EXHIBIT E

CONTRACT LABORATORY PROGRAM QUALITY ASSURANCE MONITORING PLAN

THIS PAGE INTENTIONALLY LEFT BLANK

Exhibit E - Contract Laboratory Program Quality Assurance Monitoring Plan

Table of Contents

Section		Page
1.0	OVERVIEW	5
	1.1 Quality Assurance/Quality Control (QA/QC) Activities	5
	1.2 Incentives/Sanctions	5
2.0	INTRODUCTION	6
	2.1 Quality Assurance/Quality Control (QA/QC) Program Components	6
3.0	GENERAL QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) REQUIREMENTS	7
4.0	SPECIFIC QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) MONITORING PROCEDURES	8
	4.1 Purpose	8
	4.2 Laboratory Audit and Intercomparison Study Program	8
	4.3 Annual Verification of Method Detection Limits (MDLs)	8
	4.4 Quarterly Verification of Linear Ranges/Interelement Correction Factors	9
	4.5 Quality Assurance/Quality Control Measurements	9
5.0	QUALITY ASSURANCE MANAGEMENT PLAN	10
	5.1 Introduction	10
	5.2 Required Elements of a Quality Assurance Management Plan	10
	5.3 Updating and Submitting the Quality Assurance Management Plan	12
	5.4 Incentives/Sanctions	13
6.0	STANDARD OPERATING PERFORMANCE STANDARDS	14
	6.1 Introduction	14
	6.2 Format	15
	6.3 Required SOPs	15
	6.4 Updating and Submitting SOP Requirements	18
	6.5 Incentives/Sanctions	19
7.0	CONTRACT COMPLIANCE SCREENING (CCS) PERFORMANCE STANDARDS	20
	7.1 Overview	20
	7.2 CCS Results	20
	7.3 CCS Trend Report	20
	7.4 Incentives/Sanctions	20
8.0	ANALYTICAL PERFORMANCE STANDARDS REQUIREMENTS	21
	8.1 Overview	21
	8.2 Preparation of Chemical Standards from the Neat High Purity Bulk Material	21
	8.3 Purchase of Chemical Standards Already in Solution	21
	8.4 Requesting Standards from the USEPA Standards Repository	24
	8.5 Documentation of the Verification and Preparation of Chemical Standards	24
	8.6 Incentives/Sanctions	25
9.0	DATA PACKAGE MONITORING AUDITS	26
	9.1 Overview	26
	9.2 Responding to the Data Package Audit Report	26
	9.3 Incentives/Sanctions	26
10.0	REGIONAL DATA REVIEW MONITORING	27
	10.1 Overview	27

Table of Contents (Con't)

Section — Page

11.0 QUALITY ASSURANCE (QA) PROFICIENCY MONITORING 28
11.1 Performance Evaluation (PE) Samples 28
11.2 Quarterly Blind (QB) Audits 28
11.3 Incentives/Sanctions 30

12.0 ON-SITE LABORATORY QUALITY ASSURANCE (QA) MONITORING EVALUATIONS . . 31
12.1 Overview 31
12.2 Quality Assurance On-Site Evaluation 31
12.3 Evidentiary Audit 31
12.4 Discussion of the On-Site Team's Findings 32
12.5 Incentives/Sanctions 32

13.0 ELECTRONIC DATA QUALITY ASSURANCE (QA) MONITORING AUDITS 33
13.1 Overview 33
13.2 Submission of the Instrument Electronic Data 35
13.3 Responding to the Electronic Data Audit Report 35
13.4 Incentives/Sanctions 35

14.0 DATA MANAGEMENT PERFORMANCE REQUIREMENTS 36
14.1 Overview 36
14.2 Documenting Data Changes 36
14.3 Lifecycle Management Procedures 36
14.4 Personnel Responsibilities 37

15.0 TABLES 38
TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan 38

1.0 OVERVIEW

Quality Assurance (QA) and Quality Control (QC) are integral parts of the U.S. Environmental Protection Agency's (USEPA's) Contract Laboratory Program (CLP). The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity, and ensures that data provided are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data.

1.1 Quality Assurance/Quality Control (QA/QC) Activities

During the planning of an environmental data collection program, QA activities focus on defining data quality criteria and designing a QC system to measure the quality of data being generated. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions.

1.1.1

This exhibit describes the overall QA/QC operations and the processes by which the CLP meets the QA/QC objectives defined above. This contract requires a variety of QA/QC activities. These contract requirements are the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These QC operations are designed to facilitate laboratory comparison by providing USEPA with comparable data from all Contractors. These requirements do not release the analytical Contractor from maintaining their own QC checks on method and instrument performance.

1.2 Incentives/Sanctions

The Contractor may anticipate incentives by <u>consistently</u> providing the following: (1) high quality, technically sound data as stipulated by the ILM05.3 contract; (2) on-time or early delivery of the Sample Delivery Group (SDG) Cover Sheet; (3) above average Quarterly Blind (QB) Performance Evaluation (PE) sample scores; (4) diskettes that pass the initial Contract Compliance Screening (CCS) acceptance criteria; and (5) SDGs delivered on-time. Samples are distributed routinely to Contractors based on the quality of work performed, as measured by the Performance Scheduling Algorithm (PSA) (see Section G of the contract for details). A Contractor that consistently meets the contract performance requirements as highlighted above, will earn a higher PSA score, thereby increasing the likelihood of receiving samples for analyses. If the Contractor fails to meet the requirements set forth in this Statement of Work (SOW) or elsewhere in the contract, USEPA may take, but is not limited to, the following actions (see Section E of the contract for details): reduction in the number of samples sent under the contract; suspension of sample shipments; data package audit(s); electronic data audit(s); on-site laboratory evaluation(s); and/or remedial PE sample(s).

2.0 INTRODUCTION

Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the Quality Control (QC) procedures and criteria incorporated into the ILM05.3 Statement of Work (SOW).

The data acquired from QC procedures are used to estimate and evaluate the information content of analytical results and to determine the necessity for, or the effect of, corrective action procedures. The parameters used to estimate information content include precision, accuracy, detection limit, and other quantitative and qualitative indicators. In addition, QC procedures give an overview of the activities required in an integrated program to generate data of known and documented quality required to meet defined objectives.

2.1 Quality Assurance/Quality Control (QA/QC) Program Components

2.1.1 The Contractor's QA/QC program shall include (1) internal QC criteria that demonstrate compliant levels of performance, as determined by QA review, as well as (2) external review of data and procedures accomplished by the monitoring activities of the USEPA OSRTI Analytical Services Branch (ASB), Regional Data Users, Sample Management Office (SMO), and the Quality Assurance Technical Support (QATS) Laboratory. Each external review accomplishes a different purpose. These reviews are described in specific sections of this exhibit. Laboratory evaluation samples, electronic data audits, and data packages provide an external QA reference for the program. A Contractor on-site evaluation system is also part of the external QA monitoring. A feedback loop provides the results of the various review functions to the Contractors through direct communications with the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and the USEPA OSRTI ASB Inorganic Program Manager (ASB PM).

2.1.2 This exhibit does not provide specific instructions for constructing QA Management Plans, QC systems, or a QA organization. It is, however, an explanation of the QA/QC requirements of CLP. It outlines minimum standards for QA/QC programs. It also includes specific items that are required in a Quality Assurance Management Plan (QAP) and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides USEPA with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

2.1.3 In order to assure that the product delivered by the Contractor meets the requirements of the contract, and to improve interlaboratory data comparison, the Contractor shall:

- Prepare, and adhere to, a written approved QAP, as defined in Exhibit E, Section 5;
- Prepare and adhere to, Standard Operating Procedures (SOPs) as described in Exhibit E, Section 6;
- Adhere to the analytical methods in Exhibit D and associated QC requirements specified within Exhibit E;
- Verify and document analytical standards and retain documentation of the purity of neat materials, as well as, the purity and accuracy of solutions obtained from private chemical supply houses;

- Submit all raw data and required documentation for Regional review;
- Submit results of all analyzed laboratory evaluation samples, and adhere to corrective action procedures;
- Submit, upon request, instrument data tapes and applicable documentation for tape audits, including a copy of the Sample Data Package;
- Submit to on-site laboratory evaluations, and adhere to corrective action procedures; and
- Submit all original documentation generated during sample analyses for USEPA review.

3.0 GENERAL QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) REQUIREMENTS

The Contractor shall adhere to USEPA's Good Laboratory Practices for laboratory cleanliness with regard to glassware and apparatus. The Contractor shall also adhere to good laboratory practices with regard to reagents, solvents, and gases. For additional guidelines regarding these general laboratory procedures, see the Handbook for Analytical Quality Control in Water and Wastewater Laboratories USEPA-600/4-79-019, USEPA Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, September 1982.

4.0 SPECIFIC QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) MONITORING PROCEDURES

4.1 Purpose

4.1.1 The purpose of this document is to provide (1) a uniform set of procedures for the analysis of inorganic constituents of samples, (2) documentation of methods and their performance, and (3) verification of the sample data generated. Although it is impossible to address every analytical situation in one document, this exhibit defines the minimum requirements for each major step relevant to any inorganic analysis.

4.1.2 The primary function of the Contract Laboratory Program (CLP) QA/QC program is the definition of procedures for the evaluation and documentation of analytical methodologies and the reduction and reporting of data. The location and summary of the QA/QC performance based contracting methods can be found in Exhibit E, Section 15, Table 1 - Contract Laboratory Program Quality Assurance Monitoring Plan. The objective is to provide a uniform basis for sample handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. In many instances where methodologies are available, specific QC procedures are incorporated into the method documentation (see Exhibit D).

4.1.3 The QA/QC procedures defined herein shall be used by the Contractor when performing the methods specified in Exhibit D. When QA/QC procedures are specified in Exhibit D, the Contractor shall follow those procedures, in addition to procedures specified here.

4.2 Laboratory Audit and Intercomparison Study Program

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by USEPA. The Contractor shall be required to analyze at least one Quarterly Blind (QB) sample per calendar quarter during the contract period for inorganics.

4.3 Annual Verification of Method Detection Limits (MDLs)

The Contractor shall perform and report annual verification of MDLs by the method specified in Exhibit D, by type, matrix, and model for each instrument used on this contract, to Sample Management Office (SMO), Quality Assurance Technical Support (QATS), and the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) as specified in Exhibit B. All the MDLs shall meet the requirements specified in Exhibit C.

4.4 Quarterly Verification of Linear Ranges/Interelement Correction Factors

The Contractor shall perform and report quarterly verification of linear ranges by the method specified in Exhibit D, by type and model for each instrument used on this contract, to SMO, QATS, and the USEPA Regional CLP PO as specified in Exhibit B. The Contractor shall also report, as specified in Exhibit B, integration times. For Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) methods, the Contractor shall also report, as specified in Exhibit B, wavelengths used and all interelement correction factors.

4.5 Quality Assurance/Quality Control Measurements

4.5.1 In this Exhibit, as well as other places within this Statement of Work (SOW), the term "analytical sample" discusses the required frequency or placement of certain QA/QC measurements. The term "analytical sample" is defined in the glossary, Exhibit G.

4.5.2 In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their associated QA/QC analysis shall be analyzed under the same operating and procedural conditions.

4.5.3 If any QC measurement fails to meet contract criteria, the analytical measurement must not be repeated prior to taking the appropriate corrective action as specified in Exhibit D. The exception is the CRI analysis, which may be re-analyzed once before corrective action is necessary.

4.5.4 The Contractor shall report all QC data in the exact format specified in Exhibits B and H.

4.5.5 MDLs, precision, linear dynamic range, and interference effects shall be established for each analyte on a particular instrument. All reported measurements shall be within the instrumental linear ranges. The Contractor shall maintain QC data confirming instrument performance and analytical results.

5.0 QUALITY ASSURANCE MANAGEMENT PLAN (QAP)

5.1 Introduction

The Contractor shall establish a Quality Assurance (QA) program with the objective of providing sound analytical chemical measurements. This program shall incorporate the Quality Control (QC) procedures, any necessary corrective action, all documentation required during data collection, and the quality assessment measures performed by management to ensure acceptable data production. The Contractor shall follow the USEPA EPA Requirements for Quality Management Plans (EPA QA/R-2). An electronic version can be found at http://www.epa.gov/quality1/qa_docs.html.

5.1.1 The Contractor shall prepare a written QAP which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and usability;
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility;
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable; and
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

5.1.2 The QAP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA/QC activities designed to achieve the data quality requirements in this contract. Standard Operating Procedures (SOPs) pertaining to each element shall be included or referenced as part of the QAP. The QAP shall be paginated consecutively in ascending order. The QAP shall be available during on-site laboratory evaluations and shall be submitted to the designee within 7 days of written request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA OSRTI Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM). Additional information relevant to the preparation of a QAP can be found in USEPA and ASTM publications.

5.2 Required Elements of a Quality Assurance Management Plan

The required elements of a laboratory's QAP are outlined in this section. This outline shall be used as a framework for developing the QAP.

A. Organization and Personnel

1. QA Policy and Objectives (the mission and quality policy of the organization)
2. QA Management (the specific roles, authorities, and responsibilities of management and staff with respect to QA and QC activities)
   a. Organization
   b. Assignment of QA/QC Responsibilities

c. Reporting Relationships (the means by which effective communications with personnel actually performing the work are assured)

d. QA Document Control Procedures

e. QA Program Assessment Procedures (the process used to plan, implement, and assess the work performed)

3. Personnel

a. Resumes

b. Education and Experience Pertinent to this Contract

c. Training Records and Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives

2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy

2. Sample Tracking/Custody Procedures

3. Logbook Maintenance and Archiving Procedures

4. Sample Delivery Group (SDG) File Organization, Preparation, and Review Procedures

5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs

6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency

2. Sample Preparation Procedures

3. Sample Analysis Procedures

4. Standards Preparation Procedures

5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures

2. Data Reduction Procedures

3. Data Validation Procedures

4. Data Reporting and Authorization Procedures

F. Quality Assurance (the process which measures the effectiveness of QA will be established and how frequently effectiveness will be measured)

1. Data Quality Assurance

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures (the continual improvement based on lessons learned from previous experience)

5. QA Reporting Procedures

6. Responsibility Designation

G. Quality Control

1. Solvent, Reagent, and Adsorbent Check Analysis

2. Reference Material Analysis

3. Internal QC Checks

4. Corrective Action and Determination of QC Limit Procedures

5. Responsibility Designation

5.3 Updating and Submitting the Quality Assurance Management Plan

5.3.1 The revised QAP will become the official QAP under the contract and may be used during legal proceedings. The Contractor shall maintain the QAP on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised QAP shall be paginated consecutively in ascending order. The revised QAP shall include:

- Changes resulting from (1) the Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures, and (2) the Contractor's implementation of the requirements of the contract, and

- Changes resulting from USEPA's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the pre-award on-site laboratory evaluation.

5.3.1.1 The Contractor shall send a copy of the latest version of the QAP within 7 days of a request from a USEPA Regional CLP PO or the USEPA OSRTI ASB PM. The request will designate the recipients.

5.3.2 Subsequent Updates and Submissions. During the term of the contract, the Contractor shall amend the QAP when the following circumstances occur:

- USEPA modifies the technical requirements of the Statement of Work (SOW) or contract;

- USEPA notifies the Contractor of deficiencies in the QAP document;

- USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;

- The Contractor's organization, personnel, facility, equipment, policy, or procedures change; or

- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, or procedures changes.

5.3.2.1 The Contractor shall amend the QAP within 14 days of when the circumstances listed in Exhibit E, Section 5.3, result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility. When the QAP is amended, all changes in the QAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, highlighting the change by underlining the change, bold printing the change, or using a different print font) and a copy is sent to the USEPA Regional CLP PO and Quality Assurance Technical Support (QATS). The amended section pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the latest version of the QAP document. The Contractor shall archive all amendments to the QAP document for future reference by USEPA.

5.4 Incentives/Sanctions

The Contractor shall amend the QAP as specified within this section. The QAP describes the policies and procedures for ensuring that work processes, products, or services satisfy expectations or specifications in ILM05.3. Failure to comply with the requirements of this section may result in sanctions as described in the contract.

6.0 STANDARD OPERATING PERFORMANCE STANDARDS

6.1 Introduction

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by USEPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks. The Contractor shall follow the USEPA Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents (EPA QA/G-6). An electronic version can be found at http://www.epa.gov/quality1/qa_docs.html.

6.1.1 SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts). The SOPs shall be paginated consecutively in ascending order.

6.1.2 All SOPs shall reflect Contractor activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current USEPA regulations, guidelines, and the Contract Laboratory Program (CLP) ILM05.3 contract requirements.
- Consistent with instrument(s) manufacturer’s specific instruction manuals.
- Available to USEPA during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel shall demonstrate the application of the SOPs if requested.
- Available to the designated recipients within 7 days, upon request by the USEPA Regional CLP Project Officer (CLP PO) or the USEPA OSRTI Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM).
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Capable of demonstrating the validity of data reported by the Contractor and explain the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.
- Available at specific work stations as appropriate.

- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

- Reviewed and signed by all Contractor personnel performing actions identified in the SOP.

6.2 Format

The format for SOPs may vary depending upon the type of activity for which they are prepared; however, at a minimum, the following sections shall be included:

- Title page;
- Document Control;
- Scope and Applicability;
- Summary of Method;
- Definitions (acronyms, abbreviations, and specialized forms used in the SOP);
- Health & Safety;
- Personnel Qualifications;
- Interferences;
- Apparatus & Materials (list or specify; note also designated locations where found);
- Handling & Preservation;
- Instrument or Method Calibration;
- Sample Preparation and Analysis;
- Data Calculations;
- Quality Control (QC) limits;
- Corrective action procedures, including procedures for secondary review of information being generated;
- Data Management and Records Management;
- Miscellaneous notes and precautions; and
- References.

6.3 Required SOPs

The Contractor shall maintain the following SOPs:

6.3.1 Evidentiary SOPs for required chain-of-custody and document control are discussed in Exhibit F.

6.3.2 Sample Receipt and Storage

- Sample receipt and identification logbooks,
- Refrigerator temperature logbooks, and
- Security precautions.

6.3.3 Sample Preparation

6.3.3.1 Metals

6.3.3.2 Cyanide

6.3.4 Glassware Cleaning

6.3.5 Calibration (Balances, etc.)

- Procedures;
- Frequency requirements;
- Preventative maintenance schedule and procedures;
- Acceptance criteria and corrective actions; and
- Logbook maintenance authorization.

6.3.6 Analytical Procedures (for each analytical system)

- Instrument performance specifications;
- Instrument operating procedures;
- Data acquisition system operation;
- Procedures when automatic quantitation algorithms are overridden;
- QC required parameters;
- Analytical run/injection logbooks; and
- Instrument error and editing flag descriptions and resulting corrective actions.

6.3.7 Maintenance Activities (for each analytical system)

- Preventative maintenance schedule and procedures,
- Corrective maintenance determinants and procedures, and
- Maintenance authorization.

6.3.8 Analytical Standards

- Standard coding/identification and inventory system;
- Standards preparation logbook(s);
- Standard preparation procedures;

- Procedures for equivalency/traceability analyses and documentation;
- Purity logbook (primary standards and solvents);
- Storage, replacement, and labeling requirements; and
- QC and corrective action measures.

6.3.9 Data Reduction Procedures

- Data processing systems operation;
- Outlier identification methods;
- Identification of data requiring corrective action; and
- Procedures for format and/or forms for each operation.

6.3.10 Documentation Policy/Procedures

- Contractor/analyst's notebook policy, including review policy;
- Complete Sample Delivery Group (SDG) File (CSF) contents;
- Complete SDG File organization and assembly procedures, including review policy; and
- Document inventory procedures, including review policy.

6.3.11 Data Validation/Self-Inspection Procedures

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to assure that hardcopy and electronic deliverables are complete and compliant with the requirements in the Statement of Work (SOW) Exhibits B and H;
- Procedures to assure that hardcopy deliverables are in agreement with their comparable electronic deliverables;
- Demonstration of internal Quality Assurance (QA) inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal laboratory evaluation samples, etc.);
- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

6.3.12 Data Management and Handling

- Procedures for controlling and estimating data entry errors;
- Procedures for reviewing changes to data and deliverables and ensuring traceability of updates;
- Lifecycle management procedures for testing, modifying, and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems;
- Database security, backup, and archival procedures including recovery from system failures;
- System maintenance procedures and response time;
- Individual(s) responsible for system operation, maintenance, data integrity, and security; and
- Specifications for staff training procedures.

6.4 Updating and Submitting SOP Requirements

6.4.1 The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings. The Contractor shall maintain the complete set of SOPs on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

- Changes resulting from (1) the Contractor's internal review of their procedures and (2) the Contractor's implementation of the requirements of the contract, and
- Changes resulting from USEPA's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the pre-award on-site laboratory evaluation.

6.4.1.1 The Contractor shall send a complete set of the latest version of SOPs or individually requested SOPs within 7 days of a request from an USEPA Regional CLP PO or the USEPA OSRTI ASB PM. The request will designate the recipients.

6.4.2 Subsequent Updates and Submissions. During the term of the contract, the Contractor shall amend the SOPs when the following circumstances occur:

- USEPA modifies the technical requirements of the SOW or contract;
- USEPA notifies the Contractor of deficiencies in the SOP documentation;
- USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- The Contractor's procedures change;
- The Contractor identifies deficiencies resulting from the internal review of the SOP documentation; or

- The Contractor identifies deficiencies resulting from the internal review of their procedures.

6.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 14 days of when the circumstances listed in Exhibit E, Section 6.4, result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, highlighting the change by underlining the change, bold printing the change, or using a different print font) and a copy is sent to the USEPA Regional CLP PO and Quality Assurance Technical Support (QATS). The amended/new SOPs shall have the date on which the changes were implemented.

6.4.2.2 When existing SOPs are amended or new SOPs are written, the Contractor shall document the reasons for the changes and maintain the amended SOPs or new SOPs on file. Documentation of the reasons for the changes shall be maintained on file with the amended SOPs or new SOPs.

6.4.2.3 Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs.

6.5 Incentives/Sanctions

The Contractor shall amend SOPs as specified within this section. The SOPs specify analytical procedures in greater detail than appear in Exhibit D. Adherence to these requirements will ensure that the procedure is conducted in a standard, reliable, and reproducible process described in ILM05.3. Failure to comply with the requirements specified herein may result in sanctions as described in the contract.

7.0 CONTRACT COMPLIANCE SCREENING (CCS) PERFORMANCE STANDARDS

7.1 Overview

7.1.1 CCS is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the Sample Data Package delivered to USEPA.

7.1.2 CCS is performed by the Sample Management Office (SMO) under the direction of USEPA. To assure a uniform review, a set of standardized procedures has been developed to evaluate the Sample Data Package submitted by a Contractor against the technical and completeness requirements of the contract. USEPA reserves the right to add and/or delete individual checks.

7.2 CCS Results

CCS results are distributed to the Contractor and other data recipients. The Contractor has 4 business days to correct deficiencies and shall send all corrections to the Regional client and SMO. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

7.3 CCS Trend Report

USEPA will periodically generate a CCS trend report which summarizes CCS results over a given period of time. USEPA will send the CCS trend report or discuss the CCS trend report during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and USEPA Contracting Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation.

7.4 Incentives/Sanctions

7.4.1 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

7.4.2 The Contractor shall correct deficiencies and resubmit the data within 4 business days, as specified within this section. Resubmission and correction of the data will ensure that the end user is reviewing contractually compliant data described in ILM05.3. Correct resubmission of the data may also result in a reduction in overall sanctions. Specific details on incentives can be found in the contract. If the Contractor fails to adhere to the requirements listed in this section, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

8.0 ANALYTICAL PERFORMANCE STANDARDS REQUIREMENTS

8.1 Overview

USEPA will not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories shall be required to prepare from materials or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

8.2 Preparation of Chemical Standards from the Neat High Purity Bulk Material

8.2.1 If the laboratory cannot obtain analytical reference standards, the laboratory may prepare their own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards; standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

8.2.2 The chemical standards shall be kept at manufacturer recommended conditions when not being used in the preparation of standard solutions. Proper storage of chemicals is essential in order to safeguard them from decomposition.

8.2.3 The Contractor shall be responsible for having analytical documentation proving the purity of each compound as stated. Purity confirmation, when performed, shall use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

EQ. 1 Weight of Impure Compound

$$\text{weight of impure compound} = \frac{\text{weight of pure compound}}{(\text{percent purity}/100)}$$

where “weight of pure compound” is that required to prepare a specific volume of a solution standard of a specified concentration.

8.2.4 The Contractor is responsible for obtaining analytical documentation proving that all compounds used in the preparation of solution standards are correctly identified.

8.2.5 Logbooks shall be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person. All solution standards shall be refrigerated, if required, when not in use. All solution standards shall be clearly labeled as to the identity of the analyte or analytes, the standard ID number of the solution, concentration, date prepared, solvent, expiration date of the solution, special storage requirements (if any), and initials of the preparer.

8.3 Purchase of Chemical Standards Already in Solution

Solutions of analytical reference standards can be purchased by Contractors provided the solutions meet the following criteria.

8.3.1 Reference standards shall be accompanied by documentation of the purity confirmation of the material to verify the integrity of the standard solutions.

8.3.2 The quality of reference standards purchased shall be demonstrated statistically and analytically by a method of the supplier's choice. One way this can be demonstrated is to prepare and analyze three solutions: a high standard, a low standard, and a standard at the target concentration (see Sections 8.3.2.1 and 8.3.2.2). The supplier must then demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the Student's t-test in Section 8.3.2.4. If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test in Section 8.3.2.5. Thus, the standard is certified to be within 10% of the target concentration using the equations in Section 8.3.2.6. If the procedure above is used, the supplier must document that the following have been achieved.

8.3.2.1 Two solutions of identical concentration shall be prepared independently from neat materials. An aliquot of the first solution shall be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration 10% greater than the target standard. This is called the "high standard". One further aliquot is taken from the second solution and diluted to a concentration 10% less than the target standard. This is called the "low standard".

8.3.2.2 Six replicate analyses of each standard (a total of 18 analyses) shall be performed in the following sequence: low standard; target standard; high standard; low standard; target standard; high standard; etc.

8.3.2.3 The mean and variance of the six results for each solution shall be calculated:

EQ. 2 Mean

$$\text{MEAN} = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6}$$

EQ. 3 Variance

$$\text{VARIANCE} = \frac{Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2 + Y_5^2 + Y_6^2 - 6(\text{MEAN})^2}{5}$$

The values Y_1, Y_2, Y_3, ..., represent the results of the six analyses of each standard. The means of the low, target, and high standards are designated M_1, M_2, and M_3, respectively. The variances of the low, target, and high standards are designated V_1, V_2, and V_3, respectively. Additionally, a pooled variance, V_p, is calculated.

EQ. 4 Pooled Variance

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + \frac{V_3}{1.21}}{3}$$

If the square root of V_p is less than one percent of M_2, then $M_2^2/10,000$ is to be used as the value of V_p in all subsequent calculations.

8.3.2.4 The test statistic shall be calculated:

EQ. 5 Low and High Standard Test Statistic

$$\text{TEST STATISTIC} = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}}$$

If the test statistic exceeds 2.13, then the supplier has failed to demonstrate a 20% difference between the high and low standards. In such a case, the standards are not acceptable.

8.3.2.5 The test statistic shall be calculated:

EQ. 6 Target Standard Test Statistic

$$\text{TEST STATISTIC} = \frac{\left| M_2 - \left(\frac{M_1}{1.8} \right) - \left(\frac{M_3}{2.2} \right) \right|}{\left(\frac{V_p}{4} \right)^{0.5}}$$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

8.3.2.6 The 95% confidence intervals for the mean result of each standard shall be calculated:

EQ. 7 Low Standard Interval

$$\text{Interval for Low Standard} = M_1 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

EQ. 8 Target Standard Interval

$$\text{Interval for Target Standard} = M_2 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

EQ. 9 High Standard Interval

$$\text{Interval for High Standard} = M_3 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

8.3.2.6.1 These intervals shall not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10% difference in concentrations. In such a case, the standards are not acceptable.

8.3.2.6.2 In any event, the Contractor is responsible for the quality of the standards employed for analyses under this contract.

8.4 Requesting Standards from the USEPA Standards Repository

Solutions of analytical reference materials can be ordered from the USEPA Chemical Standards Repository, depending on availability. The Contractor may place an order for standards only after demonstrating that these standards are not available from commercial vendors, either in solution or as a neat material.

8.5 Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of the Contractor to maintain the necessary documentation to show that the chemical standards it has used in the performance of Contract Laboratory Program (CLP) analysis conform to the requirements previously listed.

8.5.1 Weighing logbooks, calculations, raw data, etc., whether produced by the Contractor or purchased from chemical supply houses, shall be maintained by the Contractor and may be subject to review during on-site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to USEPA, such documentation is to be kept on file by the Contractor for a period of one year.

8.5.2 Upon request by the USEPA Regional CLP Project Officer (CLP PO), the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of the receipt of request to the designated recipients.

8.5.3 USEPA will periodically generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards. USEPA will send the report or discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO and CLP Quality Assurance Coordinator, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation.

8.5.4 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

8.6 Incentives/Sanctions

The Contractor shall obtain the highest purity possible when purchasing chemical standards specified within this section. The use of high purity standards will ensure a more accurate identification and quantitation of analytes described in the ILM05.3 Statement of Work (SOW). Failure to meet the requirements set forth in this section may result in sanctions as described in the contract.

9.0 DATA PACKAGE MONITORING AUDITS

9.1 Overview

Data package audits are performed by USEPA for program overview and specific Regional concerns. Standardized procedures have been established to assure uniformity of the auditing process. Data packages are periodically selected from recently received Cases. They are evaluated for the technical quality of hardcopy raw data, Quality Assurance (QA), and adherence to contractual requirements. This function provides external monitoring of program Quality Control (QC) requirements. Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance. Audits provide USEPA with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. A thorough review of the raw data is completed including: all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements, a check for transcription and calculation errors, a review of the qualifications of the laboratory personnel involved with the Case, and a review of the latest version of all Standard Operating Procedures (SOPs) on file.

9.2 Responding to the Data Package Audit Report

9.2.1 After completion of the data package audit, USEPA will send a copy of the data package audit report to the Contractor or discuss the data package audit report on an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and the USEPA designated recipient, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report.

9.2.2 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

9.3 Incentives/Sanctions

The Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the comments from USEPA, as specified within this section. The data package audits ensure that the policies and procedures identified in this Statement of Work (SOW) meet the requirements of this contract. If the Contractor fails to adhere to the requirements listed in this section, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

10.0 REGIONAL DATA REVIEW MONITORING

10.1 Overview

Contractor data are generated to meet the specific needs of USEPA Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end user, based on functional guidelines for data review which have been developed jointly by the Regions and the USEPA OSRTI Analytical Services Branch (ASB). Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the Regional response appropriate to the specific circumstances.

10.1.1 Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review done at the Sample Management Office (SMO), which is designed to identify contractual discrepancies, and the review done by the USEPA OSRTI ASB, which is designed to evaluate Contractor and method performance.

11.0 QUALITY ASSURANCE (QA) PROFICIENCY MONITORING

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor shall participate in USEPA's Proficiency Testing Program. USEPA's Proficiency Testing Program involves the analysis of Case specific Performance Evaluation (PE) samples and Quarterly Blind (QB) Audits. The Contractor's analytical PE samples and QB results will be used by USEPA to assess and verify the Contractor's continuing ability to produce acceptable analytical data in accordance with the contractual requirements. The Contractor shall receive a passing score of 75% to be in compliance with the contract.

11.1 Performance Evaluation (PE) Samples

11.1.1 The PE sample(s) may be scheduled with the Contractor as frequently as on a Sample Delivery Group (SDG)-by-SDG basis. The PE samples may be sent either by the Regional client or the USEPA OSRTI Analytical Services Branch (ASB). PE samples assist USEPA in monitoring Contractor performance.

11.1.2 PE samples will be provided as either single-blinds (recognizable as a PE sample but of unknown composition), or as double-blinds (not recognizable as a PE sample and of unknown composition). The Contractor will not be informed of either the analytes/parameters or the concentrations in the PE samples.

11.1.3 The Contractor may receive the PE samples as either full volume samples or ampulated/bottled concentrates from USEPA or a designated USEPA Contractor. The PE samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PE samples (i.e., the required dilution of the PE sample concentrate). PE samples are to be digested and analyzed with the rest of the routine samples in the SDG. The Contractor shall prepare and analyze the PE sample using the procedure described in the sample preparation and method analysis sections of Exhibit D. All contract required Quality Control (QC) shall be met. The PE sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.1.4 In addition to PE sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PE sample. When PE sample results are received by USEPA, the PE sample results will be evaluated for correct analytical identification and quantitation. The PE sample evaluation will be provided to the Contractor via coded evaluation sheets, by analyte. USEPA will notify the Contractor of unacceptable performance.

11.2 Quarterly Blind (QB) Audits

11.2.1 A QB Audit is a unique analytical Case containing only PE samples (i.e., referred to as QB samples). The QB samples will be scheduled by the USEPA OSRTI ASB through the Sample Management Office (SMO). QB samples assist USEPA in monitoring Contractor performance.

11.2.2 QB samples will be provided as single-blinds (recognizable as a PE sample but of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PE samples.

11.2.3 The Contractor may receive the QB samples as either full volume samples or ampulated/bottled concentrates from USEPA or a designated USEPA Contractor. The QB samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the QB samples (i.e., the required dilution of the QB sample concentrate). The Contractor shall prepare and analyze the QB samples using the procedure described in the sample preparation and method analysis sections of Exhibit D. All contract required QC shall be met, including spike and duplicate analyses. The QB sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.2.4 In addition to QB sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes included in each QB sample. When QB sample results are received by USEPA, the QB sample results will be scored for correct analytical identification and quantitation. The QB sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte. USEPA will notify the Contractor of unacceptable performance. The Contractor's QB sample performance will be assessed into one of the following three categories:

11.2.4.1 Acceptable, No Response Required: Score greater than or equal to 90%. The data meets most or all of the scoring criteria. No response is required.

11.2.4.2 Acceptable, Response Explaining Deficiencies Required: Score greater than or equal to 75%, but less than 90%. Deficiencies exist in the Contractor's performance. Corrective action response required.

11.2.4.3 Unacceptable Performance, Response Explaining Deficiencies Required: Score less than 75%. Corrective action response required.

11.2.5 In the case of Section 11.2.4.2 or 11.2.4.3, the Contractor shall describe the deficiency(ies) and the action(s) taken in a corrective action letter to the USEPA Contracting Officer, USEPA Regional Contract Laboratory Program Project Officer (CLP PO), and CLP Quality Assurance (QA) Coordinator within 14 days of receipt of notification from USEPA.

11.2.6 In the case of Section 11.2.4.2 or 11.2.4.3, if new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

11.2.7 The Contractor shall be notified by the USEPA Contracting Officer concerning agreement or disagreement with the proposed remedy for unacceptable performance.

11.2.8 A Remedial QB Audit is a unique analytical Case containing only QB samples. A Remedial QB Audit may be scheduled by the USEPA OSRTI ASB with the Contractor(s) for any of the following reasons: unacceptable PE sample performance, unacceptable QB sample performance, and/or major change in the laboratory (e.g., relocation, new owner, or high turn-over of key personnel). Sections 11.2.2 through 11.2.7 apply to the Remedial QB Audit process.

11.3 Incentives/Sanctions

The Contractor shall analyze PE and QB samples with acceptable analytical results in accordance with the contractual requirements as described in this section. If the Contractor fails to adhere to the requirements listed in this section, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

12.0 ON-SITE LABORATORY QUALITY ASSURANCE (QA) MONITORING EVALUATIONS

12.1 Overview

The USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA Contracting Officer's authorized representative will conduct an on-site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: Quality Assurance (QA) Evaluation and Evidentiary Audit.

12.2 Quality Assurance On-Site Evaluation

QA evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity, experience and education of personnel, and the acceptable performance of analytical and Quality Control (QC) procedures for adherence to the contract requirements.

12.2.1 The Contractor shall expect that items to be monitored will include, but are not limited to, the following:

- Size, cleanliness, and organization of the facility;
- Quantity, age, availability, scheduled maintenance, and performance of instrumentation;
- Availability, appropriateness, and utilization of the Quality Assurance Management Plan (QAP) and Standard Operating Procedures (SOPs);
- Staff qualifications, experience, and personnel training programs;
- Analysis of Performance Evaluation (PE) sample(s);
- Reagents, standards, and sample storage facilities;
- Standard preparation logbooks and raw data;
- Bench sheets and analytical logbook maintenance and review; and
- Review of the Contractor's sample analysis/data package inspection/data management procedures.

12.2.2 Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated into a profile package for discussion during the evaluation. Items that may be included are: previous on-site reports; Quarterly Blind (QB) and/or PE sample scores results; Regional review of data; Contractor performance information provided by the Region; data audit reports; results of Contract Compliance Screening (CCS); and data trend reports.

12.3 Evidentiary Audit

Evidence auditors conduct an on-site laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. The evidence audit comprises a procedural audit, an audit of written SOPs, and an audit of analytical project file documentation.

12.3.1 Procedural Audit. The Contractor shall perform analysis of PE sample(s) in the presence of the USEPA designated team during the procedural audit. The procedural audit will be comprised of everything from sample receipt to data package assembly and completion. This includes the review and examination of actual SOPs and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), analytical project file organization and assembly, and proper disposal of samples and cogenerated wastes.

12.3.2 Written SOPs Audit. The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

12.3.3 Analytical Project File Evidence Audit. The analytical project file evidence audit consists of review and examination of the analytical project file documentation. The auditors review the files to determine:

- The accuracy of the document inventory;
- The completeness of the file;
- The adequacy and accuracy of the document numbering system;
- Traceability of sample activity;
- Identification of activity recorded on the documents; and
- Error correction methods.

12.4 Discussion of the On-Site Team's Findings

The QA and evidentiary auditors discuss their findings with the USEPA Regional CLP PO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel. A report which discusses deficiencies found during the on-site audit will be sent to the Contractor to provide further clarification of findings. In a detailed letter to the USEPA Regional CLP PO and CLP Quality Assurance Coordinator, the Contractor shall discuss the deficiencies and the subsequent corrective actions implemented by the Contractor to resolve the deficiencies within 14 days of receipt of report or the on-site laboratory evaluation.

12.4.1 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

12.5 Incentives/Sanctions

The Contractor shall submit to on-site evaluations, as specified within this section. The on-site evaluations ensure that the policies and procedures identified in this Statement of Work (SOW) meet the requirements of this contract. If the Contractor fails to adhere to the requirements listed in this section, the Contractor will be in

noncompliance with the contract and may be subjected to sanctions as described in the contract.

13.0 ELECTRONIC DATA QUALITY ASSURANCE (QA) MONITORING AUDITS

13.1 Overview

Periodically, USEPA requests the instrument electronic data from Contractors for a specific Case in order to accomplish electronic data audits. Generally, electronic data submissions and audits are requested for the following reasons.

- Program overview;
- Indication of data quality problems;
- Support for on-site audits; and
- Specific Regional requests.

13.1.1 Depending upon the reason for an audit, the instrument electronic data from a recent Case, a specific Case, or a laboratory evaluation sample may be requested. Electronic data audits provide a mechanism to assess adherence to contractual requirements and to ensure the consistency of data reported on the hardcopy/electronic deliverables with that generated on analytical instruments. This function provides external monitoring of Program Quality Control (QC) requirements and checks adherence of the Contractor to internal Quality Assurance (QA) procedures. In addition, electronic data audits enable USEPA to evaluate the utility, precision, and accuracy of the analytical methods.

13.1.2 The Contractor shall store <u>all</u> raw and processed electronic analytical data in the appropriate instrument manufacturer's format, uncompressed, and with no security codes. The data shall include all necessary data files for a complete reconstruction of the previously submitted hardcopy and electronic deliverable data package. All associated raw data files in the instrument manufacturer proprietary software format must be submitted if those files contain data or instrumental parameters regarding any analysis and or correction applied to an instrument or analytical result. This instrument electronic data shall include data for all samples and all QC samples, including but not limited to: blanks, matrix spikes, post-digestion spikes, analytical spikes, duplicates, serial dilutions, Laboratory Control Samples (LCSs), Contract Required Quantitation Limits (CRQL) Check Standards (CRIs), Interference Check Samples (ICSs), tunes, initial calibrations and verifications, and Continuing Calibration Verifications (CCVs). In addition, the Contractor shall supply raw data for the Method Detection Limit (MDL) studies and Linear Range Analyses (LRS) which are used to set the MDL and LRV values for the year/quarter in which the Sample Delivery Group (SDG) was analyzed. The Contractor shall maintain a reference logbook of data files of EPA sample number, calibration data, standards, blanks, spikes, and duplicates. The logbook shall include EPA sample numbers, identified by Case and SDG.

13.1.3 The Contractor is required to retain the instrument electronic data for three years after submission of the reconciled Complete SDG File. Electronic media shipped to the USEPA designated recipient must be fully usable by the recipient. Diskettes must be 3.5 inch, high density, 1.44 MB MS/DOS formatted and tapes must be either 4 mm or 8 mm. Alternative means for delivery of electronic data may be utilized

by the Contractor upon prior written approval by USEPA. When submitting electronic instrument data to USEPA, the following materials shall be delivered in response to the request.

13.1.3.1 All associated raw data files for all analytical samples and all QC samples. For example, files for ICP should include raw intensities and mercury and cyanide files should include raw absorbances or integrated areas.

13.1.3.2 All processed data files and quantitation output files associated with the raw data files described in Section 13.1.3.1.

13.1.3.3 All associated identification and calculation files used to generate the data submitted in the data package. This includes, but is not limited to, result files, acquisition files, calibration files, and method files.

13.1.3.4 All Contractor-generated Inductively Coupled Plasma - Atomic Emission Spectrophotometer (ICP-AES)/ICP - Mass Spectrophotometer (ICP-MS) interference correction files must be submitted.

13.1.3.5 A copy of the Contractor's reference logbook relating data files to EPA sample number, calibration data, standards, blanks, spikes, and duplicates. The logbook shall include EPA sample numbers and laboratory file identifiers for all samples, blanks, and standards, identified by Case and SDG.

13.1.3.6 A printout of the directory of all files in each directory, including all subdirectories and the files contained therein.

13.1.3.7 A copy (hardcopy) of the completed Sample Data Package.

13.1.3.8 A statement attesting to the completeness of the electronic instrument data submission, signed and dated by the Contractor's laboratory manager. The Contractor shall also provide a statement attesting that the data reported have not been altered in any way. These statements shall be part of a Cover Sheet that includes the following information relevant to the data submission:

- Contractor name;
- Date of submission;
- Case number;
- SDG number;
- Instrument make and model number for each instrument;
- Instrument operating software name and version number;
- Data software name and version used for acquisition, re-quantitation, and hardcopy/report generation;
- Data system computer;
- System operating software;
- Data system network;
- Data backup software;

- Data backup hardware;
- Media type and volume of data (in MB) backed up; and
- Names and telephone numbers of two Contractor contacts for further information regarding the submission.

13.2 Submission of the Instrument Electronic Data

Upon request of the USEPA Regional Contract Laboratory Program Project Officer (CLP PO), the Contractor shall send the required instrument electronic data and all necessary documentation to the USEPA designated recipient [e.g., Quality Assurance Technical Support (QATS)] within 7 days of notification.

NOTE: The instrument electronic data shall be shipped according to the procedures in Exhibit F.

13.3 Responding to the Electronic Data Audit Report

After completion of the electronic data audit, USEPA will send a copy of the electronic data audit report to the Contractor or may discuss the electronic data audit report at an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the electronic data audit report within 14 days of receipt of the report or the on-site laboratory evaluation.

13.3.1 If new Standard Operating Procedures (SOPs) are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Exhibit E, Section 6.

13.4 Incentives/Sanctions

The Contractor shall submit to electronic data audits and adhere to the requirements specified in this section. Resubmission and correction of electronic data will ensure that the end user is reviewing contractually compliant data described in the ILM05.3 contract. If the Contractor fails to adhere to the requirements listed in this section, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

14.0 DATA MANAGEMENT PERFORMANCE REQUIREMENTS

14.1 Overview

14.1.1 Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage, and security of computer readable data and files. These procedures shall be in written form and contain a clear definition for all databases and files used to generate or resubmit deliverables. Key areas of concern include: system organization (including personnel and security), documentation operations, traceability, and Quality Control (QC).

14.1.2 Data manually entered from hardcopy shall be subject to QC checks and the error rates estimated. Systems should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors. In addition, data entry error rates shall be estimated and recorded on a monthly basis by re-entering a statistical sample of the data entered and calculating discrepancy rates by data element.

14.2 Documenting Data Changes

The record of changes in the form of corrections and updates to data originally generated, submitted, and/or resubmitted shall be documented to allow traceability of updates. Documentation shall include the following for each change.

- Justification or rationale for the change.
- Initials of the person making the change(s). Data changes shall be implemented and reviewed by a person or group independent of the source generating the deliverable.
- Documentation of changes shall be retained according to the schedule of the original deliverable.
- Resubmitted diskettes or other deliverables shall be re-inspected as a part of the laboratory’s internal inspection process prior to resubmission. The entire deliverable, not just the changes, shall be inspected.
- The Laboratory Manager shall approve changes to originally submitted deliverables.
- Documentation of data changes may be requested by laboratory auditors.

14.3 Lifecycle Management Procedures

Lifecycle management procedures shall be applied to computer software systems developed by the Contractor to be used to generate and edit contract deliverables. Such systems shall be thoroughly tested and documented prior to utilization.

14.3.1 A software test and acceptance plan including test requirements, test results and acceptance criteria shall be developed, followed, and available in written form.

14.3.2 System changes shall not be made directly to production systems generating deliverables. Changes shall be made first to a development system and tested prior to implementation.

14.3.3 Each version of the production system will be given an identification number, date of installation, and date of last operation and will be archived.

14.3.4 System and operations documentation shall be developed and maintained for each system. Documentation shall include a user's manual and an operations and maintenance manual.

14.3.5 This documentation shall be available for on-site review and/or upon written request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA OSRTI Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM).

14.4 Personnel Responsibilities

Individual(s) responsible for the following functions shall be identified.

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating and QC.
- Data and system security, backup and archiving.

15.0 TABLES

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit A: Summary of Requirements	Summary of Program Requirements	Performance standards are summarized in Exhibit A, Sections 1.0 through 4.0.	QA monitoring plan is outlined in Exhibit E.
Exhibit B: Reporting and Deliverables Requirements	Reporting and Deliverable Requirements	Performance standards are outlined in Exhibit B, Sections 1.0 through 4.0.	CCS in Exhibit E, Section 7.0, and CADRE will be used to monitor reporting electronic deliverables.
Exhibit C: Inorganic Target Analyte List with Contract Required Quantitation Limits	Target Analyte List with Contract Required Quantitation Limits	Performance standards are outlined in Exhibit C, Section 1.0.	QA monitoring plan is outlined in Exhibit E.
Exhibit D: Analytical Methods	ICP-AES requirements are outlined in Exhibit D, Part A, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Part A, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part A, Section 12.0, and Exhibit E.
	ICP-MS requirements are outlined in Exhibit D, Part B, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Part B, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part B, Section 12.0, and Exhibit E.
	Mercury requirements are outlined in Exhibit D, Part C, Sections 1.0 through 8.0, 14.0 and 15.0.	Performance standards are outlined in Exhibit D, Part C, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part C, Section 12.0, and Exhibit E.
	Cyanide requirements are outlined in Exhibit D, Part D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Part D, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part D, Section 12.0, and Exhibit E.
Exhibit E: Contract Laboratory Program Quality Assurance Monitoring Plan	General QA/QC Requirements	As outlined in Exhibit D, Quality Control sections.	QA Management Plan is outlined in Exhibit E, Section 5.0.

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit E: Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)	Quality Assurance Management Plan	As outlined in Exhibit E, Sections 5.1.1 and 5.1.2, a written QA Management Plan shall be used to ensure acceptable data production of known and documented quality.	USEPA will review and approve the QA Management Plan.
	Standard Operating Procedures	Performance standards are outlined in Exhibit E, Sections 6.0 through 6.4, and must be performed as stated.	SOPs will be reviewed by USEPA during Pre-Award, on-site audits, after modifications are made and randomly, as deemed appropriate.
	Contract Compliance Screening	Performance standards are outlined in Section E.2 of the ILM05.3 IFB and must be performed as stated.	The sample data package will be evaluated against the technical and completeness requirements of the contract.
	Analytical Standards	Performance standards are outlined in Exhibit E, Sections 8.0 through 8.5, and must be performed as stated.	Randomly, USEPA will review analytical standards verification and preparation documentation, as deemed appropriate.
	Data Package Audits	Performance standards are outlined in Exhibit E, Sections 9.0 through 9.2.	Data package audits are performed by USEPA to evaluate technical quality of the hardcopy raw data, QA, and adherence to contractual requirements.
	Regional Data Review	Analytical data is reviewed by each Region from the perspective of the end user to determine the usability of the data, as outlined in Exhibit E, Section 10.0.	Regional validation and/or CADRE reports are generated for all data packages.

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit E: Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)	Proficiency Testing	Performance standards are outlined in Exhibit E, Sections 11.0 through 11.2, and must be performed as stated.	Acceptable QB scores will assist in monitoring contractor performance as defined in Exhibit E, Sections 11.2.4.1 through 11.2.4.3, and 11.2.8.
	On-Site Laboratory Evaluations	Performance standards are outlined in Exhibit E, Sections 12.0 through 12.4.	USEPA will evaluate the results from quality assurance and evidentiary on-site audits as defined in Exhibit E, Sections 12.2.1 through 12.3.3, to assist in monitoring the contractor.
	Electronic Data Audits	Performance standards are outlined in Exhibit E, Sections 13.0 through 13.3.	CCS in Exhibit E, Section 7.0, will be used to monitor electronic deliverables.
	Data Management	Performance standards are outlined in Exhibit E, Sections 14.0 through 14.4, and must be performed as stated.	USEPA will monitor data management practices during quality assurance and evidentiary on-site audits.
Exhibit F: Chain-of-Custody, Document Control and Written Standard Operating Procedures	Standard Operating Procedures	Performance standards are outlined in Exhibit F, Sections 2.0 through 2.7.	SOPs will be reviewed by USEPA during Pre-Award, on-site audits, after modifications are made, and randomly as deemed appropriate.
	Written Standard Operating Procedures	Performance standards are outlined in Exhibit F, Sections 3.0 through 3.7.	SOPs will be reviewed by USEPA during Pre-Award, on-site audits, after modifications are made, and randomly as deemed appropriate.
Exhibit G: Glossary of Terms	Glossary of Terms	Contractors shall adhere to interpretation of SOW terms as defined within Exhibit G.	N/A

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit H: Data Dictionary and Format for Data Deliverables in Computer-Readable Format	Data Dictionary and Format	Performance standards are outlined in Exhibit H and Appendix A.	CCS in Exhibit E, Section 7.0, will be used to monitor electronic deliverables.
Appendix B: Modified Analysis	GFAA requirements are outlined in Appendix B, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Appendix B, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Appendix B, Section 12.0, and Exhibit E.

EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL
AND WRITTEN STANDARD OPERATING PROCEDURES

THIS PAGE INTENTIONALLY LEFT BLANK

Exhibit F - Chain-of-Custody, Document Control and
Written Standard Operating Procedures

Table of Contents

Section		Page
1.0	INTRODUCTION	5
	1.1 Purpose of Evidence Requirements	5
2.0	STANDARD OPERATING PROCEDURES	6
	2.1 Sample Receiving	6
	2.2 Sample Identification	7
	2.3 Sample Security	7
	2.4 Sample Storage	7
	2.5 Sample Tracking and Document Control	8
	2.6 Computer-Resident Sample Data Control	9
	2.7 Complete SDG File (CSF) Organization and Assembly	9
3.0	WRITTEN STANDARD OPERATING PROCEDURES	11
	3.1 Sample Receiving	11
	3.2 Sample Identification	12
	3.3 Sample Security	13
	3.4 Sample Storage	13
	3.5 Sample Tracking and Document Control	13
	3.6 Computer-Resident Sample Data Control	14
	3.7 CSF Organization and Assembly	15

THIS PAGE INTENTIONALLY LEFT BLANK

1.0 INTRODUCTION

A sample is physical evidence collected from a facility or from the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To ensure that U.S. Environmental Protection Agency's (USEPA's) sample data and records supporting sample-related activities are admissible and have weight as evidence in future litigation, Contractors are required to maintain USEPA samples under chain-of-custody and to account for all samples and supporting records of sample handling, preparation, and analysis. Contractors shall maintain sample identity, sample custody, and all sample-related records according to the requirements in this exhibit.

1.1 Purpose of Evidence Requirements

The purpose of the evidence requirements include:

- Ensuring traceability of samples while in possession of the Contractor;
- Ensuring custody of samples while in possession of the Contractor;
- Ensuring the integrity of sample identity while in possession of the Contractor;
- Ensuring sample-related activities are recorded on documents or in other formats for USEPA sample receipt, storage, preparation, analysis, and disposal;
- Ensuring all laboratory records for each specified Sample Delivery Group will be accounted for when the project is completed; and
- Ensuring that all laboratory records directly related to USEPA samples are assembled and delivered to USEPA or, prior to delivery, are available upon USEPA's request.

2.0 STANDARD OPERATING PROCEDURES

The Contractor shall implement the following Standard Operating Procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group (SDG) File (CSF) organization and assembly to ensure accountability of USEPA sample chain-of-custody as well as control of all USEPA sample-related records.

2.1 Sample Receiving

2.1.1 The Contractor shall designate a sample custodian responsible for receiving USEPA samples.

2.1.2 The Contractor shall designate a representative to receive USEPA samples in the event that the sample custodian is not available.

2.1.3 Upon receipt, the condition of shipping containers and sample containers shall be inspected and recorded on Form DC-1 by the sample custodian or a designated representative.

2.1.4 Upon receipt, the condition of the custody seals (intact/broken) shall be inspected and recorded on Form DC-1 by the sample custodian or a designated representative.

2.1.5 The sample custodian or a designated representative shall verify and record on Form DC-1 the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.

2.1.6 The sample custodian or a designated representative shall verify and record the following information on Form DC-1 as samples are received and inspected:

- Presence or absence and condition of custody seals on shipping and/or sample containers;
- Custody seal numbers when present;
- Presence or absence of Traffic Reports/Chain of Custody Records or Packing Lists;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;
- Presence or absence of sample tags;
- Sample tags listed/not listed on Traffic Reports/Chain of Custody Records;
- Condition of the sample bottles;
- Presence or absence of cooler temperature indicator bottle;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;

- pH of all aqueous samples;
- Sample tag numbers;
- Assigned laboratory numbers;
- Remarks regarding condition of sample shipment, etc.;
- Samples delivered by hand; and
- Problems and discrepancies.

2.1.7 The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., Traffic Reports/Chain of Custody Records or packing lists, and airbills).

NOTE: Initials are not acceptable.

2.1.8 The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to: absent documents; conflicting information; absent or broken custody seals; insufficient sample volume; unsatisfactory sample condition (e.g., leaking sample container); and samples not preserved to the proper pH.

2.1.9 The Contractor shall record the resolution of all problems and discrepancies communicated through SMO.

2.2 Sample Identification

2.2.1 The Contractor shall maintain the identity of USEPA samples and prepared samples (including extracted samples, digested samples, and distilled samples) throughout the laboratory.

2.2.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory sample identification number.

2.3 Sample Security

2.3.1 The Contractor shall demonstrate that USEPA sample custody is maintained from receiving through retention or disposal. A sample is in custody if:

- It is in your possession; or
- It is in your view after being in your possession; or
- It is locked in a secure area after being in your possession; or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel).

2.3.2 The Contractor shall demonstrate security of designated secure areas.

2.4 Sample Storage

The Contractor shall designate storage areas for USEPA samples and prepared samples.

2.5 Sample Tracking and Document Control

2.5.1 The Contractor shall record all activities performed on USEPA samples.

2.5.2 Titles which identify the activities recorded shall be printed on each page of all laboratory documents. (Activities include, but are not limited to: sample receipt; sample storage; sample preparation, and sample analysis.) When a document is a record of analysis, the instrument type and parameter group [e.g., ICP-AES (metals)] shall be included in the title.

2.5.3 When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.

2.5.4 Reviewers' signatures shall be identified on laboratory documents when reviews are conducted.

NOTE: Individuals recording review comments on computer-generated raw data are not required to be identified unless the written comments address data validity.

2.5.5 The laboratory name shall be identified on preprinted laboratory documents.

2.5.6 Each laboratory document entry shall be dated with the month/day/year (e.g., 01/01/1999) and signed by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

2.5.7 Notations on laboratory documents shall be recorded in ink.

2.5.8 Corrections to laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.5.9 Unused portions of laboratory documents shall be lined-out.

2.5.10 Pages in bound and unbound logbooks shall be sequentially numbered.

2.5.11 Instrument-specific run logs shall be maintained to enable the reconstruction of run sequences.

2.5.12 Logbook entries shall be in chronological order.

2.5.13 Logbook entries shall include only one SDG per page, except in the events where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs).

2.5.14 Each page in bound and unbound logbooks shall be dated (month/day/year) and signed (no initials) at the bottom by the individual recording the activity (if a single entry is made on a page) or by the last individual recording information on the page (if multiple entries are on the same page).

2.5.15 Information inserted into laboratory documents shall be affixed permanently in place. The individual responsible for inserting information shall sign and date across the insert and logbook page at the time information is inserted.

2.5.16 The Contractor shall document disposal or retention of USEPA samples, remaining portions of samples, and prepared samples.

2.6 Computer-Resident Sample Data Control

2.6.1 Contractor personnel responsible for original data entry shall be identified at the time of data input.

2.6.2 The Contractor shall make changes to electronic data in a manner which ensures that the original data entry is preserved, the editor is identified, and the revision date is recorded.

2.6.3 The Contractor shall routinely verify the accuracy of manually entered data, electronically entered data, and data acquired from instruments.

2.6.4 The Contractor shall routinely verify documents produced by the electronic data collection system to ensure accuracy of the information reported.

2.6.5 The Contractor shall ensure that the electronic data collection system is secure.

2.6.5.1 The electronic data collection system shall be maintained in a secure location.

2.6.5.2 Access to the electronic data collection system functions shall be limited to authorized personnel through utilization of software security techniques (e.g., log-ons or restricted passwords).

2.6.5.3 Electronic data collection systems shall be protected from the introduction of external programs or software (e.g., viruses).

2.6.6 The Contractor shall designate archive storage areas for electronic data and the software required to access the data.

2.6.7 The Contractor shall designate an individual responsible for maintaining archives of electronic data including the software.

2.6.8 The Contractor shall maintain the archives of electronic data and necessary software in a secure location. (Secure areas shall be accessible only to authorized personnel.)

2.7 Complete SDG File (CSF) Organization and Assembly

2.7.1 The Contractor shall designate a document control officer responsible for the organization and assembly of the CSF.

2.7.2 The Contractor shall designate a representative responsible for the organization and assembly of the CSF in the event that the document control officer is not available.

2.7.3 The Contractor shall maintain documents relating to the CSF in a secure location.

2.7.4 All original laboratory forms and copies of SDG-related logbook pages shall be included in the CSF.

2.7.5 Copies of laboratory documents in the CSF shall be photocopied in a manner to provide complete and legible replicates.

2.7.6 Documents relevant to each SDG including, but not limited to, the following shall be included in the CSF:

- logbook pages;
- bench sheets;
- screening records;
- preparation records;
- repreparation records;
- analytical records;
- re-analysis records;
- records of failed or attempted analysis;
- custody records;
- sample tracking records;
- raw data summaries;
- computer printouts;
- correspondence;
- FAX originals;
- library search results; and
- other.

2.7.7 The document control officer or a designated representative shall ensure that sample tags are encased in clear plastic bags before placing them in the CSF.

2.7.8 CSF documents shall be organized and assembled on an SDG-specific basis.

2.7.9 Original documents which include information relating to more than one SDG (e.g., Traffic Reports/Chain of Custody Records, calibration logs) shall be filed in the CSF of the lowest SDG number, and copies of these originals shall be placed in the other CSF(s). The document control officer or a designated representative shall record the following statement on the copies in (indelible) *dark ink*:

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF _____

_____
Signature

_____
Date

2.7.10 All CSFs shall be submitted with a completed Form DC-2. All resubmitted CSFs shall be submitted with a new or revised Form DC-2.

2.7.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall be recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

2.7.12 Before shipping each CSF, the document control officer or a designated representative shall verify the agreement of information recorded on all documentation and ensure that the information is consistent and the CSF is complete.

2.7.13 The document control officer or a designated representative shall document the shipment of deliverable packages including what was sent, to whom, the date, and the carrier used.

2.7.14 Shipments of deliverable packages, including resubmittals, shall be sealed with custody seals by the document control officer or a

designated representative in a manner such that opening the packages would break the seals.

2.7.15 Custody seals shall be signed and dated by the document control officer or a designated representative when sealing deliverable packages.

3.0 WRITTEN STANDARD OPERATING PROCEDURES

The Contractor shall develop and implement the following written Standard Operating Procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group (SDG) File (CSF) organization and assembly to ensure accountability for USEPA sample chain-of-custody and control of all USEPA sample-related records.

3.1 Sample Receiving

3.1.1 The Contractor shall have written SOPs for sample receiving which accurately reflect the procedures used by the laboratory.

3.1.2 The written SOPs for sample receiving shall ensure that the procedures listed below are in use at the laboratory.

3.1.2.1 The condition of shipping containers and sample containers are inspected and recorded on Form DC-1 upon receipt by the sample custodian or a designated representative.

3.1.2.2 The condition of custody seals are inspected and recorded on Form DC-1 upon receipt by the sample custodian or a designated representative.

3.1.2.3 The presence or absence of the following documents/items accompanying the sample shipment is verified and recorded on Form DC-1 by the sample custodian or a designated representative:

- Custody seals;
- Traffic Reports/Chain of Custody Records or Packing Lists;
- Airbills or airbill stickers;
- Sample tags; and
- Cooler temperature indicator bottle.

3.1.2.4 The agreement or disagreement of information recorded on shipping documents with information recorded on sample containers is verified and recorded on Form DC-1 by the sample custodian or a designated representative.

3.1.2.5 The following information is recorded on Form DC-1 by the sample custodian or a designated representative as samples are received and inspected:

- Custody seal numbers, when present;
- Airbill or airbill sticker numbers;
- Sample tag numbers listed/not listed on Traffic Reports/Chain of Custody Records;

- Condition of sample bottles;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;
- pH of all aqueous samples;
- Sample tag numbers;
- Assigned laboratory numbers;
- Remarks regarding condition of sample shipment, etc.;
- Samples delivered by hand; and
- Problems and discrepancies.

3.1.2.6 All accompanying forms are signed, dated, and the time is recorded, when applicable, at the time of sample receipt (e.g., Traffic Reports/Chain of Custody Records or packing lists, and airbills) by the sample custodian or a designated representative.

3.1.2.7 The Sample Management Office (SMO) is contacted to resolve problems and discrepancies including, but not limited to: absent documents; conflicting information; absent or broken custody seals; insufficient sample volume; unsatisfactory sample condition (e.g., leaking sample container); and samples not preserved to the proper pH.

3.1.2.8 The resolution of all problems and discrepancies communicated through SMO is recorded.

3.2 Sample Identification

3.2.1 The Contractor shall have written SOPs for sample identification which accurately reflect the procedures used by the laboratory.

3.2.2 The written SOPs for sample identification shall ensure that the procedures listed below are in use at the laboratory.

3.2.2.1 The identity of USEPA samples and prepared samples is maintained throughout the laboratory when:

- The Contractor assigns unique laboratory sample identification numbers, the written SOPs shall include a description of the procedure used to assign these numbers;
- The Contractor uses prefixes or suffixes in addition to laboratory sample identification numbers, the written SOPs shall include their definitions; and
- The Contractor uses methods to uniquely identify fractions/parameter groups and matrix type, the written SOPs shall include a description of these methods.

3.2.2.2 Each sample and sample preparation container is labeled with the SMO number or a unique laboratory sample identification number.

3.3 Sample Security

3.3.1 The Contractor shall have written SOPs for sample security which accurately reflect the procedures used by the laboratory.

3.3.2 The written SOPs for sample security shall include the items listed below.

3.3.2.1 Procedures which ensure the following:

- Sample custody is maintained; and
- The security of designated secure areas is maintained.

3.3.2.2 A list of authorized personnel who have access to locked storage areas.

3.4 Sample Storage

3.4.1 The Contractor shall have written SOPs for sample storage which accurately reflect the procedures used by the laboratory.

3.4.2 The written SOPs for sample storage shall describe locations, contents, and identities of all storage areas for USEPA samples and prepared samples in the laboratory.

3.5 Sample Tracking and Document Control

3.5.1 The Contractor shall have written SOPs for sample tracking and document control which accurately reflect the procedures used by the laboratory.

3.5.2 The written SOPs for sample tracking and document control shall include the items listed below.

3.5.2.1 Examples of all laboratory documents used during sample receiving, sample storage, sample transfer, sample analyses, CSF organization and assembly, and sample retention or disposal.

3.5.2.2 Procedures which ensure the following:

- All activities performed on USEPA samples are recorded;
- Titles which identify the activities recorded are printed on each page of all laboratory documents;
- Information recorded in columns is identified with column headings;
- Reviewers' signatures are identified on laboratory documents;
- The laboratory name is included on preprinted laboratory documents;
- Laboratory document entries are signed and dated with the month/day/year (e.g., 01/01/1999);
- Entries on all laboratory documents are recorded in ink;
- Corrections and additions to laboratory documents are made by drawing single lines through the errors, entering the correct information, and initialing and dating the new information;

- Unused portions of laboratory documents are lined-out;
- Pages in bound and unbound logbooks are sequentially numbered;
- Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
- Logbook entries are recorded in chronological order;
- Entries are recorded for only one SDG on a page, except in the event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs);
- Each page in bound and unbound logbooks shall be dated (month/day/year) and signed (no initials) at the bottom by the individual recording the activity (if a single entry is made on a page) or by the last individual recording information on the page (if multiple entries are on the same page);
- Information inserted in laboratory documents is affixed permanently, signed, and dated across the insert; and
- The retention or disposal of USEPA samples, remaining portions of samples, and prepared samples is documented.

3.6 Computer-Resident Sample Data Control

3.6.1 The Contractor shall have written SOPs for computer-resident sample data control which accurately reflect the procedures used by the laboratory.

3.6.2 The written SOPs for computer-resident sample data control shall include the items listed below.

3.6.2.1 Procedures which ensure the following:

- Contractor personnel responsible for original data entry are identified;
- Changes to electronic data are made such that the original data entry is preserved, the editor is identified, and the revision date is recorded;
- The accuracy of manually entered data, electronically entered data, and data acquired from instruments is verified;
- Report documents produced by the electronic data collection system are routinely verified to ensure the accuracy of the information reported;
- Electronic data collection system security is maintained;
- Archives of electronic data and accompanying software are maintained in a secure location; and
- Off-site backup and storage of electronic data is maintained.

3.6.2.2 Descriptions of archive storage areas for the electronic data and the software required to access data archives.

3.6.2.3 A list of authorized personnel who have access to electronic data collection system functions and to archived data.

3.7 CSF Organization and Assembly

3.7.1 The Contractor shall have written SOPs for CSF organization and assembly which accurately reflect the procedures used by the laboratory.

3.7.2 The written SOPs for CSF organization and assembly shall ensure that the procedures listed below are in use at the laboratory.

- Documents relating to the CSF are maintained in a secure location.
- All original laboratory forms and copies of SDG-related logbook pages are included in the CSF.
- Laboratory documents are photocopied in a manner to provide complete and legible replicates.
- All documents relevant to each SDG are included in the CSF.
- Sample tags are encased in clear plastic bags by the document control officer or a designated representative before placing them in the CSF.
- The CSF is organized and assembled on an SDG-specific basis.
- Original documents which contain information relating to more than one SDG are filed in the CSF of the lowest SDG and copies are referenced to originals in the event that an original document contains information relating to more than one SDG.
- Each CSF is submitted with a completed Form DC-2, and resubmitted CSFs are submitted with a new or revised Form DC-2.
- Each page of the CSF is stamped with a sequential number and the page number ranges are recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence are recorded in the "Comments" section of Form DC-2. Inserted documents are recorded in the "Other Records" section of Form DC-2.
- Consistency and completeness of the CSF are verified by the document control officer or a designated representative.
- Shipments of deliverable packages are documented by the document control officer or a designated representative.
- Deliverable packages are shipped by the document control officer or a designated representative using custody seals in a manner such that opening the packages would break the seals.
- Custody seals are signed and dated by the document control officer or a designated representative before placing them on deliverable packages.

EXHIBIT G

GLOSSARY OF TERMS

THIS PAGE INTENTIONALLY LEFT BLANK

ABSORBANCE - A measure of the decrease in incident light passing through a sample into a detector. It is defined mathematically as:

Absorbance

$$A = -\log \frac{I}{Io}$$

WHERE,

I = Radiation intensity of a sample.
I_o = Radiation intensity of a blank.

ALIQUOT - A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

ANALYSIS DATE/TIME - The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SAMPLE - Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, initial calibration verification (ICV), initial calibration blank (ICB), continuing calibration verification (CCV), continuing calibration blank (CCB), and tunes. Note the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA), matrix spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, Interference Check Samples (ICSs), Contract Required Quantitation Limit (CRQL) Check Standards (CRIs), Laboratory Control Samples (LCSs), Performance Evaluation (PE) samples, Preparation Blanks (PBs), Linear Range Samples (LRSs), and cyanide MIDRANGE samples.

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB. All sample analyses during the analytical sequence are subject to the QC protocols set forth in Exhibits D and E of this contract unless otherwise specified in the individual methods.

ANALYTICAL SERVICES BRANCH (ASB) - The division of United States Environmental Protection Agency's (USEPA) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

ANALYTICAL SPIKE - A spike that is fortified just prior to analysis by adding a known quantity of the analyte to an aliquot of the prepared sample.

ASTM - American Society for Testing and Materials. A developer and provider of voluntary consensus standards.

AUTOZERO - Zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

BACKGROUND CORRECTION - A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BASELINE - Analysis used to reset the baseline during mercury or cyanide runs.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

CALIBRATION - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation. This blank is not subjected to the preparation method.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

CASE - A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

CONCENTRATION LEVEL (low or medium) - For inorganics analysis, low or medium level is defined by the appropriate designation by the sampler on the Traffic Report/Chain of Custody Record.

CONTAMINATION - A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents laboratory environment, or analytical instruments.

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be run every 10 analytical samples or every 2 hours, whichever is more frequent.

CONTRACT COMPLIANCE SCREENING (CCS) - A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is done under USEPA direction by the SMO Contractor.

CONTRACT LABORATORY PROGRAM (CLP) - Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) of USEPA.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) - A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CYANIDE (Total) - Cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

DATE - MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 1998, 1999, 2000, 2001, etc.

DAY - Unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - An official record of the sample preparation (digestion).

DIRECT ANALYSIS - Analysis of a sample, standard, or blank that has not been taken through a preparation procedure (digestion or distillation).

DISSOLVED METALS - Analyte elements in a water/aqueous sample which will pass through a 0.45 micrometer (µm) filter.

DRY WEIGHT - The weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

FIELD BLANK - This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

FIELD QC - Any Quality Control sample submitted from the field to the laboratory. Examples include, but are not limited to: field blanks, field duplicates, and field spikes.

FIELD SAMPLE - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA sample number.

GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) - A technique for the determination of analytes in which a sample aliquot is injected into a hollow graphite tube, which is then heated to atomize the analyte. The vapor absorbs light at wavelengths characteristic of the element(s) atoms present.

HOLDING TIME - The elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis.

Holding time = (sample analysis date - sample receipt date)

INDEPENDENT STANDARD - A Contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the calibration.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES) - A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

INDUCTIVELY COUPLED PLASMA-MASS SPECTROSCOPY (ICP-MS) - A technique for the multi-element determination of elements in solution. The basis of the technique is the detection of atomic ions produced by an ICP and sorted by mass/charge ratio.

IN-HOUSE - At the Contractor's facility.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to NIST or other certified standard sources when USEPA ICV solutions are not available.

INJECTION - Introduction of the analytical sample into the instrument excitation system for the purpose of measuring absorbance, emission or concentration of an analyte. May also be referred to as exposure.

INSUFFICIENT QUANTITY - When there is not enough volume (water/aqueous sample) or weight (soil/sediment) to perform any of the required operations: sample analysis, percent solids, etc. Exhibit D provides guidance for addressing this problem.

INTERFERENCE CHECK SAMPLE - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INTERFERENTS - Substances which affect the analysis for the element of interest.

INTERNAL STANDARD - A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

LABORATORY - Synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition. Laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.

LABORATORY RECEIPT DATE - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record. Also referred to as VTSR (Validated Time of Sample Receipt).

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the instrument response remains linear.

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water/ aqueous or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - In general, the effect of particular matrix constituents.

MATRIX SPIKE - Aliquot of a sample (water/aqueous or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

MIDRANGE - A distilled standard at a concentration approximately equivalent to the midpoint of the calibration curve used to verify the reliability of the distillation procedure.

NARRATIVE (SDG Narrative) - Portion of the data package which includes laboratory, contract, Case, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete SDG Narrative specifications are included in Exhibit B.

PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values. The difference between the two values divided by one of the values.

PERCENT SOLIDS (%S) - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

PERFORMANCE EVALUATION (PE) SAMPLE - A sample of known composition provided by USEPA for Contractor analysis. Used by USEPA to evaluate Contractor performance.

PREPARATION BLANK - An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

PREPARATION LOG - An official record of the sample preparation (digestion or distillation).

QUALITY ASSURANCE TECHNICAL SUPPORT (QATS) LABORATORY - A Contractor-operated facility operated under the QATS contract, awarded and administered by USEPA.

REAGENT WATER - The purity of this water must be equivalent to ASTM Type II reagent water of Specification D1193-77, "Standard Specification for Reagent Water".

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

REPRESENTATIVE - Alternate or designee who has the knowledge and authority to perform a specific task.

RESLOPE - An analysis used to re-align the calibration curve during mercury or cyanide runs.

ROUNDING RULES - If the figure is greater than or equal to 5, round up, otherwise round down. As an example, 11.443 is rounded down to 11.44 and 11.455 is rounded up to 11.46. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures. See forms instructions (Exhibit B) for exceptions.

RUN - A continuous analytical sequence consisting of prepared samples and all associated Quality Assurance (QA) measurements as required by the contract SOW. A run begins with the instrument calibration and is to be completed within a 24-hour period.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received, or
- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil samples in one SDG, all water samples in another) at the discretion of the laboratory.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by USEPA.

SAMPLE NUMBER (EPA SAMPLE NUMBER) - A unique identification number designated by USEPA for each sample. The EPA sample number appears on the sample Traffic Report/Chain of Custody Record which documents information on that sample.

SENSITIVITY - The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

SOIL - Synonymous with soil/sediment or sediment as used herein.

SOP - Standard Operating Procedure.

SOW - Statement of Work.

STANDARD ANALYSIS - An analytical determination made with known quantities of target analytes.

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

TARGET ANALYTE LIST (TAL) - A list of Inorganic Analytes (metals and cyanide) as designated in Exhibit C.

TIME - When required to record time on any deliverable item, time shall be expressed as Military Time [i.e., a 24-hour clock (0000-2359)].

TRAFFIC REPORT/CHAIN OF CUSTODY RECORD (TR/COC) - An USEPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and is used for documenting sample identity, sample chain-of-custody, and sample receipt by the laboratory.

TUNE - Analysis of a solution containing a range of isotope masses to establish ICP-MS accuracy, resolution, and precision prior to calibration.

USEPA OSRTI ASB INORGANIC PROGRAM MANAGER (ASB PM) - The USEPA, OSRTI ASB Official who manages the CLP Inorganic Program.

USEPA REGIONAL CLP PROJECT OFFICER (CLP PO) - The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record.

WET WEIGHT - The weight of a sample aliquot including moisture (undried).

10% FREQUENCY - A frequency specification during an analytical sequence allowing for no more than 10 analytical samples between required calibration verification measurements, as specified by the contract SOW.

EXHIBIT H

DATA DICTIONARY AND FORMAT FOR DATA DELIVERABLES IN COMPUTER-READABLE FORMAT

THIS PAGE INTENTIONALLY LEFT BLANK

Exhibit H - Data Dictionary and Format for Data Deliverables in Computer-Readable Format

Table of Contents

Section		Page
1.0	USEPA AGENCY STANDARD IMPLEMENTATION	5
1.1	Format Characteristics	5
2.0	RECORD TYPES	6
2.1	Specifications	6
3.0	PRODUCTION RUNS	7
3.1	Specifications	7
3.2	Example	7
4.0	RECORD SEQUENCE	10
4.1	Specifications	10
5.0	FILE/RECORD INTEGRITY	12
6.0	DATES AND TIMES	12
7.0	MULTIPLE VOLUME DATA	12
8.0	DELIVERABLE	13
8.1	Requirements	13
9.0	RECORD LISTING	14
9.1	Production Run First Header Record (Type 10)	14
9.2	Production Run Second Header Record (Type 16)	15
9.3	Mandatory Sample Header Data Record (Type 20)	16
9.4	Sample Header Record (Type 21)	21
9.5	Associated Injection and Counter Record (Type 22)	23
9.6	Results Data Record (Type 30)	24
9.7	Instrumental Data Readout (Type 31)	27
9.8	Auxiliary Data Record (Type 32)	28
9.9	QC Limit Record (Type 34)	29
9.10	Correction Data Record (Type 35)	30
9.11	Comment Record (Type 90)	31
9.12	Sample Associated Data Record (Type 92)	32

THIS PAGE INTENTIONALLY LEFT BLANK

1.0 USEPA AGENCY STANDARD IMPLEMENTATION

1.1 Format Characteristics

The following constitutes an implementation of the USEPA Agency Standard for Electronic Data Transmission based upon analytical results and ancillary information required by the contract. All data generated by a single analysis are grouped together, and the groups are aggregated to produce files that report data from a Sample Delivery Group (SDG). Because this implementation is only a subset of the USEPA Agency Standard, some fields have been replaced by delimiters as place holders for non-Contract Laboratory Program (CLP) data elements.

1.1.1 This implementation includes detailed specifications for the required format of each record. The position in the record where each field is to be contained relevant to other fields is specified, as well as the maximum length of the field. Each field's required contents are specified as literal (contained in quotes), which must appear exactly as shown (without quotes), or as a variable for which format and/or descriptions are listed in the format/contents column. Options and examples are listed for most fields. For fields where more than three options are available, a list and description of options are supplied following the record descriptions. Fields are separated from each other by the delimiter "|" (ASCII 124). Fields that do not contain data should be zero length or a blank field (empty with no space or additional delimiters between the delimiters before and after the field) with the delimiter as a place holder. For the purposes of Section 9 of this Exhibit, wherever "blank" is given as an option under the "Format/Contents" column, it refers to a blank field as explained above.

1.1.2 Numeric fields may contain numeric digits, a decimal place, and a leading minus sign. A positive sign is assumed if no negative sign is entered in a numeric field and must not be entered into any numeric field. Values that exceed the maximum length allowed shall be reported to the maximum possible, maintaining the specified decimal place and maximum field length restrictions.

1.1.3 Requirements for significant figures and number of decimal places are specified in Exhibit B. The numeric field lengths are specified such that all possible numeric values can be written to the file. The size of the numeric field indicates the maximum number of digits, including a decimal place and negative sign, if appropriate, that can appear in the field at the same time. Therefore, the number reported may need to be rounded (using rounding rules described in Exhibit B) to fit into the field. The rounding must maintain the greatest significance possible providing the field length limitation. In addition, the rounded number that appears on the form, and therefore the field in the diskette file, must be used in any calculation that may result in other numbers reported on the same form or other forms in the SDG. Field lengths should only be as long as necessary to contain the data; packing with blanks is not allowed.

1.1.4 USEPA is currently developing a data delivery strategy that may be used as an alternative to the requirements stated in Exhibit H. This strategy's intent is to provide a neutral data delivery structure to the Contractor that will further facilitate the exchange of analytical information generated under this analytical protocol. The proposed strategy is intended to accommodate laboratories that generate data transmission files under multiple data formats. Upon implementation of this alternate electronic data delivery strategy by the USEPA and prior to submission of data in alternate format(s), the

Contractor must first demonstrate its ability to provide electronic data as stated in this Exhibit H and obtain written permission from the USEPA for the submission of data in alternate format(s). The Contractor will receive a written response to its request within 90 calendar days. However, until the implementation of this alternate electronic data delivery strategy by the USEPA, all electronic data deliverables must be provided as specified in this Exhibit H.

2.0 RECORD TYPES

2.1 Specifications

The USEPA Agency Standard consists of variable length ASCII records. Maximum field length specifications match the reporting requirements in Exhibit B. The last two bytes of each record must contain “carriage return” and “line feed”, respectively.

2.1.1 This implementation consists of twelve record types that can be summarized in four groups, designated by the first record type in each group:

Type	Type ID	Contents
Run Header	10	Information pertinent to a group of samples processed in a continuous sequence; usually several per SDG
Sample Header	20	Sample identifying, qualifying, and linking information
Results Record	30	Analyte results and qualifications
Comments Record	90	Free form comments

2.1.2 All record types given are mandatory. Type 10, representing the analytical run, contains the instrument and run IDs which act as an identifying label for the run. All 10, 20, 30, and 90 series records following that record pertain to the same analytical run. Type 20, representing the sample, contains the EPA Sample ID which acts as an identifying label for the sample. The QC code indicates whether the data is from an environmental sample, calibration, or QC sample. All 20, 30, and 90 series records following that record pertain to the same sample. Type 30, representing an individual analyte, contains an identifier to identify the analyte. All 30 series records following that record pertain to the same analyte.

3.0 PRODUCTION RUNS

3.1 Specifications

A production run represents a “group” or “batch” of samples that are processed in a continuous sequence under relatively stable conditions. Specifically:

3.1.1 Calibration - All samples in a run use the same initial calibration data. For mercury analyses, samples prepared by a certain method must be analyzed with calibration and QC standards prepared by the same method. Therefore, all samples, calibration standards, and QC standards in a run must be associated with the same Preparation Code (Type 21 record).

3.1.2 Method number - Constant throughout a run.

3.1.3 Instrument conditions - Constant throughout a run. Results obtained on different instruments cannot be combined in one run.

3.1.4 Thus, each separate group of analyses on each instrument will consist of a separate production run, and must be reported in a separate file.

3.1.5 The run numbers in a Sample Delivery Group (SDG) must be unique; that is, there shall only be one Run Number “1”, only one Run Number “2”, etc. in an SDG.

3.1.6 In addition, later runs within a method for an analyte shall have a higher run number than earlier ones. For example, if arsenic is quantitated by the Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) method on 01/01/1999 beginning at 12:02 and arsenic is later quantitated by the ICP-AES method on 01/01/1999 beginning at 18:06, then the run beginning at 12:02 shall have a lower run number than the run beginning at 18:06.

3.2 Example

The following is an example of the sequence of record types in a production run.

10 Contains run header information. Occurs once per run.

16 Contains additional run header information. Occurs once per run.

20 Acts as a header for the following method and instrument parameters information. Occurs at least once per run with EPA sample number equal to “MDL”. Analysis year, analysis month, analysis day equal the year, month and day the Method Detection Limit (MDL) was computed. Analyte count equals the number of the Type 30 records that follow.

- 21 Contains the Preparation Code (field #5) and the Preparation Date (fields #8, 9, 10) for the MDL. Occurs at least once per run with each Type 21 record preceded by the relevant Type 20 record and immediately followed by its related Type 30 record(s).

- 30 Contains the Analyte Identifier “C” (field #2), the Analyte CAS Number (field #3), the MDL Label “U” (field #20), and the MDL (field #21). Occurs once for each analyte used in the run.

20

21

30

20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA sample number equal to "LRV". Analysis year, analysis month, analysis day equal the year, month and day the linear ranges were computed. Analyte count equals the number of Type 30, 32 and 34 groups that follow.

30 Contains only the Analyte CAS Number and the Analyte Identifier. Occurs once for each analyte used in the run.

32 Contains integration time information for the preceding analyte on the Type 30 record.

34 Contains the Contract Required Quantitation Limit (CRQL) and Linear Range information for the preceding analyte on the Type 30 record. There are as many consecutive Type 34 records as there are different wavelengths or masses used for the analyte identified on preceding Type 30.

30

32

34

20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA sample number equal to "BCD". Analysis year, analysis month, analysis day equal the year, month and day the background correction factors were computed. Analyte count equals the number of the Type 30 and 35 groups that follow.

30 Contains only the Analyte CAS Number and the Analyte Identifier. Occurs once for each analyte used in the run.

35 Contains the background and interelement correction information for the preceding analyte on the Type 30 record. There are as many consecutive Type 35 records as there are interelement correction factors for the analyte identified on preceding Type 30.

30

35

20 Contains header information for sample and QC data.

21 Contains additional information for analytical and instrument QC samples. Will always be preceded by a Type 20 record.

22 Contains additional information for analytical samples. Will usually follow a Type 21 record.

30 Contains the sample level concentration, true or added value and QC value for each analyte. Occurs once for

each analytical result for the EPA sample number of the previous Type 20 record.

31 Reports any instrumental data necessary to obtain the result reported on the previous Type 30 record. Will always be preceded by a Type 30 or 31 record. For Inductively Couple Plasma - Mass Spectrometry (ICP-MS), there are as many Type 31 records as there are isotopes for the analyte identified on the preceding Type 30 record.

30 Values for the next analyte being measured.

31 Values for the next analyte being measured.

30

31

Type 30-31 record sequence continues as many times as the value of the ANALYTE COUNT on the previous Type 20 record.

20 Next Sample Header record - The following applies to the next sample data.

21

22

30

31

30

31 etc.

20

21

22

30

31 etc.

4.0 RECORD SEQUENCE

4.1 Specifications

A Run Header (Type 10) record must be present as the first record in the file (run). Further occurrences of the Type 10 record in the file are not allowed.

4.1.1 A Type 16 record must immediately follow the Type 10 record. Further occurrences of the Type 16 record in the file are not allowed.

4.1.2 The first Type 20 records with EPA sample numbers MDL, LRV, and BCD are headers for the run-wide method and instrument parameters.

4.1.3 The first Type 20 record of the Type 21, 30 group is a header for the annually determined Method Detection Limits (MDLs) and must immediately follow the Type 16 record. A Type 20 record of the Type 21, 30 group must be present for each MDL reported in the run. For ICP-AES, ICP-MS, and cyanide analyses, an MDL associated with Preparation Code “NP1” must be present in each run. This MDL shall be used in the qualification of the data reported for non-prepared samples and instrument QC analyses (except the distilled Initial Calibration Verification (ICV) standard for cyanide).

4.1.4 The next Type 20 record of the Type 30, 32, 34 group is a header for the Linear Range Values (LRVs) and must immediately follow the last Type 30 record of the Type 21, 30 group that pertains to the MDL. The linear range values for all methods except the Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) methods are the analytically determined concentrations of the highest instrument calibration standards that are used in the generation of the calibration curve at the beginning of every run. The linear range values for the ICP-AES and ICP-MS methods are the quarterly determined values that are reported on Form XI-IN of the hardcopy.

4.1.5 The next Type 20 record of the Type 30, 35 group is a header for the ICP-AES Background Correction Data (BCD) and must immediately follow the last Type 34 record of the Type 30, 32, 34 group that pertains to the linear range values. This Type 20 record is not required for methods MS, AV, CV, CA, AS and C (i.e., ICP-MS, mercury, and cyanide analyses).

4.1.6 These are the only occurrences of the Type 20 records that do not relate to actual analyses in the run. Therefore, the only fields that are not blank in these occurrences of the Type 20 record are the RECORD TYPE (“20”); EPA SAMPLE NUMBER (“MDL”, “LRV” and “BCD”); Analysis Year/Year Computed, Analysis Month/Month Computed, Analysis Day/Day Computed (“YYYY”, “MM”, “DD”); and ANALYTE COUNT.

4.1.7 A minimum of one Type 30 record must immediately follow the Type 21 record of the Type 21, 30 group with EPA sample number MDL, and the total number of Type 30 records must be equivalent to the ANALYTE COUNT on the Type 20 record.

4.1.8 A minimum of one Type 30, 32, 34 group with EPA sample number LRV must immediately follow the Type 20 record which is preceded by the last Type 30 record of the final Type 21, 30 group. The information in each Type 30, 32, 34 group must pertain to one and only one analyte. The number of Type 30, 32, 34 groups must be equivalent to the ANALYTE COUNT on the Type 20 record.

4.1.9 A minimum of one Type 30, 35 group with EPA sample number BCD must immediately follow the Type 20 record for background correction data (if required). This Type 20 is preceded by the last Type 34 record of the final Type 30, 32, 34 group. The information in each Type 30, 35 group must pertain to one and only one analyte. The number of Type 30, 35 groups must be equivalent to the ANALYTE COUNT on the Type 20 record.

4.1.10 The Type 20 record that relates to the analysis of the first instrument calibration standard must immediately follow the last Type 30, 35 group for ICP-AES, or the last Type 30, 32, 34 group for mercury and cyanide analyses. For ICP-MS, the Type 20 record for the first instrument tune standard analysis must immediately follow the last Type 30, 32, 34 group and the Type 20 record for the first instrument calibration standard must immediately follow the last 30, 31 group from the last tune standard analyzed. After the appearance of these Type 20 records in the file, further occurrences of the Type 32, 34 and 35 records in that file are not allowed.

4.1.11 Each environmental sample, calibration, or Quality Control (QC) sample is represented by a group composed of Type 20, 21, and 22 records, which hold sample level identifying information, followed by a minimum of one group composed of Type 30 and 31 records for each analyte. The Type 20 record holds a count for the number of analytes being used to determine results. The ANALYTE COUNTER must have a value equivalent to the number of Type 30 groups associated with each Type 20 record.

4.1.12 Except for the first Type 20 records (EPA sample numbers MDL, LRV, BCD) for method ICP-AES and the first two Type 20 records (EPA sample numbers MDL, LRV) for the methods for ICP-MS, mercury and cyanide analyses, all Type 20 records should occur in the order of sample analysis.

4.1.13 Type 90 comment records may be defined to occupy any position except before the Type 10 (header) record. Comments pertaining to the whole run such as ones on Cover Page must appear before the first Type 20 record. Comments pertaining to a particular sample such as ones on Forms IA-IN and IB-IN must appear after the Type 20 record for that sample, but before the first Type 30 record associated with that sample. Comments pertaining to a particular analyte must appear after the Type 30 record of that analyte, but before the Type 30 record of the following analyte.

4.1.14 The Type 92 record which contains the sample associated data that is reported at the bottom of Forms IA-IN and IB-IN must appear anywhere after the Type 22 record for that EPA Field Sample, but before the Type 20 record of the next sample.

5.0 FILE/RECORD INTEGRITY

All record types must contain the following check fields to ensure file and record integrity:

Record Position	Field Length	Field Contents	Remarks
First Field	2	Record type or identifier	"10" or as appropriate
Last Field	5	Record sequence number	00000-99999, repeated as necessary. The record sequence numbers within a run must be in ascending order and shall not repeat until 99999 has been reached.
	4	Record checksum[1]	Four hexadecimal digits. The combination of record sequence number and record checksum must be unique within each run.
	2	Must contain CR and LF	

6.0 DATES AND TIMES

Date or time-of-day information consists of successive groups of digits, each separated by delimiters. Dates are given in the order YYYY MM DD, and times as HH MM. All hours must be given as 00 to 23 using a 24 hour clock and must be local time. All days shall be given as 01 to 31. All months shall be given as 01 to 12 (e.g., 01 is January, 02 is February).

7.0 MULTIPLE VOLUME DATA

There is no requirement under this format that all the data from an entire Sample Delivery Group (SDG) fit onto a single diskette. However, each single production run must fit onto a single diskette if possible. If that is not possible, then it is necessary that all files start with a Type 10 record, and that the multiple Type 10 records for each file of the same production run be identical. Information for a single sample may not be split between files.

[1]The checksum is the sum of the ASCII representation of the data on the record up to the Record Sequence Number (not including the Record Sequence Number), plus the checksum of the previous record. The sum is taken modulo 65536 (2^{16}) and is represented as four hexadecimal digits (i.e., the remainder of the sum divided by 65536 represented as four hexadecimal digits).

8.0 DELIVERABLE

8.1 Requirements

The file shall be submitted on IBM-compatible, 3.5 inch, high density 1.44 MB diskettes. The diskettes shall be formatted and recorded using DOS/Windows Operating Systems. The diskettes shall contain all information relevant to one and only one Sample Delivery Group (SDG). An alternative means of electronic transmission may be utilized if approved in advance by USEPA.

8.1.1 USEPA Agency Standard data from an entire SDG may not fit onto a single diskette. If a single production run is being split onto multiple diskettes, then all files shall start with a Type 10 record, and the multiple Type 10 records for each file of the same production run shall be identical. Do not split the data from a single sample onto multiple diskettes.

8.1.2 Information on the diskette **must correspond** to information submitted in the hardcopy raw data package and on the hardcopy raw data package forms. Unused records shall not be included on the diskettes. If the information submitted in the hardcopy data package forms is changed, the information in the electronic file (e.g., diskette) shall be changed accordingly, and a complete electronic deliverable containing all the information for the SDG shall be resubmitted along with the hardcopy at no additional cost to USEPA.

8.1.3 Each diskette shall be identified with an external label containing (in this order) the following information:

Disk Density
File Name(s)
Laboratory Name (optional)
Laboratory Code
Contract Number
Case Number/SDG
NRAS Number (where applicable)
Initial Submission or Resubmission (as applicable) and Date

8.1.4 The format for File Name shall be XXXXXX.I01 to XXXXXX.I99, where XXXXXX is the SDG identifier, I designates inorganics, and 01 through 99 is the file number.

8.1.5 Dimensions of the label must be in the range of 2-1/2" to 2-3/4" long by 2" to 2-1/8" wide for a 3-1/2" diskette.

9.0 RECORD LISTING

The following section provides information for the usage of each of the record types. Where specified, labels indicate the nature of the value(s) that follow on that record. If the value(s) will not be reported, the label shall be omitted. Listed below is every record type required to report data from a single Sample Delivery Group (SDG).

9.1 Production Run First Header Record (Type 10)

Use: Each production run will start with a Record Type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"10"
1	Delimiter	|
4	ANALYSIS START YEAR	YYYY
1	Delimiter	|
2	ANALYSIS START MONTH	MM
1	Delimiter	|
2	ANALYSIS START DAY	DD
1	Delimiter	|
2	ANALYSIS START HOUR	HH
1	Delimiter	|
2	ANALYSIS START MINUTE	MM
1	Delimiter	|
5	METHOD TYPE	CHARACTER[2]
1	Delimiter	|
8	METHOD NUMBER	"ILM05.3" (SOW)
1	Delimiter	|
3	MANAGER'S INITIALS	CHARACTER
1	Delimiter	|
6	LAB CODE	CHARACTER
4	Delimiter	||||
11	CONTRACT NUMBER	CHARACTER
1	Delimiter	|
10	INSTRUMENT ID	CHARACTER
2	Delimiter	||
25	LABORATORY NAME	CHARACTER
1	Delimiter	|
2	RUN NUMBER	NUMERIC[3]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[2]Analysis Method Types are:
"P" for ICP-AES
"MS" for ICP-MS
"CV" for Manual Cold Vapor AA
"AV" for Automated Cold Vapor AA
"AS" for Semi-Automated Spectrophotometric
"C" for Manual Spectrophotometric

[3]Run number values are 01 through 99. Each production run will be assigned a unique Run Number. Run Numbers are to be assigned sequentially beginning with 01 and will equal the number of production runs.

9.2 Production Run Second Header Record (Type 16)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"16"
1	Delimiter	|
4	ANALYSIS END YEAR	YYYY
1	Delimiter	|
2	ANALYSIS END MONTH	MM
1	Delimiter	|
2	ANALYSIS END DAY	DD
1	Delimiter	|
2	ANALYSIS END HOUR	HH
1	Delimiter	|
2	ANALYSIS END MINUTE	MM
1	Delimiter	|
1	AUTO-SAMPLER USED	"Y" or "N"[4]
1	Delimiter	|
1	INTERELEMENT CORRECTIONS APPLIED	"Y" or "N"[5]
1	Delimiter	|
1	BACKGROUND CORRECTIONS APPLIED	"Y" or "N"[5]
1	Delimiter	|
1	RAW DATA GENERATED	"Y" or "N" or "B"[6]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[4]Enter "Y" if an auto-sampler is used with equal time and intervals between analysis.

[5]These are the answers to the first two questions on the Cover Page of the hardcopy deliverable. "Y" equals "YES", and "N" equals "NO".

[6]This is the answer to the third question on the Cover Page of the hardcopy deliverable. "Y" equals "YES", "B" equals BLANK, and "N" equals "NO".

9.3 Mandatory Sample Header Data Record (Type 20)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD	"20"
1	Delimiter	|
2	REGION	NUMERIC
1	Delimiter	|
12	EPA SAMPLE NUMBER	CHARACTER[7]
1	Delimiter	|
5	MATRIX	CHARACTER[8]
1	Delimiter	|
3	QC CODE	CHARACTER
1	Delimiter	|
3	SAMPLE QUALIFIER	CHARACTER
1	Delimiter	|
5	CASE NUMBER	CHARACTER
1	Delimiter	|
6	SDG NUMBER	CHARACTER
1	Delimiter	|
4	ANALYSIS YEAR/YEAR COMPUTED	YYYY
1	Delimiter	|
2	ANALYSIS MONTH/MONTH COMPUTED	MM
1	Delimiter	|
2	ANALYSIS DAY/DAY COMPUTED	DD
1	Delimiter	|
2	ANALYSIS HOUR	HH
1	Delimiter	|
2	ANALYSIS MINUTE	MM
2	Delimiter	||
2	SAMPLE WT/VOL UNITS	"G"/"ML"[9]
1	Delimiter	|
5	SAMPLE WT/VOL	NUMERIC[10]
1	Delimiter	|
3	ANALYTE COUNT	NUMERIC
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[7]EPA Sample Number as it appears on Form XIII-IN of the hardcopy deliverable except for the first Type 20 records. The first Type 20 record must have an EPA sample number of "MDL"; after all Type 20 records with an EPA sample number of "MDL", the next Type 20 record must have an EPA sample number of "LRV"; for ICP-AES, the Type 20 record following the "LRV" must have an EPA sample number of "BCD".

[8]For matrix, "1" equals "WATER" and "F" equals "SOIL". A matrix identifier ("1" or "F") is required for all EPA sample numbers except "BCD".

[9]"G" equals grams and "ML" equals milliliters.

[10]This is the size of the sample at the beginning of the digestion procedure.

9.3.1 SAMPLE QC CODES LISTING FOR TYPE 20

NOTE: These QC codes appear in the QC code field on the Type 20 record (R20F5). They are used to indicate the type of data that is being reported.

QCC	Name	Definition
LRB	LABORATORY (REAGENT) BLANK	The Preparation Blank (see Exhibit G).
LCB	LABORATORY CALIBRATION BLANK	The Continuing Calibration Blank (CCB) (see Exhibit G).
LIB	LABORATORY INITIAL BLANK	The Initial Calibration Blank (ICB) (see Exhibit G).
LCM	LABORATORY CONTROL SOLUTION	The Laboratory Control Sample (LCS) (see Exhibit G).
LD2	LABORATORY DUPLICATE SECOND MEMBER	This is the second aliquot and is identified as "D" on Form VI-IN of the hardcopy.
LVM	LABORATORY CALIBRATION VERIFICATION SOLUTION	These values are identified as "Initial Calibration Verification" (ICV) on Form IIA-IN of hardcopy.
LVC	LABORATORY CONTINUING CALIBRATION VERIFICATION	These values are identified as "Continuing Calibration Verification" (CCV) on Form IIA-IN of hardcopy.
LVD	LABORATORY DISTILLED VERIFICATION SOLUTION	These values are the "distilled ICV" results for cyanide. Refer to Exhibit D, Section 12.7.1 for cyanide.
LSF	LABORATORY SPIKED SAMPLE - FINAL VALUES	These are the "Spiked Sample Result (SSR)" values of Form VA-IN of hardcopy.
LDO	LABORATORY DILUTED SAMPLE BACKGROUND (ORIGINAL) VALUES	These values are the "Initial Sample Result (I)" values on Form VIII-IN of hardcopy.
LDF	LABORATORY DILUTED SAMPLE - FINAL VALUES	These are the "Serial Dilution Result(S)" values Form VIII-IN of hardcopy.

PDO	POST-DIGESTION SPIKE BACKGROUND (ORIGINAL) VALUES	This value is identified as "Sample Result" (SR) on Form VB-IN of hardcopy.
PDF	POST-DIGESTION SPIKE BACKGROUND (FINAL) VALUES	This value is identified as "Spiked Sample Result" (SSR) on Form VB-IN of hardcopy.
LPC	CRQL CHECK STANDARD	Laboratory Performance Check Solution for analysis methods P, MS, CV, AV, AS, and C (EPA sample number is CRI). These results are reported on Form IIB-IN of hardcopy.
LSA	LABORATORY INTERFERENCE CHECK SOLUTION A	The results of this solution analysis (EPA sample number is ICSA) are reported on Forms IVA and IVB-IN of hardcopy.
LSB	LABORATORY INTERFERENCE CHECK SOLUTION AB	The results of this solution analysis (EPA sample number is ICSAB) are reported on Forms IVA and IVB-IN of hardcopy.
LTS	LABORATORY TUNE SAMPLE	The results of these solution analyses are reported on Form XIV-IN of hardcopy.
FRB	FIELD BLANK	This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.
FRM	PERFORMANCE EVALUATION (PE) SAMPLE	This is a sample of known composition provided by USEPA for Contractor analysis and is used to evaluate Contractor performance.
FLD	FIELD SAMPLE	This is the sample that is identified by a unique EPA sample number on the Traffic Report/Chain of Custody Record.
ZZQ	NON-SDG SAMPLE	This is any sample that is analyzed and is not part of the SDG (EPA sample number is ZZZZZZ).
STB	CALIBRATION STANDARD	This is the instrument calibration Blank Standard (EPA sample number is S0).

STC	CALIBRATION STANDARD	This is the instrument calibration CRQL Standard (EPA sample number is Sx where x is the CRQL value of the analyte).
STD	CALIBRATION STANDARD	This is the instrument calibration standard other than the Blank Standard or the CRQL Standard (EPA sample number is S).
STM	MIDRANGE STANDARD	This is the distilled cyanide Mid-range Standard (EPA sample number is MIDRANGE). Refer to Exhibit D, Section 10.2.1.1, for cyanide.
STR	RESLOPE SAMPLE	This is the resloping that is permitted for mercury analysis (EPA sample number is RESLOPE). Refer to Exhibit D, Section 9.1.5, for mercury.
STL	BASELINE SAMPLE	This is the baseline correction that is permitted for mercury analysis (EPA sample number is BASELINE). Refer to Exhibit D, Section 9.1.5, for mercury.
MDQ	METHOD DETECTION LIMIT	These are the annually determined analyte detection limits that are reported on Form IX-IN of hardcopy. (EPA sample number is MDL).
LRQ	LINEAR RANGE VALUE	These are the quarterly determined values for ICP-AES and ICP-MS methods that are reported on Form XI-IN of hardcopy. For all other methods, these are the analytically determined concentrations of the highest instrument calibration standards that are used in the generation of the calibration curve at the beginning of every run. (EPA sample number is LRV).
BCQ	BACKGROUND CORRECTION	These are the ICP-AES annually determined interelement correction factors that are reported on Forms XA and XB-IN of hardcopy. (EPA sample number is BCD).

NOTE: All field samples that are reported on the Traffic Report/Chain of Custody Record shall contain the QC code "FLD" in Record Type 20

Field Number 5 (R20F5) except when “FLD” is superseded by “FRB” (Field Blank Sample), “FRM” (PE Sample).

For Matrix Spike and Duplicate sample analysis (Forms VA-IN and VI-IN of hardcopy), the “Sample” result shall contain the QC code “FLD” in R20F5, the “Spiked Sample Result” shall contain the QC Code “LSF” in R20F5, and the “Duplicate” result shall contain the QC code “LD2" in R20F5.

9.4 Sample Header Record (Type 21)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"21"
2	Delimiter	||
3	LEVEL	"LOW"/"MED"
2	Delimiter	||
3	PREPARATION CODE	CHARACTER[11]
1	Delimiter	|
6	NRAS NUMBER	CHARACTER
1	Delimiter	|
14	LAB SAMPLE ID	CHARACTER
1	Delimiter	|
4	PREPARATION YEAR	YYYY
1	Delimiter	|
2	PREPARATION MONTH	MM
1	Delimiter	|
2	PREPARATION DAY	DD
2	Delimiter	||
4	YEAR RECEIVED	YYYY
1	Delimiter	|
2	MONTH RECEIVED	MM

[11]Preparation Codes: A Preparation Code is required for all EPA sample numbers except "LRV", "BCD", and "TUNE".
"HW1" - Hotplate/Block digestion for ICP-AES analysis of water samples.
"HW2" - Hotplate/Block digestion for ICP-MS analysis of water samples.
"HW3" - Hotplate/Block digestion for ICP-MS analysis of water samples.
"MW1" - Microwave digestion for ICP-AES analysis of water samples.
"MW2" - Microwave digestion for ICP-AES analysis of water samples.
"HS1" - Hotplate/Block digestion for ICP-AES analysis of soil samples.
"HS2" - Hotplate/Block digestion for ICP-AES analysis of soil samples.
"MS1" - Microwave digestion for ICP-AES analysis of soil samples.
"CW1" - Preparation for the Manual Cold Vapor AA analysis of water samples.
"CS1" - Preparation for the Manual Cold Vapor AA analysis of soil samples.
"CW2" - Preparation for the Automated Cold Vapor analysis of water samples.
"DW1" - Distillation for the manual and semi-automated spectrophotometric analysis of water samples.
"DW2" - Midi-distillation for the semi-automated spectrophotometric analysis of water samples.
"DS1" - Distillation for the manual and semi-automated spectrophotometric analysis of soil samples.
"DS2" - Midi-distillation for the semi-automated spectrophotometric analysis of soil samples.
"NP1" - No preparation.

Sample Header Record (Type 21) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
2	DAY RECEIVED	DD
1	Delimiter	|
9	SOLUTION SOURCE	CHARACTER[12]
1	Delimiter	|
8	INJECTION/ALIQUOT VOLUME	NUMERIC[13]
1	Delimiter	|
2	PREPARATION START HOUR	HH[14]
1	Delimiter	|
2	PREPARATION START MINUTE	MM[14]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[12]This is the source of the solutions that are reported on Inorganic Forms IIA-IN, IIB-IN, IV-IN, and VII-IN of the hardcopy (ICV, CCV, CRI, ICS, and LCS), and the source of the instrument calibration standards.

[13]This is the portion of the sample that is injected into the instrument excitation system for the purpose of measuring the absorbence, emission, or concentration of an analyte.

[14]This is the time at which the preparation is started. It is used to differentiate between different batches on the same day.

9.5 Associated Injection and Counter Record (Type 22)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"22"
8	Delimiter	||||||||
5	VOLUME ADJUSTMENT FACTOR	NUMERIC[15]
2	Delimiter	||
8	FINAL VOLUME	NUMERIC[16]
1	Delimiter	|
8	DILUTION FACTOR	NUMERIC
3	Delimiter	|||
5	PERCENT SOLIDS	NUMERIC
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[15]This field is used to report any additional volume adjustments in the preparation method. As an example, the factor of 1.25 that results from the chloride interference volume adjustment in Preparation Method/Code HW2.

[16]This is the final volume that is currently reported on Form XII-IN of the hardcopy.

9.6 Results Data Record (Type 30)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"30"
1	Delimiter	|
1	ANALYTE IDENTIFIER	"C"[17]
1	Delimiter	|
9	ANALYTE CAS NUMBER	CHARACTER[18]
2	Delimiter	||
5	CONCENTRATION UNITS	"UG/L"/"MG/KG"
1	Delimiter	|
3	CONCENTRATION QUALIFIER	CHARACTER[19]
1	Delimiter	|
15	CONCENTRATION	NUMERIC[20,21]
1	Delimiter	|
1	VALUE DESCRIPTOR	"T"/"F"[22]
1	Delimiter	|
10	AMOUNT ADDED OR TRUE VALUE	NUMERIC
1	Delimiter	|
1	QC VALUE DESCRIPTOR, P	"P"[23]
1	Delimiter	|
10	QC VALUE	NUMERIC
1	Delimiter	|||
1	QC VALUE DESCRIPTOR, L	"L"[23]
1	Delimiter	|
10	QC VALUE	NUMERIC
1	Delimiter	|
1	MATRIX SPIKE QC LIMIT QUALIFIER	"N"[24]
1	Delimiter	|
10	QC LOWER LIMIT	NUMERIC[25]
1	Delimiter	|
10	QC UPPER LIMIT	NUMERIC[25]
1	Delimiter	|
1	QC LIMIT QUALIFIER	"*"/"E"[26]
1	Delimiter	|
1	MDL LABEL	"U"
1	Delimiter	|
10	MDL	NUMERIC[27]
2	Delimiter	||
15	RAW DATA AVERAGE	NUMERIC[28]
1	Delimiter	||
10	RAW DATA %RSD	NUMERIC
1	Delimiter	||
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

FORMAT OF THE RESULTS DATA RECORD (TYPE 30) FOOTNOTES

[17]"C" (CAS Registry Number) is used for all metals and cyanide.

[18]The CAS Numbers for metals and cyanide are in Exhibit B, Form IA-IN, and Table 1 - Inorganic Target Analyte List and Contract Required Quantitation Limits (CRQLs), in Exhibit C. NOTE: The CAS Numbers for the ICS non-target interferents are as follows: carbon (7440-44-0); chlorine (7782-50-5); molybdenum (7439-98-7); phosphorus (7723-14-0); sulfur (7704-34-9), and titanium (7440-32-6).

[19]"BDL" means below detection limit.

"NSQ" means there is not sufficient quantity to prepare sample according specification in Exhibit D; therefore, a smaller sample size is used.

"NAR" means no analysis result required.

"LTC" means less than the CRQL but greater than or equal to the MDL.

"FQC" means failed Quality Control (QC) criteria.

"GTL" means greater than the linear range. The result is reported from a re-analysis at an appropriate dilution.

"RIN" means that the analysis result was not used to report data in the SDG. The result is reported from a later re-analysis of the same sample aliquot.

"REX" means that the analysis result was not used to report data in the SDG. The result is reported from a later re-analysis of a repreparation of the same sample.

Note that, except for "NAR", none of these codes relieves the Contractor from reporting a valid result. They only explain why or if the result is qualified. Also note that ICP-MS internal standard results are not to be qualified.

[20]EPA Field Samples reported on Traffic Report/Chain of Custody Record (QC codes FLD, FRB, FRM) shall have their analytes' results reported to four decimal places.

[21]Follow the instructions for the reporting of data in Exhibit B in reporting results for samples with QC codes. For example, the LD2 QC code sample results shall be reported to four decimal places because the duplicate results on Form VI-IN have to be reported to four decimal places. Refer to Section 9.3.1 for QC codes and definitions.

[22]"T" stands for an analyte's true value in a solution. This includes the concentration of all Instrument Calibration Standards for ALL methods of analysis. "F" stands for an added concentration to a sample such as a pre- or post-digestion spike.

[23]"P" equals Percent Recovery (%R), Percent Difference (%D), Relative Percent Difference (RPD), Percent Relative Standard Deviation (%RSD), Percent Relative Intensity (%RI), or correlation coefficient. "L" equals control limit for duplicates. The matrix spike sample %R shall be entered on the Type 30 record of the EPA sample number with the "S" suffix (QC code=LSF). The post digest spike sample %R shall be entered on the Type 30 record of the EPA sample number with the "A" suffix (QC code=PDF). The RPD and the control limit for duplicates shall be entered on the Type 30 record of the EPA sample number with the "D" suffix (QC code=LD2). The ICP serial dilutions %D shall be entered on the Type 30 record of the EPA sample number with the "L" suffix (QC code=LDF). The average %RSD for ICP-MS tune analyses shall be entered on the Type 30 record of the last EPA sample number "TUNE" (QC code=LTS) in each run. The %RI for ICP-MS internal standards shall be entered on the Type 30 record of all EPA samples numbers (except "TUNE", "ZZZZZZ", "MDL", and "LRV").

The correlation coefficient for the calibration for mercury and cyanide analyses shall be reported on the Type 30 record of the EPA sample number associated with the final standard analyzed in the calibration curve (immediately preceding the ICV).

[24]"N" is the qualifier that is used on Form VA-IN of the hardcopy to indicate that the matrix or pre-digestion spike sample recovery for an analyte is not within the specified control limits. The "N" qualifier shall be entered on the Type 30 record of the EPA sample number with the "S" suffix (QC code=LSF).

[25]These are the control limits for the ICV/CCV percent recovery (%R) on Form IIA-IN, the CRI %R on Form IIB-IN, the ICSA/ICSAB %R on Forms IVA and IVB-IN, the matrix spike %R on Form VA-IN, and the LCSW %R and the LCSS upper and lower limits on Form VII-IN. The QC upper and lower limits for the Spike Sample Recovery shall be entered on the Type 30 record of the EPA sample number with the "S" suffix (QC code=LSF).

[26]"*" is the qualifier that is used on Form VI-IN of the hardcopy to indicate that the duplicate sample analysis for an analyte is out of control, and "E" is the qualifier that is used on Form VIII-IN of the hardcopy to indicate that the ICP serial dilution analysis results are estimated because of the existence of significant physical or chemical interferences. The "*" qualifier should be entered on the Type 30 record of the EPA sample number with the "D" suffix (QC code=LD2) The "E" qualifier shall be entered on the Type 30 record of the EPA sample number with the "L" suffix (QC code=LDF).

[27]The MDL shall be reported to 2 significant figures for values less than 10 and to 3 significant figures for values greater than or equal to 10. MDLs shall be reported in UG/L for water samples, ICV, ICB, CCV, CCB, CRI, ICSA, ICSAB and MIDRANGE (for cyanide), and any other samples with concentration results reported in "UG/L". MDLs shall be reported in MG/KG for soil samples.

[28]The average value of the replicate injections or exposures are reported in this field. The average values for mercury and cyanide analyses are also reported in this field. In addition, the raw data average value shall always be reported in units of UG/L to a minimum of four decimal places, regardless of the units the instrument readings are reported in, on record Type 31. The raw data average value shall not be corrected for dilutions or volume adjustments.

For Instrument Calibration Standards analyses and Instrument Tune Standards analyses, the raw data average is not required to be reported.

9.7 Instrumental Data Readout (Type 31)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"31"
1	Delimiter	|
1	TYPE OF DATA	"W"/"M"[29]
1	Delimiter	|
1	TYPE OF VALUE	CHARACTER[30]
2	Delimiter	||
8	ANALYTE WAVELENGTH/MASS	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	|
15	FIRST INSTRUMENT VALUE	NUMERIC[31]
2	Delimiter	||
15	SECOND INSTRUMENT VALUE	NUMERIC[31]
2	Delimiter	||
15	THIRD INSTRUMENT VALUE	NUMERIC[31]
2	Delimiter	||
15	FOURTH INSTRUMENT VALUE	NUMERIC[31]
2	Delimiter	||
15	FIFTH INSTRUMENT VALUE	NUMERIC[31]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[29]"W" equals wavelength, "M" equals mass.

[30]"C" equals concentration in µg/L, "B" equals absorbance, "I" equals intensity (counts per second or equivalent).

[31]Used to report data for method analyses that require replicate injections or exposures. If a single instrument measurement is used, then enter it in the first instrument value field, and leave the other four fields empty. If two instrument measurements are used, then enter them in the first and second instrument value fields in the order of their analyses, and leave the other three fields empty, etc. In addition, the instrument values shall be reported to a minimum of four decimal places.

9.8 Auxiliary Data Record (Type 32)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"32"
10	Delimiter	||||||||||
2	INTEGRATION TIME CODE	"IT"
1	Delimiter	|
10	INTEGRATION TIME	IN SECONDS
4	Delimiter	||||
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

9.9 QC Limit Record (Type 34)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"34"[32]
4	Delimiter	||||
8	ANALYTE WAVELENGTH OR MASS	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	|
10	CRQL	NUMERIC
1	Delimiter	|
10	LINEAR RANGE VALUE	NUMERIC
6	Delimiter	||||||
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

[32]There must be as many consecutive Type 34 records as there are wavelengths or masses used for the analyte identified in the preceding Type 30 record.

9.10 Correction Data Record (Type 35)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"35"
1	Delimiter	|
3	TYPE OF CORRECTION	"ICP"
1	Delimiter	|||||
9	CAS NUMBER OF INTERFERING ANALYTE	CHARACTER
1	Delimiter	|
8	ANALYTE WAVELENGTH	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	|
10	CORRECTION FACTOR	NUMERIC
1	Delimiter	|
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

9.11 Comment Record (Type 90)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"90"
1	Delimiter	|
67	ANY COMMENT	CHARACTER
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

9.12 Sample Associated Data Record (Type 92)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"92"
1	Delimiter	|
9	COLOR BEFORE	CHARACTER
1	Delimiter	|
9	COLOR AFTER	CHARACTER
1	Delimiter	|
6	CLARITY BEFORE	CHARACTER
1	Delimiter	|
6	CLARITY AFTER	CHARACTER
1	Delimiter	|
6	TEXTURE	CHARACTER
1	Delimiter	|
3	ARTIFACTS	"YES"/BLANK
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

APPENDIX A -- FORMAT OF RECORDS FOR SPECIFIC USES

DISCLAIMER

The USEPA does not warrant or guarantee the completeness and/or accuracy of the representative examples of record type uses provided in this appendix. This appendix serves as an example for the usage of record types and in no way redefines or supersedes the specifications or requirements stated in Exhibits A through H of ILM05.3.

THIS PAGE INTENTIONALLY LEFT BLANK

Appendix A -- Format of Records for Specific Uses

Table of Contents

Section		Page
1.0	ICP	5
1.1	ICP-AES	5
1.2	ICP-MS	7
2.0	MERCURY	10
2.1	Start of a Mercury Run for Water Samples with Record Types 10 and 16 and the First Type 20 Records	10
2.2	Mercury Instrument Calibration Standards: Blank (S0) and Four Other Standards	10
2.3	Spike Sample Recovery and Duplicates Performed on Different Samples (QC Codes FLD, LSF, FLD, LD2)	11
2.4	Duplicates and Spike Sample Recovery Performed on the Same Sample (QC Codes FLD, LD2, LSF)	11
2.5	Initial Calibration Verification (ICV) with LVM QC Code	12
2.6	Laboratory Control Sample (Solid) with LCM QC Code	12
3.0	CYANIDE	12
3.1	Start of a Cyanide Run with Record Types 10 and 16 and the First Type 20 Records	12
3.2	Cyanide Instrument Calibration Standards: Blank (S0) and Five Other Standards	12
3.3	Preparation Blank (Soil) with LRB QC Code	13
3.4	Laboratory Control Sample (Soil) with LCM QC Code	13
3.5	Continuing Calibration Verification (CCV) with LVC QC Code	13
3.6	Spike Sample Recovery and Post Distillation Spike Sample Recovery Performed on the Same Sample (QC Codes FLD, PDO, LSF, PDF)	13

THIS PAGE INTENTIONALLY LEFT BLANK

1.0 ICP

1.1 ICP-AES

1.1.1 Start of an ICP-AES Run with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|17|09|06|P|ILM05.3|ABC|TESLAB||||68-D2-0039|P2||TEST LABSINC.|2|000001879
16|1999|09|17|12|03|Y|Y|Y|N|000012114

20|1|MDL|1|MDQ||||1999|07|15||||||04|000044B9D
21||||NP1|||||||||||||000053CD5
30|C|7440-22-4||UG/L|||||||||||||||U|3.1||||||000065996
30|C|7429-90-5||UG/L|||||||||||||||U|21.8||||||0000767D1
30|C|7440-39-3||UG/L|||||||||||||||U|11.5||||||0000875CB
30|C|7440-41-7||UG/L|||||||||||||||U|1.1||||||0000983C5

20|1|MDL|1|MDQ||||1999|07|15||||||04|0000104B9D
21||||HW1|||1999|07|15|||||||08|00|0000113CD5
30|C|7440-22-4||UG/L|||||||||||||||U|3.4||||||0000125996
30|C|7429-90-5||UG/L|||||||||||||||U|22.8||||||00001367D1
30|C|7440-39-3||UG/L|||||||||||||||U|12.5||||||00001475CB
30|C|7440-41-7||UG/L|||||||||||||||U|2.1||||||00001583C5

20|1|MDL|F|MDQ||||1999|07|16||||||04|000164B9D
21||||HS1|||1999|07|15|||||||08|00|00017A212
30|C|7440-22-4||MG/KG|||||||||||||||U|0.82||||||00018C248
30|C|7429-90-5||MG/KG|||||||||||||||U|4.8||||||00019B321
30|C|7440-39-3||MG/KG|||||||||||||||U|3.1||||||00020CE75
30|C|7440-41-7||MG/KG|||||||||||||||U|0.42||||||00021A21B

20|1|LRV|1|LRQ||||1999|07|15||||||04|0002356C2
30|C|7440-22-4|||||||||||||||||||||||0002463D1
32||||||||||IT|5.00||||000256CDA
34||||328.00|5|40000||||||000267591
30|C|7429-90-5|||||||||||||||||||||||0002782AD
32||||||||||IT|5.00||||000288BB6
34||||308.20|200|1000000||||||0002994FB
30|C|7440-39-3|||||||||||||||||||||||00030A211
32||||||||||IT|5.00||||00031AB1A
34||||493.40|20|100000||||||00032B436
30|C|7440-41-7|||||||||||||||||||||||00033C149
32||||||||||IT|5.00||||00034CA52
34||||313.00|2|25000||||||00035D2DA

20|1|BCD||BCQ||||1999|07|01||||||04|0007894FB
30|C|7440-22-4|||||||||||||||||||||||00079A20A
35|ICP|||||7439-89-6|259.90|-0.0002500|00080AC9B
35|ICP|||||7439-96-5|257.60|0.0002200|00081B6F4
30|C|7429-90-5|||||||||||||||||||||||00082C410
35|ICP|||||7439-96-5|257.60|0.0004900|00083CE72
35|ICP|||||7440-62-2|292.40|-0.0419200|00084D8EF
30|C|7440-39-3|||||||||||||||||||||||00085E605
35|ICP|||||7439-96-5|257.60|0.0000600|00086F060
30|C|7440-41-7|||||||||||||||||||||||00087FD73
35|ICP|||||7440-50-8|324.70|0.0046200|0008914D1
35|ICP|||||7439-96-5|257.60|0.0015400|000901F30
```

1.1.2 ICP-AES Instrument Calibration Standards, S0 and S

```
20|1|S0|1|STB||20596|MAX123|1999|09|17|09|06||||04|00128D199
21||||NP1||STDB|1999|09|17|||||TESLAB||||00129DD31
22||||||||||||1.0||||00130E598
```

```
30|C|7440-22-4|||||T|0.0|||||||||||U|3.1||||||00131F8F5
31|W|I||328.00|0.0304||0.0374||0.0400|||||001320305
30|C|7429-90-5|||||T|0.0|||||||||||U|21.8||||||001331697
31|W|I||308.20|0.0104||0.0136||0.0120|||||001342137
30|C|7440-39-3|||||T|0.0|||||||||||U|11.5||||||00135348D
31|W|I||493.40|-0.0002||0.0002||0.0000|||||001363EA4
30|C|7440-41-7|||||T|0.0|||||||||||U|1.1||||||0013751FA
31|W|I||313.00|0.0006||0.0002||0.0004|||||001385C04

20|1|S|1|STD||20596|MAX123|1999|09|17|09|11||||04|00206314E
21||||NP1||STD1|1999|09|17|||||TESLAB||||002073CD5
22||||||||||||1.0||||00208453C
30|C|7440-22-4|||||T|5000|||||||||||U|3.1||||||002139157
31|W|I||328.00|1.9540||1.9610||1.9660|||||002149B6E
30|C|7429-90-5|||||T|1000|||||||||||U|21.8||||||00215ADE2
31|W|I||308.20|0.8384||0.8378||0.8440|||||00216B7EC
30|C|7440-39-3|||||T|5000|||||||||||U|11.5||||||00219E77D
31|W|I||493.40|1.9460||1.9510||1.9684|||||00220F18F
30|C|7440-41-7|||||T|5000|||||||||||U|1.1||||||002210410
31|W|I||313.00|0.9924||0.9910||1.0010|||||002220E25
```

1.1.3 Duplicates, Spike Sample Recovery, and Serial Dilutions Performed on the Same Field Sample (QC Codes FLD, LDO, LD2, LSF, LDF)

```
20|1|MAX123|F|FLD||20596|MAX123|1999|09|17|11|09||G|1.05|08|01568C5FD
21||LOW||HS1||S308233-01|1999|09|14||1999|08|24|||08|30|01569D451
22||||||||||200|1.0|||91.5|01570DE17
90|STONES|01571E154
92|GREY|GREY|||MEDIUM|YES|01572EA43
30|C|7440-22-4||MG/KG|BDL|2.0817||||||||||||U|0.82||1.1567||||01573FD12
31|W|C||328.00|4.2000||0.5500||-1.2800|||||0157409A5
30|C|7429-90-5||MG/KG||6227.0101||||||||||||U|4.8||29913.0000||||015751DCD
31|W|C||308.20|29992.0000||29654.0000||30093.0000|||||015762CA0
30|C|7440-39-3||MG/KG|LTC|21.9349||||||||||||U|3.1||105.3700||||01577400C
31|W|C||493.40|107.2400||101.6400||107.2300|||||015784DA6
30|C|7440-41-7||MG/KG|BDL|1.0409||||||||||||U|0.42||1.4900||||01579606A
31|W|C||313.00|1.4900||1.4900||1.4900|||||015806CD9

20|1|MAX123|F|LDO||20596|MAX123|1999|09|17|11|09||G|1.05|08|01650C630
21||LOW||HS1||S308233-01|1999|09|14||1999|08|24|||08|30|01651D484
22||||||||||200|1.0|||91.5|01652DE4A
30|C|7440-22-4||UG/L|BDL|10.00||||||||||||U|3.9||1.1567||||01655FC98
31|W|C||328.00|4.2000||0.5500||-1.2800|||||01656092B
30|C|7429-90-5||UG/L||29913.00||||||||||||U|23.1||29913.0000||||016571C09
31|W|C||308.20|29992.0000||29654.0000||30093.0000|||||016582ADC
30|C|7440-39-3||UG/L|LTC|105.37||||||||||||U|14.9||105.3700||||016593DCE
31|W|C||493.40|107.2400||101.6400||107.2300|||||016604B68
30|C|7440-41-7||UG/L|BDL|5.00||||||||||||U|2.0||1.4900||||01579606A
31|W|C||313.00|1.4900||1.4900||1.4900|||||015806CD9

20|1|MAX123D|F|LD2||20596|MAX123|1999|09|17|11|11||G|1.04|08|016913BCF
21||LOW||HS1||S308233-02|1999|09|14||1999|08|24|||08|30|016924A23
22||||||||||200|1.0|||90.9|0169353EC
30|C|7440-22-4||MG/KG|BDL|2.1017||||||||||||U|0.82||0.9600||||0169466BE
31|W|C||328.00|1.6400||1.6300||-0.3900|||||016957356
30|C|7429-90-5||MG/KG||6622.7406|||P|6.2|||||||||U|4.8||31511.0000||||016968784
31|W|C||308.20|31993.0000||31313.0000||31227.0000|||||016979641
30|C|7440-39-3||MG/KG|LTC|25.1387|||P|13.6|||||||||U|3.1||119.6100||||01698AAC5
31|W|C||493.40|121.4600||118.9300||118.4400|||||01699B86C
30|C|7440-41-7||MG/KG|BDL|1.0509||||||||||||U|0.42||1.5000||||01700CC13
31|W|C||313.00|1.5000||1.5000||1.5000|||||01701D86A
```

```
20|1|MAX123S|F|LSF||20596|MAX123|1999|09|17|11|14||G|1.01|08|01730BE3C
21||LOW||HS1||S308233-03|1999|09|14||1999|08|24|||08|30|01731CC90
22||||||||||200|1.0|||91.5|01732D656
30|C|7440-22-4||MG/KG||10.7212|F|10.82|P|99||||||75|125||U|0.82||49.5400||||01733EBC7
31|W|C||328.00|48.8400||49.2000||50.5800|||||01734F8DC
30|C|7429-90-5||MG/KG|NAR|6859.9253|||||||||||||U|4.8||31698.0000||||017350E27
31|W|C||308.20|31578.0000||31766.0000||31750.0000|||||017361CF1
30|C|7440-39-3||MG/KG||326.3539|F|432.83|P|70|||||N|75|125||U|3.1||1508.0000||||017373339
31|W|C||493.40|1524.0000||1504.4000||1495.6000|||||017384171
30|C|7440-41-7||MG/KG||10.4290|F|10.82|P|96||||||75|125||U|0.42||48.1900||||0173956E4
31|W|C||313.00|48.1900||48.2000||48.1800|||||0174063EB

20|1|MAX123L|F|LDF||20596|MAX123|1999|09|17|11|17||||08|017696573
21||LOW||||S308233-04|||||1999|08|24|||||017707255
22||||||||||||5.0|||91.5|017717B8D
30|C|7440-22-4||UG/L|BDL|50.00|||||||||||||U|3.9||0.6100||||017728DDF
31|W|C||328.00|1.4500||-0.3800||0.7600|||||017739A7B
30|C|7429-90-5||UG/L||25575.50|||P|15|||||||E|U|23.1||5115.1000||||01774AE69
31|W|C||308.20|5038.6000||5126.4000||5180.3000|||||01775BCAC
30|C|7440-39-3||UG/L|LTC|111.30|||P|6|||||||||U|14.9||22.2600||||01776D0AA
31|W|C||493.40|22.2600||22.7700||21.7500|||||01777DDB9
30|C|7440-41-7||UG/L|BDL|25.00|||||||||||||U|2.0||0.3000||||0173956E4
31|W|C||313.00|0.1900||0.2000||0.51|||||0174063EB
```

1.2 ICP-MS

1.2.1 Start of an ICP-MS Run with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|17|09|06|MS|ILM05.3|ABC|TESLAB||||68-D2-0039|P2||TEST LABSINC.|2|000001879
16|1999|09|17|12|03|Y|Y|Y|N|000012114

20|1|MDL|1|MDQ||||1999|07|15||||||04|000044B9D
21||||NP1|||||||||||||||00005DD31
30|C|7440-22-4||UG/L|||||||||||||||U|0.40||||||000065996
30|C|7429-90-5||UG/L|||||||||||||||U|12.8||||||0000767D1
30|C|7440-39-3||UG/L|||||||||||||||U|3.0||||||0000875CB
30|C|7440-41-7||UG/L|||||||||||||||U|0.44||||||0000983C5

20|1|MDL|1|MDQ||||1999|07|15||||||04|000044B9D
21||||HW2|||1999|07|15|||||||09|00|00005DD31
30|C|7440-22-4||UG/L|||||||||||||||U|0.41||||||000065996
30|C|7429-90-5||UG/L|||||||||||||||U|13.8||||||0000767D1
30|C|7440-39-3||UG/L|||||||||||||||U|4.0||||||0000875CB
30|C|7440-41-7||UG/L|||||||||||||||U|0.43||||||0000983C5

20|1|LRV|1|LRQ||||1999|07|15||||||04|0002356C2
30|C|7440-22-4|||||||||||||||||||||||0002463D1
32||||||||||IT|5.00||||000256CDA
34||||107.00|5|40000||||||000267591
30|C|7429-90-5|||||||||||||||||||||||0002782AD
32||||||||||IT|5.00||||000288BB6
34||||27.00|200|1000000||||||0002994FB
30|C|7440-39-3|||||||||||||||||||||||00030A211
32||||||||||IT|5.00||||00031AB1A
34||||137.00|20|100000||||||00032B436
30|C|7440-41-7|||||||||||||||||||||||00033C149
32||||||||||IT|5.00||||00034CA52
34||||111.00|2|25000||||||00035D2DA
```

1.2.2 ICP-MS Instrument Tune and Calibration Standards, S0 and S

```
20|3|TUNEA1|1|LTS||26791|MCSB00|1999|02|06|20|00||||5|000917DD7
```

```
21||||||TUNE1|||||||TESLAB||||000917DD8
22||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA2|1|LTS||26791|MCSB00|1999|02|06|20|10||||5|000917DD7
21||||||TUNE2|||||||TESLAB||||000917DD8
22||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||100000||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||100000||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA3|1|LTS||26791|MCSB00|1999|02|06|20|20||||5|000917DD7
21||||||TUNE3|||||||TESLAB||||000917DD8
22||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA4|1|LTS||26791|MCSB00|1999|02|06|20|30||||5|000917DD7
21||||||TUNE4|||||||TESLAB||||000917DD8
22||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||000914DE2
```

```
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA5|1|LTS||26791|MCSB00|1999|02|06|20|40||||5|000917DD7
21||||||TUNE5|||||||TESLAB||||000917DD8
22||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|P|0.0|||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|P|0.0|||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|P|0.0|||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|P|0.0|||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|P|0.0|||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|1|S0|1|STB||20596|MAX123|1999|09|17|09|06||||04|00128D199
21||||NP1||STDB|1999|09|17|||||TESLAB||||00129DD31
22||||||||||1.0||||00130E598
30|C|7440-22-4|||||T|0.0||||||||||U|0.40||||||00131F8F5
31|M|I||107.00|0.0304||0.0374||0.0400|||||001320305
30|C|7429-90-5|||||T|0.0||||||||||U|12.8||||||001331697
31|M|I||27.00|0.0104||0.0136||0.0120|||||001342137
30|C|7440-39-3|||||T|0.0||||||||||U|3.0||||||00135348D
31|M|I||137.00|-0.0002||0.0002||0.0000|||||001363EA4
30|C|7440-41-7|||||T|0.0||||||||||U|0.44||||||0013751FA
31|M|I||111.00|0.0006||0.0002||0.0004|||||001385C04

20|1|S|1|STD||20596|MAX123|1999|09|17|09|11||||04|00206314E
21||||NP1||STD1|1999|09|17|||||TESLAB||||002073CD5
22||||||||||1.0||||00208453C
30|C|7440-22-4|||||T|5000||||||||||U|0.40||||||002139157
31|M|I||107.00|1.9540||1.9610||1.9660|||||002149B6E
30|C|7429-90-5|||||T|1000||||||||||U|12.8||||||00215ADE2
31|M|I||27.00|0.8384||0.8378||0.8440|||||00216B7EC
30|C|7440-39-3|||||T|5000||||||||||U|3.0||||||00219E77D
31|M|I||136.00|1.9460||1.9510||1.9684|||||00220F18F
30|C|7440-41-7|||||T|5000||||||||||U|0.44||||||002210410
31|M|I||111.00|0.9924||0.9910||1.0010|||||002220E25
```

1.2.3 Field Samples

```
20|1|MAX123|1|FLD||20596|MAX123|1999|09|17|09|06||ML|100|04|00128D199
21||||HW2||S308233-01|1999|09|17||1999|09|16|TESLAB||09|30|00129DD31
22||||||||1.25||50|1.0|||0.0|00130E598
30|C|7440-22-4||UG/L|LTC|0.6625||||||||||||U|0.41||0.5300||||00131F8F5
```

```
31|M|I||107.00|0.5300||0.5300||0.5300|||||001320305
30|C|7429-90-5||UG/L||56.3750|||||||||||U|13.8||45.1000||||001331697
31|M|I||27.00|45.1000||45.1000||45.1000|||||001342137
30|C|7440-39-3||UG/L||11.0000|||||||||||U|4.0||8.8000||||00135348D
31|M|I||137.00|8.8000||8.8000||8.8000|||||001363EA4
30|C|7440-41-7||UG/L|BDL|1.000|||||||||||U|0.43||0.3210||||0013751FA
31|M|I||111.00|0.3210||0.3210||0.3210|||||001385C04

20|1|MAX124|1|FLD||20596|MAX123|1999|09|17|09|06||ML|20|04|00128D199
21||||NP1||S308234-01|1999|09|17||1999|09|16|TESLAB||09|30|00129DD31
22|||||||||20|1.0|||0.0|00130E598
30|C|7440-22-4||UG/L|LTC|0.5300|||||||||||U|0.40||0.5300||||00131F8F5
31|M|I||107.00|0.5300||0.5300||0.5300|||||001320305
30|C|7429-90-5||UG/L||45.1000|||||||||||U|12.8||45.1000||||001331697
31|M|I||27.00|45.1000||45.1000||45.1000|||||001342137
30|C|7440-39-3||UG/L|LTC|8.8000|||||||||||U|3.0||8.8000||||00135348D
31|M|I||137.00|8.8000||8.8000||8.8000|||||001363EA4
30|C|7440-41-7||UG/L|BDL|1.000|||||||||||U|0.44||0.3210||||0013751FA
31|M|I||111.00|0.3210||0.3210||0.3210|||||001385C04
```

2.0 MERCURY

2.1 Start of a Mercury Run for Water Samples with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|09|08|44|CV|ILM05.3|ABC|TESLAB||||68-D2-0039|M3||TEST LABS INC.|6|0000018F7
16|1999|09|09|14|34|N||||000012099

20|1|MDL|1|MDQ||||1999|07|15||||||1|000044AEB
21||||CW1|||1999|07|15|||||||||000053CD5
30|C|7439-97-6||UG/L|||||||||||||||U|0.042||||||0000658F4

20|1|LRV|1|LRQ||||1999|09|09||||||1|0000666A6
30|C|7439-97-6|||||||||||||||||||||||0000773CB
32|||||||||||||000087D02
34||||253.70|0.2|5||||||00009852D
```

2.1.1 Start of a Mercury Run for Soil Samples with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|09|08|44|CV|ILM05.3|ABC|TESLAB||||68-D2-0039|M3||TEST LABS INC.|6|0000018F7
16|1999|09|09|14|34|N||||000012099

20|1|MDL|F|MDQ||||1999|07|16||||||1|000074AEB
21||||CS1|||1999|07|16|||||||09|00|000083CD5
30|C|7439-97-6||MG/KG|||||||||||||||U|0.0092||||||0000958F4

20|1|LRV|1|LRQ||||1999|09|09||||||1|0001066A6
30|C|7439-97-6|||||||||||||||||||||||0001173CB
32|||||||||||||000127D02
34||||253.70|0.2|5||||||00013852D
```

2.2 Mercury Instrument Calibration Standards: Blank (S0) and Four Other Standards

```
20|1|S0|1|STB||20596|MAX123|1999|09|09|08|44||||1|00010936F
21||||CS1||0PPB|1999|09|09|||||TESLAB||07|00|000119F0C
22|||||||||||1.0||||00012A773
30|C|7439-97-6|||||T|0.0|||||||||||U|0.018||0.0122||||00013BAD9
31|W|C||253.70|0.0122|||||||||00014C4EC

20|1|S0.2|1|STC||20596|MAX123|1999|09|09|08|48||||1|00015D392
21||||CS1||0.2PPB|1999|09|09|||||TESLAB||07|00|00016DF8F
22|||||||||||1.0||||00017E7F6
```

```
30|C|7439-97-6|||||T|0.2||||||||||U|0.018||0.0896||||00018FB5E
31|W|C||253.70|0.0896|||||||||000190571

20|1|S1.0|1|STD||20596|MAX123|1999|09|09|08|53||||1|000201412
21||||CS1||1.0PPB|1999|09|09|||||TESLAB||07|00|00021200E
22||||||||||1.0||||000222875
30|C|7439-97-6|||||T|1.0||||||||||U|0.018||1.0128||||000233BDC
31|W|C||253.70|1.0128|||||||||0002445EF

20|1|S2.0|1|STD||20596|MAX123|1999|09|09|08|57||||1|000255495
21||||CS1||2.0PPB|1999|09|09|||||TESLAB||07|00|000266092
22||||||||||1.0||||0002768F9
30|C|7439-97-6|||||T|2.0||||||||||U|0.018||2.0055||||000287C61
31|W|C||253.70|2.0055|||||||||000298674

20|1|S5.0|1|STD||20596|MAX123|1999|09|09|09|01||||1|000309513
21||||CS1||5.0PPB|1999|09|09|||||TESLAB||07|00|00031A113
22||||||||||1.0||||00032A97A
30|C|7439-97-6|||||T|5.0|P|0.9997|||||||||U|0.018||4.9952||||00033BCE5
31|W|C||253.70|4.9952|||||||||00034C6F8
```

2.3 Spike Sample Recovery and Duplicates Performed on Different Samples (QC Codes FLD, LSF, FLD, LD2)

```
20|1|MAX123|F|FLD||20596|MAX123|1999|09|09|13|20||G|0.20|1|002106798
21||LOW||CS1||S308233-01|1999|09|09||1999|08|24|||07|00|0021175EF
22||||||||||100|1.0|||91.5|002127FB4
30|C|7439-97-6||MG/KG|BDL|0.1093||||||||||||U|0.0092||0.0049||||002159EC0
31|W|C||253.70|0.0049|||||||||00216A8E3

20|1|MAX123S|F|LSF||20596|MAX123|1999|09|09|13|25||G|0.20|1|00229534B
21||LOW||CS1||S308233-03|1999|09|09||1999|08|24|||07|00|0023061A2
22||||||||||100|1.0|||91.5|002316B67
30|C|7439-97-6||MG/KG||0.5664|F|0.55|P|103||||||75|125||U|0.0092||1.0366||||00232807A
31|W|C||253.70|1.0366|||||||||002338A9D

20|1|MAX126|F|FLD||20596|MAX123|1999|09|09|13|30||G|0.20|1|00217B9F5
21||LOW||CS1||S308233-06|1999|09|09||1999|08|24|||07|00|00218C84C
22||||||||||100|1.0|||85.6|00219D211
30|C|7439-97-6||MG/KG||1.5053||||||||||||U|0.0092||2.5771||||00222F11D
31|W|C||253.70|2.5771|||||||||00223FB40

20|1|MAX126D|F|LD2||20596|MAX123|1999|09|09|13|35||G|0.20|1|002240C9D
21||LOW||CS1||S308233-07|1999|09|09||1999|08|24|||07|00|002251AF4
22||||||||||100|1.0|||85.1|0022624BC
30|C|7439-97-6||MG/KG|BDL|0.1175|||P|200|||L|0.0383||||*|U|0.0092||0.0028||||002273795
31|W|C||253.70|0.0028|||||||||0022841B9
```

2.4 Duplicates and Spike Sample Recovery Performed on the Same Sample (QC Codes FLD, LD2, LSF)

```
20|1|MAX126|F|FLD||20596|MAX123|1999|09|09|16|10||G|0.20|1|002106798
21||LOW||CS1||S308233-06|1999|09|09||1999|08|24|||07|00|0021175EF
22||||||||||100|1.0|||91.5|002127FB4
30|C|7439-97-6||MG/KG||0.6429||||||||||||U|0.0092||1.1765||||002159EC0
31|W|C||253.70|1.1765|||||||||00216A8E3

20|1|MAX126D|F|LD2||20596|MAX123|1999|09|09|16|15||G|0.20|1|002240C9D
21||LOW||CS1||S308233-07|1999|09|09||1999|08|24|||07|00|002251AF4
22||||||||||100|1.0|||90.9|0022624BC
30|C|7439-97-6||MG/KG||0.2342|||P|94|||L|0.0364||||*|U|0.0092||0.4286||||002273795
31|W|C||253.70|0.4286|||||||||0022841B9
```

```
20|1|MAX126S|F|LSF||20596|MAX123|1999|09|09|16|20||G|0.20|1|00229534B
21||LOW||CS1||S308233-08|1999|09|09||1999|08|24|||07|00|0023061A2
22||||||||||100|1.0|||91.5|002316B67
30|C|7439-97-6||MG/KG||0.9710|F|0.55|P|60|||||N|75|125||U|0.0092||1.7769||||00232807A
31|W|C||253.70|1.7769|||||||||002338A9D
```

2.5 Initial Calibration Verification (ICV) with LVM QC Code

```
20|1|ICV1A|1|LVM||20596|MAX123|1999|09|09|09|06||||1|00035D687
21||||CS1||ICV-5|1999|09|09|||07|00|ICF(0791)||07|00|00036E25E
22||||||||||||2.0||||00037EAC6
30|C|7439-97-6||UG/L||4.91|T|4.9|P|100||||||80.0|120.0||U|0.018||2.4559||||00038FFD0
31|W|C||253.70|2.4559|||||||||0003909FC
```

2.6 Laboratory Control Sample (Solid) with LCM QC Code

```
20|1|LCSSC3|F|LCM||20596|MAX123|1999|09|09|12|24||G|0.20|1|001256DBA
21||||CS1||LCSHG|1999|09|09|||||QAL-0287||07|00|001267B1B
22||||||||||100|1.0||||001278443
30|C|7439-97-6||MG/KG||4.6|T|4.2|P|110||||||2.8|6.0||U|0.0092||9.2000||||00128996D
31|W|C||253.70|2.7719|||||||||00129A39A
```

3.0 CYANIDE

3.1 Start of a Cyanide Run with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|01|14|09|AS|ILM05.3|ABC|TESLAB||||68-D2-0039|C1||TEST LABS INC.|7|00000189C
16|1999|09|01|15|03|Y||||000012033

20|1|MDL|1|MDQ||||1999|07|15||||||1|000044A74
21||||NP1|||1999|07|15|||||||10|30|000053CD5
30|C|57-12-5||UG/L|||||||||||||||U|1.7||||||0000656DC

20|1|MDL|1|MDQ||||1999|07|15||||||1|000044A74
21||||DW1|||1999|07|15|||||||10|30|000053CD5
30|C|57-12-5||UG/L|||||||||||||||U|1.8||||||0000656DC

20|1|MDL|F|MDQ||||1999|07|16||||||1|000044A74
21||||DS2|||1999|07|16|||||||07|45|000053CD5
30|C|57-12-5||MG/KG|||||||||||||||U|0.092||||||0000656DC

20|1|LRV|1|LRQ||||1999|09|01||||||1|000066486
30|C|57-12-5|||||||||||||||||||||||||000076FDA
32||||||||||IT|45.00||||000087917
34||||620.00|10|400||||||000098169
```

3.2 Cyanide Instrument Calibration Standards: Blank (S0) and Five Other Standards

```
20|1|S0|1|STB||20596|MAX123|1999|09|01|14|09||||1|000108FA1
21||||NP1||0PPB||||||||TESLAB||||000119B3E
22||||||||||||1.0||||00012A3A5
30|C|57-12-5|||||T|0.0||||||||||U|1.7||0.3543||||00013B48B
31|W|C||620.00|0.3543|||||||||00014BD34

20|1|S10|1|STC||20596|MAX123|1999|09|01|14|10||||1|00015CB95
21||||NP1||10PPB||||||||TESLAB||||00016D763
22||||||||||||1.0||||00017DFCA
30|C|57-12-5|||||T|10.0||||||||||U|1.7||11.1700||||00018F0D2
31|W|C||620.00|11.1700|||||||||00019F97B

20|1|S40|1|STD||20596|MAX123|1999|09|01|14|11||||1|0002007E0
21||||NP1||40PPB||||||||TESLAB||||0002113B1
22||||||||||||1.0||||000221C18
```

```
30|C|57-12-5|||||T|40.0||||||||||U|1.7||38.4000||||000232D23
31|W|C||620.00|38.4000|||||||||0002435CC

20|1|S100|1|STD||20596|MAX123|1999|09|01|14|12||||1|00025445F
21||||NP1||100PPB|||||||TESLAB||||00026505D
22||||||||||1.0||||0002758C4
30|C|57-12-5|||||T|100.0||||||||||U|1.7||99.7400||||000232D23
31|W|C||620.00|99.7400|||||||||0002972A5

20|1|S200|1|STD||20596|MAX123|1999|09|01|14|12||||1|000308139
21||||NP1||200PPB|||||||TESLAB||||000318D38
22||||||||||1.0||||00032959F
30|C|57-12-5|||||T|200.0||||||||||U|1.7||201.3000||||00033A6D8
31|W|C||620.00|201.3000|||||||||00034AF81

20|1|S400|1|STD||20596|MAX123|1999|09|01|14|13||||1|00035BE18
21||||NP1||400 PPB|||||||TESLAB||||00036CA19
22||||||||||1.0||||00037D280
30|C|57-12-5|||||T|400.0|P|1.0000|||||||||U|1.7||399.5000||||00038E3BB
31|W|C||620.00|399.5000|||||||||00039EC64
```

3.3 Preparation Blank (Soil) with LRB QC Code

```
20|1|PBSD1|F|LRB||20596|MAX123|1999|09|01|14|23||G|1.00|1|000928FA0
21||||DS2||PB|1999|08|30|||||||08|30|000939A40
22||||||||||50|1.0||||00094A30C
30|C|57-12-5||MG/KG|BDL|1.0000||||||||||||U|0.092||-0.0030||||00095B433
31|W|C||620.00|-0.0030|||||||||00096BE6F
```

3.4 Laboratory Control Sample (Soil) with LCM QC Code

```
20|1|LCSSD1|F|LCM||20596|MAX123|1999|09|01|14|24||G|1.00|1|00097CF4D
21||||DS2||LCSCN|1999|08|30|||||QAL-0689||08|30|00098DCB0
22||||||||||50|1.0||||00099E57C
30|C|57-12-5||MG/KG||5.0|T|5.6|P|89||||||4.3|6.9||U|0.092||100.0933||||00100F89B
31|W|C||620.00|100.0933|||||||||001010315
```

3.5 Continuing Calibration Verification (CCV) with LVC QC Code

```
20|1|CCV11|1|LVC||20596|MAX123|1999|09|01|14|30||||1|0015045A3
21||||NP1||200 PPB|||||||TESLAB||||0015151A2
22||||||||||1.0||||001525A09
30|C|57-12-5||UG/L||188.48|T|200.0|P|94||||||85.0|115.0||U|1.7||188.4772||||001536E87
31|W|C||620.00|188.4772|||||||||001547916
```

3.6 Spike Sample Recovery and Post Distillation Spike Sample Recovery Performed on the Same Sample (QC Codes FLD, PDO, LSF, PDF)

```
20|1|MAX123|F|FLD||20596|MAX123|1999|09|01|14|35||G|1.06|1|001955D8E
21||LOW||DS2||S308233-01|1999|08|30||1999|08|24|||08|30|001966BDF
22||||||||||50|1.0|||71.0|001977578
30|C|57-12-5||MG/KG|LTC|0.2952||||||||||||U|0.092||4.4441||||002009309
31|W|C||620.00|4.4441|||||||||002019D4B

20|1|MAX123|F|PDO||20596|MAX123|1999|09|01|14|35||G|1.06|1|00202AE62
21||LOW||DS2||S308233-01|1999|08|30||1999|08|24|||08|30|00203BCB3
22||||||||||50|1.0|||71.0|00204C64C
30|C|57-12-5||UG/L|LTC|4.44||||||||||||U|1.4||4.4441||||00207E3DD
31|W|C||620.00|4.4441|||||||||00208EE1F

20|1|MAX123S|F|LSF||20596|MAX123|1999|09|01|14|36||G|1.02|1|00209FF7A
21||LOW||DS2||S308233-02|1999|08|30||1999|08|24|||08|30|002100DCB
22||||||||||50|1.0|||71.0|002111767
```

```
30|C|57-12-5||MG/KG||4.6341|F|6.90|P|63|||||N|75|125||U|0.092||67.1210||||0021228D6
31|W|C||620.00|67.1210|||||||||002133324

20|1|MAX123A|F|PDF||20596|MAX123|1999|09|01|14|37||G|1.06|1|0021444AD
21||LOW||||S308233-03|1999|08|30||1999|08|24|||||0021552FE
22||||||||||50|1.0|||71.0|002165C98
30|C|57-12-5||UG/L||21.23|F|20.0|P|84|||||||||U|1.4||21.2279||||0021770C0
31|W|C||620.00|21.2279|||||||||002187B4E
```

APPENDIX B - Modified Analysis

THIS PAGE INTENTIONALLY LEFT BLANK

Appendix B - Modified Analysis

Table of Contents

Section		Page
1.0	SCOPE AND APPLICATION	5
2.0	SUMMARY OF METHOD	5
3.0	DEFINITIONS	5
4.0	INTERFERENCES	6
	4.1 Spectral Interferences	6
	4.2 Nonspectral Interferences	7
5.0	SAFETY	7
6.0	EQUIPMENT AND SUPPLIES	8
	6.1 Glassware/Labware	8
	6.2 Atomic Absorption Spectrophotometer	8
7.0	REAGENTS AND STANDARDS	9
	7.1 Reagents	9
	7.2 Standards	9
8.0	SAMPLE COLLECTION, PRESERVATION, AND STORAGE	11
	8.1 Sample Collection and Preservation	11
	8.2 Procedure for Sample Storage	11
	8.3 Procedure for Sample Digestate Storage	11
	8.4 Contract Required Holding Time	11
9.0	CALIBRATION AND STANDARDIZATION	12
	9.1 Instrument Operating Conditions	12
	9.2 Graphite Furnace Atomic Absorption (GFAA) Instrument Calibration Procedure	12
10.0	PROCEDURE	13
	10.1 Sample Preparation	13
	10.2 Sample Analysis	14
11.0	DATA ANALYSIS AND CALCULATIONS	16
	11.1 Water/Aqueous Sample Calculation	16
	11.2 Soil Sample Calculation	16
	11.3 Corrections For Sample Dilutions	17
12.0	QUALITY CONTROL	17
13.0	METHOD PERFORMANCE	17
14.0	POLLUTION PREVENTION	17
15.0	WASTE MANAGEMENT	17
16.0	REFERENCES	17
17.0	TABLES/DIAGRAMS/FLOWCHARTS	17

THIS PAGE INTENTIONALLY LEFT BLANK

MODIFIED ANALYSIS

The Contractor may be requested by USEPA to perform modified analyses. These modifications will be within the scope of this SOW and may include, but are not limited to, analysis of additional analytes and/or lower quantitation limits. These requests will be made by the USEPA Regional CLP Project Officer (CLP PO), USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (PM), and USEPA Contracting Officer (CO) in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. All contract requirements specified in the SOW/Specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample scheduling.

GRAPHITE FURNACE ATOMIC ABSORPTION METHOD

1.0 SCOPE AND APPLICATION

This method is a graphite furnace atomic absorption spectroscopy procedure that is used to analyze water, sediment, sludge, and soil samples taken from hazardous waste sites. The following metals: arsenic, lead, selenium, and thallium that are contained in the Target Analyte List (TAL) in Exhibit C may be quantitated by the Graphite Furnace Atomic Absorption (GFAA) method.

2.0 SUMMARY OF METHOD

Water and soil samples are treated with acids and heat to solubilize the metals present. These digestates are then analyzed for trace metals by the Graphite Furnace Atomic Absorption (GFAA) spectroscopic technique. In this technique, a tube of graphite is located in the sample compartment of the Atomic Absorption (AA) spectrometer, with the light passing through it. A small volume of sample solution is quantitatively placed into the tube, normally through a sample injection hole located in the center of the tube wall. The tube is heated through a programmed temperature sequence until finally the analyte present in the sample is dissociated into atoms and atomic absorption occurs.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements in water and soil/sediments. Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 milligrams per Liter (mg/L). In addition, total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects.

Interferences from the Graphite Furnace Atomic Absorption (GFAA) technique can be divided into two broad categories, spectral and nonspectral interferences. Spectral interferences are those resulting from light absorption by molecules or by atoms other than those of the analyte element; that is, spectral interference exists if the atomic absorption profile of an element overlaps the emission line of another. Nonspectral interferences are those which affect the production or availability of analyte atoms which create the measured atomic absorption.

4.1 Spectral Interferences

4.1.1 Emission Interference - this interference arises when the intense light emitted by the hot graphite tube reaches the instrument’s light detector, the Photomultiplier Tube (PMT). This problem is manifested by increased signal variability (noise) which degrades analytical performance. In severe circumstances, emission interference may temporarily blind the PMT, resulting in erratic, meaningless readings at atomization.

4.1.2 Background Absorption - this is the most severe spectral interference encountered with graphite furnace analyses. Background absorption is a nonspecific attenuation of light at the analyte wavelength caused by matrix components in the sample. Unlike atomic absorption, background absorption is broad band, sometimes covering tens or even hundreds of nanometers. This broad band absorption normally is due to molecular absorption or light scattering caused by undissociated sample matrix components in the light path at atomization. Since background absorption is broad band, the chance of overlap with a desired analyte wavelength is significant.

4.1.3 Emission interference is controlled by primarily by spectrometer optical design. Techniques for controlling and reducing background absorption include matrix modification (sample treatment) and optical background correction. Through matrix modification, a reagent or “matrix modifier” is added to the sample or standard. The matrix modifier is selected to generate either an increased matrix volatility or decreased analyte volatility. One type of background correction, Zeeman, can correct for higher and more spectrally complicated background absorption and provide more precise and accurate analytical results. Zeeman background correction uses the principle that the electronic energy levels of an atom placed in a strong magnetic field are changed thereby changing the atomic spectra; the spectral nature of background absorption, on the other hand is unaffected by a magnetic field.

4.2 Nonspectral Interferences

In order for atomic absorption to occur, free atoms of the analyte element must be present in the spectrometer light path. Nonspectral interferences result when diverse components in the sample matrix inhibit the formation of free analyte atoms. An often used approach to compensate for nonspectral interferences is known as the “Method of Standard Additions”.

5.0 SAFETY

See Section 1.14 in Exhibit D - Introduction to Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here, however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 Glassware/Labware

6.1.1 250 milliliter (mL) beaker or other appropriate vessel

6.1.2 Watch glasses

6.1.3 Funnels

6.1.4 Graduated cylinders

6.1.5 Various volumetric flasks (Type A)

6.1.6 Thermometer that covers a range of 0-200°C

6.1.7 Whatman No. 42 filter paper or equivalent

6.1.8 Hot plate, block digester, or other heating source capable of maintaining 92-95°C

6.1.9 Balances - Analytical Balance, 300 gram (g) capacity, and minimum ±0.01 g.

6.2 Atomic Absorption Spectrophotometer - with graphite furnace atomizer and background correction. Hollow Cathode Lamp (HCL) and/or Electrodeless Discharge Lamp (EDL).

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. (Redistilled acids are acceptable.)

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-77). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2 Nitric acid - Concentrated (specific gravity 1.41).

7.1.3 Nitric acid, 5% (v/v) - Add 50 milliliters (mL) conc. HNO_3 to 500 mL reagent water; dilute to 1 Liter (L).

7.1.4 Hydrochloric acid - Concentrated (specific gravity 1.19).

7.1.5 Hydrogen peroxide (30%)

7.1.6 Matrix Modifiers

7.1.6.1 Ammonium Phosphate solution (40%): Dissolve 40 grams (g) of ammonium phosphate, $(NH_4)_2PO_4$ (analytical reagent grade) in reagent water and dilute to 100 mL.

7.1.6.2 Calcium Nitrate solution: Dissolve 11.8 g of calcium nitrate, $Ca(NO_3)_2 \bullet 4H_2O$ (analytical reagent grade) in reagent water and dilute to 100 mL. 1 mL = 20 mg Ca.

7.1.6.3 Lanthanum Nitrate solution: Dissolve 58.64 g of American Chemical Society (ACS) reagent grade 2. La_2O_3 in 100 mL conc. HNO_3 and dilute to 1000 mL with reagent water. 1 mL = 50 mg La.

7.1.6.4 Nickel Nitrate solution, 5%: Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \bullet 6H_2O$ in reagent water and make up to 100 mL.

7.1.6.5 Nickel Nitrate solution, 1%: Dilute 20 mL of the 5% nickel nitrate to 100 mL with reagent water.

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E, Section 8.0. The Contractor must be able to verify that the standards are certified. Manufacturer’s certificates of analysis must be retained by the Contractor and presented upon request.

7.2.2 Stock Standard Solutions

7.2.2.1 Stock standard solutions may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 hour at 105°C unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.) Typical stock solution preparation procedures follow.

7.2.2.2 Arsenic solution, stock [1 mL = 1 mg As (1000 mg/1)] - Dissolve 1.320 g of As_2O_3 in 100 mL of reagent water containing 0.4 g NaOH. Acidify the solution with 20 mL conc. HNO_3 and dilute to 1 L.

7.2.2.3 Lead solution, stock [1 mL = 1 mg Pb (1000 mg/L)] - Dissolve 1.599 g of $Pb(NO_3)_2$ in reagent water. When solution is complete, acidify with 10 mL of conc. HNO_3 and dilute to 1 L with reagent water.

7.2.2.4 Selenium solution, stock [1 mL = 1 mg Se (1000 mg/L)] - Dissolve 0.3453 g of H_2SeO_3 (actual assay 94.6%) in reagent water and make up to 200 mL.

7.2.2.5 Thallium solution stock [1 mL = 1 mg Tl (1000 mg/L)] - Dissolve 1.303 g of $TlNO_3$ in reagent water. Add 10 mL of conc. nitric acid and dilute to 1 L with reagent water.

7.2.3 Working Standards

7.2.3.1 Secondary Dilution Standards

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions". The calibration standards must be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed after sample preparation.

7.2.3.2 Calibration Blank

Prepared by diluting 1 mL of (1+1) HNO_3 and 2 mL 30% H_2O_2 to 100 mL with reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All samples must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection. All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until digestion.

8.1.1 Dissolved Metals

For the determination of dissolved metals, the sample must be filtered through a 0.45 micrometer (µm) pore diameter membrane filter at the time of collection or as soon as possible. Use a portion of the sample to rinse the filter flask, discard this portion, and collect the required volume of filtrate. Preserve the filtrate with nitric acid to pH less than 2 immediately after filtration.

8.2 Procedure for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery of a complete, reconciled data package to USEPA. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

8.3 Procedure for Sample Digestate Storage

Sample digestates must be stored until 365 days after delivery of a complete, reconciled data package to USEPA.

8.4 Contract Required Holding Time

The maximum holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR).

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Conditions

Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL), precision, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument calibrated range. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2 Graphite Furnace Atomic Absorption (GFAA) Instrument Calibration Procedure

9.2.1 Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time shall be included in the raw data.

9.2.2 Calibration standards shall be prepared fresh daily or each time an analysis is to be made and discarded after use. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One atomic absorption calibration standard shall be at the CRQL. The calibration standards shall be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following sample preparation.

9.2.3 Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Date and time of preparation and analysis shall be given in the raw data.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact Sample Management Office (SMO) to inform them of the problem. SMO will contact the Region for instructions. The Region will either require that no sample analyses be performed or will require that a reduced volume be used for the sample analysis. No other changes in the analyses will be permitted. The Contractor shall document the Region's decision in the Sample Delivery Group (SDG) Narrative.

10.1.2 If multiphase samples (e.g., two-phase liquid sample, oily sludge/sandy soil sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample and analyze one or more of the phases, separately. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.1 If all of the phases are not amenable to analysis (i.e., outside scope), the Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is(are) amenable to analysis. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other changes in the analyses will be permitted. The Contractor shall document the Region's decision in the SDG Narrative.

10.1.3 Water/Aqueous Sample Preparation

10.1.3.1 Shake sample and transfer 50-100 mL of well-mixed sample to a 250 mL heating vessel, add 1 milliliter (mL) of (1+1) HNO_3 and 2 mL of 30% H_20_2 to the sample. Cover with watch glass or similar cover and heat on a hot plate, block digester, or equivalent heating source which is adjustable and capable of maintaining a temperature of 92-95°C for 2 hours or until sample volume is reduced to between 25 and 50 mL, making certain sample does not boil. Cool sample and filter to remove insoluble material.

NOTE: In place of filtering, the sample, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

Adjust sample volume to 50-100 mL with reagent water. The sample is now ready for analysis. Concentrations so determined shall be reported as "total". If volumes less than 100 mL are used, all other reagents shall be reduced appropriately (e.g., if 50 mL is

used, reduce reagent volumes by one-half). The final volume of the digestate must equal the initial volume of the sample aliquot.

10.1.4 Soil/Sediment Sample Preparation

10.1.4.1 A representative 1.0 gram (g) (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Nitric acid is employed as the final reflux acid for the Graphite Furnace Atomic Absorption (GFAA) analysis of As, Pb, Se, and Tl. A separate sample shall be dried for a percent solids determination.

10.1.4.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a beaker.

10.1.4.3 Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 92-95°C on hot plate or block digestor, and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.

10.1.4.4 After the second reflux step has been completed and the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the heat source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heat vessel.

Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

10.1.4.5 If the sample is being prepared for the GFAA analysis of As, Pb, Se, and Tl, continue heating the acid-peroxide digestate until the volume has been reduced to approximately 2 mL, add 10 mL of reagent water, and warm the mixture. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 mL with reagent water.

NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

Dilute the digestate 1:1 (200 mL final volume) with acidified water to maintain constant acid strength. For analysis, withdraw aliquots of appropriate volume, and add any required reagent or matrix modifier. The sample is now ready for analysis.

10.2 Sample Analysis

10.2.1 Set up instrument with proper operating parameters established in Section 9.1.

10.2.2 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using calibration standard solutions.

10.2.3 Instrument Parameters - Suggested Conditions

10.2.3.1 Arsenic

10.2.3.1.1 Wavelength: 193.7 nm

10.2.3.1.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.1.3 The use of background correction is required. Background correction made by the deuterium arc method does not adequately compensate for high levels of certain interferents (i.e., Al, Fe). If conditions occur where significant interference is suspected, the laboratory must switch to an alternate wavelength or take other appropriate actions to compensate for the interference effects.

10.2.3.1.4 The use of the Electrodeless Discharge Lamps (EDLs) for the light source is recommended.

10.2.3.2 Lead

10.2.3.2.1 Wavelength: 283.3 nm

10.2.3.2.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.2.3 The use of background correction is required.

10.2.3.2.4 Greater sensitivity can be achieved using the 217.0 nm line, but the optimum concentration range is reduced. The use of an EDL at this lower wavelength has been found to be advantageous. Also a lower atomization temperature (2400°C) may be preferred.

10.2.3.2.5 To suppress sulfate interference (up to 1500 ppm), lanthanum is added as the nitrate to both samples and calibration standards.

10.2.3.2.6 Since glassware contamination is a severe problem in lead analysis, all glassware should be cleaned immediately prior to use, and once cleaned, should not be open to the atmosphere except when necessary.

10.2.3.3 Selenium

10.2.3.3.1 Wavelength: 196.0 nm

10.2.3.3.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.3.3 Selenium analysis suffers interference from chlorides (>800 mg/L) and sulfate (>200 mg/L). For the analysis of industrial effluents and samples with concentrations of sulfate from 200 to 2000 mg/L, both samples and standards should be prepared to contain 1% nickel.

10.2.3.3.4 The use of the EDL for the light source is recommended.

10.2.3.4 Thallium

10.2.3.4.1 Wavelength: 276.8 nm

10.2.3.4.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.4.3 The use of background correction is required.

10.2.3.4.4 Nitrogen may also be used as the purge gas.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Water/Aqueous Sample Calculation

The concentrations determined in the digestate are to be reported in units of microgram per Liter (µg/L):

EQ. 1 Aqueous Sample Concentration

$$\text{Concentration} = C \times \frac{V_f}{V_i}$$

WHERE,

C = Instrument value in µg/L

V_f = Final digestion volume

V_i = Initial digestion volume

11.2 Soil Sample Calculation

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of milligram per kilogram (mg/kg):

EQ. 2 Soil Sample Concentration

$$\text{Concentration (dry wt.) (mg/kg)} = \frac{C \times V}{W \times S}$$

WHERE,

C = Concentration (mg/L)

V = Final volume in liters after sample preparation

W = Weight in kg of wet sample

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

11.3 Corrections For Sample Dilutions

If dilutions were performed, the appropriate factor shall be applied to the sample values as follows:

EQ. 3 Correction for Dilution

$$C\ (\mu g/L) = C_i \times DF$$

WHERE,
- C = Concentration of analyte in sample
- C_i = Instrument value concentration
- DF = Dilution Factor

12.0 QUALITY CONTROL

For specific Quality Control (QC) requirements, the Contractor shall follow the instructions provided by the USEPA Region requesting the analysis.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 1.15 in Exhibit D - Introduction to Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 1.16 in Exhibit D - Introduction to Analytical Methods.

16.0 REFERENCES

16.1 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 206.2. March 1983.

16.2 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 239.2. March 1983.

16.3 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 270.2. March 1983.

16.4 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 279.2. March 1983.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Not applicable.