
Superfund



SUPERFUND ANALYTICAL METHODS FOR LOW CONCENTRATION WATER FOR ORGANICS ANALYSIS

SUPERFUND ANALYTICAL METHODS

FOR

LOW CONCENTRATION WATER FOR ORGANICS ANALYSIS

6/91

U.S. Environmental Protection Agency
Region 5, Library (PL-12J)
77 West Jackson Boulevard, 12th Floor
Chicago, IL 60604-3590

6/91

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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SECTION I

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

The following table reiterates the Contract reporting and deliverables requirements specified in the Contract Schedule and specifies the distribution that is required for each deliverable. NOTE: Specific recipient names and addresses are subject to change during the term of the contract. SMO will notify the Contractor in writing of such changes when they occur.

Item	No. Copies	Delivery Schedule	<u>Distribution</u>			
			(1)	(2)	(3)	(4)
1. Updated SOPs	3	60 days after contract award and as required in Exhibit E.		X	X	X
*2. Sample Traffic Reports	1	3 days after receipt of last sample in Sample Delivery Group (SDG). **	X			
***3. Sample Data Summary Package	1	14 days after receipt of last sample in SDG.	X			
***4. Sample Data Package	2	14 days after receipt of last sample in SDG.	X		X	
*** 5. Complete SDG File ****	1	14 days after receipt of last sample in SDG.			X	
***6. Data in Computer-Readable Form	1	14 days after receipt of last sample in SDG.	X			

Distribution:

- (1) Sample Management Office (SMO)
- (2) EMSL-LV
- (3) Region-Client (Technical Project Officer)
- (4) NEIC

Item	No. Copies	Delivery Schedule	Distribution			
			(1)	(2)	(3)	(4)
7.	GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request.		As Directed	
8.	Extracts	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request.		As Directed	
9.*****	QA Plan	3 Copy	Submit within 60 days after contract award, and as required in Exhibit E.		As Directed	

NOTE: Contractor must be prepared to receive the full contract sample requirement at the time of contract award.

* Also required in the Sample Data Summary Package.

** Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less and not exceeding 20 samples. 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 all samples have been delivered.

*** Concurrent delivery required. Delivery shall be made such that all designated recipients receive the item on the same calendar day.

**** Complete SDG file will contain the original sample data package plus all of the original documents described under Complete SDG File paragraph 5.

***** See Exhibit E for a more detailed description.

NOTE: As specified in the Contract Schedule (Section G, Government Furnished Supplies and Materials), unless otherwise instructed by the CLP Sample Management Office, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than sixty (60) 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.

Distribution Addresses:

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

For overnight delivery service, use street address:
300 North Lee Street, Suite 200
Alexandria, VA 22314

- (2) USEPA Environmental Monitoring
Systems Laboratory (EMSL-LV)
P. O. Box 15027
Las Vegas, NV 89114
ATTN: Data Audit Staff

For overnight delivery service, use street address:

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

- (3) USEPA REGIONS:

The CLP Sample Management Office will provide the Contractor with the list of addressees for the ten EPA 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.

- (4) USEPA National Enforcement Investigations Center (NEIC)
ATTN: CLP Audit Program
Denver Federal Center Bldg. 53
P.O. Box 25227
Denver, CO 80225

SECTION II

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

The Contractor laboratory shall provide reports and other deliverables as specified in the Contract Schedule (Performance/Delivery Schedule, Section F). The required content and form of each deliverable is described in this Exhibit.

All reports and documentation MUST BE:

- o Legible
- o Clearly labeled and completed in accordance with instructions in this Exhibit
- o Arranged in the order specified in this Section
- o Paginated consecutively in ascending order starting from the SDG Narrative

If submitted documentation does not conform to the above criteria, the Contractor will be required to resubmit such documentation with the deficiencies corrected, at no additional cost.

Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through a SMO action, or through a Regional Data Reviewer's request, the data must be clearly marked as ADDITIONAL DATA and must be sent to all three contractual data recipients (SMO, EMSL/LV, and Region). A cover letter shall be included which describes what data are being delivered, to which EPA Case(s)/SDGs it pertains, and who requested the data.

Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data must be sent to all three contractual data recipients (SMO, EMSL/LV, and Region), and in all three instances must be accompanied by a color-coded COVER SHEET (Laboratory Response To Results of Contract Compliance Screening) provided by SMO.

Descriptions of the requirements for each deliverable item cited in the Contract Performance/Delivery Schedule (Contract Schedule, Section F) are specified in this Section. Items submitted concurrently MUST BE arranged in the order listed. The components of each item MUST BE arranged in the order presented in this Section when the item is submitted.

Section III contains the form instructions to assist the Contractor in providing all the required data. Section IV of this Exhibit contains copies of the required data reporting forms in specified formats.

1. QUALITY ASSURANCE PLAN AND STANDARD OPERATING PROCEDURES

See contract for specifications.

2. SAMPLE TRAFFIC REPORTS

- 2.1 Original Sample Traffic Report page marked "Lab Copy for Return to SMO" with lab receipt information and signed in original Contractor signature, for each sample in the Sample Delivery Group.
- 2.2 Traffic Reports (TRs) shall be submitted in Sample Delivery Group (SDG) sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.
- 2.3 The SDG Cover Sheet shall contain the following items:
- o Lab name
 - o Contract number
 - o Sample analysis price - full sample price from contract.
 - o Case number
 - o List of EPA Sample Numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt (LRDs).
- 2.4 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).
- 2.5 The EPA Sample Number of the first sample received in the SDG is the SDG number. Each Traffic Report must be clearly marked with the SDG Number. This information should be entered below the Lab Receipt Date on the TR. The TR for the last sample received in the last SDG shipment must be clearly marked "SDG - FINAL SAMPLE."
- 2.6 If samples are received at the laboratory with multi-sample Traffic Reports (TRs), all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the laboratory must make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.

3. SAMPLE DATA SUMMARY PACKAGE

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

- o SDG Narrative
 - o Organics Analysis Data Sheet for target compound results (Form I) and for tentatively identified compounds (Form I, TIC) by fraction (VOA, SV, PEST) and by sample within each fraction. (No Form I, TIC for PEST fraction.)
 - o Surrogate Recovery (Form II) by fraction (VOA, SV, PEST)
 - o Laboratory Control Sample Recovery (Form III) by fraction (VOA, SV, PEST)
 - o Method Blank Summary (Form IV), Organics Analysis Data Sheet for target compound results (Form I) and for tentatively identified compounds (Form I, TIC) by fraction (VOA, SV).
 - o Peak Area and Retention Time Summary of Internal Standards for initial calibration standards (Form VIII) and samples (Form VIII) by fraction (VOA, SV only).
- 3.3 Sample data forms shall be arranged in increasing EPA Sample Number order.

4. **SAMPLE DATA PACKAGE**

- 4.1 The Sample Data Package shall include data for analyses of all samples in each Sample Delivery Group, specifically including field samples, reanalyses, dilutions, blanks, Laboratory Control Samples, and Performance Evaluation Samples. The Sample Data Package is divided into the five major units as follows:

- o SDG Narrative
- o Sample Traffic Reports
- o Volatiles Data
- o Semivolatiles Data
- o Pesticides/Aroclors Data

- 4.2 The Volatiles, Semivolatiles, and Pesticides/Aroclors data are each specific to an analytical fraction. If the analysis of that fraction is not required, then that fraction-specific unit is not a required deliverable.

The Contractor shall retain a copy of the sample data package for 365 days after final acceptance of data. After this time, the Contractor may dispose of the package.

4.3. SDG Narrative

- 4.3.1 This document shall be clearly labeled "SDG Narrative". The SDG Narrative shall contain: laboratory name; Case number; EPA Sample Numbers in the Sample Delivery Group (SDG), differentiating between initial analyses, dilutions and reanalysis; 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.

- 4.3.2 Whenever data from sample reanalyses are submitted, the Contractor shall state in the SDG Narrative for each reanalysis, whether it considers the reanalysis to be billable, and if so, why. A copy of the narrative should be sent to SMO for their review. The Contractor must also include any problems encountered; both technical and administrative, the corrective actions taken, and resolution.
- 4.3.3 The Contractor must also 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 volatiles samples were acidified in the field. No pH adjustment is to be performed by the Contractor on water samples for volatiles analysis.
- 4.3.4 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 data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or his designee, as verified by the following signature." This statement shall be directly followed by signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.
- 4.3.5 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 samples in the SDG Narrative.
- 4.3.6 The SDG Narrative itself must be signed with original signature by the Laboratory Manager or his designee and dated.

4.4 Sample Traffic Reports

Copies of the Sample Traffic Reports for all of the samples in the SDG are also included in the Sample Data Summary Package. The Traffic Reports shall be arranged in increasing EPA Sample Number order, considering both letters and numbers in ordering samples. Copies of the SDG cover sheet are to be included with the copies of the Traffic Reports.

If samples are received at the laboratory with multi-sample Traffic Reports (TRs), not all samples on one multi-sample TR are necessarily in the same SDG. In this instance, the laboratory must make the appropriate number of photocopies of the TR so that a copy is submitted with each data package to which it applies. In addition, in any instance where samples from more than one multi-sample TR are in the

same data package, the laboratory must submit a copy of the SDG cover sheet with copies of the TRs.

4.5 Volatiles Data

4.5.1 Volatiles QC Summary

If more than a single form is necessary, each type of form must be arranged in chronological order by instrument.

- o Surrogate Recovery (Form II LCV)
- o Laboratory Control Sample Recovery (Form III LCV)
- o Method Blank Summary (Form IV LCV)
- o GC/MS Tuning and Mass Calibration - BFB (Form V LCV)
- o Internal Standard Area and Retention Time Summary (Form VIII LCV)

4.5.2 Volatiles Sample Data

Sample data, including PES, shall be arranged in packets with both of the Organic Analysis Data Sheets (Form I LCV and Form I LCV-TIC), followed by the raw data for volatile samples. These sample packets should then be placed in increasing EPA Sample Number order.

4.5.2.1 Organics Analysis Data Sheet for target compound results (Form I LCV).

4.5.2.2 Organics Analysis Data Sheet for tentatively identified compounds (Form I LCV-TIC). This form must be included even if no TIC's are found.

4.5.2.3 Reconstructed total ion chromatograms (RIC)

The RIC for each sample, extract, standard, and blank must be normalized to the largest nonsolvent component, and must contain the following header information:

- o EPA Sample Number
- o Date and time of analysis
- o GC/MS instrument ID
- o Lab file ID

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

4.5.2.4 Quantitation Report

If automated data systems are used for quantitation of the target compounds, the complete data system quantitation report must be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system quantitation 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" quantitation report containing the following information must be included in the sample data package in addition to the chromatogram.

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

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 include the scan range integration.

4.5.2.5 Target Compound Mass Spectra

For each sample, by each compound identified, copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra are required. The raw spectra and the background-subtracted spectra must be labeled with EPA Sample Number, lab file ID, date and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra.

4.5.2.6 Tentatively Identified Compound Mass Spectra and Library Matches

For each sample, by each compound identified, copies of mass spectra of non-target and non-surrogate organic compounds (Tentatively Identified Compounds) with the associated spectra of the three best library matches are labeled with EPA Sample Number, lab file ID, date and time of analysis, and GC/MS instrument ID.

4.5.3 Volatiles Standards Data

4.5.3.1 Initial Calibration

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

Initial Calibration Summary (Form VI LCV).

Internal Standard Area and Retention Time Summary (Form VIII LCV)

Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five point) calibration are labeled as in Paragraphs 4.5.2.3 and 4.5.2.4. Spectra are not required.

4.5.3.2 Continuing Calibration

When more than one continuing calibration is performed, the reconstructed ion chromatogram and quantitation reports and each type of form must be in chronological order, and by instrument if more than one instrument is used.

Continuing Calibration Summary (Form VII LCV)

Internal Standard Area and Retention Time Summary (Form VIII LCV)

VOA standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12 hour) calibrations are labeled as in Paragraph 4.5.2.3 and 4.5.2.4. Spectra are not required.

4.5.4 Volatiles QC Data

4.5.4.1 GC/MS Tuning Data

GC/MS Tuning - BFB data, for each 12-hour period, shall be arranged in chronological order by instrument for each GC/MS system utilized.

GC/MS Tuning and Mass Calibration - BFB (Form V LCV)

Bar graph spectrum, labeled as in Paragraph 4.5.2.3.

Mass listing, labeled as in Paragraph 4.5.2.3.

4.5.4.2 Blank Data

Blank data shall be arranged in chronological order by instrument. NOTE: This order is different from that used for samples.

Blank data shall be arranged in packets with both of the Organic Analysis Data Sheets (Form I LCV and Form I LCV-TIC), followed by the raw data for volatile samples (see paragraphs 4.5.2.1 to 4.5.2.6).

4.5.4.3 Laboratory Control Sample Data

Organics Analysis Data Sheet for target compound results (Form I LCV). Form I LCV-TIC is not required.

Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Paragraph 4.5.2.3 and 4.5.2.4. Spectra are not required.

4.6 Semivolatiles Data

4.6.1 Semivolatiles QC Summary

If more than a single form is necessary, each type of form must be arranged in chronological order, by instrument.

- o Surrogate Recovery (Form II LCSV)
- o Laboratory Control Sample Recovery (Form III LCSV)
- o Method Blank Summary (Form IV LCSV)
- o GC/MS Tuning and Mass Calibration - DFTPP (Form V LCSV)
- o Internal Standard Area and Retention Time Summary (Form VIII LCSV-1, LCSV-2)

4.6.2 Semivolatiles Sample Data

Sample data, including PES, shall be arranged in packets with both of the Organic Analysis Data Sheets (Form I LCSV and Form I LCSV-TIC), followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing EPA Sample Number order.

4.6.2.1 Organic Analysis Data Sheet for target compound results (Form I LCSV-1, LCSV-2).

4.6.2.2 Tentatively Identified Compounds (Form I LCSV-TIC). This form must be included even if no TICs are found.

4.6.2.3 Reconstructed total ion chromatograms (RICs)

The RIC for each sample, extract, standard, and blank must be normalized to the largest nonsolvent component, and must contain the following header information:

- o EPA Sample Number
- o Date and time of analysis
- o GC/MS instrument ID
- o Lab file ID

Internal standard and surrogate spiking compounds are to be labeled with the names of the compounds, either directly on the peak, or on a print-out of retention times if retention times are printed over the peak.

4.6.2.4 Quantitation Reports

If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system quantitation report must be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system quantitation 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" quantitation report containing the following information must be included in the sample data package in addition to the chromatogram.

- o EPA Sample Number
- o Date and time of analysis

- o RT or scan number of identified target compounds
- o Ion used for quantitation with measured area
- o Copy of peak area table from data system
- o GC/MS instrument ID
- o Lab file ID

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 include the scan range integration.

4.6.2.5 Target Compound Mass Spectra

For each sample, by each compound identified, copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra are required. The raw spectra and the background-subtracted mass spectra must be labeled with EPA Sample Number, lab file ID, date and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra.

4.6.2.6 Tentatively Identified Compound Mass Spectra and Library Matches

For each sample, by each compound identified, copies of mass spectra of non-target and non-surrogate organic compounds (Tentatively Identified Compounds) with the associated spectra of the three best library matches are labeled with EPA Sample Number, lab file ID, date and time of analysis, and GC/MS instrument ID.

4.6.3. Semivolatiles Standards Data

4.6.3.1 Initial Calibration

Data must be included for all calibration analyses pertaining to the SDG. When more than one initial calibration is performed, the reconstructed ion chromatogram and quantitation reports and each type of form must be put in chronological order, by instrument.

Initial Calibration Data (Form VI LCSV-1, LCSV-2).

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

Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five point) calibration are labeled as in Paragraphs 4.6.2.3 and 4.6.2.4. Spectra are not required.

4.6.3.2 Continuing Calibration

When more than one continuing calibration is performed, the reconstructed ion chromatogram and quantitation reports and each type of form must be in chronological order, by instrument.

Continuing Calibration Summary (Form VII LCSV-1, LCSV-2).

Internal Standard Area and Retention Time Summary (Form VIII LCSV-1, LCSV-2).

Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12 hour) calibrations are labeled as in Paragraphs 4.6.2.3 and 4.6.2.4. Spectra are not required.

4.6.4. Semivolatiles QC Data

4.6.4.1 GC/MS Tuning Data

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

GC/MS Tuning and Mass Calibration-BFB (Form V LCSV)

Bar graph spectrum, labeled as in Paragraph 4.6.2.3.

Mass listing, labeled as in Paragraph 4.6.2.3.

4.6.4.2 Blank Data

Blank data shall be arranged in chronological order by instrument. NOTE: This order is different from that used for samples.

Blank data shall be arranged in packets with both of the Organic Analysis Data Sheets (Form I LCSV and Form I LCSV-TIC), followed by the raw data for

semivolatile samples (see paragraphs 4.6.2.1 to 4.6.2.6)

4.6.4.3 Laboratory Control Sample Data

Organic Analysis Data Sheet for target compounds (Form I LCSV-1, LCSV-2). Form I LCSV-TIC is not required.

Reconstructed ion chromatogram(s) and quantitation report(s), are labeled as in Paragraphs 4.6.2.3 and 4.6.2.4. Spectra are not required.

4.7 Pesticide/Aroclor Data

4.7.1 Pesticide/Aroclor QC Summary

If more than a single form is necessary, forms must be arranged in chronological order by instrument.

- o Surrogate Percent Recovery Summary (Form II LCP)
- o Laboratory Control Sample Recovery (Form III LCP)
- o Method Blank Summary (Form IV LCP)

If more than a single method blank summary form is necessary, forms must be arranged in chronological order by type (method or sulfur blank) by instrument, and by date of analyses.

4.7.2 Pesticide/Aroclor Sample Data

Sample data, including PES, shall be arranged in packets with the Organic Analysis Data Sheet (Form I LCP), followed by the raw data for pesticide samples. These sample packets should then be placed in increasing EPA Sample Number order.

- 4.7.2.1 Organic Analysis Data Sheet for target compounds (Form I LCP).
- 4.7.2.2 Pesticide Identification Summary for Single Component Analytes (Form X LCP-1), only required for positively identified analytes.
- 4.7.2.3 Pesticide Identification Summary for Multicomponent Analytes (Form X LCP-2), only required for positively identified analytes.
- 4.7.2.4 Pesticide chromatograms

All chromatograms must be labeled with the following information:

- o EPA Sample Number
- o Volume injected (ul)
- o Date and time of analyses
- o GC column identification (by stationary phase and internal diameter)
- o GC instrument identification
- o Scaling Factor
- o Positively identified compounds must be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak.

4.7.2.5 Copies of pesticide chromatograms from second GC column, labeled as in Paragraph 4.7.2.4.

4.7.2.6 Data System Printouts

Data system printouts of retention time and corresponding peak areas or height must accompany each chromatogram are labeled with the following information:

- o EPA Sample Number
- o Volume injected (ul)
- o Date and time of analyses
- o GC column identification (by stationary phase and internal diameter)
- o GC instrument identification
- o Scaling Factor
- o Positively identified compounds must be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak.

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 include the integration time range.

4.7.2.7 Manual work sheets.

4.7.3 Pesticide/Aroclor Standards Data

4.7.3.1 Initial Calibration

Data must be included for all calibration analyses pertaining to the SDG. When more than one initial calibration is performed, the data and each type of form must be put in chronological order, by instrument and GC column.

Initial Calibration for Single Component Analytes (Form VI LCP-1, LCP-2).

Initial Calibration for Multicomponent Analytes (Form VI LCP-3).

Resolution Check Summary (Form VI LCP-4).

Analytical Sequence (Form VIII LCP), containing initial calibration standards.

4.7.3.2 Calibration Verification

Calibration Verification Summary (Form VII LCP) for all GC columns.

When more than one calibration verification is performed, forms must be in chronological order, by instrument and GC column.

4.7.3.3 Chromatograms and data system printouts are required for all standards and arranged in chronological order by instrument and each GC column:

- o Resolution Check Mixture.
- o Performance Evaluation Mixtures, each initial calibration and all those that bracket samples in the SDG.
- o Individual Standard Mixture A, at three concentrations, each initial calibration, plus all those that bracket samples in the SDG.
- o Individual Standard Mixture B, at three concentrations, each initial calibration, plus all those that bracket samples in the SDG.

- o All multicomponent analytes (Toxaphene and Aroclors), each initial calibration.
- o All multicomponent analyte standards analyzed for confirmation.

4.7.3.4 Data system printouts of retention times and corresponding peak areas or peak heights must accompany each chromatogram. In addition, all chromatograms and data system printouts are required to be labeled with the following:

- o EPA Sample Number for the standard, i.e., INDA1, INDA2, etc. (See Forms Instructions for details).
- o Label all standard peaks for all individual compounds either directly out from the peak or on the printout of retention times if retention times are printed over the peak.
- o Total nanograms injected for each standard.
- o Date and time of injection.
- o GC column identification (by stationary phase and internal diameter).
- o GC instrument identification.
- o Scaling factor

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 include the integration time range.

4.7.4 Pesticide/Aroclor QC Data

4.7.4.1 Blank Data

Blank data instrument - grouped by type of blank (i.e., method and sulfur) and arranged in chronological order. NOTE: This order is different from that used for samples.

Organics Analysis Data Sheet for target compounds (Form I LCP).

Blank data shall be arranged in packets with the Organics Analysis Data Sheet (Form I LCP) followed by the raw data (paragraph 4.7.2.2, to 4.7.2.7).

4.7.4.2 Laboratory Control Sample

Organics Analysis Data Sheet for target compounds (Form I LCP).

Chromatograms and data system printouts are labeled as in Paragraph 4.7.2.4 and 4.7.2.6.

4.7.4.3 Florisil Cartridge Check

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

Each Form IX LCP shall be followed by the chromatograms and data system printouts, labeled as in 4.7.2.4 and 4.7.2.6.

5. COMPLETE SDG FILE

As specified in the Delivery Schedule, one Complete SDG File (CSF) including the original sample data package shall be delivered to the Region concurrently with delivery of copies of the Sample Data Package to SMO and EMSL/LV. The contents of the CSF will be numbered according to the method described in Section III of Exhibit B. The Document Inventory Sheet, Form DC-2, is contained in Section IV. The CSF will contain all original documents where possible. No copies will be placed in the CSF unless the originals are bound in a logbook which is maintained by the laboratory. The CSF will contain all original documents specified in Section III, and Form DC-2 of Exhibit B.

The CSF will consist of the following original documents in the order listed in paragraph 5.1 through 5.6 below:

- 5.1 The original sample data package (see Exhibit B, Section 4).
- 5.2 A completed and signed Document Inventory Sheet (Form DC-2).
- 5.3 All original shipping documents, including, but not limited to, the following documents:
 - o EPA Chain of Custody Record
 - o Airbills.
 - o EPA Traffic Reports.
 - o Sample Tags (if present) sealed in plastic bags.
- 5.4 All original receiving documents, including, but not limited to, the following documents:
 - o Form DC-1

- o Other receiving forms or copies of receiving logbooks
 - o SDG Cover Sheet
- 5.5 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:
- o Original preparation and analysis forms or copies of preparation and analysis logbook pages.
 - o Internal sample and sample extract transfer chain-of-custody records.
 - o Screening records.
 - o All instrument output, including strip charts from screening activities.
- 5.6 All other original SDG-related documents in the possession of the laboratory, including, but not limited to, the following documents:
- o Telephone contact logs
 - o Copies of personal logbook pages
 - o All hand written SDG-specific notes
 - o Any other SDG specific documents not covered by the above.

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 EPA, as well as copies that are altered in any fashion, are also deliverables to EPA. (Original to the Region and copies to SMO and EMSL/LV).

If the laboratory does submit SDG-specific documents to EPA after submission of the CSF, the documents shall be numbered as an addendum to the CSF and a revised DC-2 form shall be submitted, or the documents should be numbered as a new CSF and a new DC-2 form should be submitted. The revised DC-2 form is sent to the Region only.

6. DATA IN COMPUTER-READABLE FORM

The Contractor shall provide a computer-readable copy of the data on data reporting Forms I-X for all samples in the Sample Delivery Group, as specified in the Contract Performance/Delivery Schedule. Computer-readable data deliverables shall be submitted on IBM or IBM-compatible, 5.25 inch double-sided, double density 360 K-byte or a high density 1.2 M-byte diskette or 3.5 inch double-sided double density 720 K-byte or 1.44 M-byte diskette.

When submitted, diskettes 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) must be included in the same shipment as the hardcopy data and shall, at a minimum, be enclosed in a diskette mailer. The data shall be recorded in ASCII text file format, and shall adhere to the file, record and field specifications listed in

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

7. GC/MS TAPES

See Exhibit E for requirements.

8. EXTRACTS

The Contractor shall store sample extracts at 4°C ($\pm 2^\circ\text{C}$) in bottles/vials with Teflon-lined septa. Extract bottles/vials shall be labeled with EPA Sample Number, Case number and Sample Delivery Group (SDG) number. A logbook of stored extracts shall be maintained, listing EPA Sample Numbers and associated Case and SDG numbers.

The Contractor is required to retain extracts for 365 days following data submission. During that time, the Contractor shall submit extracts and associated logbook pages within seven days following receipt of a written request from the Sample Management Office.

SECTION III

FORM INSTRUCTION GUIDE

This section includes specific instructions for the completion of all required forms. Each of the forms is specific to a given fraction (volatile, semivolatile, pesticide/Aroclor). The Contractor shall submit only those forms pertaining to the fractions analyzed for a given sample or samples. For instance, if a sample is scheduled for volatile analysis only, submit only VOA forms. There are two pages relating to the semivolatile fraction for Forms I, VI, VII, and VIII and four pages relating to the pesticide/Aroclor fraction for Form VI. Whenever semivolatiles or pesticides/Aroclors are analyzed and one of the above named forms is required, all pages (LCSV-1, LCSV-2, etc.) must be submitted. These instructions are arranged in the following order:

1. General Information and Header Information
2. Organic Analysis Data Sheet (Form I, All Fractions)
3. Surrogate Recovery (Form II, All Fractions)
4. Laboratory Control Sample Recovery (Form III, All Fractions)
5. Method Blank Summary (Form IV, All Fractions)
6. GC/MS Tuning and Mass Calibration (Form V LCV, LCSV)
7. Initial Calibration Summary (Form VI, All Fractions)
8. Pesticide Resolution Check Summary (Form VI LCP-4)
9. Continuing Calibration Summary (Form VII LCV, LCSV)
10. Calibration Verification Summary (Form VII LCP)
11. Internal Standard Area and Retention Time Summary (Form VIII LCV, LCSV)
12. Pesticide/Aroclor Analytical Sequence (Form VIII LCP)
13. Pesticide/Aroclor Florisil Cartridge Check (Form IX LCP)
14. Pesticide/Aroclor Identification (Form X LCP)
15. Sample Log-In Sheet (Form DC-1)
16. Document Inventory Sheet (Form DC-2)

1. GENERAL INFORMATION AND HEADER INFORMATION

- 1.1 The data reporting forms presented in Section IV have been designed in conjunction with the computer-readable data format specified in Exhibit H, Data Dictionary and Format for Data Deliverables in Computer-Readable Format. The specific length of each variable for computer-readable data transmission purposes is given in the data dictionary (Exhibit H). Information entered on these forms must not exceed the size of the field given on the form, including such laboratory-generated items as Lab Name and Lab Sample ID.
- 1.2 Note that on the hardcopy forms (Section IV), the space provided for entries is greater in some instances than the length prescribed for the variable as written to diskette (see Exhibit H). Greater space is provided on the hardcopy forms for visual clarity.
- 1.3 Values must be reported on the hardcopy forms according to the individual form instructions in this Section. For example, results for concentrations of VOA target compounds must be reported to two significant figures if the value is greater than or equal to 10. Values can be written to the diskette file in any format that does not exceed the field specification as given in the record specifications and discussed in "Record Structure", in Exhibit H.
- 1.4 For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals exactly 5, round up if the digit to be retained is odd, and round down if that digit is even.
- 1.5 All characters which appear on the data reporting forms presented in the contract (Exhibit B, Section IV) must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in this Superfund Analytical Method. No information may be added, deleted, or moved from its specified position without prior written approval by SMO. The names of the various fields and compounds (i.e., "Lab Code", "Chloromethane") on the uncompleted forms must appear as they do in the this Superfund Analytical Method (Section IV of this exhibit), except that the use of uppercase and lowercase letters is optional.
- 1.6 Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters. 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. (See Exhibit H for more detailed instructions.) However, do not remove the underscores or vertical bar characters 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, see Form II LCV, line 30. If data must be entered on line 30, it will replace the underscores).

- 1.7 Forms II, IV, V, VIII, IX, and X contain a field labeled "page _ of _" in the bottom left-hand 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 them consecutively, as "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, Form II LCSV, and Form II LCP are for different data. Therefore, do not number the pages of all three versions of Form II as "1 of 3, 2 of 3, etc." Only number pages within a fraction-specific form.
- 1.8 Six pieces of information are common to the header sections of each data reporting form. They are: Lab Name, Contract, Lab Code, Case No., SAS No., and SDG No. This information, if it applies, must be entered on every form and must match on every form.
- 1.8.1 The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.
- 1.8.2 The "Lab Code" is an alphabetical abbreviation of up to 6 letters, assigned by SMO, to identify the laboratory and aid in data processing. This lab code shall be assigned at the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of SMO. If a change of name or ownership occurs at the laboratory, the lab code will remain the same until the Contractor is directed by SMO to use another lab code assigned by SMO.
- 1.8.3 The "Case No." is the assigned Case Number (up to 5 digits) associated with the sample, and reported on the Traffic Report.
- 1.8.4 The "Contract" is the number of the SMO contract under which the analyses were performed.
- 1.8.5 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).
- 1.8.6 The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services. If samples are to be analyzed under SAS only, and reported on these forms, then enter SAS No., and leave Case No. blank. If samples are analyzed according to the "Routine Analytical Services" (IFB) protocols and have additional "SAS" requirements, list both Case No. and SAS No. on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. NOTE: Some samples in an SDG may have a SAS No. while others do not.

1.9 EPA Sample Number

1.9.1 EPA Sample Number must be entered on several of the forms. This field 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 "EPA Sample No." is entered into the triple-spaced box in the upper right-hand corner of the form, it should be entered on the middle line of the three lines that comprise the box.

1.9.2 All samples, including Laboratory Control Samples and Performance Evaluation Samples, blanks, and standards shall be identified with an EPA Sample Number.

1.9.3 For samples, the EPA Sample Number is the unique identifying number given in the Traffic Report that accompanied that sample. In order to facilitate data assessment, the following identification scheme must be used for samples:

XXXXX - EPA Sample Number assigned
XXXXXRE - re-analyzed sample
XXXXXDL - sample analyzed at a dilution
XXXXXDL2 - sample analyzed at a secondary dilution (for PEST only)

1.9.4 The EPA Sample Number must be unique for each Laboratory Control Sample within an SDG. The EPA Sample Number for a Laboratory Control Sample must be FLCS##, where:

F - fraction (V for volatiles; S for semivolatiles; 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.

1.9.5 The EPA Sample Number must be unique for each blank within an SDG. Within a fraction, a laboratory must replace the "##" 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.

Volatile method blanks shall be identified as VBLK##.

Volatile storage blank shall be identified in VSBK##.

Volatile instrument blank shall be identified as VIBLK##.

Semivolatile method blanks shall be identified as SBLK##.

Pesticide/Aroclor method blanks shall be identified as PBLK##.

Pesticide/Aroclor instrument blanks shall be identified as PIBLK##.

If a separate sulfur cleanup blank is required (e.g., when not all Pesticide/Aroclor samples associated with a given method blank are subjected to sulfur cleanup) the Pesticide/Aroclor sulfur cleanup blanks shall be identified as PCBLK##.

- 1.9.6 The EPA Sample Number must be unique for each standard within an SDG.

The EPA Sample Numbers for volatile and semivolatile standards must be FSTD###, where:

F - fraction (V for volatiles; S for semivolatiles).

STD - indicates a standard.

- the concentration in ug/L of volatile standards (i.e., 001, 002, 005, 010) or the amount injected in ng for semivolatile standards (i.e., 005, 010, 020, 050, and 080). These designations will have to be concatenated with other information to uniquely identify each standard in the SDG.

For pesticide/Aroclor standards, the following scheme shall be used to enter EPA Sample Number.

<u>Name</u>	<u>EPA Sample Number</u>
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##
Aroclor 1254	AR1254##
Aroclor 1260	AR1260##
Aroclor 1016/1260	AR1660##

The laboratory must create a unique "EPA Sample No." within an SDG by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both.

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 may not be used.

- 1.10 Several other pieces of information are common to the header information on some of the data reporting forms. These include: Lab Sample ID, Lab File ID, Purge or Sample Volume, GC Column - ID, Instrument ID, Time Analyzed, Date Received, Extracted, and Date Analyzed.
- 1.10.1 "Lab Sample ID" is an optional laboratory-generated internal identifier. Up to 12 alpha-numeric characters may be reported here.
- 1.10.2 "Lab File ID" is the laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. Up to 14 alpha-numeric characters may be used here.
- 1.10.3 "Purge Volume" or "Sample Volume" is the total volume of water that was purged or extracted, in milliliters.
- 1.10.4 There are two fields to be entered under "GC Column - ID". Enter the stationary phase of the GC column after "GC Column" and enter the internal diameter in millimeters after "ID".
- 1.10.5 "Instrument ID" is the identifier that distinguishes each instrument used for analysis in the SDG.
- 1.10.6 The "Time Analyzed" shall be in military time.
- 1.10.7 "Date Received" is the date of sample receipt at the laboratory, as noted on the Sample Traffic Report (i.e., the Validated Time of Sample Receipt). "Date Received" is entered as MM/DD/YY.
- 1.10.8 Enter the date on which the extraction procedure was started for "Date Extracted". "Date Extracted" is entered as MM/DD/YY.
- 1.10.9 For each fraction, the "Date Analyzed" is the date of the sample analysis. 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. "Date Analyzed" is entered as MM/DD/YY.
- 1.11 For pesticide/Aroclors, analyses on two GC columns are required. The information on the two analyses is differentiated on some of the forms as "Date Analyzed (1)", "Date Analyzed (2)", etc. The order of reporting is not important, but must be consistent with the information reported on Form X. When simultaneous injection is made on both GC columns, the dates (and times) will be the same. If simultaneous injections are not made, the (1) shall refer to the first analysis, and (2) the second. If only one analysis is required, leave blank the fields for the second analysis.

2. ORGANIC ANALYSIS DATA SHEET (FORM I)

2.1 Target Compounds. Form I LCV, LCSV-1, LCSV-2, and LCP

This form is used for reporting the detected concentrations of the target compounds in the samples, Laboratory Control Samples, Performance Evaluation Samples, and all blanks analyzed, including method blanks, instrument blanks, sulfur cleanup blanks, and storage blanks.

Complete the header information on each Form I required, according to the instructions in paragraph 1.

Enter 1 for the "Dilution Factor", if a sample was not diluted or concentrated for analysis. If a sample has been diluted for analysis, enter the "Dilution Factor" as a single number, such as 100 when a sample is diluted by a factor of 100. Enter 0.1 when a sample is concentrated by a factor of 10.

For volatiles, the "Purge Volume" is the total volume (in mL) purged for the analysis.

For semivolatiles and pesticides, enter the "Concentrated Extract Volume" and the "Injection Volume" in microliters. The "Concentrated Extract Volume" is the actual volume of the most concentrated sample extract. 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. Enter the "pH" of the sample before extraction, reported to 0.1 pH units. Enter "Y" or "N" for "Yes" or "No" under the "Sulfur Cleanup" for the pesticides.

In the concentration column, for positively identified target compounds, the Contractor shall report the concentrations as uncorrected for blank contaminants.

For volatile and semivolatile results, report analytical results to one significant figure if the value is less than 10, and to two significant figures if greater than or equal to 10.

Report all pesticide/Aroclor results to two significant figures.

If the analytical result is greater than or equal to the quantitation limit, report the result.

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

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

The nine defined qualifiers to be used are as follows:

- U - Indicates compound was analyzed for but not detected. The numerical value is the sample quantitation limit and must be corrected for dilution. For example, 5 U for phenol in water if the sample final volume is the protocol-specified final volume. If a 1 to 10 dilution of the extract is necessary, the reported limit is 50 U.
- J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero. For example, if the sample quantitation limit is 10 ug/L, but a concentration of 3 ug/L is calculated, report it as 3J. The sample quantitation limit must be adjusted for dilution as discussed for the U flag.
- N - Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds, where the identification is based on a mass spectral library search. It is applied to all TIC results. For generic characterization of a TIC, such as chlorinated hydrocarbon, the N code is not used.
- B - This flag is used on the sample Form I when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified target compound.

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 initial calibration range of the instrument for that specific analysis. If one or more compounds have a response that exceed the initial calibration range, the sample or extract must be diluted and reanalyzed according to the specifications in Exhibit D. All such compounds should have the concentration flagged with an "E" on the Form I for the original analysis. The dilution of the extract may cause some compounds identified in the first analysis to be below the calibration range in the second analysis. The results of both analyses shall be reported on separate Forms I. The Form I for the diluted sample shall have the "DL" (or "DL2") (for pesticide samples only) suffix appended to the EPA Sample Number. NOTE: For total xylenes, where three isomers are quantified as two peaks, the calibration range 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 µg/L or the peak representing the two coeluting isomers on that GC column exceeds 50 µg/L.

- D - If a sample or extract is diluted and re-analyzed, as in the "E" flag above, all concentration values reported on that Form I are flagged with the "D" flag. The "DL" or "DL2" (for pesticide samples only) suffix is appended to the EPA Sample Number on the Form I for the diluted sample.
- A - This flag is not used under this contract, but is reserved.
- P - This flag is used for a pesticide/Aroclor target analyte when there is greater than 25.0% difference between the concentration calculated from the two GC columns (see Form X) The lower of the two values is reported on Form I and flagged with a "P".
- X - Other specific flags may be required to properly define the results. If used, they must be fully described and such description attached to the Sample Data Summary Package and the SDG Narrative. Begin by using "X". If more than one flag is required, use "Y" and "Z", as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "A", "B", and "D" flags for some sample.

2.2 Non-target Compounds. Form I LCV-TIC and LCSV-TIC

Form I LCV-TIC and LCSV-TIC are used for reporting the tentative identification and estimated concentration for up to 10 of the non-surrogate and non-target organic compounds in the volatile fraction and up to 20 of the non-surrogate and non-target organic compounds in the semivolatile fraction.

Include a Form I LCV-TIC or LCSV-TIC for every volatile and semivolatile fraction of every sample, Performance Evaluation Sample, and blank analyzed. Form I LCV-TIC or LCSV-TIC must be provided for every analysis (except for the Laboratory Control Samples) that requires a Form I for target compounds, including required dilutions and reanalyses, even if no TICs are found.

Fill in all header information as section 2.1.

Report tentatively identified compounds (TICs) including CAS number, compound name, retention time, and the estimated concentration (criteria for reporting TICs are given in Exhibit D). Retention time must be reported in minutes and decimal minutes, not seconds or minutes and seconds.

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

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

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 description to no more than 28 characters (i.e., unknown hydrocarbon, etc.).

All TIC results, except "generics" (See N flag) are flagged "JN" in the "Q" column to emphasize the quantitative and qualitative uncertainties associated with these data. This includes "unknowns".

3. SURROGATE RECOVERY. FORM II LCV, LCSV, AND LCP

Form II is used to report the recovery of the surrogate compounds added to each sample, blank, Laboratory Control Sample, and Performance Evaluation Sample.

Complete the header information on each Form II required, according to the instructions in paragraph 1.

In the table, enter EPA Sample Numbers for each analysis as described in paragraph 1. For each sample, report the percent recovery for each surrogate to the nearest whole number.

Flag each surrogate recovery outside the QC limits with an asterisk (*). The asterisk must be placed in the last space in each appropriate column, under the "#" symbol. In the far right-hand column, total the number of surrogate recoveries outside the QC limits for each sample. If no surrogates were outside the limits, enter "0".

If a sample or extract is diluted and the surrogate recovery is below the recovery limits in any analysis, enter the calculated recovery or "0" (zero) if the surrogate is not detected. Flag the surrogate recovery with a "D" in the column under the "#" symbol. Do not include results flagged "D" in the total number of recoveries for each sample outside the QC limits.

Pesticide/Aroclor samples are analyzed on two GC columns, and surrogates recoveries must be reported for both analyses. Enter the information on the stationary phases and internal diameters of the two GC columns, as described in paragraph 1.10.4, differentiating the GC columns as "(1)" and "(2)". Enter the recoveries of the two surrogates for each column in a similar fashion.

Number the Form II pages as described in paragraph 1.7.

4. LABORATORY CONTROL SAMPLE RECOVERY. FORM III LCV, LCSV, AND LCP

Form III is used to report the recovery of the spiked analytes in the Laboratory Control Sample (LCS).

Complete the header information on each Form III required, according to the instructions in Sections 1 and 2.

The "LCS Lot No." is an identification number assigned by the Agency to the LCS spiking solution, if the solution is provided by the Agency. If the LCS solution is purchased by the Contractor from a third party, report the identification number used by the laboratory under "LCS Lot No."

The "LCS Aliquot" is the volume in microliters of LCS spiking solution that was added to reagent water before purging or extraction.

For pesticides, the LCS is reported for both GC columns. Enter the Instrument ID and GC Column - ID for analyses on 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.

In the upper box in Form III, under "AMOUNT ADDED", enter the amount in nanograms of each analyte added to the sample. Under "AMOUNT RECOVERED", enter the amount in nanograms 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". Enter the limits for each analyte in the column for "QC LIMITS". The limits should be entered as two whole numbers (lower and upper limits) separated by a hyphen. 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.

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

5. METHOD BLANK SUMMARY. FORM IV LCV, LCSV, AND LCP

Form IV lists the samples including LCS and PES associated with each method blank. A copy of the appropriate Form IV is required for each method blank.

Complete the header information on each Form IV required, according to the instructions in Sections 1.

For semivolatile and pesticide/Aroclor method blanks, enter the date of extraction of the blank.

For pesticide/Aroclors, enter the "Date Analyzed", "Time Analyzed", "Instrument ID", and "GC Column - ID" for analyses on both GC columns.

For all three fractions, as appropriate, summarize the samples, including LCS and PES associated with a given method blank in the table below the header, entering EPA Sample Number and Lab Sample ID. For volatiles, enter the Lab File ID and Time Analyzed for each sample. For semivolatiles, enter the Lab File ID and Date Analyzed. For pesticides/Aroclors, enter the Date Analyzed on each GC column for each sample.

For pesticides/Aroclors, enter "Y" or "N" (for yes or no) under "Sulfur Cleanup". If a separate sulfur cleanup blank is prepared, when not all

samples associated with a method blank are subjected to sulfur cleanup, then complete a separate Form IV for the sulfur cleanup blank, listed the EPA Sample No. of the blank, as described in paragraph 1.9.6, in the box in the upper right hand corner of the form. These samples associated with the sulfur cleanup blank will be listed in the lower portion of the form, as well as on a copy of Form IV for their associated method blank. Whenever all the samples and their associated method blank are subjected to sulfur cleanup, no separate sulfur blank is required, and only one Form IV needs to be completed.

Number the Form IV pages as described in paragraph 1.7.

6. GC/MS TUNING AND MASS CALIBRATION. FORM V LCV AND LCSV

This form is used to report the results of GC/MS tuning for volatiles and semivolatiles, and to summarize the date and time of analysis of samples, standards, and blanks associated with each GC/MS tune (including Laboratory Control and Performance Evaluation Samples).

Complete the header information on each Form V required, according to the instructions in paragraph 1.

Enter the "Lab File ID" for the injection containing the GC/MS tuning compound (BFB for volatiles, DFTPP for semivolatiles). Enter the date and time of injection of the tuning compound. Enter injection time as military time.

In the upper table, for each ion listed on the form, enter the % Relative Abundance in the right-hand column. Report relative abundances to the number of significant figures given for each ion in the ion abundance criteria column.

Note that 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 base peak (m/z 95 for BFB, and m/z 198 for DFTPP). Despite this possibility, all ion abundances are to be normalized to the nominal base peaks listed on Form V (see Exhibit D).

All relative abundances must be reported as a number. If zero, enter "0", 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.

In the lower half of the form, list all samples, standards, and blanks analyzed under that tune in chronological order, by time of analysis (in military time). Refer to paragraph 1 for specific instructions for identifying standards and blanks. Enter "EPA Sample No.", "Lab Sample ID", "Lab File ID", "Date Analyzed", and "Time Analyzed" for all standards, samples including LCS and PES, and blanks.

Number the Form V pages as described in paragraph 1.7.

7. INITIAL CALIBRATION SUMMARY. FORM VI LCV, LCSV-1, LCSV-2, LCP-1, LCP-2 AND LCP-3

For each fraction, after a GC/MS or GC system has undergone an initial calibration, and after all initial calibration technical criteria have been met, the laboratory must complete and submit all Form VIs for initial calibrations performed relevant to the samples including LCS and PES and blanks in the SDG, regardless of when that calibration was performed.

Complete the header information on each Form VI required, according to the instructions in paragraph 1.

Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID" and "Calibration Date(s)". If the calendar date changes during the calibration procedure, the inclusive dates should be given on Form VI.

For the volatile and semivolatile fractions, enter the "Lab File ID" for each of the five calibration standards injected. Complete the response factor data for the five calibration points. The relative response factor (RRF) is reported for each target compound and surrogate. The laboratory must report the average RRF and the percent relative standard deviation (%RSD) for the RRFs for each target compound and surrogate.

The initial calibration of pesticides and 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.

Complete header information on Form VI, LCP-1 and LCP-2 according to the instructions in paragraph 1. 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 performed on each GC column during an initial calibration, complete one copy of Form VI for each GC column used. Enter the Instrument ID, GC Column, and ID as described previously. Enter the dates of analysis of the first and last of the six standards on each form under "Date(s) Analyzed". Under "Level (x low)", 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" for "mid". If the concentration is not exactly 4.0 times the low point, enter the appropriate multiplier in a similar format. The high point standard must 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.

For the pesticides/Aroclors fraction, one Form VI (LCP-1 and LCP-2) is required for each initial calibration performed on each GC column. In the table, on Form VI LCP-1, enter the retention time of each analyte in the low, mid, and high point Standard Mixtures A and B in the columns labeled "RT of Standards". Use the values from Standard Mixture A for the surrogates. Calculate and report in the appropriate column the mean retention time and the retention time windows for each analyte. Report the retention time window for each analyte as a range of two values, i.e., from 1.44 to 1.54. Enter the lower value of the range in the column under "RT WINDOW" labeled "FROM". Enter the upper value of the range in the column under "TO". Do not separate the two values with a hyphen, and do not enter the retention time window as a plus/minus value such as ± 0.05 . NOTE: By definition, the center of the retention time window must be the mean retention time listed to the left of the retention time window.

On Form VI LCP-2, calculate the calibration factor for each analyte in the low, mid, and high point Standard Mixtures A and B. Use the values from Standard Mixture A for the surrogates. Report the values under the columns labeled "CALIBRATION FACTORS". Calculate the mean of the three calibration factors and the percent relative standard deviation (%RSD) for the calibration factor values for each analyte. Report the calculated values under the "MEAN" column the "%RSD" columns, respectively.

On Form VI LCP-3, for the initial calibration of multicomponent analytes, enter the amount of standard injected in nanograms of each analyte, under the "AMOUNT" column. The number of peaks with an asterisk under the "Peak" column indicates the minimum number of peaks calibrated for each analyte. Enter the retention time of each peak used to quantitate under the "RT" column. Data for two additional peaks may be reported for each multicomponent analyte. Calculate and report the calibration factor for each peak used under "CALIBRATION FACTOR".

8. PESTICIDE RESOLUTION CHECK SUMMARY. FORM VI LCP-4

Pesticide Resolution Check Summary Form VI is used to report the resolution of each analyte in the Resolution Check Mixture analyzed at the beginning of each initial calibration on each GC column.

Complete the header information on each Form VI required according to the instructions in paragraph 1.

For each GC column, enter the "EPA Sample Number" of the Resolution Check Mixture, as described in paragraph 1.9.7, for the mixture injected on the first GC column. Enter the Lab Sample ID, Date Analyzed (1), and Time Analyzed (1).

In the table, under "ANALYTE", enter the name of each analyte as it appears on Form I, in elution order, starting with the first target analyte or surrogate to elute. Enter the retention time of each of the analytes listed under "RT".

Calculate the percent resolution between each pair of consecutive peaks according to Exhibit D. Enter the percent resolution of each pair in the "RESOLUTION" field of the analyte that elutes earlier (the analyte listed first). The resolution must be calculated for each adjacent peaks so that the resolution of peak 1 and peak 2 is calculated, as well as peak 2 vs. peak 3, peak 3 vs. peak 4, etc. The "RESOLUTION" field will be left blank for the last analyte in the in the table. The percent resolution must meet the QC limits listed at the bottom of the page.

Complete the information for the second GC column in the same fashion.

9. CONTINUING CALIBRATION SUMMARY. FORM VII LCV, LCSV-1 AND LCSV-2

The Continuing Calibration Summary Form VII is used to verify 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 analysis.

Complete the header information on each Form VII required, according to the instructions in paragraph 1.

Enter date and time of continuing calibration standard analysis, the Lab File ID of the continuing calibration standard, and date(s) of initial calibration. Give inclusive dates if initial calibration is performed over more than one date. Enter the average relative response factor (RRF) for each target compound that was calculated from the initial calibration data (referred to in the initial calibration date(s) analyzed field). Report the relative response factor for each target compound and surrogate from the continuing calibration standard analysis.

10. PESTICIDE/AROCLOL CALIBRATION VERIFICATION

Calibration Verification Summary. Form VII LCP-1 and LCP-2

The Calibration Verification Summary Form VII is used to report the results of the Performance Evaluation Mixtures (PEM), instrument blanks, and Individual Standard Mixtures A and B analyzed at the beginning and end of a twelve hour sequence. The laboratory must submit this form for each twelve hour sequence analyzed.

Complete the header information on each Form VII required according to the instructions in paragraph 1.

Enter the initial calibration date(s) analyzed. Give inclusive dates if initial calibration is performed over more than one date.

On Form VII, LCP-1, enter the EPA Sample No., Lab Sample ID, Date Analyzed, and Time Analyzed for the instrument blank that preceded the twelve hour sequence (PIBLK). For the PEM that initiated or terminated the twelve hour sequence (PEM), enter the EPA Sample No., Lab Sample ID, Date Analyzed, and Time Analyzed.

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

In the table, report the retention time for each analyte in the PEM as well as the retention time windows. For each analyte in the PEM, enter the amount of the analyte in nanograms, to three decimal places, calculated to be in the PEM, under "CALC AMOUNT". Enter the nominal amount of each analyte in the PEM under "NOM AMOUNT". Calculate the relative percent difference between the calculated amount and nominal amount for each analyte according to Exhibit D. Report the values under "%D".

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 is used to report the dates and times of analysis of the instrument blanks and the results of the analyses of the midpoint concentrations of Individual Standard Mixtures A and B that, along with the PEM, bracket each 12-hour period of sample analyses. One copy of Form VII LCP-2 must be completed each time the Individual Standard Mixtures are analyzed, for each GC column used. The form is completed in a similar fashion to Form VII LCP-1, entering the EPA Sample No., Lab Sample ID, Date Analyzed, and Time Analyzed for the instrument blank immediately preceding the Individual Standard Mixtures A and B, and for the standards themselves. The upper table on the form contains the retention time and amount data for Individual Standard Mixture A compounds. The lower table contains the data for Mixture B. Enter the data in these tables in a fashion similar to that for the PEM. Complete copies of Form VII LCP-1 and 2 for each standard reported in Form VIII LCP.

11. INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY FORM VIII LCV, LCSV-1 AND LCSV-2

Form VIII is used to summarize the peak areas and retention times of the internal standards added to all volatile and semivolatile samples and blanks. Form VIII is also used to check the internal standards in the initial calibration sequences. The data are used to determine when changes in internal standard responses will adversely affect quantitation of target compounds. This form must be completed each time an initial calibration or a continuing calibration is performed, or when samples are analyzed under the same GC/MS tune as an initial calibration.

Complete the header information on each Form VIII required, according to the instructions in paragraph 1.

Enter the Lab File ID, Date Analyzed, and Time Analyzed for the continuing calibration standard. If samples are analyzed immediately following an initial calibration, before another GC/MS tune and a continuing calibration, Form VIII shall be completed for the initial calibration standard that is the same concentration as the continuing calibration standard. Enter the Lab File ID, the date and time of analysis, the areas and retention times of this initial calibration standard in place of those of a continuing calibration standard.

From the results of the analysis of the continuing calibration standard, enter the area measured for each internal standard and its retention time under the appropriate column in the row labeled "12 HOUR STD". For each volatile internal standard, calculate the area upper limit as the area of the particular internal standard plus 40 percent of its area, and the area lower limit as the area of the internal standard minus 40 percent of its area. For each semivolatile internal standard, calculate the area upper limit as the area of the particular standard plus 100% of its area (i.e., two times the area in the 12 HOUR STD box), and the area lower limit as the area of the internal standard minus 50% of its area (i.e., one half the area in the 12 HOUR STD box). Report these values in the boxes labeled "UPPER LIMIT" and "LOWER LIMIT" respectively.

For each volatile and semivolatile internal standard, calculate the retention time (RT) upper limit as the RT of the particular internal standard plus 0.33 minutes. The lower limit is the RT of the internal standard minus 0.33 minutes. Report these values in the boxes labeled "UPPER LIMIT" and "LOWER LIMIT" respectively.

For each sample including LCS and PES and blank 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 or retention time is outside the upper or lower limits calculated above, flag that value with an asterisk (*). The asterisk must be placed in the far right hand space of the box for each internal standard area or retention time, directly under the "#" symbol.

If samples are analyzed immediately following an initial calibration as described above, enter the EPA Sample Number, internal standard areas, and retention times for all five of the initial calibration standards.

Number the Form VIII pages as described in paragraph 1.7.

12. PESTICIDE/AROCLOL ANALYTICAL SEQUENCE. FORM VIII LCP

Form VIII LCP is required for each analytical sequence for each GC system and for each GC column used to analyze pesticide/Aroclors in an SDG.

Complete the header information on each Form VIII required, according to the instructions in paragraph 1.

Enter the initial calibration date(s). Give inclusive dates if initial calibration is performed over more than one date.

At the top of the table, report the mean retention time for surrogates tetrachloro-m-xylene and decachlorobiphenyl calculated from the initial calibration sequence under "TCX" and "DCB", respectively. For every analysis associated with a particular analytical sequence starting with the initial calibration, enter the EPA Sample Number, Lab Sample ID, Date Analyzed, and Time Analyzed. Each sample analyzed as part of the sequence must be reported on Form VIII LCP even if it is not associated with the SDG. The laboratory may use the EPA Sample No. of "ZZZZZ" to distinguish all samples that are not part of the SDG being reported. Report the retention time of the surrogates for each analysis under "TCX RT" and "DCB RT". All sample analyses must 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 (see Exhibit D), it is not necessary to report the data from 12-hour periods when no samples in an SDG were run. The laboratory must deliver the 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 is not a routine deliverable, it must be made available on request during on-site evaluations, etc. Here again, non-EPA samples may be indicated with "ZZZZZ".

Flag all those values which do not meet the contract requirements by entering an asterisk (*) in the last column, under the "*". 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 (i.e., under "DCB") column, and document the problem in the SDG Narrative.

Number the Form VIII pages as described in paragraph 1.7.

13. PESTICIDE/AROCLOR FLORISIL CARTRIDGE CHECK. FORM IX LCP

Form IX is required for each lot of Florisil cartridges that is used with samples associated with the SDG.

Complete the header information on each Form IX required, according to the instructions in paragraph 1.

Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the cartridge check was performed. Enter the "Florisil Cartridge Lot Number". Enter under the "Date Analyzed", the date the Florisil cartridge check solution was analyzed.

In the upper table, enter the amount of spike added and spike recovered in nanograms for each analyte.

Calculate to the nearest whole percent, and enter the percent recovery in the "% REC" field. Flag each spike recovery outside the QC limits

with an asterisk (*). The asterisk must be placed in the last space in the "% Rec" column, under the "*" symbol.

In the lower table, enter the "EPA Sample No.", the "Lab Sample ID", and "Date Analyzed" for each sample and blank that was cleaned up using this lot of Florisil cartridges.

Number the Form IX pages as described in paragraph 1.7.

14. PESTICIDE/AROCLOR IDENTIFICATION. FORM X LCP-1, LCP-2

Form X summarizes the data used to identify and quantify all pesticide/Aroclor target analytes detected in a given sample. Form X LCP-1 is required for each sample (including PES and LCS) or blank in which any single component analytes is detected. Form X LCP-2 is required for each sample (including PES and LCS) or blank in which any multicomponent analyte is detected. If no single component analyte or multicomponent analyte is detected in a sample, no copy of the applicable Form X is required for that sample.

Complete the header information on each Form X required, according to the instructions in paragraph 1.

For each target pesticide or Aroclor detected, enter the name of the analyte on Form X in the column labeled "Analyte", spelling the name as it appears on Form I. For the multicomponent analytes, there are spaces (fields) for up to 5 peaks for each analyte. The asterisks indicate the number of peaks that are required, and data for additional peaks may be reported. The retention time, retention time window, and concentration are calculated separately for each peak used for a multicomponent analyte. For each GC column, enter the retention times of the analytes detected in the sample next to the appropriate column designation (1 or 2). Enter the retention time windows on each column of the appropriate standard. The lower value is entered under the "FROM" column, the upper value under the "TO" column. Do not use a hyphen. These data must correspond with those on Form VI, and are entered in a similar manner. Calculate the concentration of the analyte using the calibration factors derived from the initial calibration sequence. For the multicomponent analytes, calculate and report the mean concentration by averaging the concentration values from the peaks used for quantitation. Calculate and report the percent difference to a tenth of a percent between the concentration values (or mean concentration values for multicomponent analytes) on the two GC columns under "%D" as described in Exhibit D.

Number the Form X pages as described in paragraph 1.7.

THE FOLLOWING ARE DOCUMENT CONTROL FORMS

(To be submitted as hardcopy only)

15. SAMPLE LOG-IN SHEET (FORM DC-1)

This form is used to document the receipt and inspection of samples and containers. One original of Form DC-1 is required for each sample

shipping container. If the samples in a single sample shipping container (e.g., coolers) must be assigned to more than one Sample Delivery Group, the original Form DC-1 shall be placed with the deliverables for the Sample Delivery Group with the lowest sample number and a copy of Form DC-1 must be placed with the deliverables for the other Sample Delivery Group(s). The copies should be identified as "copy(ies)," and the location of the original should be noted on the copies.

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

Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (i.e., Traffic Reports, Packing Lists), and airbills or airbill stickers in items 3-5 on Form DC-1. Specify if there is an airbill present or an airbill sticker in item 5 on Form DC-1. Record the airbill or sticker number in item 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 (i.e., intact, broken, leaking) and presence or absence of sample tags in items 7 and 8 on Form DC-1.

Review the sample shipping documents and complete the header information described in Part A. Compare the information recorded on all the documents and samples and mark the appropriate answer in item 9 on Form DC-1.

If there are no problems observed during receipt, sign and date (include time) Form DC-1, the chain-of-custody record, and Traffic Report, and write the sample numbers on Form DC-1. Record the appropriate sample tags and assigned laboratory numbers if applicable. The log-in date should be recorded at the top of Form DC-1 and the date and time of cooler receipt at the laboratory should be recorded in items 10 and 11. Cross out unused columns and spaces.

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

Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of Form I. Sign and date the Sample Transfer block.

16. DOCUMENT INVENTORY SHEET (FORM DC-2)

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

Organize all complete SDG file documents as described in Exhibit B, Section II, paragraph 5. Assemble the documents in the order specified on Form DC-2, and stamp each page with a consecutive number. (Do not number the DC-2 form). Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided in the Form DC-2. If there are no documents for a specific document type, enter an "NA" in the empty space.

Certain laboratory specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review DC-2 to determine if it is most appropriate to place them under No. 7, 8, 9, or 10. Category 10 should be used only if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

SECTION IV

DATA REPORTING FORMS

1LCA
LOW CONC. WATER VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

b Name: _____ Contract: _____

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

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Analyzed: _____

Purge Volume: _____ (mL) Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION (ug/L)	Q
74-87-3-----	Chloromethane		
74-83-9-----	Bromomethane		
75-01-4-----	Vinyl chloride		
75-00-3-----	Chloroethane		
75-09-2-----	Methylene chloride		
67-64-1-----	Acetone		
75-15-0-----	Carbon disulfide		
75-35-4-----	1,1-Dichloroethene		
75-34-3-----	1,1-Dichloroethane		
156-59-4-----	cis-1,2-Dichloroethene		
156-60-5-----	trans-1,2-Dichloroethene		
67-66-3-----	Chloroform		
107-06-2-----	1,2-Dichloroethane		
78-93-3-----	2-Butanone		
74-97-5-----	Bromochloromethane		
71-55-6-----	1,1,1-Trichloroethane		
56-23-5-----	Carbon tetrachloride		
75-27-4-----	Bromodichloromethane		
78-87-5-----	1,2-Dichloropropane		
10061-01-5-----	cis-1,3-Dichloropropene		
79-01-6-----	Trichloroethene		
124-48-1-----	Dibromochloromethane		
79-00-5-----	1,1,2-Trichloroethane		
71-43-2-----	Benzene		
10061-02-6-----	trans-1,3-Dichloropropene		
75-25-2-----	Bromoform		
108-10-1-----	4-Methyl-2-pentanone		
591-78-6-----	2-Hexanone		
127-18-4-----	Tetrachloroethene		
79-34-5-----	1,1,2,2-Tetrachloroethane		
106-93-4-----	1,2-Dibromoethane		
108-88-3-----	Toluene		
108-90-7-----	Chlorobenzene		
100-41-4-----	Ethylbenzene		
100-42-5-----	Styrene		
1330-20-7-----	Xylenes (total)		
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		

1LCB
LOW CONC. WATER SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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) pH: _____

CAS NO.	COMPOUND	CONCENTRATION (ug/L)	Q
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)		
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		
120-82-1-----	1,2,4-Trichlorobenzene		
91-20-3-----	Naphthalene		
106-47-8-----	4-Chloroaniline		
87-68-3-----	Hexachlorobutadiene		
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		
91-58-7-----	2-Chloronaphthalene		
88-74-4-----	2-Nitroaniline		
131-11-3-----	Dimethylphthalate		
208-96-8-----	Acenaphthylene		
606-20-2-----	2,6-Dinitrotoluene		
99-09-2-----	3-Nitroaniline		
83-32-9-----	Acenaphthene		

1LCC
LOW CONC. WATER SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

b Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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) pH: _____

CAS NO.	COMPOUND	CONCENTRATION (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		
7005-72-3-----	4-Chlorophenyl-phenylether		
86-73-7-----	Fluorene		
100-01-6-----	4-Nitroaniline		
534-52-1-----	4,6-Dinitro-2-methylphenol		
86-30-6-----	N-Nitrosodiphenylamine (1)		
101-55-3-----	4-Bromophenyl-phenylether		
118-74-1-----	Hexachlorobenzene		
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-----	Dibenz(a,h) anthracene		
191-24-2-----	Benzo(g,h,i) perylene		

(1) - Cannot be separated from Diphenylamine

1LCD
LOW CONC. WATER PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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) _____ pH: _____

CAS NO.	COMPOUND	CONCENTRATION (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		
7421-36-3-----	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		

1LCE
 LOW CONC. WATER VOLATILE ORGANICS ANALYSIS DATA SHEET
 TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Name: _____ Contract: _____

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

Lab Sample ID: _____ Date Received: _____

Lab File ID: _____ Date Analyzed: _____

Purge Volume: _____ (mL) Dilution Factor: _____

Number TICs found: _____

CAS NUMBER	COMPOUND NAME	RT	EST. CONC. (ug/L)	Q
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

1LCF
 LOW CONC. WATER SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
 TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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) pH: _____

Number TICs found: _____

CAS NUMBER	COMPOUND NAME	RT	EST. CONC. (ug/L)	Q
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

2LCA
LOW CONC. WATER VOLATILE SURROGATE RECOVERY

Name: _____ Contract: _____

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

	EPA SAMPLE NO.	BFB %REC #	OTHER	TOT OUT
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				

QC LIMITS

%REC

BFB = Bromofluorobenzene (80-120)

- # Column to be used to flag recovery values.
- * Values outside of contract required QC limits.
- D Surrogate diluted out.

2LCB
LOW CONC. WATER SEMIVOLATILE SURROGATE RECOVERY

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

	EPA SAMPLE NO.	NBZ %REC #	FBP %REC #	TPH %REC #	PHL %REC #	2FP %REC #	TBP %REC #	OTHER	TOT OUT
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									

QC LIMITS
%REC

NBZ = Nitrobenzene-d5 (40-112)
FBP = 2-Fluorobiphenyl (42-110)
TPH = Terphenyl-d14 (24-140)
PHL = Phenol-d5 (17-113)
2FP = 2-Fluorophenol (16-108)
TBP = 2,4,6-Tribromophenol (18-126)

Column to be used to flag recovery values.
* Values outside of contract required QC limits.
D Surrogate diluted out.

2LCC
LOW CONC. WATER PESTICIDE SURROGATE RECOVERY

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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								
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								

QC LIMITS

%REC

TCX = Tetrachloro-m-xylene (60-150)

DCB = Decachlorobiphenyl (60-150)

Column to be used to flag recovery values.

* Values outside of contract required QC limits.

D Surrogate diluted out.

3LCA
LOW CONC. WATER VOLATILE LAB CONTROL SAMPLE RECOVERY

EPA SAMPLE NO.

Name: _____ Contract: _____

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

Lab Sample ID: _____ LCS Lot No.: _____

Lab File ID: _____ Date Analyzed: _____

Purge Volume: _____ (mL) Dilution Factor: _____

LCS Aliquot: _____ (uL)

COMPOUND	AMOUNT ADDED (ng)	AMOUNT RECOVERED (ng)	%REC #	QC LIMITS
Vinyl chloride				
1,2-Dichloroethane				
Carbon tetrachloride				
1,2-Dichloropropane				
Trichloroethene				
1,1,2-Trichloroethane				
Benzene				
cis-1,3-Dichloropropene				
Bromoform				
Tetrachloroethene				
1,2-Dibromoethane				
1,4-Dichlorobenzene				

Column to be used to flag LCS recovery with an asterisk.

* Values outside of QC limits.

LCS Recovery: _____ outside limits out of _____ total.

COMMENTS: _____

3LCB
LOW CONC. WATER SEMIVOLATILE LAB CONTROL SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

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

Lab Sample ID: _____ LCS Lot No.: _____

Lab File ID: _____ Date Extracted: _____

LCS Aliquot: _____ (uL) Date Analyzed: _____

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

Injection Volume: _____ (uL) pH: _____

COMPOUND	AMOUNT ADDED (ng)	AMOUNT RECOVERED (ng)	%REC #	QC LIMITS
Phenol				
bis(2-Chloroethyl) ether				
2-Chlorophenol				
N-Nitroso-di-n-propylamine				
Hexachloroethane				
Isophorone				
1,2,4-Trichlorobenzene				
Naphthalene				
4-Chloroaniline				
2,4,6-Trichlorophenol				
2,4-Dinitrotoluene				
Diethylphthalate				
N-Nitrosodiphenylamine				
Hexachlorobenzene				
Benzo(a)pyrene				

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

LCS Recovery: _____ outside limits out of _____ total.

COMMENTS: _____

3LCC
LOW CONC. WATER PESTICIDE LAB CONTROL SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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) _____ pH: _____

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

COMPOUND	AMOUNT ADDED (ng)	AMOUNT RECOVERED (ng)	%REC #	QC LIMITS
gamma-BHC (Lindane) _____	_____	_____	_____	_____
Heptachlor epoxide _____	_____	_____	_____	_____
Dieldrin _____	_____	_____	_____	_____
4,4'-DDE _____	_____	_____	_____	_____
Endrin _____	_____	_____	_____	_____
Endosulfan sulfate _____	_____	_____	_____	_____
gamma-Chlordane _____	_____	_____	_____	_____

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

COMPOUND	AMOUNT ADDED (ng)	AMOUNT RECOVERED (ng)	%REC #	QC LIMITS
gamma-BHC (Lindane) _____	_____	_____	_____	_____
Heptachlor epoxide _____	_____	_____	_____	_____
Dieldrin _____	_____	_____	_____	_____
4,4'-DDE _____	_____	_____	_____	_____
Endrin _____	_____	_____	_____	_____
Endosulfan sulfate _____	_____	_____	_____	_____
gamma-Chlordane _____	_____	_____	_____	_____

Column to be used to flag recovery values with an asterisk.
 * Values outside of QC limits.
 %CS Recovery: _____ outside limits out of _____ total.

COMMENTS: _____

4LCA
LOW CONC. WATER VOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Name: _____ Contract: _____

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

Lab Sample ID: _____ Date Analyzed: _____

Lab File ID: _____ Time Analyzed: _____

Instrument ID: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND LCS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
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				

COMMENTS: _____

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4LCB
LOW CONC. WATER SEMIVOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

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

Lab Sample ID: _____ Date Extracted: _____

Lab File ID: _____ Date Analyzed: _____

Instrument ID: _____ Time Analyzed: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND LCS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
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				

COMMENTS: _____

page __ of __

4LCC
LOW CONC. WATER PESTICIDE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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) _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND LCS:

	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				
24				
25				
26				

COMMENTS: _____

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5LCA
LOW CONC. WATER VOLATILE ORGANIC GC/MS TUNING AND MASS
CALIBRATION - BROMOFLUOROBENZENE (BFB)

Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
Lab File ID: _____ BFB Injection Date: _____
Instrument ID: _____ BFB Injection Time: _____
GC Column: _____ ID: _____ (mm)

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 TUNE APPLIES TO THE FOLLOWING SAMPLES, LCS, 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					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

page __ of __

5LCB
 LOW CONC. WATER SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS
 CALIBRATION - DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS 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 TUNE APPLIES TO THE FOLLOWING SAMPLES, LCS, 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					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

6LCA

LOW CONC. WATER VOLATILE ORGANICS INITIAL CALIBRATION SUMMARY

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Calibration Date(s): _____

LAB FILE ID: _____	RRF1 = _____	RRF2 = _____	RRF5 = _____	RRF10 = _____	RRF25 = _____		
RRF5 = _____	RRF10 = _____	RRF25 = _____					
COMPOUND	RRF1	RRF2	RRF5	RRF10	RRF25	RRF	% RSD
Chloromethane							
Bromomethane	*						
Vinyl chloride	*						
Chloroethane							
Methylene chloride							
Acetone							
Carbon disulfide							
1,1-Dichloroethene	*						
1,1-Dichloroethane	*						
cis-1,2-Dichloroethene							
trans-1,2-Dichloroethene							
Chloroform	*						
1,2-Dichloroethane	*						
2-Butanone							
Bromochloromethane	*						
1,1-Trichloroethane	*						
Carbon tetrachloride	*						
Bromodichloromethane	*						
1,2-Dichloropropane	*						
cis-1,3-Dichloropropene	*						
Trichloroethene	*						
Dibromochloromethane	*						
1,1,2-Trichloroethane	*						
Benzene	*						
trans-1,3-Dichloropropene	*						
Bromoform	*						
4-Methyl-2-pentanone							
2-Hexanone							
Tetrachloroethene	*						
1,1,2,2-Tetrachloroethane	*						
1,2-Dibromoethane	*						
Toluene	*						
Chlorobenzene	*						
Ethylbenzene	*						
Styrene	*						
Xylenes (total)	*						
1,3-Dichlorobenzene	*						
1,4-Dichlorobenzene	*						
1,2-Dichlorobenzene	*						
1,2-Dibromo-3-chloropropane							
Bromofluorobenzene	*						

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

6LCB
LOW CONC. WATER SEMIVOLATILE ORGANICS INITIAL CALIBRATION SUMMARY

Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
Instrument ID: _____ Calibration Date(s): _____
Calibration Times: _____

LAB FILE ID: _____ RRF5 = _____ RRF10 = _____
RRF20 = _____ RRF50 = _____ RRF80 = _____

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

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

6LCC
LOW CONC. WATER SEMIVOLATILE ORGANICS INITIAL CALIBRATION SUMMARY

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Instrument ID: _____ Calibration Date(s): _____
 Calibration Times: _____

LAB FILE ID: _____		RRF5 = _____	RRF10 = _____
RRF20= _____		RRF50= _____	RRF80= _____

COMPOUND	RRF5	RRF10	RRF20	RRF50	RRF80	RRF	% RSD
Diethylphthalate							
4-Chlorophenyl-phenylether *							
Fluorene *							
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine (1)							
4-Bromophenyl-phenylether *							
Hexachlorobenzene *							
Pentachlorophenol *							
Phenanthrene *							
Anthracene *							
-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 *							
Dibenz(a,h)anthracene *							
Benzo(g,h,i)perylene *							

Nitrobenzene-d5							
2-Fluorobiphenyl *							
Terphenyl-d14 *							
Phenol-d5 *							
2-Fluorophenol *							
2,4,6-Tribromophenol							

(1) Cannot be separated from Diphenylamine

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

11 other compounds must meet a minimum RRF of 0.010.

6LCD
LOW CONC. WATER PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS 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 Ind. Mix A analyses.

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

6LCE
LOW CONC. WATER PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Instrument ID: _____ Level (x low): low _____ mid _____ high _____
 GC Column: _____ ID: _____ (mm) Date(s) Analyzed: _____

COMPOUND	CALIBRATION FACTORS			MEAN	%RSD
	LOW	MID	HIGH		
alpha-BHC					
beta-BHC					
delta-BHC					
gamma-BHC (Lindane)					
Heptachlor					
Aldrin					
Heptachlor epoxide					
Endosulfan I					
Dieldrin					
4,4'-DDE					
Endrin					
Endosulfan II					
,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 Ind. Mix A analyses.

%RSD must be less than or equal to 20.0% for all compounds, except the surrogates, where %RSD must be less than or equal to 30.0%. Up to two target compounds, but not surrogates, may have %RSD greater than 20.0%, but less than or equal to 30.0%.

6LCF
LOW CONC. WATER PESTICIDE INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Instrument ID: _____ Date(s) Analyzed: _____
 GC Column: _____ ID: _____ (mm)

COMPOUND	AMOUNT (ng)	PEAK	RT	RT WINDOW		CALIBRATION FACTOR
				FROM	TO	
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				

* Denotes required peaks

6LCG
LOW CONC. WATER PESTICIDE RESOLUTION CHECK SUMMARY

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

GC Column (1): _____ ID: _____ (mm) Instrument ID (1): _____
EPA Sample No. (Standard 1): _____ 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. (Standard 2): _____ Lab Sample ID (2): _____
Date Analyzed (2): _____ Time Analyzed (2): _____

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

Resolution of two adjacent peaks must be calculated as a percentage of the height of the smaller peak, and must be greater than or equal to 60.0%.

7LCA

LOW CONC. WATER VOLATILE ORGANICS CONTINUING CALIBRATION SUMMARY

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Calibration Date: _____ Time: _____

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

COMPOUND	RRF	RRF5	MIN RRF	%D	MAX %D
Chloromethane					
Bromomethane			0.100		30.0
Vinyl chloride			0.100		30.0
Chloroethane					
Methylene chloride					
Acetone					
Carbon disulfide					
1,1-Dichloroethene			0.100		30.0
1,1-Dichloroethane			0.200		30.0
cis-1,2-Dichloroethene			0.100		
trans-1,2-Dichloroethene					
Chloroform			0.200		30.0
1,2-Dichloroethane			0.100		30.0
2-Butanone					
Bromochloromethane			0.050		30.0
1,1,1-Trichloroethane			0.100		30.0
Carbon tetrachloride			0.100		30.0
Bromodichloromethane			0.200		30.0
1,2-Dichloropropane					
cis-1,3-Dichloropropene			0.200		30.0
Trichloroethene			0.300		30.0
Dibromochloromethane			0.100		30.0
1,1,2-Trichloroethane			0.100		30.0
Benzene			0.500		30.0
trans-1,3-Dichloropropene			0.100		30.0
Bromoform			0.050		30.0
4-Methyl-2-pentanone					
2-Hexanone					
Tetrachloroethene			0.200		30.0
1,1,2,2-Tetrachloroethane			0.100		30.0
1,2-Dibromoethane			0.100		30.0
Toluene			0.400		30.0
Chlorobenzene			0.500		30.0
Ethylbenzene			0.100		30.0
Styrene			0.300		30.0
Xylenes (total)			0.300		30.0
1,3-Dichlorobenzene			0.600		30.0
1,4-Dichlorobenzene			0.500		30.0
1,2-Dichlorobenzene			0.400		30.0
1,2-Dibromo-3-chloropropane					
Bromofluorobenzene			0.200		30.0

All other compounds must meet a minimum RRF of 0.010.

7LCB
LOW CONC. WATER SEMIVOLATILE ORGANICS CONTINUING CALIBRATION SUMMARY

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Instrument ID: _____ Calibration Date: _____ Time: _____
 Lab File ID: _____ Init. Calib. Date(s): _____
 Init. Calib. Times: _____

COMPOUND	RRF	RRF20	MIN RRF	%D	MAX %D
Phenol			0.800		25.0
bis(2-Chloroethyl) ether			0.700		25.0
2-Chlorophenol			0.700		25.0
2-Methylphenol			0.700		25.0
2,2'-oxybis(1-Chloropropane)					
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		25.0
2,4-Dimethylphenol			0.200		25.0
bis(2-Chloroethoxy) methane			0.300		25.0
2,4-Dichlorophenol			0.200		25.0
1,2,4-Trichlorobenzene			0.200		25.0
Naphthalene			0.700		25.0
4-Chloroaniline					
Hexachlorobutadiene					
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
2-Chloronaphthalene			0.800		25.0
2-Nitroaniline					
Dimethylphthalate					
Acenaphthylene			1.300		25.0
2,6-Dinitrotoluene			0.200		25.0
3-Nitroaniline					
Acenaphthene			0.800		25.0
2,4-Dinitrophenol					
4-Nitrophenol					
Dibenzofuran			0.800		25.0
2,4-Dinitrotoluene			0.200		25.0

All other compounds must meet a minimum RRF of 0.010.

7LCC
LOW CONC. WATER SEMIVOLATILE ORGANICS CONTINUING CALIBRATION SUMMARY

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Instrument ID: _____ Calibration Date: _____ Time: _____
 Lab File ID: _____ Init. Calib. Date(s): _____
 Init. Calib. Times: _____

COMPOUND	RRF	RRF20	MIN RRF	%D	MAX %D
Diethylphthalate					
4-Chlorophenyl-phenylether			0.400		25.0
Fluorene			0.900		25.0
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine (1)					
4-Bromophenyl-phenylether			0.100		25.0
Hexachlorobenzene			0.100		25.0
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
Dibenz(a,h)anthracene			0.400		25.0
Benzo(g,h,i)perylene			0.500		25.0
Nitrobenzene-d5			0.010		25.0
2-Fluorobiphenyl			0.700		25.0
Terphenyl-d14			0.500		25.0
Phenol-d5			0.800		25.0
Fluorophenol			0.600		25.0
2,4,6-Tribromophenol					

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

7LCD
LOW CONC. WATER PESTICIDE CALIBRATION VERIFICATION SUMMARY

Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ SAS 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		CALC AMOUNT (ng)	NOM AMOUNT (ng)	%D
		FROM	TO			
alpha-BHC						
beta-BHC						
gamma-BHC (Lindane)						
Endrin						
4,4'-DDT						
Methoxychlor						

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

QC LIMITS:

Absolute values of %D of amounts in PEM must be less than or equal to 25.0%

4,4'-DDT breakdown must be less than or equal to 20.0%

Endrin breakdown must be less than or equal to 20.0%

Combined breakdown must be less than or equal to 30.0%

7LCE
LOW CONC. WATER PESTICIDE CALIBRATION VERIFICATION SUMMARY

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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. (INDA): _____ 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. (INDB): _____ 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						
Enrin aldehyde						
alpha-Chlordane						
gamma-Chlorodane						
Tetrachloro-m-xylene						
Decachlorobiphenyl						

QC LIMITS: Absolute value of %D of amounts in the Individual Mixes must be less than or equal to 25.0%.

8LCA
LOW CONC. WATER VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Lab File ID (Standard): _____ Date Analyzed: _____
 Instrument ID: _____ Time Analyzed: _____
 GC Column: _____ ID: _____ (mm)

	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 internal standard area and RT values with an asteris
 * Values outside of QC limits.

8LCB
LOW CONC. WATER SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Lab File ID (Standard): _____ Date Analyzed: _____
 Instrument ID: _____ Time Analyzed: _____

	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 internal standard area and RT values with an asterisk
 * Values outside of QC limits.

8LCC
LOW CONC. WATER SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Lab File ID (Standard): _____ Date Analyzed: _____
 Instrument ID: _____ Time Analyzed: _____

	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						
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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 internal standard area and RT values with an asterisk
 * Values outside of QC limits.

8LCD
LOW CONC. WATER PESTICIDE ANALYTICAL SEQUENCE

Lab Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 GC Column: _____ ID: _____ (mm) Init. Calib. Date(s): _____
 Instrument ID: _____

THE ANALYTICAL SEQUENCE OF PERFORMANCE EVALUATION MIXTURES, BLANKS,
 SAMPLES, 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					
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18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					
32					

QC LIMITS
 TCX = Tetrachloro-m-xylene (\pm 0.05 MINUTES)
 DCB = Decachlorobiphenyl (\pm 0.10 MINUTES)

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

9LCA
LOW CONC. WATER PESTICIDE FLORISIL CARTRIDGE CHECK

Lab Name: _____ Contract: _____
 Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
 Florisil Cartridge Lot Number: _____ Date Analyzed: _____
 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				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

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

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

	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 CONC. WATER PESTICIDE IDENTIFICATION SUMMARY
FOR SINGLE COMPONENT ANALYTES

EPA SAMPLE NO. _____

Name: _____ Contract: _____

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

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

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

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

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

10LCB
LOW CONC. WATER PESTICIDE IDENTIFICATION SUMMARY
FOR MULTICOMPONENT ANALYTES

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS 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		CONCENTRATION	MEAN CONCENTRATION	%D
			FROM	TO			
COLUMN 1	*1						
	*2						
	*3						
	4						
	5						
COLUMN 2	*1						
	*2						
	*3						
	4						
	5						
COLUMN 1	*1						
	*2						
	*3						
	4						
	5						
COLUMN 2	*1						
	*2						
	*3						
	4						
	5						
COLUMN 1	*1						
	*2						
	*3						
	4						
	5						
COLUMN 2	*1						
	*2						
	*3						
	4						
	5						

: least 3 peaks are required for identification of multicomponent analytes

SAMPLE LOG-IN SHEET				
Lab Name: _____		Page ____ of ____		
Received By (Print Name): _____		Log-in Date: _____		
Received By (Signature): _____				
Case Number: _____				
Sample Delivery Group No.: _____				
SAS Number: _____				
REMARKS:				
1. Custody Seal(s) Present/Absent* Intact/Broken				
2. Custody Seal Nos.: _____				
3. Chain-of-Custody Records Present/Absent*				
4. Traffic Reports or Packing List Present/Absent*				
5. Airbill Airbill/Sticker Present/Absent*				
6. Airbill No.: _____				
7. Sample Tags Present/Absent*				
Sample Tag Numbers Listed/Not Listed on Chain-of-Custody				
8. Sample Condition: Intact/Broken*/Leaking				
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*				
10. Date Received at Lab: _____				
11. Time Received: _____				
Sample Transfer				
Fraction: _____				
Area #: _____				
By: _____				
On: _____				

Logbook No.: _____
Logbook Page No: _____

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

LABORATORY NAME _____	CITY/STATE _____
CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ SAS NO. _____	
CONTRACT NO. _____ SOW NO. _____	

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II, PARAGRAPH 5, and SECTION III, PARAGRAPH 16.)

		PAGE NOs		CHECK	
		FROM	TO	LAB	EPA
1.	<u>Inventory Sheet</u> (Form DC-2) (Do not number)	_____	_____	_____	_____
2.	<u>SDG Case Narrative</u>	_____	_____	_____	_____
3.	<u>Traffic Report</u>	_____	_____	_____	_____
4.	<u>Volatiles Data</u>				
	a. QC Summary				
	Surrogate Percent Recovery Summary (Form II LCV)	_____	_____	_____	_____
	Lab Control Sample Recovery (Form III LCV)	_____	_____	_____	_____
	Method Blank Summary (Form IV LCV)	_____	_____	_____	_____
	Tuning and Mass Calibration (Form V LCV)	_____	_____	_____	_____
	b. Sample Data	_____	_____	_____	_____
	TCL Results - (Form I LCV)			_____	_____
	Tentatively Identified Compounds (Form I LCV-TIC)			_____	_____
	Reconstructed total ion chromatograms (RIC)			_____	_____
	and Quantitation Reports for each sample			_____	_____
	For each sample:				
	Raw spectra and background-subtracted				
	mass spectra of target compounds identified			_____	_____
	Mass spectra of all reported TICs with three				
	best library matches			_____	_____
	c. Standards Data (All Instruments)				
	Initial Calibration Summary (Form VI LCV)	_____	_____	_____	_____
	RICs and Quan Reports for all Standards	_____	_____	_____	_____
	Continuing Calibration (Form VII LCV)	_____	_____	_____	_____
	RICs and Quant Reports for all Standards	_____	_____	_____	_____
	Internal Standard Area and RT Summary	_____	_____	_____	_____
	(Form VIII LCV)	_____	_____	_____	_____
	d. QC Data				
	BFB	_____	_____	_____	_____
	Blank Data	_____	_____	_____	_____
	LCS Data	_____	_____	_____	_____
	PES Data	_____	_____	_____	_____
5.	<u>Semivolatiles Data</u>				
	a. QC Summary				
	Surrogate Percent Recovery Summary (Form II LCSV)	_____	_____	_____	_____
	Lab Control Sample Recovery (Form III LCSV)	_____	_____	_____	_____
	Method Blank Summary (Form IV LCSV)	_____	_____	_____	_____
	Tuning and Mass Calibration (Form V LCSV)	_____	_____	_____	_____

FORM DC-2-1

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

CASE NO. _____	SDG NO. _____	SDG NOS. TO FOLLOW _____	SAS NO. _____
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	PAGE NOS		CHECK	
	FROM	TO	LAB	EPA
5. <u>Semivolatiles Data</u> (cont.)				
b. Sample Data	_____	_____	_____	_____
TCL Results (Form I LCSV)				
Tentatively Identified Compounds (Form I LCSV-TIC)			_____	_____
Reconstructed total ion chromatograms (RIC)			_____	_____
and Quantitation Reports for each sample			_____	_____
For each sample:				
Raw spectra and background-subtracted				
mass spectra of TCL compounds			_____	_____
Mass spectra of TICs with three best library matches			_____	_____
GPC chromatograms (if GPC performed)			_____	_____
c. Standards Data (All Instruments)				
Initial Calibration Summary (Form VI LCSV)	_____	_____	_____	_____
RICs and Quan Reports for all Standards	_____	_____	_____	_____
Continuing Calibration (Form VII LCSV)	_____	_____	_____	_____
RICs and Quan Reports for all Standards	_____	_____	_____	_____
Internal Standard Area and RT Summary				
(Form VIII LCSV)	_____	_____	_____	_____
d. QC Data				
DFTPP	_____	_____	_____	_____
Blank Data	_____	_____	_____	_____
LCS Data	_____	_____	_____	_____
PES Data	_____	_____	_____	_____
6. <u>Pesticides</u>				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II LCP)	_____	_____	_____	_____
Lab Control Sample Recovery (Form III LCP)	_____	_____	_____	_____
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 and				
calibration plots			_____	_____
Manual work sheets			_____	_____
For pesticides/Aroclors confirmed by GC/MS, copies				
of raw spectra and copies of background-subtracted mass				
spectra of target compounds (samples & standards)			_____	_____

LOW CONCENTRATION WATER FOR ORGANICS COMPLETE SDG FILE (CSP) INVENTORY SHEET (Cont.)

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

	PAGE NOS FROM	TO	CHECK LAB	EPA
5. <u>Pesticides (cont.)</u>				
c. Standards Data				
Initial Calibration Data (Form VI LCP)	_____	_____	_____	_____
Calibration Verification (Form VII LCP)	_____	_____	_____	_____
Pesticides Analytical Sequence (Form VIII, LCP-1 and -2)	_____	_____	_____	_____
Pesticide Florisil Cartridge Check (Form IX, LCP)	_____	_____	_____	_____
Pesticide Identification (Form X LCP)	_____	_____	_____	_____
Standard chromatograms and data system printout for all Standards	_____	_____	_____	_____
For pesticides/Aroclors confirmed by GC/MS, copies of spectra for standards used	_____	_____	_____	_____
d. QC Data				
Blank Data	_____	_____	_____	_____
LCS Data	_____	_____	_____	_____
PES Data	_____	_____	_____	_____
7. <u>Miscellaneous Data</u>				
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)	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
8. <u>EPA Shipping/Receiving Documents</u>				
Airbills (No. of shipments _____)	_____	_____	_____	_____
Chain-of-Custody Records	_____	_____	_____	_____
Sample Tags	_____	_____	_____	_____
Sample Log-In Sheet (Lab & DC1)	_____	_____	_____	_____
SDG Cover Sheet	_____	_____	_____	_____
Miscellaneous Shipping/Receiving Records (describe or list)	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
9. <u>Internal Lab Sample Transfer Records and Tracking Sheets</u> (describe or list)				
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

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

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

PAGE NOs		CHECK	
FROM	TO	LAB	EPA
1	1		
2	2		
3	3		
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10. Other Records (describe or list)

Telephone Communication Log

11. Comments:

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

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

EXHIBIT C

TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

NOTE: The values in these tables are quantitation limits, not absolute detection limits. The amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to simply be detected above the background noise. Except as noted, the quantitation limits in these tables are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Volatiles	CAS Number	Quantitation Limits
		Water ug/L
1. Chloromethane	74-87-3	1
2. Bromomethane	74-83-9	1
3. Vinyl chloride	75-01-4	1
4. Chloroethane	75-00-3	1
5. Methylene chloride	75-09-2	2
6. Acetone	67-64-1	5
7. Carbon disulfide	75-15-0	1
8. 1,1-Dichloroethene	75-35-4	1
9. 1,1-Dichloroethane	75-34-3	1
10. cis-1,2-Dichloroethene	156-59-4	1
11. trans-1,2-Dichloroethene	156-60-5	1
12. Chloroform	67-66-3	1
13. 1,2-Dichloroethane	107-06-2	1
14. 2-Butanone	78-93-3	5
15. Bromochloromethane	74-97-5	1
16. 1,1,1-Trichloroethane	71-55-6	1
17. Carbon Tetrachloride	56-23-5	1
18. Bromodichloromethane	75-27-4	1
19. 1,2-Dichloropropane	78-87-5	1
20. cis-1,3-Dichloropropene	10061-01-5	1
21. Trichloroethene	79-01-6	1
22. Dibromochloromethane	124-48-1	1
23. 1,1,2-Trichloroethane	79-00-5	1
24. Benzene	71-43-2	1
25. trans-1,3-Dichloropropene	10061-02-6	1
26. Bromoform	75-25-2	1
27. 4-Methyl-2-pentanone	108-10-1	5
28. 2-Hexanone	591-78-6	5
29. Tetrachloroethene	127-18-4	1

TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(CONT'D.)

Volatiles	CAS Number	Quantitation Limits
		<u>Water</u> ug/L
30. 1,1,2,2-Tetrachloroethane	79-34-5	1
31. 1,2-Dibromoethane	106-93-4	1
32. Toluene	108-88-3	1
33. Chlorobenzene	108-90-7	1
34. Ethylbenzene	100-41-4	1
35. Styrene	100-42-5	1
36. Xylenes (total)	1330-20-7	1
37. 1,3-Dichlorobenzene	541-73-1	1
38. 1,4-Dichlorobenzene	106-46-7	1
39. 1,2-Dichlorobenzene	95-50-1	1
40. 1,2-Dibromo-3-chloropropane	96-12-8	1

NOTE: Except for Methylene chloride, the quantitation limits in this table are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

In the case of Methylene chloride, the CRQL value in this table is based on the lowest level of detection in samples contaminated with this common laboratory solvent that can be achieved by reasonable means in a production laboratory.

**TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CROL)
(CONT'D.)**

Semivolatiles	CAS Number	Quantitation Limits
		<u>Water</u> ug/L
1. Phenol	108-95-2	5
2. bis-(2-Chloroethyl)ether	111-44-4	5
3. 2-Chlorophenol	95-57-8	5
4. 2-Methylphenol	95-48-7	5
5. 2,2'-oxybis(1-Chloropropane)	108-60-1	5
6. 4-Methylphenol	106-44-5	5
7. N-Nitroso-di-n-propylamine	621-64-7	5
8. Hexachloroethane	67-72-1	5
9. Nitrobenzene	98-95-3	5
10. Isophorone	78-59-1	5
11. 2-Nitrophenol	88-75-5	5
12. 2,4-Dimethylphenol	105-67-9	5
13. bis-(2-Chloroethoxy)methane	11-91-1	5
14. 2,4-Dichlorophenol	120-83-2	5
15. 1,2,4-Trichlorobenzene	120-82-1	5
16. Naphthalene	91-20-3	5
17. 4-Chloroaniline	106-47-8	5
18. Hexachlorobutadiene	87-68-3	5
19. 4-Chloro-3-methylphenol	59-50-7	5
20. 2-Methylnaphthalene	91-57-6	5
21. Hexachlorocyclopentadiene	77-47-4	5
22. 2,4,6-Trichlorophenol	88-06-2	5
23. 2,4,5-Trichlorophenol	95-95-4	20
24. 2-Chloronaphthalene	91-58-7	5
25. 2-Nitroaniline	88-74-4	20
26. Dimethylphthalate	131-11-3	5
27. Acenaphthylene	208-96-8	5
28. 2,6-Dinitrotoluene	606-20-2	5
29. 3-Nitroaniline	99-09-2	20
30. Acenaphthene	83-32-9	5
31. 2,4-Dinitrophenol	51-28-5	20
32. 4-Nitrophenol	100-02-7	20
33. Dibenzofuran	132-64-9	5

TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(CONT'D.)

Semivolatiles	CAS Number	Quantitation Limits
		<u>Water</u> ug/L
34. 2,4-Dinitrotoluene	121-14-2	5
35. Diethylphthalate	84-66-2	5
36. 4-Chlorophenyl-phenylether	7005-72-3	5
37. Fluorene	86-73-7	5
38. 4-Nitroaniline	100-01-6	20
39. 4,6-Dinitro-2-methylphenol	534-52-1	20
40. N-Nitrosodiphenylamine	86-30-6	5
41. 4-Bromophenyl-phenylether	101-55-3	5
42. Hexachlorobenzene	118-74-1	5
43. Pentachlorophenol	87-86-5	20
44. Phenanthrene	85-01-8	5
45. Anthracene	120-12-7	5
46. Di-n-butylphthalate	84-74-2	5
47. Fluoranthene	206-44-0	5
48. Pyrene	129-00-0	5
49. Butylbenzylphthalate	85-68-7	5
50. 3,3'-Dichlorobenzidine	91-94-1	5
51. Benzo(a)anthracene	56-55-3	5
52. Chrysene	218-01-9	5
53. bis-(2-Ethylhexyl)phthalate	117-81-7	5
54. Di-n-octylphthalate	117-84-0	5
55. Benzo(b)fluoranthene	205-99-2	5
56. Benzo(k)fluoranthene	207-08-9	5
57. Benzo(a)pyrene	50-32-8	5
58. Indeno(1,2,3-cd)pyrene	193-39-5	5
59. Dibenz(a,h)anthracene	53-70-3	5
60. Benzo(g,h,i)perylene	191-24-2	5

TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)
(CONT'D.)

Pesticides/PCBs	CAS Number	Quantitation Limits
		<u>Water</u> ug/L
1. alpha-BHC	319-84-6	0.01
2. beta-BHC	319-85-7	0.01
3. delta-BHC	319-36-8	0.01
4. gamma-BHC (Lindane)	58-89-9	0.01
5. Heptachlor	76-44-8	0.01
6. Aldrin	309-00-2	0.01
7. Heptachlor epoxide	1024-57-3	0.01
8. Endosulfan I	959-98-8	0.01
9. Dieldrin	60-57-1	0.02
10. 4,4'-DDE	72-55-9	0.02
11. Endrin	72-20-8	0.02
12. Endosulfan II	33213-65-9	0.02
13. 4,4'-DDD	72-54-8	0.02
14. Endosulfan sulfate	1031-07-8	0.02
15. 4,4'-DDT	50-29-3	0.02
16. Methoxychlor	72-43-5	0.10
17. Endrin ketone	53494-70-5	0.02
18. Endrin aldehyde	7421-36-3	0.02
19. alpha-Chlordane	5103-71-9	0.01
20. gamma-Chlordane	5103-74-2	0.01
21. Toxaphene	8001-35-2	1.0
22. Aroclor-1016	12674-11-2	0.20
23. Aroclor-1221	11104-28-2	0.40
24. Aroclor-1232	11141-16-5	0.20
25. Aroclor-1242	53469-21-9	0.20
26. Aroclor-1248	12672-29-6	0.20
27. Aroclor-1254	11097-69-1	0.20
28. Aroclor-1260	11096-82-5	0.20

EXHIBIT D

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

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SECTION I
INTRODUCTION

The analytical method that follows is designed to analyze 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 from drinking water and well/ground water sources around Superfund sites. The method is based on EPA Method 524.2 and the volatile method contained in the CLP Statement of Work, "Organic Analysis, Multi Media, Multi Concentrations by GC/MS and GC/EC Techniques". Specific quality control requirements are incorporated in the method in order to minimize contamination of the samples from laboratory sources.

Problems have been associated with the following compounds analyzed by this method.

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

SECTION II

PART A - SAMPLE STORAGE AND HOLDING TIMES

1. PROCEDURES FOR SAMPLE STORAGE

The samples must be protected from light and refrigerated at 4°C ($\pm 2^\circ\text{C}$) from the time of receipt until 60 days after delivery of a complete reconciled sample data package. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

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 purgeable samples received under this contract.

Samples, sample extracts, and standards must be stored separately.

Volatiles standards must be stored separately from semivolatile and pesticide/Aroclor standards.

2. CONTRACT REQUIRED HOLDING TIMES

Analysis of samples must be completed within 10 days of the validated time of sample receipt (VTSR).

PART B

3. SUMMARY OF METHOD

- 3.1 An inert gas is bubbled through a 25 mL sample contained in a specially designed purging chamber at ambient temperature causing the purgeables to be transferred from the 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 gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer (MS).
- 3.2 Target and surrogate compounds are identified in the samples by analyzing standards under the same conditions used for samples and comparing resultant mass spectra and GC retention times. Internal standards are added to all samples and standards. A response factor is established for each target and surrogate compound during the initial and continuing calibrations by comparing the MS response from the extracted ion current profile (EICP) for the primary ion produced by the target and surrogate compound to the MS response for the primary ion produced by an internal standard compound. Each identified target and surrogate compound in a sample is quantified by comparing the responses for the target compound and the internal standard, while taking into account the response factor from the most recent calibration, the sample volume, and any sample dilutions.
- 3.3 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology Mass Spectral Library. Non-target compounds are quantified by comparing the MS response from the reconstructed ion chromatogram (RIC) for the non-target compound peaks to the MS response for a peak produced by the nearest internal standard compound. A response factor of 1 is assumed.

4. INTERFERENCES

- 4.1 Impurities in the purge gas or methanol, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. Gas lines from the gas tanks to the instrument must be either stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. When potential interfering peaks are noted in laboratory method blanks, it may be necessary to reduce solvent contamination in the laboratory, purge the methanol used to prepare standard solutions, purge the reagent water with helium or nitrogen, change the purge gas source, or regenerate the molecular sieve purge gas filter.
- 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. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing lines are to be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. Laboratory clothing worn by the analyst must be clean of solvents since clothing previously exposed to methylene chloride and other laboratory solvent fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

- 4.3 Contamination by carryover can occur whenever a sample is analyzed after a high level sample which contain high levels of organic compounds. To reduce carryover, the sample purger 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 is necessary to wash out the sample purger with a detergent solution between analyses, rinse it with distilled water, and then dry it in a 105°C oven. 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.

5. APPARATUS AND MATERIALS

Brand names, suppliers and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of the method is the responsibility of the Contractor.

- 5.1 Purge and Trap Device - The purge and trap device consists of three separate pieces of equipment; the sample purger, the trap, and the trap heater. Purge gas plumbing lines are to be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used.
- 5.1.1 Sample Purger - The sample purger must be designed to accept 25 mL samples with a water column at least 10 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 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternate sample purge devices may be utilized provided equivalent performance is demonstrated. A set of sample purgers sufficient to meet contract requirements must be reserved for exclusive use in this contract.

5.1.2 Trap Packing

- 5.1.2.1 2,6-Diphenylene oxide polymer, 60/80 mesh, chromatographic grade (Tenax GC or equivalent).
- 5.1.2.2 Methyl silicone packing, 3 percent OV-1 on Chromosorb W, 60/80 mesh (or equivalent).
- 5.1.2.3. Silica gel (35/60 mesh, Davison, grade 15 or equivalent).

5.1.3 Trap - The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch (0.2667 cm). Starting from the inlet, pack the column in the following order: 0.5 cm silanized glass wool, 1 cm methyl silicone coated packing, 15 cm 2,6-diphenylene oxide polymer, 8 cm silica gel, and 0.5 cm silanized glass wool.

5.1.4 Trap Heater

- 5.1.4.1 The trap heater must be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated to greater than 180°C and the temperature of the remaining sections should not exceed 220°C during bakeout mode.
- 5.1.4.2 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph.

5.2 Gas Chromatograph/Mass Spectrometer

5.2.1 Gas Chromatograph - The gas chromatograph (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 5.1 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; therefore, a subambient oven controller is required.

5.2.1.1 Gas Chromatography Columns

Column 1-30 m long x 0.53 mm ID VOCOL (Supelco, Inc.) or equivalent fused silica wide-bore capillary column with 3 um film thickness.

Column 2-30 m long x 0.53 mm ID DB-624 fused silica wide-bore (J&W Scientific, Inc.) or equivalent column with 3 um film thickness.

- 5.2.2 Mass Spectrometer - The mass spectrometer must be capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the tuning acceptance criteria when 50 ng of p-bromofluorobenzene (BFB) are injected through the gas chromatograph 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 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.
- 5.2.3 GC/MS Interface - Any gas chromatograph to mass spectrometer interface may be used that gives acceptable calibration points at 25 ng or less per injection for each of the purgeable target and surrogate compounds and achieves all acceptable performance criteria. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.
- 5.2.4 Data System - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching 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 most recent release of the National Institute of Standards and Technology Mass Spectral Library shall be used as the reference library.
- 5.3 Magnetic Tape Storage Device - The magnetic tape storage device must be capable of recording data and must be suitable for long-term, off-line storage.
- 5.4 pH paper - wide range.
- 5.5 Glassware - A set of glassware sufficient to meet the contract requirements must be reserved for exclusive use in this contract.
- 5.5.1 Bottles - 15 mL, screw-cap, with Teflon cap liner.
- 5.5.2 Volumetric flasks - class A with ground-glass stoppers.

- 5.6 Micro syringes - 10 uL and larger, 0.006 inch ID needle.
- 5.7 Syringe valve - two-way, with Luer ends (three each), if applicable to the purging device.
- 5.8 Syringe - 25 mL, gas tight with shut-off valve.
- 5.9 Analytical Balance - capable of accurately weighing ± 0.0001 g. The balance must be calibrated with class S weights once per each 12-hour workshift. The balance must also be annually checked by a certified technician.
- 5.10 Gases - Helium, Nitrogen. Ultra-pure grade.
- 5.11 Gas line tubing - polytetrafluoroethylene, stainless steel, or copper tubing.

6. REAGENTS

- 6.1 Reagent water - Reagent water is defined as water in which no purgeable target compound is observed at or above the CRQL listed in Exhibit C for that compound and in which no non-target compound is observed at or above 2.0 ug/L.
 - 6.1.1 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).
 - 6.1.2 Reagent water may be generated using a water purification system (Millipore Super-Q, or equivalent).
 - 6.1.3 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 Teflon-lined septum, and cap.
- 6.2 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 the Exhibit C.

7. STANDARDS

- 7.1 The Contractor must provide all standards to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E. 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 Stock Standard Solutions

Stock standard solutions may be purchased or may be prepared in methanol from pure standard materials.

- 7.2.1 Prepare stock standard solutions by placing about 9.8 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.
- 7.2.2 Add the assayed reference material as described below.
 - 7.2.2.1 If the compound is a liquid, using a 100. μ L syringe, 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.
 - 7.2.2.2 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 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 Teflon tubing to the side-arm relief valve and direct a gentle stream of the reference standard into the methanol meniscus.
- 7.2.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. For non-gaseous compounds, calculate the concentration in micrograms per microliter 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 micrograms per microliter, using the Ideal Gas Law, taking into account the temperature and pressure conditions within the laboratory.
- 7.2.4 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.

7.3 Secondary Dilution Standards

- 7.3.1 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.

- 7.3.2 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.

7.4 Working Standards

7.4.1 Tuning Solution - p-Bromofluorobenzene (BFB)

Prepare a 25 ng/uL solution of BFB in methanol. Prepare fresh BFB solution every six months, or sooner, if the solution has degraded or evaporated. Note: The 25 ng/uL concentration is used with a 2 uL injection volume. The laboratory may prepare a 50 ng/uL solution of BFB if a 1 uL injection volume is used.

7.4.2 Calibration Standard Solution

Prepare the working calibration standard solution containing all of the purgeable target compounds in methanol. The concentration of the non-ketone target compounds and the surrogate must be 5.0 ug/mL in the standard (i.e., final concentration). The concentration of the ketones (acetone, butanone, 2-hexanone, 4-methyl-2-pentanone) must be 25.0 ug/mL in the standard (i.e., final concentration). Prepare fresh working calibration standard solutions weekly, or sooner, if solutions have degraded or evaporated.

7.4.3 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 ug/mL for each internal standard. Add 10 uL of this spiking solution into 25.0 mL of sample or calibration standard for a concentration of 5.0 ug/L. Prepare fresh spiking solution every three months, or sooner, if the solution has degraded or evaporated.

7.4.4 Surrogate Compound Spiking Solution

Prepare 12.5 ng/uL solution of p-bromofluorobenzene in methanol. For samples and blanks, add 10 uL of the surrogate compound solution to 25 mL of sample or reagent water for a final concentration of 5 ug/L. The surrogate compound is added to the working calibration standards so it is not to be added again when aqueous calibration standards are prepared. Prepare fresh surrogate compound solution every six months, or sooner, if the solution has degraded or evaporated.

7.4.5 Laboratory Control Sample (LCS) Spiking Solution

Prepare the laboratory control sample spiking solution containing all of the analytes listed below at 12.5 ng/uL in methanol. The laboratory control sample is prepared by adding 10 uL of this solution into 25.0 mL of reagent water for a concentration of 5.0 ug/L. Prepare fresh LCS spiking solution every six months, or sooner, if the solution has degraded or evaporated. The LCS must contain the following compounds:

1,2-Dibromoethane	1,4-Dichlorobenzene
Benzene	cis-1,3-Dichloropropene
1,2-Dichloroethane	1,1,2-Trichloroethane
1,2-Dichloropropane	Vinyl chloride
Tetrachloroethene	Bromoform
Carbon tetrachloride	Trichloroethene

7.5 Aqueous Calibration Standard Solutions

- 7.5.1 Prepare five aqueous initial calibration standard solutions containing all of the purgeable target compounds and the surrogate compound at the 1.0, 2.0, 5.0, 10, and 25 ug/L levels, except the ketones which are at the 5.0, 10, 25, 50, and 125 ug/L levels. 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., 1.0, 2.0, 5.0, 10.0 and 25 ug/L). The preparation procedure for the aqueous calibration standard solutions is listed in Table D-1. The internal standards are added to each calibration standard according to the procedures in Section 14.3.5.

TABLE D-1

VOL OF WORKING STANDARD (uL added to 25 mL)	FINAL CONC. OF AQUEOUS STANDARD FOR NON-KETONES (ug/L)	FINAL CONC. OF AQUEOUS STANDARD FOR KETONES (ug/L)
5	1	5
10	2	10
25	5	25
50	10	50
125	25	125

7.5.2 Aqueous calibration standards may be prepared in a volumetric flask or in the syringe used to inject the standard into the purging device.

7.5.2.1 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.

7.5.2.2 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.

7.5.3 The 5 ug/L (25 ug/L for ketones) aqueous calibration standard solution is the continuing calibration standard.

7.5.4 A smaller sample or standard volume (5 to 25 mL) may be purged but the CRQL and all QC criteria must be met. The same purge volume must be used for all samples, standards, and blanks in a single SDG.

7.5.5 The methanol contained in each of the aqueous calibration standards must not exceed 1% by volume.

7.6 Storage of Standards

7.6.1 Store the stock standards in Teflon sealed screw-cap bottles with zero headspace at -10°C to -20°C. 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.

7.6.2 Store secondary dilution standards in Teflon 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.

- 7.6.3 Aqueous standards may be stored up to 24 hours if held in Teflon sealed screw-cap vials with zero headspace at 4°C ($\pm 2^\circ\text{C}$). 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.
- 7.6.4 Purgeable standards must be stored separately from other standards.

SECTION III

INSTRUMENT QUALITY CONTROL PROCEDURES AND REQUIREMENTS

PART A - INSTRUMENT OPERATING CONDITIONS

Purge and Trap

The following are the recommended purge and trap analytical conditions except as stated below:

Purge Conditions:

Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 \pm 0.1 min
Purge Flow Rate:	25-40 mL/min
Purge Temperature:	Ambient

Desorb Conditions:

Desorb Temperature:	180°C
Desorb Flow Rate:	15 mL/min
Desorb Time:	4.0 \pm 0.1 min

Trap Reconditioning Conditions:

Reconditioning Temperature:	180°C
Reconditioning Time:	7.0 min \pm 0.1 min (minimum). A longer time may be required to bake contamination or water from the system.

Before initial use, condition the trap overnight at 180°C by backflushing with at least 20 mL/min flow of inert gas. Vent the trap effluent to the hood and not to 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 analysis of samples.

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 standard, samples, blanks, Performance Evaluation Samples and Laboratory Control Samples

Gas Chromatograph

The following are the recommended gas chromatographic analytical conditions:

Carrier Gas:	Helium
Flow Rate:	15 mL/min
Initial Temperature:	10°C
Initial Hold Time:	1.0 - 5.0 min (\pm 0.1 min precision)
Ramp Rate:	6°C/min
Final Temperature:	160°C
Final Hold Time:	Until all target compounds elute

Optimize GC conditions for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, performance evaluation samples, and laboratory control samples.

Mass Spectrometer

The following are the required mass spectrometer conditions:

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

PART B - TUNING THE MASS SPECTROMETER

8. SUMMARY

It is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria prior to initiating any on-going data collection. This is accomplished through the analysis of p-bromofluorobenzene (BFB).

9. FREQUENCY

Each GC/MS used for volatile analysis must be hardware tuned once per twelve (12) hour time period of operation. Also, whenever the Contractor takes corrective action which could change or affect the tuning for BFB (e.g., ion source cleaning or repair, column replacement, etc.), the tune must be verified before continuing analysis irrespective of the twelve-hour daily tuning requirement. The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the BFB analysis that the Contractor submits as documentation of a compliant tune. The time period ends after twelve (12) hours have elapsed. In order to meet the tuning requirements, a sample, LCS, PES, standard, or blank must be injected within twelve hours of the BFB injection.

10. PROCEDURE

10.1 Inject 50 ng BFB into the GC/MS system. Alternatively, add 50 ng of BFB solution to 25.0 mL of reagent water and analyze according to Section 17. All instrument conditions must be identical to those listed in Section III, Part A, except that a different temperature program may be used.

10.2 For the tune, BFB may not be analyzed simultaneously with any calibration standards or blanks.

11. TECHNICAL ACCEPTANCE CRITERIA FOR BFB ANALYSIS

11.1 The GC/MS system must be tuned at the frequency described in Section 9.

11.2 The abundance criteria listed in Table D-2 must be met for a 50 ng injection of BFB. The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of BFB. Note: All subsequent standards and samples, including LCS, PES and blanks associated with a BFB analyses must use identical mass spectrometer instrument conditions.

11.3 The criteria listed in Table D-2 are based on adherence to the acquisition specifications identified in Section 11.2 and were developed for the specific target compound list associated with this method. The criteria are based on performance characteristics of

instruments currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for 28 target compounds (see Table D-5), are designed to control and monitor instrument performance associated with the requirements of this method. As they are performance based criteria for these specific analytical requirements, they may not be optimal for additional target compounds.

TABLE D-2 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.

12. CORRECTIVE ACTION

- 12.1 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.
- 12.2 BFB technical acceptance criteria MUST be met before any standards, samples or required blanks are analyzed. Any samples (including LCS and PES), or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis at no additional cost.

PART C - CALIBRATION OF THE GC/MS SYSTEM

14. INITIAL CALIBRATION

14.1 Summary - Prior to the analysis of samples and required blanks and after BFB 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 surrogate compounds.

14.2 Frequency

14.2.1 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 acceptance criteria have not been met.

14.2.2 If time remains in the 12 hour time period after meeting the technical acceptance criteria for the initial calibration, samples 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. Quantify all sample and quality control sample results and quality control criteria results, such as internal standard area response change and retention time shift, against the initial calibration standard that is the same concentration as the continuing calibration standard.

14.3 Procedure

14.3.1 Set-up the purge and trap GC/MS system per the requirements of Section III, Part A.

14.3.2 All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.

14.3.3 Tune the GC/MS system to meet the BFB technical acceptance criteria in Section 11.

14.3.4 Prepare a spiking solution containing the internal standards using the procedure described in Section 7.4.3.

14.3.5 Prepare five aqueous calibration standards containing all the purgeable target compounds and the surrogate using the procedure described in Section 7.5. Add 10 uL of the internal standard solution to each aqueous standard. Analyze each calibration standard, according to Section 17. If a compound saturates at the highest standard concentration (Section 7.5.1), and the GC/MS system is calibrated to achieve a

detection sensitivity of no less than 1.0 ug/L, the Contractor must document it in the SDG Narrative and attach a quantitation report and RIC. In this instance, the Contractor must calculate the results based on a four-point initial calibration for the specific compound that saturates. Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion. If secondary ion quantitation is used, calculate a relative response factor using the area response from the most intense secondary ion which is free of sample interferences and document the reasons for the use of the secondary ion in the SDG Narrative.

14.4 Calculations

NOTE: Unless otherwise stated the area response is that of the primary quantitation ion.

Calculating the relative response factor of the xylenes requires special attention. On capillary columns, the m- and p-xylene isomers coelute. Therefore, when calculating the relative response factor in the equation below, use the area response (A_x) and concentration (C_x) of the peak from o-xylene.

- 14.4.1 Calculate relative response factors (RRF) for each purgeable target compound and the surrogate using Equation D.1. See Table D-3 to associate purgeable target compounds with the proper internal standard. See Table D-4 for primary quantitation ions to be used for each purgeable target, surrogate, and internal standard compounds.

$$\text{EQ. D.1} \quad \text{RRF} = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x - Area response (EICP) for the compound to be measured.

A_{is} - Area response (EICP) for the internal standard.

C_{is} - Concentration of the internal standard.

C_x - Concentration of the compound to be measured.

- 14.4.2 Equation D.2 is the general formula for standard deviation (SD) for a statistically small set of values.

$$\text{EQ. D.2.} \quad \text{SD} = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}$$

SD - Standard deviation for a set of values.
 x_i - Value.
 \bar{x} - Mean value.
 n - Number of values.

- 14.4.3 Calculate the percent relative standard deviation (% RSD) of RRF values for each purgeable target and surrogate compound over the initial calibration range using Equation D.3 in conjunction with Equation D.2.

$$\text{EQ. D.3} \quad \% \text{RSD} = \frac{SD_{\text{RRF}}}{\bar{x}} \times 100$$

Where:

%RSD - Percent relative standard deviation.

SD_{RRF} - Standard deviation of initial calibration response factors (per compound).

From EQ. D.2:

$x_i = \text{RRF}_i$ - Relative response factors from initial calibration standard (per compound).

$\bar{x} = \overline{\text{RRF}}$ - Mean of initial calibration response factors (per compound).

- 14.4.4 Equation D.4 is the general formula for the mean of a set of values.

EQ. D.4

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$$

x_i - Value.

\bar{x} - Mean value.

n - Number of values.

- 14.4.5 Calculate the mean of the relative retention times ($\overline{\text{RRT}}$) for each purgeable target and surrogate compound over the initial calibration range using Equation D.4 and Equation D.5.

$$\text{EQ. D.5} \quad \text{RRT} = \frac{RT_c}{RT_{is}}$$

RT_c - Retention time for the purgeable target and surrogate compound.

RT_{is} - Retention time for the internal standard.

From EQ D.4:

$x_i = RRT_i$ - Relative retention times for the purgeable target or surrogate compound for each initial calibration standard.

$\bar{x} = \overline{RRT}$ - Mean relative retention time (per compound).

n - Number of values.

- 14.4.6 Calculate the area response (\bar{Y}) mean for each internal standard over the initial calibration range using Equation D.4.

Where:

$x_i = Y_i$ - Area responses for the internal standard in each of the calibration standards.

$\bar{x} = \bar{Y}$ - Area response mean.

n - Number of values.

- 14.4.7 Calculate the mean of the absolute retention times (\overline{RT}) for each internal standard over the initial calibration range using Equation D.4.

Where:

$x_i = RT_i$ - Retention times for the internal standard in each of the calibration standards.

$\bar{x} = \overline{RT}$ - Mean absolute retention time.

n - Number of values.

14.5 Technical Acceptance Criteria For Initial Calibration

- 14.5.1 All initial calibration standards must be analyzed at the concentration levels described in Section 14.3, and at the frequency described in Section 14.2 on a GC/MS system meeting the BFB technical acceptance criteria.
- 14.5.2 The relative response factor (RRF) at each calibration concentration for each purgeable target and surrogate compound that has a required minimum response factor value must be greater than or equal to the compound's minimum acceptable response factor listed in Table D-5.

- 14.5.3 The %RSD for each target or surrogate compound listed in Table D-5 must be less than or equal to that value listed.
- 14.5.4 Up to two compounds may fail the criteria listed in paragraphs 14.5.2 and 14.5.3 and still meet the minimum response factor 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%.
- 14.5.5 The relative retention time (RRT) at each calibration level must be within ± 0.06 RRT units of mean relative retention time (RRT) over all the calibration levels for each purgeable target compound and the surrogate.
- 14.5.6 The area response (Y) for each internal standard compound in each calibration standard must be within the inclusive range of ± 40.0 percent of the mean area response (\bar{Y}) of the internal standard in all of the calibration standards.
- 14.5.7 The retention time (RT) shift for each internal standard at each calibration level must be less than or equal to ± 0.33 minutes (20.0 seconds) from the mean retention time (RT) over all calibration levels for each internal standard.

14.6 Corrective Action

- 14.6.1 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.
- 14.6.2 Initial calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed. Any samples (including LCS and PES) or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost.

15. CONTINUING CALIBRATION

- 15.1 Summary - Prior to the analysis of samples and required blanks and after BFB and initial calibration 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 surrogate compounds to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the method.

15.2 Frequency

- 15.2.1 Each GC/MS used for analysis must be calibrated once per each twelve (12) hour time period of operation. The twelve hour time period begins with the injection of BFB.

- 15.2.2 If time remains in the 12 hour time period after meeting the technical acceptance criteria for the initial calibration, samples 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. Quantify all sample results against the initial calibration standard that is the same concentration as the continuing calibration standard (5 ug/L for non-ketones, 25 ug/L for ketones).

15.3 Procedure

- 15.3.1 Set up the purge and trap GC/MS system per the requirements of Section III, Part A.
- 15.3.2 All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.
- 15.3.3 Tune the GC/MS system to meet the BFB technical acceptance criteria in Section 11.
- 15.3.4 Prepare a working continuing calibration standard solution containing all the purgeable target and surrogate compounds using the procedure listed in Section 7.4.2. The concentration for the non-ketones and the surrogate compound will be 5 ug/L. The concentration of the ketones will be 25 ug/L.
- 15.3.5 Prepare the aqueous continuing calibration solution by adding 25 uL of the working calibration standard to reagent water as described in Paragraph 7.5.
- 15.3.6 Add 10 uL of internal standard spiking solution (prepared as described in Paragraph 7.4.3) to the 25 mL syringe or volumetric flask containing the continuing calibration standard. Analyze the continuing calibration standard, according to Section 17.

15.4 Calculations

- 15.4.1 Calculate a relative response factor (RRF) for each target and surrogate compound according to Section 14.4.1.
- 15.4.2 Calculate the percent difference between the continuing calibration relative response factor and the most recent initial calibration mean relative response factor for each purgeable target and surrogate compound using Equation D.6.

$$\text{EQ D.6} \quad \% \text{ D}_{\text{RRF}} = \frac{\overline{\text{RRF}}_i - \text{RRF}}{\overline{\text{RRF}}_i} \times 100$$

Where:

% D_{RRF} - Percent difference between relative response factors.

RRF - Relative response factor from current continuing calibration standard.

\overline{RRF}_1 - Mean relative response factor from the most recent initial calibration.

15.5 Technical Acceptance Criteria For Continuing Calibration

- 15.5.1 The concentration of the volatile organic target and surrogate compounds in the continuing calibration standard must be 5.0 ug/L for non-ketones and 25.0 ug/L for ketones. The continuing calibration standard must be analyzed at the frequency described in Section 15.2 on a GC/MS system meeting the BFB and the initial calibration technical acceptance criteria.
- 15.5.2 The relative response factor for each purgeable target and surrogate compound that has a required minimum response factor value must be greater than or equal to the compound's minimum acceptable response factor listed in Table D-5.
- 15.5.3 The relative response factor percent difference for each purgeable target and surrogate compound listed in Table D-5 must be less than or equal to that value listed.
- 15.5.4 Up to two compounds may fail the requirements listed in paragraph 15.5.2 and 15.5.3 and still meet the minimum response factor criteria and percent difference criteria. However, these compounds must have a minimum response factor greater than or equal to 0.010 and the percent difference must be within the inclusive range of $\pm 40.0\%$.

15.6 Corrective Action

- 15.6.1 If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 14. 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.
- 15.6.2 Continuing calibration technical acceptance criteria MUST be met before any samples (including LCS and PES) or required blanks are analyzed. Any samples, or required blanks analyzed when continuing calibration technical acceptance criteria have not been met will require reanalysis at no additional cost.

SECTION IV

SAMPLE ANALYSIS AND COMPOUND IDENTIFICATION AND QUANTITATION

16. SUMMARY

- 16.1 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 SMO for instructions.
- 16.2 Before samples or required blanks may be analyzed, the purge and trap, and the GC/MS instrument must meet the BFB (Section 11), initial calibration (Section 14) and continuing calibration (Section 15) technical acceptance criteria listed. Also, before samples may be analyzed, a method blank must be analyzed which meets blank technical acceptance criteria listed in Section 26. All samples, required blanks, and calibration standards must be analyzed under the same instrument conditions. All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before analysis.

17. PROCEDURE

- 17.1 Set up the purge and trap GC/MS system per the requirements of Section III, Part A.
- 17.2 Remove the plunger from a 25 mL syringe that has a closed syringe valve attached. Open the sample or standard container which has been allowed to come to ambient temperature (approximately 1 hour), and carefully decant 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 ($\pm 2^\circ\text{C}$).

NOTE: A smaller sample volume (5 to 25 mL) may be analyzed but the CROL and all technical acceptance criteria must be met. The same sample volume must be used for all standards, samples, and blanks that apply to a single SDG.

- 17.3 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.
- 17.4 Add 10.0 uL of the internal standard spiking solution and 10.0 uL of the surrogate standard solution through the valve bore of the syringe, then close the valve. Invert the syringe three times.

17.5 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.

17.6 Close both valves and purge the sample for 11.0 (± 0.1) minutes at ambient temperature.

17.7 **Sample Desorption**

After the 11 minute purge, attach the trap to the gas chromatograph, adjust the purge and trap system to the desorb mode, initiate the temperature program sequence of the gas chromatograph 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 gas chromatograph, 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 a 105°C oven between analyses.

17.8 **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.

17.9 **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 all target 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.

18. **TERMINATION OF DATA ACQUISITION**

When all 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).

19. **DILUTIONS**

19.1 If the initial analysis of a sample indicates the sample must be reanalyzed at a dilution, the dilution 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. 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 in the SDG Narrative.

- 19.2 All dilutions must keep the response of the target compounds that required dilution in the upper half of the initial calibration range.
- 19.3 Dilutions are made in volumetric flasks or in a 25 mL "Luerlock" syringe.
- 19.3.1 To dilute the sample in a volumetric flask, use the following procedure:
- 19.3.1.1 Select the volumetric flask that will allow for the necessary dilution (25 mL to 100 mL).
 - 19.3.1.2 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.
 - 19.3.1.3 Inject the proper sample aliquot from a syringe into the volumetric flask. Aliquots of less than 1 mL are prohibited. Dilute the flask to the mark with reagent water. Cap the flask, invert, and shake three times.
 - 19.3.1.4 Fill a 25.0 mL syringe with the diluted sample and analyze according to Section 17.
- 19.3.2 To dilute the sample in a 25 mL syringe, use the following procedure:
- 19.3.2.1 Calculate the volume of the reagent water necessary for the dilution. The final volume of the diluted sample should be 25 mL.
 - 19.3.2.2 Close the syringe valve, remove the plunger from the syringe barrel, and pour reagent water into the syringe barrel to just short of overflowing.
 - 19.3.2.3 Replace the syringe plunger and compress the water.
 - 19.3.2.4 Invert the syringe, open the syringe valve, and vent any residual air. Adjust the water volume to the desired amount.
 - 19.3.2.5 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.
 - 19.3.2.6 Analyze according to Sections 17.4 to 18.

20. IDENTIFICATION OF TARGET COMPOUNDS

- 20.1 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: (1) elution of the sample compound at the same GC relative retention time as the standard compound, and (2) correspondence of the sample compound and standard compound mass spectra.
- 20.2 For establishing correspondence of the GC relative retention time (RRT), the sample compound RRT must be within ± 0.06 RRT units of the RRT of the standard compound in the continuing calibration analysis. If co-elution of interfering compounds prohibits accurate assignment of the sample compound RRT from the extracted ion current profile for the primary ion, the RRT must be assigned by using the total ion chromatogram.
- 20.3 For comparison of standard and sample compound 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 BFB technical acceptance criteria. These standard spectra may be obtained from the continuing calibration analysis that was also used to obtain the reference relative retention times.
- 20.4 The guidelines for qualitative verification by comparison of mass spectra are as follows:
- 20.4.1 All ions present in the standard mass spectra at a relative intensity greater than 25 percent (most abundant ion in the spectrum equals 100 percent) should be present in the sample spectrum.
- 20.4.2 The relative intensities of ions specified in Section 20.4.1 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).
- 20.4.3 Ions greater than 25 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should FAVOR FALSE POSITIVES. All compounds meeting the identification criteria must be reported with their spectra.
- 20.4.4 If a compound cannot be verified by all of the spectral identification criteria listed in Sections 20.4.1 - 20.4.3, 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.

21. IDENTIFICATION OF NON-TARGET COMPOUNDS

- 21.1 A library search shall be executed for non-target sample compounds for the purpose of tentative identification. For this purpose, the most recent release of the National Institute of Standards and Technology Mass Spectral Library shall be used.
- 21.2 Up to ten (10) non-internal standard and non-surrogate organic compounds of greatest apparent concentration not listed in Exhibit C for the purgeable organic fraction shall be tentatively identified via a forward search of the NIST Mass Spectral Library. (Compounds with a peak area response less than 40 percent of the peak area response of the best matched internal standard are not required to be searched in this fashion). Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search must not use normalization routines if those routines would misrepresent the library or unknown spectra when compared to each other.
- 21.3 Guidelines for making tentative identification:
- 21.3.1 All ions present in the standard mass spectra at a relative intensity greater than 25 percent (most abundant ion in the spectrum equals 100 percent) must be present in the sample spectrum.
- 21.3.2 The relative intensities of the major ions specified in Section 20.4.1 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.)
- 21.3.3 Molecular ions present in reference spectrum should be present in sample spectrum.
- 21.3.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- 21.3.5 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 co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.
- 21.3.6 Ions greater than 25 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should FAVOR FALSE POSITIVES. All compounds meeting the identification criteria must be reported with their spectra.

- 21.3.6 If in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown aromatic, unknown hydrocarbon, unknown chlorinated compound). If a probable molecular weight can be distinguished, include it.

22. CALCULATIONS

NOTE: Unless otherwise stated the area response is that of the extracted ion current profile (EICP) of the primary quantitation ion.

22.1 Target Compounds

- 22.1.1 Calculate target compound and surrogate concentrations using Equation D.7.

$$\text{EQ. D.7 Concentration in ug/L} = \frac{(A_x)(I_s)(Df)}{(A_{is})(RRF)(V_o)}$$

Where:

- A_x - Area response (EICP) for the compound to be measured. The primary quantitation ions for the target, internal standards, and the surrogate compounds are listed in Table D-4.
- A_{is} - Area response (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 12.5 mL of sample is diluted to 25.0 mL with reagent water and purged, $Df = 25.0 \text{ mL} / 12.5 \text{ mL} = 2.0$. If no dilution is performed, $Df = 1.0$.

- 22.1.2 When target compounds are below contract required quantitation limits (CRQL) but the spectra meet the identification criteria, report the concentration with a "J". For example, if the CRQL is 1.0 ug/L and a concentration of

0.5 ug/L is calculated, report as "0.5 J". Report ALL sample concentration data as UNCORRECTED for blanks.

22.1.3 Xylenes (o-, m-, and p- isomers) are to be reported as xylenes (total). Because m- and p-xylene isomers coelute on capillary columns, special attention must be given to the quantitation of the xylenes. The relative response factor (RRF) is based on the peak that represents the single isomer on the GC column using o-xylene on capillary columns. In quantitating sample concentrations, use the areas on both 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.

22.1.4 The stereoisomers, trans-1,2-dichloroethene and cis-1,2-dichloroethene, are to be reported separately.

22.2 Non-Target Compounds

Equation D.7 is also used for calculating non-target compound concentrations. Total area counts (or peak heights) from the total ion chromatograms (RIC) are to be used for both the non-target compound to be measured (A_x) and the internal standard (A_{IS}). Associate the nearest internal standard free of interferences with the non-target compound to be measured. A relative response factor (RRF) of 1.0 is to be assumed. The value from this quantitation shall be qualified as estimated ("J"). This estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns. Do not report any non-target compound whose concentration is less than 2.0 ug/L (peak area response is less than 40% of the peak area response of the nearest internal standard).

22.3 Surrogates

Calculate the surrogate percent recovery using Equation D.8.

$$\text{EQ. D.8 Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis.

Q_a = Quantity added to sample/blank.

22.4 Internal Standards

22.4.1 Calculate the percent area response change (%ARC) between the sample/blank analysis and the most recent continuing calibration standard analysis for each of the internal standards using Equation D.9.

$$\text{EQ. D.9 } \% \text{ ARC} = \frac{A_s - A_c}{A_c} \times 100$$

Where:

% ARC = Percent area response change.

A_s = Area response of the internal standard (EICP) in the sample/blank analysis.

A_c = Area response of the internal standard (EICP) in the most recent continuing calibration standard.

- 22.4.2 Calculate the retention time shift (RTS) between the sample/blank analysis and the most recent continuing calibration standard analysis for each of the internal standards using Equation D.10.

$$\text{EQ. D.10 } \text{RTS} = \text{RT}_s - \text{RT}_c$$

Where:

RTS = Retention time shift.

RT_s = Retention time of the internal standard in a sample/blank.

RT_c = Retention time of the internal standard in the most recent continuing calibration standard.

23. TECHNICAL ACCEPTANCE CRITERIA FOR SAMPLE ANALYSIS

- 23.1 The sample must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration, and blank technical acceptance criteria.
- 23.2 The sample must be analyzed within the contract holding times. The sample must have an LCS associated with it meeting the LCS technical acceptance criteria. The sample must have a PES associated with it meeting the PES technical acceptance criteria.
- 23.3 The surrogate compound percent recovery must be between 80 and 120 percent inclusive.
- 23.4 The difference of the area response between the sample and the most recent continuing calibration standard analysis for each of the internal standards must be within the inclusive range of ± 40.0 percent of the continuing calibration standard.
- 23.5 The retention time shift between the sample and the most recent continuing calibration standard analysis for each of the internal standards must be within ± 0.33 minutes (20.0 seconds).

- 23.6 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 19.
- 23.7 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 ug/L, or saturated ions from a compound (excluding the compound peaks in the solvent front), the Contractor must either:
- 23.7.1 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 26.4), or
- 23.7.2 Monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample and that exceeded the limits above. The maximum contamination criteria are as follows: the sample must not contain a concentration above the CRQL for the target compounds or above 2 ug/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 also must meet the maximum contamination criteria.

24. CORRECTIVE ACTION

- 24.1 If the technical acceptance criteria for the internal standards and surrogate compound are not met, check calculations, internal standard solutions and instrument performance. It may be necessary to bake-out the system to remove the water from the purge and trap transfer lines of the trap, to recalibrate the instrument, or take other corrective action procedures to meet the technical acceptance criteria.
- 24.2 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 (excluding the compound peaks in the solvent front), contact SMO. SMO will contact the Region for instructions.
- 24.3 All samples to be reported must meet the maximum contamination criteria in Section 23.7. 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 contamination

criteria or an instrument blank has been analyzed that meets the technical acceptance criteria for blanks.

- 24.4 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 reanalysis at no additional cost.
- 24.5 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 SMO.
- 25. [This paragraph has been intentionally left blank and has been reserved.]

SECTION V

SAMPLE QUALITY CONTROL PROCEDURES AND REQUIREMENTS

26. BLANK ANALYSIS

26.1 Summary

There are three different types of blanks required by this method.

26.1.1 Method Blank - 25 mL of reagent water spiked with 10.0 uL internal standard and 10.0 uL surrogate solution, and carried through the entire analytical scheme. The method blank is analyzed immediately following the continuing calibration standard and before any samples are analyzed. The method blank must be analyzed immediately after the initial calibration sequence if samples are analyzed before the 12 hour time period expires. The method blank measures reagent and system contamination.

26.1.2 Storage Blank - Upon receipt of the first samples from a Sample Delivery Group, two-40 mL screw cap VOA vials with a PTFE-faced silicon septum are filled with reagent water (80 mL total). The vials are stored under the same conditions as the samples in the Sample Delivery Group. A 25.0 mL aliquot of this reagent water is spiked with a 10.0 uL internal standard and 10.0 uL of surrogate solution and analyzed after all samples in the Sample Delivery Group have been analyzed. The storage blank indicates whether contamination may have occurred during storage of samples.

26.1.3 Instrument Blank - 25 mL of reagent water spiked with 10.0 uL of internal and 10.0 uL of surrogate solution carried through the entire analytical scheme. Instrument blanks are analyzed after a sample/dilution which contains a target compound at a concentration greater than 25 ug/L (ketones 125 ug/L) or a non-target compound at a concentration greater than 100 ug/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.

26.2 Frequency

26.2.1 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 LCS, PES and storage blanks from an SDG are analyzed.

26.2.2 Storage blanks must be analyzed once per Sample Delivery Group, after all of the samples have been analyzed.

26.2.3 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 (25 ug/L for non-ketones, 125 ug/L for ketones) or non-target compounds at concentrations greater than 100 ug/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 injection port if an autosampler is used) or a sample that meets the maximum contamination criteria in Section 23.7 must be analyzed. For these purposes, if the instrument blank meets the technical acceptance criteria for blank analysis or the sample meets the maximum contamination criteria, the system is considered to be uncontaminated. If the instrument blank or sample does not meet the criteria (i.e., is contaminated), the system must be decontaminated. Until an instrument blank meets the blank technical acceptance criteria or a sample meets the maximum contamination criteria (Section 23.7), any samples analyzed since the original contaminated sample will require reanalysis at no additional expense.

26.3 Procedure

Prepare and analyze the blanks and calculate results according to Sections 17 through 22.

26.4 Technical Acceptance Criteria For Blank Analysis

- 26.4.1 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, and continuing calibration technical acceptance criteria and at the frequency described in Section 26.2.
- 26.4.2 The storage blank must be analyzed on a GC/MS system that also meets the technical acceptance criteria for the method blank.
- 26.4.3 Surrogate compound recovery in the blank must be between 80 and 120 percent inclusive.
- 26.4.4 The difference of the area response between the blank and the most recent continuing calibration standard analysis for each of the internal standards must be within the inclusive range of ± 40.0 percent of the response in the continuing calibration standard.
- 26.4.5 The retention time shift between the blank and the most recent continuing calibration standard analysis for each of the internal standards must be less than or equal to ± 0.33 minutes (20.0 seconds).
- 26.4.6 The concentration of the target compounds in the blank must be less than the CRQL for each target compound. The

concentration of non-target compounds in the blank must be less than 2.0 ug/L.

26.5 Corrective Action

26.5.1 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 Paragraph 26.4.6, 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.

26.5.2 Any method blank or instrument blank that fails to meet the technical acceptance criteria must be reanalyzed at no additional cost. Further, all samples processed within the same 12-hour time period with a method blank or instrument blank that does not meet the blank technical acceptance criteria will require reanalysis at no additional cost. Note: Storage blank data must be retained by the Contractor and be made available for inspection during the on-site laboratory evaluation.

27. LABORATORY CONTROL SAMPLES (LCS)

27.1 Summary

The LCS is an internal laboratory quality control sample designed to assess (on an SDG-by-SDG basis) the capability of the contractor to perform the analytical method listed in this Exhibit.

27.2 Frequency

The LCS must be prepared, analyzed, and reported once per Sample Delivery Group. The LCS must be prepared and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples in the SDG.

27.3 Procedure

27.3.1 Prepare the LCS using the procedure described in Section 17. Spike 25 mL of reagent water with 10.0 uL of LCS spiking solution (see Paragraph 7.4.5), 10.0 uL of internal standard, and 10.0 uL of surrogate solution.

27.3.2 Analyze the LCS as described in Sections 17 to 22.

27.4 Calculations

- 27.4.1 Calculate individual compound recoveries of the LCS using Equation D.8, substituting LCS percent recovery for surrogate percent recovery.
- 27.4.2 See Section 22 for equations necessary for other calculations.

27.5 Technical Acceptance Criteria For Laboratory Control Sample Analysis

- 27.5.1 The LCS must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration, and blank technical acceptance criteria at the frequency described in Section 27.2.
- 27.5.2 The LCS must be prepared as described in Paragraph 27.3.
- 27.5.3 The LCS must be prepared and analyzed with a method blank that meets the blank technical acceptance criteria.
- 27.5.4 Surrogate compound recovery in the LCS must be between 80 and 120 percent inclusive.
- 27.5.5 The area response change between the LCS and the most recent continuing calibration standard analysis for each of the internal standards must be within the inclusive range of ± 40.0 percent.
- 27.5.6 The retention time shift between the LCS and the most recent continuing calibration standard analysis for each of the internal standards must be within ± 0.33 minutes (20.0 seconds).
- 27.5.7 The percent recovery for each of the compounds in the LCS must be within the recovery limits listed in Table D-6.

TABLE D-6

Compound	Percent Recovery Limits
Vinyl chloride	60-140
1,2-Dichloroethane	60-140
Carbon tetrachloride	60-140
1,2-Dichloropropane	60-140
Trichloroethene	60-140
1,1,2-Trichloroethane	60-140
Benzene	60-140
cis-1,3-Dichloropropene	60-140
Bromoform	60-140
Tetrachloroethene	60-140
1,2-Dibromoethane	60-140
1,4-Dichlorobenzene	60-140

NOTE: The recovery limits for any of the compounds listed above may be expanded at any time during the period of performance if SMO determines that the limits are too restrictive.

27.6 Corrective Action

- 27.6.1 If the LCS technical acceptance criteria for the internal standards and surrogate are not met, check calculations; internal standard and surrogate solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the internal standard and surrogate criteria.
- 27.6.2 The laboratory may not submit data from an SDG until the LCS technical acceptance criteria are met. LCS contamination from laboratory sources or any LCS not meeting the criteria will require reanalysis of the LCS at no additional cost.
- 27.6.3 Further, all samples in the SDG prepared and analyzed with an LCS that does not meet the LCS technical acceptance criteria will also require reanalysis at no additional cost. Any LCS failing to meet these technical acceptance criteria must be reanalyzed at no additional cost.

28. PERFORMANCE EVALUATION SAMPLE (PES)

28.1 Summary

The PES is an external laboratory quality control sample prepared and designed to assess (on an SDG-by-SDG basis) the capability of the Contractor to perform the analytical method listed in this Exhibit.

28.2 Frequency

The Contractor must analyze and report the PES once per SDG, if available. The PES must be prepared and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples in the SDG.

28.3 Procedure

- 28.3.1 The PES will be received either as an ampulated extract or as a full volume sample. If received as an ampulated extract, the Contractor will receive instructions concerning the dilution procedure to bring the extract to full volume prior to preparation and analysis of the PES.
- 28.3.2 Prepare the PES for analysis using the procedure described in Section 17. Add 10.0 uL of internal standard and 10.0 uL of surrogate solution to the aqueous PES. Analyze the PES as described in Sections 17 to 22.

28.4 Calculations

See Section 22 for equations necessary for calculations.

28.5 Technical Acceptance Criteria for the PES

- 28.5.1 The PES must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration, and blanks technical acceptance criteria at a frequency described in Section 28.2.
- 28.5.2 The PES must be prepared according to the procedure in Section 28.3.
- 28.5.3 The surrogate compound percent recovery in the PES must be between 80 and 120 percent inclusive.
- 28.5.4 The difference of the area response between the PES and the most recent continuing calibration standard analysis for each of the internal standards must be within the inclusive range of ± 40.0 percent.
- 28.5.5 The retention time shift between the PES and the most recent continuing calibration standard analysis for each of the internal standards must be within ± 0.33 minutes (20.0 seconds).

28.6 Corrective Action

- 28.6.1 If the PES technical acceptance criteria for the internal standards and the surrogate are not met, check calculations, standard solutions and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the internal standard criteria. Any PES failing to meet these technical acceptance criteria must be reanalyzed at no additional cost. If insufficient PES spiking extract remains or if insufficient full volume PES remains, document this in the SDG Narrative by stating that the PES could not be reanalyzed because insufficient volume remains.
- 28.6.2 In addition to complying with the PES technical acceptance criteria, the Contractor will be responsible for correctly identifying and quantifying the compounds included in the PES. SMO will notify the Contractor of unacceptable performance.

Note: Unacceptable performance for identification and quantitation of compounds is defined as a score less than 75 percent.

- 28.6.3 The PES technical acceptance criteria MUST be met before sample data are reported. Also, the Contractor must demonstrate acceptable performance for compound identification and quantitation.

TABLE D-3
VOLATILE TARGET COMPOUNDS AND SURROGATE WITH CORRESPONDING
INTERNAL STANDARDS FOR QUANTITATION

1,4-DIFLUOROBENZENE	CHLOROBENZENE-d5
Acetone	Benzene
Bromochloromethane	Bromodichloromethane
Bromomethane	Carbon tetrachloride
2-Butanone	Chlorobenzene
Carbon disulfide	Dibromochloromethane
Chloroethane	1,2-Dibromoethane
Chloroform	1,2-Dichloropropane
Chloromethane	cis-1,3-Dichloropropene
1,1-Dichloroethane	trans-1,3-Dichloropropene
1,2-Dichloroethane	Ethylbenzene
1,1-Dichloroethene	2-Hexanone
cis-1,2-Dichloroethene	4-Methyl-2-pentanone
trans-1,2-Dichloroethene	Styrene
Methylene chloride	1,1,2,2-Tetrachloroethane
Vinyl chloride	Tetrachloroethene
	Toluene
4-Bromofluorobenzene	1,1,1-Trichloroethane
	1,1,2-Trichloroethane
	Trichloroethene
	Xylenes (total)
1,4-DICHLOROBENZENE-d4	
Bromoform	
1,2-Dibromo-3-chloropropane	
1,2-Dichlorobenzene	
1,3-Dichlorobenzene	
1,4-Dichlorobenzene	

TABLE D-4
PRIMARY QUANTITATION IONS (M/Z) AND SECONDARY
IONS FOR VOLATILE ORGANIC COMPOUNDS

Volatile Target Compounds	Primary Quantitation Ion	Secondary Ions
Acetone	43	58
Benzene	78	-
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
2-Butanone	43	72*
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77,114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Dibromochloromethane	129	127
1,2-Dibromo-3-chloropropane	75	155,157
1,2-Dibromoethane	107	109,188
1,2-Dichlorobenzene	146	111,148
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
cis-1,3-Dichloropropene	75	77
trans-1,3-Dichloropropene	75	77
Ethylbenzene	91	106
2-Hexanone	43	58,57,100
Methylene chloride	84	86,49
4-Methyl-2-pentanone	43	58,100
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	166	168,129
Toluene	91	92
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	97	83,85,99,132,134
Trichloroethene	95	130,132
Vinyl chloride	62	64
Xylenes (total)	106	91

SURROGATE COMPOUND AND INTERNAL STANDARDS:

4-Bromofluorobenzene	174	95,176
Chlorobenzene-d ₅	117	82,119
1,4-Dichlorobenzene-d ₄	152	115,150
1,4-Difluorobenzene	114	63,88

* Quantitation of this analyte is based on m/z 43 but m/z 72 must be present in the spectrum.

TABLE D-5
TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION FOR VOLATILE ORGANIC COMPOUNDS

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Benzene	0.500	30.0	±30.0
Bromochloromethane	0.05	30.0	±30.0
Bromodichloromethane	0.200	30.0	±30.0
Bromoform	0.05	30.0	±30.0
Bromomethane	0.100	30.0	±30.0
Carbon tetrachloride	0.100	30.0	±30.0
Chlorobenzene	0.500	30.0	±30.0
Chloroform	0.200	30.0	±30.0
Dibromochloromethane	0.100	30.0	±30.0
1,2-Dibromoethane	0.100	30.0	±30.0
1,2-Dichlorobenzene	0.400	30.0	±30.0
1,3-Dichlorobenzene	0.600	30.0	±30.0
1,4-Dichlorobenzene	0.500	30.0	±30.0
1,1-Dichloroethane	0.200	30.0	±30.0
1,2-Dichloroethane	0.100	30.0	±30.0
1,1-Dichloroethene	0.100	30.0	±30.0
cis-1,3-Dichloropropene	0.200	30.0	±30.0
trans-1,3-Dichloropropene	0.100	30.0	±30.0
Ethylbenzene	0.100	30.0	±30.0
Styrene	0.300	30.0	±30.0
1,1,2,2-Tetrachloroethane	0.100	30.0	±30.0
Tetrachloroethene	0.200	30.0	±30.0
Toluene	0.400	30.0	±30.0
1,1,1-Trichloroethane	0.100	30.0	±30.0
1,1,2-Trichloroethane	0.100	30.0	±30.0
Trichloroethene	0.300	30.0	±30.0
Vinyl chloride	0.100	30.0	±30.0
Xylenes (total)	0.300	30.0	±30.0
4-Bromofluorobenzene	0.200	30.0	±30.0

The following compounds have no maximum %RSD or maximum % Difference criteria, but must meet a minimum RRF criterion of 0.010:

Carbon disulfide	trans-1,2-Dichloroethene
Chloroethane	1,2-Dichloropropane
Chloromethane	Methylene Chloride
cis-1,2-Dichloroethene	

Note: At the present time, the Agency has not set minimum RRF or %RSD criteria for Acetone, 2-Butanone, 1,2-Dibromo-3-chloropropane, 2-Hexanone and 4-Methyl-2-pentanone.

EXHIBIT D

METHOD FOR THE ANALYSIS OF LOW CONCENTRATION WATER FOR SEMIVOLATILE ORGANIC
COMPOUNDS

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SECTION I INTRODUCTION

The analytical method that follows is designed to analyze 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 sources around Superfund sites. The method is based upon the semivolatile method contained in the CLP Statement Of Work, "Organic Analysis, Multi-Media, Multi-Concentration by GC/MS and GC/EC Techniques", except that a single extraction at a pH 2.0 is used. Incorporated in the method are specific requirements to minimize contamination of the samples from laboratory sources.

Problems have been associated with the following compounds covered by this method:

- o Dichlorobenzidine and 4-chloroaniline may be subject to oxidative losses during solvent concentration.
- o Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition.
- o N-nitrosodiphenylamine decomposes in the gas chromatographic inlet forming diphenylamine and, consequently, may be detected as diphenylamine.
- o Due to the lower quantitation limits required by this method, extra caution must be exercised when identifying compounds.

SECTION II

PART A - SAMPLE/SAMPLE EXTRACT STORAGE AND HOLDING TIMES

1. PROCEDURES FOR SAMPLE STORAGE

The samples must be protected from light and refrigerated at 4°C ($\pm 2^\circ\text{C}$) from the time of receipt until 60 days after delivery of a complete reconciled data package. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

Samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

Samples, sample extracts, and standards must be stored separately.

2. PROCEDURE FOR SAMPLE EXTRACT STORAGE

Sample extracts must be protected from light and stored at 4°C ($\pm 2^\circ\text{C}$) until 365 days after delivery of a complete data package.

Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

Samples, sample extracts, and standards must be stored separately.

3. CONTRACT REQUIRED HOLDING TIMES

The extraction shall be started within 5 days of the Validated Time of Sample Receipt (VTSR).

Extracts must be analyzed within 40 days following the start of the extraction.

PART B - EQUIPMENT AND STANDARDS

4. SUMMARY OF METHOD

- 4.1 A one liter aliquot of sample is acidified to pH 2.0 and extracted with methylene chloride using a continuous liquid-liquid extractor. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL. The extract is injected onto a gas chromatograph (GC) capillary column. The gas chromatograph is temperature programmed to separate the semivolatile compounds, which are then detected with a mass spectrometer (MS).
- 4.2 Target and surrogate compounds are identified in the samples by analyzing standards under the same conditions used for samples and comparing resultant mass spectra and GC retention times. Internal standards are added to all samples and standards. A response factor is established for each target and surrogate compound during the initial and continuing calibrations by comparing the MS response for the primary ion produced by the compound extracted ion current profile (EICP) to the MS response for the primary ion produced by an internal standard. Each identified target and surrogate compound in a sample is quantified by comparing the responses for the target compound and the internal standard, while taking into account the response factor from the most recent calibration, the sample volume, and any sample dilutions.
- 4.3 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology Mass Spectral Library. Non-target compounds are quantified by comparing the MS response from the reconstructed ion chromatogram (RIC) for the non-target compound peaks to the MS response produced by the nearest internal standard. A response factor of 1 is assumed.

5. 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 current (RIC) profiles (TICPs). 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.

6. APPARATUS AND MATERIALS

Brand names, suppliers and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of the method is the responsibility of the Contractor.

6.1 Gas Chromatograph/Mass Spectrometer

- 6.1.1 Gas Chromatograph - The gas chromatograph (GC) 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.

Gas Chromatography Column - A 30 m x 0.25 mm ID (or 0.32 mm) bonded-phase silicone coated fused silica capillary column (J&W Scientific DB-5 or equivalent). A film thickness of 0.25 to 1.0 μ m may be used.

- 6.1.2 Mass Spectrometer - The mass spectrometer must be capable of scanning from 35 to 500 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 tuning acceptance criteria when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the gas chromatograph 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.
- 6.1.3 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 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 most recent release of the National Institute of Standards and Technology Mass Spectral Library shall be used as the reference library.

- 6.2 Magnetic Tape Storage Device - The magnetic tape storage device must be capable of recording data and suitable for long-term, off-line storage.

- 6.3 Glassware: A set of glassware sufficient to meet contract requirements must be reserved for exclusive use in this contract.
- 6.3.1 Continuous liquid-liquid extractors - Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor-Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent.)
 - 6.3.2 Drying column - 19 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.)
 - 6.3.3 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes, Vineland, NJ, K-570050-1025 or equivalent).
 - 6.3.4 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001 - 0500 or equivalent). Attach to concentrator tube with springs.
 - 6.3.5 Snyder column - Kuderna-Danish, Three-ball macro (Kontes K-50300-0121 or equivalent).
 - 6.3.6 Snyder column - Kuderna-Danish, Two-ball micro (Kontes K-569001-0219 or equivalent).
 - 6.3.7 Vials - Amber glass, 2 mL capacity with Teflon-lined screw-cap.
 - 6.3.8 Syringes - 0.2 mL, 0.5 mL, and 5 mL volumes.
- 6.4 Gases - Helium, Nitrogen, ultra pure grade.
- 6.5 Gas-line tubing - stainless steel, or copper tubing.
- 6.6 Silicon carbide boiling chips - approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.
- 6.7 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.
- 6.8 Balance - Analytical, capable of accurately weighing ± 0.0001 g. The balances must be calibrated with class S weights once per each 12-hour workshift. The balances must also be annually checked by a certified technician.
- 6.9 Nitrogen evaporation device equipped with a water bath that can be maintained at 30°C to 35°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.

6.10 Micro-syringes - 10 uL and larger, 0.006 inch ID needle.

6.11 pH meter - Calibrate according to manufacturer's instructions. pH meter must be calibrated before each use.

7. REAGENTS

7.1 Reagent water - Reagent water is defined as water in which no semivolatile target compound is observed at or above the CRQL listed in Exhibit C for that compound and in which no non-target compound is observed at or above 10 ug/L.

7.1.1 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).

7.1.2 Reagent water may be generated using a water purification system (Millipore Super-Q or equivalent).

7.2 Solvents - Acetone, methanol, methylene chloride. Pesticide quality or equivalent.

7.3 Sodium sulfate - (ACS) Granular, anhydrous (J.T. Baker anhydrous powder, catalog #73898, 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.

7.4 Sulfuric acid solution (1:1) - slowly add 50 mL of concentrated H₂SO₄ (Sp. Gr. 1.84) to 50 mL of reagent water.

8. STANDARDS

8.1 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.

8.2 Stock Standard Solutions

Stock standard solutions may be purchased or prepared using the following procedure.

8.2.1 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.

8.2.2 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.

8.2.3 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.

8.3 Secondary Dilution Standards

8.3.1 Using stock standards, prepare secondary dilution standards in methylene chloride that contain the compounds of interest either singly or mixed together.

8.3.2 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.

8.4 Working Standards

8.4.1 Tuning Solution - Decafluorotriphenylphosphine (DFTPP)

Prepare a 50 ng/uL solution of DFTPP in methylene chloride. The DFTPP solution must be prepared fresh once every twelve months, or sooner, if the solution has degraded or concentrated.

8.4.2 Initial and Continuing Calibration Solutions

8.4.2.1 Five initial calibration standard solutions are required for all target and surrogate compounds. Standard concentrations of 5, 10, 20, 50, and 80 ng/uL are required for five of the surrogates and all but eight of the target compounds. Nine compounds: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol and pentachlorophenol, 2,4,6-tribromophenol (surrogate), require calibration at 20, 50, 80, 100, and 120 ng/uL.

8.4.2.2 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.

- 8.4.2.3 Add 10.0 uL of internal standard spiking solution (Paragraph 8.6.1) to 1.0 mL of each calibration standard for a concentration of 20 ng/uL for each internal standard.
- 8.4.2.4 The 20 ng/uL initial calibration solution (80 ng/uL for the nine compounds listed in 8.4.2.1) is the continuing calibration solution.
- 8.4.2.5 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.

8.5 Surrogate Standard Spiking Solution

- 8.5.1 Prepare a surrogate standard spiking solution in methanol that contains, 2,4,6-tribromophenol (an acid surrogate compound), at a concentration of 120 ug/mL. The other acid surrogate compounds: phenol-d₆ and 2-fluorophenol, and the base/neutral compounds: nitrobenzene-d₅, terphenyl-d₁₄, and 2-fluorobiphenyl are at a concentration of 40 ug/mL.
- 8.5.2 The surrogate standard spiking solution must be prepared every twelve months, or sooner, if the solution has degraded or concentrated.

8.6 Internal Standard Spiking Solution

- 8.6.1 Prepare an internal standard spiking solution in methylene chloride or another suitable solvent that contains 1,4-dichlorobenzene-d₄, naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂, and perylene-d₁₂ at 2000 ng/uL. It may be necessary to use 5 to 10 percent benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents.
- 8.6.2 The internal standard spiking solution must be prepared every six months, or sooner, if the solution has degraded or concentrated.

8.7 Laboratory Control Sample (LCS) Spiking Solution

- 8.7.1 Prepare a laboratory control sample spiking solution that contains each of the compounds at the concentrations listed below in methanol.

<u>Compounds</u>	<u>Concentration</u> <u>(ug/mL)</u>
Phenol	40.0
2-Chlorophenol	40.0
4-Chloroaniline	40.0
2,4,6-Trichlorophenol	40.0
bis(2-Chloroethyl)ether	20.0
N-Nitroso-di-n-propylamine	20.0
Hexachloroethane	20.0
Isophorone	20.0
1,2,4-Trichlorobenzene	20.0
Naphthalene	20.0
2,4-Dinitrotoluene	20.0
Diethylphthalate	20.0
N-Nitrosodiphenylamine	20.0
Hexachlorobenzene	20.0
Benzo(a)pyrene	20.0

8.7.2 The laboratory control sample solution must be prepared every twelve months, or sooner, if the solution has degraded or concentrated.

8.8 Storage of Standard Solutions

- 8.8.1 Store the stock and secondary standard solutions at -10°C to -20°C in Teflon-lined screw-cap amber bottles.
- 8.8.2 Store the working standard solutions at 4°C ($\pm 2^\circ\text{C}$) in Teflon-lined screw-cap amber bottles.
- 8.8.3 Protect all standards from light.
- 8.8.4 Samples, sample extracts, and standards must be stored separately.

SECTION III

INSTRUMENT QUALITY CONTROL PROCEDURES AND REQUIREMENTS

PART A - INSTRUMENT OPERATING CONDITIONS

Gas Chromatograph

The following are the recommended gas chromatographic analytical conditions.

Carrier Gas:	Helium
Linear Velocity:	25-30 cm/sec
Injector Temperature:	250-300°C
Injector:	Grob-type, splitless
Initial Temperature:	40°C
Initial Hold Time:	4.0 \pm 0.1 min
Ramp Rate:	10°C/min
Final Temperature:	290°C
Final Hold Time:	10 min or until all compounds of interest have eluted.

Optimize GC conditions for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, performance evaluation samples, and laboratory control samples.

Mass Spectrometer

The following are the required mass spectrometer conditions:

Transfer Line Temperature:	250-300°C
Source Temperature:	According to manufacturer's specifications.
Electron Energy:	70 volts (nominal)
Ionization Mode:	EI
Mass Range:	35 to 500 amu
Scan Time:	At least 5 scans per peak, not to exceed 1 second per scan.

PART B - TUNING THE MASS SPECTROMETER

9. SUMMARY

It is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria prior to initiating any on-going data collection. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP).

10. FREQUENCY

Each GC/MS system used must be hardware tuned once per twelve (12) hour time period of operation. Also, whenever the Contractor takes corrective action which may change or affect the tuning criteria for DFTPP (e.g., ion source cleaning or repair, column replacement, etc.), the tune must be verified irrespective of the twelve-hour tuning requirement. The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the Contractor submits as documentation of a compliant tune. The time period ends after twelve (12) hours have elapsed. In order to meet tuning requirements, samples, PES, LCS, blanks, and standards must be injected within twelve hours of the DFTPP injection.

11. PROCEDURE

- 11.1 Inject 50 ng of DFTPP into the GC/MS system. All instrument conditions must be identical to those listed in Section III, Part A, except that a different temperature program may be used.
- 11.2 DFTPP may be analyzed separately or as part of the calibration standard.

12. TECHNICAL ACCEPTANCE CRITERIA FOR DFTPP ANALYSIS

- 12.1 The GC/MS system must be tuned at the frequency described in Section 10.
- 12.2 The abundance criteria listed in Table D-7 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 prior to the elution of DFTPP. Note: All subsequent standards samples, including LCS, PES, and blanks, must use identical mass spectrometer instrument conditions.

Table D-7

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

<u>Mass</u>	<u>Ion Abundance Criteria</u>
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 abundance MUST be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

12.3 The criteria above are based on adherence to the acquisition specifications identified in paragraph 12.2. The criteria are based on performance characteristics of instruments currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for 51 target compounds (see Table D-10), are designed to control and monitor instrument performance associated with the requirements of this method.

13. CORRECTIVE ACTION

13.1 If the DFTPP acceptance criteria are not met, retune the GC/MS system. It may be necessary to clean the ion source, clean quadrupoles, or take other actions to achieve the acceptance criteria.

13.2 DFTPP acceptance criteria MUST be met before any standards, samples (including LCS and PES), or required blanks are analyzed. Any samples or required blanks analyzed when tuning criteria have not been met will require reanalysis at no additional cost.

14. [This paragraph has been intentionally left blank and has been reserved.]

PART C - CALIBRATION OF THE GC/MS SYSTEM

15. INITIAL CALIBRATION

15.1 Summary - Prior to the analysis of samples and required blanks and after tuning criteria have been met, each GC/MS system must be initially calibrated at a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

15.2 Frequency

15.2.1 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.

15.2.2 If time still remains in the 12 hour time period after meeting the technical acceptance criteria for the initial calibration, samples 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. Quantify all sample and quality control sample results and quality control criteria results, such as internal standard area response change and retention time shift, against the initial calibration standard that is the same concentration as the continuing calibration standard.

15.3 Procedure

15.3.1 Set-up the GC/MS system per the requirements of Section III, Part A.

15.3.2 All standard/spiking solutions and blanks must be allowed to warm to ambient temperature (approximately 1 hour) before preparation or analysis.

15.3.3 Prepare five calibration standards containing all the semivolatile target and surrogate compounds at the concentrations described in Paragraph 8.4.2.

15.3.4 Prepare an internal standard spiking solution using the procedure described in Section 8.6.

15.3.5 Tune the GC/MS system to meet the technical acceptance criteria in Section 12 for DFTPP.

15.3.6 Analyze each calibration standard by injecting 1.0 uL of standard. If a compound saturates when the 80 ng/uL standard is injected (120 ng/uL for the nine compounds listed in

Paragraph 8.4.2), and the GC/MS system is calibrated to achieve a detection sensitivity of no less than the CRQL for each compound, the Contractor must document it in the SDG Narrative, and attach a quantitation report and RIC. In this instance, the Contractor must calculate the results based on a four-point initial calibration for the specific compound that saturates. Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion. If secondary ion quantitation is used, calculate a relative response factor using the area response from the most intense secondary ion which is free of interferences, and document the reasons for the use of the secondary ion in the SDG Narrative.

15.4 Calculations

NOTE: Unless otherwise stated the area response is that of the primary quantitation ion.

- 15.4.1 Calculate relative response factors (RRF) for each semivolatile target and surrogate compounds using Equation D.11. See Table D-8 to associate semivolatile target and surrogate compounds with the proper internal standard. See Table D-9 for primary quantitation ions to be used for each semivolatile target compound, surrogate, and internal standard.

$$\text{EQ. D.11} \quad \text{RRF} = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the primary quantitation ion response (EICP) for the compound to be measured.

A_{is} = Area of the primary quantitation ion response (EICP) for the internal standard.

C_{is} = Concentration of the internal standard.

C_x = Concentration of the compound to be measured.

- 15.4.2 Equation D.12 is the general formula for standard deviation (SD) for a statistically small set of values.

$$\text{EQ. D.12} \quad \text{SD} = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}$$

Where:

SD = Standard deviation for set of values.

x_i = Value.

\bar{x} = Mean value.

n = Number of values.

- 15.4.3 Calculate the percent relative standard deviation (%RSD) of RRF values for each semivolatile target and surrogate compound over the initial calibration range using Equation D.13 in conjunction with Equation D.12.

EQ. D.13

$$\%RSD = \frac{SD_{RRF}}{\bar{x}} \times 100$$

Where:

%RSD = Percent relative standard deviation.

SD_{RRF} = Standard deviation of initial calibration response factors (per compound).

From EQ. D.12

x_i = RRF = Relative response factors from initial calibration standard (per compound).

\bar{x} = \overline{RRF} = Mean value of initial calibration response factors (per compound).

- 15.4.4 Equation D.14 is the general formula for the mean of a set of values.

EQ. D.14

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$$

x_i = Value.

\bar{x} = Mean value.

n = Number of values.

- 15.4.5 Calculate the mean of the relative retention times (\overline{RRT}) for each semivolatile target and surrogate compound over the initial calibration range using Equation D.14 and Equation D.15.

EQ. D.15

$$RRT = \frac{RT_c}{RT_{is}}$$

RT_c - Retention time for the semivolatile target and surrogate compound.

RT_{is} - Retention time for the internal standard.

From EQ. D.14:

$x_i = RRT_i$ - Relative retention times for the semivolatile target or surrogate compound for each initial calibration standard.

$\bar{x} = \overline{RRT}$ - Mean relative retention time.

- 15.4.6 Calculate the area response (\bar{Y}) mean for each internal standard compound over the initial calibration range using Equation D.14.

Where:

$x_i = Y$ - Area responses of the primary quantitation ion (EICP) for the internal standard for each initial calibration standard.

$\bar{x} = \bar{Y}$ - Area response mean.

- 15.4.7 Calculate the mean of the absolute retention times (\bar{RT}) for each internal standard over the initial calibration range using Equation D.14.

Where:

$x_i = RT$ - Retention time for the internal standard for each initial calibration standard.

$\bar{x} = \bar{RT}$ - Mean retention time.

n - Number of values.

15.5 Technical Acceptance Criteria For Initial Calibration

- 15.5.1 All initial calibration standards must be analyzed at the concentration levels described in paragraph 8.4.2 and at the frequency described in Section 15.2 on a GC/MS system meeting the DFTPP technical acceptance criteria.
- 15.5.2 The relative response factor (RRF) at each calibration concentration for each semivolatile target and surrogate

compound must be greater than or equal to the compound's minimum acceptable relative response factor listed in Table D-10.

- 15.5.3 The %RSD over the initial calibration range for relative response factor for each semivolatile and surrogate compound that has a required %RSD must be less than or equal to the %RSD listed in Table D-10.
- 15.5.4 Up to four compounds may fail the criteria listed in paragraph 15.5.2 and 15.5.3 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%.
- 15.5.5 The relative retention time (RRT) for each of the semivolatile target and surrogates compounds at each calibration level must be within ± 0.06 relative retention time units of the mean relative retention time (\overline{RRT}) for each compound.
- 15.5.6 The area response (Y) for each internal standard compound in each calibration standard must be within the inclusive range of -50 percent to +100 percent of the mean area response (\overline{Y}) of the internal standard in all of the calibration standards.
- 15.5.7 The retention time (RT) shift for each of the internal standards at each calibration level must be within ± 0.33 minutes (20.0 seconds) compared to the mean retention time (\overline{RT}) over the initial calibration range for each internal standard.

15.6 Corrective Action

- 15.6.1 If the technical acceptance criteria for initial calibration are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the acceptance criteria.
- 15.6.2 Initial calibration technical acceptance criteria MUST be met before any samples (including the LCS and PES) or required blanks are analyzed. Any samples or required blanks analyzed when initial calibration criteria have not been met will require reanalysis at no additional cost.

16. CONTINUING CALIBRATION

16.1 Summary

Prior to the analysis of samples and required blanks and after tuning criteria and initial calibration 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, surrogates, and internal standards.

16.2 Frequency

- 16.2.1 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.
- 16.2.2 If time still remains in the 12 hour time period after meeting the technical acceptance criteria for the initial calibration, samples 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. Quantify all sample results against the 20 ng/uL (80 ng/uL for the nine compounds listed in Section 8.4.2.1) calibration standard.

16.3 Procedure

- 16.3.1 Set up GC/MS system per the requirements of Section III, Part A.
- 16.3.2 Prepare a continuing calibration standard solution containing all the semivolatile target and surrogate compounds using the procedure listed in Section 8.4.2.
- 16.3.3 All standard/spiking solutions and blanks must be allowed to warm to ambient temperature (approximately 1 hour) before preparation or analysis.
- 16.3.4 Tune the GC/MS system to meet the DFTPP technical acceptance criteria in Section 12.
- 16.3.5 Start the analysis of the continuing calibration standard by injecting 1.0 uL of standard.

16.4 Calculations

- 16.4.1 Calculate a relative response factor (RRF) for each semivolatile target and surrogate compound using Equation D.11.
- 16.4.2 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 and surrogate compound using Equation D.17.

$$\text{EQ D.17 } \% \text{ Difference }_{\text{RRF}} = \frac{\overline{\text{RRF}}_i - \text{RRF}_c}{\overline{\text{RRF}}_i} \times 100$$

Where:

\overline{RRF}_i = Average relative response factor from the most recent initial calibration.

RRF_c = Relative response factor from continuing calibration standard.

16.5 Technical Acceptance Criteria For Continuing Calibration

- 16.5.1 The continuing calibration standard must be analyzed at the 20 ng/uL (80 ng/uL for the nine compounds listed in 8.4.2.1) concentration level at the frequency described in Section 16.2 on a GC/MS system meeting the DFTPP tuning and the initial calibration technical acceptance criteria.
- 16.5.2 The relative response factor for each semivolatile target and surrogate compound must be greater than or equal to the compound's minimum acceptable relative response factor listed in Table D-10.
- 16.5.3 The relative response factor percent difference for each semivolatile and surrogate compound that has a percent difference criteria must be within the inclusive range listed in Table D-10.
- 16.5.4 Up to four compounds may fail the requirements listed in paragraph 16.5.2 and 16.5.3 and still meet the minimum response factor and percent difference criteria. However, these compounds must have a minimum response factor greater than or equal to 0.010 and the percent difference must be within the inclusive range of $\pm 40\%$.

16.6 Corrective Action

- 16.6.1 If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 15. It may be necessary to clean the ion source, change the column or take other corrective actions to achieve the acceptance criteria.
- 16.6.2 Continuing calibration technical acceptance criteria MUST be met before any samples (including the LCS and PES) or required blanks are analyzed. Any samples or required blanks analyzed when continuing calibration criteria have not been met will require reanalysis at no additional cost.

SECTION IV

SAMPLE PREPARATION, ANALYSIS, AND COMPOUND IDENTIFICATION AND QUANTITATION

17. SUMMARY

This method is designed for analysis of 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 sources and well/ground water around Superfund sites. If, upon inspection of a sample, the Contractor suspects that the sample is not amenable to this method, contact SMO for instructions.

18. PROCEDURE

18.1 Extraction

Continuous liquid-liquid extraction is required for the extraction of the samples.

- 18.1.1 Add methylene chloride to the bottom of the extractor and fill it to a depth of at least 1 inch above the bottom side arm.
- 18.1.2 Using a 1 liter graduated cylinder, measure out a 1.0 liter sample aliquot. Transfer the 1 liter sample aliquot to the continuous extractor. Pipet 1.0 mL of surrogate standard spiking solution into the sample and mix well. Check the pH of the sample with a pH meter and adjust the pH to 2.0 with 1:1 H₂SO₄.
- 18.1.3 Add sufficient methylene chloride to the distilling flask to ensure proper solvent cycling during operation. Extract for 18 hours. Allow to cool, then detach the distilling flask and label.
- 18.1.4 If the sample was received in a 1 liter container, rinse the empty container with 60 mL of methylene chloride after taking the sample aliquot. Add the rinsate to the continuous extractor.

18.2 Concentrating the Extracts

- 18.2.1 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.
- 18.2.2 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.
- 18.2.3 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60°C

to 80°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 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. 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.

- 18.2.4 Two different types of concentration techniques are permitted to obtain the final 1.0 mL volume: micro Snyder column and nitrogen evaporation techniques.

18.2.4.1 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) 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 to dryness. Remove the Snyder column and rinse its 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 Teflon-sealed screw-cap bottle, label the bottle and store at 4°C ($\pm 2^\circ\text{C}$).

18.2.4.2 Nitrogen Evaporation Technique (taken from ASTM Method D3086)

Place the concentrator tube with an open micro Snyder column attached 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 Teflon tubing. 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 Teflon-sealed screw-cap bottle, label the bottle and store at 4°C ($\pm 2^\circ\text{C}$).

18.3 Instrumental Analysis

- 18.3.1 Set up the GC/MS system per the requirements of Section III, Part A. Before samples or required blanks can be analyzed, the instrument must meet the DFTPP, initial calibration, and continuing calibration technical acceptance criteria. All samples, required blanks, extracts, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before preparation/analysis. All sample extracts and required blanks must be analyzed under the same instrumental conditions as the calibration standards.
- 18.3.2 Add 10.0 μL of the internal standard spiking solution (2000 ng/ μL) to the 1.0 mL extract. For sample dilutions, add an appropriate amount of the internal standard spiking solution to maintain a 20 ng/ μL concentration of the internal standards in the diluted extract.
- 18.3.3 Inject 1.0 μL of sample extract into the GC/MS, and start data acquisition.
- 18.3.4 When all semivolatile target compounds have eluted from the GC, terminate the MS data acquisition and store data files on the data system storage device. Use appropriate data output software to display full range mass spectra and EICPs.

19. DILUTIONS

- 19.1 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 compound saturate the detector (excluding the compound peaks in the solvent front), the extract must be diluted and reanalyzed. Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion, 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.

19.2 Dilute the sample using the following procedure:

19.2.1 Calculate the sample dilution necessary to keep the semivolatile target compounds that required dilution within the upper half of the initial calibration range and so that no compound has ions which saturate the detector (excluding the compound peaks in the solvent front). Note: Do not dilute sample solely to get 2,4,6 tribromophenol value within the initial calibration range.

19.2.2 Dilute the sample extract with methylene chloride in a volumetric flask.

19.2.3 Analyze the sample dilution per Section 18.3, including the addition of internal standards to maintain a 20 ng/uL concentration of the internal standards (see 18.3.2).

20. IDENTIFICATION OF TARGET COMPOUNDS

20.1 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: (1) elution of the sample compound at the same GC relative retention time as the standard compound, and (2) correspondence of the sample compound and standard compound mass spectra.

20.2 For establishing correspondence of the GC relative retention time (RRT), the sample compound RRT must be within ± 0.06 RRT units of the RRT of the standard compound. For reference, the standard must be run on the same shift as the sample. If co-elution of interfering compounds prohibits accurate assignment of the sample compound RRT from the extracted ion current profile for the primary ion, the RRT must be assigned by using the total ion chromatogram.

20.3 For comparison of standard and sample compound mass spectra, mass spectra obtained on the Contractor's GC/MS are required. These standard spectra may be used for identification purposes, only if the Contractor's GC/MS meets the DFTPP daily tuning technical acceptance criteria. These standard spectra may be obtained from the analysis used to obtain reference relative retention times.

20.4 The guidelines for qualitative verification by comparison of mass spectra are as follows:

20.4.1 All ions present in the standard mass spectra at a relative intensity greater than 25 percent (most abundant ion in the spectrum equals 100 percent) must be present in the sample spectrum.

20.4.2 The relative intensities of the major ions specified in Section 20.4.1 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.)

20.4.3 Ions greater than 25 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should FAVOR FALSE POSITIVES. All compounds meeting the identification criteria must be reported with their spectra.

20.4.4 If a compound cannot be verified by all of the criteria in Sections 20.4.1 - 20.4.3, 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.

21. IDENTIFICATION OF NON-TARGET COMPOUNDS

21.1 A library search shall be executed for non-target compounds for the purpose of tentative identification. The most recent release of the National Institute of Standards and Technology Mass Spectral Library shall be used as the reference library.

21.2 Up to 20 non-surrogate/non-internal standard organic compounds of greatest apparent concentration not listed in Exhibit C for the semivolatiles shall be tentatively identified via a forward search of the National Institute of Standards and Technology Mass Spectral Library. (Compounds with a peak area response less than 50 percent of the area response for the nearest internal standard and compounds which elute before the first semivolatile target compound elutes are not required to be searched in this fashion.) Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search must not use normalization routines if those routines would misrepresent the library or unknown spectra when compared to each other.

21.3 Guidelines for making tentative identification

21.3.1 Relative intensities of major ions in the reference spectrum (ions greater than 25 percent of the most abundant ion) should be present in the sample spectrum.

21.3.2 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.)

21.3.3 Molecular ions present in reference spectrum should be present in sample spectrum.

21.3.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

- 21.3.5 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.
- 21.3.6 If in the technical judgment of the mass interpretation spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If a probable molecular weight can be distinguished, include it.

22. CALCULATIONS

NOTE: Unless otherwise stated, the area response is from the extracted ion current profile (EICP) of the primary quantitation ion.

22.1 Target Compounds

- 22.1.1 Calculate target compound concentrations using Equation D.18.

$$\text{EQ. D.18} \quad \text{Concentration in ug/L} = \frac{(A_x)(I_s)(V_t)(D_f)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where:

- A_x - Area response (EICP) for the compound to be measured. The primary quantitation ions for the target compounds, internal standards, and surrogates are listed in Table D-9.
- A_{is} - Area response (EICP) for the internal standard. The target compounds are listed with their associated internal standard in Table D-8.
- I_s - Amount of internal standard injected in nanograms (ng).
- RRF - The RRF from the most recent continuing calibration as determined in Section 15.4.1.
- V_o - Volume of water extracted in milliliters (mL).
- V_i - Volume of extract injected in microliters (uL).
- V_t - Volume of final extract in microliters (uL).
- D_f - Dilution Factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

uL most conc. extract used to make dilution + uL clean solvent
uL most conc. extract used to make dilution

If no dilution is performed, $D_f = 1.0$.

- 22.1.2 When target compounds are below contract required quantitation limits (CRQL), but the spectra meet the identification criteria, report the concentration with a "J". For example, if the CRQL is 5 ug/L and a concentration of 3.0 ug/L is calculated, report as "3.0 J". Report ALL sample concentration data as UNCORRECTED for blanks.

22.2 Non-Target Compounds

Equation D.18 is used for calculating the concentrations of the non-target compounds. Total area counts (or peak heights) from the reconstructed ion chromatograms (RIC) are to be used for both the non-target compound to be measured (A_x) and the internal standard (A_{is}). Associate the nearest internal standard free of interferences with the non-target compound to be measured. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated ("J"). This estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns.

22.3 Surrogates

Calculate the surrogate percent recovery using Equation D.19.

$$\text{EQ. D.19} \quad \text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis.

Q_a = Quantity added to sample/blank.

22.4 Internal Standards

- 22.4.1 Calculate the percent area response change (% ARC) for the sample/blank analysis compared to the most recent continuing calibration standard analysis for each of the internal standard compounds using Equation D.20.

$$\text{EQ. D.20} \quad \% \text{ ARC} = \frac{A_s - A_c}{A_c} \times 100$$

Where:

% ARC = Percent area response change.

A_s = Area response of the internal standard in the sample/blank analysis.

A_c = Area response of the internal standard in the most recent continuing calibration standard.

22.4.2 Calculate the retention time shift (RTS) between the sample/blank analysis and the most recent continuing calibration standard analysis for each of the internal standards using Equation D.21.

$$\text{EQ. D.21 } \text{RTS} = \text{RT}_s - \text{RT}_c$$

Where:

RTS = Retention time shift.

RT_s = Retention time of the internal standard in a sample/blank.

RT_c = Retention time of the internal standard in the most recent continuing calibration standard.

23. TECHNICAL ACCEPTANCE CRITERIA FOR SAMPLE ANALYSIS

- 23.1 The sample must be analyzed on a GC/MS system meeting the DFTPP, initial calibration, and continuing calibration technical acceptance criteria.
- 23.2 The sample must be extracted and analyzed within the contract holding times.
- 23.3 The sample must have an associated method blank meeting the blank technical acceptance criteria. The sample must have a Laboratory Control Sample associated with it meeting the LCS technical acceptance criteria. The sample must also have a PES associated with it meeting the PES technical acceptance criteria.
- 23.4 The percent recovery for each of the surrogates in the sample must be within the acceptance windows listed in Table D-11.
- 23.5 The difference of the area response change for each of the internal standards for the sample must be within the inclusive range of -50 percent and +100 percent of the response of the internal standards in the most recent continuing calibration analysis.
- 23.6 The retention time shift for each of the internal standards must be within ± 0.33 minutes (20.0 seconds) between the sample and the most recent continuing calibration standard analysis.

- 23.7 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 19.

Table D-11

CONTRACT REQUIRED SURROGATE SPIKE RECOVERY LIMITS

Surrogate Compound	% Recovery
Nitrobenzene-d ₅	40-112
2-Fluorobiphenyl	42-110
Terphenyl-d ₁₄	24-140
Phenol-d ₅	17-113
2-Fluorophenol	16-110
2,4,6-Tribromophenol	18-126

24. CORRECTIVE ACTION

- 24.1 If the sample technical acceptance criteria for the surrogates and internal standards are not met, check calculations, surrogate and internal standard solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and internal standard technical acceptance criteria.
- 24.2 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 SMO. SMO will contact the Region for instructions.
- 24.3 Sample analysis technical acceptance criteria MUST be met before data are reported. Samples contaminated from laboratory sources, or associated with a contaminated method blank or any samples analyzed not meeting the technical acceptance criteria will require reextraction and/or reanalysis at no additional cost.
- 24.4 Sample reruns performed as a result of suspected matrix interferences beyond the scope of the method will be reviewed on a case-by-case basis for payment purposes by SMO.
25. [This paragraph has been intentionally left blank and has been reserved.]

SECTION V

SAMPLE QUALITY CONTROL PROCEDURES AND REQUIREMENTS

26. BLANK ANALYSIS

26.1 Summary - A method blank is 1.0 liter of reagent water carried through the entire analytical scheme.

26.2 Frequency

26.2.1 A method blank must be extracted at least once:

- o every 20 samples, AND
- o whenever samples are extracted.

26.2.2 Each method blank must be analyzed on each GC/MS used to analyze the samples prepared with the method blank.

26.3 Procedure for Method Blank Preparation

26.3.1 Prepare the method blank at the frequency listed in Section 26.2. Measure out 1.0 liter of reagent water for each method blank aliquot. Extract and concentrate the method blank at the same time as the samples associated with the blank according to Sections 18.1.1 and 18.2.

26.3.2 Analyze the method blank and calculate the results according to Sections 18-22.

26.4 Technical Acceptance Criteria For Blank Analysis

26.4.1 All blanks must be analyzed at the frequency described in Section 26.2 on a GC/MS system meeting the DFTPP, initial calibration, and continuing calibration technical acceptance criteria.

26.4.2 The percent recovery for each of the surrogates in the blank must be within the acceptance windows listed in Table D-11.

26.4.3 The area response change for each of the internal standards for the blank must be within the inclusive range of -50 percent and +100 percent compared to the internal standards in the most recent continuing calibration analysis.

26.4.4 The retention time shift for each of the internal standards must be within ± 0.33 minutes (20.0 seconds) between the blank and the most recent continuing calibration analysis.

26.4.5 The concentration of the target compounds in the blanks must be less than or equal to the CRQL for each target compound. The concentration of non-target compounds in the blanks must not exceed 10 ug/L.

26.5 Corrective Action

26.5.1 If a Contractor's blanks do not meet the technical acceptance criteria, the Contractor must consider the analytical system to

be out of control. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds.

- 26.5.2 Any method blank that fails to meet the technical acceptance criteria must be reextracted and reanalyzed at no additional cost. Further, all samples processed with a method blank that is out of control (i.e., contaminated) will require reextraction and reanalysis at no additional cost.

27. LABORATORY CONTROL SAMPLES (LCS)

27.1 Summary

The LCS is an internal laboratory quality control sample designed to assess (on an SDG-by-SDG basis) the capability of the contractor to perform the analytical method listed in this Exhibit.

27.2 Frequency

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 instrumentation as the samples in the SDG.

27.3 Procedure

- 27.3.1 Measure a 1.0 liter aliquot of reagent water in a 1 liter graduated cylinder and transfer the water to a continuous extractor. Pipet 1.0 mL of the LCS spiking solution (Paragraph 8.7.1) and 1.0 mL of the surrogate standard spiking solution into the water and mix well. Extract and concentrate the sample according to Sections 18.1 and 18.2.

- 27.3.2 Analyze the LCS per Sections 18-21.

27.4 Calculations

- 27.4.1 Calculate individual compound recoveries of the LCS using Equation D.19, substituting LCS percent recovery for surrogate percent recovery.

- 27.4.2 See Section 22 for equations necessary for other calculations.

27.5 Technical Acceptance Criteria For Laboratory Control Sample Analysis

- 27.5.1 The LCS must be analyzed at the frequency described in Section 27.2 on a GC/MS system meeting the DFTPP, initial calibration, and continuing calibration technical acceptance criteria.
- 27.5.2 The LCS must be prepared as described in Paragraph 27.3.
- 27.5.3 The LCS must be prepared and analyzed with a method blank that met the blank technical acceptance criteria.
- 27.5.4 The percent recovery for each of the surrogates in the LCS must be within the acceptance windows listed in Table D-11.
- 27.5.6 The area response change for each internal standard for the LCS must be within the inclusive range of -50 percent and +100 percent compared to the internal standard in the most recent continuing calibration analysis.
- 27.5.7 The retention time shift for each of the internal standards within ± 0.33 minutes (20.0 seconds) between the LCS and the continuing calibration standard analysis.
- 27.5.8 The percent recovery for each of the compounds in the LCS must be within the recovery limits listed in Table D-12.

Table D-12

LABORATORY CONTROL SAMPLE PERCENT RECOVERY LIMITS

COMPOUND	% RECOVERY
Phenol	44 - 120
2-Chlorophenol	58 - 110
4-Chloroaniline	35 - 98
2,4,6-Trichlorophenol	65 - 110
bis(2-Chloroethyl)ether	64 - 110
N-Nitroso-di-n-propylamine	34 - 102
Hexachloroethane	32 - 77
Isophorone	49 - 110
1,2,4-Trichlorobenzene	44 - 96
Naphthalene	56 - 160
2,4-Dinitrotoluene	61 - 140
Diethylphthalate	76 - 104
N-Nitrosodiphenylamine	35 - 120
Hexachlorobenzene	30 - 95
Benzo(a)pyrene	55 - 92

NOTE: The recovery limits for any of the compounds in the LCS may be expanded at any time during the period of performance if SMO determines that the limits are too restrictive.

27.6 Corrective Action

- 27.6.1 If the LCS technical acceptance criteria for the surrogates and internal standards are not met, check calculations, surrogate and internal standard solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and internal standard criteria.
- 27.6.2 The laboratory may not submit data from an SDG until the LCS technical acceptance criteria are met. LCS contamination from laboratory sources or any LCS not meeting the criteria will require reanalysis and reextraction of the LCS at no additional cost.
- 27.6.3 Further, all samples in the SDG prepared and analyzed with an LCS that does not meet the LCS technical acceptance criteria will also require reanalysis at no additional cost. Any LCS failing to meet these technical acceptance criteria must be reanalyzed and reextracted at no additional cost.

28. PERFORMANCE EVALUATION SAMPLE (PES)

28.1 Summary

The PES is an external laboratory quality control sample prepared and designed to assess (on an SDG-by-SDG basis) the capability of the Contractor to perform the analytical method listed in this Exhibit.

28.2 Frequency

The Contractor must extract, analyze, and report the results of the PE sample once per SDG, if available. The PES must be extracted and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples in the SDG.

28.3 Procedure

- 28.3.1 The PES will be received either as an ampulated extract or as a full volume sample. If received as an ampulated extract, the Contractor will receive instructions concerning the dilution procedure to bring the extract to full volume prior to preparation and analysis of the PES.
- 28.3.2 Extract and concentrate the PES using the procedure described in Section 18. Add 1.0 mL of surrogate solution to 1 liter of reagent water spiked with the PES solution. Analyze the PES as described in Sections 18 to 21.

28.4 Calculations

See Section 22 for equations necessary for calculations.

28.5 Technical Acceptance Criteria for the PES

- 28.5.1 The PES must be analyzed on a GC/MS system meeting the DFTPP tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in Section 28.2.
- 28.5.2 The PES must be extracted and concentrated according to Section 28.3.
- 28.5.3 The PES must be prepared and analyzed with a method blank that met the blank technical acceptance criteria.
- 28.5.4 The percent recovery for each of the surrogates in the PES must be within the acceptance windows listed in Table D-11.
- 28.5.5 The area response change between the PES and the most recent continuing calibration standard analysis for each of the internal standards must be within the inclusive range of -50 percent and +100 percent.
- 28.5.6 The retention time shift between the PES and the most recent continuing calibration standard analysis for each of the internal standards must be within ± 0.33 minutes (20.0 seconds).

28.6 Corrective Action

- 28.6.1 If the PES technical acceptance criteria for the internal standards and the surrogate are not met, check calculations, standard solutions and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the internal standard criteria. Any PES failing to meet these technical acceptance criteria must be reextracted and reanalyzed at no additional cost. If insufficient PES spiking extract remains or if insufficient full volume PES remains, document this in the SDG Narrative by stating that the PES could not be reextracted and reanalyzed because insufficient volume remained.
- 28.6.2 In addition to complying with the PES technical acceptance criteria, the Contractor will be responsible for correctly identifying and quantitating the compounds included in the PES. SMO will notify the Contractor of unacceptable performance.

Note: Unacceptable performance for identification and quantitation of compounds is defined as a score less than 75 percent.

- 28.6.3 The PES technical acceptance criteria MUST be met before sample data are reported. Also, the Contractor must demonstrate acceptable performance for compound identification and quantitation.

TABLE D-8

SEMIVOLATILE INTERNAL STANDARDS WITH
CORRESPONDING TARGET ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀	Phenanthrene-d ₁₀	Chrysene-d ₁₂	Perylene-d ₁₂
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octylphthalate
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-Nitrosodiphenylamine	Butylbenzylphthalate	Benzo(b)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenyl phenyl ether	3,3-Dichlorobenzidine	Benzo(k)fluoranthene
2-Methylphenol	2,4-Dimethylphenol	2-Chloronaphthalene	Hexachlorobenzene	Benzo(a)Anthracene	Benzo(a)pyrene
bis(2-Chloroisopropyl)ether	bis(2-Chloroethoxy)methane	2-Nitroaniline	Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)Pyrene
4-Methylphenol	2,4-Dichlorophenol	Dimethyl phthalate	Phenanthrene	Chrysene	Dibenz(a,h)anthracene
N-Nitroso-di-n-propylamine	4-Chloroaniline	Acenaphthylene	Anthracene	Terphenyl-d ₁₄ (surr)	Benzo(g,h,i)perylene
2-Fluorophenol (surr)	Hexachlorobutadiene	3-Nitroaniline	Di-n-Butyl phthalate		
Phenol-d ₆ (surr)	4-Chloro-3-methylphenol	Acenaphthene	Fluoranthene		
	2-Methylnaphthalene	2,4-Dinitrophenol			
	Nitrobenzene-d ₅ (surr)	4-Nitrophenol			
		Dibenzofuran			
		2,4-Dinitrotoluene			
		2,6-Dinitrotoluene			
		Diethylphthalate			
		4-Chlorophenyl phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			

Surr = surrogate compound

SV D-39

Table D-9

Primary Quantitation Ions (m/z) and Secondary Ions
for Semivolatile Organic Compounds

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
2,2'-oxybis(1-Chloropropane)	45	77, 79
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
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
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
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

Table D-9 (continued)

Parameter	Primary Ion	Secondary Ion(s)
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
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
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
Surrogates		
Phenol-d ₅	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene d ₅	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
Internal Standards		
1,4-Dichlorobenzene-d ₄	152	115
Naphthalene-d ₈	136	68
Acenaphthene-d ₁₀	164	162, 160
Phenanthrene-d ₁₀	188	94, 80
Chrysene-d ₁₂	240	120, 236
Perylene-d ₁₂	264	260, 265

TABLE D-10
ACCEPTANCE CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION FOR SEMIVOLATILE ORGANIC COMPOUNDS

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Phenol	0.800	20.5	±25.0
bis(2-Chloroethyl)ether	0.700	20.5	±25.0
2-Chlorophenol	0.700	20.5	±25.0
2-Methylphenol	0.700	20.5	±25.0
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
1,2,4-Trichlorobenzene	0.200	20.5	±25.0
Naphthalene	0.700	20.5	±25.0
4-Chloro-3-methylphenol	0.200	20.5	±25.0
2-Methylnaphthalene	0.400	20.5	±25.0
2,4,6-Trichlorophenol	0.200	20.5	±25.0
2,4,5-Trichlorophenol	0.200	20.5	±25.0
2-Chloronaphthalene	0.800	20.5	±25.0
Acenaphthylene	1.300	20.5	±25.0
Acenaphthene	0.800	20.5	±25.0
Dibenzofuran	0.800	20.5	±25.0
2,4-Dinitrotoluene	0.200	30.0	±30.0
2,6-Dinitrotoluene	0.200	20.5	±25.0
4-Chlorophenyl-phenylether	0.400	20.5	±25.0
Fluorene	0.900	20.5	±25.0
4-Bromophenyl-phenylether	0.100	20.5	±25.0
Hexachlorobenzene	0.100	20.5	±25.0
Pentachlorophenol	0.050	20.5	±25.0
Phenanthrene	0.700	20.5	±25.0
Anthracene	0.700	20.5	±25.0
Fluoranthene	0.600	20.5	±25.0
Pyrene	0.600	20.5	±25.0
Benzo(a)anthracene	0.800	20.5	±25.0
Chrysene	0.700	20.5	±25.0
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

(continued)

TABLE D-10
ACCEPTANCE CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION FOR SEMIVOLATILE ORGANIC COMPOUNDS

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Phenol-d ₅	0.800	20.5	±25.0
2-Fluorophenol	0.600	20.5	±25.0
Terphenyl-d ₁₄	0.500	20.5	±25.0
2-Fluorobiphenyl	0.700	20.5	±25.0

The following compounds have no maximum %RSD or maximum %D criteria, but must meet a minimum RRF criterion of 0.010:

2,2'-oxybis(1-Chloropropane)	4-Nitroaniline
4-Chloroaniline	4,6-Dinitro-2-methylphenol
Hexachlorobutadiene	N-Nitrosodiphenylamine
Hexachlorocyclopentadiene	Di-n-butylphthalate
2-Nitroaniline	Butylbenzylphthalate
Dimethylphthalate	3,3'-Dichlorobenzidine
3-Nitroaniline	bis(2-Ethylhexyl)phthalate
2,4-Dinitrophenol	Di-n-octylphthalate
4-Nitrophenol	2,4,6-Tribromophenol
Diethylphthalate	Nitrobenzene-d ₅

EXHIBIT D

**METHOD FOR THE ANALYSIS OF LOW CONCENTRATION WATER FOR
PESTICIDES AND AROCLORS**

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SECTION I

INTRODUCTION

The analytical method that follows is designed to analyze water in order to determine the presence and concentration of the chlorinated pesticides and Aroclors found in the Target Compound List (Exhibit C). The majority of the samples are expected to be from drinking water and well/ground water 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. Quality control requirements are incorporated in the method in order to maximize GC/ECD sensitivity and column resolution and to minimize contamination of the samples from laboratory sources.

Resolution difficulties have been associated with the following pairs of compounds using this method:

- o On a DB-608 or equivalent column, DDE and dieldrin; Methoxychlor and Endrin ketone; and Endosulfan I and gamma-Chlordane.
- o On a DB-1701 or equivalent column, Endosulfan I and gamma-Chlordane; and Methoxychlor and Endosulfan sulfate.

SECTION II

PART A - SAMPLE STORAGE AND HOLDING TIMES.

1. PROCEDURES FOR SAMPLE STORAGE

The samples must be protected from light and refrigerated at 4°C ($\pm 2^\circ\text{C}$) from the time of receipt until 60 days after delivery of a complete reconciled sample data package. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

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 samples received under this contract.

Samples, sample extracts, and standards must be stored separately.

2. PROCEDURE FOR SAMPLE EXTRACT STORAGE

Sample extracts must be protected from light and stored at 4°C ($\pm 2^\circ\text{C}$) until 365 days after delivery of a complete data package.

Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

Samples, sample extracts, and standards must be stored separately.

3. CONTRACT REQUIRED HOLDING TIMES

The extraction of all samples must be started within 5 days of the Validated Time of Sample Receipt (VTSR).

Analysis of samples must be completed within 40 days following the start of extraction.

PART B - EQUIPMENT AND STANDARDS

3. SUMMARY OF METHOD

- 3.1 A one liter aliquot of sample is spiked with the surrogate solution and extracted with methylene chloride by using a continuous liquid-liquid extractor. 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 mL.
- 3.2 The hexane extract is injected onto two wide-bore capillary columns in a gas chromatograph (GC). The gas chromatograph 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 both GC columns.
- 3.3 A single component pesticide is identified if a peak is detected within its appropriate retention time 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 concentrations during the initial calibration.
- 3.4 Toxaphene and Aroclors are identified primarily by pattern recognition, but retention times 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.

4. INTERFERENCES

- 4.1 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 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 Section 12 must be used to remove such interferences in order to achieve the Contract Required Quantitation Limits (CRQL).

5. APPARATUS AND MATERIALS

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of method is the responsibility of the Contractor.

5.1 Gas Chromatograph/Electron Capture Detector (GC/ECD)

5.1.1 Gas Chromatograph

5.1.1.1 The gas chromatograph (GC) system must regulate temperature in order to give a reproducible temperature program 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.

5.1.1.2 Gas chromatographs 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 any 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.

5.1.2 Gas Chromatograph Columns

5.1.2.1 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 DB-1701, 30 m x 0.53 mm ID, 1.0 µm film thickness, J&W Scientific, Folsom, CA, and a DB-608, 30 m x 0.53 mm ID, 0.5 to 1.0 µm film thickness from J&W Scientific. Equivalent columns may be employed if they meet the requirements in Section III.

5.1.2.2 Columns are mounted in 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.
Laboratories should follow the manufacturer's recommendation on mounting 0.53 mm capillaries into injection ports.

- 5.1.3 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 SMO. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of a hydrogen leak detector is highly recommended if hydrogen is used as a carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used.
- 5.1.4 Electron Capture Detector - The makeup gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. The GC/ECD system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants which may interfere with the analysis. The instrument must be vented to outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.
- 5.1.5 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.
- 5.2 Glassware - A set of glassware sufficient to meet contract requirements must be reserved for exclusive use in this contract.
 - 5.2.1 Continuous liquid-liquid extractors equipped with Teflon or glass connecting lines for use with methylene chloride (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10, or equivalent).
 - 5.2.2 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes, Vineland, NJ K-570050-1025, or equivalent).
 - 5.2.3 Evaporative flask - Kuderna-Danish, 500 mL (Kontes K-570001 - 0500, or equivalent). Attach to concentrator tube with springs.
 - 5.2.4 Snyder column - Kuderna-Danish, three-ball macro (Kontes K-50300-0121, or equivalent).

- 5.2.5 Drying column, chromatographic column approximately 400 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.)
- 5.2.6 Pipet, Volumetric 1.00 mL or 2.00 mL (optional).
- 5.2.7 Microsyringe, 1.0 uL and larger, 0.006 inch ID needle.
- 5.2.8 Syringe, 1.00 mL or 2.00 mL (optional).
- 5.2.9 Flask, Volumetric 10.00 mL.
- 5.2.10 Flask, Volumetric 1.00 mL or 2.00 mL (optional).
- 5.2.11 Snyder column, micro two or three ball with a 19 mm ground glass joint.
- 5.2.12 Glass vials, minimum of 20 mL, with screw cap and Teflon or aluminum foil liner.
- 5.2.13 Vials, 10 mL, with screw cap and Teflon liner (optional).
- 5.2.14 Vials and caps, 1 or 2 mL for GC auto sampler.
- 5.2.15 Bottle or test tube, 50 mL with Teflon-lined screw cap for sulfur removal.
- 5.2.16 Centrifuge tubes, calibrated, 12 mL, for sulfur removal.
- 5.2.17 Micropipet, 250 uL, with disposable tips.
- 5.3 pH Paper, wide range.
- 5.4 Boiling chips.
 - 5.4.1 Silicon carbide boiling chips, approximately 10 to 40 mesh. Heat the chips to 400°C for 30 minutes or solvent rinse before use.
 - 5.4.2 Teflon boiling chips (optional). Solvent rinse the chips before use.
- 5.5 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.
- 5.6 Balance. Analytical, capable of accurately weighing ± 0.0001 g. The balances must be calibrated with class S weights once per each 12-hour workshift. The balances must also be annually checked by a certified technician.

- 5.7 Nitrogen evaporation device equipped with a heated bath that can be maintained at 30 to 35°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.
- 5.8 Florisil Cleanup Equipment
- 5.8.1 Florisil bonded silica. 1-g cartridges with stainless steel or Teflon frits, Catalog No. 694-313 (Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent).
- 5.8.2 Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold, Analytichem International (J.T. Baker, or Supelco) or equivalent.
- 5.8.3 Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.
- 5.8.4 Vacuum pressure gauge.
- 5.8.5 Rack for holding 10 mL volumetric flasks in the manifold.
- 5.9 Mechanical shaker or mixer, for sulfur removal.

6. REAGENTS

- 6.1 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 GC/ECD to demonstrate that it is free of interference before use (J. T. Baker anhydrous granular, Catalog No. 3375, or equivalent).
- 6.2 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 it is used. 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.
- 6.3 Mercury, triple distilled, for sulfur clean-up.
- 6.4 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.
- 6.5 Sodium hydroxide solution (10 N). Carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 mL.
- 6.6 Concentrated sulfuric acid, (Sp. Gr. 1.84).

- 6.7 Nitric acid, dilute, for sulfur removal with copper.
- 6.8 Reagent water. Reagent water is defined as a water in which no target analyte is observed at the CRQL for that compound.
- 6.8.1 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).
- 6.8.2 Reagent water may be generated using a water purification system (Millipore Super-Q or equivalent).
- 6.9 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. STANDARDS

- 7.1 The Contractor must provide all standards to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E. 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 Stock standard solutions (1.00 ug/uL) - Stock standard solutions may be purchased as certified solutions or prepared from pure standard materials.
- 7.2.1 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.
- 7.2.2 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.
- 7.2.3 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 working standards from them.

7.3 Working Standards

7.3.1 Surrogate Standard Spiking Solution

The surrogates, tetrachloro-m-xylene and decachlorobiphenyl, are added to all standards, samples, and blanks. Prepare a surrogate spiking solution of 0.20 ug/mL of each of the two compounds in acetone. The solution should be checked frequently for stability. The solution must be replaced after six months or at an earlier time, if the solution has degraded or concentrated.

7.3.2 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.

<u>Compounds</u>	<u>Concentration (ng/mL)</u>
gamma-Chlordane	10.0
Endosulfan I	10.0
p,p'-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

7.3.3 Performance Evaluation Mixture

The Performance Evaluation Mixture is prepared in hexane or iso-octane as listed below. The PEM must be prepared weekly or more often, if the solution has degraded or concentrated.

<u>Compounds</u>	<u>Concentration (ng/mL)</u>
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

7.3.4 Single Component Pesticides

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 6 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 epoxida	5.0
Dieldrin	10.0	alpha-Chlordane	5.0
Endrin	10.0	gamma-Chlordane	5.0
p,p'-DDD	10.0	p,p'-DDE	10.0
p,p'-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
		Decachlorobiphenyl	10.0

7.3.5 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 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.

7.3.6 Florisil Cartridge Check Solution

Prepare a 0.10 ug/mL solution of 2,4,5-trichlorophenol in acetone. The solution must be prepared every 6 months, or sooner, if the solution has degraded or concentrated.

7.3.7 Laboratory Control Sample (LCS) Spiking Solution

Prepare a laboratory control sample (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.

<u>Compounds</u>	<u>Concentration (ug/mL)</u>
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

7.4 Storage of Standards

- 7.4.1 Store the stock and secondary standard solutions at -10°C to -20°C in Teflon-lined screw-cap amber bottles.
- 7.4.2 Store the working standard solutions at 4°C ($\pm 2^\circ\text{C}$) in Teflon-lined screw-cap amber bottles. The working standards must be checked frequently for signs of degradation or evaporation.
- 7.4.3 Protect all standards from light.
- 7.4.4 Samples, sample extracts, and standards must be stored separately.

SECTION III

INSTRUMENT QUALITY CONTROL PROCEDURES AND REQUIREMENTS

PART A - INSTRUMENT OPERATING CONDITIONS

8. GAS CHROMATOGRAPH / ELECTRON CAPTURE DETECTOR

The following are the gas chromatographic analytical conditions. The conditions are recommended unless otherwise noted.

Carrier Gas:	Helium
Column Flow:	5 mL/min
Make-up Gas:	Argon/Mathane (P-5 or P-10) or N ₂ (required)
Injector Temperature:	≥ 200°C (required)
Injection Technique:	On-column
Injection Volume:	1 or 2 ul (see note below)
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	1/2 min
Temperature Ramp:	5°C to 6°C/min
Final Temperature:	275°C
Final Hold Time:	After Decachlorobiphenyl has eluted (approximately 10 minutes)

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, performance evaluation samples and laboratory control samples. NOTE: Manual injections must be 2.0 uL. Auto injectors may use 1.0 uL volumes. The same injection volume must be used for all standards, blanks, and samples.

The linearity of the 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.

Cold (ambient temperature) on-column injectors that allow injection directly onto a 0.53 mm ID column may be used as long as the acceptance criteria for resolution, calibration, and analyte breakdown are met.

PART B - CALIBRATION OF THE GC/ECD SYSTEM

9. INITIAL CALIBRATION

9.1 Summary

Prior to sample analysis, each GC/ECD system must be initially calibrated at a minimum of three concentrations for single component analytes in order to determine instrument sensitivity and the linearity of GC response. Each multicomponent analyte is analyzed at one concentration.

9.2 Frequency

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.

9.3 Procedure

- 9.3.1 Set up the GC/ECD system as described in Section 8.
- 9.3.2 Prepare the initial calibration standards using the procedures, the analytes, and the concentrations according to Section 7.
- 9.3.3 All standards, samples, and blanks must be allowed to warm to ambient temperature before preparation or analysis.
- 9.3.4 Analyze the initial calibration sequence as given below. NOTE: Steps 16 and 17 are used as part of the calibration verification as well (see Section 10).

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

9.4 Calculations

- 9.4.1 For each single component pesticide and surrogate, a retention time (RT) is measured in each of three of the calibration standards analyses (low point, midpoint, high point) during the initial calibration for Individual Standard Mixture A and Individual Standard Mixture B. The RT for the surrogates is measured from the Individual Standard Mixture A analyses. The mean RT is calculated as the average of the three values. Calculate a mean absolute retention time (RT) for each single component pesticide and surrogate using Equation D.22.

$$\text{EQ. D.22. } \overline{\text{RT}} = \sum_{i=1}^n \frac{\text{RT}_i}{n}$$

$\overline{\text{RT}}$ - Mean absolute retention time of analyte.

RT_i - Absolute retention time of analyte.

n - Number of measurements (3).

- 9.4.2 A retention time 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.13. Windows are centered around the average absolute retention time for the analyte established during the initial calibration.

TABLE D.13

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
DDD	±0.07
DDE	±0.07
DDT	±0.07

TABLE D.13 (continued)

RETENTION TIME WINDOWS FOR SINGLE AND MULTICOMPONENT ANALYTES AND SURROGATES.

Compound	Compound Identification Window (minutes)
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

- 9.4.3 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 is permitted to calculate linearity for endrin based on peak area and to calculate linearity for aldrin based on peak height. It is not permitted within a %RSD calculation for an analyte to use calibration factors calculated from both peak area and peak height. For example, it is not 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.

9.4.3.1 Calculate the calibration factor for each single component pesticide and surrogate over the initial calibration range using Equation D.23.

9.4.3.2 Calculate the mean and the %RSD of the calibration factors for each single component pesticide and surrogate over the initial calibration range using Equation D.24 and Equation D.25.

$$\text{EQ. D.23 } CF = \frac{\text{Peak Area (or Height) of the Standard}}{\text{Mass Injected (ng)}}$$

$$\text{EQ. D.24 } CF = \frac{\sum_{i=1}^n CF_i}{n}$$

$$\text{EQ. D.25 } \% \text{ RSD} = \frac{SD_{CF}}{\overline{CF}} \times 100$$

$$\text{Where } SD_{CF} = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n-1}} \quad \text{and } n=3$$

%RSD = Percent relative standard deviation.

SD_{CF} = Standard deviation for calibration factors.

CF_i = Calibration factor.

\overline{CF} = Mean calibration factor.

9.4.4 A calibration factor is calculated for each peak in a selected set of three to five major peaks for each multicomponent analyte using Equation D.23.

9.4.5 Calculate the percent breakdown of DDT, the percent breakdown of Endrin, and the combined breakdown of DDT and Endrin in the Performance Evaluation Mixture using Equations D.26, D.27, D.28, and D.29.

EQ. D.26

$$\text{Amount found (in ng)} = \frac{\text{Peak area (or Peak height) of compound in PEM}}{CF_{mp}}$$

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. D.27

$$\% \text{ Breakdown DDT} = \frac{\text{Amount found in ng (DDD+DDE)} * 100}{\text{Amount in ng of DDT injected}}$$

EQ. D.28

% Breakdown Endrin =

$$\frac{\text{Amount found in ng (Endrin Aldehyde + Endrin Ketone)} * 100}{\text{Amount in ng of Endrin injected}}$$

EQ. D.29 Combined % Breakdown = %Breakdown DDT + %Breakdown Endrin

9.4.6 Calculate the percent difference for each pesticide and surrogate in the Performance Evaluation Mixture and Individual Standard Mixtures A and B using Equation D.26 and Equation D.30.

$$\text{EQ. D.30} \quad \%D = \frac{|C_{nom} - C_{calc}|}{C_{nom}} \times 100$$

- %D - Percent Difference
- C_{nom} - nominal concentration of each analyte
- C_{calc} - Calculated concentration of each analyte from the analyses of the standard

- 9.4.7 Calculate the resolution between the analytes in the Resolution Check Mixture and the midpoint concentrations of individual standard mixtures A and B using Equation D.31.

$$\text{EQ. D.31} \quad \% \text{ Resolution} = \frac{V}{H} \times 100\%$$

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

9.5 Technical Acceptance Criteria For Initial Calibration

All initial calibration technical acceptance criteria apply independently to both GC columns.

- 9.5.1 The initial calibration sequence must be analyzed according to the procedure and in the order listed in Section 9.3, at the concentrations listed in Section 7, and at the frequency listed in Section 9.2. The GC/ECD operating conditions optimized in Section 8 must be followed.
- 9.5.2 The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0%. On the DB-1701 GC column, resolution difficulties are most likely between Endosulfan I and gamma-Chlordane and between Methoxychlor and endosulfan sulfate. On the DB-608 GC column, the poorest resolution will probably be between p,p'-DDE and Dieldrin; Methoxychlor and Endrin ketone; and Endosulfan I and gamma-Chlordane.
- 9.5.3 All single component pesticides and surrogates in both runs of the Performance Evaluation Mixture (PEM) must be 100 percent resolved.
- 9.5.4 The absolute retention times of each of the single component pesticides and surrogates in both runs of the PEM must be within the retention time window determined from the three-point initial calibration in paragraph 9.4.2.

- 9.5.5 The absolute value of the percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in both of the PEM runs must be less than or equal to 25.0 percent, using Equation D.30.
- 9.5.6 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.
- 9.5.7 The %RSD of the calibration factors for each single component target compound must be less than or equal to 20.0 percent, except as noted. 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 (but not surrogates) may exceed the 20.0 percent limit for %RSD, but those compounds must have a %RSD of less than or equal to 30.0 percent.
- 9.5.8 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.
- 9.5.9 All instrument blanks must meet the technical acceptance criteria in Paragraph 20.3.4.
- 9.5.10 The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The retention time 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 retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.
- 9.5.10.1 The chromatograms that result from the analyses of the Resolution Check Mixture, the Performance Evaluation Mixture, 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.
- 9.5.10.2 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.

- 9.5.10.3 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.
- 9.5.10.4 For all Resolution Check Mixtures, Performance Evaluation Mixtures, 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.
- 9.5.10.5 If a chromatogram is replotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.
- 9.5.10.6 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.

9.6 Corrective Action

- 9.6.1 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 acceptance criteria.
- 9.6.2 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.
- 9.6.3 If a laboratory decontaminates a detector using elevated temperature, the ECD electronics must be turned off during the bake out procedure.
- 9.6.4 After bake out or hydrogen reduction, the detector must be recalibrated using the initial calibration sequence.

- 9.6.5 Initial calibration technical acceptance criteria MUST be met before any samples (including the LCS and PES) or required blanks are analyzed. Any samples (including the LCS and PES) or required blanks analyzed after the initial calibration criteria have not been met will require reanalysis at no additional cost.

10. CALIBRATION VERIFICATION

10.1 Summary

Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid point concentration of Individual Standard Mixtures A and B constitute the continuing calibration. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

10.2 Frequency

- 10.2.1 An instrument blank and the Performance Evaluation Mixture must bracket one end of a 12-hour period during which sample 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.
- 10.2.2 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 that 12-hour period (see Section 13.3). Samples 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).
- 10.2.3 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 acceptance criteria in Section 10.5. 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 acceptance criteria in Section 10.5. The 12-hour time period begins with the injection of the instrument blank. Standards (PEM or Individual Standard Mixtures), samples and

required blanks may be injected for 12:00 hours from the time of injection of the instrument blank.

- 10.2.4 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(s) was injected.
- 10.2.5 After a break in sample analyses, the laboratory may only resume the analysis of samples using the current initial calibration for quantitation by analyzing an acceptable instrument blank and a PEM.
- 10.2.6 If the entire 12-hour period is not required for the analyses of all samples to be reported and all data collection is to be stopped, the incomplete 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 12-hour period.

10.3 Procedure

- 10.3.1 Set up the GC/ECD system as described in Section 8.
- 10.3.2 Prepare the PEM standard and the mid point concentration of Individual Standard Mixtures A and B as in Section 7, and the instrument blank as in Section 20.3.
- 10.3.3 The PEM, the Individual Standard Mixtures, and the instrument blank must be at ambient temperature at the time of preparation and analysis.
- 10.3.4 Analyze the PEM, instrument blank, and the mid point concentration of Individual Standard Mixtures A and B at the required frequencies (Sections 10.2 and 13.3).

10.4 Calculations

- 10.4.1 For each analysis of the Performance Evaluation Mixture used to demonstrate continuing calibration, calculate the percent difference between the amount of each analyte (including the surrogates) found in the PEM and the nominal amount, using Equations D.26 and D.30.
- 10.4.2 For each analysis of the Performance Evaluation Mixture used to demonstrate continuing calibration, calculate the percent breakdown of Endrin and DDT, and the combined breakdown, using Equations D.26, D.27, D.28, and D.29.
- 10.4.3 For each analysis of the mid point concentration of Individual Standard Mixtures A and B used to demonstrate continuing calibration, calculate the percent difference between the amount of each analyte (including the surrogates) found in the

standard mixture and the nominal amount, using Equations D.26 and D.30. 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.

10.5 Technical Acceptance Criteria For Calibration Verification

All calibration verification technical acceptance criteria apply independently to both columns.

- 10.5.1 The PEMs, Individual Standard Mixtures, and instrument blanks must be analyzed at the required frequency (paragraph 10.2), using the procedure in paragraph 10.3, on a GC/ECD system that has met the initial calibration technical acceptance criteria.
- 10.5.2 All single component pesticides and surrogates in the Performance Evaluation Mixtures used to demonstrate continuing calibration must be 100 percent resolved. 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.
- 10.5.3 The absolute retention time for each of the single component pesticides and surrogates in the PEMs and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be within the retention time windows determined from the three-point initial calibration in paragraph 9.4.2.
- 10.5.4 The absolute value of the percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEM and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be less than or equal to 25.0 percent, using Equation D.30.
- 10.5.5 The percent breakdown of DDT and endrin in the PEM must be less than or equal to 20.0 percent each on both columns. The combined breakdown of DDT and endrin must be less than or equal to 30.0 percent on both columns.
- 10.5.6 All instrument blanks must meet the technical acceptance criteria in Paragraph 20.3.4.
- 10.5.7 The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The retention time 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 retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.

- 10.5.7.1 The chromatograms that result from the analyses of the Performance Evaluation Mixture 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.
- 10.5.7.2 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.
- 10.5.7.3 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.
- 10.5.7.4 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.

10.6 Corrective Action.

- 10.6.1 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective action to achieve the acceptance criteria.
- 10.6.2 Major corrective actions such as replacing the GC column or baking out the detector will require that a new initial calibration be performed and meets the technical acceptance criteria.
- 10.6.3 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 acceptance criteria.
- 10.6.4 If a Performance Evaluation Mixture 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.

- 10.6.5 If an instrument blank does not meet the technical acceptance criteria listed in Paragraph 20.3.4, 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.
- 10.6.6 Analysts are cautioned that running an instrument blank and a Performance Evaluation Mixture 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.
- 10.6.7 If a successful instrument blank and PEM cannot be run after an interruption in analysis (Paragraph 10.2.5), an acceptable initial calibration must be run before sample data may be collected. All acceptable sample analyses must be preceded and followed by acceptable standards and instrument blanks, as described in Section 10.2.
- 10.6.8 Calibration verification technical acceptance criteria must be met before any samples (including LCS, PES) and required blanks are reported. Any samples, including LCS and PES required with a calibration verification which did not meet the technical acceptance criteria will require reanalysis at no additional cost.

SECTION IV

SAMPLE PREPARATION, EXTRACTION, AND CLEANUP

11. SUMMARY

- 11.1 This method is designed for analysis of 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 sources and well/ground water around Superfund sites. If, upon inspection of a sample, the Contractor suspects that the sample is not amenable to this method, contact SMO for instructions.

12. PROCEDURE

12.1 Extraction

Continuous Liquid-Liquid Extraction is required for the extraction of the samples.

- 12.1.1 Add methylene chloride (100 to 250 mL) to the bottom of the extractor and fill it to a depth of at least one inch above the bottom sidearm.
- 12.1.2 Measure out each 1.0 liter sample aliquot in a separate 1 liter 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. Transfer the sample aliquot into the continuous extractor.
- 12.1.3 Using a micropipet with disposable tip, add 200 uL of the surrogate solution to all samples and method blanks.
- 12.1.4 Adjust the level of methylene chloride in the extractor so that the bottom sidearm is half filled with solvent.
- 12.1.5 If the sample was received in a 1 liter container, rinse the empty container with 60 mL of methylene chloride after taking the sample aliquot. Add the rinsate to the continuous extractor.
- 12.1.6 Add sufficient methylene chloride to the distilling flask to ensure proper solvent cycling during operation. Extract the solution for 18 hours. Allow to cool, then detach the distillation flask and label.

12.2 Extract Drying and Concentration

- 12.2.1 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.

- 12.2.2 Pour the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and the sodium sulfate with at least two additional 20 to 30 mL portions of methylene chloride to complete the quantitative transfer.
- 12.2.3 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 (60-80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 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.
- 12.2.4 Proceed with the solvent exchange to hexane.
- 12.3 Solvent Exchange To Hexane
- 12.3.1 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.
- 12.3.2 Remove the Snyder column; using 1 to 2 mL of hexane, rinse the flask and its lower joint into the concentrator tube.
- 12.3.3 Use the micro Snyder column or the nitrogen blowdown technique (Section 12.5) to concentrate the hexane extract to 1.0 mL.
- 12.4 Extract Cleanup
- 12.4.1 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 GC/ECD analysis. Sulfur contamination in a sample analysis is unacceptable. Method blanks must be subjected to the same cleanup procedures as the samples.

12.4.2 Florisil Cartridge Procedure

Florisil cartridge cleanup significantly reduces matrix interferences caused by polar compounds and is required for all extracts.

12.4.2.1 Cartridge Performance Check. Every lot number of Florisil cartridges must be tested by the following procedure before they are used for sample cleanup. Add 0.50 mL of 2,4,5-trichlorophenol solution (0.1 ug/mL in acetone) and 0.50 mL of Standard Mixture A, midpoint concentration (Section 7.3.4) to 4 mL of hexane. Reduce the final volume to 1.0 mL using the extract concentration techniques in paragraph 12.5. 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. Reduce the final volume to 1.0 mL using micro Snyder column or nitrogen evaporation techniques (Section 12.5). Analyze the solution by GC/ECD. This solution must be analyzed on a GC/ECD meeting the initial and calibration verification technical acceptance criteria. The recovery of each analyte must be determined for evaluation and reporting purposes. The lot of Florisil cartridges is acceptable if all pesticides and surrogates are recovered at 80 to 120 percent, if the recovery of trichlorophenol is less than 5%, and no peaks interfering with the target analytes are detected.

12.4.2.2 Florisil cartridge cleanup procedure

12.4.2.2.1 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.

12.4.2.2.2 Place a 1 g Florisil cartridge into the vacuum manifold for each sample extract.

12.4.2.2.3 Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge in 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.

- 12.4.2.2.4 After the cartridges in 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.
- 12.4.2.2.5 Transfer the 1 mL extract to the top frit of the appropriate Florisil cartridge. Rinse the concentrator tube twice with 0.5 mL of hexane/acetone (90:10) and transfer each rinse to the top frit of the cartridge.
- 12.4.2.2.6 The pesticides/Aroclors in the extract concentrates are then eluted through the column with 8.0 mL of hexane/acetone (90:10) and are collected into the 10 mL volumetric flasks held in the rack inside the vacuum manifold.
- 12.4.2.2.7 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.
- 12.4.2.2.8 Concentrate the extract to 2.0 mL using either a micro Snyder column or nitrogen evaporation (Section 12.5). Measure the final volume with a syringe or by transferring the extract to a volumetric flask.
- 12.4.2.2.9 If crystals of sulfur are evident or if the presence of sulfur is suspected, proceed to Section 12.4.3. Sample analyses with interference caused by sulfur are not acceptable and the extracts must be cleaned up and reanalyzed.
- 12.4.2.2.10 If the extract is not contaminated with sulfur, transfer the sample to a GC vial and label the vial. The extract is ready for GC/ECD analysis. Proceed

to Section 13. Store the extracts at 4°C in the dark until analyses are completed.

12.4.3 Sulfur Removal

Sulfur can be removed by one of two methods, according to laboratory preference. Interference which is due to sulfur is not acceptable. 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.

- 12.4.3.1 If only part of a set of samples requires sulfur cleanup, then two blanks are required for that set: one that is shaken with mercury or copper, and one that is not. If only part of a set of samples requires sulfur cleanup, do not subject the associated method blank to sulfur cleanup, but prepare a separate sulfur cleanup blank as described below. If all the samples are subjected to sulfur cleanup, process the method blank along with the samples. In this case, no additional sulfur cleanup blank is required.

Sulfur cleanup blank. Add 200 µL of surrogate to 10 mL of hexane in a clean centrifuge tube or 10 mL vial. Concentrate the solution to 2.0 mL by using either nitrogen blowdown or a micro Snyder column. The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. Measure the volume with a syringe or by transferring the solution to a volumetric flask. Proceed with the sulfur removal using the same technique (mercury or copper) as the samples associated with the blank.

12.4.3.2 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 13 and analyze the extract. If the mercury turns black, repeat sulfur removal as necessary. 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.

12.4.3.3 Copper technique

Add approximately 2 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 13 and analyze the extract. If the copper changes color, repeat the sulfur removal procedure as necessary.

12.5. Extract Concentration

Two different techniques are permitted to concentrate the extract to 1.0 mL (volume before Florisil cleanup) or 2.0 mL (extract volume before instrumental analysis). They are the micro Snyder column and nitrogen evaporation techniques.

12.5.1 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 (60°C to 65°C) 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 1.0 mL (Florisil) or 2.0 mL (analysis).

12.5.2 Nitrogen Evaporation Technique (taken from ASTM Method D 3086).

12.5.2.1 Place the concentrator tube with an open micro Snyder column attached in a warm water bath (30°C to 35°C) 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) or 2.0 mL (analysis).

12.5.2.2 CAUTION: Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or Teflon tubing. The internal wall of new tubing must be rinsed several times with hexane and then dried prior to use. 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.

12.5.3 If the extract has not been put through Florisil yet, proceed to Section 12.4 for extract cleanup. Otherwise, transfer the extract to a Teflon-lined screw-cap bottle and label the bottle. Store at 4°C ($\pm 2^\circ\text{C}$).

SECTION V

SAMPLE ANALYSIS AND COMPOUND IDENTIFICATION AND QUANTITATION

13. INSTRUMENTAL ANALYSIS

Before samples or required blanks can be analyzed, the instrument must meet the initial calibration and calibration verification technical acceptance criteria. All sample extracts, including LCS and PES, required blanks, and calibration standards must be analyzed under the same instrumental conditions. All samples (including the LCS and PES), required blanks, extracts, and standard/spiking solutions must be allowed to warm to ambient temperature (approximately 1 hour) before preparation/analysis.

- 13.1 Set up the GC/ECD system per the requirements in Section III. Unless ambient temperature on-column injection is used (see Section 8), the injector must be heated to at least 200°C. The optimized gas chromatographic conditions from Section 8 must be used.
- 13.2 The injection must be made on-column by using either automatic or manual injection. If autoinjectors are used, 1.0 uL injection volumes may be used. Manual injections must use at least 2.0 uL 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 uL. However, the same injection volume must be used for all analyses.

13.3 All acceptable samples 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
	o	
	o	Subsequent Samples
	o	
12 hr.	o	Last sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd injection	Performance Evaluation Mixture
	o	Sample
	o	
	o	Subsequent Samples
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd and 3rd injections past 12 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
	etc.	

NOTE: The first 12 hours are counted from the injection #16 (the Instrument Blank at the end of the initial calibration sequence), not from injection #1. Samples 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 accomodate staff working on 8-hour shifts.

- 13.4 Included with the subsequent samples in the analysis sequence are all of the required method blanks and sulfur cleanup blanks. The Contractor may decide at what point in the sequence the method blanks and sulfur cleanup blanks are to be analyzed.

13.5 Termination of Data Acquisition

After decachlorobiphenyl has eluted from the GC column, the data acquisition may be terminated for that analysis.

14. DILUTIONS

- 14.1 The sample or blank must first be analyzed at the most concentrated level (injection taken from the 2.0 mL final extract after the clean-up steps).
 - 14.1.1 If the response of any single component pesticide is greater than the response of that analyte in the initial calibration high point standard, then the extract must be diluted to have the response of that analyte between the initial calibration low point and high point standard.
 - 14.1.2 If the response of the largest peak in a multicomponent analyte is greater than the most intense single component analyte response in the initial calibration high point standard, then the response of the largest peak in a multicomponent analyte must be diluted to have its response between the responses of the initial calibration midpoint and high point standard of that single component pesticide.
 - 14.1.3 If a greater than 10 times dilution is needed (for example, 50 to 1 dilution), then a 10 times more concentrated dilution (5 to 1 dilution) also must be analyzed.
- 14.2 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.
- 14.3 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.
- 14.4 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.

14.5 Dilute the sample using the following procedure:

14.5.1 Calculate the extract dilution in order for the single component pesticides to meet the requirement listed in paragraph 14.2.

14.5.2 Calculate the extract dilution in order for the multicomponent analytes to meet the requirement listed in 14.3.

14.5.3 Dilute the sample extract with hexane in a volumetric flask.

15. IDENTIFICATION OF TARGET ANALYTES

15.1 The laboratory will identify single component analyte peaks based on the retention time 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 GC columns.

15.2 A set of three to five major peaks is selected for each multicomponent analyte. Retention time 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 retention time window of the corresponding peaks of the standard on both GC columns. The number of potential quantitation peaks is listed in Table D.14.

TABLE D-14

<u>Multicomponent Analyte</u>	<u>No. of Potential Quantitation Peaks</u>
Aroclor 1016/1260	5/5
Aroclor 1221	3
Aroclor 1232	4
Aroclor 1242	5
Aroclor 1248	5
Aroclor 1254	5
Toxaphene	4

15.3 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.

15.4 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.

15.5 Toxaphene and Aroclors require only a single-point calibration. Identification requires visual inspection of an on-scale pattern.

16. QUANTITATION OF ANALYTES

16.1 Quantitation for all analytes and surrogates must be performed and reported on both columns.

16.2 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 SDG Narrative.

16.3 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 continuing calibration for quantitation of samples.

16.4 The Contractor must quantitate each multicomponent analyte based on the calibration factor from the most recent initial calibration standard.

16.5 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.

16.6 Before reporting data, it is required that the Contractor check for flags generated by the data system that indicate improper quantitation of analytes.

16.7 The chromatograms of all samples, standards, and blanks must be reviewed by a qualified pesticide analyst before they are reported.

16.8 Calculations.

16.8.1 Calculate the concentration of the single component pesticides and surrogates by using the following equation:

$$\text{EQ. D.32 Concentration ug/L} = \frac{(A_x)(V_t)(Df)}{(CF)(V_i)(V_x)}$$

Where:

A_x = Response (peak area or height).

CF = Calibration factor for the external standard (Paragraph 9.4.3) (per ng).

V_t = Volume of total extract (uL). (This volume is 2000 uL.)

V_i = Volume of extract injected (uL). (If a single injection is made onto two columns, use one-half the volume of 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:

$$\frac{\text{uL most conc. extract used to make dilution} + \text{uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The calibration factors used in Equation D.32 are those from the most recent initial calibration. If the calibration factors used to determine the linearity of the initial calibration were based on peak area, then the response 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.

16.8.2 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 D.32. Determine the mean concentration for all of the selected peaks. The mean value is reported on Form X (Exhibit B) for both GC columns.

16.8.3 For the single component pesticides, report the lower of the two values quantitated from the two columns of Form I. For the multicomponent analytes, report the lower of the two mean values from the two columns on Form I.

16.8.4 The recoveries of the surrogates are calculated according to Equation D.33.

EQ. D.33

$$\text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis.

Q_a = Quantity added to sample/blank.

16.8.5 The percent difference is calculated according to Equation D.34.

EQ. D.34

$$\%D = \frac{\text{Conc}_H - \text{Conc}_L}{\text{Conc}_L} \times 100$$

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 that 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.

17. TECHNICAL ACCEPTANCE CRITERIA FOR SAMPLE ANALYSIS

All requirements listed below apply independently to both GC columns and to all instruments used for these analyses.

- 17.1 Samples must be analyzed under the GC/ECD operating conditions in Section 8. The instrument must have met all initial calibration and calibration verification technical acceptance criteria. Sample data must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, Performance Evaluation Mixtures, and Individual Standard Mixtures A and B, as described in Section 10.2.
- 17.2 The sample must be extracted and analyzed within the contract holding times.
- 17.3 The LCS associated with the samples must meet the LCS technical acceptance criteria. The PES associated with the samples must meet the PES technical acceptance criteria. The method blank extracted with the samples must meet 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.
- 17.4 The retention time for each of the surrogates must be within the retention time window as calculated in Section 9.
- 17.5 The percent recovery for the surrogates must be between 60.0 and 150 percent, inclusive.
- 17.6 No target analyte concentrations may exceed the upper limit of the initial calibration (See paragraph 14.1.1 and 14.1.2) or else extracts must be diluted and reanalyzed.
- 17.7 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.

17.8 The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The retention time 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 retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.

17.8.1 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.

17.8.2 Chromatograms must display single component pesticides detected in the sample at less than full scale.

17.8.3 Chromatograms must display the largest peak of any multicomponent analyte detected in the sample at less than full scale.

17.8.4 If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale.

17.8.5 If an extract must be diluted, chromatograms must display multicomponent analytes between 25 and 100 percent of full scale.

17.8.6 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.

17.8.7 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

17.8.8 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.

18. CORRECTIVE ACTION

18.1 If the sample 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 reanalyzed at no additional cost after the corrective action.

- 18.2 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 SMO. SMO will contact the Region for instructions.
- 18.3 Sample analysis technical acceptance criteria MUST be met before data are reported. Samples contaminated from laboratory sources or associated with a contaminated method blank or sulfur cleanup blank will require reextraction and reanalysis at no additional cost. Any samples analyzed that do not meet the technical acceptance criteria will require reextraction and/or reanalysis at no additional cost.
- 18.4 Sample reextraction/reanalyses 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 SMO.

SECTION VI

SAMPLE QUALITY CONTROL PROCEDURES AND REQUIREMENTS

20. BLANKS

Summary

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.

20.1 Method Blanks

20.1.1 Summary

A method blank is 1.0 liter of reagent water carried through the entire analytical scheme.

20.1.2 Frequency

A method blank must be extracted and analyzed:

- o once every 20 samples, AND
- o every time samples are extracted.

20.1.3 Procedure

20.1.3.1 Measure 1.0 liter of reagent water for each method blank aliquot. Add 200 uL of the surrogate solution. Extract and concentrate the method blank according to Section 12.

20.1.3.2 Analyze the method blank according to Section 13. Calculate the results according to Section 16.

20.1.4 Technical Acceptance Criteria for Method Blanks

20.1.4.1 All method blanks must be prepared and analyzed at the frequency described in Paragraph 20.1.2 using the procedure in Paragraph 20.1.3 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

20.1.4.2 The concentration in the method blank of the target compounds in Exhibit C must be less than or equal to the CRQL for each target compound.

20.1.4.3 The method blank must meet all sample technical acceptance criteria in Section 17.

20.1.5 Corrective Action

- 20.1.5.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures **MUST** be taken and documented before further sample analysis proceeds.
- 20.1.5.2 Any method blank that fails to meet the technical acceptance criteria must be reextracted and reanalyzed at no additional cost. Further, all samples (including LCS and PES) processed with a method blank that does not meet the blank technical acceptance criteria (i.e., contaminated) will require reextraction and reanalysis at no additional cost.

20.2 Sulfur Cleanup Blank

20.2.1 Summary

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 procedure (Section 12.4.3).

20.2.2 Frequency

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.

20.2.3 Procedure

- 20.2.3.1 Prepare the sulfur cleanup blank per Paragraph 12.4.3.1 at the frequency listed in Paragraph 20.2.2.
- 20.2.3.2 Analyze the sulfur cleanup blank according to Section 13. Calculate the results according to Section 16.

20.2.4 Technical Acceptance Criteria for Sulfur Cleanup Blanks

- 20.2.4.1 All sulfur cleanup blanks must be prepared and analyzed at the frequency described in Paragraph 20.2.2 using the procedure in Paragraph 20.2.3 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.
- 20.2.4.2 The concentration in the sulfur cleanup blank of the target compounds in Exhibit C must be less than or equal to the CRQL for each target compound.
- 20.2.4.3 The sulfur cleanup blank must meet all sample technical acceptance criteria in Section 17.

20.2.5 Corrective Action

- 20.2.5.1 If a sulfur blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control. It is the Contractor's responsibility to ensure that 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. 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.
- 20.2.5.2 Any sulfur blank that fails to meet the technical acceptance criteria must be reextracted and reanalyzed at no additional cost. Further, all samples (including LCS and PES) processed with a sulfur blank that does not meet the blank technical acceptance criteria (i.e., contaminated) will require reextraction and reanalysis at no additional cost.

20.3 Instrument Blanks

20.3.1 Summary

An instrument blank is a volume of clean solvent containing the surrogates that is analyzed to determine the extent of contamination in the GC/ECD system.

20.3.2 Frequency

The first analysis after a 12-hour analysis sequence (see Section 10.2) must be an instrument blank. All groups of acceptable sample analyses are to be preceded and followed by acceptable instrument blanks. 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 (see Section 10.2).

20.3.3 Procedure

20.3.3.1 Prepare the instrument blank by spiking the surrogates into hexane or iso-octane for a concentration of 20.0 ng/mL of Tetrachloro-m-xylene and Decachlorobiphenyl.

20.3.3.2 Analyze the instrument blank according to Section 13 at the frequency listed in paragraph 20.3.2. Calculate the results according to Section 16.

20.3.4 Technical Acceptance Criteria for Instrument Blanks

20.3.4.1 All instrument blanks must be prepared and analyzed at the frequency described in Paragraph 20.3.2 using the procedure in Paragraph 20.3.3 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

20.3.4.2 The concentration in the instrument blank of each target analyte in Exhibit C must be less than or equal to the CRQL for that analyte.

20.3.4.3 The instrument blank must meet all sample technical acceptance criteria in Section 17.

20.3.5 Corrective Action

20.3.5.1 If analytes are detected at greater than the CRQL or the surrogate 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 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.

20.3.5.2 Analysts are cautioned that running an instrument blank once every 12 hours (see Section 10) is the minimum contract requirement. 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 more often to avoid discarding data.

21. LABORATORY CONTROL SAMPLES (LCS)

21.1 Summary

The laboratory control sample (LCS) is an internal laboratory quality control sample designed to assess (on an SDG-by-SDG basis) the capability of the contractor to perform the analytical method listed in this Exhibit.

21.2 Frequency

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 instrumentation as the samples in the SDG.

21.3 Procedure

21.3.1 Measure a 1 liter aliquot of reagent water in a 1 liter graduated cylinder and transfer the water to a continuous extractor. Pipet 1.0 mL of the LCS spiking solution (Paragraph 7.3.7) and 200 uL of the surrogate standard spiking solution into the water and mix well. Extract and concentrate the sample according to Section 12.

21.3.2 Analyze the LCS per Section 13.

21.4 Calculations

21.4.1 Calculate the results according to Section 16.

21.4.2 Calculate individual compound recoveries of the LCS using Equation D.33, substituting LCS percent recovery for surrogate percent recovery.

21.5 Technical Acceptance Criteria For Laboratory Control Sample Analysis

21.5.1 The LCS must be analyzed at the frequency described in Section 21.2 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

21.5.2 The LCS must be prepared as described in Paragraph 21.3.

21.5.3 The LCS must meet all sample technical acceptance criteria in Section 17.

21.5.4 The percent recovery for each of the compounds in the LCS must be within the recovery limits listed in Table D-15.

Table D-15

LABORATORY CONTROL SAMPLE RECOVERY LIMITS

COMPOUND	% RECOVERY
gamma-BHC	56-123
Heptachlor epoxide	74-150
Dieldrin	33-130
4,4'-DDE	50-150
Endrin	56-121
Endosulfan sulfate	50-100
gamma-Chlordane	33-130

NOTE: The recovery limits for any of the compounds in the LCS may be expanded at any time during the period of performance if SMO determines that the limits are too restrictive.

21.6 Corrective Action

- 21.6.1 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.
- 21.6.2 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 reextraction and reanalysis of the LCS at no additional cost.
- 21.6.3 All samples prepared and analyzed in an SDG with an LCS that does not meet the technical acceptance criteria will also require reextraction and reanalysis at no additional cost.

22. PERFORMANCE EVALUATION SAMPLES (PES)

22.1 Summary

The PES is an external laboratory quality control sample prepared and designed to assess (on an SDG-by-SDG basis) the capability of the contractor to perform the analytical method listed in this Exhibit.

22.2 Frequency

The Contractor must extract, analyze, and report the PES once per SDG, if available. The PES must be extracted and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples in the SDG.

22.3 Procedure

22.3.1 The PES will be received either as an ampulated extract or as a full volume sample. If received as an ampulated extract, the Contractor will receive instructions concerning the dilution procedure to bring the extract to full volume prior to preparation and analysis of the PES.

22.3.2 Add 200 uL of surrogate solution to 1 liter of reagent water spiked with the PES solution. Extract and concentrate the PES using the procedure described in Section 12. Analyze the PES as described in Section 13.

22.4 Calculations

See paragraph 16 for all equations necessary for calculations.

22.5 Technical Acceptance Criteria for Performance Evaluation Sample

22.5.1 The PES must be analyzed on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria at the frequency described in Section 22.2.

22.5.2 The PES must be extracted and concentrated according to Section 22.3.

22.5.3 The PES must meet all sample technical acceptance criteria in Section 17.

22.6 Corrective Action

22.6.1 If the PES technical acceptance criteria for the surrogates are not met, check calculations, standard solutions and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria. Any PES failing to meet these technical acceptance criteria must be reextracted and reanalyzed at no additional cost. If insufficient PES extract remains or if an insufficient volume of the PES remains, document this in the SDG Narrative by stating that the PES could not be reextracted and reanalyzed because insufficient volume remained.

22.6.2 In addition to complying with the PES technical acceptance criteria, the Contractor will be responsible for correctly identifying and quantifying the compounds included in the

Performance Evaluation Sample. SMO will notify the Contractor of unacceptable performance.

Note: Unacceptable performance for identification and quantitation of compounds is defined as a score less than 75 percent.

- 22.6.3 The PES technical acceptance criteria MUST be met before sample data are reported. Also, the Contractor must demonstrate acceptable performance for compound identification and quantitation.