FINAL
CONFIRMATORY SAMPLING WORK PLAN FORT STEWART HINESVILLE, GEORGIA

MARCH 2007

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DOCUMENT 2

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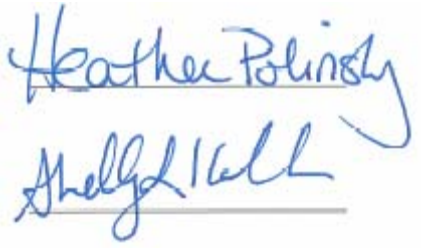

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MARCH 2007

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TABLE OF CONTENTS

TABLE OF ACRONYMS ..... **A**

1 INTRODUCTION **1-1**

1.1 PROJECT OBJECTIVES 1-2
1.2 PROJECT MANAGEMENT 1-3
1.2.1 Project Schedule 1-3
1.2.2 Project Personnel 1-3
1.2.2.1 Malcolm Pirnie Project Personnel 1-3
1.2.2.2 Other Project Personnel 1-5
1.2.2.3 Subcontractors 1-6
1.3 WORK PLAN ORGANIZATION 1-6

2 PROJECT OVERVIEW **2-1**

2.1 HRR 2-1
2.2 TPP PROCESS/STAKEHOLDER DATA QUALITY OBJECTIVE PROCESS 2-3
2.3 CS FIELD ACTIVITIES 2-6
2.4 PROJECT DELIVERABLES 2-7

3 TECHNICAL APPROACH **3-1**

3.1 MEC ACTIVITIES 3-1
3.1.1 Instrument Assisted Visual Survey 3-1
3.1.2 Triggers for Immediate Response 3-3
3.2 MC ACTIVITIES 3-4
3.2.1 Surface Soil/Sediment Sampling 3-5
3.2.2 Surface Water Sampling 3-6
3.2.3 Chemistry Analyses 3-7
3.3 UTILITY CLEARANCE 3-8
3.4 GPS SURVEYING 3-8
3.5 FIELD EQUIPMENT 3-9
3.6 LABORATORY ANALYSIS 3-9
3.7 QA/QC SAMPLES 3-10
3.7.1 QC Samples 3-10
3.7.2 Field Duplicate Samples 3-11
3.8 SAMPLING EQUIPMENT DECONTAMINATION 3-12
3.8.1 Decontamination Procedures/Sample Contaminant Sources 3-12
3.8.2 Reagents 3-12
3.8.3 Sample Contaminant Sources and Other Potential Problems 3-13
3.9 HEALTH & SAFETY 3-13

4 FIELD ACTIVITIES **4-1**

4.1 ANTI – AIRCRAFT RANGE -1 4-1
4.1.1 Site Description 4-1
4.1.2 Proposed MEC/MC Activities 4-1
4.2 ANTI – AIRCRAFT RANGE – 90MM - 2 4-2

4.2.1 Site Description 4-2
4.2.2 Proposed MEC/MC Activities 4-3
4.3 ANTI – TANK RANGE 90MM 4-3
4.3.1 Site Description 4-3
4.3.2 Proposed MEC/MC Activities 4-4
4.4 HAND GRENADE COURSE 4-4
4.4.1 Site Description 4-4
4.4.2 Proposed MEC/MC Activities 4-5
4.5 SMALL ARMS RANGE -1 4-5
4.5.1 Site Description 4-5
4.5.2 Proposed MEC/MC Activities 4-6
4.6 SMALL ARMS RANGE – 2 4-6
4.6.1 Site Description 4-6
4.6.2 Proposed MEC/MC Activities 4-7
4.7 SMALL ARMS RANGE - 3 4-7
4.7.1 Site Description 4-7
4.7.2 Proposed MEC/MC Activities 4-7
4.8 HERO ROAD TRENCH AREA 4-8
4.8.1 Site Description 4-8
4.8.2 Proposed MEC/MC Activities 4-8
4.9 SUMMARY OF FIELD ACTIVITIES 4-9

5 SITE-SPECIFIC QAPP 5-1

6 SAMPLE MANAGEMENT AND ANALYSIS 6-1

6.1 FIELD OPERATIONS DOCUMENTATION 6-1
6.2 DQCR 6-1
6.3 FIELD NOTEBOOKS 6-1
6.4 SAMPLE NUMBERING SCHEME 6-1
6.5 SAMPLE LABELS 6-2
6.6 COC 6-3
6.7 SAMPLE PACKAGING AND SHIPPING REQUIREMENTS 6-3
6.8 INVESTIGATIVE DERIVED WASTE (IDW) 6-5

7 REFERENCES 7-1

LIST OF TABLES

Table 1-1: Project Schedule 1-3
Table 1-2: Project Personnel 1-4
Table 1-3: Other Project Personnel 1-5
Table 2-1: Summary of MEC TPP Decisions 2-4
Table 2-2: Summary of MC TPP Decisions 2-5
Table 3-1: MEC Factors for Immediate Response Actions 3-4
Table 3-2: Field Equipment 3-9
Table 3-3: Quantities of Analysis 3-11

Table 4-1: Field Sample Summary Table 4-9
Table 5-1: Applicable Regulatory Standards and Comparison Values by Sampling Media 5-1
Table 5-2: Solid Laboratory Limits and Applicable Standards 5-2
Table 5-3: Aqueous Laboratory Limits and Applicable Standards 5-2
Table 5-4: EPA Method 6010B QC Limits 5-4
Table 5-5: EPA Method 8330A QC Limits 5-5
Table 5-6: Laboratory Control Sample (LCS) and Matrix Spike (MS) Control Limits for 8330 Solid Matrix 5-7
Table 5-7: Inorganic Analysis by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Methods 6020 5-7

LIST OF FIGURES

Figure 3-1: CRREL seven-sample wheel diagram 3-5

LIST OF MAPS

Map 2-1: Overview of MRSs 2-9
Map 4-1: Anti – Aircraft Range - 1 4-10
Map 4-2: Anti – Aircraft Range 90mm - 2 4-11
Map 4-3: Anti – Tank Range 90mm 4-12
Map 4-4: Hand Grenade Course 4-13
Map 4-5: Small Arms Range - 1 4-14
Map 4-6: Small Arms Range - 3 4-15
Map 4-7: Hero Road Trench Area 4-16

LIST OF APPENDICES

Appendix A: Quality Assurance Program Plan
Appendix B: Health and Safety Plan
Appendix C: Technical Project Planning Session Meeting Minutes
Appendix D: MEC/Multiple Anomaly Discovery Sheet
Appendix E: HRR Conceptual Site Models

TABLE OF ACRONYMS

Acronym	Definition
AEC	Army Environmental Command
CCC	Criterion Continuous Concentration
CMS	Corrective Measures Study
COC	Chain of Custody
CRREL	Cold Regions Research Engineering Laboratory
CS	Confirmatory Sampling
CSM	Conceptual Site Model
CTC	Cost to Complete
CTT	Closed, Transferring, and Transferred
DI	Deionized
DoD	Department of Defense
DPW	Directorate of Public Works
DQCR	Daily Quality Control Report
DQO	Data Quality Objective
EOD	Explosive Ordnance Disposal
EPA	Environmental Protection Agency
ERIS	Environmental Restoration Information System
FPM	Field Project Manager
FS	Feasibility Study
FTSW	Fort Stewart
GA	Georgia
GAEPD	Georgia Environmental Protection Division
GM	Guided Missile
GPS	Global Positioning System
HE	High Explosives
H&S	Health and Safety
HASP	Health and Safety Plan
HRR	Historical Records Review
HSD	Health and Safety Director
IDW	Investigative Derived Wastes
MC	Munitions Constituents
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
mg/kg	Milligram per Kilogram
MIDAS	Munitions Items Disposition Action System
mm	Millimeter
MMRP	Military Munitions Response Program
MR	Munitions Response
MRS	Munitions Response Site
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NA	Not Applicable

Acronym	Definition
NCO	Non Commissioned Officer
NFA	No Further Action
OE	Ordnance and Explosives
PM	Project Manager
PMP	Project Management Plan
POC	Point of Contact
PRG	Preliminary Remediation Goals
PWS	Performance Work Statement
QA	Quality Assurance
QAPP	Quality Assurance Program Plan
QC	Quality Control
RCRA	Resource Conservation Recovery Act
RFI	RCRA Facilities Investigation
RI	Remedial Investigation
RL	Reporting Limit
SEIMA	Southeast Installation Management Command
SI	Site Inspection
SSC	Site Safety Coordinator
SS-HASP	Site-Specific Health and Safety Plan
SS-QAPP	Site-Specific Quality Assurance Program Plan
TAL	Target Analyte List
TPP	Technical Project Planning
U.S.	United States
USACE	United States Army Corps of Engineers
UXO	Unexploded Ordnance

1 INTRODUCTION

Malcolm Pirnie, Inc. (Malcolm Pirnie) has prepared this Resource Conservation Recovery Act (RCRA) Confirmatory Sampling (CS) Work Plan for the Military Munitions Response Program (MMRP) eligible sites at Fort Stewart (FTSW), Georgia (GA), under United States (U.S.) Army Corps of Engineers (USACE) Contract Number W912 DR 05 D 0004, Delivery Order 26. This CS Work Plan is intended to meet the requirements of a MMRP Site Inspection (SI) Work Plan.

FTSW consists of 279,081 acres and is located north of Hinesville, GA, approximately 40 miles southwest of Savannah, GA. FTSW is the largest Army installation east of the Mississippi River, spanning portions of Bryan, Evans, Liberty, Long, and Tattnall counties. FTSW is bisected by Georgia Highway 119, which runs north to south from Pembroke to Hinesville and Georgia Highway 144, which runs east to west from Richmond Hill to Glennville. Situated south of Interstate 16 and west of Interstate 95, the installation boundaries are roughly defined by the intersection of Interstate 16 and Interstate 95 and the cities of Richmond Hill, Hinesville, Glennville, Claxton, and Pembroke.

Currently, the mission of FTSW is to sustain a quality of life and reservation support at the level necessary for divisions and non-divisional, tenant, and Reserve Component units to accomplish their training missions.

This Work Plan has been developed to provide a description of the tasks necessary to complete this project and to ensure that the project will conform with the USACE, Baltimore District project Performance Work Statement (PWS), dated 1 December 2005 and the Final Project Management Plan (PMP) dated 21 April 2006. In addition, this Work Plan incorporates the resolutions and ideas generated during the review and development process for this project. This Work Plan includes the following project specific information:

- Project objectives
- Project management
- Schedule
- Personnel

- Site location and history
- Field work
- Laboratory analyses
- Health and safety

The Quality Assurance Program Plan (QAPP) (Appendix A), Health and Safety Plan (HASP) (Appendix B), and Technical Project Planning (TPP) Meeting Minutes (Appendix C) are incorporated in this Work Plan.

This Work Plan will be used with the understanding that unanticipated conditions may dictate a change in the plan as written. Any necessary deviations from the plan will be brought to the attention of the USACE, Baltimore District Project Manager (PM) as soon as possible, and a written request for variance will be submitted to document the decision made.

1.1 PROJECT OBJECTIVES

The purpose of this project is to determine the presence or absence of munitions and explosives of concern (MEC) and munitions constituents (MC) that may remain from activities conducted by the Department of Defense (DoD) during operation of these sites and that may pose a threat to human health and/or the environment. The CS Work Plan and CS Report are intended to meet the goal of a MMRP SI Work Plan and SI Report. The primary goal of a MMRP SI and this CS is to collect information necessary to make one of the following decisions: 1) whether a RCRA Facilities Investigation (RFI)/Corrective Measures Study (CMS) is required at a MRS; 2) whether an immediate response is needed; or 3) whether the MRS qualifies for no further action (NFA). The CS Report at FTSW will investigate the explosive safety threat posed by MEC at the MMRP eligible sites (Munitions Response sites [MRSs or MRS]). It will also investigate human and ecological heath risks and environmental impacts associated with MC contamination at the MRSs on FTSW. The secondary goal of the CS is to collect information to complete the Cost to Complete (CTC) estimates and data to apply the MRS Prioritization Protocol for the MRSs. The data collected for this CS Report will be used to meet the secondary goal of the SI.

1.2 PROJECT MANAGEMENT

Malcolm Pirnie will provide all of the documents and will participate in all of the meetings and conference calls in accordance with the protocols stated in the USACE, Baltimore District project PWS and the PMP. The project schedule and personnel involved are outlined below.

1.2.1 Project Schedule

The project schedule has been established according to the performance of the following tasks as delineated by the USACE, Baltimore District project PWS.

- Task 1 – Stakeholder involvement
- Task 2 – Historical Records Review (HRR)
- Task 3 – TPP
- Task 4 – CS

The project schedule/status is provided in **Table 1-1**.

Table 1-1: Project Schedule

Task Status	Task	Completion Date
Complete	Kick-Off Meeting	04/18/06
Complete	Stakeholder Involvement	04/18/06
Complete	Stakeholder Draft HRR	08/16/06
Complete	Host TPP Session 1	09/12/06
Complete	Final HRR	09/29/06
Complete	Stakeholder Draft Work Plan	01/05/07
Complete	Final TPP Memo	11/27/06
Planned	Final Work Plan	03/02/07
Planned	CS MEC/MC Field Work	03/13/07 – 03/15/07
Planned	Stakeholder Draft CS Report	08/21/07
Planned	Host TPP Session 2	10/03/07
Planned	Final CS Report	11/12/07

1.2.2 Project Personnel

1.2.2.1 Malcolm Pirnie Project Personnel

Malcolm Pirnie project personnel and their responsibilities are listed in **Table 1-2**.

Table 1-2: Project Personnel

Name	Title
Heather Polinsky	Malcolm Pirnie Program Manager
Joseph Golden	Malcolm Pirnie Corporate Health and Safety (H&S) Director (HSD)
Shelly Kolb	Malcolm Pirnie PM
David Smith	Deputy/Field PM (FPM)
Marla Miller	Malcolm Pirnie Project Chemist
Dan Hains	Field personnel - MEC survey/ Unexploded Ordnance Health and Safety Supervisor (UXOSS)
Nicole Ukura	Field personnel - MC sampling

Malcolm Pirnie Program Manager – Heather Polinsky

The Malcolm Pirnie Program Manager oversees the Malcolm Pirnie PM and reports directly to the USACE, Baltimore District PM. Any issues or problems the USACE, Baltimore District may experience with the Malcolm Pirnie PM may be addressed to the Malcolm Pirnie Program Manager. The Malcolm Pirnie Program Manager has full authority over the performance of the project and can direct changes in project implementation.

Malcolm Pirnie Corporate HSD – Joseph Golden

The Malcolm Pirnie Corporate HSD maintains the organizational freedom and authority for ensuring full implementation of the Site-Specific Health and Safety Plan (SS-HASP) and Malcolm Pirnie's corporate H&S policy. The HSD can direct how the SS-HASP is implemented. This can include delegating authority to other personnel and directing the enforcement of the SS-HASP, including removing individuals from the project for non-compliance.

Malcolm Pirnie PM – Shelly Kolb

The Malcolm Pirnie PM has ultimate responsibility for all aspects of the project and reports directly to the Malcolm Pirnie Program Manager, Malcolm Pirnie Corporate HSD, and the USACE, Baltimore District PM. The Malcolm Pirnie PM is also responsible for project personnel safety and health, including correction of all identified unsafe acts or conditions and enforcement of procedures and regulations.

Malcolm Pirnie Deputy/FPM –David Smith

The Malcolm Pirnie FPM is the primary contact for performance of field activities. The FPM is responsible for work with field staff for the implementation of the Work Plan, including the project quality assurance/quality control (QA/QC) requirements. The FPM will be on-site during field activities.

Malcolm Pirnie UXOSS– Dan Hains

The Malcolm Pirnie UXOSS reports to the Malcolm Pirnie PM for all aspects of the fieldwork and is responsible for enforcing all aspects of safety and health rules, policies, and procedures on behalf of Malcolm Pirnie.

Malcolm Pirnie Project Chemist – Marla Miller

The Project Chemist is responsible for the day to day management of the data at all stages to ensure that all project activities related to analytical data are performed to meet the project data quality objectives (DQOs).

1.2.2.2 Other Project Personnel

Table 1-3 lists the individuals and associated agencies/organizations also involved with this project. They are also included in the document distribution list.

Table 1-3: Other Project Personnel

Name	Org Code	Title	Work Phone
Army Environmental Command (AEC)			
Timothy Rodeffer	SFIM-AEC-CDP	MMRP Project Manager	410-436-1530
Alan Freed	SFIM-AEC	Restoration Manager	410-436-0498
USACE, Baltimore District			
Kimberly Gross	CENAB-EN-HM	PM	410-962-6735
FTSW			
Algeana Stevenson	Hunter Army Airfield (HAAF) DPW, Environmental	IRP Manager	912-315-5227
Randy Powell-Jones	FSTW DPW	Restoration Manager	912-315-5109
Southeast Installation Management Command (SEIMCOM)			
Michael Riegert	SFIM-NE-PW-ER	Southeast Installation Management Command	404-464-0789

Name	Org Code	Title	Work Phone
Georgia Environmental Protection Division (GAEPD)			
Benoit Causse	Hazardous Waste Center Management Branch	Environmental Engineer	404-463-7513

1.2.2.3 Subcontractors

Subcontractors report to the Malcolm Pirnie FPM and UXOSS during the performance of the tasks associated with their fieldwork and are responsible for complying with the project Work Plan while on-site. Analytical Laboratory Services, Inc., a National Environmental Laboratory Accreditation Program certified laboratory (see Appendix A for full certification list), has been hired by Malcolm Pirnie to help complete this project. Laboratory qualifications are provided in the QAPP.

1.3 WORK PLAN ORGANIZATION

Including **Section 1: Introduction**, the Work Plan consists of seven sections and five appendices. The remaining six sections and appendices of the Work Plan are outlined below:

- **Section 2: Project Overview** discusses the proposed activities to be conducted by Malcolm Pirnie as part of the CS.
- **Section 3: Technical Approach** outlines methods and overall QA/QC procedures.
- **Section 4: Field Activities** presents a detailed description of each MRS and site-specific field activities for the CS.
- **Section 5: Site-Specific QAPP (SS-QAPP)** outlines site-specific sampling information and any exceptions or proposed changes to the QAPP.
- **Section 6: Sample Management and Analysis** outlines field guidelines, including QA/QC associated with sample management. This section includes sample packaging and shipping requirements and investigative derived wastes (IDW) procedures.
- **Section 7: References**
- **Appendix A: QAPP**
- **Appendix B: HASP**
- **Appendix C: TPP Meeting Minutes**
- **Appendix D: MEC/Multiple Anomaly Discovery Sheet**
- **Appendix E: HRR Conceptual Site Model**

2 PROJECT OVERVIEW

The MMRP SI process, being conducted under RCRA correction action process for Fort Stewart, consists of five primary tasks which include the HRR, TPP, CS Work Plan, CS fieldwork, and CS Report.

HRR – consists of identifying data gaps from the U.S. Army's Phase 3 Closed, Transferring, and Transferred (CTT) Inventory and obtaining and reviewing historical records**.** The HRR is aimed at developing a draft Conceptual Site Model (CSM), focusing field work, and providing a common understanding of the MRS.

TPP – consists of planning activities conducted with the stakeholders to identify project objectives and designing data collection programs to meet objectives.

CS Work Plan – consists of preparing and submitting a site-specific Work Plan document reflecting the agreements made during the TPP session.

CS fieldwork – consists of performing investigation activities and preparing reports of findings as described in this Work Plan.

CS Report – consists of preparing and submitting an CS Report summarizing the results of the fieldwork, to include an updated CSM developed for each MRS with an appendix containing all information necessary to complete the MRS Prioritization Protocol.

2.1 HRR

A HRR for FTSW was finalized on September 29, 2006 in support of CS. This document expanded on the information collected during the Phase 3 CTT Range Inventory and provided information pertinent to identifying, verifying, and establishing the physical limits and potential MEC and MC for each MRS. Historical records, aerial photos, existing site maps, and existing environmental restoration documents were reviewed, and interviews with installation personnel

were completed. An existing installation-specific background study, including sample analysis for metals, was reviewed. The following information is provided in the HRR:

- Project purpose/scope
- Project drivers
- Installation description/history
- Phase 3 CTT Range Inventory results
- Data collection and document review process
- MRS descriptions/HRR findings
- Draft CSM
  - MMRP site profile
    - Area and layout
    - Structures
    - Utilities
    - Boundaries
    - Security
  - Physical profile
    - Climate
    - Geology
    - Topography
    - Soil
    - Hydrogeology
    - Hydrology
    - Vegetation
  - Land use and exposure profile
    - Human receptors (current and future)
    - Zoning/land use restrictions
    - Beneficial resources
    - Demographics
  - Ecological profile
    - Habitat type
    - Degree of disturbance
    - Ecological receptors
  - Munitions/release profile
    - Munitions types and release mechanisms
    - Maximum probable penetration depth
    - MEC density
    - Munitions debris
    - Associated MC
    - Transport mechanisms/migration routes
  - Pathway analyses for MEC and MC
- Conclusions

The findings of the HRR resulted in seven sites being identified from the Phase 3 Range Inventory:

- Anti-Aircraft Range - 1
- Anti-Aircraft Range 90-millimeter (mm) - 2
- Anti-Tank Range 90-mm
- Hand Grenade Course
- Small Arms Range - 1
- Small Arms Range - 2
- Small Arms Range - 3

One additional MRS, the Hero Road Trench Area, previously identified by Installation personnel was researched and included in this HRR.

As a result of the research conducted for the HRR, the Small Arms Range – 2 was found to be ineligible for the MMRP as it is positioned completely within the operational footprint.

As a result of the findings of the HRR, there are a total of seven MMRP eligible sites (488 acres) at FTSW. The Final Fort Stewart Historical Records Review Report was submitted on 29 September 2006. Comments from the USACE, Baltimore District; AEC, FTSW, and the stakeholders were incorporated into the Final HRR Report. The MRSs identified in the HRR are presented on Map 2-1. Summaries of each MRS are provided in Section 4 of this Report.

2.2 TPP PROCESS/STAKEHOLDER DATA QUALITY OBJECTIVE PROCESS

The TPP process is a comprehensive and systematic process that involves four phases of planning activities. It was developed for identifying project objectives and designing data collection programs. Use of the TPP process is consistent with the philosophy of taking a graded approach to planning that will produce the type and quality of results needed for site-specific decision-making.

A TPP session was held at FTSW on September 12, 2006. The results of the TPP session dictated the MEC and MC sampling/field activities planned for the installation. Table 2-1

provides a summary of decisions made to address MEC, and Table 2-2 provides a summary of decisions made to address MC. The Final Meeting Minutes from the September 12, 2006, TPP are included in Appendix C.

Table 2-1: Summary of MEC TPP Decisions

MRS	MEC CS Activities	
	Activity	Purpose
Anti – Aircraft Range - 1	Magnetometer assisted visual survey during sampling activities	Support MEC no further action (NFA) or RFI/CMS determination Recommend NFA if no MEC is encountered on the surface Recommend RFI/CMS if MEC is encountered on the surface
Anti – Aircraft Range 90mm - 2	Magnetometer assisted visual survey during sampling activities	Recommend RFI/CMS for MRS based on historical evidence of multiple overlapping range fans and multiple explosive ordnance disposal (EOD) responses.
Anti – Tank Range 90mm	Document historical use in Installation Master Plan	Recommend NFA under the MMRP because current/future use as a RCRA permitted landfill.
Hand Grenade Course	Magnetometer assisted visual survey during sampling activities	Recommend RFI/CMS for MRS based on historical evidence of multiple overlapping range fans.
Small Arms Range - 1	No MEC field activities are required because only small arms were used at the MRS.	
Small Arms Range - 2	No MEC field activities are required because only small arms were used at the MRS.	
Hero Road Trench Area	Conduct a visual survey of unfenced portions of MRS to ensure no MEC or MEC debris remains on the surface.	Recommend RFI/CMS for MRS based on historical evidence and results of current investigation.

Table 2-2: Summary of MC TPP Decisions

MRS	MC CS Activities	
	Activity[1]	Purpose[2]
Anti – Aircraft Range - 1	Collect 4 composite surface soil samples Sample locations will be randomly distributed unless biased locations are identified. Analyze for explosives and metals using Environmental Protection Agency (EPA) Methods 8330 and 6010B/6020	Support CTC/Prioritization Protocol. Support MC NFA or RFI/CMS determination. Screen data using: • FTSW Inorganic/Metal Background Study • EPA Region 9 Preliminary Remediation Goal (PRG) for Residential Soil • Region 4 Ecological Screening Values for surface soil
Anti – Aircraft Range 90mm - 2	Collect 1 biased composite surface soil sample at the location of one of the EOD response locations. Analyze for explosives and metals using EPA Methods 8330 and 6010B/6020	Support CTC/Prioritization Protocol. RFI/CMS recommended for MRS based on historical evidence of multiple overlapping range fans and multiple EOD responses. Compare data to: • FTSW Inorganic/Metal Background Study • EPA Region 9 PRG for Residential Soil • Region 4 Ecological Screening Values for surface soil
Anti – Tank Range 90mm	None	Recommend NFA because RCRA permitted landfill is currently being monitored under the RCRA program.
Hand Grenade Range	Collect 1 biased composite surface soil sample in the center of the MRS. Analyze sample for explosives and metals using EPA Methods 8330 and 6010B/6021.	RFI/CMS recommended for MRS based on historical evidence of multiple overlapping range fans.
Small Arms Range - 1	Collect 4 composite surface soil samples collected in the undeveloped portions (~41 acres) of the MRS. Antimony and Lead by EPA Method 6020	Support CTC/Prioritization Protocol. Support MC NFA or RFI/CMS determination. Screen data using: • FTSW Inorganic/Metal Background Study • EPA Region 9 PRG for Residential Soil • Region 4 Ecological Screening Values for surface soil

MRS	MC CS Activities	
	Activity[1]	Purpose[2]
Small Arms Range -3	Collect 2 sediment, 2 surface water and 3 composite surface soil samples. Soil samples: 1 in northern and 2 in the southern portions. Sediment samples: 1 on each of the man-made damns of the pond. Antimony and Lead by EPA Method 6020	Support CTC/Prioritization Protocol. Support MC NFA or RFI/CMS determination. Screen data using: • FTSW Inorganic/Metal Background Study • EPA Region 9 PRG for Residential Soil • Region 4 Ecological Screening Values for surface soil • EPA Water Quality Standards for Freshwater Criterion Continuous Concentration (CCC) chronic • Region 4 Ecological Screening Values for surface water
Hero Road Trench Area	Collect 1 composite surface soil sample Explosives and metals using EPA Methods 8330 and 6010B/6020	Support CTC/Prioritization Protocol. RFI/CMS recommended for the MRS based on historical evidence and results of current investigation. Screen data using: • FTSW Inorganic/Metal Background Study • EPA Region 9 PRG for Residential Soil • Region 4 Ecological Screening Values for surface soil

1 As per an agreed upon decision made after the TPP meeting, the analysis for the full Target Analyte List (TAL) metals list will not be conducted. The metals analysis will be limited to primary or indicator compounds associated with the munitions history of each MRS. Aluminum, antimony, copper, lead and zinc have been identified as primary or indicator compounds for the munitions associated with the FTSW MRSs and the metals analysis will be limited to these compounds. The primary MC for the munitions items was determined utilizing the U.S. Army Technical Manuals 43-0001-28, 43-0001-29, 43-0001-30, and the Munitions Items Disposition Action System (MIDAS) database created by the Defense Ammunition Center Technology Directorate. For MRSs where historical evidence indicates small arms use only metals analysis will be limited to lead as agreed upon during the TPP meeting.
2 As per an agreed upon decision made after the TPP meeting, additional screening values including ecological soil / surface water and human surface water criteria were added and are presented.

2.3 CS FIELD ACTIVITIES

The goal of this project is to determine the presence or absence of MEC and MC that may remain from activities conducted by the DoD during operation of these sites and that may pose a threat to human health and/or the environment.

During the field sampling event, qualified team members (UXO Technicians III) will inspect the surface for MEC and provide anomaly avoidance support. Samples will be collected to analyze

for metals and explosives as dictated by historical site activities. The fieldwork will take place during March 2007 and will last approximately three days.

It is anticipated that 14 surface soil, two sediment, and two surface water samples will be collected for analytical laboratory analysis. The analytical methods were selected on the basis of the types of munitions known to have been used at the MRS and include the standard suite of range-related analytical parameters to account for unknown items. The standard analytical methods include metals (Aluminum, antimony, copper, lead and zinc) (EPA Methods 6010B and 6020), explosives (EPA Method 8330). Method 6010B will be used for the analysis of aluminum, copper, and zinc. Method 6020 will be used for the analysis of antimony and lead. Method 6020 will be used in lieu of 6010B to achieve the reporting limits consistent with the screening criteria agreed upon at the TPP session. All field and laboratory work will be of the quality to support screening against the following in the listed order:

- FTSW Inorganic/Metal Background Study (April 2000)
- EPA Region 9 PRG for Residential Soil
- Region 4 Ecological Screening Values for Surface Soil
- EPA Water Quality Standards for Freshwater CCC Chronic
- Region 4 Ecological Screening Values for Surface Water

2.4 PROJECT DELIVERABLES

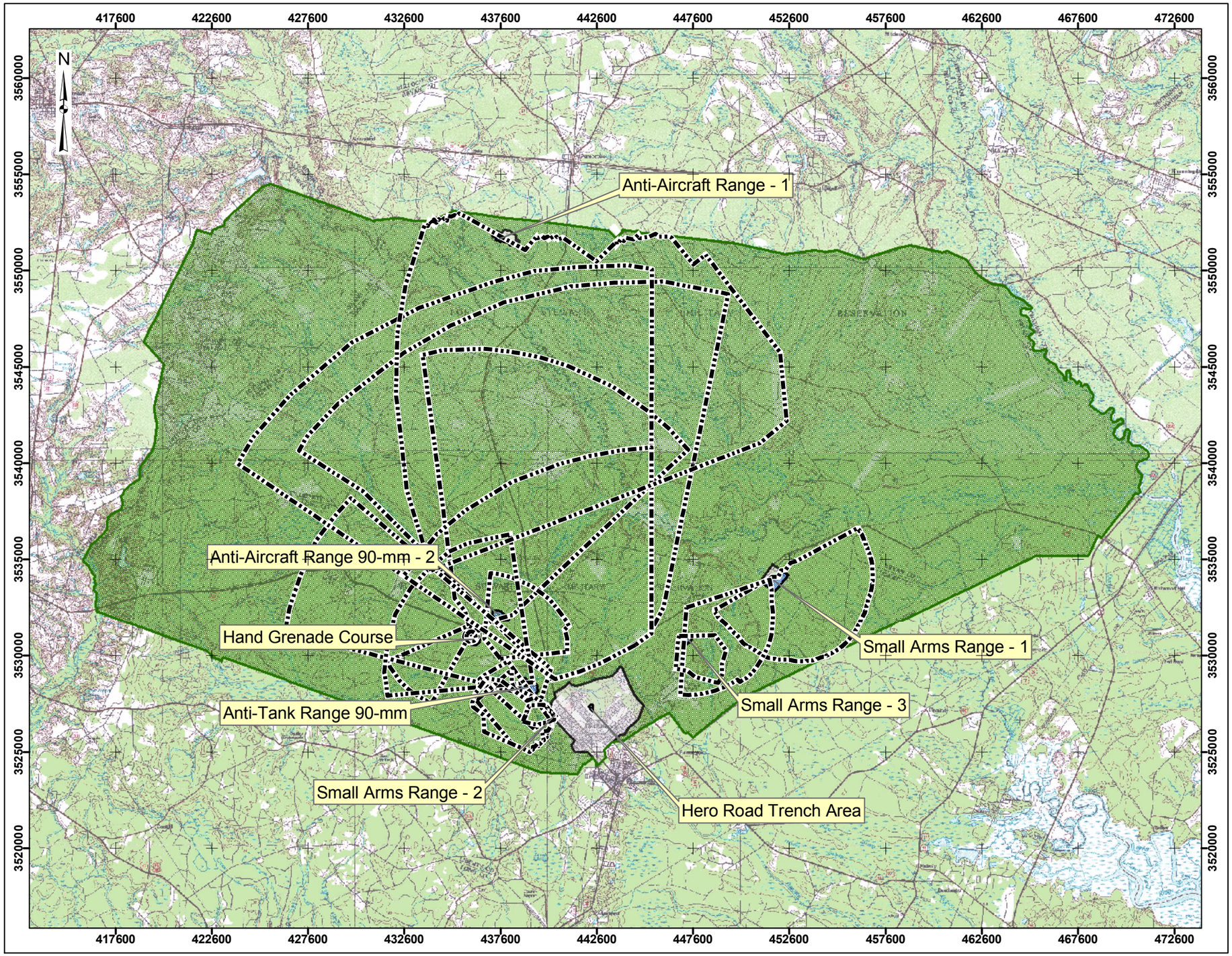
In addition to this Work Plan, Malcolm Pirnie will develop and submit an CS Report, which will include the:

- Final CSM;
- Analytical data; and
- Results of instrument assisted site walk.

In accordance with the PWS, all the analytical data generated during this field effort will be uploaded into the U.S. Army's Environmental Restoration Information Systems (ERIS) web-

based database. The data will include the following information for each sample collected: sample identification number; preservation; date sampled; media type; site location; chemical analyses; and validation review. The format requirements for the ERIS database are in the QAPP (Appendix A). If the ERIS database format is revised during MMRP investigations, the newly established database format shall be included as an appendix to the QAPP.

SI Work Plan
Fort Stewart, GA



Map 2-1
HRR Overview of Ranges

Legend

- Installation Boundary

Military Range Area

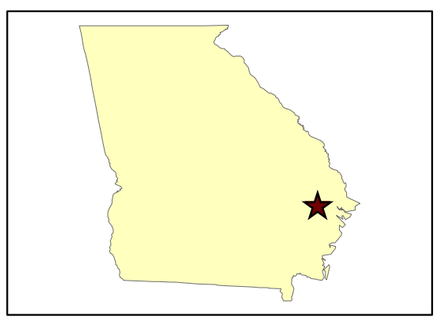
- Closed
- Operational Range Area
- Non Range, Non UXO-DMM-MC Area
- Historical Impact Areas

0 2,750 5,500 8,250 11,000 Meters

Data Source: Fort Stewart, GA

Coordinate System: UTM Zone 17N
Datum: NAD 83
Units: Meters

Contract: DACA31-00-D-0043
Edition: Final Work Plan
Date: March 2007



3 TECHNICAL APPROACH

The sampling rationale/design for the CS is to collect sufficient data to confirm the presence/absence of MEC or MC within the areas of concern. Based upon the objectives of this CS, the following items have been incorporated into the sampling program rationale/design.

3.1 MEC ACTIVITIES

This portion of the fieldwork should be such that exclusion zone impacts, engineering control requirements, clearing and grubbing efforts, and MEC disposal activities are not required. In some cases, encountering just one MEC item will be sufficient to determine that further investigation is necessary for a particular MRS. The field activities for the CS are not intended to confirm all types of MEC present, determine MEC density, or define the exact limits of the MEC impacts. The areas over which MEC activities will be conducted are discussed in detail in Section 4.

MEC that are discovered during sampling activities will not be removed, disturbed, or otherwise compromised. The sampling team will make a photographic record of the MEC item and make field notes indicating the location of the item, its conditions, and any other pertinent information. The location of the MEC item will be recorded with GPS equipment. This information will be recorded on the MEC/Multiple Anomaly Form which is provided in Appendix D. The field crew will notify the DPW, AEC, and USACE, Baltimore District of any MEC items encountered at the completion of field activities each day. If multiple MEC items are encountered during the field activities DPW and USACE, Baltimore District will be contacted to decide how to proceed.

3.1.1 Instrument Assisted Visual Survey

A limited instrument assisted visual survey of the suspected MEC sites (listed in Section 4) will be performed to locate and document MEC found during the site walk. Field team personnel will conduct the visual survey while being escorted by an UXO Technician III. This activity will be limited to a surface walkover to identify materials and/or surface features that provide information on the areas and activities in question.

A Schonstedt handheld magnetometer will be used to conduct the limited survey and detect surface MEC (primarily used for MEC anomaly avoidance for safety purposes). A transect sweep approach will be used to search the identified MRS, depending on the terrain and layout. Each transect will be approximately 5 feet in width and spaced 40 feet apart, depending on the terrain, vegetation, and line-of-site. A perimeter survey may also be conducted for visual evidence of munitions impacted areas or release of other constituents off-site. Site-specific details are provided in Section 4 for each MRS.

The following steps will be conducted during the site walk:

- Prior to entering an area requiring anomaly avoidance, the UXO Technician III will conduct a tailgate safety brief. This brief will cover emergency procedures, operations, types of suspected MEC that may be encountered during the site visit, and anomaly avoidance procedures.
- The UXO Technician III will enter the site first and will conduct a surface sweep of the path as the survey team follows behind in a single file. The team will identify target areas containing MEC, to include discarded military munitions, munitions debris and masses of buried materials.
- Target areas containing MEC will be marked and documented.
- Survey of firing points (where appropriate) will be documented, the Global Positioning System (GPS) locations will be recorded, and the areas will be photographed.
- The survey team will observe the area for pits, craters, and unusual holes—these could indicate impact areas, demolition sites or burial pits. These areas will be documented using the MEC/Multiple Anomaly Discovery Form, the GPS locations will be recorded, and the areas will be photographed.
- If MEC are discovered, the UXO Technician III will mark the item, GPS coordinates for the item will be recorded, and the MEC item will be logged as to its description, size, color, and any other distinguishable marks. Pertinent data will be entered on an MEC/Multiple Anomaly Discovery Form. A digital photograph of the item will be taken, and the photo number and item description will be noted in the logbook. At no time will the MEC item be moved or disturbed. After collecting the necessary data, the team will proceed with its survey.
- If any live or suspected live MEC are encountered during the limited visual survey, they will be marked for positive identification, and an immediate response trigger evaluation described in Section 3.1.2 will be performed. The FTSW Directorate of Public Works (DPW), AEC, and USACE, Baltimore District will be notified if any MEC item is encountered during fieldwork

The following function check procedures will be used to perform function tests on the equipment used during the visual survey:

- Hand-held metal detectors (i.e., Schonstedt,) will be swept across known selected items within an area outside of the site to demonstrate consistent effectiveness.
- Instruments and equipment used to gather and generate data will be tested with sufficient frequency and in such a manner as to ensure that accuracy and reproducibility of results are consistent with the manufactures' specifications. Instruments or equipment failing to meet the standards will be repaired, recalibrated, or replaced. Replaced instruments or equipment must meet the same specifications for accuracy and precision as the item removed from service.

In addition an all metals detector assisted visual survey will be conducted in order to locate remnants of small arms rounds that may remain. A transect sweep approach will be used to search the identified MRS. Each transect will be approximately 5 feet in width and spaced 40 feet apart, depending on terrain, vegetation, and line-of-site.

3.1.2 Triggers for Immediate Response

MEC removals will not be conducted as part of the CS. However, the field team may encounter MEC and munitions debris during site reconnaissance. An UXO Technician III will accompany the data collection team and provide MEC escort services for all data collection personnel. Any MEC and munitions debris that is encountered will be identified to help characterize the MEC and/or MC at the MRS. Under no circumstances will MEC be handled, moved, or disturbed during the visual survey. Any MEC items encountered during the CS field activities will be reported to FTSW EOD. FTSW EOD will be responsible for disposal of MEC items encountered and reported.

The CS fieldwork is not intended to include removal or disposal actions; however, if identified, an MEC or explosives hazard must be reported, and a decision must be made about its disposition, if any. The decision is based on the overall threat to human health and the environment. The level of threat is based on an overall understanding of the situation and its risk, based on site-specific data and the factors discussed in **Table 3-1**.

Table 3-1: MEC Factors for Immediate Response Actions

MEC Factor	Status Questions
Accessibility of the MEC	Is it in an area that is restricted to the public with engineering controls that preclude entry, such as fences, security guards, or posted hazards signs? Is the MEC in an area that is accessible to the public, and does this create an imminent hazard to people or the environment?
Type of MEC	What is the condition, fuzing type, net explosive weight and specific hazards of the item? Does the MEC pose an immediate threat?
Site assessment	Do the MEC and/or MC site conditions require using protective measures such as tamping, shielding, or focusing of the heat, blast, and shockwave to mitigate the explosive effects? What is the maximum fragmentation range and over-pressure distance of the MEC?
Other considerations	Can the hazard be moved? Can the area within the fragmentation and blast distance withstand a detonation, and are there critical habitats or facilities located nearby?

For the purposes of the CS, Malcolm Pirnie will immediately report the presence of MEC and the information needed to answer the questions in **Table 3-1** for determination of the appropriate action to the USACE, Baltimore PM, AEC, and the installation point of contact (POC).

3.2 MC ACTIVITIES

The goal of the field sampling activities for MC is to determine if the MRS has been impacted by MC. Anomaly avoidance techniques will be utilized during the MC field sampling activities. Analytical results exceeding background levels and appropriate regulatory limits agreed on during the TPP session will be used for justification in moving the MRS into the RFI/CMS phase. The CS field sampling activities are not intended to determine the nature and extent of all contaminants.

All fieldwork will be of the quality needed to meet the DQOs for the project as dictated in the QAPP, the TPP Meeting Minutes, and decisions agreed upon after the TPP meeting. A decision to limit the metals analysis to primary or indicator compounds associated with the munitions history of each MRS was agreed upon after the TPP meeting. As a result of this, the metals

analysis for the FTSW MRSs will be limited to aluminum, antimony, copper, lead and zinc, which are the primary MC associated with the munitions history of these MRSs. The primary MC for the munitions items was determined utilizing the U.S. Army Technical Manuals 43-0001-28, 43-0001-29, 43-0001-30, and the MIDAS database created by the Defense Ammunition Center Technology Directorate. For MRSs where historical evidence indicates small arms use only metals analysis will be limited to antimony and lead as agreed upon during the TPP meeting. The details of the planned MEC and MC field sampling activities are provided in Section 2.

3.2.1 Surface Soil/Sediment Sampling

Surface soil samples will be composite samples based on the Cold Regions Research Engineering Laboratory (CRREL) seven-sample wheel approach (as described in CRREL Special Report 96-15, Assessment of Sampling Error Associated with Collection and Analysis of Soil Samples at Explosives-Contaminated Sites). Seven grab samples of approximately equal weight will be collected from each position along the wheel. These seven grab samples will be combined on a disposable sheet of plastic and thoroughly homogenized to form one composite sample (see **Figure 3-1**). Procedures to homogenize the seven samples to form one composite sample are detailed in CRREL Special Report 96-15. Sample locations will be biased towards areas where MEC were identified during the visual survey or areas where the highest density of munitions are expected. Random sampling will only be performed if no MEC or known high-density areas are identified.

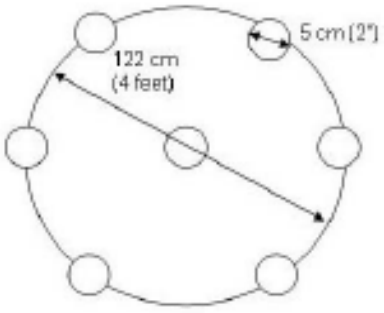


Figure 3-1: CRREL seven-sample wheel diagram

Surface soil samples will be collected with a disposable scoop or similar equipment while wearing Nitrile gloves. New scoops and gloves will be used at each sampling location. The analytical samples will be collected and placed directly into the appropriate sample containers, labeled, and placed in an ice chest chilled to a maximum temperature of 4 degrees Celsius. A portion of the sample will be set aside and used to log a description of the soil characteristics using the Unified Soil Classification System on a sample log form. After a sample is put into the ice chest, the chain of custody (COC) and Daily Quality Control Report (DQCR) forms will be filled out. The remaining soil will be disposed of on the ground surface at the locations from which they were collected. If field conditions dictate that disposable equipment cannot be used, reusable sampling equipment will be decontaminated before moving to the next sampling location. Decontamination procedures are presented in Section 4.7 in the QAPP (Appendix A) and Section 3.8 of this document. If the use of reusable equipment becomes necessary, rinse blank samples will be collected as discussed in Section 3.7 of this document and as described in the QAPP. Surface sample locations will be recorded using a handheld GPS unit.

3.2.2 Surface Water Sampling

Surface water samples will be collected directly from the water body. New disposable nitrile or latex gloves will be worn for every sampling location and discarded following sample collection. Samples will be collected from the body of water until field measurements collected indicate the pH, conductivity, temperature, and turbidity have reached equilibrium (variation between successive measurements less than 10% of the measured value). The field parameters will be measured with a water quality meter such as a Horiba U-10 Water Quality Checker or equivalent.

When collecting surface water samples, the sample bottles and bottle caps will be handled with care. The bottle will be held in one hand and the cap in the other, making sure not to touch the inside of the cap or bottle neck. The bottles and caps will be kept free of contamination from the ground or any other surfaces with which they could potentially come into contact. The presence of the Polytetrafluroethylene (PTFE) liner on the inside of the cap will be verified prior to sampling and prior to sealing the bottles after sampling is complete. The sample bottles will not be rinsed prior to collection. The water flow will not be adjusted during the sample collection.

Bottles will be filled slowly and continuously. The field crew will avoid contaminating the sample with water splash drops from the ground. The caps will be tightly secured and the absence of air within the container will be verified. All bottles will be labeled appropriately, placed into separate plastic bags, and immediately placed on ice in a cooler and maintained at 4^{o}C. The location and time of sample collection, a description of the sampled tap, field parameter monitoring results, size and type of laboratory containers, analyses requested, and observations regarding color and odor of the sample will be recorded in the field logbook at the time of sample collection.

3.2.3 Chemistry Analyses

Malcolm Pirnie will meet the project-specific DQOs for sampling and analysis and the QA/QC objectives by collecting the proper quantities and types of samples, using the correct analytical methodologies, implementing field and laboratory QA/QC procedures, and using various data validation and evaluation processes. The DQOs for each analytical method are provided in the QAPP. Laboratory requirements for the analytical methods being used for this project are provided below and in the QAPP. These procedures include requirements for sample preparation, sampling containers, preservation methods, and holding times.

The QAPP has been developed to support the sampling, analysis, and evaluation activities associated with this project. The QAPP consists of policies, procedures, specifications, standards, and documentation sufficient to produce data of quality adequate to meet the DQOs for the project, RCRA standards, and to minimize loss of data due to out-of-control conditions or malfunctions.

The QAPP has been prepared to ensure that this responsibility is met throughout the duration of this project. It addresses procedures to assure the precision, accuracy, representativeness, completeness, and comparability of field and laboratory data generated during the course of this project. It also provides a framework for evaluating existing data that may be used in this project. The QAPP defines the first stage of the QA requirements for sample and data acquisition, handling, and assessment.

QA procedures, such as tracking, reviewing and auditing, are implemented as necessary to ensure that all project work is performed in accordance with professional standards, EPA and USACE regulations and guidelines, and the specific goals and requirements stated in this Work Plan.

QC of sample collection, analysis, and assessment will be performed by technical project personnel. Laboratory equipment will be maintained and calibrated, and records of these activities will be kept in accordance with established procedures. This will include laboratory oversight by Malcolm Pirnie project personnel, as well as laboratory data and document review.

Per the EPA criteria for data quality for risk-based projects, 10% of the analytical data are required to meet a comprehensive data level of QA/QC related to sample collection, laboratory analysis, and data validation techniques. Following the processes identified in the QAPP, final data usability will be determined by the USACE Project Chemist in coordination with the Malcolm Pirnie PM and Malcolm Pirnie Project Chemist.

Overall QA review of documentation, field sampling and laboratory QC will allow determination of the acceptability of these data for use in this project.

Sample chemical analyses are discussed in greater detail in the QAPP and the SS-QAPP in Section 4.

3.3 UTILITY CLEARANCE

As requested during the TPP meeting, Malcolm Pirnie will contact the appropriate installation public works and public utility locating agency prior to conducting any soil sampling. In addition, any overhead power lines observed in the area will be avoided.

3.4 GPS SURVEYING

Each sample location will be surveyed to document the location. The GPS unit proposed for use is a Trimble GeoExplorer CE, Geo XT handheld unit. Pathfinder Office software will be used to download and post process the data to achieve sub-meter horizontal accuracy. Field conditions,

such as the number of satellites available at the reading time and density of the tree canopy, dictate the amount of time needed to acquire a reading. Coordinates will be established for each sample location to an accuracy of 1 meter.

3.5 FIELD EQUIPMENT

A variety of equipment will be used to perform the field activities for this project. Table 3-2 lists the field equipment that will be used.

Table 3-2: Field Equipment

Category	Equipment
Surface sampling	Disposable scoops (or similar), plastic sheeting, all metals detector, Schonstedt
H&S equipment	Safety boots, safety glasses, first aid kit, fire extinguisher, protective clothing, Nitrile gloves, hard hat if a danger of falling overhead objects exists.
Shipping	Packaging tape, labels, seals, COC forms, ice, zip top bags, coolers, bubble wrap, packaging material
Documentation	DQCR forms, field log book, boring logs, all applicable H&S forms
Sample containers	See Table 4-1 in the QAPP
Decontamination supplies[1]	Liquinox or Alconox detergent, potable water, deionized (DI) water, scrub brushes, decontamination tubs/buckets
GPS	Trimble GeoExplorer CE, Geo XT handheld unit

[1] If disposable equipment cannot be used, reusable sampling equipment (with decontamination supplies) will be used

3.6 LABORATORY ANALYSIS

The analytical methods are selected on the basis of the munitions items known to have been used at the MRS and include the standard suite of range-related analytical parameters to account for unknown items. As per a decision made and agreed upon after the TPP meeting the metals analysis will be limited to primary or indicator compounds associated with the munitions history of each MRS. As a result of this the metals analysis for the FTSW MRSs will be limited to aluminum, antimony, copper, lead and zinc which are the primary MC associated with the munitions history of this MRS. For MRSs where historical evidence indicates small arms use only metals analysis will be limited to copper, antimony, and lead as agreed upon during the TPP

meeting. The standard analytical methods include EPA Methods 6010B (for aluminum, copper and zinc), and 6020 (for lead and antimony) for metals, and EPA Method 8330 for explosives. Method 6020 will be used in lieu of 6010B to achieve the reporting limits consistent with the screening criteria agreed upon at the TPP session. Screening criteria are listed in the SS-QAPP.

3.7 QA/QC SAMPLES

QA and QC procedures are documented in the QAPP. QA and QC samples are samples analyzed for the purpose of assessing the quality of the sampling effort and of the analytical data. QC samples include equipment/rinsate blanks, temperature blanks, and matrix spike/matrix spike duplicates. QA samples include field duplicate samples.

3.7.1 QC Samples

Sample QC for analytical samples will be provided in the field through the use of equipment/rinsate blanks, temperature blanks, and matrix spike/matrix spike duplicates (MS/MSD). The QC samples will be handled as regular samples. In order for distinctions to be determined between study areas, the different types of samples will be submitted in separate batches for laboratory analysis. Calibrations and associated QC samples will not be mixed between sample types. Sample QC for the analytical samples will be provided in the field through the use of duplicate field samples. QC samples are used to evaluate the contract laboratory's performance. Duplicate samples are collected as a single sample, which is divided into two equal parts.

The following QC samples will be collected for analytical samples:

Matrix spikes Samples will be collected to be split in the lab and run as MS/MSD in an amount equal to at least 5% of the study area samples for laboratory analysis.

Equipment/rinsate blanks Equipment/rinsate blanks will not be collected because disposable sampling equipment will be used at the MRS. However, if field conditions dictate that equipment requiring decontamination be utilized sampling equipment will be decontaminated prior to and after each use, and equipment/rinsate blanks will be collected and analyzed in accordance with the QAPP (Appendix A) (i.e., one field blank per decontamination event per equipment type).

The number of QC samples to be collected is presented in **Table 3-3**.

3.7.2 Field Duplicate Samples

Sample QA for the analytical samples will be provided in the field through the use of field duplicate samples. QA samples are used to evaluate the contractor's laboratory performance. Duplicate samples are collected as a single sample, which is divided into two equal parts. As shown in Table-3-3, QA samples will be collected at a rate of at least 10% of the field samples collected. QA split samples will not be collected during the CS phase as discussed during negotiations between Malcolm Pirnie and USACE prior to contract award and per Malcolm Pirnie's general assumptions submitted with the cost estimate and accepted by USACE.

Table 3-3: Quantities of Analysis

Analysis	Media	Baseline Samples(1)				
		Field Samples	Matrix Spikes(2)	Matrix Spikes Duplicate(2)	Duplicate Field Samples(3)	Total Analyses
Metals(4) (aluminum, antimony, copper lead and zinc)	Soil	7	1	1	1	10
Metals (4) (antimony, copper lead and zinc)	Soil	7	1	1	1	10
Explosives	Soil	7	1	1	1	10
Metals (4) (antimony, copper lead and zinc)	Sediment	2	1	1	1	5
Metals (4) (antimony, copper lead and zinc)	Surface Water	2	1	1	1	5

(1) If equipment decontamination is necessary, then equipment blank samples must also be collected at a rate of one field blank per decontamination event per equipment type, not to exceed one per day.
(2) Two samples indicate one MS/MSD pair, collected at a rate of one pair per 10 samples.
(3) Field duplicates will be collected at a rate of one per 10 samples.
(4) As per a decision made and agreed upon after the TPP meeting the metals analysis will be limited to primary or indicator compounds associated with the munitions history of each MRS.

3.8 SAMPLING EQUIPMENT DECONTAMINATION

In an effort to achieve the highest level of QC, one time use and disposable sampling equipment will be used whenever feasible. This type of equipment includes sampling gloves, scoops, and pre-cleaned sample jars. Applicable equipment will be decontaminated as discussed in the remainder of the section.

3.8.1 Decontamination Procedures/Sample Contaminant Sources

This section provides instructions on deciding on the appropriate decontamination scheme(s) for the project field sampling equipment in order to prevent or reduce cross-contamination of project samples. The applicability of each step in a decontamination protocol will depend upon factors such as the contaminants present on-site, the subsequent analysis to be performed, and the composition of the sampling devices. The appropriateness of a decontamination protocol is vital to the eventual validity of the analytical results and decisions made based upon those results. All sampling equipment that has come in contact with a potentially contaminated media must be cleaned prior to the subsequent use of that device. Unless field conditions dictate a change in the equipment planned for use, pre-wrapped, sterile, plastic, disposable scoops will be utilized for collecting soils samples at the installation. The scoops will be used to collect one sample and then disposed of to avoid cross-contamination between samples and locations. If field conditions dictate that other sample collection methods are required and equipment decontamination becomes necessary, all equipment will be properly decontaminated prior to and following the collection of each sample. Decontamination procedures are summarized below can be found in Section 4.7 of the QAPP (Appendix A).

3.8.2 Reagents

The detergent wash is a non-phosphate detergent solution used with brushing or circulating techniques to remove gross contamination and/or used as a mild neutralizing agent. Tap water is considered a rinse-water, preferably from a water system of known chemical composition. Acid rinses are used as the inorganic solubilizing agent or as a mild neutralizing agent. These rinses are 10:1 solution of water and acid (hydrochloric acid), respectively. The solutions are prepared from reagent grade acids and DI water. Solvent rinses are used as an organic solubilizing agent. Requirements for solvent types vary depending upon the nature of known organic contamination

requiring solubilization and any impurities present within the rinse that may potentially interfere with or contribute to the subsequent analysis. All solvent rinses used must be of pesticide grade quality. Finally, the DI water is organic-free reagent water. Analyte-free water may be used as deemed appropriate.

3.8.3 Sample Contaminant Sources and Other Potential Problems

Contaminant carryover between samples and/or from leaching of the sampling devices is very complex and requires special attention. Decisions concerning the appropriateness of the device's material composition must account for these carryover or leaching potentials and whether these contaminants are of concern on the project. Disposable equipment will be used for all sampling procedures.

3.9 HEALTH & SAFETY

The HASP (Appendix B) provides general H&S procedures applicable to sampling and analytical activities to be performed at all installations where MMRP SIs are being conducted by Malcolm Pirnie (within USACE, North and South Atlantic Divisions). The HASP sets forth health and safety protocols to be used by Malcolm Pirnie employees and its subcontractors during field activities. All work will be in conformance with the HASP unless formally modified and approved by the Malcolm Pirnie UXOSS and reviewed by the Contracting Officer via a formal record of change. The intent of the HASP is to ensure the health and safety of all site personnel, the general public, and the environment. Although it is impossible to eliminate all risks, adherence to the HASP will help minimize incidents and accidents by promoting safety while maintaining productivity. It should be noted that the HASP may include discussions that are not applicable to a specific site since it is intended to encompass all sites.

It is intended that once the HASP is finalized, it will not be modified (except for programmatic changes) and will serve as a programmatic document. Site-specific sampling information and any exceptions or proposed changes to the HASP are addressed and included in the SS-HASP which is included as Attachment 1 to the HASP. The SS-HASP is not a stand-alone document from the HASP. The HASP will provide the majority of the H&S information; the SS-HASP

simply supplements the information in the HASP by providing for site-specific condition requirements.

4 FIELD ACTIVITIES

The field activities that will be completed at each of the MRSs at FTSW in order to identify whether MEC and/or MC are present were determined using the TPP process. The determination of whether further investigation is required or if a NFA determination is appropriate for each MRS will be made using a weight of evidence approach. Examples of evidence that will be included in the decision making process include historical information, analytical results (screened against established background levels, and agreed upon regulatory limits), and field observations. A brief site description and the agreed upon MEC/MC field activities are presented below for each of the seven MRSs. Map 2-1 shows the relative location of each MRS on the installation and the historical range fans that overlap and make up each of the FTSW MRSs.

4.1 ANTI – AIRCRAFT RANGE -1

4.1.1 Site Description

The MRS layout, location, and approximate sample points are presented on **Map 4-1**. This MRS is a 42-acre parcel that was overlapped by the buffer area of one historical range fan. The MRS is currently a parade field associated with the NCO Academy located in the northern most part of the installation. It appears that this MRS is located in a down range buffer area, and is not located at a firing point or an impact area. It is assumed that Anti-Aircraft Range - 1 was used continuously from 1957 to 1964. Archival documents from 1941 documenting munitions and weapons allocations confirmed that 37-mm, 40-mm, and 90-mm (M1) anti-aircraft guns were used on FTSW. Based on the range type, period of usage, and the 1941 documents, it is assumed that these munitions were used on Anti-Aircraft Range – 1. No EOD responses have been reported for this MRS. Appendix E of this Work Plan includes the Conceptual Site Model (CSM) developed for the Anti-Aircraft Range-1.

4.1.2 Proposed MEC/MC Activities

MEC Activities: Based on information presented in the HRR, the potential for MEC at the site exists; therefore, activities associated with MEC presence will be performed, including a magnetometer assisted surface sweep/visual survey during sample activities. A Schonstedt

magnetometer assisted site walk will determine the presence of MEC on the site. Field personnel (escorted by a UXO Technician III) will traverse the transects spaced 40 feet apart in order to complete the magnetometer assisted surface sweep/visual survey through the area. A visual depiction of the transects can bee found on Map 4-1. An MEC/Multiple Anomaly Discovery Sheet (Appendix D) will be completed if MEC or munitions debris are detected with the magnetometer or if potential burial sites are found during the site walk. If MEC are identified, the site will be recommended for an RFI/CMS. If no MEC are identified, then the site may be recommended for NFA, depending upon the results of the MC sampling. The FTSW DPW, AEC, and USACE, Baltimore District will be notified if a MEC item is encountered during fieldwork.

MC Activities: Four composite surface soil samples will be collected at biased locations (near MEC or munitions debris, if identified) when possible or at random locations throughout the site. Based on the historical layout and use of this MRS, berms or burial areas are not anticipated therefore only surface soil samples, at a depth of 0 – 6 inches, will be collected. Soil samples will be analyzed for aluminum, copper, zinc (EPA Method 6010B), lead, antimony (EPA Method 6020), and explosives (EPA Method 8330). Data will be compared to FTSW inorganic/metal background values, EPA Region 9 residential PRGs, Region 4 Ecological Screening Values for Surface Soil, for metals and explosives. If MC results in all of the samples fall below the applicable screening standards, the site may be recommended for NFA, depending upon the results of the MEC investigation. If MC results for any of the samples exceed the applicable screening standards, the site may be recommended for an RFI/CMS.

4.2 ANTI – AIRCRAFT RANGE – 90MM - 2

4.2.1 Site Description

The MRS layout, location, and sample point are presented on **Map 4-2**. This MRS is a 77-acre parcel, located northwest of the cantonment area, where two different types of historical munitions uses occurred. These uses included anti-aircraft and tank training and occurred on a total of six separate/collocated ranges from 1941 through 1964. The MRS is positioned in the downrange portion of these ranges and does not overlap impact/target areas or firing points. The known munitions use associated with this MRS includes 40-mm, and 90-mm anti-aircraft

projectiles. The munitions used on the tank range are unknown. However, archival documents from 1941 indicate that 90-mm (HE), 90-mm (practice with tracer), 40-mm (HE), 40-mm (practice with tracer), 37-mm (practice with tracer, HE) were issued to FTSW. Therefore, it is assumed that these munitions could have been used on this MRS. Numerous EOD calls involving C-4 plastic explosives (secondary explosives), M-222 Dragon high explosive anti-tank guided missile, M-7 grenades (riot control agent), and MK-2 fragmentation hand grenades were reported on this site. Appendix E of this Work Plan includes the CSM developed for the Anti-Aircraft Range- 90MM- 2.

4.2.2 Proposed MEC/MC Activities

MEC Activities: Based on information presented in the HRR, the potential for MEC at the site is likely; activities associated with MEC presence will be performed, including a limited magnetometer assisted surface sweep/visual survey during sample activities.. This site is recommended for RFI/CMS due to historical evidence of multiple overlapping range fans (Map2-1) and multiple EOD responses.

MC Activities: One biased composite surface soil sample will be collected at the location of documented EOD response. Based on the historical layout and use of this MRS, berms or burial areas are not anticipated therefore only surface soil samples, at a depth of 0 – 6 inches, will be collected. The soil sample will be analyzed for aluminum, copper, zinc (EPA Method 6010B), lead, antimony (EPA Method 6020), and explosives (EPA Method 8330). Data will be compared to FTSW inorganic/metal background values, EPA Region 9 residential PRGs, Region 4 Ecological Screening Values for Surface Soil, for metals and explosives. This site is recommended for RFI/CMS based on historical evidence of multiple overlapping range fans (Map 2-1) and multiple EOD responses.

4.3 ANTI – TANK RANGE 90MM

4.3.1 Site Description

The MRS layout and location is presented on **Map 4-3**. This MRS is a 124-acre parcel, which had three overlapping historic munitions uses and is currently an active landfill west of the

cantonment area. The MRS is located near the firing points of both a former 90-mm anti-tank range and the former 40-mm anti-aircraft range. The MRS is also positioned within the downrange buffer area of the small arms range. The period of usage of the 90-mm anti-tank range and the 40-mm anti-aircraft range could have been from 1941 through 1947. The history of FTSW implies that this type of training likely ceased in 1944. Based on the research conducted, the small arms ranges were in operation from 1941 through 1971. However, this small arms use only overlapped this MRS in 1941. The known munitions use associated with this MRS includes 40-mm anti-aircraft projectiles and 90-mm anti-tank projectiles. According to documents reviewed for this HRR, munitions used on the small arms range were .50-caliber (cal) or less; however, the exact caliber is unknown. No EOD responses have been reported for this MRS. Appendix E of this Work Plan includes the CSM developed for the Anti-Tank Range 90MM.

4.3.2 Proposed MEC/MC Activities

MEC Activities/MC Activities: No MEC or MC field activities are planned for former Anti - Tank Range 90mm because of the MRS' current and future anticipated use as a RCRA permitted landfill. It is recommended that the historical use of this area be documented in the Installation Master Plan.

4.4 HAND GRENADE COURSE

4.4.1 Site Description

The MRS layout, location, and approximate sample point are presented on **Map 4-4**. This MRS is a 67-acre undeveloped parcel, and is located, in an isolated area of the installation, northwest of the cantonment area. Four different types of historical munitions uses occurred from 1941 through 1994 on five different overlapping ranges. These uses included 40-mm anti-aircraft, 90-mm anti-tank, hand grenade, and small arms training. The MRS is located near the firing point of the small arms range and in the downrange portions of a 40-mm anti-aircraft and a 90-mm anti-tank range. The MRS is almost completely overlapped by the footprint of the hand grenade course. The known munitions use associated with this MRS includes 40-mm anti-aircraft projectiles, 90-mm anti-tank projectiles, small arms, and hand grenades. The exact caliber of

small arms use is unknown. Appendix E of this Work Plan includes the CSM developed for the Hand Grenade Course.

4.4.2 Proposed MEC/MC Activities

MEC Activities: Based on information presented in the HRR, the potential for MEC at the site is likely; activities associated with MEC presence will be performed, including a limited magnetometer assisted surface sweep/visual survey during sample activities. This site is recommended for RFI/CMS due to historical evidence of multiple overlapping range fans (Map 2-1) and its historical use as a hand grenade range.

MC Activities: One random composite surface soil sample will be collected on this MRS. Based on the historical layout and use of this MRS, berms or burial areas are not anticipated therefore only surface soil samples, at a dept of 0 – 6 inches, will be collected. The soil sample will be analyzed for metals using EPA Method 6010B/6020, and for explosives using EPA Method 8330. Data will be compared to FTSW inorganic/metal background values, EPA Region 9 residential PRGs, Region 4 Ecological Screening Values for Surface Soil, for metals and explosives. This site is recommended for RFI/CMS based on historical evidence of multiple overlapping range fans (Map2-1) and its historical use as a hand grenade range.

4.5 SMALL ARMS RANGE -1

4.5.1 Site Description

The MRS layout and location are presented on **Map 4-5**. This MRS is a 136-acre parcel located at Evans Heliport/Airfield, northeast of the cantonment area that was overlapped by two historical small arms ranges. These ranges were operational in 1962 and 1964. According to documents reviewed for this HRR, munitions used on the small arms range were .50-cal or less; however, the exact caliber is unknown. No EOD responses have been reported for this MRS. Appendix E of this Work Plan includes the CSM developed for the Small Arms Range- 1.

4.5.2 Proposed MEC/MC Activities

MEC Activities: No MEC field activities are recommended for this MRS because historical evidence suggests only small arms were used at this MRS.

MC Activities: An all-metals detector assisted visual survey will be conducted in order to locate remnants of small arms roundss. The all-metals detector assisted visual survey will be completed by traversing transects spaced 40 feet apart. A visual depiction of the visual survey transects can be found on Map 4-5. Four composite surface soil samples will be collected at biased locations (near remnants of small arms, if identified) when possible or at random locations on undeveloped portions of the MRS. Based on the historical layout and use of this MRS, berms or burial areas are not anticipated therefore only surface soil samples, at a depth of 0 – 6 inches, will be collected. Soil samples will be analyzed for copper using EPA Method 6010B and antimony, and lead using EPA Method 6020. Data will be screened using the FTSW background value, then the EPA region 9 residential PRG for copper, antimony, and lead and the Region 4 Ecological Screening Value for copper, antimony, and lead in surface soil. If MC results in all of the samples fall below the applicable screening standards, the MRS may be recommended for NFA. If MC results for any of the samples exceed the applicable screening standards, the MRS may be recommended for an RFI/CMS.

4.6 SMALL ARMS RANGE – 2

4.6.1 Site Description

This MRS was identified during the Phase 3 Range Inventory. As part of the HRR a thorough review of the documents used to generate the Phase 3 Range Inventory was conducted. As a result of this review it was determined that the historical small arms range fans that made up this MRS did overlap the cantonment area (non operational area) and as such this MRS is not eligible for the MMRP. It was therefore agreed upon during the TPP meeting that no further action is required for this MRS under the active installation MMRP, and no CSM was developed for this site.

4.6.2 Proposed MEC/MC Activities

As mentioned above no further action is required at this MRS, there fore no MEC/MC activities will be performed.

4.7 SMALL ARMS RANGE - 3

4.7.1 Site Description

The MRS layout and location are presented on **Map 4-6**. This MRS is a 32-acre parcel, in the area northeast of the cantonment areas within one mile of the Holbrook Pond Recreational Area. The overlapping historical munitions use is an operational small arms range used in 1964. According to documents reviewed for this HRR, munitions used on the small arms range are believed to be .50-cal or less; however, the exact caliber is unknown. No EOD responses have been reported for this MRS. Appendix E of this Work Plan includes the CSM developed for the Small Arms Range- 3.

4.7.2 Proposed MEC/MC Activities

MEC Activities: No MEC field activities are recommended for this MRS because historical evidence suggests only small arms were used at this MRS.

MC Activities: An all-metals detector assisted visual survey will be conducted in order to locate remnants of small arms rounds. The all-metals detector assisted visual survey will be completed by traversing transects spaced 40 feet apart. A visual depiction of the visual survey transects can be found on Map 4-6. Three composite surface soil samples will be collected at biased locations (near remnants of small arms, if identified) when possible or at random locations throughout the site. Two sediment, and two surface water samples will also be collected at this MRS. Based on the historical layout and use of this MRS, berms or burial areas are not anticipated therefore only surface soil samples, at a depth of 0 – 6 inches, will be collected. One soil sample will be collected in the northern portion and two samples will be collected in the southern portions of this MRS. The sediment samples: will be collected from each of the man-made damns of the pond. The surface water samples will be collected in the near the sediment sample locations. All samples will be analyzed for copper using EPA Method 6010B and antimony, and lead using

EPA Method 6020. Data will be compared to the FTSW background value and then the EPA region 9 residential PRG, Region 4 Ecological Screening Values for surface soil, EPA Water Quality Standards for Freshwater CCC chronic, and Region 4 Ecological Screening Values for surface water for copper, antimony, and lead as appropriate. If MC results in all of the samples fall below the applicable screening standards, the MRS may be recommended for NFA. If MC results for any of the samples exceed the applicable screening standards, the MRS may be recommended for an RFI/CMS.

4.8 HERO ROAD TRENCH AREA

4.8.1 Site Description

The MRS layout, location, and approximate sample point are presented on **Map 4-7**. The Hero Road Trench Area is a 10-acre parcel located within the cantonment area that was identified in January 2003 when a former FTSW DPW staff member reported to the DPW Environmental Office that materials (i.e., mustard gas) had been buried in the DPW Family Housing Maintenance parking lot located on Hero Road. The aerial photographs indicate disturbances from January 1941 to January 1957 that were indicative of possible burial activities. Items were allegedly buried at the MRS but not used on this MRS. CWM items allegedly associated with this MRS include Gas Identification Set, Detonation, M1, containing: 5% solution of mustard, 5% solution of Lewisite, 50% solution of chloropicrin, and Pure agent phosgene. No EOD responses have been reported for this MRS. This MRS is partially fenced. The red cross-hatched area found on Map 4-7 represents the unfenced portion were the field activities will take place. Appendix E of this Work Plan includes the CSM developed for the Hero Road Trench Area.

4.8.2 Proposed MEC/MC Activities

MEC Activities: MEC field activities planned for this MRS include conducting a visual survey of unfenced portions of MRS to ensure no MEC or MEC debris remains on the surface during sampling activities. This MRS is recommended for RFI/CMS based on historical evidence and results of the current investigation.

MC Activities: One composite surface soil sample will be collected from biased location when possible or at a random location throughout the MRS. Surface soil samples, at a depth of 0 – 6 inches, will be collected at this MRS. The soil sample will be analyzed for aluminum, copper, zinc (EPA Method 6010B), lead, antimony (EPA Method 6020), and explosives (EPA Method 8330). Data will be compared to FTSW inorganic/metal background values, EPA Region 9 residential PRGs, Region 4 Ecological Screening Values for Surface Soil, for metals and explosives. This MRS is recommended for RFI/CMS based on historical evidence and results of the current investigation

4.9 SUMMARY OF FIELD ACTIVITIES

The total number of field samples that will be collected and the selected laboratory analyses are presented in **Table 4-1** below.

Table 4-1: Field Sample Summary Table

MRS	Number of Field Samples/Analysis																			
	Metals[1] (6010B,6020)				Metals[2] (6010B, 6020)												Explosives (8330)			
	Surface Soil				Surface Soil				Sediment				Surface Water				Surface Soil			
Sample Type	FS	DFS	MS	MSD	FS	DFS	MS	MSD	FS	DFS	MS	MSD	FS	DFS	MS	MSD	FS	DFS	MS	MSD
Anti – Aircraft Range - 1	4	1	1	1	0	0	0	0	0	0	0	0	0	0	0	0	4	1	1	1
Anti – Aircraft Range 90mm - 2	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0
Anti – Tank Range 90mm	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Hand Grenade Course	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0
Small Arms Range - 1	0	0	0	0	4	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Small Arms Range - 3	0	0	0	0	3	1	1	1	2	1	1	1	2	1	1	1	0	0	0	0
Hero Road Trench Area	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0
Total Analysis by Media	10				10				5				5				10			

1 Metals analysis includes: aluminum, copper, zinc by EPA Method 6010B; and lead, antimony by EPA Method 6020.
2 Metals analysis includes: copper by EPA Method 6010B; and lead, antimony by EPA Method 6020.

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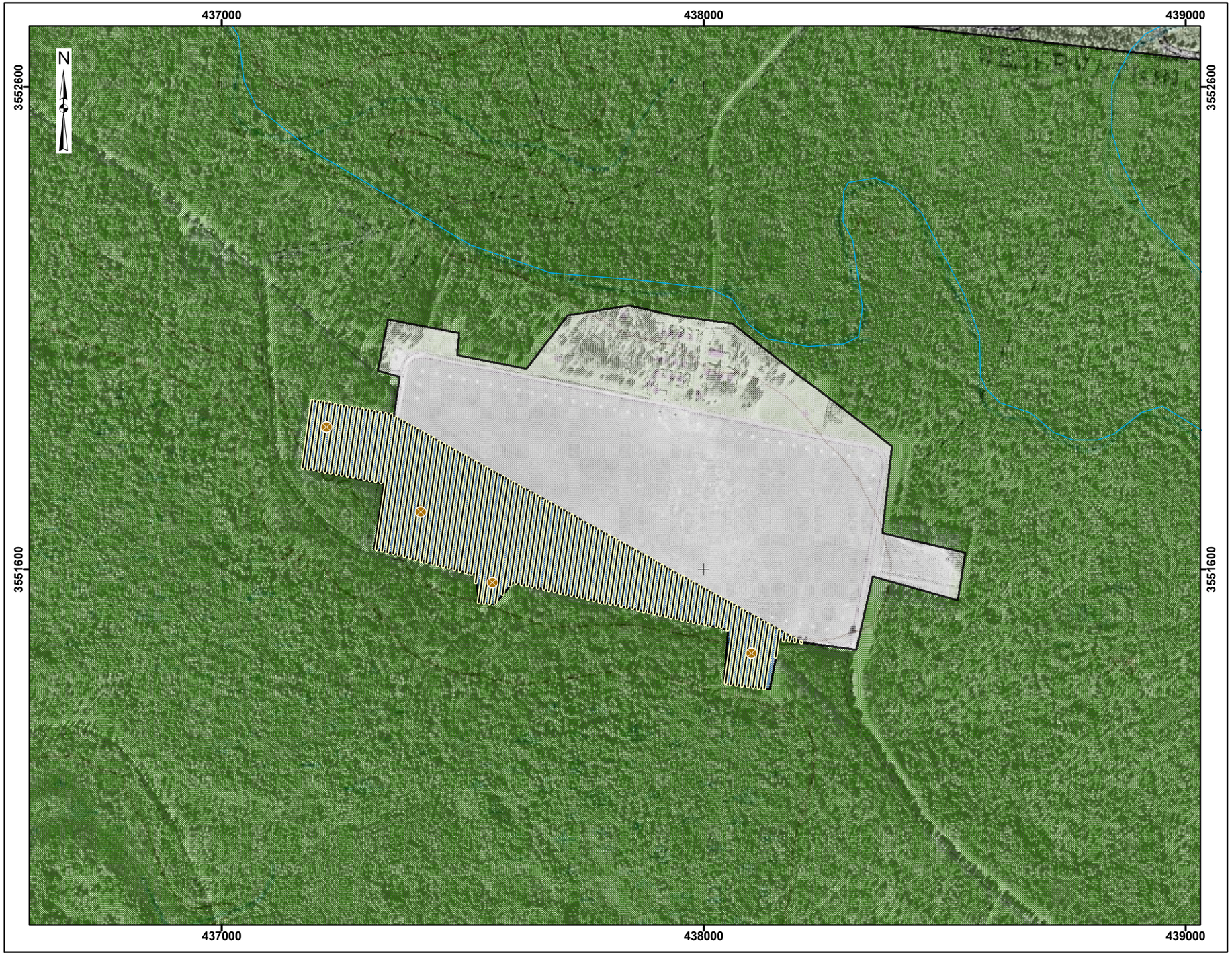

Map 4-1
Anti - Aircraft Range - 1

Legend

- Installation Boundary
- Streams/Rivers

Military Range Area

- Closed
- Operational Range Area
- Non Range, Non UXO-DMM-MC Area
- Soil Sampling Location*
- Proposed Path for Site Survey Area

*Note: All sample locations shown are approximate

0 125 250 375 500 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

Contract: DACA31-00-D-0043
Edition: Final Work Plan
Date: March 2007

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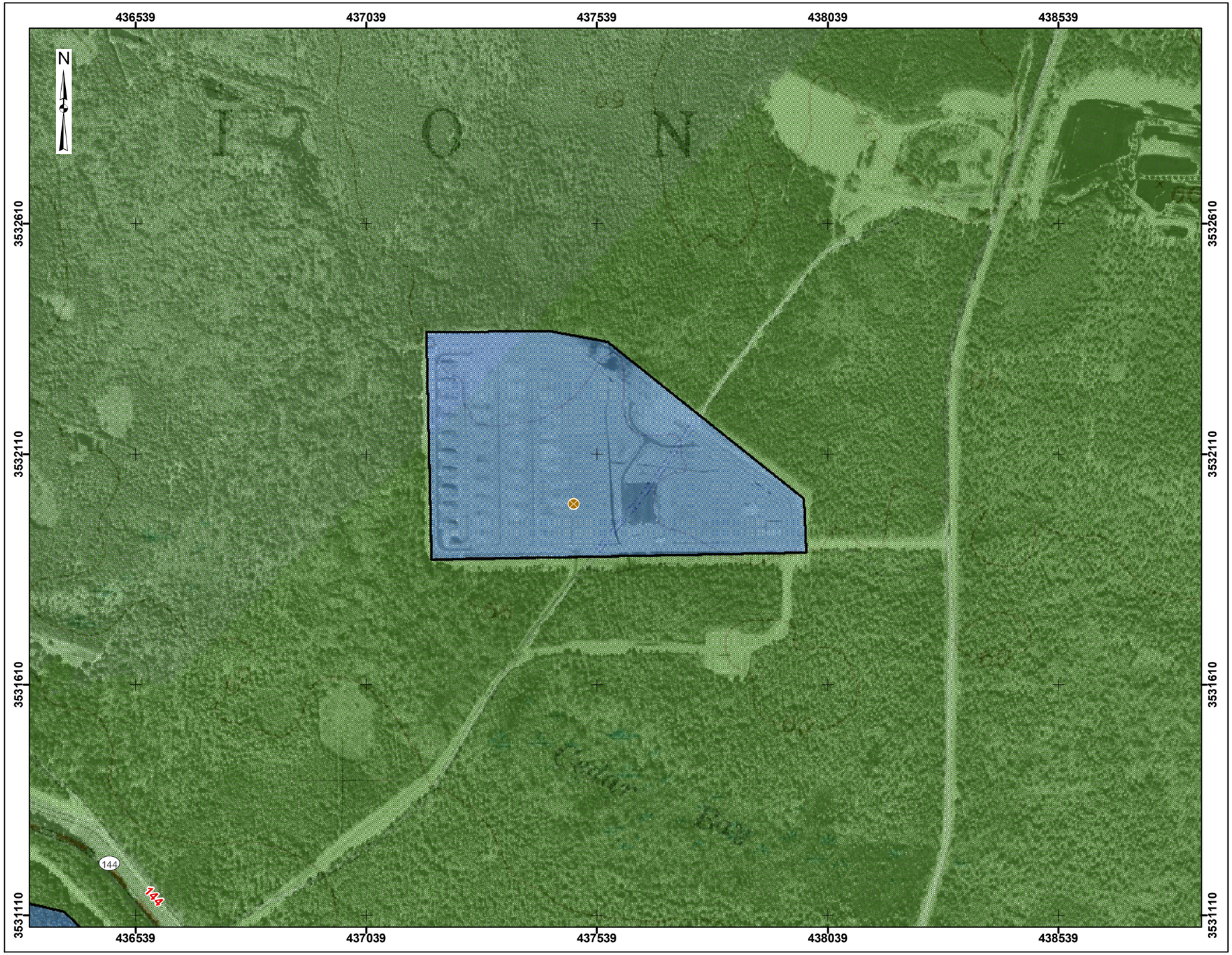

Map 4-2
Anti-Aircraft Range 90-mm - 2

Legend

- Installation Boundary

Military Range Area

- Closed
- Operational Range Area
- Non Range, Non UXO- DMM-MC Area
- Soil Sampling Location*

*Note: All sample locations shown are approximate

0 125 250 375 500 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

Contract: DACA31-00-D-0043
Edition: Final Work Plan
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Map 4-3
Anti Tank Range 90-mm

Legend

- Installation Boundary
- Streams/Rivers
- Lake or Pond

Military Range Area

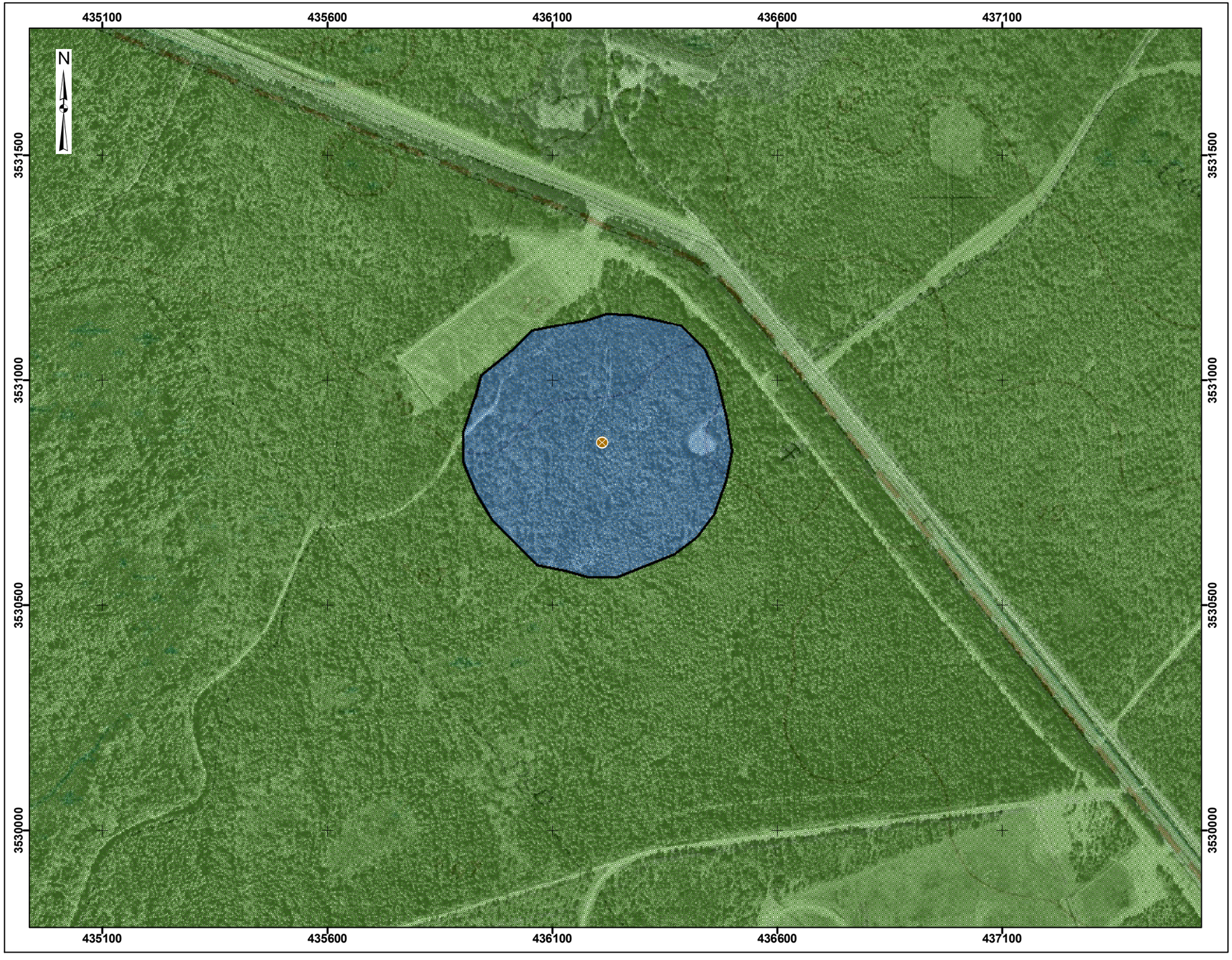
- Closed
- Operational Range Area
- Non Range, Non UXO-DMM-MC Area

0 100 200 300 400 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

Contract: DACA31-00-D-0043
Edition: Final Work Plan
Date: March 2007



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MALCOLM PIRNIE

Map 4-4
Hand Grenade Course

Legend

- Installation Boundary

Military Range Area

- Closed
- Operational Range Area
- Non Range, Non UXO-DMM-MC Area
- Soil Sampling Location*

*Note: All sample locations shown are approximate

0 125 250 375 500 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

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Map 4-5
Small Arms Range - 1

Legend

- Installation Boundary
- Lake or Pond

Military Range Area

- Closed
- Operational Range Area
- Non Range, Non UXO-DMM-MC Area
- Proposed Path for Site Survey Area

0 150 300 450 600 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

Contract: DACA31-00-D-0043
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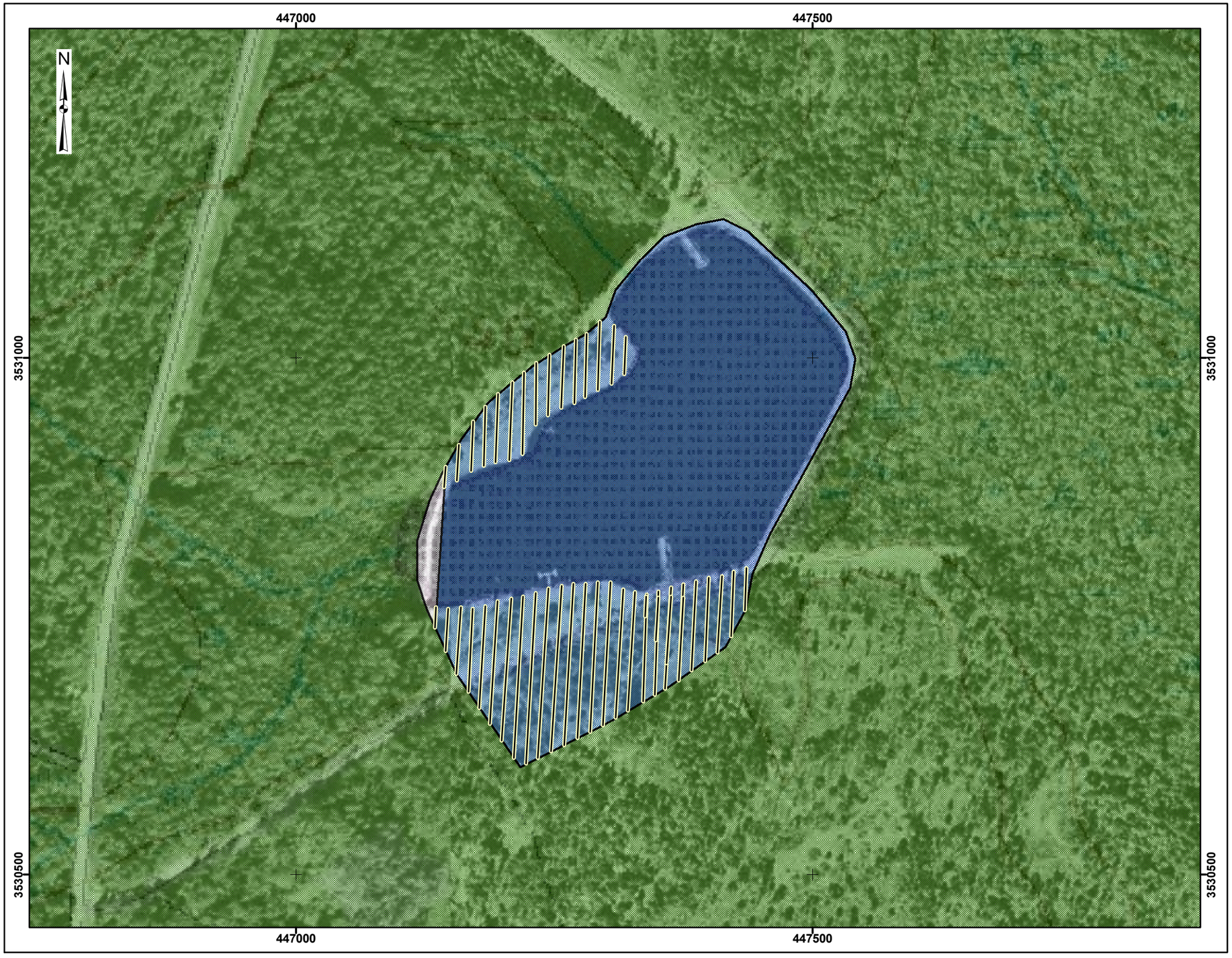
MALCOLM PIRNIE

Map 4-6
Small Arms Range - 3



Legend

- Installation Boundary
- Streams/Rivers
- Lake or Pond

Military Range Area

- Closed
- Operational Range Area
- Non Range, UXO-DMM-MC Area
- Proposed Path for Site Survey Area

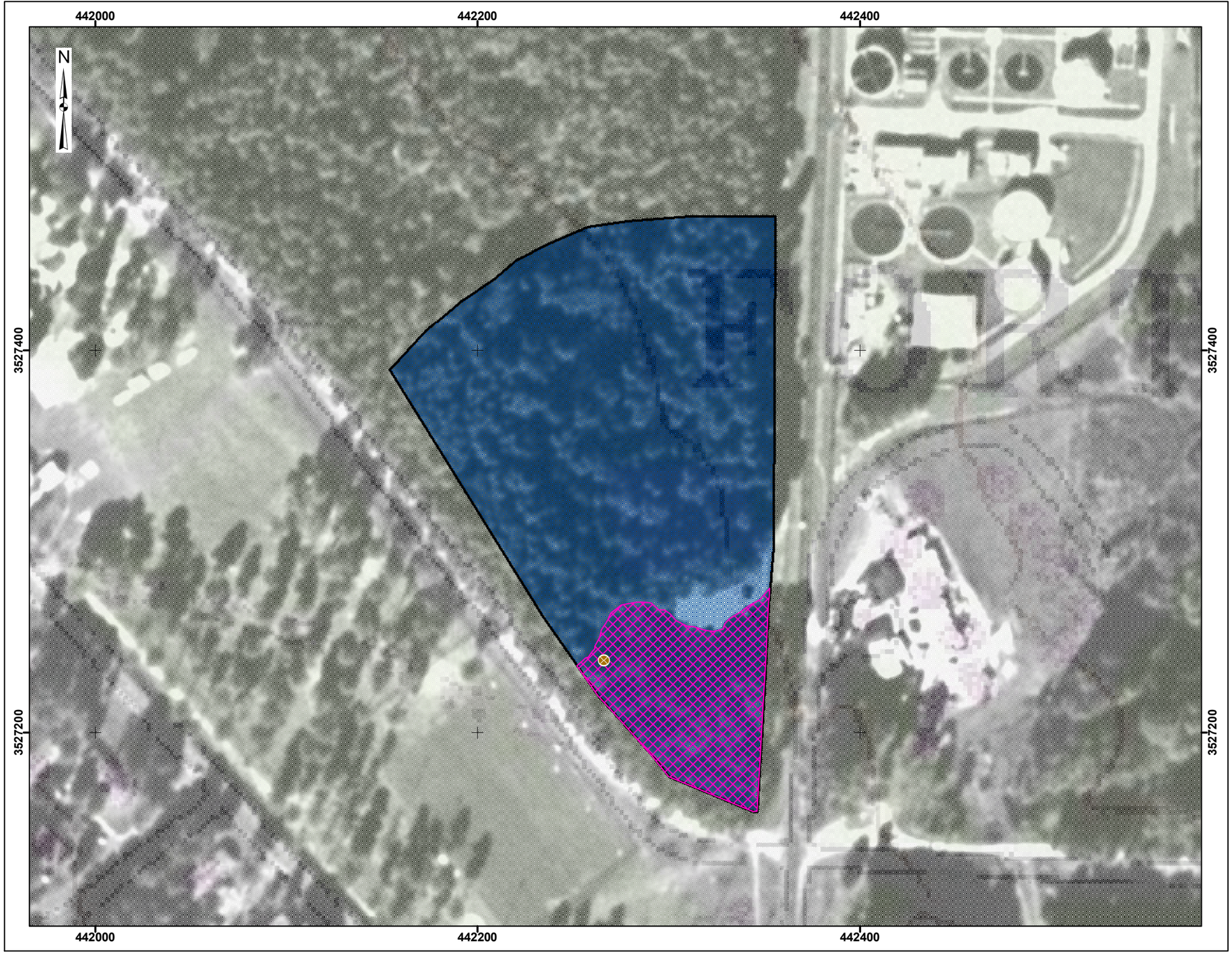
*Note: All sample locations shown are approximate

0 50 100 150 200 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

Contract: DACA31-00-D-0043
Edition: Final Work Plan
Date: March 2007



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MALCOLM PIRNIE

Map 4-7
Hero Road Trench Area

Legend

- Installation Boundary

Military Range Area

- Closed
- Operational Range Area
- Non Range, Non UXO-DMM-MC Area
- Approximate Surface Walk Area
- Soil Sampling Location*

*Note: All sample locations shown are approximate

0 25 50 75 100 Meters

Data Source: CTT Inventory Data
Microsoft TerraServer
USGS Digital Raster Graphics

Coordinate System: UTM Zone 17N
Datum: WGS 84
Units: Meters

Contract: DACA31-00-D-0043
Edition: Final Work Plan
Date: March 2007

5 SITE-SPECIFIC QAPP

This section is intended to supplement the overall MMRP CS QAPP (Appendix A). The QAPP provides general information and standard operating procedures applicable to sampling and analytical activities to be performed at all installations where MMRP CSs are being conducted by Malcolm Pirnie. The information includes definitions and generic goals for data quality and minimum requirements for QA/QC samples. The procedures address sampling and decontamination protocols; geophysical investigation; field documentation; sample handling, custody, and shipping; instrument calibration and maintenance; field and laboratory auditing; data reduction, validation, and reporting; corrective action requirements; and QA reporting. It should be noted that the QAPP may include discussions on procedures or methods that are not applicable to a specific MRS since it is intended to encompass all sites.

Per the contract, it is intended that once the QAPP is finalized, it will not be modified (except for programmatic changes) and will serve as a programmatic document. Site-specific sampling information and any exceptions or proposed changes to the QAPP are addressed and included in this SS-QAPP. This SS-QAPP is not a stand-alone document from the QAPP. The QAPP will provide the majority of the QA/QC information; the SS-QAPP simply supplements the information in the QAPP by providing for MRS-specific condition requirements.

The data collected at FTSW will be compared to applicable regulatory standards (Table 5-1).

Table 5-1: Applicable Regulatory Standards and Comparison Values by Sampling Media

Sample Media	Applicable Standard and/or Comparison Values
Soil	FTSW inorganic/metal background concentrations for surface soil
Soil	EPA Region 9 PRG for residential soils
Soil	Region 4 Ecological Screening Values for surface soil
Surface Water	EPA National Recommended Water Quality Criteria for Human Health Consumption of Water and Organism
Surface Water	Region 4 Ecological Screening Values for surface water

Table 5-2 presents the contaminants of concern for soil with the applicable standards compared to the laboratory Method Detection Limits (MDLs) and Reporting Limits (RLs). Available background concentrations for metals are also provided in **Table 5-2**.

Table 5-2: Solid Laboratory Limits and Applicable Standards

Contaminant of Concern	MDLs (mg/kg)[1]	Laboratory RLs (mg/kg)	Region 9 PRGs - Residential (mg/kg)	Region 4 Ecological Screening Values Surface Soil (mg/l)	FTSW Inorganic/Metal Background Concentrations[2] (mg/kg)
Explosives					
1,3,5-TNB (NC)	0.05	0.25	1,800	-	NA[3]
1,3-DNB (NC)	0.05	0.25	6.1	-	NA
2,4,6-TNT (CA)	0.03	0.25	16	-	NA
2,4-DNT (NC)	0.04	0.25	120	20	NA
2,6-DNT (NC)	0.05	0.25	61	-	NA
2-AM-4,6-DNT	0.1	0.5	-	-	NA
2-NT (NC)	0.03	0.25	180	-	NA
3-NT (NC)	0.02	0.25	180	-	NA
4-AM-2,6-DNT	0.1	0.5	-	-	NA
4-NT (CA)	0.03	0.25	12	-	NA
HMX (NC)	0.04	0.25	3,100	-	NA
NB (NC)	0.1	0.5	20	40	NA
RDX (CA)	0.1	0.5	4.4	-	NA
TETRYL (NC)	0.2	1	16	-	NA
Metals					
Aluminum (NC)	2	10	76,000	-	-
Antimony (NC)	0.6	3.0	31	-	-
Copper (NC)	0.3	1.5	3,100	9	-
Lead (NC)	0.3	1.5	400	2.5	11.1
Zinc (NC)	0.7	3.5	23,000	120	15.5

1 mg/kg = milligram per kilogram
2 Phase II RCRA Facility Investigation Report For 16 Solid Waste Management Units At Fort Stewart, Georgia. April 2000
3 NA = Not applicable
- indicates no available value available
(CA)- Cancer
(NC) – Non-cancer

Table 5-3: Aqueous Laboratory Limits and Applicable Standards

Contaminant of Concern	MDLs (µg/l)[1]	Laboratory RLs (µg/l)	Human Health Consumption of Water and Organism (µg/l)	Region 9 PRGs Tap Water (µg/l)	Region 4 Ecological Screening Values Surface Water (µg/l)
Antimony	0.3	2.0	5.6	15	160
Copper	0.3	2.0	1,300	15	6.54
Lead	0.3	2.0	-	-	1.32

- indicates no available value available

Weight of Evidence Based Decisions

As presented in Table 5-2 above the contaminants of concern have been designated by the EPA as either non-cancer or cancer contaminants. The USEPA Region 9 PRGs for non-cancer contaminants will be divided by 10 to meet GAEPD's recommended risk level / hazard quotient of 0.1. The adjusted criteria will be used as comparison values, which will be used along with other forms of evidence to make the determination whether further investigation is required or if a NFA determination is appropriate for each MRS . Examples of evidence that will be included in the decision making process include historical information, analytical results (screened against established background levels, and agreed upon regulatory limits), and field observations.

As noted in Table 5-3, Human Health Consumption of Water and Organism, and Region 9 PRGs Tap Water for lead do not exist for lead in surface water. As such the weight of evidence approach for human consumption of surface water will include an evaluation of the lead concentrations in the sediment and surface soils at this MRS as agreed upon after the TPP meeting.

Data Quality Control Criteria

Section 8.0 of the Final Quality Assurance Project Plan (QAPP) Military Munitions Response Program Site Inspections defines the data quality control criteria for the project including Precision, Accuracy, Representativeness, Sensitivity, Comparability and Completeness. The specific project criteria for precision, accuracy and sensitivity are presented in the attached.

Table 5-4 through **Table 5-7** which lists the laboratory quality control limits which the laboratory will employ for this project for EPA methods 8330 for explosives, EPA method 6010B will be used for the analysis of all metals with the exception of antimony and lead, in which case EPA Method 6020 will be used. EPA Method 6020 will be used in lieu of 6010B to achieve the reporting limits consistent with the screening criteria agreed upon at the TPP session. The precision acceptance criteria for field duplicate samples collected are part of this project with a relative percent difference (RPD) of at least $\leq$35% evaluated for sample results at least 5 times the detection limit.

Representativeness will be satisfied by determining that the field sample plan is followed, proper sampling techniques, preservation, and handling are used, proper analytical procedures are followed, and holding times are not exceeded in the laboratory.

The procedures used to obtain the planned analytical data are expected to provide comparable data. The procedures employed will be based upon EPA-promulgated methodologies which are commonly used for environmental investigations.

It is expected that the laboratories used for this project will provide data that meet the completeness QC acceptance criteria of 90% or more of all the samples analyzed. The completeness goal for samples collected in the field will be 95% of the quantity of samples planned in the field sample plan. Corrective action may be implemented to re-collect samples where necessary and possible. The percent completeness will be calculated per the definition and equation given in the QAPP.

Table 5-4: EPA Method 6010B QC Limits

Parameter	Frequency	Control Limits	Corrective Action
Calibration Blank	Beginning of run, after every 10 samples, and at the end of the run.	< 2 x MDL	Reanalyze the blank, prepare new blank and analyze, perform maintenance on instrument, recalibrate, reanalyze any samples since the last acceptable blank. If reanalysis is not possible, report with a qualifying comment.
Method Blank (LRB)	One per batch of no more than 20 samples. Analyze with associated sample batch.	<1/2 the RL	Reanalyze the blank. Samples in the batch must be < the reporting limit or ≥ 10x the method blank. If not, samples must be re-digested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.
High Calibration Standard	After calibration and before analysis of samples.	90-110%	Reanalyze the High Standard. If the standard is still not acceptable, re-profile and/or perform instrument maintenance, and prepare a new calibration.
Laboratory Fortified Blank (LFB or LCS)	One per batch of no more than 20 samples. Analyze with associated sample batch.	80-120%	Reanalyze the LFB. If still outside of acceptable range, samples must be re-digested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.

Parameter	Frequency	Control Limits	Corrective Action
Quality Control Sample (QCS)	Immediately after calibration.	90-110%	Reanalyze the QCS. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration.
Instrument Performance Check Solution (IPC) Same Source	Beginning of run, after every 10 samples, and at the end of the run.	90-110%	Reanalyze the IPC. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration. Reanalyze any samples since the last acceptable IPC. If reanalysis is not possible, report with a qualifying comment.
Reporting Limit Standard (RPL)	Beginning of run, after calibration.	80-120%	Reanalyze the RPL. If the standard is still not acceptable, recalibrate.
Matrix Spike (MS)	One every 10 samples with at least one per batch.	80-120%	If calibration verification standards are acceptable, reanalyze spike once. If the spike still fails perform a post-spike. Post spikes must be recovered at 85-115%. If not or if reanalysis is not possible, report the results with a qualifying comment.

Table 5-5: EPA Method 8330A QC Limits

Parameter	Frequency	Control Limits	Corrective Action
Initial Calibration: Minimum of five points for linear calibration models and six points for quadratic calibration models.	Calibration must be in place prior to sample analysis. Recalibration when qualitative and/or qualitative fail to meet method, laboratory, or project criteria.	Linear or quadratic regression: $r^2 \geq 0.99$ Averaged Calibration Factor: Relative Standard Deviation (RSD) ≤ 20%	Identify and correct problem. Re-prepare and re-analyze initial calibration standards.
Second source initial calibration verification (ICV).	Once after initial calibration.	± 15% of the expected value.	1. Prepare and analyze a fresh Second Source ICV. 2. Identify and correct the source of the problem. This may require instrument re-calibration.
Continuing calibration verification (CCV)	Daily before sample analysis, after every ten field samples, and at the end of the analytical sequence.	± 15% of the expected value.	1. Re-inject CCV 2. Re-inject all samples analyzed prior to failing CCV if results are impacted. (i.e.: If response for an analyte is high and the analyte was not detected in the samples, data are not impacted and re-analysis is not necessary)

Parameter	Frequency	Control Limits	Corrective Action
Method blank (MB)	One per preparatory batch of 20 or fewer field samples	No analytes detected > ½ RL. If contamination in the method blank is greater than ½ RL and is greater than 1/10 of the measured amount in the sample, corrective action will be taken.	1. Identify and correct the source of contamination. 2. If additional sample remains, re-extract and re-analyze. 3. If no additional sample remains, flag results for the specific analyte in all samples associated with method blank. Note: If analyte detected in method blank was not present in the samples, data were not affected. Sample re-analysis or data flagging is not necessary.
LCS	One LCS per extraction batch of 20 or fewer samples	See the following Table: LCS and MS Control Limits for EPA Method 8330 Solid Matrix	1. Check Calculations and spike solution fortification. Re-evaluate data if this indicates a problem. 2. Identify and correct problem. Re-extract and re-analyze LCS and associated field samples. If additional sample or holding time limitations prevent re-extraction and re-analysis, flag results for the specific analyte(s) in all samples in the preparatory batch.
Matrix spike (MS)	One MS per batch of twenty or fewer samples.	See the following Table: LCS and MS Control Limits for 8330 Solid Matrix	1. Check calculation
Matrix spike duplicate (MSD) or sample duplicate	One MSD or sample duplicate per twenty or fewer samples	30% RPD	
Surrogate spike	All Field and QC samples	65% to 135%	For QC samples and field samples, identify and correct problem then re-prep and reanalyze all samples with failing surrogate recoveries in preparatory batch. If chromatographic interferences or matrix affects are obvious for field samples, re-extraction may not be necessary.
Confirmation of positive results	All positive detections will be confirmed.	Calibration and quality control criteria from primary column apply to confirmation column. Precision between primary and confirmation column ≤ 40%RPD	Primary column used to report all quantitative results unless matrix interferences warrant reporting from confirmation column. Flag analyte if RPD is > 40%
Field Duplicates	Per batch of 20 Samples	≤35% Evaluated for samples 5 times the detection limits	If the limits are exceeding for field replicates this will be addressed by the data validator

Note: The laboratory QC criteria are based on Analytical Laboratory Services (ALSI) Standard Operating Procedures (SOPs): 09-8330S, Revision 1, Ultrasonication of Solids for the Analysis of Explosives by EPA Method 833A High Performance Liquid Chromatography (HPLC) and 1B-833, Revision 3, Nitroaromatics and Nitramines by HPLC with Ultraviolet Detection

Table 5-6: Laboratory Control Sample (LCS) and Matrix Spike (MS) Control Limits for 8330 Solid Matrix

Compound	Lower Control Limit (%)	Upper Control Limit (%)
HMX	75	125
RDX	70	135
1,3,5-TNB	75	125
1,3-DNB	80	125
Tetryl[2]	10	150
2,4,6-TNT	50	145
NB	50	140
4-Am-4,6-DNT	55	155
2-Am-2,6-DNT	50	155
2,4-DNT	60	135
2,6-DNT	60	135
2-NT	45	135
3-NT	50	130
4-NT	50	130

Notes:

1. The control limits listed are based upon those given in Table D-13 from the Department of Defense Quality Systems Manual for Environmental Laboratories, Version 3, and January 2006.

The control limits for Tetryl are based upon those given in Table D-2 from the Department of Defense Quality Systems Manual for Environmental Laboratories, Version 3, and January 2006.

Table 5-7: Inorganic Analysis by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Methods 6020

Parameter	Frequency	Acceptance Criteria	Corrective Action
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see DOD Quality Systems Manual , Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see DOD Quality Systems Manual, section C.1.f).
MDL	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see DOD Quality Systems Manual box D-18).	See 40 Code of Federal Regulations (CFR) 136B. MDL verification checks must produce a response at least 3 times the instrument noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see DOD Quality Systems Manual box D-18).
Instrument detection limit (IDL) study	At initial set-up and after significant change	Detection limits established shall be $\leq$ MDL.	NA
Tuning	Prior to initial calibration	Mass calibration $\leq$ 0.1 amu from true value; Resolution <0.9 amu full width at 10% peak height; For stability, RSD $\geq$ 5% for at least four replicate analytes	Retune instrument then reanalyze tuning solutions.

Parameter	Frequency	Acceptance Criteria	Corrective Action
Initial calibration for all analytes (ICAL) (minimum one high standard and a blank)	Initial calibration prior to sample analysis	If more than one standard is used, in which case $r \geq 0.995$.	Correct problem and repeat initial calibration.
Second source calibration verification	Once after each initial calibration, prior to sample analysis	Value of second source for all analytes within ± 10% of expected value (initial source)	Correct problem and verify second source standard. If that fails, then repeat initial calibration
Low level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±20% of expected value	Correct problem, then reanalyze.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ± 10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration.
Liner dynamic range or high-level check standard	Every 6 months	Within ± 10% of expected value	NA
Method blank	One per preparatory batch	No analytes detected ≥ ½ RL For common laboratory contaminants, no analytes detected RL	Correct problem, then see criteria in DoD Quality Systems Manual, box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples
Interference check solutions (ICS-A and ICSAB)	At the beginning of an analytical run	ICS-A: Absolute value of conc. For all non-spiked analytes > 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all affected samples.
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see DoD Quality Systems Manual, box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes. If sufficient sample material is available. (See full explanation in Appendix DoD-D).

Parameter	Frequency	Acceptance Criteria	Corrective Action
Dilution test	Each preparatory batch	Five-fold dilution must agree within ± 10% of the original determination	Perform post-digestion spike addition.
Post-digestion spike addition	When dilution test fails or analyte concentration in all samples < 100 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.
Method of standard additions (MSA)	When matrix interference is suspected	NA	NA
MS	One MS per every 20 project samples per matrix (see DoD Quality Systems Manual, box D-15).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.
Internal standards (IS)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in EPA Method 6020
Results reported between MDL and RL	NA	NA	NA

6 SAMPLE MANAGEMENT AND ANALYSIS

6.1 FIELD OPERATIONS DOCUMENTATION

Field documentation of the samples taken is of the utmost importance in assuring QC. Field documentation will include DQCRs, field notebooks, sample labels, and COC forms. All field documentation will be completed in indelible ink. Corrections will be made by drawing a single line through the text and legibly writing the correction.

6.2 DQCR

As described in the QAPP, the DQCR will be prepared by the FPM each day that fieldwork is performed, commencing with the first day work is performed on-site. All workdays will be documented in this report throughout the duration of the fieldwork. Malcolm Pirnie will provide DQCRs to the USACE, Baltimore District PM in the CS Report. A sample DQCR form is included as Figure 10-1 in Appendix A of the QAPP.

6.3 FIELD NOTEBOOKS

Field notes regarding all sampling and field activities will be kept in a bound notebook with pre-numbered pages. Indelible ink will be used for all entries. The field notes will be filled out while the fieldwork is taking place and will include all of the information that is reported on the DQCR forms.

6.4 SAMPLE NUMBERING SCHEME

All samples taken will employ the USACE Laboratory numbering system. This system assures that QC checks originating from the field are blind to the laboratory and that a uniform and consistent numbering system is employed in the field.

All samples collected as part of this CS Report will utilize the following standard designation format:
FTSW- [Sample media] - [Location designation] - [sample date (month) (day) (year)]

SS will be used to designate a surface soil sample (e.g., FTSW-SS-22-080104).

All duplicate samples collected will utilize the following standard designation format:
FTSW - [Sample media] - [Location designation/DUP] - [sample date (month)(day)(year)]
(e.g., FTSW-S-22/DUP-080104)

All MS/MSD samples collected will utilize the following standard designation format:
FTSW - [Sample media] - [Location designation/MSD] - [sample date (month)(day)(year)]
(e.g., FTSW-SS-22/MSD-080104)

All equipment blank samples collected will utilize the following standard labeling format:
FTSW - [Sample media] - [Location designation/EB] - [sample date (month)(day)(year)]
(e.g., FTSW-SS-22/EB-080104)

6.5 SAMPLE LABELS

Correct sample labeling and the corresponding notation of the sample identification numbers in the field notebook, DQCR, and on the COC forms will be utilized to prevent misidentification of samples and their eventual results. All sample labels will be completed legibly with indelible ink. The labels will be affixed to the sample bottle and covered with clear tape.

At a minimum, the sample labels will include the following:

a. Project name
b. Company name
c. Name/initials of the collector
d. Date and time of collection
e. Sample location and depth
f. Analysis required
g. Preservatives added
h. Matrix

6.6 COC

The COC procedures will be in accordance with USACE Sample Handling Protocol and EPA procedures. COC procedures are used to document and track samples from collection through reporting of analytical results and to serve as permanent records of sample handling and shipment. Strict COC protocol will be maintained for all samples collected during this project. The COC forms will be filled out with indelible ink by the FPM, and any mistakes made will be crossed out with a single line and initialed and dated.

The information on the COC form will include the following:

a. Sample identification numbers
b. Date and time of sample collection
c. Project name and number
d. Number of sample containers
e. Analyses required including method number
f. Turn around time required
g. Preservatives used
h. Signatures of all parties who had possession of the samples
i. Matrix

COC forms will be completed for every cooler and will be sealed in a resealable bag and taped to the inside of the lid of the cooler. The FPM will keep one copy of the COC form. The laboratory will then sign the COC form upon accepting the samples for analysis. Copies of the COC forms will be included in the CS Report as an appendix and given to the USACE, Baltimore District PM upon completion of the field sampling effort.

6.7 SAMPLE PACKAGING AND SHIPPING REQUIREMENTS

Custody of samples must be maintained throughout the shipment of samples to the selected laboratory. The following procedures will be used to send samples to be analyzed for explosives and metals to the laboratory:

- Use waterproof high-strength plastic ice chests or coolers only.

- After filling out the pertinent information on the sample label and tag, put the sample in the container and screw on the lid. Secure the bottle lid with strapping tape.
- Tape cooler drain shut.
- Place about 3 inches of inert cushioning material, such as vermiculite or styrofoam "popcorn", in the bottom of the cooler.
- Enclose the containers in clear plastic bags through which sample labels are visible, and seal the bag. Place containers upright in the cooler in such a way that they do not touch and will not touch during shipment.
- Put in additional inert packing material to partially cover sample containers (more than halfway). Place bags of ice or ice-gel packs around, among, and on top of the sample containers.
- Fill the remaining space in the cooler with cushioning material.
- If sending the samples by common carrier, sign the COC form under "Relinquished by," enter the carrier name and air bill number, retain a copy for field records, put the COC record in a waterproof plastic zip top bag and tape it with masking tape to the inside lid of the cooler.
- If sending the samples by courier or field team shipper, follow the above procedures, but also have the receiving carrier sign under "Received by."
- Apply custody seals to the front and back of the cooler, across the lid.
- Secure lid by taping. Wrap the cooler completely with strapping tape at a minimum of two locations. Do not cover any labels.
- Attach completed shipping label to top of the cooler. The shipping label will have a return address.
- Ship the cooler by overnight express or courier to the respective laboratory.

The primary laboratory address and POC are noted below:

Analytical Laboratory Services, Inc.
34 Dogwood Lane
Middletown, PA 17057
ATTN: Judy Kester/Sample Custodian
Phone: (717)944-5541
Fax: (717) 944-1430

A secondary laboratory (i.e., back-up) has been selected for the MMRP investigations, which can meet the analytical requirements of this program. The secondary laboratory, which is noted below, will analyze samples ONLY in instances when Analytical Laboratory Services cannot.

STL Savannah
5102 LaRoche Avenue

Savannah, GA 31404
ATTN: Linda Wolfe/Sample Custodian
Phone: (912) 354-7858
Fax: (912) 351-3673

6.8 INVESTIGATIVE DERIVED WASTE (IDW)

IDW will not require containerizing or special disposal procedures. Soil cuttings and excess sample material will be returned to the sample hole or boring for backfill purposes immediately after completion of sampling.

Decontamination fluids are not expected since dedicated/disposable field sampling equipment will be used. Used gloves, core liners, and any other disposable sampling equipment or personal protective equipment will be double bagged and disposed of off-site as non-hazardous waste.

7 REFERENCES

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U.S. Environmental Protection Agency. Region 9, Preliminary Remediation Goals Table. 2004

Appendix A: Quality Assurance Program Plan

FINAL
QUALITY ASSURANCE PROGRAM PLAN
MILITARY MUNTIONS RESPONSE PROGRAM
SITE INSPECTIONS

JUNE 2006

Prepared for:

U.S. ARMY CORPS OF ENGINEERS, BALTIMORE DISTRICT
P.O. Box 1715
Baltimore, Maryland 21203-1715

Prepared by:

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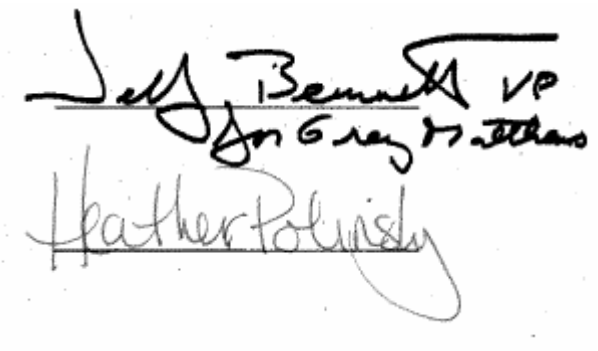
FINAL

QUALITY ASSURANCE PROGRAM PLAN
MILITARY MUNITIONS RESPONSE PROGRAM
SITE INSPECTIONS

DoD Contract Number: W912DR-05-D-0004

Reviewed and Approved by:



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Malcolm Pirnie, Inc. prepared this report at the direction of the U.S. Army Corps of Engineers (USACE). This document should be used only with the approval of the USACE. This report is based, in part, on information provided in other documents and is subject to the limitations and qualifications presented in the referenced documents.

JUNE 2006

TABLE OF CONTENTS

ACRONYMS ..... **vi**

1.0 INTRODUCTION **1-1**

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES **2-1**

3.0 QUALITY ASSURANCE AND CONTROL OBJECTIVES **3-1**

3.1 Introduction 3-1
3.2 TPP Process 3-1
3.2.1 Data Quality Objectives 3-1
3.2.2 Identify Decision Types 3-2
3.2.3 Identify Data Uses and Needs 3-3
3.2.4 Design Data Collection Program 3-6

4.0 FIELD SAMPLING TECHNIQUES **4-1**

4.1 Overview 4-1
4.2 Sample Collection 4-2
4.3 Geophysical Survey Procedures 4-2
4.4 Surface Soil and Sediment Sampling Procedures 4-2
4.5 Surface Water Sampling Procedures 4-2
4.6 Potable Water Sampling 4-2
4.7 Decontamination Procedures / Sample Contaminant Sources 4-3
4.7.1 Reagents 4-3
4.7.2 Procedure clarifications/exceptions 4-3
4.7.3 Sample Contaminant Sources and Other Potential Problem 4-4

5.0 GEOPHYSICAL INVESTIGATION **5-1**

5.1 Navigation 5-1
5.2 Quality Management 5-1

6.0 SAMPLE RECEIPT, HANDLING, AND CUSTODY PROCEDURES **6-1**

6.1 Overview 6-1
6.2 QA/QC Requirements 6-1
6.2.1 Field Notebook -Corrections to documentation 6-1
6.2.2 Photographs 6-1
6.2.3 Sample Labels - Potential Problems 6-1
6.2.4 Corrective Action 6-2
6.3 Field Corrective Action 6-2
6.4 Laboratory Corrective Action 6-2

7.0 ANALYTICAL PROCEDURES **7-1**

7.1 Preventative Maintenance 7-1
7.1.1 Field Equipment 7-1
7.1.2 Rental Equipment 7-2
7.1.3 Laboratory Equipment 7-2
7.2 Calibration Procedures & Frequency 7-2
7.3 Laboratory QC Procedures 7-3
7.4 Field Quality Control 7-3
7.5 Quality Control Samples 7-4

7.6 Performance And System Audits 7-5
7.6.1 Field Audit Procedures 7-5
7.6.2 Laboratory Audit Procedures 7-6
7.7 Nonconformance And System Audits 7-7
7.8 Routine Laboratory Analyses 7-8
7.9 Extraction Efficiencies 7-8
7.10 Method Detection Limits And Quantitation Limits 7-8

8.0 DATA REDUCTION / CALCULATION OF DATA QUALITY INDICATORS 8-1

8.1 Data Reduction 8-1
8.1.1 Field and Technical Data Reduction 8-1
8.1.2 Laboratory Data Reduction 8-1
8.2 Precision 8-1
8.3 Accuracy 8-2
8.4 Representativeness 8-2
8.5 Sensitivity 8-3
8.6 Comparability 8-3
8.7 Completeness 8-3

9.0 DATA ASSESSMENT PROCEDURES 9-1

9.1 Data Verification/Validation 9-1
9.1.1 Field and Technical Data Validation 9-1
9.1.2 Analytical Data Validation 9-1

10.0 QUALITY ASSURANCE REPORTING 10-1

10.1 Daily Quality Control Report 10-1
10.1.1 Daily Quality Control Report Procedures 10-1
10.1.2 DCQR Corrective Action 10-1
10.2 Data Report – Split Sample Analyses 10-1
10.3 Quality Control Summary Report 10-1
10.4 MMRP Databases 10-2

11.0 REFERENCES 11-1

LIST OF TABLES

TABLE 4-1: Analytical Procedure, Holding Times, Preservatives, and Sample Containers 4-1
TABLE 7-1: QC Procedures 7-3
TABLE 7-2: QC Checks 7-3

LIST OF FIGURES
(All located in Appendix B)

Figure 7- 1: Inorganic Analysis By Inductively Coupled Plasma (ICP) Methods 6010
Figure 7- 2: Organic Analysis By Gas Chromatography And High Performance Liquid Chromatography (Method 8330)
Figure 7- 3: Common Anions Analysis (Method 9058)
Figure 7- 4: Quality Control Field Audit Report
Figure 7- 5: Nonconformance and Corrective Action Report
Figure 10- 1: Daily Quality Control Report

LIST OF APPENDICIES

Appendix A: Laboratory QAPP and Standard Operating Procedures for Analytical Methods
Appendix B: Figures
Appendix C: ERIS Database Format Example (One Sample One Analyte)
Appendix D: Procedure for Decontaminating Field Sampling Equipment

ACRONYMS

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CRQL	Contract Required Quantitation Limit
CSM	Conceptual Site Model
DoD	Department of Defense
DQCR	Daily Quality Control Report
DQO	Data Quality Objectives
ERIS	Environmental Restoration Information System
FS	Feasibility Study
HRR	Historical Records Review
IDL	Instruction Detection Limit
IDW	Investigation Derived Waste
MC	Munitions Constituents
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
MMRP	Military Munitions Restoration Program
NELAP	National Environmental Accreditation Program
NFA	No Further Action
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PM	Project Manager
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAO	Quality Assurance Objectives
QAPP	Quality Assurance Program Plan
QC	Quality Control
QCSR	Quality Control Summary Report
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
SI	Site Inspection
SOW	Scope of Work
SSC	Site Safety Coordinator
SS-QAPP	Site Specific Quality Assurance Program Plan
TPP	Technical Project Planning
U.S.	United States
USACE	United States Army Corps of Engineers
USAESCH	US Army Engineering and Support Center, Huntsville
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

Malcolm Pirnie, Inc. (Malcolm Pirnie) has prepared the following Quality Assurance Program Plan (QAPP) for the Military Munitions Response Program (MMRP) Site Inspection (SI) of MMRP eligible sites at various Army Installations across the United States (US), under US Army Corps of Engineers (USACE) Baltimore District, Contract Number DACA31-00-D-0043.

This QAPP provides general information and standard operating procedures applicable to sampling and analytical activities to be performed at all installations that MMRP SIs are being conducted by Malcolm Pirnie (within USACE, North and South Atlantic Divisions). The information includes definitions and generic goals for data quality and minimum requirements for quality assurance/ quality control (QA/QC) samples. The procedures address sampling and decontamination protocols; geophysical investigation; field documentation; sample handling, custody, and shipping; instrument calibration and maintenance; field and laboratory auditing; data reduction, validation, and reporting; corrective action requirements; and quality assurance reporting. It should be noted that QAPP may include discussions on procedures or methods that are not applicable to a specific site since it is intended to encompass all sites. A Site Specific QAPP (SS-QAPP) will be prepared for each individual installation where a Site Inspection is being conducted by Malcolm Pirnie. The SS-QAPP will serve as addendums to this QAPP and will be included as part of the site specific work plan. Per the contract, it is intended that once the QAPP is finalized, it will not be modified (except for programmatic changes) and will serve as a programmatic document. Site-specific sampling information and any exceptions or proposed changes to the QAPP will be addressed and included in the SS-QAPP. The majority of information contained in this QAPP should not be repeated in the SS-QAPP. The appropriate EPA Region and State Regulatory Agency method specific reporting limits will be included in each SS-QAPP to ensure that the analytical methods selected can achieve State reporting requirements. The methods specific to each site should specify the appropriate detection limit and reporting limit information. Any deviations from this QAPP (e.g., holding times, detection limits, sampling methods, etc.) should be brought to the attention of the USACE Project Manager.

The SS-QAPP should not be a stand-alone document from this QAPP. The QAPP will provide the majority of the QA/QC information; the SS-QAPP should simply supplement this information by providing for site-specific condition requirements.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

Project specific personnel responsibilities will be identified and discussed in detail in the site specific Work Plan. Malcolm Pirnie project personnel and their responsibilities are discussed in Section 1.2.2 of the Work Plan.

The primary laboratory selected to perform analyses for samples collected at MMRP eligible sites is capable of providing complete environmental analytical services consistent with USEPA protocols, certified under the National Environmental Accreditation Program (NELAP), and approved by the USACE. Detailed information regarding the laboratory personnel, facilities and procedures are presented in Appendix A of this QAPP. In instances when the primary laboratory cannot conduct the analyses, the secondary laboratory (i.e., back-up) personnel, facilities and procedures will be identified in the SS-QAPP.

3.0 QUALITY ASSURANCE AND CONTROL OBJECTIVES

3.1 Introduction

This section discusses quality assurance objectives (QAOs) for the MMRP SI. QAOs are the requirements specifying the quality of the environmental data needed to support the decision-making process. The uncertainty must be maintained at levels that will allow the resultant data to be used for its intended purposes.

The primary goal of the MMRP SI is to collect information necessary to make one of the following decisions:

1. Whether a Remedial Investigation/Feasibility Study (RI/FS) is required at a site,
2. Whether an immediate response is needed, or
3. Whether the site qualifies for no further action (NFA).

3.2 TPP Process

Technical Project Planning (TPP) is used to identify project objectives and design data collection programs to help ensure that the requisite type, quality, and quantity of data are obtained so that informed decisions can be made for site closeout. The TPP process is a critical component of the U.S. Army Corps of Engineers (USACE) quality management system and meets the American National Standard for planning the collection and evaluation of environmental data.

The TPP Process is a comprehensive and systematic process that involves four phases of planning activities. Use of the TPP Process is consistent with the philosophy of taking a graded approach to planning that will produce the type and quality of data needed for site-specific decision making.

3.2.1 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions, and are developed to achieve the level of data quality required to meet project goals. DQOs are implemented so the data is legally and scientifically defensible. The development of DQOs for a specific site and measurement takes into account project needs, data uses and types and needs, and data collection. These factors determine whether the quality and quantity of data are adequate for its end use. Sampling protocols have been developed and sample documentation and handling procedures have been identified to realize the required data quality.

The TPP session conducted for each SI is intended to establish the site-specific DQOs. The results of the TPP are incorporated into the SS-QAPP and the Work Plan for the site location (TPP memo an appendix of the Work Plan). The DQOs discussed below will be developed for the SI, either as an element of the HRR, TPP, or during completion of the Work Plan.

3.2.2 Identify Decision Types

Stage 1 of the DQO process should identify and involve the data users, evaluate all available information, and specify investigation goals and decisions.

3.2.2.1 Data Users

Due to the interdisciplinary nature of environmental investigations and/or sampling, it becomes important that all personnel involved with the investigation be identified, including individuals associated with collecting and analyzing environmental samples, and individuals at the regulatory agencies that will review investigative results. The SS-QAPP will identify the individuals responsible for data collection and data quality.

3.2.2.1.1 Data Quality for Sample Analysis

A number of factors relate to the quality of data and its adequacy for use in the corrective action process, including the following considerations:

Age of the data;
Analytical methods used;
Detection limits of method; and
QA/QC procedures and documentation.

3.2.2.1.2 Data Quality for Sample Collection

Methods used for sample collection are as important to consider as the methods used for sample analysis. These considerations fall into two broad categories: statistical and SOPs. The statistical considerations relate to the representativeness of the data and the level of confidence that may be placed in conclusions drawn from the data.

Following SOPs ensure sample integrity and data comparability and reduces sampling and analytical error. Typical issues to consider include the following:

Sampling objective and approach;
Sample collection methods;
Chain-of-Custody documentation;
Sample preservation techniques;
Sample shipment methods; and
Holding times.

If limited or no information exists on sample collection, preservation techniques, or holding times, the data should be interpreted with caution, if they can be accepted at all.

3.2.2.1.3 Data Adequacy

The uncertainty associated with each data measurement activity should be considered when data are evaluated. Although data may be validated analytically, the level of precision of a particular data point may not provide sufficient certainty for use in a

decision. The uncertainty associated with a decision is a function of the statistical distribution of the factors that were used in reaching the decision. Assessment of data adequacy has two steps. The first step is data validation. The second step is determining if the data is sufficient to reduce the uncertainty surrounding a decision to an acceptable level.

Data validation identifies invalid data and qualifies the usability of the remaining data. The output of data validation is qualitative or quantitative statements of data quality. Once the quality of individual measurements is known, a compilation of all data points into a cohesive statement can be made. The confidence associated with a statement incorporates both the confidence in individual measurements as well as in the decision.

3.2.2.1.4 Conceptual Model

Conceptual site models (CSMs) describe a site and its environs and present hypotheses regarding the contaminants present, their route of migration, and their potential impact on sensitive receptors. For the Army SIs, a CSM is developed as a component of the HRR. The hypotheses are tested, refined and modified throughout the investigation.

3.2.3 Identify Data Uses and Needs

Stage 2 of the DQO process defines data uses and specifies the types of data needed to meet the project objectives. This process begins when the project objectives are established. The CSM and TPP become the basis for determining data uses and data needs. Stage 1 determines if existing data meet the project objectives. If the existing data are sufficient, there is no need to collect additional data. If the data are insufficient, the types, quality, and quantity of data that must be collected are determined in Stage 2.

3.2.3.1 Identifying Data Quality Needs

The identification of data uses and data types must be defined during the initial phases of the investigation. As the project proceeds and more data becomes available, data types may change.

3.2.3.1.1 Appropriate Analytical Levels

The following analytical levels can be used as a guidance to help achieve data types:

Level I - field screening or analysis using portable instruments. Results are often not compound specific and not quantitative but results are available in real-time.

Level II - field analyses using more sophisticated portable analytical instruments (i.e., mobile or on-site lab). There is a wide range in the quality of data that can be generated, depending on such factors as suitable calibration standards, sample preparation equipment, and the training of the operator. Results are available in real-time or several hours.

Level III - SW-846 routine analytical parameters. All analyses are performed in an off-site laboratory following SW-846 protocols. Level III is characterized by rigorous QA/QC procedures and documentation.

Level IV - analytical analysis by pre-approved non-standard methods. All analyses are performed in an off-site approved analytical laboratory. Method development or method modification may be required for specific constituents or detection limits. Level IV should be characterized by rigorous QA/QC procedures and documentation.

Level V - physical property and engineering material analysis by approved standard or non-standard methods. All analyses are performed in an off-site laboratory. QA/QC protocols and documentation may be required for some analyses.

The following analytical types can also be used as a guidance to help achieve data types, and are defined by the USACE as follows:

a. Screening Data with Definitive Confirmation – Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data provide analytical identification and quantification, although the quantification may be relatively imprecise. At least 10% of the screening data are confirmed using analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality. The QA/QC elements of screening data include the following: sample documentation; chain-of-custody; sampling design approach; initial and continuing calibration; determination and documentation of detection limits; analyte identification; analyte quantification; analytical error determination; and definitive confirmation of at least 10% of the samples.

b. Definitive Confirmation – Definitive data are generated using rigorous analytical methods, such as EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produced are tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. The QA/QC elements of definitive data include the following: chain-of-custody; sampling design approach; initial and continuing calibration; determination and documentation of detection limits; analyte identification; analyte quantification; QC blanks; matrix spike recoveries; performance evaluation sample results (when specified); analytical error determination (precision of analytical method); and total measurement error determination (over all precision of measurement system).

For each generic data use, several of the analytical levels may be appropriate, and the decision maker needs further criteria to select the most appropriate level. Important criteria driving the decision are the contaminants of concern and the level of concern for each contaminant.

Engineering design typically requires information beyond analytical levels for chemical analyses. Physical property data (viscosity, soil organic carbon, etc.) may be necessary for engineering design, and in all likelihood would require more than one analytical level.

3.2.3.1.2 Action and Target Levels

The action level specifies a concentration above which some form of corrective action may need to be taken. The action level is defined by the regulatory agency to be a health and environmental standard or criteria value. The action level is intimately linked with a target level that defines the level of cleanup for corrective action. Project-specific action levels for activities conducted under the MMRP investigations are specified in the SS-QAPP.

A rough estimate of a target level is necessary to ensure that the chosen analytical methods are accurate at the target level. In addition, knowledge of the target level can influence the number of samples required and the selection of the analytical method.

3.2.3.1.3 Detection Limit Requirements

The action level can directly affect data quality requirements. The sampling and analysis methods used must be accurate at the detection limit. Since sampling accuracy is hard to evaluate or control, it is extremely important that the analytical technique chosen has a detection limit well below the action level. This must be considered when evaluating analytical options.

3.2.3.1.4 Critical Samples

Critical samples are those for which valid data must be obtained to satisfy the objective of the sampling and analysis program. Critical samples may be taken in duplicate, or as appropriate.

3.2.3.1.5 Identify Data Quantity Needs

In the absence of available data, the data users and decision makers will be required to develop a rationale for selecting sampling locations. Questions to guide the data users in selecting appropriate locations could include the following:

a. Do source materials still exist on the soil surface?
b. Is there evidence of soil disturbance or vegetative stress based upon review of aerial photographs?
c. Do geologic features in the area control ground water and surface water flow patterns?

d. Do site conditions favor surficial soil erosion or wind erosion?
e. Are sensitive receptors located in the vicinity of the site?

In situations where data are available, or as new data are added to a database, statistical techniques may be utilized in determining the number of data required.

3.2.4 Design Data Collection Program

Stage 3 of the DQO process entails design of the detailed data collection program for the investigation. The process of addressing elements in Stages 1 and 2, all of the components required for the completion of Stage 3, are available.

3.2.4.1 Assemble Data Collection Components

During Stage 2, specific DQOs were developed by media or sampling activity. The intent of Stage 3 is to compile the information and DQOs developed for specific tasks into a comprehensive data collection program. A detailed list of all samples to be obtained should be assembled in a format which includes phase, media, and sample type, number of samples, sample location, analytical methods, and QA/QC samples (type and number). In addition, a schedule for all sampling activities should be developed in bar chart or critical path method format.

3.2.4.2 Develop Data Collection Documentation

The output of the DQO process is a well defined SS-QAPP. The DQO process provides a framework to ensure that all the pertinent issues related to the collection of data with known quality are addressed. The DQO levels for sampling will be outlined in SS-QAPP documents.

4.0 FIELD SAMPLING TECHNIQUES

4.1 Overview

The following section describes the standard operating procedures (SOPs) that will be followed for sample collection in order that representative samples will be collected. The number of samples for each sample location, including QA and QC samples is provided in the SS-QAPP. Table 4-1, provided below, outlines the types of sample containers and preservatives required for sample collection. All field teams will be required to strictly adhere to the procedures provided in the Work Plan, QAPP, and the Health and Safety protocols provided in the Health and safety Plan (HASP) and site Specific HASP (SS-HASP). Prior to commencement of field activities, all on-site personnel will be trained in health and safety techniques and site-specific operations.

Each Work Plan shall include a project description, sampling rationale, sampling strategy, sample collection and procedures, decontamination of field equipment, and sample documentation.

Note: The sampling procedures outlined below are a generic collection of sampling procedures. The fact that these sampling methods are listed in this document does not mean particular sampling event will be performed under this contract. However, the following SOPs will be followed in the event that such sample collection is necessary.

TABLE 4-1: Analytical Procedure, Holding Times, Preservatives, and Sample Containers

Media / Parameter	Analytical Procedure	Holding Time	Preservative	Container
Water:				
Perchlorate	SW-846 6850	28 days	4 deg C	125 mL HDPE
Explosives	SW-846 8330	7 days – extraction 40 days - analysis	4 deg. C	(2) 1 Liter amber glass
Metals	SW-846 6010B	6 months	HNO_3 to pH < 2 4 deg. C	> 150 mL glass container
Soil / Sediment:				
Explosives	SW-846 8330	14 days – extraction 40 days - analysis	4 deg. C	6 ounce wide mouth jar
Metals	SW-846 6010B	6 months	HNO_3 to pH < 2 4 deg. C	Glass or plastic container > 3.00 g

[1]Containers for metals analyses pre-preserved from the lab.

4.2 Sample Collection

Unless otherwise stated, the order of sample collection for groundwater samples will be:

1. Perchlorates.
2. Explosives.
3. Total Metals.

Unless otherwise stated, the order of sample collection for soil samples will be:

4. Explosives.
5. Total metals.
6. Propellants.

Samples collected for perchlorate analysis will be kept separate for other parameters collected; perchlorate samples MUST be kept from temperature extremes and packed in an insulated container using pick "N" pluck foam sections or similar polyurethane insulation.

Samples collected for explosive and metal analyses will be immediately placed in a cooler and held at 4°C. Disposable gloves will be worn by the sampling personnel and changed between sampling points. The information presented in Section 4.2 shall be recorded in the field logbook at the time of sampling.

Sampling equipment will be decontaminated as discussed in Section 4.7. While performing any equipment decontamination, phthalate-free gloves (neoprene or natural rubber) will be worn in order to prevent phthalate contamination of the sampling equipment by interaction between the gloves and the organic solvent(s).

4.3 Geophysical Survey Procedures

The Work Plan will include a description of the procedures, the advantages and limitations to the technique chosen, the instrumentation, survey design, and data reduction and interpretation.

4.4 Surface Soil and Sediment Sampling Procedures

Please reference the Work Plan for details on soil and sediment sampling procedures.

4.5 Surface Water Sampling Procedures

Please reference the Work Plan for details on water sampling procedures.

4.6 Potable Water Sampling

Please reference the Work Plan for details on potable water sampling procedures.

4.7 Decontamination Procedures / Sample Contaminant Sources

This section provides instruction on choosing an appropriate decontamination scheme (s) for the project field sampling equipment in order to prevent or reduce cross-contamination of project samples. The applicability of each step in a decontamination protocol will depend upon the contaminants present onsite, the subsequent analysis to be performed, the composition of the sampling devices, etc. The appropriateness of a decontamination protocol is vital to the eventual validity of the analytical results and decisions made based upon those results. All sampling equipment that has come in contact with a potentially contaminated media must be cleaned prior to the subsequent use of that device. Devices may include bailers, pumps, shovels, scoops, split spoons, tube samplers, augers, etc. Another approach to minimizing the potential for cross-contamination may be to dedicate or use disposable sampling equipment. Standard Operating Procedures for Decontaminating Field Sampling Equipment are found in Appendix D.

4.7.1 Reagents

The detergent wash is a non-phosphate detergent solution used with brushing or circulating techniques to remove gross contamination, and/or as a mild neutralizing agent. Tap water is considered a rinse-water, preferably from a water system of known chemical composition. Acid rinses are used as the inorganic solubilizing agent, or as a mild neutralizing agent. These rinses are a 10-percent to 1-percent Hydrochloric Acid (HCl) solution prepared from reagent grade acids and deionized water, respectively. Solvent rinses are used as an organic solubilizing agent. Requirements for solvent types vary depending upon the nature of known organic contamination requiring solubilization; and any impurities present within the rinse which may potentially interfere or contribute to the subsequent analysis. All solvent rinses used must be of pesticide grade quality. Finally, the deionized water is organic-free reagent water. Analyte-free water may be used as deemed appropriate. All equipment will undergo a final rinse with distilled or deionized water.

4.7.2 Procedure clarifications/exceptions

The detergent wash is used in conjunction with scrubbing for gross contamination removal, followed by the appropriate rinses. For cleaning of pumping equipment or devices with inaccessible internal mechanisms, suggest circulating/flushing the system with the applicable solutions in the order given below. Solvent rinses for pumping equipment should be limited to a 10-percent dilution (vol./vol.) of acetone or isopropyl alcohol in water. Tubing used with peristaltic pumps may be flushing with hexane or dilute HCl, followed by a distilled water rinse depending on contaminants noted onsite. The decontamination of low carbon steel sampling devices should limit the acid rinse to a dilute 1-percent acid solution. All sampling equipment should be allowed to air dry prior to the next use. For this reason it is important to have sufficient sampling devices onsite which may be alternated. This practice will allow a thorough air drying of equipment without increasing sampling downtime. Alternatively, larger equipment (e.g., drill rig components, power augers, etc.) may be cleaned with a portable power washer or a steam cleaning machine in lieu of the protocols outlined above. Finally, depending upon the project, it may be appropriate to contain spent decontamination fluids and arrange for eventual disposal as investigation derived wastes (IDW). In these cases, it is important

that these containers be suitable for the eventual disposition of the materials, and therefore complies with any potentially applicable regulations.

4.7.3 Sample Contaminant Sources and Other Potential Problem

4.7.3.1 Carryover and leaching

Contaminant carryover between samples, and/or from leaching of the sampling devices, is very complex and requires special attention. Decisions concerning the appropriateness of the device's material composition must account for these carryover or leaching potentials, and whether these contaminants are of concern on the project. Equipment blanks may be used to assess contamination of this nature.

4.7.3.2 Adsorption

Contaminant adsorption is another problem which must be considered when deciding on an applicable sampling device or the appropriate composition material. This phenomenon is more critical when sampling an aqueous or gaseous media, due to the capability of lower levels of contaminant detection and the fact that the fluid matrix is more apt to potential contaminant transfer. PVC and other plastics are known to sorb organics and to leach plasticizers and phthalate esters. Polypropylene, and other thermoplastics, have been shown to sorb organics and environmental mercury efficiently, and should therefore be avoided in sampling devices, especially tubing. For these reasons, PTFE is commonly chosen over the PVC and plastics when working with organic or mercury contaminants. In addition, some pesticides and halogenated compounds preferentially adsorb to glass surfaces. For this reason, it is recommended that when taking aqueous samples, the sample container NOT be rinsed prior to sample collection; and the same container be rinsed with the extraction solvent after the sample has been quantitatively transferred to an extraction apparatus. Inorganics (metals) adsorption to containers is dependant upon the specific metal element, the concentration, pH, contact time, complexing agents present, and container composition. This is believed to be nominal and proper preservation of samples should prevent this. In deciding appropriate tubing to be used for aqueous sample acquisition, it is important to decide applicable material composition and diameter based upon the contaminant and the purpose of the data. Adsorption is less likely to occur when there is an increase in tubing diameter.

5.0 GEOPHYSICAL INVESTIGATION

5.1 Navigation

Positional precision and accuracy is required for geophysical investigations at MMRP eligible sites. Since detection and removal of buried MEC is a multi-stage process, it is important that positional information gathered at one stage be useable at the next stage. This means that all data collected at each stage must be tied to a common positional system. The positional system can either be temporary or permanent. The use of temporary or assumed location systems is strongly discouraged. U.S. Army Engineering and Support Center, Huntsville (USAESCH) recommends that all navigation be based on the local State Grid Plane system. For investigations conducted at MMRP sites, navigation is accomplished either using ropes (traditional method) or GPS. The traditional method is referenced to grid corner stakes surveyed on centers. Marked survey ropes are then placed laterally across each survey grid at evenly spaced intervals. Alternating colored markers on the ropes facilitate straight-line profiling and identify locations for the placement of fiducial marks within the recorded data. The second method of navigation is GPS. It is accomplished with a single GPS sensor mounted over the center of the coil to provide real-time positional tracking capabilities

5.2 Quality Management

The general objective of geophysical investigations during MMRP SI field activities is to efficiently locate buried MEC so that it can be properly evaluated. Specific geophysical investigation objectives of a project are defined by the project team and must be risk-based, measurable, and attainable.

There are two elements which are subject to QA/QC: processes and products. Processes are the project-specific geophysical planning and data collection/data analysis procedures and methods that must be performed. Products are the final project-specific deliverables and results that must be achieved. Both the project processes and the project products must be part of a formal quality management process in order to demonstrate that project quality objectives are met. For investigations conducted at MMRP sites, the data collection and analysis, data storage and preliminary and post processing of the data is described in detail in the subcontractors SOP located in Appendix A of this QAPP.

To ensure process quality management the project team must periodically check the geophysical data provided by the project team to assure positional accuracy, proper instrument calibration, and analysis confirmation.

6.0 SAMPLE RECEIPT, HANDLING, AND CUSTODY PROCEDURES

6.1 Overview

Sample custody during the field investigations will be performed in three phases. The first phase encompasses sample collection, pre-laboratory treatment procedures (preservation), packaging, and shipping field custody procedures. The second custody phase involves sample shipment, where mode of shipment, airbill numbers, dates and times are documented. The third phase involves the custody procedures employed by the laboratory. All three phases of sample custody will be performed to provide that:

- All samples are uniquely identified;
- The correct samples are tested and are traceable to their source;
- Important sample characteristics are preserved;
- Samples are protected from loss, damage, or temperature extremes; and
- A record of sample integrity is established and maintained through the entire custody process.

6.2 QA/QC Requirements

6.2.1 Field Notebook -Corrections to documentation

All original data recorded in field logbooks and on sample labels, chain of custody records, and receipt for samples forms are written in waterproof ink. If an error is made on an accountable document, corrections should be made simply by crossing out the error and entering the correct information. The erroneous information should not be obliterated. Any error discovered on a document should be corrected by the person who made the entry. All corrections must be initialed and dated.

6.2.2 Photographs

The photographer should review the photographs and compare them with the photographic log to confirm that the log and photographs match.

6.2.3 Sample Labels - Potential Problems

Although most sample labels are made with water-resistant paper and are filled out using waterproof ink, inclement weather and general field conditions can affect the legibility of sample labels. It is recommended that after sample labels are filled out and affixed to the sample container, the label should be covered with wide clear tape. This will preserve the label and keep it from becoming illegible. In addition to label protection, chain of

custody and analysis request forms should be protected when samples are shipped in iced coolers. Typically, these forms should be placed inside a Ziploc bag or similar waterproof protection and taped to the inside lid of the secured shipping container with the samples.

6.2.4 Corrective Action

Corrective actions are those measures taken to rectify a laboratory or field measurement system that does not comply with this QAPP. The need for corrective action may be identified by system or performance audits or by standard QC procedures. The essential steps in the corrective action system are:

- Identifying and defining the problem.
- Assigning of responsibility for investigating the problem.
- Investigating and determining the cause of the problem.
- Determining a corrective action to eliminate the problem.
- Assigning and accepting responsibility for implementing the corrective action.
- Implementing the corrective action and evaluating its effectiveness.
- Verifying that the corrective action has eliminated the problem.

6.3 Field Corrective Action

At the end of each sampling day, the sampling team shall report any problems requiring corrective action which were encountered during the day. Corrective action will be undertaken when a non-conforming condition is identified. A non-conforming condition occurs when QA objectives for precision, accuracy, completeness, representativeness or comparability are not met, or when procedural practices or other conditions are not acceptable. A report shall be filed which documents the problems encountered and the corrective action implemented. A stop-work order may be issued by the Project QA/QC Coordinator, upon authorization by the Project Manager, if corrective action does not adequately address a problem, or if no resolution can be reached.

6.4 Laboratory Corrective Action

If a particular analysis is deemed "out-of control," corrective action will be taken to ensure continued data quality. Actions which may be taken include, but are not limited to:

- Rechecking calculations;
- Checking QC data on other samples;
- Auditing laboratory procedures;
- Reanalyzing the sample if the holding time requirements have not been exceeded;

- Accepting data with the acknowledged level of uncertainty; and
- Discarding data.

The coordinator of the laboratory's analytical section will be responsible for initiating laboratory corrective action when necessary. Recommendations for corrective actions outside the laboratory will be made by the laboratory QA Manager to the Project Manager within 48 hours of corrective action. Corrective action procedures specific to the laboratory are described in the LQAM located in Appendix A of this QAPP.

7.0 ANALYTICAL PROCEDURES

7.1 Preventative Maintenance

A preventative maintenance program is necessary to help prevent delays in project schedules, poor output performance or erroneous results in investigative operations. Preventative maintenance on laboratory analytical equipment used in this program will be performed contractually by qualified personnel. Maintenance of field equipment will be performed routinely for sampling events. More extensive maintenance will be performed based on hours of use, by a qualified servicing organization. Repairs, adjustments and calibrations will be recorded.

7.1.1 Field Equipment

The three elements of the field equipment maintenance program include normal upkeep of equipment, service and repair (when required), and formalized record-keeping of all work performed on each piece of equipment. This section addresses the normal equipment upkeep element of the maintenance program. For most of the equipment, normal maintenance will consist of cleaning outside surfaces, lubrication of all moving parts, and, if applicable, a battery level check and recharge or replacement as necessary. This program will include the maintenance of all monitoring, measuring, and test equipment returning from use or any equipment used on a daily basis. The frequency of maintenance checks will be dependent on the individual needs and use of each piece of equipment. Maintenance procedures will be only those necessary for keeping an instrument in service or in preparation for everyday use. It is beyond the scope of this document to cover repair procedures for each piece of equipment. Repair problems will be referred to the manufacturer or other qualified servicing organization.

The Project QA/QC Coordinator, or the designated task leader, will be responsible for keeping all maintenance records, making sure all equipment used is maintained properly, informing field team members of any specific maintenance requirements for equipment used at the site and shipping any instrument in need of repair to the correct source.

The field personnel responsibilities include maintaining each piece of equipment located at the site and the maintenance of equipment after use. A record of equipment maintenance and repair will be kept in the field logbook.

Equipment used during the geophysical investigations will be in accordance with maintenance procedures outlined in the geophysical SOP documented located in Appendix B of this QAPP.

7.1.2 Rental Equipment

Rental equipment used on the project will be obtained only from a certified rental supplier. The equipment will require a pre-receipt to verify accuracy, maintenance and up-keep of the equipment. A receipt indicating that the equipment has been checked upon return will be required as well.

7.1.3 Laboratory Equipment

An important factor in maintaining accuracy and precision, achieving required holding times, and addressing contract schedule is preventive maintenance. As part of the laboratory's maintenance program, service contracts are held on critical analytical instruments. Information regarding routine maintenance performed on laboratory equipment is described in the SOP documents located in Appendix A of this QAPP.

7.2 Calibration Procedures & Frequency

Measuring and test equipment shall have an initial calibration and shall be recalibrated at scheduled intervals against certified standards that have known and valid traceability to recognized national standards. Calibration intervals for each item shall be, at a minimum, in accordance with manufacturer's recommendations as defined in the equipment manual. Test equipment used for calibration of sensors shall themselves be recalibrated at least once a year or when maintenance or damage indicates a need for recalibration.

Calibration standards shall be maintained and used in an environment with temperature, humidity, and cleanliness controls that are compatible with the accuracy and operating characteristics of the standards. An inspection will be made during the equipment calibration to evaluate the physical condition of the equipment. The purpose of the inspection is to detect any abnormal wear or damage that may affect the operation of the equipment before the next calibration. Equipment found to be out of calibration or in need of maintenance or repair will be identified and removed from service.

The Project QA/QC Coordinator shall be notified if the test equipment is found to be out of tolerance during inspection and calibration. The corrective actions to be taken include evaluating the validity of previous inspection or test results; evaluating the acceptability of the items inspected or tested since the last calibration check; and repeating the original inspections or tests using calibrated equipment when it is necessary to establish the acceptability of previous inspections or tests. Specifics regarding QC checks and verification of field equipment stability are located in Appendix A of this QAPP.

Each item of measuring and test equipment in the calibration program shall be identified in such a way as to show its calibration status and calibration expiration date. Equipment history records for measurement and test equipment shall be used to indicate calibration status and conditions, corrections to be applied, results of in-service checks, and repair history. This will provide a basis for establishing calibration frequencies and for remedial action if the instrument is found out of calibration.

Laboratory instrumentation calibration procedures, frequency, and standards will be consistent with the requirements of the applicable analytical method. Information regarding laboratory calibration procedures is presented in the SOP documents located in Appendix A of this QAPP. If the secondary (i.e., back-up) laboratory is used, that laboratories analytical SOPs will be included as an attachment to the SS-QAPP documents.

7.3 Laboratory QC Procedures

This section should identify the specific internal QC measures to be used by the laboratory when performing the analytical tests. Type and frequencies of specific QC samples performed by the laboratory are dependent upon analytical requirements specific to the method analyzed. Internal QC methods require performance on a sample batch basis and include analyses of method blanks, laboratory control samples, and actual environmental samples as duplicates, matrix spikes, and matrix spike duplicates. Additional QC is incorporated into the analytical sequence. All analyses shall include the following QC procedures, where applicable:

TABLE 7-1: QC Procedures

Procedure	Frequency
Calibration	As required
Standards	Daily
Method Blanks	Daily
Duplicates	5%, per batch, or per analytical run
Matrix Spikes	5%, per batch, or per analytical run
Surrogates	Each sample
QC Check Samples	Daily

7.4 Field Quality Control

The QC checks employed for field instruments include the following:

TABLE 7-2: QC Checks

QC Method	Purpose	Frequency
Calibration Check	Ensures proper working order of field instrument.	Daily
Field Duplicate Sample	Measures accuracy and sensitivity.	One per ten samples
MS/MSD	Measures instrument precision.	One per twenty samples (minimum of 1 MS and MSD per site)
Field Rinsate Blanks	Measures cross-contamination	Daily as required*

*In the event that non-disposable/dedicated equipment is used equipment rinsate samples will be collected at a rate of one per day.

7.5 Quality Control Samples

The QA/QC samples that will be required for the sampling program shall be identified in the Work Palan documents. The types of QA/QC samples are described below:

Field Sample - The total sample collected at a specific site location. This sample may be any matrix and may be divided to provide material for QA/QC analysis.

Quality Control (QC) Samples - Samples analyzed to help identify potential problems related to sample collection or analysis. QC samples include replicate and split samples, trip blanks, rinsate blanks and filtration blanks.

Quality Assurance (QA) Samples - Split samples sent to the secondary (i.e., back-up) laboratory for analysis to evaluate the primary laboratory's performance. QA samples represent approximately 10% percent of the field samples. The collection of QA samples is not anticipated.

Matrix Spike/Matrix Spike Duplicates - Aqueous VOC and extractable organic samples collected at three times their standard volume at the frequency of approximately five percent (5%) of the field samples. After sample analysis, the additional sample volume is spiked with a known quantity and reanalyzed. The percent recovery will be used to calculate accuracy. The relative percent difference (RPD) for each component will be used to calculate precision.

Split Samples - Samples collected as a single sample, homogenized, divided into two or more equal parts and placed into separate containers. The sample shall be split in the field prior to delivery to the laboratory. Split samples will be taken at a frequency of approximately 10% per matrix.

Replicate (duplicate, triplicate, etc.) Samples - Multiple grab samples, collected separately, that equally represent a medium at a given time and location. This is the type of co-located sample required for volatile organic analyses and most ground water and surface water samples. Replicate samples will be taken at a frequency of approximately 10% per matrix.

Filtration Blank - When groundwater samples are filtered prior to collection and analysis, a filtration blank is collected. Deionized water is run through a clean filter and submitted as a blank sample to assess the potential for contamination by the filter/filtration process. The filter shall be identical as those used for the field sample filtering.

Field Rinsate Blank - Samples collected from a final rinse of sampling equipment with deionized demonstrated analyte-free water after the decontamination procedure has been performed. The purpose of the field rinsate blank is to determine whether the sampling equipment is causing cross-contamination of samples. The frequency of field blank collection is dependent on the number of decontamination events; i.e., one field blank per decontamination event per equipment type. The number of field blanks should not exceed one per day. Field blanks must be preserved in the same manner as aqueous environmental samples.

Deionized Demonstrated Analyte-Free Water - Deionized demonstrated analyte-free (DI) water is water of a known quality which has been demonstrated through analysis not to possess any contaminants of concern at levels greater than the CLP contract required quantitation limits (CRQLs), as defined in the current CLP Statements of Work (SOW). DI water is used in the final rinse step of decontamination and in the preparation of field rinsate blanks.

7.6 Performance And System Audits

Audits will include a careful evaluation of both field and laboratory quality control procedures and will be performed before or shortly after systems is operational. The audits will be conducted by an individual who is technically knowledgeable about the operation(s) under review. Systems audits provide a quantitative measure of the quality of the data produced by one section or the entire measurement process. Performance audits are conducted by introducing control samples into the data production process. These control samples may include performance evaluation samples, field samples spiked with known amounts of analyte, and split field samples that are analyzed by two or more analysts within or without the organization. Systems audits are onsite qualitative inspections and reviews of the quality assurance system used by some part of or the entire measurement system. The audits are performed against a set of requirements, which may be a quality assurance project plan or work plan, a standard method, or a project statement of work. The primary objective of the systems audits is to ensure that the QA/QC procedures are being followed.

7.6.1 Field Audit Procedures

Field performance audits will be conducted on an ongoing basis during the project as field data are generated, reduced, and analyzed. All numerical manipulations, including manual calculations, will be documented. All records of numerical analyses will be legible, of reproduction-quality, and sufficiently complete to permit logical reconstruction by a qualified individual other than the originator.

Indicators of the level of field performance include the analytical results of the blank and replicate samples. Each blank analysis will be considered an indirect audit of the effectiveness of measures taken in the field to ensure sample integrity (e.g., field decontamination procedures). The results of the field replicate analyses are an indirect audit of the ability of each field team to collect representative sample portions of each matrix type.

System audits of site activities will be accomplished by an inspection of all field site activities. During this audit, the auditor(s) will compare current field practices with standard procedures. The following elements will be evaluated during a field system audit:

- All activities conducted in accordance with the Work Plan;
- All procedures and analyses conducted according to procedures outlined in the QAPP;
- Sample documentation;
- Working order of instruments and equipment;
- Level of QA conducted per each field team;
- Contingency plans in case of equipment failure or other event preventing the planned activity from proceeding;
- Decontamination procedures;
- Level of efficiency with which each team conducts planned activities at one site and proceeds to the next; and
- Sample packaging and shipment.

After completion of the audit, any deficiencies will be discussed with the field staff and corrections identified. If any of these deficiencies could affect the integrity of the samples being collected, the auditor(s) will inform the field staff immediately, so that corrections will be implemented immediately. The audit will be performed by the Project QA/QC Coordinator or the Site Field Manager. The audit form is presented as Figure 7-4 located in Appendix B of this QAPP.

7.6.2 Laboratory Audit Procedures

7.6.2.1 Systems/Internal Audits

As part of its Quality Assurance Program, the Laboratory Quality Assurance Manager shall conduct periodic checks and audits of the analytical systems. The purpose of these is to ensure that the analytical systems are working properly and that personnel are adhering to established procedures and documenting the required information. These checks and audits will also assist in determining or detecting where problems are occurring.

The Quality Assurance Manager will periodically review laboratory control samples. These samples will check the entire analytical method, the efficiency of the preparation method and the analytical instrument performance. The results of the control samples are

reviewed by the Quality Assurance Manager. The Quality Assurance Manager reports the results to the analyst and the Laboratory Manager. When a problem is indicated, the Quality Assurance Manager will assist the analyst and laboratory management in determining the reason and in developing solutions. Rechecking of systems will be conducted by the Quality Assurance Manager as required.

7.6.2.2 Performance and External Audits

In addition to conducting internal reviews and audits, as part of its established Quality Assurance program, the laboratory is required to take part in regularly scheduled Performance Evaluations and laboratory audits from State and Federal agencies. These are conducted as part of certification processes and to monitor the laboratory performance. These provide an external quality assurance check of the laboratory and provide reviews and information on the management systems, personnel, SOPs, and analytical measurement systems. Acceptable performance on evaluation samples and audits is required for certification and accreditation. The laboratory shall use the information provided from these audits to monitor and assess the quality of its performance. Problems detected in these audits shall be reviewed by the Quality Assurance Manager and laboratory management and corrective action shall be instituted as necessary.

7.7 Nonconformance And System Audits

A nonconformance is defined as an identified or suspected deficiency in an approved document (e.g., technical report, analysis, calculation, computer program); an item where the quality of the end item itself or subsequent activities using the document or item would be affected by the deficiency; or an activity that is not conducted in accordance with the established plans or procedures. Any staff member engaged in project work that discovers or suspects a nonconformance is responsible for initiating a nonconformance report (see Figure 7-5 in Appendix B). The Project QA/QC Coordinator shall evaluate each nonconformance report and shall provide a disposition, which describes the actions to be taken. The Project Manager shall ensure that no further project work dependent on the nonconforming item or activity is performed until approval is obtained and the nonconformance report is closed out. If the nonconformance is related to material, the Project Manager shall be responsible for marking or identifying, with the nonconformance report number, the nonconforming item (if practical) and indicating that it is nonconforming and is not to be used.

Samples that are analyzed prior to the resolution of a nonconforming event will be resampled, and/or reanalyzed once the corrective action has been demonstrated to be effective.

A copy of each closed nonconformance report shall be included in the quality assurance file. Copies of all nonconformance reports shall be maintained by the Project QA/QC Coordinator.

7.8 Routine Laboratory Analyses

The analytical procedures for samples collected will follow those specified in Figures 7-1 through 7-3 provided in Appendix B. The sample holding time requirements are noted on Table 4-1. The proposed analytical methods shall be identified in the SS-QAPP documents. Test Methods for Evaluating Solid Waste, USEPA Office of Solid Waste, SW-846, 3rd Edition, Revision No. 2, June 1990; Methods for Chemical Analysis of Water and Wastes, USEPA Office of Research and Development, March 1983; and American Society for Testing Materials, Annual Book of ASTM Standards are incorporated by reference into this QAPP for the purpose of describing the standard analytical methods. The instrument and method detection limits and reporting limits specific to laboratory is included in Appendix A of this QAPP. In instances where detection and/or reporting are revised due to updates, modifications to SOPs, and/or changes in instrumentation, the revised detection and reporting limit information will be included in the Site-Specific SS-QAPP documents.

Laboratories providing analytical support must be certified by the State Regulatory Department, NELAP and be in compliance with the latest version of the DOD Quality Systems Manual. If the laboratory's state or federal certifications expire during MMRP investigations, the laboratory must follow the appropriate procedures to maintain certifications.

In the event that analytical parameters are not validated by either the State Regulatory Department and/or the USACE through the performance of proficiency samples and on-site audits, laboratory SOPs will be forwarded to the USACE chemist and state regulatory personnel for review during the stages of the work plan development.

7.9 Extraction Efficiencies

The method chosen for analyses are the standard analytical methods used within the laboratory industry. The analytical data generated by these standard methods provide information used to make critical decisions at the site. As part of the method, sample preparation or extraction techniques prepare the sample prior to analysis. A way to measure the "integrity" of the method is to introduce known amounts and concentrations of known compounds and subject them to the extraction and analysis procedures outlined in the method. These added compounds are measured after analysis and represent the response of the unknown compounds in the sample. The analytical results provide a tool to measure the extraction efficiency of a particular analysis.

7.10 Method Detection Limits And Quantitation Limits

Analyte and associated detection and quantitation limits are presented by method in Appendix A of this QAPP. Actual detection and quantitation limits for specific samples will vary depending on the amounts and types of compounds present in the sample. A significant concentration of one compound may require that the sample be diluted, which increases the detection limits and sample quantitation limits accordingly. In addition, the occurrence of one compound may interfere with the detection of other compounds.

The Method Detection Limit (MDL) is a level at which the analytical procedure referenced is capable of determining with a 99% probability that the constituent is present. The procedure for determining the MDL includes the complete analytical procedure, including any sample preparation such as extractions and digestions. This procedure involves the replicate analysis (seven replicates as a minimum) of a sample with an analyte concentration near, but greater than zero. The standard deviation at this concentration is then calculated.

The Instrument Detection Limit (IDL) establishes the noise level of the instrument under routine operating conditions.

The Practical Quantitation Limit (PQL) establishes a limit with a higher level of precision than associated with the detection limit, but does not represent the lowest achievable detection limit. The PQL is usually the laboratories reporting limit.

The current detection and reporting limit information is presented in Appendix A of this QAPP. In instances where detection and reporting limits are revised due to updates, modifications to SOPs, and/or changes in instrumentation, the current detection and reporting limit information will be included in the Site-Specific SS-QAPP documents.

8.0 DATA REDUCTION / CALCULATION OF DATA QUALITY INDICATORS

8.1 Data Reduction

8.1.1 Field and Technical Data Reduction

Field personnel will record all field data in bound field notebooks and on standard forms. After checking the validity of the data in the field notes, the Site Field Manager or his designee will reduce the data to tabular form, when possible, by entering the data into data files. Where appropriate, the data files will be set up for direct input into the project database. Subjective data will be filed as hard copies for later review by the Project Manager and incorporation into technical reports, as appropriate.

8.1.2 Laboratory Data Reduction

Data reduction is the process by which raw analytical data generated from laboratory instrument systems is converted into usable concentrations. The raw data, which may take the form of area counts, instrument responses or observations, is processed by the lab and converted into concentrations expressed in the parts-per-million (ppm) or parts-per-billion (ppb) range. Raw data from these systems include compound identifications, concentrations, retention times, and data system print-outs. Raw data is usually reported in graphic form, bar-graph form, or tabular form. The laboratories will follow SOPs consistent with the data handling requirements of the applicable methods.

The Laboratory Reporting Limits (RLs) must be less than or equal to those stipulated in the published methods and must be significantly less than the action levels developed for the site investigations. The laboratory RLs are presented in Appendix A of this QAPP. In instances where RLs are revised due to updates, modifications to SOPs, and/or changes in instrumentation, the current RL information will be included in the Site-Specific SS-QAPP.

8.2 Precision

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed conditions. Assessing precision measures the random error component of the data collection process. Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions, and is calculated as an absolute value. The degree of agreement, expressed as the relative percent difference (RPD), is calculated using the formula below.

$$\text{RPD} = \frac{(V_1 - V_2)}{\frac{(V_1 + V_2)}{2}} \times 100$$

where: V1 = value 1
V2 = value 2

Analytical precision is assessed by analyzing matrix spike/matrix spike duplicate pairs and laboratory duplicate samples. Field precision is assessed by measurement of field duplicate samples. The objective for precision is to equal or exceed the precision demonstrated for similar samples and should be with the established control limits for the methods. Precision control limits and QC RPD limits are presented as part of the SS-QAPP.

8.3 Accuracy

Accuracy is the degree of agreement of a measurement with an accepted reference or true value. Accuracy measures the bias or systematic error of the entire data collection process. Sources of these errors include the sampling process, field and laboratory contamination, sample preservation and handling, sample matrix interferences, sample preparation methods, and calibration and analytical procedures. To determine accuracy, a reference material of known concentration is analyzed or a sample which has been spiked with a known concentration is reanalyzed. Accuracy is expressed as a percent recovery and is calculated using the following formula:

$$\%\ \text{Recovery} = 100 \times \frac{\text{measured value}}{\text{true value}}$$

Recoveries are assessed to determine method efficiency and matrix interference effects. Analytical accuracy is measured by the analysis of calibration checks, system blanks, quality control samples, surrogate spikes, matrix spikes, and other checks required by the selected analytical methods. Sampling accuracy is assessed by evaluating the results of field and trip blanks. Sampling accuracy is also maintained by frequent and thorough review of field procedures. The objective is to meet or exceed the demonstrated accuracy for the analytical methods on similar samples and should be within established control limits for the methods. Accuracy control limits and MS/MSD and surrogate recovery limits are presented as part of the SS-QAPP.

8.4 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is achieved through proper development of the field sampling program. The sampling program must be designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected. The objective of obtaining representativeness of samples will be met through the implementation of the work plan and SS-QAPP.

8.5 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between small differences in analyte concentration. The sensitivity and detection limits for methods applicable to MMRP investigations are presented in Appendix A of this QAPP.

8.6 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability cannot be described in quantitative terms, but must be considered in designing the sampling program. Thus, this objective will be met by using standard methods for sampling and analyses and by following techniques and methods set forth in the project specific work plan and SS-QAPP.

8.7 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data is complete and valid if it meets all acceptance criteria including accuracy, precision, and any other criteria specified by the particular analytical method being used. Completeness is calculated as follows:

$$\% \text{ Completeness} = 100 \times \frac{V}{n}$$

where: V = number of measurements judged valid
n = total number of measurements

The objective is to generate a sufficient database with which to make informed decisions. To help meet the completeness objective, every effort must be made to avoid sample loss through accidents or inadvertence. The completeness objective for each project is stated in the SS-QAPP.

9.0 DATA ASSESSMENT PROCEDURES

9.1 Data Verification/Validation

9.1.1 Field and Technical Data Validation

Validation of objective field and technical data will be performed at two different levels. The first level of data validation will be performed at the time of collection by following standard procedures and quality control checks. The Site Field Manager who will review the data to ensure that the correct codes and units have been included will complete the second level of data validation. After data reduction into tables and arrays is complete, the Field Manager will review data sets for anomalous values. The Project Manager, who will review field reports for reasonableness and completeness, will validate subjective field and technical data. In addition, the Field Manager and/or Site QA/QC Coordinator will make random checks of sampling and field conditions.

9.1.2 Analytical Data Validation

The laboratory shall review data prior to its release from the laboratory. The analytical method performance will be determined by an examination of precision, accuracy, and completeness, as discussed in Section 8.0, as well as a review of the following quality controls:

- Method Blanks: Measure of laboratory contamination and accuracy.
- Laboratory Duplicates: Measure of laboratory precision.
- Field Duplicates: Measure of field sampling and laboratory precision.
- Matrix Spikes: Measure of laboratory accuracy and any sample matrix effects.
- Surrogate Spike Recoveries: Measure of laboratory accuracy.
- Laboratory Control Samples: Measure of laboratory accuracy.

The laboratory is required to evaluate their ability to meet these objectives. Outlying data shall be flagged in accordance with laboratory SOPs and corrective action shall be taken to rectify the problem. The laboratory case narratives shall describe how the data did or did not meet the method criteria and must describe the overall quality of the data and whether or not the data are valid and usable.

In order to ensure the analytical data generated by the laboratory are accurate, the project chemist will review the electronic data deliverable from the laboratory to ensure that the data submitted electronically correspond to the hard copy results in the laboratory data deliverable. The SS-QAPP shall address the project team members responsible for the electronic data review.

10.0 QUALITY ASSURANCE REPORTING

10.1 Daily Quality Control Report

A Daily Quality Control Report (DQCR) will be completed for each day of field activities. An in-house inspection of these reports will be reviewed as they are generated field personnel. A sample report is presented as Figure 10-1 provided in Appendix B.

10.1.1 Daily Quality Control Report Procedures

During field investigation activities, DCQR will be completed, dated, and signed by the sampling technician at the end of each workday. Copies will be distributed to the field supervisor and project chemist on a daily basis. These DQCR shall include, but are not limited to the following information:

a. Weather conditions at the time of sampling.
b. Level of Personal Protective Equipment.
c. Sample collected including reference to applicable QAPP sections.
d. Field instrument measurements and calibrations.
e. Any deviations from the QAPP, problems identified, and corrective actions taken.

10.1.2 DCQR Corrective Action

If a significant problem occurs during sampling, the DQCR will be provided to the project chemist within 48 hours accompanied by a corrective action report. The DQCR will be written by the sampling technician and will be cross checked against the field logbook for completeness at the end of each day. A sample DQCR form is shown in Figure 10-1.

10.2 Data Report – Split Sample Analyses

The data of QA/QC (split) samples is not anticipated for MMRP investigations; however, in the event QA split samples are collected, the data from the initial and confirmation analyses will be evaluated using the data quality element of precision. Data packages form the secondary laboratory will include the following information: all blank sample and internal quality control results such as spike, surrogate recoveries, and replicate analyses.

10.3 Quality Control Summary Report

A Quality Control Summary Report (QCSR) will be submitted as part of the report of investigation activities. The QCSR may be incorporated into the field investigation report. The QCSR will address:

- Project Scope,
- Project Description,
- Sampling Procedures (planned vs. implemented),
- Field Quality Control Activities (planned vs. implemented),
- Analytical Procedures,

- Significant Problems with Analytical Procedures,
- Data Presentation and Evaluation,
- Quality Control Activities including Discussion of Data Reliability,
- Lessons Learned, and
- DQCR Consolidation.

The report will also discuss any corrective actions implemented in response to problems encountered during the project. Data packages and data assessment reports will be summarized.

10.4 MMRP Databases

Analytical results will require input in the Environmental Restoration Information System (ERIS) Database. The data from MMRP investigations will be maintained in the database which includes the following information for each sample collected: sample ID; preservation; date sampled; media type; site location; chemical analyses; and validation review. The format requirements for the ERIS database are located in Appendix D of this QAPP.

If the ERIS database format is revised during MMRP investigations, the newly established database format shall be included as an appendix in the SS-QAPP.

11.0 REFERENCES

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Appendix A: Laboratory QAPP and Standard Operating Procedures for Analytical Methods

Quality Assurance Plan

Analytical Laboratory Services, Inc.

Environmental Program
34 Dogwood Lane
Middletown, PA 17057
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Prepared By:

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Approved By:

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Michael S. Farlling *Date*
President

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Laboratory Manager

Document Control No.:_____

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TABLE OF CONTENTS

1. Document History 2
2. TABLE OF CONTENTS 3
3. Quality Assurance Program Plan Identification Form 6
4. Quality Policy and Objectives 7
5. Organization and Management Structure 8
5.1. Ownership 8
5.2. Organizational Structure of Personnel 8
5.3. Job Descriptions 8
5.3.1. President 8
5.3.2. Technical Director and LIMS Administrator I 8
5.3.3. Laboratory Manager 9
5.3.4. Quality Assurance Manager 9
5.3.5. LIMS Administrator II 10
5.3.6. Laboratory Supervisor 10
5.3.7. Laboratory Analyst 11
5.3.8. Laboratory Technician 11
5.3.9. Laboratory Assistant 12
5.4. Safety 12
6. Training 12
6.1. Personnel Files 12
6.2. Technical Training 12
6.3. Method Specific Training 13
6.4. Minimum Experience and Training 13
6.5. Organizational Chart 13
7. Document Control 14
7.1. Computer Records 14
7.2. Hardcopy Records 14
7.3. Laboratory Logbooks 14
7.4. Laboratory Worksheets 14
7.5. Raw Data 15
7.6. Data Deliverable Packages 15
7.7. Quality Assurance Documents 15
7.8. External Documents 16
8. Traceability of Measurement 17
8.1. Reagents 17
8.2. Reference Standards 17
8.3. Removal of Expired Chemicals and Standards 18
8.4. Purchased Materials 19
8.4.1. Volumetric Glassware 19
8.4.2. Sample Collection Bottles 19
8.5. Balances 20
9. Scope of Tests 20
10. Review of Solicitation, Offer or Contract 20

10.1. Laboratory Capacity Review 21
10.2. Amendment to Contract 21
11. Demonstrating Method Performance 22
12. Client Services 22
12.1. Project Planning 23
12.2. Organizational and Technical Interface 23
13. Control of Nonconformance Testing 24
14. Test Method Reference 24
14.1. Analytical Procedure References 24
14.2. Standard Operating Procedures 25
14.3. Laboratory Analyses 26
14.3.1. Test Scheduling 26
14.3.2. Record of Analyses 26
14.3.3. Preps Performed Entry 27
14.3.4. Reporting Results 27
15. Sample Handling 27
15.1. Sample Collection 27
15.1.1. Sampling Procedures 27
15.1.2. Bottle Preservation 28
15.1.3. Holding Time 29
15.1.4. Turnaround Time 29
15.2. Sample Custody 29
15.2.1. Chain of Custody Form 29
15.2.2. Sample Receipt 30
15.2.3. Acceptance/Rejection Criteria 30
15.2.4. Sample Identification and Control 31
15.2.5. Sample Storage 31
15.3. Subcontracted Analyses 32
16. Laboratory Facilities and Equipment 32
16.1. General Description 32
16.2. Security 32
16.3. Laboratory Accommodations 33
16.4. Waste Management 33
16.5. Pollution Prevention 33
16.6. Deionized Water 33
16.7. Gas Storage 34
16.8. Instrumentation 34
16.9. Instrument Maintenance 34
16.10. Instrument Calibration 35
17. Data Verification Procedures 35
17.1. Data Quality Objectives 35
17.1.1. Completeness 35
17.1.2. Accuracy 35
17.1.3. Precision 35
17.1.4. Traceability 36
17.2. Laboratory Quality Control Checks 36
17.2.1. Method Blank 36
17.2.2. Reagent Blank 36

17.2.3. Field Blank 36
17.2.4. Trip Blank 36
17.2.5. Refrigerator / Storage Blank 36
17.2.6. Quality Control Reference Sample or Calibration Verification Standard (Second Source Standard) 37
17.2.7. Laboratory Control Sample 37
17.2.8. Surrogate Standards 37
17.2.9. Duplicate 37
17.2.10. Matrix Spike/Matrix Spike Duplicate 37
17.3. Proficiency Testing (PT) Samples 37
17.4. Quality Control Charts 38
17.5. Method Detection Limits 39
17.6. Data Reduction, Validation and Reporting 39
17.7. Data Deliverable Reporting 40
18. Corrective Actions and Feedback 41
18.1. Preventive Action 42
19. Departure From Procedure 42
20. Complaints 42
21. Confidentiality, Proprietary Rights, and Transfer of Ownership 43
21.1. Confidentiality and Reporting 43
21.2. Limitation of Liability 43
21.3. Transfer of Ownership 43
22. Audit and Review 43
22.1. Audits from Regulatory Agencies 43
22.2. Internal Audits 43
22.3. Management Quality Review 44
22.4. Audit Response 44
23. Uncertainty of Measurement 45
23.1. Measurement Uncertainty Calculation for Reporting 45
Appendix A – ALSI Organizational Chart 46
Appendix B – Container, Preservation, Storage, and Holding Times 47
Appendix C – Chain Of Custody Record 57
Appendix D – Laboratory Floor Plans 58
Appendix E – Instrument List 60
Appendix F – Change History Form 65
Figure 1 – Reagent Logbook 66
Figure 2 – Quality Verification Data Form 67
Figure 3 – Proposal Request Form 68
Figure 4 – Preservation Logbook 71
Figure 5 – Corrective Action Form 72
Figure 6 – LIMS Internal Chain of Custody 73
Figure 7 – Sample Non Conformance Report 74
Figure 8 – External Complaint Form 75
Figure 9 – Internal Complaint Form 76

3. Quality Assurance Program Plan Identification Form

Document Title:	Quality Assurance Plan
Organization Title:	Analytical Laboratory Services, Inc.
Organization Unit:	Environmental
Address:	34 Dogwood Lane Middletown, PA 17057
Phone:	(717) 944-5541 (717) 944-1430 fax

4. Quality Policy and Objectives

The main objective of Analytical Laboratory Services, Inc. (ALSI) is to provide our clients with high quality laboratory services. We are dedicated to providing our clients with analytical data and services that conform to ISO Guide 17025 requirements. This Quality Assurance Plan details facilities, personnel and equipment necessary for accomplishing this objective along with general procedures and practices, which will be followed to maintain adherence to the objective.

There is a firm commitment from all members of this laboratory to follow a comprehensive Quality Assurance Plan (QAP). This commitment and dedication to quality is fully supported from management to the bench level in order to meet the objectives of our analytical laboratory and best serve our clients. The following Quality Assurance Plan is an embodiment of the current practices of quality assurance/quality control implemented by Analytical Laboratory Services, Inc. to ensure the production of accurate, consistent data of known quality. The stated policies and procedures meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

ALSI's approach to Quality Assurance starts with the President who delineates policy and sets goals in conjunction with senior management personnel. Management staff and laboratory personnel implement policies. Each department assists in the process by providing assessment of operating procedures along with recommendations for improvements or corrections.

Secondly, a Quality Assurance Manager, who reports directly to the President, oversees prevention, assessment and control procedures for the analytical laboratory and various associated departments within the organization. These three functions, prevention, assessment and control, comprise the foundation of the laboratory's approach to Quality Assurance.

In addition to implementing the policies and practices established in the QAP, each laboratory section is responsible for keeping an updated version of Standard Operating Procedures (SOPs) applicable to their section to ensure continuity of analysis throughout the laboratory. Specifics in the areas such as sample handling, instrument calibration, quality control measures, data acquisition and data processing are thoroughly outlined and explained in each SOP. By continuously updating and following the guidelines stated in the QAP and SOPs, the laboratory is able to generate data of consistently high quality.

Existing in conjunction with the quality program at ALSI is a comprehensive ethics program. This ensures the prevention of data quality compromise. Each employee is expected to maintain a firm commitment to comply with all applicable laws and regulations, and to conduct business in accordance with the letter, spirit, and intent of all relevant laws and to refrain from any illegal, dishonest, or unethical conduct. ALSI's business and ethics policy is outlined in the employee handbook and ALSI Ethics Credo that each employee must read and agree to as a condition of his or her initial employment. Documentation that each employee has read and understands the significance of the Ethics Credo becomes part of an individual's training records. Thereafter, on an annual basis each employee is required to review the Ethics Credo and signs off on the Employee Training History form as part of their permanent training record.

The policies and practices of quality assurance/quality control presented in the following text are set forth as minimums. Any additional measures that a project or investigation requires can be incorporated into the project specific Quality Assurance Project Plan (QAPP).

Separate Microbiological and Field Sampling Quality Assurance Plans have been established. These plans should be referred to for the specific quality control procedures required for the microbiology and field services departments.

5. Organization and Management Structure

5.1. Ownership

Analytical Laboratory Services, Inc. (ALSI) is incorporated under the laws of the State of Pennsylvania and is privately owned.

5.2. Organizational Structure of Personnel

An organizational chart depicting the corporate structure at ALSI is provided in Appendix A.

5.3. Job Descriptions

5.3.1. President

Responsibilities:

- Insure commitment to compliance with the standards as stated in ISO17025.
- Direct the day-to-day operations and management of the laboratory.
- Guide Sales & Marketing activities.
- Provide senior management with timely information about business matters, which include financial, sales, marketing, personnel, capital expenditures, safety, and quality issues.
- Guarantee quality in the services provided by the laboratory.
- Insure the financial health of the laboratory.

Requirements:

- Education: A four-year college degree from an accredited educational institution or equivalent.

Experience:

- A minimum of five years nonacademic experience.

5.3.2. Technical Director and LIMS Administrator I

Reports To:

- President

Responsibilities:

- Insure commitment to compliance with the standards as stated in ISO 17025.
- Oversees the day-to-day management of the Laboratory Information Management Systems (LIMS).
- Maintains overall responsibility for technical operations
- Ensure that a proper training program exists for chemists and technicians.
- Recommend analytical improvements and innovations.

Requirements:

- Education: A four-year college degree from an accredited educational institution in chemistry or a related science or equivalent.

Experience:

- A minimum of three years nonacademic laboratory experience.

Deputy:

- In the absence of the Technical Director, the Laboratory Manager and LIMS Administrator II will share the above responsibilities.

5.3.3. Laboratory Manager

Reports To:

- President

Responsibilities:

- Insure commitment to compliance with the standards as stated in ISO17025.
- Maintain overall responsibility for organic and inorganic technical operations.
- Recruit, hire, and train a sufficient number of qualified personnel.
- Ensure that a proper training program exists for chemists and technicians.
- Requisition supplies and equipment necessary to complete required tasks.
- Recommend analytical improvements and innovation.
- Respond to corrective actions recommended by the QA Manager.
- Maintains day-to-day supervision of laboratory operations.
- Monitors standards of performance in Quality Control/Quality Assurance.
- Monitors validity of the analyses performed and data generated to assure reliable data.

Requirements:

- Education: A four-year college degree from an accredited educational institution in the chemical, environmental, biological sciences, physical sciences or engineering, with at least 24 credit hours in chemistry.

Experience:

- A minimum of two years experience in the environmental analysis of organic and inorganic analyses for which the laboratory maintains accreditation.

Deputy:

- In the absence of the Laboratory Manager, the President will assume the above responsibilities.

5.3.4. Quality Assurance Manager

The Quality Assurance Manager operates independently of all data generating areas.

Reports To:

- President

Responsibilities:

- Insure commitment to compliance with the standards as stated in ISO17025.
- Serve as the focal point for QA/QC in the laboratory.
- Maintain an adequate and current quality assurance plan.
- Monitor the QA program as documented in the QA plan and ensure all elements are carried out as written.

- Conduct scheduled and unscheduled audits and inspections and report findings to management.
- Ensure that current SOPs are available for all methods and that they conform to recognized standards.
- Prepare project specific quality assurance plans and data deliverable packages as needed.
- Coordinate performance evaluation and proficiency testing samples.
- Introduce "blind" PE samples.
- Maintain a record of deficiencies or "out of control" events and any corrective actions taken.
- Oversee and/or review quality control data.
- Evaluate data objectively and perform assessments without outside (e.g., managerial) influence.

Requirements

- Education: A four-year college degree from an accredited educational institution in a basic or applied science or equivalent.

Experience:

- A minimum of one-year nonacademic laboratory experience.
- The Quality Assurance Manager shall have a general knowledge of the analytical methods for which data review is performed.

Deputy:

- In the absence of the Quality Assurance Manager, the Laboratory Manager will assume the above responsibilities.

5.3.5. LIMS Administrator II

Reports To:

- Technical Director

Responsibilities:

- Direct the day-to-day operations of the Laboratory Information Management Systems (LIMS).
- Train users on LIMS.
- Develop reports to be used in LIMS.
- Develop upgrades as needed.

Requirements:

- Education: A four-year college degree from an accredited educational institution in Chemistry or a related science or equivalent.

Experience:

- A minimum of two years nonacademic laboratory experience.
- A minimum of two years as LIMS Administrator.

5.3.6. Laboratory Supervisor

Reports:

- Laboratory Manager

Responsibilities:

- Review all technical information that originates in their department.
- Ensure that analysts perform QC checks at required intervals and that all required criteria are met.
- Responsible for documenting all analytical and operational activities in their departments.
- Train analysts working in their department and maintain all documentation to assure their training and competency.
- Prepare and update SOPs.
- Ensure that method detection limit studies are performed on an annual basis.

Requirement:

- Education: A bachelor's degree in a relevant scientific field or equivalent experience.

Experience:

- A minimum of two years of nonacademic experience in relevant analyses.
- A supervisor shall be familiar with relevant test methods and associated calibrations.

5.3.7. Laboratory Analyst

Reports To:

- Laboratory Supervisor

Responsibilities:

- Follow appropriate methodologies, running QC checks as required.
- Evaluate the results of QC samples.
- Identify and report quality problems to the supervisor or QA Manager.
- Document activities and report results in a concise and accurate manner.
- Supervise a small group of technicians engaged in performing routine laboratory and fieldwork.

Requirements:

- Education: A bachelor's degree in a relevant scientific field or equivalent experience.

5.3.8. Laboratory Technician

Reports To:

- Laboratory Supervisor

Responsibilities:

- Follow appropriate methodologies, running QC checks as required.
- Evaluate the results of QC samples.
- Identify and report quality problems to the supervisor or QA Coordinator.
- Document activities and report results in a concise and accurate manner.

Requirements:

- Education: High School graduate or equivalent. Two years of education in physical or environmental science and/or two or more years work experience in a testing laboratory.

5.3.9. Laboratory Assistant

Reports To:

- Laboratory Supervisor

Responsibilities:

- Follow appropriate methodologies, running QC checks as required.
- Document activities and report results in a concise and accurate manner.

Requirements:

- Education: High school graduate or equivalent

5.4. Safety

ALSI is conscious of providing a safe and healthy work place for its employees. This is accomplished by adequate safety training for all personnel. Employees receive all the necessary safety training and information to meet the guidelines established by the Occupational Safety and Health Administration (OSHA). ALSI fully complies with the "Right-to-Know" laws established by the federal government.

ALSI's safety committee is responsible for maintaining the Material Safety Data Sheets (MSDS) within the laboratory and organizing continued safety training for all employees. The safety committee performs inspections of the facilities for compliance with safety regulations and verifies that all safety equipment is in good working condition.

It is ALSI's policy that safety glasses or splash goggles and lab coats be worn by every person entering designated laboratory areas and no food or beverages are allowed to be brought into these areas. All accidents that occur in the laboratory that involve personal injury or that could potentially have involved personal injury are reported on OSHA Form 301. The pertinent information pertaining to the incident is maintained on the form. This includes the date that the incident occurred, the person(s) involved, details of the incident, and information about the physician or health care professional, if required. A record of these reports is kept by the safety committee and summarized annually on OSHA Form 300. This summary form is posted on the Health and Safety bulletin board during the month of April each year.

ALSI maintains a Chemical Hygiene Plan (CHP), which details the safety policies of the laboratory. This manual is read and signed-off as understood by all employees following their initial orientation. These records can be found in the individual's training records maintained by the QC department.

6. Training

6.1. Personnel Files

The Controller maintains a file for each individual. Annual reviews of technical competence, achievements, and problems noted shall be maintained in this file.

6.2. Technical Training

All employees involved with the handling of samples, manipulation or generation of data or operation and maintenance of equipment used in sample processing must maintain training records. Training records are maintained in the QA Department. Evidence of Ethics Training, short courses or seminars attended along with a current resume are also kept on file in the Quality Assurance Department. Any accreditation or certification maintained by an individual, who affords the laboratory certification, shall also be stored in these training files. Demonstration that each employee has read, understands, and is using the latest version of the laboratory's in-house quality manual can be found in these files on the *Employee Training History Form.* Instructions on required training documentation are included in ALSI's Standard Operating

Procedure, 99-Train. Initial Demonstration of Capability as well as annual Demonstration of Continual Proficiency including certification that technical personnel have read, understood, and agreed to perform the most recent version of the SOP are required to be documented and to be approved by the Laboratory Manager and the QA Manager. Other measures of proficiency included in these logs will be reviewed and signed by an appropriate supervisor at regular intervals. If an employee terminates his/her employment, their training records will be archived for a minimum of seven (7) years. All employees are required to review their training records on a regular basis, and make sure they are up to date and properly archived.

The training required to perform independently within the organization varies considerably from task to task. Ultimately, the personal judgment of an employee's direct supervisor shall determine his or her ability to work alone. Demonstration of proficiency as well as training with and observation by competent co-workers or supervisors is the foundation of the training program. Documentation of training and evaluation of measures of proficiency are essential to the training program.

Once an employee has demonstrated acceptable proficiency as described, it is implied that he or she has also demonstrated a minimum level of qualification, experience, and skills necessary for working in the lab. This may include, but is not limited to, basic lab skills such as using a balance, using appropriate glassware, and using qualitative and quantitative techniques

6.3. Method Specific Training

An analyst or technician in training may perform work on samples submitted for analysis as long as they have demonstrated the ability to produce reliable results through the analysis of a proficiency testing sample or in-house quality control samples, and a supervisor or designee is available in the work area when preparing or analyzing samples. When Initial Demonstration of Capability is demonstrated, the SOP has been reviewed and sufficient training is documented in the individual's training records, the employee may work independently.

6.4. Minimum Experience and Training

In addition to the requirements listed in sections 5.3.1 through 5.3.8 persons hired for the following job functions must meet the following minimum experience and training requirements before working independently in these areas, unless a supervisor or designee is available for consultation.

- Inductively coupled plasma-emission (ICP) spectroscopy: One year experience with satisfactory completion of a short course on ICP or equivalent in-house training.
- Flameless atomic absorption spectroscopy: One year experience with satisfactory completion of a short course on graphite furnace atomic absorption (GFAA) or equivalent in-house training.
- Flame atomic absorption (FLAA) spectroscopy: One year experience with satisfactory completion of a short course on FLAA or equivalent in-house training.
- Gas chromatography: One year experience with satisfactory completion of a short course on basic GC or equivalent in-house training.
- Mass spectrometry: One year experience with satisfactory completion of a vendor's training course, professional sponsored short course or equivalent in-house training.
- Mass spectra interpretation: One year experience with satisfactory completion of a vendor's training course, professional sponsored short course or equivalent in-house training.
- General chemistry and instrumentation: Six months experience.
- Sample collection: Six months experience.

6.5. Organizational Chart

See Appendix A for ALSI's Organizational Chart by Position.

7. Document Control

Record keeping is extremely critical in an environmental laboratory to assure the validity of the data produced. ALSI produces two types of records: computerized records and hardcopy records. The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data.

7.1. Computer Records

Computerized records include those generated by the entry of information into the Laboratory Information Management System (LIMS). Information entered into the laboratory computer system is saved daily on hot swappable media. This is accomplished through a "back-up" which is performed each night. Back-ups are kept in a fireproof safe located in the field sampling building, which is not attached to the main building. A member of the IT Department may initiate retrieval and printing of these records.

7.2. Hardcopy Records

Hardcopy records produced by the laboratory are a combination of forms, reports and logbooks. Chain of custody, carrier receipts, cooler receipt checklists, and other project or sample information forms along with Lab Analysis Reports are stored in the administrative office. These are stored in filing cabinets by client name. Each ALSI client has an individual file in which the chain of custody forms and Lab Analysis Reports for all samples originating from that client are kept. Within this file, the records are separated by type (e.g., all chain of custody information is filed together and all laboratory analysis reports are stored together) and are placed in numerical order of the laboratory sample numbers. At the end of each calendar year, the previous year's records are moved from this office to an off-site storage warehouse where the test records shall be protected from loss, damage, misuse, or deterioration. The records are kept for a minimum of seven (7) years in this area filed alphabetically and chronologically in order to permit retrieval when required. Access to archived information is controlled and documented with an access log filed with the document storage facility. Drinking water data for Public Water Systems will be kept for ten (10) or twelve (12) years before disposing of records. Data, which are expected to become part of a legal action, may be maintained for a longer period of time dependent on legal counsel. Filing, copying and maintaining legible hardcopy records is the responsibility of the Customer Service Representatives and the Department Supervisors.

7.3. Laboratory Logbooks

Laboratory logbooks are bound, sequentially numbered documents used to record specific information related to sample receipt, preparation, and analysis. This information may include, but is not limited to, preservation records, instrument maintenance, sample measurement data, etc. All laboratory logbooks are assigned and labeled with a unique identification number before being placed into use. This provides a system for direct document control and tracking. The supervisor of each department is responsible for the storage and retention of any logbooks used in their department.

Laboratory logbooks are kept in the laboratories until filled or no longer in use. Once a logbook is "retired" or no longer in use, it is also placed into storage. This includes the logbooks assigned to individual analysts, those in which instrument maintenance, calibrations, etc. were recorded, reference material notebooks, sample preservation notebooks and all other bound materials generated by the laboratory. Laboratory logbooks are kept for a minimum of seven (7) years by the laboratory.

7.4. Laboratory Worksheets

Laboratory worksheets are forms used to document information relating to laboratory sample analysis processes. The forms vary and are used throughout the laboratory. Each form is controlled by the

quality control department and tracked by assigning each a dated revision number. This ensures that any changes made to the forms are documented, and each revision is appropriately dated.

7.5. Raw Data

Raw instrument data are generated daily in each department of the laboratory. Raw data include instrument printouts used to calculate and report sample data. All raw data generated is stored in each individual department for a period of time judged sufficient by the supervisor of that department. The data is then transferred to labeled file boxes and stored in locked storage for a minimum of seven (7) years from generation.

7.6. Data Deliverable Packages

All Deliverable Packages are reviewed and validated by the Quality Control Department prior to submission to the client. The client receives the original and a CD Rom of all raw data, quality control summary forms, and associated shipping documents. A copy of the original data package is retained by ALSI in electronic form only.

7.7. Quality Assurance Documents

Control of quality assurance documents is to be maintained by the Quality Assurance Department. Quality assurance plans, standard operating procedures and other controlled documents are subject to the following requirements.

1. All ALSI Quality Assurance Plans must be signed and approved by either the President, Technical Director, Lab Manager, and the Quality Assurance Manager or designee.

2. All ALSI Standard Operating Procedures (SOPs) must be signed and approved by the Quality Assurance Manager and a Validator or designee.

3. All ALSI QA documents and SOPs must have document control information placed in the upper right hand corner of each document page. The information required is the document name, revision number, date generated, and page number. (See upper right hand corner of this page for format.) The first page of the document will then be stamped with an “effective date”. The document name of an SOP will consist of the ALSI department number and a unique code to be determined by the department supervisor. For example, a hexavalent chromium SOP, written by the Wet Chemistry Department, may have the name 04-CR+6.

4. All ALSI QA documents and SOPs must have an Identification Form at the beginning of the document. The Identification Form will contain the document title, the document control number, the organization title and the appropriate signatures required for approval.

5. Reproduction and distribution of controlled documents are to be handled by the Quality Assurance Department. The original documents will be filed in the Quality Assurance Department. Documents to be distributed will be given a control number and will be listed in the Controlled Document Logbook. Employees will then sign and date the logbook when they receive a controlled document. Every time a controlled copy is produced from a master document the following stamp is used to mark all pages of the document:

CONTROLLED DOCUMENT
DO NOT DUPLICATE
If this stamp is not colored red, this is not a controlled copy

6. When a document has been revised, the old revision will be collected, the date returned will be recorded in the logbook and the new revision will be issued following the same procedure. The original copy of the superseded document will be kept in the Quality Assurance office and will be stamped "SUPERSEDED BY REVISION", directly on the sleeve and on the cover page of the SOP, with the new revision number written in. All other copies will be disposed of.

7. Any employee receiving a controlled document will be responsible for that document and the information contained in that document. Employees need to assure its safekeeping. If a controlled document is lost or damaged, the Quality Assurance Manager shall be notified immediately so it can be noted in the controlled document logbook and a new document issued.

8. All employees receive and maintain responsibility for a controlled copy of the most recent revision of the QA Plan.

9. Laboratory supervisors are responsible for reviewing SOPs annually for revision as required (i.e. methods are updated, new instrumentation, etc.). Revisions of SOPs must be submitted to the Quality Control Department for validation and approval before they are put into use.

10. If it is necessary to make a change in a controlled document before a new revision is created, the change shall be entered by hand in the document by a supervisory or managerial employee, and the change initialed and dated. All controlled copies of that document shall be changed in this manner. The document shall then be submitted to the Quality Assurance Department for revision.

7.8. External Documents

The Laboratory has established and maintains control of all external documents that form part of its quality system. These external documents include sources such as regulations, standards, test methods, accreditations/certifications, and are kept in the Quality Assurance Department for use by all laboratory personnel.

A listing of all reference procedures is given in Section 14.1 of this Quality Assurance Plan. The current versions are maintained by regularly checking Federal and State websites for the latest updates. A list of all currently controlled documents is available and updated as these revisions are implemented within the laboratory.

Originals for all accreditations/certifications are located in the reception area of the laboratory. This information can also be found on our website www.analyticallab.com.

Documents determined to be outdated, but retained for knowledge preservation will be marked as "obsolete". References removed from the Quality Assurance Department, for use by laboratory personnel, must be signed-out in the *Reference Library Logbook* and signed back in upon its return.

8. Traceability of Measurement

The laboratory has established programs for the calibration, verification and maintenance of its measuring and testing equipment. This includes balances, thermometers and reference standards. Calibration of all laboratory balances is performed annually and contracted to Mettler-Toledo, Inc., 1900 Polaris Parkway, Columbus, OH 43240. Calibration of all Class "1" weights is performed every five years and contracted to Troemner, 201 Wolf Drive, Thorofare, NJ, 08086-0087. Calibration of all in-house thermometers is performed annually against a NIST certified thermometer. The calibration of the NIST certified thermometer is performed every three years by ICL Calibration Laboratories, Inc., 1501 Decker Avenue, Stuart, FL 34994.

All certificates of calibration are kept on file in the QA Department for review by management and all regulatory agencies.

8.1. Reagents

Reagent quality is of extreme importance to laboratory results. All chemicals and reagents used in the laboratory meet purity and traceability requirements specified in the individual methods. Each laboratory area has standard operating procedures, which define the quality of reagents being used. It is the responsibility of the supervisor of each area to requisition reagents of required quality. All purchased reagents are delivered to the Sample Receipt Area to be unpacked, verified for accuracy against the invoice, and labeled with a unique reference ID number. This reference ID number is then recorded in the Chemical Reagent Logbook (See Figure 1) along with the chemical name, manufacturer, lot number, date received, receiver's initials, expiration date, number of bottles, and storage location. The storage of chemicals is governed by the manufacturer's recommendations and by the analytical procedures for which the chemicals are used. Purchased reagents are to be labeled with the reference ID number, date opened, expiration date, and initials of the analyst opening the reagent. For solid stock chemicals that do not list a supplier expiration date, ALSI will label these chemicals with an expiration date of 5 years from the date of receipt.

Verification that the chemical or reagent purchased is of the correct purity and traceability before being put into use is the responsibility of the supervisor of the department in which the reagent will be used. The preparation of working reagents is recorded in bound logbooks. These logbooks document the name of the reagent, reference ID number, and the concentration of the reagent, the reference number(s) of the stock reagent(s) used as well as the dilutions performed, date of preparation, date of expiration, and initials of the preparer. The container holding the working reagent is labeled with the reference ID number, initials of the preparer, the date of preparation, and the expiration date as determined by the method. Any health and/or safety concerns are also listed on the container.

Each lot of chemical or reagent used is monitored and controlled for any unusual contaminants that interfere with analysis as evident in results of prescreens and/or method and reagent blanks. If a working reagent is found to be suspect, it is removed from use and traced back to the original lot number, which is then investigated. If the stock reagent is found to be the source of the problem, it is completely removed from use. Any samples contained in batches in which the suspect reagent was used for analysis will be reanalyzed if sufficient remaining sample allows, or flagged with a comment on the report. The corrective action process described in Section 18.0 of this manual is used to document these assessments.

8.2. Reference Standards

Reference standards are used for calibration and calibration verification in all analyses requiring comparison to a chemical substance. The reference standards used are those specified in the reagent sections of the respective standard operating procedures. Wherever available, reference standards are

traceable to national standards of measurement. If NIST (National Institute of Standards and Technology) traceable standards are not available, reference standards of the best purity and quality from a reputable supplier are procured by the supervisors of each department by careful study and consideration of the chemically pure substances available. It is the responsibility of the supervisor of each area to requisition reference standards of required quality appropriate for the analysis being performed and to provide correlation of results per NELAC standards. All purchased reference standards are delivered to the Sample Receipt Area to be unpacked, verified for accuracy against the invoice. The standards are then delivered to the individual departments along with their respective Certificates of Analysis provided by the supplier, where the supervisor verifies that the correct standards have been received. Certificates of Analysis are kept on file for all reference standards within the respective departments. Records are maintained for each stock reference standard used in the laboratory including the identity, a unique reference ID number, dates received, lot number, and supplier. Each reference standard will itself be labeled with the reference ID number, the date received, the date opened, and the initials of the analyst opening the standard, or will be labeled with the reference ID number allowing the traceability to these items.

Working standards are prepared using quality reagents as outlined in 8.1, reference standards, Class A volumetric glassware, and properly calibrated laboratory equipment such as balances and pipettors. Bound logbooks are maintained indicating the reference ID number of the working standard, the concentration, the reference ID number(s) of the stock standard(s) used, the dilution performed, the matrix, the preparation and expiration date, and initials of the analyst preparing the standard. All prepared standards will be properly labeled with the reference ID number, the date of preparation, the preparer's initials and the expiration date of the standard, or will be labeled with the reference ID number allowing the traceability to these items. Any health and safety concerns shall also be noted on the label. This documentation allows standard traceability back to the original Certificate of Analysis.

Reference standards from an independent source are used in all methods for calibration verification. They are purchased and prepared separately from a source independent of the calibration standards. If not available from a reliable second source, they are prepared separately from a different lot than that of the calibration standards. These are used to verify and control the accuracy of the working calibration standards before client samples are analyzed. Acceptance criteria are set per method requirements for the calibration verification standards, and are listed in each respective standard operating procedure. Calibration verification standards falling outside of the acceptance criteria result in the analysis being discontinued until the cause can be investigated and corrected.

Reference standards used to spike samples before a preparative process, such as surrogate and matrix spike solutions used during extractions, are subjected to a critical solution quality verification procedure prior to use. This ensures that the standards are of acceptable quality before extraction is performed. This procedure consists of preparing organic surrogate and spiking solutions pursuant to the method required, and aliquoting a portion of each lot into a separate analysis vial. The formulated standard is then placed in a holding area to await approval, and the aliquot is delivered to the appropriate analytical department for testing. Once the analytical department grants approval, as indicated by a completed Quality Verification Data Form (see Figure 2), the solutions may be removed from the holding area and placed into use for extraction of samples. If approval is denied due to insufficient quality, all portions of the lot of surrogate or spiking solution are discarded. Verification that the standard is the correct concentration and of the correct purity before use in extracting samples is the responsibility of the supervisor of the department in which the analytical testing is being performed.

8.3. Removal of Expired Chemicals and Standards

Each laboratory group manages its own inventory for expiration date. Any chemical, reagent, solution or standard, which is past its expiration date, and cannot be verified for reliability, is removed from service

and placed in the laboratory's internal waste management system. Expired standards or chemicals will not be used for analyzing samples unless the laboratory verifies its reliability. Verification records shall be kept with the Supplier's Certificate of analysis and retained for review by local, state and federal agencies. Any expired standard, which is retained for training purposes, or as a reference material must be clearly marked, "Not for Instrument Calibration – Expired Standard" or "Not for Analytical Use – Expired Standard."

8.4. Purchased Materials

ALSI routinely purchases consumable items for the laboratory through the laboratory-purchasing agent. The department supervisors are responsible for selecting all items for laboratory use as specified in the individual method SOPs. All items shall be of the required quality as dictated in these SOPs. The Laboratory Manager reviews all *purchase requisitions* before the orders are placed. The supervisors will submit all pertinent information on a purchase order to the purchasing agent. The purchasing agent is responsible for placing the order with the appropriate vendor and assuring that items are received intact in the time period requested by the department supervisor. It is possible for a department supervisor to place an order directly with the appropriate vendor in cases where an item is needed in short periods of time and the purchasing agent and Laboratory Manager is not available to approve and place the order. Records are documented for all items ordered by the laboratory. Those from the current year are filed in the administrative office, while those from previous years are filed in locked storage for a minimum of seven (7) years.

All purchased items are delivered to a Sample Receipt Area to be unpacked, verified for accuracy against the invoice, and inspected for breakage. Any unacceptable item is returned to the purchasing agent to be sent back to the vendor in return for the correct, intact item. Items that are common to all the departments and those received in large quantities, such as sample collection bottles (8.4.2), are stored in the locked storage room until use. The Sample Receipt Custodian may initial the invoice for these items. Other items are delivered directly to the department that submitted the order for initialing of the invoice and storage. It is the responsibility of the department supervisor to insure that the items received are of the required quality. Any certificates received for goods and services that have a direct influence on quality are saved and filed for reference.

The same consumable items used in the routine analysis of samples, such as test tubes, Pasteur pipettes, etc., are also used in the preparation and analysis of calibration standards and control checks. If the quality of results for the calibration standards and control checks is within the acceptance criteria outlined in the method, then the consumable items are considered to be of acceptable quality to perform the analysis.

8.4.1. Volumetric Glassware

Volumetric glassware, including pipettes, graduated cylinders, and flasks, which influence the accuracy or validity of calibrations or tests, may be subject to a calibration check before being put into use throughout the laboratory. Volumetric glassware is verified gravimetrically and controlled using controlled tolerances as acceptance criteria. The measurements are documented with the date and analyst's initials, and maintained in the QA office.

8.4.2. Sample Collection Bottles

ALSI provides appropriate containers for sample collection. See Appendix B for a listing of the appropriate sample containers available for each method. All bottles are disposed of after a single sampling.

ALSI purchases pre-cleaned bottles for various analyses. These are cleaned in accordance with EPA specifications. Careful inspection and comparison of the bottles received against the invoice is performed to assure that the quality of the bottles is that which was ordered. Bottles used for microbiological samples are subject to a documented sterility check for every batch of the same lot number. This check must meet acceptance criteria described in the same document before the bottles can be used for sample collection. All bottles are used only once, and are disposed of.

Bottles used in the analysis of samples are also used in the periodic analysis of field and trip blanks. The department supervisors monitor results of field and trip blanks at the time of department approval. If a field or trip blank is found to have positive results, and contamination from the bottle is suspected to be the cause, the corrective action process described in Section 18.0 of this manual is followed.

8.5. Balances

ALSI maintains multiple balances for analytical measurements throughout the laboratory. Calibration checks are performed and documented on each day of use for each balance. This calibration check consists of measurement of two or more NBS Class 1 weight reference standards, which are used for calibration only, and for no other purpose, and comparison to acceptance criteria listed in the associated balance calibration standard operating procedure.

Each balance is serviced and calibrated over the range of use annually by a manufacture's certified representative. Balance Calibration Reports and Certificates of Weight Traceability are kept on file identifying traceability of test weights used to the National Institute of Standards and Technology. NBS Class 1 weights are returned to a certified calibration laboratory at least every five years for re-certification.

9. Scope of Tests

Unless superseded by client or project requirements, analysis of samples is performed using EPA or EPA-approved methods. For those analyses that do not have EPA-approved methods, the analytical methods used are taken from standard sources, where such methods exist (ASTM, API, etc.). The standard analytical methodologies performed by ALSI can be found in Appendix B along with the recommended container, preservation, storage and holding times for each methodology.

ALSI will not accept samples that require test methods that have not been published in national standards, such as those listed in Section 14.0 of this document, unless these methods have been agreed upon by and made available to the client upon request, and have been documented and validated by the laboratory. It is the responsibilities of the laboratory management to determine which validation studies are appropriate for a given analysis on a case-by-case basis. The effectiveness of a non-standard analytical method is then evaluated and approved by laboratory management as determined by the results of the validation studies performed. Examples of validation studies demonstrating effectiveness are reference standard analysis, blank studies, MDL studies, precision and accuracy studies, determination of calibration range, or proficiency sample evaluation. These studies will be made available to the client and any recipients of the reports upon request. Non-standard methods are clearly indicated on Laboratory Analysis Reports as modified or in-house methods.

10. Review of Solicitation, Offer or Contract

Upon receipt of a formal solicitation, offer or contract—also referred to as a Request for Proposal (RFP), Request for Quotation (RFQ), or similar document—containing items such as contractual language, specific QAPP information, target compound lists, delivery schedules, the document (when bid is issued) is assigned an ALSI quote number. The proposal staff—the President, Technical Director, Laboratory Manager, National Accounts Manager, Account Executive, and/or Project Coordinators—are all capable of

completing a solicitation, offer or contract. The proposal staff will review pertinent pieces for content and requirements. The President, Technical Director and/or Laboratory Manager will be involved with the technical review for method specific or client specific analytical requirements to complete the designated turnaround schedules and work plan as requested within the solicitation, offer or contract. As part of the review process the QC Manager will include the current accreditation status of the laboratory per the project requirements. If accreditation does not exist, or cannot be completed prior to the project start date the request for solicitation, offer or contract will be refused by ALSI. Additionally, if during the course of the contract the laboratory is subjected to suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation this information will be immediately communicated to the client.

The Sales Administrative Assistant is responsible for supporting the proposal staff in generating the specific qualifications information and/or promotional information and/or promotional information required within the solicitation, offer or contract.

The President, Technical Director and/or Laboratory Manager reviews the terms & conditions of sale, and the overall requirements of the scope of work for continuity, enforceable penalties, liabilities, and expectations, as indicated in the solicitation, offer or contract.

The Sales Administrative Assistant completes the final solicitation, offer or contract following the collection of the above information and resolution of outstanding questions or issues generated by each group. The proposal staff will follow up with the client after the solicitation, offer or contract is delivered.

10.1. Laboratory Capacity Review

For any solicitations, offers, or contracts generated over $10,000, a review of the equipment and personnel will be performed by the President, Technical Director, National Accounts Manager, and/or Laboratory Manager. A Proposal Request Form will be attached to the front of the proposal with a checklist (see Figure 3). The Proposal Request Form is used to define the project specifications requested by the client. If the laboratory does not have the capabilities to perform the new work, this will be noted on the cover sheet. Actions taken to provide the necessary resources will be documented on the cover sheet or it will be noted that the proposal was not accepted.

10.2. Amendment to Contract

When an amendment is requested to a contract that has been already signed and accepted by both parties and the project has not been initiated, the laboratory will reserve the right to propose adjusted rates and delivery schedules that reflect the “new scope of work.” If the changes in the work do not impact the objectives of the work plan nor necessitate a change in delivery schedule or unit pricing, then the amendment will be adopted and all parties involved will proceed under the amended contract.

An amendment to a contract that necessitates changes in the product to be delivered, turnaround times, sample delivery groups, required labor and equipment to meet objectives may require a negotiation in the unit pricing for the new analytical services package. No work outside the original scope of work will be performed until all outstanding issues and/or discrepancies are cleared between the client and the laboratory.

Amendments during an ongoing project that necessitate pricing adjustments, delivery schedule changes, or other changes to the normal laboratory routine, will be mutually addressed in a timely manner by the client and the laboratory. The laboratory will make all attempts to meet holding times on samples received under the initial contract in “good faith” that all parties will meet with a mutual understanding of the new delivery schedule and associated fees for the change orders.

All affected laboratory departments are participants in the commitments made to an amended scope of work under an amended contract.

11. Demonstrating Method Performance

When new methods are implemented or a client requires a method not routinely performed, there are certain requirements that must be fulfilled prior to performing work to demonstrate adequate method performance.

Methods published in national standards that have not previously been performed at ALSI require a demonstration of method performance that meets or exceeds minimum expectations as published in the method. These may include, but are not limited to, initial demonstration studies required by the method, blank studies, method detection limit studies, precision and accuracy studies, QC check sample performance, and calibration ranges. A standard operating procedure must also be written for the analysis. Once these validation studies have been completed and satisfactory performance has been demonstrated, samples may be analyzed by the new method.

Methods not published in national standards, such as special project procedures or screening methods are also subject to a demonstration of method performance before use in analyzing project samples. It is the responsibility of the laboratory managers to determine which studies are appropriate for a given analysis on a case-by-case basis. Once satisfactory performance has been demonstrated as determined by laboratory management, samples may be analyzed by the new method.

12. Client Services

Client Services is one of the most important and integral parts of the ALSI operation. ALSI's Client Services Department consists of project managers and project coordinators. From initial project set-up to project completion, the Project Managers/Coordinators along with the support of the individual laboratory departments have the greatest impact on the success of a client's environmental monitoring program or project. In conjunction with the success of a program or project, in-house clients or new clients are all going to measure the responsiveness, effectiveness, and overall quality of the laboratory through their interaction with the Project Managers/Coordinators. The Project Managers/ Coordinators are the laboratory's interface with the client and a major key to continuing a future relationship with them.

The ALSI Client Services Department is under the direction of the President. The Supervisor of the Client Services Department has the responsibility of overseeing the Sample Receiving Department, including bottle preparation, and the direct day-to-day supervision of all project managers. All of these functions relate directly to the initial set-up of an environmental monitoring program or project, initiation of ALSI's services, sample receipt, and project follow-up support.

The Project Manager/Coordinator's responsibilities include routine requests for coordinating technical support from new or established clients, generation of orders for bottle preparation to the Sample Receiving Department, project set-up and document review, project status requests, maintenance of pertinent discussions with clients relating to sample analysis requirements, and project follow-up upon completion of the analytical tasks.

Client Services is a major key to the laboratory's success. The Client Service Department interrelates with internal and external clients on a daily basis and without them; the laboratory would not be able to operate in an efficient manner in order to meet the demanding client requests and their ongoing projects or programs.

12.1. Project Planning

ALSI has been in existence since 1979. Standard Operating Procedures (SOPs) and required instrumentation have been continually purchased and brought online in response to markets created throughout this time period. Tests and associated instrumentation are for commercial application, and therefore, most of the work that is brought to the facility is to be completed following standardized EPA procedures that have been incorporated into the ALSI's SOPs.

The President, National Account Executive, Technical Director, and Laboratory Manager are responsible for the type and quantity of work that is accepted and moreover promoted by ALSI. Solicitations, offers, contracts and/or specific client requests that are extraordinary are reviewed by the President, Technical Director and/or Laboratory Manager as to the viability of the program and the ability of the laboratory and associated personnel to successfully complete the objective of the scope of work. Subcontracting, MDL studies for non-standardized compounds, and scheduling adjustments may all come about as a result of each project's inherent requirements. The objective of having prior notification of sample receipt or prior notification of a client's expectations is to have as much time as possible to react and to meet or be able to address the client's project requirements.

Daily work schedules are generated, validated and utilized by the Department Supervisors as they oversee production within their departments. The key components of these schedules are holding times, delivery times and the specific or extraordinary requirements of a sample delivery group. The Laboratory Manager oversees the Department Supervisors who are held accountable for the management of their respective departments.

Client Services assists the Sample Receiving Department in the review and formalization of the tests that will be performed, associated bottleware for the field, labeling, test methodology to be requested, adequate and proper preservation reagents as well as communication to the field. They also communicate within the laboratory regarding sample deliveries and holding times associated with the sample delivery groups.

As the client's samples are logged-in and processed, any discrepancies or issues will then be addressed as soon as possible to Client Services.

12.2. Organizational and Technical Interface

Client Services is the main contact for a project once the first sample delivery group has been received and logged-in by the laboratory. It is Client Services that will be tracking the project's movement, resolving any issues that arise during the sample preparation, analytical steps and final reporting of the data. Project Coordinators are assigned upon contract award.

Large projects with multiple sample delivery groups usually require constant interaction with the field to communicate coordination of supplies and sample movement into the facility. This allows the Project Coordinator to establish the ongoing dialogue needed and the openness of communication that develops between the client and the laboratory. Delays in sample processing due to instrumentation failure, matrix effects, etc. are brought to the client's attention as soon as practically possible once the Department Supervisor has notified Client Services. It is imperative that the Project Coordinator be notified in a timely manner by the laboratory personnel so as to minimize any additional costs and operational impacts in the field.

Scope of Work changes, delays in the field due to equipment failures or weather are all communicated through the Project Coordinator into the laboratory departments that are affected by the change. Work

schedules can then be adjusted and resources can be redirected within the operation for maximization of equipment utilization for the client and their samples.

Delays in fieldwork may require a re-commitment by the laboratory towards the due dates and deadlines that were initially set. The Laboratory Manager and Department Supervisors will then mutually agree upon a new commitment to the client so that all involved can plan proactively for future commitment of all resources.

13. Control of Nonconformance Testing

Ideally, all data inquiries should occur prior to the release of the analytical results to a client. However, if a client should question an analytical result, the analysis must be reviewed thoroughly in order to alleviate the client's concerns as well as to ensure that the laboratory is not suffering from a procedural problem.

If Client Services determines that further corrective action is necessary due to a data inquiry received by a Client it shall be documented on an ALSI Corrective Action Report (CAR) (see Figure 5). This form will contain the general information of the data in question. Following initiation, the appropriate department supervisor will review this inquiry. The supervisor will review all associated raw data regarding the inquiry. Comments and/or resolutions will be described on the CAR. The CAR will then be returned to the Project Coordinator with a copy to the QA Department. The Project Coordinator will inform the client of all pertinent information regarding the data inquiry. If reanalysis is required, the client will be given the approximate date that they can expect reanalysis results.

The QA Department will review the copy of the inquiry and perform follow up regarding quality issues, if required. The CARs are summarized monthly and charted by "cause". These charts are reviewed annually by ALSI management and may effect changes to the laboratory's quality assurance practices.

From the information available a CAR may precipitate a correction of the final results, reanalysis of the client sample and/or additional action by the Quality Control Department. Additional action may include performing an internal audit to determine if the problem occurred due to a non-authorized procedural change requiring method revision and/or additional analyst training.

Additional information regarding control of nonconformance testing can be found in the Standard Operating Procedure 99-Corr A.

14. Test Method Reference

14.1. Analytical Procedure References

ALSI relies primarily upon the most current EPA approved revisions of the references listed below for methodologies used in the laboratory. Procedures contained in these references are acceptable for use only after the lab has demonstrated and documented adequate performance with the method such as method detection limit studies, precision and accuracy studies, proficiency sample analysis, and linear calibration range studies. These studies are then routinely verified as long as the methods are in use in the laboratory.

- "Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, 1979. Revised 1983.

- "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 18th Edition, 19th Edition, 20th Edition.

- "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA SW-846, Third Edition, 1986, Revision 1, July 1992, Update II, September 1994 and Update III, June, 1997.

- 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants.

- 40 CFR Part 141, National Primary Drinking Water Regulations.

- "Methods for the Determination of Organic Compounds in Drinking Water", EPA 600/4-88/039, Rev. July 1991; Supplement I, EPA 600/4-90/020, July 1990; Supplement II, EPA 600/R-92/129, August 1992; Supplement III, EPA-600/R-95/131, August 1995.

- "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA 600/R-93/100, August 1993.

- "Methods for the Determination of Metals in Environmental Samples", Supplement, EPA 600/4-88/039, Rev. July 1991; Supplement I, EPA 600/4-90/020, July 1990; Supplement II, EPA 600/R-92/129, August 1992.

- "NIOSH Manual of Analytical Methods", U.S. Department of Health and Human Services, Fourth Edition, August 1994. First Supplement, May 15, 1996.

- Annual Book of ASTM Standards.

14.2. Standard Operating Procedures

A set of Standard Operating Procedures (SOPs) is available in each of the laboratory areas. The SOPs are comprised of clear, complete written instructions for completing each standard test performed by ALSI. Standard Operating Procedures include the following sections if applicable to the laboratory's direct performance of the method. Otherwise, analysts shall consult the reference method indicated in the Scope and Application Section of each SOP for the sections not included in a particular SOP. Other pertinent sections in addition to the following may be included as necessary.

- **Scope and Application** which includes:
    - Identification of the Test Method
    - Applicable Matrix or Matrices
    - Detection Limit
- **Method Summary**
- **Definitions**
- **Interferences**
- **Safety**
- **Apparatus and Materials**
- **Reagents**
    - Includes any chemicals used in the procedure, including reference standards
- **Calibration**
- **Quality Control** which includes:
    - Data Assessment and Acceptance Criteria for Quality Control Measures
    - Corrective Actions for Out-of-Control or Unacceptable Data
- **Sample Collection, Preservation, and Storage**
- **Procedure**
- **Calculations**
- **Reporting Results**

- **Method Performance**
- **Pollution Prevention**
- **Waste Management**
- **References**
- **Tables, Diagrams, Flowcharts, and Validation Data**
- **SOP Concurrence Form**

All analysts performing a particular method are expected to sign the SOP Concurrence Form, which is kept on file as part of the analyst training record, to indicate that they have read, understood, and agreed to follow this revision of the SOP. Laboratory supervisors are responsible for maintaining and updating SOPs on an annual basis, which are then validated and or reviewed by the Quality Assurance Department before being initiated for use in the laboratory. Archived revisions of Standard Operating Procedures are stored in the Quality Assurance Department for at least seven (7) years.

14.3. Laboratory Analyses

14.3.1. Test Scheduling

Test scheduling is accomplished through the Laboratory Information Management System (LIMS) and is coordinated by the laboratory supervisors. Each of the laboratory departments prints a "Backlog Report" at the beginning of each day or during the day as needed. This report lists the outstanding analyses from each of the departments. Each department supervisor is responsible for monitoring Backlog Reports pertinent to their section, which is used in the scheduling and recording of laboratory analyses. The Backlog Reports are identified as follows:

- VOGC - Gas Chromatography - Purge and Trap
- SVGC - Gas Chromatography - Direct Injection
- HPLC - High Performance Liquid Chromatography
- SVMS – Gas Chromatography/Mass Spectroscopy – Semivolatiles
- META – Metals
- WETC – Wet Chemistry
- MICR – Microbiology
- SUB - Subcontracted Analyses
- PREP – Prep Department
- FLD - Field Analyses

Using the LIMS reports, the supervisors assign tests to the laboratory analysts. The analysts enter the computer system to schedule the analyses. This is done in the "Batching" area of the LIMS. The information entered by the analyst is recorded in the system for use by other laboratory personnel. Anyone using the computer system to inquire on the scheduled samples will see this information.

14.3.2. Record of Analyses

Laboratory records are traceable, retrievable and legible and include sufficient information and explanation such that any staff that did not perform the generation can readily interpret them. This also allows repetition of the analysis at a later date if necessary.

While performing a test, all the necessary information is recorded in different manners, depending on the type of test. A test that does not use automated equipment has all necessary information recorded in bound laboratory notebooks. The logbooks are test specific and contain the date of analysis, the initials of the analyst, the laboratory sample numbers, the result obtained, the units of the results, any calculations involved, instrument ID, and any comments concerning unusual circumstances encountered. All entries into the logbooks are original and are recorded in indelible ink.

Other tests require the use of automated instruments and computers. The computer printouts generally contain the laboratory sample number or a reference ID which can be traced to the sample number using an analytical run logbook, instrument ID, analyst's initials, date of analysis, and time of analysis in the header information. The header should also contain a reference to the method being used. Included with each package of data there is a listing of samples analyzed in the sequence and a listing of the instrument parameters under which the samples were analyzed. If any deviations from the set instrument parameters are required for an individual sample, these changes are noted on that sample's printout. If an instrument is set to run over more than one shift and another analyst will be taking over the responsibility of the analysis of samples run on his/her shift, a comment is written on the printout of the first sample where responsibility was transferred.

If any corrections or amendments to the records need to be made, the incorrect data will have a single line drawn through it and the correct data entered. The change is initialed and dated by the person responsible for making the change.

14.3.3. Preps Performed Entry

Preparatory processes, such as sample digestions or extractions, frequently involve dilutions or concentrations of samples before analysis begins. These dilutions or extractions result in factors that must be taken into consideration when calculating final concentrations. In order to account for these "prep factors", the initials and final volumes/weights are entered into the LIMS after the samples are prepared and readied for analysis. This is accomplished by a preparatory analyst in the LIMS "Posting" area to create a batch of sample prep factors. Each batch is test specific and contains the date of preparation, the analyst performing the prep, the associated batch QC sample identifications, and the initial and final volumes/weights of each of the samples in the batch. These initial and final measurements are then easily retrievable and automatically used in the calculation of final sample concentrations, which are reported to the client. This system eliminates the possibility of human error resulting from manual calculations.

14.3.4. Reporting Results

Once an analysis has been completed and results checked for validity, the data is available to enter into the LIMS. All sample results and their associated quality control data are entered into the LIMS under the individual laboratory departments for the analyses performed in those departments. This is accomplished by each analyst in the LIMS "Posting" Entry/Edit area. Each batch is test specific and contains date and time of analysis, analyst completing the analysis, the instrument used for analysis, and sample results generated for one or more client samples as well as the associated quality control data produced during the analysis of those samples. Also, any comments concerning the samples themselves or the analysis of the samples, such as nonconformances or interferences with the method are entered at this time. All results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

15. Sample Handling

15.1. Sample Collection

15.1.1. Sampling Procedures

In order to produce meaningful analytical data, ALSI must have samples that are representative of the system from which they were taken. If the representation and integrity of the samples received in the

laboratory cannot be verified due to inadequate sampling procedures, the usefulness of the analytical data produced for these samples is limited. The laboratory cannot accept responsibility for improper sampling of client-procured samples and will document the condition of the samples and analyze them as delivered. If an incorrect sampling procedure is suspected, the client will be notified as soon as possible by the Customer Service Coordinator. ALSI will postpone testing, if the holding time will not be exceeded, pending client response. Sampling instructions and acceptance criteria are available to clients upon request.

Sample collection services are provided by ALSI through personnel from the field services department. Documented procedures for field sampling are outlined in the *Field Services Sampling Plan*. Sampling personnel ensure that collected samples are representative of the original systems, fully labeled and identified, and properly preserved and transported to the laboratory.

Where sampling, as in obtaining sample aliquots from a submitted sample, is carried out as part of the test method, the laboratory uses documented procedures as outlined in SOP 19-Subsampling to obtain a representative sub sample.

15.1.2. Bottle Preservation

Bottle preservation is performed by ALSI according to the information in Appendix B, which lists appropriate preservatives and holding times for commonly performed analyses. The grade of acids and bases that are used for preservation are all designated for specialized instrumental methods, including trace organic or trace element analysis. The lot numbers of the acids and bases used for preservation are recorded in a logbook kept in the sample receipt room.

Because ALSI does not know the exact nature of the matrix to be preserved, a uniform amount of acid or base is added to the bottles prior to shipment. The amount added equates to 4.0 ml of concentrated acid per liter of sample or approximately 4.0 ml of 10 Normal sodium hydroxide solution per liter of sample. For samples collected in amounts less than one liter, the preservative amounts will be decreased proportionally.

Upon return of the samples to the laboratory, the pH is measured on all samples (excluding volatiles) where pH adjustment is needed for proper preservation. If the pH of any container is outside the specified limits for a given test, additional acid/base is added to bring the pH into the proper range. An adjustment of this nature is recorded in the Preservation Logbook (See Figure 4), which is maintained in the sample receipt area, along with the lot number of the preservative used. It is also documented on the Corrective Action Form (See Figure 5), which is maintained with the chain of custody. It is the responsibility of the Sample Custodian to maintain the Preservation Logbook. Any adjustment is also noted as a comment on the final laboratory report. This testing and adjustment procedure is also followed for other preservation requirements such as the addition of sodium thiosulfate to eliminate residual chlorine content and temperature of samples.

For volatile organic samples, the pH and chlorine content is checked using two different procedures depending upon the type of autosampler used. For the Archon style autosamplers, the pH and chlorine content of the sample is not checked until the analysis of the sample is completed. This is because the analysis of a VOA sample is done in a completely closed system and the sample is never opened in the laboratory. The Archon autosampler removes an aliquot for testing by piercing the septa and withdrawing the sample at the time the sample is analyzed. At the completion of the analysis sequence, the sample remains are tested for pH and chlorine content using conventional methods. For the older style autosamplers (Tekmar 2016, etc.), the sample bottle is opened and a portion of sample is transferred to a purging chamber for analysis. The sample pH and chlorine content is checked using the remaining sample in the bottle after the sample has been loaded on the instrument.

The pH and chlorine content will be recorded on the associated laboratory logbook in both the GC or GC/MS departments. If a sample pH or chlorine content is not within the proper range the sample will be analyzed, a comment (qualifier) will be placed on the final laboratory report, and a corrective action form will be initiated. The proper customer service coordinator will be notified by the department, who will then take the proper steps to notify the client. The client can then make the decision as to whether or not to resample.

The sample custodian will check the volatile analysis bottles upon receipt to ensure no headspace is present. If sample containers have been found to contain headspace, the sample custodian will note this on the Chain of Custody. The sample custodian will notify the appropriate customer service coordinator who will then take the proper steps to notify the client. The client will be informed that the volatile sample does not conform to proper sampling procedures. It will then be the client's decision as to whether the sample is analyzed or resampled. If the sample is analyzed, a comment will be placed on the lab report stating that the sample contained headspace.

For tests that require filtration prior to preservation, this filtration is performed in the field prior to the sample entering the bottles containing preservative. If the laboratory must filter any sample prior to analysis it should not be preserved. The type of filter used and the date and time of filtration will appear as a comment on the laboratory report.

15.1.3. Holding Time

Holding time is the time from sampling until the start of analysis. The date and time of sampling documented on the chain of custody establishes the time zero. If the holding time is specified to be measured in hours, then each hour is measured from the minute the sample was collected in 60-minute intervals. When the maximum allowable holding time is expressed in days, the holding time is based on calendar day measured from time zero, the date the sample was collected. The first day of holding time is not passed until midnight of the day after the sample was collected. Holding times for analysis include any necessary re-analysis due to instrument failure or analyst error that does not yield useful data. If a re-analysis is necessary due to the nature of the matrix of the sample, such as a dilution or matrix spike failure due to matrix interference, the holding time has been met if the initial analysis was run within time. A comment is added to the final report stating that further analysis was required past hold time. The sampling time must be documented on the chain of custody form by the client. See Appendix B for a listing of analytical methods and their holding times.

15.1.4. Turnaround Time

Turnaround time is the time from receipt of samples to the transmittal of analytical data by mail, electronically or facsimile. The signing of the chain of custody form by the sample custodian sets the zero time for the reporting turnaround time. The required turnaround time will be based on a twenty four hour day multiplied by the number of days, from the time of signing of the chain of custody form, not including weekends and holidays, unless specified in the job specific quotation. The turnaround time for samples received weekdays after 3:00 PM will be the following business day.

15.2. Sample Custody

15.2.1. Chain of Custody Form

Chain of Custody documentation tracks the historical possession of samples. A completed Chain of Custody must accompany all samples entering the laboratory (Appendix C). This form provides essential

information to the laboratory regarding sample collection and analyses required, and includes the client 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.

Sample preparation logs and sample analysis logs document the custody and transfer of sample extracts/digestates from preparation to analysis. The LIMS also tracks these processes and documents them on the final laboratory analysis report. A more detailed internal chain of custody logbook is available for use upon client request for special projects or for samples being tested for litigation purposes (See Figure 6). Standard Operating Procedure 99-LCOC provides detailed instructions describing legal chain of custody procedures. Standard Operating Procedure 19-Rec/Han provides instruction on reviewing and inspecting the chain of custody for discrepancies. This is accomplished by inspection and comparison of the samples received against the chain of custody to identify any discrepancies. Sample receiving will immediately notify the customer service department of samples received without a chain of custody via US Mail, Federal Express, UPS, etc. These samples will be placed on hold until the chain of custody is received from the client. For walk-in customers dropping off samples, a copy of the COC will be given to them to fill out before sample acceptance. Complete chain of custody documentation, including memos, transmittal forms, etc., are filed and properly retained by the laboratory.

15.2.2. Sample Receipt

Upon arrival in the laboratory, samples are received by a sample custodian who ensures that all samples are accompanied by a proper Chain of Custody. The Chain of Custody 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 against the Chain of Custody to identify any discrepancies before signing the form and receiving the samples into the custody of the laboratory. The date and time relinquished/received is also recorded on the Chain of Custody. A Cooler Receipt Checklist is also filled out on the Chain of Custody 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.

15.2.3. Acceptance/Rejection Criteria

When a sample arrives in the laboratory, a decision is made to accept or reject the sample. It is the responsibility of the Sample Receiving Department to verify and document any nonconformance regarding use of appropriate sample containers, preservatives, packaging, and incorrect documentation and labeling upon receipt of samples. The condition of the sample, such as sample cooler temperature, pH, chlorine content, etc., is recorded on the Chain of Custody and in the Sample Preservation Logbook (See Figure 4). Any conditions outside of acceptance criteria are noted as comments on the final laboratory report. In cases where samples are hand delivered to the laboratory immediately after collection, they are immediately refrigerated.

ALSI reserves the right to reject a sample upon receipt in the laboratory if any of the following conditions occur:

- The sample is not properly documented on the chain of custody form and on the sample label with water resistant labels and indelible ink. Documentation shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type, and any special remarks concerning the sample.
- The sample has exceeded the holding time for the requested analysis.
- The incorrect preservative was used during sample collection.
- Incorrect sampling protocols were used during sampling (i.e., a sample not being filtered in the field for dissolved metals).

- Improper sample container was used.
- Insufficient sample is present to perform the requested analysis.
- Improper storage or transport of sample has occurred prior to receipt.
- Excessive amount of sample has been collected or other conditions exist which would make disposal difficult.
- Samples show signs of damage or contamination.
- Sample contains hazardous raw material that is not accompanied by an MSDS or material that cannot be safely handled by the laboratory (i.e.: radioactive material).

When there exists any doubt as to a samples' suitability for testing, where the sample does not conform to the description provided, when the samples show signs of damage or contamination, or where the test required is not fully specified, the appropriate customer service representative or project manager is notified. They, in turn, are responsible for notifying the client for further instruction before proceeding. Any instruction given by the client is recorded on the original chain of custody and may be noted on the Final Lab Analysis Report. If the sample does not meet the above acceptance criteria, the appropriate customer service representative or project manager shall retain correspondence and/or records of conversations concerning the final disposition of rejected samples or fully document any decision to proceed with the analysis of samples not meeting acceptance criteria. The condition of these samples shall, at a minimum, be noted on the chain of custody document. The analysis data shall be appropriately qualified on the final report.

15.2.4. Sample Identification and Control

To ensure sample accountability, all samples receive a unique sample identification number upon receipt into the laboratory. This identification number, which is used to identify and track the sample throughout the laboratory, is recorded on the chain of custody and on the durable water-resistent labels placed on all of the sample bottles. This number along with information from the chain of custody form is entered into the Laboratory Information Management System (LIMS). The LIMS contains all the information necessary to locate and track the sample. It also contains the information regarding specific analyses and turnaround commitments.

15.2.5. Sample Storage

Since samples have different storage requirements, samples are maintained in various locations throughout the laboratory. Samples are stored away from all standards, reagents, and other potentially contaminating sources in such a manner as to prevent cross-contamination. The majority of the samples received are refrigerated at 1-4°C to comply with state certification requirements. Samples requiring NELAP certification may be stored at 4+/-2°C. Samples receiving volatile analyses are segregated from other samples in order to prevent cross-contamination. High concentration material or neat chemicals shall be stored separately. Samples not requiring refrigeration, such as aqueous metals samples, are stored in room temperature cabinets. After results are reported to the client, samples are held for an approximate two-week period. At the end of the two-week holding period, samples will either be discarded by the laboratory or returned to the client. ALSI will not be responsible for disposal of materials known or suspected to contain dioxins, dibenzofurans, radioactive material, and high level PCBs. Special storage requirements for legal, project or other reasons will be met upon request.

All refrigerators and freezers used for sample and standard storage contain thermometers immersed in glycerol. The temperature of each refrigerator or freezer is recorded at least once a workday according to instructions in SOP 99-TEMP.

The thermometers used in the refrigerators and freezers will be calibrated against a NIST certified thermometer reference standard which is used for calibration only, and for no other purpose, once a year at a minimum of two points surrounding the temperature range of the thermometer being calibrated

according to instructions in SOP 09-TCAL. A hardbound logbook will be kept for recording the temperatures. The calibrations of the thermometers will be recorded in logbooks. The NIST certified thermometers will be returned to the manufacturer once every three years for recalibration.

15.3. Subcontracted Analyses

There are occasions when particular laboratory analyses cannot be completed in-house by ALSI. This may occur because the laboratory does not have the necessary instrumentation, equipment or certification to perform the analyses. ALSI also subcontracts overflow work as necessary when instrument problems occur or physical capacity is exceeded. Prospective subcontracting firms are thoroughly reviewed with an emphasis on their quality control program and associated certifications. A register of all approved subcontractors is retained in the QC Department. ALSI will ensure that the laboratory receiving the subcontracted work maintains the necessary certifications and level of quality to perform the work to project specifications.

ALSI advises its clients in each proposal of its intention to subcontract any portion of the testing to a third party. If it is necessary to subcontract work as a result of unforeseen circumstances, clients are contacted by their client service representative to gain their permission. This is documented on the Chain of Custody. When samples are sent, they are shipped to an appropriately certified subcontracting firm from ALSI's sample management department and the results of the analyses are transmitted back to ALSI for review. Any subcontracted analysis is noted as such on ALSI's final laboratory analysis report with an identification of the appropriate subcontractor. The original subcontractor analysis reports may also be attached to the associated ALSI Laboratory Analysis Report.

Samples requiring NELAP certification will be shipped to an ALSI approved laboratory accredited under NELAP for the tests to be performed and/or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed.

16. Laboratory Facilities and Equipment

16.1. General Description

ALSI is located on a one-acre lot on the north side of PA Route 283, five miles southeast of Harrisburg. ALSI's address is 34 Dogwood Lane, Middletown, PA 17057. The building has an area of approximately 16,000 square feet on two levels. The upper level contains the GC, GC/MS, sample preparation and sample receipt laboratories, sample storage areas, customer service and administrative offices (see Appendix D, Figure 1). The lower level contains the metals, wet chemistry, administrative sales office, QA department, computer office, sample storage, gas storage, chemical and waste storage areas (see Appendix D, Figure 2).

16.2. Security

The building has fire and smoke alarms and an electronic security system that are monitored by ADT. During weekends and off-shift hours the electronic security system is used to prevent unauthorized entry into the building. At approximately 7 p.m., the doors providing access to the building are locked and are kept locked until approximately 7 a.m. Any non-employee wishing to enter the building during these hours must use the door buzzer and wait for an employee to permit them access. All visitors, both during business hours and off-shift hours, must sign in and out at the receptionist's desk. The employee whom they are visiting will be notified of their presence and will come to the lobby to escort the visitor to the appropriate office or lab. People dropping off samples for analysis (and who do not leave the reception area) and field samplers entering the building for the sole purpose of dropping off samples in the Sample Receipt area will not need to sign in.

16.3. Laboratory Accommodations

ALSI has made great efforts to ensure that laboratory accommodations including things such as lighting, temperature, ventilation, and energy sources are consistent to facilitate proper performance of testing in all areas. Temperature and humidity are important factors in the operation of instrumentation in the laboratory. All areas of the laboratory have separate temperature controls in order to allow for optimal adjustment of these factors. Certain areas of the laboratory contain controls for temperature and humidity due to the operational and test method regulation specifications of the specialized equipment contained in those areas.

Regarding ventilation, extra provisions have been developed in order to provide clients with a greater assurance that their samples are being processed in a professional and quality environment. These include such things as maintenance of a negative air pressure in the preparatory laboratories and a net positive pressure in the organic laboratories and hallways leading to the preparatory laboratories. Additionally, access to and use of neighboring areas where activities are incompatible is controlled. This ensures that any solvent contamination resulting from sample extraction processes is kept out of the analysis laboratories. Also, all fume hoods meet OSHA standards for face velocity, and good housekeeping practices are maintained throughout the laboratory. This helps to ensure the safety of ALSI employees as well as the integrity of client samples.

16.4. Waste Management

ALSI conducts waste management practices consistent with all applicable EPA rules and regulations. Spent reagents, samples and method process wastes are characterized and disposed of in an acceptable manner.

Reference SOP 19 – Waste Disposal for information regarding ALSI's waste management procedures.

16.5. Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

16.6. Deionized Water

Deionized water is used for all analyses performed in the laboratory. Deionized water is provided to each individual laboratory by a laboratory-wide circulation system that has been designed and is currently serviced by an outside vendor. This system consists of tap water being introduced first to a sediment cartridge, with a 1-micron pore size, which is used for particle elimination, then to carbon filters, which are used for removal of chlorine and organic contaminant. Water is circulated through three ion exchange tanks, containing anion and cation resins that are used to deionize water, and an ultraviolet sterilizer, which is used to provide control of bacteria. A polishing tank containing mixed anion and cation resins is also used as a final deionizing source and to act as a safeguard to maintain the quality of the water before and during servicing. Finally, water flows through a 0.2 micron filter to remove any colloidal silica before distribution to all areas of the laboratory. This system provides quality water with a resistivity reading of greater than 18.0 MΩ·cm. The deionized water system is equipped with a continuous resistively monitor. The readings are monitored and recorded daily. The conductivity of the water is determined and recorded at least weekly by the water quality department.

16.7. Gas Storage

A separate gas storage room contains the necessary specialty gases that are required for specific analytical equipment. The gases leave the centralized storage room through individual lines leading to the analytical departments. Other non-flammable gas tanks can be found in wet chemistry, GC-Volatiles, and GC-Semivolatiles. All gas tanks are properly secured with chains and department personnel are trained in proper handling.

16.8. Instrumentation

The laboratory has a full complement of instrumentation and support equipment such as fume hoods, refrigerators, freezers, ovens, balances, a deionized water system, etc. required for the correct performance of all tests. All instruments are maintained by trained employees and/or by manufacturer's service personnel. Reference materials, including instrumentation manuals provided by the manufacturer, are available to provide instruction in the proper use of the instrumentation. A complete listing of instrumentation and equipment is included in Appendix E.

16.9. Instrument Maintenance

Preventative maintenance as well as some repairs can be accomplished on-site by ALSI personnel. ALSI also maintains some service agreements with instrument manufacturers to further ensure the operational viability of all equipment.

All maintenance and servicing done on instruments and equipment is recorded in hardbound notebooks. The instrument logs contain general information about the instrument, including the name of the manufacturer, instrument model, serial number, date of purchase, date placed into service, current instrument location, condition when received (e.g., new, used, reconditioned), and information concerning any service contracts maintained. They also contain information concerning any routine maintenance done by ALSI personnel. Information concerning routine maintenance should include a brief description of the maintenance performed, the frequency required, the date performed, and the initials of personnel performing the maintenance and any comments concerning the procedure. Also contained in the log is information concerning repairs done by ALSI personnel or instrument manufacturers. This information includes the date of servicing, the initials of personnel performing the service, record of why it was done and the results of the servicing relative to instrument performance. The individual logbooks are located in the laboratory with the instruments to which they pertain along with copies of manufacturers instructions, where available. Once a logbook is filled, a new logbook will be started. The general information about the instrument and the routine maintenance required will be transferred over to the new logbook. The old logbook may remain in the laboratory for at least one year so that it may be used as a reference by the analysts. When removed from the department the old logbook will be put into storage, where it will be kept for a minimum of seven years.

Instruments and other equipment, such as ovens, will also have a piece of paper attached to them indicating the status of the equipment. This is to ensure that the employees starting a new shift are aware of the calibration status of instruments and any problems that might have been encountered with any equipment. This is also to ensure that, if any piece of equipment is not functioning properly, employees are aware of it and will not accidentally use it. All information placed on the paper will be dated and initialed by the person writing the message. Instruments, which are not functioning properly, are exempt from QC checks while not in use.

It is the responsibility of the section leaders to determine the effect, if any, of an instrument defect on previous results. If an effect has been determined that questions the validity of any sample results, the QA Manager is notified and the corrective action procedure is followed.

16.10. Instrument Calibration

All instrumentation must be calibrated prior to use. The initial calibration determines the working range of the instrument by measuring the analytical response in relation to the amount of analyte present. All initial calibrations are method specific, and may be comprised of a single point or multi-points. The type of calibration performed depends on the type of instrumentation and the method of analysis in use. Step by step calibration procedures are outlined in detail in each Standard Operating Procedure. Also included is the frequency of calibration required and the materials needed to perform each calibration. Only standard reference materials as defined in section 8.2 are used for calibration.

All initial calibrations are verified for accuracy by analysis of a second source standard. This is a check standard prepared from a reference material procured from a different source than that used for the calibration. This provides verification that the calibration standard has been prepared at the correct concentration.

All initial calibrations are controlled by analysis of continuing calibration standards and/or QC check samples. These are method specific or mid-range level calibration standards that are analyzed at specific frequencies as established by the method. The amount of analyte recovered is compared to the acceptance criteria of the method. Acceptable recoveries verify the stability of the calibration and lack of instrument drift throughout the analysis. If the acceptance criteria are not met, method specific corrective action must be taken.

17. Data Verification Procedures

17.1. Data Quality Objectives

The data quality objectives discussed below ensure that data will be gathered and presented in accordance with procedures appropriate for its intended uses, and that the data will be of known and documented quality able to withstand scientific and legal scrutiny. The quality of the measurement data can be defined in terms of completeness, accuracy, precision and traceability.

17.1.1. Completeness

Completeness is defined as the percentage of measurements that are judged to be valid measurements. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, missed method prescribed holding times, lost sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or rejection of sample results due to failure to conform to QC criteria specifications.

17.1.2. Accuracy

Accuracy is the measure of agreement between an analytical result and its “true” or accepted value. Deviations from a standard value represent a change in the measurement system. Potential sources of deviations include (but are not limited to) the sampling process, sample preservation, sample handling, matrix effects, sample analysis and data reduction. Sampling accuracy is typically assessed by collecting and analyzing field and trip blanks for the parameters of interest. Analytical laboratory accuracy is determined by comparing results from the analysis of laboratory control samples or check standards to their known values. Accuracy results are generally expressed as percent recovery.

17.1.3. Precision

Precision is the determination of the reproducibility of measurements under a given set of conditions, or a quantitative measure of the variability of a group of measurements compared to their average value. Precision is typically measured by analyzing field duplicates and laboratory duplicates (sample duplicate,

matrix spike duplicate, check standard duplicate and/or laboratory duplicate). Precision is most frequently expressed as standard deviation, percent relative standard deviation or relative percent difference.

17.1.4. Traceability

Traceability is the extent to which reported analytical results can be substantiated by supporting documentation. Traceability documentation exists in two essential forms: those, which link the quantitation process to authoritative standards and those, which explicitly describe the history of each sample from collection to analysis and disposal.

17.2. Laboratory Quality Control Checks

Technical personnel are responsible for complying with all quality assurance/quality control requirements that pertain to their technical functions. ALSI uses the following internal quality controls to verify that the data produced by the laboratory has the required degree of accuracy and precision and is free from contamination due to laboratory processes. All samples are normally processed in preparation and analytical batches of no more than 20 samples per batch. The following quality control checks defined below are appropriate for the various methods performed in the laboratory. Individual SOPs will further define the specific checks to be analyzed with each method. Additionally, a client's individual Quality Assurance Project Plan may require the laboratory to include additional checks for analysis depending on the *site* requirements.

17.2.1. Method Blank

A method blank is an analytical control consisting of all reagents, internal standards, and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination contributed from the preparation or processing of the sample.

17.2.2. Reagent Blank

A reagent blank is an analyte-free sample that contains all the reagents used in a particular method. It is prepared and analyzed to determine if contamination is present at detectable levels that can be attributed to the reagents used in the process.

17.2.3. Field Blank

A field blank consists of reagent water that is transported to the sampling site, transferred from one vessel to another at the site, and preserved with the appropriate reagents. This serves as a check on reagent and environmental contamination.

17.2.4. Trip Blank

A trip blank consists of reagent water that is transported to the sampling site and returned to the laboratory without being opened. This serves as a check on sample contamination originating from sample transport, shipping, and from the site conditions.

17.2.5. Refrigerator / Storage Blank

Refrigerator storage blanks are placed in VOA sample storage refrigerators and routinely analyzed for full Volatile Organic Analytes/Target Compound List (VOA-TCL) analytes by GC and GC/MS. These blanks monitor the volatile storage refrigerators for presence of sample cross-contamination.

17.2.6. Quality Control Reference Sample or Calibration Verification Standard (Second Source Standard)

A QC reference sample is a sample prepared from a source other than that used for calibration at a concentration within the calibration range. It is used to verify that the calibration standards were prepared accurately. It is analyzed after every initial calibration performed in the laboratory.

17.2.7. Laboratory Control Sample

An LCS is a laboratory blank fortified at a known concentration. Aqueous and solid LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples. An LCS is analyzed with each preparative or analytical batch as required by the method. It provides a measure of the accuracy of the analytical system in the absence of matrix effects.

17.2.8. Surrogate Standards

Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference samples), and spiked samples prior to an organic analysis. Percent recoveries are calculated for each surrogate to detect problems in the sample preparation process and monitor the efficiency of the process.

17.2.9. Duplicate

A duplicate is a second aliquot of a sample that is prepared and analyzed in the same manner as the original sample in order to determine the precision of the method. Samples selected to be analyzed in duplicate are rotated among client samples so that various matrix problems may be noted and/or addressed. Poor precision in a sample duplicate may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate analysis. Precision is measured as relative percent difference as determined using the following formula:

$$\%RPD = (((S - D) / (S + D)) / 2) * 100\%$$

where: S = Sample Result
D = Duplicate Result

17.2.10. Matrix Spike/Matrix Spike Duplicate

A matrix spike/matrix spike duplicate is the addition of a known amount of a target analyte to a sample that is subjected to the entire analytical procedure. Samples selected for matrix spiking are rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. Accuracy is determined by calculating the percent recovery.

17.3. Proficiency Testing (PT) Samples

These reference materials provided by the EPA, state agencies, government agencies or certified commercial vendors monitor accuracy performance of the laboratory on a regular basis. It is an essential part of ALSI's quality program to maintain accreditation and certification by regulatory agencies. It is laboratory policy to analyze and report PT samples as if they were regular compliance samples. This includes analysis of all methods under the Scope of Work for NELAC and includes all matrices presently accreditated for by both NELAC and non-NELAC state agencies. Proficiency on PT samples measures

performance of our analytical capabilities. Participation in these programs provides the laboratory with evidence of correlation of results with other laboratories and national standards. Proficiency testing results are filed and archived for at least seven (7) years. ALSI participates in the following programs:

Program	**Sample Type**	**Frequency**
In-house Blind Study	Organics/Inorganics/Metals	Quarterly
Water Pollution Study (WP) provided by a NELAC and/or NIST approved provider	Organics/Inorganics/Metals/Microbiology	Semi-annually
Water Supply Study (WS) provided by a NELAC and/or NIST approved provider	Organics/Inorganics/Metals/Microbiology	Semi-annually
APG Plus Study (WP) 2-Level Proficiency Testing performed for West Virginia Certification	Organics/Inorganics/Metals	Annually
Discharge Monitoring Report provided by a NELAC and/or NIST approved provider	Inorganics/Metals	Annually
U.S.A.C.E.	Organics/Inorganics/Metals	As required
State of North Carolina	Organics/Inorganics/Metals	As required
Laboratory Soil Proficiency Testing Program	Organics/Inorganics/Metals	Semi-annually

17.4. Quality Control Charts

Control limits are used by ALSI to establish method performance of a given analysis and to monitor trends of QC results graphically over time. These limits also allow for the development of acceptance criteria where no method or regulatory criteria exist. Each analytical department records control limits and calculates the upper and lower control limits. Warning limits are at plus and minus two standard deviations, and control limits are at plus and minus three standard deviations.

Documented acceptance limits are available before an analysis is begun based upon continuing statistical evaluation of data generated by the analysis of quality control samples or specific minimum acceptance limits established by the method and/or Standard Operating Procedure. This allows any out-of-control parameters to be detected before data is reported. If the Out-of-control parameter is judged to be sample related, the analysis may continue. The corrective action policy must be followed, and the result reported with a comment qualifying the results.

When an analysis is deemed out of control by the analyst performing the analysis, the reason for the out-of-control situation is investigated immediately. The response to the out-of-control situation will depend on the analysis and the SOP shall be consulted. In addition, the supervisor is informed of the problem and he/she does not allow any further analyses until the problem has been corrected. Corrections may include reassay of the check samples, recalibration, instrument maintenance or other SOP mandated operations. If it is necessary to report results obtained when the system is judged to be out-of-control, the corrective action policy will be followed, and the data will be flagged on the laboratory analysis report and a qualifying comment will be attached.

17.5. Method Detection Limits

Method detection limits (MDLs) are determined and documented in accordance with federal guidelines contained in 40 CFR Part 136 Appendix B. The detection limit is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix. The statistical procedure allows for computation of method detection limits based upon a minimum of seven analyses of a check sample that is prepared at a concentration between 1 and 5 times the estimated MDL. Once an MDL study has been performed, the MDL concentration observed is deemed acceptable if it is between 1 and 10 times the concentration spiked. The procedure outlined in SOP 99-MDL explains in detail the procedure to be followed.

Method detection limit studies are performed where appropriate for each method and matrix of concern during initial method validations or when a major change in operating conditions or instrument configuration occurs. If operating conditions during the course of the year do not require an MDL study for a specific method, a study will be performed at least once per year for most methods to maintain adequate records of the method's performance.

Reporting Limits or Practical Quantitation Limits are defined as the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. These are listed on all lab reports along with the analytical result. They are determined by multiplying the calculated Method Detection Limit by three (2) to five (5) times and incorporating a safety factor that is developed using guidance from recovery studies and blank concentrations, or by reporting the lowest standard of the initial calibration curve.

17.6. Data Reduction, Validation and Reporting

ALSI has an established sequence of approvals that analysis results undergo before being issued to a client on a final report as described in SOP 99-DATA. Each step in the process is initialed and dated upon completion. The individual analyses on the report are initially reviewed by the analysts while performing the testing. The analyst ensures that all quality control information is in control and correct. The data generated by the analyst is then reviewed by the laboratory supervisor or designee responsible for that particular analysis. The supervisor or designee reviews the raw data including sample and QC data. The records are checked for SOP compliance and calculation errors. This check is documented on the raw data. Once all the data has received approval by the supervisors or designees, results are entered into the LIMS. Once all inorganic results of an analytical batch have been entered, they are checked by a second analyst for transcriptional errors and completeness. Once all results are confirmed to be reported accurately, the data are approved and the report is marked as complete in the LIMS. Organic data is electronically uploaded and does not need to be checked for transcriptional errors and completeness. Once all of the data for a workorder has been entered into the LIMS and the data reviews by the sections have all been completed, the workorder is scheduled to print automatically by the LIMS during the next print run. The report is then printed with the electronic signature of the Laboratory Manager. If at any point in the data reduction, validation, and reporting process, an error is found in the data entered into

the LIMS, the error will be corrected and the correction will be noted in an audit trail function in the LIMS system. The LIMS will document the sample number, the correction that was made, the reason for making the correction, the date and time the correction was made and the initials of the person making the correction.

All of the information necessary for the interpretation of the test results and all information required by the methods used is included on the report. The report lists the laboratory sample number, client name and address, project name, the project number, location identification, sample state, sample collector, purchase order number (if available), date and time (if available) sampled, date received, and a discard date. This information is included in the heading of the report. In the actual body of the report, the test/parameter being analyzed is listed along with the result, units, limit of detection and method reference. Also included may be "line item comments" where a comment is attached directly to a particular result, and "sample comments" where comments are attached at the end of a report. These comments will indicate any out of the ordinary circumstances such as a method non-conformance or interference problem. At the end of the report is included the percent recovery of any surrogate standards used.

The front page of the report supplies additional information not included in the body of the report. This information includes the definitions of qualifiers used in reporting sample results, laboratory certification information and the signature of an approved signatory.

Once a final report has been approved, only the Laboratory Manager, the Technical Director, the QC Manager and the customer service representatives have the access to change the report. If changes need to be made to a report that has already been transmitted to the client, and a comment will be put on the report as to what change was made, the date it was made and the initials of the person making the change.

If additional tests are requested on a sample that has been started but not finished by the laboratory, the test request will be added to the appropriate sample in the LIMS. Additional tests for a laboratory sample will be added by either the Laboratory Manager, Technical Director, sample custodian or the customer service representative.

17.7. Data Deliverable Reporting

ALSI is capable of developing a variety of data deliverable reports. For all deliverables produced the hardcopy data is scanned and a CD ROM is maintained by the QC department in a secure location. All packages are organized and assembled on a specific basis in order to comply with the client's regulatory or project specific requirements. They are reviewed for completeness and compliance by a QC Officer and verified by validation within the QC department. Shipments of deliverable packages are documented by the QC Officer and sent by the Project Manager through a commercial carrier. In general, ALSI Deliverables contain:

- Case Narrative – Information on sample types, tests performed, any problems encountered, and general comments.
- Analytical Data – Data are reported by sample with the appropriate significant figures and reporting limits, and have been adjusted for dilution, if appropriate. Pertinent information including dates sampled, received, prepared, extracted, and analyzed are provided in the data package.
- Laboratory Performance QC Information – The summary results and raw data of LCSs and method blanks analyzed with the project are provided. Any data or QC anomalies are discussed in the narrative or is listed as comments on the analytical report.

- Matrix Specific QC Information – The summary results and raw data of any sample duplicates and MS/MSDS analyzed with the samples as specific QC are provided. Any additional project specific QC requested by the client is also reported. All QC results include supporting information such as concentration of the spike, percent recovery, and percent difference/RPD.
- Methodology – References to analytical methodology used is cited in the deliverable package.

18. Corrective Actions and Feedback

When a system or performance audit indicates a deficiency, when established quality control limits are violated, or when professional judgment by an experienced analyst indicates that a result may be inaccurate, corrective action is necessary. Corrective actions are measures taken to correct a problem, which may adversely affect the quality of a reported result, and to prevent its reoccurrence if possible. Corrective action may consist of, but is not limited to, things such as re-preparation of a sample, reanalysis of a sample, maintenance of malfunctioning equipment, revision of standard operating procedures, qualification of a sample result with a detailed explanation, or re-training of personnel. See SOP 99-CorrA for details concerning the corrective action documentation procedure.

An experienced analyst, upon review of raw data, is expected to use professional judgment when accepting and rejecting results of an analysis. In cases in which an analysis successfully passes all method QC requirements, an analyst may use professional experience to judge the sample result questionable. This may be due to previous sample history, lack of correlation between results of multiple test methods performed, or general dissatisfaction with the result obtained. In these cases analysts are given the authorization to reanalyze the sample as a form of corrective action. In all cases of re-analysis, it must be documented clearly in the raw data which results were reported to the client.

If corrective action is required because routine data quality assessments are out-of-control, such as surrogate recoveries below acceptable limits or duplicate relative percent difference values above acceptable limits, the data is evaluated on a sample-by-sample and/or batch basis. Data is evaluated with respect to SOP criteria and the corrective action may be limited to rejecting the sample or batch or accepting it and reporting the result with a qualifying comment on the Lab Analysis Report. The decision that is made is indicated on the raw data, such as on the analytical worksheet or in the laboratory data book. If a quality control violation is judged to be matrix related, a Sample Non-conformance Form is completed (see Figure 7) and filed with the raw data. If a quality control violation is judged to be non-matrix related, a Corrective Action Form is completed (see Figure 5). If a trend is not observed during the course of data validation, additional corrective action or documentation is not necessary.

If the corrective action is required because of a systemic deficiency or if a situation occurs affecting data usability for more than one batch (i.e., standards preparation errors), a more global assessment must be made. In these cases, the deficiency, along with the corrective actions initiated, are recorded on a corrective action form. The corrective action documentation shall be completed by the department supervisor or his designee and include immediate action taken, and long term corrective action to be taken to prevent the reoccurrence of the deficiency in the future.

If at any point in ALSI's system it becomes evident that an error has occurred (either human or instrumental) which may have jeopardized the validity of reported results, the Quality Assurance Manager, Laboratory Manager and appropriate Customer Service Representative are notified immediately. An initial phone call is made by the Customer Service Representative or a letter is distributed immediately to all potentially affected clients defining the error. This letter or documented phone call will address the specific tests and samples involved and define the effect that the suspected problem would have on issued results. After a thorough review by the QA Manager, a letter may be sent more thoroughly defining the problem and associated resolution. All problems are also documented on a Corrective Action Form as explained in the Corrective Action SOP (99-Corr).

18.1. Preventive Action

Preventive actions are long-term improvements the laboratory makes to prevent non-conformances.

The Quality Assurance Manager tracks all external and internal non-conformances on a monthly basis. Based on this review the Quality Assurance Manager shall make the determination if there are any apparent problems or trends that need to be addressed within the laboratory operations. If necessary, system audits an/or method audits will be performed as a result of this review. The President and/or Laboratory Director will become involved where changes are required in personnel, instrumentation or procedures.

ALSI encourages all employees to contribute their ideas on issues relating to facility and system improvements, increased efficiency, waste elimination, ensuring a safe working environment and improving customer satisfaction while maintaining a high level of quality. For this purpose we have available a "suggestion box" that is centrally located and accessible to all laboratory personnel. Suggestions are evaluated for merit and regularly discussed at our open forum meetings held monthly with upper management and all laboratory personnel.

19. Departure From Procedure

There are occasions when it becomes necessary to deviate from documented policies and procedures. When a deviation is necessary, prior evaluation and approval by a laboratory manager is required and the customer must be notified. Customer approval can be granted verbally or in writing, but in all cases is documented. This comment states the specifics of the deviation, the applicable test(s) and the reasoning for the deviation. This comment appears on the final lab report. Deviations from SOPs will be noted with the appropriate initials and date in the lab notebook. All departures from procedures are documented on corrective action forms filed in the QA office.

20. Complaints

All complaints, whether initiated by clients, or generated internally are taken seriously. All complaints are handled according to the Standard Operating Procedure SOP 99-Complaints. This procedure outlines the steps taken to process complaints in the laboratory.

An external complaint consists of any non-compliance with customer's project specifications as indicated by the client. Upon receipt of an external complaint, an External Complaint Form is initiated (See Figure 8). Information such as customer name, client contact, date/time, applicable COC numbers, and the ALSI contact are documented. The specific items discussed are also documented. If an appropriate resolution can be worked out immediately with the client, that too is documented. Otherwise, the External Complaint Form is submitted to management for determination of an appropriate resolution. The finalized External Complaint Form is submitted to the QA Office for review and distribution. All external complaints are kept on file and archived in the QA office.

An internal complaint consists of any grievance, concern, or data quality related issues. All employees have access to Internal Complaint Forms in the Business Office at all times (see Figure 9). The Internal Complaint Form can either be returned to the laboratory "*suggestion box*" anonymously or submitted in person to the Human Resource Manager. All forms are reviewed, investigated, and distributed for resolution by the Human Resource Department.

21. Confidentiality, Proprietary Rights, and Transfer of Ownership

21.1. Confidentiality and Reporting

The confidentiality of client information is strictly maintained through rigid controls. Reports and information are issued only to the clients who have submitted the work except as otherwise indicated by the client. The laboratory will sign an acceptable confidentiality agreement as required. Copies of the final laboratory report, mailed via regular mail, are covered in the analysis fees. Additional copies of reports sent to another address may be charged per copy. Charges for express mail services, sample shipping, and fax services are extra.

21.2. Limitation of Liability

Notwithstanding any other provision herein, ALSI's liability and Client's exclusive remedy for any cause of action arising hereunder, whether based on contract, negligence, or any other cause of action, shall be limited to the compensation received by ALSI from the Client for the services rendered therewith. All claims, including negligence or any other cause whatsoever shall be deemed waived unless made in writing and received by ALSI within ninety (90) days after ALSI's completion of the services provided.

21.3. Transfer of Ownership

In the event of a transfer of ownership of the laboratory, the new owner will agree in writing, which shall be either stipulated in a purchase agreement or as a separate record retention document, that the current records shall be maintained for a period of not less than five (5) years.

In the event of a laboratory closure, the current owner/management will notify all clients, which the laboratory performed sample analysis within the last ten (10) years, in writing that the laboratory will be closing. This letter will instruct the clients to contact the laboratory to provide instructions on how previous records are to be transferred to the client's care.

22. Audit and Review

22.1. Audits from Regulatory Agencies

As a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits are usually conducted on an annual or bi-annual basis and focus on laboratory conformance to the specific program protocols for which the lab is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action within an agreed upon time frame. All audit reports are filed and archived for at least seven (7) years.

22.2. Internal Audits

The QA Manager or experienced designee is responsible for performing internal audits. All technical laboratory sections of ALSI are required to participate in these internal audits annually. The procedure for performing these internal audits is outlined in SOP 99-Intaudit. The findings of these audits are to be formally documented and submitted to the laboratory management. The Quality Assurance Manager, Laboratory Manager, Technical Director and/or Laboratory Supervisors will have the responsibility for resolving points at issue or for effecting necessary changes to the laboratory's practices within an agreed upon time frame, usually two weeks

The audit program is to focus on the following areas:

- Maintenance of acceptable and complete SOPs in company format.
- Maintenance of training records.
- Maintenance of notebooks.
- Maintenance of instrument records.
- Evaluation of standard control records.
- Evaluation of sample handling procedures.
- Evaluation of data handling and storage procedures.

When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's environmental test results, the laboratory shall take immediate corrective action. The client will be notified in writing whenever investigations show that their laboratory results are affected.

Discovery of evidence of inappropriate actions or vulnerabilities related to data integrity shall be investigated. Any investigation that results in findings of inappropriate activity shall be considered a violation of the "Ethics Credo". Actions taken shall include immediate disciplinary action, corrective action, and appropriate client notification, if necessary. All documentation relating to an investigation shall be maintained for five years.

22.3. Management Quality Review

A review of the entire quality system is to be carried out annually. The QA Manager, Laboratory Manager, Technical Director, and President will have the responsibility for resolving points at issue or for effecting necessary changes to the laboratory's quality assurance practices within an agreed upon time frame.

The purpose of these reviews is to discover:

- whether management objectives (as defined by the quality system) are being met
- whether designated duties are being carried out satisfactorily
- whether procedures described in the quality system are being followed
- opportunities for quality improvements

The review includes:

- matters arising from the previous review
- assessment reports from A2LA and other state or national certifying agencies
- reports from audits by clients
- reports from supervisory personnel
- corrective action summaries
- results of internal audits done since the last review, including corrective actions implemented
- results of participation in proficiency testing
- results of in-house quality checks
- details of any complaints from clients
- staff training (for both new and existing staff members)
- adequacy of staff, equipment and facility resources
- future plans and estimates for new work, new staff, new equipment, etc.

22.4. Audit Response

The laboratory is required to respond with corrective action to any audit findings and recommendations of regulatory agencies before certification for a particular program can be granted. If a recommendation is related to document format, then the laboratory personnel will revise the document format and a copy of the revised document format will be submitted to the appropriate representatives of the regulatory agency. If a recommendation is related to an actual procedure, then the recommendation will be

communicated to the laboratory personnel informing them of the correct procedure and a record of this communication will be submitted to the appropriate representatives of the regulatory agency. If a recommendation is related to the written procedures, then the laboratory personnel will revise the written SOPs and a copy of the new SOPs will be submitted to the appropriate representatives of the regulatory agency. The Quality Assurance Manager will conduct a follow-up to verify that corrective action has been implemented. All audit responses are filed and archived for at least seven (7) years.

23. Uncertainty of Measurement

The uncertainty components, which are of importance to a given procedure, must be accounted for using appropriate methods of analysis. Therefore, the laboratory must determine the uncertainty attributed to all steps in a testing procedure. These include uncertainty imposed by equipment, calibration, standards, reagents, preparation, cleanups, etc. Since for most analytical procedures, the laboratory control sample (LCS) is subject to the entire process of preparation through analysis, all procedural elements that would contribute to uncertainty will be inclusive in the overall LCS results. The LCS is performed with every batch of samples where appropriate for the method.

Measurement uncertainty is a statistical accuracy calculation equal to twice the standard deviation of the LCS recoveries for a given continuous set of LCS recoveries. This statistical observation is reported as standard deviation by percentage. Although there is no requirement that measurement uncertainty be reported with sample results, if requested by a client, it would be applied by multiplying the determined analyte concentration by the uncertainty percentage.

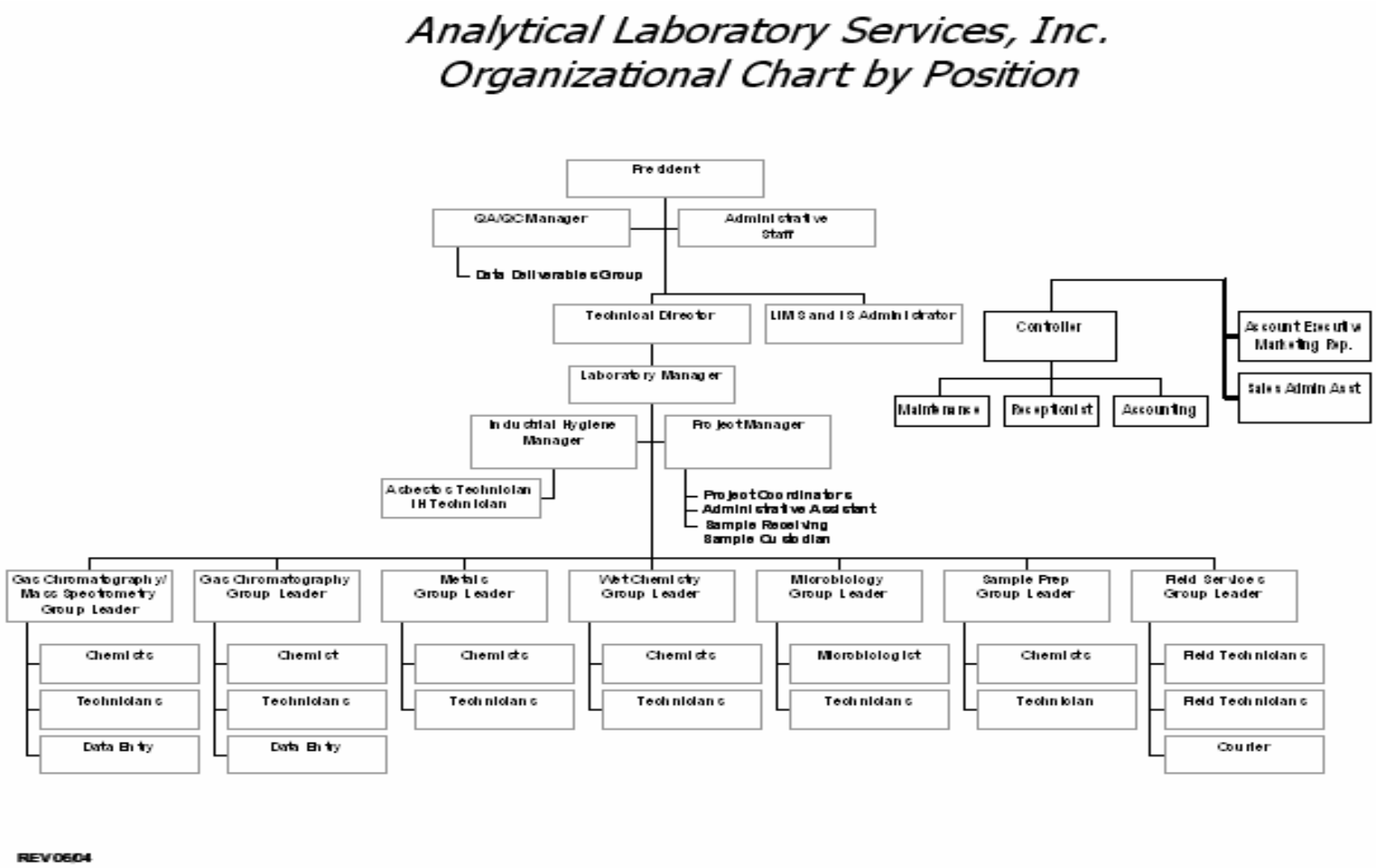
23.1. Measurement Uncertainty Calculation for Reporting

Uncertainty (at 95% confidence level, K=2) can be expressed as:
$X (1 \pm 2S_r)$

Where:

X is the analytical result
Sr is the relative standard deviation of the LCS data

Appendix A – ALSI Organizational Chart



Appendix B – Container, Preservation, Storage, and Holding Times

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
METALS						
Total Recoverable / Dissolved Metals Digestion	3005A	NW, LW	P or G	HNO_3 pH<2	6 months	500mL
Total Metals Digestion by Microwave	3015	NW, LW	P or G	HNO_3 pH<2	6 months	500mL
Total Metals Digestion by Hot Plate	3020A	NW, LW	P or G	HNO_3 pH<2	6 months	500mL
AAS Metals (except CrVI or Hg)	200 series	PW, NW	P or G	HNO_3 pH<2	6 months	500mL
	200.9	PW	P or G	HNO_3 pH<2	6 months	500mL
	3000/7000 series	S, SW	P or G	Cool 4 C	6 months	100g
ICP Metals	200.7	PW, NW	P or G	HNO_3 pH<2	6 months	500mL
	3000 series/ 6010B	S, SW	P or G	Cool 4 C	6 months	100g
ICP/MS Metals	200.8	PW, NW	P or G	HNO_3 pH<2	6 months	500mL
	3000 series/ 6020	S, SW	P or G	Cool 4 C	6 months	100g
Chromium, Hexavalent (CrVI)	3500CrD	PW,NW	P or G	Cool 4 C	24 hours	500mL
Chromium, Hexavalent (CrVI)	3000/7196A	S, SW	P or G	Cool 4 C	30 days digestion/168 hrs after digestion for analysis	100g
Mercury (Hg)	245.1 /7470A	PW, NW	P or G	HNO_3 pH<2	28 days	500mL
	7471A	S, SW	P or G	Cool 4 C	28 days	100g
INORGANICS						
Acidity	2310B	PW, NW	P or G	Cool 4 C	14 days	200mL
Albuminoid Nitrogen	221	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
Alkalinity	310.1/2320B	PW, NW	P or G	Cool 4 C	14 days	200mL
Alkalinity, Phenolphthalein	2320B	PW, NW	P or G	Cool 4 C	14 days	200mL
Ammonia (as N)	350.3	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
	350.3	S, SW	P or G	Cool 4 C	28 days	100g
Biochemical Oxygen Demand	5210B	PW, NW	P or G	Cool 4 C	48 hours	1000mL

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
(BOD)						
BOD, Carbonaceous	5210B	PW, NW	P or G	Cool 4 C	48 hours	1000mL
Bromate	300.1	PW,NW	P or G	50 mg/L EDA Cool 4 C	28 days	1000mL
Bromide	300.0	PW, NW	P or G	Cool 4 C	28 days	100mL
	9056.0	S, SW	P or G	Cool 4 C	28 days	25g
Chloride	300.0	PW, NW	P or G	Cool 4 C	28 days	100mL
	9056	S, SW	P or G	Cool 4 C	28 days	25g
Chlorine, Residual	4500G	PW, NW	P or G	Cool 4 C	Analyze immediately	200mL
Chemical Oxygen Demand	410.4	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	50mL
Chlorite	300.1	PW,NW	P or G (opaque)	50 mg/L EDA; Cool 4 C	14 days	1000mL
Color	110.2/2120B	PW, NW	P or G	Cool 4 C	48 hours	1000mL
Corrosivity	Langlier Index Calculation	PW, NW	P or G	1 bottle HNO_3 pH<2; 1 bottle 4 C	NA	50mL (2)
	150.1/ 9040B	LW	P or G	Cool 4 C	Analyze immediately	100g or 8 oz. jar
	9045C	S, SW	P or G	Cool 4 C	Analyze immediately	100g or 8 oz. jar
Cyanide	335.3	NW	P or G	NaOH pH>12; Cool 4 C	Distillation & Analysis 14 days	500ml
	335.4	PW				
	9012	S, SW	P or G	Cool 4 C	14 days	100g
Cyanide, Amenable	335.3/335.4/4500CNG	PW	P or G	NaOH pH>12; Cool 4 C	Distillation & Analysis 14 days	500ml
	9012	S, SW	P or G	Cool 4 C	14 days	100g
Dissolved Organic Carbon	5310B	PW,NW	G, amber	HCl pH<2;Cool 4 C	28 days	125mL
Flash Point	1010	LW	G, Teflon-lined septum	Cool 4 C	NA	100mL
Fluoride	4500F-C	PW, NW	P	None	28 days	100mL
	300.0	PW, NW	P	None	28 days	100mL
	9056.0	S, SW	P	None	28 days	25g
Hardness, Calcium	3500CaD	PW,NW	P or G	HNO_3 pH<2	6 months	500mL
Hardness,	3500CaD	PW,NW	P or G	HNO_3 pH<2	6 months	500mL

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
Magnesium						
Hardness, Total	2340B/200.7	PW, NW	P or G	HNO_3 pH<2	6 months	100mL
Landfill Gas	RSK175	NW	G	H_2SO_4 pH<2	14 days	40mL (2)
Nitrogen, Kjeldahl	351.3	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
	351.3	S, SW	P or G	Cool 4 C	28 days	100g
Nitrate-Nitrogen	353.2 / 4500D	PW, NW	P or G	1 bottle H_2SO_4 pH<2 Cool 4 C (NO_3); 1 bottle Cool 4 C (NO_2)	NO_3 - 28 days NO_2 - 48 hours	100mL (2)
	300.0	PW, NW	P or G	None	48 hours	100mL
	9056	S, SW	P or G	Cool 4 C	48 hours	25g
Nitrate/Nitrite-Nitrogen	353.2	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	100mL
Nitrite-Nitrogen	4500B	PW, NW	P or G	Cool 4 C	48 hours	100mL
	300.0	PW, NW	P or G	Cool 4 C	48 hours	100mL
	9056	S, SW	P or G	Cool 4 C	48 hours	25g
Nitrogen, Organic	Calculation	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	1000mL
	Calculation	S, SW	P or G	Cool 4 C	28 days	100g
Odor	2150B / 140.1	PW	G	None	24 hours	1000mL
Oil and Grease	1664	PW, NW	G	H_2SO_4 pH<2;Cool 4 C	28 days	1000mL (3)
	9070	LW	G	HCl pH<2;Cool 4 C	28 days	1000mL
	9071B	S, SW	G	Cool 4 C	28 days	100g
Orthophosphate	365.3	NW	P or G	Cool 4 C	48 hours	100mL
	4500-PE	PW, NW	P or G	Cool 4 C	48 hours	100mL
Osmotic Pressure	Interim Method	PW, NW	P or G	Cool 4 C	48 hours	100mL
Oxygen, Dissolved	360.1	PW, NW	G Bottle and Top	None	Analyze immediately	500mL
Paint Filter Liquids Test	9095	LW, S, SW	P or G	None	NA	100g or 100mL
Perchlorate	314	PW,NW	P or G	None	28 days	100mL
pH	150.1	PW, NW	P or G	None	Analyze immediately	50mL
	9040B	LW	P or G	None	Analyze immediately	50mL
	9045C	S, SW	P or G	None	Analyze immediately	50g
Phenols	420.2	NW	G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
	420.4	NW				
	420.4	S, SW	G	Cool 4 C	28 days	100g

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
Phosphorus, Polyhydrolizable	365.1	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
	365.1	S, SW	P or G	Cool 4 C	28 days	100g
Phosphorus, Soluble	365.1	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
	365.1	S, SW	P or G	Cool 4 C	28 days	100g
Phosphorus, Total	365.1	PW, NW	P or G	H_2SO_4 pH<2;Cool 4 C	28 days	500mL
	365.1	S, SW	P or G	Cool 4 C	28 days	100g
Reactive Cyanide	SW846 Section 7.3.3.2	S, SW	P or G	Cool 4 C	14 days	100g or 8 oz. jar
Reactive Sulfide	SW846 Section 7.3.4.2	S, SW	P or G	Cool 4 C	7 days	100g or 8 oz. jar
Residue, Filterable (TDS)	160.1/2540C	PW, NW	P or G	Cool 4 C	7 days	250mL
Residue, Nonfilterable (TSS)	160.2	PW, NW	P or G	Cool 4 C	7 days	250mL
Residue, Settleable	160.5/2540F	PW, NW	P or G	Cool 4 C	48 hours	1000mL
Residue, Total	160.3/2540B	PW, NW	P or G	Cool 4 C	7 days	250mL
	160.3/2540G	S, SW	P or G	Cool 4 C	7 days	100g
Residue, Volatile	160.4/2540E	PW, NW	P or G	Cool 4 C	7 days	250mL
	160.4/2540E	S, SW	P or G	Cool 4 C	7 days	100g
Silica, Dissolved	370.1/4500SiD	PW, NW	P	Cool 4 C	28 days	100mL
Specific Conductance	120.1/2510B	PW, NW	P or G	Cool 4 C	28 days	100mL
Sulfate	300.0	PW, NW	P or G	Cool 4 C	28 days	250mL
Sulfide	376.1	PW, NW	G	Zn Acetate; NaOH pH>9; Cool 4 C	7 days	500mL
	9034	S, SW	G	Zn Acetate; NaOH pH>9; Cool 4 C	7 days	50g Sludge 25g Dry
Sulfite	377.1	PW, NW	P or G	EDTA; Cool 4 C	Analyze immediately	500mL
Surfactants (MBAS)	5540C	PW, NW	P or G	Cool 4 C	48 hours	2L
Temperature	170.1	PW, NW	P or G	None	Analyze immediately	100mL
THM Formation Potential	502.2	PW	G, Teflon-lined septum	Cool 4 C; $Na_2S_2O_3$ HCl pH<2	14days	40mL
Total Organic Carbon	415.1/5310C	PW, NW	G	HCl; pH<2;Cool 4 C	28 days	125mL

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
Total Organic Halogen	9020B	PW, NW	G, Teflon-lined septum	H_2SO_4 pH<2; Cool 4 C	14 days	250mL (2)
	9023Modified	S, SW	G, Teflon-lined septum	Cool 4 C	14 days	100g
Turbidity	SM2130B	PW, NW	P or G	Cool 4 C	24 hours	100mL
UV254	5910B	PW, NW	1L amber	Cool 4 C	48 Hours	1L
MICROBIOLOGY						
Fecal Coliform	9222D	NW	P or G Sterile	Cool 4 C; 0.008% $Na_2S_2O_3$	30 hours-DW 6 hours-WW 8 hours-GW	125mL
Iron Bacteria	In-House	NW	P or G Sterile	Cool 4 C; 0.008% $Na_2S_2O_3$	24 hours	125 mL
Total Coliform	9223B	PW, NW	P or G Sterile	Cool 4 C; 0.008% $Na_2S_2O_3$	30 hours	125mL
Standard Plate Count	9215 B	PW, NW	P or G Sterile	Cool 4 C; 0.008% $Na_2S_2O_3$	8 hours	125mL
Streptococci, Fecal	9230C	NW	P or G	Cool 4 C; 0.008% $Na_2S_2O_3$	As soon as possible after collection	125mL
ORGANICS						
Separatory Funnel Extraction	3510C	NW, LW	G, Teflon-lined cap	Cool 4 C	7 days	1000mL (2)
Continuous Liquid/Liquid Extraction	3520C	NW, LW	G, Teflon-lined cap	Cool 4 C	7 days	1000mL (2)
Automated Soxhlet Extraction	3545	S, SW	G, Teflon-lined cap	Cool 4 C	14 days	100g or 8 oz. jar
Ultrasonic Extraction	3550B	S, SW	G, Teflon-lined cap	Cool 4 C	14 days	100g or 8 oz. jar
Soxhlet Extraction	3540C	S, SW	G, Teflon-lined cap	Cool 4 C	14 days	100g or 8 oz. jar
Volatiles P&T Extraction	5030B	NW, LW	G, Teflon-lined septum	$NaHSO_4$; Cool 4 C; pH<2	14 days	40mL (3)

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
Volatiles Closed-System P&T Extraction	5035	S, SW	Encore™ Sampler	Cool 4 C	48 Hours	3-5g Encores for Low Level 1-5g Encores for Med Level
			Preweigh – Low Level 1g Sodium Bisulfate, 5 mL Reagent Water in a 40 mL VOA jar with stir bar	Cool 4 C after filled with sample	Analyze 14 days after filled with sample	Low Level 2-Low Level Containers 1-Medium Level Containers
			Preweigh-Med Level 5mL MEOH in 40mL VOA vial with stir bar			Med Level 1-Medium Level Containers
			G, Teflon-lined septum	Cool 4 C	Preserve using Preweighed Containers ASAP	4 oz. Jar
Alcohols and Acetates	8015 Modified	LW	G,Teflon-lined septum	4°C	14 days	40mL (2)
		SW	G,Teflon-lined septum	4°C	14 days	4 oz
Haloacetic Acids	552.2	PW	G, Teflon-lined septum	Cool 4 C; Sodium Sulfite; HCl pH 4.5-5.0	14 days	40mL (2)
EDB/DBCP	504.1	PW	G, Teflon-lined septum	Cool 4 C; 3mg $Na_2S_2O_3$	14 days	40mL (2)
GC-Chlorinated Acids (Herbicides)	515.3	PW	G, Teflon-lined cap	Cool 4 C; 80mg $Na_2S_2O_3$	14 days/ 28 days	1000mL (2)
GC-Chlorinated Acids (Herbicides)	515.4	PW	G, Teflon-lined cap	Cool 4 C; 80mg $Na_2S_2O_3$	14 days/ 28 days	1000mL (2)
GC/MS-Solid Phase Extraction	525.2	PW	G, Teflon-lined cap	Cool 4 C; 80mg $Na_2S_2O_3$	14days	1000mL (2)
GC/MS-Endothall by Solid Phase Ion Exchange	548.1	PW	G, Teflon-lined cap	Cool 4 C; 80mg $Na_2S_2O_3$	7 days/14 days	1000mL (2)
HPLC-Glyphosate	547	PW	G, Teflon-lined cap	Cool 4 C; 80mg $Na_2S_2O_3$	14days	40mL (2)
GC-Herbicides	8151A	NW, LW	G, Teflon-lined cap	Cool 4 C	7 days/40 days	1000mL (2)
	8151A	S, SW	4 oz Plastic Jar	Cool 4 C	14 days/28 days	30g

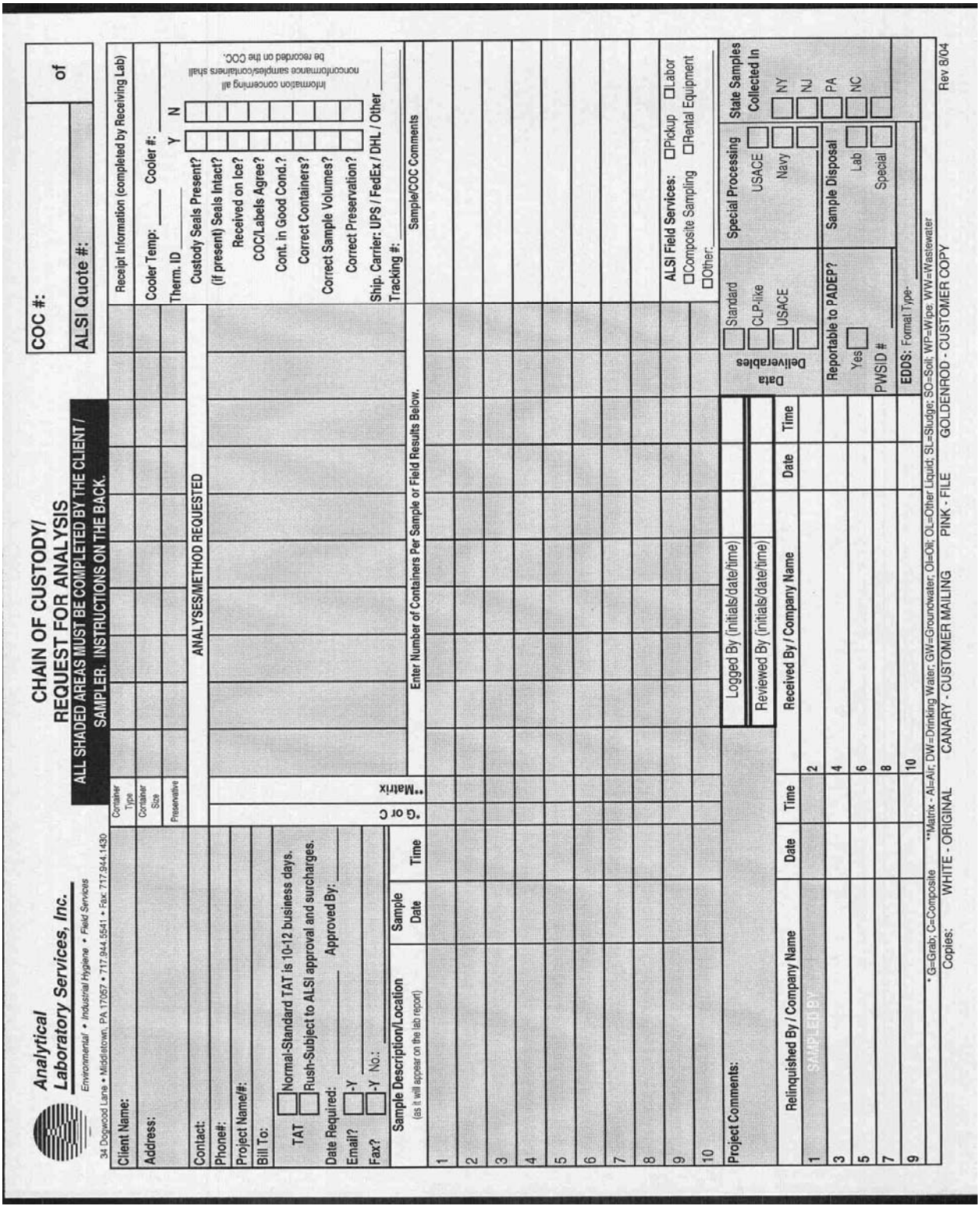
Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
GC-Organic Acids	Inhouse	NW	P or G zero headspace	None	28 days	250mL
GC-PCB Screening	508A	PW	G, Teflon-lined cap	Cool 4 C	14 days/30 days	1000mL (2)
GC-Pesticides	505	PW	G, Teflon-lined septum	Cool 4 C; 3mg $Na_2S_2O_3$	If Heptachlor is present 7 days, if not 14 days	40 mL (2)
GC-Pesticides	608 /8000 series/8081A	NW, LW	G, Teflon-lined cap	Cool 4 C; 5<pH<9	7 days/40 days	1000mL (2)
	8000 series/8081A	S, SW	G, Teflon-lined cap	Cool 4 C	14 days/40 days	100g or 8 oz. jar
GC-Pesticides	622	NW	1L Amber	Cool 4 C	7 days/40 days	1L
GC-PCBs	608 / 8000 series/8082	NW, LW	G, Teflon-lined cap	Cool 4 C; 5<pH<9	7 days/40 days	1000mL (2)
	8000 series/8082	S, SW	G, Teflon-lined cap	Cool 4 C	14 days/40 days	100g or 8 oz. jar
HPLC, PAHs	8000 Series/ 8310	NW, LW	G, Teflon-lined cap	Cool 4 C	7 days/40 days	1000mL (2)
	8000 Series/8310	S, SW	G, Teflon-lined cap	Cool 4 C	14 days/40 days	100g or 8 oz. jar
HPLC-Nitroaromatics and Nitramines (Explosives)	8000 Series/8330	NW, LW	G, Teflon-lined cap	Cool 4 C	7 days/40 days	1000mL (2)
	8000 Series/8330	S, SW	G, Teflon-lined cap	Cool 4 C	14 days/40 days	100g or 8 oz. jar

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
GC, Nitrogen Phosphorus Pesticides	8000 Series/8141A	NW	G, Teflon-lined cap	Cool 4 C 5<pH<8	7 days/40 days	1000mL (2)
	8000 Series/8141A	S, SW	G, Teflon-lined cap	Cool 4 C 5<pH<8	14 days/40 days	100g or 8 oz. jar
GC-Pesticides/PCBs, Chlorinated	508	PW	G, Teflon-lined cap	Cool 4 C; 80mg/L $Na_2S_2O_3$	7 days/14 days	1000mL (2)
GC-Pesticides, Nitrogen Phosphorus	507	PW	G, Teflon-lined septum	Cool 4 C; 80mg/L $Na_2S_2O_3$	14 days/14 days	1000mL (2)
Diquat and Paraquat	549.2	PW	PVC high-density	Cool 4 C; 80mg/L $Na_2S_2O_3$	7 days/21days	1000mL
GC-Purgeable Aromatics	602/8021B	NW	G, Teflon-lined septum	$Na_2S_2O_3$; HCl pH<2;Cool 4 C	14 days	40mL (2)
	8021B	S, SW	See Method 5035	Cool 4 C	14 days	See Method 5035
	601	NW	G, Teflon-lined septum	$Na_2S_2O_3$; Cool 4 C	14 days	40mL (2)
GC-Purgeable Organics	502.2	PW	G, Teflon-lined septum	3mg/40mL $Na_2S_2O_3$; Cool 4 C; HCl pH<2	14 days	40mL (2)
	601/602 modified	NW	G, Teflon-lined septum	$Na_2S_2O_3$;HCl pH<2;Cool 4 C	14 days	40mL (2)
GC-Non-Halogenated Organics	8015B	NW	G, Teflon-lined septum	$Na_2S_2O_3$;HCl pH<2;Cool 4 C	14 days	40mL (2)
		S, SW	See Method 5035	Cool 4 C	14 days	See Method 5035
GC-Purgeable Organics	8021B	NW	G, Teflon-lined septum	$Na_2S_2O_3$;HCl pH<2;Cool 4 C	14 days	40mL (2)
		S, SW	G, Teflon-lined septum	Cool 4 C	14 days	100g or 8 oz. jar
GC/MS-Purgeable Organics	524.2	PW	G, Teflon-lined septum	Cool 4 C;HCl pH<2; 25 mg Ascorbic Acid	14 days	40mL (2)
	624/ 8260B	NW	G, Teflon-lined septum	$Na_2S_2O_3$;HCl pH<2;Cool 4 C	14 days	40mL (2)
	8260B	S, SW	See Method 5035	Cool 4 C	14 days	See Method 5035
GC/MS-Semivolatiles	625/ 8000 series/8270C	NW	G, Teflon-lined septum	$Na_2S_2O_3$;Cool 4 C	7/40 days	1000mL (2)

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
	8000 series/ 8270C	S, SW	G, Teflon-lined septum	Cool 4 C	14/40 days	100g or 8 oz. jar
HPLC-Carbamates	531.1	PW	G, Teflon-lined cap	Mono-chloroacetic Acid to pH 3; 80 mg/L $Na_2S_2O_3$ Cool 4 C	28 days	40mL (2)
TOTAL PETROLEUM HYDROCARBONS						
Total Petroleum Hydrocarbons (TPH)	1664	NW	G	Cool 4 C; pH<2, H2SO4	28 days	1000mL (2)
Total Petroleum Hydrocarbons (TPH)	418.1	PW, NW	G	Cool 4 C; pH<2, H2SO4	28 days	1000mL (2)
Total Petroleum Hydrocarbons (TPH)	418.1 modified	S, SW	G	Cool 4 C	28 days	100g or 8 oz. jar
Total Petroleum Hydrocarbons as Diesel	8000 series/ 8015B	PW, NW	G	Cool 4 C; pH<2, H2SO4	14 days/40 days	1000mL (2)
	8000 series/ 8015B	S, SW	G	Cool 4 C	14 days/40 days	100g or 8 oz. jar
Total Petroleum Hydrocarbons as	8000 series/8015B	PW, NW	G, Teflon-lined septum	Cool 4 C, pH<2 HCl	14 days	40mL (2)
Gasoline	8000 series/8015B	S, SW	See Method 5035	Cool 4 C	14 days	See Method 5035
LEACHING PROCEDURES						
EP Toxicity Metals	1310A	LW	P or G	Cool 4 C	28 days to leaching	1000mL
	1310A	S, SW	P or G	Cool 4 C	28 days to leaching	100g
TCLP Metals Only	1311	LW	P or G	Cool 4 C	28 days to leaching	1000mL
		SW	P or G	Cool 4 C	28 days to leaching	500g
GC/MS-TCLP Volatile Organics	1311	LW	G, Teflon-lined septum	Cool 4 C	14 days to leaching	200mL
		SW	G, Teflon-lined septum	Cool 4 C	14 days to leaching	100g
GC/MS-TCLP Semivolatiles	1311	LW	G, Teflon-lined cap	Cool 4 C	14 days to leaching	3000mL
		SW	G, Teflon-lined cap	Cool 4 C	14 days to leaching	500g
SPLP Metals Only	1312	LW	P or G	Cool 4 C	28 days to leaching	1000mL

Description	Method	*Matrix PW-Potable Water NW-Nonpotable Water LW-Liquid Waste S-Soil, SW-Solid Waste	Sample Container	Preservation	Prep/ Analysis Holding Time	Minimum Volume
SPLP Metals Only	1312	SW	P or G	Cool 4 C	28 days to leaching	500g
GC/MS-SPLP Volatile Organics	1312	LW	G, Teflon-lined septum	Cool 4 C	14 days to leaching	200mL
GC/MS-SPLP Volatile Organics	1312	SW	G, Teflon-lined septum	Cool 4 C	14 days to leaching	100g
GC/MS-SPLP Semivolatiles	1312	LW	G, Teflon-lined cap	Cool 4 C	14 days to leaching	3000mL
		SW	G, Teflon-lined cap	Cool 4 C	14 days to leaching	500g

Appendix C – Chain Of Custody Record



Analytical Laboratory Services, Inc.
Environmental • Industrial Hygiene • Field Services
34 Dogwood Lane • Middletown, PA 17057 • 717.944.5541 • Fax: 717.944.1430

CHAIN OF CUSTODY/
REQUEST FOR ANALYSIS
ALL SHADED AREAS MUST BE COMPLETED BY THE CLIENT / SAMPLER. INSTRUCTIONS ON THE BACK.

COC #: of
ALSI Quote #:

Client Name:
Address:
Contact:
Phone#:
Project Name/#:
Bill To:
TAT ☐ Normal-Standard TAT is 10-12 business days. ☐ Rush-Subject to ALSI approval and surcharges.
Date Required: Approved By:
Email? -Y
Fax? -Y No.:

Container Type / Container Size / Preservative

ANALYSES/METHOD REQUESTED

Receipt Information (completed by Receiving Lab)
Cooler Temp: Cooler #:
Therm. ID Y N
Custody Seals Present?
(If present) Seals Intact?
Received on Ice?
COC/Labels Agree?
Cont. in Good Cond.?
Correct Containers?
Correct Sample Volumes?
Correct Preservation?
Ship. Carrier: UPS / FedEx / DHL / Other
Tracking #:
Information concerning all nonconformance samples/containers shall be recorded on the COC.

Sample Description/Location (as it will appear on the lab report)	Sample Date	Time	G or C	"Matrix	Enter Number of Containers Per Sample or Field Results Below.	Sample/COC Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						

Project Comments:

Logged By (initials/date/time)
Reviewed By (initials/date/time)

Data Deliverables: ☐ Standard ☐ CLP-like ☐ USACE

ALSI Field Services: ☐Pickup ☐Labor ☐Composite Sampling ☐Rental Equipment ☐Other:

Special Processing: ☐ USACE ☐ Navy

State Samples Collected In: ☐ NY ☐ NJ

Relinquished By / Company Name	Date	Time	Received By / Company Name	Date	Time
SAMPLED BY			2		
3			4		
5			6		
7			8		
9			10		

Reportable to PADEP? ☐ Yes ☐
PWSID # _____

Sample Disposal: ☐ Lab ☐ Special

☐ PA ☐ NC

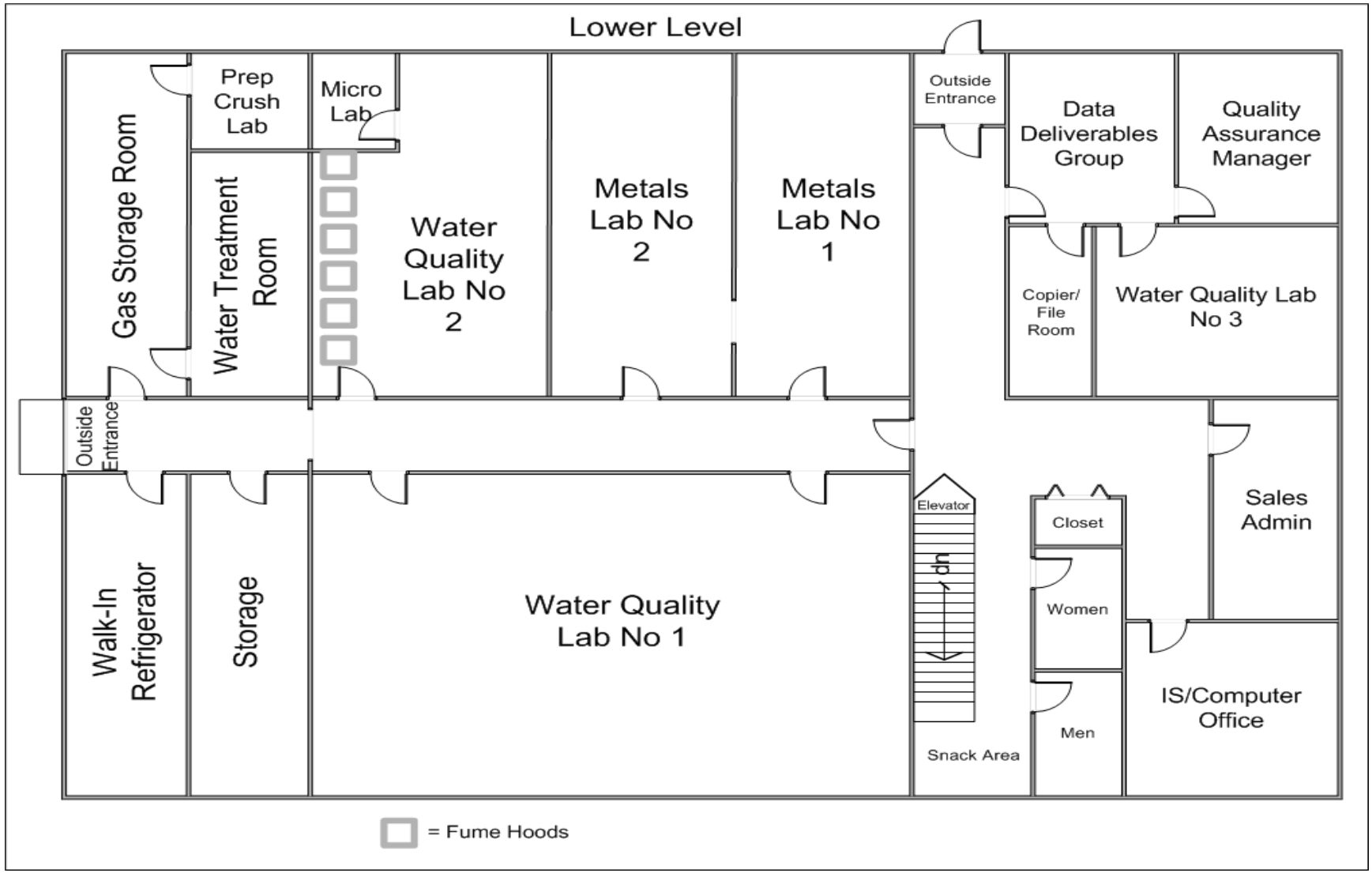
EDDS: Format Type-

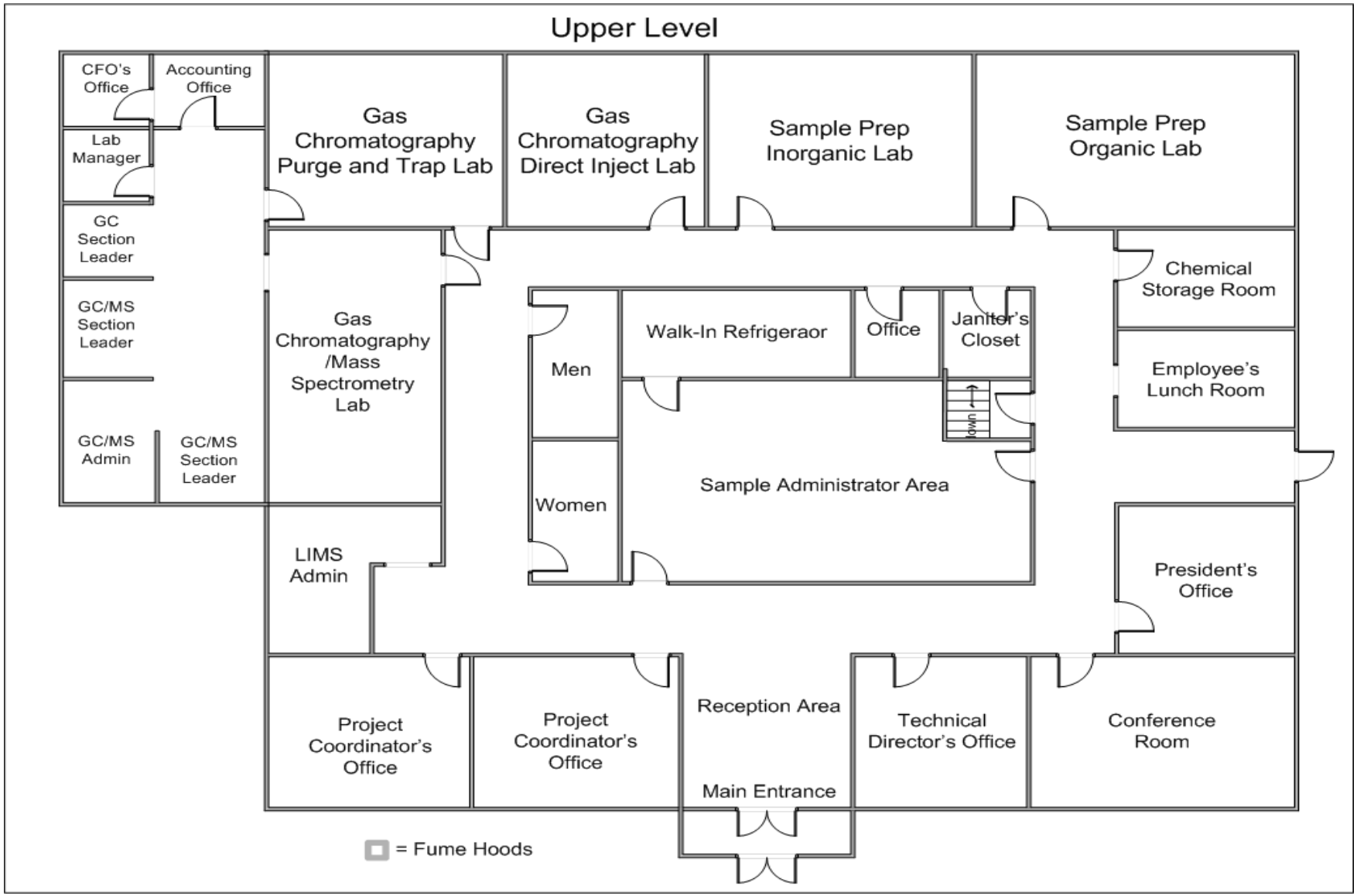
* G=Grab; C=Composite **Matrix - Al=Air; DW=Drinking Water; GW=Groundwater; Oil=Oil; OL=Other Liquid; SL=Sludge; SO=Soil; WP=Wipe; WW=Wastewater

Copies: WHITE - ORIGINAL CANARY - CUSTOMER MAILING PINK - FILE GOLDENROD - CUSTOMER COPY

Rev 8/04

Appendix D – Laboratory Floor Plans




Upper Level
CFO's Office
Accounting Office
Lab Manager
GC Section Leader
GC/MS Section Leader
GC/MS Admin
GC/MS Section Leader
Gas Chromatography Purge and Trap Lab
Gas Chromatography Direct Inject Lab
Sample Prep Inorganic Lab
Sample Prep Organic Lab
Gas Chromatography /Mass Spectrometry Lab
Men
Women
Walk-In Refrigeraor
Office
Janitor's Closet
Down
Sample Administrator Area
Chemical Storage Room
Employee's Lunch Room
LIMS Admin
President's Office
Project Coordinator's Office
Project Coordinator's Office
Reception Area
Main Entrance
Technical Director's Office
Conference Room
= Fume Hoods

Appendix E – Instrument List

Instrument	Manufacturer	Model No.	Analysis	Serial No.	Acquisition Date
LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)					
LIMS Software and Data System	Chemware Horizon		All Sections	Licensed Agreement	1996
Unix Server	Hewlett Packard	9000 E45	All Sections	3543A88230	1996
ORGANICS					
Hewlett Packard Chem Server 4920 Organic Data System	Hewlett Packard	4920	Organics/GC-GC/MS/HPLC	3240A80098	1995
Hewlett Packard Environ Unix Terminals (3)	Hewlett Packard		Organics/GC-GC/MS/HPLC	CA46391044 CA46391094 CA46391070	1995 1995 1995
Network Hub	Hewlett Packard	J2602A	Organics/GC-GC/MS/HPLC	SG5200272	1995
NIST 75,000 Compound Reference Spectra Library	Hewlett Packard	59943L	Organics/GC-GC/MS/HPLC	3525A70197	1995
GAS CHROMATOGRAPHY/MASS SPECTROMETRY					
Purge and Trap Concentrator with Autosampler and Sample Heater (MS01)	Tekmar/EST	2000/Archon EST	GC/MS	3518A10324/13449	1999
Gas Chromatograph/Mass Selective Detector (MS01) (with EI, packed or capillary columns, and autoinjector)	Hewlett Packard	5890/5970	GC/MS-VOA	2950A26771/3004A12574	1986
Gas Chromatograph/Mass Selective Detector (MS02)	Hewlett Packard	5890/5971	GC/MS-VOA	3033A31928/N.A.	2001
Gas Chromatograph/Mass Selective Detector (MS03) with Purge and Trap Concentrator/Autoinjector	Hewlett Packard	5890/5972	GC/MS-VOA	3336A59812/3501A02569	1995
Purge and Trap Concentrator with Autosampler and Sample Heater (MS03)	Tekmar/EST	3000/Archon EST	GC/MS	93133003/13137	1986
Gas Chromatograph/Mass Selective Detector (MS04) with Purge and Trap Concentrator	Hewlett Packard	5890/5972	GC/MS-BNA	US00662866/3549A03337	1997
Gas Chromatograph/Mass Selective Detector (MS05) (with EI and capillary column) with Autoinjector	Hewlett Packard	5890/5971	GC/MS-VOA	3140A38964/3022A01159	2001

Instrument	Manufacturer	Model No.	Analysis	Serial No.	Acquisition Date
Purge and Trap Concentrato with Autosampler and Sample Heater (MS05)	Tekmar/EST	3000/Archon EST	GC/MS	90169022/13679	1990
Gas Chromatograph/Mass Selective Detector (MS06)	Hewlett Packard	5890/5971A	GC/MS-BNA	N.A./N.A.	2000
Gas Chromatograph/Mass Selective Detector (MS07) (with EI and capillary column) with Autoinjector	Hewlett Packard	5890/5971	GC/MS-VOA	3133A37488/3118A02504	1992
Purge and Trap Concentrator with Autosampler and Sample Heater (MS07)	Tekmar/EST	2000/Archon EST	GC/MS	90163013/12543	1999
Gas Chromatograph/Mass Selective Detector (MS09)	Hewlett Packard	6890/5973	GC/MS-BNA (DW)	US00027847/US92522731	2000
Gas Chromatograph/Mass Selective Detector (MS11)	Finnigan	Trace CC/Trace DSQ	GC/MS	20033754/100084	2003
Purge and Trap Concentrator with Autosampler and Sample Heater (MS11)	Tekmar/EST	Velocity XPT/ Archon EST	GC/MS	US03150011/13918	2003
GAS CHROMATOGRAPHY					
Dynatech Archon	Dynatech	5100	GC	11767-695	1995
Gas Chromatograph (with TCD) (GC-1)	GOW-MAC	550	GC-Organic Acids	E63605	1991
Gas Chromatograph (with PID/ELCD) (GC-2)	Hewlett Packard	6890	GC-VOA	US00021496	1999
Gas Chromatograph (with dual ECD) (GC-4)	Hewlett Packard	5890	GC-PCBs/504.1	3310A49263	1997
Gas Chromatograph (with ECD) (GC-5)	Hewlett Packard	5890	GC-Herbicides/504/Pest/PCBs	3140A47535	1997
Gas Chromatograph (GC-7)	Hewlett Packard	5890	GC-Capillary Column	3234A00311	1997
Gas Chromatograph (with PID/FID) (GC-8)	Hewlett Packard	5890II	GC-BTEX/GRO	3203A40203	1994
Gas Chromatograph (with ECD/ELD) (GC-9)	Hewlett Packard	5890II	GC-Herbicides/Pest/PCBs/507	2950A27674	2004
Gas Chromatograph (with ECD/NPD) (GC-10)	Hewlett Packard	5890/7673	GC-Pesticides/PCBs/Herbicides	3223A42304	1992
Gas Chromatographs (with FID/FID) (GC-11)	Hewlett Packard	5890	GC-TPH/DRO	2541A08247	1990
Gas Chromatograph (with PID/ELCD) (GC-12)	Hewlett Packard	5890II	GC-VOA	3235A44488	1994
Gas Chromatograph (NPD/NPD) (GC-13)	Hewlett Packard	5890	GC	2415801131	2003

Instrument	Manufacturer	Model No.	Analysis	Serial No.	Acquisition Date
Gas Chromatographs (with ECD/ECD) (2) (GC-14)	Hewlett Packard	5890	GC-Pesticides/PCBs	3235A44019	1995
Gas Chromatograph Data System	PE Nelson	Turbochrom 4.1	GC	95074101484/95074101485	1995
Purge and Trap Concentrators (4) with Autosampler and Sample Heater	Tekmar	LSC2000/ALS2016	GC	9203001/921600890296013/9030201592014003	19921990
Purge and Trap Concentrators (4) with Autosampler	Tekmar	LSC2/ALS	GC	87042001/307 87042002/1205	1987 1987
Purge and Trap Concentrator	Tekmar	LSC2	GC	88197004	1988
Gas Chromatograph (with FID)	Tracor	540	GC-Screen - Packed Column	851286	1989
Gas Chromatograph (with PID/ELCD/FID)	Waters	Dimension I	GC-BTEX/GRO	N.A.	1997
HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)					
Waters Carbamate System	Waters	600E	HPLC	6PLEP6539	1991
Waters 486 Tunable UV Detector	Waters	486	HPLC	486-PRD499	1991
Waters 470 Scanning Fluorescence Detector	Waters	470	HPLC	470-001705	1991
METALS					
Microwave Digestion System	Milestone	Ethos 900 Plus	Metals Digestion	123635	2001
Autosampler	Varian		GF Metals	0061369	1990
Inductively Coupled Plasma-Mass Spectroscopy	Perkin Elmer	ELAN 6000	ICP-MS Metals	182960360	2001
Graphite Furnace Zeeman Background	Varian	400Z	GF Metals	0061071	1990
Inductively Coupled Plasma, IRIS Advantage Dual-View	Thermo-Jarrell Ash	IRIS	ICP Metals	N.A.	2000
with 300-Place Autosampler	Thermo-Jarrell Ash	300	ICP Metals	N.A.	2000
Mercury Analyzer	PSA	10.45	Mercury	007	2002
Mercury Analyzer	Leeman Labs	PS200	Mercury	3029110-00012	1996
Microwave Digestion System	CEM Corporation	MDS-2100	Metals Digestion	WR7089	1994
Inductively Coupled Plasma, Simultaneous	Thermo-Jarrell Ash	61E	ICP Metals	278490	1994
Hot Water Bath	Precision Scientific	180 Series	Wet Chem/Hg Digestion	N.A.	2003
CPI Mod-Block (Hot Block)	CPI		Metals Digestion	N.A.	1999
WET CHEMISTRY					
AA3 Autoanalyzer	Bran-Luebbe	AA3	Wet Chem	54449525/9528237	2002

Instrument	Manufacturer	Model No.	Analysis	Serial No.	Acquisition Date
Autoclave	Market Forge	Sterilmatic	Wet Chem	GM2304	1993
Balance	Mettler	AE100	Wet Chem	B94827	1986
Balance	Mettler	PM4800	Wet Chem	1113242887	1994
Centrifuge	Becton Dickinson	Dynac 420101	Wet Chem	2850029	1994
COD Digestor	HACH	45600	COD	920700007511	1995
COD Digestor	HACH	45600	COD	920600007361	1994
COD Digestor	HACH	16500	COD	880811313	1988
Colony Counter	Biotech	Biotech	Microbiology	N.A.	1986
Conductivity Meter	Orion	160	Wet Chem	53076047	1995
Digestor	Tecator	1015	Wet Chem	N.A.	1995
Digestor	LabConco	25Place	Wet Chem	010695394E	2001
Distillation Unit	Buchi	323	Ammonia-Nitrogen	N.A.	1993
Incubators (2)	VWR	2020, 2030	Wet Chem	0900290	1992
Incubator	American Scientific	IS-61	Microbiology	N.A.	1986
Incubator Bath	Precision Scientific	251	Microbiology	N.A.	1986
Infrared Spectrophotometer	Buck Scientific	404	TPH	634	1996
Ion Chromatographer with:	Dionex	DX-500	300.0/300.1/314.0	N.A.	2004
Autosampler	Dionex	AS40	300.0/300.1/314.0	98100646	1999
Computer	Dell	D1028L	300.0/300.1/314.0	84779-A1VJY	1999
Ion Chromatographer with:	Dionex	DX-120	300.0/300.1/314.0	00110303	2000
Autosampler	Dionex	AS40	300.0/300.1/314.0	00100638	2000
Ion Analyzer	Orion	EA940	Wet Chem	N.A.	1995
Ion Analyzer	Orion	EA920	Wet Chem	N.A.	1996
Lachat QuickChem with XYZ Autosampler and Autodiluter	Lachat		Anions	2000-600/2000-479/2000-350	1993
Microscope	Olympus	BHT2	Asbestos	022785/229024	1988
Muffle Furnace	Vulcan	3-1750	Wet Chem	N.A.	1996
Osmometer	Advanced Instruments	3W2	Wet Chem	38948	1990
Oven	Lab-Line	Imperial V	Wet Chem	N.A.	1988
Oven	Lab-Line	Imperial IV	Wet Chem	N.A.	1988
Pensky Martin Closed Cup Flash Point Apparati (2)	Pensky Martin	TA6	Wet Chem	108A-2	1991
pH Meters (3)	Corning	350	Wet Chem	1868	1988

Instrument	Manufacturer	Model No.	Analysis	Serial No.	Acquisition Date
pH Meter	Fisher	825	Wet Chem	495	1995
Quanti-Tray Sealers (2)	IDEXX	2020	Microbiology	N.A.	1996
Rapidstill (2)	Labconco	Rapidstill II	Wet Chem	N.A.	1996
Ultrasonic Cleaners (2)	Branson	2200, 2210	Wet Chem	N.A.	1988, 1994
SmartChem	Westco		Wet Chem		2002
Specific Conductance Meter	YSI		Wet Chem	1552	1990
Spectrophotometer	HACH	DR2000	COD	930500024601	1994
TOC Autosampler	Dohrman	ASM-1	TOC	HD1680	1988
TOC Autosampler	Shimadzu	ASI5000A	TOC	N.A.	1996
Total Organic Carbon Analyzer	Shimadzu	5000A	TOC	N.A.	1996
Total Organic Carbon Analyzer	Dohrman	DX-20A	TOC	HF1667	1986
Total Organic Carbon Analyzer	Shimadzu	SSM-5000A	TOC	40440748	2003
Total Organic Halogen Analyzer	Mitsubishi	TOX-10	TOX	43C32231	1991
Total Organic Halogen Analyzer	Mitsubishi	TOX-10E	TOX	75R03775	1991
Turbidimeter	HF Scientific	DRT 100B	Wet Chem	18485	1994
UV Spectrophotometer	Milton Roy	501	Wet Chem	N.A.	1998
UV/Visible Spectrophotometer	Shimadzu	UV-1201	Wet Chem	30034J	1997

Appendix F – Change History Form

Section No	Section	Reason for Change
7.6	Data Deliverable Packages	PADEP Audit 2004 Corrective Action
7.7	Quality Assurance Documents, Item 2,6,9	PADEP Audit 2004 Corrective Action
7.8	External Documents	PADEP Audit 2004 Corrective Action
8.1	Reagents	Change of LIMS System (Element to Horizon)
8.2	Reference Standards	Change of LIMS System (Element to Horizon)
8.5	Balances	Class S weights changed to Class 1
10.1	Laboratory Capacity Review	PADEP Audit 2004 Corrective Action
14.3.1	Test Scheduling	Change of LIMS System (Element to Horizon)
14.3.2	Record of Analysis	Change of LIMS System (Element to Horizon)
14.3.3	Preps Performed Entry	Change of LIMS System (Element to Horizon)
14.3.4	Reporting Results	Change of LIMS System (Element to Horizon)
15.1.2	Bottle Preservation (paragraph 3)	Change of LIMS System (Element to Horizon)
15.1.3	Holding Time	Change of LIMS System (Element to Horizon)
15.1.4	Turnaround Time	Change of LIMS System (Element to Horizon)
15.2.1	Chain of Custody Form (paragraph 2)	Change of LIMS System (Element to Horizon)
15.2.4	Sample Identification and Control	Change of LIMS System (Element to Horizon)
17.4	Quality Control Charts	Change of LIMS System (Element to Horizon)
17.6	Data Reduction, Validation and Reporting	Change of LIMS System (Element to Horizon)
17.7	Data Deliverable Reporting	PADEP Audit 2004 Corrective Action
22.2	Internal Audits	PADEP Audit 2004 Corrective Action

Figure 1 – Reagent Logbook

CHEMICAL NO.	CHEMICAL NAME	MFGR.	LOT NO.	DATE RECVD	*DATE EXPIRED	RCVR'S INITIALS	P.O. NO.	STORAGE LOCATION	NO. OF CONTAINERS
*NOTE: Include the expiration date, if indicated on the bottle									

Figure 2 – Quality Verification Data Form

Quality Verification Data

Reagent ID # and description: _____

Manufacturer: _____

Lot #: _____

Laboratory use: _____

Requested analysis: _____

Date submitted for analysis: _____

Technician submitting aliquot / department: _____

Comments:

_____

_____

_____

_____

Analytical Results:

_____

_____

_____

_____

Analysts Approval (initials and date):

_____

_____

_____

Revision 2/97

Figure 3 – Proposal Request Form

Analytical Laboratory Services, Inc.
PROPOSAL REQUEST FORM

Prepared by	Date of Request: Date Required:
Proposal No	Client Contact:
Client Name	Phone No.: ☐Fax to:
Address ***Site Name*** Government Contract No. New Client ☐ (PC assigned by Sue Baer upon contract award) Existing Client ☐ PC: Standard Discount:	One-Time Job ☐ No. of Samples: Date of Arrival: Annual Contract ☐ Weekly☐ Monthly☐ Quarterly☐ Annual☐ Other ***Turnaround Time*** ☐Routine—10 business days ☐Rush—Due: If rush, Approved by:

Project Description

☐***Samples collected from what State: _____*** ☐***Special Project Description (see attached)*** ☐***SOW (see attached)*** ☐ ***Special Detection Limits (see attached)***

If samples are collected from NY state, fill out the Addendum to the Proposal Request Form.

Project Notes:

Reporting Requirements ☐J-Values ☐Standard Deliverables ☐Specific Deliverables Page Ref: Due Date: ☐CLP-like ☐USACE ☐Raw Data ☐ NJ-Reduced ☐Saic Other- ☐EDDs: ☐Excel-Std ☐ EQuIS ☐GISkey ☐Landlinks ☐SEDD ☐Other	Subcontracted Analysis ☐ None for this job Test(s): Sub'd to: Test(s): Sub'd to: Test(s): Sub'd to: Test(s): Sub'd to:

Field Services ☐ALSI Pickup/Delivery ☐Client Delivery ☐FedEx/UPS ☐ALSI Sampling ☐Client Sampling Sampling Date(s): Expected Day(s) of Receiving:	***QC Requirements*** ☐Trip Blank ☐Field Blank ☐Equipment Blank ☐Rinsate Blank ☐Field Duplicate ☐MS/MSD ☐Method Specific ☐Other
Certification Requirements	***Sample Disposal*** ☐Standard ☐Special—No. of days:
Pricing Review-- Manager Review (required for all proposals over $10,000): Pricing Accurate? ☐Yes ☐No Sufficient Equipment available to perform the job? ☐Yes ☐No Sufficient Personnel available to perform the job? ☐Yes ☐No	
Comments	***Manager's Signature/Date:*** ***Michael S. Farlling*** Date:

Addendum to Proposal Request Form

Checklist for Samples Received from New York State

Note: The State of New York charges a fee for all samples collected and analyzed from the State of New York. Furthermore, New York has the right to audit this laboratory's accounting system for tracking all NY samples received. If discrepancies occur during an audit, a monetary fine may be assessed and ALSI could jeopardize their certification in the State of New York.

To insure proper handling of all samples received from the State of New York, please fill out this form, initial the checklist, and attach to the ALSI Proposal Request Form to ensure that all information regarding NY samples is completed and retained in the client file for future reference. If you have any questions or need assistance, please see the QA Manager.

Client Name:_____ Client Contact Name:_____

Project ID:_____ Phone No.: _____

Site Location: _____ Project Start Date: _____

ALSI Proposal No. _____ ALSI Contact: _____

Initial Here

_____ Are the samples from New York State: YES or NO

_____ If samples are from NY State, are they being taken from Federal Property. (i.e., Indian Reservation, Federal Building, Federal Landmark)? YES or NO If yes, indicate Federal Property:
_____.

_____ Indicate the sample type (i.e., DW, WW, SHW): _____.

_____ The most recent NY certificates were reviewed to confirm certification for the required analytes.
YES or NO If yes, who reviewed the certification: _____.

_____ NY certification was confirmed for the methods requested. YES or NO If yes, who confirmed the methods requested: _____.

_____ The client was directed to complete the chain-of-custody in accordance with ALSI requirements and to complete the information regarding NY samples and any required deliverable requirements.
YES or NO

Figure 4 – Preservation Logbook

DATE	SAMPLE NO.	TEST	PRESERVATION	pH RECEIVED	ADJUSTMENT AMOUNT ADDED	FINAL pH	RESIDUAL CHLORINE (+ or -)	Preservation Lot #	TURBIDITY (N.T.U.)	SULFIDE	INITIALS
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
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					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
					(mL)						
Turbidimeter was calibrated using 0.02 and 8.00 standards					Date:________		Page No.:				
					Tech:________						

Figure 5 – Corrective Action Form

ALSI Corrective Action Report (CAR)

To be Completed by Originator and Submitted to Customer Service

Date:_____ CAR Submitted By:_____
Dept:_____ Supervisor:_____

Chain of Custody No.(s):

Method:_____

Problem/Reason for Corrective Action:

___ Login Error (Double reviewed by:_____)
___ Incorrect Preservative
___ Missed Holding Time
___ Calibration failure
___ QC failure due to sample matrix
___ QC failure NOT due to sample matrix
___ Sample Lost in process
___ Reporting Error
___ Other_____

Explanation of Problem:

_____

Signature(s), where Applicable:

Analyst:_____
1st Review:_____
2nd Review:_____
Result Entered By:_____
Result Approved By:_____
Supervisor:_____

Immediate Corrective Action Taken: By:_____ Date:_____

_____

Are there policies/procedures in place to prevent this from occurring? Yes ____ No ____ Not Applicable ____

If Yes, What is the procedure, and why didn't it work?

If No, What procedure is needed to prevent this from occurring?

Target Date for Implementation:_____ Person Responsible for Implementation:_____

Customer Service Rep. Modified: _____ Date: _____

To be Completed by Customer Service and Submitted to QA

Date:_____ Customer Service Rep.:_____ Client Contacted? Yes ____ No ____

Project(s) / Client(s) Involved: Date:_____ Person:_____

Action Taken (if no action taken, explain why):

Laboratory Notified of Client Response? Yes ___ No ___ Not Applicable ___

To be Completed by QA and Dept.

Was action taken appropriate? Yes ___ No ___ Date:_____

Is further action necessary? Yes ___ No ___ QA Initials:_____

If Yes, What action is needed?

Modification of SOP needed? Yes ____ No ____

If Yes, What is the procedure? Has it been modified? Yes ___ No ___

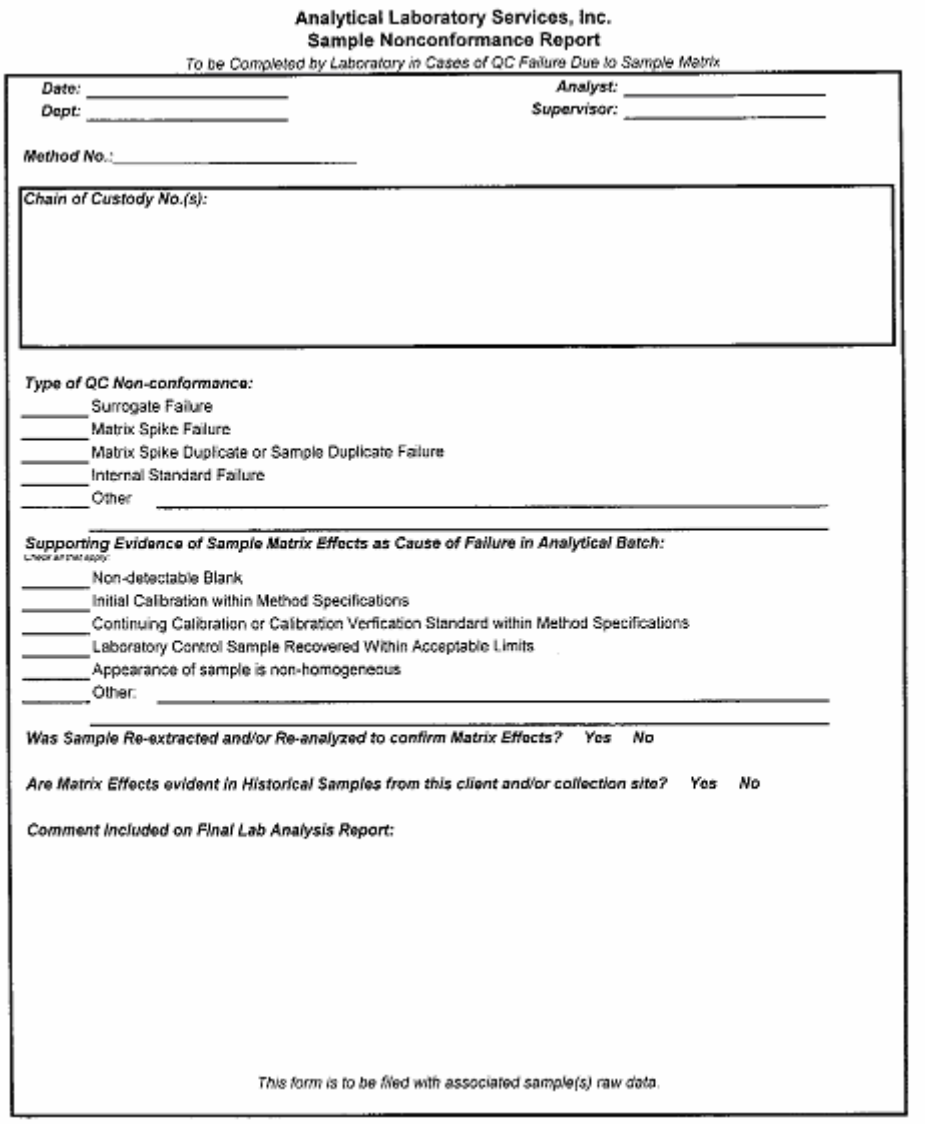
Figure 6 – LIMS Internal Chain of Custody

ALSI INTERNAL CHAIN OF CUSTODY

Storage Area:_____

Sample #	Prep Type	Relinquished By:	Date	Time	Received By:	Date	Time	Purpose:	Subsample Formed?	Prep Type

Figure 7 – Sample Non Conformance Report



Analytical Laboratory Services, Inc.
Sample Nonconformance Report

To be Completed by Laboratory in Cases of QC Failure Due to Sample Matrix

Date: _____ ***Analyst:*** _____

Dept: _____ ***Supervisor:*** _____

Method No.: _____

Chain of Custody No.(s):

Type of QC Non-conformance:

_____ Surrogate Failure
_____ Matrix Spike Failure
_____ Matrix Spike Duplicate or Sample Duplicate Failure
_____ Internal Standard Failure
_____ Other _____

Supporting Evidence of Sample Matrix Effects as Cause of Failure in Analytical Batch:

Check all that apply:

_____ Non-detectable Blank
_____ Initial Calibration within Method Specifications
_____ Continuing Calibration or Calibration Verification Standard within Method Specifications
_____ Laboratory Control Sample Recovered Within Acceptable Limits
_____ Appearance of sample is non-homogeneous
_____ Other: _____

Was Sample Re-extracted and/or Re-analyzed to confirm Matrix Effects? ***Yes*** ***No***

Are Matrix Effects evident in Historical Samples from this client and/or collection site? ***Yes*** ***No***

Comment Included on Final Lab Analysis Report:

This form is to be filed with associated sample(s) raw data.

Figure 8 – External Complaint Form

External Complaint Form

Please be as specific and complete as possible.

Customer Name:		Complaint File Number:	
Client Contact:		Specific Job/Phase/Task:	
Date/Time:		Applicable COC Numbers:	
Person Making Complaint:		ALSI Contact:	

Specific Items Discussed:

Resolution:

Departments Involved with Complaint:

☐ Biomonitoring	☐ Microbiology		
☐ Client Services	☐ Prep Lab		
☐ Field Services	☐ QA/QC		
☐ GC/MS	☐ Sample Receiving		
☐ GC (Direct Inject)	☐ TOX/TOC		
☐ GC (Volatiles)	☐ Wet Chemistry	Report Reviewed By:	
☐ Metals	☐ Miscellaneous	Customer Service Informed:	

Figure 9 – Internal Complaint Form

ALSI INTERNAL COMPLAINT FORM

Employee Initiating Complaint:	**Specific Job/Phase/Task:** **Date:**
Complaint:	
Resolution:	
Complaint Received By:	**Complaint Reviewed By:**
Complainant Signature:	

Rev. 2/03

Document Title: Nitroaromatics and Nitramines by HPLC with Ultraviolet Detection

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Alan Lopez, Date
Laboratory Technical Manager

TABLE OF CONTENTS

1 Scope and Application....3
2 Summary of Method....4
3 Interferences....4
4 Safety....4
5 Apparatus and Materials....5
6 Reagents....5
7 Instrument Calibration....9
8 Quality Control....11
9 Sample Collection, Preservation and Handling....15
10 Procedure....16
11 Reporting Results....18
12 Waste Disposal....18
13 Pollution Prevention....18
14 Definitions....19
Table 1: QA Acceptance Criteria....20
Appendix A: Sample Logbook Page....21
SOP Change History Summary....22
SOP Concurrence Form....23

1 Scope and Application

1.1 This standard operating procedure is adapted from EPA Method 8330, SW 846, "Test Methods for Evaluating Solid Waste". The method detection limits (MDL) can be found in the current HPLC method detection limit folder. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.2 This method is used to determine nitroaromatics and nitramines in a variety of solid and liquid matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The following compound can be determined by this method:

ANALYTE	ABBREVIATION	CAS#
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
1,3-Dinitrobenzene	1,3DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-75-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
2-Amino-4,6-dinitrotoluene	2-Am-DNT	35572-78-2
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0

1.3 This is a high performance liquid chromatography (HPLC) method applicable to the determination of the compounds listed above.

1.4 This method is restricted for use to use by or under the supervision of analysts experienced in the use of HPLC systems and has demonstrated the ability to generate acceptable results using the procedure described in this document.

1.5 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory including the most recent NELAC standards.

2 Summary of Method

2.1 A measured volume/weight of sample, is processed using one of the following procedures a salting-out extraction, Solid-phase extraction, ultrasonic bath extraction, or a high level direct injection. An aliquot of the extract/sample is injected into a HPLC and the compounds are detected by the ultraviolet (UV) detector.

3 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis by running method blanks.

3.2 2,4-DNT and 2,6-DNT elute at similar retention times (retention time difference of 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture should be reported.

3.3 Tertyl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tertyl should be diluted with acetonitrile prior to filtration and acidified to pH <3. All samples expected to contain tertyl should not be exposed to temperatures above room temperature.

3.4 Degradation products of tertyl appear as a shoulder on the 2,4,6-TNT peak. Peak heights rather than peak areas should be used when tertyl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a current awareness file of the OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Data Safety Sheets (MSDS) has been made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the information of the analyst.

5 Apparatus and Materials

5.1 High Performance Liquid Chromatography (HPLC) apparatus

5.1.1 HPLC1 – Waters 600E gradient pumping system; consistent flow or equivalent.

5.2 HPLC Columns

PRIMARY

5.2.1 Reversed phase column: Merck Purospher RP-18e column; 5 um particle size; length 25 cm; i.d. 4.6 mm; Merck P/N 1.50169.0001 or Agilent P/N 79925PE-584 or equivalent

CONFIRMATION

5.2.2 Reversed phase column: Column Engineering Reliasil C8 column; 5 um particle size; 100Å pore size, length 15 cm; i.d. 4.6 mm; P/N R5FI-122 or equivalent.

5.3 Data system: Data is acquired using PE Nelson Turbochrom. All data is imported to the Hewlett Packard Chemserver and processed using Target software

5.4 Autosampler: Water 717 Autosampler or equivalent.

5.5 Detectors

Ultravoilet detection: Waters 486 Tunable Absorbance detector or similar.

5.6 Microsyringes: Hamilton gas tight, various sizes purchased from Supelco.

5.7 Analytical balance: capable of reading to 0.0001 g.

5.8 Mobile Phase Filter/Degassing Apparatus

5.8.1 Filtration Apparatus using 0.45 um filter; Supelco Cat. #Z29040-8 or equivalent.

5.8.2 Degassing Apparatus: Ultrasonic cleaner; VWP Catalog No 33995-536.

6 Reagents

6.1 HPLC grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without decreasing the accuracy of the determination.

6.2 Acetonitrile: HPLC grade purchased from VWR Catalog No. BJ015-4 or equivalent

6.3 Reagent Water: HPLC grade purchased from VWR Catalog No. EM-WX0004, or equivalent.

6.4 Methanol (MeOH): HPLC grade purchased from VWR Catalog No. EM-MX0475-1, or equivalent.

6.5 Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol): HPLC grade purchased from VWR Catalog No.323-4, or equivalent.

6.6 Calcium chloride, $CaCl_2$ - Reagent grade. Prepare an aqueous solution containing 5 g/L of calcium chloride per litre of water

6.7 Primary Stock Solutions

6.7.1 Ultra Scientific Intermediate Stock Solution 1 Cat No. NAIM-833A or equivalent containing 1,3-DNB, 2,4-DNT, HMX, NB, RDX, 1,3,5-TNB, and 2,4,6-TNT at 1000 ug/ml in acetonitrile. Store at 1-4°C away from light. Discard on or before the manufacturer's expiration date. Opened vials have a one-year expiration date.

10.3.5 Ultra Scientific Intermediate Stock Solution 2 Cat No. NAIM-833B or equivalent containing 2-Am-DNT, 4-Am-DNT, 2,6-DNT, 2-NT, 3-NT, 4-NT and Tertyl at 1000 ug/ml in acetonitrile. Store at 1-4°C away from light. Discard on or before the manufacturer's expiration date. Opened vials have a one-year expiration date.

10.3.6 8330 Intermediate Stock Standard (20 ug/ml): Dilute 40 ul of NAIM-833A (6.6.1) + 40 ul NAIM-833B (6.6.2) + 200 ul IST-630 (6.8) to 2.0mL mlacetonitrile. This standard should be stored in an amber vial at 4°C and is good for six months.

6.8 Second Source Stock Solutions

6.8.1 Restek 8330 Calibration Mix #1 Cat No. 31450 or equivalent containing 1,3-

DNB, 2,4-DNT, HMX, NB, RDX, 1,3,5-TNB, and 2,4,6-TNT at 1000 ug/ml in acetonitrile. Store at 1-4°C away from light. Discard on or before the manufacturer's expiration date. Opened vials have a one-year expiration date.

10.3.5 Restek 8330 Calibration Mix #2 Cat No. 31451 or equivalent containing 2-Am-DNT 4-Am-DNT, 2,6-DNT, 2-NT, 3-NT, 4-NT and Tertyl at 1000ug/mL in acetonitrile. Store at 1-4°C away from light. Discard on or before the manufacturer's expiration date. Opened vials have a one-year expiration date.

6.8.3 Second Source Intermediate Stock Standard

6.8.3.1 8330 Intermediate Second Source Stock Standard (20 ug/ml): Dilute 40.0 ul of 31450 (6.8.1) + 40.0 ul 31451 (6.8.2) + 40.0 ul IST-630 (6.9) to 2.0 ml acetonitrile. This standard should be stored in an amber vial at 4°C and is stable for up to six months.

10.3.5.1 Second Source Calibration Check Standard (100 ug/L): Fortify 2 mL of a 1:1 mixture of acetonitrile and $CaCl_2$ solution (6.6) with 10 uL of intermediate second source stock standard (6.8.3.1). Prepare fresh daily.

6.9 Surrogate Stock: 1-Chloro-3-nitrobenzene (1000 µg/mL). Purchased from Ultra Scientific, Cat. No. IST-630. Store at 1-4°C away from light. Discard on or before the manufacturer's expiration date. Opened vials have a one-year expiration date.

10.3.5 Surrogate Intermediate Stock Solution (20 µg/mL): Dilute 200 µL of IST-630 (6.9) to 10 mL in acetonitrile. Store at 1 - 4°C. Discard within six months of preparation.

10.3.6 Surrogate spike solution (5.0 µg/mL): Dilute 250 µL of IST-630 (6.9) to 50 mL in acetonitrile. Each sample (approximately 1000 mL) will be fortified with 1.0 mL of this solution resulting in a sample concentration of 5.0 µg/L.

6.10 Intermediate Calibration Standards. These Standards are prepared as illustrated by the table. They are to be stored in amber vials at 4°C and are stable for 1 month.

Table 1

	Amount of Intermediate stock 6.7.3	Final Volume in Acetonitrile
Level 1	1.0 µL	1.0 ml
Level 2	2.5 µL	1.0 ml
Level 3	5.0 µL	1.0 ml
Level 4	12.5 µL	1.0 ml
Level 5	25 µL	1.0 ml
Level 6	50 µL	1.0 ml
Level 7	100 µL	1.0 ml

These standards are to be prepared fresh on the day of initial calibration and are good for one day.

Table 2: Calibration Levels and respective concentrations in ug/L

Compound	Stock (6.7.3) Concentration ug/mL	Level 1 ug/L	Level 2 ug/L	Level 3 ug/L	Level 4 ug/L	Level 5 ug/L	Level 6 ug/L	Level 7 ug/L
1,3-DNB	20	20	50	100	250	500	1000	2000
2,4-DNT	20	20	50	100	250	500	1000	2000
HMX	20	20	50	100	250	500	1000	2000
NB	20	20	50	100	250	500	1000	2000
RDX	20	20	50	100	250	500	1000	2000
1,3,5-TNB	20	20	50	100	250	500	1000	2000
2,4,6-TNT	20	20	50	100	250	500	1000	2000
2-Am-DNT	20	20	50	100	250	500	1000	2000
4-Am-DNT	20	20	50	100	250	500	1000	2000
2,6-DNT	20	20	50	100	250	500	1000	2000
2-NT	20	20	50	100	250	500	1000	2000
3-NT	20	20	50	100	250	500	1000	2000
4-NT	20	20	50	100	250	500	1000	2000
Tertyl	20	20	50	100	250	500	1000	2000
1-Chloro-3-nitrobenzene	20	20	50	100	250	500	1000	2000

6.11 Spike Solution: This solution will be used for fortifying reagent water for the laboratory control samples, matrix spikes, and for the quality control check samples referenced in the initial demonstration Section in Section 8.0. Prepare by diluting 25.0 uL of Restek

8330 Cal Mix #1 Cat No. 31450 and 25 uL of 8330 Cal Mix #2 Cat No. 31451 to 25.0 mL in acetonitrile. Laboratory control samples or matrix spikes will be fortified with 1.0 mL of this solution. This will result in 1.0 ug/L concentration of all analytes based on an initial sample volume of 1000 mL:

7 Instrument Calibration

7.1 An external standard procedure is used for the 8330 analysis. A valid calibration of all target compounds and the surrogate must be in place prior to sample analysis. The calibration must consist of at least five points for linear calibration models, and of at least six points for non-linear calibration models.

7.2 With the Target Software, use the data to prepare a calibration using the externalstandard calibration technique. Two general calibration types are available on the target software.

7.2.1 Averaged Response Factor: This calibration model is acceptable if the averaged response factor over the calibration range is constant (20% RSD or less). If the RSD is greater than 20%, or if it does not represent the calibration data well, proceed to 7.2.2.

7.2.2 Prepare a calibration curve using one of three regression models. When using a calibration curve, do not include the origin or force the calibration through the origin. The calibration curve will be valid if the coefficient of determination (r^2) is 0.99 or greater.

7.2.2.1 Linear regression

7.2.2.2 Quadratic regression

7.2.3 If the criteria specified in 7.3.1 or 7.3.2 is not met, sample analysis will not begin until corrective action is taken resulting in acceptable %RSD or coefficient of determination. The following are suggestions on types of corrective action that may be pursued:

7.2.3.1 If a specific calibration level is the cause of the unacceptable calibration, re-inject the standard.

7.2.3.2 A calibration level for an analyte may be removed if it does not represent the practical quantitation limit for the analyte.

7.2.3.3 If the above steps do not correct the problem, re-prepare initial calibration standards and re-inject. If the calibration is still unacceptable, instrument maintenance may be necessary.

7.3 To evaluate the initial calibration analyze the appropriate second source check standard (6.7.3.3). Acceptance criteria is ±15% of the nominal value.

7.3.1 Prepare second source and reinject.

7.3.2 If it still does not meet +/-15% criteria, identify source of problem and correct before continuing with sample analysis. This may involve recalibration.

7.4 The initial calibration must be verified by the analysis of a calibration verification check standard at the beginning of each 12-hour shift or every ten field samples, which ever is more frequent. If the response for each analyte is ±15% of the response obtained during the initial calibration then the initial calibration is considered still valid and the analyst may proceed with sample analysis. If the standard does not meet the ±15% criteria, re-inject the standard. If the second injection of the standard still does not meet calibration verification criteria, the following actions may be taken:

7.4.1 Identify and correct the source of the problem. Inject a calibration verification standard. If results meet calibration verification criteria, sample analysis may proceed.

7.4.2 Recalibrate the instrument.

7.5 Verify the initial calibration following the analysis of a group of samples to ensure the instrument is still in control. The result should be ±15% of the expected result. If this criteria is not met, re-inject a calibration verification standard. If the criteria is still not met, the following actions will be taken with the samples analyzed prior to the failing calibration verification standard.

7.5.1 If the bias is low, re-analyze the samples under a valid calibration.

7.5.2 If the bias is high, and there were detections in a field sample, re-analyze that field sample.

7.5.3 If the bias was high and analytes were not detected in the field sample, data quality is not impacted. Do not re-analyze the samples. Report to the client without qualification.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Initial Demonstration of Capability. This demonstration must be successfully performed by each analyst before being considered proficient to analyze samples by this method.

8.2.1 As described in Section 6.10, the quality control check sample concentrate will contain each analyte at 1.0 ug/ml concentration in acetonitrile.

8.2.2 Four QC check samples will be prepared by spiking four 1000 mL aliquots of reagent water or four 30g aliquots of organic free soil with 1.0 mL spike solution (6.8). Take the samples through all steps of the extraction as described in the Prep SOP 09-8330W.

8.2.3 Analyze samples by HPLC as described in Section 10.0

8.2.4 An average recovery range of 70-130% recovery will be used as guidance to assess laboratory performance. Calculate average recovery (x) in µg/L, standard deviation of the recovery(s) in µg/L. Table 1 in Appendix A gives method accuracy and precision as a function of concentration for the analytes of interest. The contents of this table will be used to evaluate the laboratory's ability to perform and generate acceptable data by this method. When sufficient data is gathered, in house control limits will be used to assess laboratory performance.

8.2.5 If one or more analytes do not meet this criteria, identify and correct source of problem and report test for those analytes that initially failed.

8.3 Laboratory Method Blank. A method blank is prepared and analyzed with each extraction batch of 20 samples or less. The method blank should be less than the method detection limit, less than 5% of the regulatory limit associated with the analyte, or less than 5% of the result for the sample analyte, whichever is greater. If the method blank does not meet this criteria the following corrective actions shall be implemented:

8.3.1 Evaluate an instrument blank (acetonitrile) to determine if the contamination is post extraction related.

8.3.2 If 8.3.1 does not reveal a problem, evaluate all the samples in the extraction batch. If there are samples without detections of the analyte in question, data was not

impacted and may be reported without qualification.

8.3.3 If samples do have detections of the analyte, and the detection of the analyte in the method blank is less than 5% of the detection in the field sample, data may be reported as acceptable.

8.3.4 If the samples have detections, and the detection in the method blank is greater than 5% of the detection in the sample, the following steps will be taken:

8.3.4.1 If additional sample is available and the sample is still within holding time, the sample will be re-extracted.

8.3.4.2 If additional sample is not available, or the sample is past it's holding time, the appropriate customer service representative will be notified, and the data will be qualified to the end user.

8.4 Assessing Surrogate Recovery: The surrogate concentration in samples will be 50 ug/L based on a 1 liter initial volume. The concentration will vary based on the initial volume of the sample. Until sufficient data has been acquired to calculate matrix and extraction specific control charts, surrogate recovery limits will be 65% to 135%. When sufficient data has been acquired (>20 data points), determine the average and standard deviation of the data points. Control limits will be the average of ± 3 standard deviations.

8.4.1 When surrogate recovery from a sample is outside the established control limits, and the method blank and laboratory control sample is in control, the following steps shall be taken:

8.4.1.1 Check calculations and volumes used for spiking.

8.4.1.2 In this situation, sample matrix may be the cause of the out of control recovery in the field sample. Examine the sample chromatogram for other indications of matrix affect. Re-extract the sample or qualify as suspect due to sample matrix.

8.4.2 If surrogate recovery is out of control in the method blank and laboratory control sample, the following actions shall be taken:

8.4.2.1 Check calculations and volumes used for spiking.

8.4.2.2 Re-validate the surrogate spike solution used to spike the samples. If the

integrity of the spike solution has degraded (ie. solution concentrated), re-evaluate recoveries based on the actual concentration of the spike solution. Additionally, dispose of the spike solution.

8.4.2.3 Check instrument performance. If the instrument is not running correctly, make the appropriate adjustments, or perform the appropriate instrument maintenance. Re-calibration may be necessary before sample analysis can resume.

8.4.2.4 If the above do not indicate a problem, and re-analysis of the blank and / or laboratory control sample do not result in acceptable surrogate recovery the following actions will be taken:

8.4.2.4.1 If surrogate recoveries for the samples are in control, no further actions will be necessary.

8.4.2.4.2 If possible, re-extract all samples with out of control surrogate recoveries.

8.4.2.4.3 If re-extraction is not possible, contact the appropriate customer service person to notify client. Qualify the samples.

8.5 Assessing Laboratory Performance - One laboratory control sample will be extracted and analyzed with each group of 20 field samples or one per extraction batch. The laboratory control sample is prepared and analyzed in the same manner as described in Section 8.2. Until sufficient data is acquired to establish in house control limits, the recoveries must be within the ranges 70 to 130 %. If a recovery for an analyte is out of control, the following actions shall be taken:

8.5.1 Check calculations and volumes used for spiking.

8.5.2 Re-validate the surrogate spike solution used to spike the samples. If the integrity of the spike solution has degraded (i.e., Solution concentrated), re-evaluate recoveries based on the actual concentration of the spike solution. Additionally, dispose of the spike solution.

8.5.3 If the above steps do not indicate a problem, and the batch matrix spike also has unacceptable spike recoveries, the following actions shall be taken:

8.5.3.1 If spike recovery for the matrix spike is acceptable, sample

data is acceptable and reported to the end user without qualification.

8.5.3.2 If spike recovery for the matrix spike is also unacceptable, samples will be re-extracted if additional sample is available and if the sample is still within holding time.

8.5.3.3 If additional sample is not available, contact the appropriate customer service representative. Data will be reported to the end user as suspect.

8.6 Assessing Analyte Recovery (Matrix Spike & Matrix Spike Duplicate): The laboratory must add a known concentration to a minimum of 5% of the routine samples or one sample concentration per set, whichever is greater. The sample selected for spiking will be done randomly. The same sample will be spiked a second time for precision data. Alternatively, when a field sample is known to have significant levels of target analyte, the field sample will be extracted in duplicate rather than a matrix spike duplicate. Spiked samples will be fortified at the same level as the laboratory control sample.

8.6.1 Calculate the percent recovery, R of the concentration for each analyte, after correcting the analytical result, X, from the fortified sample for the background concentration, b, measured in the unfortified sample, i.e.:

$$R = 100\ (X - b)\ /\ \text{fortifying concentration}.$$

Until sufficient data is acquired to establish in house control limits, the recoveries must be within the ranges 70 - 130%. If a recovery for an analyte is out of control, the following actions should be taken:

8.6.1.1 If analyte recovery in a field sample is outside laboratory control limits, and the same analyte is in control in the laboratory control sample, sample results for that analyte in the unfortified matrix must be listed as suspect due to matrix with a qualifying comment on the lab report.

8.6.2 Precision: Until sufficient data is acquired to generate precision control limits, 50% RPD will be the upper precision control limit when comparing results between the matrix spike and the matrix spike duplicate. The same precision criteria applies to results in a duplicate pair when a sample duplicate is extracted in place of a matrix spike duplicate. Relative percent difference (RPD) is calculated using the following equation:

$$\%RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

where: C_1 is the result from the sample
C_2 is the result from the sample duplicate

8.7 Accessing the internal standard (if used): The analyst will monitor the Internal Standard (IS) response (peak area) of all samples during each analysis day. The IS response for any sample chromatogram shall not deviate from the most recent calibration check standard's IS response by more than 50%.

9 Sample Collection, Preservation and Handling

9.1 Sample Collection

9.1.1 Containers used to collect samples must be specially cleaned 1-liter glass bottles. The sample containers should have screw caps with Teflon lined septa. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. A minimum of one liter is necessary for this analysis.

9.1.2 Conventional sampling practices should be followed

9.2 Sample Preservation

9.2.1 The samples must be iced or refrigerated at 1-4^{o}C ± 1^{o}C from the time of collection until extraction.

9.3 Sample Handling

9.3.1 All aqueous samples must be extracted within 7 days.

9.3.2 Solid and concentrated waste samples must be extracted within 14 days.

9.3.3 Sample extracts must be analyzed within 40 days of extraction. Store extracts away from light at $\leq$ 4^{o}C.

9.3.4 All samples not analyzed within this time frame must be discarded and re-sampled for analysis unless permission is give by the client to analyze the sample past recommended hold time.

10 Procedure

10.1 Sample Preparation: Samples must be prepared by one of the following methods prior to HPLC analysis.

Matrix	Methods
Water	8330A Salting out, 3535A SPE
Solid	8330A Soil sonication

10.2 To achieve maximum sensitivity with this method, the extract must 5.0 ml.

10.3 HPLC Analysis

10.3.1 The sample HPLC operating conditions used for the initial calibration will be used for sample analysis.

10.3.2 Verify the calibration each 12-hour shift by injecting a level 3 or level 4 standard. A calibration standard will also be injected at intervals no less than once every 20 samples. Sample analysis may continue as long as calibration verification samples continue to meet quality control criteria.

10.3.3 HPLC conditions. Use column described in Section 5.2.1. The following gradient parameters are used with a 100 ul sample injection.

Time (min)	**Flow (mL/min)**	**%H_2O**	**%MeOH**	**Curve**
Initial	1.00	90	10	*
25.0	1.00	20	80	6 (linear)
35.0	1.00	20	80	11 (hold)
40.0	1.00	90	10	9 (slow)

Ultraviolet detector is set 254nm wavelength

10.3.4 Qualitative Analysis: As previously discussed, the UV detector (5.5) will be the primary detector used for identification and quantitation of target compounds. Also, the LC-18 column (5.2.1) will be used for tentative identification and all quantitation of target compounds. Tentative identification of an analyte occurs when a peak from a sample extract fall within the relative retention time window of a compound of interest.

Confirmation Techniques:

10.3.5 Prior knowledge of sample site: Section 11.9 paragraph three of method 8000C states "Confirmation may not be necessary if the composition of the sample matrix is well established by prior analyses…" Consequently, if it has been established in previous sampling events from a site that target nitroaromatics and nitroamines are present, confirmation is not necessary.

10.3.6 Confirmation using the specified confirmation column (C8, Section 5.2.2): The confirmation column will provide good qualitative confirmation, but quantitation may be impacted due to partially co-eluting peaks on the confirmation column.

10.3.6.1 HPLC Conditions: Use column described in Section 5.2.2. The following gradient parameters are used with a 100 ul sample injection.

Time (min)	**Flow (mL/min)**	**%H_2O**	**%IPA**	**Curve**
Initial	1.70	88	12	*
10.0	1.70	88	12	11 (hold)
20.0	1.70	78	22	6 (linear)
35.0	1.70	78	22	11 (hold)
40.0	1.70	88	12	3 (fast)

Ultraviolet detector is set 254nm wavelength

10.3.5 Quantitative Analysis: As described in Section 7, all compounds will have a valid calibration curve.

10.3.8 All calculations will be performed with the Target Software. The following are equations as they would be performed manually.

Aqueous samples

$$\text{Concentration (ug/l)} = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(V_s)(1000)}$$

Non-aqueous samples

$$\text{Concentration (ug/kg)} = \frac{(A_s)(C_{is})(D)(V_i)}$$

$$(A_{is})(RF)(W_s)(1000)$$

Where: A_s = Area of the peak for the analyte in the sample
A_{is} = Area of the peak for internal standard
C_{is}= Concentration of the internal standard in the sample extract in ug/l
D = dilution factor. If no dilution D = 1
V_i = Volume of the extract injected (ul)
RF= Mean response factor from the initial calibration
W_s = Weight of sample extract (grams)

10.3.9 If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.

10.3.10 All calculations are performed by target software.

11 Reporting Results

11.1 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

11.2 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.

11.3 Report the actual result in the Horizon LIMS. The reporting limit is at or above the lowest calibration standard.

12 Waste Disposal

12.1 Refer to ALSI SOP 19-Waste Disposal.

13 Pollution Prevention

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees.

ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

14 Definitions

14.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

TABLE 1

QC Acceptance Criteria[a]

Compound	Test Conc. (ug/l)	Limit for s (ug/l)	Range for x (ug/l)	Range p, p_s (%)
HMX	1.0	20.2	D-52.9	D-124
RDX	1.0	45.1	22.1-112.1	D-139
1,3,5-TNB	1.0	1.4	0.56-5.6	D-126
1,3-DNB	1.0	2.0	1.6-5.8	12-135
Tetryl	1.0	2.0	0.1-5.5	D-128
NB	1.0	3.1	1.8-13.8	6-150
2,4,6-TNT	1.0	2.3	D-10.7	D-116
4-Am-DNT	1.0	2.5	D-7.0	D-159
2-Am-DNT	1.0	2.1	D-8.8	D-199
2,4-DNT	1.0	2.0	0.3-10.0	D-110
2,6-DNT	1.0	3.0	2.7-11.1	14-123
2-NT	1.0	4.3	D-11.9	D-142
3-NT	1.0	1.5	0.6-5.1	D-116
4-NT	1.0	20.4	10.8-50	D-122

s = Standard deviation of four recovery measurements in ug/l

x = Average recovery for four recovery measurement in ug.l

p, p_s = Percent recovery measured

D = Detected; result must be greater than zero

[a]Criteria adapted from 40 CFR part 136 for Method 610. These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limit to concentrations below those used to develop Table 1.

Appendix A Sample Logbook Page

Sequence: C:\HPLC1\ Batch: Method: Date:

Sample Number	Sample ID	Dilution	Tech	QC	Standard	Comments

Buffer Batch Number:	Water Lot #	Injection Vol
	MeOH Lot #	
PCR Reagent Batch:	CH3CN Lot #	Internal Stds
	Other	

SOP Change History Sheet

Section No.	**Section**	**Reason for Change**
Revision 3: 10/31/05		
10.3.4 – 10.3.5	Procedure	USACE audit response
Appendix A	Logbook Sample Page	USACE audit response
5.2	Apparatus and Materials	Removed reference to column guard; updated manufacturer information
6.5	Reagents	Added Isopropyl Alcohol to list
10.3.3	Procedure	Removed reference to guard column and expanded HPLC table
10.3.6	Procedure	Added gradient parameters table
Figure 1		Removed

SOP Concurrence Form

for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Document Title: Ultrasonication of Solids for the Analysis of Explosives by EPA Method 8330A (HPLC)

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

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TABLE OF CONTENTS

1 Scope and Application....3
2 Summary of Method....3
3 Interferences....3
4 Safety....3
5 Apparatus and Materials....4
6 Reagents....5
7 Glassware Cleaning....6
8 Quality Control....6
9 Sample Collection, Preservation and Handling....8
10 Procedure....8
11 Calculations....10
12 Reporting Results....10
13 Waste Disposal....11
14 Pollution Prevention....11
15 Definitions....11
16 Troubleshooting....11
APPENDIX A....12
SOP Change History Summary....13
SOP Concurrence Form....14

1 Scope and Application

1.1 This Standard Operating Procedure is adapted from U. S. EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Method 8330A, Revision 1, January 1998.

1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.3 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 Solid Samples are extracted, using acetonitrile in an ultrasonic bath, and filtered, in preparation for HPLC analysis.

3 Interferences

3.1 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics in particular must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

3.2 Soap residue on glassware may cause degradation of certain compounds. All glassware shall be rinsed carefully with deionized water to avoid the problem.

3.3 Interferences co-extracted from the samples will vary considerably from source to source.

3.4 Light decomposes various target analytes, in particular, tertyl. Precautions shall be taken as to not allow excessive amounts of light to reach sample.

3.5 Heat causes some analytes, in particular, tetryl, to decompose rapidly. Samples shall not be exposed to temperatures above room temperature.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

4.2 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.

4.3 All solvents and reagents used in this procedure shall be handled in a fume hood using

Viton gloves.

4.2 Solids containing a high amount of explosive analytes may be present as finely ground grayish-white material. Extreme caution shall be used with these samples. Samples containing large amounts of grayish-white material, or lumps of material that have a chemical appearance or are known to have come from a site containing high amounts of explosives shall be suspect and not ground as described in Section 10.1.3.

5 Apparatus and Materials

5.1 Ultrasonic Disrupter – Bronson 2510, 80 watt, or equivalent, equipped with floating tray, and a temperature-controlled bath.

5.2 Vortex Mixer.

5.3 Balance – capable of weighing ± 0.01 g.

5.4 Syringes – gastight, appropriate sizes.

5.5 Syringes – disposable 10 mL (0.90mm x 25mm).

5.6 Syringe Filters – Spartan #00760, 0.45 µm Teflon (PTFE) membrane, 25mm diameter, or equivalent.

5.7 Pasteur Pipets – glass disposable.

5.8 Mortar/Pestle – ceramic.

5.9 Culture Tubes – glass, disposable with Teflon-lined (PTFE) screw caps, 16 x 125mm.

5.10 VOA Vials – 20 mL, pre-cleaned glass.

5.11 Vials – amber, glass 2 dram.

5.12 Weigh Boats – aluminum.

5.13 Volumetric Pipets – 5 mL, 10 mL, Class A.

5.14 600 micron sieve – stainless steel.

6 Reagents

NOTE: Unless indicated otherwise, reagents listed in this section are stored at room temperature and carry a 6 month expiration date.

6.1 Acetonitrile: HPLC grade purchased from VWR Catalog #BJ015-4 or equivalent.

6.2 Methanol (MeOH): HPLC grade purchased from VWR Catalog #EM-MX0475-1 or equivalent.

6.3 Calcium chloride, dehydrate: ACS grade, purchased from VWR Catalog #EM-CX0130-1 or equivalent.

6.3.1 Add 5.0 g calcium chloride to 500 mL reagent water in a 1-L volumetric flask. Stir until dissolved and then dilute to the mark with reagent water.

6.4 Surrogate Stock Solution.

6.4.1 1-mL ampules of 1–chloro-3-nitrobenzene (1000 µg/mL) in acetonitrile are purchased, Ultra Scientific part #IST-630 or equivalent. Expiration dates are provided by the manufacturer and the ampules shall be stored at -10º to -20ºC in the dark.

6.4.2 Working Surrogate Solution (1000 µg/µL) – Ultra Scientific stock solution #IST-630 or equivalent is transferred into a crimp top vial; label with manufacturer provided expiration dates. Add 20 µL to all samples and quality controls.

6.5 LCS/matrix spike stock solution – 2 mixes are purchased commercially in 1 mL ampules provided with expiration dates. Both mixes are at 1000 µg/mL in acetonitrile.

6.5.1 Restek 8330 mix #1, part #31450 or equivalent contains the following: 1,3 – dinitrobenzene, 2,4 – dinitrotoluene, HMX, Nitrobenzene, RDX, 1,3,5 – trinitrobenzene, 2,4,6 – trinitrotoluene

6.5.2 Restek 8330 mix #2, part #31451 or equivalent contains the following: 2 – amino – 4,6 – dinitrotoluene, 4 – amino – 2,6 – dinitrotoluene, 2,6 dinitrotoluene, 2 – nitrotoluene, 3 – nitrotoluene, 4 – nitrotoluene, tetryl

6.6 Spike Solution (100 µg/mL) – Place 800 µL of acetonitrile (Section 6.1) in a 12 x 32mm amber glass vial equipped with a PTFE lined cap. Add 100 µL of Stock Solution 1 (Section 6.5.1) and 100 µL of Stock Solution 2 (Section 6.5.2). Cap and mix well. This standard shall be stored above the freezing point of water up to 6º C and expires six months after preparation.

6.7 Reagent Sand – muffle furnaced at 400º for 4 hours.

7 Glassware Cleaning

7.1 Remove surface residuals immediately after use by rinsing with tap water or the last solvent used.

7.2 Soak the glassware in hot water and Liquinox detergent to float most particulate material from glassware. Use a brush to scrub the glassware to aid in the removal of residual materials.

7.3 Hot water rinse the glassware.

7.4 If the glassware used was in contact with high level samples (greater than 100 mg/kg) and/or there is an apparent residue remaining on the glassware after the first three steps, it will be necessary to soak the glassware in a strong oxidizing agent to destroy traces of residual compounds. The oxidizing agent currently used is Chem Solv 2157, manufactured by Mallinchrodt and distributed by Baxter Scientific (part #2157-INY, 1995). Chem Solv 2157 is a safer alternative to chromic acid solutions typically suggested for cleaning glassware. The manufacturer's instructions for the safe handling of Chem Solv 2157 shall be stored with each container and reviewed before its use. Glassware suspected or known to have been in contact with high-level samples shall soak for 1 hour in the Chem Solv solution. Rinse with copious amounts of tap water and resubmit for normal cleaning procedure (Section 7.2).

7.5 Rinse the glassware thoroughly with deionized water to remove remaining materials and any metallic deposits.

7.6 Rinse the glassware with isopropyl alcohol. Drain any pooled alcohol into a solvent waste container.

7.7 Dry the glassware in an oven at 130ºC $\pm$ 30ºC for a minimum of 20 minutes. Allow the glassware to cool and cover with aluminum foil.

7.8 Flush all glassware immediately before use with the extraction solvent being used for the application.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Initial Demonstration of Capability: This demonstration must be successfully performed by each analyst prior to being considered proficient to analyze samples by this method. Ongoing proficiency must be established annually as specified in the QA Plan, under Technical Training,

8.2.1 Four QC check samples will be prepared: Spike four 2.0 g samples of reagent sand

(Section 6.7) with 20.0 µL of the Spike Solution (Section 6.6). Take the samples through all steps of the extraction as described in this SOP.

8.2.2 Analyze samples by HPLC as described in ALSI SOP 1B-8330.

8.2.3 An average recovery range of 70-130% recovery will be used as guidance to assess laboratory performance. See ALSI SOP 1B-8330 for calculation of percent recovery. A precision control limit of 30% RSD for all four check samples will be used as guidance to assess laboratory performance. When sufficient data is gathered, in house control limits will be used to assess laboratory performance.

8.2.4 If one or more analytes do not meet this criterion, identify and correct source of problem and report test for those analytes that initially failed.

8.3 A method blank must be run with each batch of samples prepared. It is imperative that the blanks be subjected to exactly the same analytical procedures as those used in the actual samples. This includes the addition of the surrogate standards and use of sodium sulfate and other chemicals used in the extraction procedures.

8.4 A matrix spike and matrix spike duplicate or a matrix spike and duplicate must be extracted with each batch. If insufficient sample is available to perform a matrix spike or duplicate, a comment must be placed in the extraction log.

8.4.1 Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.5 A laboratory control sample (LCS) must be extracted with every batch. The LCS is prepared similarly to a method blank but is spiked with the LCS spiking solution.

8.6 The size of any extraction batch cannot exceed 20 samples.

8.7 MDL Study

8.7.1 MDL studies must be performed according to ALSI SOP 99-MDL or the reference method, whichever is more frequent.

9 Sample Collection, Preservation and Handling

9.1 Soil samples shall be collected in 4 oz (or larger) soil jars with Teflon-lined lids. All samples shall be stored in the dark above the freezing point of water up to 6ºC until

extraction. No preservation is recommended.

9.2 Samples must be extracted within 14 days of collection.

9.3 Sample extracts must be analyzed within 40 days of extraction. Store extracts away from light above the freezing point of water up to 6ºC.

10 Procedure

10.1 Sample Homogenation

10.1.1 Weigh 10.0 – 12.0 g of sample into an aluminum weigh boat. 10.0 – 12.0 g of muffle furnaced reagent sand shall be used for blanks and LCS's.

10.1.2 Samples are placed in a closed desiccating cabinet, without light or heat, at room temperature or less for a minimum of 18 hours in order to dry. Samples are then weighed, their weight recorded and returned to the desiccating cabinet. A second weighing will occur no less than four hours after initial weighing. Samples must have a RPD of less than 1% in order to be considered dry. If the RPD is >1% additional drying needs to occur until RPD% $\leq$ 1.

$$RPD = \frac{\text{Difference between sample results (mg)}}{\text{Average of sample results}} \times 100\%$$

10.1.3 Once samples are dried and at a constant weight, it is desirable to homogenize the sample; however, extreme care must be exercised due to the explosive nature of the analytes. Solids containing a high amount of explosive analytes, may exhibit the inclusion of a finely ground grayish-white material. Extreme caution shall be used with these samples. Samples containing large amounts of grayish-white material, or lumps of material that have a chemical appearance or are known to have come from a site containing high amounts of explosives shall be suspect and not subjected to any homogenization. If a sample is believe to be clean, and it requires homogenization because of a non-uniform appearance, then the sample may be gently ground and homogenized in a clean, dry, acetonitrile rinsed, mortar, by pestle, followed by passing the ground sample through a 600 micron sieve. Such homogenization is performed at the discretion of the analyst involved, and it is not a requirement of this procedure.

10.2 Sample Extraction

10.2.1 Remove 2.0 – 2.2 g of subsample of each sample into separate, clean, dry, disposable culture tubes. Using a black Sharpie marker, label all tubes with the

proper sample prep ID and COC# information. Record the actual weight in extraction logbook.

10.2.2 Add 20 µL of working surrogate solution (Section 6.4.2) to the surface of all samples, blanks, and LCS's.

10.2.3 Add 20 µL of the working spiking solution (Section 6.6) to all matrix spikes and LCS.

10.2.4 Using an acetonitrile rinsed, Class A, 10 mL volumetric pipet, add 10.0 mL of acetonitrile to all samples, blanks, and LCS's. Cap all tubes with clean PTFE-lined caps.

10.2.5 Vortex swirl each tube for one minute. (In larger batches, this step may take considerable time. Precautions shall be taken as to not subject tubes to excessive light or heat.)

10.2.6 Place all tubes into the floating tray of the ultrasonic bath. (Samples shall be upright during sonication; a rubber band may be needed to hold all the tubes together and upright over the entire sonication process.) Cover the entire top of sonicator with aluminum foil so as not to allow any light to reach the samples.

10.2.7 Set the ultrasonic bath to the "run continuously" position, recording the time in the logbook. Samples are sonicated for 18 hours. (During the sonication, bath temperature must stay in a 15ºC- 25ºC temperature range. To ensure this, a chilling apparatus needs to be in place. Occasional monitoring of the bath temperature needs to be done in order to ensure the water stays within this range.)

10.2.8 After 18 hours of sonication, turn the bath off and record the time. Remove the samples from the bath and place in a tube rack and allow to settle for 30 minutes.

10.2.9 Using a separate, acetonitrile rinsed, Class A 5 mL volumetric pipet for each sample, transfer 5.0 mL of supernatant to a 20 mL VOA vial. Be sure to transfer all COC # information to the vials.

10.2.10 Using a 5 mL, Class A volumetric pipet add 5.0 mL of calcium chloride (Section 6.3.1) to each vial. Shake and let stand for 15 minutes.

10.3 Filtering of Supernatant

10.3.1 Assemble a 10 mL disposable syringe without the PTFE filter. Draw entire supernatant into syringe. (Depending on sample matrix, if any residual substrate is left on the syringe tip, it will need to be rinsed with a small amount of

acetonitrile.) Invert syringe and attach a 0.45 um PTFE filter.

10.3.2 Discard first 3 mL and retain remainder in a PTFE-capped 2 dram amber vial labeled with all appropriate COC information and LIMS ID. For dirtier samples, the syringe filter may need to be replaced before the entire sample is pushed through.

11 Calculations

11.1 Not applicable.

12 Reporting Results

12.1 In the "Daily Function" menu of the LIMS, select #6, "Preps Performed Entry".

12.2 Enter Test 8330S.

12.3 Batch #: The batch # and the COC # assigned to the reagent blank are the same six characters. The reagent blanks are sequentially numbered in the extraction logbook with a 2-letter prefix followed by 4 digits (ex. ES0001).

12.4 To assign a reagent blank a COC # and subsequently the extraction batch # in the Prep Performed Entry menu of the LIMS, consult the extraction logbook and locate the last blank for the appropriate extraction. Name the batch # the following number proceeded by the appropriate 2-letter prefix (ex. ES0001).

12.5 Enter the date/time the extraction was started.

12.6 Enter the technicians' initials finishing the method.

12.7 Enter the digit "1" for the initial volume and the digit "1" for the final volume. Analysts in the GC department will calculate the extraction factors manually based on the entries in the extraction logbook. These values entered in the LIMS will not be used in calculating the final concentration but will indicate to the analyst that the extraction is complete.

12.8 Select "B" from the bottom screen menu to create a reagent blank in the LIMS.

12.8.1 Enter the digit "1" for the final volume and "1" for the initial volume of the reagent blank.

12.9 Review the labeling on vials, the entries in the logbook and the entries in the LIMS to verify that all entries match.

12.10 Deliver the vials to the GC Department for storage above the freezing point of water up to

6ºC until analysis.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

APPENDIX A

Work continued from page:____

HPLC EXTRACTION

Work continued to page:____

EPA Method: _____ Surrogate ID: _____

ALSI SOP: _____ Surrogate Amount: _____

Extraction:	Date	Time	Tech	Sonication:	Time On	Time Off	Total Hours Sonicated	Tech

Line #	Sample ID	Sample Amount	QC Type	Spike ID	Spike Amount	Final Volume	Elution Time	Sample Specific Comments (Include weigh data for drying, if applicable.)
1.)								
2.)								
3.)								
4.)								
5.)								
6.)								
7.)								
8.)								
9.)								
10.)								
11.)								
12.)								
13.)								
14.)								
15.)								

Reagent Identification(s): _____

_____

Approved By:_____
Date Approved:_____

Page #:____

Revised 2/1/05

SOP Change History Sheet

Section No.	Section	Reason for Change
Revision 1: 04/10/2006		
Spelling, grammar and formatting revisions made throughout SOP.		
1.3	Scope and Application	Added individual project verbiage
2.1	Summary of Method	HPLC reference included
4.2	Safety	Added MSDS availability
4.3	Safety	Deleted use of Silvershield gloves
5	Apparatus and Materials	Added "PTFE" to Teflon references for clarity
6	Reagents	Numerous additions and revisions made throughout this section
8.2	Quality Control	Added details for DOC and annual recertification
8.7	Quality Control	Added SOP 99-MDL reference
10.1	Procedure	Added details for clarity and precautionary directions for identifying high-explosive analytes
10.2.2, 10.2.3	Procedure	Deleted syringe reference and revised solution volumes
15	Definitions	Added section
16	Troubleshooting	Added section
A	Appendix	Added bench worksheet
SOP Change History Summary		Added section

SOP Concurrence Form

for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

Document Title: Analysis of Total Metals by Inductively Coupled Plasma Using the TJA Trace ICP

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
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Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Susan Magness, Date
Validator

TABLE OF CONTENTS

1 Scope and Application 3
2 Summary of Method 3
3 Interferences 4
4 Safety 5
5 Apparatus and Materials 5
6 Reagents 6
7 Instrument Calibration 14
8 Quality Control 15
9 Sample Collection, Preservation and Handling 23
10 Procedure 24
11 Calculations 27
12 Reporting Results 27
13 Waste Disposal 27
14 Pollution Prevention 27
15 Definitions 28
Appendix A 29
TABLE 1 30
TABLE 2 31
TABLE 3 32
TABLE 4 33
SOP Change Summary 34
SOP Concurrence Form 36

1 Scope and Application

1.1 This method is adapted from the U.S. EPA SW846 Method 6010B Revision 2, December 1996, "Inductively Coupled Plasma - Atomic Emission Spectroscopy." This method is applicable to a wide variety of matrices including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, industrial hygiene paints, wipes, airs, and other solid wastes.

1.2 The specific elements which ALSI analyzes by this method are listed in Table 1. This table lists the wavelengths used for the appropriate elements.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of an ICP. Each analyst must also be skilled in the interpretation of raw data, including quality control data.

1.4 All samples are digested using appropriate sample preparation techniques.

1.5 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.6 Method Detection Limits can be found in the current metals department method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

2 Summary of Method

2.1 This method measures element-emitted light by optical spectrometry. The ICP instrument contains a torch through which flows argon gas. A spark is used to initiate a plasma of ionized argon, which is then maintained, by a radio-frequency field. Samples are pulled into the system by a peristaltic pump and nebulized. The resulting aerosol is transported into the plasma torch. Element specific atomic- and ionic-line emission spectra are produced by the excited atoms or ions on their return to the ground state. The emission spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. The average of two intensity exposures, along with inter-element corrections yields the final result.
*(**Note:** For all sequence runs that involve samples from the U.S. Army Corp, an average of three separate exposures is used to calculate the data.)*

2.2 Background correction is used for all element determinations. Background intensity is measured adjacent to the analytical lines from the samples during analysis in an area free from spectral interferences. It's then subtracted from the intensity measured at these lines.

2.3 The possibility for additional interferences such as spectral, chemical, and physical interferences, does exist. Appropriate corrections for these interferences must be made.

3 Interferences

3.1 Spectral interferences are caused by (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background from continuum or recombination phenomena; and (4) stray light from the line emission of high concentration elements.

3.1.1 Background contribution and stray light are normally compensated for by the background correction.

3.1.2 Unresolved overlap requires the selection of an alternate wavelength or an alternate method of analysis such as graphite furnace or flame AA.

3.1.3 Spectral overlap is compensated for in the Trace instrument by automatic correction of the raw data after monitoring and measuring the interfering elements. A linear relationship between the interferant levels and the false interferences they cause can be assumed. Inter-element correction factors are checked daily by analysis of an interference check solution and updated annually, or whenever indicated by failure of the interference check solution.

3.2 Physical interferences are effects associated with the sample introduction and flow through the instrument. These interferences are brought about due to differences in viscosity and surface tension. Physical interferences are most commonly seen in samples containing high dissolved solids or high acid concentrations. To reduce physical interferences, ALSI uses a peristaltic pump for sample introduction, dilutions of problem samples, and matrix matching of standards to samples. Internal standard addition and the method of standard additions may also be used to compensate for physical interferences.

3.2.1 Another problem that can occur while analyzing samples with high dissolved solids is salt build-up at the tip of the nebulizer, which affects aerosol flow rate and causes instrument drift. This problem is controlled by regular maintenance and by wetting the argon prior to nebulization using a humidifier.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these are not significant with the ICP. If they are encountered, they can be minimized by carefully selecting the operating conditions (RF power, torch position, and nebulizer flow rate), buffering the sample, matrix matching, and performing standard addition procedures. Chemical interferences are highly dependant on matrix type and the specific analyte of interest.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully defined; however, each chemical compound should be treated as a potential health hazard.

4.2 Analysts shall consult the Material Safety Data Sheets (MSDS) for the chemicals used in the analysis process.

4.3 Precautions should be taken when handling samples and/or chemicals in the lab. The use of gloves, safety glasses, and lab coats is required when working with samples.

5 Apparatus and Materials

5.1 Thermo Jarrell Ash 61E Trace Simultaneous ICP Emission Spectrophotometer with the following options:

5.1.1 IBM compatible 386 computer
ThermoSPEC/AE v5.06 software
Axial Torch
Computer controlled emission with background correction.
Computer inter-element correction ability
Radio frequency generator coupled to a water cooled induction coil
Water cooler
Vacuum Pump
Polychromatic optic system under vacuum
Adjustable variable speed peristaltic pump
Mass flow controllers for argon flow rate
HP LaserJet Printer
TJA 300 automatic liquid sampler
Argon humidifier

5.1.2 Sample pump tubing - CPI, catalog #4062-535 or equivalent.

5.1.3 Internal Standard pump tubing - CPI, catalog #4062-5015 or equivalent.

5.1.4 Rinse pump tubing - CPI, catalog #4062-545 or equivalent.

5.1.5 13 x 100 polystyrene tubes - Perfector Scientific, catalog #2110 or equivalent.

5.1.6 Internal Standard Mixing Kit - CPI, catalog #4062-910 or equivalent.

5.2 Various Class A volumetric pipets and flasks.
5.3 Disposable Pasteur pipets - VWR, catalog #14670-103 or equivalent.

6 Reagents

6.1 Concentrated Nitric Acid (HNO_3) - Baker Instra-analyzed Reagent Grade, VWR, catalog #JT9598-34 or equivalent. Store at room temperature and dispose of by the manufacturer's expiration date.

6.2 Concentrated Hydrochloric Acid (HCl) - Baker Analyzed Reagent Grade, VWR catalog #JT9535-33 or equivalent. Store at room temperature and dispose of by the manufacturer's expiration date.

6.3 Reagent water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification system which provides analyte free, greater than 18.0 megohm-cm deionized water on demand. This water is used for preparation of all reagents and standards.

6.4 Liquid Argon Supply - High purity grade, purchased from MG Industries.

6.5 Stock Standard Solutions are purchased as commercially prepared NIST traceable certified solutions. When received in the lab each is assigned a unique log number and is recorded in the Standard Preparation Logbook along with the manufacturer, date of receipt, expiration date, and analysts initials. These standards are stored at room temperature and disposed of on or before the manufacturer's expiration date.

6.5.1 SM-1339-002 (STD MIX #2)Stock Solution in 5% HNO_3/Trace HF. High Purity, or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
Antimony (Sb)	20 mg/L
Bismuth (Bi)	100 mg/L
Boron (B)	100 mg/L
Cobalt (Co)	100 mg/L
Cooper (Cu)	100 mg/L
Iron (Fe)	100 mg/L
Manganese (Mn)	10 mg/L
Molybdenum (Mo)	100 mg/L
Nickel (Ni)	100 mg/L
Silver (Ag)	10 mg/L
Thallium (Tl)	10 mg/L
Tin (Sn)	10 mg/L
Titanium (Ti)	10 mg/L
Zinc (Zn)	50 mg/L

6.5.2 SM-1339-001 (STD MIX #1-R) Stock Solution in 5% HNO_3. High Purity Express, or equivalent NIST certified standard. This standard contains the

following elements:

ELEMENT	CONCENTRATION
Aluminum (Al)	100 mg/L
Arsenic (As)	10 mg/L
Barium (Ba)	100 mg/L
Beryllium (Be)	20 mg/L
Calcium (Ca)	100 mg/L
Cadmium (Cd)	10 mg/L
Chromium (Cr)	10 mg/L
Lead (Pb)	10 mg/L
Magnesium (Mg)	100 mg/L
Potassium (K)	2000 mg/L
Selenium (Se)	100 mg/L
Sodium (Na)	2000 mg/L
Strontium (Sr)	10 mg/L
Vanadium (V)	5 mg/L

6.5.3 QC26 Stock Solution in 5% HNO_3. QCD, catalog #QCS26K or equivalent. This standard contains the following elements:

ELEMENT	CONCENTRATION
Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, Zn, Al, Ba, B, Si, Ag, Na, K	all at 100 mg/l

6.5.4 Strontium Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog #140-051-382 or equivalent NIST certified standard.

6.5.5 Tin Stock Standard (1000 ppm) in 20% HCl. SCP, catalog #140-052-502 or equivalent NIST certified standard.

6.5.6 Bismuth Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog #140-051-832 or equivalent NIST certified standard.

6.5.7 Scandium Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog #140-051-215 or equivalent NIST certified standard.

6.5.8 INTER18 in 5% HNO_3. CPI, Cat. #4400-INTR18-100 or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
K	20000 mg/L

Se	500 mg/L
As, Pb, Tl	1000 mg/L
Ba, Cd, Cr	300 mg/L
Co, Cu, Ni	
Ag, V, Zn	
Mn	200 mg/L
Be	100 mg/L
Hg	50 mg/L

6.5.9 INTER5 in 2% HNO_3. CPI, Cat. #4400-INTR5-500 or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
Al	1200 mg/L
Ca	6000 mg/L
Fe	5000 mg/L
Mg	3000 mg/L
Na	1000 mg/L

6.5.10 Arsenic Stock Standard (1000ppm) in 4% HNO_3. SCP, catalog # 7697-37-2 or equivalent NIST certified standard.

6.6 Working Standard Solutions. Prepare in an acid matrix similar to the samples being analyzed. This is most often a 10% HNO_3 matrix, but is dependent on the type of digestion performed on the samples, and should be adjusted to match the samples being analyzed. After preparation, each standard is assigned a unique log number and is recorded in the standard preparation logbook along with the stock solution used, the concentration of that stock, the volume used, the final volume, the matrix, the date prepared, the date it will expire, and the preparer. Prior to analysis of DOD QSM samples, the following calibration standards (6.6.2 through 6.6.5) are to be prepared fresh before analysis. *(**Note:** The associated working standards are prepared daily, and held at room temperature. The maximum storage life is no longer than one day.)*

6.6.1 High Calibration Standard. To a 100 ml volumetric flask containing 10 ml HNO_3 (or that which matches the sample) in reagent water, add 5 ml HP1019-A-500 Stock Solution and 5 ml HP1019-B-500 Stock Solution. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working Calibration standard solutions are stable for 3 months.

6.6.2 Mid Calibration Standard. Add 4 ml of High Calibration Standard to 16 ml of calibration blank in a standard vessel for the Trace ICP. Mix solution by placing cap on vessel and inverting several times.

6.6.3 Low Calibration Standard. Add 2 ml of High Calibration Standard to 18 ml of calibration blank in a standard vessel for the Trace ICP. Mix solution by placing a cap on the vessel and inverting several times.

6.6.4 Calibration Blank (<detection limit (DL)). To 1000 ml volumetric flask, add 100 ml HNO_3 (or that which matches the samples) and bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. The calibration blank standard is stable for 3 months.
***(Note**: For all sequence runs that involve samples from the U.S. Army Corp., the calibration reagent blank must have a concentration less than ½ the reporting limit.)*

Calibration Standard Concentrations (mg/L)

Element	High Cal. Std.	Mid Cal. Std.	Low Cal. Std.
Ag	0.5	0.10	0.05
Al	5.0	1.0	0.5
As	0.5	0.10	0.05
Ba	5.0	1.0	0.5
Be	1.0	0.20	0.10
Ca	5.0	1.0	0.5
Cd	0.5	0.10	0.05
Co	5.0	1.0	0.5
Cr	0.5	0.10	0.05
Cu	5.0	1.0	0.5
Fe	5.0	1.0	0.5
Mg	5.0	1.0	0.5
Mn	0.5	0.10	0.05
Mo	5.0	1.0	0.5
Ni	5.0	1.0	0.5
Pb	0.5	0.10	0.05
Se	5.0	1.0	0.5
Sn	0.5	0.10	0.05
Sr	0.5	0.10	0.05
Ti	0.5	0.10	0.05
Tl	0.5	0.10	0.05
V	0.25	0.05	0.025
Sb	1.0	0.20	0.10
Bi	5.0	1.0	0.5
B	5.0	1.0	0.5
Zn	2.5	0.50	0.25

6.6.5 Working Profile Solution (5.0 mg/L). To a 1000 ml volumetric flask containing 100 ml HNO_3 (it is not critical for this standard to be matrix matched to the samples being analyzed) in reagent water, add 5 ml Arsenic Stock Standard. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.

6.6.6 Initial Calibration Verification Standard QC26 (1.0 mg/l). To a 100 ml volumetric flask containing 2 ml HNO_3 and 1 ml HCl (or that which matches the sample) in reagent water, add 1 ml QC26 Stock Solution, 0.1 ml Strontium Stock Standard, 0.1 ml Tin Stock Standard and 0.1 ml Bismuth Stock Standard. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.

6.6.7 Working Interference Check Solution. To a 1000 ml volumetric flask containing 100 ml HNO_3 (or that which matches the sample) in reagent water, add 2.5 ml INTER18 Stock Solution and 25 ml INTER5 Stock Solution. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.

ELEMENT	CONCENTRATION
Ag	0.75 mg/L
Al	30 mg/L
As	2.5 mg/L
Ba	0.75 mg/L
Be	0.25 mg/L
Ca	150 mg/L
Cd	0.75 mg/L
Co	0.75 mg/L
Cr	0.75 mg/L
Cu	0.75 mg/L
Fe	125 mg/L
Mg	75 mg/L
Mn	0.25 mg/L
Ni	0.75 mg/L
Tl	2.5 mg/L
V	0.75 mg/L
Zn	0.75 mg/L
Pb	2.5 mg/L
Se	1.25 mg/L
Na	25 mg/L
K	25 mg/L

6.6.8 RPL Standard (CRI). To a 100 ml volumetric flask containing 1ml HNO3 (or that which matches the sample matrix) in reagent water, add 0.5ml of RPL Stock Standard(6.10). Bring up to volume using reagent water. This standard should be prepared daily, and documented in the sample analysis logbook. The RPL / CRI concentrations are listed in Table 2.

6.7 Rinse Solution. To the rinse reservoir containing reagent water, add 300 ml HNO_3 (or that which matches the samples). Bring up to volume (3 L) using reagent water. Prepare

as needed.

6.8 Working Internal Standard Solution (5.0mg/l Sc). To a 2000 mL volumetric flask containing 40 mL HNO3 in reagent water, add 10 mL of Scandium Stock Solution. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working standard solutions are stable for 3 months when stored at room temperature.

6.9 Instrument Performance Check Solution (IPC). To a 100 ml volumetric flask containing 10 ml HNO_3 (or that which matches the sample) in reagent water, add 2.5 ml SM-1339-002(STD MIX #2)(See 6.5.1) and 2.5 ml SM-1339-001(STD MIX #1-R)(See 6.5.2). Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working Calibration standard solutions are stable for 3 months when stored at room temperature. This standard contains the following elements:

ELEMENT	CONCENTRATION
Ag	0.25 mg/l
Al	2.5 mg/l
As	0.25 mg/l
Ba	2.5 mg/l
Be	0.50 mg/l
Ca	2.5 mg/l
Cd	0.25 mg/l
Co	2.5 mg/l
Cr	0.25 mg/l
Cu	2.5 mg/l
Fe	2.5 mg/l
Mg	2.5 mg/l
Mn	0.25 mg/l
Mo	2.5 mg/l
Ni	2.5 mg/l
Pb	0.25 mg/l
Se	2.5 mg/l
Sn	0.25 mg/l
Sr	0.25 mg/l
Ti	0.25 mg/l
Tl	0.25 mg/l
V	0.125 mg/l
Zn	1.25 mg/l
Sb	0.50 mg/l
Bi	2.5 mg/l
B	2.5 mg/l

6.10 Report Limit Standard (RPL / CRI) is prepared by adding the following amounts of single element standard to a 200mL volumetric flask containing 5ml conc. HNO3. This working standard is stable for three months when stored at room temperature.

ELEMENT	Volume of 1000ppm Single Element Solution in 200mL.	Final Concentration (mg/L)
Aluminum	4.0 mL	20
Antimony	0.4 mL	2.0
Arsenic	0.32 mL	1.6
Barium	0.4 mL	2.0
Beryllium	0.16 mL	0.8
Bismuth	2.0 mL	10.0
Boron	4.0 mL	20.0
Cadmium	0.04 mL	0.20
Calcium	4.0 mL	20.0
Chromium	0.2 mL	1.0
Cobalt	0.2 mL	1.0
Copper	0.4 mL	2.0
Iron	2.4 mL	12.0
Lead	0.2 mL	1.0
Magnesium	4.0 mL	20.0
Manganese	0.2 mL	1.0
Molybdenum	0.8 mL	4.0
Selenium	0.4 mL	2.0
Silver	.16 mL	0.8
Nickel	0.8 mL	4.0
Tin	0.4 mL	2.0
Strontium	0.2 mL	1.0
Titanium	0.8 mL	4.0
Thallium	0.4 mL	2.0
Vanadium	0.2 mL	1.0
Zinc	0.8 mL	4.0

6.11 Silver Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-472 or equivalent NIST certified standard.

6.12 Aluminum Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-132 or equivalent NIST certified standard.

6.13 Arsenic Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-332 or equivalent NIST certified standard.

6.14 Barium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-562 or equivalent NIST certified standard.

6.15 Beryllium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-042 or equivalent NIST certified standard.

6.16 Calcium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-202 or equivalent NIST certified standard.

6.17 Cadmium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-482 or equivalent NIST certified standard.

6.18 Cobalt Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-272 or equivalent NIST certified standard.

6.19 Chromium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-242 or equivalent NIST certified standard.

6.20 Copper Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-292 or equivalent NIST certified standard.

6.21 Iron Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-262 or equivalent NIST certified standard.

6.22 Magnesium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-122 or equivalent NIST certified standard.

6.23 Manganese Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-252 or equivalent NIST certified standard.

6.24 Molybdenum Stock Standard (1000 ppm) in H_2O. SCP catalog #140-050-422 or equivalent NIST certified standard.

6.25 Nickel Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-282 or equivalent NIST certified standard.

6.26 Lead Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-822 or equivalent NIST certified standard.

6.27 Selenium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-342 or equivalent NIST certified standard.

6.28 Titanium Stock Standard (1000 ppm) in H_2O/Trace HF. SCP catalog #140-050-222 or equivalent NIST certified standard.

6.29 Thallium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-812 or equivalent NIST certified standard.

6.30 Vanadium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-232 or equivalent NIST certified standard.

6.31 Zinc Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-302 or equivalent NIST certified standard.

6.32 Antimony Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-512 or equivalent NIST certified standard.

6.33 Boron Stock Standard (1000 ppm) in H_2O. SCP catalog #140-050-052 or equivalent NIST certified standard.

7 Instrument Calibration

7.1 Immediately preceding calibration, each spectral line must be centered on its exit slit which is positioned in front of each photomultiplier tube. Maintaining this optical alignment during operation is called profiling.

7.1.1 Only the arsenic spectral line is profiled. All other lines are preset relative to arsenic.

7.1.2 To profile the instrument, aspirate the Working Profile Solution. From the main menu, enter Analysis. Enter the method name to be used and press Enter. Press F5, Profile. The profile line should read As 189.042/2.

7.1.3 Press F3, Automatic. When the Working Profile solution has reached the plasma, press F1, Run. After scanning is complete, the arsenic peak is displayed on the screen, along with the peak position, peak intensity and peak width.

7.1.4 In order to get the peak position as close to the center as possible, the peak position number should be as close to 0.00 as possible. This is accomplished by choosing F1, calc SS, and pressing enter. The new vernier position will be displayed on the screen. Adjust the vernier knob on the instrument manually to the value displayed on the screen. Then press F9, Done/Keep.

7.1.5 Go to step 7.1.3 and repeat the process until the peak position is as close to 0.00 as possible. Then press F9, Done/Keep and F9, Done. Allow the system to rinse for 5 minutes before starting calibration.

7.1.6 Reprofiling may be performed periodically throughout the analysis to compensate for diurnal changes which are apparent by drifting QCs. It must only be adjusted, however, after instrument check standards and calibration blank have been

analyzed.

7.2 The instrument prepares a standard curve by analyzing three levels of calibration standards and a calibration blank. Starting with the blank and working toward the high standard, the standards are aspirated and emission intensity readings are recorded by the data system.

7.3 All calibration standards are analyzed in duplicate and an average intensity is reported and used by the data system to prepare the calibration curve.
(Note: All runs involving USACE samples require three replicates for calibration standards.)

7.4 A daily calibration curve is created by plotting the average intensity readings on the y-axis and concentration readings on the x-axis. The software of the data system plots the curve in a linear configuration. The calibration curve occurring most immediately preceding a particular sample is used to calculate the concentration for that sample. The acceptance criteria for a calibration curve for all analytes is a correlation coefficient of 0.995 or greater.

7.5 The calibration curve is validated using instrument check solutions prepared at known concentrations from a different source than that of the calibration standards. Validation occurs immediately following calibration and then at a frequency of 10% throughout the analysis run.

8 Quality Control

8.1 Initial demonstration of performance

8.1.1 Linear Dynamic Range (LDR) - The upper limit of linearity must be established for each element being analyzed. Analyze succeedingly higher concentrations of the analyte until the percent recovery falls under 90%. The last concentration maintaining greater or equal to 90% recovery is considered the upper limit of linearity. Samples containing analytes greater than 90% of the upper limit of linearity must be diluted and reanalyzed for those analytes. The LDRs are verified annually or any time a change in operating conditions occurs that may change the LDR.
(***Note****: Linear Dynamic Ranges cannot be used when analyzing samples for the U.S. Army Corp. All samples must be diluted to analyte concentrations which fall within the calibration curve.)*

8.1.2 Method Detection Limits (MDL) - MDLs must be established and verified annually, and any time a change in operating conditions occurs that may change the MDL (reference 99-MDL). When analyzing USACE samples, MDL studies must be performed annually and an MDL Check Standard analyzed mid-year at

2X the MDL. If all metals are detected when analyzing the check standard, it is not necessary to analyze the MDL study during the second half of the year.

8.1.3 Demonstration of Capability (DOC) – DOCs must be performed yearly by each analyst prior to performing this method and repeated at any time there is a significant change in instrument type. To perform DOC's, four consecutive Laboratory Control Samples (LCS), with a matrix matching that of the calibration standards are analyzed. The recoveries obtained must be within 80-120% of the known values for each associated metal, and consecutive reads must have an RSD less than 20%. If the DOC's are outside these acceptance limits, a new calibration curve must be established, and the LCS's reanalyzed. This process is repeated until the DOC's are completed successfully.

8.1.4 Interelement correction factors must be verified and updated every six months or at any time a change in instrument operating conditions occurs which may change the interelement correction requirements.

8.1.5 Prior to performing analysis on NLLAP samples (IH), analysts will have read through the latest AALA lead requirements, and have demonstrated ability to produce reliable results through accurate analysis of standard reference material (ie. PAT rounds or ELPAT studies), or in-house quality control samples. Their performance must be documented in their training logs.

8.1.6 Analysts/Technicians involved in IH Lead analysis shall redemonstrate their ability to adequately analyze certified reference materials (ie. PAT rounds or ELPAT studies), on a bi-yearly basis. Their performance must be documented in their training log.

8.1.7 Contamination Control – Lead dust wipe sampling must be performed in all associated areas of the lab on a quarterly basis to determine surface concentrations of lead. Sample preparation and analysis is not to proceed until surface contamination is less that the specified maximum allowable limit of 40 micrograms per square foot.

8.2 Daily demonstration of instrument performance

8.2.1 Quality Control Sample (QC26) - Initial and periodic verification of calibration standards is necessary to verify instrument performance. To verify the calibration standards, the Working Quality Control Sample QC26 must be within +/- 10% of the true value immediately following the daily calibration. If outside of this acceptable range for target elements, the problem must be corrected by re-analysis, preparation, recalibration, or instrument maintenance. Samples may not

be analyzed until the problem has been corrected and a QCS has been recovered within acceptable range.

8.2.2 A laboratory method blank (MB) is prepared with every batch of samples or one per every 20 samples digested, if the batch contains more than 20 samples. The result must be less than 2.2 x the MDL for all required analytes. If the result does not meet this requirement, the samples in that prep batch must be either less than the reporting limit or greater than or equal to 10x the reagent blank value for the effected elements. If this criteria is not met, the effected samples in that batch must be redigested. For DOD QSM samples the acceptance criteria for these blanks will be one-half the reporting limit.
***(Note**: The method blank result must be equal to or less than ½ the reporting limit for all sequence batches that include samples from the U.S. Army Corp.)*

8.2.2.1 IH Lead Wipe QC. The wipe media used for the quality control samples (ie. MB / LCS / LCSD), should be of the same lot number or manufacturer as the wipes used for sample collection. If the samples are collected by an outside source, the lab is requesting that extra wipe media is provided to perform the required QC. If the wipe media is not provided by the client, then A.L.S.I will use in-house wipe media purchased from Environmental Express.

8.2.3 A laboratory fortified blank (LFB) is processed with every batch of samples, or one every 20 samples if the batch contains more than 20 samples. The LFB must be subjected to all sample preparation steps, such as digestion if necessary. The percent recovery must be 80-120% of the true spike value. If the recovery falls outside of this range, the source of the problem should be identified and resolved before continuing analyses. LFB results are documented in the ALSI LIM system.

8.2.3.1 To prepare a laboratory fortified blank while analyzing samples which do not require digestion, add 1 mL of HP1019-A-500 and HP1019-B-500 Stock Solutions to 100 mL of sample. This will result in a mid-level concentration for the LFB.

8.2.3.2 On a monthly basis, a LFB spiked at the reporting limit of each element must be analyzed to verify accuracy. Recoveries must be within +/- 50% of the true value. If recoveries fall outside of this range the source of the problem should be identified and resolved before continuing analyses.

8.2.4 Instrument Performance Check Solution (IPC) is analyzed following the calibration, after every 10 samples and at the end of the run. The results of the analytes in the check solution immediately following calibration must be within +/- 10% of the true value with <5% RSD between replicates. Subsequent analyses

of the IPC solution must be within +/- 10% of the true value. If the result falls outside of this range for any element, the IPC may be rerun for that element. If the result still falls outside of this range, the problem must be corrected by reprofiling, preparation of new IPC, recalibration, or instrument maintenance. Samples following the last acceptable IPC requiring the elements that failed must be reanalyzed after correction of the problem and successful analysis of the IPC. All results of IPCs are documented in the ALSI LIM system.

8.2.5 The Working Interference Check Solution is analyzed to provide an adequate test of the inter-element and background correction factors. It must be analyzed at the beginning and end of an analytical run or twice during every 8-hour shift, whichever is more frequent. The result must be within +/- 20% of the true value. If the results fall outside of this range, the interference check solution may be rerun. If it is still outside this range for any element, the problem must be corrected by preparation of new working interference solution, recalibration, or adjustment of inter-element and background correction factors. Any sample requiring an element that fails must be rerun for that element after the problem had been corrected and the interference check successfully meets the criteria.

8.2.6 Calibration reagent blanks are analyzed directly after each IPC. The result for every element being analyzed must be less than 2.2 x the MDL. If the result does not meet this requirement for any element, the calibration blank may be rerun for that element. If the result is still not acceptable, the problem must be corrected by preparation of new blank, recalibration, or instrument maintenance. Samples following the last acceptable calibration reagent blank requiring the elements that failed must be reanalyzed after correction of the problem and successful analysis of the calibration reagent blank.

8.3 Daily demonstration of data quality

8.3.1 A matrix spike sample is processed at a frequency of 10% of the routine samples. Per EPA Method 6010 requirements the percent recovery must be 75-125% of the true spike value. Recovery calculations are not required if the concentration of the spike added is greater than ten times the sample background and a comment must be placed on the lab report. If the recovery falls outside of the acceptable range, and the system is found to be in control (Section 8.2), the recovery problem is judged to be matrix related and not system related. To determine if the method of standard additions is necessary, the sample must be post spiked or diluted as described below. Results of all matrix spikes are documented in the ALSI LIM system.

% Recovery *= MS Conc. – Sample Conc. / Actual Spike Concentration x 100*

8.3.2 Analyte Addition Test. To prepare a post spike, add an amount of high standard

that will produce a minimum level of 20x and a maximum level of 100x the MDL and analyze. The percent recovery must be 75-125% of the true value as determined by the following formula. If the result is outside of this range, make successive dilutions of the sample and re-spike until the recovery falls within this range. The reporting limit of the sample must then be raised to reflect the dilution used. If this raises the reporting limit higher than the client needs, the method of standard additions should be performed. NOTE: If the analyte concentration is greater than ten times the matrix spike concentration, post-spikes are not required, but a comment must be added to the lab report stating "No Spike Calculated".

$$A = \frac{B - (\frac{E}{F})(C)}{(\frac{E}{F})(D)} \times 100$$

where: A = Post spike percent recovery
B = Resulting spike concentration
C = Sample concentration
D = Working calibration standard concentration
E = Amount of spike added (ml)
F = Final volume spike solution + sample

8.3.3 Dilution Test. If (100 x MDL) <20% of the sample concentration, prepare a 1/5 dilution on the sample and reanalyze. The resulting corrected concentration should be within +/- 10% of the original sample concentration. If not, a matrix effect should be suspected, and the sample diluted until the matrix problem has been eliminated. If this raises the reporting limit higher than the client needs, the method of standard additions should be performed.

8.3.4 A matrix spike duplicate or sample duplicate is processed at a frequency of 5% of the routine samples. The relative percent difference (RPD) must be within 20%. If the RPD falls outside of the acceptable range, and the system is found to be in control, the precision problem is judged to be matrix related and not system related. A comment must be added to the lab report. Results of all duplicate analyses are documented in the ALSI LIM system.

***RPD** = Difference / Average x 100*

8.4 Method of Standard Additions - The method of standard additions is used when sample dilution and spikes fail to produce good recoveries. In the standard addition technique, two identical aliquots (Volume Vx) of the sample solution are taken. To the first, (labeled A), is added a small a volume (Vs) of a standard analyte solution of concentration Cs. To the second (labeled B), is added the same volume Vs of the matrix blank. The intensity counts of A and B are measured and corrected for non-analyte intensity counts. The unknown sample concentration (Cx) is calculated as follows:

$$Cx = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where:

S_A = intensity counts of A corrected for the blank
S_B = intensity counts of B corrected for the blank

Vs and Cs should be chosen so that S_A is roughly twice S_B on the average. It is best if Vs is much less than Vx, and thus Cs is much greater than Cx, to avoid excess dilution of the sample matrix. If a concentration or separation step is used, the additions are best made first and carried through the entire procedure. For results from this technique to be valid, the following limitations must be taken into consideration:

(1) The analytical curve must be linear.
(2) The chemical form of the analyte added must respond the same as the analyte in the sample.
(3) The interference effect must be constant over the working range of concern.
(4) The signal must be corrected for any additive interference.

8.5 Samples resulting in high negative (|conc.| > reporting limit) concentrations must be post-spiked (see 8.3.2) to determine if there exists a negative interference. If multiple samples containing the same matrix (from the same source or client) show the same negative trend, only one sample of this matrix needs to be post-spiked.

8.6 When analyzing using a scandium internal standard, the intensity of scandium in each sample must be within +/- 20% of the intensities in the initial calibration blank. If the intensities fall outside of this range, re-analyze the sample at a dilution to eliminate matrix interferences.

8.7 Each sample, QC check, and calibration standard is analyzed in duplicate and the results averaged. The relative standard deviation (RSD) between sample replicates must be less than 20% for all concentrations greater than the reporting limit to be accepted. If the RSD is greater than 20% and the sample concentration is above the reporting limit, the sample must be reanalyzed.
(Note: All runs involving USACE samples require three replicates for standards, samples, and QC Checks.)

8.8 It is recommended that whenever a new or unusual sample matrix is encountered, either of the following tests be performed to determine if either positive or negative matrix interferences are present to distort the accuracy of the reported values.

8.8.1 Serial dilution. If the analyte concentration is at least 40x the detection limit, a

1:4 dilution should agree within +/- 10% of the original determination. If not, a chemical or physical interference should be suspected and must either be diluted out and the detection level raised or the sample may be analyzed by the method of standard addition.

8.8.2 Post digestion spike. A post digestion spike prepared as directed in 8.3.2, should be recovered within 75 to 125% of the true value. If the spike is not recovered and the necessary sample dilution to recover the analyte concerned raises the detection limit of the sample above the limit needed by the client, the method of standard additions must be used.

8.9 ALSI participates regularly in applicable performance evaluation studies conducted by various certifying organizations.

8.10 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Calibration Reagent Blank	--	Beginning of run, after every 10 samples, and at the end of the run.	< 2.2 x MDL	Reanalyze the blank, prepare new blank and analyze, perform maintenance on instrument, recalibrate, reanalyze any samples since the last acceptable blank. If reanalysis is not possible, report with a qualifying comment.
Method Blank (MB)	--	One per batch of no more than 20 samples. Analyze with associated sample batch.	< 2.2 x MDL or <Reporting Limit or < ½ reporting limit for DOD QSM samples	Reanalyze the blank. Samples in the batch must be < the reporting limit or ≥ 10x the method blank. If not, samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment. For DOD QSM samples the method blank must be ½ the reporting limit (see additional requirements in Section 8.12).
High Calibration Standard	See Section 6.6.1	After calibration and before analysis of samples.	90-110%	Reanalyze the High Standard. If the standard is still not acceptable, reprofile and/or perform instrument maintenance, and prepare a new calibration.
*Laboratory Fortified Blank (LFB or LCS)	Listed in Table 3	One per batch of no more than 20 samples. Analyze with associated sample batch.	80-120%	Reanalyze the LFB. If still outside of acceptable range, samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.

Quality Control Sample (QCS) Second Source Standard	1.0 mg/L	Immediately after calibration.	90-110%	Reanalyze the QCS. If the standard is still not acceptable, reprofile and/or perform instrument maintenance, and prepare a new calibration.
Instrument Performance Check Solution (IPC) Same Source	Listed in Table 4	Beginning of run, after every 10 samples, and at the end of the run.	90-110%	Reanalyze the IPC. If the standard is still not acceptable, reprofile and/or perform instrument maintenance, and prepare a new calibration. Reanalyze any samples since the last acceptable IPC. If reanalysis is not possible, report with a qualifying comment.
Reporting Limit Standard (RPL)	Listed in Table 2	Beginning of run, after calibration.	50-150% *U.S. Army Corps. (80-120%)*	Reanalyze the RPL. If the standard is still not acceptable, reprofile and reanalyze. If still outside of range, recalibrate.
* Matrix Spike (MS)	Listed in Table 3	One every 10 samples with at least one per batch.	75-125%	If calibration verification standards are acceptable, reanalyze spike once. If the spike still fails perform a post-spike. Post spikes must be recovered at 85-115%. If not or if reanalysis is not possible, report the results with a qualifying comment.
*Duplicate or matrix spike duplicate (MSD)	--	One every 10 samples with at least one per batch.	RPD ≤ 20%	Reanalyze the duplicate. If the RPD is still >20% or if reanalysis is not possible, report the results with a qualifying comment.
Sample replicates	--	Every sample	RSD < 20% for all samples > reporting limit	Reanalyze the sample. If the RSD is still > 10% or if reanalysis is not possible, report the results with a qualifying comment.
Samples with high negative concentration	--	--	(|conc.| < reporting limit	Post-spike sample to determine if there exists a negative interference. If multiple samples containing the same matrix (from the same source or client) show the same negative trend, only one sample of this matrix needs to be post-spiked.
Scandium Internal Standard	--	Every sample	Intensity must be within 30% of the intensity of the initial calibration blank	Reanalyze the sample at a dilution to eliminate sample matrix interference.

Interference Check Solution	See Section 6.6.7	Beginning and end of every run, or every 8 hours.	80-120%	Rerun the interference check solution, preparation of new solution, recalibrate, or adjustment of interelement correction factors. Rerun any samples requiring an element that fails, or if re-analysis is not possible, report the results with a qualifying comment.

* Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

- LCS recoveries and duplicate precision limits stated in the QC Chart are also used for all IH analyses.

9 Sample Collection, Preservation and Handling

9.1 Sample Collection.

9.1.1 Samples must be collected in plastic or glass containers.

9.1.2 Soil/Sediment/Solid samples can be collected in plastic or glass containers.

9.1.3 A minimum of 3.00g for soil samples, and 150mL for water samples must be supplied by the sampler in order for the laboratory to perform analysis.

9.2 Sample Preservation

9.2.1 Preserve aqueous samples using HNO_3 to a pH<2. Sample preservation should be performed immediately upon sample collection. If this is not possible, then samples should be preserved ASAP when received by the laboratory.

9.3 Sample Handling

9.3.1 All samples must be analyzed within 180 days of collection. All samples not analyzed within this time frame must be discarded and resampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report.

9.3.2 Soil samples must be preserved at 1-4° C until analysis.

9.3.3 Water samples and sample digestates should be stored at room temperature both before and after analysis.

9.3.4 All samples and digestates must be held by the laboratory for a minimum of two weeks after the lab reports have been sent to the client.

9.3.5 For samples requiring digestion, refer to the Sample Preparation SOPs for procedures.

10 Procedure

10.1 Initial Set-up and Analysis for the TJA Trace ICP

10.1.1 Perform the daily and as needed maintenance.

(1) Refill the rinse solution.
(2) Replace the sample and rinse pump tubing.
(3) Inspect all other autosampler tubing for clogs and/or visible leaks.
(4) Inspect the nebulizer for clogs and position in the spray chamber.
(5) Inspect the spray chamber for cleanliness and clean if needed.
(6) Set the incoming Argon pressure to 70 psi.
(7) Clean the torch and tip if needed.
(8) Adjust the tension on the pump tubing in the peristaltic pump.
(9) Check the vacuum gauge. (This must read below 30 millitorr. If the pressure rises above 30, the vacuum pump oil must be changed.)
(10) Check to be sure the water cooler is on and is filled to the proper level.
(11) Check to be sure the hood is operating.
(12) Empty the waste containers.
(13) Check the reagent water level in the argon humidifier and refill if necessary.

10.1.2 Turn the computer on and using the arrow keys, move to Plasma Control Panel under Set-up. Press Enter.

10.1.3 Press F1, Start-up and then press F9, continue. After purging for 90 sec. the torch will automatically be ignited. Press Enter.

10.1.4 Press Esc. Use the arrow keys to move to Operation. Press Enter. After the Enter Method Name prompt, type in OPTIMIZE. The instrument will load and set the parameters, such as nebulizer pressure and pump speed, specified in this method. Put the probe of the autosampler in the rinse solution.

10.1.5 Allow the instrument to become thermally stable before beginning. This requires at least 30 minutes of running while aspirating rinse solution.

10.1.6 Profile the instrument using the Working Profile Solution as described in 7.1. When an acceptable profile is reached, print the screen by pressing Control F2, Print screen.

10.1.7 Leaving the probe in the profile solution, press F1, Analyze and F1, Run. The method will run 10 replicates of the Profile solution and print the %RSD. Manually adjust the position of the nebulizer and the nebulizer flow rate in method development to bring the intensity as close to 300000 counts as possible with the %RSD under 0.50 for the 10 replicates. When an acceptable %RSD is achieved, print by pressing Control F2, Print Screen. Press F9, Done/Keep. The nebulizer flow rate chosen to give the best response should then be entered into the sample analysis method by moving to Methods under Development. Press Enter. At the Enter Method Name prompt, enter the correct method. Press Enter. Then press F6, Plasma Info. Adjust the nebulizer pressure and save by pressing F9, Done/Keep.

10.1.8 The following parameters should also be checked while still in Methods. When everything has been set correctly, save any changes made to the method by pressing F9, Done/Keep. Print the method by reentering Methods and inputting the correct method. Press Enter. Press F8, Options. Press F2, Print method. When the method is done printing, save again by pressing F9, Done/Keep twice.

Sample Introduction Device: Normal
Calibration Mode: Concentration

Number of Repeats: 2 ***(3** Replicates are required for U.S. Army Corp.)*
Flush Time: 65.0 sec
Auto-Store Analysis Data? Yes
Auto-Store Stdzn Data? Yes
Store Individual Repeats? No
Autoprint Analysis Data? Yes
Autoprint Stdzn Report: None
Condensed Print Format? No

Output Mode: Concentration
Override Print Limits? Yes
Override Sig. Figures? No
Apply Background Correction? Yes
Apply Blank Subtraction? No

Torch gas: High Flow
Auxilary Gas Flow: Low (0.5 L/min)
Nebulizer Pressure: See step 10.1.7

Approximate RF Power: 950

Analysis Pump Rate: 150
Flush Pump Rate: 150
Relaxation Time (sec): 0
Pump Tubing Type: Tygon-Orange

10.1.9 Use the arrow keys to move to Operation, Autosampler set-up to create a sequence table for the Autosampler. Press Enter, then F1, Edit Set. Fill in the parameters, then press F1, Edit Samples. Build the table and save by pressing F9, Done/Keep three times. The computer will dictate the positions for the calibration blank and standards as well as the QCs that the analyst has put into the sequence table. Rinse and refill the calibration standard, blank, and QC sample wells in the Autosampler and insert them in the correct positions.

10.1.10 Go into Operation, Analysis. Enter the method name. Press Enter. Press F9, Autosampler. Enter the name of the Autosampler table to be used. Press F1, Run. The instrument will, with the use of the Autosampler and peristaltic pump, start to standardize the instrument.

10.2 Analyze by the method of standard addition (see Sect. 8.9) any samples containing matrix interferences which cannot be eliminated by dilution.

10.3 For the inter-element spectral interference correction factors to remain valid, the interferant concentration must not exceed its limit of linearity. Sample dilution is necessary in these cases, and the reporting limit must be raised to reflect the dilution performed.

11 Calculations

11.1 Sample results are reported directly from the readout of the instrument (from the calibration curve), and input into the LIMS. Appropriate prep factors are applied to the result at the time of supervisor approval.

11.2 Any sample result requiring dilution to bring the sample into the linear range, is multiplied by the dilution factor before being entered into the LIMS using the following equation:

$$A = \frac{Z\,(B)}{C}$$

where A = concentration of element in sample
Z = concentration of element in diluted sample
B = final volume of dilution (ml)
C = volume of sample aliquot used in dilution (ml)

12 Reporting Results

12.1 Report results in the Horizon LIMs system: All results available on the raw should be entered into the LIMs. This will allow the laboratory to provide j-values to clients when they are needed. When j-values are not requested, the LIMS will automatically round results off to 3 significant figures after all internal calculations are completed.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

Appendix A

Trace ICP
Analytical Worksheet

Acid Matrix: ___% HNO3 + ___% HCl

Instrument Method:_____
Sequence #:_____
Date Started:_____
Analyst:_____

*High Std:_____ml each of _____+_____ into _____ml
*Mid Std:_____ml High Std into _____ml
*Low Std:_____ml High Std into _____ml
*CCV/IPC:_____ml each of _____+_____ into _____ml
*Second Source:_____ml of _____+
_____ml each of _____+_____+_____ into _____ml
*Additional Second Source:_____ml of _____+_____ into _____ml
*Cal Blank:_____ml HNO3 Lot #:_____ HCl Lot #:_____

ICS:_____
RPL:_____ml_____into_____ml

* All Standard are prepared daily prior to analysis of samples in acid matrix listed

PDS: 3.5 ml sample + 3.5 ml High Std

BLANK		
LOW STD		
MID STD		71
HIGH STD		72
SECOND SOURCE	31	73
	32	74
	33	75
	34	76
	35	77
	36	78
	37	79
	38	80
	39	
	40	
		81
		82
	41	83
	42	84
1	43	85
2	44	86
3	45	87
4	46	88
5	47	89
6	48	90
7	49	
8	50	
9		
10		
		91
	51	92
	52	93
11	53	94
12	54	95
13	55	96
14	56	97
15	57	98
16	58	99
17	59	100
18	60	
19		
20		
		101
		102
	61	103
	62	104
21	63	105
22	64	106
23	65	107
24	66	108
25	67	109
26	68	110
27	69	
28	70	
29		
30		

TABLE 1
(Element Wavelengths)

Element	Wavelength
Ag	328.068
Al	308.215
As	189.042
B	249.678
Ba	493.409
Be	313.042
Bi	223.061
Ca	317.933
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
Fe	271.441
Mg	202.030
Mo	202.030
Mn	257.610
Ni	231.604
Pb	220.353
Sb	206.838
Se	361.384
Sn	189.989
Sr	421.552
Ti	334.941
Tl	190.864
V	292.402
Zn	213.856

TABLE 2
(Reporting Limit Concentrations)

Element	Concentration
Ag	0.004
Al	0.10
As	0.008
B	0.10
Ba	0.010
Be	0.004
Bi	0.05
Ca	0.10
Cd	0.001
Co	0.005
Cr	0.005
Cu	0.01
Fe	0.06
Mg	0.10
Mo	0.02
Mn	0.005
Ni	0.02
Pb	0.005
Sb	0.01
Se	0.01
Sn	0.01
Sr	0.005
Ti	0.02
Tl	0.01
V	0.005
Zn	0.02

TABLE 3
(Laboratory Fortified Blank and Matrix Spike Concentrations)

Element	Concentration (mg/L)
Ag	0.100
Al	1.00
As	0.100
B	1.00
Ba	1.00
Be	0.200
Bi	1.00
Ca	1.00
Cd	0.100
Co	1.00
Cr	0.100
Cu	1.00
Fe	1.00
Mg	0.100
Mo	1.00
Mn	0.100
Ni	1.00
Pb	0.100
Sb	0.20
Se	1.00
Sn	0.10
Sr	0.10
Ti	0.10
Tl	0.10
V	0.050
Zn	0.50

TABLE 4
(Interference Check Solution Concentrations)

Element	Concentration
Ag	0.75
Al	30.0
As	2.50
Ba	0.75
Be	0.25
Ca	150
Cd	0.75
Co	0.75
Cr	0.75
Cu	0.75
Fe	125
Mg	75.0
Mn	0.50
Ni	0.75
Pb	2.50
Se	1.25
Tl	2.50
V	0.75
Zn	0.75

SOP Change History Sheet

Section No.	Section	Reason for Change
2	Summary of Method	U.S. Army Corp. audit response
6.1	Reagents	Sop Update (02/24/05)
6.2	Reagents	Sop Update (02/24/05)
6.5.1-6.5.2	Reagents	SOP Update
6.5.10	Reagents	SOP Update
6.6.4	Reagents	U.S. Army Corp. audit response
6.6.5	Reagents	Sop Update (02/24/05)
6.6.6	Reagents	Sop Update (02/24/05)
6.6.7	Reagents	Sop Update (02/24/05)
6.6.8	Reagents	SOP Update (01/24/05)
6.8-6.10	Reagents	SOP Update (02/24/05)
6.9-6.11	Reagents	SOP Update
7.3	Instrument Calibration	Sop Update (02/24/05)
7.4	Instrument Calibration	Sop Update (02/24/05)
8.1.1	Quality Control	U.S. Army Corp. audit response
8.1.3	Quality Control	SOP Update
8.1.5	Quality Control	SOP Update (01/24/05)
8.1.6	Quality Control	SOP Update (01/24/05)
8.1.7	Quality Control	SOP Update (01/24/05)
8.2.2	Quality Control	U.S. Army Corp. audit response
8.2.2.1	Quality Control	SOP Update (01/24/05)

8.3.1	Quality Control	SOP Update
8.3.4	Quality Control	SOP Update (02/24/05)
8.7	Quality Control	Sop Update (02/24/05)
8.12	Quality Control	Section Removed
9.3.2-9.3.4	Sample Handling	SOP Update
10.1.8	Procedure	U.S. Army Corp. audit response
12.1	Reporting Results	SOP Update
12.2 / 12.3	Reporting Results	Sections Removed
	Appendix A (Analytical Worksheet)	U.S. Army Corp. audit response
	Table 2 (RPL Concentrations)	SOP Update
	Table 3 (LCS / MS Concentrations)	SOP Update
	Table 4 (Interference Check Concentrations)	SOP Update

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

Document Title: Mercury by Cold-Vapor Atomic Absorption Using an Automated Continuous-Flow Vapor Generator

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Anna Milliken Date
Laboratory Operations Manager

_____ _____
Natalie Hufford Date
Validator

TABLE OF CONTENTS

1 Scope and Application 3

2 Summary of Method 4

3 Interferences 4

4 Safety 4

5 Apparatus and Materials 5

6 Reagents 6

7 Instrument Calibration 6

8 Quality Control 7

9 Sample Collection, Preservation and Handling 10

10 Procedure 11

11 Calculations 13

12 Reporting Results 14

13 Waste Disposal 15

14 Pollution Prevention 15

15 Definitions 15

16 Troubleshooting 16

APPENDIX A 17
APPENDIX B 23
APPENDIX C 28
Change History Summary 30
SOP Concurrence Form 33

1.1 This document states the policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.2 This method is adapted from EPA Method 245.1, Revision 3.0, May 1994; SW-846 Method 7470A, Mercury in Liquid Waste, September 1994 Revision 1; and, Method 7471B, Mercury in Solid or Semisolid Waste, January 1998 Revision 2; Method 7000 Atomic Absorption Methods, July 1992, Revision 1

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of cold vapor analysis. Each analyst must also be skilled in the interpretation of raw data, including quality control data.

1.4 This method measures total mercury (organic-inorganic) in drinking, surface, saline, and ground waters, domestic and industrial wastes, and mobility-procedure extracts. It also applies to soils, sediments, bottom deposits, and sludge-type materials.

1.5 In addition to inorganic forms of Mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system.

1.6 All samples must be digested prior to analysis.

1.7 Method Detection Limits can be found in the metals department method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.8 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 The flameless AA procedure is a physical method based on the absorption of radiation

at 253.7 nm by mercury vapor. The samples/standards and reagents are pumped into the analyzer and mixed. Argon gas is introduced into the solution stream, which flows to a mixing coil where the samples and reagents are thoroughly combined in the mixing coil. The gas and liquid stream is transferred to the gas/liquid separator where the gas and liquid phases are separated. The liquid waste is drained off and the gas is pumped to the absorption cell. The absorption cell is positioned in the light path of the mercury lamp. Absorbance (peak height) is measured as a function of mercury concentration and recorded as ppb of mercury.

3 Interferences

3.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL). During the oxidation step, chlorides are converted to free chlorine, which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

3.4 Interference from certain volatile organic materials which will absorb at this wavelength is also possible. All positive samples must be checked for false increases due to organics by analysis without the addition of stannous chloride.

4 Safety

4.1 Operation of an atomic absorption spectrophotometer involves the use of argon gas and hazardous materials including corrosive fluids. Unskilled, improper, and careless use of equipment can create explosion hazards, fire hazards or other hazards, which can cause death, serious injury to personnel, or severe damage to equipment or property.

4.2 Caution shall be taken when handling all samples, standards, and QC material because of the acidic nature of the prepared samples as well as the possible mercury content in the samples.

4.3 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.

4.4 Proper personal protective equipment must be used, including gloves, safety glasses, and lab coat.

4.5 The fume hood must be turned on during the analysis of mercury to vent the waste vapor.

5 Apparatus and Materials

5.1 PSA Automated Mercury Analyzer

5.1.1 CETAC M-6100 Mercury Analyzer.

5.1.2 PSA Automated Mercury Analyzer.

5.2 Green-Green Santoprene tubing: P S Analytical, cat. #M025T002

5.3 Yellow-Yellow Santoprene tubing: CETAC SP5705A

5.4 Black-Black Santoprene tubing: CETAC SP5705B

5.5 Finnpipette with disposable tips: 2 mL – 10 mL VWR, cat# 53516-178 or equivalent
0.2 mL – 1 mL VWR, cat #53515-876 or equivalent
20 µL – 200 µL VWR, cat#53503-094 or equivalent

5.6 Various Class A volumetric glassware.

5.7 Various calibrated dispensers.

5.8 40 mL VOA vials: Industrial Glassware #2795FL-PC.

5.9 25 mL graduated cylinder: Class A.

5.10 8 mL polystyrene tubes: VWR 60818-849 or equivalent

5.11 14 mL polypropylene tubes: VWR #60818-618 or equivalent.

6 Reagents

6.1 Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification System, which provides analyte-free DI water greater than 16.0 megohm on demand. This water is

used for preparation of all reagents, calibration standards, and as dilution water.

6.2 Liquid Argon: high purity grade, MG Industries or equivalent.

6.3 Stannous Chloride: Prepare by adding 100 g of stannous chloride crystal (VWR, catalog #EM-SX0885-1 or equivalent) to a 1000 mL volumetric flask. Add 14.0 mL conc. H_2SO_4 and stir until dissolved. Bring up to volume with reagent water. Stored at room temperature. Maximum storage life for stannous chloride is approximately 12 hours.

6.4 Sulfuric Acid, conc. Baker Instra-analyzed grade: VWR, cat. #JT9673-33 or equivalent. Store at room temperature up to the manufacturer's expiration date.

6.5 Sodium Chloride (NaCl.) Baker Instra-analyzed grade: VWR, cat. #JT3625-15 or equivalent. Store at room temperature up to the manufacturer's expiration date.

6.6 Hydroxylamine hydrochloride decolorizing reagent: To prepare, dissolve 120 g Hydroxylamine hydrochloride crystals (VWR, cat. #JT2196-1 or equivalent) and 120 g NaCl in reagent water in a 1000 mL volumetric flask. Bring up to volume using reagent water. Stored at room temperature. Maximum storage life is 3 months from date of preparation. The amount used for decolorizing is dependent upon the type of analysis being performed. Water samples require 1.5 mL and solid sample require 6.0 mL.

7 Instrument Calibration

7.1 The instrument plots a standard calibration curve each day of use using five standards and a blank. The calibration standards for EPA Method 245.1 and 7470 are Blank, 0.2 µg/L, 1.0 µg/L, 2.0 µg/L, 4.0 µg/L, and 10.0 µg/L. The calibration standards for EPA Method 7471 are Blank, 0.5 µg/L, 1.0 µg/L, 2.0 µg/L, 4.0 µg/L, and 10.0 µg/L. The calibration standards used for low level requests are 0.050 µg/L, 0.075 µg/L, 0.100 µg/L, 0.200 µg/L, and 0.500 µg/L. Starting with the blank and working toward the high standard, the standards are introduced into the mercury analyzer by the autosampler. Absorbance readings are recorded by the data system.

7.2 A calibration curve is drawn by plotting the absorbance readings on the y-axis and concentration readings on the x-axis. The software of the data system plots the curve. The calibration curve is used to calculate the concentration for the samples. The correlation coefficient must be 0.995 or greater.

7.3 A set of calibration standards is prepared along with every batch of mercury samples digested. It is these standards, which must be used to prepare the calibration curve for that batch of samples.

7.3.1 This is especially important because Method 245.1, Method 7470 and Method 7471 batches are prepared differently. Therefore these standards shall never be interchanged.

7.4 An Initial Calibration Verification (ICV) must be analyzed after every calibration to verify the instrument performance during analysis. The ICV is prepared from the second source standard. Analysis of the ICV immediately following calibration must verify that the instrument is within ± 5% of calibration for EPA Method 245.1, and ±10% for EPA Methods 7470/7471. Subsequent analysis of this standard is called the continuing calibration verification standard (CCV) and must be within ±10% of calibration. If outside of this range, determine and correct the problem. If necessary, recalibrate. Samples may not be analyzed until an acceptable ICV/CCV is analyzed.

7.5 Laboratory Control Sample (LCS). A digested standard must be analyzed with each batch and after every calibration. It is prepared at 2.0 ppb from the same source as that of the calibration standards. The recovery must be within ± 15% of the true value for the calibration. If outside of this range, determine and correct the problem and re-analyze. If necessary, recalibrate. Samples may not be analyzed until an acceptable LCS is analyzed

7.6 If the calibration blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

7.6.1 If the calibration blank concentration is greater than or equal to 2x the MDL, the source must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement. **(DoD Requirements)**

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 A demonstration of capability shall be performed before any client samples are analyzed. The DOC is determined by the analysis of four different Laboratory Control Samples (LCS). The percent recovery of the four LCSs must be ± 15% and the percent RPD must be $\leq$ 10%. If demonstration of capability fails, reanalyze. Ongoing proficiency must be established annually as specified in the QA plan, Technical Training.

Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Acceptance Criteria	Corrective Action
Calibration Blank (ICB/CCB)	NA	Prepared with each batch of samples. Analyzed after every ICV/CCV, at a minimum frequency of 10% and after calibration.	< MDL	Re-analyzed the blank. If still out of range, the problem must be solved by preparing a new blank, recalibration, or instrument maintenance. Samples following the last acceptable blank must be rerun.
Method Blank	NA	One digested with each batch of 20 or less samples. They are analyzed with that batch of samples.	1/2 the Reporting Limit	Re-analyze the blank. The samples in the prep batch must be less than the reporting limit or greater than 10X the reagent blank value for the affected analyte. It not, the affected samples in that batch must be re-digested. If re-digestion is not possible, they will be reported with a qualifying comment.
Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB)	Water: 2.0 ug/L Soil: 100 ug/kg Low detection waters: 0.070 ug/L	One digested with each batch of 20 or less samples. They are analyzed with that batch of samples.	85-115% R As required by the Method	Re-analyze the LCS. If the recovery is still outside the given range, the source of the problem must be identified and corrected before continuing analyses. If the problem cannot be identified, the samples in that batch must be re-digested. If re-digestion is not possible, report with a qualifying comment.
Matrix Spike (MS)*	Water: 5.0 ug/L Soil: 250 ug/kg Low detection waters: 0.200 ug/L	Frequency of 10% per matrix per batch	80-120% R As required by the Method	Re-analyze the MS. If still out of range analyze a post digestion spike (85-115%). If still out of range, a qualifying comment on the final lab report.
Parameter	**Concentration**	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Matrix Spike Duplicate (MSD) or Duplicate (Dup)*	Water: 5.0 ug/L Soil: 250 ug/kg Low detection waters: 0.200 ug/L	Frequency of 10% (**DoD samples** - 100% frequency)	<20% RPD As required by the Method	Re-analyze the duplicate. If the sample is outside the range, redigest the sample. If still outside of acceptable limits, report with a comment on the lab report.

Initial Calibration Verification Standard (ICV) (Second Source)	4.0 ug/L Low detection waters: 0.100 ug/L	Immediately after calibration.	Immediately after calibration ± 5%R (245.1); ± 10%R (7470/7471).	Re-analyze the ICV. If still out of range, the problem must be identified and corrected before analyzing any samples. Any samples analyzed after the last acceptable ICV/CCV must be re-analyzed.
Continuing Calibration Verification Standard (CCV) (Same Source)	4.00 ug/L for Methods 245.1 & 7470 and 4.0 ug/L used for Method 7471 and Low detection waters: 0.100 ug/L	Immediately after calibration, after every ten samples, and after the last sample.	Immediately after calibration ± 5%R (245.1); ± 10%R (7470/7471). Thereafter it must be within ± 10%R.	Re-analyze the ICV. If still out of range, the problem must be identified and corrected before analyzing any samples. Any samples analyzed after the last acceptable ICV/CCV must be re-analyzed.

* Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

Note: See Section 11.4 for % recovery calculations.

8.3 For samples with results greater than the highest standard will be diluted and reanalyzed until the concentrations are within the calibration range.

8.4 Method detection limits are determined annually using the procedure outlined in the ALSI Quality Assurance Plan. NOTE: If **DoD samples** are to be analyzed, an MDL check sample will be used to verify the MDL. The MDL check sample is at a concentration equal to 2 x the MDL. If a positive response is detected from the MDL check sample, another MDL study is not needed for that calendar year. Theses studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.

8.4.1 Practical Quantitation Limits (PQL) or reporting limits are determined by multiplying the MDL by 3-5 times, and adding an appropriate safety factor.

8.5 If the matrix spike fails criteria, a post digestion spike is performed. If the recovery of the post digestion spike is within 85-115%, the results will be reported. If outside of this range, comment on the final report. If the LCS is acceptable and the specific matrix interference is identified, report with a qualifying statement. If the specific matrix interference is unknown, reanalyze the sample and matrix spike to determine matrix effect or analytical error.

8.6 If the method blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

8.6.1 If the method blank concentration is greater than or equal to ½ the reporting limit AND is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement. **(DoD Requirements)**

9 Sample Collection, Preservation and Handling

9.1 Sample Collection:

9.1.1 Samples can be collected in any size plastic or glass bottles. Minimum amount required for analysis is 100 mL.

9.1.2 Aqueous samples requiring dissolved metals shall be filtered immediately on site before adding preservation for dissolved metals.

9.2 Sample Preservation:

9.2.1 Preserve aqueous samples using HNO_3 to a pH <2. Sample preservation shall be performed immediately upon sample collection. If this is not possible, then samples would be preserved as soon as possible when received at the laboratory. If samples are unpreserved in the laboratory, analysis shall not take place for 16 hours.

9.3 Sample Handling:

9.3.1 All samples must be analyzed within 28 days of collection. All samples not analyzed within this time frame must be discarded and resampled for analysis.

9.3.2 All samples require digestion. Refer to the Sample Preparation SOP for procedures.

9.3.3 All samples and sample digestates shall be stored at room temperature, separated from standards.

10 Procedure

10.1 For P S Analytical Merlin Millennium.

10.1.1 Verify the Argon pressure is set at 40 psi and inspect the tubing. If it has flattened change all four lines and clamp.

10.1.2 Open the Avalon Software and name the folder using the date on appropriate matrix: W for waters, S for soils and LL for low detection waters.

10.1.3 Under the OPTIONS heading select SERVICE OPTIONS and check the Disable dryer gas check and also Disable the analysis gas.

10.1.4 Under the METHODS heading select ALSIW and click OK, if analyzing the low detection samples set the range to 10. Verify that the lines are drawing liquid by using harmless food coloring and adjust the pump clamps to set the proper tension. Allow the instrument to warm up for at least 15 minutes.

10.1.5 Fill the Stannous Chloride reservoir with 10% Stannous Chloride and insert the probe into it. Allow this to flush the system for 10 minutes.

10.1.6 Under the OPTIONS heading select MANUAL CONTROL. Adjust the reference output between 107-110 using the screw on the instrument labeled R. Adjust the Trans Loss by setting the range to 10 and using the screw on the instrument labeled TL set this between 160-190.

10.1.7 Under the CALBRATION heading select NEW CURVE and set the standards and select analyze. To each standard add Hyrdroxylamine hydrochloride. Shake until color disappears and vent. Load into spaces 1 to 6 on the autosampler. In the instrument software set the concentrations that are being used, click OK and verify that the settings are correct and click OK.

10.1.8 After the calibration is complete and acceptable, (r=0.995 or greater) under the PROGRAM heading choose the program editor and OPEN the correct autosampler table.

10.1.9 To each sample add Hydroxylamine hydrochloride. Shake until color disappears and vent. Load the samples into the autosampler tray. To begin the analysis of the samples, under the PROGRAM heading choose RUN and click OK to begin the analysis.

10.1.10 After analysis is complete, any sample that has a result above the reporting limit (0.0005 mg/L for 245.1/7470; 0.001 mg/L for SPLP/7471; 0.006 mg/L for TCLP; 0.0002 mg/L for 245.1 Low and 0.00007 for 245.1 X-Low) must be rerun without stannous chloride to determine if an organic interference is

present.

10.1.10.1 If the stannous chloride result is greater than the reporting limit, subtract the non-stannous chloride result to get the final mercury concentration.

10.2 For the CETAC M-6100 Mercury Analyzer

10.2.1 Start the Quick Trace M-6100 software (if the software was left in standby, open the M-6100 controls and start the autosampler by selecting PARK - to start the rinse cycle.)

10.2.2 Turn on the lamp and start the gas flow (40 psi). A minimum of 15 minutes is needed.

10.2.3 Inspect the sample tubing and replace if needed clamp the lines. Verify that the capillary is 0.5 mm above the Gas/liquid separator center post.

10.2.4 Wet the Gas/liquid separator (GLS) center post. Disconnect the "11→Hg Vapor→12" from the GLS vapor outlet. Set the gas pressure to 60 psi. Place the reagent line into a beaker containing DI water. Turn on the pump and release the tension on the bottom two lines (drain lines). Allow the liquid level to rise so that the gas will bubble and wet the ENTIRE post of the GLS. Once this happens re-clamp the lines, set the gas pressure to 40 psi and allow the GLS to drain. When the GLS has drained reconnect the "11→Hg Vapor→12" and place the reagent line into 10% Stannous Chloride.

10.2.5 Open the proper worksheet and using SAVE AS name it using the date followed by W for waters; S for soils; LL for low detection

10.2.6 At this point, add Hydroxylamine hydrochloride to each standard shake until color disappears and vent. Load the standards and begin the calibration. Once the calibration is complete and acceptable, (r=0.995 or greater) sample analysis can begin. Decolorize the samples using Hydroxylamine hydrochloride shake until color disappears and vent. Load the samples into the autosampler tray for analysis

10.2.7 After analysis is complete, any sample that has a result above the reporting limit (0.0005 mg/L for 245.1/7470; 0.001 mg/L for SPLP/7471; 0.006 mg/L for TCLP; 0.0002 mg/L for 245.1 Low and 0.00007 for 245.1 X-Low) must be rerun without stannous chloride to determine if an organic interference is present.

10.2.7.1 If the stannous chloride result is greater than the reporting limit,

subtract the non-stannous chloride result to get the final mercury concentration.

11 Calculations

11.1 Samples results are documented directly form the readout of the instrument in ppb (from the calibration curve).

11.2 The results are converted to ppm and input into the LIMS system.

11.3 Samples requiring dilution at the time of analysis to bring the result into calibration range are multiplied by the dilution factor used before inputting into the LIM system using the following equation:

$$A = \frac{Z\,(B)}{C}$$

where: A= Concentration of mercury in the sample
B= Final volume of the dilution (mL)
Z= Concentration of mercury in the dilution
C= Volume of sample aliquot used in the dilution

11.4 **LCS Recovery**
% recovery = (Cm/Cn)x100
Cm = measured concentration of LCS
Cn = Spiking concentration

Spike Recovery
% recovery = [(Cs-Cu)/Cn]x100
Cs = measured concentration of spiked sample aliquot
Cu = measured concentration of unspiked sample aliquot
Cn = spiking concentration

Precision (RPD)

$$\% \text{ RPD} = \frac{|(R1-R2)|}{(R1+R2)/2} \times 100$$

R1 = sample or spike result
R2 = duplicate or spike duplicate result

12 Reporting Results

12.1 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

12.2 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.

12.3 Report the actual result, even if it is less than the reporting limit. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate detection limit.

12.4 Reporting Results (DEP Reportable Samples)

12.4.1 DEP samples exceeding the Maximum Contaminant Level (MCL) must be reported to the Customer Service Representative immediately following determination in order to comply with the Pennsylvania Code; Title 25, Chapter 109, Section 109.810 for Reporting and Notification Requirements.

12.5 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

12.6 The following must be done to upload data into the Horizon LIMS system. It is instrument specific.

12.6.1 For data from the CETAC M-6100:

Select the REPORTS icon from the instrument software and choose the file to be uploaded. Next select EXPORT FILE. This file is to be exported to the CETAC folder located on the Desktop. Name the file so that it corresponds to the actual run and save. It is now in an EXCEL spreadsheet and shall be in the following format so that uploading can occur *SAMPLE NUMBER*SAMPLE TYPE*BATCH NUMBER*ANALYST INITIALS*DILUTION* (This is only needed if it is something other than 1.) Once all samples have this format the following shall be done to complete the process. Open the MERCURY_CETAC file and click the button called 'import.csv file'. Select the file name to be uploaded from the CETAC DATA folder on the desktop. The reports will print to NuGenesis and can be posted in the Horizon LIMS.

12.6.2 For data from the P S Analytical:

Select the FILE option and select SAVE AS TEXT. Name the file so that it corresponds to the actual run .txt (example: results.txt). Under the drivers select e:\\wmmet001\psadata. Open the spreadsheet named PSA and click

'import .txt file' Select the file name to be uploaded from the PSA data folder on the Desktop. The data shall be in the following format so that uploading can occur *SAMPLE NUMBER*SAMPLE TYPE*BATCH NUMBER*ANALYST INITALS*DILUTION* (This is only needed if it is something other than 1) Once all the samples have this format the following shall be done to complete the process. Click the 'send to NuGenesis' button. The reports will print to NuGenesis and can be posted in the Horizon LIMS.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

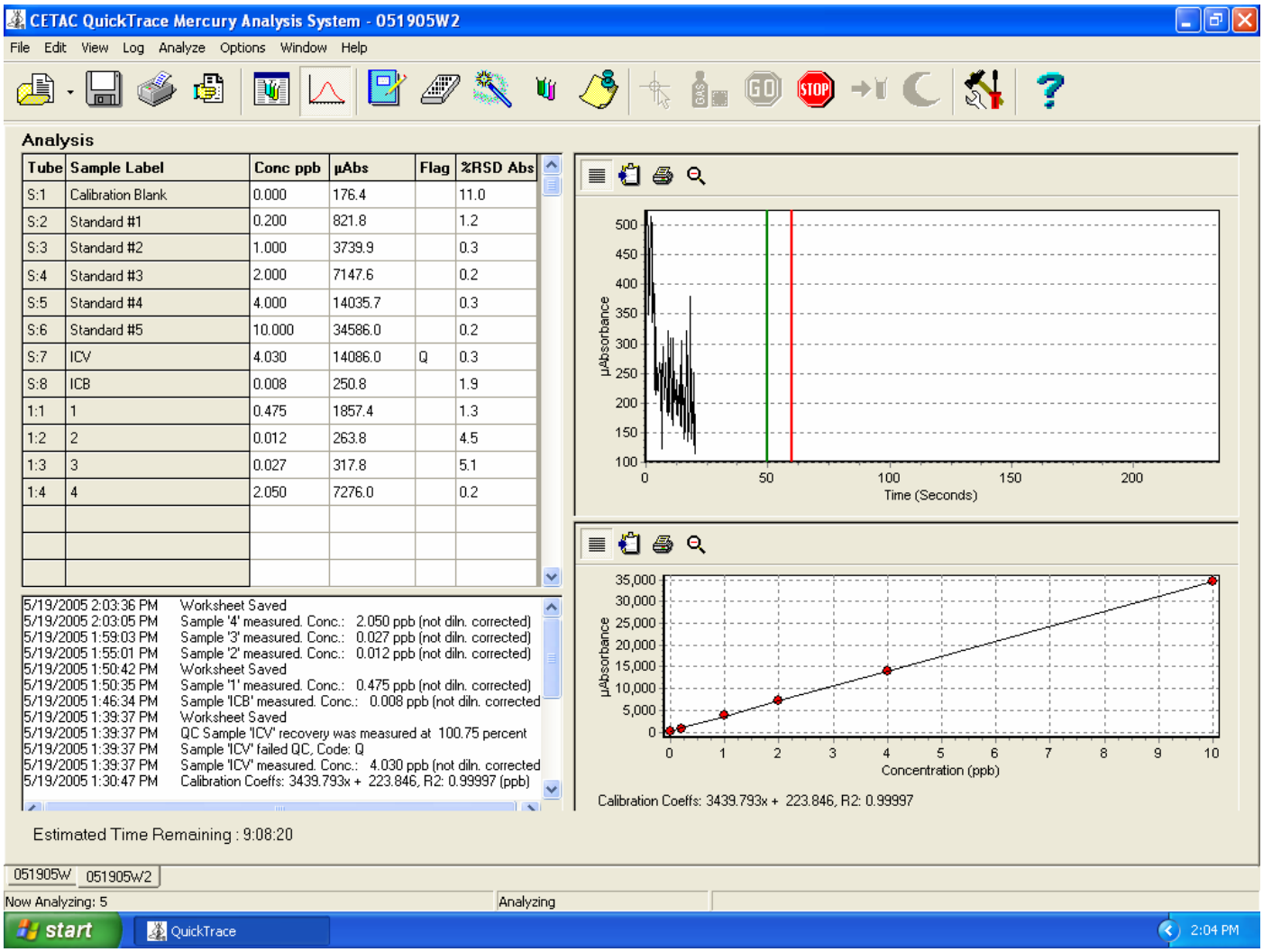
15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

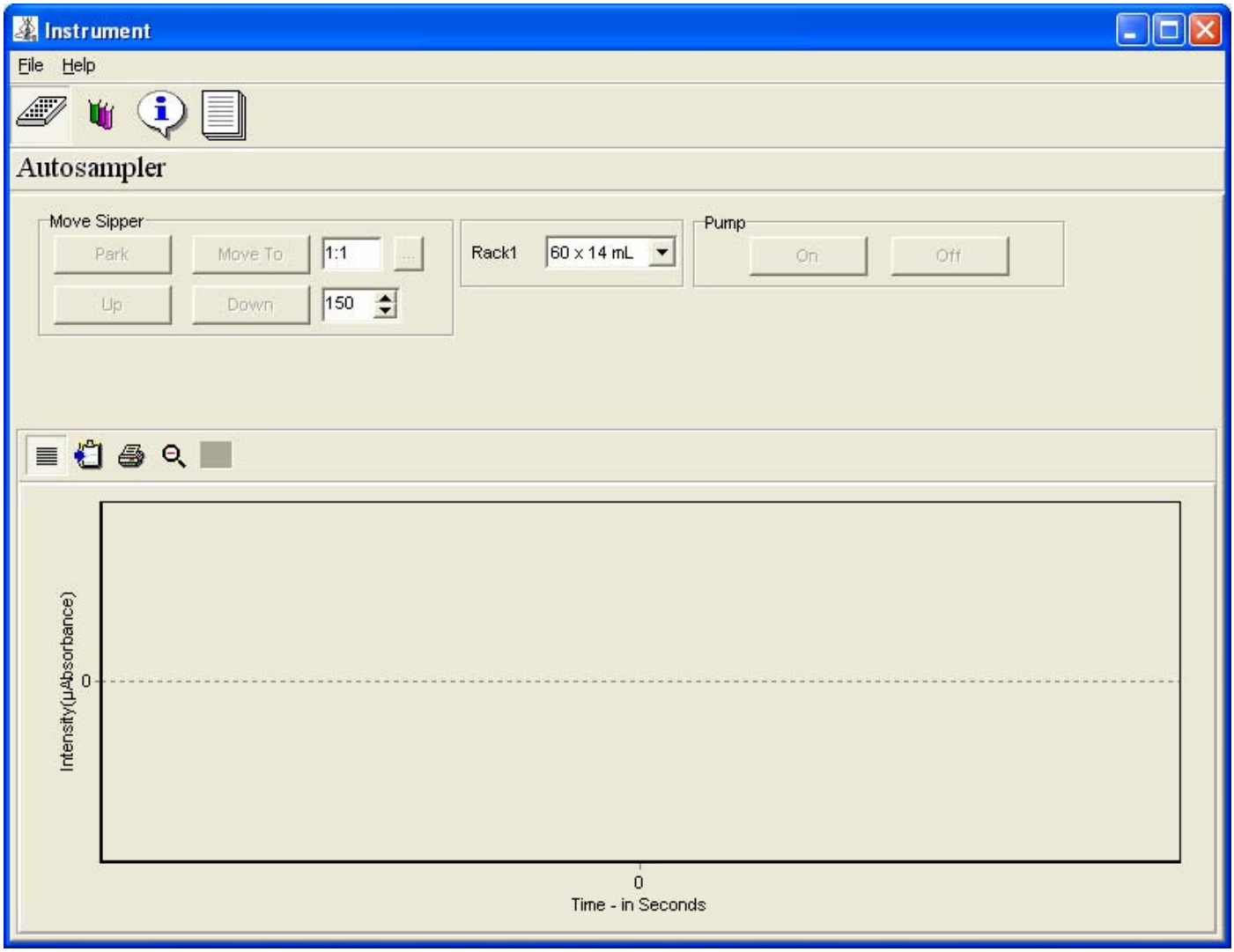
16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

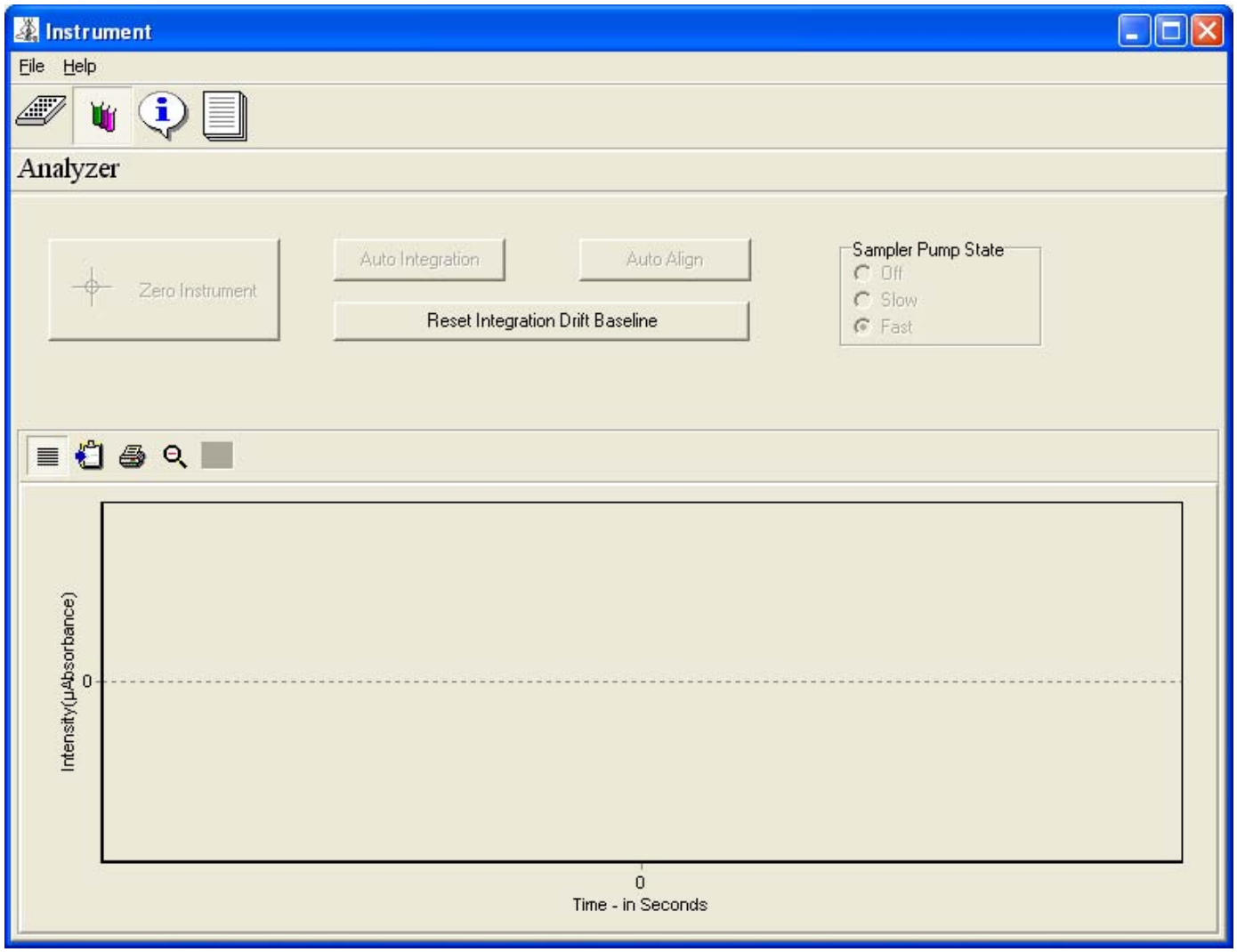
Appendix A – CETAC



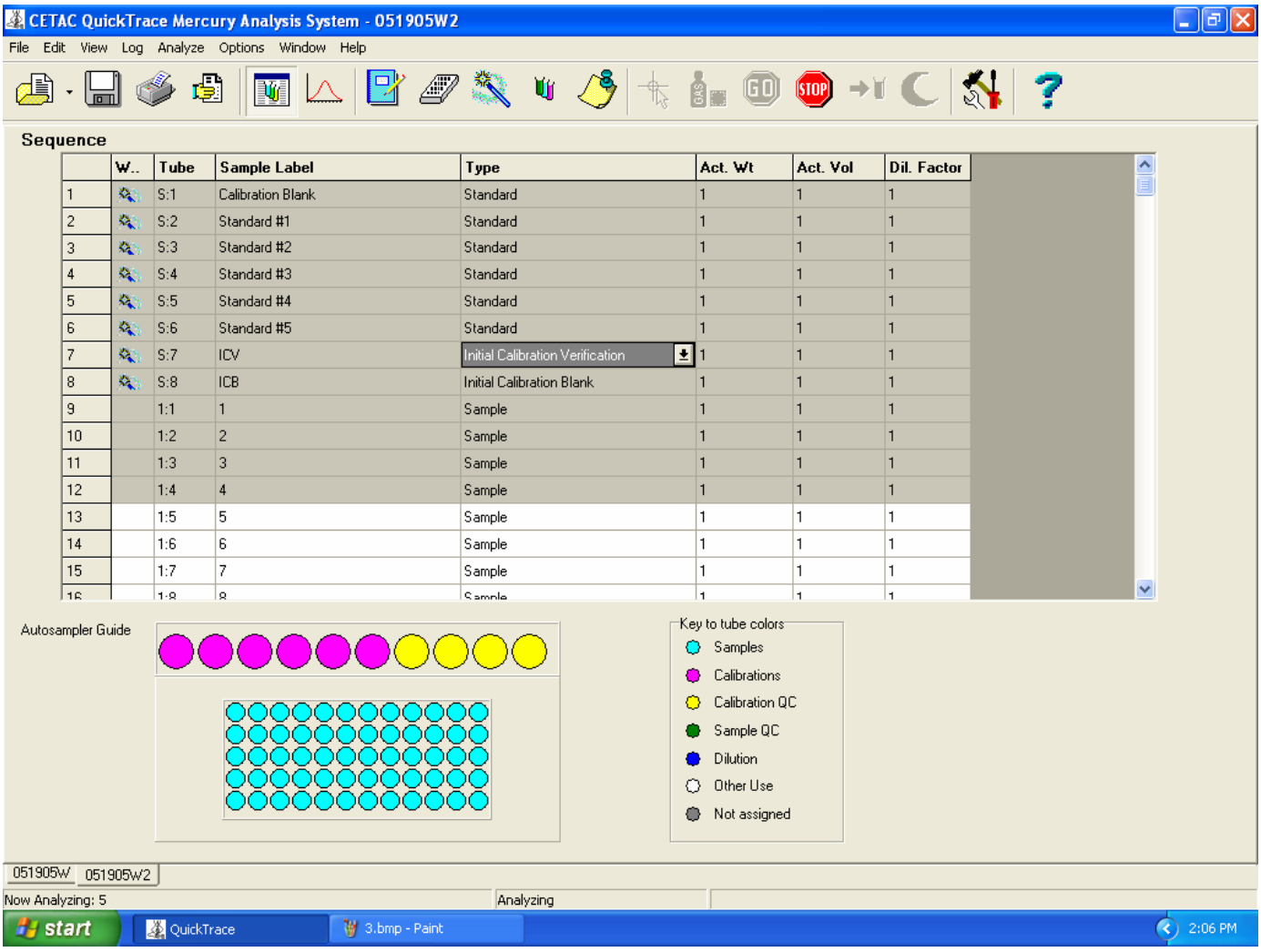
Appendix A - CETAC



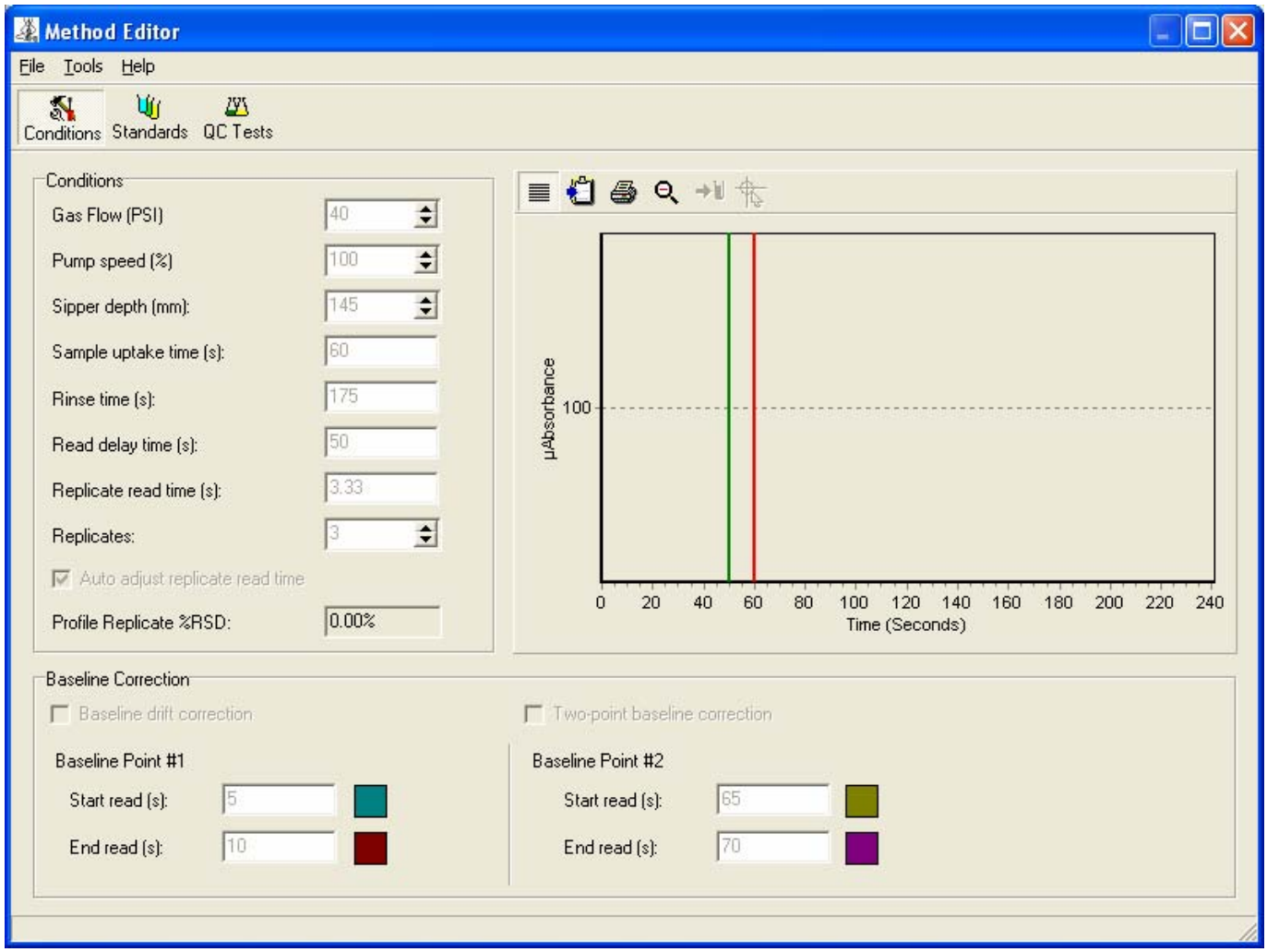
Appendix A - CETAC



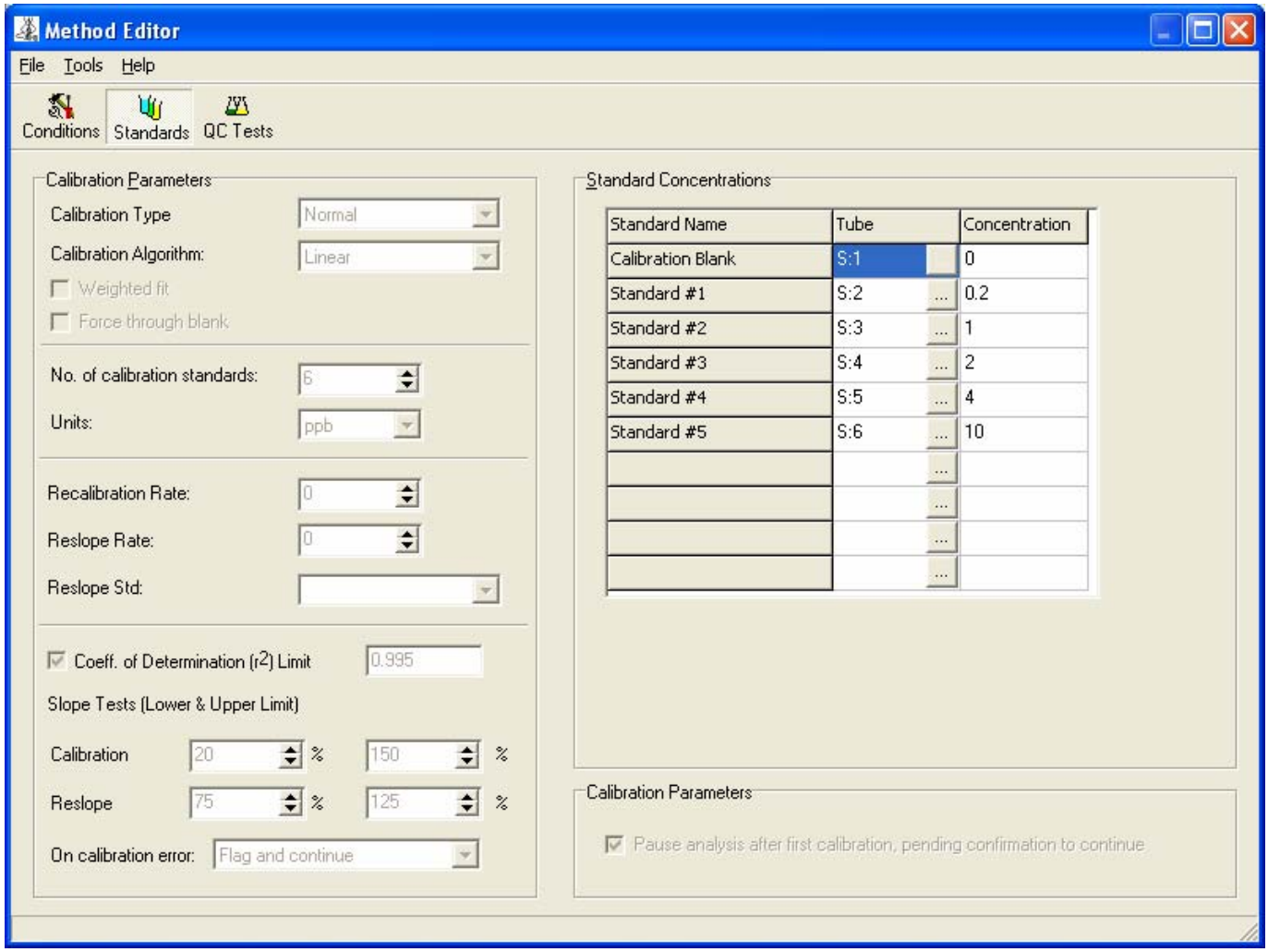
Appendix A - CETAC



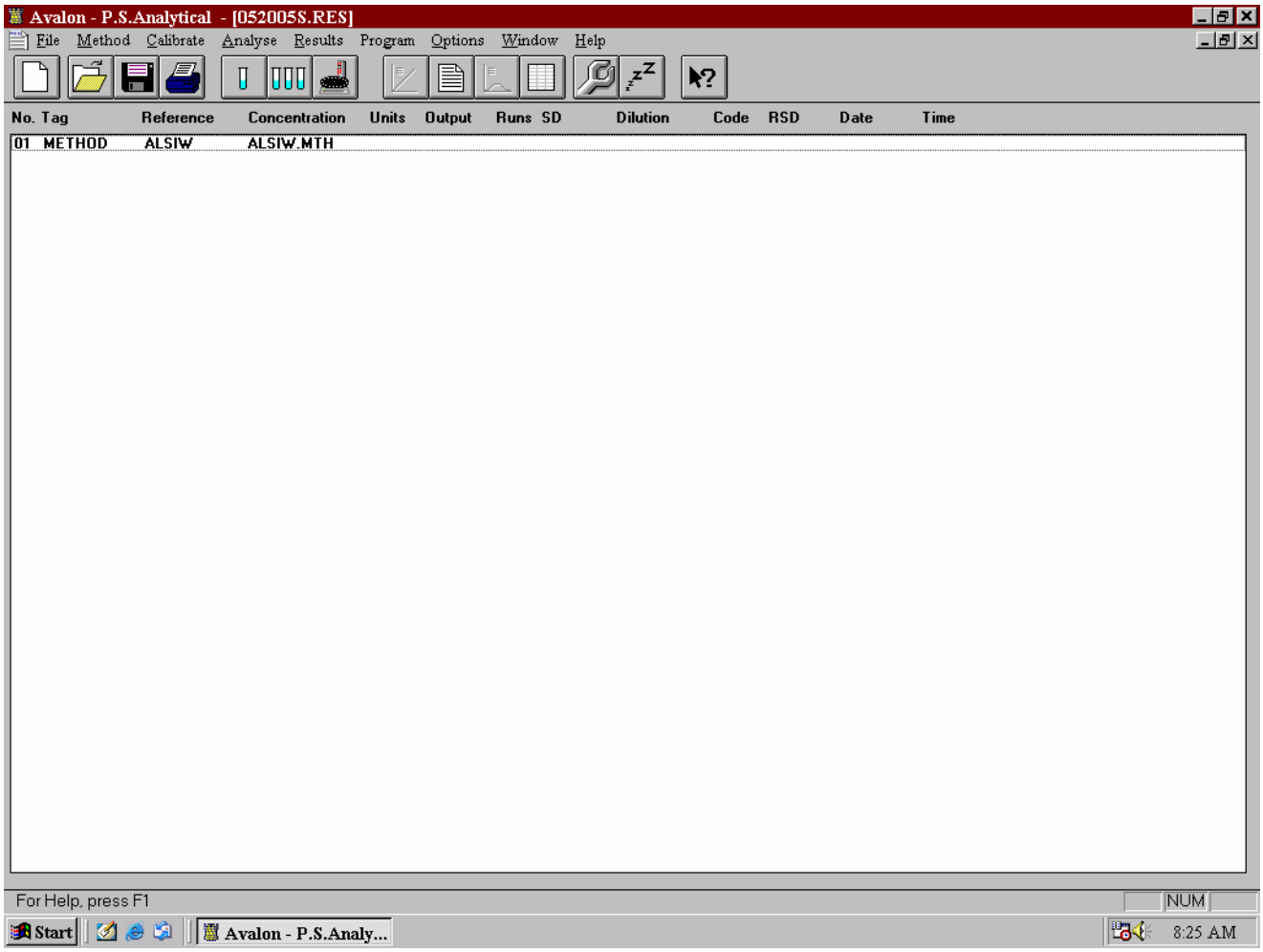
Appendix A - CETAC



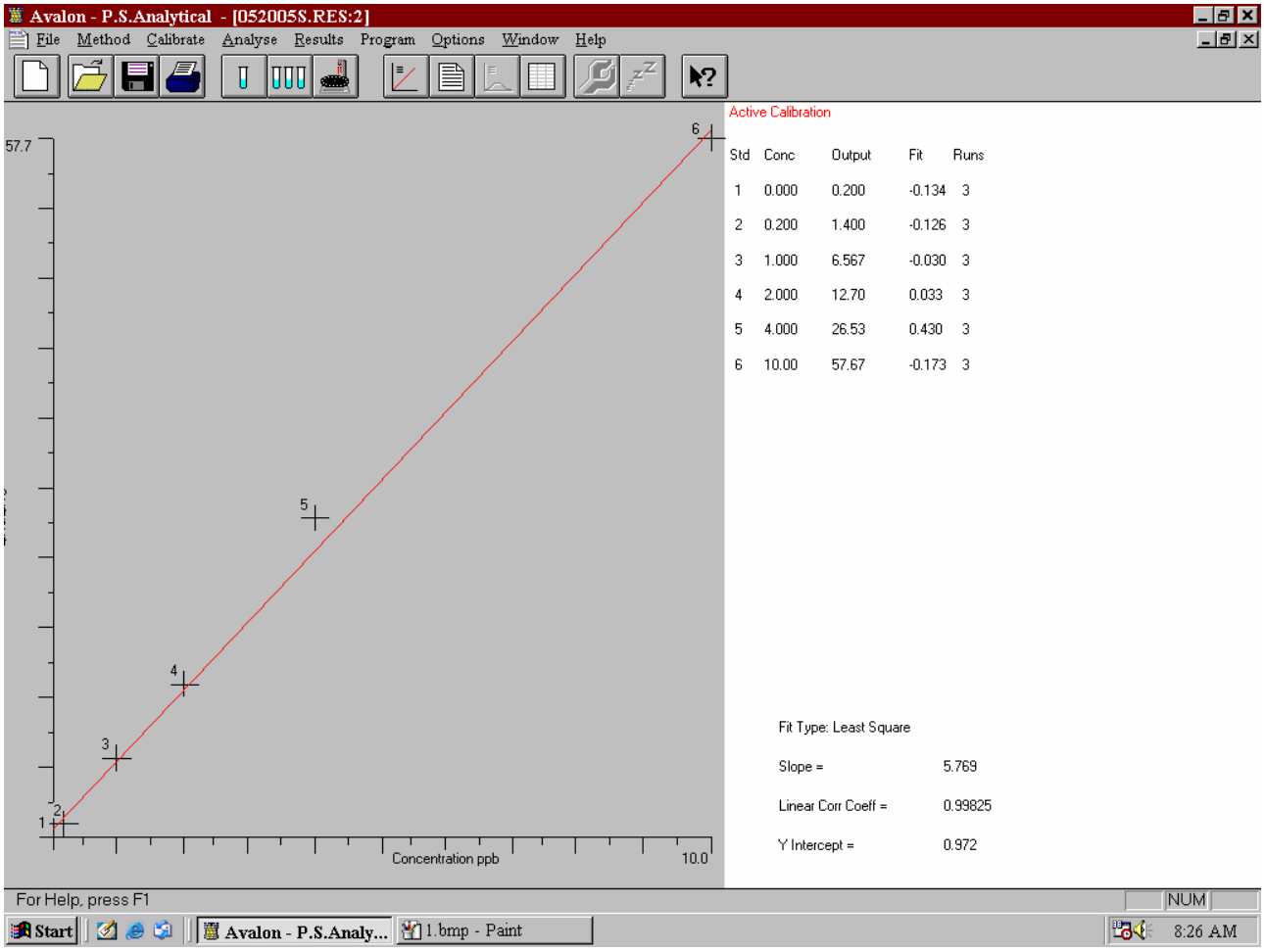
Appendix A - CETAC



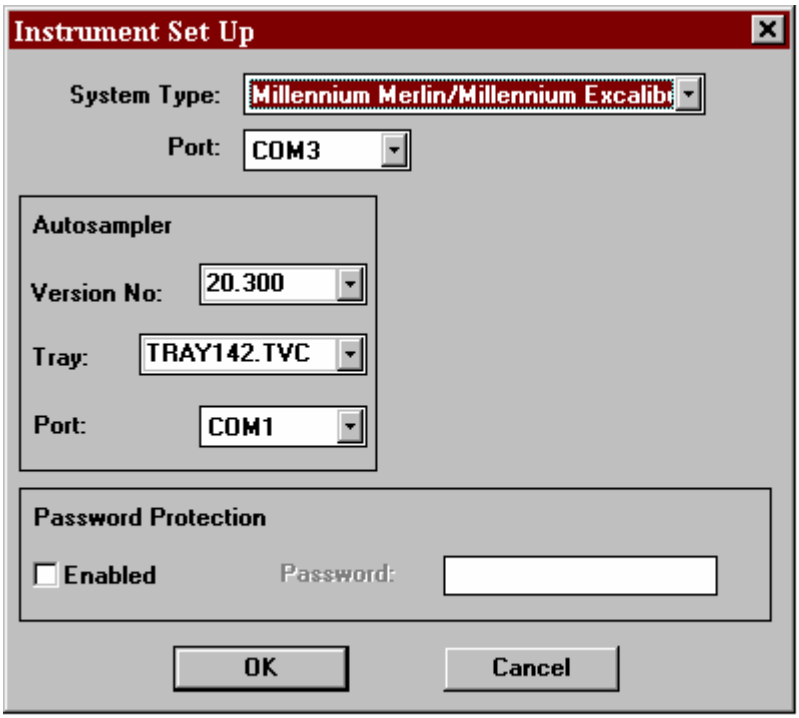
Appendix B – PSA



Appendix B – PSA



Appendix B – PSA



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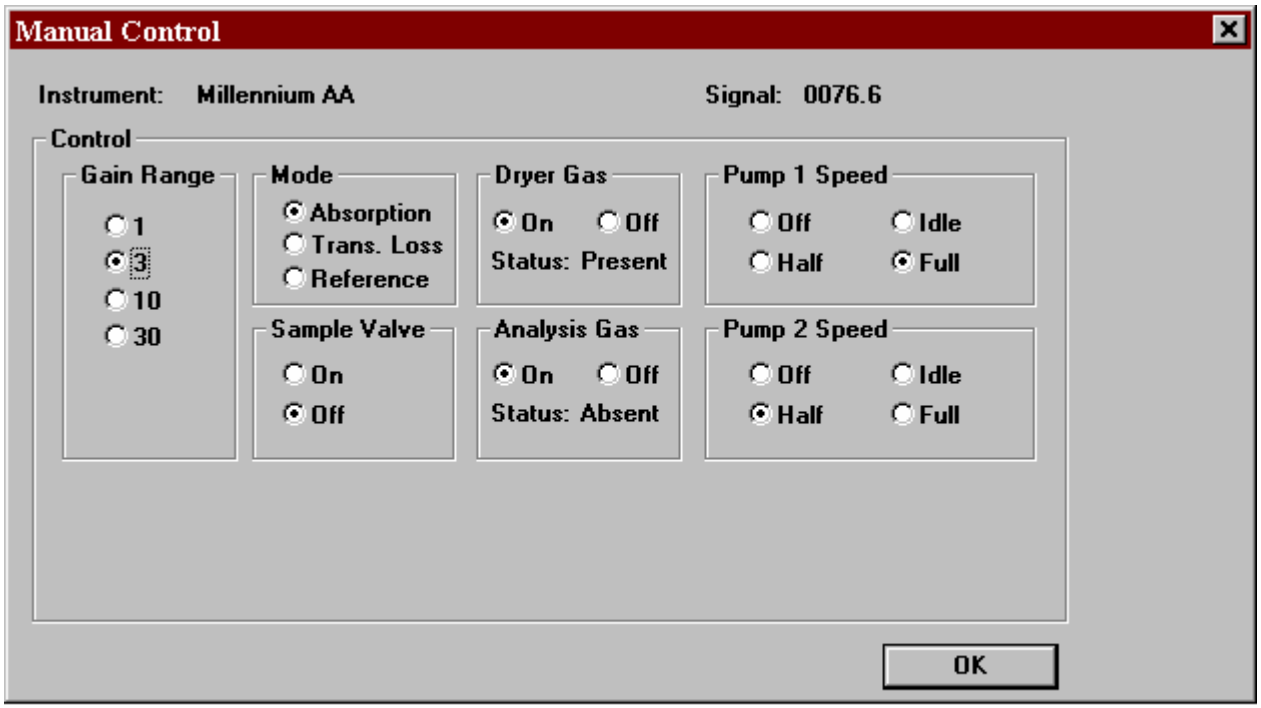
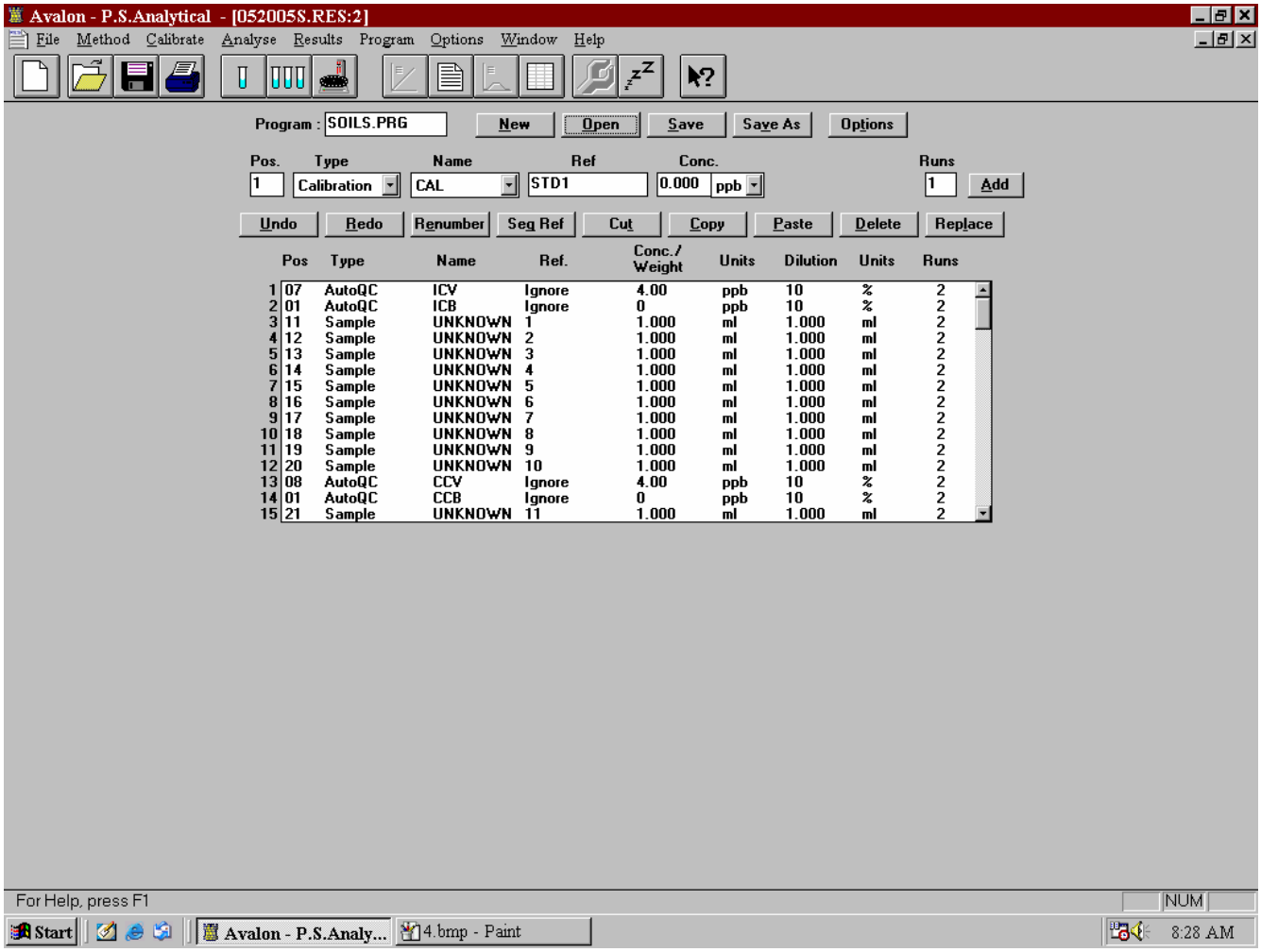


Appendix B – PSA



Appendix C

CETAC HG WORKSHEET

File:_____
Matrix:_____
Analyst:_____
Date:_____

$SnCl_2$ solution: _____g $SnCl_2$ in _____of 1.4% H_2SO_4
NH2OH / HCl log#: _____ Pipette ID#_____
Post Spike: 50 ul_____ into 10 ml of sample.

Sample	Sample
1	37
2	38
3	39
4	40
5	41
6	42
7	43
8	44
9	45
10	46
11	47
12	48
13	49
14	50
15	51
16	52
17	53
18	54
19	55
20	56
21	57
22	58
23	59
24	60
25	61
26	62
27	63
28	64
29	65
30	66
31	67
32	68
33	69
34	70
35	71
36	72

Appendix D

PSA HG WORKSHEET

File:_____
Matrix:_____
Analyst:_____
Date:_____

$SnCl_2$ solution: _____g $SnCl_2$ in _____of 1.4% H_2SO_4
NH2OH / HCl log#: _____ Pipette ID#_____
Post Spike: 50 ul_____ into 10 ml of sample.

Sample	Sample
1	37
2	38
3	39
4	40
5	41
6	42
7	43
8	44
9	45
10	46
11	47
12	48
13	49
14	50
15	51
16	52
17	53
18	54
19	55
20	56
21	57
22	58
23	59
24	60
25	61
26	62
27	63
28	64
29	65
30	66
31	67
32	68
33	69
34	70
35	71
36	72

SOP Change History Sheet

Section No.	Section	Reason for Change
1	Scope and Application	SOP update
8.2	Quality Control	PADEP Audit response
8	Quality Control Requirements	USACE audit response
12	Reporting Results	Updated for new LIMS
5	Apparatus and Materials	A2LA Audit Response
		SOP update 5/3/05
7	Instrument Calibration	SOP update to calibration
		levels 5/03/05
10	Procedure	SOP update 5/3/05
	Appendix A	Inserted New Instrument
		Set-up Records for CETAC 5/19/05
	Appendix B	Inserted New Instrument
		Set-up Records for PSA 5/19/05
	Appendix C	Insert New Instrument
		Logbook for CETAC
		5/19/05
	Appendix D	Insert New Instrument
		Logbook for PSA 5/19/05

Revision 23: 03/23/2006

1.2	Scope and Application	DoD audit response, updated methods
1.8	Scope and Application	Added project requirement verbiage
6.3	Reagents	Updated vendor information

SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
6.6	Reagents	Added decolorizing agent volume as per PADEP audit response
7.3.1	Instrument Calibration	Updated reference method and standards reference
7.6	Instrument Calibration	Added corrective actions for calibration blank concentrations as per PADEP/DoD audit responses
8.2	Quality Control	Added ongoing proficiency verbiage
8.2	Quality Control	Updated table to reflect method addition/revision and concentration revisions as per DoD audit response
8.3-8.5	Quality Control	Major revisions/additions throughout
10.1.7, 10.1.9, 10.2.6	Procedure	Added verbiage about Hydroxylamine addition as per PADEP audit response
10.1.8	Procedure	Added r-value
11.4	Calculations	Added specific calculations as per DoD audit response
12	Reporting Results	Major revisions/additions throughout

SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
16	Troubleshooting	Added section as per DoD audit response

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

Document Title: Determination of Perchlorate by Ion Chromatography

Document Control Number:_____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Susan Magness, Date
Validator

TABLE OF CONTENTS

1 Scope and Application 3
2 Summary of Method 3
3 Interferences 3
4 Safety 4
5 Apparatus and Materials 4
6 Reagents 5
7 Sample Collection, Preservation and Handling 8
8 Initial Demonstration of Capability 8
9 Quality Control 10
10 Instrument Calibration 11
11 Procedure 12
12 Data Review 15
13 Calculations 16
14 Reporting Results 16
15 References 17
16 Waste Disposal 17
17 Pollution Prevention 17
18 Definitions 17
Appendix A 18
SOP Change Summary 19
SOP Concurrence Form 20

1 Scope and Application

1.1 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.2 This method is adapted from EPA Method 314.0, Determination of Perchlorate in Drinking Water by Ion Chromatography, Revision 1.0, November 1999.

1.3 This method is restricted for use by or under the supervision of analysts trained on the use of the ion chromatograph.

1.4 This method covers the determination of perchlorate. The applicable matrices are reagent water, finished drinking water, surface water, and ground water.

2 Summary of Method

2.1 A small volume of sample, typically 1.0 mL, is introduced into an ion chromatograph. Perchlorate is separated and measured, using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector.

3 Interferences

3.1 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline noise.

3.2 Interferences can be caused by substances with retention times that are similar to and/or overlap with the retention time of perchlorate. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

3.3 Sample matrices with high concentrations of common anions such as chloride, sulfate and carbonate can make the analysis problematic by destabilizing the baseline on the retention time window for perchlorate. These common anion levels can be indirectly assessed by monitoring the conductivity of the matrix. All sample matrices must be monitored for conductivity prior to analysis. When the laboratory determined Matrix Conductivity Threshold (MCT) is exceeded sample dilution must be performed.

3.4 All sample and any reagent solutions that contain particles larger than 0.20 microns require glass membrane filtration to prevent damage to instrument columns and flow systems. (An in-line filter removes any particulates present in reagents. Sample caps with filters remove fine particulates in samples. Samples with a large amount of particulate matter should initially be filtered through an 0.45 micron filter to avoid clogging the sample cap filter. Nylon fiber filters should not be used because they alter the perchlorate concentration.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

4.2 Each analyst should become familiar with the reagents used by reference the Material Safety Data Sheets (MSDS) for each reagent. In doing so, the analyst will become familiar with the appropriate precautions for each reagent.

4.3 The laboratory also operates under a formal safety plan.

4.4 Analysts must wear a buttoned lab coat and safety glasses at all times during the analysis. PVC gloves should be worn when handling samples and reagents.

5 Apparatus and Materials

5.1 Ion Chromatograph- Dionex DX-120 including:

5.1.1 Anion Guard column (Dionex AG16, 4mm, P/N 55377), or equivalent.

5.1.2 Anion Separator column (Dionex AS16, 4mm, P/N 55376), or equivalent.

5.1.3 Anion Suppressor (Dionex Anion Self Regenerating Suppressor (4 mm ASRS ULTRA, P/N 53946) used in external water mode, or equivalent.

5.1.4 Detector (Dionex CD20), or equivalent.

5.1.5 Autosampler (Dionex AS40), or equivalent.

5.1.6 Standard Conditions:

5.1.6.1 Eluent Flow = 1.5 ml/min

5.1.6.2 Pressure = approximately 2800 psi

5.1.6.3 Suppressor current = 300mA

5.1.6.4 Background Conductivity = 2-4 Us

5.1.6.5 Run Time = 12 minutes

5.1.6.6 External Water Flow (with 300 mA current on) = 3-5 ml/min

5.2 The Dionex PeakNet Chromatography Software

5.3 Conductivity Meter – purchased from YSI Instruments, Model 3100, or equivalent.

5.4 Analytical Balance - capable of accurately weighing to the nearest 0.0001 gram. Mettler AE100 is currently in use.

5.5 Class A volumetric flasks – various sizes, purchased from VWR Scientific.

5.6 Weigh boats – purchased from VWR catalog no. 12577-051, or equivalent.

5.7 Glass fiber filters- 0.45 micron - purchased from VWR, or equivalent.

5.8 Eppendorf pipettor (20-200 ml) - purchased from VWR catalog 53513-408, or equivalent.

5.9 Eppendorf pipettor (100 – 1000ml) – purchased from VWR catalog no. 53513-582, or equivalent.

5.10 5 ml autosampler vials – purchased from Dionex catalog no. 038008, or equivalent.

5.11 Filter caps for 5.0 ml vials – purchased from Dionex catalog no. 038009, or equivalent.

6 Reagents

6.1 Reagent water: Distilled or deionized water, free of anions. Water should contain particles no larger than 0.20 microns. For this purpose, ALSI uses a deionizer that provides analyte-free, greater than 16 megohm-cm, DI water on demand.

6.2 Sodium Hydroxide (NaOH) - purchased in pellet form from VWR catalog no. JT3722-7. Store at room temperature for a maximum of 5 years.

6.3 Eluent Concentrate: 10 N Sodium Hydroxide – Dissolve 800 grams of sodium hydroxide in 2 liters of deionized water. Store at room temperature for a maximum of 6 months.

6.4 Working Eluent Solution: 50mM Sodium Hydroxide. Put a spin bar in approximately 2 liters of reagent water and place on a stir plate. Degas the reagent water by pulling a vacuum on the stirring solution for 20 to 30 minutes. Pipette 10 ml of 10N Sodium Hydroxide Eluent Concentrate into a 2-liter volumetric flask. Dilute to volume with the degassed reagent water. This solution expires after 5 days at room temperature.

6.4.1 Eluent should be purged for 10 minutes with helium prior to use (pressurizing the eluent reservoir with helium will give the same effect). When refilling the eluent reservoir, completely replace the old eluent by emptying the old eluent, rinsing the reservoir with reagent water and refilling with the new eluent.

Note: Solutions of NaOH are very susceptible to carbonate contamination resulting from adsorption of carbon dioxide from the atmosphere. This contamination will result in poor reproducibility of perchlorate retention times, elevated background conductivity, and increase in baseline noise/drift. Consequently, exposure to the atmosphere should be minimized.

6.5 Sodium Perchlorate – ACS reagent grade. Purchase Aldrich 41,024-1 (100gm) or equivalent. Dry chemical in desicator 24 hours prior to use.

6.6 Stock Perchlorate Solution, 1000 mg/l: The stock perchlorate solution is prepared from ACS reagent grade materials. Dissolve 1.231 g sodium perchlorate in reagent grade water and dilute to volume in a 1 liter flask. This solution is stable at room temperature for 12 months.

6.7 Intermediate Perchlorate Solution, 10,000µg/L: The intermediate perchlorate solution is prepared from the 1000mg/L stock perchlorate solution (6.6). Dilute 1.0 mL of the stock perchlorate solution to volume in a 100 mL Class A volumetric flask. This solution is stable for one month at room temperature.

6.8 Working Perchlorate Solution, 1,000µg/L: The working perchlorate solution is prepared from the 1000mg/L stock perchlorate solution (6.6). Dilute 1.0 mL of the stock perchlorate solution to volume in a 1000 mL Class A volumetric flask. This solution is stable for one month at room temperature.

6.9 Calibration Curve Standards: Use the intermediate perchlorate solution (6.7) to prepare serial dilutions in 100 ml Class A volumetric flasks at the following specifications: These solutions are stable at room temperature for a period of 7 days.

Calibration Level	Amount Added (mL)	Final Volume (mL)	Concentration (ug/L)
1	0.040	100	4
2	0.100	100	10

3	0.150	100	15
4	0.200	100	20
5	0.250	100	25
6	0.500	100	50
7	0.750	100	75
8	1.00	100	100

6.10 Initial Calibration Check Solution (ICCS), 4ug/l: Using the intermediate perchlorate solution (6.7) dilute 0.040 ml to a volume of 100 ml in a Class A volumetric flask using reagent water. This solution is stable at room temperature for 7 days.

6.11 Mid Level Continuing Calibration Check Solution (CCCS), 25 ug/l: Using the intermediate perchlorate solution (6.7) dilute 0.250 ml to a volume of 100 ml in a Class A volumetric flask using reagent water. This solution is stable at room temperature for 7 days.

6.12 High Level Continuing Calibration Check Solution (CCCS), 75 ug/l: Using the intermediate perchlorate solution (6.7) dilute 0.750 ml to a volume of 100 ml in a Class A volumetric flask using reagent water. This solution is stable at room temperature for 7 days.

6.13 Laboratory Fortified Blank (LFB): Dilute 0.125 ml of working perchlorate solution (6.8) into 5 ml of reagent water for a LFB of 25 ug/l. Filter using a 0.45 um filter.

6.14 Sample Matrix Spike: Pipet 0.125 ml of working perchlorate solution (6.8) into 5 ml of sample for a 25 ug/l spike.

6.15 Second Source Perchlorate Stock Solution, 1000 mg/l: Using an ACS grade chemical of a different lot number than the stock perchlorate solution, dissolve 1.231 g sodium perchlorate in reagent water and dilute to 1000 ml in a Class A volumetric. Solution is stable at room temperature for 12 months.

6.16 Second Source Intermediate Solution, 1000 µg/l: Dilute 1.0 ml of the second source perchlorate stock solution (6.15) to 100 ml in a Class A volumetric flask with reagent water. This solution is stable for one month at room temperature.

6.17 Laboratory Control Standard (LCS), 50 µg/l: Dilute 0.50 ml of the second source intermediate solution (6.16) to 100 ml in a Class A volumetric flask with reagent water. This solution is stable for seven days at room temperature.

6.18 Mixed Common Anion Solution (25 mg/ml each of chloride, sulfate, and carbonate): Dissolved 1.0 g sodium chloride (purchased from Aldrich catalog no. 20,443-9, or equivalent), 0.93 g sodium sulfate (purchased from VWR catalog no. EM-SX0760E-1,

or equivalent), and 1.1 g sodium carbonate (purchased from VWR catalog no. JT3604-01, or equivalent) in reagent grade water to a final volume of 25 ml. Solution is stable at room temperature for one month.

6.19 Instrument Performance Check (IPC): Dilute 0.80 ml mixed common anion solution (6.18) and 1.250 ml working perchlorate solution (6.8) to 50 ml in a Class A volumetric flask with reagent water. This solution is stable for seven days at room temperature.

7 Sample Collection, Preservation and Handling

7.1 Samples should be collected in plastic or glass bottles. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal. A minimum of 100 ml is required.

7.2 Samples for perchlorate have a 28-day holding time and do not need to be preserved. Samples do not need to be shipped on ice or stored cold but every effort should be taken to protect samples from temperature extremes.

8 Initial and Continuing Demonstration of Capability

8.1 Initial Demonstration of Accuracy (IDA) – Prepare and analyze seven replicate LFBs at 25.0 µg/l (6.13). Calculate the average measured concentration of the replicate values. To pass the IDA, the average concentration must be within 10% of the true value or between 22.5 µg/l and 27.5 µg/l. Independent analysis of samples should not begin until an acceptable IDA is achieved.

8.2 Initial Demonstration of Precision (IDP) – Using the data generated for Section 8.1, calculate the percent relative standard deviation (%RSD) as follows:

$$\%RSD = \frac{(Sn-1)}{Ave.Conc.} \times 100$$

where, Sn-1 = sample standard deviation (n-1) of the replicate analysis

To pass the IDP, the %RSD must be less than 10%. Independent analysis of samples should not begin until an acceptable IDP is achieved.

8.3 Method Detection Limit (MDL) – MDLs should be determined every 12 months, when a new operator begins work or whenever there is a significant change in the background or instrument response. MDLs must be established using reagent water fortified at a concentration three to five times the established instrument detection limits. Refer to previous MDL studies for suggested concentrations to be used. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through

the entire analytical method over a three day period. Calculate the MDL as follows:

$$MDL = (t) \times (Sn - 1)$$

where t=3.14 for seven replicates and Sn-1 = sample standard deviation (n-1) of the seven replicate analyses.

8.4 Matrix Conductivity Threshold (MCT)

8.4.1 Prepare a laboratory fortified blank at 25 µg/l (6.13).

8.4.2 Prepare a series of common anion fortified reagent water samples by adding 0.20 ml (200 mg/l), 0.30 ml (300mg/l), 0.40 ml (400 mg/l), 0.50 ml (500 mg/l), 0.60 ml (600 mg/l), 0.80 ml (800 mg/l) and 1.00 ml (1000 mg/l) of the mixed common anion stock solution (6.18) into separate 25 ml volumetric flasks. Next, add 0.625 ml of the working perchlorate solution (6.8) to each volumetric flask and dilute to volume with reagent water for a final perchlorate concentration of 25.0 µg/l.

8.4.3 Measure the conductivity of each solution on a calibrated conductivity meter. Refer to SOP 04-SPC.

8.4.4 Analyze each solution and record the peak area to height (A/H) ratio and the quantified concentration of perchlorate using the MCT spreadsheet in Excel.

8.4.5 Calculate the A/H ratio percent difference ($PD_{A/H}$) between the average A/H ratio for the LFB (A/H_{LFB}) and the average A/H ratios for each mixed common anion solution (A/H_{MA}) using the following equation:

$$PD_{A/H} = \frac{ABS\,(A/H_{LFB} - A/H_{MA})}{A/H_{LFB}} \times 100$$

(see excel spreadsheet for assisted calculations)

8.4.6 As the conductivity of the matrices increase, the $PD_{A/H}$ will increase. The MCT is the matrix conductance where the $PD_{A/H}$ exceeds 20%. Therefore, the MCT is the conductance level of the highest mixed anion solution which yielded a $PD_{A/H}$ value below the 20% threshold.

8.4.7 Prior to sample analysis, the conductivity of each sample must be determined. When the conductance is above the MCT, sample dilution must be performed.

8.5 Continuing Demonstration of Capability (CDC) – Evaluate the analysis of four (4) consecutive Laboratory Fortified Blanks (6.13) on an annual basis. Calculate the

average measured concentration of the replicate values. To pass the CDA, the average concentration must be within 10% of the true value or between 22.5 ug/L and 27.5 ug/L. Documentation of the CDC shall be included in the employee's training records. Independent analysis of samples should not continue if an acceptable CDA is not on file.

8.6 Continuing Demonstration of Precision (CDP) – Using the data generated for Section 8.5, calculate the percent relative standard deviation (%RSD) as shown in Section 8.2. To pass the CDP the %RSD must be less than 10%. Documentation of the CDP shall be included in the employee's training records. Independent analysis of samples should not continue if an acceptable CDP is not on file.

9 Quality Control

9.1 All policies and procedures in the most current revisions of the ALSI QA Plan shall be followed when performing this procedure.

9.2 Quality Control Requirements

Quality Control Requirements

Parameter	Criteria	Frequency	Control Limits	Corrective Action
Instrument Performance Check (IPC) (6.19)	1.Compare Conductivity to that of MCT --- 2. Compare A/H to A/H of LFB in previous batch --- 3. HClO4 = 25 ug/l --- 4. Compare Retention Time to Window Study	Beginning of each batch	1. Conductivity 90-110 % of MCT --- 2. Percent Difference of A/H < 25% --- 3. Recovery must be 80-120% --- 4. Shift > 5% may indicate an instrument problem	1. Prepare fresh IPC solution --- 2., 3., 4. Rerun once. Do not proceed with the analysis of samples until an acceptable result has been obtained. If necessary perform a new MCT study.
Laboratory Reagent Blank (LRB)	NA	Beginning of batch, every 10 samples, end of batch	< ½ Reporting Limit	Rerun once. If still unacceptable identify and correct the source of the problem. Rerun the blank and rerun all detectable samples since the last acceptable blank.
Initial Calibration Check (ICCS)	HClO4= 4 ug/l	Beginning of each batch	Recovery of 75-125 %	Do not proceed with sample analysis until an

(6.10)			If DoD QSM samples are included 85-115%	acceptable result is obtained. If necessary recalibrate system.
Laboratory Fortified Blank (LFB) (6.13)	HClO4= 25 ug/l	Beginning of each batch	Recovery of 85-115 %	Do not proceed with sample analysis until an acceptable result is obtained. If necessary recalibrate system.
Continuing Calibration Check Standards (CCCS) (6.11 and 6.12)	Alternate HClO4= 25 ug/l and 75 ug/l	Every 10 samples and end of batch	Recovery of 85-115 %	Reanalyze all samples analyzed after the last acceptable CCCS.
Laboratory Control Standard (LCS) (6.17)	HClO4= 50 ug/l	Daily at the beginning of the run	Recovery of 90-110 %	Do not proceed with sample analysis until an acceptable result is obtained. If necessary recalibrate system.
Matrix Spike (MS)* (6.14)	HClO4= 25 ug/l	Once per batch of 20	Recovery of 80-120 %	If LFB is acceptable report sample with a qualifying statement.
Matrix Spike Duplicate (MSD)* (6.14)	HClO4= 25 ug/l	Once per batch of 20	1. Recovery of 80-120 % 2. MS/MSD RPD<15%	If LFB is acceptable report sample with a qualifying statement.

* Samples selected for matrix spike and matrix spike duplicate analyses shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike or duplicate may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

10 Instrument Calibration

10.1 Prepare a series of eight (8) standards (section 6.9). All standard dilutions must be recorded in the standards logbook located in the wet chemistry area on the bookshelf with the other laboratory notebooks.

10.2 In the Peaknet methods, open the previous Method P and “save as” renaming the method with the current date (Method P DD/MM/YY). In the Peaknet schedule, identify these standards as Level 1 through Level 8 calibration standards.

10.3 After calibration standards have been analyzed, check that the acceptance criteria are met. Under method file select linear curve type. Linearity must be ≥ 0.995, include

origin. Linearity must be achieved without deleting any middle calibration points.

10.4 After the calibration has been established, it must be verified by the analysis of a Second Source Standard. (Section 6.17). If measurements exceed +/- 10% of the true value, the problem must be investigated and corrected prior to the analysis of any samples.

10.5 A new calibration should be performed every 3 months or when calibration verification standards fail to meet acceptance criteria.

11 Procedure

11.1 Starting the DX-120 Ion Chromatograph #2

11.1.1 Configure instrument to run the perchlorate method- ensure that all lines are properly fitted and suppressor is set in external water mode. Fill external reservoir with DI water and purge with helium gas for 10 minutes. Pressurize DI reservoir with helium by adjusting in-line regulator. Fill the eluent reservoir with eluent (6.4).

11.1.2 From the Windows desktop, click on the Dionex icon.

11.1.3 From the PeakNet main Menu, click on Run and on IC #2 to highlight system #2.

11.1.4 Click the “Load Method” button

11.1.5 Double click on “METHOD P”, then OK

11.1.6 The lights on the DX-120 instrument #2 should light up for The Eluent Pressure, Pump and SRS.

11.1.7 Click on the “Baseline” button and wait for the baseline to stabilize. The pressure should come up to about 2500 psi or more. The flow should read approximately 1.50 ± 0.02 ml/minute.

11.1.8 Record the pressure, flow, total conductivity and offset conductivity in the DX-120 #2 maintenance notebook. Also complete maintenance checklist daily (located in the beginning of the same book).

11.2 Edit the Sample Analysis Schedule

11.2.1 From the Dionex Main Menu, click on Schedule

11.2.2 The autosampler will hold positions for 66 analyses. An example of the sequence of samples and QC checks, is as follows:

1) IPC
2) LRB
3) ICCS
4) LFB
5) LCS
6) CCCS 25PPB
7-16) Samples
17) CCV Level 25 PPB
18) Blank
19-28) Samples
29) MS
30) MSD
31) CCV Level 75 PPB
32) Blank

NOTE: The maximum number of samples permitted in a sample batch is 20. This does not include standards or spikes but does include dilutions.

11.2.3 Under Sample, enter the sample number or the type of QC check. For samples that are diluted, follow the sample number with the dilution factor, so that it will print out on the PeakNet report.

11.2.4 Under Sample Type, enter "Sample" for all.

11.2.5 Under Level, enter nothing

11.2.6 Under Method, choose the current calibration method.

11.2.7 Under Data File, change the file name to be "the date" followed by run number, followed by "_001.DXD". For example, the file name for the first sample analysis run on January 25, 2001 would be 2-012501RUN1_001.DXD. Copy this same file name down for all samples and QC checks.

11.2.8 Under Dilution, enter "1" no matter what dilution is used. The LIMS system will multiply the result by the dilution factor.

11.2.9 If the DX-120 should shut down after the analysis run, the last line of the schedule should have a name of "Shutdown" in the sample column. Sample type should be "Sample", and choose (SHUTDOWN.MET) under the method column. Note: Analyst must be present at end of run to turn off helium gas pressurizing external water source.

11.2.10 Click the "File/Save As".

11.2.11 Give the schedule a file name similar to the data file naming procedure. The file name should be "the date" followed by run number. For example, the file name for the second schedule run on January 25, 2001 would be 2-012501RUN2_.SCH

11.2.12 Print a hard copy of the schedule and save it for the data review process.

11.2.13 Close the Schedule Editor Window.

11.2.14 Place a copy of the schedule into IC #2 Log Run Book. Record the following information on the sheet: run date, analyst, calibration date, standards, and reagents.

11.3 Sample Analysis

11.3.1 Back at the Run Window, Highlight IC #2, click on the "Load Schedule" button, then, for data acquisitions, change to Drive D.

11.3.2 Double click on the schedule name as set above, in section 11.2.7.

11.3.3 Measure and record the conductivity of each sample on a calibrated conductivity meter. Any sample with a conductivity greater than the MCT requires dilution to avoid matrix interferences with the perchlorate peak. An appropriate dilution will yield a conductance below the MCT.

11.3.4 Filter all samples containing excess particulates through a 0.45 micron glass fiber filter.

11.3.5 Pour 5 ml of the appropriate sample (or 5 ml of a diluted sample) and the QC checks into the autosampler vials. Refer to the sequence in the schedule and position them into the appropriate autosampler tray position. Load trays into autosampler.

11.3.6 Push the "Hold/Run" button on the AS40 Autosampler #2 so that the light is in the Run position.

11.3.7 Push the "Load" button on the AS40 Autosampler #2.

11.3.8 Wait for the Load button to start blinking (1 to 2 minutes).

11.3.9 On the PeakNet Run window, click on the Run Menu, then Start.

11.3.10 *** At the conclusion of the run, be sure to turn off helium gas that is

pressurizing the DI reservoir. This is absolutely crucial. If the reservoir runs dry and helium is run through the suppressor, irreversible damage to the suppressor will occur. ***

12 Data Review

12.1 Review each sample report and its chromatogram to make certain that the retention time window has properly identified perchlorate. If the retention time is off, it can be adjusted through the Optimize/Name Peaks menu item. Preview the report to make certain that the correct peak has been reintegrated.

12.1.1 Review each sample report and its chromatogram to make certain that the retention time windows have properly identified each analyte. The width of the retention time window should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size. However, due to possible shifts in retention times for an individual sample due to the sample's ionic strength, the experience of the analyst should weigh heavily in chromatogram interpretation. If the identity of a peak is in question, the sample should be spiked and reanalyzed for confirmed.

12.2 Review each sample chromatogram to make certain that the baselines are correctly drawn. If the baselines need adjusted, this can be done through the Optimize/Adjust Baseline menu item.

12.3 Review each sample report to make certain that the reported concentration is within the lowest and highest calibration standard. If the result is above the highest standard, the sample must be rerun at a different dilution.

12.4 Prepare a data package. The cover page sheet should be a summary report identifying the LIMS batch number, analyst, data reviewer, date analyzed, sample results, dilutions and sample reporting limits for dilutions. This is followed by the printed PeakNet schedule and then all copies of the PeakNet sample reports/chromatograms in the same order as run. The package should be stapled/rubber banded together.

12.5 Review that all samples names and methods were entered correctly on the PeakNet schedule.

13 Calculations

13.1 No sample calculations are required. PeakNet and the LIMS perform all calculations.

13.2 Standard recovery is calculated as:

$$\%Recovery = (Result / True\ Value) \times 100$$

13.3 Spike recovery is calculated as:

$$\%Recovery = \frac{(Spike\ Result - Unspiked\ Sample\ Result)}{Spike\ Added} \times 100$$

13.4 Precision (RPD) is calculated as:

$$\%RPD = \frac{Result\ 1 - Result\ 2}{Average\ Result} \times 100$$

14 Reporting Results

14.1 When entering results in the LIMS, enter the sample result from the instrument and the dilution factor separately. The LIMS will multiply the dilution factor.

14.2 Report only those values that are greater than the reporting limit and fall between the lowest and highest calibration standards. For samples diluted due to matrix interferences, the reporting limits are also increased by the same sample dilution factor. If a sample is diluted because of an over-range analyte, the reporting limit should not be raised.

14.3 Report results in ug/l to three significant figures. Do not report to more decimal places than the last decimal of the reporting limit.

14.4 Spikes, matrix spike duplicates, and the internal QC samples all need to be reported in the LIMS.

14.5 If a sample is below the current reporting limit, then the sample should be reported as ND (non-detectable). Remember to adjust the reporting limits for any sample receiving a dilution.

15 References

15.1 EPA Method 314.0, Determination of Perchlorate In Drinking Water by Ion Chromatography, Revision 1.0, November 1999

15.2 Dionex DX-120 Operators Manual

15.3 PeakNet Users Guide

16 Waste Disposal

16.1 Refer to ALSI SOP 19-Waste Disposal.

17 Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

18 Definitions

18.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

Appendix A

Date _____ **Ion Chromatography Run Sheet** Batch Number _____

Tech _____ Calibration Date _____

Cup #	Sample	Dilution	Comment	Cup #	Sample	Dilution	Comment
1				39			
2				40			
3				41			
4				42			
5				43			
6				44			
7				45			
8				46			
9				47			
10				48			
11				49			
12				50			
13				51			
14				52			
15				53			
16				54			
17				55			
18				56			
19				57			
20				58			
21				59			
22				60			
23				61			
24				62			
25				63			
26				64			
27				65			
28				66			
29				67			
30							
31							
32							
33							
34							
35							
36							
37							
38							

Standard Log Number:
Reagent Log Number:

Page_____

Revision 6/00

SOP Change History Sheet

Section Number	Section Description	Reason for Change
8	Initial and Continuing Demonstration of Capability	Navy audit response
8.5-8.6	Initial and Continuing Demonstration of Capability	Navy audit response
9.2	Quality Control	Navy audit response

SOP Concurrence Form
For the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

Method Detection Limits
Metals

Analysis Method: 6010B/7471 (Hg)
Prep Method: 3050B
Instrument: IRIS ICP
Matrix: Soil

Analyte	Units	Analysis Date	MDL	Reporting Limit
Aluminum	mg/kg	12/6/05	**2**	**10**
Antimony	mg/kg	12/4/05	**0.6**	**3.0**
Arsenic	mg/kg	1/6/06	**0.6**	**3.0**
Barium	mg/kg	12/4/05	**0.1**	**1.0**
Beryllium	mg/kg	12/4/05	**0.04**	**1.0**
Bismuth	mg/kg	12/4/05	**1.0**	**5.0**
Boron	mg/kg	12/4/05	**1**	**10**
Cadmium	mg/kg	12/4/05	**0.2**	**1.0**
Calcium	mg/kg	12/4/05	**2**	**10**
Chromium	mg/kg	12/4/05	**0.3**	**1.5**
Cobalt	mg/kg	12/4/05	**0.05**	**2.5**
Copper	mg/kg	12/4/05	**0.3**	**1.5**
Iron	mg/kg	12/4/05	**1**	**10**
Lead	mg/kg	12/12/05	**0.3**	**1.5**
Selenium	mg/kg	12/4/05	**2**	**10**
Magnesium	mg/kg	12/6/05	**0.6**	**10**
Mercury	mg/kg	4/4/05	**0.0031**	**0.05**
Manganese	mg/kg	12/4/05	**0.2**	**1.0**
Molybdenum	mg/kg	12/4/05	**0.2**	**2.0**
Nickel	mg/kg	12/4/05	**0.1**	**2.0**
Silver	mg/kg	12/4/05	**0.1**	**0.5**
Strontium	mg/kg	12/4/05	**0.03**	**1.0**
Thallium	mg/kg	1/7/06	**1.1**	**5.5**
Tin	mg/kg	12/4/05	**0.6**	**5.0**
Titanium	mg/kg	12/4/05	**0.2**	**2.0**
Vanadium	mg/kg	12/4/05	**0.3**	**1.5**
Zinc	mg/kg	12/7/05	**0.7**	**3.5**
Potassium	mg/kg	12/4/05	**9**	**50**
Sodium	mg/kg	12/4/05	**2**	**50**

Method Detection Limit Studies
GC

Instrument: HPLC
Analysis Method: 8330
Matrix: soil

Analyte	Units	Analysis Date	MDL in use for reporting samples	Reporting Limit
HMX	mg/kg	5/18/06	**0.04**	**0.25**
RDX	mg/kg	5/18/06	**0.1**	**0.5**
1,3,5-Trinitrobenzene	mg/kg	5/18/06	**0.05**	**0.25**
Tetryl	mg/kg	5/18/06	**0.2**	**1**
1,3-Dinitrobenzene	mg/kg	5/18/06	**0.05**	**0.25**
2,4,6-Trinitrotoluene	mg/kg	5/18/06	**0.03**	**0.25**
Nitrobenzene	mg/kg	5/18/06	**0.1**	**0.5**
4-Amino-2,6-Dinitrotoluene	mg/kg	5/18/06	**0.1**	**0.5**
2-Amino-4,6-Dinitrotoluene	mg/kg	5/18/06	**0.1**	**0.5**
2,6-Dinitrotoluene	mg/kg	5/18/06	**0.05**	**0.25**
2,4-Dinitrotoluene	mg/kg	5/18/06	**0.04**	**0.25**
2-Nitrotoluene	mg/kg	5/18/06	**0.03**	**0.25**
4-Nitrotoluene	mg/kg	5/18/06	**0.03**	**0.25**
3-Nitrotoluene	mg/kg	5/18/06	**0.02**	**0.25**

DataChem MDL and LCS Limits Report

Analytical Method	Preparatory Method	Matrix	Date Analyzed	Instrument
6850	6850	WATER	3/12/2006	LC/MS

Analyte Name	Units	MDL	PQL	LCL	UCL
Perchlorate	ug/L	0.195	0.5	78.52	121.80

MDL Studies are required to be updated annually. Valid MDL Studies when approved are used. MDLs may change at any time. The above MDLs are valid MDLs used by DataChem currently. If you require more current MDL values please contact the laboratory. For multiple instrumentation DataChem uses the highest MDL values from all instruments in the study and a date range is given.

DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: The Determination of Perchlorate in Water, Soil and Biota by Liquid Chromatography / Mass Spectrometry

DOCUMENT CONTROL NUMBER: LC/MS-CLO4 - Revision 1

EFFECTIVE DATE: July 28, 2004

APPROVALS:

MANAGER _____ Date _____

QA MANAGER _____ Date _____

LAB DIRECTOR _____ Date _____

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF PERCHLORATE IN WATER, SOIL AND BIOTA BY LIQUID CHROMATOGRAPHY / MASS SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 This DataChem Laboratories method uses a liquid chromatography / mass spectrometry (LC/MS) method applicable to the determination of perchlorate in water, soil, and biota matrices.

1.2 A 100 µL portion of the sample or extract is introduced into a LC/MS. Perchlorate is separated by liquid chromatography, and partially fragmented for measure at mass 83 using mass spectrometry.

1.3 This method meets requirements of SW846 Method 8321A, SW846 Draft Method 6850, and is recommended for use only by or under the supervision of analysts experienced in the use of liquid chromatography and in the interpretation of mass spectrometry data.

2.0 CHANGES AND CLARIFICATIONS FROM THE REFERENCE METHOD

2.1 Not Applicable.

3.0 DETECTION LIMITS, INTERFERENCES, AND SAFETY

3.1 Reporting Limits

3.1.1 Reporting Limits under the conditions of this method are:

Analyte	PQL Water (µg/L)	PQL Soil (µg/Kg)	PQL Biota (µg/Kg)
Perchlorate	0.2	2.0	6.0

3.2 Interferences

3.2.1 Method interferences can be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus. These interferences can lead to false positive results for the target analyte.

3.2.2 All reagent solutions and samples (including QC samples) must be filtered through no larger than a 0.45 micron nominal pore size membrane of frit to remove particulates and prevent damage to the instrument, columns and flow systems. Filters specifically designed for IC or LC applications should be used.

3.3 Safety Precautions

3.3.1 Protective clothing must be worn when one works with corrosive or potentially corrosive materials or samples.

3.3.2 Safety glasses must be worn at all times in the laboratory.

3.3.3 Normal, accepted laboratory safety practices shall be followed during reagent preparation and instrument operation.

3.3.4 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

3.3.5 Refer to: DCL SOP LAB-005, "General Laboratory Safety and Chemical Hygiene" and the Safety Manual and Chemical Hygiene Plan of DataChem Laboratories (DCL).

4.0 APPARATUS AND CHEMICAL REAGENTS

4.1 Glassware/Hardware

4.1.1 Volumetric flasks, 100-mL and other sizes as needed

4.1.2 Eppendorf pipettes (10-µL to 10,000-µL)

4.1.3 Disposable autosampler vials

4.1.4 Disposable centrifuge tubes

4.1.5 Disposable plastic micro-beakers

4.1.6 Sample bottles: Polyethylene (or glass) of sufficient volume to allow replicate analyses

4.1.7 Disposable PALL 0.45-µm IC Acrodisc filters, with plastic 5-mL luer-lock syringes.

4.1.8 Burdick & Jackson C18 (2000-mg) columns.

4.2 Instrumentation

4.2.1 The system is an Agilent 1100 LC/MS or equivalent. Agilent 1100 LC/MS instrument conditions are presented in Figure 1. Instrument tuning and instrument conditions are based on the manufacturer's instructions. Tuning is not required prior to daily analyses. Tuning parameters are available from the instrument manufacturer, along with mass tuning solutions and instructions on how to optimize the mass spectrometer. Similar LC/MS systems from other manufacturers may also be used.

Figure 1. LC/MS Instrument Parameters

Agilent 1100 LC/MS

Pump Control
- Flow Rate: 0.5 mL/min
- Run Time: 13.0 min
- HPLC Mobile Phase:
  - Isocratic
  - 53.00%, Solvent A (95% ACN / 4.5% Water / 0.5% Acetic Acid)
  - 47.00%, Solvent B (94.5% Water / 5% ACN / 0.5% Acetic Acid)

Mass Spectrometer
- Ionization Mode: Electrospray
- Polarity: Negative

SIM Parameters

SIM Ion	Fragmentor	Gain (EMV)	Actual Dwell
83.00	160V	3.0	192 msec
85.00			192 msec
89.00			192 msec

Spray Chamber
- Gas Temp: 320°C
- Drying Gas (Nitrogen): 12.0 L/min
- Nebulizer Pressure: 50 psig

Capillary Voltage
- Negative: 1450 V

Autosampler and Column
- Injection Volume: 100 uL
- Column Temp: 30°C

4.2.1.1 Analytical column: KP-RPPX K' (Prime) Technologies, Inc. or equivalent.

4.2.2 A Chemstation Data System is used to determine peak areas.

4.2.3 An Analytical Balance is used to accurately weigh reagents used in the preparation of eluent and aliquots of solid samples. Analytical Balances are 4 and 5 places and must meet the acceptance criteria of 0.05% on test weights. See DCL SOP LAB-015 "Balances".

4.2.5 A hand-operated stainless steel grinder (Back to Basics Model SJ-27, or equivalent) is used to grind up biota (plant) samples in order to ensure complete extraction of perchlorate from the matrix.

4.2.6 A standard laboratory centrifuge is used to spin down particulates in biota (plant) samples that interfere with sample filtration.

4.3 Reagents

4.3.1 **ASTM Type II water** (ASTM D1193). Water shall be monitored for impurities.

4.3.2 **Acetonitrile (ACN)** (HPLC Grade) (CAS [75-05-8])

4.3.3 **Acetic Acid** (Glacial) (CAS [64-19-7])

4.3.4 **Sodium Perchlorate** ($NaClO_4$, CAS [7601-89-0]).

4.3.5 **Sodium Perchlorate ^{18}O**, containing 90% ^{18}Oxygen, Isotec Inc

4.3.6 **Mobile Phase Preparation**: ASTM Type II water and ACN are mixed in two one-liter bottles. Solvent A contains 95% ACN and 4.5% water (v/v) and the Solvent B contain 94.5% water and 5% ACN. 5ml acetic acid will be added to each bottle. The HPLC Mobile phase is an isocratic mixture of Solvent A (53%) and Solvent B (47%).

4.4 Reagents for Standard Preparation

4.4.1 Perchlorate stock standard solution, 1000µg/mL: A stock standard solution can be purchased as a certified solution or prepared from ACS reagent grade sodium salt as listed below. (NOTE: Sodium perchlorate represents a molar weight fraction of 81.2% perchlorate anion.)

4.4.1.1 Perchlorate (ClO_4^-) 1000µg/mL: Dissolve 0.123g of sodium perchlorate ($NaClO_4$, CAS [7601-89-0]) in ASTM Type II water and dilute to 100mL in a volumetric flask.

4.4.1.2 Stock standards may be stored at room temperature for a period up to 12 months. Expiration dates should be clearly specified on the label.

4.4.2 Intermediate Standard Solution (10ug/mL): Dilute 1000 µL of the stock standard solution to 100 mL with ASTM Type II water.

4.4.2.1 Calibration standards (See Section 5.2) may be made by further diluting the Intermediate standard.

4.4.2.2 Intermediate Standard Solutions may be stored at room temperature for a period up to 12 months. Expiration dates should be clearly specified on the label.

4.4.3 Internal Standard Stock Solution, approximately 1000ug/mL: A stock internal standard solution can be prepared from Oxygen-18 labeled Perchlorate salt as listed below.

4.4.3.1 O-18 labeled Perchlorate (ClO_4^-) approximately 1000 ug/mL: Dissolve 0.121g of Oxygen-18 labeled $NaClO_4$ in ASTM Type II water and dilute to 100mL in a volumetric flask.

4.4.3.2 Stock standards may be stored at room temperature for a period up to 12 months. Expiration dates should be clearly specified on the label.

4.4.4 Intermediate Internal Standard Solution (10ug/mL): Dilute 1000 uL of the stock internal standard solution to 100 mL with ASTM Type II water.

4.4.4.1 Intermediate Standard Solutions may be stored at room temperature for a period up to 12 months. Expiration dates should be clearly specified on the label.

4.4.5 Internal Standard Spiking Solution (approximately 1000 ug/L): Dilute 10.0 mL of the intermediate internal standard solution to 100 mL with ASTM Type II water.

4.4.5.1 Intermediate Standard Solutions may be stored at room temperature for a period up to 6 months. Expiration dates should be clearly specified on the label.

4.4.5.2 Each standard and sample requires 50µl of Internal Standard Spiking Solution per 10mL of sample or standard.

4.5 Reagents for QC Sample Preparation

4.5.1 Prepare QC Standard Solutions as described in Sections 4.4.1. and 4.4.2. Be sure to use a separate source from that used for the preparation of the calibration standards.

5.0 CALIBRATION AND STANDARDIZATION

5.1 Demonstration and documentation of acceptable initial calibration is required prior to analysis.

5.2 Working calibration standards are generated using various dilutions of the intermediate standard solution (Section 4.4.2). Analyze a minimum of six calibration standards as well as a blank standard. A sufficient number of standards must be analyzed to allow an accurate calibration curve to be established.

5.2.1 Standard concentrations generally used to determine a calibration curve are 0.2, 0.5, 1.0, 2.0, 5.0, and 10.0µg/L.

5.2.2 Internal Standard calibration (at 5.0µg/L) is used as per SW 846 8000B section 7.10.2.

5.3 *Initial Calibration*: The standard curve for each analyte is established by plotting the area ratio response for each standard against the concentration. The acceptance criterion for the initial calibration curve is a correlation coefficient of 0.995 or higher.

5.4 *Initial Calibration Verification (ICV):* Immediately after the calibration standards have been analyzed, the accuracy of the initial calibration shall be verified and documented by the analysis of an Initial Calibration Verification. The ICV is prepared from the QC stock standard solution at a concentration of 1.0 µg/L. When measurements exceed the control limits of ±15% of the true value, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

5.4.1 The Initial Calibration Verification analysis shall be conducted using an independent standard. An independent standard is defined as a standard comprising analyte material from a different source than that used in the standards for the instrument calibration.

5.5 *Continuing Calibration Verification (CCV):* Initial calibrations may be stable for extended periods of time. Once the calibration curve has been established, it MUST be verified for each analysis batch using a CCV prior to conducting any field sample analysis, every tenth field sample, and at the end of the analysis sequence. Percent recovery for the CCV must be ±15%.

5.5.1 CCV targets should alternate between high (10µg/L) and mid-range (1.0µg/L) levels.

5.6 *Retention Time Window*

A retention time window study in need not be performed as per SW846 8000B section 7.6 when using an internal standard.

5.7 Refer to: the Environmental Quality Assurance Program Plan of DCL, Appendix 14.7, "Summary of Calibration and Corrective Action".

6.0 SAMPLE HANDLING AND PRESERVATION

6.1 Refer to DCL SOP QS-DC-001, "Sample Receipt and Log-In (Environmental)" and the Environmental Quality Assurance Program Plan of DCL, Appendix 14.8, "Sample Preservation and Holding Times".

6.2 Samples should be collected in scrupulously clean glass or polyethylene bottles that meet EPA cleaning procedure D protocols.

6.2.1 Water samples should be collected in 125-mL polyethylene bottles; soil samples should be collected in 4-oz amber glass bottles.

6.3 Sample preservation and holding times for anions (perchlorate) determined by this method are as follows:

Matrix	Preservation*	Holding Time
Water	None required	28 days
Soil/Solid	None required	28 days
Biota	Non Required	28 Days

*Care should be taken to avoid temperature extremes during shipment.

7.0 SAMPLE PREPARATION AND ANALYSIS

7.1 Sample Preparation QC Requirements

7.1.1 At least one method blank consisting of deionized ASTM Type II water, shall be processed through each sample preparation and analysis procedure at a frequency of one per batch of twenty samples.

7.1.2 A Laboratory Control Sample (LCS) must be analyzed using the same sample preparations and analytical procedures employed for the field samples. The LCS shall be prepared from a separate source from the stock standards used for calibration.

7.1.3 One matrix spike (MS) sample shall be prepared at a frequency of at least one per 20 field samples.

7.1.4 One matrix duplicate (MD) or matrix spike duplicate (MSD) sample must be prepared at a frequency of at least one per 20 field samples. Duplicates are separate sample aliquots (soil or water) that are taken through the complete analytical process from preparation through analysis.

7.2 Water Sample Preparation

7.2.1 Ten mL of sample (or standard) is aliquoted to a 15-mL disposable centrifuge tube.

7.2.2 50µL of Internal Standard Spiking Solution is added to each sample.

7.2.3 50µL of glacial acetic acid is added to each sample.

7.2.4 Each sample is filtered through a PALL 0.45-µm IC Acrodisc filter into an autosampler vial for analysis.

7.3 Soil Sample Preparation

7.3.1 10 mL of ASTM Type II water is added to 1 gram of sample soil in a 15-mL centrifuge tube. Internal Standard Spiking Solution and glacial acetic acid are added to each sample (see sections 7.2.2 and 7.2.3). The mixture is vortexed, sonicated for at least 10 minutes, and vortexed again. If necessary, the sample is centrifuged. The extract is then filtered through a PALL 0.45-µm Acrodisc IC filter into an autosampler vial for analysis.

7.4 Biota (Plant) Sample Preparation

7.4.1 A sufficient portion (at least 10 grams) of sample is ground through a hand-operated stainless steel grinder.

7.4.2 30 mL of ASTM Type II water is added to 3 grams of sample matrix in a 50-mL centrifuge tube. Internal Standard Spiking Solution and glacial acetic acid (150µL each) are added to each sample (see sections 7.2.2 and 7.2.3). The

mixture is vortexed and left overnight, which allows for complete saturation of the sample.

7.4.3 Prior to analysis, the sample is vortexed again, then centrifuged at 5000 rpm for 30 minutes.

7.4.4 Approximately 6 mL of the supernatant is then drawn through an activated B&J C18 column, which removes a large portion of organic contaminants.

7.4.4.1 To activate the C18 cartridge column, draw approximately five mL of reagent-grade methanol through the column, followed by five mL of ASTM Type II water, being careful not to let the column go dry.

7.4.4.2 The first two mL of sample drawn through the C18 cartridge are discarded, as they are diluted by the liquid already in the column from the activation process.

7.4.5 The remainder (about 4 mL) of the resulting clear liquid is then filtered through a PALL 0.45-µm Acrodisc IC filter into an autosampler vial for analysis.

NOTE: Quantitative recovery of the sample through the C18 cartridge column is not necessary because there is no dilution or concentration of the sample. As noted in Section 7.4.4.2 above a portion of sample which elutes first is discarded because it is diluted with the activation solution on the cartridge.

7.5 Matrix Dilution: If analysis exceeds the calibration range the sample may be diluted or reduced volumes may be injected into the LC/MS.

8.0 PROCEDURE

8.1 Establish a stable baseline with working eluent running through the system. This requires approximately 15 to 30 minutes.

8.2 Establish a valid initial calibration as outlined in Section 5.3.

8.3 Load and inject a fixed amount of well-mixed sample. Record the resulting peak size in area units and the retention time. The autosampler and data system perform these functions automatically.

8.4 If the response for the peak exceeds the calibration range of the system, dilute the sample as per section 7.5 and reanalyze.

8.5 If the internal standard peak area response is greater than ± 30 % from the ICV or CCV (as appropriate) the sample must be reanalyze.

8.6 If manual integration is necessary it must be performed in accordance with DCL SOP XX-DC-024 "Manual Integration".

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Identify perchlorate in the sample chromatograms by:

9.1.1 Using the retention time of the internal standard

9.1.2 Evaluating the relative abundance of masses 83 and 85 ions in the chromatogram. The ratio should between 2.2 to 3.3 to confirm the presence of perchlorate. This ratio is derived from the natural abundance of chlorine isotopes 35 and 37

9.1.3 Use mass 83 for quantitation.

9.2 Sample concentration is calculated using internal standard calculation as per SW846 8000B section 7.10.2 and reported as follows:

9.2.1 Water Samples

Final Result (µg/L) = (C)(D)

Where:

C = Concentration from internal standard curve (µg/L)
D = Dilution factor (if needed)

9.2.2 Soil/Biota Samples

$$\text{Final Result } (\mu g/g) = \frac{(C)(V)(D)}{M} = \frac{(C)(30mL)(D)}{(3.0g)}\left(\frac{1L}{1000\,mL}\right)$$

Where:
C = Concentration in extract from internal standard curve (µg/L)
V = Volume of sample extract used for analysis (mL)
D = Dilution factor (if needed)
M = Mass of initial sample extracted (g)

10.0 QUALITY CONTROL

10.1 The requirements for the quality control (QC) program for this method consist of an , Laboratory Method Blank (MB), Laboratory Control Sample (LCS), Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Matrix Spike (MS), and either a Field (MD), Laboratory (QD), or Matrix Spike duplicate (MSD) sample analysis. This section details the specific requirements for each of these QC parameters.

10.1.1 Refer to: DCL SOP XX-DC-018, "Evaluation of Quality Control Data" and the DCL Environmental Quality Assurance Program Plan, Section 10, "Quality Control Procedures", Section 11, "Data Reduction, Verification, and Reporting", Section 12, "Corrective Action", Appendix 14.7, "Summary of Calibration and Corrective Action", and Appendix 14.10, "Batch QC and Corrective Action Flowcharts". Nonconformance procedures are in accordance with DCL SOP LAB-020, "Nonconformance/Corrective Action Report (NC/CAR) Procedures".

10.2 Method Detection Limit (MDL)

10.2.1 An MDL must be established for the instrument prior to any sample analysis. Follow the procedure outlined in DCL SOP Lab-024 "Calculation of Method Detection Limits". MDLs will be analyzed in reagent water. Additionally, MDL verifications must be analyzed as per section 4.3 of the DCL SOP Lab-024.

10.2.2 MDLs should be verified at least annually.

10.3 Minimum Reporting Level (MRL)

10.3.1 The Method Reporting Limit (MRL) is the threshold concentration of an analyte that a laboratory can expect to accurately quantitate in an unknown sample. The MRL must be established at an analyte concentration at least three times the MDL. Although the lowest calibration standard may be below the MRL, the MRL must never be lower than the lowest calibration standard.

10.4 Assessing Laboratory Performance: The following must be included in every analytical batch.

10.3.1 A Method Blank (MB) must be prepared and treated exactly as a field sample, including exposure to all glassware, equipment, solvents, filtration, and reagents that are used with field samples. Data produced are used to assess instrument performance of a blank sample and evaluate contamination from the lab equipment. Any perchlorate recovery in the MB that exceeds the 1/2 the RL indicates that contamination is present. The source of the contamination must be determined prior to conducting any sample analysis. Any sample included in an analysis batch that has an invalid MB must be reanalyzed in a subsequent batch after the contamination problem is resolved.

10.3.2 A Laboratory Control Sample (LCS) must be prepared and treated exactly as a field sample, including exposure to all glassware, equipment, solvents, filtration, and reagents that are used with field samples. Data produced are used to assess efficiency of the instrument performance and preparation procedures. Perchlorate recovery in the LCS must be within required limits of 80 – 120%. Alternate limits, historical or client supplied, may be used fro specific projects. If perchlorate recovery is outside control limits the preparation batch in question must be re-prepared and reanalyzed.

10.5 Assessing Analyte Recovery and Data Quality: The following must be included in every analytical batch.

10.5.1 Matrix Spike (MS): At least one matrix spike sample should be prepared at 1.0 µg/L for every analysis batch.

10.5.1.1 Individual analyte percent recoveries (%Rec) are calculated as follows:

$$\%\,\mathrm{Rec} = \frac{(\mathrm{SSR} - \mathrm{SR})}{\mathrm{SA}} \times 100$$

Where:
SSR = Spiked Sample Result
SR = Sample Result

SA = Spike Added

When sample concentration is less than the method detection limit, use SR = 0 for purposes of calculating %Rec.

10.5.1.3 The method control limits for %Rec are 80.0 – 120.0 for perchlorate. Clients may specify alternate limits for specific projects.

10.5.1.3.1 If the perchlorate concentration in the field sample does not exceed the spiked concentration by a factor of four, and the MS recovery is outside the control limits, a matrix effect is suspected, especially if all other QC data is within limit. The associated data should be flagged according to project specifications or noted in the comments section of the report.

10.5.1.3.2 If the perchlorate concentration in the field sample exceeds the spike concentration by a factor of four, the MS amount is considered negligible and the %Rec is not representative of the analytical procedure. Associated data are not flagged in the analytical report.

10.5.1.3.3 Repeated failure to meet method MS recovery criteria indicates potential problems with the procedure and should be investigated.

10.5.2 Matrix Spike Duplicates: A matrix spike duplicate (MSD) must be included in every analytical batch in order to establish the precision of the data. Generally, an MSD prepared at 1.0µg/L is the best alternative.

10.5.2.1 Calculate the relative percent difference (RPD) between the duplicate sample recovery and the initial sample recovery as follows.

$$RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where:
RPD = Relative Percent Difference
S = Sample Result
D = Duplicate Sample Result

10.5.2.2 The method control limit for RPD is 15% for Perchlorate for all samples above the reporting limit. Clients may specify alternate limits for specific projects.

10.5.2.3 If the analyte concentration in the sample and duplicate are greater than reporting limit and a matrix effect or non-homogeneous sample matrix is suspected, the project manager is contacted and the associated data must be flagged according to project specifications and noted in the comments section of the report.

10.5.2.4 RPD failure should not be a chronic problem. If it frequently recurs (>20% of duplicate analyses), it indicates a problem with the instrument or individual technique that must be corrected.

10.6 Responsibility for Inspection

10.6.1 The operations manager or designee is responsible for inspecting the work performed by the analysts to verify completeness and data quality.

10.6.2 The analysts performing this procedure have the responsibility to inspect: notebooks and worksheets for accuracy and completeness; samples for proper volume/size; labels, forms, and tags for accuracy; and equipment for proper maintenance and operation. (See DCL SOP LAB-028, "Documentation - Maintaining Instrument Records, Notebooks and Logbooks")

10.7 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method using laboratory control samples and/or Proficiency Testing Samples.

11.0 REPORTING RESULTS

11.1 Results shall be reported in the units and format consistent with the requirements of the contract or project covered by this procedure. Analytical anomalies shall be reported consistent with the above requirements, such as suspected matrix effects determined by matrix spike data or matrix duplicate data.

11.2 It is the responsibility of the operations manager or designee to verify that the results are accurate, precise, complete, and in compliance with Section 11.1.

11.3 It is the responsibility of the operations manager or designee to approve all reports issued by DCL.

12.0 PREVENTATIVE MAINTENANCE

12.1 Refer to DCL SOP LAB-002, "Preventive Maintenance for Analytical Instrumentation."

12.2 The LC/MS system should be flushed with isopropanol periodically (at least monthly) in order to clear out contaminants and/or blockages that may build up in the system.

12.3 The source module on the mass spectrometer should be checked daily. Any salt buildup on the source cone should be removed (using DI water) and the spray needle should be inspected for wear. The shape of the mist cone sprayed from the needle will indicate if the needle needs to be replaced.

13.0 WASTE MANAGEMENT

13.1 Refer to: DCL SOP LAB-004, "Hazardous Waste Handling and Disposal" and LAB-005, "General Laboratory Safety and Chemical Hygiene".

13.2 All processed samples are stored and disposed of following the procedures outlined in EA-DC-002, "Processed Sample Storage and Disposal Control."

14.0 DEFINITIONS

14.1 Refer to: the Environmental Quality Assurance Program Plan of DCL, Appendix 14.12, "Definitions and Terms".

15.0 REFERENCES

15.1 "*Determinative Chromatographic Methods*" SW846 8000B.

15.2 *Annual Book of ASTM Standards*, (Volume 11.01, 1990), Method D4327-88.

15.3 DCL SOP QS-DC-001, "Sample Receipt and Log-In (Environmental)".

15.4 DCL Environmental Quality Assurance Program Plan.

15.5 DCL SOP LAB-002, "Preventive Maintenance for Analytical Instrumentation."

15.6 DCL SOP XX-DC-018, "Evaluation of Quality Control Data."

15.7 DCL SOP QC-DC-001, "Internal Review of QA/QC Data."

15.8 DCL SOP LAB-030, "Documentation—Maintaining Instrument Records, Notebooks and Logbooks."

15.9 DCL SOP LAB-020, "Nonconformance/Corrective Action Report (NC/CAR) Procedures".

15.10 DCL SOP LAB-004, "Hazardous Waste Handling and Disposal".

15.11 DCL SOP LAB-005, "General Laboratory Safety and Chemical Hygiene".

15.12 DCL SOP LAB-024, "Calculation of Method Detection Limits."

15.13 DCL SOP LAB-015, "Balances".

15.14 DCL SOP XX-DC-024, "Manual Integration".

15.15 "*Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection*" SW846 8321A

15.16 *"PERCHLORATE IN WATER, SOILS AND SOLID WASTES USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ELECTROSPRAY IONIZATION/MASS SPECTROMETRY (HPLC/ESI/MS)"* Draft Method 6850

Document Title: Method 6020 – Determination of Trace Elements in Water and Waste by Inductively Coupled Plasma – Mass Spectrometry

Document Control Number: _____

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TABLE OF CONTENTS

1	Scope and Application	4
2	Summary of Method	5
3	Interferences	6
4	Safety	8
5	Apparatus and Materials	8
6	Reagents	10
7	Instrument Calibration	17
8	Quality Control	18
9	Sample Collection, Preservation, and Handling	21
10	Procedure	22
11	Calculations	24
12	Reporting Results	25
13	Waste Disposal	26
14	Pollution Prevention	26
15	Definitions	26
16	Troubleshooting	28
17	References	28
	Appendix A	30
	Appendix B	32

TABLE OF CONTENTS (continued)

Table 1 33

Table 2 34

Table 3 35

Table 4 36

Table 5 37

SOP Change Summary 38

SOP Concurrence Form 42

1 Scope and Application

1.1 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.2 This Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-µg/L concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required. This method is adapted from SW-846 Method 6020A, Revision I, January, 1998.

1.3 Method detection limits (MDLS) and linear working ranges will be dependent on the sample matrix, instrumentation and selected operating conditions. These will be up-dated on an annual basis as required in this method. Method detection limits can be found in the current metals department method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.4 ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability of Method 6020 in a multi-laboratory study on solid wastes are listed in Table 1. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, instrumentation, and operating conditions. In relatively simple matrices, detection limits will generally be below 0.02 µg/L.

1.5 If Method 6020 is used to determine any analyte not listed in Table 1, it is the responsibility of the analyst to demonstrate the accuracy and precision of the Method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality (see Section 4.0).

1.6 Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS

1.7 Users of the method must document and have on file the required initial demonstration performance data described in Section 8.5 prior to using this method.

1.8 An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard shall have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.9 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 Prior to analysis, samples which require total ("acid-leachable") values must be digested using appropriate sample preparation methods (such as Methods 3005 - 3051).

2.2 Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3 Interferences

3.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z).

A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

3.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal):

corrected arsenic signal (using natural isotopes abundances for coefficient approximations) =

(m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal),
(where the final term adjusts for any selenium contribution at 77 m/z),

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes [6]).

Similarly,

corrected cadmium signal (using natural isotopes abundances for coefficient approximations) =

(m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal),
(where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114).

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are **ONLY** illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision.

The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferant. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

3.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of an internal standard is less than 30 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.4 Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type

of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4 Safety

4.1 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.

4.2 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical shall be regarded as a potential health hazard and exposure to these compounds shall be as low as reasonably achievable. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

4.3 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides.

4.4 Analytical plasma sources emit radiofrequency radiation in addition to intense UV radiation. Suitable precautions shall be taken to protect personnel from such hazards. The inductively coupled plasma shall only be viewed with proper eye protection from UV emissions.

5 Apparatus and Materials

5.1 Inductively coupled plasma-mass spectrometer: Perkin Elmer Elan 6000 and Perkin Elmer Elan DRCe are currently in use.

5.1.1 Both systems are capable of providing resolution better than or equal to 1.0 amu at 10% peak height The instruments are capable of scanning the mass range of 5-250 amu and the data systems allow corrections for isobaric interferences and the applying of the internal standard technique

Note: If an electron multiplier detector is being used, precaution shall be taken, where necessary, to prevent exposure to high ion flux. Otherwise changes in instrument response or damage to the multiplier may result.

5.1.2 Argon gas supply: high-purity grade (99.99%) When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders. Argon is supplied by GTS.

5.1.3 Radio-Frequency generator compliant with FCC regulations.

5.1.4 A mass-flow controller on the nebulizer gas supply is required. A water cooled spray chamber may reduce some types of interferences. (e.g. polyatomic oxide species.

5.2 Analytical balance, with the capacity to measure to .1 mg for use in weighing solids.(Mettler PM2000 or equivalent).

5.2.1. An assortment of auto-pipettes capable of delivering volumes ranging from 20 ul to 10 mL with an assortment of high quality disposable pipet tips. These pipettes are calibrated using SOP 19-AP (Standard Operating Procedure for Calibration Checks for Auto-pipetters / dispensers)

5.2.2 Finnpipette 2 mL-10 mL (VWR Cat #53515-050 or equivalent).

5.2.3 Finnpipette 100-1000 µL (VWR Cat #53515-044 or equivalent).

5.2.3 Finnpipette 1000 µL (VWR Cat #53511-566 or equivalent).

5.2.5 Eppendorf pipet 10-100 µL (VWR Cat #53511-577 or equivalent).

5.2.6 Finnpipette tips 2 mL-10 mL are VWR Catalog #53516-178 or equivalent,

5.2.7 Tips for the Finnpipette 200-1000 µL and the Eppendorf 1000 µL are VWR Catalog #53508-876 or equivalent.

5.2.8 Tips for the Eppendorf 10-100 µL are VWR Catalog #53503-094 or equivalent.

5.2.9 Narrow mouth storage bottles, FEP (fluorinated ethylene propylene) with ETFE (ethylene tetraflourethylene) screw closures, 125 mL to 250 mL capacities. (Greenwood Products Catalog # DB08A)

5.3 ICP-MS tubing:

5.3.1 Black/ Black sample tubing SCP Science catalog # 020-030-07 or equivalent.

5.3.2 Black/White waste tubing. SCP Science catalog #020-030-021 or equivalent.

5.4 Autosampler Supplies

5.4.1. 17 x 100 polypropylene sample tubes. VWR catalog #60818-618 or equivalent.

5.4.2 17 x 100 dual position tube caps. VWR catalog #60819-091 or equivalent.

5.5 Labware – For determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. A clean laboratory work area designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace elements (1) by contributing contaminants through surface adsorption or leaching and (2) by depleting element concentrations through adsorption processes. All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) shall be sufficiently clean for the task objectives. All glassware shall be cleaned in accordance with ALSI's glassware procedure.

NOTE: Chromic acid must not be used for cleaning glassware.

6 Reagents

6.1 Reagents may contain elemental impurities that might affect the integrity of analytical data. Owing to the high sensitivity of ICP-MS, high-purity reagents shall be used whenever possible. All acids used for this method must be of ultra high-purity grade. Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when hydrochloric acid is used, however, it shall be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for the chloride polyatomic ion interferences must be applied to all data.

6.1.1 Nitric acid, concentrated EM Science Ultra Pure Acid, Cat. #EM-NX0608-6 or equivalent. Store at room temperature until the manufacturer's expiration date.

6.1.2 Nitric acid (1+1) - Add 500 mL conc. nitric acid to 400 mL of reagent water and dilute to I L. Store at room temperature for a maximum of 6 months.

6.1.3 Nitric acid (1+9) - Add 100 mL conc. nitric acid to 400 mL of reagent grade water and dilute to I L. Store at room temperature for a maximum of 6 months.

6.1.4 Hydrochloric acid, concentrated EM Science Ultra Pure Acid, Cat. #EM-HX0608-6 or equivalent. Store at room temperature until the manufacturers expiration date.

6.1.5 Hydrochloric acid (1+1) - Add 500 mL conc. hydrochloric acid to 400 mL of reagent grade water and dilute to 1 L. Store at room temperature for a maximum of 6 months.

6.1.6 Hydrochloric acid (1+4) - Add 200 mL conc. hydrochloric acid to 400 mL of reagent grade water and dilute to I L. Store at room temperature for a maximum of 6 months.

6.2 Reagent water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification system which provides analyte free, greater than 18.0 megohm-cm DI water on demand. This water is used for preparation of all reagents and standard.

6.3 Standard Stock Solutions - Stock standards may be purchased from a reputable commercial source or prepared from ultra high-purity grade chemicals or metals (99.99 - 99.999% pure). Stock standard solutions are purchased commercially and are NIST traceable certified solutions. When received in the lab each standard is assigned a unique log number and is recorded in the Standard Preparation Logbook (Appendix B), along with the manufacturer, date of receipt, expiration date, and the analyst's initials. These stock solutions may be stored at room temperature until the manufacturer's expiration date.

6.3.1 CPI stock solution (P/N 4400-130597) in 1%HNO3 +Trace HF or an equivalent NIST certified standard. This standard contains the following elements (all elements are at a concentration of 10 µg/mL). Al, Sb, As, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, V, and Zn.

6.3.2 CPI stock solution (P/N 4400-130775) in 1% HNO3 or an equivalent NIST certified standard. This standard contains the following elements (all elements are at a concentration of 10 µg/mL). Ba, B, Ca, Fe, Mg, Ag, and Sr.

6.3.3 The second source standard (ICV or QCS) is purchased commercially from QCD Analysts (P/N QCS26QK) or equivalent. The stock solution is in a 5% HNO3 matrix. The standard contains the following elements (all

elements are at a concentration of 100 mg/L). Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Tl, Ti, V, and Zn.

6.3.4 The Interference Check Solutions are purchased commercially and contain known concentrations of interfering elements. These solutions will show the magnitude of interferences and provide adequate tests of any corrections. There are two solutions: A and AB. The final concentrations found in A and AB are listed below. These are purchased from QCD Analysts, catalog #20MICSAK and #6020ALICD or equivalent.

6.3.4.1 Working Solution A – Prepare by adding 10 mL of Stock Standard A to a 100 mL volumetric flask. Add 10 mL of HNO3 (or that which matches the samples) and take up to volume. Store at room temperature. Prepare fresh daily. This solution contains the following analytes:

ELEMENT	CONCENTRATION
Cl	1800 ppm
Ca	300 ppm
Fe and Na	250 ppm
C	200 ppm
Al, Mg, P, K, and S	100 ppm
Mo and Ti	2 ppm

6.3.4.2 Working Solution AB – Prepare by adding 10 mL of Stock Standard A and 1 mL of Stock Standard AB to a 100 mL volumetric flask. Add 10 mL of HNO3 (or that which matches the samples)and take up to volume. Store at room temperature. Prepare fresh daily. This solution contains the following analytes:

ELEMENT	CONCENTRATION
Cl	1800 ppm
Ca	300 ppm
Fe and Na	250 ppm
ELEMENT	**CONCENTRATION**
C	200 ppm
Al, Mg, P, K and S	100 ppm
Mo and Ti	2 ppm
Cr, Co, Cu, Mn, Ni, and V	200 ppb
As, Cd, Se, and Zn	100 ppb
Ag	50 ppb

NOTE: The ICS solutions in Table 2 are intended to evaluate corrections for known interferences on only the analytes in Table 1. If Method 6020 is used to determine an element not listed in Table 1, it is the responsibility of the analyst to modify the ICS solutions, or prepare an alternative ICS solution, to allow adequate verification of correction of interferences on the unlisted element.

6.4 Working Standard Solutions: Prepare these standards in an acid matrix that is similar to the samples being analyzed. This is dependent on the type of digestion performed on the samples. The acid concentration shall be adjusted to match the samples being analyzed. After preparation, each standard is assigned a log number and is recorded in the standard preparation logbook along with the stock solution used, the concentration of the stock solution, the volume used, the final volume, the matrix, the date prepared, the date it will expire, and the initials of the preparer. These standards must be prepared fresh on a daily basis.

6.4.1 Calibration Blank: To a 100 mL volumetric flask, add 10 mL of HNO3 (or that which matches the samples) and bring up to volume using reagent water.

6.4.2 Standard 1 or Reporting Limit Standard (RLS): For elements whose reporting limit is 1 ppb (Be, Cd, Ag, Tl, and V) make a 1 ppm intermediate standard by taking 0.1 mL of each 1000 ppm stock standard and adding it to a 100 mL class A volumetric flask. For elements whose reporting limit is 2 ppb (Sb, Cr, Pb, and Mo) make a 2 ppm intermediate standard by taking 0.2 mL of each 1000 ppm stock standard and adding it to a 100 mL volumetric flask. Take 1 mL of each intermediate standard and add to a 1000 mL volumetric flask containing 100 mL of HNO3 (or that which matches the samples) and 500 mL reagent water. Bring up to volume with reagent water.

6.4.2.1 Silver Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. # 140-051-472 or equivalent NIST certified standard.

6.4.2.2 Beryllium Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. # 140-051-042 or equivalent NIST certified standard.

6.4.2.3 Cadmium Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. # 140-051-482 or equivalent NIST certified standard.

6.4.2.4 Thallium Stock Solution (1000 ppm) in 4 % HNO3. SCP Cat. # 140-051-812 or equivalent NIST certified standard.

6.4.2.5 Vanadium Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. # 140-051-232 or equivalent NIST certified standard.

6.4.2.6 Antimony Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. # 140-051-512 or equivalent NIST certified standard.

6.4.2.7 Chromium Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. # 140-051-242 or equivalent NIST certified standard.

6.4.2.8 Lead Stock Solution (1000 ppm) in 4% HNO3. SCP Cat. #140-051-822 or equivalent NIST certified standard.

6.4.2.9 Molybdenum Stock Solution (1000 ppm) in H2O. SCP Cat. #140-050-422 or equivalent NIST certified standard.

6.4.3 Standard 2: To a 100 mL volumetric flask, add 10 mL of HNO3 (or that which matches the samples) in reagent water, add 50 µL of CPI stock solutions 4400-130597 and 4400-130775. Bring up to volume with reagent water. All elements in this standard will be at a concentration of 5 ppb.

6.4.4 Standard 3: To a 100 mL volumetric flask, add 10 mL. of HNO3 (or that which matches the samples) in reagent water, add 0.2 mL of CPI stock solutions 4400-130597 and 4400-130775. Bring up to volume with reagent water. All elements in this standard will be at a concentration of 20 ppb.

6.4.5 Standard 4: To a 100 mL volumetric flask, add 10 mL of HNO3 (or that which matches the samples) in reagent water, add 2.0 mL of CPI stock solutions 4400-130597 and 4400-130775. Bring up to volume with reagent water. All elements in this standard will be at a concentration of 200 ppb.

6.4.6 Continuing Calibration Check Solution (CCV): Concentrations of the CCV must be mid-range of the calibration.. Therefore, a 100 ppb CCV is prepared.. To a 100 mL Class A volumetric flask, add 10 mL of HNO3 (or that which matches the samples) in reagent water and add 1 mL of stock solutions 4400-130597 and 4400-130775. Bring up to volume with reagent water. All concentration will be at a concentration of 100 ppb.

6.4.7 Initial Calibration Check Solution (ICV): To a 100 mL Class A volumetric flask, add 10 mL of HNO3 (or that which matches the samples) in reagent water, add 0.1 mL of QC26 solution (QCD Analysts,

Cat. #QCS26QZ). Bring up to volume with reagent water. All analytes are at a concentration of 100 ppb.

6.5 Internal Standards Stock Solution: To a 100 mL volumetric flask, add 3 mL of HNO3 and the following amounts of each internal standard. Bring up to volume with reagent water.

Product	Element	Stock Conc.	mL to add	Final Conc.
High Purity #100024-1	Indium	1000 ppm	2 mL	20 ppm
High Purity #100067-1	Yttrium	1000 ppm	2 mL	20 ppm
High Purity #100023-1	Holmium	1000 ppm	2 mL	20 ppm
High Purity #100057-1	Terbium	1000 ppm	2 mL	20 ppm
High Purity #100048-1	Scandium	1000 ppm	2 mL	20 ppm
QCD # 8703006	Lithium	100 ppm	20 mL	20 ppm
High Purity #100020-1	Germanium	1000 ppm	10 mL	100 ppm
SCP Science # 140-052-451	Rhodium	1000 ppm	2 mL	20 ppm

This solution must be added to all blanks, calibration standards, and samples. 0.05 mL of the internal standard stock solution shall be added to every 5 mL of blank, standard, or sample. This stock solution is stored at room temperature and is stable for a period of 3 months.

6.6 Blanks. Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

6.6.1 The calibration blank consists of the same concentrations of the same acids used to prepare the final dilution of the calibrating solutions of the analytes (often 1% HNO3 (v/v) in reagent water) along with the selected concentrations of internal standard element for each of the analytes.

6.6.2 The preparation (or reagent) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions.

6.6.3 The rinse blank consists of 6 percent HNO_3 (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.7 Stock tuning solution. This solution is used for instrument tuning prior to analysis and to verify that the instrument has reached thermal stability. The solution is prepared by mixing 1.0 mL of the stock solutions in a 100 mL volumetric flask.

The 6000 ICP/MS uses a solution containing Pb, Ba, Mg, Ce, U, In, Rh. The ICP/MS uses a solution containing Mg, Cu, Rh, Cd, In, Ba, Ce, Pb, U.

6.8 Working Tuning Solution: Add 0.1 mL of the 10 ppm Tuning solution to a 100 mL volumetric flask. Add 3 mL of HNO_3.(or that which matches the samples) Bring up to volume with reagent water. This will give a solution of 10 ppb. Use the 10 ppb solution as the working tuning solution and do not add internal standards to this solution.

6.9 Stock Mass Calibration Standard: Prepare this solution by adding 3 mL of HNO3 to a 100 mL Class A volumetric flask. Add 1 mL of the following elements to the flask: Cerium (High Purity # 100010-1), Rhodium (SCP Science #140-052-451), Lead (SCP Science # 140-051-822), Barium (SCP Science # 140-051-562), Magnesium (SCP Science # 140-051-122), and Uranium (SCP Science #140-051-921) or equivalent NIST approved standards. Bring up to volume with reagent water. This results in a 10ppm solution of these elements. Use this stock solution to prepare the actual tuning solution.

6.10 Working Mass Calibration Solution. Take a 500 mL Class A volumetric flask and Add 50 mL of HNO3 (or that which matches the samples) and 0.5 mL of the stock solution. Take up to volume with reagent water. Do not add internal standard to this solution. This results in a 10 ppb solution of the six elements above.

6.11 Laboratory Fortified Blank (LFB or LCS_): To a 100 mL aliquot of reagent water

add 1 mL aliquots from the QCD(#Q-AL-1 and #Q-AL-2 or equivalent) custom blend multi-element solutions. The final concentration is 100 ug/L for each element. The LFB must be carried through the same preparation method as the samples. Including sample digestion, when applicable. Add internal standards after preparation is complete.

6.12 Undigested Matrix Spike: To a 100 mL aliquot of sample, add 1mL aliquots from the QCD(#Q-AL-1 and Q-AL-2) custom blend multi-element solution. The final concentration is 100 ug/L for each element . Add internal standards after preparation is completed.

7 Instrument Calibration

7.1 The argon pressure shall be set between 50 and 52 psi. The plasma shall be set at 1000 watts for aqueous solutions and 1200 watts for solids. All other instrument settings are optimized daily and may change on a regular basis. Refer to the instrument operating manuals for more information on instrument settings.

7.2 Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples.

7.3 Instrument stability must be demonstrated by analyzing the tuning solution a minimum of five times. The resulting relative standard deviation for all analytes must be less than 2%.

7.4 Mass calibration and resolution checks must also be conducted in the mass regions of interest using the working mass calibration solution. Adjust the mass calibration if analytes differ from their unit mass by more than 0.05 amu. Adjust the spectrometer resolution if the peak width for all analytes is not within 0.75 ± 0.10 amu.

7.4.1 For the Perkin Elmer 6000, magnesium intensities shall be greater than 20000, rhodium intensities shall be greater than 150000, and lead intensities shall be greater than 100000. Background shall be less than 30 and the double charged ions and oxides shall be less than or equal to 0.030.

7.4.2 For the Perkin Elmer DRCe, magnesium intensities shall be greater than 50000, indium intensities shall be greater than 250000, and uranium intensities shall be greater than 200000. Background shall be less than 2 and the double charged ions and oxides shall be less than or equal to 0.030.

7.5 Internal standards must be used in all analyses to correct for instrument drift and physical interferences. A list of acceptable internal standards is provided in Table 2. Internal standards must be present in all samples, standards, and blanks at identical levels. This may be achieved by adding 0.05 mL of the Internal Standard Stock Solution (Section 6.5) to 5 mL of sample, standard, or blank.

7.6 The instrument must be calibrated using the internal standard technique described in Section 7.5 on a daily basis. The instrument must be calibrated for the analytes to be determined using the calibration blank and standards described in Sections 6.4.1 through 6.4.5. A minimum of three replicate integrations is required for data acquisition. Use the average of the integrations for instrument calibration and data reporting. The correlation coefficient for each analyte curve must be 0.995 or greater.

7.7 The rinse blank (Section 6.6.3) shall be used to flush the system between samples, standards, and blanks. Sufficient rinse time must be allowed to remove traces of the previous sample. Currently the rinse time is set at 60 seconds. Solutions shall be aspirated for 60 seconds prior to the acquisition of data to allow equilibrium to be established.

7.8 Samples that have concentrations exceeding the linear range for an analyte must be diluted and reanalyzed or measured using an alternate line.

8 Quality Control

8.1 All policies and procedures in the most current version of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Instrument Detection Limits (IDLs) in µg/L can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least every three months.

NOTE: Only isobaric elemental, molecular, and doubly charged interference corrections which use the observed isotopic-response ratios or parent-to-oxide ratios (provided an oxide internal standard is used as described in Section 3.2) for each instrument system are acceptable corrections for use in Method 6020.

8.3 Linear calibration ranges - Linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range shall be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care shall be taken to avoid potential damage to the detector during this process. The linear calibration range, which may be used for the analysis of samples, shall be judged by the analyst from the resulting data. The upper LDR limit shall be an observed signal no more than 10% below the level extrapolated from lower standards. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs shall be verified every 6 months or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined.

8.4 Method detection limits (MDL) shall be established for all analytes, using the procedure outlined in *ALSI SOP 99-MDL*. MDLs shall be determined annually, when a new operator begins work or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined. Reporting limits (PQL) are determined by multiplying the MDL by 3-5 times plus any safety factor that may be deemed necessary.

8.5 Demonstration of Capability (DOC): DOC's must be performed prior to independent analysis using this method and established annually as specified in the QA Plan, Technical Training. To perform DOC's, four consecutive Laboratory Control Samples (LCS) with a matrix matching that of the calibration standards are analyzed. The recoveries obtained must be within 80-120 % of the known values for each of the associated elements, and the consecutive reads must have an RSD less than 20%. If the DOC's are outside these acceptance limits, a new calibration curve must be established, and the LCS's reanalyzed. This process is repeated until the DOC's are completed successfully. For recertification the successful analysis of a blind performance sample (PT) may be used for the yearly DOC.

8.6 Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Calibration Blank	--	Beginning of run, after every 10 samples, and at the end of the run.	< 3 x IDL **For DoD** < 1/2 RDL	Reanalyze the blank, prepare new blank and analyze, perform maintenance on instrument , recalibrate, reanalyze any samples since the last acceptable blank. If reanalysis is not possible, report with a qualifying comment.
Method Blank (LRB)	--	One per batch of no more than 20 samples. Analyze with associated sample batch.	<reporting limit or <10% of sample concentration **For DoD** < 1/2 RDL	Reanalyze the blank. If still not acceptable, associated samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.
Laboratory Fortified Blank (LFB or LCS)	All analytes 100ppb	One per batch of no more than 20 samples. Analyze with associated sample batch.	80-120% (**DoD samples** require 80-120% recovery with the exception of Ag, Mo, and Se. The recoveries for these metals are 75-120%)	Reanalyze the LFB. If still outside of acceptable range, samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.
Parameter	**Concentration**	**Frequency**	**Control Limits**	**Corrective Action**
Quality Control Sample (QCS or ICV) Second Source Standard	All analytes are at 100 ppb	Immediately after calibration.	Mean concentration from 3 replicates must be within 90-110%	Reanalyze the ICV. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration.
Continuing Calibration Check Standard (CCV) Same Source	CCV conc. 100 ppb	Beginning of run, after every 10 samples, and at the end of the run.	90-110%	Reanalyze the CCV. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration. Reanalyze any samples since the last acceptable CCV. If reanalysis is not possible, report with a qualifying comment.
Reporting Limit Standard (RLS)	Standard # 1 Section 6.4.2	Used in Calibration Curve	+30% Part of the Calibration Curve	This standard does not have to be analyzed if it is part of the calibration curve.
* Matrix Spike (MS)	All analytes are at 100 ppb	One every 20 samples with at least one per batch.	75-125 % Recovery calculations are not required if the conc. of the analyte is greater than	If calibration verification standards are acceptable, reanalyze spike once. If the spike still fails or if reanalysis is not possible, report the results with a qualifying comment.

			4 times the spiking level. (**DoD samples** 80-120% recovery. Exceptions for Ag, Mo, and Se. recoveries are 75-120%)	If the LCS is acceptable and the specific matrix interference is known, report with a qualifying statement. If the interference in unknown reanalyze the sample and matrix spike to determine matrix effect or analytical error.
*Duplicate or matrix spike duplicate (MSD)	--	One every 20 samples with at least one per batch.	RPD ≤ 20%	Reanalyze the duplicate. If the RPD is still >20% or if reanalysis is not possible, report the results with a qualifying comment.
**Post-Digestion Spike	All analytes are at 100 ppb	Samples that have unacceptable matrix spike recoveries.	75-125%	Dilute the sample and reanalyze to compensate for the matrix effect. Results must agree to within 10% of the original determination.
Internal Standard Response	—	Added to all samples and QCs	> 30% of the response in the initial calibration standard.	Flush the instrument with rinse blank and monitor the response in the calibration blank. If acceptable, Dilute sample by factor of 4, and reanalyze. If after flushing the calibration blank response is unacceptable, terminate the analysis and determine cause of drift. May be due to partially blocked sampling cone or change in tuning condition of instrument.
Interference Check Standard	Listed in Section 6.3.4	At the beginning and end of each run and every 8 hours thereafter	ICSA: Absolute value for all non-spiked analytes < RL (unless they are verified trace impurities) ICSAB: 80-120% of expected value.	Rerun the check standard, if still unacceptable, recalibrate. Samples shall not be analyzed until acceptable.
Dilution Test	Sample must be at least 100x greater than the reagent blank	Every 20 samples	Within 10% of the original result	Interference must be suspected. Rerun. If still not acceptable, dilute again.

* Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike analysis may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

** Post Digestion Spike is performed when dilution test fails or when both the matrix spike and matrix spike duplicate fail for an analyte. The recovery for the post digestion spike must be within 75-125%. If the post digestion spike fails dilute sample and spike until the recovery is within the acceptance limits.

9 Sample Collection, Preservation and Handling

9.1 Sample Collection.

9.1.1 Samples must be collected in plastic or glass containers. For aqueous samples, the minimum sample amount is 150 mL. For soil samples, the minimum amount is 3.00 g.

9.2 Sample Preservation

9.2.1 Preserve aqueous samples using HNO_3 to a pH<2. Sample preservation shall be performed immediately upon sample collection. If this is not possible, then samples shall be preserved ASAP when received by the laboratory.

9.2.1.1 Following acidification in the laboratory, samples must be held 16 hours and then verified to be pH<2. If for some reason, the sample pH is verified to be greater than 2, more acid must be added and the sample held for an additional 16 hours until verified to be pH<2.

9.2.2 Soil samples must be preserved above the freezing point of water up to 6°C until analysis.

9.3 Sample Handling

9.3.1 All samples must be analyzed within 180 days of collection. All samples not analyzed within this time frame must be discarded and resampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report. Digested samples can be stored at room temperature until they are analyzed. Digestates must be stored separately from standard solutions.

9.3.2 For samples requiring digestion, refer to the Sample Preparation SOPs for procedures.

10 Procedure

10.1 Aqueous Sample Preparation – Dissolved Analytes

10.1.1 For the determination of dissolved analytes in ground and surface waters, pipet an aliquot (> 20 mL) of the filtered, acid preserved sample into a 50-mL polypropylene centrifuge tube. Add an appropriate volume of (1+1) nitric acid to adjust the acid concentration of the aliquot to approximate a 1% (v/v) nitric acid solution (e.g., add 0.4 mL (1+1) HNO, to a 20 mL aliquot of sample). If the direct addition procedure is being used, add internal standards, cap the tube and mix. The sample is now ready for analysis. Allowance for sample dilution shall be made in the calculations.

NOTE: If a precipitate is formed during acidification, transport, or storage, the sample aliquot must be treated using the procedure prior to analysis.

10.2 Sample Analysis

10.2.1 For every new or unusual matrix, it is highly recommended that the sample be screened for elements at high concentration. This may help prevent potential damage to the detector during sample analysis and identify elements that are higher than their linear range. This will also screen the sample for background levels of all elements being used as internal standards in order to prevent bias in the calculation of the analytical data.

10.2.2 Initiate the instrument operating configuration. Tune and calibrate the instrument for the analytes of interest.

10.2.3 Setup the run procedures for quantitative analysis. For all sample analyses, a minimum of three replicate integrations is required for data acquisition. Use the average of the integrations for data reporting.

10.2.4 All masses that might affect data quality must be monitored during the analytical run. This information shall be used to correct the data for identified interference.

10.2.5 During the analysis of samples, the laboratory must comply with the required quality control described in this SOP. Only for the determination of dissolved analytes or the "direct analysis" of drinking water with turbidity of < I NTU is the sample digestion step of the LRB, LFB, and LFM not required.

10.2.6 The rinse blank shall be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of one minute Samples shall be aspirated for 60 sec prior to the collection of data.

10.2.7 Samples having concentrations higher than the established linear dynamic range shall be diluted into range and reanalyzed. First analyze the samples for the trace elements. Then dilute and analyze the sample for the high concentration elements. Alternatively, the dynamic range may be adjusted by selecting an alternative isotope of lower natural abundance, provided quality control data for that isotope have been established. The dynamic range must not be adjusted by altering instrument conditions to an uncharacterized state. (**DoD** samples will be diluted to within the

calibration range or high-level check standard will be analyzed as part of the analysis. The acceptance limits will agree within +10 of expected value.)

10.3 Initial Set-up and Analysis for the Perkin Elmer ELAN 6000 ICP-MS and Perkin Elmer Elan DRCe

10.3.1 Perform the daily and as needed maintenance.

10.3.1.1 Check waste containers, empty if needed.

10.3.1.2 Clean the skimmer and sampler cones.

10.3.1.3 Change the sample tubing daily.

10.3.1.4 Change the waste tubing at least weekly, more frequently if needed.

10.3.2 Turn on the computer. Click on "start" at the bottom left hand corner of the screen. Move the mouse to "Programs". Move the mouse to the right and hold it on Elan 6000 (common). Move the mouse to the right and click on :Elan:. This will allow you to enter the ICPMS software.

10.3.3 The method that was used last will be on the screen. Click on the method box and click on sampling. Initialize the autosampler by clicking on "Probe". Then, click on "Go to Rinse". Start the pump by clicking on the device icon. Click on connect and the direction arrow that points to the right. Change the speed of the pump by typing the speed wanted in the box with the "rpm" next to it.

10.3.4 To light the plasma, click on the instrument window and click on "Start" under the word plasma. Allow the instrument to warm up for at least one-half hour.

10.3.5 Click on the File icon and then click on the Daily Performance method. Be sure the Daily Performance check meets the criteria listed in Section 7.12.2. Click on the File Icon and then click on the Tuning method. Click on the Tune Mass Spec box. All measured peak widths must be between 0.625 and 0.675 amu. The measured peak must be within + 0.05 amu of the actual mass value. Once these two measurements have passed the instrument is ready to analyze samples.

10.3.6 Click on the Method icon and then move the mouse to File. Under "File" click on "Open", click on the method that is going to be used.

10.3.7 Click on the Sample icon and then move the mouse to File. Under "File" click on "Open", click on the sample program that is going to be used.

10.3.8 Click on the Data Set icon and move the mouse to File. Click on "New". Use that day's date for naming the Data Set. If a second Data Set is used on the same day, add an A at the end of the date.

10.3.9 Click on the sample page that was opened. Samples are analyzed using this page. Click on the "Batch" box in the upper right hand corner. The calibration and beginning QC are programmed into the software. If not, follow the example on Table 5.

11 Calculations

11.1 Elemental equations recommended for sample data calculations are listed in Table 3.

11.2 Data values shall be corrected for instrument drift or sample matrix induced interferences by the application of internal standardization. Corrections for characterized spectral interferences shall be applied to the data. Chloride interference corrections shall be made on all samples, regardless of the addition of hydrochloric acid, as the chloride ion is a common constituent of environmental samples.

11.3 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration shall therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. See table 1 for the preferred isotopes to be reported.

11.4 The QC data obtained during the analyses provide an indication of the quality of the sample data and shall be entered into the LIMS.

11.5 Assessing Analyte Recovery and Data Quality

11.5.1 **LCS Recovery**:

$$\% \text{ Recovery} = (C_m / C_n) \times 100$$

where C_m = measured concentration of LCS
C_n = spiking concentration

11.5.2 **Spike Recovery:**

$$\% \text{ Recovery} = [(C_s - C_u) / C_n)] \times 100$$

where C_s = measured concentration of spiked sample aliquot
C_u = measured concentration of unspiked sample aliquot
C_n = spiking concentration.

11.5.3 **Precision (RPD):**

$$\% \text{ RPD} = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100$$

where: R_1= Matrix Spike Recovery
R_2= Matrix Spike Duplicate Recovery

12 Reporting Results

12.1 When entering results in the LIMS, enter the sample result from the instrument and the dilution factor separately. The LIMS will multiply the dilution factor.

12.2 All results available on the raw data shall be entered into the LIMS. When entering data into Horizon LIMS do not round off results. Horizon will automatically perform rounding appropriate to the method. Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte. This will allow the laboratory to provide "J" values to the client when they are requested. When "J" values are not requested.

12.3 Spikes, matrix spike duplicates, and the internal QC samples all need to be reported in the LIMS.

12.4 Report the actual result even if it is less than the reporting limit. Any sample with a result less than the reporting limit is reported as a ND (non-detectable by the Horizon LIMS); LIMS will automatically report the appropriate detection limit.

12.5 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing the first and second review.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Optimum concentration range: A range below which scale expansion must be used and above which curve correction shall be considered. This concentration range will vary with the sensitivity of the instrument and the operating conditions employed.

15.2 Sensitivity: The slope of the analytical curve. The functional relationship between emission intensity and concentration.

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

15.4 Total recoverable metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

15.5 Dissolved metals: The concentration of metals determined in a sample after the sample is filtered through 0.45-um filter.

15.6 Suspended metals: The concentration of metals determined in the portion of a sample that is retained by a 0.45-um filter.

15.7 Total metals: The concentration of metals determined in a sample following digestion by Methods 3010, 3015, 3020, 3050, 3051, or 3052.

15.8 Instrument detection limit (IDL): The concentration equivalent to a signal due to the analyte which is equal to three times the standard deviation of a series of 7 replicate measurements of a reagent blank's signal at the same wavelength or mass.

15.9 Interference check sample: A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.

15.10 Initial Calibration Verification Standard (ICV): A certified or independently prepared solution used to verify the accuracy of the initial calibration.

15.11 Continuing calibration verification (CCV): Used to assure calibration accuracy during each run. It must be run for each analyte. It shall be analyzed at the beginning of the run, after every ten samples, and after the last analytical sample. The concentration of this standard shall be at or near the mid-range level of the calibration curve.

15.12 Calibration standards: A series of known standard solutions used by the analyst for calibration of the instrument.

15.13 Linear dynamic range: The concentration range over which the analytical curve remains linear.

15.14 Method blank: A volume of reagent water processed through each sample preparation procedure.

15.15 Calibration blank: A volume of reagent water acidified with the same amounts of acids as were the standards and samples.

15.16 Laboratory control standard (LCS): A volume of reagent water spiked with known concentrations of analytes and carried through the preparation and analysis procedure as a sample. It is used to monitor loss/recovery values.

15.17 Method of standard addition (MSA): This technique involves the use of the unknown.

15.18 Sample holding time: The storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

15.19 Check standard: A solution containing a known concentration of analyte derived from externally prepared test samples. The check standard is obtained from a source external to the laboratory and is used to check laboratory performance.

15.20 Refer to ALSI QA Plan under Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to instrumentation used in this method.

17 References

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16. Dobb, David E., Rowan, J.T., and Cardenas, D., Lockheed Environmental Systems and Technologies Co., Las Vegas, NV; and Butler, L.C., and Heithman, E.M., U.S. EPA, Las Vegas, NV; "Determination of Mercury by ICP-MS".

17. SW-846, Method 6020A, Revision 1, January 1998.

Appendix A

Logbook Example

ICP-MS PE ELAN 6000
Analytical Worksheet

Data Set:						Cal-St0 log #:		
Date:						Cal–St1 log #:		
Analyst:						Cal-St2 log #:		
Matrix:						Cal-St3 log #:		
Parameter:						Cal-St4 log #:		
Cal:						QC26 log #		
						Cal Blank (ccb) #:		
						Rinse #:		
						Tuning Solution #:		
Sample ID	Seq.	AS Pos.	Sample ID	Seq.	AS Pos.	Sample ID	Seq.	AS Pos.
Oxides								
Sensitivity								
Mass Cal Mg								
Mass Cal Rh								
Mass Cal Pb								
Calibration Blank								
Cal-St0								
Cal-St1								
Cal-St2								
Cal-St3								
Cal-St4								

Appendix B

Metals Standard Preparation Logbook

Standard log#	Standard Name	Standard Conc.	Stock Standard			Final Volume	Final Matrix	Prep Date	Exp. Date	Pipette ID #	Comments	Tech
			Log #	Conc.	Vol. Used							

Table 1

List of Recommended Masses

Element of Interest	Isotope
Aluminum	27
Antimony	121,(123)
Arsenic	75
Barium	135,(137)
Beryllium	9
Boron	10,(11)
Cadmium	106, 108, (111), 114
Calcium	44
Chromium	(52), 53
Cobalt	59
Copper	(63), 65
Iron	54, (56), 57
Lead	(206), (207), (208)
Magnesium	24, 25, (26)
Manganese	55
Molybdenum	95, 97, (98)
Nickel	(60),62
Selenium	77, (82)
Silver	(107), 109
Strontium	(87)
Thallium	203, (205)
Thorium	(232)
Uranium	(238)
Vanadium	51
Zinc	(66), 67, 68
Krypton	83
Ruthenium	99
Palladium	105
Tin	118

Note: Isotopes recommended for analytical determination are bracketed.

Table 2

List of Acceptable Internal Standards

Internal Standard	Mass
Lithium	6
Scandium	45
Yttrium	89
Rhodium	103
Indium	115
Terbium	159
Holmium	165
Lutetium	175
Bismuth	209

Table 3

Elemental Equations

Element	Equation
Vanadium 51	-3.127 * ClO 53 + 0.3534 * Cr 52
Chromium 50	-0.9691 * Ti 49 - .002406 * V 51
Iron 54	-0.02823 * Cr 52
Zinc 66(2)	-0.00093 * Ba ++ 138 + 0.0014 Ga 71
Zinc 66(3)	-0.00093 * Ba ++ 138 + 0.0014 Ga 71 - 145.6 SO2 68
Zinc 67	-0.0335 * Ba ++ 138 + 0.05236 * Ga 71
Zinc 68	-0.11 * Ba ++ 138 + 0.1657 * Ga 71
Arsenic 75(1)	-3.127 * Se 77 + 2.529 * Se 82
Arsenic 75(2)	-3.127 * Se 77 + 0.9894 * Se 78
Selenium 82(1)	-1.001 * Kr 83
Selenium 82(2)	-1.001 * Kr 83 - 0.027 * Br 79
Selenium 78(1)	-0.0303 * Kr 83
Selenium 78(2)	-0.187 * Ar2 76 - 0.0303 * Kr 83
Selenium 78(3)	-0.187 * Ar2 76
Strontium 87	-0.386 * Rb 85
Molybdenum 98(1)	-0.1095 * Ru 101
Molybdenum 98(2)	-0.146 * Ru 99
Silver 107(2)	-0.2186 * ZrO 106
Silver 109(2)	-0.0005688 * ZrO 106
Cadmium 114	-0.02747 * Sn 118 - 1.629 * MoO 108
Cadmium 111	-1.073 * MoO 108 + 0.764 * Pd 106
Antimony 123	-0.1245 * Te 125
Indium 115	-0.01457 * Sn 118
Lead 206(Bi)	+1.00 * Pb 207 + 1.00 * Pb 208
Lead 206(Ho)	+1.00 * Pb 207 + 1.00 * Pb 208

Table 4
Reporting Limit Standard Concentrations

Analyte	Mass	PQL (ppb)
Aluminum	27	80.0
Antimony	121	2.0
Antimony	123	2.0
Arsenic	75	5.0
Barium	135	5.0
Barium	137	5.0
Beryllium	9	1.0
Cadmium	111	1.0
Cadmium	114	1.0
Calcium	44	50.0
Chromium	50	2.0
Chromium	52	2.0
Cobalt	59	1.0
Copper	63	5.0
Copper	65	5.0
Iron	54	30.0
Iron	57	30.0
Lead	206	2.0
Manganese	55	1.0
Molybdenum	95	2.0
Molybdenum	97	2.0

Analyte	Mass	PQL (ppb)
Molybdenum	98	2.0
Nickel	60	5.0
Nickel	62	5.0
Selenium	77	5.0
Selenium	78	5.0
Selenium	82	5.0
Silver	107	1.0
Silver	109	1.0
Strontium	87	2.0
Thallium	203	1.0
Thallium	205	1.0
Vanadium	51	1.0
Zinc	67	5.0
Zinc	68	5.0
Zinc	66	5.0
Magnesium	24	40.0
Boron	10	40.0
Boron	11	40.0
Mercury	200	0.2
Mercury	202	0.2

Table 5
Sample/Batch Report

User Name: jdavies
Computer Name: MS50
Sample File: D:\elandata\Sample\Letterkenny Ag.sam
Report Date/Time: Friday, July 27, 2001 13:30:57

A/S Loc.	Batch ID	Sample ID	Description	Sample Type	Init. Quant.	Prep. Vol.	Aliquot Vol.	Diluted Vol.	Solids Ratio
7		ICV	Initial Calib. Std.						
3		ICB	Initial Calib. Blank						
8		CCV	Continuing Calib. Std.						
10		A	Interference						
11		AB	Interference						
3		CCB	Continuing Calib Blk.						

SOP CHANGE HISTORY SHEET

Section No.	Section	Reason For Change
1.6	Scope and Application	SOP Update 5/23/05
6.1.1	Apparatus and Materials	SOP Update 5/23/05
6.2.1	Apparatus and Materials	A2LA Audit 5/23/05
7.1.1	Reagents and Standards	SOP Update 5/23/05
7.1.4	Reagents and Standards	SOP Update 5/23/05
7.3	Reagents and Standards	SOP Update 5/23/05
7.3.3.1	Reagents and Standards	SOP Update 5/23/05
7.4.2	Reagents and Standards	SOP Update 5/23/05
7.4.3	Reagents and Standards	SOP Update 5/23/05
7.4.4	Reagents and Standards	SOP Update 5/23/05
7.4.5	Reagents and Standards	SOP Update 5/23/05
7.6	Reagents and Standards	SOP Update 5/23/05
7.6.1	Reagents and Standards	SOP Update 5/23/05
7.7.1	Reagents and Standards	SOP Update 5/23/05
8.3	Quality Control	SOP Update 5/23/05
8.5.3	Quality Control	Added Section 5/23/05
8.5.4	Quality Control	Added Section 5/23/05

SOP CHANGE HISTORY SHEET (continued)

Section No.	Section	Reason For Change
QC Table	Quality Control (Calibration Blank)	SOP Update 5/23/05
QC Table	Quality Control (CCV)	SOP Update 5/23/05
9.6	Calibration and Standardization	SOP Update 5/23/05
9.8	Calibration and Standardization	SOP Update 5/23/05
9.9	Calibration and Standardization	SOP Update 5/23/05
9.14	Calibration and Standardization	SOP Update 5/23/05
10.1.1	Sample Collection, Preservation and Handling	SOP Update 5/23/05
10.3.1	Sample Collection, Preservation and Handling	SOP Update 5/23/05
12.7	Calculations and Reporting Results	SOP Update 5/23/05
12.8	Calculations and Reporting Results	SOP Update 5/23/05

Revision 3: 04/03/2006

1.8	Scope and Applications	Project requirements verbiage added
4.1	Safety	Availability to MSDSs added
5.1.1-5.6	Apparatus and Materials	Added and updated apparatus and materials
6	Apparatus and Materials	Added storage parameters
6.2	Reagents and Standards	Revised DI water megohm-cm
6.3.2, 6.3.3.	Reagents and Standards	Removed mercury reference
6.3.4.1	Reagents and Standards	Revised HNO3 volume

SOP CHANGE HISTORY SHEET (continued)

Section No.	Section	Reason For Change
6.3.4.2	Reagents and Standards	Revised HNO3 volume and Mo & Ti concentration, moved note from 6.6.3 to 6.3.4.2
6.4.2	Reagents and Standards	Revised preparation process; added RLS reference and standard stability
6.4.6	Reagents and Standards	Major revisions throughout section
6.5	Reagents and Standards	Removed references to #10006-1 & 100021-2, revised preparation volumes
6.7-6.12	Reagents and Standards	Major additions and revisions throughout
7	Instrument Calibration	Major revisions throughout section
8	Quality Control	Major revisions throughout section
10.2.6	Procedure	Removed mercury reference, revised aspiration time
10.2.7	Procedure	DoD audit response
10.3	Procedure	Added instrument names
11.1	Calculations	Deleted sample data reporting from this section
11.2	Calculations	Deleted data value reporting from this section
11.5	Calculations	Added detailing on assessing analyte recovery and data quality
12.2	Reporting Results	Deleted verbiage about which data values to report and sample dilution factor reporting limits
12.2, 12.4	Reporting Results	Added directions for Horizon LIMS entries

SOP CHANGE HISTORY SHEET (continued)

Section No.	Section	Reason For Change
12.5	Reporting Results	Added raw data requirements
15.20	Definitions	Added reference to QA Plan
16.1	Troubleshooting	Added section

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Document Title: Hot Plate Acid Digestion of Sediments, Sludges and Solids

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by: _____

_____

Helen MacMinn, Date
Quality Assurance Manager

_____

_____

Anna Milliken Date
Laboratory Operations Manager

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Validator

TABLE OF CONTENTS

1 Scope and Application 3
2 Summary of Method 4
3 Interferences 4
4 Safety 4
5 Apparatus and Materials 5
6 Reagents 5
7 Glassware Cleaning 8
8 Quality Control 9
9 Sample Collection, Preservation and Handling 12
10 Procedure 12
11 Calculations 14
12 Reporting Results 14
13 Waste Disposal 15
14 Pollution Prevention 15
15 Definitions 15
16 Troubleshooting 15
Appendix A 16
SOP Change History Summary 17

SOP Concurrence Form 20

1 Scope and Application

1.1 This method is an acid digestion procedure for the hot plate used to prepare sediments, sludges, and soil samples requiring digestion by Method 3050B of EPA SW846 "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," Revision 2, December 1996 for analysis by Inductively Coupled Plasma (ICP) and ICP/MS. Samples prepared by this method may be analyzed by ICP or ICP/MS as indicated below:

ICP

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Bismuth	Selenium
Boron	Silver
Cadmium	Sodium
Calcium	Strontium
Chromium	Thallium
Cobalt	Tin
Copper	Titanium
Iron	Vanadium
Lead	Zinc

ICP/MS

Aluminum	Manganese
Antimony	Molybdenum
Barium	Nickel
Beryllium	Silver
Boron	Strontium
Cadmium	Thallium
Chromium	Tin
Cobalt	Titanium
Copper	Vanadium
Lead	Zinc

1.2 CAUTION: The digestate formed for analysis by ICP/MS cannot be interchanged with a digestate formed for analysis by ICP. The appropriate digestate must be analyzed by the appropriate analytical method.

1.3 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.4 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 A representative 1.00 gram to 2.00 gram (wet weight) sample is digested in nitric acid and hydrogen peroxide. For ICP/MS analysis, the digestate is reduced in volume on the hotplate and then diluted to a final volume of 100 mL. For ICP analysis, the digestate is refluxed with hydrochloric acid. The digestate is reduced in volume on the hotplate and then diluted to a final volume of 100 mL.

3 Interferences

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material shall be processed to aid in determining whether Method 3050B is applicable to a given waste.

3.2 See the appropriate analytical method for interferences of individual analytes.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Therefore, each chemical shall be treated as a potential health hazard.

4.2 Analysts shall consult the material Safety Data Sheets (MSDS) for each chemical used in the digestion process. MSDSs are available to all staff and are located in the QA office.

4.3 Since the chemical make-up of the samples is not known, analysts shall treat the samples with extreme caution. Proper protective equipment must be used, including powder free PVC gloves, lab coats, safety glasses and a fume hood.

5 Apparatus and Materials

5.1 Erlenmeyer flask: 250 mL, purchased from Fisher Scientific 10-090B or equivalent

5.2 Watch glasses:
Plain 3" Fisher Scientific catalog no. 02-612C or equivalent
Ribbed 75mm Fisher Scientific catalog no. 02-613A or equivalent

5.3 Volumetric flasks: 100 mL, Class A, purchased from VWR catalog no. 29621-087 or equivalent

5.4 Centrifuge tubes, 50 mL capacity, VWR catalog no. 21008-177 or equivalent

5.5 Centrifuge, Garver Electrofuge or equivalent

5.6 Balance capable of weighing 0.01 g, Mettler PM2000 or equivalent

5.7 Finnpipette repeating pipette and various tips. VWR #53515-050 or equivalent

5.8 Thermometer, calibrated and capable of reading 100°C in 1° increments. Used for monitoring hot plate temperature. See Section 8.10 for mandatory calibration requirements.

5.9 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with ETFE (ethylene tetrafluorethylene) screw closure, 125 mL to 250 mL capabilities; Greenwood Products cat #DB08A or equivalent

5.10 Hot plate - adjustable and capable of maintaining 90 - 95°C, Lindeberg model no. 53015-RC or equivalent

5.11 Gloves, powder free PVC

5.12 PTFE Boiling Stones Fisher Scientific catalog no. 09-191-20 or equivalent

6 Reagents

NOTE: The expiration date of all standards and reagents shall be labeled on their respective containers. If an expiration date is not supplied by the manufacturer, the default expiration date shall be one year from the date opened.

6.1 Reagent Water - Reagent Water is water in which an interferant is not observed at

the analyte of interest. For this purpose, ALSI uses a Filson Water Purification System which provides analyte-free, 18 megohm-cm or greater deionized water on demand. All references to water in the method refer to reagent water unless otherwise specified.
NOTE: For metals digestions and analyses all water shall be taken from the plastic taps.

6.2 Concentrated nitric acid (HNO_3) - J.T. Baker, 'Baker Analyzed' grade, purchased from VWR catalog #JT9598-34 or equivalent.

NOTE: Acid bottles must be contained inside Teflon carriers at all times. If an expiration date is not supplied by the manufacturer, the expiration will be one year from the date opened. This date shall be marked directly on the acid bottle.

6.3 Concentrated hydrochloric acid (HCl) – Mallinckrodt 'Instrumental Analysis' grade, purchased from VWR catalog #MK5587-46 or equivalent.

NOTE: Acid bottles must be contained inside Teflon carriers at all times. If an expiration date is not supplied by the manufacturer, the expiration will be one year from the date opened. This date shall be marked directly on the acid bottle

6.4 Hydrogen Peroxide 30% (H_2O_2) - E. M. Science 'Suprapur' grade, purchased from VWR catalog #EM-7298-2 or equivalent.

NOTE: This reagent needs to be refrigerated and shall be kept in the organic prep laboratory refrigerator. The manufacturer's expiration dates must be applied.

6.5 Spiking Solution Standard, SCP P/N 901-6A5-800 (A) Stock Solution in 5% HNO_3, purchased from SCP Science or equivalent NIST certified standard. This is a pre-mixed standard of Ag, Cr, Ti, Mn, V, Cd and Sr at 20 mg/mL, As, Be, and Sb at 40 mg/mL, and Tl at 100 mg/mL in 5% HNO_3. This reagent can be stored in the manufacturer's bottle at room temperature. The marked expiration dates must be applied.

6.6 Spiking Solution Standard, SCP P/N 901-6A5-803 (C2) Stock Solution in 5% HNO_3, purchased from SCP Science or equivalent NIST certified standard. This is a pre-mixed standard of Al, B, Ni, Ba, Li, Pb, Se, Co, Mo, Zn, and Cu at 200 mg/mL, Fe at 2000 mg/mL, Na, K, Ca, and Mg at 1000 mg/mL, and Zn at 200 mg/mL in 5% HNO_3. This reagent can be stored in the manufacturer's bottle at room temperature. The marked expiration dates must be applied.

6.7 Spiking Stock Solution, SCP P/N 140-052-501 or equivalent. This stock Solution is purchased from SCP Scientific and is a NIST certified standard containing Sn at 1000 ppm in 20% HCl. The stock is stored at room temperature. The manufacturer's expiration dates must be applied.

6.7.1 Tin Working Spiking Solution, 40 mg/L.
To a Class A 200 mL volumetric flask, add 20 mL HCl (concentrate). Using a Class A pipette, deliver 8.0 mL of Spiking Stock 6.7 to the flask and dilute to volume with reagent water. Store at room temperature for 3 months.

6.8 Spiking Stock Solution, SCP P/N 140-001-835 or equivalent. This stock Solution is purchased from SCP Scientific and is a NIST certified standard containing Bi at 1000 ppm in 4% HNO_3. The stock is stored at room temperature. The manufacturer's expiration dates must be applied.

6.8.1 Bismuth Working Spiking Solution, 200 mg/L.
To a Class A 200 mL volumetric flask, add 10 mL HNO_3 (concentrate). Using a Class A pipette, deliver 40.0 mL of Spiking Stock 6.8 to the flask and dilute to volume with reagent water. Store at room temperature for 3 months.

6.9 1:1 (v/v) nitric acid (HNO_3). Using a graduated cylinder, add 500 mL of concentrated nitric acid to a 1000 mL Class A volumetric flask containing approximately 200 mL of deionized water. Dilute to 1000 mL with reagent water and mix thoroughly. This reagent must be given a lot number and documented in the reagent logbook. This reagent is stable for three months when stored at room temperature.

6.10 Lead Soil ELPAT spiking standards purchased from RTI International, or equivalent NIST traceable soil. These are proficiency standards that are part of the *Environmental Lead Proficiency Testing Program.* When digesting solid that are part of the IH (NLLAP), this ELPAT soil spike must be used for all QC (i.e. LCS, MS, and MSD).

6.11 Custom Mix CM-ANL-1 (5% HNO_3) or equivalent. This stock solution is a custom mixed multi-element blend, purchased from QCD Analysts containing Sb, Sn, Ag, and Ti at 100 µg/mL each. Discard according to manufacturer's label.

6.11.1 Spiking Solution Standard Mix #1, 10µg/mL
To a 100 mL Class A volumetric flask, add 5 mL HNO_3 (concentrate) and pipette 10 mL Custom Mix CM-ANL-1 (6.11). Bring to volume with

reagent water. Solution is stable for 3 months.

6.12 Custom Mix CM-ANL-2 (5% HNO_3), or equivalent. This stock solution is a custom mixed multi-element blend, purchased from QCD Analysts containing Al, As^{+3}, Ba, Be, B, Cd, Ca, $Cr^{(+3)}$, Co, Cu, Fe, Pb, Mg, Mo, Ni, Se, Tl, V, and Zn at 100 µg/mL each. Discard according to manufacturer's label.

6.12.1 Spiking Solution Standard Mix #2, 10 µg/mL
To a 100 mL Class A volumetric flask, add 5 mL HNO_3 (concentrate) and pipette 10 mL Custom Mix CM-ANL-2 (6.12). Bring to volume with reagent water. Solution is stable for 3 months.

7 Glassware Cleaning

7.1 Glassware used in this procedure is to be cleaned as noted in the glassware washing standard operating procedure (ALSI SOP 09-GLWH).

7.2 All glassware used in this procedure shall be soap and tap water washed, tap water rinsed and D.I. water rinsed. The glassware shall then be placed in the prep acid bath for at least 4 hours. When glassware is removed from the acid bath, it shall be rinsed with D.I. water, 10% nitric acid rinsed, and D.I. water rinsed before being used. (Note: The acid baths have a normal expiration date of three months. Change baths accordingly unless evidence of contamination indicates more frequent changes.)

7.3 Glassware is stored in metals prep area and rinsed with 10% nitric acid before use.

7.4 Lab ware – For determination of trace elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. A clean laboratory work area designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface adsorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable lab ware (glass, quartz, polyethylene, PTFE, FEP, etc.) shall be sufficiently clean for the task objectives.

7.5 Chromic acid must NOT be used for cleaning glassware.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Prior to performing analyses on NLLAP samples, Lead analysts/technicians shall have completed an external and/or internal training course for Lead or applicable metals analysis and have demonstrated ability to produce reliable results through accurate analysis of standard reference materials (ELPAT), proficiency testing samples, or in-house quality control samples. Their performance must be documented.

8.3 Demonstration of Capability (DOC) – Analysts/Technicians must perform DOC's prior to performing this method. DOC's will also be performed annually and any time that there is a significant change in the digestion procedure. This demonstration can be a part of the analysis of proficiency testing materials or quality control samples associated with routine sample runs. Individual's training records must be updated as these DOC's are performed. (DOC's are prepared as four separate laboratory control samples, and the involved batch has all of the necessary QC. The DOC's are analyzed and the data is placed into an analytical spreadsheet. The analyte recoveries must be within the method requirements of 80-120% and their relative percent difference must be less than or equal to 10%. If the DOC recoveries fall outside these method requirements, the analyst must repeat the study until he or she is deemed proficient.)

8.3.1 The DOC's for the IH NLLAP Lead program must be prepped using the ELPAT Lead Soil Standard. The analyte recoveries must be within the method requirements of 80-120% and their relative percent difference must be less than or equal to 10%, 75% of the time. If the DOC recoveries fall outside these method requirements, the analyst must repeat the study until he or she is deemed proficient.

8.3.2 Ongoing proficiency must be established annually as specified in the QA Plan, Technical Training.

8.4 Contamination Control – Laboratory dust wipe sampling and analysis shall be conducted at least quarterly to determine surface contamination levels of lead in the associated areas. Sample preparation is not to proceed until surface contamination is less than the specified maximum allowable concentration of 40 $\mu g/ft^2$.

8.5 All mechanical pipettes used in this digestion process must be calibrated annually to verify the ***entire*** range of use. Select four or five settings throughout the pipette's range and perform a full calibration at each setting. This calibration data must be recorded in the metals autopipette calibration logbook. (See SOP 09-AP for calibration procedure.)

8.5.1 A single setting pipette calibration is also required on a weekly basis to verify dispensing volumes. Select a mid-range volume to perform this calibration and be certain to document the procedure in the metals autopipette logbook. (See SOP 09-AP for calibration procedure.)

8.5.2 The ALSI Lab I.D. number of all mechanical pipettes used in this digestion process must be documented in the digestion logbook (Appendix A).

8.6 A matrix spike and a matrix spike duplicate or sample duplicate must be prepared in every batch of up to 20 samples to determine accuracy and precision.

8.6.1 Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.6.2 Samples to be analyzed by ICP shall be spiked in the following manner. Using a Finnpipette, add 1 mL of Spiking Solution 901-6A5-800 (Section 6.5), 1 mL of Bismuth Working Spiking Solution (Section 6.8.1) and 1 mL of a 1:1 mixture of Spiking Solution Standard 901-6A5-803 (Section 6.6) and Tin Working Spiking Solution (Section 6.7.1) to the sample before placing on the hot plate. Record the spike amounts and lot numbers used in the digestion logbook (Appendix A).

8.6.3 Samples to be analyzed by ICP/MS shall be spiked in the following manner. Using a Finnpipette, add 1.0 mL of Spiking Solution Standard Mix #1 (Section 6.11.1) and 1.0 mL of Spiking Solution Standard Mix #2 (Section 6.12.1) to the sample before placing the sample on the hot plate. Record the spike amounts and lot numbers used in the log book (Appendix A).

8.6.4 Digestion batches that include IH NLLAP LEAD samples must be spiked

with an NIST traceable standard that is of similar matrix. Using an ELPAT Lead Soil Standard (6.10), weigh out 0.1 g into all related QC. Document the spike amounts and the ELPAT Round number in the digestion logbook (Appendix A).

8.7 The expiration date of all standards and reagents shall be labeled on their respective containers. If an expiration date is not supplied by the manufacturer, the default expiration date shall be one year from the date opened.

8.8 Method Blanks shall be performed at a frequency of one per batch of no more than 20 samples per matrix. The Method Blank must be carried through the entire sample preparation and analytical process.

8.8.1 The Method Blank is prepared by adding 1.00 g of PTFE Boiling Stones to an empty 250 mL Erlenmeyer flask and proceeding with Section 10.1

8.8.2 The results of this analysis are used to assess batch acceptance. The source of Method Blank contamination shall be investigated, and measures taken to correct, minimize, or eliminate the problem if the concentration of the Method Blank exceeds the reporting limit for all samples except DOD samples. For DOD samples corrective action is taken if the concentration exceeds one-half the reporting limit. If this criteria is exceeded and an analyte is detected in the sample, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:

8.8.2.1 The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of the sample or

8.8.2.2 The blank contamination is greater than 1/10 of the project specified limits, then

8.8.2.3 Any samples associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparatory batch. If no sample volume remains for reprocessing, the results shall be reported with an appropriate data qualifying statement.

8.8.2.4 If contamination is detected in a blank, but the analyte is detected below the reporting limit in the sample (1/2 RL for DoD), data is not impacted and the sample result is reportable.

8.9 A Laboratory Control Sample (LCS) must be prepared with each batch of up to 20 samples. This sample is a Method Blank (Section 8.8) spiked as noted in Section 8.6.2 or 8.6.3 above (depending on analysis type) and brought through the digestion process.

8.9.1 MDL studies must be performed according to SOP 99-MDL or the reference methods, whichever is more frequent. To perform an MDL Study at least 7 replicates will be performed using the spiking standards. All replicates analyzed must be used when calculating the MDL values. In addition, the same method criteria used to calculate samples must be applied to the MDL Study

8.10 Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency
Method Blank (MB)	--	One per batch of no more than 20 samples.
Laboratory Fortified Blank (LFB or LCS)	See Sections 8.6.2, 8.6.3 & 8.6.4	One per batch of no more than 20 samples.
Matrix Spike (MS)	See Sections 8.6.2, 8.6.3 & 8.6.4	One per batch of no more than 20 samples.
Duplicate or matrix spike duplicate (MSD)	For MSD see sections 8.6.2, 8.6.3 & 8.6.4	One MSD per batch of no more than 20 samples. Sample duplicates.

Refer to appropriate analytical SOP for acceptance criteria and corrective actions.

9 Sample Collection, Preservation and Handling

9.1 Plastic and glass containers are both suitable for collection of samples.

9.2 A minimum sample amount of 3.00 g is needed.

9.3 Nonaqueous samples shall be refrigerated upon receipt and digested as soon as possible. Analysis must be completed within 180 days of collection.

9.4 Digestates are stored at room temperature in the metals lab to facilitate segregation from samples.

10 Procedure

10.1 NOTE: Any apparatus coming into contact with the sample (container, beakers, volumetric flasks, etc.) must be pre-rinsed with both 10% HNO_3 and reagent water prior to use. Additionally, see Section 7.4 for laboratory work area considerations.

10.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh to the nearest 0.01 g and transfer to an Erlenmeyer flask a 1.00 gram to 2.00 gram portion of sample.

10.3 Using a Finnpipette, add 10 mL of 1:1 HNO_3 (Section 6.9), mix the slurry and cover with a plain watch glass. Heat the sample to 95°C ± 5º C and reflux for 10 to 15 minutes without boiling.

10.4 Allow the sample to cool, add 5 mL of concentrated HNO_3 (Section 6.2) using a Finnpipette, replace the watch glass, and reflux for 30 minutes. Repeat this last step over and over until no brown fumes are given off by the sample indicating complete oxidation. Maintain a temperature of 95º C ± 5º C.

10.5 Replace the plain watch glass with a ribbed watch glass and allow the sample to evaporate for about 2 hours to a low volume of approximately 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker at all times. Maintain a temperature of 95º C ± 5º C.

10.6 After Step 10.5 has been completed and the sample has cooled, add 2 mL of reagent water and 3 mL of 30% H_2O_2 (Section 6.4) with a Finnpipette. Cover the flask with a plain watch glass and return the covered flask to the hot plate for warming, 95º C ± 5º C, 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 beaker.

10.7 Continue to add 30%H_2O_2 in 1 mL increments with warming until the effervescence is minimal. Do not add more than a total volume of 10 mL of 30% H_2O_2.

10.8 Return the sample to the hot plate with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL, maintaining a covering of solution at all times and a temperature of 95º C ± 5º C.

10.9 For samples requiring ICP/MS analysis, continue with section 10.10. For samples requiring ICP analysis, skip to section 10.11.

10.10 After cooling, if the digestate contains particulates, quantitatively transfer to a 50 mL centrifuge tube. Centrifuge the sample for five minutes and pour off the supernatant into a labeled 100 mL Class A volumetric flask. Add approximately 40 mL of reagent water and centrifuge sample again for five minutes. Pour off supernatant into the same labeled 100 mL Class A volumetric flask and dilute to the mark with reagent water. If the digestate does not contain particulates, transfer by pouring the sample into a 100 mL Class A volumetric flask. Dilute to the mark with reagent water. Label the bottle with sample number, digestion type, preparation factors and batch number. Complete the digestion log book. Sample digestion is complete.

10.11 If the sample is being digested for ICP analysis, remove the sample from the hot plate and let it cool.

10.12 After cooling, add 10 mL of concentrated HCl (Section 6.3). Return the sample to the hot plate with a plain watch glass, and reflux for 15 minutes without boiling at 95°C ± 5º C.

10.13 After cooling, if the digestate contains particulates, quantitatively transfer to a 50 mL centrifuge tube. Centrifuge the sample for five minutes and pour off the supernatant into a labeled 100 mL Class A volumetric flask. Add Approximately 40 mL of reagent water and centrifuge sample again for five minutes. Pour off supernatant into the same labeled 100 mL Class A volumetric flask and dilute to the mark with reagent water If the digestate does not contain particulates, transfer by pouring the sample into a 100 mL Class A volumetric flask. Dilute to the mark with deionized water. Label the bottle with sample number, digestion type, preparation factors and batch number. Complete the digestion log book. Sample digestion is complete.

11 Calculations

11.1 Not applicable.

12 Reporting Results

12.1 Verify that all required information has been entered into the digestion logbook.

12.2 Open the production Horizon LIMS and go to “batch posting” under the

Operations TAB. Enter the Horizon Batch Number (HBN), which corresponds to MDIG digestion batch. When the batch opens, the samples will already have default initial volumes of 1.00 g and final digestate volumes of 100 mL. These weights will need to be adjusted to match the weights in the hot plate digestion logbook.

12.3 Once the sample prep factors have been updated, the analyst initials, final prep date and time need to be entered in their appropriate fields. When entering data into Horizon LIMS do not round off results; Horizon will automatically perform rounding appropriate to the method.

12.4 A second analyst must verify that all information entered in steps 12.1 through 12.3 is correct and hit the save TAB in the upper left corner of the Horizon page. Once the batch is saved, the prep factors are applied to the samples, and the META analysis code becomes available for the Metals Department. The analyst performing the second review enters the date and initials the logbook at the bottom of the page.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Check for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this method.

16.2 Other possible problems that may occur can be found in Section 7.4 and Section 8.4

APPENDIX A

Work continued from page:_____ Work continued to page:_____

EPA Method:_____ LIMS ID Number: _____ ALSI SOP:_____	Hot Plate ID Number:_____ Thermometer Number:_____ Starting Temperature:_____ Finishing Temperature:_____ Batch Number:_____	Starting Date:_____ Time:_____ Tech:_____ Finishing Date:_____ Time:_____ Tech:_____

Line#	Sample #	Spike Amount	Spike Lot #	Sample Amount	Final Volume	Comments	Pipette ID #
1.)							
2.)							
3.)							
4.)							
5.)							
6.)							
7.)							
8.)							
9.)							
10.)							
11.)							
12.)							
13.)							
14.)							
15.)							
16.)							
17.)							
18.)							
19.)							
20.)							
21.)							
22.)							
23.)							
24.)							
25.)							
26.)							

Comments:

1.) Reagent:1:1 HNO3 _____

2.) Reagent:Conc. HCl _____

3.) Reagent:Conc. HNO3 _____

4.) 30% H2O2_____

5.) _____

HOT PLATE DIGESTION

SOP Change History Summary

Section	Section Description	Reason for Change
6.0-6.8	Reagents	SOP Update 04/21/05
6.10	Reagents	A2LA Response 04/21/05
7.2	Glassware Cleaning	SOP Update 04/21/05
8.3	Quality Control	SOP Update 04/21/05
8.3.4	Quality Control	A2LS Response 04/21/05
8.7, 8.8	Quality Control	SOP Update 04/21/05
8.8.1	Quality Control	A2LS Response 04/21/05
8.9	Quality Control	SOP Update 04/21/05
8.10, 8.11	Quality Control	A2LS Response 04/21/05
Revision5: 02/21/06		
1	Scope and Application	Navy audit response
1.1	Scope and Application	Spelled out ICP, alphabetized list, replaced graphite furnace with ICP/MS references
1.5	Scope and Application	Added project requirement verbiage
2	Summary of Method	Replaced graphite furnace with ICP/MS references
4.2	Safety	Identified MSDS location
4.3	Safety	Added “powder free”

SOP Change History Summary (continued)

Section	Section Description	Reason for Change
5	Apparatus and Materials	Navy audit response
5.1	Apparatus and Materials	Change in flask detailed
5.2	Apparatus and Materials	Added vendor details
5.4	Apparatus and Materials	Added vendor details
5.5	Apparatus and Materials	Added vendor details
5.7	Apparatus and Materials	Calibration reference added
5.9	Apparatus and Materials	Added container specifications
5.11	Apparatus and Materials	Added gloves
5.12	Apparatus and Materials	Added boiling stones
6.5	Reagents	Added storage and holding parameters
6.11, 6.12	Reagents	Added reagents and spiking concentrations
7.3	Glassware Cleaning	Added storage requirements
7.4, 7.5	Glassware Cleaning	Sections added for additional instruction
8.0	Quality Control	Section re-arranged and details added
8.3.2	Quality Control	Statement of ongoing proficiency added
8.5	Quality Control	Deleted MS/MSD/SD preparation frequency
8.6	Quality Control	Section reference and volumetric changes
8.8.1	Quality Control	Use of boiling stones added

SOP Change History Summary (continued)

Section	Section Description	Reason for Change
8.8.2.4	Quality Control	Added section
8.9	Quality Control	Section reference revision
8.9.1	Quality Control	MDL studies reference added
9	Sample Collection, Preservation…	Sampling and storage details added
10	Procedure	Details added to clarify and enhance
10 (various)	Procedure	Temperature parameters added
10.2	Procedure	Flask detail added
10.10/10.13	Procedure	Additional steps included
12.2	Reporting Results	Detail added for 2nd review
12.3	Reporting Results	Rounding directions added
15	Definitions	Section added
16	Troubleshooting	Section added
A	Appendix	Digestion Logbook example page added

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Document Title: Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP or GFAA Spectroscopy

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC.
(ALSI)

Address: 34 Dogwood Lane
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Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Alan Lopez, Date
Laboratory Technical Manager

_____ _____
KaTonna Williams Date
Validator

TABLE OF CONTENTS

1 Scope and Application 3

2 Summary of Method 3

3 Interferences 4

4 Safety 4

5 Apparatus and Materials 5

6 Reagents 5

7 Glassware Cleaning 6

8 Quality Control 6

9 Sample Collection, Preservation and Handling 10

10 Procedures 10

11 Calculations 13

12 Reporting Results 13

13 Waste Disposal 14

14 Pollution Prevention 14

15 Definitions 14

16 Troubleshooting 15

Appendix A (Microwave Digestion Logbook) 16

SOP Change History Sheet 18

SOP Concurrence Form 21

1 Scope and Application

1.1 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.2 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by graphite furnace (GFAA), and inductively coupled argon plasma spectroscopy (ICP). The procedure is an acid digestion for determining total available metals on samples requiring digestion by Method 3015 of EPA SW846 "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," Revision 0, September 1994.

1.3 Samples prepared by Method 3015 may be analyzed by GFAA or ICP for the following metals:

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Barium	Molybdenum
Beryllium	Nickel
Cadmium	Potassium
Calcium	Selenium
Chromium	Silver
Cobalt	Sodium
Copper	Thallium
Iron	Vanadium
	Zinc

1.4 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 A representative 45 mL aqueous sample is digested with 5 mL of concentrated nitric acid in a Teflon digestion vessel for 20 minutes using microwave heating. The sample is transferred, with filtering if necessary, to a clean sample bottle for analysis.

3 Interferences

3.1 In order to identify problem matrices and method error, blanks, spikes, spike duplicates and check samples are run at regular intervals, as specified in each relevant analytical method.

3.2 Samples that are oily or continuously vent in the microwave shall be digested by an alternate hot plate digestion method (see prep SOP 09-3050B).

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Therefore, each chemical shall be treated as a potential health hazard.

4.2 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.

4.3 Analysts shall consult the Material Safety Data Sheets (MSDS) for each chemical used in the digestion process.

4.4 Since the chemical make-up of the samples is not known, analysts shall treat the samples with extreme caution. Proper protective equipment must be used including PVC gloves, lab coats, safety glasses and a fume hood.

4.5 Although there are many safety features built into the microwave, technicians must remember that the samples will be acidified to 10% nitric acid, heated to approximately 170°C and will be under pressures of 70 psi. All parts of the digestion vessels must be examined to ensure that there are no visible faults that could release the sample during digestion.

4.6 Analysts must be familiar with the proper assembly and use of the vessels and all related safety equipment. Information regarding the safe use of the MSD-2100 can be found in the *MSD-2100 Operation Manual* to be found in the microwave supplies drawer in the Inorganic Prep Lab.

4.7 The addition of Conc. Nitric acid (6.2) to water in Sections 7.2 and 10.7 may cause significant heat generation or a vigorous chemical reaction. The analysts must always add acid to water, not water to acid. This step must also be

performed in a fume hood.

5 Apparatus and Materials

5.1 Microwave, CEM Model MDS-2100 with advanced composite digestion vessels.

5.2 100mL graduated cylinder, Class A or Class B.

5.3 Filter Paper, Whatman #2.

5.4 Filter funnels, plastic.

5.5 8 fl-oz. Polyethylene bottles, disposable. VWR #16059-068, or equivalent.

5.6 Acid Dispensers (Finnipipettes), from Thermo capable of dispensing 0.5mL to 10mL of acid.

5.7 Thermo Finntip tips, VWR catalogue #53515-050 or equivalent.

5.8 Balance, capable of weighing to 0.01g, Mettler PM2000.

6 Reagents

6.1 Reagent Water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification System, which provides analyte-free, >16.0 megohm-cm deionized water on demand. All references to water in the method refer to reagent water unless otherwise specified.

6.2 Nitric Acid, concentrated, (HNO_3). J.T. Baker 'Baker analyzed' grade, Cat. #JT-9598-34 purchased from VWR or equivalent.

Note: Acid bottles must be contained inside Teflon carriers at all times. If an expiration date is not supplied by the manufacturer, the expiration will be five years from the date opened. This date shall be marked directly on the acid bottle.

6.3 Spiking solution Standard Mix #1-R purchased from, High Purity Standards, Cat. #SM-1339-001, or equivalent NIST certified standard. This is a pre-

mixed standard of Al, Ba, Ca, Li, Mg, and Se at 100 mg/l, As, Cd, Cr, Pb, and Sr at 10 mg/l, K and Na at 2000 mg/l, Be at 20 mg/l, and V at 5 mg/l in 5% HNO_3.

6.4 Spiking Solution Standard Mix #2 purchased from High Purity Standards, Cat. #SM-1339-002, or equivalent NIST certified standard. This is a pre-mixed standard of Bi, B, Co, Cu, Fe, Mo, and Ni at 100 mg/l, Mn, Ag, Tl, Sn, and Ti at 10 mg/l, Sb at 20 mg/l, and Zn at 50 mg/l in 5% HNO_3.

7 Glassware Cleaning

7.1 Glassware used in this procedure is to be cleaned as noted in the Glassware Washing Standard Operating Procedure (09-GLWH) for metals glassware. This procedure includes the acid washing of all glassware used for metal digestions.

7.2 Glassware used in this procedure is stored in metals prep area and rinsed with a 10% Nitric Acid (HNO_3) solution before use. Exact measurements are not necessary to produce this cleaning reagent. Production is approximately 100 mL concentrated HNO_3 into 900 mL of deionized water.

Note: Some heat will be generated from the addition of acid to water.

7.3 For determination of trace elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory glassware and apparatus and general contamination within the laboratory environment from dust, etc. A clean laboratory work area designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface adsorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable lab ware (glass, quartz, polyethylene, PTFE, FEP, etc.) shall be sufficiently clean for the task objectives.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Demonstration of Capability (DOC) – DOCs must be performed by each analyst prior to performing this method and repeated at any time there is a significant

change in instrument type. To perform DOCs, four consecutive Laboratory Fortified Blanks (LCSs), with a matrix matching that of the calibration standards, are analyzed. The recoveries obtained must be within 85-115% of the known values for each associated metal, and consecutive reads must have an RSD less than 10%. If the DOCs are outside these acceptance limits, a new set of LCS's must be digested and analyzed. This process is repeated until the DOCs are completed successfully.

8.2.1 Ongoing proficiency must be established annually as specified in the QA Plan, Technical Training.

8.3 For each analytical batch of samples processed, a method blank shall be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated. A method blank is a 45 mL aliquot of reagent water digested as a sample.

Note: The method blank concentration must be <1/2 the reporting limit for USACE / DOD samples. If the blank recoveries are outside this acceptance limit, the samples associated with the batch will need to be redigested in a new batch.

8.4 Spiked samples or standard reference materials shall be employed to determine accuracy. A spike and spike duplicate sample must be prepared for every twenty (20) samples.

Note: Some client contracts (ex. DOD) require the preparation of matrix spikes for every new matrix received. The Horizon LIMS will designate that these samples are to be prep spiked. The 1 per 20 batch spiking rule will not always be applicable to these samples. If the LIMS designates a sample MS/MSD, it is not to be disregarded by the analyst.

8.4.1 Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.4.2 Samples to be spiked for (ICP) analysis shall be spiked in the following manner. Using a Finnipipette (5.6), add 500 µL of Standard Mix #1-R (6.3) and 500 µL of Standard Mix #2 (6.4), to the samples before the

addition of Nitric Acid. Record spike amounts and lot #'s used in the digestion logbook.

Note**:** The final volume for samples spiked in this manner will be 51 mL.

8.5 A Laboratory Fortified Blank / Laboratory Control Sample (LCS) is to be run minimally every 20 samples or at least once on each 8-hour shift. The blank spike is a 45 mL aliquot of reagent water spiked as noted in Section 8.4.2. Special projects may require more frequent analysis of the LCS.

Note: The final volume for LCS's spiked in this manner will be 51 mL.

8.6 The expiration date of all standards and reagents shall be labeled on their respective containers. If an expiration date is not supplied by the manufacturer, the default expiration date shall be one year from the date opened.

8.7 The ALSI Lab I.D. number of all mechanical pipettes used in this digestion process must be documented in the prep logbook.

8.8 All mechanical pipettes used in this digestion process must calibrated annually to verify the ***entire*** range of use. Select four to five settings throughout the pipette range, and perform a full calibration at each setting. This calibration data must be recorded in the metals autopipette calibration logbook. (See 09-AP SOP for calibration procedure.)

8.8.1 A single setting pipette calibration is also required on a weekly basis to verify dispensing volumes. Select a mid-range volume to perform this calibration, and be sure to document the procedure in the metals autopipette logbook. (See 09-AP SOP for calibration procedure.)

8.9 The Mettler balance used to weigh the digestion vessels must be calibrated each day. Prior to use, the analyst must verify that daily calibration has been recorded in the appropriate Balance Calibration Logbook. The procedure for calibrating the balance can be found in SOP 09-PM2000.

8.10 All QC spiking solutions used in this method must be stored in area separate from samples and sample digestates. If the spiking solutions are going to be kept in the same room as samples, then a separate drawer or enclosed cabinet

must be designated for their storage.

Quality Control Requirements
(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Corrective Action
Method Blank (LRB)	--	One per batch of 20 samples or less.	Dependent upon analysis results. Samples in the batch must be < the reporting limit or ≥ 10X the method blank. For batches involving samples from USACE / DOD the method blank recovery must be <1/2 the reporting limit. If not, the samples associated with the blank must be redigested.
Laboratory Fortified Blank (LFB or LCS)	See Sections 8.4 & 8.5	One per 20 samples or at least once per 8-hour shift.	Dependent upon analysis results. If LCS recoveries remain outside acceptable limits of 80-120% after reanalysis, the samples associated with it must be redigested and reanalyzed.
* Matrix Spike (MS)	See Sections 8.4 & 8.5	One per batch of no more than 20 samples.	The matrix spike recovery must be within 75-125% of the expected value. If the spike fails, a post digestion spike is performed at the time of analysis, and a comment is added to the client's lab report. The metals analyst will add these comments when the sample results are entered.
*Duplicate or matrix spike duplicate (MSD)	For MSD see sections 8.4 & 8.5	One MSD per batch of no more than 20 samples. Sample duplicates are performed only when requested by the client.	The Relative Percent Difference (RPD) between the Spike and Spike duplicate must be within 20%. If the RPD is above 20%, a comment must be added to the report.

Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.11 MDL studies must be performed according to SOP 99-MDL or the reference methods, whichever is more frequent.

9 Sample Collection, Preservation and Handling

9.1 Both plastic and glass containers are suitable.

9.2 Aqueous waste waters must be acidified to a pH of <2 with HNO_3 at the time of sampling as a means of preservation.

9.3 Samples and sample digestates must be stored separately from prep spiking solutions.

10 Procedure

10.1 Any apparatus coming into contact with the sample (containers, graduated cylinders, filter paper, filter funnels, etc) must be rinsed with both 10% HNO_3 and reagent water. Also, the graduated cylinder used to measure out samples shall be rinsed at least twice with both 10% HNO_3 and reagent water in between transferring each sample.

10.2 Section 10.1.4 of EPA Method 3015A states that a microwave power check shall be periodically performed to verify the integrity of the calibration. Check to make sure that the power check has been performed on a weekly basis. If not, perform the microwave power check as specified in the Microwave Power Check SOP (09-PWRCK). According to the EPA Method, a partial power setting, obtained from the three-point calibration, shall be used for the power check. The wattage obtained from the power check shall be ± 10 w from the selected partial power setting. If the wattage is outside allowable limits, repeat the power check. If the wattage remains outside acceptable limits, the entire calibration needs to be re-evaluated. If the wattage is within allowable limits, the multiple point calibration remains valid, and the digestion process can continue.

Note: A service contract for the microwave, CEM Model MDS-2100, is maintained with the manufacturer. As per that agreement, a service technician performs a calibration twice a year. A certificate of calibration is provided and stored in the metals prep area.

10.3 Assemble, according to the manufacturer's instruction, a microwave tray with nine of each of the following: advanced composite sleeves, thread rings, and Teflon liners. These are to be placed in positions 1,2,3,5,6,7,9,10, and 11 as labeled on the carrousels. Counter balances shall be used if necessary so as to evenly distribute the weight on the turntable.

10.4 Using a graduated cylinder, Class A or Class B, transfer a 45 mL representative aliquot of the well-mixed sample to a digestion vessel. **Note:** The number of the digestion vessels must be documented with COC# of the sample it contains in the microwave digestion logbook. This is necessary because there is no place to record the sample number on the digestion vessel itself. Also, record the sample volume used in the "sample amount" column of the microwave digestion logbook. (App. A)

10.5 Using the method explained in Section 10.3, fill the rest of the tray including the blank which is a 45 mL aliquot of reagent water. Note: If fewer than nine (9) samples are to be digested, fill out the rest of the tray with vessels containing 45 mL of deionized water and 5 mL of nitric acid to balance the energy inside the cavity of the microwave.

10.6 If any of the samples are to be spiked, add the spike solutions at this time as described in Section 8.4. Record spike amounts and lot numbers in the designated columns in the microwave digestion logbook.

10.7 Using the Finnipipette acid dispenser from Thermo, add 5 mL of concentrated nitric acid to each digestion vessel. This step shall be performed in a fume hood because of the possibility of a vigorous reaction between the sample and concentrated nitric acid. If a vigorous reaction does occur, allow the sample to cool to room temperature before capping the vessel.

10.8 Place a cover and cap on each of the vessels remembering to include a pressure regulating cover on one of the samples. Tighten the cap just hand tight. The threads will expand during heating in the microwave.

10.9 Using an analytical balance, weigh each digestion liner/cover/cap assembly to the nearest 0.01g and record this weight in the **"**initial weight of vessel" column of the microwave digestion logbook.

10.10 Assemble the overflow reservoir making sure that a vent tube from the reservoir is connected to each digestion vessel.

10.11 Place an **OPEN** transparent valve on the pressure control cover.

10.12 Place the tray in the microwave cavity making sure to connect the pressure line to the transparent valve on the tray.

Note: The pressure line must be purged of any air bubbles in order to accurately read the pressure of the sample. This may require filling the syringe with deionized water.

10.13 Recall the stored program (3015) from the stored methods menu. Review the method to make sure that changes were not inadvertently made to the program. The program calls for the samples to reach a pressure of 70 psi in less than ten minutes and then hold that pressure for 10 additional minutes.

10.13.1 From information received from the manufacturer of the microwave, the pressure of 70 psi is said to place the samples in the correct temperature range specified by Method 3015. That is, the samples will be brought to with ± 4 degrees of 160ºC within the first ten minutes, and allowed to slowly rise to 165ºC-170ºC for the second ten minutes.

10.14 Verify that the fume hood is turned on and start the program.

10.15 If the tray of samples has reached 70 psi in the specified time frame, a reversed type “TAP P” will be displayed in the microwave’s LCD.

10.16 If the “TAP P” is not displayed when the program is completed, the batch will need to be cooled to room temperature and the batch redone with a fewer number of vessels per tray.

10.17 If the “TAP P” is displayed, close the transparent value in the pressure monitored sample, and remove the tray from the microwave, and place in a fume hood to cool.

10.18 After the vessels have cooled to room temperature, remove the vent tubes from the covers and the transparent valve from the pressure control cover.

10.19 Reweigh the vessels and record the weights in the “final weight of vessel” column of the microwave digestion logbook.

10.19.1 If the difference in weights from the initial weight of vessel to the final weight of vessel is greater than 5g, the sample must be discarded and redigested. This indicates a loss of sample through the rupture membrane. Re-evaluate the sample and

consider digesting by the appropriate hot plate method if the sample cannot be digested in the microwave.

10.19.2 If the difference in weights from the initial weight of the vessel to the final weight of the vessels is less than or equal to 5g, the "final volume" column can be filled out in the microwave digestion logbook. The final volume is the total volume of sample plus nitric acid plus any spike solutions added. (See Sections 8.4 and 8.5 for final volumes of spiked samples.)

10.20 Carefully uncap and vent each sample in a fume hood.

10.21 Transfer the sample into an 8 fl-oz. Polyethylene digestion bottle that has been pre-rinsed with 10% Nitric Acid and D.I water. Label the bottle with sample number, digestion type, method number, preparation factors, and batch number.

10.22 If the sample contains particulates that may be large enough to clog the nebulizer, filtration of the sample through a 10% HNO_3 and reagent water rinsed Whatman #2 filter paper will be necessary. Do not filter with any additional amount of reagent water, as this will change the preparation factors for the sample. The filtration may be performed directly into a digestion bottle that has been rinsed and labeled as noted in Section 10.21.

11 Calculations

11.1 Not applicable to this method.

12 Reporting Results

12.1 Verify that all required information has been entered into the digestion logbook.

12.2 Open the production Horizon LIMS, and go to "batch posting" under the Operations TAB. Enter the Horizon Batch Number (HBN), which corresponds to MDIG digestion batch. When the batch opens, the samples will already have default initial volumes of 45 mL and final digestate volumes of 50 mL. These volumes will need to be adjusted to match the volumes in the microwave digestion logbook. Remember that the final digestate volume for all spiked samples is 51 mL.

12.3 Once the sample prep factors have been updated, the analyst initials and final prep date and time need to be entered in their appropriate fields.

12.4 When entering data into Horizon LIMS do not round off results; Horizon will automatically perform rounding appropriate to the method.

12.5 Double check that all information entered in steps 12.1 through 12.3 is correct, have information reviewed by second analyst, and hit the save TAP in the upper left corner of the Horizon page. Once the batch is saved, the prep factors are applied to the samples, the META analysis code becomes available for the Metals Dept.)

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this

method.

Appendix A

Microwave Digestion Logbook

EPA Method: 3015	Date of Microwave Power Check:_____					Date	Time	Tech	
ALSI SOP: 09-3015	Date of Microwave Calibration:_____				Start	_____	_____	_____	
Microwave ID Number:ZR8053	Was pressure reached in	YES: _____			Finish	_____	_____	_____	
Batch Number:_____	time required by method?	NO: _____							
Batch Number:_____	T - TCLP	M - 6020							
Batch Number:_____	S - SPLP	W - 6010B							
Batch Number:_____	A - ASTM								

Position #	Sample #	Initial Weight Of Vessel (g)	Final Weight Of Vessel (g)	Spike Amount (ul of each)	Spike Lot #	Sample Amount (45 ml)	Final Volume (50 ml)	Batch Type	Pipette ID #	QC Type
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										
21										
22										
23										
24										
25										
26										

	Comments:	1.) Concentrated HNO_3 Lot #_____
		2.)All TCLP matrix spikes are spiked prior to preservation as noted in the TCLP logbook.

SOP Change History Sheet

Section No.	Section	Reason for Change
3.1	Interferences	SOP Update 05/1/205
3.2	Interferences	SOP Update 05/12/05
4.6	Safety	SOP Update 05/12/05
5.5	Apparatus and Materials	DOD Audit Response 05/12/05
5.6	Apparatus and Materials	DOD Audit Response 05/12/05
6.2	Reagents	SOP Update 05/12/05
6.3	Reagents	SOP Update 05/12/05
6.4	Reagents	SOP Update 05/12/05
8.2	Quality Control	SOP Update 05/12/05
8.3	Quality Control	SOP Update 05/12/05
8.4	Quality Control	DOD Audit Response 05/12/05
8.4.1	Quality Control	SOP Update 05/12/05
8.4.2	Quality Control	SOP Update 05/12/05
8.5	Quality Control	SOP Update 05/12/05
8.7	Quality Control	A2AL Audit Response 04/21/05
8.8.	Quality Control	A2LA Audit Response 04/21/05
8.8.1	Quality Control	A2LA Audit Response 04/21/05

SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
8.9	Quality Control	DOD Audit Response 05/12/05
8.10	Quality Control	DOD Audit Response 05/12/05
10.1	Procedure	SOP Update 05/12/05
10.3	Procedure	SOP Update 05/12/05
10.4	Procedure	SOP Update 05/12/05
10.6	Procedure	SOP Update 05/12/05
10.7	Procedure	SOP Update 05/12/05
10.13.1	Procedure	SOP Update 05/12/05
10.14	Procedure	SOP Update 05/12/05
10.17	Procedure	SOP Update 05/12/05
10.21	Procedure	SOP Update 05/12/05
12.1	Reporting Results	SOP Update 04/21/05
12.2	Reporting Results	SOP Update 05/12/05
12.3	Reporting Results	SOP Update 05/12/05
12.4	Reporting Results	SOP Update 05/12/05
Revision 7: 2/20/2006		
1.4	Scope and Application	Added as per DoD audit
2.1	Summary of Method	Added time parameters

SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
4.2	Safety	Added statements of MSDS availability
6.2	Reagents	Revised expiration dating
7.2	Glassware Cleaning	Added storage instructions
7.3	Glassware Cleaning	Added contamination considerations
8.2.1	Quality Control	Added ongoing DOC proficiency
8.11	Quality Control	MDL performance frequency
10.2	Procedure	Note added about microwave service contract
12.4	Reporting Results	Data entry instructions
12.5	Reporting Results	Review by second analyst
16	Troubleshooting	Section added as per DoD audit

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

Certifications and Accreditations for Analytical Laboratory Services, Inc.

Certification/ Accreditation	*Issuing Agency*	*Cert. Number*	*Contact Name and Number*	*Scope of Certification*	*Expiration*
State of Connecticut	Department of Public Health	PH-0224	Dermot Jones (860)509-7389	Drinking Water, Wastewater, Sewage/Effluent, Soil - inorganic and organic Also Misc. Phase II and V SOCs	12/31/2007
State of Delaware	Division of Air and Waste Management	ID 11	Brenda Haire (302)741-8630	Approved by the Department to perform analytical work at sites being investigated under HSCA, the VCP, or the Brownfield Program	1/31/2008
State of Georgia	Department of Natural Resources	914	Loretta Lambert (404)651-5164	Drinking Water - Inorganics, Organics	1/31/2008
State of Maryland	Department of Health and Mental Hygiene Laboratories Administration	128	Mary E.T. Stancovage (410)537-3738	Drinking Water - Microbiology, Inorganics, Organics	3/31/2007
National Environmental Laboratory Accreditation Program	New Jersey Department of Environmental Protection and Energy; Office of Quality Assurance	77010	Debra Waller (609)984-7732	Drinking Water, Wastewater, and Solid/Haz Waste - Chemistry, Metals, Organics	6/30/2007
National Environmental Laboratory Accreditation Program	New York State Department of Health	11759	Dan Dickenson (518)-485-5570	Environmental Analyses Potable Water Environmental Analyses Solid and Hazardous Waste	4/1/2007
National Environmental Laboratory Accreditation Program	Pennsylvania Department of Environmental Protection; Office of Management and Technical Services; Bureau of Laboratories	22-293	Bethany Piper (717)346-8214	Drinking Water - Microbiology, Inorganics, Organics Wastewater - Microbiology, Inorganics, Organics Solid & Hazardous Waste - Microbiology, Inorganics, Organics	1/31/2008
State of Tennessee	Department of Health	2847	Dr. Ruth Chen (615)532-0881	Drinking Water - Inorganics, Organics	1/31/2008
Commonwealth of Virginia	Department of General Services; Division of Consolidated Laboratory Services	421	Tracey Hunter (804)786-3411	Drinking Water - Microbiology, Inorganics, Organics	6/30/2007
West Virginia Department of Environmental Protection	Division of Water and Waste Management	343	David Wolfe (304)472-5124	Limited Chemistry, Metals, Organics	7/31/2007
A2LA - American Association for Laboratory Accreditation	American Association for Laboratory Accreditation	0818.01	Randy Querry (301) 644-3248	Potable Water, Nonpotable Water, Solid/Hazardous Waste	4/30/2007
Environmental Lead Proficiency Analytical Testing Program (ELPAT)	American Association for Laboratory Accreditation	0818.01	Randy Querry (301) 644-3248	Environmental Lead (Pb) Testing Laboratory Accreditation Program	4/30/2007
USEPA Region 8	Wyoming & Region 8 Tribal Systems	8TMS-Q	Tony Medrano (303) 312-6984	Drinking Water -- Microbiology, Inorganics, Organics	9/12/2007
USACE - Northwest Division	Formal program discontinued. Approval granted by individual districts per project.	NA	NA	Water and Soil - VOCs by 8260, SVOCs by 8270, PCBs by 8082, Herbicides by 8141/8151, TAL Metals, Anions by 300/9056, Pesticides by 8081, GRO/DRO by 8015, Cyanide by 9012/9014, Explosives by 8330, Halogenated/Aromatic VOCs by 8021, and Perchlorate by 314; Oil - PCBs by 8082	*Approval on a project basis following audit evaluation.
NAVY (NFESC)	Department of the Navy; Navy Facilities Engineering Service Center	NA	Pati Moreno (805) 982-1659	Water and Soil - VOCs by 8260, SVOCs by 8270, PCBs by 8082, Herbicides by 8141/8151, TAL Metals, Anions by 300/9056, Pesticides by 8081, GRO/DRO by 8015, Cyanide by 9012/9014, Explosives by 8330, Halogenated/Aromatic VOCs by 8021, and Perchlorate by 314; Oil - PCBs by 8082	12/1/2007

NA: Not Applicable

The USACE program was replaced by the laboratory's compliance with the DOD QSM Version 3 and NELAC programs.

Rev. 2/22/2007

Appendix B: Figures

Figure 7- 1: Inorganic Analysis By Inductively Coupled Plasma (ICP) Methods 6010

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method	QC acceptance criteria published by DoD, if available; otherwise method specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	Every 3 months	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear range or high-level calibration check standard (ICP only)	Every 6 months	Within ± 10% of expected value	NA	NA	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes (ICAL) (ICP: minimum one high standard and a blank; GFAA: minimum three standards and a blank; CVAA: minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case r ≥ 0.995.	Correct problem and repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration, prior to sample analysis	All analyte(s) within ± 10% of expected value	Correct problem and verify second source standard. If that fails, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	ICP: within ± 10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration.	Flagging criteria is not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±30% of expected value	Correct problem, then reanalyze.	Flagging criteria is not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Method blank	One per preparatory batch	No analytes detected ≥ ½ RL For common laboratory contaminants, no analytes detected $ RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected ≥ MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria is not appropriate.	No samples may be analyzed without a valid ICS.
LCS containing all analytes required to be reported by the project or contract	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch (see full explanation in Appendix DoDD).	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	ICP: Perform post-digestion spike (PDS) addition.	Flagging criteria is not appropriate.	Only applicable for samples with concentrations > 50 x MDL (ICP).
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the postdigestion spike addition.	The spike addition should produce a level between 10 and 100 x MDL.
MS	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

Figure 7- 2: Organic Analysis By Gas Chromatography And High Performance Liquid Chromatography (Method 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method	QC acceptance criteria published by DoD, if available; otherwise method specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	Not applicable (NA)	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-12)	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL.
Retention time window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	Width is ± 3 times standard deviation for each analyte retention time from 72-hour study.	NA	NA	
Breakdown check (Endrin/ DDT Method 8081A only	Daily prior to analysis of samples	Degradation < 15% for both Endrin and DDT.	Correct problem then repeat breakdown check.	Flagging criteria is not appropriate.	No samples shall be run until degradation < 15%.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum fivepoint initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below: Option 1: RSD for each analyte < 20% Option 2: Grand mean2 RSD < 20%, with no individual analyte RSD > 30% Option 3: linear – least squares regression: r > 0.995 Option 4: non-linear regression: coefficient of determination (COD) r2 ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration.	Apply J to all analytes with RSD > 20% and ≤ 30%. Identify in case narrative analytes with RSD > 20%, provide to client the actual RSD for those analytes, and document the grand mean.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. If that fails then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL	The center of the retention time window shall be set at midpoint of initial calibration curve.	NA	NA	
Retention time window verification for each analyte and surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria is not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without a verified retention time window at the initial verification.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ± 15% of expected value (%D), or grand mean ≤ 15%D with no %drift/difference for any individual analyte > 20%D	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. See section 9.4.2.2.e and box 41. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification	Identify in case narrative analytes with %D>15%, provide to client the actual %D for those analytes, and document the grand mean. ICV: Apply J to all results associated with the analytical batch for analyte(s) > 15% and < 20% of expected range. CCV: Apply Q to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	If an individual analyte is > 20% or the grand mean is > 15%, no samples may be analyzed until the problem has been corrected.
Method blank	One per preparatory batch	No analytes detected ≥ ½ RL. For common laboratory contaminants, no analytes detected > RL.	Correct problem, then see criteria in box D-4; if required, reprep then reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory control sample (LCS) containing all analytes required to be reported by the project or contract	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D)	If corrective action fails apply Q to specific analyte(s) in all samples in the associated preparatory batch	
Matrix spike (MS)	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error
Matrix spike duplicate (MSD) or sample duplicate	One per every 20 project samples per matrix	RPD $\leq$ 30% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike (analytes identified in Appendix DoDD)	All field and QC samples	QC acceptance criteria for LCS specified by DoD, if available; otherwise method specified criteria or laboratory's own in-house criteria	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met. For QC samples, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be Confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA	Apply J if RPD > 40% from primary column result or Qflag if sample is not confirmed. Discuss in the case narrative.	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the noneffected result and document in the case narrative.
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL.	

Figure 7- 3: Common Anions Analysis (Method 9058)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method	QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is completed.
MDL study	At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL.
Retention time window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change)	Width is ± 3 times standard deviation for each analyte retention time over 24-hour period	NA	NA	
Multipoint calibration for all analytes (minimum three standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression.	Correct problem, then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No sample may be run until calibration has passed.
Second source calibration verification	Once after each multipoint calibration	Value of second source for all analytes within ± 10% of expected value (initial source).	Correct problem and verify second source standard. If that fails, then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishmentfor each analyte	Once per multipoint calibration	Position shall be at midpoint of calibration curve.	NA	NA	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window verification for each analyte	Each calibration verification	Analyte within established window.	Correct problem, then reanalyze all samples analyzed since the last retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria is not appropriate.	No samples shall be run without a verified retention time window.
Initial calibration verification (ICV)	Daily before sample analysis; and when eluent is changed, and with every batch of samples	All analytes within ± 25% of expected value and retention times within appropriate windows	Correct problem, rerun ICV. If that fails, then repeat initial calibration (see section 9.4.2.2.e and box #41).	Flagging criteria is not appropriate.	No samples may be run without verifying initial calibration.
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence	Instrument response within ± 15% of expected value	Correct problem, then repeat continuing calibration verification and reanalyze all samples since last successful calibration verification	Apply Q to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	
Method blank	One per preparatory batch	No analytes detected ≥ ½ RL For common laboratory contaminants, no analysis detected $ RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
LCS containing all analytes required to be reported by the project or contract	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated prepatory batch, if sufficient sample material is available.	If corrective action fails apply Q to specific analyte(s) in all samples in the associated preparatory batch.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MS	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD	One per every 20 project samples per matrix	RPD ≤ 20% (between MS and MSD)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample Duplicate (replicate)	One per every 10 samples	%D ≤ 10%	Correct problem and reanalyze sample and duplicate.	If corrective action fails, apply Q to specific analyte(s) in the sample.	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL.	
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see DOD Quality Systems Manual , Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see DOD Quality Systems Manual, section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Detection Limit (MDL) study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see DOD Quality Systems Manual box D-18).	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times the instrument noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see DOD Quality Systems Manual box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study	At initial set-up and after significant change	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Tuning	Prior to initial calibration	Mass calibration ≤ 0.1 amu from true value; Resolution <0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate analytes	Retune instrument then reanalyze tuning solutions.	Flagging criteria is not appropriate	No analysis shall be performed without a valid MS tune.
Initial calibration for all analytes (ICAL) **(minimum one high standard and a blank)**	Initial calibration prior to sample analysis	If more than one standard is used, in which case r ≥ 0.995.	Correct problem and repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration, prior to sample analysis	Value of second source for all analytes within ± 10% of expected value (initial source)	Correct problem and verify second source standard. If that fails, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Low level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±20% of expected value	Correct problem, then reanalyze.	Flagging criteria is not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ± 10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration.	Flagging criteria is not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Liner dynamic range or high-level check standard	Every 6 months	Within ± 10% of expected value	NA	NA	
Method blank	One per preparatory batch	No analytes detected ≥ ½ RL For common laboratory contaminants, no analytes detected $ RL	Correct problem, then see criteria in DoD Quality Systems Manual, box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICSAB)	At the beginning of an analytical run	ICS-A: Absolute value of conc. For all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all affected samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see DoD Quality Systems Manual, box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes. If sufficient sample material is available. (See full explanation in Appendix DoD-D).	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test	Each preparatory batch	Five-fold dilution must agree within ± 10% of the original determination	Perform post-digestion spike addition.	Flagging criteria is not appropriate.	Only applicable for samples with concentrations > 100 x MDL.
Post-digestion spike addition	When dilution test fails or analyte concentration in all samples < 100 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	.
Method of standard additions (MSA)	When matrix interference is suspected	NA	NA	NA	Document use in the case narrative.
MS	One MS per every 20 project samples per matrix (see DoD Quality Systems Manual, box D-15).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards (IS)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in Method 6020 (8.3)	Flagging criteria is not appropriate	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

Figure 7- 4: Quality Control Field Audit Report

SUMMARY INFORMATION

1. PROJECT NAME: _____

2. PROJECT ADDRESS: _____

_____

_____

3. PRELIMINARY ASSESSMENT _____ RI/FS _____ RD _____ CONSTRUCTION_____

OTHER _____

4. DATE(S) OF QC FIELD AUDIT _____

5. AUDITOR'S NAME _____PHONE_____

6. FACILITY CONTACT _____PHONE_____

7. CONTRACTOR CONTACT_____PHONE_____

8. PERSONNEL ON-SITE

NAME	REPRESENTING	PHONE

9. AUDITOR'S COMMENTS

_____

_____

_____

_____

10. WEATHER CONDITIONS

SUNNY ; PARTLY SUNNY ; PARTLY CLOUDY ; CLOUDY ; RAIN ; DRIZZLE ; SNOW ; SLEET

TEMPERATURE_____ WIND SPEED_____ WIND DIRECTION_____

11. LEVEL OF PERSONNEL PROTECTION REQUIRED IN WORK PLAN

A B C D

LEVEL OF PERSONNEL PROTECTION ACTUALLY DONNED:

A B C D

12. FIELD SURVEY EQUIPMENT

INSTRUMENT	MODEL	CALIBRATION CHECK	CALIBRATION STANDARD	SPAN SETTING
CONDUCTIVITY METER	_____	_____	_____	_____
DISSOLVED O_2 METER	_____	_____	_____	_____
PH METER	_____	_____	_____	_____
COMBUSTIBLE GAS INDICATOR (LEL/O_2)	_____	_____	_____	_____
FLAME IONIZATION DETECTOR (OVA)	_____	_____	_____	_____
PHOTOIONIZATION DETECTOR (HNU)	_____	_____	_____	_____
TOTAL GAS INDICATOR (CO,H_2S)	_____	_____	_____	_____
OTHER	_____	_____	_____	_____

OBSERVATIONS _____

_____

_____

13. DID THE SAMPLING TEAM TAKE PERIODIC SURVEYS OF THE AMBIENT AIR CONDITIONS?

YES NO N/A

14. DID THE SAMPLING TEAM PROVIDE A DECON ZONE DESIGNATING CLEAN AND CONTAMINATED AREAS?

YES NO N/A

15. WERE PHOTOGRAPHS TAKEN? YES NO

16. AUDITOR'S COMMENTS

_____

_____

_____

MONITORING WELL SAMPLING SETUP AND EVACUATION

EVACUATION PROCEDURES

1. WELL CASING CONSTRUCTION STAINLESS STEEL TEFLON PVC OTHER _____

2. DIAMETER OF WELL CASING 2" 4" 6" OTHER _____

3. LOCKING CAPS ON THE WELLS? YES NO N/A PROTECTIVE CASING? YES NO N/A

4. METHOD UTILIZED TO DETERMINE THE STATIC WATER LEVEL

WATER LEVEL INDICATOR OTHER _____

5. REFERENCE POINT THAT THE STATIC WATER LEVEL WAS MEASURED FROM:

SURVEY POINT	TOP OF INNER CASING	TOP OF PROTECTIVE CASING	HEIGHT OF CASING ABOVE GROUND SURFACE

6. WAS THE WATER LEVEL INDICATOR DECONTAMINATED ACCORDING TO STANDARD PROCEDURES BETWEEN EACH WEL
YES NO N/A

IF NO, METHOD USED: _____

_____

_____

7. EVACUATION METHOD:

BAILER CENTRIFUGAL PUMP PERISTALTIC PUMP BLADDER PUMP SUBMERSIBLE PUMP

GAS DISPLACEMENT PUMP GAS LIFT PUMP OTHER _____

8. TYPE OF HOSE UTILIZED:

POLYETHYLENE TEFLON SILASTIC N/A OTHER _____

9. WAS THE HOSE DEDICATED TO EACH WELL LOCATION? YES NO N/A

IF NO, METHOD OF DECONTAMINATION _____

10. WAS THE PUMP DEDICATED TO EACH WELL LOCATION? YES NO N/A

11. WAS THE PUMP: LABORATORY DECONTAMINATED? FIELD DECONTAMINATED? N/A

12. WAS THE PUMP DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION _____

13. WAS THE PUMP HEAD OR END OF HOSE WITHIN 6 FEET OF THE DYNAMIC WATER LEVEL DURING EVACUATION?
YES NO N/A

14. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION?

YES NO N/A

15. AUDITOR'S COMMENTS

_____

_____

_____

AQUEOUS SAMPLING PROCEDURES

1. AQUEOUS MATRIX SAMPLED:

POTABLE WELL GROUND WATER SURFACE WATER LEACHATE RUNOFF STORM SEWER

SANITARY SEWER OTHER _____

2. TYPE OF SAMPLE: GRAB COMPOSITE IF COMPOSITE - SAMPLES/COMPOSITE _____

3. WAS THE VOA SAMPLE COLLECTED FIRST? YES NO N/A

4. TYPE OF SAMPLING EQUIPMENT:

	MATERIAL OF CONSTRUCTION			
	STAINLESS STEEL	TEFLON	GLASS	OTHER
BAILER	_____	_____	_____	_____
BLADDER PUMP	_____	_____	_____	_____
SAMPLER	_____	_____	_____	_____
COLIWASA	_____	_____	_____	_____
KEMMERER DEPTH SAMPLER	_____	_____	_____	_____
WHEATON DIP SAMPLER	_____	_____	_____	_____
TUB SAMPLER	_____	_____	_____	_____
BACON BOMB	_____	_____	_____	_____

5. TYPE OF LEADER LINE THAT COMES IN CONTACT WITH THE WELL WATER:

TEFLON TEFLON COATED STAINLESS STEEL N/A OTHER _____

6. LENGTH OF THE LEADER LINE _____

7. WAS THE SAMPLING EQUIPMENT DEDICATED? YES _____ NO _____

8. WAS THE SAMPLING EQUIPMENT: LAB DECONTAMINATED? FIELD DECONTAMINATED?

9. WAS THE SAMPLING EQUIPMENT DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION: _____

10. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION?

YES NO

11. ARE DISPOSABLE GLOVES WORN AND CHANGED BETWEEN EACH SAMPLE LOCATION? YES NO

12. AUDITOR'S COMMENTS:

_____

_____

_____

QA/QC INFORMATION

1. LABORATORY:

NAME _____ PHONE _____

CONTACT PERSON _____

CLP CLP CAPABLE CERTIFIED OTHER _____

3. SAMPLE INFORMATION:

MATRIX	PARAMETER	PRESERVATIVE	CONTAINER DESCRIPTION
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

3. WHAT ORDER BY ANALYTICAL PARAMETER ARE SAMPLES COLLECTED: _____

_____

4. FIELD BLANKS: YES NO N/A FREQUENCY _____

METHOD: _____

WAS IDENTICAL BOTTLE TO BOTTLE TRANSFER OF WATER UTILIZED? YES NO

5. TRIP BLANKS: YES NO N/A FREQUENCY _____

6. WHAT WAS THE SOURCE OF THE BLANK WATER? LABORATORY DEMONSTRATED ANALYTE-FREE
OTHER_____

7. SAMPLE PACKAGING AND HANDLING:

SAMPLE CONTAINERS LABELED	YES	NO	N/A
COC FORMS COMPLETED	YES	NO	N/A
CUSTODY SEALS	YES	NO	N/A
SAMPLES PRESERVED TO 4°C:	YES	NO	N/A

8. AUDITOR'S COMMENTS

_____

_____

_____

_____

Figure 7- 5: Nonconformance and Corrective Action Report

Date _____

NCR No. _____

Description of Nonconformance and Cause _____

_____

_____

_____

_____

_____

Proposed Disposition _____

_____

_____

_____

_____

_____

Submitted by: _____ Date: _____

Approved by: _____

DISPOSITION (by Project Manager or Designee)

Implementation of Disposition Assigned to: _____

Actual Disposition _____

_____

_____

_____

_____

_____

Disposition completed on: _____

Date

_____

Signature

VERIFICATION

Disposition reviewed and work inspected by: _____ on _____

Disposition verified by: _____ on _____

(Use additional sheet or memo if necessary)

Figure 10- 1: Daily Quality Control Report

MMRP: (Installation name)
DAILY QUALITY CONTROL REPORT

DAY	S	M	T	W	TH	F	S

USACE PROJECT MGR. _____

PROJECT _____

JOB NO. _____

CONTRACT NO. _____

WEATHER	BRIGHT SUN	CLEAR	OVERCAST	RAIN	SNOW
TEMPERATURE	< 32	32 - 50	50 - 70	70-85	>85
WIND	STILL	MODERATE	HIGH	REPORT NO.	
HUMIDITY	DRY	MODERATE	HUMID		

SUBCONTRACTORS ON-SITE:
EQUIPMENT ON SITE:
WORK PERFORMED (INCLUDING SAMPLING):
QUALITY CONTROL ACTIVITIES (INCLUDING FIELD CALIBRATIONS):
HEALTH AND SAFETY LEVELS AND ACTIVITIES:
PROBLEMS ENCOUNTERED/CORRECTIVE ACTION TAKEN:
SPECIAL NOTES:
TOMORROW'S EXPECTATIONS:

BY _____ TITLE _____

Appendix C: ERIS Database Format Example (One Sample One Analyte)

```
                                        eris file.txt
PREPARED=27APR20020000
PREPARATION_BATCH=VSCQ
Preparation_Type=
Procedure_ID=
Procedure_Name=
Comment=

 RECORD:Result
 ANALYTE_NAME=1,2,4-Trichlorobenzene
 Analyte_Type=
 Amount_Added=
 Amount_Added_Units=
 Detection_Limit=
 Detection_Limit_Type=
 Percent_Difference=
 Percent_Recovery=
 Percent_Recovery_Limit_High=
 Percent_Recovery_Limit_Low=
 Percent_Recovery_Limit_Type=
 Quantitation_Limit=
 Quantitation_Limit_Type=
 Relative_Percent_Difference=
 Relative_Percent_Difference_Limit_High=
 Relative_Percent_Difference_Limit_Type=
 Reporting_Limit=.167
 Reporting_Limit_Type=CONTRACT REQUIRED DETECTION LIMIT
 Retention_Time=
 Retention_Time_Units=
 RESULT=.167
 RESULT_UNITS=Micrograms per Gram
 Comment=

  RECORD:Qualifier
  LAB_QUALIFIER=Not detected
```

Malcolm Pirnie, Inc.
Blossom Point Research Facility
Date: August 2006

Standard Operating Procedure

Decontaminating Field Sampling Equipment

Title: Procedure for Decontaminating Field Sampling Equipment

I. Introduction

This procedure describes the methods used to decontaminate sampling equipment and sample processing tools. The procedures specifically address equipment used to collect sediment and soil samples.

II. Definitions

DCM	Dichloromethane, organic solvent
PPE	Personal Protective Equipment

III. Equipment and Supplies

The following equipment will be used to decontaminate equipment and tools used to collect sediment and soil samples:

1. **Tap water** for initial cleaning and rinsing of equipment.
2. **De-ionized water** for final rinsing of equipment after tap water or solvent rinse.
3. **Non-phosphate detergent** (e.g. Alconox™) for cleaning equipment.
4. **Dishwashing detergent** (e.g. Joy™ which provides suds in seawater) to remove oily or organic residue.
5. **Laboratory grade alcohol** for removing water from rinsed equipment (e.g. methanol, ethanol, or iso-propyl alcohol)
6. **Organic solvent** for final cleaning of equipment (e.g. hexane, dichloromethane (DCM), methylene chloride)
7. **Personnel protective equipment (PPE)** - including disposable gloves (nitrile preferred), disposable wipes, eye wash system, first aid kit, and waterproof outerwear (if necessary).
8. **Re-sealable buckets** approved for waste collection and transportation.
9. **Squirt bottles** for water, alcohol, and solvents.
10. **Brushes** for cleaning equipment.
11. **Field notebooks, pens, pencils, and digital camera** to document decontamination procedures.

IV. Guidelines

The following equipment will be used to collect sediment cores and require decontamination:

1. **Rotary drilling rig** (truck-mounted or skid type) with all associated drilling equipment.
2. **Tripod drill** and all associated drilling equipment.
3. **Calibrated Steel Rod** to investigate the sediment type and probe the depth of unconsolidated sediments at a sampling location and to determine the length of tubing to use.
4. **Shelby tubes** conforming to thin-walled tube specifications outlined in ASTM D 1587 with a 3-inch O.D.
5. **Drill Bits** – Tri-cone roller bit and/or drag bits that deflect the circulating drilling fluid horizontally will be used to advance the bits through the overburden soils.
6. **Vibracorer** and ancillary equipment.
7. **Polycarbonate or Cellulose Acetate Butyrate (CAB) Tubing** of appropriate diameter (approximately 3.75 inch O.D. and 0.07 inch wall thickness) for use with the vibracoring apparatus.
8. **Sediment Grab Sampler** (e.g., Ponar, van Veen, Smith McIntire, or Eckman Grabs) used for surface sediment colection.

Collection of sediment, soil, and water samples for chemical analysis requires that the equipment be cleaned between sample locations to avoid sample contamination. Generally, the cleaning procedures to be followed between sample locations are as follows:

Decontaminate all sample collection tools that contact the sample as well as all bowls and mixing/distribution implements in accordance with the following procedures.

1. Rinse each item with tap water to remove mud, dirt, or other visually present material.
2. Scrub the item with a brush and soapy water, using non-phosphate detergent such as Alconox™ for non-oily residue, or a detergent (e.g. Joy™) for items with oily or other sticky organic residue.
3. Rinse the item with tap water to remove all residual soap
4. Rinse the item with de-ionized water three times
5. Rinse the item with alcohol (methanol, ethanol, iso-propyl) or acetone to remove de-ionized water
6. Rinse the item with organic solvent (e.g. hexane, DCM, methylene chloride)
7. Wrap the item(s) in aluminum foil or plastic bag to protect it until it is used.

All solvents must be captured and disposed of in appropriate, labeled, aqueous waste containers. All instruments that come into contact with the sample (i.e. syringe, ruler,

collection buckets) must be cleaned in the same manner as the sampling device. Liquids collected into the chemical waste container must be discarded in an appropriate waste stream. Staff performing decontamination procedures need to wear appropriate PPE, gloves (e.g. nitrile) and eye protection. Care must be taken in cleaning not to allow contact of cleaning solutions with clothing as much as possible. If circumstances dictate contact will occur (e.g. high pressure washing, splashing, high wind), waterproof outer clothing must be worn (e.g. foul weather gear or rain gear).

Decontamination procedures may vary depending on specific workplan specifications, and unique contaminants of concern at specific locations. The project workplan may designate collection of equipment rinse samples to document effectiveness of cleaning.

This SOP does not address radioactive decontamination, PPE for radioactive waste, or disposal of radioactive contaminated waste material.

IV. References

American Society for Testing and Materials (ASTM), 1994. Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites. Designation: D 5088 – 90.

Appendix B: Health and Safety Plan

FINAL HEALTH AND SAFETY PLAN
FORT STEWART
HINESVILLE, GEORGIA

MARCH 2007

Prepared for:

U.S. ARMY CORPS OF ENGINEERS, BALTIMORE DISTRICT
P.O. Box 1715
Baltimore, Maryland 21203-1715

Prepared by:

MALCOLM PIRNIE, INC.
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FINAL HEALTH AND SAFETY PLAN
FORT STEWART
HINESVILLE, GEORGIA

DoD Contract Number: W912DR-05-D-0004

Reviewed and Approved by:

Heather Polinsky, Vice President
Program Officer
Malcolm Pirnie, Inc.

Shelly Kolb
Project Manager
Malcolm Pirnie, Inc.

MARCH 2007

TABLE OF CONTENTS

ACRONYMS ..... **iv**

1.0 INTRODUCTION **1-1**

1.1 Scope 1-1
1.2 HASP Acceptance 1-1

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY **2-1**

2.1 Project Organization of Safety Personnel 2-1
2.2 Safety Responsibilities of Personnel 2-1
2.3 Stop Work Authority 2-4
2.4 Required On-Site Documents 2-4
2.5 Project Logs, Records, and Reports 2-5

3.0 SAFETY AND HEALTH RISK ANALYSIS **3-1**

3.1 Project Tasks 3-1
3.2 Radiological Hazards 3-1
3.3 MEC Hazards 3-1
3.4 MEC Awareness Training 3-2
3.5 General Physical/Biological Hazards 3-3
3.5.1 Heat Stress 3-3
3.5.2 Noise 3-3
3.5.3 Slip, Trip and Fall Hazards 3-4
3.6 Equipment Operation 3-4
3.6.1 Utility Avoidance (Overhead and Underground) 3-5
3.6.2 Electrical 3-5
3.6.3 Falling Objects 3-5
3.6.4 Biological Hazards 3-5
3.6.5 Trench Collapse or Cave-In 3-6
3.7 Task-Specific Hazards and Control Measures 3-7

4.0 HEALTH AND SAFETY ORIENTATION TRAINING **4-1**

4.1 Specialized Training 4-2
4.1.1 Pre-Investigation Health and Safety Briefing 4-2
4.1.2 Morning Safety Meetings 4-3
4.1.3 Hazard Communication 4-3

5.0 MEDICAL SURVEILLANCE AND EXPOSURE MONITORING **5-1**

5.1 Medical Surveillance 5-1
5.2 Heat Stress Monitoring 5-1

6.0 PERSONAL PROTECTIVE EQUIPMENT **6-1**

6.1 General Protection Levels 6-1
6.2 Required Level of Protection 6-2
6.3 Inspection of PPE 6-4
6.4 PPE Doffing Guidelines 6-4

7.0 HAZARDOUS MATERIAL MONITORING 7-1

7.1 Radiological Monitoring 7-1

8.0 SITE CONTROL MEASURES 8-1

8.1 General 8-1
8.2 Site Control 8-1
8.3 Work Zones 8-1

9.0 STANDARD OPERATING PROCEDURES FOR SAFETY 9-1

10.0 DECONTAMINATION PROCEDURES 10-1

10.1 Personnel Decontamination 10-1
10.2 Disposal Procedures 10-1
10.3 Confined Space Entry Procedures 10-1

11.0 EMERGENCY RESPONSE PLAN 11-1

11.1 Emergency Planning 11-1
11.2 Emergency Equipment 11-1
11.3 Personnel Roles, Lines of Authority and Communication 11-1
11.4 Emergency Recognition and Prevention 11-2
11.5 Adverse Weather Conditions 11-2
11.6 Emergency Medical Treatment/First Aid 11-3
11.7 Evacuation Procedures/Safe Distances 11-3
11.8 Site Security and Control 11-5
11.9 Fire or Explosion 11-5
11.10 Spill Containment Plan 11-5
11.11 Emergency Response Evaluation 11-5
11.11.1 Pre-Planning and General Procedures 11-5

12.0 RECORDKEEPING 12-1

12.1 Medical Surveillance Report 12-1
12.2 Personnel Training Records 12-1
12.3 Health and Safety Plan (HASP) 12-1
12.4 Incident Reports 12-2

13.0 NEAR MISS REPORTING 13-1

14.0 SUBCONTRACTOR REPORTING 14-1

LIST OF TABLES

Table 6-1: Summary of Level D PPE Requirements 6-3

Table 11-1: Hand Signals 11-2

LIST OF ATTACHMENTS

Attachment 1: Installation-Specific Health and Safety Addendum

ACRONYMS

Acronym	Definition
ANSI	American National Standards Institute
CIH	Corporate Industrial Hygienist
EC	Emergency Coordinator
EOD	Explosive Ordnance Disposal
FPM	Field Project Manager
FSP	Field Sampling Plan
GA	Georgia
HSD	Health and Safety Director
MC	Munitions Constituents
MEC	Munitions and Explosives of Concern
MMRP	Military Munitions Restoration Program
MPPEH	Material Potentially Posing an Explosive Hazard
MSDS	Material Safety Data Sheet
OSHA	Occupational Safety and Health Administration
PM	Project Manager
PPE	Personal Protective Equipment
RCRA	Resource Conservation and Recovery Act
ROC	Record of Changes
SI	Site Inspection
SSO	Site Safety Officer
U.S.	United States
UXO	Unexploded Ordnance
UXOSS	UXO Health and Safety Supervisor

1.0 INTRODUCTION

1.1 Scope

The Malcolm Pirnie, Inc. (Malcolm Pirnie) Health and Safety Plan (HASP) has been developed for conducting site inspections (SI), at sites having a potential for munitions and explosives of concern (MEC) and munitions constituents (MC). This plan sets forth health and safety protocols to be used by Malcolm Pirnie employees and its subcontractors during field activities under contract number W912DR-05-D-0004. All work conducted under this contract should be in conformance with this plan unless formally modified and approved by the Malcolm Pirnie UXO Health and Safety Supervisor (UXOSS) and reviewed by the Contracting Officer via a formal record of change. The intent of this plan is to ensure the health and safety of all site personnel, the general public and the environment. Although it is impossible to eliminate all risks, adherence to this plan will help minimize incidents and accidents by promoting safety while maintaining productivity.

1.2 HASP Acceptance

This HASP and supporting documents will be provided at each site considered for a SI. Site employees and official visitors will be provided with a copy of this plan for review and are responsible for reading, understanding, and signing the acceptance page found in Attachment 1**.** In addition, an Installation Specific Health and Safety Addendum will be included as the installation-specific hazards are identified, and this information will be part of the daily safety briefing. The UXOSS and potentially the Corporate Industrial Hygienist (CIH) will provide an installation-specific orientation for site workers and visitors. The Site Safety Tailgate Meeting Form, enclosed at the end of this report, will be completed for each orientation. No personnel will be required to perform any activity at the site they believe will endanger their health and safety or that of others.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 Project Organization of Safety Personnel

This program will be accomplished under the direction of the individuals identified below (or alternates) in accordance with the responsibilities assigned by their respective organizations. Specific personnel to fill these positions are included in the Site Specific HASP.

Title	Organization	Function
Corporate Health and Safety Director (HSD)	Malcolm Pirnie	Responsible to the President on all matters related to the health and safety of all Malcolm Pirnie employees and its subcontractors. Has final approval authority on HASPs and modifications recommended by the Field Project Manager.
Field Project Manager (FPM)	Malcolm Pirnie	Manages all on-site activities and responsible for maintaining a healthy work environment.
Unexploded Ordnance Health and Safety Supervisor (UXOSS)[1]	Malcolm Pirnie	Works closely with the FPM and HSD and assists with all on-site activities. Responsible for all safety related to MEC. Provides the daily tailgate safety brief, site orientation, and safe escort of non-UXO personnel.

2.2 Safety Responsibilities of Personnel

All Malcolm Pirnie and subcontracted personnel are responsible for compliance with this HASP. All on-site field personnel are expected to perform only those tasks they believe can be done safely and for which they have been adequately trained. They are responsible for taking all reasonable precautions to prevent injury to themselves and to their fellow employees; for being alert to potentially harmful situations; and for immediately reporting any accidents, near misses, and/or unsafe conditions to the HSD and UXOSS or designated field representative. Specific safety responsibilities of the safety staff are described below.

[1] Also referred to as the Site Safety Officer.

Corporate Health and Safety Director (HSD)

The HSD is responsible for development and implementation of the Programmatic HASP and for the health and safety of Malcolm Pirnie personnel assigned to the field investigation. The HSD will review and approve the HASP. Other duties of the HSD include:

- Initiating actions to provide any required initial installation-specific training;
- Being available for consult by telephone for the full duration of site activities;
- Being available to conduct on-site audits as necessary to observe the effectiveness of the HASP;
- Being available for emergencies;
- Providing on-site consultation as necessary to verify that the HASP is fully implemented;
- Being available for consultation with the FPM and the UXOSS, and the Contracting Officer regarding any modifications to the Site Specific HASP;
- Being available for consultation with the FPM to evaluate changing site conditions and to recommend changes to engineering controls, work practices and personal protective equipment (PPE);
- Being available for review of accident reports and results of daily inspections; and
- Serving as a member of the quality control staff.

Field Project Manager (FPM) – The FPM serves as the Project Manager and has responsibility and authority for directing field activities without exposing or endangering site personnel or the public. The FPM enforces safe work practices, removes unfit or unqualified personnel/visitors from the site, and verifies that machinery and mechanized equipment brought to the site have been certified safe to operate. He/she works closely with the UXOSS, and they both share emergency coordinator activities with the facility and assist with accident and incident investigations. The FPM assigns field tasks only to those on-site personnel who have received adequate instruction and training. He ensures that all site personnel understand their respective safety roles, responsibilities and recommends changes in the HASP if required due to changing site conditions.

UXO Health and Safety Supervisor (UXOSS) – The UXOSS is responsible for supervising all on-site MEC activities and has final authority on field activities involving MEC. She/he may also assist the FPM with general site safety matters. Duties include examining the support zones, work zones, and material potentially posing an explosive hazard (MPPEH) for potential

MEC; providing MEC orientation and safe escort for site personnel. He or she is also responsible for certifying that all materials are positively identified, if this can be accomplished safely, and to ensure that the area around a MEC is marked.

The UXOSS will assist other team members in interpreting and documenting health and safety related data relevant to work activities at the site. As site data are obtained and evaluated, the UXOSS may modify this HASP with approval of the HSD. The levels of personnel protection outlined in this plan may be upgraded based on such information. The levels of personal protection outlined in this plan cannot be downgraded without the approval of the HSD. The UXOSS or designee will also conduct regular on-site briefings pertaining to health and safety requirements of the project.

Both the FPM and the UXOSS report to the HSD, and they have the responsibility and the authority to develop, implement, and verify compliance with the site HASP. These persons advise on all matters related to health and safety and have the authority to stop all work if conditions are judged to be hazardous to on-site personnel or the public. The UXOSS provides the support to the FPM in the event of an emergency. The UXOSS is responsible for implementing the emergency response plan, supporting responding emergency services, and coordinating with the facility contact. He/she is responsible for conducting accident and near-miss investigations and for submitting the Accident Reports and First Aid Incident Report to the HSD within 24 hours of a significant incident or within eight hours of a serious incident. Additional duties of the FPM and the UXOSS are:

- Verifying personnel training and medical certifications;
- Regularly inspecting the site for hazardous conditions;
- Conducting and reporting accident and near-miss investigations;
- Documenting that all field personnel have read and understand the requirements set forth in the HASP, and verifying that these requirements are upheld during on-site work activities;
- Conducting daily tailgate health and safety meetings for all participants before starting a specific task;
- Arranging for and providing job safety training, as required;

- Establishing work zones, evacuation routes, and assembly areas;
- Determining whether to maintain or modify levels of protection provided in the HASP based on site conditions and monitoring data;
- Ensuring that protective clothing and equipment are properly selected, used, stored, and maintained;
- Maintaining a first aid kit and availability of a vehicle in the case of an emergency;
- Maintaining contact with the facility in the event of an imminent MEC hazard;
- Ensuring that the FPM and Project Manager are informed of any situations out of the norm that may be of concern regarding the investigation, audits, and/or reports; and
- Clearing the area prior to collection of environmental media samples.

2.3 Stop Work Authority

All employees have the right to work in a safe and healthful environment that is free from recognized hazards. Conditions or situations that are unsafe must be reported immediately to the FPM and/or the UXOSS. The FPM will evaluate the situation, in consultation with the UXOSS and the HSD, and determine which appropriate actions need to be taken to ensure a safe working environment. Work will be continued only after these actions have been implemented.

2.4 Required On-Site Documents

The following information (some of which will be included in the site specific HASP Addendum) must be available at the project site:

- Installation-specific HASP
- Emergency notifications, services, points of contact phone list and procedures
- Site Evacuation Plan (including routes)
- Site Hospital Route Map
- Material Safety Data Sheets (MSDSs), if needed
- Applicable Occupational Safety and Health Administration (OSHA) records (OSHA Forms 300 and 301)

2.5 Project Logs, Records, and Reports

The FPM (or designee) must carefully document the implementation of this HASP by maintaining the installation-specific Field Binder. The binder will contain the following documents, which shall be available for review by the facility or appropriate OSHA representative:

- Daily Employee Visitor Roster
- Daily Tailgate Safety Meeting Reports
- Supervisor's Report of Injury or Illness
- First Aid Incident Report
- Project Accident First Aid Log
- Incident Reports (for unanticipated MEC discovery, environmental incidents, equipment damage, evacuations, and near-miss events)
- Record of Changes (ROCs) to this HASP
- Signed Acceptance of HASP Form (signed by all routine on-site personnel).

3.0 SAFETY AND HEALTH RISK ANALYSIS

3.1 Project Tasks

The site specific HASP Addendum will address any additional project tasks not covered in Section 1.

3.2 Radiological Hazards

Given the extent to which radioactive material has been used in industry and government, there is always a possibility of encountering other sources of radioactive contamination. It is not anticipated that any radiological hazards will be encountered during this work. However, if any radiological contamination is suspected, work will cease immediately and both the FPM and the UXOSS will be contacted.

Radium nuclear decay emits ionizing radiation in the form of alpha particles. Alpha particles can travel a few inches in the air, but cannot penetrate the skin or other barrier. However, they can be particularly damaging if ingested or inhaled. The potential routes of entry include inhalation of contaminated dusts and ingestion of contaminated dusts from hand-to-mouth contact due to poor personal hygiene.

These techniques are employed to protect workers from ionizing radiation:

- Avoid any suspected radiation emitting devices and contact the FPM immediately.
- Limit time of exposure to radioactive materials.
- Specify safe working distances from sources.
- Shield against radioactive particles using barriers and/or PPE.

3.3 MEC Hazards

Physical hazards associated with explosive compounds and MEC are anticipated at the ranges. These include reactive/explosive residues from spotting charges or phosphoric fillers associated with practice munitions and/or MEC. For the purposes of this HASP, all explosives are termed

MEC. An UXO Technician(s) will first perform a visual MEC survey of the areas that need to be accessed by walking the site and closely observing and marking any surface MEC hazards. If non-MEC trained personnel must access an area, a safe access corridor will first be marked with flagging or pin flags or a UXO Technician will provide escort for any non-MEC trained personnel. It is critical that all personnel be briefed on both the initial identification of MEC and the steps to take if potential MEC is encountered. Specific hazards will be discussed in the tailgate safety briefing and included in the installation-specific safety orientation. MEC hazards, precautions and procedures are discussed in the Malcolm Pirnie Standard Operating Procedures for Sites Contaminated with MEC.

3.4 MEC Awareness Training

The work being conducted for the preliminary assessment of ranges does not involve MEC operations as they relate to the excavation, moving and disposal of MEC. This is solely an **Anomaly Avoidance** project; no one under any circumstances shall touch or move any MEC or items that may resemble MEC. All personnel that are not UXO Trained Technicians will remain only in those areas that are marked as safe for access or will be under escort by a trained UXO Technician. At the initial on-site training, all personnel will receive an installation-specific MEC briefing by either a Malcolm Pirnie UXO Technician or Military Explosive Ordnance Disposal (EOD) Unit before beginning any site work. The briefing will include the following:

- Type of MEC items that have been found in the past;
- Number of items that have been found at the project site and in the surrounding area;
- Emergency telephone numbers to activate the MEC/EOD team;
- Safe refuge areas that will be used to retreat from the explosive areas (The safety areas are established based on the size of the explosive item encountered to ensure that no fragmentation reaches that area);
- Specific steps to take if a worker encounters MEC (Additional MEC safety precautions and safe work practices are described in the Malcolm Pirnie Anomaly Avoidance Standard Operating Procedure)

  Step 1: Make NO attempt to touch, move, uncover, recover, or disturb the item that has been found.
  Step 2: Call out to the UXOSS on-site. Do not make any quick moves. Wait for the MEC supervisor and point to identify the object. Then slowly move away

from the object by retracing your footprints until you are again on a normally used path. Go immediately to the safe area and alert the team of the situation.
Step 3: The UXOSS will ensure that others in the immediate area are alerted to the possible MEC and advise them to wait in a safe area until the item is inspected and clearly marked.
Step 4: No MEC will be moved or repositioned unless requested and authorized by the Contracting Officer. The UXOSS will notify the facility of the location, type, and condition of the item.
Step 5: The UXOSS will photograph (if possible) and document the item in the daily log.

Specific requirements while working in the area include the following:

- Entry to the area is restricted to daylight hours only;
- Vehicles must remain on roadways, designated jeep trails, or areas cleared by the MEC personnel;
- Vehicle must be positioned pointing out of the site with keys in the ignition in the event of an emergency;
- Personnel must remain in groups of two or more and remain within arms length of their partners;
- Personnel must maintain clear communications with MEC personnel and have a working knowledge of radio procedures;
- DO NOT transmit on the radio when within 35 feet of any MEC item;

3.5 General Physical/Biological Hazards

Anticipated physical/biological hazards include:

- Heat stress (high ambient temperature);
- Noise;
- Slip, Trip and Fall;
- Equipment Operation;
- Electrical;
- Utility avoidance (overhead and underground);
- Falling objects; and
- Biological hazards.

3.5.1 Heat Stress

Exposure monitoring for heat stress is described in Section 6.2.

3.5.2 Noise

OSHA requires the use of hearing protection by all employees when noise levels exceed 85 decibels. This limit may be exceeded on or near heavy equipment. A sound level meter, operating in the dBA slow response mode, will be used to monitor noise levels when personnel are working near heavy equipment. Site workers will wear hearing protection when sustained noise levels exceed 85 decibels. In addition, all Malcolm Pirnie personnel must undergo initial employment, annual, and employment termination examinations, during which a hearing test is conducted.

3.5.3 Slip, Trip and Fall Hazards

Ground irregularities due to topography or protruding materials (e.g., nails in boards, broken glass) may pose a fall, slip or trip hazard to workers. Leather shoes with puncture proof inserts will be worn by personnel to protect against sharp objects which may be protruding from the surface or when using heavy equipment. There are potential hazards from the presence of wet areas, puddles, oil and grease, debris, loose or sandy soils, or other obstructions that may be within the passageways or walkways. Field personnel will be briefed by the UXOSS each morning on the location and type of obvious hazards in the work areas. Site workers are to take care in areas where ground irregularities or protruding objects exist and may not be observed due to vegetation.

3.6 Equipment Operation

To prevent entrainment in moving machinery, Malcolm Pirnie employees will maintain a safe distance from heavy machinery. Malcolm Pirnie employees will remain outside the swing radius of heavy equipment. The UXOSS or designee will remind all site workers each morning about the hazards of moving equipment. Subcontractors will place a worker near moving heavy equipment to guide the operator and warn others.

3.6.1 Utility Avoidance (Overhead and Underground)

Underground utilities may pose an electrocution, explosion, or other hazard during activities. The location of underground utilities will be determined prior to intrusive activities. Utility companies and other responsible authorities will be contacted to locate and mark the locations. On commercial or industrial properties where underground utilities are expected and public utility companies may not have information on buried utilities, a Level 2 survey will be conducted to locate all above ground and below ground utilities. A Level 2 survey will consist of the use of remote sensing devices (e.g., electrical resistivity, ground penetrating radar, and magnetometer).

3.6.2 Electrical

Electrical storms (thunderstorms) may pose an electrocution hazard. During thunderstorms, all heavy equipment will be shut down, drilling activities will be terminated, and all personnel on-site will take refuge in buildings.

All electrical equipment, power tools, and extension lighting used on this site will be low voltage or protected by ground fault circuit interrupters.

3.6.3 Falling Objects

If there is a danger of falling objects on a property, the entire area inside the exclusion zone will be a hard hat area. Hard hats will also be worn within 50 feet of activities posing an overhead hazard.

3.6.4 Biological Hazards

Persons working on-site should be aware of the presence of biological hazards, including snakes, poisonous plants and poisonous insects. Non-poisonous snakes and poisonous snakes may be present. With the exception of some rare species of poisonous snakes, snakes will not attack unless provoked. All snakes encountered should be avoided. If a snake is discovered, the

UXOSS should be immediately informed of the snake's location, size and type, if known. In most cases, only a brief interruption of work will be necessary to allow the snake to vacate the work area on its own.

Poison ivy is a climbing plant with ternate leaves (arranged in threes) and white berries. Poison oak is similar to poison ivy, but its leaves appear oak-like in form. The leaves of these poisonous plants produce irritating oil causing an intensely itchy skin rash and characteristic bullous lesions. These plants are to be avoided.

Working in tall grass, especially in or at the edge of wooded areas, increases the potential for ticks to bite workers. Ticks can be particularly numerous in the spring and fall. Ticks are vectors of many different diseases including Rocky Mountain spotted fever, Q fever, tularemia, Colorado tick fever and Lyme disease. Ticks attach to the skin and intravenously feed on blood, creating an opportunity for disease transmission. Covering exposed areas of the body and using insect repellent containing N,N-diethyl-m-toluamide (DEET) help prevent tick bites. Periodically during the workday, employees should inspect themselves for the presence of ticks. If a tick is discovered, the following procedure should be used to remove it:

- Do not try to detach a tick with your bare fingers; bacteria from a crushed tick may be able to penetrate even unbroken skin. Fine-tipped tweezers should be used.
- Grip the tick as close to your skin as possible and gently pull it straight away from you until it releases its hold.
- Do not twist the tick as you pull and do not squeeze its body. That may actually inject bacteria into your skin.
- Thoroughly wash your hands and the bite areas with soap and water. Then apply an antiseptic to the bite area.
- Save the tick in a small container with the date, the body location of the bite and where you think the tick came from.
- Notify the UXOSS of any tick bites as soon as possible.

3.6.5 Trench Collapse or Cave-In

When working on sites that contain MEC, it is possible to encounter a camouflet. A camouflet is an underground cavity that may form when an explosive ordnance item penetrates the earth's

surface to a depth where the force of the explosion is not enough to rupture the surface. The atmosphere of the cavity is filled with carbon dioxide as well as other gasses that will not sustain life. There is a potential for a cave-in when sufficient pressure is applied to the surface.

Whenever possible, workers shall not enter trenches or test pits for any reason. If sampling is necessary, it shall be performed using remote equipment or devices (e.g., backhoe buckets, shovels, or equivalent).

If entry is required at depths greater than four feet, use OSHA protective systems (such as sloping, benching, shoring), a competent person to inspect the trench prior to entry, emergency retrieval systems, safe ladders, and a confined space entry permit, where required, to ensure safe atmospheres.

All simple slopes in excavations greater than 20 feet shall have a maximum allowable slope of 1 1/2:1 Horizontal: Vertical or 34°, as measured from the horizontal.

Store excavated materials/spoils greater than two feet from the edge of excavation and/or have retaining devices.

Properly sign and barricade all trenches/excavations to restrict unauthorized pedestrian and vehicular traffic.

As feasible, back-fill trenches upon completion of work. Do not leave open trenches unattended unless covered by steel traffic plates.

3.7 Task-Specific Hazards and Control Measures

A summarized activity hazard analysis will be prepared for all site-specific tasks and included in the installation-specific HASP in Attachment 1**.** The analysis will include a description of the

hazards and the mitigating or control measures required to prevent accidents. New activities or tasks will require a new, written hazard analysis prior to conducting the task.

4.0 HEALTH AND SAFETY ORIENTATION TRAINING

Malcolm Pirnie and subcontractor personnel involved with the investigation activities are required to have completed the 40-hour hazardous materials health and safety training as specified in 29 CFR 1910.120. This training, designed to orient personnel potentially exposed to hazardous substances, health hazards, or safety hazards, includes the following:

- Safety and health risk analysis;
- Use of PPE;
- Work practices by which the employee can minimize risks from hazards;
- Safe use of engineering controls and equipment;
- Medical surveillance requirements, including recognition of symptoms and signs which might indicate overexposure to hazards;
- Procedures for environmental monitoring, site control and decontamination;
- Emergency response plans;
- Introductory Radiological Worker Training;
- Chain-of-command;
- MEC familiarization training;
- Hazard Communication Program, including installation-specific MSDSs; and
- How to respond to media inquiries.

All personnel will also have proof of attendance at an annual eight-hour Health and Safety refresher course if their 40-hour course was completed more than a year prior to the start of field activities.

A MEC orientation program (refer to Section 5.1) will be presented to all field personnel before any work begins. Hazardous work permits, developed for this investigation, are presented in Attachment 1.

"Tailgate" or "toolbox" safety meetings will be conducted each morning by the UXOSS for all phases of work during which all field teams will be provided with a daily work order that will include a checklist with utility clearance and known conditions on the property. Topics of discussion will include work tasks and associated hazards, work zones and designated PPE,

emergency procedures, evacuation routes, and prior safety concerns. These meetings must be documented on the prescribed forms.

4.1 Specialized Training

Malcolm Pirnie, subcontractor, and other field personnel are to be knowledgeable in the particular hazards that may be encountered during this project and familiar with safe operating procedures. This will be accomplished through the review of this HASP, specialized training prior to the commencement of the field work, an audit of field activities and safety meetings during the program, as discussed below.

Field personnel should have a minimum of three days of actual field experience under a skilled supervisor and be familiar with emergency response procedures outlined in this HASP. The UXOSS and all supervisory personnel will have additional training, including cardiopulmonary resuscitation (CPR), First Aid, and eight-hour Hazardous Waste Operations and Emergency Response Supervisor training. Subcontractors will be responsible for ensuring that their employees receive specialized training for their job functions and responsibilities.

4.1.1 Pre-Investigation Health and Safety Briefing

Malcolm Pirnie and subcontractor personnel involved with the project will attend an installation-specific health and safety briefing prior to initiation of the field activities. The topics to be discussed will include:

- Characteristics and potential hazards of contaminants known to be present at the site;
- Personal protective clothing function, donning/doffing, frisking;
- Respirators: selection, use, care;
- Personal hygiene;
- Environmental monitoring;
- Decontamination procedures;
- Site control and work zone designations;
- General safety concepts;
- Emergency recognition and prevention;
- Heat stress;

- Signs and symptoms of over exposure to site specific chemical hazards;
- Hazard communication
- Emergency response plan; and
- Site contingency plans.

4.1.2 Morning Safety Meetings

The UXOSS or designee shall conduct morning safety and health briefings on a daily basis. Problems relative to respiratory protection, inclement weather, heat stress, or the interpretation of newly available environmental monitoring data are examples of topics that might be covered during these briefings. An outline report of meetings giving the date, time, attendees, subjects discussed, and instructor shall be maintained. Visitors will be properly oriented to existing site conditions, planned activities, levels of personal protection, and other procedures outlined in this HASP.

4.1.3 Hazard Communication

Malcolm Pirnie has a written hazard communication program which was established to meet the requirements of 29 CFR 1910.1200, and field activities shall be implemented in accordance with that program, as described below.

MSDSs for hazardous chemicals introduced to the site by Malcolm Pirnie and their subcontractors will be present at the site, for review by all on-site personnel. Labels on containers used by Malcolm Pirnie are as originally received (not to be defaced) and are to contain the following information: (1) the identity of the hazardous chemical(s); (2) the appropriate hazard warnings; and (3) the name and address of the chemical manufacturer. If an employee transfers chemicals from a labeled container to a portable container, a label that contains those three items must be affixed to it. If the portable container is intended only for that employee's immediate use (during the same work shift), the product name only shall be clearly

marked on the container. The employee will be responsible for properly emptying, cleaning or disposing of the portable container immediately after use.

As part of the installation-specific health and safety orientation conducted by the UXOSS, a review of our hazard communication program will be included to inform employees of hazardous chemicals to which they may be exposed during field activities. Subcontractors will also attend the hazard communication training session. If the chemical hazard changes or a new chemical hazard is introduced into the area after work begins, additional training will be provided by the UXOSS.

Installation-specific hazard communication training for hazardous chemicals introduced to the site by Malcolm Pirnie will include:

- Properties and hazards (chemical, physical, toxicological) of each hazardous chemical;
- Health hazards, including signs and symptoms of exposure and any medical condition known to be aggravated by exposure;
- Measures employees can take to protect themselves, including: appropriate work practices or methods for proper use and handling, procedures for emergency response, and the proper use and maintenance of PPE, as required;
- Work procedures for employees to follow to protect themselves when cleaning hazardous chemical spills and leaks; and
- Use of the container labeling system and the MSDSs including: where MSDSs are located, how to read and interpret the information on both labels and MSDSs, and how employees may obtain additional hazard communication information;

Installation-specific hazard communications training will also cover hazardous chemicals introduced by other employers and shall emphasize:

- Information about the hazardous chemicals to which Malcolm Pirnie's employees may be exposed;
- An explanation of the labeling system other employers are using;
- Information about the precautionary measures Malcolm Pirnie employees need to take to protect themselves during normal operating conditions and in emergencies; and
- Location of MSDSs for hazardous chemicals brought to the site by other employers.

The UXOSS shall document the training, including the agenda and list of attendees. This subsection of the HASP and the hazard communication training conducted as described above,

shall be the mechanism for informing other employers planning to be on-site of hazardous chemicals introduced to the site by Malcolm Pirnie.

5.0 MEDICAL SURVEILLANCE AND EXPOSURE MONITORING

5.1 Medical Surveillance

Malcolm Pirnie personnel who may have potential exposure to hazardous materials will have initial employment, annual, and termination examinations. Medical evaluations will be performed by an approved occupational physician in accordance with Malcolm Pirnie's Medical Monitoring Program. All Malcolm Pirnie field personnel shall be enrolled in Malcolm Pirnie's Medical Monitoring Program, be medically approved to wear respirators, and fit-tested in accordance with OSHA requirements. Subcontractors are also required to meet medical surveillance requirements for this project.

Purpose - The purposes of the medical evaluation are to: 1) determine fitness for duty on hazardous waste sites; and 2) establish baseline data for future reference. Such an evaluation is based upon the employee's occupational and medical history, a comprehensive physical examination, and an evaluation of the ability to work while wearing protective equipment. The medical examinations include an evaluation of the workers' ability to use respiratory protective equipment according to protocol published in 29 CFR 1910.134.

Supplemental Examinations - Supplemental examinations may be performed whenever there is an actual or suspected excessive exposure to chemical contaminants or upon experience of exposure symptoms or following injuries or temperature stress.

5.2 Heat Stress Monitoring

Whenever feasible, the level of protection established for workers will be based upon quantitative determinations of the radiological and chemical agents and physical stresses present in the work environment. It is proposed that work will be conducted during the summer months; therefore, heat exposure is an issue of concern.

Heat stress is probably one of the most common and potentially serious illnesses at hazardous waste sites. The potential for heat stress is dependent on a number of factors, including environmental conditions, clothing, workload, physical conditioning, and age. The effects of heat stress can range from mild symptoms, such as fatigue, irritability, and decreased mobility, to death. The body's response to heat stress includes the following:

Heat Rash: A result of continuous exposure to heat and humidity, heat rash decreases the body's ability to tolerate heat.

Heat Cramps: A result of profuse perspiration with inadequate fluid intake and chemical replacement, heat cramps are signaled by muscle spasms and pain in the abdomen and the extremities.

Heat Exhaustion: A result of increased stress on various organs. The signs of heat exhaustion include shallow breathing; pale, cool, moist skin; profuse sweating; dizziness and lassitude.

Heat Stroke: The most severe form of heat stress, heat stroke must be relieved immediately to prevent severe injury or death. The signs of heat stroke are red, hot, dry skin; no perspiration; nausea; dizziness and confusion; strong, rapid pulse; and coma. The body must be cooled and medical attention sought immediately.

Measures to prevent heat stress include regular work breaks during field activity, regular fluid replenishment, and the availability of shelter (i.e., shaded area). All personnel will be made aware of the symptoms of heat stress. Should one or more symptoms be detected, the affected worker will be assisted to seek shade, drink plenty of fluids, and seek medical attention, if required.

Several screening techniques can be used to detect early warning signs of heat stress. The following method, based on body temperature measurements, is simple and straightforward and

may be conducted by the UXOSS. Body temperature may be measured with a digital-readout clinical ear thermometer with disposable tips.

Body temperature may be measured for three minutes with an ear thermometer at the end of each work period and before drinking. Temperature at the end of the work period should not exceed 99.6°F. If the temperature does exceed 99.6°F, the next work period should be shortened by 10 minutes (or 33%), while the length of the rest period stays the same. If the temperature exceeds 99.6°F at the beginning of the next rest period, however, the following work cycle should be further shortened by 33%. Temperature should be measured again at the end of the rest period to make sure that it has dropped below 99.6°F. No worker may be permitted to continue wearing semi-permeable or impermeable garments when his/her temperature exceeds 100. 6°F.

6.0 PERSONAL PROTECTIVE EQUIPMENT

6.1 General Protection Levels

Personnel must wear protective equipment when work activities involve known or suspected radiological or chemical atmospheric contamination; when vapors, gases, or particulates may be generated; or when direct contact with dermally active substances may occur. Respirators can protect the lungs, the gastrointestinal tract and the eyes against air toxicants. Chemical-resistant clothing can protect the skin from contact with skin-destructive and skin adsorbable chemicals. Good personal hygiene limits or prevents the ingestion of materials.

Equipment designed to protect the body against contact with known or anticipated chemical hazards has been divided into four categories according to the degree of protection afforded, Levels A through D. For the site inspections, it is expected that only Level D PPE will be necessary. Level D is described below:

- Level D/Modified Level D: Level D should be selected only when there are no respiratory or skin hazards suspected or known to exist at the site. Modified Level D PPE is selected when no respiratory hazards are suspected or known to exist, yet the potential for dermal hazards including contact with contaminated soils, splashes or immersion exists. If the potential for splashes or immersion exists, coated-type chemical resistant coveralls (such as Saranex) and hard hats with face shields could be selected. If the only dermal hazards that existed were related to soil sampling, a non-coated semi-permeable-type coverall (such as Tyvek) could be selected, thereby avoiding the heat stress hazards associated with an impermeable coverall.

The level of protection selected is based primarily on:

- Types and measured concentrations of the contaminants in the ambient atmosphere and their associated toxicity; and
- Potential or measured exposure to substances in air, splashes of liquids or other indirect contact with material due to the task being performed.

In situations where the types of contaminants, concentrations, and possibilities of contact are not known, the appropriate level of protection must be selected based on professional experience and

judgment until the hazards may be further characterized. The individual components of clothing and equipment must be assembled into a full protective ensemble to protect the worker from installation-specific hazards, while at the same time minimizing hazards and drawbacks of the personal protective gear itself. Ensemble components outlined in the following subsection are based on the widely used Environmental Protection Agency (EPA) Levels of Protection.

In general:

- All protective headgear shall meet the requirements of the American National Standards Institute (ANSI) Z89.1, Class A or ANSI Z89.2, Class B.
- Personnel will be provided with eye and face protective equipment when machines or operations present potential eye or face injury from physical, chemical or radiological agents. Eye and face protective equipment shall meet the requirements in ANSI Z87.1, Practice for Occupational and Educational Eye and Face Protection.
- Persons requiring corrective lenses in eyeglasses, when required by this regulation to wear eye protection, will be protected by one of the following:
- Eyeglasses whose protective lenses provide optical correction; or
- Goggles that can be worn over corrective lenses without disturbing the adjustment of the spectacles; or
- Goggles that incorporate corrective lenses mounted behind the protective lenses.
- If excessive noise levels are encountered, particularly around heavy equipment operation, noise protection shall be provided as appropriate.
- Persons handling rough, sharp-edged, abrasive materials or whose work subjects the hand to lacerations, punctures, burns, or bruises will use general-purpose outer hand protection in addition to the chemical resistant inner and outer gloves, as required.
- Employees will wear clothing suitable for the weather and work conditions. The minimum will be long sleeved shirt, long trousers, and protective work shoes or boots. Canvas tennis or deck shoes are not acceptable.
- Protective footwear will be worn by all persons who are engaged in the work. Steel-toed boots cannot be worn for the site inspections since the metal in the shoes will limit the effectiveness of the magnetometer and EM 61.
- PPE will be inspected regularly and maintained in serviceable and sanitary condition and, before being reissued to another person or returned to storage, will be cleaned, disinfected, inspected, and repaired.

6.2 Required Level of Protection

Based upon current information regarding the hazard evaluation of the tasks to be completed (see Section 1.0), the required level of personal protection is Level D. A summary of the Level

D PPE requirements can be found in Table 6-1. The *MP Corporate Health and Safety Program Guide* (June 1988) contains the protocol for PPE and Respiratory Protection, as required by OSHA (29 CFR 1910.120).

Level D

Equipment Requirements for Level D are as follows:

- Coveralls or suitable work uniform
- Gloves (optional)
- Boots/shoes with composite toe (steel toed boots should not be worn if using a magnetometer or other geophysical instrument), leather or chemical-resistant
- Safety glasses or chemical splash goggles (optional)
- Hard hat (face shield optional)
- Hearing protection

Table 6-1: Summary of Level D PPE Requirements

Level	When Required	Equipment
Level D	No contaminants are present or contaminants are present below the action level. Work functions preclude splashes, immersion, or potential for unexpected inhalation of any radionuclides.	Non high-static work shirt and full-length cotton pants or coveralls ANSI standard Z41.4 steel-toed work boots (unless conducting magnetometer operations) ANSI standard Z89.1 hard hat (when working around heavy equipment or overhead "bump" hazards) ANSI standard Z87.1 safety glasses EPA standard hearing protectors (when working in high noise areas [e.g., steam cleaners and heavy equipment]) Reflective safety vests when working around traffic areas Heavy duty leather work gloves (when appropriate)

6.3 Inspection of PPE

Before use of protective clothing, all personnel shall determine that the clothing material is correct for the specified task at hand. The clothing is to be visually inspected for imperfect seams, non-uniform coatings, tears and malfunctioning closures. It is to be held up to the light to check for pinholes. It is to be flexed to observe for cracks or other signs of shelf deterioration. If the product has been used previously, it should be inspected inside and out for signs of chemical deterioration, such as discoloration, swelling and stiffness. During work, the clothing should be periodically inspected for evidence of chemical deterioration, closure failure, tears, punctures and seam discontinuities.

6.4 PPE Doffing Guidelines

The recommended sequence for removing PPE is as follows:

- Wash/rinse (if necessary) excess mud or other debris from outer boots, gloves, and clothing;
- Remove inner latex/nitrile gloves and cloth liners;
- Wash hands; and
- Discard disposable PPE into a properly labeled container and handled as contaminated waste.

7.0 HAZARDOUS MATERIAL MONITORING

It is not anticipated that there will be chemical exposures that would require air monitoring. Potential chemical hazards are from discrete, identifiable sources, such as oil or cleaning substances used as part of the work. Biological and explosive hazards will be monitored visually. Monitoring is not required for this project and will be addressed as a task specific evolution in the event of a scope of work change.

7.1 Radiological Monitoring

Radiological monitoring is not a part of this project nor or are the site workers trained to handle this situation. In the event that any potential radiological devices are discovered, the situation will be avoided and reported immediately.

8.0 SITE CONTROL MEASURES

8.1 General

A daily log containing the names of personnel, site entry and exit times, and their levels of personal protection shall be maintained.

8.2 Site Control

Site Control is necessary to prevent unauthorized, untrained, or unprotected personnel or visitor from being exposed to the various hazards associated with the site. Level D or greater PPE will be observed at all times during the performance of field activities. Personnel performing field activities will always use the buddy system while at the site. If separation is absolutely necessary, a communication device such as cellular phone or radio will be required unless its use is restricted due to the safety. Other site control measures may include the following.

- Requiring all personnel and visitors to sign in and out on the Personnel Visitor Daily Roster.
- Requiring all site visitors to receive prior approval from the FPM. Visitors will be allowed on-site solely for the purpose of observing site conditions or operations. Upon arrival, visitors will report to the FPM or UXOSS, where he/she will receive and sign the Visitor Health and Safety Form. Visitors may not enter controlled work areas without producing documentation that training and medical requirements have been met. Visitors must be escorted in MEC areas by UXO technician.

8.3 Work Zones

In order to control the potential spread of contamination from MC and to prevent injury to Malcolm Pirnie field personnel, work zones will be classified according to two categories outlined below: a Controlled Work Zone and a Support/Clean Zone. The Support/Clean Zone will be established outside of the Controlled Work Zone and maintained as contamination free. The controlled work zone is the area inside of the site boundaries that has a potential for MEC or MC hazards. Primary functions of locations are:

- Support/Clean Zone
    - Site access for personnel, materials, and equipment;
    - Site egress for decontaminated personnel, materials, and equipment;
    - Storage area for clean work equipment;
    - An area for breaks, consumption of food and beverages, and other related activities; and
    - Vantage point for site visitors.
- Controlled Work Zone
    - Access for only those UXO trained personnel or those escorted by UXO trained personnel.

The specific location of work zone boundaries shall be determined jointly by the FPM, the UXOSS or designee and the subcontractor prior to field mobilization. Decontamination of personnel will be performed as outlined in Section 11.0 before entering the Support/Clean Zone. Only personnel who are essential to the completion of the limited visual survey will be allowed access to work areas, if they are wearing the prescribed level of protection.

9.0 STANDARD OPERATING PROCEDURES FOR SAFETY

A range of physical and explosive hazards exist that must be understood by all field personnel assigned to work on-site. At a minimum, the safe work practices to be followed at the site shall include:

- The number of personnel and equipment on the site shall be minimized, consistent with effective site operations.
- On-site personnel shall use the "buddy" system. No one may work alone (i.e., out of earshot or visual contact with other workers). In addition, each field team will be required to carry two-way radios and have access to a cellular phone.
- Because of potential safety issues associated with abandoned and/or uninhabited buildings, site workers must stay within their designated work areas. No one should enter restricted access areas without authorization of the UXOSS.
- Site activities will be performed to minimize dust production and soil disturbance.
- Contact with surfaces/materials either suspected or known to be contaminated will be avoided to minimize the potential for transfer to personnel, the need for decontamination, and cross contamination.
- Eating, drinking, chewing gum or tobacco, smoking, or any practice that increases the probability of hand-to-mouth transfer of contaminated material, is strictly prohibited in the work area outside the designated clean zone.
- Medicine and alcohol can potentiate the effects of exposure to toxic chemicals. Due to possible contraindications, use of prescribed drugs should be reviewed with the contractor or subcontractor occupational physician. Alcoholic beverage and illegal drug intake are strictly forbidden during site work activities.
- When it is necessary for a visitor to observe the fieldwork, that person will be issued appropriate PPE, briefed on potential hazards, safety practices, decontamination procedures and site communications. All site visitors must supply respiratory equipment and proof of training/fit testing to the UXOSS or designee.
- All employees have the obligation to correct or report unsafe work conditions.

10.0 DECONTAMINATION PROCEDURES

10.1 Personnel Decontamination

The decontamination procedures for this project will consist of a soap and water wash prior to eating, smoking, or drinking. The SI should not involve any direct personal exposure to any hazardous materials. Only materials that are not hazardous or are not regulated by the Resource Conservation and Recovery Act (RCRA) will be used to prevent the generation of mixed waste. Contaminated personnel shall be decontaminated using materials such as waterless hand cleaner and paper towels or rags, whenever possible, to minimize waste volumes. Good house keeping procedures as well as a common sense approach will be practiced during the SI.

10.2 Disposal Procedures

Disposal procedures for Investigation Derived Waste are presented in the Field Sampling Plan.

10.3 Confined Space Entry Procedures

There are no permit-required confined spaces anticipated for this project. If an area is suspected to be a confined space, the FPM shall halt work in the affected area and notify the facility concerned.

11.0 EMERGENCY RESPONSE PLAN

11.1 Emergency Planning

The UXOSS or designee shall implement this emergency response plan whenever conditions at the site warrant such action. The UXOSS will be responsible for assuring the evacuation, emergency treatment, and emergency transport of site personnel as necessary and notification of emergency response units and the appropriate staff.

The UXOSS or designee will inform the local fire department about the nature and duration of work expected on the site and the type of contaminants and possible health or safety effects of emergencies involving these contaminants.

11.2 Emergency Equipment

Emergency equipment will be readily accessible and distinctly marked. Malcolm Pirnie and subcontractor personnel will be familiar with the location and trained in the use of emergency equipment. Emergency equipment that will be available on-site includes:

First Aid Kits

- First Aid Kits will conform to Red Cross requirements and the requirements of 29 CFR 1910.151.
- First Aid Kits shall consist of a weatherproof container with individually sealed packages for each type of item.
- First Aid Kits will be fully equipped before being sent to the site. It will be checked weekly by the UXOSS or designee and expended items will be immediately replaced.
- First Aid Kits will be carried in the field vehicles, distinctly marked, and readily accessible.

11.3 Personnel Roles, Lines of Authority and Communication

Working on former active training ranges requires that site personnel be in constant communication with each other. All work that involves potential exposure of personnel to

explosive hazards or MC requires the use of the buddy system. The responsibilities of workers to utilize the buddy system include:

- Providing his/her partner with routine and emergency assistance;
- Observing his/her partner for signs of chemical exposure or heat stress;
- Periodically checking the integrity of his/her partner's PPE; and
- Notifying others if emergency help is required.

Table 11-1: Hand Signals

Signal	Definition
Hands clutching throat	I cannot breathe
Hands on top of head	Need assistance
Thumbs up	I am OK; affirmative
Thumbs down	No/negative
Arms waving upright	Send backup support
Grip partners wrist	Exit area immediately
Horn - one long blast	Evacuate site
Horn - two short blast	All clear, return to site

11.4 Emergency Recognition and Prevention

As part of the initial installation-specific health and safety briefing, the UXOSS and the FPM will address emergency recognition and prevention. Topics will include hazard recognition regarding tasks to be performed in addition to hazards associated with site contaminants. Other topics relating to emergency recognition and prevention are mentioned in other chapters of the HASP.

11.5 Adverse Weather Conditions

In the event of adverse weather conditions, the FPM and UXOSS or designee will determine if work can continue without sacrificing the health and safety of site workers. Some of the items to be considered prior to determining if work should continue are:

- Potential for heat stress;
- Inclement weather-related working conditions;
- Limited visibility;
- Potential for electrical storms.

11.6 Emergency Medical Treatment/First Aid

A minimum of two site personnel will be first aid/CPR qualified. In the event of personal injury, emergency first aid will be applied on site as deemed necessary. Decontaminate as appropriate and transport the individual to the nearest medical center if needed. Appropriate medical data sheets will be provided by the Site Safety Officer (SSO) to the medical facility. A standard Malcolm Pirnie Accident Investigation Report will be filled out.

If any personnel have been directly exposed to chemicals or contaminants of concern, follow the procedures outlined below:

- 15 minutes. Decontaminate and provide medical attention. Eye wash stations will be provided on-site. If necessary, transport to the nearest medical facility.
- Inhalation: Move to fresh air and, if necessary, transport to the nearest medical facility.
- Ingestion: Decontaminate and transport to the nearest medical facility.

In the event of a serious medical emergency, the Site Specific HASP will include:

- Route to Emergency Medical Facility
- Maps to medical facility
  Emergency Numbers

11.7 Evacuation Procedures/Safe Distances

Evacuation procedures will occur at three levels: (1) withdrawal from immediate work area (100 feet or more upwind); (2) site evacuation; and (3) evacuation of surrounding area. Anticipated conditions that require these responses are described in the following subsections. If site evacuation is required, all field team members will be notified by cellular phone.

Withdrawal Upwind

Withdrawing upwind (100 feet or more) will be required when: (1) ambient air conditions contain greater contaminant concentrations than guidelines allow for the type of protection being worn (the work crew may return after donning greater protection and/or assessing the situation as transient and past) or (2) a breach in protective clothing or minor accident occurs.

The work crew will observe general wind directions while on-site. Upon observing conditions that warrant moving away from the work site, the crew will relocate upwind a distance of approximately 100 feet or farther, as indicated by the site monitoring instruments. The HSD, FPM, Installation point of contact and the Baltimore District Project Manager will be notified if a condition exists to withdraw. When access to the site is restricted and escape is thereby hindered, the crew may be instructed to evacuate the site rather than move upwind, especially if withdrawal upwind moves the crew away from escape routes.

Site Evacuation

Evacuation of the site will be required when: (1) ambient air conditions contain explosive and persistent levels of combustible gas, excessive levels of toxic gases, or excessive dust; (2) a fire or major collapse occurs; or (3) explosion is imminent or has occurred.

After determining that site evacuation is warranted, the work crew will proceed upwind of the work site and notify the UXOSS of site conditions. If the decontamination area is upwind and more than 500 feet from the work site, the crew will pass quickly through decontamination to remove contaminated outer suits. As more facts are determined from the field crew, they will be relayed to the appropriate agencies.

The evacuation route and an upwind gathering point will be determined by the UXOSS or designee each day and communicated to all field personnel prior to beginning work. Any modifications to the evacuation route or gathering point will be discussed at the morning safety meetings.

Surrounding Area Evacuation

The area surrounding the site will be evacuated when an explosive hazard is imminent.

11.8 Site Security and Control

A daily log containing the names of personnel, including site entry and exit times and their levels of personnel protection, shall be maintained by the UXOSS or designee. Site security may involve the use of security guards to protect equipment or field personnel during investigation activities.

After a site evacuation, the senior person will take a "head count" to match against the Employee/Visitor Daily Roster; search/account for missing persons; notify the emergency crews (as applicable); and limit access into the hazardous area to only necessary rescue and response personnel to prevent additional injury and possible exposures. Work shall not resume until all hazard control issues are resolved to the satisfaction of the FPM and UXOSS.

11.9 Fire or Explosion

In case of fire or explosion, sound the emergency alarm (using the radio) and contact the facility Fire Department for outside assistance, regardless of the size of the incident. The FPM will evacuate all non-response personnel and visitors to the Safe Refuge Area and conduct a head-count. Only trained Emergency Crews will control any large-scale or potentially unmanageable incident. The FPM will direct the off-site responding agencies to the site and will provide them with the site map and a hazard briefing. The FPM and or UXOSS will complete an Incident Report for submittal to the Corporate HSD.

11.10 Spill Containment Plan

As no hazardous products will be brought on-site during the SI, a spill is not anticipated.

11.11 Emergency Response Evaluation

11.11.1Pre-Planning and General Procedures

In the event of an emergency associated with the project activity, the UXOSS shall: 1) take immediate, diligent action to minimize the cause of the emergency; 2) alert the FPM and

applicable facility personnel; and 3) institute measures necessary to prevent any repetition of the emergency. Emergency contact names, telephone numbers, and hospital route maps must be posted in the work area and/or support vehicle. At the beginning of project operations, at least the FPM and UXOSS will become familiar with the emergency route(s) and the travel time required. These procedures shall be thoroughly discussed in the initial "kick-off" briefing and in daily "tailgate" safety meetings. A cellular telephone, fully charged, will be available for any emergency.

Emergency Coordinator

The emergency coordinator (EC) will normally be the FPM or the UXOSS, with the others providing assistance as directed. First-aid and rescue duties will be shared between qualified team members. The EC will contact emergency response agencies and serve as the primary point of contact when they arrive.

Emergency Services

The EC must pre-determine the location and availability of the nearest base and civilian emergency facilities and services. Medical transport may be via ambulance or life flight, depending on response times and/or weather conditions. The EC will coordinate contractor access to base services through the range management and discuss it at the initial "kickoff" meeting.

Emergency Equipment

Maintain the following emergency equipment/supplies on-site: industrial first aid kit, portable eye washes capable of a 15-minute use, blanket or visqueen, and compressed air horn.

Store the emergency and first-aid equipment in an immediately accessible area (e.g., in the staging area). Protect equipment from the elements. The UXOSS will inspect the emergency equipment at the beginning of each field event.

12.0 RECORDKEEPING

Record keeping will include Medical Training Records, Site Safety and Health Plans and Incident Reports. In addition, records of meetings on health and safety matters will be maintained by the HSD.

12.1 Medical Surveillance Report

The employer or the employer's medical center will maintain the original medical monitoring record. 29 CFR 1910.20 requires retention of medical records until termination of employment plus 30 years. The employer shall maintain a copy of the employee's Disclosure Agreement and Physician's Statement.

12.2 Personnel Training Records

Personnel health and safety training records are maintained to document personnel qualifications and capabilities and to demonstrate compliance with company training requirements. Each installation-specific training session will be documented by a training report. The UXOSS will prepare the report and include the date of training, location, a list of attendees and a description of the material covered. The original report will be filed with the HSD. Copies of CPR/first aid training certificates will be retained.

12.3 Health and Safety Plan (HASP)

HASPs will be completed and in-place prior to each work assignment involving field activities. The HASP will be signed and approved by the HSD and FPM. The original of each completed HASP will be placed in the project file. A copy will accompany the field team and be readily available at the work site under the control of the UXOSS or designee. Copies of the HASP will be available to all employees when installation-specific training is provided.

In addition to the HASP, the following documents may also be prepared, as necessary, depending on site conditions and circumstances:

- Site Health and Safety Meeting Reports - will be documented in the field laptop that becomes part of the permanent project file. Telephone conversation records on health and safety decisions will be retained.
- Site Health and Safety Follow-up Report - will be completed by the FPM after completing work covered by the HASP. This report is an internal document only and will be maintained by the HSD.
- Health and Safety Audits - The HSD or his/her designee will periodically audit field activities to determine compliance with the HASP.

12.4 Incident Reports

In case of environmental incidents, fires, property damage, power disruption, or mandated work "shut-downs" (e.g., following storms, equipment failure), the UXOSS will complete and transmit an Incident Report to the FPM and facility management. Any damage, loss, or theft of government property (items/tools/equipment purchased for the contract) will be reported via an Incident Report or equivalent. Report damage, loss, or theft of company property to the FPM.

13.0 NEAR MISS REPORTING

Near-miss incidents that do not result in injury must also be recorded and investigated for accident prevention purposes. The FPM/UXOSS will submit completed Incident Reports to the HSD.

14.0 SUBCONTRACTOR REPORTING

The field supervisor of each subcontracting crew will investigate and complete an accident report that specifies preventive measures in accordance with their internal company policy. The FPM will ensure that this report is transmitted to the HSD within 24 hours of a significant mishap and eight hours of a serious mishap. The UXOSS will record the event on the project Accident/First-Aid Incident Summary Log.

SITE SAFETY TAILGATE MEETING

MALCOLM PIRNIE

PROJECT NAME:		CLIENT NAME:	
PROJECT NUMBER:		PROJECT LEADER:	
PREPARED BY:		DATE:	

ON-SITE SAFETY MEETING RECORD

LOCATION:

Task to be Performed:

I. Purpose for meeting: (check all that apply)

	DAILY SAFETY BRIEFING
	Begin New Task. Task:
	Periodic Safety Meeting
	New Site Procedures
	New Site Conditions / Information
	New Site Workers

MEETING ATTENDEES

NAME (Print)	SIGNATURE	COMPANY
1.		
2.		
3.		
4.		
5.		

Page 2 of 2

ON-SITE SAFETY MEETING RECORD

II. Topic (check all that apply)

	Site Safety Personnel		Decontamination
	Work Area Description		Emergency Response
	Site characterization		Hazard Communication
	Equipment Hazard(s)		On-site Emergency
	Biological Hazard(s)		On-site Injuries
	Chemical Hazard(s)		Evacuation Procedures
	Physical Hazard(s)		Rally Point
	Heat Stress		Emergency Communications
	Cold Stress		Directions to Hospital
	Site Control		Emergency Equipment
	Work and Support Zones		Drug and Alcohol Policies
	PPE		Medical Monitoring
	Air Monitoring		Task Training
	Safe Work Practices		MEC

III. Remarks

V. Verification

I certify that the personnel listed on this roster received the briefing described above. Site personnel not attending this meeting will be briefed before beginning their assigned duties.

_____

Field Project Manager Date

_____

UXO Health and Safety Supervisor Date

Attachment 1: Installation-Specific Health and Safety Addendum

Fort Stewart Health and Safety Addendum

Site Description:
Fort Stewart (FTSW) consists of 279,081 acres and is located north of Hinesville, GA, approximately 40 miles southwest of Savannah, GA. FTSW is the largest Army installation east of the Mississippi River, spanning portions of Bryan, Evans, Liberty, Long, and Tattnall counties. FTSW is bisected by Georgia Highway 119, which runs north to south from Pembroke to Hinesville and Georgia Highway 144, which runs east to west from Richmond Hill to Glennville. Situated south of Interstate 16 and west of Interstate 95, the installation boundaries are roughly defined by the intersection of Interstate 16 and Interstate 95 and the cities of Richmond Hill, Hinesville, Glennville, Claxton, and Pembroke.

Health & Safety Personnel and Contact Information

Project Manager: Shelly Kolb
Mobile Phone: (410) 585-4200

Field Project Manager: David Smith
Mobile Phone: (410) 908-7340

UXO Site Safety Officer: Dan Hains, UXO Technician
Mobile Phone: (813) 404-3885

Corporate Health and Safety Manager: Joseph Golden
Work Phone: (914) 641-2978

Primary Emergency Facility:
Address: Liberty Regional Medical Center
426 Elma G Miles Pkwy

Hinesville, GA 31313
Phone: (912) 369-9400

Other Emergency Numbers:
Fort Stewart Department of Training Range Control: (912) 767-8100 or (912) 767-8777;
Fort Stewart EOD: (912) 767-8717 or (912) 767-8718
Fort Stewart Military Police Desk Operations: (912) 767-2822 or (912) 767-2823 or (912) 767-2824
Fort Stewart Range Control-Range Officer: Jim Pearson (912) 767-8679
Fort Stewart Directorate of Emergency Services (912) 767-8427
Hinesville County Fire/Police/Ambulance: 911
Fort Stewart POC: Algeana Stevenson (912) 315-5227
Fort Stewart POC: Randy Powell-Jones (912) 315-5109
Project Manager, Baltimore Corps of Engineers: Kim Gross (410) 962-6735

Directions to: Liberty Regional Medical Center
426 Elma G Miles Pkwy
Hinesville, GA 31313

1. Start out going south on HERO RD. (.3 miles)
2. Turn slight left onto GA-119 S. (1.8 miles)
3. Turn right onto GA119 / GA-196/ EG Miles PKWY. (.3 miles)
4. End at 426 Elma G Miles Pkwy. Hinesville, GA 31313-4000

Reference:www.mapquest.com

ACTIVITY HAZARD ANALYSIS

1. Phase of Project: Site Inspection		
2. Location: Fort Stewart	3. Contract No.: W912DR-05-D-0004	4. Project: MMRP Site Inspection
5. Prime Contractor: Malcolm Pirnie.	6. Date of Preparation: 8/20/06	7. Est. of Start Date: 3/13/07
Potential Safety Hazard	Procedure to Control or Mitigate Hazard	
1. Magnetometer Assisted Site Walk/Geophysical Survey	Use only trails that have been cleared by the UXO Technician. No smoking, eating or drinking. Always use the buddy system. Always check for good radio communications. Report any findings and obtain a second opinion. Do not touch or move anything. Stay within an arms reach of the UXOSS. Wear the appropriate PPE.	
2. Sampling (soil)	Do not collect samples until the area has been property cleared by UXOSS.	
3. Slip/ Trip/ Fall	Maintain firm footing while walking on uneven surfaces. Avoid open excavations. Wear work boots that are in good condition. Watch where you walk. Only walk in areas that are marked as safe to walk in.	
4. Noise	Use hearing protection in designated areas. Maintain noise control devices: mufflers.	
5. Ticks	Check for ticks following field activities. Spray repellent around shoes, ankles and neck. Avoid rubbing against bushes and trees. Advise crew of tick borne disease symptoms. Advise crew of potential haunta virus areas.	
5. Mechanical Hazards (pinch points) for mechanical equipment including off-road vehicles	Maintain belt, chain, rotating shaft and other moving part guards in their proper position. Keep hands away from rotating/ moving parts. Conduct daily equipment safety inspections.	
6. MEC	Always use trails that have been surveyed by a UXOSS. Do not pick up, move, step on or kick any objects. Immediately report if you observe potential MEC.	
7. Magnetometer Use	Always use firm footing. Pay attention to where you are walking. D o not use as a poker in animals holes.	
8. Contractor's Rep. (Signature and Date)		

Appendix C: Technical Project Planning Session Meeting Minutes

MALCOLM PIRNIE

MEETING MINUTES

Purpose: Fort Stewart Military Munitions Response Program Site Inspection Technical Project Planning Meeting
8:00 am – 3:30 pm

Location: Hunter Army Airfield, GA

Date: 12 September 2006

Attendees	Organization
Timothy Rodeffer	Army Environmental Center (AEC)
Alan Freed	AEC Remedial Manager
Kim Gross	US Army Corps of Engineers, Baltimore District Project Manager
Shelly Kolb	Malcolm Pirnie, Inc.
Afton Hess	Malcolm Pirnie, Inc.
Algeana Stevenson	Fort Stewart (FTSW) Department of Public Works (DPW) Environmental
Randy Powell-Jones	Fort Stewart DPW Restoration
Benoit Causse	Georgia Environmental Protection Division (EPD)

Shelly Kolb opened the meeting with a brief overview of the meeting goals and introductions were made around the table. Before the presentation, a discussion on various related topics occurred.

- Algeana provided the inorganic background data for 16 solid waste management units across FTSW, which will be used to screen soil samples collected during the Military Munitions Response Program (MMRP) Site Inspection (SI) field work. Benoit Causse was not working for GAEPD when the report was finalized and therefore will be reviewing the report for his information.

- In order to meet the requirements of FTSW's Resource Conservation and Recovery Act (RCRA) permit, Fort Stewart will need to submit an extension letter containing the scheduled dates for the MMRP SI field work to GAEPD.

- The Munitions Response Sites (MRS or MR site) will be "Areas of Concern (AOC)" in the RCRA program, not "Solid Waste Management Units (SWMU)". A letter reporting the discovery of the AOCs will be submitted to GAEPD to be in compliance with FTSW's RCRA permit. AEC will provide the information and FTSW will send the letter. The letter will be sent after the SI report is finalized and will include all MR sites in the Historical Records Review (HRR)

including MR sites where a no further action (NFA) is recommended prior to the SI field work (including Small Arms Range 2). Descriptions of MR sites with a NFA recommendation will include a brief explanation of why the NFA recommendation was made.

- Benoit Causse GAEPD indicated that he will be providing updated appropriate regulatory screening criteria.

The TPP presentation continued with a summary of the HRR results for each MRS. During this summary Benoit Causse GAEPD presented two comments on the Stakeholder Draft HRR. The comments were as follows:

Comment: Section 5 does not contain a conceptual site model (CSM) or munitions constituent (MC) pathway analysis figure for Small Arms Range 2.

Response: The HRR research revealed that Small Arms Range 2 did not overlap the cantonment area and therefore is not eligible for the MMRP. This information is presented in Section 4.6.3 of the HRR report. Text will be added to the introduction text of Section 5 indicating that the MRS is no longer MMRP eligible and therefore a CSM will not be created.

Comment: Figure 5-3 MEC Pathway Analysis Figure depicts an incomplete pathway for receptors to MEC on the surface. Since there has been EOD reports in this area this pathway should be potentially complete.

Response: This change will be made and reflected in the Final HRR.

The following MMRP SI field activities and outcomes were discussed and agreed upon during the TPP meeting:

MRS	Munitions of Explosive Concern (MEC) SI Activities		
	Activity	**Purpose**	**Notes**
Anti-Aircraft Range -1	Limited magnetometer assisted visual survey during sampling activities.	Support MEC NFA or further investigation under the RCRA program (equivalent to remedial investigation (RI)). NFA if no MEC is encountered on the surface. RI if MEC is encountered on the surface.	Site is well maintained/mowed so MEC or munitions debris on the surface is not expected.
Anti-Aircraft Range 90mm - 2	None	Further investigation under the RCRA program (equivalent to RI) is recommended for the MRS.	Recommendation is based on historical evidence of multiple overlapping range fans and multiple EOD responses.

MRS	Munitions of Explosive Concern (MEC) SI Activities		
	Activity	**Purpose**	**Notes**
Anti-Tank Range 90mm	Document historical use in Installation Master Plan	NFA is recommended for the MRS.	Recommendation based on current/future use as a RCRA permitted landfill.
Hand Grenade Course	Limited magnetometer assisted visual survey during sampling activities[1].	Further investigation under the RCRA program (equivalent to RI) is recommended for the MRS.	Recommendation based on historical evidence of multiple overlapping range fans and multiple EOD responses.
Hero Road Trench	Conduct a visual survey of unfenced portions of MRS to ensure no MEC or MEC debris remains on the surface.	Further investigation under the RCRA program (equivalent to RI) is recommended for the MRS.	Recommendation based on historical evidence and results of current investigation.
Small Arms Range - 1	N/A	small arms only	No MEC is associated with small arms use.
Small Arms Range - 3	N/A	small arms only	No MEC is associated with small arms use.
1 MEC field activities for the former Hand Grenade Course were updated after the TPP meeting minutes were finalized due to a previously unrecognized error. Discussions during the TPP meeting included visual survey activities during sampling activities.			

MRS	Munitions Constituents (MC) SI Activities		
	Activity	**Purpose**	**Notes**
Anti-Aircraft Range -1	Collect 4 composite surface soil samples at random locations or biased locations if MEC is encountered. Analyze sample for explosives and metals using EPA Methods 8330 and 6010B/6020.	To support CTC and Prioritization Protocol and to support MC NFA or further investigation under the RCRA program (equivalent to RI). The data will be screened using a background data and residential PRGs.	MRS is overlapped by a buffer area of the range fan, near the firing point. Potential munitions that were used are 37mm, 40mm, 90mm anti-aircraft guns. No EOD responses have been reported. The land is currently a Parade Field Associated with the NCO Academy; the field is maintained.
Anti-Aircraft Range 90mm - 2	Collect 1 composite surface soil sample. Analyze sample for explosives and metals using EPA Methods 8330 and 6010B/6020.	To support CTC and Prioritization Protocol and to support MC NFA or further investigation under the RCRA program (equivalent to RI). The data will be screened using a background data and residential PRGs.	The potential munitions used are 40mm, 90mm Anti-Aircraft Projectiles. Tank range munitions are unknown. Several EOD responses have been reported involving C-4 plastic explosives, M-222, GM Dragon Missiles, M-7, MK-2 fragmentation hand grenade. The current and future land use is an Ammunition Supply Point.

MRS	Munitions Constituents (MC) SI Activities		
	Activity	**Purpose**	**Notes**
Anti-Tank Range 90mm	None	NFA is recommended for the MRS. Historic use should be documented in the Master Plan.	The potential munitions use: 90mm, 40mm, 37mm, and various small arms. One EOD response involving an M-7 grenades and an MK-2 fragmentation grenade. MRS is currently an active RCRA permitted landfill. Recommendation based on current/future use.
Hand Grenade Course	Collect 1 biased composite surface soil sample in the center of MRS[1]. Analyze sample for explosives and metals using EPA Methods 8330 and 6010B/6021.	To support CTC and Prioritization Protocol. Further investigation under the RCRA program (equivalent to RI) is recommended for the MRS. The data will be compared to background data and residential PRGs	The potential munitions uses are hand grenades (type unknown), 90mm, 40mm, 37mm, and various small arms. One EOD response reported involving M-7 grenades and an MK-2 fragmentation grenade. The land is currently undeveloped. Recommendation is based on historical evidence of multiple overlapping range fans and multiple EOD responses.
Hero Road Trench	Collect 1 composite surface soil sample. Analyze sample for explosives and metals using EPA Methods 8330 and 6010B/6021.	To support CTC and Prioritization Protocol. Further investigation under the RCRA program (equivalent to RI) is recommended for the MRS. The data will be compared to background data and residential PRGs.	The potential Munitions Use are 5% solution of mustard gas, 5% solution of Lewisite, 50% solution of chloropicrin, pure agent phosgene. No EOD responses reported. MRS is currently fenced and undeveloped and is located adjacent to the Family Housing Maintenance Parking Lot. Recommendation is based on historical evidence and results of current investigation.
Small Arms Range - 1	Collect 4 composite surface soil samples in the undeveloped portions (~41 acres) of the site. Analyze sample for lead by EPA Method 6020.	To support CTC and Prioritization Protocol and to support MC NFA or further investigation under the RCRA program (equivalent to RI). The data will be screened using background data and residential PRG.	The site is overlapped by the firing point but the firing point is a paved heliport pad. The potential munitions used are various small arms. No EOD responses reported. The current land use is Evans's Airfield/Heliport.

MRS	Munitions Constituents (MC) SI Activities		
	Activity	**Purpose**	**Notes**
Small Arms Range - 3	Collect 2 sediment, 2 surface water and 3 composite surface soil samples. Soil samples: 1 in northern and 2 in the southern portions. Sediment samples: 1 on each of the man-made damns of the pond. Analyze samples for lead by EPA method 6020*	To support CTC and Prioritization Protocol and to support MC NFA or RI determination. The data will be screened using a background study and residential PRG for lead.	Potential munitions used are various small arms. No EOD responses reported. The current land use is undeveloped and Hallbrook Pond Recreational Area.
*MC field activities updated after MRS tour. 1 MC field activities for the former Hand Grenade Course were updated after the TPP meeting minutes were finalized due to a previously unrecognized error. Although there are EOD responses associated with the overlapping range fans none of these locations are actually within the MRS. The sample will be collected from the center of the MRS.			

After the presentation the team broke for lunch and traveled to Fort Stewart where a tour of each MRS was conducted. The following are notations from the specific sites.

Site Tour

<u>Small Arms Range 1/Evans Airfield/Helliport</u>

- This area is diagonal to SWMU 29
- The north portion grass-covered and mowed
- The south portion is mostly paved with grass covered areas and shrubs
- Samples should be taken in grass-covered areas.

<u>Small Arms Range 3/Hollbrook Pond</u>

- Site contains a manmade pond that was built in 1966
  - About 20 acres
  - Average of 6 feet in depth
  - Alligators live are present in pond
- Earthen dam is along boundary
- Benoit Causse GAEPD requests that two sediment and two surface water samples be added to the field activities for the site since pond was build after historic use.
- Sediment and surface water and sediment samples should be collected along each side man made of the dam. This is in addition to the three soil samples discussed during the presentation (this was added to the table above).

- The pond is stocked with bass, and catfish

Hero Road Trench Area

- Building 7808 and a housing area are located near the MRS
- Entire MRS does not appear to be fenced.
- Visual survey of MRS should be used to also determine bounds of trench and fill landfill if possible.

Anti-Aircraft Range 2

- MRS includes a combination of mowed grass and wooded areas

Anti-Tank Range

- This MRS was not included in the tour since it is a RCRA permitted landfill Benoit Causse GAEPD did not need to see it.

Anti-Aircraft Range - 1

- This area is completely mowed and maintained and samples should be widely disbursed across the MRS.

Meeting Generated Action Items

- Benoit Causse GAEPD will provide acceptable updated regulatory screening criteria for screening for various sampling media via email.
- Algeana will obtain actual GIS layer of fence for Hero Road Trench Area.
- The Final HRR will be distributed early based on comment received from Benoit Causse GAEPD.

Appendix D: MEC/Multiple Anomaly Discovery Sheet

MALCOLM PIRNIE

MEC/MULTIPLE ANOMALY DISCOVERY FORM

UXO Safety Supervisor:_____ **Date:**_____

Anomaly ID No. (i.e. FAR A-001)		
Anomaly Longitude X/Latitude Y (Northing and Easting) Feet		
Object length		Inches
Object Diameter/Thickness		Inches
Object Weight (Estimated)		**Lb**
Slope of terrain (Check one box)	☐ **<10°** ☐ **10° to 30°** ☐ **>30**	
Vegetation cover (Check one box)	☐ Clear ☐ vegetation ☐ Swamp	
Soil type (Check one box)	☐ Sand ☐ Clay ☐ Rock	
Inclination	**0° 45° 90° 135° 180°**	
Orientation	**N-S NW-SE E-W SW-NE**	

Item Description/Justification/Comments

Anomaly type categories (Check Appropriate Box)

☐ **UXO** ☐ **DMM** ☐ **Munitions Debris** ☐ **Practice Ordnance** ☐ **Inert Ordnance**

☐ **Other** ☐ **Metal Waste** ☐ **Sub Surface Anomaly**

Was photo taken? ☐ **Yes** ☐ **No** **File Name:**

Ordnance Positive Identification (If Known, Record Below and record fuze condition and disposition)

Quantity:	**Ordnance Mark/Mod:**	**Nose Fuze Mark/Mod:**	**Tail Fuze Mark/Mod:**

Ordnance Filler: ☐ Explosive ☐ Propellant ☐ Pyrotechnic ☐ Other N.E.W.

Ordnance Category:

☐ **Bombs** ☐ **Clusters/Dispensers** ☐ **Grenades** ☐ **Guided Missiles**
☐ **Land Mines** ☐ **Misc. Explosive Devices** ☐ **Mortars** ☐ **Projectiles**
☐ **Rockets** ☐ **Pyrotechnics and Flares** ☐ **Small Arms** ☐ **Underwater Ordnance**

Fuzing Types

☐ Piezo-Electric ☐ Proximity (VT) ☐ Impact ☐ Base Detonating
☐ All-ways Acting ☐ Electric ☐ Point Detonating (PD) ☐ Influence
☐ Mech long delay ☐ Point-initiating, Base-detonating ☐ Mechanical Time ☐ Pressure
☐ Powder Train Time Fuze (PTTF) ☐ MT Superquick

Status of MEC/UXO ☐ Armed ☐ Unarmed

Physical Condition of MEC/UXO (Check all that apply) ☐ Broken Open ☐ Soil Staining ☐ Filler Visible ☐ Soil Sample Taken

FOR SUXOSS USE

Disposition: (**Clarify Under Remarks**) ☐ Transport ☐ Leave In Place ☐ Other		**Date:**
Notifications To Installation By:	**Signature:**	**Date**
Transported By:	**Signature:**	**Date:**
Transferred To:	**Signature:**	**Date:**
Storage Location:		
Destroyed By:	**Signature**	**Date:**

Remarks:_____

Signature: _____
SUXOS

UXO – Ordnance fuzed, armed or otherwise prepared for action and fired or placed in such a manner that it constitutes a hazard
DMM – Ordnance that was disposed of by abandonment; may have been fuzed or armed, but was not employed
Inert – Same physical features as an ordnance item but does not and never did contain energetic material
Munitions Debris – Ordnance material that contained or was in contact with energetic material, which has been expended (e.g., fragments from projectile)

Appendix E: HRR Conceptual Site Models

5 CONCEPTUAL SITE MODEL

The CSM was developed following guidance documents issued by the United States Environmental Protection Agency (USEPA) for hazardous waste sites and by the USACE for OE sites. Guidance documents included the USEPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA* (EPA/540/G-89/004) and the USACE's *CSM Guidance Development of Integrated Conceptual Site Models for Environmental Ordnance and Explosives (OE) Sites*, which was final as of February 2003. The CSM uses preliminary findings presented in this HRR to describe the site and its environmental setting. The CSM presents information regarding: (1) MEC and/or MC known or suspected to be at the site; (2) current and future reasonably anticipated or proposed uses of the real property; and (3) actual, potentially complete, or incomplete exposure pathways that link them.

Installation-wide CSM profiles are provided in Section 5.1, and site-specific CSM profiles are provided in Section 5.2 for each of the eight MMRP eligible range. As stated previously in Section 4.6.3, based on the findings of this HRR, Small Arms Range – 2 does not overlap a non-operational area, and as such is not eligible for the MMRP. Therefore, a CSM profile for Small Arms Range – 2 is not provided below.

5.1 INSTALLATION-WIDE CSM PROFILES

5.1.1 MMRP Site Profile

5.1.1.1 Utilities

FSTW is a fully functioning installation containing various basic utilities such as the following: water distribution system, electricity, and communications. Natural gas is distributed by the Defense Energy Supply Center through a 30-mile network via high-pressure mains. There is a wastewater treatment facility located within the cantonment area (FTSW0093).

5.1.1.2 Security

Security has been removed from the installation access gate located on Highway 144; therefore, vehicle access is not restricted at this location (it is uncertain if this is temporary or permanent). The majority of FTSW is currently not fenced. Therefore, people can potentially access FTSW through many of the boundaries that are not fenced. Access is unrestricted, except for fenced or guarded areas.

5.1.2 Physical Profile

5.1.2.1 Climate

The climate of FTSW is humid subtropical. Temperatures range from an average of 52 degrees Fahrenheit (°F) in January to 81°F in July. The annual precipitation is approximately 48 inches, with slightly over half falling from June to September. Average wind speed is from 0 to 5 miles per hour (mph), with the prevailing wind direction to the northwest. However, thunderstorms, hurricanes, and tropical storms, occurring most frequently from May through September, produce gusty surface winds with speeds over 5 mph (FTSW0094).

5.1.2.2 Geology

Known geology of coastal Georgia dates to the Paleozoic epoch and extends to 4000 m below the ocean surface. The sedimentary section consists of 700 m of Paleozoic rocks of Late Devonian age overlain by 2300 m of Early and Late Cretaceous sediments from the Mesozoic era. Cretaceous rocks are overlain by 100 m of Cenozoic sediments, most of which are Eocene in age (FTSW0095).

FTSW is located within the Southern Atlantic Coastal Plain Physiographic Province. It is characterized by a wedge of gentle, southeast-dipping, clastic sediments that cover crystalline basement rock. The unconsolidated clastic (sand, silt and clay) sediments thicken in an easterly direction (FTSW0094). The basement rocks underlying the sediments dip coastward at about 5.7 m per kilometer from the fall line near Macon and Augusta; they appear near the surface in the Savannah area. The basement complex is

composed of metamorphic and igneous rocks that range in age from Precambrian to Triassic. The overlying coastal plain sediments are dominated by clastics in the western areas (near the fall line) and become more nonclastic near the coast (FTSW0093).

5.1.2.3 Topography

Most of the installation is flat lying, with typical elevations of 2 to 30 m above mean sea level (amsl). The northwestern portion is characterized by rolling hills and has typical elevations from 30 to 55 m (FTSW0093).

5.1.2.4 Soil

The most common soil series are Ellabelle loamy sand, Ogeechee, Pelham, Stilson, Rutlege, Leefield and Mascotte (FTSW0095). Most of the soils exhibit a sandy surface layer overlying a subsoil that may be sandy, clayey, loamy, or any combination thereof (FTSW0093). The natural soil types range from excessively drained to poorly drained; the poorly drained soil tends to be higher in organic matter than other soils. The excessively drained soil tends to occur at lower elevations in associations with swamps. The soil is especially vulnerable to erosion once vegetation has been removed (FTSW0094). In coastal Georgia, drainage from three physiographic provinces (the Blue Ridge Mountains, Piedmont Plateau, and Coastal Plain) affects the composition of the alluvial deposits. Near FTSW, the parent material for all soils is water-lain sediments deposited prior to and during the Pleistocene Age (FTSW0095).

5.1.2.5 Hydrogeology

There are three distinct aquifer systems in the FTSW region. The principle artesian aquifer is a deep sequence of limestone of the Eocene to Oligocene age, the primary source of large groundwater withdrawals in the coastal area. This aquifer is generally 92 to 153 m below the surface and is comprised of two different layers. The upper layer is derived from the Oligocene series of sandy, phosphatic limestone and is generally not used as a water source. It is underlain by the Ocala Limestone of Eocene age

(FTSW0095). Primary recharge to the principal aquifer occurs approximately 50 to 90 miles northwest of FTSW, where the rocks composing the aquifer outcrop at the surface. The principal artesian aquifer is overlain by two shallow aquifer systems. A 120- to 150-meter-thick series of Miocene clays, sandy clays, and gravel lies directly above the principal artesian aquifer. The surface aquifer is composed of a relatively thin layer of sands, gravels and clays. It is recharged directly from rainfall percolating through sediments. It is used almost exclusively as a source for domestic water, but primarily as a secondary water supply rather than for drinking water (FTSW0093, FTSW0095).

FTSW has its own potable water distribution system. There are 31 groundwater wells located on the installation; five of these are used to supply water through the distribution system to the cantonment area. The cantonment area wells range in depth from 500 to 800 feet and are cased to depths of 400 to 470 feet. There are four other active groundwater supply wells located elsewhere on the installation that act as individual water supplies. These wells reportedly range from depths of 500 to 560 feet and are cased to about 400 feet. The remaining 22 wells are distributed across the installation. Of these, 2 are on standby and the remaining 20 wells are no longer in use (FTSW0093, FTSW0094). The potable water capacity from the five active wells is approximately 10.4 million gallons per day (FTSW0096).

5.1.2.6 Hydrology

The majority of FTSW is located within the Canoochee River watershed. Most of the surface waters on FTSW drain into the Canoochee River, which passes through the northwestern, central, and southeastern areas of the installation and joins the southward-flowing Ogeechee River (FTSW0093). The Canoochee River merges with the Ogeechee River about 35 miles inland from the Ossabaw Sound. The northeastern section of the installation drains directly into the Ogeechee River, and the southwestern section drains into the Altamaha River (FTSW0095). The Ogeechee River forms part of the northeastern boundary of FTSW. The remaining surface waters represent a relatively small percentage of the total volume of water leaving the area. In the eastern half of the

installation, 60% of the surface area is comprised of marshes and swamps. Four major lakes and ponds are located on FTSW: Pineview Lake, Glissons Pond, Holbrook Pond, and Cantonment Pond (FTSW0093).

5.1.2.7 Vegetation

On a broad scale, there are four types of ecosystems on FTSW: sand hills, pine flat woods, upland forests and wetlands (FTSW0096). The installation acreage is made up of approximately 57% upland forest, approximately 29% forested wetlands and approximately 14% cleared areas (FTSW0095). Major tree species found at FTSW include longleaf pine (*Pinus palustris*), slash pine (*Pinus elliottii*), loblolly pine (*Pinus taeda*), tupelo (*Nyssa sylvatica*), other gums (*Nyssa spp*.), water oak (*Quercus nigra*) and bald cypress (*Taxodium distichum*) (FTSW0093).

5.1.3 Land Use and Exposure Profile

5.1.3.1 Current Human Receptors

Current human receptors include residents (on-installation housing and nearby off-installation residential areas), authorized installation personnel (on-installation and nearby off-installation areas), fisherman/hunters (on-installation and nearby off-installation areas for recreation), visitors and trespassers. For fishing and hunting, access is restricted in some portions of the installation. Therefore, fisherman and hunters are not included as current receptors in some site-specific profiles.

5.1.3.2 Future Human Receptors

There is no anticipated change in access control to the site or land use, so future human receptors are expected to remain the same as current human receptors.

5.1.3.3 Zoning/Land Use Restriction

There are no known zoning or access restrictions at FTSW.

5.1.3.4 Beneficial Resources

Specific beneficial resources include various aquatic habitats that provide fish and crustaceans for human consumption, wetland habitats, and water recreational areas. FTSW has a number of natural or man-made ponds and lakes, the Canoochee River, Canoochee Creek and tributaries, and a number of bottomland swamps and pools. Dense growth of aquatic vegetation is typical, especially during the summer months. FTSW contains approximately 82,148 acres of wetlands, covering approximately 30% of the installation (FTSW0095). Forested areas also serve as a habitat to game, which are hunted for recreation and human consumption. FTSW contains more than 158,869 acres of forested land (FTSW0095). It also contains a large amount of grassland, which serves as a habitat to many species. FTSW acts as a home to many threatened, endangered or special concern plants and animals.

5.1.3.5 Demographics

According to the 2000 U.S. Census, the population at FTSW was 11,205. The city of Hinesville, which is located at the southern boundary of FSTW, has a population of 30,392 according to the 2000 U.S. Census. The city of Savannah, located northeast of FTSW, has a population of 131,510.

5.1.4 Ecological Profile

5.1.4.1 Habitat Type

FTSW has a large portion of forested property and wetlands; therefore, it serves as a habitat for the many animals and fish that reside on FTSW.

5.1.4.2 Ecological Receptors

There are four basic types of vegetative ecosystems on FTSW: sand hills, pine flat woods, upland forests and wetlands. Mixed coniferous and deciduous trees can be found in the sand hills and the upland forests. Pine species can be found in the flat woods. Wetlands provide critical nursery areas, as well as a habitat, for numerous fish, bird and

reptile species. The wetlands range from seasonally saturated to permanently inundated. Wetland ecosystems found on FTSW include black water swamps, bay forests, stream head pocosins, wet pine flat woods and cypress-gum swamps. The fauna at FTSW typically consist of birds (such as: Wood duck, Eastern wild turkey, Bobwhite quail, and Mourning dove), mammals (such as: Eastern gray squirrel, Eastern fox squirrel, Eastern cottontail rabbit, Feral hog, and White-tailed deer), and fish (such as: Largemouth bass, Bluefill, Redear sunfish, Channel catfish, Black crappie, and Hybrid striped bass). FTSW is also a home to many state and federally threatened, endangered, and/or species of concern. The federally listed threatened/endangered species include: Southern bald eagle (*Haliaeetus leucocephalus l.*), Wood stork (*Mycteria Americana*), Red-cockaded woodpecker (*Picoides borealis*), Eastern indigo snake (*Drymarchon coralis couperi*), Flatwoods slalmander (*Ambystoma cingulatum*), and Shortnose sturgeon (*Acipenser brevirostrum*). The state listed threatened/endangered species include the species listed above as well as the following: Peregin Falcon (*Falco peregrinus*) and Gopher tortoise (*Gopherus polyphemus*), (FTSW0095).

5.2 SITE-SPECIFIC CSM PROFILES

The site-specific CSM profiles are provided in the following sections. Information is only provided in the site-specific CSM profiles if it is more detailed than that provided in the installation-wide CSM. If no site-specific information was available, reference is provided to the appropriate installation-wide CSM profile in Section 5.1.

5.2.1 Anti-Aircraft Range - 1

5.2.1.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-1:

Table 5-1: Site Profile – Anti-Aircraft Range - 1

Information Needs	CSM Findings
Area and Layout	Approximately 42 acres Located along the northern boundary of the installation
Structures	None

	NCO Academy buildings are located approximately 250 m north of Anti-Aircraft Range - 1
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped area S: Unidentified road E: Undeveloped area W: Undeveloped area
Security	Fences

5.2.1.2 Physical Profile

The site-specific physical profile is presented in Table 5-2:

Table 5-2: Physical Profile – Anti-Aircraft Range - 1

Information Needs	CSM Findings
Topography	Gently rolling terrain Approximately 20 m amsl
Soil	Sand-silt/sand-clay
Hydrology	Unidentified stream flows north of the site
Vegetation	Grasses

5.2.1.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-3:

Table 5-3: Land Use and Exposure Profile – Anti-Aircraft Range - 1

Information Needs	CSM Findings
Current Land Use/Activities	Parade field for NCO Academy
Current Human Receptors	Installation personnel Visitors Contractors Trespassers
Potential Future Land Use	Same as current use

Information Needs	CSM Findings
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	Undeveloped grassland habitat A potable water well is located north of the MRS.

5.2.1.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-4:

Table 5-4: Ecological Profile – Anti-Aircraft Range - 1

Information Needs	CSM Findings
Habitat Type	Grassy area
Degree of Disturbance	Medium; forest has been cleared

5.2.1.5 Munitions/Release Profile

5.2.1.5.1 Munitions Types

Table 5-5 presents a summary of MEC types that are expected to exist within Anti-Aircraft Range - 1 based on the information collected for this HRR. The mechanisms by which the MEC may have been released into the environment are also presented in this table. The typical release mechanisms for the Anti-Aircraft Range - 1 were intentional activities, such firing munitions, or unintentional activities, such as malfunctioned munitions.

Table 5-5: Summary of Potential MEC Types – Anti-Aircraft Range - 1

Potential Munitions	Primary Release Mechanisms	Potential MEC
37-mm HE M54, 40-mm, 40-mm High Explosive Plastic (HEP),	Munitions firing Malfunctioned munitions Discarded munitions	Partially/fully functioned projectiles/fuzes

Potential Munitions	Primary Release Mechanisms	Potential MEC
90-mm, 90-mm HE 90-mm M71 HE projectiles		

5.2.1.5.2 Maximum Probability Penetration Depth

Table 5-6 provides the expected penetration depths for various types of soils for potential MEC that are anticipated to be found at Anti-Aircraft Range - 1. The depth to which ordnance may penetrate is affected by the types of soil and the groundcover. These expected depths were obtained from Engineering Manual 1110-1-4009 *Ordnance and Explosives Response*, prepared by USACE. For the Anti-Aircraft Range - 1, the soil type is considered sand-silt/sand-clay. The definition of loam states that it is soil that is well mixed with sand and lesser amounts of clay and/or silt. Therefore, the depth of penetration for Anti-Aircraft Range - 1 are based upon the penetration depth for a loamy soil. These penetration depths are estimated on a worst-case scenario, which assumes that the impact is perpendicular to ground surface and that the ordnance item does not become deformed upon impact.

Table 5-6: Summary of Expected MEC Penetration Depths – Anti-Aircraft Range - 1

Ordnance Item/Weapon	Depth of Penetration (feet bgs[1])		
	Sand	Loam	Clay
37-mm projectiles	3.9	5.2	7.9

Ordnance Item/Weapon	Depth of Penetration (feet bgs[1])		
	Sand	Loam	Clay
40-mm, 40-mm HEP projectiles	0.2	0.3	0.4
90-mm, 90-mm HE, 90-mm M71 HE projectiles	0	7	1

(1) below ground surface

5.2.1.5.3 MEC Density

A visual survey has not been conducted at this time to verify the presence or absence of MEC at the Anti-Aircraft Range 90-mm - 1. However, the forested area has been cleared, so it is anticipated that any potential MEC would be found in the subsurface. Since this MRS is overlapped by what appears to be a buffer area of the range the MEC density is expected to be low.

5.2.1.5.4 Munitions Debris

A visual survey was not conducted as part of this HRR. No MEC or munitions debris are known to have been reported at the range. However, based on the activities that occurred at the former range, there is the potential for munitions debris items. No EOD calls have been reported at this site (FTSW0086).

5.2.1.5.5 Associated Munitions Constituents

Potential MC associated with 37-mm, 40-mm, 40-mm HEP, 90-mm, 90-mm HE, 90-mm M71 HE projectiles include: CMP AB, Tetryl, and trinitrotoluene(TNT). (Refer to the appropriate Ordnance Technical Data Sheets in Appendix D.) Soil samples within the boundary of Anti-Aircraft Range - 1 have not been analyzed for explosives or metals;

therefore, it is unknown whether explosives or metals are present in concentrations that exceed regulatory levels. Previous sampling activities conducted at the closed Fort Stewart's Non-Commissioned Officer Academy (TAC-X) Landfill (Solid Waste Management Unit [SWMU] 3) located southwest of the Anti-Aircraft Range - 1 indicate no contamination of CERCLA-listed hazardous constituents in the groundwater detected above regulatory limits and surface water in the area is not being significantly degraded by the past operation of the TAC-X Landfill (FTSW0094).

5.2.1.5.6 Transport Mechanisms/Migration Routes

The primary transport mechanisms identified for Anti-Aircraft Range - 1 include:

Erosion: Anti-Aircraft Range - 1 is a grassy area surrounded by heavily forested area and grassy land; therefore, erosion is not expected in this area and is not a factor in transporting and migrating possible MC contaminated soil.

Soil Disturbance: The current degree of disturbance is medium; the forested area has been cleared. Future development could unveil potential MC that are in the surface and/or subsurface.

Infiltration: Based on the soil types associated with Anti-Aircraft Range - 1, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.1.6 Pathway Analysis

5.2.1.6.1 MEC

Based on the historical use of the site as a buffer area for an anti-aircraft range, the potential exists for MEC to be present on the site. However, MEC on the surface are not expected, as the site is currently the parade field for the NCO Academy and is well maintained (mowed). As illustrated in the Exposure Pathway Analysis for MEC (Figure 5-1), no complete or potentially complete pathways for human or ecological receptors for MEC on the surface are expected to exist. Potentially complete pathways exist for

installation personnel, contractors, and biota for MEC in the subsurface as these receptors have the potential to conduct intrusive activities. The pathway for MEC in the subsurface is incomplete for all other receptors.

5.2.1.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-2), soil and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Based on this, potentially complete exposure pathways may exist for surface and subsurface soil and groundwater through various exposure routes for both human receptors and biota.

Food Chain

Potentially complete pathways to MC in the source media through uptake into vegetation exist for grazing/foraging biota. This exposure pathway is incomplete for all other receptors. As there are no domestic animals on FTSW, the pathway to MC in the source media through this exposure route is incomplete for all receptors. The pathway to MC in the source media through the game/fish/prey exposure route is potentially complete for biota. This exposure pathway is incomplete for all other receptors as their activities are not expected to include hunting.

Groundwater

Precipitation infiltration may provide for contaminant mobility into the shallow or surficial groundwater aquifer. However, based on a review of hydrogeological data (Section 5.1.2.5), it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW. Receptor contact with groundwater is possible if the soil is disturbed through excavation or construction activities, creating possible migration routes/mechanisms for MC in shallow groundwater. As such, installation personnel, contractors, and biota have potentially complete pathways to MC in subsurface soil and/or shallow groundwater through the (incidental) ingestion and dermal contact exposure routes. Given that it is unlikely that

MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW, the dermal and ingestion exposure routes are incomplete for all other receptors. Since the upper aquifer is not used as a potable water source and MC are typically not volatile, the inhalation (vapor) exposure route is incomplete for all receptors.

Subsurface Soil

Since the potential exists for MC in the subsurface soil in the Anti-Aircraft Range - 1 area exists, receptor contact with subsurface soil is possible if the soil is disturbed through excavation or construction activities, creating possible receptor pathways to MC in subsurface soils. As such, installation personnel, contractors, and biota have potentially complete pathways to MC in subsurface soil through the (incidental) ingestion, dermal contact, and inhalation (dust) exposure routes. Visitors and trespassers would not be expected to participate in any intrusive activities; therefore, these exposure routes are incomplete for these receptors.

Surface Soil

All human and ecological receptors within the Anti-Aircraft Range - 1 area may be exposed to surface soil during daily activities. Therefore, the pathways to MC in surface soil through the (incidental) ingestion, dermal contact, and inhalation of dust exposure routes are potentially complete for all receptors.

Figure 5-1: MEC Pathway Analysis- Anti-Aircraft Range - 1

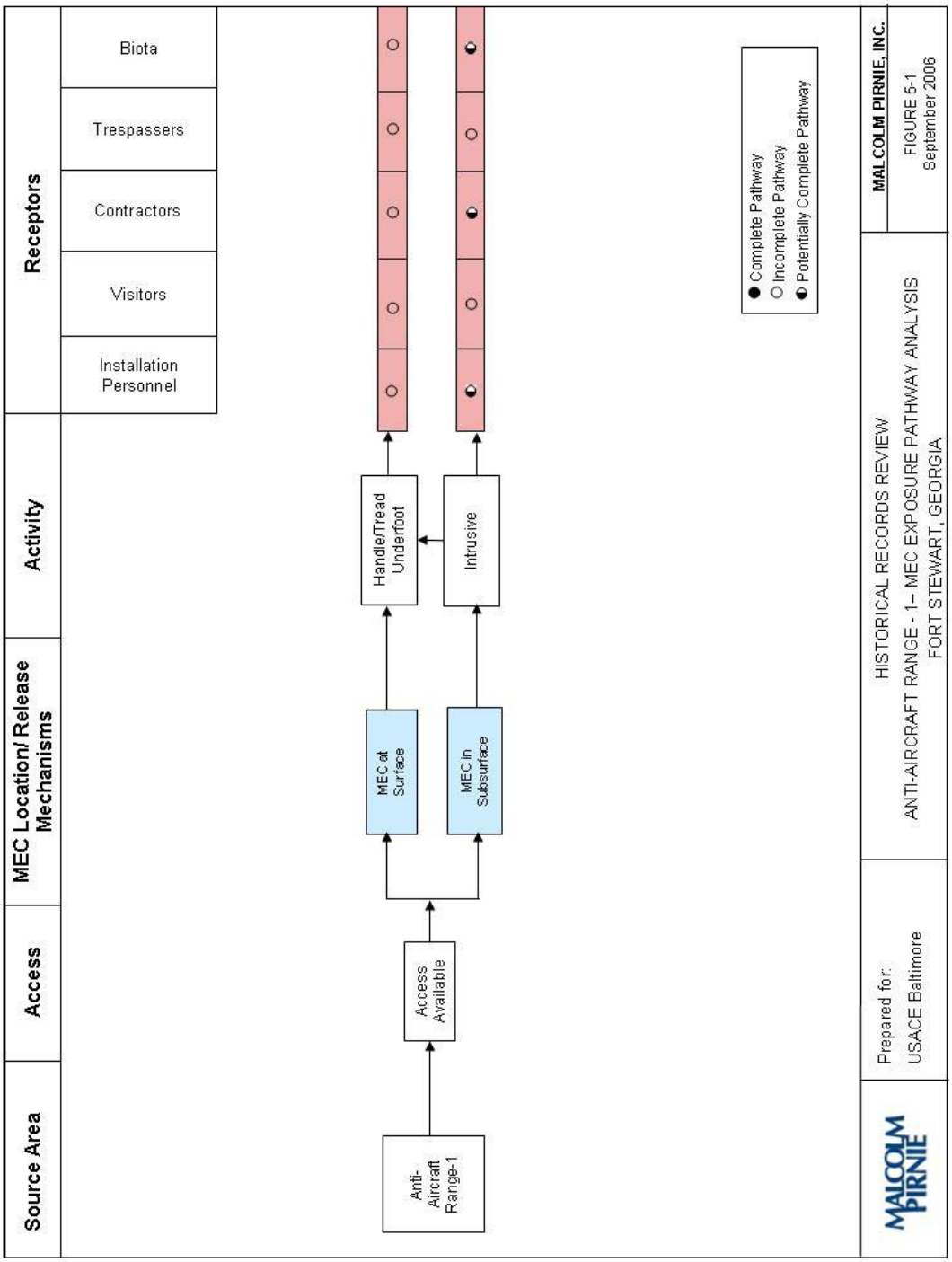
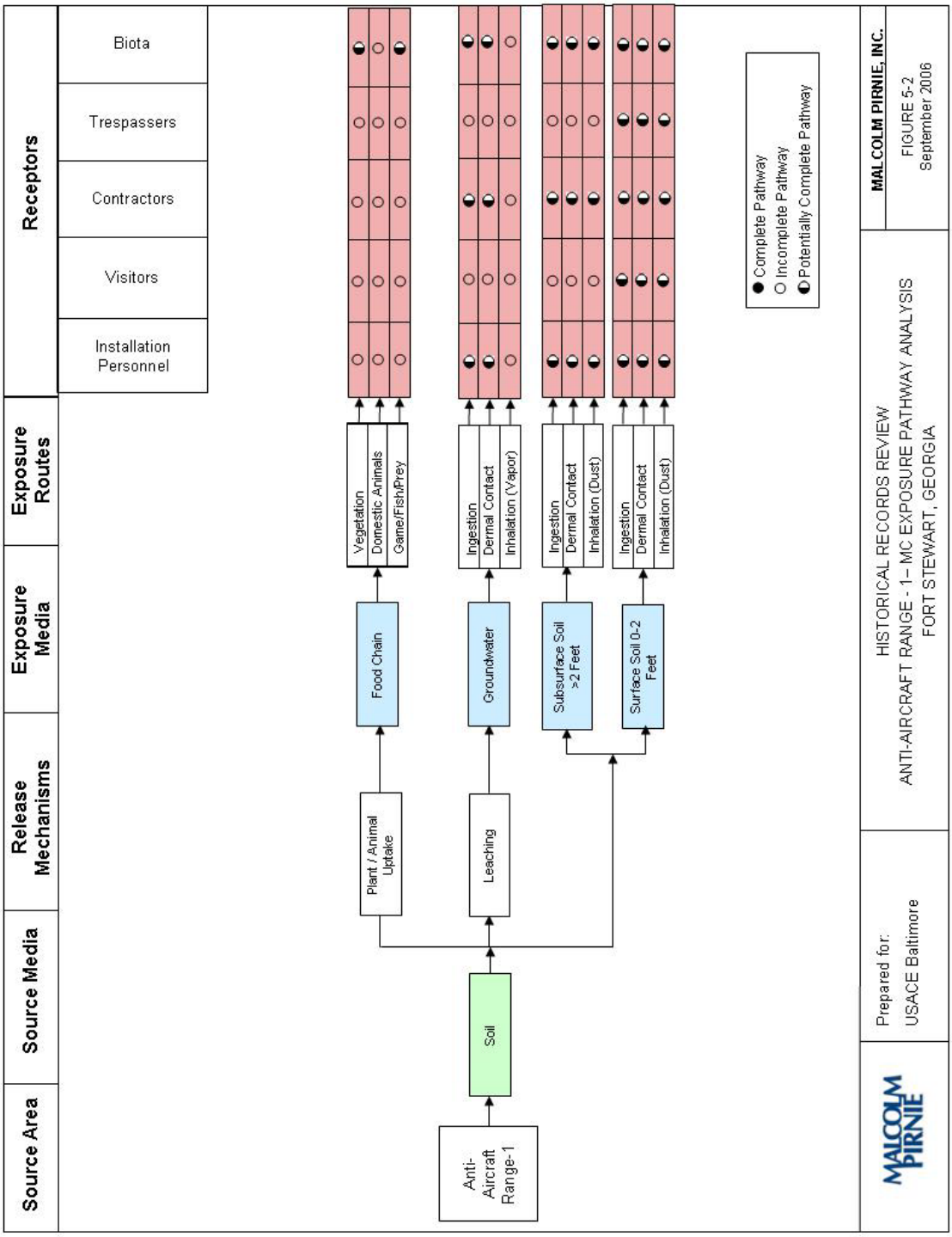


Figure 5-2: MC Pathway Analysis - Anti-Aircraft Range – 1



5.2.2 Anti-Aircraft Range 90-mm - 2

5.2.2.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-7:

Table 5-7: Site Profile – Anti-Aircraft Range 90-mm - 2

Information Needs	CSM Findings
Area and Layout	Approximately 77 acres Located in the southern portion of the installation Located approximately 5000 m northwest of the cantonment area
Structures	Ammunition supply point 42 buildings
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped area S: Undeveloped area E: Undeveloped area W: Undeveloped area
Security	Fences and Guards

5.2.2.2 Physical Profile

The site-specific physical profile is presented in Table 5-8:

Table 5-8: Physical Profile – Anti-Aircraft Range 90-mm - 2

Information Needs	CSM Findings
Topography	Approximately 20 m amsl Flat, level terrain
Soil	Sand-silt/sand-clay
Hydrology	None
Vegetation	Developed property Few grasses

5.2.2.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-9:

Table 5-9: Land Use and Exposure Profile - Anti-Aircraft Range 90-mm - 2

Information Needs	CSM Findings
Current Use	Ammunition supply point
Current Human Receptors	Installation personnel Contractors Trespassers
Potential Future Use	Same as current use
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	None

5.2.2.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-10:

Table 5-10: Ecological Profile - Anti-Aircraft Range 90-mm - 2

Information Needs	CSM Findings
Habitat Type	None Forested area adjacent to site
Degree of Disturbance	High; developed property

5.2.2.5 Munitions/Release Profile

5.2.2.5.1 Munitions Types

Table 5-11 presents a summary of MEC types that are expected to exist within Anti-Aircraft Range 90-mm - 2 based on the information collected for this HRR. The mechanisms by which the MEC may have been released into the environment are also presented in Table 5-11. The typical release mechanisms for Anti-Aircraft Range 90-mm - 2 were intentional activities, such firing munitions into a target area, or unintentional activities, such as munitions malfunctioning.

Table 5-11: Summary of Potential MEC Types – Anti-Aircraft Range 90-mm - 2

Potential Munitions	Primary Release Mechanism	Potential MEC
MK-2 fragmentation, M-7 grenades	Hand thrown	Partially/fully functioned grenades
M-222 and Dragon guided missiles (ground)	Munitions firing Malfunctioned munitions Discarded munitions	Partially/fully functioned missiles
37-mm HE M54, 40-mm, 40-mm HEP, 90-mm, 90-mm HE, 90-mm M71 HE projectiles	Munitions firing Malfunctioned munitions Discarded munitions	Partially/fully functioned projectiles/fuzes

Maximum Probability Penetration Depth

Table 5-12 provides the expected penetration depths for MEC for various types of soils that are expected to be found at Anti-Aircraft Range 90-mm - 2 (USACE, Engineering Manual 1110-1-4009 *Ordnance and Explosives Response*). For the Anti-Aircraft Range 90-mm - 2, the soil type is considered sand-silt/sand-clay. Therefore, the depths of penetration for Anti-Aircraft Range 90-mm - 2 are based upon the penetration depth for a loamy soil. As discussed in Section 5.2.1.5.2, these penetration depths are estimated on a worst-case scenario. Anti-Aircraft Range 90-mm - 2 was developed after its use as a range. It is currently used as an ammunition supply point. The site was filled and graded during the construction of the ammunition supply point. Thus, the depths to MEC may not be representative of the depths presented in

Table 5-12 and MEC could be encountered at any depth within the construction or fill

areas.

Table 5-12: Summary of Expected MEC Penetration Depths – Anti-Aircraft Range 90-mm - 2

Ordnance Item/Weapon	Depth of Penetration (feet bgs)		
	Sand	Loam	Clay
MK-2 fragmentation, M-7 grenades	0.0	0.0	0.0
M-222 and Dragon guided missiles (ground)	9.0	1.0	7.0
37-mm projectiles	3.9	5.2	7.9
40-mm, 40-mm HEP projectiles	0.2	0.3	0.4
90-mm, 90-mm HE, 90-mm M71 HE projectiles	0.0	7.0	1.0

5.2.2.5.2 MEC Density

A visual survey has not been conducted at this time to verify the presence or absence of MEC at the Anti-Aircraft Range 90-mm - 2; however, it is anticipated that potential MEC would be found in the surface and subsurface.

5.2.2.5.3 Munitions Debris

A visual survey was not conducted as part of this HRR; however, based on the activities that occurred at the former range, there is the potential for munitions debris items. The EOD has responded to several emergency calls in the area. They have encountered MK-2 fragmentation hand grenades, M-7 grenades, C-4 plastic explosives, and M-222 and GM Dragon missiles (FTSW0086).

5.2.2.5.4 Associated Munitions Constituents

Associated MC from MK-2 hand grenades includes: black powder (potassium nitrate, sulfur, and charcoal); TNT; CMP ABC; Tetryl; hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX); Cyclotetramethylenetetranitramine (HMX); and High Blast Explosive (HBX). Potential MC associated with M-7 grenades include Octol. Potential MC associated with M-222 and GM Dragon Missiles includes: Octol, perchlorate, pyrotechnic smoke, and a tearing agent. Potential MC associated with 37-mm, 40-mm, 40-mm HEP, 90-mm, 90-mm HE, 90-mm M71 HE projectiles include Tetryl, CMP AB, and TNT. (Refer to the appropriate Ordnance Technical Data Sheets in Appendix D.) Soil samples within the boundary of Anti-Aircraft Range 90-mm - 2 have not been analyzed for explosives or metals; therefore, it is not known whether explosives or metals are present in concentrations that exceed regulatory levels. There is one potable water well located just south of the boundary of the MRS. It is unknown if this is currently used as drinking water.

5.2.2.5.5 Transport Mechanisms/Migration Routes

The primary transport mechanisms identified for Anti-Aircraft Range 90-mm - 2 include:

Erosion: Anti-Aircraft Range 90-mm - 2 is a heavily developed area; therefore, erosion is not expected in this area and is not a factor in transporting and migrating possible MC contaminated soil.

Soil Disturbance: The current degree of disturbance is relatively high, as the area has been developed and cleared since the range was used. Future development could unveil potential MC that are in the surface or subsurface.

Infiltration: Based on the soil types associated with Anti-Aircraft Range 90-mm - 2, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.2.6 Pathway Analysis

5.2.2.6.1 MEC

Based on the historical use of the site as a 90-mm anti-aircraft range fan, the potential exists for MEC to be present on the site. The potential for MEC to exist on the surface; however, it is unlikely, as the site is currently an ammunition supply point and is well maintained (mowed). As illustrated in the Exposure Pathway Analysis for MEC (Figure 5-3), the pathway for all human and ecological receptors are potentially complete as the potential for these receptors to encounter MEC on the surface exists. Potentially complete pathways for installation personnel, contractors, and biota for MEC in the subsurface exist as these receptors have the potential to conduct intrusive activities. The pathway for MEC in the subsurface is incomplete for all other receptors.

5.2.2.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-4), soil and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Based on this, potentially complete exposure pathways may exist for surface and subsurface soil and groundwater through various exposure routes for both human receptors and biota.

<u>Food Chain</u>

Potentially complete pathways to MC in the source media through uptake into vegetation exist for grazing/foraging biota. This exposure pathway is incomplete for all other receptors. As there are no domestic animals on FTSW, the pathway to MC in the source media through this exposure route is incomplete for all receptors. The pathway to MC in

the source media through the game/fish/prey exposure route is potentially complete for biota. This exposure pathway is incomplete for all other receptors as their activities are not expected to include hunting.

Groundwater

Precipitation infiltration may provide for contaminant mobility into the shallow or surficial groundwater aquifer. However, based on a review of hydrogeological data (Section 5.1.2.5), it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW. Receptor contact with groundwater is possible if the soil is disturbed through excavation or construction activities, creating possible migration routes/mechanisms for MC in shallow groundwater. As such, installation personnel, contractors, and biota have potentially complete pathways to MC in subsurface soil and/or shallow groundwater through the (incidental) ingestion and dermal contact exposure routes. Given that it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW, the dermal and ingestion exposure routes are incomplete for all other receptors. Since the upper aquifer is not used as a potable water source and MC are typically not volatile, the inhalation (vapor) exposure route is incomplete for all receptors.

Subsurface Soil

Since the potential exists for MC in the subsurface soil in the Anti-Aircraft Range 90-mm - 2 area, receptor contact with subsurface soil is possible if the soil is disturbed through excavation or construction activities, creating possible receptor pathways to MC in subsurface soils. As such, installation personnel, contractors, and biota have potentially complete pathways to MC in subsurface soil through the (incidental) ingestion, dermal contact, and inhalation (dust) exposure routes. Trespassers would not be expected to participate in any intrusive activities; therefore, these exposure routes are incomplete for this receptor.

Surface Soil

All human and ecological receptors within the Anti-Aircraft Range 90-mm - 2 area may be exposed to surface soil during daily activities. Therefore, the pathways to MC in surface soil through the (incidental) ingestion, dermal contact, and inhalation of dust exposure routes are potentially complete for all receptors.

Figure 5-3: MEC Pathway Analysis - Anti-Aircraft Range 90-mm - 2

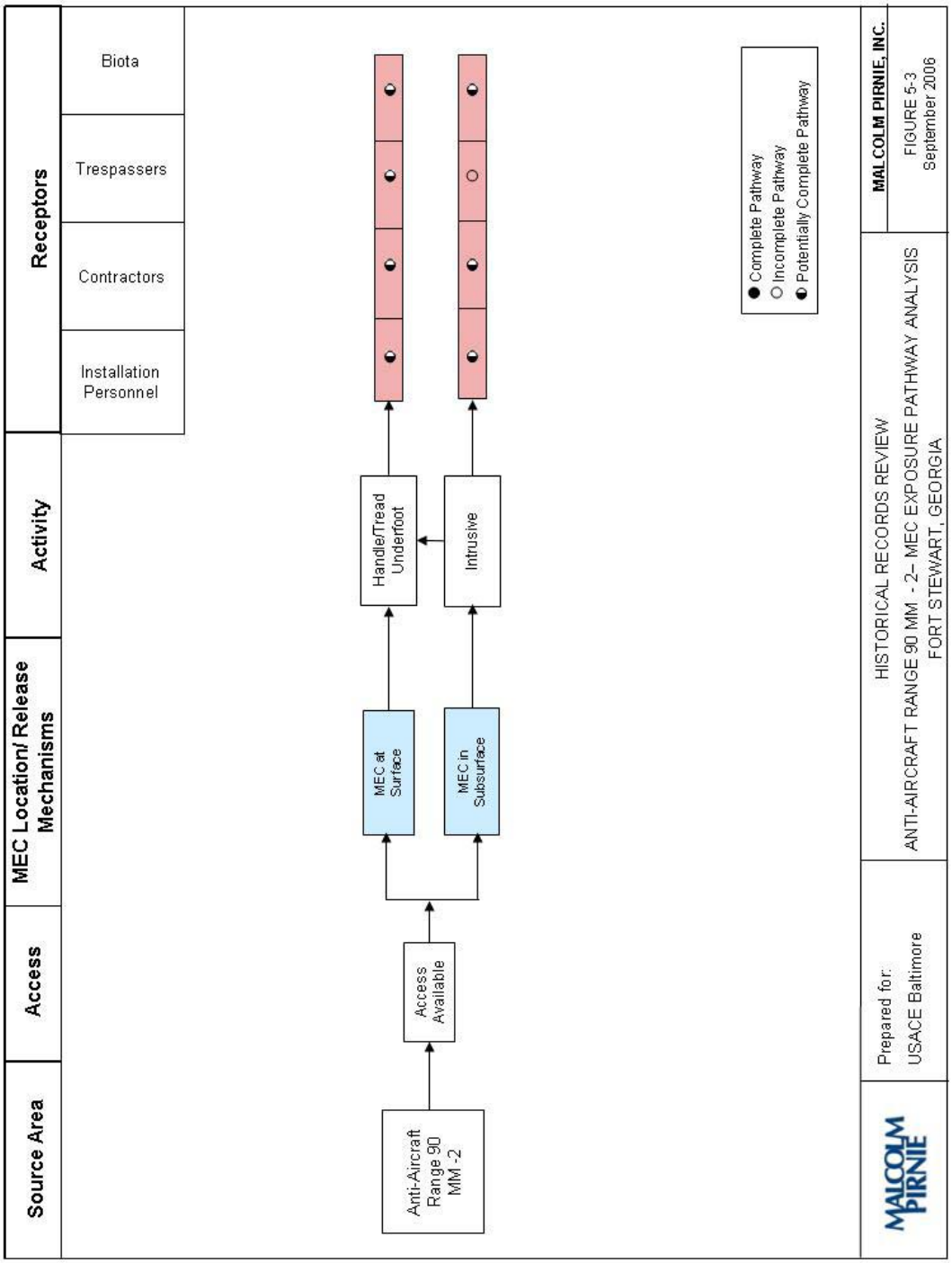
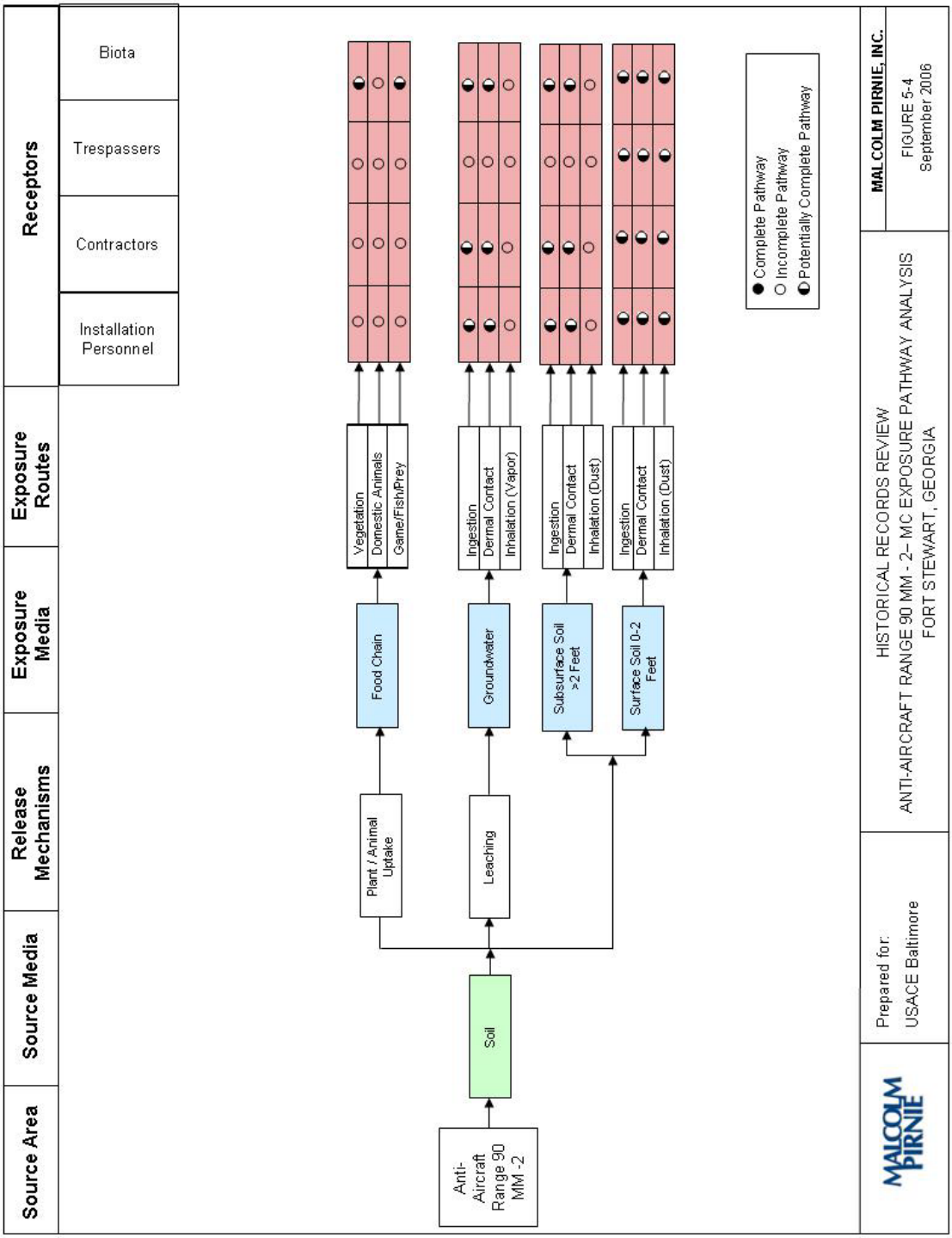


Figure 5-4: MC Pathway Analysis - Anti-Aircraft Range 90-mm - 2



5.2.3 Anti-Tank Range 90-mm

5.2.3.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-13:

Table 5-13: Site Profile - Anti-Tank Range 90-mm

Information Needs	CSM Findings
Area and Layout	Approximately 124 acres Located along the southern portion of the installation Located approximately 1,000 m northwest of the cantonment area
Structures	Active landfill (since 1963) Four buildings
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped property S: Unidentified road E: Undeveloped property W: Mill Creek
Security	Fences

5.2.3.2 Physical Profile

The site-specific physical profile is presented in Table 5-14:

Table 5-14: Physical Profile - Anti-Tank Range 90-mm

Information Needs	CSM Findings
Topography	Approximately 20 m amsl Flat, level terrain
Soil	Sand-silt/sand-clay
Hydrology	Taylor's Creek flows to the north of the site Mill Creek flows to the west and south of the site Engineer's Pond is located southeast of the site
Vegetation	Few grasses

5.2.3.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-15:

Table 5-15: Land Use and Exposure Profile - Anti-Tank Range 90-mm

Information Needs	CSM Findings
Current Land Use	Active landfill (since 1963)
Current Human Receptors	Installation personnel Contractors Trespassers Hunters/fisherman
Potential Future Use	Same as current use
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	None

5.2.3.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-16:

Table 5-16: Ecological Profile - Anti-Tank Range 90-mm

Information Needs	CSM Findings
Habitat Type	None
Degree of Disturbance	High; cleared property used currently as a landfill

5.2.3.5 Munitions/Release Profile

5.2.3.5.1 Munitions Types

Table 5-17 presents a summary of potential munitions types that are expected to exist within Anti-Tank Range 90-mm based on the information collected for this HRR. The mechanisms by which munitions may have been released into the environment are also presented in. The typical release mechanisms for the Anti-Tank Range 90-mm were intentional activities, such firing munitions, or unintentional activities, such as munitions falling outside of the target area.

Table 5-17: Summary of Potential Munitions Types – Anti-Tank Range 90-mm

Potential Munitions	Primary Release Mechanism	Potential MEC
37-mm HE M54, 40-mm, 40-mm HEP, 90-mm, 90-mm HE 90-mm M71 HE projectiles	Munitions firing Malfunctioned munitions Discarded munitions	Partially/fully functioned projectiles/fuzes
0.22-cal, 0.30-cal, 0.30-cal (with tracer), 0.45-cal, 0.50-cal, 0.50-cal (with tracer), 0.50 cal (armor piercing) small arms	Munitions firing Malfunctioned munitions Discarded munitions	No MEC expected

5.2.3.5.2 Maximum Probability Penetration Depth

Since the entire site is an active landfill, the depth at which MEC could be located depends on the amount (depth) of fill located at the site. Therefore, MEC could be encountered at any depth within the landfill

5.2.3.5.3 MEC Density

A visual survey has not been conducted at this time to verify the presence or absence of MEC at the Anti-Tank Range 90-mm. However, the entire area is an active landfill, so it is anticipated that any potential MEC could be found in the subsurface.

5.2.3.5.4 Munitions Debris

A visual survey was not conducted as part of this HRR; however, based on the activities that occurred at the former range, there is the potential for munitions debris items. No MEC or munitions debris are known to have been reported at the range. No EOD calls have been reported at this site (FTSW00086).

5.2.3.5.5 Associated Munitions Constituents

Potential MC associated with 37-mm HE M54, 40-mm, 40-mm HEP, 90-mm, 90-mm HE, 90-mm M71 HE projectiles include: Tetryl, CMP AB, and TNT. Potential MC associated with 0.22-cal, 0.30-cal, 0.45-cal, and 0.50-cal small arms munitions include: lead, antimony, tin, arsenic, copper, zinc, iron, strontium, magnesium, and lead/styphante/lead azide. (Refer to the appropriate Ordnance Technical Data Sheets in Appendix D.) Soil samples within the boundary of Anti-Tank Range 90-mm have not been analyzed for explosives or metals; therefore, it is unknown whether explosives or metals are present in concentrations that exceed regulatory levels.

5.2.3.5.6 Transport Mechanisms/Migration Route

The primary transport mechanisms identified for the Anti-Tank Range 90-mm include:

Erosion: Anti-Tank Range 90-mm may potentially be disturbed by flooding of the Mill Creek and other creeks, which could result in erosion.

Soil Disturbance: The current degree of disturbance is relatively high, as the area has been developed and the forest has been cleared. Future development could unveil potential MC that are in the surface or subsurface.

Infiltration: Based on the soil types associated with Anti-Tank Range 90-mm, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.3.6 Pathway Analysis

5.2.3.6.1 MEC

Based on the historical use of the site as a buffer area near the firing point of an anti-tank range and an anti-aircraft range and a portion of a small arms range, the potential exists for MEC to be present on the site. However, MEC on the surface are not expected, as the site is currently a landfill. As illustrated in the Exposure Pathway Analysis for MEC (Figure 5-5), no complete or potentially complete pathways for human or ecological receptors for MEC on the surface are expected to exist. The munitions related use of the Anti-Tank Range 90-mm area ceased in 1947, and in 1963, the installation began using the site as a landfill. As a result, any potential MEC remaining on the site are expected to be buried deep below the waste that has been placed in the landfill. Due to the extremely high cost of excavating a landfill, intrusive activities at the site are highly unlikely. Therefore, no complete or potentially complete pathways exist for MEC in the subsurface for human or ecological receptors.

5.2.3.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-6), soil and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Additionally, since the site has been used as a landfill for approximately 43 years, it is extremely difficult to directly attribute any potential impacts to the former use of this site as a range area. Currently, the installation conducted groundwater monitoring at the landfill on a semi-annual basis; however, it is not possible to decipher if the impact from potential munitions contributes to the sampling results (FTSW00101). Based on the fact that area has been maintained as a landfill for approximately 43 years, no complete or potentially complete pathways for MC in the surface/subsurface soil or groundwater exist for human or ecological receptors.

Figure 5-5: MEC Pathway Analysis - Anti-Tank Range 90-mm

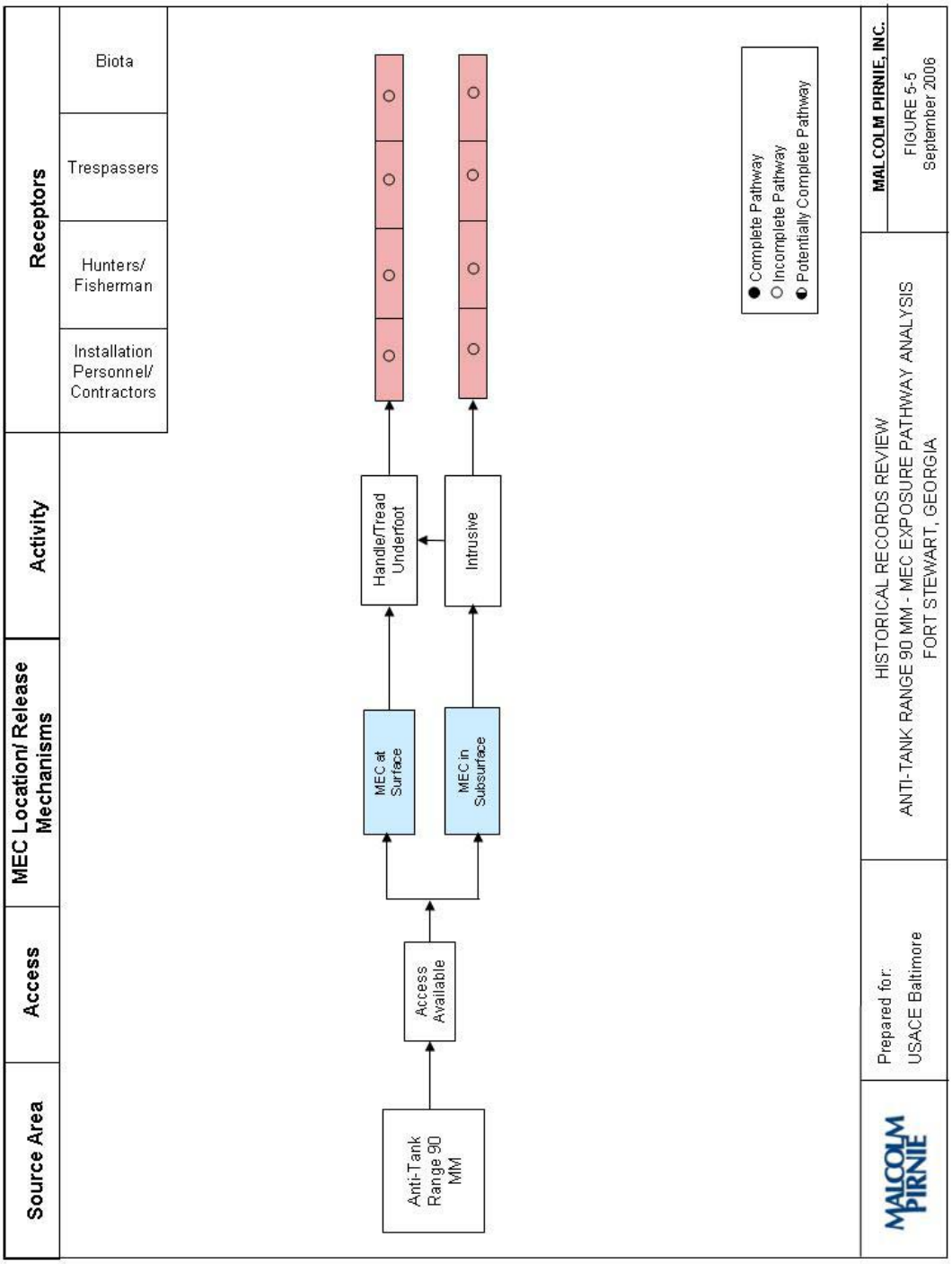
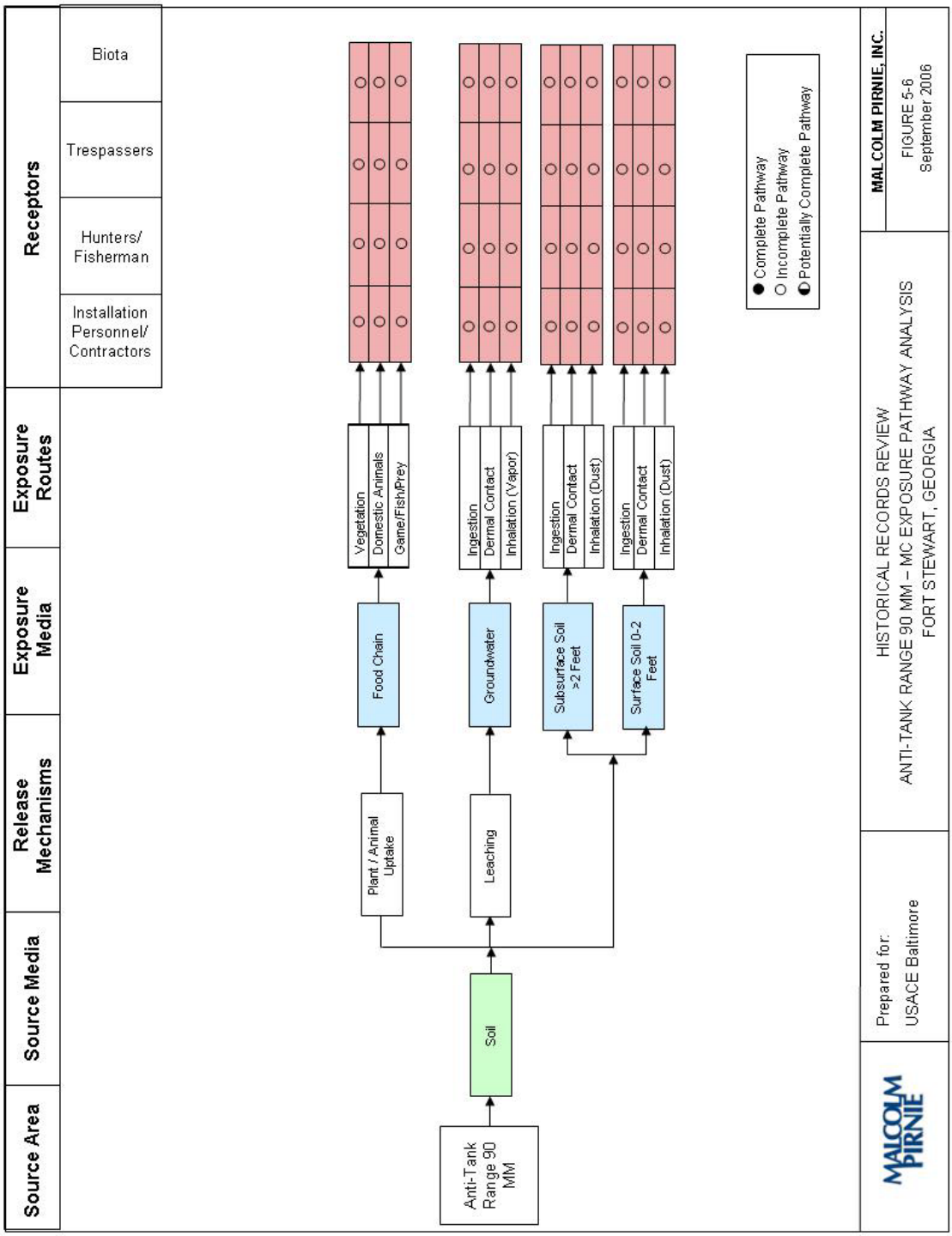


Figure 5-6: MC Pathway Analysis - Anti-Tank Range 90-mm



5.2.4 Hand Grenade Course

5.2.4.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-18:

Table 5-18: Site Profile – Hand Grenade Course

Information Needs	CSM Findings
Area and Layout	Approximately 67 acres Located along the southern portion of the installation Located approximately 5,000 m northwest of the cantonment area
Structures	None
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped property/GA Highway 144 S: Undeveloped property E: Undeveloped property W: Undeveloped property
Security	Signs

5.2.4.2 Physical Profile

The site-specific physical profile is presented in Table 5-19:

Table 5-19: Physical Profile – Hand Grenade Course

Information Needs	CSM Findings
Topography	Approximately 20 m amsl Flat, level terrain
Soil	Sand-silt/sand-clay
Hydrology	None
Vegetation	Heavily forested area

5.2.4.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-20:

Table 5-20: Land Use and Exposure Profile – Hand Grenade Course

Information Needs	CSM Findings
Current Land Use	Undeveloped property
Current Human Receptors	Authorized Installation personnel Authorized Contractors Trespassers
Potential Future Use	Same as current use
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	Forested land acts as a habitat

5.2.4.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-21:

Table 5-21: Ecological Profile - Hand Grenade Course

Information Needs	CSM Findings
Habitat Type	Forested and grassy area
Degree of Disturbance	Low; forested area

5.2.4.5 Munitions/Release Profile

5.2.4.5.1 Munitions Types

Table 5-22 presents a summary of MEC types that are expected to exist within the Hand Grenade Course based on the information collected for this HRR. The mechanisms by which the MEC may have been released into the environment are also presented in this table. The typical release mechanisms for the Hand Grenade Course were intentional activities, such as throwing a grenade into a target area or firing munitions, and unintentional activities, such as grenades or munitions falling outside the target area. The Hand Grenade course was overlapped by various other areas including: an impact area for a 90-mm anti-tank range, an impact area for a 40-mm anti-aircraft range, and a firing point for a small arms range; therefore, the MEC associated with these areas are also MEC types that are expected to exist at the Hand Grenade Course.

Table 5-22: Summary of Potential MEC Types – Hand Grenade Course

Potential Munitions	Primary Release Mechanism	Potential MEC
MK-2 fragmentation grenades M-7 grenades	Hand thrown	Partially/fully functioned grenades
37-mm HE M54, 40-mm, 40-mm HEP, 90-mm, 90-mm HE, 90-mm M71 HE projectiles	Munitions firing Malfunctioned munitions Discarded munitions	Partially/fully functioned projectiles/fuzes
0.22-cal, 0.30-cal, 0.30-cal (with tracer), 0.45-cal, 0.50-cal, 0.50-cal (with tracer), 0.50-cal (armor piercing) small arms	Munitions firing Malfunctioned munitions Discarded munitions	No MEC expected

5.2.4.5.2 Maximum Probability Penetration Depth

Table 5-23 provides the expected penetration depths for various types of soils for MEC that are expected to be found at Hand Grenade Course (USACE, Engineering Manual 1110-1-4009 *Ordnance and Explosives Response*). No MEC are expected from the small arms range; therefore, a penetration depth is not be presented for small arms. For the Hand Grenade Course, the soil type is considered sand-silt/sand-clay. Therefore, the depth of penetration for Hand Grenade Course is based upon the penetration depth for a loamy soil. As noted in Section 5.2.1.5.2, these penetration depths are estimated on a worst-case scenario.

Table 5-23: Summary of Expected MEC Penetration Depths – Hand Grenade Course

Ordnance Item/Weapon	Depth of Penetration (feet bgs)		
	Sand	Loam	Clay
MK-2 fragmentation grenades M-7 grenade	0.0	0.0	0.0
37-mm projectiles	3.9	5.2	7.9
40-mm, 40-mm HEP projectiles	0.2	0.3	0.4
90-mm, 90-mm HE, 90-mm M71 HE projectiles	0.0	7.0	1.0

5.2.4.5.3 MEC Density

A visual survey has not been conducted at this time to verify the presence or absence of MEC at the Hand Grenade Course. However, the area is mostly forested, so it is anticipated that any potential MEC could be found on the surface or subsurface.

5.2.4.5.4 Munitions Debris

A visual survey was not conducted as part of this HRR; however, based on the activities that occurred at the former range, there is the potential for munitions debris items. No MEC or munitions debris are known to have been reported at the range. No EOD calls have been reported at the site.

5.2.4.5.5 Associated Munitions Constituents

Associated MC from MK-2 fragmentation and M-7 hand grenades includes: black powder (potassium nitrate, sulfur, and charcoal), CN, TNT, CMP ABC, Tetryl, RDX, HMX, HBX, and PETN. Potential MC associated with 40-mm, 40-mm HEP, 90-mm, 90-mm HE, 90-mm M71 HE projectiles include: Tetryl, CMP AB, and TNT. Potential MC associated with 0.22-cal, 0.30-cal, 0.45-cal, and 0.50-cal small arms munitions include: lead, antimony, tin, arsenic, copper, zinc, iron, strontium, magnesium, and lead styphante/lead azide. (Refer to the appropriate Ordnance Technical Data Sheets in Appendix D.) Soil samples within the boundary of Hand Grenade Course have not been analyzed for explosives or metals; therefore, it is not known whether explosives or metals are present in concentrations that exceed regulatory levels.

5.2.4.5.6 Transport Mechanisms/Migration Routes

The primary transport mechanisms identified for the Hand Grenade Course include:

Erosion: The Hand Grenade Course is a heavily forested area; therefore, erosion is not expected in this area and is not a factor in transporting and migrating possible MC contaminated soil.

Soil Disturbance: The current degree of disturbance is low. Future development could unveil potential MC that are in the surface or subsurface.

Infiltration: Based on the soil types associated with the Hand Grenade Course, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.4.6 Pathway Analysis

5.2.4.6.1 MEC

Based on the multiple historical uses of the site as a hand grenade course, an impact area of an anti-tank range and an anti-aircraft range, and a firing point of a small arms range, the potential exists for MEC to be present on the site. As illustrated in the Exposure

Pathway Analysis for MEC (Figure 5-7), potentially complete pathways to MEC on the surface exist for human and ecological receptors. Potentially complete pathways for authorized installation personnel, authorized contractors, and biota for MEC in the subsurface exist as these receptors have the potential to conduct intrusive activities. The pathway for MEC in the subsurface is incomplete for trespassers. These potentially complete pathways are currently managed for authorized installation personnel and authorized contractors through the use of signage.

5.2.4.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-8), soil and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Based on this, potentially complete exposure pathways may exist for surface and subsurface soil and groundwater through various exposure routes for human receptors and biota.

Food Chain

Potentially complete pathways to MC in the source media through uptake into vegetation exist for grazing/foraging biota. This exposure pathway is incomplete for all other receptors. As there are no domestic animals on FTSW, the pathway to MC in the source media through this exposure route is incomplete for all receptors. The pathway to MC in the source media through the game/fish/prey exposure route is potentially complete for biota. This exposure pathway is incomplete for all other receptors as their activities are not expected to include hunting.

Groundwater

Precipitation infiltration may provide for contaminant mobility into the shallow or surficial groundwater aquifer. However, based on a review of hydrogeological data (Section 5.1.2.5), it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW. Receptor contact with groundwater is possible if the soil is disturbed through excavation or construction activities, creating possible migration routes/mechanisms for MC in shallow

groundwater. As such, authorized installation personnel, authorized contractors, and biota have potentially complete pathways to MC in subsurface soil and/or shallow groundwater through the (incidental) ingestion and dermal contact exposure routes. Given that it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW, the dermal and ingestion exposure routes are incomplete for all other receptors. Since the upper aquifer is not used as a potable water source and MC are typically not volatile, the inhalation (vapor) exposure route is incomplete for all receptors.

Subsurface Soil

Since the potential exists for MC in the subsurface soil in the Hand Grenade Course area, receptor contact with subsurface soil is possible if the soil is disturbed through excavation or construction activities, creating possible receptor pathways to MC in subsurface soils. As such, authorized installation personnel, authorized contractors, and biota have potentially complete pathways to MC in subsurface soil through the (incidental) ingestion, dermal contact, and inhalation (dust) exposure routes. Trespassers would not be expected to participate in any intrusive activities; therefore, these exposure routes are incomplete for these receptors.

Surface Soil

All human and ecological receptors within the Hand Grenade Course area may be exposed to surface soil during daily activities. Therefore, the pathways to MC in surface soil through the (incidental) ingestion, dermal contact, and inhalation of dust exposure routes are potentially complete for all receptors.

Figure 5-7: MEC Pathway Analysis - Hand Grenade Course

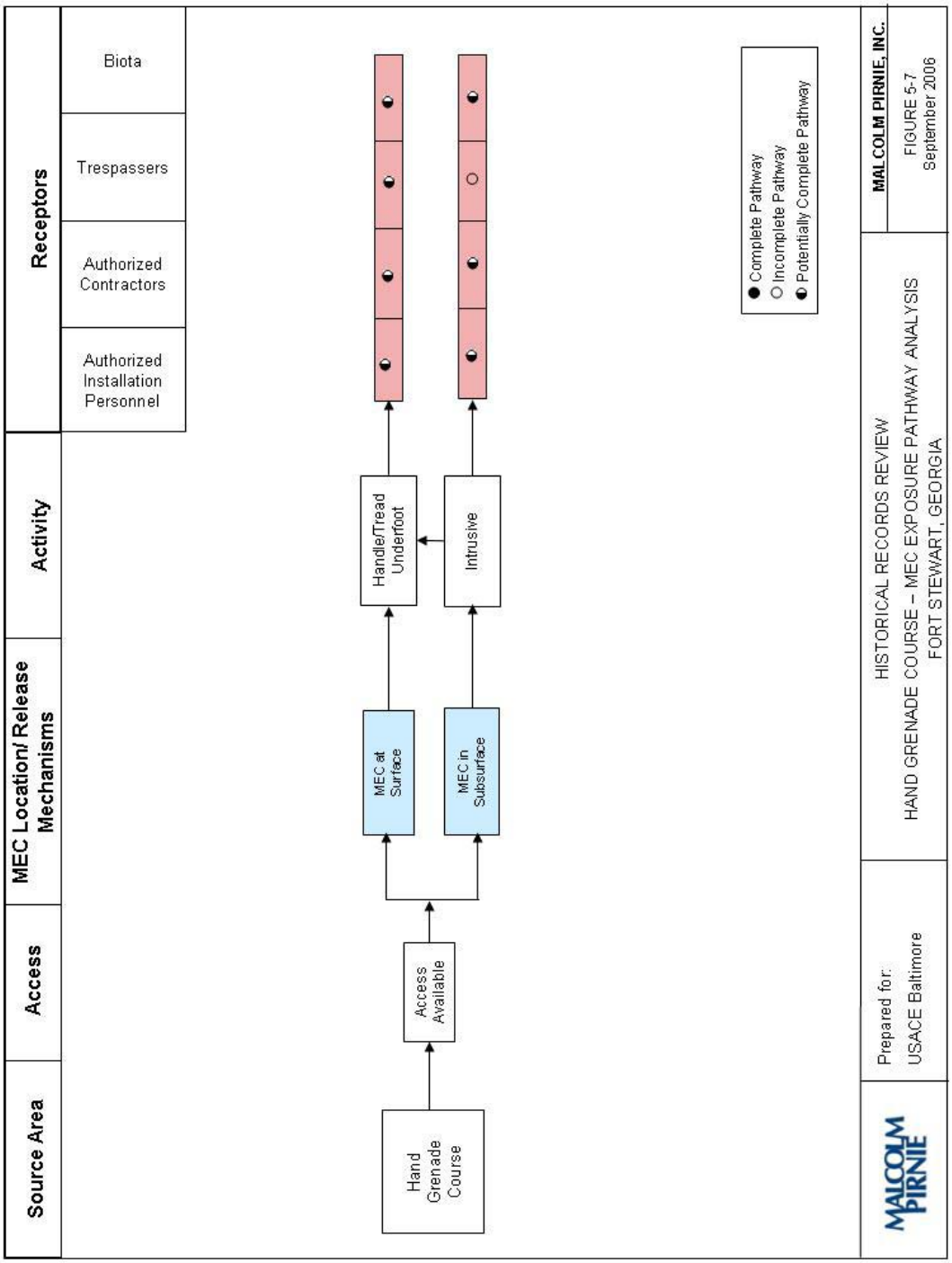
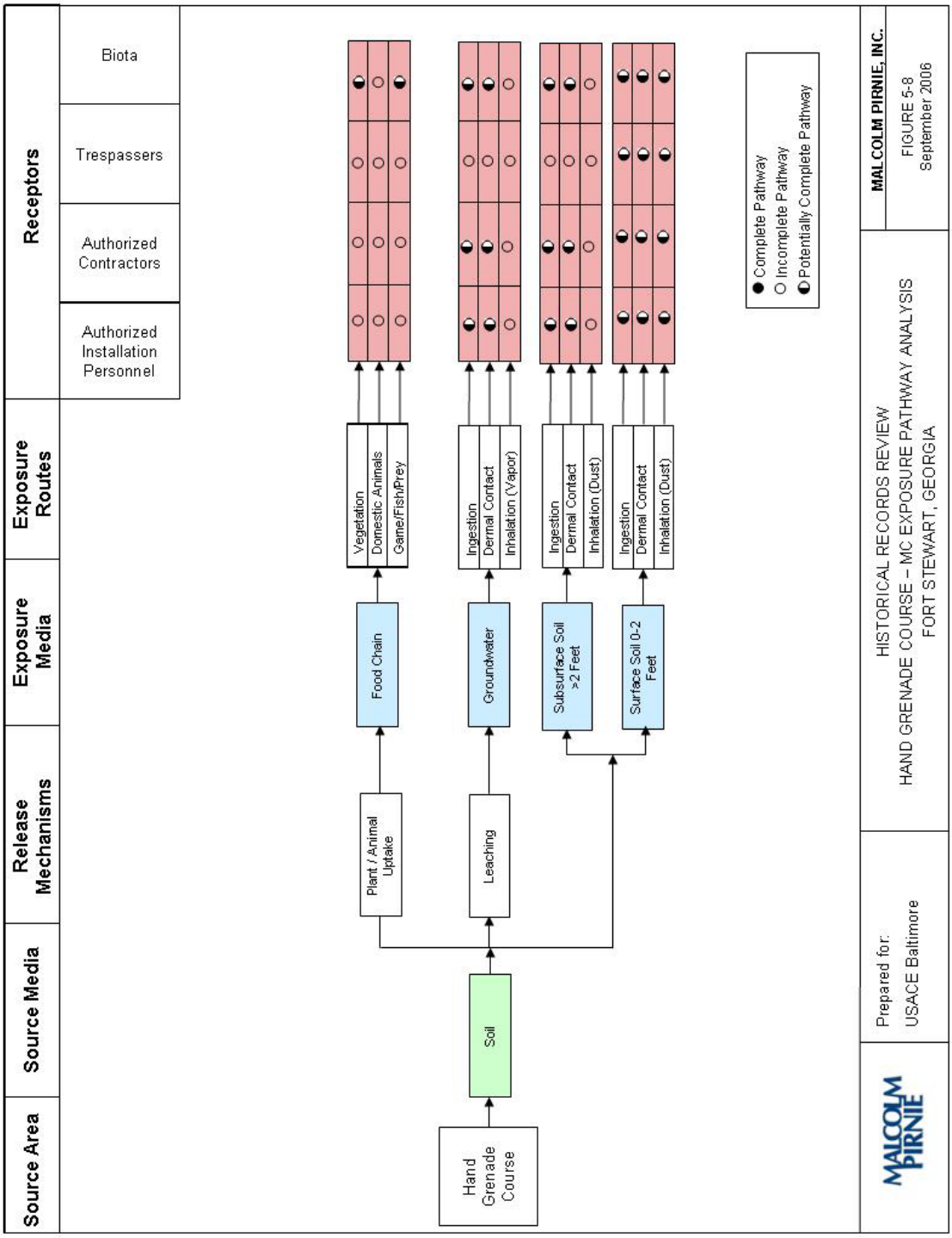


Figure 5-8: MC Pathway Analysis - Hand Grenade Course



5.2.5 Small Arms Range - 1

5.2.5.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-24:

Table 5-24: Site Profile – Small Arms Range - 1

Information Needs	CSM Findings
Area and Layout	Approximately 136 acres Located along the southern portion of the installation Located approximately 9,000 m northeast of the cantonment area
Structures	31 buildings
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped property/Buildings 19EVN, 20EVN S: Undeveloped property E: Undeveloped property/Buildings 19104, 19108 W: Unidentified road/GA Highway 144
Security	None

5.2.5.2 Physical Profile

The site-specific physical profile is presented in Table 5-25:

Table 5-25: Physical Profile - Small Arms Range - 1

Information Needs	CSM Findings
Topography	Approximately 10 m amsl Flat, level
Soil	Sand-silt/sand-clay
Hydrology	Evan's Field Pond located northwest of the site
Vegetation	Some forested areas Some grasses

5.2.5.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-26:

Table 5-26: Land Use and Exposure Profile - Small Arms Range - 1

Information Needs	CSM Findings
Current Land Use	Evans Heliport/Airfield
Current Human Receptors	Installation personnel Visitors Contractors Trespassers
Potential Future Use	Same as current use
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	Partial forest habitat Grassland habitat

5.2.5.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-27:

Table 5-27: Ecological Profile - Small Arms Range - 1

Information Needs	CSM Findings
Habitat Type	Some forest and grassland
Degree of Disturbance	Medium; forest and grasses remain on portion of site

5.2.5.5 Munitions/Release Profile

5.2.5.5.1 Munitions Types

Table 5-28 presents a summary of the types of munitions that could be expected to exist based on information collected during this HRR process. Also presented in this table are the mechanisms by which munitions would be expected to be released into the environment if present. It is important to note that because this area was used as a small arms range, MEC are not expected, and the primary concern would be associated with MC.

Table 5-28: Summary of Potential Munitions Types – Small Arms Range - 1

Potential Munitions	Primary Release Mechanism
0.22-cal, 0.30-cal, 0.30-cal (with tracer), 0.45-cal, 0.50-cal, 0.50-cal (with tracer), 0.50-cal (armor piercing) small arms	Munitions firing Malfunctioned munitions Discarded munitions

5.2.5.5.2 MEC Density

Due to the nature of small arms ammunition, MEC are not expected.

5.2.5.5.3 Munitions Debris

A visual survey was not conducted as part of this HRR; however, based on the activities that occurred at the former range, there is the potential for munitions debris items. Potential munitions debris associated with small arms ammunition include spent projectiles, fragments, and shell casings. No EOD calls have been reported at this site (FTSW00086).

5.2.5.5.4 Associated Munitions Constituents

Potential MC associated with 0.22-cal, 0.30-cal, 0.45-cal and 0.50-cal small arms include: lead, antimony, tin, arsenic, copper, zinc, iron, strontium, magnesium, and lead styphante/lead azide. (Refer to appropriate Ordnance Technical Data Sheets in Appendix D.) Soil samples within the boundary of Small Arms Range - 1 have not been analyzed for metals; therefore, it is not known whether metals are present in concentrations that exceed regulatory levels.

5.2.5.5.5 Transport Mechanisms/Migration Routes

The primary transport mechanisms identified for Small Arms Range - 1 include:

Erosion: Small Arms Range - 1 is mostly developed land; therefore, erosion is not expected in this area and is not a factor in transporting and migrating possible MC contaminated soil.

Soil Disturbance: The current degree of disturbance is relatively high, as most of the area has been developed and cleared since the range was used. Future development, especially in the forested area, could unveil potential MC that are in the surface or subsurface.

Infiltration: Based on the soil types associated with Small Arms Range - 1, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.5.6 Pathway Analysis

5.2.5.6.1 MEC

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at Small Arms Range - 1 as only small arms ammunition is assumed to have been used (FTSW0086, FTSW0090). MEC are not associated with small arms ranges; therefore, an Exposure Pathway Analysis was not created.

5.2.5.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-9), soil, sediment, surface water, and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Based on this, potentially complete exposure pathways may exist for surface and subsurface soil, sediment, surface water, and groundwater through various exposure routes for human receptors and biota.

Food Chain

Potentially complete pathways to MC in the source media through uptake into vegetation exist for grazing/foraging biota. This exposure pathway is incomplete for all other receptors. As there are no domestic animals on FTSW, the pathway to MC in the source media through this exposure route is incomplete for all receptors. The pathway to MC in the source media through the game/fish/prey exposure route is potentially complete for biota. This exposure pathway is incomplete for all other receptors as their activities are not expected to include hunting.

Surface Water/Sediment

The surface water/sediment exposure pathway is considered to be potentially complete for both human and ecological receptors since the Evan's Field Pond area is approximately 1,400 feet to the northwest of Small Arms Range - 1

Groundwater

Precipitation infiltration may provide for contaminant mobility into the shallow or surficial groundwater aquifer. However, based on a review of hydrogeological data (Section 5.1.2.5), it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW. Receptor contact with groundwater is possible if the soil is disturbed through excavation or construction activities, creating possible migration routes/mechanisms for MC in shallow groundwater. As such, authorized installation personnel, authorized contractors, and biota have potentially complete pathways to MC in subsurface soil and/or shallow groundwater through the (incidental) ingestion and dermal contact exposure routes. Given that it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW, the dermal and ingestion exposure routes are incomplete for all other receptors. Since the upper aquifer is not used as a potable water source and MC are typically not volatile, the inhalation (vapor) exposure route is incomplete for all receptors.

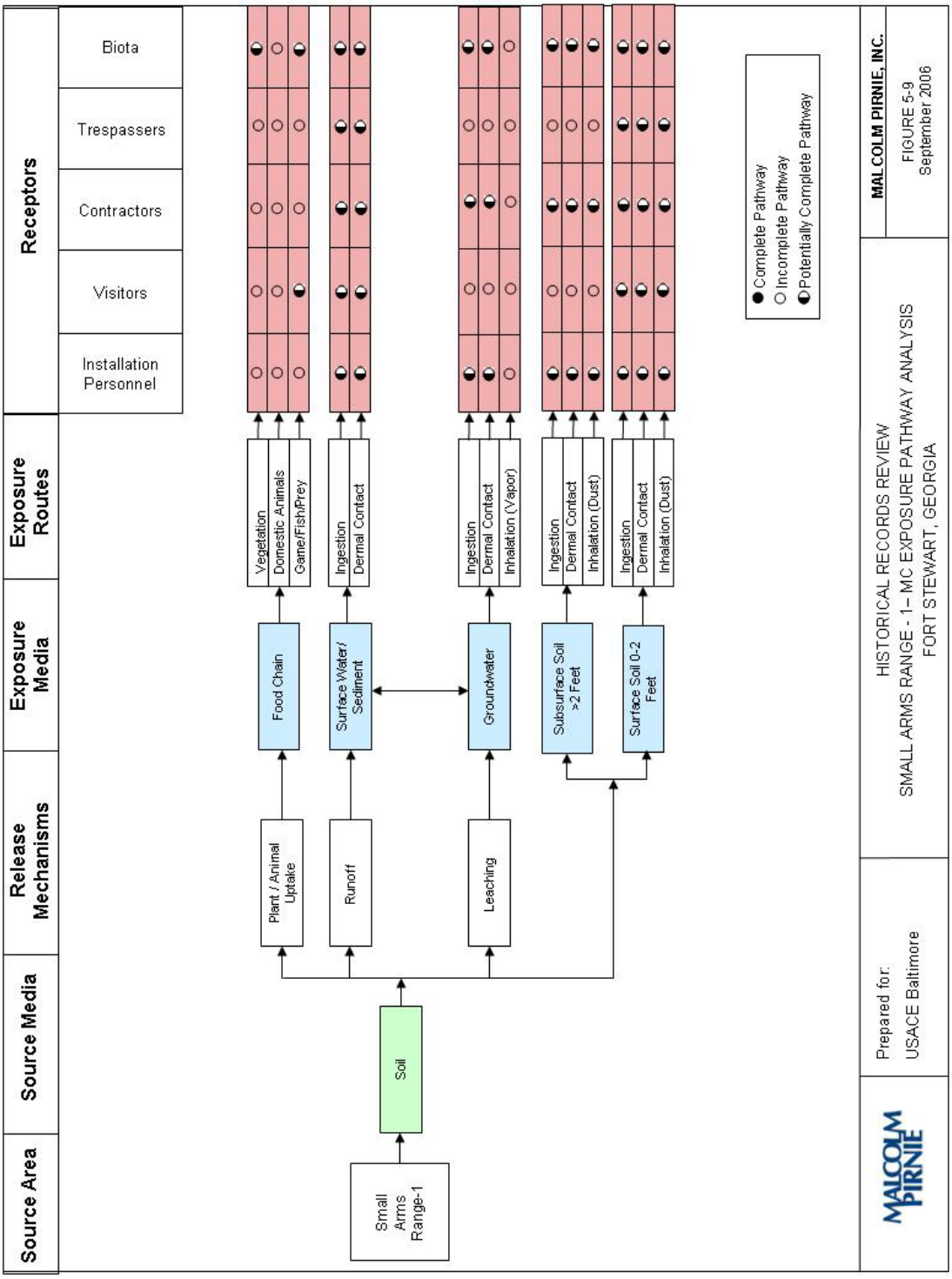
Subsurface Soil

Since the potential exists for MC in the subsurface soil in the Small Arms Range - 1 area, receptor contact with subsurface soil is possible if the soil is disturbed through excavation or construction activities, creating possible receptor pathways to MC in subsurface soils. As such, installation personnel, contractors, and biota have potentially complete pathways to MC in subsurface soil through the (incidental) ingestion, dermal contact, and inhalation (dust) exposure routes. Visitors and trespassers would not be expected to participate in any intrusive activities; therefore, these exposure routes are incomplete for these receptors.

Surface Soil

All human and ecological receptors within the Small Arms Range - 1 area may be exposed to surface soil during daily activities. Therefore, the pathways to MC in surface soil through the (incidental) ingestion, dermal contact, and inhalation of dust exposure routes are potentially complete for all receptors.

Figure 5-9: MC Pathway Analysis - Small Arms Range – 1



5.2.6 Small Arms Range - 3

5.2.6.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-29:

Table 5-29: Site Profile – Small Arms Range - 3

Information Needs	CSM Findings
Area and Layout	Approximately 32 acres Located along the southern portion of the installation Located 4,000 m northeast of the cantonment area
Structures	Five structures
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped property S: Undeveloped property E: Unidentified road W: Undeveloped property
Security	None

5.2.6.2 Physical Profile

The site-specific physical profile is presented in Table 5-30:

Table 5-30: Physical Profile - Small Arms Range - 3

Information Needs	CSM Findings
Topography	Approximately 10 m amsl Flat, level terrain
Soil	Sand-silt/sand-clay
Hydrology	Holbrook Pond covers approximately 75% of the site. Stream located northeast of site
Vegetation	Forested area and grasses Wetland vegetation

5.2.6.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-31:

Table 5-31: Land Use and Exposure Profile - Small Arms Range - 3

Information Needs	CSM Findings
Current Land Use	Pond Recreational area Undeveloped property Five buildings
Current Human Receptors	Installation personnel Contractors Recreational Users Visitors Trespassers
Potential Future Use	Same as current use
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	Pond and forested areas act as habitat

5.2.6.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-32:

Table 5-32: Ecological Profile - Small Arms Range - 3

Information Needs	CSM Findings
Habitat Type/System	Pond Forested and grassy area
Degree of Disturbance	Low; mostly Holbrook Pond and forested area, very little development

5.2.6.5 Munitions/Release Profile

5.2.6.5.1 Munitions Types

Table 5-33 presents a summary of the types of munitions that could be expected to exist

based on information collected during this HRR process. Also presented in this table are the mechanisms by which munitions would be expected to be released into the environment if present. It is important to note that because this area is suspected of being a small arms range, MEC are not expected, and the primary concern would be associated with MC.

Table 5-33: Summary of Potential Munitions Types – Small Arms Range - 3

Potential Munitions	Primary Release Mechanism
0.22-cal, 0.30-cal, 0.30-cal (with tracer), 0.45-cal, 0.50-cal, 0.50-cal (with tracer), 0.50-cal (armor piercing) small arms	Munitions firing Malfunctioned munitions Discarded munitions

5.2.6.5.2 MEC Density

Due to the nature of small arms ammunition, MEC are not expected.

5.2.6.5.3 Munitions Debris

A visual survey was not conducted as part of this HRR; however, based on the activities that occurred at the former range, there is the potential for munitions debris items. Potential munitions debris associated with small arms ammunition include spent projectiles, fragments, and shell casings. No EOD calls have been reported at this site (FTSW00086).

5.2.6.5.4 Associated Munitions Constituents

Potential MC associated with small arms estimated to be used on Small Arms Range - 3

include lead, antimony, tin, arsenic, copper, zinc, iron, strontium, magnesium, and lead styphante/lead azide. (Refer to appropriate Ordnance Technical Data Sheets in Appendix D.) Soil samples within the boundary of Small Arms Range – 3 have not been analyzed for metals; therefore, it is not known whether metals are present in concentrations that exceed regulatory levels. Currently, a potable water well is located on the southern portion of the MRS. It is unknown if this is used for drinking water.

5.2.6.5.5 Transport Mechanisms/Migration Routes

The primary transport mechanisms identified for Small Arms Range - 3 include:

Erosion: Small Arms Range - 3 is mostly a pond; therefore, erosion is possible in this area and is a factor in transporting and migrating possible MC contaminated soil.

Soil Disturbance: The current degree of disturbance is relatively low, as most of the area has not been developed since the range was used. More development, especially in the forested area, could unveil potential MC that are in the surface or subsurface.

Infiltration: Based on the soil types associated with Small Arms Range - 3, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.6.6 Pathway Analysis

5.2.6.6.1 MEC

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at Small Arms Range - 3 as only small arms ammunition is assumed to have been used (FTSW0086). MEC are not associated with small arms ranges; therefore, an Exposure Pathway Analysis was not created.

5.2.6.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-10), soil, sediment, surface water, and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Based on this, potentially complete exposure pathways may exist for surface and subsurface soil, sediment, surface water, and groundwater through various exposure routes for human receptors and biota.

Food Chain

Potentially complete pathways to MC in the source media through uptake into vegetation exist for grazing/foraging biota. This exposure pathway is incomplete for all other receptors. As there are no domestic animals on FTSW, the pathway to MC in the source media through this exposure route is incomplete for all receptors. The pathway to MC in the source media through the game/fish/prey exposure route is potentially complete for biota and recreational users as fishing may occur since approximately 75% of the site is covered by Holbrook Pond. Hunting including shotgun, muzzle loader and archery are also permitted on Small Arms Range - 3.

Groundwater

Precipitation infiltration may provide for contaminant mobility into the shallow or surficial groundwater aquifer. However, based on a review of hydrogeological data (Section 5.1.2.5), it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW. Receptor contact with groundwater is possible if the soil is disturbed through excavation or construction activities, creating possible migration routes/mechanisms for MC in shallow groundwater. As such, authorized installation personnel, authorized contractors, and biota have potentially complete pathways to MC in subsurface soil and/or shallow groundwater through the (incidental) ingestion and dermal contact exposure routes. Given that it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW, the dermal and ingestion exposure routes are incomplete for all other receptors. Since the upper aquifer is not used as a potable water source and MC are typically not volatile, the inhalation (vapor) exposure route is incomplete for all receptors.

Surface Water/Sediment

The surface water/sediment exposure pathway is considered to be potentially complete for human and ecological receptors since a majority of the site is covered by Holbrook Pond.

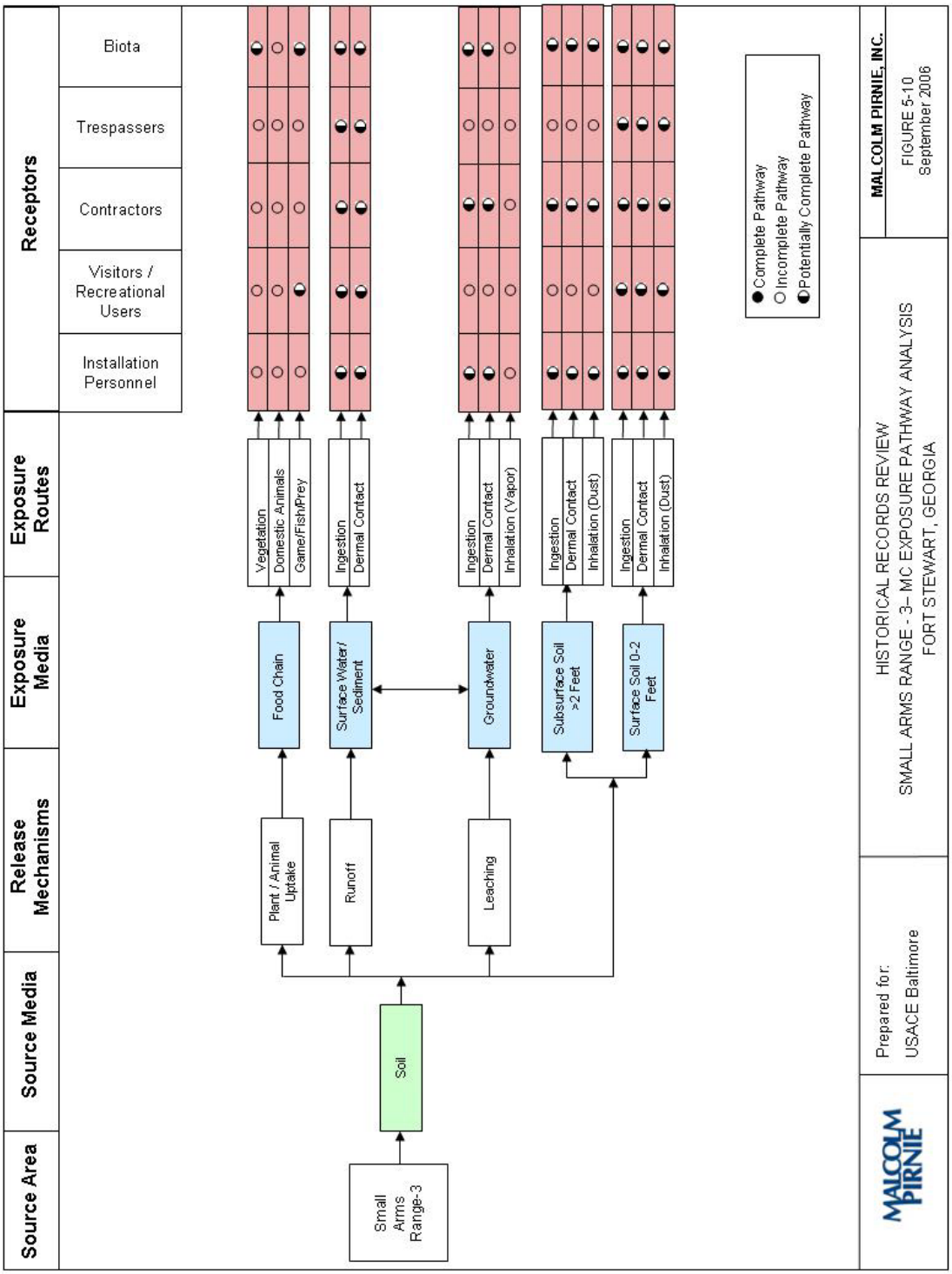
Subsurface Soil

Since the potential exists for MC in the subsurface soil in the Small Arms Range - 3 area, receptor contact with subsurface soil is possible if the soil is disturbed through excavation or construction activities, creating possible receptor pathways to MC in subsurface soils. As such, installation personnel, contractors, and biota have potentially complete pathways to MC in subsurface soil through the (incidental) ingestion, dermal contact, and inhalation (dust) exposure routes. Visitors and trespassers would not be expected to participate in any intrusive activities; therefore, these exposure routes are incomplete for these receptors.

Surface Soil

All human and ecological receptors within the Small Arms Range - 3 area may be exposed to surface soil during daily activities. Therefore, the pathways to MC in surface soil through the (incidental) ingestion, dermal contact, and inhalation of dust exposure routes are potentially complete for all receptors.

Figure 5-10: MC Pathway Analysis - Small Arms Range – 3



5.2.7 Hero Road Trench Area

5.2.7.1 MMRP Site Profile

The site-specific site profile is presented in Table 5-34:

Table 5-34: Site Profile - Hero Road Trench Area

Information Needs	CSM Findings
Area and Layout	Approximately 10 acres Located in center of cantonment area
Structures	None
Utilities	Yes, refer to Section 5.1.1.1.
Boundaries	N: Undeveloped property S: Undeveloped property E: Undeveloped property W: Undeveloped property
Security	Fences

5.2.7.2 Physical Profile

The site-specific physical profile is presented in Table 5-35:

Table 5-35: Physical Profile - Hero Road Trench Area

Information Needs	CSM Findings
Topography	Approximately 20 m amsl Flat, level terrain
Soil	Clay-sand/clay-silt
Hydrology	Wetland is located near site
Vegetation	Forest Grass

5.2.7.3 Land Use and Exposure Profile

The site-specific land use and exposure profile is presented in Table 5-36:

Table 5-36: Land Use and Exposure Profile - Hero Road Trench Area

Information Needs	CSM Findings
Current Land Use	Undeveloped
Current Human Receptors	Authorized Installation personnel Authorized Contractors Trespassers
Potential Future Use	Same as current use There is a potential to develop a childcare center near the site.
Potential Future Human Receptors	Same as current receptors
Beneficial Resources	Forest and grass habitat

5.2.7.4 Ecological Profile

The site-specific ecological profile is presented in Table 5-37:

Table 5-37: Ecological Profile - Hero Road Trench Area

Information Needs	CSM Findings
Habitat Type/System	Forest and grassy area
Degree of Disturbance	Low; forest and grass remains

5.2.7.5 Munitions/Release Profile

5.2.7.5.1 Munitions Types

Table 5-38 presents a summary of the types of munitions that could be expected to exist based on information collected during this HRR process. Also presented in this table are the mechanisms by which munitions would be expected to be released into the environment if present.

Table 5-38: Summary of Potential Munitions Types – Hero Road Trench Area

Potential Munitions	Primary Release Mechanism
Chemical Agent Identification Sets Kits (M1)	Intentionally or unintentionally disposed items

5.2.7.5.2 Maximum Probability Penetration Depth

There is no associated maximum probability penetration depth for the Hero Road Trench Area due to the fact that this site is a former trench and landfill area. The depth at which MEC could be located depends on the amount of fill placed on top of it and is not representative of the depths presented in Engineering Manual 1110-1-4009 *Ordnance and Explosives Response*. MEC could be encountered at any depth within the landfill.

5.2.7.5.3 MEC Density

The MEC density of the Hero Road Trench Area is considered to be low due to the fact that activities conducted at Hero Road Trench Area did not include the firing of explosives. However, M1 detonation kits may be buried at the Hero Road Trench Area and a small explosive charge is associated with M1 detonation. There have been no reported finds of MEC; however, the majority of the area is undeveloped.

5.2.7.5.4 Munitions Debris

A visual survey was not conducted as part of the HRR; however, based on the activities that occurred, there is the potential for munitions debris items. A geophysical survey was conducted in September 2003 on 4 acres off of Hero Road around the Family Housing Maintenance parking lot. Anomalies were recorded but it could not be determined if they were from burial items or interference. No MEC or munitions debris are known to have been reported; however, a significant portion of the area is undeveloped.

5.2.7.5.5 Associated Munitions Constituents

Potential MC associated with the Hero Road Trench Area include tear gas and smoke composition (see Ordnance Technical Data Sheets in Appendix D). Soil samples within the boundary of the Hero Road Trench Area have not been analyzed for constituents; therefore, it is not known whether constituents exist in concentrations that exceed regulatory levels.

5.2.7.5.6 Transport Mechanisms/Migration Routes

The primary transport mechanisms identified for the Hero Road Trench Area include:

Erosion: The Hero Road Trench Area is near a wetland; therefore, erosion is possible in this area and is a factor in transporting and migrating possible MC contaminated soil.

Soil Disturbance: The current degree of disturbance is relatively low, as most of the area has not been developed since the range was used. More development, especially in the forested area, could unveil potential MC that are in the surface or subsurface.

Infiltration: Based on the soil types associated with Hero Road Trench Area, the potential exists for MC to migrate from one environmental medium to another (surface to subsurface soil to groundwater) through filtration.

5.2.7.6 Pathway Analysis

5.2.7.6.1 MEC

Based on historical documents and information obtained during the data collection process, M1 detonation kits may be buried at the Hero Road Trench Area. A small explosive charge is associated with M1 detonation kits; therefore, the potential for MEC on the MRS exists. The MRS is currently fenced; therefore, access is controlled. Since the site is reportedly a burial site, no MEC are expected to be present on the surface. As illustrated in the Exposure Pathway Analysis for MEC (Figure 5-11), no complete or potentially complete pathways for human or ecological receptors for MEC on the surface

are expected to exist. Potentially complete pathways exist for authorized installation personnel, authorized contractors, and biota for MEC in the subsurface as these receptors have the potential to conduct intrusive activities. The pathway for MEC in the subsurface is incomplete for all other receptors.

5.2.7.6.2 MC

As illustrated in the MC Exposure Pathway Analysis (Figure 5-12), soil and groundwater with potential MC impacts represent the potential primary source media. Given the absence of analytical data, the presence or absence of MC across the site cannot be verified. Based on this, potentially complete exposure pathways may exist for surface and subsurface soil and groundwater through various exposure routes for human receptors and biota.

Food Chain

Potentially complete pathways to MC in the source media through uptake into vegetation exist for grazing/foraging biota. This exposure pathway is incomplete for all other receptors. As there are no domestic animals on FTSW, the pathway to MC in the source media through this exposure route is incomplete for all receptors. The pathway to MC in the source media through the game/fish/prey exposure route is potentially complete for biota. This exposure pathway is incomplete for all other receptors as their activities are not expected to include hunting.

Groundwater

Precipitation infiltration may provide for contaminant mobility into the shallow or surficial groundwater aquifer. However, based on a review of hydrogeological data (Section 5.1.2.5), it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW. Receptor contact with groundwater is possible if the soil is disturbed through excavation or construction activities, creating possible migration routes/mechanisms for MC in shallow groundwater. As such, authorized installation personnel, authorized contractors, and biota have potentially complete pathways to MC in subsurface soil and/or shallow

groundwater through the (incidental) ingestion and dermal contact exposure routes. Given that it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as a water supply for FTSW, the dermal and ingestion exposure routes are incomplete for all other receptors. Since the upper aquifer is not used as a potable water source and MC are typically not volatile, the inhalation (vapor) exposure route is incomplete for all receptors.

Subsurface Soil

Since the potential exists for MC in the subsurface soil in the Hero Road Trench Area, receptor contact with subsurface soil is possible if the soil is disturbed through excavation or construction activities, creating possible receptor pathways to MC in subsurface soils. As such, authorized installation personnel, authorized contractors, and biota have potentially complete pathways to MC in subsurface soil through the (incidental) ingestion, dermal contact, and inhalation (dust) exposure routes. Trespassers would not be expected to participate in any intrusive activities; therefore, these exposure routes are incomplete for these receptors.

Surface Soil

Since the Hero Road Trench Area is a suspected burial site, the presence of MC on the surface is not expected. Therefore, the pathways to MC in surface soil through the (incidental) ingestion, dermal contact, and inhalation of dust exposure routes are expected to be incomplete for all receptors.

Figure 5-11: MEC Pathway Analysis - Hero Road Trench Area

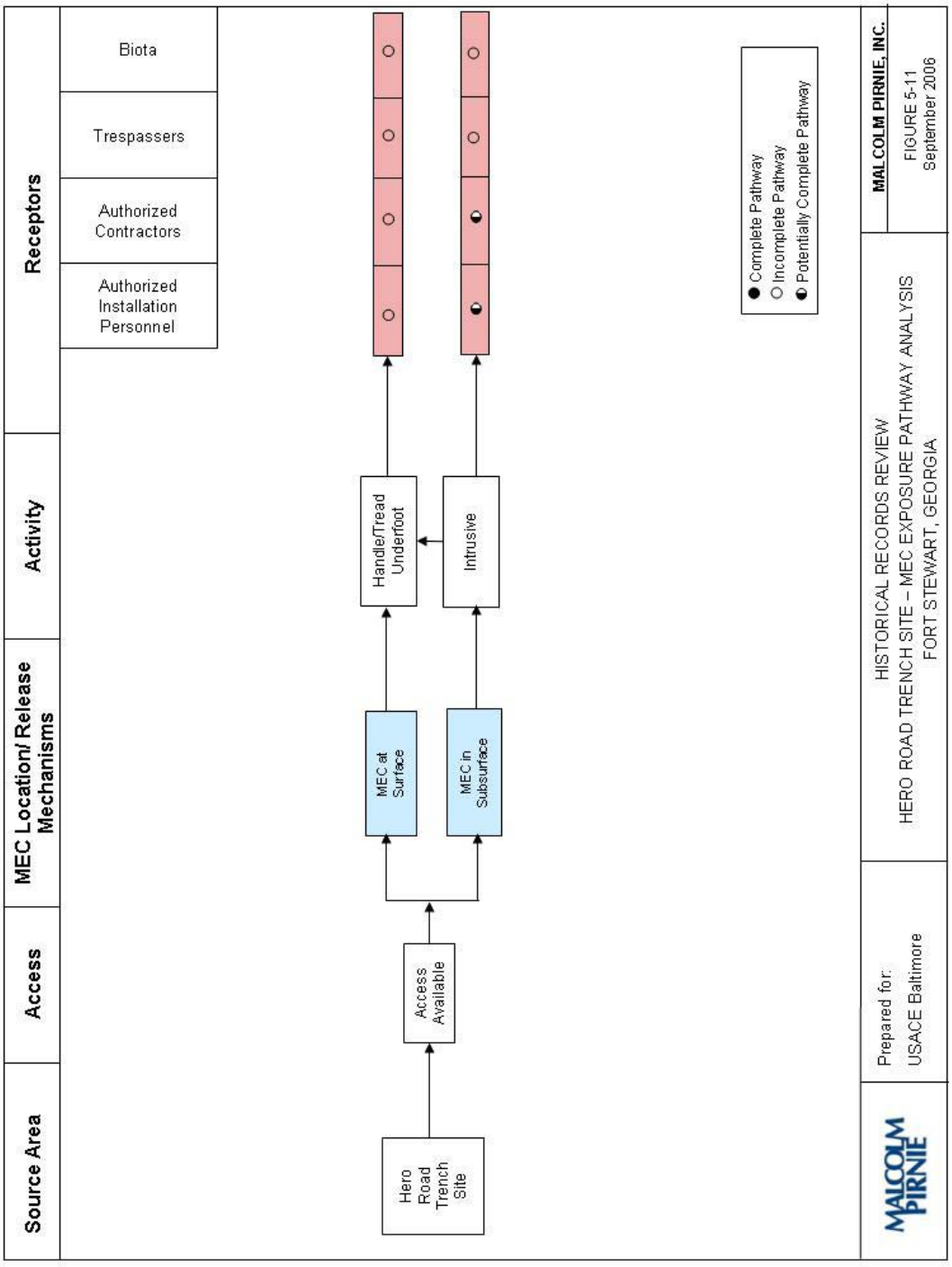


Figure 5-12: MC Pathway Analysis - Hero Road Trench Area