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Superfund

USEPA CONTRACT LABORATORY PROGRAM

VOLATILE ORGANICS ANALYSIS OF AMBIENT AIR IN CANISTERS

VCAAO 1.0

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December, 1991

Revision VCAA01.0

VOLATILE ORGANICS ANALYSIS OF
AMBIENT AIR IN CANISTERS

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PREFACE

The purpose of this contract is to provide the U.S. Environmental Protection Agency (EPA) with chemical analytical services, quality control procedures, and an analysis structure which will generate data of known and documented quality. This document was developed with the guidance of the Air Toxics Workgroup to ensure that the needs of regional, state, and local air pollution programs are addressed.

The samples to be analyzed are of ambient air collected in canisters at or in the vicinity of known or suspected hazardous waste sites and may contain potentially hazardous organic in significant concentrations. The Contractor should be aware of the potential hazards associated with the handling and analyses of these samples. It is the Contractor's responsibility to take all necessary measures and precautions to ensure the health and safety of its employees. The Contractor is responsible for providing a safe working environment and making its employees aware of the potential hazards of working with and analyzing these samples.

Procedures specified herein shall be used in the preparation of canisters and analysis of air samples in canisters for the presence and quantitation of certain volatile organic compounds. The Contractor shall employ safe handling procedures and generally accepted laboratory practices in the performance of contract requirements and shall follow the quality assurance and quality control (QA/QC) program specified herein.

The data obtained under this contract will be used by EPA to determine the existence and extent of risk posed by hazardous waste disposal sites to the public, to individuals involved in Superfund site cleanups, and to the environment. The data may be used in civil and/or criminal litigation which requires the strictest adherence to chain-of-custody protocol, document control, and quality assurance procedures.

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR VOLATILE ORGANICS ANALYSIS OF AMBIENT AIR IN CANISTERS

December, 1991

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR
VOLATILE ORGANICS ANALYSIS OF AMBIENT AIR IN CANISTERS

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SECTION 1

GENERAL REQUIREMENTS

1.1 The Contractor shall employ procedures specified in this contract in the preparation and analysis of the ambient air samples for the presence and quantitation of the organic compounds listed in Exhibit C.

1.2 The Contractor shall use proven techniques to identify and measure the organic parameters presented in the Target Compound List (TCL) as specified in Exhibit C. The Contractor shall perform sample preparation and analysis procedures as prescribed in Exhibit D, and meet specified sample preservation and holding time requirements.

1.3 For all samples analyzed under this contract, the Contractor shall adhere to the QA/QC protocols specified in Exhibit E and abide by the evidentiary protocols specified in Exhibit F.

1.4 Following sample analysis, the Contractor shall perform data reduction and shall report analytical activities, sample data, and quality control documentation as designated in Exhibit B. Exhibit B contains all reporting and deliverables requirements for this contract, including copies of the data reporting forms and form instructions guide.

1.5 To ensure proper understanding of the language in this contract, Exhibit G contains a glossary of terms. When a term is used in the text without explanation, the glossary meaning shall be applicable. Glossary definitions do not replace or take precedence over specific information included in the document text.

1.6 The samples to be analyzed by the Contractor are from known or suspected hazardous waste sites and may contain hazardous organic and/or inorganic materials at high concentration levels. The Contractor should be aware of the potential hazards associated with the handling and analysis of these samples. It is the Contractor's responsibility to take all necessary measures to ensure the health and safety of its employees. It is also the Contractor's responsibility to follow appropriate disposal procedures according to state and federal regulations.

1.7 In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, as it may be used to make major decisions regarding public health and environmental welfare. In addition, data generated under this contract may be used in litigation against potentially responsible parties in the enforcement of Superfund legislation.

SECTION 2

SPECIFIC REQUIREMENTS

For each sample, the Contractor shall perform the following tasks:

2.1 TASK I: RECEIVE AMBIENT AIR SAMPLES IN CANISTERS

2.1.1 The Contractor shall receive and handle samples under the chain-of-custody and document control procedures described in Exhibit F.

2.1.2 The Contractor shall provide the required analytical expertise and instrumentation for analyses of the TCL analytes equal to or lower than the quantitation limits specified in Exhibit C. In Exhibit D, EPA provides the Contractor with an appropriate set of analytical procedures that shall be used.

2.1.3 The Contractor shall analyze samples within the maximum holding times specified in Exhibit D, even if these times are less than the maximum data submission time allowed in this contract.

2.1.4 The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the preparation and analysis with appropriate caution. The Contractor shall be responsible for all necessary measures and precautions to ensure the health and safety of laboratory employees.

2.2 TASK II: ANALYZE SAMPLES FOR THE IDENTIFICATION AND QUANTITATION OF SPECIFIC PARAMETERS

2.2.1 For each sample received, the Contractor shall be required to perform the analyses described in Exhibit D. The documentation that accompanies the sample(s) to the Contractor facility shall indicate specific analytical requirements for that sample or set of samples.

2.2.2 Exhibit D specifies the analytical procedures that shall be used. Exhibit D contains instructions and references for the analysis of ambient air samples containing low-to-medium concentrations of volatile organics for GC/MS analysis. GC/MS may use automated computer programs to facilitate the identification of organic compounds.

2.2.3 All samples must initially be run undiluted. When an analyte concentration exceeds the calibrated or linear range, appropriate dilution (but not below the contract required quantitation limit (CRQL)) and reanalysis of the sample is required, as specified in Exhibit D.

2.2.4 For the purpose of this contract, a full sample analysis is defined as analysis for all of the TCL constituents identified in Exhibit C in accordance with the methods in Exhibit D and performance of related QA/QC as specified in Exhibit D and Exhibit E. Laboratory Control Samples (LCS) analyses shall be considered a separate full sample analysis. All other QA/QC requirements are considered an inherent part of this contract and are included in the contract sample unit price.

2.2.5 The volatile compounds analyzed by GC/MS techniques and initially identified shall be verified by an analyst competent in the interpretation of mass spectra by comparison of the suspect mass spectra to the mass spectrum of a standard of the suspected compound. This procedure requires the use of multiple internal standards. Two criteria must be satisfied to verify the identifications:

2.2.5.1 Elution of the sample component at the same GC relative retention time as the standard component.

2.2.5.2 Correspondence of the sample component and standard component mass spectra.

2.2.6 For each sample analysis, the Contractor shall conduct mass spectral library searches of non-target compound sample components to determine tentative compound identifications as follows:

2.2.6.1 For each volatile organics analysis, the Contractor shall conduct a search to determine the possible identity of up to 10 organic compounds of greatest concentration which are not internal standards and not listed in Exhibit C.

2.2.6.2 In performing searches, the most recent release of the National Institute of Standards and Technology (NIST)/EPA/MSDC mass spectral library must be used.

NOTE: Substances with responses of less than 10 percent of the nearest internal standard are not required to be searched in this fashion.

2.2.6.3 Only after visual comparison of sample spectra with the spectra from the library searches will the mass spectral interpretation specialist assign a tentative identification. If the compound does not meet the identification criteria, it shall be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, they also should be included.

2.3 TASK III: PERFORM REQUIRED QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

2.3.1 All specific QA/QC procedures prescribed in Exhibits D and E shall be strictly adhered to by the Contractor. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibit B requirements.

2.3.2 The Contractor shall establish and use on a continuing basis QA/QC procedures including the daily or (as required) more frequent use of standard reference solutions from EPA, NIST, or secondary standards traceable thereto, where available at appropriate concentrations (i.e., standard solutions designed to ensure that operating parameters of equipment and procedures, from sample receipt through identification and quantitation, produce reliable data). Exhibits D and E provide specific QA/QC requirements.

2.3.3 Additional QA/QC shall be required quarterly or more frequently, i.e., with each Case or Sample Delivery Group (SDG), in the form of Laboratory Control Samples (LCS) and Performance Evaluation (PE) samples for volatile organics submitted to EPA for Contractor analysis, and in the form of verification of instrument parameters, as described in Exhibit E.

2.3.3.1 EPA has provided to the Contractor formats for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in this contract and within the time specified in the Contract Performance/Delivery Schedule.

2.3.3.2 Use of formats other than those designated by EPA will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government will be required.

2.3.3.3 Computer generated forms may be submitted in the hardcopy data package(s) provided that the forms are in exact EPA format. This means that the order of data elements is the same as on each EPA required form, including form numbers and titles, page numbers and header information, columns, and lines.

2.3.4 The Contractor shall provide analytical equipment and technical expertise for this contract as specified by the following:

2.3.4.1 Gas chromatograph/mass spectrometer (GC/MS) data system capable of meeting all the terms and conditions of the Contract with the following requirements:

2.3.4.1.1 The computer shall be interfaced by hardware to the mass spectrometer and be capable of acquiring continuous mass scans for the duration of the chromatographic program.

2.3.4.1.2 The computer shall be equipped with mass storage devices for saving all data from the GC/MS runs.

2.3.4.1.3 Computer software shall be available to allow searching GC/MS runs for specific ions and plotting the intensity of the ions with respect to time or scan number.

2.3.4.1.4 A computer data system must be interfaced to the MS that allows the continuous acquisition and storage, on machine-readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP) or Selected Ion Current Profile (SICP). Software must also be available that allows intergrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

2.3.4.1.5 The GC/MS shall be equipped with a GC to MS interface capable of extending a fused silica capillary column into the ion source. The column is to be 50 meters long by 0.25 to 0.53 mm I.D. 100% methyl silicone or 5% phenyl, 95% methyl silicone capillary column, or equivalent.

2.3.4.2 The Contractor shall use a magnetic tape storage device capable of recording data and suitable for long-term, off-line storage. The Contractor shall retain all raw GC/MS data acquired under this contract on magnetic tape in appropriate instrument manufacturer's format. The Contractor is required to retain the magnetic tapes with associated hardcopy tape logbook identifying tape contents (see Exhibit B) for 365 days after data submission. During that time, the Contractor shall submit tapes and logbook within seven days of request, as specified in the Contract Performance/Delivery Schedule.

2.3.4.3 The Contractor shall have a computerized MS library search system capable of providing a forward comparison, using the

standard spectra contained in the mass spectral library. The 1985 (or most recent) release of the NIST library (containing 42,261 spectra) must be used.

2.3.4.4 The system shall provide a numerical ranking of the standard spectra most closely corresponding to the sample spectra examined, and the data system shall have software capable of removing background signals from spectra.

2.3.4.5 The Contractor shall have, in-house and operable, a device capable of analyzing volatile organics as described in Exhibit D.

2.3.4.6 The Contractor shall have, in-house, the appropriate standards for all target compounds listed in Exhibit C prior to accepting any samples from the Sample Management Office (SMO). Standards provided by EPA for use in the Preaward Performance Evaluation may not contain all the target compounds and thus shall not be used for routine analyses unless or until they have been supplemented with commercially-available standard materials.

2.3.5 The minimum functional requirements necessary to meet the terms and conditions of this contract are listed below. The Contractor shall designate and use qualified key personnel to perform these functions. The EPA reserves the right to review personnel qualifications and experience.

2.3.5.1 Project Manager

2.3.5.2 GC/MS Laboratory Supervisor

2.3.5.3 Quality Assurance Officer

2.3.5.4 Systems Manager

2.3.5.5 Programmer Analyst

2.3.5.6 GC/MS Operators

2.3.5.7 Mass Spectral Interpreter

2.3.5.8 Chemist (back-up)

NOTE: The Contractor shall designate a Sample Custodian and a Document Control Officer.

2.3.6 The Contractor shall respond within 10 days to requests from data recipients for additional information or explanations that result from the Government's inspection activities.

2.3.7 The Contractor is required to retain unused sample volumes and used sample containers for a period of 60 days after data submission unless otherwise instructed in Exhibit B or Exhibit D.

2.3.8 The Contractor shall adhere to the chain-of-custody and document control procedures described in Exhibit F. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported in the Complete Case File Purge (Exhibit B).

2.3.9 Sample shipments to the Contractor's facility will be scheduled and coordinated by SMO, acting on behalf of the Administrative Project Officer (APO). The Contractor shall communicate with SMO personnel by telephone as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

2.3.10 If there are problems with the samples (e.g., mixed media, containers broken) or sample documentation/paperwork (e.g., Traffic Reports not with shipment, or sample and Traffic Report numbers do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

2.3.11 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case. A Case consists of one or more SDG(s). An SDG is defined by the following:

2.3.11.1 Each Case of field samples received; or

2.3.11.2 Each 20 field samples within a Case; or

2.3.11.3 Each seven 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).

2.3.12 Data for all samples in an SDG must be submitted together (in one package) in the order specified in Exhibit B. The SDG number is the EPA number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number is the lowest sample number (considering both alpha and numeric

designations) in the first group of samples received under the SDG. The SDG number is reported on all data reporting forms. The SDG Receipt Date is the day that the last sample in the SDG is received.

2.3.13 The Contractor is responsible for identifying each SDG as samples are received, through proper sample documentation (see Exhibit B) and communication with SMO personnel.

2.3.14 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report (TR) form bearing the sample number and descriptive information regarding the sample. The Contractor shall complete and sign the TR, recording the date of sample receipt and sample condition on receipt for each sample container.

2.3.15 The Contractor shall submit signed copies of TRs for all samples in an SDG to SMO within three calendar days following receipt of the last sample in the SDG. TRs shall be submitted in SDG sets (i.e., all TRs for a SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

2.3.16 EPA Case numbers (including SDG numbers) and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.

2.3.17 Samples will be routinely shipped directly to the Contractor through a delivery service. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays and holidays. As necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station, or other carrier service within the Contractor's geographical area.

2.3.18 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

SECTION 3

DETAILED TECHNICAL & MANAGEMENT REQUIREMENTS

The Contractor shall have the following technical and management capabilities:

3.1 PERSONNEL

3.1.1 Project Manager

3.1.1.1 Responsible for all technical efforts of the laboratory to meet all terms and conditions of the contract.

3.1.1.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.1.3 Experience: Minimum of three years of laboratory experience, including at least one year in a supervisory position.

3.1.2 GC/MS Laboratory Supervisor

3.1.2.1 Responsible for all technical efforts of the GC/MS laboratory to meet all terms and conditions of the contract.

3.1.2.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.2.3 Experience: Minimum of three years of laboratory experience in operating a GC/MS, including at least one year in a supervisory position.

3.1.3 Quality Assurance Officer

3.1.3.1 Responsible for overseeing the quality assurance aspects of data generation and reporting directly to upper management.

3.1.3.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.3.3 Experience: Minimum of three years of laboratory experience, including at least one year of applied experience with QA principles and practices in an analytical laboratory.

3.1.4 Systems Manager

3.1.4.1 Responsible for the management and quality control of

all computing systems (hardware, software, documentation, and procedures), generating, updating, and performing quality control on automated deliverables.

3.1.4.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, database management systems, or systems requirements analysis.

3.1.4.3 Experience: Minimum of three years experience in data or systems management or programming including one year experience with software used for data management and generation of deliverables.

3.1.5 **Program Analyst**

3.1.5.1 Responsible for the installation, operation, and maintenance of software and programs; generating, updating, and performing quality control procedures on analytical databases and automated deliverables.

3.1.5.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, information systems, or systems requirements analysis.

3.1.5.3 Experience: Minimum of two years experience in systems or applications programming including one year of experience with software used for data management and generation of deliverables.

3.1.6 **Gas Chromatography/Mass Spectrometer (GC/MS) Operator**

3.1.6.1 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.6.2 Experience: Minimum of one year of experience in operating and maintaining GC/MS instruments in conjunction with the education requirement; or in lieu of education requirement, three additional years of experience in operating and maintaining GC/MS instrumentation.

3.1.7 **Mass Spectral Interpreter**

3.1.7.1 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline with specialized training in GC/MS.

3.1.7.2 Experience: Minimum of two years of applied experience with GC/MS analysis of environmental samples.

3.1.8 Technical Staff Redundancy

3.1.8.1 In order to ensure continuous operations to accomplish the required work as specified by the contract, the bidder shall have a minimum of one chemist available at all times as a back-up technical person with the following qualifications.

3.1.8.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.8.3 Experience: Minimum of one year of experience in each of the following areas: GC/MS operation and maintenance.

3.2 FACILITIES

The adequacy of the facilities and equipment is as important as the technical staff for accomplishing the required work as specified by the EPA contract.

3.2.1 Sample Receipt Area

Adequate, contamination-free, well-ventilated work space with chemical resistant bench top shall be available for receipt and safe handling of EPA samples.

3.2.2 Storage Area

Sufficient refrigerator space to maintain unused EPA sample volume for up to 60 days after data submission shall be provided. Volatile samples must be stored in a refrigerator used only for storage of volatile samples from this contract. Samples must be stored in an atmosphere demonstrated to be free from all potential contaminants. Samples and standards must be stored separately.

3.2.3 Sample/Standard Preparation Area

Adequate, contamination-free, well-ventilated work space shall be provided with:

3.2.3.1 Benches with chemical resistant tops;

3.2.3.2 Exhaust hoods;

3.2.3.3 Glove box or isolated area in which to prepare standard materials;

3.2.3.4 Source of distilled or demineralized organic-free water; and

3.2.3.5 Analytical balance(s) located away from draft and rapid change in temperature.

3.3 INSTRUMENTATION

At a minimum, the Contractor shall have the following instruments operative at the time of the Preaward Site Evaluation and committed for the full duration of the contract.

3.3.1 100 Samples/Month Capacity Requirements

No. of Instrument(s)	Type of Instrument
1	GC/MS

NOTE: The Contractor shall have 1 complete GC/MS system available (operational) at all times as a back-up system. These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.2 200 Samples/Month Capacity Requirements

No. of Instrument(s)	Type of Instrument
2	GC/MS

NOTE: These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.3 Instrument Specifications

Further information on instrument specifications and required ancillary equipment may be found in Exhibit D and other Exhibits in this contract.

3.4 DATA HANDLING AND PACKAGING

The Contractor shall be able to submit reports and data packages as specified in Exhibit B. To complete this task, the Contractor shall be required to:

3.4.1 Provide space, tables, and copy machines to meet the contract requirements; and

3.4.2 Designate personnel responsible for report preparations and submission.

3.5 LABORATORY MANAGEMENT CAPABILITY

The Contractor shall have an organization with well-defined responsibilities for each individual in the management system to ensure sufficient resources for EPA contract(s) and to maintain a successful operation. To establish this capability, the Contractor shall designate personnel to carry out the following responsibilities for the EPA contract. Functions include, but are not limited to, the following:

3.5.1 Technical Staff

Responsible for all technical efforts for the EPA contract such as sample analysis, sample validation, and trouble-shooting of all instruments.

3.5.2 Project Manager

Responsible for overall aspects of EPA contract(s) (from sample receipt through data delivery) and shall be the primary contact for EPA Headquarters APO and Regional Technical Project Officers (TPO).

3.5.3 Sample Custodian

Responsible for receiving the EPA samples (logging, handling, and storage).

3.5.4 Quality Assurance Officer

Responsible for overseeing the quality assurance aspects of the data and reporting directly to upper management.

3.5.5 Document Control Officer

Responsible for ensuring that all documents generated are placed in the Complete SDG File for inventory and are delivered to the appropriate EPA Region or other receiver as designated by EPA.

EXHIBIT B

REPORTING

AND

DELIVERABLES REQUIREMENTS

December, 1991

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

PAGE NO.

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SECTION 1

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 The following table summarizes the contract reporting and deliverables requirements specified in the Contract Schedule and includes the distribution of each deliverable. **NOTE**: Specific recipient names and addresses are subject to change during the term of the contract. The EPA APO or SMO will notify the Contractor in writing of such changes when they occur.

Item	No. of Copies	Schedule and Delivery	Distribution		
			(1)	(2)	(3)
Updated Standard Operating Procedures (SOPs)	2	45 days after contract award.		X	X
*Sample Traffic Reports	1	***3 days after receipt of last sample in Sample Delivery Group (SDG).	X		
**Sample Data Summary Package	1	14 days after receipt of last sample in SDG.	X		
**Sample Data Package including the Performance Evaluation (PE) Sample	3	35 days after receipt of last sample in SDG	X	X	X
Results of Intercomparison Study/Preaward Performance Evaluation (PPE) Sample	2	35 days after receipt of last sample in SDG	X	X	
Complete SDG File	1	35 days after data receipt of last sample in SDG.		X	
GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO.	As Directed		
****Quality Assurance Plan	1	Submit copy within 7 days by written request by APO.	As Directed		

Distribution

(1) Sample Management Office
(2) Environmental Monitoring Systems Laboratory-Las Vegas
(3) USEPA Region

* Also required in each Sample Data Package.

** Concurrent delivery of these items to all recipients is required.

*** An SDG is a group of samples within a Case, received over a period of seven days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. (See Exhibit A, Task III, for further description).

**** See Exhibit E for description.

NOTE: As specified in the Contract Schedule in the IFB (Government Furnished Supplies and Materials), unless otherwise instructed by SMO, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of analytical data.

Address

(1) USEPA Contract Laboratory Program
Sample Management Office
P.O. Box 818
Alexandria, VA 22313

For overnight delivery service, use street address:

300 North Lee Street
Alexandria, VA 22313

(2) USEPA Environmental Monitoring Systems Laboratory
P.O. Box 93478
Las Vegas, NV 89193-3478
ATTN: Data Audit Staff

For overnight delivery service, use street address:

944 E. Harmon, Executive Center
Las Vegas, NV 89109
ATTN: Data Audit Staff

(3) USEPA REGIONS:

SMO, acting on behalf of the EPA APO, will provide the Contractor with the list of addresses for the 10 EPA Regions. SMO will provide the Contractor with updated Regional name/address lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The APO will notify the Contractor in writing of such changes when they occur.

SECTION 2

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

2.1 The Contractor shall provide reports and other deliverables according to the schedule specified in Section F of the IFB, "SCHEDULE INFORMATION." The required content and form of each deliverable is described in this Exhibit.

2.1.1 All reports and documentation shall be:

2.1.1.1 Legible;

2.1.1.2 Clearly labeled and completed in accordance with instructions in this Exhibit;

2.1.1.3 Arranged in the order specified in this section;

2.1.1.4 Paginated; and

2.1.1.5 Single-sided.

2.1.2 If submitted documentation does not conform to the above criteria, the Contractor will be required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the Government.

2.1.3 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation or through an APO/TPO action, the data shall be clearly marked as "ADDITIONAL DATA" and shall be sent to all three contractual data recipients (SMO, EMSL-LV, and Region). A cover letter shall be included that describes which data are being delivered, to which EPA Case(s) the data pertain, and who requested the data.

2.1.4 Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with the required documentation and contains the required data forms in EPA-specified format.

2.1.5 Descriptions of the requirements for each deliverable item cited in the Contract Performance/Delivery Schedule (see Section F of the IFB "SCHEDULE INFORMATION") are specified in this Section. Items submitted concurrently must be arranged in the order listed. Additionally, the components of each item must be arranged in the order presented herein.

2.2 UPDATED STANDARD OPERATING PROCEDURES

2.2.1 The Contractor shall submit updated copies of all required Standard Operating Procedures (SOPs) that were submitted with the Prebid Performance

Evaluation (PPE) sample results. The updated SOPs must address any and all issues of laboratory performance and operation identified by the Contractor in the review of the PPE sample data and the evaluation of Bidder-Supplied Documentation.

2.2.2 The Contractor must supply SOPs for the following.

2.2.2.1 Evidentiary SOPs.

2.2.2.2 Sample receipt and logging.

2.2.2.3 Sample and extract storage area.

2.2.2.4 Preventing sample contamination.

2.2.2.5 Security for laboratory and samples.

2.2.2.6 Traceability/equivalency of standards.

2.2.2.7 Maintaining instrument records and bound logbooks.

2.2.2.8 Glassware cleaning.

2.2.2.9 Technical and managerial review of laboratory operation and data package preparation.

2.2.2.10 Internal review of contractually-required QA/QC data for each individual data package.

2.2.2.11 Sample analysis, data handling, and data reporting.

2.2.2.12 Chain-of-custody and document control, including case file preparation.

2.2.2.13 Sample data validation/self-inspection system, including:

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to ensure that hardcopy data are complete and compliant with the requirements in Exhibit B;
- Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE

samples, etc.);

- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

2.2.2.14 Data Handling.

2.2.2.14.1 Data Management procedures are defined as written procedures that are clearly defined for all databases and files used to generate or re-submit deliverables specifying the acquisition or entry, update, correction, deletion, storage, and security of computer-readable data and files. Key areas of concern include: system organization including personnel and security, demonstration, operations, traceability, and quality control.

2.2.2.14.2 Data manually entered from hardcopy must be subjected to quality control procedures and error rates estimated.

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

- Justification or rationale for the change;
- Initials of the person making the changes or changes. Data changes must be identified when generating the deliverables;
- Changed documentation must be retained according to the schedule of the original deliverable;
- Resubmitted deliverables must be reinspected as a part of the laboratory's internal inspection process prior to submission. The entire deliverable and not just the changes must be reinspected;
- The laboratory manager must approve changes to originally submitted deliverables; and
- Documentation of data changes may be requested by laboratory

auditors.

2.2.2.14.4 Life cycle management procedures must be applied to computer systems used to generate and edit contract deliverables. Such systems must be thoroughly tested and documented prior to utilization.

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

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

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

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

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

- System operation and maintenance including documentation and training; and
- Database integrity including data entry, data updating and QC.

2.2.2.14.10 Data and system security, backup, and archiving.

2.3 SAMPLE TRAFFIC REPORTS

2.3.1 The original sample TR page marked "Lab Copy for Return to SMO" shall be submitted to SMO with laboratory receipt information and signed in original Contractor signature, for each sample in the SDG.

2.3.2 TRs shall be submitted in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

2.3.3 The SDG Cover Sheet shall contain the following items:

2.3.3.1 Laboratory name.

2.3.3.2 Contract number.

2.3.3.3 Sample analysis price - full sample price from contract.

2.3.3.4 Case number.

2.3.3.5 List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations), and the "last" sample received would be the highest sample number (considering both alpha and numeric designations).

2.3.4 Each TR shall be clearly marked with the SDG Number and the EPA sample number of the first sample in the SDG. This information shall be entered below the laboratory receipt date on the TR. The TR for the last sample received in the SDG shall be clearly marked "SDG - FINAL SAMPLE."

2.3.5 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the laboratory shall make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.

2.4 SAMPLE DATA SUMMARY PACKAGE

2.4.1 As specified in the Delivery Schedule, one Sample Data Summary Package shall be delivered to SMO concurrently with delivery of other required sample data. The Sample Data Summary Package shall be submitted separately (i.e., separated by rubber bands, clips or other means) directly preceding the Sample Data Package.

2.4.2 The Sample Data Summary Package shall contain data for samples in one SDG of the Case, as follows:

2.4.2.1 Cover Page.

2.4.2.2 By sample, tabulated target compound results (FORM I-AAVC) and tentatively identified compounds (FORM I-AAVC-TIC).

2.4.2.3 Laboratory Control Sample results (FORM III-AAVC).

2.4.2.4 Blank summary (FORM II-AAVC) and tabulated results (FORM I) including tentatively identified compounds (FORM I-AAVC-TIC).

2.4.2.5 Internal standard area and retention time data (FORM VII-AAVC).

2.5 SAMPLE DATA PACKAGE

2.5.1 The sample data package shall be complete, consecutively paginated, and shall include data for analysis of all samples in an SDG such as field samples, blanks, and laboratory control samples.

2.5.2 The sample data package is divided into five units as follows:

2.5.2.1 Cover page.

2.5.2.1.1 This document shall be clearly labeled "Cover Page." The Cover Page shall contain: laboratory name; laboratory code; contract number; Case Number; SDG Number; SOW number (appears on cover page of SOW); EPA sample numbers in alphanumeric order, showing EPA sample number cross-referenced with laboratory ID numbers; and comments, describing in detail any problems encountered in processing the samples in the data package.

2.5.2.1.2 The Cover Page shall contain the following statement, verbatim:

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature."

2.5.2.1.3 This statement shall be directly followed by the signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

2.5.2.1.4 In the event that the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

2.5.2.2 Sample data (Results).

2.5.2.2.1 Sample data shall be arranged in packets with the Analysis Data Sheet (FORM I-AAVC, including FORM I AAVC-TIC), followed by the raw data for volatile samples. These sample packets should then be placed in increasing EPA sample number

order, considering both letters and numbers.

NOTE: FORM I AAVC-TIC is the tabulated list of the highest probable match for up to 10 organic compounds that are not surrogates and internal standards and are not listed in Exhibit C (TCL). It includes the Chemical Abstracts Service (CAS) Registry Number, tentative identification, and estimated concentration.

2.5.2.2.1.1 Reconstructed total ion chromatograms (RIC) for each sample or sample extract.

2.5.2.2.1.2 RICs must be normalized to the largest nonsolvent component, and must contain the following header information:

- EPA sample number;
- Date and time of analysis;
- GC/MS instrument ID; and
- Laboratory file ID.

2.5.2.2.1.3 Internal standards are to be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak.

2.5.2.2.1.4 Quantitation Report: The complete data system report must be included in all sample data packages, in addition to the reconstructed ion chromatogram for preliminary identification and/or quantitation using either the automated or manual data system procedures. The complete data system report shall include all of the information listed below:

- EPA sample number;
- Date and time of analysis;
- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- GC/MS instrument ID; and
- Laboratory file ID.

2.5.2.2.1.5 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report.

2.5.2.2.1.6 Target Compound Mass Spectra: For each sample, by each compound identified, copies of the raw spectra and copies of background subtracted mass spectra of target compounds listed in Exhibit C that are identified in the sample and corresponding background subtracted target compound standard mass spectra shall be included in the data package. Spectra must be labeled with EPA sample number, laboratory file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.

2.5.2.2.1.7 Tentatively Identified Compound Mass Spectra and Library Matches: For each sample, by each compound identified, copies of mass spectra of organic compounds not listed in Exhibit C, Tentatively Identified Compounds, with associated best-match spectra (three best matches), labeled as above shall be included in the data package.

2.5.2.2.2 Volatile Standard Data:

2.5.2.2.2.1 Initial Calibration: All initial calibration data must be included for all analyses associated with the SDG. When more than one initial calibration is performed, the reconstructed ion chromatograms and quantitation reports and each type of form must be put in chronological order, by instrument as follows:

- Initial Calibration Data Sheet (FORM V-AAVC);
- Internal Standard Area and Retention Time Summary (FORM VII-AAVC); and
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimiles) for the initial (five point) calibration are labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.2.2.2 Continuing Calibration: When more than one continuing calibration is performed, the reconstructed ion chromatograms and quantitation reports and each type of form must be put in chronological order, by instrument if more than one instrument is used as follows:

- Continuing Calibration Data Sheet (FORM VI-AAVC);
- Internal Standard Area and Retention Time Summary (FORM VII-AAVC); and
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimiles) for the initial (five point) calibration are labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.3 Quality control summary.

2.5.2.3.1 The quality control summary shall contain the following forms:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order by date of analysis or instrument.

- Blank Summary (FORM Ii-AAVC);
- GC/MS Instrument Performance Check (FORM IV-AAVC); and
- Internal Standard Area and RT Summary (FORM VII-AAVC).

2.5.2.3.2 The quality control summary shall also contain the following:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order by date of analysis or instrument.

- GC/MS Tuning Data
  - GC/MS Tuning - BFB data, for each 12-hour period, shall be arranged in chronological order by instrument for each GC/MS system utilized;
  - GC/MS Tuning and Mass Calibration - BFB (FORM IV-AAVC);
  - Bar graph spectrum, labeled as in 2.5.2.2.1.2 and 2.5.2.2.1.4; and
  - Mass listing, labeled as in 2.5.2.2.1.2. and 2.5.2.2.1.4.
- Blank data shall be arranged in chronological order by instrument. The blank data shall be arranged in packets with both of the Organic Analysis Data Sheets (FORM I VOA and FORM I VOA-TIC), followed by the raw data for volatile samples.

- Laboratory Control Sample Data

  - Laboratory Control Sample Data Sheet (FORM III-AAVC); and

  - Reconstructed ion chromatograms and quantitation reports or legible facsimile (GC/MS), labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.4 Raw data.

2.5.2.4.1 For each reported value, the Contractor shall include all raw data from the instrument used to obtain the sample values (except for raw data for quarterly verifications of instrument parameters). Raw data shall contain all instrument readouts used for the sample results, including those readouts that may fall below the method quantitation limit. All GC/MS instruments must provide legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included.

2.5.2.4.2 All raw data shall include concentration units for GC/MS.

2.5.2.4.3 Organic raw data must be labeled with EPA sample number and appropriate codes as shown in Tables B-1 and B-2 respectively, to identify unequivocally the following:

- Initial and continuing calibration standards;

- Blanks;

- Diluted and undiluted samples (by EPA sample number) and all values used to obtain the reported values. If the dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient;

- Duplicates;

- Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation;

- Data and EPA sample number for GC/MS analyses clearly and sequentially identified on the raw data;

- All calculations for sample data, including percent recovery,

coefficient of variation, slope and y-intercept of linear fit; and

- Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide time of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as interference check samples and linear range analysis standards.

2.5.2.5 Preparation logs.

These logs must include the following:

- Date;
- Standard weights and/or volumes;
- Sample canister pressures;
- Sufficient information to identify unequivocally which QC samples (e.g., laboratory control sample, blank) correspond to each batch prepared; and
- Comments describing any significant sample changes or reactions which occur during preparation.

2.5.2.6 Sample TRs.

A legible copy of the sample TRs and SDG Cover Sheet shall be submitted as described in part 2.3 of this Exhibit for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample number order, considering both alpha and numeric designations.

2.6 RESULTS OF INTERCOMPARISON/PERFORMANCE EVALUATION SAMPLE ANALYSES

The reporting of analytical results for Intercomparison Study/Preaward Performance Evaluation (PPE) sample analyses includes all requirements specified in part 2.4 for reporting of sample data. The PPE sample shall be carried through the exact same process as an analytical and field samples.

2.7 COMPLETE CASE FILE PURGE

2.7.1 The Complete SDG File package includes all laboratory records

received or generated for a specific Case that have not been previously submitted to EPA as a deliverable. These items shall be submitted to EPA as a deliverable. These items shall be submitted along with their Document Inventory Sheet FORM AADC-2 (see Exhibit E for description of document numbering and inventory procedure). These items include, but are not limited to, sample tags, custody records, sample tracking records, analysts' logbook pages, bench sheets, instrument readout records, computer printouts, raw data summaries, instrument logbook pages (including instrument conditions), correspondence, and the document inventory.

2.7.2 Shipment of the Complete SDG File package by first class mail, overnight courier, priority mail, or equivalent, is acceptable. Custody seals, which are provided by EPA, shall be placed on shipping containers and a document inventory and transmittal letter included. The Contractor is not required to maintain any documents for a sample Case after submission of the Complete SDG File package; however, the Contractor should maintain a copy of the document inventory and transmittal letter.

2.8 GC/MS TAPES

2.8.1 The Contractor must store all raw and processed GC/MS data on magnetic tape, in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, laboratory control samples, initial calibrations, continuing calibrations, and BFB, as well as all laboratory generated spectral libraries and quantitation reports required to generate the data package. The Contractor shall maintain a written reference logbook of tape files to EPA sample number, calibration data, standards, blanks, and laboratory control samples. The logbook should include EPA sample numbers and standard and blank IDs, identified by Case and SDG.

2.8.2 The Contractor is required to retain the GC/MS tapes for 365 days after data submission. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the APO.

2.9 QUALITY ASSURANCE PLAN (QAP)

2.9.1 The Contractor shall prepare a written Quality Assurance Plan (QAP) which describes the procedures that are implemented to achieve the following: maintain data integrity, validity, and useability; ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility; detect problems through data assessment and established corrective action procedures which keep the analytical process reliable; and document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

2.9.2 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, SOPs pertaining to each parameter shall be included or referenced as part of the QAP. The QAP must be available during on-site laboratory evaluation and upon written request by the APO.

Table B-1
Codes for Labeling Organic Data

Sample	XXXXX
Reanalyzed Sample	XXXXXRE
Sample Analyzed at a Dilution	XXXXXDL
Laboratory Control Sample Number	VCLCS##
Laboratory Method Blank	VCBLK##
Standards	VCSTD###

SECTION 3

FORM INSTRUCTIONS GUIDE/DATA REPORTING FORMS

3.1 Form Instructions Guide

3.1.1 This section includes specific instructions for the completion of all required forms for volatile organics analysis utilizing canister. Each of the forms is specific to a given function. These instructions are arranged in the following order:

3.1.1.1 General Information and Header Information

3.1.1.2 Cover Page [COVER PAGE - AAVC]

3.1.1.3 Analysis Data Sheet [FORM I - AAVC]

3.1.1.4 Tentatively Identified Compounds [FORM I - AAVC-TIC]

3.1.1.5 Blank Summary [FORM II - AAVC]

3.1.1.6 Laboratory Control Sample Data Sheet [FORM III - AAVC]

3.1.1.7 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AAVC]

3.1.1.8 Initial Calibration Data Sheet [FORM V - AAVC]

3.1.1.9 Continuing Calibration Data Sheet [FORM VI - AAVC]

3.1.1.10 Internal Standard Area and Retention Times Summary [FORM VII - AAVC]

3.1.1.11 Canister Certification [FORM VIII - AAVC]

3.1.1.12 Analytical Sequence [FORM IX - AAVC]

3.1.1.13 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.1.1.14 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

3.1.2 General Information and Header Information

3.1.2.1 Values must be reported on the hardcopy forms according to the individual form instructions in this Section. For example, results for concentrations of volatile organic target compounds must be reported to three significant figures if the value is greater than

or equal to 10, and to two significant figures for values less than 10.

3.1.2.2 All characters which appear on the data reporting forms presented in the contract must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the EPA APO. The names of the various fields and compounds (i.e., "Lab Code," "Chloromethane") must appear as they do on the forms in the contract.

3.1.2.3 Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low").

3.1.2.4 Six (6) pieces of information are common to the header sections of each data reporting form. They are Lab Name, Lab Code, Contract No., Case No., SDG No., and SAS No. These pieces of information must be entered on every form and must match on every form.

3.1.2.4.1 The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

3.1.2.4.2 The "Lab Code" is an alphabetical abbreviation of up to 6 letters, assigned by the EPA, to identify the laboratory and aid in data processing. This code shall be assigned by the EPA at the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of the EPA. If a change of name or ownership occurs at the laboratory, the Lab Code will remain the same until the contractor is directed by the EPA to use another Lab Code assigned by the EPA.

3.1.2.4.3 The "Contract No." is the number of the EPA contract under which the analyses were performed. In the case of multiple laboratories operating under a corporate-wide contract, the contract number entered shall be that of the corporate contract, regardless of the facility performing the analyses (see Lab Code).

3.1.2.4.4 The "Case No." is the EPA-assigned case number associated with the sample, and reported on the Traffic Report.

3.1.2.4.5 The "SDG No." is the Sample Delivery Group (SDG) number. The SDG No. is the EPA Sample Number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

3.1.2.4.6 The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services (SAS). If samples are to be analyzed under SAS only and reported on these forms, then enter SAS No. and leave Case No. blank. If samples are analyzed according to the "Routine Analytical Services" (IFB) protocols and have additional SAS requirements, list both Case No. and SAS No. on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. Note that some samples in an SDG may have a SAS No. while others do not.

3.1.2.5 The "EPA Sample No." is the other information common to most of the forms. This number appears either in the upper right corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper right corner, it should be entered on the middle line of the three lines that comprise the box.

3.1.2.5.1 All samples, spikes, blanks, and standards shall be identified with an EPA Sample Number. For field samples, the EPA Sample Number is the unique identifying number given in the Traffic Report that accompanied that sample.

3.1.2.5.2 In order to facilitate data assessment, the following sample suffixes must be used:

XXXXX	-	EPA sample number
XXXXXRE	-	Reanalyzed sample
XXXXXDL	-	Sample analyzed at a secondary dilution

3.1.2.5.3 VOC standards prepared in canisters shall be identified as VCSTD###, where ### is the concentration in ppbv of volatile standards in canister (i.e., 002, 005, 010, 020, and 050).

3.1.2.5.4 As for the blank identifiers, these designations will have to be combined with other information to uniquely identify each standard. Blanks shall be identified as VCBLK##. The "EPA Sample No." must be unique for each blank analysis within an SDG. The laboratory must achieve this by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for volatiles-canister blanks would be VCBLK01, VCBLK02, etc.

3.1.2.5.5 LCSs shall be identified as VCLCS##. The "EPA Sample No." must be unique for each LCS analysis within an SDG. The laboratory must achieve this by replacing the two-character "##"

terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for volatiles-canister LCSs would be VCLCS01, VCLCS02, etc.

3.1.2.6 Several other pieces of information are common to many of the Data Reporting Forms. These include Lab Sample ID, Lab File ID, Date Received, etc. Following is a brief description of each of these entries.

3.1.2.6.1 "Lab Sample ID" is an optional laboratory-generated internal identifier. Up to 12 alpha-numeric characters may be reported here. If the contractor does not have a Lab Sample ID, this field may be left blank.

3.1.2.6.2 "Lab File ID" is the laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. Up to 14 alpha-numeric characters may be used here.

3.1.2.6.3 "Date Received" is the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the VTSR). It should be entered as MM/DD/YY.

3.1.2.6.4 "Date Analyzed" should be entered in a similar fashion. The date of sample receipt will be compared with the analysis dates to ensure that contract holding times were not exceeded.

3.1.2.6.5 "Instrument ID" is common to many of the forms, particularly those containing calibration data. The identifier used by the laboratory must include some indication of the manufacturer and/or model of the instrument, and contain additional characters that differentiate between all instrument of the same type in the laboratory.

3.1.2.6.6 "GC Column ID" or "Column ID" is common to various other forms. This field is used to identify the GC column.

3.1.2.7 For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is odd, and round down if that digit is even.

3.1.2.8 All results must be transcribed to the forms in the raw data with the specified number of decimal places that are described in

Exhibit B. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros must be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry
5.9	6.3	5.900
5.99653	6.3	5.997
95.99653	6.3	95.997
995.99653	6.3	995.997
9995.996	6.3	9996.00
99995.9	6.3	99995.9
999995.9	6.3	invalid

NOTE: 6.3 stands for a maximum of six significant figures and up to three decimal places.

3.1.3 **Cover Page [COVER PAGE - ~~AAVC~~]**

3.1.3.1 This form is used to list all billable samples analyzed within an SDG, and to provide certain analytical information and general comments. It is also the document which is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

3.1.3.2 Under the "EPA Sample No." column, enter up to 7 characters for the EPA sample number (including blanks and duplicates) for each required analysis within the SDG. Duplicates must contain a "D" suffix. These sample numbers must be listed on the form in ascending alphanumeric order using the Extended Binary Coded Decimal Interchange Code convention. Thus, if MAB123A is the lowest (considering both alpha and numeric characters) EPA Sample No. within the SDG, it would be entered in the first EPA Sample No. field. Samples listed below it would be ~~in ascending~~ sequence - MAB124A, MAB124B, MAB125A, MAC111A, MA1111A, MA1111AD, etc.

3.1.3.3 All EPA sample numbers must be listed in ascending alphanumeric order, continuing to the following Cover Page if applicable.

3.1.3.4 Under "Lab Sample ID", a Lab Sample ID (up to 10 characters) may be entered for each associated EPA Sample No. If a Lab Sample ID is entered, it must be entered identically (for each EPA Sample No.) on all associated data.

3.1.3.5 Under "Comments", enter any problems encountered, both technical and administrative, the corrective action taken, and resolution performed for all of the samples in the SDG.

3.1.3.6 Each Cover Page must be signed, in original, by the Laboratory Manager or the Manager's designee, and dated to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.1.4 Analysis Data Sheet [FORM I - AAVC]

3.1.4.1 This form is used for tabulating and reporting results for analysis of canister samples for the compounds in a Target Compound List for Volatiles as given in Exhibit C.

3.1.4.2 This form is used for reporting the detected concentrations of the target compounds in the field samples, blanks, laboratory control samples, and performance evaluation samples.

3.1.4.3 Complete the header information on each page of Form I-AAVC according to the instructions in section 3.1.2.

3.1.4.4 The use of a Nafion dryer invalidates all results for polar compounds. Indicate by checking the appropriate item whether or not a Nafion dryer was used.

3.1.4.5 Fill in the pressure readings and injection volume (trapped sample volume) in the appropriate blanks. Determine the dilution factor, if any, according to Exhibit D, section 4.5 and fill in the appropriate blank.

3.1.4.6 For each positively identified target compound, the Contractor shall multiply the detected concentration in ppbv by the dilution factor, and if this value is greater than or equal to the quantitation limit, report the resulting true concentration uncorrected for blank contaminants. Report analytical results to two significant figure if the value is less than 10, and three significant figures if the value is greater than or equal to 10.

3.1.4.7 Under the column labeled "Q" for qualifier, flag each result with the specific Data Reporting Qualifiers listed below. The Contractor is encouraged to use additional flags or footnotes. The definition of such flags must be explicit and must be included in the SDG Narrative.

3.1.4.8 For reporting results to the Agency, the following contract specific qualifiers are to be used. The seven qualifiers defined below are not subject to modification by the laboratory. Up to five qualifiers may be reported on Form I-AAVC for each compound. The

seven EPA-defined qualifiers to be used are as follows:

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero. For example, if the sample quantitation limit is 2 ppbv, but a concentration of 1 ppbv is calculated, report it as "1J". The sample quantitation limit must be adjusted for dilution.

N - Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds, where the identification is based on a mass spectral library search. It is applied to all TIC results.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified target compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. If one or more compounds have a response greater than full scale, except as noted in Exhibit D, the sample must be diluted and reanalyzed according to the specifications in Exhibit D. All such compounds with a response greater than full scale should have the concentration flagged with an "E" on the Form I-AAVC for the original analysis. If the dilution of the sample causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported on separate copies of Form I-AAVC. The Form I-AAVC for the diluted sample shall have the "DL" suffix appended to the sample number.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample is reanalyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the sample number on the Form I-AAVC for the diluted sample, and all concentration values reported on that Form I-AAVC are flagged with the "D" flag. This flag alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample.

X - Other specific flags may be required to properly define the results. If used, they must be fully described, and such description attached to the Sample Data Summary Package and the SDG Narrative. Begin by using "X". If more than one flag is required, use "Y" and "Z" as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "B", and "D" flags for some sample. The laboratory-defined flags are limited to the letters "X", "Y", and "Z".

NOTE: The combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are detected in the sample.

3.1.5 Tentatively Identified Compounds [FORM I - AAVC-TIC]

3.1.5.1 Fill in all header information as above.

3.1.5.2 Report Tentatively Identified Compounds (TICs) including CAS number, compound name, retention time (RT), and the estimated concentration (criteria for reporting TICs are given in Exhibit D). Retention time must be reported in minutes and decimal minutes, not seconds or minutes:seconds.

3.1.5.3 If, in the opinion of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown.

3.1.5.4 Include a Form I-AAVC-TIC for every sample and blank analyzed, even if no TICs are found. Total the number of TICs found, and enter this number in the "No. of TICs found." If none were found, enter "0" (zero). Form I-AAVC-TIC must be provided for every analysis, including required dilutions and reanalyses, even if no TICs are found.

3.1.6 Blank Summary [FORM II - AAVC]

3.1.6.1 This form summarizes the samples associated with each field and laboratory blank analysis. A copy of the appropriate Form II-AAVC is required for each blank reported on a Form I-AAVC.

3.1.6.2 Complete the header information on Form II-AAVC as described in section 3.1.2. The "EPA Sample No." entered in the box at the top of Form II-AAVC shall be the same number entered on the Form I-AAVC when reporting results for the blank itself.

3.1.6.3 On the numbered lines, enter the EPA sample numbers associated with the blank, along with the other information which

identifies the EPA samples. The Canister ID for each sample must be provided under the "Canister" column, if available.

3.1.7 Laboratory Control Sample Data Sheet [FORM III - AAVC]

3.1.7.1 Form III-AAVC is used to report the recovery of the spiked analytes in the laboratory control samples (LCS).

3.1.7.2 Complete the header information according to the instructions in section 3.1.2.

3.1.7.3 Enter the date and time the LCS was analyzed.

3.1.7.4 In the table under "Spiked," enter the spiked concentration in ppbv of each LCS compound. Under "Reported," enter the concentration obtained in ppbv calculated from the analysis of the LCS. Calculate the percent recovery of each LCS compound to the nearest whole percent and enter in the column under "% Recovery". At the bottom of the table are the QC limits for LCS percent recoveries. Flag all values outside of the limits with an "*" in the column under the "Q" symbol.

3.1.7.5 Summarize the values outside the QC limits at the lower part of the form.

3.1.7.6 Enter any comments pertinent to the analysis of the LCS.

3.1.8 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AAVC]

3.1.8.1 This form is used to report the results of GC/MS instrument performance check (also known as "tuning") and to summarize the date and time of analysis of samples, standards, and blanks associated with each analysis of the instrument performance check solution.

3.1.8.2 Complete the header information as in section 3.1.2. Enter the "Lab File ID" for the injection containing the instrument performance check mixture of BFB. Enter the date and time (military time) of injection of the instrument performance check mixture.

3.1.8.3 For each ion listed on the form, enter the percent relative abundance in the right column. Report relative abundances to the number of significant figures given for each ion in the ion abundance criteria column.

3.1.8.4 Under "to m/e 95", all ion abundances are to be normalized to the nominal base peak listed on Form IV-AAVC. For some of the ions, determine the percentage of the ion abundance to the specified mass and report under "to specified mass". For example, if the relative

ion abundance of mass 96 and mass 174 ions are 4 and 80 (under the "to m/e 95" column), respectively, then enter "5.0" (under the "to specified mass" column) as the ion abundance of mass 96 relative to mass 174.

3.1.8.5 All relative abundances must be reported as a number. If zero, enter "0", not a dash or other non-numeric character.

3.1.8.6 In the lower half of the form, list all samples and standards analyzed under that instrument performance check in chronological order, by time of analysis (in military time). Refer to section 3.1.2 for specific instructions for identifying standards and blanks. Enter "EPA Sample No.", "Lab Sample ID", "Lab File ID", "Date Analyzed", and "Time Analyzed" for all standards, samples, and blanks.

3.1.8.7 The GC/MS instrument performance check must be analyzed again twelve hours from the time of injection of the instrument performance check solution of BFB listed at the top of the form. In order to meet these requirements, samples, standards, or blanks must be injected within twelve hours of the injection of the instrument performance check solution.

3.1.9 Initial Calibration Data Sheet [FORM V - AAVC]

3.1.9.1 Each time the GC/MS system undergoes a five-point calibration to initialize subsequent quantitation of VOCs in sample and blank analysis, the laboratory must complete and submit a Form V-AAVC.

3.1.9.2 Complete all header information as in section 3.1.2.

3.1.9.3 Enter the "Case No." and "SDG No." for the current data package, regardless of the original case for which the initial calibration was performed. Enter "Instrument ID", "GC Column ID", and "Injection Volume".

3.1.9.4 Check the appropriate standard preparation method used to calibrate the GC/MS system. The following define the acronyms provided on the form:

DD(Direct)	-	Dynamic Dilution Direct Injection
DD(Canister)	-	Dynamic Dilution via Canister
SDB	-	Static Dilution Bottle Technique
HPC	-	High Pressure Cylinder
WPT	-	Water Purge and Trap Method

3.1.9.5 Enter the injection dates and times of each of the calibration standards analyzed under "Date injected" and "Time injected", respectively.

3.1.9.6 Enter the "EPA Sample No." and "Lab File ID" for each of the five calibration standards.

3.1.9.7 Complete the relative response factor (RRF) calculation for the five calibration points, and then calculate and report the average relative response factor ($\overline{RRF}$) and %RSD of the RRF values for each target compound in the space provided.

3.1.10 Continuing Calibration Data Sheet [FORM VI - AAVC]

3.1.10.1 Each time the GC/MS system undergoes a continuing calibration to check for the validity of the initial calibration, the laboratory must complete and submit a Form VI-AAVC.

3.1.10.2 Complete all header information as in section 3.1.2. Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID", "GC Column ID", and the date(s) of the most recent initial calibration. If the calendar date changes during the calibration procedure, the inclusive dates should be given on Form VI.

3.1.10.3 Enter the date and time of injection of the continuing calibration standard.

3.1.10.4 Complete the relative response factor (RRF) calculation for each target compound in the space provided.

3.1.10.5 Under the column "IC mean RRF", enter the mean relative response factor for each target compound as determined in the most recent valid initial calibration.

3.1.10.6 A value of percent difference (%D) is calculated as part of a continuing calibration check in which only the calibration standard with a concentration of 10 ppbv is analyzed. A RRF is calculated for this concentration and compared to the mean RRF value in the most recent valid initial calibration.

3.1.11 Internal Standard Area and Retention Time Summary [FORM VII - AAVC]

3.1.11.1 This form is used to summarize the peak areas and retention times of the internal standards added to all samples and blanks. The data are used to determine when changes in internal standard responses will adversely affect quantification of target compounds. This form must be completed each time an initial or continuing calibration is performed for each GC/MS system.

3.1.11.2 Complete the header information according to section 3.1.2.

3.1.11.3 Enter the Lab File ID of the 12-hour calibration standard, as well as the date and time of analysis of the calibration standard. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a continuing calibration, a Form VI-AAVC shall be completed on the basis of the internal standard areas of the mid level (10 ppbv) initial calibration sequence standard. Use the date and time of analysis of this standard, and its Lab File ID and areas in place of those of a continuing calibration standard.

3.1.11.4 From the results of the analysis of the 12-hour calibration standard, enter the area measured for each internal standard and its retention time (in decimal minutes) under the appropriate column. For each internal standard, calculate the upper limit of the area as the area of the particular standard plus 40 percent of its area (i.e., 1.4 times the area in the 12 HOUR STD box), and the lower limit of the area as the area of the internal standard minus 40% of its area (i.e., 0.6 times the area in the 12 HOUR STD box). Report these values in the boxes labeled "UPPER LIMIT" and "LOWER LIMIT", respectively.

3.1.11.5 Calculate the upper limit of the retention time as the retention of the internal standard plus 0.33 minutes (20 seconds), and the lower limit of the retention time as the retention time in the standard minus 0.33 minutes (20 seconds).

3.1.11.6 For each sample and blank under a given 12-hour analytical sequence, enter the EPA Sample Number and the area measured for each internal standard and its retention time. If the internal standard area is outside the upper or lower limits calculated above, flag that area with an asterisk (*) placed in the far right-hand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the retention time of any internal standard that is outside the limits with an asterisk.

3.1.12 Canister Certification [FORM VIII - AAVC]

3.1.12.1 This form is used to document the certification of canisters prior to use.

3.1.12.2 Complete the header information on each page of Form VII-AAVC according to the instructions in section 3.1.2.

3.1.12.3 Enter the results of the leak test procedure in the appropriate spaces provided.

3.1.12.4 Enter in Column 1, the results of the analysis of an unspiked certified clean canister. For target compounds that are not detected, enter the CRQL of the compound followed by a "U". If the detected level is less than the CRQL, enter the value followed by a

"J".

3.1.12.5 Enter in Column 2, under the "Initial" spiked concentration, the results of the initial analysis of a canister spiked with the target compounds.

3.1.12.6 Enter in Column 3, under the "Final" spiked concentration, the results of the analysis of the spiked canister after ageing for seven days.

3.1.12.7 For each standard, enter the percent difference (%D) in Column 4 using the formula given in Exhibit D, section 3.3.4.2. If this value is within ±30 percent, the canister is certified.

3.1.13 **Analytical Sequence [FORM IX - AAVC]**

3.1.13.1 A Form IX-AAVC is required for each analytical sequence for each GC/MS system used to perform VOA on canister samples in an SDG.

3.1.13.2 Complete the header information on each page of Form VII-AAVC according to the instructions in section 3.1.2.

3.1.13.3 On the numbered lines, enter the EPA sample numbers along with the other information which identifies the samples, blanks, and standards. The first item in the table must be the BFB since the 12-hour time period starts at the injection of the instrument performance check standard. Arrange the items in chronological order for each GC/MS system.

3.1.14 **Sample Receipt/Log-In Sheet [FORM AADC-1]**

3.1.14.1 This form is used to document the receipt and inspection of sample containers and samples. One original of Form AADC-1 is required for each sample shipping container. If the samples in a single sample shipping container must be assigned to more than one Sample Delivery Group, the original Form AADC-1 shall be placed with the deliverables for the Sample Delivery Group of the lowest Arabic number and a copy of Form AADC-1 must be placed with the deliverables for the other Sample Delivery Group(s). The copies should be identified as "copy(ies)," and the location of the original should be noted on the copies.

3.1.14.2 Sign and date the airbill (if present). Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in item 1 on Form AADC-1. Record the custody seal numbers in item 2.

3.1.14.3 Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody

record(s), SMO forms (i.e., Traffic Reports, Packing Lists), and airbills or airbill stickers in items 3-5 on Form AADC-1. Specify if there is an airbill present or an airbill sticker in item 5 on Form AADC-1. Record the airbill or sticker number, if present.

3.1.14.4 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the canister (i.e., intact, dent, leaking) and presence of absence of sample tags in items 6 and 7 on Form AADC-1.

3.1.14.5 Review the sample shipping documents and complete the header information described in section 3.1.2. Compare the information recorded on all the documents and samples and circle the appropriate answer in item 8 on Form AADC-1.

3.1.14.6 If there are no problems observed during receipt, sign and date (include time) Form AADC-1, the chain-of-custody record, and Traffic Report, and write the sample numbers on Form AADC-1. Record the appropriate sample tags and assigned laboratory numbers, if applicable. The log-in date should be recorded at the top of Form AADC-1 and the date and time of sample receipt at the laboratory should be recorded in items 9 and 10. Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of Form AADC-1. Sign and date the Sample Transfer block. Cross out unused columns and spaces.

3.1.14.7 If there are problems observed during receipt or an answer marked with an asterisk (i.e., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.1.15 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

3.1.15.1 This form is used to record the inventory of the SDG File Purge documents and count of documents in the original Sample Data Package which is sent to the Region.

3.1.15.2 Organize all EPA-CSF documents as described in Exhibit B, Section 1. Assemble the documents in the order specified on Form AADC-2, and stamp each page with a consecutive number. (Do not number the AADC-2 form). Inventory the CSF by reviewing the document numbers and recording page numbers ranges in the columns provided in the Form AADC-2. If there are no documents for a specific document type, enter an "NA" in the empty space.

3.1.15.3 Certain laboratory specific documents related to the CSF may

not fit into a clearly defined category. The laboratory should review AADC-2 to determine if it is most appropriate to place them under No. 17, 18, 19, or 20. Category 20 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.2 **Data Reporting Forms**

3.2.1 Cover Page [COVER PAGE - AAVC]

3.2.2 Analysis Data Sheet [FORM I - AAVC]

3.2.3 Tentatively Identified Compounds [FORM I - AAVC-TIC]

3.2.4 Blank Summary [FORM II - AAVC]

3.2.5 Laboratory Control Sample Data Sheet [FORM III - AAVC]

3.2.6 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AAVC]

3.2.7 Initial Calibration Data Sheet [FORM V - AAVC]

3.2.8 Continuing Calibration Data Sheet [FORM VI - AAVC]

3.2.9 Internal Standard Area and Retention Times Summary [FORM VII - AAVC]

3.2.10 Canister Certification [FORM VIII - AAVC]

3.2.11 Analytical Sequence [FORM IX - AAVC]

3.2.12 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.2.13 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

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COVER PAGE

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

	EPA Sample No.	Lab Sample ID
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

Comments:

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data (if submitted) on floppy diskette has been authorized by the Laboratory Manager's designee, as verified by the following signature:

Signature:_____ Name:_____
Date:_____ Title:_____

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SAMPLE RECEIPT/LOG–IN SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

ITEM	REMARKS	EPA Sample No.	Sample Tag No.	Assigned Lab No.	Spl Vol (m^3)	REMARKS: Conditions of Sample Shipment,etc.
1. Custody Seal(s)	Present/Absent/Intact/Broken*					
2. Custody Seal No(s).						
3. Chain–of–Custody records	Present/Absent*					
4. Traffic Reports or	Present/Absent*					
Packing List						
5. Airbill	Sticker/Present/Absent*					
Airbill No(s).						
6. Sample Tags	Present/Absent*					
Sample Tag No(s):	Listed/Not Listed on COC					
7. Sample Condition	Intact/Broken/Leaking*					
8. Do informations on custody						
records, traffic reports, and						
sample tags agree?	Yes/No*					
9. Date Received at Lab:						
10. Time Received at Lab:						
Sample Transfer						
Area #:						
By:						
On:						

* If circled, contact SMO and attach record of resolution.

Received by:
Signature:_____ Log–in Date:_____
Print Name:_____

Reviewed by:
Signature:_____ Date:_____
Logbook No.:_____ Logbook Page No.:_____

FORM AADC–1

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COMPLETE SDG FILE (CSF) DOCUMENT INVENTORY SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No:_____ SDG No.:_____

DOCUMENT	Page Nos.		Please Check	
	From	To	Lab	Reg
1. Cover Page (Cover Page – AAVC)				
2. Sample Receipt/Log–In Sheet (FORM AADC–1)				
3. CSF Document Inventory Sheet (FORM AADC–2)				
4. Analysis Data Sheet (FORM I – AAVC)				
5. Tentatively Identified Compounds (FORM I – AAVC–TIC)				
6. Blank Summary Form (FORM II – AAVC)				
7. Laboratory Control Sample Data Sheet (FORM III – AAVC)				
8. GC/MS Tuning with BFB (FORM IV – AAVC)				
9. Initial Calibration Data Sheet (FORM V – AAVC)				
10. Continuing Calibration Data Sheet (FORM VI – AAVC)				
13. Internal Standard Area and RT Summary (FORM VII – AAVC)				
14. Canister Certification Data Sheet (FORM VIII – AAVC)				
15. Analytical Sequence (FORM IX – AAVC)				
16. EPA Shipping/Receiving Documents				
Airbill (No. of shipments:___)				
Chain–of–Custody Records				
Sample Tags				
Sample Log–In Sheet (Lab & AADC–1)				
17. Misc. Shipping/Receiving Records (list individual records)				
Telephone Logs				
18. Internal Lab Sample Transfer Records				
19. Internal Original Sample Preparation and Analysis Records				
20. Other Records (describe or list)				

Comments:_____

Completed by (CLP Lab):
Signature:_____ Date:_____
Printed Name/Title:_____
Audited by (EPA):
Signature:_____ Date:_____
Printed Name/Title:_____

FORM AADC–2

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ANALYSIS DATA SHEET

EPA Sample No. _____

Lab Name: _____ Contract No.: _____
Lab Code: _____ Case No.: _____
SAS No.: _____ SDG No.: _____ Nafion Dryer: ( )Y (/)N
Lab Sample ID: _____ Date Received: _____ Instrument ID: _____
Lab File ID: _____ Date Analyzed: _____ Column ID: _____
Canister pressure on tag (units): _____( ) Injection volume (cm^3): _____
Canister pressure as received (units): _____( ) Dilution factor: _____

CAS RN	COMPOUND NAME	Concentration (ppbv)	Q
67-64-1	Acetone		
75-05-8	Acetonitrile		
107-02-8	Acrolein		
107-13-1	Acrylonitrile		
71-43-2	Benzene		
100-44-7	Benzyl chloride		
75-27-4	Bromodichloromethane		
74-83-9	Bromomethane		
106-99-0	1,3-Butadiene		
78-93-3	2-Butanone		
56-23-5	Carbon tetrachloride		
108-90-7	Chlorobenzene		
75-45-6	Chlorodifluoromethane		
75-00-3	Chloroethane		
67-66-3	Chloroform		
74-87-3	Chloromethane		
107-05-1	3-Chloro-1-propene		
124-48-1	Dibromochloromethane		
106-93-4	1,2-Dibromoethane		
95-50-1	1,2-Dichlorobenzene		
541-73-1	1,3-Dichlorobenzene		
106-46-7	1,4-Dichlorobenzene		
75-71-8	Dichlorodifluoromethane		
75-34-3	1,1-Dichloroethane		
107-06-2	1,2-Dichloroethane		
75-35-4	1,1-Dichloroethene		
156-59-2	cis-1,2-Dichloroethene		
156-60-5	trans-1,2-Dichloroethene		
78-87-5	1,2-Dichloropropane		
10061-01-5	cis-1,3-Dichloropropene		

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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____ Nafion Dryer: ( )Y ( )N
Lab Sample ID:_____ Date Received:_____ Instrument ID:_____
Lab File ID:_____ Date Analyzed:_____ Column ID:_____
Canister pressure on tag (units):_____( ) Injection volume (cm^3):_____
Canister pressure as received (units):_____( ) Dilution factor:_____

CAS RN	COMPOUND NAME	Concentration (ppbv)	Q
10061-02-6	trans-1,3-Dichloropropene		
1320-37-2	1,2-Dichloro-1,1,2,2-tetrafluoroethane		
100-41-4	Ethylbenzene		
142-82-5	Heptane		
87-68-3	Hexachlorobutadiene		
110-54-3	Hexane		
167-56-1	Methanol		
75-09-2	Methylene chloride		
80-62-6	Methyl methacrylate		
108-10-1	4-Methyl-2-pentanone		
98-83-9	alpha-Methyl styrene		
111-65-9	Octane		
109-66-0	n-Pentane		
115-07-1	Propylene		
100-42-5	Styrene		
79-43-5	1,1,2,2-Tetrachloroethane		
127-18-4	Tetrachloroethylene		
108-88-3	Toluene		
120-82-1	1,2,4-Trichlorobenzene		
71-55-6	1,1,1-Trichloroethane		
79-06-5	1,1,2-Trichloroethane		
79-01-6	Trichloroethylene		
75-69-4	Trichlorofluoromethane		
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane		
95-63-6	1,2,4-Trimethylbenzene		
108-67-8	1,3,5-Trimethylbenzene		
108-05-4	Vinyl acetate		
75-01-4	Vinyl chloride		
1330-20-7	Xylenes, m- & p-		
95-47-6	Xylene, o-		

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TENTATIVELY IDENTIFIED COMPOUNDS

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SAS No.:_____ Date Received:_____
SDG No:_____ Date Analyzed:_____
GC Column ID:_____ Instrument ID:_____
No. of TICs Found:_____

	CAS RN	COMPOUND NAME	Estimated Concentration (ppbv)	RT (minutes)	Q
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

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CONTRACT LABORATORY PROGRAM
Volatile Organics in Ambient Air – Canister

BLANK SUMMARY

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Analyzed:_____ Instrument ID:_____
Lab File ID:_____ Time Analyzed:_____ Column ID:_____

THIS BLANK APPLIES TO THE FOLLOWING SAMPLES:

	EPA Sample No.	Laboratory ID			Analysis	
		Sample	File	Canister	Date	Time
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

Comments:

U. S. ENVIRONMENTAL PROTECTION AGENCY
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LABORATORY CONTROL SAMPLE DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Analyzed:_____ Instrument ID:_____
Lab File ID:_____ Time Analyzed:_____ Column ID:_____
Injection Volume (cm^3):_____

		Concentration (ppbv)		%	
CAS RN	COMPOUND NAME	Spiked	Reported	Recovery	Q
71–43–2	Benzene				
56–23–5	Carbon tetrachloride				
106–93–4	1,2–Dibromoethane				
106–46–7	1,4–Dichlorobenzene				
107–06–2	1,2–Dichloroethane				
78–87–5	1,2–Dichloropropane				
10061–02–6	trans–1,3–Dichloropropene				
127–18–4	Tetrachloroethylene				
79–06–5	1,1,2–Trichloroethane				
79–01–6	Trichloroethylene				
75–01–4	Vinyl chloride				

%Recovery QC Limits: 60–140%

LCS Recovery: _____ outside limits out of _____ total.

Comments:

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GC/MS INSTRUMENT PERFORMANCE CHECK AND MASS CALIBRATION

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Injected:_____ Instrument ID:_____
Lab File ID:_____ Time Injected:_____ GC Column ID:_____
Injection Volume (cm^3):_____ Mass BFB Injected (ng):_____

m/e	ION ABUNDANCE CRITERIA	% Relative Abundance	
		to m/e 95	to specified mass
50	8.0 – 40.0% of m/e 95		
75	30.0 – 66.0 of m/e 95		
95	Base peak, 100% relative abundance		
96	5.0 – 9.0% of m/e 174		
173	Less than 2.0% of m/e 174		
174	50.0 – 120.0% of m/e 95		
175	4.0 – 9.0% of m/e 174		
176	93.0 – 101.0% of m/e 174		
177	5.0 – 9.0% of m/e 176		

THIS TUNE APPLIES TO THE FOLLOWING:

	EPA Sample No.	Lab Sample ID	Lab File ID	Date Analyzed	Time Analyzed
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

U. S. ENVIRONMENTAL PROTECTION AGENCY
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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Instrument ID:_____ GC Column ID:_____ Injection Volume (cm^3):_____
Standard Preparation Method: ___DD(Direct) ___DD(Canister) ___SDB ___HPC ___WPT

STANDARD	2	5	10	20	50
EPA Sample No.					
Lab File ID					
Date injected					
Time injected					

COMPOUND NAME	Relative Response Factor (RRF) 2	5	10	20	50	mean RRF	%RSD
Acetone							
Acetonitrile							
Acrolein							
Acrylonitrile							
Benzene							
Benzyl chloride							
Bromodichloromethane							
Bromomethane							
1,3–Butadiene							
2–Butanone							
Carbon tetrachloride							
Chlorobenzene							
Chlorodifluoromethane							
Chloroethane							
Chloroform							
Chloromethane							
3–Chloro–1–propene							
Dibromochloromethane							
1,2–Dibromoethane							
1,2–Dichlorobenzene							
1,3–Dichlorobenzene							
1,4–Dichlorobenzene							
Dichlorodifluoromethane							
1,1–Dichloroethane							
1,2–Dichloroethane							
1,1–Dichloroethene							
cis–1,2–Dichloroethene							
trans–1,2–Dichloroethene							
1,2–Dichloropropane							
cis–1,3–Dichloropropene							

U. S. ENVIRONMENTAL PROTECTION AGENCY
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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Instrument ID:_____ GC Column ID:_____ Injection Volume (cm^3):_____
Standard Preparation Method: ___DD(Direct) ___DD(Canister) ___SDB ___HPC ___WPT

STANDARD	2	5	10	20	50
EPA Sample No.					
Lab File ID					
Date injected					
Time injected					

COMPOUND NAME	Relative Response Factor (RRF) 2	5	10	20	50	mean RRF	%RS
trans-1,3-Dichloropropene							
1,2-Dichloro-1,1,2,2-tetrafluoroethane							
Ethylbenzene							
Heptane							
Hexachlorobutadiene							
Hexane							
Methanol							
Methylene chloride							
Methyl methacrylate							
4-Methyl-2-pentanone							
alpha-Methyl styrene							
Octane							
n-Pentane							
Propylene							
Styrene							
Tetrachloroethylene							
1,1,2,2-Tetrachloroethane							
Toluene							
1,2,4-Trichlorobenzene							
1,1,1-Trichloroethane							
1,1,2-Trichloroethane							
Trichloroethylene							
Trichlorofluoromethane							
1,1,2-Trichloro-1,2,2-trifluoroethane							
1,2,4-Trimethylbenzene							
1,3,5-Trimethylbenzene							
Vinyl acetate							
Vinyl chloride							
Xylenes, m- & p-							
Xylene, o-							

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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
EPA Sample No.:_____ Lab File ID:_____
Instrument ID:_____ GC Column ID:_____
Date Injected:_____ Time Injected:_____
Injection Volume (cm^3):_____ Date of Init. Cal.:_____

COMPOUND NAME	IC mean RRF	RRF	%D
Acetone			
Acetonitrile			
Acrolein			
Acrylonitrile			
Benzene			
Benzyl chloride			
Bromodichloromethane			
Bromomethane			
1,3–Butadiene			
2–Butanone			
Carbon tetrachloride			
Chlorobenzene			
Chlorodifluoromethane			
Chloroethane			
Chloroform			
Chloromethane			
3–Chloro–1–propene			
Dibromochloromethane			
1,2–Dibromoethane			
1,2–Dichlorobenzene			
1,3–Dichlorobenzene			
1,4–Dichlorobenzene			
Dichlorodifluoromethane			
1,1–Dichloroethane			
1.2–Dichloroethane			
1,1–Dichloroethene			
cis–1,2–Dichloroethene			
trans–1,2–Dichloroethene			
1,2–Dichloropropane			
cis–1.3–Dichloropropene			

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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
EPA Sample No.:_____ Lab File ID:_____
Instrument ID:_____ GC Column ID:_____
Date Injected:_____ Time Injected:_____
Injection Volume (cm^3):_____ Date of Init. Cal.:_____

COMPOUND NAME	IC mean RRF	RRF	%D
trans–1,3–Dichloropropene			
1,2–Dichloro–1,1,2,2–tetrafluoroethane			
Ethylbenzene			
Heptane			
Hexachlorobutadiene			
Hexane			
Methanol			
Methylene chloride			
Methyl methacrylate			
4–Methyl–2–pentanone			
alpha–Methyl styrene			
Octane			
n–Pentane			
Propylene			
Styrene			
1,1,2,2–Tetrachloroethane			
Tetrachloroethylene			
Toluene			
1,2,4–Trichlorobenzene			
1,1,1–Trichloroethane			
1,1,2–Trichloroethane			
Trichloroethylene			
Trichlorofluoromethane			
1,1,2–Trichloro–1,2,2–trifluoroethane			
1,2,4–Trimethylbenzene			
1,3,5–Trimethylbenzene			
Vinyl acetate			
Vinyl chloride			
Xylenes, m– & p–			
Xylene, o–			

U. S. ENVIRONMENTAL PROTECTION AGENCY
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INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

	Chlorobenzene–d_5				Bromochloromethane				1,4–Difluorobenzene			
	Area	#	RT	#	Area	#	RT	#	Area	#	RT	#
12–HOUR STANDARD												
Upper Limit												
Lower Limit												
EPA Sample No.												
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

AREA: Upper Limit: +40% of internal standard area. RT: Upper Limit: +0.33 minutes of internal standard RT.
Lower Limit: –40% of internal standard area. Lower Limit: –0.33 minutes of internal standard RT.

All values outside of the QC limits must be followed by an "*" under the "#" column.

FORM VII – AAVC

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Volatile Organics in Ambient Air – Canister

CANISTER CERTIFICATION

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No:_____
Case No.:_____ SDG No.:_____
Canister ID:_____ SAS No.:_____
Leak Test (psi/kPa):Initial Pressure:_____ Initial Vacuum:_____
Final Pressure:_____ Final Vacuum:_____

	COMPOUND NAME	1	2	3	4
		Unspiked Concentration	Spiked Concentration		%D (2 and 3)
			Initial	Final	
1	Acetone				
2	Acetonitrile				
3	Acrolein				
4	Acrylonitrile				
5	Benzene				
6	Benzyl chloride				
7	Bromodichloromethane				
8	Bromomethane				
9	1,3–Butadiene				
10	2–Butanone				
11	Carbon tetrachloride				
12	Chlorobenzene				
13	Chlorodifluoromethane				
14	Chloroethane				
15	Chloroform				
16	Chloromethane				
17	3–Chloro–1–propene				
18	Dibromochloromethane				
19	1,2–Dibromoethane				
20	1,2–Dichlorobenzene				
21	1,3–Dichlorobenzene				
22	1,4–Dichlorobenzene				
23	Dichlorodifluoromethane				

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Volatile Organics in Ambient Air – Canister

CANISTER CERTIFICATION

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No:_____
Case No.:_____ SDG No.:_____
Canister ID:_____ SAS No.:_____
Leak Test (psi/kPa):Initial Pressure:_____ Initial Vacuum:_____
Final Pressure:_____ Final Vacuum:_____

	COMPOUND NAME	1	2	3	4
		Unspiked Concentration	Spiked Concentration Initial	Final	%D (2 and 3)
24	1,1–Dichloroethane				
25	1,2–Dichloroethane				
26	1,1–Dichloroethene				
27	cis–1,2–Dichloroethene				
28	trans–1,2–Dichloroethene				
29	1,2–Dichloropropane				
30	cis–1,3–Dichloropropene				
31	trans–1,3–Dichloropropene				
32	1,2–Dichloro–1,1,2,2–tetrafluoroethane				
33	Ethylbenzene				
34	Heptane				
35	Hexachlorobutadiene				
36	Hexane				
37	Methanol				
38	Methylene chloride				
39	Methyl methacrylate				
40	4–Methyl–2–pentanone				
41	alpha–Methyl styrene				
42	Octane				
43	n–Pentane				
44	Propylene				
45	Styrene				
46	1,1,2,2–Tetrachloroethane				

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CONTRACT LABORATORY PROGRAM
Volatile Organics in Ambient Air – Canister

CANISTER CERTIFICATION

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No:_____
Case No.:_____ SDG No.:_____
Canister ID:_____ SAS No.:_____
Leak Test (psi/kPa):Initial Pressure:_____ Initial Vacuum:_____
Final Pressure:_____ Final Vacuum:_____

	COMPOUND NAME	1	2	3	4
		Unspiked Concentration	Spiked Concentration		%D
			Initial	Final	(2 and 3)
47	Tetrachloroethylene				
48	Toluene				
49	1,2,4–Trichlorobenzene				
50	1,1,1–Trichloroethane				
51	1,1,2–Trichloroethane				
52	Trichloroethylene				
53	Trichlorofluoromethane				
54	1,1,2-Trichloro-1,2,2-trifluoroethane				
55	1,2,4–Trimethylbenzene				
56	1,3,5–Trimethylbenzene				
57	Vinyl acetate				
58	Vinyl chloride				
59	Xylenes, m– & p–				
60	Xylene, o–				
	OTHERS				
1					
2					
3					
4					
5					
6					
7					
8					

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Volatile Organics in Ambient Air – Canister

ANALYTICAL SEQUENCE

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Instrument ID:_____ GC Column ID:_____

	EPA Sample No.	Lab Sample ID	Analysis Time	Analysis Date
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

EXHIBIT C

TARGET COMPOUND LIST

TABLE 1

VOA OF AMBIENT AIR IN CANISTERS TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Target Compound	CAS RN	CRQL, ppbv*
Acetone	67-64-1	5
Acetonitrile	75-05-8	5
Acrolein	107-02-8	5
Acrylonitrile	107-13-1	5
Benzene	71-43-2	2
Benzyl chloride	100-44-7	5
Bromodichloromethane	75-27-4	2
Bromomethane	74-83-9	5
1,3-Butadiene	106-99-0	5
2-Butanone	78-93-3	5
Chloromethane	74-87-3	2
Carbon tetrachloride	56-23-5	2
Chlorobenzene	108-90-7	2
Chlorodifluoromethane	75-45-6	2
Chloroethane	75-00-3	2
Chloroform	67-66-3	2
3-Chloro-1-propene	107-05-1	2
Dibromochloromethane	124-48-1	2
1,2-Dibromoethane	106-93-4	2
1,2-Dichlorobenzene	95-50-1	2
1,3-Dichlorobenzene	541-73-1	5
1,4-Dichlorobenzene	106-46-7	5
Dichlorodifluoromethane	75-71-8	2
1,1-Dichloroethane	75-34-3	2
1,2-Dichloroethane	107-06-2	2
1,1-Dichloroethene	75-35-4	5
cis-1,2-Dichloroethylene	156-59-2	2
trans-1,2-Dichloroethylene	156-60-5	2
1,2-Dichloropropane	78-87-5	2
cis-1,3-Dichloropropene	10061-01-5	2
trans-1,3-Dichloropropene	10061-02-6	2
1,2-Dichloro-1,1,2,2-tetrafluoroethane	1320-37-2	2
Ethylbenzene	100-41-4	2
Heptane	142-82-5	2
Hexachlorobutadiene	87-68-3	5
Hexane	110-54-3	2

* ppbv referenced to 25°C and 760 mm Hg.

TABLE 1

VOA OF AMBIENT AIR IN CANISTERS
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(continued)

Target Compound	CAS RN	CRQL, ppbv*
Methanol	67-56-1	5
Methylene chloride	75-09-2	5
Methyl methacrylate	80-62-6	2
4-Methyl-2-pentanone	108-10-1	5
alpha-Methyl styrene	98-83-9	5
Octane	111-65-9	2
n-Pentane	109-66-0	2
Propylene	115-07-1	5
Styrene	100-42-5	5
1,1,2,2-Tetrachloroethane	79-43-5	2
Tetrachloroethylene	127-18-4	5
Toluene	108-88-3	5
1,2,4-Trichlorobenzene	120-82-1	2
1,1,1-Trichloroethane	71-55-6	5
1,1,2-Trichloroethane	79-06-5	5
Trichloroethylene	79-01-6	2
Trichlorofluoromethane	75-69-4	2
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2
1,2,4-Trimethylbenzene	95-63-6	2
1,3,5-Trimethylbenzene	108-67-8	2
Vinyl acetate	108-05-4	5
Vinyl chloride	75-01-4	2
Xylenes, m- and p-	1330-20-7	5
Xylene, o-	95-47-6	5

NOTE: The values in Table 1 are Contract Required Quantitation Limits (CRQL), not absolute detection limits. The quantitation limits in these tables are set at or slightly above the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

* ppbv referenced to 25°C and 760 mm Hg.

TABLE 2

VOLATILE ORGANIC COMPOUNDS FOR TENTATIVE IDENTIFICATION AND DEVELOPMENT OF CRQL DATA*

Target Compound	CAS RN
Carbon disulfide	75-15-0
Pyridine	110-86-1
1,2-Dibromo-3-chloropropane	96-12-8
2-Chloro-1,3-butadiene	126-99-8
trans-1,4-Dichlorobutene	110-57-6
Propanal	123-38-6
2-Hexanone	591-78-6
Cyclohexanone	108-94-1
1-Bromobutane	109-65-9
2-Methylnaphthalene	91-57-6
1,3,4-Trimethylbenzene	95-63-6
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
n-Propylbenzene	103-65-1
tert-Butylbenzene	98-06-6
sec-Butylbenzene	135-98-8
1,2,3-Trichlorobenzene	87-61-6

This table lists additional compounds for which the method has not been validated, but which were ranked as air toxics of concern or identified as compounds of Regional interest during the development of this document. Data developed during method validation and subsequent analysis under SAS may lead to validation of the VOC methods in this document for some or all of these compounds.

EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED IN SUMMA® CANISTERS AND ANALYZED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

December, 1991

EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED IN SUMMA® CANISTERS AND ANALYZED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

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EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED IN SUMMA® CANISTERS AND ANALYZED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1 INTRODUCTION

1.1 SCOPE AND APPLICATION

1.1.1 The analytical methods that follow are designed to analyze whole air samples collected in SUMMA® polished, stainless steel canisters for the volatile organic compounds on the Target Compound List for Volatiles in Exhibit C. The compounds to be identified and quantified from canister samples are those listed in Table D/VC-1 with the Contract Required Quantitation Limits shown therein. The methods are based on EPA Compendium Method TO-14, "The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMA® Passivated Canister Sampling and Gas Chromatographic Analysis," with appropriate modifications. The modifications include a more generic description of the method for preconcentration of target compounds and for generation of standards. One result of the modifications is that many systems currently in use for VOC analysis can be adapted to these analytical methods. Any reasonable analytical system can be used provided the system produces results equivalent to the results specified in Section 5.

1.1.2 This method is divided into the following sections: sample storage and holding times; canister cleaning and certification; optional sample screening; GC/MS analysis; and analytical performance requirements. As described in the screening section, a portion of the sample may be analyzed by GC/FID to determine the concentration level of the volatiles and the possible need for sample dilution. The GC/MS analysis section contains a description of the full scan MS analysis after gas chromatographic separation. The sample preconcentration technique is included in the analysis section because GC/MS operation and the preconcentration technique are interrelated. The section on performance requirements discusses detection limits, audit accuracy, and replicate precision that have been achieved in EPA-sponsored network operations, and which the laboratory must meet when performing analyses under this document.

1.1.3 This method is applicable to specific VOCs that have been tested and determined to be stable when stored in SUMMA® polished canisters. The volatile organic compounds that have been collected in pressurized canisters and successfully analyzed by this method are listed in Table D/VC-1 along with target compounds for which storage stability in

canisters has not been fully documented. These compounds have been successfully measured at the parts per billion by volume (ppbv) level.

1.2 SUMMARY OF METHOD

1.2.1 In the field, a sample of air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated SUMMA® passivated canister. After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to a predetermined laboratory for analysis.

1.2.2 During analysis, water vapor may be reduced in the gas stream by a Nafion® dryer (if applicable), although the use of the dryer invalidates all analysis results of polar compounds. The VOCs are then concentrated by collection in a cryogenically-cooled trap. The cryogen is then removed and the temperature of the trap is raised. The VOCs originally collected in the trap are revolatilized, separated on a GC column, then detected by mass spectrometry.

1.3 INTERFERENCES

1.3.1 Chloromethane, vinyl chloride, bromomethane, and chloroethane can display peak broadening if the compounds are not delivered to the GC column in a tight band.

1.3.2 Polar compounds such as methanol, ethanol, butanol, acetonitrile, and methyl ethyl ketone have been found to be less stable than the more nonpolar VOCs when stored in canisters over seven-day periods.

1.3.3 In cases where Nafion® tubing is used to dry the sample stream, polar compounds are not reported.

1.3.4 Interferences in canister samples may result from contamination of the canisters due to poor manufacturing practices, contamination of the canister cleaning apparatus, contamination of the sampling system or analytical system, and improper use or storage. Attention to the following details will help to minimize the possibility of contamination of canisters.

1.3.5 Canisters should be manufactured using high quality welding and cleaning techniques, and new canisters should be filled with humidified zero air which is then analyzed to determine cleanliness. The cleaning apparatus, sampling system, and analytical system should be assembled of clean, high quality components and each system should be shown to be free of contamination.

1.3.6 Canisters should be stored in a contaminant-free location and should be capped tightly during shipment and when not in use to prevent

leakage of ambient air into or out of the canister in the event that a leak develops in the valve.

1.3.7 Impurities in the dilution gas (if applicable) and carrier gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humid zero air blanks. The use of tubing other than chromatographic grade stainless steel, non-TFE thread sealants, or flow controllers with rubber components must be avoided.

1.3.8 Significant contamination of the analytical equipment can occur whenever analysis of samples containing high VOC concentrations are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of humid zero air to check for cross-contamination.

1.3.9 The laboratory where analysis of volatiles is performed should be completely free of solvents.

1.4 DEFINITIONS

NOTE: Definitions used in this document and any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E355. All pertinent abbreviations and symbols are defined within this document at point of use.

1.4.1 Absolute canister pressure = Pg + Pa, where Pg = gauge pressure in the canister (kPa, psi) and Pa = barometric pressure.

1.4.2 Absolute pressure: Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mm Hg, or psia.

1.4.3 Cryogen: A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp -195.8) or liquid argon (bp -185.7°C).

1.4.4 Dynamic calibration: Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

1.4.5 Dynamic dilution: Means of preparing calibration mixtures in which standard gas(es) from pressurized cylinders are continually blended with humidified zero air in a manifold so that a flowing stream of

calibration mixture is available at the inlet of the analytical system.

1.4.6 Gauge pressure: Pressure measured above ambient atmospheric pressure (as opposed to absolute pressure). Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

1.4.7 MS-SCAN: The GC is coupled to a MS programmed in the SCAN mode to scan a range of ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

1.4.8 Megabore® column: Chromatographic column having an internal diameter (I.D.) greater than 0.50 mm. The Megabore® column is a trademark of the J&W Scientific Co. For purposes of this method, Megabore® refers to chromatographic columns with 0.53 mm I.D.

1.4.9 Qualitative accuracy: The ability of an analytical system to correctly identify compounds.

1.4.10 Quantitative accuracy: The ability of an analytical system to correctly measure the concentration of an identified compound.

2 SAMPLE STORAGE AND HOLDING TIMES

2.1 CANISTER RECEIPT

2.1.1 Receipt of each canister is recorded in a laboratory notebook dedicated to this use. The overall condition of each sample canister is observed and noted in the dedicated laboratory notebook. Each canister should be received with an attached sample identification tag. Note on the identification tag the date received and initials of recipient.

2.1.2 The pressure of the canister is checked by attaching a pressure gauge to the canister inlet. The canister valve is opened briefly and the gauge pressure (kPa, psig) is recorded.

2.1.3 If gauge pressure is <83 kPa (<12 psig), the user may wish to pressurize the canisters, as an option, with humid zero air up to a gauge pressure of approximately 137 kPa (20 psig) to ensure that enough sample is available for analysis.

NOTE: Pressurizing the canister can introduce additional error, increase the method detection limit (MDL), and consume time. The user should weigh these limitations as part of his program objectives before pressurizing.

2.1.4 If the pressure is significantly different (greater than 10 percent) from the original pressure recorded on the canister ID tag, the integrity of the sample is suspect and the data must be flagged with a "P".

2.2 PROCEDURES FOR SAMPLE STORAGE

2.2.1 The samples must be retained from the validated time of sample receipt (VTSR) until 45 days after delivery of a complete sample data package to the Agency. After 45 days, the samples may be disposed of in a manner that complies with all applicable regulations. Disposal is expected to occur during the process of canister recleaning.

2.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants. The canisters should not be stored at elevated temperatures. High temperatures may precipitate leaks and may lead to thermal alteration of target compounds.

2.2.3 Samples and standards must be stored separately.

2.3 CONTRACT REQUIRED HOLDING TIMES

Analysis of air samples in canisters must be completed within 14 days of the VTSR.

3 CANISTER PREPARATION AND CERTIFICATION

3.1 CANISTER PREPARATION

3.1.1 Summary

3.1.2 The SUMMA© polished stainless steel canisters are cleaned by alternate pressurization and evacuation. Two methods of cleaning techniques are described in this section. In the alternate cleaning procedure, the canisters are placed in an oven.

3.1.3 After cleaning, prior to field sampling, canisters are tested to ensure that the canister seals are leak-tight by determining the change, if any, in canister pressure after 24 hours from a pressurized and an evacuated state.

3.1.4 Apparatus and Materials

3.1.4.1 Canisters: Stainless steel vessels (e.g., 6-L, 15-L) which are polished internally by the SUMMA® process and fitted with a stainless steel, high temperature, packless, leak-tight bellows valve capped with a ¼ inch stainless steel fitting.

3.1.4.2 Vacuum pump: Capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.

3.1.4.3 Manifold: Stainless steel manifold with connections for simultaneously cleaning several canisters.

3.1.4.4 Shut-off valve(s): On-off toggle valves.

3.1.4.5 Thermocouple vacuum gauge: Capable of measuring vacuum in the manifold to an absolute pressure of 0.05 mm Hg or less.

3.1.4.6 Cryogenic trap (2 required): Stainless steel U-shaped open tubular trap cooled with liquid argon or liquid nitrogen to prevent contamination from back diffusion of oil from vacuum pump and to provide clean zero air to sample canister(s).

3.1.4.7 Stainless steel pressure gauges (2): 0-345 kPa (0-50 psig) to monitor zero air pressure.

3.1.4.8 Stainless steel flow control valve: To regulate flow of zero air into canister(s).

3.1.4.9 Humidifier: Pressurizable water bubbler or other system capable of providing moisture to the zero air supply.

3.1.4.10 Isothermal oven (optional): For heating canisters.

3.1.5 Reagents

3.1.5.1 Deionized water: High performance liquid chromatography (HPLC) grade for humidified zero air stream.

3.1.5.2 Cryogen: Liquid argon or liquid nitrogen.

3.1.6 Cleaning Procedure

3.1.6.1 A canister cleaning system may be assembled, as illustrated in Figure D/VC-1, where cryogen is added to both the vacuum pump and zero air supply traps. Figure D/VC-5 provides a flow diagram of the canister cleaning process by this method.

3.1.6.2 Connect the canister(s) to the manifold. Open the vent shut-off valve and the canister valve(s) to release any remaining pressure in the canister(s). Start the vacuum pump, close the vent shut-off valve, and then open the vacuum shut-off valve. Evacuate the canister(s) to <0.05 mm Hg and hold at this vacuum for at least one hour.

NOTE: On a daily basis or more often if necessary, purge the cryogenic traps with zero air to remove any trapped water from previous canister cleaning cycles.

3.1.6.3 Close the vacuum and vacuum/pressure gauge shut-off valves and open the zero air shut-off valve to pressurize the canister(s) with humid zero air to a gauge pressure of approximately 206 kPa (30 psig). If a zero gas generator system is used, the flow rate may need to be limited to maintain the zero air quality.

3.1.6.4 Close the zero air shut-off valve and allow the canister(s) to vent down to atmospheric pressure through the vent shut-off valve. Close the vent shut-off valve. Repeat this procedure two (2) additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.

3.1.6.5 At the end of the evacuation/pressurization cycle, re-evacuate to <0.05 mm Hg. The evacuated canister is now ready for a leak test and/or certification analyses.

3.1.7 Alternate Cleaning Procedure

3.1.7.1 An alternate canister cleaning system is shown in Figure D/VC-2 and the cleaning procedure is described below.

3.1.7.2 Initially, the system valve and manifold valves are in the closed position and the pump is on. Place canisters, which have been vented to atmospheric pressure in the oven, and attach to the

manifold. Close the canister valves. Bring the oven temperature to 100°C.

3.1.7.3 Place liquid nitrogen in the Dewar flask to immerse the cold trap and replenish as needed during the cleanup cycle. Open the system valve and evacuate the system lines to <0.05 mm Hg. If the system lines will not pump down, the trap may be blocked. To correct this, remove the cryogen and warm the trap with a heat gun while air is pulled through the lines into the pump.

NOTE: Care should be taken to always keep the system valve closed unless the cold trap is immersed in cryogen or air is being pulled through the lines to the pump. This will ensure that pump oil does not backstream into the line between the trap and canisters.

3.1.7.4 Once the system line is evacuated, open the manifold valve and evacuate the manifold tubing to <0.05 mm Hg. This will indicate whether or not the canister/manifold connections are leak-tight. Open the canister valves, and evacuate the canisters to <0.05 mm Hg and hold at this vacuum for a minimum of one hour. Close the canister valves, close the manifold valve, and disconnect the canisters from the manifold. Open the manifold valve slightly so that air is flowing to the pump, and then remove the cryogen from the trap. Allow the trap to warm to room temperature and close the system valve.

3.1.7.5 This cleaning method may be enhanced by adding a cycle in which the canisters are pressurized with humidified zero air and then re-evacuated to <0.05 mm Hg. The evacuated canister is now ready for a leak test and/or certification analyses.

3.2 LEAK TEST PROCEDURE

3.2.1 Summary

The canisters are tested to ensure that the valves and seams are leak-free. Leak testing involves a pressurized and an evacuated state.

3.2.2 Frequency

All canisters are leak tested prior to each sampling use.

3.2.3 Procedure

3.2.3.1 Test all canisters by pressurizing them to a gauge pressure of approximately 206 kPa (30 psig) with zero air. Measure and record the initial pressure and close the canister valve. After 24 hours, open the valve, and measure and record the pressure.

3.2.3.2 Evacuate each canister to <0.05 mm Hg, record the vacuum reading, close the canister valve, and setting the canister aside for 24 hours. After 24 hours, open the canister valve and record the vacuum.

3.2.4 **Technical Acceptance Criteria**

3.2.4.1 In the pressurized test, if leak-tight, the canister pressure should not decrease by more than 0.2 kPa (0.029 psi) over the 24-hour period.

3.2.4.2 The vacuum reading in the canisters in the evacuation tests should be within 0.02 mm Hg of the initial vacuum.

3.2.5 **Corrective Action**

Any significant change from the initial pressure or initial vacuum may indicate that compounds are off-gassing from the canister walls or that the valve or weld seam are not leak-tight. Any problem must be resolved after which the canister shall undergo a leak test and be subject to the same criteria for leak-tightness. If the results are still not within the limits, the canister must be set aside.

3.2.6 **Documentation**

Results of the leak tests shall be reported on Form VIII-AAVC.

3.3 **CANISTER CERTIFICATION**

3.3.1 **Summary**

Canister certification involves two procedures: blank analysis and spiked analysis. The purpose of the blank analysis is to determine the cleanliness of the canister, and the spiked analysis determines the condition of the interior of the canister as indicated by the stability of the spiked target compounds in the canister over a one week period.

3.3.2 **Frequency**

Initially, all canisters must be checked after cleaning to establish the percentage that pass the cleanliness criteria. A total of forty individual canisters should be analyzed in ten batches to check for individual canister contamination and for batch contamination by the cleaning apparatus. If and when only two or less individual canisters are contaminated and no batch contamination is evident, the laboratory may reduce the number of canisters tested for cleanliness after cleaning, but must continue to check 10 percent

of the canisters or one canister from each cleaning run, whichever is greater.

3.3.3 **Procedure**

3.3.3.1 Calibrate the GC/FID or the GC/MS system that has met all the tuning criteria, using a single injection of the 10 ppbv level of standard containing all the target compounds in Table D/VC-1 for volatile organics, prepared according to one of the calibration standard preparation procedures described in section 5.4.

3.3.3.2 For the blank (unspiked) analysis, analyze an aliquot of the contents of a clean canister, pressurized (206 kPa or 30 psig) with humid zero air following the preconcentration procedure in 5.12.3.

3.3.3.3 For the spiked analysis, the same canister used for the blank analysis or another clean canister may be used. Spike the canister with the target compounds then pressurize with humid zero air to 206 kPa or 30 psig. The canister should contain each target compound at a nominal level of 10 ppbv. Immediately analyze an aliquot of the canister contents following the preconcentration procedure in section 5.12.3.

3.3.3.4 Allow the spiked canister to sit at room temperature (ageing) for 7 days, then reanalyze the canister and compare the results with the results of the initial analysis.

3.3.3.5 After analysis, re-evacuate the unspiked canister to <0.05 mm Hg. If clean, the evacuated canister is now ready for storage and/or collection of air sample from the field.

3.3.3.6 The spiked canister must undergo the full canister cleaning cycles as a used canister prior to field use.

3.3.3.7 The certified canisters are considered clean for a period of one month after cleaning. If a canister is stored for a period of one month or more after being successfully cleaned, it must be recleaned by going through one cycle of pressurization and evacuation; however, no subsequent analytical confirmation of cleanliness is required before using.

3.3.4 **Calculation**

3.3.4.1 For the blank analysis, target compound concentrations are determined using the external standard quantitation method. Use the following equation to determine target compound concentration levels:

$$Concentration,\ ppbv = \frac{(A_x)\,(C_s)\,(V_s)}{(A_s)\,(V_x)} \qquad \text{Eq. D/VC-1}$$

where: A_x = peak response of target analyte in the sample aliquot;
A_s = peak response of target analyte in the calibration standard;
V_x = volume of sample aliquot injected, cm^3;
V_s = volume of calibration standard aliquot injected, cm^3; and
C_s = concentration of target analyte in the calibration standard, ppbv.

3.3.4.2 For the spiked analysis, calculate the percent difference (%D) for each target compound between the peak responses at the initial day and the seventh day using the following equation:

$$\%D = \frac{A_0 - A_7}{A_0} \times 100 \qquad \text{Eq. D/VC-2}$$

where: A_0 = peak response of target analyte at initial analysis; and
A_7 = peak response of target analyte after 7 days.

3.3.4.3 For non-target compounds, use the peak response of the target analyte in the calibration standard with the closest retention time to that of the non-target analyte peak in the sample.

3.3.5 Technical acceptance Criteria

3.3.5.1 No target compound shall be present in the unspiked canister at a level higher than its CRQL. The total concentration of compounds in the canister (target and non-target) shall not exceed 10 ppbv.

3.3.5.2 For the spiked canister, the acceptable percent difference for any target compound at a nominal 10 ppbv concentration in humidified zero air is ±30 percent.

3.3.6 Corrective Action

3.3.6.1 Any unspiked canister with a target analyte concentration greater than the its CRQL or a total concentration greater than 10 ppbv (target and non-target) shall be recleaned and reanalyzed subject to the same criteria for cleanliness, or be set aside.

3.3.6.2 For the spiked analysis, a percent difference greater than ±30 percent indicates a problem with the canister, e.g., leak at seams and active sites inside the canister. Any problem must be resolved after which the canister shall undergo a spiked analysis and be subject to the same criteria for cleanliness. If after reanalysis, the percent difference is still not within the limits, the

canister must be set aside.

3.3.7 Documentation

Results of the certification of the canisters shall be reported on Form VIII-AAVC.

4 OPTIONAL GC/FID SCREENING OF SAMPLES IN CANISTERS

4.1 SUMMARY

The air sample is screened by GC/FID to determine the approximate range of concentrations of VOCs in the sample and to determine whether a dilution of the sample will be required prior to GC/MS analysis. Since FID identifications are based on retention time alone, an FID screening analysis is useful only in establishing tentative identifications and concentrations of the components in a sample, and in determining whether a dilution of the sample will be required prior to GC/MS analysis.

4.2 APPARATUS AND MATERIALS

4.2.1 Gas chromatograph: An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including analytical columns, gases, flame ionization detector, and integrator. A data system is recommended for measuring peak areas. Sub-ambient oven control and oven temperature programming are required.

4.2.2 Chromatographic column: 30 m x 0.53 mm fused silica column such as DB-1, DB-5, or DB-624, or equivalent.

NOTE: The wider Megabore® column is less susceptible to plugging as a result of trapped water, thus reducing the need for a Nafion® dryer in the analytical system. However, greater retention time variability has been observed when the Nafion® dryer is not used to remove water vapor from the sample stream, and the analyst, therefore, must be careful to consider any retention time shifts when identifications are made. The Megabore® column has sample capacity approaching that of a packed column, while retaining much of the peak resolution traits of narrower columns. GC operating parameters are to be optimized by the analyst.

4.2.3 Six-port gas chromatographic valve.

4.2.4 Cryogenic trap with temperature control assembly.

4.2.5 Electronic mass flow controllers: Capable of maintaining a constant flow (for carrier gas, sample gas and canister sample flow) and to provide analog output to monitor flow anomalies (two 0-100 cm^3/min units for air and one 0-10 cm^3/min unit for helium, or equivalent).

4.2.6 Vacuum pump: General purpose laboratory pump, capable of reducing the downstream pressure of the flow controller to provide the minimum pressure differential necessary to maintain controlled flow rates.

4.2.7 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings.

4.2.8 Vacuum/pressure gauges: Stainless steel gauges, capable of measuring vacuum and pressure.

4.2.9 Stainless steel cylinder pressure regulators: Standard, two-stage cylinder regulators with pressure gauges for helium, air, nitrogen, and hydrogen gas cylinders.

4.2.10 Gas purifiers: Used to remove organic impurities and moisture from gas streams.

4.2.11 Calibration system and manifold described in section 5.2.3.

4.3 **REAGENTS**

4.3.1 Gases: Cylinders of helium, hydrogen, nitrogen, and air, ultrahigh purity grade.

4.3.2 Liquid nitrogen: For cooling GC oven and cryogenic trap.

4.3.3 Deionized water: HPLC grade, for humidifying gas streams.

4.4 **PROCEDURE**

4.4.1 Prepare working calibration standards as described in section 5.4, and calibrate the GC at three of the five concentrations (2, 10, and 20 ppbv) to determine the instrument linearity and sensitivity. Once the FID linearity range has been established, the GC/FID may be calibrated using a single-point (10 ppbv) calibration during each 12-hour period.

4.4.2 Analyze the sample with the procedure described in section 5.12.3. Use the retention times of the target compounds in the calibration standard to tentatively identify target compounds in the sample.

4.4.3 Based on the concentrations of tentatively identified target compounds in the sample, determine whether the sample must be diluted or whether the target compounds are within the range of the GC/MS calibration.

4.4.4 If target compounds are all within the range of the GC/MS calibration, proceed to Section 5.

4.4.5 If target compounds are present at concentrations higher than the calibration range of the GC/MS, then the sample must be diluted as described in the following section prior to GC/MS analysis.

4.5 **DILUTION**

The GC/FID screening procedure, if used, will have shown the approximate concentrations of sample components. If a dilution of the

sample was indicated, first determine the dilution factor needed to bring the concentration into the upper half of the GC/MS calibration, then use one of the following procedures to dilute the sample.

4.5.1 Estimate the final pressure of the canister needed to achieve the dilution. Pressurize the canister with humid zero air which has been demonstrated to be free of contaminants. Measure the final canister pressure and calculate the actual dilution factor using the following equation:

$$DF = \frac{Y_a}{X_a} \qquad \text{Eq. D/VC-3}$$

where: DF = dilution factor;
X_a = canister pressure absolute before dilution; and
Y_a = canister pressure absolute after dilution.

NOTE: (X_a and Y_a must be in same units).

4.5.2 Using a gastight syringe, transfer an aliquot of sample from the original canister to a clean, evacuated canister. Pressurize the dilution canister with humid zero air which has been demonstrated to be free of contaminants. Calculate the dilution factor from the volume of original sample injected and the volume of dilution gas which was added using the following equation.

$$DF = \frac{Volume_{original\ sample}}{Volume_{dilution\ gas}} \qquad \text{Eq. D/VC-4}$$

5 GC/MS ANALYSIS OF VOLATILES FROM CANISTERS

5.1 SUMMARY

In this analytical procedure, a whole air sample is passed through a preconcentrator where the VOCs are condensed on a reduced temperature surface (cold trap). Subsequently the condensed gases are thermally desorbed and backflushed from the trap with an inert gas onto a gas chromatographic column. The preconcentration technique discussed here is similar to that discussed in EPA Compendium Method TO-14, "The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMA® Passivated Canister Sampling and Gas Chromatographic Analysis". Other preconcentration techniques (e.g, those using solid sorbents) may be employed if equivalency is established for the analysis procedure of which it is a part. The gas chromatograph (GC) is temperature-programmed to separate the individual VOCs, which are then detected with a mass spectrometer operated in a full scan mode. The steps of the analytical method are diagrammed in Figure D/VC-3.

5.2 APPARATUS AND MATERIALS

5.2.1 Sampling/Preconcentrator system

5.2.1.1 Electronic mass flow controllers: used to maintain constant flow (for carrier gas and sample gas) and to provide an analog output to monitor flow anomalies.

5.2.1.2 Vacuum pump: general purpose laboratory pump, capable of reducing the downstream pressure of the flow controller to provide the minimum pressure differential necessary to maintain controlled flow rates.

5.2.1.3 Stainless steel tubing and stainless steel fittings: chromatographic grade.

5.2.1.4 Stainless steel cylinder pressure regulators: standard, two-stage cylinder regulators with pressure gauges.

5.2.1.5 Gas purifiers: used to remove organic impurities and moisture from gas streams.

5.2.1.6 Nafion® dryer (optional): consisting of Nafion® tubing coaxially mounted within larger tubing, used to remove water vapor from the sample stream if the MS in use does not have sufficient pumping capacity to handle water vapor.

5.2.1.7 Six-port gas chromatographic valve: for routing sample and carrier gas flows.

5.2.1.8 Cryogenic preconcentrator: complete units are commercially available from several sources.

5.2.1.8.1 The cooling unit is comprised of a 0.32 cm outside diameter (O.D.) nickel tubing loop packed with 60-80 mesh Pyrex beads with glass wool plugs at each end. The nickel tubing loop is wound onto a cylindrically formed tube heater (250 watt). A cartridge heater (25 watt) is sandwiched between pieces of aluminum plate at the trap inlet and outlet to provide additional heat to eliminate cold spots in the transfer tubing.

NOTE: During operation, the trap is inside a two-section stainless steel shell which is well insulated. Rapid heating (-150 to +100°C in 55 sec.) is accomplished by direct thermal contact between the heater and the trap tubing. Cooling is achieved by vaporization of the cryogen. In the shell, efficient cooling (+120 to -150°C in 225 sec.) is facilitated by confining the vaporized cryogen to the small open volume surrounding the trap assembly.

5.2.1.8.2 The trap assembly and chromatographic valve are mounted on a baseplate fitted into the injection and auxiliary zones of the GC on an insulated pad directly above the column oven.

NOTE: Alternative trap assembly and connection to the GC may be used depending upon user's requirements.

5.2.1.8.3 The carrier gas line is connected to the injection end of the analytical column with a zero-dead-volume fitting that is usually held in the heated zone above the GC oven. An aluminum box is fitted over the sample handling elements to complete the package. Vaporized cryogen is vented through the top of the box.

5.2.2 **GC/MS System**

5.2.2.1 Gas chromatograph: The gas chromatographic (GC) system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout desorption and temperature program operations. The system must include or be interfaced to a preconcentrator (see above) and have all required accessories including analytical columns and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with rubber components are not to be used. The column oven must be cooled to -50°C at the start of the gas chromatographic run; therefore, a sub-ambient oven temperature controller is required.

5.2.2.2 Chromatographic columns: 100% methyl silicone or 5% phenyl, 95% methyl silicone capillary columns of 0.25 to 0.53 mm I.D. x 50 m length, or equivalent, are required to provide separation of the target compounds.

5.2.2.3 Mass spectrometer: Capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng of p-bromofluorobenzene (BFB) is analyzed.

NOTE: BFB criteria must be met before any samples are analyzed. Any samples analyzed when BFB criteria have not been met will require reanalysis at no cost to the Agency. To ensure sufficient precision of mass spectral data, the MS scan rate should allow acquisition of at least five spectra while a sample compound elutes from the GC. The preconcentration GC/MS system must be in a room whose atmosphere is demonstrated to be free of all potential contaminants which will interfere with the analysis. The instrument must be vented to the outside of the facility or to a trapping system which prevents the release of contaminants into the instrument room.

5.2.2.4 GC/MS interface: Any gas chromatograph to mass spectrometer interface, that gives acceptable calibration points for each of the parameters of interest and achieves all acceptable performance criteria, may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass should be deactivated and this can be accomplished by silanizing with dichlorodimethylsilane.

5.2.2.5 Data system: The computer system that is interfaced to the mass spectrometer must allow the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as a Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The 1990 (or most recent) release of the NIST Library shall be used as the reference library.

5.2.2.6 Magnetic tape storage device: Capable of recording data and must be suitable for long-term, off-line storage.

5.2.3 Calibration System and Manifold

NOTE: The following calibration system is based on EPA Compendium Method TO-14 which discusses gaseous calibration standards. Other calibration methods such as those used in purge and trap procedures may be used if, through such use, the analytical procedure is not compromised with respect to the performance specifications in of the analytical system.

5.2.3.1 Calibration manifold: Glass or high purity quartz manifold (1.25 cm I.D. x 66 cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing. The manifold should be heated to 50°C.

5.2.3.2 Humidifier: 500-mL impinger flask containing HPLC grade deionized water.

5.2.3.3 Electronic mass flow controllers: One 0 to 5 L/min unit and one or more 0 to 10 cm^3/min units for air, depending on number of cylinders in use for calibration.

5.2.3.4 Teflon filter(s): 47 mm Teflon filter for particulate control, best source.

5.2.3.5 Gastight syringes: For injecting internal standards mixtures.

5.3 **REAGENTS**

5.3.1 Neat standards or manufacturer-certified solutions/mixtures.

5.3.2 Helium and air: Ultrahigh purity grade in gas cylinders.

5.3.3 Liquid nitrogen.

5.3.4 Deionized water: HPLC grade, ultrahigh purity (for humidifier).

5.4 **STANDARDS**

5.4.1 The Contractor must provide all standards to be used with this contract. The Contractor must be able to verify that the standards are certified traceable to a NIST Standard Reference Material (SRM) or to a NIST/EPA approved Certified Reference Material (CRM). Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

5.4.2 Neat standards must have a purity of 98 percent or greater. The weight may be used without correction to calculate the concentration of the stock solution.

5.4.3 Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source, or traceable to EPA-supplied standards. Fresh stock standards must be prepared once every twelve months, or sooner, if standards have degraded or concentrated. Stock standards must be checked for signs of degradation or concentration just prior to preparing secondary dilution and working standards from them.

5.4.4 **Gas calibration stock standards**

Cylinder(s) containing approximately 10 ppmv of each of the target compounds are used as primary stock standards. The components may be purchased in one cylinder or in separate cylinders. Refer to manufacturer's specifications for guidance on purchasing and mixing VOCs in gas cylinders.

5.4.5 **Working Standards**

5.4.5.1 **Instrument Performance Check Standard**

Prepare a standard solution of BFB in humidified zero air at a concentration which will allow collection of 50 ng of BFB under the optimized preconcentration parameters.

5.4.5.2 **Calibration Standards**

Prepare five working calibration standards in humidified zero air at a concentration which will allow collection at the 2, 5, 10, 20, and 50 ppbv level for each component under the optimized preconcentration parameters.

5.4.5.3 **Internal Standard Spiking Mixture**

Prepare an internal standard spiking mixture containing bromochloromethane, chlorobenzene-d_5, and 1,4-difluorobenzene at 10 ppmv each in humidified zero air to be added to the sample or calibration standard. 500 μL of this solution spiked into 500 mL of sample will result in a concentration of 10 ppbv. The internal standard is introduced into the trap during the collection time for all calibration, blank, and sample (field and QC) analyses. The volume of internal standard spiking mixture spiked for each analysis must be the same from run to run.

5.4.6 **Standard Preparation by Dynamic Dilution Technique**

5.4.6.1 Standards may be prepared by dynamic dilution of the gaseous standards in the cylinder(s) with humidified zero air using mass flow controllers and a calibration manifold. The working standard may be delivered from the manifold to a clean, evacuated canister using a

pump and mass flow controller.

5.4.6.2 Alternatively, the analytical system may be calibrated by sampling directly from the manifold if the flow rates are optimized to provide the desired amount of calibration standards. However, the use of the canister as a reservoir prior to introduction into the preconcentration system resembles the similar procedure as in the sample analysis and is preferred. Flow rates of the dilution air and cylinder standards (all expressed in the same units) are measured using a bubble meter or calibrated electronic flow measuring device, and the concentrations of target compounds in the manifold are then calculated using the dilution ratio and the original concentration of each compound.

$$Manifold\ Conc. = \frac{Original\ Conc. \times Std.\ Gas\ Flowrate}{Air\ Flowrate + Std.\ Gas\ Flowrate} \quad \text{Eq. D/VC-5}$$

5.4.6.3 Example of 1 cm^3/min flow of 10 ppmv standard diluted with 1,000 cm^3/min of humid air provides a nominal 10 ppbv mixture, as calculated below:

$$Manifold\ Conc. = \frac{(10 \times 10^{-6})(1\ cm^3/min)}{(1000\ cm^3/min) + (1\ cm^3/min)} = 10\ ppb \quad \text{Eq. D/VC-6}$$

5.4.7 Standard Preparation by Static Dilution Bottle Technique

Standards may be prepared in canisters by spiking the canister with a mixture of components prepared in a static dilution bottle. This technique is used specifically for liquid standards. A Standard Operating Procedure for the preparation of static dilution bottles is available from the Atmospheric Research and Exposure Assessment Laboratory, Office of Research and Development, USEPA, Research Triangle Park, NC, 27711, Document AREAL/RTP-SOP-MRDD-036. A summarized version of the SOP is provided below.

5.4.7.1 Determine the approximate volume of a clean 2-liter round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, by weighing the amount of water required to completely fill up the flask. Assuming a specific gravity for the water of 1, the weight of the water in grams is taken as the volume of the flask in milliliters.

5.4.7.2 Flush the flask with helium for by attaching a tubing onto the glass neck to deliver the helium. After a few minutes, remove the tubing and immediately close the glass neck with a Mininert septum cap.

5.4.7.3 Place the flask in a 60°C oven and allow to equilibrate at that temperature for about 15 minutes. Inject, with a gastight

syringe, predetermined aliquots of liquid standards into the flask making sure to keep the flask temperature constant at 60°C.

5.4.7.4 Before withdrawing aliquots from the flask, allow the contents to equilibrate in the oven for at least 30 minutes. Syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots to avoid condensation.

5.4.7.5 Aliquots may then be taken for introduction into the cryofocusing trap. An aliquot or aliquots totaling greater than 1 percent of the flask volume should be avoided.

5.4.7.6 Standards prepared by this method as stable for one week. The septum must be replaced with each freshly prepared standard.

5.4.7.7 Calculate the concentration of each component in the flask using the following equation:

$$Concentration,\ mg/L = \frac{(V_a)(d)}{V_f} \qquad \text{Eq. D/VC-7}$$

where: V_a = volume of liquid neat standard injected into the flask in μL;
d = density of the liquid neat standard in $mg/\mu L$; and
V_f = volume of the flask in liters.

5.4.7.8 To obtain concentrations in ppbv, the approximate volume at the vaporized phase can be determined using the equation given in section 5.4.8.7.

CAUTION: In the preparation of standards by this technique, the analyst should make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask. Precautions should also be taken to avoid a significant decrease in pressure inside the flask after withdrawal of aliquot(s).

5.4.8 Standard Preparation Procedure in High Pressure Cylinders

Standards may be prepared in high pressure cylinders as described in the procedure of Pollack and Holdren (1990)[1]. A modified summary of the procedure is provided below.

[1] A.J. Pollack, M.W. Holdren, "Multi-Adsorbent Preconcentration and Gas Chromatographic Analysis of Air Toxics with an Automated Collection/Analytical System," in the Proceedings of the 1990 EPA/A&WMA International Symposium on Measurement of Toxic and Related Air Pollutants, USEPA Report Number EPA/600/9-90/026, pp. 209-218.

5.4.8.1 Obtain the standard compounds as gases or neat liquids (greater than 98 percent purity).

5.4.8.2 Flush a 17.3 m^3 Teflon-lined aluminum cylinder with high-purity nitrogen gas and then evacuate.

5.4.8.3 Accurately measure predetermined amounts of each neat standard compound using a gastight syringe and inject into the cylinder.

5.4.8.4 Pressurize the cylinder to 1200 psig with humid zero air. The flow rate of the zero air should be kept at a constant in order to determine the total volume of diluent used by multiplying the flow rate by the delivery time.

5.4.8.5 Allow the contents of the cylinder to equilibrate prior to withdrawal of aliquots into the GC system.

5.4.8.6 Calculate the concentrations using the following equation:

$$Concentration,\ ppbv = \frac{Volume_{standard}}{Volume_{dilution\ gas}} \qquad \text{Eq. D/VC-8}$$

(Both values must be expressed in the same units.)

5.4.8.7 If the neat standard is a liquid, the approximate volume at the vaporized phase can be determined using the following equations:

$$V = \frac{nRT}{P} \qquad \text{Eq. D/VC-9}$$

and

$$n = \frac{(uL)(p)}{MW} \qquad \text{Eq. D/VC-10}$$

where:
- V = volume at gaseous state in liters;
- n = moles;
- R = gas constant in (0.08206 L-atm/mole °K);
- T = 273°K (standard temperature);
- P = 1 atm (standard pressure);
- μL = volume of liquid neat standard in microliters;
- p = specific gravity of the neat standard; and
- MW = molecular weight of the neat standard expressed in mg/mole.

NOTE: For the above compounds, the calculated cylinder concentrations are within ±20 percent of the 200 ppbv nominal targeted values. For lower concentrations, an aliquot of this

solution should be diluted according to one of the methods described under section 4.5.

5.4.9 **Standard Preparation by Water Methods**

Standards may be prepared by a water purge and trap method described in the procedure by Stephenson, Allen, and Slagle (1990)[2] and summarized below.

5.4.9.1 Pressurize a previously cleaned canister still under vacuum to 760 mm Hg absolute (1 atm) with zero grade air.

5.4.9.2 Remove the air gauge from the canister and connect the sparging vessel to the canister with the shortest length of 1/16 in. stainless steel tubing possible. (Extra effort should be made to minimize possible areas of dead volume to maximize transfer of analytes from the water to the canister.)

5.4.9.3 Spike 5 mL of water with the stock standard solution and the internal standard solution.

5.4.9.4 Transfer this water into the sparge vessel and purge the water with nitrogen for 10 mins at 100 cm^3/min while being heated at 40°C.

5.4.9.5 At the end of 10 mins, remove the sparge vessel from the canister, re-install the air gauge, and pressurize the canister with pure nitrogen to 1500 mm Hg absolute (approximately 15 psi).

5.4.9.6 Allow the canister to equilibrate overnight before use.

5.4.10 **Storage of Standards**

Working standards and internal standards prepared in canisters may be stored for thirty days in an atmosphere free of potential contaminants.

5.5 INSTRUMENT OPERATING CONDITIONS

5.5.1 **Preconcentrator**

The following are suggested cryogenic preconcentrator analytical conditions which may be optimized by the operator.

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2 J.H.M. Stephenson, F. Allen, T. Slagle, "Analysis of Volatile Organics in Air via Water Methods," in Proceedings of the 1990 EPA/A&WMA International Symposium on Measurement of Toxic and Related Air Pollutants, USEPA Report Number EPA/600/9-90/026, pp. 194-199.

5.5.1.1 Sample Collection Conditions

The cryogenic trap is at a setpoint from -150 to -170°C. A 500-cm^3 sample of whole air is passed through the trap during the sample collection period.

NOTE: The analyst should optimize the flow rate, duration of sampling, and absolute sample volume to be used. Other preconcentration systems may be used provided performance standards are realized.

5.5.1.2 Desorption Conditions

Desorb Temperature:	120°C
Desorb Flow Rate:	4 cm^3/min helium
Desorb Time:	<60 sec

5.5.1.3 Trap Reconditioning Conditions

Before initial use, condition the trap overnight at 120°C by flushing with 10 cm^3/min of inert gas. Vent the trap effluent to the room and not to the analytical column. Prior to daily use, condition the trap by heating at 120°C for 30 minutes while flushing with inert gas. The trap may be vented to the analytical column during daily conditioning.

Reconditioning Temperature:	120°C
Reconditioning Time:	30 min

5.5.2 GC/MS System

5.5.2.1 Optimize GC conditions for compound separation and sensitivity. Baseline separation of benzene and carbon tetrachloride is an indication of optimum chromatographic performance.

5.5.2.2 The following are the recommended gas chromatographic analytical conditions:

Carrier Gas:	Helium	
Flow Rate:	4 cm^3/min	
Temperature Program:	Initial Temperature:	-50°C
	Initial Hold Time:	2 min
	Ramp Rate:	8° C/min
	Final Temperature:	150°C
	Final Hold Time:	Until all target compounds elute.

5.5.2.3 The following are the required mass spectrometer conditions:

Electron Energy: 70 Volts (nominal)
Mass Range: 35-300 amu
Scan Time: To give at least 5 scans per peak, not to exceed 1 second per scan.

5.6 ANALYTICAL SEQUENCE

The GC/MS analytical sequence for each 12-hour time period shall be as follows:

- Instrument Performance Check (BFB)
- Initial or continuing calibration
- Laboratory Method Blank
- LCS
- ≤20 field samples
- Performance Evaluation (PE) Sample (if available)

5.7 INSTRUMENT PERFORMANCE CHECK

5.7.1 Summary

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. The GC/MS system is set up according to the manufacturer's specifications, and the mass calibration and resolution of the GC/MS system are then verified by the analysis of the instrument performance check standard, bromofluorobenzene (BFB).

5.7.2 Frequency

5.7.2.1 Prior to the analyses of any samples, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard containing BFB. The instrument performance check solution must be analyzed initially and once per 12-hour time period of operation. Also, whenever the laboratory takes corrective action which may change or affect the mass spectral criteria (e.g., ion source cleaning or repair, column replacement, etc.), the instrument performance check must be verified irrespective of the 12-hour laboratory requirement.

5.7.2.2 The 12-hour time period for GC/MS instrument performance check and standards calibration (initial calibration and continuing calibration check criteria) begins at the injection of the BFB which the laboratory submits as documentation of a compliance tune. The time period ends after 12 hours have elapsed. In order to meet instrument performance check requirements, samples, blanks, and standards must be injected within 12 hours of the BFB injection.

5.7.3 Procedure

5.7.3.1 The analysis of the instrument performance check standard is performed by trapping 50 ng of BFB under the optimized preconcentration parameters. The BFB is introduced from a cylinder into the GC/MS via a sample loop valve injection system similar to that in the sample analysis section.

5.7.3.2 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of BFB.

NOTE: All instrument conditions must be identical to those used in the sample analysis.

5.7.4 Technical Acceptance Criteria

5.7.4.1 Prior to the analysis of any samples, blanks, or calibration standards, the Laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution as specified in Table D/VC-2.

5.7.4.2 The instrument performance check standard must be injected once at the beginning of each 12-hour period during which samples, blanks, or standards are analyzed.

5.7.5 Corrective Action

5.7.5.1 If the BFB acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other necessary actions to achieve the acceptance criteria.

5.7.5.2 BFB acceptance criteria MUST be met before any standards, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when tuning criteria have not been met will require reanalysis at no additional cost to the Agency.

5.7.6 Documentation

Reporting requirements are listed in Exhibit B. Results of the BFB tuning are reported on Form IV-AAVC.

5.8 INITIAL CALIBRATION

5.8.1 Summary

5.8.1.1 Prior to the analysis of samples and required blanks and after the instrument performance check standard criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations in an initial calibration sequence to determine instrument sensitivity and the linearity of GC/MS response for the target compounds.

5.8.1.2 All sample results, for sample analyses performed in the same 12-hour sequence as the initial calibration, are quantified against the initial calibration standard that is the same concentration as the continuing calibration standard (10 ppbv).

5.8.2 Frequency

5.8.2.1 Each GC/MS system must be calibrated upon award of the contract, whenever the Contractor takes corrective action which may change or affect the initial calibration criteria (e.g., ion source cleaning or repair, column replacement, etc.), or if the daily calibration acceptance criteria have not been met.

5.8.2.2 If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed.

5.8.2.3 If time does not remain in the 12-hour period after meeting the acceptance criteria for the initial calibration, a new analytical sequence shall commence with the analysis of the instrument performance standard.

5.8.3 Procedure

5.8.3.1 Verify that the GC/MS system meets the instrument performance criteria in section 5.7.

5.8.3.2 The GC must be operated using temperature and flow rate parameters equivalent to those in section 5.5. Calibrate the preconcentration-GC/MS system by drawing 500 cm^3 of standard into the system following one of the methods described under section 5.4. Add the equivalent of 10 ppbv of each internal standard into the trap.

5.8.4 Calculations

NOTE: In the following calculations, the area response is that of the primary quantitation ion unless otherwise stated.

5.8.4.1 Relative Response Factor: Calculate the relative response factors (RRF) for each target compound to the appropriate internal standard (see Table D/VC-3) using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \qquad \text{Eq. D/VC-11}$$

where: RRF = relative response factor;
A_x = area of the primary ion for the compound to be measured;
A_{is} = area of the primary ion for the internal standard;
C_{is} = concentration of internal standard spiking mixture, ppbv; and
C_x = concentration of the compound in the calibration standard, ppbv.

NOTE: The equation above is valid under the condition that the volume (500 μL) of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume (500 cm^3) of field and QC sample introduced into the trap is the same for each analysis. C_{is} and C_x must be in the same units.

5.8.4.2 Mean Relative Response Factor: Calculate the mean RRF ($\overline{RRF}$) for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n} \qquad \text{Eq. D/VC-12}$$

where: $\overline{RRF}$ = mean relative response factor;
x_i = RRF of the compound; and
n = number of values.

5.8.4.3 Percent Relative Standard Deviation (%RSD): Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100 \qquad \text{Eq. D/VC-13}$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(RRF_i - \overline{RRF})^2}{N - 1}} \qquad \text{Eq. D/VC-14}$$

where: SD_{RRF} = standard deviation of initial response factors (per compound);
RRF_i = relative response factor at a concentration level; and
$\overline{RRF}$ = mean of initial relative response factors (per compound).

5.8.4.4 Relative Retention Times (RRT): Calculate the RRTs for each target compound over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{IS}} \qquad \text{Eq. D/VC-15}$$

where: RT_c = retention time of the target compound; and
RT_{IS} = retention time of the internal standard.

5.8.4.5 Mean of the Relative Retention Times ($\overline{RRT}$): Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target compound over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT}{n} \qquad \text{Eq. D/VC-16}$$

where: $\overline{RRT}$ = mean relative retention time for the target compound for each initial calibration standard; and
RRT = relative retention time for the target compound at each calibration level.

5.8.4.6 Tabulate the area response (Y) of the primary ion (see Table D/VC-3) and the corresponding concentration for each compound and internal standard.

5.8.4.7 Mean Area Response ($\overline{Y}$) for Internal Standard: Calculate the mean area response ($\overline{Y}$) for each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n} \qquad \text{Eq. D/VC-17}$$

where: $\overline{Y}$ = mean area response; and
Y = area response for the primary quantitation ion for the internal standard for each initial calibration standard.

5.8.4.8 Percent Area Response Change (%ARC): Calculate the %ARC at each calibration level for each of the internal standards using the following equation:

$$\%ARC = \frac{A_x - \bar{Y}}{\bar{Y}} \times 100 \qquad \text{Eq. D/VC-18}$$

where: %ARC = percent area response change;
A_x = area response of the internal standard at a concentration level; and
$\bar{Y}$ = mean area response of the internal standard over the entire calibration range.

5.8.4.9 Mean of the Retention Times ($\overline{RT}$) For Internal Standard: Calculate the mean of the retention times ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n} \qquad \text{Eq. D/VC-19}$$

where: $\overline{RT}$ = mean retention time; and
RT = retention time for the internal standard for each initial calibration standard.

5.8.4.10 Internal Standard Retention Time Shift (RTS): Calculate the RTS between the RT of each internal standard at each concentration level and the $\overline{RT}$ for that internal standard over the entire calibration range using the following equation:

$$RTS = \overline{RT_i} - RT_x \qquad \text{Eq. D/VC-20}$$

where: $\overline{RT_i}$ = mean of the retention time for the internal standard in the initial calibration; and
RT_x = retention time of the internal standard at a concentration level.

5.8.5 Technical Acceptance Criteria

5.8.5.1 All initial calibration standards must be analyzed at the concentration levels and frequency described in this section on a GC/MS system meeting the BFB instrument performance check criteria.

5.8.5.2 The %RSD for all target compounds in the initial curve must be less than or equal to 30 percent. Up to two compounds may exceed the maximum %RSD criteria; the %RSD for those compounds, however, must not exceed 40 percent.

5.8.5.3 The RRT for each of the target compounds at each calibration level must be within ±0.06 RRT units of the mean relative retention time ($\overline{RRT}$) for the compound.

5.8.5.4 The %ARC at each calibration level must be within ±40

percent of the mean area response ($\bar{Y}$) over the initial calibration range for each internal standard.

5.8.5.5 The retention time shift for each of the internal standards at each calibration level must be within ±20.0 seconds compared to the mean retention time ($\overline{RT}$) over the initial calibration range for each internal standard.

5.8.6 Corrective Action

5.8.6.1 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the initial calibration technical acceptance criteria.

5.8.6.2 Initial calibration acceptance criteria MUST be met before any field samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when initial calibration criteria have not been met will require reanalysis at no additional cost to the Agency.

5.8.7 Documentation

Reporting requirements are listed in Exhibit B. Results of the initial calibration are reported on Form V-AAVC; Internal standard area and RT shall be tabulated on Form VII-AAVC.

5.9 CONTINUING CALIBRATION

5.9.1 Summary

Prior to the analysis of samples and required blanks and after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a continuing calibration standard to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the method. The continuing calibration standard, which is the 10 ppbv level calibration standard, shall contain all the target compounds and internal standards.

5.9.2 Frequency

5.9.2.1 A check of the calibration curve must be performed once every 12 hours on a GC/MS system that has met the tuning criteria.

5.9.2.2 The continuing calibration sequence starts with the injection of the BFB. If the BFB analysis meets the ion abundance criteria for BFB, then a continuing calibration standard may be analyzed.

5.9.3 Procedure

Analyze the mid level standard (10 ppbv) in a GC/MS system that has met the tuning and mass calibration criteria following the same procedure in section 5.8.3 .

5.9.4 Calculations

NOTE: In the following calculations, the area response is that of the primary quantitation ion unless otherwise stated.

5.9.4.1 Relative Response Factor (RRF): Calculate a relative response factor (RRF) for each target compound using the equation in section 5.8.4.1.

5.9.4.2 Percent Difference (%D): Calculate the percent difference in the RRF of the daily RRF (12-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100 \qquad \text{Eq. D/VC-21}$$

where: RRF_c = RRF of the compound in the continuing calibration standard; and

$\overline{RRF_i}$ = mean RRF of the compound in the most recent initial calibration.

5.9.5 Technical Acceptance Criteria

5.9.5.1 The continuing calibration standard must be analyzed at the concentration level and frequency described in this section on a GC/MS system meeting the BFB instrument performance check criteria.

5.9.5.2 The %D for each target compound in a daily calibration sequence must be within ±30 percent in order to to proceed with the analysis of samples and blanks.

5.9.6 Corrective Action

5.9.6.1 If the continuing calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the continuing calibration technical acceptance criteria.

5.9.6.2 Continuing calibration acceptance criteria MUST be met before any field samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when the continuing calibration criteria have not been met

will require reanalysis at no additional cost to the Agency.

5.9.7 **Documentation**

Reporting requirements are listed in Exhibit B. Results of the continuing calibration are reported on Form VI-AAVC; Internal standard area and RT shall be tabulated on Form VII-AAVC.

5.10 BLANK ANALYSIS

5.10.1 Summary

5.10.1.1 To monitor for possible laboratory contamination, laboratory method blanks are analyzed with each SDG at least once in a 12-hour analytical sequence. All steps in the analytical procedure are performed on the blank using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

5.10.1.2 A laboratory method blank is an unused, certified canister that has not left the laboratory. The blank canister is pressurized with zero air and carried through the same analytical procedure as a field sample. The injected aliquot of the blank must contain the same amount of internal standards that are added to each sample. All field samples must be analyzed with associated blanks.

5.10.2 Frequency

5.10.2.1 The laboratory method blank must be analyzed after the calibration standard(s) and before any samples are analyzed. A LMB shall be analyzed immediately before the LCS along with each batch of ≤20 samples and shall be carried through the entire analytical procedure.

5.10.2.2 Whenever an unusually concentrated sample is encountered, a LMB analysis shall be performed immediately after the sample analysis.

5.10.3 Procedure

5.10.3.1 Determine the average pressure of all the sample canisters in the SDG. Pressurize the blank canisters, which are evacuated to <0.05 mm Hg at the end of the canister certification procedure, to the average pressure of the sample canisters in the SDG (approximately 130 kPa) with humidified zero air.

5.10.3.2 Analyze the blanks following the same procedure outlined under section 5.12, "Sample Analysis".

5.10.4 Calculations

The blanks are analyzed similar to a field sample and the equations in section 5.12.4 apply.

5.10.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the blank are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.

5.10.5.1 All blanks must be analyzed at the frequency described in section 5.10.2 on a GC/MS system meeting the BFB instrument performance check and initial calibration or continuing calibration technical acceptance criteria.

5.10.5.2 The area response for each IS in the blank must be within ±40 percent of area response of the IS in the most recent valid calibration.

5.10.5.3 The retention time for each of the internal standards must be within ±20.0 seconds between the blank and the most recent valid calibration.

5.10.5.4 The blank must not contain any target analyte at a concentration greater than its CRQL and must not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte at its CRQL. The total level of analytes in the blank must not exceed 10 ppbv.

5.10.6 Corrective Action

5.10.6.1 If a Contractor's blanks do not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds.

5.10.6.2 All samples processed with a blank that is out of control (i.e., contaminated) shall be flagged with a "B".

5.10.7 Documentation

Reporting requirements are listed in Exhibit B. Laboratory method blank results are reported on Form II-AAVC, Laboratory Blank Summary Form. Internal standard area and RT shall be tabulated on Form VII-AAVC.

5.11 LABORATORY CONTROL SAMPLES (LCS)

5.11.1 Summary

The LCS is an internal laboratory quality control sample designed to assess (on an SDG-by-SDG basis) the capability of the Contractor to perform the analytical method listed in this Exhibit. Table D/VC-7 provides a list of LCS compounds and the corresponding percent recovery limits.

5.11.2 Frequency

The LCS must be analyzed and reported once per 12-hour analytical sequence, and concurrently with the samples in the SDG.

5.11.3 Procedure

5.11.3.1 Prepare a canister containing all the LCS compounds at a concentration of 10 ppbv for each compound.

5.11.3.2 Analyze an aliquot according to the same procedure as described in section 5.12.3.

5.11.4 Calculations

5.11.4.1 Calculate individual compound recoveries of the LCS using the following equation:

$$LCS\ \%Recovery = \frac{Concentration_{reported}}{Concentration_{spiked}} \times 100 \qquad \text{Eq. D/VC-22}$$

5.11.4.2 Field sample calculations in section 5.12 also apply to the LCS.

5.11.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the LCS are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.

5.11.5.1 The LCS must be analyzed on a GC/MS system meeting the BFB, initial or continuing calibration, and blank technical acceptance criteria at the frequency described in section 5.11.2.

5.11.5.2 The percent recovery for each of the compounds in the LCS must be within the recovery limits listed on Table D/VC-7.

5.11.5.3 The area response change between the LCS and the most recent valid calibration for each of the internal standards must be less than or equal to ±40 percent.

5.11.5.4 The retention time shift between the LCS and the most recent valid calibration for each of the internal standards must be within ±20.0 seconds.

5.11.6 **Corrective Action**

5.11.6.1 If the technical acceptance criteria for the internal standards are not met, check calculations and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria.

5.11.6.2 The laboratory may not submit data from an SDG until all the LCS technical acceptance criteria are met. LCS contamination from laboratory sources or any LCS analyzed not meeting all the technical acceptance criteria will require reanalysis at no additional cost to the Agency.

5.11.6.3 LCS acceptance criteria MUST be met before any field samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when the LCS technical acceptance criteria have not been met will require reanalysis at no additional cost to the Agency.

5.11.7 **Documentation**

Reporting requirements are listed in Exhibit B. Laboratory Control Sample analysis data are reported on Form III-AAVC. Internal standard area and RT shall be tabulated on Form VII-AAVC.

5.12 **SAMPLE ANALYSIS**

5.12.1 **Summary**

An aliquot of the air sample collected in a canister is cryofocused and subsequently analyzed by GC/MS under conditions in section 5.5. Guidelines for qualitative and quantitative analysis are discussed in sections 5.13 and 5.14.

5.12.2 **Frequency**

5.12.2.1 If time remains in the 12-hour period in which an initial calibration is performed, samples may be analyzed without analysis of a continuing calibration standard.

5.12.2.2 If time does not remain in the 12-hour period since the injection of the instrument performance check standard in which an initial calibration is performed, both the instrument performance check standard and the continuing calibration standard must be analyzed before sample analysis may begin.

5.12.3 **Procedure - Instrumental Analysis**

5.12.3.1 All canister samples must be at ambient temperature before analysis.

5.12.3.2 Check and adjust the mass flow controllers to provide correct flow rates for the system.

5.12.3.3 Connect the sample canister to the inlet of the GC/MS analytical system, as shown in Figure D/VC-4. For pressurized samples with sufficient pressure to drive an electronic mass flow controller, place the controller on the canister, open the canister valve, and vent the canister flow past a tee inlet to the analytical system. The flow rate out of the canister is higher than the optimized sample flow rate so that excess sample is vented and exhausted from the pump, while the desired sample flow is established through the six-port chromatographic valve and the preconcentrator to the downstream flow controller. The absolute volume of sample being pulled through the trap must be consistent from run to run.

5.12.3.4 Cool the GC oven and cryogenic trap to their set points of -50°C and -150°C, respectively. As soon as the cryogenic trap reaches its lower set point of -150°C, the six-port chromatographic valve is cycled to the trap position to begin sample collection. Utilize the sample collection time which has been optimized by the analyst.

5.12.3.5 Use a gastight syringe or some alternate method of introduction of the internal standard during the sample collection period. Add sufficient internal standard equivalent to 10 ppbv in the sample. For example, a 0.5 cm^3 volume of a mixture of internal standard compounds, each at 10 ppmv concentration, added to a sample volume of 500 cm^3, will result in 10 ppbv of each internal standard in the sample.

5.12.3.6 After the sample and internal standards are preconcentrated on the cryogenic trap, the GC sampling valve is cycled to the inject position and the cryogenic trap is heated (-150°C to 120°C in 60 sec)

and swept with helium. The trapped analytes are thermally desorbed onto the head of the capillary column and are separated on the column using the GC oven temperature program. The canister valve is closed and the canister is disconnected from the mass flow controller and capped. The trap is maintained at 120°C until the beginning of the next analysis.

5.12.3.7 Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least five scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra.

5.12.3.8 Each analytical run must be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a laboratory blank analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

5.12.3.9 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative.

5.12.4 Calculations

5.12.4.1 The equation below is used for calculating concentrations.

$$X_a = \frac{A_x C_{is} DF}{A_{is} RRF} \qquad \text{Eq. D/VC-23}$$

where: X_a = compound concentration, ppbv;
A_x = area of the characteristic ion for the compound to be measured;
A_{is} = area of the characteristic ion for the specific internal standard;
C_{is} = concentration of the internal standard spiking mixture, ppbv;
RRF = relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration; and
DF = dilution factor calculated as described in 4.5. If no dilution is performed, DF = 1.

NOTE: The equation above is valid under the condition that the volume (500 µL) of internal standard spiking mixture added in all

field and QC analyses is the same from run to run, and that the volume (500 cm^3) of field and QC sample introduced into the trap is the same for each analysis.

5.12.4.2 Percent Area Response Change (%ARC): Calculate the change in area response for each internal standard by comparing with the most recent valid calibration using the following equation:

$$\%ARC = \frac{A_c - A}{A_c} \times 100 \qquad \text{Eq. D/VC-24}$$

where: %ARC = percent area response change;
A_c = area response of the IS in the most recent valid calibration; and
A = area response of the IS in the sample.

5.12.4.3 Internal Standard Retention Time Shift (RTS): Calculate the shift in retention time between the RT in the sample and in the most recent valid calibration standard for each of the internal standards using the following equation:

$$RTS = RT_c - RT \qquad \text{Eq. D/VC-25}$$

where: RT_c = retention time of the IS in the most recent valid calibration; and
RT = retention time of the IS in the sample.

5.12.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the sample are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.

5.12.5.1 The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in section 5.12.2.

5.12.5.2 The field sample must be analyzed with a laboratory method blank that met the blank technical acceptance criteria.

5.12.5.3 All of the target analyte peaks must be within the initial calibration range.

5.12.5.4 The retention time for each internal standard must be within ±20.0 seconds of the retention time of the internal standard in the most recent valid calibration.

5.12.5.5 The %ARC for each of the internal standards must be within ±40 percent of the most recent valid calibration.

5.12.6 **Corrective Action**

5.12.6.1 If the on-column concentration of any compound in any sample exceeds the initial calibration range, an aliquot of the original sample must be diluted as discussed in section 4.5 and reanalyzed. Guidance in performing dilutions, and exceptions to this requirement are given below.

5.12.6.1.1 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

5.12.6.1.2 The dilution factor chosen should keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

5.12.6.1.3 Do not submit data for more than two analyses, i.e., the original sample and one dilution, or, if the screening procedure was employed, the most concentrated dilution analyzed and one further dilution.

5.12.6.2 Internal standard responses and retention times must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 seconds from the latest daily (12-hour) calibration standard or mid level standard if samples are analyzed in an inital calibration analytical sequence, the GC/MS system must be inspected for malfunctions, and corrections made as required.

5.12.6.3 If the SICP area for any internal standard changes by more than ±40 percent between the sample and the most recent valid calibration, the GC/MS system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

5.12.6.4 If after reanalysis, the SICP areas or the RTs for all internal standards are inside the contract limits, then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with SICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

5.12.6.5 If the reanalysis of the sample does not solve the problem, i.e., the SICP areas or internal standard RTs are outside the contract limits for both analyses, then submit the SICP data and

sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the SDG Narrative all inspection and corrective actions taken.

5.12.6.6 When target compounds are below contract required quantitation limits (CRQL), but the spectra meet the identification criteria, report the concentration with a "J." For example, if the CRQL is 5 ppbv and a concentration of 2 ppbv is calculated, report as "2J."

5.12.7 Documentation

Reporting requirements are listed in Exhibit B. Sample analysis data are reported on Form I-AAVC; Internal standard area and RT shall be tabulated on Form VII-AAVC.

5.13 PERFORMANCE EVALUATION SAMPLES

5.13.1 Summary

The performance evaluation (PE) samples will assist the Agency in monitoring Contractor performance. The laboratory will not be informed as to which compounds are contained in the PE samples or the concentrations.

5.13.2 Frequency

The laboratory must analyze, and report the results of the PE sample once per sample delivery group, if available.

5.13.3 Procedure

5.13.3.1 The laboratory will receive humidified PE samples in SUMMA® canisters from the Agency. The samples will come with instructions concerning the analysis procedure required for the PE samples.

5.13.3.2 Each laboratory must analyze the PE sample using the procedure described in section 5.12 for those target compounds listed in Table D/VC-1.

5.13.4 Calculations

Calculations for PE samples are the same as those for field samples. Use the equations in section 5.12 for determining technical acceptance criteria compliance.

5.13.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial

calibration, internal standard area responses and RTs in the PE sample are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.

5.13.5.1 The PE sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in section 5.13.2.

5.13.5.2 The PE sample must be analyzed with a method blank that met the blank technical acceptance criteria.

5.13.5.3 The retention time for each internal standard in the PE sample analsis must be within ±20.0 seconds of the retention time of the internal standard in the most recent valid calibration.

5.13.5.4 The %ARC for each of the internal standards in the PE sample analysis must be within ±40 percent of the most recent valid calibration.

5.13.5.5 The results of analysis must identify the target compounds provided in the performance evaluation sample and must meet precision and accuracy criteria in comparison with the known results, as outlined in section 6.

5.13.6 Corrective Action

5.13.6.1 If the PE sample technical acceptance criteria for the internal standards are not met, check calculations and instrument performance. It may be necessary to reanalyze the PE sample or take other corrective action procedures to meet the internal standard criteria.

5.13.6.2 If after reanalysis, the SICP areas or the RTs for all internal standards are inside the contract limits, then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with SICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

5.13.6.3 If the reanalysis of the PE sample does not solve the problem, i.e., the SICP areas or internal standard RTs are outside the contract limits for both analyses, then submit the SICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the SDG Narrative all inspection and corrective actions taken.

5.13.6.4 When target compounds are below contract required quantitation limits (CRQL), but the spectra meet the identification criteria, report the concentration with a "J." For example, if the CRQL is 5 ppbv and a concentration of 2 ppbv is calculated, report as "2J."

5.13.6.5 In addition to complying with the PE sample technical acceptance criteria, the Contractor will be responsible for correctly identifying the compounds included in the PE sample. The Agency will notify the Contractor of unacceptable performance.

5.13.6.6 If the PE sample is provided with the SDG, technical acceptance criteria for the PE sample MUST be met before sample data are reported. Also, the Contractor must demonstrate acceptable performance for compound identification and quantification. If the Contractor fails to meet the PE sample technical acceptance criteria, the Agency may take, but is not limited to, the following actions: reduction of the number of samples, suspension of sample shipment, a site visit, a full data audit, and/or require the laboratory to analyze a remedial PE sample.

5.13.7 **Documentation**

Reporting requirements are listed in Exhibit B. Sample analysis data are reported on Form I-AAVC; Internal standard area and RT shall be tabulated on Form VII-AAVC.

5.14 QUALITATIVE ANALYSIS

5.14.1 The compounds in Table D/VC-1 of this section shall be identified by an analyst competent in the interpretation of mass spectra (see Bidder Responsibility description) by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the target compound identifications: (1) elution of the sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

5.14.2 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run in the same 12-hour time period as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

5.14.3 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes

only if the Contractor's GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRFs.

5.14.4 Requirements for Qualitative Verification

5.14.4.1 All ions present in the standard mass spectra at a relative intensity greater than 10 percent (most abundant ion in the spectrum equals 100 percent) must be present in the sample spectrum.

5.14.4.2 The relative intensities of the specified ions must agree within ±20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent).

5.14.4.3 Ions greater than 10 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL report the actual value followed by a "J", e.g., "3J."

5.14.4.4 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

NOTE: If a compound cannot be verified by all of the above criteria, but in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantification.

5.14.5 Guidelines for Making Tentative Identification

5.14.5.1 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the 1990 (or more recent) release of the NIST Library, containing 50,000 spectra, shall be used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

5.14.5.2 Up to 10 VOCs of greatest apparent concentration not listed in Table D/VC-1 shall be tentatively identified via a forward search of the NIST Library. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification using the

following guidelines:

5.14.5.2.1 Characteristic ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

5.14.5.2.2 The relative intensities of the major ions should agree within ±20 percent. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

5.14.5.2.3 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compour s.

5.14.5.2.4 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

NOTE: If in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

5.15 QUANTITATIVE ANALYSIS

5.15.1 Target Compounds

5.15.1.1 Target compounds identified shall be quantified by the internal standard method using the SICP area of the characteristic ions of analytes listed in Table D/VC-3.

5.15.1.2 The relative response factor (RRF) from the daily calibration standard analysis or from the mid level standard, if samples are analyzed in the same 12-hour time period as the initial calibration, is used to calculate the concentration in the sample. The equation for determining concentration is provided in section 5.12.4.

5.15.2 Non-Target Compounds

5.15.2.1 An estimated concentration for non-target components that are tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard

free of interferences shall be used.

5.15.2.2 The formula for calculating concentrations is the same as in section 5.12.4. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. An RRF of 1 is to be assumed. The value from this quantitation shall be qualified as estimated (i.e., flagged "J"). This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

6 REQUIREMENTS FOR DEMONSTRATING METHOD ACCEPTABILITY FOR VOC ANALYSIS FROM CANISTERS

There are three performance criteria which must be met for a laboratory to demonstrate that its analysis method of choice can successfully perform VOC analysis on air samples. Criteria are established for: the method detection limit, replicate precision, and audit accuracy. These criteria are a detection limit of ≤5 ppbv, replicate precision within 25 percent, and audit accuracy within 30 percent for concentrations normally expected in ambient air. Specific criteria for each compound on the Target Compound List must be met by the participating laboratories. These criteria were established using monitoring data from the Toxics Air Monitoring System (TAMS) Network and the Urban Air Toxics Monitoring Program (UATMP) network. The primary reason to base the acceptability of analysis method on performance is to allow systems currently being used for the analysis of VOCs in water to be used for VOCs in air. Essentially these analytical systems would be used for analysis of an ambient air stream instead of the carrier air stream from a purge and trap system. Details for the determination of each of the criteria follow.

6.1 METHOD DETECTION LIMIT

The procedure chosen to define the method detection limit is that given in 40CFR136 Appendix B. The method detection limit is defined by each laboratory by making seven replicate measurements of a concentration of the compound of interest near (within a factor of five) the expected detection limit, computing the standard deviation for the seven replicate concentrations, and multiplying this value by 3.14 (the Student's t value for 99 percent confidence for 7 values). Employing this method, the detection limits given in Table D/VC-4 were obtained for some of the VOCs on the Target Compound List.

6.2 REPLICATE PRECISION

6.2.1 The measure of precision used for this program is the absolute value of the relative difference between replicate measurements of the same sample expressed as a percentage as follows:

$$\textit{percent difference} = \frac{|x_1 - x_2|}{\bar{x}} \times 100 \qquad \text{Eq. D/VC-26}$$

where: x_1 = first measurement value;
x_2 = second measurement value; and
$\bar{x}$ = average of the two values.

6.2.2 There are several factors which may affect the precision of the measurement. The nature of the compound of interest itself may have some effect on the precision, such as the observation that styrene generally shows slightly poorer precision than the bulk of nonpolar VOCs. The

primary influence on precision is the concentration level of the compound of interest in the sample, i.e., the precision degrades as the concentration approaches the detection limit. A "best case" measure of replicate precision was found to be within 6-7 percent for replicate calibration samples at the 10 ppbv level. A more conservative measure was obtained from replicate analysis of "real world" canister samples from the TAMS and UATMP networks. These data are given in Table D/VC-5. The information presented in Table D/VC-5 was used to determine the replicate precision value of 25 percent as a goal to be achieved for each of the target compounds.

6.3 AUDIT ACCURACY

6.3.1 A measure of analytical accuracy is the degree of agreement with audit standards. Audit accuracy is defined as the relative difference between the measurement result and the nominal concentration of the audit compound;

$$\textit{Audit Accuracy, \%} = \frac{\textit{Spiked Value - Observed Value}}{\textit{Spiked Value}} \times 100 \quad \text{Eq. D/VC-27}$$

6.3.2 Audit standards will be supplied to the participating laboratories, these audit standards analyzed, and the results used to obtain audit accuracy values. Audit accuracy values will be compared to similar values obtained from network data. Audit accuracy results for TAMS and UATMP analyses are summarized in Table D/VC-6. These values form the basis for a selection of 30 percent as the performance criterion for audit accuracy.

TABLE D/VC-1. TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL) FOR VOLATILES COLLECTED IN CANISTERS

Compound	CAS RN	CRQL, ppbv
Acetone	67-64-1	5
Acetonitrile	75-05-8	5
Acrolein	107-02-8	5
Acrylonitrile	107-13-1	5
Benzene	71-43-2	2
Benzyl chloride	100-44-7	5
Bromodichloromethane	75-27-4	2
Bromomethane	74-83-9	5
1,3-Butadiene	106-99-0	5
2-Butanone	78-93-3	5
Carbon tetrachloride	56-23-5	2
Chlorobenzene	108-90-7	2
Chlorodifluoromethane	75-45-6	2
Chloroethane	75-00-3	2
Chloroform	67-66-3	2
Chloromethane	74-87-3	2
3-Chloro-1-propene	107-05-1	2
Dibromochloromethane	124-48-1	2
1,2-Dibromoethane	106-93-4	2
1,2-Dichlorobenzene	95-50-1	2
1,3-Dichlorobenzene	541-73-1	5
1,4-Dichlorobenzene	106-46-7	5
Dichlorodifluoromethane	75-71-8	2
1,1-Dichloroethane	75-34-3	2
1,2-Dichloroethane	107-06-2	2
1,1-Dichloroethene	75-35-4	5
cis-1,2-Dichloroethene	156-59-2	2
trans-1,2-Dichloroethene	156-60-5	2
1,2-Dichloropropane	78-87-5	2
cis-1,3-Dichloropropene	10061-01-5	2
trans-1,3-Dichloropropene	10061-02-6	2
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	2
Ethyl benzene	100-41-4	2
Heptane	142-82-5	2
Hexachlorobutadiene	87-68-3	5
Hexane	110-54-3	2

TABLE D/VC-1. TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL) FOR VOLATILES COLLECTED IN CANISTERS
(continued)

Compound	CAS RN	CRQL ppbv
Methanol	167-56-1	5
Methylene chloride	75-09-2	5
Methyl methacrylate	80-62-6	2
4-Methyl-2-pentanone	108-10-1	5
alpha-Methyl styrene	98-83-9	5
Octane	111-65-9	2
n-Pentane	109-66-0	2
Propylene	115-07-1	5
Styrene	100-42-5	5
1,2,4-Trichlorobenzene	120-82-1	2
1,1,1-Trichloroethane	71-55-6	5
1,1,2-Trichloroethane	79-06-5	5
1,1,2,2-Tetrachloroethane	79-43-5	2
Tetrachloroethylene	127-18-4	5
Toluene	108-88-3	5
Trichloroethylene	79-01-6	2
Trichlorofluoromethane	75-69-4	2
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2
1,2,4-Trimethylbenzene	95-63-6	2
1,3,5-Trimethylbenzene	108-67-8	2
Vinyl acetate	108-05-4	5
Vinyl chloride	75-01-4	2
Xylenes, m- and p-	1330-20-7	5
Xylene, o-	95-47-6	5

TABLE D/VC-2. REQUIRED BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	base peak, 100 percent relative abundance
96	5.0 to 9.0 percent of m/e 95 (See note)
173	less than 2.0 percent of m/e 174
174	50.0 to 120.0 percent of m/e 95
175	4.0 to 9.0 percent of m/e 174
176	93.0 to 101.0 percent of m/e 174
177	5.0 to 9.0 percent of m/e 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE D/VC-3. QUANTITATION IONS FOR TARGET COMPOUNDS

Target Compound	Primary Ion*	Secondary Ions
Acetone+,°	43	58
Acetonitrile+,°	41	40
Acrolein	27	56, 25, 55, 28
Acrylonitrile+,°	53	26, 53, 52, 51
Benzene+,°	78	77, 50
Benzyl chloride°	91	126
Bromodichloromethane	83	85
Bromomethane+,°	94	96
1,3-Butadiene+	39	54, 27, 53, 28
2-Butanone+,°	72	57
Carbon tetrachloride+,°	117	119
Chlorobenzene+,°	112	77,114
Chlorodifluoromethane	51	31
Chloroethane°	64	29, 27
Chloroform+,°	83	85, 47
Chloromethane°	50	52
3-Chloro-1-propene°	41	39, 76
Dibromochloromethane	129	208, 206
1,2-Dibromoethane+,°	107	109, 27
1,2-Dichlorobenzene°	146	148, 111
1,3-Dichlorobenzene°	146	148, 111
1,4-Dichlorobenzene°	146	148, 111
Dichlorodifluoromethane+,°	85	87
1,1-Dichloroethane°	63	27, 65
1,2-Dichloroethane+,°	62	27, 64
1,1-Dichloroethene+,°	61	96, 63
cis-1,2-Dichloroethene°	61	96, 98
trans-1,2-Dichloroethene	61	96, 98
1,2-Dichloropropane+,°	63	41, 62
cis-1,3-Dichloropropene°	75	39, 77
trans-1,3-Dichloropropene°	75	39, 77
1,2-Dichloro-1,1,2,2-tetrafluoroethane+,°	85	135, 87
Ethyl benzene+,°	91	106

*The primary ion should be used unless interferences are present, in which case a secondary ion may be used.

+NIST certified gaseous standards are available for these compounds.

°Gaseous standards prepared gravimetrically using NIST traceable weights are available for these compounds.

TABLE D/VC-3. QUANTITATION IONS FOR TARGET COMPOUNDS
(continued)

Target Compound	Primary Ion*	Secondary Ions
Heptane	43	41, 57, 71, 29
Hexachlorobutadiene°	225	227, 223
Hexane	57	43, 41, 29, 27
Methanol°	31	32, 29
Methylene chloride+,°	49	84, 86
Methyl methacrylate°	41	39, 69, 100
4-Methyl-2-pentanone	43	58, 100
alpha-Methyl styrene	118	117, 103, 78, 115
Octane	43	41, 29, 57, 85
n-Pentane	43	42, 41, 27, 29
Propylene	41	42, 39, 40, 27
Styrene°	104	78, 103
1,1,2,2-Tetrachloroethane°	83	85
Tetrachloroethene+,°	164	129, 131,166
Toluene+,°	91	92
1,2,4-Trichlorobenzene°	180	182, 184
1,1,1-Trichloroethane+,°	97	99, 61
1,1,2-Trichloroethane°	97	83, 61
Trichloroethene+,°	130	132, 95
Trichlorofluoromethane+,°	101	103
1,1,2-Trichloro-1,2,2-trifluoroethane+,°	151	101, 103
1,2,4-Trimethylbenzene°	105	120
1,3,5-Trimethylbenzene°	105	120
Vinyl acetate	43	86
Vinyl chloride+,°	62	27, 64
Xylenes, o-, m-, and p-°	91	106

*The primary ion should be used unless interferences are present, in which case a secondary ion may be used.

+NIST certified gaseous standards are available for these compounds.

°Gaseous standards prepared gravimetrically using NIST traceable weights are available for these compounds.

TABLE D/VC-4. METHOD DETECTION LIMITS (MDL)*

TO-14 List	Lab #1, SCAN	Lab #2, SIM	MDL, ppbv
Benzene	0.34	0.29	0.4
Benzyl Chloride	--	--	--
Carbon tetrachloride	0.42	0.15	0.5
Chlorobenzene	0.34	0.02	0.4
Chloroform	0.25	0.07	0.3
m-Dichlorobenzene	0.36	0.07	0.4
1,2-Dibromoethane	--	0.08	0.3
p-Dichlorobenzene	0.70	0.12	0.7
o-Dichlorobenzene	0.44	--	0.5
1,1-Dichloroethane	0.27	0.05	0.3
1,2-Dichloroethane	0.24	--	0.3
1,1-Dichloroethene	--	0.22	1.1
cis-1,2-Dichloroethene	--	0.06	0.3
Dichloromethane	1.38	0.84	1.4
1,2-Dichloropropane	0.21	--	0.3
cis-1,3-Dichloropropene	0.36	--	0.4
trans-1,3-Dichloropropene	0.22	--	0.3
Ethylbenzene	0.27	0.05	0.3
Ethyl Chloride	0.19	--	0.2
4-Ethyltoluene	--	--	--
Freon 11	--	--	--
Freon 113	--	--	--
Freon 114	--	--	--
Freon 12	--	--	--
Hexachlorobutadiene	--	--	--
Methyl Bromide	0.53	--	0.6
Methyl Chloride	0.40	--	0.4
Styrene	1.64	0.06	1.7
1,1,2,2-Tetrachloroethane	0.28	0.09	0.3
Tetrachloroethylene	0.75	0.10	0.8
Toluene	0.99	0.20	1.0
1,2,4-Trichlorobenzene	--	--	--
1,1,1-Trichloroethane	0.62	0.21	0.7
1,1,2-Trichloroethane	0.50	--	0.5
Trichloroethene	0.45	0.07	0.5
1,2,4-Trimethylbenzene	--	--	--
1,3,5-Trimethylbenzene	--	--	--
Vinyl Chloride	0.33	0.48	0.4
m,p-Xylene	0.76	0.08	0.8
o-Xylene	0.57	0.28	0.6

*Method Detection Limits (MDLs) are defined as the product of the standard deviation of seven replicate analyses and the student's "t" test value for 99% confidence. For Lab #2, the MDLs represent an average over four studies. MDLs are for MS/SCAN for Lab #1 and for MS/SIM for Lab #2. For those compounds measured by SIM but not by SCAN, the SIM MDL has been multiplied by a factor of 5 and this value has been used as a substitute SCAN MDL. The resultant list of MDLs are given in the last column. Ten of the compounds have no listed MDLs.

TABLE D/VC-5. SUMMARY OF EPA DATA ON REPLICATE PRECISION (RP) FROM EPA NETWORK OPERATIONS*

Monitoring Compound Identification	Urban Air Toxics Monitoring Program (UATMP)			Toxics Air Monitoring Stations (TAMS)		
	%RP	#	ppbv	%RP	#	ppbv
Freon 12	--		--	13.9	47	0.9
Dichloromethane	16.3	07	4.3	19.4	47	0.6
1,2 Dichloroethane	36.2	31	1.6	--	--	--
1,1,1 Trichloroethane	14.1	44	1.0	10.6	47	2.0
Benzene	12.3	56	1.6	4.4	47	1.5
Trichloroethene	12.8	08	1.3	--	--	--
Toluene	14.7	76	3.1	3.4	47	3.1
Tetrachloroethene	36.2	12	0.8	--	--	--
Chlorobenzene	20.3	21	0.9	--	--	--
Ethylbenzene	14.6	32	0.7	5.4	47	0.5
m,p-Xylene	14.7	75	4.0	5.3	47	1.5
Styrene	22.8	59+	1.1	8.7	47	0.2+
o-Xylene	--		--	6.0	47	0.5
m-Dichlorobenzene	49.1	06	0.6	--	--	--
p-Dichlorobenzene	14.7	14	6.5	--	--	--

* # denotes the number of replicate or duplicate analysis used to generate the statistic. The replicate precision is defined as the mean ratio of absolute difference to the average value.

\+ Styrene and o-Xylene coelute from the GC column used in UATMP. For the TAMS entries, both values were below detection limits for 18 of 47 replicates and were not included in the calculation.

TABLE D/VC-6. AUDIT ACCURACY (AA) VALUES* FOR SELECTED TO-14 COMPOUNDS

Selected Compounds From TO-14 List	FY-88 TAMS AA(%), N=30	FY-88 UATMP AA(%), N=3
Vinyl chloride	4.6	17.9
Methyl bromide	--	6.4
Freon 11	6.4	--
Dichloromethane	8.6	31.4
Chloroform	--	4.2
1,2-Dichloroethane	6.8	11.4
1,1,1-Trichloroethane	18.6	11.3
Benzene	10.3	10.1
Carbon tetrachloride	12.4	9.4
1,2-Dichloropropane	--	6.2
Trichloroethene	8.8	5.2
Toluene	8.3	12.5
Tetrachloroethylene	6.2	--
Chlorobenzene	10.5	11.7
Ethylbenzene	12.4	12.4
o-Xylene	16.2	21.2

* Audit accuracy is defined as the average of multiple determinations of the absolute difference between the audit measurement result and its nominal value divided by the audit value. N denotes the number of audits averaged to obtain the audit accuracy value. Information is not available for other TO-14 compounds because they were not present in the audit materials.

TABLE D/VC-7. LABORATORY CONTROL SAMPLE (LCS) PERCENT RECOVERY LIMITS

LCS Compound	Percent recovery Limits
Benzene	60-140
Bromoform	60-140
Carbon tetrachloride	60-140
1,2-Dibromoethane	60-140
1,4-Dichlorobenzene	60-140
1,2-Dichloroethane	60-140
1,2-Dichloropropane	60-140
cis-1,3-Dichloropropene	60-140
Tetrachloroethylene	60-140
1,1,2-Trichloroethane	60-140
Trichloroethylene	60-140
Vinyl chloride	60-140

TABLE D/VC-8. CORRESPONDING INTERNAL STANDARDS FOR SOME VOLATILE TARGET COMPOUNDS FOR GC/MS QUANTITATION

Bromochloromethane

Acetone
Bromomethane
2-Butanone
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
Methylene chloride
Vinyl chloride

1,4-Difluorobenzene

Benzene
Bromodichloromethane
Carbon tetrachloride
Dibromochloromethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethylene

Chlorobenzene-d_5

Chlorobenzene
1,2-Dibromoethane
Ethylbenzene
4-Methyl-2-pentanone
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethylene
Toluene
Xylenes, m- and p-
Xylene, o-

NOTE: For the following target compounds in the TCL, the internals standard used is the one closest in retention time to the target compound. In the future as more data become available, each target compound will be assigned a specific internal standard for quantitation.

Compound

Acetonitrile
Acrolein
Acrylonitrile
Benzyl chloride
1,3-Butadiene
Chlorodifluoromethane
3-Chloro-1-propene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Dichlorodifluoromethane
1,2-Dichloro-1,1,2,2-tetrafluoroethane
Heptane
Hexachlorobutadiene
Hexane
Methanol
Methyl methacrylate
alpha-Methyl styrene
Octane
n-Pentane
Propylene
1,2,4-Trichlorobenzene
Trichlorofluoromethane
1,1,2-Trichloro-1,2,2-trifluoroethane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene
Vinyl acetate

Figure D/VC-1

Canister Cleaning System

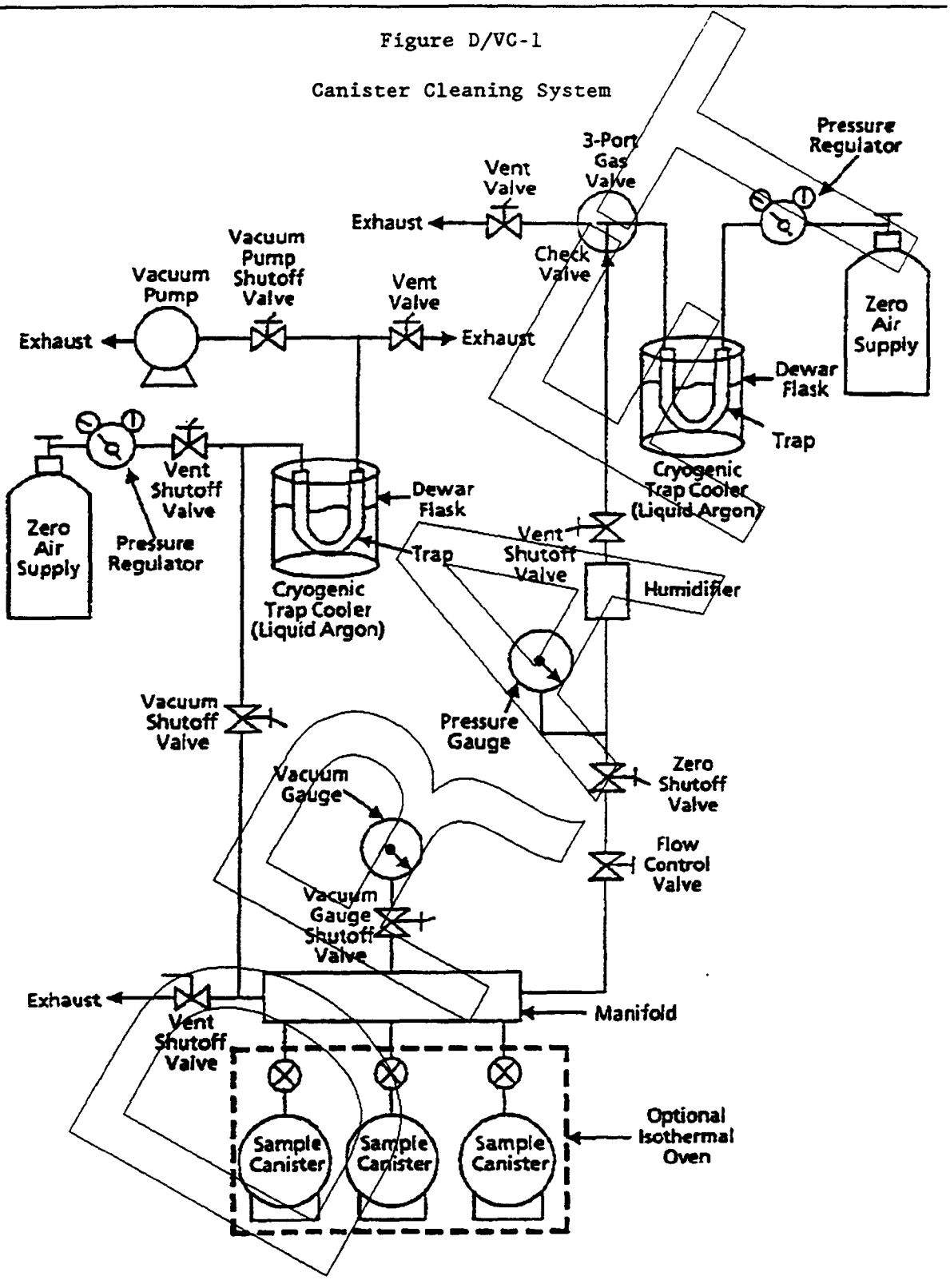


Figure D/VC-2

Alternate Canister Cleaning System

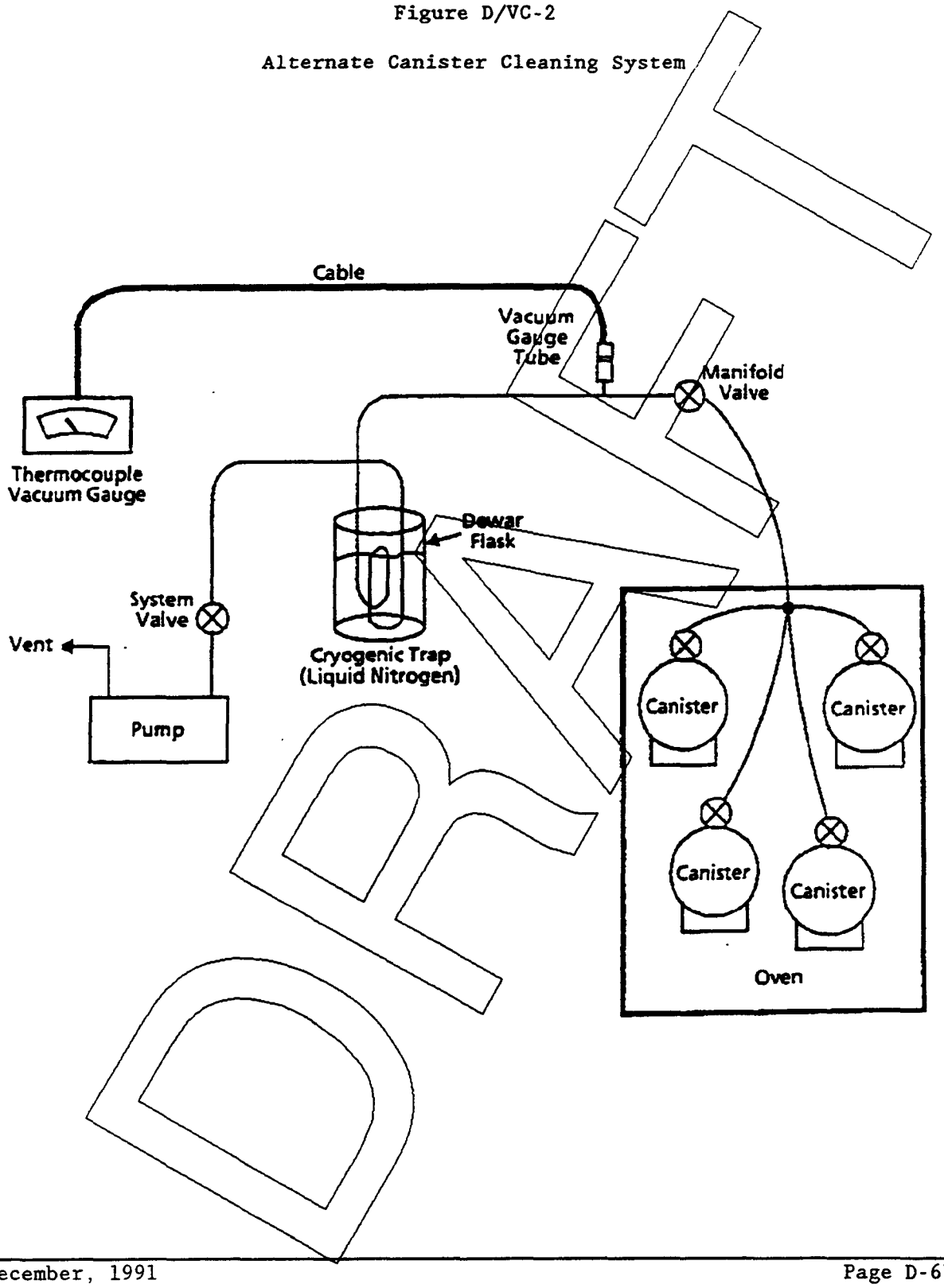
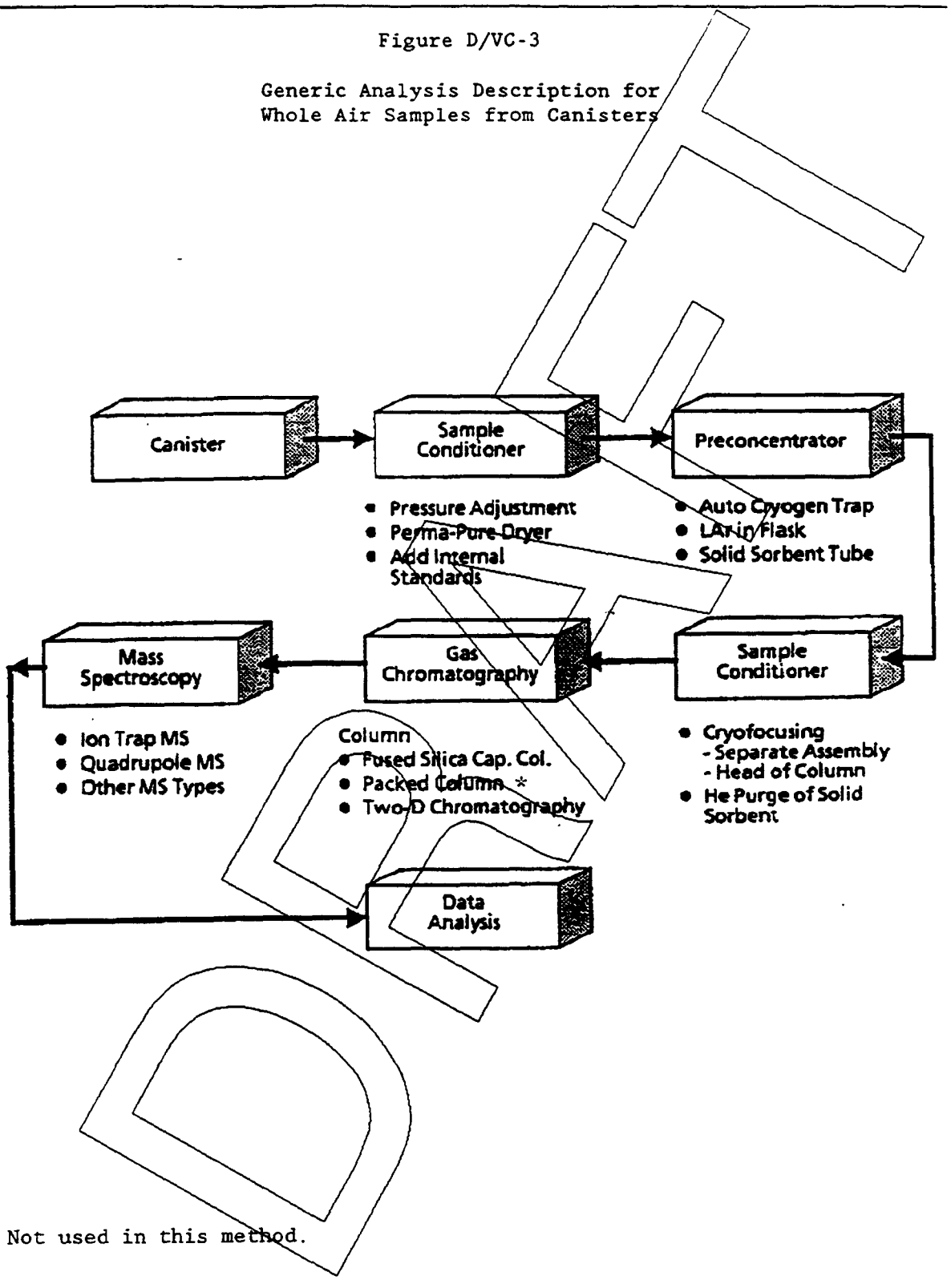


Figure D/VC-3

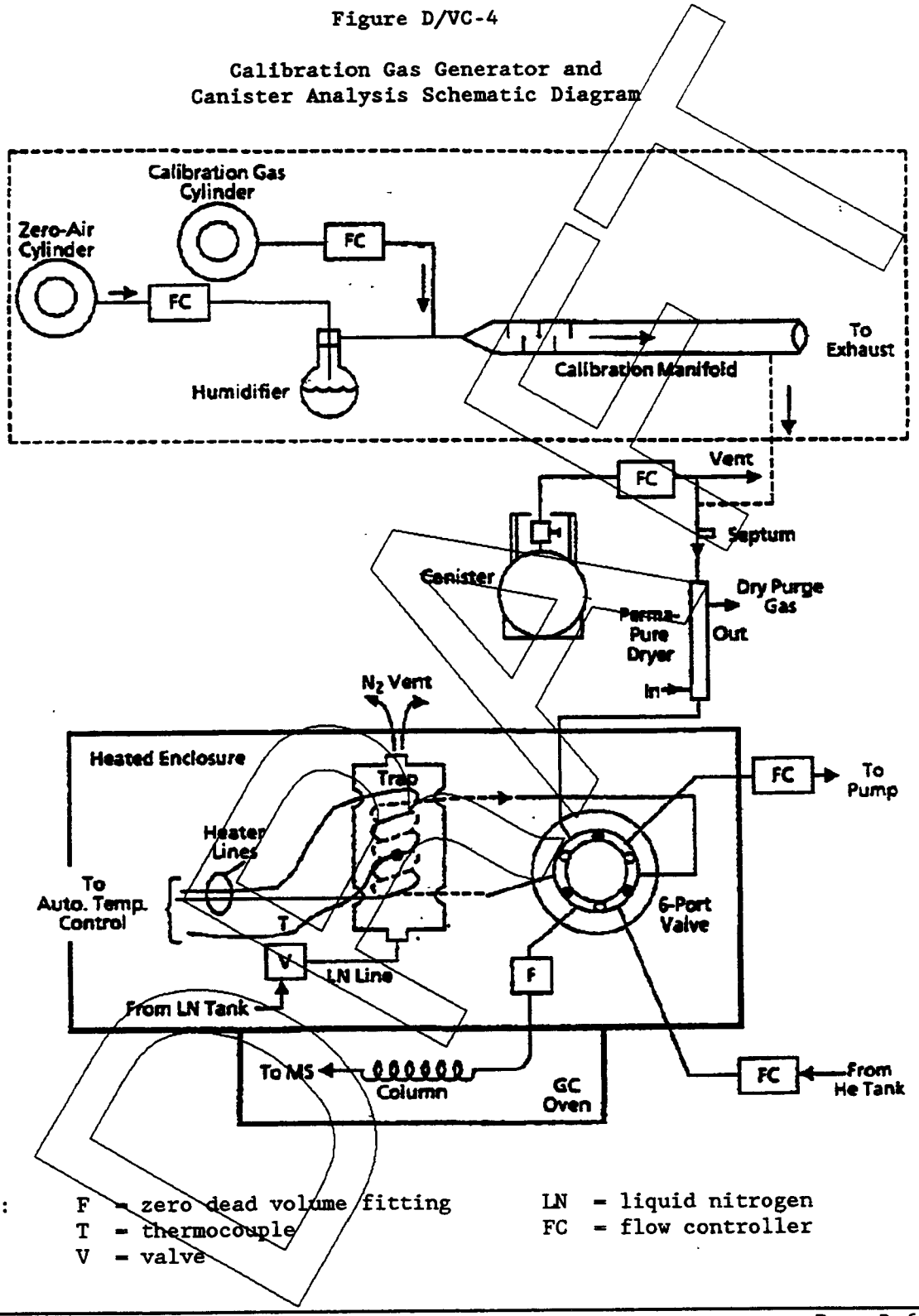
Generic Analysis Description for
Whole Air Samples from Canisters



* Not used in this method.

Figure D/VC-4

Calibration Gas Generator and
Canister Analysis Schematic Diagram



LEGEND:
F = zero dead volume fitting
T = thermocouple
V = valve
LN = liquid nitrogen
FC = flow controller

Figure D/VC-5

Canister Certification Flowchart

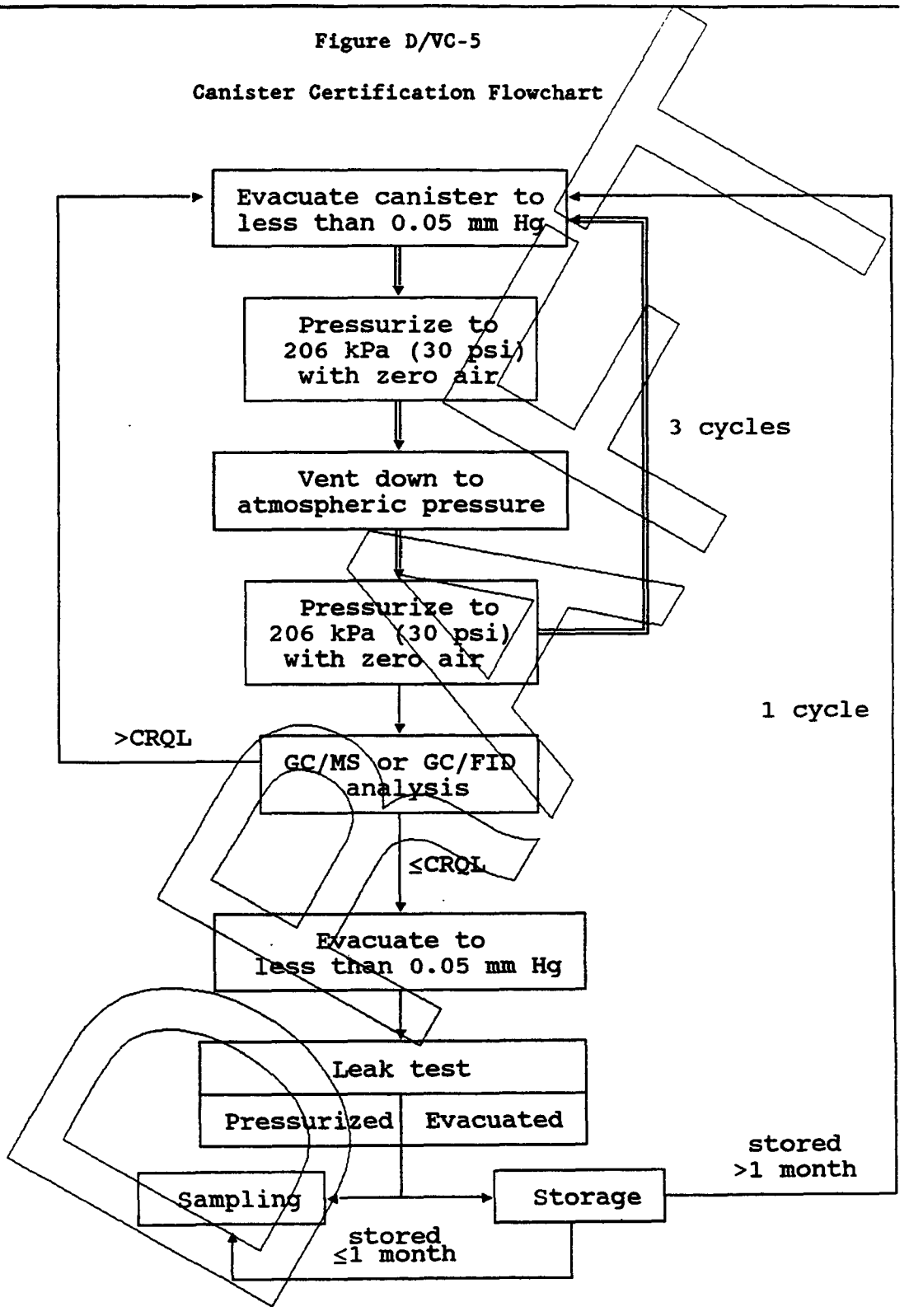
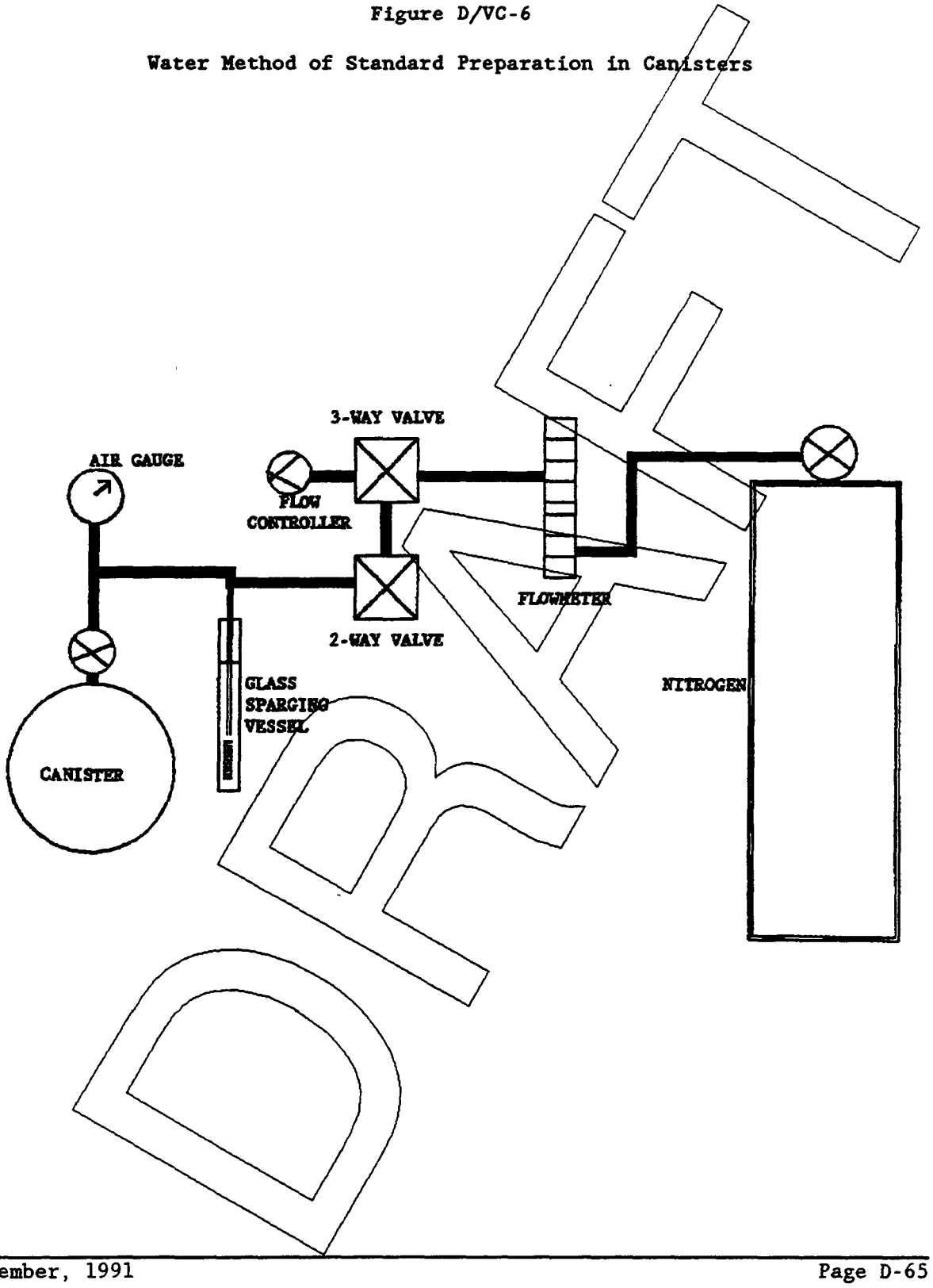


Figure D/VC-6

Water Method of Standard Preparation in Canisters



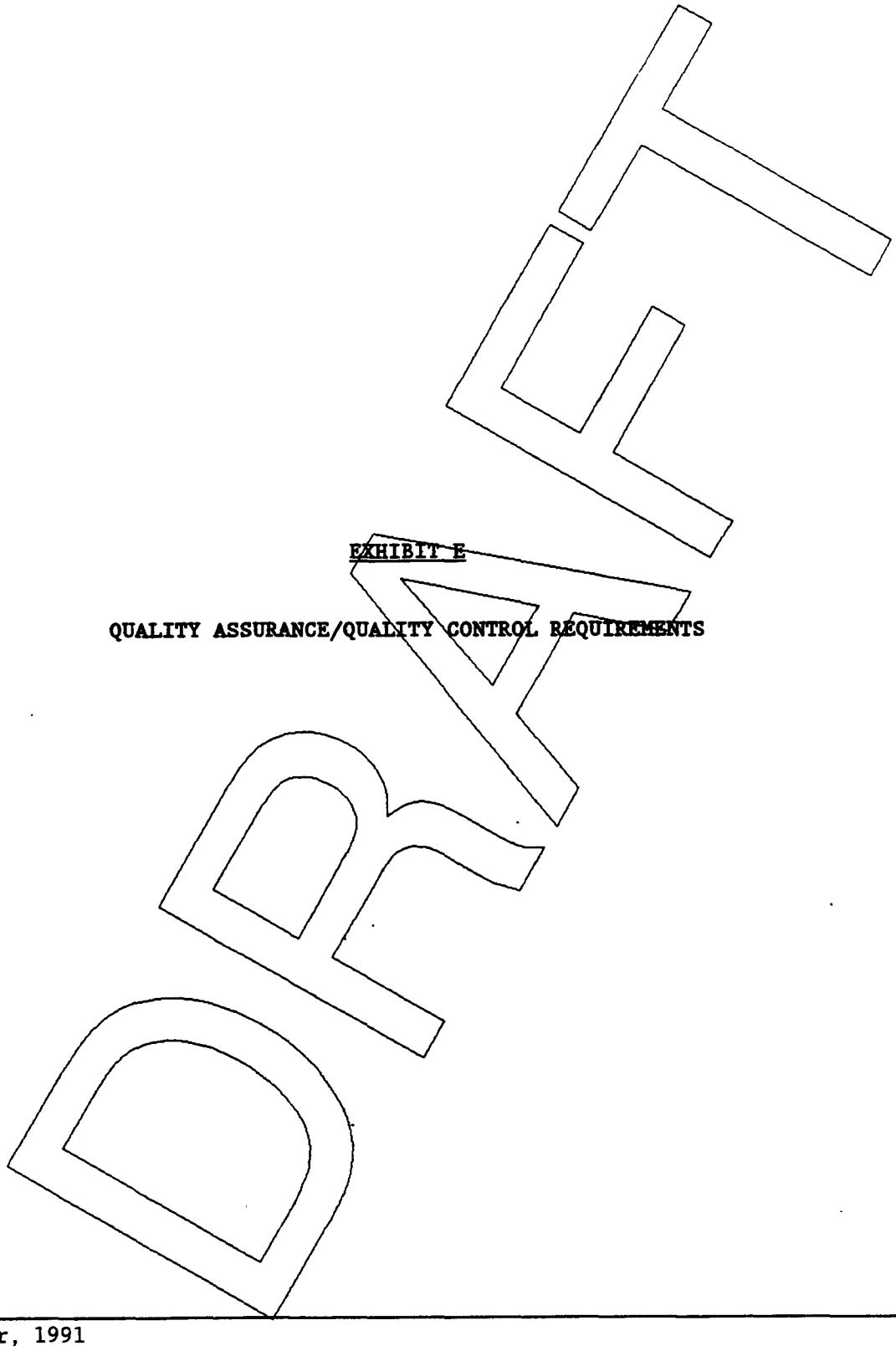


EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

December, 1991

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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SECTION 1

INTRODUCTION

1.1 Quality assurance (QA) and quality control (QC) are integral parts of EPA's Contract Laboratory Program (CLP). The CLP QA program consists of management review and oversight at the planning, implementation, and completion stages of environmental data generation activities, to ensure that data provided are of the quality required. The CLP QC program includes those activities required as part of data generation to ensure that the data are of known and documented quality.

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

1.3 The purpose of this Exhibit is to describe 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 QA/QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These operations are designed to facilitate laboratory comparison by providing the EPA with comparable data from all Contractors. These requirements do not release the laboratory from maintaining its own QC checks on method and instrument performance.

1.4 Appropriate use of data generated under the great range of analytical conditions encountered in ambient air analyses requires reliance on the QC procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories participating in the CLP. However, the validation of these methods does not guarantee that they perform equally well for all samples collected under actual field conditions. Inaccuracies can result from causes such as sampling artifacts, equipment malfunctions, and human error. Therefore, the QC component of each method is indispensable.

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

1.6 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, NEIC, and EMSL/LV. Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Performance evaluation samples provide an external QA reference for the program. A laboratory 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 contract laboratories through direct communications with the Administrative Project Officers (APOs) and Technical Project Officers (TPOs).

1.7 This Exhibit is not a guide to constructing QA project plans, QC systems, or a QA organization. It is, however, an explanation of the QC and QA requirements of the CLP. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a QA Plan and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides the Agency with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

1.8 To ensure that the product delivered by the Contractor meets the requirements of the contract and to improve interlaboratory data comparison, the Agency requires the following from the Contractor.

1.8.1 Development and implementation of a QA program, and documentation of the key elements of that QA program through a written QA Plan, as described in Section 2 of this Exhibit.

1.8.2 Preparation of and adherence to written Standard Operating Procedures (SOPs) as described in Section 5 of this Exhibit.

1.8.3 Adherence to the analytical methods and associated QC requirements specified in the contract.

1.8.4 Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical houses.

1.8.5 Participation in the analysis of laboratory performance evaluation (PE) samples, including adherence to corrective action procedures.

1.8.6 Participation in on-site laboratory evaluations, including adherence to corrective action procedures.

1.8.7 Submission of all raw data and pertinent documentation for Regional review.

1.8.8 Submission, upon request, of GC/MS tapes and applicable documentation for tape audits.

1.8.9 Submission for Agency review of all original documentation generated during sample analyses.

SECTION 2

QUALITY ASSURANCE PLANS

2.1 The Contractor shall establish a QA program with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

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

2.2.1 Maintain data integrity, validity, and usability.

2.2.2 Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility .

2.2.3 Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable.

2.2.4 Document all aspects of the measurement process in order to provide data that are technically sound and legally defensible.

2.3 The QAP must present, in specific terms, the policies, organization, objectives, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during On-Site Laboratory evaluation and upon written request by the Administrative Project Officer. Additional information relevant to the preparation of a QAP can be found in EPA and ASTM publications[2,4].

2.4 **ELEMENTS OF A QUALITY ASSURANCE PLAN**

2.4.1 The following key elements of the Contractor's quality assurance program shall be addressed in the QAP:

2.4.1.1 Contractor QA Policy and Objectives

2.4.1.2 Organization and Personnel

- QA Management;
- Organization;
- Assignment of QC and QA responsibilities; and

- Reporting relationships.

• Personnel;

- Staff resumes;
- Education and experience requirements pertinent to this Contract; and
- Training progress.

• Facilities and Equipment;

- Instrumentation and backup alternatives; and
- Maintenance activities and schedules.

• Document Control;

- Laboratory notebook policy;
- Sample and data tracking/custody procedures and documentation requirements;
- Logbook maintenance and archiving procedures;
- Case file organization, preparation, and review procedures; and
- Procedures for preparation, approval, review, revision, and distribution of SOPs.

• Analytical Methodology;

- Calibration procedures and frequency;
- Sample handling and storage procedures;
- Sample preparation procedures;
- Sample analysis procedures; and
- Standards preparation procedures.

• Data Generation;

- Data collection procedures;
- Data reduction procedures;

- Data review procedures;
- Data reporting and authorization procedures; and
- Data management procedures.

• Quality Control Program; and

- Solvent, reagent, and adsorbent check analysis;
- Reference material analysis;
- Internal QC checks; and
- Corrective action and determination of QC limit procedures.

• Quality Assurance Program Assessment.

- Data audits;
- Systems audits;
- Performance audits;
- Corrective action procedures;
- QA reporting procedures.

SECTION 3

STANDARD OPERATING PROCEDURES

3.1 In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document that 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.

3.2 SOPs prepared by the Contractor must be functional, i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs, as presented to the Agency, must reflect activities as they are currently performed in the laboratory. In addition, all SOPs must:

3.2.1 Be consistent with current EPA regulations, guidelines, and the CLP contract's requirements;

3.2.2 Be consistent with instrument manufacturer's specific instruction manuals;

3.2.3 Be available to the EPA 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 evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs;

3.2.4 Provide for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol;

3.2.5 Describe the mechanism for demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results;

3.2.6 Describe the corrective measures and feedback mechanism used when analytical results do not meet protocol requirements;

3.2.7 Be reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made;

3.2.8 Be archived for future reference in usability or evidentiary situations;

3.2.9 Be available at specific work stations as appropriate; and

3.2.10 Be subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

3.3 SOP SPECIFICATIONS AND FORMAT

3.3.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA sa the basis for laboratory evidence audits.

3.3.2 The format for SOPs may vary depending upon the kind of activity for which they are prepared. However, at a minimum, the following sections must be included.

3.3.2.1 Title page.

3.3.2.2 Scope and application.

3.3.2.3 Definitions.

3.3.2.4 Procedures.

3.3.2.5 QC acceptance criteria.

3.3.2.6 Corrective Action Procedures, including procedures for secondary review of information being generated.

3.3.2.7 Documentation Description and example forms.

3.3.2.8 Miscellaneous notes and precautions.

3.3.2.9 References.

3.4 REQUIRED SOPS

3.4.1 Evidentiary SOPs

The Contractor shall develop and use adequate written SOPs to ensure sample and data accountability. Evidentiary SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.4.1.1 **Sample receipt and logging**

3.4.1.1.1 The Contractor shall have written SOPs for receiving and logging in the samples. The procedures shall include, documentation of the following information:

- Presence or absence of EPA chain-of-custody forms;
- Presence or absence of airbills or airbill stickers;
- Presence or absence of EPA Traffic Reports or SAS packing lists;
- Presence or absence of custody seals on shipping and/or sample containers and their condition;
- Custody seal numbers, when present;
- Presence or absence of sample tags;
- Sample tag ID numbers;
- Condition of the shipping container;
- Condition of the sample container;
- Verification of agreement or nonagreement of information on receiving documents and sample containers;
- Resolution of problems or discrepancies with SMO; and
- The definition of any terms used to describe sample condition upon receipt.

3.4.1.1.2 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.4.1.2 **Sample identification**

3.4.1.2.1 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.4.1.2.2 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.4.1.2.3 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

3.4.1.3 Sample security

The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access to secure storage areas.

3.4.1.4 Internal chain-of-custody of samples and data.

The Contractor shall have written SOPs for the chain-of-custody consisting of sample identification, chain-of-custody procedures, sample receiving procedures, and sample tracking procedures. For more information concerning the chain-of-custody procedures see Section 4 of this Exhibit.

3.4.1.5 Internal tracking of samples and data.

The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

- A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses;
- A description of the documentation used to record instrument calibration and other QA/QC activities; and
- Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.4.1.6 **Laboratory document and information control**

3.4.2 **Analytical SOPs**

The Contractor shall develop and use adequeate written SOPs to ensure that all data generated for the CLP are of known, documented, and acceptable quality. Analytical SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.4.2.1 The Contractor shall have written SOPs for preventing sample contamination, during sample preparation, cleaning of glassware, storage, and analysis.

3.4.2.2 The Contractor shall have SOPs to ensure traceability of standards used in sample analysis QA/QC.

3.4.3 **Quality Management SOPs**

3.4.3.1 The Contractor shall have written SOPs for technical and managerial review of laboratory operation and data package preparation, laboratory data review/laboratory self inspection system. The procedures shall include but not be limited to documenting the following information:

3.4.3.1.1 Data flow and chain-of-command for data review;

3.4.3.1.2 Procedures for measuring precision and accuracy.

3.4.3.1.3 Evaluation of parameters for identifying systematic errors.

3.4.3.1.4 Procedures to assure that hardcopy deliverables are complete and compliant with the requirements in Exhibit B.

3.4.3.1.5 Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.).

3.4.3.1.6 Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).

3.4.3.1.7 Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).

3.4.3.1.8 Documentation of audit reports, (internal and external), response, corrective action, etc.

3.4.3.2 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure. For more information concerning document control and case file preparation, see Section 5 of this Exhibit.

3.4.3.3 The Contractor shall have written SOPs for sample analysis, management and handling, and reporting of data. The procedures shall include but not be limited to documenting the following information:

3.4.3.3.1 Procedures for controlling and estimating data entry errors.

3.4.3.3.2 Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

3.4.3.3.3 Life cycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

3.4.3.3.4 Database security, backup and archival procedures including recovery from system failures.

3.4.3.3.5 System maintenance procedures and response time.

3.4.3.3.6 Individual(s) responsible for system operation, maintenance, data integrity and security.

3.4.3.3.7 Specifications for staff training procedures.

3.4.3.4 The Contractor shall have written SOPs for laboratory safety.

3.5 HANDLING OF CONFIDENTIAL INFORMATION

3.5.1 A Contractor conducting work under this contract may receive EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.5.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.5.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

3.6 SOPS DELIVERY REQUIREMENTS

Within forty-five (45) days of contract receipt, a complete set of SOPs relevant to this contract shall be sent to the TPO, SMO and EMSL/LV. Also, during the term of performance of the contract, copies of SOPs which have been amended or new SOPs which have been written shall be sent to the TPO, EMSL/LV (quality assurance SOPs) and NEIC (evidentiary SOPs).

SECTION 4

CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

4.1 SAMPLE IDENTIFICATION

4.1.1 To ensure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

4.1.2 Each sample and sample preparation container shall be labeled with the EPA number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA number.

4.2 CHAIN-OF-CUSTODY PROCEDURES

Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented. A sample is under custody if the following applies:

4.2.1 It is in your possession, or

4.2.2 It is in your view after being in your possession, or

4.2.3 It was in your possession and you locked it up, or

4.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

4.3 SAMPLE RECEIVING PROCEDURES

4.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

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

4.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment.

4.3.5.1 Airbills or airbill stickers.

4.3.5.2 Custody seals.

4.3.5.3 EPA custody records.

4.3.5.4 EPA traffic reports or SAS packing lists.

4.3.5.5 Sample tags.

4.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

4.3.7 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

4.3.8 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

4.3.9 The following information shall be recorded on appropriate Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected.

4.3.9.1 Condition of the shipping container.

4.3.9.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

4.3.9.3 Custody seal numbers, when present.

4.3.9.4 Condition of the sample bottles.

4.3.9.5 Presence or absence of airbills or airbill stickers.

4.3.9.6 Airbill or airbill sticker numbers.

4.3.9.7 Presence or absence of EPA custody records.

4.3.9.8 Presence or absence of EPA traffic reports or SAS packing lists.

4.3.9.9 Presence or absence of sample tags.

4.3.9.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

4.3.9.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

4.3.9.12 Problems or discrepancies.

4.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 5

DOCUMENT CONTROL

The goal of the laboratory document control program is to assure that all documents for a specified Sample Delivery Group (SDG) will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

5.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

5.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

5.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

5.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

5.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

5.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA,

the laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

5.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be crossed out.

5.2 CONSISTENCY OF DOCUMENTATION

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

5.2.2 All copies of laboratory documents shall be complete and legible.

5.2.3 Original documents which include information relating to more than one SDG shall be filled in the CSF of the lowest SDG number. The copy(s) shall be placed in the other CSF(s) and the Contractor shall record the following information on the copy(ies) in red ink:

"COPY

ORIGINAL IS FILED IN CSF _____"

The Contractor shall sign and date this addition to the copy(ies).

5.2.4 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, lab bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

5.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURE

5.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

5.3.2 All documents relevant to each sample delivery group, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

5.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 is an example of a document inventory.

Figure E-1

Example

DOCUMENT INVENTORY

Document Control #*	Document Type	# Pages
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	50
232-2-0005	SMO Organics Traffic Reports	10
232-2-0006	Analysis Data Sheets	41
232-2-0007	Analysts' Organics Notebook Pages	14
232-2-0008	GC/MS and GC Instrument Logbook Pages	12
etc.	etc.	etc.

*This number is to be recorded on each set of documents.

5.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

5.5 SHIPPING DATA PACKAGES AND CSF

5.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

5.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

5.5.3 A copy of the transmittal letter for the CSF will be sent to the NEIC and the SMO.

5.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one (1) original FORM AADC-1 for each shipping container.

5.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

5.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

5.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 6

ANALYTICAL STANDARDS REQUIREMENTS

The U.S. Environmental Protection Agency will not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories will be required to prepare from neat materials, from cylinders of compressed gases traceable to NIST Standard Reference Materials or NIST/EPA approved certified reference material, or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

6.1 PREPARATION OF CHEMICAL STANDARDS FROM THE NEAT HIGH PURITY BULK MATERIAL

6.1.1 A laboratory may prepare their chemical standards from neat materials. Commercial sources for neat chemical standards pertaining to analytes listed on the TCL are given in Appendix C of the "Quality Assurance Materials Bank: Analytical Reference Standards," Seventh Edition, January 1988. Laboratories should obtain the highest purity possible when purchasing neat chemical standards; standards purchased at less than 98% purity must be documented as to why a higher purity could not be obtained.

6.1.2 Neat chemical standards must be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of neat chemicals is essential in order to safeguard them from decomposition.

6.1.3 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 contract laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use either differential scanning calorimetry, gas chromatography with flame ionization detection, high performance liquid chromatography, infrared spectrometry, or other 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:

$$\text{Wt. of impure compound} = \frac{\text{Wt. of pure compound}}{\left(\frac{\text{percent purity}}{100}\right)} \quad \text{Eq. E-1}$$

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

6.1.4 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 contract laboratory's responsibility to have analytical documentation confirming that all compounds used in the preparation of solution standards are correctly identified. Identification confirmation, when performed, should use GC/MS analysis on at least two different analytical columns, or other appropriate techniques.

6.1.5 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 must verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights. All weighing should 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 should be compatible with the protocol in which the standard is to be used; the solute should be soluble, stable, and nonreactive with the solvent. (For standards in canisters, the solvent is humid zero air.) In the case of a multicomponent solution, the components must not react with each other.

6.1.6 The primary standard is the solution of gas(es) in humid zero air prepared from neat standards. All subsequent dilutions must be traceable back to the primary standard.

6.1.7 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 when not in use. All solution standards are to be clearly labeled as to the identity of the compound or compounds, concentration, date prepared, solvent, and initials of the preparer.

6.2 PREPARATION OF GASEOUS STANDARDS

6.2.1 As discussed in Exhibit D, the Contractor may prepare gaseous standards in a dynamic dilution system from compressed gases traceable to a National Institute of Standards (NIST) Standard Reference Material (SRM) or to a NIST/EPA approved Certified Reference Material (CRM). The components may be purchased in one cylinder or separate cylinders. Other methods of gaseous standards preparation are described in Exhibit D. For these alternate methods, equivalence must be established by the Contractor through an EPA audit procedure. In either method, the Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

6.3 PURCHASE OF CHEMICAL STANDARDS IN SOLUTION

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

6.3.1.1 Contract laboratories must maintain the following documentation to verify the integrity of the standard solutions they purchase:

- Mass spectral identification confirmation of the neat material;
- Purity confirmation of the neat material; and
- Chromatographic and quantitative documentation that the solution standard was QC checked according to the following section.

6.3.1.2 The Contractor must 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 parts a and b below). 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 part 6.3.1.3 which follows. If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test. Thus the standard is certified to be within 10 percent of the target concentration.

6.3.1.3 If the procedure above is used, the supplier must document that the following have been achieved.

- Two solutions of identical concentration must be prepared independently from neat materials. An aliquot of the first solution must be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration ten percent 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 percent less that the target standard. This is called the "low standard";
- Six replicate analyses of each standard (a total of 18 analyses) must be performed in the following sequence: low

standard, target, high standard, low standard, target standard, high standard; and

- The mean and variance of the six results for each solution must be calculated.

$$Mean = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} \quad \text{Eq. E-2}$$

$$Variance = \frac{Y_{12} + Y_{22} + Y_{32} + Y_{42} + Y_{52} + Y_{62} - (6 \times Mean)_2}{5} \quad \text{Eq. E-3}$$

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, Vp, is calculated.

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + V_3}{\frac{1.21}{3}} \quad \text{Eq. E-4}$$

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

- The test statistic must be calculated:

$$TEST\ STATISTIC = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}} \quad \text{Eq. E-5}$$

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

- The test statistic must be calculated:

$$TEST\ STATISTIC = \frac{\left|M_2 - \left(\frac{M_1}{1.8}\right) - \left(\frac{M_3}{2.2}\right)\right|}{\left(\frac{V_p}{4}\right)^{0.5}} \qquad \text{Eq. E-6}$$

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.

- The 95 percent confidence intervals for the mean result of each standard must be calculated:

$$INTERNAL\ FOR\ LOW\ STANDARD = M_1 \pm (2.13)\left(\frac{V_p}{6}\right)^{0.5} \qquad \text{Eq. E-7}$$

$$INTERNAL\ FOR\ TARGET\ STANDARD = M_2 \pm (2.13)\left(\frac{V_p}{6}\right)^{0.5} \qquad \text{Eq. E-8}$$

$$INTERNAL\ FOR\ HIGH\ STANDARD = M_3 \pm (2.13)\left(\frac{V_p}{6}\right)^{0.5} \qquad \text{Eq. E-9}$$

These intervals must not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable. In any event, the laboratory is responsible for the quality of the standards employed for analyses under this contract.

6.4 REQUESTING STANDARDS FROM THE EPA STANDARDS REPOSITORY

6.4.1 Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, depending on availability. The Contractor can 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.

6.5 DOCUMENTATION OF THE VERIFICATION AND PREPARATION OF CHEMICAL STANDARDS

It is the responsibility of each laboratory to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra, etc, whether produced by the laboratory or purchased from chemical supply houses, must be maintained by the laboratory and may be subject to review during on-site inspection visits. Documentation of

standards preparation may be required to be sent to EPA for verification of contract compliance. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the laboratories for a period of one year.

SECTION 7

METHOD SPECIFIC QA/QC REQUIREMENTS

7.1 This section outlines the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the volatile organic target compounds listed in Exhibit C, using the procedures in Exhibit D for samples of ambient air in canisters. This section is not intended as a comprehensive QC document, but rather as a guide to the specific QC operations that must be considered for volatile analyses using this method. At a minimum, the laboratory is expected to address these operations in preparing the QAP discussed in Section 2 and SOPs discussed in Section 3.

7.2 The specific QC operations that must be considered for volatile organics analysis include the following:

- Canister Cleaning and Certification;
- GC/MS Mass Calibration and Ion Abundance Patterns;
- GC/MS Initial and Continuing Calibration;
- Blank Analysis;
- Laboratory Control Sample Analysis;
- Sample Analysis;
- Internal Standard Responses and Retention Times; and
- Performance Evaluation (PE) Samples.

7.3 **Canister Cleaning and Certification**

7.3.1 Prior to the initial use of any canister in an analytical scheme, the laboratory shall certify the cleanliness of the canister. Before each use, the laboratory shall verify that the canister is leak-free and shall clean the canister and analyze a sample of humidified zero air to verify cleanliness.

7.3.2 Canisters must be certified clean before initial use as outlined in Exhibit D, Section 3. Results documenting initial certification are reported on Form VIII-AAVC, Canister Certification.

7.3.3 Before each use, the laboratory must clean each canister according to the procedures and meet the criteria described in Exhibit D, Section 3. Canisters cleaned and stored more than one month before use must be recleaned through one cycle of the standard three-cycle cleaning

procedure; however, no subsequent analytical confirmation of cleanliness is required before use.

7.3.4 As a blank check of the canister(s) cleanup procedure, the final humid zero air fill of 100% of the canisters must be analyzed until the cleanup system and canisters are proven reliable as outlined in Exhibit D, Section 3.

7.3.5 Only canisters determined to be leak-free according to the procedures and criteria described in Exhibit D, Section 3 shall be used by the laboratory for sampling and analysis under this document.

7.4 GC/MS Mass Calibration and Ion Abundance Patterns

7.4.1 Before analysis of samples, blanks, or standards, the laboratory must demonstrate that a given GC/MS system meets the instrument performance check standard specified in Exhibit D, Section 5. The purpose of this instrument performance check is to ensure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of bromofluorobenzene (BFB).

7.4.2 BFB analysis (once every 12 hours on each GC/MS system) is described in detail in Exhibit D, Section 5.7

7.4.3 The key ions produced during the analysis of BFB and their respective ion abundance criteria must be met.

7.4.4 The documentation includes reporting data on Form IV-AAVC, GC/MS Instrument Performance Check and Mass Calibration.

7.5 GC/MS Initial Calibration for Target Compounds

7.5.1 Prior to the analysis of samples and required blanks, and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound standards.

7.5.2 The concentrations of the initial standards for volatile target compounds and system monitoring compounds are 2, 5, 10, 20, and 50 ppbv, as described in Exhibit D.

7.5.3 The standards are to be analyzed according to the procedures and at the frequency given in Exhibit D, Section 5.8.

7.5.4 The relative response factors (RRFs) are determined according to the procedures in Exhibit D, using the assignment of internal standards to target compounds given in Exhibit D, Table D/VC-8.

7.5.5 The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors (RRF) of each target compound. The minimum RRF of each compound in the initial calibration and the percent relative standard deviation (%RSD) across all five points must meet the criteria given in Exhibit D, Section 5.8.

7.5.6 The documentation includes reporting data on Form V-AAVC, a GC/MS data system printout for the analysis of each VOC calibration standard, and the mass spectrum of each target compound.

7.6 **GC/MS Continuing Calibration for Target Compounds**

7.6.1 Once the GC/MS system has been calibrated, the calibration must be verified each 12-hour time period for each GC/MS system.

7.6.2 The standard is to be analyzed according to the procedures and at the frequency given in Exhibit D, Section 5.9, using Form VI-AAVC to report results.

7.6.3 The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum RRF of each compound in the continuing calibration and the percent difference must meet the criteria given in Exhibit D, Section 5.9.

7.7 **Internal Standard Responses and Retention Times**

7.7.1 The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results, because the quantitative determination of volatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

7.7.2 The specific compounds used as internal standards are given in Exhibit D. The concentration of each internal standard in the aliquot of sample analyzed by GC/MS must be 10 ppbv.

7.7.3 The retention time and the selected ion current profile (SICP) of each internal standard must be monitored for all analyses.

7.7.4 The area response of each internal standard from the SICP and the retention time of the internal standard are evaluated for stability, according to the procedures in Exhibit D. The area of the internal standard in a sample must not vary by more than a factor ±40 percent from the area of the same internal standard in the associated continuing calibration standard. Likewise, the retention time of an internal standard must be within ± 0.33 minutes (20 seconds) of its retention time

in the continuing calibration standard. Internal standard areas and retention times are reported on Form VII-AAVC.

7.8 Sample Analysis

7.8.1 The GC/MS must be set up per requirements of Exhibit D and meet BFB instrument performance and mass calibration criteria.

7.8.2 The internal standard must be added at a concentration outlined in Exhibit D, Section 5.12.

7.8.3 Guidelines for qualitative verification must be met as discussed in Exhibit D and outlined below:

7.8.3.1 All ions present in the standard mass spectra at a relative intensity greater than 10 percent (most abundant ion in the spectrum equals 100 percent) must be present in the sample spectrum.

7.8.3.2 The relative intensities of ions must agree within ± 20 percent between the standard and sample spectra as specified in Exhibit D. (Example: For an ion with an abundance of 50 percent in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent).

7.8.3.3 Ions greater than ten percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positive. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL report the actual value followed by a "J", e.g., "3J."

7.8.4 Guidelines for making tentative identification as presented in Exhibit D are outlined below:

7.8.4.1 Relative intensities of major ions in the reference spectrum (ions greater than ten percent of the most abundant ion) should be present in the sample spectrum.

7.8.4.2 The relative intensities of the major ions should agree within ± 20 percent. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

7.8.4.3 Molecular ions present in reference spectrum should be present in sample spectrum.

7.8.4.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination of presence of coeluting compounds.

7.8.4.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

7.8.5 Results are reported on Form I-AAVC. Other documentation includes Form VIII-AAVC, a GC/MS data system printout for the analysis of the volatile calibration standard, and the mass spectrum of each target compound.

7.9 Laboratory Method Blank Analysis

7.9.1 A laboratory method blank (LMB) is a certified canister spiked with humid zero air (air with no detectable levels of any analyte of interest), from which an aliquot is carried through the entire analytical procedure. The air aliquot volume must be equal to the sample aliquot associated with the blank. The purpose of a LMB is to determine the levels of contamination associated with the processing and analysis of samples.

7.9.2 An LMB must be analyzed once every 12 hours on each GC/MS system, as required in Exhibit D.

7.9.3 For the purposes of this protocol, an acceptable LMB analysis must result in no target compound at or above the CRQL as specified in Exhibit D, Table D/VC-1.

7.9.4 If a LMB exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. All samples processed with an LMB that is out of control must be reanalyzed at no additional cost to the agency. The laboratory manager or his designee must address problems and solutions in the SDG Narrative (Exhibit B).

7.9.5 An EPA sample number must be assigned to each LMB, as described in Exhibit B, Section 3.1. This EPA sample number is listed on Form III-AAVC Blank Summary, and the results of the blank analysis are reported on Form I-AAVC under this unique EPA sample number.

7.10 Performance Evaluation (PE) Samples

7.10.1 Performance evaluation (PE) samples are intended to assist the Agency in monitoring Contractor and method performance. The laboratory will not be informed as to which compounds are contained in the PE samples or the concentrations.

7.10.2 The laboratory shall analyze, and report the results (Form I-AAVC) of the PE sample once per sample delivery group, if available.

7.10.3 The laboratory will receive PE samples in SUMMA® canisters from the Agency. The samples will come with instructions concerning the extraction procedure required for the PE samples.

7.10.4 The laboratory must meet the following PE sample technical acceptance criteria:

7.10.4.1 The PE sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in Exhibit D.

7.10.4.2 The PE sample must be preconcentrated according to Exhibit D.

7.10.4.3 The PE sample must be prepared and analyzed with a blank that meets the blank technical acceptance criteria.

7.10.4.4 The percent recovery for each of the target compounds in the PE Sample must be within replicate precision and accuracy, as outlined in Section 5 of this Exhibit.

SECTION 8

REGIONAL DATA REVIEW

8.1 Contract laboratory data are generated to meet the specific needs of the Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of end-user, based upon functional aspects of data quality. General guidelines for data review have been developed jointly by the Region and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problems under investigation and the Regional response appropriate to the specific circumstances.

8.2 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, which is designed to identify contractual discrepancies, and the review done at EMSL/LV, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for program and laboratory administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

SECTION 9

LABORATORY EVALUATION SAMPLES

9.1 Although intralaboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in interlaboratory comparison studies conducted by the EPA. Results from the analysis of these laboratory evaluation samples will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

9.2 Sample sets may be provided to participating Contractors as frequently as on an SDG-by-SDG basis as a recognizable QC sample of known composition; as a recognizable QC sample of unknown composition; or not recognizable as a QC material. The laboratory evaluation samples may be sent either by the Regional client or the National Program Office, and may be used for contract action.

9.3 Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

9.4 At a minimum, the results are evaluated for compound identification, quantification, and sample contamination. Confidence intervals for the quantification of target compounds are based on reported values using population statistics. EPA may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample. Normally, a fraction of the compounds spiked into the sample are not specifically listed in the contract. Contractors are required to use the NIST/EPA/MSDC mass spectral library to tentatively identify a maximum number of non-target compounds in each fraction that are present above a minimal response. Tentative identification of these compounds based on contractually described spectral interpretation procedures is evaluated and integrated into the evaluation process.

9.5 A Contractor's results on the laboratory evaluation samples will determine the Contractor's performance as follows:

9.5.1 No response is required for a score of 90 percent or above.

9.5.2 For a score of 75 to 89, the Contractor shall describe the deficiency(ies) and the corrective action(s) taken in a letter to the APO, TPO, and EMSL/LV, within 14 days of receipt of notification from EPA.

9.5.3 For a score less than 75, the Contractor shall be notified by the APO or TPO concerning the remedy for its unacceptable performance. The Contractor may expect, but EPA is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, a site visit, a full data audit, analysis of remedial PE samples, and/or a contract sanction, such as a Cure Notice.

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

SECTION 10

GC/MS TAPE AUDITS

10.1 Periodically, EPA requests from Contractors the GC/MS magnetic tapes corresponding to a specific Case in order to accomplish tape audits. Generally, tape submissions and audits are requested for the following reasons:

- Program overview;
- Indication of data quality problems from EMSL/LV, SMO, or Regional data reviews;
- Support for on-site audits; and
- Specific Regional requests.

10.2 Depending upon the reason for an audit, the 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 GC/MS tapes. This function provides external monitoring of Program QC requirements and checks adherence of the Contractor to internal QA procedures. In addition, tape audits enable EPA to evaluate the utility, precision, and accuracy of the analytical methods.

10.3 The GC/MS tape shall include raw data and quantitation reports for samples, blanks, laboratory evaluation samples, initial calibrations, continuing calibration, and BFB associated with the Case requested. The specific requirements for submissions of GC/MS tapes are discussed in Exhibit B.

10.4 Upon request of the Administrative Project Officer or EMSL/LV, the required tapes and all necessary documentation shall be submitted to EPA within seven (7) days of notification.

SECTION 11

ON-SITE LABORATORY EVALUATIONS

11.1 At a frequency dictated by a contract laboratory's performance, the Administrative Project Officer, Technical Project Officer 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 Evaluation and an Evidentiary Audit.

11.2 QUALITY ASSURANCE ON-SITE EVALUATION

11.2.1 Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures. The Contractor should expect that items to be monitored will include but not be limited to the following items:

- Size and appearance of the facility;
- Quantity, age, availability, scheduled maintenance and performance of instrumentation;
- Availability, appropriateness, and utilization of 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 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, laboratory evaluation sample scores, Regional review of data, Regional QA materials, GC/MS tape audit reports, results of CCS, and data trend reports.

11.3 EVIDENTIARY AUDIT

11.3.1 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. The evidence audit is comprised of the following three activities.

11.3.1.1 Procedural Audit

The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following laboratory operations:

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

11.3.1.2 Written SOPs Audit

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

11.3.1.3 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:

- Accuracy of the document inventory;
- Completeness of the file;
- 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 Administrative Project Officer/Technical Project Officer 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

11.5.1 Following an on-site evaluation, QA and evidentiary audit reports which discuss deficiencies found during the on-site evaluation will be forwarded to the Contractor. The Contractor must discuss the corrective actions taken to resolve the deficiencies discussed during the on-site visit and discussed in the on-site reports in a letter to the Administrative Project Officer, Technical Project Officer, EMSL/LV (response to the QA report) and NEIC (response to the evidentiary report) within 14 days of receipt of the finding or within the time agreed upon between the Administrative Project Officer/Technical Project Officer and the Contractor. If SOPs are required to be written or SOPs are required to be amended, the Contractor must provide the SOPs to the Technical Project Officer, EMSL/LV (QA/technical SOPs) and NEIC (evidentiary SOPs) within 30 days of receipt of the finding or within the time agreed upon between the Administrative Project Officer/Technical Project Officer and the Contractor.

11.5.2 If the Contractor fails to take appropriate corrective action to resolve the deficiencies discussed in the on-site reports, a Contractor may expect, but the Agency is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, a follow-up site visit, a full data audit, analysis of remedial PE samples and/or contract sanction, such as a Cure Notice.

SECTION 12

QUALITY ASSURANCE AND DATA TREND ANALYSIS

12.1 Data submitted by laboratories are subject to review from several aspects: compliance with contract-required QC, usability, and full data package evaluation. Problems resulting from any of these reviews may determine the need for a GC/MS 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 Agency 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 data base. Statistical reports that evaluate specific anomalies or disclose trends in many areas, including the following, are generated from this data base:

- Laboratory Control Sample;
- Blanks;
- GC/MS Instrument Performance Checks;
- Initial and Continuing Calibration Data; and
- Other QC and Method Parameters.

12.2 Program-wide statistical results are used to rank laboratories in order to observe the relative performance of each Contractor using a given protocol against its peers. The reports are also used to identify trends within laboratories. The results of many of these trends analyses are included in overall evaluation of a Contractor's performance, and are reviewed to determine if corrective action or an on-site laboratory evaluation is indicated in order to meet the QA/QC requirements of the contract.

12.3 Contractor performance over time is monitored using these trend analysis techniques to detect departures of Contractor output from required or desired levels of QC, and to provide an early warning of Contractor QA/QC problems which may not be apparent from the results of an individual case.

12.4 As a further benefit to the Program, the data base 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 contract laboratories is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of QC and performance criteria specifications of what is routinely achievable and expected of environmental chemistry laboratories in mass production analysis

of environmental samples. This, in turn, assists the Agency in meeting its objectives of obtaining data of known and documented quality.

SECTION 13

DATA MANAGEMENT

13.1 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 should 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.

13.2 Data manually entered from hard-copy must be quality controlled 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 must be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

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

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

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

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

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

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

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

13.5 Individual(s) responsible for the following functions must be identified:

- System operation and maintenance including documentation and training;

- Database integrity, including data entry, data updating and quality control; and

- Data and system security, backup and archiving.

SECTION 14

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14.2 Office of Monitoring Systems and Quality Assurance, U.S. Environmental Protection Agency, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80, December 1980.

14.3 Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Third Edition, SW-846, November 1986.

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14.5 Health Effects Research Laboratory, U.S. Environmental Protection Agency, Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, EPA-600/8-80-036, June, 1980.

14.6 Environmental Protection Agency, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule", 40 CFR Part 136, Federal Register, Vol. 49, No. 209., pp 43234-43442, October 26, 1984.

14.7 Health Effects Research Laboratory, U.S. Environmental Protection Agency, Manual of Analytical Quality Control for Pesticides and Related Compounds In Human and Environmental Samples-Second Revision, EPA-600/2-81-059, April 1981.

14.8 Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Analytical Reference Standards and Supplemental Data: The Pesticides and Industrial Chemicals Repository, EPA-600/4-84-082, October 1984.

14.9 American Chemical Society Committee on Environmental Improvement, and Subcommittee on Environmental Analytical Chemistry, "Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry", Analytical Chemistry, Volume 52, Number 14, December 1980.

14.10 Moore, J.M. and Pearson, J.C. "Quality Assurance Support for the Superfund Contract Laboratory Program", Quality Control in Remedial Site Investigation: Hazardous and Industrial Solid Waste Testing, Fifth Volume, ASTM STP 925, C.L. Perket, ed., American Society for Testing and Materials, Philadelphia, 1986.

EXHIBIT F

EVIDENTIARY REQUIREMENTS

December, 1991

EXHIBIT F

EVIDENTIARY REQUIREMENTS

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SECTION 1

SAMPLE CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

1.1 SAMPLE IDENTIFICATION

1.1.1 To assure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

1.1.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA sample number.

1.2 CHAIN-OF-CUSTODY PROCEDURES

1.2.1 Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented.

1.2.2 A sample is under custody if the following applies:

1.2.2.1 It is in your possession.

1.2.2.2 It is in your view after being in your possession.

1.2.2.3 It was in your possession and you locked it up.

1.2.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

1.3 SAMPLE RECEIVING PROCEDURES

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available. The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.3 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

1.3.4.1 Airbills or airbill stickers.

1.3.4.2 Custody seals.

1.3.4.3 EPA custody records.

1.3.4.4 EPA traffic reports or SAS packing lists.

1.3.4.5 Sample tags.

1.3.5 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

1.3.6 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

1.3.7 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

1.3.8 The following information shall be recorded on appropriate Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected:

1.3.8.1 Condition of the shipping container.

1.3.8.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

1.3.8.3 Custody seal numbers, when present.

1.3.8.4 Condition of the sample bottles.

1.3.8.5 Presence or absence of airbills or airbill stickers.

1.3.8.6 Airbill or airbill sticker numbers.

1.3.8.7 Presence or absence of EPA custody records.

1.3.8.8 Presence or absence of EPA traffic reports or SAS packing lists.

1.3.8.9 Presence or absence of sample tags.

1.3.8.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

1.3.8.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

1.3.8.12 Problems or discrepancies.

1.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 2

DOCUMENT CONTROL PROCEDURES

The goal of the laboratory document control program is to assure that all documents for a specified SDG will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

2.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

2.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the laboratory may exercise the option of using only laboratory or EPA

sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

2.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be crossed out.

2.2 CONSISTENCY OF DOCUMENTATION

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

2.2.2 All copies of laboratory documents shall be complete and legible.

2.2.3 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

2.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURES

2.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

2.3.2 All documents relevant to each SDG, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

2.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 of Exhibit E is an example of a document inventory.

2.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

2.5 SHIPPING DATA PACKAGES AND CSF

2.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

2.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

2.5.3 A copy of the transmittal letter for the CSF will be sent to NEIC and SMO.

2.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one original FORM AADC-1 for each shipping container.

2.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

2.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

2.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 3

STANDARD OPERATING PROCEDURES

The Contractor must have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, tracking the analysis of samples, and assembly of completed data.

3.1 SPECIFICATIONS FOR WRITTEN STANDARD OPERATING PROCEDURES

3.1.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation.

3.1.2 The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits. The Contractor must have written standard operating procedures (SOPs) for:

3.1.2.1 Sample receipt and logging.

3.1.2.2 Sample storage.

3.1.2.3 Preventing sample contamination.

3.1.2.4 Security for laboratory and samples.

3.1.2.5 Traceability of standards.

3.1.2.6 Maintaining instrument records and logbooks.

3.1.2.7 Sample analysis and data control systems.

3.1.2.8 Glassware cleaning.

3.1.2.9 Technical and managerial review of laboratory operation and data package preparation.

3.1.2.10 Internal review of contractually-required quality assurance and quality control data for each individual data package.

3.1.2.11 Sample analysis, data handling, and reporting.

3.1.2.12 Chain-of-Custody.

3.1.2.13 Document control, including Case file preparation.

3.1.3 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.1.4 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.1.4.1 Presence or absence of EPA chain-of-custody forms.

3.1.4.2 Presence or absence of airbills or airbill stickers.

3.1.4.3 Presence or absence of EPA Traffic Reports or SAS packing lists.

3.1.4.4 Presence or absence of custody seals on shipping and/or sample containers and their condition.

3.1.4.5 Custody seal numbers, when present.

3.1.4.6 Presence or absence of sample tags.

3.1.4.7 Sample tag ID numbers.

3.1.4.8 Condition of the shipping container.

3.1.4.9 Condition of the sample container.

3.1.4.10 Verification of agreement or nonagreement of information on receiving documents and sample containers.

3.1.4.11 Resolution of problems or discrepancies with the SMO.

3.1.4.12 The definition of any terms used to describe sample condition upon receipt.

3.1.5 The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.1.6 The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the

following:

3.1.6.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.1.6.2 A description of the documentation used to record instrument calibration and other QA/QC activities.

3.1.6.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.1.7 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.1.8 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.1.9 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions. The Contractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.

3.1.10 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure.

3.1.11 The Contractor shall have written SOPs for laboratory safety.

3.1.12 The Contractor shall have written SOPs for cleaning of glassware used in preparing and analyzing samples under this contract.

3.1.13 The Contractor shall have SOPs for traceability of standards used in sample analysis QA/QC.

3.2 HANDLING OF CONFIDENTIAL INFORMATION

3.2.1 A Contractor conducting work under this contract may receive EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed

under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.2.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.2.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

EXHIBIT G

GLOSSARY OF TERMS

December, 1991

EXHIBIT G

GLOSSARY OF TERMS

Aliquot - A measured portion of a field sample taken for analysis.

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

Analysis Group - An analysis group is a set of no more than twenty analytical samples (as defined below) for the purpose of method Quality Assurance/Quality Control (QA/QC), such that the QA/QC required by Exhibit E is, at a minimum, prepared and analyzed at a frequency of once per twenty analytical samples.

Analysis Replicate - A single analytical sample prossed through the analytical preparation method and analyzed in replicate.

Analyte - The compound an analysis seeks to determine; the compound of interest.

Analytical Sample - Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), duplicate samples, and laboratory control sample (LCS).

ASTM Type II Water - Distilled water with a conductivity of less than 1.0 μmho/cm at 25°C. For additional specifications refer to ASTM D1193-77, "Standard Specification for Reagent Water".

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

Batch - A group of samples prepared at the same time.

Calibration - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. Calibration procedures differ for the various methods included in this document.

Calibration Standards - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions are not subjected to the preparation method but contain the same matrix as the sample preparations to be analyzed.

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

Continuing Calibration - Analytical standard run at least every 12 hours to verify the calibration of the analytical system.

Contract Required Quantitation Limit (CRQL) - Minimum level of quantitation acceptable under the contract. Generally defined as 3.3 (or more) times the standard deviation of seven replicate analyses of the method blank.

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

Correlation Coefficient - A number (r) which indicates the degree of dependence between two variables (e.g., concentration - absorbance). The more dependent they are the closer the value to one. Determine on the basis of the least squares line.

Cryogen - A liquified gas used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp - 195.8°C).

Data System - For the purpose of this document, computer system that allows the continuous acquisition and printout of time vs. intensity data throughout the chromatographic program.

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

Deuterated Chemicals - Those chemicals which contain deuterium (hydrogen isotope that is twice the mass of hydrogen); used as tracers for system quality assurance.

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

Dynamic Calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

Dynamic Dilution - Means of preparing calibration mixtures in which standard gas(es) from pressurized cylinders are continually blended with humidified zero air in a manifold so that a flowing stream of calibration mixture is available at the inlet of the analytical system.

EBCDIC - Extended Binary Coded Decimal Interchange Code.

External Standards - Target analytes analyzed at a known concentration prior to sample analysis, to determine response factors.

Field Sample - A portion of material received to be analyzed that is contained

in single or multiple containers and identified by a unique EPA Sample Number.

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

In-House - At the Contractor's facility.

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

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

Internal Standards - Analytes added to every standard, blank, sample (for VOAs) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target analysis.

Laboratory - Synonymous with Contractor as used herein.

Laboratory Control Sample - Aliquot spiked with known concentration of specific analytes and subjected to the entire analytical procedure in order to monitor method and contractor performance.

Laboratory Receipt Date - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

Linear Range - The concentration range over which the analytical curve remains linear. The range of the instrument for a specific analyte, as determined using calibration standards. The upper limit of this linear range (determined at each analysis) is the highest concentration calibration standard that has a determined value within 10% of the known value.

Mass Spectral Interference - Defined as the inability to detect the internal standard quantification ion due to presence of high levels of mass spectral "noise" at the same mass.

Matrix - The predominant material of which the sample to be analyzed is composed.

Megabore® Column - One of two types of capillary columns, the other being the narrow bore, for the analysis of target compounds under this contract.

Method Detection Limit (MDL) - The chemical concentration that produces a signal, due to an analyte, which is equal to the student $t_{.99}$ times the standard deviation of a series of measurements on at least seven separate method blanks. In practice, a method detection limit will be substantially higher than an instrumental detection limit. The method detection limit for

metals is $t_{.99}$ times the standard deviation of seven method blank analyses. Of course, all spectral background techniques must be operative and the same integration times must be utilized as when actual samples are analyzed.

MS-SCAN - The gas chromatograph (GC) is coupled to a mass selective detector where the instrument is programmed to acquire all mass for target analytes and to disregard all others.

Narrative (SDG Narrative) - Portion of the data package which includes laboratory, contract, SDG 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.

Narrow-Bore Capillary Column - One of two capillary columns, the other being the wide-bore (Mega-bore®) capillary column, for the analysis of compounds under this contract.

Performance Evaluation (PE) Sample - A sample of known composition provided by EPA for Contractor analysis. Used by EPA to evaluate Contractor performance.

Protocol - A compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

Qualitative Accuracy - The ability of an analytical system to correctly identify compounds.

Quantitative Accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

Reconstructed Ion Chromatogram (RIC) - A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Recovery - A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

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

Relative Response Factor (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \qquad \text{Eq. F-1}$$

where:
A = area of the characteristic ion measured
C = concentration
is = internal standard
x = analyte of interest

Resolution - Also termed separation, the separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Retention Time (RT) - The time to elute a specific chemical from a chromatographic column for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until its maximum concentration appears at the detector.

Retention Time Window - Retention time window is determined for each analyte of interest and is the time from injection to elution of a specific chemical from a chromatographic column. The window is determined by three injections of a single component standard over a 24-hour period as plus or minus three times the standard deviation of the absolute retention time for that analyte.

Run - A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract.

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

Sample Delivery Group (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 samples within a Case; or
- Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

Samples may be assigned to Sample Delivery Groups by sample collection method (i.e., all Tenax® tubes in one SDG, all canisters in another).

Sample Number (EPA Sample Number) - A unique identification number designated by EPA for each sample. The EPA Sample Number appears on the sample Traffic

Report which documents information on that sample.

Selected Ion Current Profile (SICP) - A plot of ion abundance vs. time or scan number for ions of a specified mass.

Standard Analysis - An analytical determination made with known quantities of target compounds; used to determine response factors.

Static Calibration - Calibration of an analytical system with known concentrations of calibration gas, obtained from a source such as gas cylinders or prepared from standard stock solutions.

Stock Solution - A standard solution which can be diluted to derive other standards.

Surrogates (Surrogate Standard) - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media.

Tentatively Identified Compounds (TIC) - Compounds detected in samples that are not target compounds, internal standards or surrogate standards. Up to 10 peaks are subjected to mass spectral library searches for tentative identification.

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 is used for documenting sample condition and receipt by the laboratory.

Twelve-Hour Time Period - The twelve (12) hour time period for GC/MS system tuning, standards calibration (initial or continuing calibration) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of compliant tune. The time period ends after 12 hours has 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.

Volatile Compounds - Target compounds with normal vapor pressures $\geq$ 0.1 mm Hg.

ATTACHMENT IV

USEPA CONTRACT LABORATORY PROGRAM

VOLATILE ORGANICS ANALYSIS OF AMBIENT AIR ON TENAX®

December, 1991

Revision VTAA01.0

VOLATILE ORGANICS ANALYSIS OF
AMBIENT AIR ON TENAX®

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PREFACE

The purpose of this contract is to provide the U.S. Environmental Protection Agency (EPA) with chemical analytical services, quality control procedures, and an analysis structure which will generate data of known and documented quality. This document was developed with the guidance of the Air Toxics Workgroup to ensure that the needs of regional, state, and local air pollution programs are addressed.

The samples to be analyzed are of ambient air collected on Tenax® at or in the vicinity of known or suspected hazardous waste sites and may contain potentially hazardous organic and inorganic material at significant concentrations. The Contractor should be aware of the potential hazards associated with the handling and analyses of these samples. It is the Contractor's responsibility to take all necessary measures and precautions to ensure the health and safety of its employees. The Contractor is responsible for providing a safe working environment and making its employees aware of the potential hazards of working with and analyzing these samples.

Procedures specified herein shall be used in the preparation of Tenax® cartridges and analysis of air samples for the presence and quantitation of certain volatile organic compounds (VOCs). The Contractor shall employ safe handling procedures and generally accepted laboratory practices in the performance of contract requirements and shall follow the quality assurance and quality control (QA/QC) program specified herein.

The data obtained under this contract will be used by EPA to determine the existence and extent of risk posed by hazardous waste disposal sites to the public, to individuals involved in Superfund site cleanups, and to the environment. The data may be used in civil and/or criminal litigation which requires the strictest adherence to chain-of-custody protocol, document control, and quality assurance procedures.

DRAFT

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR VOLATILE ORGANICS ANALYSIS OF AMBIENT AIR ON TENAX®

December, 1991

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR VOLATILE ORGANICS ANALYSIS OF AMBIENT AIR ON TENAX®

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SECTION 1

GENERAL REQUIREMENTS

1.1 The Contractor shall employ procedures specified in this contract in the preparation and analysis of the ambient air samples for the presence and quantitation of the organic compounds listed in Exhibit C.

1.2 The Contractor shall use proven techniques to identify and measure the organic compounds presented in the Target Compound List (TCL) as specified in Exhibit C. The Contractor shall perform sample preparation and analysis procedures as prescribed in Exhibit D, and meet specified sample preservation and holding time requirements.

1.3 For all samples analyzed under this contract, the Contractor shall adhere to the QA/QC protocols specified in Exhibit E and abide by the evidentiary protocols specified in Exhibit F.

1.4 Following sample analysis, the Contractor shall perform data reduction and shall report analytical activities, sample data, and quality control documentation as designated in Exhibit B. Exhibit B contains all reporting and deliverables requirements for this contract, including copies of the data reporting forms and form instructions guide.

1.5 To ensure proper understanding of the language in this contract, Exhibit G contains a glossary of terms. When a term is used in the text without explanation, the glossary meaning shall be applicable. Glossary definitions do not replace or take precedence over specific information included in the document text.

1.6 The samples to be analyzed by the Contractor are from known or suspected hazardous waste sites and may contain hazardous organic and/or inorganic materials at high concentration levels. The Contractor should be aware of the potential hazards associated with the handling and analysis of these samples. It is the Contractor's responsibility to take all necessary measures to ensure the health and safety of its employees. It is also the Contractor's responsibility to follow appropriate disposal procedures according to state and federal regulations.

1.7 In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, as it may be used to make major decisions regarding public health and environmental welfare. In addition, data generated under this contract may be used in litigation against potentially responsible parties in the enforcement of Superfund legislation.

SECTION 2

SPECIFIC REQUIREMENTS

For each sample, the Contractor shall perform the following tasks:

2.1 TASK I: RECEIVE AMBIENT AIR SAMPLES ON TENAX® CARTRIDGES

2.1.1 The Contractor shall receive and handle samples under the chain-of-custody and document control procedures described in Exhibit F.

2.1.2 The Contractor shall provide the required analytical expertise and instrumentation for analyses of the TCL compounds equal to or lower than the quantitation limits specified in Exhibit C. In Exhibit D, EPA provides the Contractor with an appropriate set of analytical procedures that shall be used.

2.1.3 The Contractor shall analyze samples within the maximum holding times specified in Exhibit D, even if these times are less than the maximum data submission time allowed in this contract.

2.1.4 The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the preparation and analysis with appropriate caution. The Contractor shall be responsible for all necessary measures and precautions to ensure the health and safety of laboratory employees.

2.2 TASK II: ANALYZE SAMPLES FOR THE IDENTIFICATION AND QUANTITATION OF SPECIFIC COMPOUNDS

2.2.1 For each sample received, the Contractor shall be required to perform the analyses described in Exhibit D. The documentation that accompanies the sample(s) to the Contractor facility shall indicate specific analytical requirements for that sample or set of samples.

2.2.2 Exhibit D specifies the analytical procedures that shall be used. Exhibit D contains instructions and references for the analysis of ambient air samples containing low-to-medium concentrations of volatile organics for GC/MS analysis. GC/MS may use automated computer programs to facilitate the identification of organic compounds.

2.2.3 For the purpose of this contract, a full sample analysis is defined as analysis for all of the TCL constituents identified in Exhibit C in accordance with the methods in Exhibit D and performance of related QA/QC as specified in Exhibit D and Exhibit E. Laboratory Control Samples (LCS) analyses shall be considered a separate full sample analysis. All other QA/QC requirements are considered an inherent part

of this contract and are included in the contract sample unit price.

2.2.4 The volatile compounds analyzed by GC/MS techniques and initially identified shall be verified by an analyst competent in the interpretation of mass spectra by comparison of the suspect mass spectra to the mass spectrum of a standard of the suspected compound. This procedure requires the use of multiple internal standards. Two criteria must be satisfied to verify the identifications:

2.2.4.1 Elution of the sample component at the same GC relative retention time as the standard component.

2.2.4.2 Correspondence of the sample component and standard component mass spectra.

2.2.5 For each sample analysis, the Contractor shall conduct mass spectral library searches of non-target compound sample components to determine tentative compound identifications as follows:

2.2.5.1 For each volatile organics analysis, the Contractor shall conduct a search to determine the possible identity of up to 10 organic compounds of greatest concentration which are not internal standards, surrogate compounds, and not listed in Exhibit C.

2.2.5.2 In performing searches, the most recent release of the National Institute of Standards and Technology (NIST)/EPA/MSDC mass spectral library must be used.

NOTE: Substances with responses of less than 10 percent of the nearest internal standard are not required to be searched in this fashion.

2.2.5.3 Only after visual comparison of sample spectra with the spectra from the library searches will the mass spectral interpretation specialist assign a tentative identification. If the compound does not meet the identification criteria, it shall be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, they also should be included.

2.3 TASK III: PERFORM REQUIRED QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

2.3.1 All specific QA/QC procedures prescribed in Exhibits D and E shall be strictly adhered to by the Contractor. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance

with Exhibit B requirements.

2.3.2 The Contractor shall establish and use on a continuing basis QA/QC procedures including the daily or (as required) more frequent use of standard reference solutions from EPA, NIST, or secondary standards traceable thereto, where available at appropriate concentrations (i.e., standard solutions designed to ensure that operating parameters of equipment and procedures, from sample receipt through identification and quantitation, produce reliable data). Exhibits D and E provide specific QA/QC requirements.

2.3.3 Additional QA/QC shall be required quarterly or more frequently, i.e., with each Case or Sample Delivery Group (SDG), in the form of Laboratory Control Samples (LCS) and Performance Evaluation (PE) samples for volatile organics submitted to EPA for Contractor analysis, and in the form of verification of instrument parameters, as described in Exhibit E.

2.3.3.1 EPA has provided to the Contractor formats for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in this contract and within the time specified in the Contract Performance/Delivery Schedule.

2.3.3.2 Use of formats other than those designated by EPA will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government will be required.

2.3.3.3 Computer generated forms may be submitted in the hardcopy data package(s) provided that the forms are in exact EPA format. This means that the order of data elements is the same as on each EPA required form, including form numbers and titles, page numbers and header information, columns, and lines.

2.3.4 The Contractor shall provide analytical equipment and technical expertise for this contract as specified by the following:

2.3.4.1 Gas chromatograph/mass spectrometer (GC/MS) data system capable of meeting all the terms and conditions of the Contract with the following requirements:

2.3.4.1.1 The computer shall be interfaced by hardware to the mass spectrometer and be capable of acquiring continuous mass scans for the duration of the chromatographic program.

2.3.4.1.2 The computer shall be equipped with mass storage devices for saving all data from the GC/MS runs.

2.3.4.1.3 Computer software shall be available to allow searching GC/MS runs for specific ions and plotting the intensity of the ions with respect to time or scan number.

2.3.4.1.4 A computer data system must be interfaced to the MS that allows the continuous acquisition and storage, on machine-readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP) or Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

2.3.4.1.5 The GC/MS shall be equipped with a GC to MS interface capable of extending a fused silica capillary column into the ion source. The column is to be 50 meters long by 0.25 to 0.53 mm I.D. 100% methyl silicone or 5% phenyl, 95% methyl silicone capillary column, or equivalent.

2.3.4.2 The Contractor shall use a magnetic tape storage device capable of recording data and suitable for long-term, off-line storage. The Contractor shall retain all raw GC/MS data acquired under this contract on magnetic tape in appropriate instrument manufacturer's format. The Contractor is required to retain the magnetic tapes with associated hardcopy tape logbook identifying tape contents (see Exhibit B) for 365 days after data submission. During that time, the Contractor shall submit tapes and logbook within seven days of request, as specified in the Contract Performance/Delivery Schedule.

2.3.4.3 The Contractor shall have a computerized MS library search system capable of providing a forward comparison, using the standard spectra contained in the mass spectral library. The 1985 (or most recent) release of the NIST library (containing 42,261 spectra) must be used.

2.3.4.4 The system shall provide a numerical ranking of the standard spectra most closely corresponding to the sample spectra examined, and the data system shall have software capable of removing background signals from spectra.

2.3.4.5 The Contractor shall have, in-house and operable, a device capable of analyzing volatile organics as described in Exhibit D.

2.3.4.6 The Contractor shall have, in-house, the appropriate standards for <u>all</u> target compounds listed in Exhibit C prior to accepting any samples from the Sample Management Office (SMO). Standards provided by EPA for use in the Preaward Performance Evaluation may not contain all the target compounds and thus shall <u>not</u> be used for routine analyses unless or until they have been supplemented with commercially-available standard materials.

2.3.5 The minimum functional requirements necessary to meet the terms and conditions of this contract are listed below. The Contractor shall designate and use qualified key personnel to perform these functions. The EPA reserves the right to review personnel qualifications and experience.

2.3.5.1 Project Manager

2.3.5.2 GC/MS Laboratory Supervisor

2.3.5.3 Quality Assurance Officer

2.3.5.4 Systems Manager

2.3.5.5 Programmer Analyst

2.3.5.6 GC/MS Operators

2.3.5.7 Mass Spectral Interpreter

2.3.5.8 Chemist (back-up)

NOTE: The Contractor shall designate a Sample Custodian and a Document Control Officer.

2.3.6 The Contractor shall respond within 10 days to requests from data recipients for additional information or explanations that result from the Government's inspection activities.

2.3.7 The Contractor is required to retain unused sample volumes and used sample containers for a period of 60 days after data submission unless otherwise instructed in Exhibit B or Exhibit D.

2.3.8 The Contractor shall adhere to the chain-of-custody and document control procedures described in Exhibit F. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported in the Complete Case File Purge (Exhibit B).

2.3.9 Sample shipments to the Contractor's facility will be scheduled and coordinated by SMO, acting on behalf of the Administrative Project Officer (APO). The Contractor shall communicate with SMO personnel by telephone as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

2.3.10 If there are problems with the samples (e.g., mixed media, containers broken) or sample documentation/paperwork (e.g., Traffic Reports not with shipment, or sample and Traffic Report numbers do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

2.3.11 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case. A Case consists of one or more SDG(s). An SDG is defined by the following:

2.3.11.1 Each Case of field samples received, or

2.3.11.2 Each 20 field samples within a Case, or

2.3.11.3 Each seven 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).

2.3.12 Data for all samples in an SDG must be submitted together (in one package) in the order specified in Exhibit B. The SDG number is the EPA number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number is the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. The SDG number is reported on all data reporting forms. The SDG Receipt Date is the day that the last sample in the SDG is received.

2.3.13 The Contractor is responsible for identifying each SDG as samples are received, through proper sample documentation (see Exhibit B) and communication with SMO personnel.

2.3.14 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report (TR) form bearing

the sample number and descriptive information regarding the sample. The Contractor shall complete and sign the TR, recording the date of sample receipt and sample condition on receipt for each sample container.

2.3.15 The Contractor shall submit signed copies of TRs for all samples in an SDG to SMO within three calendar days following receipt of the last sample in the SDG. TRs shall be submitted in SDG sets (i.e., all TRs for a SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

2.3.16 EPA Case numbers (including SDG numbers) and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.

2.3.17 Samples will be routinely shipped directly to the Contractor through a delivery service. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays and holidays. As necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station, or other carrier service within the Contractor's geographical area.

2.3.18 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

SECTION 3

DETAILED TECHNICAL & MANAGEMENT REQUIREMENTS

The Contractor shall have the following technical and management capabilities:

3.1 PERSONNEL

3.1.1 Project Manager

3.1.1.1 Responsible for all technical efforts of the laboratory to meet all terms and conditions of the contract.

3.1.1.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.1.3 Experience: Minimum of three years of laboratory experience, including at least one year in a supervisory position.

3.1.2 GC/MS Laboratory Supervisor

3.1.2.1 Responsible for all technical efforts of the GC/MS laboratory to meet all terms and conditions of the contract.

3.1.2.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.2.3 Experience: Minimum of three years of laboratory experience in operating a GC/MS, including at least one year in a supervisory position.

3.1.3 Quality Assurance Officer

3.1.3.1 Responsible for overseeing the quality assurance aspects of data generation and reporting directly to upper management.

3.1.3.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.3.3 Experience: Minimum of three years of laboratory experience, including at least one year of applied experience with QA principles and practices in an analytical laboratory.

3.1.4 Systems Manager

3.1.4.1 Responsible for the management and quality control of all computing systems (hardware, software, documentation, and procedures), generating, updating, and performing quality control on

automated deliverables.

3.1.4.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, database management systems, or systems requirements analysis.

3.1.4.3 Experience: Minimum of three years experience in data or systems management or programming including one year experience with software used for data management and generation of deliverables.

3.1.5 Program Analyst

3.1.5.1 Responsible for the installation, operation, and maintenance of software and programs; generating, updating, and performing quality control procedures on analytical databases and automated deliverables.

3.1.5.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, information systems, or systems requirements analysis.

3.1.5.3 Experience: Minimum of two years experience in systems or applications programming including one year of experience with software used for data management and generation of deliverables.

3.1.6 Gas Chromatography/Mass Spectrometer (GC/MS) Operator

3.1.6.1 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.6.2 Experience: Minimum of one year of experience in operating and maintaining GC/MS instruments in conjunction with the education requirement; or in lieu of education requirement, three additional years of experience in operating and maintaining GC/MS instrumentation.

3.1.7 Mass Spectral Interpreter

3.1.7.1 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline with specialized training in GC/MS.

3.1.7.2 Experience: Minimum of two years of applied experience with GC/MS analysis of environmental samples.

3.1.8 Technical Staff Redundancy

3.1.8.1 In order to ensure continuous operations to accomplish the required work as specified by the contract, the bidder shall have a minimum of one chemist available at all times as a back-up technical

person with the following qualifications.

3.1.8.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.8.3 Experience: Minimum of one year of experience in each of the following areas: GC/MS operation and maintenance.

3.2 FACILITIES

The adequacy of the facilities and equipment is as important as the technical staff for accomplishing the required work as specified by the EPA contract.

3.2.1 Sample Receipt Area

Adequate, contamination-free, well-ventilated work space with chemical resistant bench top shall be available for receipt and safe handling of EPA samples.

3.2.2 Storage Area

Sufficient refrigerator space to maintain unused EPA sample volume for up to 60 days after data submission shall be provided. Volatile samples must be stored in a refrigerator used only for storage of volatile samples from this contract. Samples must be stored in an atmosphere demonstrated to be free from all potential contaminants. Samples and standards must be stored separately.

3.2.3 Sample/Standard Preparation Area

Adequate, contamination-free, well-ventilated work space shall be provided with:

3.2.3.1 Benches with chemical resistant tops.

3.2.3.2 Exhaust hoods.

3.2.3.3 Glove box or isolated area in which to prepare standard materials.

3.2.3.4 Source of distilled or demineralized organic-free water.

3.2.3.5 Analytical balance(s) located away from draft and rapid change in temperature.

3.3 INSTRUMENTATION

At a minimum, the Contractor shall have the following instruments

operative at the time of the Preaward Site Evaluation and committed for the full duration of the contract.

3.3.1 100 Samples/Month Capacity Requirements

No. of Instrument(s)	Type of Instrument
1	GC/MS

NOTE: The Contractor shall have one (1) complete GC/MS system available (operational) at all times as a back-up system. These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.2 200 Samples/Month Capacity Requirements

No. of Instrument(s)	Type of Instrument
2	GC/MS

NOTE: These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.3 Instrument Specifications

Further information on instrument specifications and required ancillary equipment may be found in Exhibit D and other Exhibits in this contract.

3.4 DATA HANDLING AND PACKAGING

The Contractor shall be able to submit reports and data packages as specified in Exhibit B. To complete this task, the Contractor shall be required to:

3.4.1 Provide space, tables, and copy machines to meet the contract requirements.

3.4.2 Designate personnel responsible for report preparations and submission.

3.5 LABORATORY MANAGEMENT CAPABILITY

The Contractor shall have an organization with well-defined responsibilities for each individual in the management system to ensure

sufficient resources for EPA contract(s) and to maintain a successful operation. To establish this capability, the Contractor shall designate personnel to carry out the following responsibilities for the EPA contract. Functions include, but are not limited to, the following:

3.5.1 Technical Staff

Responsible for all technical efforts for the EPA contract such as sample analysis, sample validation, and troubleshooting of all instruments.

3.5.2 Project Manager

Responsible for overall aspects of EPA contract(s) (from sample receipt through data delivery) and shall be the primary contact for EPA Headquarters Administrative Project Officer (APO) and Regional Technical Project Officers (TPO).

3.5.3 Sample Custodian

Responsible for receiving the EPA samples (logging, handling, and storage).

3.5.4 Quality Assurance Officer

Responsible for overseeing the quality assurance aspects of the data and reporting directly to upper management.

3.5.5 Document Control Officer

Responsible for ensuring that all documents generated are placed in the Complete SDG File for inventory and are delivered to the appropriate EPA Region or other receiver as designated by EPA.

EXHIBIT B

REPORTING

AND

DELIVERABLES REQUIREMENTS

December, 1991

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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SECTION 1

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

The following table summarizes the contract reporting and deliverables requirements specified in the Contract Schedule and includes the distribution of each deliverable.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The EPA APO or SMO will notify the Contractor in writing of such changes when they occur.

Item	No. of Copies	Schedule and Delivery	Distribution		
			(1)	(2)	(3)
Updated Standard Operating Procedures (SOPs)	2	45 days after contract award.		X	X
*Sample Traffic Reports	1	***3 days after receipt of last sample in Sample Delivery Group (SDG).	X		
**Sample Data Summary Package	1	14 days after receipt of last sample in SDG.	X		
**Sample Data Package including the Performance Evaluation (PE) Sample	3	35 days after receipt of last sample in SDG	X	X	X
Results of Intercomparison Study/Preaward Performance Evaluation (PPE) Sample	2	35 days after receipt of last sample in SDG	X	X	
Complete SDG File	1	35 days after data receipt of last sample in SDG.		X	
GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO.	As Directed		
****Quality Assurance Plan	1	Submit copy within 7 days by written request by APO.	As Directed		

Distribution

(1) Sample Management Office
(2) Environmental Monitoring Systems Laboratory-Las Vegas
(3) USEPA Region

* Also required in each Sample Data Package.

** Concurrent delivery of these items to all recipients is required.

*** An SDG is a group of samples within a Case, received over a period of seven days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. (See Exhibit A, Task III, for further description).

**** See Exhibit E for description.

NOTE: As specified in the Contract Schedule in the IFB (Government Furnished Supplies and Materials), unless otherwise instructed by SMO, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of analytical data.

Address

(1) USEPA Contract Laboratory Program
Sample Management Office
P.O. Box 818
Alexandria, VA 22313

For overnight delivery service, use street address:

300 North Lee Street
Alexandria, VA 22313

(2) USEPA Environmental Monitoring Systems Laboratory
P.O. Box 93478
Las Vegas, NV 89193-3478
ATTN: Data Audit Staff

For overnight delivery service, use street address:

944 E. Harmon, Executive Center
Las Vegas, NV 89109
ATTN: Data Audit Staff

(3) USEPA REGIONS:

SMO, acting on behalf of the EPA APO, will provide the Contractor with the list of addresses for the 10 EPA Regions. SMO will provide the Contractor with updated Regional name/address lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The APO will notify the Contractor in writing of such changes when they occur.

SECTION 2

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

2.1 INTRODUCTION

2.1.1 The Contractor shall provide reports and other deliverables according to the schedule specified in Section F of the IFB, "SCHEDULE INFORMATION." The required content and form of each deliverable is described in this Exhibit.

2.1.2 All reports and documentation shall be:

2.1.2.1 Legible;

2.1.2.2 Clearly labeled and completed in accordance with instructions in this Exhibit;

2.1.2.3 Arranged in the order specified in this section;

2.1.2.4 Paginated; and

2.1.2.5 Single-sided.

2.1.3 If submitted documentation does not conform to the above criteria, the Contractor will be required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the Government.

2.1.4 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation or through an APO/TPO action, the data shall be clearly marked as "ADDITIONAL DATA" and shall be sent to all three contractual data recipients (SMO, EMSL-LV, and Region). A cover letter shall be included that describes which data are being delivered, to which EPA Case(s) the data pertain, and who requested the data.

2.1.5 Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with the required documentation and contains the required data forms in EPA-specified format.

2.1.6 Descriptions of the requirements for each deliverable item cited in the Contract Performance/Delivery Schedule (see Section F of the IFB "SCHEDULE INFORMATION") are specified in this Section. Items submitted concurrently must be arranged in the order listed. Additionally, the components of each item must be arranged in the order presented herein.

2.2 UPDATED STANDARD OPERATING PROCEDURES

2.2.1 The Contractor shall submit updated copies of all required Standard Operating Procedures (SOPs) that were submitted with the Prebid Performance Evaluation (PPE) sample results. The updated SOPs must address any and all issues of laboratory performance and operation identified by the Contractor in the review of the PPE sample data and the evaluation of Bidder-Supplied Documentation.

2.2.2 The Contractor must supply SOPs for the following.

2.2.2.1 Evidentiary SOPs.

2.2.2.2 Sample receipt and logging.

2.2.2.3 Sample storage area.

2.2.2.4 Preventing sample contamination.

2.2.2.5 Security for laboratory and samples.

2.2.2.6 Traceability/equivalency of standards.

2.2.2.7 Maintaining instrument records and bound logbooks.

2.2.2.8 Glassware cleaning.

2.2.2.9 Technical and managerial review of laboratory operation and data package preparation.

2.2.2.10 Internal review of contractually-required QA/QC data for each individual data package.

2.2.2.11 Sample analysis, data handling, and data reporting.

2.2.2.12 Chain-of-custody and document control, including case file preparation.

2.2.2.13 Sample data validation/self-inspection system, including:

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to ensure that hardcopy data are complete and compliant

with the requirements in Exhibit B;

- Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.);
- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

2.2.2.14 Data Handling.

2.2.2.14.1 Data Management procedures are defined as written procedures that are clearly defined for all databases and files used to generate or re-submit deliverables specifying the acquisition or entry, update, correction, deletion, storage, and security of computer-readable data and files. Key areas of concern include: system organization including personnel and security, demonstration, operations, traceability, and quality control.

2.2.2.14.2 Data manually entered from hardcopy must be subjected to quality control procedures and error rates estimated.

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

- Justification or rationale for the change;
- Initials of the person making the changes or changes. Data changes must be identified when generating the deliverables;
- Changed documentation must be retained according to the schedule of the original deliverable;
- Resubmitted deliverables must be reinspected as a part of the laboratory's internal inspection process prior to submission. The entire deliverable and not just the changes must be reinspected;
- The laboratory manager must approve changes to originally

submitted deliverables; and

- Documentation of data changes may be requested by laboratory auditors.

2.2.2.14.4 Life cycle management procedures must be applied to computer systems used to generate and edit contract deliverables. Such systems must be thoroughly tested and documented prior to utilization.

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

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

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

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

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

- System operation and maintenance including documentation and training; and

- Database integrity including data entry, data updating and QC.

2.2.2.14.10 Data and system security, backup, and archiving.

2.3 SAMPLE TRAFFIC REPORTS

2.3.1 The original sample TR page marked "Lab Copy for Return to SMO" shall be submitted to SMO with laboratory receipt information and signed in original Contractor signature, for each sample in the SDG.

2.3.2 TRs shall be submitted in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

2.3.3 The SDG Cover Sheet shall contain the following items:

2.3.3.1 Laboratory name.

2.3.3.2 Contract number.

2.3.3.3 Sample analysis price - full sample price from contract.

2.3.3.4 Case number.

2.3.3.5 List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations), and the "last" sample received would be the highest sample number (considering both alpha and numeric designations).

2.3.4 Each TR shall be clearly marked with the SDG Number and the EPA sample number of the first sample in the SDG. This information shall be entered below the laboratory receipt date on the TR. The TR for the last sample received in the SDG shall be clearly marked "SDG - FINAL SAMPLE."

2.3.5 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the laboratory shall make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.

2.4 SAMPLE DATA SUMMARY PACKAGE

2.4.1 As specified in the Delivery Schedule, one Sample Data Summary Package shall be delivered to SMO concurrently with delivery of other required sample data. The Sample Data Summary Package shall be submitted separately (i.e., separated by rubber bands, clips or other means) directly preceding the Sample Data Package.

2.4.2 The Sample Data Summary Package shall contain data for samples in one SDG of the Case, as follows:

2.4.2.1 Cover Page.

2.4.2.2 By sample, tabulated target compound results (FORM I-AAVT) and tentatively identified compounds (FORM I-AAVT-TIC).

2.4.2.3 Laboratory Control Sample results (FORM III-AAVT).

2.4.2.4 Blank summary (FORM II-AAVT) and tabulated results (FORM I) including tentatively identified compounds (FORM I-AAVT-TIC).

2.4.2.5 Initial and Continuing Calibration Data (FORM V-AAVT and FORM VI-AAVT).

2.4.2.6 Internal standard area and retention time data (FORM VII-AAVT).

2.4.2.7 Surrogate Recovery (FORM IX-AAVT).

2.4.2.8 Analytical Sequence (FORM X-AAVT).

2.5 SAMPLE DATA PACKAGE

2.5.1 The sample data package shall be complete, consecutively paginated, and shall include data for analysis of all samples in an SDG such as field samples, blanks, and laboratory control samples.

2.5.2 The sample data package is divided into five units as follows:

2.5.2.1 Cover page.

2.5.2.1.1 This document shall be clearly labeled "Cover Page." The Cover Page shall contain: laboratory name; laboratory code; contract number; case number; SDG number; SAS number; EPA sample numbers in alphanumeric order, showing EPA sample number cross-referenced with laboratory ID numbers; and comments, describing in detail any problems encountered in processing the samples in the data package.

2.5.2.1.2 The Cover Page shall contain the following statement, verbatim:

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature."

2.5.2.1.3 This statement shall be directly followed by the signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

2.5.2.1.4 In the event that the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

2.5.2.2 Sample data (Results).

2.5.2.2.1 Sample data shall be arranged in packets with the Analysis Data Sheet (FORM I-AAVT, including FORM I AAVT-TIC), followed by the raw data for volatile samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.

NOTE: FORM I AAVT-TIC is the tabulated list of the highest probable match for up to 10 organic compounds that are not surrogates and internal standards and are not listed in Exhibit C (TCL). It includes the Chemical Abstracts Service (CAS) Registry Number, tentative identification, and estimated concentration.

2.5.2.2.1.1 Reconstructed total ion chromatograms (RIC) for each sample or sample extract.

2.5.2.2.1.2 RICs must be normalized to the largest nonsolvent component, and must contain the following header information:

- EPA sample number;
- Date and time of analysis;
- GC/MS instrument ID; and
- Laboratory file ID.

2.5.2.2.1.3 Internal standards are to be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak.

2.5.2.2.1.4 Quantitation Report: The complete data system report must be included in all sample data packages, in addition to the reconstructed ion chromatogram for preliminary identification and/or quantitation using either the automated or manual data system procedures. The complete data system report shall include all of the information listed below:

- EPA sample number;
- Date and time of analysis;
- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;

- Copy of area table from data system;
- GC/MS instrument ID; and
- Laboratory file ID.

2.5.2.2.1.5 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report.

2.5.2.2.1.6 Target Compound Mass Spectra: For each sample, by each compound identified, copies of the raw spectra and copies of background subtracted mass spectra of target compounds listed in Exhibit C that are identified in the sample and corresponding background subtracted target compound standard mass spectra shall be included in the data package. Spectra must be labeled with EPA sample number, laboratory file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.

2.5.2.2.1.7 Tentatively Identified Compound Mass Spectra and Library Matches: For each sample, by each compound identified, copies of mass spectra of organic compounds not listed in Exhibit C, Tentatively Identified Compounds, with associated best-match spectra (three best matches), labeled as above shall be included in the data package.

2.5.2.2.2 Volatile Standard Data:

2.5.2.2.2.1 Initial Calibration: All initial calibration data must be included for all analyses associated with the SDG. When more than one initial calibration is performed, the reconstructed ion chromatograms and quantitation reports and each type of form must be put in chronological order, by instrument as follows:

- Initial Calibration Data Sheet (FORM V-AAVT);
- Internal Standard Area and Retention Time Summary (FORM VII-AAVT); and
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimiles) for the initial (five point) calibration are labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.2.2.2 Continuing Calibration: When more than one continuing

calibration is performed, the reconstructed ion chromatograms and quantitation reports and each type of form must be put in chronological order, by instrument if more than one instrument is used as follows:

- Continuing Calibration Data Sheet (FORM VI-AAVT);
- Internal Standard Area and Retention Time Summary (FORM VII-AAVT); and
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimiles) for the initial (five point) calibration are labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.3 Quality control summary.

2.5.2.3.1 The quality control summary shall contain the following forms:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order by date of analysis or instrument.

- Blank Summary (FORM Ii-AAVT);
- GC/MS Instrument Performance Check (FORM IV-AAVT); and
- Internal Standard Area and RT Summary (FORM VII-AAVT).

2.5.2.3.2 The quality control summary shall also contain the following:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order by date of analysis or instrument.

- GC/MS Tuning Data.
  - GC/MS Tuning - BFB data, for each 12-hour period, shall be arranged in chronological order by instrument for each GC/MS system utilized;
  - GC/MS Tuning and Mass Calibration - BFB (FORM IV-AAVT);
  - Bar graph spectrum, labeled as in 2.5.2.2.1.2 and 2.5.2.2.1.4; and
  - Mass listing, labeled as in 2.5.2.2.1.2. and 2.5.2.2.1.4.

- Blank data shall be arranged in chronological order by instrument. The blank data shall be arranged in packets with both of the Organic Analysis Data Sheets (FORM I-AAVT and FORM I-AAVT-TIC), followed by the raw data for volatile samples.

- Laboratory Control Sample Data.

  - Laboratory Control Sample Data Sheet (FORM III-AAVT); and

  - Reconstructed ion chromatograms and quantitation reports or legible facsimile (GC/MS), labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.4 Raw data.

2.5.2.4.1 For each reported value, the Contractor shall include all raw data from the instrument used to obtain the sample values (except for raw data for quarterly verifications of instrument parameters). Raw data shall contain all instrument readouts used for the sample results, including those readouts that may fall below the method quantitation limit. All GC/MS instruments must provide legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included.

2.5.2.4.2 All raw data shall include concentration units for GC/MS.

2.5.2.4.3 Organic raw data must be labeled with EPA sample number and appropriate codes as shown in Tables B-1 to identify unequivocally the following:

- Initial and continuing calibration standards;

- Blanks;

- Duplicates;

- Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation;

- Data and EPA sample number for GC/MS analyses clearly and sequentially identified on the raw data;

- All calculations for sample data, including percent recovery, coefficient of variation, slope and y-intercept of linear fit; and

- Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide time of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as interference check samples and linear range analysis standards.

2.5.2.5 Preparation logs.

These logs must include the following:

- Date;

- Standard weights and/or volumes;

- Sufficient information to identify unequivocally which QC samples (e.g., laboratory control sample, blank) correspond to each batch prepared; and

- Comments describing any significant sample changes or reactions which occur during preparation.

2.5.2.6 Sample TRs

A legible copy of the sample TRs and SDG Cover Sheet shall be submitted as described in section 2.3 of this Exhibit for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample number order, considering both alpha and numeric designations.

2.6 RESULTS OF INTERCOMPARISON/PERFORMANCE EVALUATION SAMPLE ANALYSES

The reporting of analytical results for Intercomparison Study/Preaward Performance Evaluation (PPE) sample analyses includes all requirements specified in section 2.4 for reporting of sample data. The PPE sample shall be carried through the exact same process as an analytical and field samples.

2.7 COMPLETE CASE FILE PURGE

2.7.1 The Complete SDG File package includes all laboratory records received or generated for a specific Case that have not been previously

submitted to EPA as a deliverable. These items shall be submitted to EPA as a deliverable. These items shall be submitted along with their Document Inventory Sheet FORM AADC-2 (see Exhibit E for description of document numbering and inventory procedure). These items include, but are not limited to, sample tags, custody records, sample tracking records, analysts' logbook pages, bench sheets, instrument readout records, computer printouts, raw data summaries, instrument logbook pages (including instrument conditions), correspondence, and the document inventory.

2.7.2 Shipment of the Complete SDG File package by first class mail, overnight courier, priority mail, or equivalent, is acceptable. Custody seals, which are provided by EPA, shall be placed on shipping containers and a document inventory and transmittal letter included. The Contractor is not required to maintain any documents for a sample Case after submission of the Complete SDG File package; however, the Contractor should maintain a copy of the document inventory and transmittal letter.

2.8 GC/MS TAPES

2.8.1 The Contractor must store all raw and processed GC/MS data on magnetic tape, in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, laboratory control samples, initial calibrations, continuing calibrations, and BFB, as well as all laboratory generated spectral libraries and quantitation reports required to generate the data package. The Contractor shall maintain a written reference logbook of tape files to EPA sample number, calibration data, standards, blanks, and laboratory control samples. The logbook should include EPA sample numbers and standard and blank IDs, identified by Case and SDG.

2.8.2 The Contractor is required to retain the GC/MS tapes for 365 days after data submission. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the APO.

2.9 QUALITY ASSURANCE PLAN (QAP)

2.9.1 The Contractor shall prepare a written Quality Assurance Plan (QAP) which describes the procedures that are implemented to achieve the following: maintain data integrity, validity, and useability; ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility; detect problems through data assessment and established corrective action procedures which keep the analytical process reliable; and document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

2.9.2 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, SOPs pertaining to each parameter shall be included or referenced as part of the QAP. The QAP must be available during on-site laboratory evaluation and upon written request by the APO.

Table B-1

Codes for Labeling Organic Data

Sample	XXXXX
Duplicate Sample	XXXXXD
Laboratory Control Sample	VTLCS##
Laboratory Method Blank	VTMBLK##
Field Blank	VTFBLK##
Standards	VTSTD###

SECTION 3

FORM INSTRUCTIONS GUIDE/DATA REPORTING FORMS

3.1 Form Instructions Guide

3.1.1 This section includes specific instructions for the completion of all required forms for volatile organics analysis utilizing Tenax® and other adsorbents. Each of the forms is specific to a given function. These instructions are arranged in the following order:

3.1.1.1 General Information and Header Information

3.1.1.2 Cover Page [COVER PAGE - AAVT]

3.1.1.3 Analysis Data Sheet [FORM I - AAVT]

3.1.1.4 Tentatively Identified Compounds [FORM I - AAVT-TIC]

3.1.1.5 Blank Summary [FORM II - AAVT]

3.1.1.6 Laboratory Control Sample Data Sheet [FORM III - AAVT]

3.1.1.7 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AAVT]

3.1.1.8 Initial Calibration Data Sheet [FORM V - AAVT]

3.1.1.9 Continuing Calibration Data Sheet [FORM VI - AAVT]

3.1.1.10 Internal Standard Area and Retention Times Summary [FORM VII - AAVT]

3.1.1.11 Tenax® Cartridge Certification [FORM VIII - AAVT]

3.1.1.12 Surrogate Recovery [FORM IX - AAVT]

3.1.1.13 Analytical Sequence [FORM X - AAVT]

3.1.1.14 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.1.1.15 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

3.1.2 General Information and Header Information

3.1.2.1 Values must be reported on the hardcopy forms according to the individual form instructions in this Section. For example, results for concentrations of volatile organic target compounds must

be reported to three significant figures if the value is greater than or equal to 10, and to two significant figures for values less than 10.

3.1.2.2 All characters which appear on the data reporting forms presented in the contract must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the EPA APO. The names of the various fields and compounds (i.e., "Lab Code," "Chloromethane") must appear as they do on the forms in the contract.

3.1.2.3 Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low").

3.1.2.4 Six (6) pieces of information are common to the header sections of each data reporting form. They are Lab Name, Lab Code, Contract No., Case No., SDG No., and SAS No. These pieces of information must be entered on every form and must match on every form.

3.1.2.4.1 The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

3.1.2.4.2 The "Lab Code" is an alphabetical abbreviation of up to 6 letters, assigned by the EPA, to identify the laboratory and aid in data processing. This code shall be assigned by the EPA at the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of the EPA. If a change of name or ownership occurs at the laboratory, the Lab Code will remain the same until the contractor is directed by the EPA to use another Lab Code assigned by the EPA.

3.1.2.4.3 The "Contract No." is the number of the EPA contract under which the analyses were performed.

3.1.2.4.4 The "Case No." is the EPA-assigned case number associated with the sample, and reported on the Traffic Report.

3.1.2.4.5 The "SDG No." is the Sample Delivery Group (SDG) number. The SDG No. is the EPA Sample Number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

3.1.2.4.6 The "SAS No." is the EPA-assigned number for analyses

performed under Special Analytical Services (SAS). If samples are to be analyzed under SAS only and reported on these forms, then enter SAS No. and leave Case No. blank. If samples are analyzed according to the "Routine Analytical Services" (RAS) protocols and have additional SAS requirements, list both Case No. and SAS No. on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. Note that some samples in an SDG may have a SAS No. while others do not.

3.1.2.5 The "EPA Sample No." is the other information common to most of the forms. This number appears either in the upper right corner of the form, or as the left column of a table summarizing data from a number of samples. The "EPA Sample No." should be entered on the center part of the box.

3.1.2.5.1 All samples, spikes, blanks, and standards shall be identified with an EPA Sample Number. For field samples, the EPA Sample Number is the unique identifying number given in the Traffic Report that accompanied that sample.

3.1.2.5.2 In order to facilitate data assessment, the following sample suffixes must be used:

XXXXX = EPA sample number

XXXXXD = Duplicate sample

3.1.2.5.3 VOC standards prepared on Tenax® cartridges shall be identified as VTSTD###, where ### is the concentration in ng (on column) of the volatile standards (i.e., 005, 050, 100, 200, and 250).

3.1.2.5.4 As for the blank identifiers, these designations will have to be combined with other information to uniquely identify each blank. Field blanks shall be identified as VTFBLK## and laboratory method blanks shall be identified as VTMBLK##. The "EPA Sample No." must be unique for each blank analysis within an SDG. The laboratory must achieve this by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for volatiles-Tenax® blanks would be VTMBLK01, VTMBLK02, etc.

3.1.2.5.5 LCSs shall be identified as VTLCS##. The "EPA Sample No." must be unique for each LCS analysis within an SDG. The laboratory shall achieve this by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for volatiles-Tenax® cartridge LCSs would be VTLCS01, VTLCS02, etc.

3.1.2.6 Several other pieces of information are common to many of the Data Reporting Forms. These include Lab Sample ID, Lab File ID, Date Received, etc. Following is a brief description of each of these entries.

3.1.2.6.1 "Lab Sample ID" is an optional laboratory-generated internal identifier. Up to 12 alpha-numeric characters may be reported here. If the contractor does not have a Lab Sample ID, this field may be left blank.

3.1.2.6.2 "Lab File ID" is the laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. Up to 14 alpha-numeric characters may be used here.

3.1.2.6.3 "Date Received" is the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the VTSR). It should be entered as MM/DD/YY.

3.1.2.6.4 "Date Analyzed" should be entered in a similar fashion. The date of sample receipt will be compared with the analysis dates to ensure that contract holding times were not exceeded.

3.1.2.6.5 "Instrument ID" is common to many of the forms, particularly those containing calibration data. The identifier used by the laboratory must include some indication of the manufacturer and/or model of the instrument, and contain additional characters that differentiate between all instrument of the same type in the laboratory.

3.1.2.6.6 "GC Column ID" or "Column ID" is common to various other forms. This field is used to identify the GC column.

3.1.2.7 For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is odd, and round down if that digit is even.

3.1.2.8 All results must be transcribed to the forms in the raw data with the specified number of decimal places that are described in Exhibit B. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros must be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry
5.9	6.3	5.900
5.99653	6.3	5.997
95.99653	6.3	95.997
995.99653	6.3	995.997
9995.996	6.3	9996.00
99995.9	6.3	99995.9
999995.9	6.3	invalid

NOTE: 6.3 stands for a maximum of six significant figures and up to three decimal places.

3.1.3 **Cover Page [COVER PAGE - AAVT]**

3.1.3.1 This form is used to list all billable samples analyzed within an SDG, and to provide certain analytical information and general comments. It is also the document which is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

3.1.3.2 Under the "EPA Sample No." column, enter up to 7 characters for the EPA sample number (including blanks and duplicates) for each required analysis within the SDG. Duplicates must contain a "D" suffix. These sample numbers must be listed on the form in ascending alphanumeric order using the Extended Binary Coded Decimal Interchange Code convention. Thus, if MAB123A is the lowest (considering both alpha and numeric characters) EPA Sample No. within the SDG, it would be entered in the first EPA Sample No. field. Samples listed below it would be in ascending sequence - MAB124A, MAB124B, MAB125A, MAC111A, MA1111A, MA1111AD, etc.

3.1.3.3 All EPA sample numbers must be listed in ascending alphanumeric order, continuing to the following Cover Page if applicable.

3.1.3.4 Under "Lab Sample ID", a Lab Sample ID (up to 10 characters) may be entered for each associated EPA Sample No. If a Lab Sample ID is entered, it must be entered identically (for each EPA Sample No.) on all associated data.

3.1.3.5 Under "Comments", enter any problems encountered, both technical and administrative, the corrective action taken, and resolution performed for all of the samples in the SDG.

3.1.3.6 Each Cover Page must be signed, in original, by the Laboratory Manager or the Manager's designee, and dated to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.1.4 Analysis Data Sheet [FORM I - AAVT]

3.1.4.1 This form is used for tabulating and reporting results for analysis of samples on Tenax® cartridges for the compounds in a Target Compound List for Volatiles as given in Exhibit C.

3.1.4.2 This form is used for reporting the detected concentrations of the target compounds in the field samples, blanks, laboratory control samples, and performance evaluation samples.

3.1.4.3 Complete the header information on each page of Form I-AAVT according to the instructions in section 3.1.2.

3.1.4.4 Enter the volume of air pumped into the cartridge during sample collection if indicated on the sample tag. If the sample tag provides the sampling flowrate and sampling time, the volume of air sampled may be calculated by multiplying the sampling flowrate by the sampling time.

3.1.4.5 For each positively identified target compound, the Contractor shall enter the detected concentration in ng/tube. If this value is greater than or equal to the quantitation limit, report the resulting true concentration uncorrected for blank contaminants. Report analytical results to two significant figure if the value is less than 10, and three significant figures if the value is greater than or equal to 10. If the air sample volume is known to the laboratory, convert the values in ng/tube to ng/m_3 and enter in the appropriate column.

3.1.4.6 Under the column labeled "Q" for qualifier, flag each result with the specific Data Reporting Qualifiers listed below. The Contractor is encouraged to use additional flags or footnotes. The definition of such flags must be explicit and must be included in the SDG Narrative.

3.1.4.7 For reporting results to the Agency, the following contract specific qualifiers are to be used. The seven qualifiers defined below are not subject to modification by the laboratory. Up to five qualifiers may be reported on Form I-AAVT for each compound. The seven EPA-defined qualifiers to be used are as follows:

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the

sample quantitation limit but greater than zero. For example, if the sample quantitation limit is 20 ng, but a concentration of 10 ng is calculated, report it as "10J". The sample quantitation limit must be adjusted for dilution.

N - Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds, where the identification is based on a mass spectral library search. It is applied to all TIC results.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified target compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. If one or more compounds have a response greater than full scale, except as noted in Exhibit D, the sample should have the concentration flagged with an "E" on the Form I-AAVT for the original analysis.

X - Other specific flags may be required to properly define the results. If used, they must be fully described, and such description attached to the Sample Data Summary Package and the SDG Narrative. Begin by using "X". If more than one flag is required, use "Y" and "Z" as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "B", and "D" flags for some sample. The laboratory-defined flags are limited to the letters "X", "Y", and "Z".

NOTE: The combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are detected in the sample.

3.1.5 **Tentatively Identified Compounds [FORM I - AAVT-TIC]**

3.1.5.1 Fill in all header information as above.

3.1.5.2 Report Tentatively Identified Compounds (TICs) including CAS number, compound name, retention time (RT), and the estimated concentration (criteria for reporting TICs are given in Exhibit D). Retention time must be reported in minutes and decimal minutes, not seconds or minutes:seconds.

3.1.5.3 If, in the opinion of the mass spectral interpretation

specialist, no valid tentative identification can be made, the compound shall be reported as unknown.

3.1.5.4 Include a Form I-AAVT-TIC for every sample and blank analyzed, even if no TICs are found. Total the number of TICs found, and enter this number in the "No. of TICs found." If none were found, enter "0" (zero). Form I-AAVT-TIC must be provided for every analysis, including required dilutions and reanalyses, even if no TICs are found.

3.1.6 **Blank Summary [FORM II - AAVT]**

3.1.6.1 This form summarizes the samples associated with each field and laboratory blank analysis. A copy of the appropriate Form II-AAVT is required for each blank reported on a Form I-AAVT.

3.1.6.2 Complete the header information on Form II-AAVT as described in section 3.1.2. The "EPA Sample No." entered in the box at the top of Form II-AAVT shall be the same number entered on the Form I-AAVT when reporting results for the blank itself.

3.1.6.3 On the numbered lines, enter the EPA sample numbers associated with the blank, along with the other information which identifies the EPA samples. The Cartridge ID for each sample must be provided under the "Cartridge" column, if available.

3.1.7 **Laboratory Control Sample Data Sheet [FORM III - AAVT]**

3.1.7.1 Form III-AAVT is used to report the recovery of the spiked analytes in the laboratory control samples (LCS).

3.1.7.2 Complete the header information according to the instructions in section 3.1.2.

3.1.7.3 Enter the date and time the LCS was analyzed.

3.1.7.4 In the table under "Spiked," enter the spiked concentration in ng/tube of each LCS compound. Under "Reported," enter the concentration obtained in ng/tube calculated from the analysis of the LCS. Calculate the percent recovery of each LCS compound to the nearest whole percent and enter in the column under "% Recovery". At the bottom of the table are the QC limits for LCS percent recoveries. Flag all values outside of the limits with an "*" in the column under the "Q" symbol.

3.1.7.5 Summarize the values outside the QC limits at the lower part of the form.

3.1.7.6 Enter any comments pertinent to the analysis of the LCS.

3.1.8 **GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AAVT]**

3.1.8.1 This form is used to report the results of GC/MS instrument performance check (also known as "tuning") and to summarize the date and time of analysis of samples, standards, and blanks associated with each analysis of the instrument performance check solution.

3.1.8.2 Complete the header information as in section 3.1.2. Enter the "Lab File ID" for the injection containing the instrument performance check mixture of BFB. Enter the date and time (military time) of injection of the instrument performance check mixture.

3.1.8.3 For each ion listed on the form, enter the percent relative abundance in the right column. Report relative abundances to the number of significant figures given for each ion in the ion abundance criteria column.

3.1.8.4 Under "to m/e 95", all ion abundances are to be normalized to the nominal base peak listed on Form IV-AAVT. For some of the ions, determine the percentage of the ion abundance to the specified mass and report under "to specified mass". For example, if the relative ion abundance of mass 96 and mass 174 ions are 4 and 80 (under the "to m/e 95" column), respectively, then enter "5.0" (under the "to specified mass" column) as the ion abundance of mass 96 relative to mass 174.

3.1.8.5 All relative abundances must be reported as a number. If zero, enter "0", not a dash or other non-numeric character.

3.1.8.6 In the lower half of the form, list all samples and standards analyzed under that instrument performance check in <u>chronological order</u>, by time of analysis (in military time). Refer to section 3.1.2 for specific instructions for identifying standards and blanks. Enter "EPA Sample No.", "Lab Sample ID", "Lab File ID", "Date Analyzed", and "Time Analyzed" for all standards, samples, and blanks.

3.1.8.7 The GC/MS instrument performance check must be analyzed again twelve hours from the time of injection of the instrument performance check solution of BFB listed at the top of the form. In order to meet these requirements, samples, standards, or blanks must be injected within twelve hours of the injection of the instrument performance check solution.

3.1.9 **Initial Calibration Data Sheet [FORM V - AAVT]**

3.1.9.1 Each time the GC/MS system undergoes an initial calibration, the laboratory must complete and submit a Form V-AAVT.

3.1.9.2 Complete all header information as in section 3.1.2.

3.1.9.3 Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID" and "GC Column ID".

3.1.9.4 Check the appropriate standard preparation method used to calibrate the GC/MS system - flash vaporization, static dilution bottle technique, or permeation calibration generator.

3.1.9.5 Enter the injection dates and times of each of the calibration standards analyzed under "Date injected" and "Time injected", respectively.

3.1.9.6 Enter the "EPA Sample No." and "Lab File ID" for each of the five calibration standards.

3.1.9.7 Complete the relative response factor (RRF) calculation for the five calibration points, and then calculate and report the average relative response factor ($\overline{RRF}$) and %RSD of the RRF values for each target and surrogate compound in the space provided.

3.1.10 **Continuing Calibration Data Sheet [FORM VI - AAVT]**

3.1.10.1 Each time the GC/MS system undergoes a continuing calibration to check for the validity of the initial calibration, the laboratory must complete and submit a Form VI-AAVT.

3.1.10.2 Complete all header information as in section 3.1.2. Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID", "GC Column ID", and the date(s) of the most recent initial calibration. If the calendar date changes during the calibration procedure, the inclusive dates should be given on Form VI.

3.1.10.3 Enter the date and time of injection of the continuing calibration standard.

3.1.10.4 Complete the relative response factor (RRF) calculation for each target compound in the space provided.

3.1.10.5 Indicate with a check mark the method used for the continuing calibration. Note that direct injection calibration is allowed only for the continuing calibration check. If the continuing calibration standard is in a Tenax® cartridge, the same desorption method (flash vaporization, static dilution bottle technique, or permeation calibration generator) used for the initial calibration standards must be used for the continuing calibration standard.

3.1.10.6 Under the column "IC mean RRF", enter the mean relative response factor for each target compound as determined in the most recent valid initial calibration.

3.1.10.7 A RRF is calculated for this concentration and compared to the mean RRF value in the most recent valid initial calibration. Calculate the percent difference (%D) between the continuing calibration RRF and the mean RRF from the most recent valid initial calibration.

3.1.11 Internal Standard Area and Retention Time Summary [FORM VII - AAVT]

3.1.11.1 This form is used to summarize the peak areas and retention times of the internal standards added to all samples and blanks. The data are used to determine when changes in internal standard responses will adversely affect quantification of target compounds. This form must be completed each time an initial or continuing calibration is performed for each GC/MS system.

3.1.11.2 Complete the header information according to section 3.1.2.

3.1.11.3 Enter the Lab File ID of the 12-hour calibration standard, as well as the date and time of analysis of the calibration standard. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a continuing calibration, a Form VII-AAVT shall be completed on the basis of the internal standard areas of the mid level (CAL 3) initial calibration standard. Use the date and time of analysis of this standard, and its Lab File ID and areas in place of those of a continuing calibration standard.

3.1.11.4 From the results of the analysis of the 12-hour calibration standard, enter the area measured for each internal standard and its retention time (in decimal minutes) under the appropriate column. For each internal standard, calculate the upper limit of the area as the area of the particular standard plus 40 percent of its area (i.e., 1.4 times the area in the 12 HOUR STD box), and the lower limit of the area as the area of the internal standard minus 40% of its area (i.e., 0.6 times the area in the 12 HOUR STD box). Report these values in the boxes labeled "UPPER LIMIT" and "LOWER LIMIT", respectively.

3.1.11.5 Calculate the upper limit of the retention time as the retention of the internal standard plus 0.33 minutes (20 seconds), and the lower limit of the retention time as the retention time in the standard minus 0.33 minutes (20 seconds).

3.1.11.6 For each sample and blank under a given 12-hour analytical sequence, enter the EPA Sample Number and the area measured for each internal standard and its retention time. If the internal standard

area is outside the upper or lower limits calculated above, flag that area with an asterisk (*) placed in the far right-hand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the retention time of any internal standard that is outside the limits with an asterisk.

3.1.12 Tenax® Cartridge Certification [FORM VIII - AAVT]

3.1.12.1 This form is used to document the certification of Tenax® cartridges prior to use.

3.1.12.2 Complete the header information on each page of Form VII-AAVT according to the instructions in section 3.1.2.

3.1.12.3 Enter under "Tenax® Cartridge ID" (shaded cells) the identification number of up to five Tenax® cartridges used for certification.

3.1.12.4 Enter the results of the analysis of an unspiked certified clean cartridge. For target compounds that are not detected, enter the CRQL of the compound followed by a "U". If the detected level is less than the CRQL, enter the value followed by a "J".

3.1.12.5 At the bottom of each column, enter the total or subtotal level of VOCs found. Include on those that are positive hits; values flagged with a "U" are not included.

3.1.12.6 If the none of the values for a particular cartridge are more than the corresponding CRQL of any of the target compounds, and if the total level of VOCs is not greater than 10 ng/tube, then the cartridge is certified.

3.1.13 Surrogate Recovery [FORM IX - AAVT]

3.1.13.1 Form IX - AAVT is used to report the recoveries of the surrogate compounds added to each Tenax® adsorbent tube.

3.1.13.2 Complete the header information and enter EPA Sample Numbers as described in section 3.2.1. For each surrogate, report the percent recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.

3.1.13.3 Flag each surrogate recovery outside the QC limits with an asterisk (*). The asterisk must be placed in the last space in each appropriate column, under the "#" symbol. In the far righthand column, total the number of surrogate recoveries outside the QC limits for each sample (Total Out). If no surrogates were outside the limits, enter "0".

3.1.14 Analytical Sequence [FORM X - AAVT]

3.1.14.1 A Form X-AAVT is required for each analytical sequence for each GC/MS system used to perform VOA on Tenax® cartridge samples in an SDG.

3.1.14.2 Complete the header information on each page of Form VII-AAVT according to the instructions in section 3.1.2.

3.1.14.3 On the numbered lines, enter the EPA sample numbers along with the other information which identifies the samples, blanks, and standards. The first item in the table must be the BFB since the 12-hour time period starts at the injection of the instrument performance check standard. Arrange the items in chronological order for each GC/MS system.

3.1.15 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.1.15.1 This form is used to document the receipt and inspection of sample containers and samples. One original of Form AADC-1 is required for each sample shipping container. If the samples in a single sample shipping container must be assigned to more than one Sample Delivery Group, the original Form AADC-1 shall be placed with the deliverables for the Sample Delivery Group of the lowest Arabic number and a copy of Form AADC-1 must be placed with the deliverables for the other Sample Delivery Group(s). The copies should be identified as "copy(ies)," and the location of the original should be noted on the copies.

3.1.15.2 Sign and date the airbill (if present). Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in item 1 on Form AADC-1. Record the custody seal numbers in item 2.

3.1.15.3 Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (i.e., Traffic Reports, Packing Lists), and airbills or airbill stickers in items 3-5 on Form AADC-1. Specify if there is an airbill present or an airbill sticker in item 5 on Form AADC-1. Record the airbill or sticker number, if present.

3.1.15.4 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample (i.e., intact, dent, leaking) and presence of absence of sample tags in items 6 and 7 on Form AADC-1.

3.1.15.5 Review the sample shipping documents and complete the header information described in section 3.1.2. Compare the information recorded on all the documents and samples and circle the appropriate

answer in item 8 on Form AADC-1.

3.1.15.6 If there are no problems observed during receipt, sign and date (include time) Form AADC-1, the chain-of-custody record, and Traffic Report, and write the sample numbers on Form AADC-1. Record the appropriate sample tags and assigned laboratory numbers, if applicable. The log-in date should be recorded at the top of Form AADC-1 and the date and time of sample receipt at the laboratory should be recorded in items 9 and 10. Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of Form AADC-1. Sign and date the Sample Transfer block. Cross out unused columns and spaces.

3.1.15.7 If there are problems observed during receipt or an answer marked with an asterisk (i.e., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.1.16 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

3.1.16.1 This form is used to record the inventory of the SDG File Purge documents and count of documents in the original Sample Data Package which is sent to the Region.

3.1.16.2 Organize all EPA-CSF documents as described in Exhibit B, Section 1. Assemble the documents in the order specified on Form AADC-2, and stamp each page with a consecutive number. (Do not number the AADC-2 form). Inventory the CSF by reviewing the document numbers and recording page numbers ranges in the columns provided in the Form AADC-2. If there are no documents for a specific document type, enter an "NA" in the empty space.

3.1.16.3 Certain laboratory specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review AADC-2 to determine if it is most appropriate to place them under No. 17, 18, 19, or 20. Category 20 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.2 Data Reporting Forms

3.2.1 Cover Page [COVER PAGE - AAVT]

3.2.2 Analysis Data Sheet [FORM I - AAVT]

3.2.3 Tentatively Identified Compounds [FORM I - AAVT-TIC]

3.2.4 Blank Summary [FORM II - AAVT]

3.2.5 Laboratory Control Sample Data Sheet [FORM III - AAVT]

3.2.6 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AAVT]

3.2.7 Initial Calibration Data Sheet [FORM V - AAVT]

3.2.8 Continuing Calibration Data Sheet [FORM VI - AAVT]

3.2.9 Internal Standard Area and Retention Times Summary [FORM VII - AAVT]

3.2.10 Tenax® Cartridge Certification [FORM VIII - AAVT]

3.2.11 Surrogate Recovery [FORM IX - AAVT]

3.2.12 Analytical Sequence [FORM X - AAVT]

3.2.13 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.2.14 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

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COVER PAGE

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

	EPA Sample No.	Lab Sample ID
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

Comments:

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data (if submitted) on floppy diskette has been authorized by the Laboratory Manager's designee, as verified by the following signature:

Signature: Name:
Date: Title:

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SAMPLE RECEIPT/LOG–IN SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

ITEM	REMARKS	EPA Sample No.	Sample Tag No.	Assigned Lab No.	Spl Vol (m^3)	REMARKS: Conditions of Sample Shipment,etc.
1. Custody Seal(s)	Present/Absent/Intact/Broken*					
2. Custody Seal No(s).						
3. Chain–of–Custody records	Present/Absent*					
4. Traffic Reports or Packing List	Present/Absent*					
5. Airbill	Sticker/Present/Absent*					
Airbill No(s).						
6. Sample Tags	Present/Absent*					
Sample Tag No(s):	Listed/Not Listed on COC					
7. Sample Condition	Intact/Broken/Leaking*					
8. Do informations on custody records, traffic reports, and sample tags agree?	Yes/No*					
9. Date Received at Lab:						
10. Time Received at Lab:						
Sample Transfer						
Area #:						
By:						
On:						

* If circled, contact SMO and attach record of resolution.

Received by:
Signature:_____ Log–in Date:_____
Print Name:_____

Reviewed by:
Signature:_____ Date:_____
Logbook No.:_____ Logbook Page No.:_____

FORM AADC–1

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COMPLETE SDG FILE (CSF) DOCUMENT INVENTORY SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No:_____ SDG No.:_____

DOCUMENT	Page Nos. From	Page Nos. To	Please Check Lab	Please Check Reg
1. Cover Page (Cover Page – AAVT)				
2. Sample Receipt/Log–In Sheet (FORM AADC–1)				
3. CSF Document Inventory Sheet (FORM AADC–2)				
4. Analysis Data Sheet (FORM I – AAVT)				
5. Tentatively Identified Compounds (FORM I – AAVT–TIC)				
6. Blank Summary Form (FORM II – AAVT)				
7. Laboratory Control Sample Data Sheet (FORM III – AAVT)				
8. GC/MS Tuning with BFB (FORM IV – AAVT)				
9. Initial Calibration Data Sheet (FORM V – AAVT)				
10. Continuing Calibration Data Sheet (FORM VI – AAVT)				
13. Internal Standard Area and RT Summary (FORM VII – AAVT)				
14. Tenax Cartridge Certification Data Sheet (FORM VIII – AAVT)				
15. Surrogate Recovery Form (FORM IX – AAVT)				
16. Analytical Sequence (FORM X – AAVT)				
17. EPA Shipping/Receiving Documents				
Airbill (No. of shipments:___)				
Chain–of–Custody Records				
Sample Tags				
Sample Log–In Sheet (Lab & AADC–1)				
17. Misc. Shipping/Receiving Records (list individual records)				
Telephone Logs				
18. Internal Lab Sample Transfer Records				
19. Internal Original Sample Preparation and Analysis Records				
20. Other Records (describe or list)				

Comments:

Completed by (CLP Lab):
Signature:_____ Date:_____
Printed Name/Title:_____
Audited by (EPA):
Signature:_____ Date:_____
Printed Name/Title:_____

FORM AADC–2

U. S. ENVIRONMENTAL PROTECTION AGENCY
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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Received:_____ Instrument ID:_____
Lab File ID:_____ Date Analyzed:_____ Column ID:_____
Sampled Air Volume, m^3 (corrected to STP):_____

CAS RN	COMPOUND NAME	Concentration		Q
		ng/tube	ng/m^3	
100–52–7	Benzaldehyde			
71–43–2	Benzene			
100–47–0	Benzonitrile			
108–86–1	Bromobenzene			
74–97–5	Bromochloromethane			
109–70–6	1–Bromo–3–chloropropane			
74–96–4	Bromoethane			
75–62–7	Bromotrichloromethane			
104–51–8	n–Butylbenzene			
56–23–5	Carbon tetrachloride			
108–90–7	Chlorobenzene			
78–86–4	2–Chlorobutane			
106–89–8	1–Chloro–2,3–epoxypropane			
110–75–8	2–Chloroethoxyethene			
67–66–3	Chloroform			
540–54–5	1–Chloropropane			
75–29–6	2–Chloropropane			
107–05–1	3–Chloro–1–propene			
108–41–8	m–Chlorotoluene			
95–49–8	o–Chlorotoluene			
106–43–4	p–Chlorotoluene			
106–93–4	1,2–Dibromoethane			
74–95–3	Dibromomethane			
78–75–1	1,2–Dibromopropane			
95–50–1	1,2–Dichlorobenzene			
541–73–1	1,3–Dichlorobenzene			
106–46–7	1,4–Dichlorobenzene			
1190–22–3	1,3–Dichlorobutane			
110–56–5	1,4–Dichlorobutane			

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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Received:_____ Instrument ID:_____
Lab File ID:_____ Date Analyzed:_____ Column ID:_____
Sampled Air Volume, m^3 (corrected to STP):_____

CAS RN	COMPOUND NAME	Concentration		Q
		ng/tube	ng/m^3	
7581-97-7	2,3-Dichlorobutane			
764-41-0	cis-1,4-Dichloro-2-butene			
760-23-6	3,4-Dichloro-1-butene			
75-34-3	1,1-Dichloroethane			
107-06-2	1,2-Dichloroethane			
75-35-4	1,1-Dichloroethene			
78-87-5	1,2-Dichloropropane			
142-28-9	1,3-Dichloropropane			
123-91-1	1,4-Dioxane			
1073-67-2	1-Ethenyl-4-chlorobenzene			
100-41-4	Ethylbenzene			
98-82-8	(1-Methylethyl) benzene			
99-87-6	1-Methyl-4-(1-methylethyl) benzene			
76-01-7	Pentachloroethane			
98-86-2	1-Phenylethanone			
100-42-5	Styrene			
630-20-6	1,1,1,2-Tetrachloroethane			
79-43-5	1,1,2,2-Tetrachloroethane			
127-18-4	Tetrachloroethylene			
109-99-9	Tetrahydrofuran			
108-88-3	Toluene			
75-25-2	Tribromomethane			
71-55-6	1,1,1-Trichloroethane			
79-06-5	1,1,2-Trichloroethane			
79-01-6	Trichloroethylene			
96-18-4	1,2,3-Trichloropropane			
108-67-8	1,3,5-Trimethylbenzene			
1330-20-7	Xylenes, m- and p-			
95-47-6	Xylene, o-			

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TENTATIVELY IDENTIFIED COMPOUNDS

EPA Sample No. _____

Lab Name: _____ Contract No.: _____
Lab Code: _____ Case No.: _____
Lab Sample ID: _____ Lab File ID: _____
SAS No.: _____ Date Received: _____
SDG No: _____ Date Analyzed: _____
GC Column ID: _____ Instrument ID: _____
No. of TICs Found: _____ Sampled Air Volume, m^3 (at STP): _____

	CAS RN	COMPOUND NAME	Estimated Concentration ng/tube	Estimated Concentration ng/m^3	RT (mins.)	Q
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						

U. S. ENVIRONMENTAL PROTECTION AGENCY
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BLANK SUMMARY

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Analyzed:_____ Instrument ID:_____
Lab File ID:_____ Time Analyzed:_____ Column ID:_____

THIS BLANK APPLIES TO THE FOLLOWING SAMPLES:

	EPA Sample No.	Laboratory ID			Analysis	
		Sample	File	Cartridge	Date	Time
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						

Comments:

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LABORATORY CONTROL SAMPLE DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Analyzed:_____ Instrument ID:_____
Lab File ID:_____ Time Analyzed:_____ Column ID:_____

CAS RN	COMPOUND NAME	Concentration (ng/tube)		% Recovery	Q
		Spiked	Reported		
71–43–2	Benzene				
56–23–5	Carbon tetrachloride				
106–93–4	1,2–Dibromoethane				
106–46–7	1,4–Dichlorobenzene				
107–06–2	1,2–Dichloroethane				
78–87–5	1,2–Dichloropropane				
127–18–4	Tetrachloroethylene				
79–06–5	1,1,2–Trichloroethane				
79–01–6	Trichloroethylene				

%Recovery QC Limits: 60–140%

LCS Recovery: _____ outside limits out of _____ total.

Comments:

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GC/MS INSTRUMENT PERFORMANCE CHECK AND MASS CALIBRATION

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Injected:_____ Instrument ID:_____
Lab File ID:_____ Time Injected:_____ GC Column ID:_____
Mass BFB Injected (ng):_____

m/e	ION ABUNDANCE CRITERIA	% Relative Abundance	
		to m/e 95	to specified mass
50	8.0 – 40.0% of m/e 95		
75	30.0 – 66.0 of m/e 95		
95	Base peak, 100% relative abundance		
96	5.0 – 9.0% of m/e 174		
173	Less than 2.0% of m/e 174		
174	50.0 – 120.0% of m/e 95		
175	4.0 – 9.0% of m/e 174		
176	93.0 – 101.0% of m/e 174		
177	5.0 – 9.0% of m/e 176		

THIS TUNE APPLIES TO THE FOLLOWING:

	EPA Sample No.	Lab Sample ID	Lab File ID	Date Analyzed	Time Analyzed
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					

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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Instrument ID:_____ GC Column ID:_____
Standard Preparation Method:____flash vaporization ____static dilution ____permeation calibration

STANDARD	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5		
EPA Sample No.							
Lab File ID							
Date injected							
Time injected							
	Relative Response Factor (RRF)					mean	
COMPOUND NAME	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	RRF	%RSD
Benzene–d_6							
Chlorobenzene–d_5							
1,2–Dichlorobenzene–d_4							
Benzaldehyde							
Benzene							
Benzonitrile							
Bromobenzene							
Bromochloromethane							
1–Bromo–3–chloropropane							
Bromoethane							
Bromotrichloromethane							
n–Butylbenzene							
Carbon tetrachloride							
Chlorobenzene							
2–Chlorobutane							
1–Chloro–2,3–epoxypropane							
2–Chloroethoxyethene							
Chloroform							
1–Chloropropane							
2–Chloropropane							
3–Chloro–1–propene							
m–Chlorotoluene							
o–Chlorotoluene							
p–Chlorotoluene							
1,2–Dibromoethane							
Dibromomethane							
1,2–Dibromopropane							
1,2–Dichlorobenzene							
1,3–Dichlorobenzene							
1,4–Dichlorobenzene							
1,3–Dichlorobutane							

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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Instrument ID:_____ GC Column ID:_____
Standard Preparation Method:____flash vaporization ____static dilution ____permeation calibration

STANDARD	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5		
EPA Sample No.							
Lab File ID							
Date injected							
Time injected							
	Relative Response Factor (RRF)					mean	
COMPOUND NAME	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	RRF	%RSD
1,4–Dichlorobutane							
2,3–Dichlorobutane							
cis–1,4–Dichloro–2–butene							
3,4–Dichloro–1–butene							
1,1–Dichloroethane							
1,2–Dichloroethane							
1,1–Dichloroethene							
1,2–Dichloropropane							
1,3–Dichloropropane							
1,4–Dioxane							
1–Ethenyl–4–chlorobenzene							
Ethylbenzene							
(1–Methylethyl) benzene							
1–Methyl–4–(1–methylethyl)benzene							
Pentachloroethane							
1–Phenylethanone							
Styrene							
1,1,1,2–Tetrachloroethane							
1,1,2,2–Tetrachloroethane							
Tetrachloroethylene							
Tetrahydrofuran							
Toluene							
Tribromomethane							
1,1,1–Trichloroethane							
1,1,2–Trichloroethane							
Trichloroethylene							
1,2,3–Trichloropropane							
1,3,5–Trimethylbenzene							
Xylenes, m– and p–							
Xylene, o–							

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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
EPA Sample No.:_____ Lab File ID:_____
Instrument ID:_____ GC Column ID:_____
Date Injected:_____ Time Injected:_____
Calibration Method:____direct injection ____via cartridge Date of Init. Cal.:_____

COMPOUND NAME	IC mean RRF	RRF	%D
Benzene–d_6			
Chlorobenzene–d_5			
1,2–Dichlorobenzene–d_4			
Benzaldehyde			
Benzene			
Benzonitrile			
Bromobenzene			
Bromochloromethane			
1–Bromo–3–chloropropane			
Bromoethane			
Bromotrichloromethane			
n–Butylbenzene			
Carbon tetrachloride			
Chlorobenzene			
2–Chlorobutane			
1–Chloro–2,3–epoxypropane			
2–Chloroethoxyethene			
Chloroform			
1–Chloropropane			
2–Chloropropane			
3–Chloro–1–propene			
m–Chlorotoluene			
o–Chlorotoluene			
p–Chlorotoluene			
1,2–Dibromoethane			
Dibromomethane			
1,2–Dibromopropane			
1,2–Dichlorobenzene			
1,3–Dichlorobenzene			
1,4–Dichlorobenzene			
1,3–Dichlorobutane			

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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
EPA Sample No.:_____ Lab File ID:_____
Instrument ID:_____ GC Column ID:_____
Date Injected:_____ Time Injected:_____
Calibration Method:_____direct injection _____via cartridge Date of Init. Cal.:_____

COMPOUND NAME	IC mean RRF	RRF	%D
1,4–Dichlorobutane			
2,3–Dichlorobutane			
cis–1,4–Dichloro–2–butene			
3,4–Dichloro–1–butene			
1,1–Dichloroethane			
1,2–Dichloroethane			
1,1–Dichloroethene			
1,2–Dichloropropane			
1,3–Dichloropropane			
1,4–Dioxane			
1–Ethenyl–4–chlorobenzene			
Ethylbenzene			
(1–Methylethyl) benzene			
1–Methyl–4–(1–methylethyl) benzene			
Pentachloroethane			
1–Phenylethanone			
Styrene			
1,1,1,2–Tetrachloroethane			
1,1,2,2–Tetrachloroethane			
Tetrachloroethylene			
Tetrahydrofuran			
Toluene			
Tribromomethane			
1,1,1–Trichloroethane			
1,1,2–Trichloroethane			
Trichloroethylene			
1,2,3–Trichloropropane			
1,3,5–Trimethylbenzene			
Xylenes, m– and p–			
Xylene, o–			

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INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

	Perfluorotoluene		1,2-Dichlorobenzene-d_4		1,4-Difluorobenzene	
	Area #	RT #	Area #	RT #	Area #	RT #
12-HOUR STANDARD						
Upper Limit						
Lower Limit						
EPA Sample No.						
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

AREA: Upper Limit: +40% of internal standard area. RT: Upper Limit: +0.33 minutes of internal standard RT.
Lower Limit: −40% of internal standard area. Lower Limit: −0.33 minutes of internal standard RT.

All values outside of the QC limits must be followed by an "*" under the "#" column.

FORM VII – AAVT

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TENAX CARTRIDGE CERTIFICATION

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No:_____
SAS No.:_____ SDG No.:_____

	COMPOUND NAME	Tenax Cartridge ID				
1	Benzaldehyde					
2	Benzene					
3	Benzonitrile					
4	Bromobenzene					
5	Bromochloromethane					
6	1–Bromo–3–chloropropane					
7	Bromoethane					
8	Bromotrichloromethane					
9	n–Butylbenzene					
10	Carbon tetrachloride					
11	Chlorobenzene					
12	2–Chlorobutane					
13	1–Chloro–2,3–epoxypropane					
14	2–Chloroethoxyethene					
15	Chloroform					
16	1–Chloropropane					
17	2–Chloropropane					
18	3–Chloro–1–propene					
19	m–Chlorotoluene					
20	o–Chlorotoluene					
21	p–Chlorotoluene					
22	1,2–Dibromoethane					
23	Dibromomethane					
24	1,2–Dibromopropane					
25	1,2–Dichlorobenzene					
26	1,3–Dichlorobenzene					
27	1,4–Dichlorobenzene					
28	1,3–Dichlorobutane					
29	1,4–Dichlorobutane					
	Subtotal VOCs (ng/tube)					

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TENAX CARTRIDGE CERTIFICATION

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No:_____
SAS No.:_____ SDG No.:_____

	COMPOUND NAME	Tenax Cartridge ID				
30	2,3–Dichlorobutane					
31	cis–1,4–Dichloro–2–butene					
32	3,4–Dichloro–1–butene					
33	1,1–Dichloroethane					
34	1,2–Dichloroethane					
35	1,1–Dichloroethene					
36	1,2–Dichloropropane					
37	1,3–Dichloropropane					
38	1,4–Dioxane					
39	1–Ethenyl–4–chlorobenzene					
40	Ethylbenzene					
41	(1–Methylethyl) benzene					
42	1–Methyl–4–(1–methylethyl)benzene					
43	Pentachloroethane					
44	1–Phenylethanone					
45	Styrene					
46	1,1,1,2–Tetrachloroethane					
47	1,1,2,2–Tetrachloroethane					
48	Tetrachloroethylene					
49	Tetrahydrofuran					
50	Toluene					
51	Tribromomethane					
52	1,1,1–Trichloroethane					
53	1,1,2–Trichloroethane					
54	Trichloroethylene					
55	1,2,3–Trichloropropane					
56	1,3,5–Trimethylbenzene					
57	Xylenes, m– and p–					
58	Xylene, o–					
Total VOCs (ng/tube)						

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Volatile Organics in Ambient Air – Tenax

SURROGATE RECOVERY

Lab Name: _____ Contract No.: _____
Lab Code: _____ Case No.: _____
SAS No.: _____ SDG No.: _____

	EPA Sample No.	Surrogate Percent Recovery						Total Out
		benzene–d_6	#	chlorobenzene–d_5	#	dichlorobenzene–d_4	#	
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

Surrogate %Recovery Limits: 80–120%
Values outside of QC limits are flagged with a "*" under the "#" column

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Volatile Organics in Ambient Air – Tenax

ANALYTICAL SEQUENCE

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Instrument ID:_____ GC Column ID:_____

	EPA Sample No.	Lab Sample ID	Analysis Time	Analysis Date
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

FORM X – AAVT

EXHIBIT C

TARGET COMPOUND LISTS

TABLE 1

VOLATILES IN AMBIENT AIR
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Target Compound	CAS RN	ng, on column
Benzaldehyde	100-52-7	20
Benzene	71-43-2	9
Benzonitrile	100-47-0	5
Bromobenzene	108-86-1	45
Bromochloromethane	74-97-5	7
1-Bromo-3-chloropropane	109-70-6	5
Bromoethane	74-96-4	26
Bromotrichloromethane	75-62-7	6
n-Butylbenzene	104-51-8	3
Carbon tetrachloride	56-23-5	7
Chlorobenzene	108-90-7	6
2-Chlorobutane	78-86-4	12
1-Chloro-2,3-epoxypropane	106-89-8	26
2-Chloroethoxyethene	110-75-8	27
Chloroform	67-66-3	9
1-Chloropropane	540-54-5	6
2-Chloropropane	75-29-6	11
3-Chloro-1-propene	107-05-1	6
m-Chlorotoluene	108-41-8	2
o-Chlorotoluene	95-49-8	2
p-Chlorotoluene	106-43-4	2
1,2-Dibromoethane	106-93-4	11
Dibromomethane	74-95-3	15
1,2-Dibromopropane	78-75-1	48
1,2-Dichlorobenzene	95-50-1	4
1,3-Dichlorobenzene	541-73-1	5
1,4-Dichlorobenzene	106-46-7	41
1,3-Dichlorobutane	1190-22-3	2
1,4-Dichlorobutane	110-56-5	27
2,3-Dichlorobutane	7581-97-7	17
1,4-Dichloro-2-butene	764-41-0	7
3,4-Dichloro-1-butene	760-23-6	22

TABLE 1

VOLATILES IN AMBIENT AIR
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Target Compound	CAS RN	ng, on column
1,1-Dichloroethane	75-34-3	19
1,2-Dichloroethane	107-06-2	13
1,1-Dichloroethene	75-35-4	23
1,2-Dichloropropane	78-87-5	13
1,3-Dichloropropane	142-28-9	32
1,4-Dioxane	123-91-1	13
1-Ethenyl-4-chlorobenzene	1073-67-2	7
Ethylbenzene	100-41-4	6
1-(methylethyl)benzene	98-82-8	4
1-Methyl-4-(1-methylethyl)benzene	99-87-6	13
Pentachloroethane	76-01-7	6
1-Phenylethanone	98-86-2	10
Styrene	100-42-5	6
1,1,1,2-Tetrachloroethane	630-20-6	3
1,1,2,2-Tetrachloroethane	79-43-5	22
Tetrachloroethylene	127-18-4	9
Tetrahydrofuran	109-99-9	4
Toluene	108-88-3	7
Tribromomethane	75-25-2	28
1,1,1-Trichloroethane	71-55-6	6
1,1,2-Trichloroethane	79-06-5	7
Trichloroethylene	79-01-6	3
1,2,3-Trichloropropane	96-18-4	16
1,3,5-Trimethylbenzene	108-67-8	9
Xylenes, m- and p-	1330-20-7	2
Xylene, o-	95-47-6	2

NOTE: The values in Table 1 are Contract Required Quantitation Limits (CRQL), not absolute detection limits. The quantitation limits in these tables are set near the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

TABLE 2

VOLATILE ORGANIC COMPOUNDS FOR
TENTATIVE IDENTIFICATION AND DEVELOPMENT OF CRQL DATA*

Target Compound	CAS RN
Carbon disulfide	75-15-0
Pyridine	110-86-1
1,2-Dibromo-3-chloropropane	96-12-8
2-Chloro-1 3-butadiene	126-99-8
trans-1,4-Dichlorobutene	110-57-6
Propanal	123-38-6
2-Hexanone	591-78-6
Cyclohexanone	108-94-1
1-Bromobutane	109-65-9
2-Methylnaphthalene	91-57-6
1,3,4-Trimethylbenzene	95-63-6
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
n-Propylbenzene	103-65-1
tert-Butylbenzene	98-06-6
sec-Butylbenzene	135-98-8
1,2,3-Trichlorobenzene	87-61-6

* This table lists additional compounds for which the adsorbent method has not been validated, but which were ranked as air toxics of concern or identified as compounds of Regional interest during the development of this document. Data developed during method validation and subsequent analysis under SAS and RAS may lead to validation of the VOC methods in this document for some or all of these compounds.

EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED ON TENAX® AND ANALYZED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

December, 1991

EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED ON TENAX® AND ANALYZED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

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EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED ON TENAX® AND ANALYZED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1 INTRODUCTION

1.1 Scope and Application

1.1.1 This document describes a method for determining the presence and concentration of specific volatile organic compounds (VOCs) in ambient air included in the Target Compound List (Exhibit C) and in Table D/VT-1. The method is based on the collection of VOCs on Tenax® solid adsorbent, which is made of poly(2,6-diphenylphenylene oxide), and subsequently thermally desorbing and analyzing the VOCs by gas chromatography/mass spectrometry (GC/MS).

1.1.2 While this method outlines the use of Tenax® as the adsorbent, alternative adsorbents (e.g., multibed) will be allowed. The Laboratory must demonstrate audit accuracy and replicate precision limits of the alternative adsorbent as well as adsorbent characteristic data for selected analytes in order for it to be equivalent. In addition, the analysis of Performance Evaluation (PE) samples must meet required audit accuracy and replicate precision limits as outlined in Section 5. These accuracy and precision limits were based upon data acquired over the last five (5) years from USEPA's Toxic Air Monitoring System (TAMS). The TAMs utilized Tenax® adsorbent tubes as part of the distributive air volume methodology where four tubes are arranged in close proximity, each pulling a different air volume through the resin bed to help address breakthrough, artifact formation, and biases in the sampling methodology.

1.1.3 This method is designed to allow some flexibility in order to accommodate procedures currently in use. However, such flexibility also places considerable responsibility on the user to document that such procedures give acceptable results, i.e., documentation of method performance within each laboratory situation is required. Each contractor must generate standard operating procedures (SOPs) describing specific stepwise instructions for the Tenax® preparation and handling as well as the analytical procedures. SOPs should be readily available to and understood by all involved personnel.

1.1.4 This method is based upon procedures developed by the U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, NC and outlined in "Standard Operating Procedure for the GC/MS Determination of Volatile Organic Compounds Collected on Tenax®." Compounds which can be determined by this method are nonpolar organics having boiling points in the range of

approximately 80 to 200°C. However, not all compounds falling into this category can be determined. Exhibit C and Table D/VT-1 of this exhibit list the target compounds to be analyzed by this method. Other compounds (e.g., semi-polar) may yield satisfactory results but validation by the individual user is required.

1.2 Summary of Method

1.2.1 In the field, ambient air is drawn through an adsorbent cartridge containing approximately 1 to 2 grams of Tenax® adsorbent. While highly volatile organic compounds and most inorganic atmospheric constituents pass through the cartridge, certain volatile organic compounds are retained on the adsorbent bed. The cartridge is then tagged and transported back to the laboratory for analysis.

1.2.2 Prior to desorption and subsequent analysis by GC/MS, internal standards are added to the cartridges. During the preconcentration step, the VOCs trapped on the Tenax® are thermally desorbed by heating the sample cartridge under a flow of helium. The desorbed vapors are collected in a cryogenic trap which is cooled by liquid nitrogen temperature. The cryogenic trap containing the organics is then heated to transfer the sample to the capillary GC column, the front of which is cooled to liquid nitrogen temperatures (-150°C). This step is essential to focus the organic compounds and allow their application to the front of the capillary column in a discrete band.

1.2.3 The scan of the mass spectrometer is initiated and the analytical procedure is begun. Under a flow of helium, the GC column is programmed to a temperature to allow the elution of all of the organic compounds while the mass spectrometer is scanning. Data are recorded by the computer for subsequent processing.

1.2.4 Components are identified in a computerized library search routine, on the basis of the GC retention time and mass spectral characteristics. Quantitation is performed by the method of relative response factors, where the proportionate system responses for analyte and standard are determined prior to the analysis of the sample and this relative system response is used to determine the quantity of compound present on the sample cartridge.

1.2.5 Quantitative calculations are performed using the method of relative response factors. Data are reported as "ng/tube" since the entire contents of each cartridge is desorbed and analyzed in one GC/MS run. If the air sample volume in m^3 pumped through each sample cartridge is known to the laboratory, the concentration may also be reported in "ng/m^3."

1.3 Interferences and Limitations

1.3.1 In the use of Tenax® adsorbent, artifacts can arise from chemical reactions due to oxidants in the sample, degradation of the Tenax®, or thermal alterations of certain VOCs. This can usually be resolved by running blank and laboratory control samples prior to analysis and using multiple sampling volumes.

1.3.2 Excessive concentrations of water vapor on high humidity days may cause some changes in retention properties of Tenax®. In general, this can be minimized by multiple sampling volumes, smaller sampling volumes, and the use of desiccants in the culture tubes used for storage.

1.3.3 Contamination of the Tenax® adsorbent with the compound(s) of interest is a commonly encountered problem in the method. The user must be extremely careful in the preparation, storage, and handling of the cartridges throughout the entire sampling and analysis process to minimize this problem. Otherwise, false positive detection of chloroform, toluene, benzene, and other common volatile organics may occur. Precautions should be taken for sampling caustic atmospheres which contain levels of NO_x and molecular halogens greater than 2-5 ppm and 25 ppb, respectively.

1.3.4 Breakthrough volumes of the compounds of interest must be known or determined prior to use. Breakthrough volumes for some compounds (e.g., vinyl chloride, chloroform, etc.) may be so small that quantitative collection is impractical. Other characteristics of Tenax® which may adversely affect its preparation and use in this method are: (1) poor desorption of highly polar compounds; (2) possible retention of oxygen, leading to sample oxidation; and (3) high benzene background due to manufacturing.

1.3.5 Tenax® breakthrough volume also influences the linear range of the method, detection limits, and reproducibility. The linear range depends upon two factors. First, it is a function of the breakthrough volume of each specific compound and second, it is related to the limits of MS detection for each analyte. Thus, the range and limit of detection are a direct function of each compound which is present in the sampled air. The nominal linear range for quantitation using a capillary GC/MS/data system is generally two to three orders of magnitude (5-500 ng, 5-5,000 ng). Absolute limit of detection may vary from 0.1 ng to approximately 50 ng. Curvature of the calibration plot may begin at levels as low as 1000 ng and must be determined for each compound.

1.3.6 The reproducibility of this method is generally ±30 percent RSD, but depends on the chemical and physical nature of each analyte. The inherent analytical errors are a function of several factors: (1) the ability to accurately determine the breakthrough volume and its relation to field sampling conditions for each of the organic compounds identified;

(2) the accurate measurement of sample volume; (3) the percent recovery of the organic from the sampling cartridge after a period of storage; (4) the reproducibility of thermal desorption for a compound from the cartridge and its introduction into the analytical system; (5) the accuracy of determining the response factor ratios between the identified compound and the quantitat n standard used for calibrating the analytical system; and (6) the reproducibility of transmitting the sample through the high resolution GC column.

1.4 **Definitions**

NOTE: Definitions used in this test method and any user-prepared SOPs should be consistent with ASTM Test Methods D1356, B260, and E355. All abbreviations and symbols are defined within this document at the initial point of use.

1.4.1 Cryogen: A liquified gas used to obtain very low temperatures (-150°C) in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen.

1.4.2 Dynamic calibration: Calibration of an analytical system with calibration gas concentrations that are generated in a dynamic, flowing system, by metering known volumetric flow rates of concentrated gas standards and zero gas into a common inlet line to the system.

1.4.3 MS-SCAN: The GC is coupled to a mass selective detector where the instrument is programmed to acquire all mass data for the target compounds and to disregard all others.

1.4.4 Deuterated compounds: Those chemicals which contain deuterium (hydrogen isotope that is twice the mass of hydrogen) used as tracers for system quality assurance.

1.4.5 Static calibration: Calibration of an analytical system with known concentrations of calibrations gas, obtained from a source such as gas cylinders or prepared from standard stock solutions.

1.4.6 Retention time (RT): The time to elute a specific chemical from a chromatographic column for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until its maximum concentration appears at the detector.

1.4.7 Relative retention time (RRT): Ratio of RTs of two different chemicals for the same GC column and carrier gas flow rate, where the denominator represents the retention time for a reference chemical.

2 SAMPLE STORAGE AND HOLDING TIMES

2.1 Receipt of Exposed Cartridges

2.1.1 Receive all exposed Tenax® cartridge tubes in a sealed can with the appropriate Chain-of-Custody sheet. Match the Chain-of-Custody sheet with the corresponding sample to ensure that no mixup has occurred.

2.1.2 Check each Chain-of-Custody Sheet carefully for the following items: (1) a signature of a person relinquishing custody; (2) the amounts of surrogate loaded on the cartridge; (3) the sampling temperature and volume collected; and (4) the Tenax® batch number. Do not analyze any sample for which Chain-of-Custody sheet or any of the above information is missing.

2.2 Procedures for Sample Storage

2.2.1 Put all cans of samples in the cartridge freezer when received, and log each sample in the appropriate notebook as received. Place each Chain-of-Custody sheet in the project notebook (with all other information regarding that particular sample) after signing and dating it, and store any used cartridges in sealed cans so they can be recycled, cleaned, and used again.

2.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants. The sample cartridges should not be stored at elevated temperatures. High temperatures may result in leaking and thermal alteration of target compounds.

2.2.3 Samples and standards must be stored separately.

2.3 Contract Required Holding Times

Analysis of air samples must be completed within 14 days of the validated time of sample receipt (VTSR).

3 CARTRIDGE PREPARATION AND CERTIFICATION

3.1 Cartridge Preparation

3.1.1 Summary

The following routine shall be followed (as illustrated in Figure D/.T-5) in the preparation of Tenax® cartridges for air sampling:

3.1.1.1 Cleaning and preparation of Tenax® adsorbent;

3.1.1.2 Preparation of the empty tube and selection of the cartridge design to be used;

3.1.1.3 Packing of the Tenax® adsorbent into the tubes; and

3.1.1.4 Pre-conditioning of the Tenax® cartridges.

3.1.2 Apparatus and Materials

NOTE: All glassware must be soaked for at least one hour in Amway SA-8 laundry compound, or equivalent, followed by several rinses with Deionized water, and baking for a minimum of four hours at 500-550°C.

3.1.2.1 Extraction thimbles: Cellulose (60 mm x 180 mm).

3.1.2.2 Soxhlet extraction apparatus: Extraction flask and condenser Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA, 15219, or equivalent.

3.1.2.3 Aluminum shipping cylinder: 17.8 cm x 1.6 cm O.D., TEKMAR Co., P.O. Box 371856, Cincinnati, OH 45222.

3.1.2.4 Vacuum oven equipped with a dry ice trap and connected to water apparatus vacuum supply - Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA, 15219, or equivalent.

3.1.2.5 Beaker: 100-mL.

3.1.2.6 Variable transformer.

3.1.2.7 Heating mantle for 1000-mL flask.

3.1.2.8 Mettler balance: Type H15 for weighing Tenax®, Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA, 15219, or equivalent.

3.1.2.9 Desiccator with gas connectors and desiccant (Drierite).

3.1.2.10 Jar, wide-mouth, amber.

3.1.2.11 Crystallizing dish, Kimax®.

3.1.2.12 Aluminum foil.

3.1.2.13 Pyrex disks: For drying Tenax®.

3.1.2.14 Sieves: 40 and 60 mesh.

3.1.2.15 Glass funnel.

3.1.2.16 Cotton gloves.

3.1.2.17 Pre-washed glass wool, unsilanized.

3.1.2.18 Glass wool, silanized.

3.1.2.19 Teflon cap liners: 24 mm.

3.1.2.20 Stainless steel tweezers.

3.1.2.21 Screw caps: 24 mm.

3.1.2.22 Silicone septa: Teflon®-backed.

3.1.2.23 One-gallon clean metal paint cans: To hold clean Tenax® cartridges.

3.1.2.24 Stainless steel tubes: 10 cm x 1.6 cm O.D., TEKMAR Co., P.O. Box 371856, Cincinnati, OH 45222.

3.1.2.25 Glass jar: Capped with Teflon®-lined screw cap. For storage of purified Tenax®.

3.1.3 Reagents

3.1.3.1 Tenax®, 60/80 mesh (2,6-diphenylphenylene oxide polymer), GC or TA - Alltech Associates, Inc., Deerfield, IL 60015.

3.1.3.2 Granular activated charcoal: For preventing contamination of Tenax® cartridges during storage.

3.1.3.3 Acetone: Pesticide quality or equivalent.

3.1.3.4 Methanol: Distilled in glass.

3.1.3.5 n-Pentane: Distilled in glass.

3.1.4 **Tenax® Cleaning**

NOTE: The following adsorbent purification procedure is based on U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory (AREAL), Research Triangle Park, NC, Standard Operating Procedure (SOP) manual entitled "SOP for Preparation of Clean Tenax® Cartridges". Deviations from this procedure should be carefully evaluated before implementation into the user prepared SOP. Shorter cleaning procedures of the Tenax® have been documented. It is the laboratory's responsibility to provide documentation to the variation from this cleaning procedure to the Agency prior to implementation into the laboratory SOP.

3.1.4.1 All Tenax®, whether new or recycled, must be purified through solvent extraction and thermal treatment before it is used for sample collection of organic compounds. All glassware used in Tenax® purification as well as cartridge materials should be thoroughly cleaned by water rinsing followed by an acetone rinse and dried in an oven at 250°C.

3.1.4.2 To the batch of Tenax®, assign a unique number and record on the Tenax® Cleanup Worksheet, as illustrated in Figure D/VT-6. If possible, new Tenax® should be taken from a single batch that has been certified clean by the manufacturer.

3.1.4.3 If the Tenax® is new, also record batch number on the Worksheet. If the Tenax® is used, record previous Tenax® blank value and matrix in which Tenax® was used (i.e., fixed-site monitoring, breath or personal air).

3.1.4.4 In a hood, set up a sufficient number of Soxhlet extraction units, each with a 1000-mL round flask and a water-cooled condenser. Load approximately 50 g of Tenax® into each thimble and cover the Tenax® with approximately 2 cm of unsilanized glass wool.

3.1.4.5 Place the thimble in the Soxhlet, add 600 mL of methanol to the 1000-mL flask, and carefully pour an additional 300 mL of methanol onto the Tenax®.

NOTE: The 300 mL of extra methanol is added directly onto the Tenax® to ensure sufficient solvent for the extraction process after the initial adsorption of solvent.

3.1.4.6 Turn on the condenser water and the temperature-controlled heating mantle and record on the Tenax® Worksheet the date and time the extraction was started. After the first extraction cycle, adjust the temperature with the variable transformer to obtain five cycles per hour. Continue the extraction for 16-24 hours, checking the

extraction units twice daily and entering the information on the Worksheet.

NOTE: To avoid solvent losses, ensure that sufficient water is flowing to cool the condensers.

3.1.4.7 After 16 to 24 hours, cool the system and discard the methanol. With a pair of tweezers carefully pull out the thimble and let it drain in a 100-mL beaker for 10 minutes. Rinse the thimble with 50 mL of clean n-pentane. Repeat the rinse twice and then return the thimble to the Soxhlet. Discard the n-pentane.

NOTE: To avoid contamination, do not handle the thimble with your hands.

3.1.4.8 Transfer 700 mL of clean pentane to the flask. Reposition the Soxhlet and heat to reflux. Record in the Worksheet the date and time that the pentane extraction began. After the first cycle, adjust the temperature to obtain five cycles per hour.

3.1.4.9 Obtain and fill out a Tenax® cleanup worksheet, recording date and time of each operation. Complete the information on the Worksheet for this Tenax® batch, and continue the extraction for 16-24 hours. After extraction, cool the system to room temperature, remove the thimble from the Soxhlet with a pair of tweezers, and discard the pentane.

3.1.4.10 Place the beakers containing the thimbles in the desiccator at room temperature under a slow nitrogen flow (e.g., 25 mL/min) through a cryogenic trap to remove residual organics. After at least 24 hours, transfer the contents of the two thimbles to a large crystallizing dish. Cover the dish loosely with aluminum foil, set the dish in the vacuum oven, and recharge the cryogenic trap with dry ice/isopropanol and dry the Tenax® overnight at 100°C and slight vacuum.

3.1.4.11 After 24 hours, turn off the heater and allow the oven to reach room temperature (about 3 hours) before opening the oven. To open the vacuum oven, first close off the valve leading to the pump. Connect the nitrogen line to the other valve connector on the vacuum oven and slowly turn on the nitrogen flow with one hand while opening the valve with the other hand. Ensure that the nitrogen is vented out the oven through an activated charcoal tube.

3.1.4.12 Remove the Tenax® from the vacuum oven, open the valve leading to the pump and then immediately turn the vacuum pump off.

3.1.4.13 Store the Tenax® tubes, protected from the light, in a clean

wide mouth jar with Teflon®-lined cap.

3.1.4.14 Dry the rest of the Tenax® batch following the above procedures from step 3.1.4.10.

3.1.4.15 Combine the contents of the jars containing Tenax® from the same batch.

3.1.4.16 As an option, sieve the combined material and collect the fraction in the 40/60 mesh range. Return this fraction to the jar. Label the jar "sieved Tenax®" and indicate the date. Record this operation on the Worksheet.

3.1.5 Tube Preparation

NOTE: A 10-cm tube (stainless steel or glass) packed with Tenax® is referred to as a Tenax® cartridge. This section describes preparation of the empty cartridge prior to packing with the adsorbent.

3.1.5.1 The first step in preparing the cartridge is to select the type of design to be used. The most common design is shown in Figure D/VT-1a where the contact of the sample with metal surfaces is minimized. However, a disadvantage of this design is the need to rigorously avoid contamination of the outside portion of the cartridge since the entire surface is subjected to the purge gas stream during the desorption process. Clean cotton gloves must be worn at all times when handling such cartridges and exposure of the open cartridge to ambient air must be minimized.

3.1.5.2 A second common type of design is shown in Figure D/VT-1b. This design eliminates the need to avoid direct contact with the exterior surface, since only the interior of the cartridge is purged.

3.1.5.3 For adsorbent packing, a multibed option has been developed by Supelco, as illustrated in Figure D/VT-1c. The tube contains three adsorbent beds to capture the more volatile organics which Tenax® cannot retain.

3.1.5.4 Regardless of the cartridge design chosen, the thermal desorption module and sampling system must be selected to be compatible with that particular cartridge design.

3.1.5.5 Place the Teflon® liners in a beaker and sonicate them in methanol for 10 minutes, and rinse the liners with fresh methanol.

3.1.5.6 Repeat the above with n-pentane instead of methanol.

3.1.5.7 Dry the Teflon® liners in the vacuum oven for five hours at

100°C and slight vacuum. Store the liners in a wide-mouth jar, protected from light.

NOTE: To avoid contamination of the Teflon®, always use a pair of tweezers to handle the liners.

3.1.5.8 Clean the silicone septa by the above procedures from step 3.1.5.5.

3.1.5.9 Soak the 24-mm screw caps in methanol for 30 minutes, then remove the paper-lined foil from the caps with a spatula. Rinse the caps in clean methanol and dry them in the vacuum oven overnight at 100°C.

3.1.5.10 Wrap the Kimax® culture tube with aluminum foil and secure it with clear tape and place a 4-cm glass wool plug at the bottom of the culture tube.

3.1.5.11 Place a silicone septum in the screw cap. Cover the septum with a cleaned Teflon-liner, and loosely close the culture tube with the screw cap.

3.1.6 Tube Packing

NOTE: To avoid contamination of the Tenax® and the tube, always use a pair of tweezers and a pair of gloves.

3.1.6.1 Carefully inspect the tubes before packing. Discard glass tubes with rough ends or cracks.

3.1.6.2 Set the tubes in a rack and insert a 4-cm glass wool plug into one end of the tube and press the glass wool plug lightly into place with a dowel.

3.1.6.3 Using a glass funnel, transfer a known amount (approximately 2 g) of Tenax® to the tube.

3.1.6.4 Insert another 4-cm glass wool plug into the other end of the tube (see Figure D/VT-1) and lightly compress it with a dowel.

3.1.6.5 Store the Tenax® cartridges in the prepared culture tubes until pre-conditioning.

3.1.7 Tenax® Cartridge Pre-conditioning

3.1.7.1 Place the Tenax® cartridge into the conditioning unit and turn on the helium tank. This allows oxygen to be purged from the cartridge before heating.

3.1.7.2 Turn on the desorption unit to 250°C, place liquid nitrogen in the cryogenic trap, and open the helium line to the desorption chambers.

NOTE: Ensure that a cryogenic trap has been placed in the helium line to remove residual organics.

3.1.7.3 Adjust the helium flow under each chamber to approximately 15 mL/min., then condition the Tenax® cartridges for five hours at 250°C. Make sure that helium flow is maintained to each cartridge throughout desorption.

3.1.7.4 Refill the cryogenic trap with liquid nitrogen every hour, or when the level of liquid nitrogen is less than one-third full.

NOTE: If liquid nitrogen in the trap is depleted, all the impurities trapped in the line will be transported to the Tenax®.

3.1.7.5 Record all pertinent information on the Tenax® Cleanup Worksheet for specific Tenax® batch.

3.1.7.6 Allow cartridges to cool to room temperature under the helium flow. Remove each cartridge with a pair of tweezers and immediately place the hot cartridge in a shipping container.

3.1.7.7 Seal the tube and label the screw cap with the Tenax® batch number and the culture tube with the pre-conditioning date, and place in a tightly sealed friction-top container. (For cartridges of the type shown in Figure D/VT-1a, the culture tube, not the cartridge, is labeled).

3.1.7.8 The Tenax® cartridges are now ready for certification.

3.2 Tenax® Cartridge Certification

3.2.1 Summary

The Tenax® cartridges are analyzed by GC/MS or GC/FID to ensure the integrity of the cleaning and desorbing procedures. While analysis can be accomplished by GC/MS, laboratories may choose to use GC/FID due to logistical and cost considerations.

3.2.2 Frequency

Initially, all Tenax® cartridges must be checked after cleaning/preparation to establish the percentage that pass the cleanliness criteria. A total of forty individual cartridges should be analyzed in ten batches to check for individual cartridge contamination and for batch contamination by the cleaning apparatus.

If and when only two or less individual cartridges are contaminated and no batch contamination is evident, the laboratory may reduce the number of cartridges tested for cleanliness after cleaning, but must continue to check 10 percent of the cartridges or one cartridge from each Tenax® cartridge batch, whichever is greater.

3.2.3 Procedure

3.2.3.1 Calibrate the GC/FID or the GC/MS system that has met all the tuning criteria, using a single injection of the mid level (CAL 3) standard containing all the target and surrogate compounds, prepared according to one of the calibration standard preparation procedures described in Section 4.

3.2.3.2 Analyze the clean, preconditioned cartridges following the desorption and preconcentration method outlined in section 5.6.

3.2.3.3 Cartridges should be used for sampling within two weeks after preparation and analyzed within two weeks after sampling. If possible, the cartridges should be stored at -10°C in a clean freezer (i.e., no solvent extracts or other sources of volatile organics are contained in the freezer).

3.2.3.4 The certified cartridges are considered clean for a period of two weeks after certification. If a certified cartridge is stored for a period of two weeks or more after being successfully cleaned, it must be reconditioned according to section 3.1.7 above; however, no subsequent analytical confirmation of cleanliness is required before using.

3.2.4 Calculations

For the GC screening analysis, target compound concentrations are determined using the external standard quantitation method. Use the following equation to determine target compound concentration levels:

$$Concentration,\ ng/tube = \frac{(A_x)\,(C_s)}{(A_s)} \qquad \text{EQ. D/VT-1}$$

where: A_x = peak response of analyte in the sample tube;
A_s = peak response of analyte in the calibration standard; and
C_s = amount of analyte in the injected calibration standard, ng.

3.2.5 Technical Acceptance Criteria

3.2.5.1 Each batch of Tenax® cartridges prepared must be checked for contamination by analyzing 10 percent of the cartridges or at least

one cartridge in each batch, whichever is greater, immediately after preparation. The analyzed cartridge(s) must not contain any of the target VOCs at levels greater than the CRQL, and the total level of VOCs per cartridge must not exceed 10 ng.

3.2.5.2 While acceptance criteria can vary depending on the components of interest and anticipated concentration level, at a minimum, the clean cartridge should be demonstrated to contain less than the CRQL of each component. For most compounds, the blank level should be less than 10 ng total VOCs per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory.

3.2.6 Corrective Action

3.2.6.1 Any cartridge with a target analyte concentration greater than the its CRQL or a total concentration greater than 10 ng (target and non-target) shall be recleaned and reanalyzed subject to the same criteria for cleanliness, or be set aside.

3.2.6.2 If a cartridge does not meet these acceptance criteria, the entire batch should be rejected.

3.2.7 Documentation

Results of the certification of the Tenax® cartridges shall be reported on Form VIII-AAVT.

3.3 Supplying Cartridges for Collection of Samples

3.3.1 As a quality assurance indicator, all Tenax® cartridges shall be spiked with three surrogate standards (100 ng each of benzene-d_6, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4) as indicators of performance during sampling and analysis.

3.3.2 The surrogate compounds can be added to the adsorbent cartridge by either flash vaporization, static dilution, or by the permeation gas generation (see Section 4). The same spiking technique must be employed for all samples, blanks, and standards in an analytical sequence.

3.3.3 Prepare the Chain-of-Custody and Field Data Sheet for all samples to be collected. Remove and label the requested number of Tenax® cartridges from the Tenax® storage area. Store all of the labeled Tenax® cartridges in the Tenax® storage area until needed.

NOTE: If more than one Tenax® batch number has been assigned per matrix, use Tenax® from same batch for all the field and duplicate samples.

4 CALIBRATION STANDARDS PREPARATION PROCEDURES

4.1 Flash Vaporization

4.1.1 Summary

4.1.1.1 A dilute solution of one or more organic compounds in methanol is injected into a heated zone in a helium stream. The methanol and the solute compounds are rapidly vaporized and then swept onto a sorbent cartridge. Methanol has little affinity for Tenax® sorbent and is rapidly eluted from the cartridge while the target VOCs are retained in the cartridge.

4.1.1.2 The solute compounds remain in the sorbent bed when the cartridge is removed from the flow system, and may subsequently be desorbed from the cartridge and delivered to an analytical instrument for analysis.

4.1.1.3 Since the quantity of each compound in the cartridge can be determined from its concentration in the solution and the volume of solution injected, this method may be used to spike quantitative standards on sorbent cartridges.

4.1.2 Interferences

4.1.2.1 Contamination of the methanol solvent with compounds to be calibrated or with compounds producing similar instrumental responses will result in false high or false positive responses.

4.1.2.2 Chemical reactions between compounds can deplete them from the mixture and might also result in unexpected reaction products. Absorption of a compound into the matrix of sorbent particles will probably result in part of it being retained in the cartridge during desorption, with consequent decreased response.

4.1.2.3 Use of a syringe for consecutive injections from the same bottle without cleaning after each injection may result in erratic responses due to buildup of sample residues in the syringe. Rinse individual syringes with methanol and acetone and dry in a vacuum syringe cleaner for approximately 30 seconds. (A heat gun is used to heat the barrel of the syringe during vacuum drying.)

NOTE: Syringes must be rigorously cleaned after each injection to remove traces of sample. Even if more than one injection is needed from any given source, a freshly cleaned syringe must be used for each injection. Failure to do so may result in erratic responses.

4.1.3 **Apparatus and Materials**

4.1.3.1 Flash vaporization unit (see Figure D/VT-3).

4.1.3.2 Liquid microsyringes: 5-, 10-, 50-, and 100-µL for injecting liquid standards into flash vaporization system.

4.1.3.3 Volumetric flasks: 25-, 50-, 100-, 250-mL.

4.1.3.4 Helium cylinder and pressure regulator and needle valves for controlling flow rate.

4.1.3.5 Flow meter (i.e., soap bubble, rotameter).

4.1.3.6 Thermal conductivity detector.

4.1.3.7 Vacuum syringe cleaner.

4.1.4 **Procedure**

4.1.4.1 Assemble the flash vaporization unit, as illustrated in Figure D/VT-3.

4.1.4.2 Adjust the helium flow to 30 mL/min and the heating mantle to 310 ± 10°C.

4.1.4.3 Allow the helium to flow for approximately 30 minutes to equilibrate the system.

4.1.4.4 The volume of helium required to elute methanol from a sorbent cartridge is determined by using a thermal conductivity detector. Several different flow rates are tried to find one which results in as sharp a methanol peak as possible without sweeping volatile solutes out of the cartridge before it can be removed from the system.

4.1.4.5 Set the helium flow to 30 mL/min and the heater to 310° ± 10°C. Place a clean Tenax® cartridge in line, and pass helium through the cartridge for a period of 5 minutes.

4.1.4.6 Using a heated syringe, retrieve from the individual standard flask an aliquot using the solvent flushing technique.

4.1.4.7 With the aliquot of the standard in the syringe, inject smoothly, at the standard injection point, the syringe contents over a period of about 5 seconds. Allow the helium containing the injection standard to pass through the cartridge for 50 minutes or until 1500 mL of helium has passed. Remove the cartridge from the system, cap, and store at 5°C.

4.1.5 Calculations

4.1.5.1 The approximate volume of solution to be injected is calculated by working backward from the size of the spike to be placed in the sorbent cartridge. An example of this calculation follows.

- If a 500 ng spike is needed, it could be done by injecting 10 µL of methanol containing 50 ng/µL of solute. Therefore, 10 µL x 50 ng/µL solution = 500 ng.
- A solution containing 50 ng/µL of solute is prepared by dissolving 5 mg of neat compound in a 100-mL volumetric flask and diluting to mark with methanol.
- If the density of the neat compound is 0.9726 g/mL (0.9726 mg/µL), then the measured neat compound would be 5 mg/(0.9726 mg/µL) = 5.14 µL.
- Therefore, 5.14 µL of solute measured with a syringe would produce 50 ng/µL solution when diluted to 100 mL with methanol
- It is not practical to measure fractions of microliters, so usual practice would be to dissolve 5 µL of sample in 100 mL of methanol to produce a concentration of: (0.9726 mg/µL x 5 µL)/100 mL = 0.0486 mg/mL = 48.63 ng/µL.
- A 10 µL aliquot of this solution would contain 486.3 ng.

4.1.5.2 As a further example, a typical mixture can be prepared as follows:

Standard	Density g/mL	Volume µL	Weight mg	Deliverable Volume µL	Spiked on Cartridges ng
ethylbenzene	0.867	11.0	9.54	3	286
p-xylene	0.861	12.0	10.33	3	310
acetophenone	1.028	10.0	10.28	3	308
2-nonanone	0.821	12.0	9.85	3	296

Each compound is measured into a 100-mL volumetric flask using a microsyringe. The flasks are filled to the mark with spectrographic grade methanol and the contents mixed thoroughly. Three microliters (3 µL) each of these solutions, when injected into the flash vaporization unit, will deposit approximately 100 ng of each compound on a sorbent cartridge as shown in the table above. The solution must be used within 12 hours.

4.2 Static Dilution Bottle Technique

4.2.1 Summary

4.2.1.1 A quantity of liquid organic compound is injected into a 2-L round bottom helium-filled flask through a septum cap. After injections are completed, the flask is agitated and heated to achieve complete vaporization.

4.2.1.2 Aliquots of the resulting vapor are then delivered to sorbent cartridges or analytical instruments. The weight of each compound delivered is calculated from: (1) the density of the liquid; (2) the volume of liquid injected into the known volume of the bottle; and (3) the volume of the vapor aliquot removed.

NOTE: The quantity of any compound injected into the dilution flask must be substantially less than that which would result in a partial pressure equal to its vapor pressure at ambient temperature. Vaporization of liquid aliquots injected into the bottle must not result in a large positive pressure, and removal of vapor aliquots from the flask must not result in a substantial vacuum. If these precautions are not taken erratic responses may occur.

4.2.1.3 The static dilution bottle technique for preparing standards has been validated for the following 22 compounds:

Acetophenone	1-Ethenyl-4-chlorobenzene
Benzonitrile	3-Chloro-1-propene
1,1,1,2-Tetrachloroethane	1,4-Dichlorobutane
1,4-Dioxane	1,2,3-Trichloropropane
1-Chloro-2,3-epoxypropane	1,1-Dichloroethane
1,3-Dichlorobutane	2-Chlorobutane
1,4-Dichlorobenzene	2-Chloroethoxyethene
cis-1,4-Dichloro-2-butene	1-Methylethylbenzene
3,4-Dichloro-1-butene	1,3,5-Trimethylbenzene
Perfluorotoluene	Butylbenzene
Fluoroiodobenzene	1-Methyl-4-(1-Methylethyl)benzene

4.2.1.4 Amounts used have ranged between 0.3 and 4 μL of liquid samples. Repeatability of daily injections of a mixture of the 22 compounds into the GC/MS is about ±10 percent RSD. Precision depends on the substance introduced, the skill of the individual producing the flask standard, and the skill of the operator of the instrument used to analyze the flask contents. Accuracy has not been established.

4.2.2 Interferences

4.2.2.1 Adsorption of vapor molecules on the walls of the bottle or

on the septum will result in loss of material, with a consequent decrease in response. This is especially likely when new, freshly annealed bottles are used. Contamination of apparatus may result in adsorption loss or provide unexpected sources of compounds in a mixture.

4.2.2.2 Chemical reactions between compounds can deplete them from the mixture and might also result in unexpected reaction products. Use of a syringe for consecutive injections from the same bottle without cleaning after each injection may result in erratic responses due to buildup of sample residues in the syringe. Rinse individual syringes with methanol and acetone and dry in a vacuum syringe cleaner for approximately 30 seconds. (A heat gun is used to heat the barrel of the syringe during vacuum drying.)

NOTE: Syringes must be rigorously cleaned after each injection to remove traces of sample. Even if more than one injection is needed from any given source a freshly cleaned syringe must be used for each injection. Failure to do so may result in erratic responses.

4.2.3 **Apparatus and Materials**

4.2.3.1 Round-bottom flask (2-L) containing 30 3-mm diameter glass beads and a 1-in. Teflon®-coated magnetic stirring bar - the flask is modified to accept a screw-on Mininert septum cap, TEKMAR Co., P.O. Box 371856, Cincinnati, OH 45222.

4.2.3.2 Gas-tight glass microsyringes: 10-, 25-, 50-, 100-, 500-, 1000-, and 2500-μL.

4.2.3.3 Laboratory oven: large enough to contain at least two dilution bottles and capable of maintaining 60 ± 5°C.

4.2.3.4 Drying oven capable of 300°C.

4.2.3.5 Helium cylinder and pressure regulator connected to a length of flexible tubing.

4.2.3.6 Vacuum syringe cleaner.

4.2.3.7 Magnetic stirrer.

4.2.3.8 Heat gun.

4.2.3.9 50-mL vial fitted with a septum cap.

4.2.4 **Procedure**

4.2.4.1 Wash the 2-L flask with detergent and water, rinse several

times with deionized water, and dry in an oven at 300°C for 4 hours.

4.2.4.2 Place 30 3-mm glass beads inside the flask and weigh on an analytical balance to an accuracy of 0.01 g.

4.2.4.3 Fill the flask with deionized water to the level of the septum cap.

4.2.4.4 Weigh the flask containing the glass beads and water on an analytical balance to an accuracy of 0.01 g.

4.2.4.5 The weight of the water required to fill the bottle is the difference between the two weights, as calculated below:

$$V_f = Wt_f - Wt_i \qquad \text{EQ. D/VT-2}$$

where: V_f = volume of flask, mL;
Wt_f = final weight of flask with beads and water, g; and
Wt_i = initial weight of flask with beads, g.

4.2.4.6 Two methods have been used to load the dilution flask with organic components for standards - direct injection of each compound separately into the flask and a single direct injection of a previously prepared mixture of compounds. These methods have been shown to produce indistinguishable results.

- The first method involves injecting each compound (one at a time) into the flask. The flask is inverted after each injection with the syringe in place through the septum, in order for the beads to remove any liquid remaining on the syringe needle. The second method involves preparing a master solution by injecting 1 mL of each component into a culture vial fitted with a septum cap. After all the compounds have been added, the vial is agitated to produce a homogeneous liquid mixture. The vial is then recapped with a new septum. Aliquots of this master solution are removed and injected into the dilution flask as needed in the same manner as indicated above; and

- The second method involves retrieving a clean, dry 2-L flask containing 70 3-mm glass beads and flushing the flask with helium for a period of five minutes. At the end of the flushing process, immediately cap with a Mininert septum cap. Place the 2-L flask on a magnetic stirring apparatus and set at the maximum speed. Using the syringes, inject the calculated volume of each compound (one at a time) or from the mixture solution into the flask while the glass beads are agitated by the stirring bar at the maximum setting of the magnetic stirrer. Invert the flask after each injection with the syringe in place through the septum,

in order for the beads to remove any liquid remaining on the syringe needle. After all substances have been introduced, place the flask in the oven at 60°C for 30 minutes to equilibrate. Store the flask in the oven at 60°C until needed. Bottles are stable for one week after preparation.

NOTE: The technique of injecting a solution of targeted compounds rather than individual injection of specific compounds is preferred if many substances are involved, because it is more rapid, and the master solution can be used over a long period if it is refrigerated at 0°C. Before use, the refrigerated solution is allowed to sit at room temperature for about an hour. It is recommended that a total less than 90 μL of liquid be injected and a total less than 20,000 μL of gas be removed.

4.2.4.7 Remove the flask from the oven and place on the magnetic stirrer for approximately 15 seconds.

4.2.4.8 Place the syringe to be used in the extraction procedure in the oven at 60°C to prevent condensation in the syringe during delivery. Using the heated syringe, insert its needle through the septum and pump three times slowly.

4.2.4.9 After the third pump, fill the syringe to approximately 25 percent greater volume than needed. After a 5-second pause, withdraw the needle from the Mininert septum valve. Flush the excess sample from the syringe, then draw a small quantity of air into the syringe to retard diffusion of sample through the syringe tip. The aliquot sample must be used immediately.

4.2.4.10 If the aliquot is to be injected into a clean sorbent cartridge, the tip of the needle is inserted to the center of the sorbent bed. Then the plunger is depressed over a 10 second period while the needle tip is being withdrawn about half the distance to the end of the bed.

4.2.4.11 If the aliquot is injected directly into the analytical instrument, injection is made in the normal manner unless column-head freeze-trapping (cryofocusing) is being employed, in which case the plunger is depressed over about a 10 second period.

4.2.4.12 If an aliquot is too large to be injected in one step, two or more injections may be made. This causes no complication for injection into a sorbent cartridge, but cryofocusing must be employed when multiple injections are made directly into a gas chromatograph.

4.2.5 Calculations

4.2.5.1 Volumes to be introduced into the 2-L flask are calculated by

working backwards from the quantity of material to be delivered. For example, if a 500 ng delivery is needed, it could conveniently be accomplished by using a 50-µL syringe containing 10 ng/µL of compound. Therefore:

$$50\ \mu L \times 10\ ng/\mu L = 500\ ng$$

4.2.5.2 If the typical volume of the flask is 2.065 L, then to get that concentration (10 ng/µL) in the flask, one would have to add 20.65 mg of liquid compound to the flask. The calculation would therefore involve: 10 ng/µL x 2.065 L = quantity of liquid needed to develop a flask concentration of 10 ng/µL.

$$10\ ng/\mu L \times 2.065\ L = 20.65\ mg$$

4.2.5.3 If the density of the solution was 0.9726 g/mL (or 0.9726 mg/µL), then the volume of solution needed to add to the flask to maintain a concentration of 10 ng/µL or a deliverable of 500 ng would be 21.23 µL, as calculated below:

$$20.65\ mg/(0.9726\ mg/\mu L) = 21.23\ \mu L$$

4.2.5.4 It is not practical to deliver and measure fractions of a microliter; therefore, in practice, 21 µL would be used. The deliverable would be calculated:

$$(21\ \mu L \times 0.9726\ mg/\mu L)/2065\ mL = 0.00989\ mg/mL = 0.00989\ \mu g/\mu L$$

4.2.5.5 This is equivalent to 9.89 ng/µL, so a 50-µL injection of the vapor compound from the static dilution flask would contain 494.5 ng of compound delivered as calculated below:

$$9.89\ ng/\mu L \times 50\ \mu L = 494.5\ ng$$

4.3 Permeation Calibration Generator

4.3.1 Summary

4.3.1.1 A permeation calibration generator is designed to allow the permeation of gas through Teflon® or other plastic material at a constant rate in a water bath at constant temperature to generate test atmospheres.

4.3.1.2 The permeation tube is made by sealing a liquid chemical in a tube made of some permeable material. It is essential that the chemical be in the liquid state for the permeation tube to operate properly. In many cases the chemical is a gas at atmospheric pressure, but is maintained in the liquid state under its own saturation vapor pressure in the permeation tube. The tube is sealed

at both ends with a non-permeable plug.

4.3.1.3 Permeation of the vapor within the tube occurs through the exposed sidewalls because of the concentration gradient that exists between the inner and outer tube walls. By passing different flows of diluent gas over the tube, gases of varying concentration can be generated. If the tube is held at a constant temperature, the permeation rate will remain constant. By measuring the weight loss at this constant temperature over a given period of time, the permeation rate may be determined. The output rate of the tube will remain essentially constant until nearly all of the liquid in the tube has permeated through the walls. In general, permeation tubes can be used to generate known concentrations of target compounds between 0.7 to 200 ppbv.

4.3.2 Apparatus and Materials

4.3.2.1 Permeation system (see Figure D/VT-4).

4.3.2.2 Nylon gloves.

4.3.2.3 Long glass hook (for retrieving permeation tubes).

4.3.2.4 Permeation tubes.

4.3.2.5 Lint-free tissues (e.g., Kimwipes®).

4.3.2.6 Stopwatch.

4.3.3 Procedure

4.3.3.1 Before a permeation device can be used in the laboratory or in the field, its permeation rate must be determined. The permeation rate, R, is determined gravimetrically. The tube is weighed, then placed in a temperature bath (±1°C) for a period of time. The tube is removed and reweighed. This process is repeated over several days to calculate a permeation rate at that specific temperature. The difference between initial and recorded weight (ng), divided by time (min) determines the permeation rate at that specific temperature.

$$R = \frac{W}{T} \qquad \text{EQ. D/VT-3}$$

where: R = Permeation rate, ng/min;
W = Weight change, ng; and
T = Time, minutes.

4.3.3.2 The permeation rate can be calculated either manually, as

shown in the above equation, or recorded automatically. At different temperatures, different permeation rates can be calculated.

4.3.3.3 Permeation tubes should be kept at the temperature specified by the manufacturer and at a constant temperature (±0.05°C) during calibration procedures. Changes in temperature as small as 0.1°C can significantly affect the permeation rate. Tubes should initially be allowed to equilibrate for 24 hours. After small changes in temperature (1 to 5°C), the tube should be allowed to equilibrate for at least half an hour.

4.3.3.4 A permeation tube system has been developed for application of loading known standards onto Tenax® cartridges for use in determining the relative response factor and the column performance evaluation of the GC/MS-DS system in conjunction with the flash vaporization system.

4.3.3.5 In addition, the permeation tube system may be used for generating internal standards to be loaded by syringe onto the Tenax® tube to determine relative retention times, relative response factors, and stability of the GC/MS system and for generating surrogate standards used in the evaluation of breakthrough volumes associated with Tenax®.

4.3.3.6 A permeation system consists of three main parts (see Figure D/VT-4): (1) a temperature-controlled chamber containing permeation tubes; (2) a mixing chamber; and (3) permeation tube storage chamber. A stream of nitrogen flows through the system. The amounts of compounds transported downstream remain constant once the system has become equilibrated with the compounds to be loaded. The amount of compounds can be determined by measuring the time and the gas flow through the cartridge.

4.3.3.7 The permeation system may be used to load any volatile compound that will permeate at a constant rate under controlled conditions, and to inject a calibration standard onto a sorbent via syringe.

NOTE: Because the compounds in the permeation tubes may be toxic or contain suspected carcinogen, the Permeation Generator Assembly should be housed in a properly ventilated hood.

4.3.3.8 The following routine should be followed when Tenax® cartridges are loaded with standards via a permeation system: (1) determine the number of cartridges to be loaded, 2) select the permeation tubes; (3) determine the loading conditions to be used; (4) equilibrate the system (5) load the cartridges; (6) calculate the amounts of compounds loaded (7) ensure the integrity of the loading

procedure; and (8) pack and store the cartridges.

- Obtain a copy of the field sampling schedule from the Monitoring Coordinator or Program Manager;
- Determine the number of standards to satisfy the sampling objectives;
- Check the permeation notebooks (located in the laboratory) to see which permeation tubes are available for the needed standards; and
- Select only the permeation tubes whose permeation rates are stable.

NOTE: A permeation rate is considered stable when the mean permeation rate has a coefficient of variation (CV) of less than 10 percent. The mean permeation rate is calculated using the last five individual permeation rates. Do not use permeation tubes with permeation rates below 100 ng/min or above 1×10^5 ng/min.

- In a bound notebook assigned for the specific project, prepare a table including the numbers of the tubes to be used, the names of the compounds, and the corresponding mean permeation rates.

4.3.4 Calculations

4.3.4.1 For any compound, calculate the amounts needed to be loaded onto a Tenax® cartridge using the following formula:

$$G = \frac{(P)(t)(F_1)}{(F_1 + F_2)} \qquad \text{EQ. D/VT-4}$$

where: G = Amount loaded of compound onto Tenax® tube;
P = Permeation rate of specific compound, ng/min;
t = Loading time of compound onto Tenax® tube, minutes;
F_1 = Flow rate through the Tenax® cartridge, mL/min; and
F_2 = Exhaust flow rate, mL/min.

NOTE: The four variables G, t, F_1, and F_2 determine the loading conditions. Any three may be fixed and the fourth one calculated from the equation.

4.3.4.2 The following restrictions must be followed to minimize error: (1) do not load for less than two minutes; (2) do not load with a cartridge flow below 50 mL/min or above 150 mL/min; and (3) do not operate the system with a total flow below 250 mL/min.

4.3.4.3 If the GC/MS system needs to operate in the range of 200-500

ng per analyte, then the analyst must generate standards concurrent with that range. Fixing three of the four variables of the above equation will enable calculation of the needed loading onto the Tenax® tube.

4.3.4.4 As an example, the following calculations are provided to assist the user in determining operating parameters of the permeation tube system in generating standards on QA/QC checks.

OBJECTIVE: To load chlorobenzene and chloroform onto a cartridge in the range of 200-500 per analyte.

GIVEN: G = 200 ng per analyte
F_1 = 80 mL/min
t = 4 min
P = 270 ng/min for chlorobenzene
= 520 ng/min for chloroform

Chlorobenzene

$G = (P)(t)[F_1/(F_1+F_2)]$
200 ng = [(270 ng/min)(4 min)] x [(80 mL/min)/(80 mL/min+F_2)]
F_2 = {[(270 ng/min)(4 min)(80 mL/min)]/200 ng) - 80 mL/min
F_2 = 352 mL/min

Now, since all tubes are in the permeation device together, the flow (F_2) for chloroform will be 352 mL/min. Therefore, the loading on the Tenax® tube for chloroform must be calculated to verify that it falls within the 200-500 ng per tube loading.

Chloroform

$G = (P)(t)[F_1/(F_1+F_2)]$
G = (520 ng/min)(4 min)[(80 mL/min)/(80 mL/min + 352 mL/min)]
G = 385 ng

All values obtained are within the acceptable range.
NOTE: Permeation tubes may contain toxic or carcinogenic materials. The following procedures should be carried out in a properly ventilated glove box or hood. Wear nylon gloves when handling permeation tubes.

4.3.4.5 Locate the chambers in which the selected permeation tubes are stored. With a long glass hook, remove the selected permeation tubes from the storage chamber and transfer immediately to the loading chamber of the permeation system.

NOTE: When a cartridge is not being loaded, a dummy cartridge is placed in the loading position.

4.3.4.6 Direct nitrogen flow to the side where the Tenax® cartridge will be loaded and allow the system to equilibrate for 30 minutes before loading cartridges with the generated test atmospheres.

4.3.4.7 Divert the nitrogen flow to the side that will not be used for loading cartridges and insert the Tenax® cartridge into the chamber.

4.3.4.8 Start the stopwatch and immediately direct the test atmospheres gas flow through the cartridge.

4.3.4.9 Calculate the time needed to load the amounts desired as follows:

$$t = (G/P) \times [(F_1 + F_2)/F_1]$$

NOTE: Base the calculation only on the compound whose permeation tube has the highest or lowest permeation rate.

4.3.4.10 At the calculated time, rotate the two stopcocks to direct gas flow away from the cartridge being loaded.

4.3.4.11 Handling the cartridge with a Kimwipe®, remove the cartridge and return it to its culture tube. Seal the tube, then label with the following information: (1) project number; (2) standard mixture followed by a number indicating the order of loading; and (3) the date.

4.3.4.12 For every compound, calculate the amount loaded onto a cartridge using the general formula:

$$G = (P)(t)[F_1/(F_1 + F_2)]$$

4.3.4.13 Deliver the loaded permeation tubes to the GC/MS for use in the quality assurance program.

5 GC/MS ANALYSIS OF VOLATILES FROM TENAX® CARTRIDGES

5.1 Summary

5.1.1 This section includes a description of the GC/MS system and associated procedures for GC/MS tuning, characterizing the performance of the GC/MS calibration procedures, and analysis of the exposed Tenax® cartridges and QC samples.

5.1.2 The analytical system is comprised of a GC equipped with a mass spectrometer set in the full scan mode. The GC/MS is set up for automatic, repetitive analysis and programmed to acquire data for the target compounds. The Contract Required Quantitation Limits (CRQL) range from 1 to 50 ng in the full scan mode with an analytical precision of about 5 percent RSD. Concentration of compounds based upon a previously installed calibration table is reported by an automated data reduction program.

5.1.3 The relative response factor (RRF) from the continuing calibration standard analysis is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion.

5.1.4 Quantitation is based on integration of the SICP of a quantitation mass or primary ion for the compound. This mass has been previously selected for each compound based on its spectral uniqueness, intensity, and lack of potential interferences from known coeluting compounds. Currently used masses are listed in Table D/VT-5 for both primary and secondary ions.

5.2 Apparatus and Materials

NOTE: Considerable variation from one laboratory to another is expected in terms of instrument configuration. Therefore, each laboratory must be responsible for verifying that their particular system yields satisfactory results. Section 6 discusses specific performance requirements which must be met.

5.2.1 Sampling/Preconcentration System

5.2.1.1 Sample Desorption/Injection Unit: Designed for thermally heating a Tenax® sample cartridge (glass or stainless steel) for sample transfer into a suitable GC/MS system for analysis. The configuration of the thermal desorption unit should permit the enclosure and rapid heating of the Tenax® cartridge from room

temperature to approximately 250°C while purging with an inert gas (helium) into a cryogenically cooled (liquid nitrogen) trap. The cryogenically cooled sample must then be rapidly heated to a preselected temperature (200-250°C) and a helium gas supply allowed to sweep the sample from the trap onto the gas chromatographic column. A schematic diagram of a typical thermal desorption/GC/MS configuration is shown in Figure D/VT-2.

5.2.1.2 A block diagram of the typical system required for analysis of Tenax® cartridges is shown in Figure D/VT-2. The thermal desorption module must be designed to accommodate the specific cartridge configuration used in the sampling protocol. Steel or nickel metal surfaces should be employed. The volume of tubing and fittings leading from the cartridge to the GC column must be minimized and all areas must be well-swept by helium carrier gas.

5.2.1.3 Sample cartridge: Sampling cartridges consist of 13.5 x 100 mm borosilicate glass with polished-flat end surfaces. One end is etched with an I (inlet) and the other with an E (exit). Stainless steel cartridges (12.7 mm OD x 100 mm long) may also be used. (see Figure D/VT-1). Cartridges must fit into the thermal desorption unit.

5.2.1.4 Glass fiber filters, 25 mm (optional).

5.2.1.5 Forceps.

5.2.1.6 Lint-free tissues: (e.g., Kimwipes®).

5.2.1.7 Filter holder: Stainless steel or aluminum (to accommodate 1 inch diameter filter).

5.2.1.8 Cotton gloves: For handling Tenax® cartridges.

5.2.2 GC/MS System

5.2.2.1 The GC/MS system should be capable of subambient temperature programming, exhibit unit mass resolution up to 400 amu, and be capable of scanning a 35-300 amu region every second. Equipped with data system for instrument control as well as data acquisition, data processing using spectral enhancement algorithms, and historical library screening and storage.

5.2.2.2 The GC column required for this method is fused silica, 0.30 mm ID x 50 m, SE-30 or OV-1 coating. The GC column inlet should be capable of being cooled to 10°C and subsequently increased rapidly to approximately 30°C. This can be most readily accomplished using a GC equipped with an automated subambient cooling capability (liquid nitrogen), although other approaches such as manually cooling the

inlet of the column with a cotton swab containing liquid nitrogen may be acceptable.

5.2.2.3 The specific GC column and temperature program employed will be dependent on the specific compounds of interest. Appropriate conditions are described in Table D/VT-2. In general a nonpolar stationary phase (e.g., SE-30, OV-1) temperature-programmed from 30 to 245°C at 4°/min will be suitable.

5.2.3 Data System

5.2.3.1 GC/MS analysis is based on a combination of retention times and relative abundances of target ions. These qualifiers are stored on the hard disk of the GC/MS computer and are applied for identification of each chromatographic peak. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The 1990 (or more recent) release of the National Institute of Standards and Technology (NIST) Mass Spectral Library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel. The retention time qualifier must be determined to be within ±6 seconds of the library retention time of the compound. The acceptance level for relative abundance must be determined to be within ± 20 percent of the expected abundance. Any peak that fails any of the qualifying tests is flagged. All the data are manually examined by the analyst to determine the reason for the flag and whether the compound should be reported as detected. While this adds some subjective judgment to the analysis, computer generated identification problems can be clarified by an experienced operator. Manual inspection of the quantitative results is also performed to verify concentrations outside the expected range.

5.3 Reagents

5.3.1 Nitrogen gas: certified 99.995 percent.

5.3.2 Helium: certified 99.995 percent, with regulator.

5.3.3 Neat standards: chemical compounds to be used as standards.

5.3.4 Spectrograde methanol: distilled in glass.

5.3.5 Spectrograde acetone: distilled in glass.

NOTE: Individual chemicals to be used for standards, as well as isotopic standards, must have a manufacturer's determined purity of ≥98 percent or better. Purity should be checked by NMR or direct probe MS. Each chemical received by the laboratory is checked by injection of an aliquot into a GC, using a 50-m SE-30 WCOT glass capillary bonded (cross-linked) column and FID. The resulting chromatogram is examined for extraneous peaks. If such peaks are observed and amount to more than 2 percent of the standard peak, the standard is unacceptable.

5.4 Standards

5.4.1 The procedures for the preparation of calibration standards are described in more detail in Section 4. This section presents three procedures for generating known concentrations of targeted VOCs for direct injection into the GC/MS for calibration, or for deposition on Tenax® for calibration of the entire GC/MS analytical system. (Note that direct injection is allowed only for a continuing calibration standard.) They are: (1) use of flash vaporization technique for loading targeted VOC standards upon Tenax® tubes; (2) preparation of known concentrations utilizing static dilution bottles; and (3) use of permeation tube system for generating known concentrations of VOC standards on Tenax®.

5.4.2 The Contractor must provide all standards to be used with this contract. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

5.4.3 Neat standards must have a purity of 98 percent or greater. The weight may be used without correction to calculate the concentration of the stock solution.

5.4.4 Alternate methods of generating standards may be used if equivalency is established through an EPA audit procedure. Standards on Tenax® may be stored for no more than two weeks.

5.4.5 **Calibration Stock Standards**

Primary stock standards containing the target compounds at 10 mg/mL are used as primary stock standards.

5.4.6 **Working Standards**

5.4.6.1 **Instrument Performance Check Standard**

Prepare a standard solution of BFB in methanol at a concentration which will allow injection of 50 ng of BFB under the

optimized instrument parameters. Prepare fresh BFB solution every six months, or sooner, if the solution has degraded or evaporated.

5.4.6.2 Calibration Standards

Prepare five calibration standards at the concentration levels for each component as outlined in Table D/VT-4.

5.4.6.3 Internal Standard Spiking Mixture

Prepare an internal standard spiking mixture containing perfluorotoluene (PFT), 1,2-dichlorobenzene-d_4, and 1,4-difluorobenzene at a concentration that will allow deposition of 100 ng of each internal standard on the Tenax® cartridges. The amount of internal standard spiking mixture used for each analysis must be the same from run to run. Internal standards are spiked into the samples, blanks, and standard cartridges just prior to thermal desorption and subsequent GC/MS analysis.

5.4.6.4 Surrogate Compounds Spiking Mixture

Prepare the surrogate compounds spiking mixture containing benzene-d_6, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4 at a concentration that will allow deposition of 100 ng of each surrogate compound on the Tenax® cartridges. The amount of surrogate spiked into each cartridge must be the same for all analysis. The surrogate compounds are spiked onto the sample cartridges prior to field deployment.

5.4.6.5 Laboratory Control Sample (LCS) Spiking Mixture

Prepare the LCS spiking mixture containing all of the compounds listed below to allow deposition of 100 ng of each compound onto a Tenax® cartridge:

benzene	1,2-dichloropropane
carbon tetrachloride	tetrachloroethylene
1,2-dibromoethane	1,1,2-trichloroethane
1,2-dichlorobenzene	trichloroethylene
1,2-dichloroethane	

5.4.7 Storage of Standards in Cartridges

5.4.7.1 Secure the cartridge inside the Kimax® tube with a glass wool plug to avoid breakage during transport. For deuterated standards, label the top of the screw cap with the symbol, "D*".

NOTE: The star (*) indicates that deuterated standards have been loaded onto the cartridge. This symbol will also be added to the participant's code.

5.4.7.2 Store the cartridges in a sealed paint can in the freezer until they are ready to be sent to the field.

5.5 Instrument Operating Conditions

The typical instrument conditions for the analysis of VOCs on Tenax® sampling cartridge are outlined below using a NuTech Model 320, Tekmar Model 5000, or equivalent thermal desorption unit. The thermal desorption chamber and the six port Valco valve are maintained at 250°C during analysis. The detector is a quadrupole spectrometer capable of electron impact ionization. The nickel capillary trap on the inlet manifold should be cooled with liquid nitrogen.

5.5.1 Thermal Desorption Unit

Purge gas (prior to desorption)	-	Helium @ 1.2 mL/min
Desorption cycle		8 minutes
Initial desorption temperature	-	25°C
Final desorption temperature	-	150°C
Thermal desorption unit purge	-	10 mL/min

5.5.2 Gas Chromatography

Injection/Detector temperature	-	200°C
Initial column temperature	-	30°C
Initial hold time	-	0.1 minutes
Program (ramp rate)	-	4°C/min to 240°C
Final hold temperature	-	240°C
Final hold time	-	0.1 minutes
Maximum oven temperature	-	245°C
Carrier gas	-	Helium; velocity 20 cm^3/sec at 250°C
GC/MS interface	-	Direct coupling or glass jet
Sample injection to MS	-	Direct Probe
Column	-	Hewlett-Packard OV-1 glass capillary crosslinked methyl silicone (50 m x 0.3 mm, 0.17 μm film thickness) Scientific Glass Engineering SE-30 glass capillary crosslinked methyl silicone (50 m x 0.5 mm, 0.80 μm film thickness),or equivalent.

5.5.3 Mass Spectrometer

Mass range	-	35 to 300 amu
Scan time	-	1 sec/10 min over entire range
EI condition	-	70 eV
Mass scan and detector mode	-	Follow manufacturer instruction for select MSD and scan mode
Routine tuning	-	p-bromofluorobenzene (BFB)
Preamp sensitivity	-	10^7
Emission current	-	0.45
Electron multiplier voltage	-	1000 to 1500
Mass filter	-	10 amu/sec
Filter	-	x 100
Total Ion Current sensitivity	-	1
Resolution	-	Normal
Display	-	TIC
Response	-	Fast

5.6 Instrumental Analysis

5.6.1 Initial Start-Up

5.6.1.1 Prior to instrument calibration or sample analysis, helium purge flows (through the desorption unit) and carrier gas flow to the GC/MS are set at approximately 10 mL/min and 1-2 mL/min, respectively. If applicable, the injector sweep flow is set at 2-4 mL/min.

5.6.1.2 After the column and other system components are assembled, condition the column as specified by the manufacturer.

5.6.1.3 The MS and data system are set according to the manufacturer's instructions. The mass range should be from 35 to 300 amu, the scan time should be at least five scans per peak and not to exceed one second per scan. Table D/VT-2 outlines general operating conditions for the GC/MS system.

5.6.1.4 Once the entire GC/MS system has been set up, the user should prepare a detailed standard operating procedure describing the operation of the specific instrument being used.

5.6.1.5 Turn on the power to the mass-flow controllers and set line pressures for the following gases:

- Helium: 60 psig
- Compressed Air: 40 psig
- Nitrogen: 30 psig

5.6.1.6 Flow rates for the thermal desorption and GC system should be established according to instrument requirements.

5.6.1.7 Turn on the master power switch to the chromatograph, set manifold temperature to 105 ± 5°C, and set source ionization temperature to 260°C.

5.6.1.8 Turn on the power to the thermal desorption unit and set the temperatures for the valve, trap, and transfer line on the vernier dials on the control box of the thermal desorption unit according to manufacturer's specification. Typical values are:

- Valves 275°C
- Trap 250°C
- Line 210°C

5.6.2 Thermal Desorption of Tenax® Adsorbent Tubes

5.6.2.1 Initially, the thermal desorption unit is cold and the Tenax® cartridges are placed inside while flowing helium through them. This allows oxygen to be purged from the trap, reducing oxidative degradation of Tenax®. Then, during the thermal desorption cycle, helium gas continues to flow through the cartridge to purge the organic vapors on the Tenax® into the liquid nitrogen capillary trap.

5.6.2.2 After the desorption has been completed, the six-port valve is rotated and the temperature on the capillary loop is rapidly raised (greater than 100°C/min); the carrier gas then introduces the vapors onto the high resolution GC column. The bonded-phase fused silica capillary column is temperature-programmed from 40°C (5 min hold) to 240°C at 4°C/min and held at the upper limit until all target compounds elute.

5.6.2.3 The column is programmed to a temperature that will allow the elution of all of the organic compounds while the mass spectrometer is scanning. Data are recorded by the computer for subsequent processing. Quantitation is performed by the method of relative response factors, where the proportionate system responses for analyte and standard are determined prior to the analysis of the sample and this relative system response is used to determine the quantity of compound present on the sample cartridge.

5.6.2.4 The following outlines typical steps associated with sample handling immediately prior to thermal desorption using the NuTech device. They are presented as a guideline to follow when using this or similar equipment.

5.6.2.4.1 Remove the sealed paint can containing the desired Tenax® cartridge from the freezer.

NOTE: Use the freezer in the laboratory designated for cartridge storage ONLY for this purpose. Inadvertent storage of containers of solvent in this freezer will result in contamination of all cartridges stored in the freezer and will compromise the analysis, since organic solvents are frequently target compounds for quantitative analysis. Verify that the laboratory personnel are not involved in any process which exposes open containers of organic solvents, as organic solvent vapor will contaminate a Tenax® cartridge exposed to this atmosphere in only a few seconds, thus compromising the quantitative and/or qualitative assay.

5.6.2.4.2 Open the sealed lid of the paint can, using a flat-bladed screwdriver, beverage can opener, or other convenient tool for this purpose.

NOTE: The cartridge will be in a stainless steel tube with a Teflon-lined screw cap.

5.6.2.4.3 Remove a single Tenax® tube from the paint can. Seal the paint can and replace in the freezer, and release the Teflon cap of the desorption chamber.

5.6.2.4.4 Remove the cartridge from the shipping tube using forceps.

NOTE: DO NOT TOUCH THE CARTRIDGE WITH YOUR HANDS! Organic compounds present on the fingertips can be sufficient to compromise the analysis. If the cartridge is inadvertently touched, make careful note of the circumstances in both the instrument log and the project notebook.

5.6.2.4.5 Insert the cartridge immediately into the desorption chamber. Close the Teflon cap of the desorption chamber, and initiate the timing of the eight-minute desorption cycle. During this time period, helium is flowing through the desorption chamber.

5.6.2.5 At the end of the eight-minute desorption cycle, turn the desorption unit valve to the INJECT mode. The following steps are automatic on some commercially available instruments.

5.6.2.5.1 Start the GC temperature program;

5.6.2.5.2 Initiate heating of the nickel trap;

5.6.2.5.3 Turn on data acquisition system; and

5.6.2.5.4 Turn off the trap after it has heated to 250°C.

5.6.2.6 Turn the thermal desorption unit valve back to desorb and remove the Tenax® cartridge. At the end of the run, the GC will recycle and cool to 30°C, and the data acquisition will stop automatically after all compounds have eluted.

5.6.2.7 Repeat this procedure for each Tenax® cartridge.

5.6.2.8 Data from GC/MS runs are normally processed by the data system in an automated program which locates the compounds of interest in the data set, quantifies those compounds for which calibration data are available, and prints a report. A typical report will present the quantification parameters and result for those compounds identified and quantifiable. The report will typically list those compounds which were searched for in the sample, indicate which ones were not found, print the identifying characteristics and quantification results for those which were found, and present comments for the operator's benefit, such as the criteria which caused a peak to be rejected or the center scan for any search which failed. The information in the report can also be saved in a DS file for archival storage and DS transfer purposes.

5.6.2.9 The DS library should contain a file containing one entry for each compound of interest. For each entry, the library contains the compound name, its mass spectrum from the Mass Spectral Data Base, its absolute retention time, and its retention time relative to the internal standards, as determined from calibration standards. Response lists are compound specific DS files containing the quantitative calibration data for each of the target compounds.

5.6.2.10 The automated procedure attempts to locate chromatographic peaks corresponding to target compounds by a reverse library search using the following recommended criteria for scan window:

5.6.2.10.1 For internal standards:	±100 seconds from library scan number.
5.6.2.10.2 For single compounds:	±20 seconds from the calculated scan.
5.6.2.10.3 For isomer groups:	-20 and +20 seconds from the calculated seconds for the earliest and latest eluting members of the group, respectively.

5.6.2.10.4	Peak identification:	As determined by the laboratory.
5.6.2.10.5	Peak selection:	The scan list is partitioned in order of increasing distance from the center of the scan window, except for isomer groups.

5.6.2.11 The automated procedure begins by attempting to locate the two retention time markers (perfluorotoluene and 1,2-dichlorobenzene-d_4). If the early eluting standard, PFT, is not located, a warning message is printed and the procedure is terminated. If only the late eluting internal standard is not found, the procedure uses the scan number calculated from the library retention time for this standard as a default value.

NOTE: Alternatively, the operator may specify scan numbers for the internal standards and then initiate the remainder of the automated procedure. The procedure cycles through the compounds in the library list attempting to locate each compound in turn.

5.6.2.12 If one or more peaks are identified in the search for a target compound, the resulting scan list is partitioned to order the scans in increasing distance from the center of the search window. The mass spectra in the partitioned list are sequentially compared to the library entry for the target compound in order to the mass weighted purity, fit and rfit.

5.6.2.13 If the mass spectrum at the peak maximum passes either of the above tests, the procedure attempts to quantify the peak. If the target is a single compound, only the first peak to pass the qualitative criteria is processed further. If the target is an isomer group, all peaks detected by the search are processed through the qualitative filters and all that pass these filters are quantified. If no peaks are found by the search or pass through the qualitative filters, a "not detected" entry is placed in the report.

NOTE: The failure of a peak to satisfy these criteria does not necessarily prove the absence of the compound in the sample. Interfering compounds or low levels of the compound of interest may cause the test values to fall outside of the acceptance range. It is also possible to obtain acceptable values for fit/purity and rfit/purity, but have a questionable identification. If the absence of a particular compound is of crucial importance and the DS procedure fails to locate the compound, manual inspection of the data by a person skilled in the interpretation of GC/MS data is necessary for confirmation.

5.7 **Analytical Sequence**

The GC/MS analytical sequence for each 12-hour time period shall be as follows:

5.7.1 Instrument Performance Check (BFB);

5.7.2 Initial or continuing calibration;

5.7.3 Laboratory Method Blank;

5.7.4 LCS;

5.7.5 Field Blank;

5.7.6 ≤20 field samples; and

5.7.7 Performance Evaluation (PE) Sample (if available).

5.8 **Instrument Performance Check**

5.8.1 **Summary**

Instrument performance check and mass standardization of the MS system is performed according to manufacturer's instructions and relevant information from the user-prepared SOP. The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check standard.

5.8.2 **Frequency**

5.8.2.1 Prior to the analyses of any samples, blanks, or calibration standards, the Contractor must establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of p-bromofluorobenzene (BFB). The instrument conditions required for the acquisition of the BFB mass spectrum are given in Exhibit E.

5.8.2.2 Each GC/MS used for analysis must be checked for performance daily or once per each twelve hour period of operation, whichever is most frequent, to meet the technical acceptance criteria for BFB as outlined in Table D/VT-3. Also, whenever corrective action which could change or affect the instrument performance check for BFB (e.g., ion source cleaning or repair, column replacement, etc.), the instrument performance check must be verified immediately irrespective of the 12-hour or daily performance check requirement.

5.8.3 **Procedure**

5.8.3.1 Prepare a 25 ng/μL solution of BFB in methanol. Prepare fresh BFB solution every six months or sooner if the solution has degraded or evaporated.

NOTE: The 25 ng/μL concentration is used with a 2 μL injection volume. The laboratory may prepare a 50 ng/μL solution of BFB if a 1 μL injection volume is used.

5.8.3.2 Inject 50 ng BFB into the GC/MS.

5.8.3.3 Set time and parameters for the acquisition of the data and initiate data acquisition by following instructions in the operator's manual.

5.8.3.4 The instrument parameters (e.g., lens voltages, resolution) should be adjusted to give the relative ion abundances shown in Table D/VT-3 as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source or quadrapoles may require cleaning according to manufacturer's instructions. The Contractor must obtain the required relative ion abundances for BFB before proceeding with sample analysis.

5.8.4 **Technical Acceptance Criteria**

5.8.4.1 Prior to the analysis of any samples, blanks, or calibration standards, the Laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution.

5.8.4.2 The instrument performance check standard must be injected once at the beginning of each 12-hour period during which samples or standards are to be analyzed.

5.8.5 **Corrective Action**

5.8.5.1 If the BFB acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other actions to achieve the acceptance criteria.

5.8.5.2 BFB acceptance criteria MUST be met before any standards, performance evaluation (PE) samples, or required blanks are analyzed.

5.8.6 **Documentation**

Reporting requirements are listed in Exhibit B. Results of the GC/MS tuning are reported on Form IV-AAVT.

5.9 Initial Calibration

5.9.1 Summary

5.9.1.1 Prior to the analysis of samples and required blanks and after the instrument performance check standard criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations in an initial calibration sequence to determine instrument sensitivity and the linearity of GC/MS response for the target and surrogate compounds.

5.9.1.2 All sample results, for sample analyses performed in the same 12-hour sequence as the initial calibration, are quantified against the initial calibration standard that is the same concentration as the continuing calibration standard.

5.9.2 Frequency

5.9.2.1 Each GC/MS system must be calibrated upon award of the contract, whenever the Contractor takes corrective action which may change or affect the initial calibration criteria (e.g., ion source cleaning or repair, column replacement, etc.), or if the continuing calibration standard acceptance criteria have not been met.

5.9.2.2 If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. If time does not remain in the 12-hour period after meeting the acceptance criteria for the initial calibration, a new analytical sequence shall commence with the analysis of the instrument performance standard.

5.9.3 Procedure

5.9.3.1 Verify that the GC/MS system meets the instrument performance criteria in section 5.8.

5.9.3.2 The GC must be operated using temperature and flow rate parameters equivalent to those in section 5.5.

5.9.3.3 Prepare five calibration standards containing all the target compounds spiked on clean Tenax® tubes as outlined in Section 4 and at concentrations outlined in Table D/VT-4. To each of these tubes, add known concentrations of internal standards and surrogate standards at 100 ng/cartridge.

5.9.3.4 Analyze each tube according to section 5.6.

5.9.4 Calculations

NOTE: In the following calculations, the area response is that of the primary quantitation ion unless otherwise stated.

5.9.4.1 Relative Response Factor: Calculate the relative response factors (RRF) for each target and surrogate compound to the appropriate internal standard using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \qquad \text{EQ. D/VT-5}$$

where: RRF = relative response factor;
A_x = area of the primary ion for the compound to be measured;
A_{is} = area of the primary ion for the internal standard;
C_{is} = amount of internal standard, ng; and
C_x = amount of the compound in the calibration standard, ng.

5.9.4.2 Mean Relative Response Factor: Calculate the mean RRF ($\overline{RRF}$) for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n} \qquad \text{EQ. D/VT-6}$$

where: $\overline{RRF}$ = mean relative response factor;
x_i = RRF of the compound; and
n = number of values.

5.9.4.2.1 Percent Relative Standard Deviation (%RSD): Using the RRFs from the initial calibration, calculate the %RSD for all target and surrogate compounds using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100 \qquad \text{EQ. D/VT-7}$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(RRF_i - \overline{RRF})^2}{N - 1}} \qquad \text{EQ. D/VT-8}$$

where: SD_{RRF} = standard deviation of initial response factors (per compound);
RRF_i = relative response factor at a concentration level; and
$\overline{RRF}$ = mean of initial RRFs (per compound).

5.9.4.3 Relative Retention Times (RRT): Calculate the RRTs for each target and surrogate compound over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{IS}} \qquad \text{EQ. D/VT-9}$$

where: RT_c = retention time of the target or surrogate compound; and
RT_{IS} = retention time of the internal standard.

25.9.4.4 Mean of the Relative Retention Times ($\overline{RRT}$): Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target and surrogate compound over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT}{n} \qquad \text{EQ. D/VT-10}$$

where: $\overline{RRT}$ = mean relative retention time for the target or surrogate compound for each initial calibration standard; and
RRT = relative retention time for the target or surrogate compound at each calibration level.

5.9.4.5 Tabulate the area response (Y) of the primary ion (see Table D/VT-5) and the corresponding concentration for each compound and internal standard.

5.9.4.6 Mean Area Response ($\overline{Y}$) for Internal Standard: Calculate the mean area response ($\overline{Y}$) for each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n} \qquad \text{EQ. D/VT-11}$$

where: $\overline{Y}$ = mean area response; and
Y_i = area response for the primary quantitation ion for the internal standard for each initial calibration standard.

5.9.4.7 Percent Area Response Change (%ARC): Calculate the %ARC at each calibration level for each of the internal standards using the following equation:

$$\%ARC = \frac{A_x - \overline{Y}}{\overline{Y}} \times 100 \qquad \text{EQ. D/VT-12}$$

where: %ARC = percent area response change;
A_x = area response of the internal standard at a concentration level; and
$\overline{Y}$ = mean area response of the internal standard over the entire calibration range.

5.9.4.8 Mean of the Retention Times ($\overline{RT}$) For Internal Standard: Calculate the mean retention time ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n} \qquad \text{EQ. D/VT-13}$$

where: $\overline{RT}$ = mean retention time; and
RT = retention time for the internal standard for each initial calibration standard.

5.9.4.9 Internal Standard Retention Time Shift (RTS): Calculate the RTS between the RT of each internal standard at each concentration level and the $\overline{RT}$ for that internal standard over the entire calibration range using the following equation:

$$RTS = \overline{RT_i} - RT_x \qquad \text{EQ. D/VT-14}$$

where: $\overline{RT_i}$ = mean of the retention time for the internal standard in the initial calibration; and
RT_x = retention time of the internal standard at a concentration level.

5.9.4.10 Tabulate peak height or area responses against concentration for each compound and internal standard. Table D/VT-5 contains primary quantitation ions to be used for each target and surrogate compound and internal standard.

5.9.4.11 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition.

5.9.5 **Technical Acceptance Criteria**

5.9.5.1 All initial calibration standards must be analyzed at the concentration levels and frequency described in this section on a GC/MS system meeting the BFB instrument performance check criteria.

5.9.5.2 The %RSD for all target and surrogate compounds in the initial curve must be less than or equal to 30.0 percent. Up to two compounds may exceed the maximum %RSD criteria; the %RSD for those compounds, however, must not exceed 40.0 percent.

5.9.5.3 The RRT for each of the target and surrogate compounds at each calibration level must be within ±0.06 RRT units of the mean relative retention time ($\overline{RRT}$) for the compound.

5.9.5.4 The %ARC at each calibration level must be within ±40 percent of the mean area response ($\overline{Y}$) over the initial calibration range for each internal standard.

5.9.5.5 The retention time shift for each of the internal standards at each calibration level must be within ±20.0 seconds compared to the mean retention time ($\overline{RT}$) over the initial calibration range for each internal standard.

5.9.6 **Corrective Action**

5.9.6.1 If the retention time for any internal standard changes by more than 20 seconds from mean retention time from the entire calibration range, the chromatographic system must be inspected for malfunctions, and corrections made as required.

5.9.6.2 If the %RSD of the RRFs in the initial curve is not within ±30.0 percent, initial calibration standards are rerun until the %RSD is within the QC limits.

5.9.6.3 If the SICP area for any internal standard is not within 40 percent of the mean initial calibration area ($\overline{Y}$), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. Document in the SDG Narrative all inspection and corrective actions taken.

5.9.6.4 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the initial calibration technical acceptance criteria.

5.9.6.5 Initial calibration acceptance criteria MUST be met before any field samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when initial calibration criteria have not been met will require analysis of additional sample or blank cartridges (if available) at no additional cost to the Agency.

5.9.7 Documentation

Reporting requirements are listed in Exhibit B. Results of the initial calibration are reported on Form V-AAVT; Internal standard area and RT shall be tabulated on Form VII-AAVT.

5.10 Continuing Calibration

5.10.1 Summary

5.10.1.1 Prior to the analysis of samples and required blanks and after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a continuing calibration standard to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the method.

5.10.1.2 The continuing calibration standard, which is the mid level (CAL 3) standard, shall contain all the target compounds, surrogate compounds, and internal standards.

5.10.1.3 All sample results are quantified against the RRFs obtained from the continuing calibration standard.

5.10.2 Frequency

5.10.2.1 A check of the calibration curve must be performed once every 12 hours on a GC/MS system that has met the tuning criteria.

5.10.2.2 The continuing calibration sequence starts with the injection of the BFB. If the BFB analysis meets the ion abundance criteria for BFB, then a continuing calibration standard may be analyzed.

5.10.3 Procedure

Analyze the mid level standard (CAL 3) in a GC/MS system that has met the tuning and mass calibration criteria following the same procedure under Instrumental Analysis, section 5.6.

5.10.4 Calculations

NOTE: In the following calculations, the area response is that of the primary quantitation ion unless otherwise stated.

5.10.4.1 Relative Response Factor (RRF): Calculate a relative response factor (RRF) for each target compound and surrogate using the equation in section 5.9.4.1.

5.10.4.2 Percent Difference (%D): Calculate the percent difference in the RRF of the daily RRF (12-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target and surrogate compound using the following equation:

$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100 \qquad \text{EQ. D/VT-15}$$

where: %D = percent difference;
RRF_c = RRF of the compound in the continuing calibration standard; and
$\overline{RRF_i}$ = mean RRF of the compound in the most recent initial calibration.

5.10.5 **Technical Acceptance Criteria**

5.10.5.1 The continuing calibration standard must be analyzed at the concentration level and frequency described in this section on a GC/MS system meeting the BFB instrument performance check criteria.

5.10.5.2 The %D for each target and surrogate compound RRF in a continuing calibration sequence must be within ±30.0 percent of the initial calibration mean RRF in order to proceed with the analysis of samples and blanks. Up to two compounds may exceed the maximum %D criteria; the %D for those compounds, however, must not exceed 40.0 percent.

5.10.5.3 **Corrective Action**

5.10.5.4 If the retention time for any internal standard changes by more than 20 seconds from the latest continuing (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required.

5.10.5.5 If the continuing calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the continuing calibration technical acceptance criteria.

5.10.5.6 Continuing calibration acceptance criteria MUST be met before any field samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when the continuing calibration criteria have not been met will require analysis of additional sample or blank cartridges (if available) at no additional cost to the Agency.

5.10.6 **Documentation**

Reporting requirements are listed in Exhibit B. Results of the continuing calibration are reported on Form VI-AAVT; Internal standard area and RT shall be tabulated on Form VII-AAVT.

5.11 **Blank Analysis**

5.11.1 **Summary**

5.11.1.1 To monitor for possible laboratory contamination, laboratory method blanks are analyzed with each SDG at least once in a 12-hour analytical sequence. All steps in the analytical procedure are performed on the blank using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

5.11.1.2 A field blank is designed to detect potential sample contamination during the handling and shipping process of a field sample. The Tenax® cartridge used as a field blank must be associated with the actual sampling process; therefore, the blank cartridge is opened with the other cartridges, resealed, and carried through the same handling process as those used to sample ambient air.

5.11.1.3 A laboratory method blank (LMB) is an unused, certified Tenax® that has not left the laboratory. The blank cartridge is spiked with the same amount of surrogate compounds and internal standard and carried through the same analytical procedure as a field sample.

5.11.1.4 The same amount of internal standards that are added to each sample is added to each blank. All field samples must be analyzed with associated blanks.

5.11.2 **Frequency**

5.11.2.1 A field blank is analyzed once per sample delivery group. A field blank shall be analyzed along with each batch of ≤20 samples and shall be carried through the entire analytical procedure.

5.11.2.2 The LMB must be analyzed after the calibration standard(s) before any samples are analyzed.

5.11.2.3 Whenever an unusually concentrated sample is encountered, an LMB analysis shall be performed immediately after the sample analysis.

5.11.3 **Procedure**

5.11.3.1 Spike the blank cartridges with the same amount of internal

standards and surrogate compounds and using the same spiking technique as the field samples.

5.11.3.2 Analyze the blanks following the same procedure outlined under section 5.13, "Sample Analysis".

5.11.4 Calculations

The blanks are analyzed similar to a field sample and the equations in section 5.13.4 apply.

5.11.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the blank are evaluated against the corresponding internal standard area responses and RTs in the mid level (CAL 3) standard of the initial calibration.

5.11.5.1 All blanks must be analyzed at the frequency described in section 5.11.2 on a GC/MS system meeting the BFB instrument performance check and initial calibration or continuing calibration technical acceptance criteria.

5.11.5.2 The percent recovery of each of the surrogate compounds in the blanks must be between 80 and 120 percent.

5.11.5.3 The area response for each IS in the blank must be within ±40 percent of area response of the IS in the most recent valid calibration.

5.11.5.4 The retention time for each of the internal standards must be within ±20.0 seconds between the blank and the most recent valid calibration.

5.11.5.5 The blank must not contain any target analyte at a concentration greater than its CRQL and must not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte at its CRQL. The total level of analytes in the blank other than the surrogates and internal standards must not exceed 10 ng.

5.11.6 Corrective Action

5.11.6.1 If a Contractor's blanks do not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample storage and processing hardware

that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds.

5.11.6.2 All samples processed with a blank that is out of control (i.e., contaminated) shall be flagged with a "B".

5.11.7 **Documentation**

Reporting requirements are listed in Exhibit B. Blank results are reported on Form I-AAVT. and the associated samples are summarized on Form II-AAVT. Internal standard area and RT shall be tabulated on Form VII-AAVT.

5.12 Laboratory Control Samples (LCS)

5.12.1 Summary

The LCS is an internal laboratory quality control sample designed to assess the capability of the Contractor to perform the analytical method listed in this Exhibit. Section 5.4.6.5 lists the LCS compounds.

5.12.2 Frequency

The LCS must be analyzed and reported once per 12-hour analytical sequence, and concurrently with the samples in the SDG.

5.12.3 Procedure

5.12.3.1 Prepare a Tenax® cartridge containing all the LCS compounds at a concentration of 100 ng for each compound according to section 5.4.6.5, and the surrogates and internal standards at a concentration of 100 ng each using the same spiking technique as the samples and blanks.

5.12.3.2 Analyze the LCS following the same procedure described in section 5.13, Sample Analysis.

5.12.4 Calculations

5.12.4.1 Calculate individual compound recoveries of the LCS using the following equation:

$$LCS\ \%Recovery = \frac{Concentration_{reported}}{Concentration_{spiked}} \times 100 \qquad \text{EQ. D/VT-16}$$

5.12.4.2 Field sample calculations in section 5.13 also apply to the LCS in monitoring the area and retention time of each of the internal standards.

5.12.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the LCS are evaluated against the corresponding internal standard area responses and RTs in the mid level (CAL 3) standard of the initial calibration.

5.12.5.1 The LCS must be analyzed on a GC/MS system meeting the BFB, initial or continuing calibration, and blank technical acceptance criteria at the frequency described in section 5.12.2.

5.12.5.2 The percent recovery of each of the surrogate compounds in the LCS must be between 80 and 120 percent.

5.12.5.3 The percent recovery for each of the LCS compounds must be within the percent recovery limits of 60 to 140 percent.

5.12.5.4 The area response change between the LCS and the most recent valid calibration for each of the internal standards must be within ±40 percent.

5.12.5.5 The retention time shift between the LCS and the most recent valid calibration for each of the internal standards must be within ±20.0 seconds.

5.12.6 Corrective Action

5.12.6.1 If the technical acceptance criteria for the internal standards are not met, check calculations and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria.

5.12.6.2 The laboratory may not submit data from an SDG until all the LCS technical acceptance criteria are met. LCS contamination from laboratory sources or any LCS analyzed not meeting all the technical acceptance criteria will require analysis of additional LCS cartridges at no additional cost to the Agency.

5.12.6.3 LCS acceptance criteria MUST be met before any field

samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when the LCS technical acceptance criteria have not been met will require analysis of additional LCS cartridges at no additional cost to the Agency.

5.12.7 Documentation

Reporting requirements are listed in Exhibit B. Laboratory Control Sample analysis data are reported on Form III-AAVT. Internal standard area and RT shall be tabulated on Form VII-AAVT.

5.13 Sample Analysis

5.13.1 Summary

Prior to the analysis of samples, the appropriate GC/MS operating conditions are established, instrument performance check standard is analyzed, and the GC/MS system is calibrated. The contents of the sample cartridge is desorbed, preconcentrated, and subsequently analyzed by GC/MS under conditions in section 5.5. Guidelines for qualitative and quantitative analysis are discussed in sections 5.14 and 5.15.

5.13.2 Frequency

5.13.2.1 If time remains in the 12-hour period in which an initial calibration is performed, samples may be analyzed without analysis of a continuing calibration standard.

5.13.2.2 If time does not remain in the 12-hour period since the injection of the instrument performance check standard in which an initial calibration is performed, both the instrument performance check standard and the continuing calibration standard must be analyzed before sample analysis may begin.

5.13.3 Procedure

5.13.3.1 Analyze samples under the instrument conditions provided in section 5.5 and according to the GC/MS instrumental analysis procedure outlined in section 5.6.

5.13.3.2 Upon introduction of the cryofocused sample onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least five scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra.

5.13.3.3 Each analytical run must be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank analyzed is found to be free of interferences.

5.13.3.4 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative.

5.13.4 Calculations

5.13.4.1 The equation below is used for calculating concentrations.

$$X_a = \frac{A_x C_{is}}{A_{is} RRF} \qquad \text{EQ. D/VT-17}$$

where: X_a = compound concentration, ng/tube;
A_x = area of the characteristic ion for the compound to be measured;
A_{is} = area of the characteristic ion for the specific internal standard;
C_{is} = amount of the internal standard spiked into the cartridge, ng; and
RRF = relative response factor from the analysis of the continuing calibration standard or the mid level (CAL 3) standard of the initial calibration.

5.13.4.2 Surrogates percent recovery (%R): Calculate the surrogate percent recovery using the following equation:

$$\%R = \frac{Q_d}{Q_a} \times 100 \qquad \text{EQ. D/VT-18}$$

where: Q_d = quantity determined by analysis, ng; and
Q_a = quantity added to sample/blank, ng.

5.13.4.3 Percent Area Response Change (%ARC): Calculate the change in area response for each internal standard by comparing with the most recent valid calibration using the following equation:

$$\%ARC = \frac{A_c - A}{A_c} \times 100 \qquad \text{EQ. D/VT-19}$$

where: %ARC = percent area response change;
A_c = area response of the IS in the most recent valid calibration; and
A = area response of the IS in the sample.

5.13.4.4 Internal Standard Retention Time Shift (RTS): Calculate the shift in retention time between the RT in the sample and in the most recent valid calibration standard for each of the internal standards using the following equation:

$$RTS = RT_c - RT \qquad \text{EQ. D/VT-20}$$

where: RT_c = retention time of the IS in the most recent valid calibration; and
RT = retention time of the IS in the sample.

5.13.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the sample are evaluated against the corresponding internal standard area responses and RTs in the mid level (CAL 3) standard of the initial calibration.

5.13.5.1 The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in section 5.13.2.

5.13.5.2 The retention time for each internal standard must be within ±20.0 seconds of the retention time of the internal standard in the most recent valid calibration.

5.13.5.3 The %ARC for each of the internal standards must be within ±40 percent of the most recent valid calibration.

5.13.5.4 The sample must be desorbed and analyzed within the contract holding times.

5.13.5.5 The sample must have a laboratory method blank meeting the blank technical acceptance criteria of less than 10 ng of total VOCs per blank analysis.

5.13.5.6 The percent recovery of each of the surrogate compounds in the sample must be between 80 and 120 percent.

5.13.5.7 All target compound concentrations must not exceed the upper limit of the initial calibration range and no compound ion (excluding the compound peaks in the solvent front) may saturate the detector.

5.13.6 Corrective Action

5.13.6.1 If the on-column concentration of any compound in any sample exceeds the initial calibration range, flag results for the compounds that are out of range.

5.13.6.2 Internal standard responses and retention times must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 seconds (0.33 minutes) from the latest continuing (12-hour) calibration standard or mid level (CAL 3) standard if samples are analyzed in an initial calibration analytical sequence, the GC/MS system must be inspected for malfunctions, and corrections made as required.

5.13.6.3 If the SICP area for any internal standard changes by more than ±40 percent between the sample and the most recent valid calibration, the GC/MS system must be inspected for malfunction and corrections made as appropriate.

5.13.6.4 When target compounds are below contract required quantitation limits (CRQL), but the spectra meet the identification criteria, report the concentration with a "J." For example, if the CRQL is 5 ng and a concentration of 2 ng is calculated, report as "2J."

5.13.6.5 If the sample technical acceptance criteria for the surrogates and internal markers are not met, check calculations, surrogate and internal standard solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and internal standard technical acceptance criteria.

5.13.6.6 If the Contractor needs to analyze more than one (1) sample to have all the target compounds within the initial calibration range and to have all compound ions not saturating the detector (excluding the peaks in the solvent front), contact Sample Management Office (SMO). SMO will contact the Region for instructions.

5.13.6.7 Samples that do not meet technical acceptance criteria must be flagged.

5.13.7 Documentation

Reporting requirements are listed in Exhibit B. Sample analysis

data are reported on Form I-AAVT; Internal standard area and RT shall be tabulated on Form VII-AAVT.

5.14 Performance Evaluation Samples

5.14.1 Summary

5.14.1.1 The performance evaluation (PE) samples will assist the Agency in monitoring Contractor performance. The laboratory will not be informed as to which compounds are contained in the PE samples or the concentrations.

5.14.1.2 The laboratory will receive PE samples on Tenax® adsorbent tubes from the Agency. The samples will come with instructions concerning the desorption procedure required for the PE samples. Add internal standards and surrogate compounds to the PE sample, following procedures in Section 4.

5.14.1.3 In addition to complying with the PE sample technical acceptance criteria, the laboratory will be responsible for correctly identifying the quantifying the compounds included in the PE sample. The agency will notify the laboratory of unacceptable performance.

5.14.2 Frequency

The Laboratory must desorb, analyze, and report the results of the PE sample once per SDG, if available.

5.14.3 Procedure

Desorb and analyze the PE sample using the procedure described in section 5.13, Sample Analysis.

5.14.4 Calculations

Calculations for PE samples are the same as those for field samples. Use the equations in section 5.13 for determining technical acceptance criteria compliance.

5.14.5 Technical Acceptance Criteria

NOTE: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the PE sample are evaluated against the corresponding internal standard area responses and RTs in the mid level (CAL 3) standard of the initial calibration.

5.14.5.1 The PE sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in section 5.14.2

5.14.5.2 The PE sample must be analyzed with a method blank that met the blank technical acceptance criteria.

5.14.5.3 The percent recovery of each of the surrogate compounds in the sample must be between 80 and 120 percent.

5.14.5.4 The retention time for each internal standard in the PE sample analysis must be within ±20.0 seconds of the retention time of the internal standard in the most recent valid calibration.

5.14.5.5 The %ARC for each of the internal standards in the PE sample analysis must be within ±40 percent of the most recent valid calibration.

5.14.5.6 The results of analysis must identify the target compounds provided in the performance evaluation sample and must meet precision and accuracy criteria in comparison with the known results, as outlined in Section 6.

5.14.6 Corrective Action

5.14.6.1 If the PE sample technical acceptance criteria for the internal standard and surrogates are not met, check calculations, standard solutions and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria.

5.14.6.2 If duplicate cartridges of the PE samples are provided, the PE sample must be reanalyzed if technical acceptance criteria are not met. If after reanalysis, the SICP areas or the RTs for all internal standards are inside the contract limits, then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with SICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

5.14.6.3 If the reanalysis of the PE sample does not solve the problem, i.e., the SICP areas or internal standard RTs are outside the contract limits for both analyses, then submit the SICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the SDG Narrative all inspection and corrective actions taken.

5.14.6.4 If no other PE sample cartridges are provided for reanalysis, report the results after flagging the data as required. Put comments in the SDG narrative, if necessary.

5.14.6.5 In addition to complying with the PE sample technical acceptance criteria, the Contractor will be responsible for correctly identifying the compounds included in the PE sample. The Agency will notify the Contractor of unacceptable performance.

5.14.6.6 If the PE sample is provided with the SDG, PE sample technical acceptance criteria MUST be met before sample data are reported. Also, the Contractor must demonstrate acceptable performance for compound identification and quantification. If the Contractor fails to meet the PE sample technical acceptance criteria, the Agency may take, but is not limited to the following actions: reduction of the number of samples, suspension of sample shipment, a site visit, a full data audit, and/or require the laboratory to analyze a remedial PE sample, and/or a contract sanction, such as a Cure Notice.

5.14.7 Documentation

Reporting requirements are listed in Exhibit B. Sample analysis data are reported on Form I-AAVT; Internal standard area and RT shall be tabulated on Form VII-AAVT.

5.15 Qualitative Analysis

5.15.1 Target Compounds

5.15.1.1 The compounds listed in the Target Compound List (TCL) in Exhibit C and in Table D/VT-1 of this chapter shall be identified by an analyst competent in the interpretation of mass spectra (see Bidder Responsibility description) by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound.

5.15.1.2 Two criteria must be satisfied to verify the identifications:

- Elution of the sample component at the same GC retention time as the standard component; and
- Correspondence of the sample component and standard component mass spectra (primary and secondary ion identification).

5.15.1.3 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run in the same 12-hour time period as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using SICPs for ions unique to the component of

interest.

5.15.1.4 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes only if the Contractor's GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRTs.

5.15.1.5 The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10 percent (most abundant ion in the spectrum equals 100 percent) must be present in the sample spectrum;
- The relative intensities of such ions must agree within ±20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent);
- For each internal standard, determine that the area measured in the sample extract has not changed by greater than 40 percent from the area measured during the most recent continuing calibration check or by greater than 50 percent from the mean area measured during initial calibration. If either criterion is not met, remedial action must be taken to improve sensitivity; and
- Ions greater than 25 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL report the actual value followed by a "J", e.g., "3J."

5.15.1.6 If a compound cannot be verified by all of the above criteria, but in the technical judgement of the mass spectral interpretation specialist the identification is correct, then the Contractor shall report that identification and proceed with quantification.

5.15.2 Non-Target Compounds

5.15.2.1 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this

purpose, the 1990 (or more recent) release of the NIST Library, containing 50,000 spectra, shall be used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Up to 10 organic compounds of greatest apparent concentration not listed in Exhibit C shall be tentatively identified via a forward search of the NIST Library. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

5.15.2.2 **Guidelines For Making Tentative Identification:**

- Relative intensities of major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum;

- The relative intensities of the major ions should agree within ±20 percent. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent);

- Molecular ions present in reference spectrum should be present in sample spectrum;

- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds; and

- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

5.15.2.3 If in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

5.16 Quantitative Analysis

The quantitative analysis is performed by a combination of manual and computerized procedures: the computer identifies both primary and secondary characteristic ions in a full scan mode. At this point the operator intervenes to determine if the compound of interest has been

located correctly. If the compound identification is correct, the computer then performs the quantitative calculation using the method of relative response factors. Data are reported as ng or ng/tube (since the entire contents of the tube is introduced into the GC/MS system, and can be subsequently converted to ng/m³ if the volume of air sampled is known to the laboratory.

5.16.1 Target Compound Quantitation

5.16.1.1 Sample quantitation is performed by the data processing system for all desired ions of all target compounds. Target compounds are quantified according to the following equation:

$$X,\ ng/m^3 = \frac{A_x ng_{IS}}{A_s V_o RRF} \qquad \text{EQ. D/VT-21}$$

where: X = target compound air concentration, ng/m³,
A_x = area of ion of analyte,
ng_{IS} = mass of internal standard applied to tube, ng,
A_s = area of standard,
RRF = relative response factor (RRF), and
V_o = volume of air sampled (m³), STP.

If the volume of air sampled is not known to the laboratory, the above equation becomes:

$$X,\ ng = \frac{A_x ng_{IS}}{A_s RRF} \qquad \text{EQ. D/VT-22}$$

5.16.1.2 The computer must be able to print out peak number, m/e, scan number, time, relative retention time, area and amount.

5.16.1.3 Standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any standard changes by more than 30 seconds from the latest continuing (12 hour) calibration, the chromatographic system must be inspected for malfunctions, and corrections made as required. The SICP of the internal standards must be monitored and evaluated for each sample and blank. If the SICP area for any internal standard changes by more than 40 percent, the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of duplicate samples analyzed while the system was malfunctioning is necessary.

5.16.2 Non-Target Compound Quantitation

5.16.2.1 An estimated concentration for non-target components

tentatively identified shall be quantified by the standard method. The nearest internal standard free of interferences shall be used.

5.16.2.2 The formula for calculating concentrations is the same as in section 5.16.1.1. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the standard. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated (i.e., flagged "J"). This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

5.16.2.3 An estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns. This estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns.

6 PERFORMANCE REQUIREMENTS FOR DEMONSTRATING METHOD ACCEPTABILITY FOR THE ANALYSIS OF AMBIENT AIR

6.1 Summary

6.1.1 Although this method describes the GC/MS analysis of target compounds collected on Tenax®, collection on other adsorbents is acceptable if the performance criteria described in this section are met. Specifically, the analyst must demonstrate that collection on an alternate solid adsorbent and subsequent GC/MS analysis produce results meeting these general criteria:

- Minimum contract required quantitation limits (CRQL) listed in Table D/VT-1;
- Replicate precision within 30 percent RSD; and
- Audit accuracy of 30 percent* for target compound concentrations normally expected in ambient air.

* Exceptions are carbon tetrachloride and 1,1,1-trichloroethane, for which higher audit accuracies are reported in the USEPA TAMs study.

6.1.2 These criteria were established using historical data from the application of TO-10 methodology to samples from the Toxics Air Monitoring System (TAMs) and the Urban Air Toxics Monitoring Program (UATMP). The primary reason to base the acceptability of analysis method on performance is to allow systems currently being used for the analysis of VOCs in water to be used for VOCs in air. Solutions rather than compressed gas standards may be used for calibration. However, audit standards must be humidified gas standards, to most closely resemble the air matrix. Details for the determination of each of the criteria follow.

6.2 Minimum Contract Required Quantitation Limits (CRQL)

The minimum CRQL is defined by each laboratory by making seven replicate measurements of a concentration of the compound of interest near the expected detection limit, the standard deviation computed for the seven replicate concentrations, and this value multiplied by 3.14 (the Student's t value for 99 percent confidence for 7 values).

6.3 Replicate Precision

The measure of precision used for this program is the unsigned relative difference between replicate measurements of the same sample. There are several factors which may affect the quoted precision of the measurement. The nature of the compound of interest itself may have some effect on the precision such as the observation that styrene generally

shows slightly poorer precision than the bulk of nonpolar VOCs. The primary influence on precision is the concentration level of the compound of interest in the sample, i.e., the percent relative standard deviation for a set of replicate values degrades as the concentration approaches the detection limit. A conservative measure of precision was obtained from replicate analysis of Tenax® samples from the TAMs network. This is the source for the 30 percent figure of merit for overall replicate precision given above.

6.4 Audit Accuracy

Audit Bias is defined as the relative difference between the measurement result and the nominal concentration of the audit spiked compound, i.e., [(True - Found x 100]/True. Audit standards will be supplied to the participating laboratories, these audit standards analyzed, and the results judged against criteria based on historical data.

Table D/VT-1

Target Compound List for Volatiles Collected on Tenax® Tubes and Contract Required Quantitation Limits (CRQL)

Compound	CAS RN	CRQL, on column (ng)
Benzaldehyde	100-52-7	20
Benzene	71-43-2	9
Benzonitrile	100-47-0	5
Bromobenzene	108-86-1	45
Bromochloromethane	74-97-5	7
1-Bromo-3-chloropropane	109-70-6	5
Bromoethane	74-96-4	26
Bromotrichloromethane	75-62-7	6
n-Butylbenzene	104-51-8	3
Carbon Tetrachloride	56-23-5	7
Chlorobenzene	108-90-7	6
2-Chlorobutane	78-86-4	12
1-Chloro-2,3-epoxypropane	106-89-8	26
2-Chloroethoxyethene	110-75-8	27
Chloroform	67-66-3	9
1-Chloropropane	540-54-5	6
2-Chloropropane	75-29-6	11
3-Chloro-1-propene	107-05-1	6
m-Chlorotoluene	108-41-8	2
o-Chlorotoluene	95-49-8	2
p-Chlorotoluene	106-43-4	2
1,2-Dibromoethane	106-93-4	11
Dibromomethane	74-95-3	15
1,2-Dibromopropane	78-75-14	8
1,2-Dichlorobenzene	95-50-1	4
1,3-Dichlorobenzene	541-73-1	4
1,4-Dichlorobenzene	106-46-7	41
1,3-Dichlorobutane	1190-22-3	2
1,4-Dichlorobutane	110-56-5	27
2,3-Dichlorobutane	7581-97-7	17
cis-1,4-Dichloro-2-butene	764-41-0	7
3,4-Dichloro-1-butene	760-23-6	22
1,1-Dichloroethane	75-34-3	19
1,2-Dichloroethane	107-06-2	13
1,1-Dichloroethene	75-35-4	23
1,2-Dichloropropane	78-87-5	13
1,3-Dichloropropane	142-28-9	32
1,4-Dioxane	123-91-1	13
1-Ethenyl-4-chlorobenzene	1073-67-2	7

Table D/VT-1
(continued)

Target Compound List for Volatiles Collected on Tenax® Tubes
and Contract Required Quantitation Limits (CRQL)

Compound	CAS RN	CRQL, on column (ng)
Ethylbenzene	100-41-4	6
(1-Methylethyl)benzene	98-82-8	4
1-Methyl-4-(1-methylethyl)benzene	99-87-6	13
Pentachloroethane	76-01-7	6
1-Phenylethanone	98-86-2	10
Styrene	100-42-5	6
1,1,1,2-Tetrachloroethane	630-20-6	3
1,1,2,2-Tetrachloroethane	79-43-5	22
Tetrachloroethylene	127-18-4	9
Tetrahydrofuran	109-99-9	4
Toluene	108-88-3	7
Tribromomethane	75-25-2	28
1,1,1-Trichloroethane	71-55-6	6
1,1,2-Trichloroethane	79-06-5	7
Trichloroethylene	79-01-6	3
1,2,3-Trichloropropane	96-18-4	16
1,3,5-Trimethylbenzene	108-67-8	9
m- and p-Xylenes	1330-20-7	2
o-Xylene	95-47-6	2

Table D/VT-2

Typical Operating Conditions for a GC/MS System

Thermal Desorption Unit - NuTech Model 320, Tekman Model 5000, or equivalent

Purge gas (prior to desorption)	Helium @ 1.2 mL/min
Desorption cycle	8 minutes
Initial desorption temperature	25°C
Final desorption temperature	150°C
Thermal desorption unit purge	10 mL/min

Gas Chromatography

Injection/Detector temperature	200°C
Initial column temperature	30°C
Initial hold time	0.1 minutes
Program (ramp rate)	4°C/min to 240°C
Final hold temperature	240°C
Final hold time	0.1 minutes
Maximum oven temperature	245°C
Carrier gas	Helium; velocity 20 cm^3/sec at 250°C
GC/MS interface	Direct coupling or glass jet
Sample injection to MS	Direct Probe
Column	Hewlett-Packard OV-1 glass capillary crosslinked methyl silicone (50 m x 0.3 mm, 0.17 μm film thickness) Scientific Glass Engineering SE-30 glass capillary crosslinked methyl silicone (50 m x 0.5 mm, 0.80 μm film thickness),or equivalent.

Mass Spectrometer - Quadrupole Spectrometer, Electron Impact (EI)

Mass range	35 to 300 amu
Scan time	1 sec-10 min over entire range
EI condition	70 eV
Mass scan and detector mode	Follow manufacturer instruction for select mass selective detector (MS) and scan mode
Routine tuning	p-bromofluorobenzene (BFB)
Preamp sensitivity	10^{-7}
Emission current	-0.45
Electron multiplier voltage	1000 to 1500
Mass filter	10 amu/sec
Filter	x 100
Total Ion Current sensitivity	1
Resolution	Normal
Display	TIC
Response	Fast

Table D/VT-3

Required BFB Key Ions and Ion Abundance Criteria

M/e	Ion Abundance Criteria
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	base peak, 100 percent relative abundance
96	5.0 t0 9.0 percent of m/e 95
173	less than 2.0 percent of m/e 174
174	50.0 to 120.0 percent of m/e 95
175	4.0 to 9.0 percent of m/e 174
176	93.0 to 101.0 percent of m/e 174
177	5.0 to 9.0 percent of m/e 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

Table D/VT-4

Composition and Concentration (ng, on column injection) of Calibration Standards

Compound	Cal 1	Cal 2	Cal 3	Cal 4	Cal 5
benzaldehyde	5	50	100	150	200
benzene	5	50	100	150	200
benzonitrile	5	50	100	150	200
bromobenzene	20	75	150	200	250
bromochloromethane	5	50	100	150	200
1-bromo-3-chloropropane	5	50	100	150	200
bromoethane	20	50	150	200	250
bromotrichloromethane	5	50	100	150	200
butylbenzene	5	50	100	150	200
carbon tetrachloride	5	50	100	150	200
chlorobenzene	5	50	100	150	200
2-chlorobutane	5	50	100	150	250
1-chloro-2,3-epoxypropane	20	75	150	200	250
2-chloroethoxyethene	20	75	150	200	250
chloroform	5	50	100	150	200
1-chloropropane	5	50	100	150	200
2-chloropropane	5	50	100	150	200
3-chloro-1-propene	5	50	100	150	200
m-chlorotoluene	5	50	100	150	200
o-chlorotoluene	5	50	100	150	200
p-chlorotoluene	5	50	100	150	200
1,2-dibromoethane	5	50	100	150	200
dibromomethane	5	50	100	150	200
1,2-dibromopropane	20	75	150	200	250
1,2-dichlorobenzene	20	75	150	200	250
1,3-dichlorobenzene	20	75	150	200	250
1,4-dichlorobenzene	20	75	150	200	250
1,3-dichlorobutane	5	50	100	150	200
1,4-dichlorobutane	20	75	150	200	250
2,3-dichlorobutane	20	75	150	200	250
3,4-dichloro-1-butene	20	75	150	200	250
cis-1,4-dichloro-2-butane	5	50	100	150	200
1,1-dichloroethane	5	50	100	150	200
1,2-dichloroethane	5	50	100	150	200

Table D/VT-4
(continued)

Composition and Concentration (ng, on column injection) of Calibration Standards

Compound	Cal 1	Cal 2	Cal 3	Cal 4	Cal 5
1,1-dichloroethene	20	75	150	200	250
1,2-dichloropropane	5	50	100	150	200
1,3-dichloropropane	20	75	150	200	250
1,2-dimethylbenzene	5	50	100	150	200
1,4-dioxane	5	50	100	150	200
1-ethenyl-4-chlorobenzene	5	50	100	150	200
ethylbenzene	5	50	100	150	200
(1-methylethyl) benzene	5	50	100	150	200
1-methyl-4-(1-methylethyl)benzene	5	50	100	150	200
pentachloroethane	5	50	100	150	200
1-phenylethanone	5	50	100	150	200
styrene	5	50	100	150	200
1,1,1,2-tetrachloroethane	5	50	100	150	200
1,1,2,2-tetrachloroethane	20	75	150	200	250
tetrachloroethene	5	50	100	150	200
tetrachloromethane	5	50	100	150	200
tetrahydrofuran	5	50	100	150	200
tribromomethane	20	75	150	200	250
1,1,1-trichloroethane	5	50	100	150	200
1,1,2-trichloroethane	5	50	100	150	200
trichloroethene	5	50	100	150	200
1,2,3-trichloropropane	5	50	100	150	200
1,3,5-trimethylbenzene	5	50	100	150	200
toluene	5	50	100	150	200
xylene, m- and p-	5	50	100	150	200
xylene, o-	5	50	100	150	200

Table D/VT-5

Target Compound List for Volatiles Collected on Tenax® Cartridges with Characteristic Ions (Primary and Secondary)

Compound	Primary Ion	Secondary Ions
Benzene	78	77,50
Bromobenzene	156	158,77,51
Bromochloromethane	130	128,51
1-Bromo-3-chloropropane	158	156,77,76
Bromoethane	108	110,29,27
Bromotrichloromethane	163	161,82,47
n-Butylbenzene	91	134,92
Carbon Tetrachloride	117	119
Chlorobenzene	112	77,114
2-Chlorobutane	57	56,41
1-Chloro-2,3-epoxypropane	57	92
2-Chloroethoxyethene	63	43,44,106
Chloroform	83	85,47
1-Chloropropane	42	29,26
2-Chloropropane	43	27,63,78
3-Chloro-1-propene	41	39,76
m-Chlorotoluene	126	91,63
o-Chlorotoluene	126	91,39,63
p-Chlorotoluene	126	125,91,28
1,2-Dibromoethane	107	109,27
Dibromomethane	174	93,94,81
1,2-Dibromopropane	121	123,41
1,2-Dichlorobenzene	146	148,111
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,3-Dichlorobutane	55	27,90
2,3-Dichlorobutane	90	55,65
1,4-Dichlorobutane	55	27,41
cis-1,4-Dichloro-2-butene	75	53,89
3,4-Dichloro-1-butene	75	89,77
1,1-Dichloroethane	63	27,65
1,2-Dichloroethane	62	27,64
1,1-Dichloroethene	61	96,63
1,2-Dichloropropane	63	41,62

Table D/VT-5
(continued)

Target Compound List for Volatiles Collected on Tenax® Cartridges with Characteristic Ions (Primary and Secondary)

Compound	Primary Ion	Secondary Ions
1,3-Dichloropropane	76	41,27
1,4-Dioxane	88	58,29,31
Ethylbenzene	91	106
1-Ethenyl-4-chlorobenzene	138	103,140
(1-Methylethyl) benzene	105	120
1-Methyl-4-(1-methylethyl)benzene	119	134,91
Pentachloroethane	167	119,95
1-Phenylethanone	105	77,51,120
Styrene	104	78,103
1,1,1,2-Tetrachloroethane	131	117,119,96
1,1,2,2-Tetrachloroethane	83	85
Tetrahydrofuran	72	43,41,73
Tetrachloroethylene	164	129,131,166
Tribromomethane	171	175,93,81
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	97	83,61
Trichloroethylene	130	132,95
1,2,3-Trichloropropane	75	39,49,110
1,3,5-Trimethylbenzene	105	120
Toluene	91	92
Xylenes, o-, m-, and p-	91	106

Table D/VT-6

Typical Compounds Used in Response Factor (RF) Determination with Specific Mass Loading onto Spiked Cartridge

Compound	ng Loaded
Benzene	304
Chloroform	114
1,1,1-Trichloroethane	174
Carbon Tetrachloride	201
1,2-Dichloroethane	329
Trichloroethylene	403
1,1,2,2-Tetrachloroethane	409
Chlorobenzene	140
Tetrachloroethylene	323
Ethylbenzene	346
o-, m-, and p-Xylenes	344
Styrene	362
o-Dichlorobenzene	260
p-Dichlorobenzene	260

Figure D/VT-1

Common Designs of Adsorbent Cartriges

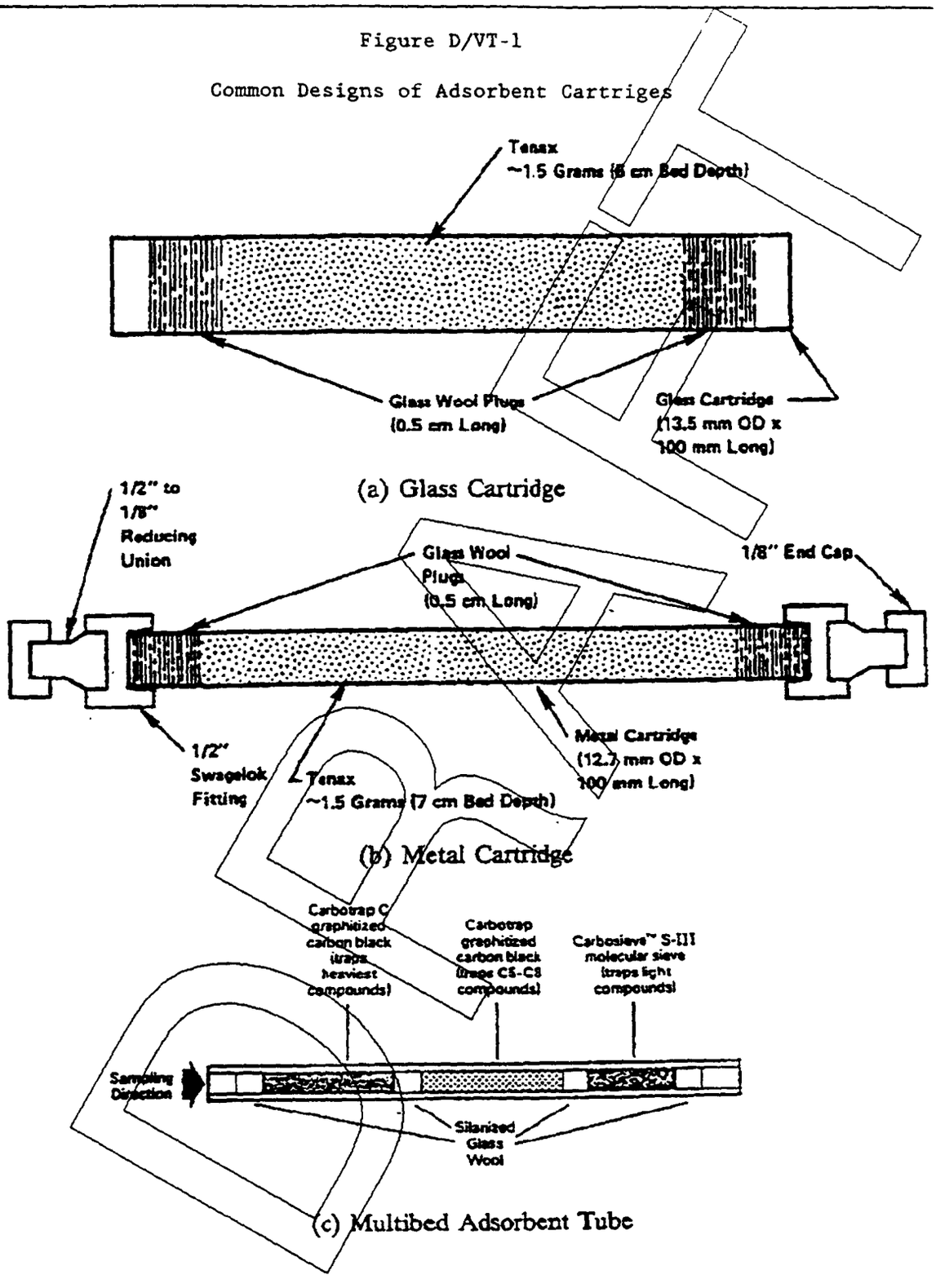

Figure D/VT-2

Typical Desorption GC/MS Configuration

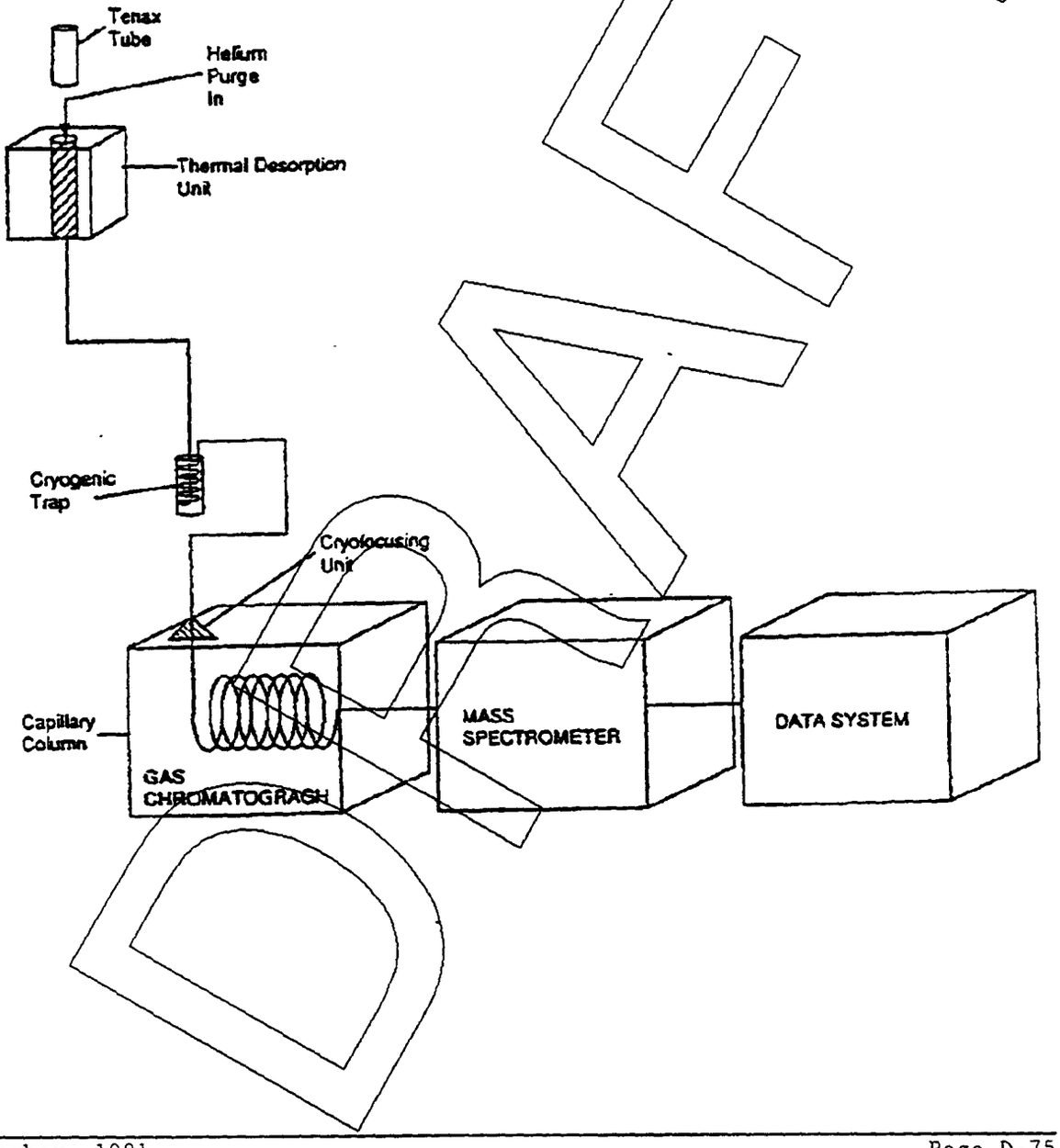

Figure D/VT-3

Flash Evaporation Unit

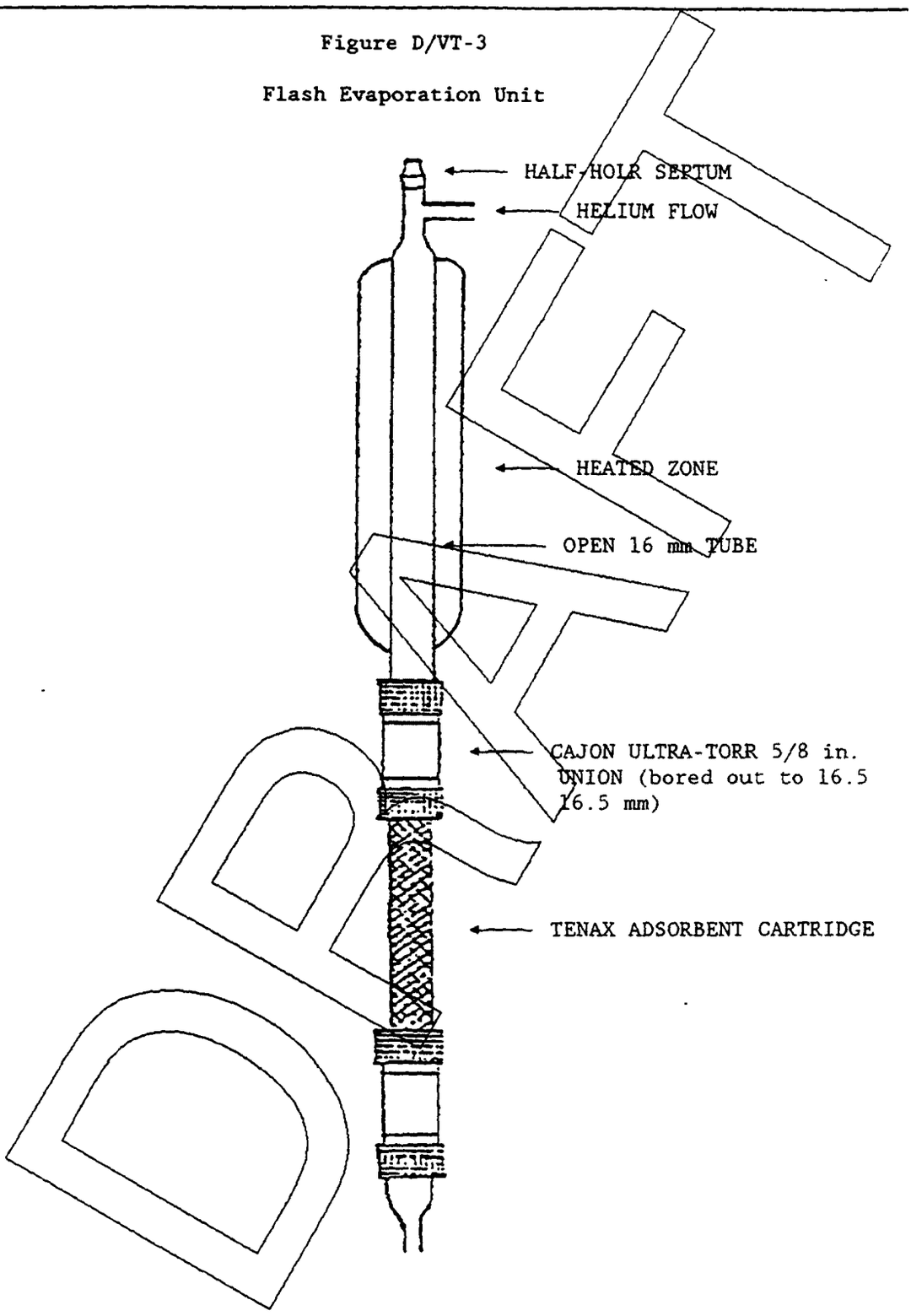

Figure D/VT-4

Permeation Tube System for
Generating Standard Gas Atmospheres

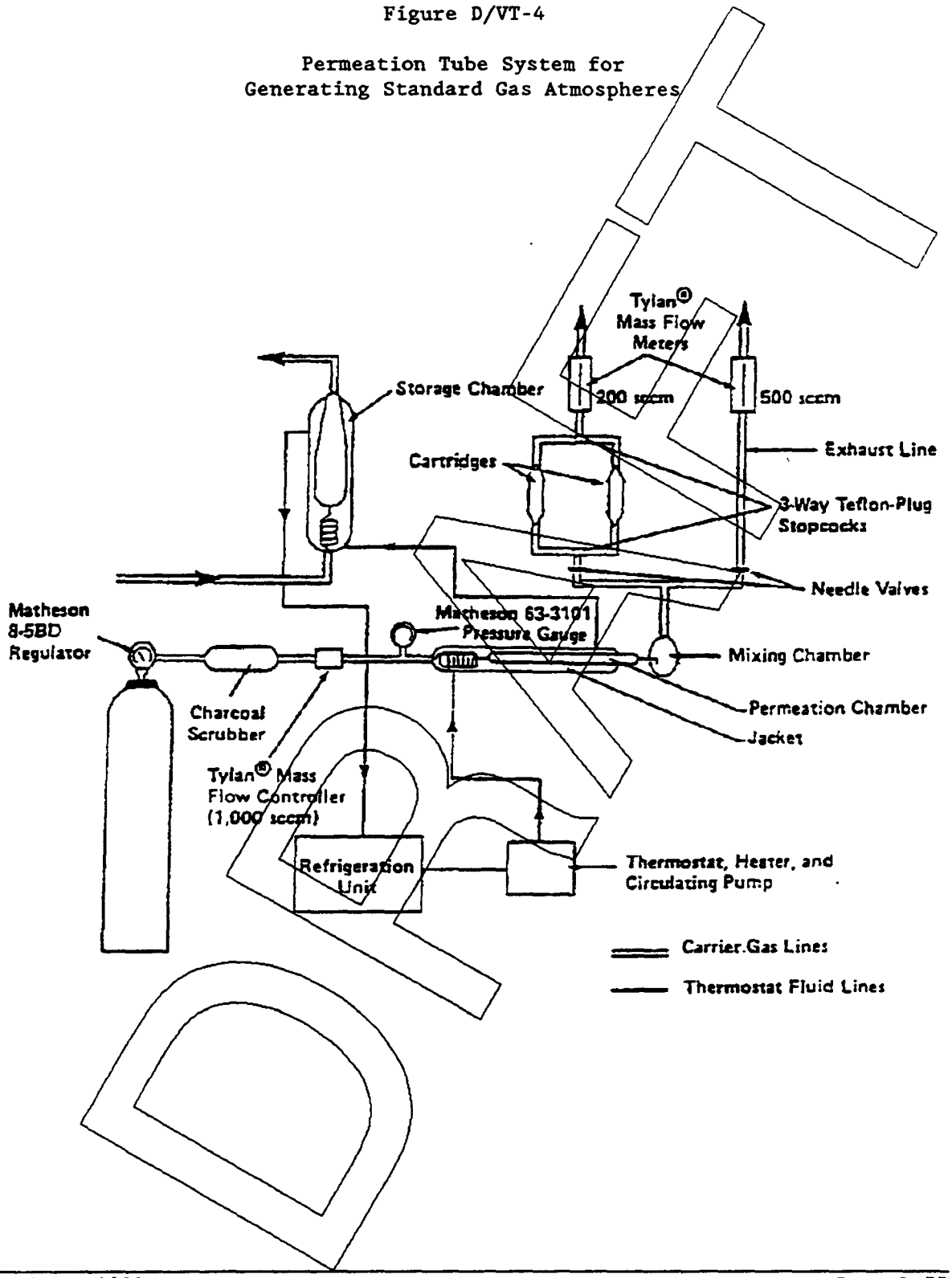

Figure D/VT-5

Tenax Cleanup Schematic Diagram

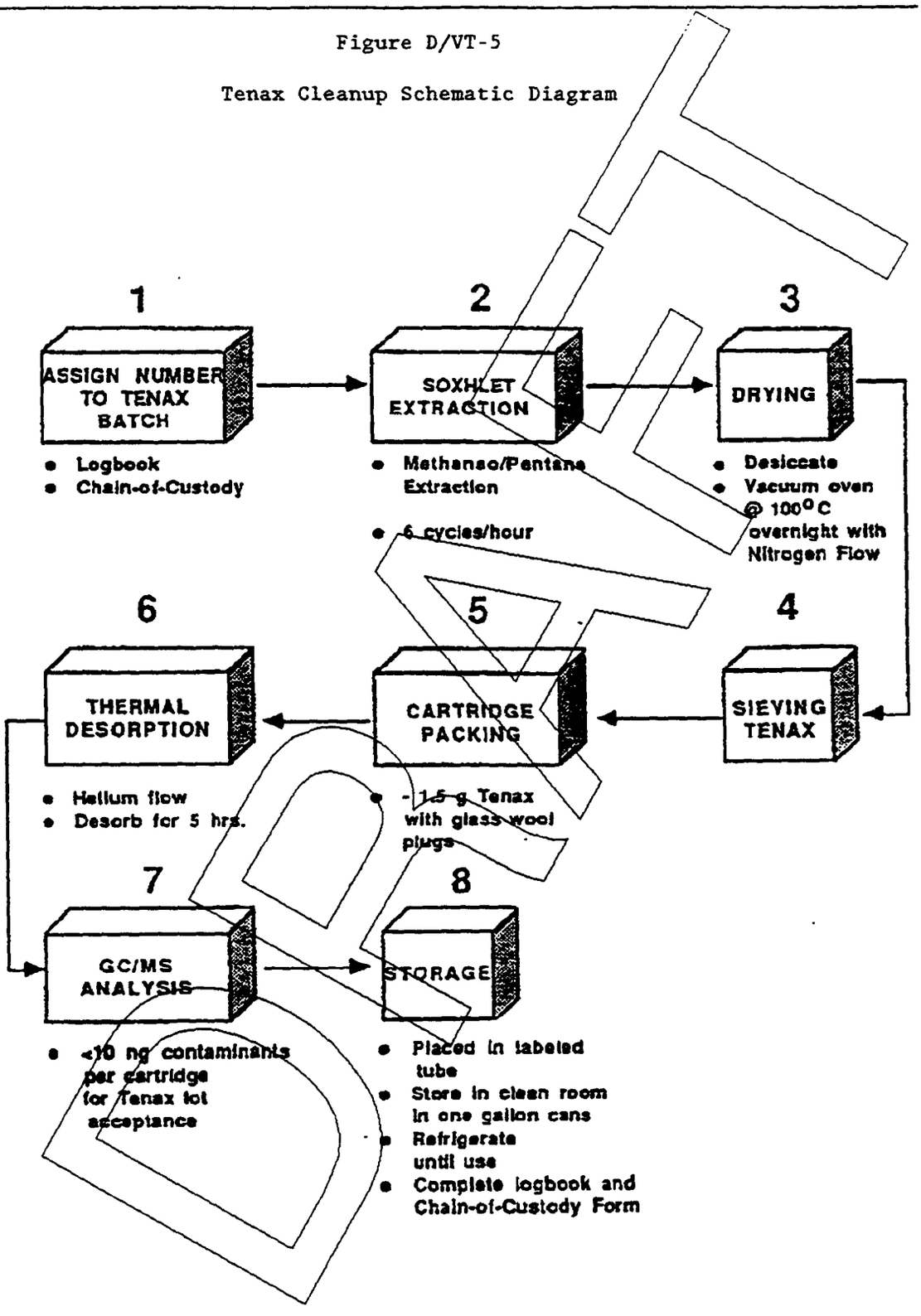

Figure D/VC-6

Tenax Cleanup Worksheet

Tenax Batch No.:_____ Projected Use:_____

Virgin_____ Recycled_____ (check one) Recycled Source_____

No. of cartridges:_____

EXTRACTION

No. of Soxhlet units (circle one) 1 2 3 4 5

Methanol extraction:
Date:
Hours:
Siphon rate:

Pentane extraction:
Date:
Hours:
Siphon rate:

DRYING

Nitrogen chamber:
Date:
Hours:
Approximate Flowrate:

Vacuum oven:
Date:
Hours:
Pump trap:
Cooldown (hours):
N_2 vent through Act. C:

SIEVING/PACKING

Sieve (40/60) Date:

Packing Date:

CLEANUP

Teflon septum Date:

Teflon liner Date:

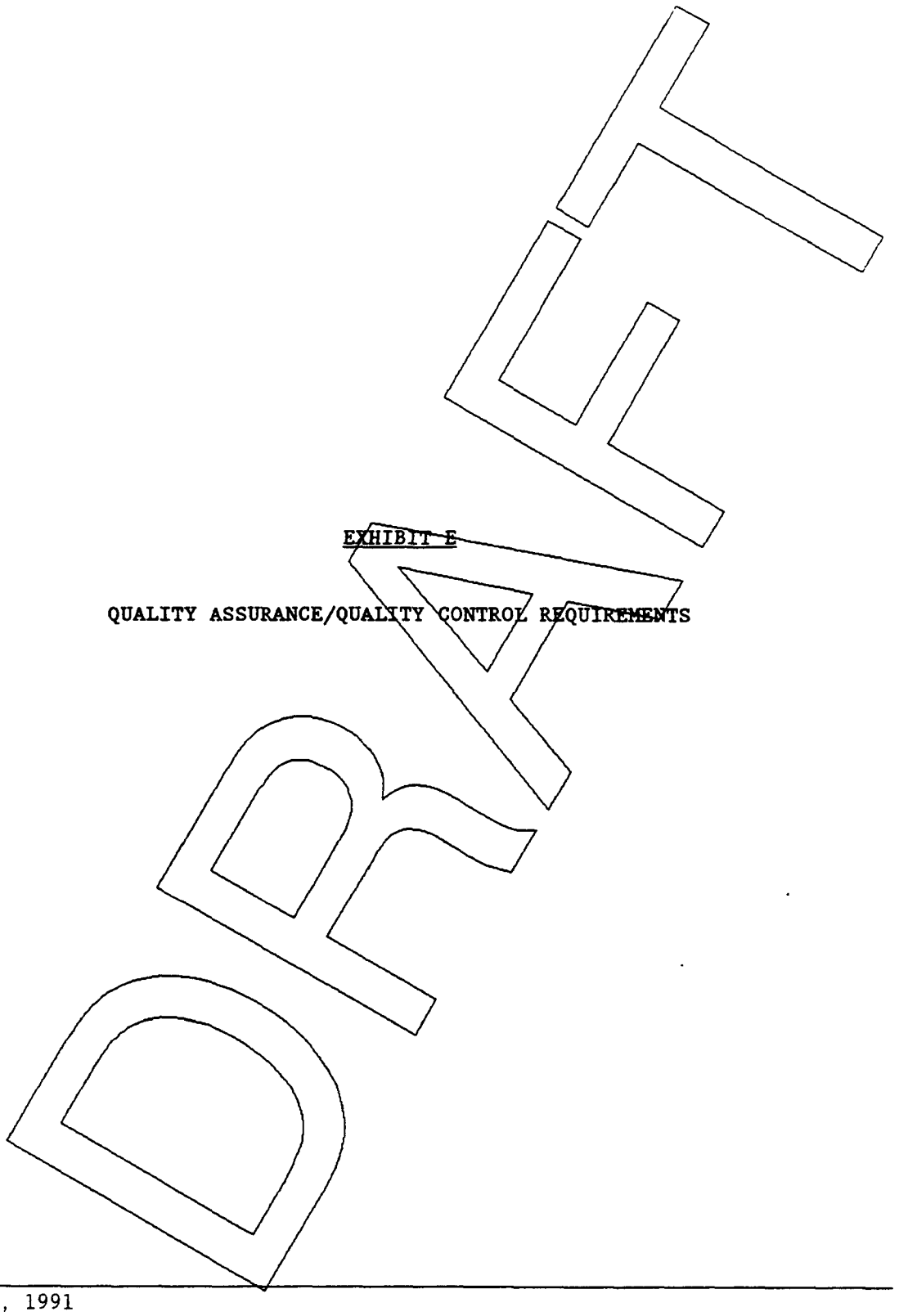

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

December, 1991

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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SECTION 1

INTRODUCTION

1.1 Quality assurance (QA) and quality control (QC) are integral parts of EPA's Contract Laboratory Program (CLP). The CLP QA program consists of management review and oversight at the planning, implementation, and completion stages of environmental data generation activities, to ensure that data provided are of the quality required. The CLP QC program includes those activities required as part of data generation to ensure that the data are of known and documented quality.

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

1.3 The purpose of this Exhibit is to describe 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 QA/QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These operations are designed to facilitate laboratory comparison by providing the EPA with comparable data from all Contractors. These requirements do not release the laboratory from maintaining its own QC checks on method and instrument performance.

1.4 Appropriate use of data generated under the great range of analytical conditions encountered in ambient air analyses requires reliance on the QC procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories participating in the CLP. However, the validation of these methods does not guarantee that they perform equally well for all samples collected under actual field conditions. Inaccuracies can result from causes such as sampling artifacts, equipment malfunctions, and human error. Therefore, the QC component of each method is indispensable.

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

1.6 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, NEIC, and EMSL/LV. Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Performance evaluation samples provide an external QA reference for the program. A laboratory 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 contract laboratories through direct communications with the Administrative Project Officers (APOs) and Technical Project Officers (TPOs).

1.7 This Exhibit is not a guide to constructing QA project plans, QC systems, or a QA organization. It is, however, an explanation of the QC and QA requirements of the CLP. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a QA Plan and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides the Agency with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

1.8 To ensure that the product delivered by the Contractor meets the requirements of the contract and to improve interlaboratory data comparison, the Agency requires the following from the Contractor.

1.8.1 Development and implementation of a QA program, and documentation of the key elements of that QA program through a written QA Plan, as described in Section 2 of this Exhibit.

1.8.2 Preparation of and adherence to written Standard Operating Procedures (SOPs) as described in Section 5 of this Exhibit.

1.8.3 Adherence to the analytical methods and associated QC requirements specified in the contract.

1.8.4 Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical houses.

1.8.5 Participation in the analysis of laboratory performance evaluation (PE) samples, including adherence to corrective action procedures.

1.8.6 Participation in on-site laboratory evaluations, including adherence to corrective action procedures.

1.8.7 Submission of all raw data and pertinent documentation for Regional review.

1.8.8 Submission, upon request, of GC/MS tapes and applicable documentation for tape audits.

1.8.9 Submission for Agency review of all original documentation generated during sample analyses.

SECTION 2

QUALITY ASSURANCE PLANS

2.1 The Contractor shall establish a QA program with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

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

2.2.1 Maintain data integrity, validity, and usability.

2.2.2 Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility .

2.2.3 Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable.

2.2.4 Document all aspects of the measurement process in order to provide data that are technically sound and legally defensible.

2.3 The QAP must present, in specific terms, the policies, organization, objectives, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during On-Site Laboratory evaluation and upon written request by the Administrative Project Officer. Additional information relevant to the preparation of a QAP can be found in EPA and ASTM publications[2,4].

2.4 **ELEMENTS OF A QUALITY ASSURANCE PLAN**

2.4.1 The following key elements of the Contractor's quality assurance program shall be addressed in the QAP:

2.4.1.1 **Contractor QA Policy and Objectives**

2.4.1.2 **Organization and Personnel**

- QA Management;
- Organization;
- Assignment of QC and QA responsibilities; and

- Reporting relationships.

• Personnel;

- Staff resumes;
- Education and experience requirements pertinent to this Contract; and
- Training progress.

• Facilities and Equipment;

- Instrumentation and backup alternatives, and
- Maintenance activities and schedules.

• Document Control;

- Laboratory notebook policy;
- Sample and data tracking/custody procedures and documentation requirements;
- Logbook maintenance and archiving procedures;
- Case file organization, preparation, and review procedures; and
- Procedures for preparation, approval, review, revision, and distribution of SOPs.

• Analytical Methodology;

- Calibration procedures and frequency;
- Sample handling and storage procedures;
- Sample preparation procedures;
- Sample analysis procedures; and
- Standards preparation procedures.

• Data Generation;

- Data collection procedures;
- Data reduction procedures;

- Data review procedures;
- Data reporting and authorization procedures; and
- Data management procedures.

• Quality Control Program; and

- Solvent, reagent, and adsorbent check analysis;
- Reference material analysis;
- Internal QC checks; and
- Corrective action and determination of QC limit procedures.

• Quality Assurance Program Assessment.

- Data audits;
- Systems audits;
- Performance audits;
- Corrective action procedures; and
- QA reporting procedures.

SECTION 3

STANDARD OPERATING PROCEDURES

3.1 In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document that 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.

3.2 SOPs prepared by the Contractor must be functional, i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs, as presented to the Agency, must reflect activities as they are currently performed in the laboratory. In addition, all SOPs must:

3.2.1 Be consistent with current EPA regulations, guidelines, and the CLP contract's requirements;

3.2.2 Be consistent with instrument manufacturer's specific instruction manuals;

3.2.3 Be available to the EPA 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 evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs;

3.2.4 Provide for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol;

3.2.5 Describe the mechanism for demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results;

3.2.6 Describe the corrective measures and feedback mechanism used when analytical results do not meet protocol requirements;

3.2.7 Be reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made;

3.2.8 Be archived for future reference in usability or evidentiary situations;

3.2.9 Be available at specific work stations as appropriate; and

3.2.10 Be subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

3.3 SOP SPECIFICATIONS AND FORMAT

3.3.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA sa the basis for laboratory evidence audits.

3.3.2 The format for SOPs may vary depending upon the kind of activity for which they are prepared. However, at a minimum, the following sections must be included.

3.3.2.1 Title page.

3.3.2.2 Scope and application.

3.3.2.3 Definitions.

3.3.2.4 Procedures.

3.3.2.5 QC acceptance criteria.

3.3.2.6 Corrective Action Procedures, including procedures for secondary review of information being generated.

3.3.2.7 Documentation Description and example forms.

3.3.2.8 Miscellaneous notes and precautions.

3.3.2.9 References.

3.4 REQUIRED SOPS

3.4.1 Evidentiary SOPs

The Contractor shall develop and use adequate written SOPs to ensure sample and data accountability. Evidentiary SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.4.1.1 Sample receipt and logging

3.4.1.1.1 The Contractor shall have written SOPs for receiving and logging in the samples. The procedures shall include, documentation of the following information:

- Presence or absence of EPA chain-of-custody forms;
- Presence or absence of airbills or airbill stickers;
- Presence or absence of EPA Traffic Reports or SAS packing lists;
- Presence or absence of custody seals on shipping and/or sample containers and their condition;
- Custody seal numbers, when present;
- Presence or absence of sample tags;
- Sample tag ID numbers;
- Condition of the shipping container;
- Condition of the sample container;
- Verification of agreement or nonagreement of information on receiving documents and sample containers;
- Resolution of problems or discrepancies with SMO; and
- The definition of any terms used to describe sample condition upon receipt.

3.4.1.1.2 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.4.1.2 Sample identification

3.4.1.2.1 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.4.1.2.2 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.4.1.2.3 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

3.4.1.3 Sample security

The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access to secure storage areas.

3.4.1.4 Internal chain-of-custody of samples and data.

The Contractor shall have written SOPs for the chain-of-custody consisting of sample identification, chain-of-custody procedures, sample receiving procedures, and sample tracking procedures. For more information concerning the chain-of-custody procedures see Section 4 of this Exhibit.

3.4.1.5 Internal tracking of samples and data.

The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

- A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses;
- A description of the documentation used to record instrument calibration and other QA/QC activities; and
- Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.4.1.6 Laboratory document and information control

3.4.2 Analytical SOPs

The Contractor shall develop and use adequate written SOPs to ensure that all data generated for the CLP are of known, documented, and acceptable quality. Analytical SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.4.2.1 The Contractor shall have written SOPs for preventing sample contamination, during sample preparation, cleaning of glassware, storage, and analysis.

3.4.2.2 The Contractor shall have SOPs to ensure traceability of standards used in sample analysis QA/QC.

3.4.3 **Quality Management SOPs**

3.4.3.1 The Contractor shall have written SOPs for technical and managerial review of laboratory operation and data package preparation, laboratory data review/laboratory self inspection system. The procedures shall include but not be limited to documenting the following information:

3.4.3.1.1 Data flow and chain-of-command for data review;

3.4.3.1.2 Procedures for measuring precision and accuracy.

3.4.3.1.3 Evaluation of parameters for identifying systematic errors.

3.4.3.1.4 Procedures to assure that hardcopy deliverables are complete and compliant with the requirements in Exhibit B.

3.4.3.1.5 Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.).

3.4.3.1.6 Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).

3.4.3.1.7 Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).

3.4.3.1.8 Documentation of audit reports, (internal and external), response, corrective action, etc.

3.4.3.2 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure. For more information

concerning document control and case file preparation, see Section 5 of this Exhibit.

3.4.3.3 The Contractor shall have written SOPs for sample analysis, management and handling, and reporting of data. The procedures shall include but not be limited to documenting the following information:

3.4.3.3.1 Procedures for controlling and estimating data entry errors.

3.4.3.3.2 Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

3.4.3.3.3 Life cycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

3.4.3.3.4 Database security, backup and archival procedures including recovery from system failures.

3.4.3.3.5 System maintenance procedures and response time.

3.4.3.3.6 Individual(s) responsible for system operation, maintenance, data integrity and security.

3.4.3.3.7 Specifications for staff training procedures.

3.4.3.4 The Contractor shall have written SOPs for laboratory safety.

3.5 HANDLING OF CONFIDENTIAL INFORMATION

3.5.1 A Contractor conducting work under this contract may receive EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.5.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.5.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each

working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

3.6 SOPS DELIVERY REQUIREMENTS

Within forty-five (45) days of contract receipt, a complete set of SOPs relevant to this contract shall be sent to the TPO, SMO and EMSL/LV. Also, during the term of performance of the contract, copies of SOPs which have been amended or new SOPs which have been written shall be sent to the TPO, EMSL/LV (quality assurance SOPs) and NEIC (evidentiary SOPs).

SECTION 4

CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

4.1 SAMPLE IDENTIFICATION

4.1.1 To ensure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

4.1.2 Each sample and sample preparation container shall be labeled with the EPA number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA number.

4.2 CHAIN-OF-CUSTODY PROCEDURES

Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented. A sample is under custody if the following applies:

4.2.1 It is in your possession, or

4.2.2 It is in your view after being in your possession, or

4.2.3 It was in your possession and you locked it up, or

4.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

4.3 SAMPLE RECEIVING PROCEDURES

4.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

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

4.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment.

4.3.5.1 Airbills or airbill stickers.

4.3.5.2 Custody seals.

4.3.5.3 EPA custody records.

4.3.5.4 EPA traffic reports or SAS packing lists.

4.3.5.5 Sample tags.

4.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

4.3.7 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

4.3.8 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

4.3.9 The following information shall be recorded on appropriate Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected.

4.3.9.1 Condition of the shipping container.

4.3.9.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

4.3.9.3 Custody seal numbers, when present.

4.3.9.4 Condition of the sample bottles.

4.3.9.5 Presence or absence of airbills or airbill stickers.

4.3.9.6 Airbill or airbill sticker numbers.

4.3.9.7 Presence or absence of EPA custody records.

4.3.9.8 Presence or absence of EPA traffic reports or SAS packing lists.

4.3.9.9 Presence or absence of sample tags.

4.3.9.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

4.3.9.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

4.3.9.12 Problems or discrepancies.

4.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 5

DOCUMENT CONTROL

The goal of the laboratory document control program is to assure that all documents for a specified Sample Delivery Group (SDG) will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

5.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

5.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

5.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

5.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

5.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

5.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the

laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

5.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be crossed out.

5.2 CONSISTENCY OF DOCUMENTATION

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

5.2.2 All copies of laboratory documents shall be complete and legible.

5.2.3 Original documents which include information relating to more than one SDG shall be filled in the CSF of the lowest SDG number. The copy(s) shall be placed in the other CSF(s) and the Contractor shall record the following information on the copy(ies) in red ink:

"COPY

ORIGINAL IS FILED IN CSF _____"

The Contractor shall sign and date this addition to the copy(ies).

5.2.4 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, lab bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

5.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURE

5.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

5.3.2 All documents relevant to each sample delivery group, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

5.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 is an example of a document inventory.

Figure E-1

Example

DOCUMENT INVENTORY

Document Control #*	Document Type	# Pages
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	30
232-2-0005	SMO Organics Traffic Reports	10
232-2-0006	Analysis Data Sheets	41
232-2-0007	Analysts' Organics Notebook Pages	14
232-2-0008	GC/MS and GC Instrument Logbook Pages	12
etc.	etc.	etc.

*This number is to be recorded on each set of documents.

5.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

5.5 SHIPPING DATA PACKAGES AND CSF

5.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

5.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

5.5.3 A copy of the transmittal letter for the CSF will be sent to the NEIC and the SMO.

5.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one (1) original FORM AADC-1 for each shipping container.

5.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

5.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

5.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 6

ANALYTICAL STANDARDS REQUIREMENTS

The U.S. Environmental Protection Agency will not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories will be required to prepare from neat materials, from cylinders of compressed gases traceable to NIST Standard Reference Materials or NIST/EPA approved certified reference material, or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

6.1 PREPARATION OF CHEMICAL STANDARDS FROM THE NEAT HIGH PURITY BULK MATERIAL

6.1.1 A laboratory may prepare their chemical standards from neat materials. Commercial sources for neat chemical standards pertaining to analytes listed on the TCL are given in Appendix C of the "Quality Assurance Materials Bank: Analytical Reference Standards," Seventh Edition, January 1988. Laboratories should obtain the highest purity possible when purchasing neat chemical standards; standards purchased at less than 98% purity must be documented as to why a higher purity could not be obtained.

6.1.2 Neat chemical standards must be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of neat chemicals is essential in order to safeguard them from decomposition.

6.1.3 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 contract laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use either differential scanning calorimetry, gas chromatography with flame ionization detection, high performance liquid chromatography, infrared spectrometry, or other 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:

$$\text{Wt. of impure compound} = \frac{\text{Wt. of pure compound}}{\left(\frac{\text{percent purity}}{100}\right)} \qquad \text{Eq. E-1}$$

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

6.1.4 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 contract laboratory's responsibility to have analytical documentation confirming that all compounds used in the preparation of solution standards are correctly identified. Identification confirmation, when performed, should use GC/MS analysis on at least two different analytical columns, or other appropriate techniques.

6.1.5 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 must verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights. All weighing should 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 should be compatible with the protocol in which the standard is to be used; the solute should be soluble, stable, and nonreactive with the solvent. In the case of a multicomponent solution, the components must not react with each other.

6.1.6 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 when not in use. All solution standards are to be clearly labeled as to the identity of the compound or compounds, concentration, date prepared, solvent, and initials of the preparer.

6.2 PURCHASE OF CHEMICAL STANDARDS IN SOLUTION

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

6.2.1.1 Contract laboratories must maintain the following documentation to verify the integrity of the standard solutions they purchase:

- Mass spectral identification confirmation of the neat material;
- Purity confirmation of the neat material; and
- Chromatographic and quantitative documentation that the solution standard was QC checked according to the following section.

6.2.1.2 The Contractor must 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 6.2.1.3 and 6.2.1.4 below). 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 part 6.3.1.3 which follows. If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test. The standard is certified to be within 10 percent of the target concentration.

6.2.1.3 If the procedure above is used, the supplier must document that the following have been achieved.

- Two solutions of identical concentration must be prepared independently from neat materials. An aliquot of the first solution must be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration ten percent 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 percent less that the target standard. This is called the "low standard";

- Six replicate analyses of each standard (a total of 18 analyses) must be performed in the following sequence: low standard, target, high standard, low standard, target standard, high standard; and

- The mean and variance of the six results for each solution must be calculated.

$$Mean = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} \qquad \text{Eq. E-2}$$

$$Variance = \frac{Y_{12} + Y_{22} + Y_{32} + Y_{42} + Y_{52} + Y_{62} - (6 \times Mean)_2}{5} \qquad \text{Eq. E-3}$$

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, Vp, is calculated.

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + V_3}{\frac{1.21}{3}} \qquad \text{Eq. E-4}$$

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

- The test statistic must be calculated:

$$TEST\ STATISTIC = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3}\right)^{0.5}} \qquad \text{Eq. E-5}$$

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

- The test statistic must be calculated:

$$TEST\ STATISTIC = \frac{\left| M_2 - \left(\frac{M_1}{1.8}\right) - \left(\frac{M_3}{2.2}\right) \right|}{\left(\frac{V_p}{4}\right)^{0.5}} \qquad \text{Eq. E-6}$$

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.

- The 95 percent confidence intervals for the mean result of each standard must be calculated:

$$INTERNAL\ FOR\ LOW\ STANDARD = M_1 \pm (2.13)\left(\frac{V_p}{6}\right)^{0.5} \qquad \text{Eq. E-7}$$

$$INTERNAL\ FOR\ TARGET\ STANDARD = M_2 \pm (2.13)\left(\frac{V_p}{6}\right)^{0.5} \qquad \text{Eq. E-8}$$

$$INTERNAL\ FOR\ HIGH\ STANDARD = M_3 \pm (2.13)\left(\frac{V_p}{6}\right)^{0.5} \qquad \text{Eq. E-9}$$

These intervals must not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable. In any event, the laboratory is responsible for the quality of the standards employed for analyses under this contract.

6.3 REQUESTING STANDARDS FROM THE EPA STANDARDS REPOSITORY

Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, depending on availability. The Contractor can 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.

6.4 DOCUMENTATION OF THE VERIFICATION AND PREPARATION OF CHEMICAL STANDARDS

It is the responsibility of each laboratory to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra, etc, whether produced by the laboratory or purchased from chemical supply houses, must be maintained by the laboratory and may be subject to review during on-site inspection visits. Documentation of standards preparation may be required to be sent to EPA for verification of contract compliance. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the laboratories for a period of one year.

SECTION 7

METHOD SPECIFIC QA/QC REQUIREMENTS

7.1 This section outlines the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of the volatile organic compounds listed in Exhibit C, using the procedures in Exhibit D for samples of ambient air captured on Tenax®. This section is not intended as a comprehensive quality control document, but rather is a guide to the specific QC operations that must be addressed during analysis of volatiles using this method. The laboratory is expected to address these operations in preparing the quality assurance plan and Standard Operating Procedures discussed in Sections 2 and 3 of this Exhibit.

7.2 The specific QC operations that must be considered for volatile organics analysis include the following:

- Testing and Spiking of Tenax® cartridges;
- GC/MS Instrument Performance Check and Ion Abundance Patterns;
- GC/MS Initial and Continuing Calibration;
- Internal Standard Area and Retention Time;
- Blank Analysis;
- Surrogate Compound Recoveries; and
- Performance Evaluation (PE) Samples;

7.2.1 Testing and Spiking of Tenax® Cartridges

7.2.1.1 As part of the procedure for preparing Tenax® cartridges for sampling and subsequent analysis, the laboratory is required to analyze by GC/MS or FID one cartridge from each batch of prepared Tenax® cartridges, and confirm that total VOCs are less than 10 ng. If the total VOCs exceed 10 ng, the entire batch of Tenax® cartridges shall be rejected.

7.2.1.2 For acceptable batches, the laboratory is also required to spike each cartridge with 100 ng each of PFT, 1,2-dichlorobenzene-d_4, as internal standards, and benzene-d_6, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4, as the surrogate compounds.

7.2.2 GC/MS Performance Checking the Mass Spectrometer and Ion Abundance Patterns

7.2.2.1 Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given

GC/MS system meets the instrument performance criteria specified in Exhibit D. The purpose of this instrument performance check is to ensure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of bromofluorobenzene (BFB).

7.2.2.2 The required frequency of BFB analysis (once every 12 hours on each GC/MS system) is described in detail in Exhibit D.

7.2.2.3 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Exhibit D, Section 2, Table 3.

7.2.2.4 The documentation includes Form IV-AAVT and a mass listing and bar graph spectrum for BFB analysis.

7.2.3 **Initial Calibration of GC/MS for Target and Surrogate Compounds**

Prior to the analysis of samples and required blanks and after instrument performance criteria have been met, the GC/MS system must be initially calibrated utilizing target compound (see Table 4) and surrogate compounds.

7.2.4 **Internal Standard Calibration Procedures**

7.2.4.1 The GC/MS may be calibrated by analyzing standards spiked onto Tenax® tubes, and calculating concentrations by the relative response factor (RRF) method.

7.2.4.2 Calibration standards containing the target compounds of interest are prepared as outlined in Exhibit D.

7.2.4.3 The Tenax® tubes are then spiked with known concentrations of internal markers and surrogate standards as described in Exhibit D.

7.2.4.4 The tubes are then analyzed as described in Exhibit D.

7.2.4.5 Relative response factors (RRFs) are calculated as described in Exhibit D.

7.2.4.6 The documentation includes Form VII-AAVT, a GC/MS data system printout for the analysis of each volatile calibration standard, and the mass spectrum of each target and surrogate compound.

7.2.5 **GC/MS Continuing Calibration for Target and Surrogate Compounds**

7.2.5.1 Once the GC/MS system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

7.2.5.2 The standard is to be analyzed according to the procedures and at the frequency given in Exhibit D.

7.2.5.3 The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum response factors of each compound in the continuing calibration and the percent difference must meet the criteria given in Exhibit D. Allowance is made for any two volatile compounds that fail to meet these criteria. The minimum or RRF of those two compounds must be greater than or equal to 0.010, and the percent difference must be less than or equal to 30.0 percent for the continuing calibration to be acceptable.

7.2.5.4 The documentation included Form VI-AAVT, a GC/MS data system printout for the analysis of the volatile calibration standard, and the mass spectrum of each target surrogate compound.

7.2.6 Internal Markers and Surrogate Standards and Retention Times

7.2.6.1 The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results, because the quantitative determination of volatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

7.2.6.2 The specific compounds used as internal markers and surrogate standards are given in Exhibit D. Each internal marker and surrogate standard is spiked on the Tenax® cartridge at a level of approximately 300 ng.

7.2.6.3 The retention time and the selected ion current profile (SICP) of each internal marker and surrogate standard must be monitored for all analyses.

7.2.6.4 The area response of each internal marker and surrogate standard from the SICP and the retention time of the internal marker and surrogate standard are evaluated for stability, according to the procedures in Exhibit D. The area of the internal marker and surrogate standard in a sample must not vary by more than 40 percent from the area of the same internal marker and surrogate standard in the associated continuing calibration standard. Likewise, the retention time of an internal marker and surrogate standard must be within 30 seconds of its retention time in the continuing calibration standard, as described in Exhibit D.

7.2.6.5 Requirements for analysis of samples when internal marker and surrogate standards do not meet specifications are given in Exhibit D.

7.2.6.6 The documentation includes Form III-AAVT and the GC/MS data system printout for the analysis of each sample, blank, matrix spike, and standard.

7.2.7 Method Blank Analysis

7.2.7.1 A method blank is a certified clean Tenax® tube thermally desorbed with a volume of helium carried through the entire analytical procedure. The volume of helium must be approximately equal to the volume associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

7.2.7.2 A method blank shall be analyzed once every 12 hours on each GC/MS system, as described in detail in Exhibit D.

7.2.7.3 For the purposes of this protocol, an acceptable method blank must contain less than or equal to the Contract Required Quantitation Limit (see Exhibit C) of any single target compound, or less than 10 ng/cartridge for total VOCs, whichever is less.

7.2.7.4 If a method blank exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination shall be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reanalysis of associated samples are given in Exhibit D.

7.2.7.5 The documentation includes Form IV-AAVT for the blank analysis and a GC/MS data system printout for the analysis of the method blank.

7.2.8 Surrogate Compound Recoveries

7.2.8.1 The recoveries of the three surrogate compounds are calculated from the analysis of each sample, blank, and matrix spike. The purpose of the surrogate compounds is to evaluate the performance of the Tenax® sampling and desorption system. Poor purging efficiency, leaks, and cold spots in transfer lines are only a few of the potential causes of poor recovery of these compounds.

7.2.8.2 The surrogate compounds are added to each Tenax® tube prepared for sampling at the concentration described in Exhibit D.

7.2.8.3 The recoveries of the surrogate compounds are calculated according to the procedures in Exhibit D. The recoveries must be within the quality control limits given in Exhibit D. If the recovery of any one surrogate compound is outside these limits, the Contractor must follow the steps outlined in Exhibit D.

7.2.8.4 The documentation includes Form IX-AAVT, and a GC/MS data system printout for the analysis of each sample, blank, and matrix spike.

7.2.9 Performance Evaluation (PE) Samples

7.2.9.1 Performance and method evaluation samples are intended to assist the Agency in monitoring Contractor performance. The laboratory will not be informed as to which compounds are contained in the PE samples or the concentrations.

7.2.9.2 The Laboratory shall extract, analyze, and report the results of the PE sample once per sample delivery group, if available.

7.2.9.3 The laboratory will receive PE samples on Tenax® cartridges from the Agency. The samples will come with instructions concerning the desorption procedure required for the PE samples. The Laboratory must add internal markers and surrogate compounds to the PE sample, following procedures in Exhibit D.

7.2.9.4 Each laboratory must extract and concentrate the PE sample using the procedure described in Exhibit D for those target compounds listed in Exhibit C, Table 1.

7.2.9.5 The laboratory must meet the following PE sample technical acceptance criteria as detailed in Exhibit D.

7.2.9.5.1 The PE sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria.

7.2.9.5.2 The PE sample must be prepared and analyzed with a method blank that meets the blank technical acceptance criteria.

7.2.9.5.3 The percent recovery for each of the surrogates must be within acceptable windows as outlined in Exhibit D.

7.2.9.5.4 The area response change between the PE sample and the most recent calibration standard analysis for each of the surrogate standards must be within 40 percent.

7.2.9.5.5 The percent recovery for each of the target compounds must be within replicate precision and audit accuracy, as outlined in Section 5, Exhibit D.

SECTION 8

REGIONAL DATA REVIEW

8.1 Contract laboratory data are generated to meet the specific needs of the Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of end-user, based upon functional aspects of data quality. General guidelines for data review have been developed jointly by the Region and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problems under investigation and the Regional response appropriate to the specific circumstances.

8.2 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, which is designed to identify contractual discrepancies, and the review done at EMSL/LV, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for program and laboratory administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

SECTION 9

LABORATORY EVALUATION SAMPLES

9.1 Although intralaboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in interlaboratory comparison studies conducted by the EPA. Results from the analysis of these laboratory evaluation samples will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

9.2 Sample sets may be provided to participating Contractors as frequently as on an SDG-by-SDG basis as a recognizable QC sample of known composition; as a recognizable QC sample of unknown composition; or not recognizable as a QC material. The laboratory evaluation samples may be sent either by the Regional client or the National Program Office, and may be used for contract action.

9.3 Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

9.4 At a minimum, the results are evaluated for compound identification, quantification, and sample contamination. Confidence intervals for the quantification of target compounds are based on reported values using population statistics. EPA may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample. Normally, a fraction of the compounds spiked into the sample are not specifically listed in the contract. Contractors are required to use the NIST/EPA/MSDC mass spectral library to tentatively identify a maximum number of non-target compounds in each fraction that are present above a minimal response. Tentative identification of these compounds based on contractually described spectral interpretation procedures is evaluated and integrated into the evaluation process.

9.5 A Contractor's results on the laboratory evaluation samples will determine the Contractor's performance as follows:

9.5.1 No response is required for a score of 90 percent or above.

9.5.2 For a score of 75 to 89, the Contractor shall describe the deficiency(ies) and the corrective action(s) taken in a letter to the APO, TPO, and EMSL/LV, within 14 days of receipt of notification from EPA.

9.5.3 For a score less than 75, the Contractor shall be notified by the APO or TPO concerning the remedy for its unacceptable performance. The

Contractor may expect, but EPA is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, a site visit, a full data audit, analysis of remedial PE samples, and/or a contract sanction, such as a Cure Notice.

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

SECTION 10

GC/MS TAPE AUDITS

10.1 Periodically, EPA requests from Contractors the GC/MS magnetic tapes corresponding to a specific Case in order to accomplish tape audits. Generally, tape submissions and audits are requested for the following reasons:

- Program overview;
- Indication of data quality problems from EMSL/LV, SMO, or Regional data reviews;
- Support for on-site audits; and
- Specific Regional requests.

10.2 Depending upon the reason for an audit, the 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 with that generated on the GC/MS tapes. This function provides external monitoring of Program QC requirements and checks adherence of the Contractor to internal QA procedures. In addition, tape audits enable EPA to evaluate the utility, precision, and accuracy of the analytical methods.

10.3 The GC/MS tape shall include raw data and quantitation reports for samples, blanks, laboratory evaluation samples, initial calibrations, continuing calibration, and BFB associated with the Case requested. The specific requirements for submissions of GC/MS tapes are discussed in Exhibit B.

10.4 Upon request of the Administrative Project Officer or EMSL/LV, the required tapes and all necessary documentation shall be submitted to EPA within seven (7) days of notification.

SECTION 11

ON-SITE LABORATORY EVALUATIONS

11.1 At a frequency dictated by a contract laboratory's performance, the Administrative Project Officer, Technical Project Officer 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 Evaluation and an Evidentiary Audit.

11.2 QUALITY ASSURANCE ON-SITE EVALUATION

11.2.1 Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures. The Contractor should expect that items to be monitored will include but not be limited to the following items:

- Size and appearance of the facility;
- Quantity, age, availability, scheduled maintenance and performance of instrumentation;
- Availability, appropriateness, and utilization of 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 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, laboratory evaluation sample scores, Regional review of data, Regional QA materials, GC/MS tape audit reports, and data trend reports.

11.3 EVIDENTIARY AUDIT

11.3.1 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. The evidence audit is comprised of the following three activities.

11.3.1.1 Procedural Audit

The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following laboratory operations:

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

11.3.1.2 Written SOPs Audit

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

11.3.1.3 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:

- Accuracy of the document inventory;
- Completeness of the file;
- 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 Administrative Project Officer (APO)/Technical Project Officer (TPO) 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

11.5.1 Following an on-site evaluation, QA and evidentiary audit reports which discuss deficiencies found during the on-site evaluation will be forwarded to the Contractor. The Contractor must discuss the corrective actions taken to resolve the deficiencies discussed during the on-site visit and discussed in the on-site reports in a letter to the APO/TPO, EMSL/LV (response to the QA report) and NEIC (response to the evidentiary report) within 14 days of receipt of the finding or within the time agreed upon between APO/TPO and the Contractor. If SOPs are required to be written or SOPs are required to be amended, the Contractor must provide the SOPs to the TPO, EMSL/LV (QA/technical SOPs) and NEIC (evidentiary SOPs) within 30 days of receipt of the finding or within the time agreed upon between the APO/TPO and the Contractor.

11.5.2 If the Contractor fails to take appropriate corrective action to resolve the deficiencies discussed in the on-site reports, a Contractor may expect, but the Agency is not limited to, the following actions:

- reduction of the number of samples sent under the contract;
- suspension of sample shipment to the Contractor;
- a follow-up site visit, a full data audit; and
- analysis of remedial PE samples and/or contract sanction, such as a Cure Notice.

SECTION 12

QUALITY ASSURANCE AND DATA TREND ANALYSIS

12.1 Data submitted by laboratories are subject to review from several aspects: compliance with contract-required QC, usability, and full data package evaluation. Problems resulting from any of these reviews may determine the need for a GC/MS 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 Agency 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 data base. Statistical reports that evaluate specific anomalies or disclose trends in many areas, including the following, are generated from this data base:

- Laboratory Control Sample;
- Blanks;
- GC/MS Instrument Performance Checks;
- Initial and Continuing Calibration Data; and
- Other QC and Method Parameters.

12.2 Program-wide statistical results are used to rank laboratories in order to observe the relative performance of each Contractor using a given protocol against its peers. The reports are also used to identify trends within laboratories. The results of many of these trends analyses are included in overall evaluation of a Contractor's performance, and are reviewed to determine if corrective action or an on-site laboratory evaluation is indicated in order to meet the QA/QC requirements of the contract.

12.3 Contractor performance over time is monitored using these trend analysis techniques to detect departures of Contractor output from required or desired levels of QC, and to provide an early warning of Contractor QA/QC problems which may not be apparent from the results of an individual case.

12.4 As a further benefit to the Program, the data base 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 contract laboratories is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of QC and performance criteria specifications of what is routinely achievable and expected of environmental chemistry laboratories in mass production analysis

of environmental samples. This, in turn, assists the Agency in meeting its objectives of obtaining data of known and documented quality.

SECTION 13

DATA MANAGEMENT

13.1 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 should 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.

13.2 Data manually entered from hard-copy must be quality controlled 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 must be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

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

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

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

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

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

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

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

13.5 Individual(s) responsible for the following functions must be identified:

- System operation and maintenance including documentation and training;

- Database integrity, including data entry, data updating and quality control; and

- Data and system security, backup and archiving.

SECTION 14

REFERENCES

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EXHIBIT F

EVIDENTIARY REQUIREMENTS

December, 1991

EXHIBIT F

EVIDENTIARY REQUIREMENTS

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SECTION 1

SAMPLE CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

1.1 SAMPLE IDENTIFICATION

1.1.1 To assure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

1.1.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA sample number.

1.2 CHAIN-OF-CUSTODY PROCEDURES

1.2.1 Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented.

1.2.2 A sample is under custody if the following applies:

1.2.2.1 It is in your possession.

1.2.2.2 It is in your view after being in your possession.

1.2.2.3 It was in your possession and you locked it up.

1.2.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

1.3 SAMPLE RECEIVING PROCEDURES

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available. The condition of the shipping containers and sample bottles shall be

inspected upon receipt by the sample custodian or his/her representative.

1.3.3 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

1.3.4.1 Airbills or airbill stickers.

1.3.4.2 Custody seals.

1.3.4.3 EPA custody records.

1.3.4.4 EPA traffic reports or SAS packing lists.

1.3.4.5 Sample tags

1.3.5 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

1.3.6 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

1.3.7 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

1.3.8 The following information shall be recorded on Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected:

1.3.8.1 Condition of the shipping container.

1.3.8.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

1.3.8.3 Custody seal numbers, when present.

1.3.8.4 Condition of the sample bottles.

1.3.8.5 Presence or absence of airbills or airbill stickers.

1.3.8.6 Airbill or airbill sticker numbers.

1.3.8.7 Presence or absence of EPA custody records.

1.3.8.8 Presence or absence of EPA traffic reports or SAS packing lists.

1.3.8.9 Presence or absence of sample tags.

1.3.8.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

1.3.8.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

1.3.8.12 Problems or discrepancies.

1.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 2

DOCUMENT CONTROL PROCEDURES

The goal of the laboratory document control program is to assure that all documents for a specified SDG will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

2.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

2.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the laboratory may exercise the option of using only laboratory or EPA

sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

2.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be crossed out.

2.2 CONSISTENCY OF DOCUMENTATION

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

2.2.2 All copies of laboratory documents shall be complete and legible.

2.2.3 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

2.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURES

2.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

2.3.2 All documents relevant to each SDG, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

2.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 of Exhibit E is an example of a document inventory.

2.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

2.5 SHIPPING DATA PACKAGES AND CSF

2.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

2.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

2.5.3 A copy of the transmittal letter for the CSF will be sent to NEIC and SMO.

2.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one original FORM AADC-1 for each shipping container.

2.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

2.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

2.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 3

STANDARD OPERATING PROCEDURES

The Contractor must have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, tracking the analysis of samples, and assembly of completed data.

3.1 SPECIFICATIONS FOR WRITTEN STANDARD OPERATING PROCEDURES

3.1.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation.

3.1.2 The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits. The Contractor must have written standard operating procedures (SOPs) for:

3.1.2.1 Sample receipt and logging.

3.1.2.2 Sample storage.

3.1.2.3 Preventing sample contamination.

3.1.2.4 Security for laboratory and samples.

3.1.2.5 Traceability of standards.

3.1.2.6 Maintaining instrument records and logbooks.

3.1.2.7 Sample analysis and data control systems.

3.1.2.8 Glassware cleaning.

3.1.2.9 Technical and managerial review of laboratory operation and data package preparation.

3.1.2.10 Internal review of contractually-required quality assurance and quality control data for each individual data package.

3.1.2.11 Sample analysis, data handling, and reporting.

3.1.2.12 Chain-of-Custody.

3.1.2.13 Document control, including Case file preparation.

3.1.3 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.1.4 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.1.4.1 Presence or absence of EPA chain-of-custody forms.

3.1.4.2 Presence or absence of airbills or airbill stickers.

3.1.4.3 Presence or absence of EPA Traffic Reports or SAS packing lists.

3.1.4.4 Presence or absence of custody seals on shipping and/or sample containers and their condition.

3.1.4.5 Custody seal numbers, when present.

3.1.4.6 Presence or absence of sample tags.

3.1.4.7 Sample tag ID numbers.

3.1.4.8 Condition of the shipping container.

3.1.4.9 Condition of the sample container.

3.1.4.10 Verification of agreement or nonagreement of information on receiving documents and sample containers.

3.1.4.11 Resolution of problems or discrepancies with SMO.

3.1.4.12 The definition of any terms used to describe sample condition upon receipt.

3.1.5 The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.1.6 The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

3.1.6.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.1.6.2 A description of the documentation used to record instrument calibration and other QA/QC activities.

3.1.6.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.1.7 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.1.8 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.1.9 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions. The Contractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.

3.1.10 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure.

3.1.11 The Contractor shall have written SOPs for laboratory safety.

3.1.12 The Contractor shall have written SOPs for cleaning of glassware used in preparing and analyzing samples under this contract.

3.1.13 The Contractor shall have SOPs for traceability of standards used in sample analysis QA/QC.

3.2 HANDLING OF CONFIDENTIAL INFORMATION

3.2.1 A Contractor conducting work under this contract may receive

EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.2.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.2.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

EXHIBIT G

GLOSSARY OF TERMS

December, 1991

EXHIBIT G

GLOSSARY OF TERMS

Aliquot - A measured portion of a field sample taken for analysis.

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

Analysis Group - An analysis group is a set of no more than twenty analytical samples (as defined below) for the purpose of method Quality Assurance/Quality Control (QA/QC), such that the QA/QC required by Exhibits D and E are, at a minimum, prepared and analyzed at a frequency of once per twenty analytical samples.

Analytical Sample - Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: field samples, duplicate samples, laboratory control sample (LCS), and performance evaluation (PE) samples.

ASTM Type II Water - Distilled water with a conductivity of less than 1.0 μmho/cm at 25°C. For additional specifications refer to ASTM D1193-77, "Standard Specification for Reagent Water".

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

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

Batch - A group of samples prepared at the same time.

Breakthrough volume (V_B) - Sample volume at which point a particular component will be initially detected in the eluate from the Tenax® sample cartridge.

Calibration - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. Calibration procedures differ for the various methods included in this document. Refer to the method of interest for a definition specific to that method.

Calibration Standards - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions are not subjected to the preparation method but contain the same matrix as the sample preparations to be analyzed.

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

Coefficient of Variation (CV) - The standard deviation as a percent of the arithmetic mean.

Continuing Calibration Standard - A standard solution prepared by the analyst to be used to verify the stability of the instrument calibration with time and the instrument performance during the analysis of samples. The continuing calibration standard should have a concentration in the middle of the calibrated range. Analytical standard run every 20 analytical samples or every 12 hours, whichever is more frequent, to verify the calibration of the analytical system.

Contract Required Quantitation Limit (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work. Generally defined as 3.3 (or more) times the standard deviation of seven replicate analyses of the method blank.

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

Correlation Coefficient - A number (r) which indicates the degree of dependence between two variables (e.g., concentration - absorbance). The more dependent they are the closer the value to one. Determine on the basis of the least squares line.

Cryogen - A liquified gas used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp - 195.8 °C).

Data System - For the purpose of this contract, computer system that allows the continuous acquisition and printout of time vs intensity data throughout the chromatographic program.

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

DDI - Deionized Distilled water.

Deuterated Chemicals - Those chemicals which contain deuterium (hydrogen isotope that is twice the mass of hydrogen); used as tracers for system quality assurance.

Field Blank - Any sample submitted from the field identified as a blank.

Field Sample - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

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

In-House - At the Contractor's facility.

Initial Calibration - Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical instrument to the target compounds.

Interferents - Substances which affect the analysis for the compound of interest.

Internal Standards -Compounds added to every standard, blank, sample at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target analysis.

Laboratory - Synonymous with Contractor as used herein.

Laboratory Control Sample (LCS)- Aliquot spiked with known concentration of specific compounds and subjected to the entire analytical procedure in order to monitor method and contractor performance.

Laboratory Method Blank (LMB)- A clean unused cartridge analyzed along with and performing the same analytical procedure as the field samples. The LMB cartridge never leaves the laboratory.

Laboratory Receipt Date - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

Linear Range - The concentration range over which the analytical curve remains linear. The range of the instrument for a specific compound, as determined using calibration standards. The upper limit of this linear range (determined at each analysis) is the highest concentration calibration standard that has a determined value within 10% of the known value.

Mass Spectral Interference - Defined as the inability to detect the internal standard quantification ion due to presence of high levels of mass spectral "noise" at the same mass.

Matrix - The predominant material of which the sample to be analyzed is composed.

Megabore® Column - One of two types of capillary columns, the other being the narrow bore, for the analysis of target compounds under this contract.

Method Detection Limit (MDL) - The chemical concentration that produces a signal, due to an analyte, which is equal to the student $t_{.99}$ times the standard deviation of a series of measurements on at least seven separate method blanks. In practice, a method detection limit will be substantially higher than an instrumental detection limit. The method detection limit for metals is $t_{.99}$ times the standard deviation of seven method blank analyses. Of course, all spectral background techniques must be operative and the same integration times must be utilized as when actual samples are analyzed.

MS-SCAN - The gas chromatograph (GC) is coupled to a mass selective detector where the instrument is programmed to acquire all mass for target compounds and to disregard all others.

Narrative (SDG Narrative) - Portion of the data package which includes laboratory, contract, SDG 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.

Narrow-Bore Capillary Column - One of two capillary columns, the other being the wide-bore (Megabore®) capillary column, for the analysis of compounds under this contract.

Performance Evaluation (PE) Sample - A sample of known composition provided by EPA for Contractor analysis. Used by EPA to evaluate Contractor performance.

Protocol - A compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

Qualitative Accuracy - The ability of an analytical system to correctly identify compounds.

Quantitative Accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

Reconstructed Ion Chromatogram (RIC) - A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Recovery - A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

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

Relative Response Factor (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where: A = area of the characteristic ion measured;
C = concentration;
is = internal standard; and
x = compound of interest.

Resolution - Also termed separation, the separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Retention Time (RT) - The time to elute a specific chemical from a chromatographic column for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until its maximum concentration appears at the detector.

Retention Time Window - Retention time window is determined for each compound of interest and is the time from injection to elution of a specific chemical from a chromatographic column. The window is determined by three injections of a single component standard over a 24-hour period as plus or minus three times the standard deviation of the absolute retention time for that compound.

Rounding Rules - If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no

figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

See forms instructions (Exhibit B) for exceptions.

Run - A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by this contract.

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

Sample Delivery Group (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. An SDG is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 samples within a Case; or
- Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

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

Selected Ion Current Profile (SICP) - A plot of ion abundance vs time or scan number for ions of a specified mass.

Standard Analysis - An analytical determination made with known quantities of target compounds; used to determine response factors.

Static Calibration - Calibration of an analytical system with known concentrations of calibration gas, obtained from a source such as gas cylinders or prepared from standard stock solutions.

Stock Solution - A standard solution which can be diluted to derive other standards.

Surrogates (Surrogate Standard) - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media.

Target compound - The compound an analysis seeks to determine; the compound of interest.

Tentatively Identified Compounds (TIC) - Compounds detected in samples that are not target compounds, internal standards or surrogate standards. Up to 10 peaks are subjected to mass spectral library searches for tentative identification.

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 is used for documenting sample condition and receipt by the laboratory.

Twelve-Hour Time Period - The twelve (12) hour time period for GC/MS system tuning, standards calibration (initial or continuing calibration) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of compliant tune. The time period ends after 12 hours has 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.

Volatile Compounds - Target compounds with normal vapor pressures $\geq$ 0.1 mm Hg by the analytical method in this document.

ATTACHMENT V

USEPA CONTRACT LABORATORY PROGRAM

SEMIVOLATILE ORGANICS ANALYSIS OF AMBIENT AIR

January, 1992

Revision SVAA01.0

SEMIVOLATILE ORGANICS ANALYSIS
OF AMBIENT AIR

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PREFACE

The purpose of this contract is to provide the U.S. Environmental Protection Agency (EPA) with chemical analytical services, quality control procedures, and an analysis structure which will generate data of known and documented quality. This document was developed with the guidance of the Air Toxics Workgroup to ensure that the needs of regional, state, and local air pollution programs are addressed.

The samples to be analyzed are of ambient air collected on PUF/XAD-2 at or in the vicinity of known or suspected hazardous waste sites and may contain potentially hazardous organic and inorganic materials at significant concentrations. The Contractor should be aware of the potential hazards associated with the handling and analyses of these samples. It is the Contractor's responsibility to take all necessary measures and precautions to ensure the health and safety of its employees. The Contractor is responsible for providing a safe working environment and making its employees aware of the potential hazards of working with and analyzing these samples.

Procedures specified herein shall be used in the preparation of PUF/XAD-2 cartridges and analysis of air samples for the presence and quantitation of certain semivolatile organic compounds (SVOCs). The Contractor shall employ safe handling procedures and generally accepted laboratory practices in the performance of contract requirements and shall follow the quality assurance and quality control (QA/QC) program specified herein.

The data obtained under this contract will be used by EPA to determine the existence and extent of risk posed by hazardous waste disposal sites to the public, to individuals involved in Superfund site cleanups, and to the environment. The data may be used in civil and/or criminal litigation which requires the strictest adherence to chain-of-custody protocol, document control, and quality assurance procedures.

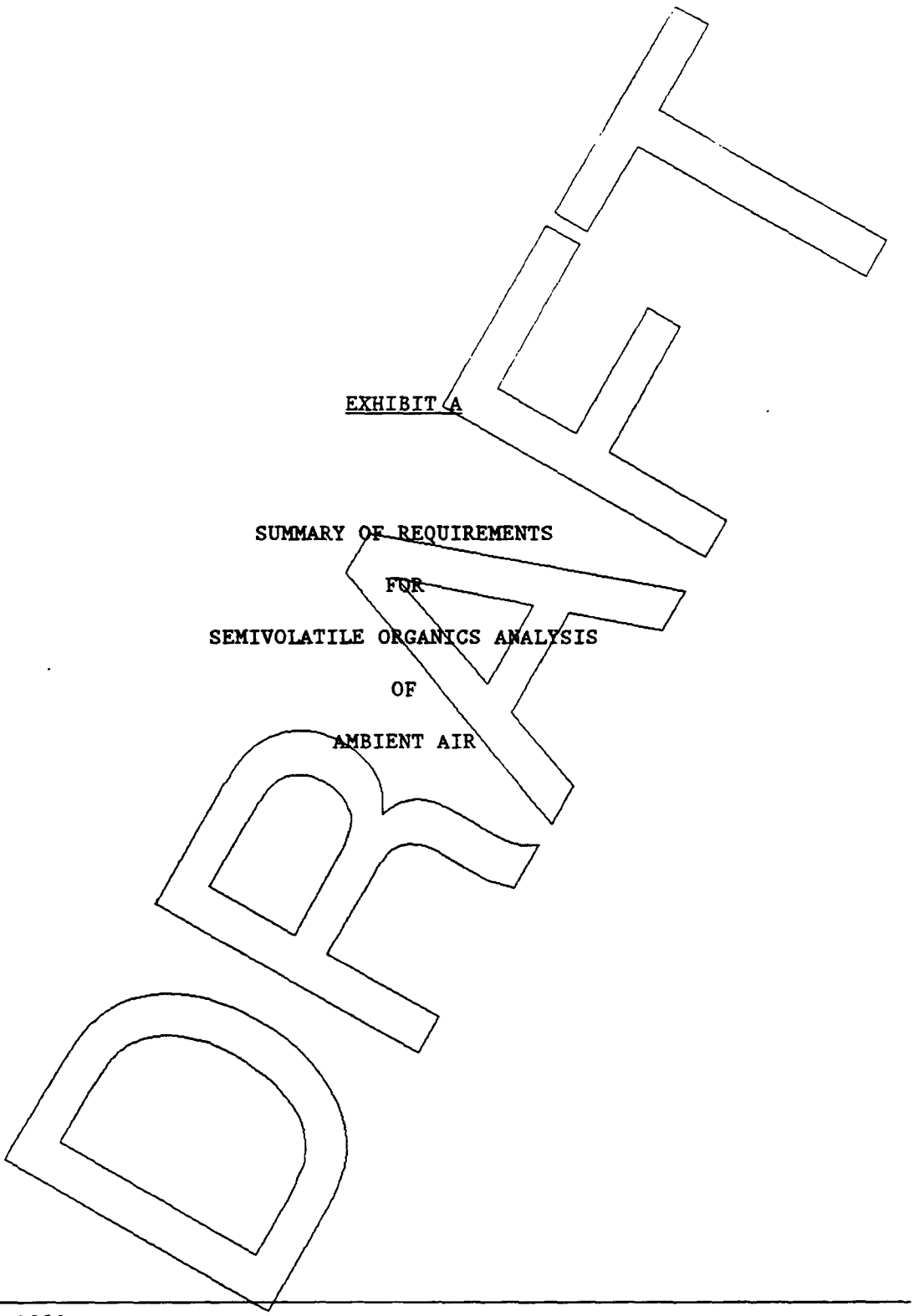

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR SEMIVOLATILE ORGANICS ANALYSIS OF AMBIENT AIR

January, 1992

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR
SEMIVOLATILE ORGANICS ANALYSIS OF AMBIENT AIR

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SECTION 1

GENERAL REQUIREMENTS

1.1 The Contractor shall employ procedures specified in this contract in the preparation and analysis of the ambient air samples for the presence and quantitation of the organic compounds listed in Exhibit C.

1.2 The Contractor shall use proven techniques to identify and measure the organic compounds presented in the Target Compound List (TCL) as specified in Exhibit C. The Contractor shall perform sample preparation and analysis procedures as prescribed in Exhibit D, and meet specified sample and extract preservation and holding time requirements.

1.3 For all samples analyzed under this contract, the Contractor shall adhere to the QA/QC protocols specified in Exhibit E and abide by the evidentiary protocols specified in Exhibit F.

1.4 Following sample analysis, the Contractor shall perform data reduction and shall report analytical activities, sample data, and quality control documentation as designated in Exhibit B. Exhibit B contains all reporting and deliverables requirements for this contract, including copies of the data reporting forms and form instructions guide.

1.5 To ensure proper understanding of the language in this contract, Exhibit G contains a glossary of terms. When a term is used in the text without explanation, the glossary meaning shall be applicable. Glossary definitions do not replace or take precedence over specific information included in the document text.

1.6 The samples to be analyzed by the Contractor are from known or suspected hazardous waste sites and may contain hazardous organic and/or inorganic materials at high concentration levels. The Contractor should be aware of the potential hazards associated with the handling and analysis of these samples. It is the Contractor's responsibility to take all necessary measures to ensure the health and safety of its employees. It is also the Contractor's responsibility to follow appropriate disposal procedures according to state and federal regulations.

1.7 In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, as it may be used to make major decisions regarding public health and environmental welfare. In addition, data generated under this contract may be used in litigation against potentially responsible parties in the enforcement of Superfund legislation.

SECTION 2

SPECIFIC REQUIREMENTS

For each sample, the Contractor shall perform the following tasks:

2.1 TASK I: RECEIVE AMBIENT AIR SAMPLES ON PUF/XAD-2 CARTRIDGES

2.1.1 The Contractor shall receive and handle samples under the chain-of-custody and document control procedures described in Exhibit F.

2.1.2 The Contractor shall provide the required analytical expertise and instrumentation for analyses of the TCL compounds equal to or lower than the quantitation limits specified in Exhibit C. In Exhibit D, EPA provides the Contractor with an appropriate set of analytical procedures that shall be used.

2.1.3 The Contractor shall extract samples and analyze the extracts within the maximum holding times specified in Exhibit D, even if these times are less than the maximum data submission time allowed in this contract.

2.1.4 The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic and/or inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the preparation and analysis with appropriate caution. The Contractor shall be responsible for all necessary measures and precautions to ensure the health and safety of laboratory employees.

2.2 TASK II: ANALYZE SAMPLES FOR THE IDENTIFICATION AND QUANTITATION OF SPECIFIC COMPOUNDS

2.2.1 For each sample received, the Contractor shall be required to perform the sample extraction and analysis procedures described in Exhibit D. The documentation that accompanies the sample(s) to the Contractor facility shall indicate specific analytical requirements for that sample or set of samples.

2.2.2 Exhibit D specifies the analytical procedures that shall be used. Exhibit D contains instructions and references for the analysis of ambient air samples containing low-to-medium concentrations of semivolatile organics for GC/MS analysis. GC/MS may use automated computer programs to facilitate the identification of organic compounds.

2.2.3 For the purpose of this contract, a full sample analysis is defined as analysis for all of the TCL constituents identified in Exhibit C in accordance with the methods in Exhibit D and performance of related

QA/QC as specified in Exhibit D and Exhibit E. Laboratory Control Samples (LCS) analyses shall be considered a separate full sample analysis. All other QA/QC requirements are considered an inherent part of this contract and are included in the contract sample unit price.

2.2.4 The semivolatile compounds analyzed by GC/MS techniques and initially identified shall be verified by an analyst competent in the interpretation of mass spectra by comparison of the suspect mass spectra to the mass spectra of a standard of the suspected compound. This procedure requires the use of multiple internal standards. Two criteria must be satisfied to verify the identifications:

2.2.4.1 Elution of the sample component at the same GC relative retention time as the standard component.

2.2.4.2 Correspondence of the sample component and standard component mass spectra.

2.2.5 For each sample analysis, the Contractor shall conduct mass spectral library searches of non-target compound sample components to determine tentative compound identifications as follows:

2.2.5.1 For each semivolatile organics analysis, the Contractor shall conduct a search to determine the possible identity of up to 10 organic compounds of greatest concentration which are not internal standards, surrogate compounds, and not listed in Exhibit C.

2.2.5.2 In performing searches, the most recent release of the National Institute of Standards and Technology (NIST)/EPA/MSDC mass spectral library must be used.

NOTE: Substances with responses of less than 10 percent of the nearest internal standard are not required to be searched in this fashion.

2.2.5.3 Only after visual comparison of sample spectra with the spectra from the library searches will the mass spectral interpretation specialist assign a tentative identification. If the compound does not meet the identification criteria, it shall be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, they also should be included.

2.3 TASK III: PERFORM REQUIRED QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

2.3.1 All specific QA/QC procedures prescribed in Exhibits D and E shall

be strictly adhered to by the Contractor. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibit B requirements.

2.3.2 The Contractor shall establish, and use on a continuing basis, QA/QC procedures including the daily or (as required) more frequent use of standard reference solutions from EPA, NIST, or secondary standards traceable thereto, where available at appropriate concentrations (i.e., standard solutions designed to ensure that operating parameters of equipment and procedures, from sample receipt through identification and quantitation, produce reliable data). Exhibits D and E provide specific QA/QC requirements.

2.3.3 Additional QA/QC shall be required quarterly or more frequently, i.e., with each Case or Sample Delivery Group (SDG), in the form of Laboratory Control Samples (LCS) and Performance Evaluation (PE) samples for semivolatile organics submitted to EPA for Contractor analysis, and in the form of verification of instrument parameters, as described in Exhibit E.

2.3.3.1 EPA has provided to the Contractor formats for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in this contract and within the time specified in the Contract Performance/Delivery Schedule.

2.3.3.2 Use of formats other than those designated by EPA will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government will be required.

2.3.3.3 Computer generated forms may be submitted in the hardcopy data package(s) provided that the forms are in exact EPA format. This means that the order of data elements is the same as on each EPA required form, including form numbers and titles, page numbers and header information, columns, and lines.

2.3.4 The Contractor shall provide analytical equipment and technical expertise for this contract as specified by the following:

2.3.4.1 Gas chromatograph/mass spectrometer (GC/MS) data system capable of meeting all the terms and conditions of the Contract with the following requirements:

2.3.4.1.1 The computer shall be interfaced by hardware to the mass spectrometer and be capable of acquiring continuous mass scans for the duration of the chromatographic program.

2.3.4.1.2 The computer shall be equipped with mass storage devices for saving all data from the GC/MS runs.

2.3.4.1.3 Computer software shall be available to allow searching GC/MS runs for specific ions and plotting the intensity of the ions with respect to time or scan number.

2.3.4.1.4 A computer data system must be interfaced to the MS that allows the continuous acquisition and storage, on machine-readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP) or Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

2.3.4.1.5 The GC/MS shall be equipped with a GC to MS interface capable of extending a fused silica capillary column into the ion source. The column is to be 50 meters long by 0.25 to 0.53 mm I.D. 100% methyl silicone or 5% phenyl, 95% methyl silicone capillary column, or equivalent.

2.3.4.2 The Contractor shall use a magnetic tape storage device capable of recording data and suitable for long-term, off-line storage. The Contractor shall retain all raw GC/MS data acquired under this contract on magnetic tape in appropriate instrument manufacturer's format. The Contractor is required to retain the magnetic tapes with associated hardcopy tape logbook identifying tape contents (see Exhibit B) for 365 days after data submission. During that time, the Contractor shall submit tapes and logbook within seven days of request, as specified in the Contract Performance/Delivery Schedule.

2.3.4.3 The Contractor shall have a computerized MS library search system capable of providing a forward comparison, using the standard spectra contained in the mass spectral library. The 1985 (or most recent) release of the NIST library (containing 42,261 spectra) must be used.

2.3.4.4 The system shall provide a numerical ranking of the standard spectra most closely corresponding to the sample spectra examined,

and the data system shall have software capable of removing background signals from spectra.

2.3.4.5 The Contractor shall have, in-house and operable, a device capable of analyzing semivolatile organics as described in Exhibit D.

2.3.4.6 The Contractor shall have, in-house, the appropriate standards for all target compounds listed in Exhibit C prior to accepting any samples from the Sample Management Office (SMO). Standards provided by EPA for use in the Preaward Performance Evaluation may not contain all the target compounds and thus shall not be used for routine analyses unless or until they have been supplemented with commercially available standard materials.

2.3.5 The minimum functional requirements necessary to meet the terms and conditions of this contract are listed below. The Contractor shall designate and use qualified key personnel to perform these functions. The EPA reserves the right to review personnel qualifications and experience.

2.3.5.1 Project Manager

2.3.5.2 GC/MS Laboratory Supervisor

2.3.5.3 Quality Assurance Officer

2.3.5.4 Systems Manager

2.3.5.5 Program Analyst

2.3.5.6 GC/MS Operators

2.3.5.7 Mass Spectral Interpreter

2.3.5.8 Sample Preparation/Extraction Supervisor;

2.3.5.9 Sample Preparation/Extraction Specialist;

2.3.5.10 Chemist (back-up)

NOTE: The Contractor shall designate a Sample Custodian and a Document Control Officer.

2.3.6 The Contractor shall respond within 10 days to requests from data recipients for additional information or explanations that result from the Government's inspection activities.

2.3.7 The Contractor is required to retain unused samples and sample extracts, and used sample containers for a period of 60 days after data

submission unless otherwise instructed in Exhibit B or Exhibit D.

2.3.8 The Contractor shall adhere to the chain-of-custody and document control procedures described in Exhibit F. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported in the Complete Case File Purge (Exhibit B).

2.3.9 Sample shipments to the Contractor's facility will be scheduled and coordinated by SMO, acting on behalf of the Administrative Project Officer (APO). The Contractor shall communicate with SMO personnel by telephone as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

2.3.10 If there are problems with the samples (e.g., mixed media, containers broken) or sample documentation/paperwork (e.g., Traffic Reports not with shipment, or sample and Traffic Report numbers do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

2.3.11 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case. A Case consists of one or more SDG(s). An SDG is defined by the following:

2.3.11.1 Each Case of field samples received, or

2.3.11.2 Each 20 field samples within a Case, or

2.3.11.3 Each seven 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).

2.3.12 Data for all samples in an SDG must be submitted together (in one package) in the order specified in Exhibit B. The SDG number is the EPA number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number is the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. The SDG number is reported on all data reporting forms. The SDG Receipt Date is the day

that the last sample in the SDG is received.

2.3.13 The Contractor is responsible for identifying each SDG as samples are received, through proper sample documentation (see Exhibit B) and communication with SMO personnel.

2.3.14 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report (TR) form bearing the sample number and descriptive information regarding the sample. The Contractor shall complete and sign the TR, recording the date of sample receipt and sample condition on receipt for each sample container.

2.3.15 The Contractor shall submit signed copies of TRs for all samples in an SDG to SMO within three calendar days following receipt of the last sample in the SDG. TRs shall be submitted in SDG sets (i.e., all TRs for an SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

2.3.16 EPA Case numbers (including SDG numbers) and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.

2.3.17 Samples will be routinely shipped directly to the Contractor through a delivery service. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays and holidays. As necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station, or other carrier service within the Contractor's geographical area.

2.3.18 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

SECTION 3

DETAILED TECHNICAL & MANAGEMENT REQUIREMENTS

The Contractor shall have the following technical and management capabilities:

3.1 PERSONNEL

3.1.1 Project Manager

3.1.1.1 Responsible for all technical efforts of the laboratory to meet all terms and conditions of the contract.

3.1.1.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.1.3 Experience: Minimum of three years of laboratory experience, including at least one year in a supervisory position.

3.1.2 GC/MS Laboratory Supervisor

3.1.2.1 Responsible for all technical efforts of the GC/MS laboratory to meet all terms and conditions of the contract.

3.1.2.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.2.3 Experience: Minimum of three years of laboratory experience in operating a GC/MS, including at least one year in a supervisory position.

3.1.3 Quality Assurance Officer

3.1.3.1 Responsible for overseeing the quality assurance aspects of data generation and reporting directly to upper management.

3.1.3.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.3.3 Experience: Minimum of three years of laboratory experience, including at least one year of applied experience with QA principles and practices in an analytical laboratory.

3.1.4 Systems Manager

3.1.4.1 Responsible for the management and quality control of all computing systems (hardware, software, documentation, and procedures), generating, updating, and performing quality control on

automated deliverables.

3.1.4.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, database management systems, or systems requirements analysis.

3.1.4.3 Experience: Minimum of three years experience in data or systems management or programming including one year experience with software used for data management and generation of deliverables.

3.1.5 **Program Analyst**

3.1.5.1 Responsible for the installation, operation, and maintenance of software and programs; generating, updating, and performing quality control procedures on analytical databases and automated deliverables.

3.1.5.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, information systems, or systems requirements analysis.

3.1.5.3 Experience: Minimum of two years experience in systems or applications programming including one year of experience with software used for data management and generation of deliverables.

3.1.6 **Gas Chromatography/Mass Spectrometer (GC/MS) Operator**

3.1.6.1 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.6.2 Experience: Minimum of one year of experience in operating and maintaining GC/MS instruments in conjunction with the education requirement; or in lieu of education requirement, three additional years of experience in operating and maintaining GC/MS instrumentation.

3.1.7 **Mass Spectral Interpreter**

3.1.7.1 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline with specialized training in GC/MS.

3.1.7.2 Experience: Minimum of two years of applied experience with GC/MS analysis of environmental samples.

3.1.8 **Sample Preparation/Extraction Supervisor**

3.1.8.1 The bidder shall have a minimum of one extraction specialist responsible for all technical efforts of sample preparations and extractions to meet all terms and conditions of the EPA contract.

3.1.8.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.8.3 Experience: Minimum of three years of laboratory experience, including at least one year of supervisory experience.

3.1.9 **Sample Extraction/Preparation Specialist**

3.1.9.1 Education: Minimum of Bachelor's degree in chemistry, or equivalent.

3.1.9.2 Experience: Minimum of one year of applied experience in an analytical laboratory.

3.1.10 **Technical Staff Redundancy**

3.1.10.1 In order to ensure continuous operations to accomplish the required work as specified by the contract, the bidder shall have a minimum of one chemist available at all times as a back-up technical person with the following qualifications.

3.1.10.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.10.3 Experience: Minimum of one year of experience in each of the following areas: GC/MS operation and maintenance.

3.2 **FACILITIES**

The adequacy of the facilities and equipment is as important as the technical staff for accomplishing the required work as specified by the EPA contract.

3.2.1 **Sample Receipt Area**

Adequate, contamination-free, well-ventilated work space with chemical resistant bench top shall be available for receipt and safe handling of EPA samples.

3.2.2 **Storage Area**

Sufficient refrigerator space to maintain unused EPA sample volume for up to 60 days after data submission shall be provided. Semivolatile samples must be stored in a refrigerator used only for storage of semivolatile samples from this contract. Samples must be stored in an atmosphere demonstrated to be free from all potential contaminants. Samples, sample extracts, and standards must be stored separately.

3.2.3 **Sample/Standard Preparation Area**

Adequate, contamination-free, well-ventilated work space shall be provided with:

3.2.3.1 Benches with chemical resistant tops.

3.2.3.2 Exhaust hoods.

3.2.3.3 Glove box or isolated area in which to prepare standard materials.

3.2.3.4 Source of distilled or demineralized organic-free water.

3.2.3.5 Analytical balance(s) located away from draft and rapid change in temperature.

3.3 **INSTRUMENTATION**

At a minimum, the Contractor shall have the following instruments operative at the time of the Preaward Site Evaluation and committed for the full duration of the contract.

3.3.1 **100 Samples/Month Capacity Requirements**

No. of Instrument(s)	Type of Instrument
1	GC/MS

NOTE: The Contractor shall have one (1) complete GC/MS system available (operational) at all times as a back-up system. These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.2 **200 Samples/Month Capacity Requirements**

No. of Instrument(s)	Type of Instrument
2	GC/MS

NOTE: These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.3 **Instrument Specifications**

Further information on instrument specifications and required ancillary equipment may be found in Exhibit D and other Exhibits in this contract.

3.4 **DATA HANDLING AND PACKAGING**

The Contractor shall be able to submit reports and data packages as specified in Exhibit B. To complete this task, the Contractor shall be required to:

3.4.1 Provide space, tables, and copy machines to meet the contract requirements.

3.4.2 Designate personnel responsible for report preparations and submission.

3.5 **LABORATORY MANAGEMENT CAPABILITY**

The Contractor shall have an organization with well-defined responsibilities for each individual in the management system to ensure sufficient resources for EPA contract(s) and to maintain a successful operation. To establish this capability, the Contractor shall designate personnel to carry out the following responsibilities for the EPA contract. Functions include, but are not limited to, the following:

3.5.1 **Technical Staff**

Responsible for all technical efforts for the EPA contract such as sample analysis, sample validation, and troubleshooting of all instruments.

3.5.2 **Project Manager**

Responsible for overall aspects of EPA contract(s) (from sample receipt through data delivery) and shall be the primary contact for EPA Headquarters Administrative Project Officer (APO) and Regional Technical Project Officers (TPO).

3.5.3 **Sample Custodian**

Responsible for receiving the EPA samples (logging, handling, and storage).

3.5.4 **Quality Assurance Officer**

Responsible for overseeing the quality assurance aspects of the data and reporting directly to upper management.

3.5.5 Document Control Officer

Responsible for ensuring that all documents generated are placed in the Complete SDG File for inventory and are delivered to the appropriate EPA Region or other receiver as designated by EPA.

EXHIBIT B

REPORTING

AND

DELIVERABLES REQUIREMENTS

January, 1992

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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SECTION 1

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 The following table summarizes the contract reporting and deliverables requirements specified in the Contract Schedule and includes the distribution of each deliverable.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The EPA APO or SMO will notify the Contractor in writing of such changes when they occur.

Item	No. of Copies	Schedule and Delivery	Distribution		
			(1)	(2)	(3)
Updated Standard Operating Procedures (SOPs)	2	45 days after contract award.		X	X
*Sample Traffic Reports	1	***3 days after receipt of last sample in Sample Delivery Group (SDG).	X		
**Sample Data Summary Package	1	14 days after receipt of last sample in SDG.	X		
**Sample Data Package including the Performance Evaluation (PE) Sample	3	35 days after receipt of last sample in SDG	X	X	X
Results of Intercomparison Study/Preaward Performance Evaluation (PPE) Sample	2	35 days after receipt of last sample in SDG	X	X	
Complete SDG File	1	35 days after data receipt of last sample in SDG.		X	
GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO.	As Directed		
****Quality Assurance Plan	1	Submit copy within 7 days by written request by APO.	As Directed		

Distribution

(1) Sample Management Office
(2) Environmental Monitoring Systems Laboratory-Las Vegas
(3) USEPA Region

* Also required in each Sample Data Package.

** Concurrent delivery of these items to all recipients is required.

*** An SDG is a group of samples within a Case, received over a period of seven days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. (See Exhibit A, Task III, for further description).

**** See Exhibit E for description.

NOTE: As specified in the Contract Schedule in the IFB (Government Furnished Supplies and Materials), unless otherwise instructed by SMO, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of analytical data.

Address

(1) USEPA Contract Laboratory Program
Sample Management Office
P.O. Box 818
Alexandria, VA 22313

For overnight delivery service, use street address:

300 North Lee Street
Alexandria, VA 22313

(2) USEPA Environmental Monitoring Systems Laboratory
P.O. Box 93478
Las Vegas, NV 89193-3478
ATTN: Data Audit Staff

For overnight delivery service, use street address:

944 E. Harmon, Executive Center
Las Vegas, NV 89109
ATTN: Data Audit Staff

(3) USEPA REGIONS:

SMO, acting on behalf of the EPA APO, will provide the Contractor with the list of addresses for the 10 EPA Regions. SMO will provide the Contractor with updated Regional name/address lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The APO will notify the Contractor in writing of such changes when they occur.

SECTION 2

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

2.1 The Contractor shall provide reports and other deliverables according to the schedule specified in Section F of the IFB, "SCHEDULE INFORMATION." The required content and form of each deliverable is described in this Exhibit.

2.1.1 All reports and documentation shall be:

2.1.1.1 Legible;

2.1.1.2 Clearly labeled and completed in accordance with instructions in this Exhibit;

2.1.1.3 Arranged in the order specified in this section;

2.1.1.4 Paginated; and

2.1.1.5 Single-sided.

2.1.2 If submitted documentation does not conform to the above criteria, the Contractor will be required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the Government.

2.1.3 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation or through an APO/TPO action, the data shall be clearly marked as "ADDITIONAL DATA" and shall be sent to all three contractual data recipients (SMO, EMSL-LV, and Region). A cover letter shall be included that describes which data are being delivered, to which EPA Case(s) the data pertain, and who requested the data.

2.1.4 Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with the required documentation and contains the required data forms in EPA-specified format.

2.1.5 Descriptions of the requirements for each deliverable item cited in the Contract Performance/Delivery Schedule (see Section F of the IFB "SCHEDULE INFORMATION") are specified in this Section. Items submitted concurrently must be arranged in the order listed. Additionally, the components of each item must be arranged in the order presented herein.

2.2 UPDATED STANDARD OPERATING PROCEDURES

2.2.1 The Contractor shall submit updated copies of all required Standard Operating Procedures (SOPs) that were submitted with the Prebid Performance

Evaluation (PPE) sample results. The updated SOPs must address any and all issues of laboratory performance and operation identified through the review of the PPE sample data and the evaluation of Bidder-Supplied Documentation.

2.2.2 The Contractor must supply SOPs for the following.

2.2.2.1 Evidentiary SOPs.

2.2.2.2 Sample receipt and logging.

2.2.2.3 Sample and extract storage area.

2.2.2.4 Preventing sample contamination.

2.2.2.5 Security for laboratory and samples.

2.2.2.6 Traceability/equivalency of standards.

2.2.2.7 Maintaining instrument records and bound logbooks.

2.2.2.8 Glassware cleaning.

2.2.2.9 Technical and managerial review of laboratory operation and data package preparation.

2.2.2.10 Internal review of contractually-required QA/QC data for each individual data package.

2.2.2.11 Sample extraction and analysis, data handling, and data reporting.

2.2.2.12 Chain-of-custody and document control, including case file preparation.

2.2.2.13 Sample data validation/self-inspection system, including:

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to ensure that hardcopy data are complete and compliant with the requirements in Exhibit B;
- Demonstration of internal QA inspection procedure (demonstrated

by supervisory sign-off on personal notebooks, internal PE samples, etc.);

- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

2.2.2.14 Data Handling.

2.2.2.14.1 Data Management procedures are defined as written procedures that are clearly defined for all databases and files used to generate or re-submit deliverables specifying the acquisition or entry, update, correction, deletion, storage, and security of computer-readable data and files. Key areas of concern include: system organization including personnel and security, demonstration, operations, traceability, and quality control.

2.2.2.14.2 Data manually entered from hardcopy must be subjected to quality control procedures and error rates estimated.

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

- Justification or rationale for the change;
- Initials of the person making the changes or changes. Data changes must be identified when generating the deliverables;
- Changed documentation must be retained according to the schedule of the original deliverable;
- Resubmitted deliverables must be reinspected as a part of the laboratory's internal inspection process prior to submission. The entire deliverable and not just the changes must be reinspected;
- The laboratory manager must approve changes to originally submitted deliverables; and

- Documentation of data changes may be requested by laboratory auditors.

2.2.2.14.4 Life cycle management procedures must be applied to computer systems used to generate and edit contract deliverables. Such systems must be thoroughly tested and documented prior to utilization.

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

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

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

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

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

- System operation and maintenance including documentation and training; and
- Database integrity including data entry, data updating and QC.

2.2.2.14.10 Data and system security, backup, and archiving.

2.3 SAMPLE TRAFFIC REPORTS

2.3.1 The original sample TR page marked "Lab Copy for Return to SMO" shall be submitted to SMO with laboratory receipt information and signed in original Contractor signature, for each sample in the SDG.

2.3.2 TRs shall be submitted in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

2.3.3 The SDG Cover Sheet shall contain the following items:

2.3.3.1 Laboratory name.

2.3.3.2 Contract number.

2.3.3.3 Sample analysis price - full sample price from contract.

2.3.3.4 Case number.

2.3.3.5 List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations), and the "last" sample received would be the highest sample number (considering both alpha and numeric designations).

2.3.4 Each TR shall be clearly marked with the SDG Number and the EPA sample number of the first sample in the SDG. This information shall be entered below the laboratory receipt date on the TR. The TR for the last sample received in the SDG shall be clearly marked "SDG - FINAL SAMPLE."

2.3.5 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the laboratory shall make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.

2.4 SAMPLE DATA SUMMARY PACKAGE

2.4.1 As specified in the Delivery Schedule, one Sample Data Summary Package shall be delivered to SMO concurrently with delivery of other required sample data. The Sample Data Summary Package shall be submitted separately (i.e., separated by rubber bands, clips or other means) directly preceding the Sample Data Package.

2.4.2 The Sample Data Summary Package shall contain data for samples in one SDG of the Case, as follows:

2.4.2.1 Cover Page (COVER PAGE - AASV).

2.4.2.2 By sample, tabulated target compound results (FORM I-AASV) and tentatively identified compounds (FORM I-AASV-TIC).

2.4.2.3 Laboratory Control Sample results (FORM III-AASV).

2.4.2.4 Blank summary (FORM II-AASV) and tabulated results (FORM I-AASV) including tentatively identified compounds (FORM I-AASV-TIC).

2.4.2.5 Initial and Continuing Calibration Data (FORM V-AASV and FORM VI-AASV).

2.4.2.6 Internal Standard Area and Retention Time Summary (FORM VII-AASV).

2.4.2.7 Filter/Adsorbent Cartridge Certification Data Sheet (FORM VIII-AASV)

2.4.2.8 Surrogate Recovery (FORM IX-AASV).

2.4.2.9 Analytical Sequence (FORM X-AASV).

2.5 SAMPLE DATA PACKAGE

2.5.1 The sample data package shall be complete, consecutively paginated, and shall include data for analysis of all samples in an SDG such as field samples, blanks, and laboratory control samples.

2.5.2 The sample data package is divided into five units as follows:

2.5.2.1 Cover page.

2.5.2.1.1 This document shall be clearly labeled "Cover Page." The Cover Page shall contain: laboratory name; laboratory code; contract number; case number; SDG number; SAS number; EPA sample numbers in alphanumeric order, showing EPA sample number cross-referenced with laboratory ID numbers; and comments, describing in detail any problems encountered in processing the samples in the data package.

2.5.2.1.2 The Cover Page shall contain the following statement, verbatim:

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature."

2.5.2.1.3 This statement shall be directly followed by the signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

2.5.2.1.4 In the event that the Laboratory Manager cannot validate

all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

2.5.2.2 Sample data (Results).

2.5.2.2.1 Sample data shall be arranged in packets with the Analysis Data Sheet (FORM I-AASV, including FORM I-AASV-TIC), followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.

NOTE: FORM I AASV-TIC is the tabulated list of the highest probable match for up to 10 organic compounds that are not surrogates and internal standards and are not listed in Exhibit C (TCL). It includes the Chemical Abstracts Service (CAS) Registry Number, tentative identification, and estimated concentration.

2.5.2.2.1.1 Reconstructed total ion chromatograms (RIC) for each sample or sample extract.

2.5.2.2.1.2 RICs must be normalized to the largest nonsolvent component, and must contain the following header information:

- EPA sample number;
- Date and time of analysis;
- GC/MS instrument ID; and
- Laboratory file ID.

2.5.2.2.1.3 Internal standards are to be labeled with the names of compounds, either directly out from the peak, or on a printout of retention times if retention times are printed over the peak.

2.5.2.2.1.4 Quantitation Report: The complete data system report must be included in all sample data packages, in addition to the reconstructed ion chromatogram for preliminary identification and/or quantitation using either the automated or manual data system procedures. The complete data system report shall include all of the information listed below:

- EPA sample number;
- Date and time of analysis;

- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- GC/MS instrument ID; and
- Laboratory file ID.

2.5.2.2.1.5 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report.

2.5.2.2.1.6 Target Compound Mass Spectra: For each sample, by each compound identified, copies of the raw spectra and copies of background subtracted mass spectra of target compounds listed in Exhibit C that are identified in the sample and corresponding background subtracted target compound standard mass spectra shall be included in the data package. Spectra must be labeled with EPA sample number, laboratory file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.

2.5.2.2.1.7 Tentatively Identified Compound Mass Spectra and Library Matches: For each sample, by each compound identified, copies of mass spectra of organic compounds not listed in Exhibit C, Tentatively Identified Compounds, with associated best-match spectra (three best matches), labeled as above shall be included in the data package.

2.5.2.2.2 Semivolatile Standard Data:

2.5.2.2.2.1 Initial Calibration: All initial calibration data must be included for all analyses associated with the SDG. When more than one initial calibration is performed, the reconstructed ion chromatograms and quantitation reports and each type of form must be put in chronological order, by instrument as follows:

- Initial Calibration Data Sheet (FORM V-AASV);
- Internal Standard Area and Retention Time Summary (FORM VII-AASV); and
- Semivolatile standard(s) reconstructed ion chromatograms

and quantitation reports (or legible facsimiles) for the initial (five point) calibration are labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.2.2.2 Continuing Calibration: When more than one continuing calibration is performed, the reconstructed ion chromatograms and quantitation reports and each type of form must be put in chronological order, by instrument if more than one instrument is used as follows:

- Continuing Calibration Data Sheet (FORM VI-AASV);
- Internal Standard Area and Retention Time Summary (FORM VII-AASV); and
- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimiles) for the initial (five point) calibration are labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are not required.

2.5.2.3 Quality control summary.

2.5.2.3.1 The quality control summary shall contain the following forms:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order by date of analysis or instrument.

- Blank Summary (FORM II-AASV);
- GC/MS Instrument Performance Check (FORM IV-AASV); and
- Internal Standard Area and RT Summary (FORM VII-AASV).

2.5.2.3.2 The quality control summary shall also contain the following:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order by date of analysis or instrument.

- GC/MS Tuning Data
  - GC/MS Tuning - BFB data, for each 12-hour period, shall be arranged in chronological order by instrument for each GC/MS system utilized;
  - GC/MS Tuning and Mass Calibration - BFB (FORM IV-AASV);

- Bar graph spectrum, labeled as in 2.5.2.2.1.2 and 2.5.2.2.1.4; and

- Mass listing, labeled as in 2.5.2.2.1.2. and 2.5.2.2.1.4.

• Blank data shall be arranged in chronological order by instrument. The blank data shall be arranged in packets with both of the Semivolatile Organics Analysis Data Sheets (FORM I-AASV and FORM I-AASV-TIC), followed by the raw data for semivolatile samples.

• Laboratory Control Sample Data

- Laboratory Control Sample Data Sheet (FORM III-AASV); and

- Reconstructed ion chromatograms and quantitation reports or legible facsimile (GC/MS), labeled according to 2.5.2.2.1.2 and 2.5.2.2.1.4. Spectra are <u>not</u> required.

2.5.2.4 Raw data.

2.5.2.4.1 For each reported value, the Contractor shall include all raw data from the instrument used to obtain the sample values (except for raw data for quarterly verifications of instrument parameters). Raw data shall contain all instrument readouts used for the sample results, including those readouts that may fall below the method quantitation limit. All GC/MS instruments must provide legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included.

2.5.2.4.2 All raw data shall include concentration units for GC/MS.

2.5.2.4.3 Organic raw data must be labeled with EPA sample number and appropriate codes as shown in Table B-1 to identify unequivocally the following:

• Initial and continuing calibration standards;

• Blanks;

• Duplicates;

• Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation;

- Data and EPA sample number for GC/MS analyses clearly and sequentially identified on the raw data;
- All calculations for sample data, including percent recovery, coefficient of variation, slope and y-intercept of linear fit; and
- Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide time of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as interference check samples and linear range analysis standards.

2.5.2.5 Preparation logs.

These logs must include the following:

- Date;
- Standard weights and/or volumes;
- Sufficient information to identify unequivocally which QC samples (e.g., laboratory control sample, blank) correspond to each batch prepared; and
- Comments describing any significant sample changes or reactions which occur during preparation.

2.5.2.6 Sample TRs.

A legible copy of the sample TRs and SDG Cover Sheet shall be submitted as described in section 2.3 of this Exhibit for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample number order, considering both alpha and numeric designations.

2.6 RESULTS OF INTERCOMPARISON/PERFORMANCE EVALUATION SAMPLE ANALYSES

The reporting of analytical results for Intercomparison Study/Preaward Performance Evaluation (PPE) sample analyses includes all requirements specified in section 2.4 for reporting of sample data. The PPE sample shall be carried through the exact same process as an analytical and field samples.

2.7 COMPLETE CASE FILE PURGE

2.7.1 The Complete SDG File package includes all laboratory records received or generated for a specific Case that have not been previously submitted to EPA as a deliverable. These items shall be submitted to EPA as a deliverable. These items shall be submitted along with their Document Inventory Sheet FORM AADC-2 (see Exhibit E for description of document numbering and inventory procedure). These items include, but are not limited to, sample tags, custody records, sample tracking records, analysts' logbook pages, bench sheets, instrument readout records, computer printouts, raw data summaries, instrument logbook pages (including instrument conditions), correspondence, and the document inventory.

2.7.2 Shipment of the Complete SDG File package by first class mail, overnight courier, priority mail, or equivalent, is acceptable. Custody seals, which are provided by EPA, shall be placed on shipping containers and a document inventory and transmittal letter included. The Contractor is not required to maintain any documents for a sample Case after submission of the Complete SDG File package; however, the Contractor should maintain a copy of the document inventory and transmittal letter.

2.8 GC/MS TAPES

2.8.1 The Contractor must store all raw and processed GC/MS data on magnetic tape, in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, laboratory control samples, initial calibrations, continuing calibrations, and BFB, as well as all laboratory generated spectral libraries and quantitation reports required to generate the data package. The Contractor shall maintain a written reference logbook of tape files to EPA sample number, calibration data, standards, blanks, and laboratory control samples. The logbook should include EPA sample numbers and standard and blank IDs, identified by Case and SDG.

2.8.2 The Contractor is required to retain the GC/MS tapes for 365 days after data submission. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the APO.

2.9 QUALITY ASSURANCE PLAN (QAP)

2.9.1 The Contractor shall prepare a written Quality Assurance Plan (QAP) which describes the procedures that are implemented to achieve the following: maintain data integrity, validity, and useability; ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility; detect problems through data assessment and established corrective action procedures which keep the analytical process reliable; and document all aspects of the measurement process in order to

provide data which are technically sound and legally defensible.

2.9.2 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, SOPs pertaining to each parameter shall be included or referenced as part of the QAP. The QAP must be available during on-site laboratory evaluation and upon written request by the APO.

Table B-1

Codes for Labeling Organic Data

Sample	XXXXX
Duplicate Sample	XXXXXD
Reanalyzed Sample	XXXXXRE
Laboratory Control Sample	SVLCS##
Laboratory Method Blank	SVMBLK##
Field Blank	SVFBLK##
Standards	SVSTD###

SECTION 3

FORM INSTRUCTIONS GUIDE/DATA REPORTING FORMS

3.1 Form Instructions Guide

3.1.1 This section includes specific instructions for the completion of all required forms for semivolatile organics analysis utilizing PUF/XAD-2. Each of the forms is specific to a given function. These instructions are arranged in the following order:

3.1.1.1 General Information and Header Information

3.1.1.2 Cover Page [COVER PAGE - AASV]

3.1.1.3 Analysis Data Sheet [FORM I - AASV]

3.1.1.4 Tentatively Identified Compounds [FORM I - AASV-TIC]

3.1.1.5 Blank Summary [FORM II - AASV]

3.1.1.6 Laboratory Control Sample Data Sheet [FORM III - AASV]

3.1.1.7 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AASV]

3.1.1.8 Initial Calibration Data Sheet [FORM V - AASV]

3.1.1.9 Continuing Calibration Data Sheet [FORM VI - AASV]

3.1.1.10 Internal Standard Area and Retention Times Summary [FORM VII - AASV]

3.1.1.11 Filter/Adsorbent Cartridge Certification [FORM VIII - AASV]

3.1.1.12 Surrogate Recovery [FORM IX - AASV]

3.1.1.13 Analytical Sequence [FORM X - AASV]

3.1.1.14 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.1.1.15 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

3.1.2 General Information and Header Information

3.1.2.1 Values must be reported on the hardcopy forms according to the individual form instructions in this Section. For example,

results for concentrations of semivolatile organic target compounds must be reported to three significant figures if the value is greater than or equal to 10, and to two significant figures for values less than 10.

3.1.2.2 All characters which appear on the data reporting forms presented in the contract must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the EPA APO. The names of the various fields and compounds (i.e., "Lab Code," "Pentachlorophenol") must appear as they do on the forms in the contract.

3.1.2.3 Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low").

3.1.2.4 Six (6) pieces of information are common to the header sections of each data reporting form. They are Lab Name, Lab Code, Contract No., Case No., SDG No., and SAS No. These pieces of information must be entered on every form and must match on every form.

3.1.2.4.1 The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

3.1.2.4.2 The "Lab Code" is an alphabetical abbreviation of up to 6 letters, assigned by the EPA, to identify the laboratory and aid in data processing. This code shall be assigned by the EPA at the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of the EPA. If a change of name or ownership occurs at the laboratory, the Lab Code will remain the same until the contractor is directed by the EPA to use another Lab Code assigned by the EPA.

3.1.2.4.3 The "Contract No." is the number of the EPA contract under which the analyses were performed. In the case of multiple laboratories operating under a corporate-wide contract, the contract number entered shall be that of the corporate contract, regardless of the facility performing the analyses (see Lab Code).

3.1.2.4.4 The "Case No." is the EPA-assigned case number associated with the sample, and reported on the Traffic Report.

3.1.2.4.5 The "SDG No." is the Sample Delivery Group (SDG) number. The SDG No. is the EPA Sample Number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest

sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

3.1.2.4.6 The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services (SAS). If samples are to be analyzed under SAS only and reported on these forms, then enter SAS No. and leave Case No. blank. If samples are analyzed according to the "Routine Analytical Services" (IFB) protocols and have additional SAS requirements, list both Case No. and SAS No. on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. Note that some samples in an SDG may have a SAS No. while others do not.

3.1.2.5 The "EPA Sample No." is the other information common to most of the forms. This number appears either in the upper right corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper right corner, it should be entered on the middle line of the three lines that comprise the box.

3.1.2.5.1 All samples, blanks, and standards shall be identified with an EPA Sample Number. For field samples, the EPA Sample Number is the unique identifying number given in the Traffic Report that accompanied that sample.

3.1.2.5.2 In order to facilitate data assessment, the following sample suffixes must be used:

XXXXX	=	EPA sample number
XXXXXD	=	Duplicate sample
XXXXXRE	=	Reanalyzed sample

3.1.2.5.3 Semivolatile standards prepared on PUF/XAD-2 cartridges shall be identified as SVSTD###, where ### is the concentration in ng (on column) of the semivolatile standards (e.g., 010, 025, 050, 100, and 200).

3.1.2.5.4 As for the blank identifiers, these designations will have to be combined with other information to uniquely identify each standard. Field blanks shall be identified as SVFBLK## and laboratory method blanks shall be identified as SVMBLK##. The "EPA Sample No." must be unique for each blank analysis within an SDG. The laboratory must achieve this by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for semivolatiles-PUF/XAD-2 blanks would be SVMBLK01, SVMBLK02, etc.

3.1.2.5.5 LCSs shall be identified as SVLCS##. The "EPA Sample No." must be unique for each LCS analysis within an SDG. The laboratory must achieve this by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for semivolatiles-PUF/XAD-2 cartridge LCSs would be SVLCS01, SVLCS02, etc.

3.1.2.6 Several other pieces of information are common to many of the Data Reporting Forms. These include Lab Sample ID, Lab File ID, Date Received, etc. Following is a brief description of each of these entries.

3.1.2.6.1 "Lab Sample ID" is an optional laboratory-generated internal identifier. Up to 12 alpha-numeric characters may be reported here. If the contractor does not have a Lab Sample ID, this field may be left blank.

3.1.2.6.2 "Lab File ID" is the laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. Up to 14 alpha-numeric characters may be used here.

3.1.2.6.3 "Date Received" is the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the VTSR). It should be entered as MM/DD/YY.

3.1.2.6.4 "Date Extracted" is the date the sample was extracted by the laboratory. It should be entered as MM/DD/YY.

3.1.2.6.5 "Date Analyzed" should be entered in a similar fashion. The date of sample receipt will be compared with the analysis dates to ensure that contract holding times were not exceeded.

3.1.2.6.6 "Instrument ID" is common to many of the forms, particularly those containing calibration data. The identifier used by the laboratory must include some indication of the manufacturer and/or model of the instrument, and contain additional characters that differentiate between all instrument of the same type in the laboratory.

3.1.2.6.7 "GC Column ID" or "Column ID" is common to various other forms. This field is used to identify the GC column.

3.1.2.7 For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit

to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is odd, and round down if that digit is even.

3.1.2.8 All results must be transcribed to the forms in the raw data with the specified number of decimal places that are described in Exhibit B. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros must be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry
5.9	6.3	5.900
5.99653	6.3	5.997
95.99653	6.3	95.997
995.99653	6.3	995.997
9995.996	6.3	9996.00
99995.9	6.3	99995.9
999995.9	6.3	invalid

NOTE: 6.3 stands for a maximum of six significant figures and up to three decimal places.

3.1.3 Cover Page [COVER PAGE - AASV]

3.1.3.1 This form is used to list all billable samples analyzed within an SDG, and to provide certain analytical information and general comments. It is also the document which is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

3.1.3.2 Under the "EPA Sample No." column, enter up to 7 characters for the EPA sample number (including blanks and duplicates) for each required analysis within the SDG. Duplicates must contain a "D" suffix. These sample numbers must be listed on the form in ascending alphanumeric order using the Extended Binary Coded Decimal Interchange Code convention. Thus, if MAB123A is the lowest (considering both alpha and numeric characters) EPA Sample No. within the SDG, it would be entered in the first EPA Sample No. field. Samples listed below it would be in ascending sequence - MAB124A, MAB124B, MAB125A, MAC111A, MA1111A, MA1111AD, etc.

3.1.3.3 All EPA sample numbers must be listed in ascending alphanumeric order, continuing to the following Cover Page if applicable.

3.1.3.4 Under "Lab Sample ID", a Lab Sample ID (up to 10 characters) may be entered for each associated EPA Sample No. If a Lab Sample ID is entered, it must be entered identically (for each EPA Sample No.) on all associated data.

3.1.3.5 Under "Comments", enter any problems encountered, both technical and administrative, the corrective action taken, and resolution performed for all of the samples in the SDG.

3.1.3.6 Each Cover Page must be signed, in original, by the Laboratory Manager or the Manager's designee, and dated to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.1.4 **Analysis Data Sheet [FORM I - AASV]**

3.1.4.1 This form is used for tabulating and reporting results for analysis of samples on PUF/XAD-2 cartridges for the compounds in a Target Compound List for Semivolatiles as given in Exhibit C.

3.1.4.2 This form is used for reporting the detected concentrations of the target compounds in the field samples, blanks, laboratory control samples, and performance evaluation samples.

3.1.4.3 Complete the header information on each page of Form I-AASV according to the instructions in section 3.1.2.

3.1.4.4 For "Lab Sample ID", enter the lab sample ID for the sample, as listed on the Cover Page. Enter the "Lab File ID", if applicable.

3.1.4.5 For "Cleanup Procedure", enter the cleanup procedure used (Lobar or 610). If no cleanup procedure was used, enter "NONE".

3.1.4.6 For "Date Received," enter the date (formatted month/day/year) the sample was received at the laboratory, as recorded on the Traffic Report (TR) [i.e., the Validated Time of Sample Receipt (VTSR)].

3.1.4.7 For "Date Extracted," enter the date (formatted month/day/year) the sample cartridge was extracted.

3.1.4.8 For "Date Analyzed," enter the date (formatted month/day/year) the sample extract was analyzed.

3.1.4.9 Enter "Instrument ID", "GC Column ID", and "Injection Volume (μL)".

3.1.4.10 Enter the dilution factor. If no dilution was performed, enter "1".

3.1.4.11 Enter the "Air Sample Volume" corrected to standard temperature and pressure (STP) in cubic meter (m^3). If the volume of ambient air sampled is not known, enter "NA".

3.1.4.12 Under the column labeled "Concentration", if the analytical result is greater than or equal to the Contract Required Quantitation Limit (CRQL), report the result. If the result is lower than the CRQL, report the value followed with a "J" under the "Q" column (see section 3.1.15). For example, if the CRQL of an analyte is 5 ng and the detected level is 3 ng, then enter "3" under the "ng" column and "J" under "Q". In this analysis, the CRQL is expressed in "ng" on-column injected and "ng/m^3". Both columns must be filled if the volume of ambient air sampled is known to the laboratory. If the volume of ambient air sampled is not known to the laboratory, then only the levels in ng injected is reported.

3.1.4.13 Analytical results must be reported to two significant figures if the result value is less than 10. Values greater than or equal to 10 shall be reported to three significant figures.

3.1.4.14 The requirement for reporting results to two or three significant figures applies to FORM I-AASV only. Follow the specific instructions for reporting all other results on required forms as described in this Exhibit.

3.1.4.15 For reporting results to the Agency, the following contract specific qualifiers are to be used. The seven qualifiers defined below are not subject to modification by the laboratory. Up to five qualifiers may be reported on Form I-AASV for each compound. The seven EPA-defined qualifiers to be used are as follows:

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero. For example, if the sample quantitation limit is 20 ng, but a concentration of 10 ng is calculated, report it as "10J". The sample quantitation limit must be adjusted for dilution.

N - Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds, where the identification is based on a mass spectral library search. It is applied to all TIC results.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified target compound.

E - This flag identifies compounds whose concentrations exceed the initial calibration range of the instrument for that specific analysis. If one or more compounds have a response that exceed the initial calibration range, the sample or extract must be diluted and reanalyzed according to the specifications in Exhibit D. All such compounds should have the concentration flagged with an "E" on the Form I for the original analysis. The dilution of the sample may cause some compounds identified in the first analysis to be below the calibration range in the second analysis. The results of both analyses shall be reported on separate FORM Is. The FORM I for the diluted sample shall have the "DL" suffix appended to the EPA Sample Number.

X - Other specific flags may be required to properly define the results. If used, they must be fully described, and such description attached to the Sample Data Summary Package and the SDG Narrative. Begin by using "X". If more than one flag is required, use "Y" and "Z" as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "B", and "D" flags for some sample. The laboratory-defined flags are limited to the letters "X", "Y", and "Z".

NOTE: The combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are detected in the sample.

3.1.5 Tentatively Identified Compounds [FORM I - AASV-TIC]

3.1.5.1 FORM I-AASV-TIC is used for reporting the identification and estimated concentration for up to 10 of the non-surrogate, non-internal standard, and non-target compounds.

3.1.5.2 Include a FORM I-AASV-TIC for every sample, performance evaluation sample, and blank analyzed. FORM I-AASV-TIC must be provided for every analysis that requires a FORM I-AASV for target compounds, including required dilutions and reanalyses, even if no TICs are found.

3.1.5.3 Complete the header information according to the header instructions in section 3.1.2.

3.1.5.4 Total the number of TICs found, and enter this number in the "No. of TICs Found". If no TICs were found, enter "0" (zero).

3.1.5.5 Report tentatively identified compounds (TICs) including "CAS RN", "Compound Name", "RT" (retention time), and the estimated concentration (criteria for reporting TICs are given in Exhibit D). Retention time must be reported in minutes and decimal minutes, not seconds or minutes and seconds.

3.1.5.6 If in the opinion of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown.

3.1.5.7 Under the column labeled "Q", enter result qualifiers as identified in section 3.1.4. If additional qualifiers are used, their explicit definitions must be included on the Cover Page in the Comments section.

3.1.6 **Blank Summary [FORM II - AASV]**

3.1.6.1 This form summarizes the samples associated with each field and laboratory method blank analysis. A copy of the appropriate Form II-AASV is required for each blank reported on a Form I-AASV.

3.1.6.2 Complete the header information on Form II-AASV as described in section 3.1.2. The "EPA Sample No." entered in the box at the top of Form II-AASV shall be the same number entered on the Form I-AASV when reporting results for the blank itself.

3.1.6.3 On the numbered lines, enter the EPA sample numbers associated with the blank, along with the other information which identifies the EPA samples. The Cartridge ID for each sample must be provided under the "Cartridge" column, if available.

3.1.7 **Laboratory Control Sample Data Sheet [FORM III - AASV]**

3.1.7.1 Form III-AASV is used to report the recovery of the spiked analytes in the laboratory control samples (LCS).

3.1.7.2 Complete the header information according to the instructions in section 3.1.2.

3.1.7.3 Enter the date and time the LCS was analyzed.

3.1.7.4 In the table under "Spiked," enter the spiked concentration in ng (per injection) of each LCS compound. Under "Reported," enter the concentration obtained in ng (per injection) calculated from the analysis of the LCS. Calculate the percent recovery of each LCS compound to the nearest whole percent and enter in the column under

"% Recovery". At the bottom of the table are the QC limits for LCS percent recoveries. Flag all values outside of the limits with an "*" in the column under the "Q" symbol.

3.1.7.5 Summarize the values outside the QC limits at the lower part of the form.

3.1.8 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AASV]

3.1.8.1 This form is used to report the results of GC/MS instrument performance check (also known as "tuning") and to summarize the date and time of analysis of samples, standards, and blanks associated with each analysis of the instrument performance check solution.

3.1.8.2 Complete the header information as in section 3.1.2. Enter the "Lab File ID" for the injection containing the instrument performance check mixture. Enter the date and time (military time) of injection of the instrument performance check mixture.

3.1.8.3 For each ion listed on the form, enter the percent relative abundance in the right column. Report relative abundances to the number of significant figures given for each ion in the ion abundance criteria column.

3.1.8.4 Under "to m/e 198", all ion abundances are to be normalized to the nominal base peak listed on Form IV-AASV. For some of the ions, determine the percentage of the ion abundance to the specified mass and report under "to specified mass". For example, if the relative ion abundance of mass 443 and mass 442 ions under the "to m/e 198" column are 10 and 50 , respectively, then enter "20" (under the "to specified mass" column) as the ion abundance of mass 443 relative to mass 442.

3.1.8.5 All relative abundances must be reported as a number. If zero, enter "0", not a dash or other non-numeric character.

3.1.8.6 In the lower half of the form, list all samples and standards analyzed under that instrument performance check in <u>chronological order</u>, by time of analysis (in military time). Refer to section 3.1.2 for specific instructions for identifying standards and blanks. Enter "EPA Sample No.", "Lab Sample ID", "Lab File ID", "Date Analyzed", and "Time Analyzed" for all standards, samples, and blanks.

3.1.8.7 The GC/MS instrument performance check must be analyzed again 12 hours from the time of injection of the instrument performance check solution listed at the top of the form. In order to meet these requirements, samples, standards, or blanks must be injected within 12 hours of the injection of the instrument performance check solution.

3.1.9 **Initial Calibration Data Sheet [FORM V - AASV]**

3.1.9.1 Each time the GC/MS system undergoes a five-point calibration to initialize subsequent quantitation of semivolatiles in sample and blank analysis, the laboratory must complete and submit a Form V-AASV.

3.1.9.2 Complete all header information as in section 3.1.2.

3.1.9.3 Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID", "GC Column ID", and "Injection Volume" in microliters.

3.1.9.4 Enter the "EPA Sample No." and "Lab File ID" for each of the five calibration standards.

3.1.9.5 Enter the injection dates and times of each of the calibration standards analyzed under "Date injected" and "Time injected", respectively.

3.1.9.6 Complete the relative response factor (RRF) calculation for the five calibration points, and then calculate and report the average relative response factor ($\overline{RRF}$) and %RSD of the RRF values for each target and surrogate compound in the space provided.

3.1.9.7 At the bottom of the last Form V-AASV, enter the EPA Sample number, Lab Sample ID, and date and time of analysis of the samples and blanks associated with the initial calibration.

3.1.10 **Continuing Calibration Data Sheet [FORM VI - AASV]**

3.1.10.1 Each time the GC/MS system undergoes a continuing calibration to check for the validity of the initial calibration, the laboratory must complete and submit a Form VI-AASV.

3.1.10.2 Complete all header information as in section 3.1.2. Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID", "GC Column ID", and the date(s) of the most recent initial calibration. If the calendar date changes during the calibration procedure, the inclusive dates should be given on Form VI-AASV.

3.1.10.3 Enter the date and time of injection of the continuing calibration standard.

3.1.10.4 Under the column "IC mean RRF", enter the mean relative response factor for each target compound as determined in the most recent valid initial calibration.

3.1.10.5 Complete the relative response factor (RRF) calculation for each target compound in the space provided.

3.1.10.6 Calculate the percent difference (%D) between the continuing calibration RRF and the mean RRF from the most recent valid initial calibration for each target and surrogate compound and enter the values obtained under the "%D" column.

3.1.10.7 At the last Form VI-AASV, enter the EPA Sample number, Lab Sample ID, and date and time of analysis of the samples and blanks associated with the continuing calibration.

3.1.11 **Internal Standard Area and Retention Time Summary [FORM VII - AASV]**

3.1.11.1 This form is used to summarize the peak areas and retention times of the internal standards added to all samples and blanks. The data are used to determine when changes in internal standard responses will adversely affect quantification of target compounds. This form must be completed each time an initial or continuing calibration is performed for each GC/MS system.

3.1.11.2 Complete the header information according to section 3.1.2.

3.1.11.3 Enter the Lab File ID of the 12-hour calibration standard, as well as the date and time of analysis of the calibration standard. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a continuing calibration, a Form VII-AASV shall be completed on the basis of the internal standard areas of the mid level (CAL 3) initial calibration sequence standard. Use the date and time of analysis of this standard, and its Lab File ID and areas in place of those of a continuing calibration standard.

3.1.11.4 From the results of the analysis of the 12-hour calibration standard, enter the area measured for each internal standard and its retention time (in decimal minutes) under the appropriate column. For each internal standard, calculate the upper limit of the area as the area of the particular standard plus 40 percent of its area (i.e., 1.4 times the area in the 12 HOUR STD box), and the lower limit of the area as the area of the internal standard minus 40% of its area (i.e., 0.6 times the area in the 12 HOUR STD box). Report these values in the boxes labeled "UPPER LIMIT" and "LOWER LIMIT", respectively.

3.1.11.5 Calculate the upper limit of the retention time as the retention of the internal standard plus 0.33 minutes (20 seconds), and the lower limit of the retention time as the retention time in the standard minus 0.33 minutes (20 seconds).

3.1.11.6 For each sample and blank under a given 12-hour analytical sequence, enter the EPA Sample Number and the area measured for each internal standard and its retention time. If the internal standard area is outside the upper or lower limits calculated above, flag that area with an asterisk (*) placed in the far right-hand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the retention time of any internal standard that is outside the limits with an asterisk.

3.1.12 Filter/Adsorbent Cartridge Certification [FORM VIII - AASV]

3.1.12.1 This form is used to document the certification of PUF/XAD-2 cartridges prior to use.

3.1.12.2 Complete the header information on each page of Form VIII-AASV according to the instructions in section 3.1.2.

3.1.12.3 Enter "Filter Batch No.", "PUF Batch No.", and "XAD-2 Batch No.", where applicable.

3.1.12.4 Enter the results of the analysis of an unspiked certified clean cartridge. For target compounds that are not detected, enter the CRQL of the compound followed by a "U". If the detected level is less than the CRQL, enter the value followed by a "J".

3.1.12.5 If none of the values for a particular cartridge are more than the corresponding CRQL of any of the target compounds, and if the total level of semivolatiles is not greater than 10 μg/cartridge, then the cartridge is certified.

3.1.13 Surrogate Recovery [FORM IX - AASV]

3.1.13.1 Form IX - AASV is used to report the recoveries of the surrogate compounds added to each PUF/XAD-2 adsorbent cartridge.

3.1.13.2 Complete the header information and enter EPA Sample Numbers as described in section 3.2.1. For each surrogate, report the percent recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.

3.1.13.3 Flag each surrogate recovery outside the QC limits with an asterisk (*). The asterisk must be placed in the last space in each appropriate column, under the "#" symbol. In the far right-hand column, total the number of surrogate recoveries outside the QC limits for each sample (Total Out). If no surrogates were outside the limits, enter "0".

3.1.14 Analytical Sequence [FORM X - AASV]

3.1.14.1 A Form X-AASV is required for each analytical sequence for each GC/MS system used to perform semivolatile analysis on PUF/XAD-2 cartridge samples in an SDG.

3.1.14.2 Complete the header information on each page of Form X-AASV according to the instructions in section 3.1.2.

3.1.14.3 On the numbered lines, enter the EPA sample numbers along with the other information which identifies the samples, blanks, and standards. The first item in the table must be the DFTPP since the 12-hour time period starts at the injection of the instrument performance check standard. Arrange the items in chronological order for each GC/MS system.

3.1.15 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.1.15.1 This form is used to document the receipt and inspection of sample containers and samples. One original of Form AADC-1 is required for each sample shipping container. If the samples in a single sample shipping container must be assigned to more than one Sample Delivery Group, the original Form AADC-1 shall be placed with the deliverables for the Sample Delivery Group of the lowest Arabic number and a copy of Form AADC-1 must be placed with the deliverables for the other Sample Delivery Group(s). The copies should be identified as "copy(ies)," and the location of the original should be noted on the copies.

3.1.15.2 Sign and date the airbill (if present). Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in item 1 on Form AADC-1. Record the custody seal numbers in item 2.

3.1.15.3 Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (i.e., Traffic Reports, Packing Lists), and airbills or airbill stickers in items 3-5 on Form AADC-1. Specify if there is an airbill present or an airbill sticker in item 5 on Form AADC-1. Record the airbill or sticker number, if present.

3.1.15.4 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample (i.e., intact, dent, leaking) and presence of absence of sample tags in items 6 and 7 on Form AADC-1.

3.1.15.5 Review the sample shipping documents and complete the header information described in section 3.1.2. Compare the information recorded on all the documents and samples and circle the appropriate

answer in item 8 on Form AADC-1.

3.1.15.6 If there are no problems observed during receipt, sign and date (include time) Form AADC-1, the chain-of-custody record, and Traffic Report, and write the sample numbers on Form AADC-1. Record the appropriate sample tags and assigned laboratory numbers, if applicable. The log-in date should be recorded at the top of Form AADC-1 and the date and time of sample receipt at the laboratory should be recorded in items 9 and 10. Record the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of Form AADC-1. Sign and date the Sample Transfer block. Cross out unused columns and spaces.

3.1.15.7 If there are problems observed during receipt or if an answer marked with an asterisk (i.e., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.1.16 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

3.1.16.1 This form is used to record the inventory of the SDG File Purge documents and count of documents in the original Sample Data Package which is sent to the Region.

3.1.16.2 Organize all EPA-CSF documents as described in Exhibit B, Section 1. Assemble the documents in the order specified on Form AADC-2, and stamp each page with a consecutive number. (Do not number the AADC-2 form). Inventory the CSF by reviewing the document numbers and recording page numbers ranges in the columns provided in the Form AADC-2. If there are no documents for a specific document type, enter an "NA" in the empty space.

3.1.16.3 Certain laboratory specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review AADC-2 to determine if it is most appropriate to place them under No. 16, 17, 18, or 19. Category 19 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.2 Data Reporting Forms

3.2.1 Cover Page [COVER PAGE - AASV]

3.2.2 Analysis Data Sheet [FORM I - AASV]

3.2.3 Tentatively Identified Compounds [FORM I - AASV-TIC]

3.2.4 Blank Summary [FORM II - AASV]

3.2.5 Laboratory Control Sample Data Sheet [FORM III - AASV]

3.2.6 GC/MS Instrument Performance Check and Mass Calibration [FORM IV - AASV]

3.2.7 Initial Calibration Data Sheet [FORM V - AASV]

3.2.8 Continuing Calibration Data Sheet [FORM VI - AASV]

3.2.9 Internal Standard Area and Retention Times Summary [FORM VII - AASV]

3.2.10 Filter/Adsorbent Cartridge Certification [FORM VIII - AASV]

3.2.11 Surrogate Recovery [FORM IX - AASV]

3.2.12 Analytical Sequence [FORM X - AASV]

3.2.13 Sample Receipt/Log-In Sheet [FORM AADC-1]

3.2.14 Complete SDG File (CSF) Document Inventory Sheet [FORM AADC-2]

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COVER PAGE

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____

	EPA Sample No.	Lab Sample ID
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

Comments: _____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data on floppy diskette has been authorized by the Laboratory Manager's Designee, as verified by the following signature:

Signature: _____ Name: _____
Date: _____ Title: _____

ENVIRONMENTAL PROTECTION AGENCY
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SAMPLE RECEIPT/LOG-IN SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____

ITEM	REMARKS	EPA Sample No.	Sample Tag No.	Assigned Lab No.	Sample Volume	REMARKS: Conditions of Sample Shipment,etc.
1. Custody Seal(s)	Present/Absent/Intact/Broken*					
2. Custody Seal No(s).						
3. Chain-of-Custody records	Present/Absent*					
4. Traffic Reports or	Present/Absent*					
Packing List						
5. Airbill	Sticker/Present/Absent*					
Airbill No(s).						
6. Sample Tags	Present/Absent*					
Sample Tag No(s):	Listed/Not Listed on COC					
7. Sample Condition	Intact/Broken/Leaking*					
8. Do informations on custody						
records, traffic reports, and						
sample tags agree?	Yes/No*					
9. Date Received at Lab:						
10. Time Received at Lab:						
Sample Transfer						
Area #:						
By:						
On:						

* Contact SMO and attach record of resolution.

Received by:
Signature:_____ Log-in Date:_____
Print Name:_____

Reviewed by:
Signature:_____ Date:_____
Logbook No.:_____ Logbook Page No.:_____

FORM AADC-1

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COMPLETE SDG FILE (CSF) DOCUMENT INVENTORY SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____

DOCUMENT	Page Nos. From	Page Nos. To	Please Check Lab	Please Check Reg
1. Cover Sheet (Cover Page – AASV)				
2. Sample Receipt/Log–In Form (FORM AADC–1)				
3. CSF Document Inventory Sheet (FORM AADC–2)				
4. Analysis Data Sheet (FORM I – AASV)				
5. Tentatively Identified Compounds (FORM I – AASV–TIC)				
6. Blank Summary (FORM II – AASV)				
7. Laboratory Control Sample Data Sheet (FORM III – AASV)				
8. GC/MS Tuning with DFTPP (FORM IV –AASV)				
9. Initial Calibration Data Sheet (FORM V – AASV)				
10. Continuing Calibration Data Sheet (FORM VI – AASV)				
11. Internal Standard Area and RT Summary (FORM VII – AASV)				
12. Filter/Cartridge Certification Data Sheet (FORM VIII – AASV)				
13. Analytical Sequence (FORM IX –AASV)				
14. Percent Surrogate Recovery (FORM X – AASV)				
15. EPA Shipping/Receiving Documents				
Airbill (No. of shipments: ___)				
Chain–of–Custody Records				
Sample Tags				
Sample Log–In Sheet (Lab & AADC–1)				
16. Misc. Shipping/Receiving Records (list individual records)				
Telephone Logs				
17. Internal Lab Sample Transfer Records				
18. Internal Original Sample Preparation and Analysis Records				
19. Other Records (describe or list)				

Comments:_____

Completed by (CLP Lab):
Signature:_____ Printed Name/Title:_____ Date:_____
Audited by (EPA):
Signature:_____ Printed Name/Title:_____ Date:_____

FORM AADC–2

ENVIRONMENTAL PROTECTION AGENCY
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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Received:_____ Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Cleanup Procedure:_____ Dilution Factor:_____
Air Sample Volume (STP):_____ m^3 Injection Volume (uL):_____

CAS RN	COMPOUND NAME	Concentration ng	Concentration ng/m^3	Q
83-32-9	Acenaphthene			
208-96-8	Acenaphthylene			
98-86-2	Acetophenone			
116-06-3	Aldicarb			
309-00-2	Aldrin			
62-53-3	Aniline			
120-12-7	Anthracene			
22781-23-3	Bendiocarb			
92-87-5	Benzidine			
56-55-3	Benz[a]anthracene			
50-32-8	Benzo[a]pyrene			
205-99-2	Benzo[b]fluoranthene			
192-97-2	Benzo[e]pyrene			
191-24-2	Benzo[g,h,i]perylene			
207-08-9	Benzo[k]fluoranthene			
100-51-6	Benzyl alcohol			
319-84-6	alpha-BHC			
58-89-9	gamma-BHC (Lindane)			
92-67-1	p-Biphenylamine			
84-74-2	Bis(n-butyl)phthalate			

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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Received:_____ Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Cleanup Procedure:_____ Dilution Factor:_____
Air Sample Volume (STP):_____ m^3 Injection Volume (uL):_____

CAS RN	COMPOUND NAME	Concentration		Q
		ng	ng/m^3	
85-68-7	Butylbenzylphthalate			
133-06-2	Captan			
5103-71-9	alpha-Chlordane			
5103-74-2	gamma-Chlordane			
59-50-7	4-Chloro-3-methylphenol			
106-47-8	4-Chloroaniline			
111-91-1	Bis(2-chloroethoxy)methane			
111-44-4	Bis(2-chloroethyl)ether			
1897-45-6	Chlorothalonil			
2921-88-2	Chlorpyrifos			
218-01-9	Chrysene			
1861-32-1	Dacthal (DCPA)			
72-54-8	4,4'-DDD			
72-55-9	4,4'-DDE			
50-29-3	4,4'-DDT			
333-41-5	Diazinon			
53-70-3	Dibenz[a,h]anthracene			
62-73-7	Dichlorvos (DDVP)			
115-32-2	Dicofol			
60-57-1	Dieldrin			

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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Received:_____ Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Cleanup Procedure:_____ Dilution Factor:_____
Air Sample Volume (STP):_____ m^3 Injection Volume (uL):_____

CAS RN	COMPOUND NAME	Concentration		Q
		ng	ng/m^3	
84-66-2	Diethylphthalate			
105-67-9	2,4-Dimethylphenol			
131-11-3	Dimethylphthalate			
534-52-1	4,6-Dinitro-2-methylphenol			
51-28-5	2,4-Dinitrophenol			
121-14-2	2,4-Dinitrotoluene			
959-98-8	Endosulfan I			
33213-65-9	Endosulfan II			
72-20-8	Endrin			
7421-93-4	Endrin aldehyde			
533-41-5	Endrin ketone			
117-81-7	Bis(2-ethylhexyl)phthalate			
206-44-0	Fluoranthene			
86-73-7	Fluorene			
133-07-3	Folpet			
76-44-8	Heptachlor			
1024-57-3	Heptachlor epoxide			
118-74-1	Hexachlorobenzene			
77-47-4	Hexachlorocyclopentadiene			
67-72-1	Hexachloroethane			

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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Received:_____ Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Cleanup Procedure:_____ Dilution Factor:_____
Air Sample Volume (STP):_____ m^3 Injection Volume (uL):_____

CAS RN	COMPOUND NAME	Concentration		Q
		ng	ng/m^3	
193-39-5	Indeno(1,2,3-c,d)pyrene			
78-59-1	Isophorone			
72-43-5	Methoxychlor			
91-57-6	2-Methylnaphthalene			
95-48-7	2-Methylphenol			
106-44-5	4-Methylphenol			
2385-85-5	Mirex			
91-20-3	Naphthalene			
91-59-8	2-Naphthylamine			
88-74-4	2-Nitroaniline			
100-01-6	3-Nitroaniline			
98-95-3	Nitrobenzene			
92-93-3	4-Nitrodiphenyl			
88-75-5	2-Nitrophenol			
100-02-7	4-Nitrophenol			
117-84-0	Bis(n-octyl)phthalate			
27304-13-8	Oxychlordane			
56-38-2	Parathion			
608-93-5	Pentachlorobenzene			
87-86-5	Pentachlorophenol			

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ANALYSIS DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Received:_____ Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Cleanup Procedure:_____ Dilution Factor:_____
Air Sample Volume (STP):_____ m^3 Injection Volume (uL):_____

CAS RN	COMPOUND NAME	Concentration		Q
		ng	ng/m^3	
52645-53-1	cis/trans-Permethrin			
85-01-8	Phenanthrene			
108-95-2	Phenol			
90-43-7	o-Phenylphenol			
114-26-1	Propoxur			
129-00-0	Pyrene			
10453-86-8	Resmethrin			
299-84-3	Ronnel			
95-95-4	2,4,5-Trichlorophenol			
88-06-2	2,4,6-Trichlorophenol			
27323-18-8	Monochlorobiphenyls			
25512-42-9	Dichlorobiphenyls			
25323-68-6	Trichlorobiphenyls			
26914-33-0	Tetrachlorobiphenyls			
25429-29-2	Pentachlorobiphenyls			
26601-64-9	Hexachlorobiphenyls			
28655-71-2	Heptachlorobiphenyls			
31472-83-0	Octachlorobiphenyls			
53742-07-7	Nonachlorobiphenyls			
2051-24-3	Decachlorobiphenyls			

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ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Received:_____ Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Injection Volume (uL):_____ Dilution Factor:_____
Air Sample Volume (STP):_____ m^3 Cleanup Procedure:_____

	CAS RN	COMPOUND NAME	Concentration ng	Concentration ng/m^3	RT	Q
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						

U. S. ENVIRONMENTAL PROTECTION AGENCY
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BLANK SUMMARY

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Analyzed:_____ Instrument ID:_____
Lab File ID:_____ Time Analyzed:_____ Column ID:_____

THIS BLANK APPLIES TO THE FOLLOWING SAMPLES:

	EPA Sample No.	Laboratory ID			Analysis	
		Sample	File	Cartridge	Date	Time
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						

Comments:_____

U. S. ENVIRONMENTAL PROTECTION AGENCY
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LABORATORY CONTROL SAMPLE DATA SHEET

EPA Sample No.

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Date Analyzed:_____ Instrument ID:_____
Lab File ID:_____ Time Analyzed:_____ Column ID:_____

COMPOUND NAME	Concentration (ng) Spiked	Reported	% Recovery	Q
Bis-(2-chloroethyl)ether				
Benzyl alcohol				
Methylphenol				
Hexachloroethane				
Nitrobenzene				
2,4-Dimethylphenol				
Bis(2-chloroethoxy)methane				
2-Methylnaphthalene				
Hexachlorocyclopentadiene				
3-Nitroaniline				
4-Nitrophenol				
Diethylphthalate				
Fluorene				
Bendiocarb				
Pentachlorophenol				
Diazinon				
Fluoranthene				
Folpet				
Endrin				
Butylbenzylphthalate				
Endrin ketone				
Dicofol				
2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl				
Indeno(1,2,3-cd)pyrene				
Dibenz(a,h)anthracene				

%Recovery QC Limits:

LCS Recovery: _____ outside limits out of _____ total.

FORM III - AASV

ENVIRONMENTAL PROTECTION AGENCY
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GC/MS INSTRUMENT PERFORMANCE CHECK AND MASS CALIBRATION

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
Lab Sample ID:_____ Lab File ID:_____
Date Injected:_____ Time Injected:_____
Instrument ID:_____ GC Column ID:_____
Injection Volume (uL):_____ Mass DFTPP Injected (ng):_____

m/e	ION ABUNDANCE CRITERIA	% Relative Abundance	
		to m/e 198	to specified mass
51	30–80% of mass 198		
68	Less than 2.0% of mass 69		
69	Present		
70	Less than 2.0% of mass 69		
127	25–75% of mass 198		
197	Less than 1.0% of mass 198		
198	Base peak, 100% relative abundance		
199	5–9% of mass 198		
275	10–30% of mass 198		
365	Greater than 0.75% of mass 198		
441	Present, but less than mass 443		
442	40–110% of mass 198		
443	15–24% of mass 442		

THIS TUNE APPLIES TO THE FOLLOWING:

	EPA Sample No.	Lab Sample ID	Lab File ID	Date Analyzed	Time Analyzed
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

ENVIRONMENTAL PROTECTION AGENCY
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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
GC Column ID:_____ Instrument ID:_____
Injection Volume (uL):_____

STANDARD	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5
EPA Sample No.					
LAB FILE ID					
MASS INJECTED (ng)					
DATE INJECTED					
TIME INJECTED					

	Relative Response Factor (RRF)					mean	
COMPOUND NAME	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	RRF	%RSD
Acenaphthene							
Acenaphthylene							
Acetophenone							
Aldicarb							
Aldrin							
Aniline							
Anthracene							
Bendiocarb							
Benzidine							
Benz[a]anthracene							
Benzo[a]pyrene							
Benzo[b]fluoranthene							
Benzo[e]pyrene							
Benzo[g,h,i]perylene							
Benzo[k]fluoranthene							
Benzyl alcohol							
alpha-BHC							
gamma-BHC (Lindane)							
p-Biphenylamine							
Bis(n-butyl)phthalate							
Butylbenzylphthalate							
Captan							
alpha-Chlordane							
gamma-Chlordane							
4-Chloro-3-methylphenol							
4-Chloroaniline							
Bis(2-chloroethoxy)methane							
Bis(2-chloroethyl)ether							
Chlorothalonil							

ENVIRONMENTAL PROTECTION AGENCY
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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
GC Column ID:_____ Instrument ID:_____
Injection Volume (uL):_____

COMPOUND NAME	Relative Response Factor (RRF)					mean RRF	%RSD
	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5		
Chlorpyrifos							
Chrysene							
Dacthal (DCPA)							
4,4'-DDD							
4,4'-DDE							
4,4'-DDT							
Diazinon							
Dibenz[a,h]anthracene							
Dichlorvos (DDVP)							
Dicofol							
Dieldrin							
Diethylphthalate							
2,4-Dimethylphenol							
Dimethylphthalate							
4,6-Dinitro-2-methylphenol							
2,4-Dinitrophenol							
2,4-Dinitrotoluene							
Endosulfan I							
Endosulfan II							
Endrin							
Endrin aldehyde							
Endrin ketone							
Bis(2-ethylhexyl)phthalate							
Fluoranthene							
Fluorene							
Folpet							
Heptachlor							
Heptachlor epoxide							
Hexachlorobenzene							
Hexachlorocyclopentadiene							
Hexachloroethane							
Indeno(1,2,3-c,d)pyrene							
Isophorone							
Methoxychlor							

ENVIRONMENTAL PROTECTION AGENCY
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INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
GC Column ID:_____ Instrument ID:_____
Injection Volume (uL):_____

COMPOUND NAME	Relative Response Factor (RRF) CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	mean RRF	%RSD
Hexachlorocyclopentadiene							
Hexachloroethane							
Indeno(1,2,3-c,d)pyrene							
Isophorone							
Methoxychlor							
2-Methylnaphthalene							
2-Methylphenol							
4-Methylphenol							
Mirex							
Naphthalene							
2-Naphthylamine							
2-Nitroaniline							
3-Nitroaniline							
Nitrobenzene							
4-Nitrodiphenyl							
2-Nitrophenol							
4-Nitrophenol							
Bis(n-octyl)phthalate							
Oxychlordane							
Parathion							
Pentachlorobenzene							
Pentachlorophenol							
cis/trans-Permethrin							
Phenanthrene							
Phenol							
o-Phenylphenol							
Propoxur							
Pyrene							
Resmethrin							
Ronnel							
2,4,5-Trichlorophenol							
2,4,6-Trichlorophenol							

ENVIRONMENTAL PROTECTION AGENCY
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Semivolatile Organics in Air

INITIAL CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____
GC Column ID:_____ Instrument ID:_____
Injection Volume (uL):_____

	Relative Response Factor (RRF)					mean	
COMPOUND NAME	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	RRF	%RSD
PCBs							
Monochlorobiphenyls							
Dichlorobiphenyls							
Trichlorobiphenyls							
Tetrachlorobiphenyls							
Pentachlorobiphenyls							
Hexachlorobiphenyls							
Heptachlorobiphenyls							
Octachlorobiphenyls							
Nonachlorobiphenyls							
Decachlorobiphenyls							
SURROGATES							
Nitrobenzene–d_5							
2–Fluorobiphenyl							
p–Terphenyl–d_{14}							
Phenol–d_5							
2–Fluorophenol							
2,4,6–Tribromophenol							
Anthracene–d_{10}							
Benzo(a)pyrene–d_{12}							

THIS INITIAL CALIBRATION APPLIES TO THE FOLLOWING:

EPA Sample No.	Lab Sample ID	Date Analyzed	Time Analyzed

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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____
EPA Sample No.:_____ Lab File ID:_____
GC Column ID:_____ Instrument ID:_____
Date injected:_____ Time injected:_____
Injection volume (uL):_____ Date of I.C.:_____

COMPOUND NAME	IC mean RRF	CAL 3 RRF	% D
Acenaphthene			
Acenaphthylene			
Acetophenone			
Aldicarb			
Aldrin			
Aniline			
Anthracene			
Bendiocarb			
Benzidine			
Benz[a]anthracene			
Benzo[a]pyrene			
Benzo[b]fluoranthene			
Benzo[e]pyrene			
Benzo[g,h,i]perylene			
Benzo[k]fluoranthene			
Benzyl alcohol			
alpha-BHC			
gamma-BHC (Lindane)			
p-Biphenylamine			
Bis(n-butyl)phthalate			
Butylbenzylphthalate			
Captan			
alpha-Chlordane			
gamma-Chlordane			
4-Chloro-3-methylphenol			
4-Chloroaniline			
Bis(2-chloroethoxy)methane			
Bis(2-chloroethyl)ether			

ENVIRONMENTAL PROTECTION AGENCY
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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____
EPA Sample No.:_____ Lab File ID:_____
GC Column ID:_____ Instrument ID:_____
Date injected:_____ Time injected:_____
Injection volume (uL):_____ Date of I.C.:_____

COMPOUND NAME	IC mean RRF	CAL 3 RRF	% D
Chlorothalonil			
Chlorpyrifos			
Chrysene			
Dacthal (DCPA)			
4,4'-DDD			
4,4'-DDE			
4,4'-DDT			
Diazinon			
Dibenz[a,h]anthracene			
Dichlorvos (DDVP)			
Dicofol			
Dieldrin			
Diethylphthalate			
2,4-Dimethylphenol			
Dimethylphthalate			
4,6-Dinitro-2-methylphenol			
2,4-Dinitrophenol			
2,4-Dinitrotoluene			
Endosulfan I			
Endosulfan II			
Endrin			
Endrin aldehyde			
Endrin ketone			
Bis(2-ethylhexyl)phthalate			
Fluoranthene			
Fluorene			
Folpet			
Heptachlor			

ENVIRONMENTAL PROTECTION AGENCY
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Semivolatile Organics in Air

CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____
EPA Sample No.:_____ Lab File ID:_____
GC Column ID:_____ Instrument ID:_____
Date injected:_____ Time injected:_____
Injection volume (uL):_____ Date of I.C.:_____

COMPOUND NAME	IC mean RRF	CAL 3 RRF	% D
Heptachlor epoxide			
Hexachlorobenzene			
Hexachlorocyclopentadiene			
Hexachloroethane			
Indeno(1,2,3-c,d)pyrene			
Isophorone			
Methoxychlor			
Hexachlorocyclopentadiene			
Hexachloroethane			
Indeno(1,2,3-c,d)pyrene			
Isophorone			
Methoxychlor			
2-Methylnaphthalene			
2-Methylphenol			
4-Methylphenol			
Mirex			
Naphthalene			
2-Naphthylamine			
2-Nitroaniline			
3-Nitroaniline			
Nitrobenzene			
4-Nitrodiphenyl			
2-Nitrophenol			
4-Nitrophenol			
Bis(n-octyl)phthalate			
Oxychlordane			
Parathion			
Pentachlorobenzene			

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Semivolatile Organics in Air

CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____
EPA Sample No.:_____ Lab File ID:_____
GC Column ID:_____ Instrument ID:_____
Date injected:_____ Time injected:_____
Injection volume (uL):_____ Date of I.C._____

COMPOUND NAME	IC mean RRF	CAL 3 RRF	% D
Pentachlorophenol			
cis/trans-Permethrin			
Phenanthrene			
o-Phenylphenol			
Phenol			
Propoxur			
Pyrene			
Resmethrin			
Ronnel			
2,4,5-Trichlorophenol			
2,4,6-Trichlorophenol			
Monochlorobiphenyls			
Dichlorobiphenyls			
Trichlorobiphenyls			
Tetrachlorobiphenyls			
Pentachlorobiphenyls			
Hexachlorobiphenyls			
Heptachlorobiphenyls			
Octachlorobiphenyls			
Nonachlorobiphenyls			
Decachlorobiphenyls			
Nitrobenzene-d_5			
2-Fluorobiphenyl			
p-Terphenyl-d_{14}			
Phenol-d_5			
2-Fluorophenol			
2,4,6-Tribromophenol			
Anthracene-d_{10}			
Benzo(a)pyrene-d_{12}			

ENVIRONMENTAL PROTECTION AGENCY
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CONTINUING CALIBRATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SDG No.:_____ SAS No.:_____
EPA Sample No.:_____ Lab File ID:_____
GC Column ID:_____ Instrument ID:_____
Date injected:_____ Time injected:_____
Injection volume (uL):_____ Date of I.C.:_____

THIS CONTINUING CALIBRATION APPLIES TO THE FOLLOWING:

EPA Sample No.	Lab Sample ID	Date Analyzed	Time Analyzed

U. S. ENVIRONMENTAL PROTECTION AGENCY
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Semivolatile Organics in Ambient Air

INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

	Dichlorobenzene–d_4				Naphthalene–d_8				Acenaphthene–d_{10}			
	Area	#	RT	#	Area	#	RT	#	Area	#	RT	#
12–HOUR STANDARD												
Upper Limit												
Lower Limit												
EPA Sample No.												
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

AREA: Upper Limit: +40% of internal standard area.
Lower Limit: −40% of internal standard area.

RT: Upper Limit: +0.33 minutes of internal standard RT.
Lower Limit: −0.33 minutes of internal standard RT.

All values outside of the QC limits must be followed by an "*" under the "#" column.

U. S. ENVIRONMENTAL PROTECTION AGENCY
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INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

		Phenanthrene-d_{10}			Chrysene-d_{12}			Perylene-d_{12}		
		Area	#	RT	#	Area	#	RT	#	Area	#	RT	#
12-HOUR STANDARD													
Upper Limit													
Lower Limit													
EPA Sample No.													
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													

AREA: Upper Limit: +40% of internal standard area. RT: Upper Limit: +0.33 minutes of internal standard RT.
Lower Limit: −40% of internal standard area. Lower Limit: −0.33 minutes of internal standard RT.

All values outside of the QC limits must be followed by an "*" under the "#" column.

ENVIRONMENTAL PROTECTION AGENCY
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Semivolatile Organics in Air

FILTER/ADSORBENT CARTRIDGE CERTIFICATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Filter Batch No.:_____ PUF Batch No.:_____
Injection Volume:_____(uL) XAD-2 Batch No.:_____

CAS RN	COMPOUND NAME	Concentration ug/assembly
83-32-9	Acenaphthene	
208-96-8	Acenaphthylene	
98-86-2	Acetophenone	
116-06-3	Aldicarb	
309-00-2	Aldrin	
62-53-3	Aniline	
120-12-7	Anthracene	
22781-23-3	Bendiocarb	
92-87-5	Benzidine	
56-55-3	Benz[a]anthracene	
50-32-8	Benzo[a]pyrene	
205-99-2	Benzo[b]fluoranthene	
192-97-2	Benzo[e]pyrene	
191-24-2	Benzo[g,h,i]perylene	
207-08-9	Benzo[k]fluoranthene	
100-51-6	Benzyl alcohol	
319-84-6	alpha-BHC	
58-89-9	gamma-BHC (Lindane)	
92-67-1	p-Biphenylamine	
84-74-2	Bis(n-butyl)phthalate	
85-68-7	Butylbenzylphthalate	
133-06-2	Captan	
5103-71-9	alpha-Chlordane	
5103-74-2	gamma-Chlordane	
59-50-7	4-Chloro-3-methylphenol	
106-47-8	4-Chloroaniline	

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FILTER/ADSORBENT CARTRIDGE CERTIFICATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Filter Batch No.:_____ PUF Batch No.:_____
Injection Volume:_____(uL) XAD-2 Batch No.:_____

CAS RN	COMPOUND NAME	Concentration ug/assembly
111-91-1	Bis(2-chloroethoxy)methane	
111-44-4	Bis(2-chloroethyl)ether	
1897-45-6	Chlorothalonil	
2921-88-2	Chlorpyrifos	
218-01-9	Chrysene	
1861-32-1	Dacthal (DCPA)	
72-54-8	4,4'-DDD	
72-55-9	4,4'-DDE	
50-29-3	4,4'-DDT	
333-41-5	Diazinon	
53-70-3	Dibenz[a,h]anthracene	
62-73-7	Dichlorvos (DDVP)	
115-32-2	Dicofol	
60-57-1	Dieldrin	
84-66-2	Diethylphthalate	
105-67-9	2,4-Dimethylphenol	
131-11-3	Dimethylphthalate	
534-52-1	4,6-Dinitro-2-methylphenol	
51-28-5	2,4-Dinitrophenol	
121-14-2	2,4-Dinitrotoluene	
959-98-8	Endosulfan I	
33213-65-9	Endosulfan II	
72-20-8	Endrin	
7421-93-4	Endrin aldehyde	
533-41-5	Endrin ketone	
117-81-7	Bis(2-ethylhexyl)phthalate	

ENVIRONMENTAL PROTECTION AGENCY
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FILTER/ADSORBENT CARTRIDGE CERTIFICATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Filter Batch No.:_____ PUF Batch No.:_____
Injection Volume:_____(uL) XAD-2 Batch No.:_____

CAS RN	COMPOUND NAME	Concentration ug/assembly
206-44-0	Fluoranthene	
86-73-7	Fluorene	
133-07-3	Folpet	
76-44-8	Heptachlor	
1024-57-3	Heptachlor epoxide	
118-74-1	Hexachlorobenzene	
77-47-4	Hexachlorocyclopentadiene	
67-72-1	Hexachloroethane	
193-39-5	Indeno(1,2,3-c,d)pyrene	
78-59-1	Isophorone	
72-43-5	Methoxychlor	
91-57-6	2-Methylnaphthalene	
95-48-7	2-Methylphenol	
106-44-5	4-Methylphenol	
2385-85-5	Mirex	
91-20-3	Naphthalene	
91-59-8	2-Naphthylamine	
88-74-4	2-Nitroaniline	
100-01-6	3-Nitroaniline	
98-95-3	Nitrobenzene	
92-93-3	4-Nitrodiphenyl	
88-75-5	2-Nitrophenol	
100-02-7	4-Nitrophenol	
117-84-0	Bis(n-octyl)phthalate	
27304-13-8	Oxychlordane	
56-38-2	Parathion	

ENVIRONMENTAL PROTECTION AGENCY
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Semivolatile Organics in Air

FILTER/ADSORBENT CARTRIDGE CERTIFICATION DATA SHEET

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
Lab Sample ID:_____ Lab File ID:_____
SDG No.:_____ SAS No.:_____
Date Extracted:_____ Date Analyzed:_____
Instrument ID:_____ GC Column ID:_____
Filter Batch No.:_____ PUF Batch No.:_____
Injection Volume:_____(uL) XAD-2 Batch No.:_____

CAS RN	COMPOUND NAME	Concentration ug/assembly
608-93-5	Pentachlorobenzene	
87-86-5	Pentachlorophenol	
52645-53-1	cis/trans-Permethrin	
85-01-8	Phenanthrene	
108-95-2	Phenol	
90-43-7	o-Phenylphenol	
114-26-1	Propoxur	
129-00-0	Pyrene	
10453-86-8	Resmethrin	
299-84-3	Ronnel	
95-95-4	2,4,5-Trichlorophenol	
88-06-2	2,4,6-Trichlorophenol	
	PCBs	
27323-18-8	Monochlorobiphenyls	
25512-42-9	Dichlorobiphenyls	
25323-68-6	Trichlorobiphenyls	
26914-33-0	Tetrachlorobiphenyls	
25429-29-2	Pentachlorobiphenyls	
26601-64-9	Hexachlorobiphenyls	
28655-71-2	Heptachlorobiphenyls	
31472-83-0	Octachlorobiphenyls	
53742-07-7	Nonachlorobiphenyls	
2051-24-3	Decachlorobiphenyls	

U. S. ENVIRONMENTAL PROTECTION AGENCY
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SURROGATE RECOVERY

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

		Surrogate Percent Recovery					Total
	EPA Sample No.	Nitrobenzene-d_6	#	2-Fluorobiphenyl	#	p-Terphenyl-d_{14}	#	Out
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

%Recovery QC Limits: 25-150%
Values outside of QC limits are flagged with a "*" under the "#" column

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SURROGATE RECOVERY

Lab Name:_____ Contract No.:_____
Lab Code:_____ Case No.:_____
SAS No.:_____ SDG No.:_____

	EPA Sample No.	Surrogate Percent Recovery					Total out
		Anthracene-d_{10} #	Benzo(a)pyrene-d_{12} #	Tribromophenol #	2-Fluorophenol #	Phenol-d_5 #	
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							

%Recovery QC Limits: 25-150%, except for 2-Fluorophenol, 10-150%.
Values outside of QC limits are flagged with a "*" under the "#" column

FORM IX - AASV-2

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Semivolatile Organics in Ambient Air

ANALYTICAL SEQUENCE

Lab Name: _____ Contract No.: _____
Lab Code: _____ Case No.: _____
SAS No.: _____ SDG No.: _____
Instrument ID: _____ GC Column ID: _____

THE FOLLOWING IS THE ANALYTICAL SEQUENCE ARRANGED IN CHRONOLOGICAL ORDER:

	EPA Sample No.	Lab Sample ID	Analysis Time	Analysis Date
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
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21				
22				
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24				
25				
26				
27				
28				
29				
30				

FORM X – AASV

EXHIBIT C

TARGET COMPOUND LISTS

EXHIBIT C

SEMIVOLATILES IN AMBIENT AIR
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

I. PAHs, PESTICIDES, AND OTHER SEMIVOLATILES

Target Compound	CAS RN	CRQL	
		On-column Injection, ng	ng/m^3
Acenaphthene	83-32-9	10	37
Acenaphthylene	208-96-8	10	37
Acetophenone	98-86-2	10	37
Aldicarb	116-06-3	40	146
Aldrin	309-00-2	40	146
Aniline	62-53-3	20	73
Anthracene	120-12-7	10	37
Bendiocarb	22781-23-3	50	183
Benzidine	92-87-5	20	73
Benzo(a)anthracene	56-55-3	10	37
Benzo(a)Pyrene	50-32-8	10	37
Benzo(b)fluoranthene	205-99-2	10	37
Benzo(e)pyrene	192-97-2	10	37
Benzo(g,h,i)perylene	191-24-2	10	37
Benzo(k)fluoranthene	207-08-9	10	37
Benzyl alcohol	100-51-6	10	37
alpha-BHC	319-84-6	40	146
gamma-BHC (Lindane)	58-89-9	40	146
p-Biphenylamine	92-67-1	50	183
Bis(n-butyl)phthalate	84-74-2	10	37
Butylbenzylphthalate	85-68-7	10	37
Captan	133-06-2	50	183
alpha-Chlordane	5103-71-9	40	146
gamma-Chlordane	5103-74-2	40	146
4-Chloro-3-methylphenol	59-50-7	50	183
4-Chloroaniline	106-47-8	20	73
Bis(2-chloroethoxy)methane	111-91-1	10	183
Bis(2-chloroethyl)ether	111-44-4	10	37
Chlorothalonil	1897-45-6	50	183
Chlorpyrifos	2921-88-2	50	183
Chrysene	218-01-9	10	37
Dacthal (DCPA)	1861-32-1	50	183
4,4'-DDD	72-54-8	40	146
4,4'-DDE	72-55-9	40	146
4,4'-DDT	50-29-3	40	146

TABLE 3

SEMIVOLATILES IN AMBIENT AIR
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(continued)

I. PAHs, PESTICIDES, AND OTHER SEMIVOLATILES (continued)

Target Compound	CAS RN	CRQL On-column Injection, ng	CRQL ng/m³
Diazinon	333-41-5	50	183
Dibenzo(a,h)anthracene	53-70-3	10	37
Dichlorvos (DDVP)	62-73-7	50	183
Dicofol	115-32-2	50	183
Dieldrin	60-57-1	40	146
Diethyl Phthalate	84-66-2	20	73
2,4-Dimethyl phenol	105-67-9	20	73
Dimethylphthalate	131-11-3	10	37
4,6-Dinitro-2-methylphenol	534-52-1	50	183
2,4-Dinitrophenol	51-28-5	20	73
2,4-Dinitrotoluene	121-14-2	10	37
Endosulfan I	959-98-8	10	37
Endosulfan II	33213-65-9	10	37
Endrin	72-20-8	10	37
Endrin Aldehyde	7421-93-4	10	37
Endrin ketone	533-41-5	50	183
Bis(2-ethylhexyl)phthalate	117-81-7	10	37
Fluoranthene	206-44-0	10	37
Fluorene	86-73-7	10	37
Folpet	133-07-3	50	183
Heptachlor	76-44-8	40	146
Heptachlor Epoxide	1024-57-3	40	146
Hexachlorobenzene	118-74-1	10	37
Hexachlorocyclopentadiene	77-47-4	10	37
Hexachloroethane	67-72-1	10	37
Indeno(1,2,3-c,d)pyrene	193-39-5	10	37
Isophorone	78-59-1	10	37
Methoxychlor	72-43-5	40	146
2-Methylnaphthalene	91-57-6	10	37
2-Methylphenol	95-48-7	50	183
4-Methylphenol	106-44-5	50	183
Mirex	2385-85-5	40	146
Naphthalene	91-20-3	10	37
2-Naphthylamine	91-59-8	50	183

SEMIVOLATILES IN AMBIENT AIR
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(continued)

I. **PAHs, PESTICIDES, AND OTHER SEMIVOLATILES** (continued)

Target Compound	CAS RN	CRQL On-column Injection, ng	CRQL ng/m^3
2-Nitroaniline	88-74-4	20	73
3-Nitroaniline	100-01-6	20	73
Nitrobenzene	98-95-3	10	37
4-Nitrodiphenyl	92-93-3	50	183
2-Nitrophenol	88-75-5	20	73
4-Nitrophenol	100-02-7	50	183
Bis(n-octyl)phthalate	117-84-0	10	37
Oxychlordane	27304-13-8	50	183
Parathion	56-38-2	40	146
Pentachlorobenzene	608-93-5	50	183
Pentachlorophenol	87-86-5	50	183
cis/trans-Permethrin	52645-53-1	50	183
Phenanthrene	85-01-8	10	37
Phenol	108-95-2	50	183
o-Phenylphenol	90-43-7	50	183
Propoxur	114-26-1	50	183
Pyrene	129-00-0	10	37
Resmethrin	10453-86-8	50	183
Ronnel	299-84-3	50	183
2,4,6-Trichlorophenol	88-06-2	50	183
2,4,5-Trichlorophenol	95-95-4	50	183

SEMIVOLATILES IN AMBIENT AIR
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(continued)

II. PCBs (Congeners)

Target Compound	CAS RN	CRQL	
		On-column Injection, ng	ng/m^3
Monochlorobiphenyls	27323-18-8	40	146
Dichlorobiphenyls	25512-42-9	40	146
Trichlorobiphenyls	25323-68-6	40	146
Tetrachlorobiphenyls	26914-33-0	40	146
Pentachlorobiphenyls	25429-29-2	40	146
Hexachlorobiphenyls	26601-64-9	40	146
Heptachlorobiphenyls	28655-71-2	80	293
Octachlorobiphenyls	31472-83-0	80	293
Nonachlorobiphenyls	53742-07-7	100	366
Decachlorobiphenyl	2051-24-3	100	366

EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF SEMIVOLATILE ORGANICS COLLECTED ON PUF/XAD-2 AND ANALYZED BY GAS CHROMATOGRAPHY AND MASS SPECTROMETRY (GC/MS)

January, 1992

EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF SEMIVOLATILE ORGANICS COLLECTED ON PUF/XAD-2 AND ANALYZED BY GAS CHROMATOGRAPHY AND MASS SPECTROMETRY (GC/MS)

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EXHIBIT D

ANALYTICAL METHOD FOR THE DETERMINATION OF SEMIVOLATILE ORGANICS COLLECTED ON PUF/XAD-2 AND ANALYZED BY GAS CHROMATOGRAPHY AND MASS SPECTROMETRY (GC/MS)

1 INTRODUCTION

1.1 SCOPE AND APPLICATION

1.1.1 Polynuclear aromatic hydrocarbons (PAHs), pesticides, polychlorinated biphenyls (PCBs), and other semivolatile compounds (SVOCs) have received increased attention in recent years in air pollution studies because some of these compounds are highly carcinogenic or mutagenic. In particular, benzo[a]pyrene (B[a]P), 4,4'-DDD, 4,4'-DDT, and PCBs have all been identified as being highly carcinogenic.

1.1.2 The analytical method that follows is designed to analyze samples containing the compounds listed on the Target Compound List in Exhibit C.

1.1.3 The analysis technique is based on modifications of EPA Test Methods 610 and 625, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater; Methods 8000, 8270, and 8310, Test Methods for Evaluation of Solid Waste (SW-846); the USEPA Contract Laboratory Program Statement of Work for Low Concentration Water for Organics Analysis; and EPA Method 680, Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry (GC/MS).

1.1.4 Three surrogate compounds (pre-sampling) are added to each PUF/XAD-2 cartridge brought to the field. Five additional surrogate compounds (post-sampling) are spiked on the cartridges received from the field just prior to extraction. For the laboratory method blank (LMB), laboratory control sample (LCS), and performance evaluation (PE) samples, all of the eight surrogate compounds are spiked onto the cartridges just prior to extraction, since these cartridges are not sent to the field. Internal standards are added to each blank and sample extract before GC/MS analysis. Each concentration measurement is based on an integrated ion abundance of one characteristic ion. Analytes are identified as individual compounds, and a concentration is calculated by relating the MS response of each compound to the MS response of the corresponding internal standard. Table D/SV-3 provides the internal standard assignments for each target and surrogate compound.

1.1.5 An interfaced data system (DS) to control data acquisition and to store, retrieve, and manipulate mass spectral data is essential.

1.1.6 Applicability of the Method to PCBs

1.1.6.1 This method is applicable to samples containing PCBs as single congeners. PCBs are identified and measured as isomer groups (i.e., by level of chlorination) by GC/MS using a special software.

1.1.6.2 The existence of 209 possible PCB congeners makes the listing of the Chemical Abstracts Service Registry Number (CAS RN) for each potential method analyte impractical.

1.1.6.3 A concentration is measured for each PCB isomer group, and total PCB concentration for each sample extract is obtained by summing isomer group concentrations. Nine selected PCB congeners are used as calibration standards and one internal standard, chrysene-d_{12}, is used to calibrate MS response to PCBs. Because PCBs are identified and measured as isomer groups, the non-specific CAS RN for each level of chlorination is used to describe method analytes.

1.1.7 Detection Limits

Detection limits vary among method analytes and with sample matrix, sample preparation procedures, condition of the GC/MS system, type of data acquisition, and individual samples. Based on numerous analyses of calibration solutions using one instrument over a period of approximately six months, the following comments are provided to assist the analyst:

1.1.7.1 Pesticide analytes other than endosulfans I and II can be identified and accurately measured when the injected aliquot contains 2 ng of each analyte. The endosulfans require about 4 ng each.

1.1.7.2 Detection limits for individual PCB congeners increase with increasing number of chlorine atoms, with the detection limit for decachlorobiphenyl being about 5 to 10 times higher than that of a monochlorobiphenyl. A monochlorobiphenyl can be identified and accurately measured when the injected extract aliquot contains 1 ng and full scan data acquisition is used.

1.1.7.3 The detection limit for total PCBs will depend on the number of individual PCB congeners present.

1.1.8 Safety

1.1.8.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for

maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the analyst.

1.1.8.2 Care must be exercised when working with these substances. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use. The user should be thoroughly familiar with the chemical and physical properties of targeted substances.

1.1.8.3 Treat all analytes as carcinogens. Neat compounds should be weighed in a glove box. Spent samples and unused standards are toxic wastes and should be disposed according to regulations. Regularly check counter tops and equipment with "black light" for fluorescence as an indicator of semivolatile contamination.

1.1.8.4 Precautions must be taken with diethyl ether because of its extreme fire hazard and the possibility that peroxides may form producing an explosive mixture should the solution be allowed to evaporate to dryness. It is the laboratory's duty to instruct all its personnel on the proper use and handling of this solvent. Standard safety practices for handling ethers must be implemented.

1.2 SUMMARY OF METHOD

1.2.1 Prior to field use, the filter and adsorbent cartridge (PUF/XAD-2/PUF) are cleaned in solvents and vacuum-dried. The cartridge is then spiked with a minimum of three surrogate compounds. The filter and spiked adsorbent cartridge are stored in screw-capped jars wrapped in aluminum foil prior to installation on the sampler.

1.2.2 In the field, approximately 273 m^3 of ambient air is drawn through the filter and adsorbent cartridge where the analytes of interest are retained. At the end of the specified sampling period, the amount of air sampled is recorded, and the filter and cartridge are placed in an appropriately labeled container and shipped along with blank filter and adsorbent cartridges to the analytical laboratory for analysis.

1.2.3 Each pair of the filters and PUF/XAD-2 adsorbent cartridge are extracted together by Soxhlet extraction with diethyl ether/hexane solvent (10 percent v/v). The extract is concentrated using a Kuderna-Danish (K-D) evaporator and further concentrated to 1.0 mL by micro-Snyder column or by nitrogen blowdown technique. Prior to analysis by GC/MS, internal

standards are added to the concentrate. (If applicable, the concentrated extract is screened for levels of semivolatiles utilizing a GC with a flame ionization detector (FID) or an electron capture detector (ECD).)

1.2.4 For final identification and quantification, 1 µL of the extract is injected onto a GC with a capillary column and temperature-programmed to separate the compounds, which are then detected with a mass spectrometer (MS) in the full scan data acquisition mode.

NOTE: This method is based upon full scan data acquisition. All concentrations are based upon the use of 1-µL injections. If different volumes are used, the concentrations must be appropriately adjusted.

1.2.5 Target compounds are identified in the samples by analyzing standards under the same analytical conditions used as the samples and comparing resultant mass spectra and GC retention times. A relative response factor is established for each target compound during the initial and continuing calibrations by comparing the MS response for the primary ion produced by the compound to the MS response for the primary ion produced by an internal standard. Each identified target compound in a sample is quantified by comparing the responses for the target compound and the internal standard, while taking into account the relative response factor from the most recent calibration, the initial and final sample volume, and any sample dilutions.

1.2.6 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library. Non-target compounds are quantified by comparing the MS response for the non-target compound peaks to the MS response produced by the nearest internal standard. A response factor of 1 is assumed.

1.3 INTERFERENCES AND LIMITATIONS

1.3.1 Contaminants in solvents, reagents, glassware and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). Laboratory method blanks are analyzed with each analytical sequence to demonstrate that these materials are free of interferences under the analytical conditions used for samples. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source.

1.3.2 To minimize interferences, glassware should be meticulously cleaned. As soon as possible after use, rinse glassware with the last solvent used. Then wash with detergent in hot water and rinse with tap water followed by distilled water. Drain dry and heat in a muffle furnace at 450°C for a few hours (volumetric glassware should not be heated in a

muffle furnace). After cooling, store glassware inverted or covered with aluminum foil. Before using, rinse each piece with an appropriate solvent.

NOTE: Some thermally stable materials such as PCBs may not be eliminated by this treatment. Solvent rinses with acetone and pesticide-quality hexane may be substituted for the muffle furnace.

1.3.3 For both pesticides and PCBs, interference can be caused by the presence of much greater quantities of other sample components that overload the capillary column; additional sample extract preparation procedures must then be used to eliminate interferences. Capillary column GC retention times and the compound-specific characteristics of mass spectra eliminate many interferences that formerly were of concern with pesticide/PCB determinations by electron capture detection. The approach and identification criteria used in this method for PCBs eliminate interference by most chlorinated compounds other than PCBs. With the isomer group approach, coeluting PCBs that contain the same number of chlorines are identified and measured together. Therefore, coeluting PCBs are a problem only if they contain a different number of chlorine atoms.

1.4 DEFINITIONS

NOTE: Definitions used in this document and in any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, D1605-60, E260, and E255. All abbreviations and symbols are defined within this document at the point of use. A detailed glossary of terms can be found in Exhibit G.

1.4.1 CAL: Calibration standards are defined in Table D/SV-1 in which 5 levels of calibration are defined; CAL 1, CAL 2, CAL 3, CAL 4, and CAL 5. CAL 1 is the lowest concentration and CAL 5 is the highest concentration. CAL 3, which is the mid level standard, is designated as the solution to be used for continuing calibrations.

1.4.2 Continuing calibration standard: A solution of method analytes used to evaluate the mass spectrometer response over a period of time. A check of the initial calibration is performed once each 12-hour period. The continuing calibration standard is CAL 3 of the initial calibration curve.

1.4.3 GC response (A_x): The peak area or height of analyte, x.

1.4.4 Internal standard (IS): A compound added to a sample extract in known amounts and used to calibrate concentration measurements of other compounds that are sample components. The internal standard must be a compound that is not a sample component.

1.4.5 Laboratory method blank (LMB): The concentrate from the extraction

of a clean, certified filter/adsorbent cartridge solution that is treated as a sample. The filter/adsorbent cartridge is carried through the same analytical procedure as a field sample. The surrogates are spiked onto the cartridge bed prior to extraction. The purpose of the LMB is to monitor for possible laboratory contamination.

1.4.6 Mass spectral interference: Defined as the inability to detect the analyte quantification ion due to presence of high levels of mass spectral "noise" at the same mass.

1.4.7 Method detection limit (MDL): A statistically determined value indicating the minimum concentration of an analyte that can be identified and measured in a sample matrix with 99 percent confidence that the analyte concentration is greater than zero. This value varies with the precision of the replicate measurements used for the calculation. (See 40CFR 136 APP.B)

1.4.8 PAH: Polynuclear aromatic hydrocarbon.

1.4.9 PCB: Polychlorinated biphenyl.

1.4.10 Performance evaluation (PE) sample: A sample containing known concentrations of method analytes that has been analyzed to statistically determine the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte identification and concentrations are unknown to the analyst. PE samples are supplied by the Agency.

1.4.11 PUF: Polyurethane foam used as an adsorbent and as a support for XAD-2.

1.4.12 Retention time window: Retention time is determined for each analyte of interest as the time from injection to elution of a specific chemical from a chromatographic column. The window is determined by five injections of a initial calibration standard over a 24-hour period as plus or minus three times the standard deviation of the absolute retention time for that analyte. For this document, the window is ±0.06 RRT units of the continuing or mid level calibration standard RRT for each target and surrogate compound.

1.4.13 Selected Ion Current Profile (SICP): A plot of ion abundances of the ions of an analyte produced by the mass spectrometer. SICP is used interchangeably with EICP, extracted ion current profile.

1.4.14 Stock standard solution: A solution used to prepare calibration standards. Normally, this solution will be at a concentration which is easily diluted to a level which can be injected into the GC.

1.4.15 Surrogate compound: A compound not expected to be found in the

sample that is added to each sample prior to field deployment and before extraction, and is measured with the same procedures used to measure sample components. The purpose of a surrogate compound is to monitor method performance with each sample.

1.4.16 VTSR: Validated Time of Sample Receipt is the time the sample is logged-in on the Chain-of-Custody Log-In Sheet by the laboratory.

1.4.17 Working standard solution: A solution which is prepared as a calibration standard. Normally, this solution will be injected into the GC without additional dilution.

1.4.18 XAD-2: A resin used as an adsorbent for semivolatile compounds.

2 SAMPLE STORAGE AND HOLDING TIMES

2.1 SAMPLE IDENTIFICATION

2.1.1 In the field, the samples are packed in dry ice to ensure that the glass sample container containing the filter and adsorbent cartridge are properly chilled, and shipped to the designated laboratory.

2.1.2 The samples are logged in the laboratory logbook or appropriate data tracking system according to sample location, filter and adsorbent cartridge number identification, and total air volume sampled, corrected to standard temperature and pressure (STP). The time the samples are logged-in on the Chain of Custody Log-In Sheet at the laboratory is the validated time of sample receipt (VTSR).

2.1.3 If the time between sample receipt and extraction is greater than 24 hours, then the samples must be kept refrigerated. Minimize exposure of samples to fluorescent light. All samples must be extracted within five (5) days of VTSR.

2.2 SAMPLE/SAMPLE EXTRACT STORAGE

2.2.1 The samples must be contained in air-tight glass containers, protected from light, and refrigerated at 4°C (±2°C) from the time of receipt until extraction. Unused samples must be disposed of or regenerated in a manner that complies with all applicable regulations.

2.2.2 Sample extracts must be protected from light and stored at 4°C (±2°C) until 30 days after delivery of a complete data package to the Agency.

2.2.3 Samples and sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

2.2.4 Samples, sample extracts, and standards must be stored separately.

2.3 CONTRACT REQUIRED HOLDING TIMES

2.3.1 The extraction shall be started within 5 days of the VTSR.

2.3.2 Extracts must be analyzed within 25 days following the start of the extraction.

3 CARTRIDGE PREPARATION AND CERTIFICATION

3.1 SUMMARY OF METHOD

This section discusses pertinent information regarding the preparation and cleaning of the filter, adsorbents, and filter/adsorbent cartridge assembly. The separate batches of filters and adsorbents are extracted with the appropriate solvent. At least one PUF/XAD-2/PUF cartridge assembly and one filter from each batch, or 10 percent of the batch, whichever is greater, must be extracted and certified before the batch is considered for field use. Prior to field sampling, the cartridges are spiked with surrogate compounds.

3.2 APPARATUS AND MATERIALS

3.2.1 Acid-washed Pallflex filter: 4 inch Pallflex filter, General Metal Works, Inc., Cat. No. GMW QMA-4, 145 South Miami Ave., Village of Cleves, OH, 45002, 800-543-7412, or Supelco Inc., Cat. No. 1-62, Supelco Park, Bellefonte, PA, 16823-0048, or equivalent.

3.2.2 Polyurethane foam (PUF): Two separates plugs, 6.5 cm by 2.5 cm and 6.5 cm by 5 cm. These can be manufactured from a 3 inch thick sheet stock, polyether type (density 0.022 g/cm^3) used in furniture upholstering, General Metal Works, Inc., Cat. No. PS-1-16, 145 South Miami Ave., Village of Cleves, OH, 45002, 800-543-7412, or Supelco Inc., Cat. No. 1-63, Supelco Park, Bellefonte, PA, 16823-0048, or equivalent. The two separate plugs are also commercially available from General Metal Works.

3.2.3 XAD-2 resin: Supelco Inc., Cat. No. 2-02-79, Supelco Park, Bellefonte, PA, 16823-0048, or equivalent.

3.2.4 Aluminum foil: baked in an oven overnight at 500°C after rinsing with hexane.

3.2.5 Soxhlet extractors: Capable of extracting GMW Model PS-1 filter and adsorbent cartridges (2.3" x 5" length), 1000-mL flask, and condenser.

3.2.6 Minivials: 2-mL, borosilicate glass, with conical reservoir and screw caps lined with Teflon®-faced silicone septa, and a vial holder.

3.2.7 Glass vials: 40-mL.

3.2.8 Erlenmeyer flasks: 50-mL.

3.2.9 Spatulas and spoons: Teflon®-coated stainless steel.

3.2.10 Kuderna-Danish (K/D) apparatus: 500-mL evaporation flask (Kontes K-570001-500, or equivalent), 10-mL graduated concentrator tubes (Kontes K-570050-1025, or equivalent) with ground-glass stoppers, and 3-ball

macro-Snyder Column (Kontes K-5700010500, K-50300-0121, and K-569001-219, or equivalent).

3.2.11 Adsorption columns for column chromatography: 1 cm x 10 cm with stands.

3.2.12 White cotton gloves: For handling cartridges and filters.

3.2.13 Glove box: For working with extremely toxic standards and reagents, with explosion-proof hood for venting fumes from solvents, reagents, etc.

3.2.14 Vacuum oven: Vacuum drying oven system capable of maintaining a vacuum at 240 torr (flushed with nitrogen) overnight.

3.2.15 Concentrator tubes and a nitrogen evaporation apparatus: with variable flow rate.

3.2.16 Laboratory refrigerator.

3.2.17 Boiling chips: Solvent-extracted 10/40 mesh silicon carbide, or equivalent.

3.2.18 Water bath: Heated, with concentric ring cover, capable of ±5°C temperature control.

3.2.19 Vortex evaporator (optional).

3.2.20 Teflon® sleeves for the ground-glass joints on the Soxhlet extraction apparatus.

3.3 REAGENTS

3.3.1 Hexane: chromatographic grade, glass distilled.

3.3.2 Diethyl ether: chromatographic grade, glass distilled.

3.3.3 Methylene chloride: Chromatographic grade, glass-distilled.

3.3.4 Sodium sulfate: Anhydrous (ACS), granular (purified by washing with methylene chloride followed by heating at 400°C for 4 hours in a shallow tray).

3.3.5 Nitrogen: High purity grade.

3.4 PROCEDURES FOR PREPARATION OF FILTER, ADSORBENT, AND CARTRIDGE

3.4.1 Glass Fiber Filter Preparation

3.4.1.1 The quartz fiber filters are baked at 600°C for five hours

before use. To ensure acceptable filters, they are extracted with methylene chloride in a Soxhlet apparatus, similar to the cleaning of the XAD-2 resin. The extract may be analyzed for the purpose of determining cleanliness prior to certification.

3.4.1.2 The extracted filters are set aside in a clean container prior to combined extraction (certification) with the PUF/XAD-2/PUF glass cartridge assembly.

3.4.2 **XAD-2 Adsorbent Preparation**

3.4.2.1 For initial cleanup of the XAD-2 resin, a batch of XAD-2 (approximately 50-60 grams) is placed in a Soxhlet apparatus and extracted with methylene chloride for 16 hours at approximately 4 cycles per hour.

3.4.2.2 At the end of the initial Soxhlet extraction, the spent methylene chloride is discarded and replaced with fresh reagent. The XAD-2 resin is once again extracted for 16 hours at approximately 4 cycles per hour.

3.4.2.3 The XAD-2 resin is removed from the Soxhlet apparatus, placed in a vacuum oven connected to an ultra-pure nitrogen gas stream and dried at room temperature for approximately 2-4 hours (until no solvent odor is detected).

NOTE: Alternatively, a 600 g batch of XAD-2 resin is extracted with methylene chloride for 16 hours. After extraction, the resin is transferred to a clean drying column. Then the resin is dried with high-purity nitrogen using Teflon® tubing from the nitrogen cylinder with a charcoal tube in the line. In an alternative method of drying, the XAD-2 resin is placed in a Pyrex® column (10 cm x 60 cm), allowing sufficient space for fluidizing. The column is wrapped with heat tape, maintained at 40°C, during the drying process. High purity air, scrubbed through a charcoal trap, is forced through the resin bed, fluidizing the bed while generating a minimum load at the exit of the column.

3.4.2.4 The extract from the Soxhlet extraction procedure from each batch may be analyzed for the purpose of determining initial cleanliness prior to certification.

3.4.3 **PUF Adsorbent Preparation**

3.4.3.1 The PUF adsorbent is a polyether-type polyurethane foam (density 0.0225 g/cm^3).

3.4.3.2 The adsorbent assembly is composed of two inserts, as illustrated in Figure D/SV-2. The bottom PUF is 6.5-cm by 2.5-cm, while the top plug is 6.5-cm by 5.0-cm. The PUF inserts are cut from

sheet stock and should fit with slight compression in the glass cartridge, supported by the wire screen. The two PUF plugs (i.e., 1-inch and 2-inch deep) are also commercially available as pre-cut separate pieces.

3.4.3.3 For initial cleanup, the PUF plugs are placed in a Soxhlet apparatus and extracted with acetone for 16 hours at approximately 4 cycles per hour. When cartridges are reused, diethyl ether/hexane (5 to 10 percent v/v) can be used as the cleanup solvent.

NOTE: A modified PUF cleanup procedure can be used to remove unknown interference components of the PUF blank. This method consists of compressed rinsing 50 times with toluene, acetone, and diethyl ether/hexane (5 to 10 percent v/v) and followed by Soxhlet extraction.

3.4.3.4 The extracted PUF is placed in a vacuum oven connected to a water aspirator and a source of clean dry nitrogen or air. Nitrogen or air is purged through the oven to dry the PUF at room temperature for approximately 2 to 4 hours. At the end of the drying period, there should be no solvent odor detected.

3.4.3.5 The extract from the Soxhlet extraction procedure from each batch may be analyzed for the purpose of determining initial cleanliness prior to certification.

3.4.4 **PUF/XAD-2 Adsorbent Cartridge Preparation**

3.4.4.1 A nickel or stainless steel screen (mesh size 200/200) is fitted to the bottom of a hexane-rinsed glass sampling cartridge to retain the PUF/XAD-2 adsorbents, as illustrated in Figures D/SV-1 and D/SV-2. A Soxhlet-extracted, vacuum dried PUF (2.5-cm thick by 6.5-cm diameter) is placed on top of the screen in the glass sampling cartridge using polyester gloves.

3.4.4.2 Approximately 10 g of 35/60 mesh Soxhlet-extracted, vacuum dried XAD-2 resin is placed into the sampling cartridge (using clean white cotton gloves) on top of the PUF. A second Soxhlet-extracted, vacuum-dried PUF adsorbent (6.5-cm x 5.0-cm) is placed on top of the XAD-2 resin bed. Finally, the top nickel or stainless steel screen is fitted to the sampling cartridge.

3.4.4.3 The glass module containing the PUF/XAD-2 adsorbent is wrapped with hexane-rinsed aluminum foil, placed in a labeled container, and tightly sealed with Teflon® tape or liner.

NOTE: The aluminum foil should be baked in an oven overnight at 500°C after rinsing with hexane to ensure that no residuals remain.

3.5 PROCEDURE FOR CERTIFICATION OF PUF/XAD-2 CARTRIDGE ASSEMBLIES

3.5.1 Assemble the Soxhlet apparatus. Charge the Soxhlet apparatus with 500 mL of the extraction solvent (10 percent v/v diethyl ether/hexane) and reflux for 2 hours. Let the apparatus cool, disassemble it, and discard the used extraction solvent. Transfer the filter and PUF/XAD-2 glass cartridge to the Soxhlet apparatus (the use of an extraction thimble is optional).

NOTE: The filter and adsorbent assembly are tested together in order to reach detection limits and to minimize cost.

3.5.2 Add 500 mL of diethyl ether/hexane (10 percent v/v) to the Soxhlet apparatus. Reflux the sample for 18 hours at a rate of at least 3 cycles per hour. Allow to cool, then disassemble the apparatus.

3.5.3 Assemble a K-D concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D, if equivalency is demonstrated for all the target analyte compounds listed in Exhibit C.

3.5.4 Transfer the extract by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate, and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of 10% diethylether/hexane to complete the quantitative transfer.

3.5.5 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL of the extraction solvent to the top of the column. Place the K-D apparatus on a hot water bath (50°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in one hour. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 5 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 5 mL of hexane. A 5-mL syringe is recommended for this operation.

3.5.6 Two different types of concentration techniques are permitted to obtain a final volume of 1.0 mL: micro-Snyder column and nitrogen blowdown techniques outlined in sections 4.4.2.4 and 4.4.2.5, respectively.

3.5.7 Analyze the extract by GC/MS according to the conditions in section 6.6. Only a single point standard is used for certification. The

analytes in the standard should have concentrations at or slightly above the analyte CRQLs per injection (similar to CAL 1).

3.5.8 The level of each target analyte for the extract each pair of filter and adsorbent assembly analyzed must be less than or equal to the analyte's CRQL, and the total level of target compounds must not exceed 10 μg.

3.5.9 Document results on Form VIII-AASV, Filter/Adsorbent Cartridge Certification Form. Reporting requirements are listed in Exhibit B.

3.6 DEPLOYMENT OF CARTRIDGES FOR FIELD SAMPLING

3.6.1 Prior to field deployment, a minimum of three surrogate compounds (i.e., chemically inert compounds not expected to occur in an environmental sample) are added to the center bed of the top PUF cartridge, using a microsyringe. The surrogate compounds must be added to each cartridge assembly.

3.6.2 The recoveries of the surrogate compounds are used to monitor for unusual matrix effects and gross sampling processing errors. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits.

3.6.3 The following surrogate standards have been successfully used in determining matrix effects, breakthrough, and analytical problems by GC/MS. Refer to section 6.5.4 for preparation of surrogate spiking mixture.

	Surrogate Compound	Spiked Amount (μg/cartridge)
	Anthracene-d_{10}	100
	Benzo(a)pyrene-d_{12}	100
	2,4,6-Tribromophenol	100
	2-Fluorophenol	100
	Phenol-d_5	100
*	Nitrobenzene-d_5	100
*	2-Fluorobiphenyl	100
*	p-Terphenyl-d_{14}	100

* These three surrogate compounds must be added to each adsorbent cartridge sent out to the field as pre-sampling surrogates. The other five surrogate compounds must be added to the cartridges just prior to extraction. These concentrations are based upon a 1-μL injection of the resulting extract.

4 SAMPLE PREPARATION FOR GC/MS ANALYSIS

4.1 SUMMARY OF METHOD

The filters and adsorbent cartridges are spiked with post-sampling surrogate compounds and extracted with a Soxhlet apparatus with the appropriate solvent. The extract is concentrated by Kuderna-Danish (K-D) evaporator, followed by optional GC/ECD or GC/FID screening, and finally GC/MS analysis, as illustrated in Figure D/SV-3.

4.2 APPARATUS AND MATERIALS

4.2.1 Soxhlet extractors: Capable of extracting GMW Model PS-1 filter and adsorbent cartridges (2.3 in. x 5 in. length), 1000-mL flask, and condenser.

4.2.2 Pyrex glass tube furnace system: For activating silica gel at 180°C under purified nitrogen gas purge for an hour, with the capability of raising temperature gradually, best source.

4.2.3 Glass vials: 40-mL.

4.2.4 Erlenmeyer flask: 50-mL.

4.2.5 Spatulas and spoons: Teflon-coated stainless steel.

4.2.6 Kuderna-Danish (K-D) apparatus: 500-mL evaporation flask (Kontes K-570001-500, or equivalent), 10-mL graduated concentrator tubes (Kontes K-570050-1025, or equivalent) with ground-glass stoppers, and 3-ball macro Snyder Column (Kontes K-5700010500, K-50300-0121, and K-569001-219, or equivalent).

4.2.7 Adsorption columns for column chromatography: 1 cm x 10 cm with stands.

4.2.8 White cotton gloves: For handling cartridges and filters.

4.2.9 Minivials: 2-mL, borosilicate glass, with conical reservoir and screw caps lined with Teflon-faced silicone disks, and a vial holder.

4.2.10 Glove box: For working with extremely toxic standards and reagents with explosion-proof hood for venting fumes from solvents, reagents, etc.

4.2.11 Vacuum oven: Vacuum drying oven system capable of maintaining a vacuum at 240 torr (flushed with nitrogen) overnight.

4.2.12 Concentrator tubes and a nitrogen evaporation apparatus: With a variable flow rate.

4.2.13 Laboratory refrigerator.

4.2.14 Boiling chips: Solvent-extracted 10/40 mesh silicon carbide, or equivalent.

4.2.15 Water bath: Heated, with concentric ring cover, capable of ±5°C temperature control.

4.2.16 Vortex evaporator (optional).

4.2.17 Teflon® sleeves for the ground-glass joints on the Soxhlet extraction apparatus.

NOTE: Reuse of glassware should be minimized to avoid the risk of cross-contamination. All glassware that is used, especially glassware that is reused, must be scrupulously cleaned as soon as possible after use. Rinse glassware with the last solvent used in it and then with high-purity acetone and hexane. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water. Drain dry and heat in a muffle furnace at 400°C for 4 hours. Volumetric glassware must not be heated in a muffle furnace; rather, it should be rinsed with high-purity acetone and hexane. After the glassware is dry and cool, rinse it with hexane, and store it inverted or capped with solvent-rinsed aluminum foil in a clean environment.

4.3 REAGENTS

4.3.1 Methylene chloride: Chromatographic grade, glass-distilled.

4.3.2 Sodium sulfate: Anhydrous (ACS), granular (purified by washing with methylene chloride followed by heating at 400°C for 4 hours in a shallow tray).

4.3.3 Diethyl ether: Chromatographic grade, glass-distilled.

4.3.4 Hexane: Chromatographic grade, glass-distilled.

4.3.5 Nitrogen: High purity grade.

4.4 PROCEDURE

4.4.1 Soxhlet Extraction

4.4.1.1 Assemble the Soxhlet apparatus. Charge the Soxhlet apparatus with 500 mL of the extraction solvent and reflux for 2 hours. Let the apparatus cool, disassemble it, transfer the PUF/XAD-2 adsorbent and filter to the Soxhlet apparatus (the use of an extraction thimble is optional), and discard the spent solvent.

NOTE: The filter and adsorbent are analyzed together in order to reach Contract Required Quantitation Limits (CRQLs), to avoid questionable interpretation of the data, and to minimize cost.

4.4.1.2 Prior to extraction, the following five additional surrogate compounds are spiked onto each cartridge received from the field.

Surrogate Compound	Spiked Amount (µg/cartridge)
Anthracene-d_{10}	100
Benzo(a)pyrene-d_{12}	100
2,4,6-Tribromophenol	100
2-Fluorophenol	100
Phenol-d_5	100

For blank and sample cartridges not sent out to the field, these surrogate compounds are spiked along with the three pre-sampling surrogates (nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}) just prior to extraction. The surrogate spiking technique is illustrated in Figure D/SV-5 at the end of this exhibit.

NOTE: The recovery of the surrogate standards are used to monitor for unusual matrix effects, gross sample processing errors, problems with the analytical method, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits.

4.4.1.3 Add 500 mL of diethyl ether/hexane (10 percent v/v) to the Soxhlet apparatus. Reflux the sample for 18 hours at a rate of at least 3 cycles per hour. Allow to cool then disassemble the apparatus.

4.4.2 Extract Concentration

4.4.2.1 Assemble a K-D concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D, if equivalency is demonstrated for all the target analyte compounds listed in Exhibit C.

4.4.2.2 Transfer the extract from section 4.4.1.3 by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate, and collect the extract into the K-D concentrator. Rinse the flask and column with 20 to 30 mL of 10% diethyl ether/hexane to complete the quantitative transfer.

4.4.2.3 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL of the extraction solvent to the top of the column. Place the K-D apparatus on a hot water bath (50°C) so that the

concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in one hour. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 5 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 5 mL of hexane. A 5-mL syringe is recommended for this operation. The extract is now ready for further concentration to 1.0 mL by either the micro-Snyder column or nitrogen blowdown techniques.

4.4.3 Micro-Snyder Column Technique

4.4.3.1 Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro-Snyder column.

4.4.3.2 Pre-wet the Snyder column by adding about 1 mL of hexane to the top of the column.

4.4.3.3 Place the K-D apparatus on a hot water bath (50°) so that the concentrator tube is partially immersed in hot water.

4.4.3.4 Adjust the vertical position of the apparatus and the water temperature required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent.

4.4.3.5 When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for a least 10 minutes while cooling.

4.4.3.6 Remove the Snyder column and rinse its flask and its lower joint into the concentrator tube with 0.2 mL of hexane.

4.4.3.7 Adjust the final volume to 1.0 mL with hexane.

4.4.3.8 Transfer the extract to a Teflon-sealed screw-cap amber vial. Label the vial and store at 4°C (±2°C). The extract is now ready for optional GC/FID or GC/ECD screening, followed by GC/MS analysis.

4.4.4 Nitrogen Blowdown Technique (from ASTM Method D3086)

CAUTION: Gas lines from the gas source to the blowdown apparatus must be stainless steel, copper, or Teflon tubing.

4.4.4.1 Place the concentrator tube with an open micro-Snyder attachment in a warm water bath (30° to 35°C) and evaporate the solvent volume to just below 1 mL by blowing a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon) above the extract.

4.4.4.2 The internal wall of the concentrator tube must be rinsed down several times with hexane during the operation.

4.4.4.3 During evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry.

4.4.4.4 Bring the final volume brought to 1.0 mL with hexane. Transfer the extract to a Teflon-sealed screw-cap amber vial, label the vial and store at 4°C (±2°C). The extract is now ready for optional GC/FID or GC/ECD screening, followed by GC/MS analysis.

4.5 DILUTIONS

4.5.1 When a sample extract is analyzed that has an analyte target compound concentration greater than the upper limit of the initial calibration range or saturated ions from a compound (excluding the compound peaks in the solvent front), the extract must be diluted and reanalyzed. Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion. If secondary ion quantitation is used, calculate a relative response factor using the area response from the most intense secondary ion which is free of sample interferences, and document the reasons for the use of the secondary ion in the SDG Narrative.

4.5.2 Calculate the sample dilution necessary to keep the semivolatile target compounds that required dilution within the upper half of the initial calibration range and so that no compound has saturated ions (excluding the compound peaks in the solvent front). Dilute the sample in hexane in a volumetric flask. Analyze the diluted sample per section 6.13.3.

4.5.3 The dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the initial calibration range of the instrument.

4.5.4 If the on-column concentration of any target compound in any sample exceeds the initial calibration range, that sample must be diluted, the internal standard concentration readjusted, and the sample extract reanalyzed.

4.5.5 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak

within the initial calibration range.

4.5.6 Do not submit data for more than two analyses, i.e., the original sample extract and one dilution, or, if the semivolatile screening procedure was employed, from the most concentrated dilution analyzed and one further dilution.

5 GC/FID OR GC/ECD OPTIONAL SCREENING METHODOLOGY

5.1 SUMMARY OF METHOD

As an option, the sample extract may be screened prior to GC/MS analysis to approximate the range of concentration of target compounds for dilution prior to quantitation with the GC/MS. Screening is accomplished utilizing a gas chromatograph coupled to specific detectors. For pesticides and other chlorinated compounds, electron capture detector (ECD) is used, and for the other semivolatiles, a flame ionization detector (FID) is employed.

5.2 APPARATUS

5.2.1 Gas chromatograph: The gas chromatograph (GC) system must adequately regulate temperature in order to give a reproducible temperature program and have a flow controller that maintains a constant column flow rate throughout temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases.

5.2.2 GC column: 30 m x 0.25 mm I.D. fused silica column, 1.0 μm filter thickness (DB-5, J&W Scientific, Folsom, CA, or equivalent).

5.2.3 Electron capture detector: The makeup gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. The GC/ECD system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants which may interfere with the analysis. The instrument must be vented to the outside of the facility or to a trapping system which prevents the release of contaminants into the instrument room.

5.2.4 Flame ionization detector.

5.2.5 Data system: A data system must be interfaced to the GC/FID or GC/ECD. The data system must allow the continuous acquisition of data throughout the duration of the chromatographic program and must permit, at the minimum, the output of time vs intensity (peak height or peak area) data. Also, the data system must be able to rescale chromatographic data in order to report chromatograms meeting the requirements listed within this method.

5.3 REAGENTS

The carrier gas for routine applications is helium. Laboratories may choose to use hydrogen as a carrier gas, but must clearly identify its use in the SDG Narrative and on all divider pages preceding raw chromatographic data in submissions to the Agency. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of

a hydrogen leak detector is highly recommended when hydrogen is used as the carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with rubber components are not to be used.

5.4 STANDARD PREPARATION

5.4.1 The CAL 3 working standard solutions in hexane prepared according to section 6.5.11 may be used for screening purposes.

5.4.2 If the level of chlorinated solvent (from the stock solutions) in the standards prepared for GC/MS analysis interferes with the GC screening methods, stock standard solutions made purely in non-chlorinated solvents such as hexane may be prepared, then accurately diluted in an appropriate solvent to contain CAL 3 levels outlined in Table D/SV-1.

5.5 INSTRUMENT OPERATING CONDITIONS

Suggested GC operating conditions are as follows:

GC Column:	DB-5 fused silica, 1.0 μm 5% phenyl methyl siloxane bonded, 30 m x 0.25 mm I.D.
Carrier Gas:	Helium, 28-30 cm/sec
Flow Rate:	1 cm/minute.
Column Program:	Initial Temperature: 40°C
Initial Time:	1 min
Ramp Rate:	15°C/min to 200°C, increase to 3°C/min
Final Temperature:	300°C.

5.6 PROCEDURE

5.6.1 Before analysis can be performed, make sure that the system is within optimum operating conditions. This is an optimal screening procedure, and it is up to the laboratory to ensure that the GC is properly calibrated, and that stable retention times are obtained.

5.6.2 Inject the calibration standard which may be the mid level (CAL 3) standard in the GC/MS analysis initial calibration range.

5.6.3 Inject the appropriate extract from Section 4 including the blanks. The laboratory must note that if an ECD detector is used, the internal standards which are in methylene chloride should not be in the extract when performing GC/ECD screening.

5.6.4 If no peaks are observed from the extract, it may be analyzed by GC/MS without dilution.

5.6.5 If peaks are detected, calculate the approximate concentrations and

the necessary dilutions, if any, to reduce the major peaks to between half and full scale deflection when performing GC/MS analysis.

5.6.6 Make any necessary dilutions and proceed with GC/MS analysis (Section 6).

5.6.7 It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

6 GC/MS ANALYSIS

6.1 SUMMARY OF METHOD

This method outlines a GC/MS procedure for the analysis of semivolatiles following sample preparation (Section 4) or optional screening (Section 5).

6.2 INTERFERENCES

6.2.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that result in discrete artifacts and/or elevated baselines in the detector profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running a laboratory method blanks (LMB).

6.2.2 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water and rinsing with tap water and reagent water. It should then be drained dry, solvent rinsed with acetone and spectrographic grade hexane. After drying and rinsing, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Glassware should be stored inverted or capped with aluminum foil.

NOTE: The glassware, except for volumetrics, may be further cleaned by placing in a muffle furnace at 450°C for 8 hours to remove trace organics.

6.2.3 The use of high purity water, reagents, and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

6.2.4 Matrix interferences may be caused by contaminants that are coextracted from the sample.

6.2.5 The extent of interferences that may be encountered has not been fully assessed. Although GC conditions described allow for unique resolution of compounds covered by this method, other compounds may interfere. The analytical system must be routinely demonstrated to be free of internal contaminants such as contaminated solvents, glassware, or other reagents which may lead to method interferences. A laboratory reagent blank may be run for each batch of reagents used to determine if reagents are contaminant-free.

6.2.6 There are concerns that during sample transport and analysis, heat, ozone, NO_2, and ultraviolet (UV) light may cause sample degradation. These problems should be addressed as part of the user-prepared SOP manual. Where possible, incandescent or UV-shielded fluorescent lighting in the laboratory should be used during analysis.

6.3 APPARATUS AND MATERIALS

6.3.1 Gas chromatograph: An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection equipped with a flow controller that maintains a constant column flow rate throughout temperature program operations, and all required accessories, including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with rubber components should not be used.

6.3.2 Mass spectrometer: Capable of scanning from 35 to 510 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact (EI) ionization mode and producing a mass spectrum which meets all the instrument performance criteria when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet. To ensure sufficient precision of mass spectral data, the MS scan rate must allow acquisition of at least five scans while a sample compound elutes from the GC. The GC/MS system must be in a room with atmosphere demonstrated to be free of all potential contaminants which will interfere with the analysis. The instrument must be vented outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

NOTE: DFTPP criteria must be met before any sample extracts are analyzed. Any samples analyzed when DFTPP criteria have not been met will require reanalysis at no cost to the Agency.

6.3.3 GC/MS interface: Any gas chromatograph to mass spectrometer interface that gives acceptable calibration points for each of the parameters of interest, and achieves all acceptable performance criteria (Exhibit E), may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.3.4 Data system: A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching the GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defined as Selected Ion Current Profile (SICP). Software must be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows comparing sample spectra against reference spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

6.3.5 Magnetic tape storage device: The magnetic tape storage device must be capable of recording data and suitable for long-term, off-line storage.

6.3.6 Chromatographic Column: A 30 m x 0.25 mm I.D. (or 0.32 mm I.D.) bonded-phase silicone-coated fused silica capillary column (J&W Scientific DB-5, Alltech Associates SE-54, or equivalent) must be used. A film thickness of 1.0 μm is recommended because of its larger capacity. A film thickness of 0.25 μm may also be used. A megabore column (0.53 mm I.D.) may also be used as long as all QC criteria are met.

6.4 REAGENTS

6.4.1 Hydrogen and helium: Gas cylinders of ultra high purity, (99.9999 percent).

6.4.2 Combustion air: Ultra high purity.

6.4.3 Zero air: May be obtained from a cylinder or zero-grade compressed air scrubbed with Drierite or silica gel and 5A molecular sieve or activated charcoal, or by catalytic cleanup of ambient air. All zero air must pass through a liquid argon cold trap for final cleanup.

6.5 STANDARDS

6.5.1 The Contractor must provide all standards to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E or by the manufacturer. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request. Each calibration standard solution shall contain all the appropriate surrogates and internal standards. Neat standards with compound purity of at least 97 percent must be used.

6.5.2 Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source, or traceable to EPA-supplied standards. Fresh stock standards must be prepared once every twelve months, or sooner, if standards have degraded or concentrated. Stock standards should be checked for signs of degradation or concentration just prior to preparing secondary dilution and working standards from them. Table D/SV-1 outlines the concentrations of the working calibration standards.

CAUTION: Each time a vial containing small volumes of solution is warmed to room temperature and opened, a small volume of solvent in the vial headspace evaporates, significantly affecting concentration.

6.5.3 Instrument performance check solution

Prepare a solution of decafluorotriphenylphosphine (DFTPP), such

that a 2-μL injection will contain 50 ng of DFTPP. The DFTPP should also be included in the calibration standards at this level.

6.5.4 Surrogate Compounds Spiking Solution

Prepare separate solutions to contain approximately 1000 μg/mL of each surrogate compound. From these solutions, prepare the spiking solution to obtain the following amounts when 1 mL is spiked into the cartridges. Fill a 1-mL syringe with the surrogate spiking solution and randomly inject a 200-μL portion into each of the five locations in the cartridge as illustrated in Figure D/SV-5.

	Surrogate Compound	Amount Spiked on Cartridge, μg
	Anthracene-d_{10}	100
	Benzo(a)pyrene-d_{12}	100
	2,4,6-Tribromophenol	100
	2-Fluorophenol	100
	Phenol-d_5	100
*	Nitrobenzene-d_5	100
*	2-Fluorobiphenyl	100
*	p-Terphenyl-d_{14}	100

* These surrogate compounds are spiked into the cartridges that are sent out to the field prior to field deployment (pre-sampling surrogates).

6.5.5 Internal Standards

Internal standard solutions of 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} can be prepared by dissolving 100 mg of each compound in 100 mL of methylene chloride. It may be necessary to use 5 to 10 percent benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. The resulting solution will contain each standard at a concentration of 1000 ng/μL. A 40-μL portion of this solution should be added to each 1 mL of sample extract. This will result in 40 ng of each internal standard in the 1-μL volume of extract injected into the GC/MS. See Table D/SV-3 for the internal standards used for each target and surrogate compound.

6.5.6 Laboratory Control Sample Spiking Solution

Table D/SV-11 lists the compounds used for the laboratory control sample (LCS). Prepare two spiking mixtures containing the acid-neutral LCS compounds and the base-neutral LCS compounds at a concentration level of 1000 μg/mL. Spike a clean cartridge with the mixtures to contain 50 μg of each LCS compound.

NOTE: Each mixture (acid-neutral and base-neutral) may be spiked

separately onto the cartridge or may be mixed into one solution just prior to injection into the cartridge.

6.5.7 **PAHs Stock Standard Solutions**

6.5.7.1 Place 0.1 gram of native PAHs on a tared aluminum weighing disk and weigh on a balance.

6.5.7.2 Quantitatively transfer each to a 10-mL volumetric flask. Rinse the weighing disk with several small portions of methylene chloride. Ensure all material has been transferred. Dilute to mark with methylene chloride. The concentration of the stock standard solution of PAHs in the flask is 10 $\mu g/\mu L$.

6.5.7.3 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C (±2°C) and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing working standards.

6.5.7.4 Secondary standards may be prepared by diluting the stock standard solutions. Working standards are then prepared from the secondary standard solutions.

6.5.8 **Pesticides Stock Standard Solutions**

6.5.8.1 Prepare from pure standard materials. Weigh approximately 25.0 mg (with accuracy of 0.1 mg) of each pure pesticide analyte, except Endosulfan I and Endosulfan II. For these two pesticides, prepare a stock solution twice as concentrated as that prepared for other pesticide analytes. Dissolve each compound in hexane and dilute to volume in a 10-mL (5-mL for the two Endosulfans) volumetric flask. (Concentration of each component = 3.5 mg/mL, except the Endosulfans, which should be 5 mg/mL.) Smaller or larger volumes of stock solution may be used if desired.

6.5.8.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C (±2°C) and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing working standards.

6.5.8.3 Secondary standards may be prepared by diluting the stock standard solutions. Working standards are then prepared from the secondary standard solutions.

6.5.9 **PCBs Stock Standard Solutions**

6.5.9.1 Prepare a stock solution of each of the nine PCB concentration calibration congeners at a concentration of 1 $\mu g/\mu L$ in

hexane. Place each solution in a clean glass vial with a Teflon-lined screw cap. Fill to the top so no headspace is evident. Store at 4°C if solutions are not to be used right away. Solutions are stable indefinitely if solvent evaporation is prevented.

6.5.9.2 The 9 individual PCB congeners listed in Table D/SV-4 are used as the calibration compounds for PCB determinations. One isomer at each level of chlorination is used as the concentration calibration standard for all other isomers at that level of chlorination, except decachlorobiphenyl (Cl_{10}) which is used for both Cl_9 and Cl_{10} isomer groups. The basis for selection of these calibration congeners has been reported and referenced in Section 7 (Citation 9) and Table D/SV-4.

6.5.9.3 Take aliquots of the stock solutions of the nine PCB concentration calibration congeners and mix together in the proportions that will provide a primary dilution standard solution of the composition ratios illustrated in Table D/SV-1. Place each solution in a clean glass vial with a Teflon-lined screw cap and store at 4°C. Mark the meniscus on the vial wall to monitor solution volume during storage; solutions are stable indefinitely if solvent evaporation is prevented.

6.5.9.4 Five calibration solutions are required containing the internal standards, surrogates, and target compounds as specified in Table D/SV-1. Because MS response to PCBs decreases with increasing level of chlorination, PCB congener concentrations in calibration solutions increase with level of chlorination. Components of the highest concentration solution (CAL 5) are present at a concentration that allow injections of 1 μL aliquots without MS saturation or GC column overloading.

6.5.10 **Stock Standard Solutions of Other Semivolatiles**

6.5.10.1 Place 0.1 gram of each native semivolatile target analyte on a tared aluminum weighing disk and weigh on a balance.

6.5.10.2 Quantitatively transfer to a 10-mL volumetric flask. Rinse the weighing disk with several small portions of methylene chloride. Ensure that all material have been transferred. Dilute to mark with methylene chloride. The concentration of the stock standard solution of semivolatiles in the flask is 10 μg/μL.

6.5.10.3 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C (±2°C) and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards.

6.5.10.4 Secondary standards may be prepared by diluting the stock

standard solutions. Working standards are then prepared from the secondary standard solutions.

6.5.11 **Working Calibration Standards**

Prepare calibration standards in hexane at a minimum of five concentration levels as outlined in Table D/SV-1. Each calibration standard shall contain the appropriate target compounds, internal standards, and surrogate compounds as outlined in Tables D/SV-1. Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at 4°C (±2°C) in screw-cap amber bottles with Teflon liners. Fresh standards should be prepared every twelve months at a minimum. The continuing calibration standard (see Table D/SV-1, CAL 3) should be prepared weekly and stored at 4°C (±2°C).

6.5.12 **Storage of Standard Solutions**

6.5.12.1 Store the stock and secondary standard solutions at 4°C (±2°C) in Teflon-lined screw-cap amber bottles. Store the working standard solutions at 4°C (±2°C) in Teflon-lined screw-cap amber bottles.

6.5.12.2 Protect all standards from light. Samples, sample extracts, and standards must be stored separately.

6.5.12.3 Stock standard solutions must be replaced every twelve months, or sooner, if comparison with quality control check samples indicates a problem.

6.6 **INSTRUMENT OPERATING CONDITIONS**

6.6.1 Gas chromatograph: The following are the recommended gas chromatographic analytical conditions, as outlined also in Table D/SV-2, to optimize conditions for compound separation and sensitivity.

Carrier Gas:	Helium
Linear Velocity:	28-29 cm^3/sec
Injector Temperature:	250-300°C
Injector:	Grob-type, splitless.
Temperature Program:	Initial Temperature: 50°C
Initial Hold Time:	4.0 ± 0.1 min.
Ramp Rate:	8°C/min
Final Temperature:	280°C
Analytical Time:	approximately 50 minutes
Injection Volume*:	1-µL

NOTE: A volume of 1-µL has been found to work with the concentrations shown in this document. If a different volume is used, the laboratory

must make appropriate adjustments to the calibration concentrations. Smaller volumes may be used only with automated systems.

6.6.2 Mass spectrometer: The following are the required mass spectrometer conditions for full range data acquisition.

Transfer Line Temperature:	250-300°C
Source Temperature:	According to Manufacturer's Specifications
Electron Energy:	70 volts (nominal)
Ionization Mode:	EI
Mass Range:	35 to 500 amu, full range data acquisition
Scan Time:	At least 5 scans per peak, not to exceed 1 second per scan.

6.7 ANALYTICAL SEQUENCE

The GC/MS analytical sequence for each 12-hour time period shall be as follows:

6.7.1 Instrument Performance Check (DFTPP)

6.7.2 Initial or continuing calibration

6.7.3 Laboratory method blank (LMB)

6.7.4 Laboratory Control Sample (LCS)

6.7.5 Field Blank

6.7.6 ≤20 field sample extracts

6.7.7 Performance Evaluation (PE) Sample (if available)

6.8 INSTRUMENT PERFORMANCE CHECK

6.8.1 Summary

It is necessary to establish that a given GC/MS meet tuning and standard mass spectral abundance criteria prior to initiating any on-going data collection, as illustrated in Figure D/SV-4. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP).

6.8.2 Frequency

6.8.2.1 The instrument performance check solution of DFTPP must be analyzed initially and once per 12-hour time period of operation. Also, whenever the laboratory takes corrective action which may change

or affect the mass spectral criteria (e.g., ion source cleaning or repair, column replacement, etc.), the instrument performance check must be verified irrespective of the 12-hour laboratory requirement.

6.8.2.2 The 12-hour time period for GC/MS analysis begins at the injection of the DFTPP which the laboratory submits as documentation of a compliance tune. The time period ends after 12 hours have elapsed. In order to meet instrument performance check requirements, samples, blanks, and standards must be injected within 12 hours of the DFTPP injection.

6.8.3 **Procedure**

6.8.3.1 Inject 50 ng of DFTPP into the GC/MS system. All instrument conditions must be identical to those listed in section 6.6 except that a different temperature program may be used.

6.8.3.2 The DFTPP may be analyzed separately or as part of the calibration standard.

6.8.4 **Technical Acceptance Criteria**

6.8.4.1 Prior to the analysis of any samples, blanks, or calibration standards, the laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing DFTPP.

6.8.4.2 The GC/MS system must be checked for instrument performance at the frequency described in section 6.8.2. The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant. The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution.

6.8.4.3 The abundance criteria listed in Table D/SV-5 must be met for a 50 ng injection of DFTPP. The mass spectrum of DFTPP must be acquired by averaging three scans (the peak apex scan and the scans immediately preceding and following the apex). Background subtraction is required, and must be accomplished using a single scan prior to the elution of DFTPP.

NOTE: All ion abundance **MUST** be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

6.8.4.4 The criteria above are based on adherence to the acquisition specifications identified in Table D/SV-5 and were developed for the specific target compound list associated with this document. The criteria are based on performance characteristics of instruments

currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for target analytes are designed to control and monitor instrument performance associated with the requirements of this document. As they are performance-based criteria for these specific analytical requirements, they may not be optimal for additional target compounds.

6.8.5 Corrective Action

6.8.5.1 If the DFTPP acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other actions to achieve the acceptance criteria.

6.8.5.2 The DFTPP acceptance criteria MUST be met before any standards, field samples, or required blanks are analyzed. Any standards, field samples, or required blanks analyzed when tuning criteria have not been met will require reanalysis at no additional cost to the Agency.

6.8.6 Documentation

Reporting requirements are listed in Exhibit B. Instrument Performance Check data results are reported on Form IV-AASV, GC/MS Instrument Performance Check and Mass Calibration Form.

6.9 INITIAL CALIBRATION

6.9.1 Summary

6.9.1.1 Prior to the analysis of samples and required blanks, and after tuning criteria (instrument performance check) have been met, each GC/MS system must be initially calibrated at a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the analyte compounds and the surrogates.

6.9.1.2 All sample results are quantified using the RRFs from CAL 3 in the initial calibration or the RRFs from the most recent valid continuing calibration standard within the same 12-hour period.

6.9.2 Frequency

Each GC/MS system must be initially calibrated upon award of the contract, whenever the laboratory takes corrective action which may change or affect the initial calibration criteria (e.g., ion source cleaning or repair, column replacement, etc.), or if the continuing calibration acceptance criteria (see section 6.10.5) have not been met.

6.9.3 **Procedure**

6.9.3.1 Set up the GC/MS system under the conditions outlined in section 6.6.

6.9.3.2 All working standard solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.

6.9.3.3 Tune the GC/MS system to meet the technical acceptance criteria in section 6.8.4 for DFTPP.

6.9.3.4 Prepare five calibration standards containing the target compounds, internal standards, and surrogate compounds at the concentrations outlined in Table D/SV-1 and according to the procedure in section 6.5.

6.9.3.5 Calibrate the GC/MS by injecting 1.0 µL of each standard. If a compound saturates when the CAL 5 standard is injected, and the GC/MS system is calibrated to achieve a detection sensitivity of no less than the CRQL for each compound, the laboratory must document it in the SDG Narrative, and attach a quantitation report and chromatogram (see Exhibit B). In this instance, the laboratory must calculate the results based on a four-point initial calibration for the specific compound that saturates. Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion. If secondary ion quantitation is used, calculate a relative response factor using the area response from the most intense secondary ion which is free of interferences, and document the reasons for the use of the secondary ion in the SDG Narrative.

6.9.3.6 Record a spectrum of each target compound. Background subtraction and spectrum averaging may be needed. Judge the acceptability of recorded spectra by comparing them to spectra in libraries. If an acceptable spectrum of a calibration standard component is not acquired, take necessary actions to correct GC/MS performance. If performance cannot be corrected, report sample extract data for the particular compound(s), but document the affected compound(s) and the nature of the problem.

6.9.3.7 For PCBs, compare the quantitation ion to the confirmation ion. A proper calibration using the CAL 3 standard should document the ratio in the ranges found in Table D/SV-7.

6.9.4 **Calculations**

NOTE: In the following calculations, the area response is that of the primary quantitation ion unless otherwise stated.

6.9.4.1 Relative Response Factors (RRF): Table D/SV-8 outlines

characteristic ions for the surrogate compounds and internal standards. Table D/SV-9 outlines primary and secondary quantitation ions for the target compounds. Calculate RRFs for each analyte target compound and surrogate to the appropriate internal standard using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \qquad \text{Eq. D/SV-1}$$

where: A_x = area of the primary quantitation ion for the compound to be measured;
A_{is} = area of the primary quantitation ion for the internal standard;
C_{is} = concentration or amount of the internal standard; and
C_x = concentration or amount of the compound to be measured.

NOTE: C_{is} and C_x must be in the same units.

6.9.4.2 Percent Relative Standard Deviation (%RSD): Using the RRFs from the initial calibration, calculate the %RSD for all target compounds and surrogates using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\bar{x}} \times 100 \qquad \text{Eq. D/SV-2}$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N - 1}} \qquad \text{Eq. D/SV-3}$$

where: SD_{RRF} = standard deviation of initial response factors (per compound);
$\bar{x}$ = mean of initial relative response factors (per compound); and
x_i = RRF for each calibration level.

6.9.4.3 Relative Retention Times (RRT): Calculate the RRTs for each target compound and surrogate over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{is}} \qquad \text{Eq. D/SV-4}$$

where: RT_c = retention time of the target compound; and
RT_{is} = retention time of the internal standard.

6.9.4.4 Mean of the Relative Retention Times ($\overline{RRT}$): Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target compound and surrogate over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n} \qquad \text{Eq. D/SV-5}$$

where: $\overline{RRT}$ = mean relative retention time for the target compound or surrogate for each initial calibration standard; and
RRT_i = relative retention time for the target compound or surrogate for each initial calibration standard.

6.9.4.5 Mean Area Response ($\overline{Y}$) for Internal Standard: Calculate the area response (Y) mean for primary quantitation ion each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n} \qquad \text{Eq. D/SV-6}$$

where: $\overline{Y}$ = mean area response; and
Y_i = area response for the primary quantitation ion for the internal standard for each calibration level.

6.9.4.6 Percent Area Response Change (%ARC): Calculate the %ARC at each calibration level for each of the internal standards using the following equation.

$$\%ARC = \frac{A_x - \overline{Y}}{\overline{Y}} \times 100 \qquad \text{Eq. D/SV-7}$$

where: %ARC = percent area response change;
A_x = area response of the internal standard at a concentration level; and
$\overline{Y}$ = mean area response of the internal standard in the entire calibration range.

6.9.4.7 Mean of the Retention Times ($\overline{RT}$) For Internal Standard: Calculate the mean of the retention times ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n} \qquad \text{Eq. D/SV-8}$$

where: $\overline{RT}$ = mean retention time; and
RT = retention time for the internal standard for each initial calibration standard.

6.9.4.8 Internal Standard Retention Time Shift (RTS): Calculate the RTS between the RT of each internal standard at each concentration level and the $\overline{RT}$ for that internal standard over the entire calibration range using the following equation:

$$RTS = \overline{RT_i} - RT_x \qquad \text{Eq. D/SV-9}$$

where: $\overline{RT_i}$ = mean of the retention time for the internal standard in the initial calibration; and
RT_x = retention time of the internal standard at a concentration level.

6.9.5 Technical Acceptance Criteria

6.9.5.1 All initial calibration standards must be analyzed at the concentration levels and frequency described in this section on a GC/MS system meeting the DFTPP instrument performance check criteria.

6.9.5.2 The performance criteria for initial calibration of the CAL 3 must show:

- Baseline separation of beta-BHC and gamma-BHC;
- Baseline separation of endrin ketone and chrysene-d_{12};
- Signal/noise ratio of ≥5 for m/z 499 of PCB congener Cl_{10}-PCB to illustrate MS sensitivity;
- Lack of degradation of endrin: examine an extracted ion current profile (EICP) for m/z 67 in the retention time window between 4,4'-DDE and endosulfan sulfate; confirm that the abundance of m/z 67 at the retention time of endrin aldehyde is greater than 10 percent of the abundance of m/z 67 produced by endrin; and

6.9.5.3 The RRFs at each calibration concentration for each target compound and surrogate that has a required minimum response factor value must be greater than or equal to the compound's minimum acceptable relative response factor see Table D/SV-6.

6.9.5.4 The %RSD over the initial calibration range for each target compound and surrogate that has a required maximum %RSD must be less than or equal to the required maximum value. For all the other target compounds, the value for %RSD must be less than or equal to 25 percent. When the value for %RSD exceeds 25 percent, analyze additional aliquots of appropriate CALs to obtain an acceptable %RSD of RRFs over the entire concentration range, or take action to improve GC/MS performance.

6.9.5.5 The RRT for each of the target compounds and surrogates at

each calibration level must be within ±0.06 relative retention time units of the mean relative retention time for the compound.

6.9.5.6 The internal standard %ARC at each calibration level must be within ±40 percent of the mean area response ($\bar{Y}$) over the initial calibration range for each internal standard.

6.9.5.7 The retention time shift for each of the internal standards at each calibration level must be within ±20.0 seconds compared to the mean retention time ($\overline{RT}$) over the initial calibration range for each internal standard.

6.9.5.8 The compounds listed in Exhibit C must meet the minimum RRF and maximum %RSD criteria for the initial calibration, with allowance made for up to four target and surrogate compounds. However, the RRFs for those four compounds must be greater than 0.010, and the %RSD of those four compounds must be less than or equal to 40.0 percent for the initial calibration to be acceptable.

6.9.6 Corrective Action

6.9.6.1 If the technical acceptance criteria for initial calibration are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the acceptance criteria.

6.9.6.2 All initial calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed in a 12-hour time period for an initial calibration analytical sequence. Any samples or required blanks analyzed when initial calibration criteria have not been met will require reanalysis at no additional cost to the Agency.

6.9.7 Documentation

Reporting requirements are listed in Exhibit B. Initial calibration data results are reported on Form V-AASV, Initial Calibration Data Sheet. Internal standard area and retention times shall be tabulated on Form VII-AASV.

6.10 CONTINUING CALIBRATION

6.10.1 Summary

Prior to the analysis of samples and required blanks and after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a continuing calibration standard (CAL 3) to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the method, as illustrated in Figure D/SV-4. The continuing calibration standard

(CAL 3) shall contain the appropriate target compounds, surrogates, and internal standards.

6.10.2 **Frequency**

The GC/MS system shall be calibrated with a continuing calibration standard (CAL 3) at the beginning of every 12-hour time period after the injection of DFTPP, to check for the validity of the initial calibration.

6.10.3 **Procedure**

6.10.3.1 Set up the GC/MS system under the conditions outlined in section 6.6 and as specified by the manufacturer, and tune the GC/MS system to meet the technical acceptance criteria in section 6.8.4 for DFTPP.

6.10.3.2 All working standard solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.

6.10.3.3 Start the analysis of the continuing calibration by injecting 1.0 μL of the CAL 3 standard solution which contains the appropriate target analytes, surrogate compounds, and internal standards using the procedure listed the initial calibration section.

6.10.4 **Calculations**

6.10.4.1 Relative Response Factor (RRF): Calculate a relative response factor (RRF) for each target compound and surrogate using the equation in section 6.9.4.1.

6.10.4.2 Percent Difference (%D): Calculate the percent difference between the mean relative response factor ($\overline{RRF}$) from the most recent initial calibration and the continuing calibration RRF for each analyte target compound and surrogate using the following equation:

$$\%D_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100 \qquad \text{Eq. D/SV-10}$$

where: $\%D_{RRF}$ = percent difference between relative response factors;
$\overline{RRF_i}$ = average relative response factor from the most recent initial calibration; and
RRF_c = relative response factor from the continuing calibration standard.

6.10.5 **Technical Acceptance Criteria**

6.10.5.1 The continuing calibration standard must be analyzed for the compounds listed in Exhibit C at the frequency described in section

6.10.2 on a GC/MS system meeting the DFTPP instrument performance check and the initial calibration technical acceptance criteria.

6.10.5.2 The RRF for each target analyte and surrogate that has a required minimum relative response factor value must be greater than or equal to the compound's minimum acceptable relative response factor.

6.10.5.3 For an acceptable continuing calibration, the %D between the measured RRF for each target/surrogate compound of the CAL 3 standard and the mean value calculated during initial calibration must be within ±25.0 percent. If the criteria for %D are not met for any target or surrogate compound, remedial action must be taken and recalibration may be necessary.

6.10.6 Corrective Action

6.10.6.1 If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument according to section 6.9. It may be necessary to clean the ion source, change the column or take other corrective actions to achieve the acceptance criteria.

6.10.6.2 Continuing calibration technical acceptance criteria **MUST** be met before any samples or required blanks are analyzed in a 12-hour continuing calibration analytical sequence. Any samples or required blanks analyzed when continuing calibration criteria have not been met will require reanalysis at no additional cost to the Agency.

6.10.6.3 Remedial actions, which include but are not limited to the following, must be taken if criteria are not met:

- Check and adjust GC and/or MS operating conditions;
- Clean or replace injector liner;
- Flush column with solvent according to manufacturers instructions;
- Break off a short portion (approximately 0.33 m) of the column;
- Replace GC column (performance of all initial calibration procedures are then required);
- Adjust MS for greater or lesser resolution;
- Calibrate MS mass scale;
- Prepare and analyze new continuing calibration; and
- Prepare a new initial calibration curve.

6.10.7 Documentation

Reporting requirements are listed in Exhibit B. Continuing calibration data results are reported on Form VI-AASV, Continuing Calibration Data Form. Internal standard area and retention time shall be tabulated on Form VII-AASV.

6.11 BLANK ANALYSIS

6.11.1 Summary

6.11.1.1 To monitor for possible laboratory and field contamination, laboratory method blank (LMB) and field blanks are extracted with each SDG and analyzed at least once in a 12-hour analytical sequence. All steps in the analytical procedure are performed on the LMB using all reagents, standards, surrogate compounds, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

6.11.1.2 An LMB is an unused, certified filter/cartridge assembly which is carried through the same extraction procedure as a field sample. The LMB extract must contain the same amount of surrogate compounds and internal standards that is added to each sample. All field samples must be extracted and analyzed with an associated LMB.

6.11.1.3 A field blank is designed to detect potential sample contamination during the handling and shipping process of a field sample. The cartridge used as a field blank must be associated with the actual sampling process; therefore, the blank cartridge is opened with the other cartridges, resealed, and carried through the same handling process as those used to sample ambient air.

6.11.1.4 The same amount of internal standards that are added to each sample is added to each blank. All field samples must be analyzed with associated blanks.

6.11.2 Frequency

6.11.2.1 An LMB along shall be prepared along with each batch of ≤20 samples and shall be carried through the entire extraction, concentration, and analysis procedures. The laboratory method blank must be analyzed after the calibration standard(s) before any samples are analyzed. Whenever an unusually concentrated sample is encountered, an LMB analysis shall be performed immediately after the sample analysis.

6.11.2.2 A field blank is analyzed once per sample delivery group. A field blank shall be analyzed along with each batch of ≤20 samples and shall be carried through the entire analytical procedure.

6.11.2.3 The laboratory may also analyze a laboratory reagent blank which is the same as an LMB except that no cartridge is extracted, and no surrogate compounds or internal standards are added; this demonstrates that reagents do not contain impurities that produce an ion current above the level of background noise for any target compound quantitation ions.

6.11.3 Procedure

6.11.3.1 Spike the blank cartridges with the same amount of surrogate compounds using the same spiking technique as the field samples (see Figure D/SV-5). For the field blank, the three pre-sampling surrogates are spiked onto the cartridge prior to field deployment, along with the cartridges to be used for sampling ambient air. For the LMB, all eight surrogate compounds are spiked into the cartridges just prior to extraction.

6.11.3.2 Extract the blanks following the procedures in Section 4.

6.11.3.3 Add the internal standards to the blank extracts at the same concentration as the field sample extracts.

6.11.3.4 Analyze the blank extracts following the same analysis procedures as the field sample extracts.

6.11.4 Calculations

The equations in section 6.13.4 apply to the blanks.

6.11.5 Technical Acceptance Criteria

6.11.5.1 All blanks must be analyzed at the frequency described in section 6.11.2 on a GC/MS system meeting the DFTPP instrument performance check and initial calibration or continuing calibration technical acceptance criteria.

6.11.5.2 The percent recovery for each of the surrogates in the blank must be within the acceptance windows listed in section 6.13.5.3.

6.11.5.3 The percent area response change (%ARC) for each of the internal standards for the blank must be within ±40 percent compared to the internal standards in the most recent CAL 3 analysis.

6.11.5.4 The retention time for each of the internal standards must be within ±20.0 seconds between the blank and the most recent CAL 3 analysis.

6.11.5.5 The blank must not contain any target analyte at a concentration greater than its CRQL and must not contain additional

compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte at its CRQL. The total level of analytes in the blank filter/cartridge must not exceed 10 µg. If the LMB that was extracted along with a batch of samples is contaminated, the reported values for the entire batch of samples must be flagged with a "B."

6.11.6 Corrective Action

6.11.6.1 If a Contractor's blanks do not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds.

6.11.6.2 All samples processed with a method blank that is out of control (i.e., contaminated) must be flagged with a "B".

6.11.7 Documentation

Reporting requirements are listed in Exhibit B. Blank results are reported on Form I-AASV and associated samples are entered in Form II-AASV, except the surrogate recoveries, which are reported on Form IX-AASV. Internal standard area and retention time shall be tabulated on Form VII-AASV.

6.12 LABORATORY CONTROL SAMPLES (LCS)

6.12.1 Summary

The LCS is an internal laboratory quality control sample designed to assess (on an SDG-by-SDG basis) the capability of the Contractor to perform the analytical method listed in this Exhibit.. Table D/SV-11 lists the LCS compounds and the corresponding QC recovery limits.

6.12.2 Frequency

The LCS must be analyzed and reported once per 12-hour analytical sequence, and concurrently with the samples in the SDG.

6.12.3 Procedure

6.12.3.1 Prepare a PUF/XAD-2 cartridge spiked with the LCS spiking mixture(s), prepared according to section 6.5.6, to contain each LCS

compound at a concentration of 50 μg.

6.12.3.2 Spike the LCS cartridge with the surrogate compounds at the same concentration as the field samples.

6.12.3.3 Extract the LCS extract following the procedures in Section 4.

6.12.3.4 Add the internal standards to the LCS extract at the same concentration as the field sample extracts.

6.12.3.5 Analyze the LCS extract following the same analysis procedures as the field sample extracts.

6.12.4 **Calculations**

6.12.4.1 Calculate individual compound recoveries of the LCS using the following equation:

$$LCS\ \%Recovery = \frac{Concentration_{reported}}{Concentration_{spiked}} \times 100 \qquad \text{Eq. D/SV-11}$$

6.12.4.2 Field sample calculations in section 6.13 also apply to the LCS for the internal standards.

6.12.5 **Technical Acceptance Criteria**

6.12.5.1 The LCS must be analyzed on a GC/MS system meeting the tuning, initial or continuing calibration, and blank technical acceptance criteria at the frequency described in section 6.12.2.

6.12.5.2 The percent recovery for each of the surrogates in the LCS must be within the acceptance windows listed in section 6.13.5.3.

6.12.5.3 The percent recovery for each of the LCS compounds must be within the QC recovery limits listed in Table D/SV-11.

6.12.5.4 The area response change between the LCS and the most recent valid CAL 3 analysis for each of the internal standards must be less than or equal to ±40 percent.

6.12.5.5 The retention time shift between the LCS and the most recent valid CAL 3 analysis for each of the internal standards must be within ±20.0 seconds.

6.12.6 Corrective Action

6.12.6.1 If the technical acceptance criteria for the internal standards are not met, check calculations and instrument performance.

It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria.

6.12.6.2 The laboratory may not submit data from an SDG until all the LCS technical acceptance criteria are met. LCS contamination from laboratory sources or any LCS analyzed not meeting all the technical acceptance criteria will require analysis of additional LCS cartridges at no additional cost to the Agency.

6.12.6.3 LCS acceptance criteria MUST be met before any field samples, performance evaluation (PE) samples, or required blanks are analyzed. Any samples or required blanks analyzed when the LCS technical acceptance criteria have not been met will require analysis of additional LCS cartridges at no additional cost to the Agency.

6.12.7 Documentation

Reporting requirements are listed in Exhibit B. Laboratory Control Sample analysis data are reported on Form III-AASV. Internal standard area and RT shall be tabulated on Form VII-AASV, and surrogate recoveries are reported on Form IX-AASV.

6.13 SAMPLE ANALYSIS

6.13.1 Summary

The sample extract from Section 4 is analyzed by GC/MS and quantitated by the internal standard method.

6.13.2 Frequency

6.13.2.1 Before samples can be analyzed, the instrument must meet the GC/MS tuning and initial calibration or continuing calibration technical acceptance criteria.

6.13.2.2 If there is time remaining in the 12-hour time period with a valid initial calibration or continuing calibration, samples may be analyzed in the GC/MS system that meets the instrument performance check criteria.

6.13.3 Procedure

6.13.3.1 Set up the GC/MS system under the conditions outlined in section 6.6.

6.13.3.2 All sample extracts must be allowed to warm to ambient temperature (approximately 1 hour) before analysis. All sample extracts must be analyzed under the same instrumental conditions as

the calibration standards.

6.13.3.3 Add the internal standard spiking solution to the 1.0 mL extract to contain 40 ng/μL. For sample dilutions, add an appropriate amount of the internal standard spiking solution to maintain the concentration of the internal standards at 40 ng/μL in the diluted extract.

6.13.3.4 Inject 1.0 μL of sample extract into the GC/MS, and start data acquisition.

6.13.3.5 When all semivolatile target compounds have eluted from the GC, terminate the MS data acquisition and store data files on the data system storage device. Use appropriate data output software to display full range mass spectra and SICPs.

6.13.4 Calculations

6.13.4.1 Calculate target compound concentrations using the following equation:

$$Concentration\ (ug/cartridge) = \frac{A_x I_s V_t D_f}{A_{is} V_i RRF} \qquad \text{Eq. D/SV-12}$$

where: A_x = area response for the compound to be measured. The primary quantitation ions for the target compounds, internal standards, and surrogates are listed in Table D/SV-8 and D/SV-9;

A_{is} = area response for the internal standard. The target compounds are listed with their associated internal standard in Table D/SV-3;

I_s = amount of internal standard, in micrograms (μg), spiked into the total sample extract;

RRF = the RRF from the most recent continuing calibration or CAL 3;

V_i = volume of extract injected in microliters (μL);

V_t = volume of final extract in microliters (μL); and

D_f = dilution factor for the extract. If there was no dilution, D_f equals 1. If the sample was diluted, the D_f is greater than 1.

NOTE: Total PCB concentration in each sample extract is obtained by summing isomer group concentrations.

6.13.4.2 If the volume of air sampled is known to the laboratory, the above equation becomes:

$$Concentration\ (ng/m^3) = \frac{A_x I_s V_t D_f}{A_{is} V_o V_i RRF} \times \frac{1000\ ng}{ug} \qquad \text{Eq. D/SV-13}$$

where: V_o = volume of air sampled (m^3), STP.

6.13.4.3 The equation in section 6.13.6.1 is also used for calculating the concentrations of the non-target compounds. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the non-target compound to be measured (A_x) and the internal standard (A_{is}). Associate the nearest internal standard free of interferences with the non-target compound to be measured. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated ("J") (estimated, due to lack of a compound-specific response factor) and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns. This estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns.

6.13.4.4 Surrogate Percent Recovery (%R): Calculate the surrogate percent recovery using the following equation:

$$\%R = \frac{Q_d}{Q_a} \times 100 \qquad \text{Eq. D/SV-14}$$

where: Q_d = Quantity determined by analysis; and
Q_a = Quantity added to sample/blank.

6.13.4.5 Percent Area Response Change (%ARC): Calculate the percent area response change (%ARC) for the sample/blank analysis compared to the most recent CAL 3 analysis for each of the internal standard compounds using the following equation:

$$\%ARC = \frac{A_s - A_x}{A_x} \times 100 \qquad \text{Eq. D/SV-15}$$

where: %ARC = percent area response change;
A_s = area response of the internal standard in the sample/blank analysis; and
A_x = area response of the internal standard in the most recent CAL 3 analysis.

The %ARC for the internal standard must not exceed ±40 percent.

6.13.4.6 Internal Standard Retention Time Shift (RTS): Calculate the retention time shift (RTS) between the sample/blank analysis and the most recent CAL 3 analysis for each of the internal standards using the following equation:

$$RTS = RT_s - RT_x \qquad \text{Eq. D/SV-16}$$

where: RT_s = retention time of the IS in the sample: and
RT_x = retention time of the IS in the most recent CAL 3 analysis.

6.13.5 **Technical Acceptance Criteria**

6.13.5.1 All target compound concentrations must not exceed the upper limit of the initial calibration range and no compound ion (excluding the compound peaks in the solvent front) may saturate the detector.

6.13.5.2 Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 seconds from the latest continuing calibration standard or CAL 3 if samples are analyzed in the same 12-hour sequence as the initial calibration, the chromatographic system must be inspected for malfunctions, and corrections made as required. The SICP of the internal standards must be monitored and evaluated for each field and QC sample. The criteria are described in detail in the instructions for FORM VII-AASV, Internal Standard Area Summary. If the SICP area for any internal standard changes by more than a factor of ±40 percent, the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required.

6.13.5.3 The percent surrogate recoveries must be within the limits specified as follows:

Surrogate	Recovery Limits
Anthracene-d_{10}	25-150%
Benzo(a)pyrene-d_{12}	25-150%
Nitrobenzene-d_5	25-150%
2-Fluorobiphenyl	25-150%
p-Terphenyl-d_{14}	25-150%
2,4,6-Tribromophenol	25-150%
2-Fluorophenol	10-150%
Phenol-d_5	25-150%

6.13.5.4 When target compounds are below CRQLs but the spectrum meets the identification criteria, report the concentration with a "J." For example, if the CRQL is 3 ng and concentration of 1 ng is calculated, report as "1J."

6.13.6 Corrective Action

6.13.6.1 If the sample technical acceptance criteria for the surrogates and internal standards are not met, check calculations, surrogate and internal standard solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and internal standard technical acceptance criteria.

6.13.6.2 If the Contractor needs to analyze more than one (1) sample dilution to have all the target compounds within the initial calibration range and to have all compound ions not saturating the detector (excluding the peaks in the solvent front), contact SMO. SMO will contact the Region for instructions.

6.13.6.3 Sample analysis technical acceptance criteria MUST be met before data are reported. Samples contaminated from laboratory sources, or associated with a contaminated method blank or any samples analyzed not meeting the technical acceptance criteria will require reanalysis at no additional cost to the Agency.

6.13.6.4 Sample reruns performed as a result of suspected matrix interferences beyond the scope of the method will be reviewed on a case-by-case basis for payment purposes by the Project Officer.

6.13.6.5 The samples or standards with SICP areas outside the limits must be reanalyzed. If corrections are made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that meets the SICP criteria. After corrections are made, the reanalysis of samples analyzed while the system was malfunctioning is required.

6.13.6.6 If after reanalysis, the SICP areas for all internal standards are inside the contract limits (±40 percent), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with SICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

6.13.6.7 If the reanalysis of the sample does not solve the problem, i.e., the SICP areas are outside the contract limits for both analyses, then submit the SICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the SDG Narrative all inspection and corrective actions taken.

6.13.7 **Documentation**

Reporting requirements are listed in Exhibit B. Sample analysis results are reported on Form I-AASV, Analysis Data Sheet. Surrogate recoveries are reported on Form IX-AASV, and tentatively identified compounds (TICs) are reported on Form I-AASV-TIC. Internal standard area and RT shall be tabulated on Form VII-AASV.

6.14 **PERFORMANCE EVALUATION (PE) SAMPLES**

6.14.1 **Summary**

6.14.1.1 Performance evaluation (PE) samples will assist the Agency in monitoring laboratory performance. The Laboratory will not be informed as to which compounds are contained in the PE samples or the concentrations.

6.14.1.2 The PE sample containing known concentrations of analytes is analyzed by the laboratory to demonstrate that it can obtain acceptable identifications and measurements with procedures used to analyze environmental samples containing the same or similar analytes. Analyte and their concentrations are unknown by the analyst.

6.14.2 **Frequency**

The Laboratory shall extract, analyze, and report the results of the PE sample once per SDG, if available.

6.14.3 **Procedure**

6.14.3.1 The laboratory will receive PE samples on PUF/XAD-2 cartridges from the Agency. The samples may come with special instructions concerning the extraction procedure required for the PE samples.

6.14.3.2 Extract and concentrate the PE sample using the procedure described in Section 4. Add the surrogate and internal standards solution to the PE sample solution. Analyze the PE sample as described in section 6.13.

6.14.4 **Calculations**

See section 6.13 for equations necessary for calculations.

6.14.5 **Technical Acceptance Criteria**

6.14.5.1 The PE sample must be analyzed on a GC/MS system meeting the DFTPP tuning, initial calibration, and continuing calibration technical acceptance criteria at the required frequency.

6.14.5.2 The PE sample must be extracted and concentrated according to Section 4.

6.14.5.3 The PE sample must be prepared and analyzed with a method blank that met the blank technical acceptance criteria.

6.14.5.4 The percent recovery for each of the surrogates must be within the acceptance windows listed in section 6.13.5.3.

6.14.5.5 The area response change between the PE sample and the most recent continuing calibration check standard analysis for each of the internal standards must be within ±40 percent.

6.14.5.6 The retention time shift between the PE sample and the most recent CAL 3 analysis for each of the internal standards must be within ±20.0 seconds.

6.14.5.7 In addition to complying with the PE sample technical acceptance criteria, the laboratory will be responsible for correctly identifying and quantifying the compounds included in the PE sample. The Agency will notify the laboratory of unacceptable performance.

6.14.6 Corrective Action

6.14.6.1 If the PE sample technical acceptance criteria for the internal standard and surrogates are not met, check calculations, standard solutions and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria.

6.14.6.2 Specifically, the Laboratory must meet the following criteria associated with the PE sample:

- Minimum detection limits listed in Exhibit C;
- Replicate precision within ±30 percent RSD; and
- Audit accuracy of less than or equal to 30 percent for identified analytes in PE sample.

6.14.6.3 The PE sample technical acceptance criteria **MUST** be met before sample data are reported if the PE sample is provided with the SDG. Also, the Contractor must demonstrate acceptable performance for compound identification and quantification. If the Contractor fails to meet the PE sample technical acceptance criteria or achieves a score of less than 75 percent, the Agency may take, but is not limited to the following actions: reduction of the number of samples, suspension of sample shipment, a site visit, a full data audit, and/or require the laboratory to analyze a remedial PE sample, and/or a

contract sanction, such as a Cure Notice.

6.14.7 Documentation

Reporting requirements are listed in Exhibit B. Performance evaluation (PE) sample results are reported on Form I-AASV. Surrogate recoveries are reported on Form IX-AASV.

6.15 QUALITATIVE ANALYSIS

6.15.1 Target Compounds

6.15.1.1 The compounds listed in the Target Compound List (TCL), Exhibit C, shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications:

- Elution of the sample compound at the same GC relative retention time as the standard compound, and
- Correspondence of the sample compound and standard compound mass spectra.

6.15.1.2 PCBs are identified and measured with a special software (available from Hewlett-Packard and Finnigan), using information found in Table D/SV-7 and the criteria presented in Table D/SV-4. The intensity ratio of the two major ions in the molecular cluster for each homolog is the major identification criteria used. A ±20 percent range in this ratio around the theoretical or expected value is considered acceptable. The labeled surrogates are examined for correct retention time and the absence of interference as shown by the ratio of selected ion abundances from the molecular ion cluster.

6.15.1.3 The identification and quantification extends from the calibration utilizing the relative response factors (RRFs) for each PCB homolog group with respect to the chrysene-d_{12} internal standard. The primary ion is used to calculate a response factor with respect to the primary ion of the internal standard. This response factor is used to quantify each member of a specific homolog group. Thus, reported PCB congener concentrations are corrected for the different instrumental sensitivities of each Cl_x (homolog) group. This method of quantitation does not, however, take into account the range of sensitivities that may occur within a single homolog series, e.g., among the 42 tetrachloro PCB isomers. Therefore, use the mean RRF calculated during initial calibration or the RRF calculated in the continuing calibration.

NOTE: For PCB analyses with automated data interpretation, a linear fit algorithm will produce erroneous concentration data.

6.15.1.4 Examine results obtained on the special PCB qualitative report (for individual components identified as PCBs) and the quantitation report (for pesticide analytes) and PCB isomer groups. Individual spectra should be examined and compared to appropriate spectra acquired during calibration. Report calculated values to two significant figures.

6.15.1.5 For establishing correspondence of the GC relative retention time, the same compound RRT must be within ±0.06 RRT units of the RRT of the standard compound. For reference, the standard must be run on the same shift as the sample. If coelution of interfering compounds prohibits accurate assignment of the sample compound RRT from the extracted ion current profile for the primary ion, the RRT must be assigned by using the total ion chromatogram.

6.15.1.6 For comparison of standard and sample compound mass spectra, mass spectra obtained on the Contractor's GC/MS are required. These standard spectra may be used for identification purposes, only if the Contractor's GC/MS meets the DFTPP daily tuning technical acceptance criteria. These standard spectra may be obtained from the analysis used to obtain reference relative retention times.

6.15.1.7 The guidelines for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10 percent must be present in the sample spectrum.
- The relative intensities of the major ions must agree within ±20 percent between the standard and sample spectra. (Example: for an ion with an abundance of 50 percent in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)
- Ions greater than 25 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should FAVOR FALSE POSITIVE. All compounds meeting the identification criteria must be reported with their spectra.
- If a compound cannot be verified by all of the above criteria, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantification.

6.15.2 **Non-target Compounds**

6.15.2.1 All non-target compounds with a response greater than 10 percent of the nearest internal standard shall be tentatively identified via a forward search of the NIST Mass Spectral Library. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search must not use normalization routines if those routines would misrepresent the library or unknown spectra when compared to each other.

6.15.2.2 The following are guidelines for tentative identification:

- Relative intensities of major ions in the reference spectrum (ions greater than 25 percent of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20 percent. (Example: For an ion with an abundance of 50 percent in the standard spectra, the corresponding sample ion abundance should be between 30 and 70 percent.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.
- If, in the technical judgement of the mass interpretation spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If a probable molecular weight can be distinguished, include it.

6.16 QUANTITATIVE ANALYSIS

6.16.1 Target Compound Quantitation

6.16.1.1 Target components identified shall be quantified by the internal standard method. The internal standard used for the target compounds are outlined in Table D/SV-3 or shall be the one nearest the retention time to that of a given analyte. The SICP area of characteristic ions of analytes listed in Table D/SV-9 are used for quantitation.

6.16.1.2 The relative response factor (RRF) from the daily continuing calibration standard analysis (or RRF of CAL 3 if the sample is analyzed in the same 12-hour sequence as the initial calibration) is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion.

6.16.1.3 A retention time window is calculated for each single component analyte and surrogate. Windows are established as ±0.01 RRT units of the retention time for the analyte in CAL 3 of the initial calibration or the continuing calibration.

6.16.1.4 Sample quantitation is performed by the data processing system for all desired ions of all target compounds. Target compounds are quantified according to the equation in section 6.13.4.1.

6.16.1.5 The computer must be able to print out peak number, m/e, scan #, time, relative retention time, area and amount.

6.16.1.6 Standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any standard changes by more than 30 seconds from the latest daily (12 hour) calibration, the chromatographic system must be inspected for malfunctions, and corrections made as required. The SICP of the internal standards must be monitored and evaluated for each sample and blank. If the SICP area for any internal standard changes by more than 40 percent, the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of duplicate samples analyzed while the system was malfunctioning is necessary.

6.16.2 Non-Target Compound Quantitation

6.16.2.1 An estimated concentration for non-target components tentatively identified shall be quantified by the standard method.

The nearest internal standard free of interferences shall be used.

6.16.2.2 The formula for calculating concentrations is the same as in section 6.13.4.1. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the standard. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated (i.e., flagged "J"). This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

6.16.2.3 An estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns. This estimated concentration must be reported for ten highest tentatively identified compounds as well as those identified as unknowns.

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TABLE D/SV-1a

CONCENTRATION OF CALIBRATION SOLUTIONS

Target Compound	Solution†	Concentration, ng/on column				
		CAL 1	CAL 2	CAL 3††	CAL 4	CAL 5
Acenaphthene	B	10	25	50	100	200
Acenaphthylene	B	10	25	50	100	200
Acetophenone	B	10	25	50	100	200
Aldicarb	A	10	25	50	100	200
Aldrin	B	10	25	50	100	200
Aniline	B	10	25	50	100	200
Anthracene	B	10	25	50	100	200
Bendiocarb	A	10	25	50	100	200
Benzidine	B	10	25	50	100	200
Benz(a)anthracene	B	10	25	50	100	200
Benzo(a)Pyrene	B	10	25	50	100	200
Benzo(b)fluoranthene	B	10	25	50	100	200
Benzo(e)pyrene	B	10	25	50	100	200
Benzo(g,h,i)perylene	B	10	25	50	100	200
Benzo(k)fluoranthene	B	10	25	50	100	200
Benzyl alcohol	A	10	25	50	100	200
alpha-BHC	B	10	25	50	100	200
gamma-BHC (Lindane)	B	10	25	50	100	200
p-Biphenylamine	B	10	25	50	100	200
Bis(n-butyl)phthalate	B	10	25	50	100	200
Butylbenzylphthalate	B	10	25	50	100	200
Captan	A	10	25	50	100	200
alpha-Chlordane	A	10	25	50	100	200
gamma-Chlordane	A	10	25	50	100	200
4-Chloro-3-methylphenol	A	10	25	50	100	200
4-Chloroaniline	B	10	25	50	100	200
Bis(2-chloroethoxy)methane	A	10	25	50	100	200
Bis(2-chloroethyl)ether	B	10	25	50	100	200
Chlorothalonil	B	10	25	50	100	200
Chlorpyrifos	A	10	25	50	100	200
Chrysene	B	10	25	50	100	200
Dacthal (DCPA)	A	10	25	50	100	200
4,4'-DDD	B	10	25	50	100	200
4,4'-DDE	B	10	25	50	100	200
4,4'-DDT	B	10	25	50	100	200

† Recommended solutions A and B may be used as separate calibration mixtures or mixed just prior to injection.

†† CAL 3 is used for continuing calibration.

TABLE D/SV-1a (continued)

CONCENTRATION OF CALIBRATION SOLUTIONS

Target Compound	Solution†	Concentration, ng/on column CAL 1	CAL 2	CAL 3††	CAL 4	CAL 5
Diazinon	A	10	25	50	100	200
Dibenz(a,h)anthracene	B	10	25	50	100	200
Dichlorvos (DDVP)	A	10	25	50	100	200
Dicofol	A	10	25	50	100	200
Dieldrin	B	10	25	50	100	200
Diethyl Phthalate	B	10	25	50	100	200
2,4-Dimethyl phenol	A	10	25	50	100	200
Dimethylphthalate	B	10	25	50	100	200
4,6-Dinitro-2-methylphenol	A	10	25	50	100	200
2,4-Dinitrophenol	A	10	25	50	100	200
2,4-Dinitrotoluene	A	10	25	50	100	200
Endosulfan I	B	10	25	50	100	200
Endosulfan II	B	10	25	50	100	200
Endrin	B	10	25	50	100	200
Endrin Aldehyde	B	10	25	50	100	200
Endrin ketone	B	10	25	50	100	200
Bis(2-ethylhexyl)phthalate	B	10	25	50	100	200
Fluoranthene	B	10	25	50	100	200
Fluorene	B	10	25	50	100	200
Folpet	A	10	25	50	100	200
Heptachlor	B	10	25	50	100	200
Heptachlor Epoxide	B	10	25	50	100	200
Hexachlorobenzene	A	10	25	50	100	200
Hexachlorocyclopentadiene	A	10	25	50	100	200
Hexachloroethane	A	10	25	50	100	200
Indeno(1,2,3-c,d)pyrene	B	10	25	50	100	200
Isophorone	A	10	25	50	100	200
Methoxychlor	B	10	25	50	100	200
2-Methylnaphthalene	A	10	25	50	100	200
2-Methyl phenol	A	10	25	50	100	200
4-Methyl phenol	A	10	25	50	100	200
Mirex	B	10	25	50	100	200
Naphthalene	B	10	25	50	100	200
2-Naphthylamine	B	10	25	50	100	200
2-Nitroaniline	B	10	25	50	100	200

† Recommended solutions A and B may be used as separate calibration mixtures or mixed just prior to injection.

†† CAL 3 is used for continuing calibration.

TABLE D/SV-1a (continued)

CONCENTRATION OF CALIBRATION SOLUTIONS

Target Compound	Solution†	Concentration, ng/on column CAL 1	CAL 2	CAL 3††	CAL 4	CAL 5
3-Nitroaniline	B	10	25	50	100	200
Nitrobenzene	A	10	25	50	100	200
4-Nitrodiphenyl	A	10	25	50	100	200
2-Nitrophenol	A	10	25	50	100	200
4-Nitrophenol	A	10	25	50	100	200
Bis(n-octyl)phthalate	B	10	25	50	100	200
Oxychlordane	A	10	25	50	100	200
Parathion	A	10	25	50	100	200
Pentachlorobenzene	B	10	25	50	100	200
Pentachlorophenol	A	10	25	50	100	200
cis/trans-Permethrin	A	10	25	50	100	200
Phenanthrene	B	10	25	50	100	200
Phenol	A	10	25	50	100	200
o-Phenylphenol	A	10	25	50	100	200
Propoxur	A	10	25	50	100	200
Pyrene	B	10	25	50	100	200
Resmethrin	A	10	25	50	100	200
Ronnel	A	10	25	50	100	200
2,4,5-Trichlorophenol	A	10	25	50	100	200
2,4,6-Trichlorophenol	A	10	25	50	100	200
PCB Calibration Congeners†††						
Cl_1	A	10	25	50	100	200
Cl_2	A	10	25	50	100	200
Cl_3	A	10	25	50	100	200
Cl_4	A	10	25	50	100	200
Cl_5	A	10	25	50	100	200
Cl_6	A	10	25	50	100	200
Cl_7	A	10	25	50	100	200
Cl_8	A	10	25	50	100	200
Cl_9	A	10	25	50	100	200
Cl_{10}	A	10	25	50	100	200

† Recommended solutions A and B may be used as separate calibration mixtures or mixed just prior to injection.

†† CAL 3 is used for continuing calibration.

††† These congeners are used to quantitate PCBs using the special software.

TABLE D/SV-1b

APPROXIMATE CONCENTRATION OF INTERNAL STANDARDS AND SURROGATE COMPOUNDS IN CALIBRATION SOLUTIONS

Compound Name		Concentration, ng/on column CAL 1	CAL 2	CAL 3	CAL 4	CAL 5
Internal Standards						
1,4-Dichlorobenzene-d_4		40	40	40	40	40
Naphthalene-d_8		40	40	40	40	40
Acenaphthene-d_{10}		40	40	40	40	40
Phenanthrene-d_{10}		40	40	40	40	40
Chrysene-d_{12}		40	40	40	40	40
Perylene-d_{12}		40	40	40	40	40
Surrogate Compounds						
Anthracene-d_{10}		10	25	50	100	200
Benzo(a)pyrene-d_{12}		10	25	50	100	200
2,4,6-Tribromophenol		10	25	50	100	200
2-Fluorophenol		10	25	50	100	200
Phenol-d_5		10	25	50	100	200
Nitrobenzene-d_5	Pre-sampling	10	25	50	100	200
2-Fluorobiphenyl	Pre-sampling	10	25	50	100	200
p-Terphenyl-d_{14}	Pre-sampling	10	25	50	100	200

TABLE D/SV-2

GC/MS OPERATING CONDITIONS

Activity	Conditions
Gas Chromatography	
Column	J&W Scientific, DB-5 crosslinked 5% phenylmethyl silicone (30 m x 0.32 mm, 1.0 μm film thickness) or equivalent
Carrier Gas	Helium, velocity between 28-29 cm^3/sec at 250°C
Injection Volume	1 μL, Grob-type, splitless
Injector Temperature:	250-300°C
Temperature Program	
Initial Column Temp.	50°C
Initial Hold Time	4 ± 0.1 min.
Program	8°C/min
Final Temperature	280°C
Final Hold Time	15 min. or until all compounds of interest have eluted
Mass Spectrometer	
Transfer Line Temperature:	250-300°C
Source Temperature:	According to Manufacturer's Specifications.
Electron Energy:	70 volts (nominal)
Ionization Mode:	EI
Mass Range:	35 to 500 amu, full range data acquisition (SCAN) mode
Scan Time:	At least 5 scans per peak, not to exceed 1 second per scan.

TABLE D/SV-3
INTERNAL STANDARDS FOR SEMIVOLATILE GC/MS ANALYSIS

Naphthalene-d_8

Acetophenone
p-Biphenylamine
Bis(2-chloroethoxy)methane
4-Chloro-3-methylphenol
4-Chloroaniline
2,4-Dimethyl phenol
Dichlorvos (DDVP)
Isophorone
2-Methylnaphthalene
Naphthalene
Nitrobenzene
Nitrobenzene-d_5
2-Nitrophenol
Nitrophenols (mixed)

Acenaphthene-d_{10}

Acenaphthene
Acenaphthylene
Diethyl Phthalate
Dimethylphthalate
2,4-Dinitrophenol
2,4-Dinitrotoluene
2-Fluorobiphenyl
Fluorene
Hexachlorocyclopentadiene
2-Naphthylamine
2-Nitroaniline
3-Nitroaniline
o-Phenylphenol
Propoxur
2,4,5-Trichlorophenol
2,4,6-Tribromophenol
2,4,6-Trichlorophenol

1,4-Dichlorobenzene-d_4

Aldicarb
Aniline
Benzyl alcohol
Bis(2-chloroethyl)ether
2-Fluorophenol
Hexachloroethane
2-Methyl phenol
4-Methyl phenol
Phenol-d_5

Phenanthrene-d_{10}

Aldrin
alpha-BHC
gamma-BHC (Lindane)
Anthracene
Anthracene-d_{10}
Bendiocarb
Bis(n-butyl)phthalate
Butylbenzylphthalate
Captan
Chlorothalonil
Chlorpyrifos
Dacthal (DCPA)
Diazinon
4,6-Dinitro-2-methylphenol
Endosulfan I
Fluoranthene
Folpet
Heptachlor
Heptachlor Epoxide
Hexachlorobenzene
Oxychlordane
4-Nitrodiphenyl
Parathion
Pentachlorobenzene
Pentachlorophenol
Phenanthrene
Polybrominated biphenyls
Ronnel

TABLE D/SV-3 (continued)

INTERNAL STANDARDS FOR SEMIVOLATILE GC/MS ANALYSIS

Chrysene-d_{12}

Benzidine
Benzo(a)anthracene
Bis(2-ethylhexyl)phthalate
alpha-Chlordane
gamma-Chlordane
Chrysene
4,4'-DDD
4,4'-DDE
4,4'-DDT
$^{13}C_{12}$-4,4'-DDT
Dicofol
Dieldrin
Endosulfan II
Endrin
Endrin Aldehyde
Methoxychlor
cis/trans-Permethrin
Pyrene
Resmethrin
p-Terphenyl-d_{14}
Monochlorobiphenyls
Dichlorobiphenyls
Trichlorobiphenyls
Tetrachlorobiphenyls
Pentachlorobiphenyls
Heptachlorobiphenyls
Hexachlorobiphenyls
Octachlorobiphenyls
Nonachlorobiphenyls
Decachlorobiphenyl

Perylene-d_{12}

Benzo(a)pyrene
Benzo(a)pyrene-d_{12}
Benzo(b)fluoranthene
Benzo(e)pyrene
Benzo(g,h,i)perylene
Benzo(k)fluoranthene
Bis(n-octyl)phthalate
Dibenzo(a,h)anthracene
Endrin ketone
Indeno(1,2,3-c,d)pyrene
Mirex

TABLE D/SV-4

PCB CONGENERS USED AS CALIBRATION STANDARDS

PCB Isomer Group	Congener Number[a]	Chlorine Substitution
Concentration Calibration Standard		
Monochlorobiphenyl	1	2
Dichlorobiphenyl	5	2,3
Trichlorobiphenyl	29	2,4,5
Tetrachlorobiphenyl	50	2,2',4,6
Pentachlorobiphenyl	87	2,2',3,4,5'
Hexachlorobiphenyl	154	2,2'4,4',5,6'
Heptachlorobipehnyl	188	2,2'3,4',5,6,6'
Octachlorobiphenyl	200	2,2'3,3',4,5',6,6'
Nonachlorobiphenyl[b]	-	---
Decachlorobiphenyl	209	2,2'3,3',4,4',5,5',6,6'

[a] Numbered according to the system of Ballschmiter and Zell. (see section 3.7, Citation 2).
[b] Decachlorobiphenyl is used as the calibration congener for both nona-and decachlorobiphenyl isomer groups.

TABLE D/SV-5

DFTPP KEY IONS & ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	Less than 2% of mass 69
69	Present
70	Less than 2% of mass 69
127	25-75% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	Greater than 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

TABLE D/SV-6
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF SEMIVOLATILE TARGET COMPOUNDS

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
2-Methylnaphthalene	0.400	25.0	25.0
2-Methylphenol	0.700	25.0	25.0
2-Nitrophenol	0.100	25.0	25.0
2,4-Dimethylphenol	0.200	25.0	25.0
2,4-Dinitrotoluene	0.200	25.0	25.0
2,4,5-Trichlorophenol	0.200	25.0	25.0
2,4,6-Trichlorophenol	0.200	25.0	25.0
4-Chloro-3-methylphenol	0.200	25.0	25.0
4-Methylphenol	0.600	25.0	25.0
Acenaphthylene	1.300	25.0	25.0
Acenaphthene	0.800	25.0	25.0
Anthracene	0.700	25.0	25.0
Benz(a)anthracene	0.800	25.0	25.0
Benzo(a)pyrene	0.700	25.0	25.0
Benzo(b)fluoranthene	0.700	25.0	25.0
Benzo(g,h,i)perylene	0.500	25.0	25.0
Benzo(k)fluoranthene	0.700	25.0	25.0
bis(-2-Chloroethoxy)methane	0.300	25.0	25.0
bis(-2-Chloroethyl)ether	0.700	25.0	25.0
Chrysene	0.700	25.0	25.0
Dibenzo(a,h)anthracene	0.400	25.0	25.0
Fluoranthene	0.600	25.0	25.0
Fluorene	0.900	25.0	25.0
Hexachlorobenzene	0.100	25.0	25.0
Hexachloroethane	0.300	25.0	25.0
Indeno(1,2,3-cd)pyrene	0.500	25.0	25.0
Isophorone	0.400	25.0	25.0
Naphthalene	0.700	25.0	25.0
Nitrobenzene	0.200	25.0	25.0
Pentachlorophenol	0.050	25.0	25.0
Phenanthrene	0.700	25.0	25.0
Phenol-d_5	0.800	25.0	25.0
Pyrene	0.600	25.0	25.0
Terphenyl-d_{14}	0.500	25.0	25.0

TABLE D/SV-6
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF SEMIVOLATILE TARGET COMPOUNDS
(continued)

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Naphthalene	0.700	25.0	25.0
Acenaphthylene	1.300	25.0	25.0
Acenaphthene	0.800	25.0	25.0
Fluorene	0.900	25.0	25.0
Phenanthrene	0.700	25.0	25.0
Anthracene	0.700	25.0	25.0
Fluoranthene	0.600	25.0	25.0
Pyrene	0.600	25.0	25.0
Benz(a)anthracene	0.800	25.0	25.0
Chrysene	0.700	25.0	25.0
Benzo(b)fluoranthene	0.700	25.0	25.0
Benzo(k)fluoranthene	0.700	25.0	25.0
Benzo(a)pyrene	0.700	25.0	25.0
Indeno(1,2,3-cd)pyrene	0.500	25.0	25.0
Dibenz(a,h)anthracene	0.400	25.0	25.0
Benzo(g,h,i)perylene	0.500	25.0	25.0

TABLE D/SV-7
QUANTITATION, CONFIRMATION, AND INTERFERENCE CHECK IONS FOR PCBs, INTERNAL STANDARDS, AND SURROGATE COMPOUNDS

Analyte/ Internal Std	Quant. Ion	Confirm. Ion	Expected Ratio[a]	Accepted Ratio[a]
PCB Isomer Group				
Cl_1	188	190	3.0	2.5-3.5
Cl_2	222	224	1.5	1.3-1.7
Cl_3	256	258	1.0	0.8-1.2
Cl_4	292	290	1.3	1.1-1.5
Cl_5	326	324	1.6	1.4-1.8
Cl_6	360	362	1.2	1.0-1.4
Cl_7	394	396	1.0	0.8-1.2
Cl_8	430	428	1.1	0.9-1.3
Cl_9	464	466	1.3	1.1-1.5
Cl_{10}	498	500	1.1	0.9-1.3
Internal Standard (IS)				
Chrysene-d_{12}	240	241	5.1	4.3-5.9

[a] Ratio of quantitation ion to confirmation ion

TABLE D/SV-8

CHARACTERISTIC IONS FOR SURROGATES AND INTERNAL STANDARDS

Classification	Primary Ion	Secondary
Surrogates		
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2.4,6-Tribromophenol	330	332, 54
Nitrobenzene-d_5	82	128, 54
2-Fluorobiphenyl	172	171
p-Terphenyl-d_{14}	244	122, 212
Anthracene-d_{10}	188	
Benzo(a)pyrene-d_{12}	264	
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

TABLE D/SV-9

QUANTITATION IONS FOR TARGET COMPOUNDS

TARGET COMPOUND	PRIMARY	SECONDARY
Acenaphthene	153	152, 154
Acenaphthylene	152	151, 153
Acetophenone	100	77, 120
Aldicarb	144	86, 89, 58
Aldrin	66	263, 220
Aniline	93	66, 65
Anthracene	178	179, 176
Bendiocarb	151	126, 166, 223, 58, 51
Benzidine	184	-
Benz(a)anthracene	228	229, 226
Benzo(a)Pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(e)pyrene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzyl alcohol	108	107, 79
alpha-BHC	183	181, 109
gamma-BHC (Lindane)	183	181, 109
p-Biphenylamine	169	168, 170
Bis(n-butyl)phthalate	149	150,104
Butylbenzylphthalate	149	91, 206
Captan	79	119, 117, 77, 149
alpha-Chlordane	373	375, 377
gamma-Chlordane	373	375, 377
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Chlorothalonil	266	264, 268, 109, 124, 133
Chlorpyrifos	197	97, 199, 125, 258
Chrysene	228	226, 229
Dacthal (DCPA)	299	45, 44, 142, 221
4,4'-DDD	235	237, 165
4,4'-DDE	246	248, 176
4,4'-DDT	235	237, 165
Diazinon	137	179, 199, 93, 97
Dibenz(a,h)anthracene	278	139, 279
Dichlorvos (DDVP)	109	79, 185, 187, 202
Dicofol	139	111, 141, 250, 75, 140
Dieldrin	263	82, 81
Diethyl Phthalate	149	177, 150
2,4-Dimethyl phenol	107	121, 122

TABLE D/SV-9 (continued)

QUANTITATION IONS FOR TARGET COMPOUNDS

TARGET COMPOUND	PRIMARY	SECONDARY
Dimethylphthalate	163	194, 164
4,6-Dinitro-2-methylphenol	198	182, 77
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 182
Endosulfan I	195	339, 341
Endosulfan II	337	339, 341
Endrin	263	82, 81
Endrin Aldehyde	67	345, 250
Endrin Ketone	317	67, 319
Bis(2-ethylhexyl)phthalate	149	167, 279
Fluoranthene	202	101, 100
Fluorene	166	165, 167
Folpet	260	262, 130, 117, 104, 76
Heptachlor	100	272, 274
Heptachlor Epoxide	353	355, 351
Hexachlorobenzene	284	142, 249
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95, 138
Methoxychlor	227	228
2-Methylnaphthalene	142	144
2-Methylphenol	108	107
4-Methylphenol	108	107
Mirex	272	274, 270
Naphthalene	128	129, 127
2-Naphthylamine	143	115
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	108, 92
Nitrobenzene	77	123, 65
4-Nitrodiphenyl	199	152
2-Nitrophenol	139	65, 109
4-Nitrophenol	109	139,65
Bis(n-octyl)phthalate	149	-
Oxychlordane	115	57, 87, 117, 149, 121
Parathion	291	109, 97, 155, 137, 139
Pentachlorobenzene	250	252, 248
Pentachlorophenol	266	264, 268
cis/trans-Permethrin	183	
Phenanthrene	178	179, 176
o-Phenylphenol	170	169, 168, 141, 115
Propoxur	110	111, 41, 43, 81

TABLE D/SV-9 (continued)

QUANTITATION IONS FOR TARGET COMPOUNDS

TARGET COMPOUND	PRIMARY	SECONDARY
Pyrene	202	101, 100
Resmethrin	123	128, 143, 141, 171, 81
Ronnel	125	119, 93, 79, 57, 62
2,4,5-Trichlorophenol	196	198, 200
2,4,6-Trichlorophenol	196	198, 200
PCB Calibration Congeners		
Cl_1	188	189, 190
Cl_2	222	224, 223, 225
Cl_3	256	258, 260, 257
Cl_4	292	290, 294, 296, 291
Cl_5	326	328, 324, 330
Cl_6	360	362, 358, 364, 361
Cl_7	394	396, 398, 392, 400
Cl_8	430	428, 432, 426, 434, 431
Cl_9	(464)	(462, 466, 468, 460)
Cl_{10}	498	500, 496, 502, 494, 504

[a] Congener Number

TABLE D/SV-10

KNOWN RELATIVE ABUNDANCES OF IONS IN PCB MOLECULAR ION CLUSTERS

m/z	Relative Intensity
Monochlorobiphenyls	
188	100
189	13.5
190	33.4
192	4.41
Dichlorobiphenyls	
222	100
223	13.5
224	66.0
225	8.82
226	11.2
227	1.44
Trichlorobiphenyls	
256	100
257	13.5
258	98.6
259	13.2
260	32.7
261	4.31
262	3.73
263	0.47
Tetrachlorobiphenyls	
290	76.2
291	10.3
292	100
293	13.4
294	49.4
295	6.57
296	11.0
297	1.43
298	0.95
Pentachlorobiphenyls	
324	61.0
325	8.26
326	100
327	13.5
328	65.7
329	8.78
330	21.7
331	2.86
332	3.62
333	0.47
334	0.2
Hexachlorobiphenyls	
358	50.9
359	6.89
360	100
361	13.5
362	82.0
363	11.0
364	36.0
365	4.77
366	8.92
367	1.17
368	1.20
369	0.15
Heptachlorobiphenyls	
392	43.7
393	5.91
394	100
395	13.5
396	98.3
397	13.2
398	53.8
399	7.16
400	17.7
401	2.34
402	3.52
403	0.46
404	0.40
Octachlorobiphenyls	
426	33.4
427	4.51
428	87.3
429	11.8
430	100
431	13.4
432	65.6
433	8.76
434	26.9
435	3.57
436	7.10
437	0.93
438	1.18
439	0.15
440	0.11
Nonachlorobiphenyls	
460	26.0
461	3.51
462	76.4
463	76.4
464	100
465	13.4
466	76.4
467	10.2
468	37.6
469	5.00
470	12.4
471	1.63
472	2.72
473	0.35
474	0.39
Decachlorobiphenyl	
494	20.8
495	2.81
496	68.0
497	9.17
498	100
499	13.4
500	87.3
501	11.7
502	50.0
503	6.67
504	19.7
505	2.61
506	5.40
507	0.71
508	1.02
509	0.13

TABLE D/SV-11

LABORATORY CONTROL SAMPLE COMPUNDS

Target Compound	%Recovery QC Limit
Bendiocarb	
Benzo(b)fluoranthene	
Benzo(k)fluoranthene	
Benzyl alcohol	
Butylbenzylphthalate	
Bis(2-chloroethoxy)methane	
Bis(2-chloroethyl)ether	
Decachlorobiphenyl	
Diazinon	
Dibenz(a,h)anthracene	
Dicofol	
Diethyl Phthalate	
2,4-Dimethyl phenol	
Endrin	
Endrin ketone	
Fluoranthene	
Fluorene	
Folpet	
Hexachlorocyclopentadiene	
Hexachloroethane	
Indeno(1,2,3-c,d)pyrene	
2-Methylnaphthalene	
2-Methyl phenol	
3-Nitroaniline	
Nitrobenzene	
4-Nitrophenol	
Nonachlorobiphenyl	
Pentachlorophenol	

NOTE: Percent Recovery Limits are not established at this time. The appropriate values shall be determined from multi-laboratory studies conducted under SAS analyses.

Figure D/SV-1. Sampling Head Configuration for Semivolatiles

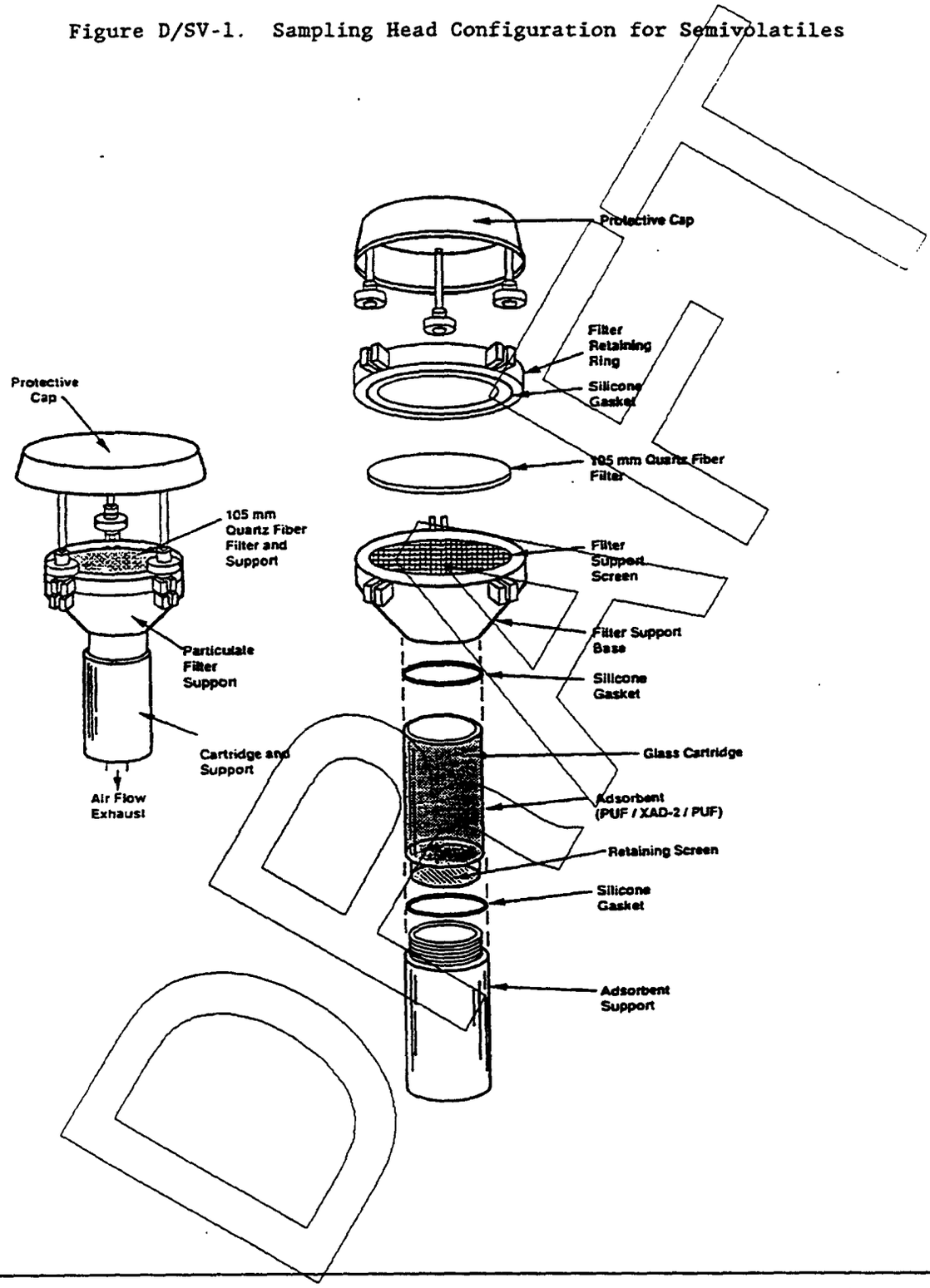

Figure D/SV-2. Configuration of the PUF/XAD-2/PUF Assembly

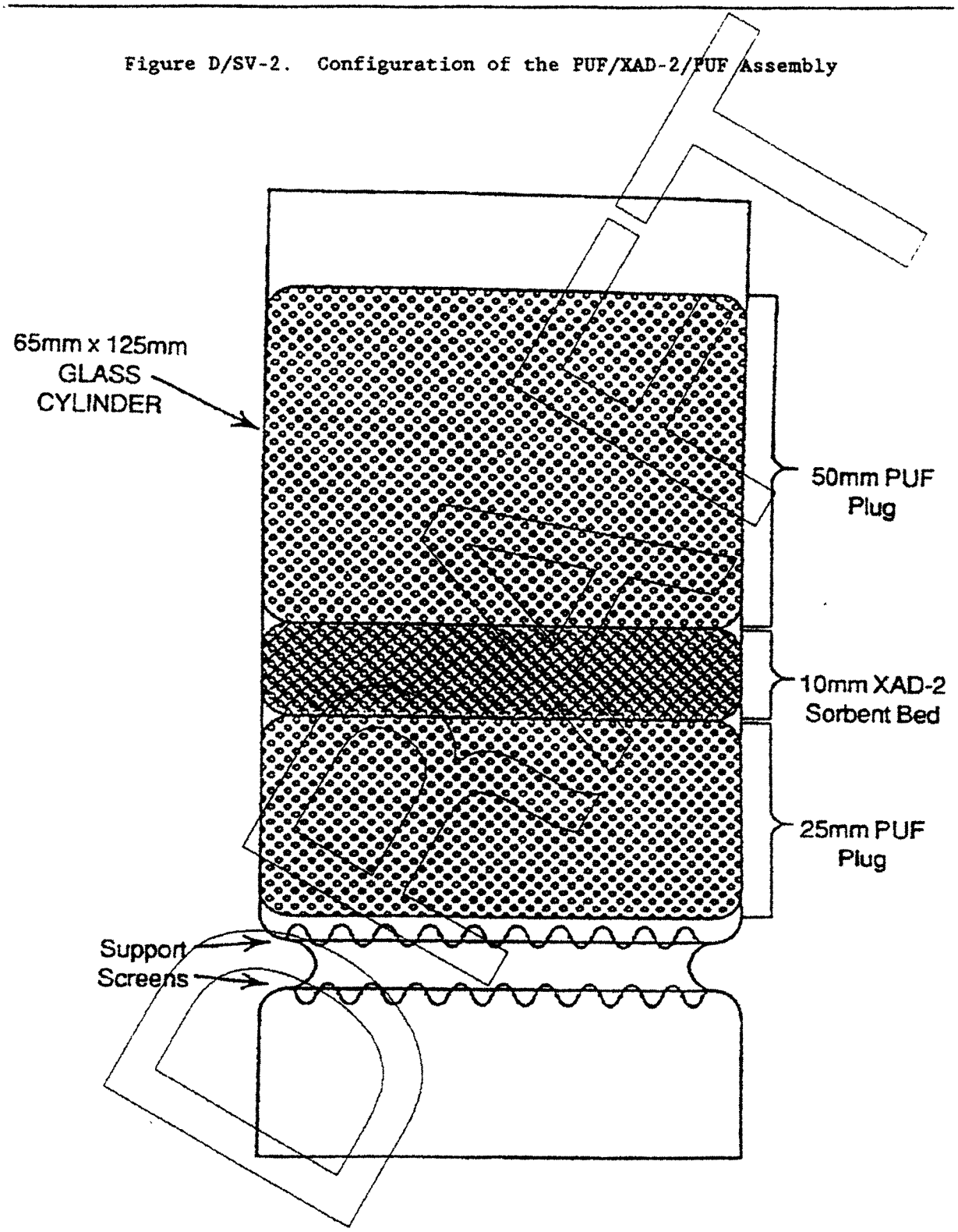

Figure D/SV-3. Analytical Scheme for the Analysis of Semivolatiles

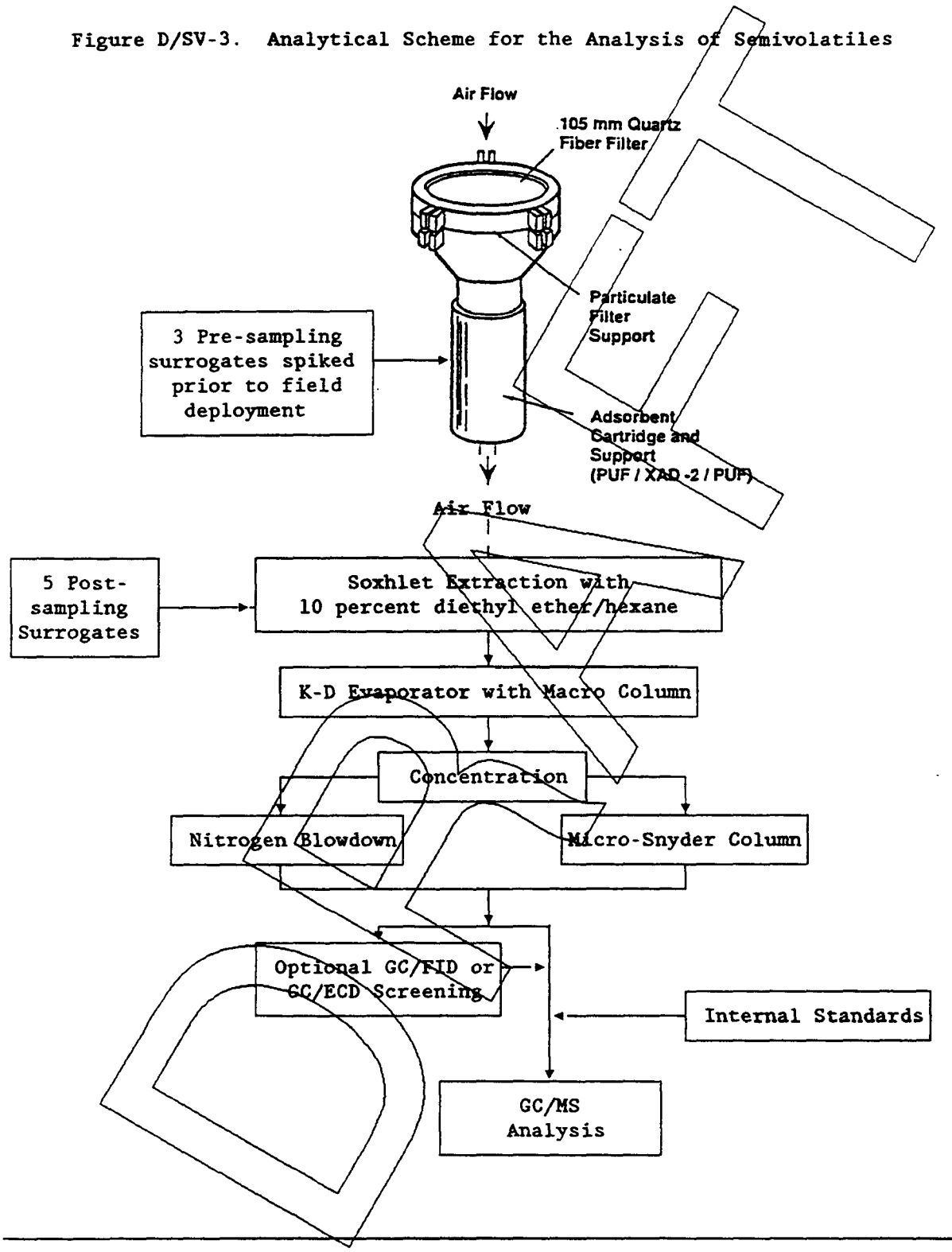

Figure D/SV-4. GC/MS Calibration and Analysis Schematic Diagram

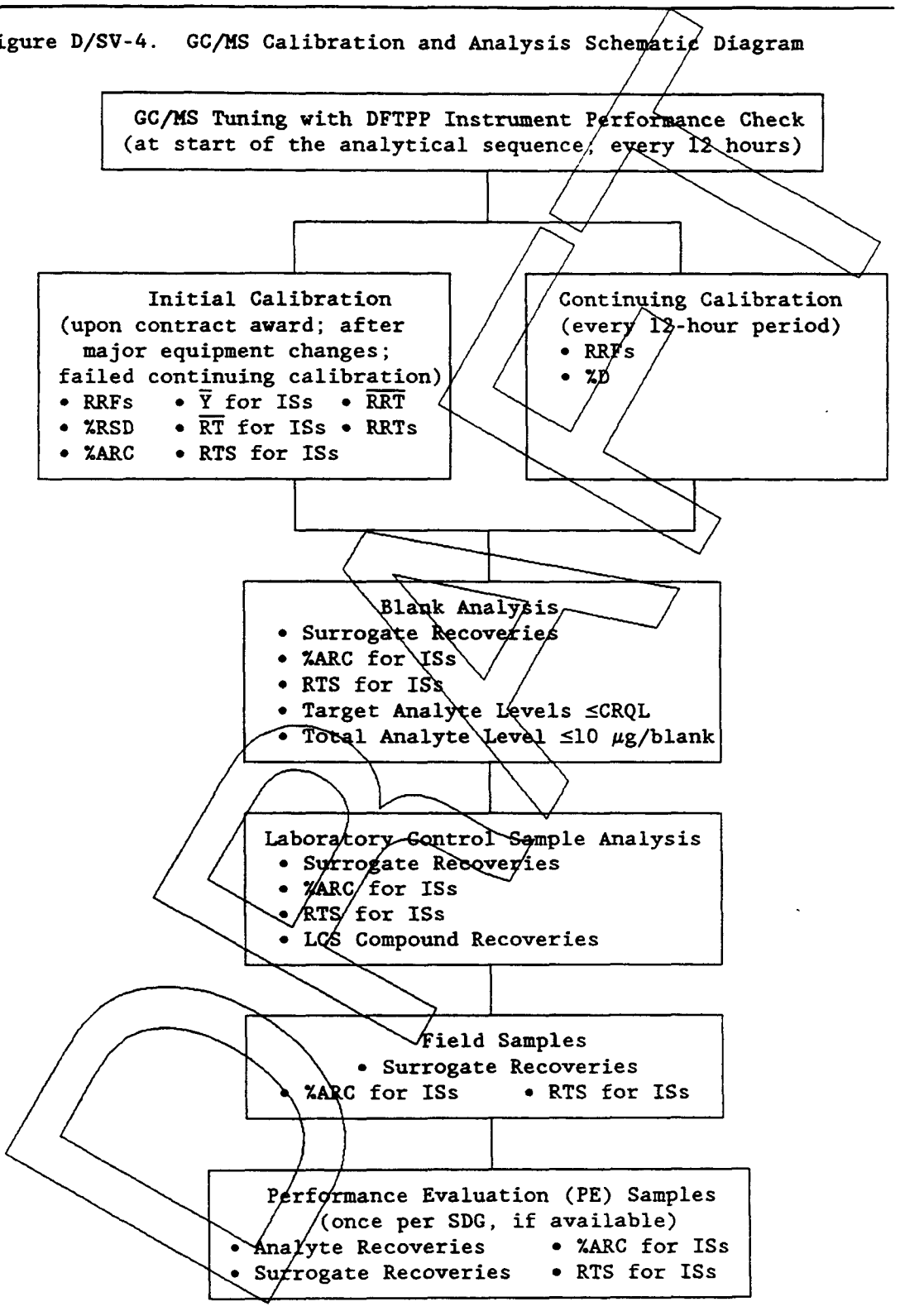

Figure D/SV-5. Semivolatiles Loading in PUF/XAD-2 Cartridges

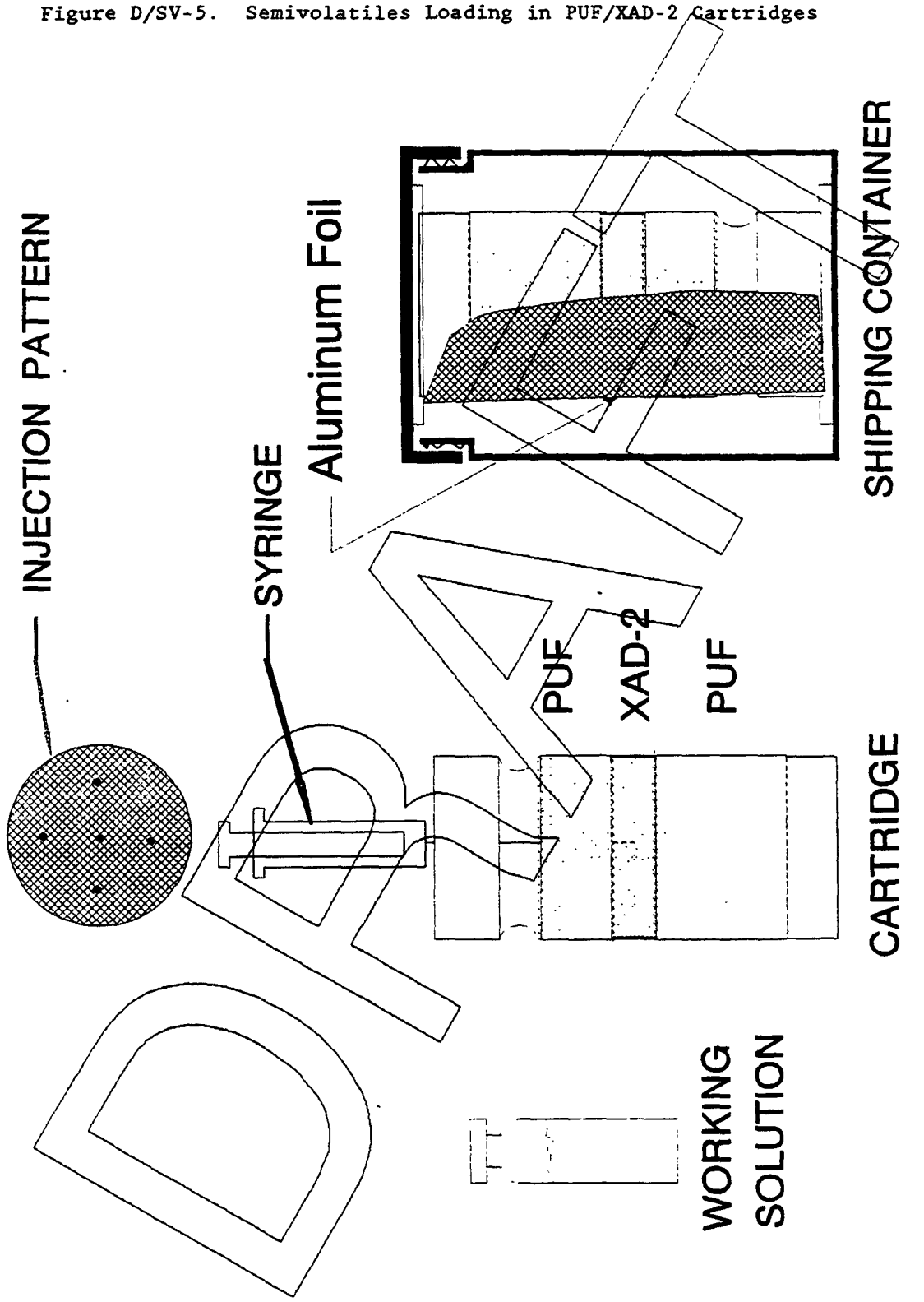

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

January, 1992

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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SECTION 1

INTRODUCTION

1.1 Quality assurance (QA) and quality control (QC) are integral parts of EPA's Contract Laboratory Program (CLP). The CLP QA program consists of management review and oversight at the planning, implementation, and completion stages of environmental data generation activities, to ensure that data provided are of the quality required. The CLP QC program includes those activities required as part of data generation to ensure that the data are of known and documented quality.

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

1.3 The purpose of this Exhibit is to describe 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 QA/QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These operations are designed to facilitate laboratory comparison by providing the EPA with comparable data from all Contractors. These requirements do not release the laboratory from maintaining its own QC checks on method and instrument performance.

1.4 Appropriate use of data generated under the great range of analytical conditions encountered in ambient air analyses requires reliance on the QC procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories participating in the CLP. However, the validation of these methods does not guarantee that they perform equally well for all samples collected under actual field conditions. Inaccuracies can result from causes such as sampling artifacts, equipment malfunctions, and human error. Therefore, the QC component of each method is indispensable.

1.5 The data acquired from QC procedures are used to estimate and evaluate analytical results and to determine the necessity for or the effect of corrective actions. The means used for evaluating the analytical results include quantitative and qualitative indicators of quality such as precision, accuracy, detection limit, and other quantitative and qualitative indicators. In addition, QC data give an overview of the activities required in an

integrated program to generate environmental data of known and documented quality required to meet defined objectives.

1.6 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, NEIC, and EMSL/LV. Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Performance evaluation samples provide an external QA reference for the program. A laboratory 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 contract laboratories through direct communications with the Administrative Project Officers (APOs) and Technical Project Officers (TPOs).

1.7 This Exhibit is not a guide to constructing QA project plans, QC systems, or a QA organization. It is, however, an explanation of the QC and QA requirements of the CLP. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a QA Plan and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides the Agency with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

1.8 To ensure that the product delivered by the Contractor meets the requirements of the contract and to improve interlaboratory data comparison, the Agency requires the following from the Contractor.

1.8.1 Development and implementation of a QA program, and documentation of the key elements of that QA program through a written QA Plan, as described in Section 2 of this Exhibit.

1.8.2 Preparation of and adherence to written Standard Operating Procedures (SOPs) as described in Section 5 of this Exhibit.

1.8.3 Adherence to the analytical methods and associated QC requirements specified in the contract.

1.8.4 Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical houses.

1.8.5 Participation in the analysis of laboratory performance evaluation (PE) samples, including adherence to corrective action procedures.

1.8.6 Participation in on-site laboratory evaluations, including adherence to corrective action procedures.

1.8.7 Submission of all raw data and pertinent documentation for Regional review.

1.8.8 Submission, upon request, of GC/MS tapes and applicable documentation for tape audits.

1.8.9 Submission for Agency review of all original documentation generated during sample analyses.

SECTION 2

QUALITY ASSURANCE PLANS

2.1 The Contractor shall establish a QA program with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

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

2.2.1 Maintain data integrity, validity, and usability.

2.2.2 Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility .

2.2.3 Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable.

2.2.4 Document all aspects of the measurement process in order to provide data that are technically sound and legally defensible.

2.3 The QAP must present, in specific terms, the policies, organization, objectives, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during On-Site Laboratory evaluation and upon written request by the Administrative Project Officer. Additional information relevant to the preparation of a QAP can be found in EPA and ASTM publications[2,4].

2.4 ELEMENTS OF A QUALITY ASSURANCE PLAN

2.4.1 The following key elements of the Contractor's quality assurance program shall be addressed in the QAP:

2.4.1.1 Contractor QA Policy and Objectives

2.4.1.2 Organization and Personnel

- QA Management;
  - Organization;
  - Assignment of QC and QA responsibilities; and

- Reporting relationships.

- Personnel;
  - Staff resumes;
  - Education and experience requirements pertinent to this Contract; and
  - Training progress.
- Facilities and Equipment;
  - Instrumentation and backup alternatives; and
  - Maintenance activities and schedules.
- Document Control;
  - Laboratory notebook policy;
  - Sample and data tracking/custody procedures and documentation requirements;
  - Logbook maintenance and archiving procedures;
  - Case file organization, preparation, and review procedures; and
  - Procedures for preparation, approval, review, revision, and distribution of SOPs.
- Analytical Methodology;
  - Calibration procedures and frequency;
  - Sample handling and storage procedures;
  - Sample preparation procedures;
  - Sample analysis procedures; and
  - Standards preparation procedures.

Data Generation;

- Data collection procedures;
- Data reduction procedures;

- Data review procedures;
- Data reporting and authorization procedures; and
- Data management procedures.

• Quality Control Program; and

- Solvent, reagent, and adsorbent check analysis;
- Reference material analysis;
- Internal QC checks; and
- Corrective action and determination of QC limit procedures.

• Quality Assurance Program Assessment.

- Data audits;
- Systems audits;
- Performance audits;
- Corrective action procedures; and
- QA reporting procedures.

SECTION 3

STANDARD OPERATING PROCEDURES

3.1 In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document that 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.

3.2 SOPs prepared by the Contractor must be functional, i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs, as presented to the Agency, must reflect activities as they are currently performed in the laboratory. In addition, all SOPs must:

3.2.1 Be consistent with current EPA regulations, guidelines, and the CLP contract's requirements;

3.2.2 Be consistent with instrument manufacturer's specific instruction manuals;

3.2.3 Be available to the EPA 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 evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs;

3.2.4 Provide for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol;

3.2.5 Describe the mechanism for demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results;

3.2.6 Describe the corrective measures and feedback mechanism used when analytical results do not meet protocol requirements;

3.2.7 Be reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made;

3.2.8 Be archived for future reference in usability or evidentiary situations;

3.2.9 Be available at specific work stations as appropriate; and

3.2.10 Be subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

3.3 SOP SPECIFICATIONS AND FORMAT

3.3.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA sa the basis for laboratory evidence audits.

3.3.2 The format for SOPs may vary depending upon the kind of activity for which they are prepared. However, at a minimum, the following sections must be included.

3.3.2.1 Title page.

3.3.2.2 Scope and application.

3.3.2.3 Definitions.

3.3.2.4 Procedures.

3.3.2.5 QC acceptance criteria.

3.3.2.6 Corrective Action Procedures, including procedures for secondary review of information being generated.

3.3.2.7 Documentation Description and example forms.

3.3.2.8 Miscellaneous notes and precautions.

3.3.2.9 References.

3.4 REQUIRED SOPS

3.4.1 Evidentiary SOPs

The Contractor shall develop and use adequate written SOPs to ensure sample and data accountability. Evidentiary SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.4.1.1 **Sample receipt and logging**

3.4.1.1.1 The Contractor shall have written SOPs for receiving and logging in the samples. The procedures shall include, documentation of the following information:

- Presence or absence of EPA chain-of-custody forms;
- Presence or absence of airbills or airbill stickers;
- Presence or absence of EPA Traffic Reports or SAS packing lists;
- Presence or absence of custody seals on shipping and/or sample containers and their condition;
- Custody seal numbers, when present;
- Presence or absence of sample tags;
- Sample tag ID numbers;
- Condition of the shipping container;
- Condition of the sample container;
- Verification of agreement or nonagreement of information on receiving documents and sample containers;
- Resolution of problems or discrepancies with SMO; and
- The definition of any terms used to describe sample condition upon receipt.

3.4.1.1.2 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.4.1.2 **Sample identification**

3.4.1.2.1 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.4.1.2.2 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.4.1.2.3 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

3.4.1.3 Sample security

The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access to secure storage areas.

3.4.1.4 Internal chain-of-custody of samples and data.

The Contractor shall have written SOPs for the chain-of-custody consisting of sample identification, chain-of-custody procedures, sample receiving procedures, and sample tracking procedures. For more information concerning the chain-of-custody procedures see Section 4 of this Exhibit.

3.4.1.5 Internal tracking of samples and data.

The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

- A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses;
- A description of the documentation used to record instrument calibration and other QA/QC activities; and
- Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.4.1.6 Laboratory document and information control

3.4.2 Analytical SOPs

The Contractor shall develop and use adequate written SOPs to ensure that all data generated for the CLP are of known, documented, and acceptable quality. Analytical SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.4.2.1 The Contractor shall have written SOPs for preventing sample contamination, during sample preparation, cleaning of glassware, storage, and analysis.

3.4.2.2 The Contractor shall have SOPs to ensure traceability of standards used in sample analysis QA/QC.

3.4.3 **Quality Management SOPs**

3.4.3.1 The Contractor shall have written SOPs for technical and managerial review of laboratory operation and data package preparation, laboratory data review/laboratory self inspection system. The procedures shall include but not be limited to documenting the following information:

3.4.3.1.1 Data flow and chain-of-command for data review;

3.4.3.1.2 Procedures for measuring precision and accuracy.

3.4.3.1.3 Evaluation of parameters for identifying systematic errors.

3.4.3.1.4 Procedures to assure that hardcopy deliverables are complete and compliant with the requirements in Exhibit B.

3.4.3.1.5 Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.).

3.4.3.1.6 Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).

3.4.3.1.7 Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).

3.4.3.1.8 Documentation of audit reports, (internal and external), response, corrective action, etc.

3.4.3.2 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure. For more information

concerning document control and case file preparation, see Section 5 of this Exhibit.

3.4.3.3 The Contractor shall have written SOPs for sample analysis, management and handling, and reporting of data. The procedures shall include but not be limited to documenting the following information:

3.4.3.3.1 Procedures for controlling and estimating data entry errors.

3.4.3.3.2 Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

3.4.3.3.3 Life cycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

3.4.3.3.4 Database security, backup and archival procedures including recovery from system failures.

3.4.3.3.5 System maintenance procedures and response time.

3.4.3.3.6 Individual(s) responsible for system operation, maintenance, data integrity and security.

3.4.3.3.7 Specifications for staff training procedures.

3.4.3.4 The Contractor shall have written SOPs for laboratory safety.

3.5 HANDLING OF CONFIDENTIAL INFORMATION

3.5.1 A Contractor conducting work under this contract may receive EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.5.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.5.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each

working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

3.6 SOPS DELIVERY REQUIREMENTS

Within forty-five (45) days of contract receipt, a complete set of SOPs relevant to this contract shall be sent to the TPO, SMO and EMSL/LV. Also, during the term of performance of the contract, copies of SOPs which have been amended or new SOPs which have been written shall be sent to the TPO, EMSL/LV (quality assurance SOPs) and NEIC (evidentiary SOPs).

SECTION 4

CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

4.1 SAMPLE IDENTIFICATION

4.1.1 To ensure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

4.1.2 Each sample and sample preparation container shall be labeled with the EPA number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA number.

4.2 CHAIN-OF-CUSTODY PROCEDURES

Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented. A sample is under custody if the following applies:

4.2.1 It is in your possession, or

4.2.2 It is in your view after being in your possession, or

4.2.3 It was in your possession and you locked it up, or

4.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

4.3 SAMPLE RECEIVING PROCEDURES

4.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

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

4.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment.

4.3.5.1 Airbills or airbill stickers.

4.3.5.2 Custody seals.

4.3.5.3 EPA custody records.

4.3.5.4 EPA traffic reports or SAS packing lists.

4.3.5.5 Sample tags.

4.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

4.3.7 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

4.3.8 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

4.3.9 The following information shall be recorded on appropriate Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected.

4.3.9.1 Condition of the shipping container.

4.3.9.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

4.3.9.3 Custody seal numbers, when present.

4.3.9.4 Condition of the sample bottles.

4.3.9.5 Presence or absence of airbills or airbill stickers.

4.3.9.6 Airbill or airbill sticker numbers.

4.3.9.7 Presence or absence of EPA custody records.

4.3.9.8 Presence or absence of EPA traffic reports or SAS packing lists.

4.3.9.9 Presence or absence of sample tags.

4.3.9.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

4.3.9.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

4.3.9.12 Problems or discrepancies.

4.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 5

DOCUMENT CONTROL

The goal of the laboratory document control program is to assure that all documents for a specified Sample Delivery Group (SDG) will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

5.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

5.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

5.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

5.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

5.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

5.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the

laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

5.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be crossed out.

5.2 CONSISTENCY OF DOCUMENTATION

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

5.2.2 All copies of laboratory documents shall be complete and legible.

5.2.3 Original documents which include information relating to more than one SDG shall be filled in the CSF of the lowest SDG number. The copy(s) shall be placed in the other CSF(s) and the Contractor shall record the following information on the copy(ies) in red ink.

"COPY

ORIGINAL IS FILED IN CSF _____"

The Contractor shall sign and date this addition to the copy(ies).

5.2.4 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, lab bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

5.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURE

5.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B.

CSF # - Region - Serialized number (For example: 75-2-0240).

5.3.2 All documents relevant to each sample delivery group, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

5.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 is an example of a document inventory.

Figure E-1

Example

DOCUMENT INVENTORY

Document Control #*	Document Type	# Pages
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	50
232-2-0005	SMO Organics Traffic Reports	10
232-2-0006	Analysis Data Sheets	41
232-2-0007	Analysts' Organics Notebook Pages	14
232-2-0008	GC/MS and GC Instrument Logbook Pages	12
etc.	etc.	etc.

*This number is to be recorded on each set of documents.

5.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

5.5 SHIPPING DATA PACKAGES AND CSF

5.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

5.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

5.5.3 A copy of the transmittal letter for the CSF will be sent to the NEIC and the SMO.

5.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one (1) original FORM AADC-1 for each shipping container.

5.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

5.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

5.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 6

ANALYTICAL STANDARDS REQUIREMENTS

The U.S. Environmental Protection Agency will not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories will be required to prepare from neat materials, from cylinders of compressed gases traceable to NIST Standard Reference Materials or NIST/EPA approved certified reference material, or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

6.1 PREPARATION OF CHEMICAL STANDARDS FROM THE NEAT HIGH PURITY BULK MATERIAL

6.1.1 A laboratory may prepare their chemical standards from neat materials. Commercial sources for neat chemical standards pertaining to analytes listed on the TCL are given in Appendix C of the "Quality Assurance Materials Bank: Analytical Reference Standards," Seventh Edition, January 1988. Laboratories should obtain the highest purity possible when purchasing neat chemical standards; standards purchased at less than 98% purity must be documented as to why a higher purity could not be obtained.

6.1.2 Neat chemical standards must be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of neat chemicals is essential in order to safeguard them from decomposition.

6.1.3 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 contract laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use either differential scanning calorimetry, gas chromatography with flame ionization detection, high performance liquid chromatography, infrared spectrometry, or other 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:

$$\frac{\text{Wt. of impure compound}}{\left(\frac{\text{percent purity}}{100}\right)} = \text{Wt. of pure compound} \qquad \text{Eq. E-1}$$

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

6.1.4 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 contract laboratory's responsibility to have analytical documentation confirming that all compounds used in the preparation of solution standards are correctly identified. Identification confirmation, when performed, should use GC/MS analysis on at least two different analytical columns, or other appropriate techniques.

6.1.5 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 must verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights. All weighing should 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 should be compatible with the protocol in which the standard is to be used; the solute should be soluble, stable, and nonreactive with the solvent. In the case of a multicomponent solution, the components must not react with each other.

6.1.6 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 when not in use. All solution standards are to be clearly labeled as to the identity of the compound or compounds, concentration, date prepared, solvent, and initials of the preparer.

6.2 PURCHASE OF CHEMICAL STANDARDS IN SOLUTION

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

6.2.1.1 Contract laboratories must maintain the following documentation to verify the integrity of the standard solutions they purchase:

- Mass spectral identification confirmation of the neat material;
- Purity confirmation of the neat material; and
- Chromatographic and quantitative documentation that the solution standard was QC checked according to the following section.

6.2.1.2 The Contractor must 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 6.2.1.3 and 6.2.1.4 below). 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 part 6.3.1.3 which follows. If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test. The standard is certified to be within 10 percent of the target concentration.

6.2.1.3 If the procedure above is used, the supplier must document that the following have been achieved.

- Two solutions of identical concentration must be prepared independently from neat materials. An aliquot of the first solution must be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration ten percent 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 percent less that the target standard. This is called the "low standard";

- Six replicate analyses of each standard (a total of 18 analyses) must be performed in the following sequence: low standard, target, high standard, low standard, target standard, high standard; and

- The mean and variance of the six results for each solution must be calculated.

$$Mean = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} \qquad \text{Eq. E-2}$$

$$Variance = \frac{Y_{12} + Y_{22} + Y_{32} + Y_{42} + Y_{52} + Y_{62} - (6 \times Mean)_2}{5} \qquad \text{Eq. E-3}$$

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.

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + V_3}{\frac{1.21}{3}} \qquad \text{Eq. E-4}$$

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

- The test statistic must be calculated:

$$TEST\ STATISTIC = \frac{\left| \frac{M_2}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}} \qquad \text{Eq. E-5}$$

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

- The test statistic must be calculated:

$$TEST\ STATISTIC = \frac{\left| M_2 - \left(\frac{M_1}{1.8} \right) - \left(\frac{M_3}{2.2} \right) \right|}{\left(\frac{V_p}{4} \right)^{0.5}} \qquad \text{Eq. E-6}$$

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.

- The 95 percent confidence intervals for the mean result of each standard must be calculated:

$$INTERNAL\ FOR\ LOW\ STANDARD = M_1 \pm (2.13) \left(\frac{V_p}{6} \right)^{0.5} \qquad \text{Eq. E-7}$$

$$INTERNAL\ FOR\ TARGET\ STANDARD = M_2 \pm (2.13) \left(\frac{V_p}{6} \right)^{0.5} \qquad \text{Eq. E-8}$$

$$INTERNAL\ FOR\ HIGH\ STANDARD = M_3 \pm (2.13) \left(\frac{V_p}{6} \right)^{0.5} \qquad \text{Eq. E-9}$$

These intervals must not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate

the 10 percent difference in concentrations. In such a case, the standards are not acceptable. In any event, the laboratory is responsible for the quality of the standards employed for analyses under this contract.

6.3 REQUESTING STANDARDS FROM THE EPA STANDARDS REPOSITORY

Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, depending on availability. The Contractor can 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.

6.4 DOCUMENTATION OF THE VERIFICATION AND PREPARATION OF CHEMICAL STANDARDS

It is the responsibility of each laboratory to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra, etc, whether produced by the laboratory or purchased from chemical supply houses, must be maintained by the laboratory and may be subject to review during on-site inspection visits. Documentation of standards preparation may be required to be sent to EPA for verification of contract compliance. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the laboratories for a period of one year.

SECTION 7

METHOD SPECIFIC QA/QC REQUIREMENTS

7.1 The purpose of this section is to outline the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of the semivolatile organic compounds listed in Exhibit C, using the procedures in Exhibit D. This section is not intended as a comprehensive quality control document, but rather as a guide to the specific QC operations that must be addressed during analysis of semivolatiles captured on PUF/XAD-2. The laboratory is expected to address these operations in preparing the quality assurance plan and Standard Operating Procedures discussed in Sections 2 and 3.

7.2 The specific QC operations that must be considered for semivolatile organics analysis include the following:

7.2.1 Testing and Spiking of PUF/XAD-2 cartridges;

7.2.2 GC/MS Instrument Performance Check and Ion Abundance Patterns;

7.2.3 GC/MS Initial and Continuing Calibration;

7.2.4 Internal Standard Area and Retention Time;

7.2.5 Blank Analysis;

7.2.6 Surrogate Compound Recoveries; and

7.2.7 Performance Evaluation (PE) Samples.

7.2.8 Testing and Spiking of PUF/XAD-2 Cartridges

7.2.8.1 As part of the procedure for preparing PUF/XAD-2 cartridges for sampling and subsequent analysis, the laboratory is required to analyze by GC/MS or GC/ECD one cartridge or 10 percent, whichever is greater, of the cartridges from each batch of prepared PUF/XAD-2 cartridges, and confirm that the total level of semivolatiles per cartridge assembly (filter and PUF/XAD-2 sandwich) is less than 10 μg. If the total semivolatiles exceed 10 μg, the entire batch shall be rejected or cleaned until acceptable.

7.2.8.2 The laboratory is also required to spike each cartridge with surrogate compounds as discussed in Exhibit D.

7.2.9 GC/MS Performance Checking the Mass Spectrometer and Ion Abundance Patterns

7.2.9.1 Prior to initiating any data collection activities involving QC or field samples, it is necessary to establish that a given GC/MS system

meets the instrument performance criteria specified in Exhibit D. The purpose of this instrument performance check is to ensure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP).

7.2.9.2 The required frequency of DFTPP analysis (once every 12 hours on each GC/MS system) is described in detail in Exhibit D.

7.2.9.3 The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Exhibit D, Table D\SV-5.

7.2.9.4 The documentation includes Form IV-AASV.

7.2.10 **Initial Calibration of the GC/MS System**

7.2.10.1 Prior to the analysis of samples and required blanks and after instrument performance criteria have been met, the GC/MS system must be initially calibrated using calibration standards containing the target compounds, surrogates, and internal standards.

7.2.10.2 The detailed procedure is given in Exhibit D.

7.2.10.3 The GC/MS is calibrated by analyzing standards spiked onto the PUF/XAD-2 cartridge assemblies, and calculating concentrations by the relative response factor (RRF) method.

7.2.10.4 Calibration standards containing the target compounds of interest are prepared as outlined in Exhibit D and spiked onto the PUF/XAD-2 cartridges.

7.2.10.5 The PUF/XAD-2 cartridges are spiked with known concentrations of internal standards and surrogate standards as described in Exhibit D.

7.2.10.6 The cartridges are then analyzed as described in Exhibit D.

7.2.10.7 Relative response factors (RRFs) are calculated as described in Exhibit D.

7.2.10.8 The documentation includes Form V-AASV, Form VII-AASV, a GC/MS data system printout for the analysis of each semivolatile calibration standard, and the mass spectrum of each target and surrogate compound.

7.2.11 **GC/MS Continuing Calibration**

7.2.11.1 Once the GC/MS system has been calibrated, the calibration must be verified each 12-hour time period for each GC/MS system.

7.2.11.2 The standard is to be analyzed according to the procedures and at the frequency given in Exhibit D.

7.2.11.3 The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum response factors of each compound with minimum RRFs in the continuing calibration and the percent difference must meet the criteria given in Exhibit D.

7.2.11.4 The documentation includes, Form VI-AASV, Form VII-AASV, a GC/MS data system printout for the analysis of the semivolatile calibration standard, and the mass spectrum of each target compound.

7.2.12 Internal Standard Area and Retention Times

7.2.12.1 The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results, because the quantitative determination of semivolatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

7.2.12.2 The specific compounds used as internal standards are given in Exhibit D. Each internal standard is spiked on the PUF/XAD-2 cartridge at the level specified in Exhibit D.

7.2.12.3 The retention time and the area response of the primary quantitation ion of each internal standard must be monitored for all analyses.

7.2.12.4 The area response and retention times of each internal standard are evaluated for stability, according to the procedures in Exhibit D. The area of the internal standard in a sample must be within ±40 percent of the area of the same internal standard in the associated initial or continuing calibration standard. Likewise, the retention time of an internal standard in a sample must be within ±0.06 RRT units of its retention time in the associated initial or continuing calibration standard, as described in Exhibit D.

7.2.12.5 Requirements for analysis of samples when internal standards do not meet specifications are given in Exhibit D.

7.2.12.6 The documentation includes Form VII-AASV; and a GC/MS data system printout for the analysis of each semivolatile sample, blank, and calibration standard.

7.2.13 Blank Analysis

7.2.13.1 A blank is a certified clean PUF/XAD-2 cartridge carried through the entire analytical procedure. The field blank is spiked with the surrogates and sent out to the field along with cartridges used for air

sampling. The laboratory method blank cartridge never leaves the laboratory. The purpose of a laboratory method blank is to determine the levels of contamination associated with the processing and analysis of samples at the laboratory, while the field blank monitors contamination due to cartridge handling during sampling.

7.2.13.2 A laboratory method blank shall be analyzed once every 12 hours on each GC/MS system while a field blank must be analyzed at least once per SDG, as described in detail in Exhibit D.

7.2.13.3 For the purposes of this protocol, an acceptable blank must contain less than or equal to the Contract Required Quantitation Limit (see Exhibit C) of any single target compound, or less than 10 μg/cartridge for total semivolatiles, whichever is less.

7.2.13.4 If a laboratory blank exceeds the limits for contamination above, the Contractor shall consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reanalysis of associated samples are given in Exhibit D.

7.2.13.5 The documentation includes Form II-AASV Blank Summary Form, Form I-AASV for the blank analysis, Form VII-AASV for internal standards in the blank, Form IX for surrogate recoveries in the blank, and a GC/MS data system printout for the analysis of the blank.

7.2.14 **Surrogate Compound Recoveries**

7.2.14.1 The recoveries of the surrogate compounds are calculated from the analysis of each sample and blank. The purpose of the surrogate compounds is to evaluate the performance of the entire PUF/XAD-2 cartridge and GC/MS system. Poor extraction efficiency, leaks, and cold spots in transfer lines are only a few of the potential causes of poor recovery of these compounds.

7.2.14.2 Three surrogate compounds are added to each PUF/XAD-2 cartridge assembly prior to field sampling at the concentrations described in Exhibit D. Five additional surrogate compounds are added to the cartridges just prior to extraction.

7.2.14.3 The recoveries of the surrogate compounds are calculated according to the procedures in Exhibit D. The recoveries must be within the quality control limits given in Exhibit D. If the recovery of any one surrogate compound is outside these limits, the Contractor shall follow the steps outlined in Exhibit D.

7.2.14.4 The documentation includes Form IX-AASV and a GC/MS data system printout for the analysis of each sample and blank.

7.2.15 **Performance Evaluation (PE) Samples**

7.2.15.1 Performance evaluation samples are intended to assist the Agency in monitoring Contractor performance. The laboratory will not be informed as to which compounds are contained in the PE samples or the concentrations.

7.2.15.2 The Laboratory shall extract, analyze, and report the results of the PE sample once per sample delivery group, if available.

7.2.15.3 The laboratory will receive PE samples on PUF/XAD-2 cartridges from the Agency. The samples will come with instructions concerning the extraction procedure required for the PE samples. The Laboratory shall add internal and surrogate compounds to the PE sample, following procedures in Exhibit D.

7.2.15.4 Each laboratory shall extract and concentrate the PE sample using the procedure described in Exhibit D for those semivolatile target compounds listed in Exhibit C.

7.2.15.5 The laboratory shall meet the following PE sample technical acceptance criteria as detailed in Exhibit D:

7.3.8.5.1 The PE sample must be analyzed on a GC/MS system meeting the DFTPP tuning and initial calibration or continuing calibration technical acceptance criteria at the frequency described in Exhibit D.

7.3.8.5.2 The PE sample must be desorbed according to Exhibit D.

7.3.8.5.3 The PE sample must be prepared and analyzed with a laboratory method blank that meets the blank technical acceptance criteria.

7.3.8.5.4 The percent recovery for each of the surrogates in the PE sample must be within acceptable windows as outlined in Exhibit D.

7.3.8.5.5 The area response change between the PE sample and the most recent calibration standard analysis for each of the internal standards must be within 40 percent.

7.3.8.5.5 The retention time shift between the PE sample and the most recent calibration standard analysis for each of the internal standards must be within 20 seconds.

7.3.8.5.6 The percent recovery for each of the target compounds must be within replicate precision and audit accuracy, as outlined in Exhibit D.

SECTION 8

REGIONAL DATA REVIEW

8.1 Contract laboratory data are generated to meet the specific needs of the Regions. In order to verify the useability of data for the intended purpose, each Region reviews data from the perspective of end-user, based upon functional aspects of data quality. General guidelines for data review have been developed jointly by the Region and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problems under investigation and the Regional response appropriate to the specific circumstances.

8.2 Regional data reviews relating useability of the data to a specific site are part of the collective assessment process. They complement the review done at the Sample Management Office, which is designed to identify contractual discrepancies, and the review done at EMSL/LV, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for program and laboratory administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

SECTION 9

LABORATORY EVALUATION SAMPLES

9.1 Although intralaboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in interlaboratory comparison studies conducted by the EPA. Results from the analysis of these laboratory evaluation samples will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

9.2 Sample sets may be provided to participating Contractors as frequently as on an SDG-by-SDG basis as a recognizable QC sample of known composition; as a recognizable QC sample of unknown composition; or not recognizable as a QC material. The laboratory evaluation samples may be sent either by the Regional client or the National Program Office, and may be used for contract action.

9.3 Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

9.4 At a minimum, the results are evaluated for compound identification, quantification, and sample contamination. Confidence intervals for the quantification of target compounds are based on reported values using population statistics. EPA may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample. Normally, a fraction of the compounds spiked into the sample are not specifically listed in the contract. Contractors are required to use the NIST/EPA/MSDC mass spectral library to tentatively identify a maximum number of non-target compounds in each fraction that are present above a minimal response. Tentative identification of these compounds based on contractually described spectral interpretation procedures is evaluated and integrated into the evaluation process.

9.5 A Contractor's results on the laboratory evaluation samples will determine the Contractor's performance as follows:

9.5.1 No response is required for a score of 90 percent or above.

9.5.2 For a score of 75 to 89, the Contractor shall describe the deficiency(ies) and the corrective action(s) taken in a letter to the APO, TPO, and EMSL/LV, within 14 days of receipt of notification from EPA.

9.5.3 For a score less than 75, the Contractor shall be notified by the APO or TPO concerning the remedy for its unacceptable performance. The

Contractor may expect, but EPA is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, a site visit, a full data audit, analysis of remedial PE samples, and/or a contract sanction, such as a Cure Notice.

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

SECTION 10

GC/MS TAPE AUDITS

10.1 Periodically, EPA requests from Contractors the GC/MS magnetic tapes corresponding to a specific Case in order to accomplish tape audits. Generally, tape submissions and audits are requested for the following reasons.

10.1.1 Program overview;

10.1.2 Indication of data quality problems from EMSL/LV, SMO, or Regional data reviews;

10.1.3 Support for on-site audits; and

10.1.4 Specific Regional requests.

10.2 Depending upon the reason for an audit, the 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 with that generated on the GC/MS tapes. This function provides external monitoring of Program QC requirements and checks adherence of the Contractor to internal QA procedures. In addition, tape audits enable EPA to evaluate the utility, precision, and accuracy of the analytical methods.

10.3 The GC/MS tape shall include raw data and quantitation reports for samples, blanks, laboratory evaluation samples, initial calibrations, continuing calibration, and DFTPP tuning associated with the Case requested. The specific requirements for submissions of GC/MS tapes are discussed in Exhibit B.

10.4 Upon request of the Administrative Project Officer or EMSL/LV, the required tapes and all necessary documentation shall be submitted to EPA within seven (7) days of notification.

SECTION 11

ON-SITE LABORATORY EVALUATIONS

11.1 At a frequency dictated by a contract laboratory's performance, the Administrative Project Officer(APO)/Technical Project Officer(TPO) 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 Evaluation and an Evidentiary Audit.

11.2 QUALITY ASSURANCE ON-SITE EVALUATION

11.2.1 Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures. The Contractor should expect that items to be monitored will include but not be limited to the following items:

- Size and appearance of the facility;
- Quantity, age, availability, scheduled maintenance and performance of instrumentation;
- Availability, appropriateness, and utilization of 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 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, laboratory evaluation sample scores, Regional review of data, Regional QA materials, GC/MS tape audit reports, and data trend reports.

11.3 EVIDENTIARY AUDIT

11.3.1 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. The evidence audit is comprised of the following three activities.

11.3.1.1 Procedural Audit

The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following laboratory operations:

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

11.3.1.2 Written SOPs Audit

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

11.3.1.3 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:

- Accuracy of the document inventory;
- Completeness of the file;
- 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 Administrative Project Officer (APO)/Technical Project Officer (TPO) 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

11.5.1 Following an on-site evaluation, QA and evidentiary audit reports which discuss deficiencies found during the on-site evaluation will be forwarded to the Contractor. The Contractor must discuss the corrective actions taken to resolve the deficiencies discussed during the on-site visit and discussed in the on-site reports in a letter to the APO/TPO, EMSL/LV (response to the QA report) and NEIC (response to the evidentiary report) within 14 days of receipt of the finding or within the time agreed upon between APO/TPO and the Contractor. If SOPs are required to be written or SOPs are required to be amended, the Contractor must provide the SOPs to the TPO, EMSL/LV (QA/technical SOPs) and NEIC (evidentiary SOPs) within 30 days of receipt of the finding or within the time agreed upon between the APO/TPO and the Contractor.

11.5.2 If the Contractor fails to take appropriate corrective action to resolve the deficiencies discussed in the on-site reports, a Contractor may expect, but the Agency is not limited to, the following actions:

- reduction of the number of samples sent under the contract;
- suspension of sample shipment to the Contractor;
- a follow-up site visit, a full data audit; and
- analysis of remedial PE samples and/or contract sanction, such as a Cure Notice.

SECTION 12

QUALITY ASSURANCE AND DATA TREND ANALYSIS

12.1 Data submitted by laboratories are subject to review from several aspects: compliance with contract-required QC, usability, and full data package evaluation. Problems resulting from any of these reviews may determine the need for a GC/MS 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 Agency 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 data base. Statistical reports that evaluate specific anomalies or disclose trends in many areas, including the following, are generated from this data base:

12.1.1 Laboratory Control Sample;

12.1.2 Blanks;

12.1.3 GC/MS Instrument Performance Checks;

12.1.4 Initial and Continuing Calibration Data; and

12.1.5 Other QC and Method Parameters.

12.2 Program-wide statistical results are used to rank laboratories in order to observe the relative performance of each Contractor using a given protocol against its peers. The reports are also used to identify trends within laboratories. The results of many of these trends analyses are included in overall evaluation of a Contractor's performance, and are reviewed to determine if corrective action or an on-site laboratory evaluation is indicated in order to meet the QA/QC requirements of the contract.

12.3 Contractor performance over time is monitored using these trend analysis techniques to detect departures of Contractor output from required or desired levels of QC, and to provide an early warning of Contractor QA/QC problems which may not be apparent from the results of an individual case.

12.4 As a further benefit to the Program, the data base 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 contract laboratories is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of QC and performance criteria specifications of what is routinely achievable and expected of environmental chemistry laboratories in mass production analysis

of environmental samples. This, in turn, assists the Agency in meeting its objectives of obtaining data of known and documented quality.

SECTION 13

DATA MANAGEMENT

13.1 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 should 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.

13.2 Data manually entered from hard-copy must be quality controlled 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 must be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

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

13.3.1 Justification or rationale for the change;

13.3.2 Initials of the person making the change or changes. Data changes must be implemented and reviewed by a person or group independent of the source generating the deliverable;

13.3.3 Change documentation must be retained according to the schedule of the original deliverable;

13.3.4 Resubmitted diskettes or other deliverables must be reinspected as a part of the laboratory's internal inspection process prior to resubmission. The entire deliverable, not just the changes, must be inspected;

13.3.5 The Laboratory Manager must approve changes to originally submitted deliverables; and

13.3.6 Documentation of data changes may be requested by laboratory auditors.

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

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

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

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

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

13.5 Individual(s) responsible for the following functions must be identified:

13.5.1 System operation and maintenance including documentation and training;

13.5.2 Database integrity, including data entry, data updating and quality control; and

13.5.3 Data and system security, backup and archiving.

SECTION 14

REFERENCES

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14.5 Health Effects Research Laboratory, U.S. Environmental Protection Agency, Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, EPA-600/8-80-036, June, 1980.

14.6 Environmental Protection Agency, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule", 40 CFR Part 136, Federal Register, Vol. 49, No. 209, pp 43234-43442, October 26, 1984.

14.7 Health Effects Research Laboratory, U.S. Environmental Protection Agency, Manual of Analytical Quality Control for Pesticides and Related Compounds In Human and Environmental Samples-Second Revision, EPA-600/2-81-059, April 1981.

14.8 Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Analytical Reference Standards and Supplemental Data: The Pesticides and Industrial Chemicals Repository, EPA-600/4-84-082, October 1984.

14.9 American Chemical Society Committee on Environmental Improvement, and Subcommittee on Environmental Analytical Chemistry, "Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry", Analytical Chemistry, Volume 52, Number 14, December 1980.

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EXHIBIT F

EVIDENTIARY REQUIREMENTS

January, 1992

EXHIBIT F

EVIDENTIARY REQUIREMENTS

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SECTION 1

SAMPLE CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

1.1 SAMPLE IDENTIFICATION

1.1.1 To assure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

1.1.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA sample number.

1.2 CHAIN-OF-CUSTODY PROCEDURES

1.2.1 Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented.

1.2.2 A sample is under custody if the following applies:

1.2.2.1 It is in your possession.

1.2.2.2 It is in your view after being in your possession.

1.2.2.3 It was in your possession and you locked it up.

1.2.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

1.3 SAMPLE RECEIVING PROCEDURES

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available. The condition of the shipping containers and sample bottles shall be

inspected upon receipt by the sample custodian or his/her representative.

1.3.3 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

1.3.4.1 Airbills or airbill stickers.

1.3.4.2 Custody seals.

1.3.4.3 EPA custody records.

1.3.4.4 EPA traffic reports or SAS packing lists.

1.3.4.5 Sample tags

1.3.5 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

1.3.6 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

1.3.7 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

1.3.8 The following information shall be recorded on Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected:

1.3.8.1 Condition of the shipping container.

1.3.8.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

1.3.8.3 Custody seal numbers, when present.

1.3.8.4 Condition of the sample bottles.

1.3.8.5 Presence or absence of airbills or airbill stickers.

1.3.8.6 Airbill or airbill sticker numbers.

1.3.8.7 Presence or absence of EPA custody records.

1.3.8.8 Presence or absence of EPA traffic reports or SAS packing lists.

1.3.8.9 Presence or absence of sample tags.

1.3.8.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

1.3.8.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

1.3.8.12 Problems or discrepancies.

1.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 2

DOCUMENT CONTROL PROCEDURES

The goal of the laboratory document control program is to assure that all documents for a specified SDG will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

2.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

2.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the laboratory may exercise the option of using only laboratory or EPA

sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

2.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be crossed out.

2.2 CONSISTENCY OF DOCUMENTATION

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

2.2.2 All copies of laboratory documents shall be complete and legible.

2.2.3 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

2.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURES

2.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

2.3.2 All documents relevant to each SDG, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

2.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 of Exhibit E is an example of a document inventory.

2.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

2.5 SHIPPING DATA PACKAGES AND CSF

2.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

2.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

2.5.3 A copy of the transmittal letter for the CSF will be sent to NEIC and SMO.

2.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one original FORM AADC-1 for each shipping container.

2.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

2.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

2.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 3

STANDARD OPERATING PROCEDURES

The Contractor must have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, tracking the analysis of samples, and assembly of completed data.

3.1 SPECIFICATIONS FOR WRITTEN STANDARD OPERATING PROCEDURES

3.1.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation.

3.1.2 The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits. The Contractor must have written standard operating procedures (SOPs) for:

3.1.2.1 Sample receipt and logging.

3.1.2.2 Sample storage.

3.1.2.3 Preventing sample contamination.

3.1.2.4 Security for laboratory and samples.

3.1.2.5 Traceability of standards.

3.1.2.6 Maintaining instrument records and logbooks.

3.1.2.7 Sample analysis and data control systems.

3.1.2.8 Glassware cleaning.

3.1.2.9 Technical and managerial review of laboratory operation and data package preparation.

3.1.2.10 Internal review of contractually-required quality assurance and quality control data for each individual data package.

3.1.2.11 Sample analysis, data handling, and reporting.

3.1.2.12 Chain-of-Custody.

3.1.2.13 Document control, including Case file preparation.

3.1.3 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.1.4 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.1.4.1 Presence or absence of EPA chain-of-custody forms.

3.1.4.2 Presence or absence of airbills or airbill stickers.

3.1.4.3 Presence or absence of EPA Traffic Reports or SAS packing lists.

3.1.4.4 Presence or absence of custody seals on shipping and/or sample containers and their condition.

3.1.4.5 Custody seal numbers, when present.

3.1.4.6 Presence or absence of sample tags.

3.1.4.7 Sample tag ID numbers.

3.1.4.8 Condition of the shipping container.

3.1.4.9 Condition of the sample container.

3.1.4.10 Verification of agreement or nonagreement of information on receiving documents and sample containers.

3.1.4.11 Resolution of problems or discrepancies with SMO.

3.1.4.12 The definition of any terms used to describe sample condition upon receipt.

3.1.5 The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.1.6 The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

3.1.6.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.1.6.2 A description of the documentation used to record instrument calibration and other QA/QC activities.

3.1.6.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.1.7 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.1.8 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.1.9 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions. The Contractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.

3.1.10 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure.

3.1.11 The Contractor shall have written SOPs for laboratory safety.

3.1.12 The Contractor shall have written SOPs for cleaning of glassware used in preparing and analyzing samples under this contract.

3.1.13 The Contractor shall have SOPs for traceability of standards used in sample analysis QA/QC.

3.2 HANDLING OF CONFIDENTIAL INFORMATION

3.2.1 A Contractor conducting work under this contract may receive

EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.2.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.2.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

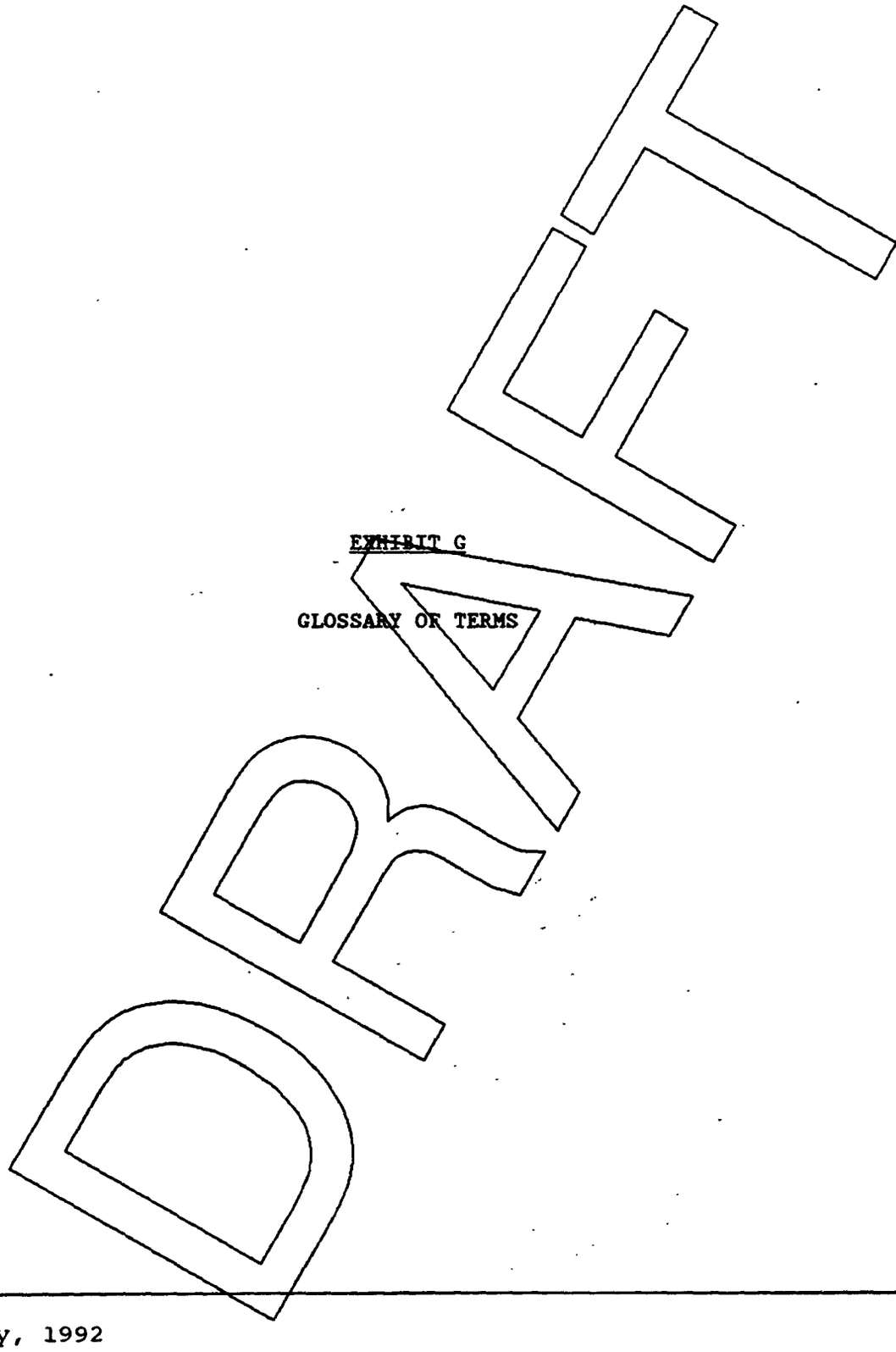

EXHIBIT G

GLOSSARY OF TERMS

January, 1992

EXHIBIT G

GLOSSARY OF TERMS

Aliquot - A measured portion of a field sample taken for analysis.

Analysis Date/Time - The date and military time (24 hour clock) of the introduction of the sample, standard, or blank into the GC/MS system.

Analysis Group - An analysis group is a set of no more than twenty analytical samples (as defined below) for the purpose of method Quality Assurance/Quality Control (QA/QC), such that the QA/QC required by Exhibit E is, at a minimum, prepared and analyzed at a frequency of once per twenty analytical samples.

Analysis Run - The actual instrumental analysis of the sample preparations from the time of instrument calibration through the running of the final sample. All sample preparation analyses during the analysis run are subject to the QC protocols set forth in Exhibit E of this contract unless otherwise specified in the individual methods.

Analytical Preparation Method - A method (i.e, extraction) used to dissolve or otherwise release the compound(s) of interest from its matrix and provide a final solution containing the compound(s) which is suitable for instrumental or other analysis methods.

Analytical Sample - Any solution or media introduced into an instrument on which an analysis is performed excluding initial calibration and continuing calibration standards. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), duplicate samples, laboratory control sample (LCS), and blanks.

ASTM Type II Water - Distilled water with a conductivity of less than 1.0 μmho/cm at 25°C. For additional specifications refer to ASTM D1193-77, "Standard Specification for Reagent Water".

Background Correction - A technique to compensate for variable background contribution to the instrument signal in the determination of target compounds.

Batch - A group of samples prepared at the same time.

Breakthrough volume (V_B) - Sample volume at which point a particular component will be initially detected in the eluate from the PUF/XAD-2 cartridge.

Calibration - The establishment of an analytical curve based on the response

or other measured characteristic of known standards.

Calibration Standards - A series of known standard solutions used to determine instrument stability and reliability (preparation of the analytical curve).

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.

Coefficient of Variation (CV) - The standard deviation as a percent of the arithmetic mean.

Continuing Calibration - Analytical standard run at least every 12 hours to verify the initial calibration of the analytical system.

Contract Required Quantitation Limit (CRQL) - Minimum level of quantitation acceptable under the contract. Generally defined as 3.3 (or more) times the standard deviation of seven replicate analyses of the method blank.

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

Correlation Coefficient - A number (r) which indicates the degree of dependence between two variables (e.g., concentration - absorbance). The more dependent they are the closer the value to one. Determined on the basis of the least squares line.

Cryogen - A liquified gas used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp - 195.8°C).

Data System - For the purpose of this contract, computer system that allows the continuous acquisition and printout of time vs. intensity data throughout the chromatographic program.

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

DDI - Deionized Distilled water.

Deuterated Chemicals - Those chemicals which contain deuterium (hydrogen isotope that is twice the mass of hydrogen); used as tracers for system quality assurance.

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

EBCDIC - Extended Binary Coded Decimal Interchange Code.

External Standards - Target analytes analyzed at a known concentration prior to sample analysis, to determine calibration factors.

Extractable - A compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile organic compounds.

Field Blank - Any blank PUF/XAD-2 cartridges spiked with the same surrogates and sent to the field, along with cartridges for field sampling, but through which ambient air is not sampled.

Field Sample - Material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

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

In-House - At the Contractor's facility.

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

Instrument Detection Limit (IDL) - Determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

Interferents - Substances which affect the analysis for the compound of interest.

Internal Standards - Analytes added to every standard, blank, and sample extract at a known concentration prior to analysis. Internal standards are used as the basis for quantitation of the target and surrogate compounds.

Laboratory - Synonymous with Contractor as used herein.

Laboratory Control Sample (LCS) - Aliquot spiked with known concentration of specific analytes and subjected to the entire analytical procedure in order to monitor method and contractor performance.

Laboratory Method Blank (LMB) - A solution produced by performing the analytical preparation method without the addition of a sample. The solution thus contains the same concentrations of reagents as all other analytical preparations plus any impurities derived from the preparation process. For preparations containing reagents of variable concentrations, the LMB should

match the maximum reagent concentration used in the sample extraction.

Laboratory Receipt Date - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

Linear Range - The concentration range over which the analytical curve remains linear.

Mass Spectral Interference - Defined as the inability to detect the internal standard quantification ion due to presence of high levels of mass spectral "noise" at the same mass.

Matrix - The predominant material of which the sample to be analyzed is composed.

Megabore® Column - One of two types of capillary columns, the other being the narrow bore, for the analysis of target compounds under this contract.

Method Detection Limit (MDL) - The chemical concentration that produces a signal, due to an analyte, which is equal to the student $t_{.99}$ times the standard deviation of a series of measurements on at least seven separate method blanks. In practice, a method detection limit will be substantially higher than an instrumental detection limit.

MS-SCAN - The gas chromatograph (GC) is coupled to a mass selective detector where the instrument is programmed to acquire all mass for target analytes and to disregard all others.

Narrative (SDG Narrative) - Portion of the data package which includes laboratory, contract, SDG 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.

Narrow-Bore Capillary Column - One of two capillary columns, the other being the wide-bore (Megabore®) capillary column, for the analysis of compounds under this contract.

Performance Evaluation (PE) Sample - A sample of known composition provided by EPA for Contractor analysis. Used by EPA to evaluate Contractor performance.

Protocol - A compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

Qualitative Accuracy - The ability of an analytical system to correctly identify compounds.

Quantitative Accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

Reconstructed Ion Chromatogram (RIC) - A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Recovery - A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

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

Relative Response Factor (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where:
- A = area of the characteristic ion measured;
- C = concentration;
- is = internal standard; and
- x = compound of interest.

Resolution - Also termed separation, the separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Retention Time (RT) - The time to elute a specific chemical from a chromatographic column for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until its maximum concentration appears at the detector.

Retention Time Window - Retention time window is determined for each analyte of interest and is the time from injection to elution of a specific chemical from a chromatographic column. The window is determined by three injections of a single component standard over a 24-hour period as plus or minus three times the standard deviation of the absolute retention time for that analyte.

Rounding Rules - The following are the rules for rounding off numbers.

If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

See forms instructions guide (Exhibit B) for exceptions.

Run - A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by this contract.

Sample Delivery Group (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 samples within a Case; or
- Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

Samples may be assigned to Sample Delivery Groups by sample collection method.

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

Selected Ion Current Profile (SICP) - A plot of ion abundance vs. time or scan

number for ions of a specified mass.

Semivolatile Organic Compounds - Target compounds with normal vapor pressures between 1 x 10^{-1} and 1 x 10^{-7} mm Hg, and which are amenable to analysis by this method.

Standard Analysis - An analytical determination made with known quantities of target compounds; used to determine response factors.

Static Calibration - Calibration of an analytical system with known concentrations of calibration gas, obtained from a source such as gas cylinders or prepared from standard stock solutions.

Stock Solution - A standard solution which can be diluted to derive other standards.

Surrogates Compounds - Compounds added to every blank, sample, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media.

Target compound - The semivolatile organic compound an analysis seeks to determine; the compound of interest.

Tentatively Identified Compounds (TIC) - Compounds detected in samples that are not target compounds, internal standards or surrogate standards. Up to 10 peaks are subjected to mass spectral library searches for tentative identification.

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 is used for documenting sample condition and receipt by the laboratory.

Twelve-Hour Time Period - The twelve (12) hour time period for GC/MS system tuning, standards calibration (initial or continuing calibration) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of compliant tune. The time period ends after 12 hours has 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.

ATTACHMENT VI

USEPA CONTRACT LABORATORY PROGRAM

METAL

ANALYSIS

OF

AMBIENT AIR

(MAA)

December, 1991

Revision MAA01.0

USEPA CONTRACT LABORATORY PROGRAM

METAL ANALYSIS OF AMBIENT AIR (MAA)

December, 1991

Revision MAA01.0

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PREFACE

The purpose of this contract is to provide the U.S. Environmental Protection Agency (EPA) with chemical analytical services, quality control procedures, and an analysis structure which will generate data of known and documented quality. The methods for the analysis of ambient air was developed with the guidance of the air toxics workgroup to ensure that the needs of regional, state and local air pollution programs were addressed.

The samples to be analyzed are of ambient air collected at or in the vicinity of known or suspected hazardous waste sites and may contain potentially hazardous organic and/or inorganic material in high concentrations. The Contractor should be aware of the potential hazards associated with the handling and analyses of these samples. It is the Contractor's responsibility to take all necessary measures and precautions to ensure the health and safety of its employees. The Contractor is responsible for providing a safe working environment and making its employees aware of the potential hazards of working with and analyzing these samples.

Procedures specified herein shall be used in the preparation and analysis of air samples for the presence and quantitation of inorganic parameters. The Contractor shall employ safe handling procedures and generally accepted laboratory practices in the performance of contract requirements and shall follow the quality assurance and quality control (QA/QC) program specified herein.

The data obtained under this contract will be used by EPA to determine the existence and extent of risk posed by hazardous waste disposal sites to the public, individuals involved in Superfund site cleanups, and the environment. The data may be used in civil and/or criminal litigation which requires the strictest adherence to chain-of-custody protocol, document control, and quality assurance procedures.

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR METAL ANALYSIS OF AMBIENT AIR

December, 1991

EXHIBIT A

SUMMARY OF REQUIREMENTS FOR METAL ANALYSIS OF AMBIENT AIR

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SECTION 1

GENERAL REQUIREMENTS

The Contractor shall employ procedures specified in this Contract in the preparation and analysis of the ambient air samples for the presence and quantitation of the metal parameters indicated in Exhibit C.

The Contractor shall employ proven techniques to identify and measure the metal parameters presented in the Target Compound List (TCL) as specified in Exhibit C. The Contractor shall perform sample preparation, analysis procedures, and quailty control procedures as prescribed in Exhibit D, and meet specified sample preservation and holding time requirements.

For all samples analyzed under this contract, the Contractor shall adhere to the quality assurance/quality control (QA/QC) protocols specified in Exhibit E and abide by the evidentiary protocols specified in Exhibit F.

Following sample analysis, the Contractor shall perform data reduction and shall report analytical activities, sample data, and quality control documentation as designated in Exhibit B. Exhibit B contains all reporting and deliverables requirements for this contract, including copies of the data reporting forms and forms instruction guide.

To ensure proper understanding of the language in this contract, Exhibit G contains a glossary of terms. When a term is used in the text without explanation, the glossary meaning shall be applicable. Glossary definitions do not replace or take precedence over specific information included in the Contract text.

The samples to be analyzed by the Contractor are from known or suspected hazardous waste sites and may contain hazardous organic and/or metal materials at high concentration levels. The Contractor should be aware of the potential hazards associated with the handling and analysis of these samples. It is the Contractor's responsibility to take all necessary measures to ensure the health and safety of its employees.

In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, as it is used to make major decisions regarding public health or welfare and the environment. In addition, data generated under this contract may be used in litigation against potentially responsible parties in the enforcement of Superfund legislation.

SECTION 2

SPECIFIC REQUIREMENTS

For each sample, the Contractor shall perform the following tasks:

2.1 TASK I: RECEIVE AND PREPARE AMBIENT AIR SAMPLES

2.1.1 The Contractor shall receive and handle samples under the chain-of-custody and document control procedures described in Exhibit F.

2.1.2 The Contractor shall provide the required analytical expertise and instrumentation for analyses of the TCL analytes equal to or lower than the quantitation limits specified in Exhibit C. In Exhibit D, EPA provides the Contractor with the specific sample preparation techniques for ambient air samples and an appropriate set of analytical procedures that shall be used.

2.1.3 The Contractor shall prepare and analyze samples within the maximum holding time specified in Exhibit D, even if these times are less than the maximum data submission time allowed in this contract.

2.1.4 The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic and metal materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the preparation and analysis with appropriate caution. The Contractor shall be responsible for all necessary measures and precautions to ensure the health and safety of laboratory employees.

2.2 TASK II: ANALYZE SAMPLES FOR THE IDENTIFICATION AND QUANTITATION OF SPECIFIC PARAMETERS

2.2.1 For each sample received, the Contractor shall be required to perform the analyses described as follows. The documentation that accompanies the sample(s) to the Contractor facility shall indicate specific analytical requirements for that sample or set of samples.

2.2.2 Exhibit D specifies the analytical procedures that shall be used. Exhibit D contains instructions and references for preparation of ambient air samples containing low-to-medium concentrations of metals for ICP, ICP/MS, or AA analysis.

2.2.3 All samples must initially be run undiluted (i.e., final product of sample preparation procedure). When an analyte concentration exceeds the calibrated or linear range, appropriate dilution [but not below the contract required quantitation limit (CRQL)] and reanalysis of the prepared sample is required, as specified in Exhibit D.

2.2.4 For the purpose of this contract, a full sample analysis is defined as analysis for all of the TCL constituents identified in Exhibit C in accordance with the methods and quality control (QC) procedures in Exhibit D and performance of related QA/QC as specified in Exhibit E. Laboratory Control Samples (LCS) and spike sample analyses shall each be considered a separate full sample analysis. All other QA/QC requirements are considered an inherent part of this Contract and are included in the contract sample unit price.

2.3 TASK III: PERFORM REQUIRED QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

2.3.1 All specific QA procedures prescribed in Exhibits E and QC procedures prescribed in Exhibit D shall be strictly adhered to by the Contractor. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibit B requirements.

2.3.2 The Contractor shall establish and use on a continuing basis QA/QC procedures, including the daily or (as required) more frequent use of standard reference solutions from EPA, NIST, or secondary standards traceable thereto, where available at appropriate concentrations (i.e., standard solutions designed to ensure that operating parameters of equipment and procedures, from sample receipt through identification and quantitation, produce reliable data). Exhibits D and E provide specific QA/QC requirements.

2.3.3 Additional QA/QC shall be required quarterly or more frequently; i.e., with each Case or Sample Delivery Group (SDG), in the form of Laboratory Control Samples (LCS) and Performance Evaluation Samples (PES) for metals submitted to EPA for Contractor analysis, and in the form of verification of instrument parameters, as described in Exhibit D and E.

2.3.3.1 EPA has provided to the Contractor formats for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in this Contract and within the time specified in the Contract Performance/ Delivery Schedule.

2.3.3.2 Use of formats other than those designated by EPA will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government will be required.

2.3.3.3 Computer generated forms may be submitted in the hardcopy data package(s) provided that the forms are in exact EPA format. This means that the order of data elements is the same as on each EPA required form, including form numbers and titles, page numbers and header information, columns, and lines.

2.3.4 The Contractor shall provide analytical equipment and technical expertise for this contract as specified by the following.

2.3.4.1 Inductively coupled plasma (ICP) emission spectrometer with the capability to analyze metals sequentially or simultaneously.

2.3.4.2 Inductively coupled plasma-mass spectrometer (ICP-MS) with the capability to analyze metals (optional).

2.3.4.3 Atomic absorption (AA) spectrometer equipped with graphite furnace and flame analysis capabilities.

2.3.4.4 The Contractor shall have, in-house, the appropriate standards for all target compounds listed in Exhibit C prior to accepting any samples from the Sample Management Office (SMO). Standards provided by EPA for use in the Preaward Performance Evaluation may not contain all the target compounds and thus shall not be used for routine analyses unless or until they have been supplemented with commercially-available standard materials.

2.3.5 The minimum functional requirements necessary to meet the terms and conditions of this contract are listed below. The Contractor shall designate and use qualified key personnel to perform these functions. The EPA reserves the right to review personnel qualifications and experience.

2.3.5.1 Project Manager.

2.3.5.2 Inorganic Laboratory Supervisor.

2.3.5.3 Quality Assurance Officer.

2.3.5.4 Systems Manager.

2.3.5.5 Programmer Analyst.

2.3.5.6 Inductively Coupled Plasma (ICP) Operators.

2.3.5.7 Inductively Coupled Plasma (ICP) Spectroscopist.

2.3.5.8 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) Operators.

2.3.5.9 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) Spectroscopist.

2.3.5.10 Graphite Furnace Atomic Absorption (GFAA) Operators.

2.3.5.11 Sample Preparation Laboratory Supervisor.

2.3.5.12 Sample Preparation Specialists.

2.3.5.13 Chemist (back-up).

NOTE: The Contractor shall designate a Sample Custodian and a Document Control Officer.

2.3.6 The Contractor shall respond within 10 days to requests from data recipients for additional information or explanations that result from the Government's inspection activities.

2.3.7 The Contractor is required to retain unused sample volumes and used sample containers for a period of 60 days after data submission unless otherwise instructed in Exhibit B or Exhibit D.

2.3.8 The Contractor shall adhere to the chain-of-custody and document control procedures described in Exhibit F. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported in the Complete Case File Purge (Exhibit B).

2.3.9 Sample shipments to the Contractor's facility will be scheduled and coordinated by SMO, acting on behalf of the Administrative Project Officer (APO). The Contractor shall communicate with SMO personnel by telephone as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

2.3.9.1 If there are problems with the samples (e.g., mixed media, containers broken) or sample documentation/paperwork (e.g., Traffic Reports not with shipment, or sample and Traffic Report numbers do not correspond) the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

2.3.10 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case. A Case consists of one or more SDG(s). An SDG is defined by the following:

2.3.10.1 Each Case of field samples received; or

2.3.10.2 Each 20 field samples within a Case; or

2.3.10.3 Each seven 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).

2.3.11 Data for all samples in an SDG must be submitted together (in one package) in the order specified in Exhibit B. The SDG number is the EPA number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number is the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. The SDG number is reported on all data reporting forms. The SDG Receipt Date is the day that the last sample in the SDG is received.

2.3.12 The Contractor is responsible for identifying each SDG as samples are received, through proper sample documentation (see Exhibit B) and communication with SMO personnel.

2.3.13 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report (TR) form bearing the sample number and descriptive information regarding the sample. The Contractor shall complete and sign the TR, recording the date of sample receipt and sample condition on receipt for each sample container.

2.3.14 The Contractor shall submit signed copies of TRs for all samples in an SDG to SMO within three calendar days following receipt of the last sample in the SDG. TRs shall be submitted in SDG sets (i.e., all TRs for a SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

2.3.15 EPA Case numbers (including SDG numbers) and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.

2.3.16 Samples will be routinely shipped directly to the Contractor through a delivery service. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays and holidays. As necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station, or other carrier service within the Contractor's geographical area.

2.3.17 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

SECTION 3

DETAILED TECHNICAL & MANAGEMENT REQUIREMENTS

The Contractor shall have the following technical and management capabilities:

3.1 PERSONNEL

3.1.1 Project Manager

3.2.1.1 Responsible for all technical efforts of the laboratory to meet all terms and conditions of the contract.

3.1.1.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.1.3 Experience: Minimum of three years of laboratory experience, including at least one year in a supervisory position.

3.1.2 Inorganic Laboratory Supervisor

3.1.2.1 Responsible for all technical efforts of the Inorganic laboratory to meet all terms and conditions of the contract.

3.1.2.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.2.3 Experience: Minimum of three years of laboratory experience in operating a ICP, ICP-MS, or AA, including at least one year in a supervisory position.

3.1.3 Quality Assurance Officer

3.1.3.1 Responsible for overseeing the quality assurance aspects of data generation and reporting directly to upper management.

3.1.3.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.3.3 Experience: Minimum of three years of laboratory experience, including at least one year of applied experience with QA principles and practices in an analytical laboratory.

3.1.4 Systems Manager

3.1.4.1 Responsible for the management and quality control of all computing systems (hardware, software, documentation, and procedures), generating, updating, and performing quality control on automated deliverables.

3.1.4.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, database management systems, or systems requirements analysis.

3.1.4.3 Experience: Minimum of three years experience in data or systems management or programming including one year experience with software used for data management and generation of deliverables.

3.1.5 Program Analyst

3.1.5.1 Responsible for the installation, operation, and maintenance of software and programs; generating, updating, and performing quality control procedures on analytical databases and automated deliverables.

3.1.5.2 Education: Minimum of Bachelor's degree with four or more intermediate courses in programming, information management, information systems, or systems requirements analysis.

3.1.5.3 Experience: Minimum of two years experience in systems or applications programming including one year of experience with software used for data management and generation of deliverables.

3.1.6 Inductively Coupled Plasma (ICP) Spectroscopist

3.1.6.1 Responsible for the operation, interpretation, and maintenance of the ICP spectrometer(s) to meet all terms and conditions of the contract.

3.1.6.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline and specialized training in ICP spectroscopy.

3.1.6.3 Experience: Minimum of two years of applied experience with ICP analysis of environmental samples.

3.1.7 ICP Operator

3.1.7.1 Responsible for the operation and maintenance of the ICP spectrometer(s) to meet all terms and conditions of the contract.

3.1.7.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.7.3 Experience: Minimum of one year of applied experience with ICP analysis of environmental samples; or in lieu of educational requirement, three additional years of experience operating and maintaining ICP instrumentation.

3.1.8 **ICP-MS Spectroscopist (Required if ICP-MS is to be used.)**

3.1.8.1 Responsible for the operation, interpretation, and maintenance of the ICP-MS spectrometer(s) to meet all terms and conditions of the contract.

3.1.8.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline and specialized training in ICP and ICP-MS spectroscopy.

3.1.8.3 Experience: Minimum of one year of applied experience with ICP-MS analysis of environmental samples and a minimum of two years of applied experience with ICP analysis of environmental samples.

3.1.9 **ICP-MS Operator (Required if ICP-MS is to be used.)**

3.1.9.1 Responsible for the operation and maintenance of the ICP-MS spectrometer(s) to meet all terms and conditions of the contract.

3.1.9.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.9.3 Experience: Minimum of one year of applied experience with ICP and ICP-MS analysis of environmental samples; or in lieu of educational requirement, three additional years of experience operating and maintaining ICP instrumentation including ICP-MS techniques.

3.1.10 **Graphite Furnace Atomic Absorption (GFAA) Operator**

3.1.10.1 Responsible for the operation and maintenance of the GFAA spectrometer(s) to meet all terms and conditions of the contract.

3.1.10.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.10.3 Experience: Minimum of one year of applied experience with AA analysis of environmental samples; or in lieu of educational requirement, three additional years of experience operating and maintaining AA instrumentation.

3.1.11 **Sample Preparation Laboratory Supervisor**

3.1.11.1 Responsible for all technical efforts of sample preparations to meet all terms and conditions of the EPA contract.

3.1.11.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.11.3 Experience: Minimum of three years of laboratory experience, including at least one year of supervisory experience.

3.1.12 **Sample Preparation Specialist**

3.1.12.1 Responsible for all aspects of sample preparations to meet all terms and conditions of the EPA contract.

3.1.12.2 Education: Minimum of high school diploma and a college level course in general chemistry or equivalent.

3.1.12.3 Experience: Minimum of six months of applied experience in an analytical laboratory.

3.1.13 **Technical Staff Redundancy**

3.1.13.1 In order to ensure continuous operations to accomplish the required work as specified by the contract, the bidder shall have a minimum of one chemist available at all times as a back-up technical person with the following qualifications.

3.1.13.2 Education: Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

3.1.13.3 Experience: Minimum of one year of experience in each of the following areas: ICP and ICP-MS operation and maintenance; AA operation and maintenance; classical chemistry analytical procedures; and sample preparation for metals analysis.

3.2 **FACILITIES**

The adequacy of the facilities and equipment is as important as the technical staff for accomplishing the required work as specified by the EPA contract.

3.2.1 **Sample Receipt Area**

Adequate, contamination-free, well-ventilated work space with chemical resistant bench top shall be available for receipt and safe handling of EPA samples.

3.2.2 **Storage Area**

Sufficient refrigerator space to maintain unused EPA sample volume for up to 60 days after data submission shall be provided. Volatile samples must be stored in a refrigerator used only for storage of volatile samples from this contract. Samples must be stored in an atmosphere demonstrated to be free from all potential contaminants. Samples, extracts, and standards must be stored separately.

3.2.3 **Sample Preparation Area**

Adequate, contamination-free, well-ventilated work space shall be provided with:

3.2.3.1 Benches with chemical resistant tops.

3.2.3.2 Exhaust hoods.

3.2.3.3 Glove box or isolated area in which to prepare standard materials.

3.2.3.4 Source of distilled or demineralized organic-free water.

3.2.3.5 Analytical balance(s) located away from draft and rapid change in temperature.

3.3 INSTRUMENTATION

At a minimum, the Contractor shall have the following instruments operative at the time of the Preaward Site Evaluation and committed for the full duration of the contract.

3.3.1 100 Samples/Month Capacity Requirements

Fraction	No. of Instrument(s)	Type of Instrument
ICP Metals	1	ICP Emission Spectrometer with MS Dectector (if appropriate)
GFAA Metals	1	Atomic Absorption Spectrometer with Graphite Furnace Atomizer

NOTE: The Contractor shall have the following instruments available (operational) at all times as a back-up system:

Quantity	Instruments
1	GFAA

These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.2 200 Samples/Month Capacity Requirements

Fraction	No. of Instrument(s)	Type of Instrument
ICP Metals	1	ICP Emission Spectrometer with MS Dectector (if appropriate)
GFAA Metals	1	Atomic Absorption Spectrometer with Graphite Furnace Atomizer

NOTE: The Secondary Instrument Requirement for 200 Samples/Month Capacity is one GFAA.

These instruments must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

3.3.3 Instrument Specifications

Further information on instrument specifications and required ancillary equipment may be found in Exhibit D and other Exhibits in this Contract.

3.4 DATA HANDLING AND PACKAGING

The Contractor shall be able to submit reports and data packages as specified in Exhibit B. To complete this task, the Contractor shall be required to:

- Provide space, tables, and copy machines to meet the contract requirements; and
- Designate personnel responsible for report preparations and submission.

3.5 LABORATORY MANAGEMENT CAPABILITY

The Contractor shall have an organization with well-defined responsibilities for each individual in the management system to ensure sufficient resources for EPA contract(s) and to maintain a successful operation. To establish this capability, the Contractor shall designate personnel to carry out the following responsibilities for the EPA contract. Functions include, but are not limited to, the following:

3.5.1 Technical Staff

Responsible for all technical efforts for the EPA contract such as sample preparation, sample analysis, sample validation, and trouble-shooting of all instruments.

3.5.2 **Project Manager**

Responsible for overall aspects of EPA contract(s) (from sample receipt through data delivery) and shall be the primary contact for EPA Headquarters APO and Regional Technical Project Officers (TPO).

3.6.3 **Sample Custodian**

Responsible for receiving the EPA samples (logging, handling, and storage).

3.6.4 **Quality Assurance Officer**

Responsible for overseeing the quality assurance aspects of the data and reporting directly to upper management.

3.6.5 **Document Control Officer**

Responsible for ensuring that all documents generated are placed in the Complete SDG File for inventory and are delivered to the appropriate EPA Region or other receiver as designated by EPA.

EXHIBIT B

REPORTING

AND

DELIVERABLES REQUIREMENTS

December, 1991

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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SECTION 1

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

The following table summarizes the contract reporting and deliverables requirements specified in the Contract Schedule and includes the distribution of each deliverable. **NOTE:** Specific recipient names and addresses are subject to change during the term of the contract. The EPA APO or SMO will notify the Contractor in writing of such changes when they occur.

Item	No. of Copies	Schedule and Delivery	Distribution		
			(1)	(2)	(3)
Updated Standard Operating Procedures (SOPs)	2	45 days after contract award.		X	X
*Sample Traffic Reports	1	***3 days after receipt of last sample in Sample Delivery Group (SDG).	X		
**Sample Data Package including the Performance Evaluation Sample (PES)	3	35 days after receipt of last sample in SDG	X	X	X
Results of Intercomparison Study/Preaward Performance Evaluation (PPE) Sample	2	35 days after receipt of last sample in SDG	X	X	
Complete SDG File	1	35 days after data receipt of last sample in SDG.		X	
*Verification of Parameters	1	15th day of January, April, July, and October.		X	
****Quality Assurance Plan	1	Submit copy within 7 days by written request by APO.	As Directed		

Distribution

(1) Sample Management Office
(2) Environmental Monitoring Systems Laboratory-Las Vegas
(3) USEPA Region

* Also required in each Sample Data Package.

** Concurrent delivery of these items to all recipients is required.

*** An SDG is a group of samples within a Case, received over a period of seven days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. (See Exhibit A, Task III, for further description).

**** See Exhibit E for description.

NOTE: As specified in the Contract Schedule in the IFB (Government Furnished Supplies and Materials), unless otherwise instructed by SMO, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of analytical data.

Address

(1) USEPA Contract Laboratory Program
Sample Management Office
P.O. Box 818
Alexandria, VA 22313

For overnight delivery service, use street address:

300 North Lee Street
Alexandria, VA 22313

(2) USEPA Environmental Monitoring Systems Laboratory
P.O. Box 93478
Las Vegas, NV 89193-3478
ATTN: Data Audit Staff

For overnight delivery service, use street address:

944 E. Harmon, Executive Center
Las Vegas, NV 89109
ATTN: Data Audit Staff

(3) USEPA REGIONS:

SMO, acting on behalf of the EPA APO, will provide the Contractor with the list of addresses for the 10 EPA Regions. SMO will provide the Contractor with updated Regional name/address lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

SECTION 2

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

The Contractor shall provide reports and other deliverables according to the schedule specified in Section F of the IFB, "SCHEDULE INFORMATION." The required content and form of each deliverable is described in this Exhibit.

All reports and documentation shall be:

- Legible;
- Clearly labeled and completed in accordance with instructions in this Exhibit;
- Arranged in the order specified in this section;
- Paginated; and
- Single-sided.

If submitted documentation does not conform to the above criteria, the Contractor will be required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the Government.

Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation or through an APO/TPO action, the data shall be clearly marked as "ADDITIONAL DATA" and shall be sent to all three contractual data recipients (SMO, EMSL-LV, and Region). A cover letter shall be included that describes which data are being delivered, to which EPA Case(s) the data pertain, and who requested the data.

Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with the required documentation and contains the required data forms in EPA-specified format.

Descriptions of the requirements for each deliverable item cited in the Contract Performance/Delivery Schedule (see Section F of the IFB "SCHEDULE INFORMATION") are specified as follows in this Section. Items submitted concurrently must be arranged in the order listed. Additionally, the components of each item must be arranged in the order presented herein.

2.1 UPDATED STANDARD OPERATING PROCEDURES

2.1.1 The Contractor shall submit updated copies of all required Standard Operating Procedures (SOPs) that were submitted with the Prebid Performance Evaluation (PPE) sample results. The updated SOPs must address any and all

issues of laboratory performance and operation identified by the Contractor in the review of the PPE sample data and the evaluation of Bidder-Supplied Documentation.

2.1.2 The Contractor must supply SOPs for the following.

2.1.2.1 Evidentiary SOPs.

2.1.2.2 Sample receipt and logging.

2.1.2.3 Sample and extract storage area.

2.1.2.4 Preventing sample contamination.

2.1.2.5 Security for laboratory and samples.

2.1.2.6 Traceability/equivalency of standards.

2.1.2.7 Maintaining instrument records and bound logbooks.

2.1.2.8 Glassware cleaning.

2.1.2.9 Technical and managerial review of laboratory operation and data package preparation.

2.1.2.10 Internal review of contractually-required QA/QC data for each individual data package.

2.1.2.11 Sample analysis, data handling, and data reporting.

2.1.2.12 Chain-of-custody and document control, including case file preparation.

2.1.2.13 Sample data validation/self-inspection system, including:

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to ensure that hardcopy data are complete and compliant with the requirements in Exhibit B;
- Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.);

- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

2.1.2.14 Data Handling.

2.1.2.14.1 Data Management procedures defined as written procedures that are clearly defined for all databases and files used to generate or re-submit deliverables specifying the acquisition or entry, update, correction, deletion, storage, and security of computer readable data and files. Key areas of concern include: system organization including personnel and security, demonstration, operations, traceability, and quality control.

2.1.2.14.2 Data manually entered from hardcopy must be subjected to quality control procedures and error rates estimated.

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

- Justification or rationale for the change;
- Initials of the person making the changes or changes. Data changes must be identified when generating the deliverables;
- Changed documentation must be retained according to the schedule of the original deliverable;
- Resubmitted deliverables must be reinspected as a part of the laboratory's internal inspection process prior to submission. The entire deliverable and not just the changes must be reinspected;
- The laboratory manager must approve changes to originally submitted deliverables; and
- Documentation of data changes may be requested by laboratory auditors.

2.1.2.14.4 Life Cycle Management procedures must be applied to computer systems used to generate and edit contract deliverables. Such systems must be thoroughly tested and documented prior to utilization.

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

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

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

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

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

- System operation and maintenance including documentation and training; and
- Database integrity including data entry, data updating and QC.

2.1.2.14.10 Data and system security, backup, and archiving.

2.2 SAMPLE TRAFFIC REPORTS

2.2.1 The original sample TR page marked "Lab Copy for Return to SMO" shall be submitted to SMO with laboratory receipt information and signed in original Contractor signature, for each sample in the SDG.

2.2.2 TRs shall be submitted in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

2.2.3 The SDG Cover Sheet shall contain the following items:

- Laboratory name;
- Contract number;
- Sample analysis price - full sample price from contract;

- Case number; and
- List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations), and the "last" sample received would be the highest sample number (considering both alpha and numeric designations).

2.2.4 Each TR shall be clearly marked with the SDG Number and the EPA sample number of the first sample in the SDG. This information shall be entered below the laboratory receipt date on the TR. The TR for the last sample received in the SDG shall be clearly marked "SDG - FINAL SAMPLE."

2.2.5 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the laboratory shall make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.

2.3 SAMPLE DATA PACKAGE

2.3.1 The sample data package shall be complete, consecutively paginated, and shall include data for analysis of all samples in an SDG. These samples include analytical and field samples, sample reanalyses, blanks, spikes, duplicates, and laboratory control samples.

2.3.2 The sample data package is divided into six units as follows:

- Cover page;
- Sample data (Results);
- Quality control summary;
- Raw data;
- Preparation logs; and
- Sample TRs.

2.3.2.1 Cover Page

This document shall be clearly labeled "Cover Page." The Cover Page shall contain: laboratory name; laboratory code; contract number; Case Number; SDG Number; SAS number (appears on cover page of SAS); EPA

sample numbers in alphanumeric order, showing EPA sample number cross-referenced with laboratory ID numbers; and comments, describing in detail any problems encountered in processing the samples in the data package.

The Cover Page shall contain the following statement, verbatim:

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature."

This statement shall be directly followed by the signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

In the event that the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

2.3.2.2 **Sample Data**

Sample data shall be submitted with FORM I - Analysis Data Sheet, for all samples in the SDG, arranged in increasing alphanumeric EPA sample number order.

2.3.2.3 **Quality Control Summary**

The quality control summary shall contain the following forms:

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

- Initial and Continuing Calibration Verification [FORM II - AAIN];
- CRQL Standards/Linear Range Standards (Quarterly) [FORM III - AAIN];
- Blanks [FORM IV - AAIN];
- ICP and ICP-MS Interference Check Sample [FORM V - AAIN];
- Spike Sample Recovery [FORM VI - AAIN];
- Duplicates [FORM VII - AAIN];
- Laboratory Control Sample [FORM VIII - AAIN];
- Method of Standard Additions [FORM IX - AAIN];

- ICP and ICP-MS Serial Dilutions [FORM X - AAIN];
- Method Quantitation Limits (Quarterly) [FORM XI - AAIN];
- ICP and ICP-MS Interelement Correction Factors (Annual) [FORM XII - AAIN];
- ICP-MS Tuning and Response Factor Criteria [FORM XIII - AAIN];
- ICP-MS Internal Standards Summary [FORM XIV - AAIN];
- Preparation Log [FORM XV - AAIN];
- Analysis Run Log [FORM XVI - AAIN]; and
- Standard Solutions Source [FORM XVII - AAIN].

2.3.2.4 **Raw Data**

2.3.2.4.1 For each reported value, the Contractor shall include all raw data from the instrument used to obtain the sample values (except for raw data for quarterly verifications of instrument parameters). Raw data shall contain all instrument readouts used for the sample results, including those readouts that may fall below the Method Quantitation Limit (MQL). All ICP, ICP-MS, and GFAA instruments must provide legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included.

2.3.2.4.2 The order of raw data in the data package shall be: ICP, ICP-MS, and GFAA. All raw data shall include concentration units for ICP and ICP-MS (if appropriate) and absorbencies or concentration units for GFAA.

2.3.2.4.3 Metal raw data must be labeled with EPA sample number and appropriate codes as shown in Table B-1, to identify unequivocally the following:

- Calibration standards, including source and preparation date;
- Initial and continuing calibration blanks and preparation blanks;
- Initial and continuing calibration verification standards, interference check samples, CRQL standard, and linear range standard;
- Diluted and undiluted samples (by EPA sample number) and all weights, dilutions, and volumes used to obtain the reported values. If the volumes, weights, and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient;
- Duplicates;

- Spikes (indicating standard solutions used, final spike concentrations, volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlying these parameters is sufficient;

- Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation;

- Data and EPA sample number for ICP, ICP-MS, and GFAA analyses clearly and sequentially identified on the raw data;

- All calculations for sample and analytical spike data, including percent recovery, coefficient of variation, slope and y-intercept of linear fit;

- Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide time of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as interference check samples and linear range analysis standards; and

- Integration times for GFAA analysis.

2.3.2.5 **Preparation Logs**

These logs must include the following:

- Date;

- Sample weights and volumes;

- Sufficient information to identify unequivocally which QC samples (i.e., laboratory control sample, preparation blank) correspond to each batch prepared; and

- Comments describing any significant sample changes or reactions which occur during preparation.

2.3.2.6 **Sample Traffic Report**

A legible copy of the sample TRs and SDG Cover Sheet shall be submitted as described in part 2.2 of this Exhibit for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample number order, considering both alpha and numeric designations.

TABLE B-1

Codes for Labelling Metal Data

Sample	XXXXXX
Duplicate	XXXXXXD
Sample Spike	XXXXXXS
Instrument Calibration Standards:	
ICP and ICP-MS	S or S0 for blank standard
Atomic Absorption	S0, S10, etc.
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
CRQL Standard for AA	CRA
CRQL Standard for ICP and ICP-MS	CRI
Laboratory Control Samples	LCS
Preparation Blank	PB
Linear Range Analysis Standard	LRS

NOTES:

1. When an MSA is performed on samples other than field samples, the "0", "1", "2" or "3" suffixes must be the last to be added to the EPA Sample Number. For instance, an MSA spike of a duplicate must be formatted "XXXXXXD0".

2. The numeric suffix that follows the "S" suffix for the standards indicates the true value of the concentration of the standard in µg/L.

3. ICP and ICP-MS calibration standards usually consist of several analytes at different concentrations. Therefore, no numeric suffix can follow the ICP and ICP-MS calibration standards unless all the analytes in the standard are prepared at the same concentrations.

4. The CRQL standard for AA is considered to be a calibration standard if it was a part of the calibration curve, thus it must be formatted like any other standard. The "CRA" format must be used if the CRQL standard for AA is not used to establish the calibration curve.

2.4 RESULTS OF INTERCOMPARISON/PERFORMANCE EVALUATION SAMPLE ANALYSES

The reporting of analytical results for Intercomparison Study/Preaward Performance Evaluation (PPE) sample analyses includes all requirements specified in part 2.3 for reporting of sample data. The PPE sample shall be carried through the exact same process as an analytical and field sample.

2.5 COMPLETE CASE FILE PURGE

2.5.1 The Complete SDG File package includes all laboratory records received or generated for a specific Case that have not been previously submitted to EPA as a deliverable. These items shall be submitted to EPA as a deliverable. These items shall be submitted along with their Document Inventory Sheet FORM AADC-2 (see Exhibit E for description of document numbering and inventory procedure). These items include, but are not limited to, sample tags, custody records, sample tracking records, analysts' logbook pages, bench sheets, instrument readout records, computer printouts, raw data summaries, instrument logbook pages (including instrument conditions), correspondence, and the document inventory.

2.5.2 Shipment of the Complete SDG File package by first class mail, overnight courier, priority mail or equivalent is acceptable. Custody seals, which are provided by EPA, shall be placed on shipping containers and a document inventory and transmittal letter included. The Contractor is not required to maintain any documents for a sample Case after submission of the Complete SDG File package; however, the Contractor should maintain a copy of the document inventory and transmittal letter.

2.6 VERIFICATION OF INSTRUMENT PARAMETERS

The Contractor shall perform and report quarterly verification of MQLs by methods specified in Exhibit D and E for each type and model number of instrument used under this contract. For ICP and ICP-MS instrumentations and methods, the Contractor shall also report annual interelement correction factors (including method of determination), wavelengths used, and integration times. Annual Verification of Instrument Parameters forms for the current year shall be submitted in each SDG data package, using appropriate forms. Submission of Quarterly Verification of Instrument Parameters shall include the raw data used to determine those values reported.

2.7 QUALITY ASSURANCE PLAN (QAP)

2.7.1 The Contractor shall prepare a written Quality Assurance Plan (QAP) which describes the procedures that are implemented to achieve the following: maintain data integrity, validity, and useability; ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility; detect problems through data assessment and established corrective action procedures which keep the analytical process

reliable; and document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

2.7.2 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, SOPs pertaining to each parameter shall be included or referenced as part of the QAP. The QAP must be available during on-site laboratory evaluation and upon written request by the APO.

SECTION 3

FORM INSTRUCTIONS GUIDE

This section contains specific instructions for the completion of all required Data Reporting Forms for analysis of metal constituents in ambient air. This section is organized into the following parts:

- General Information and Header Information
- Cover Page - [COVER PAGE - AAIN]
- Analysis Data Sheet [FORM I - AAIN]
- Initial and Continuing Calibration Verification [FORM II - AAIN]
- CRQL Standards/Linear Range Standards (Quarterly) [FORM III - AAIN]
- Blanks [FORM IV - AAIN]
- ICP Interference Check Sample [FORM V - AAIN]
- Spike Sample Recovery [FORM VI - AAIN]
- Duplicates [FORM VII - AAIN]
- Laboratory Control Sample [FORM VIII - AAIN]
- Method of Standard Additions [FORM IX - AAIN]
- ICP Serial Dilution [FORM X - AAIN]
- Method Quantitation Limits (Quarterly) [FORM XI - AAIN]
- ICP and ICP-MS Interelement Correction Factors (Annual) [FORM XII - AAIN]
- ICP-MS Tuning and Response Factor Criteria [FORM XIII - AAIN]
- ICP-MS Internal Standards Summary [FORM XIV - AAIN]
- Preparation Log [FORM XV - AAIN]
- Analysis Run Log [FORM XVI - AAIN]
- Standard Solutions Sources [FORM XVII - AAIN]
- Sample Log-In Sheet [FORM AADC-IN-1]
- Document Inventory Sheet [FORM AADC-IN-2]

3.1 GENERAL INFORMATION AND HEADER INFORMATION

3.1.1 Values must be reported on the hardcopy forms according to the individual form instructions in this Section. Each form submitted must be filled out completely for all analytes. Multiple forms cannot be submitted in place of one form if the information on those forms can be submitted on one form.

3.1.2 For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5 and there are no digits to the right of the 5 or all digits to the right of the 5 equals zero, then round up if the digit to be retained is odd, or round down if that digit is even.

3.1.3 All characters which appear on the data reporting forms presented in the contract must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the EPA APO. The names of the various fields and analytes (i.e., "Lab Code", "Aluminum") on the forms must appear as they do on the forms, except that the use of uppercase and lowercase letters is optional.

3.1.4 All alphabetic entries made onto the forms by the Contractor must be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low").

3.1.5 Six pieces of information are common to the header sections of each data reporting form. These are: Lab Name, Contract, Lab Code, Case No., SAS No., and SDG No. This information must be entered on every form and must match on all forms.

3.1.5.1 The "Lab Name" is the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

3.1.5.2 The "Contract" is the number of the EPA contract, including hyphens, under which the analyses were performed.

3.1.5.3 The "Lab Code" is an alphabetic abbreviation of up to 6 characters, assigned by the EPA, to identify the laboratory and aid in data processing. This lab code shall be assigned by the EPA at the time a contract is awarded, and must not be modified by the Contractor, except at the direction of EPA. If a change of name or ownership occurs at the laboratory, the lab code will remain the same until the Contractor is directed by the EPA to use another lab code assigned by the EPA.

3.1.5.4 The "Case No." is the EPA-assigned Case number (up to 5 characters) associated with the sample and recorded on the TR.

3.1.5.5 The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services (SAS). If samples are to be analyzed under SAS only and reported on these forms, then enter SAS No. and leave Case No. blank. If samples are analyzed according to this Contract and have additional SAS requirements, then list both Case No. and SAS No. on all forms. If the analyses have no SAS requirements, leave "SAS No." blank.

NOTE: Some samples in an SDG may have a SAS No., while others may not.

3.1.5.6 The "SDG No." is the Sample Delivery Group number. The SDG number is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number must be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

3.1.6 The EPA sample number is the unique identifying number given in the TR that accompanied that sample. This number is assigned by the EPA and it must be used exactly as assigned.

3.1.6.1 The "EPA SAMPLE No." must be entered on several of the forms. This number appears either in the upper right hand corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA SAMPLE No." is entered into the triple-spaced box in the upper right hand corner of a form, it must be centered on the middle line of the three lines that comprise the box.

3.1.6.2 All field samples and quality control samples associated with field samples must be identified with an EPA sample number. In addition, the sample suffix and quality control sample abbreviations listed in Table B-1, Section 2 of this Exhibit must be used as appropriate.

3.1.7 All results must be transcribed to FORMs II-XV in the raw data with the specified number of decimal places that are described in Exhibit B. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros must be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry on Form
5.9	6.3 (to three decimal places)	5.900
5.99653	6.3 (to three decimal places)	5.997
95.99653	6.3 (to three decimal places)	95.997
995.99653	6.3 (to three decimal places)	996.0
9995.996	6.3 (to three decimal places)	9996.0
99995.9	6.3 (to three decimal places)	99996.0
999995.9	6.3 (to three decimal places)	invalid

NOTE: 6.3 stands for a maximum of six significant figures and up to three decimal places.

3.1.8 Before evaluating a number for being in control or out of control in relationship to a certain limit, the number evaluated must be rounded using EPA rounding rules to the significance reported for that limit. For instance, the control limit for an ICV is ± 10 percent of the true value. A percent recovery value of 110.4 would be considered in control while a value of 110.6 would be considered out of control. In addition, a calculated value of 110.50 would be in control while a calculated value of 110.55 would be out of control.

3.2 COVER PAGE [Cover Page - AAIN]

3.2.1 This form is used to list all billable samples analyzed within an SDG, and to provide certain analytical information and general comments. It is also the document which is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

3.2.2 Under "EPA Sample No.," enter up to seven characters for the EPA sample number (including spikes and duplicates) for each required analysis within the SDG. Samples spikes must contain an "S" suffix and duplicates a "D" suffix. Other spikes (analytical, post digestion/distillation) must have an "A" suffix. These sample numbers must be listed on the form in ascending alphanumeric order using the Extended Binary Coded Decimal Interchange Code convention. Thus, if MAB123A is the lowest (considering both alpha and numeric characters) EPA sample number within the SDG, it would be entered in the first EPA sample number field. Samples listed below it would be in ascending sequence - MAB124A, MAB124B, MAB125A, MAC111A, MA1111A, MA1111AD, etc.

3.2.3 All EPA sample numbers must be listed in ascending alphanumeric order, continuing to the following Cover Page if applicable.

3.2.4 Under "Lab Sample ID," a laboratory sample identification (up to ten characters) may be entered for each associated EPA sample number. If a laboratory sample ID is entered, it must be entered identically (for each EPA Sample No.) on all associated data.

3.2.5 Enter "Y" or "N" for "YES" or "NO," respectively, in answer to each of the two questions concerning ICP and ICP-MS corrections. Each question must be explicitly answered with a "Y" or an "N." The third question must be answered with a "Y" or "N" if the answer to the second question is "Y." It should be left blank if the answer to the second question is "N."

3.2.6 Under "Comments," enter any problems encountered, both technical and administrative, the corrective action taken, and resolution performed for all of the samples in the SDG.

3.2.7 Each Cover Page must be signed, in original, by the Laboratory Manager or the Manager's designee, and dated to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.3 ANALYSIS DATA SHEET [FORM I - AAIN]

3.3.1 This form is used to tabulate and report sample analysis results for target analytes (Exhibit C).

3.3.2 Complete the header information according to the header instructions and as follows.

3.3.3 For "Lab Sample ID," enter the laboratory sample identification for the sample, as listed on the Cover Page.

3.3.4 For "Date Received," enter the date (formatted MM/DD/YY) the sample was received at the laboratory, as recorded on the TR [i.e., the Validated Time of Sample Receipt (VTSR)].

3.3.5 For "Air Volume Sampled, Std. m^3," enter the air volume sampled (to two decimal places), as recorded on the TR.

3.3.6 For "Date Analyzed," enter the date (formatted MM/DD/YY) the sample was analyzed by the laboratory.

3.3.7 Under "CAS No.," enter the chemical abstract services register number for the analysis if applicable. If a case number doesn't exist, enter "NA" in the space provided.

3.3.8 Under the column labeled "CONCENTRATION," if the analytical result is greater than or equal to the MQL, report the result. If the result is lower than the MQL, report the CRQL value.

NOTE: Analytical results must be reported to two significant figures if the result value is less than ten, and to three significant figures if the result value is greater than or equal to ten.

NOTE: The requirement for reporting results to two or three significant figures applies to FORM I-AAIN only. Follow the specific instructions for reporting all other results on required forms as described in this Exhibit.

3.3.8.1 Under "µg/L," enter the value obtained (to two significant figures if the result value is less than ten, and to three significant figures if the result value is greater than or equal to ten) for the analyte.

3.3.8.2 Under "µg/m³," enter the value obtained (to two significant figures if the result value is less than ten, and to three significant figures if the result value is greater than or equal to ten) from the following equation:

$$\text{Analyte Conc. air, } \mu g/m^3 = \frac{9 \times \text{Interference-Corrected Analyte Value, } \mu g/L \times \text{Extract Volume, L}}{\text{no. of strips digested} \times \text{air volume sampled, std. } m^3} \qquad \text{Eq. B-1}$$

3.3.9 Under the columns labeled "C," "Q," and "M" enter result qualifiers as identified in the following paragraphs. If additional qualifiers are used, their explicit definitions must be included on the Cover Page in the Comments section.

3.3.10 FORM I-AAIN includes fields for three types of result qualifiers. These qualifiers must be completed as follows:

3.3.10.1 C (Concentration) qualifier: Enter "B" if the reported value was obtained from a reading that was less than the CRQL but greater than or equal to the MQL. Enter "U" if the reported value was obtained from a reading that was less than the MQL.

3.3.10.2 Q (Quality Control) qualifier: Specified entries and their meanings are as follows:

- E - The reported value is estimated because of the presence of interference;
- I - The sum of the values of the interference correction(s) is greater than the result concentration;
- M - Duplicate injection/exposure precision not met;
- N - Spiked sample recovery not within control limits;

- S - The reported value was determined by the Method of Standard Additions (MSA);
- * - Duplicate analysis not within control limits; and
- + - Correlation coefficient for the MSA is less than 0.995.

NOTE: Entering "S," or "+" is mutually exclusive. No combination of these qualifiers can appear in the same field for an analyte.

3.3.10.3 M (Method) qualifier: Enter, as appropriate, the following:

- "P" for ICP;
- "PM" for ICP-MS;
- "A" for Flame Atomic Absorption;
- "F" for Graphite Furnace Atomic Absorption; or
- NR if the analyte is not required to be analyzed.

3.3.11 Under "Comments," enter any sample-specific comments concerning the analyte results and note any significant changes that occurred during sample analysis (e.g., MSA determination, interferences), both technical and administrative, the corrective action taken, and resolution performed for the sample in the SDG.

3.4 **INITIAL AND CONTINUING CALIBRATION VERIFICATION [FORM II - AAIN]**

3.4.1 This form is used to report analyte recoveries from analyses of calibrations solutions.

3.4.2 Complete the header information according to the header instructions and as follows.

3.4.3 Enter the "Initial Calibration Source" (twelve characters maximum) and the "Continuing Calibration Source" (twelve characters maximum). Enter "EPA-LV" or "EPA-CI" to indicate EPA EMSL-Las Vegas or EMSL-Cincinnati, respectively, as the source of EPA standards. When additional EPA supplied solutions are prepared in the future, the Contractor must use the codes supplied with those solutions for identification. If other sources were used, enter sufficient information in the available twelve spaces to identify the manufacturer and the solution used.

3.4.4 Use additional copies of FORM II-AAIN if more calibration sources were used.

3.4.5 Under "WOMN," enter the number of the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength number is a number assigned to each wavelength used when more

than one wavelength is used to obtain data for analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is assigned to the greatest mass used for the analyte in the SDG. A mass number "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength or mass is used to obtain data for an analyte in the SDG.

3.4.6 Under "INITIAL CALIBRATION True," enter the true concentration (in µg/L, to three decimal places) of each analyte in the Initial Calibration Verification Solution.

3.4.7 Under "INITIAL CALIBRATION Found," enter the most recent found concentration (in µg/L, to three decimal places) of each analyte measured in the Initial Calibration Verification Solution.

3.4.8 Under "INITIAL CALIBRATION %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{Found\,(ICV)}{True\,(ICV)} \times 100 \qquad \text{Eq. B-2}$$

Where True (ICV) is the true concentration of the analyte in the Initial Calibration Verification solution and Found (ICV) is the found concentration of the analyte in the Initial Calibration Verification Solution.

3.4.9 Under "CONTINUING CALIBRATION True," enter the true concentration (in µg/L, to three decimal places) of each analyte in the Continuing Calibration Verification Solution.

3.4.10 Under "CONTINUING CALIBRATION Found," enter the found concentration (in µg/L, to three decimal places) of each analyte measured in the Continuing Calibration Verification Solution.

NOTE: The form contains two "CONTINUING CALIBRATION Found" columns. The column to the left must contain values for the first Continuing Calibration Verification and the column to the right must contain values for the second Continuing Calibration Verification. The column to the right should be left blank if no second Continuing Calibration Verification was performed.

3.4.11 If more than one FORM II-AAIN is required to report multiple Continuing Calibration Verifications, then the column to the left on the second form must contain values for the third Continuing Calibration Verification, the column to the right must contain values for the fourth Continuing Calibration Verification, and so on.

3.4.12 Under "CONTINUING CALIBRATION %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{Found\,(CCV)}{True\,(CCV)} \times 100 \qquad \text{Eq. B-3}$$

Where True (CCV) is the true concentration of each analyte, and Found (CCV) is the found concentration of the analyte in the Continuing Calibration Verification Solution.

NOTE: The form contains two "CONTINUING CALIBRATION %R" columns. Entries to these columns must follow the sequence detailed above for entries to the "Continuing Calibration Found" columns.

3.4.13 Under "M," enter the method used, as in part 3.3.10.3.

3.4.14 If more than one wavelength or mass is used to analyze an analyte, then submit additional copies of FORM II-AAIN as appropriate.

3.4.15 The order of reporting ICVs and CCVs for each analyte must follow the chronological order in which the standards were run starting with the first FORM II-AAIN and moving from the left to the right, continuing to the following FORM II-AAIN as appropriate. For instance, the first ICV for all analytes must be reported on the first FORM II-AAIN. In a run where three CCVs were analyzed, the first CCV must be reported in the left CCV column on the first FORM II-AAIN and the second CCV must be reported in the right column of the same form. The third CCV must be reported in the left CCV column of the second FORM II-AAIN. On the second FORM II-AAIN, the ICV column and the right CCV column must be left empty in this example. In the previous example, if a second run for an analyte was needed, the ICV of that run must be reported on a third FORM II-AAIN and the CCVs follow in the same fashion as explained before.

3.4.16 In the case where more than one wavelength or mass is used for an analyte in the SDG, all ICV and CCV results of the longest wavelength from all runs must be reported before proceeding to report the results of the second wavelength or mass used and so on.

3.4.17 Under "Comments," enter any ICV and CCV specific comments concerning the analyte results, any significant problems encountered during the ICV and CCV analysis (i.e., percent recovery outside the control limits, interferences), both technical and administrative, the corrective action taken, and resolution performed for the ICV and CCV.

3.5 CONTRACT REQUIRED QUANTITATION LIMIT STANDARDS/LINEAR RANGE STANDARDS (Quarterly) [FORM III - AAIN]

3.5.1 Contract Required Quantitation Limit Standards (CRQL)

3.5.1.1 This form is used to report analyte recoveries from analyses of the CRQL Standards.

3.5.1.2 Complete the header information according to the header instructions and as follows.

3.5.1.3 Under "CRQL," enter the sources of the CRQL Standards for "ICP Source," "ICP-MS Source," and "GFAA Source" analyses in their respective fields (twelve characters maximum each), as explained in part 3.4.3.

3.5.1.4 Under "WOMN," enter the wavelength or mass number as explained in part 3.4.5.

3.5.1.5 Under "True," enter the true concentration (in µg/L, to three decimal places) of each analyte in the CRQL Standard Source Solution that was analyzed for analytical samples associated with the SDG.

3.5.1.6 Under "INITIAL Found," enter the found concentration (in µg/L, to three decimal places) of each analyte measured in the CRQL Standard Solution analyzed at the beginning of each run.

3.5.1.7 Under "INITIAL %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\textit{CRQL Standard Initial Found}}{\textit{CRQL Standard True}} \times 100 \qquad \text{Eq. B-4}$$

3.5.1.8 Under "FINAL Found," enter the found concentration (in µg/L, to three decimal places) of each analyte measured in the CRQL Standard Solution analyzed at the end of each run.

3.5.1.9 Under "FINAL %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\textit{CRQL Standard Final Found}}{\textit{CRQL Standard True}} \times 100 \qquad \text{Eq. B-5}$$

NOTE: For every initial solution reported there must be a final one. However, the opposite is not true. If a CRQL Standard was required to be analyzed in the middle of a run (to avoid exceeding the 8-hour limit), it must be reported in the "FINAL Found" section of this form.

3.5.1.10 If more CRQL standards analyses were required or analyses were performed using more than one wavelength or mass per analyte, submit additional copies of FORM III-AAIN in the appropriate order.

3.5.1.11 The order of reporting CRQL standards for each analyte must follow the chronological order in which the standards were run starting with the first FORM III-AAIN and continuing to the following FORM III-AAIN as appropriate. When multiple wavelengths and masses are used for

one analyte, all the results of one wavelength must be reported before proceeding to the next wavelength or mass.

3.5.1.12 Under "Comments," enter any CRQL-specific comments concerning the analyte results, any significant problems encountered during the CRQL standard analysis (e.g., percent recovery outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the standard.

3.5.2 **Linear Range Standards (LRS) (Quarterly)**

3.5.2.1 FORM III-AAIN is also used to report analyte recoveries from the quarterly analyses of the Linear Range Standards (LRSs).

3.5.2.2 Complete the header information according to the header instructions and as follows.

3.5.2.3 Under "LRS," enter the source of the LRSs for "ICP Source", "ICP-MS Source", and "GFAA Source" analyses in their respective fields (12 characters maximum each), as explained in part 3.4.3.

3.5.2.4 Under "INITIAL True," enter the true concentration (in µg/L, to three decimal places) of each analyte in the LRS Source Solution that was analyzed for the analytical samples associated with the SDG.

3.5.2.5 Under "INITIAL Found," enter the found concentration (in µg/L, to three decimal places) of each analyte measured in the LRS Solution analyzed at the beginning of each run.

3.5.2.6 Under "INITIAL %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\textit{LRS Initial Found}}{\textit{LRS True}} \times 100 \qquad \text{Eq. B-6}$$

3.5.2.7 Under "FINAL Found," enter the found concentration (in µg/L, to three decimal places) of each analyte measured in the LRS Solution analyzed at the end of each run.

3.5.2.8 Under "FINAL %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\textit{LRS Final Found}}{\textit{LRS True}} \times 100 \qquad \text{Eq. B-7}$$

3.5.2.9 If more LRS analyses were required or analyses were performed using more than one wavelength per analyte, submit additional copies of FORM III-AAIN in the appropriate order.

3.5.2.10 The order of reporting the LRS for each analyte must follow the chronological order in which the standards were run starting with the first FORM III-AAIN and continuing to the following FORM III-AAIN as appropriate. If multiple wavelengths or masses are used for one analyte, all the results of one wavelength or mass must be reported before proceeding to the next wavelength or mass.

3.5.2.11 Under "Comments," enter any LRS-specific comments concerning the analytes results, any significant problems encountered during the LRS analysis (e.g., percent recovery outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the standard.

3.6 BLANKS [FORM IV - AAIN]

3.6.1 This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), the Continuing Calibration Blanks (CCB), and the Preparation Blank (PB).

3.6.2 Complete the header information according to the header instructions and as follows.

3.6.3 Under "WOMN," enter the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength or mass number is a number assigned to each wavelength or mass used when more than one wavelength or mass is used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is assigned to the greatest mass used for the analyte in the SDG. A mass number of "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength or mass is used to obtain data for an analyte in the SDG.

3.6.4 Under "INITIAL CALIB. BLANK (ICB)," enter the concentration (in µg/L, to three decimal places) of each analyte in the most recent Initial Calibration Blank (ICB).

3.6.5 For all blanks, enter the concentration of each analyte (positive or negative) measured above the MQL or below the negative value of the MQL.

3.6.6 Under the "C" qualifier field, for any analyte enter "B" if the absolute value of the analyte concentration is less than the CRQL but greater than or equal to the MQL. Enter "U" if the absolute value of the analyte in the blank is less than the MQL.

3.6.7 Under "CONTINUING CALIBRATION BLANK 1," enter the concentration (in µg/L, to three decimal places) of each analyte detected for the first CCB analyzed after the ICB. Enter any appropriate qualifier, as

explained for the "Initial Calibration Blank," in the "C" qualifier column immediately following the "CONTINUING CALIBRATION BLANK (CCB)" column.

3.6.8 If only one CCB was analyzed, then leave the columns labeled "Conc. 2" and "Conc. 3" blank. If up to three CCBs were analyzed, complete the columns labeled "Conc. 2" and "Conc. 3," in accordance with the instructions for the "CONTINUING CALIBRATION BLANK (CCB)" column. If more than three CCBs were analyzed, then complete additional copies of FORM IV-AAIN as appropriate.

3.6.9 Under "PREPARATION BLANK (PB)," enter the concentration (in µg/L, to three decimal places) of each analyte in the PB. Enter any appropriate qualifier, as explained for the ICB, in the "C" qualifier column immediately following the "PREPARATION BLANK" column.

3.6.10 Under "M," enter the method used, as explained in part 3.3.10.3.

3.6.11 If more than one wavelength or mass is used to analyze an analyte, submit additional copies of FORM IV-AAIN as appropriate.

3.6.12 The order of reporting ICBs and CCBs for each analyte must follow the chronological order in which the blanks were run starting with the first FORM IV-AAIN and moving from left to right and continuing to the following FORM IV-AAIN as explained previously. When multiple wavelengths or mass are used for the analysis of one analyte, all the results of one wavelength or mass must be reported before proceeding to the next wavelength or mass.

3.6.13 Under "Comments," enter any ICB and CCB specific comments concerning the analyte results, any significant problems encountered during the ICB, CCB and PB analysis (e.g., blanks outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.7 ICP INTERFERENCE CHECK SAMPLE [FORM V - AAIN]

3.7.1 This form is used to report ICP Interference Check Sample (ICS) results for each ICP and/or ICP-MS instruments used in SDG analyses.

3.7.2 Complete the header information according to the header instructions and as follows.

3.7.3 For "ICP ID No.," enter an identifier that uniquely identifies the specific instrument within the Contractor laboratory. No two ICP instruments within a laboratory may have the same ICP ID Number. If ICP-MS is used, leave blank.

3.7.4 For "ICP-MS ID No.," enter an identifier that uniquely identifies the specific instrument within the Contractor laboratory. No

two ICP-MS instruments within a laboratory may have the same ICP-MS ID Number. If ICP is used leave blank.

3.7.5 For "ICS Source," enter the ICS source (twelve characters maximum each), as previously explained in part 3.4.3. For EPA solutions, include the name and number identifying it (e.g., EPA-LV87). The laboratory must use the identification supplied by the EPA.

3.7.6 Under "WOMN," enter the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength number is a number assigned to each wavelength used when more than one wavelength is used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is assigned to the greatest mass used for the analyte in the SDG. A mass number of "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength or mass is used to obtain data for an analyte in the SDG.

3.7.7 Under "TRUE Sol. A," enter the true concentration (in µg/L, to three decimal places) of each analyte present in Solution A.

3.7.8 Under "TRUE Sol. AB," enter the true concentration (in µg/L, to three decimal places) of each analyte present in Solution AB.

3.7.9 Under "INITIAL FOUND Sol. A," enter the found concentration (in µg/L to three decimal places) of each analyte measured in the initial analysis of Solution A as required in Exhibit E.

3.7.10 Under "INITIAL FOUND Sol. AB," enter the found concentration (in µg/L to three decimal places) of each analyte measured in the initial analysis of Solution AB as required in Exhibit E.

3.7.11 Under initial found "%R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\textit{Initial Found Solution AB}}{\textit{True Solution AB}} \times 100 \qquad \text{Eq. B-8}$$

3.7.12 Under "FINAL FOUND," enter the found concentration (in µg/L, to three decimal places) of each analyte measured in the final analysis as required in Exhibit E.

3.7.13 For all found values, enter the concentration (positive, negative, or zero) of each analyte at each wavelength or mass used for analysis by ICP or ICP-MS.

3.7.14 Under "FINAL FOUND %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\textit{Final Found Solution AB}}{\textit{True Solution AB}} \times 100 \qquad \text{Eq. B-9}$$

NOTE: For every initial solution reported there must be a final one. However, the opposite is not true. If an ICS was required to be analyzed in the middle of a run (to avoid exceeding the 8-hour limit), it must be reported in the "FINAL FOUND" section of this form.

3.7.15 If more ICS analyses were required, submit additional copies of FORM V-AAIN as appropriate.

3.7.16 The order of reporting ICSs for each analyte must follow the temporal order in which the standards were run starting with the first FORM V-AAIN and continuing to the following FORM V-AAIN as appropriate. When multiple wavelengths or masses are used for one analyte, all the results of one wavelength must be reported before proceeding to the next wavelength in the same manner.

3.7.17 Under "Comments," enter any ICS specific comments concerning the analyte results, any significant problems encountered during the ICS analysis (e.g., percent recovery outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.8 SPIKE SAMPLE RECOVERY [FORM VI - AAIN]

3.8.1 This form is used to report results for the spike sample recovery which is based on the addition of a known quantity of analyte to the pre-digest sample.

3.8.2 Complete the header information according to the header instructions and as follows.

3.8.3 In the "EPA SAMPLE No." box, enter the EPA sample number (7 characters maximum) of the sample from which the spike results on this form were obtained. The number must be centered in the box. Note that the EPA sample number must include the spike sample suffix for which the spike analyses are reported.

3.8.4 Under "WOMN," enter the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength or mass number is a number assigned to each wavelength or mass used when more than one wavelength or mass is used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is

assigned to the greatest mass used for the analyte in the SDG. A mass number of "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength or mass is used to obtain data for an analyte in the SDG.

3.8.5 Under "CONTROL LIMIT %R," enter "75-125" if the spike added value was greater than or equal to one-fourth of the sample result value. If not, leave the field empty.

3.8.6 Under "SPIKED SAMPLE RESULT (SSR)," enter the concentration (in µg/L, to three decimal places) of each analyte in the spike sample. Enter any appropriate qualifier in the "C" qualifier column immediately following the Spiked Sample Result (SSR) column.

3.8.7 Under "SAMPLE RESULT (SR)," enter the concentration (in µg/L, to three decimal places) of each analyte measured in the sample (reported in the EPA sample number box) on which the matrix spike was performed. Enter any appropriate qualifier in the "C" qualifier column immediately following the Sample Result (SR) column.

3.8.8 Under "SPIKE ADDED (SA)," enter the concentration (in µg/L, to three decimal places) of each analyte added to the sample. If the spike added concentration is specified in the contract, the value added and reported must be that specific concentration in µg/L.

3.8.9 Under "%R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{SSR - SR}{SA} \times 100 \qquad \text{Eq. B-10}$$

NOTE: %R must be reported, whether it is negative, positive or zero. A value of zero must be used for SSR or SR if the analyte value is less than the MQL.

3.8.10 Under "Q," enter "N" if the spike recovery (%R) is out of the control limits (75-125%) and the spiked added is greater than or equal to one-fourth of the sample result.

3.8.11 Under "M," enter the method used or enter "NR" if the analyte is not required in the spike.

NOTE: If different samples were used for spike sample analysis of different analytes, additional copies of FORM VI-AAIN must be submitted for each sample as appropriate.

3.8.12 Use additional copies of FORM VI-AAIN for each sample on which a required spike sample analysis was performed.

3.8.13 Under "Comments," enter any spike sample specific comments concerning the analyte results, any significant problems encountered during the spike sample analysis (e.g , percent recovery outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.9 DUPLICATES [FORM VII - AAIN]

3.9.1 This form is used to report results of duplicate analyses for determining the precision of the method.

3.9.2 Complete the header information according to the header instructions and as follows.

3.9.3 In the "EPA SAMPLE No." box, enter the EPA sample number (7 characters maximum) of the sample from which the duplicate results on this form were obtained. The number must be centered in the box.

3.9.4 Under "WOMN," enter the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength or mass number is a number assigned to each wavelength or mass used when more than one wavelength or mass is used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is assigned to the greatest mass used for the analyte in the SDG. A mass number of "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength is used to obtain data for an analyte in the SDG.

3.9.5 Under "CONTROL LIMIT," enter the CRQL (in appropriate units, µg/L) for the analyte if the sample or duplicate values were less than five times CRQL and greater than or equal to the CRQL. If the sample and duplicate values were less than the CRQL or greater than or equal to five times CRQL, leave the field empty.

3.9.6 Under "SAMPLE (S)," enter the concentration (in the required concentration units, to three decimal places) of each analyte in the original sample (reported in the EPA sample number box) on which a duplicate analysis was performed. Enter any appropriate qualifier in the "C" qualifier column immediately following the "SAMPLE (S)" column.

3.9.7 Under "DUPLICATE (D)," enter the concentration (in the required concentration units, to three decimal places) of each analyte measured in the Duplicate sample (reported in the EPA sample number box). Enter any appropriate qualifier in the "C" qualifier column immediately following the "DUPLICATE (D)" column.

3.9.8 Under "RPD," enter the absolute value (to one decimal place) of the Relative Percent Difference for all analytes detected above the MQL

in either the sample or the duplicate, computed according to the following equation:

$$RPD = \frac{|S - D|}{\frac{S + D}{2}} \times 100 \qquad \text{Eq. B-11}$$

3.9.9 A value of zero must be substituted for S or D if the analyte concentration is less than the MQL in either one. If the analyte concentration is less than the MQL in both S and D, leave the RPD field empty.

3.9.10 Under "Q," enter "*" if the duplicate analysis for the analyte is out of the control limits. If both sample and duplicate values are greater than or equal to five times CRQL, then the RPD must be less than or equal to 20 percent to be in control. If either sample or duplicate values are less than five times CRQL, then the absolute difference between the two values must be less than or equal to the CRQL to be in control. If both values are below the CRQL, then no control limit is applicable.

3.9.11 Under "M," enter the method used.

3.9.12 If different samples were used for duplicate sample analysis of different analytes, then additional copies of FORM VII-AAIN must be submitted for each sample as appropriate.

3.9.13 Use additional copies of FORM VII-AAIN for each sample on which a required duplicate sample analysis was performed.

3.9.14 Under "Comments," enter any duplicate sample specific comments concerning the analyte results, any significant problems encountered during the duplicate sample analysis (i.e., percent recovery outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.10 LABORATORY CONTROL SAMPLE [FORM VIII-AAIN]

3.10.1 This form is used to report results for the Laboratory Control Sample (LCS).

3.10.2 Complete the header information according to the header instructions and as follows.

3.10.3 For "LCS USED," enter the appropriate source solution identifier (12 characters maximum) provided by the EPA for the LCS solution that was analyzed by the methods in Exhibit D.

3.10.4 If no analyte was analyzed by a certain method or if the analyte was not required to be analyzed, then leave the appropriate spaces empty.

3.10.5 Under "WOMN," enter the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength or mass number is a number assigned to each wavelength or mass used when more than one wavelength or mass is used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is assigned to the greatest mass used for the analyte in the SDG. A mass number of "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength or mass is used to obtain data for an analyte in the SDG.

3.10.6 Under "M," enter the method used.

3.10.7 Under "LIMITS," enter the lower limit concentration value (to the nearest whole number) in the left column, and the upper limit concentration value (to the nearest whole number) in the right column for each analyte in the LCS according to the ± 20 percent criteria.

3.10.8 Under "TRUE," enter the true concentration (to three decimal places) of each analyte in the LCS.

3.10.9 Under "FOUND," enter the found concentration (to three decimal places) of each analyte measured in the LCS.

3.10.10 Under "C," enter "B" or "U" or leave empty to describe the found value of the LCS.

3.10.11 Under "%R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{LCS\ FOUND}{LCS\ TRUE} \times 100 \qquad \text{Eq. B-12}$$

3.10.12 If the analyte concentration is less than the MQL, a value of zero must be substituted for the LCS Found.

3.10.13 Submit additional copies of FORM VIII-AAIN as appropriate, if more than one LCS was required. In addition, submit additional copies of FORM VIII-AAIN if more than one wavelength was used to determine an analyte for a sample.

3.10.14 Under "Comments," enter any LCS specific comments concerning the analyte results, any significant problems encountered during the LCS analysis (e.g., percent recovery outside the control limits), both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.11 METHOD OF STANDARD ADDITIONS [FORM IX - AAIN]

3.11.1 This form is used to report the results of samples analyzed using the Method of Standard Additions (MSA) for GFAA analysis.

3.11.2 Complete the header information according to the header instructions and as follows.

3.11.3 Under "EPA SAMPLE No.," enter the EPA sample numbers (nine characters maximum) of the sample from which the standard additions results on this form were obtained.

NOTE: Only field samples and duplicates may be reported on this form, thus the EPA sample number usually has no suffix or a "D".

3.11.4 A maximum of 32 samples can be entered on this form. If additional samples required MSA, submit additional FORMs IX-AAIN. Samples are listed in alphanumeric order per analyte. Submit additional FORMs IX-AAIN as appropriate.

3.11.5 Under "An," enter the chemical symbol (two characters maximum) for each parameter for which MSA was required for each sample listed. The parameters must be in alphabetic listing of the chemical symbols.

NOTE: Results for different samples for each parameter must be reported sequentially, with the analytes ordered according to the alphabetic listing of their chemical symbols. For instance, results for Al (Aluminum) in samples BBB120, BBB121, and BBB122 would be reported in sequence, followed by the results for Ca (Calcium) in BBB120 etc.

3.11.6 Under "ADDITIONS," enter the "ZERO," "FIRST," "SECOND," "THIRD," observed value in absorbance units (to three decimal places) of the analyte before addition is performed.

3.11.6.1 Under "ZERO, Found," enter the concentration in µg/L (to three decimal places) of the analyte (excluding sample contribution) after first addition to the sample analyzed by MSA.

3.11.6.2 Under "FIRST, Added, Found," enter the concentration in µg/L (to three decimal places) of the analyte added in the left column and the found value in µg/L (to three decimal places) of the analyte (excluding sample contribution) after the first addition to the sample analyzed by MSA in the right column.

3.11.6.3 Under "SECOND, Added, Found," enter the concentration in µg/L (to three decimal places) of the analyte added in the left column and the found value in µg/L (to three decimal places) of the analyte (excluding sample contribution) after second addition to the sample analyzed by MSA in the right column.

3.11.6.4 Under "THIRD, Added, Found," enter the concentration in µg/L (to three decimal places) of the analyte added in the left column and the found value in µg/L (to three decimal places) of the analyte (excluding sample contribution) after third addition to the sample analyzed by MSA in the right column.

NOTE: The "ZERO, Found," "FIRST, Found," "SECOND, Found," and "THIRD, Found" must have the same dilution factor.

3.11.7 Under "Final Conc.," enter the final analyte concentration (in µg/L to two decimal places) in the sample as determined by MSA computed according to the following formula:

$$Final\ Conc. = (-1) \times (x\text{-}intercept) \qquad \text{Eq. B-13}$$

NOTE: The final concentration of a parameter does not have to equal the value for that parameter which is reported on FORM I-AAIN for that sample.

3.11.8 Under "r," enter the correlation coefficient (to four decimal places) this obtained for the least squares regression line representing the following points (x,y):(0.0, "ZERO, Found"), ("FIRST, Added," "FIRST, Found"), ("SECOND, Added," "SECOND, Found"), and ("THIRD, Added," "THIRD, Found"). The correlation coefficient must be calculated using the ordinary least square linear regression (unweighted) according to the following formula:

$$r = \frac{N\Sigma x_i y_i - \Sigma x_i \Sigma y_i}{[N\Sigma x_i^2 - (\Sigma x_i)^2]^{\frac{1}{2}} [N\Sigma y_i^2 - (\Sigma y_i)^2]^{\frac{1}{2}}} \qquad \text{Eq. B-14}$$

3.11.16 Under "Q," enter "+" if r is less than 0.995. If r is greater than or equal to 0.995, then leave the field empty.

3.11.17 Under "Comments," enter any significant problems encountered during the method of standard addition analysis, both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.12 ICP SERIAL DILUTION [FORM X - AAIN]

3.12.1 This form is used to report results for ICP serial dilution.

3.12.2 Complete the header information according to the header instructions and as follows.

3.12.3 In the "EPA SAMPLE No." box, enter the EPA sample number (seven characters maximum) of the sample from which the serial dilution analysis results on this form were obtained. The number must be centered in the box.

3.12.4 Under "WOMN," enter the wavelength number for which the results of each analyte are reported on the form. The wavelength number is a number assigned to each wavelength used when more than one wavelength is used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. The field must be left blank if a single wavelength is used to obtain data for an analyte in the SDG.

3.12.5 Under "Initial Sample Result (I)," enter the concentration (in µg/L, to three decimal places) of each analyte in the original sample (reported in the EPA sample number box) on which a serial dilution analysis was performed. Enter any appropriate qualifier in the "C" qualifier column immediately following the "Initial Sample Result (I)" column.

NOTE: The Initial Sample Concentration for an analyte does <u>not</u> have to equal the value for that analyte reported on FORM I-AAIN for that sample. It is the value of the analyte concentration (uncorrected for dilution) that is within the linear range of the instrument.

3.12.6 Under "Serial Dilution Result (S)," enter the concentration (in µg/L, to three decimal places) of each analyte in the diluted sample (reported in the EPA sample number box) on which a serial dilution analysis was performed. Enter any appropriate qualifier in the "C" qualifier column immediately following the "Serial Dilution Result (S)" column.

NOTE: The Serial Dilution Result (S) is obtained by multiplying by five the instrument measured concentration value (in µg/L) of the serial diluted sample and that the "C" qualifier for the serial dilution must be established based on the serial dilution result before correcting it for the dilution regardless of the value reported on the form.

3.12.7 Under "%Difference," enter the absolute value (to one decimal place) of the percent difference in concentration of required analytes, between the original sample and the diluted sample (adjusted for dilution) according to the following equation:

$$\%\ Difference = \frac{|I - S|}{I} \times 100 \qquad \text{Eq. B-15}$$

NOTE: The values for I and S used to calculate % Difference in the equation must be exactly those reported on this form. A value of zero must be substituted for S if the analyte concentration is less that the MQL. If the analyte concentration in (I) is less than the MQL concentration, leave the "% Difference" field empty.

3.12.8 Under "Q," enter "E" if the percent difference is greater than 10 percent and the original sample concentration (reported on FORM I-AAIN) is greater than 50x the MQL reported on FORM XII-AAIN.

3.12.9 Under "M," enter the method used.

3.13 METHOD QUANTITATION LIMITS [FORM XI - AAIN]

3.13.1 This form documents the Method Quantitation Limit (MQL) for each instrument that the laboratory used to obtain data for the SDG. Only the instrument and wavelengths or masses used to generate data for the SDG must be included.

3.13.2 Complete the header information according to the header instructions and as follows.

3.13.3 For "Date," enter the date (formatted MM/DD/YY) on which the MQL values were determined (or became effective).

3.13.4 Enter the instrument ID numbers for the fields "ICP ID No.," "ICP-MS ID No.," and "GFAA ID No." (twelve characters maximum each). These ID Numbers are used to uniquely identify each instrument that the laboratory uses for CLP analyses.

3.13.5 Under "WOMN," enter the wavelength or mass number for which the results of each analyte are reported on the form. The wavelength or mass number is a number assigned to each wavelength used when two or more wavelengths or masses are used to obtain data for an analyte in the SDG. A wavelength number of "1" is assigned to the longest wavelength used for the analyte in the SDG. A wavelength number of "2" is assigned to the second longest wavelength and so on. A mass number of "1" is assigned to the greatest mass used for the analyte in the SDG. A mass number of "2" is assigned to the second greatest mass and so on. The field must be left blank if a single wavelength or mass is used to obtain data for an analyte in the SDG.

3.13.6 Under "WAVELENGTH (nm)," enter the wavelength in nanometers (to two decimal places) for each analyte for which an MDL has been established and is listed in the MQL column. If more than one wavelength is used for an analyte, use other copies of FORM XI-AAIN as appropriate to report the MDL.

3.13.7 Under "Mass," enter the mass to charge ratio (m/z, to four decimal places) for each analyte for which an MDL has been established and is listed in the MDL column. If more than one wavelength is used for an analyte, use other copies of FORM XI-AAIN as appropriate to report the MDL.

3.13.8 Under "INTEG. TIME," enter the integration time (in seconds, to two decimal places) used for each measurement taken from each instrument.

3.13.9 Under "BACKGROUND," enter the type of background correction used to obtain GFAA data. Enter "BS" for Smith Hieftje, "BD" for Deuterium Arc, or "BZ" for Zeeman background correction.

3.13.10 The Contract Required Quantitation Limits (in µg/L) in Exhibit C must appear in the column headed "CRQL".

3.13.11 Under "MQL," enter the MQL (in µg/L to three decimal places) as determined by the laboratory for each analyte analyzed by the instrument for which the ID Number is reported on this form. MQLs shall be reported to two significant figures if the MQL value is less than 100 and to three significant figures for values above or equal to 100.

3.13.12 Under "M," enter the method used to determine the MQL for each wavelength or mass used.

3.13.13 Use additional copies of FORM XI-AAIN if more instruments and wavelengths are used. Note that the data on this form must not exceed the analysis dates in the SDG data package or precede them by more than three months.

3.13.14 Under "Comments," enter alternative wavelengths or masses and the conditions under which they are used, any significant problems encountered during the MQL analysis, the corrective action taken, and resolution performed for the sample.

3.14 ICP AND ICP-MS INTERELEMENT CORRECTION FACTORS (Annual) [FORM XII - AAIN]

3.14.1 This form documents the ICP and ICP-MS Interelement Correction Factors for each ICP and/or ICP-MS instruments that the laboratory used to obtain data for the SDG. Only the instrument and wavelengths or masses used to generate data for the SDG must be included.

3.14.2 The correction factors are determined annually (every twelve months). A copy of the results of the annual interelement correction factors must be included with each SDG data package on FORM XII-AAIN.

3.14.3 Complete the header information according to the header instructions and as follows.

3.14.4 For "Date," enter the date (formatted as MM/DD/YY) on which these correction factors were determined for use. This date must not exceed the ICP and/or ICP-MS analyses dates in the SDG data package. Also, it must not precede them by more than twelve calendar months.

3.14.5 For "ICP ID No.," enter the instrument ID number (twelve characters maximum) used to produce the data for the SDG. If more than one ICP instrument is used, submit additional FORMs XII-AAIN as appropriate.

3.14.6 For "ICP-MS ID No.," enter the instrument ID number (twelve characters maximum) used to produce the data for the SDG. If more than

one ICP-MS instrument is used, submit additional FORMs XII-AAIN as appropriate.

3.14.7 Under "WOMN," list the wavelength in nanometers (to two decimal places) for ICP or the mass to charge ratio (m/z, to four decimal places) for ICP-MS used for each analyte. If more than one wavelength or mass is used, submit additional copies of FORM XII-AAIN as appropriate.

3.14.8 In the "INTERELEMENT CORRECTION FACTORS FOR:" column, enter the correction factor (negative, positive or zero, to seven decimal places, 10 characters maximum) for each corrected analyte analyzed by ICP and/or ICP-MS. If an analyte was not corrected for an analyte that is listed in the header of a column, a zero must be entered to indicate that the correction was determined to be zero.

3.14.9 Use additional copies of FORM XII-AAIN as appropriate if correction factors for more than six analytes were applied.

3.14.10 Columns of correction factors for analytes requiring interelement correction must be entered left to right starting on FORM XII-AAIN according to the alphabetic order of their chemical symbols starting on the first FORM XII-AAIN and proceeding to the following FORM XII-AAIN as appropriate.

3.14.11 Under "Comments," enter alternative wavelengths or masses and the conditions under which they are used, any significant problems encountered during the interelement correction analysis, both technical and administrative, the corrective action taken, and resolution performed for the sample.

3.15 ICP-MS TUNING AND RESPONSE FACTOR CRITERIA [FORM XIII - AAIN]

3.15.1 This form documents the tuning and response factor, and mass calibration results for each ICP-MS used to obtain data for the SDG.

3.15.2 Complete the header information according to the header instructions and as follows

3.15.3 For "Method," enter the method code (two characters maximum) according to 3.3.10.3. If more instruments or analyte masses are used, submit additional FORMS XIII-AAIN as appropriate.

3.15.4 For "Date," enter the date (formatted as MM/DD/YY) on which the initial tuning solution were determined for use. This date must not exceed the ICP-MS analysis dates in the SDG data package.

3.15.5 For "ICP-MS ID No.," enter the instrument ID number (twelve characters maximum) used to produce the data for the SDG. If more than one ICP-MS instrument is used, submit additional FORMs XIII-AAIN as appropriate.

3.15.6 For "Run No.," enter the run number (two characters maximum) which is the number that applies to a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract.

3.15.7 Under "Analysis Time," enter the initial and final time according to the following:

3.15.7.1 For "Initial Time," enter the time (in military format - HH:MM) at which each initial tuning solution analysis was performed.

3.15.7.2 For "Final Time," enter the time (in military format - HH:MM) at which each final tuning solution analysis was performed.

3.15.8 For "% Relative Abundance," enter the initial and final percent relative abundance according to the following:

3.15.8.1 Under "Initial," enter the percent relative abundance (to two decimal places) calculated from the intensities measured, for each of the isotopes listed, as a result of analyzing the 100 ppb tuning solution at the beginning of each ICP-MS run. The isotopes are listed in the first column from the left in the Tuning Section of the Form.

3.15.8.2 Under "Final," enter the percent relative abundance (to two decimal places) calculated from the intensities measured, for each of the isotopes listed, as a result of analyzing the 100 ppb tuning solution at the end of each ICP-MS run. The isotopes are listed in the first column from the left in the Tuning Section of the Form.

3.15.9 For "RF100 (1) Response," enter the initial and final percent relative abundance according to the following:

3.15.9.1 Under "Initial," enter the measured response factor (in counts per second, to the nearest whole number) in the 100 ppb tuning solution at the at the beginning of each ICP-MS run, for each mass to charge ratio listed in the first column from the left in the Response Factor Section of the Form.

3.15.9.2 Under "Final," enter the measured response factor (in counts per second, to the nearest whole number) in the 100 ppb tuning solution at the at the end of each ICP-MS run, for each mass to charge ratio listed in the first column from the left in the Response Factor Section of the Form.

3.15.10 Under "Observed Mass," enter the observed mass (to four decimal places) in the 100 ppb tuning solution at the beginning of each ICP-MS run, for each mass to charge ratio listed in the first column from the left in the Mass Calibration Section of the Form.

NOTE: The values measured and reported in the Tuning, Response Factor, and Mass Calibration Sections of the Form must be within the control limits listed in the second column from the left in each of the Sections.

NOTE: For every initial solution reported there must be a final one. However, the opposite is not true. If a tuning solution was required to be analyzed in the middle of a run (to avoid exceeding the 8-hour limit), it must be reported in the "Final" section of this form.

3.16 ICP-MS INTERNAL STANDARDS SUMMARY [FORM XIV -AAIN]

3.16.1 This form is used to report the internal standards intensity levels for ICP-MS. The relative intensity of each of the internal standards in all analyses performed in each ICP-MS must be reported on the FORM XIV-AAIN.

NOTE: A run is defined as the continuous totality of analyses performed by an instrument throughout the sequence initiated by, and including, the initial and the final tuning solution, the first required calibration standard and terminated by, and including, the CCV and blank following the last required analytical sample. For example, all field samples and all quality control analyses (including tuning solutions, calibration standards, ICVs, CCVs, ICBs, CCBs, MTS, CRIs, ICSs, IRSs, LCSs, PBs, duplicates, and matrix spikes) associated with the SDG must be reported on FORM XIV-AAIN. The run must be continuous and inclusive of all analyses performed on the particular instrument during the run.

3.16.3 Submit one FORM XIV-AAIN per run if no more than 32 analyses including instrument calibration were performed. If more than 32 analyses per run were performed, then submit additional copies of FORM XIV-AAIN as appropriate. Each new run must be started on the first line of FORM XIV-AAIN.

3.16.4 Complete the header information according to the header instructions and as follows.

3.16.5 For "Method," enter the method code (two characters maximum) according to 3.3.10.3. If more instruments or analyte masses are used, submit additional FORMS XIV-AAIN as appropriate.

3.16.6 For "Run No.," enter the run number (two characters maximum) which is the number that applies to a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract.

3.16.7 For "ICP-MS ID No.," enter the instrument ID number (twelve characters maximum) used to produce the data for the SDG. If more than one ICP-MS instrument is used, submit additional FORMs XIV-AAIN as appropriate.

3.16.8 For "Start Date," enter the start date (formatted as MM/DD/YY) on which the analyses was started. This date must not exceed the ICP-MS analysis dates in the SDG data package.

3.16.9 For "End Date," enter the end date (formatted as MM/DD/YY) on which the analyses run was ended. This date must not exceed the ICP-MS analysis dates in the SDG data package.

3.16.10 Under "EPA SAMPLE No.," enter the EPA sample number of each sample in the SDG and of all other preparations, such as duplicates, spikes, LCSs, PBs, and repreparations (all formatted according to Table B-2 of this Exhibit). All EPA Sample Numbers must be listed in increasing temporal (date and time) order of analysis, continuing to the next FORM XV-AAIN if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, must be reported. Those analyses must be identified with the EPA SAMPLE NUMBER of "ZZZZZZ". Samples identified as "ZZZZZZ" need not have intensities reported for internal standards.

3.16.11 Under "Time," enter the time (in military format - HH:MM) at which the analysis was performed.

NOTE: For any particular ICP-MS run, the EPA sample number and time sequence on FORM XIV-AAIN and XVI-AAIN must be identical.

3.16.12 Under "Internal Standards %D For:," enter the chemical symbol of the internal standard in the two spaces header field provide to indicate the internal standard for which the percent differences in that column were reported.

3.16.12.1 In the column, enter the percent difference (to the nearest whole number) between the intensity of the internal standard in the blank calibration standard (SO) and the intensity of the internal standard in the EPA sample number for each sample analyze listed on the form (excluding "ZZZZZZ"). The percent difference (%D) is calculated using the following equation:

$$\%D = \frac{|SO_I - S_I|}{SO_I} \times 100 \qquad \text{Eq. B-16}$$

where:

SO_I = The intensity of the internal standard in the blank calibration; and

S_I = The intensity of internal standard in the EPA sample number in the same units.

3.16.13 Under "Q", enter an "E" if the %D for a field sample, duplicate, or spike is greater than 50% for the second time after being run at a five fold dilution. If the percent relative intensity is less than or equal to 50%, then leave the field empty.

NOTE: A comment on the appropriate FORMs I-AAIN, VI-AAIN, or VII-AAIN explaining which analytes are affected by this flag must be included.

NOTE: Columns of internal standard %D must be entered left to right starting with the internal standards of the lower mass on the first FORM XIV-AAIN and proceeding to the following FORM XIV-AAIN as appropriate.

3.17 Preparation Log [FORM XV - AAIN]

3.17.1 This form is used to report the sample analysis log for ICP and ICP-MS analyses only. In addition, the samples reported on this form must have been prepared in the same manner using no pre-preparation dilution or concentration steps. The results reported on FORM I-AAIN for the samples listed on this form for each analyte must be obtained by multiplying each analyte's concentration (in µg/L) from the instrument by the dilution factor (DF) listed on the form.

3.17.2 All field samples and all quality control preparations (including duplicates, spikes, LCS's, PB's and reprepared samples) associated with the SDG must be reported on FORM XV-AAIN. Only the preparations associated with the SDG may be submitted on this form.

3.17.3 Submit one FORM XV-AAIN per method if no more than 32 preparations including quality control preparations were performed. In more than 32 preparations per method were performed, then submit additional copies of FORM XV-AAIN as appropriate.

3.17.4 Complete the header information according to the header instructions and as follows.

3.17.5 For "Analyte," enter the name of the analyte as identified in the Target Analyte List in Exhibit C.

3.17.6 For "Run No.," enter the run number (two characters maximum) which is the number that applies to a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract.

3.17.7 For "Instrument ID No.," enter the instrument ID number (12 characters maximum) which is the identifier that distinguishes each instrument used for analysis in the SDG. If more than one instrument is used, submit additional copies of FORM XV-AAIN as appropriate.

3.17.8 For "Start Date," enter the date (formatted MM/DD/YY) on which the analysis run was started.

3.17.9 For "End Date," enter the date (formatted MM/DD/YY) on which the analysis run was ended.

3.17.10 Under "EPA SAMPLE No.," enter the EPA sample number of each sample in the SDG and of all other preparations, such as duplicates, spikes, LCSs, PBs, and repreparations (all formatted according to Table B-2 of this Exhibit). All EPA sample numbers must be listed in ascending alphanumeric order, continuing to the next FORM XV-AAIN if applicable. If a sample was reprepared, list the same EPA sample number in the order of increasing preparation date.

3.17.11 Under "PREP. DATE," enter the date (formatted MM/DD/YY) on which each sample was prepared for analysis by the method indicated in the header section of the form.

3.17.12 Under "Final Volume," enter the final volume (in mL, to the nearest whole number) of the preparation for each sample prepared for analysis by the method indicated in the header section of the form. This field must have a value for each sample listed.

3.17.13 Under "Time," enter the time (in military format - HH:MM) at which each analysis was performed.

3.17.14 Under "DF," enter the dilution factor (to two decimal places) by which the final product of preparation needed to be diluted for each analysis performed.

NOTE: A DF of "1" must be entered if the preparation product was analyzed without adding any further volume of dilutant or any other solutions to the sample or an aliquot of that sample taken for preparation.

NOTE: For EPA supplied solutions such as ICVs, ICSs, and LCSs, a DF must be entered if the supplied solution was used at a dilution different from that specified by the instructions provided with the solution. The DF reported in such a case must be that which would make the reported true values on the appropriate form for the solution equal those that were supplied with the solution by the EPA. For instance, if the ICV has a true value of 2500.0 µg/L for aluminum at a 20 fold dilution, and if the solution is prepared at a 40 fold dilution, a DF of "2" must be entered on FORM XIV-AAIN and the uncorrected instrument reading is compared to a true value of 1250 µg/L. In this example, FORM III-AAIN will have a true value of 2500.0 regardless of the dilution used. The found value for the ICV must be corrected for the dilution listed on FORM XV-AAIN using the following formula:

$$\textit{Found value on FORM II-AAIN} = \textit{Instrument readout in µg/L} \times DF \qquad \text{Eq. B-17}$$

3.17.15 Under "%R," enter the percent recovery (to two decimal places) for each analytical spike analyzed. Leave the field blank if the analysis reported is not an analytical spike.

3.18 Analysis Run Log [FORM XVI - AAIN]

3.18.1 This form is used to report the sample analysis run log for each instrument used for analyses in the SDG. This includes any analysis run where conditions for reporting on FORM XV-AAIN were not met. FORM XV-AAIN is analyte and method specific.

3.18.2 A run is defined as the totality of analyses performed by an instrument throughout the sequence initiated by, and including, the first required calibration standard and terminated by, and including, the continuing calibration verification and blank analyses following the last required field sample.

3.18.3 All field samples and all quality control preparations (including tuning solutions, calibration standards, ICVs, CCVs, ICBs, CCBs, MTSs, CRIs, ICSs, LRSs, LCSs, PBs, duplicates, pre-digestion spikes, analytical spikes, and spike addition analyzed by the MSA) associated with the SDG must be reported on FORM XVI-AAIN. The run must be continuous and inclusive of all analyses performed on the particular instrument during the run.

3.18.4 Submit one FORM XVI-AAIN per run if no more than 32 analyses including instrument calibration were analyzed in the run. If more than 32 analyses were performed in the run, submit additional copies of FORM XVI-AAIN as appropriate.

3.18.5 Complete the header information according to the header instructions and as follows:

3.18.6 For "Run No.," enter the run number (two characters maximum) which is the number that applies to a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract.

3.18.7 For "Start Date," enter the date (formatted MM/DD/YY) on which the analysis run was started.

3.18.8 For "Method," enter the method code (two characters maximum).

3.18.9 For "End Date," enter the date (formatted MM/DD/YY) on which the analysis run was ended.

3.18.10 For "Instrument ID No.," enter the instrument identification number (twelve characters maximum) which is the identifier that distinguishes each instrument used for analysis in the SDG. If more than one instrument is used, submit additional copies of FORM XVI-AAIN as appropriate.

3.18.11 Under "EPA SAMPLE No.," enter the EPA sample number, including the QC suffix of each sample (formatted according to Table B-2 of this

Exhibit). All EPA sample numbers must be listed in increasing temporal (date and time) order of analysis, continuing to the next FORM XV-AAIN for the instrument run if applicable. The analysis date and time of other analyses not associated with the SDG but analyzed by the instrument in the reported analytical run must be reported. Those analyses must be identified with the EPA Sample No. of "ZZZZZ".

3.18.12 Under "PREP. DATE," enter the date (formatted MM/DD/YY) on which each sample was prepared for analysis by the method indicated in the header section of the form.

3.18.13 Under "VOLUME," enter the final volume (in mL, to the nearest whole number) of the preparation for each sample prepared for analysis by the method indicated in the header section of the form. This field must have a value for each field listed.

3.18.14 Under "TIME," enter the time (in military format - HH:MM) at which each analysis was performed. If an autosampler is used with equal analysis time and intervals between analyses, then only the start time of the run (the time of analysis of the first calibration standard) and end time of the run (the time of analysis of the final CCV or CCB, whichever is later) needs to be reported.

3.18.15 Under "DF," enter the dilution factor (to three decimal places) by which the final product of the preparation procedure (digestate or distillate) can be analyzed within the instrument standard range. The DF does not include the dilution inherent in the preparation as specified by the preparation procedures in Exhibit D.

NOTE: A "1" must be entered if the preparation product was analyzed without adding any further volume of dilutant or any other solutions to the "Volume" or an aliquot of the "Volume" listed on FORM XV-AAIN for that sample.

NOTE: For EPA supplied solutions such as ICVs, ICSs, and LCSs, a DF must be entered if the supplied solution was used at a dilution different from that specified by the instructions provided with the solution. The DF reported in such a case must be that which would make the reported true values on the appropriate form for the solution equal those that were supplied with the solution by the EPA. For instance, if the ICV has a true value of 2500.0 µg/L for aluminum at a 20 fold dilution, and if the solution is prepared at a 40 fold dilution, a DF of "2" must be entered on FORM XIV-AAIN and the uncorrected instrument reading is compared to a true value of 1250 µg/L. In this example, FORM II-AAIN will have a true value of 2500.0 regardless of the dilution used. The found value for the ICV must be corrected for the dilution listed on FORM XV-AAIN using the following formula:

$$\text{Found value on FORM II-AAIN} = \text{Instrument readout in mg/L} \times DF \qquad \text{Eq. B-18}$$

3.18.16 Under "ANALYTES," enter "X" in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data on any of the forms in the SDG. Leave the column empty for each analyte if the analysis was not used to report the particular analyte.

3.19 STANDARD SOLUTIONS SOURCES [FORM XVII - AAIN]

3.19.1 This form is used to report the source of each standard solution on an analyte-by-analyte basis for ICVs, CCVs, CRQLs, LRSs, ICSs, and LCSs standards used for QC analyses in the SDG.

3.19.2 For EPA supplied solutions, entering "EPA" is not sufficient. EPA solutions must be identified using the codes EPA supplied with the solutions identification.

3.19.3 For non-EPA supplied solutions, enter sufficient information in the available twelve spaces to unequivocally identify the manufacturer and the solution used.

3.19.4 Complete the header information according to the instructions and as follows:

3.19.5 Under "ICV Standard Source," enter the initial calibration source (twelve spaces maximum) for each analyte for which the ICV results were reported on FORM II-AAIN.

3.19.6 Under "CCV Standard Source," enter the continuing calibration source (twelve spaces maximum) for each analyte for which the CCV results were reported on FORM II-AAIN.

3.19.7 Under "CRQL Standard Source," enter the contract required quantitation limit source (twelve spaces maximum) for each analyte for which the CRI results were reported on FORM III-AAIN.

3.19.8 Under "LRS Standard Source," enter the linear range standard source (twelve spaces maximum) for each analyte for which the LRS results were reported on FORM III-AAIN.

3.19.9 Under "ICS Standard Source," enter the interference check sample standard source (twelve spaces maximum) for each analyte for which the ICS results were reported on FORM V-AAIN.

3.19.10 Under "LCS Standard Source," enter the laboratory control sample standard source (twelve spaces maximum) for each analyte for which ICS results were reported on FORM VIII-AAIN.

3.20 SAMPLE LOG-IN SHEET [FORM AADC - IN - 1]

3.20.1 This form is used to document the receipt and inspection of shipping containers and samples. One original FORM AADC-IN-1 is required for each shipping container.

3.20.2 If the samples in a single shipping container must be assigned to more than one SDG, then the original FORM AADC-IN-1 shall be placed with the deliverables for the SDG of the lowest alphanumeric number and a copy of FORM AADC-IN-1 must be placed with the deliverables for the other SDG(s). The copies should be identified as "copy(ies)," and the location of the original should be noted on the copies.

3.20.3 Sign and date the airbill (if present). Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in item 1 on FORM AADC-IN-1. Record the custody seal numbers in item 2.

3.20.4 Open the shipping container, remove the enclosed sample documentation, and record on FORM AADC-IN-1, items 3-5, the presence/absence of chain-of-custody record(s), SMO forms (i.e., TRs, Packing Lists), and airbills or airbill stickers. Specify if there is an airbill or an airbill sticker present in item 5 on FORM AADC-IN-1. Record the airbill or sticker number in item 6.

3.20.5 Remove the samples from the shipping container(s), examine the samples and the sample tag (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking), and the presence or absence of sample tags in items 7 and 8 on FORM AADC-IN-1.

3.20.6 Review the sample shipping documents and complete the header information. Compare the information recorded on all the documents and samples, and circle the appropriate answer in item 9 on FORM AADC-IN-1.

3.20.7 If there are no problems observed during receipt, sign and date (include time) FORM AADC-IN-1, the chain-of-custody record, the TR, and write the sample numbers on FORM AADC-IN-1. For each sample number entered, enter the corresponding air volume sampled in standard cubic meters (std. m^3) in the appropriate column. Record the appropriate sample tags and assigned laboratory numbers if applicable. The log-in date should be recorded at the top of FORM AADC-IN-1, and the date and time of sample receipt at the laboratory should be recorded in items 10 and 11. Cross out unused columns and spaces.

3.20.8 If there are problems observed during receipt (e.g., data on air volume sampled is missing for one or more samples) or if an answer marked with an asterisk (i.e., "absent*") was circled, then contact SMO and document the resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the

preceding paragraph and note, where appropriate, the resolution of the problem.

3.20.9 For "Sample Transfer," enter the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of FORM AADC-IN-1. Sign and date the sample transfer block.

3.21 DOCUMENT INVENTORY SHEET [FORM AADC - IN - 2]

3.21.1 This form is used to record the inventory of the Complete Sample Delivery Group Case file (CSF) documents which are sent to the Region.

3.21.2 Organize all EPA-CSF documents as described in Exhibit B. Assemble the documents in the order specified on FORM AADC-IN-2 and Exhibit B, and stamp each page with the consecutive numbers (Do not number the DC-IN-2 form). Inventory the CSF by reviewing the document numbers and recording page number ranges in the column provided on FORM AADC-IN-2. If there are no documents for a specific document type, enter "NA" in the empty space.

3.21.3 Certain laboratory specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review DC-IN-2 to determine if it is most appropriate to place them under items 25, 26, 27, or 28. Item 28 should be used if there is no appropriate item. These types of documents should be described or listed in the blanks under each appropriate item.

SECTION 4

DATA REPORTING FORMS

PAGE NO.

1. Cover Page - [COVER PAGE - AAIN] B-50
2. Analysis Data Sheet [FORM I - AAIN] B-51
3. Initial and Continuing Calibration Verification [FORM II - AAIN] . . B-52
4. CRQL Standards/Linear Range Standards (Quarterly) [FORM III - AAIN] B-53
5. Blanks [FORM IV - AAIN] B-54
6. ICP Interference Check Sample [FORM V - AAIN] B-55
7. Spike Sample Recovery [FORM VI - AAIN] B-56
8. Duplicates [FORM VII - AAIN] B-57
9. Laboratory Control Sample [FORM VIII - AAIN] B-58
10. Method of Standard Additions [FORM IX - AAIN] B-59
11. ICP Serial Dilution [FORM X - AAIN] B-60
12. Method Quantitation Limits (Quarterly) [FORM XI - AAIN] B-61
13. ICP and ICP-MS Interelement Correction Factors (Annual) [FORM XII- AAIN] B-62
14. ICP-MS Tuning and Response Factor Criteria [FORM XIII - AAIN] . . . B-63
15. ICP-MS Internal Standards Summary [FORM XIV - AAIN] B-64
16. Preparation Log [FORM XV - AAIN] B-65
17. Analysis Run Log [FORM XVI - AAIN] B-66
18. Standard Solutions Sources [FORM XVII - AAIN] B-67
19. Sample Log-In Sheet [FORM AADC-IN-1] B-68
20. Document Inventory Sheet [FORM AADC-IN-2] B-69

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

COVER PAGE

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

EPA Sample No.	Lab Sample ID

Enter a "Y" for Yes or an "N" for No

ICP and ICP-MS interelement corrections applied? _____

ICP and ICP-MS background corrections applied? _____

If yes, were raw data generated before application of background corrections? _____

Comments: _____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, excluding the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM
Metals in Ambient Air

EPA SAMPLE NO.

ANALYSIS DATA SHEET

Lab Name: _____ Contract: _____

Lab Code: _____ Lab Sample ID: _____

Case No.: _____ Date Received: _____

SAS No.: _____ Air Volume Sampled, Std. m^3: _____

SDG No.: _____ Date Analyzed: _____

CAS No.	ANALYTE	CONCENTRATION		C	Q	M
		µg/L	µg/m^3			
7429-90-5	Aluminum					
7440-36-0	Antimony					
7440-38-2	Arsenic					
7440-39-3	Barium					
7440-41-7	Beryllium					
7440-43-9	Cadmium					
7440-70-2	Calcium					
7440-47-3	Chromium					
7440-48-4	Cobalt					
7440-50-8	Copper					
7439-89-6	Iron					
7439-92-1	Lead					
7439-95-4	Magnesium					
7439-96-5	Manganese					
7439-97-6	Molybdenum					
7440-02-0	Nickel					
7440-09-7	Potassium					
7782-49-2	Selenium					
7440-22-4	Silver					
7440-23-5	Sodium					
7440-28-0	Thallium					
7440-31-5	Tin					
7440-62-2	Vanadium					
7440-66-6	Zinc					

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____

SAS No.: _____ Initial Calibration Source: _____

SDG No.: _____ Continuing Calibration Source: _____

Concentration Units: µg/L

ANALYTE	WOMN	INITIAL CALIBRATION (ICV)			CONTINUING CALIBRATION (CCV) CCV No.: ____			CCV No.: ____			M
		True	Found	%R	True	Found	%R	True	Found	%R	
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Molybdenum											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Tin											
Vanadium											
Zinc											

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

CRQL STANDARDS/LINEAR RANGE STANDARDS

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

CRQL: LRS:

ICP Source: _____ ICP Source: _____

ICP-MS Source: _____ ICP-MS Source: _____

GFAA Source: _____ GFAA Source: _____

Concentration Units: µg/L

ANALYTE	WOMN	CRQL STANDARDS						LINEAR RANGE STANDARDS (LRS)					
		INITIAL			FINAL			INITIAL			FINAL		
		True	Found	%R	Found	%R	M	True	Found	%R	Found	%R	M
Aluminum													
Antimony													
Arsenic													
Barium													
Beryllium													
Cadmium													
Calcium													
Chromium													
Cobalt													
Copper													
Iron													
Lead													
Magnesium													
Manganese													
Molybdenum													
Nickel													
Potassium													
Selenium													
Silver													
Sodium													
Thallium													
Tin													
Vanadium													
Zinc													

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Concentration Units: µg/L

ANALYTE	WOMN	INITIAL CALIB. BLANK (ICB)		CONTINUING CALIBRATION BLANK (CCB)						PREPARATION BLANK (PB)		M
		Conc.	C	Conc. 1	C	Conc. 2	C	Conc. 3	C	Conc.	C	
Aluminum												
Antimony												
Arsenic												
Barium												
Beryllium												
Cadmium												
Calcium												
Chromium												
Cobalt												
Copper												
Iron												
Lead												
Magnesium												
Manganese												
Molybdenum												
Nickel												
Potassium												
Selenium												
Silver												
Sodium												
Thallium												
Tin												
Vanadium												
Zinc												

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

ICP INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

ICP ID No.: _____ ICS Source: _____

Concentration Units: µg/L ICP-MS ID No.: _____

ANALYTE	WOMN	TRUE		INITIAL FOUND			FINAL FOUND			M
		Sol. A	Sol. AB	Sol. A	Sol. AB	%R	Sol. A	Sol. AB	%R	
Aluminum										
Antimony										
Arsenic										
Barium										
Beryllium										
Cadmium										
Calcium										
Chromium										
Cobalt										
Copper										
Iron										
Lead										
Magnesium										
Manganese										
Molybdenum										
Nickel										
Potassium										
Selenium										
Silver										
Sodium										
Thallium										
Tin										
Vanadium										
Zinc										

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Concentration Units: µg/L

ANALYTE	WOMN	CONTROL LIMIT %R	SPIKED SAMPLE RESULT (SSR)		SAMPLE RESULT (SR)		SPIKE ADDED (SA)			
			Conc.	C	Conc.	C	Conc.	%R	Q	M
Aluminum										
Antimony										
Arsenic										
Barium										
Beryllium										
Cadmium										
Calcium										
Chromium										
Cobalt										
Copper										
Iron										
Lead										
Magnesium										
Manganese										
Molybdenum										
Nickel										
Potassium										
Selenium										
Silver										
Sodium										
Thallium										
Tin										
Vanadium										
Zinc										

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM
Metals in Ambient Air

DUPLICATES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Concentration Units: µg/L

ANALYTE	WOMN	CONTROL LIMIT	SAMPLE (S)		DUPLICATE (D)		RPD	Q	M
			Conc.	C	Conc.	C			
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Molybdenum									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Tin									
Vanadium									
Zinc									

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Metals in Ambient Air

LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

GFAA Source: _____ ICP Source: _____

ICP-MS Source: _____ Concentration Units: µg/L

ANALYTE	LCS USED	WOMN	LIMITS		TRUE	FOUND	C	%R	M
			LOWER	UPPER					
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Molybdenum									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Tin									
Vanadium									
Zinc									

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

STANDARD ADDITION RESULTS

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Concentration Units: µg/L

				A D D I T I O N S									
			ZERO	FIRST		SECOND		THIRD		FINAL			
			Found	Added	Found	Added	Found	Added	Found	CONC.	r	Q
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
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22													
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24													
25													
26													
27													
28													
29													
30													
31													
32													

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

ICP and ICP–MS SERIAL DILUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Concentration Units: µg/L

Analyte	WOMN	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	%Difference	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Molybdenum								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Tin								
Vanadium								
Zinc								

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

METHOD QUANTITATION LIMIT

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Date: _____ ICP ID No.: _____

GFAA ID No.: _____ Concentration Units: µg/L

ANALYTE	WOMN	WAVE-LENGTH (nm)	MASS (m/z)	INTEG. TIME (sec)	BACK-GROUND	CRQL (µg/L)	MQL (µg/L)	M
Aluminum						226		
Antimony						92		
Arsenic						92		
Barium						11		
Beryllium						7		
Cadmium						18		
Calcium						381		
Chromium						44		
Cobalt						55		
Copper						37		
Iron						126		
Lead						118		
Magnesium						89		
Manganese						15		
Molybdenum						33		
Nickel						52		
Potassium						758		
Selenium						577		
Silver						229		
Sodium						592		
Thallium						562		
Tin						155		
Vanadium						26		
Zinc						444		

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

ICP AND ICP-MS INTERELEMENT CORRECTION FACTORS (ANNUAL)

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

ICP ID No.: _____ Date: _____

ICP-MS ID No.: _____

ANALYTE	W O M N	INTERELEMENT CORRECTION FACTORS FOR:				
		___	___	___	___	___
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Tin						
Vanadium						
Zinc						

Comments: _____

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

ICP–MS TUNING AND RESPONSE FACTOR CRITERIA

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Date: _____ ICP-MS ID No.: _____

Run No.: _____ Method: _____

Analysis Times:

Initial: _____

Final: _____

TUNING

m/z	Ion Abundance Criteria	% Relative Abundance	
		Initial	Final
$^{7}Li/^{59}Co$	(0.20 - 1.00)		
$^{56}Co/^{59}Co$	(1.00)		
$^{115}In/^{59}Co$	(0.75 - 2.00)		
$^{205}Tl/^{59}Co$	(0.50 - 1.20)		

RESPONSE FACTOR (counts per second)

m/z	Response Factor Criteria	RF100 (1) Response	
		Initial	Final
^{7}Li	(> 2,000)		
^{59}Co	(> 20,000)		
^{115}In	(> 10,000)		
^{102}Ru	(> 25)		
^{205}Tl	(> 1,000)		

(1) Background (RF0) for ^{102}Ru.

MASS CALIBRATION

m/z	Acceptable Mass Range	Observed Mass
^{7}Li	(6.9160 - 7.1160)	
^{59}Co	(58.8332 - 59.0332)	
^{115}In	(114.8040 - 115.0040)	
^{205}Tl	(204.8744 - 205.0744)	

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

ICP-MS INTERNAL STANDARDS SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Run No.: _____ Method: _____

Start Date: _____ ICP-MS ID No.: _____

End Date: _____

	EPA SAMPLE NUMBER	Time	Internal Standards %D For:									
			___	Q	___	Q	___	Q	___	Q	___	Q
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
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32												

U. S. ENVIRONMENTAL PROTECTION AGENCY

CONTRACT LABORATORY PROGRAM

Metals in Ambient Air

PREPARATION LOG

Lab Name: _____ Contract: _____

Lab Code: _____ SAS No.: _____

Case No.: _____ SDG No.: _____

Run No.: _____ Analyte: _____

Start Date: _____ Instrument ID No.: _____

End Date: _____

	EPA SAMPLE No.	PREP. DATE	Final Volume (mL)	Time	DF	%R
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						

U.S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Metals in Ambient Air
ANALYSIS RUN LOG

Lab Name: ______________________ Contract: ______________________

Lab Code: ______________________ SDG No.: ______________________

Case No.: ______________________ SAS No.: ______________________

Start Date: ______________________ Run No.: ______________________

End Date: ______________________ Method: ______________________

Instrument ID No.: ______________________

	EPA SAMPLE No.	PREP. DATE	VOLUME (mL)	TIME	DF	ANALYTES																							
						AL	SB	AS	BA	BE	CD	CA	CR	CO	CU	FE	PB	MG	MN	MO	NI	K	SE	AG	NA	TL	SN	V	ZN
1																													
2																													
3																													
4																													
5																													
6																													
7																													
8																													
9																													
10																													
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U.S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM
Metals in Ambient Air

STANDARD SOLUTIONS SOURCES

Lab Name: _____ Contract: _____

Lab Code: _____ SDG No.: _____

Case No.: _____ SAS No.: _____

Analyte	ICV Standard Source	CCV Standard Source	CRQL Standard Source	LRS Standard Source	ICS Standard Source	LCS Standard Source
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Potassium						
Selenium						
Silver						
Thallium						
Tin						
Vanadium						
Zinc						

Comments: _____

SAMPLE LOG-IN SHEET

Lab Name: ______________________ Page: ________ of ________

Received By (Print Name): ______________________ Log-in Date: ______________

Received by (Signature): ______________________

Case Number: ________ Sample Delivery Group No.: ________ SAS Number: ________	EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	AIR VOLUME SAMPLED STD. m^3	REMARKS: CONDITION OF SAMPLE SHIPMENT ETC.
CIRCLE THE APPROPRIATE RESPONSE:					
1. Custody Seal(s) — Present/Absent*					
Intact/Broken					
2. Custody Seal Nos.: ________					
________					
3. Chain-of-Custody Records — Present/Absent*					
4. Traffic Reports or Packing List — Present/Absent*					
5. Airbill — Airbill/Sticker Present/Absent*					
6. Airbill No.: ________					
7. Sample Tags — Present/Absent*					
Sample Tag Numbers — Listed/Not Listed on Chain-of-Custody					
8. Sample Condition: — Intact/Broken*/ Leaking					
9. Does information on custody records traffic reports, an sample tags agree. — Yes/No*					
10. Date Received at Lab: ________					
11. Time Received: ________					
Sample Transfer					
Fraction: ________ ________					
Area #: ________ ________					
By: ________ ________					
On: ________ ________					

*If Circled, contact SMO and attach record of resolution

Reviewed by: ______________________ Logbook No.: ______________________

Date: ______________________ Logbook Page No.: ______________________

DRAFT

METAL IN AMBIENT AIR ANALYTES
COMPLETE SDG FILE (CSF)
INVENTORY SHEET

Lab Name: _____ City/State: _____

Case No.: _____ SDG No.: _____ SDG Nos. to Follow: ____ - ____ - ____ - ____

SAS No.: _____ Contract No.: _____

All documents delivered in the complete SDG file must be original documents where possible. (Reference Exhibit B, Section 3)

	Page Nos.		(Please Check:)	
	From	To	Lab	Region
1. Inventory Sheet (AADC-2) (Do not number)				
2. Cover Page				
3. Inorganic Analysis Data Sheet (FORM I-AAIN)				
4. Initial & Continuing Calibration Verification (FORM II-AAIN)				
5. CRQL Standards/Linear Range Standards (Quarterly) (FORM III-AAIN)				
6. Blanks (FORM IV-AAIN)				
7. ICP and ICP-MS Interference Check Sample (FORM V-AAIN)				
8. Spike Sample Recovery (FORM VI-AAIN)				
9. Duplicates (FORM VII-AAIN)				
10. Laboratory Control Sample (FORM VIII-AAIN)				
11. Method of Standard Addition Results (FORM IX-AAIN)				
12. ICP and ICP-MS Serial Dilution (FORM X-AAIN)				
13. Method Detection Limit (FORM XI-AAIN)				
14. ICP and ICP-MS Interelement Correction Factors (Annual) (FORM XII-AAIN)				
15. ICP-MS Tuning and Response Factor Criteria (FORM XIII-AAIN)				
16. ICP-MS Internal Standards Summary (FORM XIV-AAIN)				
17. Preparation Log (FORM XV-AAIN)				
18. Analysis Run Log (B) (FORM XVI-AAIN)				
19. ICP Raw Data				
20. ICP-MS Raw Data				
21. GFAA Raw Data				
22. Traffic Report				

METAL IN AMBIENT AIR ANALYTES
COMPLETE SDG FILE (CSF)
INVENTORY SHEET

	From	To	Lab	Region
23. EPA Shipping/Receiving Documents				
Airbill (No. of Shipments ____)				
Chain-of-custody Records				
Sample Tags				
Sample Log-In Sheet (Lab & HDC-1)				
SDG Cover Sheet				
24. Misc. Shipping/Receiving Records				
(list all individual records)				
Telephone Logs				
_____				
_____				
25. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
26. Internal Originial Sample Preparation & Analysis Records (describe or list)				
Preparation Records _____				
Analysis Records _____				
Description _____				
27. Other Records (describe or list)				
Telephone Communication Log				
_____				
_____				

28. Comments: _____

_____

_____

_____

Completed by (CLP Lab):

_____ _____ _____

(Signature) (Print Name & Title) (Date)

Audited by (EPA):

_____ _____ _____

(Signature) (Print Name & Title) (Date)

DRAFT

EXHIBIT C

TARGET COMPOUND LISTS

TABLE 1

METALS IN AMBIENT AIR
TARGET ANALYTE LIST (TAL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Analyte	CAS RN	Contract Required Quantitation Limit[1,2,3] ng/m³	µg/L
Aluminum	7429-90-5	33	226
Antimony	7440-36-0	13	92
Arsenic	7440-36-2	13	92
Barium	7440-39-3	2	11
Beryllium	7440-41-7	1	7
Cadmium	7440-43-9	3	18
Calcium	7440-70-2	55	381
Chromium	7440-47-3	6	44
Cobalt	7440-48-4	8	55
Copper	7440-50-8	5	37
Iron	7439-89-6	18	126
Lead	7439-92-1	17	118
Magnesium	7439-95-4	13	89
Manganese	7439-96-5	2	15
Molybdenum	7439-98-7	5	33
Nickel	1313-99-1	8	52
Potassium	7440-09-7	109	758
Selenium	7782-49-2	83	577
Silver	7440-22-4	33	229
Sodium	7440-23-5	85	592
Thallium	7440-28-0	81	562
Tin	7440-31-5	22	155
Vanadium	7440-62-2	4	26
Zinc	7440-66-6	64	444

(1) The analytical methods specified in Exhibit D, Sections 3 and 5 must be utilized and the achieved instrument detection limits must meet the Contract Required Quantitation Limits (CRQL) requirements. Higher detection levels may only be used in the following circumstance:

- If the sample concentration exceeds two times the detection limit of the instrument or method in use, the value may be reported even though the instrument or instrument detection limit may not equal the contract required quantitation limit. The method detection limit must be documented as described in Exhibits D.

(2) These CRQL's are the method detection limits obtained from actual method blank preparations that must be met using the procedure in Exhibits D.

(3) Assume 2500 m³ air volume per Hi-Vol filter and 40 mL final extract volume. See Exhibit D, Sections 3.9.4 and 5.11.2 for calculations.

TABLE 2

METALS IN AMBIENT AIR
TARGET ANALYTE LIST (TAL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
FOR ICP-MS ANALYSES

Analyte	CAS RN	Contract Required Quantitation Limit[1,2] ng/m³	µg/L
Aluminum	7429-90-5	33	226
Antimony	7440-36-0	13	92
Arsenic	7440-36-2	13	92
Barium	7440-39-3	2	11
Beryllium	7440-41-7	1	7
Cadmium	7440-43-9	3	18
Chromium	7440-47-3	6	44
Cobalt	7440-48-4	8	55
Copper	7440-50-8	5	37
Iron	7439-89-6	18	126
Lead	7439-92-1	17	118
Manganese	7439-96-5	2	15
Nickel	1313-99-1	8	52
Selenium	7782-49-2	83	577
Silver	7440-22-4	33	229
Thallium	7440-28-0	81	562
Vanadium	7440-62-2	4	26
Zinc	7440-66-6	64	444

(1) The ICP-MS method specified in Exhibit D, Section 4 may be utilized provided the documented method detection limits meet the Contract Required Quantitation Limit (CRQL) requirements.

(2) The CRQL is the method detection limits obtained in pure water that must be met using the procedure in Exhibit D. The method detection limits for samples may be considerably higher depending on the sample matrix.

(3) Assume 2500 m³ air volume per Hi-Vol filter and 40 mL final extract volume. See Exhibit D, Section 4.9.2 for calculations.

TABLE 3

INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS FOR ICP AND ICP-MS

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	500.0	500.0
Ca	500.0	500.0
Fe	500.0	500.0
Mg	500.0	500.0
Na	500.0	500.0
P	500.0	500.0
K	500.0	500.0
S	500.0	500.0
C	1,000.0	1,000.0
Cl	3,600.0	3,600.0
Mo	10.0	20.0
Ti	10.0	20.0
As	0.0	0.100
Cd	0.0	0.050
Cr	0.0	0.100
Co	0.0	0.200
Cu	0.0	0.100
Mn	0.0	0.100
Ni	0.0	0.200
Se	0.0	0.100
Ag	0.0	0.100
V	0.0	0.200
Zn	0.0	0.100

TABLE 4

INITIAL AND CONTINUING CALIBRATION VERIFICATION
CRQL STANDARD CONTROL LIMITS, AND LCS STANDARD CONTROL LIMITS
FOR INORGANIC ANALYSES

INITIAL AND CONTINUING CALIBRATION VERIFICATION LIMITS

Analytical Method	Inorganic Species	% of True Value (EPA Set) Low Limit	High Limit
ICP	Metals	90	110
ICP-MS	Metals	90	110
GFAA	Metals	90	110

CRQL STANDARD CONTROL LIMITS

Analytical Method	Inorganic Species	% of True Value (EPA Set) Low Limit	High Limit
ICP	Metals	85	115
ICP-MS	Metals	85	115
GFAA	Metals	85	115

LCS STANDARD CONTROL LIMITS

The LCS Standard Control Limits are the same for all inorganics species. The limits are 80-120 percent.

EXHIBIT D

ANALYTICAL METHODS FOR THE DETERMINATION OF METAL COMPOUNDS COLLECTED ON HI-VOL FILTERS AND ANALYZED BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY, INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICP-MS) OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) SPECTROMETRY

EXHIBIT D

ANALYTICAL METHODS FOR THE DETERMINATION OF METAL COMPOUNDS COLLECTED ON HI-VOL FILTERS AND ANALYZED BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY, INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICP-MS) OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) SPECTROMETRY

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SECTION 1

ANALYTICAL METHODS FOR THE DETERMINATION OF METAL COMPOUNDS COLLECTED ON HI-VOL FILTERS AND ANALYZED BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY, INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICP-MS) OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) SPECTROMETRY

1. INTRODUCTION

1.1 SCOPE AND APPLICATION

1.1.1 The methods specified in Exhibit D must be used and the documented instrument or method detection limits must meet the Contract Required Quantitation Limits (CRQL) (Tables 1 and 2, in Exhibit C). Analytical methods with higher detection limits may be used only if the sample concentration exceeds five times the documented detection limit of the instrument or method.

1.1.2 All samples must initially be run undiluted (i.e., final product of the sample preparation procedure). When an analyte concentration exceeds the calibrated or linear range (as appropriate), reanalysis for that analyte(s) is required after appropriate dilution. The Contractor must use the least dilution necessary to bring the analyte(s) within the valid analytical range (but not below the CRQL) and report the highest valid value for each analyte as measured from the undiluted and diluted analyses. Unless the Contractor can submit proof that dilution was required to obtain valid results, both diluted and undiluted sample measurements must be contained in the raw data. All sample dilutions shall be made with deionized water appropriately acidified to maintain constant acid strength.

1.1.3 The Contractor is reminded and cautioned that Exhibit D is a compendium of required and permitted analytical methods to be used in the performance of analyses under this contract. The quality assurance/quality control (QA/QC) procedures or measurements to be performed in association with these methods or analyses are specified in Exhibit E.

1.1.4 The Contractor is reminded and cautioned that the collection and provision of raw data may or may not be referred to within the individual methods of Exhibit D or the Quality Assurance Protocol of Exhibit E. The Raw Data Deliverables requirements are specified in Exhibit B. Raw data collected and provided in association with the performance of analyses under this contract shall conform to the appropriate provisions of Exhibit B.

1.1.5 Laboratory glassware to be used in metals analysis must be acid cleaned according to EPA's manual "Methods for Chemical Analysis of Water

and Wastes" or an equivalent procedure (see part 2.1.6.3). Samples must be opened and digested in a hood. Stock solutions to be used for preparing instrument or method calibration standards may be purchased or prepared as described in parts 3.6 and 4.6. All other solutions to be used for QA/QC measurements shall conform to the specific requirements of Exhibit E.

1.1.6 Background corrections are required for all GFAA measurements. Each GFAA analysis requires a minimum of two injections (burns) except during full Method of Standard Additions (MSAs). All ICP and ICP-MS measurements shall require a minimum of two replicate exposures. Appropriate hardcopy raw data for each exposure/injection shall be included in the data package in accordance with Exhibit B. The average of each set of exposures/injections shall be used for standardization, sample analysis, and reporting as specified in Exhibit D. All exposures must be reported in the raw data in concentration units.

SECTION 2

SAMPLE PREPARATION

2.1 MICROWAVE EXTRACTION AND RELATED HANDLING PROCEDURES

2.1.1 Introduction

2.1.1.1 This Section describes a microwave extraction procedure to extract the metals from the particulate loaded glass-fiber filter. Following microwave extraction, target analytes are analyzed by ICP, ICP-MS, or GFAA.

2.1.2 Sample Preservation

2.1.2.1 Ambient air glass-fiber filters should be received folded in half lengthwise with the particulate material inward and enclosed in protective envelopes. These protective envelopes shall be stored at 15 to 30°C until analysis.

2.1.2.2 The maximum sample holding time under this contract is 180 days. To be compliant with this contract, the Contractor must analyze samples within 180 days even if these times are less than the maximum data submission times allowed in this contract.

2.1.3 Summary of Method

2.1.3.1 A 1" x 8" strip is cut from the 8" x 10" filter as described in the Federal Reference Method for Lead (see Figure D-1 and Reference 11). The metals are extracted from the filter strip by a hydrochloric acid/nitric acid solution using a laboratory microwave digestion system. After cooling, the digestate is centrifuged to remove any insoluble material.

2.1.3.2 Microwave extraction is used to prepare samples for ICP analysis. This method (using nitric acid only) or an alternate hot extraction technique may be used to prepare samples for ICP-MS and GFAA analysis.

2.1.4 Apparatus and Materials

2.1.4.1 Microwave digestive system and capping station: With programmable power settings up to 600 watts (see Figure D-2).

NOTE: Commercial kitchen or home-use microwave shall NOT be used for the digestion of samples under this contract. The oven cavity must be corrosion resistant and well ventilated. All electronics must be protected against corrosion for safe operation.

2.1.4.2 PFA Teflon digestion vessels: Capable of withstanding pressures of up to 100 psi. Pressure venting vessels capable of controlled pressure relief at pressures exceeding 100 psi. (60- to 120-mL capacity).

2.1.4.3 Teflon PFA overflow vessel: Double ported (60- to 120-mL capacity).

2.1.4.4 Rotating table: For uniform exposure of samples within the oven.

2.1.4.5 Volumetric glassware: 50- to 100-mL capacity (Class A borosilicate).

2.1.4.6 Bottles: Linear polyethylene or polypropylene with leakproof caps, for storing samples; Teflon bottles for storing multielement standards (500-mL, 125-mL, and 30-mL).

2.1.4.7 Centrifuge tubes: Polypropylene tube with screw tops of polypropylene, 50-mL (Nalgene 3119-0050 or equivalent).

2.1.4.8 Pipette: Automatic dispensing with an accuracy of setting of 0.1 mL or better and repeatability of 20 µL (Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent).

2.1.4.9 Rack: Wire (Fisher 14-793-1) or polypropylene (Fisher 14-8090), for holding centrifuge tubes during shaking.

2.1.4.10 Particle mask (3M, No. 8500): To be worn while cutting and handling glass-fiber filters.

2.1.4.11 Centrifuge: Capable of maintaining speeds of 2000 rpm (International Equipment company Model UV or equivalent).

2.1.4.12 Template: To aid in sectioning the glass-fiber filter. Federal Register 1978 43 (Oct. 5), 46258-46261. (See Figure D-1).

2.1.4.13 Pizza cutter: Thin wheel (< 1 mm).

2.1.4.14 Mechanical shaker: Eberbach Corporation Model 6460 or equivalent.

2.1.5 Reagents

2.1.5.1 Hydrochloric acid: American Chemical Society (ACS) reagent grade, concentrated (12.3M) (or equivalent), for preparing samples.

2.1.5.2 Nitric acid: Redistilled spectrographic grade (16M) (or eqivalent), for preparing samples.

2.1.5.3 ASTM Type II water (ASTM D1193).

2.1.6 Microwave Extraction Procedure

2.1.6.1 Microwave Calibration Procedure

2.1.6.1.1 Calibration of the microwave unit is a critical step prior to its use. In order that absolute power settings may be interchanged from one microwave unit to another, the actual delivered power must be determined.

2.1.6.1.2 Calibration of a laboratory microwave unit (see Figure D-2) depends on the type of electronic system used by the manufacturer. If the unit has a precise and accurate linear relationship between the output power and the scale used in controlling the microwave unit, then the calibration can be a single-point calibration at maximum power. If the unit is not accurate or precise for some portion of the controlling scale, then a multiple-point calibration is necessary. If the unit power calibration needs multiple-point calibration, then the point where the linearity begins must be identified. For example, a calibration at 100, 99, 98, 97, 95, 90, 80, 70, 60, and 50 percent power settings can be applied and the data plotted. The nonlinear portion of the calibration curve can be excluded or restricted in use. Each percent is equivalent to approximately 5.5 - 6.5 W and becomes the smallest unit of power that can be controlled. If 20 - 40 W are contained from 99 - 100 percent, that portion of the microwave calibration is not controllable by three to seven times that of the linear portion of the control scale and will prevent duplication of precise power conditions specified in that portion of the power scale.

2.1.6.1.3 The equations in the following paragraph evaluate the power available for heating in a microwave cavity. This is accomplished by measuring the temperature rise in 1 kg of water exposed to electromagnetic radiation for a fixed period of time. Measurements are made on weighed replicates (five replicates) of 1 kg samples of room temperature distilled water in thick-walled microwave transparent (Teflon) vessels. The containers must be circulated continuously through the field for at least two minutes at full power. The vessels are removed from the microwave and the contents are stirred. After stirring, the temperature of the water is measured and recorded for use in the formula below.

2.1.6.2 Calibration Formula

2.1.6.2.1 Measure the initial temperature of the water, (T_i), to within 0.1°C. The starting temperature should be between 22 and 26°C. Irradiate 1 kg of water at full power, 100 percent (99, 98, 97, 95, 90, 80, 70, 60, or 50 percent power setting) for 120 seconds. The container must be circulated through the cavity at a rate of at least

one revolution every 30 seconds during the irradiation. Measure the final temperature of the water, after stirring, (T_f), to within 0.1°C, while stirring the water (an electronic stirrer using a large stir bar works best) within 30 seconds of the end of irradiation. Use the maximum reading. Repeat for a new sample, for a total of five replicates per microwave setting, of distilled room-temperature water using a new, clean container. Calculate the microwave power according to the formula:

$$Power = \frac{K \times C_p \times M \times T}{t} \qquad \text{Eq. D/IN-1}$$

$$34.87 = \frac{K \times C_p \times M}{t} \qquad \text{Eq. D/IN-2}$$

$$Power = 34.87 \times T \qquad \text{Eq. D/IN-3}$$

where:

Power = The apparent power absorbed by the sample in watts (W = joule-sec^{-2});
K = The conversion factor for thermochemical calories-sec^{-1} to W (equal to 4.184);
C_p = The heat capacity, thermal capacity, or specific heat (cal-g^{-1}-°C^{-1} = 1.0 for water);
M = The mass of the sample in grams;
T = $T_f - T_i$ in °C; and
t = Time in seconds.

2.1.6.2.2 Derive an equation for the linear portion of the calibration range and determine the equivalent value in watts of the arbitrary setting scale. Use the actual power in watts to determine the appropriate setting of the particular microwave unit being used. Each microwave unit will have its own setting that corresponds to the actual power delivered to the samples.

2.1.6.3 Cleaning Procedure for RFA Vessels

2.1.6.3.1 Prior to first use, new vessels must be annealed before they are used. A pretreatment/cleaning procedure must be followed. This procedure calls for heating the vessels for 96 hours to 200°C. The vessels must be disassembled during annealing and the sealing surfaces (the top of the vessel or the rim) must not be used to support the vessel during annealing.

2.1.6.3.2 Rinse with distilled water. Immerse in a cleaning bath of 1:1 HCl for a minimum of three hours after the bath temperature has reached a temperature just below boiling. Rinse with distilled water. Immerse in a cleaning bath of 1:1 HNO_3 for a minimum of three hours

after the bath temperature has reached a temperature just below boiling. The vessels are then rinsed with copious amounts of deionized, distilled water prior to use for any analyses under this contract. Between sample digestions, the PFA vessels should be detergent washed and 1:1 HNO_3 rinsed followed by a deionized, distilled water rinse.

2.1.6.4 Digestion Procedure for Microwave Extraction

2.1.6.4.1 Prepare extracting acid (2M HCl, 0.9M HNO_3) for use with ICP analysis. In a 1-L volumetric flask, combine in order and mix well 500 mL of deionized, distilled water, 55.5 mL of concentrated (16M) redistilled spectrographic-grade nitric acid, and 167.5 mL of ACS reagent-grade concentrated hydrochloric acid (12M). Cool and dilute to 1 L with deionized, distilled water.

NOTE: Nitric and hydrochloric acid fumes are toxic. Prepare in a well-ventilated fume hood. Mixing results in an exothermic reaction. Stir slowly.

2.1.6.4.2 Prepare nitric acid (3M HNO_3) for use with ICP-MS and GFAA analyses. In a 1-L volumetric flask combine in order and mix well, 500 mL of deionized distilled water and 192 mL of concentrated nitric acid. Slowly dilute to 1 L.

2.1.6.4.3 Cut a 1" x 8" strip from the folded particulate bearing filter using a template and a pizza cutter as described in the Federal Reference Method for Lead (see Figure D-1 and Reference 11). Using vinyl gloves or plastic forceps, accordion fold or tightly roll the filter strip and place it on its edge in a centrifuge tube.

NOTE: A breathing mask and vinyl gloves are required for safety of personnel handling dry glass-fiber filters. The breathing mask prevents the inhalation of minute glass shards and particulate material. The gloves protect the skin from the same materials and also prevent contamination of the sample by skin secretions.

NOTE: It is suggested that more than one strip from a filter be extracted to ensure adequate sample volume for sample and QC sample analysis.

2.1.6.4.4 Add 10.0 mL of extracting acid for ICP analysis or 10 mL of 3 M nitric acid for GFAA or ICP-MS analysis, using a preset calibrated automatic dispensing pipette or Class A regular pipette. (The acid should cover the strip completely). The sequence of adding the filter strip and acid to the centrifuge tube may be reversed, if more convenient, without affecting the results. Place the centrifuge tube in a Teflon PFA vessel containing 31 mL of deionized water.

2.1.6.4.5 The caps with the pressure release valves are placed on the vessels hand-tight, and then tightened using constant torque to 12 ft./lbs. Connect the sample vessel to the overflow vessel using the Teflon PFA connecting tube. Weigh the two vessel assembly to the nearest 0.01 g. Place the vessels in the microwave carousel. Connect the overflow vessels to the center well of the oven (see Figure D-2).

2.1.6.4.6 Place the 12 vessels on the turntable of the microwave unit. Any vessels containing 10 mL of acid solution for analytical blank purposes are counted as sample vessels. Irradiate the sample vessels at 486 W for 23 minutes. (Based on the calibration of the microwave as previously described).

2.1.6.4.7 At the end of the program, remove the turntable containing the microwave vessels and cool it in tap water for 10 minutes. Open the microwave vessels and discard the water that they contain.

2.1.6.4.8 Add 10 mL of deionized distilled water to the centrifuge tube, using the preset calibrated automatic dispensing pipette or using a Class A regular pipette. Cap the centrifuge tube tightly and mechanically shake for five minutes. Place the tube in the centrifuge and operate for 25 minutes at 2000 rpm. Decant the clear supernatant from the centrifuge tube into an weighed acid-cleaned 20-mL polypropylene bottle (bearing sample ID label), taking care not to disturb the solids in the bottom of the tube and weigh the bottle and contents. The matrix is 0.45M nitric acid, 1.03M hydrochloric acid for ICP analysis or 1.5M nitric acid, for ICP-MS and GFAA analyses and deionized, distilled water. The sample is now ready for analysis.

2.2 ALTERNATE SAMPLE PREPARATION FOR ICP-MS AND GFAA ANALYSIS

2.2.1 Introduction

2.2.1.1 This method describes a hot extraction procedure to extract the metals from the particulate loaded glass-fiber filter, for subsequent analysis by inductively coupled plasma-mass spectrometry (ICP-MS) and graphite furnace atomic absorption (GFAA) spectrometry.

2.2.2 Summary of Method

2.2.2.1 The metals are extracted by hot nitric acid from the filter. After cooling to room temperature, the extract is allowed to settle.

2.2.3 Apparatus and Materials

2.2.3.1 Volumetric glassware: 30- to 150-mL capacity (Class A Borosilicate).

2.2.3.2 Hot plate.

2.2.3.3 Centrifuge: Capable of maintaining speeds of 2000 rpm (International Equipment Company Model UV or equivalent).

2.2.3.4 Centrifuge tubes: Polypropylene tubes and screw tops, 100-mL.

2.2.3.5 Pipette: Automatic dispensing with an accuracy of 0.1 mL or better and repeatability of 20 μL, (Grumman Automatic Dispensing Pipet, Model ADP-30 DT or equivalent).

2.2.3.6 Particle mask: To be worn while cutting and handling glass-fiber filters.

2.2.3.7 Template: To aid in sectioning the glass-fiber filter. Federal Register 1978, 43 (Oct. 5), 46258-46261. See Figure D-1.

2.2.3.8 Pizza cutter: Thin wheel (< 1 mm).

2.2.4 **Reagents**

2.2.4.1 Nitric Acid (HNO_3): Concentrated, 16M, redistilled spectrographic grade.

2.2.4.1.1 Nitric Acid, 3M: Prepare by adding 192 mL of concentrated nitric acid (2.2.4.1) to distilled, deionized water and slowly diluting to 1 L.

NOTE: Nitric acid fumes are toxic. Prepare in a well-ventilated fume hood. Stir slowly.

2.2.4.2 ASTM Type II Water (ASTM D1193): Water must be monitored.

2.2.5 **Sample Preservation and Handling**

2.2.5.1 Exposed Hi-Vol filters should be folded in half with particulate material inward, enclosed in protective envelopes, and stored at 15 to 30°C until analysis.

2.2.6 **Hot Extraction Procedure**

2.2.6.1 Cut a 1" x 8" strip as described in the Federal Reference Method for Lead (see Figure D-1).

2.2.6.2 Fold the strip in half twice, and place in a 150-mL beaker. Add 15 mL of 3M HNO_3 to completely cover the sample. Cover the beaker with a watch glass. It is important to keep the sample covered so that corrosion products (formed on fume hood surface which may contain target analytes) are not deposited in the extract.

NOTE: It is suggested that more than one strip per filter be extracted to ensure adequate volume for sample and QC analysis.

2.2.6.3 Gently boil the sample in a beaker on a hot plate under a fume hood for 30 minutes. Do not let the sample evaporate to dryness.

NOTE: Nitric acid fumes are toxic.

2.2.6.4 After 30 minutes, remove the beaker from the hot plate, and cool to near room temperature. Rinse watch glass and sides of beaker with distilled, deionized water.

2.2.6.5 Decant extract and rinsings into a 100-mL volumetric flask. Add distilled, deionized water to the 40-mL mark on beaker, cover with watch glass and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted since it allows the HNO_3 trapped in the filter to diffuse into the rinse water.

2.2.6.6 Decant the water from the filter into the volumetric flask, rinse filter and beaker twice with distilled, deionized water, and add the rinse to the volumetric flask until the total volume is 80 to 85 mL.

2.2.6.7 Stopper flask and shake vigorously, and set aside for approximately five minutes or until foam has dissipated.

2.2.6.8 Bring solution to volume with distilled, deionized water, and mix thoroughly. Allow solution to settle for one hour before proceeding with analysis.

NOTE: Do not filter the extracted sample to remove particulate matter because of possible loss of target analytes due to filtration. The final extract can be centrifuged at 2000 RPM for 30 minutes to remove any suspended solids.

2.2.6.9 If sample is to be stored for subsequent analysis, transfer to a polyethylene bottle, being careful not to disturb the settled solids.

2.2.6.10 Samples prepared by hot extraction procedure are now in 0.45M HNO_3.

2.2.6.11 Blank filter samples should be extracted and analyzed, and digestion blanks should be run to ensure low levels of metals in the reagents used.

SECTION 3

SAMPLE ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY

3.1 INTRODUCTION

3.1.1 Metals for which this method is applicable are listed in Table 1, Exhibit C, and are determined by ICP after sample preparation by microwave digestion. Appropriate steps must be taken to correct for potential interference effects.

3.1.2 Table D-1 lists analytes along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other analytes may be added as more information becomes available.

3.1.3 Because of the differences among various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

3.2 SUMMARY OF METHOD

3.2.1 The analyte concentrations are determined by ICP atomic emission spectroscopic analysis. The acid extracts are pumped into a pneumatic nebulizer. The aerosol formed is transported into an inductively coupled plasma and the metals are excited into higher electronic states. Atomic and ionic line emission spectra characteristic of the particular metals are produced when the electrons decay back to the lower energy levels. The spectra are dispersed by a spectrometer and the intensity of specific line radiation(s) are monitored simultaneously or sequentially by a photomultiplier tube(s). The photocurrent produced by the photomultiplier tube will increase in direct proportion to the concentration of the element in the sample within the linear range of a specific emission line. The photocurrent is processed by a computer system and related to concentration through a calibration procedure.

3.2.2 Calibration is performed by standardizing the instrument with a series of mixed element standards and a blank that are matrix matched to the acid extract solution. Every solution analyzed, such as a dilution, calibration stability standard, or reference sample must be matrix matched to the sample extract.

3.2.3 Appropriate steps must be taken to ensure that potential interferences are corrected. This is especially important for spectral

interferences. Recommendations for correcting for interferences are briefly summarized below under headings that categorize the type of interference that is being considered.

3.2.3.1 **Recommended Corrections for Physical Interferences**

- The use of peristaltic pump to introduce the acid extract into the nebulizer.
- Frequent (20 percent or better) analysis of the calibration stability standard.
- Adequate rinsing (one minute or more) between sample analyses using ten percent HNO_3 or ten percent HCl, and optional use of humidified argon or a nebulizer tip washer as necessary.

3.2.3.2 **Recommended Corrections for Chemical Interferences**

- Matrix matching between samples and all standards and sample diluting the sample during analysis.

3.2.3.3 **Recommended Corrections for Spectral Interferences**

- Use of calculated interelement corrections in the form of factors or first or second order equations that describe the interference function (on-peak correction).
- Optional use of measurement of background shift on either side or both sides of the analyte line (off-peak correction).
- Optionally, wavelength scans (for each of the analyte element wavelengths) for each of the samples simultaneously plotted with a calibration blank scan and a calibration standard scan may be performed.

3.2.4 Every solution, including calibration standards, calibration and method blanks, and reference samples, must be analyzed using two full exposures (peak scan), each of which is sufficient to meet the method quantitation limit (MQL) for each analyte emission line. All exposure times must be the same for all analyses and all quarterly analyses (i.e., MQL and interelement correction factor.)

3.2.5 Both the off-peak (background) and on-peak (interelement correction coefficients or equations) interference corrections made for all analytes must be calculated and reported with the analysis results.

3.2.6 If the sum of the values of the interference correction(s) made on any analyte is greater than the resulting analyte concentration, the reported value is to be flagged with an "I" on FORM I-AAIN.

3.2.7 If the analyte requiring dilution interferes with another analyte, the interference correction(s) must reflect the actual concentration of the interferent in the undiluted samples.

3.2.8 The specific spectral lines that are employed must be reported.

3.2.9 All reported analyte data must have been obtained within the linear range of the respective analyte emission line. If any analyte concentration results in the linear range of the spectral line being exceeded, the sample must be diluted such that the resulting solution concentration falls within the linear range, but not below the CRQL.

3.3 SAFETY

3.3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets shall be available to all personnel involved in the chemical analysis.

3.4 INTERFERENCES

3.4.1 ICP emission spectroscopy is prone to interelement effects, which in practice are experienced in two main forms. Interferences that cause a translation of the analytical curve are caused by spectral line overlap or increase in background due to an electron/metal ion recombination continuum and/or scattered light within the spectrometer. For a given matrix and spectral line, a constant additive interference is produced that is independent of the analyte concentration. Rotational interference of the analytical curve, experienced essentially as a change in sensitivity, is due to the combined effects of variations in nebulizer performance produced by the physical properties of the sample solution and changes in the excitation conditions in the plasma caused by the matrix metals. Both forms of interference can operate simultaneously for a particular sample matrix.

3.4.2 Spectral interference from poorly resolved metal spectral lines, such as scattered light, or broad continuum spectral background, will lead to systematic error in the analytical results unless proper corrections are made. Molecular band emission will lead to a deterioration of the quantitation limit and will increase the difficulty of off-peak background correction. Methods of correcting translational interference (other than exact matrix matching of the standards to the sample) include the on-peak correction technique. This method can be applied to both spectral line overlap and background enhancement, but it requires specific knowledge of the metals causing the interference. On-peak correction can only be performed on a quantitative basis if the interfering metals are included

in the multi-metal analysis, although uncertainties may still exist in whether the correction coefficients employed match those required for the particular sample matrix.

3.4.3 Rotational calibration curve interference manifests itself for a given matrix and spectral line as a change in the slope of the analyte calibration. This type of interference includes: sample nebulization and transport effects, often called physical interference including lateral diffusion interferences; and chemical interferences such as solute vaporization interference and ionization interferences. Such interferences can be reduced by matrix matching of the standards and samples and by the method of standard additions, (although standard additions can become quite lengthy and impracticable for multi-metal analyses) and by the use of internal standards. Matrix matching can correct for any of these interferences but the correction is dependent on the accuracy of the matching. Variations in the matrix from sample to sample will cause corresponding inaccuracies in the analyte results.

3.4.4 Listed in Table D-2 are some interference effects for the recommended wavelengths given in Table D-1. The data in Table D-2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed. The interference information, which was collected at the Ames Laboratory (USOOE, Iowa State University, Ames, Iowa 50011), is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 mg/L of the interferent element.

3.5 **APPARATUS AND EQUIPMENT**

3.5.1 Computer-controlled inductively coupled plasma atomic emission spectrometer system with:

- Polychromator with associated dispersion and detector system such that the metals can be determined simultaneously, or a sequential scanning instrument that allows achievement of the quality control requirements for this method;
- Radio frequency generator and coupling system;
- Pneumatic nebulizer; and
- Software capable of performing both off-peak (background correction) and on-peak (coefficients of first or second order regression equations describing expected interference) spectral interference corrections. In addition, the software must be capable of creating a hardcopy output of both types of corrections in either concentration units or analyte raw intensity data along with net calculated concentrations.

3.5.2 Argon gas supply: Welding grade or better.

3.5.3 Assorted laboratory volumetric glassware, pipets and micropipets.

3.5.4 Operating conditions: Because of the differences among various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

3.6 REAGENTS AND STANDARDS

3.6.1 In the determination of trace elements, containers can introduce either positive or negative errors in the measurement of trace elements by contributing contaminants through leaching or surface desorption and depleting concentrations through adsorption. Thus the collection and treatment of the samples prior to analysis require particular attention. The following cleaning treatment sequence has been determined to be adequate to minimize contamination in the sample bottles, whether borosilicate glass, linear polyethylene, or Teflon is used: detergent, ASTM Type II water, 1:1 hydrochloric acid, ASTM Type I water, 1:1 nitric acid, and ASTM Type I water.

NOTE: Chromic acid shall not be used because chromium is one of the contract required analytes, and its use may lead to cross contamination.

NOTE: Acids used in the preparation of standards and for sample processing must be below the CRQLs for the analytes of interest for the purpose of this document.

3.6.2 Nitric acid and hydrochloric acid used in the preparation of standards and for sample processing must be of high purity.

3.6.3 Water equivalent to ASTM Type II is used throughout.

3.6.4 Stock standard solutions: Standards must be from ultrapure materials. The stock standard solutions may be the same as the spiking standard solutions, if desired.

3.6.5 Spiking standard solutions: Standards must be made from ultrapure materials. Both multi-metal and single metal solutions will be needed. Because of the limited sample volume (100 mL), multi-metal solutions will be needed to maintain the sample matrix at 95 percent original strength after the addition of the spike volume.

3.6.6 No more than five multi-metal stock standards will be required containing metals in the following concentrations:

Metal	Conc. mg/L
Na, Al, Ca, As, Se	1000
Ba, Co, Mn, Ni, Pb, Ag, Tl, V, K	500
Be, Cd, Cu, Cr, Zn, Sb, Sn, Mo, Fe, Mg	100

3.6.6.1 Using the appropriate metal salts and solution matrices, the following standards have been found to be stable for one year.

Metals Mixes	Matrix
Al, Ba, Be, Fe, Ni, Ag, Na, Tl	20 percent conc. HCl
Ca, Cd, Co, Cu, Pb, Mg, Mn, Zn	10 percent conc. HNO_3
Cr	Water
V	2 percent conc. HNO_3

3.6.7 A single metal stock standard at 1000 mg/L will be needed for each metal.

3.6.8 Calibration standards: Prepare calibration standards by dilution of stock or spiking standard solutions. All calibration standards must be matrix matched with the extracting acid solution according to the preparation procedure used in the analysis. Concentrated hydrochloric acid can be used instead of HNO_3 if required for stabilization of a metal(s).

3.6.9 Calibration blank: Prepare calibration blanks such that the resulting solution is matrix matched with the extracting acid solution according to the preparation procedure used in the analysis.

3.6.10 Initial calibration verification: The initial calibration verification solution shall be from a different source than that used for the calibration standards and shall be approximately in the middle of the respective calibration curve. This verification standard must be in the same acid matrix as the calibration standards.

3.6.11 ICP interference check sample: Prepare by dilution of the stock standards if it is not available from the EPA. If the solution is prepared by the analyst, it shall be made using the concentrations in Table 3, Exhibit C. It shall be run at least five times and the mean standard deviation shall be reported in the raw data.

NOTE: The interference check solution(s) (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

3.7 QUALITY CONTROL

3.7.1 Instrument Calibration

3.7.1.1 Summary

Prior to the analysis of samples and required QC, each ICP system shall be initially calibrated to determine instrument sensitivity.

3.7.1.2 Frequency

Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up.

3.7.1.3 Procedure

Calibration standards shall be prepared using the same type of matrix and at the same concentration as the preparation blank following sample preparation.

Calibrate according to instrument manufacturer's recommended procedures using at least two standards, one being a blank.

Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample.

3.7.1.4 Calculations

$$\% \ Recovery = \frac{Found \ Concentration}{True \ Concentration} \times 100 \qquad \text{Eq. D-4}$$

3.7.1.5 Technical Acceptance Criteria

Recovery for the highest mixed calibration standard shall be within ± five percent of the true value (i.e., 95-105 percent).

3.7.1.6 Corrective Action

Follow instrument manufacturer's recommendations to correct the problem. Also, baseline correction is acceptable as long as it is performed after every sample or after the continuing calibration verification and blank check; resloping is acceptable as long as it is immediately preceded and immediately followed by a CCV and a CCB.

3.7.1.7 Documentation

The instrument standardized data and time shall be included in the raw data. The final concentration should be in μg/L.

3.7.2 Initial Calibration Verification

3.7.2.1 Summary

Immediately after the ICP system has been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of EPA Initial Calibration Verification Solution(s) (ICV) at each wavelength used for analysis.

3.7.2.2 Frequency

Each time the instrument is calibrated, the ICV shall be run immediately following the calibration, before any samples are analyzed.

3.7.2.3 Procedure

If the ICV solution(s) are not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the linear range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

3.7.2.4 Calculations

$$\%\ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-5}$$

3.7.2.5 Technical Acceptance Criteria

Recovery for the ICV shall be within ± 10 percent of the true value (i.e., 90-110 percent).

3.7.2.6 Corrective Action

When recoveries of the ICV exceed the technical acceptance criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

3.7.2.7 Documentation

Report the ICV found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM II-AAIN.

3.7.3 Continuing Calibration Verification

3.7.3.1 Summary

To ensure calibration accuracy during an analysis run, a continuing calibration verification solution (CCV) is analyzed and reported for every wavelength used for the analysis of each analyte.

3.7.3.2 Frequency

The CCV is run at a frequency of 10 percent or every two hours during an analysis run, whichever is more frequent.

The CCV is also run after the last analytical sample in the analysis run.

3.7.3.3 Procedure

The same CCV shall be used throughout the analysis runs for a Case of samples received. The analyte concentrations in the continuing calibration standard shall be an EPA solution or a Contractor prepared standard solution and should be at or near ± 10 percent of the mid-range levels of the calibration curve.

Each CCV analyzed shall reflect the conditions of analysis for all of the associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses and other related operations that may affect the CCV measured result shall not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it as well as the difference in time between the CCV and the analytical sample immediately preceding it shall not exceed the smallest difference in time between any two consecutive analytical samples associated with the CCV.

3.7.3.4 Calculations

$$\%\ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-6}$$

3.7.3.5 Technical Acceptance Criteria

Recovery for the CCV shall be within ± 10 percent of the true value (i.e., 90-110 percent).

3.7.3.6 Corrective Action

When recoveries of the CCV exceed the technical acceptance criteria, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration reverified, and the preceding 10 analytical samples reanalyzed (or all analytical samples since the last compliant CCV).

3.7.3.7 Documentation

Report the CCV found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM II-AAIN.

3.7.4 CRQL Standard

3.7.4.1 Summary

To verify linearity near the CRQL, the Contractor shall analyze an ICP standard at two times the MQL or two times CRQL, whichever is greater. This standard shall be run for every wavelength used for analysis.

3.7.4.2 Frequency

The CRQL standard shall be run at the beginning and end of each sample analysis run, or a minimum of twice per eight hours, whichever is more frequent.

3.7.4.3 Procedure

The CRQL standard shall not be run before the ICV solution.

3.7.4.4 Calculations

$$\%\ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-7}$$

3.7.4.5 Technical Acceptance Criteria

Recovery of the CRQL standard shall be within ± 15 percent of the true value for each wavelength used for analysis.

3.7.4.6 Corrective Action

If the CRQL standard does not fall within the control limit, the analysis shall be terminated, the problem corrected and the analytical samples since the last compliant CRQL standard reanalyzed.

3.7.4.7 Documentation

Report the CRQL standard found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM III-AAIN.

3.7.5 **Linear Range Analysis (Quarterly)**

3.7.5.1 Summary

The concentration range over which the ICP calibration curve remains linear must be determined and any values above this linear range shall be diluted and reanalyzed.

3.7.5.2 Frequency

For all ICP analyses, a linear range verification check standard shall be analyzed and reported quarterly (every 3 calendar months) for each element on the target analyte list (Table 1, Exhibit C). The standard shall be analyzed during a routine analytical run performed under this contract. This standard shall be run for all wavelengths used for each analyte reported by ICP.

3.7.5.3 Procedure

The standard shall be analyzed as though it were a separate analytical sample (i.e., each measurement shall be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples).

3.7.5.4 Calculations

$$\% \ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-8}$$

3.7.5.5 Technical Acceptance Criteria

Recovery for the linear range standard shall be within ± 5 percent of the true value (i.e., 95-105 percent).

3.7.5.6 Corrective Action

If the recovery of the linear range standard does not meet the technical acceptance criteria, then the analysis shall be terminated and successive dilutions of the standard shall be reanalyzed until the control limits are met. The concentration of this standard that meets the control limits is the upper limit of the instrument linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

3.7.5.7 Documentation

Report the linear range standard found concentration (in µg/L), true concentration (in µg/L) and percent recovery for each analyte on FORM III-AAIN.

3.7.6 Initial Calibration Blank

3.7.6.1 Summary

To verify that the ICP system is not contaminated, an initial calibration blank (ICB) shall be analyzed after calibration.

3.7.6.2 Frequency

The ICB shall be analyzed each time the system is calibrated and immediately after the ICV.

3.7.6.3 Procedure

If the absolute value of the ICB is greater than the MQL, the result shall be reported.

3.7.6.4 Calculations

Not applicable.

3.7.6.5 Technical Acceptance Criteria

The absolute value of the ICB must be less than the CRQL.

3.7.6.6 Corrective Action

When the ICB concentration does not meet the technical acceptance criteria, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the ICB.

3.7.6.7 Documentation

Report the ICB values in µg/L on FORM IV-AAIN.

3.7.7 Continuing Calibration Blanks

3.7.7.1 Summary

To ensure that the system is not contaminated during the analysis run, continuing calibration blanks (CCB) are analyzed.

3.7.7.2 Frequency

Analyze the CCB at a frequency of 10 percent or every two hours, whichever is more frequent.

Analyze the CCB after every CCV.

3.7.7.3 Procedure

A CCB shall be run after the last CCV in the analysis run. If the absolute value of the CCB is greater than the MQL, the result shall be reported.

3.7.7.4 Calculations

Not applicable.

3.7.7.5 Technical Acceptance Criteria

The absolute value of the CCB must be less than the CRQL.

3.7.7.6 Corrective Action

When the CCB concentration does not meet the technical acceptance criteria, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the preceding 10 analytical samples (or all analytical samples since the last compliant CCB).

3.7.7.7 Documentation

Report the CCB values in µg/L on FORM IV-AAIN.

3.7.8 Preparation Blanks

3.7.8.1 Summary

To ensure against contamination during sample preparation, a preparation blank (PB) is analyzed.

3.7.8.2 Frequency

At least one PB must be prepared and analyzed with every SDG or with each batch of samples digested, whichever is more frequent.

3.7.8.3 Procedure

The PB shall consist of ASTM Type II water processed through each sample preparation and analysis procedure step.

The first batch of samples in a SDG shall be assigned to PB one, the second batch of samples to PB two, etc.

3.7.8.4 Calculations

Not applicable.

3.7.8.5 Technical Acceptance Criteria

The absolute value of the PB must be less than the CRQL.

3.7.8.6 Corrective Action

If the absolute value of the concentration of the PB is less than or equal to the CRQL, no corrective action is required.

If any analyte concentration in the PB is above the CRQL, the lowest concentration of the analyte in the associated samples must be 10 times the PB concentration. Otherwise, all samples associated with the PB and with the analyte's concentration less than 10 times the PB concentration and above the CRQL shall be redigested and reanalyzed for that analyte. The sample concentration is not to be corrected for the PB value.

If an analyte concentration in the PB is below the negative CRQL, then all samples reported below 10 times CRQL associated with the PB shall be redigested and reanalyzed.

3.7.8.7 Documentation

The values for the PB shall be recorded in µg/L on FORM IV-AAIN.

3.7.9 ICP Interference Check Sample

3.7.9.1 Summary

To verify interelement and background correction factors, an ICP Interference Check Sample (ICS) is analyzed.

3.7.9.2 Frequency

Analyze the ICS at the beginning and end of each analysis run or a minimum of twice per eight hour working shift, whichever is more frequent, but not before the ICV.

3.7.9.3 Procedure

The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of

the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP.

The ICP ICS shall be obtained from EPA (EMSL/LV) if available and analyzed according to the instructions supplied with the ICS.

If the ICP ICS is not available from EPA, an independent ICP ICS shall be prepared with interferent and analyte concentrations at the levels specified in Table 3, Exhibit C. The mean value and standard deviation shall be established by initially analyzing the ICS at least five times repetitively for each analyte.

If true values for analytes contained in the ICS and analyzed by ICP are not supplied with the ICS, the mean shall be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination shall be made during an analytical run where the results for the previously supplied EPA ICS met all contract specifications. Additionally, the result of this initial mean determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

3.7.9.4 Calculations

$$\% \ Recovery = \frac{Found \ Concentration}{True \ Concentration} \times 100 \qquad \text{Eq. D-9}$$

3.7.9.5 Technical Acceptance Criteria

Recovery for the ICS shall be within ± 20 percent of the true value (i.e., 80-120 percent).

3.7.9.6 Corrective Action

If the ICS recoveries does not meet the technical acceptance criteria, terminate the analysis, correct the problem, recalibrate the instrument, verify the calibration, and reanalyze all of the analytical samples since the last compliant ICS was analyzed.

3.7.9.7 Documentation

Report the ICS found concentration (µg/L), true concentration (µg/L), percent recovery, mean and standard deviation on FORM V-AAIN.

The mean and standard deviation shall be reported in the raw data.

3.7.10 Spike Sample Analysis

3.7.10.1 Summary

To provide information about the effect of the sample matrix on the digestion, a known amount of analyte is added (spiked) into a sample.

3.7.10.2 Frequency

At least one spike sample analysis shall be performed on each group of samples for each SDG. EPA may require additional spike sample analysis upon special request by the Project Officer, for which the Contractor will be paid.

If two analytical methods are used to obtain the reported values for the same analyte within a SDG (e.g., ICP and GFAA), then spike samples shall be run by each method used.

3.7.10.3 Procedure

The spike is added before the sample preparation (i.e., prior to digestion) at concentration levels in the Spike Sample solution as indicated in Table D-3.

Samples identified as field blanks cannot be used for spiked sample analysis. EPA may require that a specific sample be used for the spike sample analysis. In the instance where there is more than one spike sample per SDG and one spike sample recovery is not within contract criteria, flag all the samples of the same matrix, level, and method in the SDG.

3.7.10.4 Calculations

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100 \qquad \text{Eq. D-10}$$

where:

SSR = Spiked Sample Result;
SR = Sample Result; and
SA = Spike Added.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see part 3.7.11, Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery.

When the sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating percent recovery.

3.7.10.5 Technical Acceptance Criteria

Recovery for the spike should be within ± 25 percent of the spiked amount (i.e., 75-125 percent).

3.7.10.6 Corrective Action

If the spike recovery is not within the limits of 75-125 percent, the data of all samples received associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N" on FORMs I-AAIN and VI-AAIN.

An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125 percent recovery criteria.

3.7.10.7 Documentation

Report the spiked sample results, sample results, spike added and percent recovery for the spike sample analysis on FORM VI-AAIN.

The units for reporting spike sample results will be in µg/L.

3.7.11 Duplicate Sample Analysis

3.7.11.1 Summary

Duplicate aliquots of a sample are carried through the preparation and analysis steps to provide information about the precision of the analytical methods as well as matrix effects.

3.7.11.2 Frequency

At least one duplicate sample analysis shall be performed on each group of samples for each SDG. EPA may require additional duplicate sample analysis upon special request by the Project Officer, for which the Contractor will be paid.

If two analytical methods are used to obtain the reported values for the same analyte within a SDG (e.g., ICP and GFAA), then duplicate samples shall be run by each method used.

3.7.11.3 Procedure

Samples identified as field blanks cannot be used for duplicate sample analysis.

EPA may require that a specific sample be used for the duplicate sample analysis.

In the instance where there is more than one duplicate sample per SDG and one duplicate result is not within contract criteria, flag all the samples of the SDG.

Duplicate sample analyses are required for calculations of relative percent difference.

3.7.11.4 Calculations

$$RPD = \frac{|S - D|}{\frac{S + D}{2}} \times 100 \qquad \text{Eq. D-11}$$

where:

RPD = Relative Percent Difference;
S = First Sample Value (original); and
D = Second Sample Value (duplicate).

Duplicates cannot be averaged for reporting on FORM I-AAIN.

3.7.11.5 Technical Acceptance Criteria

A control limit of ± 20 percent for RPD shall be used for original and duplicate sample values greater than or equal to five times CRQL (Exhibit C). A control limit of ± CRQL shall be used for sample values less than five times CRQL.

If one result is above the five times CRQL level and the other is below, use the ± CRQL criteria.

If both sample values are less than the MQL, the RPD is not calculated.

Specific control limits for each analyte will be added to FORM IX-AAIN at a later date, based on precision results.

3.7.11.6 Corrective Action

If the duplicate sample results are outside the control limits, flag with an asterisk all the data for samples received associated with that duplicate sample.

3.7.11.7 Documentation

The results in µg/L of the duplicate sample analyses shall be reported on FORM VIII-AAIN.

The absolute value of the control limit (CRQL) shall be entered in the "CONTROL LIMIT" column on FORM VIII-AAIN.

3.7.12 Laboratory Control Samples

3.7.12.1 Summary

A laboratory control sample (LCS) is digested and analyzed to ensure against analyte loss in the sample preparation.

3.7.12.2 Frequency

One LCS shall be prepared and analyzed for every group of samples in a SDG, or for each batch of samples, whichever is more frequent.

3.7.12.3 Procedure

A LCS shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

The LCS solution shall be obtained from EPA. (If unavailable, other EPA Quality Assurance Check samples or other certified materials may be used.)

3.7.12.4 Calculations

$$\% \ Recovery = \frac{Found \ Concentration}{True \ Concentration} \times 100 \qquad \text{Eq. D-12}$$

3.7.12.5 Technical Acceptance Criteria

Recovery for the LCS shall be within ± 20 percent of the true value (i.e., 80-120 percent), with the exception of Ag and Sb.

3.7.12.6 Corrective Action

If the percent recovery for the LCS falls outside the technical acceptance criteria, then the analyses shall be terminated, the problem corrected, and the samples associated with that LCS reprepared and reanalyzed.

3.7.12.7 Documentation

Report the LCS found concentration (µg/L), true concentration (in µg/L), and percent recovery on FORM IX-AAIN.

3.7.13 Performance Evaluation Sample (PES)

3.7.13.1 Summary

The performance evaluation sample (PES) assists the Agency in monitoring the laboratory performance for analyte identification and quantification.

3.7.13.2 Frequency

One PES shall be delivered, prepared, and analyzed for every group of samples in a SDG, or for each batch of samples, whichever is more frequent.

3.7.13.3 Procedure

A PES shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

The PES solution shall be obtained from EPA. (If unavailable, the contractor must contact the Agency and SMO for instructions.)

3.7.13.4 Calculations

Not applicable.

3.7.13.5 Technical Acceptance Criteria

Recovery for the PES shall be within 75 percent of the true value of analyte.

3.7.13.6 Corrective Action

If the percent recovery for the PES falls below 75 percent, the Agency may take, but is not limited to the following actions:

- Show Cause and/or Cure Notice for unacceptable performance;
- Reduction of the number of samples shipped to the laboratory;
- Suspension of sample shipment;

- A site visit;
- A full data audit; or
- Require the laboratory to analyze remedial PES.

In addition to the above cited actions, the laboratory shall also provide the Technical and Administrative Project Officers a written correction action report on every unacceptable (less than 75 percent) PES score within seven days of written or verbal request by Agency personnel.

3.7.13.7 Documentation

Report the PES on Form I-AAIN (in µg/L) for all analytes.

3.7.14 Method Quantitation Limits

3.7.14.1 Summary

Prior to sample analysis, the method quantitation limit (MQL) shall be determined for each instrument that will be used.

3.7.14.2 Frequency

MQLs shall be determined within 30 days of the start of the contract and at least quarterly (every three calendar months) until the end of the contract.

3.7.14.3 Procedure

MQLs (in µg/L) shall be determined by multiplying by three, the average of the standard deviations (σ_{n-1}) obtained on three nonconsecutive days from the consecutive analysis of seven different PB extracts. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). MQLs shall be determined and reported for each wavelength used in the analysis of the samples.

The quarterly determined MQL for an instrument shall always be used as the MQL for that instrument during that quarter. If the instrument is adjusted in anyway that may affect the MQL, the MQL for that instrument shall be redetermined and the results submitted for use as the established MQL for that instrument for the remainder of the quarter.

3.7.14.4 Calculations

$$MQL = 3 \times (\sigma_{n-1}) \qquad \text{Eq. D-13}$$

3.7.14.5 Technical Acceptance Criteria

The MQLs shall be ≤ the CRQLs in Exhibit C.

3.7.14.6 Corrective Action

If an instrument's MQL exceeds the CRQL for an analyte, that instrument cannot be used in this document unless the analyte concentration is greater than or equal to five times the reported MQL.

3.7.14.7 Documentation

MQLs shall be submitted with each data package and reported on FORM XI-AAIN for each instrument used. If multiple instruments are used for the analysis of an analyte within an SDG, the highest MQL for the analyte shall be used for reporting concentration values for that SDG.

3.7.15 ICP Serial Dilution

3.7.15.1 Summary

The serial dilution shall be performed to check for the presence of matrix interference.

3.7.15.2 Frequency

One serial dilution shall be performed for each SDG.

3.7.15.3 Procedure

The serial dilution shall be performed by diluting a prepared sample aliquot five fold (1:4). The dilution must be performed on an analyte by analyte bases. The serial dilution is the dilution of the sample, or an aliquot of the sample, that contains a concentration level of the analyte within the linear range.

3.7.15.4 Calculations

$$\%D = \frac{|I - S|}{I} \times 100 \qquad \text{Eq. D-14}$$

where:

I = Initial Sample Result; and
S = Serial Dilution Result.

3.7.15.5 Technical Acceptance Criteria

If the original sample concentration is greater than 50 times the MQL, the %D should be within ± 10 percent of the initial sample result.

3.7.15.6 Corrective Action

If the serial dilution results are outside the control limits, flag all the data for the affected analytes in the samples received associated with that serial dilution with an "E" on FORM XI-AAIN and FORM I-AAIN.

3.7.15.7 Documentation

The values for the serial dilution shall be recorded in µg/L on FORM XI-AAIN.

3.7.16 Interelement Correction Factors

3.7.16.1 Summary

To ensure against spectral interferences, interelement correction factors are determined for all wavelengths used for each analyte reported by ICP.

3.7.16.2 Frequency

Before any field samples are analyzed under this contract, the ICP interelement correction factors shall be determined within three months prior to the start of contract analyses and at least annually thereafter.

3.7.16.3 Procedure

Correction factors for spectral interference due to Al, Ca, Fe, K, Na, and Mg shall be determined for all ICP instruments at all wavelengths used for each analyte reported by ICP. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, K, Na, and Mg shall be reported if they were applied.

If the instrument was adjusted in any way that may affect the ICP interelement correction factors, the factors shall be redetermined and the results submitted for use.

Follow the instrument manufacturer's recommendations for applying interelement correction factors.

3.7.16.4 Calculations

Not applicable

3.7.16.5 Technical Acceptance Criteria

Not applicable

3.7.16.6 Corrective Action

Not applicable

3.7.16.7 Documentation

Results from interelement correction factors determination shall be reported on FORM XI-AAIN for all ICP parameters.

3.8 INSTRUMENT OPERATION

3.8.1 No detailed operating instructions as to the optimization of the plasma power, argon flows, torch and coil configuration, etc. will be given. The analyst should follow the instructions provided by the manufacturer of the particular instrument.

3.8.2 The sample introduction system is to be of a pneumatic type. The use of a peristaltic pump instead of direct aspiration is required. A tip washer is a very useful aid and can be easily inserted into the sample flow system by placing a "tee connector" on the carrier argon flow line just before entering the nebulizer. One arm of the "tee connector" runs to the nebulizer, another to the carrier argon flow line and the third is connected to a line from the peristaltic pump. When a drop in the carrier flow is observed, a small pulse of water is pumped into the carrier argon flow and blown through the nebulizer orifice, dissolving the salt buildup and restarting the original carrier argon flow.

3.8.3 Changes in the carrier argon flow may change the emission characteristics of the analyte. The use of a digital mass flow controller is recommended to control the carrier argon flow.

3.8.4 For direct reading instruments, every solution, including calibration standards, calibration and method blanks, reference samples, and samples shall be analyzed using two full exposures (peak scan), each of which is sufficient to meet the MQL (at each analyte emission line). All exposure times shall be the same for all analyses and all quarterly analyses (i.e., LRS, MQL, and interelement correction factor). Each

background spectral region shall have an exposure time equivalent to a full exposure time for direct reading instruments.

3.8.5 Selection of the appropriate background spectral region for each analyte shall account for the major interferents within that region and for the possibility of analyte line broadening at high concentrations. One of the best ways to select the appropriate background spectral region is to perform a wavelength scan around the analyte wavelengths in the presence of metals frequently encountered at high levels in the samples. Alternately, if the instrument does not have automatic scanning capability, selection of the background spectral region will have to be determined on the basis of manual scans and experience.

3.8.6 A calibration blank rinse between each sample aspiration should be sufficient to prevent carry-over between samples.

3.8.7 The determination of the linear range of each analyte line, interference effects, and any type of detection limit or precision measurement shall be established under the same conditions used for the analysis of the samples, including the background correction scheme.

3.9 PROCEDURE

3.9.1 Calibration

3.9.1.1 Set up the instrument with proper operating parameters. The instrument shall be allowed to become thermally stable before beginning analysis. This requires at least 30 minutes of operation with the plasma lit prior to calibrations.

3.9.1.2 Initiate appropriate operating configuration of the instrument computer.

3.9.1.3 Perform the appropriate steps recommended by the manufacturer to align the exit slits with the entrance slit. These steps are commonly called the profile or wavelength calibration procedure.

3.9.2 Analysis Sequence

3.9.2.1 Before beginning the sample analysis run, analyze the initial calibration blank (ICB), initial calibration verifications (ICV), interference check sample, and the CRQL standard (CRI). The ICV and LRS concentration values shall not deviate from the actual values by more than 10 percent, while the CRI shall not deviate from its actual values by more than 15 percent. The calibration blank values shall not exceed the CRQL. The interference check sample found values shall not deviate by more than 20 percent of the true values. If these conditions are not met for any analyte, discontinue the analysis and refer to Exhibits D

(Quality Control - Initial and Continuing Calibration Blanks) and E for additional information.

3.9.2.2 Upon successful analysis of the ICV, ICB, CRI, and ICS, analyze all PB extract(s) prepared with the samples. If any of the blank(s) values are not less than or equal to the CRQL, see Exhibits D and E for the appropriate action.

3.9.2.3 If the PB and LCS values are within the acceptable ranges, analyze the spike sample. If the recovery of any analyte deviates from the acceptable ranges, see Exhibit E for the appropriate action. Proceed to the analysis of samples if the recoveries are acceptable or after consulting Exhibits D and E.

3.9.2.4 The continuing calibration verification standard (ICV) and the continuing calibration blank (CCB) shall be analyzed after every 10 analytical sample analyses. The analyst shall run CCV and CCB samples after the analysis of the previous sample, but prior to use of a tip wash or other clean out device. CCV concentration values shall not deviate from the actual values by more than 10 percent. In addition, the absolute values for the CCB shall be lower than the CRQLs. If these conditions are not met at any time during samples analysis, discontinue the analysis and see Exhibits D and E for the appropriate action.

3.9.2.5 At the end of the sample analysis run, analyze the ICS, CRI, and CCB. If the values for any of these samples deviates from the required limits, see Exhibits D and E for additional information.

3.9.3 **Sample Analyses**

3.9.3.1 All sample extracts shall first be analyzed without any dilution. Diluting sample extracts is permissible if necessary, provided that the dilution does not produce results below CRQL.

3.9.3.2 All concentrations within the linear range of the analyte shall be reported. All concentrations reported shall be obtained within the established linear range for that analysis run, and interference corrections shall be made based on the actual concentration of the interferent and not the apparent concentration obtained when the interferent concentration is above the linear range.

3.9.4 **Calculations**

3.9.4.1 To obtain the analyte air concentration (in $\mu g/m^3$), multiply the interference-corrected analyte values (in $\mu g/L$) by nine times the appropriate volume (in liters) used in the extraction and divide by the volume of air (in standard m^3) sampled and the number of 1"x 8" strips digested for the sample. The factor "9" represents the total area (63

in^2) of the Hi-Vol filter exposed to the air stream divided by the exposed area (7 in^2) in one 1"x 8" strip.

$$\text{Analyte Conc. air, } \mu g/m^3 = \frac{9 \times \text{Interference-Corrected Analyte Value, } \mu g/L \times \text{Extract Volume, L}}{\text{no. of strips digested} \times \text{air volume sampled, std. } m^3} \quad \text{Eq. D-15}$$

3.9.5 Documentation

3.9.5.1 Report the air concentration values in $\mu g/m^3$ and the interference-corrected analyte value in $\mu g/L$ on FORM I-AAIN.

SECTION 4

SAMPLE ANALYSIS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS)

4.1 SCOPE AND APPLICATION

4.1.1 Metals for which this method is applicable are listed in Table 2, Exhibit C in are determined by ICP-MS after sample preparation by microwave digestion (Section 2 of this Exhibit). Instrument quantitation limits, sensitivities, and linear ranges for these elements will vary with the matrices, instrumentation, and operating conditions. Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and the correction of spectral, chemical, and physical interferences in ICP-MS. The experience requirement is one year on a commercially available ICP-MS.

4.2 SUMMARY OF METHOD

4.2.1 The method describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and by means of a water cooled interface, introduced into a quadrupole mass spectrometer, capable of providing a resolution better than or equal to 1 amu peak width at 10 percent of the peak height. The water-cooled interface consisting of tandem skimmers, is differentially pumped and leads into the high vacuum chamber of the mass spectrometer. The ions and ion clusters produced in the plasma and those formed during the introduction of the ion beam into the mass spectrometer, are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

4.3 INTERFERENCES

4.3.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Table D-10, shows isobaric interferences and the secondary masses which would be analyzed to correct for these interferences. A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting out the appropriate signal from the isotope of interest. Data that is corrected must be noted in the report along with the exact calculations used. Commercial ICP-MS instruments nominally provide unit

resolution at 10 percent of the peak height, and very high ion currents at adjacent masses can contribute to ion signals at the mass of interest. Table D-9 shows approximate concentrations at which adjacent masses give rise to a contribution of 10 µg/L to the analyte of interest at a resolution of 1 amu at 10 percent peak height, if the mass were chosen for quantitation. It should be noted that the information described in Table D-9, was experimentally derived and the interferences which are described occur from several different sources. One interference is the effect of resolution on adjacent peaks. This has a larger effect at 1 amu less than the interferant than at one amu greater than the interferant's mass due to the trapezoidal peak shape associated with a quadrupole mass spectrometer. Another interference which would be observed is the formation of a hydride ion. These interferences only cause an interference at 1 amu greater than the interferant's mass. It should also be remembered that these interferences are not necessarily linear and attempts should not be made to extrapolate the values to a particular data set. The table has been included for its informational content alone.

4.3.2 Isobaric molecular and doubly charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge. Table D-10, lists isobaric molecular-ion interferences which could affect the analytes. It should be noted that many of these interferences are extremely rare, but adverse effects on data quality could occur if the individual constituents occurred in the sample at sufficiently high concentrations. When the interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections to the data must be applied. Corrections for molecular-ion interferences may either be based upon the natural isotope ratios of the molecular ion or a determination of the actual amount of interference which occurs when the interferant is present.

If a correction for an oxide ion is used, the correction may be normalized to the extent of oxide formation of an appropriate internal standard previously demonstrated to form the same level of oxide as the interferant. This second type of correction has been reported for oxide ion corrections using ThO/Th for use on rare earth elements. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature.

4.3.3 Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Nebulization and transport processes are those in which the matrix component causes a change in surface tension or viscosity in a manner different from the standards used in performing calibration. Internal standards have been used to correct for these interferences. The interferences are primarily suppressions and are seen by the lighter elements more than the heavier elements. The effects are greater for matrix components with heavier atomic mass than for matrix components with lighter atomic mass. Changes in matrix composition therefore can cause

significant suppressions and enhancements. Dissolved-solid levels can contribute deposits on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2 percent (2,000 ppm) have been recommended to minimize solid deposition. Internal standards must be affected to the same degree as the analyte to demonstrate that they compensate for these interferences. A minimum of three internal standards, listed in Table D-7, bracketing the mass range, must be used. When the intensity level of an internal standard is less than 30 percent or greater than 125 pecent of the intensity of the first standard used during calibration, the sample must be reanalyzed after performing a five fold (1:4) dilution. The intensity levels of the internal standards for the CCB and CCV solutions must agree within ± 20 percent of the intensity level of the internal standard of the ICB solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples at no additional cost.

4.3.4 Memory interferences are effects which are dependant upon the relative concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer used, affect the extent of the memory interferences which are present. To verify that memory effects do not have an adverse impact on data quality, the memory test must be performed on the tuned and calibrated instrument before any analyses are performed. A multielement memory test solution containing levels of analytes as specified in Table D-6, is aspirated into the system for a normal sample exposure period. A blank solution is then introduced, noting the time when the uptake tube is switched to the blank solution. After the normal routine rinse time has elapsed, begin a routine analysis of the blank solution. Inspect the resulting data to see if any analytes are in excess of the MQL. If there are, reanalyze the blank to eliminate the possibility of actual blank contamination. A decreased value on the second analysis indicates a memory problem rather than blank contamination. If a memory problem does exist (see Exhibit E) for a given analyte, increase the rinse time until the system passes the memory test. If the increased rinse time is not feasible from a sample throughput standpoint, a hardware change may be necessary.

4.4 Apparatus and Materials

4.1 Inductively coupled plasma - mass spectrometer: System capable of 1 amu resolution from 6-253 amu with a data system that allows corrections for isobaric interferences and the application of the internal standard technique. Use of a mass-flow controller for the nebulizer argon and a peristaltic pump for the sample solution are recommended.

4.4.2 Argon gas supply: High-purity grade (99.99%)

4.5 OPERATIONAL REQUIREMENTS

4.5.1 System configuration: Because of the differences between various makes and models of satisfactory instruments, no detailed operating instruction can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, method quantitation limits (MQL's), precision, linear dynamic range and interference effects must be established for each analyte on a particular instrument. All reported measurements must be within the instrumental linear ranges. The analyst must maintain quality control data confirming instrument performance and analytical results.

IT IS THE RESPONSIBILITY OF THE ANALYST TO VERIFY THAT THE INSTRUMENT CONFIGURATION AND OPERATING CONDITIONS USED SATISFY THE ANALYTICAL REQUIREMENTS SET FORTH IN THIS DOCUMENT AND TO MAINTAIN QUALITY CONTROL DATA CONFIRMING INSTRUMENT PERFORMANCE AND ANALYTICAL RESULTS.

The data must include hardcopies and computer readable storage media which can be readily examined by an EPA audit team. The data must demonstrate defendable choices of instrument operating conditions which mimimize interferences such as oxides.

4.5.2 Precautions must be taken to protect the channel electron multiplier from high ion currents. The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this time period, response factors are constantly changing which invalidates the calibration curve, causes instability, and invalidates sample analyses. Samples run during such periods are required reruns at no additional cost to the Government under this contract.

4.5.3 Sensitivity, MQL's, precision, linear dynamic range, and interference effects must be established for each analyte on a particular instrument. These parameters must be determined for each configuration used if an instrument is equipped with dual detector hardware. All reported measurements must be within the instrumental linear dynamic ranges. All reported measurements from a less sensitive detector configuration must exceed five times the documented instrumental detection limit for that detector configuration. The analyst must maintain quality control data confirming instrument performance and analytical results.

4.6 REAGENTS AND STANDARDS

4.6.1 Acids used in the preparation of standards and for sample processing must be below the MQL's for the analytes of interest. Redistilled acids or ultra-pure acids are required for use with ICP-MS because of the high sensitivity of ICP-MS. Nitric acid at less than two percent (v/v) is preferred for ICP-MS to minimize damage to the interface and to minimize isobaric molecular-ion interferences with the analytes.

Many more molecular-ion interferences are observed on the analytes when hydrochloric and sulfuric acids are used, as demonstrated in Table D-10. Concentrations of antimony and silver above 300 µg/L require one percent (v/v) HCl for stability.

4.6.2 Internal standards must be used to monitor and correct for changes that occur from differences between standards and samples. This information must be clearly reported in the raw data. The changes for which internal standards correct, are primarily physical interferences. Internal standards must be present in all standards and samples at identical levels by mixing the internal standard to the solution being nebulized prior to the nebulizer. This may be accomplished by using a second channel of the peristaltic pump to add the internal standard to the uptake tube. If adding the solution to the uptake tube is not used then the internal standard must be added in two separate aliquots to the samples and standards to prevent the possibility of improperly spiking the internal standard levels. The double spiking ensures that misquantitation will not occur based upon a single internal standard spike. Double spiking may occur either by adding a constant volume of internal standard concentrate to identical volumes of the standards and prepared samples, or by diluting the internal standard to the appropriate level for its use in the analyses. One typical example is to measure out 10.0 mL of all standards and samples into individual containers, then 0.100 mL of a 10 mg/L solution of the internal standard is added to each of the containers. This adds identical amounts of the internal standard to each solution for analysis. The concentrations of the analyte levels in the standards do not have to be corrected for the dilution which occurs because the dilution is canceled out when corrections to the samples are made for their dilution.

4.6.2.1 Bismuth internal standard solution, stock, 1 mL = 100 µg Bi: Dissolve 0.1115 g Bi_2O_3 in a minimum amount of dilute HNO_3. Add 10 mL conc. HNO_3 and dilute to 1,000 mL with ASTM Type I water.

4.6.2.2 Holmium internal standard solution, stock, 1 mL = 100 µg Ho: Dissolve 0.1757 g $Ho_2(CO_3)_3 \cdot 5H_2O$ in 10 mL ASTM Type I water and 10 mL HNO_3. After dissolution is complete, warm the solution to degas. Add 10 mL conc. HNO_3 and dilute to 1,000 mL with ASTM Type I water.

4.6.2.3 Indium internal standard solution, stock, 1 mL = 100 µg In: Dissolve 0.1000 g indium metal in 10 mL conc. HNO_3. Dilute to 1,000 mL with ASTM Type I water.

4.6.2.4 Lithium internal standard solution, stock, 1 mL = 100 µg ^6Li: Dissolve 0.6312 g 95 atom percent enriched ^6Li, Li_2CO_3 in 10 mL of ASTM Type I water and 10 mL HNO_3. After dissolution is complete, warm the solution to degas. Add 10 mL conc. HNO_3 and dilute to 1,000 mL with ASTM Type I water.

4.6.2.5 Rhodium internal standard solution, stock, 1 mL = 100 μg Rh: Dissolve 0.3593 g $(NH_4)_3RhCl_6$ in 10 mL ASTM Type I water. Add 100 mL conc. HCl and dilute to 1,000 mL with ASTM Type I water.

4.6.2.6 Scandium internal standard solution, stock, 1 mL = 100 μg Sc: Dissolve 0.15343 g Sc_2O_3 in 10 mL (1:1) hot HNO_3. Add 5 mL conc. HNO_3 and dilute to 1,000 mL with ASTM Type I water.

4.6.2.7 Terbium internal standard solution, stock, 1 mL = 100 μg Tb: Dissolve 0.1828 g $Tb_2(CO_3)_3 \cdot 5H_2O$ in 10 mL (1:1) HNO_3. After dissolution is complete, warm the solution to degas. Add 5 mL conc. HNO_3 and dilute to 1,000 mL with ASTM Type I water.

4.6.2.8 Yttrium internal standard solution, stock, 1 mL = 100 μg Y: Dissolve 0.2316 g $Y_2(CO_3)_3 \cdot 3H_2O$ in 10 mL (1:1) HNO_3. Add 5 mL conc. HNO_3 and dilute to 1,000 mL with ASTM Type I water.

4.6.3 Mixed calibration standard solutions: Dilute the stock-standard solutions to levels in the linear range for the instrument in a solvent consisting of one percent (v/v) HNO_3 in ASTM Type I water along with the selected concentration of internal standards such that there is an appropriate internal standard element for each of the analytes (see Table D-7). Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to freshly acid-cleaned not previously used FEP fluorocarbon bottles for storage. Fresh mixed standards must be prepared as needed with the realization that concentrations can change on aging. Calibration standards must be initially verified using a quality control sample and monitored weekly for stability. Although not specifically required, some typical calibration standard combinations follow.

4.6.3.1 Mixed standard solution I: Manganese, beryllium, cadmium, lead, silver, barium, copper, cobalt, nickel and zinc.

4.6.3.2 Mixed standard solution II: Arsenic, chromium, thallium, and aluminum.

4.6.3.3 Mixed standard solution III: Antimony, vanadium, iron.

4.6.3.4 Mixed standard solution IV: Bismuth, holmium, indium, scandium, yttrium, and terbium.

4.6.3.5 Mixed standard solution V: Rhodium.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of ASTM Type I water and

warm the flask until the solution clears. Cool and dilute to 100 mL with ASTM Type I water. For this acid combination the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days.

4.6.4 Three types of blanks are required for the analysis. The calibration blank is used in establishing and monitoring the calibration curve, the preparation blank is used to monitor the blank standard solution (SO), ICB, and CCB for possible contamination resulting from the sample preparation procedure, and the rinse blank is used to flush the system between all samples and standards.

4.6.4.1 The calibration blank consists of one percent HNO_3 (v/v) in ASTM Type I water along with the selected concentration of internal standards such that there is an appropriate internal standard element for each of the analytes (see Table D-7).

4.6.4.2 The preparation blank must contain all the reagents in the same volumes as used in processing the samples. The preparation blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solutions used for analysis (see Exhibit E).

4.6.4.3 The rinse blank consists of two percent HNO_3 (v/v) in ASTM Type I water. Prepare a sufficient quantity to flush the system between standards and samples.

4.6.5 The Interference Check Solution(s) (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. The ICS solution is detailed in Table 3, Exhibit C. The chloride concentration provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Since the natural abundance of ^{35}Cl at 75.8 percent is 3.13 times the ^{37}Cl abundance of 24.2 percent, the ion corrections can be calculated with adjustments for isobaric contributions. Iron is used to demonstrate adequate resolution of the spectrometer on manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement scheme to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

4.6.5.1 Stock solutions for preparing ICS solutions A and AB may be provided if available from EPA. Otherwise, refer to Table 3, Exhibit C. They must be diluted before use according to the instruction provided. The prepared ICS solutions A and AB must be prepared weekly.

4.6.5.2 Mixed ICS solution I may be prepared by adding 13.903 g $Al(NO_3)_3 \cdot 9H_2O$, 2.498 g $CaCO_3$ dried at 180°C for 1 hour before weighing, 1.000 g Fe, 1.658 g MgO, 2.305 g Na_2CO_3, and 1.767 g K_2CO_3 to 25 mL of ASTM Type I water. Slowly add 40 mL of (1:1) HNO_3. After dissolution is complete, warm the solution to degas. Cool and dilute to 1,000 mL with ASTM Type I water.

4.6.5.3 Mixed ICS solution II may be prepared by slowly adding 7.444 g 85 percent H_3PO_4, 6.373 g 96 percent H_2SO_4, 40.024 g 37 percent HCl, and 10.664 g $C_6O_7H_8$ to 100 mL of ASTM Type I water. Dilute to 1,000 mL with ASTM Type I water.

4.6.5.4 Mixed ICS solution III may be prepared by adding 5 mL each of arsenic stock solution, chromium stock solution, copper stock solution, and zinc stock solution, 10 mL each of cobalt stock solution, nickel stock solution, and vanadium stock solution, and 2.5 mL of cadmium stock solution. Dilute to 100 mL with two percent HNO_3.

4.6.5.5 ICS A may be prepared by adding 10 mL of mixed ICS solution I, 10 mL each of titanium stock solution, and molybdenum stock solution, and 5 mL of mixed ICS solution II. Dilute to 100 mL with ASTM Type I water. ICS solution A must be prepared fresh weekly.

4.6.5.6 ICS AB may be prepared by adding 10 mL of mixed ICS solution I, 10 mL each of titanium stock solution and molybdenum stock solution, 5 mL of mixed ICS solution II, and 2 mL of mixed ICS solution III. Dilute to 100 mL with ASTM Type I water. ICS solution AB must be prepared fresh weekly.

4.7 QUALITY CONTROL

To obtain analyte data of known quality, it is necessary to measure for more than the analytes of interest in order to know the required interference corrections. If the concentrations of interference sources (such a C, Cl, Mo, Zr, W) are below the levels that show an effect on the analyte level, uncorrected equations may be used provided all QA criteria are met. It should be noted that monitoring the interference sources does not necessarily require monitoring the interference itself, but that a molecular species may be monitored to indicate the presence of the interference. When corrected equations are used all QA criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose oxygen, hydroxyl, chloride, nitrogen, carbon and sulfur molecular ions which could impact the analytes of interest. When an interference source is present, the sample elements impacted must be flagged to indicate (a) the percentage interference correction applied to the data or (b) an uncorrected interference. The isotope proportions for an element or molecular-ion cluster provide information useful for quality assurance. These tests will enable the analyst to detect positive or negative interferences that distort the accuracy of the reported values.

4.7.1 Instrument Calibration

4.7.1.1 Summary

Prior to the analysis of samples and required QC, each ICP-MS system shall be initially calibrated to determine instrument sensitivity.

4.7.1.2 Frequency

Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up.

4.7.1.3 Procedure

Calibration standards shall be prepared using the same type of matrix and at the same concentration as the preparation blank following sample preparation.

Calibrate according to instrument manufacturer's recommended procedures using at least two standards, one being a blank.

Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample.

4.7.1.4 Calculations

$$\% \ Recovery = \frac{Found \ Concentration}{True \ Concentration} \times 100 \qquad \text{Eq. D-16}$$

4.7.1.5 Technical Acceptance Criteria

Recovery for the highest mixed calibration standard shall be within ± five percent of the true value (i.e., 95-105 percent).

4.7.1.6 Corrective Action

Follow instrument manufacturer's recommendations to correct the problem. Also, baseline correction is acceptable as long as it is performed after every sample or after the continuing calibration verification and blank check; resloping is acceptable as long as it is immediately preceded and immediately followed by a CCV and a CCB.

4.7.1.7 Documentation

The instrument standardized data and time shall be included in the raw data. The final concentration should be in µg/L.

4.7.2 Initial Calibration Verification

4.7.2.1 Summary

Immediately after the ICP-MS system has been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of EPA Initial Calibration Verification Solution(s) (ICV) at each mass used for analysis.

4.7.2.2 Frequency

Each time the instrument is calibrated, the ICV shall be run immediately following the calibration, before any samples are analyzed.

4.7.2.3 Procedure

If the ICV solution(s) are not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the linear range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

4.7.2.4 Calculations

$$\% \ Recovery = \frac{Found \ Concentration}{True \ Concentration} \times 100 \qquad \text{Eq. D-17}$$

4.7.2.5 Technical Acceptance Criteria

Recovery for the ICV shall be within ± 10 percent of the true value (i.e., 90-110 percent).

4.7.2.6 Corrective Action

When recoveries of the ICV exceed the technical acceptance criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

4.7.2.7 Documentation

Report the ICV found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM II-AAIN.

4.7.3 Continuing Calibration Verification

4.7.3.1 Summary

To ensure calibration accuracy during an analysis run, a continuing calibration verification solution (CCV) is analyzed and reported for every mass used for the analysis of each analyte.

4.7.3.2 Frequency

The CCV is run at a frequency of 10 percent or every two hours during an analysis run, whichever is more frequent.

The CCV is also run after the last analytical sample in the analysis run.

4.7.3.3 Procedure

The same CCV shall be used throughout all analysis runs for a Case of samples received. The analyte concentrations in the continuing calibration standard shall be an EPA solution or a Contractor prepared standard solution and should be at or near ± 10 percent of the mid-range levels of the calibration curve.

Each CCV analyzed shall reflect the conditions of analysis for all of the associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses and other related operations that may affect the CCV measured result shall not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it as well as the difference in time between the CCV and the analytical sample immediately preceding it shall not exceed the smallest difference in time between any two consecutive analytical samples associated with the CCV.

4.7.3.4 Calculations

$$\%\ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-18}$$

4.7.3.5 Technical Acceptance Criteria

Recovery for the CCV shall be within ± 10 percent of the true value (i.e., 90-110 percent).

4.7.3.6 Corrective Action

When recoveries of the CCV exceed the technical acceptance criteria, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration reverified, and the preceding 10 analytical samples reanalyzed (or all analytical samples since the last compliant CCV).

4.7.3.7 Documentation

Report the CCV found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM II-AAIN.

4.7.4 **CRQL Standard**

4.7.4.1 Summary

To verify linearity near the CRQL, the Contractor shall analyze an ICP-MS standard at two times the MQL or two times CRQL, whichever is greater. This standard shall be run for every mass used for analysis.

4.7.4.2 Frequency

The CRQL standard shall be run at the beginning and end of each sample analysis run, or a minimum of twice per eight hours, whichever is more frequent.

4.7.4.3 Procedure

The CRQL standard shall not be run before the ICV solution.

4.7.4.4 Calculations

$$\% \text{ Recovery} = \frac{\text{Found Concentration}}{\text{True Concentration}} \times 100 \qquad \text{Eq. D-19}$$

4.7.4.5 Technical Acceptance Criteria

Recovery of the CRQL standard shall be within ± 15 percent of the true value for each mass used for analysis.

4.7.4.6 Corrective Action

If the CRQL standard does not fall within the control limit, the analysis shall be terminated, the problem corrected and the analytical samples since the last compliant CRQL standard reanalyzed.

4.7.4.7 Documentation

Report the CRQL standards found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM III-AAIN.

4.7.5 Linear Range Analysis (Quarterly)

4.7.5.1 Summary

The concentration range over which the ICP-MS calibration curve remains linear must be determined and any values above this linear range shall be diluted and reanalyzed.

4.7.5.2 Frequency

For all ICP-MS analyses, a linear range verification check standard shall be analyzed and reported quarterly (every 3 calendar months) for each element on the target analyte list (Table 2, Exhibit C). The standard shall be analyzed during a routine analytical run performed under this contract. This standard shall be run for all masses used for each analyte reported by ICP-MS.

4.7.5.3 Procedure

The standard shall be analyzed as though it were a separate analytical sample (i.e., each measurement shall be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples).

4.7.5.4 Calculations

$$\%\ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-20}$$

4.7.5.5 Technical Acceptance Criteria

Recovery for the linear range standard shall be within ± 5 percent of the true value (i.e., 95-105 percent).

4.7.5.6 Corrective Action

If the recovery of the linear range standard does not meet the technical acceptance criteria, then the analysis shall be terminated and successive dilutions of the standard shall be reanalyzed until the control limits are met. The concentration of this standard that meets the control limits is the upper limit of the instrument linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

4.7.5.7 Documentation

Report the linear range standards found concentration (in µg/L), true concentration (in µg/L) and percent recovery for each analyte on FORM III-AAIN.

4.7.6 Initial Calibration Blank

4.7.6.1 Summary

To verify that the ICP-MS system is not contaminated, an initial calibration blank (ICB) shall be analyzed after calibration.

4.7.6.2 Frequency

The ICB shall be analyzed each time the system is calibrated and immediately after the ICV.

4.7.6.3 Procedure

If the absolute value of the ICB is greater than the MQL, the result shall be reported.

4.7.6.4 Calculations

Not applicable.

4.7.6.5 Technical Acceptance Criteria

The absolute value of the ICB must be less than the CRQL.

4.7.6.6 Corrective Action

When the ICB concentration does not meet the technical acceptance criteria, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the ICB.

4.7.6.7 Documentation

Report the ICB values in µg/L on FORM IV-AAIN.

7.7 Continuing Calibration Blanks

4.7.7.1 Summary

To ensure that the system is not contaminated during the analysis run, continuing calibration blanks (CCB) are analyzed.

4.7.7.2 Frequency

Analyze the CCB at a frequency of 10 percent or every two hours, whichever is more frequent.

Analyze the CCB after every CCV.

4.7.7.3 Procedure

A CCB shall be run after the last CCV in the analysis run. If the absolute value of the CCB is greater than the MQL, the result shall be reported.

4.7.7.4 Calculations

Not applicable.

4.7.7.5 Technical Acceptance Criteria

The absolute value of the CCB must be less than the CRQL.

4.7.7.6 Corrective Action

When the CCB concentration does not meet the technical acceptance criteria, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the preceding 10 analytical samples (or all analytical samples since the last compliant CCB).

4.7.7.7 Documentation

Report the CCB values in µg/L on FORM IV-AAIN.

4.7.8 Preparation Blanks

4.7.8.1 Summary

To ensure against contamination during sample preparation, a preparation blank (PB) is analyzed.

4.7.8.2 Frequency

At least one PB must be prepared and analyzed with every SDG or with each batch of samples digested, whichever is more frequent.

4.7.8.3 Procedure

The PB shall consist of ASTM Type I water processed through each sample preparation and analysis procedure step.

The first batch of samples in a SDG shall be assigned to PB one, the second batch of samples to PB two, etc.

4.7.8.4 Calculations

Not applicable.

4.7.8.5 Technical Acceptance Criteria

The absolute value of the PB must be less than the CRQL.

4.7.8.6 Corrective Action

If the absolute value of the concentration of the PB is less than or equal to the CRQL, no corrective action is required.

If any analyte concentration in the PB is above the CRQL, the lowest concentration of the analyte in the associated samples must be 10 times the PB concentration. Otherwise, all samples associated with the PB and with the analyte's concentration less than 10 times the PB concentration and above the CRQL shall be redigested and reanalyzed for that analyte. The sample concentration is not to be corrected for the PB value.

If an analyte concentration in the PB is below the negative CRQL, then all samples reported below 10 times CRQL associated with the PB shall be redigested and reanalyzed.

4.7.8.7 Documentation

The values for the PB shall be recorded in µg/L on FORM IV-AAIN.

7.9 ICP Interference Check Sample

4.7.9.1 Summary

To verify interelement and background correction factors, an ICP Interference Check Sample (ICS) is analyzed.

4.7.9.2 Frequency

Analyze the ICS at the beginning and end of each analysis run or a minimum of twice per eight hour working shift, whichever is more frequent, but not before the ICV.

4.7.9.3 Procedure

The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of

the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all masses used for each analyte reported by ICP-MS.

The ICS shall be obtained from EPA (EMSL/LV) if available and analyzed according to the instructions supplied with the ICS.

If the ICP ICS is not available from EPA, an independent ICP ICS shall be prepared with interferent and analyte concentrations at the levels specified in Table 3, Exhbit C. The mean value and standard deviation shall be established by initially analyzing the ICS at least five times repetitively for each analyte.

If true values for analytes contained in the ICS and analyzed by ICP-MS are not supplied with the ICS, the mean shall be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination shall be made during an analytical run where the results for the previously supplied EPA ICS met all contract specifications. Additionally, the result of this initial mean determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

4.7.9.4 Calculations

$$\% \textit{ Recovery} = \frac{\textit{Found Concentration}}{\textit{True Concentration}} \times 100 \qquad \text{Eq. D-21}$$

4.7.9.5 Technical Acceptance Criteria

Recovery for the ICS shall be within ± 20 percent of the true value (i.e., 80-120 percent).

4.7.9.6 Corrective Action

If the ICS recoveries does not meet the technical acceptance criteria, terminate the analysis, correct the problem, recalibrate the instrument, verify the calibration, and reanalyze all of the analytical samples since the last compliant ICS was analyzed.

4.7.9.7 Documentation

Report the ICS found concentration (µg/L), true concentration (µg/L), percent recovery, mean and standard deviation on FORM V-AAIN.

The mean and standard deviation shall be reported in the raw data.

4.7.10 Spike Sample Analysis

4.7.10.1 Summary

To provide information about the effect of the sample matrix on the digestion, a known amount of analyte is added (spiked) into a sample.

4.7.10.2 Frequency

At least one spike sample analysis shall be performed on each group of samples for each SDG. EPA may require additional spike sample analysis upon special request by the Project Officer, for which the Contractor will be paid.

If two analytical methods are used to obtain the reported values for the same analyte within a SDG (e.g., ICP-MS and GFAA), then spike samples shall be run by each method used.

4.7.10.3 Procedure

The spike is added before the sample preparation (i.e., prior to digestion) at concentration levels in the Spike Sample solution as indicated in Table D-3.

Samples identified as field blanks cannot be used for spiked sample analysis. EPA may require that a specific sample be used for the spike sample analysis. In the instance where there is more than one spike sample per SDG and one spike sample recovery is not within contract criteria, flag all the samples of the same matrix, level, and method in the SDG.

4.7.10.4 Calculations

$$\% \ Recovery = \frac{SSR - SR}{SA} \times 100 \qquad \text{Eq. D/IN-22}$$

where:

SSR = Spiked Sample Result;
SR = Sample Result; and
SA = Spike Added.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see part 4.7.11, Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery.

When the sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating percent recovery.

4.7.10.5 Technical Acceptance Criteria

Recovery for the spike should be within ± 25 percent of the spiked amount (i.e., 75-125 percent).

4.7.10.6 Corrective Action

If the spike recovery is not within the limits of 75-125 percent, the data of all samples received associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N" on FORMs I-AAIN and VI-AAIN.

An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125 percent recovery criteria.

4.7.10.7 Documentation

Report the spiked sample results, sample results, spike added and percent recovery for the spike sample analysis on FORM VI-AAIN.

The units for reporting spike sample results will be in µg/L.

7.11 Duplicate Sample Analysis

4.7.11.1 Summary

Duplicate aliquots of a sample are carried through the preparation and analysis steps to provide information about the precision of the analytical methods as well as matrix effects.

4.7.11.2 Frequency

At least one duplicate sample analysis shall be performed on each group of samples for each SDG. EPA may require additional duplicate sample analysis upon special request by the Project Officer, for which the Contractor will be paid.

If two analytical methods are used to obtain the reported values for the same analyte within a SDG (e.g., ICP-MS and GFAA), then duplicate samples shall be run by each method used.

4.7.11.3 Procedure

Samples identified as field blanks cannot be used for duplicate sample analysis.

EPA may require that a specific sample be used for the duplicate sample analysis.

In the instance where there is more than one duplicate sample per SDG and one duplicate result is not within contract criteria, flag all the samples of the SDG.

Duplicate sample analyses are required for calculations of relative percent difference.

4.7.11.4 Calculations

$$RPD = \frac{|S - D|}{\frac{S + D}{2}} \times 100 \qquad \text{Eq. D/IN-23}$$

where:

RPD = Relative Percent Difference;
S = First Sample Value (original); and
D = Second Sample Value (duplicate).

Duplicates cannot be averaged for reporting on FORM I-AAIN.

4.7.11.5 Technical Acceptance Criteria

A control limit of ± 20 percent for RPD shall be used for original and duplicate sample values greater than or equal to five times CRQL (Exhibit C). A control limit of ± CRQL shall be used for sample values less than five times CRQL.

If one result is above the five times CRQL level and the other is below, use the ± CRQL criteria.

If both sample values are less than the MQL, the RPD is not calculated.

Specific control limits for each analyte will be added to FORM IX-AAIN at a later date, based on precision results.

4.7.11.6 Corrective Action

If the duplicate sample results are outside the control limits, flag with an asterisk all the data for samples received associated with that duplicate sample.

4.7.11.7 Documentation

The results in µg/L of the duplicate sample analyses shall be reported on FORM VIII-AAIN.

The absolute value of the control limit (CROL) shall be entered in the "CONTROL LIMIT" column on FORM VIII-AAIN.

4.7.12 Laboratory Control Samples

4.7.12.1 Summary

A laboratory control sample (LCS) is digested and analyzed to ensure against analyte loss in the sample preparation.

4.7.12.2 Frequency

One LCS shall be prepared and analyzed for every group of samples in a SDG, or for each batch of samples, whichever is more frequent.

4.7.12.3 Procedure

A LCS shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

The LCS solution shall be obtained from EPA. (If unavailable, other EPA Quality Assurance Check samples or other certified materials may be used.)

4.7.12.4 Calculations

$$\% \text{ Recovery} = \frac{\text{Found Concentration}}{\text{True Concentration}} \times 100 \qquad \text{Eq. D/IN-24}$$

4.7.12.5 Technical Acceptance Criteria

Recovery for the LCS shall be within ± 20 percent of the true value (i.e., 80-120 percent), with the exception of Ag and Sb.

4.7.12.6 Corrective Action

If the percent recovery for the LCS falls outside the technical acceptance criteria, then the analyses shall be terminated, the problem corrected, and the samples associated with that LCS reprepared and reanalyzed.

4.7.12.7 Documentation

Report the LCS found concentration (µg/L), true concentration (in µg/L), and percent recovery on FORM IX-AAIN.

4.7.13 Performance Evaluation Sample (PES)

4.7.13.1 Summary

The performance evaluation sample (PES) assists the Agency in monitoring the laboratory performance for analyte identification and quantification.

4.7.13.2 Frequency

One PES shall be delivered, prepared, and analyzed for every group of samples in a SDG, or for each batch of samples, whichever is more frequent.

4.7.13.3 Procedure

A PES shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

The PES solution shall be obtained from EPA. (If unavailable, the contractor must contact the Agency and SMO for instructions.)

4.7.13.4 Calculations

Not applicable.

4.7.13.5 Technical Acceptance Criteria

Recovery for the PES shall be within 75 percent of the true value of analyte.

4.7.13.6 Corrective Action

If the percent recovery for the PES falls below 75 percent, the Agency may take, but is not limited to the following actions:

- Show Cause and/or Cure Notice for unacceptable performance;
- Reduction of the number of samples shipped to the laboratory;
- Suspension of sample shipment;
- A site visit;

- A full data audit; or
- Require the laboratory to analyze remedial PES.

In addition to the above cited actions, the laboratory shall also provide the Technical and Administrative Project Officers a written correction action report on every unacceptable (less than 75 percent) PES score within seven days of written or verbal request by Agency personnel.

4.7.13.7 Documentation

Report the PES results on Form I-AAIN (in µg/L) for all analytes.

4.7.14 Internal Standards for ICP-MS

4.7.14.1 Summary

A minimum of three internal standards, bracketing the mass range, shall be used to check and correct the ICP-MS system for the presence of physical interferences.

4.7.14.2 Frequency

Analyze with each sample, duplicate, spike analysis, PES, CCV, and CCB.

4.7.14.3 Procedure

Each sample, blank, and QA/QC sample must be spiked with the internal standard and shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

4.7.14.4 Calculations

$$\%D = \frac{|(SO_I) - (S_I)|}{SO_I} \times 100 \qquad \text{Eq. D/IN-25}$$

where:

SO_I = Intensity of the internal standard in the blank calibration standard;
S_I = Intensity of internal standard in the EPA Sample No.; and
%D = Percent difference.

4.7.14.5 Technical Acceptance Criteria

The intensity level of an internal standard for each sample, duplicate, spike sample, and PES shall agree within ± 50 percent of

the intensity level of the internal standard of the initial calibration blank standard solution (SO_I). MQLs shall be ≤ the CRQLs in Exhibit C.

The intensity levels of the internal standard for the ICV, ICB, CCV, and CCB solutions must agree within ± 20 percent of the instensity level of the internal standard of the initial calibration blank solution (SO_I).

4.7.14.6 Corrective Action

If the internal standard intensity level does not agree ± 50 percent of the sample, the sample must be analyzed after performing a five fold (1:4) dilution and reanalyzed. If the intensity level percent difference (%D) remains greater than 50 percent, a physical interference must be suspected, and the data on FORM XV-AAIN must be flagged with an "E." The analytes affected by the interferences must be listed in the comment section on the appropriate FORM I-AAIN, VII-AAIN, and VIII-AAIN.

If the internal standard intensity level does not agree within ± 20 percent for the ICV and ICB, the analysis shall be terminated, the problem corrected, and a new analytical run shall be started.

If the internal standard intensity level does not agree within ± 20 percent for the CCV and CCB, terminated the analysis, correct the problem, and reanalyze the CCV/CCB only once. If the first CCV/CCB reanalysis yields a %D value within the control limits, then the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification may be reanalyzed for the analytes affected. Otherwise, the instrument shall be recalibrated, the calibration verified and the affected analytical samples rerun in the context of a new run.

4.7.14.7 Documentation

The internal standard percent difference (%D) shall be reported for each ICP-MS analysis on FORM XV-AAIN.

4.7.15 **Method Quantitation Limits**

4.7.15.1 Summary

Prior to sample analysis, the method quantitation limit (MQL) shall be determined for each instrument that will be used.

4.7.15.2 Frequency

MQLs shall be determined within 30 days of the start of the contract and at least quarterly (every three calendar months) until the end of the contract.

4.7.15.3 Procedure

MQLs (in $\mu g/L$) shall be determined by multiplying by three, the average of the standard deviations (σ_{n-1}) obtained on three nonconsecutive days from the consecutive analysis of seven different PB extracts. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). MQLs shall be determined and reported for each mass and mass number used in the analysis of the samples.

The quarterly determined MQL for an instrument shall always be used as the MQL for that instrument during that quarter. If the instrument is adjusted in anyway that may affect the MQL, the MQL for that instrument shall be redetermined and the results submitted for use as the established MQL for that instrument for the remainder of the quarter.

4.7.15.4 Calculations

$$MQL = 3 \times (\sigma_{n-1}) \qquad \text{Eq. D/IN-26}$$

4.7.15.5 Technical Acceptance Criteria

The MQLs shall be ≤ the CRQLs in Table 2, Exhibit C.

4.7.15.6 Corrective Action

If an instrument's MQL exceeds the CRQL for an analyte, that instrument cannot be used in this document unless the analyte concentration is greater than or equal to five times the reported MQL.

4.7.15.7 Documentation

MQLs shall be submitted with each data package and reported on FORM XI-AAIN for each instrument used. If multiple instruments are used for the analysis of an analyte within a SDG, the highest MQL for the analyte shall be used for reporting concentration values for that SDG.

4.7.16 Interelement Correction Factors

4.7.16.1 Summary

To ensure against spectral and isobaric interferences, interelement correction factors are determined for all masses used for each analyte reported by ICP-MS.

4.7.16.2 Frequency

Before any field samples are analyzed under this contract, the ICP interelement correction factors shall be determined within three months prior to the start of contract analyses and at least annually thereafter.

4.7.16.3 Procedure

Correction factors shall be determined under the same instrument conditions used for sample analysis. If the instrument was adjusted in any way that may affect the interelement correction factors, the factors shall be redetermined and the results submitted for use.

Follow the instrument manufacturer's recommendations for applying interelement correction factors.

4.7.16.4 Calculations

Not applicable.

4.7.16.5 Technical Acceptance Criteria

Not applicable.

4.7.16.6 Corrective Action

Not applicable.

4.7.16.7 Documentation

Results from interelement correction factors determination shall be reported on FORM XI-AAIN for all ICP-MS parameters.

4.8 PROCEDURE

4.8.1 Initiate appropriate operating configuration of instrument computer.

4.8.2 Set up the instrument with the proper operating parameters. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. Instrument calibration is verified by running the tuning solution (Table D-5) at least four times with relative standard deviations of less than 10 percent for the analytes contained in the tuning solution.

4.8.3 Conduct mass calibration and resolution checks using the tuning solution (100 ppb of the elements Li, Co, In, and Tl). The intensities on the forms in Exhibit B (see Table D-6) for the response factor criteria

are recommendations which might be helpful when setting up the instruments but are not required criteria. The mass calibration must meet the criteria specified in Table D-6, if mass calibration exceed those criteria then the mass calibration must be adjusted to the correct values. The resolution must also be verified to be less than 1.0 amu full width at 10 percent peak height. To verify, the tuning solution must be analyzed at the beginning and end of each eight hour shift, and pass the tuning criteria.

4.8.4 Calibration and Sample Analysis

4.8.4.1 Calibrate the instrument for the analytes of interest using the calibration blank and at least a single standard according to the manufacturer's recommended procedure for each detector configuration which will be used in analysis. Flush the system with the rinse blank between each standard solution. Report each integration during the calibration and sample analysis and use the average of the multiple integrations for both standardization and sample analysis. A minimum of two replicate integrations are required for both calibration and sample analysis. The raw data must include the concentrations of elements in each integration as well as the average. Additionally, if different detector configurations are used, the raw data must indicate which detector configuration is being used.

NOTE: Some elements (such as Hg, W, and Mo) require extended flushing times which need to be determined for each instrumental system. Run the memory tests on the solution in Table D-7 to verify that memory problems will not affect the data quality.

4.8.5 As a minimum, all masses which would affect data quality must be monitored to determine potential effects from matrix components on the analyte peaks. This information is to be used to assess data quality and as a minimum must include the masses which are boldfaced and underlined, listed in Table D-12, for each element. These masses must be monitored simultaneously in a separate scan or at the time quantification occurs.

4.8.6 Flush the system with the rinse blank solution for a least 30 seconds before the analysis of each sample. Aspirate each sample for at least 30 seconds before collecting data.

4.8.7 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte.

4.9 CALCULATIONS

4.9.1 If dilutions were performed, the appropriate corrections must be applied to the sample values.

4.9.2 To obtain the analyte air concentration (in $\mu g/m^3$), multiply the interference-corrected analyte values (in $\mu g/L$) by nine times the

appropriate volume (in liters) used in the extraction and divide by the volume of air (in standard m^3) sampled and the number of 1" x 8" strips digested for the sample. The factor "9" represents the total area (63 in^2) of the Hi-Vol filter exposed to the air stream divided by the exposed area (7 in^2) in one 1" x 8" strip.

$$\text{Analyte Conc. In air, } \mu g/m^3 = \frac{(9)\left(\text{Interference-Corrected Value, } \mu g/L\right)\left(\text{Extract Volume, } L\right)}{\text{no. of strips digested} \times \text{air volume sampled, std. } m^3} \qquad \text{Eq. D-27}$$

4.10 DOCUMENTATION

4.10.1 Report the air concentration values in $\mu g/m^3$ and the interference-corrected analyte value in $\mu g/L$ om FORM I-AAIN.

SECTION 5

SAMPLE ANALYSIS BY GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) SPECTROMETRY

5.1 INTRODUCTION

5.1.1 GFAA analysis procedures are provided to achieve lower quantitation limits (where required) for the analysis of metal analytes listed in Table 1 the Target Analyte List in Exhibit C.

5.1.2 Because of the differences among various makes and models of satisfactory instruments, no detailed instrument operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of that instrument.

5.2 SUMMARY OF METHOD

5.2.1 Using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in a graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state element in the vapor. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from a hollow cathode lamp or an electrodless furnace lamp, and a photosensitive device measures the attenuated transmitted radiation.

5.3 INTERFERENCES

5.3.1 The composition of the sample matrix can have a major effect on the analysis. By modifying the sample matrix, either to remove interferences or to stabilize the analyte, interferences can be minimized. Examples are the addition of ammonium nitrate to remove alkali chlorides and the addition of ammonium phosphate to retain cadmium.

5.3.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Therefore the use of background correction is required for all furnace analysis.

5.3.3 Continuum background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, choose an alternate wavelength, chemically separate the

analyte from the interferant, or use an alternate form of background correction (e.g., Zeeman background correction).

5.3.4 Interferences from a smoke producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken to prevent loss of analyte.

5.4 APPARATUS

5.4.1 Atomic absorption spectrophotometer: Single or dual channel, single or double beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190- to 800-nm, background correction, and provisions for interfacing with a recording device.

5.4.2 Graphite furnace: Any furnace device capable of reaching the specified temperatures is satisfactory.

5.4.3 Operational requirements: Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrument detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte on that particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements set forth in this document and to maintain quality control data confirming instrument performance and analytical results.

5.5 Reagents and Standards

5.5.1 Matrix matching with the samples is mandatory for all blanks, standards, and quality control samples, to avoid inaccurate concentration values due to possible standard curve deviations.

5.5.2 Calibration standards are prepared by diluting stock metal solutions at the time of analysis and are discarded after use. Prepare at least three calibration standards in graduated amounts in the appropriate range by combining an appropriate volume of stock solution in a volumetric flask. The calibration standards must be prepared using the same type of acid or combination of acids at the same concentration.

5.5.3 Two types of blanks are required for GFAA analysis; the calibration blank is used in establishing the analytical curve while the preparation blank is used to indicate possible contamination resulting from the reagents or apparatus used in the sample processing.

5.6 QUALITY CONTROL

5.6.1 Instrument Calibration

5.6.1.1 Summary

Prior to the analysis of samples and required QC, each GFAA system shall be initially calibrated to determine instrument sensitivity.

5.6.1.2 Frequency

Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up.

5.6.1.3 Procedure

Calibration standards shall be prepared by diluting the stock solutions at the time of analysis, and are discarded after use.

Calibration standards shall be prepared using the same type of acid or combination of acids, and at the same concentration as will result in the samples following sample preparation.

The instrument shall be calibrated according to instrument manufacturer's recommended procedures using at least four calibration points. Beginning with a calibration blank and working towards the highest standard, run at least three other standards. One calibration standard shall be a blank, and another shall be at the CRQL.

Baseline correction is acceptable as long as it is performed after each and every sample, or after the CCV and CCB, respectively.

Resloping is acceptable as long as it is immediately preceded and immediately followed by a CCV and CCB.

5.6.1.4 Calculations

Not applicable.

5.6.1.5 Technical Acceptance Criteria

Not applicable.

5.6.1.6 Corrective Action

Not applicable.

5.6.1.7 Documentation

The instrument standardization date and time shall be included in the raw data. Concentrations shall be in $\mu g/L$.

5.6.2 Initial Calibration Verification

5.6.2.1 Summary

Immediately after the GFAA system has been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of EPA Initial Calibration Verification Solutions(s) (ICV) at each wavelength used for analysis.

5.6.2.2 Frequency

Each time the instrument is calibrated, the ICV shall be run immediately following the calibration and before any samples are analyzed.

5.6.2.3 Procedure

If the ICV solution(s) are not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

5.6.2.4 Calculations

$$\% \ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-28}$$

5.6.2.5 Technical Acceptance Criteria

Recovery for the ICV shall be within ± 10 percent of the true value (i.e., 90-110 percent).

5.6.2.6 Corrective Action

When recoveries of the ICV exceed the technical acceptance criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

5.6.2.7 Documentation

Report the ICV found concentration (in µg/L), true concentration (in µg/L), and percent recovery on FORM II-AAIN.

5.6.3 Continuing Calibration Verification

5.6.3.1 Summary

To ensure calibration accuracy during an analysis run, a continuing calibration verification solution (CCV) is analyzed and reported for every wavelength used for the analysis of each analyte.

5.6.3.2 Frequency

The CCV is run at a frequency of 10 percent or every two hours during an analysis run, whichever is more frequent.

The CCV is also run after the last analytical sample in the analysis run.

5.6.3.3 Procedure

The CCV shall contain the analytes at a concentration at or near the mid-range of the calibration curve.

The same CCV shall be used throughout all analysis runs for a Case of samples received.

If the ICV solution(s) are not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

Each CCV analyzed shall reflect the conditions of analysis for all the associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses and other related operations that may affect the CCV measured result shall not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it as well as the difference in time between the CCV and the analytical sample immediately preceding it shall not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

5.6.3.4 Calculations

$$\%\ Recovery = \frac{Found\ Concentration}{True\ Concentration} \times 100 \qquad \text{Eq. D-29}$$

5.6.3.5 Technical Acceptance Criteria

Recovery for the CCV shall be within ± 10 percent of the true value (i.e., 90-110 percent).

5.6.3.6 Corrective Action

When recoveries of the CCV exceed the technical acceptance criteria, the analysis shall be stopped, the problem corrected, the

instrument recalibrated, the calibration reverified, and the preceding 10 analytical samples reanalyzed (or all analytical samples since the last compliant CCV).

5.6.3.7 Documentation

Report the CCV found concentration (in µg/L), true concentration (in µg/L), and percent recovery on FORM II-AAIN.

5.6.4 CRQL Standard

5.6.4.1 Summary

To verify linearity near the CRQL, the Contractor shall analyze a GFAA standard at two times the CRQL or two times the MQL, whichever is greater. This standard shall be run for every wavelength used for analysis.

5.6.4.2 Frequency

The CRQL standard shall be run at the beginning and end of each sample analysis run, or a minimum of twice per eight hours, whichever is more frequent.

5.6.4.3 Procedure

The CRQL standard is not to be run before the ICV solution.

5.6.4.4 Calculations

$$\% \text{ Recovery} = \frac{\text{Found Concentration}}{\text{True Concentration}} \times 100 \qquad \text{Eq. D-30}$$

5.6.4.5 Technical Acceptance Criteria

Recovery of the CRQL standard shall be within ± 15 percent of the true value for each wavelength used for analysis.

5.6.4.6 Corrective Action

If the CRQL standard recovery does not fall within the technical acceptance criteria, terminate the analysis, correct the problem and reanalyze all analytical samples since the last compliant CRQL standard.

5.6.4.7 Documentation

Report the CRQL standard's found concentration (in µg/L), true concentration (in µg/L), and percent recovery on FORM III-AAIN.

5.6.5 Initial Calibration Blank

5.6.5.1 Summary

To verify that the GFAA system is not contaminated, an initial calibration blank (ICB) shall be analyzed after calibration.

5.6.5.2 Frequency

The ICB shall be analyzed each time the system is calibrated and immediately after the ICV.

5.6.5.3 Procedure

If the absolute value of the ICB is greater than the MQL, the result shall be reported.

5.6.5.4 Calculations

Not applicable.

5.6.5.5 Technical Acceptance Criteria

The absolute value of the ICB must be less than the CRQL.

5.6.5.6 Corrective Action

When the ICB concentration does not meet the technical acceptance criteria, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the ICB.

5.6.5.7 Documentation

Report the ICB values in µg/L on FORM IV-AAIN.

5.6.6 Continuing Calibration Blanks

5.6.6.1 Summary

To ensure that the system is not contaminated during the analysis run, continuing calibration blanks (CCB) are analyzed.

5.6.6.2 Frequency

Analyze the CCB at a frequency of 10 percent or every two hours, whichever is more frequent.

Analyze the CCB after every CCV.

5.6.6.3 Procedure

A CCB shall be run after the last CCV in the analysis run.

If the absolute value of the CCB is greater than the MQL, the result shall be reported.

5.6.6.4 Calculations

Not applicable.

5.6.6.5 Technical Acceptance Criteria

The absolute value of the CCB must be less than the CRQL.

5.6.6.6 Corrective Action

When the CCB concentration does not meet the technical acceptance criteria, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the preceding 10 analytical samples (or all analytical samples since the last compliant CCB).

5.6.6.7 Documentation

Report the ICB values in µg/L on FORM IV-AAIN.

5.6.7 **Preparation Blanks**

5.6.7.1 Summary

To ensure against contamination during sample preparation, a preparation blank (PB) is analyzed.

5.6.7.2 Frequency

At least one PB shall must be prepared and analyzed with every SDG or with each batch of samples digested, whichever is more frequent.

5.6.7.3 Procedure

The PB shall consist of ASTM Type II water processed through each sample preparation and analysis procedure step (see Exhibit D).

The first batch of samples in an SDG is to be assigned to PB one, the second batch of samples to PB two, etc.

5.6.7.4 Calculations

Not applicable.

5.6.7.5 Technical Acceptance Criteria

The absolute value of the PB must be less than the CRQL.

5.6.7.6 Corrective Action

If the absolute value of the PB concentration is less than or equal to the CRQL, no corrective action is required.

If any analyte concentration in the blank is above the CRQL, the lowest concentration of that analyte in the associated samples must be 10 times the blank concentration. Otherwise, all samples associated with the blank and with the analyte's concentration less than 10 times the blank concentration and above the CRQL shall be redigested and reanalyzed for that analyte. The sample concentration is not to be corrected for the blank value.

If the concentration of the blank is below the negative CRQL, then all samples reported below 10 times CRQL associated with the blank shall be redigested and reanalyzed.

5.6.7.7 Documentation

The values for the PB shall be recorded in µg/L on FORM IV-AAIN.

5.6.8 Spike Sample Analysis

5.6.8.1 Summary

To provide information about the effect of the sample matrix on the digestion, a known amount of analyte is added (spiked) into a sample.

5.6.8.2 Frequency

At least one spike sample analysis shall be performed on each group of samples for each SDG. (EPA may require additional spike sample analysis upon special request by the Project Officer, for which the Contractor will be paid.)

If two analytical methods are used to obtain the reported values for the same metal within a SDG (e.g., ICP and GFAA), then spike samples shall be run by each method used.

5.6.8.3 Procedure

The spike is added before the sample preparation (i.e., prior to digestion) at the concentration levels in the Spike Sample solution as indicated in Table D-3.

Samples identified as field blanks cannot be used for spiked sample analysis.

EPA may require that a specific sample be used for the spike sample analysis.

In the instance where there is more than one spike sample per method per SDG and one spike sample recovery is not within contract criteria, flag all the samples for the same method in the SDG.

5.6.8.4 Calculations

$$\% \ Recovery = \frac{SSR - SR}{SA} \times 100 \qquad \text{Eq. D-31}$$

where:

SSR = Spiked Sample Result, µg/L;
SR = Sample Result, µg/L; and
SA = Spike Added, µg/L.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see 5.6.9 Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery.

When the sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating percent recovery.

5.6.8.5 Technical Acceptance Criteria

Recovery for the spike should be within ± 25 percent of the spiked amount (i.e., 75-125 percent).

5.6.8.6 Corrective Action

If the spike recovery is not at or within the limits of 75-125 percent, the data of all samples received associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N" on FORMs I-AAIN and VII-AAIN.

An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125 percent recovery criteria.

When the digestion spike recovery falls outside the technical acceptance criteria and the sample result does not exceed four times the spike added, an analytical spike must be performed for those metals that do not meet the specified criteria (exception: Ag). Spike the unspiked aliquot of the sample at two times the indigenous level or two times CRQL, whichever is greater.

5.6.8.7 Documentation

Report the spiked sample results, sample results, spike added and percent recovery for the digestion spike sample analysis on FORM VI-AAIN.

The units for reporting spike sample results are µg/L.

5.6.9 Duplicate Sample Analysis

5.6.9.1 Summary

Duplicate aliquots of a sample are carried through the preparation and analysis steps to provide information about the precision of the analytical methods as well as matrix effects.

5.6.9.2 Frequency

At least one duplicate sample analysis shall be performed on each group of samples for each SDG. (EPA may require additional duplicate sample analysis upon special request by the Project Officer, for which the Contractor will be paid.)

If two analytical methods are used to obtain the reported values for the same metal within a SDG (e.g., ICP and GFAA), then duplicate samples shall be run for each method used.

5.6.9.3 Procedure

Samples identified as field blanks cannot be used for duplicate sample analysis.

EPA may require that a specific sample be used for the duplicate sample analysis.

In the instance where there is more than one duplicate sample per method per SDG and one duplicate result is not within contract criteria, then flag all the samples for the method in the SDG.

Duplicate sample analyses are required for calculation of relative percent difference.

Duplicates cannot be averaged for reporting on FORM I-AAIN.

5.6.9.4 Calculations

$$RPD = \frac{|S - D|}{\frac{S + D}{2}} \times 100 \qquad \text{Eq. D-32}$$

where:

RPD = Relative Percent Difference;
S = First Sample Value (original), µg/L; and
D = Second Sample Value (duplicate), µg/L.

5.6.9.5 Technical Acceptance Criteria

A control limit of ± 20 percent for RPD shall be used for original and duplicate sample values greater than or equal to five times CRQL (Exhibit C). A control limit of ± CRQL shall be used for sample values less than five times CRQL.

If one result is above the five times CRQL level and the other is below, use the ± CRQL criteria.

For duplicate results less than five times CRQL, enter the absolute value of the CRQL in the "CONTROL LIMIT" column of FORM VIII-AAIN.

If both sample values are less than the MQL, the RPD is not calculated.

Specific control limits for each metal will be added to FORM VIII-AAIN at a later date based on precision results.

5.6.9.6 Corrective Action

If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an asterisk "*."

5.6.9.7 Documentation

The results of the duplicate sample analyses must be reported on FORM VIII-AAIN in µg/L.

The absolute value of the control limit (CRQL) shall be entered in the "CONTROL LIMIT" column of FORM VIII-AAIN.

5.6.10 Laboratory Control Samples

5.6.10.1 Summary

A laboratory control sample (LCS) is digested and analyzed to ensure against analyte loss in the sample preparation.

5.6.10.2 Frequency

One LCS shall be prepared and analyzed for every group of samples in a SDG or for each batch (i.e., a group of samples prepared at the same time) of samples, whichever is more frequent.

5.6.10.3 Procedure

The LCS shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

The LCS solution shall be obtained from EPA (if unavailable, the ICV solutions may be used).

If the EPA LCS is unavailable, other EPA Quality Assurance Check samples or other certified materials may be used.

5.6.10.4 Calculations

$$\% \text{ Recovery} = \frac{\text{Found Concentration}}{\text{True Concentration}} \times 100 \qquad \text{Eq. D-33}$$

5.6.10.5 Technical Acceptance Criteria

Recovery for the LCS shall be within ± 20 percent of the true value (i.e., 80-120 percent) with exception of Ag and Sb.

Technical acceptance criteria for Ag and Sb in the LCS will be determined at a later date.

5.6.10.6 Corrective Action

If the percent recovery for the LCS falls outside the technical acceptance criteria, the analyses shall be terminated, the problem corrected, and all samples associated with that LCS reprepared and reanalyzed.

5.6.10.7 Documentation

Report the LCS found concentration (µg/L), true concentration (µg/L), and percent recovery on FORM IX-AAIN.

5.6.11 Performance Evaluation Sample (PES)

5.6.11.1 Summary

The performance evaluation sample (PES) assists the Agency in monitoring the laboratory performance for analyte identification and quantification.

5.6.11.2 Frequency

One PES shall be delivered, prepared, and analyzed for every group of samples in a SDG, or for each batch of samples, whichever is more frequent.

5.6.11.3 Procedure

A PES shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received.

The PES solution shall be obtained from EPA. If unavailable, the contractor must contact the Agency and SMO for instructions.

5.6.11.4 Calculations

Not applicable.

5.6.11.5 Technical Acceptance Criteria

Recovery for the PES shall be within 75 percent of the true value of analyte.

5.6.11.6 Corrective Action

If the percent recovery for the PES falls below 75 percent, the Agency may take, but is not limited to the following actions:

- Show Cause and/or Cure Notice for unacceptable performance;
- Reduction of the number of samples shipped to the laboratory;
- Suspension of sample shipment;
- A site visit;
- A full data audit; or
- Require the laboratory to analyze a remedial PES.

In addition to the above cited actions, the laboratory shall also provide the Technical and Administrative Project Officers a written correction action report on every unacceptable (less than 75 percent) PES score within seven days of written or verbal request by Agency personnel.

5.6.11.7 Documentation

Report the PES on Form I-AAIN (in µg/L) for all analytes.

5.6.12 Analytical Spike Sample Analysis/Method of Standard Additions

5.6.12.1 Summary

To ensure against bias resulting from interference effects in GFAA analyses, the Method of Standard Additions (MSA) is utilized. The special procedures summarized in Figure D-3 will be required for quantitation.

5.6.12.2 Frequency

All GFAA analyses for each analytical sample will require at least one analytical spike.

The frequency of MSA will depend on the recovery of the analytical spike.

A maximum of 10 full sample analyses to a maximum of 20 injections may be performed between consecutive calibration verifications and blanks. Each full MSA counts as two analytical samples towards determining 10 percent CCV/CCB frequency (i.e., five full MSAs can be performed between calibration verifications).

5.6.12.3 Procedure

All GFAA analyses including MSA shall fall within the calibration range.

Except during full MSA, all analyses require duplicate injections. Only single injections are required for MSA quantitation. Average concentration values are used for reporting purposes.

The analytical spike (at two times CRQL) of a sample must be run immediately after that sample. The percent recovery of the analytical spike will determine the method of quantitation for the sample.

The requirement for an analytical spike will include the LCS and the preparation blank. The LCS must be quantitated from the calibration curve and corrective action, if needed, taken accordingly.

MSAs are not to be performed on the LCS or preparation blank, regardless of spike recovery results. If the preparation blank analytical spike recovery is out of the control limits 85-115 percent, the spiking solution must be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem and respike and reanalyze all analytical samples associated with the blank. An analytical spike is not required for the pre-digestion spike sample.

If the spike recovery is < 40 percent, the sample must be diluted by a factor of five to ten and rerun with another spike. This step must only be performed once. If after the dilution the spike recovery is still < 40 percent, report data from the initial undiluted analysis and flag with an "E" to indicate interference problems.

If the spike recovery is ≥ 40 percent and the sample absorbance or concentration is < 50 percent of the spike, report the sample results to the MQL. If the spike is less than 85 percent or greater than 115 percent, flag the result with a "W."

If the sample absorbance or concentration is < 50 percent of the spike and the spike recovery is at or between 85 percent and 115 percent, the sample must be quantitated directly from the calibration curve and reported down to the MQL.

If the sample absorbance or concentration is ≤ 50 percent of the spike and the spike recovery is > 85 percent or greater than 115 percent, the sample must be quantitated by MSA.

For analytical runs containing only MSAs, single injections can be used for QC samples during that run. For instruments that operate in an MSA mode only, MSA can be used to determine QC samples during that run.

The sample and three spikes must be analyzed consecutively for MSA quantitation (the "initial" spike run data is specifically excluded from use in the MSA quantitation).

MSA spikes shall be prepared such that:

a) Spike 1 is approximately 50 percent of the sample concentration;

b) Spike 2 is approximately 100 percent of the sample concentration; and

c) Spike 3 is approximately 150 percent of the sample concentration (all concentrations expressed in μg/L).

5.6.12.4 Calculations

$$\% \ Recovery = \frac{SSR - SR}{SA} \times 100 \qquad \text{Eq. D-34}$$

where:
SSR = Spiked Sample Result;
SR = Sample Result; and
SA = Spike Added.

$$RSD = \frac{\sigma_{n-1}}{\bar{x}} \times 100 \qquad \text{Eq. D-35}$$

where:
RSD = Relative Standard Deviation
σ_{n-1} = Standard deviation; and
$\bar{x}$ = Mean

5.6.12.5 Technical Acceptance Criteria

For concentrations ≥ CRQL, the duplicate injections must agree within ± 20 percent of RSD or CV.

The analytical spike recoveries for the LCS and PB shall be within control limits of ± 15 percent (i.e., MSA is NOT performed on the LCS or PB).

5.6.12.6 Corrective Action

If the RSD (CV) technical acceptance criteria are not met, rerun the sample once. If the criteria are still not met, flag the value reported on FORM I-AAIN with the letter "M".

NOTE: The "M" flag is required for the analytical spike as well as the sample.

If the PB analytical spike technical acceptance criteria are not met, verify the spiking solution by respiking and rerunning the PB once. If the criteria are still not met, correct the problem and reanalyze all analytical samples associated with the blank.

If the LCS analytical spike technical acceptance criteria are not met, correct the problem and reanalyze all analytical samples associated with that LCS.

5.6.12.7 Documentation

If the spike recovery is < 40 percent, the sample must be diluted by a factor of five to ten and rerun with another spike. This step

must only be performed once. If after the dilution the spike recovery is still < 40 percent, report data from the initial undiluted analysis and flag with an "E" to indicate interference problems.

If the spike recovery is ≥ 40 percent and the sample absorbance or concentration is < 50 percent of the spike, report the sample results to the MQL. If the spike is less than 85 percent or greater than 115 percent, flag the result with a "W."

If the sample absorbance or concentration is < 50 percent of the spike and the spike recovery is at or between 85 percent and 115 percent, the sample must be quantitated directly from the calibration curve and reported down to the MQL.

The raw data package must include absorbance and concentration values for both injections, the average value, and the coefficient of variation (or RSD).

The data for each MSA analysis must be clearly identified in the raw data documentation (using added concentration as the x-variable and absorbance as the y-variable) along with the slope, x-intercept, y-intercept, and correlation coefficient (r) for the least squares fit of the data.

Reported values obtained by MSA must be flagged with the letter "S" on FORM I-AAIN if the correlation coefficient is ≥ 0.995. If the correlation coefficient is ≤ 0.995, flag the data on FORMs I-AAIN and XII-AAIN with a "+".

5.6.13 Method Quantitation Limits

5.6.13.1 Summary

The method quantitation limit (MQL) shall be determined before any samples are analyzed for every instrument that will be used.

5.6.13.2 Frequency

MQLs must be determined within 30 days of the start of the contract and at least quarterly (every three calendar months).

5.6.13.3 Procedure

MQLs (in μg/L) shall be determined by multiplying by three the average of the standard deviations (σ_{n-1}) obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration three times to five times of the instrument manufacturer's suggested instrument detection limit (IDL), with seven consecutive measurements per day. Each measurement

must be performed as though it were a separate analytical sample (i.e., each measurement shall be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). MQLs shall be determined and reported for each wavelength used in the analysis of the samples.

The quarterly determined MQL for an instrument shall always be used as the MQL for that instrument during that quarter. If the instrument is adjusted in any way that may affect the MQL, the MQL for that instrument shall be redetermined and the results submitted for use as the established MQL for that instrument for the remainder of the quarter.

MQL must be determined in μg/L.

5.6.13.4 Calculations

$$MQL = 3 \times \sigma_{n-1} \qquad \text{Eq. D-36}$$

where:

σ_{n-1} = Standard Deviation

5.6.13.5 Technical Acceptance Criteria

The MQL shall meet the CRQLs established in Exhibit C.

5.6.13.6 Corrective Action

If an instrument's MQL cannot meet the CRQL for an analyte, that instrument cannot be used to quantitate an analyte unless the sample concentration exceeds five times the MQL.

5.6.13.7 Documentation

For each instrument used, MQLs shall be reported on FORM XV-AAIN and submitted with each data package. If multiple GFAA instruments are used for the analysis of a metal within a SDG, the highest MQL for the GFAAs shall be used for reporting concentration values for that SDG.

5.7 INSTRUMENT OPERATION

5.7.1 Set up the instrument with the proper operating parameters established by the instrument manufacturer. The individual steps (drying, charring and atomization) require careful consideration to ensure each process is carried out effectively. The instrument shall be allowed to become thermally stable before beginning any analysis. This usually requires at least 30 minutes of operation prior to calibration. Background correction shall be used.

5.7.2 Calibrate the instrument according to the manufacturer's recommended procedures using calibration standard solutions.

5.7.3 In order to determine if the sample result is to be calculated by MSA, an analytical spike at two times CRQL shall be performed and analyzed after each sample analysis. The analytical spike recovery shall be used to determine the need for MSA as explained in 5.6.12. The spiking solution volume shall not exceed 10 percent of the sample volume.

5.7.4 Dilute and reanalyze samples that are more concentrated than the linear range (i.e., top calibration standard) for an analyte.

5.8 **PROCEDURE**

5.8.1 **Calibration**

5.8.1.1 Set up the instrument with proper operating parameters. The instrument shall be allowed to become thermally stable before beginning analysis. This requires at least 30 minutes of operation with the lamp lit prior to calibration.

5.8.1.2 Initiate appropriate operating configuration of the computer, if used.

5.8.1.3 Calibrate the instrument using the appropriate matrix matched calibration standard solution(s). The number of standards utilized is left to the discretion of the operator but shall include a minimum of a calibration blank and at least three standards. The operator should be aware of the requirements in Exhibits D and E that provide for the assurance that all sample values are within the calibration range.

5.8.1.4 All standards, blanks, and sample solutions shall be matrix matched. A change in the acid strength changes the slope of the calibration curve and can cause inaccurate results.

5.9 **Analysis Sequence**

5.9.1 Before beginning the sample analysis run, analyze the ICB ICV, and CRA under the same operating conditions intended for sample analyses. The ICV found concentration values shall not deviate from the true values by more than 10 percent. The CRA recovery values must be within ± 15 percent of the true values. The calibration blank values may not exceed the CRQL. If these conditions are not met for any element, the analysis shall be discontinued and corrective action applied until the conditions are met (see Exhibits D and E for additional information).

5.9.2 Upon successful analysis of the ICV and ICB, analyze all method PB extract(s) prepared with the digested samples. If any of the

blank(s) are not less than or equal to the CRQL, see Exhibits D and E for the appropriate action.

5.9.3 If the method blank(s) values are acceptable, analyze the LCS. If any LCS values deviate from the acceptable ranges, see Exhibits D and E for the appropriate action.

5.9.4 If the LCS values are within the acceptable ranges, analyze the method spike sample. If the recovery of any element deviates from the acceptable ranges, see Exhibits D and E for the appropriate action. Proceed to the analysis of samples if the recoveries are acceptable or after consulting Exhibits D and E.

5.9.5 The CCV and the CCB must be analyzed after every 10 analytical sample analyses. CCV values shall not deviate from the actual values by more than ± 10 percent. In addition, the absolute values for the calibration blank shall be lower than the required quantitation limits. If these conditions are not met at any time during samples analysis, discontinue the analysis and see Exhibits D and E for the appropriate action.

5.9.6 At the end of the sample analysis run, analyze the CRA, CCB, and CCV. If the values for any of these samples deviates from the required limits, see Exhibits D and E.

5.10 **Sample Analyses**

5.10.1 All sample extracts must first be analyzed without any dilution. Diluting sample extracts is permissible if necessary provided that the dilution does not result in a sample concentration below CRQL.

5.10.2 All concentrations reported shall be obtained within the established calibration range for that analysis run. All concentrations within the calibration range of the analyte are to be reported.

5.10.3 In order to determine if the sample result is to be calculated by MSA, an analytical spike at two times CRQL shall be performed and analyzed immediately after each sample analysis. The analytical spike recovery is used to determine the need for MSA as explained in part 5.6.12 and Exhibit E. The spiking solution volume shall not exceed 10 percent of the sample volume.

5.10.4 Add appropriate volumes of the spiking standard to the remaining three aliquots that result in concentrations of 50 percent, 100 percent, and 150 percent of the sample concentration. The spiking standard solution volume added to each aliquot shall not exceed 10 percent of the volume of the aliquot. Add the appropriate amount of blank solution to each aliquot to make the total of spike plus blank volumes added equal.

5.10.5 Using a calculator or a statistical package on a computer, determine the slope, the intercepts of the ordinate (y-axis) and the abscissa (x-axis), and the correlation coefficient using the found concentration as the ordinate and the standard addition concentration as the abscissa. The absolute value of the intercept of the abscissa is the concentration of the analyte in the dilute solution. If the correction coefficient is less than 0.995, then the analyses shall be repeated. If the second analysis correlation coefficient is less than 0.995, then repeat the analysis using a smaller aliquot of the sample digest. Be certain to correct for the difference in volume created by using a smaller sample digest aliquot. If the correlation coefficient is still less than 0.995, flag the sample data with a "+."

5.11 Calculations

5.11.1 Determine the method detection limit (MQL) from the standard deviation of the method blank analyte analyses, based on the standard addition plot.

5.11.2 Calculate sample extract concentrations (µg/L) by multiplying the analyte concentration calculated by the appropriate dilution factors used in Sections 5.8.1. Calculate the air concentration for each analyte in µg/m³ by multiplying the analyte concentration in µg/L by the extract volume (liters) times 9 and dividing by the volume of the air sampled (standard m³) per filter and the number of filter strips digested. The factor "9" represents the area (63 in²) of the Hi-Vol filter exposed to the air stream divided by the exposed area (7 in²) in one 1"x8" strip.

$$\text{Analyte Conc. In air, } \mu g/m^3 = \frac{(9)\left(\text{Interference-Corrected Value, } \mu g/L\right)\left(\text{Extract Volume, } L\right)}{\text{no. of strips digested} \times \text{air volume sampled, std. } m^3} \quad \text{Eq. D-37}$$

Calculate all method spike levels relative to the corresponding unspiked sample concentration in units of µg/L.

Calculate the relative percent difference (RPD) for both the method and analysis duplicates. Calculate the RPD by dividing the absolute value of the difference between the sample value and the duplicate value by their mean and multiplying by 100.

$$RPD = \frac{|S - D|}{\frac{S + D}{2}} \times 100 \qquad \text{Eq. D-38}$$

where:

RPD = Relative Percent Difference;
S = First Sample Value (original), µg/L; and
D = Second Sample Value (duplicate), µg/L.

SECTION 6

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TABLE D-1

RECOMMENDED ICP WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength, nm[1]	Estimated Detection Limit, µg/L[2]
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	(See 3)
Selenium	196.026	75
Silver	238.068	7
Sodium	588.955	29
Thallium	190.864	40
Tin	189.99	42
Vanadium	292.402	8
Zinc	213.856	2

(1) The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. The use of alternate wavelengths shall be reported (in nm) with the sample data.

(2) The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines," EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

(3) Highly dependent on operating conditions and plasma position.

TABLE D-2

EXAMPLE OF ANALYTE CONCENTRATION EQUIVALENTS (MG/L) ARISING FROM INTERFERENTS AT THE 100 MG/L LEVEL

Analyte	Wavelength (nm)	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Boron	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Silicon	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

TABLE D-3

SPIKING LEVELS FOR SPIKE SAMPLE ANALYSIS
FOR INORGANIC ANALYSES

Analyte	For ICP and ICP-MS (µg/L)	For GFAA (µg/L)
Aluminum	2,000	
Antimony	500	100
Arsenic	2,000	40
Barium	2,000	
Beryllium	2,000	
Cadmium	50	5
Calcium	*	
Chromium	200	
Cobalt	500	
Copper	250	
Iron	1,000	
Lead	500	20
Magnesium	*	
Manganese	500	
Nickel	500	
Potassium	*	
Selenium	2,000	10
Silver	50	
Sodium	*	
Thallium	2,000	50
Tin	500	
Vanadium	500	
Zinc	500	

* No spike required.

NOTE: Elements without spike levels and not designated with an asterisk, must be spiked at appropriate levels.

TABLE D-4

TUNIG SOLUTION FOR ICP-MS

Analyte	Concentration (µg/L)
^{7}Lithium	100
Cobalt	100
Indium	100
Thallium	100

NOTE: The tuning solution shall consist of the following analytes at the stated concentrations.

TABLE D-5

TUNING, RESPONSE FACTOR AND MASS CALIBRATION CRITERIA

TUNING CRITERIA	
m/z	**Ion Abundance Criteria**
^{7}Lithium/^{59}Cobalt	0.20 - 1.00
^{59}Cobalt/^{59}Cobalt	1.00
^{115}Indium/^{59}Cobalt	0.75 - 2.00
^{205}Thallium/^{59}Cobalt	0.50 - 1.20
RESPONSE FACTOR CRITERIA	
m/z	**Response Factor Criteria**
^{7}Lithium	> 2,000
^{59}Cobalt	> 20,000
^{115}Indium	> 10,000
^{102}Ruthenium	< 25
^{205}Thallium	> 1,000
MASS CALIBRATION CRITERIA	
m/z	**Exact Mass**
^{7}Lithium	6.9160 - 7.1160
^{59}Cobalt	58.8332 - 59.0332
^{115}Indium	114.8040 - 115.0040
^{205}Thallium	204.8744 - 205.0744

TABLE D-6

MEMORY TEST SOLUTION FOR ICP-MS

The memory solution shall consist of the following analytes:

Analyte
Aluminum (Al)
Antimony (Sb)
Arsenic (As)
Barium (Ba)
Beryllium (Be)
Calcium (Ca)
Cadmium (Cd)
Carbon (C)
Chloride (Cl)
Chromium (Cr)
Cobalt (Co)
Copper (Cu)
Iron (Fe)
Lead (Pb)
Magnesium (Mg)
Manganese (Mn)
Molybdenum (Mo)
Nickel (Ni)
Phosphorus (P)
Potassium (K)
Selenium (Se)
Silver (Ag)
Sodium (Na)
Sulfur (S)
Titanium (Ti)
Tritium (T)
Vanadium (V)
Zinc (Zn)

TABLE D-7

INTERNAL STANDARDS WHICH MAY BE USED IN ICP-MS

Internal Standard
Scandium (Sc)
Yttrium (Y)
Rhodium (Rh)
Indium (In)
Terbium (Tb)
Holmium (Ho)
Bismuth (Bi)
Lithium (Li)

TABLE D-8

RECOMMENDED ELEMENTAL EXPRESSIONS FOR ISOBARIC INTERFERENCES

Analyte	Isobaric Correction	Expression Proportional to Analyte Concentration
Aluminum (Al)	None	$(1.0000)(^{27}M)$
Antimony (Sb)	None	$(1.0000)(^{121}M)$
Arsenic (As)	ArCl, Se	$(1.0000)(^{75}M) - (3.1277)(^{77}M) + (2.5288)(^{82}M)$
Barium (Ba)	None	$(1.0000)(^{135}M)$
Beryllium (Be)	None	$(1.0000)(^{9}M)$
Cadmium (Cd)	MoO, Sn	$(1.0000)(^{114}M) - (0.0268)(^{118}M) - (1.6285)(^{108}M)$
Calcium (Ca)	None	$(1.0000)(^{44}M)$
Chromium (Cr)	None	$(1.0000)(^{52}M)$
Cobalt (Co)	None	$(1.0000)(^{59}M)$
Copper (Cu)	None	$(1.0000)(^{65}M)$
Iron (Fe)	None	$(1.0000)(^{57}M)$
Lead (Pb)	None	$(1.0000)(^{208}M) + (1.0000)(^{207}M) + (1.0000)(^{206}M)$
Magnesium (Mg)	None	$(1.0000)(^{25}M)$
Manganese (Mn)	None	$(1.0000)(^{55}M)$
Nickel (Ni)	None	$(1.0000)(^{60}M)$
Silver (Ag)	None	$(1.0000)(^{107}M)$
Thallium (Tl)	None	$(1.0000)(^{205}M)$
Vanadium (V)	ClO, Cr	$(1.0025)(^{51}M) - (3.1158)(^{53}M) + (0.3533)(^{52}M)$
Zinc (Zn)	None	$(1.0000)(^{66}M)$
^{6}Lithium (Li)	None	$(1.0000)(^{6}M)$
Scandium (Sc)	None	$(1.0000)(^{45}M)$
Yttrium (Y)	None	$(1.0000)(^{89}M)$
Rhodium (Rh)	None	$(1.0000)(^{103}M)$
Indium (In)	Sn	$(1.0000)(^{115}M) - (0.0005)(^{118}M)$
Terbium (Tb)	None	$(1.0000)(^{159}M)$
Holmium (Ho)	None	$(1.0000)(^{165}M)$
Bismuth (Bi)	None	$(1.0000)(^{165}M)$

M = The total ion count rate at the specified mass.

TABLE D-9

CONTRIBUTIONS OF CONCOMITANT ANALYTES TO NEARY ANALYTES FOR ICP-MS WHEN RESOLUTION AND MEASUREMENT SCHEMES VARY

Analyte	Interferent Analyte	Peak Width at 10% of the Peak Height			
		1.0 amu Integration Width		0.8 amu Integration Width	
		0.9 amu	0.3 amu	0.9 amu	0.3 amu
^{121}Sb	^{120}Sn	60	300	3	310
^{121}Sb	^{122}Te	360	730	940	600
^{75}As	^{74}Se, ^{76}Se	50	40	160	35
^{9}Be	^{10}B	100	None	None	None
^{112}Cd	^{113}In	40	560	680	None
^{114}Cd	^{115}In	1.5	380	20	670
^{116}Cd	^{115}In	15	25	0.5	25
^{52}Cr	^{51}V	140	110	110	90
^{53}Cr	^{54}Fe	5	7	15	7
^{59}Co	^{58}Ni, ^{60}Ni	70	610	300	660
^{63}Cu	^{62}Ni, ^{64}Ni	30	30	20	20
^{63}Cu	^{64}Zn	25	25	10	20
^{65}Ni	^{64}Ni	30	30	20	20
^{65}Cu	^{64}Zn, ^{66}Zn	20	25	10	20
^{206}Pb	^{205}Tl	110	50	1	30
^{208}Pb	^{209}Bi	2	130	160	90
^{55}Mn	^{54}Fe, ^{56}Fe	20	250	120	310
^{202}Hg	^{203}Tl	0.01	70	40	None
^{60}Ni	^{59}Co	110	75	160	90
^{62}Ni	^{63}Cu	1	50	20	60
^{107}Ag	^{106}Pd, ^{108}Pd	30	210	5	280
^{107}Ag	^{106}Cd, ^{108}Cd	500	530	80	540
^{109}Ag	^{108}Pd, ^{110}Pd	60	210	5	270
^{109}Ag	^{108}Cd, ^{110}Cd	70	510	90	530
^{205}Tl	^{206}Pb	30	60	140	80
^{51}V	^{52}Cr	15	200	170	220
^{64}Zn	^{65}Cu, ^{63}Cu	20	110	70	140
^{66}Zn	^{65}Cu	60	60	80	60

NOTE: Concentrations listed are the approximate level (mg/L) of interferent which gives an analyte concentration of 10 μg/L.

TABLE D-10

ISOBARIC MOLECULAR-ION INTERFERENCES WHICH COULD AFFECT THE ANALYTES

Analyte	Oxygen Inter.	Hydroxyl Inter.	Nitrogen Inter.	Chlorine Inter.	Sulfur Inter.	Carbon Inter.	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	SeCl, AsCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	Mo^{++}
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Sn^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	ArNa
^{59}Co	CaO	CaOH	ScN	MgCl	AlS	TiC	
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	
^{65}Cu	TiO	TiOH	VN	SiCl	SS, SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							

TABLE D-10 (Continued)

Analyte	Oxygen Inter.	Hydroxyl Inter.	Nitrogen Inter.	Chlorine Inter.	Sulfur Inter.	Carbon Inter.	Other
^{196}Hg			WN			WC	
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	ScO	CaOH	TiN	MgCl	SiS	TiC	Sn^{++}
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	NiOH	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	FeOH	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo^{++}
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, CrOH	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

TABLE D-11

MASS CHOICES FOR ANALYTES WHICH MUST BE MONITORED
EITHER DURING THE ANALYTICAL RUN OR IN A SEPARATE SCAN FOR ICP-MS

Analytes of Interest	Mass
Aluminum	27
Antimony	121, 123
Arsenic	75
Barium	138, 137, 136, 135, 134, 132, 130
Beryllium	9
Cadmium	114, 112, 111, 110, 113, 116, 106, 108
Calcium	42, 43, 44, 46, 48
Cerium	140
Chlorine	35, 37
Chromium	52, 53, 50, 54
Cobalt	59
Copper	63, 65
Gallium	69
Germanium	72
Iron	56, 54, 57, 58
Krypton	83
Lanthanum	139
Lead	208, 207, 206, 204
Magnesium	24, 25, 26

TABLE D-11 (Continued)

Analytes of Interest	Mass
Manganese	55
Mercury	202, 200, 199, 201
Molybdenum	98, 96, 92, 97, 94
Nickel	58, 60, 62, 61, 64
Palladium	105
Potassium	39
Selenium	80, 78, 82, 76, 77, 74
Silver	107, 109
Sodium	23
Tellurium	125
Thallium	205, 203
Tin	118
Titanium	47, 49
Vanadium	51, 50
Xenon	129
Zinc	64, 66, 68, 67, 70

NOTE: The masses which must be monitored are indicated by underlining, it is strongly recommended that the other elements be monitored to indicate other potential molecular interferences which could affect the data quality.

FIGURE D-1

TEMPLATE FOR CUTTING GLASS-FIBER FILTERS

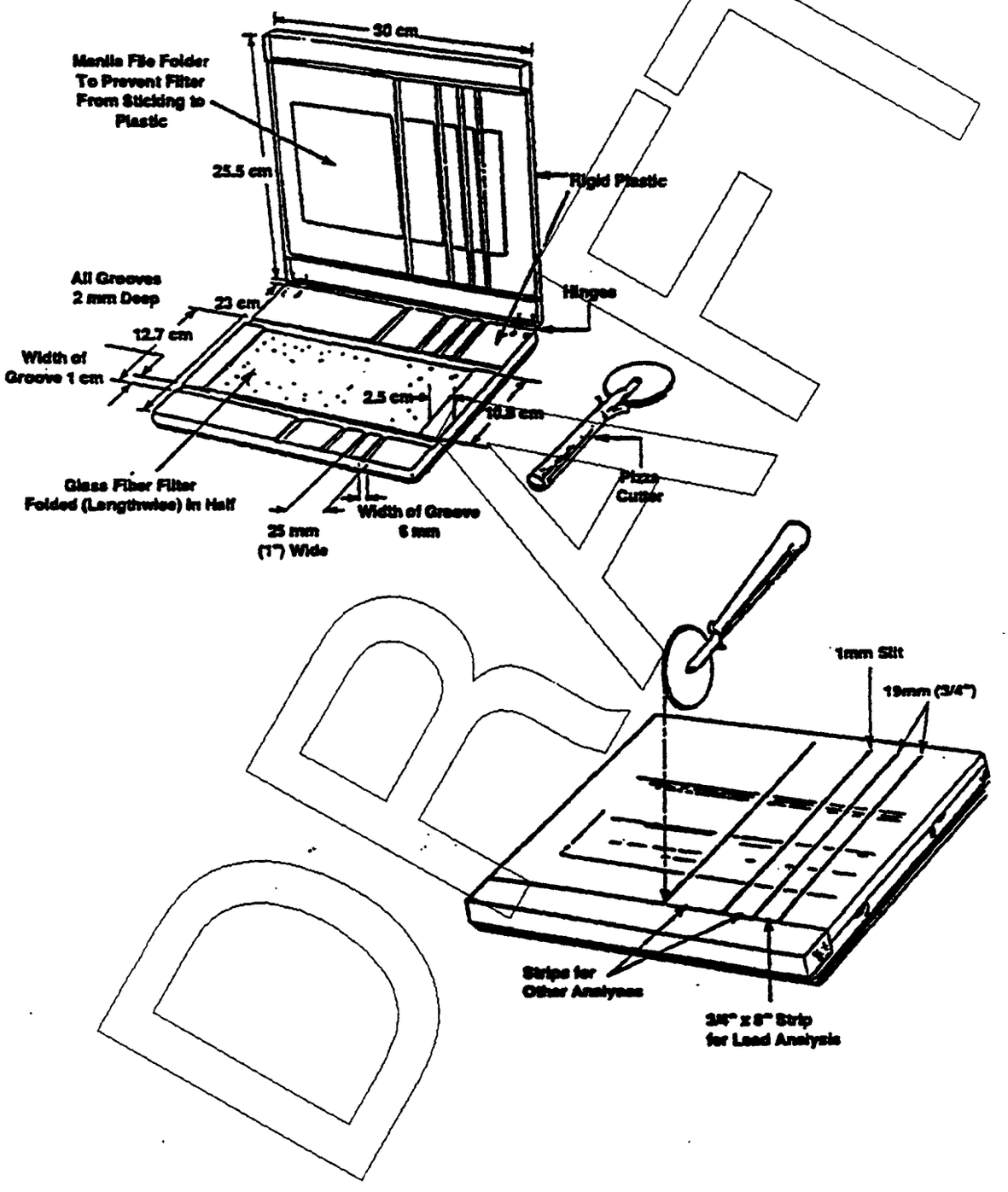

FIGURE D-2

MICROWAVE DIGESTION SYSTEM

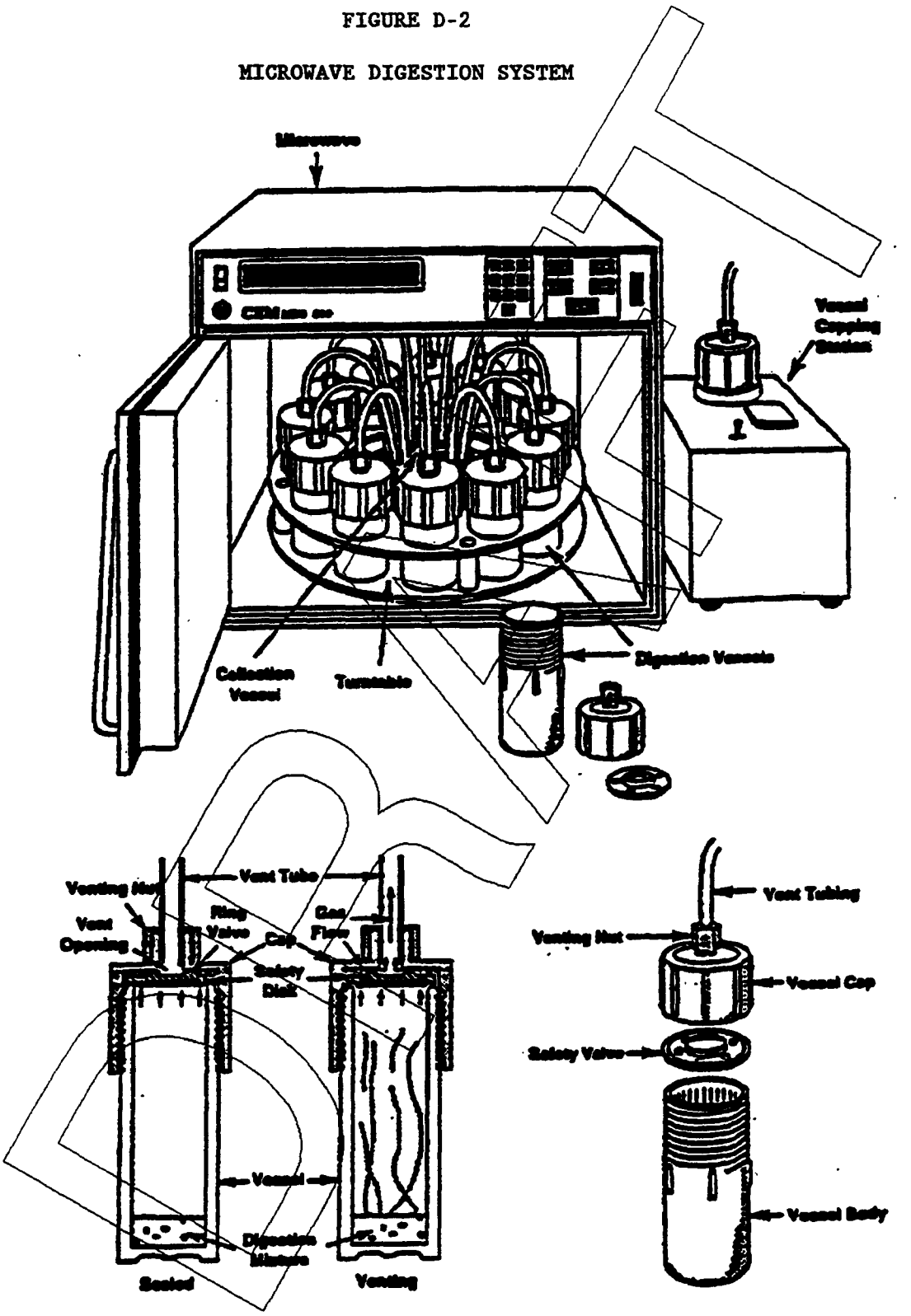

FIGURE D-3
FURNACE ATOMIC ABSORPTION ANALYSES SCHEME

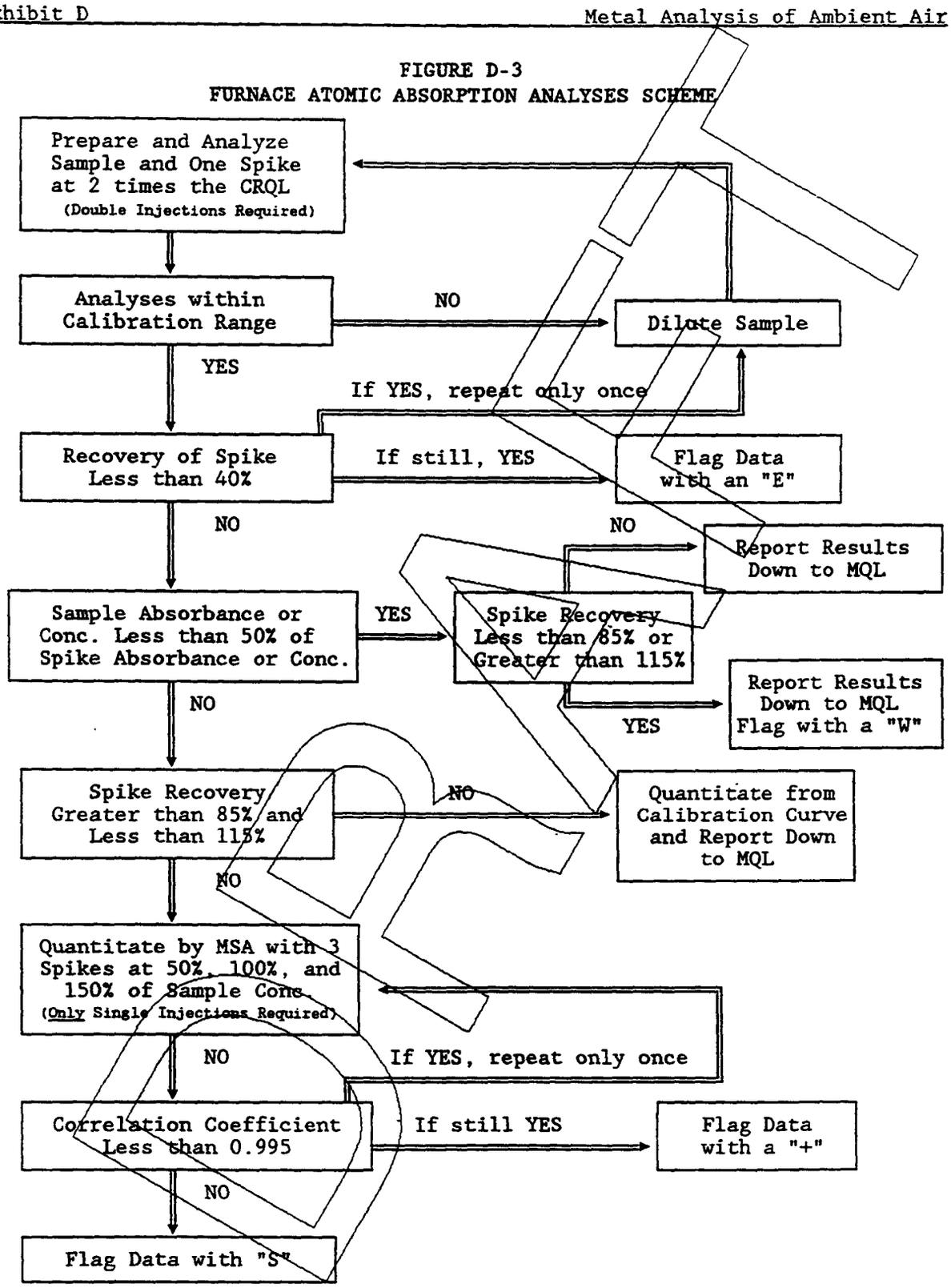

EXHIBIT E

QUALITY ASSURANCE REQUIREMENTS

December, 1991

EXHIBIT E

QUALITY ASSURANCE REQUIREMENTS

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SECTION 1

INTRODUCTION

Quality assurance (QA) and quality control (QC) are integral parts of EPA's Contract Laboratory Program (CLP). The CLP QA program consists of management review and oversight at the planning, implementation, and completion stages of environmental data generation activities to ensure that data provided are of the quality required. The CLP QC program includes those activities required as part of data generation to ensure that the data are of known and documented quality.

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

The purpose of this Exhibit is to describe the processes by which the CLP meets the quality objectives defined. This contract requires a variety of activities which represent the minimum QA/QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These operations are designed to ensure the generation of comparable data from all Contractors. These requirements do not release the laboratory from maintaining its own QC checks on method and instrument performance.

Appropriate use of data generated under the great range of analytical conditions encountered in ambient air analyses requires reliance on the QC procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories participating in the CLP. However, the validation of these methods does not guarantee that they perform equally well for all samples collected under actual field conditions. Inaccuracies can result from causes such as sampling artifacts, equipment malfunctions, and human error. Therefore, the QC component of each method is indispensable.

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

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, SMO, NEIC, and EMSL/LV. Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Performance evaluation samples provide an external QA reference for the program. A laboratory 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 contract laboratories through direct communications with the APOs and TPOs.

This Exhibit is not a guide to constructing quality assurance project plans, quality control systems, or a quality assurance organization. It is, however, an explanation of the QA requirements of the CLP. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a QA Plan, and the QA/QC documentation required by this contract. Delivery of this documentation provides the Agency with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

To ensure that the product delivered by the Contractor meets the requirements of the contract and to improve inter-laboratory data comparison, the Agency requires the following from the Contractor:

- Development and implementation of a QA program and documentation of the key elements of that QA program through a written QA Plan, as described in Section 2 of this Exhibit.
- Preparation of and adherence to written Standard Operating Procedures (SOPs) as described in Section 3 of this Exhibit.
- Adherence to the analytical methods and associated QC and documentation requirements specified in the contract.
- Verification of analytical standards and documentation of the purity of reference standard materials and the purity and accuracy of solutions obtained from private chemical houses.
- Participation in the analysis of laboratory performance evaluation samples, including adherence to corrective action procedures.
- Participation in on-site laboratory evaluations, including adherence to corrective action procedures.
- Submission of all raw data and pertinent documentation for Regional review.
- Submission for Agency review of all original documentation generated during sample analyses.

SECTION 2

QUALITY ASSURANCE PLANS

The Contractor shall establish a quality assurance program with the objective of providing sound analytical chemical measurements. This program shall incorporate the quality control procedures, a corrective action system, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

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

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

The QAP must present, in specific terms, the policies, organization, objectives, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during on-site laboratory evaluation and upon written request by the APO. Additional information relevant to the preparation of a QAP can be found in EPA and ASTM publications.

2.1 ELEMENTS OF A QUALITY ASSURANCE PLAN

The following key elements of the Contractors quality program shall be address in the QAP.

2.1.1 Contractor QA Policy and Objectives

2.1.2 Organization and Personnel

- QA Management;
  - Organization;
  - Assignment of QC and QA responsibilities; and

- Reporting relationships.

• Personnel;

- Staff resumes;
- Education and experience requirements pertinent to this Contract; and
- Training progress.

• Facilities and Equipment;

- Instrumentation and backup alternatives; and
- Maintenance activities and schedules.

• Document Control;

- Laboratory notebook policy;
- Sample and data tracking/custody procedures and documentation requirements;
- Logbook maintenance and archiving procedures;
- Case file organization, preparation, and review procedures; and
- Procedures for preparation, approval, review, revision, and distribution of SOPs.

• Analytical Methodology;

- Calibration procedures and frequency;
- Sample/extract handling and storage procedures;
- Sample preparation/extraction procedures;
- Sample analysis procedures; and
- Standards preparation procedures.

• Data Generation;

- Data collection procedures;
- Data reduction procedures;

- Data review procedures;
- Data reporting and authorization procedures; and
- Data management procedures.

• Quality Control Program; and

- Solvent, reagent, and adsorbent check analysis;
- Reference material analysis;
- Internal QC checks; and
- Corrective action and determination of QC limit Procedures.

• Quality Assurance Program Assessment.

- Data audits;
- Systems audits;
- Performance audits;
- Corrective action procedures;
- QA reporting procedures.

SECTION 3

STANDARD OPERATING PROCEDURES

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document that 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.

SOPs prepared by the Contractor must be functional; i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs, as presented to the Agency, must reflect activities as they are currently performed in the laboratory. In addition, all SOPs must:

- Be consistent with current EPA regulations, guidelines, and the CLP contract's requirements.
- Be consistent with instrument manufacturer's specific instruction manuals.
- Be available to the EPA 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 evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs.
- Provide for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Describe the mechanism for demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results.
- Describe the corrective measures and feedback mechanism used when analytical results do not meet protocol requirements.
- Be reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Be archived for future reference in usability or evidentiary situations.
- Be available at specific work stations as appropriate.

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

3.1 SOP SPECIFICATIONS AND FORMAT

3.1.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits.

3.1.2 The format for SOPs may vary depending upon the kind of activity for which they are prepared. However, at a minimum, the following sections must be included:

3.1.2.1 Title page.

3.1.2.2 Scope and application.

3.1.2.3 Definitions.

3.1.2.4 Procedures.

3.1.2.5 QC acceptance criteria.

3.1.2.6 Corrective Action Procedures, including procedures for secondary review of information being generated.

3.1.2.7 Documentation Description and example forms.

3.1.2.8 Miscellaneous notes and precautions.

3.1.2.9 References.

3.2 REQUIRED SOPS

3.2.1 Evidentiary SOPs

The Contractor shall develop and use adequate written SOPs to ensure sample and data accountability. Evidentiary SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.2.1.1 Sample receipt and logging

3.2.1.1.1 The Contractor shall have written SOPs for receiving and logging in the samples. The procedures shall include, documentation of the following information:

- Presence or absence of EPA chain-of-custody forms;
- Presence or absence of airbills or airbill stickers;
- Presence or absence of EPA Traffic Reports or SAS packing lists;
- Presence or absence of custody seals on shipping and/or sample containers and their condition;
- Custody seal numbers, when present;
- Presence or absence of sample tags;
- Sample tag ID numbers;
- Condition of the shipping container;
- Condition of the sample container;
- Verification of agreement or nonagreement of information on receiving documents and sample containers;
- Resolution of problems or discrepancies with SMO; and
- The definition of any terms used to describe sample condition upon receipt.

3.2.1.1.2 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.2.1.2 Sample identification

3.2.1.2.1 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.2.1.2.2 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.2.1.2.3 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

3.2.1.3 Sample security

The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access to secure storage areas.

3.2.1.4 Internal chain-of-custody of samples and data.

The Contractor shall have written SOPs for the chain-of-custody consisting of sample identification, chain-of-custody procedures, sample receiving procedures, and sample tracking procedures. For more information concerning the chain-of-custody procedures see Section 4 of this Exhibit.

3.2.1.5 Internal tracking of samples and data.

3.2.1.5.1 The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

- A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses;
- A description of the documentation used to record instrument calibration and other QA/QC activities; and
- Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.2.1.6 Laboratory document and information control

3.2.2 **Analytical SOPs**

The Contractor shall develop and use adequeate written SOPs to ensure that all data generated for the CLP are of known, documented, and acceptable quality. Analytical SOPs shall include specific procedures for the following processes as they are performed by the Contractor:

3.2.2.1 The Contractor shall have written SOPs for preventing sample contamination, during sample preparation, cleaning of glassware, storage, and analysis.

3.2.2.2 The Contractor shall have SOPs to ensure traceability of standards used in sample analysis QA/QC.

3.2.3 **Quality Management SOPs**

3.2.3.1 The Contractor shall have written SOPs for technical and managerial review of laboratory operation and data package preparation, laboratory data review/laboratory self inspection system. The procedures shall include but not be limited to documenting the following information:

3.2.3.1.1 Data flow and chain-of-command for data review;

3.2.3.1.2 Procedures for measuring precision and accuracy.

3.2.3.1.3 Evaluation of parameters for identifying systematic errors.

3.2.3.1.4 Procedures to assure that hardcopy deliverables are complete and compliant with the requirements in Exhibit B.

3.2.3.1.5 Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.).

3.2.3.1.6 Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).

3.2.3.1.7 Demonstration of problem identification-corrective actions and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).

3.2.3.1.8 Documentation of audit reports, (internal and external), response, corrective action, etc.

3.2.3.2 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure. For more information concerning document control and case file preparation, see Section 5 of this Exhibit.

3.2.3.3 The Contractor shall have written SOPs for sample analysis, management and handling, and reporting of data. The procedures shall include but not be limited to documenting the following information:

3.2.3.3.1 Procedures for controlling and estimating data entry errors.

3.2.3.3.2 Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

3.2.3.3.3 Life cycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

3.2.3.3.4 Database security, backup and archival procedures including recovery from system failures.

3.2.3.3.5 System maintenance procedures and response time.

3.2.3.3.6 Individual(s) responsible for system operation, maintenance, data integrity and security.

3.2.3.3.7 Specifications for staff training procedures.

3.2.3.4 The Contractor shall have written SOPs for laboratory safety.

3.3 HANDLING OF CONFIDENTIAL INFORMATION

3.3.1 A Contractor conducting work under this contract may receive EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.3.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.3.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

3.4 SOPS DELIVERY REQUIREMENTS

Within forty-five (45) days of contract receipt, a complete set of SOPs relevant to this contract shall be sent to the TPO, SMO and EMSL/LV. Also, during the term of performance of the contract, copies of SOPs which have been amended or new SOPs which have been written shall be sent to the TPO, EMSL/LV (quality assurance SOPs) and NEIC (evidentiary SOPs).

SECTION 4

CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

4.1 SAMPLE IDENTIFICATION

4.1.1 To ensure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification and tracking of samples throughout the laboratory.

4.1.2 Each air sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA sample number.

4.2 CHAIN-OF-CUSTODY PROCEDURES

4.2.1 Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until the associated data are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample and data custody are maintained and documented. A sample is under custody if the following applies:

4.2.1.1 It is in your possession, or

4.2.1.2 It is in your view after being in your possession, or

4.2.1.3 It was in your possession and you locked it up, or

4.2.1.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

4.3 SAMPLE RECEIVING PROCEDURES

4.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

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

4.3.3 The condition of the shipping containers and canisters shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

4.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

4.3.5.1 Airbills or airbill stickers.

4.3.5.2 Custody seals.

4.3.5.3 EPA custody records.

4.3.5.4 EPA traffic reports or SAS packing lists.

4.3.5.5 Sample tags.

4.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

4.3.7 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

4.3.8 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

4.3.9 The following information shall be recorded on appropriate Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected:

4.3.9.1 Condition of the shipping container.

4.3.9.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

4.3.9.3 Custody seal numbers, when present.

4.3.9.4 Condition of the sample canisters.

4.3.9.5 Presence or absence of airbills or airbill stickers.

4.3.9.6 Airbill or airbill sticker numbers.

4.3.9.7 Presence or absence of EPA custody records.

4.3.9.8 Presence or absence of EPA traffic reports or SAS packing lists.

4.3.9.9 Presence or absence of sample tags.

4.3.9.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

4.3.9.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

4.3.9.12 Problems or discrepancies.

4.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 5

DOCUMENT CONTROL

The goal of the laboratory document control program is to assure that all documents for a specified SDG are accountable upon completion of the project. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

5.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

5.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from an SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

5.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

5.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

5.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

5.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

5.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than

government agency or commercial client names to preserve the confidentiality of commercial clients.

5.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be "crossed" out.

5.2 CONSISTENCY OF DOCUMENTATION

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

5.2.2 All copies of laboratory documents shall be complete and legible.

5.2.3 Original documents what include information relating to more than one SDG shall be filled in the CSF of the lowest SDG number. The copy(s) shall be placed in the other CSF(s) and the Contractor shall record the following information on the copy(s) in red ink:

"COPY"

"ORIGINAL IS FILED IN CSF _____"

The Contractor shall sign and date this addition to the copy(s).

5.2.4 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

5.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURE

5.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

5.3.2 All documents relevant to each SDG, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried as a group.

5.3.3 The DCO shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 is an example of a document inventory.

FIGURE E-1

Example

DOCUMENT INVENTORY

Document Control #*	Document Type	No. of Pages
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	50
232-2-0005	SMO Inorganics Traffic Reports	10
232-2-0006	Inorganics Analysis Data Summary Sheets	17
232-2-0007	Analysts' Inorganics Notebook Pages	14
232-2-0008	ICP, ICP-MS and AA Instrument Logbook Pages	12
etc.	etc.	etc.

*This number is to be recorded on each set of documents.

5.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

5.5 SHIPPING DATA PACKAGES AND CSF

5.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

5.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

5.5.3 A copy of the transmittal letter for the CSF will be sent to NEIC and SMO.

5.5.4 The Document Control Form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one (1) original FORM AADC-1 for each shipping container.

5.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, and record the presence or absence of custody seals and their conditions.

5.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

5.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 6

ANALYTICAL STANDARDS REQUIREMENTS

The U.S. Environmental Protection Agency will not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All Contractors will be required to prepare reference standards from traceable National Institute of Standards and Technology Standard Reference Materials or NIST/EPA approved certified reference material. If the NIST/EPA reference standards are not available, the Contractor can purchase them from private chemical supply houses or venders, those standards necessary to successfully and accurately perform the analyses required in this protocol.

6.1 PREPARATION OF CHEMICAL STANDARDS FROM THE HIGH PURITY BULK MATERIAL

6.1.1 A Contractor may prepare its chemical standards from high purtiy materials. Contractor laboratories should obtain the highest purity possible when purchasing chemical standards; standards purchased at less than 97% purity must be documented as to why a higher purity could not be obtained.

6.1.2 Proper storage of chemical standards is essential in order to safeguard them from decomposition.

6.1.3 The weight of material to be weighed out for a specified volume should be calculated by taking into account the purity of the analyte and the desired concentration. The calculation should be verify by a second person for accuracy. All weighing should be performed on an analytical balance to the nearest 0.1 mg that has been standardized using standard weights and verified by a second person. The solvent used to dissolve the solute should be compatible with the protocol in which the standard is to be used; the solute should be soluble, stable, and nonreactive with the solvent. In the case of a multi-component solution, the components must not react with each other.

6.1.4 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 is to be called the primary standard and all subsequent dilutions must be traceable back to the primary standard.

6.1.5 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 when not in use. All solution standards are to be clearly labeled as to the identity

of the compound or compounds, concentration, date prepared, solvent, and initials of the preparer.

6.2 PURCHASE OF CHEMICAL STANDARDS IN SOLUTION

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

6.2.1 Contractor laboratories must maintain the following documentation to verify the integrity of the standard solutions they purchase:

6.2.1.1 Purity confirmation of the standard material.

6.2.1.2 Chromatographic and quantitative documentation that the solution standard was QC checked according to the following:

6.2.1.2.1 The Contractor must 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 below). The Contractor 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 part 6.2.1.2.2 which follows. If this is achieved, the Contractor 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. Thus the standard is certified to be within 10 percent of the target concentration.

6.2.1.2.2 If the procedure above is used, the supplier must document that the following have been achieved:

- Two solutions of identical concentration must be prepared independently from neat materials. An aliquot of the first solution must be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration 10 percent 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 percent less that the target standard. This is called the "low standard";

- Six replicate analyses of each standard (a total of 18 analyses) must be performed in the following sequence: low standard, target standard, high standard, low standard, target standard, high standard; and

- The mean and variance of the six results for each solution must be calculated using the following equations:

$$Mean = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} \qquad \text{Eq. E-1}$$

$$Variance = \frac{Y_{12} + Y_{22} + Y_{32} + Y_{42} + Y_{52} + Y_{62} - (6 \times Mean)_2}{5} \qquad \text{Eq. E-2}$$

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, Vp, is calculated using the following equation:

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + V_3}{\frac{1.21}{3}} \qquad \text{Eq. E-3}$$

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

- The test statistic must be calculated use the following equation:

$$TEST\ STATISTIC = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3}\right)^{0.5}} \qquad \text{Eq. E-4}$$

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

- The test statistic must be calculated using the following equation:

$$TEST\ STATISTIC = \frac{\left| M_2 - \left(\frac{M_1}{1.8}\right) - \left(\frac{M_3}{2.2}\right) \right|}{\left(\frac{V_p}{4}\right)^{0.5}} \qquad \text{Eq. E-5}$$

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.

- The 95 percent confidence intervals for the mean result of each standard must be calculated using the following equation:

$$\textit{INTERVAL FOR LOW STANDARD} = M_1 \pm (2.13)\left(\frac{V_P}{6}\right)^{0.5} \qquad \text{Eq. E-6}$$

$$\textit{INTERVAL FOR TARGET STANDARD} = M_2 \pm (2.13)\left(\frac{V_P}{6}\right)^{0.5} \qquad \text{Eq. E-7}$$

$$\textit{INTERVAL FOR HIGH STANDARD} = M_3 \pm (2.13)\left(\frac{V_P}{6}\right)^{0.5} \qquad \text{Eq. E-8}$$

These intervals must not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable. In any event, the laboratory is responsible for the quality of the standards employed for analyses under this contract.

6.3 REQUESTING STANDARDS FROM THE EPA STANDARDS REPOSITORY

Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, depending on availability. The Contractor can place an order for standards only after demonstrating that these standards are not available from commercial vendors in solution.

6.4 DOCUMENTATION OF THE VERIFICATION AND PREPARATION OF CHEMICAL STANDARDS

It is the Contractor's responsibility to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra, whether produced by the Contractor or purchased from chemical supply houses, must be maintained by the Contractor and may be subject to review during on-site inspection visits. Documentation of standards preparation may be required to be sent to EPA for verification of contract compliance. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the Contractors for a period of one year.

SECTION 7

METHOD SPECIFIC QC REQUIREMENTS

The purpose of this section is to outline the minimum QC operations that are an integral part of the analytical requirements associated with the determination of target analytes listed in Exhibit C, using the procedures in Exhibit D, Section 3 for metals collected on filter and analyzed by inductively coupled plasma (ICP) spectrometry, inductively coupled plasma-mass spectrometry (ICP-MS), and graphite furnace atomic absorption (GFAA) spectrometry. This section is <u>not</u> intended as a comprehensive quality control document, rather as a guide to the specific QC operations that must be considered for inorganic analyses. At a minimum, the laboratory is expected to address these operations in preparing the QAP discussed in Section 2 of this Exhibit, and the SOPs discussed in Section 3.

7.1 These operations include the following:

- Instrument Calibration;
- Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV);
- CRQL Standards;
- Linear Range Standard (LRS) Analyses (Quarterly);
- Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses;
- ICP and ICP-MS Interference Check Sample (ICS) Analyses;
- Spike Sample Analyses;
- Duplicate Sample Analyses;
- Laboratory Control Sample (LCS) Analyses;
- Performance Evaluation Sample (PES) Analyses;
- ICP Serial Dilution Analyses;
- Method Quantitation Limit (MQL) Determination; and
- Interelement Corrections for ICP and ICP-MS;
- Internal Standards for ICP-MS; and

- Furnace AA QC Analyses (Method of Standard Additions).

7.1.1 Instrument Calibration

7.1.1.1 Guidelines for instrument calibration are outlined in Exhibit D, Inorganics, Section 3.7.1 for ICP, Section 4.7.1 for ICP-MS, and Section 5.6.1 for GFAA. Each instrument shall be calibrated daily or once every 24 hours and each time the instrument is set up.

7.1.1.2 The calibration standards shall be prepared using the same type of matrix and at the same concentration as the preparation blank, following the procedures outlined in Exhibit D, Section 3.6 for ICP, Section 4.6 for ICP-MS, and Section 5.5 for GFAA.

7.1.2 Initial Calibration Verification (ICV)

7.1.2.1 Immediately after each of the ICP, ICP-MS, and GFAA systems have been calibrated, the accuracy of the initial calibration shall be verified and documented for each analyte by the analysis of EPA ICV solution(s). When resulting measurements exceed the control limits in Exhibit D, Section 3, Table D/IN-3, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. For ICP, ICP-MS, and GFAA, the ICV shall be run and reported at each wavelength and mass used for analysis.

7.1.2.2 If the ICVs are not available from EPA, or a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for regular instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

7.1.3 Continuing Calibration Verification (CCV)

7.1.3.1 To ensure calibration accuracy during each analysis run as required in Exhibit D, Section 3.7.3 for ICP, Section 4.7.3 for ICP-MS, and Section 5.6.3 for GFAA, the laboratory shall analyze a CCV solution, either prepared by EPA or Contractor-prepared, for every wavelength or mass used for analysis at the beginning of the run and after the last analytical sample.

7.1.3.2 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses, and other related operations that may affect the CCV measured result may not apply to the CCV to a greater extent than the extent applied to the associated analytical samples.

7.1.3.3 If the deviation of the CCV is greater than the control limits specified in Exhibit D, Section 3, Table D/IN-3, the analysis shall be terminated, the problem corrected, and the CCV reanalyzed only once. If the reanalysis yields a CCV value within control limits, then the preceding 10 analytical samples or all analytical samples analyzed since the last acceptable calibration verification shall be analyzed for the analytes affected. Otherwise the instrument shall be recalibrated, the calibration verified, and the affected analytical samples rerun.

7.1.4 **CRQL Standards**

7.1.4.1 To verify linearity near the CRQL for ICP, ICP-MS, and AA analysis, the Contractor shall analyze a CRQL standard (CRI and CRA) at two times the CRQL or two times the MQL, whichever is greater. The CRQL standard shall be analyzed at the beginning and end of each sample analysis run or a minimum of twice per eight hour working shift, whichever is more frequent, but not before ICV, as outlined in Exhibit D, Section 3.7.4 for ICP, Section 4.7.4 for ICP-MS, and 5.6.4 for AA. This standard must be run for every wavelength or mass used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na, and K for ICP.

7.1.4.2 Results for the analysis of the CRQL standard must be within ± 15 percent of the true value for each wavelength used for analysis. If not, the analysis shall be terminated, the problem corrected, and the analytical samples since the last acceptable CRI or CRA reanalyzed.

7.1.5 **Linear Range Standard Analysis (Quarterly)**

7.1.5.1 For all ICP and ICP-MS analyses, a linear range verification check standard (LRS) shall be analyzed and reported quarterly (every three calendar months) for each element on FORM III-AAIN as outlined in Exhibit D, Section 3.7.5 for ICP and Section 4.7.5 for ICP-MS. This standard must be run for every wavelength or mass used for analysis.

7.1.5.2 Results for the analysis of the LRS must be within ± 5 percent of the true value for each wavelength or mass used for analysis. If not, the analysis must be terminated and successive dilutions of the standard must be reanalyzed until the control limits are met. The concentration of this standard that meets the control limits is the upper limit of the instrument linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

7.1.6 **Initial and Continuing Calibration Blanks Analyses**

7.1.6.1 A calibration blank must be analyzed at each wavelength or mass used for analysis immediately after every initial and continuing calibration verification, at a frequency of 10 percent of every two

hours during the run, whichever is more frequent, as outlined in Exhibit D, Sections 3.7.6 and 3.7.7 for ICP, Sections 4.7.6 and 4.7.7 for ICP-MS, and Sections 5.6.5 and 5.6.6 for GFAA. The blank must be analyzed at the beginning of the run and after the last analytical sample.

7.1.6.2 If the absolute value of the blank result exceeds the CRQL (Exhibit C), analysis must be terminated, the problem corrected, and the continuing calibration blank (CCB) reanalyzed. If the reanalysis yields a CCB with an absolute value below the CRQL, all analytical samples analyzed since the last acceptable calibration blank must be reanalyzed. Otherwise, the instrument must be recalibrated, the calibration verified, and the affected analytical sample(s) rerun.

7.1.6.3 Each analytical sample must be bracketed by two consecutive CCBs that have been analyzed within two hours of each other with no more than 10 analytical samples run between the CCBs. The absolute value for each analyte in these two CCBs must fall below the CRQL.

7.1.7 **Preparation Blank (PB) Analysis**

7.1.7.1 At least one PB (or reagent blank) consisting of reagent water processed through each sample preparation and analysis procedure must be prepared and analyzed with every SDG or with each batch of samples digested, (whichever is more frequent), as outlined in Exhibit D, Section 3.7.8 for ICP, Section 4.7.8 for ICP-MS, and Section 5.6.7 for GFAA.

7.1.7.2 The first batch of samples in an SDG shall to be assigned to PB one, the second batch of samples to PB two, etc. Each package must contain the results of all the PB analyses associated with the samples in that SDG.

7.1.7.3 The PB is to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

7.1.7.3.1 If the absolute value of the concentration of the PB is less than or equal to the CRQL (Exhibit C), no correction of sample results is performed.

7.1.7.3.2 If any analyte concentration in the PB is above the CRQL, all associated samples containing less than ten times the blank concentration must be redigested and reanalyzed for that analyte. The sample concentrations are <u>not</u> to be corrected for the blank value.

7.1.7.3.3 If an analyte concentration in the PB is below the negative CRQL, all samples with reported analyte values below ten

times CRQL and associated with the blank must be redigested and reanalyzed.

7.1.7.3.4 The values for the PB must be recorded in µg/L.

7.1.8 **ICP and ICP-MS Interference Check Sample Analysis**

7.1.8.1 To verify interelement and background correction factors, the Contractor must analyze and report the results for an ICP and ICP-MS Interference Check Sample (ICS). The ICS must be analyzed at the beginning and end of each analysis run or a minimum of twice per eight hour working shift, whichever is more frequent, but not before the ICV (see Exhibit D, Section 3.7.9 for ICP and Section 4.7.9 for ICP-MS). The ICP and ICP-MS ICS must be obtained from EPA (EMSL-LV), if available, and analyzed according to the instructions supplied with the ICS.

7.1.8.2 The ICS consists of Solutions A and AB that must contain both analytes and potential interferents. An ICS analysis consists of analyzing the solution for all wavelengths or masses used for each analyte reported by ICP.

7.1.8.3 Results for the ICP analyses of Solutions A and AB during the analytical runs must be within ± 20 percent of the true value for the analytes included in the ICS. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the analytical samples analyzed since the last acceptable ICS. If true values for analytes contained in the ICS and analyzed by ICP and ICP-MS are not supplied with the ICS, the mean concentration for each analyte must be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination must be made during an analytical run where the results for the previously supplied EPA ICS met all contract specifications. Additionally, the result of this initial mean determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

7.1.9 **Spike Sample Analysis**

7.1.9.1 The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the sample preparation (i.e., digestion, and/or distillation). At least one spike sample analysis must be performed on each group of samples for each SDG, according to Exhibit D, Section 3.7.10 for ICP, Section 4.7.10 for ICP-MS, and Section 5.6.8 for GFAA and Exhibit D, Table D/IN-3.

7.1.9.2 If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations must be performed using the results of the sample designated as the "original sample" (see Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks cannot be used for the spike sample analysis.

7.1.9.3 If two analytical methods are used to obtain the reported values for the same analyte within an SDG, spike samples must be run by each method used.

7.1.9.4 If the spike recovery is not within the limits of 75-125 percent, the data for all of the samples received associated with that spike sample and determined by the same analytical method must be flagged with the letter "N", unles the sample concentration is more than four times the spike concentration.

7.1.10 Duplicate Sample Analysis

7.1.10.1 One duplicate sample must be analyzed from each group of samples for each SDG, as outlined in Exhibit D, Section 3.7.11 for ICP, Section 4.7.11 for ICP-MS, and Section 5.6.9 for GFAA. Duplicates cannot be averaged in reporting results.

7.1.10.2 Samples identified as field blanks cannot be used for duplicate sample analysis. EPA may require that a specific sample be used for duplicate sample analysis. If two analytical methods are used to obtain the reported values for the same element for a SDG (e.g., ICP and GFAA), duplicate samples must be run by each method used.

7.1.11 Laboratory Control Sample Analysis

7.1.11.1 The Laboratory Control Sample (LCS) must be analyzed for each analyte using the same sample preparation, analytical methods and QA/QC procedures employed for the EPA samples received, as outlined in Exhibit D, Section 3.7.12 for ICP, Section 4.7.12 for ICP-MS, and Section 5.6.10 for GFAA.

7.1.11.2 The EPA-provided LCS must be prepared and analyzed using each of the procedures applied to the samples received. If the EPA LCS is unavailable, other EPA quality assurance check samples or other certified materials may be used. One LCS must be prepared and analyzed for every group of samples in a SDG or for each batch of samples prepared, whichever is more frequent.

7.1.11.3 If the results of the LCS are not within the control limits established by EPA, the analyses must be terminated, the problem corrected, and the samples associated with the LCS prepared again and

reanalyzed. A control limit of ± 20 percent of the true value must be used if no control limits are provided with the LCS solution.

7.1.12 **Performance Evaluation Sample (PES)**

7.1.12.1 The Performance Evaluation Sample (PES) will assist the Agency in monitoring Contractor performance. The laboratory will not be informed in advance of the analytes in the PES or their concentration.

7.1.12.2 The Contractor must prepare, analyze and report the results of one PES per each SDG, if available, as outlined in Exhibit D, Section 3.

7.1.12.3 Prepare the PES using the procedure described in the sample preparation section of Exhibit D, Section 2, and analyze the PES using the methods described in Exhibit D for the analysis of EPA field samples. All contract required QC must also be met.

7.1.12.4 The Contractor must demonstrate acceptable performance for analyte identification and quantification. If the Contractor achieves a score of less than 75 percent, the Agency may take but is not limited to the following actions: Show Cause and/or Cure Notice, reduction of the number of samples shipped to the laboratory, suspension of sample shipment, a site visit, a full data audit, and/or require the laboratory to analyze a remedial PES.

7.1.13 **ICP Serial Dilution Analyses**

7.1.13.1 The ICP serial dilution analyses is performed to check for the presence of matrix interferences for all ICP instruments at all wavelengths used for each analyte reported by ICP. A serial dilution is not required for ICP-MS determinations. One serial dilution analysis must be performed for each SDG. Identified field blanks may not be used for serial dilution analysis.

7.1.13.2 The serial dilution analysis is performed by diluting a prepared sample aliquot five-fold (1:4). The dilution must be prepared on an analyte-by-analyte bases. The serial dilution is the dilution of the sample, or an aliquot of sample, that contains a concentration level of the analyte within the linear range.

7.1.13.3 If the analyte concentration in the field sample is sufficiently high (minimally factor of 50 above the the MQL), the serial dilution must agree within 10 percent of the initial sample concentration determination after correction for the five fold dilution. If the serial dilution analysis for one or more analyte is not within the 10 percent criteria, a chemical or physical interference effect must be suspected and the data for all analytes exceeding the limit in the samples associated with that serial dilution must be flagged with an "E" on FORMs I-AAIN and XI-AAIN.

7.1.13.4 The values for the initial sample and serial dilution must be recorded on FORM XI-AAIN for all analysis systems, as indicated in Exhibit B, Section 3.

7.1.14 **Method Quantitation Limit Determination**

7.1.14.1 The Method Quantitation Limit (MQL) (in µg/L) must be determined for each instrument used, within 30 days prior to the start of any contract analysis and at least quarterly (every 3 calendar months), as outlined in Exhibit D, Section 3.7.14 for ICP, Section 4.7.15 for ICP-MS, and Section 5.6.13 for GFAA. The MQL must be equal to or less than CRQLs (µg/L) specified in Exhibit C.

7.1.14.2 The MQLs must be determined by multiplying by three the average of the standard deviations obtained on three nonconsecutive days (each analyte in reagent water) at a concentration three times the MQL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). MQLs must be determined and reported for each wavelength or mass used in the analysis of the samples.

7.1.14.3 The quarterly determined MQL for an instrument must always be used as the MQL for that instrument during that quarter. If the instrument is adjusted in any way that may affect the MQL, the MQL for that instrument must be redetermined and the results submitted for use as the established MQL for that instrument for the remainder of the quarter.

7.1.14.4 MQLs must be reported for each instrument and submitted with each data package. If multiple instruments are used for the analysis of an analyte within a SDG, the highest MQL for the analyte must be used for reporting concentration values for that SDG.

7.1.14.5 The MQL for each analyte must be less than or equal to the CRQL. An exception is granted if the analyte concentration in the samples is greater than or equal to five times the reported MQL.

7.1.15 **Interelement Corrections for ICP and ICP-MS**

7.1.15.1 The ICP and ICP-MS interelement correction factors must be determined within three months prior to beginning sample analyses under this contract and at least annually thereafter, as outlined in Exhibit D, Section 3.7.16 for ICP and Section 4.7.16 for ICP-MS. Correction factors for spectral interference must be determined at all wavelengths or masses used for each analyte reported by ICP and ICP-MS.

7.1.15.2 The correction factors must be determined under the same instrument conditions used for sample analysis. If the instrument was adjusted in any way that may affect the ICP and ICP-MS interelement correction factors, the factors must be redetermined and the results submitted for use. The interelement factors determination must be reported for all ICP and ICP-MS analytes, for each instrument used to generate data in the SDG.

7.1.16 **Internal Standard for ICP-MS**

7.1.16.1 The internal standard analyses is performed to check for the presence of physical interferences and correct for them for analyses performed by ICP-MS.

7.1.16.2 A minimum of three internal standards, listed in Table D/IN-7, Exhibit D, bracketing the mass range must be used. The intensity level of an internal standard for each sample, duplicate, spike analysis, and PES must agree within ± 50 percent of the intensity level of the internal standard of the initial calibration blank standard solution (SO). If not, the sample must be reanalyzed after performing a five fold (1:4) dilution. If the intensity level percent difference, %D, remains greater than 50 percent, a physical interference must be suspected, and the data on FORM XV-AAIN must be flagged with an "E." The analytes affected by the interference must be listed in the comment section on the appropriate FORMs I-AAIN, VI-AAIN, VII-AAIN, and VIII-AAIN.

7.1.16.3 The intensity levels of the internal standards for the CCV and CCB solutions must agree within ± 20 percent of the intensity level of the internal standard of the initial calibration blank standard solution (SO). If not, the analysis must be terminated, the problem corrected and the CCV/CCB reanalyzed only once. If the first reanalysis yields a CCV/CCB %D value within control limits, then the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification must be reanalyzed for the analytes affected. Otherwise, the instrument must be recalibrated, the calibration verified and the affected analytical samples rerun in the context of a new run.

7.1.16.4 The intensity level of the internal standards for the ICV and ICB solutions must agree within ± 20 percent of the intensity level of the internal standard of the initial calibration blank standard solution (SO). If not, the analysis must be terminated, the problem corrected, and a new analytical run must be started.

7.1.16.5 The values for the Internal Standard Percent Difference (%D) must be reported for each ICP-MS analysis on FORM XV-AAIN as indicated in Exhibit B, Section 3.

7.1.17 **Furnace AA QC Analyses (Method of Standard Additions)**

7.1.17.1 Because of the nature of the GFAA techniques, the special procedures summarized in Figure E-2 will be required for quantitation. (These procedures do not replace those in Exhibit D, Section 5.6.12 of this document, but supplement the guidance provided therein.)

7.1.17.1.1 All analyses must fall within the calibration range. In addition, all analyses, except during full Method of Standard Additions (MSA), will require duplicate injection must be reported in raw data as well as the average intensity and concentration values. A maximum of 10 full sample analyses to a maximum of 20 injections may be performed between each consecutive calibration verification and blank. The raw data package must contain intensity and concentration values for both injections, the average value and the relative standard deviation (RSD) or coefficient of variation (CV). For concentrations greater than CRQL, the duplicate injection readings must agree within 15 percent RSD or CV, or the analytical sample must be rerun once (i.e., two additional injections). If the readings are still out, flag the value reported on FORM I-AAIN with an "M." The "M" flag is required for the analytical spike as well as the sample. If the analytical spike for a sample requires an "M" flag, the flag must be reported on FORM I-AAIN for that sample.

If the preparation blank analytical spike recovery is out of control 85-115 percent, the spiking solution must be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem, respike and reanalyze all analytical samples associated with that blank.

7.1.17.1.2 All GFAA analyses for each analytical sample, including those requiring an "M" flag, will be require at least an analytical spike to determine if the MSA will be required for quantitation. The analytical spike will be required to be at a concentration (in the sample) of two times the CRQL (except for lead which must be at 20 µg/L). This requirement for an analytical spike will include the LCS and the preparation blank. The LCS must be quantitated from the calibration curve and corrective action, if needed, taken accordingly. MSAs are not to be performed on the LCS or preparation blank, regardless of spike recovery results. If the preparation blank analytical spike recovery is out of the control limits 85-115 percent, the spiking solution must be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem and respike and reanalyze all analytical samples associated with the blank. An analytical spike is not required on the pre-digestion spike sample.

7.1.17.1.3 The analytical spike of a sample must be run immediately after that sample. The percent recovery (%R) of the spike, calculated by the same formula as Spike Sample Analyses (see Spike Sample Analysis, this Exhibit), will then determine how the sample will be quantitated, as follows:

7.1.17.1.3.1 If the spike recovery is < 40 percent, the sample must be diluted by a factor of five to ten and rerun with another spike. This step must only be performed once. If after the dilution the spike recovery is still < 40 percent, report data from the initial undiluted analysis and flag with an "E" to indicate interference problems.

7.1.17.1.3.2 If the spike recovery is ≥ 40 percent and the sample absorbance or concentration is < 50 percent of the spike, report the sample results to the MQL. If the spike is less than 85 percent or greater than 115 percent, flag the result with a "W."

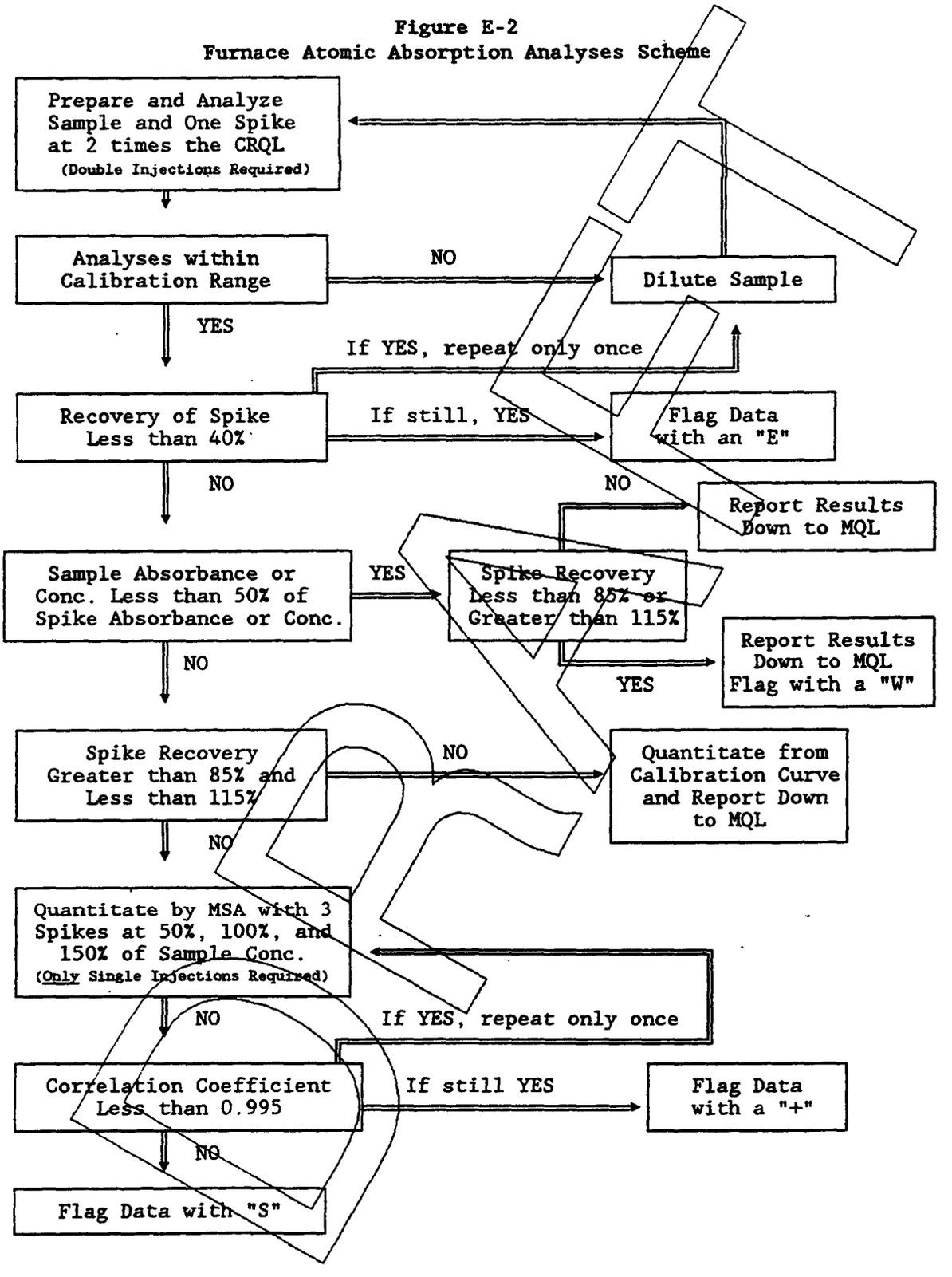
7.1.17.1.3.3 If the sample absorbance or concentration is < 50 percent of the spike and the spike recovery is at or between 85 percent and 115 percent, the sample must be quantitated directly from the calibration curve and reported down to the MQL.

7.1.17.1.3.4 If the sample absorbance or concentration is ≤ 50 percent of the spike and the spike recovery is < 85 percent or greater than 115 percent, the sample must be quantitated by MSA.

7.1.17.1.3.5 The following procedures will be incorporated into MSA analyses.

- Data from MSA calculations must be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.
- The sample and three spikes must be analyzed consecutively for MSA quantitation (the "initial" spike run data is specifically excluded from use in the MSA quantitation). Only single injections are required for MSA quantitation.
- Each full MSA counts as two analytical samples towards determining 10 percent QC frequency (i.e., five full MSAs can be performed between calibration verifications).
- For analytical runs containing only MSAs, single injections can be used for QC samples during that run.
- Spikes must be prepared such that:

Figure E-2
Furnace Atomic Absorption Analyses Scheme



1) Spike 1 is approximately 50 percent of the sample concentration.

2) Spike 2 is approximately 100 percent of the sample concentration.

3) Spike 3 is approximately 150 percent of the sample concentration.

- The data for each MSA analysis must be clearly identified in the raw data documentation (spike concentrations measured intensities or concentration, x-intercept, y-intercept and correlation coefficient). The results shall be reported on FORM X-AAIN. Reported values obtained by MSA must be flagged on FORM I-AAIN with the letter "S" if the correlation coefficient (r) is greater than or equal to 0.995.

- If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analysis must be repeated once. If the correlation coefficient is still less than 0.995, report the results on FORM I-IN from the run with the best "r" and flag the result with a "+." On FORM X-AAIN report the results of both MSA analysis and flag with a "+" for any MSA result that yields a correlation coefficient less than 0.995.

SECTION 8

REGIONAL DATA REVIEW

Contract laboratory data are generated to meet the specific needs of the Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end-user, based upon functional aspects of data quality. General guidelines for data review have been developed jointly by the Regions and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problems under investigation and the Regional response appropriate to the specific circumstances.

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 SMO, which is designed to identify contractual discrepancies, and the review done at EMSL/LV, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for program and laboratory administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

SECTION 9

LABORATORY EVALUATION SAMPLES

Although intra-laboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in inter-laboratory comparison studies conducted by the EPA. Results from the analysis of these laboratory evaluation samples will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

Sample sets may be provided to participating Contractors as frequently as on an SDG-by-SDG basis as a recognizable QC sample of known composition; as a recognizable QC sample of unknown composition; or not recognizable as a QC material. The laboratory evaluation samples may be sent either by the Regional client or the National Program Office, and may be used for contract action.

Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

At a minimum, the results are evaluated for parameter identification, quantification, and sample contamination. Confidence intervals for the quantification of target compounds are based on reported values using population statistics. EPA may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample. Normally, a fraction of the compounds spiked into the sample are not specifically listed in the contract.

The Contractor shall provide a response regarding its performance on laboratory evaluation samples as specified below:

- No Response is required for a score of 90 or above.
- For a score of 89 to 75, the Contractor shall describe the deficiency(ies) and the corrective action(s) taken in a letter to the APO, TPO, and EMSL/LV, within 14 days of receipt of notification from EPA.
- For a score of less than 75, the Contractor shall be notified by the APO or TPO concerning the remedy for their unacceptable performance. A Contractor may expect, but EPA is not limited to, the following actions: reduction of the number of samples sent under the contract; suspension of sample shipment to the Contractor; a follow-up site visit; a full data audit; analysis of remedial PE samples; and/or contract sanction, such as a Cure Notice.

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

SECTION 10

ON-SITE LABORATORY EVALUATIONS

At a frequency dictated by a contract laboratory's performance, the APO, TPO, or their authorized representative will conduct on-site laboratory evaluations. On-site laboratory evaluations are conducted 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 Evaluation and an Evidentiary Audit.

10.1 QUALITY ASSURANCE ON-SITE EVALUATION

10.1.1 Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures. The Contractor should expect items to be monitored will include but not be limited to the following items:

10.1.1.1 Size and appearance of the facility.

10.1.1.2 Quantity, age, availability, scheduled maintenance, and performance of instrumentation.

10.1.1.3 Availability, appropriateness, and utilization of SOPs.

10.1.1.4 Staff qualifications, experience, and personnel training programs.

10.1.1.5 Reagents, standards, and sample storage facilities.

10.1.1.6 Standard preparation logbooks and raw data.

10.1.1.7 Bench sheets and analytical logbook maintenance and review.

10.1.1.8 Review of the Contractor's sample analysis/data package inspection procedures.

10.1.2 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, laboratory evaluation sample scores, Regional review of data, Regional QA materials, and data trend reports.

10.2 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. The evidence audit is comprises the following three activities.

10.2.1 Procedural Audit

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

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

10.2.2 Written SOPs Audit

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

10.2.3 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:

- Accuracy of the document inventory;
- Completeness of the file;
- Adequacy and accuracy of the document numbering system;
- Traceability of sample activity;
- Identification of activity recorded on the documents; and

- Error correction methods.

10.3 DISCUSSION OF THE ON-SITE TEAM'S FINDINGS

The quality assurance and evidentiary auditors discuss their findings with the APO/TPO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

10.4 CORRECTIVE ACTION REPORTS IN RESPONSE TO QUALITY ASSURANCE AND EVIDENTIARY AUDIT REPORTS

10.4.1 Following an on-site evaluation, quality assurance and evidentiary audit reports which discuss deficiencies found during the on-site evaluation will be forwarded to the Contractor. The Contractor must discuss the corrective actions taken to resolve the deficiencies discussed during the on-site visit and discussed in the on-site reports in a letter to the APO, TPO, EMSL/LV (response to the quality assurance report) and NEIC (response to the evidentiary report) within 14 days of receipt of the finding or within the time agreed upon between the APO/TPO and the Contractor. If SOPs are required to be written or SOPs are required to be amended, the Contractor must provide the SOPs to the TPO, EMSL/LV (quality assurance/technical SOPs) and NEIC (evidentiary SOPs) within 30 days of receipt of the finding or within the time agreed upon between the APO/TPO and the Contractor.

10.4.2 If the Contractor fails to take appropriate corrective action to resolve the deficiencies discussed in the on-site reports, a Contractor may expect, but the Agency is not limited to, the following actions: reduction of the number of samples sent under the contract; suspension of sample shipment to the Contractor; a follow-up site visit; a full data audit; analysis of remedial PE samples; and/or contract sanction, such as a Cure Notice.

SECTION 11

DATA MANAGEMENT

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 should 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.

11.1 DATA ENTRY

Data manually entered from hard copy must be checked for accuracy 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 must be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

11.2 CORRECTIONS AND UPDATES

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

11.2.1 Justification or rationale for the change.

11.2.2 Initials of the person making the change or changes. Data changes must be implemented and reviewed by a person or group independent of the source generating the deliverable.

11.2.3 Change documentation must be retained according to the schedule of the original deliverable.

11.2.4 Resubmitted diskettes or other deliverables must be reinspected as a part of the laboratory's internal inspection process prior to resubmission. The entire deliverable, not just the changes, must be inspected.

11.2.5 The Laboratory Manager must approve changes to originally submitted deliverables.

11.2.6 Documentation of data changes may be requested by laboratory auditors.

11.3 LIFE CYCLE MANAGEMENT

Life cycle management procedures must be applied to computer software systems developed by the laboratory to be used to generate and edit contract deliverables. Such systems must be thoroughly tested and documented prior to utilization:

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

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

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

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

11.4 PERSONNEL IDENTIFICATION REQUIREMENTS

Individual(s) responsible for the following functions must be identified:

11.4.1 System operation and maintenance including documentation and training.

11.4.2 Data base integrity, including data entry, data updating and quality control.

11.4.3 Data and system security, backup and archiving.

SECTION 12

REFERENCES

Office of Monitoring Systems and Quality Assurance, U.S. Environmental Protection Agency, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80, December 1980.

Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Third Edition, SW-846, November 1986.

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Environmental Protection Agency, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule", 40 CFR Part 136, Federal Register, Vol. 49, No. 209., pp 43234-43442, October 26, 1984.

American Chemical Society Committee on Environmental Improvement, and Subcommittee on Environmental Analytical Chemistry, "Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry", Analytical Chemistry, Volume 52, Number 14, December 1980.

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EXHIBIT F

EVIDENTIARY REQUIREMENTS

December, 1991

EXHIBIT F

EVIDENTIARY REQUIREMENTS

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SECTION 1

SAMPLE CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

1.1 SAMPLE IDENTIFICATION

1.1.1 To assure traceability of samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

1.1.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA sample number.

1.2 CHAIN-OF-CUSTODY PROCEDURES

1.2.1 Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented.

1.2.2 A sample is under custody if the following applies:

1.2.2.1 It is in your possession.

1.2.2.2 It is in your view after being in your possession.

1.2.2.3 It was in your possession and you locked it up.

1.2.2.4 It is in a designated secure area (secure areas shall be accessible to authorized personnel only).

1.3 SAMPLE RECEIVING PROCEDURES

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available. The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.3 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

1.3.4.1 Airbills or airbill stickers.

1.3.4.2 Custody seals.

1.3.4.3 EPA custody records.

1.3.4.4 EPA traffic reports or SAS packing lists.

1.3.4.5 Sample tags.

1.3.5 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

1.3.6 The Contractor shall contact SMO to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

1.3.7 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

1.3.8 The following information shall be recorded on appropriate Form AADC-1 by the sample custodian or his/her representative as samples are received and inspected:

1.3.8.1 Condition of the shipping container.

1.3.8.2 Presence or absence and condition of custody seals on shipping and/or sample containers.

1.3.8.3 Custody seal numbers, when present.

1.3.8.4 Condition of the sample bottles.

1.3.8.5 Presence or absence of airbills or airbill stickers.

1.3.8.6 Airbill or airbill sticker numbers.

1.3.8.7 Presence or absence of EPA custody records.

1.3.8.8 Presence or absence of EPA traffic reports or SAS packing lists.

1.3.8.9 Presence or absence of sample tags.

1.3.8.10 Sample tag identification numbers cross-referenced to the EPA sample numbers.

1.3.8.11 Verification of agreement or non-agreement of information recorded on shipping documents and sample containers.

1.3.8.12 Problems or discrepancies.

1.4 SAMPLE TRACKING PROCEDURES

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

SECTION 2

DOCUMENT CONTROL PROCEDURES

The goal of the laboratory document control program is to assure that all documents for a specified SDG will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include but not be limited to logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to the EPA or are available upon request from the EPA prior to the delivery schedule.

2.1 PREPRINTED LABORATORY FORMS AND LOGBOOKS

2.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents which is directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to the EPA, the laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than

government agency or commercial client names to preserve the confidentiality of commercial clients.

2.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. Unused portions of documents shall be "crossed" out.

2.2 CONSISTENCY OF DOCUMENTATION

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

2.2.2 All copies of laboratory documents shall be complete and legible.

2.2.3 Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

2.3 DOCUMENT NUMBERING AND INVENTORY PROCEDURES

2.3.1 In order to provide document accountability of the completed analysis records, each item in a CSF shall be inventoried and assigned a serialized number as described in Exhibit B, Section 2.

CSF # - Region - Serialized number (For example: 75-2-0240).

2.3.2 All documents relevant to each SDG, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc., shall be inventoried.

2.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the EPA. The DCO shall place the sample tags in plastic bags in the file. Figure E-1 of Exhibit E is an example of a document inventory.

2.4 STORAGE OF EPA FILES

The Contractor shall maintain EPA laboratory documents in a secure location.

2.5 SHIPPING DATA PACKAGES AND CSF

2.5.1 The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

2.5.2 The Contractor shall purge the CSF deliverable to the appropriate EPA Region 180 days after the report submission.

2.5.3 A copy of the transmittal letter for the CSF will be sent to NEIC and SMO.

2.5.4 The Document Control form is used to document the receipt and inspection of shipping containers and samples. The Contractor shall submit one original FORM AADC-1 for each shipping container.

2.5.5 The Contractor shall sign and date the airbill (if present), examine the shipping containers, record the presence or absence of custody seals and their conditions.

2.5.6 The Contractor shall note any problems with the samples and follow the instructions explained in Exhibit B, Sample Log-In Sheet.

2.5.7 The Contractor shall submit a completed Document Control Form with each SDG package.

SECTION 3

STANDARD OPERATING PROCEDURES

The Contractor must have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, tracking the analysis of samples, and assembly of completed data.

3.1 SPECIFICATIONS FOR WRITTEN STANDARD OPERATING PROCEDURES

3.1.1 An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation.

3.1.2 The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits. The Contractor must have written standard operating procedures (SOPs) for:

3.1.2.1 Sample receipt and logging.

3.1.2.2 Sample storage.

3.1.2.3 Preventing sample contamination.

3.1.2.4 Security for laboratory and samples.

3.1.2.5 Traceability of standards.

3.1.2.6 Maintaining instrument records and logbooks.

3.1.2.7 Sample analysis and data control systems.

3.1.2.8 Glassware cleaning.

3.1.2.9 Technical and managerial review of laboratory operation and data package preparation.

3.1.2.10 Internal review of contractually-required quality assurance and quality control data for each individual data package.

3.1.2.11 Sample analysis, data handling, and reporting.

3.1.2.12 Chain-of-Custody.

3.1.2.13 Document control, including Case file preparation.

3.1.3 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.1.4 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.1.4.1 Presence or absence of EPA chain-of-custody forms.

3.1.4.2 Presence or absence of airbills or airbill stickers.

3.1.4.3 Presence or absence of EPA Traffic Reports or SAS packing lists.

3.1.4.4 Presence or absence of custody seals on shipping and/or sample containers and their condition.

3.1.4.5 Custody seal numbers, when present.

3.1.4.6 Presence or absence of sample tags.

3.1.4.7 Sample tag ID numbers.

3.1.4.8 Condition of the shipping container.

3.1.4.9 Condition of the sample container.

3.1.4.10 Verification of agreement or nonagreement of information on receiving documents and sample containers.

3.1.4.11 Resolution of problems or discrepancies with the SMO.

3.1.4.12 The definition of any terms used to describe sample condition upon receipt.

3.1.5 The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.1.6 The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

3.1.6.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.1.6.2 A description of the documentation used to record instrument calibration and other QA/QC activities.

3.1.6.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.1.7 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.1.8 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the EPA sample number.

3.1.9 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions. The Contractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.

3.1.10 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure.

3.1.11 The Contractor shall have written SOPs for laboratory safety.

3.1.12 The Contractor shall have written SOPs for cleaning of glassware used in preparing and analyzing samples under this contract.

3.1.13 The Contractor shall have SOPs for traceability of standards used in sample analysis QA/QC.

3.2 HANDLING OF CONFIDENTIAL INFORMATION

3.2.1 A Contractor conducting work under this contract may receive EPA-designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

3.2.2 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

3.2.3 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

EXHIBIT G

GLOSSARY OF TERMS

DRAFT

EXHIBIT G

GLOSSARY OF TERMS

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

$$A = \frac{I_{solvent}}{I_{solution}} = \log\frac{I_o}{I} \qquad \text{Eq. G-1}$$

where:

I = Radiation intensity.

Aliquot - A measured portion of a field sample taken for analysis.

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

Analysis Group - An analysis group is a set of no more than twenty analytical samples (as defined below) for the purpose of method Quality Assurance/Quality Control (QA/QC), such that the QA/QC required by Exhibit E is, at a minimum, prepared and analyzed at a frequency of once per twenty analytical samples.

Analysis Replicate - A single analytical sample prossed through the analytical preparation method and analyzed in replicate.

Analysis Run - The actual instrumental analysis of the sample preparations from the time of instrument calibration through the running of the final CCV. All sample preparation analyses during the analysis run are subject to the QC protocols set forth in Exhibit E of this contract unless otherwise specified in the individual methods.

Analysis Spike Sample - An analytical sample taken through the analytical preparation method and then spiked prior to analysis.

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

Analytical Preparation - An analytical sample taken through the analytical preparation method. Also referred to as preparation or sample preparation.

Analytical Preparation Method - A method (digestion, dilution, extraction, fusion, etc.) used to dissolve or otherwise release the analyte(s) of interest from its matrix and provide a final solution containing the analyte which is suitable for instrumental or other analysis methods.

Analytical Sample - Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, interference check samples (ICS), CRQL standard for AA (CRA), CRQL standard for ICP (CRI), laboratory control sample (LCS), preparation blank (PB) and linear range analysis sample (LRS).

Analytical Spike - A post-digestion spike to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the prepared sample. The unspiked sample aliquot must compensate for any volume change in the spike samples by addition of ASTM Type II water to the unspiked sample aliquot. The volume of the spiking solution added must not exceed 10% of the analytical sample volume.

ASTM Type II Water - Distilled water with a conductivity of less than 1.0 μmho/cm at 25°C. For additional specifications refer to ASTM D1193-77, "Standard Specification for Reagent Water".

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

Average Intensity - The average of two different injections (exposures).

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

Batch - A group of samples prepared at the same time.

Calibration - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. Calibration procedures differ for the various methods included in this SOW. Refer to the method of interest for a definition specific to that method.

Calibration Blank - For Inorganics, a volume of deionized distilled water acidified with nitric and/or hydrochloric acid, and containing all of the reagents and in the same concentration as those used in the analytical sample preparation. This blank is not subjected to the preparation method but is produced synthetically.

Calibration Standards - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical

curve). The solutions are not subjected to the preparation method but contain the same matrix as the sample preparations to be analyzed.

Calibration Verification (CCV) - A single element or multi-element standard solution prepared by the analyst to be used to verify the stability of the instrument calibration with time and the instrument performance during the analysis of samples. The CCV can either be one or more of the calibration standards and/or an ICV. However, all analyte elements being measured by the particular system must be represented in this standard and the standard must have the same matrix as the samples. The CCV should have a concentration in the middle of the calibrated range. Analytical standard run every 10 analytical samples or every 2 hours, whichever is more frequent, to verify the calibration of the analytical 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.

Coefficient of Variation (CV) - The standard deviation as a percent of the arithmetic mean.

Continuing Calibration - Analytical standard run at least every 12 hours to verify the calibration of the analytical system.

Contract Required Quantitation Limit (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work. Generally defined as 3.3 (or more) times the standard deviation of seven replicate analyses of the method blank.

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

Correlation Coefficient - A number (r) which indicates the degree of dependence between two variables (e.g., concentration - absorbance). The more dependent they are the closer the value to one. Determine on the basis of the least squares line.

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

DDI - Deionized Distilled water.

Digestion Log - An official record of the sample preparation (digestion).

Dissolved Metals - Analyte elements which have not been digested prior to analysis and which will pass through a 0.45 μm filter.

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

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

EBCDIC - Extended Binary Coded Decimal Interchange Code.

Exposure - A full measurement of an emission line of an analyte from which the concentration of the analyte can be determined in the excitation system in a manner that meets the systems detection limit. It is also referred to as a peak scan.

External Standards - Target analytes analyzed at a known concentration prior to sample analysis, to determine response factors.

Field Blank - Any sample submitted from the field identified as a blank.

Field Sample - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

Flame Atomic Absorption (AA) - Atomic absorption which utilizes flame for excitation.

Graphite Furnace Atomic Absorption (GFAA) - Atomic absorption which utilizes a graphite cell for excitation.

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

Independent Standard - A contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

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

In-House - At the Contractor's facility.

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

Initial Calibration Verification (ICV) - Solution(s) obtained from the EPA or prepared from stock standard solutions, metals or salts obtained from a source separate from that (those) utilized to prepare the calibration standards, and that have known concentration values. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration of the calibration standards and the adequacy of the instrument calibration. The ICV is not restricted to preparations made by official agencies when EPA sources are not available but should be traceable to an NBS or other certified standard.

Instrument Blank - For inorganics, acidified water analyzed to determine whether carry-over from a previous sample has occurred.

Instrument Detection Limit (IDL) - Determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

Interference Check Sample - Solution(s) containing both interfering and analyte elements of known concentrations that can be used to verify background and interelement correction factors. This solution must also contain the same matrix as the analytical preparations.

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

Internal Standards - Analytes added to every standard, blank, and sample at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target analysis.

Laboratory - Synonymous with Contractor as used herein.

Laboratory Control Sample - Aliquot spiked with known concentration of specific analytes and subjected to the entire analytical procedure in order to monitor method and contractor performance.

Laboratory Receipt Date - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

Linear Range - The concentration range over which the analytical curve remains linear. The range of the instrument for a specific analyte, as determined using calibration standards. The upper limit of this linear range (determined at each analysis) is the highest concentration calibration standard that has a determined value within 10% of the known value.

Mass Spectral Interference - Defined as the inability to detect the internal standard quantification ion due to presence of high levels of mass spectral "noise" at the same mass.

Matrix - The predominant material of which the sample to be analyzed is composed.

Matrix Modifier - Salts used in AA to lessen the effects of chemical interferents, viscosity, and surface tension.

Matrix Spike - Aliquot of a sample fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Method Blank - A solution produced by performing the analytical preparation method without the addition of a sample. The solution thus contains the same concentrations of reagents as all other analytical preparations plus any impurities derived from the preparation process. For preparations containing reagents of variable concentrations, the method blank should match the maximum reagent concentration used in the sample preparation(s).

Method of Standard Additions (MSA) - The addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). MSA may counteract matrix effects; it will not counteract spectral effects. Also referred to as Standard Addition.

Method Detection Limit (MDL) - The chemical concentration that produces a signal, due to an analyte, which is equal to the student t_{99} times the standard deviation of a series of measurements on at least seven separate method blanks. In practice, a method detection limit will be substantially higher than an instrumental detection limit. The method detection limit for metals is t_{99} times the standard deviation of seven method blank analyses. Of course, all spectral background techniques must be operative and the same integration times must be utilized as when actual samples are analyzed.

MS-SCAN - The ICP is coupled to a mass selective detector where the instrument is programmed to acquire all mass for target analytes and to disregard all others.

Performance Evaluation (PE) Sample - A sample of known composition provided by EPA for Contractor analysis. Used by EPA to evaluate Contractor performance.

Preparation Blank (also reagent, procedure, and method blank) - For inorganics, an analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed).

Preparation Log - An official record of the sample preparation (digestion).

Protocol - A compilation of the 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).

Qualitative Accuracy - The ability of an analytical system to correctly identify compounds.

Quality Control (QC) Check Sample - A sample containing known concentrations of analytes that is analyzed by a laboratory to demonstrate that it can obtain acceptable identifications and measurements with procedures to be used to analyze environmental samples containing the same or similar analytes. Analyte concentrations are known by the analyst. Preparation of the QC check sample by a laboratory or standard supplier other than the laboratory performing the analysis is highly desirable.

Quality Control Set - A group of 10 analytical samples plus the CCVs and CCBs that bracket those samples.

Quantitative Accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

Recovery - A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

$$\%\,Surrogate\ Recovery = \frac{Measured\ value}{spiked\ value} \times 100\% \qquad \text{Eq. G-2}$$

Relative Response Factor (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the

calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \qquad \text{Eq. G-3}$$

where:

A = Area of the characteristic ion measured;
C = Concentration;
is = Internal standard; and
x = Analyte of interest.

Resolution - Also termed separation, the separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Retention Time (RT) - The time to elute a specific chemical from a chromatographic column for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until its maximum concentration appears at the detector.

Retention Time Window - Retention time window is determined for each analyte of interest and is the time from injection to elution of a specific chemical from a chromatographic column. The window is determined by three injections of a single component standard over a 24-hour period as plus or minus three times the standard deviation of the absolute retention time for that analyte.

Rounding Rules - If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

- If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

- If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

- If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

NOTE: See forms instructions (Exhibit B) for exceptions.

Run - A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract Statement of Work.

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

Sample Delivery Group (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A SDG is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 samples within a Case; or
- Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

NOTE: Samples may be assigned to SDGs by sample collection method (i.e., all Tenax® tubes in one SDG, all canisters in another).

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

Sample Recovery (SR) - The quantity of a component measured in a sample as compared to a known quantity.

Sensitivity - The slope of the analytical curve, i.e., functional relationship between emission intensity and concentration.

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

Standard Analysis - An analytical determination made with known quantities of target compounds; used to determine response factors.

Stock Solution - A standard solution which can be diluted to derive other standards.

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

Total Metals - Analyte elements which have been digested prior to analysis.

Traffic Report (TR) - An EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which is used for documenting sample condition and receipt by the laboratory.

Twelve-Hour Time Period - The twelve (12) hour time period for GC/MS system tuning, standards calibration (initial or continuing calibration) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of compliant tune. The time period ends after 12 hours has 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.

Viewing Area Adjustment Standard: A solution containing a standard of a strong atom line (i.e., Cu) and a weak ion line (e.g., Ba) used to verify the proper adjustment of the observation height in the plasma for metals analysis by ICP (see Method 200.62-B for details).

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