EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND REQUIREMENTS

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Exhibit E - Quality Assurance/Quality Control Procedures and Requirements

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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 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.

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 methods. Inaccuracies can also result from causes other than unanticipated matrix effects, such as sampling artifacts, equipment malfunctions, and operator error. Therefore, the QC component of each method is indispensable.

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, 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 necessary components of a complete QA/QC program include internal QC criteria that demonstrate acceptable levels of performance, as determined by QA review. External review of data and procedures is accomplished by the monitoring activities of the National Program Office, 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 CLP Project Officer (CLP PO).

2.1.2 This exhibit does not provide specific instructions for constructing QA Plans, QC systems, or a QA organization. It is, however, an explanation of the QA/QC requirements of the program. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a Quality Assurance Plan (QAP) and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides USEPA with a complete stand-alone data package, and limits the need for contact with the Contractor or 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, USEPA requires the following from the Contractor:

- Preparation of, and adherence to, a written QAP, the elements of which are designated in Section 4.0 of this exhibit;

- Preparation of, and adherence to, Standard Operating Procedures (SOPs), as described in Section 5.0 of this exhibit;

- Adherence to the analytical methods and associated QC requirements specified in the contract;

- Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical supply houses;

- Submission of all raw data and pertinent documentation for Regional review;

- Participation in the analysis of laboratory evaluation samples, including adherence to corrective action procedures;

- Submission, upon request, of instrument data tapes and applicable documentation for tape audits, including a copy of the Sample Data Package;

- Participation in on-site laboratory evaluations, including adherence to corrective action procedures; and

- Submission of all original documentation generated during sample analyses for USEPA review.

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

The Contractor shall adhere to 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 QUALITY ASSURANCE PLAN (QAP)

4.1 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.

4.1.1 As evidence of such a program, the Contractor shall prepare a written Quality Assurance Plan (QAP) which describes the procedures that are implemented to:

- 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 to provide data which are technically sound and legally defensible.

4.1.2 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. Where applicable, 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 within 7 days of written request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO). Additional information relevant to the preparation of a QAP can be found in USEPA and American Society for Testing and Materials (ASTM) publications.

4.2 Required Elements of a Quality Assurance Plan

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

A. Organization and Personnel

1. QA Policy and Objectives

2. QA Management

   a. Organization

   b. Assignment of QA/QC Responsibilities

c. Reporting Relationships

d. QA Document Control Procedures

e. QA Program Assessment Procedures

3. Personnel

a. Resumes

b. Education and Experience Pertinent to this Contract

c. Training 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

1. Data Quality Assurance

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures

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

4.3 Updating and Submitting the Quality Assurance Plan

4.3.1 Initial Submission: During the contract solicitation process, the Contractor is required to submit its QAP to the CLP Contracting Officer. Within 60 days after contract award, the Contractor shall maintain on-file a revised QAP, fully compliant with the requirements of this contract. 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.

4.3.1.1 The Contractor shall send a copy of the latest version of the QAP within 7 days of a request from a USEPA Regional Contract

Laboratory Program Project Officer (CLP PO). The USEPA requestor will designate the recipients.

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

- C USEPA modifies the contract;
- C USEPA notifies the Contractor of deficiencies in the QAP document;
- C USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- C The Contractor identifies deficiencies resulting from their internal review of their QAP document;
- C The Contractor's organization, personnel, facility, equipment, policy, or procedures change; or
- C The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, or procedure changes.

4.3.2.1 The Contractor shall amend the QAP within 30 days of when the circumstances listed above 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, or highlighting the change by underlining the change, bold printing the change, or using a different print font). 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.

4.3.2.2 The Contractor shall send a copy of the latest version of the QAP document within 7 days of a written request by the USEPA Regional CLP PO, as directed. The USEPA requestor will designate the recipients.

4.4 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions: reduction in the numbers of samples sent under this contract; suspension of sample shipment to the Contractor; data package audit; electronic data audit; an on-site laboratory evaluation; remedial performance evaluation sample, and/or contract sanctions.

5.0 STANDARD OPERATING PROCEDURES (SOPs)

5.1 Introduction

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.

5.1.1 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.

5.1.2 All SOPs shall reflect 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) contract's 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 may be asked to demonstrate the application of the SOPs;
- Available to the designated recipients within 7 days, upon request by the USEPA Regional CLP Project Officer (CLP PO);
- 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 explaining 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 workstations, as appropriate; and

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

5.2 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;
- Scope and application;
- Definitions;
- Procedures;
- Quality Control (QC) limits;
- Corrective action procedures, including procedures for secondary review of information being generated;
- Documentation description and example forms;
- Miscellaneous notes and precautions; and
- References.

5.3 Requirements

The Contractor shall maintain the following SOPs:

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

5.3.2 Sample Receipt and Storage SOPs to include:

- Sample receipt and identification logbooks;
- Refrigerator temperature logbooks;
- Extract storage logbooks; and
- Security precautions.

5.3.3 Sample Preparation SOPs

5.3.3.1 Reagent purity check procedures and documentation

5.3.3.2 Extraction procedures

5.3.3.3 Extraction bench sheets

5.3.3.4 Extraction logbook maintenance

5.3.4 Glassware Cleaning SOPs

5.3.5 Calibration (Balances, etc.) SOPs to include:

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

5.3.6 Analytical Procedures (for each analytical system) to include:

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

5.3.7 Maintenance Activities (for each analytical system) to include:

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

5.3.8 Analytical Standards to include:

- 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

- C QC and corrective action measures.

5.3.9 Data Reduction Procedures to include:

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

5.3.10 Documentation Policy/Procedures to include:

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

5.3.11 Data Validation/Self-Inspection Procedures to include:

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

5.3.12 Data Management and Handling to include:

- C 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 to include hardware, software, and documentation or installation of 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;

- Specifications for staff training procedures; and

- Storage, retrieval and verification of the completeness and readability of HRGC/HRMS files transferred to magnetic media.

5.4 Updating and Submitting SOPs

5.4.1 Initial Submission: During the contract solicitation process, the Contractor is required to submit SOPs to the CLP Contracting Officer. Within 60 days after contract award, the Contractor shall maintain on-file a complete revised set of SOPs, fully compliant with the requirements of this contract. 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 of SOPs 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.

5.4.1.1 The Contractor shall send a complete set of the latest version of SOPs or individually requested SOPs within 7 days of a request from a USEPA Regional CLP PO.

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

- USEPA modifies the contract;

- C USEPA notifies the Contractor of deficiencies in their SOP's documentation;
- C USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- C The Contractor's procedures change;
- C The Contractor identifies deficiencies resulting from the internal review of SOP documentation; or
- C The Contractor identifies deficiencies resulting from the internal review of procedures.

5.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 30 days of when the circumstances listed above 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, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended/new SOPs shall have the date on which the changes were implemented.

5.4.2.2 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.

5.4.2.3 Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs. An alternate delivery schedule for submitting the letter and amended/new SOPs may be proposed by the Contractor, but it is the sole decision of the USEPA Contracting Officer to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe, in a letter to the USEPA Regional CLP PO and the Contracting Officer, why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 30 days for amending/writing new SOPs. An extension for amending/writing new SOPs beyond 30 days must be approved by the USEPA Contracting Officer. Similarly, an extension of up to 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs may be approved by the USEPA Regional CLP PO. An extension beyond the 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the USEPA Regional CLP PO and/or Contracting Officer.

5.5 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions:

- reduction in the number of samples sent under this contract;
- suspension of sample shipment to the Contractor; data package audit;
- electronic data audit;
- an on-site laboratory evaluation;
- remedial performance evaluation sample, and/or
- contract sanctions.

6.0 CONTRACT COMPLIANCE SCREENING (CCS)

6.1 Overview

6.1.1 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.

6.1.2 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.

6.2 CCS Results

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

6.3 CCS Trend Report

USEPA may generate a CCS trend report which summarizes CCS results over a given period of time. USEPA may 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 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. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe, in a letter to the USEPA Regional CLP PO and Contracting Officer, why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response to the CCS trend report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

6.4 Corrective Actions

6.4.1 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be re-written 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 Section 5.0.

6.4.2 If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to the following actions:

- reduction in the number of samples sent under the contract;
- suspension of sample shipment to the Contractor;
- data package audit;
- electronic data audit;
- an on-site laboratory evaluation;
- a remedial performance evaluation sample; and/or
- contract sanctions.

7.0 ANALYTICAL STANDARDS REQUIREMENTS

7.1 Overview

USEPA may not supply analytical reference standards for either direct analytical measurements or 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.

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

7.2.1 If the laboratory cannot obtain analytical reference standards, the laboratory may prepare its 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.

7.2.2 If required by the manufacturer, the chemical standards shall be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of chemicals is essential to safeguard them from decomposition.

7.2.3 The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the Contractor's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should 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 determined using the following equation:

EQ. 1

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

Where:

weight of pure compound = That required to prepare a specific volume of a solution standard of a specified concentration.

7.2.4 When compound purity is assayed to be 97% or greater, the weight may be used without correction to calculate the concentration of the stock standard. If the compound purity is assayed to be less than 97%, the weight shall be corrected when calculating the concentration of the stock solution.

7.2.5 Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. It is the Contractor's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards are correctly identified. Identification confirmation, when performed, shall use gas chromatography/mass spectrometry analysis on at least two different analytical columns, or other appropriate techniques.

7.2.6 Calculate the weight of material to be weighed out for a specified volume, taking into account the purity of the compound and the desired concentration. A second person shall verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights every 12 hours. All weighing shall be performed on an analytical balance to the nearest 0.1 mg and verified by a second person. The solvent used to dissolve the solute shall be compatible with the protocol in which the standard is to be used; the solute shall be soluble, stable, and nonreactive with the solvent. In the case of a multicomponent solution, the components must not react with each other.

7.2.7 Transfer the solute to a volumetric flask and dilute to the specified solution volume with solvent after ensuring dissolution of the solute in the solvent. Sonication or warming may be performed to promote dissolution of the solute. This solution shall be called the primary standard and all subsequent dilutions shall be traceable back to the primary standard.

7.2.8 Log notebooks are to be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the calculations for determining their concentrations are to be recorded and verified by a second person. All solution standards are to be refrigerated, if required, when not in use. All solution standards are to be clearly labeled to include the identity of the analyte or analytes, concentration, date prepared, solvent, and initials of the preparer.

7.3 Purchase of Chemical Standards Already in Solution

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

7.3.1 Contractors shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions they purchase.

7.3.2 The Contractor shall purchase standards for which the quality is 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 7.3.2.1 and 7.3.2.2). 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 7.3.2.4. If consistency 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 7.3.2.5. Then, the standard is certified to be within 10% of the target concentration using the equations in Section 7.3.2.6. If the procedure above is used, the supplier must document that the following have been achieved.

7.3.2.1 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".

7.3.2.2 Using the equation below, 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.

EQ. 2

$$\text{Mean} = \frac{\sum_{i=1}^{6} Y_i}{6}$$

7.3.2.3 The mean and variance of the six results for each solution shall be calculated using the following equation:

EQ. 3

$$\text{Variance} = \frac{\sum_{i=0}^{5} Y_i^2 - 6(\text{MEAN})^2}{5}$$

Where:

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 using the following equation.

EQ. 4

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

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

7.3.2.4 The test statistic shall be calculated using the following equation:

EQ. 5

$$\text{TEST STATISTIC} = \frac{\left| \frac{M_2}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}}$$

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

7.3.2.5 The test statistic shall be calculated using the following equation:

EQ. 6

$$\text{TEST STATISTIC} = \frac{\left| M_1 - \left(\frac{M_1}{1.6} \right) - \left(\frac{M_2}{2.2} \right) \right|}{\left(\frac{V_p}{4} \right)^{0.5}}$$

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.

7.3.2.6 The 95% confidence intervals for the mean result of each standard shall be calculated using the following equations:

EQ. 7

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

EQ. 8

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

EQ. 9

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

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

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

7.4 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.

7.5 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 used in the performance of Contract Laboratory Program (CLP) analysis conform to the requirements previously listed.

7.5.1 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.

7.5.2 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 receipt of the request to the designated recipients.

7.5.3 USEPA may generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards, or they may discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the USEPA

Regional CLP PO and Quality Assurance Technical Support (QATS), 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. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer, why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the standards documentation report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

7.5.4 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 Section 5.0.

7.6 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this Section, a Contractor may expect, but USEPA is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor; data package audit; electronic data audit; an on-site laboratory evaluation; a remedial laboratory evaluation sample, and/or contract sanctions.

8.0 DATA PACKAGE AUDITS

8.1 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 the 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 to include:

- 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.

8.2 Responding to the Data Package Audit Report

8.2.1 After completion of the data package audit, USEPA may 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.

8.2.2 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer, why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor’s response letter to the data package report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

8.2.3 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 Section 5.0.

8.3 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions:

- reduction in the number of samples sent under the contract;
- suspension of sample shipment to the Contractor;
- an on-site laboratory evaluation;
- data package audit;
- an electronic data audit;
- a remedial performance evaluation sample, and/or
- contract sanctions.

9.0 REGIONAL DATA REVIEW

9.1 Overview

Contractor data are generated to meet the specific needs of USEPA Regions. To verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end user. 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.

9.1.1 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 NPO, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for Program and Contractor administration, and management and may be used to take appropriate action to correct deficiencies in the Contractor’s performance.

10.0 PROFICIENCY TESTING

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor must participate in USEPA's Proficiency Testing Program. USEPA's Proficiency Testing Program involves the analysis of case-specific Performance Evaluation (PE) samples and the participation in interlaboratory 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.

10.1 Performance Evaluation (PE) Samples

10.1.1 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 National Program Office (NPO). PE samples will assist USEPA in monitoring Contractor performance.

10.1.2 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.

10.1.3 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 extracted 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 also be met. The PE sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

10.1.4 In addition to PE sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes/parameters 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/parameter. USEPA will notify the Contractor of unacceptable performance. USEPA reserves the right to adjust the PE sample acceptance windows to compensate for any unanticipated difficulties with a particular PE sample.

10.1.5 The Contractor shall demonstrate acceptable analytical performance for both identification and quantitation of PE sample analytes/ parameters. For unacceptable PE sample performance, USEPA may take, but is not limited to, the following actions:

- reduce value or rejection of data for the samples, SDG, or Case impacted;
- contract sanctions;
- reduction in the number of samples shipped to the laboratory;
- suspension of sample shipment;
- an on-site laboratory inspection;
- a full data package audit;
- electronic data audit; and/or

NOTE: A Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of full sample delivery.

10.2 Quarterly Blind (QB) Audits

10.2.1 QB Audits may be scheduled concurrently with all contract laboratories. A QB Audit is a unique analytical Case containing only PE samples (referred to as QB samples). The QB samples will be scheduled by the NPO through the Sample Management Office (SMO). QB samples will assist USEPA in monitoring Contractor performance.

10.2.2 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.

10.2.3 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 also be met. The QB sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

10.2.4 In addition to QB sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes/parameters 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/parameter. USEPA will notify the Contractor of unacceptable performance. USEPA reserves the right to adjust the PE sample acceptance windows to compensate for any unanticipated difficulties with a particular PE sample. The

Contractor's QB sample performance will be assessed into one of the following three categories:

10.2.4.1 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.

10.2.4.2 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.

10.2.4.3 Unacceptable Performance, Response Explaining Deficiencies Required: Score less than 75%. Deficiencies exist in the Contractor's performance to the extent that the NPO has determined that the Contractor has not demonstrated the capability to meet the contract requirements. Corrective action response required.

10.2.5 In the case of Section 10.2.4.2 or 10.2.4.3, the Contractor shall describe the deficiency(ies) and the action(s) taken in a corrective action letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and CLP Quality Assurance (QA) Coordinator within 14 days of receipt of notification from USEPA.

10.2.5.1 An alternate delivery schedule for the corrective action letter may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe, in a letter to the USEPA Regional CLP PO and Contracting Officer, why the laboratory is unable to meet the original delivery schedule listed in Section 10.2.5. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's corrective action letter. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

10.2.6 In the case of Section 10.2.4.2 or 10.2.4.3, 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 Section 5.0.

10.2.7 The Contractor shall be notified by the USEPA Contracting Officer concerning agreement or disagreement with the proposed remedy for unacceptable performance. For unacceptable QB sample performance (Section 10.2.4.3), USEPA may take, but is not limited to, the following actions:

- reduction in the number of samples shipped to the laboratory;
- suspension of sample shipment;
- an on-site laboratory inspection;

- C electronic data audit;
- C a full data package audit;
- C require the laboratory to analyze a Remedial QB sample; and/or
- C contract sanctions.

NOTE: A Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of full sample delivery.

10.2.8 A Remedial QB Audit is a unique analytical Case containing only QB samples. A Remedial QB Audit may be scheduled by the NPO with the Contractor(s) for any of the following reasons:

- C unacceptable PE sample performance;
- C unacceptable QB sample performance, and/or
- C major change in the laboratory (e.g., relocation, new owner, or high turn-over of key personnel).

Sections 10.2.2 through 10.2.7 apply to the Remedial QB Audit process.

10.3 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions:

- C reduction in the number of samples sent under the contract;
- C suspension of sample shipment to the Contractor;
- C a full data package audit;
- C an electronic data audit;
- C an on-site laboratory inspection;
- C a Remedial QB sample, and/or
- C contract sanctions.

11.0 ON-SITE LABORATORY EVALUATIONS

11.1 Overview

As dictated by a contract laboratory's performance, the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or their 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 an Evidentiary Audit.

11.2 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.

11.2.1 The Contractor should expect that items to be monitored will include, but not be limited to, the following:

- Size and appearance of the facility;
- Quantity, age, availability, scheduled maintenance, and performance of instrumentation;
- Availability, appropriateness, and utilization of the Quality Assurance Plan (QAP) and Standard Operating Procedures (SOPs);
- Staff qualifications, experience, and personnel training programs;
- 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.

11.2.2 Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are:

- previous on-site reports;
- performance evaluation sample scores;
- Regional review of data;
- Regional QA materials;

- C data audit reports;
- C results of Contract Compliance Screening (CCS); and
- C data trend reports.

11.3 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.

11.3.1 Procedural Audit: The procedural audit consists of review and examination of actual SOPs and accompanying documentation for the following laboratory operations:

- C sample receiving;
- C sample storage;
- C sample identification;
- C sample security;
- C sample tracking (from receipt to completion of analysis);
- C analytical project file organization and assembly; and
- C proper disposal of samples and cogenerated wastes.

11.3.2 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:

- C sample receiving;
- C sample storage;
- C sample identification;
- C sample security;
- C sample tracking (from receipt to completion of analysis); and
- C analytical project file organization and assembly.

11.3.3 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:

- C 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.

11.4 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.

11.5 Corrective Action Reports for Follow-Through to Quality Assurance and Evidentiary Audit Reports

Following an on-site laboratory evaluation, QA and/or evidentiary audit reports which discuss deficiencies found during the on-site evaluation may be sent to the Contractor. In a detailed letter, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies discussed during the on-site evaluation, and discussed in the report(s) to the USEPA Regional CLP PO, within 14 days of receipt of the report.

11.5.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe, in a letter to the USEPA Regional CLP PO and the Contracting Officer, why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the QA and evidentiary audit report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

11.5.2 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 Section 5.0.

11.6 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but USEPA is not limited to, the following actions:

- reduction in the number of samples sent under the contract;
- suspension of sample shipment to the Contractor;

- an on-site laboratory evaluation;
- data package audit;
- an electronic data audit;
- a remedial performance evaluation sample; and/or
- contract sanctions.

12.0 ELECTRONIC DATA AUDITS

12.1 Overview

12.1.1 Periodically, the USEPA requests the High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS) magnetic tapes from Contractors for a specific Case to perform tape audits. Generally, tape submissions and audits are requested for the following reasons:

- C Program overview;
- C Indication of data quality problems from QATS, Sample Management Office (SMO), or Regional data reviews;
- C Support for on-site audits; and
- C Specific Regional requests.

12.1.2 Depending upon the reason for an audit, tapes from a recent Case, a specific Case, or a laboratory evaluation sample may be requested. Tape audits provide a mechanism to assess adherence to contractual requirements, and to ensure the consistency of data reported on the hardcopy/floppy diskettes with that generated on the HRGC/HRMS tapes. 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, tape audits enable the USEPA to evaluate the utility, precision, and accuracy of the analytical methods.

12.1.3 The Contractor shall store <u>all</u> raw and processed HRGC/HRMS data on magnetic tape (i.e., magnetic tape, zip disk, diskette, etc.) or CD-ROM, in appropriate instrument manufacturer's format. This tape shall include data for samples, blanks, Laboratory Control Samples (LCSs), LCS duplicates (LCSDs), initial calibrations, calibration verifications, system performance checks (PFKs), Window Defining Mixture (WDM), isomer specificity checks, and column performance standards, as well as all Contractor-generated spectral libraries and quantitation reports required to generate the data package. The Contractor shall maintain a written reference logbook of tape files of the EPA sample number, calibration data, standards, blanks, matrix spikes, and duplicates. The logbook shall include EPA sample numbers and standard and blank IDs, identified by Case and Sample Delivery Group (SDG).

12.1.4 The Contractor is required to retain the HRGC/HRMS tapes for 3 years after submission of the reconciled Complete SDG File. When submitting HRGC/HRMS tapes to the USEPA, the following materials shall be delivered in response to the request.

12.1.4.1 All associated raw data files for samples, including laboratory evaluation and QC samples, blanks, LCS, laboratory control sample duplicate (LCSD), initial calibration and calibration verification standards, and system instrument Performance Check Solutions (WDM, CPS, and PFK).

12.1.4.2 All processed data files and quantitation output files associated with the raw data files described in Section 12.1.4.1.

12.1.4.3 All associated identifications and calculation files used to generate the data submitted in the data package.

12.1.4.4 A copy of the Contractor's written reference logbook relating tape files to EPA sample number, calibration data, standards, blanks, and LCS/LCSD. The logbook shall include EPA sample numbers and lab file identifiers for all samples, blanks, and standards, identified by Case and Sample Delivery Group.

12.1.4.5 A directory of files on all tapes.

12.1.4.6 A copy of the completed sample data package, if audit request within the period during which the Contractor must retain a copy of the sample data package.

12.1.4.7 A statement attesting to the completeness of the HRGC/HRMS data tape 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 tape submission:

- Contractor name;
- Date of submission;
- Case number;
- SDG number;
- HRGC/HRMS make and model number;
- Software version;
- Disk drive type (e.g., CDC, PRIAM, etc.);
- File transfer method (e.g., DSD, DTD, FTP, Aquarius, etc.);
- Data System Computer;
- System Operating Software;
- Data System Network;
- Tape Backup Software;
- Tape Backup Hardware;
- Data Analysis Software;
- Volume of data (in MB) backed up on each tape; and

- C Names and telephone numbers of two Contractor contacts for further information regarding the submission.

12.2 Submission of the HRGC/HRMS Tape

12.2.1 Upon request of the USEPA Regional CLP PO, the Contractor shall send the required HRGC/HRMS tapes and all necessary documentation to the designated recipient within 7 days of notification. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe, in a letter to the USEPA Regional CLP PO and the Contracting Officer, why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 7 days for submission of the HRGC/HRMS tapes. An extension beyond 7 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the USEPA Regional CLP PO.

NOTE: The HRGC/HRMS tapes shall be shipped according to the procedures described in Exhibit F.

12.3 Responding to the HRGC/HRMS Tape Audit Report.

After completion of the HRGC/HRMS tape audit, the USEPA may send a copy of the HRGC/HRMS tape audit report to the Contractor or may discuss the HRGC/HRMS tape 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 HRGC/HRMS tape audit report within 14 days of receipt of the report.

12.3.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the USEPA, represented by the USEPA Regional CLP PO, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why the laboratory is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the HRGC/HRMS tape report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the USEPA Regional CLP PO.

12.3.2 If new 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 Section 5.0.

12.4 Corrective Actions

12.4.1 If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the USEPA is not limited to, the following actions:

- reduction in the number of samples sent under the contract;
- suspension of sample shipment to the Contractor;
- an on-site laboratory evaluation;
- an HRGC/HRMS tape audit;
- a data package audit;
- a remedial laboratory evaluation sample; and/or
- contract sanctions, such as a Cure Notice.

13.0 QUALITY ASSURANCE AND DATA TREND ANALYSIS

13.1 Data Review

Data submitted by Contractors are subject to review from several aspects: compliance with contract-required Quality Control (QC); usability, and full data package evaluation. Problems resulting from any of these reviews may determine the need for a HRGC/HRMS tape audit, an on-site laboratory evaluation, and/or a remedial laboratory evaluation sample. In addition, QC prescribed in the methods provides information that is continually used by the USEPA to assess sample data quality, Contractor data quality, and Program data quality via data trend analysis. Trend analysis is accomplished by entering data into a computerized database. Statistical reports that evaluate specific anomalies or disclose trends in many areas, including the following, are generated from this database:

- Recovery standard recovery;
- Laboratory evaluation sample results;
- Blanks;
- HRGC/HRMS system performance checks (PFKs);
- Initial calibration and calibration verification data; and
- Other QC and method parameters.

13.2 Program-wide trend Analysis

Program-wide statistical results are used to rank Contractors to observe the relative performance of each Contractor using a given protocol against its peers. The reports are also used to identify trends within Contractors. The results of many of these trend analyses are included in the overall evaluation of a Contractor's performance, and are reviewed to determine if corrective action or an on-site laboratory evaluation may be required to ensure that the Contractor can meet the Quality Assurance/Quality Control (QA/QC) requirements of the contract. Contractor performance is monitored over time using these trend analysis techniques to detect departures of Contractor output from required or desired levels of quality control, and to provide an early warning of Contractor QA/QC problems which may not be apparent from the results of an individual Case.

13.3 Performance Based Monitoring

As a further benefit to the Program, the database provides the information needed to establish performance-based criteria in updated analytical protocols, where advisory criteria have been previously used. The vast empirical data set produced by Contractors is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of quality control and performance criteria specifications of what is routinely achievable and expected of environmental chemistry Contractors

engaged in mass production analysis of environmental samples. This, in turn, assists the USEPA in meeting its objectives of obtaining data of known and documented quality.

14.0 DATA MANAGEMENT

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; and
- 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 CLP PO.

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; and
- Data and system security, backup, and archiving.

EXHIBIT F

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

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

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1.0 INTRODUCTION

A sample is physical evidence collected from a facility or 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 (SDG) 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;
- Condition of the sample bottles;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;
- Presence or absence of Chain-of-Custody records;
- Sample tags listed/not listed on Chain-of-Custody records;
- Presence or absence of Traffic Reports or Packing Lists;
- Cooler temperature;
- Date of receipt;

- Time of receipt;
- EPA sample numbers;
- Presence or absence of sample tags;
- Sample tag numbers;
- Assigned laboratory numbers;
- 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., Chain-of-Custody records, Traffic Reports 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; and unsatisfactory sample condition (e.g., leaking sample container).

2.1.9 The Contractor shall record resolution of problems and discrepancies by Sample Management Office (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 USEPA 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, 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 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 pre-printed laboratory documents.

2.5.6 Each laboratory document entry shall be dated with the month/day/year (e.g., 01/01/2000) and signed (or initialed) 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 event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs).

2.5.14 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.

2.5.15 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).

2.5.16 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 that 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:

- C logbook pages;
- C bench sheets;
- C screening records;
- C preparation records;
- C repreparation records;
- C analytical records;
- C re-analysis records;
- C records of failed or attempted analysis;
- C custody records;
- C sample tracking records;
- C raw data summaries;
- C computer printouts;
- C correspondence;
- C FAX originals;
- C library search results, and
- C 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., Chain-of-Custody records, Traffic Reports, 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 packages were sent, the date, and the carrier used.

2.7.14 Shipments of deliverable packages, including re-submittals, 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;
- Chain-of-Custody records;
- Traffic Reports or Packing Lists;
- Airbills or airbill stickers, and
- Sample tags.

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 Chain-of-Custody records;

- C Cooler temperature;
- C Date of receipt;
- C Time of receipt;
- C EPA sample numbers;
- C Sample tag numbers;
- C Assigned laboratory numbers;
- C Samples delivered by hand, and
- C 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., Chain-of-Custody records, Traffic Reports 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 and unsatisfactory sample condition (e.g., leaking sample container).

3.1.2.8 The resolution of problems and discrepancies by 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:

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

- C Laboratory document entries are signed and dated with the month/day/year (e.g., 01/01/2000);
- C Entries on all laboratory documents are recorded in ink;
- C 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;
- C Unused portions of laboratory documents are lined-out;
- C Pages in bound and unbound logbooks are sequentially numbered;
- C Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
- C Logbook entries are recorded in chronological order;
- C Entries are recorded for only one SDG per page, except in the event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs);
- C 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);
- C Information inserted in laboratory documents is affixed permanently, signed, and dated across the insert, and
- C 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:

- C Contractor personnel responsible for original data entry are identified;
- C Changes to electronic data are made such that the original data entry is preserved, the editor is identified, and the revision date is recorded;
- C The accuracy of manually entered data, electronically entered data, and data acquired from instruments is verified;

- C Report documents produced by the electronic data collection system are routinely verified to ensure the accuracy of the information reported;
- C Electronic data collection system security is maintained;
- C Archives of electronic data and accompanying software are maintained in a secure location, and
- C 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.

- C Documents relating to the CSF are maintained in a secure location;
- C All original laboratory forms and copies of SDG-related logbook pages are included in the CSF;
- C Laboratory documents are photocopied in a manner to provide complete and legible replicates;
- C All documents relevant to each SDG are included in the CSF;
- C Sample tags are encased in clear plastic bags by the document control officer or a designated representative before being placed in the CSF;
- C The CSF is organized and assembled on an SDG-specific basis;
- C 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;
- C Each CSF is submitted with a completed Form DC-2, and re-submitted CSFs are submitted with a new or revised Form DC-2;
- C 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, and

- 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

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

ANALYSIS DATE/TIME - The date and military time of the injection of the sample, standard, or blank into the HRGC/HRMS.

CALIBRATION STANDARD (CS) - A solution prepared from a secondary standard and/or stock solutions, and used to calibrate the response of the instrument with respect to analyte concentration.

CALIBRATION VERIFICATION STANDARD - The mid-point calibration standard (CS3) that is used to verify the initial calibration of the system.

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. A Case consists of one or more Sample Delivery Groups.

CLEANUP STANDARD - A standard containing $^{37}Cl_4$-2,3,7,8-TCDD which is added to all extracts prior to cleanup. The purpose of this standard is to measure the efficiency of the cleanup process.

COLUMN PERFORMANCE SOLUTION (CPS) - When Window Defining Mixture and the Isomer specificity check solutions are combined, the solution is identified as CPS.

CONGENER - Individual compound belonging to a group or class of compounds with a similar general structure.

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.

DATE - MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 1999, 2000, 2001,2002, etc.

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

ESTIMATED DETECTION LIMIT (EDL) - The concentration of an analyte required to produce a signal with peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2,3,7,8-substituted isomer for which the response of the primary and secondary ions is less than 2.5 times the background level.

ESTIMATED MAXIMUM POSSIBLE CONCENTRATION (EMPC) - The EMPC is calculated for 2,3,7,8-substituted isomers for which the quantitation and/or confirmation ion(s) has signal to noise in excess of 2.5, but does not meet identification criteria.

EXTRACTABLE - A compound that can be partitioned into an organic solvent from the sample matrix, and is amenable to gas chromatography.

GEL PERMEATION CHROMATOGRAPHY (GPC) - A size-exclusion chromatographic technique that is used as a cleanup procedure for removing large organic

molecules, particularly naturally occurring macro-molecules such as lipids, polymers, viruses, etc.

HOMOLOGUE - A group of compounds that have the same molecular weight, but not necessarily the same structural arrangement.

HPLC - High Performance Liquid Chromatograph or High Performance Liquid Chromatography.

HRGC - High Resolution Gas Chromatograph or Gas Chromatography.

HRMS - High Resolution Mass Spectrometer or Mass Spectrometry.

IN-HOUSE - At the Contractor's facility.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations. It is used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INTEGRATION SCAN RANGE - Range from the scan number of the scan at the beginning of the area of integration, to the scan number at the end of the area of integration.

INTEGRATION TIME RANGE - The retention time at the beginning of the area of integration, to the retention time at the end of the area of integration.

INTERNAL STANDARD - $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD standards are added to every blank, quality control sample, and sample extract aliquot just prior to analysis.

ISOMER - Chemical compounds that have the same molecular formula, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are structural isomers.

LABELED COMPOUNDS - Isotopically labeled compounds in Exhibit D (see Table 1) that are added to every sample and are present at the same concentration in every blank, quality control sample, and calibration solution. The labeled compounds (internal standards) are added to the sample before extraction and are used to measure the concentrations of the analytes.

LABORATORY - Synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE/LABORATORY CONTROL SAMPLE DUPLICATE - Aliquots of a reference matrix fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure to determine the accuracy and precision of the method by measuring recovery and RPD.

m/z - Mass to charge ratio, synonymous with “m/e”.

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, soil, sediment, sludge, tissue (no human tissue), ash, oil, or oily matrices. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX EFFECT - In general, the effect of a particular matrix on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes. In addition, non-target analytes may be extracted from the matrix and cause interferences.

METHOD BLANK - An analytical control consisting of reference material, labeled compounds, internal standards, and cleanup standards, that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

NARRATIVE (SDG Narrative) - Portion of the data package which includes laboratory, contract, Case and 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 SOLIDS - An approximation of the dry solids content in a soil/sediment sample made by drying an aliquot of the sample for 12 hours at 110°C. The percent solids determined in this manner also excludes contributions from all compounds that may volatilize at or below 110°C. The percent solids is calculated as follows:

$$\% \text{ solids} = \frac{\text{weight of sample aliquot after drying}}{\text{weight of sample aliquot before drying}} \times 100\%$$

PERFLUOROKEROSENE (PFK) - The mixture of compounds used to calibrate the exact m/z scale in the HRMS.

PROTOCOL - Describes the exact procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Used synonymously with Statement of Work (SOW).

REAGENT WATER - Water demonstrated to be free from the analytes of interest and potentially interfering substances.

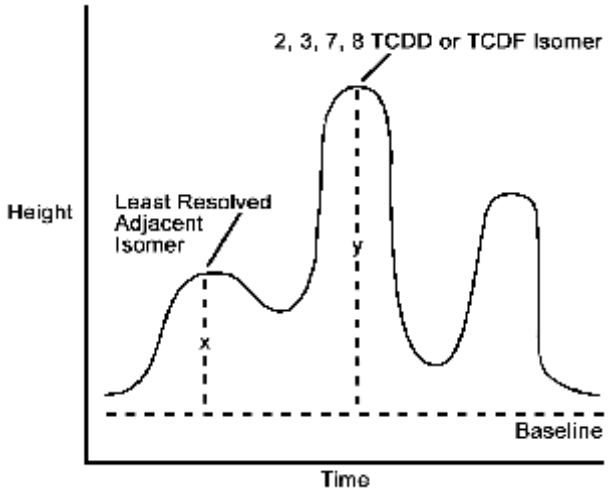
RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

RELATIVE RESPONSE FACTOR (RRF) - The ratio of the response of a given compound to its corresponding internal standard. Response factors are determined using the area responses of both the primary and secondary exact m/z's for each compound in each calibration standard.

RELATIVE RESPONSE (RR) - a measure of the relative mass spectral response of the native compound compared to its labeled compound analog. Relative responses are determined using the area responses of both the primary and secondary exact m/z's for each compound in each calibration standard.

RELATIVE STANDARD DEVIATION (%RSD) - The standard deviation times 100, divided by the mean. Also termed Coefficient of Variation.

RESOLUTION - Also termed separation or percent resolution, the separation between the least resolved adjacent isomer and the 2,3,7,8 TCDD or TCDF isomer, calculated by dividing the height of the 2,3,7,8- TCDD or TCDF isomer peak (y) by the height from the valley of the least resolved adjacent peak to the baseline (x), multiplied by 100.



RESPONSE or Instrumental Response - A measurement of the output of the HRGC/HRMS detector in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

RETENTION TIME (RT) - The time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

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. A Sample Delivery Group is defined by the following, whichever is most frequent:

- Each Case if field samples received, or
- Each 20 field samples (excluding Performance Evaluation (PE) samples) within a Case, or
- Each 7 calendar day period 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 (e.g., 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 Contract Laboratory Analytical Services Support (CLASS) contract, awarded and administered by USEPA.

SAMPLE NUMBER (EPA Sample Number) - A unique identification number designated by the USEPA to each sample. The EPA sample number appears on the sample Traffic Report which documents information on that sample.

SELECTED ION MONITORING (SIM) - A mode of MS operation in which specific m/e ratios are monitored, as opposed to scanning the entire mass range.

SIGNAL TO NOISE (S/N) RATIO - The ratio of analyte signal to random background signal. The noise is defined as the height of the largest signal (excluding signal due to CDDs/CDFs or other chemicals) within the 100 scan window. The signal is defined as the height of the CDD/CDF peak.

SOIL - Used herein synonymously with soil/sediment, sediment, and sludge.

SOLID-PHASE EXTRACTION (SPE) - An extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed Liquid-solid extraction.

SOXHLET/DEAN-STARK EXTRACTOR (SDS) - An extraction device applied to the extraction of solid and semi-solid materials.

STOCK SOLUTION - A solution containing an analyte that is prepared using a reference material traceable to the USEPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

TARGET COMPOUND LIST (TCL) - A list of compounds designated by the Statement of Work (See Exhibit C) for analysis.

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

TRAFFIC REPORT (TR) - An EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which documents sample condition and receipt by the laboratory.

TOXICITY EQUIVALENCE FACTOR (TEF) - A coefficient relating the risks from a particular compound to another compound.

TWELVE-HOUR TIME PERIOD - For dioxin/furan analyses performed by HRGC/HRMS, the 12-hour time period in the analytical sequence begins at the moment of injection of the window defining mixture that precedes sample analyses, and ends after 12 hours have elapsed according to the system clock.

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.

WINDOW DEFINING MIXTURE (WDM) - Prior to analyzing the calibration solutions, blanks, samples, and QC samples, the retention time window defining solution is analyzed to define the beginning retention times for the dioxin and furan isomers and evaluate descriptor switching times.

EXHIBIT H

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

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

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1.0 ELECTRONIC DELIVERABLE FOR CDD/CDF ANALYSIS

1.1 Requirements

The Contractor shall submit a diskette for CDD/CDF.

1.1.1 The CLP is currently developing a data delivery strategy that may be used as an alternate 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 CLP 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 CLP 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 CLP, all electronic data deliverables must be provided as specified in this Exhibit H.

1.1.2 The file shall be submitted on IBM-compatible, 3.5 inch, high density 1.44 M-byte diskettes. The diskettes shall be formatted and recorded using DOS/Windows Operating Systems. The diskettes shall include all information relevant to one, and only one, Sample Delivery Group (SDG). Multiple diskettes may be submitted. The data from a single production run may be split onto multiple diskettes, however, do not split the data from a sample analysis onto multiple diskettes. Each diskette shall be identified with an external label containing the following information: disk density; laboratory code; contract number; Case number; SDG number; Client number; initial submission or resubmission, as applicable, and date.

1.1.3 The deliverable consists of a tab delimited text file containing the data elements listed in Section 1.2. The file must contain the fields in the order specified in Section 1.2 and the field names exactly as specified in Section 1.2. For specific field contents, and requirements for significant figures and number of decimal places, refer to Exhibit B of this SOW. Information on the diskette must correspond to information submitted in the hardcopy raw data package and on the hardcopy raw data package forms. 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. Report results for field samples, PE samples, method blanks, and laboratory control samples/laboratory control sample duplicates.

1.2 Required Fields

Field Name	Data type	Format	Description
Labname	Character string	Max length 25	Ex. B
Labcode	Character string	Max length 6	Ex. B
Contract	Character string	Max length 11	Ex. B
Case Number	Character string	Max length 5	Ex. B
SDG Number	Character string	Max length 20	Ex. B
Client Number	Character string	Max length 11	Ex. B
GC Column Identifier	Character string	Max length 12	GC Column. Ex. B
GC Column Internal Diameter	Numeric	Max length 4	Ex. B (report in mm)
EPA Sample Number	Character string	Max length 20	The EPA sample number per Ex. B
Lab Sample ID	Character string	Max length 14	Ex. B
Lab File ID	Character string	Max length 14	Ex. B
Matrix	Character string	WATER, SOIL, TISSUE, OIL, ASH	The predominant material of which the sample is composed.
Date Received	Character string	YYYYMMDD	Lab Receipt Date. The date the sample was received at the laboratory.
Date Extracted	Character string	YYYYMMDD	The date sample extraction was begun.
Date Analyzed	Character string	YYYYMMDD	The date the sample was injected onto the HRGC/HRMS.
Time Analyzed	Character string	HHMM	The time the sample was injected onto the HRGC/HRMS.
Sample Weight/Volume	Numeric	Max length 6	Ex. D
Final Volume	Numeric	Max length 10	Concentrated extract volume.
Injection Volume	Numeric	Max length 10	Ex. B

Field Name	Data type	Format	Description
Percent Solids/Lipids	Numeric	Max length 7	Ex. B
Dilution Factor	Numeric	Max length 10	Ex. B
CAS Number	Character string	Ex. C	The analyte being determined in an analysis.
Concentration	Numeric	Max length 13	Enter the value as it appears on the appropriate Form.
Concentration Units	Character string	PG/L, NG/KG	Ex. B
Concentration Qualifier	Character string	Max length 5	Concentration qualifier from Form I-CDD/CDF.
EMPC	Numeric	Max length 13	Estimated Maximum Possible Concentration. Enter the value as it appears on the appropriate Form
Estimated Detection Limit	Numeric	Per Ex.B Form I-CDD/CDF	Ex. D
Amount Added	Numeric	Per Ex. B	For LCS, LCSD
Percent Recovery	Numeric	Per Ex. B	For LCS, LCSD

1.2.1 Except for the Date Analyzed, Date Extracted, and Date Received fields, all fields should contain data exactly identical to that reported on the data forms for the package.