

**U.S. Army  
Environmental  
Center**

## **Final Quality Control Plan**

# **Feasibility Study (FS) and Remedial Investigation/Site Inspection (RI/SI) Addendum for Fort George G. Meade, Maryland**

**Submitted to**

**U.S. Army Environmental  
Center (USAEC)  
Aberdeen, Maryland**

**Revision 2  
November 19, 1993**

**Arthur D. Little, Inc.  
Acorn Park  
Cambridge, Massachusetts  
02140-2390**

**ADL Reference 67069-14**

**DAAA15-91-D-0016,  
Delivery Order 0009**

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**Arthur D Little**

**Feasibility Study (FS)  
and Remedial  
Investigation/Site  
Inspection (RI/SI)  
Addendum for Fort  
George G. Meade,  
Maryland**



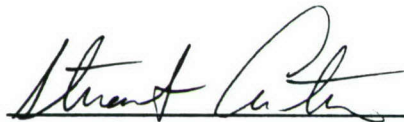
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Date

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## List of Acronyms and Abbreviations

|        |                                                                       |
|--------|-----------------------------------------------------------------------|
| AAS    | Atomic Absorption Spectroscopy                                        |
| ADL    | Arthur D. Little, Inc.                                                |
| AR     | Analytical Reagent                                                    |
| ASL    | Active Sanitary Landfill                                              |
| ASTM   | American Society for Testing and Materials                            |
| BRAC   | Base Realignment and Closure Act                                      |
| BFB    | Bromofluorobenzene                                                    |
| CAR    | Corrective Action Report                                              |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| CFD    | Clean Fill Dump                                                       |
| CFR    | Code of Federal Regulations                                           |
| CLP    | Contract Laboratory Program                                           |
| CNCL   | Cyanogen Chloride                                                     |
| COC    | Chain-of-Custody                                                      |
| COR    | Contracting Officer's Representative                                  |
| CRL    | Certified Reporting Limit                                             |
| CV     | Cold Vapor                                                            |
| DFTPP  | Decafluorotriphenylphosphine                                          |
| DL     | Detection Limit                                                       |
| DPDO   | Defense Property Disposal Office                                      |
| DSY    | DPDO Salvage Yard                                                     |
| ECD    | Electron Capture Detector                                             |
| EOD    | Explosive Ordnance Disposal                                           |
| EPA    | Environmental Protection Agency                                       |
| FGGM   | Fort George G. Meade                                                  |
| FID    | Flame Ionization Detector                                             |
| FS     | Feasibility Study                                                     |
| FSP    | Field Sampling Plan                                                   |
| FTA    | Fire Training Area                                                    |
| GC/FID | Gas Chromatography/Flame Ionization Detector                          |
| GC/MS  | Gas Chromatography/Mass Spectrometry                                  |
| GC     | Gas Chromatography                                                    |
| GC/ECD | Gas Chromatography/Electron Capture Detector                          |
| GFAA   | Graphite Furnace Atomic Absorption Spectrophotometer                  |
| HCL    | Hydrochloric Acid                                                     |
| HCN    | Hydrocyanic Acid                                                      |
| HHA    | Helicopter Hangar Area                                                |
| HNU    | HNU Inc., Manufacturer of Photoionization Detector                    |
| HPLC   | High Performance Liquid Chromatograph(y)                              |
| IC     | Ion Chromatograph(y)                                                  |
| ICAP   | Inductively Coupled Plasma-Emission Spectroscopy                      |
| ICP    | Inductively Coupled Argon Plasma emission spectroscopy                |
| IL2    | Inactive Landfill No. 2                                               |
| IR     | Installation Restoration                                              |

## List of Acronyms and Abbreviations

|           |                                                             |
|-----------|-------------------------------------------------------------|
| IRDMIS    | Installation Restoration Data Management Information System |
| IRP       | Installation Restoration Program                            |
| LCL       | Lower Control Limit                                         |
| LEL       | Lower Explosivity Limit                                     |
| LOF       | Lack of Fit                                                 |
| LWL       | Lower Warning Limit                                         |
| MS        | Mass Spectrometry                                           |
| MS/MSD    | Matrix Spike/Matrix Spike Duplicate                         |
| MTR       | Minimum Testing Range                                       |
| NA        | Not Analyzed                                                |
| NCP       | National Contingency Plan                                   |
| NCR       | Non Conformance Record                                      |
| ND        | Not Detected                                                |
| NIST      | National Institute of Standards and Technology              |
| No.       | Number                                                      |
| ODA       | Ordnance Demolition Area                                    |
| OSHA      | Occupational Safety and Health Administration               |
| OVA       | Organic Vapor Detector                                      |
| PA        | Preliminary Assessment                                      |
| PAT/GC/MS | Purge and Trap/Gas Chromatography/Mass Spectrometry         |
| PCB       | Polychlorinated Biphenyls                                   |
| PID       | Photoionization Detector                                    |
| PRI       | Potomac Research, Inc.                                      |
| PWRC      | Patuxent Wildlife Research Center                           |
| QA        | Quality Assurance                                           |
| QAC       | Quality Assurance Coordinator                               |
| QA/QC     | Quality Assurance/Quality Control                           |
| QAPjP     | Quality Assurance Project Plan                              |
| QC        | Quality Control                                             |
| QCP       | Quality Control Plan                                        |
| RCRA      | Resource Conservation and Recovery Act                      |
| RI        | Remedial Investigation                                      |
| RI/SI     | Remedial Investigation/Site Investigation                   |
| RIA       | Remedial Investigation Addendum                             |
| ROD       | Record of Decision                                          |
| RPD       | Relative Percent Difference                                 |
| SARM      | Standard Analytical Reference Material                      |
| SI        | Site Investigation                                          |
| SIA       | Site Investigation Addendum                                 |
| SLI       | Site Location Identity                                      |
| SOP       | Standard Operating Procedure                                |
| SOW       | Statement of Work                                           |
| SRM       | Standard Reference Material                                 |
| SW        | Solid Waste                                                 |
| TAL       | Target Analyte List                                         |

## List of Acronyms and Abbreviations

|          |                                                         |
|----------|---------------------------------------------------------|
| TCL      | Target Compound List                                    |
| TCLP     | Toxicity Characteristic Leaching Procedure              |
| TDS      | Total Dissolved Solids                                  |
| TEPS     | Total Environmental Program Support                     |
| TIC      | Tentatively Identified Compounds                        |
| TPHC     | Total Petroleum Hydrocarbons                            |
| TQM      | Total Quality Management                                |
| UCL      | Upper Control Limit                                     |
| USAEC    | United States Army Environmental Center                 |
| USATHAMA | United States Army Toxic and Hazardous Materials Agency |
| USEPA    | United States Environmental Protection Agency           |
| USC      | Unique Sample Code                                      |
| UV       | Ultraviolet                                             |
| UWL      | Upper Warning Limit                                     |
| UXO      | Unexploded Ordnance                                     |
| VOA      | Volatile Organic Analysis                               |
| VOC      | Volatile Organic Compound                               |
| ZI       | Zero Intercept                                          |



## 1.0 Project Description

### 1.1 Introduction

This Quality Control Plan has been prepared to address the Feasibility Study (FS) and Remedial Investigation/Site Inspection (RI/SI) Addendum activities and a Change Order to Delivery Order No. 0009 dated July 15, 1993, at the Fort George G. Meade (FGGM). It has been prepared for the U.S. Army Environmental Center (USAEC), formerly the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA), to fulfill the requirement of deliverable ELIN A004 under Task Order 0009 of TEPS contract DAAA15-91-D-0016. It has been developed in accordance with the USATHAMA Quality Assurance Program, USATHAMA *Geotechnical Requirements*, and *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA*.

Arthur D. Little's Corporate Policy includes a commitment to a high standard of quality in the work it performs for and delivers to its clients. Our commitment to quality is reflected in our general policies and procedures (hiring practices, performance evaluations, project management and control tools, and technical review procedures) and also in specific, written Quality Assurance Program and Project Plans that we develop and implement for major new assignments that we undertake. We expect similar commitment to quality from our subcontractors.

The objective of the USAEC Quality Assurance Program is to establish a QA system and proper QC procedures associated with the Quality Control Plan for specific projects, in this case, Fort George G. Meade, Delivery Order 0009. USAEC defines QA as "the system whereby an organization provides assurance that monitoring of quality related activities has occurred"; QC as "specific actions taken to ensure that system performance is consistent with established limits. It is these actions which ensure accuracy, precision, and comparability of results." A project specific QC plan is developed to address QA/QC activities. These activities ensure that the results of the field investigation program are properly documented and of adequate quality to support decisions about the necessity for and nature of further investigations and remedial actions.

This QC Plan for the FS and RIA/SIA for FGGM has been developed to comply with the requirements of the USAEC Quality Assurance Program, PAM 11-41, Revision No. 0, January 1990. We will be using a subcontracted USAEC-performance demonstrated laboratory, DataChem Laboratories, of Salt Lake City, Utah for chemical analyses of samples collected at FGGM. Therefore, we have attached the Quality Assurance Program Plan from DataChem to this QC Plan. The DataChem plan describes specific laboratory QA/QC activities, while our plan describes Arthur D. Little QA/QC activities, including sufficient details to assure, through reviews, that laboratory results meet USAEC requirements.

## **1.0 Project Description**

### **1.2 Site Background**

The Work Plan, provided by Arthur D. Little as a separate document for this project, provides details about the FGGM site and history of previous investigations. The information relevant to the QCP is summarized briefly in the following sections.

#### **1.2.1 Site Description**

FGGM is located in the northwest corner of Anne Arundel County, Maryland, approximately halfway between Baltimore and Washington, D.C (Figure 1-1). The installation contains administrative, recreational, and housing facilities, as well as limited training areas and firing/combat ranges. The FGGM community consists of a residential population and daytime work force of approximately 20,000.

#### **1.2.2 Site History**

In 1988, the U.S. Army Base Realignment and Closure Act (BRAC) recommended that 9,000 acres of the 13,000-acre facility be closed or excessed. The 9,000-acre area encompassed the southernmost two-thirds of the installation (Figure 1-2). On October 1, 1991, the U.S. Army transferred 7,600 of the 9,000 acres to the Department of the Interior, specifically the Patuxent Wildlife Research Center (PWRC). An additional 500 acres are also proposed for transfer to the Department of Interior, PWRC, however this transfer has not yet been completed. No determination has been made about the transfer of the Tipton Army Airfield and additional acreage (Hill, 1993). The remaining acres includes some areas to be retained by FGGM and additional areas to be excessed. These 1,400 acres consist of approximately 1,000 acres of woodlands and wetlands and approximately 400 acres associated with the Tipton Army Airfield.

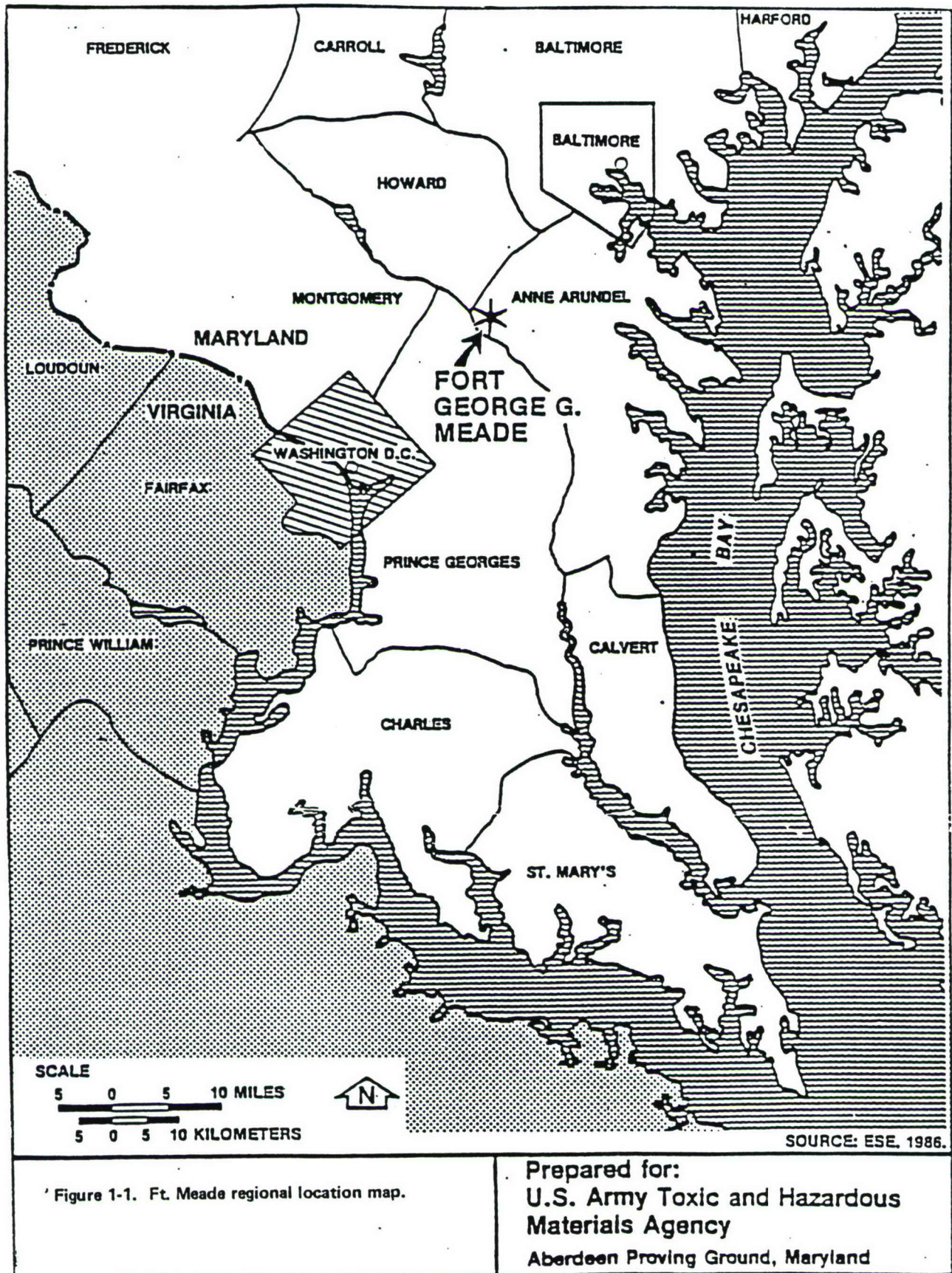
The areas to be studied during this task are briefly described below, along with an indication of what investigation and/or study will be undertaken:

- The Inactive Landfill No. 2 (IL2) is located adjacent to and south of the Tipton Army Airfield and is approximately 450 feet north of the Little Patuxent River. (SIA)
- The DPDO Salvage Yard (DSY) is located north of Route 32, northeast of the Tipton Airfield. (SIA)
- The Helicopter Hangar Area (HHA) is located west of Tipton Airfield and approximately 800 feet west of the Fire Training Area. (SIA)
- The Fire Training Area (FTA) is located immediately north of the Tipton Airfield (SIA).
- The Ordnance Demolition Area (ODA) is located at Training Range 16, which is in the southwestern area of the Base Closure parcel. (SIA)

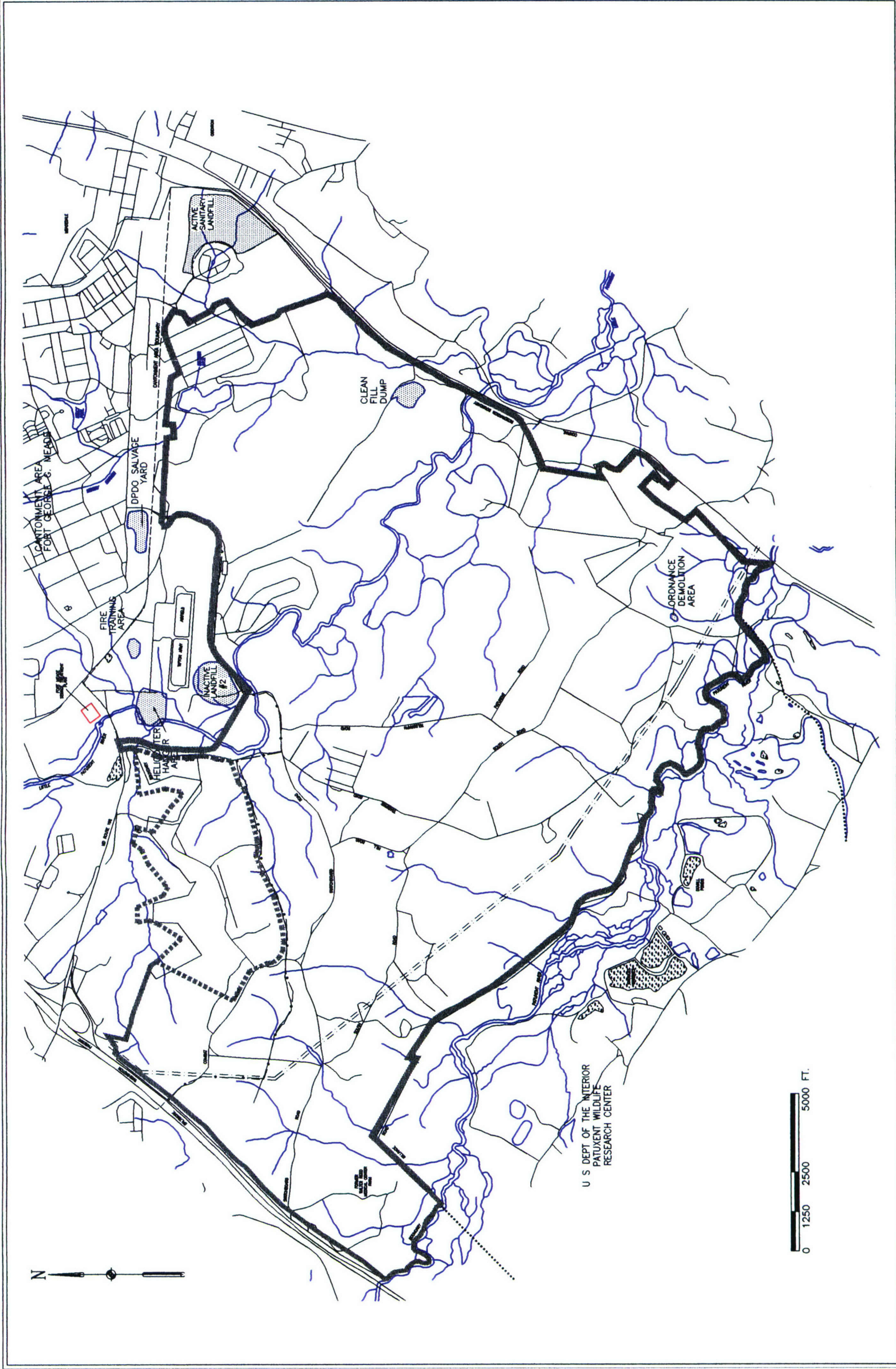


## 1.0 Project Description

Figure 1-1: Site Location Map







|                  |  |                      |  |                 |  |                |  |                                               |  |                        |  |                                                             |  |
|------------------|--|----------------------|--|-----------------|--|----------------|--|-----------------------------------------------|--|------------------------|--|-------------------------------------------------------------|--|
| PREPARED FOR:    |  | USAEC                |  | SOURCE:         |  | USATHAMA, 1992 |  | LEGEND:                                       |  | <b>Arthur D Little</b> |  | TITLE: FIGURE 1-2: GENERAL SITE MAP<br>FORT GEORGE G. MEADE |  |
| DATE: APRIL 1993 |  | SCALE: 1" = 3000 FT. |  | DWG. NO. FMBM-E |  |                |  | 500+/- Acre Boundary<br>7600+/- Acre Boundary |  |                        |  |                                                             |  |



## 1.0 Project Description

- The Active Sanitary Landfill (ASL) is located on 130 acres of land and is situated along the FGGM eastern boundary, south of Route 32. The surrounding land to the east and southeast of the ASL is privately owned. The ASL and adjacent property to the north, west, and south of the site is to be retained by FGGM and is not included in the 7,600-acre transfer to the PWRC. (RIA, FS)
- The Clean Fill Dump (CFD) is located within the 7,600 acres that were transferred to PWRC along the eastern border of the Patuxent Wildlife Refuge. (RIA)

Figure 1-2 shows the location of these sites.

## 1.3 Task Objectives and Scope of Work

The purpose of this task order is to conduct a Feasibility Study (FS) of the active sanitary landfill (ASL) and to address data gaps remaining from the previous Remedial Investigation (RI) and Site Inspection (SI). The purpose of this document is to ensure that data collected during this task are of sufficient quality to complete the task objectives. Detailed objectives for each deliverable and activity in the task are included in the Arthur D. Little FGGM Work Plan, which has been provided as a separate document.

The overall purpose of an SI is to evaluate if releases or potential contamination has occurred at suspected sites and to determine if further investigation is warranted. Arthur D. Little's study is an addendum to a previous SI conducted by EA Engineering, Science and Technology, Inc. (EA) and addresses remaining data gaps identified in the previous SI. The sites and tasks included in the SI addendum (SIA) are:

- Inactive Landfill No. 2 (IL2)
  - Collect and analyze ground water samples from six existing monitoring wells to evaluate the continued presence of elevated metals in this area.
  - Collect and analyze one seep sample to evaluate source water chemistry.
- DPDO Salvage Yard and Transformer Storage Area (DSY)
  - Install two additional monitoring wells. Collect and analyze seven ground water samples. These data will be used to address the continued presence and extent of contamination by volatile organic compounds.
- Helicopter Hangar Area (HHA)
  - Collect and analyze a total of five surface water and five sediment samples from outfalls and the Little Patuxent River to determine if site conditions and/or discharges are affecting the river chemistry.



## 1.0 Project Description

- Install one ground water monitoring well and collect samples from the new and existing wells to evaluate ground water contamination.
  - Collect eight soil samples from four locations (two depths per location) to evaluate the oil-water separator. Headspace screening will be conducted for all samples and the highest concentration samples sent for analysis.
  - Collect one subsurface soil sample during advancement of a soil boring to determine soil quality.
- Fire Training Area (FTA)
  - Install three monitoring wells and analyze ground water samples from the three new wells to determine if site activities have resulted in ground water contamination.
  - Collect and analyze one sludge sample from the oil-water separator to evaluate source chemistry.
- Ordnance Demolition Area (ODA)
  - Drill four soil borings and collect three soil samples from each for laboratory analysis. Complete three of the borings as monitoring wells and sample the ground water from each. These data will be used to determine if ordnance demolition has impacted soil or ground water quality.
  - Collect and analyze one surface water and one sediment sample from the seep to evaluate ground water discharge to the ground surface.
- Background Soil Samples
  - Collect 30 surficial soil samples to provide baseline background soil quality data.
- Soldiers Lake
  - Collect two surface water samples to evaluate background water chemistry.

The overall purpose of an RI is to evaluate the extent and rate of migration of contamination at sites which, according to historical and site data, may present adverse effects on the environment. This study is an addendum to a previous RI conducted by EA and addresses data gaps remaining from or identified in that document. The two sites, and their associated tasks, included in the RI addendum (RIA) are:

- Active Sanitary Landfill (ASL)
  - Install eight additional monitoring wells to help delineate the ground water plume.
  - Collect ground water, surface water, sediment, and leachate samples to evaluate changes in environmental quality.
  - Conduct a human health risk assessment and feasibility study.

## 1.0 Project Description

- Clean Fill Dump (CFD)
  - Collect ground water samples from the existing wells to evaluate the continued presence of contaminants.
  - Collect three surface water samples to further evaluate if ground water is impacting the surface water.
  - Collect one seep sample to evaluate source chemistry.
  - Conduct a human health risk assessment.

The purpose of an FS is to review the applicability of various remedial technologies to determine whether they are appropriate remedies for the site and to evaluate each remedy with regards to effectiveness, implementability, and costs. The FS is only being conducted for the ASL. It includes a Proposed Plan, a Responsiveness Summary, and a Record of Decision (ROD).

A summary of activities at each of the sites is provided in Figure 1-3, and a summary of analytical samples is provided in Table 4-1.

### 1.4 Application of Project QC Plan

This Project QC Plan is applicable to both the analytical and the field investigation component of the task order.

QA refers to the system whereby an organization provides assurance that monitoring of quality-related activities has occurred; QA is generally interpreted as a recordkeeping system for documentation of activities including traceability, completeness, and security of documents. Through implementation of this QA program in the field, in the office, and at the laboratory, the validity and reliability of site data and other documents will be monitored such that decisions based on the data or documents can be substantiated. QC refers to specific actions taken to verify that activities performed are consistent with established limits of acceptable quality. It is through these actions that accuracy, precision, and comparability of results are verified. QC activities must be conducted within a QA program to document that QC exists.

This Project QC Plan establishes a QA system and appropriate QC procedures for use by Arthur D. Little and its subcontractors. The emphasis of this plan is on activities that generate field and analytical data; the plan also addresses field activities that may affect the integrity of these data. This plan documents specific instructions for environmental sampling and chemical analyses; requirements for all chain-of-custody procedures, and field activities; QC of computer and document-related activities; and QC of final calculations. Arthur D. Little and its subcontracted analytical laboratory will adhere to the procedures stated in this Project QC Plan.



Figure 1-3: Summary of Activities

| Site Name                | Investigative Activities |                        |                |            |                    |                     |                                    |                      |              |                  |                      |                  |                |              |
|--------------------------|--------------------------|------------------------|----------------|------------|--------------------|---------------------|------------------------------------|----------------------|--------------|------------------|----------------------|------------------|----------------|--------------|
|                          | Site Inspection          | Remedial Investigation | Records Search | UXO Survey | Supply Well Survey | Soil Boring Samples | Surface Water and Sediment Samples | Surface soil Samples | Soil Borings | Monitoring Wells | Ground Water Samples | Leachate Samples | Sludge Samples | Seep Samples |
| SI Addendum              |                          |                        |                |            |                    |                     |                                    |                      |              |                  |                      |                  |                |              |
| Inactive Landfill No. 2  | ●                        |                        |                |            |                    |                     |                                    |                      |              |                  | 6                    |                  |                | 1            |
| DPDO Salvage Yard        | ●                        |                        |                | ●          |                    |                     |                                    |                      |              | 2                | 8                    |                  |                |              |
| Transformer Storage Area | ●                        |                        |                | ●          |                    |                     |                                    | 6                    |              |                  |                      |                  |                |              |
| Helicopter Hangar Area   | ●                        |                        |                |            |                    |                     | 5/5                                | 8                    |              |                  | 6                    |                  |                |              |
| Fire Training Area       | ●                        |                        |                | ●          |                    |                     |                                    |                      |              | 3                | 3                    |                  | 1              |              |
| Ordnance Demolition Area | ●                        |                        |                | ●          |                    | 12                  | 1/1                                |                      | 4            | 3                | 3                    |                  |                |              |
| Background               | ●                        |                        |                |            |                    |                     |                                    | 30                   |              |                  |                      |                  |                |              |
| Soldiers Lake            | ●                        |                        |                |            |                    |                     | 2/0                                |                      |              |                  |                      |                  |                |              |
| Drilling Water           |                          |                        |                |            |                    |                     |                                    |                      |              |                  | 2                    |                  |                |              |
|                          |                          |                        |                |            |                    |                     |                                    |                      |              |                  |                      |                  |                |              |
| RI Addendum              |                          |                        |                |            |                    |                     |                                    |                      |              |                  |                      |                  |                |              |
| Active Sanitary Landfill |                          | ●                      | ●              | ●          | ●                  |                     | 6/6                                |                      |              | 8                | 34                   | 3                |                |              |
| Clean Fill Dump          |                          | ●                      |                |            |                    |                     | 3/0                                |                      |              |                  | 6                    |                  |                | 1            |
| Totals                   |                          |                        |                |            |                    | 12                  | 17/12                              | 44                   | 4            | 16               | 67                   | 3                | 1              | 2            |

## 1.0 Project Description

### 1.5 Organization of Document

This Project QC Plan has been prepared using the guidance provided in the USAEC QA Program Manual (January 1990); the Plan has been organized into the sections indicated in the guidance document. Sections 1.0 through 3.0 of this plan provide an overview of the project scope, organization, and objectives. Section 1.0 describes the project, project objectives, summary of relevant past investigations, and scope of the current investigation. Section 2.0 presents the organization of the project team and identification of specific QA responsibilities. The QA objectives for the data collected during this investigation are provided in Section 3.0.

Sections 4.0 through 9.0 provide details of the procedures for sample collection and analysis and data reporting. The specific sampling procedures to be used in the collection of field samples for this FGGM project are provided in Section 4.0. The sample custody procedures, for both field and laboratory activities, are summarized in Section 5.0. Section 6.0 provides the required calibration procedures for the field and laboratory instruments to be used. Section 7.0 specifies the procedures for field and laboratory data collection; most of the analytical procedures to be used for the FGGM project are USAEC-performance demonstrated methods. The procedures to be followed for data reduction, validation, and reporting are provided in Section 8.0; these procedures conform with the USAEC IRDMS requirements. Section 9.0 identifies the QA procedures internal to the sample collection and analysis activities and specifies the frequency for each of these checks.

Section 10.0 summarizes the performance and system audits to be conducted within this investigation. Section 11.0 addresses the procedures and schedule for preventive maintenance of field and laboratory instrumentation. The specific procedures routinely used to assess data quality (precision, accuracy and completeness) are provided in Section 12.0; for the USAEC-performance demonstrated methods, these procedures are specified within the method and the calculations are performed using the USAEC software. The recommended corrective actions and QA reports to management are addressed in Sections 13.0 and 14.0, respectively.

A list of acronyms and abbreviations is provided at the beginning of this plan. In addition, three Appendices are included. Appendix A provides QA Program Plan for USAEC, prepared by DataChem Laboratories, Inc.; Appendix B includes the available SOPs for field and laboratory activities; and Appendix C provides a checklist to be used during field and laboratory activities to assure compliance with this QC Plan.



## **2.0 Project and QA/QC Organization and Responsibilities**

This section describes the organizational structure for the FGGM investigations being conducted by Arthur D. Little. This structure indicates the overall assignment of responsibility for all aspects of the project and the functional and communication relationships among the organizational elements participating in this project. The organizational structure for the FGGM RIA, SIA, and FS is presented in Figure 2-1. The roles and responsibilities of key project team personnel are described below.

### **2.1 Project Organization**

#### **2.1.1 Program Manager**

Dr. Robert N. Lambe is the Arthur D. Little Program Manager for the USAEC Total Environmental Program Support (TEPS) contract. He will be responsible for: monitoring technical progress; reviewing and approving all work products; reviewing and approving all deliverables before submission to USAEC; monitoring financial and schedule control; and instituting corrective action, if necessary.

#### **2.1.2 Task Manager**

Ms. Kathleen Thrun is the Arthur D. Little Task Manager for Delivery Order 0009 and will work directly with Dr. Lambe. As Task Manager, her responsibilities include: project staffing and direct management of all staff assigned to Delivery Order 0009; direct financial and schedule control; review and approval of all deliverables; recommending corrective actions, if necessary, to the Program Manager; and maintaining a liaison with the USAEC Project Officer, and FGGM Environmental Office Manager. In this role, the Task Manager will be responsible for ensuring that the USAEC Project Officer and FGGM Environmental Office Manager are kept informed of all technical progress as necessary.

#### **2.1.3 Task Staff**

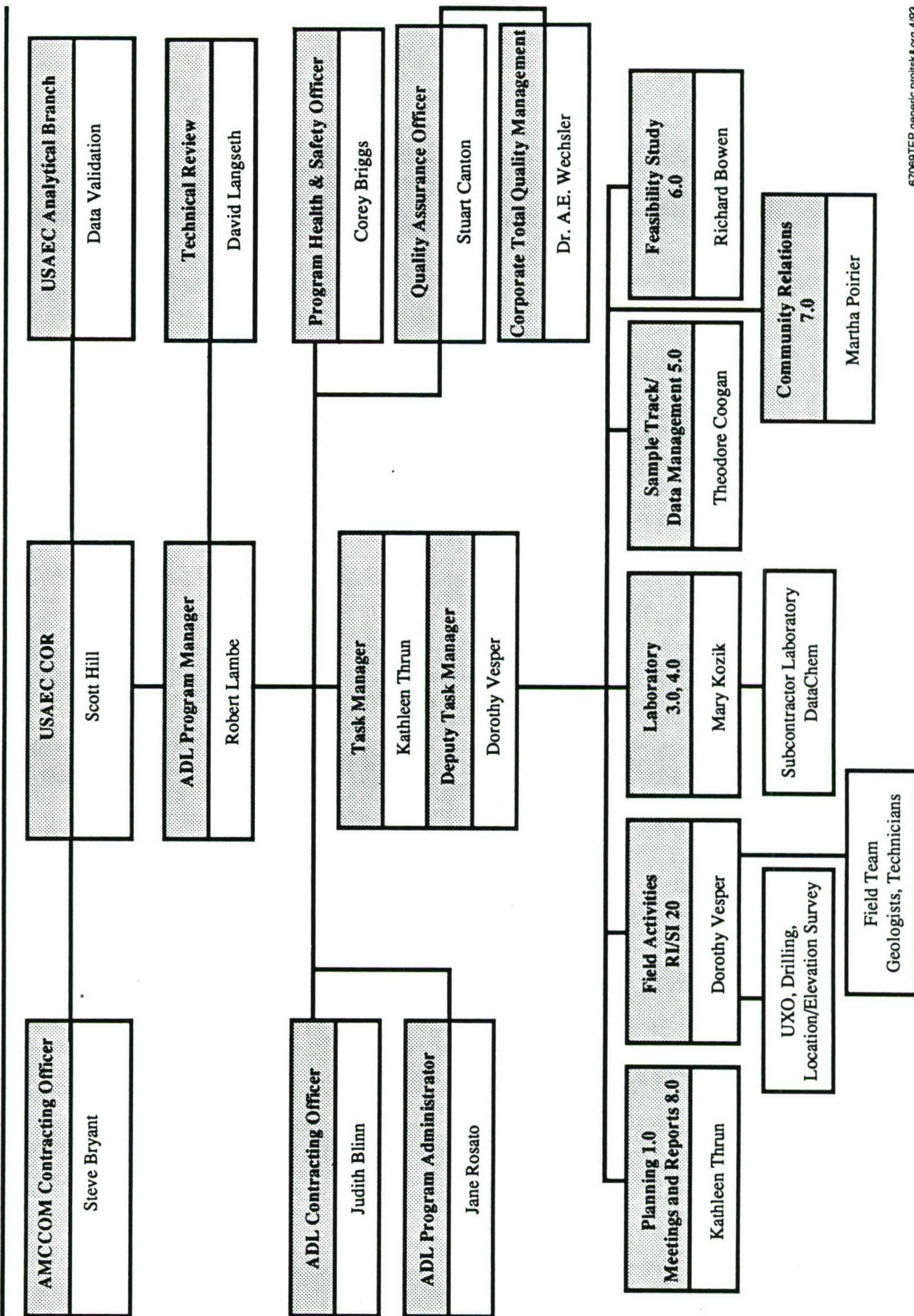
To assist Ms. Thrun in the performance of duties as Task Manager, the following Subtask Managers have been assigned to this project:

- Field Activities and Deputy Task Manager - Dorothy Vesper
- Laboratory Analysis and QA/Data Review - Mary Kozik
- Sample Tracking Database Management - Ted Coogan
- Risk Assessment - Scot Gnewuch
- Feasibility Study - Richard Bowen

The Subtask Managers are responsible for coordinating all phases of activities required to complete the stated goals of their subtask assignment, including tracking and reporting on technical quality, schedule, budget, deliverables, problems and corrective actions. Subtask Managers are responsible for ensuring that the Task Manager is kept informed of all technical progress and potential problem areas. Consistency in approach for each subtask will be assured through management by the



**Figure 2-1  
Fort Meade Project Tasks and Organization**





## **2.0 Project and QA/QC Organization and Responsibilities**

Task and Subtask Managers, brief weekly meetings, and use of a common resource base will be used to perform the specific work assignments. Technical staff members will take direction from the Subtask Managers.

Field activities will be managed by Ms. Dorothy Vesper. During the on-site field investigation at FGGM, the field team will include a site coordinator who will be Ms. Vesper or her designee and a designated on-site Health and Safety supervisor. In addition to field geologists and technicians, the subcontractors selected for the UXO survey, drilling, and the elevation/location survey will also report to the site coordinator.

Laboratory activities will be overseen by the Lead Chemist, Ms. Mary Kozik. She or her designee will be responsible for coordinating field and laboratory activities, and reviewing the operations and data files/packages of our subcontracted laboratory, DataChem.

Dr. David E. Langseth, Vice President in charge of Earth Sciences and Engineering, will serve as Technical Reviewer, serving USAEC in two ways. First, he will provide a high level of corporate attention to the task to ensure that the staffing that is needed to complete the Delivery Order within the proposed schedule is available. Second, because Dr. Langseth is an engineer who has spent considerable time evaluating and selecting technologies for site remediation and hazardous waste treatment, he will provide the Army with both a technical review, as well as a managerial review.

### **2.2 Arthur D. Little QA/QC Organization**

In order to ensure that all aspects of QA/QC are followed according to the USAEC Quality Assurance Program and this Quality Control Plan, the responsibilities to oversee this project have been assigned to the Project QA Officer and the Project Lead Chemist, in addition to specific responsibilities for QA in our subcontracted laboratory.

#### **2.2.1 Program QA Officer**

Arthur D. Little's Total Quality Management (TQM) Program is under the direction of Dr. Alfred E. Wechsler, Senior Vice President and Chief Professional Officer. Dr. Wechsler has selected Mr. Stuart Canton as the Project Quality Assurance Officer for the USAEC TEPS Contract. In his role as an independent evaluator of Arthur D. Little's performance during this Delivery Order, Mr. Canton will discuss our performance, as necessary, with officials at USAEC and other U.S. Army officials in the chain of command. Mr. Canton's findings and recommendations will be communicated directly to Dr. Lambe, Program Manager, Ms. Thrun, Task Manager, and Dr. Wechsler, Chief Professional Officer during the course of the FGGM project.

## **2.0 Project and QA/QC Organization and Responsibilities**

The primary focus of the Project Quality Assurance Officer will be to ensure that systems are in place and adequate to maintain the maximum level of quality throughout all aspects of the project.

Specific functions and duties of the Project Quality Assurance Officer include:

- Reviewing and approving of QA policies and procedures
- Reporting the adequacy, status, and effectiveness of the QA program on a regular basis to the project management
- Maintaining responsibility for documentation of corporate QA records, documents, and communications
- Conducting field audits
- Coordinating with the Lead Chemist, as needed, to ensure QC procedures specific to the laboratory and data management are followed and documented

The purpose of the field audits is to ensure that sampling is conducted in a manner consistent with the QA Program and other USAEC guidelines. This responsibility includes making trips to the site to inspect sampling where applicable. A minimum of coordination with the Arthur D. Little Project Manager prior to the inspection is acceptable. Each major type of sampling (e.g., ground water, surface water, soil, sediment) will be inspected at least once per installation investigation. The visit(s) will occur approximately during the first sampling effort for each matrix. Additional inspections may occur at the discretion of the Project QA Officer, with approval of the USAEC Project Officer and Arthur D. Little Task Manager. The Project QA Officer will document (Appendix U of the USATHAMA QA Program, January 1990) each inspection and ensure that procedures described in the Scope of Work, Project Work Plan, and Project QC Plan are followed. The Program QA Officer has the authority to require resampling of any site whose sampling integrity was determined to have been affected by faulty sampling procedures, after obtaining approval from the USAEC Project Officer or the Contracting Officer's Representative.

### **2.2.2 Lead Chemist**

Arthur D. Little's Lead Chemist is Ms. Mary Kozik. She will assist with oversight of the laboratory activities for this project. Specific functions and duties include:

- Maintaining copies of our subcontracted laboratory documentation, including USAEC-performance demonstrated methods and Quality Assurance Plans
- Providing an external and, thereby, independent QA review of our subcontracted laboratory activities and documentation (including all control charts and a 10 percent review of data packages and IRDMIS data files)



## 2.0 Project and QA/QC Organization and Responsibilities

- Coordinating with USAEC, Arthur D. Little, and DataChem to ensure that QA objectives appropriate to the project are established and that DataChem personnel are aware of these objectives
- Coordinating with DataChem management and personnel to ensure that QC procedures, appropriate to demonstrating data validity and sufficient to meet QA objectives, are developed and in place
- Ensuring data are properly reviewed by an Arthur D. Little QA chemist, including resolving any discrepancies between DataChem and the validator
- Requiring and/or reviewing corrective actions taken in the event of QC failures
- Reporting non-conformance with QC criteria or QA objectives, including an assessment of the impact of the data quality or project objectives, to the Program QA Officer and Task Manager

## 2.3 DataChem Project QA/QC Organization

The DataChem laboratory organization is described in the DataChem QA Program Plan, Section 3, Organization and Responsibilities, provided in Appendix A.

Responsibilities of the DataChem Analytical Task Manager (James H. Nelson, Ph.D.) include but are not limited to:

- Through the Arthur D. Little Task Manager, submit to Arthur D. Little for approval a detailed Project QC Plan specific to the USAEC project being supported
- Support a Quality Assurance Coordinator (QAC) who will not be subordinate to or be in charge of any person having direct responsibility for sampling or analyses
- Provide sufficient equipment, space, resources, and personnel to conduct analyses and implement the USAEC project and QA Program
- Submit the required documentation and laboratory performance demonstration data to Arthur D. Little prior to analyzing field samples
- Ensure that subsampling and other handling procedures in the laboratory are adequate for the sample types received
- Oversee the quality of purchased laboratory materials, reagents, and chemicals to ensure that these supplies do not jeopardize the quality of analytical results

## 2.0 Project and QA/QC Organization and Responsibilities

- Ensure implementation of corrective action for any QA/QC deficiencies

The DataChem Quality Assurance Coordinator (Lance M. Eggenberger, M.S.) will:

- Monitor the QA and QC activities of the laboratory to ensure conformance with authorized policies, procedures, and sound practices, and recommend improvements as necessary
- Inform the Arthur D. Little Task Manager, Arthur D. Little Lead Chemist, and laboratory management of nonconformance to the QA Program
- Request analytical reference materials from USAEC through the USAEC Chemistry Branch
- Ensure that all records, logs, standard procedures, project plans, and standing operating procedures are distributed to all laboratory personnel involved in the project
- Establish, with the analysts and the Arthur D. Little Lead Chemist, the correct analytical lot size, the correct QC samples to be included in each lot, and the correct procedures for evaluating acceptable, in-control analytical performance
- Ensure that logging of received samples includes establishing appropriate lot size for each analysis and allocating sample numbers for the correct control samples in each lot and that checklist is filled out and maintained
- Review all laboratory data before those data are transmitted to permanent storage, reported to other project participants, or submitted via the USAEC Installation Restoration Data Management Information System (IRDMIS). Before data are released, the QAC must have completed the Contractor QAC Checklist (Appendix P) and inspected calibration data, control charts, and other performance indicators to verify that the data were collected under conditions consistent with laboratory performance demonstration and that the analytical systems were in control
- Ensure that a signed Data Package Checklist is included in each completed data package
- Ensure that analysts are preparing QC samples, maintaining control charts, and implementing and documenting corrective action when necessary
- Ensure that all sampling logs, instrument logs, and QC documents are maintained and are completed with the required information



## 2.0 Project and QA/QC Organization and Responsibilities

- Collect control charts from analysts, discuss control chart results with the Analytical Task Manager, and submit the charts to Arthur D. Little and the USAEC Chemistry Branch on a weekly basis
- Maintain an awareness of the entire laboratory operation to detect conditions that might directly or indirectly jeopardize controls of the various analytical systems (e.g., improper calibration of equipment, cross contamination through improper storage of samples)
- Audit sampling documentation and procedures to ensure that samples are labeled, preserved, stored, and transported according to prescribed methods following approved chain-of-custody procedures

### **3.0 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability**

#### **3.1 Introduction**

QA objectives are qualitative and quantitative statements that specify the quality of data necessary for regulatory and/or project specific decisions. The process of developing QA objectives for a given study helps to ensure that generated data are of adequate quality for the intended use. QA objectives are expressed in terms of precision, accuracy, representativeness, completeness, and comparability.

#### **3.2 QA Objectives for FGGM Data**

QA objectives for the data collected under the FGGM investigations covered by this Project QC Plan have been defined to ensure that the collected data will be of sufficient quality to support the RI/FS/SI decision-making needs of the USAEC program. In order to provide a common point of reference for all projects and ensure comparability of the data generated within the USAEC program, USAEC prescribes the use of standardized analytical methods that provide sufficient information to evaluate data quality. For specific methods, the USAEC QA program defines QA objectives through a process of method performance demonstration, including pre-performance demonstration calibration and performance demonstration analyses: the USAEC Chemistry Branch determines whether the results of these analyses demonstrate proficiency of the laboratory and, if proficiency is demonstrated, assigns method numbers to be used when reporting data. This effort also provides the baseline for establishing control limits for daily analyses. Where possible, USAEC-performance demonstrated analytical methods will be used for the analysis of FGGM samples; for non-performance demonstrated methods, analyses will be performed based on standard EPA methods. A USAEC-performance demonstrated laboratory, DataChem Laboratories, will be used to perform all analyses on the field samples collected for this project; DataChem's QA Program Plan for USAEC Laboratory Analyses is attached as Appendix A to this Project QC Plan. All analytical methods used for the FGGM project will generate appropriate QC data to enable data quality to be assessed with respect to the QA objectives of the project.

USATHAMA analytical methods are characterized by rigorous QA/QC protocols and documentation requirements. The USAEC data is of high quality, comparable to EPA Level IV data quality (Data Quality Objectives, 1987). USAEC-performance demonstrated methods will be used for the following analyses: Target Analyte List (TAL) metals (analyzed by inductively coupled argon plasma emission spectroscopy, ICP), Target Compound List (TCL) volatiles (analyzed by purge and trap/gas chromatography/mass spectrometry, PAT/GC/MS), TCL semivolatiles (analyzed by extraction followed by gas chromatography with mass spectrometry, GC/MS), PCBs (analyzed by extraction followed by gas chromatography with electron capture detection, GC/ECD), explosives (analyzed by high performance liquid chromatography with ultraviolet detection, HPLC), and chloride, sulfate and nitrate/nitrite (analyzed by ion chromatography, IC).



### **3.0 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability**

The Target Analyte List (TAL) of metals and Target Compound List (TCL) of volatile and semivolatile organics are defined by the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP). The specific constituents analyzed as part of these multi-analyte methods, as well as the other multi-analyte methods, HPLC explosives and GC/ECD PCBs, are provided in Table 7-2.

Non-performance demonstrated methods will be used for: Petroleum Hydrocarbons, total dissolved solids (analyzed by a gravimetric method), Toxicity Characteristic Leaching Procedure (TCLP) volatiles, TCLP semivolatiles, TCLP metals, TCLP herbicides, and TCLP pesticides. These analyses will be performed using standard EPA methods, with specified QA/QC requirements. The quality of the data generated using these methods is comparable to EPA Level III data quality (Data Quality Objectives, 1987).

Field screening measurements will also be collected using portable equipment in order to provide real-time data to assist in the optimization of the field sampling activities and for health and safety purposes. Field measurements such as pH, temperature, conductivity, and volatile organics (using a photoionization detector) will be obtained. The quality of these data is generally comparable to EPA Level I data quality (Data Quality Objectives, 1987).

Table 3-1 presents the data quality objectives for critical measurements in terms of precision, accuracy and completeness for all parameters analyzed for this investigation. The table specifies whether the measurement will be made in the field or in the laboratory. Estimated accuracy is expressed as percent recovery and estimated precision is expressed as a relative percent difference (for two values) or a standard deviation (for three or more values). Completeness is expressed in terms of the percentage of valid data generated out of the total number of data points. The information regarding precision and accuracy of the methods presented in this plan has been obtained from a number of sources. For the EPA methods used in this investigation, the precision and accuracy values come from a program for evaluating analytical methods and laboratories that is directed by the EPA. For the USAEC-performance demonstrated methods precision and accuracy are evaluated as part of the control chart program. All these indicators of data quality are explained in further detail in the sections that follow.

#### **3.2.1 Precision**

Precision is the degree of mutual agreement among individual measurements of the same parameter, using prescribed conditions and a single test procedure. Overall precision includes variability associated with field and laboratory operations. The results of analyzing field duplicate samples are used to assess variability associated with field activities, which is a function of sample collection/handling as well as matrix homogeneity. Analytical precision can be expressed in several ways, including standard deviation, relative standard deviation, range, and relative percent difference (RPD).

### 3.0 QA Objectives for Measurement Data In Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability

**Table 3-1: Data Quality Objectives for Critical Measurements: Precision, Accuracy, and Completeness**

| Lab/Field QC              | Parameter                  | Matrix         | Estimated Accuracy <sup>a</sup> | Estimated Precision <sup>a</sup>  | Completeness |
|---------------------------|----------------------------|----------------|---------------------------------|-----------------------------------|--------------|
| Lab USAEC-PD <sup>1</sup> | TCL VOAs                   | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | TCL SEMIVOAs               | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | TAL Metals                 | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | PCBs                       | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | HPLC Explosives            | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | Chloride                   | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | Nitrate/Nitrite            | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | Sulfate                    | Soil/Sed       | USAEC                           | USAEC, RPD <50% <sup>b</sup>      | 90%          |
| Lab Non-PD <sup>2</sup>   | Hydrocarbons               | Soil/Sed       | 50 - 120%                       | RPD <75% <sup>b</sup>             | 90%          |
| Lab USAEC-PD <sup>1</sup> | TCL VOAs                   | Grd/Surf Water | USAEC                           | USAEC, RPD <30% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | TCL SEMIVOAs               | Grd/Surf Water | USAEC                           | USAEC, RPD <30% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | TAL Metals                 | Grd/Surf Water | USAEC                           | USAEC, RPD <30% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | Chloride                   | Grd/Surf Water | USAEC                           | USAEC, RPD <30% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | Nitrate/Nitrite            | Grd/Surf Water | USAEC                           | USAEC, RPD <30% <sup>b</sup>      | 90%          |
| Lab USAEC-PD <sup>1</sup> | Sulfate                    | Grd/Surf Water | USAEC                           | USAEC, RPD <30% <sup>b</sup>      | 90%          |
| Field Non-PD <sup>3</sup> | pH                         | Ground Water   | +0.2 pH units                   | +0.2 pH units <sup>b</sup>        | 90%          |
| Field Non-PD <sup>3</sup> | Temperature                | Ground Water   | +1°C                            | +1°C <sup>b</sup>                 | 90%          |
| Field Non-PD <sup>3</sup> | Conductivity               | Ground Water   | +2% scale                       | +2% scale <sup>b</sup>            | 90%          |
| Field Non-PD <sup>3</sup> | Turbidity                  | Ground Water   | +2% scale                       | +2% scale <sup>b</sup>            | 90%          |
| Lab Non-PD <sup>4</sup>   | TCLP Volatile Organics     | TCLP Extract   | Compound Dependent              | Compound Dependent                | 90%          |
| Lab Non-PD <sup>4</sup>   | TCLP Semivolatile Organics | TCLP Extract   | Compound Dependent              | Compound Dependent                | 90%          |
| Lab Non-PD <sup>4</sup>   | TCLP Metals                | TCLP Extract   | +15%                            | RPD <10%                          | 90%          |
| Lab Non-PD <sup>4</sup>   | Total Dissolved Solids     | Grd/Surf Water | +20%                            | RPD <30%<br>RPD <50% <sup>b</sup> | 90%          |

Sources:

1. USAEC, Quality Assurance Program, January 1990
2. Modified method based on SW-846 8015 and ASTM D3328-78
3. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983
4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, January 1990

a. For the USAEC-performance demonstrated (PD) methods, the precision and accuracy limits will be based on the historical control chart data of DataChem Laboratories. For the non-performance demonstration methods, the precision will be based on recovery of spikes using USAEC standard soil and water.

b. RPD-DQO is for the analysis of field duplicates.



### **3.0 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability**

- For the USAEC-performance demonstrated methods, laboratory precision is evaluated as part of the control chart program. A three-day moving average control chart is maintained for each control analyte by plotting the range of recovery of spiked QC samples; an updated three-day average range of recovery for each compound is plotted on the control chart as part of the daily laboratory control program. This procedure is intended to monitor variations in the precision of routine analyses and detect trends in observed variations.
- For non-performance demonstrated methods, laboratory precision is generally assessed through the use of laboratory duplicate samples or as specified in the method.

#### **3.2.2 Accuracy**

Accuracy is the difference between individual analytical measurements and the true or expected value of a measured parameter. It is a measure of the bias corresponding to systematic and random errors in the entire data collection process. Sources of error include the sampling process, field and laboratory contamination, sample preservation and handling, sample matrix interferences, sample preparation methods, and calibration and analysis procedures. Sampling accuracy can be assessed, in part, by evaluating the results of analyzing field/trip blanks; analytical accuracy can be evaluated through the use of calibration and method blanks, calibration verification samples, laboratory control samples, and matrix spikes.

- For the USAEC-performance demonstrated methods, accuracy is assessed as part of the control chart program. A three-day moving average control chart is maintained for each control analyte by plotting the recovery of spiked QC samples; an updated three-day average recovery for each compound is plotted on the control chart as part of the daily laboratory control program. This procedure is intended to monitor variations in the accuracy of routine analyses and detect trends in the observed variations.
- For non-performance demonstrated methods, laboratory accuracy is generally assessed through the use of laboratory spiked samples or as specified in the method.

#### **3.2.3 Representativeness**

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, or an environmental condition. A representative sample should possess the same qualities or properties relevant to the investigation as the material under investigation. Representativeness reflects the design of the sampling program; representativeness is maximized by proper selection of sampling locations and collection of a sufficient number of samples. Sampling locations for the FGGM investigations covered in this project were selected using a targeted sampling design.



### 3.0 QA Objectives for Measurement Data In Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability

#### 3.2.4 Completeness

Completeness is defined as the a measure of the amount (percent) of valid data obtained from a measurement system, either field or laboratory, compared to the amount expected from the system. Completeness will be assessed in terms of the actual number and type of sample results received from the laboratory as compared with the planned number and type of results. A target of 90 percent completeness for all field and laboratory data is expected for this project.

#### 3.2.5 Comparability

Comparability addresses the confidence with which one data set can be compared to another. Use of appropriate sampling methods, chain-of-custody procedures, and USAEC-performance demonstrated and EPA-approved analytical methods, as well as adherence to strict QA/QC procedures, provide the basis for uniformity in sample collection and analysis activities.

For this project, data will be considered valid with respect to the comparability objectives if the USAEC acceptance criteria for precision, accuracy, and any other method-specified quality criteria are achieved. This project is being conducted under the USAEC requirements for field sampling activities and laboratory analysis. To the extent possible, USAEC-performance demonstrated methods are being used in a USAEC-performance demonstrated laboratory. For non-performance demonstrated analyses, USAEC requirements have been followed for using standardized methods with appropriate QA/QC protocols to generate data of known quality.

In addition, comparability is assured through the consistent use of units. The data collected as part of this program will be reported in the following units:

| Parameter                             | Water                 | Soil/Sediment |
|---------------------------------------|-----------------------|---------------|
| TCL Volatiles                         | µg/L                  | µg/g          |
| TCL Semivolatiles                     | µg/L                  | µg/g          |
| TCL PCBs                              | µg/L                  | µg/g          |
| TAL Metals                            | µg/L                  | µg/g          |
| HPLC Explosives                       | µg/L                  | µg/g          |
| IC Chloride, Sulfate, Nitrate/Nitrite | µg/L                  | µg/g          |
| Total Dissolved Solids                | µg/L                  | NA            |
| Petroleum Hydrocarbons                | µg/L                  | µg/g          |
| pH                                    | pH Units              | pH Units      |
| Temperature                           | °C                    | NA            |
| Conductivity                          | µmhos/cm <sup>2</sup> | NA            |
| Turbidity                             | NTU                   | NA            |



### **3.0 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability**

Areas of concern were selected to address data gaps from previous investigations; sampling locations will be identified based on existing information and field survey data. Parameter variations at a sampling point can be evaluated on the basis of field duplicate results.

## 4.0 Sample Collection

The quality of the data collected for the FGGM investigations is a function of the overall design and planning of the sample collection program and the specific sample collection and handling procedures employed. In addition to the collection of samples, activities included within the sample collection and handling phase of field investigations include preparation of sample containers, sample preservation, sample identification, sample handling and shipment, and chain-of-custody documentation.

### 4.1 Sampling Program for FGGM Investigations

The sampling program for FGGM is described in the Work Plan, provided as a separate document, and is summarized on Table 4-1. In order to ensure that collected field samples are representative of the matrices under investigation and to ensure that the physical and chemical integrity of the samples is maintained prior to analysis in the subcontracted laboratory, detailed procedures for all aspects of sample collection and handling have been specified. These procedures comply with USAEC and EPA specifications and guidelines for the collection of environmental samples. The following sections summarize these procedures; a list of the Standard Operating Procedures (SOPs) that will be followed by the Arthur D. Little sampling staff is provided in Appendix B.

### 4.2 Sampling Equipment and Procedures

The various sampling and data collection procedures that will be followed during completion of the FGGM SIA and RIA field investigation activities are presented below, and include discussions of the various sampling and data acquisition equipment which will be used for each activity. All SOPs referenced in this section are listed in Appendix B.

#### 4.2.1 Geologic Characterization and Soil Quality Assessment

An exploratory boring and surface soil sampling program will be conducted to collect soil samples from the near surface, and at depth, for geotechnical and chemical characterizations of the various subsurface environments near suspected contaminant source areas. This program will also provide the means for installing ground water monitoring wells at some locations so that the ground water quality and specific hydraulic characteristics of the various subsurface environments can be monitored and evaluated.

**4.2.1.1 Subsurface Clearance Program.** The final location of each surface soil sample and exploratory borehole will be determined prior to drilling.

**4.2.1.2 Exploratory Boring Program.** Each exploratory boring will be advanced in accordance with SOP USA-4001, using a truck-mounted hydraulic hollow stem auger drill rig that has the capability of converting to a drive and wash drilling method, as necessary.



Table 4-1: Analytical Program Summary

| SOIL/SOLID SAMPLES                |    | LOC FREQ |    | TCL SVOCs |    | TCL VOCs |   | PHC | FILTERED METALS |    | UNFILTERED METALS |   | TCLP ORGANICS/ METALS |    | PCB | EXP | CI | NO3 | TDS | SO4 | PEST |
|-----------------------------------|----|----------|----|-----------|----|----------|---|-----|-----------------|----|-------------------|---|-----------------------|----|-----|-----|----|-----|-----|-----|------|
| SOIL SAMPLES                      |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
| DPDO Salvage Area                 | 6  | 1        | 0  | 0         | 0  | 0        | 0 | 0   | 0               | 0  | 0                 | 0 | 0                     | 6  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Ordnance Demolition Area          | 4  | 3        | 0  | 0         | 0  | 0        | 0 | 0   | 0               | 12 | 0                 | 0 | 0                     | 0  | 12  | 0   | 0  | 0   | 0   | 0   | 0    |
| Helicopter Hangar Area - shallow  | 4  | 1        | 4  | 4         | 4  | 4        | 4 | 4   | 0               | 4  | 0                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Helicopter Hangar Area - deep     | 1  | 1        | 0  | 0         | 1  | 1        | 1 | 1   | 0               | 0  | 0                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Background                        | 30 | 1        | 0  | 0         | 0  | 0        | 0 | 0   | 0               | 30 | 0                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 30   |
| SEDIMENT SAMPLES                  |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
| Helicopter Hangar Area            | 5  | 1        | 5  | 5         | 5  | 5        | 5 | 5   | 0               | 5  | 0                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Ordnance Demolition Area          | 1  | 1        | 0  | 0         | 1  | 0        | 0 | 0   | 0               | 0  | 0                 | 0 | 0                     | 0  | 1   | 0   | 0  | 0   | 0   | 0   | 0    |
| Active Sanitary Landfill          | 6  | 1        | 6  | 6         | 6  | 6        | 6 | 6   | 0               | 6  | 0                 | 0 | 0                     | 0  | 0   | 6   | 6  | 6   | 0   | 6   | 0    |
| SLUDGE SAMPLES                    |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
| Fire Training Area                | 1  | 1        | 1  | 1         | 1  | 1        | 1 | 1   | 0               | 0  | 0                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| INVESTIGATION-DERIVED WASTE       |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
|                                   | 6  | 1        | 0  | 0         | 0  | 0        | 0 | 0   | 0               | 0  | 6                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| TOTAL SOIL/SEDIMENT SAMPLES       |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
|                                   | 16 | 18       | 11 | 0         | 57 | 6        | 6 | 13  | 6               | 6  | 6                 | 0 | 0                     | 6  | 0   | 6   | 30 |     |     |     |      |
| QUALITY CONTROL SAMPLES           |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
| FIELD QC SAMPLES                  |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
| FIELD BLANK                       | 2  | 2        | 1  | 0         | 3  | 0        | 0 | 1   | 1               | 1  | 0                 | 0 | 1                     | 1  | 1   | 1   | 1  | 1   | 0   | 1   | 0    |
| RINSEATE BLANK                    | 3  | 3        | 2  | 0         | 4  | 0        | 0 | 2   | 2               | 2  | 0                 | 0 | 2                     | 2  | 2   | 2   | 2  | 2   | 0   | 2   | 0    |
| TRIP BLANK                        | 0  | 7        | 0  | 0         | 0  | 0        | 0 | 0   | 0               | 0  | 0                 | 0 | 0                     | 0  | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| FIELD COLLOCATE                   | 4  | 4        | 3  | 0         | 4  | 0        | 0 | 3   | 0               | 4  | 0                 | 0 | 1                     | 1  | 1   | 1   | 1  | 1   | 0   | 1   | 0    |
| TOTAL SOIL/SEDIMENT QC SAMPLES    |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
|                                   | 9  | 16       | 6  | 0         | 11 | 0        | 0 | 4   | 4               | 4  | 0                 | 0 | 4                     | 4  | 4   | 4   | 4  | 4   | 0   | 4   | 0    |
| GRAND TOTAL SOIL/SEDIMENT SAMPLES |    |          |    |           |    |          |   |     |                 |    |                   |   |                       |    |     |     |    |     |     |     |      |
|                                   | 25 | 34       | 17 | 0         | 68 | 6        | 6 | 10  | 17              | 10 | 10                | 0 | 10                    | 10 | 10  | 10  | 10 | 10  | 0   | 10  | 30   |

Table 4-1 Analytical Program Summary (continued)

| AQUEOUS SAMPLES                            |     | LOC | FREQ | SVOCS | TCL | VOCs | PHC | METALS | TAL | METALS | TAL | ORGANICS/<br>METALS | PCB | EXP | CI | NO3 | TDS | SO4 | PEST |
|--------------------------------------------|-----|-----|------|-------|-----|------|-----|--------|-----|--------|-----|---------------------|-----|-----|----|-----|-----|-----|------|
| <b>GROUND WATER SAMPLES</b>                |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
| Inactive Landfill #2                       | 6   | 1   | 0    | 0     | 0   | 0    | 0   | 6      | 6   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| DPDO Salvage Yard - Existing Wells         | 5   | 1   | 5    | 5     | 0   | 0    | 0   | 5      | 5   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| DPDO Salvage Yard - New Wells              | 2   | 1   | 2    | 2     | 0   | 0    | 0   | 2      | 2   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Fire Training Area                         | 3   | 1   | 3    | 3     | 3   | 3    | 3   | 3      | 3   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Helicopter Hangar Area                     | 6   | 1   | 6    | 6     | 6   | 6    | 6   | 6      | 6   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Ordnance Demolition Area                   | 3   | 1   | 3    | 3     | 0   | 0    | 0   | 3      | 3   | 0      | 0   | 0                   | 0   | 3   | 0  | 0   | 0   | 0   | 0    |
| Active Landfill - Water Supply Well Survey | 4   | 1   | 4    | 4     | 0   | 0    | 0   | 4      | 4   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Active Landfill - Existing Wells           | 26  | 1   | 26   | 26    | 0   | 0    | 0   | 26     | 26  | 0      | 0   | 0                   | 0   | 0   | 26 | 26  | 26  | 26  | 0    |
| Active Landfill - New Wells                | 3   | 1   | 3    | 3     | 0   | 0    | 0   | 3      | 3   | 0      | 0   | 0                   | 0   | 0   | 3  | 3   | 3   | 3   | 0    |
| Clean Fill Dump                            | 6   | 1   | 6    | 6     | 0   | 0    | 0   | 6      | 6   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| <b>SURFACE WATER SAMPLES</b>               |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
| Helicopter Hangar Area                     | 5   | 1   | 5    | 5     | 5   | 0    | 0   | 5      | 5   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Ordnance Demolition Area                   | 1   | 1   | 0    | 1     | 0   | 0    | 0   | 0      | 0   | 0      | 0   | 0                   | 0   | 1   | 0  | 0   | 0   | 0   | 0    |
| Active Landfill                            | 6   | 1   | 6    | 6     | 0   | 0    | 0   | 6      | 6   | 0      | 0   | 0                   | 0   | 0   | 6  | 6   | 6   | 6   | 0    |
| Clean Fill Dump - Existing Location        | 1   | 1   | 1    | 1     | 0   | 0    | 0   | 1      | 1   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Clean Fill Dump - New Locations            | 2   | 1   | 2    | 2     | 0   | 0    | 0   | 2      | 2   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Soldiers Lake                              | 2   | 1   | 0    | 0     | 0   | 0    | 0   | 2      | 2   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 2    |
| <b>LEACHATE</b>                            |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
| Active Landfill                            | 3   | 1   | 3    | 3     | 0   | 0    | 0   | 3      | 3   | 0      | 0   | 0                   | 0   | 0   | 3  | 3   | 3   | 3   | 0    |
| <b>SEEPS</b>                               |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
| Inactive Landfill #2                       | 1   | 1   | 1    | 1     | 0   | 1    | 0   | 1      | 1   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| Clean Fill Dump                            | 1   | 1   | 1    | 1     | 0   | 1    | 0   | 1      | 1   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| <b>DRILLING WATER SUPPLY</b>               |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
|                                            | 1   | 2   | 2    | 2     | 2   | 2    | 2   | 2      | 2   | 0      | 0   | 0                   | 0   | 2   | 2  | 2   | 2   | 2   | 0    |
| <b>INVESTIGATION-DERIVED WASTE</b>         |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
|                                            | 2   | 1   | 0    | 0     | 0   | 0    | 0   | 0      | 0   | 2      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| <b>TOTAL AQUEOUS SAMPLES</b>               |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
|                                            | 79  | 80  | 16   | 70    | 87  | 2    | 0   | 6      | 40  | 40     | 40  | 40                  | 40  | 40  | 40 | 40  | 40  | 40  | 2    |
| <b>QUALITY CONTROL SAMPLES</b>             |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
| FIELD BLANK                                | 8   | 8   | 2    | 8     | 8   | 0    | 0   | 3      | 6   | 6      | 6   | 6                   | 6   | 3   | 0  | 0   | 0   | 0   | 0    |
| FNSATE BLANK                               | 8   | 8   | 2    | 8     | 8   | 0    | 0   | 3      | 6   | 6      | 6   | 6                   | 6   | 3   | 0  | 0   | 0   | 0   | 0    |
| TRIP BLANK                                 | 0   | 16  | 0    | 0     | 0   | 0    | 0   | 0      | 0   | 0      | 0   | 0                   | 0   | 0   | 0  | 0   | 0   | 0   | 0    |
| FIELD DUPLICATE                            | 7   | 7   | 1    | 8     | 8   | 0    | 0   | 0      | 6   | 6      | 6   | 6                   | 6   | 0   | 0  | 0   | 0   | 0   | 0    |
| <b>TOTAL AQUEOUS QC SAMPLES</b>            |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
|                                            | 23  | 39  | 5    | 24    | 24  | 0    | 0   | 6      | 18  | 18     | 18  | 18                  | 18  | 6   | 0  | 0   | 0   | 0   | 0    |
| <b>GRAND TOTAL AQUEOUS SAMPLES</b>         |     |     |      |       |     |      |     |        |     |        |     |                     |     |     |    |     |     |     |      |
|                                            | 102 | 119 | 21   | 94    | 111 | 2    | 0   | 12     | 58  | 58     | 58  | 58                  | 58  | 8   | 0  | 0   | 0   | 0   | 0    |

## NOTES:

LOC - Locations

FREQ - Frequency

TCL, VOCs - Volatile Organics, Target Compound List

TCL, SEMIVOCs - Semivolatile Organics, Target Compound List

PHC - Petroleum hydrocarbons

TCLP - Toxicity Characteristic Leaching Procedure

PCB - Polychlorinated biphenyls

EXP - Explosives

METALS, TAL - Metals on the Target Analyte List  
Analyte lists are provided in the ADL QCP, 1993.



## 4.0 Sample Collection

All drilling supplies will be maintained by the drilling subcontractor. These supplies are likely to include extra hollow stem augers, steel casing, well construction materials (e.g., PVC well screens and riser pipe, bags of sand packing material, buckets of bentonite pellets, and bags of grout), and well completion materials (e.g., protective steel surface casings and concrete).

Each drill rig and all drilling equipment such as hollow stem augers, steel casing, drill rods, mud tubs, and split spoon samplers will be steam cleaned immediately prior to initiation of drilling activities. The drilling subcontractor will supply steam cleaners and water trucks (as necessary). Drill water will be obtained from a tested and approved location during the mobilization subtask.

Decontamination of all sampling equipment will be conducted prior to each use in accordance with the Geotechnical Requirements. Each drill rig and all drilling equipment will be decontaminated prior to arrival on site, prior to relocation on site, and prior to leaving the site as specified in SOP USA-4001. Drill rig and drilling equipment will be decontaminated in an area designated for this activity by the Base Commander through the USAEC Project Officer.

Split spoon sampling at each drilling location will occur at the ground surface and at 5 foot intervals. Split spoon sampling procedures will be performed in accordance with SOP USA-4002. The final depth of these borings is dependent upon local stratigraphy and contaminant levels detected in each borehole and in surrounding bore hole locations. The procedures for installing ground water monitoring wells in designated boreholes is presented in Section 4.2.2.1 of this plan. For exploratory borings that will not have monitoring wells installed, the borehole will be abandoned in accordance with SOP USA-4003.

**4.2.1.3 Surface Soil Sampling Program.** Two types of shallow soil samples will be collected by Arthur D. Little personnel using a decontaminated stainless steel hand auger. The hand auger will be rinsed with distilled water prior to collection of each sample designated for chemical analyses. Soil samples will be collected as follows:

- Six surface soil sample will be collected from 0 to 6 inches to evaluate if stained areas have affected the soil quality.
- Three shallow soil samples will be collected from each investigation site at FGGM at depths of 2 to 3 feet.
- Each of the sampling locations will be cleared of surface debris and vegetation to expose fresh soil.



## 4.0 Sample Collection

- The sample collected from each sampling location will be composited in a stainless steel bowl prior to distribution into the various chemical sample jars. However, if a sample is scheduled for volatile organic compound analysis, the appropriate sample bottle will be filled using a representative portion of soil from the first portion of soil at depth.
- Completion of sampling activities will include the return of auger spoils to the borehole or survey flag and the placement of a stake painted florescent orange and marked with the sample point code number for future reference.

Documentation of these procedures will be maintained in a dedicated field notebook and on appropriate field sampling forms in accordance with SOP ADL-4014. Records will include detailed sketches of each sample location for future reference, and each location will also be plotted on the detailed site basemap.

### 4.2.2 Ground Water Quality Assessment

A series of ground water investigations, including ground water quality and hydraulic flow investigations, will be conducted at a majority of the FGGM SIA, RIA, and FS field areas. The objective of the ground water quality investigation is to collect representative ground water samples from discrete hydraulic zones within the subsurface for chemical analyses. The objective of the hydraulic survey is to identify hydraulic flow gradients within the subsurface. The results of the ground water chemical analyses will be used to determine the concentrations and distributions of detected chemicals within the various hydraulic flow regimes. The hydraulic data, in conjunction with geologic and location/elevation data, form the basis for theoretical chemical transport evaluations. All water level measurements will be collected in accordance with SOP USA-4012.

#### 4.2.2.1 Ground Water Monitoring Well Installation and Development Program.

All ground water monitoring wells will be constructed in accordance with SOP USA-4008, and will generally include a 4-inch diameter, 10-foot length, of slotted PVC screen with a 4-inch diameter solid PVC riser extending to approximately 2.5 feet above the ground surface. Each well will be constructed with a sandpack filling the annular space around the screened interval from no more than 3-feet below the bottom of the well screen to a minimum of 5-feet above the top of the screen. A bentonite seal will be placed above the sand pack with a maximum slurry thickness, or a minimum pellet thickness, of 5-feet. Each PVC well will covered with a PVC slip-cap and protected with a locking steel standpipe and surface finish in conformance with the USATHAMA *Geotechnical Requirements for Drilling, Monitor Wells, Data Acquisition, and Reports* (March 1987).

Each newly installed ground water monitoring well will be developed to restore the aquifer's hydraulic conductivity and to remove well drilling fluids, solids, and other mobile particles from within, and adjacent to, the newly installed well. Well development will be conducted in accordance with SOP USA-4010 no sooner than 48



## 4.0 Sample Collection

consecutive hours after, nor longer than 7 calendar days beyond, initial mortar collar placement.

**4.2.2.2 Ground Water Monitoring Well Sampling Program.** Ground water samples will be collected in accordance with SOP USA-1011 from all monitoring wells identified in the Work Plan. For newly constructed wells, ground water sampling will be conducted no sooner than 14 days after well development. The depth to water, total well depth, and thickness of any free-phase product that may be present will be measured and recorded in accordance with SOP USA-4012 prior to ground water sampling. A total of five purge volumes will be removed from the well immediately prior to sampling. The purge volume for each well includes the volume of standing water in the well plus the volume of water in the annular space surrounding the well over the same height. The volume of water within the annular space assumes 30 percent porosity.

Immediately upon initiation and at completion of purging, the following aquifer stabilization parameters will be measured and recorded: pH, temperature, specific conductivity, and turbidity. All purging and sampling procedures will be conducted using a decontaminated, chemically inert, variable flow, submersible, pump. All sample bottles and lids will be rinsed with the well water prior to filling, except for the volatile sample vials. Each sample that requires filtering will be collected by attaching an in-line, 0.45 micron, disposable filter to the pump outflow. A new filter will be used at each sampling location. All samples will be preserved in the field as described in Section 4.3.2.

**4.2.2.3 Drinking Water Supply Well Sampling Program.** Residential drinking water wells are to be purged and sampled using the same procedures defined above for ground water sampling, with the following exceptions:

- Specific details regarding actual well location and construction details will be obtained from residents prior to sampling.
- Household residents will be interviewed to determine whether any water purifiers, filters, or softeners are in use, and if so, whether these systems can be bypassed prior to sample collection. If a drinking water sample can not be collected without passing through these systems, specific details regarding each system with which the sample passed prior to collection will be recorded by the sampler in a dedicated field notebook.
- Well purging will be conducted by opening a household faucet. If an aerator is present on the faucet, the aerator will be removed prior to purging and sampling.
- Well purging will be accomplished by allowing the water to flow from the open cold water faucet a minimum of 30 minutes prior to sampling to assure that fresh water from the aquifer is available for sampling.

## 4.0 Sample Collection

- Water quality parameters (i.e., pH, temperature, specific conductivity, and turbidity) will be collected at the purge faucet upon initiation and completion of purging.
- Upon completion of sampling the aerator will be replaced and a sample receipt will be presented to the homeowner.

### 4.2.3 Surface Water and Seep Sampling Procedures

Surface water and seep samples will be collected in conformance with the procedures set forth in Section C.3.3.1.3 of the USAEC TEPS Contract DAA15-90-R-0120 as follows:

- Surface water and seep samples will be collected from seeps, ditches, streams, lakes and rivers during periods of moderate flow. Precipitation records for the week prior to sampling will be maintained to confirm the relative flow state.
- The surface water column will be measured and recorded using a weighted tape. The position of the sampling point to the shoreline will also be measured and recorded. Records will include detailed sketches of each sample location for future reference. Each location will also be plotted on the detailed site basemap.
- Continuous vertical profile temperature measurements will be collected along the water column of each pond and lake surface water sampling location to determine the presence of a thermocline. If a thermocline is present, surface water samples will be collected both above and below the thermocline depth using a decontaminated stainless steel discrete bomb sampler.
- Samples from ditches, streams, lagoons, and ponds will be taken at approximately one half to two thirds of the water depth using a decontaminated stainless steel discrete bomb sampler. In cases where the depth to water is less than 1-foot, samples will be collected by direct submergence of the sample containers.
- At locations where surface seeps provide insufficient flow to fill sample containers by direct submergence, a shallow sample collection basin will be established by installing a 2-foot length of 4-inch diameter slotted PVC well screen into the subsurface approximately 1.5 feet then placing a PVC slip cap over the sampling port.
- The pH, temperature, specific conductivity, and turbidity of each surface water and seep sample will be measured immediately prior to collection.
- All sample containers and lids, except for the volatile sample vials, will be rinsed with the sampled surface water prior to filling.



## **4.0 Sample Collection**

### **4.2.4 Sediment Sampling Procedures**

Each sediment sample will be collected using a procedure similar to that described for Surface Soil Sampling (Section 4.2.1.3) with the following exceptions:

- The surface water column above each sediment sampling location will be measured and recorded using a weighted tape. The position of the sampling point to the shoreline will also be measured and recorded. Records will include detailed sketches of each sample location for future reference. Each location will also be plotted on the detailed site basemap.
- For sediment collection below relatively shallow surface water bodies (i.e., less than four feet deep) the sampling location will be accessed by the sampler from the downstream direction to minimize disruption of bottom sediment in the sample area. The sampler will be wearing chest waders and will be accompanied by a co-worker who will observe activities from shore in case of emergency and will document all sampling activities.
- For sediment collection below relatively deep surface water bodies (i.e., greater than four feet deep) the sampling location will be accessed by boat with a two-person crew (one to maintain position and document activities and one to perform sample collection). Samples will be collected using either a decontaminated stainless steel hand auger or a weighted stainless steel dredge.

### **4.2.5 Location and Elevation Survey**

All sampling points will be plotted on an installation map provided by the USAEC COR. Where sediment, soil, and surface water samples are involved, sampling point coordinates (Universal Transverse Mercator) will be established from a USGS Topographic Map. The location and elevation of all newly installed ground water monitoring wells will be determined by a licensed surveyor within 15 days of completion of the last monitoring well. All locations will be recorded in a dedicated field notebook, entered in the USAEC data management system, and located on an installation map.

### **4.2.6 Investigation-Derived Waste**

Waste generated during the field investigation are managed under this subtask. Potentially hazardous wastes generated include drill cuttings, drill fluids, development water, decontamination fluids, and protective clothing.

In accordance with Section C3.1.9 (Disposal of Wastes Generated Incidental to Investigations) of the basic contract, the contractor shall containerize all soil cuttings, drilling mud, drilling water, decontamination fluids, and other investigation-derived wastes. The contractor shall provide for the characterization of this waste in order to determine the appropriate disposal requirements.

Composite samples will be collected from the drummed materials and will be analyzed by RCRA Toxicity Characteristic Leaching Procedure (TCLP) for organics



## 4.0 Sample Collection

and metals. If the material is not classified as a RCRA hazardous waste according to the TCLP analysis, it will be disposed of at FGGM at locations specified by the Environmental Officer. If the material is classified as a RCRA hazardous waste, it will be disposed of in accordance with 40 CFR Part 262, Standards Applicable to Generators of Hazardous Waste and the FGGM Environmental Officer. Sampling of the investigation derived waste drums will be completed under this subtask. We have assumed that we will be able to composite representative sample aliquots from two to three drums into a single sample for analysis.

We will select individual drums for compositing on the basis of obtaining the material from the sample location and visual similarities. We have assumed that the drilling subcontractor will provide support in moving the drums from the point of generation to a common accumulation area to be designated. A licensed hazardous material disposal firm will be engaged to provide the transport and disposal of the RCRA hazardous waste generated during this investigation. We expect, however, the FGGM staff will issue any necessary manifests, including FGGM as the waste generator.

We expect to be able to dispose of non-hazardous wastes generated during the investigation, such as packing materials, in FGGM waste handling facilities.

## 4.3 Sample Containers, Preservation, and Handling

### 4.3.1 Sample Containers

To ensure the integrity of the field samples, specific steps must be taken to minimize the potential for contamination from the containers in which the samples are stored. Sample containers must be compatible with the analytes of interest; a complete list of sample containers is provided by USAEC for analytical samples collected in support of the Installation Restoration Program. The following general recommendations will be followed: septum-sealed amber glass vial for volatile compounds; amber glass bottles with Teflon-lined lids for organic compounds other than volatiles; polyethylene bottles for inorganic analytes; and wide-mouth amber glass bottles for all soil and sediment samples. The sample containers required for the collection of the various analytical samples for the FGGM investigation are indicated in Table 4-2. A complete list of the recommended sample containers is provided in the appropriate SOP listed in Appendix B.

For the FGGM investigation, all sample containers will be supplied by the subcontracted laboratory, which is performance demonstrated to perform USAEC analyses. All sample containers will be cleaned prior to shipment to the field. Cleaning procedures will be applied to new containers; reuse of sample containers is expressly prohibited. The cleaning procedures used by the laboratory are described in the appropriate SOP provided in Appendix C to this plan. These procedures meet the specifications of the sample container cleaning procedures outlined in the USAEC QA Program.



## 4.0 Sample Collection

### 4.3.2 Sample Preservation and Holding Times

The purpose of sample preservation is to prevent or retard the degradation or transformation of target analytes in the field samples during transport and storage. Preservation efforts to ensure sample integrity will be initiated at the time of sampling and will continue until the analyses are performed. Preservatives will be added to the sample container at the time of sample collection. The required preservatives for specific analytical samples to be collected for the FGGM investigation are indicated in Table 4-2; a complete list of required preservation procedures is contained in the appropriate SOP provided in Appendix B.

Chemical preservatives will be supplied to the field by the USAEC-performance demonstrated analytical laboratory subcontracted for this project. Bottles for aqueous samples will be triple-rinsed with the water being sampled, according to USAEC requirements, before the addition of preservatives, except for the volatile sample vials. For volatiles analyses, the preservative will be added before sample container is filled; for all other analyses, the sample container will be filled and then the preservative will be added.

After collection and preservation, all samples will be stored and shipped at 4 degrees Celsius. Samples will be sent to the laboratory for analysis as expeditiously as possible to ensure data quality. The recommended maximum holding times for analytical samples are indicated in Table 4-2; maximum holding times are calculated from the date of sample collection. The indicated holding times will be adhered to by the laboratory subcontracted for analysis of the FGGM samples. Freezing of samples to extend the holding time is not permitted.

### 4.4 Field Quality Control Samples

Field Quality Control (QC) samples to be collected as part of the FGGM field investigation are included in Table 4-1. The following types of QC samples will be included at a rate of 1 per lot or 1 per 20 field samples, per sampling technique:

- The results of analyzing field blanks are used to check the cleanliness and effectiveness of field handling methods.
- The results of analyzing trip blanks are used to assess potential contamination during sample transport.
- The results of analyzing equipment/rinsate blanks are used to evaluate potential cross-contamination from field sampling equipment, and the effectiveness of the decontamination procedures.
- The results of analyzing field duplicates/collocates are used for assessing the consistency of the field and analytical program.

#### 4.0 Sample Collection

**Table 4-2: Containers, Preservation, and Holding Times for Analytical Samples**

| <b>Analysis</b>                      | <b>Sample Containers</b>                            | <b>Preservation</b>      | <b>Holding Times</b>                            |
|--------------------------------------|-----------------------------------------------------|--------------------------|-------------------------------------------------|
| TCL Volatiles<br>- water             | Two 40-mL amber glass VOA vials, Teflon-lined cap   | HCl to pH<2<br>Cool, 4°C | 14 days                                         |
| TCL Volatiles<br>- soil/sediment     | 250-mL amber wide-mouth glass jar, Teflon-lined cap | Cool, 4°C                | 14 days                                         |
| TCL Semivolatiles<br>- water         | 1-L amber glass jar, Teflon-lined cap               | Cool, 4°C                | 7 days to extraction; 40 days after extraction  |
| TCL Semivolatiles<br>- soil/sediment | 250-mL amber wide-mouth glass jar, Teflon-lined cap | Cool, 4°C                | 7 days to extraction; 40 days after extraction  |
| PCBs<br>- water                      | 1-L amber glass bottle, Teflon-lined cap            | Cool, 4°C                | 7 days to extraction; 40 days after extraction  |
| PCBs<br>- soil/sediment              | 250-mL amber wide-mouth glass jar, Teflon-lined cap | Cool, 4°C                | 7 days to extraction; 40 days after extraction  |
| Explosives<br>- soil/sediment        | 250-mL amber wide-mouth glass jar, Teflon-lined cap | Cool, 4°C                | 7 days to extraction; 40 days after extraction* |
| Explosives<br>- water                | 1-L amber glass bottle, Teflon-lined cap            | Cool, 4°C                | 7 days to extraction; 40 days after extraction  |
| TAL Metals (ICP/GFAA)<br>- water     | 1-L Polyethylene bottle, Teflon-lined cap           | HNO <sub>3</sub> to pH<2 | 6 months                                        |



## 4.0 Sample Collection

**Table 4-2: Containers, Preservation, and Holding Times for Analytical Samples (continued)**

| <b>Analysis</b>                                        | <b>Sample Containers</b>                                                 | <b>Preservation</b>                                 | <b>Holding Times</b>                           |
|--------------------------------------------------------|--------------------------------------------------------------------------|-----------------------------------------------------|------------------------------------------------|
| TAL Metals<br>(ICP/GFAA)<br>-soil/sediment             | 250-mL amber wide-mouth glass jar,<br>Teflon-lined cap                   | Cool, 4°C                                           | 6 months                                       |
| Mercury<br>- water                                     | 1-L polyethylene bottle, Teflon-lined cap                                | HNO <sub>3</sub> to pH<2                            | 28 days                                        |
| Mercury<br>- soil/sediment                             | 250-mL amber wide-mouth glass jar,<br>Teflon-lined cap                   | Cool, 4°C                                           | 28 days                                        |
| Chloride/Sulfate<br>- water                            | 250-mL polyethylene bottle                                               | Cool, 4°C                                           | 28 days                                        |
| Chloride/Sulfate<br>- soil/sediment                    | 250-mL amber wide-mouth glass jar                                        | Cool, 4°C                                           | 28 days                                        |
| Nitrate plus Nitrite<br>- water                        | 250-mL polyethylene bottle                                               | Cool, 4°C<br>H <sub>2</sub> SO <sub>4</sub> to pH<2 | 28 days                                        |
| Nitrate plus Nitrite<br>- soil/sediment                | 250-mL amber wide-mouth glass jar                                        | Cool, 4°C                                           | 28 days                                        |
| Total Dissolved Solids (TDS)<br>- water                | 250-mL polyethylene bottle                                               | Cool, 4°C                                           | 7 days                                         |
| Total Petroleum Hydrocarbons (TPHC)<br>- water         | 1-L amber glass bottle, Teflon-lined cap                                 | Cool, 4°C<br>H <sub>2</sub> SO <sub>4</sub> to pH<2 | 7 days to extraction; 40 days after extraction |
| Total Petroleum Hydrocarbons (TPHC)<br>- soil/sediment | 250-mL amber wide-mouth glass jar,<br>Teflon-lined cap                   | Cool, 4°C                                           | 28 days                                        |
| TCLP Analytes<br>-water                                | Two 40-mL VOA vials and Two 1-L amber glass bottles,<br>Teflon-lined cap | Cool, 4°C                                           | **                                             |

#### 4.0 Sample Collection

**Table 4-2: Containers, Preservation, and Holding Times for Analytical Samples (continued)**

| <b>Analysis</b>                 | <b>Sample Containers</b>                                       | <b>Preservation</b> | <b>Holding Times</b> |
|---------------------------------|----------------------------------------------------------------|---------------------|----------------------|
| TCLP Analytes<br>-soil/sediment | Two 250-mL amber<br>wide-mouth glass<br>jars, Teflon-lined cap | Cool, 4°C           | **                   |

\* The holding times for the Explosives analysis were specified by USAEC.

\*\* The analytical holding times for the TCLP samples are provided below.

| <b>TCLP Analysis</b>              | <b>Max. Time:<br/>Sampling to<br/>TCLP<br/>Extraction</b> | <b>Max. Time:<br/>TCLP<br/>Extraction to<br/>Sample Prep.</b> | <b>Max. Time:<br/>Sample Prep.<br/>to Analysis</b> | <b>Max. Total<br/>Elapsed Time<br/>from Sample<br/>Collection</b> |
|-----------------------------------|-----------------------------------------------------------|---------------------------------------------------------------|----------------------------------------------------|-------------------------------------------------------------------|
| Volatiles                         | 14 days                                                   | -                                                             | 14 days                                            | 28 days                                                           |
| Semivolatiles/<br>Pesticides/PCBs | 7 days                                                    | 7 days                                                        | 40 days                                            | 54 days                                                           |
| Metals                            | 180 days                                                  | -                                                             | 180 days                                           | 360 days                                                          |
| Mercury                           | 28 days                                                   | -                                                             | 28 days                                            | 56 days                                                           |

Source: USAEC Quality Assurance Program (January 1990). TCLP information was taken from 40 CFR 261.



## 4.0 Sample Collection

The field QC samples will be treated by the laboratory as field samples. The purpose of the field QC samples and the frequency of collection are further discussed in Section 9.0 of this Project QC Plan. The QC samples are described in Section 9.1.

## 4.5 Sample Handling

All samples, including field duplicates, trip blanks, and equipment blanks will be maintained in a manner that assures the integrity and representativeness of each sample from the time of collection to laboratory analysis. This maintenance includes the accurate completion of all required documents and the secure packaging of samples prior to transport and shipment. Secure packaging includes the following steps:

- Each sample label is individually wrapped in clear tape to protect the label from water damage, and to assure the sample label is not detached from the sample.
- Each sample bottle will be individually wrapped in bubblewrap to reduce the potential for breakage during transport.
- All samples associated with a shipment will be placed in a rigid pre-cooled container with ample coolant to maintain the samples at 4°C during transport and shipping.
- Individual cooler packing lists and chain-of-custody forms will be placed inside the coolers and will accompany each sample shipment.
- Any open space remaining in the cooler(s) will be filled with bubblewrap to eliminate motion within the cooler.
- Each packed cooler will have a signed and dated custody seal placed across the opening to ensure that the cooler will not be opened until it reaches the laboratory.
- Each cooler custody seal will be protected with clear tape to insure its integrity during transport and shipping.
- The individual shipping numbers will be maintained in a field notebook in case tracking of the shipment is required.

## 5.0 Sample Custody

This section describes procedures for sample chain-of-custody to be followed by Arthur D. Little sampling personnel and the subcontracted laboratory. The primary objective of the chain-of-custody procedures is to provide an accurate written record that can be used to trace the possession and handling of a sample from the moment of its collection through its analyses. A sample is considered to be in custody if it is: in someone's physical possession; in someone's view; locked up; or kept in a secured area that can only be accessed by authorized personnel.

The purpose of these procedures is to ensure that the quality of the samples is maintained during sample collection, transportation, storage, and analysis.

Sample identification documents must be carefully prepared so that sample identification and chain-of-custody can be maintained and sample disposition controlled. Sample identification documents include field notebooks, sample labels, custody seals, and chain-of-custody records. Example of the custody forms are provided in Figures 5-1 and 5-2.

### 5.1 Field Custody Procedures

The field custody procedures to be followed by the field sampling crew are summarized in this section. The specific field custody SOPs to be used during this investigation are listed in Appendix B to this Project QC Plan. All SOPs have been prepared in accordance with the programmatic QA requirements specified by USAEC and EPA.

All samples collected for chemical analysis during the performance of the FGGM SIA and RIA are assigned unique sample designation codes so that all chemical and physical data collected in association with each sample can be directly linked to a specific location, depth, time, and sample media prior to interpretation. Each assigned sample designation code is composed of a predetermined Site Location Identity (SLI) and a Unique Sample Code (USC). The SLI is composed of an alphanumeric code which includes the IRDMIS Site ID, Site Type, and Media Code. The USC provides further detail on the area identification, sample interval, and sample media. The SLI will remain consistent for all samples collected from a single location, regardless of depth, and may therefore correspond to several data sets from a particular event. The USC, when compared with the SLI, serves to uniquely delineate a data set. All sampling locations that were established previously and which are scheduled for resampling during these field activities will use the previously established Site ID to maintain consistency with USAEC's IRDMIS. All newly established sites will be assigned Site IDs consistent with those already in existence.

To enhance sampling efficiency, field documentation accuracy, and database management activities, Arthur D. Little has developed a proprietary automated sample labeling and tracking system that includes the use of bar codes. The system is operated in the field during sampling activities by the site coordinator as follows:



## 5.0 Sample Custody

Figure 5-1: Example of Sample Tracking Identification Label

| Arthur D Little Sample Tracking Systems                                             |                       | USAEC          |
|-------------------------------------------------------------------------------------|-----------------------|----------------|
| November 1992                                                                       | Sample #1             |                |
| Ft. Devens SA32/RI                                                                  | 5 Feet/Regular Sample |                |
| Chemical Soil/Bore Hole                                                             | Pesticide/PCB         |                |
| Boring #12                                                                          |                       |                |
|  |                       | DOW: _____     |
| DV32OØ512A92KØ5Ø1BXX                                                                |                       | Sampler: _____ |

### Figure 5-2: Example of Chain-of-Custody Record

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## 5.0 Sample Custody

Prior to collection of a particular sample, all background information associated the samples including the SLI, sample media, date, and required analyses, is entered into the database through a menu-driven computer program to produce sample labels for all analyte bottles associated with the sample. Included on each label are the SLI, USC, required preservation, relative sample time, and space for the collector's initials. Information such as the SLI and USC are also printed on the label as a bar code. As the background information for each sample is entered into the software for sample label production, the data are simultaneously and automatically stored in a database used for sample tracking and chain-of-custody production. The bar coding process increases:

- The accuracy of all sample documentation procedures by eliminating the potential for transcription errors.
- The efficiency of technical personnel by reducing the amount of time needed to complete the required documentation.
- The efficiency of the database management operations and the accuracy of critical field data by having the majority of the field data entered into a database in an IRDMIS-compatible format during sampling activities.

Once sample labels are produced and affixed to the appropriate sample bottles, they are grouped by sample location and distributed to the appropriate field teams for sample collection. Duplicate copies of each sample label are affixed in a dedicated sample notebook for future reference and as hardcopy documentation of field sampling activities. As the sample bottles return from the field, the bar code on each sample is scanned and chain-of-custody documents consistent with those required by USAEC are automatically produced.

The specific sample codes for FGGM are provided in Tables 4-4 through 4-10 of the Work Plan.

## 5.2 Laboratory Custody Procedures

The laboratory chain-of-custody of the samples begins with sample receipt and continues through final disposition of the field samples and other analytical samples (e.g., extracts) generated during analysis. The areas of concern for laboratory custody of samples include the following: sample receipt and log-in; internal chain-of-custody during analysis; sample lotting and labeling; sample splitting; storage of samples and sample extracts; and disposal.

A copy of applicable field chain-of-custody records will be maintained with each sample. In addition, each lot of samples will be maintained under separate laboratory chain-of-custody records that include: the unique laboratory sample identification

## 5.0 Sample Custody

number; date and time of collection, preparation and analysis; source of sample; analyses required; signatures of laboratory personnel relinquishing and receiving sample custody, and any other pertinent information.

For this project, custody of field samples will be relinquished to the subcontracted laboratory at the time of sample receipt and log-in. Specific procedures will be followed by the laboratory to ensure maintenance of an accurate written record that can be used to trace the possession and handling of a sample from the moment of its collection through its analysis and disposal and to ensure that the integrity of the sample is maintained throughout the analytical process. The laboratory selected for the analysis of FGGM samples is DataChem Laboratories, a USAEC-performance demonstrated laboratory has prepared SOPs for all aspects of sample custody during the analytical phase of the investigation; these SOPs conform to the requirements of the USAEC QA program. The laboratory custody procedures are summarized in the DataChem QA Program Plan for USAEC Laboratory Analysis provided in Appendix A of this Project QC Plan; the appropriate SOPs are listed in Appendix B.



## 6.0 Calibration Procedures and Frequency

This section presents information regarding the calibration of field and laboratory instrumentation to be used by Arthur D. Little and the subcontracted analytical laboratory during this project. All instruments and equipment used during sampling and analysis will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as the criteria set forth in the applicable field and laboratory procedures addressed in this section. Operation, calibration, and maintenance and calibration information will be maintained in an appropriate logbook or reference file for each instrument, and will be available upon request. If daily calibration cannot be achieved, the instrument will be scheduled for service and an alternate instrument will be used.

A description of the calibration procedures or reference to applicable SOPs is provided in the sections below. Calibration standards and frequency requirements are also summarized. Additional analytical method-specific calibration information is provided in the QA Program for the analytical laboratory (Appendix A) for USAEC-performance demonstrated analyses and within the analytical methods for the non-performance demonstrated analytes (Appendix D).

Two types of calibration are discussed in this section:

- Operational calibration, which is routinely performed as part of instrument usage, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operation calibration is generally performed for instrument systems.
- Periodic calibration, which is performed at prescribed intervals for equipment, such as balances and thermometers. In general, equipment that can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance.

### 6.1 Field Instrumentation

All field instrumentation will be maintained according to manufacturer's recommendations, including those regarding initial and routine calibration, as outlined in the appropriate operating manual. Maintenance and calibration procedures will be documented in the instrument logbook. In general, instruments will be calibrated at the start of each day of sampling and at the end of the day to check for instrument drift. All calibration data and calibration checks will be entered into the field notebook. Failure of an instrument to maintain accurate calibration will be reported to the site coordinator who will take immediate action to ensure that accurate field data are collected. The faulty instrument will be tagged and will not be used until it has been repaired or recalibrated.

## 6.0 Calibration Procedures and Frequency

Field measurements will be made for the following parameters: pH, temperature, conductivity, and turbidity. Total volatile organic emissions data will be collected in the field for health and safety purposes and for VOC contaminant screening purposes.

The instruments used to obtain field pH, temperature, conductivity, and turbidity measurements are factory calibrated and are routinely checked for accuracy against known standards; if necessary, recalibration will be performed. The specific procedures used to check the accuracy of these various field instruments are summarized below. SOPs for use and calibration of each of the field instruments are provided in Appendix B.

- pH: The accuracy of pH measurements obtained in the field is ensured by calibrating the pH meter against standard buffer solutions of known pH. The pH electrode is initially calibrated against a pH 7.0 buffer and then recalibrated at either pH 4.0 or 10.0 (depending on the anticipated range of sample pH). These procedures are performed at the beginning of each day of field sampling activities and at the end of the day to check for drift. The procedures for use and calibration of the pH meter are provided in SOP ADL-5013.
- Temperature: The accuracy of the field instrumentation used to obtain temperature data will be checked against a NIST thermometer at the beginning of each day of sampling and again at the end of the day to check for instrument drift.
- Conductivity: The accuracy of the conductivity meter will be checked daily during field sampling activities. A standard potassium chloride solution of known conductivity (0.1 N KCl) will be used; if necessary, recalibration of the instrument will be performed as indicated in SOP ADL-5011.
- Turbidity: The accuracy of the turbidity meter will be checked against a standard of known turbidity (0.02 NTU) before each reading in the field.

Data for total volatile organic emissions will be obtained in the field using a photoionization detector (PID). The procedures for use and calibration of the PID are provided in SOP ADL-5012. Calibration is verified prior to use in the field and at the beginning of each day of field sampling activities; calibration is verified at the end of the day to check for drift. Isobutylene is in air at a concentration of 25 to 100 ppm; calibration will be performed at ambient temperature and pressure.

An explosimeter will be used to determine percent oxygen for health and safety purposes and will be calibrated as follows:

- The instrument will be inspected and calibrated on a daily basis.
- The instrument will be inspected to ensure that entry and exit ports are clear.



## 6.0 Calibration Procedures and Frequency

- Turn the switch to the ON position. At this point the alarm will sound and the meter dials will jump.
- Allow the meters to stabilize and press the red RESET button. If the alarm continues, turn switch to HORN OFF position.
- Check the battery by depressing the black BATTERY button and note reading on the explosimeter display.
- Calibrate the oxygen meter to 20.8 percent by using the CALIBRATE knob.
- Zero the explosimeter to zero with the ZERO knob.
- If horn was turned off, return the switch to the ON position.
- Check alarm levels by adjusting the CALIBRATE knob for oxygen levels and the ZERO knob for explosimeter levels and note readings when alarm sounds. Return readings to normal and depress RESET button.

## 6.2 Laboratory Calibration

The laboratory analyses for samples collected during the investigations undertaken in this project will be performed by DataChem Laboratories. All analytical instruments and equipment used by DataChem are controlled by a formal calibration program. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with the specified requirements of the investigation. Calibration is performed internally by laboratory personnel using reference standards or externally by calibration agencies or equipment manufacturers.

This section prescribes the routine laboratory practices used to implement a calibration program. Development and documentation of the laboratory calibration program is the responsibility of the Laboratory Managers. Implementation is the responsibility of the supervisors and analysts; the Laboratory Quality Assurance Coordinator (QAC) monitors the procedures.

### 6.2.1 Laboratory Instrumentation Calibration

**6.2.1.1 Calibration Standards.** Two types of reference standards are used for calibration of laboratory instrumentation:

- Physical standards, such as weights for calibrating balances and performance demonstrated thermometers for calibrating working thermometers, refractors and ovens, which are generally used for periodic calibration.

## 6.0 Calibration Procedures and Frequency

- Chemical standards, such as Standard Reference Materials (SRMs) provided by the National Institute of Standards and Technology (NIST) or the EPA. These may include vendor-performance demonstrated materials traceable to NIST or EPA SRMs. These are primarily used for operational calibration.

Whenever possible, physical reference standards have known relationships to nationally recognized standards (e.g., NIST) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference is documented.

Physical reference standards are used only for calibration and are stored separately from equipment used in analyses. In general, physical reference standards are at least four to ten times as accurate as the requirements for the equipment which they are used to calibrate. In general, physical standards are recalibrated annually by a performance demonstrated external agency.

Whenever possible, chemical reference standards are directly traceable to NIST SRMs. If SRMs are not available, compounds of vendor-performance demonstrated high purity are used to prepare calibration standards.

**6.2.1.2 Calibration Frequency.** Instruments and equipment shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment. Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended data use, specified analytical methods, effect of error upon the measurement process, and prior experience.

Equipment that cannot be calibrated or becomes inoperable during use is removed from service and tagged to indicate it is out of calibration. Such equipment must be repaired and satisfactorily recalibrated before reuse. For equipment that fails calibration, Nonconformance Record (NCR) is used to record the corrective action and to demonstrate satisfactory calibration.

The following data-generating laboratory instrument requires *annual calibration*.

- Analytical Balance

The following data-generating laboratory instrument requires *semi-annual calibration*.

- UV-VIS Spectrophotometer



## 6.0 Calibration Procedures and Frequency

The following data-generating laboratory instruments require *calibration before each use*.

- a. The first group includes the instruments for which the calibration procedure is the establishment of a calibration curve.
  - UV-VIS Spectrophotometer (when used for relative analyses)
  - Technicon Autoanalyzer
  - Total Organic Carbon Analyzer
  - Atomic Absorption Spectrophotometer
  - IR Spectrophotometer
  - Selective Ion Meter
  - Inductively Coupled Plasma/Atomic Emission Spectrophotometer
- b. The second group includes instruments for which the calibration procedure is the measurement of standard response factors as described in the individual analytical methods. The documentation of the calibration is the record of standard concentrations and responses stored in the files of the standard runs.
  - Gas Chromatograph
  - Gas Chromatograph/Mass Spectrometer
- c. The third group includes instruments for which the calibration procedure consists of the measurement of one or two standards. From the standard measurements either the instrument is set to read the appropriate value or a calibration factor is calculated. The results of the standard measurements are recorded on the laboratory data sheets.
  - pH Meter
  - Selective Ion Meter (when used for pH measurements)
  - Conductivity Meter
  - Dissolved Oxygen Meter
  - Turbidimeter/Nephelometer

**6.2.1.3 Tuning and GC/MS Mass Calibration.** Prior to initiating any ongoing data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP) or p-bromofluorobenzene (BFB). The ion abundance criteria for each calibration compound *must* be met before any samples, blanks, or standards can be analyzed.

**6.2.1.4 Decafluorotriphenylphosphine (DFTPP):** Each GC/MS system used for the analysis of semivolatile or pesticide compounds must be hardware-tuned to meet the abundance criteria for a 50-ng injection of decafluorotriphenylphosphine (DFTPP). DFTPP may be analyzed separately or as part of the calibration standard. The criteria

## 6.0 Calibration Procedures and Frequency

must be demonstrated daily or for each 12-hour period, whichever is more frequent. DFTPP must be injected to meet this criterion. Post-acquisition manipulation of ion abundance is *not* acceptable. Documentation of the calibration is provided in the form of a mass listing (Table 6-1).

**6.2.1.5 *p*-Bromofluorobenzene (BFB).** Each GC/MS system used for the analysis of volatile compounds must be hardware-tuned to meet the abundance criteria for a maximum of a 50-ng injection of BFB. Alternately, 50 ng of BFB solution is added to 5.0 mL of reagent or standard solution and analyte. This criterion must be demonstrated daily or for each 12-hour period, whichever is more frequent. Post-acquisition manipulation of ion abundance is *not* acceptable. Documentation of the calibration is provided in the form of a mass listing (Table 6-2).

DFTPP and BFB criteria *must* be met before any samples, sample extracts, blanks, or standards are analyzed. Any samples analyzed when tuning criteria have not been met may require reanalysis at no cost to the client.

Definition: The 12-hour period for tuning and calibration criteria begins at the moment of injection of the DFTPP and BFB analysis that the laboratory submits as documentation of complaint tune. The period ends after 12 hours according to the system clock.

### 6.2.2 Operational Calibration

Operational calibration is generally performed as part of the analytical procedure. Included may be the analysis of a method blank and the preparation of a standard response (standard calibration) curve. Following is a brief discussion of the analysis of method blanks and preparation of standard curves.

**6.2.2.1 General Calibration Procedures.** The initial phase of a laboratory testing program requires the selection and performance demonstration of the method best suited for an individual parameter. Performance demonstration, or verification, is the elimination, or minimizing, of determinate errors that may be due to analyst error or the use of less- than-optimum equipment, reagents, solvents, or gases. The quality of materials, even though they are analytical reagent (AR) grade or better, may vary from one source to another. The analyst must determine, through the use of reagent and/or solvent blanks, if materials are free from interfering substances that could affect the analysis. Other steps in performance demonstrating the method include the determination of a method blank and the preparation of a standard calibration curve.

**6.2.2.2 Method Blank.** The analyst will prepare a method blank to evaluate background levels of contamination associated with sample preparation and analysis. The method blank will be prepared and analyzed in the same manner as field samples using all reagents used in processing the samples. In the USAEC program, a method blank must be used at a frequency of one per lot and is prepared using the standard water or soil matrix. The standard water matrix consists of Type I water for inorganic



## 6.0 Calibration Procedures and Frequency

Table 6-1: DFTPP Key Ions and Abundance Criteria

| Mass | Ion Abundance Criteria                    |
|------|-------------------------------------------|
| 51   | 30.0 - 60.0 percent of mass 198           |
| 68   | less than 2.0 percent of mass 69          |
| 70   | less than 2.0 percent of mass 69          |
| 127  | 40.0 - 60.0 percent of mass 198           |
| 197  | less than 1.0 percent of mass 198         |
| 198  | base peak, 100 percent relative abundance |
| 199  | 5.0 - 9.0 percent of mass 198             |
| 275  | 10.0 - 30.0 percent of mass 198           |
| 365  | greater than 1.0 percent of mass 198      |
| 441  | present but less than mass 443            |
| 442  | greater than 40.0 percent of mass 198     |
| 443  | 17.0 - 23.0 percent of mass 442           |

Note: Whenever the laboratory takes corrective action that may change or affect the tuning criteria for DFTPP (e.g., ion source cleaning or repair, etc.), the tune is verified irrespective of the 12-hour tuning requirements.

## 6.0 Calibration Procedures and Frequency

**Table 6-2: BFB Key Ions and Abundance Criteria**

| Mass | Ion Abundance Criteria                                            |
|------|-------------------------------------------------------------------|
| 50   | 15.0 - 40.0 percent of the base peak                              |
| 75   | 30.0 - 60.0 percent of the base peak                              |
| 95   | base peak, 100 percent relative abundance                         |
| 96   | 5.0 - 9.0 percent of the base peak                                |
| 173  | less than 2.0 percent of mass 174                                 |
| 174  | greater than 50.0 percent of the base peak                        |
| 175  | 5.0 - 9.0 percent of mass 174                                     |
| 176  | greater than 95.0 percent but less than 101.0 percent of mass 174 |
| 177  | 5.0 - 9.0 percent of mass 176                                     |

Note: Whenever the laboratory takes corrective action that may change or affect the tuning criteria for BFB (e.g., ion source cleaning or repair, etc.), the tune must be verified irrespective of the 12-hour tuning requirements.



## 6.0 Calibration Procedures and Frequency

analyses and Type II water containing 100 mg/L of chloride and sulfate for organic analyses. The standard soil matrix is provided to the laboratory by USAEC.

**6.2.2.3 Calibration Curve.** For all "relative" analyses, a calibration or standard curve is required to calculate sample concentrations from the measured instrument responses. A calibration curve is prepared by measuring the instrument responses for a series of standard solutions of the analyte. The sample concentrations are then calculated by comparison to the standard points. One way to perform these calculations is to use regression analysis to fit a curve through the standard data. The sample concentrations can then be calculated using the resulting regression equation. The regression analysis also provides parameters that can be used to assess the condition of the analysis. The majority of analyses in the laboratory give linear calibration curves or can be transformed to a linear form. Other analyses can be fitted to a parabolic curve.

### 6.2.3 Calibration for USAEC-Performance Demonstrated Methods

The USAEC QA Program delineates, in detail, the requirements for instrument calibration, initial calibration for analysis, and daily calibration during sample analysis. DataChem Laboratories has implemented the USAEC specifications for all performance demonstrated methods. The specific calibration procedures for USAEC-performance demonstrated methods are summarized in the DataChem QA Program Plan provided in Appendix A.

Table 6-1 summarizes the general instrumental systems controls associated with the USAEC calibration program. The concentration range of the calibration standards brackets the performance demonstrated range of the method. For the minimum testing range (MTR), initial calibration for Class 1 methods includes a minimum of one blank and five levels of calibration standards plus the check standard; for Class 1A methods, initial calibration includes a minimum of one blank and three levels of calibration standards. When order-of-magnitude extensions are performed, additional high level standards are required.

Initial calibration procedures are performed in the following events:

- The first day that performance demonstrated analyses are performed.
- The instrument is started up (other than daily start-up and shut-down).
- The instrument is used to analyze analytes different from those for which the instrument was previously calibrated.
- The instrument fails daily calibration.

Daily calibration procedures are performed each day of instrumental analysis to verify that the instrument response has not changed from the previous calibration.

## 6.0 Calibration Procedures and Frequency

Calibration and spiking standards are prepared from Standard Analytical Reference Materials (SARMs) or interim SARMs obtained from the USAEC Repository Program, whenever possible. Materials purchased from outside vendors are classified as "off-the-shelf" and used only when SARMs are not available. Off-the-shelf materials are characterized against NIST or EPA standards for purity and identification. Standards characterization data are kept on file at the laboratory. Chain-of-custody procedures are maintained for all standard reference materials. Materials are stored in locked areas at ambient temperature or below 4°C for inorganics and organics, respectively.



## 7.0 Analytical Procedures

### 7.1 Analytical Program

The chemical analysis program for this FGGM project is directed towards generating data from field and laboratory tests that will define contamination profiles at the FGGM site and facilitate the development and evaluation of remedial action alternatives. For this task, a specific set of analytes for laboratory analysis has been specified for each sample collected from the site. The chemical analysis program has been designed to obtain quantitative data on the presence of these selected chemicals at detection limits consistent with USAEC target reporting limits and federal and state regulations. In addition to measuring the concentration of specific analytes, all tentatively identified organic compounds (TIC) detected during the GC/MS analyses with an area of greater than 10 percent of the internal standard will be library searched. This technique lends some assurance that major organic species that may be present in the FGGM samples will be detected and reported. As an indicator of a broader spectrum of oil-related contamination, total petroleum hydrocarbons (TPHC) will be measured at selected locations. This technique indicates the presence of contamination from a variety of oils and/or fuels that may have been used at FGGM. Tests for total volatile organic emissions will also be conducted in the field to provide "real time" information about ground water well development and the presence of broad indicators of contamination, in soil, water, and air (headspaces and/or soil gases).

Our subcontracted laboratory for this task, DataChem Laboratories, is committed to providing services to this contract as a high priority. In addition, we have identified a backup laboratory to provide added assurance that there will be excess capacity to carry out the required chemical analyses within the specified holding times and project schedules. DataChem Laboratories provides qualifications and commitment within the USAEC program, with over 40 USAEC-performance demonstrated methods and performance on USAEC contracts since 1981.

Table 7-1 lists the analyses to be performed on the samples collected during the FGGM investigations within this project. Table 7-2 provides a complete list of analytes. For each of the analyses, the reference analytical method is provided. Most of the analyses cited in Table 7-1 will be performed using USAEC-performance demonstrated methods. The referenced USAEC-performance demonstrated methods are unique to DataChem Laboratories and all USAEC-performance demonstrated analyses will be conducted according to the requirements of the specific method, without deviation. For the TCLP organics and inorganics, an EPA-approved method will be used. The TCLP analyses will be performed on investigation-derived waste samples and are not part of the site characterization data base. The Total Petroleum Hydrocarbons (TPHC) method is a USAEC non-performance demonstrated method based on EPA and ASTM methods.

Details of the USAEC analyses, including the CRL for each analyte, are provided within the DataChem QA Program Plan provided in Appendix A to this Project QC Plan. A copy of the complete USAEC-performance demonstrated DataChem method



## 7.0 Analytical Procedures

**Table 7-1: Summary of Analytical Methods for Site Characterization at FGGM**

| <b>Analysis</b>                                     | <b>Method Type</b> | <b>USAEC-Performance Demonstrated Method Number*</b> |
|-----------------------------------------------------|--------------------|------------------------------------------------------|
| TCL Volatiles - water                               | Class 1A           | UM21                                                 |
| TCL Volatiles - soil/sediment                       | Class 1A           | LM23                                                 |
| TCL Semivolatiles - water                           | Class 1A           | UM25                                                 |
| TCL Semivolatiles - soil/sediment                   | Class 1A           | LM25                                                 |
| PCBs - soil/sediment                                | Class 1            | LH17                                                 |
| Explosives - soil/sediment                          | Class 1            | LW23                                                 |
| Explosives - water                                  |                    |                                                      |
| TAL Metals (ICP) - water                            | Class 1            | SS12                                                 |
| TAL Metals (ICP) - soil/sediment                    | Class 1            | JS12                                                 |
| Cyanide - water                                     | Class 1            | TF34                                                 |
| Cyanide - soil/sediment                             | Class 1            | KF15                                                 |
| Mercury - water                                     | Class 1            | CC8                                                  |
| Mercury - soil/sediment                             | Class 1            | Y9                                                   |
| Chloride/Sulfate - water                            | Class 1            | TT09                                                 |
| Chloride/Sulfate - soil/sediment                    | Class 1            | KT07                                                 |
| Nitrate - water                                     | Class 1            | LL8                                                  |
| Nitrate - soil/sediment                             | Class 1            | KF17                                                 |
| Total Petroleum Hydrocarbons (TPHC) - soil/sediment | GC/FID **          | Non-performance demonstrated                         |
| Water                                               |                    |                                                      |
| Total Dissolved Solids (TDS) - water                | Gravimetric **     | Non-performance demonstrated                         |

\* USAEC-performance demonstrated method numbers are unique to DataChem Laboratories. Analyte CRLs for USAEC-performance demonstrated methods are on file at Arthur D. Little.

\*\* Non-performance demonstrated analytical methods are provided in Appendix D.



## 7.0 Analytical Procedures

Table 7-2: Summary of Specific Constituents in Multi-Analyte Methods

### USAEC VOLATILE ORGANIC COMPOUNDS

| USAEC ANALYTE                         | ANALYTE CODE |
|---------------------------------------|--------------|
| 1,1,1-TRICHLOROETHANE                 | 111TCE       |
| 1,1,2-TRICHLOROETHANE                 | 112TCE       |
| 1,1-DICHLOROETHENE                    | 11DCE        |
| 1,1-DICHLOROETHANE                    | 11DCLE       |
| * 1,2-DICHLOROETHENES (CIS AND TRANS) | 12DCE        |
| 1,2-DICHLOROETHANE                    | 12DCLE       |
| 1,2-DICHLOROPROPANE                   | 12DCLP       |
| 1,3-DICHLOROPROPENE                   | 13DCPE       |
| 2-CHLOROETHYL VINYL ETHER             | 2CLEVE       |
| ACETONE                               | ACET         |
| BROMODICHLOROMETHANE                  | BRDCLM       |
| CIS-1,3-DICHLOROPROPENE               | C13DCP       |
| VINYL ACETATE                         | C2AVE        |
| VINYL CHLORIDE                        | C2H3CL       |
| CHLOROETHANE                          | C2H5CL       |
| BENZENE                               | C6H6         |
| CARBONTETRACHLORIDE                   | CCL4         |
| METHYLENECHLORIDE                     | CH2CL2       |
| BROMOMETHANE                          | CH3BR        |
| CHLOROMETHANE                         | CH3CL        |
| BROMOFORM                             | CHBR3        |
| CHLOROFORM                            | CHCL3        |
| DICHLOROMETHANE                       | CL2CH2       |
| CHLOROBENZENE                         | CLC6H5       |
| CARBONDISULFIDE                       | CS2          |
| DIBROMOCHLOROMETHANE                  | DBRCLM       |
| ETHYLBENZENE                          | ETC6H5       |
| TOLUENE                               | MEC6H5       |
| METHYLETHYLKETONE                     | MEK          |
| METHYLISOBUTYLKETONE                  | MIBK         |
| STYRENE                               | STYR         |
| TRANS-1,2-DICHLOROETHENE              | T12DCE       |
| TRANS-1,3-DICHLOROPROPENE             | T13DCP       |
| 1,1,2,2-TETRACHLOROETHANE             | TCLEA        |
| TETRACHLOROETHENE                     | TCLEE        |
| TRICHLOROETHENE                       | TRCLE        |
| XYLENES, TOTAL                        | TXYLEN       |
| TRICHLOROFLUOROMETHANE                | TCFM         |
| DICHLORODIFLUOROMETHANE               | DCDFM        |

## 7.0 Analytical Procedures

**Table 7-2: Summary of Specific Constituents in Multi-Analyte Methods  
(continued)**

### USAEC SEMIVOLATILE ORGANIC COMPOUNDS

| USAEC ANALYTE                                   | ANALYTE CODE |
|-------------------------------------------------|--------------|
| 1,2,4-TRICHLOROBENZENE                          | 124TCB       |
| 1,2-DICHLOROBENZENE                             | 12DCLB       |
| 1,3-DICHLOROBENZENE                             | 13DCLB       |
| 1,4-DICHLOROBENZENE                             | 14DCLB       |
| 2,4,5-TRICHLOROPHENOL                           | 245TCP       |
| 2,4,6-TRICHLOROPHENOL                           | 246TCP       |
| 2,4-DICHLOROPHENOL                              | 24DCLP       |
| 2,4-DIMETHYLPHENOL                              | 24DMPN       |
| 2,4-DINITROPHENOL                               | 24DNP        |
| 2,4-DINITROTOLUENE                              | 24DNT        |
| 2,6-DINITROTOLUENE                              | 26DNT        |
| 2-CHLOROPHENOL                                  | 2CLP         |
| 2-CHLORONAPHTHALENE                             | 2CNAP        |
| 2-METHYLNAPHTHALENE                             | 2MNAP        |
| 2-METHYLPHENOL / 2-CRESOL                       | 2MP          |
| 2-NITROANILINE                                  | 2NANIL       |
| 2-NITROPHENOL                                   | 2NP          |
| 3,3'-DICHLOROBENZIDINE                          | 33DCBD       |
| 3,4-Dinitrotoluene                              | 34DNT        |
| 3-NITROANILINE                                  | 3NANIL       |
| 3-NITROTOLUENE                                  | 3NT          |
| 4,6-DINITRO-2-CRESOL / METHYL-4,6-DINITROPHENOL | 46DN2C       |
| 4-BROMOPHENYLPHENYL ETHER                       | 4BRPPE       |
| 4-CHLOROANILINE                                 | 4CANIL       |
| 4-CHLORO-3-CRESOL / 3-METHYL-4-CHLOROPHENOL     | 4CL3C        |
| 4-CHLOROPHENYLPHENYL ETHER                      | 4CLPPE       |
| 4-METHYLPHENOL / 4-CRESOL                       | 4MP          |
| 4-NITROANILINE                                  | 4NANIL       |
| 4-NITROPHENOL                                   | 4NP          |
| ACENAPHTHENE                                    | ANAPNE       |
| ACENAPHTHYLENE                                  | ANAPYL       |
| ANTHRACENE                                      | ANTRC        |
| BIS (2-CHLOROETHOXY) METHANE                    | B2CEXM       |
| BIS (2-CHLOROISOPROPYL) ETHER                   | B2CIPE       |
| BIS (2-CHLOROETHYL) ETHER                       | B2CLEE       |
| BIS (2-ETHYLHEXYL) PHTHALATE                    | B2EHP        |
| BENZO [A] ANTHRACENE                            | BAANTR       |
| BENZO [A] PYRENE                                | BAPYR        |
| BENZO [B] FLUORANTHENE                          | BBFANT       |
| BUTYLBENZYL PHTHALATE                           | BBZP         |
| BENZOIC ACID                                    | BENZOA       |
| BENZO [G,H,I] PERYLENE                          | BGHIPY       |
| BENZO [K] FLUORANTHENE                          | BKFANT       |



## 7.0 Analytical Procedures

**Table 7-2: Summary of Specific Constituents In Multi-Analyte Methods  
(continued)**

### USAEC SEMIVOLATILE ORGANIC COMPOUNDS

| USAEC ANALYTE              | ANALYTE CODE |
|----------------------------|--------------|
| BENZYL ALCOHOL             | BZALC        |
| CHRYSENE                   | CHRY         |
| HEXACHLOROBENZENE          | CL6BZ        |
| HEXACHLOROCYCLOPENTADIENE  | CL6CP        |
| HEXACHLOROETHANE           | CL6ET        |
| DIBENZ [A,H] ANTHRACENE    | DBAHA        |
| DIBENZOFURAN               | DBZFUR       |
| DIETHYL PHTHALATE          | DEP          |
| DIMETHYL PHTHALATE         | DMP          |
| DI-N-BUTYL PHTHALATE       | DNBP         |
| DI-N-OCTYL PHTHALATE       | DNOP         |
| FLUORANTHENE               | FANT         |
| FLUORENE                   | FLRENE       |
| HEXACHLOROBUTADIENE        | HCBD         |
| INDENO [1,2,3-C,D] PYRENE  | ICDPYR       |
| ISOPROPYLAMINE             | IPA          |
| ISOPHORONE                 | ISOPHR       |
| NAPHTHALENE                | NAP          |
| NITROBENZENE               | NB           |
| NITROSO DI-N-PROPYLAMINE   | NDNPA        |
| N-NITROSO DI-N-PROPYLAMINE | NNDNPA       |
| N-NITROSO DIPHENYLAMINE    | NNDPA        |
| PENTACHLOROPHENOL          | PCP          |
| PHENANTHRENE               | PHANTR       |
| PHENOL                     | PHENOL       |
| PYRENE                     | PYR          |

## 7.0 Analytical Procedures

**Table 7-2: Summary of Specific Constituents in Multi-Analyte Methods  
(continued)**

### USAEC METALS COMPOUNDS

| USAEC ANALYTE | ANALYTE CODE |
|---------------|--------------|
| SILVER        | AG           |
| ALUMINUM      | AL           |
| ARSENIC       | AS           |
| BARIUM        | BA           |
| BERYLLIUM     | BE           |
| CALCIUM       | CA           |
| CADMIUM       | CD           |
| COBALT        | CO           |
| CHROMIUM      | CR           |
| COPPER        | CU           |
| CYANIDE       | CYN          |
| IRON          | FE           |
| MERCURY       | HG           |
| POTASSIUM     | K            |
| MAGNESIUM     | MG           |
| MANGANESE     | MN           |
| SODIUM        | NA           |
| NICKEL        | NI           |
| LEAD          | PB           |
| ANTIMONY      | SB           |
| SELENIUM      | SE           |
| THALLIUM      | TL           |
| VANADIUM      | V            |
| ZINC          | ZN           |

### USAEC EXPLOSIVES

| USAEC ANALYTE                                  | ANALYTE CODE |
|------------------------------------------------|--------------|
| 1,3,5-TRINITROBENZENE                          | 135TNB       |
| 1,3-DINITROBENZENE                             | 13DNB        |
| 2,4,6-TRINITROTOLUENE                          | 246TNT       |
| 2,4-DINITROTOLUENE                             | 24DNT        |
| 2,6-DINITROTOLUENE                             | 26DNT        |
| 3,4-Dinitrotoluene                             | 34DNT        |
| CYCLOTETRAMETHYLENETETRANITRAMINE              | HMX          |
| NITROBENZENE                                   | NB           |
| NITROCELLULOSE                                 | NC           |
| NITROGLYCERINE                                 | NG           |
| PENTAERYTHRITOL TETRANITRATE                   | PETN         |
| CYCLOTRIMETHYLENETRINITRAMINE / CYCLONITE      | RDX          |
| N-METHYL-N,2,4,6-TETRANITROANILINE / NITRAMINE | TETRYL       |



## 7.0 Analytical Procedures

**Table 7-2: Summary of Specific Constituents In Multi-Analyte Methods  
(continued)**

### USAEC POLYCHLORINATED BIPHENYL COMPOUNDS

| USAEC ANALYTE | ANALYTE CODE |
|---------------|--------------|
| PCB 1016      | PCB016       |
| PCB 1221      | PCB221       |
| PCB 1232      | PCB232       |
| PCB 1242      | PCB242       |
| PCB 1248      | PCB248       |
| PCB 1254      | PCB254       |
| PCB 1260      | PCB260       |

## **7.0 Analytical Procedures**

for each of these analyses will be maintained in the Arthur D. Little files for this project. The method performance demonstration procedures and analyte performance demonstration procedures for the USAEC-performance demonstrated analyses are summarized in Sections 7.2 and 7.3, respectively. Brief summaries of the analytical methods to be used to generate site characterization data are provided in Section 7.4.

### **7.2 Laboratory Method Performance Demonstration**

In order to provide a common point of reference for all projects and to provide a means of evaluating laboratory performance, USAEC prescribes the use of standardized methods for commonly encountered analytes. These methods are sufficiently general to be used in almost any laboratory, yet specify all critical elements. The standardized methods are based on published methods of analysis, USAEC standing methods, or past USAEC experience (e.g., for military unique compounds). Methods have been evaluated in terms of sound analytical practice and applicability to environmental projects. In addition to specifying sample preparation and analysis, each method also specifies calibration procedures and frequency, calibration check acceptance criteria, methods of preparing standard solutions, and preparation of QC samples.

Four different types of analyses are recognized by the USAEC QA Program: Class 1, 1A, 1B, and Class 2; for this project, Class 1 and 1A analyses will be performed, as indicated in Table 7-1. The difference between the classes is the procedure used to characterize laboratory performance of the method. Class 1A performance demonstration is reserved exclusively for GC/MS methods; whereas Class 1B are reserved for low sample-throughput methods (i.e., non-GC/MS). Class 2 performance demonstration is used for methods that screen for the presence or absence of contaminants. Each type of analysis requires a different level of documentation, including precision and accuracy data, and a different set of daily or batch-related QC criteria.

#### **7.2.1 Laboratory Methods Requiring Performance Demonstration**

The Class 1 USAEC-performance demonstrated methods being used for the FGGM project are:

- Metals
- Explosives
- Nitrate
- Sulfate
- PCBs
- Chloride



## 7.0 Analytical Procedures

The Class 1A (GC/MS) USAEC-performance demonstrated methods being used for the FGGM project are:

- Volatile organics
- Semivolatile (acid/base/neutral) organics

### 7.2.2 Methods Not Requiring Performance Demonstration

Some methods, including calibration of test and measurement equipment, do not require performance demonstration, due to either the nature of the measurement or the intended use of the data. When such methods are part of a project, USAEC will not provide a standardized method. However, laboratories must submit sufficient information in test plans, work plans, project QC plans, etc., to describe the procedures to be used. A copy of the methods must be submitted to the USAEC Chemistry Branch before it is used on any project.

The non-performance demonstrated methods to be used for analysis of site characterization samples are for TDS (Total Dissolved Solids) and TPHC (Total Petroleum Hydrocarbons). Copies of the proposed analytical methods for these analyses are provided in Appendix D of this Project QC plan. Methods for analysis of hazardous waste characteristics, i.e., TCLP organics and inorganics, would also be non-performance demonstrated methods. However, these analyses are not part of site characterization and apply only to disposal of investigation-derived waste.

## 7.3 Analyst Qualification

It is the responsibility of the organization to establish personnel qualifications and training requirements for all positions. Each member of the FGGM analytical team will have the education, training, technical knowledge, and experience, or a combination thereof, to enable that individual to perform their assigned functions. Personnel qualifications are documented in terms of education, experience, and training. Training is provided for each staff member to properly perform their functions.

Copies of the performance demonstrated methods will be maintained by the laboratory QA staff and the Arthur D. Little Lead Chemist. Analysts will demonstrate their proficiency in conducting a particular chemical analysis by showing evidence of acceptable performance on past routine QC samples analyzed with each batch of samples. New analysts performing an established analytical procedure will be considered conditionally qualified until the first set of QA/QC data is generated. These QC data are required for every lot of samples analyzed. If these QC data are in control based on control charts, the analyst or analytical team will be considered qualified to run that particular analysis. QC data that do not meet established QC requirements will be rejected, and corrective action, which may include re-analysis of the lot of samples and further training of the analytical team, will be taken.

## **7.0 Analytical Procedures**

The analysts and other subcontracted lab support personnel are responsible for adherence to the QA Program Plan and to the requirements of the USAEC program.

### **7.4 Analytical Methods**

This section provides a brief summary of the USAEC-performance demonstrated analytical methods, as well as non-performance demonstrated methods, for the analysis of samples for this project.

#### **7.4.1 Nitrate, Sulfate, and Chloride**

For these analyses, a small volume of sample, typically 2 to 3 milliliters, is introduced into an ion chromatograph (IC). The anions of interest are separated and measured using a system comprised of a guard column, separator column, suppressed column, and conductivity detector.

#### **7.4.2 Volatile Organics (GC/MS)**

The method for volatile organics is based on USEPA Method 8240 and is used to determine volatile organic compounds in a variety of matrices. An inert gas is bubbled through a 5-milliliter water sample or 5-gram soil sample contained in a specially designed purging chamber at ambient temperature. The purgeable organics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped.

After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph (GC) is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

#### **7.4.3 Semivolatile (Acid/Base/Neutral) Organics (GC/MS)**

The method for semivolatiles is based on USEPA Method 8270 to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. For the analysis, a measured volume of sample, approximately 1 liter for aqueous samples or 30 grams for soil/sediment samples, is extracted with methylene chloride. The methylene chloride extract is dried, concentrated to a volume of 1 milliliter, and analyzed by GC/MS.

#### **7.4.4 PCBs (GC/ECD)**

The method for PCBs is based on USEPA Method 8080. For the analysis, a measured volume of sample, approximately one liter for aqueous samples and 10 grams for soil/sediment samples, is extracted with methylene chloride. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 milliliters for less. The extract is separated by GC and the parameters are then measured with an electron capture detector. The method provides a Florisil column



## 7.0 Analytical Procedures

cleanup procedure and an elemental sulfur removal procedure to aid in the elimination of interferences that may be encountered.

### 7.4.5 Metals

**7.4.5.1 Inductively Coupled Plasma Atomic Emission Spectroscopy (ICAP).** For analysis, samples are solubilized or digested using a method based on USEPA Method 3010 for water and Method 3050 for soils. These methods are from "Test Methods for Evaluating Solid Waste", SW-846, third edition, USEPA, September 1986. The analysis procedure follows USEPA Method 6010 for multi-elemental determination of elements by ICAP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes.

**7.4.5.2 Cold Vapor (Mercury).** The method for mercury analysis is based on USEPA Methods 7470 and 7471. Mercury-containing compounds from solid or aqueous samples are digested under acid conditions in the presence of heat and strong oxidant. Following digestion, mercury is reduced to its elemental state and aerated from solution in a cold vapor adsorption cell of fixed path length. The absorption cell is positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured (peak height) as a function of concentration at 253.7 nm. A calibration curve is constructed by plotting peak height concentration of known standards using a second order regression. The instrumental concentration is determined and the final sample concentration is calculated, accounting for any dilution or concentration process utilized and the initial volume of sample used for the analysis.

**7.4.5.3 Graphite Furnace Atomic Absorption.** USEPA reference methods for these analyses are Methods 7060 (arsenic), 7740 (selenium), 7421 (lead), 7841 (thallium), 3020 (water digestion), and 3050 (soil digestion).

Metallic constituents from solid or aqueous samples are made soluble through sample reflux digestion under acid conditions. Sample digestates are introduced into a temperature-programmed graphite furnace atomic absorption spectrophotometer (GFAA) that has been calibrated in accordance with specification. The sample is dried, charred, and atomized. The metal atoms are placed in a beam of radiation by increasing the temperature, causing the specimen to volatilize. Characteristic radiation from a hollow cathode lamp is absorbed and the attenuated transmitted radiation is measured. Quantification of the analyte of interest in the digestate is based on a standard curve of absorption response versus known concentration using linear regression. The instrumental concentration is determined and the final sample concentration is calculated, accounting for any dilution or concentration process utilized.



## **7.0 Analytical Procedures**

### **7.4.6 Explosives**

This method is based on USAEC Method CERTNF for aqueous samples and CERTNF for soils by HPLC.

For aqueous samples, the method employs solid phase extraction of 500 milliliters of an environmental water using a tube packed with Porapak R. The target analytes are desorbed with 3 milliliters of acetonitrile and the extract is diluted to a final volume of 10 milliliters with water. The analytes are separated by HPLC using isocratic elution and detected using ultraviolet absorbance (uv) at 250 nm.

For soil samples, the method employs extraction of one gram of an environmental soil using two milliliters of acetonitrile. Extraction is accomplished by vortexing followed by sonication of the sample for two hours. The resulting extract is filtered and diluted one to eight with water. The target analytes are separated on a HPLC column using isocratic elution and detected using UV at 230 nm.

### **7.4.7 TDS (Total Dissolved Solids)**

The method that will be used for this analysis is EPA 160.1. Dissolved solids, also known as filtrable residue, is material that is passed through a standard glass fiber filter disk and remains after evaporation and drying to constant weight at 180°C. An aliquot of a 100 ml, or more, of well mixed sample is filtered through a glass fiber filter done under vacuum. The filtered aqueous sample is then transferred to a weighted evaporating dish, and evaporated, then dried in an oven for at least an hour at 180° and weighed. The result is calculated by subtracting the weight of the dish from the weight of dried residue then dividing it by the volume of filtrate used.

### **7.4.8 TPHC (Total Petroleum Hydrocarbons by Gas Chromatography/Flame Ionization Detector)**

This method is designed to identify and quantify petroleum products contaminants and can be applied to water, soil, and waste samples. The sample is extracted in methylene chloride, and the extract is concentrated and then analyzed by capillary column gas chromatography with flame ionization detection. A result is reported for the total petroleum hydrocarbons detected, and a qualitative identification of the contaminants is made. This method is based in part on EPA Methods 8015, and ASTM D3328-78. It is intended to provide higher resolution than these methods and to provide a greater amount of qualitative information. The technique has been applied to a wide variety of environmental investigations.

## **7.5 Field Analytical Methods**

The non-performance demonstrated methods for the analyses to be performed in the field are conductivity, pH, temperature and turbidity. Conductivity is measured by using a self-contained conductivity meter. It measures the ability of a water sample to carry an electric current as specified in SOP ADL-5011 and in accordance with



## 7.0 Analytical Procedures

method EPA 120.1. For this project, a single instrument will provide the pH and temperature measurements as specified in SOP ADL-5013 in accordance with methods EPA 150.1 and EPA 170.1, respectively. The pH is determined electrometrically using a gas electrode in combination with a reference potential. In addition, this instrument will measure the temperature with a thermometer that is incorporated in the probe. Finally, turbidity analysis is the comparison of the intensity of light scattered by a standard reference suspension under the same conditions. For this project, a portable turbidimeter will be used as specified in SOP ADL-5026 and in accordance with method EPA 180.1.

## **8.0 Data Reduction, Validation, and Reporting**

### **8.1 Arthur D. Little's Data Management**

Data management for this project refers to the effective management of all project related information; map, geotechnical and chemical data. Arthur D. Little's and the subcontracted laboratory's data management systems will be integrated in order to achieve an efficient flow of information from the laboratory to Arthur D. Little to USAEC.

#### **8.1.1 Flow of Map Data Into IRDMIS**

IRDMIS map data entry refers to registering sampling locations by a specific convention and a coordinate system using a USAEC software program called PC IRDMIS or PC TOOL. Arthur D. Little will acquire the latest FGGM map data base from Patomac Research, Inc. (PRI) and will send this map database to the subcontracted laboratory so that proper record and group checks will be possible. Arthur D. Little will also be responsible for providing both the subcontracted laboratory and USAEC with updated map files based on sampling efforts at FGGM. When a new site is being sampled, Arthur D. Little will enter the map data to ensure proper processing of the associated analytical data.

#### **8.1.2 Flow of Geotechnical Data Into IRDMIS**

Arthur D. Little will provide USAEC with updated geotechnical files based on sampling efforts at FGGM. The geotechnical data from new well sites will be processed and entered into IRDMIS by Arthur D. Little. These data will be transferred into an ASCII-based "transfer" file, which will be sent to PRI for processing, validation, and loading to the USAEC legal repository known as Level III.

#### **8.1.3 Flow of Chemical Data Into the IRDMIS**

Arthur D. Little will be responsible for the final review of 10 percent of the analytical data associated with the sampling efforts at FGGM. This review is in addition, but identical to, the checks that are to be performed by the Arthur D. Little subcontracted laboratory. After the laboratory has analyzed FGGM field samples and created the IRDMIS transfer file, data will be submitted to PRI for eventual Level III status. This transfer will be confirmed as indicated by the USAEC weekly status report for each lot. Arthur D. Little's internal tracking system will also ensure that all field samples have had the proper analysis performed and will contact the laboratory and the USAEC Project Officer whenever and wherever discrepancies arise.

### **8.2 Data Reduction**

All the processes that change either the form of expression or quantity of data values or numbers of data items are part of the data reduction process.

Raw data from quantitative analysis procedures such as Gas Chromatography (GC), Gas Chromatography/Mass Spectrometry (GC/MS), High Performance Liquid



## 8.0 Data Reduction, Validation, and Reporting

Chromatography (HPLC), Inductively Coupled Argon Plasma (ICAP) and Ion Chromatography (IC) generally consist of peak areas (or peak heights) for the analytes of concern, internal standards, and surrogates. This applies to Class 1, 1A and TPH/GC-FID (a non-USAEC-performance demonstrated method). These raw data will be converted to concentrations by use of calibration curves or relative response factors that relate peak area to the quantity of analyte introduced in the instrument. For field methods, the calibration procedures are generally less rigorous than those for Class 1 and 1A.

Generally, data will be collected during the analysis of samples either into computer based data files or onto hard copy sheets, which, in turn, are either machine generated or hand written. In reporting results, rounding to the correct number of significant figures (this varies with the method) will occur only after all calculations and manipulations are completed. For dilutions, the number of significant figures will be reduced by one. Each analytical method referenced in Table 7-1 will describe the data reduction procedures for laboratory analysis results. In addition, it will describe the correct procedure for using method blank results.

All uncorrected values less than the certified (performance demonstrated) reporting limit, including no response, will be reported as "less than" the reporting limit. Results of the analyses will be entered into the USAEC IRDMIS as outlined in the IR Data Management User's Guide (USATHAMA, September 1992). Non-performance demonstrated analytes will be reported using detection limits documented in the appropriate method and will be flagged for data entry into the IRDMIS Non THAMA Approved Methods (NTAM) database.

### 8.3 Data Validation

Data validation is an integral part of this QA program. Data validation will be performed on one hundred percent of all data packages by the DataChem QA Coordinator. Even though the primary responsibility for this review and validation rests with the laboratory performing the analyses, Arthur D. Little will be responsible for reviewing 10 percent of the data, following USAEC guidelines for data review which are the same procedures followed by Datachem. See Section 8.1 - Arthur D. Little's Data Management.

The following is a brief outline of the data review and validation process:

- Evaluate for completeness of laboratory data.
- Evaluate data with respect to reporting limits.
- Evaluate data with respect to control limits.

## 8.0 Data Reduction, Validation, and Reporting

- Review holding time data.
- Correlate laboratory data from related laboratory tests.
- Examine chain-of-custody records to ensure that custody was properly maintained.
- Compare data on instrument print-outs with data recorded on worksheets or in notebooks.
- Check to ensure that the same calibration was used for all samples within a lot.
- Examine chromatographic outputs and documentation of the reasons for manual integrations.
- Compare standard and sample preparation and injection records with instrument output to ensure that each output is associated with the correct sample.
- Examine calibration and tuning results to ensure that requirements are met.
- Check calculations on selected samples to ensure correctness.
- Check that GC/MS library searches have been performed for all unknowns, as required, and that the results have been evaluated and recorded.
- Examine all papers and notebooks to ensure that all pages are initialed, dated, and have sufficient explanation for the changes, and that all items are legible.
- Compare transfer file, record and group check results with analysis results.

### 8.4 Data Validation Procedures

The data processed through the DataChem Data Management System, where automated QC checks are performed, are reviewed by the analyst supervisor and analytical task manager. The data package containing the computerized reports and all raw data are completed and submitted with the data package to the QA supervisor. See Appendix C for checklist used in the data package review.

The project QA Coordinator or assistant, is responsible for reviewing and approving all data packets before submittal of data to Arthur D. Little. Data validation involves a thorough review of all data documentation from the raw data to the reported results contained in the lot folders. Data are considered complete only after they are approved by the QA staff prior to submittal. The reviews are performed on every batch to ensure that all QC checks required by the method are included in the batch.



## 8.0 Data Reduction, Validation, and Reporting

With the use of the USAEC Data Review Checklist (see Appendix C), a through-package audit is performed. This includes checking the control charts, method blanks, standard matrix and sample matrix spike recoveries, surrogate recoveries, calibration curves, certified (performance demonstrated) reporting limits, and units. The lab QA Coordinator or assistant makes an initial judgment on the acceptability of method blank and other data. Also included in the reviews are analyst's notebook pages, number of samples and sample identifications, dilutions, percent moisture, sample weights, chain-of-custody forms, standard preparation notebooks, instrument logbooks, etc. After ensuring that all these items are present and complete, the QA staff proceeds to review the raw data for precision, accuracy, and completeness. The raw data are checked against the reported values, and the appropriate calculations are spot checked.

Any discrepancies pertaining to any of the previously mentioned QA/QC checks are directed to the analytical task manager for verification, clarification, and/or correction, if necessary. Other queries regarding the data transmission file (e.g., improper method codes or incomplete field data) are addressed directly to Data Management. The questions are usually written under the "Comments" section of the USAEC Data Review Checklist (see Figure 8-3) or on separate attachments. Once the questions are satisfactorily answered, the QA staff initials and dates the batch and appropriate sections. The batch folder is then returned to Data Management for entry into IRDMIS.

The control charts are reviewed and transmitted to USAEC and Arthur D. Little weekly by the laboratory QA Supervisor. The control charts are reviewed by the laboratory coordinator, analytical task manager, and QA staff before any data are transmitted to USAEC IRDMIS data files.

Three data levels are used to indicate increasing QA and validation performed on the data. Data reviewed by Arthur D. Little QA staff and subsequently transmitted to USAEC IRDMIS are considered to be Level I data. At USAEC, Potomac Research, Inc. (PRI), the USAEC on-site data management contractor, loads the data into a computer for group and record checks. Errors, if present, are reported to the USAEC COR and chemist. Based on the nature of the error, the data are corrected or rejected. When the data have successfully passed group and record checks, they are elevated to Level II. Level II data become Level III when they are uploaded into the USAEC computer system. Level III data are available to users to create reports and graphs, but they cannot be changed by contractors. Generally, only Level III data are available to the USAEC COR. Under unique circumstances, the COR may request and receive Level I data. Level I data are used for information purposes only. Major decisions and risk assessments are based on Level III data only.

## 8.0 Data Reduction, Validation, and Reporting

### 8.5 IRDMIS Record and Group Checks

After each data packet has been reviewed by key individuals and validated by QA and data management staff, the data file from the packet is loaded into the USAEC IRDMIS systems at DataChem and run through the first record check and then the group check. Every data point is checked using these two routines. IRDMIS record check determines the following:

- Whether file names (such as CGW, CSW) and site type (BORE, WELL) combinations are valid.
- Validity of sampling program and technique, and existence or absence of depth measurement.
- Sample date, preparation/extraction date, and analysis date are compared to determine any holding-time violations.
- All test names are verified as valid, and either performance demonstrated or flagged as non-performance demonstrated, at the time of analysis or at present.
- Value compliance with Certified (Performance Demonstrated) Reporting Limit and Upper Certified (Performance Demonstrated) Limit.
- Correct Boolean values, such as ND, LT.
- Correct QC test, mantissa and exponent values, and uncorrected mantissa and exponent values.
- If required, dilution mantissa, exponent, and moisture content inclusion.
- Whether all required flagging codes are included.

IRDMIS group check determines the following:

- That all test names/analytes found in QC are present in all of the samples.
- That all required QC spikes exist, all spiking levels are valid as determined by the methods table, and no aberrations exist in QC or sample data.

Specific criteria for record checks are based on the specific analytical method and on the current performance demonstration status of the laboratory performing the analysis. These criteria are stored in IRDMIS as certifications (performance demonstrations) tables.



## **8.0 Data Reduction, Validation, and Reporting**

If any errors are found in group and record check that are not addressed on the Data Review Checklist by the laboratory analysts, laboratory project coordinator, or the QA Coordinator, the lot is returned to the laboratory project coordinator, so that the problem can be rectified. If changes to the analytical data are required, the lot is then resubmitted for QA review and, after re-validation, it is again processed through IRDMIS to ensure that any errors have been corrected.

After the data in a lot have successfully passed QA validation and IRDMIS record check and group check, a transfer file of the lot is created and sent to USAEC via modem. The data are again run through record and group check by USAEC, and after passing the data checks, are elevated to Level II.

### **8.6 Data Reporting**

The results for samples analyzed for USAEC projects are entered into the USAEC-provided software program (IRDMIS). Data created using the IRDMIS can then be electronically transmitted to PRI or a diskette together with hard copy printouts can be submitted.

All the subcontracted laboratory data are entered on a coding form by the analyst, which is verified by the peer checker and group leader/section manager. QA personnel review data for obvious errors. These data are encoded onto a diskette, checked through two USAEC software routines, then printed out and verified by visual inspection by a Data Entry Specialist. Verified analytical results are then submitted to PRI. DataChem retains a duplicate diskette of all data submitted.

All information pertaining to the analysis of a lot of samples is collected into a data package at the completion of analysis. The contents of data packages varies with methods of analysis. The package is reviewed by Quality Assurance to eliminate technical errors that might affect the litigation quality of the data. The reported data are also reviewed by Data Entry for completeness before release.

The subcontracted laboratory subsequently sends data packages to Arthur D. Little for final review (10 percent of all data packages). Subsequent to the final review, all pertinent documentation in appropriately labeled boxes is delivered to USAEC.

## **9.0 Internal QC Checks and Frequency**

### **9.1 Control Samples**

Control samples are those that are introduced into the train of environmental samples to function as monitors of the analytical method. All required QC samples will be prepared from standard matrices or actual field samples and processed through the complete performance demonstrated analytical method. Stock solutions used to spike QC samples will be prepared independently of stocks used for calibration or performance demonstration samples.

### **9.2 Field Control Samples**

Various types of field QC samples are used to check the cleanliness and effectiveness of field handling methods. Field QC samples help indicate whether project data quality objectives have been met by providing quantitative and qualitative measures of precision, accuracy, representativeness, completeness, and comparability parameters. They are analyzed in the laboratory as samples, and their purpose is to assess the sampling and transport procedures as possible sources of sample contamination and document overall sampling and analytical precision. Field staff may add blanks or duplicates if field circumstances are such that they consider normal procedures insufficient to prevent or control sample contamination, or at the direction of the Task Manager. Rigorous documentation of all field QC samples in the site logbooks is mandatory.

Field QC samples and the programmatic recommendations for frequency of collection are briefly described below. The specification and number of field QC samples to be collected at the FGGM site are provided in Table 4-1.

#### **9.2.1 Trip Blanks**

Trip blanks are not exposed to field conditions; results from the analysis of trip blanks are used to assess potential contamination from everything except ambient field conditions. Trip blanks are prepared at the laboratory prior to the sampling event by adding reagent ground water to a 40-ml VOA vial containing two to three drops of concentrated hydrochloric acid; they are shipped with the sample bottles. One trip blank will be used with every shipment of water samples for volatile organic analysis. Each trip blank will be transported to the sampling location, handled in the same manner as a field sample (except the bottle cap is not removed), and returned to the laboratory for analysis without having been opened in the field.

#### **9.2.2 Field Equipment/Rinsate Blanks**

The results of analyzing field equipment/rinsate blanks are used to document that sampling equipment have been properly prepared and cleaned before field use and that cleaning procedures between samples are sufficient to minimize cross-contamination. Rinsate blanks are prepared onsite by passing analyte-free water over sampling equipment; they are analyzed for all applicable parameters. If a sampling team is familiar with a particular site, it may be possible to predict the areas or



## 9.0 Internal QC Checks and Frequency

samples that are likely to have the highest concentration of contaminants. The equipment blank sample should be collected after a sample is expected to exhibit high concentrations of target analytes.

Rinsate blanks will generally be collected at a frequency of one per day per equipment type used that day. Rinsate blanks will not be collected for sampling activities using dedicated equipment to collect each sample.

### 9.2.3 Field Duplicates

Field duplicates are two samples collected independently at a sampling location during a single sampling event. The results of analyzing field duplicates are used to assess the consistency of the overall sampling and analytical system. Field duplicate samples are generally collected at a rate of 1 per 20 or fewer samples per matrix.

## 9.3 Laboratory Control Samples

QC data are necessary to determine precision and accuracy and to provide quantitative evidence that the method is performing comparably or better than when documented during method development and performance demonstration. Laboratory-based control samples will consist of standards, surrogates, spikes, and blanks. Data generated from control samples included in each lot will be plotted on control charts to monitor day-to-day variations in routine analyses. For this program DataChem will follow the approach described by the USAEC QA Program for performance demonstrated methods with respect to laboratory control samples. For non-performance demonstrated methods will follow the specific method directives. Generally, a blank, a spike, and a duplicate will be included in each lot of 20 or fewer samples.

The types of laboratory control samples and the minimum acceptable performance for non-performance demonstrated methods for USAEC projects are briefly described below.

### 9.3.1 Laboratory Blanks

In addition to field blank samples, three types of blanks that may be analyzed in the laboratory are calibration blanks, method blanks, and reagent blanks. Method blanks and reagent blanks are used to assess laboratory procedures as possible sources of sample contamination. Calibration blanks establish the analytical baseline against which all other blanks are measured.

- Method blanks are laboratory blanks that correspond to the first step in sample preparation and as such, provide a check on contamination resulting from sample preparation and measurement activities. For USAEC-performance demonstrated procedures, method blanks for water and soil samples consist of a standard matrix that is subjected to the entire sample procedure as appropriate for the analytical

## 9.0 Internal QC Checks and Frequency

method being utilized. For non-performance demonstrated methods, the method blank is typically an appropriate volume of laboratory water carried through the entire preparation and analysis procedure.

- Reagent/Solvent blanks are closely related to method blanks, but they do not incorporate all sample preparation materials and analytical reagents in one sample. When a method blank reveals significant contamination, one or more reagent blanks may be prepared and analyzed to identify the source of contamination.
- Calibration blanks consist of pure reagent matrix and are used to zero an instrument's response to the level of analytes in the pure reagent matrix. They do not provide a direct indication of the types, sources, or levels of contamination, but they establish the analytical baseline.

### 9.3.2 Laboratory Duplicates

Laboratory duplicate samples are defined as two sample aliquots taken from the same sample container and analyzed independently. The results of these analyses serve as an indicator of the precision of the method and the sample results. The frequency of these duplicates is specified in the performance demonstrated methods. For non-performance demonstrated methods, duplicates will be prepared with the frequency specified in the referenced method.

### 9.3.3 Calibration Standards

A calibration standard is prepared in the laboratory by dissolving a known amount of a pure compound in an appropriate matrix. The final concentration calculated from the known quantities is the true value of the standard. The results obtained from these standards are used to generate a standard curve and thereby quantify the compound in the environmental sample. See Section 7.0 for calibration procedures.

### 9.3.4 Spike Sample

A sample spike is prepared by adding to an environmental sample or standard matrix (for USAEC-performance demonstrated methods; before extraction or digestion), a known amount of pure compound of the same type that is to be analyzed for in the analysis. The spike may also be a surrogate compound for the analyte of interest. These spikes simulate the background and interferences found in the actual samples and provide a mechanism to verify overall method performance. The calculated percent recovery of the spike is taken as a measure of the accuracy of the total analytical method. For USAEC-performance demonstrated methods, between one and three spiked samples, as specified in each method, will be included in each lot. For non-performance demonstrated procedures, spiked samples will be analyzed with the frequency specified in the method.



## 9.0 Internal QC Checks and Frequency

### 9.3.5 Internal Standard

An internal standard is prepared by adding a known amount of pure compound to the environmental sample; the compound selected is not one expected to be found in the sample, but is similar in nature to the compound of interest. Internal standards are added to the environmental sample just prior to analysis.

## 9.4 Concentration and Frequency of Control Samples

One method blank shall be included in each analytical lot, regardless of performance demonstration class. A single method blank/spike for GC/MS procedures (Class 1A) serves as a standard matrix QC blank and spike. The frequency of QA samples is summarized in Table 9-1. The spiked QC samples described below will be included in each analytical lot:

### 9.4.1 Class 1 Performance Demonstrated Method

- Two independently-prepared spiked standard matrix QC samples shall contain all the control analytes at a concentration near the upper end of the certified (performance demonstrated) range or approximately 10 times certified (performance demonstrated) reporting limit (CRL).
- One spiked standard matrix QC sample prepared at the regulatory action level or approximately two times certified (performance demonstrated) reporting limit.

Control analytes will be specified in USAEC standardized method. For multi-analyte methods, USAEC will designate the required control analytes. Control limits will be initialized for all analytes.

Control charts will be maintained for each control analyte. Out-of-control situations are discussed in Section 12.0.

### 9.4.2 Class 1A Performance Demonstrated Method (GC/MS only)

- One independently-prepared standard matrix QC sample (method blank/spike), containing all the performance demonstrated surrogate analytes at approximately 10 times certified (performance demonstrated) reporting limit (not to exceed the upper limit of the certified (performance demonstrated) range). For the method blank/spike, surrogate results represent the QC spike, while unspiked, non-surrogate results represent the method blank.
- Every field sample will be spiked with performance demonstrated surrogate analytes at approximately 10 times certified (performance demonstrated) reporting limit. The spike concentration will be the same for all the samples.

## 9.0 Internal QC Checks and Frequency

**Table 9-1: Frequency of Laboratory QC Samples for USAEC-Performance Demonstrated Methods**

| USAEC CLASS | ANALYSIS               | QC SAMPLES FREQUENCY/LOT |         |
|-------------|------------------------|--------------------------|---------|
|             |                        | Method Blank             | Splikes |
| 1           | Metals                 | 1                        | 3       |
|             | Explosives             | 1                        | 3       |
|             | Nitrate                | 1                        | 3       |
|             | PCBs                   | 1                        | 3       |
|             | Sulfate                | 1                        | 3       |
|             | Chloride               | 1                        | 3       |
| 1A          | VOAs                   | 1*                       | 1       |
|             | BNAs                   | 1*                       | 1       |
| 2           | PCBs<br>(Confirmation) | 1                        | 1       |

\* = Surrogates only



## 9.0 Internal QC Checks and Frequency

Control analytes will be specified in the USAEC standardized method. Additional non-surrogate target analytes may be specified by the USAEC project officer.

Control charts will be maintained for each control analyte. Out-of-control situations are discussed in Section 9.6.

Results of natural matrix surrogate spikes are reported to IRDMIS. Appropriate flagging codes will be used to indicate any problems with surrogate recoveries.

## 9.5 Data Reporting for Quality Control

### 9.5.1 Class 1, Class 1A, and Class 1B Performance Demonstrated Methods

Results for each analyte in the spiked QC sample will be determined using the same acceptable calibration curve that is used for analytical samples in the lot. Raw values below the CRL will be reported as "less than" the reporting limit. All certified (performance demonstrated) data will be entered into IRDMIS by personnel trained in the use of IRDMIS.

The results for the method blank and spiked QC samples will be quantified each day of analysis. A new lot of samples will not be introduced into the analytical instrument until the results for QC samples in the previous lot have been calculated, plotted on control charts, and the entire analytical method has been shown to be in control.

Data from the method blank will be reported, usually as "less than" the CRL for each analyte. Any values above the terms of concentration, will be entered into IRDMIS. Data collected from analyses with contaminated blanks will not be used or will be reported flagged.

## 10.0 Performance and System Audits

Performance audits are a quantitative evaluation of a measurement system and generally consist of evaluation of a laboratory's performance in analyzing performance evaluation samples and blind samples. DataChem Laboratories has participated in performance audits by USAEC and has also participated in EPA's water pollution and water supply performance evaluation program.

System audits are a qualitative on-site review and evaluation of the components and implementation of USAEC's QA Program (January 1990). They consist of field, laboratory, and project audits that are performed by qualified personnel from the Arthur D. Little QA or technical staff or from external regulatory agencies.

The Quality Assurance reviews under this subtask are systematic evaluations of four aspects of the Fort Meade project: (1) field/geotechnical activities, (2) laboratory analysis activities, (3) data files and packages, and (4) overall project activities and documentation. The field Quality Assurance reviews will be undertaken by the Arthur D. Little Project QA Officer or his designee. The laboratory Quality Assurance reviews will largely be undertaken by our subcontracted laboratory, with QA oversight provided by the Arthur D. Little Lead Chemist or her designee. The Arthur D. Little Lead Chemist will also review IRDMIS data files and USAEC data packages from our subcontracted laboratory prior to sending files and packages to USAEC. These reviews will assure that activities and data are implemented in accordance with this Work Plan and the Quality Control Plan and associated Standard Operating Procedures, provided as a separate document. These documents adhere to the requirements specified in the *USATHAMA QA Program*, and the *USATHAMA Geotechnical Requirements for Drilling, Monitoring Wells, Data Acquisition, and Reports*.

### 10.1 Field Audits

Field audits will be performed on a variety of projects to determine the accuracy of the field sampling, documentation, and measurement systems. A schedule for field audits for the FGGM field sampling effort will be determined by the Arthur D. Little Task Manager or the Project QA Officer, and USAEC.

Field Quality Assurance reviews will be performed on site for one day during field investigation activities. The reviews will be conducted by the Project Quality Assurance Officer or his designee. Through a combination of on-site observations and on-site and off-site review of documentation, the following will be reviewed to ensure conformance with the above referenced documents:

- Field logbooks and forms
- Field chemical/physical analyses including calibration and QC samples
- Containers and sample preservation used for collected samples



## 10.0 Performance and System Audits

- Sample storage and security
- Sample containers
- Location and elevation survey
- On-site steam cleaning drill rig procedures prior to drilling activities, between each well, and before leaving the site
- "Dig-safe" and UXO screening procedures
- Confinement and containerization of drilling wastes (waste steam cleaning condensates from drill rigs and the PVC pipe used for casings; drilling fluid, if used; surface runoff, and antifreeze if used)
- Drilling activities (water sources used) and well materials (Ottawa sand, bentonite and grout)
- Well development and presample purging techniques
- Depth measuring techniques
- Well construction and security
- Accurate drawings and notes of the well's location and drilling operations
- Specified numbers and types of soil, ground water, surface water, and sediment samples are collected and sent to the laboratory
- Custody forms, including sample labels and chain-of-custody records

The Field Checklist provided in Appendix W of the USAEC QA Program, PAM-11-4, will be used during this audit. External audits may also be performed by a representative of the USAEC Chemistry Branch.

## 10.2 Laboratory Audits

A system internal audit by the DataChem Laboratories Project Manager and QA Coordinator (or designees) is made before any new experimental procedures are implemented. Systems audits are also made for critical functions during the sampling and analysis program. The system audit is of a qualitative nature and consists of an on-site review of the laboratory's QA system and physical facilities for sampling, calibration, and measurement. The results of these reviews will be documented in initial and final laboratory visit checklists.

## 10.0 Performance and System Audits

Critical functions will be audited by the QA Coordinator to verify that:

- Standards, procedures, records, charts, floppy disks, and notebooks are properly maintained
- Actual procedures agree with written instructions
- QA records are adequately filed and maintained to assure protection and retrievability

The QA Coordinator or designee will also assess the results of QC sample analyses.

In addition to internal laboratory audits, USAEC will perform external audits. Currently, DataChem Laboratories is audited by USAEC every six months by representatives of the USAEC Chemistry Branch.

Findings from DataChem audits will be documented in a bound notebook and maintained in a Project QA file. Findings will include observations and notations as to whether approved practices are followed. A summary of findings will be distributed to the DataChem Laboratories Corporate QA Officer, the Project Manager, Analytical Coordinator, Arthur D. Little Task Manager and Lead Chemist, and USAEC.

### 10.2.1 Data Review

As required by the USAEC QA Plan, all data packages will be reviewed by the DataChem Quality Assurance Coordinator. This review serves two purposes; it ensures that all required data and documentation are provided in the package and it checks the content for technical and recordkeeping errors. The reviewer's name and date of review will be recorded on the QAC Checklist, any corrective actions required will also be noted. When the corrective action has been completed the QAC will initial and date the original comment. The QAC's signature on the checklist will indicate that the data are considered valid and usable.

Our subcontracted laboratory will provide Arthur D. Little with USAEC data packages and IRDMIS data files. We will review data packages and files and transfer reviewed files to IRDMIS.

An additional review of approximately 10 percent of the data packages will be performed by the Arthur D. Little Lead Chemist. The packages will be chosen to cover as broad as possible a range of analyses and matrices. In some cases, a particular lot may be selected for additional review by the Arthur D. Little or USAEC Project Manager. The Lead Chemist will assess the completeness of the documentation provided, adherence to the performance demonstrated or other published method, adherence to USAEC quality control requirements and acceptability of the quality control data. The Lead Chemist will also provide a



## **10.0 Performance and System Audits**

technical review of the data and verify at least one calculation for standard preparation and final reported analyte values from the raw data contained in the data packages to the final reported value on IRDMIS. Any discrepancies or omissions will be discussed promptly with DataChem. A copy of the Arthur D. Little Lead Chemist's review will be added to the data package.

Any deviations or problems with data packages will be reviewed with the subcontractor laboratory, and appropriate corrective actions will be taken as necessary and will be fully documented.

### **10.3 Project Audits**

Project audits may also be performed on files containing relevant project documentation. These audits will be triggered by apparent non-conformance to the USAEC QA Program and/or in response to corrective actions. Project files are evaluated against internal document control SOPS. Project audits may be performed on a random percentage of projects by the Project QA Officer or his designee.

## **11.0 Preventive Maintenance**

### **11.1 Field Instruments**

All field instruments and equipment used for sample analysis will be serviced and maintained only by qualified personnel. All repairs, adjustments, routine maintenance, and calibrations will be documented in an appropriate logbook or data sheet that will be kept on file at the field equipment warehouse. The instrument maintenance logbooks will clearly document the date, the description of the problems, the corrective action taken, the result, and who performed the work. Arthur D. Little maintains a sufficient number of spare parts for all field instruments and, in many cases, backup instrumentation to minimize downtime of instruments and delays in analyses.

### **11.2 Laboratory Equipment**

The subcontracted laboratory, DataChem, maintains maintenance contracts with the major instrument manufacturers for 24-hour, 7 day per week emergency call service. DataChem performs routine maintenance to prevent instrument malfunction and minimize downtime, and to optimize instrument capabilities.

The schedule of preventative or routine maintenance checks are, in general, outlined within the specific equipment's operation manuals and in the analytical procedures performed. DataChem adheres to these schedules, and it is the responsibility of both the project analyst and management to monitor that these checks are completed. Appendix B provides the SOP Reference for Instrumentation Maintenance for our subcontracted laboratory.

The laboratory maintains an inventory of replacement parts for all analytical instrumentation; this enables analysts to perform routine maintenance and repair of instruments as needed.



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

This section describes the statistical analysis of data obtained during analysis of FGGM samples by USAEC-performance demonstrated methods. The calculations described in this section are contained in computer software developed by USAEC.

The statistical calculations compare the measured concentration of standards in spiked samples with the known spiked concentrations of these target analytes. The measured concentrations are determined from calibration curves constructed according to the standardized method. Recovery factors will not be used to correct measured concentrations during analysis of the performance demonstration data. These calculations must be performed for each target analyte in a method.

### 12.1 Lack of Fit (LOF) and Zero Intercept (ZI) Tests

All data must be collected during periods when instrumental calibration was in control (i.e., within plus or minus 10 percent of the mean response for inorganics analyses in surface/ground waters and within plus or minus 25 percent of the mean response for all other analyses). Data obtained from valid methods using properly calibrated instruments are expected to be linear and have a zero intercept, when measured concentrations are compared to the target concentrations. This relationship must be tested because calculation of the CRL assumes that a linear relationship exists.

Data obtained during performance demonstration analyses shall be first examined for any outliers before being tested for linearity using the LOF and ZI tests. In the absence or replacement of an outlier, data from each of the performance demonstration analyses shall be pooled and tested for LOF.

### 12.2 Certified (Performance Demonstrated) Reporting Limit (CRL)

Before any analytical system is employed in a survey, sufficient spikes and blanks will be run to statistically establish the lowest sample concentration to be reported. This concentration is the CRL. For USAEC projects, CRLs shall be determined by using the USAEC program with 95 percent confidence limits. This CRL is associated with the entire method and reflects all sample preparation and measurement steps.

The CRL is derived from the following assumptions:

- The relationship between the measured concentration and target concentration is linear
- The variance about the least squares linear regression line is homogeneous over the tested concentration range
- Measured concentrations for a given target concentration are normally distributed

## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

Based on these assumptions, the least squares linear regression line, of the form indicated in Equation 1, can be determined. The performance demonstration performance data (X, Y paired data) are used to determine the slope and Y-intercept of the least squares regression line according to the formulae provided below in Equations 2 and 3; these equations assume that errors occur only in the measured concentration.

### Equation (1)

$$Y = Y_o + bX$$

where:

Y = found concentration;

$Y_o$  = Y axis (found concentration) intercept;

b = slope of the line; and

X = target concentration.

### Equation (2)

$$\text{slope} = b = \frac{N \sum X_i Y_i - \sum X_i \sum Y_i}{N \sum X_i^2 - (\sum X_i)^2}$$

where:

N = number of data points;

$X_i$  = the i-th target concentration; and

$Y_i$  = the i-th found concentration.

### Equation (3)

$$Y \text{ axis intercept} = Y_o = \frac{\sum Y_i - b \sum X_i}{N}$$

where:

b = slope of the least squares linear regression line from Equation 2.



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

The equations for the upper confidence limit (Equation 4) and the lower confidence limit (Equation 5) about the regression line are provided below:

### Equation (4)

$$Y = Y_o + S_{YX} t \left[ \frac{1}{N} + \frac{(X_i - \bar{X})^2}{\sum (X_i - \bar{X})^2} \right]^{1/2}$$

### Equation (5)

$$Y = Y_o + bX S_{YX} t \left[ \frac{1}{N} + \frac{(X_i - \bar{X})^2}{\sum (X_i - \bar{X})^2} \right]^{1/2}$$

where:

### Equation (6)

$$= S_{Y, X} \left[ \frac{\sum (Y_i - [\bar{Y} + b(X_i - \bar{X})])^2}{N - 2} \right]^{1/2}$$

$Y_o$  = calculated Y axis intercept;

$t$  = Student's t-test for 2-tailed  $P = 0.10$  and  $N - 2$  degrees of freedom;

$\bar{X}$  = the average of all target concentrations; and

$\bar{Y}$  = the average of all found concentrations.

The calculated reporting limit,  $X_d$ , is the value of  $X$  corresponding to a point on the lower confidence limit curve where the value of  $Y$  equals the value of  $Y$  on the upper confidence limit curve at  $X = 0$ . An example of the statistical analysis of reporting limit using the USAEC computer software is shown in the USAEC QA Program manual (January 1990).

The calculated reporting limit will be reported as the CRL of the method, provided that at least one of the tested concentrations is at or below the calculated reporting

## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

limit. Otherwise, the lowest tested concentration is the minimum level that can be reported as the CRL. The CRL will not be less than the lowest tested concentration.

The data provide an optimistic estimate of the method reporting limit because interferences found in natural samples will be absent. The highest tested concentration will represent the upper limit of reportable data. All sample measurements must be performed within the tested range. A calculated reporting limit higher than the highest target concentration indicates that either an invalid range was chosen or the method is not suitable for analysis of that compound.

### 12.3 Method Performance Demonstration Accuracy

As calculated according to Section 12.2, the slope,  $b$ , of the least squares linear regression line of a plot of observed versus target concentrations is a measure of the accuracy of the method. A slope (accuracy) of "plus one" (1.00) indicates 100 percent recovery over the complete analytical method and tested range. Failure to consider the intercept, if it is significantly different from zero, could result in an erroneous estimate of the accuracy. If the intercept is significantly different from zero, then there is a need to investigate whether the blank was correctly applied or if there is some other systematic error in the system. At no time should the laboratory continue until this is investigated. Experimental values may deviate from this expected value. The performance demonstration data will provide an optimistic estimate of the method accuracy because interferences found in natural samples will be absent. The accuracy estimate for the complete performance demonstration data set is incorporated into the USAEC IRDMIS. The slope for the complete data set will be used to indicate the accuracy of the method.

### 12.4 Method Performance Demonstration Standard Deviation

For all method performance demonstration, the standard deviation,  $s$ , will be calculated at each target concentration according to Equation 7. The standard deviation provides an indication of the precision of the analysis. This calculation is performed by the USAEC software.

Equation (7)

$$\text{Standard deviation} = S = \left[ \frac{\sum Y_i^2 - \frac{(\sum Y_i)^2}{N}}{N - 1} \right]^{1/2}$$



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

where:

$Y_i$  = the measured concentration; and

$N$  = total number of  $Y$  values at each target concentration.

### 12.5 Method Performance Demonstration Percent Inaccuracy

For all method performance demonstration, the percent inaccuracy will be calculated at each target concentration according to Equation 8. This calculation is performed by the USAEC software.

Equation (8)

$$\text{Percent inaccuracy} = \frac{\bar{Y} - X}{X} (100)$$

where:

$X$  = target concentration; and

$\bar{Y}$  = average measured concentration at the target concentration.

### 12.6 Method Performance Demonstration Percent Imprecision

For all method performance demonstration, the percent imprecision will be calculated at each target concentration according to Equation 9. This calculation is performed by the USAEC software.

Equation (9)

$$\text{Percent imprecision} = \frac{S}{\bar{Y}} (100)$$

where:

$S$  = standard deviation; and

$\bar{Y}$  = average measured concentration at the particular target concentration.

## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

### 12.7 Data Moving-Average Accuracy and Precision

Moving-average control charts will be maintained for the specified surrogates in the spiked standard matrix sample (Class 1A). The X - R three-point moving-average control chart will be constructed for each control analyte as follows:

- Use percent recovery to allow for minor variations in spiking concentrations
- The first plotted point is the average of the first three recoveries (from performance demonstration, at concentrations nearest the spiking level)
- Subsequent points are obtained by averaging the three most recent individual recovery values (outliers excluded from calculation but not from plot)
- The range for each point is the difference between the highest and lowest value for each group of three values
- The central line, upper warning limit (UWL), upper control limit (UCL), lower warning limit (LWL), and lower control limit (LCL) for the control charts are calculated using the following formulas:

Equation (10)

$$\text{Average } \bar{\bar{X}} = \frac{\sum \bar{X}}{K}$$

Equation (11)

$$\text{Range } \bar{R} = \frac{\sum R}{K}$$

where:

$\bar{\bar{X}}$  = between-group average of the average recovery of the three points (within group);

$\bar{X}$  = average within-group recovery for the three points;

R = within-group difference between recoveries for data pairs; and



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

$K$  = cumulative number of pairs in the database.

Upper Warning Limit (UWL) on Average:

$$UWL_x = \bar{\bar{X}} + 0.682 \bar{R}$$

Upper Control Limit (UCL) on Average:

$$UCL_x = \bar{\bar{X}} + 1.023 \bar{R}$$

Lower Warning Limit (LWL) on Average:

$$LWL_x = \bar{\bar{X}} - 0.682 \bar{R}$$

Lower Control Limit (LCL) on Average:

$$LCL_x = \bar{\bar{X}} - 1.023 \bar{R}$$

Upper Warning Limit (UWL) on Range:

$$UWL_R = 2.050 \bar{R}$$

Upper Control Limit (UCL) on Range:

$$UCL_R = 2.575 \bar{R}$$

Lower Warning Limit (LWL) on Range:

$$LWL_R = 0$$

Lower Control Limit (LCL) on Range:

$$LCL_R = 0$$

All data will be plotted, regardless of whether the lot is in control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual measurement value will be tested as an outlier using Dixon's test at the 98 percent confidence level (USATHAMA QA Program manual (January 1990), Appendix K). If the datum is not classified as an outlier by the test, the point will be included in updating the control chart limits. If an individual measurement is classified as an outlier, it will be used in calculating the three-point moving average for plotting purposes only; the measurement is then excluded from calculations based on the three most recent acceptable individual points that are used to determine moving-average and the control chart limits. Method control will be judged according to the criteria in Section 8.0.

## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

After the first control chart points, control limits will be recalculated using only in-control data points. Any points falling outside of the control limits (UCL or LCL) will be dropped from the calculations (but left on the charts) and the control limits recalculated using only points between the UCL and LCL. Charts will then be updated with the newly calculated control limits and all points plotted.

Lots associated with points outside of the new control limits may require resampling and/or reanalysis as determined by USAEC COR on a case-by-case basis. These limits will then be used to control analysis of the next 20 lots. The control charts are now the outlier test, although individual measurements will continue to be tested as outliers if they appear not to be representative of the data set. A maximum of the 40 most recent lots will be used to recalculate control limits for 60 or more lots (40-point slide).

When, as a result of audits or QC sample analysis, sampling or analysis systems are shown to be unsatisfactory, a corrective action shall be implemented. The Laboratory QA Coordinator will be notified and the necessary corrective action taken.

### 12.8 Control Charts

For Class 1, Class 1A, and Class 1B performance demonstrated methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and to detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts are not adjusted for accuracy. Control charts are not used with Class 2 performance demonstrated methods.

Control charts include the analyte, method number, DataChem laboratory code of UB, spike concentration, and chart title. All data presented on a control chart are also presented in tabular form. The following charts may be selected from the USAEC-supplied computer control chart program:

- Single-Day X-Bar Control Chart (High Spike Concentration)
- Single-Day Range Control Chart (High Spike Concentration)
- Three-Day X-Bar Control Chart (Low Spike Concentration)
- Three-Day Range Control Chart (Low Spike Concentration)

In addition, the following information is also included on each control chart:

- Three-letter lot designation for each point, shown on the X-axis
- Percent recovery (for X-bar control charts), or range (for R control charts) along the Y-axis
- Upper control limit (UCL)



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

- Upper warning limit (UWL)
- Mean
- Lower warning limit (LWL), on X-bar charts
- Lower control limit (LCL), on X-bar charts

For some analytes specified by USAEC, warning limits on X-bar charts are deleted and replaced by modified control limits based upon data quality specifications.

### 12.8.1 Control Chart Plotting: Single-Day

The initial control chart is prepared using the four days of performance demonstration data closest to the spiking concentration used during analysis. The average (X-bar), average range (R), and control limits for both are updated after each in-control lot for the first 20 lots. Limits established after lot 20 are used for the next 20 lots. Control charts are updated after each 20 lots thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (1-20), the limits established from the previous lots are used to control the current lot.

When modified limits are established, data for samples are accepted if the control data fall between the modified limits. If modified limits have not been established, data for samples are accepted, based upon the recoveries established during performance demonstration and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control chart limits. Out-of-control or outlier points are plotted; however, such lots are not utilized in lot number requirements or control chart calculations.

All recoveries are plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and are not the individual measurement values. Each individual recovery measurement value is tested as an outlier using Dixon's Test at the 98 percent confidence level. If the datum is not classified as an outlier, it is not used in updating the control chart limits. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits are recalculated using only in-control data points. The control limits are then drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) are dropped from the calculations (but left on the charts) and the control limits recalculated using only points between those limits. This practice of dropping points and recalculating limits is performed only once, at the initialization of stable limits. Charts are then updated with newly calculated control limits and all points plotted.



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

### 12.8.2 Three-Point Moving Average

Analytical data for analytes prepared in the single low concentration QC sample are plotted and evaluated on a three-day-moving-average control chart. Data for the surrogates spiked in a standard matrix and used in GC/MS analyses are also charted on a three-day-moving-average control chart. Plotting criteria for the three-point moving average control charts are similar to those described above for single-day control charts. Data for analytes prepared in duplicate QC samples at high concentrations are plotted and evaluated on single-day control charts.

Computer generated control charts maintained by Quality Assurance are updated and printed weekly, while analysts plot data points by hand as sample lots are analyzed. This allows for both computer maintenance and evaluation of a large data base with software calculation of control limits, and immediate daily surveillance of analytical trends.

### 12.9 Out-of-Control Conditions

Results of the analysis of quality control samples are reported to QA within 48 hours of completion through the analyst's submission of a Preliminary QC Report.

The analyst quantifies each analyte in the method blank and spiked QC sample each data of analysis. Processing of additional lots will not occur until the results of the previous lots have been calculated, plotted on control charts as required, and the entire analytical method shown to be in control.

An indication of an out-of-control situation may include: a value outside the control limits or classified as outlier by statistical test; a series of seven successive points on the same side of the mean; a series of five successive points going in the same direction; a cyclical pattern of control values; or two consecutive points between the UWL and UCL or the LWL and LCL.

If the points for at least two-thirds of the control analytes for a multi-analyte method are classified as in-control, the method is in-control and environmental sample data may be reported. A method may be deemed out-of-control even if greater than or equal to two-thirds of the control analytes meet control criteria. Of the remaining control analytes (less than one-third possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is deemed out-of-control. If data points for fewer than two-thirds of the control analytes are classified as in-control, the method is considered to be out-of-control and all work on that method must cease immediately. No data for environmental samples in that lot may be reported.

In all cases, investigation by the analyst and the Quality Assurance Coordinator is required to determine the cause of the condition and to decide on appropriate



## 12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

corrective action. The pertinent details of the situation and the corrective action taken are fully documented in a Corrective Action Report (CAR). (See also Section 10.0.) Field sample data effected by the situation are evaluated and reanalyzed as necessary.

When a method is determined to be out-of-control, the analysis of field samples by that method is suspended. Corrective action must be documented and the method must be demonstrated to be in-control before analysis of field samples is reinstated. Analytical control is demonstrated through the acceptable analysis of an appropriate set of QA samples.

### 12.10 Non-USAEC Methods

For non-USAEC methods, including laboratory tests for Total Dissolved Solids (TDS) and Total Petroleum Hydrocarbons (TPHC) and field tests for pH, temperature, conductivity, turbidity, and total volatile organics (by photoionization detection), the QC samples and procedures for assessing data precision and accuracy are provided in the referenced method or Standard Operating Procedure.

### 12.11 Completeness

Completeness is a measure of the amount of usable data obtained from a measurement system compared to the total amount expected to be obtained. It is calculated as follows:

$$\text{Completeness (\%)} = \frac{\text{Number of valid analyses}}{\text{Number of analyses requested}} \times 100$$

### 13.0 Corrective Actions

When, as a result of staff observations, audits or QC sample analysis, sampling or analysis systems are shown to be unsatisfactory, corrective action will be implemented. Staff and management at Arthur D. Little and/or DataChem may be involved in the corrective action. If previously reported data are affected by the situation requiring correction or if the corrective action will impact the project budget or schedule, the action will directly involve the Task Manager, the USAEC COR, and the USAEC Quality Assurance Chemist. Corrective actions are of two kinds:

- Immediate - to correct or repair nonconforming equipment and systems. The need for such an action will most frequently be identified by the field technician or analyst actually doing the work.
- Long-term - to eliminate causes of nonconformance. The need for such actions will probably be identified by audits. Examples of this type of action include:
  - Staff training in technical skills or in implementing the QA Program
  - Rescheduling of laboratory and/or sampling routines to ensure analysis within allowed holding times
  - Identifying vendors to supply reagents of sufficient purity for field work
  - Revising QA system or replacing personnel
  - Personnel reassignment
  - Field instrumentation replacement

For either immediate or long-term corrective actions, the steps comprising a closed-loop corrective action system are as follows:

- Define the problem
- Assign responsibility for investigating the problem
- Investigate and determine the cause of the problem
- Determine a corrective action to eliminate the problem
- Assign and accept responsibility for implementing the corrective action
- Establish effectiveness of the corrective action and implement the correction
- Verify that the corrective action has eliminated the problem

Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, corrective action employed, and verification that the problem has been eliminated will be documented.

In addition, if the corrective action results in the preparation of a new standard or calibration solution(s), then a comparison of the new versus the old solution will be performed and the results supplied with the weekly QC submittal as verification that the problem has been eliminated.



## **13.0 Corrective Actions**

### **13.1 Field Situations**

Deviations from quality in field operations that require corrective action in the field will be identified by field audits as described in Section 10.0 and by other more immediate occurrences, such as equipment malfunction and on-site observations by the field supervisor. Once the problem has been identified, prompt and appropriate action will be taken by the field staff, Task Manager or field supervisor to correct the situation. After a corrective action has been implemented, its effectiveness will be verified and documented in the site log. If the action does not resolve the problem, appropriate personnel will be assigned by the Program Manager or Task Manager to investigate and effectively remediate the problem.

Documentation of all corrective action is required. Immediate corrective actions taken in the field will be documented in the field logbooks and approved by the field supervisor or Task Manager. Corrective actions that result in deviations from the Work Plan or Project QC Plan will also be documented in a memorandum to the Arthur D. Little Task Manager and QA Officer. They will ensure appropriate changes are incorporated into the final report. Corrective actions initiated as a result of a field audit must be documented in a memorandum from the Program QA Officer to the Task Manager.

### **13.2 Laboratory Situations**

If weaknesses or problems are uncovered during system or performance audits or QC sample analysis, corrective action will be initiated immediately. The DataChem Laboratories Project Manager, Analytical Coordinator, QA Coordinator, and analyst must be involved in the corrective action. If previously reported data or project schedule or budget will be affected, then the corrective actions planned will be directly reported to the DataChem Laboratories Project Manager, Arthur D. Little Program Manager, Arthur D. Little Task Manager, and Arthur D. Little Lead Chemist. Corrective actions may also be initiated by the analyst as required from daily review of control charts.

Corrective action might include, but not necessarily be limited to: recalibration of instruments using freshly prepared calibration standards; replacement of lots of solvent or other reagents that give unacceptable values; instrument repair, additional training of laboratory personnel in correct implementation of sample preparation and analysis methods; and reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements.

## 14.0 Quality Assurance Reports to Management

### 14.1 Laboratory Reports

Each daily report generated has a QA section associated with the text. Any matrix characteristics or other physical parameters are noted. The laboratory must confirm that all characteristics indicated by field investigation team match the sample being analyzed by the laboratory. Any discrepancies cause the analysis sequence to be halted.

Normal submissions to the USAEC Chemistry Branch include the IRDMIS submissions (Section 8.0) and the results of QC activities. During those periods when analyses are being conducted, all QC charts (tabular and graphical), as described in Section 12.0, must be submitted to the USAEC Chemistry Branch and Arthur D. Little on a weekly basis. The QC report must be provided to the Chemistry Branch and Arthur D. Little no later than five working days after analyses for a week are completed. Analysis data shall be defined by the day the analytical instrument was run. All points that indicate an out-of-control situation must be evaluated and explained. Any corrective measures and reanalysis of samples must be fully explained and documented, including procedural changes to prevent recurrence. Printouts generated from control chart software programs provided by USAEC shall be utilized, when available. A checklist included with each control chart submission is shown in Appendix Q of the USAEC QA Program, January 1990.

As an appendix to the project final report, the QAC, in coordination with the Analytical Task Manager and the Project Manager, will provide tabulation of all QC sample data, as well as specific observations delineating the control effectiveness for each analytical method. These observations will include the following:

- QC samples in each lot and how analytical results were combined to prepare control charts
- Spike levels and rationale for choosing those levels
- Possible effects on environmental sample results of detected concentrations in method blanks
- Unique matrix characteristics of environmental samples

If any time during the analytical effort a process was not in control, a discussion will be submitted on:

- Rationale for judging a point as in control, if it appears to satisfy an out-of-control criterion listed in Section 9.0
- Investigation of the out-of-control situation
- Actions taken to bring the process back into control



## **14.0 Quality Assurance Reports to Management**

- Actions taken to ensure that the out-of-control situation did not recur
- Disposition of data acquired while the process was out-of-control

### **14.2 Program QA Officer and Lead Chemist Reports**

The Arthur D. Little Program QA Officer and the Lead Chemist will routinely generate reports to maintain the Program and Task managers informed of the QA/QC activities during the course of the FGGM project. These reports will be verbal or in the form of a memorandum and will address any findings encountered during their audits and reviews.

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## Appendix A: DataChem Laboratories Quality Assurance Program Plan

**QUALITY ASSURANCE  
PROGRAM PLAN**

**for  
U.S. ARMY TOXIC AND HAZARDOUS  
MATERIALS AGENCY**

**September 1991**

**Laboratory Analysis  
of Environmental Samples**

**DCL Document QA-3/87**

**DataChem Laboratories  
960 West LeVoy Drive  
Salt Lake City, Utah 84123**



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**1.0**  
**DOCUMENT IDENTIFICATION**

|                          |                                                                               |
|--------------------------|-------------------------------------------------------------------------------|
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| Document Control Number: | QA-3/87                                                                       |
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| Quality Assurance:       | Lance M. Eggenberger, M.S.<br>Phone: 801-266-7700                             |



## 2.0 INTRODUCTION

This document is the DCL Quality Assurance/Quality Control Plan, prepared in compliance with the requirements of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) with analytical laboratory services in support of the implementation of various installation restoration programs. This plan adheres to, and is an implementation of, the USATHAMA QA Program, January 1990, First Edition.

DCL is committed, in strictly following this plan, to provide to USATHAMA analytical data that are of a quality that may be used in litigation. All deviations from this plan or the USATHAMA QA Program will be submitted to USATHAMA for approval prior to implementation in the laboratory. Such deviations will be properly and fully documented.

DCL has conducted analyses for USATHAMA since 1984 under the 1982 USATHAMA QA Program, the Second Edition (March 1987) of the 1985 USATHAMA QA Program, and the January 1990 USATHAMA QA Program, First Edition.

### 3.0

## ORGANIZATION AND RESPONSIBILITIES

#### 3.1 Introduction

Ultimate responsibility for the conduct of all projects, and approval for the implementation of all programs at DCL resides with the Laboratory Director, Dr. James H. Nelson. Functional responsibility for the analytical work is delegated to the Project Manager, Mr. David W. Gayer; to the Analytical Task Managers, Mr. A. Brent Torgensen, and Mr. Richard Wade; and to the Quality Assurance Coordinator, Mr. Ronald H. Marsden.

#### 3.2 Laboratory Director

The Laboratory Director is responsible to assure that DCL resources are adequately allocated to the project and that sufficient staffing and equipment are provided. He oversees and supports the Quality Assurance Coordinator.

#### 3.3 Project Manager

The Project Manager has the responsibility of communication with the USATHAMA Program Contract Officer and oversees and supports the Analytical Task Managers in development, implementation, and operation of the analytical program organization. He is directly responsible for the interpretation of the provisions of the contract for DCL. The Project Manager is also responsible to assure that QA/QC recommendations and corrective actions are implemented.

The Project Manager is authorized to conduct official discussions with the Program Contract Officer concerning the original contractual agreement and delivery orders, and any subsequent modifications to the contractual agreement and/or delivery orders. Laboratory personnel matters are decided in concert with the Analytical Task Manager and appropriate Section Managers.

#### 3.4 Analytical Task Manager

The Analytical Task Manager has the responsibility of implementing the USATHAMA 1990 QA Plan, and for coordinating the sample analysis flow in the laboratory. This will be achieved through the following:

1. Assuring the provision of sufficient equipment, laboratory space, resources, personnel, and quality reagents and materials to properly conduct the required analyses;
2. Supporting the Quality Assurance Coordinator;



3. Submitting documented analytical methods and laboratory certification data to the USATHAMA Project Officer prior to the analysis of field samples;
4. Ensuring that all provisions of the approved Project Quality Control Plan are fully implemented in the laboratory;
5. Ensuring the implementation of corrective action for any QA/QC deficiencies.

The Analytical Task Manager has the authority to suspend analytical work for quality control problems and to implement corrective actions recommended by the Quality Assurance Coordinator. He also has authority to accept or reject increases in the delivery rate of samples, within the bounds set by the contract. He confers with section managers and the Project Manager on personnel matters when they impact on the project.

### 3.5 Quality Assurance Coordinator

The Quality Assurance Coordinator (QAC) has the responsibility of establishing, overseeing, and auditing specific procedures for documenting, controlling, and validating analytical data quality. This is accomplished, in part, through the following:

1. Monitoring the QA and QC activities of the laboratory to ensure conformance with authorized policies, procedures, and good laboratory practices, and recommending improvements as necessary;
2. Informing the Project Manager and/or the Analytical Task Manager of noncompliance with the approved QA Program;
3. Requesting standard analytical reference materials from USATHAMA;
4. Ensuring that all records, logs, standard operating procedures, project plans and analytical results are maintained in a retrievable fashion;
5. Ensuring that standard operating procedures and project QA/QC plans are distributed to all appropriate laboratory personnel;
6. In consultation with the analysts and the Analytical Task Manager, establishing appropriate analytical lot size, including the correct QC samples;
7. Establishing the correct procedures and criteria for evaluating whether analytical performance is acceptable and in-control;
8. Ensuring that samples are received and logged properly, including lot sizing, introduction of required QC samples, and numbering of field samples and control samples;
9. Reviewing all laboratory data before those data are released, verifying that data were collected properly under an in-control analytical system;

10. Ensuring that the DCL quality control chemist, or appropriate analysts, are properly preparing QC samples;
11. Maintaining quality control charts, ensuring timely distribution of such charts, documenting corrective actions, and ensuring that analysts implement and document corrective actions as they become necessary;
12. Ensuring that sample logs, instrumentation logs, and all QC documents are properly maintained, including frequency of entries;
13. Discussing control chart results with the Analytical Task Manager and submitting updated, current charts to the USATHAMA Project Officer on a weekly basis, or as required by USATHAMA;
14. Maintaining an awareness of the entire laboratory operation to detect conditions which might jeopardize controls of the various analytical systems;
15. As directed by USATHAMA, auditing sampling documentation and procedures to ensure proper labeling, handling, transportation, and storage.

The Quality Assurance Coordinator has the authority to:

1. Approve all analytical reports;
2. Reject analytical data which does not meet applicable quality control criteria;
3. Require re-performance of sample analyses which are determined to be out-of-control;
4. Evaluate data and determine apparent long-term trends which may require corrective action;
5. Suspend analytical work, when necessary, to assure corrective actions are taken and that an analysis is again in control.

The Quality Assurance Coordinator also attends and participates in conferences for discussion of quality control and quality assurance problems and procedures.



## 4.0 CERTIFICATION

### 4.1 Laboratory Certification

DCL, as a laboratory, rather than as individual analysts, certifies as proficient in conducting analyses for USATHAMA. Each member of the organization has the education and training necessary to enable that individual to perform assigned functions. A personnel training file is maintained for each individual. Each individual updates the training file as necessary.

Management personnel have earned a Baccalaureate degree from an accredited college or university.

Analytical Chemists have earned a Baccalaureate Degree in Science or related fields from an accredited college or university.

Technical Staff have applicable training, including on the job training, and/or experience in related fields.

### 4.2 Analytical Methods

Analytical methods used for the analysis of environmental samples are described in a set of written instructions completely defining the procedure to be followed to process a sample and obtain an analytical result. An analytical method describes, as a minimum, the analytes for which it is valid, the matrix type, sample preparation, reagent and standards preparation, instrument calibration, and computations used to evaluate the analytical results. Standards and quality control sample requirements are also defined.

Analytical methods are either supplied by USATHAMA or, with approval, developed by DCL. The documentation for proposed methods development includes:

1. The submission of documentation to USATHAMA.
2. A statement of the problem.
3. A description of the technical approach to include specific details on procedures, solvents, instrumentation, etc.
4. An estimate of resources required (to include labor hours, funds and schedule).

When the testing of the analytical procedures has been successfully completed, the method is documented in the standardized USATHAMA format. The format for documentation of all analytical methods is provided in Table 1. The format for data analysis is established by USATHAMA-provided statistical analysis computer software. Updates to the software are implemented upon receipt.

**Table 1.**  
**FORMAT FOR DOCUMENTATION OF METHOD CERTIFICATION**

- I. Summary**
  - A. Analytes
  - B. Matrix
  - C. General Method
- II. Application**
  - A. Tested Concentration Range
  - B. Sensitivity
  - C. Reporting Limit
  - D. Interferences
  - E. Analysis Rate
  - F. Safety Information
- III. Glassware and Chemicals**
  - A. Glassware/Hardware
  - B. Instrumentation
  - C. Analytes
  - D. Reagents and SARMS
- IV. Calibration**
  - A. Initial Calibration
  - B. Daily Calibration
- V. Certification Testing**
- VI. Sample Handling and Storage**
  - A. Sampling Procedure
  - B. Containers
  - C. Storage Conditions
  - D. Holding Time Limits
  - E. Solution Verification
- VII. Procedure**
  - A. Separations
  - B. Chemical Reactions
  - C. Instrumental Analysis
  - D. Confirmational Analysis
- VIII. Calculations**
- IX. Daily Quality Control**
  - A. Control Samples
  - B. Control Charts
- X. References**
- XI. Data**



The analytical method, once certified, is followed for all USATHAMA analyses. Instrumental conditions are optimized within the limits specified by method and documented by the analyst. Any deviation, other than the optimization of instrumental conditions, is pre-approved by USATHAMA before implementation.

All copies of USATHAMA-certified methods are individually numbered. Each distributed method copy must be signed for and dated. A comprehensive list of all distributed methods is kept by the Quality Assurance Coordinator.

#### 4.3 Method Certification

Before field samples may be analyzed by the laboratory, the methods of analysis must be certified. Certification for selected methods, accomplished under other USATHAMA contracts, may be determined by USATHAMA to be acceptable for the work performed under this contract for identical analytes and matrices. If analytes are required for a particular certified method in addition to those which have already been certified, the additional analytes are appended to the current certified method by following full certification procedures for the additional analytes. The current certified method standards, concentrations and analytical conditions are used to certify the additional compounds.

Some methods, including calibration of test and measurement equipment, do not require certification, due to either the nature of the measurement or the intended use of the data. When such methods are part of a project, USATHAMA will not provide a standardized method. However, laboratories must submit sufficient information in test plans, work plans, and project QC plans to describe exactly the procedures to be used. A copy of a proposed method must be submitted to the USATHAMA Chemistry Branch before it is used on any project.

The following methods do not require USATHAMA certification by the USATHAMA Chemistry Branch: temperature, conductivity, pH, oil and grease, hardness, asbestos, alkalinity (carbonate/bicarbonate/hydroxide), total organic carbon, biochemical oxygen demand, chemical oxygen demand, total dissolved solids, total suspended solids, totals solids, total petroleum hydrocarbons, salinity, and acidity.

##### 4.3.1 Written Method

A draft of the analytical method proposed for certification is submitted to USATHAMA for approval with the precertification performance data package.

##### 4.3.2 Standards

Standard Analytical Reference Materials (SARMs), provided by USATHAMA, are used in all method certification analyses. DCL obtains suitable, certified Reference Materials from the EPA or other commercial sources for analytes for which USATHAMA is not able to provide SARMs. Standard water and standard soil are used by DCL for all USATHAMA analyses done.

#### 4.3.3 Standard Water

Standard water samples are prepared by adding a known quantity of target analyte to a known volume of water. The volume of water is specific in the method being performed. All target analytes for the method are added. ASTM Type I grade water is used for inorganic methods. ASTM Type II grade water containing 100 mg/L each of added sulfate and chloride is used for organic methods. The method and reagents used to prepare spiking solutions are specified in the standardized methods.

#### 4.3.4 Standard Soil

Standard soil samples are prepared by adding a known quantity of target analyte to a known weight of selectively blended standard soil as provided by the Chemistry Branch of USATHAMA.

#### 4.3.5 Precertification Calibration

Before initiating method certification, precertification calibration is performed. DCL holds discussions with USATHAMA delineating anticipated environmental concentrations. The concentration range tested includes the Target Reporting Limit (TRL). Additional concentrations of calibration standards may be included for expanding the range of certification. Duplicate analyses are performed on all of the calibration standards.

The certified check standards are obtained from a source other than USATHAMA, whenever possible. In the absence of suitable commercially prepared mixtures, the DCL Quality Control Chemist prepares appropriate mixtures from certified pure stock reagents. The mixtures contain the analyte(s) of interest at concentrations near the high end of the certification range.

The calibration standard data is tabulated and graphed for analysis of Lack of Fit (LOF) and Zero Intercept (ZI), then submitted to USATHAMA for evaluation. The check standard results are required to fall within the acceptability limits defined by the originator.

#### 4.3.6 Certification

Certified methods meet the following conditions: The Target Reporting Limit (TRL) and the range of certification are selected in consultation with USATHAMA. A pre-certification analysis is performed and reported to USATHAMA, with a copy of the analytical method. Upon approval from USATHAMA, a Class 1, Class 1A, Class 1B, or Class 2 certification process is initiated. See Table 2.

Data derived from certification is processed using USATHAMA supplied software, and submitted to USATHAMA for evaluation. The method Certified Reporting Limit (CRL) and certified range are determined from this data evaluation.



Methods certified under previous editions of the USATHAMA Quality Assurance Program and determined by USATHAMA to be valid for current work do not require recertification.

All certification data are properly maintained in archive files.

#### 4.3.7 Method Modifications and Control

Any modifications, additions, or deletions proposed to any USATHAMA-certified method must be submitted to USATHAMA for approval before such a change is made. Following approval, the revised method (with changes plainly noted) shall be distributed to appropriate laboratory personnel as described in DCL SOP-GLP-002, and the old method collected for retirement.

#### 4.4 Analyst Training

An analyst certifying a new method is qualified to perform that method during routine field sample analysis. An analyst who is required to perform on a procedure which has already been certified is required to satisfactorily analyze an appropriate set of quality control samples to demonstrate ability to perform the method. The demonstration sample data must pass current quality control criteria. Successful certification performance is reflected by an addition to the analyst's training file.

The analyst prepares all data records and a data package, as required for field sample analysis data. The data and the data package must be approved by Quality Assurance. The data and data package are maintained in archives.

**Table 2.**  
**NUMBERS AND CONCENTRATIONS OF CALIBRATION STANDARDS**  
**(LINEAR AND ZERO-INTERCEPT)**

PRECERTIFICATION - CLASS 1

Minimum Testing Range (MTR): 12 Standards + 1 Check Standard (SC)  
Blank, \*0.5, 1, 2, 5, & \*10 TRL (Duplicate) + CS  
MTR + 1 Order of Magnitude Extension: 18 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, & \*100 TRL (Duplicate) + CS  
MTR + 2 Orders of Magnitude Extension: 24 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, & \*1000 TRL (Duplicate) + CS

PRECERTIFICATION - CLASS 1A

Minimum Testing Range (MTR): 8 Standards  
Blank, \*0.5, 2, & \*10 TRL (Duplicate)  
MTR + 1 Order of Magnitude Extension: 12 Standards  
Blank, \*0.5, 2, 10, 50, & \*200 TRL (Duplicate)  
MTR + 2 Orders of Magnitude Extension: 16 Standards  
Blank, \*0.5, 2, 10, 50, 200, 500, & \*2000 TRL (Duplicate)

PRECERTIFICATION - CLASS 1B

Minimum Testing Range (MTR): 8 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, & \*10 TRL (Duplicate) + CS  
MTR + 1 Order of Magnitude Extension: 12 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, 10, 50, & \*200 TRL (Duplicate) + CS  
MTR + 2 Orders of Magnitude Extension: 16 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, 10, 50, 200, 500, & \*2000 TRL (Duplicate) + CS

PRECERTIFICATION - CLASS 2  
(Not Required)

INITIAL CALIBRATION - CLASS 1

Minimum Testing Range (MTR): 7 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 1, 2, 5, \*10, & \*10 TRL + CS  
MTR + 1 Order of Magnitude Extension: 10 Standards + 1 Check Standard  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, \*100, & \*100 TRL + CS  
MTR + 2 Orders of Magnitude Extension: 13 Standards + 1 Check Standard  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, \*1000, & \*1000 TRL + CS

\* 10 percent to 25 percent Range Extension



**Table 2**  
**(Continued)**

INITIAL CALIBRATION - CLASS 1A

Minimum Testing Range (MTR): 5 Standards  
Blank, \*0.5, 2, \*10, & \*10 TRL  
MTR + 1 Order of Magnitude Extension: 7 Standards  
Blank, \*0.5, 2, 10, 50, \*200, & \*200 TRL  
MTR + 2 Orders of Magnitude Extension: 9 Standards  
Blank, \*0.5, 2, 10, 50, 200, 500, \*2000, & \*2000 TRL

INITIAL CALIBRATION - CLASS 1B

Minimum Testing Range (MTR): 5 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, \*10, & \*10 TRL + CS  
MTR + 1 Order of Magnitude Extension: 7 Standards + 1 Check Standard  
Blank, \*0.5, 2, 10, 50, \*200, & \*200 TRL + CS  
MTR + 2 Orders of Magnitude Extension: 9 Standards + 1 Check Standard  
Blank, \*0.5, 2, 10, 50, 200, 500, \*2000, & \*2000 TRL + CS

INITIAL CALIBRATION - CLASS 2

Minimum Testing Range (MTR): 6 Standards  
Blank and 1 TRL (Triplicate)

DAILY CALIBRATION - CLASS 1/CLASS 1A/CLASS 1B

Minimum Testing Range (MTR): 2 Standards  
\*10 & \*10 TRL  
MTR + 1 Order of Magnitude Extension: 2 Standards  
\*100 & \*100 TRL  
MTR + 2 Orders of Magnitude Extension: 2 Standards  
\*1000 & \*1000 TRL

DAILY CALIBRATION - CLASS 2

Minimum Testing Range (MTR): 4 Standards  
Blank and 1 TRL (Duplicate)

**Table 2**  
**(Continued)**

CERTIFICATION - CLASS 1

Minimum Testing Range (MTR): 9 Initial, 6 Daily  
MTR + 1 Order of Magnitude Extension: 12 Initial, 6 Daily  
MTR + 2 Orders of Magnitude Extension: 15 Initial, 6 Daily

CERTIFICATION - CLASS 1A

Minimum Testing Range (MTR): 5 Initial  
MTR + 1 Order of Magnitude Extension: 7 Initial  
MTR + 2 Orders of Magnitude Extension: 9 Initial

CERTIFICATION - CLASS 1B

Minimum Testing Range (MTR): 6 Initial, 6 Daily  
MTR + 1 Order of Magnitude Extension: 8 Initial, 6 Daily  
MTR + 2 Orders of Magnitude Extension: 10 Initial, 6 Daily

CERTIFICATION - CLASS 2

Minimum Testing Range (MTR): 6 Initial

INITIAL FIELD SAMPLE LOT - CLASS 1

Minimum Testing Range (MTR): 9 Initial  
MTR + 1 Order of Magnitude Extension: 12 Initial  
MTR + 2 Orders of Magnitude Extension: 15 Initial

INITIAL FIELD SAMPLE LOT - CLASS 1A

Minimum Testing Range (MTR): 5 Initial  
MTR + 1 Order of Magnitude Extension: 7 Initial  
MTR + 2 Orders of Magnitude Extension: 9 Initial



**Table 2**  
**(Continued)**

INITIAL FIELD SAMPLE LOT - CLASS 1B

Minimum Testing Range (MTR): 6 Initial  
MTR + 1 Order of Magnitude Extension: 8 Initial  
MTR + 2 Orders of Magnitude Extension: 10 Initial

INITIAL FIELD SAMPLE LOT - CLASS 2

Minimum Testing Range (MTR): 6 Initial

ADDITIONAL FIELD SAMPLE LOT - CLASS 1/CLASS 1A/CLASS 1B

Minimum Testing Range (MTR): 2 Daily  
MTR + 1 Order of Magnitude Extension: 2 Daily  
MTR + 2 Orders of Magnitude Extension: 2 Daily

ADDITIONAL FIELD SAMPLE LOT - CLASS 2

Minimum Testing Range (MTR): 4 Daily

## 5.0 SAMPLE HANDLING AND ANALYSIS

### 5.1 Sample Management

In most instances, DCL does not perform sample collection, but receives samples from designated field crews. Samples received by DCL are received by designated sample custodians. The protocols of sample management are delineated below.

#### 5.1.1 Sample Containers

As directed by USATHAMA, DCL will supply sample bottles and/or shipping coolers for use in the collection of field samples. A copy of DCL's "Field Sampling Information," to be used as guidance in sampling and in the completion of chains-of-custody, is included in the initial shipment of coolers to the field sampling site. All sample containers shall be cleaned before use according to the protocols specified in Appendix C. Use of commercially cleaned bottles is acceptable provided that cleaning is performed as specified in Appendix C or meets the requirements of the EPA's Contract Laboratory Program.

Generally, for water samples, this includes: septum-sealed glass vials for volatile compounds; amber glass bottles with Teflon-lined lids for organic constituents other than volatiles; and polyethylene bottles for inorganic analytes. Exceptions are noted in the certified method. For soil and sediment samples wide-mouth amber-glass bottles shall be used. Preservatives, as delineated in the DCL USATHAMA Analyte Summary (Appendix B), are provided (as necessary) with sample containers shipped to the field, for proper addition at the site.

#### 5.1.2 Sample Receipt

Samples are received at DCL by the designated Sample Receipt Officer (SRO), or his designee. At the time of receipt of a sample shipment, the sample shipping containers are opened and the samples are inspected. A Sample Receipt Form is initiated at this time. This form includes entries for date and time of receipt, airbill number, a record of the condition of seals on the shipping container and samples, documentation present, temperature and general condition of the shipment, and correlation of sample document and sample labeling information.

Any discrepancies between the samples and the documentation, including missing, broken, or damaged samples, will be reported to USATHAMA or its contractor within 24 hours.

The SRO or his designee signs the field chain-of-custody record at the time that the shipping container is opened. In the case of water samples, which do not usually require splitting, the SRO or his designee opens the shipping container and completes the sample inspection form and field chain-of-custody record. Sufficient copies of the field chain-of-custody record are made to allot one copy for each analytical procedure, plus one for moisture and one as a back-up.



### 5.1.3 Sample Logging

The field chain-of-custody record is used by the Sample Receipt Coordinator (SRC) to initiate sample logging procedures. Initial logging entries include field sample number, date of receipt at DCL, analyses requested, and comments on sample condition at the time of receipt as noted on the Sample Receipt Record. These are recorded in both a computer based log and in a bound logbook. After sample lotting is completed, the USATHAMA sample identification number for each sample and analysis is entered into the logs.

### 5.1.4 Sample Splitting

Following initial sample inspection, the SRC splits the samples into the required number of aliquots (one for each analytical procedure, one for moisture if the sample is a soil, and a large portion for back-up). The SRC properly labels the aliquots with the field sample identification number and the method of analysis, and relinquishes custody of the sample aliquots to the SRC.

### 5.1.5 Sample Lotting and Labeling

The number of samples which can be analyzed by a given method on a single day, as determined by the rate-limiting step in the analytical scheme, is designated as a "lot". The samples in a lot are labeled with a USATHAMA sample identification number consisting of a three letter lot code and individual three number sample designations (e.g. AAA001, AAA002). As split sample aliquots for a particular analytical procedure are received by the SRC, they are given the next alphabetical lot designation in sequence. Samples received and split at various times are grouped together in the same lot such that sample holding times are not jeopardized. The unique sample number is written in black permanent marker on white laboratory labeling tape, which is prominently placed on each sample container.

Quality control (QC) samples are a part of every lot, and are spiked according to the specific method requirements. The QC samples are provided upon request of the analyst.

### 5.1.6 Sample Storage

Samples are stored in a location appropriate to the holding requirements of the requested analytes. Heat-sensitive, light-sensitive, radioactive, or other samples having unusual physical characteristics or requiring special handling, are properly stored and maintained.

## 5.2 Chain-of-Custody

DCL maintains chain-of-custody records for all USATHAMA samples received at the laboratory.

A copy of applicable field chain-of-custody records is maintained with each sample lot. In addition, each lot of samples is maintained under a separate laboratory chain-of-custody record. The chain-of-custody includes unique sample number(s), date and time, source of sample(s), analyses required, signatures of relinquishing and receiving entities, and any other pertinent information. Copies of DCL's field and in-house chains-of-custodies for USATHAMA projects are provided in Appendix D.

### 5.3 Sample Handling Procedures

After samples have been received, split, and lotted, those not requiring extraction procedures are transferred to a central walk-in cold storage area. They are stored in this area until they are scheduled for analysis. Samples not requiring extraction procedures are prepared for analysis, within the required holding times, by the analyst or by a technician working under the direction of the analyst. These samples are usually analyzed within hours after preparation.

Samples which require extraction, distillation, or digestion procedures are prepared for analysis by the appropriate Inorganic or Organic Sample Preparation groups after lotting procedures have been completed. Extracts or distillates are stored in refrigerators in appropriate analytical areas of the laboratory.

The samples and extracts are maintained in their designated lots and under chain-of-custody, at all times. Separate preparation logbooks are maintained by the sample preparation groups to document sample handling.

### 5.4 Toxicity Characteristic Leaching Procedure

Samples which require Toxicity Characteristic Leaching Procedure (TCLP) are split and assigned a unique three-letter lot code. Chains-of-custody for these samples are signed off in the same manner as other samples requiring a USATHAMA-certified analysis. At the same time, chains-of-custody are printed (but not "initiated") for all prospective analyses to be generated from the TCLP leachate(s).

Once the original sample has been satisfactorily leached, both the chain-of-custody and any remaining original sample are transferred to Long Term Storage. The chains-of-custody for all generated leachates are now initiated by TCLP personnel. These leachates (along with their chains-of-custody) are stored and handled as any other USATHAMA samples which have been prepared for analysis.

The chains-of-custody for the original sample and the leachates are cross-referenced to facilitate traceability.

### 5.5 Holding Times

The holding times specified in DCL's USATHAMA Analyte Summary (Appendix B) are adhered to for all USATHAMA samples, extracts, distillates, and digestates.



## 5.6 Sample Analysis

### 5.6.1 Standards

Analytical standards are prepared either from Standard Analytical Reference Materials (SARMs) or Interim Reference Materials (IRM) supplied by USATHAMA, or from standard materials obtained by DCL from the EPA, the National Institute of Standards and Technology (NIST), or other commercial sources. Secondary standard materials may be used when SARM materials are available in only limited quantity. The secondary standards, which must be positively identified with an estimation of purity, are referenced to SARMs and periodically checked against them.

Standard materials procured from commercial sources other than USATHAMA, the Environmental Protection Agency (EPA), or the NIST are considered as "off-the-shelf" materials. The purity and identity of these materials is established from both analysis documentation supplied by the vendor and DCL analytical data. Materials are characterized by two independent methods whenever possible, including, but not limited to IR, GC, GC/MS, HPLC, and other inorganic techniques.

Metals are traceable to NIST, whenever possible. "Off-the-shelf" materials are characterized against EPA or NBS known standards whenever possible. All SARMS are stored in the quality control laboratory, under controlled access conditions. Generally, organic compounds are stored under refrigeration, while metals solutions are stored at room temperature.

### 5.6.2 Solutions

Analytical standard working solutions are normally prepared by the analyst performing the analysis, in accordance with the protocol defined in the approved analytical method. In some analytical procedures, a designated analyst prepares the standards, while other analysts carry out the procedure.

As new or replacement standard solutions are prepared, they are validated against either the previously used standard, a commercially prepared quantitative standard, or a standard prepared by another analyst for the purpose of validation.

Although validation acceptance criteria are established for each analytical method, protocol guidelines for acceptance of a new solution is that it is found, by analysis, to be within  $\pm 5\%$  of the target value. All validations are documented either in the analyst's notebook or in a standards preparation logbook unique to USATHAMA and the analytical area using the standards.

### 5.6.3 Sample Preparation

Soil and water field samples are prepared for analysis according to the protocol defined in the analytical method for the specific analyte(s) being analyzed. Procedures for the preparation of mixed-matrix field samples, such as sediment, sludge, sewer, or lake-bottom samples, are discussed with USATHAMA on a case-by-case basis.



#### 5.6.4 Instrument Calibration

The USATHAMA QA Program delineates, in detail, the requirements for instrument calibration for precertification, full method certification, initial calibration for analysis work, and daily calibration during sample analysis. DCL has implemented these guidelines for all USATHAMA work, as follows. Also see Section 4.3.6 (Certification) for additional details.

Instruments are tuned, as applicable, and the required number and concentrations of standards are analyzed daily with each lot of samples. Calibration criteria are either passed or corrective action is pursued by the analyst. If daily calibration criteria are not met, then initial calibration procedures are instituted to bring the analytical system back into calibration.

#### 5.6.5 Initial Calibration

During initial calibration, a minimum of one blank and five calibration standards (Class 1) or one blank and three calibration standards (Class 1A and Class 1B) that bracket the certification testing range is analyzed singularly on one day. The concentrations of the calibration standards, in the solvent that results from all the preparation steps of the method, take into account any concentration steps that are part of the method. Concentrations in the solvent correspond to those in an environmental matrix as if the method preparation steps had been performed.

In addition to the initial calibration standards, Class 1 and 1B methods require the analysis of calibration check standards (Section 5.6.7). During a Class 1 or Class 1B initial calibration, a calibration check standard is analyzed at the completion of calibration. If the method requires what could be an initial calibration each day analysis is performed, then the calibration check standards are analyzed once a week rather than each day.

If the results of the calibration check standard are not acceptable, immediate reanalysis of the calibration check standard is required. If the results of the reanalysis still exceed the limits of acceptability, the system is considered to have failed calibration. Sample analysis is halted and will not resume until successful completion of initial calibration. Corrective actions taken to restore initial calibration are documented in the analysts' notebook.

#### 5.6.6 Daily Calibration

Calibration standards are analyzed each day to verify that instrument response has not changed from previous calibration. Each day before sample analysis, the highest concentration standard is analyzed. The response must fall within the required percentage or two standard deviations of the mean response for the same concentration, as determined from precertification, certification, and prior initial/daily calibrations. If the response fails this test, the daily standard is reanalyzed. If the response from the second analysis fails this range, initial calibration is performed before analyzing samples.



Each day after sample analyses are completed, the highest concentration standard is analyzed. If the response is not within the required percentage or two standard deviations of the mean response from precertification, certification, and prior initial/daily calibrations, the daily standard shall be reanalyzed. If the response from the second analysis fails the range, the system is considered to have failed calibration. Initial calibration is performed and all samples analyzed since the last acceptable calibration are reanalyzed.

For non-linear or non-zero-intercept calibration curves, daily calibration consists of analysis of the low, middle, and high standards at the beginning of the day. When sample analyses are completed at the end of the day, the low and high standards are analyzed. Instrument responses for each concentration determination must fall within two standard deviations of the mean response, as described previously, for the appropriate standard. For calibrations fitted by the quadratic equation, a minimum of four standards over the certified range are required and the highest level standard analyzed at the end of the day. For all other equations, one more standard than needed to meet the degrees of freedom for any lack-of-fit is required, as a minimum.

#### 5.6.7 Calibration Check Standards

Calibration check standards are required for all Class 1 and 1B methods and are analyzed during precertification and with each initial certification. The calibration check standard contains all analytes of interest for the method in question at a concentration near the upper end of the calibration range. Results of the calibration check standards shall fall within the limits of acceptability as described below:

##### CASE 1.

A certified check standard is available from the EPA or some other source with both the true value and limits of acceptability specified by the supplier. The results must fall within the limits specified by the supplier, or  $\pm 10$  percent for inorganics,  $\pm 25$  percent for organics, whichever is less.

##### CASE 2.

A certified check standard is available from the EPA or some other source with a true value specified but without limits of acceptability. The results must fall within  $\pm 10$  percent for inorganics and within  $\pm 25$  percent for organics.

##### CASE 3.

If no certified check standard is available, the contractor laboratory shall prepare a check standard using a second source of reference material. This standard shall be prepared by a different analyst than the one who prepared the calibration standard. If weighing of the material is required, a different balance should be used, if possible. The results must fall within  $\pm 10$  percent for inorganics and within  $\pm 25$  percent for organics.

##### CASE 4.

If there is only one source of reference material available, then the calibration and calibration check standards must be prepared from the same material. The standards shall be prepared by different analysts. If weighing is required, different balances should be used, if possible. The results must fall within  $\pm 10$  percent for inorganics and within  $\pm 25$  percent for organics.

For all cases listed above, after the seventh acceptable calibration check standard, the limits of acceptability are +/- two standard deviations, as determined from the first seven points.

For multi-analyte methods, the calibration check standard contains all analytes of interest. For the check standard to be deemed acceptable at least 2/3 of the analytes must meet the limits of acceptability as defined above (also see Table 3). In addition, if a single analyte falls outside the limits of acceptability for two consecutive times, then the calibration check standard is deemed unacceptable. If a calibration check standard is not acceptable, the procedures detailed above are followed.

**Table 3.**  
**MINIMUM NUMBER OF IN-CONTROL POINTS**  
**FOR MULTI-ANALYTE METHODS**

| <u>Required Control</u><br><u>Analytes Per Method</u> | <u>Required Number of</u><br><u>Data Values Falling</u><br><u>Between the UCL and LCL</u> |
|-------------------------------------------------------|-------------------------------------------------------------------------------------------|
| 1                                                     | 1                                                                                         |
| 2                                                     | 2                                                                                         |
| 3                                                     | 2                                                                                         |
| 4                                                     | 3                                                                                         |
| 5                                                     | 4                                                                                         |
| 6                                                     | 4                                                                                         |
| 7                                                     | 5                                                                                         |
| 8                                                     | 6                                                                                         |
| 9                                                     | 6                                                                                         |
| 10                                                    | 7                                                                                         |
| 11                                                    | 8                                                                                         |
| 12                                                    | 8                                                                                         |
| 13                                                    | 9                                                                                         |
| 14                                                    | 10                                                                                        |
| 15                                                    | 10                                                                                        |
| 16                                                    | 11                                                                                        |
| 17                                                    | 12                                                                                        |
| 18                                                    | 12                                                                                        |
| 19                                                    | 13                                                                                        |
| 20                                                    | 14                                                                                        |
| 21                                                    | 14                                                                                        |
| 22                                                    | 15                                                                                        |
| 23                                                    | 16                                                                                        |
| 24                                                    | 16                                                                                        |
| 25                                                    | 17                                                                                        |

#### 5.6.8 Analytical Procedures

All field samples are analyzed according to approved, laboratory certified USATHAMA analytical methods. All deviations shall be approved by USATHAMA prior to implementation. These deviations are also documented in the analyst's notebook.



#### 5.6.9 Second-Column Confirmation

In several GC and HPLC methods (e.g., organochlorine pesticides and explosives), the presence of compounds is routinely confirmed on a second column. The confirmation is usually performed on the basis of a Class 2 certification. Confirmation does not necessarily have to be performed within holding times, but must be accomplished within ten (10) days of sample analysis.

### 5.7 Data Handling

Although the primary emphasis of the USATHAMA QA Program is the control of sample analysis and the handling of data, record keeping maintains its importance in the overall assessment of the production of quality of data and is used in part to document the control of sample analysis. The degree of rigor used in documenting sampling and analysis activities cannot be understated. All activities require extensive documentation and special handling protocols. All activities are to be performed under chain-of-custody procedures. Particularly in these situations, the attitude is: "If you didn't write it down, you didn't do it."

For most USATHAMA projects, this degree of documentation is required. For some projects, documentation in the form of an EPA CLP package is required. In any case, the records described in this Quality Assurance document shall be maintained and will be available for inspection by USATHAMA.

#### 5.7.1 Data Reduction

Generally, data have been collected during the analysis of samples either into computer based data files or onto hard copy sheets, which, in turn, are either machine generated or hand written. All of the data are eventually compiled in computer files. The data pertaining to analytical standards are either compared to the most recent initial calibration curve, in the case of a daily calibration, or used to generate new initial calibration curves, in accordance with those generated during pre-certification. The appropriate standard curve is used to evaluate the field sample data to determine the amount of analyte present. Finally, all of the computer generated calculations are generated as hard copy output.

#### 5.7.2 Data Validation

Initial data validation is accomplished during data collection through the use of quality control samples and calibration check standards. Errors detected through a review of these monitors by Quality Assurance during analysis are corrected during the data collection phase of the analysis. Only analytically valid data are processed further.

Following an analyst's computer-based reduction of data and production of a numerical results report, the entire assemblage of data is given to a peer analyst for review and validation. The peer analyst checks that the analytical method was followed, that there are no errors in the transcription of data, that the best-fit curve was used, and that the numerical report of data contains no calculation or transcription errors.

The data package is then reviewed by the appropriate Group Leader or Section Manager. The data report is particularly scrutinized to assure that all reported data values are in the proper range or have dilution factors, that the method has been carefully followed, that instrumentation was properly tuned or calibrated, and that the instrumental data was properly interpreted. A general review of the data package is also made to assure that all required documentation is present.

The final step in data validation is the review by Quality Assurance. The content of each data package is closely checked for errors or omissions that would negatively impact on the admissibility of the data in litigation proceedings. Corrective action is initiated and documented as outlined in section 10.0.

### 5.7.3 Data Reporting

The results for samples analyzed for USATHAMA projects are entered into the USATHAMA-provided software program (IRDMS). Data created using the IRDMS can then be electronically transmitted to USATHAMA Via Potomac Research Inc. (PRI), or a diskette together with hard copy printouts can be submitted.

Data is entered on a coding form by the analyst, which is verified by the peer checker and, group leader/section manager. QA personnel review data for obvious errors. These data are encoded onto a diskette, checked through two USATHAMA software routines, then printed out and verified by visual inspection by a Data Entry Specialist. Verified analytical results are then submitted to USATHAMA. DCL retains a copy diskette of all data submitted.

All information pertaining to the analysis of a lot of samples is collected into a data package at the completion of analysis. The contents of data packages varies with methods of analysis. The package is reviewed by Quality Assurance to eliminate technical errors that might affect the litigation quality of the data. The reported data is also reviewed by Data Entry for completeness before release.

All data packages are archived at DCL until a task or delivery order at a particular installation is complete. At that time, all pertinent documentation filed in appropriately-labeled boxes is delivered either to USATHAMA directly, or to the prime contractor responsible for final review of the data packages. In the second case, the prime contractor is responsible for the delivery of DCL data boxes to USATHAMA.



## 6.0 ANALYTICAL SYSTEM CONTROLS

### 6.1 Sample Control

As discussed in the section of this QA Plan on Sample Management, DCL is not generally responsible for the collection of samples from sites in the field. However, DCL efforts in sample control may extend into field sample collection. As directed by USATHAMA or the prime contractor, DCL provides proper sample collection bottles, sample preservatives, labeling material, sample shipping containers (coolers), and technical assistance to field sample collection crews. DCL also works in concert with USATHAMA or the prime contractor on sample shipping and receiving.

Samples received at DCL are under the control of Sample Receipt personnel from receipt at the lab to acceptance by an analyst for extraction or preparation. Samples are not released for processing until all documentation is completed and the samples are properly lotted and labeled. Holding times are closely monitored by the analysts, Sample Receipt and laboratory management.

DCL Project Managers communicate regularly with USATHAMA and/or other involved prime contractors to alleviate sample shipping, holding time, and analysis difficulties.

### 6.2 Document Control

Document control is primarily the responsibility of Quality Assurance. Sample documents generated in the field during sample collection and shipping are maintained in QA files. Laboratory chain-of-custody records, sample receipt and tracking records, data reporting forms and analysis data packages, and corrective action records are maintained by Quality Assurance. On a schedule determined by contract requirements, QA also archives or otherwise controls all bound notebooks and logbooks containing data pertinent to USATHAMA work.

### 6.3 Quality Control Samples

Quality control chemists within the Quality Assurance Section of DCL prepare most of the quality control samples required during sample analysis. These samples are prepared from USATHAMA-supplied SARM and IRM stocks, and other reference materials. Other reference materials include EPA, and NIST standard materials, and "off-the-shelf" materials. "Off-the-shelf" materials are analyzed by DCL, with positive identification and estimate of purity, with EPA standard reference materials, where possible, using at least two different methods.

Quality control stock and dilute working solutions are prepared and maintained separately from those used by analysts as standards. Exceptions to this procedure are made only when primary stock material is in very short supply, or when the primary solution is unstable. In these cases, the same primary solution is used to prepare separate dilute working solutions. Samples are prepared in accordance with parameters defined in each analytical method. These parameters include the control analytes, the concentration levels at which the analytes should be spiked, control sample matrix, spike equilibration time, and procedures for preparation of the sample for analysis.

Quality control samples which are not regularly prepared by the quality control chemists include surrogate spiking solutions and spiked samples required in the GC/MS methods for volatile and semi-volatile organic compounds. These surrogate preparations are handled by the GC/MS Group and the Extraction Group, respectively.

Quality control samples are included in every lot of USATHAMA samples, as required in the USATHAMA QA Program and specified in each certified analytical method. The control samples are processed through the entire analytical method and quantitated on the same calibration curve as the field samples. The results for the quality control samples are evaluated first by the analyst, and then by Quality Assurance, to determine their acceptability.

Calibration check standards are prepared by someone other than the person preparing the standards. Calibration check standards are analyzed at the time of an initial calibration, or once per week when routine initial calibrations replace daily calibrations. The analysis results must meet the criteria established by their originator.

#### 6.4 Control Charts

For Class 1, Class 1A, and Class 1B certified methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and to detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts is not adjusted for accuracy. Control charts are not used with Class 2 certified methods.

Control charts include the analyte, method number, DCL laboratory code of UB, spike concentration, and chart title. All data presented on a control chart are also presented in tabular form. The following charts may be selected from the USATHAMA-supplied computer control chart program:

1. Single-Day X-Bar Control Chart (High Spike Conc.)
2. Single-Day Range Control Chart (High Spike Conc.)
3. Three-Day X-Bar Control Chart (Low Spike Conc.)
4. Three-Day Range Control Chart (Low Spike Conc.)



In addition, the following information is also included on each control chart:

- Three-letter lot designation for each point, shown on the x-axis;
- Percent recovery (for X-bar control charts), or range (for R control charts) along the y-axis;
- Upper control limit (UCL);
- Upper warning limit (UWL);
- Mean;
- Lower warning limit (LWL), on X-bar charts; and
- Lower control limit (LCL), on X-bar charts.

For some analytes specified by USATHAMA, warning limits on X-bar charts are deleted and replaced by modified control limits based upon data quality specifications.

#### 6.4.1 Control Chart Plotting: Single-Day

The initial control chart is prepared using the four days of certification data closest to the spiking concentration used during analysis. The average (X-bar), average range (R), and control limits for both are updated after each in-control lot for the first 20 lots. Limits established after lot 20 are used for the next 20 lots. Control charts are updated after each 20 lots thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (1-20), the limits established from the previous lots are used to control the current lot.

When modified limits are established, data for samples are accepted if the control data fall between the modified limits. If modified limits have not been established, data for samples are accepted, based upon the recoveries established during certification and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control chart limits. Out-of-control or outlier points are plotted; however, such lots are not utilized in lot number requirements or control chart calculations.

All recoveries are plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual recovery measurement value is tested as an outlier using Dixon's Test at the 98% confidence level. If the datum is not classified as an outlier by the test, the point is included in updating the control chart limits. If the datum is classified as an outlier, it is not used in updating the control chart limits. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits are recalculated using only in-control data points. The control limits are then drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) are dropped from the calculations (but left on the charts) and the control limits recalculated using only points between those limits. This practice of dropping points and recalculating limits is performed only once, at the initialization of stable limits. Charts are then updated with newly calculated control limits and all points plotted.



#### 6.4.2 Three-Point Moving Average

Analytical data for analytes prepared in the single low concentration QC sample are plotted and evaluated on a three-day-moving-average control chart. Data for the surrogates spiked in a standard matrix and used in GC/MS analyses are also charted on a three-day-moving-average control chart. Plotting criteria for the three-point moving average control charts are similar to those described above (Section 6.4.1) for single-day control charts. Data for analytes prepared in duplicate QC samples at high concentrations are plotted and evaluated on single-day control charts.

Computer generated control charts maintained by Quality Assurance are updated and printed weekly, while analysts plot data points by hand as sample lots are analyzed. This allows for both computer maintenance and evaluation of a large data base with software calculation of control limits, and immediate daily surveillance of analytical trends.

#### 6.5 Out-of-Control Conditions

Results of the analysis of quality control samples are reported to QA within 48 hours of completion through the analyst's submission of a Preliminary QC Report.

The analyst quantifies each analyte in the method blank and spiked QC sample each day of analysis. Processing of additional lots will not occur until the results of the previous lots have been calculated, plotted on control charts as required, and the entire analytical method shown to be in control.

An indication of an out-of-control situation may include: A value outside the control limits or classified as outlier by statistical test; A series of seven successive points on the same side of the mean; A series of five successive points going in the same direction; A cyclical pattern of control values, or; Two consecutive points between the UWL and UCL or the LWL and LCL.

If the points for at least two-thirds of the control analytes for a multi-analyte method are classified as in-control, the method is in control and environmental sample data may be reported. A method may be deemed out-of-control even if greater than or equal to 2/3 of the control analytes meet control criteria. Of the remaining control analytes (less than 1/3 possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is deemed out-of-control. If data points for fewer than 2/3 of the control analytes are classified as in control, the method is considered to be out-of-control and all work on that method must cease immediately. No data for environmental samples in that lot may be reported.

In all cases, investigation by the analyst and the Quality Assurance Coordinator is required to determine the cause of the condition and to decide on appropriate corrective action. The pertinent details of the situation and the corrective action taken are fully documented in a Corrective Action Report (CAR). (See also section 10.0.) Field sample data effected by the situation are evaluated and reanalyzed as necessary.



When a method is determined to be out of control, the analysis of field samples by that method is suspended. Corrective action must be documented and the method must be demonstrated to be in control before analysis of field samples is reinstated. Analytical control is demonstrated through the acceptable analysis of an appropriate set of QA samples.

## **7.0 PREVENTATIVE MAINTENANCE**

All analytical instrumentation used at DCL is maintained to provide consistent, high-quality performance. Most instruments are maintained by the manufacturer, under contract. Each instrument is labeled with a unique number and instrument information peculiar to USATHAMA requirements. Instrument service records and maintenance calibrations are maintained by the appropriate section and in a logbook unique for each instrument.

The primary objective of the instrument maintenance program is to assure the quality of the analytical data generated by the instrument. While there are analytical systems which require absolute calibration, such as balances, the majority of analytical systems used by DCL for the analysis of USATHAMA samples are calibrated at the time of use by the analyst. This is accomplished through generation of a chemical calibration curve, based upon instrument response verses analyte concentration. This curve is used to evaluate field sample data through instrument responses.

Major instrument systems which are calibrated on an "as used" basis are maintained under either an "on call" or a preventative maintenance contract with the manufacturer. Preventative maintenance is scheduled in each instrument contract. When an instrument cannot perform to specifications and DCL technicians cannot return it to specification, a contracted repair service (usually the manufacturer) is called.

Instrument systems which must maintain an absolute calibration, such as analytical balances, are serviced under contract with the manufacturer, usually on an annual basis. Balances are also checked, on at least a weekly basis, for accuracy by Quality Assurance, using NIST-traceable weights. Temperatures of freezers, refrigerators, and walk-in coolers are recorded every working day by QA. When temperatures are noted outside the acceptable range, appropriate personnel are notified for correction. Ovens are calibrated and their temperatures maintained regularly by the appropriate section personnel.



## 8.0 RECORDKEEPING

### 8.1 Laboratory Notebooks

Bound, sequentially-numbered laboratory notebooks with pre-numbered pages are utilized by all analysts for analytical recordkeeping. Notebooks are generally issued to and used by an individual analyst. Any loose sheets of data which must be included in a notebook are securely taped into the notebook and signed and dated across the edges, halfway on the inserted sheet and halfway on the notebook page. Each data page is signed and dated by the analyst entering data on that page, as well as reviewed, signed, and dated by a witness. All entries are required to be in black ink. Corrections are made by a single strikeout, which is dated and initialed.

### 8.2 Logbooks

#### 8.2.1 General

Individual logbook entries are signed and dated by the analyst or technician making the entry. These notebooks include, for example, instrument use and maintenance/calibration logs, pH logs, sample moisture determination logs, and sample receipt logs.

Recordkeeping for sample receipt is discussed under the Sample Management Section 5.1.

#### 8.2.2 Standards

A bound logbook is maintained for all analytical reference materials used for USATHAMA work. The record includes the date of receipt, preparer, source, purity, composition, storage requirements, and expiration date, if applicable. Characterization data for purchased reference material is also included.

The preparation of working standards from reference materials is recorded in a bound logbook. This logbook may be of general use by several analysts for USATHAMA standards preparation, or an individual analyst's notebook, as for preparation of standards used for a single analytical run associated with a single lot of samples.

#### 8.2.3 Instrument

Instrument maintenance records and, where applicable, instrument tuning and calibration data, are maintained in instrument specific logbooks. Actual analytical conditions pertaining to an individual lot analysis are recorded in the analyst's notebook, along with other pertinent analytical information.

### 8.3 Hard-Copy Output

Hard-copy output, (e.g., chromatograms and computer generated data evaluations) is labeled with date, time (where applicable), analytical method, sample numbers, the name or initials of the analyst generating the output, and other pertinent information. Storage of hard-copy output is with related analytical data pertaining to an individual lot analysis. All such data, comprising a complete record of an analysis, are compiled into one or more envelopes for archiving. The envelopes are properly labeled with the lot designation, method of analysis, matrix, analyst, analyst's notebook, and date of completion. When samples from multiple sites or projects are grouped together in a single lot, the data pertaining to each site are compiled (or copied) and stored separately, as directed by USATHAMA. All copies indicate the location of the original data.

### 8.4 Data Package Preparation

In general, all data should be maintained in two separate locations, the data package and the laboratory notebook(s).

Records to be contained in the data package should include, but are not limited to the following:

- Optimized instrumental conditions
- Original chromatograms, strip charts, and/or other instrument output
- Original chain-of-custody form and carrier transmittal documents
- All hardcopy GC/MS outputs
- Expanded scale blow-up of manually integrated peak(s).
- All data sheets or other pre-printed forms used by the contractor or laboratory.
- Copies of all relevant notebook pages. This should include preparation of standards, calibration, sample preparation/extraction, moisture determinations, calculations, and any other relevant comments.

Each data package should contain all information related to one lot for one installation. In cases where a lot has samples from more than one installation, then the information should be copied and placed in separate packages for each installation. In those packages which receive copies, the location of the original material should be identified.

Each data package should contain a contents and approval checklist. This should identify all materials which must be placed into the data package. This list should also list reviewer's names, dates of review, provide space for comments, notes, and corrective actions.



It is the responsibility of the contractor laboratory to review data packages for both content and correctness.

Included in the data package should be a discussion on the observations on the data contained in that data package. This discussion shall include, but not be limited to, observed matrix effects, blank results, control problems, deviations from approved SOPs, digressions from normal practices (i.e., manual integrations) and reasons thereof, etc. The impact on the usability of the data shall be discussed. Explanations on the use of the applicable flagging codes shall be provided.

A detailed SOP is currently in development at DCL.

## **9.0 AUDITS**

DCL facilities are always available for any required audits, announced or unannounced, by USATHAMA representatives.

The DCL Quality Assurance Coordinator conducts internal audits of critical functions within the laboratory, including verification that record keeping procedures are adequate, verification that general good laboratory practices, analytical methods and standard operating procedures are being followed, and continual assessment of quality control sample results. A summary of such audits is available for review at the laboratory. Internal audits shall be conducted by DCL QA personnel at a minimum rate of twice per month.



## 10.0 CORRECTIVE ACTION

When, as a result of audit procedures or the analysis of quality control samples, the analytical or other laboratory systems are found to be unsatisfactory, a corrective action is initiated. The unsatisfactory situation may be either immediate or long term in nature. Immediate short term problems may include unsatisfactory performance on quality control samples (which may be more involved than simply out-of-control data), errors or omissions in the compilation of the data package, or other problems peculiar to a single lot of samples. Long-term problems include trends or cycles in quality control sample analysis data, standard and solution preparation control, staff training in analytical and quality control procedures, or other problems which affect several analytical methods or multiple lots of samples.

To enhance the timeliness of corrective action and thereby reduce the generation of unacceptable data, problems identified by assessment procedures are resolved at the lowest possible management level. Problems that cannot be resolved at this level are reported to the Quality Assurance Coordinator (QAC) for resolution. The QAC determines the management level at which the problem can best be resolved, and notifies the appropriate manager. Weekly progress reports detail all problems and subsequent resolutions.

Steps included in the corrective action system include:

1. Defining the problem;
2. Assigning responsibility for problem investigation;
3. Investigating and determining the cause of the problem;
4. Assigning responsibility for problem resolution; and
5. Verifying that the resolution has corrected the problem.

Problems requiring corrective action may not be easy to identify or define. The situation may not be producing out-of-control data, but simply producing data not of the quality desired. The project manager, section managers, analysts, and the quality assurance staff combine efforts in solving long-term unsatisfactory situations.

All corrective actions are documented by Quality Assurance. Final corrective action reports, which relate to a particular lot analysis, are included in the data package for that lot.

## 11.0 QUALITY CONTROL REPORTS

DCL provides weekly quality assurance evaluation reports to USATHAMA, in conjunction with weekly interim technical reports from project management. The QA reports include charts and tables of quality control data, a control chart checklist delineating contracts and lots, and copies of Corrective Action Reports (CARs). These CARs include explanations of analytical or quality control problems and discussions of the corrective actions taken to alleviate those problems. Observations of data trends or situations which could develop into problems are also discussed in this report, as well as preliminary acceptance or rejection of analytical data.



# APPENDIX A

## APPENDIX A

### LACK OF FIT AND ZERO INTERCEPT TESTS

#### B.1 LACK OF FIT TEST FOR CALIBRATION CURVES AND CERTIFICATION DATA

For most routinely used analytical systems, the instrument response is assumed to be a linear function of analyte concentration. The linear model can be tested by analyzing standards that have been prepared in replicate at each concentration. In addition to the calibration data (target versus instrument response), certification data (target versus found) is also subjected to the Lack of Fit (LOF) test. The usual method of least squares fitting assumes no error in the concentrations of standards.

There are two distinct linear first-order regression models that are generally encountered in analytical calibration. The non-zero intercept model is the most familiar, given by:

$$Y = Y_0 + bX$$

where:

$Y$  = Dependent Variable (Instrument Response or Found Concentration);

$Y_0$  = Y Axis Intercept;

$b$  = Slope of the Line; and

$X$  = Target Concentration.

The estimates  $Y_0$  and  $b$  are calculated to minimize the Sum of Squares (SS) of the deviations from the line without restrictions. For some analyses, however, theory predicts that the response of the instrument should be linear with concentration and should also be zero when there is no analyte present. Thus, if the instrument has been calibrated correctly, the calculated line should pass through the origin by definition. The proper regression model would then be the Zero Intercept model:



$$\hat{Y} = b_0 X$$

where:

$\hat{Y}$  = Predicted Value of Dependent Variable;

$b_0$  = Slope of Line Through Origin; and

$X$  = Target Concentration.

The estimate of  $b_0$  is calculated to minimize the SS of deviations from the line with the restriction that the line must pass through the origin.

For the model with an intercept:

$$b = \frac{N \sum X_i Y_i - \sum X_i \sum Y_i}{N \sum X_i^2 - (\sum X_i)^2}$$

$$Y_0 = \frac{\sum Y_i - b \sum X_i}{N}$$

For the model through the origin:

$$b_0 = \frac{\sum X_i Y_i}{\sum X_i^2}$$

$$Y_0 = 0$$

where:

$N$  = Number of Data Points;

$X_i$  =  $i$ -th Target Concentration; and

$Y_i$  =  $i$ -th Value of Dependent Variable.

The correlation coefficient is a measure of the relationship between two independent variables. In calibration and certification problems, it is assumed that a definite functional relationship exists between the dependent (response or found concentration) and independent (target concentration) variables. Therefore, the correlation coefficient is an insensitive tool for evaluating the quality of the fitted equation.

A more sensitive tool for evaluating the fitted equation is a regression analysis, in which the sources of variation are fractionated into the SS attributable to regression and the SS for residuals. When replicate measurements have been made, the residual SS can be separated into a systematic error component and a random error component. The SS due to systematic error is designated the SS due to LOF because it arises from the inadequacy of the fitted regression model to describe the experimental points.

For the model with intercept, the equation for calculating the SS of residuals is:

$$SS \text{ Residual} = \left[ \sum Y^2 - \frac{(\sum Y)^2}{N} \right] - b^2 \left[ \sum X^2 - \frac{(\sum X)^2}{N} \right]$$

where:

Y = Values of Dependent Variable;

X = Target Concentration;

N = Total Number of Measurements; and

b = Slope of Best Fit Line.

The number of degrees of freedom (df) is N - 2, because two regression coefficients were fitted (slope and Y-axis intercept).

The SS for random error is independent of the regression model employed, depending only on the distribution of replicates around the mean at each concentration. When duplicate measurements have been acquired at each concentration, the SS for random error is given by:

$$SS \text{ Random Error} = \frac{\sum d^2}{2}$$

where:

d = Difference in Values for Each Set of Duplicates.

The total df in this error estimate would be equal to the number of duplicates sets because each would contribute 1 df (2 - 1 = 1). When more than two replicates measurements are made, the SS random error for each set is given by:

$$SS \text{ Random Error} = \sum Y^2 - \frac{(\sum Y)^2}{n}$$

where:

n = Number of Replicates in Each Set. (df is n - 1).



Both the SS random error and the df are then summed across all sets to get the total SS random error and the total df.

After the total SS random error has been calculated, the SS for LOF can be obtained by difference according to:

$$SS\ LOF = (SS\ Residual) - (Total\ SS\ Random\ Error)$$

Similarly, the df associated with LOF is given by:

$$df\ LOF = (df\ Residual) - (df\ Total\ Random\ Error)$$

Regression analysis tables are used to determine whether the data fit the linear models and which linear model is more appropriate. The tables are calculated as shown in Table A-1. For calibration curves and certification data, the replicate analyses of the blank (zero concentration) are not used to obtain regression equations.

After calculating the regression analysis table, the F-ratio for LOF is compared to an F Table (Table A-2) to determine if the regression model is an adequate description of the data. The df LOF is used as  $v_1$ , df random error for  $v_2$ , and 95 percent confidence level. If the calculated F-ratio exceeds the value in the table, there is statistically significant LOF and the data are not linear.

The nature of this test is such that large random error will mask nonlinearity in the data. Very small random error can cause very small (and possibly unimportant) nonlinearity to be found significant (e.g., significant LOF). In fact, when random error is large (or very small), it is difficult to detect systematic variations that might cause LOF.

Table A.1. Regression Analysis Table for Model with Intercept

| Source of Variation                                              | Sum of Square (SS)                                                                                    | Degrees of Freedom (df)               | Mean Square (MS)                                      | F-Ratio                                       |
|------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------|---------------------------------------|-------------------------------------------------------|-----------------------------------------------|
| Residual                                                         | $\left[ \sum Y^2 - \frac{(\sum Y)^2}{N} \right] - b^2 \left[ \sum X^2 - \frac{(\sum X)^2}{N} \right]$ | N-2                                   | $\frac{\text{Residual SS}}{N-2}$                      | -                                             |
| Individual Error<br>(for each set of data at each concentration) | $\sum Y^2 - \frac{(\sum Y)^2}{n}$<br>(for duplicates -- $\frac{nd^2}{2}$ )                            | n-1                                   | -                                                     | -                                             |
| Total Error                                                      | $\sum \text{Individual Error SS}$                                                                     | $\sum \text{df for Individual Error}$ | $\frac{\text{Total Error SS}}{\text{df Total Error}}$ | -                                             |
| Lack of Fit (LOF)                                                | Residual SS - Total Error SS                                                                          | df Residual - df Total Error          | $\frac{\text{LOF SS}}{\text{df LOF}}$                 | $\frac{\text{MS LOF}}{\text{MS Total Error}}$ |

where  
Y = Values for Dependent Variable  
X = Target Concentration  
N = Total Number of Measurement  
n = Number of Replicates at each Concentration  
d = Difference between Duplicates

Do not round off intermediate numbers in calculations. Carry through at least six digits to avoid rounding off errors, even though in the final results less than six digits will be significant.



Table A.2. F-Ratio Critical Values (From Scheffe, 1959)

| THE ANALYSIS OF VARIANCE                                   |      |      |      |      |      |      |      |      |      |  |
|------------------------------------------------------------|------|------|------|------|------|------|------|------|------|--|
| UPPER $\alpha$ POINT* OF $F$ WITH $\nu_1$ AND $\nu_2$ D.F. |      |      |      |      |      |      |      |      |      |  |
| $\alpha = 0.05$                                            |      |      |      |      |      |      |      |      |      |  |
| $\nu_2 \backslash \nu_1$                                   | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    |  |
| 1                                                          | 161  | 200  | 216  | 225  | 230  | 234  | 237  | 239  | 241  |  |
| 2                                                          | 18.5 | 19.0 | 19.2 | 19.2 | 19.3 | 19.3 | 19.4 | 19.4 | 19.4 |  |
| 3                                                          | 10.1 | 9.55 | 9.28 | 9.12 | 9.01 | 8.94 | 8.89 | 8.85 | 8.81 |  |
| 4                                                          | 7.71 | 6.94 | 6.59 | 6.39 | 6.26 | 6.16 | 6.09 | 6.04 | 6.00 |  |
| 5                                                          | 6.61 | 5.79 | 5.41 | 5.19 | 5.05 | 4.95 | 4.88 | 4.82 | 4.77 |  |
| 6                                                          | 5.99 | 5.14 | 4.76 | 4.53 | 4.39 | 4.28 | 4.21 | 4.15 | 4.10 |  |
| 7                                                          | 5.59 | 4.74 | 4.35 | 4.12 | 3.97 | 3.87 | 3.79 | 3.73 | 3.68 |  |
| 8                                                          | 5.32 | 4.46 | 4.07 | 3.84 | 3.69 | 3.58 | 3.50 | 3.44 | 3.39 |  |
| 9                                                          | 5.12 | 4.26 | 3.86 | 3.63 | 3.48 | 3.37 | 3.29 | 3.23 | 3.18 |  |
| 10                                                         | 4.96 | 4.10 | 3.71 | 3.48 | 3.33 | 3.22 | 3.14 | 3.07 | 3.02 |  |
| 11                                                         | 4.84 | 3.98 | 3.59 | 3.36 | 3.20 | 3.09 | 3.01 | 2.95 | 2.90 |  |
| 12                                                         | 4.75 | 3.89 | 3.49 | 3.26 | 3.11 | 3.00 | 2.91 | 2.85 | 2.80 |  |
| 13                                                         | 4.67 | 3.81 | 3.41 | 3.18 | 3.03 | 2.92 | 2.83 | 2.77 | 2.71 |  |
| 14                                                         | 4.60 | 3.74 | 3.34 | 3.11 | 2.96 | 2.85 | 2.76 | 2.70 | 2.65 |  |
| 15                                                         | 4.54 | 3.68 | 3.29 | 3.06 | 2.90 | 2.79 | 2.71 | 2.64 | 2.59 |  |
| 16                                                         | 4.49 | 3.63 | 3.24 | 3.01 | 2.85 | 2.74 | 2.66 | 2.59 | 2.54 |  |
| 17                                                         | 4.45 | 3.59 | 3.20 | 2.96 | 2.81 | 2.70 | 2.61 | 2.55 | 2.49 |  |
| 18                                                         | 4.41 | 3.55 | 3.16 | 2.93 | 2.77 | 2.66 | 2.58 | 2.51 | 2.46 |  |
| 19                                                         | 4.38 | 3.52 | 3.13 | 2.90 | 2.74 | 2.63 | 2.54 | 2.48 | 2.42 |  |
| 20                                                         | 4.35 | 3.49 | 3.10 | 2.87 | 2.71 | 2.60 | 2.51 | 2.45 | 2.39 |  |
| 21                                                         | 4.32 | 3.47 | 3.07 | 2.84 | 2.68 | 2.57 | 2.49 | 2.42 | 2.37 |  |
| 22                                                         | 4.30 | 3.44 | 3.05 | 2.82 | 2.66 | 2.55 | 2.46 | 2.40 | 2.34 |  |
| 23                                                         | 4.28 | 3.42 | 3.03 | 2.80 | 2.64 | 2.53 | 2.44 | 2.37 | 2.32 |  |
| 24                                                         | 4.26 | 3.40 | 3.01 | 2.78 | 2.62 | 2.51 | 2.42 | 2.36 | 2.30 |  |
| 25                                                         | 4.24 | 3.39 | 2.99 | 2.76 | 2.60 | 2.49 | 2.40 | 2.34 | 2.28 |  |
| 26                                                         | 4.23 | 3.37 | 2.98 | 2.74 | 2.59 | 2.47 | 2.39 | 2.32 | 2.27 |  |
| 27                                                         | 4.21 | 3.35 | 2.96 | 2.73 | 2.57 | 2.46 | 2.37 | 2.31 | 2.25 |  |
| 28                                                         | 4.20 | 3.34 | 2.95 | 2.71 | 2.56 | 2.45 | 2.36 | 2.29 | 2.24 |  |
| 29                                                         | 4.18 | 3.33 | 2.93 | 2.70 | 2.55 | 2.43 | 2.35 | 2.28 | 2.22 |  |
| 30                                                         | 4.17 | 3.32 | 2.92 | 2.69 | 2.53 | 2.42 | 2.33 | 2.27 | 2.21 |  |
| 40                                                         | 4.08 | 3.23 | 2.84 | 2.61 | 2.45 | 2.34 | 2.25 | 2.18 | 2.12 |  |
| 60                                                         | 4.00 | 3.15 | 2.76 | 2.53 | 2.37 | 2.25 | 2.17 | 2.10 | 2.04 |  |
| 120                                                        | 3.92 | 3.07 | 2.68 | 2.45 | 2.29 | 2.17 | 2.09 | 2.02 | 1.96 |  |
| $\infty$                                                   | 3.84 | 3.00 | 2.60 | 2.37 | 2.21 | 2.10 | 2.01 | 1.94 | 1.88 |  |

\* Rounded off to three significant figures from tables of M. Merrington and C. M. Thompson in *Biometrika*, Vol. 33, pp. 78-87, 1943. Reproduced with the kind permission of the authors and the editor.

## A.2 ZERO INTERCEPT TEST FOR CALIBRATION CURVES AND CERTIFICATION DATA

If the linear model with intercept is acceptable, the intercept must be tested to determine if it is significantly different from zero. The expression for calculating the slope of the line through the origin is:

$$b_1 = \frac{\sum X_i Y_i}{\sum X_i^2}$$

Before testing the hypothesis that the intercept is zero, a regression analysis table is constructed (Table A-3). If the LOF for the model through the origin is not statistically significant, the Zero Intercept hypothesis is tested using the differences between the residual SS for the intercept and origin models.

To test the hypothesis that the intercept does not differ significantly from zero, calculate:

$$F = \frac{\frac{\text{SS Residual for Zero Intercept Model} - \text{SS Residual of Model with Intercept}}{\text{MS Residual of Model with Intercept}}}{1}$$

The df in the numerator will always be 1 because  $(N - 1) - (N - 2) = 1$  and, therefore, the difference in these SS are divided by 1 to get the MS. The df in the denominator is  $N - 2$ .

The calculated F-ratio is compared to the critical values of F in Table A-2, at  $v_1 = 1$  and  $v_2 = N - 2$ . If the calculated F-ratio is less than the critical value, the Zero Intercept model is accepted.

Generally, certification data will be expected to have intercepts not statistically different from zero. The procedures for daily calibration assume that the zero intercept model can be accepted. If intercepts are statistically different from zero, more rigorous calibration controls will be required and will be specified on a case-by-case basis in the project QC plan.



Table A.3. Regression Analysis Table for Model Through the Origin

| Source of Variation                                                    | Sum of Square (SS)                                                                   | Degrees of Freedom (df)                  | Mean Square (MS)                                      | F-Ratio                                       |
|------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------------------------------|-------------------------------------------------------|-----------------------------------------------|
| Residual                                                               | $\Sigma Y^2 - \frac{(\Sigma XY)^2}{\Sigma X^2}$                                      | N-1                                      | $\frac{\text{Residual SS}}{N-1}$                      | -                                             |
| Individual Error<br>(for each set of<br>data at each<br>concentration) | $\Sigma Y^2 - \frac{(\Sigma Y)^2}{n}$<br>(for duplicates -- $\frac{\Sigma d^2}{2}$ ) | n-1                                      | -                                                     | -                                             |
| Total Error                                                            | $\Sigma \text{ Individual Error SS}$                                                 | $\Sigma \text{ df for Individual Error}$ | $\frac{\text{Total Error SS}}{\text{df Total Error}}$ | -                                             |
| Lack of Fit (LOF)                                                      | Residual SS - Total Error SS                                                         | df Residual - df Total Error             | $\frac{\text{LOF SS}}{\text{df LOF}}$                 | $\frac{\text{MS LOF}}{\text{MS Total Error}}$ |

where Y = Values for Dependent Variable

X = Target Concentration

N = Total Number of Measurement

n = Number of Replicates at each Concentration

d = Difference between Duplicates

Do not round off intermediate numbers in calculations. Carry through at least six digits to avoid rounding off errors, even though in the final results less than six digits will be significant.

# APPENDIX B



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APPENDIX B

| SOIL                                      |              |                      |                                  |                                |               |                    |                                        |          |                      | WATER                             |                                |               |                    |                                          |                                             |
|-------------------------------------------|--------------|----------------------|----------------------------------|--------------------------------|---------------|--------------------|----------------------------------------|----------|----------------------|-----------------------------------|--------------------------------|---------------|--------------------|------------------------------------------|---------------------------------------------|
| USATHAMA Analyte                          | Analyte Code | Amount Sample Needed | Container And Fixative (Solvent) | HOLD TIMES Extr./Analysis hrs. | METHOD INSTR. | Certified Method # | CRL (µg/g)                             | Comments | Amount Sample Needed | Container And Fixative (Solvent)  | HOLD TIMES Extr./Analysis hrs. | METHOD INSTR. | Certified Method # | CRL (µg/L)                               | Comments                                    |
| ARSENIC                                   | As           | 10 g                 | 20 mL Polyeth. Vial              | -/180                          | GFAA          | BB                 | 2.5                                    |          | 500 mL               | Plastic                           | -/180                          | GFAA          | AX8                | 2.35                                     |                                             |
| HEXACHROMIUM Cr+6                         | CR#EX        | 10 g                 | 4 oz. w/m Plastic                | -/24 hrs.                      | Auto Analyzer | JY03               | 1.0                                    |          | 125 mL               | Plastic                           | -/24 hrs.                      | Auto Analyzer | SF01               | 2.5                                      |                                             |
| IRON                                      | Fe           |                      | CERTIFICATION NOT REQUIRED       |                                |               |                    |                                        |          | 500 mL               | Plastic                           | -/7                            | Spec          | SY04               | 50.0                                     |                                             |
| LEAD                                      | Pb           | 10 g                 | 20 mL Polyeth. Vial              | -/180                          | GFAA          | JD21               | 0.467                                  |          | 250 mL               | Plastic                           | -/180                          | GFAA          | SD18               | 4.47                                     |                                             |
| MAGNESIUM                                 | Mg           | 10 g                 | 20 mL Polyeth. Vial              | -/180                          | FLAA          | JA02               | 2.37                                   |          | 250 mL               | Plastic                           | -/180                          | FLAA          | SC07               | 8.35                                     |                                             |
| MERCURY                                   | Hg           | 10 g                 | 20 mL Polyeth. Vial              | -/28                           | CV            | Y9                 | 0.05                                   |          | 500 mL               | Plastic                           | -/28                           | CV            | OC8                | 0.10                                     |                                             |
| SELENIUM                                  | Se           | 10 g                 | 20 mL Polyeth. Vial              | -/180                          | GFAA          | JD20               | 0.449                                  |          | 250 mL               | Plastic                           | -/180                          | GFAA          | SD25               | 2.53                                     |                                             |
| SILVER                                    | Ag           | 10 g                 | 20 mL Polyeth. Vial              | -/180                          | GFAA          | JD22               | 0.0124                                 |          | 250 mL               | Plastic                           | -/180                          | GFAA          | SD26               | 0.333                                    |                                             |
| VANADIUM                                  | V            | 10 g                 | 20 mL Polyeth. Vial              | -/180                          | GFAA          | JD23               | 0.941                                  |          | 500 mL               | Plastic                           | -/180                          | GFAA          | SD29               | 4.36                                     |                                             |
| AMMONIA                                   | NH3N2        |                      | CERTIFICATION NOT REQUIRED       |                                |               |                    |                                        |          | 125 mL               | Plastic                           | -/28                           | Auto Analyzer | TF30               | 8.42                                     |                                             |
| ANIONS Bromide* Fluoride Chloride Sulfate | Br F CL SO4  | 5 g                  | 20 mL Polyeth. Vial              | -/28                           | Ion Chrom.    | KT07               | Br 6.0<br>F 6.36<br>CL 7.12<br>SO4 5.0 |          | 125 mL               | Plastic                           | -/28                           | Ion Chrom.    | TT09               | Br 407.<br>F 153.<br>CL 278.<br>SO4 175. |                                             |
| CYANIDE                                   | CYN          | 10 g                 | 4 oz. w/m Plastic                | -/14                           | Auto Analyzer | KF15               | 0.25                                   |          | 250 mL               | Plastic                           | -/14                           | Auto Analyzer | TF34               | 5.0                                      |                                             |
| NITRATE/NITRITE                           | NIT          | 10 g                 | 4 oz. w/m Plastic                | -/28                           | Auto Analyzer | KF17               | 1.00                                   |          | 125 mL               | Plastic                           | -/28                           | Auto Analyzer | LL8                | 10.0                                     |                                             |
| NITRITE                                   | NO2          |                      | CERTIFICATION NOT REQUIRED       |                                |               |                    |                                        |          | 125 mL               | Plastic                           | -/48 hrs.                      | Auto Analyzer | TF31               | 5.0                                      |                                             |
| NITROCELLULOSE                            | NC           | 10 g                 | 4 oz. w/m Plastic                | -/28                           | Auto Analyzer | LF05               | 23.1                                   |          | 125 mL               | Plastic                           | -/28                           | Auto Analyzer | UF05               | 222                                      |                                             |
| T-PHOSPHORUS                              | P4           | 10 g                 | 4 oz. w/m Plastic                | -/28                           | Auto Analyzer | KF18               | 41.6                                   |          | 125 mL               | Plastic                           | -/28                           | Auto Analyzer | TF29               | 1.0                                      |                                             |
| TKN                                       | N2KJEL       |                      | CERTIFICATION NOT REQUIRED       |                                |               |                    |                                        |          | 125 mL               | Plastic                           | -/28                           | Auto Analyzer | TF28               | 64.0                                     |                                             |
| SULFIDE                                   | S            |                      | CERTIFICATION NOT REQUIRED       |                                |               |                    |                                        |          | 125 mL               | Plastic<br>1 mL ZnAc<br>1 mL NaOH | -/7                            | Spec          | TY15               | 11.0                                     | Preservatives added to samples sequentially |

| WATER                                                                                                                                                                                                                                                                                    |                      |                                  |                            |                     |                    |                                                                                                                                                                                                                                                                                          |                   |                      |                                    |                            |                     |                    |                                                                                                                                                                                                                                                                                 |                |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|----------------------------------|----------------------------|---------------------|--------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------|----------------------|------------------------------------|----------------------------|---------------------|--------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------|
| SOIL                                                                                                                                                                                                                                                                                     |                      |                                  |                            |                     |                    |                                                                                                                                                                                                                                                                                          |                   |                      |                                    |                            |                     |                    |                                                                                                                                                                                                                                                                                 |                |
| Analyte Code                                                                                                                                                                                                                                                                             | Amount Sample Needed | Container And Fixative (Solvent) | HOLD TIMES Extr./ Analysis | METHOD INSTR.       | Certified Method # | CRL ( $\mu\text{g/g}$ )                                                                                                                                                                                                                                                                  | Comments          | Amount Sample Needed | Container And Fixative (Solvent)   | HOLD TIMES Extr./ Analysis | METHOD INSTR.       | Certified Method # | CRL ( $\mu\text{g/L}$ )                                                                                                                                                                                                                                                         | Comments       |
| USATHAMA Analyte                                                                                                                                                                                                                                                                         |                      |                                  |                            |                     |                    |                                                                                                                                                                                                                                                                                          |                   |                      |                                    |                            |                     |                    |                                                                                                                                                                                                                                                                                 |                |
| ICP METALS I                                                                                                                                                                                                                                                                             |                      |                                  |                            |                     |                    |                                                                                                                                                                                                                                                                                          |                   |                      |                                    |                            |                     |                    |                                                                                                                                                                                                                                                                                 | RMA Analytes   |
| Cadmium<br>Calcium<br>Chromium<br>Copper<br>Lead<br>Magnesium<br>Potassium<br>Sodium<br>Zinc                                                                                                                                                                                             | 10 g                 | 20 mL<br>Polyeth. Vial           | - / 180                    | ICP<br>Sequential   | P9                 | CD 0.74<br>--<br>CR 6.5<br>CU 4.7<br>PB 8.4<br>--<br>--<br>--<br>--<br>ZN 8.7                                                                                                                                                                                                            | RMA<br>Analytes   | 500 mL               | Plastic<br>0.5 mL HNO <sub>3</sub> | - / 180                    | ICP<br>Sequential   | GG8                | CD 8.4<br>CA 500.0<br>CR 24.0<br>CU 26.0<br>PB 74.0<br>MG 500.0<br>K 250.0<br>NA 940.0<br>ZN 22.0                                                                                                                                                                               | RMA Analytes   |
| ICP METALS II                                                                                                                                                                                                                                                                            |                      |                                  |                            |                     |                    |                                                                                                                                                                                                                                                                                          |                   |                      |                                    |                            |                     |                    |                                                                                                                                                                                                                                                                                 | CLASS Analytes |
| Aluminum<br>Antimony<br>Arsenic<br>Barium<br>Beryllium<br>Boron<br>Cadmium<br>Calcium<br>Chromium<br>Cobalt<br>Copper<br>Iron<br>Lead<br>Magnesium<br>Manganese<br>Molybdenum<br>Nickel<br>Potassium<br>Selenium<br>Silver<br>Sodium<br>Tellurium<br>Thallium<br>Tin<br>Vanadium<br>Zinc | 10 g                 | 20 mL<br>Polyeth. Vial           | - / 180                    | ICP<br>Simultaneous | JS12               | AL 11.2<br>SB 19.6<br>AS 16.4<br>BA 3.26<br>BE 0.427<br>B 6.64<br>CD 1.20<br>CA 26.3<br>CR 1.04<br>CO 2.50<br>CU 2.84<br>FE 6.66<br>PB 7.44<br>MG 10.1<br>MN 9.87<br>MO 14.3<br>NI 2.74<br>K 131<br>SE 20.7<br>AG 0.803<br>NA 38.7<br>TE 14.9<br>TL 34.3<br>SN 7.43<br>V 1.41<br>ZN 2.34 | CLASS<br>Analytes | 500 mL               | Plastic<br>0.5 mL HNO <sub>3</sub> | - / 180                    | ICP<br>Simultaneous | SS12               | AL 112<br>SB 60.0<br>AS 117<br>BA 2.82<br>BE 1.12<br>B 230<br>CD 6.78<br>CA 105<br>CR 16.8<br>CO 25.0<br>CU 18.8<br>FE 77.5<br>PB 43.4<br>MG 135<br>MN 9.67<br>MO 52.7<br>NI 32.1<br>K 1240<br>SE 97.1<br>AG 10.0<br>NA 279<br>TE 118<br>TL 125<br>SN 59.9<br>V 27.6<br>ZN 18.0 | CLASS Analytes |



# USATHAMA ANALYTE SUMMARY ORGANIC

| SOIL                           |                                                                                                                                                |                      |                                                |                            |               |              |                                                                                                                          |          |                      | WATER                             |                            |               |              |                                                                                                                                 |          |
|--------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|------------------------------------------------|----------------------------|---------------|--------------|--------------------------------------------------------------------------------------------------------------------------|----------|----------------------|-----------------------------------|----------------------------|---------------|--------------|---------------------------------------------------------------------------------------------------------------------------------|----------|
| USATHAMA Analyte               | Analyte Code                                                                                                                                   | Amount Sample Needed | Container And Fixative/ (Solvent)              | HOLD TIMES Exir./ Analysis | METHOD INSTR. | Cert. Method | ORL (µg/g)                                                                                                               | Comments | Amount Sample Needed | Container And Fixative/ (Solvent) | HOLD TIMES Exir./ Analysis | METHOD INSTR. | Cert. Method | ORL (µg/L)                                                                                                                      | Comments |
| DBCP                           | DBCP                                                                                                                                           | 10 g                 | 40 mL VOA Vial                                 | 7/40                       | GC/EC         | 88           | 0.005                                                                                                                    |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | GC/EC         | AY8          | 0.20                                                                                                                            |          |
| DMP                            | DMP                                                                                                                                            |                      |                                                | CERTIFICATION NOT REQUIRED |               |              |                                                                                                                          |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | GC/FPD        | AW8A         | 0.66                                                                                                                            |          |
| DIMP/DMMP (Phosphonates)       | DIMP DMMP                                                                                                                                      |                      |                                                | CERTIFICATION NOT REQUIRED |               |              |                                                                                                                          |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | GC/FPD        | AT8          | DIMP 0.362<br>DMMP 0.188                                                                                                        |          |
| EXPLOSIVES                     | HMX<br>RDX<br>NB<br>Tetryl<br>1,3,5-Trinitrobenzene<br>1,3-Dinitrobenzene<br>2,4,6-Trinitrotoluene<br>2,4-Dinitrotoluene<br>2,6-Dinitrotoluene | 10 g                 | 4 oz. w/m Amber Glass w/TFE Cap                | 7/40                       | HPLC          | LW23         | HMX 2.00<br>RDX 1.26<br>NB 1.14<br>TETRYL 2.11<br>135TNB 0.922<br>13DNB 0.804<br>246TNT 2.00<br>24DNT 2.80<br>26DNT 2.00 |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | HPLC          | UW28         | HMX 0.533<br>RDX 0.418<br>NB 0.682<br>TETRYL 0.831<br>135TNB 0.210<br>13DNB 0.458<br>246TNT 0.428<br>24DNT 0.367<br>26DNT 0.600 |          |
| HERBICIDES                     | 2,4-D<br>2,4,5-T<br>2,4,5-TP<br>SLVEX                                                                                                          | 20 g                 | 4 oz. w/m Amber Glass w/TFE Cap                | 7/40                       | GC/EC         | LH18         | 24D 0.0300<br>24ET 0.0201<br>SLVEX 0.0064                                                                                |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | GC/EC         | UH10         | 24D 0.283<br>24ET 0.180<br>SLVEX 0.006                                                                                          |          |
| HYDRAZINES                     | Hydrazine<br>Monomethylhydrazine<br>UDMH                                                                                                       |                      |                                                | CERTIFICATION NOT REQUIRED |               |              |                                                                                                                          |          | 2 x 40 mL            | 40 mL VOA Vial w/o Septum         | 7/40                       | GC/NP         | ZZ8          | HYDRZ 30.8<br>MHYDRZ 2.970<br>UDMH 21.8                                                                                         |          |
| HYDROCARBONS                   | Methyl Isobutyl Ketone<br>Dicyclopentadiene<br>Bicycloheptadiene                                                                               | 28 g                 | 4 oz. w/m Amber Glass w/TFE Cap (MeCp)         | 7/40                       | GC/FID        | PP6          | MBK 0.84<br>DCPD 0.45<br>BCHPD 1.10                                                                                      |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | GC/FID        | P8           | MBK 4.90<br>DCPD 4.98<br>BCHPD 5.87                                                                                             |          |
| NITROGEN PHOSPHORUS PESTICIDES | Altrazine<br>Parathion<br>Malathion<br>Supona<br>Vapona                                                                                        | 20 g                 | 4 oz. w/m Amber Glass w/TFE Cap (Acetone/Hex.) | 7/40                       | GC/EC         | LH15         | ATZ 0.181<br>PRTHN 0.156<br>MLTHN 0.126<br>SUPONA 0.148<br>DDVP 0.080                                                    |          | 1 Liter              | Amber Glass with TFE Cap          | 7/40                       | GC/EC         | UH11         | ATZ 4.03<br>PRTHN 0.647<br>MLTHN 0.373<br>SUPONA 0.787<br>DDVP 0.384                                                            |          |

# USATHAMA ANALYTE SUMMARY ORGANIC

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| SOIL                                                                                 |                                                                                                                                                                                                                                                                                                                                                  |                      |                                                |                            |               |              |                                                                                                                                                                                                                                                                                                                                                                                                                                                          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| USATHAMA Analyte                                                                     | Analyte Code                                                                                                                                                                                                                                                                                                                                     | Amount Sample Needed | Container And Fixative (Solvent)               | HOLD TIMES Extr./ Analysis | METHOD INSTR. | Cert. Method | QRL (µg/g)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      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Method | QRL (µg/L)                                | Comments                                 |
| NITROGLYCERIN/PEIN                                                                   | NG<br>PETN                                                                                                                                                                                                                                                                                                                                       | 10 g                 | 4 oz. w/m Amber Glass w/TFE Cap                | 7/40                       | HPLC/ DAD     | LW27         | NG 0.810<br>PETN 1.00                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |          | 1 Liter              | Amber Glass with TFE Cap         | 7/40                       | HPLC/DAD      | UN27         | NG 1.48<br>PETN 2.00                      |                                          |
| NITROGLANDINE                                                                        | NG                                                                                                                                                                                                                                                                                                                                               | 10 g                 | 4 oz. w/m Amber Glass w/TFE Cap                | 7/40                       | HPLC/ DAD     | LW30         | 0.0434                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |          | 1 Liter              | Amber Glass with TFE Cap         | 7/40                       | HPLC/DAD      | UN28         | 21.1                                      |                                          |
| NITROSAMINES<br>Nitrosodimethylamine<br>Nitrosodipropylamine<br>Nitrosodiphenylamine | NNDMEA<br>NNDNPA<br>NNDPA                                                                                                                                                                                                                                                                                                                        | 2 x 10 g             | 40 mL VOA Vial w/te Septum (Methanol)          | 7/40                       | GC/NPD        | LN08         | NNDMEA 0.010<br>NNDNPA 0.0552<br>NNDPA 0.080                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |          | 1 Liter              | Amber Glass with TFE Cap         | 7/40                       | GC/NPD        | LN10         | NNDMEA 0.101<br>NNDNPA 1.80<br>NNDPA 1.93 | *Do not use without consulting R. Goebel |
| ORGANOCHLORINE PESTICIDES I                                                          | ALDRIN<br>Alpha-BHC<br>Beta-BHC<br>Delta-BHC<br>Lindane<br>Chlordane<br>Dieldrin<br>Endrin<br>Endrin Alkyls<br>Endosulfen I<br>Endosulfen II<br>Isodrin<br>Methoxychlor<br>Heptachlor<br>Heptachlor Epoxide<br>p,p'-DDE<br>p,p'-DDD<br>p,p'-DDT<br>PCB-1016<br>PCB-1221<br>PCB-1232<br>PCB-1242<br>PCB-1248<br>PCB-1254<br>PCB-1260<br>Toxaphene | 50 g                 | 4 oz. w/m Amber Glass w/TFE Cap (Acetone/Hex.) | 7/40                       | GC/EC         | LH17         | ALDRIN 0.0014<br>Alpha-BHC 0.0028<br>Beta-BHC 0.0077<br>Delta-BHC 0.0068<br>Lindane 0.0010<br>Chlordane 0.0064<br>Dieldrin 0.0016<br>Endrin 0.0086<br>Endosulfen I -<br>Endosulfen II 0.0010<br>AENSILF 0.0007<br>BENSILF 0.0007<br>ISODR 0.0030<br>MEXCLR 0.0056<br>HPCL 0.0022<br>HPCLE 0.0013<br>PPDDE 0.0027<br>PPDDO 0.0027<br>PPDDT 0.0036<br>PCB016 0.100<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br>..<br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## SOIL

| USATHAMA Analyte                                                                     | Analyte Code                                                                                                                                                                                                                                                                                                                                     | Amount Sample Needed | Container And Fixative (Solvent)               | HOLD TIMES Extr./ Analysis | METHOD INSTR. | Cert. Method | QRL (µg/g)                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | Comments             |
|--------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|------------------------------------------------|----------------------------|---------------|--------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|
| NITROGLYCERIN/PEIN                                                                   | NG<br>PEIN                                                                                                                                                                                                                                                                                                                                       | 10 g                 | 4 oz. w/m Amber Glass w/TFE Cap                | 7/40                       | HPLC/DAD      | LW27         | NG 0.810<br>PEIN 1.00                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                      |
| NITROGLANDINE                                                                        | NG                                                                                                                                                                                                                                                                                                                                               | 10 g                 | 4 oz. w/m Amber Glass w/TFE Cap                | 7/40                       | HPLC/DAD      | LW30         | 0.0434                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |                      |
| NITROSAMINES<br>Nitrosodimethylamine<br>Nitrosodipropylamine<br>Nitrosodiphenylamine | NNDMEA<br>NNDNPA<br>NNDPA                                                                                                                                                                                                                                                                                                                        | 2 x 10 g             | 40 mL VOA Vial w/te Septum (Methanol)          | 7/40                       | GC/NPD        | UN08         | NNDMEA 0.010<br>NNDNPA 0.0652<br>NNDPA 0.0680                                                                                                                                                                                                                                                                                                                                                                                                                                   |                      |
| ORGANOCHLORINE PESTICIDES I                                                          | ALDRIN<br>Alpha-BHC<br>Beta-BHC<br>Delta-BHC<br>Lindane<br>Chlordane<br>Dieldrin<br>Endrin<br>Endrin Alkyls<br>Endosulfen I<br>Endosulfen II<br>Isodrin<br>Methoxychlor<br>Heptachlor<br>Heptachlor Epoxide<br>p,p'-DDE<br>p,p'-DDD<br>p,p'-DDT<br>PCB-1016<br>PCB-1221<br>PCB-1232<br>PCB-1242<br>PCB-1248<br>PCB-1254<br>PCB-1260<br>Toxaphene | 50 g                 | 4 oz. w/m Amber Glass w/TFE Cap (Acetone/Hex.) | 7/40                       | GC/EC         | LH17         | ALDRIN 0.0014<br>Alpha-BHC 0.0028<br>Beta-BHC 0.0077<br>Delta-BHC 0.0068<br>Lindane 0.0010<br>Chlordane 0.0064<br>Dieldrin 0.0016<br>Endrin 0.0086<br>Endrin Alkyls -<br>Endosulfen I 0.0010<br>Endosulfen II 0.0007<br>Isodrin 0.0030<br>Methoxychlor 0.0056<br>Heptachlor 0.0022<br>Heptachlor Epoxide 0.0013<br>p,p'-DDE 0.0027<br>p,p'-DDD 0.0027<br>p,p'-DDT 0.0036<br>PCB-1016 0.100<br>PCB-1221<br>PCB-1232<br>PCB-1242<br>PCB-1248<br>PCB-1254<br>PCB-1260<br>Toxaphene | FOR ALL NON-RMA WORK |



## USATHAMA ANALYTE SUMMARY

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# APPENDIX C



## APPENDIX C

### SAMPLE CONTAINER CLEANING PROCEDURES

To ensure the integrity of aqueous and solid samples, steps must be taken to minimize contamination from the containers in which they are stored. If the analyte(s) to be determined are organic in nature, the container should be made of amber glass. If the analyte(s) are inorganic, the container should be polyethylene. When both organic and inorganic substances are expected to be present, separate samples should be taken. New sample bottles must be cleaned according to either of the procedures presented below; reuse of sample containers is expressly prohibited. The procedure that was used must be documented. Commercially cleaned containers may be utilized if cleaning procedures comply with those provided in this appendix and prior USATHAMA Chemistry Branch approval is obtained. The procedures for cleaning the glass and polyethylene containers and their caps are as follows:

#### ALTERNATE A:

- Polyethylene Bottles and Polyethylene Caps
  - (1) Rinse bottles and lids with 5 percent sodium hydroxide.
  - (2) Rinse with deionized water.
  - (3) Rinse with 5 percent Ultrex (or equivalent) nitric acid in deionized water.
  - (4) Rinse with deionized water.
  - (5) Drain and air dry.
- Amber-Glass Bottles or 40-ml Vials
  - (1) Scrub and wash bottles in detergent.
  - (2) Rinse with copious amounts of distilled water.
  - (3) Rinse with acetone.
  - (4) Rinse with methylene chloride (Nanograde or equivalent).
  - (5) Rinse with hexane (Nanograde or equivalent).
  - (6) Air dry.

(7) Heat to 200°C.

(8) Allow to cool.

(9) Cap with clean caps with Teflon liners.

- Bottle Caps

(1) Remove paper liners from caps.

(2) Wash with detergent.

(3) Rinse with distilled water.

(4) Dry at 40°C.

- Teflon Liners (avoid contact with fingers)

(1) Wash with detergent.

(2) Rinse with distilled water.

(3) Rinse with acetone.

(4) Rinse with hexane (Nanograde or equivalent).

(5) Air dry.

(6) Place liners in cleaned caps.

(7) Heat to 40°C for 2 hours.

(8) Allow to cool.

(9) Use to cap cleaned bottles.

#### ALTERNATE B: (Specified by EPA for CLP)

- Amber Glass Bottles

(1) Wash containers, closures, and teflon liners in hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.



- (3) Rinse with 1:1 nitric acid.
- (4) Rinse three times with ASTM Type 1 deionized water.
- (5) Rinse with pesticide grade methylene chloride.
- (6) Oven dry.
- (7) Remove containers, closures, and teflon liners from oven.
- (8) Place teflon liners in closures and place closures on containers.  
Attendant to wear gloves and containers not to be removed from preparation room until sealed.

- 40 mL Borosilicate Glass Vials

- (1) Wash vials, septa, and closures in hot tap water with laboratory grade non-phosphate detergent.
- (2) Rinse three times with tap water.
- (3) Rinse three times with ASTM Type 1 deionized water.
- (4) Oven dry vials, septa, and closures.
- (5) Remove vials, septa, and closures from oven.
- (6) Place septa in closures, teflon side down, and place on vials.  
Attendant to wear gloves and vials not to be removed from preparation room until sealed.

- High Density Polyethylene Bottles

- (1) Wash bottles, closures, and teflon liners with hot tap water with laboratory grade non-phosphate detergent.
- (2) Rinse three times with tap water.
- (3) Rinse with 1:1 nitric acid.
- (4) Rinse three times with ASTM Type 1 deionized water.
- (5) Air dry in contaminant-free environment.

- (6) Place liners in closures and place closures on bottles. Attendant to wear gloves and bottles not to be removed from preparation room until sealed.

Documentation must be provided to the USATHAMA Chemistry Branch validating that the bottles are in fact "clean." Documentation may consist of the results of "bottle blank" analysis using the method(s) that will be applied to the sample that will be placed in that bottle. QC results from the supplier of commercially cleaned containers, demonstrating that the bottle(s) are "clean," will be acceptable. The documentation must be provided before the bottles are used to collect samples in the field. This validation is to be performed or provided for each batch or "lot" of bottles cleaned together and must be provided at least once for each installation where they are used.



# APPENDIX D

## PARAMETERS/METHOD NUMBERS FOR ANALYSIS

|                  |                     |                        |
|------------------|---------------------|------------------------|
| PRIME CONTRACTOR | PRIME CONT.<br>CODE | BASE CLOSURE<br>YES NO |
| INSTALLATION     | INSTALL<br>CODE     | MATRIX                 |

[illegible]



### Analysis:

**SAMPLE ANALYSIS DATE:**

**Appendix B: Lists of Standard Operating Procedures**



## DataChem Laboratory SOPs Cross Reference

| USATHAMA SOP Requirement                                                                                                    | DataChem SOP Title                                                                   | Document Control Number      |
|-----------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------------------|
| Sample receipt and log-in                                                                                                   | Sample Receipt and Logging Chain-of-Custody and Laboratory Tracking                  | SOP-EPA-100<br>SOP-EPA-1210  |
| Laboratory personnel training                                                                                               | Record of Training                                                                   | SOP-GLP-007                  |
| Sample storage                                                                                                              | Security of Laboratory Samples                                                       | SOP-EPA-400                  |
| Sample scheduling                                                                                                           | Chain-of-Custody and Laboratory Tracking                                             | SOP-EPA-1210                 |
| Preventing sample contamination                                                                                             | Preventing Sample Contamination                                                      | SOP-EPA-300                  |
| Security for laboratory, samples and standards                                                                              | Security of Laboratory Samples                                                       | SOP-EPA-400                  |
| Traceability/Equivalency of standards                                                                                       | Standards Purity, Traceability and Verification                                      | SOP-EPA-500                  |
| Standard solution verification                                                                                              | Standards Purity, Traceability and Verification                                      | SOP-EPA-500                  |
| Maintaining instrument records and logbooks                                                                                 | Documentation - Maintaining Instrument Records, Notebooks and Logbooks               | SOP-EPA-600                  |
| Sample analysis and data control systems                                                                                    | Data Control Systems - Calibration                                                   | SOP-EPA-700                  |
| Glassware cleaning                                                                                                          | Glassware Cleaning - Organic Analysis<br>Glassware Cleaning - Inorganic Analysis     | SOP-EPA-810<br>SOP-EPA-820   |
| Technical and managerial review of laboratory operation and data package preparation                                        | Technical and managerial review of laboratory operation and data package preparation | SOP-EPA-900                  |
| Internal review of contractually-required quality assurance and quality control data for each individual data and reporting | Internal Review of QA/QC Data<br>Document Control and Report Preparation             | SOP-EPA-1000<br>SOP-EPA-1220 |
| Data reduction and validation                                                                                               | Sample Data Validation/Self-Inspection System                                        | SOP-EPA-1300                 |

# Arthur D. Little Field SOPs Cross Reference

| USATHAMA SOP Requirement        | Arthur D. Little SOP Title                                                                                                                                                                                                                | Document Control Number                                  |
|---------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------|
| Training                        | Training of Field Personnel                                                                                                                                                                                                               | USA-0003                                                 |
| Sample Management               | Sample Receipt and Log-in                                                                                                                                                                                                                 | ADL-1002                                                 |
| Numbering and Labeling          | Management of USATHAMA Samples                                                                                                                                                                                                            | USA-6003                                                 |
| Sample Tracking                 | Sample Custody                                                                                                                                                                                                                            | ADL-1001                                                 |
| Sample Containers               | Sample Containers, Preservatives and Holding Times                                                                                                                                                                                        | USA-1000                                                 |
| Sample Preservation and Storage | Sample Containers, Preservatives and Holding Times<br>Sample and Extract Storage                                                                                                                                                          | USA-1000<br>ADL-1005                                     |
| Holding Times                   | Sample Containers, Preservatives and Holding Times                                                                                                                                                                                        | USA-1000                                                 |
| Shipping                        | Sample Custody                                                                                                                                                                                                                            | ADL-1001                                                 |
| Sample Collection Procedures    | Standard Penetration Test and Split Spoon Sampling<br>Ground Water Monitoring Well Sampling                                                                                                                                               | USA-4002<br>USA-1011                                     |
| Corrective Action               | Corrective Actions for Field Operations                                                                                                                                                                                                   | USA-7006                                                 |
| Records Management              | Geotechnical Documentation                                                                                                                                                                                                                | ADL-4014                                                 |
| Chemical and Sample Disposal    | Chemical and Sample Disposal                                                                                                                                                                                                              | USA-3000                                                 |
| Reporting                       | Geotechnical Documentation                                                                                                                                                                                                                | ADL-4014                                                 |
| Field Analyses                  | Conductivity Meter Calibration and Measurement<br>HNU-PI-101 Organic Vapor Analyzer Operation<br>pH Meter Operation<br>Inspection and Use of the MSA 261 Combustible gas/Oxygen Meter<br>Calibration and Use of the Portable Turbidimeter | ADL-5011<br>ADL-5012<br>ADL-5013<br>ADL-5018<br>ADL-5026 |
| Geotechnical*                   | Exploratory Boring Procedures                                                                                                                                                                                                             | USA-4001                                                 |
| Geotechnical*                   | Standard Penetration Tests and Split Spoon Sampling                                                                                                                                                                                       | USA-4002                                                 |



Arthur D. Little Field SOPs Cross Reference (continued)

| USATHAMA SOP Requirement | Arthur D. Little SOP Title                                                             | Document Control Number |
|--------------------------|----------------------------------------------------------------------------------------|-------------------------|
| Geotechnical*            | Grouts Methods and Criteria                                                            | USA-4003                |
| Geotechnical*            | Monitoring Well Development                                                            | USA-4010                |
| Geotechnical*            | Monitoring Well Water Level Measurement Procedure                                      | USA-4012                |
| Geotechnical*            | In-Situ Permeability Testing (Slug Testing) with Hydraulic Conductivity Data Reduction | ADL-4018                |

\* Arthur D. Little requirement, not a USATHAMA requirement

## Appendix C: Checklists for Field and Laboratory Activities



## DATA PACKAGE CHECKLIST

Lot

Method Number

I have checked this report and data package to make certain that the following conditions are in compliance with USATHAMA requirements:

### I. GENERAL

- ☐ 1. All enclosed copies are legible and not excessively reduced.
- ☐ 2. There are no "yellow stickies," tablet sheets, or other undocumented forms in the data package.
- ☐ 3. All required documents, including a completed chain-of-custody form, are enclosed.
- ☐ 4. The data block on the outside of the data package are complete, with all other relevant information included.

### II. NOTEBOOK PAGES

- ☐ 5. All copies of notebook pages are identified by notebook number and page number.
- ☐ 6. All units ("ug/L"; "ug/g"; "mL") are clearly defined.
- ☐ 7. Each page has been signed and dated by the analyst and reviewer.
- ☐ 8. All written explanations have all of the necessary information included and may stand alone as written.

### III. COMPUTER DATA SHEET

- ☐ 9. The preliminary computer data sheet has been signed and dated by both the reviewer and the analyst.

### IV. CHROMATOGRAMS AND STRIP CHARTS

- ☐ 10. All enclosed chromatograms and/or strip charts have been labelled properly, signed, and dated by the analyst.

**DATA PACKAGE CHECKLIST**

## V. CHECKLISTS

- \_\_\_ 11. All enclosed checklists are the current version, and have either each blank initialled or the blanks checked with a signature at the bottom of the page.

## VII. CORRECTIONS

- \_\_\_ 12. No white-out or correction tape has been used on any raw data.
- \_\_\_ 13. All cross-outs consist of only a single line, and have been initialled and dated.
- \_\_\_ 14. All cross-outs have a legitimate, sufficient explanation alongside.

\_\_\_\_\_  
Analyst Signature      Date

\_\_\_\_\_  
Checker Signature      Date

Data were obtained while the analytical process was in-control and meet the agreed upon Data Quality Objectives.

\_\_\_\_\_  
QAC Signature

\_\_\_\_\_  
Date

**DATA PACKAGE CHECKLIST**



## DATA PACKAGE DOCUMENT INVENTORY LIST

Lot

Method Number

**Analyst:** If the listed document is in the data package, please initial inventory list.

- \_\_\_\_\_ Review sign-off sheet;
- \_\_\_\_\_ Chain-of-custody sheet, laboratory;
- \_\_\_\_\_ Chain-of-custody sheet, field;
- \_\_\_\_\_ Reagent blank report form;
- \_\_\_\_\_ Screening chromatogram - dated and initialed by analyst;
- \_\_\_\_\_ Unknown analyte report sheet;
- \_\_\_\_\_ Best fit spectra for each unknown peak;
- \_\_\_\_\_ NIST library search for unknowns;
- \_\_\_\_\_ Coding form or approved data reporting form;
- \_\_\_\_\_ Copy of extraction logbook pages;
- \_\_\_\_\_ Copy of sample preparation logbook pages;
- \_\_\_\_\_ Copy of analyst's notebook pages;
- \_\_\_\_\_ Copy of moisture logbook pages;
- \_\_\_\_\_ Copy of standards preparation (logbook pages);
- \_\_\_\_\_ Raw data output - dated and initialed by analyst (printouts, etc.);
- \_\_\_\_\_ DFTPP 12 hour tuning and mass calibration report(s);
- \_\_\_\_\_ BFB 12 hour tuning and mass calibration report(s);
- \_\_\_\_\_ Initial calibration data, including RIC, and quantitation reports for four standards;
- \_\_\_\_\_ Daily calibration data, including RIC, and quantitation report;
- \_\_\_\_\_ RIC and quantitation report for: field samples, QC samples, blank samples;
- \_\_\_\_\_ Check standard results;
- \_\_\_\_\_ Chromatogram or strip chart recorded output with analyte peak indicated, dated, and initialed by analyst;
- \_\_\_\_\_ Expanded scale blow-up of manually integrated peak;
- \_\_\_\_\_ Unknown report, library search, best fit spectra;
- \_\_\_\_\_ Raw data for quantitated analytes (when positively identified - including difference display, and enhanced and unenhanced spectra);
- \_\_\_\_\_ Example calculations.

NA - item not applicable to analytical procedure.

## **DATA PACKAGE DOCUMENT INVENTORY LIST**

USATHAMA DATA REVIEW CHECKLIST

Lot

Method Number

HOLDING TIMES

YES NO N/A

COMMENTS

1. Was extraction/digestion holding time met for all samples?

2. Was analysis holding time met for all extracts/digestates?

3. Were all reported dilutions performed within holding times?

PAPER TRAIL \_\_\_\_\_

4. Is chain-of-custody information present and complete?

5. Are all necessary forms present, complete, and filled out in blue or black ink?

6. Are all changes made properly, and initialed/dated?

**USATHAMA DATA REVIEW CHECKLIST**



DAILY CALIBRATION

YES NO N/A

COMMENTS

7. Was a standard curve for each analyte (as specified in the method) plus a blank analyzed with each daily lot?

8. Was a new standard curve run on the day of reanalysis of diluted extracts, and was it used for sample calculation for that date?

9. Do the calibration standards equal or bracket the concentration equivalent to the CRL and the UCR (if appropriate)?

10. Do the calibration standards equal or bracket the CRL and the highest sample or spike response in the daily lot (if appropriate)?

11. Was the standard specified in the method reanalyzed at the end of each daily lot, and at the appropriate interval within that lot and did the response meet criteria?

CONTROL SPIKES \_\_\_\_\_

12. Were standard matrix control spikes (spiked with the appropriate analytes and at the designated levels) and a standard matrix blank extracted/digested and analyzed on the same date as the daily lot?

**USATHAMA DATA REVIEW CHECKLIST**

YES NO N/A COMMENTS

13. If dilution and reanalysis have been performed on a different day, was at least one control spike reanalyzed with the diluted samples? Has this spike been reported with the data on the appropriate date?

14. Did control spikes pass control chart criteria? If not, has an acceptable explanation been provided, and correction taken as necessary?

#### SAMPLE ANALYSIS

15. Are reported sample and control spike concentrations within the certified concentration range of the method?

16. If sample concentrations above the UCR are reported, were they diluted into the certified range with the dilution factors clearly indicated?

17. Are reported detection limits the certified reporting limits?

18. Are justifications supplied for all non-use of data, analyses, etc.

19. Are all reanalyzed samples clearly marked and explanation presented?

### **USATHAMA DATA REVIEW CHECKLIST**



YES NO N/A COMMENTS

20. Are all manual integration justified?

QUALITY ASSURANCE REVIEWER ONLY

21. For randomly selected data points, can the reported concentrations be back calculated using the available raw data?

REVIEWER'S SIGNATURE

CHEM: \_\_\_\_\_ DATE \_\_\_\_\_

SUPERVISOR: \_\_\_\_\_ DATE \_\_\_\_\_

QA: \_\_\_\_\_ DATE \_\_\_\_\_

USATHAMA DATA REVIEW CHECKLIST

## USATHAMA REPORT CHECKLIST

Lot

Method Number

I have reviewed and checked the enclosed report for the following items:

### Transcriptions

- \_\_\_\_\_ 1. Soil weights and liquid volumes have been copied correctly.
- \_\_\_\_\_ 2. All information from strip charts, chromatograms, and lab notebooks has been correctly transferred to the computer.
- \_\_\_\_\_ 3. All information from the field chain-of-custody has been correctly copied onto the coding form.
- \_\_\_\_\_ 4. Sample results and dilution factors derived from computer printouts or notebook calculations have been accurately copied onto the coding form.

### Calculations

- \_\_\_\_\_ 5. All calculations have been verified.
- \_\_\_\_\_ 6. All reported values are uncorrected for moisture, dilution, or other factors.

### Coding Form and QC Form

- \_\_\_\_\_ 7. The mantissa and exponent for each sample result and dilution factor have been accurately entered onto the coding form.
- \_\_\_\_\_ 8. The correct CRL has been used on the coding form.
- \_\_\_\_\_ 9. The correct method ID has been noted on both the coding form and the outside of the data package.
- \_\_\_\_\_ 10. Preparation date and analysis date on the coding form agree with those on the chain-of-custody.
- \_\_\_\_\_ 11. The QC form indicating whether or not the QC spikes are within control has been completely and accurately completed.
- \_\_\_\_\_ 12. Sample results are not reported below the CRL or above the highest standard.

\_\_\_\_\_  
Analyst Signature

\_\_\_\_\_  
Date

\_\_\_\_\_  
Checker Signature

\_\_\_\_\_  
Date

**USATHAMA REPORT CHECKLIST**



## AUDIT CHECKLIST

| <u>YES</u> | <u>NO</u> | <u>COMMENT</u> |
|------------|-----------|----------------|
|------------|-----------|----------------|

### PRE-AUDIT

1. Notified laboratory
2. Notified project officer
3. Made travel arrangements
4. Reviewed background information/  
data
5. Requested laboratory to have data/  
methods/personnel available
6. Prepared agenda

### IN-BRIEFING

7. Introduced participants
8. Described goals and objectives of  
audit/agenda
9. Identified specific areas for  
review that could require some  
laboratory preparation
10. Discussed general overview/status  
on project
11. Discussed problem areas

USATHAMA AUDIT CHECKLIST

YES    NO    COMMENT

GENERAL

12. a. Has detailed Project QC Plan (QAPjP) been submitted?
- b. Has individual been appointed as QAC who is independent from analysis?
- c. Have sufficient facilities, personnel, and instrumentation been provided to perform the required analyses?
- d. Does the QAC have the resources to function effectively?
- e. Are chemicals and reagents of sufficient quality so as not to compromise the analytical system?
- f. Is housekeeping commensurate with analytical techniques?
- g. Has a training plan been developed and training been documented?
- h. Is the correct version of USATHAMA supplied software being used?



AUDIT

YES

NO

COMMENT

13. Samples chosen to follow through laboratory:

Inorganic

Organic

14. Sample receiving:

- a. Are procedures/SOPs available?
- b. Are samples checked upon receipt?
- c. Is the sample checking documented?
- d. Is area secure?
- e. Are chain-of-custody forms filed?
- f. Are internal chain-of-custody forms generated?
- g. Are samples logged in according to SOP?
- h. Are USATHAMA numbers assigned?
- i. Are numbers allocated for QC samples?

**USATHAMA AUDIT CHECKLIST (Cont.)**

AUDIT (cont)

YES   NO   COMMENT

j. Are samples stored in refrigerator until needed?

k. Is the temperature of refrigerator monitored?

l. Is there a sign-out system for samples?

m. Are VOA samples isolated from other samples?

15. Inorganics Section:

a. Are logbooks kept for:

Digestion?

Analysis?

Instrument maintenance?

Standard preparation?

b. Are logbooks identified with unique number?

c. Are pages of logbooks numbered?

d. Are reagents/solvents/acids checked for purity, etc.?



Inorganics (cont)

YES   NO   COMMENT

e. Are standards stored correctly?

f. Is inventory of standards maintained?

g. Are standard solutions labelled with date prepared?

h. Are solution validity checks documented?

i. Are standards traceable from receipt to use?

j. Are samples maintained and stored according to SOP?

k. Are procedures in place to minimize cross contamination?

l. Are samples analyzed according to certified methods?

m. Are results of analyses stored in data packages?

16. Organics Section:

a. Are logbooks kept for:

Extraction?

Analysis?

**USATHAMA AUDIT CHECKLIST (Cont.)**

# Hydrocarbon Fingerprinting by GC

The composition of various petroleum products may vary depending on refinery operation, seasonal additives, and crude oil sources. As such, identification of products, especially weathered products, can be very complicated. If a match can be determined, the following products can be identified.

- Gasoline
- Aviation Gasoline
- Kerosene (or Jet Fuel A)
- Paint Thinner
- Turpentine
- Fuel Oil #2 (or Diesel #2)
- Fuel Oil #4
- Fuel Oil #6
- Coal Tar
- Creosote
- Lubricating Oils (such as motor oils)
- Processing Oils (such as cutting fluids)
- Vegetation Hydrocarbons
- Asphalt

Other products can also be identified. Pattern matching to a specific reference material can also be performed. If a reference solution is submitted, specific comparisons can be made.

Additional information (e.g., distribution ranges based on n-alkanes, degree of weathering based on pristane to n-C17 and phytane to n-C18 ratios, concentration of n-alkanes and

ratio of resolved to unresolved can be provided on request. If standards are available, specific products can be identified using pattern matching as described in ASTM D3328-78.

Duplicate laboratory control spikes of fuel oil #2 are analyzed every 20 samples. A method blank containing a surrogate spike is analyzed with each batch. The blank amount should be less than the method reporting limit. In addition, each sample is spiked with the surrogate to monitor extraction efficiency. Matrix spikes, duplicates, and field blanks are recommended, as needed, to satisfy a program's specific data quality objectives.

In application, this method has a broad set of uses. It is especially helpful in cases where a identification of an unknown petroleum contaminant is necessary. It is also frequently used to delineate areas of contamination.

For example, the test was used at a Superfund site that was contaminated with fuel oil #2 and coal tar from a coal gasification operation. The relative concentration of the two products

from the analysis of soil borings was used to assign responsibility for cleanup costs to the responsible parties.

In another case, an unknown petroleum product was observed in a drinking water well. The well was located down gradient of a heating oil distributor and a gasoline service station. The analysis of a water sample from the well identified the contamination to be fuel oil #2 (heating oil).

Although the test is generally used in conjunction with a standard test for measuring total petroleum hydrocarbons (e.g., Method 418.1), it can sometimes provide the more useful data. This is especially true in cases where the samples contain compounds that interfere with the procedures. In one case, high values were detected in soil samples collected during an underground storage tank replacement. The values were not consistent with field observations. Subsequent testing using this method revealed that the samples contained a high level of leak hydrocarbons (i.e., waxes) that were not petroleum in origin.

The test has also been useful on non-conventional matrices such as seawater, drummed wastes, and air samples.

Sources: ASTM D3338-78, Standard Test Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography, American Society for Testing and Materials, 1982.

EPA 8015, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, January 1990.