USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

ANALYSIS OF
LOW CONCENTRATION ORGANIC

OLC03.2
December 2000

STATEMENT OF WORK

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EXHIBIT A

SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

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1.0 PURPOSE

The purpose of the Low Concentration (Water) Organic analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (USEPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other USEPA Program Offices that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

The organic analytical service provides a contractual framework for laboratories to apply USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 50 volatile, 65 semivolatile, and 28 pesticide/Aroclor target compounds in water samples. The analytical service provides the methods to be used and the specific contractual requirements by which USEPA will evaluate the data. This service uses Gas Chromatography/Mass Spectrometry (GC/MS) and Gas Chromatography/Electron Capture Detector (GC/ECD) methods to analyze the target compounds.

3.0 DATA USES

This analytical service provides data which USEPA uses for a variety of purposes such as: determining the nature and extent of contamination at a hazardous waste site; assessing priorities for response based on risks to human health and the environment; determining appropriate clean-up actions; and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections; Hazard Ranking System (HRS) scoring; remedial investigation/feasibility studies; remedial design; treatability studies; and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/ litigation activities.

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the Organic Low Concentration Statement of Work

This Statement of Work (SOW) is designed as part of the documentation for a contract between USEPA and a commercial laboratory performing analyses in support of USEPA Superfund programs. The SOW is comprised of eight exhibits. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and instructions. Exhibit C specifies the Organic Target Compound list for this SOW with the contract required quantitation limits for the sample matrix. Exhibit D details the required analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required Quality Assurance/Quality Control (QA/QC), Standard Operating Procedures (SOPs), QA/QC performance, and the reporting of data. Exhibit F contains Chain-of-Custody and sample documentation requirements which the Contractor shall follow. To ensure proper understanding of the terms utilized in this SOW, a glossary can

be found in Exhibit G. When a term is used in the text without explanation, the glossary meaning shall be applicable. Specifications for reporting data in computer-readable format appear in Exhibit H.

4.2 Overview of Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the exhibits referenced.

4.2.1 Task I: Sample Receiving, Storage, and Disposal

4.2.1.1 Chain-of-Custody

The Contractor shall receive and maintain samples under proper Chain-of-Custody. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete Sample Delivery Group File (CSF) (Exhibit B). The Contractor shall establish and use appropriate procedures to handle confidential information received from USEPA.

4.2.1.2 Sample Scheduling/Shipments

Sample shipments to the Contractor's facility will be scheduled and coordinated by the Contract Laboratory Program (CLP) Sample Management Office (SMO). 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.

4.2.1.2.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays.

4.2.1.2.2 If there are problems with the samples (e.g., mixed media, containers broken or leaking) or sample documentation and paperwork [e.g., Traffic Reports (TRs) not with shipment, sample and TR 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 immediately notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

4.2.1.2.3 To monitor the temperature of the sample shipping cooler more effectively, each USEPA Regional Office may include a sample shipping cooler temperature blank with each cooler shipped. The temperature blank will be clearly labeled: USEPA COOLER TEMPERATURE INDICATOR. The Contractor shall record the presence or absence of the cooler temperature indicator bottle on Form DC-1, Item 9.

4.2.1.2.3.1 When the USEPA Regional Office supplies a cooler temperature indicator bottle in the sample shipping cooler, the Contractor shall use the USEPA supplied cooler temperature indicator bottle to determine the cooler temperature. The temperature of the cooler shall be measured at the time of sample receipt by the Contractor.

4.2.1.2.3.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler, and prior to unpacking the samples or removing the packing material.

4.2.1.2.3.3 To determine the temperature of the cooler, the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer (±1°C) shall have a measurable range of 0-50°C. Other devices which can measure temperature may be used if they can be calibrated to ±1°C and have a range of 0-50°C. If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the Sample Delivery Group (SDG) Narrative.

4.2.1.2.3.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10°C, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the Region from which the samples were shipped for instruction on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and the EPA sample numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on Form DC-1, under Item 10 - Cooler Temperature, and in the SDG Narrative.

4.2.1.2.4 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.

4.2.1.2.5 The Contractor is required to retain unused sample volume and partially used sample volume in original sample containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain all water (preserved and unpreserved) samples at 4°C (±2°C).

4.2.1.2.6 The Contractor shall be required to routinely return sample shipping containers (e.g., coolers) to the appropriate sampling office within 14 calendar days following shipment receipt (see clause titled, "Government Furnished Supplies and Materials").

4.2.1.3 Modified Analysis

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

4.2.2 Task II: Sample Preparation and Analysis

4.2.2.1 Overview

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high (greater than 15 percent) levels of organic and inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.2.2.2 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique USEPA 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.

4.2.2.2.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

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

4.2.2.2.2 Samples shall be assigned to SDGs at the time the samples are received, and shall not be assigned retroactively. However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case.

4.2.2.2.3 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a TR 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.

4.2.2.2.4 The Contractor shall submit signed copies of TRs for all samples in an SDG to SMO within **three working days** following receipt of the last sample in the SDG. Faxed copies of TRs do not meet this requirement. 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.

4.2.2.2.5 USEPA Case numbers, 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.

4.2.2.3 If insufficient sample volume (less than the required amount) is received to perform the analysis, the Contractor shall contact SMO to apprise them of the problem. SMO will contact the Region for instructions. The Region will either approve that no sample analysis be performed, or require that a reduced volume be used for the sample analysis. No other changes in the analysis will be permitted. SMO will notify the Contractor of the Region's

decision. The Contractor shall document the Region's decision in the SDG Narrative.

4.2.2.4 Analytical Techniques: The Target Compounds listed in Exhibit C shall be identified, as described in the methodologies given in Exhibit D. Automated computer programs may be used to facilitate the identification of compounds.

4.2.2.5 Preparation Techniques. The Contractor will prepare samples as described in Exhibit D. For semivolatile and pesticide/Aroclor samples, an aliquot is extracted with a solvent and concentrated. The concentrated extract is subjected to cleanup procedures and then analyzed by Gas Chromatography/Mass Spectrometry (GC/MS) for semivolatile or Gas Chromatography/Electron Capture Detector (GC/ECD) for the pesticide/Aroclor target compounds listed in Exhibit C. For volatile samples, an aliquot is purged with an inert gas, trapped on a solid sorbent, and then desorbed onto the GC/MS for analysis of the target compounds listed in Exhibit C.

4.2.2.6 Qualitative Verification of Compounds. The volatile and semivolatile compounds identified by GC/MS techniques shall be verified by an analyst competent in the interpretation of mass spectra by comparison of the suspect mass spectrum to the mass spectrum of a standard of the suspected compound. This procedure requires the use of multiple internal standards.

4.2.2.6.1 If a compound initially identified by GC/MS techniques cannot be verified, 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 quantitation.

4.2.2.6.2 The pesticide/Aroclor compounds identified by GC/ECD techniques shall be verified by an analyst competent in the interpretation of gas chromatograms and by comparison of the retention times of the suspected unknowns with the retention times of respective standards of the suspected compounds. Compounds shall also be confirmed by GC/MS techniques if the compounds are of sufficient concentration to be detected by the GC/MS.

4.2.2.7 Quantitation of Verified Compounds. The Contractor shall quantitate components identified by GC/MS techniques by the internal standard method stipulated in Exhibit D. Where multiple internal standards are required by USEPA, the Contractor shall perform quantitation utilizing the internal standards specified in Exhibit D. The Contractor shall quantitate components analyzed by GC/ECD techniques by the external standard method stipulated in Exhibit D. The Contractor shall also perform an initial calibration, verify its linearity, determine the breakdown of labile components, and determine calibration factors for all standards analyzed by GC/ECD techniques as described in Exhibit D.

4.2.2.8 Tentative Identification of Non-Target Sample Components. For each analysis of a sample, the Contractor shall conduct mass spectral library searches to determine tentative compound

identifications as follows: For each volatile sample, the Contractor shall conduct a search to determine the possible identity of up to 30 organic compounds of greatest concentration which are not deuterated monitoring compounds or internal standards and are not listed in Exhibit C under volatiles or semivolatiles. For each semivolatile sample, the Contractor shall conduct a search to determine the possible identification of up to 30 organic compounds of greatest concentration which are not surrogates or internal standards and are not listed in Exhibit C under volatiles or semivolatiles. In performing searches, the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent, mass spectral library shall be used.

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

4.2.2.9 Quality Assurance/Quality Control (QA/QC) Procedures

4.2.2.9.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. 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 Exhibits B and H.

4.2.2.9.2 The Contractor shall maintain a Quality Assurance Plan (QAP) 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.

4.2.2.9.3 Additional QC shall be conducted in the form of the analysis of Performance Evaluation samples submitted to the laboratory by USEPA. Unacceptable results of all such QC or Performance Evaluation samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to USEPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values, as determined by USEPA, as well as meeting the contract requirements for analysis (Exhibit D), QA/QC (Exhibit E), data reporting and other deliverables (Exhibits B and H), and sample custody, sample documentation, and Standard Operating Procedure (SOP) documentation (Exhibit F). As an alternative to data rejection, USEPA may require re-analysis of non-compliant samples. Re-analysis will be performed by the Contractor at no additional cost to USEPA, unless it is determined that the Performance Evaluation sample(s) was defective.

4.2.3 Task III: Sample Reporting and Resubmission of Data

4.2.3.1 USEPA has provided, to the Contractor, formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and submitting analysis data sheets and computer-readable data on diskette (or via an alternate means of electronic transmission approved in advance by USEPA) in a format specified in this SOW and within the time specified in Exhibit B, Section 1.1.

4.2.3.2 Use of formats other than those designated by USEPA will be deemed as non-compliant. Such data are unacceptable. Resubmission in the specified format, at no additional cost to USEPA, shall be required.

4.2.3.3 Computer-generated forms may be submitted in the hardcopy Sample Data Package(s), provided that the forms are in **exact USEPA format**. This means that the order of data elements is the same as on each USEPA-required form, including form numbers and titles, page numbers, and header information.

4.2.3.4 If the submitted data package does not conform to the specified contractual or technical criteria, the Contractor will be required to resubmit the data package with all deficiencies corrected at its own expense. The Contractor will respond within seven days to requests for additional information or explanations that result from the Government's inspection activities. If the Contractor is required to submit or resubmit data as a result of a Regional request, the data shall be clearly marked as ADDITIONAL DATA. The Contractor shall include a cover letter which describes which data are being delivered, to which USEPA project the data pertain, and who requested the data. Any and all resubmissions must be in accordance with the documentation requirements of this SOW.

4.2.3.5 The data reported by the Contractor on the hardcopy data forms and the associated computer-readable data submitted by the Contractor on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the corrected hardcopy forms or the corrected computer-readable data, or both sets of corrected data, at no additional cost to USEPA.

4.2.3.6 In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, since it is used to make major decisions regarding public health and environmental welfare. The data may also be used in litigation against Potentially Responsible Parties (PRPs) in the enforcement of Superfund legislation.

4.3 Technical and Management Capability

4.3.1 Personnel

The Contractor shall have adequate personnel at all times during the performance of the contract to ensure that USEPA receives data that meet the terms and conditions of the contract.

4.3.2 Instrumentation

The Contractor shall have a sufficient Gas Chromatograph/Electron Capture/Data System (GC/ECD/DS), Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/DS), including magnetic tape storage devices to meet all the terms and conditions of the contract.

4.3.3 Facilities

The Contractor shall maintain a facility suitable for the receipt, storage, analysis of the samples, and delivery of the product meeting the terms and conditions of the contract.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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Exhibit B - Reporting and Deliverable Requirements

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRICTION

1.1 Report Deliverable Schedule. The following table reiterates the contract reporting and deliverable requirements specified in the Contract Schedule (Performance/Delivery Schedule) and specifies the distribution that is required for each deliverable. The turnaround times for items B through E listed below are 7, 14, and 21 days.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The USEPA Analytical Operations/Data Quality Center (AOC) Organic Program Manager will notify the Contractor in writing of such changes when they occur.

Table 1

	Item	No. of Copies[A]	Delivery Schedule	Distribution: SMO	Distribution: Region
A.[2]	Sample Traffic Reports	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).[1]	X	
B.[2]	Sample Data Package[C]	1	XX[B] days after receipt of last sample in SDG.	X	
C.[2]	Data in Computer-Readable Format	1	XX[B] days after receipt of last sample in SDG.	X	X
D.[2, 3]	Complete SDG File	1	XX[B] days after receipt of last sample in SDG.		X
E.[5]	Preliminary Results (VOA Analyses)	1	Within 48 hours after receipt of each sample at laboratory, if requested.	X	X
	Preliminary Results (SV and Pest Analyses)	1	Within 72 hours after receipt of each sample at laboratory, if requested.	X	X

Exhibit B -- Section 1
Contract Reports/Deliverables Distribution (Con't)

	Item	No. of Copies	Delivery Schedule	Distribution SMO	Distribution Region
F.[4]	Standard Operating Procedures-- Technical and Evidentiary	1	Revise within 60 days after contract award. Submit within 7 days of receipt of written request to recipients as directed.		As directed
G.[4]	Quality Assurance Plan	1	Revise within 60 days after contract award. Submit within 7 days of receipt of written request to recipients as directed.		As directed
H.	GC/MS and GC/ECD Tapes	Lot	Retain for 3 years after data submission. Submit within 7 days after receipt of written request by CLP PO.		As directed
I.	Extracts	Lot	Retain for 365 days after data submission. Submit within 7 days after receipt of written request by CLP PO or SMO, at USEPA's direction.		As directed
J.[6]	Method Detection Limit Study		Submit to USEPA within 7 days after receipt of written request by CLP PO or SMO, at USEPA's discretion.		As directed

Footnotes:

[A]The number of copies specified is the number of copies required to be delivered to each recipient.

[B]The number of days associated with these elements will be provided in the associated laboratory contract document, and will also be provided at the time of the sample scheduling by the Sample Management Office (SMO) Contractor.

[C]Contractor-concurrent delivery to USEPA designated recipient (e.g., QATS) may be required upon request by the CLP PO. Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the CLP PO.

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[1]A Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less, not exceeding 20 samples, (excluding PE samples) and scheduled under the same contractual turnaround (Preliminary Results have no impact on defining the SDGs). Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

[2]**DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date all samples have been delivered. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.**

[3]Complete SDG File will contain the original Sample Data Package plus all of the original documents described under Section 2.6.

[4]See Exhibit E and Exhibit F for a more detailed description.

[5]If requested at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of Form I and Form I TIC analytical results, by fraction, for field and QC sample analyses via telefacsimile (fax). The Contractor may submit Preliminary Results in electronic format after obtaining permission from USEPA. The Contractor will be notified of the fax number or E-mail address at the time of sample scheduling. Sample Traffic Reports (TRs) and SDG cover sheets shall be submitted with the Preliminary Results. The Contractor shall contact SMO after confirming transmission. The Contractor shall document all communication in a telephone contact log.

[6]Method Detection Limit Study is performed annually or for each new instrument, whichever is more frequent. The information should be available on file and provided to USEPA within 7 days after the receipt of a written request.

Preliminary Results Delivery Schedule:

If the sample arrives before 5 p.m., the Preliminary Results for that sample are due within the required turnaround time. If the sample is received after 5 p.m., the Preliminary Results for that sample are due within the required turnaround time beginning at 8 a.m. the following day. **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent**

Footnotes (Con't):

delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.

NOTE: As specified in the Contract Schedule (Government Furnished Supplies and Materials), unless otherwise instructed by the CLP SMO based on a Regional decision, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than sixty days following submission of the reconciled Complete SDG File. Sample disposal and disposal of unused sample bottles/containers is the responsibility of the Contractor, and should be done in accordance with all applicable laws and regulations governing disposal of such materials.

1.2 Distribution

The following addresses correspond to the "Distribution" column in the tables in Sections 1.1.

SMO:
USEPA Contract Laboratory Program
Sample Management Office (SMO)[1]
2000 Edmund Halley Dr.
Reston, VA 20191-3436

Region: USEPA Region: The SMO will provide the Contractor with the list of addresses for the 10 USEPA Regions. SMO will provide the Contractor with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

USEPA AOC Organic Program Manager:

Mailing Address:
USEPA OERR Analytical Operations/
Data Quality Center
Ariel Rios Building (5204G)
1200 Pennsylvania Avenue, N.W.
Washington, DC 20469
Attn: CLP Organic Program Manager

Fed-Ex/Overnight Delivery:
USEPA OERR Analytical Operations/
Data Quality Center
1235 Jefferson Davis Highway
Crystal Gateway I, 12th Floor
Arlington, VA 22202
Attn: CLP Organic Program Manager

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[1]The Sample Management Office (SMO) is a contractor-operated facility operating under the CLASS contract awarded and administered by USEPA.

USEPA Regional CLP Project Officer (CLP PO):

SMO will provide the Contractor with the list of addresses for the CLP POs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

QATS

USEPA Contract Laboratory Program (CLP)
Quality Assurance Technical Support (QATS) Laboratory[2]
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Attn: Data Audit Staff

[2]The Quality Assurance Technical Support (QATS) Laboratory is a contractor operated facility operating under the QATS contract awarded and administered by the USEPA.

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in the Contract Schedule (Performance/Delivery Schedule). The required content and form of each deliverable is described in this exhibit. All reports and documentation must be:

- Legible;
- Clearly labeled and completed in accordance with instructions in this exhibit;
- Arranged in the order specified in this section;
- Paginated consecutively in ascending order starting from the Sample Delivery Group (SDG) Narrative;
- Copies must be legible and double-sided; and
- **Information reported on the forms listed in Exhibit B (excluding the Sample Log-in Sheet [DC-1] and the Complete SDG File (CSF) Inventory Sheet [DC-2]) must be either typewritten or computer-generated. Handwritten corrections of the information must be legible, signed, and dated.**

NOTE: Complete SDG files need not be double-sided. (The CSF is composed of original documents.) However, Sample Data Packages delivered to Sample Management Office (SMO) must be double-sided.

2.1.1 Requirements for each deliverable item cited in the Contract Schedule (Contract Performance/Delivery Schedule) are specified in Sections 2.3 to 2.9. Prior to submission, the Contractor shall arrange items and the components of each item in the order listed in these sections.

2.1.2 The Contractor shall use USEPA Case numbers (including SDG numbers) and EPA sample numbers to identify samples received under this contract, both verbally and in reports/correspondence. The contract number shall be specified in all correspondence.

2.2 Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor shall resubmit such documentation with deficiency(ies) corrected, at no additional cost to USEPA.

2.2.1 The Contractor shall respond within seven days to written requests from data recipients for additional information or explanations that result from the Government's inspection activities, unless otherwise specified in the contract.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, or through a Contract Laboratory Program Project Office (CLP PO) action, or through a Regional data reviewer's request, the data shall be clearly marked as ADDITIONAL DATA and shall be sent to both contractual data recipients (SMO and the Region; to USEPA designated recipient, upon written request). The Contractor shall include a cover letter which describes which data are being delivered, to which USEPA Case(s) the data pertain, and **who requested the data**.

2.2.3 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to both contractual data recipients (SMO and the Region; to the USEPA designated recipient (e.g., QATS), when a written request for the Sample Data Package has been made). In all instances, the Contractor shall include a color-coded COVER SHEET (Laboratory Response to Results of Contract Compliance Screening) provided by SMO.

2.3 Quality Assurance Plan (QAP) and Standard Operating Procedures (SOPs)

The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Sample Traffic Reports (TRs)

Each sample received by the Contractor will be labeled with a EPA sample number, and will be accompanied by a Sample TR bearing the sample number and descriptive information regarding the sample. The Contractor shall complete the TR (marked "Lab Copy for Return to SMO"), recording the date of sample receipt and sample condition upon receipt for each container, and shall sign the TR. Information shall be recorded for each sample in the SDG.

2.4.1 The Contractor shall submit TRs in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG cover sheet attached. The SDG cover sheet shall contain the following items:

- Laboratory name;
- Contract number;
- Sample analysis price (full sample price from the contract);
- Case number; and
- List of EPA sample numbers of all samples in the SDG, identifying the <u>first</u> and <u>last</u> samples received, and their dates of receipt (LRDs).

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); the "last" sample received would be the highest sample number (considering both alpha and numeric designations).

2.4.2 Each TR shall be clearly marked with the SDG number, entered below the laboratory receipt date on the TR. The TR for the <u>last</u> sample received in the SDG shall be clearly marked "SDG--FINAL SAMPLE". 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 shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

2.4.3 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 Contractor shall make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.

2.5 Sample Data Package

The Sample Data Package is divided into the five major units described in this section. The last three units are each specific to an analytical fraction (volatiles, semivolatiles, and pesticides/Aroclors). If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable. The Sample Data Package shall include data for the analyses of all samples in one SDG, including field samples, dilutions, re-analyses, blanks, Laboratory Control Samples, and any requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD). The Contractor shall retain a copy of the CSF for 365 days after final acceptance of data. After this time, the Contractor may dispose of the package.

2.5.1 SDG Narrative. This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; Case number; EPA sample numbers in the SDG, differentiating between initial analyses and re-analyses; SDG number; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package. The Contractor shall include any technical and administrative problems encountered, the corrective actions taken, the resolution, and an explanation for all flagged edits (e.g., manual edits) on quantitation lists. This includes documenting the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Contractor shall also provide, in the SDG Narrative, sufficient information, including equations or curves (at least one equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any flexibility SOW modification. This includes attaching a copy of the approved modification form to the SDG Narrative. Additionally the Contractor shall also identify and explain any differences which exist between the Form Is and supporting documentation provided in the data package and those previously provided as preliminary results.

All Gas Chromatograph (GC) columns used for analysis shall be documented here, by fraction. List the GC column identification--brand name, the internal diameter, in millimeters (mm), and the length, in meters, packing/coating material and film thickness. The trap used for volatile analysis shall be described here. List trap name, when denoted by manufacturer, its composition [packing material/brand name, amount of packing material, in length, centimeters (cm)]. All tentatively identified (semi-volatile) alkanes and their estimated concentrations are to be reported here. The EPA sample number, the CAS number, when available, the alkane compound (or series) name, and its estimated concentration shall be provided in tabular format. The Contractor shall document in the SDG Narrative all instances of manual integration. The SDG Narrative 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 and in the computer-readable data submitted on diskette has been authorized by the laboratory manager or his/her designee, as verified by the following signature." This statement shall be directly followed by an original signature of the laboratory manager or their designee with a typed line below it containing the signer's name and title, and the date of signature.

2.5.1.1 The samples analyzed under this contract should not exhibit a matrix effect which would prevent the Contractor from meeting the requirements of the contract. Sample re-extraction/re-analyses 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 CLP PO. Send or telefax to the CLP PO a copy of the SDG Narrative (include your contract number), a description of the situation and the requested CLP PO action, either prior or concurrent with the delivery of the Sample Data Package.

2.5.1.2 The Contractor shall list the pH determined for each water sample submitted for volatiles analysis. This information may appear as a simple list or table in the SDG Narrative. The purpose of this pH determination is to ensure that all water volatiles samples were acidified in the field. No pH adjustment is to be performed by the Contractor on water samples for volatiles analysis.

2.5.2 Traffic Reports. The Contractor shall include a copy of the TRs submitted in Section 2.4 for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample number order, considering both letters and numbers. Copies of the SDG cover sheet are to be included with the copies of the TRs. (See Section 2.4 for more detail on reporting requirements for TRs.) In the case of multi-sample TRs, the Contractor shall make the appropriate number of photocopies of the TR so that a copy is submitted with each applicable data package. In addition, in any instance where samples from more than one multi-sample TR are in the same data package, the Contractor shall submit a copy of the SDG cover sheet with copies of the TRs.

2.5.3 Volatiles Data

2.5.3.1 Volatiles QC Summary

2.5.3.1.1 Deuterated Monitoring Compound Recovery (Form II LCV-1, LCV-2).

2.5.3.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III LCV). This data shall be provided upon Region's request.

2.5.3.1.3 Method Blank Summary (Form IV LCV): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

2.5.3.1.4 GC/MS instrument performance check (Form V LCV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.5.3.1.5 Internal Standard Area and RT Summary (Form VIII LCV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.5.3.2 Volatiles Sample Data. Sample data, including dilutions, and re-analyses data, shall be arranged in packets with the Organic Analysis Data Sheet (Form I LCV-1, LCV-2, including Form I LCV-TIC), followed by the raw data for volatile samples. These sample packets shall be placed in increasing EPA sample number order, considering both letters and numbers.

2.5.3.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I LCV-1, LCV-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C,

Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (Section 2.5.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.3.2.2 Tentatively Identified Compounds (Form I LCV-TIC). Form I LCV-TIC is the tabulated list of the highest probable match for up to 30 organic compounds that are not deuterated monitoring compounds or internal standard compounds and are not listed in Exhibit C. It includes the Chemical Abstracts Service (CAS) registry number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. If no compounds are found, indicate this on the form by entering "0" in the field for "Number Found".

2.5.3.2.3 Reconstructed Total Ion Chromatograms (for each sample or sample extract, including dilutions and re-analyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:

- EPA sample number;
- Date and time of analysis;
- GC/MS instrument identifier;
- Lab file identifier, and
- Analyst ID.

2.5.3.2.3.1 Internal standards and deuterated monitoring compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times, if retention times (or scan numbers) are printed above the peak.

2.5.3.2.3.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the Sample Data Package, in addition to the chromatogram:

- EPA sample number;
- Date and time of analysis;
- Retention time or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;

- GC/MS instrument identifier;
- Lab file identifier; and
- Analyst ID.

2.5.3.2.3.3 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, and shall include the integration scan range. In addition, a hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standards and deuterated monitoring compounds.

- EICPs displaying each manual integration.

2.5.3.2.4 Other Required Information. For each sample, by each compound identified, the following items shall be included in the data package.

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (Volatiles), that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Spectra shall be labeled with EPA sample number, lab file identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.
- Copies of mass spectra of non-deuterated monitoring/non-internal standard organic compounds not listed in Exhibit C with associated best-match spectra (minimum of one, maximum of three best matches). Spectra shall be labeled with EPA sample number, lab file identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.

2.5.3.3 Volatiles Standards Data

2.5.3.3.1 Initial calibration data (Form VI LCV-1, LCV-2, LCV-3) shall be included in order by instrument, if more than one instrument is used.

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.5.3.2.3. Spectra are not required.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- EICPs displaying each manual integration.

2.5.3.3.2 Continuing calibration data (Form VII LCV-1, LCV-2, LCV-3) shall be included in order by instrument, if more than one instrument is used.

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibrations, labeled as in Section 2.5.3.2.3. Spectra are not required.
- When more than one continuing calibration is performed, forms shall be in chronological order, by instrument.
- EICPs displaying each manual integration.

2.5.3.3.3 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, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standards and deuterated monitoring compounds.

2.5.3.4 Volatiles Raw QC Data

2.5.3.4.1 4-Bromofluorobenzene (BFB) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.5.3.2.4.
- Mass listing, labeled as in Section 2.5.3.2.4.
- Reconstructed total ion chromatogram, labeled as in Section 2.5.3.2.3.

2.5.3.4.2 Blank data shall be arranged by type of blank (method, storage, instrument) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I LCV-1, LCV-2).
- Tentatively identified compounds (Form I LCV-TIC) even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.3.2.3.
- Target compound spectra with laboratory-generated standard spectra, labeled as in Section 2.5.3.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:

-- Raw target compound spectra.

-- Enhanced or background-subtracted spectra.

-- Laboratory-generated standard spectra.

- GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.5.3.2.4.

- Quantitation/calculation of tentatively identified compound concentrations.

2.5.3.4.3 Volatiles Matrix Spike Data

- Tabulated results (Form I LCV-1, LCV-2) of target compounds. Form I LCV-TIC is not required.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.3.2.3. Spectra are not required.

2.5.3.4.4 Volatiles Matrix Spike Duplicate Data

- Tabulated results (Form I LCV-1, LCV-2) of target compounds. Form I LCV-TIC is not required.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.3.2.3. Spectra are not required.

2.5.4 Semivolatiles Data

2.5.4.1 Semivolatiles QC Summary

2.5.4.1.1 Deuterated Monitoring Compound Recovery (Form II LCSV-1, LCSV-2).

2.5.4.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III LCSV): This data shall be provided upon Region's request.

2.5.4.1.3 Method Blank Summary (Form IV LCSV): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

2.5.4.1.4 GC/MS Instrument Performance Check (Form V LCSV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.5.4.1.5 Internal Standard Area and RT Summary (Form VIII LCSV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.5.4.2 Semivolatiles Sample Data. Sample data, including dilutions and re-analysis samples, shall be arranged in packets with the Organic Analysis Data Sheet **(Form I LCSV-1, LCSV-2, including Form I LCSV-TIC)**, followed by the raw data for semivolatile samples. These sample packets shall be placed in increasing EPA sample number order, considering both letters and numbers.

2.5.4.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I LCSV-1, LCSV-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Semivolatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (Section 2.5.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed

description of the problems associated with the sample in the SDG Narrative.

2.5.4.2.2 Tentatively Identified Compounds (Form I LCSV-TIC). Form I LCSV-TIC is the tabulated list of the highest probable match for up to 30 organic compounds that are not deuterated monitoring compounds, or internal standard organic compounds and are not listed in Exhibit C (Volatiles, Semivolatiles). It includes the CAS registry number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. If no compounds are found, indicate this on the form by entering "0" in the field for "Number TICs found."

2.5.4.2.3 Reconstructed Total Ion Chromatograms (for each sample, including dilutions and re-analyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:

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

2.5.4.2.3.1 Internal standards and deuterated monitoring compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times (or scan numbers) are printed over the peak.

2.5.4.2.3.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the Sample Data Package, in addition to the chromatogram:

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

2.5.4.2.3.3 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, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards and deuterated monitoring compounds.

- EICPs displaying each manual integration.

2.5.4.2.4 Other Required Information. For each sample, by each compound identified, the following shall be included in the data package.

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (Semivolatiles) that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier compound names shall be clearly marked on all spectra.

- Copies of mass spectra of non-deuterated monitoring compounds/non-internal standard organic compounds not listed in Exhibit C (Volatiles and Semivolatiles) with associated best-match spectra (minimum of one, maximum of three best matches). This includes the mass spectra for tentatively identified alkanes. Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier compound names shall be clearly marked on all spectra.

2.5.4.3 Semivolatiles Standards Data

2.5.4.3.1 Initial calibration data (Form VI LCSV-1, LCSV-2, LCSV-3) shall be included in order by instrument, if more than one instrument is used.

- Semivolatile standard(s), reconstructed ion chromatograms, and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.5.4.2.3. Spectra are not required.

- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.

- EICPs displaying each manual integration.

2.5.4.3.2 Continuing calibration data (Form VII LCSV-1, LCSV-2, LCSV-3) shall be included in order by instrument, if more than one instrument used.

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour)

calibrations, labeled as in Section 2.5.4.2.3. Spectra are not required.

- When more than one continuing calibration is performed, forms shall be in chronological order, by instrument.

- EICPs displaying each manual integration.

2.5.4.3.3 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, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and deuterated monitoring compounds.

2.5.4.4 Semivolatiles Raw QC Data

2.5.4.4.1 Decafluorotriphenylphosphine (DFTPP) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.5.4.2.4.

- Mass listing, labeled as in Section 2.5.4.2.4.

- Reconstructed total ion chromatogram, labeled as in Section 2.5.4.2.3.

2.5.4.4.2 Blank data shall be included in chronological order by extraction date.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I LCSV-1, LCSV-2).

- Tentatively identified compounds (Form I LCSV-TIC) even if none are found.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.4.2.3.

- Target compound spectra with laboratory-generated standard spectra, labeled as in Section 2.5.4.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:

-- Raw target compound spectra.

-- Enhanced or background-subtracted spectra.

-- Laboratory-generated standard spectra.

- GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.5.4.2.4.

- Quantitation/calculation of tentatively identified compound concentrations.

2.5.4.4.3 Semivolatiles Matrix Spike Data

- Tabulated results (Form I LCSV-1, LCSV-2) of target compounds. Form I LCSV-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.4.2.3. Spectra are not required.

2.5.4.4.4 Semivolatiles Matrix Spike Duplicate Data

- Tabulated results (Form I LCSV-1, LCSV-2) of target compounds. Form I LSV-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.4.2.3. Spectra are not required.

2.5.5 Pesticide/Aroclor Data

2.5.5.1 Pesticide/Aroclor QC Summary

2.5.5.1.1 Surrogate Percent Recovery Summary (Form II LCP).

2.5.5.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III LCP-1): This data shall be provided upon Region's request.

2.5.5.1.3 Laboratory Control Sample Recovery (Form III LCP-2).

2.5.5.1.4 Method Blank Summary (Form IV LCP): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

2.5.5.2 Pesticide/Aroclor Sample Data. Sample data including dilutions and re-analyses shall be arranged in packets with the Organic Analysis Data Sheet (Form I LCP) and the raw data for pesticide samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.

2.5.5.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I LCP). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Pesticides/Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 2.5.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.5.2.2 Copies of Pesticide Chromatograms. Positively identified (identified according to the criteria in Exhibit D Pesticides and Aroclors) compounds for each column shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D Pesticides and Aroclors, and shall be labeled with the following information:

- EPA sample number;
- Volume injected (µL);

- Date and time of injection;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor.

2.5.5.2.3 Copies of pesticide chromatograms from the second GC column shall be included and labeled as in Section 2.5.5.2.2.

2.5.5.2.4 Data System Printout. A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.5.5.2.5 All manual work sheets shall be included in the Sample Data Package.

2.5.5.3 Pesticide/Aroclor Standards Data

2.5.5.3.1 Initial Calibration of Single Component Analytes (Form VI LCP-1, LCP-2): for all GC columns, all instruments, in chronological order by GC column and instrument.

2.5.5.3.2 Initial Calibration of Multicomponent Analytes (Form VI LCP-3): for all GC columns, all instruments, in chronological order by GC column and instrument.

2.5.5.3.3 Analyte Resolution Summary (Form VI LCP-4): for all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.4 Performance Evaluation Mixture (Form VI LCP-5): for all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.5 Individual Standard Mixture A (Form VI LCP-6): for all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.6 Individual Standard Mixture B (Form VI LCP-7): for all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.7 Calibration Verification Summary (Form VII LCP-1): for all performance evaluation mixtures and instrument blanks, on all GC columns and instruments, in chronological order by GC column and instrument. Report for each associated instrument blank.

2.5.5.3.8 Calibration Verification Summary (Form VII LCP-2): for all mid-point concentrations of Individual Standard Mixtures A and B and reported for all instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.9 Analytical Sequence (Form VIII LCP): for all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.10 Florisil Cartridge Check (Form IX LCP): for all lots of cartridges used to process samples in the SDG.

2.5.5.3.11 Pesticide Identification Summary for Single Component Analytes (Form X LCP-1): for all samples with positively identified single component analytes, in order by increasing EPA sample number.

2.5.5.3.12 Pesticide Identification Summary for Multicomponent Analytes (Form X LCP-2): for all samples with positively identified multicomponent analytes, in order by increasing EPA sample number.

2.5.5.3.13 Chromatograms and data system printouts shall be included for all standards including the following:

- Resolution check mixture;
- Performance evaluation mixtures, all;
- Individual Standard Mixture A, at three concentrations, each initial calibration;
- Individual Standard Mixture B, at three concentrations, each initial calibration;
- All multicomponent analytes (toxaphene and Aroclors), each initial calibration;
- All mid-point concentrations of Individual Standard Mixtures A and B used for calibration verification; and
- All multicomponent analyte standards analyzed for confirmation.

2.5.5.3.14 A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D Pesticides and Aroclors, and shall be labeled with the following:

- EPA sample number for the standard, e.g., INDA, INDB, etc. (See Section 3.3.7.6 for details);
- Labels of all standard peaks for all individual compounds either directly out from the peak on the chromatogram or on the printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram;
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram;
- Date and time of injection;

- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor.

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/EC operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.5.5.4 Pesticide/Aroclor Raw QC Data

2.5.5.4.1 Blank data shall be arranged by type of blank (method, instrument, sulfur cleanup) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I LCP).
- Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, labeled as in Sections 2.5.5.2.2 and 2.5.5.2.4.

2.5.5.4.2 Laboratory Control Sample Data

- Tabulated results (Form I LCP) of target compounds.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.5.5.2.2 and 2.5.5.2.4 and for both columns as in Section 2.5.5.2.3.

2.5.5.4.3 Pesticides Matrix Spike

- Tabulate results (Form I LCP) of target compounds.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Section 2.5.5.2.2 and 2.5.5.2.4 and for both columns as in Section 2.5.5.2.3.

2.5.5.4.4 Pesticides Matrix Spike Duplicate Data

- Tabulate results (Form I LCP) of target compounds.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Section 2.5.5.2.2 and 2.5.5.2.4 and for both columns as in Section 2.5.5.2.3.

2.5.5.5 Raw Florisil Data. The chromatogram and data system report(s) shall be arranged in chronological order by Florisil cartridge performance check analyses.

- Chromatograms and data system reports labeled as specified in Sections 2.5.5.2.2 and 2.5.5.2.4 for the florisil cartridge performance check analyses.
- Chromatograms and data system reports for standard analyses used to quantify compounds in the Florisil cartridge performance check analysis, labeled as specified in Section

2.5.5.3.14 (i.e., Individual Standard Mixture A and Individual Standard Mixture B and the 2,4,5 Trichlorophenol solution).

2.6 Complete SDG File (CSF)

As specified in Section 1, the Contractor shall deliver one Complete SDG File (CSF) including the original Sample Data Package to the Region concurrently with delivery of the Sample Data Package to SMO. (Delivery to a USEPA designated recipient is only required upon written request.)

2.6.1 The CSF will contain all original documents as specified in Section 3 and Exhibit F, and in Form DC-2 (see Section 4). No photocopies of original documents will be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to USEPA with another Case/SDG in accordance with the requirements described in Exhibit F. The contents of the CSF shall be numbered according to the specifications described in Section 3.20.

2.6.2 The CSF will consist of the following original documents in addition to the documents in the Sample Data Package.

NOTE: All SDG-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other SDG-specific documents generated after the CSF is sent to USEPA, as well as copies that are altered in any fashion, are also deliverables to USEPA. (Deliver the original to the Region and a copy to SMO. Delivery to a USEPA-designated recipient is only upon written request.)

2.6.2.1 The original Sample Data Package.

2.6.2.2 A completed and signed document inventory sheet (Form DC-2).

2.6.2.3 All original shipping documents including, but not limited to, the following documents:

- USEPA Chain-of-Custody Record;
- Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information);
- USEPA TRs; and
- Sample tags (if present) sealed in plastic bags.

2.6.2.4 All original receiving documents including, but not limited to, the following documents:

- Form DC-1;
- Other receiving forms or copies of receiving logbooks; and
- SDG cover sheet.

2.6.2.5 All original laboratory records, not already submitted in the Sample Data Package, of sample transfer, preparation and analysis including, but not limited to, the following documents:

- Original preparation and analysis forms or copies of preparation and analysis logbook pages;

- Internal sample and sample extract transfer chain-of-custody records;
- Screening records; and
- All instrument output, including strip charts from screening activities.

2.6.2.6 All other original SDG-specific documents in the possession of the Contractor including, but not limited to, the following documents:

- Telephone contact logs;
- Copies of personal logbook pages;
- All handwritten SDG-specific notes; and
- Any other SDG-specific documents not covered by the above.

2.6.3 If the Contractor does submit SDG-specific documents to USEPA after submission of the CSF, the documents should be identified with unique accountable numbers, a revised Form DC-2 should be submitted, and the unique accountable numbers and the locations of the documents in the CSF should be recorded in the "Other Records" section on the revised Form DC-2. Alternatively, the Contractor may number the newly submitted SDG-specific documents to USEPA as a new CSF and submit a new Form DC-2. The revised Form DC-2 or new Form DC-2 should be submitted to the USEPA Regions only.

2.7 Data in Computer-Readable Format

The Contractor shall provide a computer-readable copy of the data on data reporting Forms I-X for all samples in the SDG as specified in Exhibit H, and delivered as specified in the Contract Schedule (Contract Performance/Delivery Schedule). Computer-readable data deliverables shall be submitted on IBM or IBM-compatible, 3.5-inch high-density 1.44 MB diskette (or via an alternate means of electronic transmission approved in advance by the USEPA).

2.7.1 When submitted, the diskette(s) shall be packaged and shipped in such a manner that the diskette(s) cannot be bent or folded, and will not be exposed to extreme heat or cold or any type of electromagnetic radiation. The diskette(s) shall be included in the same shipment as the hardcopy data and shall, at a minimum, be enclosed in a diskette mailer. The diskette(s) shall be labeled as specified in Exhibit H, Section 8.4.

2.7.2 The data shall be recorded in ASCII, text file format, and shall adhere to the file, record, and field specifications listed in Exhibit H.

2.8 Preliminary Results

The Form I data results shall be submitted for all samples in one SDG of a Case. This includes tabulated target compound results (Form I) for the volatile, semivolatile, and pesticide fractions, and tentatively identified compounds (Form I TIC) for the volatile and semivolatile fractions. The Contractor shall clearly identify the Preliminary Results by labeling each Form I and Form I TIC as "Preliminary Results" under each form title (e.g., under Volatile Organics Analysis Data Sheet, Volatile Organics Analysis Data Sheet Tentatively Identified Compounds).

2.8.1 The Contractor shall also include a disclaimer at the bottom of all Form Is stating that the "Data results contained on this Form I are for scanning purposes only and may not have been validated fro CLP criteria."

2.8.2 Sample Traffic Reports and SDG Cover Sheets shall be submitted with the Preliminary Results.

2.9 GC/MS and GC/ECD Tapes

The Contractor shall adhere to the requirements in Section 13 of Exhibit E.

2.10 Extracts

The Contractor shall preserve sample extracts at 4°C (±2°C) in bottles/vials with PTFE-lined septa. Extract bottles/vials shall be labeled with EPA sample number, Case number, and SDG number. The Contractor shall maintain a logbook of stored extracts, listing EPA sample numbers and associated Case and SDG numbers. The Contractor shall retain extracts for 365 days following submission of the reconciled complete Sample Data Package. During that time, the Contractor shall submit extracts and associated logbook pages within seven days following receipt of a written request from the CLP PO.

3.0 FORM INSTRUCTIONS

3.1 Introduction

This section includes specific instructions for completing the data reporting forms required under this contract. Each of the forms is specific to a given fraction (volatile, semivolatile, or pesticide/Aroclor). The Contractor shall submit only those forms pertaining to the fractions analyzed for a given sample(s). For instance, if a sample is scheduled for volatiles analysis only, the Contractor shall provide only forms for the volatile fraction.

3.2 General Information

The Contractor shall report values on the hardcopy forms according to the individual form instructions in this section. For instance, all results for concentrations of target compounds shall be reported to two significant figures. Values that exceed the maximum length allowed shall be reported to the maximum possible, maintaining the specified significance.

3.2.1 The data reporting forms presented in Section 4 have been designed in conjunction with the computer-readable data format specified in Exhibit H. The specific length of each variable for computer-readable data transmission purposes is also given in Exhibit H. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as lab name and lab sample identifier.

NOTE: The space provided for entries on the hardcopy forms (Section 4) is greater in some instances than the length prescribed for the variable as written to the electronic deliverable (see Exhibit H). Greater space is provided on the hardcopy forms for visual clarity.

3.2.2 When submitting data, the Contractor shall reproduce <u>all</u> characters that appear on the data reporting forms in Section 4. The format of the forms submitted shall be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval from the Contract Laboratory Program Project Officer (CLP PO). The names of the various fields and compounds (e.g., "Lab Code," "Chloromethane") shall appear as they do on the forms in the contract. For items appearing on the uncompleted forms (Section 4), the use of uppercase and lowercase letters is optional.

3.2.3 Alphabetical entries made on the forms by the Contractor shall be in ALL UPPERCASE letters (e.g., "ABCDE", not "Abcde" or "abcde"). If an entry does not fill the entire blank space provided on the form, null characters shall be used to remove the remaining underscores that comprise the blank line. However, the Contractor shall <u>not</u> remove the underscores or vertical bars that delineate "boxes" on the forms. The only exception would be those underscores at the bottom of a "box" that are intended as a data entry line. (For instance, on Form 2LCV, line 30, if data is entered on line 30, it will replace the underscores.)

3.3 Header Information

Six pieces of information are common to the header section of each data reporting form: lab name, contract, lab code, Case number, Client number and Sample Delivery Group (SDG) number. Except as noted for Client number, this information shall be entered on every form and shall match on every form.

3.3.1 Lab Name. The lab name shall be the name chosen by the Contractor to identify its laboratory. It shall not exceed 25 characters.

3.3.2 Contract. Contract refers to the number of the USEPA contract under which the analyses were performed.

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

3.3.4 Case Number. The Case number is the USEPA-assigned Case number associated with the sample. This number is reported on the Traffic Report (TR).

3.3.5 Client Number. The Client number is a unique number identifying the client and the project. This number may be the USEPA-assigned number for analyses performed under Non-Routine Analytical Services (NRAS). If samples are to be analyzed under NRAS only, and reported on these forms, then enter the NRAS number as "Client No." and leave the Case number blank. If samples are analyzed according to the Routine Analytical Services (RAS) protocol and have additional NRAS requirements, list both the Case number and NRAS number on all forms. If the analyses have no NRAS requirements, leave the "Client No." field blank.

NOTE: Some samples in an SDG may have a Client Number where as other may not.

3.3.6 SDG Number. The "SDG No." is the Sample Delivery Group (SDG) number. The SDG number is the EPA sample number of the first sample received in the SDG, except when this would cause duplication. 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. If fractions of the same field samples are scheduled under different turnaround times, thus creating separate SDG's containing the same sample numbers, a different sample number shall be utilized in the assignment of the SDG number for each SDG. If a situation arises where there are an insufficient number of samples for assignment of SDG numbers the Contractor shall contact SMO for the assignment of an SDG number.

3.3.7 Sample Number. 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 number is entered in the triple-spaced box in the upper right-hand corner of Form I, Form IV, or Form X, it should be entered on the middle line of the three lines that comprise the box.

3.3.7.1 The Contractor shall identify all samples, including dilutions and re-analyses, Laboratory Control Samples, requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD) (as described in Section 3.3.7.4), blanks, and standards with an EPA sample number. For field samples, the EPA sample number is the five digit unique identifying number given in the TR that accompanied that sample. In order to facilitate data assessment, the Contractor shall use the following sample suffixes:

XXXXX	=	EPA sample number
XXXXXMS	=	Matrix spike sample
XXXXXMSD	=	Matrix spike duplicate sample
XXXXXRE	=	Re-extracted and re-analyzed sample
XXXXXDL	=	Sample analyzed at a dilution
XXXXXDL2	=	Sample analyzed at a secondary dilution
XXXXXDL3	=	Sample analyzed at a third dilution

NOTE: The Region may approve up to one additional dilution be performed beyond the one dilution for volatiles and semivolatiles and two dilutions for pesticides specified in Exhibit D. The approval of the additional dilution by the Region must be documented in the SDG Narrative and include the Telephone Record Conversation between SMO and the Contractor communicating USEPA's decision.

3.3.7.2 There may be instances when all samples analyzed must be listed on the form, regardless of whether or not they are part of the SDG being reported (e.g., Form VIII LCP). In these instances, use ZZZZZ as the EPA sample number for any sample analysis not associated with the SDG being reported.

3.3.7.3 For blanks, the Contractor shall use the following identification scheme for the EPA sample number:

- Volatile method blanks shall be identified as VBLK##;
- Volatile instrument blanks shall be identified as VIBLK##;
- Volatile storage blanks shall be identified as VHBLK##;
- Semivolatile method blanks shall be identified as SBLK##;
- Pesticide/Aroclor method blanks and/or sulfur cleanup blanks shall be identified as PBLK##; and
- Pesticide/Aroclor instrument blanks shall be identified as PIBLK##.

3.3.7.3.1 The EPA sample number shall be unique for each blank within an SDG. Within a fraction, the Contractor 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 volatile blanks would be VBLK1, VBLK2, VBLKA1, VBLKB2, VBLK10, VBLKAB, etc.

3.3.7.3.2 If the method blank is analyzed on multiple instruments, then an additional two-character suffix shall be added to make the blank EPA sample number unique.

3.3.7.4 The EPA sample number shall be unique for each Laboratory Control Sample within an SDG. The EPA sample number for a Laboratory Control Sample must be PLCS##.

Where:

P = fraction (P for pesticides/Aroclors)

LCS = indicates a Laboratory Control Sample

= suffix consisting of characters or numbers or both that makes the EPA sample number for the LCS unique in the SDG.

(1) = When reporting results on Form I, a "(1)" is appended on to the sample number to indicate that the results are from Gas Chromatograph (GC) column(1) [e.g., PLCS01(1)].

(2) = When reporting results on Form I, a "(2)" is appended on to the sample number to indicate that the results are from GC column(2) [e.g., PLCS01(2)].

3.3.7.5 Volatile and semivolatile standards shall be identified as FSTD***##.

Where:

F = Fraction code (V for volatiles; S for semivolatiles)

STD = Standard

*** = Concentration of volatile standards in µg/L (e.g., 0.5, 001, 005, 010, and 025) or the amount injected in ng for semivolatile standards (e.g., 005, 010, 020, 050, and 080)

= One or two characters, numbers, or combinations of both to create a unique EPA sample number within an SDG.

3.3.7.6 The Contractor shall use the following scheme to identify pesticide/Aroclor standards:

Name	EPA Sample Number
Individual Mix A (low point)	INDAL##
Individual Mix A (mid-point)	INDAM##
Individual Mix A (high point)	INDAH##
Individual Mix B (low point)	INDBL##
Individual Mix B (mid-point)	INDBM##
Individual Mix B (high point)	INDBH##
Resolution Check	RESC##
Performance Evaluation Mixture	PEM##
Toxaphene	TOXAPH##
Aroclor 1016	AR1016##
Aroclor 1221	AR1221##
Aroclor 1232	AR1232##
Aroclor 1242	AR1242##
Aroclor 1248	AR1248##

Name	EPA Sample Number
Aroclor 1254	AR1254##
Aroclor 1260	AR1260##
Aroclor 1016/1260 Mix	AR1660##

The Contractor shall replace the two-character terminator (##) of the identifier with one or two characters or numbers, or a combination of both, to create a unique EPA sample number within an SDG.

3.3.7.6.1 If the standards are injected onto both GC columns on the same instrument simultaneously, the same EPA sample number may be used for reporting data for the standards for both columns. If simultaneous injections are not made, then the same number shall not be used.

3.3.7.7 The EPA sample number for florisil shall be FLO#########, where ######### is the florisil cartridge lot number. If the florisil cartridge lot number is more than nine characters, truncate at the ninth character.

3.3.8 Other Common Fields. Several other pieces of information are common to many of the data reporting forms. These include purge volume/sample volume, lab sample identifier, lab file identifier, instrument ID, and page _ of _.

3.3.8.1 "Purge Volume" or "Sample Volume" is the total volume of water that was purged or extracted, in milliliters (mL).

3.3.8.2 The lab sample identifier is a unique laboratory-generated internal identifier pertaining to a particular analysis. The Contractor can enter up to 12 alpha-numeric characters in the "Lab Sample ID" field. The Contractor may use the EPA sample number as the lab sample identifier.

3.3.8.3 The lab file identifier is the unique laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. The Contractor can enter up to 14 alpha-numeric characters in the "Lab File ID" field.

3.3.8.4 The "Instrument ID" field is common to the forms containing calibration data. The identifier used by the Contractor shall include some indication of the manufacturer and/or model of the instrument, and shall contain additional characters that differentiate between all instruments of the same type in the laboratory.

3.3.8.5 The GC column identifier, and inner diameter are common to many of the reporting forms for the volatile and pesticide fractions. In addition, column length is entered on the volatile reporting forms. Under "GC Column", enter the column identification as denoted by the manufacturer. Enter the inner diameter in the "ID" field in millimeters (mm) (to two decimal places), and the column length in the "Length" field in m (in whole numbers).

3.3.8.6 Forms II, III, IV, V, VIII, IX, and X contain a field labeled "page _ of _" in the bottom lefthand corner. If the number of entries required on any of these forms exceeds the available space, continue entries on another copy of the same fraction-specific form, duplicating all header information. If a second page is required, number the pages consecutively (i.e.,

"page 1 of 2" and "page 2 of 2"). If a second page is not required, number the page "page 1 of 1."

NOTE: These forms are fraction-specific. For example, Form II LCV and Form II LCSV are for different data. Therefore, do not number the pages of all three versions of Form II as "1 of 6," "2 of 6," etc. Number only pages corresponding to the fraction-specific form.

3.3.9 Rounding Rule. For rounding off numbers to the appropriate level of precision, the Contractor shall follow these rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than or equal to 5, drop it and increase the last digit to be retained by 1 (round up).

3.4 Organic Analysis Data Sheet (Form I, All Fractions)

3.4.1 Purpose. This form is used for tabulating and reporting sample analysis, including dilutions, re-analysis, blank, Laboratory Control Sample for target compounds and requested MS/MSD. If all fractions are not requested for analysis, only the pages for the fractions required shall be submitted. For example, if only volatiles analysis is requested, Form I LCV-1, LCV-2 and Form I LCV-TIC shall be submitted. If only the pesticide/Aroclor fraction is requested for analysis, Form I LCP shall be submitted. **Furthermore, pesticide instrument blanks (PIBLKs) shall be reported on a per column/per analysis basis on Form I LCP.** Each PIBLK shall be named with a unique EPA sample number. **Also, the Laboratory Control Sample and the MS/MSD shall be reported on a per column basis. Distinguish between GC column(1) and GC column(2) results by appending a suffix "(1)" for GC column(1) and "(2)" for GC column(2).**

3.4.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.2.1 Enter the date of sample receipt at the laboratory, as noted on the TR (i.e., the VTSR), in the "Date Received" field. The date shall be entered as MM/DD/YYYY.

3.4.2.2 Complete the "Date Extracted" and "Date Analyzed" fields in the same format (MM/DD/YYYY). For the continuous liquid-liquid extraction procedures, enter the date that the procedure was started in the "Date Extracted" field. If separatory funnel (pesticides only) was used, enter the date the procedure was completed in the "Date Extracted" field. For pesticide/Aroclor samples, enter the date of the first GC analysis performed in the "Date Analyzed" field. The date of sample receipt will be compared with the extraction and analysis dates of each fraction to ensure that contract holding times were not exceeded.

3.4.2.3 For volatiles on Form I LCV-1 and LCV-2, enter the GC column identifier in the "GC Column" field, the internal diameter in mm, to two decimal places in the "ID" field, and the length in meters (m), as a whole number, as described in Section 3.3.

3.4.2.4 For pesticides/Aroclors, enter the method of extraction in the "Extraction" field on Form I LCP as "SEPF" for separatory funnel, or "CONT" for continuous liquid-liquid extraction.

3.4.2.5 For semivolatiles and pesticides/Aroclors, enter the actual volume of the most concentrated sample extract, in microliters (µL), in the "Concentrated Extract Volume" field on Form I LCSV-1, LCSV-2 or LCP. For semivolatiles, this volume will typically be 1,000 µL. For pesticides/Aroclors, the volume of the most concentrated extract will typically be 2,000 µL. If a dilution of the sample extract is made in a subsequent analysis, this volume will remain the same, but the dilution factor will change.

3.4.2.6 For semivolatiles and pesticides/Aroclors, enter the volume of the sample extract injected into the GC in the "Injection Volume" field on Form I LCSV-1, LCSV-2 or LCP. Report this volume in microliters (µL) to one decimal place (e.g., 1.0 µL).

3.4.2.7 If pesticides/Aroclors are analyzed using two GC columns connected to a single injection port, enter the amount of half the volume in the syringe in the "Injection Volume" field (i.e., assume that the extract injected is evenly divided between the two columns).

3.4.2.8 If a sample or sample extract has been diluted for analysis, enter the dilution factor as a single number (e.g., enter 100.0 for a 1 to 100 dilution of the sample) in the "Dilution Factor" field. The dilution factor shall not be entered as a fraction. If a sample was not diluted, enter 1.0. Report dilution factors to one decimal place.

3.4.2.9 If sulfur cleanup is employed, enter Y in the "Sulfur Cleanup" field; if not, enter N on Form I LCP.

3.4.2.10 For positively identified target compounds, the Contractor shall report the concentrations as uncorrected for blank contaminants.

3.4.2.11 Report all analytical results to two significant figures.

3.4.2.12 Under the column labeled "Q" for qualifier, flag each result with the specific data reporting qualifiers listed below. When reporting results to USEPA, the Contractor shall use these contract-specific qualifiers. The Contractor shall not modify the qualifiers. Up to five qualifiers may be reported on Form I for each compound. The Contractor is encouraged to use additional flags or footnotes (see the X qualifier).

The USEPA-defined qualifiers to be used are:

U: This flag indicates the compound was analyzed for but not detected. The Contract Required Quantitation Limit (CRQL) shall be adjusted according to the equation listed in Exhibit D. CRQLs are listed in Exhibit C.

J: This flag indicates an estimated value. This flag is used (1) when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, (2) when the mass spectral and retention time data indicate the presence of a compound that meets the volatile and semivolatile GC/MS identification criteria, and the result is less than the CRQL but greater than zero, and (3) when the retention time data indicate the presence of a compound that meets the identification criteria for a pesticide and/or an Aroclor, and the result is less than the CRQL but greater than zero. For example, if the sample quantitation limit is 5.0 µg/L, but a concentration of 3.0 µg/L is calculated, report it as 3.0J.

NOTE: The J flag is not used and the compound is not reported as being identified for pesticide/Aroclor results less than the CRQL if the pesticide residue analysis expert determines that the peaks used for compound identification resulted from instrument noise or other interferences (column bleed, solvent contamination, etc.).

N: This flag indicates presumptive evidence of a compound. This flag is only used for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search. It is applied to all TIC results. For generic characterization of a TIC, such as chlorinated hydrocarbon, the N flag is not used.

P: This flag is used for a pesticide/Aroclor target analyte when there is greater than 25% difference for detected concentrations between the two GC columns (see Form X). **The lower of the two values is reported on Form I and flagged with a P.** The P flag is not used unless a compound is identified on both columns.

C: This flag is not used under this contract, but it is reserved for USEPA use.

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

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

E: This flag identifies compounds whose concentrations exceed the upper level of the calibration range of the instrument for that specific analysis. If one or more compounds have a concentration greater than the upper level of the calibration range, the sample or extract shall be diluted and re-analyzed according to the specifications in Exhibit D; exceptions are also noted in Exhibit D. All such compounds with concentrations greater than the upper level of the calibration range shall have the concentrations flagged with an E on Form I for the original analysis. The results of both analyses shall be reported on separate copies of Form I. The Form I for the diluted sample shall have for the volatile and semivolatile dilutions "DL" or ("DL2", when this additional dilution was approved by the Region) and for the pesticides dilution "DL" or "DL2" (or "DL3", when approved by the Region) suffix appended to the sample number.

NOTE: For total xylenes, where three isomers are quantified as two peaks, the calibration range of <u>each peak</u> shall be considered separately. For example, a diluted analysis is <u>not</u> required for total xylenes unless the concentration of the peak representing the single isomer exceeds 25 µg/L or the peak representing the two co-eluting isomers on that GC column exceeds 50 µg/L.

D: This flag is used for all compounds identified in an analysis as diluted. If a sample or extract is re-analyzed with a dilution factor greater than 1, for example, when the concentration of the analyte exceeds the upper calibration range, the "DL", "DL2" or "DL3" suffix is appended to the sample number on Form I for the more diluted sample, and <u>all</u> reported

concentrations on that Form I are flagged with the D flag. This flag alerts data users that any discrepancies between the reported concentrations may be due to dilution of the sample or extract.

NOTE 1: The D flag is not applied to compounds which are not detected in the sample analysis, i.e., compounds reported with the CRQL and the U flag.

NOTE 2: Separate Form Is are required for reporting the original analysis (EPA Sample No. XXXXX) and the more diluted sample analyses, i.e., the results from these analyses cannot be combined on a single Form I.

A: This flag indicates that a Tentatively Identified Compound (TIC) is a suspected aldol-condensation product.

X:Other specific flags may be required to properly define the results. If used, the flags shall be fully described in 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 represent a combination of several flags. For instance, the X flag might combine the B and D flags for some samples. The laboratory-defined flags are limited to X, Y, and Z.

3.5 Organic Analysis Data Sheet: Tentatively Identified Compounds (Form I LCV-TIC and Form I LCSV-TIC)

3.5.1 Purpose. These forms are used to report analysis results for non-target compounds (e.g., compounds not listed in Exhibit C), excluding deuterated monitoring compounds and internal standards. See Exhibit D for instructions on identification and quantitation. The Contractor shall submit Form I LCV-TIC or LCSV-TIC for every analysis, including required dilutions and re-analyses, and blanks, even if no TICs are found.

3.5.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions in addition to the instructions in Section 3.4.

3.5.2.1 Report all TICs including CAS number (if applicable), compound name, retention time, and the estimated concentration as uncorrected for blank contaminants. Report all analytical results to two significant figures. (Criteria for reporting TICs are given in Exhibit D, Section 11). Retention time shall be reported in minutes and decimal minutes, not seconds or minutes:seconds.

3.5.2.2 Total the number of TICs found, and enter this number in the "Number TICs found" field. If no TICs were found, enter 0 (zero).

3.5.2.3 If the name of a compound exceeds the 28 spaces in the TIC column, truncate the name to 28 characters. If the compound is an unknown, restrict the description to no more than 28 characters (e.g., unknown hydrocarbon).

3.6 Deuterated Monitoring Compound (DMC) Recovery (Form II LCV-1, LCV-2 and Form II LCSV-1, LCSV-2)

3.6.1 Purpose. For volatiles and semivolatiles, Form II LCV-1, LCV-2 and Form II LCSV-1, LCSV-2 are used to report the recoveries of the DMCs added to each volatile and semivolatile sample, including dilutions, re-analyses, blanks and requested MS/MSD. The DMCs are used to monitor the performance of the purge and trap for volatiles, the extraction and injection for semivolatiles, and the GC/MS system as a whole.

3.6.2 Instructions. Complete the header information according to the instructions in Section 3.3.

3.6.2.1 For each volatile DMC listed in Table 2 and each semivolatile DMC listed in Table 3, 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.

Table 2
Volatile Deuterated Monitoring Compounds

Volatile Deuterated Monitoring Compounds		CAS Number
VDMC1	Vinyl Chloride-d3	6745-35-3
VDMC2	Chloroethane-d5	19199-91-8
VDMC3	1,1-Dichloroethene-d2	22280-73-5
VDMC4	2-Butanone-d5	24313-50-6
VDMC5	Chloroform-d	865-49-6
VDMC6	1,2-Dichloroethane-d4	17060-07-0
VDMC7	Benzene-d6	1076-43-3
VDMC8	1,2-Dichloropropane-d6	93952-08-0
VDMC9	Toluene-d8	2037-26-5
VDMC10	trans-1,3-Dichloropropene-d4	93951-86-1
VDMC11	2-Hexanone-d5	4840-82-8
VDMC12	Bromoform-d	2909-52-6
VDMC13	1,1,2,2-Tetrachloroethane-d2	33685-54-0
VDMC14	1,2-Dichlorobenzene-d4	2199-69-1

Table 3
Semivolatile Deuterated Monitoring Compounds

Semivolatile Deuterated Monitoring Compounds		CAS Number
SDMC1	Phenol-d5	4165-62-2
SDMC2	bis-(2-Chloroethyl)ether-d8	93952-02-4
SDMC2	2-Chlorophenol-d4	93951-73-6
SDMC4	4-Methylphenol-d8	190780-66-6
SDMC5	Nitrobenzene-d5	4165-60-0
SDMC6	2-Nitrophenol-d4	93951-78-1
SDMC7	2,4-Dichlorophenol-d3	93951-74-7
SDMC8	4-Chloroaniline-d4	191656-33-4
SDMC9	Dimethylphthalate-d6	85448-30-2
SDMC10	Acenaphthylene-d8	93951-97-4
SDMC11	4-Nitrophenol-d4	93951-79-2
SDMC12	Fluorene-d10	81103-79-9
SDMC13	4,6-Dinitro-methylphenol-d2	93951-76-9
SDMC14	Anthracene-d10	1719-06-8
SDMC15	Pyrene-d10	1718-52-1
SDMC16	Benzo(a)pyrene-d12	63466-71-7

3.6.2.2 Flag each DMC recovery outside the QC limits with an asterisk (*). The asterisk shall be placed in the last space in each appropriate column, under the "#" symbol.

3.6.2.3 In the "TOT OUT" column, total the number of DMC recoveries that were outside the QC limits for each sample. If no DMCs were outside the limits, enter 0 (zero).

3.6.2.4 If the sample is a dilution and the deuterated monitoring compounds (DMCs) are outside the acceptance window, enter the calculated recovery and flag the DMC recoveries with a D in the column under the "#" symbol. Do <u>not</u> include recoveries flagged with a D in the total number of recoveries for each sample outside the QC limits.

3.6.2.5 Number all pages as described in Section 3.3.

3.7 Surrogate Recovery (Form II LCP)

3.7.1 Purpose. Form II LCP is used to report the recoveries of the surrogate compounds added to each pesticide/Aroclor sample, blank, Laboratory Control Sample and requested MS/MSD.

3.7.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.7.2.1 For each surrogate listed in Table 4, report the percent recovery to the nearest whole percentage point.

3.7.2.2 Flag each surrogate recovery outside the QC limits with an asterisk (*). The asterisk shall be placed in the last space in each appropriate column, under the "#" symbol.

3.7.2.3 In the "TOT OUT" column, total the number of surrogate recoveries that were outside the QC limits for each sample. If no surrogates were outside the limits, enter 0 (zero).

3.7.2.4 If the samples is a dilution and the surrogates are outside the acceptance window in any analysis, enter the calculated recovery, and flag the surrogate recoveries with a D in the column under the "#" symbol. Do not include results flagged with a D in the total number of recoveries for each sample outside the QC limits.

3.7.2.5 The pesticide surrogate recoveries shall be reported from each GC column used for the analyses. Therefore, identify each GC column at the top of Form II LCP, entering the stationary phase in the "GC Column" field, and the internal diameter of the column in mm in the "ID" field.

3.7.2.6 The assignment of columns as "1" and "2" is left to the discretion of the Contractor when the analyses are performed by simultaneous injection into a GC containing two columns. If so analyzed, the assignment of "GC Column 1" and "GC Column 2" shall be consistent across all the reporting forms. If the analysis is not performed by simultaneous injection, then the assignment of GC column number shall be based on the chronological order of the two analyses.

3.7.2.7 Number all pages as described in Section 3.3.

Table 4
Pesticide Surrogates

Pesticide Surrogates	CAS Number
Decachlorobiphenyl (DCB)	2051-24-3
Tetrachloro-m-xylene (TCX)	877-09-8

3.8 Matrix Spike/Matrix Spike Duplicate Recovery (Form III, All Fractions, LCV, LCSV, LCP-1)

3.8.1 Matrix Spike/Matrix Spike Duplicate Recovery and Laboratory Control Sample Recovery

3.8.1.1 Purpose. This form is used to report the results of the analyses of MS/MSD. This form should only be submitted if the analysis of MS/MSD samples have been requested by the Region. Complete Form III LCP-1 for each GC column used for analysis.

3.8.1.2 Instructions. Complete the header information according to the instructions in Section 3.3. Include the EPA sample number for the matrix spike, **without** the suffixes MS or MSD. Complete the remainder of the form using the following instructions. For each

Form III LCP-1 enter the Instrument ID, the stationary phase in the "GC Column" field, and the internal diameter of the column in mm in the "ID" field. The order of reporting is not important but must be consistent with Form X.

3.8.1.2.1 In the first table under the "SPIKE ADDED" column, enter the amount of spike added in µg/L for each analyte.

3.8.1.2.2 Enter the sample concentration in the next column of each spike compound detected in the original sample. If a spike compound was not detected during the analysis of the original sample, enter the sample result as 0 (zero).

3.8.1.2.3 In the "MS CONCENTRATION" column, enter the actual concentration of each spike compound detected in the matrix spike aliquot.

3.8.1.2.4 Calculate the percent recovery of each spike compound in the matrix spike aliquot to the nearest whole percent, according to Exhibit D. Enter the percent recovery in the "MS % REC" column.

3.8.1.2.5 Flag all percent recoveries outside the QC limits with an asterisk (*). The asterisk shall be placed in the last space of the "MS % REC" column, under the "#" symbol.

3.8.1.2.6 Follow Sections 3.8.1.2.1 through 3.8.1.2.5 to complete the lower table, using the results of the analysis of the MSD aliquot.

3.8.1.2.7 Calculate the relative percent difference (RPD) between the matrix spike recovery and the matrix spike duplicate recovery, and enter this value in the "RPD" column. Report the RPD to the nearest whole percent.

3.8.1.2.8 Compare the RPDs to the QC limits given on the form, and flag each RPD outside the QC limits with an asterisk (*) in the last space of the "RPD" column, under the "#" symbol.

3.8.1.2.9 Summarize the values outside the QC limits at the bottom of the page. No further action is required by the Contractor.

3.8.2 Laboratory Control Sample Recovery (Form III LCP-2)

3.8.2.1 Purpose. Form III LCP-2 is used to report the results of the analyses of the Laboratory Control Samples.

3.8.2.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.8.2.2.1 If the LCS solution is purchased by the Contractor from a third party, report the identification number used by the third party to identify the LCS lot, if available. If the LCS solution was prepared in-house, leave this entry blank.

3.8.2.2.2 The "LCS Aliquot" is the volume in microliters (µL) of LCS spiking solution that was added to reagent water before extraction.

3.8.2.2.3 The LCS is reported for each GC column. Enter the Instrument ID, GC column, and internal diameter (ID) for both GC columns. The order of reporting is not important, but must be consistent with the information reported on Form X. If simultaneous injections are not made, the "Date Analyzed" is the earlier date of the two LCS analyses. The dates should be entered in MM/DD/YYYY format.

3.8.2.2.4 In the box (upper for Pesticides) in Form III, under "AMOUNT ADDED", enter the amount in nanograms (ng) of each analyte added to the sample. Under "AMOUNT RECOVERED", enter the amount in ng of each analyte in the sample calculated from analysis. Calculate the percent recovery of each compound in the sample to the nearest whole percent, according to Exhibit D, and enter under "% REC". Flag all percent recoveries which do not meet the contract requirements with an asterisk (*). The asterisk must be placed in the last space of the percent recovery column, under the "#" symbol.

3.8.2.2.5 Complete the lower box according to the instructions in Section 3.8.2.2.4.

3.8.2.2.6 Summarize the values outside the QC limits at the bottom of the page.

NOTE: This means the results for both columns.

3.9 Method Blank Summary (Form IV, All Fractions)

3.9.1 Purpose. This form summarizes the samples associated with each method blank analysis. The Contractor shall submit the appropriate Form IV for each blank.

3.9.2 Instructions. Complete the header information according to the instructions in Section 3.3. The EPA sample number entered in the upper right-hand corner shall be the same number entered on Form I for the blank. Complete the remainder of the form using the following instructions.

3.9.2.1 Complete the following fields: "Instrument ID", "Date Analyzed", and "Time Analyzed". Dates shall be entered as MM/DD/YYYY. The time shall be reported in military time.

3.9.2.2 Pesticide/Aroclor contaminants shall meet the identification criteria requiring analysis of the blank on two different GC columns (see Exhibit D PEST). Enter the date, time, and instrument ID of both analyses of the blank on the pesticide method blank summary (Form IV LCP). The information on the two analyses is differentiated as Date Analyzed (1), Date Analyzed (2), etc. If the analyses were run simultaneously, the order of reporting is not important, but shall be consistent with the information reported on all other pesticide forms. Otherwise, Date Analyzed (1) shall indicate the analysis on column 1, and Date Analyzed (2) shall indicate the analysis on column 2.

3.9.2.3 For pesticide/Aroclor blanks, enter the method of extraction as "SEPF" for separatory funnel, or "CONT" for continuous liquid-liquid extraction on Form IV LCP.

3.9.2.4 Identify the GC column, internal diameter, and length in the appropriate fields, as indicated in Section 3.3.

3.9.2.5 For semivolatile and pesticide/Aroclor method blanks, enter the date of extraction of the blank on Form IV LCSV or LCP.

3.9.2.6 If the samples associated with pesticide/Aroclor blank are subjected to sulfur cleanup, then the blank shall also be subjected to sulfur cleanup. If sulfur cleanup is employed, enter Y in the "Sulfur Cleanup" field; if not, enter N on Form IV LCP. If only some of the samples associated with the method blank are subjected to sulfur cleanup, a separate sulfur cleanup blank is required (see Exhibit D PEST). If a separate sulfur cleanup blank is prepared, complete one version of Form IV associating all the samples with the method blank, and a second version of Form IV listing only those samples associated with the separate sulfur cleanup blank.

NOTE: Subjecting all samples associated with a method blank to sulfur cleanup avoids the need for two forms.

3.9.2.7 For all three fractions, as appropriate, summarize the samples associated with a given method blank in the table, entering the EPA sample number and lab sample identifier. For volatiles, enter the lab file identifier and the time of analysis of each sample. For semivolatiles, enter the lab file identifier and date of analysis. For pesticides/Aroclors, enter the dates of both analyses as Date Analyzed (1) and Date Analyzed (2), as discussed previously.

3.9.2.8 Number all pages as described in Section 3.3.

3.10 GC/MS Instrument Performance Check (Form V LCV and Form V LCSV)

3.10.1 Purpose. This form is used to report the results of the GC/MS instrument performance check for the volatile and semivolatile fractions and to summarize the date and time of analyses of samples, including dilutions and re-analyses, standards, blanks, and requested MS/MSD associated with each analysis of the instrument performance check solution.

3.10.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.10.2.1 Enter the date and time of injection of the instrument performance check solution (BFB for volatiles--CAS Number 460004, DFTPP for semivolatiles--CAS Number 5074715). The date shall be entered as MM/DD/YYYY. The time shall be reported as military time.

3.10.2.2 For volatiles, identify the GC column, internal diameter, and column length on Form V LCV, as described in Section 3.3.

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

NOTE: For both BFB and DFTPP, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak, m/z 95 for BFB and m/z 198 for DFTPP. Despite this possibility, all ion abundances shall be normalized to the nominal base peaks listed on Form V (see Exhibits D).

3.10.2.4 All relative abundances shall be reported as a number. If the relative abundance is zero, enter 0 (zero), not a dash or other non-numeric character. Where parentheses appear, compute the percentage of the ion abundance of the mass given in the appropriate footnote, and enter that value in the parentheses.

3.10.2.5 In the lower table, list all samples, including dilutions, re-analyses, standards, blanks and requested MS/MSD analyzed under that instrument performance check in chronological order, by time of analysis (in military time). Refer to Section 3.3.7 for specific instructions for identifying standards and blanks.

3.10.2.6 Complete the following fields for all standards, samples, including dilutions, re-analyses, and blanks: "EPA Sample No.", "Lab Sample ID", "Lab File ID", "Date Analyzed", and "Time Analyzed".

3.10.2.7 Number all pages as described in Section 3.3.

3.11 GC/MS Initial Calibration Data (Form VI LCV-1, LCV-2, LCV-3 and Form VI LCSV-1, LCSV-2, LCSV-3)

3.11.1 Purpose. After a GC/MS system has undergone an initial five-point calibration at the specific concentration levels described in Exhibit D, and after all initial calibration criteria have been met, the Contractor shall complete and submit this form for each volatile or semivolatile target compound initial calibration performed which is relevant to the samples, including dilutions, re-analyses, and blanks in the SDG, regardless of when that calibration was performed.

3.11.2 Instructions. Complete the header information according to the instructions in Section 3.3. Enter the Case number and SDG number for the current data package, regardless of the original Case for which the initial calibration was performed. Complete the remainder of the form using the following instructions.

3.11.2.1 Enter the date(s) of the calibration. If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded. Dates shall be entered as MM/DD/YYYY.

3.11.2.2 Enter the injection times of the first and last of the standards analyzed in the "Calibration Times" field. Times shall be reported in military time.

3.11.2.3 Enter the lab file identifier for each of the five calibration standards injected. Complete the response factor data for the five calibration points, and then calculate and report the average Relative Response Factor (RRF) for all target compounds.

3.11.2.4 For volatiles and semivolatiles, report the RRFs for the deuterated monitoring compounds in the calibration standards. The Contractor shall report the Relative Standard Deviation (%RSD) for <u>all</u> compounds. See Exhibit D for equations.

3.12 GC/EC Initial Calibration Data (Form VI LCP-1, LCP-2, LCP-3)

3.12.1 Purpose. The initial calibration of pesticides/Aroclors involves the determination of retention times, retention time windows, and calibration factors. For single component pesticide target compounds, these data are calculated from the analyses of the Individual Standard Mixtures A and B at three different concentration

levels. For the multicomponent target compounds, these data are calculated from a single point calibration.

3.12.2 Instructions. Complete one Form VI for <u>each</u> GC column used for the three analyses of Individual Standard Mixture A (low-point, mid-point, and high-point) and the three analyses of Individual Standard Mixture B during an initial calibration. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.12.2.1 In the "Level (x low)" field, enter the concentration of the low-point, mid-point, and high-point calibration standards as a multiplier of the low-point. Therefore, for the low-point, enter "1.0." The concentration of the mid-point standard is specified in Exhibit D as four times the low-point; therefore, enter "4.0." The high-point standard shall be at least 16 times the low-point, but may be higher, if that value lies within the linear range of the instrument, as specified in Exhibit D. Therefore, enter the appropriate multiplier to the high-point standard concentration to one decimal place.

3.12.2.2 Identify the GC column and internal diameter (in mm) in the appropriate fields.

3.12.2.3 Enter the dates of analysis of the first and last of the six standards on each form in the "Date(s) Analyzed" field. Dates shall be entered as MM/DD/YYYY.

3.12.2.4 For each standard analyzed, enter the retention time of each applicable analyte in minutes and decimal minutes, under the appropriate concentration level in the "RT OF STANDARDS" column on Form VI LCP-1.

3.12.2.5 Calculate the mean retention time of each analyte from the three individual mixtures, and report it in the "MEAN RT" column on Form VI LCP-1.

3.12.2.6 Calculate the retention time window for each analyte using the specifications in Exhibit D, and enter the lower limit of the window in the "RT WINDOW" column under "FROM," and the upper limit of the window under "TO" on Form VI LCP-1. The retention times of the surrogates are reported from the analyses of Individual Mixture A and the windows are only required to be calculated for Individual Mixture A.

3.12.2.7 For the six analyses of the Individual Standard Mixtures, the Contractor shall also complete the calibration factor data on Form VI LCP-2. Prepare one form for each instrument and GC column used. Enter the calibration factor for each compound in each of the standards. Calculate and enter a mean calibration factor and a relative standard deviation (%RSD). As with surrogate retention times, the surrogate calibration factors are only required from Individual Mixture A analyses.

3.12.2.8 For the multicomponent target compounds, the retention times, retention time windows, and calibration factors shall be reported in a similar fashion for each single point calibration standard. For each multicomponent compound, the Contractor shall select at least three peaks from each analyte, according to the specifications in Exhibit D. The retention time and calibration factor data apply to <u>each</u> peak. Complete one version of Form VI

LCP-3 for each GC column, for each initial calibration that applies to samples in the data package.

3.12.3 Form VI (LCP-4) is also used to report the results of analysis of the Resolution Check Solution that shall begin each pesticide/Aroclor initial calibration sequence. The Contractor shall submit one Form VI LCP-4 for both GC columns.

3.12.3.1 Complete the header information as described in Section 3.3. Using the same assignment of first and second GC columns made for Form IV, enter the GC column identifier, internal diameter, and date and time of analysis. Enter the EPA sample number for the Resolution Check Standard. If simultaneous injections on a single GC are used, the EPA sample number may be the same for both Resolution Check Standards. If simultaneous injections are not used, use different suffixes to identify the standards. Complete the remainder of the form using the following instructions.

3.12.3.2 List each analyte, in retention time order, including both surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the Resolution Check Mixture as it appears on Form I LCP. Spell out the names of the surrogates as they appear on Form II LCP-2.

3.12.3.3 Enter the retention time of each analyte from the analysis in the "RT" column.

3.12.3.4 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered.

NOTE: Only eight of the nine resolution fields will be filled.

3.12.4 Form VI (LCP-5, LCP-6 and LCP-7 for each PEM, initial mid-level calibration mixture A, and initial mid-level calibration mixture B, respectively) shall be used to report the percent resolution between each pair of analytes according to the definition in Exhibit D Pesticides and Aroclors.

NOTE: These forms shall also be used to report all percent resolution data for the PEM and midpoint concentration Individual Mixtures A and B analyzed as part of calibration verification (Exhibit D/PEST, Section 9.3).

3.12.4.1 Complete the header information as described in Section 3.3. Using the same assignment of first and second GC columns made for Form IV, enter the GC column identifier, internal diameter, and date and time of analysis. Enter the EPA sample number for the respective standards. If simultaneous injections are not used, use different suffixes to identify the standards. Complete the remainder of the form using the following instructions.

3.12.4.2 List each analyte, in retention time order, including both surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of

each target analyte in the standard as it appears on Form I LCP. Spell out the names of the surrogates as they appear on Form II LCP-2.

3.12.4.3 Enter the retention time of each analyte from the analysis in the "RT" column.

3.12.4.4 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered.

NOTE: The last resolution field will be left blank in each table.

3.13 GC/MS Continuing Calibration Data (Form VII LCV-1, LCV-2, LCV-3 and Form VII LCSV-1, LCSV-2, LCSV-3)

3.13.1 Purpose. For volatiles and semivolatiles, this form is used to report the calibration of the GC/MS system by the analysis of specific calibration standards. Form VII is required for each 12-hour time period for both volatile and semivolatile target compound analyses. The Contractor shall analyze calibration standards and meet all criteria outlined in Exhibit D for the minimum Relative Response Factors (RRF) and maximum percent difference between initial and continuing calibrations.

3.13.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.13.2.1 Enter the date and time of the continuing calibration and the dates and times of the initial calibration (give inclusive dates if the initial calibration is performed over more than one date). Dates shall be entered as MM/DD/YYYY. Times shall be reported in military time.

3.13.2.2 Using the appropriate initial calibration (volatile or semivolatile), enter the average RRF for each target compound and each deuterated monitoring compound for volatiles and semivolatiles.

3.13.2.3 Report the RRF (RRF5 for Volatiles and RRF20 for Semivolatiles) from the continuing calibration standard analysis.

3.13.2.4 Calculate the percent difference (%D) for all compounds. See Exhibit D for equation. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.

3.14 GC/ECD Calibration Verification Summary (Form VII, LCP-1, LCP-2)

3.14.1 Purpose. Form VII is used to report the results of the Performance Evaluation Mixtures (PEMs) and the mid-point concentrations of Individual Standard Mixtures A and B that, along with the PEM, bracket each 12-hour period of sample analyses. The Contractor shall submit Form VII LCP-1 for each 12-hour sequence analyzed. Form VII LCP-2 shall be completed each time the Individual Standard Mixtures are analyzed, for each GC column used.

3.14.2 Instructions. Complete Form VII LCP-1 and LCP-2 for each standard reported on Form VIII LCP. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

FORM VII LCP-1

3.14.2.1 Enter the date(s) of the initial calibration(s). Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YYYY.

3.14.2.2 Identify the GC column and internal diameter in the appropriate fields.

3.14.2.3 On Form VII LCP-1, enter the EPA sample number, lab sample identifier and date and time of analysis for the instrument blank that preceded the 12-hour sequence (PIBLK). For the PEM that initiated or terminated the 12-hour sequence (PEM), enter the EPA sample number, lab sample identifier, and date and time of analysis.

3.14.2.4 When reporting data for the PEM at the beginning of the initial calibration sequence, leave the "EPA Sample No.", "Lab Sample ID", "Date Analyzed", and "Time Analyzed" fields blank for the instrument blank (PIBLK), when no instrument blank is analyzed before the PEM. When reporting all other PEM analyses, the instrument blank fields shall be completed.

3.14.2.5 In the table, report the retention time for each analyte in the PEM as well as the retention time windows.

3.14.2.6 For each analyte in the PEM, enter the amount of the analyte found in the PEM, in ng to three decimal places, in the "CALC AMOUNT" column.

3.14.2.7 Enter the nominal amount (amount added) of each analyte in the PEM in ng to three decimal places in the "NOM AMOUNT" column.

3.14.2.8 Calculate the percent difference between the calculated amount and nominal amount for each analyte according to Exhibit D. Report the values in the "%D" column. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.

3.14.2.9 Calculate the percent breakdown for endrin and 4,4'-DDT and the combined percent breakdown in the PEM according to Exhibit D. Enter the values for the breakdown of endrin and 4,4'-DDT in their respective fields immediately under the table.

FORM VII LCP-2

3.14.2.10 The upper table on Form VII LCP-2 contains the retention time and amount data for Individual Standard Mixture A compounds. The lower table contains the data for Mixture B. Complete the form using the instructions in Sections 3.14.2.1 through 3.14.2.8 for Form VII LCP-1.

3.15 Internal Standard Area and RT Summary (Form VIII LCV and Form VIII LCSV-1, LCSV-2)

3.15.1 Purpose. This form is used to summarize the peak areas and retention times of the internal standards added to the initial calibration

standards, continuing calibration standards and all volatile and semivolatile samples, including dilutions, re-analyses, and blanks. The data are used to determine when changes in internal standard responses will adversely affect quantitation of target compounds. This form shall be completed each time an initial calibration and continuing calibration is performed, or when samples are analyzed under the same GC/MS instrument performance check as an initial calibration.

3.15.2 Instructions. Complete the header information according to Section 3.3. Complete the remainder of the form using the following instructions. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a continuing calibration, Form VIII shall be completed on the basis of the internal standard areas of the 5 µg/L initial calibration standard for volatiles, and the 20 ng initial calibration standard for semivolatiles. Use the date and time of analysis of this standard and the lab file identifier and areas in place of those of a continuing calibration standard.

3.15.2.1 Enter the date and time of analysis of the continuing calibration standard. The date shall be entered as MM/DD/YYYY. The time shall be reported in military time.

3.15.2.2 For volatiles, enter the GC column identifier, internal diameter, and length as directed in Section 3.3.

3.15.2.3 From the results of the analysis of the continuing calibration standard, enter the area measured for each internal standard and its retention time (in decimal minutes) under the appropriate column in the "12 HOUR STD" row.

3.15.2.4 For each internal standard listed in Tables 5 and 6, calculate the upper limit of the area and the lower limit of the area from the internal standard area. For FORM VIII LCV, calculate the upper limit of the area as the area of the particular internal standard plus 40 percent of its area (i.e., 1.4 times the area in the "12 HOUR STD" field), and the lower limit of the area as the area of the internal standard minus 40 percent of its area (i.e., 0.6 times the area in the "12 HOUR STD" field). For FORMS VIII LCSV-1 and VIII LCSV-2, calculate the upper limit of the area as the area of the particular internal standard plus 100 percent of its area (i.e., two times the area in the "12 HOUR STD" field), and the lower limit of the area as the area of the internal standard minus 50 percent of its area (i.e., 0.5 times the area in the "12 HOUR STD" field). Report these values in the "UPPER LIMIT" and "LOWER LIMIT" rows, respectively. Calculate the upper limit of the retention time and the lower limit of the retention time. The upper limit of the retention time is calculated by adding 0.33 minutes to the retention time of the internal standard and the lower limit of the retention time is the retention time of the internal standard minus 0.33 minutes. Report these values in the "UPPER LIMIT" and "LOWER LIMIT" rows in the applicable RT columns.

3.15.2.5 For each sample, including dilutions, re-analyses, blanks, and requested MS/MSD analyzed under a given continuing calibration, 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 in Section 3.15.2.4, flag that area with an asterisk (*). The asterisk shall be 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.

3.15.2.6 Number all pages as described in Section 3.3.

Table 5
Volatile Internal Standards

Volatile Internal Standards	CAS Number
IS1: Chlorobenzene-d5 (CBZ)	3114-55-4
IS2: 1,4-Difluorobenzene (DFB)	540-36-3
IS3: 1,4-Dichlorobenzene-d4 (DCB)	3855-82-1

Table 6
Semivolatile Internal Standards

Semivolatile Internal Standards	CAS Number
IS1: 1,4-Dichlorobenzene-d4 (DCB)	3855-82-1
IS2: Naphthalene-d8 (NPT)	1146-65-2
IS3: Acenaphthene-d10 (ANT)	15067-26-2
IS4: Phenanthrene-d10 (PHN)	1517-22-2
IS5: Chrysene-d12 (CRY)	1719-03-5
IS6: Perylene-d12 (PRY)	1520-96-3

3.16 Pesticide Analytical Sequence (Form VIII LCP)

3.16.1 Purpose. This form is used to report the analytical sequence for pesticide analysis. At least one form is required for each GC column used for pesticide/Aroclor analyses.

3.16.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.16.2.1 Enter the date(s) of the initial calibration. Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YYYY.

3.16.2.2 Identify the GC column and internal diameter in the appropriate fields.

3.16.2.3 At the top of the table, report the mean retention time for tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) calculated from the initial calibration sequence (from INDA).

3.16.2.4 For every analysis associated with a particular analytical sequence starting with the initial calibration, enter the EPA sample number, lab sample identifier, and date and time of analysis. Each sample analyzed as part of the sequence shall be reported on Form VIII LCP <u>even</u> if it is not associated with the SDG. The Contractor shall use ZZZZZ as the EPA sample number to

distinguish all samples that are not part of the SDG being reported.

3.16.2.5 Report the retention time of the surrogates for each analysis in the "TCX RT" and "DCB RT" columns. All sample analyses shall be bracketed by acceptable analyses of instrument blanks, a PEM, and Individual Standard Mixtures A and B. Given the fact that the initial calibration may remain valid for some time (Exhibit D), it is only necessary to report the data from 12-hour periods when samples, dilutions, re-analyses, Laboratory Control Samples, requested MS/MSD, blanks, or multicomponent standard analytes for the 72-hour confirmation requirement in an SDG were analyzed. The Contractor shall submit Form VIII for the initial calibration sequence and forms that include the PEMs and Individual Standard Mixtures that bracket any and all samples in the SDG. While the data for time periods between the initial calibration and samples in the SDG are not a routine deliverable, the data shall be available as requested (e.g., at on-site evaluations). Non-USEPA samples shall be numbered ZZZZZ.

3.16.2.6 Flag all those values which do not meet the contract requirements by entering an asterisk (*) in the "RT" column, under the "#" symbol. If the retention time cannot be calculated due to interfering peaks, leave the "RT" column blank for that surrogate, enter an asterisk in the last column, and document the problem in the SDG Narrative.

3.16.2.7 If more than a single copy of Form VIII LCP is required, enter the same header information on all subsequent pages for that GC column and instrument, and number each page as described in Section 3.3.

3.17 Pesticide Cleanup Summary (Form IX, LCP)

3.17.1 Purpose. Form IX LCP is used to report the results of the check of the Florisil cartridges used to process all sample extracts and to associate the lot of cartridges with particular sample results so that problems with a particular cartridge lot may be tracked across all associated samples.

3.17.2 Instructions. Complete the header information according to the instructions in Section 3.3. Enter the Case number and SDG number for the current data package, regardless of the original Case for which the cartridge check was performed. Complete the remainder of the form using the following instructions.

3.17.2.1 Enter the Florisil cartridge lot number.

3.17.2.2 Enter the date the Florisil cartridge check solution was analyzed in the "Date of Analysis" field. The date shall be entered as MM/DD/YYYY.

3.17.2.3 Complete the "GC Column" and "ID" fields for the two GC columns used to analyze the samples, including blanks, re-analyses, Laboratory Control Samples, and requested MS/MSD. Report all results from either GC Column 1 or GC Column 2.

3.17.2.4 In the first table, enter the amount of spike added and spike recovered in ng for each analyte.

3.17.2.5 Calculate the percent recovery to the nearest whole percent, and enter the number in the "% REC" field. Flag each spike recovery

outside the QC limits (shown on the form) with an asterisk (*). The asterisk shall be placed in the last space in the "% REC" column, under the "#" symbol.

3.17.2.6 In the second table, complete the "EPA Sample No.," the "Lab Sample ID," and "Date Analyzed" fields for each sample and blank that were cleaned up using this lot of Florisil cartridges.

3.17.2.7 Number the pages as described in Section 3.3.

3.18 Pesticide/Aroclor Identification (Form X, LCP-1, LCP-2)

3.18.1 Purpose. This form summarizes the quantitations of all target pesticides/Aroclors detected in a given sample. It reports the retention times of the compound on both columns on which it was analyzed, as well as the retention time windows of the standard for that compound on both of these columns. In addition, it is used to report the concentration determined from each GC column, and the percent difference between the two quantitative results. Separate forms are used for single component analytes and multicomponent analytes.

Form X is required for each sample, including dilutions, re-analyses, blanks, Laboratory Control Samples and requested MS/MSD in which compounds listed in Exhibit C (Pesticides/Aroclors) are reported on Form I. **Do not generate a Form X for pesticide instrument blanks.**

3.18.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.18.2.1 Enter the date(s) of analysis. Dates shall be entered as MM/DD/YYYY.

3.18.2.2 Enter the GC column and internal diameter for each of the two columns.

3.18.2.3 For each single component pesticide positively identified, enter the name of the compound in the "ANALYTE" column as it appears on Form I.

3.18.2.4 Enter the retention times on each column of the compounds detected in the sample next to the appropriate column designation (1 or 2).

3.18.2.5 Enter the retention time windows on each column from the initial calibration standard. These data shall correspond with those on Form VI and shall be entered in a similar manner. The lower value is entered under the "FROM" column, the upper value under the "TO" column.

3.18.2.6 Enter the concentration calculated from each GC column under the "CONCENTRATION" column. Although the units are the same as those used on Form I, do <u>not</u> enter any units on Form X.

3.18.2.7 Calculate the percent difference between the concentrations entered on this form, using the equation found in Exhibit D, and report it to a tenth of a percent in the "%D" column. If %D is greater that 999.9, report it as 999.9.

3.18.2.8 The <u>lower</u> of the two concentrations is reported on Form I for each pesticide compound. The lower concentration is used because, if

present, coeluting interferences are likely to increase the calculated concentration of any target compound. If the percent difference between the calculated concentrations is greater than 25.0 percent, flag the concentration on Form I, as described previously. This will alert the data user to the potential problems in quantitating this analyte.

3.18.2.9 If more pesticide compounds are identified in an individual sample than can be reported on one Form X, complete as many additional copies of Form X as necessary, duplicating all header information and numbering the pages as described in Section 3.3.

3.18.2.10 Report multicomponent analytes detected in samples on Form X LCP-2. Complete the header information and GC column fields as described above. For multicomponent analytes, it is necessary to report the retention time and concentration of each peak chosen for quantitation in the target analyte in a fashion similar to that for single component pesticides. The concentrations of all peaks quantitated (three are required, up to five may be used) are averaged to determine the mean concentration. Report the lower of the two mean concentrations on Form I. Flag this value if the mean concentrations from the two GC columns differ by more than 25 percent, as described previously.

3.18.2.11 If more multicomponent compounds are identified in an individual sample than can be reported on one Form X, complete as many additional copies of Form X as necessary, duplicating all header information and numbering the pages as described in Section 3.3.

3.19 Sample Log-In Sheet (Form DC-1)

3.19.1 Purpose. This form is used to document the receipt and inspection of sample containers and samples. One original of Form DC-1 is required for each sample shipping container (only the hardcopy form is required). If the samples in a single sample shipping container are assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG of the lowest alpha-numeric number; and a copy of Form DC-1 shall be placed with the deliverables for the other SDGs. The copies shall be identified as "copy(ies)", and the location of the original shall be noted on the copies.

3.19.2 Instructions

3.19.2.1 Sign and date the airbill. (If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information).

3.19.2.2 Complete the header information on the form, including the log-in date.

3.19.2.3 Examine the shipping container and record the presence/absence of custody seals and their condition (e.g., intact, broken) in item 1.

3.19.2.4 Record the custody seal numbers in item 2.

3.19.2.5 Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (e.g., TRs, Packing Lists), and airbills or airbill stickers in items 3-5. Specify if there is an airbill present or an

airbill sticker in item 5. Record the airbill or sticker number in item 6.

3.19.2.6 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (e.g., intact, broken, leaking) and presence or absence of sample tags in items 7 and 8.

3.19.2.7 Record the presence of the cooler temperature indicator bottle in item 9 and cooler temperature in item 10.

3.19.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and circle the appropriate answer in item 11.

3.19.2.9 Record the date and time of cooler receipt at the laboratory in items 12 and 13.

3.19.2.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1, the Chain-of-Custody record, and the TR, and write the sample numbers on Form DC-1 in the "EPA Sample #" column.

3.19.2.11 Record the appropriate sample tags and assigned laboratory numbers, if applicable.

3.19.2.12 Any comments should be made in the "Remarks" column.

3.19.2.13 Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the "Sample Transfer" block. Sign and date the "Sample Transfer" block.

3.19.2.14 Cross out unused columns and spaces. Initial and date all cross outs.

3.19.2.15 If there are problems observed during receipt or an answer marked with an asterisk (e.g., "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 and note, where appropriate, the resolution of the problem.

3.20 Complete SDG File (CSF) Inventory Sheet (Form DC-2)

3.20.1 Purpose. Form DC-2 is used to record both the CSF documents and the number of documents in the original Sample Data Package sent to the USEPA Region.

3.20.2 Instructions

3.20.2.1 Organize all USEPA CSF documents as described in Section 2.6. Assemble the documents in the order specified on Form DC-2 and Section 2.6, and stamp each page with a consecutive number; however, do not number Form DC-2. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (e.g., "page numbers not used, XXXX - XXXX, YYYY - YYYY. If there are no documents for a specific document type, enter an "NA" in the empty space.

3.20.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The Contractor shall review Form DC-2 to determine if it is most appropriate to place them under categories 7, 8, 9, or 10. Category 10 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 on Form DC-2.

3.20.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall identify the documents with unique accountable numbers and record the unique accountable numbers and the locations of the documents in the CSF (in the "Other Records" section on Form DC-2).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

1LCA
LOW CONCENTRATION WATER VOLATILE ORGANICS ANALYSIS
DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Analyzed: _____

Purge Volume: _____(ML) Dilution Factor: _____

GC Column: _____ ID: _____(MM) Length: _____(M)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (UG/L)	Q
75-71-8	Dichlorodifluoromethane		
74-87-3	Chloromethane		
75-01-4	Vinyl Chloride		
74-83-9	Bromomethane		
75-00-3	Chloroethane		
75-69-4	Trichlorofluoromethane		
75-35-4	1,1-Dichloroethene		
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane		
67-64-1	Acetone		
75-15-0	Carbon Disulfide		
79-20-9	Methyl Acetate		
75-09-2	Methylene Chloride		
156-60-5	trans-1,2-Dichloroethene		
1634-04-4	Methyl tert-Butyl Ether		
75-34-3	1,1-Dichloroethane		
156-59-2	cis-1,2-Dichloroethene		
78-93-3	2-Butanone		
74-97-5	Bromochloromethane		
67-66-3	Chloroform		
71-55-6	1,1,1-Trichloroethane		
110-82-7	Cyclohexane		
56-23-5	Carbon Tetrachloride		
71-43-2	Benzene		
107-06-2	1,2-Dichloroethane		

1LCB
LOW CONCENTRATION WATER VOLATILE ORGANICS ANALYSIS
DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Analyzed: _____

Purge Volume: _____(ML) Dilution Factor: _____

GC Column: _____ ID: _____(MM) Length: _____(M)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (UG/L)	Q
79-01-6	Trichloroethene		
108-87-2	Methylcyclohexane		
78-87-5	1,2-Dichloropropane		
75-27-4	Bromodichloromethane		
10061-01-5	cis-1,3-Dichloropropene		
108-10-1	4-Methyl-2-pentanone		
108-88-3	Toluene		
10061-02-6	trans-1,3-Dichloropropene		
79-00-5	1,1,2-Trichloroethane		
127-18-4	Tetrachloroethene		
591-78-6	2-Hexanone		
124-48-1	Dibromochloromethane		
106-93-4	1,2-Dibromoethane		
108-90-7	Chlorobenzene		
100-41-4	Ethylbenzene		
1330-20-7	Xylene (total)		
100-42-5	Styrene		
75-25-2	Bromoform		
98-82-8	Isopropylbenzene		
79-34-5	1,1,2,2-Tetrachloroethane		
541-73-1	1,3-Dichlorobenzene		
106-46-7	1,4-Dichlorobenzene		
95-50-1	1,2-Dichlorobenzene		
96-12-8	1,2-Dibromo-3-chloropropane		
120-82-1	1,2,4-Trichlorobenzene		
87-61-6	1,2,3-Trichlorobenzene		

1LCC
LOW CONCENTRATION WATER SEMIVOLATILE ORGANICS
ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Extracted: _____

Sample Volume: _____ (ML) Date Analyzed: _____

Concentrated Extract Volume: _____ (UL) Dilution Factor: _____

Injection Volume: _____ (UL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (UG/L)	Q
100-52-7	Benzaldehyde		
108-95-2	Phenol		
111-44-4	bis(2-Chloroethyl)ether		
95-57-8	2-Chlorophenol		
95-48-7	2-Methylphenol		
108-60-1	2,2'-oxybis(1-Chloropropane)		
98-86-2	Acetophenone		
106-44-5	4-Methylphenol		
621-64-7	N-Nitroso-di-n-propylamine		
67-72-1	Hexachloroethane		
98-95-3	Nitrobenzene		
78-59-1	Isophorone		
88-75-5	2-Nitrophenol		
105-67-9	2,4-Dimethylphenol		
111-91-1	bis(2-Chloroethoxy)methane		
120-83-2	2,4-Dichlorophenol		
91-20-3	Naphthalene		
106-47-8	4-Chloroaniline		
87-68-3	Hexachlorobutadiene		
105-60-2	Caprolactam		
59-50-7	4-Chloro-3-methylphenol		
91-57-6	2-Methylnaphthalene		
77-47-4	Hexachlorocyclopentadiene		
88-06-2	2,4,6-Trichlorophenol		
95-95-4	2,4,5-Trichlorophenol		
92-52-4	1,1'-Biphenyl		
91-58-7	2-Chloronaphthalene		
88-74-4	2-Nitroaniline		
131-11-3	Dimethylphthalate		
606-20-2	2,6-Dinitrotoluene		
208-96-8	Acenaphthylene		
99-09-2	3-Nitroaniline		
83-32-9	Acenaphthene		

1LCD

LOW CONCENTRATION WATER SEMIVOLATILE ORGANICS ANALYSIS
DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Extracted: _____

Sample Volume: _____ (ML) Date Analyzed: _____

Concentrated Extract Volume: _____ (UL) Dilution Factor: _____

Injection Volume: _____ (UL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (UG/L)	Q
51-28-5	2,4-Dinitrophenol		
100-02-7	4-Nitrophenol		
132-64-9	Dibenzofuran		
121-14-2	2,4-Dinitrotoluene		
84-66-2	Diethylphthalate		
86-73-7	Fluorene		
7005-72-3	4-Chlorophenyl-phenylether		
100-01-6	4-Nitroaniline		
534-52-1	4,6-Dinitro-2-methylphenol		
86-30-6	N-Nitrosodiphenylamine (1)		
95-94-3	1,2,4,5 Tetrachlorobenzene		
101-55-3	4-Bromophenyl-phenylether		
118-74-1	Hexachlorobenzene		
1912-24-9	Atrazine		
87-86-5	Pentachlorophenol		
85-01-8	Phenanthrene		
120-12-7	Anthracene		
84-74-2	Di-n-butylphthalate		
206-44-0	Fluoranthene		
129-00-0	Pyrene		
85-68-7	Butylbenzylphthalate		
91-94-1	3,3'-Dichlorobenzidine		
56-55-3	Benzo(a)anthracene		
218-01-9	Chrysene		
117-81-7	bis(2-Ethylhexyl)phthalate		
117-84-0	Di-n-octylphthalate		
205-99-2	Benzo(b)fluoranthene		
207-08-9	Benzo(k)fluoranthene		
50-32-8	Benzo(a)pyrene		
193-39-5	Indeno(1,2,3-cd)pyrene		
53-70-3	Dibenzo(a,h)anthracene		
191-24-2	Benzo(g,h,i)perylene		

(1) Cannot be separated from Diphenylamine

1LCE

LOW CONCENTRATION WATER PESTICIDE ORGANICS ANALYSIS
DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Sample Volume: _____ (ML) Date Extracted: _____

Concentrated Extract Volume: _____ (UL) Date Analyzed: _____

Injection Volume: _____ (UL) Dilution Factor: _____

Sulfur Cleanup: (Y/N) _____ Extraction: (Sepf/Cont) _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (UG/L)	Q
319-84-6	alpha-BHC		
319-85-7	beta-BHC		
319-86-8	delta-BHC		
58-89-9	gamma-BHC (Lindane)		
76-44-8	Heptachlor		
309-00-2	Aldrin		
1024-57-3	Heptachlor epoxide		
959-98-8	Endosulfan I		
60-57-1	Dieldrin		
72-55-9	4,4'-DDE		
72-20-8	Endrin		
33213-65-9	Endosulfan II		
72-54-8	4,4'-DDD		
1031-07-8	Endosulfan sulfate		
50-29-3	4,4'-DDT		
72-43-5	Methoxychlor		
53494-70-5	Endrin ketone		
7421-93-4	Endrin aldehyde		
5103-71-9	alpha-Chlordane		
5103-74-2	gamma-Chlordane		
8001-35-2	Toxaphene		
12674-11-2	Aroclor-1016		
11104-28-2	Aroclor-1221		
11141-16-5	Aroclor-1232		
53469-21-9	Aroclor-1242		
12672-29-6	Aroclor-1248		
11097-69-1	Aroclor-1254		
11096-82-5	Aroclor-1260		

1LCF

LOW CONCENTRATION WATER VOLATILE ORGANICS ANALYSIS

DATA SHEET TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Analyzed: _____

Purge Volume: _____ (ML) Dilution Factor: _____

GC Column: _____ ID: _____ (MM) Length: _____(M)

Number TICs found: _____

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC. (UG/L)	Q
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					

1LCG

LOW CONCENTRATION WATER SEMIVOLATILE ORGANICS ANALYSIS

DATA SHEET TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Extracted: _____

Sample Volume: _____ (ML) Date Analyzed: _____

Concentrated Extract Volume: _____ (UL) Dilution Factor: _____

Injection Volume: _____ (UL)

Number TICs found: _____

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC. (UG/L)	Q
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
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19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					

2LCA

LOW CONCENTRATION WATER VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

	EPA SAMPLE NO.	VDMC1 (VCL) #	VDMC2 (CLA) #	VDMC3 (DCE) #	VDMC4 (BUT) #	VDMC5 (CLF) #	VDMC6 (DCA) #	VDMC7 (BEN) #
01								
02								
03								
04								
05								
06								
07								
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			QC LIMITS
VDMC1	(VCL)	= Vinyl Chloride-d3	(49-138)
VDMC2	(CLA)	= Chloroethane-d5	(60-126)
VDMC3	(DCE)	= 1,1-Dichloroethene-d2	(65-130)
VDMC4	(BUT)	= 2-Butanone-d5	(42-171)
VDMC5	(CLF)	= Chloroform-d	(80-123)
VDMC6	(DCA)	= 1,2-Dichloroethane-d4	(78-129)
VDMC7	(BEN)	= Benzene-d6	(78-121)

\# Column to be used to flag recovery values

* Values outside of contract required QC limits

Page __ of __

2LCB

LOW CONCENTRATION WATER VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

	EPA SAMPLE NO.	VDMC8 (DPA) #	VDMC9 (TOL) #	VDMC10 (TDP) #	VDMC11 (HEX) #	VDMC12 (BRF) #	VDMC13 (TCA) #	VDMC14 (DCZ) #	TOT OUT
01									
02									
03									
04									
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28									
29									
30									

		QC LIMITS
VDMC8 (DPA)	= 1,2-Dichloropropane-d6	(84-123)
VDMC9 (TOL)	= Toluene-d8	(77-120)
VDMC10 (TDP)	= trans-1,3-Dichloropropene-d4	(80-128)
VDMC11 (HEX)	= 2-Hexanone-d5	(37-169)
VDMC12 (BRF)	= Bromoform-d	(76-135)
VDMC13 (TCA)	= 1,1,2,2-Tetrachloroethane-d2	(75-131)
VDMC14 (DCZ)	= 1,2-Dichlorobenzene-d4	(50-150)

\# Column to be used to flag recovery values

* Values outside of contract required QC limits

Page __ of __

2LCC

LOW CONCENTRATION WATER SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

	EPA SAMPLE NO.	SDMC1 (PHL) #	SDMC2 (BCE) #	SDMC3 (2CP) #	SDMC4 (4MP) #	SDMC5 (NBZ) #	SDMC6 (2NP) #	SDMC7 (DCP) #	SDMC8 (4CA) #
01									
02									
03									
04									
05									
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28									
29									
30									

			QC LIMITS
SDMC1	(PHL)	= Phenol-d5	(10-110)
SDMC2	(BCE)	= bis-(2-Chloroethyl)ether-d8	(41-94)
SDMC3	(2CP)	= 2-Chlorophenol-d4	(33-110)
SDMC4	(4MP)	= 4-Methylphenol-d8	(38-95)
SDMC5	(NBZ)	= Nitrobenzene-d5	(35-114)
SDMC6	(2NP)	= 2-Nitrophenol-d4	(40-106)
SDMC7	(DCP)	= 2,4-Dichlorophenol-d3	(42-98)
SDMC8	(4CA)	= 4-Chloroaniline-d4	(8-70)

\# Column to be used to flag recovery values

* Values outside of contract required QC limits

D DMC diluted out

Page __ of __

2LCD

LOW CONCENTRATION WATER SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

	EPA SAMPLE NO.	SDMC9 (DMP) #	SDMC10 (ACY) #	SDMC11 (4NP) #	SDMC12 (FLR) #	SDMC13 (NMP) #	SDMC14 (ANC) #	SDMC15 (PYR) #	SDMC16 (BAP) #	TOT OUT
01										
02										
03										
04										
05										
06										
07										
08										
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29										
30										

		QC LIMITS
SDMC9 (DMP)	= Dimethylphthalate-d6	(62-102)
SDMC10 (ACY)	= Acenaphthylene-d8	(49-98)
SDMC11 (4NP)	= 4-Nitrophenol-d4	(9-181)
SDMC12 (FLR)	= Fluorene-d10	(50-97)
SDMC13 (NMP)	= 4,6-Dinitro-methylphenol-d2	(53-153)
SDMC14 (ANC)	= Anthracene-d10	(55-116)
SDMC15 (PYR)	= Pyrene-d10	(47-114)
SDMC16 (BAP)	= Benzo(a)pyrene-d12	(54-120)

\# Column to be used to flag recovery values

* Values outside of contract required QC limits

D DMC diluted out

2LCE

LOW CONCENTRATION WATER PESTICIDE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column(1): _____ ID: _____(MM) GC Column(2): _____ ID: _____(MM)

	EPA SAMPLE NO.	TCX 1 %REC #	TCX 2 %REC #	DCB 1 %REC #	DCB 2 %REC #	OTHER (1)	OTHER (2)	TOT OUT
01								
02								
03								
04								
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07								
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29								
30								

			QC LIMITS
TCX	=	Tetrachloro-m-xylene	(30-150)
DCB	=	Decachlorobiphenyl	(30-150)

\# Column to be used to flag recovery values

* Values outside of QC limits

D Surrogate diluted out

Page __ of __

3LCA
LOW CONCENTRATION WATER VOLATILE
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (UG/L)	SAMPLE CONCENTRATION (UG/L)	MS CONCENTRATION (UG/L)	MS % REC #	QC LIMITS REC.
1,1-Dichloroethene					61-145
Benzene					76-127
Trichloroethene					71-120
Toluene					76-125
Chlorobenzene					75-130

COMPOUND	SPIKE ADDED (UG/L)	MSD CONCENTRATION (UG/L)	MSD % REC #	RPD #	QC LIMITS RPD	QC LIMITS REC.
1,1-Dichloroethene					14	61-145
Benzene					11	76-127
Trichloroethene					14	71-120
Toluene					13	76-125
Chlorobenzene					13	75-130

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: _____ out of _____ outside limits
Spike Recovery: _____ out of _____ outside limits

COMMENTS: _____

_____

3LCB

LOW CONCENTRATION WATER SEMIVOLATILE

MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (UG/L)	SAMPLE CONCENTRATION (UG/L)	MS CONCENTRATION (UG/L)	MS % REC #	QC LIMITS REC.
Phenol					12-110
2-Chlorophenol					27-123
N-Nitroso-di-n-prop.(1)					41-116
4-Chloro-3-methylphenol					23-97
Acenaphthene					46-118
4-Nitrophenol					10-80
2,4-Dinitrotoluene					24-96
Pentachlorophenol					9-103
Pyrene					26-127

COMPOUND	SPIKE ADDED (UG/L)	MSD CONCENTRATION (UG/L)	MSD % REC #	RPD #	QC LIMITS RPD	QC LIMITS REC.
Phenol					42	12-110
2-Chlorophenol					40	27-123
N-Nitroso-di-n-prop.(1)					38	41-116
4-Chloro-3-methylphenol					42	23-97
Acenaphthene					31	46-118
4-Nitrophenol					50	10-80
2,4-Dinitrotoluene					38	24-96
Pentachlorophenol					50	9-103
Pyrene					31	26-127

(1) N-Nitroso-di-n-propylamine

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: _____out of _____ outside limits

Spike Recovery: _____ out of _____ outside limits

COMMENTS: _____

_____

3LCC

LOW CONCENTRATION WATER PESTICIDE

MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

Instrument ID: _____ GC Column: _____ ID: _____(mm)

COMPOUND	SPIKE ADDED (UG/L)	SAMPLE CONCENTRATION (UG/L)	MS CONCENTRATION (UG/L)	MS % REC #	QC LIMITS REC.
gamma-BHC (Lindane)					56-123
Heptachlor					40-131
Aldrin					40-120
Dieldrin					52-126
Endrin					56-121
4,4'-DDT					38-127

COMPOUND	SPIKE ADDED (UG/L)	MSD CONCENTRATION (UG/L)	MSD % REC #	RPD #	QC LIMITS RPD	QC LIMITS REC.
gamma-BHC (Lindane)					15	56-123
Heptachlor					20	40-131
Aldrin					22	40-120
Dieldrin					18	52-126
Endrin					21	56-121
4,4'-DDT					27	38-127

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: _____ out of _____ outside limits

Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

_____

Page __ of __

3LCD

LOW CONCENTRATION WATER PESTICIDE LAB CONTROL SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ LCS Lot No.: _____

LCS Aliquot: _____(UL) Date Extracted: _____

Concentrated Extract Volume: _____(UL) Date Analyzed: _____

Injection Volume: _____(UL) Dilution Factor: _____

Sulfur Cleanup: (Y/N) _____

Instrument ID (1): _____ GC Column (1): _____ ID: _____(MM)

COMPOUND	AMOUNT ADDED (NG)	AMOUNT RECOVERED (NG)	%REC #	QC LIMITS
gamma-BHC (Lindane)				50-120
Heptachlor epoxide				50-150
Dieldrin				30-130
4,4'-DDE				50-150
Endrin				50-120
Endosulfan sulfate				50-120
gamma-Chlordane				30-130

Instrument ID (2): _____ GC Column (2): _____ ID: _____(MM)

COMPOUND	AMOUNT ADDED (NG)	AMOUNT RECOVERED (NG)	%REC #	QC LIMITS
gamma-BHC (Lindane)				50-120
Heptachlor epoxide				50-150
Dieldrin				30-130
4,4'-DDE				50-150
Endrin				50-120
Endosulfan sulfate				50-120
gamma-Chlordane				30-130

Column to be used to flag recovery values with an asterisk

* Values outside of QC limits

LCS Recovery:_____ outside limits out of _____total.

4LCA

LOW CONCENTRATION WATER

VOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Analyzed: _____

Lab File ID: _____ Time Analyzed: _____

Instrument ID: _____

GC Column: _____ ID: _____ (MM) Length: _____(M)

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLE ANALYSES:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
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12				
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26				
27				
28				
29				
30				

COMMENTS: _____

_____

4LCB

LOW CONCENTRATION WATER

SEMIVOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date Extracted: _____

Lab File ID: _____ Date Analyzed: _____

Instrument ID: _____ Time Analyzed: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING ANALYSES:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01				
02				
03				
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27				
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29				
30				

COMMENTS: _____

_____

Page __ of __

4LCC

LOW CONCENTRATION WATER

PESTICIDE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Date Extracted: _____ Lab Sample ID: _____

Date Analyzed (1): _____ Date Analyzed (2): _____

Time Analyzed (1): _____ Time Analyzed (2): _____

Instrument ID (1): _____ Instrument ID (2): _____

GC Column (1): _____ ID: _____(MM) GC Column (2): _____ ID: _____(MM)

Sulfur Cleanup: (Y/N) _____ Extraction (Sepf/Cont): _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLE ANALYSES:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
01				
02				
03				
04				
05				
06				
07				
08				
09				
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14				
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16				
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18				
19				
20				
21				
22				
23				
24				
25				
26				

COMMENTS: _____

_____

Page __ of __

5LCA

LOW CONCENTRATION WATER VOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
BROMOFLUOROBENZENE (BFB)

Lab Name: _____ Contract: _____

Lab_Code: _____ Case_No.: _____ Client_No.: _____ SDG_No.: _____

Lab File ID: _____ BFB Injection Date: _____

Instrument ID: _____ BFB Injection Time: _____

GC Column: _____ ID: _____(MM) Column Length: _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	8.0 - 40.0% of mass 95	
75	30.0 - 66.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of mass 95	
173	Less than 2.0% of mass 174	( )1
174	50.0 - 120.0% of mass 95	
175	4.0 - 9.0 % of mass 174	( )1
176	93.0 - 101.0% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

1-Value is % mass 174 2-Value is % mass 176

THIS CHECK APPLIES TO THE FOLLOWING SAMPLES, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
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12					
13					
14					
15					
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18					
19					
20					
21					
22					

5LCB

LOW CONCENTRATION WATER SEMIVOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK

DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.:_____ SDG No.:_____

Lab File ID: _____ DFTPP Injection Date: _____

Instrument ID: _____ DFTPP Injection Time: _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0- 80.0% of mass 198	
68	Less than 2.0% of mass 69	( )1
69	Mass 69 relative abundance	
70	Less than 2.0% of mass 69	( )1
127	25.0 - 75.0% of mass 198	
197	Less than 1.0% of mass 198	
198	Base Peak, 100% relative abundance	
199	5.0 to 9.0% of mass 198	
275	10.0- 30.0% of mass 198	
365	Greater than 0.75% of mass 198	
441	Present, but less than mass 443	
442	40.0 - 110.0% of mass 198	
443	15.0 - 24.0% of mass 442	( )2

1-Value is % mass 69 2-Value is % mass 442

THIS CHECK APPLIES TO THE FOLLOWING SAMPLES, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
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6LCA
LOW CONCENTRATION WATER VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Times: _____ _____

GC Column: _____ ID: _____(MM) Length: _____ (M)

LAB FILE ID: RRF0.5 = _____ RRF1 = _____

RRF5 = _____ RRF10 = _____ RRF25 = _____

COMPOUND	RRF0.5	RRF1	RRF5	RRF10	RRF25	$\overline{RRF}$	% RSD
Dichlorodifluoromethane							
Chloromethane							
Vinyl Chloride *							*
Bromomethane *							*
Chloroethane							
Trichlorofluoromethane							
1,1-Dichloroethene *							*
1,1,2-Trichloro-1,2,2-trifluoroethane							
Acetone							
Carbon Disulfide							
Methyl Acetate							
Methylene Chloride							
trans-1,2-Dichloroethene							
Methyl tert-Butyl Ether							
1,1-Dichloroethane *							*
cis-1,2-Dichloroethene							
2-Butanone							
Bromochloromethane *							*
Chloroform *							*
1,1,1-Trichloroethane *							*
Cyclohexane							
Carbon Tetrachloride *							*
Benzene *							*
1,2-Dichloroethane *							*
Trichloroethene *							*
Methylcyclohexane							

*Compounds with required minimum RRF and maximum %RSD values.
All other compounds must meet a minimum RRF of 0.010.

6LCB

LOW CONCENTRATION WATER VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Times: _____ _____

GC Column: _____ ID: _____(MM) Length: _____(M)

LAB FILE ID: RRF0.5 = _____ RRF1 = _____

RRF5 = _____ RRF10 = _____ RRF25 = _____

COMPOUND	RRF0.5	RRF1	RRF5	RRF10	RRF25	$\overline{RRF}$	% RSD
1,2-Dichloropropane							
Bromodichloromethane *							*
cis-1,3-Dichloropropene *							*
4-Methyl-2-pentanone							
Toluene *							*
trans-1,3-Dichloropropene *							*
1,1,2-Trichloroethane *							*
Tetrachloroethene *							*
2-Hexanone							
Dibromochloromethane *							*
1,2-Dibromoethane *							*
Chlorobenzene *							*
Ethylbenzene *							*
Xylene (total) *							*
Styrene *							*
Bromoform *							*
Isopropylbenzene							
1,1,2,2-Tetrachloroethane *							*
1,3-Dichlorobenzene *							*
1,4-Dichlorobenzene *							*
1,2-Dichlorobenzene *							*
1,2-Dibromo-3-chloropropane							
1,2,4-Trichlorobenzene *							*
1,2,3-Trichlorobenzene *							*

*Compounds with required minimum RRF and maximum %RSD values.
All other compounds must meet a minimum RRF of 0.010.

6LCC

LOW CONCENTRATION WATER VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Times: _____ _____

GC Column: _____ ID: _____(MM) Length: _____(M)

LAB FILE ID: RRF0.5 = _____ RRF1 = _____

RRF5 = _____ RRF10 = _____ RRF25 = _____

COMPOUND	RRF0.5	RRF1	RRF5	RRF10	RRF25	$\overline{RRF}$	% RSD
Vinyl chloride-d3							
Chloroethane-d5							
1,1-Dichloroethene-d2							
2-Butanone-d5							
Chloroform-d							
1,2-Dichloroethane-d4							
Benzene-d6							
1,2-Dichloropropane-d6							
Toluene-d8							
trans-1,3-Dichloropropene-d4							
2-Hexanone-d5							
Bromoform-d							
1,1,2,2-Tetrachloroethane-d2							
1,2-Dichlorobenzene-d4							

*Compounds with required minimum RRF and maximum %RSD values.
All other compounds must meet a minimum RRF of 0.010.

6LCD

LOW CONCENTRATION WATER SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Times: _____ _____

LAB FILE ID: RRF5 = _____ RRF10 = _____

RRF20 = _____ RRF50 = _____ RRF80 = _____

COMPOUND	RRF5	RRF10	RRF20	RRF50	RRF80	$\overline{RRF}$	% RSD
Benzaldehyde							
Phenol *							*
bis-(2-Chloroethyl)ether *							*
2-Chlorophenol *							*
2-Methylphenol *							*
2,2'-oxybis(1-Chloropropane)							
Acetophenone							
4-Methylphenol *							*
N-Nitroso-di-n-propylamine *							*
Hexachloroethane *							*
Nitrobenzene *							*
Isophorone *							*
2-Nitrophenol *							*
2,4-Dimethylphenol *							*
bis(2-Chloroethoxy)methane *							*
2,4-Dichlorophenol *							*
Naphthalene *							*
4-Chloroaniline							
Hexachlorobutadiene							
Caprolactam							
4-Chloro-3-methylphenol *							*
2-Methylnaphthalene *							*
Hexachlorocyclopentadiene							
2,4,6-Trichlorophenol *							*
2,4,5-Trichlorophenol *							*
1,1'-Biphenyl							
2-Chloronaphthalene *							*
2-Nitroaniline							
Dimethylphthalate							
2,6-Dinitrotoluene *							*
Acenaphthylene *							*
3-Nitroaniline							
Acenaphthene *							*
2,4-Dinitrophenol							
4-Nitrophenol							
Dibenzofuran *							*

* Compounds with required minimum RRF and maximum %RSD values.
All other compounds must meet a minimum RRF of 0.010.

6LCE

LOW CONCENTRATION WATER SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Times: _____ _____

LAB FILE ID: RRF5 = _____ RRF10 = _____

RRF20 = _____ RRF50 = _____ RRF80 = _____

COMPOUND	RRF5	RRF10	RRF20	RRF50	RRF80	$\overline{RRF}$	% RSD
2,4-Dinitrotoluene *							*
Diethylphthalate							
Fluorene *							*
4-Chlorophenyl-phenylether *							*
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine (1)							
1,2,4,5 Tetrachlorobenzene							
4-Bromophenyl-phenylether *							*
Hexachlorobenzene *							*
Atrazine							
Pentachlorophenol *							*
Phenanthrene *							*
Anthracene *							*
Di-n-butylphthalate							
Fluoranthene *							*
Pyrene *							*
Butylbenzylphthalate							
3,3'-Dichlorobenzidine							
Benzo(a)anthracene *							*
Chrysene *							*
bis(2-Ethylhexyl)phthalate							
Di-n-octylphthalate							
Benzo(b)fluoranthene *							*
Benzo(k)fluoranthene *							*
Benzo(a)pyrene *							*
Indeno(1,2,3-cd)pyrene *							*
Dibenzo(a,h)anthracene *							*
Benzo(g,h,i)perylene *							*

(1) Cannot be separated from Diphenylamine

* Compounds with required minimum RRF and maximum %RSD values.

All other compounds must meet a minimum RRF of 0.010.

6LCF

LOW CONCENTRATION WATER SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Times: _____ _____

LAB FILE ID: RRF5 = _____ RRF10 = _____

RRF20 = _____ RRF50 = _____ RRF80 = _____

COMPOUND	RRF5	RRF10	RRF20	RRF50	RRF80	$\overline{RRF}$	% RSD
Phenol-d5							
bis-(2-Chloroethyl) ether-d8							
2-Chlorophenol-d4							
4-Methylphenol-d8							
Nitrobenzene-d5							
2-Nitrophenol-d4							
2,4-Dichlorophenol-d3							
4-Chloroaniline-d4							
Dimethylphthalate-d6							
Acenaphthylene-d8							
4-Nitrophenol-d4							
Fluorene-d10							
4,6-Dinitro-methylphenol-d2							
Anthracene-d10							
Pyrene-d10							
Benzo(a)pyrene-d12							

6LCG

LOW CONCENTRATION WATER PESTICIDE

INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Level (x low): low _____ mid _____ high _____

GC Column: _____ ID: _____(MM) Date(s) Analyzed: _____ _____

COMPOUND	RT OF STANDARDS			MEAN RT	RT WINDOW	
	LOW	MID	HIGH		FROM	TO
alpha-BHC						
beta-BHC						
delta-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Aldrin						
Heptachlor epoxide						
Endosulfan I						
Dieldrin						
4,4'-DDE						
Endrin						
Endosulfan II						
4,4'-DDD						
Endosulfan sulfate						
4,4'-DDT						
Methoxychlor						
Endrin ketone						
Endrin aldehyde						
alpha-Chlordane						
gamma-Chlordane						
Tetrachloro-m-xylene						
Decachlorobiphenyl						

* Surrogate retention times are measured from Standard Mix A analyses.

Retention time windows are ±0.05 minutes for all compounds that elute before Heptachlor expoxide, ±0.07 minutes for all other compounds, except ±0.10 minutes for Decachlorobiphenyl.

6LCH

LOW CONCENTRATION WATER PESTICIDE

INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Level (x low): low _____ mid _____ high _____

GC Column: _____ ID: _____(MM) Date(s) Analyzed: _____ _____

COMPOUND	CALIBRATION FACTORS LOW	MID	HIGH	MEAN	%RSD
alpha-BHC					
beta-BHC					
delta-BHC					
gamma-BHC (Lindane)					
Heptachlor					
Aldrin					
Heptachlor epoxide					
Endosulfan I					
Dieldrin					
4,4'-DDE					
Endrin					
Endosulfan II					
4,4'-DDD					
Endosulfan sulfate					
4,4'-DDT					
Methoxychlor					
Endrin ketone					
Endrin aldehyde					
alpha-Chlordane					
gamma-Chlordane					
Tetrachloro-m-xylene					
Decachlorobiphenyl					

* Surrogate calibration factors are measured from Standard Mix A analyses.

6LCI

LOW CONCENTRATION WATER PESTICIDE
INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Date(s) Analyzed: _____ _____

GC Column: _____ ID: _____(MM)

COMPOUND	AMOUNT (NG)	PEAK[1]	RT	RT WINDOW FROM	RT WINDOW TO	CALIBRATION FACTOR
Toxaphene		1				
		2				
		3				
		4				
		5				
Aroclor 1016		1				
		2				
		3				
		4				
		5				
Aroclor 1221		1				
		2				
		3				
		4				
		5				
Aroclor 1232		1				
		2				
		3				
		4				
		5				
Aroclor 1242		1				
		2				
		3				
		4				
		5				
Aroclor 1248		1				
		2				
		3				
		4				
		5				
Aroclor 1254		1				
		2				
		3				
		4				
		5				
Aroclor 1260		1				
		2				
		3				
		4				
		5				

[1]At least 3 peaks for each column are required for identification of multicomponent analytes.

6LCJ

LOW CONCENTRATION WATER PESTICIDE ANALYTE RESOLUTION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column (1): _____ ID: ____(MM) Instrument ID (1): _____

EPA Sample No. (RESC##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			

GC Column (2): _____ ID: ____(MM) Instrument ID (2): _____

EPA Sample No. (RESC##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			

6LCK

LOW CONCENTRATION WATER PERFORMANCE EVALUATION MIXTURE (PEM)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column (1): _____ ID: ____(MM) Instrument ID (1): _____

EPA Sample No. (PEM##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			

GC Column (2): _____ ID: _____(MM) Instrument ID (2): _____

EPA Sample No. (PEM##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			

6LCL

LOW CONCENTRATION WATER INDIVIDUAL STANDARD MIXTURE A (INDA)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column (1): _____ ID: ____(MM) Instrument ID (1): _____

EPA Sample No. (INDAM##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			

GC Column (2): _____ ID: ____(MM) Instrument ID (2): _____

EPA Sample No. (INDAM##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			

6LCM

LOW CONCENTRATION WATER INDIVIDUAL STANDARD MIXTURE B (INDB)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column (1): _____ ID: _____(MM) Instrument ID (1): _____

EPA Sample No. (INDBM##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			

GC Column (2): _____ ID: _____(MM) Instrument ID (2): _____

EPA Sample No. (INDBM##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			

7LCA

LOW CONCENTRATION WATER VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.:_____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

EPA Sample No.(VSTD005##): _____ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(MM) Length: _____ (M)

COMPOUND	$\overline{RRF}$	RRF5	MIN RRF	%D	MAX %D
Dichlorodifluoromethane					
Chloromethane					
Vinyl Chloride			0.100		30.0
Bromomethane			0.100		30.0
Chloroethane					
Trichlorofluoromethane					
1,1-Dichloroethene			0.100		30.0
1,1,2-Trichloro-1,2,2-trifluoroethane					
Acetone					
Carbon Disulfide					
Methyl Acetate					
Methylene Chloride					
trans-1,2-Dichloroethene					
Methyl tert-Butyl Ether					
1,1-Dichloroethane			0.200		30.0
cis-1,2-Dichloroethene					
2-Butanone					
Bromochloromethane			0.050		30.0
Chloroform			0.200		30.0
1,1,1-Trichloroethane			0.100		30.0
Cyclohexane					
Carbon Tetrachloride			0.100		30.0
Benzene			0.400		30.0
1,2-Dichloroethane			0.100		30.0
Trichloroethene			0.300		30.0
Methylcyclohexane					

All other compounds must meet a minimum RRF of 0.010.

7LCB

LOW CONCENTRATION WATER VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

EPA Sample No.(VSTD005##): _____ Init. Calib. Times: _____ _____

GC Column:_____ID:_____(MM) Length: _____ (M)

COMPOUND	$\overline{RRF}$	RRF5	MIN RRF	%D	MAX %D
1,2-Dichloropropane					
Bromodichloromethane			0.200		30.0
cis-1,3-Dichloropropene			0.200		30.0
4-Methyl-2-pentanone					
Toluene			0.400		30.0
trans-1,3-Dichloropropene			0.100		30.0
1,1,2-Trichloroethane			0.100		30.0
Tetrachloroethene			0.100		30.0
2-Hexanone					
Dibromochloromethane			0.100		30.0
1,2-Dibromoethane			0.100		30.0
Chlorobenzene			0.500		30.0
Ethylbenzene			0.100		30.0
Xylene (total)			0.300		30.0
Styrene			0.300		30.0
Bromoform			0.050		30.0
Isopropylbenzene					
1,1,2,2-Tetrachloroethane			0.100		30.0
1,3-Dichlorobenzene			0.400		30.0
1,4-Dichlorobenzene			0.400		30.0
1,2-Dichlorobenzene			0.400		30.0
1,2-Dibromo-3-chloropropane					
1,2,4-Trichlorobenzene			0.200		30.0
1,2,3-Trichlorobenzene			0.200		30.0

All other compounds must meet a minimum RRF of 0.010.

7LCC

LOW CONCENTRATION WATER VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

EPA Sample No.(VSTD005##): _____ Init. Calib. Times: _____ _____

GC Column:_____ID:_____(MM) Length: _____(M)

COMPOUND	$\overline{RRF}$	RRF5	MIN RRF	%D	MAX %D
Vinyl Chloride-d3					
Chloroethane-d5					
1,1-Dichloroethene-d2					
2-Butanone-d5					
Chloroform-d					
1,2-Dichloroethane-d4					
Benzene-d6					
1,2-Dichloropropane-d6					
Toluene-d8					
trans-1,3-Dichloropropene-d4					
2-Hexanone-d5					
Bromoform-d					
1,1,2,2-Tetrachloroethane-d2					
1,2-Dichlorobenzene-d4					

7LCD

LOW CONCENTRATION WATER SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

EPA Sample No.(SSTD020##): _____ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(MM)

COMPOUND	$\overline{RRF}$	RRF20	MIN RRF	%D	MAX %D
Benzaldehyde					
Phenol			0.800		25.0
bis-(2-Chloroethyl)ether			0.700		25.0
2-Chlorophenol			0.800		25.0
2-Methylphenol			0.700		25.0
2,2'-oxybis(1-Chloropropane)					
Acetophenone					
4-Methylphenol			0.600		25.0
N-Nitroso-di-n-propylamine			0.500		25.0
Hexachloroethane			0.300		25.0
Nitrobenzene			0.200		25.0
Isophorone			0.400		25.0
2-Nitrophenol			0.100		30.0
2,4-Dimethylphenol			0.200		30.0
bis(2-Chloroethoxy)methane			0.300		25.0
2,4-Dichlorophenol			0.200		25.0
Naphthalene			0.700		25.0
4-Chloroaniline					
Hexachlorobutadiene					
Caprolactam					
4-Chloro-3-methylphenol			0.200		25.0
2-Methylnaphthalene			0.400		25.0
Hexachlorocyclopentadiene					
2,4,6-Trichlorophenol			0.200		25.0
2,4,5-Trichlorophenol			0.200		25.0
1,1'-Biphenyl					
2-Chloronaphthalene			0.800		25.0
2-Nitroaniline					
Dimethylphthalate					
2,6-Dinitrotoluene			0.200		25.0
Acenaphthylene			0.900		25.0
3-Nitroaniline					
Acenaphthene			0.900		25.0
2,4-Dinitrophenol					
4-Nitrophenol					
Dibenzofuran			0.800		25.0

All other compounds must meet a minimum RRF of 0.010.

7LCE
LOW CONCENTRATION WATER SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

EPA Sample No.(SSTD020##): _____ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(MM)

COMPOUND	$\overline{RRF}$	RRF20	MIN RRF	%D	MAX %D
2,4-Dinitrotoluene			0.200		30.0
Diethylphthalate					
Fluorene			0.900		25.0
4-Chlorophenyl-phenylether			0.400		25.0
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine (1)					
1,2,4,5 Tetrachlorobenzene					
4-Bromophenyl-phenylether			0.100		25.0
Hexachlorobenzene			0.100		25.0
Atrazine					
Pentachlorophenol			0.050		25.0
Phenanthrene			0.700		25.0
Anthracene			0.700		25.0
Di-n-butylphthalate					
Fluoranthene			0.600		25.0
Pyrene			0.600		25.0
Butylbenzylphthalate					
3,3'-Dichlorobenzidine					
Benzo(a)anthracene			0.800		25.0
Chrysene			0.700		25.0
bis(2-Ethylhexyl)phthalate					
Di-n-octylphthalate					
Benzo(b)fluoranthene			0.700		25.0
Benzo(k)fluoranthene			0.700		25.0
Benzo(a)pyrene			0.700		25.0
Indeno(1,2,3-cd)pyrene			0.500		25.0
Dibenzo(a,h)anthracene			0.400		25.0
Benzo(g,h,i)perylene			0.500		25.0

(1) Cannot be separated from Diphenylamine
All other compounds must meet a minimum RRF of 0.010.

.7LCF

LOW CONCENTRATION WATER SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date: _____ Time: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

EPA Sample No.(SSTD020##): _____ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(MM)

COMPOUND	$\overline{RRF}$	RRF20	MIN RRF	%D	MAX %D
Phenol-d5					
bis-(2-Chloroethyl)ether-d8					
2-Chlorophenol-d4					
4-Methylphenol-d8					
Nitrobenzene-d5					
2-Nitrophenol-d4					
2,4-Dichlorophenol-d3					
4-Chloroaniline-d4					
Dimethylphthalate-d6					
Acenaphthylene-d8					
4-Nitrophenol-d4					
Fluorene-d10					
4,6-Dinitro-methylphenol-d2					
Anthracene-d10					
Pyrene-d10					
Benzo(a)pyrene-d12					

7LCG

LOW CONCENTRATION WATER PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: ____(MM) Init. Calib. Date(s): _____ _____

EPA Sample No. (PIBLK##): _____ Date Analyzed: _____

Lab Sample ID (PIBLK): _____ Time Analyzed: _____

EPA Sample No. (PEM##): _____ Date Analyzed: _____

Lab Sample ID (PEM): _____ Time Analyzed: _____

PEM COMPOUND	RT	RT WINDOW FROM	RT WINDOW TO	CALC AMOUNT (NG)	NOM AMOUNT (NG)	%D
alpha-BHC						
beta-BHC						
gamma-BHC (Lindane)						
Endrin						
4,4'-DDT						
Methoxychlor						

4,4'-DDT % Breakdown (1): _____ Endrin % breakdown (1): _____

Combined % Breakdown (1): _____

7LCH

LOW CONCENTRATION WATER PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (MM) Init. Calib. Date(s): _____ _____

EPA Sample No. (PIBLK##): _____ Date Analyzed: _____

Lab Sample ID (PIBLK): _____ Time Analyzed: _____

EPA Sample No. (INDAM##): _____ Date Analyzed: _____

Lab Sample ID (INDA): _____ Time Analyzed: _____

INDIVIDUAL MIX A COMPOUND	RT	RT WINDOW		CALC AMOUNT (NG)	NOM AMOUNT (NG)	%D
		FROM	TO			
alpha-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Endosulfan I						
Dieldrin						
Endrin						
4,4'-DDD						
4,4'-DDT						
Methoxychlor						
Tetrachloro-m-xylene						
Decachlorobiphenyl						

EPA Sample No. (INDBM##): _____ Date Analyzed: _____

Lab Sample ID (INDB): _____ Time Analyzed: _____

INDIVIDUAL MIX B COMPOUND	RT	RT WINDOW		CALC AMOUNT (NG)	NOM AMOUNT (NG)	%D
		FROM	TO			
beta-BHC						
delta-BHC						
Aldrin						
Heptachlor epoxide						
4,4'-DDE						
Endosulfan II						
Endosulfan sulfate						
Endrin ketone						
Endrin aldehyde						
alpha-Chlordane						
gamma-Chlordane						
Tetrachloro-m-xylene						
Decachlorobiphenyl						

8LCA

LOW CONCENTRATION WATER VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

EPA Sample No.(VSTD005##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____

GC Column: _____ ID: _____(MM) Length:_____(M)

		IS1 (CBZ) AREA	#	RT	#	IS2 (DFB) AREA	#	RT	#	IS3 (DCB) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS1 (CBZ) = Chlorobenzene-d5
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (DCB) = 1,4-Dichlorobenzene-d4

AREA UPPER LIMIT = +40% of internal standard area
AREA LOWER LIMIT = -40% of internal standard area
RT UPPER LIMIT = +0.33 minutes of internal standard RT
RT LOWER LIMIT = -0.33 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.
* Values outside of QC limits

8LCB

LOW CONCENTRATION WATER SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

EPA Sample No.(SSTD020##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____ GC Column: _____ ID: _____ (MM)

		IS1 (DCB) AREA	#	RT	#	IS2 (NPT) AREA	#	RT	#	IS3 (ANT) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d10

AREA UPPER LIMIT = +100% of internal standard area
AREA LOWER LIMIT = -50% of internal standard area
RT UPPER LIMIT = +0.33 minutes of internal standard RT
RT LOWER LIMIT = -0.33 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.
* Values outside of QC limits

Page __ of __

8LCC

LOW CONCENTRATION WATER SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

EPA Sample No.(SSTD020##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____ GC Column: _____ ID: _____(MM)

		IS4 (PHN) AREA	#	RT	#	IS5 (CRY) AREA	#	RT	#	IS6 (PRY) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS4 (PHN) = Phenanthrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (PRY) = Perylene-d12

AREA UPPER LIMIT = +100% of internal standard area
AREA LOWER LIMIT = -50% of internal standard area
RT UPPER LIMIT = +0.33 minutes of internal standard RT
RT LOWER LIMIT = -0.33 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.
* Values outside of QC limits

Page __ of __

8LCD

LOW CONCENTRATION WATER PESTICIDE ANALYTICAL SEQUENCE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____(MM) Init. Calib. Date(s): _____ _____

Instrument ID: _____

THE ANALYTICAL SEQUENCE OF SAMPLES, BLANKS, AND STANDARDS IS GIVEN BELOW:

	MEAN SURROGATE RT FROM INITIAL CALIBRATION TCX:_____ DCB:_____					
	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED	TCX RT #	DCB RT #
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						

QC LIMITS

TCX = Tetrachloro-m-xylene (±0.05 MINUTES)

DCB = Decachlorobiphenyl (±0.10 MINUTES)

Column used to flag retention time values with an asterisk.

* Values outside of QC limits.

Page __ of __

9LCA

LOW CONCENTRATION WATER PESTICIDE FLORISIL CARTRIDGE CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Florisil Cartridge Lot Number: _____ Date of Analysis: _____

GC Column (1): _____ ID:_____(MM) GC Column(2): _____ ID: ____(MM)

COMPOUND	SPIKE ADDED (NG)	SPIKE RECOVERED (NG)	% REC #	QC LIMITS
alpha-BHC				80-120
gamma-BHC (Lindane)				80-120
Heptachlor				80-120
Endosulfan I				80-120
Dieldrin				80-120
Endrin				80-120
4,4'-DDD				80-120
4,4'-DDT				80-120
Methoxychlor				80-120
Tetrachloro-m-xylene				80-120
Decachlorobiphenyl				80-120
2,4,5-Trichlorophenol				<5

Column to be used to flag recovery with an asterisk.
* Values outside of QC limits.

THIS CARTRIDGE LOT APPLIES TO THE FOLLOWING SAMPLES, LABORATORY CONTROL SAMPLES, AND BLANKS:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				

10LCA

LOW CONCENTRATION WATER PESTICIDE IDENTIFICATION
SUMMARY FOR SINGLE COMPONENT ANALYTES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date(s) Analyzed: _____ _____

Instrument ID (1): _____ Instrument ID (2): _____

GC Column:(1): _____ ID:_____(MM) GC Column:(2): _____ ID: _____(MM)

ANALYTE	COL	RT	RT WINDOW FROM	RT WINDOW TO	CONCENTRATION	%D
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					

Page __ of __

10LCB

LOW CONCENTRATION WATER PESTICIDE IDENTIFICATION
SUMMARY FOR MULTICOMPONENT ANALYTES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Lab Sample ID: _____ Date(s) Analyzed: _____ _____

Instrument ID (1): _____ Instrument ID (2): _____

GC Column:(1): _____ ID: _____(MM) GC Column:(2): _____ ID: _____(MM)

ANALYTE	PEAK	RT	RT WINDOW FROM	RT WINDOW TO	CONCENTRATION	MEAN CONCENTRATION	%D
_____	1						
	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						
	1						
	2						
_____	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						
	1						
	2						
_____	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						

At least 3 peaks for each column are required for identification of multicomponent analytes.

Page __ of __

SAMPLE LOG-IN SHEET

Lab Name				Page __ of __
Received By (Print Name)				Log-in Date
Received By (Signature)				
Case Number	Sample Delivery Group No.			Client Number

Remarks:		EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____ _____				
3. Chain of Custody Records	Present/Absent*				
4. Traffic Reports or Packing Lists	Present/Absent*				
5. Airbill	Airbill/Sticker Present/Absent*				
6. Airbill No.	_____ _____				
7. Sample Tags	Present/Absent*				
Sample Tag Nos.	Listed/Not Listed on Chain-of-Custody				
8. Sample Condition	Intact/Broken*/ Leaking				
9. Cooler Temperature Indicator Bottle	Present/Absent*				
10. Cooler Temperature	_____				
11. Does information on custody records, traffic reports, and sample tags agree?	Yes/No*				
12. Date Received at Lab	_____				
13. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

LOW CONCENTRATION WATER ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ CLIENT NO. _____

CONTRACT NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible.

	PAGE NOs		CHECK	
	FROM	TO	LAB	EPA
1. **Inventory Sheet** (Form DC-2) (Do Not Number)				
2. **SDG Case Narrative**				
3. **SDG Cover Sheet/Traffic Report**				
4. **Volatiles Data**				
a. QC Summary				
Deuterated Monitoring Compound Recovery (Form II LCV)				
Matrix Spike/Matrix Spike Duplicate Recovery (Form III LCV) (if Region requests)				
Method Blank Summary (Form IV LCV)				
GC/MS Instrument Performance Check (Form V LCV)				
Internal Standard Area and RT Summary (Form VIII LCV)				
b. Sample Data				
TCL Results - (Form I LCV-1, LCV-2)				
Tentatively Identified Compounds (Form I LCV-TIC)				
Reconstructed Total Ion Chromatograms (RIC) for each sample				
For each sample:				
Raw Spectra and background-subtracted mass spectra of target compounds identified				
Quantitation reports				
Mass Spectra of all reported TICs with three best library matches				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI LCV-1, LCV-2, LCV-3)				
RICs and Quantitation Reports for all Standards				
Continuing Calibration Data (Form VII LCV-1, LCV-2, LCV-3)				
RICs and Quantitation Reports for all Standards				
d. Raw QC Data				
BFB				
Blank Data				
Matrix Spike/Matrix Spike Duplicate Data (if Region requests)				

LOW CONCENTRATION WATER ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Con't)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ CLIENT NO. _____

	PAGE NOs FROM	PAGE NOs TO	CHECK LAB	CHECK EPA
5. Semivolatiles Data				
a. QC Summary				
Deuterated Monitoring Compound (Form II LCSV)	____	____	____	____
MS/MSD Summary (Form III LCSV) (if Region requests)	____	____	____	____
Method Blank Summary (Form IV LCSV)	____	____	____	____
GC/MS Instrument Performance Check (Form V LCSV)	____	____	____	____
Internal Standard Area and RT Summary (Form VIII LCSV)	____	____	____	____
b. Sample Data	____	____		
TCL Results - (Form I LCSV-1, LCSV-2)			____	____
Tentatively Identified Compounds (Form I LCSV-TIC)			____	____
Reconstructed Total Ion Chromatograms (RIC) for each sample			____	____
For each sample:				
Raw Spectra and background-subtracted mass spectra of target compounds			____	____
Quantitation reports			____	____
Mass Spectra of TICs with three best library matches			____	____
c. Standards Data (All Instruments)	____	____		
Initial Calibration Data (Form VI LCSV-1, LCSV-2, LCSV-3)			____	____
RICs and Quantitation Reports for all Standards			____	____
Continuing Calibration Data (Form VII LCSV-1, LCSV-2, LCSV-3)			____	____
RICs and Quantitation Reports for all Standards			____	____
d. Raw QC Data				
DFTPP	____	____	____	____
Blank Data	____	____	____	____
Matrix Spike/Matrix Spike Duplicate Data (if Region requests)	____	____	____	____
6. Pesticides Data				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II LCP)	____	____	____	____
MS/MSD Duplicate Summary (Form III LCP-1) (if Region requests)	____	____	____	____
Laboratory Control Sample Recovery (Form III LCP-2)				
Method Blank Summary (Form IV LCP)	____	____	____	____
b. Sample Data	____	____		
TCL Results - Organic Analysis Data Sheet (Form I LCP)			____	____
Chromatograms (Primary Column)			____	____
Chromatograms from second GC column confirmation			____	____
GC Integration report or data system printout			____	____
Manual work sheets			____	____

LOW CONCENTRATION WATER ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Con't)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ CLIENT NO. _____

	PAGE NOs FROM	PAGE NOs TO	CHECK LAB	CHECK EPA
6. **Pesticides Data** (Con't)				
c. Standards Data	____	____		
Initial Calibration of Single Component Analytes (Form VI LCP-1 and LCP-2)			____	____
Initial Calibration of Multicomponent Analytes (Form VI LCP-3)			____	____
Analyte Resolution Summary (Form VI LCP-4)			____	____
Performance Evaluation Mixture (Form VI LCP-5)			____	____
Individual Standard Mixture A (Form VI LCP-6)			____	____
Individual Standard Mixture B (Form VI LCP-7)			____	____
Calibration Verification Summary (Form VII LCP-1)			____	____
Calibration Verification Summary (Form VII LCP-2)			____	____
Analytical Sequence (Form VIII LCP)			____	____
Florisil Cartridge Check (Form IX LCP)			____	____
Pesticide Identification Summary for Single Component Analytes (Form X LCP-1)			____	____
Pesticide Identification Summary for Multicomponent Analytes (Form X LCP-2)			____	____
Chromatograms and data system printouts				
A printout of retention times and corresponding peak areas or peak heights			____	____
d. Raw QC Data				
Blank Data	____	____	____	____
Matrix Spike/Matrix Spike Duplicate Data (if Region requests)	____	____	____	____
Laboratory Control Sample Data	____	____	____	____
e. Raw Florisil Data	____	____	____	____
7. **Miscellaneous Data**				
Original preparation and analysis forms or copies of preparation and analysis logbook pages	____	____	____	____
Internal sample and sample extract transfer chain-of-custody records	____	____	____	____
Screening records	____	____	____	____
All instrument output, including strip charts from screening activities (describe or list)				
_____	____	____	____	____
_____	____	____	____	____

LOW CONCENTRATION WATER ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Con't)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ CLIENT NO. _____

	PAGE NOs FROM	TO	CHECK LAB	EPA
8. **EPA Shipping/Receiving Documents**				
Airbills (No. of shipments _____)	____	____	____	____
Chain-of-Custody Records	____	____	____	____
Sample Tags			____	____
Sample Log-in Sheet (Lab & DC1)	____	____	____	____
Miscellaneous Shipping/Receiving Records (describe or list)				
_____	____	____	____	____
_____	____	____	____	____
9. **Internal Lab Sample Transfer Records and Tracking Sheets**				
(describe or list)				
_____	____	____	____	____
_____	____	____	____	____
10. **Other Records**				
(describe or list)	____	____	____	____
Telephone Communication Log				
_____	____	____	____	____
_____	____	____	____	____

11. **Comments:** _____

_____

_____

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

Verified by: (CLP Lab) _____ (Signature) _____ (Printed Name/Title)

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

EXHIBIT C

TARGET COMPOUND LIST AND
CONTRACT REQUIRED QUANTITATION LIMITS

NOTE: Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

The CRQL values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D.

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Exhibit C - Target Compound List and Contract Required Quantitation Limits

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1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Volatiles	CAS Number	Quantitation Limits Water µg/L
1.	Dichlorodifluoromethane	75-71-8	0.50
2.	Chloromethane	74-87-3	0.50
3.	Vinyl Chloride	75-01-4	0.50
4.	Bromomethane	74-83-9	0.50
5.	Chloroethane	75-00-3	0.50
6.	Trichlorofluoromethane	75-69-4	0.50
7.	1,1-Dichloroethene	75-35-4	0.50
8.	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.50
9.	Acetone	67-64-1	5.0
10.	Carbon Disulfide	75-15-0	0.50
11.	Methyl Acetate	79-20-9	0.50
12.	Methylene Chloride	75-09-2	0.50
13.	trans-1,2-Dichloroethene	156-60-5	0.50
14.	Methyl tert-Butyl Ether	1634-04-4	0.50
15.	1,1-Dichloroethane	75-34-3	0.50
16.	cis-1,2-Dichloroethene	156-59-2	0.50
17.	2-Butanone	78-93-3	5.0
18.	Bromochloromethane	74-97-5	0.50
19.	Chloroform	67-66-3	0.50
20.	1,1,1-Trichloroethane	71-55-6	0.50
21.	Cyclohexane	110-82-7	0.50
22.	Carbon Tetrachloride	56-23-5	0.50
23.	Benzene	71-43-2	0.50
24.	1,2-Dichloroethane	107-06-2	0.50
25.	Trichloroethene	79-01-6	0.50
26.	Methylcyclohexane	108-87-2	0.50
27.	1,2-Dichloropropane	78-87-5	0.50
28.	Bromodichloromethane	75-27-4	0.50
29.	cis-1,3-Dichloropropene	10061-01-5	0.50
30.	4-Methyl-2-pentanone	108-10-1	5.0
31.	Toluene	108-88-3	0.50
32.	trans-1,3-Dichloropropene	10061-02-6	0.50
33.	1,1,2-Trichloroethane	79-00-5	0.50
34.	Tetrachloroethene	127-18-4	0.50
35.	2-Hexanone	591-78-6	5.0
36.	Dibromochloromethane	124-48-1	0.50
37.	1,2-Dibromoethane	106-93-4	0.50
38.	Chlorobenzene	108-90-7	0.50

1.0 VOLATILE TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (Con't)

	Volatiles	CAS Number	Quantitation Limits Water µg/L
39.	Ethylbenzene	100-41-4	0.50
40.	Xylenes (total)	1330-20-7	0.50
41.	Styrene	100-42-5	0.50
42.	Bromoform	75-25-2	0.50
43.	Isopropylbenzene	98-82-8	0.50
44.	1,1,2,2-Tetrachloroethane	79-34-5	0.50
45.	1,3-Dichlorobenzene	541-73-1	0.50
46.	1,4-Dichlorobenzene	106-46-7	0.50
47.	1,2-Dichlorobenzene	95-50-1	0.50
48.	1,2-Dibromo-3-chloropropane	96-12-8	0.50
49.	1,2,4-Trichlorobenzene	120-82-1	0.50
50.	1,2,3-Trichlorobenzene	87-61-6	0.50

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Semivolatiles	CAS Number	Quantitation Limits Water • g/L
51.	Benzaldehyde	100-52-7	5.0
52.	Phenol	108-95-2	5.0
53.	bis-(2-Chloroethyl)ether	111-44-4	5.0
54.	2-Chlorophenol	95-57-8	5.0
55.	2-Methylphenol	95-48-7	5.0
56.	2,2'-oxybis(1-Chloropropane)[1]	108-60-1	5.0
57.	Acetophenone	98-86-2	5.0
58.	4-Methylphenol	106-44-5	5.0
59.	N-Nitroso-di-n-propylamine	621-64-7	5.0
60.	Hexachloroethane	67-72-1	5.0
61.	Nitrobenzene	98-95-3	5.0
62.	Isophorone	78-59-1	5.0
63.	2-Nitrophenol	88-75-5	5.0
64.	2,4-Dimethylphenol	105-67-9	5.0
65.	bis(2-Chloroethoxy)methane	111-91-1	5.0
66.	2,4-Dichlorophenol	120-83-2	5.0
67.	Naphthalene	91-20-3	5.0
68.	4-Chloroaniline	106-47-8	5.0
69.	Hexachlorobutadiene	87-68-3	5.0
70.	Caprolactam	105-60-2	5.0
71.	4-Chloro-3-methylphenol	59-50-7	5.0
72.	2-Methylnaphthalene	91-57-6	5.0
73.	Hexachlorocyclopentadiene	77-47-4	5.0
74.	2,4,6-Trichlorophenol	88-06-2	5.0
75.	2,4,5-Trichlorophenol	95-95-4	20
76.	1,1'-Biphenyl	92-52-4	5.0
77.	2-Chloronaphthalene	91-58-7	5.0
78.	2-Nitroaniline	88-74-4	20
79.	Dimethylphthalate	131-11-3	5.0
80.	2,6-Dinitrotoluene	606-20-2	5.0
81.	Acenaphthylene	208-96-8	5.0
82.	3-Nitroaniline	99-09-2	20
83.	Acenaphthene	83-32-9	5.0
84.	2,4-Dinitrophenol	51-28-5	20

[1]Previously known by the name bis(2-Chloroisopropyl)ether.

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (Con't)

	Semivolatiles	CAS Number	Quantitation Limits Water • g/L
85.	4-Nitrophenol	100-02-7	20
86.	Dibenzofuran	132-64-9	5.0
87.	2,4-Dinitrotoluene	121-14-2	5.0
88.	Diethylphthalate	84-66-2	5.0
89.	Fluorene	86-73-7	5.0
90.	4-Chlorophenyl-phenylether	7005-72-3	5.0
91.	4-Nitroaniline	100-01-6	20
92.	4,6-Dinitro-2-methylphenol	534-52-1	20
93.	N-Nitrosodiphenylamine	86-30-6	5.0
94.	1,2,4,5 Tetrachlorobenzene	95-94-3	5.0
95.	4-Bromophenyl-phenylether	101-55-3	5.0
96.	Hexachlorobenzene	118-74-1	5.0
97.	Atrazine	1912-24-9	5.0
98.	Pentachlorophenol	87-86-5	5.0
99.	Phenanthrene	85-01-8	5.0
100.	Anthracene	120-12-7	5.0
101.	Di-n-butylphthalate	84-74-2	5.0
102.	Fluoranthene	206-44-0	5.0
103.	Pyrene	129-00-0	5.0
104.	Butylbenzylphthalate	85-68-7	5.0
105.	3,3'-Dichlorobenzidine	91-94-1	5.0
106.	Benzo(a)anthracene	56-55-3	5.0
107.	Chrysene	218-01-9	5.0
108.	bis(2-Ethylhexyl)phthalate	117-81-7	5.0
109.	Di-n-octylphthalate	117-84-0	5.0
110.	Benzo(b)fluoranthene	205-99-2	5.0
111.	Benzo(k)fluoranthene	207-08-9	5.0
112.	Benzo(a)pyrene	50-32-8	5.0
113.	Indeno(1,2,3-cd)pyrene	193-39-5	5.0
114.	Dibenzo(a,h)anthracene	53-70-3	5.0
115.	Benzo(g,h,i)perylene	191-24-2	5.0

3.0 PESTICIDES/AROCLORS TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Pesticides/Aroclors	CAS Number	Quantitation Limits Water • g/L
116.	alpha-BHC	319-84-6	0.01
117.	beta-BHC	319-85-7	0.01
118.	delta-BHC	319-86-8	0.01
119.	gamma-BHC (Lindane)	58-89-9	0.01
120.	Heptachlor	76-44-8	0.01
121.	Aldrin	309-00-2	0.01
122.	Heptachlor epoxide[2]	1024-57-3	0.01
123.	Endosulfan I	959-98-8	0.01
124.	Dieldrin	60-57-1	0.02
125.	4,4'-DDE	72-55-9	0.02
126.	Endrin	72-20-8	0.02
127.	Endosulfan II	33213-65-9	0.02
128.	4,4'-DDD	72-54-8	0.02
129.	Endosulfan sulfate	1031-07-8	0.02
130.	4,4'-DDT	50-29-3	0.02
131.	Methoxychlor	72-43-5	0.10
132.	Endrin ketone	53494-70-5	0.02
133.	Endrin aldehyde	7421-93-4	0.02
134.	alpha-Chlordane	5103-71-9	0.01
135.	gamma-Chlordane	5103-74-2	0.01
136.	Toxaphene	8001-35-2	1.0
137.	Aroclor-1016	12674-11-2	0.20
138.	Aroclor-1221	11104-28-2	0.40
139.	Aroclor-1232	11141-16-5	0.20
140.	Aroclor-1242	53469-21-9	0.20
141.	Aroclor-1248	12672-29-6	0.20
142.	Aroclor-1254	11097-69-1	0.20
143.	Aroclor-1260	11096-82-5	0.20

[2]Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is reported on the data reporting forms (Exhibit B).

EXHIBIT D

METHOD FOR THE ANALYSIS OF LOW CONCENTRATION WATER FOR VOLATILE (PURGEABLE) ORGANIC COMPOUNDS

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Exhibit D -- Analytical Methods for Volatiles

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water samples containing low concentrations of the volatile compounds listed in the Target Compound List (TCL) in Exhibit C. The majority of the samples are expected to be obtained from drinking water and well/ground water type sources around Superfund sites. The method is based on EPA Method 524.2 and the volatile method contained in the Contract Laboratory Program (CLP) Statement of Work (SOW), "Organic Analysis, Multi-Media, Multi-Concentration". The sample preparation and analysis procedures included in this method are based on purge and trap Gas Chromatograph/Mass Spectrometer (GC/MS) techniques.[1]

1.2 Dichlorodifluoromethane, Trichlorofluoromethane, 1,1,2-Trichloro-1,2,2-trifluoroethane, Methyl Acetate, 1,2,3-Trichlorobenzene, Methyl tert-butyl ether, Cyclohexane, Methylcyclohexane, and Isopropylbenzene have been added to the TCL.

1.3 Problems that have been associated with the following compounds analyzed by this method include:

- Chloromethane, vinyl chloride, bromomethane, and chloroethane may display peak broadening if the compounds are not delivered to the GC column in a tight band.
- Acetone, hexanone, 2-butanone, and 4-methyl-2-pentanone have poor purge efficiencies.
- 1,1,1-Trichloroethane and all of the dichloroethanes may dehydrohalogenate during storage or analysis.
- Tetrachloroethane and 1,1-dichloroethane may be degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.
- Chloromethane may be lost if the purge flow is too fast.
- Bromoform is one of the compounds most likely to be adversely affected by cold spots and/or active sites in the transfer lines. Response of its quantitation ion (m/z 173) is directly affected by the tuning of 4-Bromofluorobenzene (BFB) at ions m/z 174/176. Increasing the m/z 174/176 ratio within the specified Quality Control (QC) limits may improve bromoform response.
- Due to the lower quantitation limits required by this method, extra caution must be exercised when identifying compounds.

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[1]This analytical method includes the use of Deuterated Monitoring Compounds (DMC) for precision and accuracy assessment.

2.0 SUMMARY OF METHOD

2.1 An inert gas is bubbled through a 25 milliliter (mL) sample contained in a specially designed purging chamber at ambient temperature causing the purgeables to be transferred from the water/aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a Gas Chromatograph (GC) wide-bore capillary column. The GC is temperature programmed to separate the purgeables, which are then detected with a Mass Spectrometer (MS).

2.2 Deuterated Monitoring Compounds (DMCs) and internal standards are added to all samples and blanks. The target compounds and DMCs are identified in the samples and blanks by analyzing standards that contain all target compounds, DMCs, and internal standards under the same conditions and comparing resultant mass spectra and GC retention times. A Relative Response Factor (RRF) is established for each target compound and DMC during the initial and continuing calibrations. The mass spectra response from the Extracted Ion Current Profile (EICP) for the primary quantitation ion produced by that compound is compared to the mass spectra response for the primary quantitation ion produced by the associated internal standard compound. Each identified target compound and DMC is quantitated by comparing the instrument response for the compound in the sample or blank with the instrument response of the associated internal standard, while taking into account the RRF from the most recent mid-point calibration, the sample volume, and any sample dilutions.

2.3 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later) or equivalent mass spectral library. Non-target compounds are quantitated by comparing the mass spectra response from the total ion chromatograms to the mass spectra response of the nearest internal standard compound. A RRF of 1 is assumed.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method interference may be caused by impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory method and instrument blanks as described in Section 12. The use of non-polytetrafluoroethylene (PTFE) tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples must be stored separately from other laboratory samples and standards and must be analyzed in a room whose atmosphere is demonstrated to be free of all potential contaminants which will interfere with the analysis.

4.3 Contamination by carryover can occur whenever high level and low level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank, or the next sample must be closely monitored to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution between analyses, rinse it with distilled water, and then dry it in an oven at 105°C. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4.4 The laboratory where volatile analysis is performed should be completely free of solvents.

5.0 SAFETY

5.1 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 OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

5.2 The following analytes covered by this method have been tentatively classified as *known or suspected, human or mammalian carcinogens:* benzene; carbon tetrachloride; chloroform; and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA-approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 EQUIPMENT AND SUPPLIES

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

6.1 Glassware

6.1.1 Syringes - 25 milliliters (mL), gas-tight with shut-off valve. Micro syringes - 10 microliters (• L) and larger, 0.006 inch (0.15 mm) ID needle.

6.1.2 Syringe Valve - two-way, with Luer ends (three each), if applicable to the purging device.

6.1.3 Pasteur Pipets - disposable.

6.1.4 Vials and Caps - assorted sizes.

6.1.5 Volumetric Flasks, class A with ground-glass stoppers.

6.1.6 Bottles - 15 mL, screw-cap, with PTFE cap liner.

6.2 pH Paper - wide range

6.3 Balances

Balances must be analytical and capable of accurately weighing ±0.0001 g. The balance must be calibrated with class S weights or known reference weights once per each 12-hour work shift. The balance must be calibrated with class S weights at a minimum of once per month. The balance must also be annually checked by a certified technician.

6.4 Purge and Trap Device

The purge and trap device consists of three separate pieces of equipment: the sample purge chamber, trap, and the desorber. Several complete devices are now commercially available.

6.4.1 The sample purge chamber must be designed to accept 25 mL samples with a water column at least 10 centimeters (cm) deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 millimeters (mm) at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

6.4.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch (2.667 mm). The trap must be packed to contain the following minimum lengths of absorbents: (starting from inlet) 0.5 cm silanized glass wool, 1 cm methyl silicone, 8 cm of 2,6-

diphenylene oxide polymer (Tenax-GC, 60/80 mesh), 8 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15 or equivalent), 7 cm of coconut charcoal (prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen), and 0.5 cm silanized glass wool. A description of the trap used for analysis shall be provided in the SDG Narrative.

6.4.3 The desorber must be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated higher than 180°C and the remaining sections should not exceed 220°C during bakeout mode.

6.4.4 Trap Packing

6.4.4.1 2,6-Diphenylene oxide polymer, 60/80 mesh chromatographic grade (Tenax GC or equivalent).

6.4.4.2 Methyl silicone packing, 3.0 percent OV-1 on Chromosorb W, 60/80 mesh (or equivalent).

6.4.4.3 Silica gel, 35/60 mesh, Davison, grade 15 (or equivalent).

6.4.4.4 Coconut charcoal (prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen).

6.4.4.5 Alternate sorbent traps may be used if:

- The trap packing materials do not introduce contaminants which interfere with identification and quantitation of the compounds listed in Exhibit C (Volatiles);
- The analytical results generated using the trap meet the initial and continuing calibration technical acceptance criteria listed in the SOW and the Contract Required Quantitation Limits (CRQLs) listed in Exhibit C (Volatiles); or
- The trap can accept up to 1000 nanograms (ng) of each compound listed in Exhibit C (Volatiles) without becoming overloaded.

6.4.4.5.1 The alternate trap must be designed to optimize performance. Follow the manufacturer's instructions for the use of its product. Before use of any trap other than the one specified in Section 6.4.2, the Contractor must first meet the criteria listed in Section 6.4.4.5. Once this has been demonstrated, the Contractor must document its use in each SDG Narrative by specifying the trap composition (packing material/brand name, amount of packing material). Other sorbent traps include, but are not limited to, Tenax/Silica Gel/Carbon Trap from EPA Method 524.2, Tenax - GC/Graphpac-D Trap (Alltech) or equivalent, and Vocarb 4000 Trap (Supelco) or equivalent.

6.4.4.5.2 The Contractor must maintain documentation that the alternate trap meets the criteria listed in Section 6.4.4.5. The minimum documentation requirements are as follows:

6.4.4.5.2.1 Manufacturer-provided information concerning the performance characteristics of the trap.

6.4.4.5.2.2 Reconstructed ion chromatograms and data system reports generated on the Contractor's GC/MS used for CLP analyses:

- From instrument blank analyses which demonstrate that there are no contaminants which interfere with the volatile analysis when using the alternate trap; and
- From initial and continuing calibration standards analyzed using the trap specified in Section 6.4.4.

6.4.4.5.2.3 Based on Contractor-generated data described above, the Contractor must complete a written comparison/review, which has been signed by the Laboratory Manager, certifying that:

- The alternate trap performance meets the technical acceptance criteria listed in Sections 9.3.5 and 9.4.5;
- The low point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;
- The high point initial calibration standard analysis was not overloaded; and
- The alternate trap materials do not introduce contaminants which interfere with the identification and/or quantitation of the compounds listed in Exhibit C (Volatiles).

6.4.4.5.2.4 The documentation must be made available to USEPA during on-site laboratory evaluations or sent to USEPA upon request of the USEPA CLP Project Officer (CLP PO) or the Organic Program Manager at the Analytical Operations/Data Quality Center (AOC).

6.4.5 The purge and trap apparatus may be assembled as a separate unit or be an integral unit coupled with a Gas Chromatograph (GC).

6.5 Gas Chromatograph/Mass Spectrometer (GC/MS) System

6.5.1 Gas Chromatograph - The 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 purge and trap system as specified in Section 6.4 and have 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, are not to be used. The column oven must be cooled to 10°C if adequate separation of gaseous compounds is not achieved (Section 9.1.2.3); therefore, a subambient oven controller is required.

6.5.2 Gas Chromatography Columns

A description of the column used for analysis shall be provided in the SDG Narrative.

6.5.2.1 Minimum length 30 m x 0.53 mm ID VOCOL (Supelco) or equivalent fused silica widebore capillary column with 3 micrometers (µm) film thickness.

6.5.2.2 Minimum length 30 m x 0.53 mm ID DB-624 (J & W Scientific) or equivalent fused silica widebore capillary column with 3 µm film thickness.

6.5.2.3 Minimum length 30 m x 0.53 mm ID AT-624 (Alltech) or equivalent fused silica widebore capillary column with 3 µm film thickness.

6.5.2.4 Minimum length 30 m x 0.53 mm ID HP-624 (Hewlett-Packard) or equivalent fused silica widebore capillary column with 3 µm film thickness.

6.5.2.5 Minimum length 30 m x 0.53 mm ID RTx-624 (Restek) or equivalent fused silica widebore capillary column with 3 µm film thickness.

6.5.2.6 Minimum length 30 m x 0.53 mm ID BPX-624 (SGE) or equivalent fused silica widebore capillary column with 3 µm film thickness.

6.5.2.7 Minimum length 30 m x 0.53 mm ID CP-Sil 13CB (Chrompack) or equivalent fused silica widebore capillary column with 3 µm film thickness.

6.5.3 A capillary column is considered equivalent if:

- The column does not introduce contaminants which interfere with the identification and quantitation of the compounds listed in Exhibit C (Volatiles);
- The analytical results generated using the column meet the initial and continuing calibration technical acceptance criteria listed in the SOW, and the CRQLs listed in Exhibit C (Volatiles);
- The column can accept up to 1000 ng of each compound listed in Exhibit C (Volatiles) without becoming overloaded; and
- The column provides equal or better resolution of the compounds listed in Exhibit C (Volatiles) than the columns listed in Section 6.5.2.

6.5.3.1 As applicable, follow the manufacturer's instructions for use of its product.

6.5.3.2 The Contractor must maintain documentation that the column met the criteria in Section 6.5.3. The minimum documentation is as follows:

6.5.3.2.1 Manufacturer provided information concerning the performance characteristics of the column.

6.5.3.2.2 Reconstructed ion chromatograms and data system reports generated on the GC/MS used for the CLP analyses:

- From instrument blanks which demonstrate that there are no contaminants which interfere with the volatile analysis when using the column; and
- From initial and continuing calibration standards analyzed using the alternate column.

6.5.3.5 Based on the Contractor-generated data described above, the Contractor shall complete a written comparison/review, signed by the Laboratory Manager, certifying that:

- The alternate column performance meets the technical acceptance criteria in Sections 9.3.5 and 9.4.5;
- The low point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;
- The high point initial calibration standard analysis was not overloaded; and
- The column does not introduce contaminants which interfere with the identification and/or quantitation of compounds listed in Exhibit C (Volatiles).

6.5.3.6 The documentation must be made available to USEPA during on-site laboratory evaluations or sent to USEPA upon request of the CLP PO or the Organic Program Manager at the AOC.

6.5.4 **PACKED COLUMNS CANNOT BE USED.**

6.5.5 Mass Spectrometer (MS) - The MS must be capable of scanning from 35 to 300 atomic mass unit (amu) every 2 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the 4-Bromo-fluorobenzene (BFB) GC/MS performance check technical acceptance criteria in Table D-1 when 50 ng of BFB are injected through the GC inlet.

NOTE: To ensure sufficient precision of mass spectral data, the MS scan rate must allow acquisition of at least five spectra while a sample compound elutes from the GC. The purge and trap 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 outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.5.6 GC/MS Interface - Any GC/MS interface may be used that gives acceptable calibration points at 25 ng or less per injection for each

of the purgeable target compounds and deuterated monitoring compounds and achieves all acceptable performance criteria. GC/MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.5.7 Data System - A computer 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 of 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). 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 comparing sample spectra against reference library spectra. The NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later) or equivalent 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.

6.5.8 Magnetic Tape Storage Device - Capable of recording data and must be suitable for long-term, off-line storage.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1.1 Reagent water - Reagent water is defined as water in which no purgeable target compound is observed at or above the Contract Required Quantitation Limit (CRQL) listed in Exhibit C for that compound and in which no non-target compound is observed at or above 2.0 micrograms per liter (µg/L).

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g (1 lb.) of activated carbon (Calgon Corp., Filtrasorb-300, or equivalent).

7.1.1.2 Reagent water may be generated using a water purification system (Millipore Super-Q or equivalent).

7.1.1.3 Reagent water may be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle, seal with a PTFE-lined septum, and cap.

7.1.2 Methanol - HPLC quality or equivalent -- Each lot of methanol used for analysis under this contract must be purged with nitrogen and must be demonstrated to be free of contaminants that interfere with the measurement of purgeable compounds listed in Exhibit C.

7.2 Standards

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. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

7.2.1 Stock Standard Solutions

Stock standard solutions may be purchased or may be prepared in methanol from pure standard materials.

7.2.1.1 Prepare stock standard solutions by placing about 9.8 milliliters (mL) of methanol into a 10.0 mL ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

7.2.1.2 Add the assayed reference material as described below.

7.2.1.2.1 If the compound is a liquid, use a 100 microliters (µL) syringe to immediately add two or more drops of assayed reference

material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

7.2.1.2.2 If the compound is a gas at room temperature, fill a 5 mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to 5 millimeters (mm) above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The gas will rapidly dissolve in the methanol. This may also be accomplished by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach PTFE tubing to the side-arm relief valve and direct a gentle stream of the reference standard into the methanol meniscus.

7.2.1.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. For non-gaseous compounds, calculate the concentration in µg/µL from the net gain in weight. When compound purity is assayed to be 97 percent or greater, the weight may be used without correction to calculate the concentration of the stock standard. If the compound purity is assayed to be less than 97 percent, the weight must be corrected when calculating the concentration of the stock solution. For gaseous compounds, calculate the concentration in µg/µL using the Ideal Gas Law, taking into account the temperature and pressure conditions within the laboratory.

7.2.1.4 Prepare fresh stock standards every two months for gases or for reactive compounds such as styrene. All other stock standards for non-gases/non-reactive purgeable compounds must be replaced after six months, or sooner if standard has degraded or evaporated.

7.2.2 Secondary Dilution Standards

7.2.2.1 Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. Secondary dilution standard solutions should be prepared at concentrations that can be easily diluted to prepare working standard solutions.

7.2.2.2 Prepare fresh secondary dilution standards for gases and for reactive compounds, such as styrene, every month or sooner if standard has degraded or evaporated. Secondary dilution standards for the other purgeable compounds must be replaced after six months, or sooner if standard has degraded or evaporated.

7.2.3 Working Standards

7.2.3.1 Instrument Performance Check Solution (4-Bromofluorobenzene)

Prepare a 25 nanograms per microliter (ng/µL) solution of 4-Bromofluorobenzene (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.

7.2.3.2 Calibration Standard Solution

Prepare the working calibration standard solution containing all of the purgeable target compounds in methanol (Exhibit C). The concentration of the non-ketone target compounds and the associated Deuterated Monitoring Compounds (DMCs) must be 2.5 µg/mL in the standard (i.e., final concentration). The concentration of the ketones (acetone, butanone, 2-hexanone, 4-methyl-2-pentanone) and their associated DMCs must be 12.5 µg/mL in the standard (i.e., final concentration). Prepare fresh working calibration standard solutions weekly, or sooner if solutions have degraded or evaporated.

7.2.3.3 Internal Standard Spiking Solution

Prepare an internal standard spiking solution containing 1,4-dichlorobenzene-d4, chlorobenzene-d5, and 1,4-difluorobenzene in methanol at the concentration of 12.5 µg/mL for each internal standard. Add 10 µL of this spiking solution into 25.0 mL of samples, blanks, requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), and calibration standards for a concentration of 5.0 µg/L. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

7.2.3.4 Deuterated Monitoring Compound (DMC) Spiking Solution

Prepare a DMC spiking solution in methanol containing the compounds listed below at the following concentrations:

Compound	Concentration µg/mL
Vinyl Chloride-d3	12.5
Chloroethane-d5	12.5
1,1-Dichloroethene-d2	12.5
2-Butanone-d5	12.5
Chloroform-d	12.5
1,2-Dichloroethane-d4	12.5
Benzene-d6	12.5
1,2-Dichloropropane-d6	12.5
Toluene-d8	12.5
trans-1,3-Dichloropropene-d4	12.5
2-Hexanone-d5	12.5
Bromoform-d	12.5
1,1,2,2-Tetrachloroethane-d2	12.5
1,2-Dichlorobenzene-d4	12.5

Add 10 µL of this spiking solution into 25 mL of sample, and blank for a concentration of 5.0 µg/L. The DMC spiking solution is added to the working calibration standards so it is not to be added again when aqueous calibration standards are prepared.

Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

7.2.3.5 Matrix Spiking Solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 12.5 µg/mL; 1,1-Dichloroethene, Trichloroethene, Chlorobenzene, Toluene, and Benzene. Prepare fresh spiking solution weekly, or sooner, if the solution has degraded or evaporated.

7.2.3.6 Aqueous Calibration Standard Solutions -- Initial and Continuing

7.2.3.6.1 Prepare five aqueous initial calibration standard solutions containing all of the purgeable target compounds and the DMCs at the following levels: all non-ketone target compounds and non-ketone DMCs at 0.50, 1.0, 5.0, 10, and 25 µg/L; all ketones and their associated DMCs at 5.0, 10, 25, 50 and 125 µg/L. It is required that all three xylene isomers (o-, p-, and m-xylene) be present in the calibration standards at concentrations of each isomer equal to that of the other target compounds (i.e., 0.50, 1.0, 5.0, 10, and 25 µg/L). The internal standards are added to each calibration standard according to the procedure in Section 9.3.3.4.

7.2.3.6.2 Aqueous calibration standards may be prepared in a volumetric flask or in the syringe used to inject the standard into the purging device.

7.2.3.6.2.1 Volumetric flask - Add an appropriate volume of working calibration standard solution to an aliquot of reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcohol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the head of the flask.

7.2.3.6.2.2 Syringe - Remove the plunger from a 25 mL syringe and close the syringe valve. Pour reagent water into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open the syringe valve, and vent any residual air. Adjust the water volume to 25.0 mL minus the amount of calibration standard to be added. Withdraw the plunger slightly and add an appropriate volume of working calibration standard through the valve bore of the syringe. Close the valve and invert three times.

7.2.3.6.3 The 5.0 µg/L (25 µg/L for ketones) aqueous calibration standard solution is the continuing calibration standard.

7.2.3.6.4 The methanol contained in each of the aqueous calibration standards must not exceed 1 percent by volume.

7.2.4 Ampulated Standard Extracts

Standard solutions purchased from a chemical supply house as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor which are immediately ampulated in glass vials may be retained for 2 years from preparation date. Upon breaking the glass seal, the expiration times listed in Sections 7.2.1 to 7.2.3 will apply. The Contractor is responsible for assuring that the integrity of the standards has not degraded (Section 7.2.5.5).

7.2.5 Storage of Standards

7.2.5.1 Store the stock standards in PTFE-sealed screw-cap bottles with zero headspace at -10°C to -20°C, and protect the standards from light. Once one of the bottles containing the stock standard solution has been opened, it may be used for no longer than one week.

7.2.5.2 Store secondary dilution standards in PTFE-sealed screw-cap bottles with minimal headspace at -10°C to -20°C. Protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to preparing the working calibration standards from them.

7.2.5.3 Aqueous standards may be stored up to 24 hours if held in PTFE-sealed screw-cap vials with zero headspace at 4°C (±2°C), and protect the standards from light. If not so stored, they must be discarded after one hour unless they are set up to be purged by an autosampler. When using an autosampler, the standards may be kept up to 12 hours in purge tubes connected via the autosampler to the purge and trap device.

7.2.5.4 Purgeable standards must be stored separately from other standards.

7.2.5.5 The Contractor is responsible for maintaining the integrity of standard solutions and verifying prior to use. This means that standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in the solution.

7.2.6 Temperature Records for Storage of Standards

7.2.6.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.6.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.6.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in glass containers having a total volume of at least 40 milliliters (mL) with a PTFE-lined septum and an open top screw-cap. Headspace should be avoided. The specific requirements for site sample collection are outlined by the Region.

8.1.2 The containers must be filled in such a manner that no air bubbles pass through the sample as the container is being filled. Seal the vial so that no air bubbles are entrapped in it.

8.1.3 Water samples are preserved to a pH of 2 at the time of collection.

8.1.4 All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until analysis.

8.2 Procedure for Sample Storage

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

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants and in a refrigerator used only for storage of volatile samples received under this contract.

8.2.3 All volatile samples in an SDG must be stored together in the same refrigerator.

8.2.4 Storage blanks shall be stored with samples until all samples within an SDG are analyzed.

8.2.5 Samples, sample extracts, and standards must be stored separately.

8.2.6 Volatile standards must be stored separately from semivolatile and pesticide/Aroclor standards.

8.3 Temperature Records for Sample Storage

8.3.1 The temperature of all sample storage refrigerators shall be recorded daily.

8.3.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

8.3.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators.

8.4 Contract Required Holding Times

Analysis of water samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR). As part of USEPA's Quality Assurance (QA) program, USEPA may provide Performance Evaluation (PE) Samples as standard extracts which the Contractor is required to prepare per the instructions provided by USEPA. PE samples must be prepared and analyzed concurrently with the samples in the SDG. The contract-required 10-day holding time does not apply to PE Samples received as standard extracts.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Conditions

9.1.1 Purge and Trap

9.1.1.1 The following are the recommended purge and trap analytical conditions. The conditions are recommended unless otherwise noted:

Purge Conditions

Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 ±0.1 minute
Purge Flow Rate:	25-40 mL/minute
Purge Temperature:	Ambient temperature (required)

Desorb Conditions

Desorb Temperature:	180°C
Desorb Flow Rate:	15 mL/minute
Desorb Time:	4.0 ±0.1 minute

Trap Reconditioning Conditions

Reconditioning Temperature:	180°C
Reconditioning Time:	7.0 ±0.1 minute (minimum). A longer time may be required to bake contamination or water from the system.

9.1.1.2 Before initial use, condition the trap overnight at 180°C by backflushing with at least 20 milliliters (mL)/minute flow of inert gas. Do not vent the trap effluent onto the analytical column. Prior to daily use, condition the trap by heating at 180°C for 10 minutes while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to the analysis of samples and blanks.

9.1.1.3 Optimize purge and trap conditions for sensitivity and to minimize cross-contamination between samples. Once optimized, the same

purge and trap conditions must be used for the analysis of all standards, samples, and blanks.

9.1.1.4 A moisture reduction/water management system may be used to improve the chromatographic performance by controlling moisture or water if:

- The system does not introduce contaminants which interfere with identification and quantitation of compounds listed in Exhibit C (Volatiles),
- The analytical results generated when using the moisture reduction/water management system meet the initial and continuing calibration technical acceptance criteria listed in the Statement of Work (SOW) and the Contract Required Quantitation Limits (CRQLs) listed in Exhibit C (Volatiles);
- All calibration standards, samples, and blanks are analyzed under the same conditions; and
- The Contractor performs acceptably on the Performance Evaluation Samples using this system.

9.1.2 Gas Chromatograph (GC)

9.1.2.1 The following are the recommended GC analytical conditions. The conditions are recommended unless otherwise noted:

Capillary Columns	
Carrier Gas:	Helium
Flow Rate:	15 mL/minute
Initial Temperature:	10°C
Initial Hold Time:	1.0 - 5.0 (±0.1) minutes
Ramp Rate:	6°C/minute
Final Temperature:	160°C
Final Hold Time:	Until three minutes after all compounds listed in Exhibit C (Volatiles) elute (required)

9.1.2.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, and blanks.

9.1.2.3 If the gaseous compounds chloromethane, bromomethane, vinyl chloride, and chloroethane fail to exhibit narrow, symmetrical peak shape, are not separated from the solvent front, or are not resolved greater than 90.0 percent from each other, then a subambient oven controller must be used, and the initial temperature must be less than or equal to 10°C.

9.1.3 Mass Spectrometer (MS)

The following are the required MS analytical conditions:

Electron Energy:	70 volts (nominal)
Mass Range:	35-300 amu
Ionization Mode:	EI
Scan Time:	To give at least five scans per peak, not to exceed 2 seconds per scan for capillary column.

9.2 Instrument Performance Check -- 4-Bromofluorobenzene (BFB)

9.2.1 Summary of Instrument Performance Check

9.2.1.1 The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as perfluoro-tri-n-butylamine (FC-43) or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Section 7.2.3.1).

9.2.1.2 Prior to the analysis 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 solution containing BFB.

9.2.2 Frequency of Instrument Performance Check

The instrument performance check solution must be injected once at the beginning of each 12-hour period, during which samples, blanks or standards are to be analyzed. The 12-hour time period for GC/MS performance check, calibration standards (initial or continuing calibration), blank, and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after 12 hours have elapsed according to the system clock.

9.2.3 Procedure for Instrument Performance Check

The analysis of the instrument performance check solution may be performed as follows:

- As an injection of up to 50 nanograms (ng) of BFB into the GC/MS (Section 7.2.3.1); or
- By adding 50 ng of BFB to a calibration standard (Section 7.2.3.2) and analyzing the calibration standard.

9.2.4 Technical Acceptance Criteria for Instrument Performance Check

9.2.4.1 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scan immediately preceding and the scan immediately following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the beginning of the elution of BFB. Do not background subtract part of the BFB peak.

NOTE: All subsequent standards, samples, and blanks associated with a BFB analysis must use identical MS instrument conditions.

9.2.4.2 The analysis of the instrument performance check solution must meet the ion abundance criteria given in Table D-1.

9.2.5 Corrective Action for Instrument Performance Check

9.2.5.1 If the BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective actions to achieve the technical acceptance criteria.

9.2.5.2 BFB technical acceptance criteria **must** be met before any standards, samples, or required blanks are analyzed. Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require re-analysis at no additional cost to USEPA.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to the analysis of samples and required blanks and after the instrument performance check technical acceptance criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the purgeable target and Deuterated Monitoring Compounds (DMCs).

9.3.2 Frequency of Initial Calibration

9.3.2.1 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 (i.e., ion source cleaning or repair, column replacement, etc.), or if the continuing calibration technical acceptance criteria have not been met.

9.3.2.2 If time remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed. It is not necessary to analyze a continuing calibration standard if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration technical

acceptance criteria. A method blank is required. Quantitate all samples and blank results against the initial calibration standard that is the same concentration as the continuing calibration standard. Compare Quality Control (QC) criteria such as internal standard area response change and retention time shift to the initial calibration standard that is the same concentration as the continuing calibration standard.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Assemble a purge and trap device that meets the specifications in Section 6.4. Condition the device as described in Section 9.1.1.

9.3.3.2 Connect the purge and trap device to the GC. The GC must be operated using temperature and flow rate parameters equivalent to those in Section 9.1.2.

9.3.3.3 All samples, blanks, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.

9.3.3.4 Add 10 microliters (µL) of the internal standard solution (Section 7.2.3.3) to each aqueous standard containing the DMCs for a concentration of 5 micrograms per liter (µg/L) at time of purge. Analyze each calibration standard according to Section 10.2.

9.3.4 Calculations for Initial Calibration

Calculating the Relative Response Factor (RRF) of the xylenes requires special attention. On capillary columns, the – and p-xylene isomers coelute. Therefore, when calculating the relative response factor in the equation below, use the area response (Ax) and concentration (Cx) of the peak from o-xylene.

9.3.4.1 Calculate RRF for each purgeable target compound and DMC using Equation 1. See Table D-3 to associate purgeable target compounds and DMCs with the proper internal standard. See Table D-4 for primary quantitation ions to be used for each purgeable target compound, DMC, and internal standard compound.

NOTE: Unless otherwise stated the area response is that of the primary quantitation ion.

EQ. 1

$$RRF = \frac{A_x}{A_{is}} * \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion (EICP) for the compound to be measured (Table D-4).

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (Tables D-3 and D-4).

C_{is} = Concentration of the internal standard.

C_x = Concentration of the compound to be measured.

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9.3.4.2 The mean Relative Response Factor (RRF) must be calculated for all compounds.

9.3.4.3 Calculate the Percent Relative Standard Deviation (%RSD) of RRF values for each purgeable target and DMC over the initial calibration range using Equation 2 in conjunction with Equation 3.

EQ. 2

$$\%RSD = \frac{SD_{RRF}}{\bar{x}} * 100$$

Where:

SD_{RRF} = Standard deviation of initial calibration relative response factors (per compound). From EQ. 3.

$\bar{x}$ = Mean value of the initial calibration relative response factors (per compound).

9.3.4.4 Equation 3 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 3

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n - 1}}$$

Where:

x_i = Each individual value used to calculate the mean.

$\bar{x}$ = The mean of n values.

n = Total number of values.

9.3.4.5 Equation 4 is the general formula for the mean of a set of values.

EQ. 4

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

Where:

x_i = Value.

$\bar{x}$ = Mean value.

n = Number of values.

9.3.5 Technical Acceptance Criteria For Initial Calibration

9.3.5.1 All initial calibration standards must be analyzed at the concentration levels described in Section 7.2.3.6.1, and at the frequency described in Section 9.3.2 on a GC/MS system meeting the BFB technical acceptance criteria (Section 9.2.4).

9.3.5.2 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the instrument manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.3.5.3 The RRF at each calibration concentration for each purgeable target and DMC that has a required minimum relative response factor value must be greater than or equal to the compound's minimum acceptable relative response factor listed in Table D-2.

9.3.5.4 The %RSD for each target or DMC listed in Table D-2 must be less than or equal to that value listed.

9.3.5.5 Up to two compounds may fail the criteria listed in Sections 9.3.5.3 and 9.3.5.4 and still meet the minimum RRF and %RSD requirements. However, these compounds must have a minimum RRF greater than or equal to 0.010, and the %RSD must be less than or equal to 40.0 percent.

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 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, service the purge and trap device, or take other corrective actions to achieve the technical acceptance criteria.

9.3.6.2 Initial calibration technical acceptance criteria **MUST** be met before any samples or required blanks are analyzed. Any samples

or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require re-analysis at no additional cost to USEPA.

9.4 Continuing Calibration

9.4.1 Summary of Continuing Calibration

Prior to the analysis of samples and required blanks and after BFB and initial calibration technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a continuing calibration standard containing all the purgeable target and DMCs and internal standards to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the SOW.

9.4.2 Frequency of Continuing Calibration

9.4.2.1 A check of the calibration curve must be performed once per every 12-hour time period of operation. The 12-hour time period begins with the injection of BFB.

9.4.2.2 If time remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed. It is not necessary to analyze a continuing calibration standard, if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration technical acceptance criteria. A method blank is required. Quantitate all sample and blank results against the initial calibration standard that is the same concentration as the continuing calibration standard (5.0 µg/L for non-ketones, 25 µg/L for ketones).

If time does not remain in the 12-hour period beginning with the injection of the instrument performance check solution, a new injection of the instrument performance check solution must be made. If the new injection meets the ion abundance criteria for BFB, then a continuing calibration standard may be injected.

9.4.3 Procedure for Continuing Calibration

9.4.3.1 Set up the purge and trap GC/MS system per the requirements in Section 9.1.

9.4.3.2 All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.

9.4.3.3 Add 10 µL of internal standard spiking solution (prepared as described in Section 7.2.3.3 to the 25 mL syringe or volumetric flask containing the continuing calibration standard in Section 7.2.3.6.3). Analyze the continuing calibration standard, according to Section 10.2.

9.4.4 Calculations for Continuing Calibration

9.4.4.1 Calculate a RRF for each target and DMC according to Section 9.3.4.1.

9.4.4.2 Calculate the percent difference between the continuing calibration RRF and the most recent initial calibration mean RRF for each purgeable target and DMC using Equation 5.

EQ. 5

$$\%Difference = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} * 100$$

Where:

RRF_c = Relative response factor from current continuing calibration standard.

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration.

9.4.5 Technical Acceptance Criteria for Continuing Calibration

9.4.5.1 The concentration of the volatile organic target and deuterated monitoring compounds in the continuing calibration standard must be 5.0 µg/L for non-ketones and 25 µg/L for ketones. The continuing calibration standard must be analyzed at the frequency described in Section 9.4.2 on a GC/MS system meeting the BFB (Section 9.2.4) and the initial calibration (Section 9.3.5) technical acceptance criteria.

9.4.5.2 The RRF for each purgeable target and DMC that has a required minimum RRF value must be greater than or equal to the compound's minimum acceptable RRF listed in Table D-2.

9.4.5.3 The RRF percent difference for each purgeable target and DMC listed in Table D-2 must be less than or equal to that value listed.

9.4.5.4 Up to two compounds may fail the requirements listed in Sections 9.4.5.2 and 9.4.5.3 and still meet the minimum RRF criteria and percent difference criteria. However, these compounds must have a minimum RRF greater than or equal to 0.010 and the percent difference must be within the inclusive range of ±40.0 percent.

9.4.5.5 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.4.6 Corrective Action for Continuing Calibration

9.4.6.1 If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the continuing calibration technical acceptance criteria.

9.4.6.2 Continuing calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed. Any samples or required blanks analyzed when continuing calibration technical acceptance criteria have not been met will require re-analysis at no additional cost to USEPA.

10.0 SAMPLE ANALYSIS

10.1 Summary of Sample Analysis

10.1.1 This method is designed for analysis of samples that contain low concentrations of the target compounds listed in Exhibit C. It is expected that the samples will come from drinking water and well/ground water type sources around Superfund sites. If, upon inspection of a sample, the Contractor suspects that the sample is not amenable to this method, contact Sample Management Office (SMO). SMO will contact the Region for instructions.

10.1.2 Prior to the analysis of samples, establish the appropriate purge and trap Gas Chromatograph/Mass Spectrometer (GC/MS) operating conditions, as outlined in Section 9.1, analyze the instrument performance check solution (Section 9.2), and calibrate the GC/MS system according to Sections 9.3 through 9.4.6. Also prior to sample analysis, a method blank must be analyzed that meets blank technical acceptance criteria in Section 12.1.5. All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis. All samples, required blanks, and calibration standards must be analyzed under the same instrument conditions.

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

10.2 Procedure for Sample Analysis

10.2.1 Remove the plunger from a 25 milliliters (mL) syringe that has a closed syringe valve attached. Open the sample or standard container which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Invert the syringe, open the syringe valve, and vent any residual air while adjusting the sample volume to 25.0 mL. This process of taking an aliquot destroys the validity of the sample for future analysis, unless the excess sample is immediately transferred to a smaller vial with zero headspace and stored at 4°C (±2°C).

10.2.2 Once the sample aliquots have been taken from the VOA vial, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do not add pH paper to the vial). Record the pH of each sample and report these data in the SDG Narrative, following the instructions in Exhibit B. No pH adjustment is to be performed by the Contractor.

Add 10.0 microliters (µL) of the internal standard spiking solution and 10.0 µL of the Deuterated Monitoring Compound (DMC) standard solution through the valve bore of the syringe, then close the valve. Invert the syringe three times.

10.2.3 Attach the valve assembly on the syringe to the valve on the sample purger. Open the valves and inject the sample into the purging chamber.

10.2.4 Close both valves and purge the sample for 11.0 (±0.1) minutes at ambient temperature.

10.2.5 Sample Desorption - After the 11-minute purge, attach the trap to the GC, adjust the purge and trap system to the desorb mode, initiate the temperature program sequence of the GC and start data acquisition. Introduce the trapped material to the GC column by rapidly heating the trap to 180°C while backflushing the trap with inert gas at 15 mL/min for 4.0 ±0.1 min. While the trapped material is being introduced into the GC, empty the sample purger and rinse it with reagent water. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high purgeable levels, it may be necessary to wash out the sample purger with a detergent solution, rinse it with reagent water, and then dry it in an oven at 105°C.

10.2.6 Trap Reconditioning - After desorbing the sample, recondition the trap for a minimum of 7.0 ±0.1 min at 180°C by returning the purge and trap system to purge mode.

10.2.7 Gas Chromatography - Hold the column temperature at 10°C for 1.0 to 5.0 min, then program at 6°C/min to 160°C and hold until three minutes after all target volatile compounds have eluted.

NOTE: Once an initial hold time has been chosen and the GC operating conditions optimized, the same GC condition must be used for the analysis.

10.2.8 Termination of Data Acquisition - Three minutes after all the purgeable 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 appropriate Extracted Ion Current Profiles (EICPs).

10.2.9 Dilutions

10.2.9.1 An original undiluted analysis must be made and results reported for all samples. If the on-column concentration of any target compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and purged. Guidance for performing dilutions and exceptions to this requirement are given in Sections 10.2.9.2 through 10.2.9.8.

NOTE 1: If the laboratory has evidence or highly suspects, because of sample color or other physical property, that a sample may contain high concentrations of either target or non-target

compounds, then SMO shall be contacted immediately. SMO will seek regional recommendations for diluted analysis.

NOTE 2: Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion, not when saturation occurs. If secondary ion quantitation is used, calculate a Relative Response Factor (RRF) using the area response (EICP) from the most intense secondary ion which is free of sample interferences, and document the reasons in the SDG Narrative.

10.2.9.2 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.

10.2.9.3 The dilution factor chosen should keep the concentration of the volatile target compounds that required dilution in the upper half of the initial calibration range.

10.2.9.4 All dilutions must be made just prior to GC/MS analysis of the sample. Until the diluted sample is in a gas-tight syringe, all steps in the dilution procedure must be performed without delay.

10.2.9.5 Samples may be diluted in a volumetric flask or in a 25 mL "Luerlock" syringe.

10.2.9.6 To dilute the sample in a volumetric flask, use the following procedure:

10.2.9.6.1 Select the volumetric flask that will allow for necessary dilution (25 mL to 100 mL).

10.2.9.6.2 Calculate the approximate volume of reagent water which will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

10.2.9.6.3 Inject the proper sample aliquot from a syringe into the volumetric flask. Only aliquots of 1 mL increments are permitted. Dilute the aliquot to the mark with reagent water. Cap the flask and invert it three times.

10.2.9.6.4 Fill a 25 mL syringe with the diluted sample and analyze according to Section 10.2.

10.2.9.7 To dilute the sample in a 25 mL syringe, use the following procedure:

10.2.9.7.1 Calculate the volume of the reagent water necessary for the dilution. The final volume of the diluted sample should be 25 mL.

10.2.9.7.2 Close the syringe valve, remove the plunger from the syringe barrel, and pour reagent water into the syringe barrel to just short of overflowing.

10.2.9.7.3 Replace the syringe plunger and compress the water.

10.2.9.7.4 Invert the syringe, open the syringe valve, and vent any residual air. Adjust the water volume to the desired amount.

10.2.9.7.5 Adjust the plunger to the 25 mL mark to accommodate the sample aliquot. Inject the proper aliquot of sample from another syringe through the valve bore of the 25 mL syringe. Close the valve and invert three times. Analyze according to Section 10.2.

10.2.9.8 For total xylenes where three isomers are quantified as two peaks, the calibration of each peak should be considered separately, e.g., a diluted analysis is not required for total xylenes unless the concentration of the peak representing the single isomer exceeds 25 micrograms per liter (µg/L) on-column, or the peak representing the two co-eluting isomers exceeds 50 µg/L on-column.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification of Target Compounds

11.1.1 The compounds listed in the Target Compound List (TCL), Exhibit C (Volatiles), 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 component at the same Gas Chromatograph (GC) Relative Retention Time (RRT) as the standard component; and
- Correspondence of the sample component and calibration standard component mass spectra.

11.1.2 For establishing correspondence of the GC RRT, the sample component RRT must be 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 samples are analyzed during the 12-hour time period as the initial calibration, use the RRT values from the 5 micrograms per liter (µg/L) standard. If co-elution of interfering compounds prohibits accurate assignment of the sample component RRT from the total ion chromatogram, then the RRT should be assigned using the Extracted Ion Current Profile (EICP) for ions unique to the component of interest.

11.1.3 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 4-Bromofluorobenzene (BFB). These standard spectra may be obtained from the standard analysis that was also used to obtain the RRTs.

11.1.4 The guidelines for qualitative verification by comparison of mass spectra are as follows:

11.1.4.1 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.

11.1.4.2 The relative intensities of ions specified in Section 11.1.4.1 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.)

11.1.4.3 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 Contract Required Quantitation Limit (CRQL), report the actual value followed by a "J" (e.g., "0.3J").

11.1.4.4 If a compound cannot be verified by all of the spectral identification criteria listed in Section 11.1.4, 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 quantitation.

11.2 Qualitative Identification of Non-Target Compounds

11.2.1 A library search shall be executed for non-target sample components for the purpose of tentative identification. The NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later) or equivalent mass spectral library, shall be used as the reference library.

11.2.2 Up to 30 organic compounds of greatest apparent concentration not listed in Exhibit C for the volatile or semivolatile organic fraction, excluding the Deuterated Monitoring Compounds (DMCs) and internal standard compounds, shall be tentatively identified via a forward search of the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent mass spectral library. The following are not to be reported:

- Compounds with a response of less than 10 percent of the internal standard (as determined by inspection of the peak areas or heights);
- Compounds which elute earlier than 30 seconds before the first purgeable compound listed in Exhibit C (Volatiles) or three minutes after the last purgeable compound listed in Exhibit C (Volatiles) are not required to be searched in this fashion;
- Carbon dioxide; and
- Semivolatile TCL compounds listed in Exhibit C.

Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

NOTE: Computer-generated library search routines must not use normalizations which would misrepresent the library or unknown spectra when compared to each other.

11.2.3 Up to 20 peaks of greatest apparent concentration (as determined by inspection of peak areas or heights) that are suspected to be straight-chain, branched, or cyclic alkanes, alone or part of an alkane series, shall be library searched. Documentation for the tentative identification must be supplied. Alkanes are not counted as part of the 30 organic compounds described in Section 11.2.2.

11.2.4 Guidelines for making tentative identification:

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

11.2.4.2 The relative intensities of the major ions specified in Section 11.2.4.1 must agree within ±20 percent between the reference and sample spectra. (Example: For an ion with an abundance of 50 percent in the reference spectrum, the corresponding sample ion abundance must be between 30 and 70 percent.)

11.2.4.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

11.2.4.5 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.

11.2.4.6 Non-target compounds receiving a library search match of 85 percent or higher should be considered a "probable match". The compound should be reported unless the mass spectral interpretation specialist feels there is just evidence not to report the compound as identified by the library search program. The lab should include in the Sample Delivery Group (SDG) Narrative the justification for not reporting a compound as listed by the search program.

11.2.4.7 If the library search produces more than one compound at or above 85 percent, report the compound with the highest percent match (report first compound if percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels that the highest match compound should not be reported, or another compound with a lower match should be reported. The lab should include in the SDG Narrative the justification for not reporting the compound with the highest spectral match.

11.2.4.8 If the library search produces a series of obvious isomer compounds with library search matches greater than 85 percent (e.g., tetramethyl naphthalenes), the compound with the highest library search percent match should be reported (or first compound if library search matches are the same). A note should be placed in the SDG Narrative indicating the exact isomer configuration, as reported, may not be accurate.

11.2.4.9 If the library search produces no matches at or above 85 percent and in the technical judgement 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 (e.g., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

11.2.4.10 Straight-chain, branched, or cyclic alkanes are <u>not</u> to be reported as tentatively identified compounds on Form I LCV-TIC. When the above alkanes are tentatively identified, the concentration(s) are to be estimated as described in Section 11.3.2 and <u>reported in the SDG Narrative as alkanes, by class</u> (i.e., straight-chain, branched, or cyclic, as a series, as applicable).

11.3 Calculations

11.3.1 Target Compounds

11.3.1.1 Target compounds identified shall be quantified by the internal standard method using Equation 6. The internal standard used shall be that which is assigned in Table D-3. The Relative Response Factor (RRF) from the continuing calibration standard is used to calculate the concentration in the sample. When a target compound concentration is below its CRQL but the spectra meets the identification criteria, report the concentration with a "J". For example, if the CRQL is 0.50 µg/L and a concentration of 0.30 µg/L is calculated, report as "0.30 J". Report ALL sample concentration data as UNCORRECTED for blanks.

EQ. 6

$$\text{Concentration in ug/L} = \frac{(A_x)\ (I_s)\ (Df)}{(A_{is})\ (RRF)\ (V_o)}$$

Where:

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target compounds, internal standards, and the DMCs are listed in Table D-4.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target compounds are listed with their associated internal standards in Table D-3.

I_s = Amount of internal standard added in nanograms (ng).

RRF = The relative response factor from the continuing calibration standard.

V_o = Total volume of water purged, in milliliters (mL).

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. For example, if 5.0 mL of sample is diluted to 25.0 mL with reagent water and purged, Df=25.0 mL/5 mL = 5.0. If no dilution is performed, Df =1.0.

11.3.1.2 Xylenes (o-, m-, and p- isomers) are to be reported as xylenes (total). Because – and p-xylene isomers coelute on capillary columns, special attention must be given to the quantitation of the xylenes. The RRF determined in Section 9.4.4.1, is based on the peak that represents the single isomer on the GC column (o-xylene on capillary columns). In quantitating sample concentrations, use the areas on <u>both</u> peaks and the RRF. The areas of the two peaks may be summed and the concentration determined, or the concentration represented by each of the two peaks may be determined separately and then summed.

11.3.1.3 The stereoisomers, trans-1,2-dichloroethene, and cis-1,2-dichloroethene, are to be reported separately.

11.3.1.4 The requirements listed in Sections 11.3.1.5 and 11.3.1.6 apply to all standards, samples, and blanks.

11.3.1.5 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitation. This normally occurs when there is compound co-elution, baseline noise, or matrix interferences. In these circumstances the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific TCL, deuterated monitoring, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instances of manual integration must be documented in the SDG Narrative.

11.3.1.6 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator <u>must</u> identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standard, and DMCs.

11.3.2 Non-Target Compounds

11.3.2.1 An estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used.

11.3.2.2 Equation 6 is also used for calculating non-target compound concentrations. Total area counts (or peak heights) from the total Reconstructed Ion Chromatograms (RICs) are to be used for both the non-target compound to be measured (A_x) and the internal standard (A_{is}). A RRF of 1.0 is to be assumed. The value from this quantitation shall be qualified by a "J" (estimate due to lack of a compound-specific relative response factor), and "N" (presumptive evidence of presence), indicating the qualitative and quantitative uncertainties associated with this non-target compound. An estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns.

11.3.3 CRQL Calculation

Calculate the adjusted CRQL for volatiles by using Equation 7.

EQ. 7

$$\text{Adjusted CRQL} = \text{Contract CRQL} * \frac{V_c}{V_o} * Df$$

Where:

Contract CRQL = Exact CRQL values in Exhibit C of the SOW.

V_o = Total volume of water purged in milliliters.
NOTE: Must not exceed the contract sample volume.

V_c = Contract sample volume in milliliters (25 mL).

Df = Same as EQ. 6.

NOTE: If the adjusted CRQL is less than the CRQL listed in Exhibit C (Volatiles), report the CRQL listed in Exhibit C (Volatiles).

11.3.4 Deuterated Monitoring Compound Recoveries

11.3.4.1 Calculate the concentration of each DMC using the same equation as used for target compounds (Equation 6).

11.3.4.2 Calculate the recovery of each DMC in all samples and blanks using Equation 8. Report the recoveries on appropriate forms.

EQ. 8

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} * 100$$

Where:

Q_d = Concentration or amount determined by analysis.

Q_a = Concentration or amount added to sample/blank.

11.3.5 Internal Standard Responses and Retention Times

Internal standard responses and retention times in all samples and blanks must be evaluated during or immediately after data acquisition. Compare the sample/blank internal standard responses and retention times to the continuing calibration internal standard responses and retention times. For samples and blanks analyzed during the same 12-hour time period as the initial calibration standards, compare the internal standard responses and retention times against the 5 µg/L calibration standard. The EICP of the internal standards must be monitored and evaluated for each sample and blank.

11.4 Technical Acceptance Criteria for Sample Analysis

11.4.1 The sample must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration, and blank technical acceptance criteria.

11.4.2 The sample and any required dilution must be analyzed within the contract holding time.

11.4.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

11.4.4 The percent recovery of each of the DMCs in the sample must be within the acceptance windows in Table D-5. Up to three DMCs per sample may fail to meet the recovery limits listed in Table D-5.

11.4.5 The EICP area for each of the internal standards in the sample must be within the inclusive range of ±40.0 percent of its response in the most recent continuing calibration standard analysis.

11.4.6 The retention time shift for each of the internal standards in the sample must be within ±0.33 minutes (20.0 seconds) of its retention time in the most recent continuing calibration standard analysis.

11.4.7 The RRT of each of the DMCs in the sample must be within ±0.06 RRT units of its relative retention time in the most recent continuing calibration standard analysis.

11.4.8 Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the initial calibration range unless a more dilute aliquot of the sample is also analyzed according to the procedures in Section 10.2.9.

11.4.9 The Contractor must demonstrate that there is no carryover from a contaminated sample before data from subsequent analyses may be submitted. After a sample that contains a target compound at a level exceeding the initial calibration range, or a non-target compound at a concentration greater than 100 µg/L, or saturated ions from a compound (excluding the compound peaks in the solvent front), the Contractor must either:

11.4.9.1 Analyze an instrument blank immediately after the contaminated sample. If an autosampler is used, an instrument blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The instrument blanks must meet the technical acceptance criteria for blank analysis (Section 12.1.5); or

11.4.9.2 Monitor the analyzed sample immediately after the contaminated sample for all the compounds that were in the contaminated sample and that exceeded the limits above. The maximum carryover criteria are as follows: the sample must not contain a concentration above the CRQL for the target compounds, or above 2 µg/L for the non-target compounds that exceeded the limits in the contaminated sample. If an autosampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample must also meet the maximum carryover criteria.

11.5 Corrective Action for Sample Analysis

11.5.1 Sample technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or any samples not meeting the sample technical acceptance criteria will require re-analysis at no additional cost to USEPA.

11.5.2 Corrective actions for failure to meet instrument performance checks, initial calibration, continuing calibration, and method blanks must be completed before the analysis of samples.

11.5.3 If the technical acceptance criteria for any of the internal standards and DMCs are not met, check calculations, internal standard and DMC spiking solutions, and instrument performance. It may be necessary to bake-out the system to remove the water from the purge and trap transfer lines, to recalibrate the instrument, or take other corrective action procedures to meet the technical acceptance criteria.

11.5.4 Sample reruns performed as a result of suspected matrix interference beyond the scope of the method will be evaluated on a case-by-case basis for payment purposes by the USEPA Contract Laboratory Program Project Officer (CLP PO). Send a copy of the SDG Narrative

(including your contract number), a description of the situation, and the requested action to the CLP PO.

11.5.5 If the contractor needs to analyze more than one (1) sample dilution other than the original analysis to have all the target compounds within the initial calibration range, contact Sample Management Office (SMO). SMO will contact the Region for instruction.

11.5.6 All samples to be reported to USEPA must meet the maximum carryover criteria in Section 11.4.9. If any sample fails to meet these criteria, each subsequent analysis must be checked for cross contamination. The analytical system is considered contaminated until a sample has been analyzed that meets the maximum carryover criteria or an instrument blank has been analyzed that meets the technical acceptance criteria for blanks.

12.0 QUALITY CONTROL (QC)

12.1 Blank Analyses

12.1.1 Summary of Blank Analyses

There are three different types of blanks required by this method.

12.1.1.1 Method Blank - 25 milliliters (mL) of reagent water spiked with 10.0 microliters (µL) internal standard solution and 10.0 µL Deuterated Monitoring Compound (DMC) solution, and carried through the entire analytical procedure. The purpose of the method blank is to determine the levels of contamination associated with processing and analysis of samples.

12.1.1.2 Storage Blank - Upon receipt of the first samples in a Sample Delivery Group (SDG), two 40 mL screw cap VOA vials with a PTFE-faced silicone septum are filled with reagent water (80 mL total). The vials are stored with the samples in the SDG under the same conditions. A 25.0 mL aliquot of this reagent water is spiked with a 10.0 µL internal standard solution and 10.0 µL of DMC solution and analyzed after all samples in the SDG have been analyzed. The storage blank indicates whether contamination may have occurred during storage of samples.

12.1.1.3 Instrument Blank - 25 mL of reagent water spiked with 10.0 µL of internal standard solution and 10.0 µL of DMC solution and carried through the entire analytical procedure. Instrument blanks are analyzed after a sample/dilution which contains a target compound at a concentration greater than 25 micrograms per liter (µg/L) (ketones 125 µg/L), or a non-target compound at a concentration greater than 100 µg/L or saturated ions from a compound (excluding the compound peaks in the solvent front). The results from instrument blank analysis indicate whether there is contamination from a previous sample.

12.1.2 Frequency of Blank Analyses

12.1.2.1 The method blank must be analyzed at least once during every 12-hour time period on each Gas Chromatograph/Mass Spectrometer (GC/MS) system used for volatile analysis (see Section 9.2.2 for the definition of the 12-hour time period).

12.1.2.2 The method blank must be analyzed after the continuing calibration standard and before any samples or storage blanks are analyzed. The method blank must be analyzed after the initial calibration sequence if samples are analyzed before the 12-hour time period expires. A method blank must be analyzed in each 12-hour time period in which samples (including dilutions) and storage blanks from an SDG are analyzed.

12.1.2.3 A minimum of one storage blank must be analyzed per SDG, after all samples for the SDG have been analyzed, unless the SDG contains only ampulated Performance Evaluation (PE) samples. Analysis of a

storage blank is not required for SDGs that contain only ampulated PE samples.

12.1.2.4 The Contractor must demonstrate that there is no carryover from contaminated samples before data from subsequent analyses may be used. Samples may contain target compounds at levels exceeding the initial calibration range or non-target compounds at concentrations greater than 100 µg/L, or ions from a compound that saturate the detector (excluding the compound peaks in the solvent front). An instrument blank must be analyzed immediately after the contaminated sample (also in the same purge inlet if an autosampler is used), or a sample that meets the maximum carryover criteria in Section 11.4.9 must be analyzed. For these purposes, if the instrument blank meets the technical acceptance criteria for blank analysis or the sample meets the maximum carryover criteria, the system is considered to be uncontaminated. If the instrument blank or sample does not meet the criteria (i.e., contaminated), the system must be decontaminated. Until an instrument blank meets the blank technical acceptance criteria or a sample meets the maximum carryover criteria, any samples analyzed since the original contaminated sample will require re-analysis at no additional expense to USEPA.

NOTE: Only the instrument blank which demonstrates that there was no carryover from the previous sample or the instrument blank that demonstrates that the system is clean (Section 12.1.5.7) must be reported. Instrument blanks analyzed during the instrument decontamination process which exceed the requirements listed in Section 11.4.9 do not need to be reported.

12.1.3 Procedure for Blank Analyses

12.1.3.1 Spike 25 mL of reagent water with 10.0 µL of the internal standard solution (Section 7.2.3.3), and 10.0 µL of the DMC solution (Section 7.2.3.4).

12.1.3.2 Prepare and analyze the blanks as described in Section 10.2.

12.1.4 Calculations for Blank Analyses

Perform data analysis and calculations according to Section 11.

12.1.5 Technical Acceptance Criteria for Blank Analyses

12.1.5.1 All blanks must be analyzed on a GC/MS system meeting the 4-Bromofluorobenzene (BFB), initial calibration, and continuing calibration technical acceptance criteria, and at the frequency described in Section 12.1.2.

12.1.5.2 The storage blank must be analyzed on a GC/MS system that also meets the technical acceptance criteria for the method blank.

12.1.5.3 The percent recovery of each of the DMCs in the blank must be within the acceptance windows in Table D-5.

12.1.5.4 The EICP area for each of the internal standards in the blank must be within the inclusive range of ±40.0 percent of its response in the most recent continuing calibration standard analysis.

12.1.5.5 The retention time shift for each of the internal standards in the blank must be within ±0.33 minutes (20.0 seconds) of its retention time in the most recent continuing calibration standard analysis.

12.1.5.6 The Relative Retention Time (RRT) of each of the DMCs in the blank must be within ±0.06 RRT units of its relative retention time in the most recent continuing calibration standard analysis.

12.1.5.7 The concentration of each target compound found in the storage and method blanks must be less than its CRQL listed in Exhibit C (Volatiles), except for methylene chloride and cyclohexane which must be less than 10 times their respective CRQLs, and acetone and 2-butanone, which must be less than two times their respective CRQLs. The concentration of each target compound in the instrument blank must be less than its CRQL listed in Exhibit C (Volatiles). The concentration of non-target compounds in all blanks must be less than 2.0 µg/L.

12.1.6 Corrective Action for Blank Analyses

12.1.6.1 It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms, be eliminated. If a Contractor's blanks exceed the criteria in Section 12.1.5.7, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds.

12.1.6.2 Any method blank or instrument blank that fails to meet the technical acceptance criteria must be re-analyzed at no additional cost to USEPA. Further, all samples processed within the 12-hour time period with a method blank or instrument blank that does not meet the blank technical acceptance criteria will require re-analysis at no additional cost to USEPA.

12.1.6.3 If the storage blank does not meet the technical acceptance criteria for blank analyses in Sections 12.1.5.1 to 12.1.5.6, correct system problems and re-analyze the storage blank. If the storage blank does not meet the criteria in Section 12.1.5.7, re-analyze the blank to determine whether the contamination occurred during storage or during analyses. If upon re-analysis, the storage blank meets the criteria in Section 12.1.5.7, the problem occurred during the analysis and the re-analyzed storage blank results must be reported. If upon re-analysis the storage blank did not meet the criteria in Section 12.1.5.7, the problem occurred during storage. The laboratory manager or his/her designee must address the problem in the SDG Narrative and discuss the corrective actions implemented to prevent future occurrences.

NOTE: A copy of the storage blank data must be retained by the Contractor and be made available for inspection during on-site laboratory evaluations.

12.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

12.2.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the method used for volatile analysis, USEPA has prescribed a mixture of volatile target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method, upon request.

12.2.2 Frequency of MS/MSD

12.2.2.1 A MS/MSD shall only be analyzed if requested by the Region (through the Sample Management Office (SMO)) or specified on the Traffic Report (TR). If requested, a matrix spike and a matrix spike duplicate must be performed for each group of 20 field samples in an SDG, or each SDG, whichever is most frequent.

12.2.2.2 As part of USEPA's Quality Assurance (QA)/Quality Control (QC) program, water rinsate samples and/or field/trip blanks (field QC) may be delivered to a laboratory for analysis. The Contractor shall not perform MS/MSD analysis on any of the field QC samples.

12.2.2.3 If the USEPA Region requesting MS/MSD designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample, less than the required amount, remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify the Region (through SMO) that insufficient sample was received and identify the USEPA sample selected for the MS/MSD analysis. The rationale for the choice of a sample other than the one designated by the Region shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample remaining in any of the samples in an SDG to perform an MS/MSD, then the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no MS/MSD is required, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the resolution. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2.1, the Contractor shall contact SMO. SMO will contact the Region to determine which samples should have an MS/MSD performed on them. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative. If this procedure is not followed, the Contractor will not be paid for MS/MSD analysis performed at a greater frequency than required by the contract.

12.2.2.6 When a Contractor receives **only** Performance Evaluation (PE) sample(s), no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the requested MS/MSD analysis when the Region did not designate samples to be used for this purpose.

12.2.3 Procedure for Preparing MS/MSD

12.2.3.1 To prepare MS/MSD samples, add 10 µL of the matrix spike solution (Section 7.2.3.5) to each of the 25 mL aliquots of the sample chosen for spiking. Process samples according to Section 10.2. Disregarding any dilutions, this is equivalent to a concentration of 5 µg/L of each matrix spike compound.

12.2.3.2 MS/MSD samples must be analyzed at the same concentration as the most concentrated aliquot for which the original sample results will be reported. Sample dilutions must be performed in accordance with Section 10.2.9. Do **not** further dilute MS/MSD samples to get **either** spiked **or** non-spiked analytes within calibration range.

12.2.4 Calculations for MS/MSD

12.2.4.1 Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds (Equation 6). Calculate the recovery of each matrix spike compound as follows:

EQ. 9

$$\textbf{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result.

SR = Sample result.

SA = Spike added.

12.2.4.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each compound in the MS/MSD as follows:

EQ. 10

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

MSR = Matrix spike recovery.

MSDR = Matrix spike duplicate recovery.

12.2.5 Technical Acceptance Criteria for MS/MSD

12.2.5.1 If requested, all MS/MSD must be prepared and analyzed at the frequency described in Section 12.2.2. All MS/MSDs must be analyzed on a GC/MS system meeting the BFB, initial and continuing calibration technical acceptance criteria, and the blank technical acceptance criteria.

12.2.5.2 The MS/MSD must be analyzed within the contract holding time.

12.2.5.3 The retention time shift for each of the internal standards in the MS/MSD must be within ±0.33 minutes (20 seconds) of its retention time in the most recent continuing calibration standard analysis.

12.2.5.4 The limits for matrix spike compound recovery and RPD are given in Table D-6. As these limits are only advisory, no further action by the laboratory is required. However, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questions from USEPA.

12.2.5.5 The relative retention time for the DMCs must be within ±0.06 RRT units of its standard retention time in the Continuing Calibration Standard.

12.2.6 Corrective Action for MS/MSD

Any MS/MSD that does not meet the technical acceptance criteria in Sections 12.2.5.1 through 12.2.5.3 must be re-analyzed at no additional cost to USEPA.

12.3 Method Detection Limit (MDL) Determination

12.3.1 Before any field samples are analyzed under this contract, the MDL for each volatile target compound shall be determined on each instrument used for analysis. The MDLs must be verified annually thereafter (see Section 12.3.2 for MDL verification procedures), until the contract expires or is terminated, or after major instrument maintenance. Major instrument maintenance includes, but is not limited to cleaning or replacement of the mass spectrometer source, mass filters (e.g., quadrupole, ion trap, etc.), or electron multiplier (or similar device), replacement of gas chromatographic column, and replacement or overhaul of the purge and trap device.

12.3.2 To determine the MDLs, the Contractor shall run an MDL study following the procedures specified in 40 CFR Part 136. The Contractor shall analyze the MDL samples on each instrument used for field sample analyses. MDL verification is achieved by analyzing a single reagent water blank spiked with each volatile target compound

at a concentration equal to two times the analytical determined MDL. The resulting mass spectra of each target compound must meet the qualitative identification criteria outlined in Sections 11.1.1 through 11.1.4.3

12.3.3 The determined concentration of the MDL must be less than the CRQL.

12.3.4 All documentation for the MDL studies shall be maintained at the laboratory and provided to USEPA upon written request.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibility reduced at the source, USEPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington D.C., 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

15.1 USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. USEPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel*, available from the American Chemical Society at the address listed in Section 14.2.

16.0 REFERENCES

Not applicable.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE D-1 BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95 (see note)
173	less than 2.0 percent of mass 174
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 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-2
TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION FOR VOLATILE ORGANIC COMPOUNDS

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Dichlorodifluoromethane	0.010	none	none
Chloromethane	0.010	none	none
Vinyl chloride	0.100	30.0	±30.0
Bromomethane	0.100	30.0	±30.0
Chloroethane	0.010	none	none
Trichlorofluoromethane	0.010	none	none
1,1-Dichloroethene	0.100	30.0	±30.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.010	none	none
Acetone	0.010	none	none
Carbon disulfide	0.010	none	none
Methyl Acetate	0.010	none	none
Methylene chloride	0.010	none	none
trans-1,2-Dichloroethene	0.010	none	none
Methyl tert-Butyl Ether	0.010	none	none
1,1-Dichloroethane	0.200	30.0	±30.0
cis-1,2-Dichloroethene	0.010	none	none
2-Butanone	0.010	none	none
Bromochloromethane	0.050	30.0	±30.0
Chloroform	0.200	30.0	±30.0
1,1,1-Trichloroethane	0.100	30.0	±30.0
Cyclohexane	0.010	none	none
Carbon tetrachloride	0.100	30.0	±30.0
Benzene	0.400	30.0	±30.0
1,2-Dichloroethane	0.100	30.0	±30.0
Trichloroethene	0.300	30.0	±30.0
Methylcyclohexane	0.010	none	none
1,2-Dichloropropane	0.010	none	none
Bromodichloromethane	0.200	30.0	±30.0
cis-1,3-Dichloropropene	0.200	30.0	±30.0
4-Methyl-2-pentanone	0.010	none	none
Toluene	0.400	30.0	±30.0
trans-1,3-Dichloropropene	0.100	30.0	±30.0
1,1,2-Trichloroethane	0.100	30.0	±30.0
Tetrachloroethene	0.100	30.0	±30.0
2-Hexanone	0.010	none	none
Dibromochloromethane	0.100	30.0	±30.0
1,2-Dibromoethane	0.100	30.0	±30.0
Chlorobenzene	0.500	30.0	±30.0
Ethylbenzene	0.100	30.0	±30.0
Xylene (total)	0.300	30.0	±30.0
Styrene	0.300	30.0	±30.0
Bromoform	0.050	30.0	±30.0
Isopropylbenzene	0.010	none	none

TABLE D-2
TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION FOR VOLATILE ORGANIC COMPOUNDS (Con't)

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
1,1,2,2-Tetrachloroethane	0.100	30.0	±30.0
1,3-Dichlorobenzene	0.400	30.0	±30.0
1,4-Dichlorobenzene	0.400	30.0	±30.0
1,2-Dichlorobenzene	0.400	30.0	±30.0
1,2-Dibromo-3-chloropropane	0.010	none	none
1,2,4-Trichlorobenzene	0.200	30.0	±30.0
1,2,3-Trichlorobenzene	0.200	30.0	±30.0
DEUTERATED MONITORING COMPOUNDS			
Vinyl Chloride-d3	0.010	none	none
Chloroethane-d5	0.010	none	none
1,1-Dichloroethene-d2	0.010	none	none
2-Butanone-d5	0.010	none	none
Chloroform-d	0.010	none	none
1,2-Dichloroethane-d4	0.010	none	none
Benzene-d6	0.010	none	none
1,2-Dichloropropane-d6	0.010	none	none
Toluene-d8	0.010	none	none
trans-1,3-Dichloropropene-d4	0.010	none	none
2-Hexanone-d5	0.010	none	none
Bromoform-d	0.010	none	none
1,1,2,2-Tetrachloroethane-d2	0.010	none	none
1,2-Dichlorobenzene-d4	0.010	none	none

TABLE D-3
VOLATILE TARGET COMPOUNDS AND DEUTERATED MONITORING COMPOUND (DMC)
WITH CORRESPONDING INTERNAL STANDARDS FOR QUANTITATION

1,4-Difluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	1,1,1-Trichloroethane	Bromoform
Chloromethane	Cyclohexane	1,3-Dichlorobenzene
Vinyl Chloride	Carbon Tetrachloride	1,4-Dichlorobenzene
Bromomethane	Benzene	1,2-Dichlorobenzene
Chloroethane	Trichloroethene	1,2-Dibromo-3-chloropropane
Trichlorofluoromethane	Methylcyclohexane	1,2,4-Trichlorobenzene
1,1-Dichloroethene	1,2-Dichloropropane	1,2,3-Trichlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Bromodichloromethane	1,2-Dichlorobenzene-d4 (DMC)
Acetone	cis-1,3-Dichloropropene	Bromoform-d (DMC)
Carbon Disulfide	4-Methyl-2-pentanone	
Methyl Acetate	Toluene	
Bromochloromethane	trans-1,3-Dichloropropene	
Methylene Chloride	1,1,2-Trichloroethane	
trans-1,2-Dichloroethene	Tetrachloroethene	
Methyl tert-Butyl Ether	2-Hexanone	
1,1-Dichloroethane	Dibromochloromethane	
cis-1,2-Dichloroethene	1,2-Dibromoethane	
2-Butanone	Chlorobenzene	
Chloroform	Ethylbenzene	
1,2-Dichloroethane	Xylenes (total)	
Vinyl Chloride-d3 (DMC)	Styrene	
Chloroethane-d5 (DMC)	Isopropylbenzene	
1,1-Dichloroethene-d2 (DMC)	1,1,2,2-Tetrachloroethane	
2-Butanone-d5 (DMC)	Benzene-d6 (DMC)	
Chloroform-d (DMC)	1,2-Dichloropropane-d6 (DMC)	
1,2-Dichloroethane-d4 (DMC)	trans-1,3-Dichloropropene-d4 (DMC)	
	Toluene-d8 (DMC)	
	2-Hexanone-d5 (DMC)	
	1,1,2,2-Tetrachloroethane-d2 (DMC)	

TABLE D-4
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Target Compound	Primary Quantitation Ion	Secondary Ion(s)
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61, 63
1,1,2-Trichloro-1,2,2-trifluoroethane	101	85, 151
Acetone	43	58
Carbon disulfide	76	78
Methyl Acetate	43	74
Methylene chloride	84	49, 86
trans-1,2-Dichloroethene	96	61, 98
Methyl tert-Butyl Ether	73	43, 57
1,1-Dichloroethane	63	65, 83
cis-1,2-Dichloroethene	96	61, 98
2-Butanone	43*	72
Chloroform	83	85
Bromochloromethane	128	49, 130, 51
1,1,1-Trichloroethane	97	99, 61
Cyclohexane	56	69, 84
Carbon Tetrachloride	117	119
Benzene	78	-
1,2-Dichloroethane	62	98
Trichloroethene	95	97, 132, 130
Methylcyclohexane	83	55, 98
1,2-Dichloropropane	63	112
Bromodichloromethane	83	85, 127
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58, 100
Toluene	91	92
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Tetrachloroethene	164	129, 131, 166
2-Hexanone	43	58, 57, 100
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Chlorobenzene	112	77, 114
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
Bromoform	173	175, 254

*m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

TABLE D-4
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS (Con't)

Analyte	Primary Quantitation Ion	Secondary Ion(s)
Isopropylbenzene	105	120, 77
1,1,2,2-Tetrachloroethane	83	85, 131
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene	146	111, 148
1,2-Dibromo-3-Chloropropane	75	157, 155
1,2,4-Trichlorobenzene	180	182, 145
1,2,3-Trichlorobenzene	180	182, 145
Deuterated Monitoring Compounds		
Vinyl Chloride-d3	65	67
Chloroethane-d5	69	71, 51
1,1-Dichloroethene-d2	63	98, 65
2-Butanone-d5	46	77
Chloroform-d	84	86, 47, 49
1,2-Dichloroethane-d4	65	67, 51
Benzene-d6	84	82, 54, 52
1,2-Dichloropropane-d6	67	65, 46, 42
Toluene-d8	98	100, 42
trans-1,3-Dichloropropene-d4	79	81, 42
2-Hexanone-d5	63	46
Bromoform-d	174	172
1,1,2,2-Tetrachloroethane-d2	84	86
1,2-Dichlorobenzene-d4	152	150
Internal Standards		
1,4-Dichlorobenzene-d4	152	115, 150
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d5	117	82, 119

TABLE D-5
DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

Compound	Percent Recovery Limits
Vinyl Chloride-d3	49-138
Chloroethane-d5	60-126
1,1-Dichloroethene-d2	65-130
2-Butanone-d5	42-171
Chloroform-d	80-123
1,2-Dichloroethane-d4	78-129
Benzene-d6	78-121
1,2-Dichloropropane-d6	84-123
Toluene-d8	77-120
trans-1,3-Dichloropropene-d4	80-128
2-Hexanone-d5	37-169
Bromoform-d	76-135
1,1,2,2-Tetrachloroethane-d2	75-131
1,2-Dichlorobenzene-d4	50-150

NOTE: The recovery limits for any of the compounds listed above may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

TABLE D-6
MATRIX SPIKE RECOVERY AND
RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery	RPD
1,1-Dichloroethene	61-145	14
Benzene	76-127	11
Trichloroethene	71-120	14
Toluene	76-125	13
Chlorobenzene	75-130	13

EXHIBIT D

METHOD FOR THE ANALYSIS OF LOW CONCENTRATION WATER FOR
SEMIVOLATILE ORGANIC COMPOUNDS

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Exhibit D -- Analytical Methods for Semivolatiles

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water samples containing low concentrations of the semivolatile compounds listed on the Target Compound List (TCL) in Exhibit C. The majority of the samples are expected to be from drinking water and well/ground water/aqueous type sources around Superfund sites. The method is based upon the semivolatile method contained in the Contract Laboratory Program (CLP) Statement of Work, "Organic Analysis, Multi-Media, Multi-Concentration Analyses". The analytical method includes the use of Deuterated Monitoring Compounds (DMC) for precision and accuracy assessment.

1.2 Benzaldehyde, Acetophenone, Caprolactam, 1,1'-Biphenyl, Atrazine, and 1,2,4,5-Tetrachlorobenzene have been added to the TCL.

1.3 Problems have been associated with the following compounds analyzed by this method:

- 3,3'-Dichlorobenzidine and 4-chloroaniline may be subject to oxidative losses during solvent concentration.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the Gas Chromatograph (GC), chemical reactions in acetone solution, and photochemical decomposition.
- N-nitrosodiphenylamine decomposes in the gas chromatographic inlet forming diphenylamine and, consequently, may be detected as diphenylamine.
- Due to the lower quantitation limits required by this method, extra caution must be exercised when identifying compounds.

2.0 SUMMARY OF METHOD

2.1 A one liter aliquot of sample is acidified to pH 2.0 and extracted with methylene chloride using a continuous liquid-liquid extractor. Separatory funnel extraction is not permitted. The methylene chloride extract is dried with sodium sulfate and concentrated to a volume of 1.0 milliliter (mL). The extract is injected onto a Gas Chromatograph (GC) capillary column. The GC is temperature programmed to separate the semivolatile compounds, which are then detected with a Mass Spectrometer (MS).

2.2 Deuterated Monitoring Compounds (DMCs) and internal standards are added to all samples, standards, requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD) and blanks. The target compounds and DMCs are identified in the samples and blanks by analyzing standards that contain all target compounds, DMCs, and internal standards under the same conditions and comparing resultant mass spectra and GC retention times. A Relative Response Factor (RRF) is established for each target compound and DMC during the initial and continuing calibrations by comparing the mass spectra response from the Extracted Ion Current Profile (EICP) for the primary quantitation ion produced by that compound to the mass spectra response for the primary quantitation ion produced by the associated internal standard compound. Each identified target compound and DMC is quantitated by comparing the instrument response for the compound in the sample, standard, requested MS/MSD or blank with the instrument response of the associated internal standard, while taking into account the RRF from the most recent mid-point calibration, the sample volume, and any sample dilutions.

2.3 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later) or equivalent mass spectral library. Non-target compounds are quantitated by comparing the mass spectra response from the Reconstructed Ion Chromatogram (RIC) for the non-target compound peaks to the mass spectra response produced by the nearest internal standard. An RRF of 1 is assumed.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the Reconstructed Ion Chromatogram (RIC) profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. 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.

5.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. 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 data handling sheets should be made available to all personnel involved in these analyses. Specifically, concentrated sulfuric acid presents some hazards and is moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

6.0 EQUIPMENT AND SUPPLIES

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

6.1 Glassware

6.1.1 Continuous liquid-liquid extractors - Equipped with PTFE or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor-Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent) or Hydrophobic Membrane-based Extractor (Accelerated One Step™ Extractor, Corning series 3195 or equivalent).

6.1.2 Drying column - 19 millimeter (mm) ID chromatographic column with coarse frit (substitution of a small pad of Pyrex pre-extracted glass wool for the frit will prevent cross contamination of sample extracts).

6.1.3 Kuderna-Danish Apparatus

6.1.3.1 Concentrator tube - Kuderna-Danish, 10 milliliter (mL), graduated (Kontes, Vineland, NJ, K-570050-1025 or equivalent).

6.1.3.2 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001 -0500 or equivalent). Attach to concentrator tube with springs.

6.1.3.3 Snyder column - Kuderna-Danish, Three-ball macro (Kontes K-50300-0121 or equivalent).

6.1.3.4 Snyder column - Kuderna-Danish, Two-ball micro (Kontes K-569001-0219 or equivalent).

6.1.4 Vials - Amber glass, 2 mL capacity with PTFE-lined screw-cap.

6.1.5 Syringes - 0.2 mL, 0.5 mL, 10 mL volumes with Luerlock.

6.1.6 Micro-syringes - 10 microliter (µL) and larger, 0.006 inch (0.15 mm) ID needle.

6.2 Gases - Helium, Nitrogen, ultra pure grade.

6.3 Gas-line Tubing - Stainless steel or copper tubing.

6.4 Silicon Carbide Boiling Chips - Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride. PTFE boiling chips solvent rinsed prior to use are acceptable.

6.5 Water Bath - Heated, with concentric ring cover, capable of temperature control. To prevent the release of solvent fumes into the laboratory, the bath must be used in a hood.

6.6 Balance - Analytical, capable of accurately weighing ±0.0001 grams (g). The balances must be calibrated with class S weights or known reference weights once per each 12-hour workshift. The balances must be calibrated with class S weights at a minimum of once a month. The balances must also be annually checked by a certified technician.

6.7 Nitrogen Evaporation Device - Equipped with a water bath that can be maintained at 35°C to 40°C. To prevent the release of solvent fumes into the laboratory, the nitrogen evaporation device must be used in a hood. The N-Evap by Organomation Associates, Inc. South Berlin, MA (or equivalent) is suitable.

6.8 pH Meter - With a combination glass electrode, calibrated according to manufacturer's instructions. The pH meter shall be calibrated before each use.

6.9 pH Paper - Including narrow range capable of measuring a pH of 2.

6.10 Gas Chromatograph/Mass Spectrometer (GC/MS)

6.10.1 Gas Chromatograph - The gas chromatograph system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout the temperature program. The system must be suitable for splitless injection and have 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 are not to be used.

6.10.2 Gas Chromatography Column - Minimum length 30 meters (m) x 0.25 millimeter (mm) ID (or 0.32 mm) bonded-phase silicon coated fused silica capillary column DB-5 (J&W Scientific); RTx-5 (Restek); SPB-5 (Supelco); AT-5 (Alltech); HP-5 (Hewlett-Packard); CP-Sil 8CB (Chrompack); 007-2 (Quadrex); BP-5 (SGE); or equivalent. Note that this is a minimum requirement for column length. Longer columns may be used. Although a film thickness of 1.0 micron is recommended because of its larger capacity, a film thickness of 0.25 micron may be used. A description of the GC column used for analysis shall be provided in the SDG Narrative.

6.10.2.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants which interfere with the identification and quantitation of the compounds listed in Exhibit C (Semivolatiles).
- The analytical results generated using the column meet the initial and continuing calibration technical acceptance criteria listed in the SOW, and the Contract Required Quantitation Limits (CRQLs) listed in Exhibit C (Semivolatiles).
- The column can accept up to 120 nanograms (ng) of each compound listed in Exhibit C (Semivolatiles) without becoming overloaded.
- The column provides equal or better resolution of the compounds listed in Exhibit C (Semivolatiles) than the columns listed in Section 6.10.2.

6.10.2.2 As applicable, follow manufacturer's instructions for use of its product.

6.10.2.3 The Contractor must maintain documentation that the alternate column met the criteria in Section 6.10.2.1. The minimum documentation is as follows:

6.10.2.3.1 Manufacturer provided information concerning the performance characteristics of the column.

6.10.2.3.2 Reconstructed Ion Chromatograms (RICs) and data system reports generated on the GC/MS used for CLP analyses:

- From blanks which demonstrate that there are no contaminants which interfere with the semivolatile analysis when using the alternate column;
- For initial calibration standards analyzed using the alternate column;
- For continuing calibration standards analyzed using the alternate column.

6.10.2.4 Based on the Contractor generated data described in Section 6.10.2.3.2, the Contractor must complete a written comparison and review signed by the Laboratory Manager certifying that:

- The column performance meets the technical acceptance criteria in Sections 9.3.5 and 9.4.5.
- The low point initial calibration standard analysis has adequate sensitivity to meet the semivolatile CRQLs.
- The high point initial calibration standard analysis was not overloaded.
- The column does not introduce contaminants which interfere with the identification and/or quantitation of compounds listed in Exhibit C (Semivolatiles).

6.10.2.5 The documentation must be made available to USEPA during on-site laboratory evaluations or sent to USEPA upon request of the Contract Laboratory Program Project Officer (CLP PO) or the Organic Program Manager at Analytical Operations/Data Quality Center (AOC).

6.10.2.6 **PACKED COLUMNS CANNOT BE USED.**

6.10.3 Mass Spectrometer - The mass spectrometer must be capable of scanning from 35 to 500 atomic mass units (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 decafluorotriphenylphosphine (DFTPP) GC/MS performance check technical acceptance criteria (Table D-1) when 50 ng of 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 spectra while a sample compound elutes from the GC. The 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 outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.10.4 GC/MS interface - any GC/MS interface which provides acceptable sensitivity at CRQLs. GC/MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.10.5 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 any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). 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 NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent mass spectral library shall be used as the reference library. The operational data system must be able to flag all data files that have been edited manually by laboratory personnel.

6.10.6 Magnetic tape storage device - must be capable of recording data and suitable for long-term, off-line storage of GC/MS data.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1.1 Reagent water - defined as water in which no semivolatile target compound is observed at or above the Contract Required Quantitation Limit (CRQL) listed in Exhibit C for that compound and in which no non-target compound is observed at or above 10 micrograms per liter (µg/L).

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 grams (g) (1 lb) of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

7.1.1.2 Reagent water may be generated using a water purification system (Millipore Super-Q or equivalent).

7.1.2 Solvents - Acetone, methanol, methylene chloride, isooctane, 2-propanol, toluene. Pesticide quality or equivalent.

7.1.3 Sodium sulfate - (ACS) Granular or powdered, anhydrous (J.T. Baker anhydrous powder, catalog #73898, J.T. Baker anhydrous granular #3375, or equivalent). Purify by heating at 400°C for four hours in a shallow tray, cool in a desiccator, and store in a glass bottle. CAUTION: An open container of sodium sulfate may become contaminated during storage in laboratory.

7.1.4 Sulfuric acid solution (1:1) - slowly add 50 milliliters (mL) of concentrated H_2SO_4 (Sp. Gr. 1.84; 36N) to 50 mL of reagent water.

7.2 Standards

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

7.2.1 Stock Standard Solutions

Stock standard solutions may be purchased or prepared using the following procedure.

7.2.1.1 Accurately weigh about 0.0100 g of pure material. Dissolve the material in methylene chloride or another suitable solvent and dilute to volume in a 10 mL volumetric flask. Larger volumes may be used at the convenience of the analyst.

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

7.2.1.3 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.

7.2.2 Secondary Dilution Standards

7.2.2.1 Using stock standards, prepare secondary dilution standards in methylene chloride that contain the compounds of interest either singly or mixed together.

7.2.2.2 Fresh secondary dilution standards must be prepared once every twelve months, or sooner, if standards have degraded or concentrated. Secondary dilution standards must be checked for signs of degradation or concentration just prior to preparing working standards from them.

7.2.3 Working Standards

7.2.3.1 Deuterated Monitoring Compound (DMC) Standard Spiking Solution

7.2.3.1.1 Prepare a DMC standard spiking solution that contains the following compounds at concentrations shown in methanol:

DMC	Concentration • g/mL
Phenol-d5	40
bis-(2-Chloroethyl)ether-d8	40
2-Chlorophenol-d4	40
4-Methylphenol-d8	40
Nitrobenzene-d5	40
2-Nitrophenol-d4	40
2,4-Dichlorophenol-d3	40
4-Chloroaniline-d4	40
Dimethylphthalate-d6	40
Acenaphthylene-d8	40
4-Nitrophenol-d4	40
Fluorene-d10	40
4,6-Dinitro-methylphenol-d2	40
Anthracene-d10	40
Pyrene-d10	40
Benzo(a)pyrene-d12	40

7.2.3.1.2 DMC standards are added to all samples, blanks, requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), and calibration solutions. The DMC standard spiking solution must be prepared every twelve months or sooner if the solution has degraded or concentrated.

7.2.3.2 Matrix Spiking Solution

7.2.3.2.1 The matrix spiking solution consists of the following:

Bases/Neutrals	Acids
Acenaphthene	Pentachlorophenol
2,4-Dinitrotoluene	Phenol
Pyrene	2-Chlorophenol
N-Nitroso-di-n-propylamine	4-Chloro-3-methylphenol
	4-Nitrophenol

7.2.3.2.2 Prepare a spiking solution that contains each of the base/neutral compounds above at 20 micrograms per milliliter (• g/mL) in methanol and the acid compounds at 80 • g/mL in methanol.

7.2.3.2.3 The matrix spiking solution must be prepared every twelve months or sooner if the solution has degraded or concentrated.

7.2.3.3 Instrument Performance Check Solution--DFTPP

Prepare a 50 nanograms per microliter (ng/µL) solution of decafluorotriphenylphosphine (DFTPP) in methylene chloride. The DFTPP solution must be prepared fresh once every twelve months or sooner if the solution has degraded or concentrated.

7.2.3.4 Initial and Continuing Calibration Solutions

7.2.3.4.1 Five initial calibration standard solutions are required for all target compounds and DMCs. Standard concentrations of 5, 10, 20, 50, and 80 ng/µL are required for the DMCs and all but seven of the target compounds. The seven compounds: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, and 4,6-dinitro-2-methylphenol require calibration at 20, 50, 80, 100, and 120 ng/µL.

7.2.3.4.2 To prepare a calibration standard solution, add an appropriate volume of secondary dilution standard to methylene chloride in a volumetric flask. Dilute to volume with methylene chloride.

7.2.3.4.3 The 20 ng/µL initial calibration solution (80 ng/µL for the seven compounds listed in Section 7.2.3.4.1) is the continuing calibration solution.

7.2.3.4.4 The five initial calibration solutions must be prepared fresh before use. The continuing calibration standard solution must be prepared weekly or sooner if the solution has degraded or concentrated.

7.2.3.5 Internal Standard Spiking Solution

7.2.3.5.1 Prepare an internal standard spiking solution in methylene chloride or another suitable solvent that contains 1,4 dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} at 2000 ng/µL. It may be necessary to use 5 to 10 percent toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents.

NOTE: For automated systems using an injection volume of less than 10 µL, the internal standard solution may need to be prepared at a different concentration.

7.2.3.5.2 The internal standard spiking solution must be prepared every twelve months or sooner if the solution has degraded or concentrated.

7.2.4 Ampulated Standard Extracts

Standard solutions purchased from a chemical supply house as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor which are immediately ampulated in glass vials may be retained for 2 years from preparation date. Upon breaking the glass seal, the expiration times

listed in Sections 7.2.1 to 7.2.3.5 will apply. The Contractor is responsible for assuring that the integrity of the standards has not degraded (Section 7.2.5.5).

7.2.5 Storage of Standard Solutions

7.2.5.1 Store the stock and secondary standard solutions at 4°C (±2°C) in PTFE-lined screw-cap amber bottles.

7.2.5.2 Store the working standard solutions at 4°C (±2°C) in PTFE-lined screw-cap amber bottles.

7.2.5.3 Protect all standards from light.

7.2.5.4 Samples, sample extracts, and standards must be stored separately.

7.2.5.5 The Contractor is responsible for maintaining the integrity of standard solutions and verifying prior to use. Storage of standard solutions in the freezer may cause some compounds to precipitate. This means at the minimum, the standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in solution. Additional steps may be necessary to ensure all components are in solution.

7.2.6 Temperature Records for Storage of Standards

7.2.6.1 The temperature of all standard storage refrigerators shall be recorded daily.

7.2.6.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.6.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in 1 liter (L) (or 1 quart) amber glass containers and fitted with screw-caps lined with PTFE. If amber containers are not available, the samples should be protected from light. The specific requirements for site sample collection are outlined by the Region.

8.1.2 All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until extraction.

8.2 Procedure for Sample Storage

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

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3 Procedure for Sample Extract Storage

8.3.1 Sample extracts must be protected from light and stored at less than 4°C (±2°C) until 365 days after delivery of a reconciled, complete data package to USEPA.

8.3.2 Samples, sample extracts, and standards must be stored separately.

8.4 Records for Sample and Sample Extract Storage

8.4.1 The temperature of all sample and sample extract storage refrigerators shall be recorded daily.

8.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

8.4.3 Corrective action SOPs shall be posted on the refrigerators.

8.5 Contract Required Holding Times

8.5.1 Extraction of water samples by continuous liquid-liquid procedures shall be started within 5 days of Validated Time of Sample Receipt (VTSR).

NOTE: Separatory funnel extraction procedures are not permitted.

8.5.2 As part of USEPA's QA program, USEPA may provide Performance Evaluation (PE) samples as standard extracts which the Contractor is required to prepare per the instructions provided by USEPA. PE samples must be extracted and analyzed concurrently with the samples in the SDG. The extraction holding time (5 days after VTSR) does not apply to PE samples received as standard extracts.

8.5.3 Extracts of water samples must be analyzed within 40 days following extraction.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Conditions

9.1.1 Gas Chromatograph (GC)

9.1.1.1 The following are the gas chromatographic analytical conditions. The conditions are recommended unless otherwise noted.

Initial Column Temperature Hold	40°C for 4 minutes
Column Temperature Program	40-270°C at 10°C/min.
Final Column Temperature Hold	270°C for 3 minutes after all compounds listed in Exhibit C (Semivolatiles) have eluted (required)
Injector Temperature	250-300°C
Transfer Line Temperature	250-300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, splitless
Sample Volume	1 • L
Carrier Gas	Helium at 30 cm/sec

9.1.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, matrix spikes, and matrix spike duplicates, if required.

9.1.2 Mass Spectrometer (MS)

The following are the required mass spectrometer analytical conditions:

Electron Energy	70 volts (nominal)
Mass Range	35 to 500 amu
Scan Time	Not to exceed 1 second per scan
Ionization Mode	EI

9.2 Instrument Performance Check (DFTPP)

9.2.1 Summary of Instrument Performance Check

The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as perfluoro-tri-n-butylamine (FC-43) or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Section 7.2.3.3). Prior to the analysis of any samples (including requested Matrix Spike/Matrix Spike Duplicate (MS/MSD) and PE Samples) blanks and calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing decafluoro-triphenylphosphine (DFTPP).

9.2.2 Frequency of Instrument Performance Check

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

9.2.2.2 The 12-hour time period for a instrument performance check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after 12-hours have elapsed according to the system clock.

9.2.3 Procedure for Instrument Performance Check

The analysis of the instrument performance check solution may be performed as an injection of up to 50 nanograms (ng) of DFTPP into the GC/MS or by adding 50 ng of DFTPP to a calibration standard (Section 7.2.3.4.3) and analyzing the calibration standard.

9.2.4 Technical Acceptance Criteria for Instrument Performance Check

9.2.4.1 The GC/MS system must be tuned at the frequency described in Section 9.2.2.

9.2.4.2 The abundance criteria listed in Table D-1 must be met for a 50 ng injection of DFTPP. The mass spectrum of DFTPP 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 acquired no more than 20 scans prior to the beginning of the elution of DFTPP. Do not subtract part of the DFTPP peak.

NOTE: All subsequent standards, samples, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

9.2.5 Corrective Action for Instrument Performance Check

9.2.5.1 If the GC/MS performance check technical acceptance criteria are not met, re-tune the GC/MS system. It may be necessary to clean the ion source, clean quadrupoles, or take other actions to achieve the technical acceptance criteria.

9.2.5.2 GC/MS performance check technical acceptance criteria MUST be met before any standards, samples, and required blanks are analyzed. Any standards, samples, and required blanks analyzed when GC/MS performance check technical acceptance criteria have not been met will require re-analysis at no additional cost to USEPA.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to the analysis of samples and required blanks and after GC/MS performance check technical acceptance criteria have been met, each GC/MS system must be initially calibrated at a minimum of five concentrations (Section 7.2.3.4) to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and Deuterated Monitoring Compounds (DMCs).

9.3.2 Frequency of Initial Calibration

9.3.2.1 Each GC/MS system must be initially 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 technical acceptance criteria have not been met.

9.3.2.2 If time still remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed. It is not necessary to analyze a continuing calibration standard within this 12-hour time period, if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration technical acceptance criteria. Quantitate all sample and blank results against the initial calibration standard that is the same concentration as the continuing calibration standard (Section 7.2.3.4.3). Compare quality control criteria such as internal standard area response change and retention time shift to the initial calibration standard that is the same concentration as the continuing calibration standard.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Set-up the GC/MS system per the requirements of Section 9.1.

9.3.3.2 All standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before preparation or analysis.

9.3.3.3 Prepare five calibration standards containing all the semivolatile target and DMCs at the concentrations described in Section 7.2.3.4.1.

9.3.3.4 Add 10 microliters (µL) of the internal standard spiking solution (Section 7.2.3.5) to 1.0 milliliters (mL) of each of the five calibration standards for a concentration of 20 nanograms per microliter (ng/µL) for each internal standard compound.

9.3.3.5 Tune the GC/MS system to meet the technical acceptance criteria in Section 9.2.4 for DFTPP.

9.3.3.6 Analyze each calibration standard by injecting 1.0 µL of standard.

9.3.4 Calculations for Initial Calibrations

9.3.4.1 Calculate Relative Response Factors (RRF) for each semivolatile target compound and DMC using Equation 1. See Table D-2 to associate semivolatile target and deuterated monitoring compounds with the proper internal standard. See Table D-3 for primary quantitation ions to be used for each semivolatile target compound, DMC, and internal standard.

NOTE: Unless otherwise stated the area response is that of the primary quantitation ion.

EQ. 1

$$RRF = \frac{A_x}{A_{is}} * \frac{C_{is}}{C_x}$$

Where:

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 (Table D-2).

C_{is} = Amount of the internal standard injected (µg).

C_x = Amount of the compound to be measured injected (µg).

9.3.4.2 Calculate the mean Relative Response Factor for each compound using Equation 2.

EQ. 2

$$\bar{x} = \frac{\sum_{i=1} x_i}{n}$$

Where:

x_i = Each individual value.

• = The mean of n values.

n = The total number of values.

9.3.4.3 Calculate the percent relative standard deviation (%RSD) of Relative Response Factor (RRF) values for each semivolatile target compound and DMC over the initial calibration range using Equation 3 in conjunction with Equation 4.

EQ. 3

$$\%RSD = \frac{(\text{Standard Deviation})}{\text{Mean}} \times 100$$

Where:

%RSD = Percent relative standard deviation.

9.3.4.4 Equation 4 is the general formula for standard deviation for a statistically small set of values.

EQ. 4

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1} (x_i - \bar{x})^2}{n-1}}$$

Where:

x_i, • and n are as defined in Equation 2.

9.3.5 Technical Acceptance Criteria for Initial Calibration

9.3.5.1 All initial calibration standards must be analyzed at the concentration levels described in Section 7.2.3.4.1 and at the frequency described in Section 9.3.2 on a GC/MS system meeting the DFTPP technical acceptance criteria.

9.3.5.2 The relative response factor (RRF_i) at each calibration concentration for each semivolatile target compound and DMC must be greater than or equal to the compound's minimum acceptable relative response factor listed in Table D-4.

9.3.5.3 The %RSD over the initial calibration range for relative response factor for each semivolatile target compound that has a required %RSD must be less than or equal to the %RSD listed in Table D-4.

9.3.5.4 Up to four compounds may fail the criteria listed in Sections 9.3.5.2 and 9.3.5.3 and still meet the minimum RRF and %RSD requirements. However, these four compounds must have a minimum RRF greater than 0.010 and %RSD less than or equal to 40.0%.

9.3.5.5 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument manual to determine how saturation is indicated for your instrument.

9.3.6 Corrective Action for Initial Calibrations

9.3.6.1 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 technical acceptance criteria.

9.3.6.2 Initial calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed. Any samples or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require re-analysis at no additional cost to USEPA.

9.4 Continuing Calibration

9.4.1 Summary of Continuing Calibration

Prior to the analysis of samples and required blanks and after GC/MS performance check technical acceptance criteria and initial calibration technical acceptance criteria have been met, 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 contains all the semivolatile target compounds, DMCs, and internal standards.

9.4.2 Frequency of Continuing Calibration

9.4.2.1 Each GC/MS used for analysis must be calibrated once every twelve (12) hour time period of operation. The 12-hour time period begins with the injection of DFTPP.

9.4.2.2 If time still remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed. It is not necessary to analyze a continuing calibration standard within this 12-hour time period, if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration technical acceptance criteria. Quantitate all sample and blank results against the 20 ng/µL (80 ng/µL for the seven compounds listed in Section 7.2.3.4.1) calibration standard.

9.4.3 Procedure for Continuing Calibration

9.4.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before preparation or analysis.

9.4.3.2 Add 10 µL of the internal standard solution (Section 7.2.3.5) to 1.0 mL of the continuing calibration standard (Section 7.2.3.4.3) for a concentration of 20 ng/µL for each internal standard compound.

9.4.3.3 Analyze the continuing calibration standard by injecting 1.0 µL of standard.

9.4.4 Calculations for Continuing Calibration

9.4.4.1 Calculate a RRF for each semivolatile target compound and DMC using Equation 1 for the primary characteristic ions found in Table D-3.

9.4.4.2 Calculate the percent difference between the mean relative response factor from the most recent initial calibration and the continuing calibration relative response factor for each semivolatile target compound and DMC using Equation 5. For internal standards, use the primary ions listed in Table D-3 unless interferences are present. If interferences prevent the use of the primary ion for a given internal standard, use the secondary ion(s) listed in Table D-3.

EQ. 5

$$\%Difference_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} * 100$$

Where:

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria.

RRF_c = Relative response factor from continuing calibration standard.

9.4.5 Technical Acceptance Criteria for Continuing Calibration

9.4.5.1 The continuing calibration standard must be analyzed at the 20 ng/µL (80 ng/µL for the seven compounds listed in 7.2.3.4.1) concentration level, and at the frequency described in Section 9.4.2, on a GC/MS system meeting the DFTPP and the initial calibration technical acceptance criteria.

9.4.5.2 The relative response factor for each semivolatile target compound and DMC must be greater than or equal to the compound's minimum acceptable relative response factor listed in Table D-4.

9.4.5.3 The relative response factor percent difference for each semivolatile target compound that has a percent difference criteria must be within the inclusive range listed in Table D-4.

9.4.5.4 Up to four compounds may fail the requirements listed in Sections 9.4.5.2 and 9.4.5.3 and still meet the minimum relative response factor and percent difference criteria. However, these compounds must have a minimum relative response factor greater than or equal to 0.010 and the percent difference must be within the inclusive range of ±40%.

9.4.5.6 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.4.6 Corrective Action for Continuing Calibration

9.4.6.1 If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3.3. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the technical acceptance criteria.

9.4.6.2 Continuing calibration technical acceptance criteria MUST be met before any samples (including requested MS/MSD) or required blanks are analyzed. Any samples or required blanks analyzed when continuing calibration criteria have not been met will require re-analysis at no additional cost to USEPA.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 This method is designed for analysis of water samples that contain low concentrations of the semivolatile compounds listed in Exhibit C. The majority of the samples are expected to come from drinking water and well/ground water type sources around Superfund sites. If, upon inspection of a sample, the Contractor suspects that the sample is not amenable to this method, contact Sample Management Office (SMO). SMO will contact the Region for instructions.

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

10.1.3 Extraction of Sample

10.1.3.1 Allow the sample to come to ambient temperature (approximately 1 hour).

10.1.3.2 Continuous liquid-liquid extraction is used to extract the samples. Separatory funnel extraction cannot be used.

10.1.3.3 Continuous Liquid-Liquid Extraction Without Hydrophobic Membrane

10.1.3.3.1 Follow manufacturer's instructions for set-up.

10.1.3.3.2 Add methylene chloride to the bottom of the extractor and fill it to a depth of at least one inch above the bottom sidearm.

10.1.3.3.3 Measure out a 1 L sample aliquot in a separate, clean graduated cylinder; transfer the aliquot to the continuous extractor. Measure and record the initial pH of the sample with a pH meter or narrow range pH paper. Adjust the pH to **2.0** with 1:1 H_2SO_4 and record the final pH.

NOTE: With some samples, it may be necessary to place a layer of glass wool between the methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.3.3.4 Using a syringe or volumetric pipet, add 1.0 mL of the Deuterated Monitoring Compound (DMC) standard spiking solution (Section 7.2.3.1) into the sample and mix well.

10.1.3.3.5 Rinse the graduated cylinder with 50 mL of methylene chloride and transfer the rinsate to the continuous extractor. If the sample container is empty, rinse the container with 50 mL of methylene chloride and add rinsate to the continuous extractor.

10.1.3.3.6 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5 to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 18 hours.

NOTE 1: When a minimum drip rate of 10-15 mLs/min. is maintained throughout the extraction, the extraction time may be reduced to a minimum of twelve hours. Allow to cool, then detach the distillation flask. Proceed to Section 10.1.4.

NOTE 2: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.3.4 Continuous Liquid-Liquid Extraction With Hydrophobic Membrane

10.1.3.4.1 Follow the manufacturer's instructions for set-up..

10.1.3.4.2 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder. If the sample container is empty, rinse the container with 50 mL of methylene chloride and add the rinsate to the continuous extractor. If the sample container is not empty, add 50 mL of methylene chloride to the continuous extractor. Slowly transfer the aliquot to the continuous extractor. Measure and record the initial pH of the sample with a pH meter or a narrow range pH paper. Adjust the pH to **2.0** with 1:1 H_2SO_4 and record the final pH.

10.1.3.4.3 Using a syringe or volumetric pipet, add 1.0 mL of the DMC standard spiking solution (Section 7.2.3.1) into the sample and mix well.

10.1.3.4.4 Rinse the graduated cylinder with 50 mL of methylene chloride and transfer the rinsate to the continuous extractor.

10.1.3.4.5 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours.

NOTE 1: Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion which will consume the solvent volume, preventing the efficient extraction of the sample. When this occurs, add additional solvent to assure efficient extraction of the sample, and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used.

Allow to cool, then detach the distillation flask. Proceed to Section 10.1.4.

NOTE 2: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane type extractor, it may not be necessary to dry the extract with sodium sulfate.

NOTE 3: If low DMC recoveries occur, assure 1) the apparatus was properly assembled to prevent leaks; 2) the drip rate/solvent cycling was optimized; and 3) there was proper cooling for condensation of solvent.

NOTE 4: Alternate continuous liquid-liquid extractor types that meet the requirements of the SOW may also be used. If using alternate extractors or design types, follow the manufacturer's instructions for set-up.

10.1.4 Concentrating the Sample Extract

10.1.4.1 Assemble a Kuderna-Danish (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 semivolatile target compounds listed in Exhibit C.

10.1.4.2 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 a K-D concentrator. Rinse the distilling flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.1.4.3 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 methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60°C to 80°C recommended) 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 10 to 15 minutes. 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 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATIVE FLASK TO GO DRY. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5 mL syringe is recommended for this operation.

10.1.4.4 Two different types of concentration techniques are permitted to obtain the final 1.0 mL volume: micro Snyder column and nitrogen evaporation techniques.

10.1.4.4.1 Micro Snyder Column Technique

Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Pre-wet the Snyder column by adding about 0.5 mL of methylene chloride to the top of the column. Place the K-D apparatus in a hot water bath (60°C to 80°C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. 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 about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. DO NOT LET THE EXTRACT GO DRY. Remove the Snyder column and rinse the evaporative flask and its lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL with methylene chloride. Transfer the extract to a PTFE-sealed screw-cap bottle, label the bottle and store at 4°C (±2°C).

10.1.4.4.2 Nitrogen Evaporation Technique (taken from ASTM Method D3086)

Place the concentrator tube in a warm water bath (30°C 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. Caution: Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or PTFE tubing. New plastic tubing must not be used between the carbon trap and the sample since it may introduce interferences. The internal wall of the concentrator tube must be rinsed down several times with methylene chloride during the operation and the final volume brought to 1.0 mL with methylene chloride. 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. Transfer the extract to a PTFE-sealed screw-cap bottle, label the bottle and store at 4°C (±2°C).

10.2 Instrument Analysis of Sample

10.2.1 Set up the Gas Chromatograph/ Mass Spectrometer (GC/MS) system per the requirements of Section 9.1. Before samples or required blanks can be analyzed, the instrument must meet the decafluoro-triphenylphosphine (DFTPP), initial calibration, and continuing calibration technical acceptance criteria. All sample, blank extracts, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis. All sample extracts [including requested Matrix Spike/Matrix Spike Duplicate (MS/MSD)] and required blanks must be analyzed under the same instrumental conditions as the calibration standards.

10.2.2 Add 10.0 µL of the internal standard spiking solution (Section 7.2.3.5) to the 1.0 mL extract. For sample dilutions, add an appropriate amount of the internal standard spiking solution to maintain a 20 nanograms per microliter (ng/µL) concentration of the internal standards in the diluted extract.

NOTE: An alternate amount of internal standard solution may be added, however the internal standards must be added to maintain the required 20 ng/µL of each internal standard in the sample extract.

10.2.3 Inject 1.0 µL of sample extract into the GC/MS, and start data acquisition.

10.2.4 Three minutes after 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 Extracted Ion Current Profiles (EICPs).

10.2.5 Sample Dilutions

An original undiluted analysis must be made and results reported for all samples.

10.2.5.1 When a sample extract is analyzed that has a semivolatile target compound concentration greater than the upper limit of the initial calibration range or in which ions from a target compound saturate the detector (excluding the compound peaks in the solvent front), the extract must be diluted, the internal standard concentration must be readjusted, and the sample extract must be re-analyzed. Secondary ion quantitation is only allowed when there are sample

interferences with the primary quantitation ion, not when saturation occurs. If secondary ion quantitation is used, calculate a relative response factor using the area response (EICP) 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.

NOTE: If the laboratory has evidence or highly suspects, because of sample color or other physical property, that a sample may contain extremely high concentrations of either target or non-target compounds, then SMO shall be immediately contacted. SMO will seek regional recommendations for diluted analysis.

10.2.5.2 Dilute the sample using the following procedure:

10.2.5.2.1 Calculate the sample dilution necessary to keep the semivolatile target compounds that required dilution above the mid-point standard in the initial calibration range and so that no target compound has ions which saturate the detector (excluding the compound peaks in the solvent front).

10.2.5.2.2 Dilute the sample extract quantitatively with methylene chloride.

10.2.5.2.3 Analyze the sample dilution per Section 10.2, including the addition of internal standards to maintain a 20 ng/µL concentration of the internal standards (Section 10.2.2).

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification of Target Compounds

11.1.1 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 analyte within Gas Chromatograph (GC) Relative Retention Time (RRT) unit window established from the 12-hour calibration standard.
- Correspondence of the sample analyte and calibration standard component mass spectra.

11.1.2 For establishing correspondence of the GC RRT, the sample component RRT must be within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. For samples analyzed during the same 12-hour time period as the initial calibration standards, compare the analyte retention times to those from the 20 nanograms per microliter (ng/µL) (80 ng/µL for the seven compounds listed in Section 7.2.3.4.1) calibration standard. If coelution of interfering compounds prohibits accurate assignment of the sample component RRT from the Extracted Ion Current Profile (EICP) for the primary ion, the RRT must be assigned by using the total ion chromatogram.

11.1.3 For comparison of standard and sample component 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 decafluorotriphenylphosphine (DFTPP) technical acceptance criteria. These standard spectra may be obtained from the analysis used to obtain reference RRTs.

11.1.4 The requirements for qualitative verification by comparison of mass spectra are as follows:

11.1.4.1 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.

11.1.4.2 The relative intensities of the major ions specified in Section 11.1.4.1 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.)

11.1.4.3 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. When target compounds are below Contract Required Quantitation Limits (CRQLs) but the spectrum meets the identification criteria, report the concentration with a "J". For example, if the CRQL is 5.0 micrograms per liter (µg/L) and a concentration of 3.0 µg/L is calculated, report the data as "3.0J".

11.1.5 If a compound cannot be verified by all of the criteria in Sections 11.1.1 through 11.1.4, 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 quantitation.

11.2 Qualitative Identification of Non-Target Compounds

11.2.1 A library search shall be executed for non-target compounds for the purpose of tentative identification. The NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent mass spectral library shall be used as the reference library (Section 6.10.5).

11.2.2 Up to 30 non-DMC/non-internal standard organic compounds of greatest apparent concentration not listed in Exhibit C for the volatiles and semivolatiles shall be tentatively identified via a forward search of the NIST/EPA/NIH and/or Wiley mass spectral library, or equivalent mass spectral library. The following are not to be reported:

- Compounds with responses less than 10 percent of the internal standard (as determined by inspection of peak areas or heights);
- Compounds which elute earlier than 30 seconds before the first semivolatile compound listed in Exhibit C (Semivolatiles) or three minutes after the last semivolatile compound listed in Exhibit C (Semivolatile) has eluted; and
- Volatile compounds listed in Exhibit C. Only after visual comparison of sample spectra to spectra resulting from the library search(es) will the mass spectral interpretation specialist assign a tentative identification.

NOTE: Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

11.2.3 Up to 20 peaks of greatest apparent concentration (as determined by inspection of peak areas or heights) that are suspected to be straight-chain, branched, or cyclic alkanes, alone or part of an alkane series shall be library searched. When the above alkanes are tentatively identified, the concentration(s) are to be estimated as described in Section 11.4 and reported in the SDG Narrative as alkanes, by class (i.e., straight chain, branched, or cyclic; as a series; as applicable). Alkanes are not counted as part of the 30 organic compounds described in Section 11.2.2.

11.2.4 Peaks that are suspected to be aldol-condensation reaction products (e.g., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be searched, reported, and counted as part of the 30 most intense non-target semivolatile compounds, and qualified with an "A" flag on Form I LCSV-TIC.

11.2.5 Guidelines for making Tentative Identification

11.2.5.1 Major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

11.2.5.2 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.)

11.2.5.3 Molecular ions present in reference spectrum should be present in sample spectrum.

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

11.2.5.5 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.

NOTE: Data system library reduction programs can sometimes create these discrepancies.

11.2.5.6 Non-target compounds receiving a library search match of 85% or higher should be considered a "probable match". The compound should be reported unless the mass spectral interpretation specialist feels there is just evidence not to report the compound as identified by the library search program. The lab should include in the SDG Narrative the justification for not reporting a compound as listed by the search program.

11.2.5.7 If the library search produces more than one compound at or above 85%, report the compound with the highest percent match (report first compound if percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels that the highest match compound should not be reported, or another compound with a lower match should be reported. The lab should include in the Sample Delivery Group (SDG) Narrative the justification for not reporting the compound with the highest spectral match.

11.2.5.8 If the library search produces a series of obvious isomer compounds with library search matches greater than 85% (e.g., tetramethyl naphthalenes), the compound with the highest library search percent match should be reported (or first compound if library search matches are the same). A note should be placed in the SDG Narrative indicating the exact isomer configuration as reported may not be accurate.

11.2.5.9 If library search matches of less than 85% are produced and in the technical judgement 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 (e.g., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If a probable molecular weight can be distinguished, include it.

11.2.5.10 The Contractor shall report pesticide target compounds listed in Exhibit C (Pesticides) that appear as semivolatile tentatively identified compounds.

11.3 Calculations for Target Compounds

11.3.1 Target compounds identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (Table D-2). The Relative

Response Factor (RRF) from the continuing calibration analysis is used to calculate the concentration in the sample. For samples analyzed during the same 12-hour time period as the initial calibration standards, use the RRF values from the mid-point initial calibration standard.

11.3.2 Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reason in the SDG Narrative. The area of a secondary ion cannot be used for the area of the primary ion unless a relative response factor is calculated using the secondary ion.

NOTE: Unless otherwise stated, the area response is from the EICP of the primary quantitation ion. The primary quantitation ions for the target compounds, internal standards, and DMCs are listed in Table D-3.

11.3.3 Calculate target compound concentrations using Equation 6.

EQ. 6

$$\text{Concentration } \mu g/L = \frac{(A_x)(I_s)(V_o)(Df)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where:

A_x = Area response (EICP) of the characteristic ion for the compound to be measured.

A_{is} = Area response (EICP) of the characteristic ion for the internal standard. The target compounds are listed with their associated internal standard in Table D-2.

I_s = Amount of internal standard injected in nanograms (ng).

RRF = Relative response factor from the most recent continuing calibration as determined in Section 9.4.

V_o = Volume of water extracted in milliliters (mL).

V_i = Volume of extract injected in microliters (µL).

V_t = Volume of concentrated extract in microliters (µL). (V_t = 1000 µL)

Df = Dilution Factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\text{µL most conc. extract used to make dilution + µL clean solvent}}{\text{µL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

11.3.4 When a target compound concentration is below the CRQL, but the spectra meet the identification criteria, report the concentration with a "J". For example, if the CRQL is 5.0 µg/L and a concentration of 3.0 µg/L is calculated, report as "3.0 J". Report ALL sample concentration data as UNCORRECTED for blanks.

11.3.5 Calculate the adjusted CRQL for semivolatile compounds using Equation 7.

NOTE: If the adjusted CRQL is less than the CRQL listed in Exhibit C (Semivolatiles), report the CRQL listed in Exhibit C (Semivolatiles).

EQ. 7

$$\text{Adjusted CRQL} = \text{Contract CRQL} * \frac{\text{Contract Sample Vol (1000 mL)}}{V_o} * Df * \frac{V_t}{\text{Contract Ext. Vol (1000 uL)}} * \frac{\text{Contract Injection Vol (1 uL)}}{V_i}$$

Where:

Contract CRQL = CRQL values reported in Exhibit C of the SOW.

V_o = Same as EQ. 6.

V_i = Same as EQ. 6.

V_t = Same as EQ. 6.

Df = Same as EQ. 6.

11.3.6 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitations. This normally occurs when there is compound co-elution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific TCL, DMC, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instance of manual integration must be documented in the SDG Narrative.

11.3.7 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator must identify such edits or manual procedures by initialing and dating the changes made to the report and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and DMCs.

11.3.8 The requirements listed in 11.3.4 and 11.3.5 apply to all standards, samples [including requested Matrix Spike/Matrix Spike Duplicate (MS/MSD)], and blanks.

11.3.9 Internal Standard Responses and Retention Times

Internal standard responses and retention times in all samples and blanks must be evaluated during or immediately after data acquisition. Compare the sample/blank internal standard responses

and retention times to the continuing calibration internal standard responses and retention times. For samples and blanks analyzed during the same 12-hour time period as the initial calibration standards, compare the internal standard responses and retention times against the 20 ng/µL (80 ng/µL for the seven compounds listed in Section 7.2.3.4.1) calibration standard. The EICP of the internal standards must be monitored and evaluated for each sample and blank.

11.4 Calculations for Non-Target Compounds

Equation 6 is used for calculating the concentrations of the non-target compounds. Total area counts (or peak heights) from the Reconstructed Ion Chromatograms (RICs) 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 RRF of one (1) is to be assumed. The value from this quantitation shall be qualified as "J" (estimated due to lack of a compound-specific relative response factor), and "N" (presumptive evidence of presence), indicating the qualitative and quantitative uncertainties associated with this non-target compound. This estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns.

11.5 Calculations for Deuterated Monitoring Compounds (DMCs)

11.5.1 Calculate the concentration of the DMCs using the same equation as used for the target compounds.

11.5.2 Calculate the DMC percent recovery in all samples and blanks using Equation 8. Determine if recovery is within limits (Table D-5) and report on appropriate form.

EQ. 8

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} * 100$$

Where:

Q_d = Concentration or amount determined by analysis.

Q_a = Concentration or amount added to sample/blank.

11.6 Technical Acceptance Criteria for Sample Analysis

11.6.1 The sample must be analyzed on a GC/MS system meeting the GC/MS performance check, initial calibration, and continuing calibration technical acceptance criteria.

11.6.2 The sample must be extracted and analyzed within the contract holding times.

11.6.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

11.6.4 The percent recovery for the DMCs in the sample must be within the acceptance windows listed in Table D-5. Up to four DMCs per sample may fail to meet the recovery limits listed in Table D-5.

NOTE: The DMC recovery requirements do not apply to a sample that has been diluted.

11.6.5 The instrumental area response (EICP area) for each of the internal standards in the sample must be within the inclusive range of -50 percent and +100 percent of its response in the most recent continuing calibration standard analysis.

11.6.6 The retention time shift for each of the internal standards in the sample must be within ±0.33 minutes (20.0 seconds) of its retention time in the most recent continuing calibration standard analysis.

11.6.7 The RRT of each DMC in the sample must be within ±0.06 RRT units of its relative retention time in the most recent continuing calibration standard analysis.

11.6.8 Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the initial calibration range unless a more dilute aliquot of the sample extract is also analyzed according to the procedures in Section 10.2.5.

11.7 Corrective Action for Sample Analysis

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

11.7.2 If the Contractor needs to analyze more than one (1) sample dilution other than the original analysis to have all the target compounds within the initial calibration range and to have no ions saturating the detector (excluding the peaks in the solvent front), contact Sample Management Office (SMO). SMO will contact the Region for instructions.

11.7.3 Corrective actions for failure to meet instrument performance checks, initial calibration, continuing calibration and method blanks must be completed before the analysis of samples.

11.7.4 Sample analysis technical acceptance criteria MUST be met before data is 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 re-extraction and/or re-analysis at no additional cost to USEPA.

11.7.5 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 USEPA Contract Laboratory Program Project Officer (CLP PO). Send a copy of the SDG Narrative (including your contract number), a description of the situation, and the requested action to the CLP PO.

12.0 QUALITY CONTROL

12.1 Method Blank

12.1.1 Summary of Method Blank

A method blank is 1.0 liter (L) of reagent water carried through the entire analytical scheme. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.2 Frequency of Method Blank

12.1.2.1 A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples (excluding Matrix Spike/Matrix Spike Duplicate(s) MS/MSD, if required, and Performance Evaluation (PE) samples).

12.1.2.2 Each method blank must be analyzed on each Gas Chromatograph/Mass Spectrometer (GC/MS) system used to analyze the samples prepared with the method blank.

12.1.3 Procedure for Method Blank

Measure out 1.0 L of reagent water for each method blank aliquot. Extract, concentrate and analyze the method blank at the same time as the samples associated with the blank according to Section 10.

12.1.4 Calculations for Method Blank

Perform data analysis and calculations according to Section 11.

12.1.5 Technical Acceptance Criteria For Method Blank

12.1.5.1 All blanks must be analyzed at the frequency described in Section 12.1.2 on a GC/MS system meeting the GC/MS performance check, initial calibration, and continuing calibration technical acceptance criteria.

12.1.5.2 The percent recovery for each of the Deuterated Monitoring Compounds (DMCs) in the blank must be within the acceptance windows listed in Table D-5.

12.1.5.3 The area response for each of the internal standards in the blank must be within the inclusive range of -50 percent and +100 percent of its response in the most recent continuing calibration standard analysis.

12.1.5.4 The retention time shift for each of the internal standards in the blank must be within ±0.33 minutes (20.0 seconds) of its retention time in the most recent continuing calibration standard analysis.

12.1.5.5 The Relative Retention Time (RRT) of each DMC in the blank must be within ±0.06 RRT units of its relative retention time in the most recent continuing calibration standard analysis.

12.1.5.6 The concentration of all target compounds (except the phthalate esters listed in Exhibit C) in the blanks must be less than the Contract Required Quantitation Limit (CRQL) for each target compound. A method blank for semivolatile analysis must contain less than five times (5X) the CRQL of the phthalate esters listed

in Exhibit C. The concentration of non-target compounds in the blanks must not exceed 10 micrograms per liter (µg/L).

12.1.6 Corrective Action for Method Blank

12.1.6.1 If a Contractor's blank does not meet the technical acceptance criteria for method blanks the Contractor shall consider the analytical system to be out of control.

12.1.6.2 It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.6.3 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. Further, all samples (including requested MS/MSD and PE samples) processed with a method blank that does not meet the blank technical acceptance criteria (i.e., contaminated) will require re-extraction and re-analysis at no additional cost to USEPA.

12.1.6.4 If DMC recoveries in the method blank do not meet the technical acceptance criteria (Section 12.1.5.2), first re-analyze the method blank. If the DMC recoveries do not meet the technical acceptance criteria after re-analysis, then all samples (including requested MS/MSD and PE samples) associated with that method blank must be re-extracted and re-analyzed at no additional cost to USEPA.

12.1.6.5 If the method blank fails to meet a technical acceptance criteria other than Sections 12.1.5.6 and 12.1.5.2, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary), and re-analyze the method blank. Sample analysis cannot proceed until the method blank meets these technical acceptance requirements.

12.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

12.2.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the methods used for semivolatile analyses, USEPA has prescribed a mixture of semivolatile target compounds to be spiked into two aliquots of a sample and analyzed in accordance with the appropriate method upon request.

12.2.2 Frequency of MS/MSD Analyses

12.2.2.1 A matrix spike and matrix spike duplicate shall only be analyzed if requested by the Region [(through the Sample Management Office (SMO)] or specified on the Traffic Report (TR). If requested, a matrix spike and matrix spike duplicate must be extracted and analyzed for every 20 field samples in a Sample Delivery Group (SDG), or each SDG, whichever is most frequent.

12.2.2.2 As part of USEPA's QA/QC program, water rinsate samples and/or field/trip blanks (field QC) may be delivered to the laboratory for analysis. The Contractor shall not perform MS/MSD analysis on any of the field QC samples.

12.2.2.3 If the USEPA Region requesting MS/MSD designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample remaining to perform an MS/MSD, then the Contractor shall choose another sample on which to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify the Region (through SMO) that insufficient sample was received and identify the USEPA sample selected for the MS/MSD analysis. The rationale for the choice of another sample other than the one designated by USEPA shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample remaining in any of the samples in an SDG to perform the requested MS/MSD, then the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no MS/MSD is required, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2.1, then the Contractor shall contact SMO. SMO will contact the Region to determine which samples should have MS/MSD performed on them. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative. If this procedure is not followed, the Contractor will not be paid for MS/MSD analysis performed at a greater frequency than required by the contract.

12.2.2.6 When a Contractor receives only PE samples, no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a performance evaluation (PE) sample as part of a larger SDG, a sample other than the PE sample must be chosen for the requested MS/MSD analysis when the Region did not designate a sample to be used for this purpose.

12.2.3 Procedure for Preparing MS/MSD

12.2.3.1 Measure out two additional 1 L aliquots of the sample chosen for spiking in two continuous extractors. Add 1.0 mL of DMC spiking solution (Section 7.2.3) and 1.0 mL of the matrix spiking solution (Section 7.2.3.2) to each aliquot. Extract, concentrate, and analyze the MS/MSD according to the procedures described in Section 10.

12.2.4 Dilution of MS/MSD

MS/MSD samples must be analyzed at the same concentration as the most concentrated extract for which the original sample results will be reported. For example, if the original sample is to be reported at a 1:1 dilution and a 1:10 dilution, then analyze and report the MS/MSD at a 1:1 dilution only. However, if the original sample is to be reported at a 1:10 dilution and a 1:100 dilution, then the MS/MSD must be analyzed and reported at a 1:10 dilution only. Do <u>not</u> further dilute the MS/MSD samples to get either spiked or non-spiked analytes within calibration range. Dilution of the sample must be performed in accordance to the conditions in Section 10.2.5.

12.2.5 Calculations for MS/MSD

12.2.5.1 Calculate the recovery of each matrix spike compound in the matrix spike and matrix spike duplicate samples and report on the appropriate form. Calculate the concentrations of the matrix spike compounds using the same equation as used for target compounds (Equation 6). Calculate the recovery of each matrix spike compound as follows:

EQ. 9

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} * 100$$

Where:

SSR = Spike sample result.

SR = Sample result.

SA = Spike added.

12.2.5.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

EQ. 10

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} * 100$$

Where:

MSR = Matrix spike recovery.

MSDR = Matrix spike duplicate recovery.

12.2.6 Technical Acceptance Criteria for MS/MSD

12.2.6.1 If requested, all MS/MSD must be prepared and analyzed at the frequency described in Section 12.2.2. All MS/MSD must be analyzed on a GC/MS system meeting decafluorotriphenylphosphine (DFTPP), initial and continuing calibration technical acceptance criteria, and the method blank technical acceptance criteria.

12.2.6.2 The MS/MSD must have an associated method blank meeting the blank technical acceptance criteria.

12.2.6.3 The MS/MSD must be extracted and analyzed within the contract holding time.

12.2.6.4 The retention time shift for each of the internal standards must be within ±0.33 minutes (20 seconds) between the MS/MSD sample and the most recent continuing calibration standard.

12.2.6.5 The relative retention time for the DMCs must be within ±0.06 RRT units of its standard retention time in the continuing calibration standard.

12.2.6.6 The limits for matrix spike compound recovery and RPD are given in Table D-6. As these limits are only advisory, no further action by the laboratory is required; however, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questions from USEPA.

12.2.6.7 Corrective Action for Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Any MS/MSD which fails to meet the technical acceptance criteria in Sections 12.2.6.1 through 12.2.6.5 must be re-analyzed at no additional cost to USEPA.

12.3 Method Detection Limit (MDL) Determination

12.3.1 Before any field samples are analyzed under this contract, the MDL for each semivolatile target compound shall be determined for each sample extraction procedure and on one of the instruments to be used for sample analysis. The MDLs must be verified annually thereafter (see Section 12.3.2 for MDL verification procedures), until the contract expires or is terminated or after major instrument maintenance. Major instrument maintenance includes, but is not limited to cleaning or replacement of the mass spectrometer source, mass filters (e.g., quadrupole, ion trap, etc.), or electron multiplier (or similar device), or replacement of gas chromatographic column.

12.3.2 To determine the MDLs, the Contractor shall run an MDL study following the procedures specified in 40 CFR Part 136. The Contractor is only required to analyze the MDL samples on one instrument used for field sample analyses. MDL verification only is then required on all other instruments used for field sample analysis and at the frequency specified in Section 12.3.1. MDL verification is achieved by analyzing a single reagent water blank spiked with each target compound at a concentration equal to two times the analytical determined MDL. The resulting mass spectra of each target compound must meet the qualitative identification criteria outlined in Sections 11.1.1 through 11.1.4.3.

12.3.3 The determined concentration of the MDL must be less than the CRQL.

12.3.4 All documentation for the MDL studies shall be maintained at the laboratory and provided to USEPA upon written request.

13.0 METHOD PERFORMANCE

Not Applicable.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, USEPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult Less is Better: Laboratory Chemical Management for Waste Reduction, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

USEPA requires that laboratory waste management practices be conducted consistently with all applicable rules and regulations. USEPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult The Waste Management Manual for Laboratory Personnel, available from the American Chemical Society at the address listed in Section 14.2.

16.0 REFERENCES

Not Applicable.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Table D-1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0 - 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Present
70	Less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see note)
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	Greater than 0.75 percent of mass 198
441	Present but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

NOTE: All ion abundances 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.

Table D-2

Semivolatile Internal Standards With Corresponding
Target and Deuterated Monitoring Compounds Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Benzaldehyde	Nitrobenzene	Hexachlorocyclopentadiene
Phenol	Isophorone	2,4,6-Trichlorophenol
bis(2-Chloroethyl)ether	2-Nitrophenol	2,4,5-Trichlorophenol
2-Chlorophenol	2,4-Dimethylphenol	1,1'-Biphenyl
2-Methylphenol	bis(2-Chloroethoxy)methane	2-Chloronaphthalene
2,2'-oxybis-(1-Chloro-propane)	2,4-Dichlorophenol	2-Nitroaniline
Acetophenone	4-Chloroaniline	Dimethylphthalate
4-Methylphenol	Hexachlorobutadiene	Acenaphthylene
N-Nitroso-Di-n-propylamine	Caprolactam	3-Nitroaniline
Hexachloroethane	4-Chloro-3-methylphenol	Acenaphthene
Phenol-d_5 (DMC)	2-Methylnaphthalene	2,4-Dinitrophenol
Bis(2-chloroethyl)ether-d_8 (DMC)	Naphthalene	4-Nitrophenol
2-Chlorophenol-d_4 (DMC)	Nitrobenzene-d_5 (DMC)	Dibenzofuran
4-Methylphenol-d_8 (DMC)	2-Nitrophenol-d_4 (DMC)	2,4-Dinitrotoluene
	2,4-Dichlorophenol-d_3 (DMC)	2,6-Dinitrotoluene
	4-Chloroaniline-d_4 (DMC)	1,2,4,5-Tetrachlorobenzene
		Diethylphthalate
		4-Chlorophenyl-phenylether
		Fluorene
		4-Nitroaniline
		Acenaphthylene-d_8 (DMC)
		4-Nitrophenol-d_4 (DMC)
		Dimethylphthalate-d_6 (DMC)
		Fluorene-d_{10} (DMC)

Table D-2 (Con't)

Semivolatile Internal Standards With Corresponding
Target and Deuterated Monitoring Compounds Assigned for Quantitation

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octylphthalate
N-nitrosodiphenylamine	Butylbenzylphthalate	Benzo(b)fluoranthene
4-Bromophenyl-phenylether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
Atrazine	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
Pentachlorophenol	Chrysene	Dibenzo(a,h)anthracene
Phenanthrene	Pyrene-d_{10} (DMC)	Benzo(g,h,i)perylene
Anthracene		Benzo(a)pyrene-d_{12} (DMC)
Di-n-butylphthalate		
Fluoranthene		
4,6-Dinitro-2-methylphenol-d_2 (DMC)		
Anthracene-d_{10} (DMC)		

Table D-3

Characteristic Ions for Semivolatile Organic Compounds

Target Compounds	Primary Ion	Secondary Ion(s)
Benzaldehyde	77	105, 106
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
2-Methylphenol	108	107
2,2'-oxybis(1-Chloropropane)	45	77, 79
Acetophenone	105	77, 51
4-Methylphenol	108	107
N-nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(-2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
Caprolactam	113	55, 56
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
1,1'-Biphenyl	154	153, 76
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
1,2,4,5-Tetrachlorobenzene	216	214, 179, 108, 143, 218
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167

Table D-3 (Con't)

Characteristic Ions for Semivolatile Organic Compounds

Target Compounds	Primary Ion	Secondary Ion(s)
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Atrazine	200	173, 215
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octyl phthalate	149	-
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Deuterated Monitoring Compounds		
Phenol-d_5	99	71, 42
bis-(2-Chloroethyl)ether-d_8	67	99, 69
2-Chlorophenol-d_4	132	134, 68, 66
4-Methylphenol-d_8	113	115, 54
Nitrobenzene-d_5	128	82, 54
2-Nitrophenol-d_4	143	69, 41, 42
2,4-Dichlorophenol-d_3	165	167, 101
4-Chloroaniline-d_4	131	133, 69
Dimethylphthalate-d_6	166	78
Acenaphthylene-d_8	160	80, 158
4-Nitrophenol-d_4	143	113, 41, 42
Fluorene-d_{10}	176	174, 87, 86
4,6-Dinitro-2-methylphenol-d_2	200	170, 52
Anthracene-d_{10}	188	94, 80
Pyrene-d_{10}	212	106, 104
Benzo(a)pyrene-d_{12}	264	132, 118

Table D-3 (Con't)

Characteristic Ions for Semivolatile Organic Compounds

Internal Standard Compounds	Primary Ion	Secondary Ion(s)
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenapthene-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-4

Acceptance Criteria for Initial and Continuing Calibration of
Semivolatile Target Compounds and Deuterated Monitoring Compounds

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Benzaldehyde	0.010	none	none
Phenol	0.800	20.5	±25.0
bis-(2-Chloroethyl)ether	0.700	20.5	±25.0
2-Chlorophenol	0.800	20.5	±25.0
2-Methylphenol	0.700	20.5	±25.0
2,2'-oxybis-(1-Chloropropane)	0.010	none	none
Acetophenone	0.010	none	none
4-Methylphenol	0.600	20.5	±25.0
N-Nitroso-di-n-propylamine	0.500	20.5	±25.0
Hexachloroethane	0.300	20.5	±25.0
Nitrobenzene	0.200	20.5	±25.0
Isophorone	0.400	20.5	±25.0
2-Nitrophenol	0.100	30.0	±30.0
2,4-Dimethylphenol	0.200	30.0	±30.0
bis-(2-Chloroethoxy)methane	0.300	20.5	±25.0
2,4-Dichlorophenol	0.200	20.5	±25.0
Naphthalene	0.700	20.5	±25.0
4-Chloroaniline	0.010	none	none
Hexachlorobutadiene	0.010	none	none
Caprolactam	0.010	none	none
4-Chloro-3-Methylphenol	0.200	20.5	±25.0
2-Methylnaphthalene	0.400	20.5	±25.0
Hexachlorocyclopentadiene	0.010	none	none
2,4,6-Trichlorophenol	0.200	20.5	±25.0
2,4,5-Trichlorophenol	0.200	20.5	±25.0
1,1'-Biphenyl	0.010	none	none
2-Chloronaphthalene	0.800	20.5	±25.0
2-Nitroaniline	0.010	none	none
Dimethylphthalate	0.010	none	none
2,6-Dinitrotoluene	0.200	20.5	±25.0
Acenaphthylene	0.900	20.5	±25.0
3-Nitroaniline	0.010	none	none
Acenaphthene	0.900	20.5	±25.0
2,4-Dinitrophenol	0.010	none	none
4-Nitrophenol	0.010	none	none
Dibenzofuran	0.800	20.5	±25.0
2,4-Dinitrotoluene	0.200	30.0	±30.0
Diethylphthalate	0.010	none	none
1,2,4,5-Tetrachlorobenzene	0.010	none	none
4-Chlorophenyl-phenylether	0.400	20.5	±25.0
Fluorene	0.900	20.5	±25.0
4-Nitroaniline	0.010	none	none
4,6-Dinitro-2-Methylphenol	0.010	none	none
4-Bromophenyl-phenylether	0.100	20.5	±25.0
N-Nitrosodiphenylamine	0.010	none	none
Hexachlorobenzene	0.100	20.5	±25.0
Atrazine	0.010	none	none
Pentachlorophenol	0.050	20.5	±25.0
Phenanthrene	0.700	20.5	±25.0

Table D-4 (Con't)

Acceptance Criteria for Initial and Continuing Calibration of
Semivolatile Target Compounds and Deuterated Monitoring Compounds

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Anthracene	0.700	20.5	±25.0
Di-n-butylphthalate	0.010	none	none
Fluoranthene	0.600	20.5	±25.0
Pyrene	0.600	20.5	±25.0
Butylbenzylphthalate	0.010	none	none
3,3'-Dichlorobenzidine	0.010	none	none
Benzo(a)anthracene	0.800	20.5	±25.0
Chrysene	0.700	20.5	±25.0
bis-(2-Ethylhexyl)phthalate	0.010	none	none
Di-n-Octylphthalate	0.010	none	none
Benzo(b)fluoranthene	0.700	20.5	±25.0
Benzo(k)fluoranthene	0.700	20.5	±25.0
Benzo(a)pyrene	0.700	20.5	±25.0
Indeno(1,2,3-cd)pyrene	0.500	20.5	±25.0
Dibenzo(a,h)anthracene	0.400	20.5	±25.0
Benzo(g,h,i)perylene	0.500	20.5	±25.0
Deuterated Monitoring Compounds			
Phenol-d_5	0.010	none	none
bis-(2-Chloroethyl)ether-d_8	0.010	none	none
2-Chlorophenol-d_4	0.010	none	none
4-Methylphenol-d_8	0.010	none	none
Nitrobenzene-d_5	0.010	none	none
2-Nitrophenol-d_4	0.010	none	none
2,4-Dichlorophenol-d_3	0.010	none	none
4-Chloroaniline-d_4	0.010	none	none
Dimethylphthalate-d_6	0.010	none	none
Acenaphthylene-d_8	0.010	none	none
4-Nitrophenol-d_4	0.010	none	none
Fluorene-d_{10}	0.010	none	none
4,6-Dinitro-2-methylphenol-d_2	0.010	none	none
Anthracene-d_{10}	0.010	none	none
Pyrene-d_{10}	0.010	none	none
Benzo(a)pyrene-d_{12}	0.010	none	none

Table D-5

Deuterated Monitoring Compound Recovery Limits

Compound	% Recovery
Phenol-d_5	10-110
bis-(2-Chloroethyl)ether-d_8	41-94
2-Chlorophenol-d_4	33-110
4-Methylphenol-d_8	38-95
Nitrobenzene-d_5	35-114
2-Nitrophenol-d_4	40-106
2,4-Dichlorophenol-d_3	42-98
4-Chloroaniline-d_4	8-70
Dimethylphthalate-d_6	62-102
Acenaphthylene-d_8	49-98
4-Nitrophenol-d_4	9-181
Fluorene-d_{10}	50-97
4,6-Dinitro-2-methylphenol-d_2	53-153
Anthracene-d_{10}	55-116
Pyrene-d_{10}	47-114
Benzo(a)pyrene-d_{12}	54-120

Table D-6

Matrix Spike Recovery and Relative Percent Difference Limits

Compound	% Recovery	RPD
Phenol	12-110	42
2-Chlorophenol	27-123	40
N-Nitroso-di-n-propylamine	41-116	38
4-Chloro-3-methylphenol	23-97	42
Acenaphthene	46-118	31
4-Nitrophenol	10-80	50
2,4-Dinitrotoluene	24-96	38
Pentachlorophenol	9-103	50
Pyrene	26-127	31

EXHIBIT D

METHOD FOR THE ANALYSIS OF LOW CONCENTRATION WATER FOR PESTICIDES AND AROCLORS

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Exhibit D -- Analytical Methods for Pesticides/Aroclors

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water in order to determine the presence and concentrations of the chlorinated pesticides and Aroclors found in the Target Compound List (Exhibit C - Pesticides). The majority of the samples are expected to be from drinking water and well/ground water type sources around Superfund sites. The method can be used for determining analyte concentrations as low as ten parts per trillion. The method is based on EPA Method 608. The method includes sample extraction, extract cleanup techniques, and the Gas Chromatograph/Electron Capture Detector (GC/ECD) analytical method for chlorinated pesticides and aroclors.

1.2 Resolution difficulties have been associated with the following pairs of compounds using this method:

- On a DB-608 or equivalent column, DDE and Dieldrin; Methoxychlor and Endrin ketone; and Endosulfan I and gamma-Chlordane.

- On a DB-1701 or equivalent column, Endosulfan I and gamma-Chlordane; and Methoxychlor and Endosulfan sulfate.

1.3 There are two isomers of heptachlor epoxide, the endo epoxy isomer (isomer A) and the exo epoxy isomer (isomer B). The two isomers are separable using current GC capillary columns. Only the exo epoxy isomer (isomer B) is of environmental significance. This is the isomer that must be used as an analytical standard, identified and quantitated in sample analysis, and reported on appropriate forms as heptachlor epoxide.

2.0 SUMMARY OF METHOD

2.1 A one liter aliquot of sample is spiked with the surrogate solution and extracted with methylene chloride by using a continuous liquid-liquid extractor or separatory funnel. The methylene chloride extract is dried and concentrated, exchanged to hexane, cleaned up to remove interferences, and adjusted to a final volume of 2.0 milliliters (mL).

2.2 The hexane extract is injected onto two wide-bore capillary columns in a Gas Chromatograph (GC). The GC is temperature programmed to separate the pesticides and Aroclors which are then detected with an Electron Capture Detector (ECD). Calibration and run sequence specifications of the GC/ECD method apply independently to each GC column.

2.3 A single component pesticide is identified if a peak is detected within its appropriate Retention Time (RT) window on each of two GC columns. Quantitative analysis of pesticides/Aroclors must be accomplished by the external standard method. Single component analytes and the surrogates must be analyzed at three concentration levels during the initial calibration.

2.4 Toxaphene and Aroclors are identified primarily by pattern recognition, but RTs of three to five major peaks must also be taken into consideration. Single-point calibrations for multicomponent analytes are sufficient for quantitation by this method. Standards for identified Aroclors and Toxaphene must be run within 72 hours of the sample analysis in which they were observed. These standards are used to verify identification only; quantitation is based on the standards analyzed during initial calibration.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in gas chromatograms. These materials must be routinely demonstrated to be free from interferences under the sample preparation and analysis conditions by analyzing instrument blanks and method blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Because common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.2 Matrix Interferences

Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. The cleanup procedures in this method must be used to remove such interferences in order to achieve the Contract Required Quantitation Limits (CRQL).

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. 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 data handling sheets should be made available to all personnel involved in these analyses. Specifically, concentrated sulfuric acid and the 10 N sodium hydroxide solution are moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

5.2 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the Aroclors. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here, but demonstration of equivalent performance meeting the requirements of the Statement of Work (SOW) is the responsibility of the Contractor. The Contractor must document in its Sample Delivery Group (SDG) Narrative when it uses equipment and supplies other than those specified here.

6.1 Glassware

6.1.1 Continuous Liquid-Liquid Extractors

Continuous Liquid-Liquid Extractors equipped with PTFE or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf extractor, Ace Glass Company, Vineland, NJ P/N 6841-10 or equivalent) or Hydrophobic Membrane-based Extractor (Accelerated One Step™ Extractor, Corning series 3195 or equivalent).

6.1.2 Separatory Funnels - 2 liter with PTFE stopcock.

6.1.3 Graduated Cylinder - 1 liter capacity.

6.1.4 Drying column, chromatographic column approximately 400 millimeters (mm) long x 19 mm ID, with coarse frit. (Substitution of a small pad of disposable Pyrex glass wool for the frit will help prevent cross-contamination of sample extracts.)

6.1.5 Kuderna-Danish Apparatus

6.1.5.1 Concentrator Tubes - 10 milliliters (mL), graduated (Kontes, K-570050-1025, or equivalent).

6.1.5.2 Evaporative Flasks - 500 mL (Kontes K-570001-0500, or equivalent). Attach to concentrator tube with springs.

6.1.5.3 Snyder Columns - three-ball macro (Kontes K-50300-0121, or equivalent).

6.1.5.4 Snyder Columns, micro two or three ball with a 19 mm ground glass joint.

6.1.6 Pipet, Volumetric 1.00 mL or 2.00 mL.

6.1.7 Microsyringe, 1.0 microliter (µL) and larger, 0.006 (0.15 mm) inch ID needle.

6.1.8 Syringe, 1.00 mL or 2.00 mL (optional).

6.1.9 Volumetric flask, 10.00 mL, and 1 or 2 mL.

6.1.10 Vials and caps, 20 and 10 mL, with screw cap and PTFE or aluminum foil liner, 2 or 1 mL for Gas Chromatograph (GC) auto sampler.

6.1.11 Bottle or test tube, 20 mL with PTFE-lined screw cap for sulfur removal.

6.1.12 Centrifuge tubes, calibrated, 12 mL, for sulfur removal.

6.1.13 Micropipet, 200 µL, with disposable tips.

6.2 Florisil Cleanup Equipment

6.2.1 Florisil bonded silica. 1 g cartridges with stainless steel or PTFE frits, Catalog No. 694-313 (Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent).

6.2.2 Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold, Analytichem International, J.T. Baker, or Supelco (or equivalent).

6.2.3 Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

6.2.4 Vacuum pressure gauge.

6.2.5 Rack for holding 10 mL volumetric flasks in the manifold.

NOTE: Other types of equivalent systems, such as an automated system using syringe pressure are considered acceptable for elution of Florisil cartridges, as long as all quality control (QC) and sample technical acceptance criteria are met.

6.3 pH Paper, Wide Range

6.4 pH Meter -- With a combination glass electrode. Calibrate according to manufacturer's instructions. pH meter must be calibrated prior to each use.

6.5 Boiling Chips

6.5.1 Silicon carbide boiling chips, approximately 10 to 40 mesh. Heat the chips to 400°C for 30 minutes or solvent rinse before use.

6.5.2 PTFE boiling chips (optional). Solvent rinse the chips before use.

6.6 Water Bath, heated, with concentric ring cover, capable of temperature control.

NOTE: To prevent the release of solvent fumes into the laboratory, the water bath must be used in a hood.

6.7 Balances

Analytical balances, capable of weighing accurately to ±0.0001 g. The balances must be calibrated with class S weights or known reference weights once per each 12-hour work shift. The balances must be calibrated with class S weights at a minimum of once per month. The balances must also be annually checked by a certified technician.

6.8 Nitrogen Evaporation Device

Nitrogen evaporation device equipped with a heated bath that can be maintained at 35 to 40°C (N-Evap by Organomation Associates, Inc., South Berlin, MA, or equivalent). To prevent the release of solvent fumes into the laboratory, the nitrogen evaporation device must be used in a hood.

6.9 Mechanical Shaker or Mixer, for Sulfur Removal

6.10 Gas Chromatograph/Electron Capture Detector (GC/ECD) System

6.10.1 Gas Chromatograph (GC)

6.10.1.1 The GC 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 have all required accessories including syringes, analytical columns, and gases.

6.10.1.2 GCs that are available from some manufacturers may have difficulty in meeting certain method QC requirements because of endrin and DDT breakdown in the injector. This problem can be minimized by operating the injector at 200-205°C, using a Pyrex (not quartz) methyl silicone deactivated injector liner, and deactivating the metal parts in the injector with dichlorodimethyl silane. In some cases, using a 0.25 inch packed column injector converted for use with 0.53 mm capillary columns works better than a Grob-type injector. If a Grob-type injector is used, a 4 mm liner may be required to meet breakdown criteria.

6.10.1.3 Gas Chromatograph Columns -- Two wide-bore (0.53 mm ID) fused silica GC columns are required. A separate detector is required for each column. The specified analytical columns are a 30 m x 0.53 mm ID, 1.0 µm film thickness DB-1701 (J&W Scientific); SPB 1701 (Supelco); AT 1701 (Alltech); RTX-1701 (Restek); CP-Sil 19CB (Chrompack); 007-1701 (Quadrex); BP-10 (SGE); or equivalent, and a 30 m x 0.53 mm ID, 0.5 to 1.0 micrometer (• m) film thickness DB-608 (J&W Scientific); HP-608 (Hewlett Packard); SPB-608 (Supelco); 007-608 (Quadrex); BP-608 (SGE); CP-Sil 8CB (Chrompack); or equivalent.

NOTE: The column length stated above is the minimum requirement. Longer columns that meet resolution and calibration requirements may be used. A description of the GC columns used for analysis shall be provided in the SDG Narrative.

6.10.1.3.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants which interfere with identification and quantitation of the compounds listed in Exhibit C (Pesticides).
- The analytical results generated using the column meet the initial calibration and calibration verification technical acceptance criteria listed in the SOW and the Contract Required Quantitation Limits (CRQLs) listed in Exhibit C (Pesticides).
- The column can accept at least 16 times the low point standard for individual standard mixtures A and B for each compound listed in Exhibit C (Pesticides) without becoming overloaded.
- The column pair chosen must have dissimilar phases/chemical properties in order to separate the compounds of interest in different Retention Time (RT) order.

6.10.1.3.2 Although the instructions included in the SOW are for wide bore capillary columns, narrower bore capillary columns may be evaluated for use. Follow manufacturer's instructions for use

of its product. Document in SDG Narrative if other columns are used by specifying the column used (Exhibit B Section 2.5.1).

6.10.1.3.3 As applicable, follow the manufacturer's instructions for use of its product.

6.10.1.3.4 The Contractor must maintain documentation that the alternate column met the criteria in Sections 9.2.5 and 9.3.5. The minimum documentation is as follows:

6.10.1.3.4.1 Manufacturer provided information concerning the performance characteristics of the column;

6.10.1.3.4.2 GC chromatograms and data system reports generated on the GC/ECD and used for CLP analyses:

- From instrument blanks which demonstrate that there are no contaminants which interfere with the pesticide analysis when using the alternate column;
- For initial calibration standards analyzed using the alternate column; and
- For calibration verification standards analyzed using the alternate column.

6.10.1.3.5 Based on the Contractor generated data described in Section 6.10.1.3.4.2, the Contractor must complete a written comparison and review, signed by the Laboratory Manager certifying that:

- The alternate column performance is comparable to the required column performance in its ability to produce initial calibrations and calibration verifications which meet the technical acceptance criteria in Sections 9.2.5 and 9.3.5;
- The low point initial calibration standard analyses have adequate sensitivity to meet the pesticide CRQLs;
- The high point initial calibration standard analyses were not overloaded; and
- The alternate column does not introduce contaminants which interfere with identification and quantitation of compounds listed in Exhibit C (Pesticides).

6.10.1.3.6 The documentation must be made available to USEPA during on-site laboratory evaluations or sent to USEPA upon request of the Contract Laboratory Program Project Officer (CLP PO).

6.10.1.3.7 PACKED COLUMNS CANNOT BE USED.

6.10.1.4 Columns are mounted in a 0.25-inch injector ports by using glass adapters available from a variety of commercial sources (J&W Scientific, Supelco, Inc., Hewlett-Packard, Varian, Inc., Perkin Elmer, or equivalent). The two columns may be mounted into a single injection port with a tee adapter (Supelco, Inc., Bellefonte, PA, Catalog No. 2-3660, or equivalent). Use of this adapter allows simultaneous injection onto both columns. The laboratory should follow manufacturer's recommendations for mounting 0.53 mm capillary columns in injector ports.

6.10.1.5 The carrier gas for routine applications is helium. Laboratories may choose to use hydrogen as a carrier gas, but they must clearly identify its use in the SDG Narrative and on all divider pages preceding raw chromatographic data in submissions to USEPA. 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.

6.10.2 Electron Capture Detector (ECD)

The linearity of the response of the ECD may be greatly dependent on the flow rate of the make-up gas. The make-up gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector. 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 outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.10.3 Data System

A data system must be interfaced to the 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.

6.10.4 Magnetic Tape Storage Device

Magnetic tape storage devices must be capable of recording data and suitable for long-term, off-line storage of GC/ECD data.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1.1 Reagent water -- Defined as water in which no target analyte is observed at the Contract Required Quantitation Limits (CRQL) for that compound.

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 grams (g) (1 pound) of activated carbon (Calgon Corp., Filtrasorb-300, or equivalent).

7.1.1.2 Reagent water may be generated using a water purification system (Millipore Super-Q or equivalent).

7.1.2 Sodium sulfate, granular-anhydrous reagent grade, heated at 400°C for 4 hours, or at 120°C for 16 hours, cooled in a desiccator, and stored in a glass bottle. Each lot must be extracted with hexane and analyzed by Gas Chromatograph/Electron Capture Detector (GC/ECD) to demonstrate that it is free of interference before use. J. T. Baker anhydrous granular, Catalog No. 3375, or equivalent.

CAUTION: An open container of sodium sulfate may become contaminated during storage in the laboratory.

7.1.3 Methylene chloride, hexane, acetone, toluene, iso-octane, and methanol (optional), pesticide quality, or equivalent. It is recommended that each lot of solvent be analyzed to demonstrate that it is free of interference before use. Methylene chloride must be certified as acid free or must be tested to demonstrate that it is free of hydrochloric acid. Acidic methylene chloride must be passed through basic alumina and then demonstrated to be free of hydrochloric acid.

7.1.4 Mercury, triple distilled, for sulfur clean-up.

7.1.5 Copper powder (optional), fine, granular (Mallinckrodt 4649 or equivalent). Copper may be used instead of mercury for sulfur clean-up. Remove oxides by treating with dilute nitric acid, rinse with distilled water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

7.1.6 Sodium hydroxide solution (10 N). Carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 milliliters (mL).

7.1.7 Concentrated sulfuric acid, (Sp. Gr. 1.84)-36N.

7.1.8 Nitric acid, dilute, for sulfur removal with copper.

7.1.9 Ten percent acetone in hexane (v/v). Prepare by adding 10.0 mL of acetone to 90.0 mL of hexane.

NOTE: Prepare this mixture accurately or the results from the Florisil cartridge cleanup will be adversely affected. Water in the acetone will also adversely affect Florisil performance.

7.2 Standards

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. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

7.2.1 Stock Standard Solutions

7.2.1.1 Stock standard solutions may be purchased as certified solutions or prepared from pure standard materials.

7.2.1.2 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in toluene, dilute to volume in a 10 mL volumetric flask with toluene or acetone. Larger volumes may be used at the convenience of the analyst.

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

7.2.1.4 Fresh stock standards must be prepared once every six months, or sooner, if standards have degraded or concentrated. Stock standards must be checked for signs of degradation or concentration just prior to preparing working standards from them.

7.2.2 Secondary Dilution Standards

7.2.2.1 Using stock standards, prepare secondary dilution standards in acetone that contain the compounds of interest either singly or mixed together.

7.2.2.2 Fresh secondary dilution standards must be prepared once every six months, or sooner, if standards have degraded or concentrated. Secondary dilution standards must be checked for signs of degradation or concentration just prior to preparing working standards from them.

7.2.3 Working Standards

7.2.3.1 Surrogate Standard Spiking Solution

The surrogates, tetrachloro-m-xylene and decachlorobiphenyl, are added to all standards, samples (including Laboratory Control Samples), matrix spike, matrix spike duplicates and if required, Performance Evaluation (PE) samples, and required blanks (method/sulfur clean-up/instrument). Prepare a surrogate spiking solution of 0.20 micrograms per milliliter (µg/mL) of each of the two compounds in acetone. The solution should be checked frequently for stability. The solution must be replaced every 6 months, or sooner if the solution has degraded or concentrated.

7.2.3.2 Matrix Spiking Solution

Prepare a matrix spiking solution in acetone or methanol that contains the following pesticides at the concentrations specified. The solution must be replaced every six months, or sooner if the solution has degraded or concentrated.

Pesticide	Concentration (ug/mL)
gamma- BHC (Lindane)	0.1
4,4'-DDT	0.2
Endrin	0.2
Heptachlor	0.1
Aldrin	0.1
Dieldrin	0.2

7.2.3.3 Resolution Check Mixture

The Resolution Check Mixture is composed of the pesticides and surrogates at the concentrations listed below in hexane or iso-octane. The mixture must be prepared every six months, or sooner, if the solution has degraded or concentrated.

Compounds	Concentration (ng/mL)
gamma- Chlordane	10.0
Endosulfan I	10.0
4,4'-DDE	20.0
Dieldrin	20.0
Endosulfan sulfate	20.0
Endrin ketone	20.0
Methoxychlor	100.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

7.2.3.4 Performance Evaluation Mixture (PEM)

The PEM is prepared in hexane or iso-octane, as listed below. The PEM must be prepared weekly, or sooner if the solution has degraded or concentrated.

Compounds	Concentration (ng/mL)
gamma-BHC	10.0
alpha-BHC	10.0
4,4'-DDT	100.0
beta-BHC	10.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

7.2.3.5 Individual Standard Mixtures A and B

The Individual Standard Mixture solutions must be prepared in either hexane or iso-octane. The concentrations of the pesticides in the low point standard mixtures are given below. The midpoint concentration must be 4 times the low point concentration for each analyte, including the surrogates. The high concentration must be at least 16 times the low point concentration for each analyte, including the surrogates, but a higher concentration may be chosen by the Contractor. The high point concentration defines the upper end of the concentration range for which the calibration is valid. The solution must be prepared every six months, or sooner, if the solution has degraded or concentrated.

Individual Standard Mix A	Low Point Concentration (ng/mL)	Individual Standard Mix B	Low Point Concentration (ng/mL)
alpha-BHC	5.0	beta-BHC	5.0
Heptachlor	5.0	delta-BHC	5.0
gamma-BHC	5.0	Aldrin	5.0
Endosulfan I	5.0	Heptachlor-epoxide (exo-epoxy isomer)	5.0
Dieldrin	10.0	alpha-Chlordane	5.0
Endrin	10.0	gamma-Chlordane	5.0
4,4'-DDD	10.0	4,4'-DDE	10.0
4,4'-DDT	10.0	Endosulfan sulfate	10.0
Methoxychlor	50.0	Endrin aldehyde	10.0
Tetrachloro-m-xylene	5.0	Endrin ketone	10.0
Decachlorobiphenyl	10.0	Endosulfan II	10.0
		Tetrachloro-m-xylene	5.0
		Decachloro-biphenyl	10.0

NOTE: Only the exo-epoxy isomer (isomer B) of heptachlor expoxide is used as an analytical standard.

7.2.3.6 Multicomponent Standards

Toxaphene and Aroclor standards must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture. The calibration standards for the Aroclors must be prepared at concentrations of 100 nanograms (ng)/mL, except for Aroclor 1221 which must be prepared at 200 ng/mL. Toxaphene must be prepared at 500 ng/mL. All multicomponent standards must contain the surrogates at 20.0 ng/mL. The Aroclor and toxaphene solutions must be prepared in hexane or iso-octane. Each solution must be prepared every 6 months, or sooner, if the solution has degraded or concentrated.

7.2.3.7 Florisil Cartridge Check Solution

Prepare a 0.10 µg/mL solution of 2,4,5-trichlorophenol in acetone. The solution must be prepared every six months, or sooner, if the solution has degraded or concentrated.

7.2.3.8 Laboratory Control Sample (LCS) Spiking Solution

Prepare a LCS spiking solution that contains each of the analytes at the concentrations listed below in methanol or acetone. The LCS solution must be prepared every six months, or sooner, if the solution has degraded or concentrated.

Compounds	Concentration (µg/mL)
gamma-BHC	0.10
Heptachlor epoxide	0.10
Dieldrin	0.20
4,4'-DDE	0.20
Endrin	0.20
Endosulfan sulfate	0.20
gamma-Chlordane	0.10

7.2.4 Ampulated Standard Extracts

Standard solutions purchased from a chemical supply house as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor which are immediately ampulated in glass vials may be retained for 2 years from preparation date. Upon breaking the glass seal, the expiration times listed in Sections 7.2.1 to 7.2.3 will apply. The Contractor is responsible for assuring that the integrity of the standards has not degraded (Section 7.2.5.5).

7.2.5 Storage of Standards

7.2.5.1 Store the stock and secondary standard solutions at 4°C (±2°C) in PTFE-lined, screw-cap, amber bottles/vials.

7.2.5.2 Store the working standard solutions at 4°C (±2°C) in PTFE-lined screw-cap, amber bottles/vials. The working standards must be checked frequently for signs of degradation or evaporation.

7.2.5.3 Protect all standards from light.

7.2.5.4 Samples, sample extracts, and standards must be stored separately.

7.2.5.5 The Contractor is responsible for maintaining the integrity of standard solutions and verifying prior to use. Storage of standard solutions in the freezer may cause some compounds to precipitate. This means at the minimum, the standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in solution. Additional steps may be necessary to ensure all components are in solution.

7.2.6 Temperature Records for Storage of Standards

7.2.6.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.6.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.6.3 Corrective action SOPs shall be posted on the refrigerators.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in 1 L (or 1 quart) amber glass containers, fitted with screw-caps lined with PTFE. If amber containers are not available, the samples should be protected from light. The specific requirements for site sample collection are outlined by the Region.

8.1.2 All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until extraction.

8.2 Procedure for Sample Storage

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

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3 Procedure for Sample Extract Storage

8.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C) until 365 days after delivery of a complete reconciled data package to USEPA.

8.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3.3 Samples, sample extracts, and standards must be stored separately.

8.4 Records for Sample and Sample Extract Storage

8.4.1 The temperature of all sample and sample extract storage refrigerators shall be recorded daily.

8.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

8.4.3 Corrective action SOPs shall be posted on the refrigerators.

8.5 Contract Required Holding Times

8.5.1 Extraction of water samples by separatory funnel procedures must be completed within 5 days of the Validated Time of Sample Receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction must be started within 5 days of VTSR.

8.5.2 As part of USEPA's QA program, USEPA may provide Performance Evaluation (PE) Samples as standard extracts which the Contractor is required to prepare per instructions provided by USEPA. PE samples must be prepared and analyzed concurrently with the samples in the SDG. The extraction holding time (5 days after VTSR) does not apply for PE Samples received as standard extracts.

8.5.3 Analysis of sample extracts must be completed within 40 days following the start of extraction.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Gas Chromatograph (GC) Operating Conditions

9.1.1 The following are the gas chromatographic analytical conditions. The conditions are recommended unless otherwise noted.

Carrier Gas:	Helium (Hydrogen may be used, Section 6.10.1.5)
Column Flow:	5 mL/min
Make-up Gas:	Argon/Methane (P-5 or P-10) or N_2 (required)
Injector Temperature:	> 200°C (Section 9.1.5)
Injection Technique:	On-column
Injection Volume:	1 or 2 µl (Section 9.1.3)
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	0.5 min
Temperature Ramp:	5°C to 6°C/min
Final Temperature:	275°C
Final Hold Time:	After Decachlorobiphenyl has eluted (approximately 10 minutes)

9.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples (including Laboratory Control Samples, requested matrix spike, and matrix spike duplicate), and required blanks (method/sulfur clean-up/instrument).

9.1.3 Manual injections must be 2.0 microliter (µL). Auto injectors may use 1.0 µL volumes. The same injection volume must be used for all standards, samples (including Laboratory Control Samples, requested matrix spike, and matrix spike duplicate) and required blanks (method/sulfur clean-up/instrument).

9.1.4 The linearity of the Electron Capture Detector (ECD) may be greatly dependent on the flow rate of the make-up gas. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector.

9.1.5 Cold (ambient temperature) on-column injectors that allow injection directly onto a 0.53 mm ID column may be used as long as the initial calibration and calibration verification technical acceptance criteria are met.

9.2 Initial Calibration

9.2.1 Summary of Initial Calibration

Prior to sample (including Laboratory Control Samples, requested matrix spike and matrix spike duplicate) and required blanks

(method/sulfur clean-up) analysis, each GC/ECD system must be initially calibrated at a minimum of three concentrations for single component analytes and surrogates in order to determine instrument sensitivity and the linearity of GC response. Multicomponent target analytes are calibrated at a single point.

9.2.2 Frequency of Initial Calibration

Each GC/ECD system must be initially calibrated upon award of the contract, whenever major instrument maintenance or modification is performed (e.g., column replacement or repair, cleaning or replacement of ECD, etc.), or if the calibration verification technical acceptance criteria have not been met.

9.2.3 Procedure for Initial Calibration

9.2.3.1 Set up the GC/ECD system as described in Section 9.1.

9.2.3.2 Prepare the initial calibration standards using the procedures, the analytes, and the concentrations according to Section 7.2.

9.2.3.3 All standards, samples (including Laboratory Control Samples, requested matrix spike, and matrix spike duplicate) and required blanks (method/sulfur clean-up) extracts must be allowed to warm to ambient temperature before analysis.

9.2.3.4 Analyze the initial calibration sequence as given below.

NOTE: Steps 16 and 17 are used as part of the calibration verification as well (Section 9.3).

INITIAL CALIBRATION SEQUENCE

1. Resolution Check
2. Performance Evaluation Mixture
3. Aroclor 1016/1260
4. Aroclor 1221
5. Aroclor 1232
6. Aroclor 1242
7. Aroclor 1248
8. Aroclor 1254
9. Toxaphene
10. Low Point Standard A
11. Low Point Standard B
12. Midpoint Standard A
13. Midpoint Standard B
14. High Point Standard A
15. High Point Standard B
16. Instrument Blank
17. Performance Evaluation Mixture

9.2.4 Calculations for Initial Calibration

9.2.4.1 During the initial calibration sequence, absolute Retention Times (RTs) are determined for all single component pesticides, the surrogates, and at least three major peaks of each multicomponent analyte.

9.2.4.2 For each single component pesticides, a RT is measured in each of the three calibration standards (low point, midpoint, high point) for Individual Standard Mixture A and Individual Standard Mixture B. **The RT for the surrogates is measured from each of the three analyses of Individual Standard Mixture A during the initial calibration.** The mean RT is calculated for each single component pesticide and surrogate as the average of the three values. Calculate a mean absolute RT for each single component pesticide and surrogate using Equation 1.

EQ. 1

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where:

$\overline{RT}$ = Mean absolute retention time of analyte.

RT_i = Absolute retention time of analyte.

n = Number of measurements (3).

9.2.4.3 A RT window is calculated for each single component analyte and surrogate and for the major peaks (3 to 5) of each multicomponent analyte by using Table D-1. Windows are centered around the average absolute RT for the analyte established during the initial calibration. Analytes are identified when peaks are observed in the RT window for the compound on both GC columns.

9.2.4.4 The linearity of the instrument is determined by calculating a percent relative standard deviation (%RSD) of the calibration factors from a three-point calibration curve for each of the single component pesticide and surrogates. Either peak area or peak height may be used to calculate calibration factors used in the %RSD equation. For example, it <u>is</u> permitted to calculate linearity for endrin based on peak area and to calculate linearity for aldrin based on peak height. It is <u>not</u> permitted within a %RSD calculation for an analyte to use calibration factors calculated from both peak area and peak height. For example, it is <u>not</u> permitted to calculate the calibration factor for the low point standard for endrin using peak height and calculate the midpoint and high point standard calibration factors for endrin using peak area.

9.2.4.5 Calculate the calibration factor for each single component pesticide and surrogate over the initial calibration range using Equation 2. The calibration factors for surrogates are calculated from the three analyses of Individual Standard A mixture only.

9.2.4.6 Calculate the mean and the %RSD of the calibration factors for each single component pesticide and surrogate over the initial calibration range using Equation 3 and Equation 4.

EQ. 2

$$CF = \frac{\text{Peak area (or Height) of the standard}}{\text{Mass Injected (ng)}}$$

EQ. 3

$$\overline{CF} = \frac{\sum_{i=1} CF_i}{n}$$

EQ. 4

$$\%RSD = \frac{SD_{CF}}{\overline{CF}} * 100$$

Where:

$$SD_{CF} = \sqrt{\frac{\sum_{i=1} (CF_i - \overline{CF})^2}{(n-1)}}$$

%RSD = Percent relative standard deviation.

SD_{CF} = Standard deviation of calibration factors.

CF_i = Calibration factor.

$\overline{CF}$ = Mean calibration factor.

n = Total number of values (3).

9.2.4.7 A calibration factor is calculated for each peak in a selected set of three to five major peaks for each multicomponent analyte using Equation 2.

9.2.4.8 Calculate the percent breakdown of DDT, the percent breakdown of Endrin, and the combined breakdown of DDT and Endrin in the Performance Evaluation Mixture (PEM) using Equations 5, 6, 7, and 8.

EQ. 5

$$\text{Amount found (ng)} = \frac{\text{Peak area (or Peak height) of compound in PEM}}{CF_{mp}}$$

Where:

CF_{mp} = The calibration factor for the compound determined from the midpoint standard in the most recent initial calibration.

NOTE: If during the initial calibration, linearity was determined based on peak area for the compound, then the midpoint CF must be based on peak area. If during the initial calibration, the linearity for the compound was determined based on peak height for the compound, then the midpoint CF must be based on peak height.

EQ. 6

$$\%\text{Breakdown DDT} = \frac{\text{Amount found (ng) (DDD+DDE)}}{\text{Amount (ng) of DDT injected}} * 100$$

EQ. 7

$$\%\text{Breakdown Endrin} = \frac{\text{Amount found (ng) (endrin aldehyde + endrin ketone)}}{\text{Amount (ng) of endrin injected}} * 100$$

EQ. 8

$$\text{Combined \%Breakdown} = \%\text{Breakdown DDT} + \%\text{Breakdown Endrin}$$

9.2.4.9 Calculate the percent difference for each pesticide and surrogate in the PEM using Equations 5 and 9.

EQ. 9

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} * 100$$

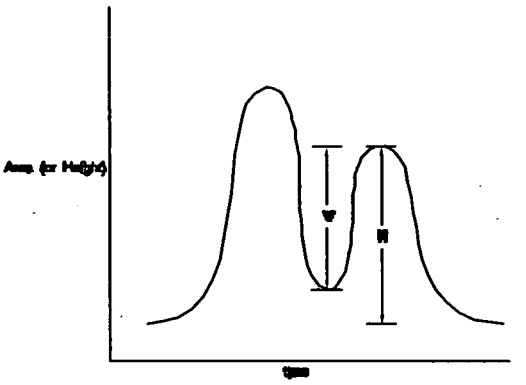
Where:

%D = Percent Difference.

C_{nom} = Nominal concentration of each analyte.

C_{calc} = Calculated concentration of each analyte from the analysis of the standard.

9.2.4.10 Calculate the resolution between the analytes in the Resolution Check Mixture, PEM and the midpoint concentrations of Individual Standard Mixtures A and B using Equation 10.



EQ. 10

$$\%\text{Resolution} = \frac{V}{H} * 100$$

Where:

V = Depth of the valley between the two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter of the adjacent peaks.

9.2.5 Technical Acceptance Criteria for Initial Calibration

All initial calibration technical acceptance criteria apply independently to each GC column.

9.2.5.1 The initial calibration sequence must be analyzed according to the procedure and in the order listed in Section 9.2.3, at the concentrations listed in Section 7.2.3, and at the frequency listed in Section 9.2.2. The GC/ECD operating conditions optimized in Section 9.1 must be followed.

9.2.5.2 The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0 percent.

9.2.5.3 All single component pesticides and surrogates in both runs of the PEM must be greater than or equal to 90.0 percent resolved on each column.

9.2.5.4 The absolute RTs of each of the single component pesticides and surrogates in both runs of the PEM must be within the RT window determined from the three-point initial calibration in Section 9.2.4.3.

9.2.5.5 The percent difference of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in both of the PEM runs of each GC column must be greater than or equal to -25 percent and less than or equal to 25 percent using Equation 9.

9.2.5.6 The percent breakdown of DDT and endrin in each of the PEM runs must be less than or equal to 20.0 percent. The combined breakdown of DDT and endrin must be less than or equal to 30.0 percent.

9.2.5.7 The %RSD of the calibration factors for each single component target compound must be less than or equal to 20.0 percent, except alpha-BHC and delta-BHC. The %RSD of the calibration factors for alpha-BHC and delta-BHC must be less than or equal to 25.0 percent. The %RSD of the calibration factors for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (not surrogates) per column may exceed the 20.0 percent limit for %RSD (25.0 percent for alpha-BHC and delta-BHC), but those compounds must have a %RSD of less than or equal to 30.0 percent.

9.2.5.8 The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

9.2.5.9 All instrument blanks must meet the technical acceptance criteria in Section 12.1.4.5.

9.2.5.10 The identification of single component pesticides by gas chromatographic methods is based primarily on RT data. The RT of the apex of a peak can only be verified from an on-scale chromatogram. The identification of multicomponent analytes by gas chromatographic methods is based primarily on recognition of patterns of RTs displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.

- The chromatograms that result from the analyses of the Resolution Check Mixture, the PEM, and Individual Standard Mixtures A and B during the initial calibration sequence must display the single component analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

- The chromatograms for at least one of the three analyses each of Individual Standard Mixtures A and B from the initial calibration sequence must display the single component analytes at greater than 50 percent and less than 100 percent of full scale.

- The chromatograms of the standards for the multicomponent analytes analyzed during the initial calibration sequence must display the peaks chosen for identification of each analyte at greater than 25 percent and less than 100 percent of full scale.

- For all Resolution Check Mixtures, PEMs, Individual Standard Mixtures, and blanks, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

- If a chromatogram is replotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.

- If the chromatogram of any standard needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

9.2.6 Corrective Action for Initial Calibration

9.2.6.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the technical acceptance criteria.

9.2.6.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. In the case of low level contamination, baking out the detector at elevated temperature (350°C) should be sufficient to achieve acceptable performance. In the case of heavy contamination, passing hydrogen through the detector for 1-2 hours at elevated temperature may correct the problem. In the case of severe contamination, the detector may require servicing by the ECD manufacturer. DO NOT OPEN THE DETECTOR. THE ECD CONTAINS RADIOCHEMICAL SOURCES.

9.2.6.3 If a laboratory cleans out a detector using elevated temperature, the ECD electronics must be turned off during the bake out procedure.

9.2.6.4 After bake out or hydrogen reduction, the detector must be recalibrated using the initial calibration sequence.

9.2.6.5 Initial calibration technical acceptance criteria MUST be met before any samples (including Laboratory Control Samples, matrix spike, and matrix spike duplicate, if required) or required blanks (method/sulfur clean-up) are analyzed. Any samples or required blanks analyzed when the initial calibration technical acceptance criteria have not been met will require re-analysis at no additional cost to USEPA.

9.3 Calibration Verification

9.3.1 Summary of Calibration Verification

Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, PEMs, and the mid point concentration of Individual Standard Mixtures A and B constitute the calibration verification. Sample (including Laboratory Control Sample and matrix spike and matrix spike duplicate, if required) and required blank (method/sulfur clean-up) data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

9.3.2 Frequency of Calibration Verification

9.3.2.1 An instrument blank and the PEM must bracket one end of a 12-hour period during which sample and required blank data are collected, and a second instrument blank and the mid point concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period.

9.3.2.2 For the 12-hour period immediately following the initial calibration sequence, the instrument blank and the PEM that are the last two steps in the initial calibration sequence bracket the front end of that 12-hour period. The injection of the instrument blank starts the beginning of the 12-hour period (Section 10.2.2.1). Samples [including Laboratory Control Samples (LCSs) and matrix spike and matrix spike duplicate (MS/MSD), if required] and required blanks (method/sulfur clean-up) may be injected for 12 hours from the injection of the instrument blank. The first three injections immediately after that 12-hour period must be an instrument blank, Individual Standard Mixture A, and Individual Standard Mixture B. The instrument blank must be analyzed first, before either standard. The Individual Standard Mixtures may be analyzed in either order (A,B or B,A).

9.3.2.3 The analyses of the instrument blank and Individual Standard Mixtures A and B immediately following one 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the technical acceptance criteria in Section 9.3.5. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and a PEM, in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks, PEMs, or Individual Standard Mixtures fails to meet the technical acceptance criteria in Section 9.3.5. The 12-hour time period begins with the injection of the instrument blank.

9.3.2.4 Standards (PEM or Individual Standard Mixtures), samples and required blanks may be injected for 12 hours from the time of injection of the instrument blank.

9.3.2.5 If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an acceptable instrument blank and PEM must be analyzed in order to start a new sequence. This requirement applies even if no analyses were performed since that standard was injected.

9.3.2.6 The requirements for running the instrument blanks, PEM, and Individual Standard Mixtures A and B are waived when no samples (including Laboratory Control Samples, requested matrix spike and matrix spike duplicate) dilutions, re-analyses, required blanks (method/sulfur clean-up), and multicomponent analytes for the 72-hour confirmation requirement are analyzed during that 12-hour period. To resume analysis, using the existing initial calibration, the Contractor must first analyze an instrument blank and PEM which meet the technical acceptance criteria.

9.3.2.7 If the entire 12-hour period is not required for the analyses of all samples and blanks to be reported and all data collection is to be stopped, the sequence must be ended with either the instrument blank/PEM combination or the instrument blank/Individual Standard Mixtures A and B combination, whichever was due to be performed at the end of the 12-hour period.

9.3.3 Procedure for Calibration Verification

9.3.3.1 All Standards and blanks must warm to ambient temperature prior to analysis.

9.3.3.2 Analyze the instrument blank, PEM, and the mid point concentration of Individual Standard Mixtures A and B at the required frequencies (Section 9.3.2).

9.3.4 Calculations for Calibration Verification

9.3.4.1 For each analysis of the PEM used to demonstrate calibration verification, calculate the percent difference between the amount of each analyte (including the surrogates) found in the PEM and the nominal amount, using Equations 5 and 9.

9.3.4.2 For each analysis of the PEM used to demonstrate calibration verification, calculate the percent breakdown of Endrin and DDT, and the combined breakdown, using Equations 5, 6, 7, and 8.

9.3.4.3 For each analysis of the mid point concentration of Individual Standard Mixtures A and B used to demonstrate calibration verification, calculate the percent difference between the amount of each analyte (including the surrogates) found in the standard mixture and the nominal amount, using Equations 5 and 9. Do not attempt to calculate the breakdown of Endrin and DDT in the Individual Standard Mixtures, as these standards contain the breakdown products as well as the parent compounds.

9.3.5 Technical Acceptance Criteria For Calibration Verification

All calibration verification technical acceptance criteria apply independently to each GC column.

9.3.5.1 The PEMs, Individual Standard Mixtures, and instrument blanks must be analyzed at the required frequency (Section 9.3.2), on a GC/ECD system that has met the initial calibration technical acceptance criteria.

9.3.5.2 All single component pesticides and surrogates in the PEMs used to demonstrate calibration verification must be greater than or equal to 90.0 percent resolved. The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B used to demonstrate calibration verification must be greater than or equal to 90.0 percent.

9.3.5.3 The absolute RT for each of the single component pesticides and surrogates in the PEMs and mid point concentration of the Individual Standard Mixtures used to demonstrate calibration verification must be within the RT windows determined from the three-point initial calibration in Section 9.2.4.3.

9.3.5.4 The percent difference of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM and midpoint concentration of the Individual Standard Mixtures used to demonstrate calibration verification must be greater than or equal to -25.0 percent and less than or equal to 25.0 percent.

9.3.5.5 The percent breakdown of 4,4'-DDT in the PEM must be less than or equal to 20.0 percent on each column. The percent breakdown of Endrin in the PEM must be less than or equal to 20.0 percent on each column. The combined breakdown of DDT and endrin must be less than or equal to 30.0 percent on each column.

9.3.5.6 All instrument blanks must meet the technical acceptance criteria in Section 12.1.4.5.

9.3.5.7 The identification of single component pesticides by gas chromatographic methods is based primarily on RT data. Since the RT of the apex of a peak can only be verified from an on-scale chromatogram, the following requirements must be met for calibration verification to be acceptable:

- The chromatograms that result from the analyses of the PEM and the Individual Standard Mixtures must display the single component analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale;
- For any PEM, Individual Standard Mixture, or blank, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl;
- If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram; and
- If the chromatogram of any standard or blank needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram(s) must be submitted in the data package.

9.3.6 Corrective Action for Calibration Verification

9.3.6.1 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective action to achieve the technical acceptance criteria.

9.3.6.2 Major corrective actions such as replacing the GC column or baking out the detector will require that a new initial calibration be performed that meets the technical acceptance criteria in Section 9.2.5.

9.3.6.3 Minor corrective actions may not require performing a new initial calibration, provided that a new analysis of the standard (PEM or Individual Mixtures) that originally failed the criteria and an associated instrument blank immediately after the corrective action do meet all the technical acceptance criteria.

9.3.6.4 If a PEM or Individual Standard Mixture does not meet the technical acceptance criteria listed above, it must be reinjected immediately. If the second injection of the PEM or Individual Standard Mixture meets the criteria, sample analysis may continue. If the second injection does not meet the criteria, all data collection must be stopped. Appropriate corrective action must be taken, and a new initial calibration sequence must be run before more sample data are collected.

9.3.6.5 If an instrument blank does not meet the technical acceptance criteria listed in Section 12.1.4.5, all data collection must be stopped. Appropriate corrective action must be taken to clean out the system, and an acceptable instrument blank must be analyzed before more sample data are collected.

9.3.6.6 Analysts are reminded that running an instrument blank and a PEM or Individual Standard Mixtures once every 12 hours are the minimum contract requirements. Late eluting peaks may carry over from one injection to the next if highly complex samples are

analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to run instrument blanks and standards more often to avoid discarding data.

9.3.6.7 If a successful instrument blank and PEM cannot be run after an interruption in analysis (Section 9.3.2.6), an acceptable initial calibration must be run before sample data may be collected. All acceptable sample (including Laboratory Control Samples, requested matrix spike and matrix spike duplicate) and required blank (method/sulfur clean-up) analyses must be preceded and followed by acceptable standards and instrument blanks, as described in Section 9.3.2.

9.3.6.8 Calibration verification technical acceptance criteria must be met before any samples (including the Laboratory Control Sample, requested matrix spike and matrix spike duplicate) and required blanks (method/sulfur clean-up) are reported. Any samples and required blanks associated with a calibration verification which did not meet the technical acceptance criteria will require re-analysis at no additional cost to USEPA.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 This method is designed for analysis of water samples that contain low concentrations of the pesticides and Aroclors listed in Exhibit C. The majority of the samples are expected to come from drinking water and well/ground water type sources around Superfund sites. If, upon inspection of a sample, the Contractor suspects that the sample is not amenable to this method, contact Sample Management Office (SMO). SMO will contact the Region for instructions.

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

10.1.3 Extraction of Samples

Water samples may be extracted by either separatory funnel procedure or a continuous liquid-liquid extraction procedure. If an emulsion prevents acceptable solvent recovery with the separatory funnel procedure, continuous liquid-liquid extraction must be employed. Allow the samples to come to ambient temperature (approximately one hour).

10.1.3.1 Separatory Funnel Extraction

10.1.3.1.1 Measure out each 1 L sample aliquot in a separate graduated cylinder. Measure and record the pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring pH adjustment must be noted in the SDG narrative. Place the sample aliquot into a 2 L separatory funnel.

10.1.3.1.2 Using a syringe or a volumetric pipet add 200 microliter (µL) of the surrogate solution (Section 7.2.3.1) to all water samples.

10.1.3.1.3 Rinse the graduated cylinder with 30 milliliters (mL) of methylene chloride and transfer the rinsate to the separatory funnel. If the sample container is empty, rinse the container with 30 mL of methylene chloride and add the rinsate to the separatory funnel. If the sample container is not rinsed, then add another 30 mL of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure.

NOTE: The total volume of solvent used for extraction is 60 mL. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration through glass wool, centrifugation or other physical means. Drain the methylene chloride into a 250 mL Erlenmeyer flask.

10.1.3.1.4 Add a second 60 mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.1.3.2 Continuous Liquid-Liquid Extraction

10.1.3.2.1 Continuous Liquid-Liquid Extraction Without Hydrophobic Membrane

10.1.3.2.1.1 Follow manufacturer's instructions for set-up.

10.1.3.2.1.2 Add methylene chloride to the bottom of the extractor and fill it to a depth of at least one inch above the bottom sidearm.

10.1.3.2.1.3 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder; transfer the aliquot to the continuous extractor. Measure the pH of the sample with wide range pH paper or pH meter and record pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, as required. Samples requiring the pH adjustment must be noted in the SDG Narrative.

NOTE: With some samples, it may be necessary to place a layer of glass wool between the methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.3.2.1.4 Using a syringe or volumetric pipet, add 200 µL of the surrogate standard spiking solution (Section 7.2.3.1) into the sample and mix well.

10.1.3.2.1.5 Rinse the graduated cylinder with 50 mL of methylene chloride and transfer the rinsate to the continuous extractor. If the sample container is empty, rinse the container with 50 mL of methylene chloride and add the rinsate to the continuous extractor.

10.1.3.2.1.6 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5 to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a <u>minimum</u> of 18 hours.

NOTE 1: When a minimum drip rate of 10-15 mLs/minute is maintained throughout the extraction, the extraction time may be reduced to a minimum of 12 hours. Allow to cool, then detach the distillation flask. Proceed to Section 10.1.4.

NOTE 2: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.3.2.2 Continuous Liquid-Liquid Extraction With Hydrophobic Membrane

10.1.3.2.2.1 Follow the manufacturer's instructions for set-up.

10.1.3.2.2.2 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder. If the sample container is empty, rinse the container with 50 mL of methylene chloride and add the rinsate to the continuous extractor. If the sample was not received in a 1 L container, add 50 mL of methylene chloride to the continuous extractor. Slowly transfer the aliquot to the continuous extractor. Measure the pH of the sample with wide range pH paper or pH meter and record pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, as required. Samples requiring the pH adjustment must be noted in the SDG Narrative.

10.1.3.2.2.3 Using a syringe or volumetric pipet, add 200 µL of the surrogate standard spiking solution (Section 7.2.3.1) into the sample and mix well.

10.1.3.2.2.4 Rinse the graduated cylinder with 50 mL of methylene chloride and transfer the rinsate to the continuous extractor.

10.1.3.2.2.5 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours.

NOTE 1: Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion which will consume the solvent volume, preventing the efficient extraction of the sample. When this occurs, add additional solvent to ensure efficient extraction of the sample, and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used.

Allow to cool, then detach the distillation flask. Proceed to Section 10.1.4.

NOTE 2: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane, it may not be necessary to dry the extract with sodium sulfate.

NOTE 3: If low surrogate recoveries occur, assure 1) the apparatus was properly assembled to prevent leaks; 2) the drip rate/solvent cycling was optimized; and 3) there was proper cooling for condensation of solvent. Document the problem and the corrective action.

NOTE 4: Alternate continuous liquid-liquid extractor types that meet the requirements of the SOW may also be used. If using alternate extractors or design types, follow the manufacturer's instructions for set-up. Optimize the extraction procedure.

10.1.4 Extract Drying and Concentration

10.1.4.1 Assemble a Kuderna-Danish (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 pesticides and Aroclors listed in Exhibit C.

10.1.4.1 Pour the extract through a drying column containing about 10 centimeters (cm) of anhydrous granular sodium sulfate and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and the sodium sulfate with at least two additional 20 to 30 mL portions of methylene chloride to complete the quantitative transfer.

10.1.4.3 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-90°C recommended) 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 15 to 30 minutes. 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 3 to 5 mL, remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY. Proceed with the solvent exchange to hexane.

10.1.4.4 Solvent Exchange to Hexane

10.1.4.4.1 Momentarily remove the three-ball Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Pre-wet the column by adding about 1 mL of hexane to the top. Concentrate the solvent extract as before. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.1.4.4.2 Remove the Snyder column; using 1 to 2 mL of hexane, rinse the flask and its lower joint into the concentrator tube.

10.1.4.4.3 Use the micro Snyder column or the nitrogen blowdown technique to concentrate the hexane extract to 2.0 mL.

10.1.4.5 Final Concentration of Extract

Two different techniques are permitted to concentrate the extract to 2.0 mL. They are the micro Snyder column and nitrogen evaporation techniques.

10.1.4.5.1 Micro Snyder Column Technique

Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Pre-wet the Snyder column by adding about 0.5 mL of hexane to the top of the column. Place the K-D apparatus in a hot water bath (80°C to 90°C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. 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 about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse its flask and lower joint into the concentrator tube with 0.2 mL of hexane. Adjust the final volume with hexane to 2.0 mL.

10.1.4.5.2 Nitrogen Evaporation Technique (taken from ASTM Method D 3086).

10.1.4.5.2.1 Place the concentrator tube with an open micro Snyder column attached in a warm water bath (30°C to 35°C recommended) and evaporate the solvent volume to just below 1 or 2 mL by blowing a gentle stream of clean, dry nitrogen filtered through a column of activated carbon above the solvent. Adjust the final volume with hexane to 1.0 mL (Florisil cartridge check) or 2.0 mL (sample extract).

10.1.4.5.2.2 **CAUTION:** Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or PTFE tubing. Plastic tubing must not be used between the carbon trap and the sample as it may introduce interferences. The internal wall of new tubing must be rinsed several times with hexane and then dried prior to use.

10.1.4.5.2.3 During evaporation, the tube solvent level must be kept below the water level of the bath. DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS.

10.1.4.6 Proceed to Section 10.1.5 for extract cleanup. Otherwise, transfer the extract to a PTFE-lined screw-cap bottle and label the bottle. Store at 4°C (±2°C) but not greater than 6°C.

10.1.5 Extract Cleanup

10.1.5.1 The two cleanup procedures specified in this method are Florisil cartridge and sulfur cleanup. Florisil cartridge cleanup is required for all extracts. Sulfur cleanup must be performed on all extracts containing sulfur at levels that interfere with Gas Chromatograph/Electron Capture Detector (GC/ECD) analysis. Sulfur contamination in a sample analysis is unacceptable. Method blanks must be subjected to the same cleanup procedures as the samples.

10.1.5.2 Florisil Cleanup

10.1.5.2.1 Introduction

Florisil cartridge cleanup significantly reduces matrix interference caused by polar compounds and is required for all extracts. The same volume of the concentrated extract taken for Florisil cleanup must be maintained after Florisil cleanup (2 mL).

10.1.5.2.2 Florisil Cartridge Performance Check

10.1.5.2.2.1 Summary of Florisil Cartridge Performance Check

Every lot number of Florisil cartridges must be tested before they are used for sample cleanup.

10.1.5.2.2.2 Frequency of Florisil Cartridge Performance Check

Cartridge performance check must be conducted at least once on each lot of cartridges used for sample cleanup or every 6 months whichever is most frequent.

10.1.5.2.2.3 Procedure for Florisil Cartridge Performance Check

Add 0.5 mL of 2,4,5-trichlorophenol solution (0.1 µg/mL in acetone, Section 7.2.3.7) and 0.5 mL of Standard Mixture A, midpoint concentration, Section 7.2.3.5) to 4 mL of hexane. Reduce the volume to 0.5 mL using nitrogen (Section 10.1.4.5.2). Place the mixture onto the top of a washed Florisil cartridge, and elute it with 9 mL of hexane/acetone [(90:10)(V/V)]. Use two additional 1 mL hexane rinses to ensure quantitative transfer of standard from the cartridge. Concentrate to a final volume of 1 mL and analyze the solution by GC/ECD using at least one of the GC columns specified for sample analysis. Determine the recovery of each analyte for evaluation and reporting purposes. Calculate the percent recovery using Equation 11.

EQ. 11

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} * 100$$

Where:

Q_d = Quantity determined by analysis.

Q_a = Quantity added.

10.1.5.2.2.4 Technical Acceptance Criteria for Florisil Cartridge Performance Check

10.1.5.2.2.4.1 The cartridge performance check solution must be analyzed on a GC/ECD meeting the initial calibration and calibration verification technical acceptance criteria.

10.1.5.2.2.4.2 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 120 percent, if the recovery of trichlorophenol is less than 5 percent, and if no peaks interfering with the target analytes are detected.

10.1.5.2.2.5 Corrective Action for Florisil Cartridge Performance Check

Any lot of Florisil cartridges that does not meet the criteria above must be discarded and a new lot, meeting criteria, used for sample cleanup.

10.1.5.2.3 Sample Cleanup by Florisil Cartridge

The required Florisil cartridge size is a 1 g cartridge and the final volume of the extract after Florisil cleanup is 2 mL.

10.1.5.2.3.1 Frequency of Sample Cleanup by Florisil Cartridge

All sample extracts (including Laboratory Control Samples and requested matrix spike and matrix spike duplicate) and method blank extracts are required to be cleaned up by the Florisil cartridge technique.

10.1.5.2.3.2 Procedure for Sample Cleanup by Florisil Cartridge

10.1.5.2.3.2.1 Attach the vacuum manifold to a water aspirator or to a vacuum pump with a trap installed between the manifold and the vacuum source. Adjust the vacuum pressure in the manifold to between 5 and 10 pounds of vacuum.

10.1.5.2.3.2.2 Place one Florisil cartridge into the vacuum manifold for each sample extract.

10.1.5.2.3.2.3 Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge on the vacuum manifold, by pulling a vacuum, and by passing at least 5 mL of the hexane/acetone solution through the cartridge. While the cartridges are being washed, adjust the vacuum applied to each cartridge so that the flow rate through each cartridge is approximately equal. DO NOT ALLOW THE CARTRIDGES TO GO DRY AFTER THEY HAVE BEEN WASHED.

10.1.5.2.3.2.4 After the cartridges on the manifold are washed, the vacuum is released, and a rack containing labeled 10 mL volumetric flasks is placed inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside of the appropriate volumetric flask as the manifold top is replaced.

10.1.5.2.3.2.5 After the volumetric flasks are in place, the vacuum to the manifold is restored, and a volume of extract equal to the required final volume from each sample and method blank extract is transferred to the top frit of the appropriate Florisil cartridge. This must equal the final volume after Florisil cleanup.

10.1.5.2.3.2.6 Because the volumes marked on concentrator tubes are not necessarily accurate at the 1 mL level, the use of a syringe or a volumetric pipet is required to transfer the extract to the cleanup cartridge.

10.1.5.2.3.2.7 The pesticides/Aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL volumetric flasks held in the rack inside the vacuum manifold.

10.1.5.2.3.2.8 Transfer the eluate in each volumetric flask to a clean centrifuge tube or 10 mL vial. Use two additional 1 mL hexane rinses to ensure quantitative transfer of the cartridge eluate.

10.1.5.2.3.2.9 Adjust the extract to 2 mL aliquot volume as was taken for cleanup using either nitrogen blowdown or a micro Snyder column (Section 10.1.4.5). Measure the final volume with a syringe or by transferring the extract to a volumetric flask.

10.1.5.2.3.2.10 If sulfur cleanup is to be performed, proceed to Section 10.1.5.3.3. Otherwise, transfer the sample to a GC vial and label the vial. The extract is ready for GC/ECD analysis.

10.1.5.3 Sulfur Cleanup

10.1.5.3.1 Introduction

10.1.5.3.1.1 Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analyses of the later eluting pesticides. If crystals of sulfur are evident or if the presence of sulfur is suspected, sulfur removal must be performed. Interference which is due to sulfur is not acceptable. Sulfur can be removed by one of two methods, according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and remove the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean centrifuge tube or clean concentrator tube before proceeding with further sulfur cleanup.

10.1.5.3.1.2 If only part of a set of samples require sulfur cleanup, then, a sulfur cleanup blank is required for that part of the set (Section 12.1.3.2).

10.1.5.3.2 Frequency of Sulfur Cleanup

Sulfur removal is required for all sample extracts that contain sulfur.

10.1.5.3.3 Procedure for Sulfur Cleanup

10.1.5.3.3.1 Mercury Technique

Add one to three drops of mercury to each hexane extract in a clean vial. Tighten the top on the vial and agitate the sample for 30 seconds. Filter or centrifuge the extract. Pipet the extract to another vial and leave all solid precipitate and liquid mercury. If the mercury appears shiny, proceed to Section 10.2 and analyze the extract. If the mercury turns black, repeat sulfur removal as necessary. The extract transferred to the vial still represents the 2.0 mL final volume. CAUTION: Waste containing mercury should be segregated and disposed of properly.

NOTE: Mercury is a highly toxic metal and therefore must be used with great care. Prior to using mercury, it is recommended that the analyst become acquainted with proper handling and cleanup techniques associated with this metal.

10.1.5.3.3.2 Copper Technique

Add approximately 2 grams (g) of cleaned copper powder to the extract in the centrifuge or concentrator tube (2 g will fill the tube to about the 0.5 mL mark). Mix the copper and extract for at least 1 minute on a mechanical shaker. Separate the extract from the copper powder by drawing off the extract with a disposable pipet, and transfer the extract to a clean vial. The extract transferred to the

vial still represents the 2.0 mL final volume. The separation of the extract from the copper powder is necessary to prevent degradation of the pesticides. If the copper appears bright, proceed to Section 10.2 and analyze the extracts. If the copper changes color, repeat the sulfur removal procedure as necessary.

10.2 GC Analysis

10.2.1 Introduction

10.2.1.1 Before samples (including Laboratory Control Samples and requested matrix spike and matrix spike duplicate) and required blanks (method/sulfur clean-up) can be analyzed, the instrument must meet the initial calibration and calibration verification technical acceptance criteria. All sample extracts, required blanks, and calibration standards must be analyzed under the same instrumental conditions. All sample extracts, required blank extracts, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before preparation/analysis. Sample analysis on both GC columns is required for all samples and blanks.

10.2.1.2 Set up the GC/ECD system per the requirements in Section 9.1. Unless ambient temperature on-column injection is used (Section 9.1), the injector must be heated to at least 200°C. The optimized gas chromatographic conditions from Section 9.1 must be used.

10.2.2 Procedure for Sample Analysis by GC/ECD

The injection must be made on-column by using either automatic or manual injection. If autoinjectors are used, 1.0 µL injection volumes may be used. Manual injections must use at least 2.0 µL injection volumes. The same injection volume must be used for all standards, samples, and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2 µL. However, the same injection volume must be used for all analyses.

10.2.2.1 Analytical Sequence

All samples and required blanks must be analyzed within a valid analysis sequence as given below.

Time	Injection #	Material Injected
	1-15	First 15 steps of the initial calibration
0 hr.	16	Instrument blank at end of initial calibration
	17	PEM at end of initial calibration
	18	First sample
	0	
	0	Subsequent samples
	0	
12 hr.	0	Last sample

Time	Injection #	Material Injected
	1st injection past 12:00 hr.	Instrument blank
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	0	Sample
	0	
	0	Subsequent samples
	0	
	0	
Another 12 hr.	0	Last sample
	1st injection past 12 hr.	Instrument blank
	2nd injection	PEM
	0	Sample
	0	
	0	Subsequent samples
	0	
	0	
Another 12 hr.	0	Last sample
	1st injection past 12:00 hr.	Instrument blank
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	0	Sample
	0	
	0	Subsequent samples
	0	
	0	
	etc.	

10.2.2.1.1 The first 12 hours are counted from injection #16 (the Instrument Blank at the end of the initial calibration sequence), not from injection #1. Samples and required blanks may be injected until 12:00 hours have elapsed. All subsequent 12-hour periods are timed from the injection of the instrument blank that brackets the front end of the samples. Because the 12-hour time period is timed from injection of the instrument blank until the injection of the last sample, each 12-hour period may be separated by the length of one chromatographic run, that of the analysis of the last sample. While the 12-hour period may not be exceeded, the laboratory may run instrument blanks and standards more frequently, for instance, to accommodate staff working on 8-hour shifts.

10.2.2.1.2 After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks, PEMs and Individual Standard Mixtures A and B are analyzed at the required frequency. This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the Contractor; however, the blanks and standards must also satisfy the

criteria presented in Section 9 in order to continue the run sequence.

10.2.2.1.3 An analysis sequence must also include all samples and required blank analyses, but the Contractor may decide at what point in the sequence they are to be analyzed.

10.2.2.1.4 The requirements for the analysis sequence apply to each GC column and for all instruments used for these analyses.

10.2.3 Sample Dilutions

10.2.3.1 The sample must first be analyzed at the most concentrated level (injection taken from the 2.0 mL final extract after the clean-up steps).

10.2.3.2 If the concentration of any single component pesticide is greater than the upper limit of the initial calibration range on both GC columns, then the extract must be diluted. If the concentration of any single component pesticide is greater than the upper limit of the initial calibration range on one GC column, but not the other, then the extract must be diluted only if the percent difference between the two concentrations is less than or equal to 25%. The on-column concentration of the pesticide compound(s) in the diluted extract must be between the initial calibration low point and high point standards.

10.2.3.3 If the calculated concentration of any multicomponent peak, used for quantitation is greater than the concentration of the most intense single component analyte in the initial calibration high point standard, then the sample must be diluted to have the concentration of the largest peak in the multicomponent analyte between the initial calibration midpoint and high point standards of that single component pesticide.

10.2.3.4 If dilution is employed solely to bring a peak within the calibration range or to get a multicomponent pattern on scale, the results for both the more and the less concentrated extracts must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

10.2.3.5 If the dilution factor is greater than 10, an additional extract 10 times more concentrated than the diluted sample extract must be analyzed and reported with the sample data. If the dilution factor is less than 10, but greater than 1, the results of the original undiluted analysis must also be reported.

10.2.3.6 If the analysis of the most concentrated extract does not meet the requirement for dilution in Section 10.2.3.2 and 10.2.3.3, then the analysis is at no additional cost to USEPA.

10.2.3.7 When diluted, the chromatographic data for the single component pesticide must be able to be reported at greater than 10 percent of full scale but less than 100 percent of full scale.

10.2.3.8 When diluted, multicomponent analytes must be able to be reported at greater than 25 percent of full scale but less than 100 percent of full scale.

10.2.3.9 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram. If the chromatogram of any sample needs to be replotted electronically to meet these requirements, both the

initial chromatogram and the replotted chromatogram(s) must be submitted in the data package.

10.2.3.10 Dilute the sample using the following procedure:

10.2.3.10.1 Calculate the extract dilution in order for the single component pesticides to meet the requirement listed in Section 10.2.3.7.

10.2.3.10.2 Calculate the extract dilution in order for the multicomponent analytes to meet the requirement listed in Section 10.2.3.8.

10.2.3.10.3 Dilute the sample extract quantitatively with hexane.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification of Target Compounds

11.1.1 The laboratory will identify single component analyte peaks based on the Retention Time (RT) windows established during the initial calibration sequence. Single component analytes are identified when peaks are observed in the RT window for the analyte on both Gas Chromatograph (GC) columns.

11.1.2 A set of three to five major peaks is selected for each multicomponent analyte. RT windows for each peak are determined from the initial calibration analysis. Identification of a multicomponent analyte in the sample is based on pattern recognition in conjunction with the elution of three to five sample peaks within the RT window of the corresponding peaks of the standard on both GC columns. The number of potential quantitation peaks is listed in Table D-2.

11.1.3 A standard of any identified multicomponent analyte must be run within 72 hours of its detection in a sample chromatogram within a valid 12-hour sequence.

11.1.4 The choice of the peaks used for multicomponent analyte identification and the recognition of those peaks may be complicated by the environmental alteration of the toxaphene or Aroclors, and by the presence of coeluting analytes, or matrix interferences, or both. Because of the alteration of these materials in the environment, multicomponent analytes in samples may give patterns similar to, but not identical with, those of the standards.

11.1.5 Toxaphene and Aroclors require only a single-point calibration. Identification requires visual inspection of an on-scale pattern.

11.2 Calculations

11.2.1 Target Compounds

11.2.1.1 Quantitation for all analytes and surrogates must be performed and reported for each GC column.

11.2.1.2 Manual integration of peaks (e.g., measuring peak height with a ruler) is only permitted when accurate electronic integration of peaks cannot be done. If manual integration of peaks is required, it must be documented in the Sample Delivery Group (SDG) Narrative.

11.2.1.3 The Contractor must quantitate each single component analyte and surrogate based on the calibration factor from the most recent initial calibration midpoint standard mixture analyses. Do not use the analyses of the Individual Standard Mixtures used to demonstrate calibration verification for quantitation of samples.

11.2.1.4 The Contractor must quantitate each multicomponent analyte based on the calibration factor from the most recent initial calibration standard.

11.2.1.5 If more than one multicomponent analyte is present, the Contractor must choose separate peaks to quantitate the different multicomponent analytes. A peak common to both analytes present in the sample must not be used to quantitate either analyte.

11.2.1.6 Before reporting data to USEPA, it is required that the Contractor check for flags generated by the data system that indicate improper quantitation of analytes.

11.2.1.7 The chromatograms of all samples (including Laboratory Control Samples, requested matrix spike/matrix spike duplicate) standards, and required blanks must be reviewed by a qualified pesticide analyst before they are reported.

11.2.1.8 Calculate the concentration of the single component pesticides and surrogates by using the following equation:

EQ. 12

$$\text{Concentration } \mu g/L = \frac{(A_x)(V_t)(Df)}{(CF_{mp})(V_i)(V_x)}$$

Where:

A_x = Response (peak area or height) of the compound to be measured.

CF_{mp} = Calibration factor for the mid-point from initial calibration standard (area per ng).

V_t = Volume of concentrated extract (µL). (This volume is 2000 µL).

V_i = Volume of extract injected (uL). (If a single injection is made onto two columns, use one-half the volume in the syringe as the volume injected onto each column).

V_x = Volume of water extracted (mL). (NOTE: for instrument blanks and sulfur cleanup blanks, assume a 1,000 mL volume).

Df = Dilution factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\text{µL most concentrated extract used to make dilution + µL clean solvent}}{\text{µL most concentrated extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The calibration factors used in Equation 12 are those from the most recent mid-point standard from the initial calibration. If the calibration factors used to determine the linearity of the initial calibration were based on peak area, then the concentration of the analyte in the sample must be based on peak area. Similarly, if peak height was used to determine linearity, use peak height to determine the concentration in the sample.

11.2.1.9 Contract Required Quantitation Limit (CRQL) Calculation

If the adjusted CRQL is less than the CRQL listed in Exhibit C (Pesticides), report the CRQL in Exhibit C (Pesticides). Calculate the adjusted CRQL for pesticides by using the following equation:

EQ. 13

$$\text{Adjusted CRQL} = \text{Contract CRQL} * \frac{\text{Contract Sample Vol (1000 mL)}}{V_x} * Df * \frac{Vt}{\text{Contract Ext. Vol (2000uL)}}$$

Where:

Contract CRQL = Take exact CRQL values reported in Exhibit C of the SOW.

V_x = Same as EQ. 12.

V_t = Same as EQ. 12.

Df = Same as EQ. 12.

11.2.1.10 During initial calibration, a set of three to five quantitation peaks was chosen for each multicomponent analyte. Calculate the concentration of each of the selected Aroclor or toxaphene peaks individually using Equation 12. Determine the mean concentration for all of the selected peaks. The mean value is reported on Form X (Exhibit B) for both GC columns.

11.2.1.11 For the single component pesticides, report the lower of the two values quantitated from the two GC columns of Form I. For the multicomponent analytes, report the lower of the two mean values from the two GC columns on Form I.

11.2.1.12 The percent difference is calculated according to Equation 14.

EQ. 14

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} * 100$$

Where:

$Conc_H$ = The higher of the two concentrations for the target compound in question.

$Conc_L$ = The lower of the two concentrations for the target compound in question.

NOTE: Using this equation will result in percent difference values that are always positive. The value will also be greater than a value calculated using the higher concentration in the denominator, however, given the likelihood of a positive interference raising the concentration determined on one GC column, this is a conservative approach to comparing the two concentrations.

11.2.2 Surrogate Recoveries

11.2.2.1 The concentrations of the surrogates are calculated separately for each GC column in a similar manner as the other analytes using Equation 12. Use the calibration factors from the midpoint concentration of Individual Standard Mixture A from the initial calibration.

11.2.2.2 The recoveries of the surrogates are calculated according to Equation 15.

EQ. 15

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} * 100$$

Where:

Q_d = Quantity determined by analysis.

Q_a = Quantity added to sample/blank.

11.3 Technical Acceptance Criteria for Sample Analyses

The requirements below apply independently to each GC column and to all instruments used for these analyses. (See exception in Section 11.3.7) Quantitation must be performed on each GC column.

11.3.1 Samples must be analyzed under the Gas Chromatograph/Electron Capture Detector (GC/ECD) operating conditions in Section 9.1. The instrument must have met all initial calibration and calibration verification technical acceptance criteria. Samples must be cleaned-up using Florisil that meets the technical acceptance criteria for Florisil. Sample data must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, Performance Evaluation Mixtures (PEMs), and Individual Standard Mixtures A and B, as described in Section 10.2.2.1.

11.3.2 The sample must be extracted and analyzed within the contract holding times.

11.3.3 The Laboratory Control Sample (LCS) associated with the samples must meet the LCS technical acceptance criteria.

11.3.4 The samples must have an associated method blank meeting the method blank technical acceptance criteria. If a sulfur cleanup blank is associated with the samples, that blank must meet the sulfur cleanup blank technical acceptance criteria.

11.3.5 The RT for each of the surrogates must be within the RT window as calculated in Section 9.2.4.3, for both GC columns.

11.3.6 The percent recovery for the surrogates must be between 30.0 and 150 percent, inclusive. These limits are <u>not</u> advisory.

NOTE: The surrogate recovery requirements do not apply to a sample that has been diluted.

11.3.7 No target analyte concentrations may exceed the upper limit of the initial calibration or else extracts must be diluted and re-analyzed. If a target analyte concentration exceeds the upper limit of the initial calibration on one GC column, but not the other, the extract must be diluted and re-analyzed only if the percent difference between the two concentrations is less than or equal to 25%.

11.3.8 A standard for any identified multicomponent analyte must be analyzed on the same instrument within 72 hours of its detection in a sample within a valid 12 hour sequence.

11.3.9 The identification of single component pesticides by gas chromatographic methods is based primarily on RT data. The RT of the apex of a peak can only be verified from an on-scale chromatogram. The identification of multicomponent analytes by gas chromatographic methods is based primarily on recognition of patterns of RTs displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.

11.3.9.1 When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low point standard of the initial calibration associated with those analyses.

11.3.9.2 Chromatograms must display single component pesticides detected in the sample at less than full scale.

11.3.9.3 Chromatograms must display the largest peak of any multicomponent analyte detected in the sample at less than full scale.

11.3.9.4 If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale.

11.3.9.5 If an extract must be diluted, chromatograms must display multicomponent analytes between 25 and 100 percent of full scale.

11.3.9.6 For any sample or blank, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

11.3.9.7 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

11.3.9.8 If the chromatogram of any sample needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram(s) must be submitted in the data package.

11.4 Corrective Action for Sample Analysis

11.4.1 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 sulfur cleanup blank will require re-extraction and re-analysis at no additional cost to USEPA. Any samples analyzed that do not meet the technical acceptance criteria will require re-extraction and/or re-analysis at no additional cost to USEPA.

11.4.2 If the sample analysis technical acceptance criteria are not met, check calculations, surrogate solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria, in which case, the affected samples must be re-analyzed at no additional cost to USEPA after the corrective action.

11.4.3 If the Contractor needs to analyze more than the most concentrated extract and two (2) sample dilutions to have all the pesticide/Aroclor compounds within the calibration range of the instrument, contact Sample Management Office (SMO). SMO will contact the Region for instructions.

11.4.4 Sample re-extraction/re-analyses 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 USEPA Contract Laboratory Program Project Officer (CLP PO). Send a copy of the SDG Narrative (including your contract number), a description of the situation, and the requested action to the CLP PO.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Introduction

There are two types of blanks required by this method: the method blank and the instrument blank. A separate sulfur cleanup blank may also be required if some, but not all of the samples are subjected to sulfur cleanup. Samples that are associated with a sulfur cleanup blank are also associated with the method blank with which they were extracted. Both the method and sulfur cleanup blanks must meet the respective technical acceptance criteria for the sample analysis technical acceptance criteria to be met.

12.1.2 Method Blank

12.1.2.1 Summary of Method Blank

A method blank is 1.0 liter of reagent water carried through the entire analytical scheme. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.2.2 Frequency of Method Blank

A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples (excluding matrix spike/matrix spike duplicate, PE samples, and Laboratory Control Samples). In addition, a method blank shall:

- Be extracted by the same procedure used to extract samples; and
- Be analyzed on each Gas Chromatograph/Electron Capture (GC/ECD) system used to analyze associated samples.

12.1.2.3 Procedure for Method Blank

Measure 1.0 liter of reagent water for each method blank aliquot. Add 200 µL of the surrogate solution (Section 7.2.3.1). Extract, concentrate and analyze the method blank according to Section 10.

12.1.2.4 Calculations for Method Blank

Calculate method blank results according to Section 11.

12.1.2.5 Technical Acceptance Criteria for Method Blank

12.1.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.1.2.5.2 All method blanks must be prepared and analyzed at the frequency described in Section 12.1.2.2, using the procedure in Section 12.1.2.3 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria. Method blanks must be cleaned-up using Florisil meeting the technical acceptance criteria for florisil.

12.1.2.5.3 Method blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and

individual Standard Mixtures A and B as described in Section 10.2.2.1.

12.1.2.5.4 The concentration of the target compounds (Exhibit C - Pesticides)in the method blank must be less than the Contract Required Quantitation Limit (CRQL) for each target compound.

12.1.2.5.5 The method blank must meet all sample technical acceptance criteria in Sections 11.3.5 to 11.3.9.

12.1.2.5.6 Surrogate recoveries must fall within the acceptance window of 30-150 percent. These limits are not advisory.

12.1.2.6 Corrective Action for Method Blank

12.1.2.6.1 If a method blank does not meet the technical acceptance criteria the Contractor must consider the analytical system to be out of control.

12.1.2.6.2 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. Further, all samples (including Laboratory Control Samples, requested matrix spike/matrix spike duplicate, and PE samples) processed with a method blank that does not meet the method blank technical acceptance criteria (i.e., contaminated) will require re-extraction and re-analysis at no additional cost to USEPA. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.2.6.3 If surrogate recoveries in the method blank do not meet the technical acceptance criteria, listed in 12.1.2.5.6, first re-analyze the method blank. If the surrogate recoveries do not meet the technical acceptance criteria after re-analysis, then the method blank and all samples (including Laboratory Control Samples, requested matrix spike/matrix spike duplicate, and PE samples) associated with that method blank must be re-extracted and re-analyzed at no additional cost to USEPA.

12.1.2.6.4 If the method blank fails to meet a technical acceptance criteria other than Sections 12.1.2.5.4 and 12.1.2.5.6, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary) and re-analyze the method blank.

12.1.3 Sulfur Cleanup Blank

12.1.3.1 Summary of Sulfur Cleanup Blank

The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and passed through the sulfur cleanup and analysis procedures. The purpose of the sulfur cleanup is to determine the levels of contamination associated with the separate sulfur cleanup steps.

12.1.3.2 Frequency of Sulfur Cleanup Blank

The sulfur cleanup blank is prepared when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur

cleanup blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then no separate sulfur cleanup blank is required.

12.1.3.3 Procedure for Sulfur Cleanup Blank

The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. The sulfur blank must also contain the surrogates at the same concentrations as the sample extracts (assuming 100.0 percent recovery). Therefore, add 0.2 milliliters (mL) of the surrogate spiking solution (Section 7.2.3.1) to 1.8 mL of hexane in a clean vial.

12.1.3.3.2 Proceed with the sulfur removal (Section 10.1.5.3) using the same technique (mercury or copper) as the samples associated with the blank.

12.1.3.3.3 Analyze the sulfur blank according to Section 10.2.

12.1.3.4 Calculations for Sulfur Cleanup Blank

12.1.3.4.1 Assuming that the material in the sulfur blank resulted from the extraction of a 1 L water sample, calculate the concentration of each analyte using Equation 12 in Section 11.2.1.8. Compare the results to the CRQL values in Exhibit C (Pesticides).

12.1.3.4.2 See Section 11.2 for the equations for the other calculations.

12.1.3.5 Technical Acceptance Criteria for Sulfur Cleanup Blanks

12.1.3.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each column.

12.1.3.5.2 All sulfur cleanup blanks must be prepared and analyzed at the frequency described in Section 12.1.3.2 using the procedure in Section 12.1.3.3 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

12.1.3.5.3 Sulfur cleanup blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs and Individual Standard Mixtures A and B, as described in Section 10.2.2.1.

12.1.3.5.4 The concentration of the target compounds (Exhibit C - Pesticides) in the sulfur cleanup blank must be less than the CRQL for each target compound.

12.1.3.5.5 The sulfur cleanup blank must meet all sample technical acceptance criteria in Sections 11.3.5 to 11.3.9.

12.1.3.5.6 Surrogate recoveries must fall within the acceptance windows of 30-150 percent.

12.1.3.6 Corrective Action for Sulfur Cleanup Blank

12.1.3.6.1 If a sulfur cleanup blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.3.6.2 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. Further, all samples processed with a sulfur cleanup blank that does not meet the sulfur cleanup blank technical acceptance criteria (i.e., contaminated) will require re-extraction and re-analysis at no additional cost to USEPA. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.3.6.3 If surrogate recoveries in the sulfur cleanup blank do not meet the technical acceptance criteria, in Section 12.1.3.5.6, first re-analyze the sulfur cleanup blank. If the surrogate recoveries do not meet the technical acceptance criteria after re-analysis, then the sulfur cleanup blank and all samples associated with that sulfur cleanup blank must be re-prepared/re-extracted and re-analyzed at no additional cost to USEPA.

12.1.3.6.4 If the sulfur cleanup blank fails to meet a technical acceptance criteria other than Sections 12.1.3.5.4, and 12.1.3.5.6, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary) and re-analyze the sulfur cleanup blank.

12.1.4 Instrument Blank

12.1.4.1 Summary of Instrument Blank

An instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis particularly with regard to carryover of analytes from standards or highly contaminated samples into other analysis.

12.1.4.2 Frequency of Instrument Blank

The first analysis in a 12-hour analysis sequence (Section 9.3.2) must be an instrument blank. All groups of acceptable sample analyses are to be preceded and followed by acceptable instrument blanks (Section 10.2.2.1). If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an instrument blank must be analyzed to initiate a new 12-hour sequence (Section 9.3.2).

12.1.4.3 Procedure for Instrument Blank

12.1.4.3.1 Prepare the instrument blank by spiking the surrogates into hexane or iso-octane for a concentration of 20.0 nanograms per milliliter (ng/mL) of Tetrachloro-m-xylene and Decachlorobiphenyl.

12.1.4.3.2 Analyze the instrument blank according to Section 10.2 at the frequency listed in Section 12.1.4.2.

12.1.4.4 Calculations for Instrument Blank

12.1.4.4.1 Assuming that the material in the instrument blank resulted from the extraction of a 1 L water sample, calculate the concentration of each analyte using Equation 12 in Section 11.2.1.8. Compare the results to the CRQL values for water samples in Exhibit C (Pesticides).

12.1.4.4.2 See Section 11.2 for the equations for the other calculations.

12.1.4.5 Technical Acceptance Criteria for Instrument Blanks

12.1.4.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.1.4.5.2 All instrument blanks must be prepared and analyzed at the frequency described in Section 12.1.4.2 using the procedure in Section 10.2 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

12.1.4.5.3 The concentration of each target analyte (Exhibit C - Pesticides) in the instrument blank must be less than the CRQL for that analyte.

12.1.4.5.4 The instrument blank must meet all sample technical acceptance criteria in Sections 11.3.5 to 11.3.9.

12.1.4.6 Corrective Action for Instrument Blank

12.1.4.6.1 If analytes are detected at concentrations greater than the CRQL or the surrogate Retention Times (RTs) are outside the RT windows, all data collection must be stopped, and corrective action must be taken. Data for samples which were run between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be run before additional data are collected. All samples (including Laboratory Control Samples, requested matrix spike/matrix spike duplicate, and PE samples) and required blanks which were run after the last acceptable instrument blank must be reinjected during a valid run sequence and must be reported at no additional cost to USEPA.

12.2 Laboratory Control Sample (LCS)

12.2.1 Summary of LCS

The LCS is an internal laboratory quality control sample designed to assess [on a Sample Delivery Group (SDG)-by-SDG basis] the capability of the contractor to perform the analytical method listed in this Exhibit.

12.2.2 Frequency of LCS

The LCS must be prepared, extracted, analyzed, and reported once per Sample Delivery Group. The LCS must be extracted and analyzed concurrently with the samples in the SDG using the same extraction protocol and instrumentation as the samples in the SDG.

12.2.3 Procedure for LCS

Measure a 1 liter aliquot of reagent water in a 1 liter graduated cylinder and transfer the water to a continuous extractor or 2 L separatory funnel. Pipet 1.0 mL of the LCS spiking solution (Section 7.2.3.8) and 200 uL of the surrogate standard spiking solution into the water and mix well. Extract, concentrate, and analyze the sample according to Section 10.

12.2.4 Calculations for LCS

12.2.4.1 Calculate the results according to Section 11.

12.2.4.2 Calculate individual compound recoveries of the LCS using Equation 15.

12.2.5 Technical Acceptance Criteria For LCS

12.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.2.5.2 The LCS must be analyzed at the frequency described in Section 12.2.2 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

12.2.5.3 The LCS must be prepared as described in Section 12.2.3.

12.2.5.4 The LCS must meet all sample technical acceptance criteria in Sections 11.3.5 to 11.3.9.

12.2.5.5 The percent recovery for each of the compounds in the LCS must be within the recovery limits listed in Table D-3.

12.2.6 Corrective Action for LCS

12.2.6.1 If the LCS technical acceptance criteria for the surrogates or the LCS compound recovery are not met, check calculations, the surrogate and LCS solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and LCS recovery criteria.

12.2.6.2 LCS technical acceptance criteria MUST be met before data are reported. LCS contamination from laboratory sources or any LCS analyzed not meeting the technical acceptance criteria will require re-extraction and re-analysis of the LCS at no additional cost to USEPA.

12.2.6.3 All samples (including matrix spike/matrix spike duplicate and PE samples) and required blanks, prepared and analyzed in an SDG with an LCS that does not meet the technical acceptance criteria, will also require re-extraction and re-analysis at no additional cost to USEPA.

12.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

12.3.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the methods used for pesticide/Aroclor analyses, USEPA has prescribed a mixture of pesticide/Aroclor target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

12.3.2 Frequency of MS/MSD Analysis

12.3.2.1 MS/MSD samples shall only be analyzed if requested by the Region [through Sample Management Office (SMO)] or specified on the Traffic Report (TR). If requested, a matrix spike and matrix spike duplicate must be extracted and analyzed for every 20 field samples in an SDG.

12.3.2.2 As part of USEPA's QA/QC program, water rinsate samples and/or field blanks may be delivered to a laboratory for analysis. Do not perform MS/MSD analysis on a water rinsate sample or field blank.

12.3.2.3 If the USEPA Region requesting MS/MSD designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample volume remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify the Region (through SMO) that insufficient sample was received and identify the USEPA sample selected for the MS/MSD analysis. The rationale for the choice of another sample other than the one designated by USEPA shall be documented in the SDG Narrative.

12.3.2.4 If there is insufficient sample volume remaining in any of the samples in an SDG to perform the requested MS/MSD, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no MS/MSD be performed, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative.

12.3.2.5 If it appears that the Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.3.2.1, the Contractor shall contact SMO. SMO will contact the Region to determine which samples should have MS/MSD performed on them. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative. If this procedure is not followed, the Contractor will not be paid for MS/MSD analysis performed at a greater frequency than required by the contract.

12.3.2.6 When a Contractor receives <u>only</u> Performance Evaluation (PE) samples, no MS/MSD shall be performed within that SDG.

12.3.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the requested MS/MSD analysis when the Region did not designate a sample to be used for this purpose.

12.3.3 Procedure for Preparing MS/MSD

12.3.3.1 Measure out two additional 1 L aliquots of the sample chosen for spiking. Adjust the pH of the samples (if required) and fortify each with 1 mL of the matrix spiking solution (Section 7.2.3.2). Using a syringe or volumetric pipet, add 200 uL of the surrogate spiking solution (Section 7.2.3.1) to each sample. Extract, concentrate, cleanup, and analyze the matrix spike and matrix spike duplicate according to Section 10.0.

12.3.3.2 Matrix spike and matrix spike duplicate samples must be analyzed at the same concentration as the most concentrated extract for

which the original sample results will be reported. Do not further dilute the MS/MSD samples to get either spiked or nonspiked analytes within calibration range.

12.3.4 Calculations for MS/MSD

The percent recoveries and the relative percent difference between the recoveries of each of the compounds in the matrix spike and matrix spike duplicate samples will be calculated and reported by using the following equations:

EQ. 16

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} * 100$$

Where:

SSR = Spike sample result.

SR = Sample result.

SA = Spike added.

EQ. 17

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} * 100$$

Where:

RPD = Relative percent difference.

MSR = Matrix spike recovery.

MSDR = Matrix spike duplicate recovery.

12.3.5 Technical Acceptance Criteria for MS/MSD

12.3.5.1 The requirements below apply independently to <u>each</u> GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.3.5.2 If requested, all MS/MSD must be prepared and analyzed at the frequency described in Section 12.3.2, using the procedure above and in Section 10 on a GC/ECD system meeting the initial calibration, calibration verification, and blank technical acceptance criteria. MS/MSD must be cleaned-up using florisil meeting the technical acceptance criteria for florisil. MS/MSD must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and individual standard mixtures A and B as described in Section 10.2.2.1.

12.3.5.3 The samples must be extracted and analyzed within the contract required holding times.

12.3.5.4 The RT for each of the surrogates must be within the RT window as calculated in Section 9 for both GC columns.

12.3.5.5 The limits for matrix spike compound recovery and RPD are given in Table D-4. As these limits are only advisory, no further action by the laboratory is required. However, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questions from USEPA.

12.3.6 Corrective Action for MS/MSD

Any MS/MSD which fails to meet the technical acceptance criteria in Sections 12.3.5.1 through 12.3.5.4 must be re-analyzed at no additional cost to USEPA.

12.4 Method Detection Limit (MDL) Determination

12.4.1 Before any field samples are analyzed under this contract, the MDL for each pesticide target compound shall be determined for each sample extraction procedure and on one of the instruments to be used for sample analysis. The MDLs must be verified annually thereafter (see Section 12.4.2 for MDL verification procedures), until the contract expires or is terminated or after major instrument maintenance. Major instrument maintenance includes, but is not limited to replacement of gas chromatographic column or replacement of the electron capture detector.

12.4.2 To determine the MDLs, the Contractor shall run an MDL study following the procedures specified in 40 CFR Part 136. The Contractor is only required to analyze the MDL samples on one instrument used for field sample analyses. MDL verification only is then required on all other instruments used for field sample analysis and at the frequency specified in Section 12.4.1. MDL verification is achieved by analyzing a single reagent water blank spiked with each target compound at a concentration equal to two times the analytical determined MDL. The resulting chromatogram must meet the qualitative identification criteria outlined in Sections 11.1.1 through 11.1.5.

12.4.3 The determined concentration of the MDL must be less than the CRQL.

12.4.4 All documentation for the MDL studies shall be maintained at the laboratory and provided to USEPA upon written request.

13.0 METHOD PERFORMANCE

Not Applicable.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasiblely reduced at the source, USEPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction,* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. USEPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult *The Waste Management Manual for Laboratory Personnel,* available from the American Chemical Society at the address listed in Section 14.2.

16.0 REFERENCES

Not Applicable.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE D-1

RETENTION TIME WINDOWS FOR SINGLE AND MULTICOMPONENT ANALYTES AND SURROGATES.

Compound	Compound Identification Window (minutes)
alpha-BHC	±0.05
beta-BHC	±0.05
gamma-BHC	±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin	±0.05
alpha-Chlordane	±0.07
gamma-Chlordane	±0.07
Heptachlor epoxide	±0.07
Dieldrin	±0.07
Endrin	±0.07
Endrin aldehyde	±0.07
Endrin ketone	±0.07
4,4'-DDD	±0.07
4,4'-DDE	±0.07
4,4'-DDT	±0.07
Endosulfan I	±0.07
Endosulfan II	±0.07
Endosulfan sulfate	±0.07
Methoxychlor	±0.07
Aroclors	±0.07
Toxaphene	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

TABLE D-2

NUMBER OF POTENTIAL QUANTITATION PEAKS

Multicomponent Analyte	No. of Potential Quantitation Peaks
Aroclor 1016/1260	5/5
Aroclor 1221	3
Aroclor 1232	4
Aroclor 1242	5
Aroclor 1248	5
Aroclor 1254	5
Toxaphene	4

TABLE D-3

LABORATORY CONTROL SAMPLE RECOVERY LIMITS

Compound	% Recovery
gamma-BHC	50-120
Heptachlor epoxide	50-150
Dieldrin	30-130
4,4'-DDE	50-150
Endrin	50-120
Endosulfan sulfate	50-120
gamma-Chlordane	30-130

NOTE: The recovery limits for any of the compounds in the LCS may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

TABLE D-4

MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery	RPD
gamma-BHC (Lindane)	56-123	15
Heptachlor	40-131	20
Aldrin	40-120	22
Dieldrin	52-126	18
Endrin	56-121	21
4,4'-DDT	38-127	27

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND REQUIREMENTS

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Exhibit E -- Quality Assurance/Quality Control Procedures and Requirements

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1.0 OVERVIEW

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

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

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

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

2.0 INTRODUCTION

Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the Quality Control (QC) procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories in the Contract Laboratory Program (CLP). However, the validation of these methods does not guarantee that they perform equally well for all sample matrices encountered. Inaccuracies can also result from causes other than unanticipated matrix effects, such as sampling artifacts, equipment malfunctions, and operator error. Therefore, the QC component of each method is indispensable.

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

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

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

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

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

- Preparation of, and adherence to, a written QAP, the elements of which are designated in Section 5;
- Preparation of, and adherence to, Standard Operating Procedures (SOPs) as described in Section 6;

- Adherence to the analytical methods and associated QC requirements specified in the contract;
- Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical supply houses;
- Submission of all raw data and pertinent documentation for Regional review;
- Participation in the analysis of laboratory evaluation samples, including adherence to corrective action procedures;
- Submission, upon request, of instrument data tapes and applicable documentation for tape audits, including a copy of the Sample Data Package;
- Participation in on-site laboratory evaluations, including adherence to corrective action procedures; and
- Submission of all original documentation generated during sample analyses for USEPA review.

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

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

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

The QA/QC procedures defined herein shall be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified in the methods in Exhibit D, the Contractor shall also follow these procedures.

NOTE: The cost of performing all QA/QC procedures specified in this Statement of Work (SOW) is included in the price of performing the bid lot.

4.1 Purpose

4.1.1 The purpose of this document is to provide a uniform set of procedures for the analysis of organic samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation. Although it is impossible to address all analytical situations in one document, the approach taken here is to define the minimum requirements for all major steps relevant to any organic low concentration analysis.

4.1.2 The primary function of the QA/QC program is the definition of procedures for the evaluation and documentation of analytical methodologies and the reduction and reporting of data. The objective is to provide a uniform basis for sample handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. In many instances where methodologies are available, specific QC procedures are incorporated into the method documentation (Exhibit D).

4.2 Laboratory Audit and Intercomparison Study Program

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by USEPA. The Contractor can expect to analyze at least two Performance Evaluation (PE) samples per calendar quarter during the contract period for organic low concentration analyses.

4.3 Annual Verification of Method Detection Limits (MDLs)

The Contractor shall perform annual verification of MDLs in accordance with the specifications in Exhibit D. All the MDLs shall meet the Contract Required Quantitation Limits (CRQLs) specified in Exhibit C.

4.4 Quality Assurance/Quality Control Measurements

4.4.1 In this Exhibit, as well as other places within this SOW, the term "analytical sample" is used in discussing the required frequency or placement of certain QA/QC measurements. As the term is used, analytical sample includes all field samples, including PE samples, received from an external source. It also includes all required QA/QC samples [requested Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), and Laboratory Control Sample (LCS)] except those directly related to instrument calibration or calibration verification (calibration standards, Initial Calibration, Continuing Calibration, and tunes).

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

4.4.3 If any QC measurement fails to meet contract criteria, the analytical measurement must not be repeated prior to taking the appropriate corrective action as specified in Exhibit D.

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

4.4.5 In addition, 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.

5.0 QUALITY ASSURANCE PLAN (QAP)

5.1 Introduction

The Contractor shall establish a Quality Assurance (QA) program with the objective of providing sound analytical chemical measurements. This program shall incorporate the Quality Control (QC) procedures, any necessary corrective action, all documentation required during data collection, and the quality assessment measures performed by management to ensure acceptable data production.

5.1.1 As evidence of such a program, the Contractor shall prepare a written QAP which describes the procedures that are implemented to achieve the following:

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

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

5.2 Required Elements of a Quality Assurance Plan

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

A. Organization and Personnel

1. QA Policy and Objectives

2. QA Management

a. Organization

b. Assignment of QA/QC Responsibilities

c. Reporting Relationships

d. QA Document Control Procedures

e. QA Program Assessment Procedures

3. Personnel

a. Resumes

b. Education and Experience Pertinent to this Contract

c. Training Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives

2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy

2. Sample Tracking/Custody Procedures

3. Logbook Maintenance and Archiving Procedures

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

5. Procedures for Preparation, Approval, Review, Revision, and Distribution of Standard Operating Procedures (SOPs)

6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency

2. Sample Preparation Procedures

3. Sample Analysis Procedures

4. Standards Preparation Procedures

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

E. Data Generation

1. Data Collection Procedures

2. Data Reduction Procedures

3. Data Validation Procedures

4. Data Reporting and Authorization Procedures

F. Quality Assurance

1. Data Quality Assurance

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures

5. QA Reporting Procedures

6. Responsibility Designation

G. Quality Control

1. Solvent, Reagent, and Adsorbent Check Analysis

2. Reference Material Analysis

3. Internal QC Checks

4. Corrective Action and Determination of QC Limit Procedures

5. Responsibility Designation

5.3 Updating and Submitting the Quality Assurance Plan

5.3.1 Initial Submission. During the contract solicitation process, the Contractor is required to submit its QAP to the CLP Contracting Officer. Within sixty days after contract award, the Contractor shall maintain on file a revised QAP, fully compliant with the requirements of this contract. The revised QAP will become the official QAP under the contract and may be used during legal proceedings. The Contractor shall maintain the QAP on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised QAP shall be paginated consecutively in ascending order. The revised QAP shall include:

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

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

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

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

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

5.3.2.1 The Contractor shall amend the QAP within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility. When the QAP is amended, all changes in the QAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended section pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the latest version of the QAP document. The Contractor shall archive all amendments to the QAP document for future reference by USEPA.

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

5.4 Corrective Actions

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

6.0 STANDARD OPERATING PROCEDURES (SOPs)

6.1 Introduction

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by USEPA, a SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks.

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

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

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

6.2 Format

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

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

6.3 Requirements

The Contractor shall maintain the following SOPs:

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

6.3.2 Sample Receipt and Storage

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

6.3.3 Sample Preparation

- Reagent purity check procedures and documentation;
- Extraction procedures;
- Extraction bench sheets; and
- Extraction logbook maintenance.

6.3.4 Glassware Cleaning

6.3.5 Calibration (Balances, etc.)

- Procedures;
- Frequency requirements;
- Preventive maintenance schedule and procedures; and
- Acceptance criteria and corrective actions.

6.3.6 Analytical Procedures (for each analytical system)

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

6.3.7 Maintenance Activities (for each analytical system)

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

6.3.8 Analytical Standards

- Standard coding/identification and inventory system;
- Standards preparation logbook(s);
- Standard preparation procedures;
- Procedures for equivalency/traceability analyses and documentation;
- Purity logbook (primary standards and solvents);
- Storage, replacement, and labeling requirements; and
- QC and corrective action measures.

6.3.9 Data Reduction Procedures

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

6.3.10 Documentation Policy/Procedures

- Contractor/analyst's notebook policy, including review policy;
- Complete SDG File contents;
- Complete SDG File organization and assembly procedures, including review policy; and
- Document inventory procedures, including review policy.

6.3.11 Data Validation/Self-Inspection Procedures

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

6.3.12 Data Management and Handling

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

6.4 Updating and Submitting SOPs

6.4.1 Initial Submission. During the contract solicitation process, the Contractor is required to submit their SOPs to the CLP Contracting Officer. Within 60 days after contract award, the Contractor shall maintain on file a complete revised set of SOPs, fully compliant with the requirements of this contract. The revised SOPs will become the official SOPs under the contract and may be used during legal

proceedings. The Contractor shall maintain the complete set of SOPs on file at the Contractor's facility for the term of the contract. Both the initial submission of SOPs and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

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

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

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

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

6.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended/new SOPs shall have the date on which the changes were implemented.

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

6.4.2.3 Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs. An alternate delivery schedule for submitting the letter and amended/new SOPs may be proposed by the Contractor, but it is the sole decision of the USEPA Contracting Officer to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed,

the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 30 days for amending/writing new SOPs. An extension for amending/writing new SOPs beyond 30 days must be approved by the USEPA Contracting Officer. Similarly, an extension of up to 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs may be approved by the USEPA Regional CLP PO. An extension beyond the 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the USEPA Regional CLP PO and/or Contracting Officer.

6.5 Corrective Action

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

7.0 CONTRACT COMPLIANCE SCREENING (CCS)

7.1 Overview

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

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

7.2 CCS Results

CCS results are mailed to the Contractor and all other data recipients. The Contractor has a period of time to correct deficiencies. The Contractor shall send all corrections to the Regional client and SMO within 6 business days. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

7.3 CCS Trend Report

USEPA may generate a CCS trend report which summarizes CCS results over a given period of time. USEPA may send the CCS trend report or discuss the CCS trend report during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and Contracting Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report of the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response to the CCS trend report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

7.4 Corrective Actions

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

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

8.0 ANALYTICAL STANDARDS REQUIREMENTS

8.1 Overview

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

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

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

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

8.2.3 The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the Contractor's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

EQ. 1

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

Where: "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

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

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

8.2.6 Calculate the weight of material to be weighed out for a specified volume, taking into account the purity of the compound and the

desired concentration. A second person shall verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights every 12 hours. All weighing shall be performed on an analytical balance to the nearest 0.1 milligram (mg) and verified by a second person. The solvent used to dissolve the solute shall be compatible with the protocol in which the standard is to be used; the solute shall be soluble, stable, and non-reactive with the solvent. In the case of a multicomponent solution, the components must not react with each other.

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

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

8.3 Purchase of Chemical Standards Already in Solution

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

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

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

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

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

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

EQ. 2

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

EQ. 3

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

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

EQ. 4

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

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

8.3.2.4 The test statistic shall be calculated:

EQ. 5

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

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

8.3.2.5 The test statistic shall be calculated:

EQ. 6

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

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

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

EQ. 7

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

EQ. 8

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

EQ. 9

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

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

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

8.4 Requesting Standards from the USEPA Standards Repository

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

8.5 Documentation of the Verification and Preparation of Chemical Standards

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

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

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

8.5.3 USEPA may generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards or may discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO and Quality Assurance Technical Support (QATS), the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the standards documentation report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

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

8.6 Corrective Actions

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

9.0 DATA PACKAGE AUDITS

9.1 Overview

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

9.2 Responding to the Data Package Audit Report

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

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

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

9.3 Corrective Actions

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

10.0 REGIONAL DATA REVIEW

10.1 Overview

Contractor data are generated to meet the specific needs of USEPA Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end user, based on functional guidelines for data review which have been developed jointly by the Regions and the 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 problem under investigation and the Regional response appropriate to the specific circumstances.

10.1.1 Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review performed at the Sample Management Office (SMO), which is designed to identify contractual discrepancies, and the review performed by the National Program Office, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for Program and Contractor administration and management, and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

11.0 PROFICIENCY TESTING

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

11.1 Performance Evaluation (PE) Samples

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

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

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

sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.1.4 In addition to PE sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes/parameters included in each PE sample. When PE sample results are received by USEPA, the PE sample results will be evaluated for correct analytical identification and quantitation. The PE sample evaluation will be provided to the Contractor via coded evaluation sheets, by analyte/parameter. USEPA will notify the Contractor of unacceptable performance. USEPA reserves the right to adjust the PE sample acceptance windows in order to compensate for any unanticipated difficulties with a particular PE sample.

11.1.5 The Contractor shall demonstrate acceptable analytical performance for both identification and quantitation of PE sample analytes/parameters. For unacceptable PE sample performance, USEPA may take, but is not limited to, the following actions: reduce value or rejection of data for the samples, SDG, or Case impacted, contract sanctions, reduction in the number of samples shipped to the laboratory, suspension of sample shipment, an on-site laboratory inspection, a full data package audit, electronic data audit, and/or require the laboratory to analyze a Remedial QB.

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

11.2 Quarterly Blind (QB) Audits

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

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

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

11.2.4 In addition to QB sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes/parameters included in each QB sample. When QB sample results are received by USEPA, the QB sample results will be scored for correct analytical identification and quantitation. The QB sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte/parameter. USEPA will notify the

Contractor of unacceptable performance. USEPA reserves the right to adjust the PE sample acceptance windows in order to compensate for any unanticipated difficulties with a particular PE sample. The Contractor's QB sample performance will be assessed into one of the following three categories:

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

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

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

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

11.2.5.1 An alternate delivery schedule for the corrective action letter may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and Contracting Officer why the laboratory is unable to meet the original delivery schedule listed in Section 11.2.5. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's corrective action letter. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

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

11.2.7 The Contractor shall be notified by the USEPA Contracting Officer concerning agreement or disagreement with the proposed remedy for unacceptable performance. For unacceptable QB sample performance (Section 11.2.4.3), USEPA may take, but is not limited to, the following actions: reduction in the number of samples shipped to the laboratory, suspension of sample shipment, an on-site laboratory inspection, electronic data audit, a full data package audit, and/or require the laboratory to analyze a Remedial QB sample, and/or contract sanctions.

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

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

11.3 Corrective Actions

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

12.0 ON-SITE LABORATORY EVALUATIONS

12.1 Overview

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

12.2 Quality Assurance On-Site Evaluation

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

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

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

12.2.2 Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous on-site reports, performance evaluation sample scores, Regional review of data, Regional QA materials, data audit reports, results of Contract Compliance Screening (CCS), and data trend reports.

12.3 Evidentiary Audit

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

12.3.1 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), analytical project file organization and assembly, and proper disposal of samples and cogenerated wastes.

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

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

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

12.4 Discussion of the On-Site Team's Findings

The QA and evidentiary auditors discuss their findings with the USEPA Regional CLP PO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

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

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

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

12.5.2 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the

Contractor shall write/amend the SOPs per the requirements listed in Section 6.

12.6 Corrective Actions

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

13.0 ELECTRONIC DATA AUDITS

13.1 Overview

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

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

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

13.1.2 The Contractor shall store all raw and processed electronic analytical data in the appropriate instrument manufacturer's format, uncompressed, and with no security codes. The data shall include all necessary data files for a complete reconstruction of the previously submitted hardcopy and electronic deliverable data package. All associated raw data files in the instrument manufacturer proprietary software format must be submitted if those files contain data or instrumental parameters regarding any analysis and or correction applied to an instrument or analytical result. This instrument electronic data shall include data for all samples and all QC samples, including but not limited to: blanks, Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), Laboratory Control Sample (LCS), instrument performance checks [4-Bromofluorobenzene (BFB) and decafluorotriphenylphosphine (DFTPP)], initial calibrations, Continuing Calibration, as well as all Contractor-generated spectral libraries and quantitation reports required to generate the data package. In addition, the Contractor shall supply raw data for the Method Detection Limit (MDL) studies and values for the year in which the Sample Delivery Group (SDG) was analyzed. The Contractor shall maintain a written reference logbook of data files of EPA sample number, calibration data, standards, blanks, spikes, and duplicates. The logbook shall include EPA sample numbers, identified by Case and SDG.

13.1.3 The Contractor is required to retain the instrument electronic data for three years after submission of the reconciled Complete SDG File. Electronic media shipped to USEPA designated recipient must be fully usable by the recipient. Diskettes must be 3.5 inch, high density, 1.44 MB MS DOS formatted and tapes must be either 4 mm or 8 mm. Alternative means for delivery of electronic data may be utilized by the Contractor upon prior written approval by USEPA. When submitting electronic instrument data to a USEPA, the following materials shall be delivered in response to the request.

13.1.3.1 All associated raw data files for all analytical samples and all QC samples. For example, files for LCS, blanks, initial and continuing calibration standards and instrument performance check solutions (BFB and DFTPP).

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

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

13.1.3.4 All Contractor-generated Mass Spectral library files (NIST/EPA/NIH and/or Wiley, or equivalent, library not required).

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

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

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

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

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

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

13.2 Submission of the Instrument Electronic Data

Upon request of the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO), the Contractor shall send the required instrument electronic data and all necessary documentation to USEPA designated recipient [e.g., Quality Assurance Technical Support (QATS)] within 7 days of notification. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in this section. The USEPA Regional CLP PO may grant an extension of up to 7 days for submission of the instrument electronic data. An extension beyond 7 days must be approved by the USEPA Contracting Officer (CO). The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

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

13.3 Responding to the Electronic Data Audit Report

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

13.3.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of USEPA to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the USEPA Regional CLP PO and the Contracting Officer why it is unable to meet the delivery schedule listed in Section 13.3. The USEPA Regional CLP PO may grant an extension of up to 14 days for the Contractor's response letter to the electronic data report. An extension beyond 14 days must be approved by the USEPA Contracting Officer. The Contractor shall proceed and not assume that an extension will be granted until so notified by the appropriate USEPA official.

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

13.4 Corrective Actions

If the Contractor fails to adhere to the requirements listed in Section 13, the Contractor may expect, but USEPA is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, an electronic data audit, a data package audit, a remedial laboratory evaluation sample, and/or contract sanctions.

13.5 Maintenance of the Magnetic Tape Storage Device

13.5.1 The Contractor shall certify that the tape head alignment on the magnetic tape storage device is in compliance with the ANSI standards for nine track magnetic tapes. If the Contractor does not have documentation of alignment within the last 12 months, the Contractor must perform or have performed the manufacturer's documented head alignment procedure within 60 days of contract award. This is generally performed with a "skew" tape, certified to be in conformance with ANSI standards. The alignment must be performed by qualified personnel. The tape head alignment must be performed at a minimum once every 12 months, or whenever there is evidence that the tape head may be out of alignment.

13.5.2 The tape system, including recording head, must be in conformance with the manufacturer's physical and electrical standards. Alignment of the remaining components of the tape system such as the retracting arms, must be performed at intervals not to exceed 24 months. If the Contractor cannot demonstrate that the remaining components of the tape system are in alignment, then the Contractor must perform or have performed the manufacturer's recommended alignment procedure.

14.0 DATA MANAGEMENT

14.1 Overview

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

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

14.2 Documenting Data Changes

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

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

14.3 Lifecycle Management Procedures

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

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

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

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

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

14.3.5 This documentation shall be available for on-site review and/or upon written request by the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO).

14.4 Personnel Responsibilities

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

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

EXHIBIT F

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

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

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1.0 INTRODUCTION

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

1.1 Purpose of Evidence Requirements

The purpose of the evidence requirements include:

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

2.0 STANDARD OPERATING PROCEDURES

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

2.1 Sample Receiving

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

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

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

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

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

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

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

- Presence or absence of sample tags;
- Sample tag numbers;
- Assigned laboratory numbers;
- Samples delivered by hand; and
- Problems and discrepancies.

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

NOTE: Initials are not acceptable.

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

2.1.9 The Contractor shall record resolution of problems and discrepancies by SMO.

2.2 Sample Identification

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

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

2.3 Sample Security

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

- It is in your possession; or
- It is in your view after being in your possession; or
- It is locked in a secure area after being in your possession; or
- It is in a designated secure area, accessible only to authorized personnel.

2.3.2 The Contractor shall demonstrate security of designated secure areas.

2.4 Sample Storage

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

2.5 Sample Tracking and Document Control

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

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

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

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

2.5.5 The laboratory name shall be identified on pre-printed laboratory documents.

2.5.6 Each laboratory document entry shall be dated with the month/day/year (e.g., 01/01/2000) and signed (or initialed) by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

2.5.7 Notations on laboratory documents shall be recorded in ink.

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

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

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

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

2.5.12 Logbook entries shall be in chronological order.

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

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

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

2.6 Computer-Resident Sample Data Control

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

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

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

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

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

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

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

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

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

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

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

2.7 Complete SDG File (CSF) Organization and Assembly

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

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

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

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

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

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

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

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

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

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

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF _____

_____
Signature

_____
Date

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

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

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

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

2.7.14 Shipments of deliverable packages, including re-submittals, shall be sealed with custody seals by the document control officer or a designated representative in a manner such that opening the packages would break the seals.

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

3.0 WRITTEN STANDARD OPERATING PROCEDURES

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

3.1 Sample Receiving

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

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

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

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

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

- Custody seals;
- Chain-of-Custody records;
- Traffic Reports (TRs) or Packing Lists;
- Airbills or airbill stickers;
- Sample tags; and
- Cooler temperature indicator bottle.

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

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

- Presence or absence and condition of custody seals on shipping and/or sample containers;
- Custody seal numbers when present;
- Condition of the sample bottles;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;

- Presence or absence of Chain-of-Custody records;
- Sample tags listed/not listed on Chain-of-Custody records;
- Presence or absence of TRs or Packing Lists;
- Presence or absence of cooler temperature indicator bottle;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;
- Presence or absence of sample tags;
- Sample tag numbers;
- Assigned laboratory numbers;
- Samples delivered by hand; and
- Problems and discrepancies.

3.1.2.6 The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., Chain-of-Custody records, TRs or packing lists, and airbills).

NOTE: Initials are not acceptable.

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

3.1.2.8 The Contractor shall record resolution of problems and discrepancies by SMO.

3.2 Sample Identification

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

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

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

- The Contractor assigns unique laboratory sample identification numbers, thus the written SOPs shall include a description of the procedure used to assign these numbers;
- The Contractor uses prefixes or suffixes in addition to laboratory sample identification numbers, thus the written SOPs shall include their definitions; and

- The Contractor uses methods to uniquely identify fractions/parameter groups and matrix type, thus the written SOPs shall include a description of these methods.

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

3.3 Sample Security

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

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

3.3.2.1 Procedures which ensure the following:

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

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

3.4 Sample Storage

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

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

3.5 Sample Tracking and Document Control

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

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

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

3.5.2.2 Procedures which ensure the following:

- All activities performed on USEPA samples are recorded;
- Titles which identify the activities recorded are printed on each page of all laboratory documents;
- Information recorded in columns is identified with column headings;
- Reviewers' signatures are identified on laboratory documents;
- The laboratory name is included on pre-printed laboratory documents;

- Laboratory document entries are signed and dated with the month/day/year (e.g., 01/01/2000);
- Entries on all laboratory documents are recorded in ink;
- Corrections and additions to laboratory documents are made by drawing single lines through the errors, entering the correct information, and initialing and dating the new information;
- Unused portions of laboratory documents are lined-out;
- Pages in bound and unbound logbooks are sequentially numbered;
- Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
- Logbook entries are recorded in chronological order;
- Entries are recorded for only one SDG per page, except in the event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs);
- Information inserted in laboratory documents is affixed permanently, signed or initialed, and dated across the insert; and
- The retention or disposal of USEPA samples, remaining portions of samples, and prepared samples is documented.

3.6 Computer-Resident Sample Data Control

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

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

3.6.2.1 Procedures which ensure the following:

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

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

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

3.7 CSF Organization and Assembly

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

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

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

EXHIBIT G

GLOSSARY OF TERMS

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

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

BAR GRAPH SPECTRUM - A plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

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

BREAKDOWN - A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4-BROMOFLUOROBENZENE (BFB) - The compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION FACTOR (CF) - A measure of the gas chromatographic response of a target analyte to the mass injected.

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

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

CONTINUING CALIBRATION - Analytical standard run every 12 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - Used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

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

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

DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) - Compound chosen to establish mass spectral instrument performance for semivolatile analysis.

DEUTERATED MONITORING COMPOUNDS (DMCs) - Compounds added to every calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the gas chromatograph/mass spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

EXTRACTABLE - A compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (SVOA) and pesticide/Aroclor compounds.

EXTRACTED ION CURRENT PROFILE (EICP) - A plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

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

GAS CHROMATOGRAPH (GC) - The instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA), or injected as extracts (SVA and PEST). In VOA and SVA analysis, the compounds are detected by a Mass Spectrometer. In PEST analysis, the compounds are detected by an Electron Capture Detector.

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 mass spectrometer or electron capture detector to the target compounds.

INTEGRATION SCAN RANGE - The scan number of the scan at the beginning of the area of integration to the scan number at the end of the area of integration. Performed in accordance with Exhibit D VOA and SVOA.

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

INTERNAL STANDARDS - Compounds added to every standard, blank, sample (for volatiles), sample extract (for semivolatiles), including Laboratory Control Sample, at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

INSTRUMENT BLANK - A blank designed to determine the level of contamination associated with the analytical instruments.

LABORATORY - Synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) - 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.

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

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, the sample matrix is water.

MATRIX EFFECT - In general, the effect of a particular matrix (water) on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, nontarget analytes may be extracted from the matrix causing interferences.

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

MATRIX SPIKE DUPLICATE - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - An analytical control consisting of all reagents, internal standards, and surrogate standards (or DMCs for VOA and SV), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

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

PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero.

PERFORMANCE EVALUATION MIXTURE - A calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PERFORMANCE EVALUATION SAMPLE - An external quality control sample prepared by USEPA and is designed to assess the capability of the Contractor to perform the analytical method.

PRIMARY QUANTITATION ION - A contract specified ion used to quantitate a target analyte.

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

PURGE AND TRAP (DEVICE) - Analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

PURGEABLES - Volatile compounds.

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.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values.

RELATIVE RESPONSE FACTOR (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RELATIVE RETENTION TIME (RRT) - The ratio of the retention time of a compound to that of a standard (such as an internal standard).

RESOLUTION - Also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RESOLUTION CHECK MIXTURE - A solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

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

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

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

- Each Case of field samples received, or
- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- Each 7 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.

SAMPLE MANAGEMENT OFFICE (SMO) - A contractor operated facility operated under the Contract Laboratory Analytical Services Support (CLASS) contract, awarded and administered by USEPA.

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

SECONDARY QUANTITATION ION - Contract specified ion(s) to be used in quantitation of target analytes when interferences prevent the use of the primary quantitation ion.

SEMIVOLATILE (SV) COMPOUNDS - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

STANDARD ANALYSIS - An analytical determination made with known quantities of target compounds; used to determine response factors.

STORAGE BLANK - Reagent water (two 40.0 mL aliquots) stored with volatile samples in an SDG. It is analyzed after all samples in that SDG have been analyzed; and it is used to determine the level of contamination acquired during storage.

SULFUR CLEANUP BLANK - A modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, then the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur cleanup blank is required.

SURROGATES (Surrogate Standard) - For pesticides/Aroclors, compounds added to every blank, sample, including Laboratory Control Sample, requested MS/MSD, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

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

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - Compounds detected in samples that are not target compounds, internal standards, deuterated monitoring compounds, or surrogates. Up to 30 peaks, not including those identified as alkanes (those greater than 10% of the peak area or height of the nearest internal standard), 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 (0000-2359).

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

TWELVE-HOUR TIME PERIOD - The twelve (12)-hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the 12-hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

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

VOLATILE COMPOUNDS - Compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

WIDE BORE CAPILLARY COLUMN - A gas chromatographic column with an internal diameter (ID) that is greater than or equal to 0.53 mm. Columns with lesser diameters are classified as narrow bore capillary columns.

EXHIBIT H

AGENCY STANDARD IMPLEMENTATION

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Exhibit H - Agency Standard Implementation

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1.0 FORMAT CHARACTERISTICS

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

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

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

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

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

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

2.0 RECORD TYPES

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

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

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

2.3 A separate run header is used for volatiles (VOA), semivolatiles (SV), and for each column analysis for pesticides (PEST) [minimum of four type 10 series for VOA/SV/PEST Sample Delivery Group (SDG)]. The 20 series records contain sample characteristics and link samples within an SDG to the corresponding calibrations, blanks, and other Quality Controls (QCs). The 30 series records contain the actual analytical results by analyte within each sample. The 10, 20, and 30 records are associated with each other by their position in the file (i.e., 30 series records follow the corresponding 20 series, which in turn follow the 10 series run header records).

3.0 PRODUCTION RUNS

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

3.1.1 Calibration - All samples in a run use the same initial calibration data.

3.1.2 Method number - Constant throughout a run.

3.1.3 Instrument conditions - Constant throughout a run.

3.2 Each instrumental analysis consists of a separate production run and is reported in a separate file. There will be a separate production run for each of the two pesticide Gas Chromatograph (GC) columns utilized. Thus, a full three fraction analysis will consist of a minimum of four production runs.

3.3 Example of the Sequence of Record Types in a File

10			Contains Run Header information.
	11		Contains additional run-wide information.
	20		Occurs once for each sample, calibration, mean response factor, matrix spike duplicate result, etc. Acts as a header.
	21		
	22		Contains additional information for samples.
	23		
	27		
		30	Occurs once for each final analytical result. Reports the value being determined as defined by the type 20.
		32	Reports any auxiliary data necessary.
		33	Reports compound names for tentatively identified compounds (TICs) if necessary.
		36	Reports any instrumental data necessary.
		30	Values for the next analyte or parameter being measured.
		32	Additional data may vary for each parameter, and may
		33	occur in any order. Multiple occurrences of the same
		36	record type, however, must be consecutive.
		30	Continues for as many as are necessary.
		32	
		33	
		36	
		30	
		32	
		33	
		36	
	20		Next Sample Header record. The following applies to the
	21		next sample or other group of data.
	22		
		30	
		32	
		33	
		36	
		30	
		32	
		33	
		36	

etc.

20
21
30
32
33
36
etc.

4.0 RECORD SEQUENCE

4.1 The sequence of records for Agency Standard files is as follows: A Run Header (type 10) record shall be present once and once only (per file) as the first record in a file. Therefore, a complete VOA/SV/PEST SDG will consist of several files.

4.2 Each environmental sample, calibration standard, or Quality Control (QC) sample is represented by a group composed of type 20, 21, 22, 23, and 27 records, that hold sample level identifying information, followed by type 30, 32, 33, and 36 records for each method analyte including surrogates, deuterated monitoring compounds, and internal standards in the sample. The type 20 record holds a count for the number of method analytes being determined and includes all target compounds, surrogates, deuterated monitoring compounds, and internal standards plus each peak of the multi-component pesticides [do not include Tentatively Identified Compounds (TICs) in this count]. A separate field on the type 23 record contains the number of TICs found. Type 20 records shall occur in the order of sample analysis. In addition, a type 20 record with a QC code "MNC", followed by a type 30 record for each method analyte (reporting values such as mean response factors) will appear after the type 10 or type 11 record and before the type 20 record that initiates the analytical sequence. Similarly, for pesticide runs, a type 20 record with a Quality Control (QC) code "FLO" for Florisil recovery, followed by type 30 records for each of the method analytes (and the two surrogates) included in the Florisil check will appear before the type 20 record that initiates the analytical sequence.

4.3 Type 90 comment records may be defined to occupy any position after the type 10 (header) record.

5.0 FILE/RECORD INTEGRITY

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

Record Position	Field Length	Field Contents	Remarks
First Field	2	Record type	"10" or as appropriate
Last Field	5	Record sequence number	00001-99999, numbered within file sequentially
	4	Record checksum[1]	Four hexadecimal digits
	2	Must contain CR and LF	

6.0 DATES AND TIMES

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

7.0 MULTIPLE VOLUME DATA

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

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

8.0 DELIVERABLE

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

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

8.3 Information on the diskette **must correspond** to information submitted in the hardcopy raw data package and on the hardcopy raw data package forms. For example, type 30 results field specifies maximum length of 13. When reporting Contract Required Quantitation Limits (CRQLs) or results on Form 1, maximum length is 13 as is specified in this exhibit; when reporting 'calculated amounts' on Form 7D, hardcopy specified maximum length is 8. Unused records shall not be included on the diskettes. If the information submitted in the hardcopy data package forms is changed, the information in the electronic file (e.g., diskette) shall be changed accordingly, and a complete electronic deliverable containing all the information for the SDG shall be resubmitted along with the hardcopy at no additional cost to the USEPA.

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

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

8.5 The format for File Name shall be XXXXX.001 to XXXXX.099. Where XXXXX is the SDG identifier, 0 designates Organics, and 01 through 99 is the file number.

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

8.7 Section 9.0 (Record Listing) provides information for the usage of each of the record types. Where specified, labels indicate the nature of the value(s) that follow on that record. If the value(s) will not be reported, the label shall be omitted.

8.7.1 A record type 30 for each Target Compound List (TCL) compound, surrogate, deuterated monitoring compound, and internal standard shall be reported. If the TCL is not detected, the 'U' qualifier in the appropriate field shall be indicative of that.

8.7.2 For multicomponent analytes (Aroclors/toxaphene), if the multicomponent analyte is detected, a record type 30 and 32 shall be reported for each peak identified.

9.0 RECORD LISTING

The following lists every record type required to report data from a single Sample Delivery Group (SDG).

9.1 Production Run Header Record (Type 10)

Use: Each production run will start with a record type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"10"
6	Delimiters	||||||
5	INSTRUMENT/DETECTOR	Character[2]
1	Delimiter	|
8	METHOD NUMBER	Character[3]
2	Delimiters	||
6	LAB CODE	Character
4	Delimiters	||||
11	CONTRACT NUMBER	Character
1	Delimiter	|
10	INSTRUMENT ID	Character
2	Delimiters	||
25	LABORATORY NAME	Character
2	Delimiters	||
5	RECORD SEQUENCE NUMBER	Numeric
4	CHECKSUM	Character

[2]General descriptor (GC/MS for VOA/SV analysis or GC for pesticide analysis on GC/ECD).

[3]OLC03.1V for volatiles; OLC03.1B for semivolatiles; OLC03.1P for pesticides. (O for Organic, L for Low, C for Concentration, zero three point zero for document number, V for volatiles, B for semivolatiles, P for pesticides.)

9.2 Chromatography Record (Type 11)

Use: To describe chromatograph condition. Must be present once for each production run immediately following the record type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"11"
1	Delimiter	|
10	GC COLUMN IDENTIFICATION	Character
2	Delimiters	||
4	GC COLUMN ID[4]	Numeric (mm)
11	Delimiters	|||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[4]Internal Diameter of the GC column used.

9.3 Sample Header Data Record (Type 20)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"20"
2	Delimiters	||
12	EPA SAMPLE NUMBER	As is exactly on the hardcopy form
1	Delimiter	|
1	MATRIX	CHARACTER[5]
1	Delimiter	|
3	QC CODE	Character (See Section 10)
1	Delimiter	|
3	SAMPLE QUALIFIER	RIN/REX/REJ/SRN/blank[6]
1	Delimiter	|
5	CASE NUMBER	Numeric
1	Delimiter	|
6	SDG NO.	Character
1	Delimiter	|
4	SAMPLE/BLANK/STANDARDS YEAR ANALYZED	YYYY
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS MONTH ANALYZED	MM
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS DAY ANALYZED	DD
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS HOUR ANALYZED	HH
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS MINUTE ANALYZED	MM
2	Delimiters	||
2	SAMPLE VOL UNITS	"ML"/blank[7]
1	Delimiter	|
5	SAMPLE VOL	Numeric[8]

[5]"0" if not applicable (calibration, tune, etc.); "1" for water.

[6]"RIN" for reinjection; "REX" for re-extractions; "REJ" for rejected samples; "SRN" for dilutions; and leave blank (empty field with zero length) when none of the previous conditions apply. In case of multiple operations on a sample, the final operation will be indicated (e.g., reinjection of a dilution; AAA12DLRE would have a QC Code of "RIN").

[7]Sample VOL unit is ML (milliliters) for liquids. Leave blank (zero length) if not applicable.

[8]Sample VOL is the volume in milliliters for liquid. Sample VOL includes the purge volume.

Sample Header Data Record (Type 20) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
3	ANALYTE COUNT	Numeric[9]
3	Delimiters	|||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[9]Counts TCL analytes, surrogates, deuterated monitoring compounds (DMC), internal standards, and all peaks reported for multi-component PCBs. Do not include the count for TICs in this field. For calibrations, also count DFTPP, if included in calibration solution.

9.4 Sample Header Data Record (Type 21)

Use: Continuation of Type 20.
Position: Follows the Type 20 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"21"
1	Delimiter	|
1	PURGE	"N" (for VOA); blank (for SV or PEST[10]
3	Delimiters	|||
1	EXTRACTION	S/C/H/blank (for all volatile samples)[11]
2	Delimiters	||
6	CLIENT NUMBER	Character
1	Delimiter	|
14	LAB FILE/SAMPLE ID	Character[12]
1	Delimiter	|
4	YEAR EXTRACTED	YYYY/blank (for volatiles)
1	Delimiter	|
2	MONTH EXTRACTED	MM/blank (for volatiles)
1	Delimiter	|
2	DAY EXTRACTED	DD/blank (for volatiles)
2	Delimiters	||
4	YEAR RECEIVED	YYYY/blank (for standards, tunes, and blanks)
1	Delimiter	|
2	MONTH RECEIVED	MM/blank (for standards, tunes, and blanks)
1	Delimiter	|
2	DAY RECEIVED	DD/blank (for standards, tunes, and blanks)
1	Delimiter	|
7	IDENTIFICATION/LOT NUMBER OF LCS	Character[13]
1	Delimiter	|

[10]"N" for not heated purge. All low concentration volatile samples are required to be purged at ambient temperature.

[11]"S" for separatory funnel; "C" for continuous liq-liq without hydrophobic membrane; "H" for continuous liq-liq with hydrophobic membrane; blank (zero length field) for all volatile samples.

[12]Lab File ID for volatile and semivolatile analyses. Lab Sample ID for pesticides in same format as on forms.

[13]If the LCS solution was purchased by the Contractor from a third party, report the identification number used by the third party to identify the LCS lot, if available (Form 3). If the LCS solution was prepared in-house, leave this entry blank.

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

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
8	INJECTION VOLUME	Numeric/blank (for VOA)[14]
2	Delimiters	||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[14]Injection volume, in µL, for SVs and PESTs.

9.5 Sample Condition Record (Type 22)

Use: Continuation of type 20. Used to describe additional Sample Conditions.
Position: Follows the type 20 and 21 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"22"
1	Delimiter	|
4	CALIBRATION YEAR	YYYY/blank (for PEST)[15]
1	Delimiter	|
2	CALIBRATION MONTH	MM/blank (for PEST)
1	Delimiter	|
2	CALIBRATION DAY	DD/blank (for PEST)
1	Delimiter	|
2	CALIBRATION HOUR	HH/blank (for PEST)
1	Delimiter	|
2	CALIBRATION MINUTE	MM/blank (for PEST)
1	Delimiter	|
14	CALIBRATION FILE ID	Character/blank (for PEST)[16]
4	Delimiters	||||
8	EXTRACT VOLUME	Numeric/blank (for VOA)[17]
1	Delimiter	|
8	DILUTION FACTOR	Numeric[18]
3	Delimiters	|||
5	LEVEL	Numeric/blank (for VOA/SV)[19]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[15]For volatiles and semivolatiles, enter the date and time of analysis of the most recent 5 ug/L (VOAs) or the 20 ng/uL (SVs) standard run prior to the sample reported in the associated type 20 record. Leave blank for pesticides.

[16]Lab File ID of standard specified in 15 above (volatiles/semivolatiles only). This field must match the Lab File ID on Type 21 for the associated calibration (VSTD005/SSTD020). Leave blank for pesticides.

[17]Enter the Concentrated Extract Volume for all SV and PEST. The value should be reported in microliters.

[18]Dilution factor of sample analyzed.

[19]Concentration level of Pesticide Individual Mix A and B standards. Concentration of low point, mid point and high point calibration standards as a multiplier of low point. Low point = 1.0; Mid point = 4.0; High point = 16.0.

9.6 Associated Injection and Counter Record (Type 23)

Use: Continuation of type 20. Used to identify associated blanks and tunes, and the number of surrogates/DMCs and spikes outside of the Quality Control (QC) limits and the number of tentatively identified compounds (TICs).

Position: Follows the type 20, 21, and 22 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"23"
1	Delimiter	|
1	INSTRUMENT PERFORMANCE CHECK (IPC/TUNE) LABEL	"P" (for BFB and DFTPP IPC) or blank (for pesticides)
1	Delimiter	|
4	IPC/TUNE INJECTION YEAR	YYYY/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION MONTH	MM/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION DAY	DD/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION HOUR	HH/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION MINUTE	MM/blank (for PEST)
1	Delimiter	|
14	DFTPP/BFB LAB FILE ID	Character/blank (for PEST)
1	Delimiter	|
2	VOLATILE STORAGE BLANK LABEL	"HB" (for VOA) or blank (for SV and PEST)
1	Delimiter	|
4	STORAGE BLANK INJECTION YEAR	YYYY/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION MONTH	MM/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION DAY	DD/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION HOUR	HH/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION MINUTE	MM/blank (for SV and PEST)
1	Delimiter	|
14	STORAGE BLANK LAB FILE ID (VOA ONLY)	Character
4	Delimiters	||||

Associated Injection and Counter Record (Type 23) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	METHOD BLANK LABEL	"MB"/blank (for standard, tune, and method blanks)
1	Delimiter	|
4	METHOD BLANK INJECTION YEAR	YYYY/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION MONTH	MM/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION DAY	DD/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION HOUR	HH/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION MINUTES	MM/blank (for standard, tune, and method blanks)
1	Delimiter	|
14	METHOD BLANK LAB FILE (for VOA and SV)/SAMPLE ID (for PEST)	CHARACTER
1	Delimiter	|
1	SURROGATE (for PEST)/ DMC (for VOA and SV) RECOVERY LABEL	"P" for % recoveries/blank (for STD/IPC)
1	Delimiter	|
2	SURROGATE (for PEST)/ DMC (for VOA and SV) RECOVERIES OUT	Numeric[20]
1	Delimiter	|
1	TIC LABEL	"T" (for VOA and SV TICs)/blank (for PEST)
1	Delimiter	|
2	NO. OF TICS	Numeric
1	Delimiter	|
1	SPIKE RECOVERY LABEL	"S" for LCS (Pest)/MS/MSD (all fractions)/blank for anything else
1	Delimiter	|

[20]This will be the number of surrogate (for PEST) or DMC (for VOA and SV) recoveries outside QC limits for a specific column. It should not be cumulative of the two columns for pesticides.

Associated Injection and Counter Record (Type 23) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	SPIKE RECOVERIES OUT	Numeric/blank[21]
1	Delimiter	|
1	RPD LABEL	"R" for RPD/blank[22]
1	Delimiter	|
2	RPD OUT	Numeric
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[21]Enter the number of spike recoveries out. Enter "0"(zero) if none of the spike recoveries are outside of the QC limit.

[22]"R" for Matrix Spike/Matrix Spike Duplicate Recovery Relative Percent Difference. Leave blank for all other samples.

9.7 Sample Cleanup Record (Type 27)

Use: Continuation of type 20. Used to identify sample/blank cleanup procedures and QC results.
Position: Follows type 20, 21, 22, and 23 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"27"
8	Delimiters	||||||||
1	FLORISIL CLEANUP TYPE	"F"/blank (for VOA and SV)
1	Delimiter	|
4	FLORISIL LOT CHECK YEAR	YYYY/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK MONTH	MM/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK DAY	DD/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK HOUR	HH/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK MINUTE	MM/blank (for VOA and SV)
1	Delimiter	|
14	FLORISIL DATA DESCRIPTOR	Character[23]
1	Delimiter	|
1	SULFUR CLEANUP	Y/N (for PEST)/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK LABEL	"SB"/blank (if no separate sulfur blank was prepared for pesticides; also blank for VOA and SV)
1	Delimiter	|
4	SULFUR BLANK INJECTION YEAR	YYYY/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION MONTH	MM/blank (for VOA and SV)
2	Delimiters	||
2	SULFUR BLANK INJECTION DAY	DD/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION HOUR	HH/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION MINUTE	MM/blank (for VOA and SV)
1	Delimiter	|
14	SULFUR BLANK LABORATORY/ SAMPLE ID	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[23]Lab Sample ID of associate Florisil lot check. This is a unique identifier assigned to a lot of Florisil cartridges.

Results Data Record (Type 30) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
1	MEAN CONCENTRATION LABEL	"M" for Mean conc. (for multicomponent PEST only)/blank (for VOA and SV)
1	Delimiter	|
13	MEAN CONCENTRATION	Numeric (for PEST)/blank (for VOA and SV)[32]
1	Delimiter	|
1	PERCENT DIFFERENCE LABEL	"F" or "P" (PEST)/blank (for VOA and SV field sample analysis)[33]
1	Delimiter	|
5	PERCENT DIFFERENCE	Numeric
1	Delimiter	|
1	INTERNAL STANDARD AREA LABEL	"I" for IS Area (for VOA and SV)/blank (for PEST)
1	Delimiter	|
13	INTERNAL STANDARD AREA	Numeric (for VOA and SV)/blank (for PEST)
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[32]Mean Concentration for Multicomponent analytes detected in pesticide analyses.

[33]"P" for Percent Difference between concentrations from two columns in pesticide analyses, or "F" for Percent Difference between average RRF (initial calibration) and RRF5/RRF20 (continuing calibration) in VOA/SV analyses. Leave blank for volatile and semivolatile sample, blank, and tune analysis.

9.9 Auxiliary Data Record (Type 32)

Use: Used to report retention time (in minutes) for Internal Standards and for TICs (for Volatiles and Semivolatiles). Used to report retention time data and percent breakdown (for pesticides).

Position: Follows type 30. (Record will only be required as specified above.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"32"
3	Delimiters	|||
2	RETENTION TIME LABEL	"RT"
1	Delimiter	|
5	RETENTION TIME	Numeric
1	Delimiter	|
3	FIRST LIMIT LABEL	"RTF"
1	Delimiter	|
5	RT WINDOW LOWER LIMIT	Numeric
1	Delimiter	|
3	SECOND LIMIT LABEL	"RTT"
1	Delimiter	|
5	RT WINDOW UPPER LIMIT	Numeric
2	Delimiters	||
2	% BREAKDOWN LABEL	"PB" for % breakdown/blank (for VOA and SV)
1	Delimiter	|
5	% BREAKDOWN	Numeric (DDT/ENDRIN)/blank (for VOA and SV)
1	Delimiter	|
5	COMBINED % BREAKDOWN	Numeric/blank (for VOA and SV)[34]
2	Delimiters	||
1	PEAK	1 THROUGH 5 (for pesticide multicomponent compounds)/blank (for VOA and SV)[35]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

[34]The combined %breakdown will be reported on both the record type 32s for DDT and Endrin.

[35]For positively identified compounds, a minimum of 3 peaks and a maximum of 5 peaks are allowed. Types 30 and 32 will be repeated for each peak that is reported (a minimum of three, a maximum of five times). This is for multicomponent analytes in pesticide analyses.

9.10 Name Record (Type 33)

Use: This record type is used for volatile and semivolatile analyses only to carry an analyte name for TICs. This record is not used for pesticide analysis.

Position: Follows types 30 and 32 for TICs.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"33"
1	Delimiter	|
67	NAME OF COMPOUND	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.11 Instrumental Data Readout Record (Type 36)

Use: This record type is only used for volatile and semivolatile analyses to describe decafluorotriphenylphosphine/4-bromofluorobenzene (DFTPP/BFB) percent abundances. This record is not used for pesticide analysis.

Position: Follows type 30 for DFTPP/BFB data.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"36"
1	Delimiter	|
1	MASS LABEL	"M"
3	Delimiters	|||
3	FIRST MASS (DFTPP/BFB)	Numeric (DFTPP for SV or BFB for VOA)
2	Delimiters	||
5	FIRST PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
3	SECOND MASS	Numeric
1	Delimiter	|
5	SECOND PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 69	Numeric, DFTPP only/blank (for VOA)
1	Delimiter	|
3	THIRD MASS	Numeric
1	Delimiter	|
5	THIRD PERCENT RELATIVE ABUNDANCE	Numeric
2	Delimiters	||
3	FOURTH MASS	Numeric
1	Delimiter	|
5	FOURTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 69	Numeric, DFTPP only/blank (for VOA)
1	Delimiter	|
3	FIFTH MASS	Numeric
1	Delimiter	|
5	FIFTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	SIXTH MASS	Numeric
1	Delimiter	|

Instrumental Data Readout Record (Type 36) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
5	SIXTH PERCENT RELATIVE ABUNDANCE	Numeric
2	Delimiters	||
3	SEVENTH MASS	Numeric
1	Delimiter	|
5	SEVENTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	EIGHTH MASS	Numeric
1	Delimiter	|
5	EIGHTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	NINTH MASS	Numeric
1	Delimiter	|
5	NINTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 176	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	TENTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	TENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	ELEVENTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	ELEVENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	TWELFTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	TWELFTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	THIRTEENTH MASS	Numeric/blank (for VOA)
2	Delimiters	||
5	THIRTEENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
1	Delimiter	|
5	PERCENT MASS OF 442	Numeric, DFTPP only (blank for VOA)
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.12 Comment Record (Type 90)

Use: To provide for operator-entered comments.
Position: May occur anywhere in the file after the type 10 record.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"90"
1	Delimiter	|
67	ANY COMMENT	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

10.0 DEFINITIONS OF VARIOUS CODES USED IN AGENCY STANDARD RECORDS

10.1 Quality Control and Related Codes (QCC) in Type 20 Records

10.1.1 NOTE: These codes appear in the quality control (QC) code fields of type 20 records. They are used to indicate the type of data that is being reported.

QCC	Name	Definition
LRB	LABORATORY (REAGENT) BLANK	The "Method Blank" (Exhibit G).
LIB	LABORATORY INSTRUMENT BLANK	The "Instrument Blank".
LSB	LABORATORY SULFUR BLANK	If different from "Method Blank" (pesticides).
LHB	LABORATORY STORAGE BLANK	The storage blank (volatiles).
FRB	FIELD BLANK	This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.
FRM	FIELD REFERENCE SAMPLE	This is any sample that is submitted for a Case and is identified as a Performance Evaluation (PE) sample.
LSD	LABORATORY SPIKE DUPLICATE BACKGROUND (ORIGINAL) VALUES	An environmental sample which is analyzed according to the analytical method, and subsequently used for the matrix spike, and the matrix spike duplicate (Exhibit G).
LF1	LABORATORY SPIKE SAMPLE _FINAL_ FIRST MEMBER	The "Matrix spike" (Exhibit G); must precede LF2.
LF2	LABORATORY SPIKE SAMPLE _FINAL_ SECOND MEMBER	The "Matrix spike Duplicate" (Exhibit G).
LCM	LABORATORY CONTROL SAMPLE	The Laboratory Control Sample (LCS) (Exhibit G).
LPC	LABORATORY PERFORMANCE CHECK SOLUTION	A solution of DFTPP (SV) or BFB (VOA) or method analytes (PEST/PCB) used to evaluate the performance of an instrument with respect to a defined set of criteria (Tune or Resolution Check Sample) (Exhibit G).
FLO	FLORISIL CHECK SOLUTION	A solution of pesticides used to check recovery from each lot of Florisil cartridges. These recovery results will be provided in every production run where associated samples are analyzed.

QCC	Name	Definition
CLM	INITIAL CALIBRATION - MULTI-POINT	The Initial Calibration for GC/MS (Exhibit G), or the Initial Individual Standard Mixes (A, B) for pesticides (Exhibit D PEST). Response factors (GC/MS) or Calibration Factors (pesticides) will be reported on the following type 30 records.
CLS	INITIAL CALIBRATION SINGLE POINT	The Initial Toxaphene/Aroclor Mixes used to determine all calibration factors (Exhibit D PEST).
CLC	CONTINUING CALIBRATION CHECK	The continuing calibration (VSTD005/ SSTD020) for GC/MS.
CLE	CONTINUING PERFORMANCE CHECK	The subsequent Individual Standard Mixes (A,B), Performance Evaluation Mixture, and for subsequent injections of Toxaphene/Aroclor mixes for pesticides (Exhibit D PEST).
CLD	DUAL PURPOSE CALIBRATION	A calibration solution as above used both as an initial calibration (CLM) and a continuing check (CLC).

10.1.2 The following QCC values are used on type 20 records which act as a header, and indicate that additional (usually calculated) analyte specific data will be present on type 30 (and following type) records. Usually, these data will apply to an entire production run, in which case they will appear immediately following the type 10 record or type 11 record if present. If the data apply to only a portion of the samples in the run, they shall be placed immediately preceding the samples to which they apply. Much of the rest of the information in the type 20 record may be blank, indicating that these data do not apply to these results.

MNC	MEAN VALUES FROM CALIBRATIONS	The data following represent mean values and percent RSDs from the initial calibration (GC/MS) or the mean calibration factors, mean retention times, and retention time windows (pesticides).

10.2 Codes For Sample Medium (Matrix, Sources)

Medium	Code
All Media, Specific Medium not Applicable. Use for Calibrations, Tunes, etc.	0 (zero)
Water	1

10.3 List of Sample and Result Qualifiers

Definition: A sample qualifier consists of three characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, or (b) produced a numeric result for an entire sample but it is qualified in some respect relating to the type or validity of the result.

10.3.1 Sample Qualifiers

Qualifier	Full Name	Definition
RIN	RE-ANALYZED	The indicated analysis results were generated from a re-injection of the same sample extract or aliquot (RE SUFFIX).
REX	RE-PREPARED	The indicated analysis results were generated from a re-extraction of the same sample (RE SUFFIX).
REJ	REJECTED	The results for the entire sample analysis have been rejected for an unspecified reason by the laboratory. For initial calibration data, these data were not utilized in the calculation of the mean.
SRN	DILUTED	The indicated analysis results were generated from a dilution of the same sample (DL SUFFIX).

10.3.2 Result Qualifiers in Type 30 Records

A result qualifier consists of three characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, or (b) produced a numeric result for a single analyte but it is qualified in some respect relating to the type or validity of the result. This qualifier is complementary to the flags field on a type 30 record. A tentatively identified compound (TIC) **must** have either a TIE, TFB, or PRE result qualifier.

BDL	BELOW DETECTABLE LIMITS	Indicates compound was analyzed for but not detected (Form 1 "U" Flag).
NAR	NO ANALYSIS RESULT	There is no analysis result required for this subject parameter.
AVG	AVERAGE VALUE	Average value -- used to report a range of values (e.g., relative response factors).
CBC	CANNOT BE CALCULATED	The analysis result cannot be calculated because an operand value is qualified (e.g., identifies analytes whose internal standard is not found) (Form 1 "X" Flag).

LTL	LESS THAN LOWER CALIBRATION LIMIT	Analysis result is from a diluted sample (DL suffix) and may be less accurate than the result from an undiluted sample (Form 1 "D" Flag).
GTL	GREATER THAN UPPER CALIBRATION LIMIT	Actual value is known to be greater than the upper calibration range (Form 1 "E" Flag).
LLS	LESS THAN LOWER STANDARD	The analysis result is less than the sample quantitation limit (Form 1 "J" Flag).
TIE	TENTATIVELY IDENTIFIED ESTIMATED VALUE	The indicated analyte is a tentatively identified analyte; its concentration has been estimated (Form 1LCF or 1LCG "J" Flag).
REJ	REJECTED	Results for the analyte are rejected by the laboratory.
STD	INTERNAL STANDARD	The indicated compound is an internal standard.
STB	INTERNAL STANDARD BELOW DETECTION LIMITS	A combination of "STD" and "BDL".
FBK	FOUND IN BLANK	The indicated compound was found in the associated method blank (LRB) as well as the sample (Form 1 "B" Flag).
TFB	TENTATIVELY IDENTIFIED AND FOUND IN BLANK	A Combination of "TIE" and "FBK" (Form 1LCF or 1LCG "B" Flag).
NRP	NON-REPRODUCIBLE	Results of two or more injections are not comparable (Form 1LCE "P" flag), e.g., Aroclor target analyte with greater than 25% difference between mean concentrations of the two column analyses.
PRE	PRESUMPTIVE PRESENCE	Presumptive evidence of presence of material for TIC (Form 1LCF or 1LCG "N" Flag).
ALC	ALDOL CONDENSATION	Labels a suspected Aldol Condensation product for TICs (Form 1LCG)

PRINTED NAME OF PURCHASE CARDHOLDER: Mary S. Price

ACCOUNT NUMBER: 4486 8600 0014 0972

ITEMS PURCHASED	VENDOR/ MERCHANT	APPROVALS (IF APPROPRIATE)	DATE ORDERED	TOTAL COST	OBJECT CLASS	DATE RECONCILED IN EAGLS	DEFAULT DCN*
Plastic inserts for ID's (500)	Identicard	SIGNATURE/TITLE	05/02/01	$148.43	26.15	05/09/01	0103JM0013
Toner for Fax (E126/A224)	Dove Data	SIGNATURE/TITLE	05/02/01	$116.70	26.15	05/11/01	0103JM0010
Floor Tread/Sealer/Floor Prep	Lab Safety	SIGNATURE/TITLE	05/03/01	$606.39	25.82	05/11/01	0103JM0010
Safety Signs/labels	Lab Safety	SIGNATURE/TITLE	5//7/01	$438.54	25.82	05/11/01	0103JM0010
Battery for 2-way radio (John J.)	Townex Communications	SIGNATURE/TITLE	05/07/01	$69.00	31.48	05/17/01	0103JM0013
Overhead bulbs (FXL)/labels	Mali	SIGNATURE/TITLE	05/10/01	$109.95	26.15	05/17/01	0103JM0010
Repair signaling module on fire pump	Honeywell	SIGNATURE/TITLE	05/14/01	$305.00	25.82	06/04/01	0103JM0013
Cartridges for Label Maker used for name plates	Mali	SIGNATURE/TITLE	05/14/01	$78.32	26.15	05/21/01	0103JM0013
Safety Glasses (Luisa S.)	Unicor	SIGNATURE/TITLE	05/15/01	$53.00	26.29		0103JM0012
Battery for 2-way Radio (Skip)	Townex Communications	SIGNATURE/TITLE	05/22/01	$116.00	31.48	06/04/01	0103JM0013
Whiteboard (A235)	Herman Miller	SIGNATURE/TITLE	05/22/01	$92.54	31.43		0103JM0010

SIGNATURE OF PURCHASE CARDHOLDER: _____

SIGNATURE OF APPROVING OFFICIAL: _____

ACCOUNT NUMBER: 4486 8600 0014 0972

FINANCIAL DATA FOR PURCHASES NOT CHARGEABLE TO CARDHOLDER'S "DEFAULT DCN"

DCN	BUDGET FISCAL YEAR	FUND	ORG	PRC	SITE/PROJ	COST/ORG	OBJ CLASS	AMOUNT

PRINTED NAME OF
PURCHASE CARDHOLDER: Mary S. Price

ACCOUNT NUMBER: 4486 8600 0014 0972

ITEMS PURCHASED	VENDOR/ MERCHANT	APPROVALS (IF APPROPRIATE)	DATE ORDERED	TOTAL COST	OBJECT CLASS	E RECONC IN EAGLS	DEFAULT DCN*
Sign for B202	Environmental Sig	SIGNATURE/TITLE	05/25/01	$60.00	25.82		0103JM0012
Toner for Xerox copier (A101)	XEROX	SIGNATURE/TITLE	05/25/01	$103.00	26.15		0103JM0013
E-Z Seal for Postage Meter	Pitney Bowes	SIGNATURE/TITLE	05/31/01	$93.09	26.15		0103JM0013
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00
		SIGNATURE/TITLE					0103JM00

SIGNATURE OF
PURCHASE CARDHOLDER: _____
TELEPHONE NUMBER: 410-305-2646 DATE: _____

SIGNATURE OF
APPROVING OFFICIAL: _____
TELEPHONE NUMBER: 215-814-5 DATE:

0003JM0009
0003JM0009
0003JM0009
0003JM0009

*PAGE 2 MUST BE COMPLETED IF ITEM PURCHASED WAS NOT CHARGED TO DEFAULT DCN/ACCOUNT NUMBER(S).

PRINTED NAME OF
PURCHASE CARDHOLDER: Mary S. Price

ACCOUNT NUMBER: 4486 8600 0000 9714

ITEMS PURCHASED	VENDOR/ MERCHANT	APPROVALS (IF APPROPRIATE)	DATE ORDERED	TOTAL COST	OBJECT CLASS	TE RECONCIL IN EAGLS	DEFAULT DCN*
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					0003JM0009
		SIGNATURE/TITLE					
		SIGNATURE/TITLE					

SIGNATURE OF
PURCHASE CARDHOLDER: _____
TELEPHONE NUMBER: 410-305-26 DATE: _____

SIGNATURE OF
APPROVING OFFICIAL: _____
TELEPHONE NUMBER: 215-814- DATE: _____

*PAGE 2 MUST BE COMPLETED IF ITEM PURCHASED WAS NOT CHARGED TO DEFAULT DCN/ACCOUNT NUMBER(S).