USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

ORGANICS ANALYSIS

Multi-Media, Multi-Concentration

OLM04.1
September 1998

STATEMENT OF WORK

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EXHIBIT A

SUMMARY OF REQUIREMENTS

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1.0 PURPOSE

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

2.0 DESCRIPTION OF SERVICE

The organic analytical service provides a contractual framework for laboratories to apply EPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of *48* volatile, *65* semivolatile, and 28 pesticide/Aroclor target compounds in water and soil/sediment environmental samples. The analytical service provides the methods to be used, and the specific contractual requirements by which EPA will evaluate the data. This service uses gas chromatography/mass spectrometry (GC/MS) and gas chromatography/electron capture (GC/EC) methods to analyze the target compounds.

3.0 DATA USES

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

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the SOW. This SOW is designed as part of the documentation for a contract between EPA and a commercial laboratory performing analyses in support of EPA Superfund programs. The SOW is comprised of eight exhibits. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and the forms instructions. Exhibit C specifies the target compound list for this SOW with the contract-required quantitation limits for sample matrices. Exhibit D details the specific analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required quality assurance/quality control (QA/QC), standard operating procedures, and procedures used for evaluating analytical methodologies, QA/QC performance, and the reporting of data. Exhibit F contains chain-of-custody and sample documentation requirements which the Contractor shall follow. To ensure proper understanding of the terms utilized in this SOW, a glossary can

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

4.2 Overview of Major Task Areas. For each sample, the Contractor shall perform the tasks described in this section. Specific requirements for each task are detailed in the exhibits as referenced.

4.2.1 Task I: Chain-of-Custody

4.2.1.1 Chain-of-Custody. The Contractor shall receive and maintain samples under proper chain-of-custody procedures. All associated document control and inventory procedures shall be developed and followed. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported as the Complete Sample Delivery Group File (CSF) (see Exhibit B). The Contractor shall establish and use appropriate procedures to handle confidential information received from the Agency. See Exhibit F for specific requirements.

4.2.1.2 Sample Scheduling/Shipments. Sample shipments to the Contractor's facility will be scheduled and coordinated by the CLP Sample Management Office (SMO). The Contractor shall communicate with SMO personnel by telephone, as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed. *At the time of sample scheduling, the Contractor will be notified if the Modified SW-846 Method 5035 is to be used in the preparation and analysis of low level soil samples for volatiles.*

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

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

4.2.1.2.3 To effectively monitor the temperature of the sample shipping cooler, each USEPA Regional office may include a sample

shipping cooler temperature blank with each cooler shipped. The temperature blank will be clearly labeled: USEPA COOLER TEMPERATURE INDICATOR.

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

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

4.2.1.2.3.3 To determine the temperature of the cooler, the contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer ($\pm 1^{\circ}C$) shall have a measurable range of 0 to 50 degrees Celsius. Other devices which can measure temperature may be used if they can be calibrated to $\pm 1^{\circ}C$ and have a range of 0 to $20^{\circ}C$. *If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. However, under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the SDG Narrative.*

4.2.1.2.3.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10 degrees Celsius, the Contractor shall contact SMO and inform them of the temperature deviation. The SMO will contact the Region from which the samples were shipped for instructions on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). The SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision in the SDG Narrative. Also in the SDG Narrative, the Contractor shall list by fraction, the USEPA sample number of all samples which were shipped in a cooler which exceeded 10 degrees Celsius.

4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on the DC-1 Form, under Remark #9 - *Cooler Temperature*, and in the SDG Narrative.

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

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

4.2.2 Task II: Analysis of Samples

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

4.2.2.1.1 A Case consists of one or more Sample Delivery Group(s). A Sample Delivery Group (SDG) is defined by the following, whichever is most frequent:

- Each Case of field samples received, OR
- Each 20 field samples *(excluding PE samples)* within a Case, OR
- Each 7 calendar day period *(excluding Sundays and Government holidays)* during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

4.2.2.1.2 Samples may be assigned to SDGs by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory. *However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case.* Such assignment shall be made at the time the samples are received, and shall <u>not</u> be made retroactively.

4.2.2.2 Preparation Techniques. The Contractor will prepare samples as described in Exhibit D. For semivolatile and pesticide/Aroclor samples, an aliquot is extracted with a solvent and concentrated. The concentrated extract is subjected to fraction-specific cleanup procedures and then analyzed by GC/MS for semivolatile or GC/EC for the pesticide/Aroclor target compounds listed in Exhibit C.

For volatile samples, an aliquot is purged with an inert gas, trapped on a solid sorbent, and then desorbed onto the GC/MS for analysis of the target compounds listed in Exhibit C.

4.2.2.3 Analytical Techniques. The target compounds listed in Exhibit C shall be identified as described in the methodologies given in Exhibit D. Automated computer programs may be used to facilitate the identification of compounds.

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

4.2.2.4.1 If a compound initially identified by GC/MS techniques cannot be verified, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the Contractor shall report that identification and proceed with quantitation.

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

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

4.2.2.6 Tentative Identification of Non-Target Sample Components. For each analysis of a sample, the Contractor shall conduct mass spectral library searches to determine tentative compound identifications as follows: For each volatile sample, the Contractor shall conduct a search to determine the possible identity of up to 30 organic compounds of greatest concentration which are not system monitoring compounds or internal standards and are not listed in Exhibit C under volatiles or semivolatiles. For each semivolatile sample, the Contractor shall conduct a search to determine the possible identification of up to 30 organic compounds of greatest concentration which are not surrogates or internal standards and are not listed in Exhibit C

under volatiles or semivolatiles. In performing searches, the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent, mass spectral library shall be used.

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

4.2.2.7 Quality Assurance/Quality Control Procedures. The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibit B and Exhibit H.

4.2.2.7.1 The Contractor shall maintain a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the quality control procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

4.2.2.7.2 Additional quality control shall be conducted in the form of the analysis of laboratory evaluation samples submitted to the laboratory by the Agency. The results of all such quality control or laboratory evaluation samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to the Agency or rejection of data for: sample(s), a fraction within an SDG, or the entire SDG, and/or may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct compound identification and concentration values as determined by the Agency, as well as meeting the contract requirements for analysis (Exhibit D), quality assurance/quality control (Exhibit E), data reporting and other deliverables (Exhibits B and H), and sample custody, sample documentation, and standard operating procedure documentation (Exhibit F).

4.2.2.8 The Contractor may be requested by EPA to perform modified analyses. These modifications may include, but are not limited to, additional compounds, sample matrices other than soil/sediment or water, and lower quantitation limits. These requests will be made by the EPA Administrative Project Officer and Contracting Officer in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. In addition, all applicable contract requirements specified in the Statement of Work/Specifications will remain in effect.

4.2.3 Task III: Reporting Requirements

4.2.3.1 EPA has provided the Contractor with formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for

completing and *submitting* analysis data sheets and computer-readable data on diskette *(or via an alternate means of electronic transmission approved in advance by the EPA)* in the format specified in this SOW and within the time specified in the Contract Performance/Delivery Schedule in Exhibit B.

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

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

4.2.3.4 The data reported by the Contractor on the hardcopy data forms and the associated computer-readable data submitted by the Contractor shall contain identical information. If discrepancies are found during government inspection, the Contractor shall be required to resubmit either the hardcopy forms or the computer-readable data, or both sets of data, at no additional cost to the Agency.

4.3 Technical and Management Capability

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

4.3.2 Instrumentation. The Contractor shall have sufficient gas chromatograph/electron capture/data system (GC/EC/DS), gas chromatograph/mass spectrometer/data system (GC/MS/DS), including magnetic tape storage devices, and gel permeation chromatography system (GPC) capability to meet all the terms and conditions of the contract.

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

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

Exhibit B - Reporting and Deliverables Requirements

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

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

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

Table 1

	Item	No. of Copies[A]	Delivery Schedule	Distribution: SMO	Distribution: Region
A.[1, 3]	Sample Traffic Reports	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).[2]	X	
B.[3]	Sample Data Summary Package	1	*XX*[B] days after receipt of last sample in SDG.	X	
C.[3]	Sample Data Package[C]	1	*XX*[B] days after receipt of last sample in SDG.	X	
D.[3]	Data in Computer Readable Format	1	*XX*[B] days after receipt of last sample in SDG.	X	X
E.[3, 4]	Complete SDG File	1	*XX*[B] days after receipt of last sample in SDG.		X
F.[6]	Preliminary Results (VOA Analyses)	1	Within 48 hours after receipt of last sample in SDG at laboratory, if requested.	X	X
	Preliminary Results (SV and Pest Analyses)	1	Within 72 hours after receipt of last sample in SDG at laboratory, if requested.	X	X

	Item	No. of Copies	Delivery Schedule	Distribution SMO	Distribution Region
G.[5]	Standard Operating Procedures--Technical and Evidentiary	1	*Revise within 60 days after contract award.* Submit within 7 days of receipt of written request to recipients as directed.		*As directed*
H.[5]	Quality Assurance Plan	1	*Revise within 60 days after contract award.* Submit within 7 days of receipt of written request to recipients as directed.		*As directed*
I.	GC/MS *and GC/EC* Tapes	Lot	Retain for 365 days after data submission. Submit within 7 days after receipt of written request by APO.		As directed
J.	Extracts	Lot	Retain for 365 days after data submission. Submit within 7 days after receipt of written request by APO or SMO, at the Agency's direction.		As directed

Footnotes:

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

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

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

1 Also required in the Sample Data Summary Package.

2 A 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 *(excluding PE 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 the last sample in the SDG is received. See Exhibit A for further description.

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

4 Complete SDG File will contain the original sample data package plus all of the original documents described under Section 2.7.

5 See Exhibit E and Exhibit F for a more detailed description.

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

Footnotes (con't):

Preliminary *Results Delivery Schedule*:

If the last sample in the SDG arrives before 5 p.m., the Preliminary Results are due within the required turnaround time. If the last sample in the SDG is received after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day. ***DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day.***

If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.

NOTE: As specified in the Contract Schedule (Government Furnished Supplies and Materials), unless otherwise instructed by the CLP Sample Management Office based on a Regional decision, 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.

1.2 Distribution. The following addresses correspond to the "Distribution" column in Table 1 of Section 1.1.

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

Region: USEPA Region: The Sample Management Office will provide the Contractor with the list of addresses for the 10 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.

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

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

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

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

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

- Legible,
- Clearly labeled and completed in accordance with instructions in this exhibit,
- Arranged in the order specified in this section,
- Paginated consecutively in ascending order starting from the SDG Narrative, and
- Copies must be legible and double-sided.

NOTE: Complete SDG files need not be double-sided. (The CSF is composed of original documents.) However, sample data packages delivered to SMO must be double-sided.

2.1.1 Requirements for each deliverable item cited in the Contract Schedule (Contract Performance/Delivery Schedule) are specified in Sections 2.3 through *2.11*. Prior to submission, the Contractor shall arrange items and the components of each item in the order listed in these sections.

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

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

2.2.1 The Contractor shall respond within seven (7) days to written requests from data recipients for additional information or explanations that result from the Government's inspection activities unless otherwise specified in the contract.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, or through an Administrative Project Officer/Technical Project Officer action, or through a Regional data reviewer's request, the data shall be clearly marked as ADDITIONAL DATA and shall be sent to both contractual data recipients (SMO and the Region; and to the EPA designated recipient (e.g., QATS) when a written request for the sample data package has been made). The Contractor shall include a cover letter which describes which data are being delivered, to which EPA Case(s) the data pertain, and **who requested the data**.

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

2.3 Quality Assurance Plan and Standard Operating Procedures. The Contractor shall adhere to the requirements in Exhibits E and F.

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

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

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

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

2.4.2 Each TR shall be clearly marked with the SDG number, entered below the laboratory receipt date on the TR. The TR for the **last** sample received in the SDG shall be clearly marked "SDG--FINAL SAMPLE." The SDG number is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

2.4.3 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the Contractor shall make the

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

2.5 Sample Data Summary Package. The sample data summary package shall be ordered as follows and shall be submitted separately (i.e., separated by rubber bands, clips, or other means) directly **preceding** the sample data package. Sample data forms shall be arranged in increasing EPA sample number order, considering **both** letters and numbers. For example, BE400 is a lower sample number than BF100, as E precedes F in the alphabet. The SDG number shall be reported on all data reporting forms. The sample data summary package shall be arranged in the same manner as the sample data package. The sample data summary package shall contain data for all samples in one SDG of the Case, as follows: (See Section 2.6 for a detailed description of each item.)

2.5.1 SDG Narrative.

2.5.2 Arranged by fraction and by sample within each fraction: tabulated target compound results (Form I) for the volatile, semivolatile, and pesticide fractions and tentatively identified compounds (Form I TIC) for the volatile and semivolatile fractions only.

2.5.3 Arranged by fraction: system monitoring compound or surrogate spike analysis results (Form II) by matrix (water and/or soil) *for the volatile, semivolatile, and pesticide fractions;* and for soil, by concentration (low or medium), for *volatile and* semivolatile fractions.

2.5.4 Arranged by fraction: matrix spike/matrix spike duplicate results (Form III) for the volatile, semivolatile, and pesticide fractions.

2.5.5 Arranged by fraction: blank data (Form IV) and tabulated results (Form I) for the volatile, semivolatile, and pesticide fractions including tentatively identified compounds (Form I TIC) for the volatile and semivolatile fractions only.

2.5.6 Arranged by fraction: internal standard area data (Form VIII) for the volatile and semivolatile fractions only.

2.6 Sample Data Package. The sample data package is divided into the five major units described in this section. The last three units are each specific to an analytical fraction (volatiles, semivolatiles, and pesticides/Aroclors). If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable. The sample data package shall include data for the analyses of all samples in one SDG, including: field samples, dilutions, reanalyses, blanks, matrix spikes, and matrix spike duplicates. 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.

2.6.1 SDG Narrative. This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; Case number; EPA sample numbers in the SDG, differentiating between initial analyses and reanalyses; 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. *All volatile low level soil samples prepared according to the Modified SW-846 Method 5035 must be noted in the SDG Narrative. When using the Modified SW-846 Method 5035, all discrepancies between sample weights determined in the field and in the laboratory shall be documented in the SDG Narrative.*

All GC columns used for analysis shall be documented here, by fraction. List the GC column identification--brand name, the internal diameter, in mm, and the length, in meters, packing/coating material and film thickness. The trap used for volatile analysis shall be described here. List trap name, when denoted by the manufacturer, its composition (packing material/brand name, amount of packing material, in length, cm). All tentatively identifed alkanes and their estimated concentrations are to be reported here. The EPA sample number, the CAS number, when available, the alkane compound (or series) name, and its estimated concentration shall be provided in a tabular format. The Contractor shall include any technical and administrative problems encountered, the corrective actions taken, the resolution, and an explanation for all flagged edits (e.g., manual edits) on quantitation lists. The Contractor shall document in the SDG Narrative all instances of manual integration.

The SDG Narrative shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the laboratory manager or his/her designee, as verified by the following signature." This statement shall be directly followed by an original signature of the laboratory manager or his/her designee with a typed line below it containing the signer's name and title, and the date of signature.

2.6.1.1 Whenever data from sample reanalyses are submitted, the Contractor shall state in the SDG Narrative for **each** reanalysis whether the reanalysis is billable, and if so, why.

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

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

Contractor shall submit a copy of the SDG cover sheet with copies of the TRs.

2.6.3 Volatiles Data

2.6.3.1 Volatiles QC Summary

2.6.3.1.1 System Monitoring Compound Summary (Form II, VOA-1, VOA-2).

2.6.3.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III, VOA-1, VOA-2).

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

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

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

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

2.6.3.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I, VOA-1, VOA-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

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

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

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

2.6.3.2.3.1 Internal standards and system monitoring compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times are printed over the peak.

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

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

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

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

- EICPs displaying each manual integration.

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

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

- Copies of mass spectra of organic compounds not listed in Exhibit C with associated best-match spectra (minimum of one, maximum of three best matches). Spectra shall be labeled with EPA sample number, lab file identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.

2.6.3.3 Volatiles Standards Data

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

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.6.3.2.3. Spectra are not required.

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

- EICPs displaying each manual integration.

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

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

calibrations, labeled as in Section 2.6.3.2.3. Spectra are not required.

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

- EICPs displaying each manual integration.

2.6.3.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standards and system monitoring compounds.

2.6.3.4 Volatiles Raw QC Data

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

- Bar graph spectrum, labeled as in Section 2.6.3.2.3.

- Mass listing, labeled as in Section 2.6.3.2.3.

- Reconstructed total ion chromatogram, labeled as in Section 2.6.3.2.3.

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

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

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

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

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.3.2.3.

- Target compound spectra with laboratory-generated standard, labeled as in Section 2.6.3.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:

  -- Raw target compound spectra.

  -- Enhanced or background-subtracted spectra.

  -- Laboratory-generated standard spectra.

- GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.6.3.2.4.

- Quantitation/calculation of tentatively identified compound concentrations.

2.6.3.4.3 Volatiles Matrix Spike Data

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

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.3.2.3. Spectra are not required.

2.6.3.4.4 Volatiles Matrix Spike Duplicate Data

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

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.3.2.3. Spectra are not required.

2.6.4 Semivolatiles Data

2.6.4.1 Semivolatiles QC Summary

2.6.4.1.1 Surrogate Percent Recovery Summary (Form II, SV-1, SV-2).

2.6.4.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III, SV-1, SV-2)

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

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

2.6.4.1.5 Internal Standard Area and RT Summary (Form VIII, SV-1, SV-2): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

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

2.6.4.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I SV-1, SV-2). Tabulated results (identification and

quantitation) of the specified target compounds (Exhibit C, Semivolatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

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

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

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

2.6.4.2.3.1 Internal standards and surrogate compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times are printed over the peak.

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

- EPA sample number,
- Date and time of analysis,

- Retention time or scan number of identified target compounds,
- Ion used for quantitation with measured area,
- Copy of area table from data system,
- GC/MS instrument identifier, and
- Lab file identifier.

2.6.4.2.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and system monitoring compounds.

- EICPs displaying each manual integration.

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

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

2.6.4.3 Semivolatiles Standards Data

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

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point)

calibration, labeled as in Section 2.6.4.2.3. Spectra are not required.

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

- EICPs displaying each manual integration.

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

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibrations, labeled as in Section 2.6.4.2.3. Spectra are not required.

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

- EICPs displaying each manual integration.

2.6.4.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and system monitoring compounds.

2.6.4.4 Semivolatiles Raw QC Data

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

- Bar graph spectrum, labeled as in Section 2.6.4.2.3.

- Mass listing, labeled as in Section 2.6.4.2.3.

- Reconstructed total ion chromatogram, labeled as in Section 2.6.4.2.3.

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

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

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

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

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.4.2.3.

- Target compound spectra with laboratory-generated standard, labeled as in Section 2.6.4.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:

  -- Raw target compound spectra.

  -- Enhanced or background-subtracted spectra.

  -- Laboratory-generated standard spectra.

- GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.6.4.2.4.

- Quantitation/calculation of tentatively identified compound concentrations.

2.6.4.4.3 Semivolatiles Matrix Spike Data

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

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.4.2.3. Spectra are not required.

2.6.4.4.4 Semivolatiles Matrix Spike Duplicate Data

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

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.4.2.3. Spectra are not required.

2.6.4.4.5 Semivolatile GPC Data. The UV traces for the GPC calibration, *the GPC continuing calibration verification,* and the reconstructed ion chromatogram and data system reports for the GPC blank shall be arranged in chronological order by GPC for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of retention times when the retention times are printed directly over the peak.

- Reconstructed ion chromatogram and data system report(s) labeled as specified in Section 2.6.4.2.3 for GPC blank analysis.

- Reconstructed ion chromatogram and data system report(s) for all standards used to quantify compounds in the GPC blank labeled as specified in Section 2.6.4.2.3 (continuing calibration standard).

2.6.5 Pesticide/Aroclor Data

2.6.5.1 Pesticide/Aroclor QC Summary

2.6.5.1.1 Surrogate Percent Recovery Summary (Form II, PEST-1, PEST-2).

2.6.5.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III, PEST-1, PEST-2).

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

2.6.5.2 Pesticide/Aroclor Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I PEST), followed by the raw data for pesticide samples. These sample packets should then be placed in order of increasing EPA sample number, considering both letters and numbers.

2.6.5.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I PEST). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Pesticides/Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.6.5.2.2 Copies of Pesticide Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D PEST, and shall be labeled with the following information:

- EPA sample number,

- Volume injected (μL),

- Date and time of injection,

- GC column identifier (by stationary phase and internal diameter),
- GC instrument identifier, and
- Scaling factor *(label the x and y axes using a numerical scale)*.

2.6.5.2.3 Copies of pesticide chromatograms from the second GC column shall be included and labeled as in Section 2.6.5.2.2.

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

2.6.5.2.5 All manual work sheets shall be included in the sample data package.

2.6.5.2.6 Other Required Information. If pesticides/Aroclors are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms, raw spectra, and background-subtracted mass spectra of target compounds listed in Exhibit C (Pesticides/Aroclors) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Compound names shall be clearly marked on all spectra. For multicomponent pesticides/Aroclors confirmed by GC/MS, the Contractor shall submit mass spectra of three major peaks of multicomponent compounds from samples and standards.

2.6.5.3 Pesticide/Aroclor Standards Data

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

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

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

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

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

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

2.6.5.3.7 Calibration Verification Summary (Form VII PEST-1): for all performance evaluation mixtures and instrument blanks, on all GC columns and instruments, in chronological order by GC column and instrument.

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

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

2.6.5.3.10 Florisil Cartridge Check (Form IX PEST-1): for all lots of cartridges used to process samples in the SDG.

2.6.5.3.11 Pesticide GPC Calibration *Verification* (Form IX PEST-2): for all GPC columns, in chronological order by calibration *verification* date.

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

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

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

- Resolution check mixture.
- Performance evaluation mixtures, all.
- Individual Standard Mixture A, at three concentrations, each initial calibration.
- Individual Standard Mixture B, at three concentrations, each initial calibration.

- All multicomponent analytes (toxaphene and Aroclors), each initial calibration.

- All mid-point concentrations of Individual Standard Mixtures A and B used for calibration verification.

- All multicomponent analyte standards analyzed for confirmation.

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

- EPA sample number for the standard. (e.g., INDAL1, INDAM2, etc.). See Section 4 for details.

- Label all standard peaks for all individual compounds either directly out from the peak on the chromatogram or on the printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram.

- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram.

- Date and time of injection.

- GC column identifier (by stationary phase and internal diameter).

- GC instrument identifier.

- Scaling factor *(label the x and y axes using a numerical scale)*.

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

2.6.5.4 Pesticide/Aroclor Raw QC Data

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

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

- Tabulated results (Form I PEST).

- Chromatogram(s) and data system printout(s) for each GC column and instrument used for analysis, labeled as in Sections 2.6.5.2.2 and 2.6.5.2.4.

2.6.5.4.2 Matrix Spike Data

- Tabulated results (Form I PEST) of target compounds.

- Chromatogram(s) and data system printout(s), labeled as in Sections 2.6.5.2.2 through 2.6.5.2.4.

2.6.5.4.3 Matrix Spike Duplicate Data

- Tabulated results (Form I PEST) of target compounds.

- Chromatogram(s) and data system printout(s), labeled as in Sections 2.6.5.2.2 through 2.6.5.2.4.

2.6.5.5 Raw GPC Data

2.6.5.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of retention times when the retention times are printed directly over the peak.

- Chromatogram and data system report(s) labeled as specified in Sections 2.6.5.2.2 and 2.6.5.2.4 for GPC blank analyses.

- Chromatogram and data system report(s) for all standards used to quantify compounds in the GPC blank labeled as specified in Section 2.6.5.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, and the Aroclor/toxaphene standards).

2.6.5.5.2 GPC Calibration *Verification*. The chromatogram and the data system report(s) shall be arranged in chronological order for the GPC calibration check.

- Chromatograms and data system printouts labeled as specified in Sections 2.6.5.2.2 and 2.6.5.2.4 for the GPC calibration *verification* solution analyses.

- Chromatogram and data system report(s) for standards used to quantify compounds in the GPC calibration *verification* solution or used to assess the Aroclor pattern labeled as specified in Section 2.6.5.3.15 (i.e., Individual Standard Mixtures A and B and Aroclor Standard Mixture 1016/1260 from the initial calibration sequence).

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

- Chromatograms and data system reports labeled as specified in Sections 2.6.5.2.2 and 2.6.5.2.4 for the florisil cartridge performance check analyses.

- Chromatograms and data system reports for standard analyses used to quantify compounds in the Florisil cartridge performance check analysis, labeled as specified in Section 2.6.5.3.15 (i.e., Individual Standard Mixture A and Individual Standard Mixture B and the 2,4,5 Trichlorophenol solution).

2.7 Complete SDG File. As specified in Section 1, the Contractor shall deliver one Complete SDG File (CSF) including the original sample data package to the Region concurrently with delivery of the sample data package to SMO. Delivery to EPA designated recipient (e.g., QATS) is only required upon written request.

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

2.7.2 The CSF will consist of the following original documents in addition to the documents in the sample data package.

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

2.7.2.1 The original sample data package.

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

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

- EPA Chain-of-Custody Record,

- Airbills *(if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information),*

- EPA Traffic Reports, and

- Sample tags (if present) sealed in plastic bags.

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

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

2.7.2.5 All original laboratory records, not already submitted in the sample data package, of sample transfer, preparation, and analysis including, but not limited to, the following documents:

- Original preparation and analysis forms or copies of preparation and analysis logbook pages,
- Internal sample and sample extract transfer chain-of-custody records,
- Screening records, and
- All instrument output, including strip charts from screening activities.

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

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

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

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

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

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

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

2.10 GC/MS *and GC/EC* Tapes. The Contractor shall adhere to the requirements in Exhibit E.

2.11 Extracts. The Contractor shall preserve sample extracts at 4° C *(± 2°C)* in bottles/vials with Teflon-lined septa. Extract bottles/vials shall be labeled with EPA sample number, Case number, and SDG number. The Contractor shall maintain a logbook of stored extracts, listing EPA sample numbers and associated Case and SDG numbers. The Contractor shall retain extracts for 365 days following submission of the reconciled complete sample data package. During that time, the Contractor shall submit extracts and associated logbook pages within seven days following receipt of a written request from the Administrative Project Officer or Technical Project Officer.

3.0 FORMS INSTRUCTIONS

3.1 Introduction. This section includes specific instructions for completing the data reporting forms required under this contract. Each of the forms are specific to a given fraction (volatile, semivolatile, or pesticide/Aroclor) and, in some instances, specific to a given matrix (water or soil) within each fraction. The Contractor shall submit only those forms pertaining to the fractions analyzed for a given sample(s). For instance, if a sample is scheduled for volatiles analysis only, the Contractor shall provide only forms for the volatile fraction. NOTE: There are two pages relating to the *volatile fraction for Forms I, VI, and VII. There are also two pages relating to the* semivolatile fraction for Forms I, VI, VII, and VIII. Whenever *volatiles or* semivolatiles are analyzed and one of these forms is required, **both** pages (*VOA-1 and VOA-2;* SV-1 and SV-2) shall be submitted.

3.2 General Information. The Contractor shall report values on the hardcopy forms according to the individual form instructions in this section. For example, results for concentrations of volatile target compounds shall be reported to two significant figures if the value is greater than or equal to 10. Values that exceed the maximum length allowed shall be reported to the maximum possible, maintaining the specified decimal place.

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

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

3.2.2 When submitting data, the Contractor shall reproduce **all** characters that appear on the data reporting forms in Section 4. The format of the forms submitted shall be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the Administrative Project Officer. The names of the various fields and compounds (e.g., "Lab Code," "Chloromethane") shall appear as they do on the forms in the contract, including the options specified in the form (e.g., "Matrix: (soil/water)" shall appear, not just "Matrix"). For items appearing on the **uncompleted** forms (Section 4), the use of uppercase and lowercase letters is optional.

3.2.3 Alphabetical entries made on the forms by the Contractor shall be in ALL UPPERCASE letters (e.g., "LOW", not "Low" or "low"). 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, the Contractor shall **not** remove the underscores or vertical bars that delineate "boxes" on the forms. The only exception would be those underscores at the bottom of a "box" that are intended as a data entry line. (For instance, on Form 2A, line 30, if data is entered on line 30, it will replace the underscores.)

3.3 Header Information. Six pieces of information are common to the header section of each data reporting form: lab name, contract, lab code, case number, SAS number, and SDG number. Except as noted for SAS number, this information shall be entered on every form and shall match on every form.

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

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

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

3.3.4 Case Number. The Case number is the EPA-assigned Case number associated with the sample. This number is reported on the Traffic Report.

3.3.5 SAS Number. The SAS number 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 the SAS number and leave the Case number blank. If samples are analyzed according to the Routine Analytical Services (RAS) protocols and have additional SAS requirements, list both the Case number and the SAS number on all forms. If there are no SAS requirements, leave the "SAS No." field blank.

NOTE: Some samples in an SDG may have a SAS number while others may not.

3.3.6 SDG Number. The "SDG No." field is for the sample delivery group number. It is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

3.3.7 Sample Number. This number appears either in the *header information* of the form, or as the left column of a table summarizing data from a number of samples. When the EPA sample number is entered in the triple-spaced box in the upper righthand corner of Form I, Form IV,

or Form X, it should be entered on the middle line of the three lines that comprise the box.

3.3.7.1 The Contractor shall identify **all** samples, including: dilutions, reanalyses, matrix spikes, matrix spike duplicates, blanks, and standards with an EPA sample number. For field samples, matrix spikes and matrix spike duplicates, 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 Contractor shall use the following sample suffixes:

XXXXX	=	EPA sample number
XXXXXMS	=	Matrix spike sample
XXXXXMSD	=	Matrix spike duplicate sample
XXXXXRE	=	Re-extracted and reanalyzed sample
XXXXXDL	=	The suffix DL is appended to the EPA sample number to indicate that the analytical results are a result of a dilution of the original analysis (reported as EPA sample XXXXX). See Exhibit D for requirements for dilutions.

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

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

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

3.3.7.4 The EPA sample number shall be unique for each blank within an SDG. Within a fraction, the Contractor shall achieve this by replacing the two-character terminator (##) of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for volatile blanks would be VBLK1, VBLK2, VBLKA1, VBLKB2, VBLK10, VBLKAB, etc. If the method blank

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

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

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

STD = Standard.

*** = Concentration of volatile standards in ug/L (e.g., 010, 020, 050, 100, and 200) or the amount injected in ng for semivolatile standards (e.g., 020, 050, 080, 120, and 160).

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

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

Name	EPA Sample Number
Individual Mix A (low point)	INDAL##
Individual Mix A (mid-point)	INDAM##
Individual Mix A (high point)	INDAH##
Individual Mix B (low point)	INDBL##
Individual Mix B (mid-point)	INDBM##
Individual Mix B (high point)	INDBH##
Resolution Check	RESC##
Performance Evaluation Mixture	PEM##
Toxaphene	TOXAPH##
Aroclor 1016	AR1016##
Aroclor 1221	AR1221##
Aroclor 1232	AR1232##
Aroclor 1242	AR1242##
Aroclor 1248	AR1248##
Aroclor 1254	AR1254##
Aroclor 1260	AR1260##
Aroclor 1016/1260 Mix	AR1660##

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

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

3.3.7.8 The EPA sample number for GPC shall be GPC#########, where ######### is the GPC column ID. If the GPC column ID is more than nine characters, truncate at the ninth character.

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

3.3.8 Other Common Fields. Several other pieces of information are common to many of the data reporting forms. These include matrix, sample weight/volume, level, lab sample identifier, and lab file identifier.

- In the "Matrix" field, enter SOIL for soil/sediment samples and WATER for water samples.

  NOTE: The matrix shall be spelled out. Abbreviations such as S or W shall **not** be used.

- In the "Sample wt/vol" field, enter the number of grams (for soil) or milliliters (for water) of sample used in the first blank. Enter the units, either G or ML, in the second blank.

- The "Level" field is used for the volatile and semivolatile fractions. Enter the determination of concentration level made from the screening of soils. Enter as LOW or MED, **not** L or M. All water samples shall be entered as LOW.

  NOTE: There is no differentiation between low and medium soil samples for the pesticide/Aroclor fraction, and no level is entered on any of these forms.

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

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

3.3.8.1 The "Instrument ID" field is common to the forms containing calibration data. The identifier used by the Contractor shall include some indication of the manufacturer and/or model of the instrument, and shall contain additional characters that

differentiate between all instruments of the same type in the laboratory.

3.3.8.2 Forms II, IV, V, VIII, IX, and X contain a field labeled "page _ of _" in the bottom lefthand corner. If the number of entries required on any of these forms exceeds the available space, continue entries on another copy of the same fraction-specific form, duplicating all header information. If a second page is required, number the pages consecutively (i.e., "page 1 of 2" and "page 2 of 2"). If a second page is **not** required, number the page "page 1 of 1."

NOTE: These forms are fraction-specific, and often matrix-specific within a fraction. For example, Form II VOA-1 and Form II VOA-2 are for different data. Therefore, **do not** number the pages of all six versions of Form II as "1 of 6," "2 of 6," etc. Number only pages corresponding to the fraction-specific and matrix-specific form.

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

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

3.4.1 Purpose. This form is used for tabulating and reporting sample analysis, including blank, matrix spike, and matrix spike duplicate results for target compounds. If all fractions are not requested for analysis, only the pages for the fractions required shall be submitted. For example, if only volatiles analysis is requested, Form I, VOA-1, VOA-2 and Form I VOA-TIC shall be submitted. If only the pesticide/Aroclor fraction is requested for analysis, Form I PEST shall be submitted. **Furthermore, pesticide instrument blanks (PIBLKs) shall be reported on a per column/per analysis basis on Form I PEST.** Each PIBLK shall be named with a unique EPA sample number.

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

3.4.2.1 For soil samples analyzed for volatiles, enter the non-decanted percent moisture in the "% Moisture: not dec." field on Form I, VOA-1, VOA-2. This is the only percent moisture determination made for volatiles since the entire contents of the VOA vial are considered as the sample. For water samples, leave this field blank.

3.4.2.2 For soil samples analyzed for semivolatiles and pesticides/Aroclors, enter the values for the percent moisture determined during the analysis in the "% Moisture" field on Form I SV-1, SV-2, or PEST. In the "decanted (Y/N)" field, enter Y if the sample had standing water above the soil/sediment that was

decanted, or N if no water was decanted off the surface of the sample. Report percent moisture (decanted or not decanted) to the nearest whole percentage point (e.g., 5%, not 5.3%). For water samples, method blanks, sulfur cleanup blanks, and instrument blanks, leave these fields on Form I blank.

3.4.2.3 For volatiles, enter the GC column identifier in the "GC Column" field on Form I, VOA-1, VOA-2, and the internal diameter in millimeters (mm), to two decimal places, in the "ID" field. For packed columns, convert the internal diameter from inches to millimeters as necessary before entering in the "ID" field.

3.4.2.4 For *semivolatiles and* pesticides/Aroclors, enter the method of extraction in the "Extraction" field on Form I *SV-1, SV-2, SV-TIC, and* PEST as SEPF for separatory funnel, CONT for continuous liquid-liquid extraction *without hydrophobic membrane, CONH for continuous liquid-liquid extraction with hydrophobic membrane,* SONC for sonication (soils only), *SOXH for Automated Soxhlet Extraction (soils only), or PFEX for Pressurized Fluid Extraction (soils only).*

3.4.2.5 If gel permeation chromatography (GPC) was performed, enter Y in the "GPC Cleanup" field on Form I SV-1, SV-2, or PEST. Enter N in this field if GPC was not performed.

NOTE: GPC is **required** for all **soil** samples analyzed for semivolatiles and pesticides/Aroclors; therefore, all forms for soil samples will contain a Y in this field.

3.4.2.6 For soil samples only, enter the pH for semivolatiles and pesticides/Aroclors, reported to 0.1 pH units, on Form I SV-1, SV-2, or PEST.

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

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

3.4.2.9 If a medium soil sample is analyzed for volatiles, enter total volume of the methanol extract in microliters (uL) in the "Soil Extract Volume" field on Form I, VOA-1, VOA-2. This volume includes any methanol not collected from the filtration of the

extract through glass wool; the volume is typically 10,000 uL (i.e., the 10 mL of methanol used for the extraction). If a medium soil sample is analyzed, enter the volume of the methanol extract added to the reagent water in the purge tube and analyzed in the "Soil Aliquot Volume" field. Enter this volume in microliters (uL).

3.4.2.10 For semivolatiles and pesticides/Aroclors, enter the actual volume of the **most** concentrated sample extract, in microliters (uL), in the "Concentrated Extract Volume" field on Form I SV-1, SV-2 or PEST. For semivolatiles, this volume will typically be 1,000 uL (for water) or 500 uL (for water and soil) when GPC is performed. For pesticides/Aroclors, the volume of the most concentrated extract will typically be 10,000 uL (for water) or 5,000 uL (for water and soil) when GPC is performed. For pesticides/Aroclors, the volume of the most concentrated extract is **not** the volume taken through the Florisil and sulfur cleanup steps. If a dilution of the sample extract is made in a subsequent analysis, this volume will remain the same, but the dilution factor will change.

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

NOTE: A 2.0 microliter injection is **required** for semivolatile analyses.

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

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

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

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

3.4.2.16 For volatile and semivolatile results, report analytical results to one significant figure if the value is less than 10, and two significant figures if the value is 10 or above. Report all pesticide/Aroclor results to two significant figures.

3.4.2.17 Enter the appropriate concentration units, ug/L or ug/Kg.

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

The EPA-defined qualifiers to be used are:

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

J: This flag indicates an estimated value. This flag is used (1) when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, (2) when the mass spectral and retention time data indicate the presence of a compound that meets the volatile and semivolatile GC/MS identification criteria, and the result is less than the CRQL but greater than zero, and (3) when the retention time data indicate the presence of a compound that meets the pesticide/Aroclor identification criteria, and the result is less than the CRQL 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.

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

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

P: This flag is used for a pesticide/Aroclor target analyte when there is greater than 25% difference for detected concentrations between the two GC columns (see Form X). **The lower of the two values is reported on Form I and flagged with a P.**

C: This flag applies to pesticide results where the **identification** has been confirmed by GC/MS. If GC/MS confirmation was attempted but was unsuccessful, do **not** apply this flag; use a laboratory-defined flag instead (see the X qualifier).

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

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

E: This flag identifies compounds whose concentrations exceed the upper level of the calibration range of the instrument for that specific analysis. If one or more compounds have a response greater than the upper level of the calibration range, the sample or extract shall be diluted and reanalyzed according to the specifications in Exhibit D; exceptions are also noted in Exhibit D. All such compounds with a response greater than the upper level of the calibration range shall have the concentration flagged with an E on Form I for the original analysis.

NOTE: For total *xylene*, where three isomers are quantified as two peaks, the calibration range of **each peak** shall be considered separately. For example, a diluted analysis is **not** required for total *xylene* unless the concentration of the peak representing the single isomer exceeds 200 ug/L or the peak representing the two co-eluting isomers on that GC column exceeds 400 ug/L.

D: If a sample or extract is reanalyzed at a higher dilution factor, for example when the concentration of an analyte exceeds the upper calibration range, the DL suffix is appended to the sample number on Form I for the more diluted sample, and **all** reported concentrations on that Form I are flagged with the D flag. This flag alerts data users that any discrepancies between the reported concentrations may be due to dilution of the sample or extract. NOTE 1: The D flag is not applied to compounds which are not detected in the sample analysis (i.e., compounds reported with the CRQL and the U flag). NOTE 2: Separate Form Is are required for reporting the original analysis (EPA Sample No. XXXXX) and the more diluted sample analysis (EPA Sample No. XXXXXDL) (i.e., the results from both analyses cannot be combined on a single Form I).

A: This flag indicates that a tentatively identified compound is a suspected aldol-condensation product.

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

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

3.5.1 Purpose. This form is used to report analysis results for non-target compounds (e.g., compounds not listed in Exhibit C), excluding system monitoring compounds, surrogates, and internal standards. See Exhibit D for instructions on identification and quantitation. The Contractor shall submit Form I VOA-TIC or SV-TIC for **every analysis**, including required dilutions and reanalyses, even if no TICs are found.

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

3.5.2.1 Report all TICs including CAS number (if applicable), compound name, retention time, and the estimated concentration as uncorrected for blank contaminants. If the analytical result is less than 10, report to one significant figure. If the analytical result is 10 or greater, report to two significant figures. (Criteria for reporting TICs are given in Exhibit D, Section 11). Retention time shall be reported in minutes and decimal minutes, **not** seconds or minutes:seconds. If, in the opinion of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown.

3.5.2.2 Total the number of TICs found, **including** aldol-condensation products (see Section 3.5.2.4), and enter this number in the "Number TICs found" field. If no TICs were found, enter 0 (zero).

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

3.5.2.4 Peaks that are suspected to be aldol-condensation reaction products (e.g., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be summarized on this form, flagged A, and included in the "Number TICs found" field. The peaks shall be counted as part of the 30 most intense non-target semivolatile compounds to be searched.

3.6 System Monitoring Compound Recovery (Form II, VOA-1, VOA-2)

3.6.1 Purpose. For volatiles, Form II, VOA-1, VOA-2 is used to report the recoveries of the system monitoring compounds added to each volatile sample, including dilutions and reanalyses, blank, matrix spike, and matrix spike duplicate. The system monitoring compounds are used to monitor the performance of the purge and trap-gas chromatograph-mass spectrometer system as a whole. Form II VOA is matrix-specific, so that system monitoring compound recoveries for water samples are reported on a different version of Form II than the recoveries for soil samples. Soil sample recoveries are further differentiated by concentration level.

3.6.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete one form for each level. **Do not** mix low and medium level samples on one form. NOTE: For volatile **soil** samples only, specify the level as LOW or MED. Complete the remainder of the form using the following instructions.

3.6.2.1 For each system monitoring compound listed in Table 1, report the percent recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.

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

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

3.6.2.4 Number all pages as described in Section 3.3.

Table 1
System Monitoring Compounds

Volatile System Monitoring Compounds	CAS Number
SMC 1: Toluene-d8 (TOL)	2037-26-5
SMC 2: Bromofluorobenzene (BFB)	460-00-4
SMC 3: 1,2-Dichloroethane-d4 (DCE)	17060-07-0

3.7 Surrogate Recovery (Form II, SV-1, SV-2 and Form II, PEST-1, PEST-2)

3.7.1 Purpose. Form II, SV-1, SV-2 and Form II, PEST-1, PEST-2 are used to report the recoveries of the surrogate compounds added to each semivolatile and pesticide/Aroclor sample, blank, matrix spike, and matrix spike duplicate. Form II SV and Form II PEST are matrix-specific as well as fraction-specific, so surrogate recoveries

for semivolatile and pesticide water samples are reported on a different version of Form II than surrogate recoveries for semivolatile and pesticide soil samples.

3.7.2 Instructions. Complete the header information according to the instructions in Section 3.3. NOTE: For **semivolatile soil** samples only, specify the level as LOW or MED. Complete one form for each level. **Do not** mix low and medium level samples on one form. Complete the remainder of the form using the following instructions.

3.7.2.1 For each surrogate listed in Tables 2 and 3, report the percent recovery to the nearest whole percentage point.

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

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

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

3.7.2.5 The pesticide surrogate recoveries shall be reported from **both** GC columns used for the analyses. Therefore, identify each GC column at the top of Form II, PEST-1. PEST-2, entering the stationary phase in the "GC Column" field, and the internal diameter of the column in millimeters (mm) in the "ID" field.

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

3.7.2.7 Although the pesticide surrogate recovery limits for samples, matrix spike and matrix spike duplicates are only advisory, the Contractor shall flag those recoveries that are outside the advisory QC limits or are diluted out. The total number of recoveries that are outside the QC limits shall include all values from both of GC columns. In counting the total number of recoveries that are outside the QC limits, do not include the results flagged with a D.

3.7.2.8 Number all pages as described in Section 3.3.

Table 2
Semivolatile Surrogates

Semivolatile Surrogates	CAS Number
S1: Nitrobenzene-d5 (NBZ)	4165-60-0
S2: 2-Fluorobiphenyl (FBP)	321-60-8
S3: Terphenyl-d14 (TPH)	98904-43-9
S4: Phenol-d5 (PHL)	4165-62-2
S5: 2-Fluorophenol (2FP)	367-12-4
S6: 2,4,6-Tribromophenol (TBP)	118-79-6
S7: 2-Chlorophenol-d4 (2CP)	93951-73-6
S8: 1,2-Dichlorobenzene-d4 (DCB)	2199-69-1

Table 3
Pesticide Surrogates

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

3.8 Matrix Spike/Matrix Spike Duplicate Recovery (Form III, All Fractions)

3.8.1 Purpose. This form is used to report the results of the analyses of matrix spikes and matrix spike duplicates (MS/MSD). The form is matrix-specific for volatiles, semivolatiles, and pesticides.

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

3.8.2.1 For volatile and semivolatile soil samples, specify level as LOW or MED on Form III, VOA-2, and SV-2. SDGs containing soil samples at both levels require a MS/MSD at each level; therefore, for soils, prepare one form for each level.

3.8.2.2 In the first table under the "SPIKE ADDED" column, enter the calculated concentration in ug/L or ug/Kg (according to the matrix) that results from dividing each spike compound amount added to the aliquot weight/volume chosen for the matrix spike. For instance, for base/neutral compounds in medium level soils, if 50 ug of spike are added to 1 g of soil, the resulting concentration is 50,000 ug/Kg.

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

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

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

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

3.8.2.7 For pesticide/Aroclor matrix spikes and matrix spike duplicates, the MS concentration and MSD concentration shall be the concentration of the spiked analyte reported on Form I for those analyses. Of the two concentrations calculated for each pesticide/Aroclor target compound, one on each GC column, the **lower** concentration shall be reported on Form I, and **both** concentrations shall be reported on Form X. The **lower** concentration is also reported on Form III and used in the calculation of spike recovery, even if that concentration yields a recovery value that is outside the advisory QC limits.

3.8.2.8 Follow Sections 3.8.2.2 through 3.8.2.7 to complete the lower table, using the results of the analysis of the MSD aliquot.

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

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

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

3.9 Method Blank Summary (Form IV, All Fractions)

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

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

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

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

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

3.9.2.4 For volatiles, indicate the purging method by entering Y for heated purge or N for ambient temperature purge in the "Heated Purge: Y/N" field on Form IV VOA.

3.9.2.5 For pesticide/Aroclor blanks, enter the method of extraction as SEPF for separatory funnel, *CONH for continuous liquid-liquid extraction with hydrophobic membrane,* CONT for continuous liquid-liquid extraction *without hydrophobic membrane,* SONC for sonication, *SOXH for automated soxhlet extraction or PFEX for pressurized fluid extraction* on Form IV PEST.

3.9.2.6 For semivolatile and pesticide/Aroclor method blanks, enter the date of extraction of the blank on Form IV SV or PEST.

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

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

3.9.2.9 For pesticide/Aroclor fraction, enter the lab file identifier only if GC/MS confirmation was attempted. Otherwise, leave this field blank.

3.9.2.10 Number all pages as described in Section 3.3.

3.10 GC/MS Instrument Performance Check and Mass Calibration (Form V VOA and Form V SV)

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

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

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

3.10.2.2 For volatiles, identify the GC column and internal diameter on Form V VOA.

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

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

3.10.2.4 All relative abundances shall be reported as a number. If the relative abundance is 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.

3.10.2.5 In the lower table, list all samples, including dilutions and reanalyses, standards, blanks, matrix spikes, and matrix spike duplicates analyzed under that instrument performance check in chronological order, by time of analysis (in military time). Refer to Section 3.3.7 for specific instructions for identifying standards and blanks.

3.10.2.6 Complete the following fields for all standards, samples, including dilutions and reanalyses, blanks, matrix spikes, and matrix spike duplicates: "EPA Sample No.," "Lab Sample ID," "Lab File ID," "Date Analyzed," and "Time Analyzed."

3.10.2.7 Number all pages as described in Section 3.3.

3.11 GC/MS Initial Calibration Data (Form VI, VOA-1, VOA-2 and Form VI, SV-1, SV-2)

3.11.1 Purpose. After a GC/MS system has undergone an initial five-point[3] calibration at the specific concentration levels described in Exhibit D, and after all initial calibration criteria have been met, the Contractor shall complete and submit this form for each volatile or semivolatile target compound initial calibration performed which is relevant to the samples, including dilutions and reanalyses, blanks, matrix spikes, or matrix spike duplicates in the SDG, regardless of when that calibration was performed.

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

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

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

3.11.2.3 For volatiles, complete the "GC Column" and "ID" fields. *Indicate the purging method by entering "Y" for heated purge or "N" for ambient temperature purge in the "Heated Purge: (Y/N)" field.*

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

3.11.2.5 For volatiles, report the relative response factors for the system monitoring compounds in the calibration standards. For semivolatiles, report the response factors for all surrogate

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[3]For semivolatiles, eight compounds (2,4-Dinitrophenol, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, 4,6-Dinitro-2-Methylphenol, and Pentachlorophenol) will only require a four-point initial calibration at 50, 80, 120, and 160 total nanograms because detection at less than 50 ng per injection is difficult. If a four-point calibration is performed for these compounds, leave the "RRF20" column blank.

compounds in the calibration standards. The Contractor shall report the relative standard deviation (%RSD) for **all** compounds. See Exhibit D for equations.

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

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

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

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

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

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

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

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

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

Mixture A and the windows are only required to be calculated for Individual Mixture A.

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

3.12.2.8 For the multicomponent target compounds, the retention times, retention time windows, and calibration factors shall be reported in a similar fashion for each single point calibration standard. For each multicomponent compound, the Contractor shall select at least three peaks from each analyte, according to the specifications in Exhibit D. The retention time and calibration factor data apply to **each** peak. Complete one version of Form VI PEST-3 for each GC column, for each initial calibration that applies to samples in the data package.

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

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

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

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

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

of adjacent analytes have been entered. NOTE: Only eight of the nine resolution fields will be filled.

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

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

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

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

3.12.4.5.3 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered. NOTE: The last resolution field will be left blank in each table.

3.13 GC/MS Continuing Calibration Data (Form VII, VOA-1, VOA-2 and Form VII, SV-1, SV-2)

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

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

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

3.13.2.2 For volatiles, enter the purge method, GC column identifier, and internal diameter. *For semivolatiles, enter GC column identifier and internal diameter.*

3.13.2.3 Using the appropriate initial calibration (volatile or semivolatile), enter the average relative response factor (RRF) for each target compound, for each system monitoring compound for volatiles, and for each surrogate for semivolatiles.

3.13.2.4 Report the relative response factor (RRF50) from the continuing calibration standard analysis.

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

3.14 GC/EC Calibration Verification Summary (Form VII, PEST-1, PEST-2)

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

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

FORM VII PEST-1

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

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

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

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

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

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

3.14.2.7 Enter the nominal amount of each analyte in the PEM in the "NOM AMOUNT" column.

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

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

FORM VII PEST-2

3.14.2.10 The upper table on Form VII PEST-2 contains the retention time and amount data for Individual Standard Mixture A compounds. The lower table contains the data for Mixture B. Complete the form using the instructions in Sections 3.14 2.1 through 3.14.2.8 for Form VII PEST-1.

3.15 Internal Standard Area and RT Summary (Form VIII VOA and Form VIII, SV-1, SV-2)

3.15.1 Purpose. This form is used to summarize the peak areas and retention times of the internal standards added to all volatile and semivolatile samples, including: dilutions, reanalyses, blanks, matrix spikes, and matrix spike duplicates. The data are used to determine when changes in internal standard responses will adversely affect quantification of target compounds. This form shall be completed each time a continuing calibration is performed, or when samples are analyzed under the same GC/MS instrument performance check as an initial calibration.

3.15.2 Instructions. Complete the header information according to Section 3.3. Complete the remainder of the form using the following instructions. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a continuing calibration, Form VIII shall be completed on the basis of the internal standard areas of the 50 ug/L initial calibration

standard for volatiles, and the 50 ng initial calibration standard for semivolatiles. Use the date and time of analysis of this standard and the lab file identifier and areas in place of those of a continuing calibration standard.

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

3.15.2.2 For volatiles, enter the purge method, GC column identifier, and internal diameter. *For semivolatiles, enter GC column identifier and internal diameter.*

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

3.15.2.4 For each internal standard listed in Tables 4 and 5, calculate the upper limit of the area as the area of the particular standard plus 100 percent of its area (i.e., two times the area in the "12 HOUR STD" field), and the lower limit of the area as the area of the internal standard minus 50 percent of its area (i.e., one half the area in the "12 HOUR STD" field). Report these values in the "UPPER LIMIT" and "LOWER LIMIT" rows, respectively. Calculate the upper limit of the retention time as the retention of the internal standard plus 0.50 minutes (30 seconds), and the lower limit of the retention time as the retention time in the standard minus 0.50 minutes (30 seconds).

3.15.2.5 For each sample, including dilutions, reanalyses, blanks, matrix spikes, and matrix spike duplicates, analyzed under a given continuing calibration, enter the EPA sample number and the area measured for each internal standard and its retention time. If the internal standard area is outside the upper or lower limits calculated in step 4, flag that area with an asterisk (*). The asterisk shall be placed in the far righthand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the retention time of any internal standard that is outside the limits with an asterisk.

3.15.2.6 Number all pages as described in Section 3.3.

Table 4
Volatile Internal Standards

Volatile Internal Standards	CAS Number
IS1: Bromochloromethane (BCM)	74-97-5
IS2: 1,4-Difluorobenzene (DFB)	540-36-3
IS3: Chlorobenzene-d5 (CBZ)	3114-55-4

Table 5
Semivolatile Internal Standards

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

3.16 Pesticide Analytical Sequence (Form VIII PEST)

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

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

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

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

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

3.16.2.4 For every analysis associated with a particular analytical sequence starting with the initial calibration, enter the EPA sample number, lab sample identifier, and date and time of analysis. Each sample analyzed as part of the sequence shall be reported on Form VIII PEST **even** if it is not associated with the SDG. The Contractor shall use ZZZZZ as the EPA sample number to distinguish all samples that are not part of the SDG being reported.

3.16.2.5 Report the retention time of the surrogates for each analysis in the "TCX RT" and "DCB RT" columns. All sample analyses shall be bracketed by acceptable analyses of instrument blanks, a PEM, and Individual Standard Mixtures A and B. Given the fact that the initial calibration may remain valid for some time (see Exhibit D), it is only necessary to report the data from 12-hour periods when samples, dilutions, reanalyses, matrix spike, matrix spike duplicate, blanks, or multicomponent analytes for the 72 hour

confirmation requirement in an SDG were analyzed. *All data necessary to demonstrate compliance with the requirements specified in Exhibit D-Pest Section 9.3 must be reported.* The Contractor shall submit Form VIII for the initial calibration sequence and forms that include the PEMs and Individual Standard Mixtures that bracket **any** and **all** samples in the SDG. While the data for time periods between the initial calibration and samples in the SDG are not a routine deliverable, the data shall be available as requested (e.g., at on-site evaluations). Non-EPA samples *or samples from SDGs not being reported* shall be numbered ZZZZZ.

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

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

3.17 Pesticide Cleanup Summary (Form IX, PEST-1, PEST-2)

3.17.1 Purpose: This form summarizes the results of the checks performed for both cleanup procedures employed during the preparation of pesticide extracts for analysis. Form IX PEST-1 is used to report the results of the check of the Florisil cartridges used to process all sample extracts and to associate the lot of cartridges with particular sample results so that problems with a particular cartridge lot may be tracked across all associated samples. Form IX PEST-2 summarizes the results of the calibration *verification* of the Gel Permeation Chromatography (GPC) device that shall be used to process all soil sample extracts for pesticide/Aroclor analyses.

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

FORM IX PEST-1

3.17.2.1 Enter the Florisil cartridge lot number.

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

3.17.2.3 Complete the "GC Column" and "ID" fields for the two GC columns used to analyze the samples, including blanks, matrix spikes, and

matrix spike duplicates. Report all results from either GC column 1 or GC column 2.

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

3.17.2.5 Calculate the percent recovery to the nearest whole percent, and enter the number in the "% REC" field. Flag each spike recovery outside the QC limits (shown on the form) with an asterisk (*). The asterisk shall be placed in the last space in the "% REC" column, under the "#" symbol.

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

3.17.2.7 Number the pages as described in Section 3.3.

FORM IX PEST-2

3.17.2.8 On Form IX PEST-2, enter an identifier for the GPC column and the *analysis* date of calibration *verification* in the appropriate fields.

3.17.2.9 Complete the "GC Column" and "ID" fields as on Form IX PEST-1 for florisil. Report all results from a single column.

3.17.2.10 For each of the pesticide matrix spike compounds listed in the first table, enter the amount of the spike added to the GPC column and the amount recovered, in nanograms (ng).

3.17.2.11 Calculate the percent recovery of each analyte, and enter these values on the form, to the nearest percent. Compare the recoveries to the QC limits shown on the form, and flag all those values outside the limits with an asterisk (*) in the "% REC" column under the "#" symbol.

3.17.2.12 For each sample in the data package that was subjected to GPC under this calibration *verification*, enter the EPA sample number, lab sample identifier, and the date of **both** analyses in the second table.

3.17.2.13 If more than one copy of Form IX PEST-2 is required, number all pages as described in Section 3.3.

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

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

forms are used for single component analytes and multicomponent analytes.

Form X is required for each sample, including dilutions and reanalyses, blank, matrix spike, and matrix spike duplicate in which compounds listed in Exhibit C (Pesticides/Aroclors) are reported on Form I. **Do not generate a Form X for pesticide instrument blanks.**

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

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

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

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

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

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

3.18.2.6 Enter the concentration calculated from each GC column under the "CONCENTRATION" column. Although the units are the same as those used on Form I, ug/L for water samples and ug/Kg for soil samples, do **not** enter any units on Form X.

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

3.18.2.8 The **lower** of the two concentrations is reported on Form I for each pesticide compound. The lower concentration is used because, if present, coeluting interferences are likely to increase the calculated concentration of any target compound. If the percent difference between the calculated concentrations is greater than 25.0 percent, flag the concentration on Form I, as described previously. This will alert the data user to the potential problems in quantitating this analyte.

3.18.2.9 If more pesticide compounds are identified in an individual sample than can be reported on one Form X, complete as many additional copies of Form X as necessary, duplicating all

header information and numbering the pages as described in Section 3.3.

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

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

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

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

3.19.2 Instructions

3.19.2.1 Sign and date the airbill (if present).

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

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

3.19.2.4 Record the custody seal numbers in item 2.

3.19.2.5 Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (e.g., Traffic Reports, Packing Lists), and airbills or airbill stickers in items 3-5. Specify if there is an airbill present or an airbill sticker in item 5. Record the airbill or sticker number in item 6.

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

3.19.2.7 Record the cooler temperature in item 9.

3.19.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and circle the appropriate answer in item *10*.

3.19.2.9 Record the date and time of cooler receipt at the laboratory in items *11 and 12*.

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

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

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

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

3.19.2.14 Cross out unused columns and spaces.

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

3.20 Document Inventory Sheet (Form DC-2)

3.20.1 Purpose. The Document Inventory Sheet (Form DC-2) is used to record both the inventory of Complete SDG File (CSF) documents and the number of documents in the original sample data package which is sent to the EPA Region.

3.20.2 Instructions

3.20.2.1 Organize all EPA CSF documents as described in Exhibit B, Sections II and III. Assemble the documents in the order specified on Form DC-2 and Sections II and III, and stamp each page with a consecutive number; however, do not number Form DC-2. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (for example, "page numbers not used, XXXX - XXXX, XXXX - XXXX." If

there are no documents for a specific document type, enter a "NA" in the empty space.

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

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

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

EXHIBIT C

TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

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

All CRQLs are rounded to two significant figures.

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

For soil samples, the moisture content of the samples must be used to adjust the CRQL values appropriately.

Exhibit C - Target Compound List and Contract Required Quantitation Limits

Table of Contents

1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Volatiles	CAS Number	Quantitation Limits Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg	On Column (ng)
1.	*Dichlorodifluoromethane*	*75-71-8*	*10*	*10*	*1200*	*(50)*
2.	Chloromethane	74-87-3	10	10	1200	(50)
3.	Vinyl Chloride	75-01-4	10	10	1200	(50)
4.	Bromomethane	74-83-9	10	10	1200	(50)
5.	Chloroethane	75-00-3	10	10	1200	(50)
6.	*Trichlorofluoromethane*	*75-69-4*	*10*	*10*	*1200*	*(50)*
7.	1,1-Dichloroethene	75-35-4	10	10	1200	(50)
8.	*1,1,2-Trichloro-1,2,2-trifluoroethane*	*76-13-1*	*10*	*10*	*1200*	*(50)*
9.	Acetone	67-64-1	10	10	1200	(50)
10.	Carbon Disulfide	75-15-0	10	10	1200	(50)
11.	*Methyl Acetate*	*79-20-9*	*10*	*10*	*1200*	*(50)*
12.	Methylene Chloride	75-09-2	10	10	1200	(50)
13.	*trans-1,2-Dichloroethene*	*156-60-5*	10	10	1200	(50)
14.	*tert-Butyl Methyl Ether*	*1634-04-4*	*10*	*10*	*1200*	*(50)*
15.	1,1-Dichloroethane	75-34-3	10	10	1200	(50)
16.	*cis-1,2-Dichloroethene*	*156-59-2*	10	10	1200	(50)
17.	2-Butanone	78-93-3	10	10	1200	(50)
18.	Chloroform	67-66-3	10	10	1200	(50)
19.	1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
20.	*Cyclohexane*	*110-82-7*	*10*	*10*	*1200*	*(50)*
21.	Carbon Tetrachloride	56-23-5	10	10	1200	(50)
22.	Benzene	71-43-2	10	10	1200	(50)
23.	1,2-Dichloroethane	107-06-2	10	10	1200	(50)
24.	Trichloroethene	79-01-6	10	10	1200	(50)
25.	*Methylcyclohexane*	*108-87-2*	*10*	*10*	*1200*	*(50)*
26.	1,2-Dichloropropane	78-87-5	10	10	1200	(50)
27.	Bromodichloromethane	75-27-4	10	10	1200	(50)
28.	cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
29.	4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
30.	Toluene	108-88-3	10	10	1200	(50)
31.	trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
32.	1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
33.	Tetrachloroethene	127-18-4	10	10	1200	(50)
34.	2-Hexanone	591-78-6	10	10	1200	(50)
35.	Dibromochloromethane	124-48-1	10	10	1200	(50)

	Volatiles	CAS Number	Quantitation Limits Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg	On Column (ng)
36.	*1,2-Dibromoethane*	*106-93-4*	*10*	*10*	*1200*	*(50)*
37.	Chlorobenzene	108-90-7	10	10	1200	(50)
38.	Ethylbenzene	100-41-4	10	10	1200	(50)
39.	Xylenes (total)	1330-20-7	10	10	1200	(50)
40.	Styrene	100-42-5	10	10	1200	(50)
41.	Bromoform	75-25-2	10	10	1200	(50)
42.	*Isopropylbenzene*	*98-82-8*	*10*	*10*	*1200*	*(50)*
43.	1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
44.	*1,3-Dichlorobenzene*	*541-73-1*	*10*	*10*	*1200*	*(50)*
45.	*1,4-Dichlorobenzene*	*106-46-7*	*10*	*10*	*1200*	*(50)*
46.	*1,2-Dichlorobenzene*	*95-50-1*	*10*	*10*	*1200*	*(50)*
47.	*1,2-Dibromo-3-chloropropane*	*96-12-8*	*10*	*10*	*1200*	*(50)*
48.	*1,2,4-Trichlorobenzene*	*120-82-1*	*10*	*10*	*1200*	*(50)*

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Semivolatiles	CAS Number	Quantitation Limits Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg	On Column (ng)
49.	*Benzaldehyde*	*100-52-7*	*10*	*330*	*10000*	*(20)*
50.	Phenol	108-95-2	10	330	10000	(20)
51.	bis-(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)
52.	2-Chlorophenol	95-57-8	10	330	10000	(20)
53.	2-Methylphenol	95-48-7	10	330	10000	(20)
54.	2,2'-oxybis(1-Chloropropane)[1]	108-60-1	10	330	10000	(20)
55.	*Acetophenone*	*98-86-2*	*10*	*330*	*10000*	*(20)*
56.	4-Methylphenol	106-44-5	10	330	10000	(20)
57.	N-Nitroso-di-n propylamine	621-64-7	10	330	10000	(20)
58.	Hexachloroethane	67-72-1	10	330	10000	(20)
59.	Nitrobenzene	98-95-3	10	330	10000	(20)
60.	Isophorone	78-59-1	10	330	10000	(20)
61.	2-Nitrophenol	88-75-5	10	330	10000	(20)
62.	2,4-Dimethylphenol	105-67-9	10	330	10000	(20)
63.	bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)
64.	2,4-Dichlorophenol	120-83-2	10	330	10000	(20)
65.	Naphthalene	91-20-3	10	330	10000	(20)
66.	4-Chloroaniline	106-47-8	10	330	10000	(20)
67.	Hexachlorobutadiene	87-68-3	10	330	10000	(20)
68.	*Caprolactam*	*105-60-2*	*10*	*330*	*10000*	*(20)*
69.	4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)
70.	2-Methylnaphthalene	91-57-6	10	330	10000	(20)
71.	Hexachlorocyclo-pentadiene	77-47-4	10	330	10000	(20)
72.	2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)
73.	2,4,5-Trichlorophenol	95-95-4	25	830	25000	(50)

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

	Semivolatiles	CAS Number	Quantitation Limit Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg	On Column (ng)
74.	*1,1'-Biphenyl*	*92-52-4*	*10*	*330*	*10000*	*(20)*
75.	2-Chloronaphthalene	91-58-7	10	330	10000	(20)
76.	2-Nitroaniline	88-74-4	25	830	25000	(50)
77.	Dimethylphthalate	131-11-3	10	330	10000	(20)
78.	2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
79.	Acenaphthylene	208-96-8	10	330	10000	(20)
80.	3-Nitroaniline	99-09-2	25	830	25000	(50)
81.	Acenaphthene	83-32-9	10	330	10000	(20)
82.	2,4-Dinitrophenol	51-28-5	25	830	25000	(50)
83.	4-Nitrophenol	100-02-7	25	830	25000	(50)
84.	Dibenzofuran	132-64-9	10	330	10000	(20)
85.	2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
86.	Diethylphthalate	84-66-2	10	330	10000	(20)
87.	Fluorene	86-73-7	10	330	10000	(20)
88.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
89.	4-Nitroaniline	100-01-6	25	830	25000	(50)
90.	4,6-Dinitro-2-methylphenol	534-52-1	25	830	25000	(50)
91.	N-Nitroso diphenylamine	86-30-6	10	330	10000	(20)
92.	4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
93.	Hexachlorobenzene	118-74-1	10	330	10000	(20)
94.	*Atrazine*	*1912-24-9*	*10*	*330*	*10000*	*(20)*
95.	Pentachlorophenol	87-86-5	25	830	25000	(50)
96.	Phenanthrene	85-01-8	10	330	10000	(20)
97.	Anthracene	120-12-7	10	330	10000	(20)
98.	Carbazole	86-74-8	10	330	10000	(20)
99.	Di-n-butylphthalate	84-74-2	10	330	10000	(20)
100.	Fluoranthene	206-44-0	10	330	10000	(20)
101.	Pyrene	129-00-0	10	330	10000	(20)
102.	Butylbenzylphthalate	85-68-7	10	330	10000	(20)
103.	3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
104.	Benzo(a)anthracene	56-55-3	10	330	10000	(20)
105.	Chrysene	218-01-9	10	330	10000	(20)
106.	bis(2-Ethylhexyl) phthalate	117-81-7	10	330	10000	(20)
107.	Di-n-octylphthalate	117-84-0	10	330	10000	(20)

	Semivolatiles	CAS Number	Quantitation Limits Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg	On Column (ng)
108.	Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
109.	Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
110.	Benzo(a)pyrene	50-32-8	10	330	10000	(20)
111.	Indeno(1,2,3-cd)-pyrene	193-39-5	10	330	10000	(20)
112.	Dibenzo(a,h)-anthracene	53-70-3	10	330	10000	(20)
113.	Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)

3.0 PESTICIDES/AROCLORS TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[2]

	Pesticides/Aroclors	CAS Number	Quantitation Limits Water µg/L	Soil µg/Kg	On Column (pg)
114.	alpha-BHC	319-84-6	0.050	1.7	5
115.	beta-BHC	319-85-7	0.050	1.7	5
116.	delta-BHC	319-86-8	0.050	1.7	5
117.	gamma-BHC (Lindane)	58-89-9	0.050	1.7	5
118.	Heptachlor	76-44-8	0.050	1.7	5
119.	Aldrin	309-00-2	0.050	1.7	5
120.	Heptachlor epoxide[3]	*1024-57-3*	0.050	1.7	5
121.	Endosulfan I	959-98-8	0.050	1.7	5
122.	Dieldrin	60-57-1	0.10	3.3	10
123.	4,4'-DDE	72-55-9	0.10	3.3	10
124.	Endrin	72-20-8	0.10	3.3	10
125.	Endosulfan II	33213-65-9	0.10	3.3	10
126.	4,4'-DDD	72-54-8	0.10	3.3	10
127.	Endosulfan sulfate	1031-07-8	0.10	3.3	10
128.	4,4'-DDT	50-29-3	0.10	3.3	10
129.	Methoxychlor	72-43-5	0.50	17	50
130.	Endrin ketone	53494-70-5	0.10	3.3	10
131.	Endrin aldehyde	7421-93-4	0.10	3.3	10
132.	alpha-Chlordane	5103-71-9	0.050	1.7	5
133.	gamma-Chlordane	5103-74-2	0.050	1.7	5
134.	Toxaphene	8001-35-2	5.0	170	500
135.	Aroclor-1016	12674-11-2	1.0	33	100
136.	Aroclor-1221	11104-28-2	2.0	67	200
137.	Aroclor-1232	11141-16-5	1.0	33	100
138.	Aroclor-1242	53469-21-9	1.0	33	100
139.	Aroclor-1248	12672-29-6	1.0	33	100
140.	Aroclor-1254	11097-69-1	1.0	33	100
141.	Aroclor-1260	11096-82-5	1.0	33	100

[2]There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides/Aroclors.

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

EXHIBIT D

ANALYTICAL METHODS
FOR PESTICIDES/AROCLORS

Exhibit D - Analytical Methods for Pesticides/Aroclors

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1.0 SCOPE AND APPLICATION

1.1 In 1978, EPA Headquarters and Regional representatives designed analytical methods for the analysis of chlorinated pesticides and Aroclors in hazardous waste samples. These methods were based on EPA Method 608, Organochlorine Pesticides and PCBs. In 1980, these methods were adopted for use in the Contract Laboratory Program (CLP). As the requirements of CERCLA evolved, the CLP methods, as well as their precedent EPA 600 Series methods, established the basis for other EPA methods to perform the analysis of chlorinated pesticides and Aroclors in hazardous waste samples (i.e., SW-846). The following CLP method has continuously improved to incorporate technological advancements promulgated by EPA, and has continued to set the standard for the preparation, extraction, isolation, identification, and reporting of chlorinated pesticides and Aroclors at hazardous waste sites.

1.2 The analytical method that follows is designed to analyze water, sediment, and soil from hazardous waste sites to determine the presence and concentration of the chlorinated pesticides and Aroclors found in the Target Compound List (TCL) in Exhibit C. The method can be used for determining analyte concentrations in the range from the contract required quantitation limits (CRQL) to one million times the CRQL in these matrices when appropriate dilutions are made. The method *includes* sample extraction, extract cleanup techniques and GC/EC analytical methods for pesticides and Aroclors.

1.3 This analytical method provides the use of SW-846 Methods 3541 (Revision 0, September 1994) and 3545 (Revision 0, December 1996) for the extraction of soil/sediment samples. However, prior to using either one of these alternate extraction procedures, the Contractor must first demonstrate that these procedures are equivalent to the existing procedures, and obtain approval for use of these alternate extraction procedures from the EPA CLP National Program Manager. The process for determining and documenting equivalency can be found in Exhibit E Section 6.0.

1.4 Problems, including resolution difficulties, have been associated with the following pairs of compounds using this method.

- On a DB-608 or equivalent column, DDE and dieldrin; methoxychlor and endrin ketone; and endosulfan I and gamma-Chlordane.

- On a DB-1701 or equivalent column, endosulfan I and gamma-Chlordane, and methoxychlor and endosulfan sulfate

1.5 There are two isomers of heptachlor epoxide, the endo isomer (isomer A) and the exo isomer (isomer B). The two isomers are separable using current GC capillary columns. Only the exo isomer (isomer B) is of environmental significance. This is the isomer that must be used as an analytical standard, identified and quantitated in sample analysis, and reported on appropriate forms as heptachlor epoxide.

2.0 SUMMARY OF METHOD

2.1 Water

Continuous liquid-liquid or separatory funnel extraction procedures are employed for aqueous samples. A 1 L volume of sample is spiked with the surrogate solution and extracted with methylene chloride using a separatory funnel or a continuous extractor. The methylene chloride extract is dried with anhydrous sodium sulfate, concentrated, and cleaned up by GPC (GPC is required when higher molecular weight compounds are present that interfere with the analyses of target compounds; GPC is optional for all other circumstances). The extract is then solvent exchanged into hexane, cleaned up by Florisil cartridges, and the final volume adjusted to 1 mL or 2 mL. The extract is analyzed using a dual column wide-bore capillary Gas Chromatography/Electron Capture (GC/EC) technique.

2.2 Soil/Sediment

A 30 g aliquot of sample is spiked with the surrogate and then mixed with anhydrous sodium sulfate and extracted with a 1:1 acetone/methylene chloride solvent mixture by sonication. The extract is filtered, concentrated and solvent-exchanged into methylene chloride. The methylene chloride extract is then cleaned up by GPC (mandatory), solvent-exchanged into hexane, cleaned up by Florisil cartridge, and adjusted to a final volume of 1 mL or 2 mL. The extract is analyzed using a dual column wide-bore capillary Gas Chromatography/Electron Capture (GC/EC) technique.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blank. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so 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 must be used to remove such interferences in order to achieve the contract required quantitation limits.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses. Specifically, concentrated sulfuric acid and the 10 N sodium hydroxide solution are moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

5.2 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the Aroclors. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here, but demonstration of equivalent performance meeting the requirements of this Statement of Work is the responsibility of the Contractor. The Contractor must document in its Narrative when it uses equipment and supplies other than those specified here.

6.1 Glassware

6.1.1 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) or Hydrophobic Membrane-based Extractor (Accelerated One Step™ Extractor, Corning series 3195, or equivalent).

6.1.2 Separatory Funnels - 2 L with Teflon stopcock.

6.1.3 Beakers - 400 mL.

6.1.4 Erlenmeyer Flasks - 250 mL.

6.1.5 Syringes - 10 mL with Luerlok fitting, 1 mL or 2 mL.

6.1.6 Vials and Caps - 20 mL and 10 mL (optional) with screw cap and Teflon or aluminum foil liner, 2 mL capacity for GC auto sampler.

6.1.7 Pipets - glass volumetric 1 mL or 2 mL.

6.1.8 Centrifuge Tube - 12 to 15 mL with 19 mm ground glass joint (optional).

6.1.9 Graduated Cylinder - 1 L capacity.

6.1.10 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).

6.1.11 Volumetric Flasks - 10 mL and 1 or 2 mL.

6.1.12 Bottle or Test Tube - 20 mL with Teflon-lined screw cap for sulfur removal and a glass bottle - 1 L volume, for use in preparation of Bio Beads for packing into a column.

6.1.13 Powder Funnels - 10 cm diameter, for filtration/drying.

6.1.14 Buchner Funnels - 9 cm diameter, for filtration.

6.2 Kuderna-Danish (K-D) Apparatus.

6.2.1 Concentrator Tubes - 10 mL, graduated (Kontes K-570040-1029, or equivalent).

6.2.2 Evaporative Flasks - 500 mL (Kontes K-470001-0500, or equivalent).

6.2.3 Snyder Column - three-ball macro (Kontes K-503000-0121, or equivalent).

6.2.4 Snyder Column - two-ball micro (Kontes K-569001 -0219, or equivalent).

6.3 Vacuum System for Eluting Multiple Cleanup Cartridges.

6.3.1 Vac Elute Manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.

6.3.2 Vacuum Trap - made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

6.3.3 Vacuum Pressure Gauge.

6.3.4 Rack for holding 10 mL volumetric flasks in the manifold.

NOTE: Other types of equivalent systems, such as an automated system using syringe pressure, are considered to be acceptable for elution of florisil cartridges, as long as all QC and sample technical acceptance criteria are met.

6.4 pH Paper - wide range (Hydrion Papers, Micro-essential Laboratory, Brooklyn, NY, or equivalent).

6.5 Spatula - stainless steel or Teflon.

6.6 Centrifuge - table top (optional).

6.7 Balances - top loading, capable of weighing accurately to ± 0.01 g, analytical, capable of weighing accurately to ± 0.0001 g. The balances must be calibrated with class S weights or known reference weights once per each 12-hour work shift. The balances must be calibrated with class S weights at a minimum of once per month. The balances must also be annually checked by a certified technician.

6.8 Ultrasonic Cell Disrupter - Heat Systems, Ultrasonics, Inc., Model W-385 (475 watt with pulsing capability, No. 207 3/4-inch tapered disruptor horn) or equivalent device with a minimum 375 watt output capability. NOTE: In order to ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. Erosion of the tip is evidenced by a rough surface.

6.9 Sonabox Acoustic Enclosure (or equivalent) - for use with disruptor to decrease noise level.

6.10 Filter Paper - No. 41 Whatmann (or equivalent), 9 cm circles (optional).

6.11 Pyrex Glass Wool - rinsed with methylene chloride and dried before use.

6.12 Boiling chips.

6.12.1 Silicon carbide boiling chips - approximately 10 to 40 mesh. Heat the chips to 400 °C for 30 minutes or solvent rinse before use.

6.12.2 Teflon boiling chips (optional) - solvent rinse the chips before use.

6.13 Water Bath - heated, with concentric ring cover, capable of temperature control. NOTE: The water bath should be used in a hood.

6.14 GPC Cleanup System

6.14.1 Gel Permeation Chromatography System - GPC Autoprep Model 1002 A or B, Analytical Biochemical Laboratories, Inc., or equivalent. Systems that perform satisfactorily have been assembled from the following components: an HPLC pump, an auto sampler or a valving system with sample loops, and a fraction collector. All systems, whether automated or manual, must meet the calibration requirements of Section 10.1.8.1. NOTE: GPC cleanup is required for extracts for all soils/sediments and for water extracts containing higher molecular weight contaminants that interfere with the analyses of the target compounds.

6.14.1.1 Chromatographic column - 700 mm x 25 mm ID glass column. Flow is upward. To simplify switching from the UV detector during calibration to the GPC collection device during extract cleanup, an optional double 3-way valve (Rheodyne Type 50 Teflon Rotary Valve #10-262 or equivalent) may be attached to that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

6.14.1.2 Guard column (optional) - 5 cm, with appropriate fittings to connect to the inlet side of the analytical column (Supelco 5-8319 or equivalent).

6.14.1.3 Bio Beads (SX-3) - 200 to 400 mesh, 70 g (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750, or equivalent). An additional 5 g of Bio Beads is required if the optional guard column is employed. The quality of Bio Beads may vary from lot to lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they can also pass through the column screens and damage the valve.

6.14.1.4 Ultraviolet detector - fixed wavelength (254 nm) with a semi-prep flow-through cell.

6.14.1.5 Strip chart recorder - recording integrator or laboratory data system.

6.14.1.6 Syringe filter assembly, disposable - Bio-Rad "Prep Disc" sample filter assembly #343-0005, 25 mm, and 5 micron filter discs or equivalent. Note: Some instrument manufacturer's recommend a smaller micron filter disc. Consult your instrument operation manual to determine the proper size filter disc to use in your system. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

6.15 Florisil - 500 mg or 1 g cartridges with stainless steel or Teflon frits, (catalog No. 694-313, Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent).

6.16 Nitrogen Evaporation Device - equipped with a heated bath that can be maintained at 35 to 40 °C (N-Evap by Organomation Associates, Inc., South Berlin, MA, or equivalent).

6.17 Oven - drying.

6.18 Desiccator.

6.19 Crucibles - porcelain crucibles or aluminum weighing pans.

6.20 pH Meter - with a combination glass electrode. Calibrate according to manufacturer's instructions. pH meter must be calibrated prior to each use.

6.21 Magnetic Stirrer Motor - Model PC 353, Corning Co., Corning, NY, or equivalent.

6.22 Magnetic Stirrer Bar - Teflon coated, at least 4 cm long.

6.23 Gas Chromatograph/Electron Capture Detector (GC/EC) System.

6.23.1 Gas Chromatograph - must adequately regulate temperature in order to give a reproducible temperature program and have a flow controller that maintains a constant column flow rate throughout temperature program operations. The system must be suitable for split less injection and have all required accessories including syringes, analytical columns, and gases.

6.23.2 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 the metal parts in the injector with dichlorodimethyl silane. In some cases, using a 0.25 inch packed column injector converted for use with 0.53 mm capillary columns works better than a Grob-type injector. If a Grob-type injector is used, a 4 mm liner may be required to meet breakdown criteria.

6.23.3 Gas Chromatograph Columns - two wide-bore (0.53 mm ID) fused silica GC columns are required. A separate detector is required for each column. The specified analytical columns are a 30 m x 0.53 mm ID, 1.0 µm film thickness, DB-1701 (J&W Scientific); SPB 1701 (Supelco); AT 1701 (Alltech); RTX-1701 (Restek); CP-Sil 19CB (Chrompack); 007-1701 (Quadrex); BP-10 (SGE); or equivalent, and a 30 m x 0.53 mm ID, 0.5 to 1.0 µm film thickness DB-608 (J&W Scientific); HP-608 (Hewlett Packard); SPB-608 (Supelco); 007-608 (Quadrex); BP-608 (SGE); CP-Sil 8CB (Chrompack); or equivalent. NOTE: The column length stated above is the minimum requirement. Longer columns that meet resolution and calibration requirements may be used. A description of the GC columns used for analysis shall be provided in the SDG Narrative.

6.23.3.1 PACKED COLUMNS CANNOT BE USED

6.23.3.2 A capillary column is considered equivalent if:

- The column does not introduce contaminants which interfere with identification and quantitation of the compounds listed in Exhibit C (Pesticides)
- The analytical results generated using the column meet the initial calibration and calibration verification technical acceptance criteria listed in the SOW and the CRQLs listed in Exhibit C (Pesticides).
- The column can accept at least 16 times the low point standard for individual standard mixtures A and B for each compound listed in Exhibit C (Pesticides) without becoming overloaded.
- The column pair chosen must have dissimilar phases/chemical properties in order to separate the compounds of interest in different RT order.

6.23.3.3 Although the instructions included in the SOW are for wide bore capillary columns, narrower bore capillary columns may be evaluated for use.

6.23.3.4 As applicable, follow the manufacturer's instructions for use of its product.

6.23.3.5 The Contractor must maintain documentation that the column met the criteria in Section 6.23.3.2. The minimum documentation is as follows:

6.23.3.5.1 Manufacturer provided information concerning the performance characteristics of the column;

6.23.3.5.2 Chromatograms and data system reports generated on the GC/ECD and used for CLP analyses:

- From instrument blanks which demonstrate that there are no contaminants which interfere with the pesticide analysis when using the alternate columns;
- For initial calibration standards analyzed using the column;
- For calibration verification standards analyzed using the alternate column.

6.23.3.5.3 Based on the Contractor generated data described in Section 6.23.3.5.2, the Contractor must complete a written review, aligned by the Laboratory Manager certifying that:

- The column performance is comparable to the required column performance in its ability to produce initial calibration and calibration verifications which meet the technical acceptance criteria in Sections 9.2.5 and 9.3.5.

- The low point initial calibration standard analyses have adequate sensitivity to meet the pesticide CRQLs.

- The high point initial calibration standard analyses were not overloaded.

- The column does not introduce contaminants which interfere with identification and quantitation of compounds listed in Exhibit C (Pesticides).

6.23.3.5.4 The documentation must be made available to the Agency during on-site laboratory evaluations or sent to the Agency upon request of the Technical Project Officer or the Administrative Project Officer.

6.23.3.6 Columns are mounted in a 0.25-inch injector ports by using glass adapters available from a variety of commercial sources (J&W Scientific, Supelco, Inc., Hewlett-Packard, Varian, Inc., Perkin Elmer, or equivalent). The two columns may be mounted into a single injection port with a tee adapter (Supelco, Inc., Bellefonte, PA, Catalog No. 2-3660, or equivalent). Use of this adapter allows simultaneous injection onto both columns. The laboratory should follow manufacturer's recommendations for mounting 0.53 mm capillary columns in injector ports.

6.23.3.7 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 the Agency. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of a hydrogen leak detector is highly recommended when hydrogen is used as the carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with rubber components are not to be used.

6.23.4 Electron Capture Detector (ECD) - the linearity of the response of the ECD may be greatly dependent on the flow rate of the make-up gas. The make-up gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector. The GC/EC system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants which may interfere with the analysis. The instrument must be vented to outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.23.5 Data System - a data system must be interfaced to the GC/EC. 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.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Reagent water - defined as water in which no interferant is observed at one-half the CRQL of any pesticide/Aroclor when one liter of the reagent water is extracted and prepared by using the same workup procedure as for a water sample.

7.1.2 Sodium sulfate - granular-anhydrous reagent grade, heated at 400 °C for 4 hours, or at 120 °C for 16 hours, cooled in a desiccator, and stored in a glass bottle. Each lot must be extracted with hexane and analyzed by GC/EC to demonstrate that it is free of interference before use. Baker anhydrous granular, Catalog No. 3375, or equivalent. CAUTION: An open container of sodium sulfate may become contaminated during storage in the laboratory.

7.1.3 Concentrated sulfuric acid (H_2SO_4)- 18 N.

7.1.4 Sodium hydroxide solution (NaOH) (10 N) - carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 mL.

7.1.5 10 percent acetone in hexane (v/v) - prepare by adding 10 mL of acetone to 90 mL of hexane. NOTE: Prepare this mixture accurately or the results from the Florisil cartridge cleanup will be adversely affected. Water in the acetone also will adversely affect Florisil performance.

7.1.6 Methylene chloride, hexane, acetone, toluene, iso-octane, and methanol (optional) - pesticide quality or equivalent. It is recommended that each lot of solvent used be analyzed to demonstrate that it is free of interference before use. Methylene chloride must be certified as acid free or must be tested to demonstrate that it is free of hydrochloric acid. Acidic methylene chloride must be passed through basic alumina and then demonstrated to be free of hydrochloric acid.

7.1.7 Mercury - triple distilled, for sulfur cleanup.

7.1.8 Copper powder (optional) - fine, granular (Mallinckrodt 4649 or equivalent). Copper may be used instead of mercury for sulfur cleanup. Remove oxides by treating with dilute nitric acid, rinse with distilled water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

7.2 Standards

7.2.1 Introduction

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

7.2.2 Stock standard solutions (1 µg/µL) - can be prepared from pure standard materials or purchased as certified solutions.

7.2.2.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in toluene and dilute to volume in a 10 mL volumetric flask with toluene or acetone. Larger volumes may be used at the convenience of the analyst.

7.2.2.2 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. (See Exhibit E Analytical Standards Requirements.)

7.2.2.3 Fresh stock standards must be prepared once every six months, or sooner if standards have degraded or concentrated. Stock standards must be checked for signs of degradation or concentration just prior to preparing working standards from them.

7.2.3 Secondary Dilution Standards

Using stock standards, prepare secondary dilution standards in acetone that contain the compounds of interest either singly or mixed together. Fresh secondary dilution standards must be prepared once every six months, or sooner if standards have degraded or concentrated. Secondary dilution standards must be checked for signs of degradation or concentration just prior to preparing working standards from them.

7.2.4 Working Standards

7.2.4.1 Surrogate Standard Spiking Solution

The surrogates, tetrachloro-m-xylene and decachlorobiphenyl, are added to all standards, samples, matrix spikes, and blanks. Prepare a surrogate spiking solution of 0.2 µg/mL of each of the two compounds in acetone.

7.2.4.2 Matrix Spiking Solution

Prepare a matrix spiking solution in acetone or methanol that contains the following pesticides at the concentrations specified:

Pesticide	Concentration µg/mL
gamma-BHC (Lindane)	0.5
4,4'-DDT	1.0
Endrin	1.0
Heptachlor	0.5
Aldrin	0.5
Dieldrin	1.0

7.2.4.3 GPC Calibration *and Calibration Verification Solutions*

7.2.4.3.1 Prepare a *GPC calibration* solution in methylene chloride that contains the following analytes at the minimum concentrations listed below:

Analyte	Concentration mg/mL
Corn oil	25.0
Bis-2-ethylhexyl phthalate	0.5
Methoxychlor	0.1
Perylene	0.02
Sulfur	0.08

7.2.4.3.2 NOTE: Sulfur is not very soluble in methylene chloride, but it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds.

7.2.4.3.3 GPC Calibration Verification Solution

Prepare a GPC calibration verification solution in methylene chloride that contains the following compounds. The concentrations listed below are for a 5 mL GPC injection loop. See section 10.1.8.1.4.3 for compound concentrations if a smaller size loop is being used.

Compound	*Concentration ug/mL*
gamma-BHC (Lindane)	*0.1*
Heptachlor	*0.1*
Aldrin	*0.1*
4,4'-DDT	*0.2*
Endrin	*0.2*
Dieldrin	*0.2*

The Aroclor mixture contains 2 ug/mL each of Aroclor 1016 and 1260 in methylene chloride.

7.2.4.4 Florisil Cartridge Check Solution

Prepare a solution of 2,4,5-Trichlorophenol in acetone, at a concentration of 0.1 µg/mL.

7.2.4.5 Resolution Check Mixture

Prepare a mixture in hexane or iso-octane that contains the following pesticides and surrogates at the concentrations listed below.

Compound	Concentration (ng/mL)
gamma-Chlordane	10.0
Endosulfan I	10.0
4,4'-DDE	20.0
Dieldrin	20.0
Endosulfan sulfate	20.0
Endrin ketone	20.0
Methoxychlor	100.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

7.2.4.6 Performance Evaluation Mixture (PEM)

Prepare the PEM in hexane or iso-octane at the concentration levels listed below.

Compound	Concentration (ng/mL)
gamma-BHC	10.0
alpha-BHC	10.0
4,4'-DDT	100.0
beta-BHC	10.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

7.2.4.7 Individual Standard Mixtures A and B

The single component pesticide standards must be prepared in hexane or iso-octane at three concentrations for each analyte, including the surrogates. Two separate calibration mixtures, A and B (listed below), are used to ensure that each peak is adequately resolved. The low point concentration corresponds to the CRQL for each analyte. The midpoint concentration must be 4 times the low point concentration. The high point concentration must be at least 16 times that of the low point, 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.

Individual Standard Mixture A
Low Point Concentration

alpha-BHC	5.0 ng/mL
Heptachlor	5.0 ng/mL
gamma-BHC	5.0 ng/mL
Endosulfan I	5.0 ng/mL
Dieldrin	10.0 ng/mL
Endrin	10.0 ng/mL
4,4'-DDD	10.0 ng/mL
4,4'-DDT	10.0 ng/mL
Methoxychlor	50.0 ng/mL
Tetrachloro-m-xylene	5.0 ng/mL
Decachlorobiphenyl	10.0 ng/mL

Individual Standard Mixture B
Low Point Concentration

beta-BHC	5.0 ng/mL
delta-BHC	5.0 ng/mL
Aldrin	5.0 ng/mL
Heptachlor Epoxide (exo-epoxy isomer)	5.0 ng/mL
alpha-Chlordane	5.0 ng/mL
gamma-Chlordane	5.0 ng/mL
4,4'-DDE	10.0 ng/mL
Endosulfan sulfate	10.0 ng/mL
Endrin aldehyde	10.0 ng/mL
Endrin ketone	10.0 ng/mL
Endosulfan II	10.0 ng/mL
Tetrachloro-m-xylene	5.0 ng/mL
Decachlorobiphenyl	10.0 ng/mL

NOTE: Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is used as an analytical standard.

7.2.4.8 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/μL, except for Aroclor 1221 which must be prepared at 200 ng/μL. Toxaphene must be prepared at 500 ng/mL. All multicomponent standards must contain the surrogates at 20 ng/mL. The Aroclor and toxaphene solutions must be prepared in hexane or iso-octane.

7.2.5 Ampulated Standard Extracts

Standard solutions purchased from a chemical supply house as ampulated extracts in glass vials may be retained until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor which are

immediately ampulated in glass vials may be retained 2 years from the preparation date. Upon breaking the glass seal, the expiration times listed in Sections 7.2.2 through 7.3 will apply. The Contractor is responsible for assuring that the integrity of the standards have not degraded (see Section 7.3.5).

7.3 Storage of Standard Solutions

7.3.1 Store the stock and secondary dilution standard solutions at *4°C (± 2°C)* in Teflon-lined screw cap amber bottles/vials. Fresh standards should be prepared every six months, or sooner if comparison with check standards indicates a problem.

7.3.2 Store the GPC calibration solution in an amber glass bottle with a Teflon lined screw-cap at *4°C (± 2°C)* and protect from light. (Refrigeration may cause the corn oil to precipitate. Before use, allow the calibration solution to stand at room temperature until the corn oil dissolves.) Replace the calibration standard solution every six months, or more frequently if necessary.

7.3.3 Store all other working standard solutions in amber glass bottles or vials with Teflon lined screw caps at *4°C (± 2°C)* and protect from light. The standard solution must be checked frequently for stability. Replace all working standard solutions after six months, or sooner if comparison with quality control check samples indicates a problem, except for the PEM solution which must be prepared weekly. CAUTION: Analysts must allow all standard solutions to equilibrate to room temperature before use.

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

7.3.5 The Contractor is responsible for maintaining the integrity of standard solutions and verifying prior to use. This means that standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in the solution.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in 1 L (or 1 quart) amber glass containers, fitted with screw-caps lined with Teflon. If amber containers are not available, the samples should be protected from light. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis. The specific requirements for site sample collection are outlined by the Region.

8.1.2 All samples must be iced or refrigerated at *4°C (± 2°C)* from the time of collection until extraction.

8.2 Procedure for Sample Storage

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

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3 Procedure for Sample Extract Storage

8.3.1 Sample extracts must be protected from light and stored at *4°C (± 2°C)* until 365 days after delivery of a complete reconciled data package to the Agency.

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

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

8.4 Contract Required Holding Times

8.4.1 Extraction of water samples by separatory funnel procedures must be completed within five days of the Validated Time of Sample Receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction procedures must be started within five days of VTSR. Extraction of soil/sediment samples by sonication must be completed within 10 days of VTSR.

8.4.2 As part of the Agency's QA program, the Agency may provide Performance Evaluation (PE) samples as standard extracts which the Contractor is required to prepare per instructions provided by the Agency. *PE samples must be prepared and analyzed concurrently with the samples in the SDG.* The extraction holding times (five days after VTSR for water, 10 days after VTSR for soil/sediment) do not apply for PE *samples* received as standard extracts.

8.4.3 Analysis of sample extracts must be completed within 40 days following the start of extraction.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Gas Chromatograph Operating Conditions

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

Carrier Gas:	Helium (hydrogen may be used, see Section 6.23.3.7)
Column Flow:	5 mL/min
Make-up Gas:	P-5/P-10 or N_2 (required)
Injector Temperature:	> 200 °C (see Section 9.1.4)
Injection:	On-column
Injection Volume:	1 or 2 µL (see Section 9.1.3)
Injector:	Grob-type, split less
Initial Temperature:	150 °C
Initial Hold Time:	½ min
Temperature Ramp:	5 to 6° C/min
Final Temperature:	275 °C
Final Hold Time:	Until after decachlorobiphenyl has eluted (approximately 10 minutes)

9.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, and MS/MSD. 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.

9.1.3 Manual injections must be 2 µL. Auto injectors may use 1 µL volumes. The same injection volume must be used for all standards, blanks, and samples, including MS/MSD.

9.1.4 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.

9.2 Initial Calibration

9.2.1 Summary of Initial Calibration

Prior to the analysis of samples, including MS/MSD and required blanks, each GC/EC system must be initially calibrated at a minimum of three concentrations to determine instrument sensitivity and the linearity of response utilizing single component target compound and surrogate standards. Multicomponent target compounds are calibrated at a single point.

9.2.2 Frequency of Initial Calibration

Each GC/ECD system must be initially calibrated upon award of the contract, whenever major instrument maintenance or modification is

performed (e.g., column replacement or repair, cleaning or replacement of ECD, etc.) or if the calibration verification technical acceptance criteria have not been met.

9.2.3 Procedure for Initial Calibration

9.2.3.1 Set up the GC/ECD systems as described in Section 9.1.

9.2.3.2 Prepare the initial calibration standards using the analytes and the concentrations specified in Sections 7.2.4.5 through 7.2.4.8.

9.2.3.3 All standards, samples, MS/MSD, blanks, and extracts must be allowed to warm to ambient temperature before analysis.

9.2.3.4 Analyze the initial calibration sequence as given below. NOTE: Steps 16 and 17 are used as part of the calibration verification as well (see Section 9.3).

INITIAL CALIBRATION SEQUENCE

1. Resolution Check
2. Performance Evaluation Mixture
3. Aroclor 1016/1260
4. Aroclor 1221
5. Aroclor 1232
6. Aroclor 1242
7. Aroclor 1248
8. Aroclor 1254
9. Toxaphene
10. Low Point Standard A
11. Low Point Standard B
12. Midpoint Standard A
13. Midpoint Standard B
14. High Point Standard A
15. High Point Standard B
16. Instrument Blank
17. Performance Evaluation Mixture

9.2.4 Calculations for Initial Calibration

9.2.4.1 During the initial calibration sequence, absolute retention times (RT) are determined for all single component pesticides, the surrogates, and at least three major peaks of each multicomponent analyte.

9.2.4.2 For single component pesticides, an RT is measured in each of three calibration standards and the mean RT is calculated as the average of the three values. An RT is measured for the surrogates in each of the three analyses of Individual Standard Mixture A during the initial calibration and the mean RT is calculated as the average of the three values. Calculate a mean absolute retention time ($\overline{RT}$) for each single component pesticide and surrogate using Equation 1.

EQ. 1

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where,

$\overline{RT}$ = Mean absolute retention time of analyte.

RT_i = Absolute retention time of analyte.

n = Number of measurements (3).

9.2.4.3 A 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 the list in Table 1. Windows are centered around the mean absolute retention time for the analyte established during the initial calibrations. Analytes are identified when peaks are observed in the RT window for the compound on both GC columns.

9.2.4.4 The linearity of the instrument is determined by calculating a percent relative standard deviation (%RSD) of the calibration factors from a three-point calibration curve for each single component pesticide and surrogate. 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.

9.2.4.5 Calculate the calibration factor for each single component pesticide and surrogate over the initial calibration range using Equation 2. The calibration factors for the surrogates are calculated from the three analyses of Individual Standard Mixture A only.

9.2.4.6 Calculate the mean and the %RSD of the calibration factors for each single component pesticide and surrogate over the initial calibration range using Equations 3 and 4.

EQ. 2 $$CF = \frac{\text{Peak area (or height) of the standard}}{\text{Mass injected (ng)}}$$

EQ. 3 $$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

EQ. 4 $$\%RSD = \frac{SD_{CF}}{\overline{CF}} \times 100$$

Where,

$$SD_{CF} = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{(n-1)}}$$

%RSD = Percent relative standard deviation

SD_{CF} = Standard deviation of calibration factors

CF_i = Calibration factor

$\overline{CF}$ = Mean calibration factor

n = Total number of values (3)

9.2.4.7 A calibration factor is calculated for each peak in a selected set of three to five major peaks for each multicomponent analyte using Equation 2.

9.2.4.8 Calculate the percent breakdown of DDT, the percent breakdown of endrin, and the combined breakdown of DDT and endrin in the PEM using Equations 5, 6, 7, and 8.

EQ. 5

$$\text{Amount found (ng)} = \frac{\text{Peak area (height) of compound in PEM}}{CF_{mp}}$$

Where,

Cf_{mp} = The calibration factor for the compound determined from the midpoint standard in the most recent initial calibration. NOTE: If during the initial calibration, linearity was determined based on peak area for the compound, then the midpoint CF must be based on peak area. If during the initial calibration, the linearity for the compound was determined based on peak height for the compound, then the midpoint CF must be based on peak height.

EQ. 6

$$\%\text{Breakdown DDT} = \frac{\text{Amount found (ng) (DDD+DDE)}}{\text{Amount (ng) of DDT injected}} \times 100$$

EQ. 7

$$\%\text{Breakdown Endrin} = \frac{\text{Amount found (ng) (endrin aldehyde + enrin ketone)}}{\text{Amount (ng) of endrin injected}} \times 100$$

EQ. 8

Combined %Breakdown = %Breakdown DDT + %Breakdown Endrin

9.2.4.9 Calculate the percent difference for each single component pesticide and surrogate in the PEM using Equations 5 and 9.

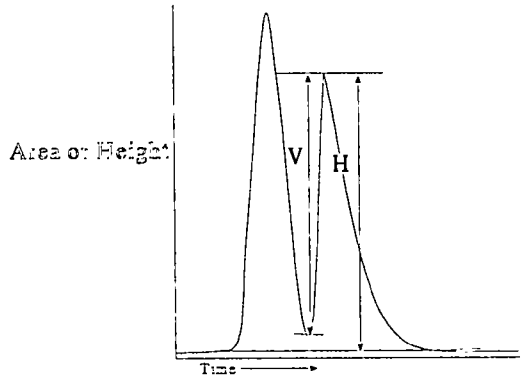
EQ. 9

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

Where,

%D = Percent difference
C_{nom} = Nominal concentration of each analyte
C_{calc} = Calculated concentration of each analyte from the analyses of the standards.

9.2.4.10 Calculate the resolution between the analytes in the Resolution Check Mixture, the Performance Evaluation Mixture, and the midpoint concentration of Individual Standard Mixtures A and B using Equation 10.



EQ. 10

$$\%\text{Resolution} = \frac{V}{H} \times 100$$

Where,

V = Depth of the valley between the two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter of the adjacent peaks.

9.2.5 Technical Acceptance Criteria for Initial Calibration

All initial calibration technical acceptance criteria apply independently to both GC columns.

9.2.5.1 The initial calibration sequence must be analyzed according to the procedure and in the order listed in Section 9.2.3, at the concentrations listed in Sections 7.2.4.5 through 7.2.4.8, and at the frequency listed in Section 9.2.2. The GC/ECD operating conditions optimized in Section 9.1 must be followed.

9.2.5.2 The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0 percent.

9.2.5.3 All single component pesticide and surrogate peaks in both runs of the PEM must be greater than or equal to 90.0 percent resolved on each column.

9.2.5.4 The absolute retention times of each of the single component pesticides and surrogates in both runs of the PEM must be within

the retention time windows determined from the three-point initial calibration in Section 9.2.4.3.

9.2.5.5 The percent difference of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in both of the PEM runs of each GC column must be greater than or equal to -25.0 AND less than or equal to 25.0 percent using Equation 9.

9.2.5.6 The percent breakdown of DDT and endrin in each of the PEM runs must be less than or equal to 20.0 percent. The combined breakdown of DDT and endrin must be less than or equal to 30.0 percent.

9.2.5.7 The %RSD of the calibration factors for each single component target compound must be less than or equal to 20.0 percent, except alpha-BHC and delta-BHC. The %RSD of the calibration factors for alpha-BHC and delta-BHC must be less than or equal to 25.0 percent. The %RSD of the calibration factors for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (but not surrogates) per column may exceed the 20.0 percent limit for %RSD (25.0 percent for alpha-BHC and delta-BHC), but those compounds must have a %RSD of less than or equal to 30.0 percent.

9.2.5.8 The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

9.2.5.9 All instrument blanks must meet the technical acceptance criteria in Section 12.1.4.4.

9.2.5.10 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.

- The chromatograms that result from the analyses of the Resolution Check Mixture, the PEM, and Individual Standard Mixtures A and B during the initial calibration sequence must display the single component analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

- The chromatograms for at least one of the three analyses each of Individual Standard Mixtures A and B from the initial calibration sequence must display the single component analytes at greater than 50 percent and less than 100 percent of full scale.

- The chromatograms of the standards for the multicomponent analytes analyzed during the initial calibration sequence must display the peaks chosen for identification of each analyte at greater than 25 percent and less than 100 percent of full scale.

- For any standard containing alpha-BHC, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

- If a chromatogram is replotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.

- If the chromatogram of any standard needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

9.2.6 Corrective Action for Initial Calibration

9.2.6.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

9.2.6.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. In the case of low level contamination, baking out the detector at an 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 an elevated temperature may correct the problem. In the case of severe contamination, the detector may require servicing by the ECD manufacturer. DO NOT OPEN THE DETECTOR. THE ECD CONTAINS RADIOCHEMICAL SOURCES.

9.2.6.3 If a laboratory cleans out a detector using an elevated temperature, the ECD electronics must be turned off during the bake out procedure.

9.2.6.4 After bake out or hydrogen reduction, the detector must be recalibrated using the initial calibration sequence.

9.2.6.5 Initial calibration technical acceptance criteria must be met before any samples, including MS/MSD or required blanks, are analyzed. Any samples or required blanks analyzed before the initial calibration technical acceptance criteria have been met will require reanalysis at no additional cost to the Agency.

9.3 Calibration Verification

9.3.1 Summary of Calibration Verification

Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, PEMs, and the midpoint 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, PEMs, and both Individual Standard Mixtures A and B.

9.3.2 Frequency of Calibration Verification

9.3.2.1 An instrument blank and the PEM must bracket one end of a 12-hour period during which sample data are collected, and a second instrument blank and the midpoint concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period.

9.3.2.2 For the 12-hour period immediately following the initial calibration sequence, the instrument blank and the PEM that are the last two steps in the initial calibration sequence bracket the front end of that 12-hour period. The injection of the instrument blank starts the beginning of that 12-hour period (see Section 10.2.2.1). Samples may be injected for 12 hours from the injection of the instrument blank. The three injections immediately after that 12-hour period must be an instrument blank, Individual Standard Mixture A, and Individual Standard Mixture B. The instrument blank must be analyzed first, before either standard. The Individual Standard Mixtures may be analyzed in either order (A,B or B,A).

9.3.2.3 The analyses of the instrument blank and Individual Standard Mixtures A and B immediately following one 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the acceptance criteria in Section 9.3.5. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and a PEM, in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks, PEMs, or Individual Standard Mixtures fails to meet the acceptance criteria in Sections 9.3.5. The 12-hour time period begins with the injection of the instrument blank.

9.3.2.3.1 Standards (PEM or Individual Standard Mixtures), samples and required blanks may be injected for 12 hours from the time of injection of the instrument blank.

9.3.2.4 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 was injected.

9.3.2.5 The requirements for running the instrument blanks, PEM, and Individual Standard Mixtures A and B are waived when no samples, dilutions, reanalyses, method/sulfur blanks, MS/MSD, and multicomponent analytes for the 72-hour confirmation requirement are analyzed during that 12-hour period. To resume analysis, using the existing initial calibration, the Contractor first must analyze an instrument blank and PEM which meet the technical acceptance criteria.

9.3.2.6 If the entire 12-hour period is not required for the analyses of all samples to be reported, the sequence must be ended with either the instrument blank/PEM combination or the instrument blank/Individual Standard Mixtures A and B combination, whichever was due to be performed at the end of the 12-hour period.

9.3.3 Procedure for Calibration Verification

9.3.3.1 Analyze the PEM, instrument blank, and the midpoint concentration of Individual Standard Mixtures A and B at the required frequencies (Section 9.3.2).

9.3.3.2 All standards and blanks must be at ambient temperature at the time of preparation and analysis.

9.3.4 Calculations for Calibration Verification

9.3.4.1 For each analysis of the PEM 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 5 and 9.

9.3.4.2 Calculate the percent breakdown of DDT and endrin, and the combined breakdown in each PEM analyzed using Equations 5, 6, 7, and 8.

9.3.4.3 For each analysis of the midpoint 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 5 and 9. Do not attempt to calculate the breakdown of Endrin and DDT in the Individual Standard Mixtures, as these standards contain the breakdown products as well as the parent compounds.

9.3.5 Technical Acceptance Criteria for Calibration Verification

All calibration verification technical acceptance criteria apply independently to each column. Each column must meet criteria.

9.3.5.1 The PEMs, Individual Standard Mixtures A and B and instrument blanks must be analyzed at the required frequency on a GC/EC

system that has met the initial calibration technical acceptance criteria.

9.3.5.2 All single component pesticides and surrogates in the PEMs used to demonstrate continuing calibration must be greater than or equal to 90.0 percent resolved. The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B *used to demonstrate* initial calibration must be greater than or equal to 90.0 percent.

9.3.5.3 The absolute retention time for each of the single component pesticides and surrogates in the PEMs and midpoint concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be within the retention time window determined from the three-point initial calibration in Section 9.2.4.3.

9.3.5.4 The percent difference between the calculated amount and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM must be greater than or equal to -25.0 percent and less than or equal to 25.0 percent.

9.3.5.5 The percent difference between the calculated amount and the nominal amount (amount added) for each of the single component pesticides and surrogates in the INDA and INDB that have been used as calibration verification must be greater than or equal to -25.0 percent and less than or equal to 25.0 percent.

9.3.5.6 The percent breakdown of DDT in the PEM must be less than or equal to 20.0 percent on each column. The percent breakdown of endrin in the PEM must be less than or equal to 20.0 percent on each column. The combined breakdown of both DDT and endrin must be less than or equal to 30.0 percent on each column.

9.3.5.7 All instrument blanks must meet the technical acceptance criteria in Section 12.1.4.4.

9.3.5.8 The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. Since the retention time of the apex of a peak can only be verified from an on-scale chromatogram, the following requirements must be met for continuing calibration to be acceptable.

9.3.5.8.1 The chromatograms that result from the analyses of the PEM and the Individual Standard Mixtures must display the single component analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

9.3.5.8.2 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.

9.3.5.8.3 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

9.3.5.8.4 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.

9.3.6 Corrective Action for Calibration Verification

9.3.6.1 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective action to achieve the acceptance criteria.

9.3.6.2 Major corrective actions such as replacing the GC column or baking out the detector will require that a new initial calibration be performed that meets the technical acceptance criteria in Section 9.2.5.

9.3.6.3 Minor corrective actions may not require performing a new initial calibration, provided that a new analysis of the standard (PEM or Individual Standard Mixture) that originally failed the criteria and an associated instrument blank immediately after the corrective action do meet all the acceptance criteria.

9.3.6.4 If a PEM or Individual Standard Mixture does not meet technical acceptance criteria listed above, it must be reinjected immediately. If the second injection of the PEM or Individual Standard Mixture meets the criteria, sample analysis may continue. **If the second injection does not meet the criteria, all data collection must be stopped. Appropriate corrective action must be taken, and a new initial calibration sequence must be run before more sample data are collected.**

9.3.6.5 If an instrument blank does not meet the technical acceptance criteria listed in Section 12.1.4.4, 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.

9.3.6.6 Analysts are reminded that running an instrument blank and a PEM or Individual Standard Mixtures once every 12 hours 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 and standards more often to avoid discarding data.

9.3.6.7 If a successful instrument blank and PEM cannot be run after an interruption in analysis (Section 9.3.2.5), 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 9.3.2.

9.3.6.8 Calibration verification technical acceptance criteria must be met before any samples, including MS/MSD and required blanks are reported. Any samples, including MS/MSD and required blanks associated with a calibration verification which did not meet the technical acceptance criteria will require reanalysis at no additional cost to the Agency.

10.0 PROCEDURE

10.1 Sample Preparation

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

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

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

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

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

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

10.1.3 Extraction of Water Samples

Water samples may be extracted by either a separatory funnel procedure or a continuous liquid-liquid extraction procedure. If an emulsion prevents acceptable solvent recovery with the separatory funnel procedure, continuous liquid-liquid extraction must be employed.

10.1.3.1 Separatory Funnel Extraction

10.1.3.1.1 Measure out each 1 L sample aliquot in a separate graduated cylinder. Measure and record pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring pH adjustment must be noted in the SDG Narrative. Place the sample into a 2 L separatory funnel.

10.1.3.1.2 Using a syringe or a volumetric pipet, add 1.0 mL of the surrogate solution to all water samples.

10.1.3.1.3 Rinse the graduated cylinder with 30 mL of methylene chloride and transfer the rinsate to the separatory funnel. If the sample was received in a 1 L container, rinse the empty container with 30 mL of methylene chloride and add rinsate to the separatory funnel. If the sample container is not rinsed, then add another 30 mL of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure. NOTE: The total volume of solvent used for extraction is 60 mL. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation or other physical means. Drain the methylene chloride into a 250 mL Erlenmeyer flask.

10.1.3.1.4 Add a second 60 mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.1.3.2 Continuous Liquid-Liquid Extraction

10.1.3.2.1 Continuous Liquid-Liquid Extraction Without Hydrophobic Membrane

10.1.3.2.1.1 Follow manufacturer's instructions for set-up.

10.1.3.2.1.2 Add methylene chloride to the bottom of the extractor and fill it to a depth of at least one inch above the bottom sidearm.

10.1.3.2.1.3 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder; transfer the aliquot to the continuous extractor. Measure the pH of the sample with wide range pH paper and record pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, as required. Samples requiring the pH adjustment must be noted in the SDG Narrative. NOTE: With some samples, it may be necessary to place a layer of glass wool between the

methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.3.2.1.4 Using a syringe or volumetric pipet, add 1.0 mL of the surrogate standard spiking solution (Section 7.2.4.1) into the sample and mix well.

10.1.3.2.1.5 Rinse the graduated cylinder with 50 mL of methylene chloride and transfer the rancid to the continuous extractor. If the sample was received in a 1 L container, rinse the empty container with 50 mL of methylene chloride and add the rancid to the continuous extractor.

10.1.3.2.1.6 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5 to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 18 hours. NOTE: When a minimum drip rate of 10-15 mLs/minute is maintained throughout the extraction, the extraction time may be reduced to a minimum of 12 hours. Allow to cool, then detach the distillation flask. Proceed to Section 10.1.4.

10.1.3.2.1.7 NOTE: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.3.2.2 Continuous Liquid-Liquid Extraction With Hydrophobic Membrane

10.1.3.2.2.1 Follow the manufacturer's instructions for set-up.

10.1.3.2.2.2 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder. If the sample was received in a 1 L container, rinse the empty container with 50 mL of methylene chloride and add the rinsate to the continuous extractor. If the sample was not received in a 1 L container, add 50 mL of methylene chloride to the continuous extractor. Slowly transfer the aliquot to the continuous extractor. Measure the pH of the sample with wide range pH paper and record pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, as required. Samples requiring the pH adjustment must be noted in the SDG Narrative.

10.1.3.2.2.3 Using a syringe or volumetric pipet, add 1.0 mL of the surrogate standard spiking solution (Section 7.2.4.1) into the sample and mix well.

10.1.3.2.2.4 Rinse the graduated cylinder with 50 mL of methylene chloride and transfer the rinsate to the continuous extractor.

10.1.3.2.2.5 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours. (NOTE: Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion which will consume the solvent volume, preventing the efficient extraction of the sample. When this occurs, add additional solvent to assure efficient extraction of the sample, and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used.) Allow to cool, then detach the distillation flask.

10.1.3.2.2.6 NOTE: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction act-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane, it may not be necessary to dry the extract with sodium sulfate.

10.1.3.2.3 NOTE: If low surrogate recoveries occur, assure 1) the apparatus was properly assembled to prevent leaks; 2) the drip rate/solvent cycling was optimized; and 3) there was proper cooling for condensation of solvent.

10.1.3.2.4 NOTE: Alternate continuous liquid-liquid extractor types that meet the requirements of the SOW may also be used. If using alternate extractors or design types, follow the manufacturer's instructions for set-up. Optimize the extraction procedure.

10.1.4 Extract Drying

10.1.4.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D if equivalency is demonstrated for all the pesticide/Aroclor target compounds listed in Exhibit C.

10.1.4.2 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 column with at least two additional 20 to 30 mL portions of methylene chloride to complete the quantitative transfer.

10.1.5 Soil/Sediment Samples

Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks. Also, decant and discard any standing aqueous phase.

10.1.5.1 pH Determination

10.1.5.1.1 Transfer 50 g of soil/sediment to a 100 mL beaker. Add 50 mL of water and stir the solution with a magnetic stirrer for 1 hour. Determine the pH of the sample by using a combination glass electrode and pH meter while the sample is stirred. Report the pH value on the appropriate data sheet. If the pH of the soil/sediment is > 9 or < 5, document any subsequent problems ln the analysis related to pH in the SDG Narrative, but do not attempt to adjust the pH of the sample. Discard the portion of the sample used for pH determination.

10.1.5.1.2 NOTE: If insufficient volume of soil/sediment is received, use a smaller 1:1 ratio of grams of sample to mLs of water for the pH determination, and note in the SDG Narrative.

10.1.5.2 Percent Moisture

Weigh 5 to 10 g of the soil/sediment to the nearest 0.01 g into a tared crucible or aluminum weighing pan. Determine the weight percent volatilized by drying overnight at 105 °C (hereafter referred to as percent moisture). After the sample is dry, remove the sample and pan and allow them to cool in a desiccator before weighing. Calculate the percent moisture according to Equation 11 below. Concentrations of individual analytes will be reported relative to the dry weight of soil/sediment. CAUTION: Gases volatized from some soil/sediment samples may require that this drying procedure be carried out in a hood.

EQ. 11

$$\%\text{Moisture} = \frac{\text{grams of wet sample - grams of dry sample}}{\text{grams of wet sample}} \times 100$$

10.1.5.3 Soil/Sediment Extraction

The procedure described below is for the extraction of soil/sediment samples by sonication. The Contractor may also use Automated Soxhlet Extraction (SW-846 Method 3541 Revision 0, September 1994) or Pressurized Fluid Extraction (SW-846 Method 3545 Revision 0, December 1996) techniques for soil/sediment samples. The above SW-846 methods are provided as reference only and the laboratory supplied Standard Operating Procedures must be accepted by the Agency before the laboratory can utilize these methods (See Exhibit E for required IPR studies). The requirements of this SOW must be met at all times (i.e., original sample weight). As applicable, follow manufacturer's instructions for use of all extraction equipment. If one of the above alternative extraction procedures is used, the Contractor must maintain documentation of the procedure utilized and document its equivalence to the sonication procedure described below. If the sample weight must be adjusted to utilize one of the alternative extraction procedures, the Contractor shall immediately contact

SMO to inform them of the problem. SMO will contact the Region for instructions.

Note: All soil/sediment samples in a Case must be extracted by the same procedure.

10.1.5.3.1 Tune the sonicator according to the manufacturer's directions prior to extracting samples by this procedure.

10.1.5.3.2 Weigh approximately 30 g of sample (to the nearest 0.1 g) into a 250 or 400 mL beaker and add 60 g of anhydrous sodium sulfate (granular).

10.1.5.3.3 Add 2.0 mL of surrogate solution to all soil/sediment samples by using a volumetric pipet or a syringe. Mix the sample well. The sample and the added sodium sulfate should be a homogeneous, granular mixture at this point. Twice as much of the surrogate solution is added to soil/sediment samples than to water samples.

10.1.5.3.4 Immediately add 80 to 100 mL of 1:1 methylene chloride/acetone to the sample.

10.1.5.3.5 Place the bottom surface of the sonicator probe about ½ inch below the surface of the solvent but above the sediment layer.

10.1.5.3.6 Sonicate for 3 minutes using a 3/4 inch disruptor horn at full power (output control knob at 10) with pulse on and percent duty cycle knob set at 50 percent. Do <u>not</u> use a microtip. NOTE: These settings refer to the Model W-385. When using a sonicator other then Model W-385, refer to the instructions provided by the manufacturer for appropriate output settings.

10.1.5.3.7 The extracted sample can be filtered by using gravity or vacuum filtration.

10.1.5.3.8 For gravity filtration prepare a filtration/drying bed by placing a plug of glass wool in the neck of a 10 cm powder funnel and filling the funnel to approximately half its depth (4 or 5 cm) with anhydrous sodium sulfate (80-100 g). Decant the extract through the packed funnel and collect it in a 500 mL evaporative (K-D) flask attached to a concentrator tube.

10.1.5.3.9 For vacuum filtration, use Whatman No. 41 paper in the Buchner funnel. Pre-wet the paper with methylene chloride/acetone before decanting the solvent.

10.1.5.3.10 Repeat the extraction two more times with additional 80 to 100 mL portions of the 1:1 methylene chloride/acetone. Before each extraction, thoroughly mix the solid residue and make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula. Decant and filter the extraction solvent after each sonication by using the same funnel described above.

After the final sonication, pour the entire sample into the funnel and rinse the beaker and funnel with 60 mL of 1:1 methylene chloride/acetone.

10.1.6 Concentrating the Extract

10.1.6.1 Concentration by K-D

Other concentration devices or techniques may be used in place of the K-D if equivalency is demonstrated for all the pesticide/Aroclor target compounds listed in Exhibit C.

10.1.6.1.1 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 °C to 70 °C recommended) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 3 to 5 mL for water samples (and less than 10 mL for soil/sediment samples), remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.1.6.1.2 For water extracts which do not require GPC cleanup, and for water and soil/sediment extracts which have been through the GPC cleanup step, proceed with the hexane exchange described in Section 10.1.6.2.

10.1.6.1.3 For water extracts which require GPC cleanup, remove the Snyder column, rinse the flask and its lower joint, collect the rinsate in the concentrator tube, and adjust the volume to 10 mL with methylene chloride. Proceed to Section 10.1.8.1.

10.1.6.1.4 For soil/sediment extracts that have not been cleaned-up using GPC, it is absolutely necessary to further reduce the volume of all soil/sediment extracts to 1 mL in order to remove most of the acetone. This is best accomplished using the nitrogen evaporation technique (Section 10.1.7.2). The presence of acetone will cause a dead volume to develop in the GPC column and thus will cause loss of surrogates and analytes during GPC cleanups. Adjust the soil/sediment extract volume to 10 mL with methylene chloride. Proceed to Section 10.1.8.1 for mandatory GPC.

10.1.6.2 Solvent Exchange into Hexane

This procedure applies to both extracts of water samples and extracts of soil/sediment samples.

10.1.6.2.1 With the extract in a K-D apparatus, remove the 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 described previously (Section 10.1.6.1), but increase the temperature of the water bath (to between 80 and 90 °C recommended). When the apparent volume of liquid reaches 3 to 5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.1.6.2.2 Remove the Snyder column; using 1 to 2 mL of hexane, rinse the flask and its lower joint into the concentrator tube. Complete quantitative transfer of the extract to a 10 mL vial by using hexane.

10.1.6.2.3 For samples which have <u>not</u> been subjected to GPC cleanup, adjust the volume of the hexane extract to 10 mL. For samples which <u>have</u> been subjected to GPC cleanup, concentrate the hexane extract to 5 mL using a micro Snyder column or nitrogen evaporation, as described in Section 10.1.7.1 or 10.1.7.2. Proceed to Section 10.1.8.2 for Florisil cartridge cleanup.

10.1.7 Final Concentration of Extract

Two different techniques are permitted to concentrate the extract to volume before Florisil cleanup or extract volume before instrumental analysis. They are the Micro Snyder Column and Nitrogen Evaporation Technique.

10.1.7.1 Micro Snyder Column Concentration

Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Pre-wet the Snyder column by adding about 0.5 mL of hexane to the top of the column. Place the K-D apparatus in a hot water bath (80 °C to 90 °C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 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 or 2 mL (see Sample Cleanup by Florisil Cartridge, Section 10.1.8.2.3).

10.1.7.2 Nitrogen Evaporation Technique (taken from ASTM Method D 3086)

10.1.7.2.1 Place the Concentrator tube in a warm water bath (30 °C to 35 °C recommended) and evaporate the solvent volume to the final volume by blowing a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon) onto the solvent. DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS. Adjust the soil/sediment extract volume to 10 mL with methylene chloride. Proceed to *Section 10.1.8* for mandatory GPC cleanup procedures.

10.1.7.2.2 Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or Teflon tubing. Plastic tubing must not be used between the carbon trap and the sample as it may introduce interferences. The internal wall of new tubing must be rinsed several times with hexane and then dried prior to use.

10.1.7.2.3 During evaporation, the tube solvent level must be kept below the water level of the bath.

10.1.8 Cleanup Procedures

There are three cleanup procedures specified in this method: GPC, Florisil cartridge, and sulfur cleanup. GPC must be performed for all soil/sediment extracts. GPC must be performed for water extracts that contain higher molecular weight contaminants that interfere with the analysis of the target analytes. Florisil cartridge cleanup is mandatory for all extracts. Sulfur cleanup must be performed for all sample extracts contaminated with sulfur. Blanks and matrix spike and matrix spike duplicate samples must be subjected to the same cleanup as the unspiked samples.

10.1.8.1 Sample Cleanup by Gel Permeation Chromatography (GPC)

10.1.8.1.1 Introduction

Gel Permeation Chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of natural (and synthetic) macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated.

10.1.8.1.2 GPC Column Preparation

The instructions listed below for GPC column preparation are for Bio Beads. Alternate column packings may be used if 1) the column packings have equivalent or better performance than the Bio Beads and meet the technical acceptance criteria for GPC calibration and GPC calibration checks, and 2) the column packings do not introduce contaminants/artifacts into the sample which interfere with the analysis of the pesticide

compounds. Follow the manufacturer's instructions for preparation of the GPC column packing.

10.1.8.1.2.1 Weigh out 70 g of Bio Beads (SX-3). Transfer them to a 1 L bottle with a Teflon-lined cap or a 500 mL separatory funnel with a large bore stopcock, and add approximately 300 mL of methylene chloride. Swirl the container to ensure the wetting of all beads. Allow the beads to swell for a minimum of 2 hours. Maintain enough solvent to cover the beads sufficiently at all times. If a guard column is to be used, repeat the above with 5 g of Bio Beads in a 125 mL bottle or a beaker, using 25 mL of methylene chloride.

10.1.8.1.2.2 Turn the column upside down from its normal position and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger). Position and tighten the outlet bed support plunger as near the end as possible, but no closer than 5 cm (measured from the gel packing to the collar).

10.1.8.1.2.3 Raise the end of the outlet tube to keep the solvent in the GPC column, or close the column outlet stopcock. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

10.1.8.1.2.4 Swirl the bead/solvent slurry to get a homogeneous mixture and, if the wetting was done in a quart bottle, quickly transfer it to a 500 mL separatory funnel with a large bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and then start draining the slurry into the column by placing the separatory funnel tip against the column wall. This will help to minimize bubble formation. Swirl occasionally to keep the slurry homogeneous. Drain enough to fill the column. Place the tubing from the column outlet into a waste beaker below the column, open the stopcock (if attached), and allow the excess solvent to drain. Raise the tube to stop the flow, and close the stopcock when the top of the gel begins to look dry. Add additional methylene chloride to just rewet the gel.

10.1.8.1.2.5 Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

10.1.8.1.2.6 Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe off the plunger. Slurry any remaining beads and transfer them into the column. Repeat the step in Section 10.1.8.1.2.5 and reinsert the plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

10.1.8.1.2.7 Push the plunger until it meets the gel, then compress the column bed about four centimeters.

10.1.8.1.2.8 Pack the optional 5 cm column with approximately 5 g of pre swelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

10.1.8.1.2.9 Connect the column inlet to the solvent reservoir (reservoir should be placed higher than the top of the column) and place the column outlet tube in a waste container. Placing a restrictor in the outlet tube will force air out of the column more quickly. A restrictor can be made from a piece of capillary stainless steel tubing of 1/16" OD x 0.010" ID x 2". Pump methylene chloride through the column at a rate of 5 mL/min for one hour.

10.1.8.1.2.10 After washing the column for at least one hour, connect the column outlet tube, without the restrictor, to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. A restrictor (same size as the one in Section 10.1.8.1.2.9 above) in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not affect flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi backpressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

10.1.8.1.2.11 When the GPC column is not to be used for several days, connect the column outlet line to the column inlet to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, reswelled, and repoured as described above. If drying occurs, methylene chloride should be pumped through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify retention volumes have not changed. NOTE: The description of solvent flow rate and column pressure applies only to the ABC GPC apparatus.

Laboratories using equivalent equipment must develop the parameters for their apparatus which give acceptable performance as described in Section 10.1.8.1.3.

10.1.8.1.3 Calibration of GPC

10.1.8.1.3.1 Summary of GPC Calibration

10.1.8.1.3.1.1 The GPC calibration procedure is based on monitoring the elution of standards with a UV detector connected to the GPC column.

10.1.8.1.3.1.2 The UV detector calibration procedure described in Section 10.1.8.1.3.3 is needed for the analyses of organochlorine pesticides and Aroclors listed in Exhibit C. IT MUST NOT BE USED FOR THE ANALYSIS OF GC/MS EXTRACTABLES OR OTHER ANALYTES WITHOUT A RECOVERY STUDY.

10.1.8.1.3.2 Frequency of GPC Calibration

Each GPC system must be calibrated upon award of the contract, when the GPC *calibration verification* solution fails to meet criteria, when the column is changed, when channeling occurs, *and once every seven days*.

10.1.8.1.3.3 Procedure for GPC Calibration

Follow the manufacturer's instructions for operating the GPC system. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored.

10.1.8.1.3.3.1 *Using a 10 mL syringe, load the calibration solution (Section 7.2.4.3) onto the GPC.* Determine the elution times for the phthalate, methoxychlor, and perylene. Phthalate will elute first; perylene, last.

10.1.8.1.3.3.2 Choose a "DUMP" time which removes > 85 percent of the phthalate. Choose a "COLLECT" time so that > 95 percent of the methoxychlor is collected, and continue to collect until just prior to the elution time of sulfur. Use a "WASH" time of 10 minutes.

NOTE: The DUMP and COLLECT times must be adjusted to compensate for the difference in volume of the lines between the detector and the collection flask.

10.1.8.1.3.3.3 Reinject the calibration solution after appropriate collect and dump cycles have been set, and the solvent flow and column pressure have been established.

10.1.8.1.3.3.4 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed may be used to indicate

problems with the system during sample processing.

10.1.8.1.3.3.5 Analyze a GPC blank of methylene chloride. Concentrate the methylene chloride that passed through the system during the collect cycle using Kuderna-Danish (K-D) evaporator. Exchange the solvent to hexane and analyze the concentrate by GC/EC according to the procedure in Section 10.2 (usual protocol). Assuming that the blank represents the extract from a 1 L water sample, calculate the analyte concentrations using Equation 13.

10.1.8.1.3.4 Technical Acceptance Criteria for GPC Calibration

10.1.8.1.3.4.1 The GPC system must be calibrated at the frequency described in Section 10.1.8.1.3.2. The UV trace must meet the following requirements:

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit > 85 percent resolution.
- Phthalate and methoxychlor peaks must exhibit > 85 percent resolution.
- Methoxychlor and perylene peaks must exhibit > 85 percent resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit > 90 percent baseline resolution.

10.1.8.1.3.4.2 The solvent flow rate and column pressure must be within *the manufacturer's specified ranges*.

10.1.8.1.3.4.3 The retention times for bis (2-ethylhexyl) phthalate and perylene must not vary more than ± 5 percent between calibrations. If the retention time shift is > 5 percent, take corrective action. Excessive retention time shifts are caused by the following:

- Poor laboratory temperature control or system leaks.
- An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight.
- Excessive laboratory temperatures causing outgassing of the methylene chloride.

10.1.8.1.3.4.4 The analyte concentrations in a GPC blank must be less than the CRQL of any compound in Exhibit C (Pesticides).

10.1.8.1.3.5 Corrective Action for GPC Calibration

10.1.8.1.3.5.1 If the flow rate and/or column pressure do not fall within the *manufacturer's specified* ranges, a new column should be prepared.

10.1.8.1.3.5.2 A UV trace that does not meet the criteria in Section 10.1.8.1.3.4 would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads if the column fails all the criteria.

10.1.8.1.3.5.3 If the GPC blank is equal to or exceeds the CRQL of any compound in Exhibit C (Pesticides), pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

10.1.8.1.4 GPC Calibration *Verification*

10.1.8.1.4.1 Summary of GPC Calibration *Verification*

The GPC calibration must be routinely verified with two check mixtures *(Section 7.2.4.3.3)*.

10.1.8.1.4.2 Frequency of GPC Calibration *Verification*

10.1.8.1.4.2.1 The calibration *verification* must be performed at least once every 7 days *(immediately following the GPC Calibration)* whenever samples, including matrix spikes, matrix spike duplicates, and blanks are cleaned up using the GPC.

10.1.8.1.4.2.2 Some samples may contaminate the SX-3 Bio Beads and change the retention volume of the GPC column. Therefore, system calibration and analyte recovery must be checked whenever a sample causes significant discoloration of the GPC column. Even if no darkening is visible, GPC calibration must be checked not less than once every seven days.

10.1.8.1.4.3 Procedure for GPC Calibration *Verification*

The instructions below are for a GPC injection loop of 5 mL. If a 2 mL injection loop is used, the Contractor will adjust the volume to 4 mL instead of 10 mL before injection extract on the GPC.

10.1.8.1.4.3.1 The pesticide GPC calibration *verification* solution contains the following six compounds in methylene chloride: gamma-BHC (Lindane), Heptachlor, and Aldrin each at a concentration of 0.1 µg/mL for a 5 mL GPC loop (0.25 µg/mL when a 2 mL GPC loop is used) and 4,4'-DDT , Endrin, and Dieldrin at 0.2 µg/mL (*0.50* µg/mL for a 2 mL loop). The Aroclor mixture contains 2 µg/mL each of

Aroclor 1016 and 1260 in methylene chloride (*5.0* µg/mL when a 2 mL GPC loop is used).

10.1.8.1.4.3.2 Load the first 5 mL sample loop by using a 10 mL syringe containing 8 mL of the pesticide GPC calibration *verification* solution. The Aroclor mixture is loaded into loop #2 in the same manner. Fractions are collected in an auto sequence by using the GPC program established be the UV detector calibration procedure (Section 10.1.8.1.3).

10.1.8.1.4.3.3 The collected GPC calibration *verification* fraction is transferred to a K-D apparatus, and the collection vessel is rinsed with two additional 10 mL portions of methylene chloride to complete the transfer. The volume of methylene chloride is reduced according to Section 10.1.6.1. After cooling, the solvent is exchanged to hexane according to the instructions in Section 10.1.6.2. The final volume is adjusted to 10 mL, and the sample is analyzed by GC according to the procedure in Section 10.2 (usual protocol). The analysis must be performed on only one of the GC columns used for sample analysis.

10.1.8.1.4.3.4 The pattern of the Aroclor quantitation peaks and the recovery of each single component analyte must be determined for evaluation and reporting purposes.

10.1.8.1.4.4 Technical Acceptance Criteria for GPC Calibration *Verification*

10.1.8.1.4.4.1 The recovery of each of the single component analytes must be between 80 - 110 percent.

10.1.8.1.4.4.2 The Aroclor patterns must be the same as those from the Aroclor 1016 and Aroclor 1260 standards in the initial calibration sequence *(Section 9.2.5.10)*.

10.1.8.1.4.5 Corrective Action for GPC Calibration *Verification*

Analysts may continue to use the GPC column if the technical acceptance criteria for the GPC calibration *verification* are met. If the recoveries are out of the acceptance window or if changes in the relative peak heights of the patterns of the Aroclor are observed, the columns must be replaced and the GPC recalibrated according to the instructions in Section 10.1.8.1.3 before proceeding with any GPC cleanup on samples, including blanks - method and/or sulfur and MS/MSD.

10.1.8.1.5 Daily UV Calibration Check (Optional)

The calibration of the GPC may be monitored daily by use of the UV-GPC calibration solution (Section 7.2.4.3) and the UV detector calibration procedure (Section 10.1.8.1.3.3). The UV detector should be used to monitor the elution times for the

phthalate, methoxychlor and perylene. in that order. The pre-calibrated GPC program should "dump' > 85 percent of the phthalate and should "collect" > 95 percent of the methoxychlor and perylene. Significant changes in elution times of the analytes (e.g., > 0.5 minutes) indicate that the column is out of calibration and must be recalibrated or replaced.

10.1.8.1.6 Sample Cleanup by GPC

10.1.8.1.6.1 Introduction to Sample Cleanup by GPC

10.1.8.1.6.1.1 It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift, and the dump and collect times determined by the calibration standard no longer will be appropriate. The ideal laboratory temperature to prevent out gassing of the methylene chloride is 22 °C.

10.1.8.1.6.1.2 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of 1:1 glycerol:water solution must be diluted and loaded into several loops. Similarly, extracts containing more than 40 mg/mL of non-volatile residue must be diluted and loaded into several loops. The non-volatile residue may be determined by evaporating a 100 µL aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container. When multiple loops/runs are necessary for an individual sample, be sure to combine all of the sample eluates collected.

10.1.8.1.6.1.3 Systems using automated injection devices to load the sample on the column must be carefully monitored to assure that the required amount is injected onto the column. Viscous extracts, or extracts containing a large amounts of non-volatile residue, will cause problems with injecting the proper amount of sample extract onto the column using automated injection systems. After the sample extract has been processed, the remaining sample extract in an injection vial must be checked to assure that the proper amount of extract was injected on the column before proceeding with the sample analysis. If the proper amount of extract was not injected, the sample must be reprepared and the sample extract must be either diluted and loaded into several loops or the sample extract must be injected manually.

10.1.8.1.6.2 Frequency of Sample Cleanup by GPC

GPC cleanup must be performed at least once for each soil/sediment extract, water extracts that contain high molecular weight contaminants that interfere with the

analysis of the target analytes and all associated QC (blanks and spikes). If cleanup procedure is inadequate contact SMO.

10.1.8.1.6.3 Procedure for Sample Cleanup by GPC

10.1.8.1.6.3.1 Particles greater than 5 microns may scratch the valve, which may result in a system leak and cross contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 5 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 mL syringe. Draw the sample extract through the filter assembly and into the 10 mL syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container (e.g., a 15 mL culture tube with a Teflon-lined screw cap). Alternatively, draw the extract into the syringe without the filter assembly. Attach the filter assembly and force the extract through the filter and into the glass container. Draw a minimum of 8 mL of extract into a 10 mL syringe.

Note: Some GPC instrument manufacturer's recommend using a smaller micron size filter disc. In this instance, follow the manufacturer's recommended operating instructions.

10.1.8.1.6.3.2 INTRODUCTION OF PARTICULATES OR GLASS WOOL INTO THE GPC SWITCHING VALVES MAY REQUIRE FACTORY REPAIR OF THE APPARATUS.

10.1.8.1.6.3.3 *Follow the manufacturer's instructions for operation of the GPC system being utilized.* Note: These instructions were written for a 5 mL GPC injection loop. A 2 mL injection loop may be used in place of a 5 mL injection loop. If a 2 mL loop is used, concentrate the 10 mL extract to 4 mL, and then inject 2 mL from the 4 mL.

10.1.8.1.6.3.4 If the sample is difficult to load, some part of the system may be blocked. Take appropriate corrective action *following manufacturer's recommendations. The problem must be resolved prior to loading sample extracts.*

10.1.8.1.6.3.5 After loading each sample loop, wash the loading port with methylene chloride to minimize cross contamination. Inject approximately 10 mL of methylene chloride to rinse the common tubes.

10.1.8.1.6.3.6 After loading all sample loops, process each sample using the collect and dump cycle times established in Section 10.1.8.1.3.

10.1.8.1.6.3.7 Collect each sample in a 250 mL Erlenmeyer flask covered with aluminum foil to reduce solvent evaporation, or directly into a Kuderna-Danish evaporator. Monitor sample volumes collected. Changes in sample volumes collected may indicate one or more of the following problems:

- Change in solvent flow rate, caused by channeling in the column or changes in column pressure.
- Increase in column operating pressure due to the absorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.
- Leaks in the system or significant variances in room temperature.

10.1.8.1.6.3.8 After the appropriate GPC fraction has been collected for each sample, concentrate the extract as per Section 10.1.6.1 and proceed to solvent exchange into hexane as described in Section 10.1.6.2 and Florisil cleanup in 10.1.8.2.

10.1.8.1.6.3.9 Any samples that were loaded into two or more loops must be recombined before proceeding with concentration.

10.1.8.2 Florisil Cleanup

10.1.8.2.1 Introduction

Florisil cartridge cleanup significantly reduces matrix interference caused by polar compounds and is required for all extracts. The same volume of the concentrated extract taken for Florisil cleanup must be maintained after Florisil cleanup (1 or 2 mL).

10.1.8.2.2 Florisil Cartridge Performance Check

10.1.8.2.2.1 Summary of Florisil Cartridge Performance Check

Every lot number of Florisil cartridges must be tested before they are used for sample cleanup.

10.1.8.2.2.2 Frequency of Florisil Cartridge Performance Check

Cartridge performance check must be conducted at least once on each lot of cartridges used for sample cleanup *or every 6 months, whichever is most frequent*.

10.1.8.2.2.3 Procedure for Florisil Cartridge Performance Check

Add 0.5 mL of 2,4,5-trichlorophenol solution (0.1 µg/mL in acetone, Section 7.2.4.4) and 0.5 mL of Standard Mixture A, midpoint concentration, (Section 7.2.4.7) to 4 mL of hexane. Reduce the final volume to 0.5 mL using nitrogen (Section 10.1.7.2). Place the mixture onto the top of a washed Florisil cartridge, and elute it with 9 mL of hexane/acetone [(90:10)(V/V)]. Use two additional 1 mL hexane rinses toensure quantitative transfer of standard from the cartridge. Reduce the final volume to 1 mL using nitrogen (Section 10.1.7.2) and analyze the solution by GC/EC using at least one of the GC columns specified for sample analysis. Determine the recovery of each analyte for evaluation and reporting purposes. Calculate the percent recovery using Equation 12.

EQ. 12

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Quantity determined by analysis
Q_a = Quantity added

10.1.8.2.2.4 Technical Acceptance Criteria for Florisil Cartridge Performance Check

10.1.8.2.2.4.1 The cartridge performance check solution must be analyzed on a GC/EC meeting the initial calibration and calibration verification technical acceptance criteria.

10.1.8.2.2.4.2 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 120 percent, if the recovery of trichlorophenol is less than 5 percent, and if no peaks interfering with the target analytes are detected.

10.1.8.2.2.5 Corrective Action for Florisil Cartridge Performance Check

Any lot of Florisil cartridges that does not meet the criteria above must be discarded and a new lot, meeting criteria, used for sample cleanup.

10.1.8.2.3 Sample Cleanup by Florisil Cartridge

The required Florisil cartridge size and the final volume of the extract after Florisil cleanup are a function of the GC autosampler that a laboratory uses. If the autosampler operates reliably with 1 mL of sample extract, then a 500 mg cartridge is used and the required final volume is 1 mL. If the autosampler requires more sample, prepare 2 mL of sample extract using a 1 g cartridge. Manual injection requires only a 1 mL final extract and a 500 mg cartridge.

10.1.8.2.3.1 Frequency of Sample Cleanup by Florisil Cartridge

All sample extracts are required to be cleaned up by the Florisil cartridge technique.

10.1.8.2.3.2 Procedure for Sample Cleanup by Florisil Cartridge

10.1.8.2.3.2.1 Attach the vacuum manifold to a water aspirator or to a vacuum pump with a trap installed between the manifold and the vacuum source. Adjust the vacuum pressure in the manifold to between 5 and 10 pounds of vacuum.

10.1.8.2.3.2.2 Place one Florisil cartridge into the vacuum manifold for each sample extract.

10.1.8.2.3.2.3 Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge on the vacuum manifold, by pulling a vacuum, and by passing at least 5 mL of the hexane/acetone solution through the cartridge. While the cartridges are being washed, adjust the vacuum applied to each cartridge so that the flow rate through each cartridge is approximately equal. DO NOT ALLOW THE CARTRIDGES TO GO DRY AFTER THEY HAVE BEEN WASHED.

10.1.8.2.3.2.4 After the cartridges on the manifold are washed, the vacuum is released, and a rack containing labeled 10 mL volumetric flasks is placed inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside of the appropriate volumetric flask as the manifold top is replaced.

10.1.8.2.3.2.5 After the volumetric flasks are in place, the vacuum to the manifold is restored, and a volume of extract equal to the required final volume (1 or 2 mL) from each sample, blank or matrix spike extract is transferred to the top frit of the appropriate Florisil cartridge. This must equal the final volume after Florisil cleanup.

10.1.8.2.3.2.6 Because the volumes marked on concentrator tubes are not necessarily accurate at the 1 mL level, the use of a syringe or a volumetric pipet is required to transfer the extract to the cleanup cartridge.

10.1.8.2.3.2.7 The pesticides/Aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL volumetric flasks held in the rack inside the vacuum manifold.

10.1.8.2.3.2.8 Transfer the eluate in each volumetric flask to a clean centrifuge tube or 10 mL vial. Use two additional 1 mL hexane rinses to ensure quantitative transfer of the cartridge eluate.

10.1.8.2.3.2.9 Adjust the extract to the same 1 or 2 mL aliquot volume as was taken for cleanup using either nitrogen blowdown (Section 10.1.7.2) or a micro Snyder column (Section 10.1.7.1). Measure the final volume with a syringe or by transferring the extract to a volumetric flask.

10.1.8.2.3.2.10 If sulfur cleanup is to be performed, proceed to Section 10.1.8.3. Otherwise, transfer the sample to a GC vial and label the vial. The extract is ready for GC/EC analysis.

10.1.8.3 Sulfur Cleanup

10.1.8.3.1 Introduction to Sulfur Cleanup

10.1.8.3.1.1 Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analyses of the later eluting pesticides. If crystals of sulfur are evident or if the presence of sulfur is suspected, sulfur removal must be performed. Interference which is due to sulfur is not acceptable. Sulfur can be removed by one of two methods, according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and remove the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean centrifuge tube or clean concentrator tube before proceeding with further sulfur cleanup.

10.1.8.3.1.2 If only part of a set of samples require sulfur cleanup, then, a sulfur cleanup blank is required for that part of the set (Section 12.1.3).

10.1.8.3.2 Frequency of Sulfur Cleanup

Sulfur removal is required for all sample extracts that contain sulfur.

10.1.8.3.3 Procedure for Sulfur Cleanup

10.1.8.3.3.1 Mercury Technique

Add one to three drops of mercury to each hexane extract in a clean vial. Tighten the top on the vial and agitate the sample for 30 seconds. Filter or centrifuge the extract. Pipet the extract to another vial and leave all solid precipitate and liquid mercury. If the mercury appears shiny, proceed to Section 10.2 and analyze the extract. If the mercury turns black, repeat sulfur removal as necessary. The extract transferred to the vial still represents the 1.0 or 2.0 mL final volume. CAUTION: Waste containing mercury should be segregated and disposed of properly. NOTE: Mercury is a highly toxic metal and therefore must be used with great care. Prior to using mercury, it is recommended that the analyst become acquainted with proper handling and cleanup techniques associated with this metal.

10.1.8.3.3.2 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 1.0 or 2.0 mL final volume. The separation of the extract from the copper powder is necessary to prevent degradation of the pesticides. If the copper appears bright, proceed to Section 10.2 and analyze the extracts. If the copper changes color, repeat the sulfur removal procedure as necessary.

10.2 GC/EC Analysis

10.2.1 Introduction to Sample Analysis by GC/EC

10.2.1.1 Before samples or required blanks can be analyzed, the instrument must meet the initial calibration and calibration verification technical acceptance criteria. Sample analysis on both GC columns is required for <u>all</u> samples, blanks, matrix spikes, and matrix spike duplicates.

10.2.1.2 Sample extracts, standards, and blanks must be analyzed within an analytical sequence as defined in Section 10.2.2.1, under the same instrumental conditions.

10.2.1.3 Set up the GC/EC system per the requirements in Section 9.0. Unless ambient temperature on-column injection is used (see Section 9.1.4), the injector must be heated to at least 200 °C. The optimized gas chromatographic conditions from Section 9.1 must be used.

10.2.2 Procedure for Sample Analysis by GC/EC

The injection must be made on-column by using either automatic or manual injection. If autoinjectors are used, 1 µL injection volumes may be used. Manual injections shall use at least 2 µL injection volumes. The same injection volume must be used for all standards, samples, MS/MSD, and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2 µL. However, the same injection volume must be used for all analyses.

10.2.2.1 Analytical Sequence

All 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
	.	Subsequent samples
	.	
	.	
12 hr.	.	Last Sample
	1st injection past 12 hr.	Instrument blank
	2nd and 3rd injections past 12 hr.	Individual Standard Mixtures A and B
	.	Sample
	.	
	.	
	.	Subsequent samples
	.	
Another 12 hr.	.	Last Sample
	1st injection past 12 hr.	Instrument blank
	2nd injection past 12 hr.	PEM
	.	Sample
	.	
	.	
	.	Subsequent samples

Time	Injection #	Material Injected
Another 12 hr.		Last Sample
	1st injection past 12 hr.	Instrument blank
	2nd and 3rd injections past 12 hr.	Individual Standard Mixtures A and B
		Sample
	etc.	

10.2.2.1.1 NOTE: The first 12 hours are counted from injection #16 (the instrument blank at the end of the initial calibration sequence), not from injection #1. Samples may be injected until 12 hours have elapsed. All subsequent 12-hour periods are timed from the injection of the instrument blank that brackets the front end of the samples. Because the 12-hour time period is timed from injection of the instrument blank until the injection of the last sample, each 12-hour period may be separated by the length of one chromatographic run, that of the analysis of the last sample. While the 12-hour period may not be exceeded, the laboratory may run instrument blanks and standards more frequently for instance, to accommodate staff working 8-hour shifts.

10.2.2.1.2 After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks, PEMs and Individual Standard Mixtures A and B are analyzed at the required frequency. This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the Contractor: these must also satisfy the criteria presented in Section 9 in order to continue the run sequence.

10.2.2.1.3 An analysis sequence must also include all required matrix spike/matrix spike duplicate and method (and/or sulfur) blank analyses, but the Contractor may decide at what point in the sequence they are to be analyzed.

10.2.2.1.4 The requirements for the analysis sequence apply to both GC columns and for all instruments used for these analyses.

10.2.3 Sample Dilutions

10.2.3.1 All samples must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined in Section 11.3).

10.2.3.2 If the response of any single component pesticide is greater than the response of that analyte in the initial calibration high pointstandard, then the extract must be diluted to have the response of that analyte between the initial calibration low point and high point standards.

10.2.3.3 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 sample must be diluted to have the response of the largest peak in the multicomponent analyte between the responses of the initial calibration midpoint and high point standards of that single component pesticide.

10.2.3.4 If dilution is employed solely to bring a peak within the calibration range or to get a multicomponent pattern on scale, the results for both the more and the less concentrated extracts must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

10.2.3.5 If the Contractor has reason to believe that diluting the final extracts will be necessary, *a less diluted run may still be required. If an acceptable chromatogram (as defined in Section 11.3) is achieved with the diluted extract, then:*

- *If the dilution factor is greater than 10, an additional extract 10 times more concentrated than the diluted sample extract must be injected and reported with the sample data.*

- *If the dilution factor is less than or equal to 10, then an undiluted sample extract must be injected and reported with the sample data.*

If the analysis of the most concentrated extract does not meet the requirement for dilution in Section 11.3.5, then the analysis is at no additional cost to the Agency.

10.2.3.6 When diluted, the chromatographic data for the single component pesticides must be able to be reported at greater than 10.0 percent of full scale but less than 100.0 percent of full scale.

10.2.3.7 When diluted, multicomponent analytes must be able to be reported at greater than 25.0 percent of full scale but less than 100.0 percent of full scale.

10.2.3.8 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 must be submitted in the data package.

10.2.3.9 Samples with analytes detected at a level greater than the high calibration point must be diluted until the response is within the linear range established during calibration or to a maximum of 1:100,000.

10.2.3.10 If the response is still above the high calibration point after the dilution of 1:100,000, the Contractor shall contact SMO immediately.

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

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

10.2.3.13 Sample dilutions must be made quantitatively. Dilute the sample extract with hexane.

10.2.3.14 Do not submit data for more than two analyses, i.e., *from* the original sample extract and one dilution, or, from the most concentrated dilution analyzed and one further dilution. This statement does not refer to reanalyses required due to failed technical acceptance criteria.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Compounds

11.1.1.1 The laboratory will identify and quantitate analyte peaks based on the RT windows and the calibration factors of the midpoint standard (single component pesticides) established during the initial calibration sequence.

11.1.1.2 Analytes are identified when peaks are observed in the RT window for the analyte on both GC columns.

11.1.1.3 A set of three to five major peaks is selected for each multicomponent analyte. The retention time window for each peak is 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 windows of the corresponding peaks of the standard on both GC columns. Calibration factors used to quantitate toxaphene and the Aroclors are based on the single-point calibration standard analyzed during the initial calibration. The number of potential quantitation peaks is listed in Table 2.

11.1.1.4 When any multicomponent analyte is detected in a sample, a standard must be run within 72 hours of the analyte's detection (from time of injection), and within a valid 12-hour sequence.

11.1.1.5 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 co-eluting analytes or matrix interferences, or both. Because of the alteration of these materials in the environment, multicomponent analytes in samples may give patterns similar to, but not identical with, those of the standards.

11.1.1.6 If more than one multicomponent analyte is observed in a sample, the Contractor must choose different peaks to quantitate each multicomponent analyte. A peak common to both analytes present in the sample must not be used to quantitate either compound.

11.1.2 GC/MS Confirmation of Pesticides and Aroclors

11.1.2.1 Any pesticide or Aroclor analyte listed in Exhibit C for which a concentration is reported from a GC/EC analysis must have the identification confirmed by GC/MS if the concentration is sufficient for that purpose. The following paragraphs are to be used as guidance in performing GC/MS confirmation. If the Contractor fails to perform GC/MS confirmation as appropriate, the Agency may require reanalysis of any affected samples at no additional cost to the Agency.

11.1.2.2 The GC/MS confirmation may be accomplished by one of three general means:

- Examination of the semivolatile GC/MS library search results (i.e.,TIC data), or
- A second analysis of the semivolatile extract, or
- Analysis of the pesticide/Aroclor extract, following any solvent exchange and concentration steps that may be necessary.

11.1.2.3 The semivolatile GC/MS analysis procedures outlined in Exhibit D SVOA are based on the injection into the instrument of approximately 20 ng of a target compound in a 2 µL volume. The semivolatile CRQL values in Exhibit C are based on the sample concentration that corresponds to an extract concentration of 10 ng/µL of target analyte. However, these are quantitation limits, and the detection of analytes and generation of reproducible mass spectra will routinely be possible at levels 3-10 times lower. The sample concentration corresponding to 10 ng/µL in extract will depend on the sample matrix.

11.1.2.3.1 For water samples, 20 ng/2µL corresponds to a sample concentration of 10 µg/L.

11.1.2.3.2 For soil/sediment samples prepared according to the semivolatile low level soil/sediment method (i.e., 30 g of soil/sediment), the corresponding sample concentration is 330 µg/Kg.

11.1.2.3.3 For soil/sediment samples prepared according to the semivolatile medium level soil/sediment method (i.e., 1 g of soil/sediment), the corresponding sample concentration is 10,000 µg/Kg.

11.1.2.3.4 Therefore, based on the values given above, any pesticide sample in which compound concentration in the sample extract is greater than or equal to 10 ng/µL for single component pesticides, 50 ng/µL for Aroclors, and 125 ng/µL for Toxaphene should enable the laboratory to confirm the pesticide/Aroclor by GC/MS analysis of the semivolatile extract.

11.1.2.4 In order to confirm the identification of the target pesticide/Aroclor, the laboratory must also analyze a reference standard for the analyte. In order to demonstrate the ability of the GC/MS system to identify the analyte in question, the concentration of the standard should be 10 ng/µL for single component pesticides, 50 ng/µL for Aroclors, and 125 ng/µL for Toxaphene.

11.1.2.5 To facilitate the confirmation of the single component pesticide analytes from the semivolatile library search data, the laboratory may wish to include these analytes in the semivolatile continuing calibration standard at a concentration of 10 ng/µL or less. Do

not include the Aroclors and toxaphene mixture in the semivolatile initial and continuing calibration standard. If added to this GC/MS standard, the response factors, retention times, etc. For these analytes would be reported on the GC/MS quantitation report, but not on the GC/MS calibration data reporting forms. As only a single concentration of each analyte would be analyzed, no linearity (%RSD) or percent difference criteria would be applied to the response factors for these additional analytes.

11.1.2.6 The laboratory is advised that library search results from the NIST/EPA/NIH (May 1992 release or later) and Wiley (1991 release or later) mass spectral library will not likely list the name of the pesticide/Aroclor analyte as it appears in this SOW, hence, the mass spectral interpretation specialist is advised to compare the CAS Registry numbers for the pesticides/Aroclors to those from the library search routine.

11.1.2.7 If the analyte cannot be confirmed from the semivolatile library search data for the original semivolatile GC/MS analysis, the laboratory may analyze another aliquot of the semivolatile sample extract after further concentration of the aliquot. This second aliquot must either be analyzed as part of a routine semivolatile GC/MS analysis, including instrument performance checks (DFTPP), calibration standards containing the pesticides/Aroclors as described in Section 11.1.2.5, or it must be analyzed along with separate reference standards for the analytes to be confirmed.

11.1.2.8 If the analyte cannot be confirmed by either the procedures in Sections 11.1.2.5 or 11.1.2.7, then an aliquot of the extract prepared for the GC/EC analysis must be analyzed by GC/MS, following any necessary solvent exchange and concentration steps. As in Section 11.1.2.4, analysis of a reference standard is required if the GC/MS continuing calibration standard does not contain the analyte to be confirmed.

11.1.2.9 Regardless of which of the three approaches above is used for GC/MS confirmation, the appropriate blank must also be analyzed by GC/MS to demonstrate that the presence of the analyte was not the result of laboratory contamination. If the confirmation is based on the analysis of the semivolatile extract, then the semivolatile method blank extracted with the sample must also be analyzed. If the confirmation is based on the analysis of the extract prepared for the GC/EC analysis, then the pesticide method blank extracted with the sample must be analyzed.

11.1.2.10 If the identification of the analyte cannot be confirmed by any of the GC/MS procedures above and the concentration calculated from the GC/EC analysis is greater than or equal to the concentration of the reference standard analyzed by GC/MS, then report the analyte as undetected, adjust the sample quantitation limit (the value associated with the "U" qualifier) to a sample concentration equivalent to the concentration of the GC/MS reference standard, and qualify the results on Form I with one of the laboratory defined qualifiers ("X," "Y," or "Z"). In this instance, define

the qualifier explicitly in the SDG Narrative, and describe the steps taken to confirm the analyte in the SDG Narrative.

11.1.2.11 For GC/MS confirmation of single component analytes, the required deliverables are copies of the library search results (best TIC matches) or analyte spectrum and the spectrum of the reference standard. For multicomponent analytes, spectra of three characteristic peaks are required for both the sample component and the reference standard.

11.1.2.12 The purpose of the GC/MS analysis for the single component pesticides is for identification. The purpose of the GC/MS analysis for the multicomponent analytes is to confirm the presence of chlorinated biphenyls in Aroclor and the presence of chlorinated camphenes in Toxaphene. The GC/MS analytical results for the pesticides/Aroclors shall not be used for quantitation and the GC/MS results shall not be reported on Form I and Form X. The exception noted in Section 11.1.2.10 applies only to analytes that cannot be confirmed above the reference standard concentration.

11.2 Calculations

11.2.1 Target Compounds

The concentrations of the single component pesticides and surrogates are calculated separately for both GC columns by using the following equations.

11.2.1.1 Water

EQ. 13

$$\text{Concentration } \mu g/L = \frac{(A_x)\,(V_t)\,(Df)\,(GPC)}{(CF)\,(V_o)\,(V_i)}$$

Where,

A_x = Area of the peak for the compound to be measured.
CF = Calibration factor from the initial calibration for the midpoint concentration external standard (area per ng)
V_o = Volume of water extracted in milliliters (mL).
V_i = Volume of extract injected in microliters (μL). (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)

V_t = Volume of the concentrated extract in microliters (μL). (If GPC is <u>not</u> performed, then V_t = 10,000 μL. If GPC is performed, then V_t = 5,000 μL.)

Df = Dilution factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\mu L \text{ most conc. extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

GPC = GPC factor. (If no GPC is performed, GPC = 1. If GPC is performed, then GPC = 2.0

11.2.1.2 Soil/Sediment

EQ. 14

$$\text{Concentration } \mu g/Kg \text{ (Dry weight basis)} = \frac{(A_x)\,(V_t)\,(Df)\,(GPC)}{(CF)\,(V_i)\,(W_s)\,(D)}$$

Where,

A_x and CF are as given for water, above.
V_t = 5,000 μL.
V_i = Volume of extract injected in microliters (μL). (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)

$$D = \frac{100 - \%\text{ moisture}}{100}$$

W_s = Weight of sample extracted in grams (g)
Df = Dilution factor. The dilution factor for analysis of soil/sediment samples by this method is defined as follows:

$$\frac{\mu L \text{ most conc. Extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

GPC = GPC factor = 2.

11.2.1.2.1 The GPC factor is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collected after GPC to 5.0 mL rather than 10.0 mL for water samples not subjected to GPC maintains the sensitivity of the soil/sediment method comparable to that of the water method, but correction of the numerical results is still required.

11.2.1.2.2 Note that the calibration factors used for the quantitation of the single component pesticides are the calibration factors from the midpoint concentration on standard in the most recent initial calibration.

11.2.1.2.3 Because of the likelihood that compounds co-eluting with the target compounds will cause positive interferences and increase the concentration determined by the method, the lower of the two concentrations calculated for each single component pesticide is reported on Form I. In addition, the concentrations calculated for both the GC columns are reported on Form X, along with a percent difference comparing the two concentrations. The percent difference is calculated according to Equation 15.

EQ. 15

Where,

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

$Conc_H$ = 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

11.2.1.2.4 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.

11.2.1.2.5 The quantitative determination of Toxaphene or Aroclors is somewhat different from that of single component pesticides. Quantitation of peaks within the detector linear range CRQL to > 16 times CRQL is based on a single calibration point assuming linear detector response. Alternatively, a linear calibration range may be established during a run sequence by a three-point calibration curve for any multicomponent analyte. If the concentration is calculated to be $1C^6$ times the CRQL, the Contractor shall contact SMO immediately.

11.2.1.2.6 The quantitation of toxaphene or Aroclors must be accomplished by comparing the heights or the areas of each of the three to five major peaks of the multicomponent analyte in the sample with the calibration factor for the same peaks established during the initial calibration sequence. The concentration of multicomponent analytes is calculated by using Equations 13 and 14, where A_x is the area for each of the major peaks of the multicomponent analyte. The concentration of each peak is determined and then a mean concentration for the three to five major peaks is determined on each column.

11.2.1.2.7 The reporting requirements for Toxaphene and the Aroclors are similar to those for the single component analytes, except that the lower mean concentration (from three to five peaks) is reported on Form I, and the two mean concentrations reported on Form X. The two mean concentrations are compared by calculating the percent difference using Equation 15.

11.2.2 CRQL Calculation

If the adjusted CRQL is less than the CRQL listed in Exhibit C (Pesticides), report the CRQL in Exhibit C (Pesticides).

11.2.2.1 Water Samples

EQ. 16

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(V_x)\,(V_t)\,(V_y)\,(Df)}{(V_0)\,(V_c)\,(V_i)}$$

Where,

V_t, Df, V_0, and V_i are as given in Equation 13.
V_x = Contract sample volume (1000 mL).
V_y = Contract injection volume (1 μL or 2 μL).
V_c = Contract concentrated extract volume (10,000 μL if GPC was not performed and 5,000 μL if GPC was performed).

11.2.2.2 Soil/Sediment Samples

EQ. 17

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)\,(V_t)\,(V_y)\,(Df)}{(W_s)\,(V_c)\,(V_i)\,(D)}$$

Where,

V_t, Df, W_s, V_i and D are as given in Equation 14.
W_x = Contract sample weight (30 g).
V_y = Contract injection volume (1 μL or 2 μL).
V_c = Contract concentrated extract volume (GPC is required: 5,000 μL).

11.2.3 Surrogate Recoveries

11.2.3.1 The concentrations of the surrogates are calculated separately for each GC column in a similar manner as the other analytes, using Equations 13 and 14. Use the calibration factors from the midpoint concentration of Individual Standard Mixture A from the initial calibration. The recoveries of the surrogates calculated for each GC column according to Equation 12, repeated below.

EQ. 12

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Quantity determined by analysis
Q_a = Quantity added

11.2.3.2 The advisory limits for the recovery of the surrogates are 30-150 percent for both surrogate compounds.

11.2.3.3 As these limits are only advisory, no further action is required by the laboratory; however, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory, and may result in questions from the Agency. Surrogate recovery data from both GC columns are reported (see Exhibit B).

11.3 Technical Acceptance Criteria for Sample Analysis

The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

11.3.1 Samples must be analyzed under the GC/EC operating conditions in Section 9. The instrument must have met all initial calibration, calibration verification, and blank technical acceptance criteria. Samples must be cleaned-up, when required, on a GPC meeting the technical acceptance criteria for GPC calibration and GPC calibration checks. Samples must be cleaned-up using florisil meeting the technical acceptance criteria for florisil. Sample data must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs and Individual Standard Mixtures A and B, as described in Section 10.2.2.1.

11.3.2 The samples must be extracted and analyzed within the contract required holding times.

11.3.3 The samples must have an associated method blank meeting the technical acceptance criteria for method blanks. When sulfur cleanup blanks are required, the samples must have associated with it a sulfur cleanup blank meeting the technical acceptance criteria for sulfur cleanup blanks.

11.3.4 The retention time for each of the surrogates must be within the retention time window as calculated in Section 9 for both GC columns.

11.3.5 No target analyte concentrations may exceed the upper limit of the initial calibration or else the extract must be diluted and reanalyzed.

11.3.6 A standard for any identified multicomponent analyte must be analyzed during a valid analytical sequence on the same instrument and column within 72 hours of its detection in a sample.

11.3.7 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 be verified only from an on-scale chromatogram. The identification of multicomponent analytes 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.

11.3.7.1 When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low point standard of the initial calibration associated with those analyses.

11.3.7.2 Chromatograms must display single component pesticides detected in the sample at less than full scale.

11.3.7.3 Chromatograms must display the largest peak of any multicomponent analyte detected in the sample at less than full scale.

11.3.7.4 If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale.

11.3.7.5 If an extract must be diluted, chromatograms must display the peaks chosen for quantitation of multicomponent analytes between 25 and 100 percent of full scale.

11.3.7.6 For any sample or blank, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

11.3.7.7 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

11.3.7.8 If the chromatogram of any sample needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample analysis technical acceptance criteria MUST be met before data are reported. Samples contaminated from laboratory sources or associated with a contaminated method blank or sulfur cleanup blank will require re-extraction and reanalysis at no additional cost to the Agency. Any samples analyzed that do not meet the technical acceptance criteria will require re-extraction and/or reanalysis at no additional cost to the Agency.

11.4.2 If the sample analysis technical acceptance criteria are not met, check calculations, surrogate solutions, and instrument performance.

It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria, in which case, the affected samples must be reanalyzed at no additional cost to the Agency after the corrective action.

11.4.3 The extract from samples which were cleaned-up by GPC using an automated injection system and have surrogate recoveries outside the lower advisory surrogate acceptance limits must be checked to assure that the proper amount was injected on the GPC column. If insufficient volume was injected, the sample must be reprepared and reanalyzed at no additional cost to the Agency.

11.4.4 If sample chromatograms have a high baseline or interfering peaks, inspect the system to determine the cause of the problem (e.g., carryover, column bleed, dirty ECD, contaminated gases, leaking septum, etc.). After correcting the problem, analyze an instrument blank to demonstrate that the system is functioning properly. Reanalyze the sample extracts. If the problem with the samples still exists, then those samples must be re-extracted and reanalyzed. Samples which cannot be made to meet the given specifications after one re-extraction and three-step cleanup (GPC, Florisil, and sulfur cleanups) are reported in the SDG Narrative and do not require further analysis.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Introduction

There are two types of blanks always required by this method: the method blank and the instrument blank. A separate sulfur cleanup blank may be required if all samples associated with a given method blank are not 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 acceptance criteria for the sample analysis acceptance criteria to be met.

12.1.2 Method Blanks

12.1.2.1 Summary of Method Blanks

A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.2.2 Frequency of Method Blanks

A method blank must be extracted once for the following, whichever is most frequent, and analyzed on each GC/EC system used to analyze samples:

- Each SDG (not to exceed 20 field samples *excluding matrix spikes/matrix spike duplicates*), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted

12.1.2.3 Procedure for Method Blank Preparation

12.1.2.3.1

For pesticide/Aroclor analyses, a method blank for water samples consists of a 1 L volume of reagent water spiked with 1.*0* mL of the surrogate spiking solution (Section 7.2.4.1). For soil/sediment samples, the method blank consists of 30 g of sodium sulfate spiked with 2.*0* mL of the surrogate spiking solution.

12.1.2.3.2

Extract, concentrate, and analyze method blanks according to Section 10.

12.1.2.3.3 Calculate method blank results according to Section 11.

12.1.2.4 Technical Acceptance Criteria for Method Blanks

12.1.2.4.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.1.2.4.2 All method blanks must be prepared and analyzed at the frequency described in Section 12.1.2.2, using the procedure above and in Section 10 on a GC/EC system meeting the initial calibration and calibration verification technical acceptance criteria. Method blanks must undergo cleanup, when required, on a GPC meeting the technical acceptance criteria for GPC calibration and GPC calibration checks. Method blanks must be cleaned-up using Florisil meeting the technical acceptance criteria for Florisil. Method blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMS, and individual standard mixtures A and B as described in Section 10.2.2.1.

12.1.2.4.3 The concentration of the target compounds (Exhibit C (Pesticides)) in the method blank must be less than the CRQL for each target compound.

12.1.2.4.4 The method blank must meet all sample technical acceptance criteria in Sections 11.3.4 to 11.3.7.

12.1.2.4.5 Surrogate recoveries must fall within the acceptance windows of 30-150%. In the case of the method blank(s), these limits are <u>not</u> advisory.

12.1.2.5 Corrective Action for Method Blanks

12.1.2.5.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the system to be out of control.

12.1.2.5.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and sample processing hardware that lead to discrete artifacts and/or elevated baselines be investigated and appropriate corrective actions be taken and documented before further sample analysis. All samples associated with a contaminated method blank must be re-extracted/reanalyzed at no additional cost to the Agency.

12.1.2.5.3 If the surrogate recoveries in the method blank do not meet the acceptance criteria listed in Section 12.1.2.4.5, first reanalyze the method blank. If surrogate recoveries do not meet the acceptance criteria after reanalysis, the method blank

and all samples associated with that method blank must be re-extracted and reanalyzed at no additional cost to the Agency.

12.1.2.5.4 If the method blank failed to meet the criteria listed in Sections 12.1.2.4.2 and 12.1.2.4.4, then there is an instrument problem. Correct the instrument problem and reanalyze the method blank.

12.1.3 Sulfur Cleanup Blanks

12.1.3.1 Summary of Sulfur Cleanup Blanks

The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and carried through the sulfur cleanup and analysis procedures. The purpose of the sulfur cleanup blank is to determine the levels of contamination associated with the separate sulfur cleanup steps.

12.1.3.2 Frequency of Sulfur Cleanup Blanks

The sulfur cleanup blank is prepared separately 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 the method blank must be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

12.1.3.3 Procedure for Sulfur Cleanup Blank

12.1.3.3.1 The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. The sulfur blank must also contain the surrogates at the same concentrations as the sample extracts (assuming 100.0 percent recovery). Therefore, add 0.1 mL of the surrogate solution to 0.9 mL of hexane in a clean vial, or for a sulfur blank with a final volume of 2 mL, add 0.2 mL of the surrogate solution to 1.8 mL of hexane in a clean vial.

12.1.3.3.2 Proceed with the sulfur removal (Section 10.1.8.3.3.1 or 10.1.8.3.3.2) using the same technique (mercury or copper) as the samples associated with the blank.

12.1.3.3.3 Analyze the sulfur cleanup blank according to Section 10.2. Assuming that the material in the sulfur cleanup blank resulted from the extraction of a 1 L water sample, calculate the concentration of each analyte using the equation in Section 11.2.1.1. Compare the results to the CRQL values for water samples in Exhibit C (Pesticides).

12.1.3.4 Technical Acceptance Criteria For Sulfur Cleanup Blanks

12.1.3.4.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.1.3.4.2 All sulfur cleanup blanks must be prepared and analyzed at the frequency described in Section 12.1.3.2 using the procedure referenced in Section 12.1.3.3 on a GC/EC system meeting the initial calibration and calibration verification technical acceptance criteria.

12.1.3.4.3 Sulfur cleanup blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and Individual Standard Mixtures A and B, as described in Section 10.2.2.1.

12.1.3.4.4 The concentration of the target compounds (Exhibit C (Pesticides)) in the sulfur cleanup blank must be less than the CRQL for each target compound.

12.1.3.4.5 The sulfur cleanup blank must meet all sample technical acceptance criteria in Sections 11.3.4 to 11.3.7.

12.1.3.4.6 Surrogate recoveries must fall within the acceptance windows of 30-150%. In the case of the sulfur cleanup blank, these limits are not advisory.

12.1.3.5 Corrective Action for Sulfur Cleanup Blanks

12.1.3.5.1 If a sulfur cleanup blank does not meet the technical acceptance criteria, the Contractor must consider the system to be out of control.

12.1.3.5.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the Contractor's responsibility to insure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and sample processing hardware that lead to discrete artifacts and/or elevated baselines be investigated and appropriate corrective actions be taken and documented before further sample analysis. All samples associated with a contaminated sulfur cleanup blank must be re-extracted/reanalyzed at no additional cost to the Agency.

12.1.3.5.3 If the surrogate recoveries in the sulfur cleanup blank do not meet the acceptance criteria listed in Section 12.1.3.4.5, first reanalyze the sulfur cleanup blank. If surrogate recoveries do not meet the acceptance criteria after reanalysis, the sulfur cleanup blank and all samples associated with that sulfur cleanup blank must be re-extracted and reanalyzed at no additional cost o the Agency.

12.1.3.5.4 If the sulfur cleanup blank failed to meet the criteria used in Sections 12.1.3.4.2 and 12.1.3.4.5, then there is an instrument problem. Correct the instrument problem and reanalyze the sulfur cleanup blank.

12.1.4 Instrument Blanks

12.1.4.1 Summary of Instrument Blanks

An instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry over of analytes from standards or highly contaminated samples into other analyses.

12.1.4.2 Frequency of Instrument Blanks

The first analysis in a 12-hour analysis sequence must be an instrument blank. All acceptable sample analyses are to be bracketed by acceptable instrument blanks, as described in Section 10.2.2.1. If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an instrument blank must be analyzed to initiate a new 12-hour sequence.

12.1.4.3 Procedure for Instrument Blanks

12.1.4.3.1 Prepare the instrument blank by spiking the surrogates into hexane or iso-octane for a concentration of 20 ng/mL of tetrachloro-m-xylene and decachlorobiphenyl.

12.1.4.3.2 Analyze the instrument blank according to Section 10.2 at the frequency listed in Section 12.1.4.2.

12.1.4.3.3 For comparing the results of the instrument blank analysis to the CRQLs, assume that the material in the instrument resulted from the extraction of a 1 L water sample and calculate the concentration of each analyte using the equation in Section 11.2.1.1. Compare the results to <u>one-half</u> the CRQL values for water samples in Exhibit C (Pesticides).

12.1.4.4 Technical Acceptance Criteria for Instrument Blanks

12.1.4.4.1 *The requirements below apply independently to each GC Column and to all instruments used for these analyses. Quantitation must be performed and reported independently (on Form I PEST) for each GC Column.*

12.1.4.4.2 All instrument blanks must be prepared and analyzed at the frequency described in Section 12.1.4.2, using the procedure in Section 12.1.4.3 on a GC/EC system meeting the initial calibration and calibration verification technical acceptance criteria.

12.1.4.4.3 The concentration of each of the target analytes (Exhibit C (Pesticides)) in the instrument blank must be less than 0.5 times the CRQL for that analyte.

12.1.4.4.4 The instrument blank must meet all sample technical acceptance criteria in Sections 11.3.4 to 11.3.7

12.1.4.5 Corrective Action for Instrument Blanks

12.1.4.5.1 If analytes are detected at greater than half 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. After an acceptable instrument blank is run, all samples which were considered suspect as defined by the criteria described above must be reinjected during a valid run sequence at no additional cost to the Agency and must be reported.

12.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

12.2.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the methods used for pesticide/Aroclor analyses, the Agency has prescribed a mixture of pesticide/Aroclor target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

12.2.2 Frequency of MS/MSD Analysis

12.2.2.1 A matrix spike and matrix spike duplicate must be extracted and analyzed *for* every 20 *field* samples of *a similar* matrix. NOTE: There is no differentiation between "low" and "medium" level soil/sediment samples in this method. Therefore only one soil/sediment MS/MSD is to be submitted per Sample Delivery Group (SDG). *MS/MSD samples shall be analyzed unless otherwise specified on the Traffic Report (TR). If no MS/MSD samples are specified on the TR, the Contractor shall contact SMO to confirm that MS/MSD analyses are not required.*

12.2.2.2 As part of the Agency's QA/QC program, water rinsate samples and/or field blanks may be delivered to a laboratory for analysis. Do not perform MS/MSD analysis on a water rinsate sample or field blank.

12.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample volume remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify the Region (through SMO) that insufficient-sample was received and identify the EPA sample selected for the MS/MSD analysis. The rationale

for the choice of another sample other than the one designated by the Agency shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample volume remaining in any of the samples in an SDG to perform an MS/MSD, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no MS/MSD be performed, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the Region has requested MS/MSD analysis at a greater frequency then required by the contract, the Contractor shall contact SMO. SMO will contact the Region to determine which samples should have an MS/MSD performed on them. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative. *If this procedure is not followed, the Contractor will not be paid for MS/MSD analysis performed at a greater frequency than required by the contract.*

12.2.2.6 When a Contractor receives only performance evaluation (PE) samples, no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the MS/MSD when the Region did not designate samples to be used for this purpose. If the PE sample is received as an ampulated standard extract, the ampulated PE sample is not considered to be another matrix type.

12.2.3 Procedure for Preparing MS/MSD

12.2.3.1 Water Samples

For water samples, measure out two additional 1 L aliquots of the sample chosen for spiking. Adjust the pH of the samples (if required) and fortify each with 1 mL of matrix spiking solution. Using a syringe or volumetric pipet, add 1 mL of surrogate spiking solution to each sample. Extract, concentrate, cleanup, and analyze matrix spikes and matrix spike duplicate according to Section 10.0.

12.2.3.2 Soil/Sediment Samples

For soil/sediment samples weigh out two additional 30 g (record weight to the nearest 0.1 g) aliquots of the sample chosen for spiking. Add 1 mL of matrix spiking solution and 2 mL of surrogate solution. Extract, concentrate, cleanup, and analyze matrix spikes and matrix spike duplicates according to Section 10.0.

12.2.3.3 Note: Before any MS/MSD analysis, analyze the original sample, then analyze the MS/MSD at the same concentration as the most concentrated extract for which the original sample results will be reported. For example, if the original sample is to be reported

at a 1:1 dilution and a 1:10 dilution, then analyze and report the MS/MSD at a 1:1 dilution only. However, if the original sample is to be reported at a 1:10 dilution and a 1:100 dilution, then the MS/MSD must be analyzed and reported at a 1:10 dilution only. Do not further dilute the MS/MSD samples to get either spiked or nonspiked analytes within calibration range.

12.2.4 Calculations for MS/MSD

12.2.4.1 The percent recoveries and the relative percent difference between the recoveries of each of the compounds in the matrix spike samples will be calculated and reported by using the following equations:

EQ. 18

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike sample result
SR = Sample result
SA = Spike added

EQ. 19

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where,

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

12.2.4.2 The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

12.2.5 Technical Acceptance Criteria for MS/MSD

12.2.5.1 The requirements below apply independently to <u>each</u> GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.2.5.2 All MS/MSD must be prepared and analyzed at the frequency described in Section 12.2.2, using the procedure above and in Section 10 on a GC/EC system meeting the initial calibration, calibration verification, and blank technical acceptance criteria. MS/MSD must be cleaned-up, when required, on a GPC meeting the technical acceptance criteria for GPC calibration and GPC

calibration checks. MS/MSD must be cleaned-up using florisil meeting the technical acceptance criteria for florisil. MS/MSD must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMS, and individual standard mixtures A and B as described in Section 10.2.2.1.

12.2.5.3 The samples must be extracted and analyzed within the contract required holding times.

12.2.5.4 The retention time for each of the surrogates must be within the retention time window as calculated in Section 9 for both GC columns.

12.2.5.5 The limits for matrix spike compound recovery and RPD are given in Table 3. As these limits are only advisory, no further action by the laboratory is required. However, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questions from the Agency.

12.2.6 Corrective Action for MS/MSD

Any MS/MSD which fails to meet the technical acceptance criteria for MS/MSD must be reanalyzed at no additional cost to the Agency.

13.0 METHOD PERFORMANCE

Not Applicable

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction,* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult *The Waste management Manual for Laboratory Personnel,* available from the American Chemical Society at the address listed in Section 14.2.

16.0 REFERENCES

Not Applicable

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Table 1
Retention Time Windows for Single and
Multicomponent Analytes and Surrogates

Compound	Retention Time Window (minutes)
alpha-BHC	± 0.05
beta-BHC	± 0.05
gamma-BHC (Lindane)	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
Aldrin	± 0.05
alpha-Chlordane	± 0.07
gamma-Chlordane	± 0.07
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
4,4'-DDD	± 0.07
4,4'-DDE	± 0.07
4,4'-DDT	± 0.07
Endosulfan I	± 0.07
Endosulfan II	± 0.07
Endosulfan sulfate	± 0.07
Methoxychlor	± 0.07
Aroclors	± 0.07
Toxaphene	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

Table 2
Number of Potential Quantitation Peaks

Multicomponent Analyte	No. of Potential Quantitation Peaks
Aroclor 1016/1260	5/5
Aroclor 1221	3
Aroclor 1232	4
Aroclor 1242	5
Aroclor 1248	5
Aroclor 1254	5
Toxaphene	4

Table 3
Matrix Spike Recovery and
Relative Percent Difference Limits

Compound	%Recovery Water	RPD Water	%Recovery Soil	RPD Soil
gamma-BHC (Lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

EXHIBIT D

ANALYTICAL METHODS
FOR VOLATILES

Exhibit D - Analytical Methods for Volatiles

Table of Contents

1.0 SCOPE AND APPLICATION

1.1 In 1978, EPA Headquarters and Regional representatives designed analytical methods for the analysis of volatiles in hazardous waste samples. These methods were based on EPA Method 624, Purgeables. In 1980, these methods were adopted for use in the Contract Laboratory Program (CLP). As the requirements of CERCLA evolved, the CLP methods, as well as their precedent EPA 600 Series methods, established the basis for other EPA methods to perform the analysis of volatiles contained in hazardous waste samples (i.e., SW-846). The following CLP method has continuously improved to incorporate technological advancements promulgated by EPA, and has continued to set the standard for the preparation, extraction, isolation, identification, and reporting of volatiles at hazardous waste sites.

1.2 The analytical method that follows is designed to analyze water, sediment, and soil from hazardous waste sites for the *volatile* organic compounds on the Target Compounds List (TCL, see Exhibit C). The method includes sample preparation, screening to determine the approximate concentration of organic constituents in the sample, and the actual analysis which is based on a purge and trap gas chromatograph/mass spectrometer (GC/MS) method.

1.3 This analytical method includes the use of the Modified SW-846 Method 5035 for the preparation and analysis of low level soil/sediment samples. A detailed description of the sample preparation, analysis, and quality control procedures to be followed when this method option is requested can be found in Appendix B.

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

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

- Acetone, hexanone, 2-butanone, and 4-methyl-2-pentanone have poor purge efficiencies.

- 1,1,1-trichloroethane and all the dichloroethanes can dehydrohalogenate during storage or analysis.

- Chloromethane can be lost if the purge flow is too fast.

- Bromoform is one of the compounds most likely to be adversely affected by cold spots and/or active sites in the transfer lines. Response of its quantitation ion (m/z 173) is directly affected by the tuning of the GC/MS to meet the instrument performance criteria for 4-bromofluorobenzene (BFB) at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

2.0 SUMMARY OF METHOD

2.1 Water

An inert gas is bubbled through a 5 mL sample contained in a specifically designed purging chamber at ambient temperature. The purgeable compounds are efficiently 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 purgeable compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

2.2 Low *Level* Soil

An inert gas is bubbled through a mixture of reagent water and 5 g of sample contained in a specifically designed purging chamber that is held at an elevated temperature. The purgeable compounds are efficiently 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 purgeable compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

2.3 Medium *Level* Soil

A measured amount of soil is *collected/*extracted with methanol. A portion of the methanol is diluted to 5 mL with reagent water. An inert gas is bubbled through this solution in a specifically designed purging chamber at ambient temperature. The purgeable compounds are effectively 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 purgeable compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

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

4.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling.

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

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

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

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

6.0 EQUIPMENT AND SUPPLIES

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

6.1 Glassware

6.1.1 Syringes - 5 mL, gas-tight with shut-off valve. Micro syringes - 25 µL and larger, 0.006 inch ID needle.

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

6.1.3 Pasteur Pipets - disposable.

6.1.4 Vials and Caps - 2 mL for GC.

6.1.5 Volumetric Flasks.

6.1.6 Bottle - 15 mL, screw-cap, with Teflon cap liner.

6.2 pH Paper - wide range

6.3 Balances - analytical, capable of accurately weighing ± 0.0001 g, and a top-loading balance capable of weighing 100 g ± 0.01 g. The balances must be calibrated with class S weights or known reference weights once per each 12-hour work shift. The balances must be calibrated with class S weights at a minimum of once per month. The balances must also be annually checked by a certified technician.

6.4 Purge and Trap Device - consists of three separate pieces of equipment: the sample purge chamber, trap, and the desorber. Several complete devices are now commercially available.

6.4.1 The sample purge chamber must be designed to accept 5 mL samples with a water column at least 3 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.

6.4.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of absorbents: (starting from inlet) 0.5 cm silanized glass wool, 1 cm methyl silicone, 8 cm of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh), 8 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15 or equivalent), 7 cm of coconut charcoal (prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen), and 0.5 cm silanized glass wool. A description of the trap used for analysis shall be provided in the SDG Narrative.

6.4.3 The desorber should be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 220 °C during bakeout mode.

6.4.4 Trap Packing

6.4.4.1 2,6-Diphenylene oxide polymer, 60/80 mesh chromatographic grade (Tenax GC or equivalent).

6.4.4.2 Methyl silicone packing, 3.0 percent OV-1 on Chromasorb W, 60/80 mesh (or equivalent).

6.4.4.3 Silica gel, 35/60 mesh, Davison, grade 15 (or equivalent).

6.4.4.4 Coconut charcoal (prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen).

6.4.4.5 Alternate sorbent traps may be used if:

- The trap packing materials do not introduce contaminants which interfere with identification and quantitation of the compounds listed in Exhibit C (Volatiles).
- The analytical results generated using the trap meet the initial and continuing calibration technical acceptance criteria listed in the SOW and the CRQLs listed in Exhibit C (Volatiles).
- The trap can accept up to 1000 ng of each compound listed in Exhibit C (Volatiles) without becoming overloaded.

6.4.4.5.1 The alternate trap must be designed to optimize performance. Follow manufacturer's instructions for the use of its product. Before use of any trap, other than the one specified in Section 6.4.2, the Contractor must first meet the criteria listed in Section *6.4.4.5*. Once this has been demonstrated, the Contractor must document its use in each SDG Narrative by specifying the trap composition (packing material/brand name, amount of packing material). Other sorbent traps include, but are not limited to, Tenax/Silica Gel/Carbon Trap from EPA Method 524.2, Tenax - GC/Graphpac-D Trap (Alltech) or equivalent, and Vocarb 4000 Trap (Supelco) or equivalent.

6.4.4.5.2 The Contractor must maintain documentation that the alternate trap meets the criteria listed in Section *6.4.4.5*. The minimum documentation requirements are as follows:

6.4.4.5.2.1 Manufacturer provided information concerning the performance characteristics of the trap.

6.4.4.5.2.2 Reconstructed ion chromatograms and data system reports generated on the Contractor's GC/MS used for CLP analyses:

- From instrument blank analyses which demonstrate that there are no contaminants which interfere with the volatile analysis when using the alternate trap;

- From initial and continuing calibration standards analyzed using the trap specified in Section 6.4.4.

6.4.4.5.2.3 Based on Contractor generated data described above, the Contractor must complete a written comparison/review, which has been signed by the Laboratory Manager, certifying that:

- The alternate trap performance meets the technical acceptance criteria listed in Sections 9.3.5 and 9.4.5;

- The low point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;

- The high point initial calibration standard analysis was not overloaded;

- The alternate trap materials do not introduce contaminants which interfere with the identification and/or quantitation of the compounds listed in Exhibit C (Volatiles).

6.4.4.5.2.4 The documentation must be made available to the Agency during on-site laboratory evaluations or sent to the Agency upon request of the Technical Project Officer or the Administrative Project Officer.

6.4.5 The purge and trap apparatus may be assembled as a separate unit or be an integral unit coupled with a gas chromatograph.

6.5 A heater or heated bath capable of maintaining the purge chamber at 40 °C (± 1 °C) is to be used for low level soil/sediment analysis, but <u>not</u> for water or medium level soil/sediment analyses.

6.6 *Gas Chromatography/Mass Spectrometer (GC/MS) System*

6.6.1 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 6.4 and have all required accessories including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants, or flow controllers with rubber components are not to be used.

6.6.2 Gas Chromatography Columns - *a description of the column used for analysis shall be provided in the SDG Narrative.*

6.6.2.1 Packed columns - 6 ft. long x 0.1 inch ID glass, packed with 1.0

percent SP-1000 on Carbopack B (60/80) mesh or equivalent.

6.6.2.2 Capillary Columns

6.6.2.2.1 Minimum length 30 m x 0.53 mm ID VOCOL (Supelco) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.2.2 Minimum length 30 m x 0.53 mm ID DB-624 (J & W Scientific) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.2.3 Minimum length 30 m x 0.53 mm ID AT-624 (Alltech) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.2.4 Minimum length 30 m x 0.53 mm ID HP-624 (Hewlett-Packard) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.2.5 Minimum length 30 m x 0.53 mm ID RTx-624 (Restek) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.2.6 Minimum length 30 m x 0.53 mm ID BPX-624 (SGE) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.2.7 Minimum length 30 m x 0.53 mm ID CP-Sil 13CB (Chrompack) or equivalent fused silica widebore capillary column with 3 μm film thickness.

6.6.2.3 A capillary column is considered equivalent if:

- The column does not introduce contaminants which interfere with the identification and quantitation of the compounds listed in Exhibit C (Volatiles).
- The analytical results generated using the column meet the initial and continuing calibration technical acceptance criteria listed in the SOW, and the CRQLs listed in Exhibit C (Volatiles).
- The column can accept up to 1000 ng of each compound listed in Exhibit C (Volatiles) without becoming overloaded.
- The column provides equal or better resolution of the compounds listed in Exhibit C (Volatiles) than the columns listed in Section 6.6.2.2.

6.6.2.4 As applicable, follow the manufacturer's instructions for use of its product.

6.6.2.5 The Contractor must maintain documentation that the column met the criteria in Section 6.6.2.3. The minimum documentation is as follows:

6.6.2.5.1 Manufacturer provided information concerning the performance characteristics of the column.

6.6.2.5.2 Reconstructed ion chromatograms and data system reports generated on the GC/MS used for the CLP analyses:

- From instrument blanks which demonstrate that there are no contaminants which interfere with the volatile analysis when using the *alternate* column;
- From initial and continuing calibration standards analyzed using the alternate column.

6.6.2.5.3 Based on the Contractor-generated data described above, the Contractor shall complete a written review, signed by the Laboratory Manager, certifying that:

- The column performance meets the technical acceptance criteria in Sections 9.3.5 and 9.4.5;
- The low point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;
- The high point initial calibration standard analysis was not overloaded;
- The column does not introduce contaminants which interfere with the identification and/or quantitation of compounds listed in Exhibit C (Volatiles).

6.6.2.5.4 The documentation must be made available to the Agency during on-site laboratory evaluations or sent to the Agency upon request of the Technical Project Officer or Administrative Project Officer.

6.6.3 Mass Spectrometer - must be capable of scanning from 35 to 300 amu every 1 second or less to every 2 seconds or less utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng of BFB is injected through the gas chromatograph inlet. The instrument conditions required for the acquisition of the BFB mass spectrum are given in Section 9.

6.6.3.1 NOTE: The MS scan rate should 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 the outside of the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.6.4 GC/MS interface - any gas chromatograph to mass spectrometer interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest, and achieves all acceptance criteria, may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.6.5 Data system - a computer system 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 for the comparison of sample spectra against reference library spectra. The NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

6.6.6 Magnetic tape storage device - capable of recording data and must be suitable for long-term, off-line storage.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Reagent water - defined as water in which an interferant is not observed at or above the CRQL of the analytes of interest. Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g (1 lb) of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

7.1.1.1 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

7.1.1.2 Reagent water may also 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 and seal with a Teflon-lined septum and cap.

7.1.2 Methanol - pesticide quality or equivalent

7.2 Standards

7.2.1 Introduction

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

7.2.2 Stock Standard Solutions

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

7.2.2.2 Prepare stock standard solutions by placing about 9.8 mL of methanol into a 10 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.

7.2.2.3 Add the assayed reference material as described below.

7.2.2.3.1 If the compound is a liquid, using a 100 µL 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.

7.2.2.3.2 If the compound is a gas at room temperature, fill a 5 mL valved gas-tight syringe with the reference standard to the 5 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.

7.2.2.3.3 The procedure in Section 7.2.2.3.2 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.

7.2.2.3.4 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 97.0 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 97.0 percent, the weight must be corrected when calculating the concentration of the stock solution. See Exhibit E (Analytical Standards Requirements). 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.

7.2.2.3.5 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 the standard has degraded or evaporated.

7.2.3 Secondary Dilution Standards

7.2.3.1 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.

7.2.3.2 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.

7.2.4 Working Standards

7.2.4.1 System Monitoring Compound (SMC) Spiking Solution

Prepare a system monitoring compound spiking solution containing toluene-d8, 4-bromofluorobenzene (BFB), and 1,2-dichloroethane-d4 in methanol at a concentration of 25 µg/mL. Add 10 µL of this spiking solution into 5 mL of sample, sample extract or calibration standard for a concentration of 50 µg/L. Prepare fresh spiking solution weekly, or sooner, if the solution has degraded or evaporated.

7.2.4.2 Matrix Spiking Solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 25 µg/mL: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Prepare fresh

spiking solution weekly, or sooner, if the solution has degraded or evaporated.

7.2.4.3 Internal Standard Spiking Solution

Prepare an internal standard spiking solution containing bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene in methanol at a concentration of 25 µg/mL for each internal standard. Add 10 µL of this spiking solution into 5 mL of sample or calibration standard for a concentration of 50 µg/L. Prepare fresh spiking solution weekly, or sooner, if the solution has degraded or evaporated.

7.2.4.4 Instrument Performance Check Solution - 4-Bromofluorobenzene (BFB)

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

7.2.4.5 Calibration Standard Solution

Prepare a calibration standard solution containing all of the purgeable target compounds in methanol. The recommended concentration of the target compounds is 100 µg/mL. Prepare fresh calibration standard solutions weekly, or sooner, if solutions have degraded or evaporated.

7.2.4.6 Initial and Continuing Calibration Standards

7.2.4.6.1 Prepare five aqueous initial calibration standard solutions containing all of the purgeable target compounds and system monitoring compounds at 10, 20, 50, 100, and 200 µg/L levels. It is required that all three xylene isomers (o-, m-, and p-xylene) be present in the calibration standards at concentrations of each isomer equal to that of the other target compounds (i.e., 10, 20, 50, 100, and 200 µg/L). Similarly, the cis and trans isomers of 1,2-dichloroethene must both be present in the standards at concentrations of each isomer equal to that of the other target compounds.

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

7.2.4.6.2.1 Volumetric flask - add an appropriate volume of the 100 µg/mL calibration standard solution (Section 7.2.4.5) to an aliquot of reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcohol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the head of the flask.

7.2.4.6.2.2 Syringe - remove the plunger from a 5 mL "Luerlock" syringe. 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 5 mL minus the amount of calibration standard to be added. Withdraw the plunger slightly and add an appropriate volume of working calibration standard through the valve bore of the syringe. Close the valve and invert three times.

7.2.4.6.2.3 The 50 µg/L aqueous calibration standard solution is the continuing calibration standard.

7.2.4.6.3 The methanol contained in each of the aqueous calibration standards must not exceed 1.0 percent by volume.

7.2.5 Ampulated Standard Extracts

Standard solutions purchased from a chemical supply house as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions, prepared by the Contractor which are immediately ampulated in glass vials, may be retained for 2 years from the preparation date. Upon breaking the glass seal, the expiration times listed in Sections 7.2.2 through 7.3 will apply. The Contractor is responsible for assuring that the integrity of the standards has not degraded (see Section 7.3.5).

7.3 Storage of Standard Solutions

7.3.1 Store the stock standards in Teflon-sealed screw-cap bottles with zero headspace at -10 °C to -20 °C, and protect the standards from light. Once one of the bottles containing the stock standard solution has been opened, it may be used for no longer than one week.

7.3.2 Store secondary dilution standards in Teflon-sealed screw-cap bottles with minimal headspace at -10 °C to -20 °C. and protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to preparing working standards from them.

7.3.3 Aqueous standards may be stored for up to 24 hours if held in Teflon-sealed screw-cap vials with zero headspace at 4 °C (±2 °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 for up to 12 hours in purge tubes connected via the autosampler to the purge and trap device. All other working standards may be stored at -10 °C to -20 °C.

7.3.4 Purgeable standards must be stored separately from other standards.

7.3.5 The Contractor is responsible for maintaining the integrity of

standard solutions and verifying prior to use. This means that standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in the solution.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in glass containers having a total volume of at least 40 mL with a Teflon-lined septum and an open top screw-cap. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis. Headspace should be avoided. *Soil samples for medium level analysis may also be collected in pre-weighed vials containing 10 ml of methanol.* The specific requirements for site sample collection are outlined by the Region.

8.1.2 For collection of water samples, the containers must be filled in such a manner that no air bubbles pass through the sample as the container is being filled. Seal the vial so that no air bubbles are entrapped in it.

8.1.3 Water samples are preserved to a pH of 2 at the time of collection.

8.1.4 For collection of medium level soil samples with methanol, the sample vial, with 10 ml of methanol and all labeling, is weighed to the nearest 0.1 g prior to the addition of sample. Approximately 5 g of sample is added to the vial. The sample vial with sample is weighed to the nearest 0.1 g. The initial weight, final weight and sample weight will be recorded and provided to the laboratory.

8.1.5 All samples must be iced or refrigerated at 4 °C (±2 °C) from the time of collection until analysis.

8.2 Procedure for Sample Storage

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

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants and in a refrigerator used only for storage of volatile samples.

8.2.3 All volatile samples in an SDG must be stored together in the same refrigerator.

8.2.4 Storage blanks shall be stored with samples until all samples are analyzed.

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

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

8.3 Contract Required Holding Times

Analysis of water and soil/sediment samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR). As part of the Agency's QA program, the Agency may provide Performance Evaluation (PE) samples as standard extracts which the Contractor is required to prepare per the instructions provided by the Agency. *PE samples must be prepared and analyzed concurrently with the samples in the SDG.* The contract required 10 day holding time does not apply to PE samples received as standard extracts.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Conditions

9.1.1 Purge and Trap

9.1.1.1 The following are the recommended purge and trap analytical conditions. The conditions are recommended unless otherwise noted.

Purge conditions

Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 ± 0.1 minute
Purge Flow Rate:	25-40 mL/minute
Purge Temperature:	Ambient temperature for water or medium level soil/sediment samples (required); 40 °C low level soil/sediment samples (required)

Desorb Conditions

Desorb Temperature:	180 °C
Desorb Flow Rate:	15 mL/minute
Desorb Time:	4.0 ± 0.1 minute

Trap Reconditioning Conditions

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

9.1.1.2 Before initial use, condition the trap overnight at 180 °C by backflushing with at least 20 mL/minute flow of inert gas. Do not vent the trap effluent onto the analytical column. Prior to daily use, condition the trap by heating at 180 °C for 10 minutes while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to the analysis of samples.

9.1.1.3 Optimize purge and trap conditions for sensitivity and to minimize cross-contamination between samples. Once optimized, the same purge and trap conditions must be used for the analysis of all standards, samples, and blanks.

9.1.1.4 A moisture reduction/water management system may be used to improve the chromatographic performance by controlling moisture or water if:

- The system does not introduce contaminants which interfere with identification and quantitation of compounds listed in Exhibit C (Volatiles),
- The analytical results generated when using the moisture

reduction/water management system meet the initial and continuing calibration technical acceptance criteria listed in the SOW and the CRQLs listed in Exhibit C (Volatiles),

- All calibration standards and samples, including blanks and MS/MSDs, are analyzed under the same conditions,

- The Contractor performs acceptably on the Performance Evaluation samples using this system.

9.1.2 Gas Chromatograph

9.1.2.1 The following are the recommended GC analytical conditions. These conditions are recommended unless otherwise noted.

Packed columns

Carrier Gas:	Helium
Flow Rate:	30 mL/minute
Initial Temperature:	45 °C
Initial Hold Time:	3 minutes
Ramp Rate:	8 °C/minute
Final Temperature:	220 °C
Final Hold Time:	Until three minutes after all compounds listed in Exhibit C (Volatiles) elute (required)
Transfer Line Temperature:	250-300 °C

Capillary Columns

Carrier Gas:	Helium
Flow Rate:	15 mL/minute
Initial Temperature:	10 °C
Initial Hold Time:	1.0 - 5.0 (± 0.1) minutes
Ramp Rate:	6 °C/minute
Final Temperature:	160 °C
Final Hold Time:	Until three minutes after all compounds listed in Exhibit C (Volatiles) elute (required)

9.1.2.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, matrix spikes, and matrix spike duplicates.

9.1.2.3 For capillary columns, if the gaseous compounds chloromethane, bromomethane, vinyl chloride, and chloroethane fail to exhibit narrow, symmetrical peak shape, are not separated from the solvent front, or are not resolved greater than 90.0 percent from each other, then a subambient oven controller must be used, and the initial temperature must be less than or equal to 10 °C.

9.1.3 Mass Spectrometer

The following are the required mass spectrometer analytical conditions:

Electron Energy:	70 volts (nominal)
Mass Range:	35-300 amu
Scan Time:	To give at least 5 scans per peak, not to exceed 2 seconds per scan for capillary column.
	To give at least 5 scans per peak, not to exceed 3 seconds per scan for packed column.

9.2 GC/MS Calibration (Tuning) and Ion Abundance

9.2.1 Summary of GC/MS Performance Check

9.2.1.1 The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as perfluoro-tri-n-butylamine (FC-43) or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Section 7.2.4.4).

9.2.1.2 Prior to the analysis of any samples, including MS/MSDs, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing BFB.

9.2.2 Frequency of GC/MS Performance Check

The instrument performance check solution must be injected once at the beginning of each 12-hour period, during which samples or standards are to be analyzed. The twelve (12) hour time period for GC/MS instrument performance check (BFB), standards calibration (initial or continuing calibration criteria), blank and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after twelve (12) hours have elapsed according to the system clock.

9.2.3 Procedure for GC/MS Performance Check

9.2.3.1 The analysis of the instrument performance check solution may be performed as follows:

- As an injection of up to 50 ng of BFB into the GC/MS.
- By adding 50 ng of BFB to 5 mL of reagent water and analyzing the resulting solution as if it were an environmental sample (See Section 10).

9.2.3.2 The instrument performance check solution must be analyzed alone without calibration standards. NOTE: The calibration standards contain BFB as a system monitoring compound (SMC).

9.2.4 Technical Acceptance Criteria for GC/MS Performance Check

9.2.4.1 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

9.2.4.2 NOTE: All subsequent standards, samples, MS/MSD, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

9.2.4.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given in Table 1.

9.2.5 Corrective Action for GC/MS Performance Check

9.2.5.1 If the technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective actions to achieve the technical acceptance criteria.

9.2.5.2 BFB technical acceptance criteria must be met before any standards, samples, including MS/MSDs or required blanks are analyzed. Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis at no additional cost to the Agency.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to the analysis of samples and required blanks, and after the instrument performance check solution criteria have been met, each GC/MS system must be calibrated at five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the purgeable target compounds.

9.3.2 Frequency of Initial Calibration

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

9.3.2.2 If time remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples 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, 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.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Assemble a purge and trap device that meets the specifications in Section 6.4. Condition the device as described in Section 9.1.1.

9.3.3.2 Connect the purge and trap device to the gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Section 9.1.2.

9.3.3.3 Add 10 µL of the internal standard solution (Section 7.2.4.3) to each of the five aqueous calibration standard solutions containing the system monitoring compounds (Section 7.2.4.6) for a concentration of 50 µg/L at time of purge. Analyze each calibration standard according to Section 10.

9.3.3.4 Separate initial and continuing calibrations must be performed for water samples and low level soil/sediment samples (unheated purge vs. heated purge). Extracts of medium level soil/sediment samples may be analyzed using the calibrations of water samples.

The laboratory may run different matrices in the same 12-hour time period under the same tune, as long as separate calibrations are performed for each matrix within that 12-hour period.

9.3.4 Calculations for Initial Calibration

9.3.4.1 Calculate the relative response factor (RRF) for each volatile target and system monitoring compound using Equation 1. The primary characteristic ions used for quantitation are listed in Table 2 and Table 4. Assign the target compounds and system monitoring compound to an internal standard according to Table 3. If an interference prevents the use of a primary ion for a given internal standard, use a secondary ion listed in Table 4. NOTE: Unless otherwise stated, the area response of the primary characteristic ion is the quantitation ion.

EQ. 1

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (see Table 2)
A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (see Tables 3 and 4)
C_{is} = Concentration of the internal standard
C_x = Concentration of the compound to be measured

9.3.4.2 Calculating the relative response factor of the xylenes and the cis and trans isomers of 1,2-dichloroethene requires special attention. On packed columns, o- and p-xylene isomers co-elute. On capillary columns, the m- and p-xylene isomers co-elute. Therefore, when calculating the relative response factor in the equation above, use the area response (A_x) and concentration (C_x) of the peak that represents the single isomer on the GC column used for analysis.

9.3.4.3 The mean relative response factor ($\overline{RRF}$) must be calculated for all compounds.

9.3.4.4 Calculate the % Relative Standard Deviation (%RSD) of the RRF values over the working range of the curve.

EQ. 2

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

X_i = each individual value used to calculate the mean
$\overline{X}$ = the mean of n values
n = the total number of values

9.3.5 Technical Acceptance Criteria for Initial Calibration

9.3.5.1 All initial calibration standards must be analyzed at the concentration levels described in Section 7.2.4.6.1, and at the frequency described in Section 9.3.2 on a GC/MS system meeting the BFB technical acceptance criteria.

9.3.5.2 The relative response factor (RRF) at each calibration concentration for each purgeable target and system monitoring compound must be greater than or equal to the compound's minimum acceptable response factor listed in Table 5.

9.3.5.3 The %RSD for each target or system monitoring compound listed in Table 5 must be less than or equal to that value listed.

9.3.5.4 Up to two compounds may fail the criteria listed in Sections 9.3.5.2 and 9.3.5.3 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 percent.

9.3.5.5 Excluding those ions in the solvent front, and the combined xylenes in the 200 µg/L standard, no quantitation ion may saturate the detector. Follow the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, service the purge and trap device, or take other corrective actions to achieve the technical acceptance criteria.

9.3.6.2 Initial calibration technical acceptance criteria must be met before any samples or required blanks are analyzed. Any samples including MS/MSD or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the Agency.

9.4 Continuing Calibration

9.4.1 Summary of Continuing Calibration

Prior to the analysis of samples and required blanks and after BFB and initial calibration 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 system monitoring compounds to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the SOW.

9.4.2 Frequency of Continuing Calibration

9.4.2.1 A check of the calibration curve must be performed once every 12 hours (see Section 9.2.2 for the definition of the 12-hour time period). 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 (50 µg/L).

9.4.2.2 If time does not remain in the 12-hour period beginning with the injection of the instrument performance check solution, a new injection of the instrument performance check solution must be made. If the new injection meets the ion abundance criteria for BFB, then a continuing calibration standard may be injected.

9.4.3 Procedure for Continuing Calibration

9.4.3.1 Set up the purge and trap GC/MS system per the requirements in

Section 9.1.1.

9.4.3.2 Add 10 µL of internal standard solution (Section 7.2.4.3) to the 5 mL syringe or volumetric flask containing the continuing calibration standard (Section 7.2.4.6). Analyze the continuing calibration standard according to Section 10.

9.4.4 Calculations for Continuing Calibration

9.4.4.1 Calculate a relative response factor (RRF) for each target and system monitoring compound using Equation 1.

9.4.4.2 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 system monitoring compound using Equation 3.

EQ. 3

$$\%\text{Difference} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where,

RRF_c = Relative response factor from continuing calibration standard

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

9.4.5 Technical Acceptance Criteria for Continuing Calibration

9.4.5.1 The continuing calibration standard must be analyzed at the frequency described in Section 9.4.2 on a GC/MS system meeting the BFB and initial calibration technical acceptance criteria.

9.4.5.2 The relative response factor (RRF) for each purgeable target and system monitoring compound listed in Table 5 must be greater than or equal to the compound's minimum acceptable response factor listed in Table 5.

9.4.5.3 The relative response factor percent difference for each purgeable target and system monitoring compound listed in Table 5 must be less than or equal to the value listed.

9.4.5.4 Up to two compounds may fail the requirements listed in Sections 9.4.5.2 and 9.4.5.3 and still meet the minimum relative response factor criteria and percent difference criteria. However, these compounds must have a minimum relative response factor greater than or equal to 0.010 and the percent difference must be within the inclusive range of ±40.0 percent.

9.4.5.5 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument

operating manual to determine how saturation is indicated for your instrument.

9.4.6 Corrective Action for Continuing Calibration

9.4.6.1 If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3.3. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the continuing calibration technical acceptance criteria.

9.4.6.2 Continuing calibration technical acceptance criteria must be met before any samples, which include MS/MSD samples, 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 to the Agency.

10.0 PROCEDURE

10.1 Sample Preparation

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

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

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

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

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

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

10.1.3 Water Samples

10.1.3.1 All water samples must be allowed to warm to ambient temperature before analysis.

10.1.3.2 Prior to the analysis of samples, establish the appropriate purge and trap GC/MS operating conditions, as outlined in Section 9.1, analyze the instrument performance check solution (Section 9.2),

and calibrate the GC/MS system according to Sections 9.3 through 9.4.6.

10.1.3.3 If time remains in the 12-hour period (as described in Section 9.3.2), samples may be analyzed without analysis of a continuing calibration standard.

10.1.3.4 If time does not remain in the 12-hour period since the injection of the instrument performance check solution, both the instrument performance check solution and the continuing calibration standard must be analyzed before sample analysis may begin (see Section 9.4.2).

10.1.3.5 Adjust the purge gas (helium) flow rate to 25-40 mL/minute. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

10.1.3.6 Remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample or standard bottle which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5 mL. This process of taking an aliquot destroys the validity of the sample for future analysis so, if there is only one VOA vial, the analyst must fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly. Filling one 5 mL syringe would allow the use of only one syringe. If an analysis is needed from the second 5 mL syringe, it must be performed within 24 hours. Care must also be taken to prevent air from leaking into the syringe.

10.1.3.7 Add 10 μL of the system monitoring compound spiking solution (Section 7.2.4.1) and 10 μL of the internal standard spiking solution (Section 7.2.4.3) through the valve bore of the syringe, then close the valve. The system monitoring compounds and internal standards may be mixed and added as a single spiking solution. The addition of 10 μL of the system monitoring compound spiking solution to 5 mL of sample is equivalent to a concentration of 50 μg/L of each system monitoring compound.

10.1.3.8 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.1.3.9 Close both valves and purge the sample for 11.0 ($\pm$ 0.1) minutes at ambient temperature.

10.1.3.10 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program. Concurrently, introduce

the trapped materials to the gas chromatographic column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/minute for four minutes.

10.1.3.11 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 mL flushes of reagent water to avoid carryover of target compounds. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution between analyses, rinse it with distilled water, and then dry it in an oven at 105 °C.

10.1.3.12 After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. Trap temperatures up to 220 °C may be employed. However, the higher temperature will shorten the useful life of the trap. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

10.1.4 Low Level Soil/Sediment Samples

10.1.4.1 The Contractor must determine whether a soil/sediment sample should be analyzed by the low or medium method. It is the responsibility of the Contractor to analyze the sample at the correct level.

10.1.4.2 Three approaches may be taken to determine whether the low level or medium level method must be followed.

- Assume the sample is low level and analyze a 5 g sample.
- Use the X factor calculated from the hexadecane screen (Appendix A) to determine the appropriate method for analysis.
- Use *other EPA approved* screening procedures, *or an in-house laboratory screening procedure. The* procedure must be documented and available for review during on-site laboratory evaluation or when requested by the Technical Project Officer or Administrative Project Officer.

10.1.4.3 If the on column concentration of any *target* compound exceeds the initial calibration range from the analysis of 5 g sample, a smaller sample size must be analyzed. However, the smallest sample size permitted is 0.5 g. If smaller than 0.5 g sample size is needed to prevent the on column concentration of *target* compounds from exceeding the initial calibration range, the medium level method must be used.

10.1.4.4 The low level soil/sediment method is based on a heated purge of a

soil/sediment sample mixed with reagent water containing the system monitoring compounds and the internal standards. Analyze all matrix spike/matrix spike duplicate samples, blanks, and standards under the same condition as the samples.

10.1.4.5 *The procedure described below is to be utilized for volatile low level soil samples unless the Modified SW-846 Method 5035 is specified at the time of sample scheduling. If the Modified SW-846 Method 5035 is to be utilized for low level soil samples, use the procedure outlined in Appendix B and follow the equipment manufacturer's instructions. Note: The requirements of this SOW must be met at all times. If the above method is specified at the time of sample scheduling, but the Contractor believes that the samples cannot be processed by this method, the Contractor shall immediately contact SMO. SMO will contact the Region for instructions.*

10.1.4.6 Use 5 grams of sample, or use the X Factor (Appendix A) or your in-house screening procedure to determine the sample size for purging.

10.1.4.7 Prior to the analysis of samples, establish the appropriate purge and trap GC/MS operating conditions, as outlined in Section 9.1, analyze the instrument performance check solution (Section 9.2), and calibrate the GC/MS system according to Sections 9.3 through 9.4.6. This should be done prior to the preparation of the sample to avoid lose of volatiles from standards and sample. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low level method. Follow the initial and daily calibration instructions (Sections 9.3.3 and 9.4.3), but increase the purge temperature to 40 °C.

10.1.4.8 To prepare the reagent water containing the system monitoring compounds and the internal standards, remove the plunger from a 5 mL "Luerlok" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5 mL. Add 10 µL of the system monitoring compound spiking solution and 10 µL of the internal standard solution to the syringe through the valve. NOTE: Up to 10 mL of reagent water may be added to a soil sample to increase purge gas/sample interaction. **All soil samples including MS and MSD, standards, and blanks within an SDG must have the same amount of reagent water added. Do not increase/change the amount of system monitoring compound and internal standard solution added.**

10.1.4.9 The sample (for volatile organics) is defined as the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 5 g or the amount determined in using the screening procedure in Appendix A or an in-house screening procedure into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

10.1.4.10 Add the spiked reagent water to the purge device and connect the device to the purge and trap system.

10.1.4.11 NOTE: Prior to the attachment of the purge device, the steps in Sections *10.1.4.8* and *10.1.4.10* above must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

10.1.4.12 Heat the sample to 40 °C (± 1 °C) and purge the sample for 11.0 (± 0.1) minutes.

10.1.4.13 Proceed with the analysis as outlined in Sections 10.1.3.9 through 10.1.3.12.

10.1.5 Medium Level Soil/Sediment Samples

10.1.5.1 The medium level soil/sediment method is based on extracting the soil/sediment sample with methanol. An aliquot of the methanol extract is added to reagent water containing the system monitoring compounds and the internal standards. The reagent water containing the methanol extract is purged at ambient temperature. When using the screening method in Appendix A, all samples with an X Factor > 1.0 should be analyzed by the medium level method.

10.1.5.2 Prior to the analysis of samples, establish the appropriate purge and trap GC/MS operating conditions, as outlined in Section 9.1, analyze the instrument performance check solution (Section 9.2), and calibrate the GC/MS system according to Sections 9.3 through 9.4.6. This should be done prior to the addition of the methanol extract to reagent water. Because the methanol extract and reagent water mixture is purged at ambient temperature, the instrument performance check, initial calibration, and continuing calibration for water samples may be used for analyses of medium level soil/sediment sample extracts.

10.1.5.3 The sample (for volatile organics) is defined as the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 g (wet weight) into a tared 15 mL vial. Use a top loading balance. Record the actual weight to the nearest 0.1 g.

NOTE: If methanol preserved sample is to be analyzed, weigh sample vial and contents to the nearest 0.1 g and record the weight. Record any discrepancies between laboratory determined weight and sampler determined weight in the SDG Narrative and utilize the sampler determined weight in any calculations. Proceed to Section 10.1.5.6.

10.1.5.4 Quickly add 10 mL of methanol to the vial. Cap and shake for 2 minutes.

10.1.5.5 NOTE: The steps in Sections 10.1.5.3 and 10.1.5.4 must be performed rapidly to avoid loss of volatile organics. These steps

must be performed in a laboratory free of solvent fumes.

10.1.5.6 Let the solution settle. Then, using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be discarded. The 1 mL extract may be stored in the dark at *4 °C (±2 °C)* prior to the analysis.

10.1.5.7 Table 6 can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. If the hexadecane screen procedure (Appendix A) was followed, use the estimated concentration (Option A) or the X Factor (Option B) to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low level analysis or from the in-house screening procedure to determine the appropriate volume.

10.1.5.8 Remove the plunger from a 5 mL "Luerlok" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5 mL to allow volume for the addition of sample and standards. Add 10 µL of system monitoring compound and 10 µL of the internal standard solution. Also add the volume of methanol extract determined in Section 10.1.5.7 and a volume of clean methanol to total 100 µL (excluding methanol in system monitoring/internal standard solutions).

10.1.5.9 Attach the syringe-syringe valve assembly to the syringe valve on the purge device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

10.1.5.10 Proceed with the analysis as outlined in Section 10.1.3.9 through 10.1.3.12.

10.1.6 Sample Dilutions

10.1.6.1 If the on-column concentration of any *target* compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and purged. Guidance in performing dilutions and exceptions to this requirement are given in Sections *10.1.6.2* through *10.1.6.10*.

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

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

10.1.6.4 For medium level soil/sediment analyses, the purgeable organics screening procedure (Appendix A), if used, will show the approximate concentrations of major sample components. If a dilution of the sample was indicated, this dilution shall be made

just prior to GC/MS analysis of the sample. All steps in the dilution procedure must be performed without delays until the point at which the diluted sample is in a gas tight syringe.

10.1.6.5 *For water samples, all dilutions are made in volumetric flasks (10 mL to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions. Calculate the approximate volume of reagent water which will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.*

10.1.6.6 For water samples, inject the proper aliquot from the syringe prepared in Section 10.1.3.6 into the volumetric flask. *Only* aliquots of 1 mL increments are *permitted*. Dilute the *aliquot* to the mark *on the flask* with reagent water. Cap the flask, invert, and shake three times.

10.1.6.7 Fill a 5 mL syringe with the diluted sample as in Section 10.1.3.6.

10.1.6.8 If this is an intermediate dilution, use it and repeat the above procedure to achieve larger dilutions.

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

10.1.6.10 For total xylenes, where three isomers are quantified as two peaks, the calibration of each peak should be considered separately, i.e., a diluted analysis is not required for total xylenes unless the concentration of the peak representing the single isomer exceeds 200 µg/L (µg/kg for soils/sediment) or the peak representing the two co-eluting isomers on the GC column exceeds 400 µg/L (µg/kg for soils/sediment).

10.2 pH Determination (Water Samples)

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.

10.3 Percent Moisture Determination

Immediately after weighing the sample for analysis, weigh 5-10 g of the soil/sediment into a tared crucible. Determine the percent moisture by drying overnight at 105 °C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of soil/sediment.

EQ. 4

$$\%\text{Moisture} = \frac{\text{grams of wet sample - grams of dry sample}}{\text{grams of wet sample}} \times 100$$

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Compounds

11.1.1.1 The compounds listed in the Target Compound List (TCL) in Exhibit C (Volatiles) shall be identified by an analyst competent in the interpretation of mass spectra (see Exhibit A, Section 4.3.1) 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 component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

11.1.1.2 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run in the same 12-hour time period as the sample. If samples are analyzed during the same 12-hour time period as the initial calibration standards, use the RRT values from the 50 µg/L standard. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

11.1.1.3 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, **only** if the Contractor's GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRTs.

11.1.1.4 The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) **must** be present in the sample spectrum.

- The relative intensities of ions specified above must agree within ± 20.0 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50.0 percent in the standard spectra, the corresponding sample abundance must be between 30.0 and 70.0 percent).

- Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Exhibit A, Task II, the verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL, report the actual value followed by a "J", e.g., "3J".

11.1.1.5 If a compound cannot be verified by all of the criteria in Section 11.1.1.4, but in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantitation in Section 11.2.

11.1.2 Identification of Non-Target Compounds

11.1.2.1 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent mass spectral library, shall be used.

11.1.2.2 Up to 30 organic compounds of greatest apparent concentration not listed in Exhibit C for the volatile and semivolatile organic fraction, excluding the system monitoring compounds and internal standard compounds, shall be tentatively identified via a forward search of the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent mass spectral library. The following are not to be reported: 1) Substances with responses less than 10 percent of the internal standard (as determined by inspection of the peak areas or height), 2) Substances which elute earlier than 30 seconds before the first purgeable compound listed in Exhibit C (Volatiles) or three minutes after the last purgeable compound listed in Exhibit C (Volatiles) has eluted are not required to be searched in this fashion, 3) Carbon dioxide, and 4) Semivolatile TCL compounds listed in Exhibit C. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

11.1.2.3 NOTE: Computer generated library search routines must not use normalizations which would misrepresent the library or unknown spectra when compared to each other.

11.1.2.4 Guidelines for making tentative identification:

- Relative intensities of major ions in the reference spectrum (ions greater than 10.0 percent of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ±20.0 percent. Example: For an ion with an abundance of 50.0 percent of the standard spectra, the corresponding sample ion abundance must be between 30.0 and 70.0 percent.

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

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

- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the

sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

11.1.2.5 *If, after careful review and in the technical judgement of the mass spectral interpretation specialist, no valid identification can be made, the compound should be reported as follows:*

- *If the library search produces a match at or above 85%, report that compound.*
- *If the library search produces more than one compound at or above 85%, report the first compound (highest).*
- *If the library search produces no matches at or above 85%, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e. unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.*

11.2 Calculations

11.2.1 Target Compounds

11.2.1.1 Target compounds identified shall be quantified by the internal standard method using the equations below. The internal standard used shall be that which is assigned in Table 3. The relative response factor (RRF) from the continuing calibration standard is used to calculate the concentration in the sample.

11.2.1.2 Water

EQ. 5

$$\text{Concentration } (\mu g/L) = \frac{(A_x)\ (I_s)\ (Df)}{(A_{is})\ (RRF)\ (V_o)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (see Table 2)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (see Tables 3 and 4)

I_s = Amount of internal standard added in nanograms (ng)

RRF = Relative response factor from the ambient temperature purge of the calibration standard.

V_o = 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 2.0 mL of sample is diluted to 5 mL with reagent water and purged, Df = 5 mL/2.0 mL = 2.5.
If no dilution is performed, Df = 1.

11.2.1.3 Low *Level* Soil/Sediment

EQ. 6

$$\text{Concentration } (\mu g/Kg) \text{ (dry weight basis)} = \frac{(A_x)\ (I_s)}{(A_{is})\ (RRF)\ (W_s)\ (D)}$$

Where,

A_x, I_s, A_{is} are as given for water, Equation 5.
RRF = Relative response factor from the heated purge of the calibration standard.

$$D = \frac{100 - \%\text{moisture}}{100}$$

W_s = Weight of sample added to the purge tube, in grams (g).

11.2.1.4 Medium *Level* Soil/Sediment

EQ. 7

$$\text{Concentration } \mu g/Kg \text{ (dry weight basis)} = \frac{(A_x)\ (I_s)\ (V_t)\ (1000)\ (Df)}{(A_{is})\ (RRF)\ (V_a)\ (W_s)\ (D)}$$

Where,

A_x, I_s, A_{is} are as given for water, Equation 5.
RRF = Relative response factor from the **ambient** temperature purge of the calibration standard.
V_t = Total volume of the methanol extract in milliliters (mL).
NOTE: This volume is typically 10 mL, even though only 1 mL is transferred to the vial in Section 10.1.5.6.
V_a = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 µL) in microliters (µL) added to reagent water for purging.
W_s = Weight of soil/sediment extracted, in grams (g).

$$D = \frac{100 - \%\text{moisture}}{100}$$

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

$$\frac{\mu L \text{ most conc. extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

11.2.1.5 For water, low level and medium level soil/sediment samples, xylenes (o-,m-, and p-isomers) are to be reported as xylenes (total).

Because the o- and p-xylene isomers co-elute on packed columns, and the m- and p-xylene isomers co-elute on capillary columns, special attention must be given to the quantitation of the xylenes. The relative response factor (RRF) determined in Section 9.4.4 is based on the peak that represents the single isomer on the GC column used (m-xylene on packed columns, o-xylene on capillary columns). In quantitating sample concentrations, use the areas on both peaks and the RRF from Section 9.4.4. 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. It is required that all three xylene isomers be present in the initial and continuing calibration standards.

11.2.1.6 The cis and trans stereo isomers of 1,2-dichloroethene are to be reported *separately*.

11.2.1.7 Secondary ion quantitation is allowed **only** when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. A secondary ion cannot be used unless a relative response factor is calculated using the secondary ion.

11.2.1.8 The requirements listed in Sections 11.2.1.9 and 11.2.1.10 apply to all standards, samples including MS/MSDs, and blanks.

11.2.1.9 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitations. This normally occurs when there is compound co-elution, baseline noise, or matrix interferences. In these circumstances the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific TCL compound. The area integrated shall not include baseline background noise. The area integrated shall not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instances of manual integration must be documented in the SDG Narrative.

11.2.1.10 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standards, and system monitoring compounds.

11.2.2 Non-Target Compounds

11.2.2.1 An estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method. For

quantitation, the nearest internal standard free of interferences shall be used.

11.2.2.2 The formulas for calculating concentrations are the same as in Sections 11.2.1.2, 11.2.1.3, and 11.2.1.4. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A relative response factor (RRF) of one (1) is to be assumed. The resulting concentration shall be qualified as "J" (estimated, due to lack of a compound-specific response factor), and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration must be calculated for all tentatively identified compounds as well as those identified as unknowns.

11.2.3 CRQL Calculations

NOTE: If the adjusted CRQL is less than the CRQL listed in Exhibit C (Volatiles), report the CRQL listed in Exhibit C (Volatiles).

11.2.3.1 Water

EQ. 8

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{V_x}{V_o} \times Df$$

Where,

V_o and Df are as given in Equation 5
V_x = Contract Sample Volume (5 mL)

11.2.3.2 Low Level Soil/Sediment

EQ. 9

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)}{(W_s)(D)}$$

Where,

W_s and D are as given in Equation 6
W_x = Contract Sample Weight (5 g)

11.2.3.3 Medium Level Soil/Sediment

EQ. 10

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)(V_t)(V_y)(1000)(Df)}{(W_s)(V_c)(V_a)(D)}$$

Where,

V_t, Df, W_s, V_a and D are as given in Equation 7
W_x = Contract Sample Weight (4 g)
V_y = Contract Soil Aliquot Volume from soil methanol extract (100 µL)
V_c = Contract Soil Methanol Extract Volume (10,000 µL)

11.2.4 System Monitoring Compound Recoveries

11.2.4.1 Calculate the recovery of each system monitoring compound in all samples, blanks, matrix spikes, and matrix spike duplicates. Determine if the recovery is within limits (see Table 7), and report on the appropriate form.

11.2.4.2 Calculate the concentrations of the system monitoring compounds using the same equations as used for target compounds.

11.2.4.3 Calculate the recovery of each system monitoring compound as follows:

EQ. 11

$$\%\text{Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

11.2.5 Internal Standard Responses and Retention Times

Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. Compare the sample internal standard responses and retention times to the continuing calibration internal standard response and retention times.

For samples analyzed during the same 12-hour time period as the initial calibration standards, compare the internal standard responses and retention times against the 50 µg/L calibration standard. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate.

11.3 Technical Acceptance Criteria for Sample Analysis

11.3.1 The samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration, and blank technical acceptance criteria.

11.3.2 The sample must be analyzed within the contract holding time.

11.3.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

11.3.4 The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows in Table 7.

11.3.5 The EICP area for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis.

11.3.6 The retention time shift for each of the internal standards must be within ± 0.50 minutes (30 seconds) between the sample and the most recent continuing calibration standard analysis.

11.3.7 The relative retention time (RRT) of the system monitoring compound in a sample must be within ±0.06 (RRT) units of its relative retention time in the continuing calibration standard.

11.3.8 Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the initial calibration range unless a more diluted aliquot of the sample is also analyzed according to the procedures in Section 10.1.6.

11.3.9 The Contractor must demonstrate that there is no carryover from a contaminated sample before data from subsequent analyses may be submitted. After a sample that contains a target compound at a level exceeding the initial calibration range, the Contractor must either:

- 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 (see Section 12.1.4), or

- Monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample and that exceeded the calibration range. The maximum contamination criteria are as follows: the sample must not

contain a concentration above the CRQL for the target compounds that exceeded the limits in the contaminated sample. If an auto sampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample must also meet the maximum contamination criteria. If the maximum criteria were exceeded, then all samples affected by the carryover must be reanalyzed at no additional cost to the Agency.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or sample results which failed to meet the sample technical acceptance criteria require reanalysis at no additional cost to the Agency.

11.4.2 Corrective actions for failure to meet instrument performance checks, initial and continuing calibration, and method blanks must be completed before the analysis of samples.

11.4.3 Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria.

11.4.3.1 If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria:

- Check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria.

- If the instrument logs indicate that the incorrect amount of system monitoring compound or internal standard compound spiking solution was added, then reanalyze the sample after adding the correct amount of system monitoring compound and internal standard spiking solutions.

- If the system monitoring compound spiking solution or internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare the solutions and reanalyze the samples.

- If the instrument malfunctioned, correct the instrument problem and reanalyze the sample. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample. Verify that the system monitoring compound recoveries meet acceptance criteria.

11.4.3.2 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was a matrix effect, take the following corrective action steps:

- Reanalyze the sample. EXCEPTION: If system monitoring compound recoveries or internal standard compound response in a sample

used for a matrix spike or matrix spike duplicate were outside the acceptance criteria, then it should be reanalyzed only if system monitoring compound recoveries and internal standard compound response met acceptance criteria in both the matrix spike and matrix spike duplicate analyses.

- If the system monitoring compound recoveries and the internal standard compound responses meet the acceptance criteria in the reanalyzed sample, then the problem was within the Contractor's control. Therefore, submit data only from the reanalysis.

- If the system monitoring compound recoveries and/or the internal standard compound responses fail to meet the acceptance windows in the reanalysis, then submit data from both analyses. Distinguish between the initial analysis and the reanalysis on all deliverables using the suffixes on Exhibit B.

11.4.4 Corrective action for system monitoring compounds relative retention times/internal standard compounds retention times outside acceptance criteria.

11.4.4.1 If the system monitoring compounds relative retention times or internal standard compounds retention times are not within their acceptance criteria, check the instrument for malfunctions. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the samples.

11.4.4.2 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was a matrix effect, take the following corrective action steps:

- Reanalyze the sample. EXCEPTION: If the system monitoring compounds relative retention times or internal standard compounds retention times in a sample used for a matrix spike or matrix spike duplicate were outside the acceptance criteria, then it should be reanalyzed only if the system monitoring compounds and internal standard compounds retention times were within the acceptance criteria in both the matrix spike and matrix spike duplicate analyses.

- If the system monitoring compounds relative retention times and internal standard compounds retention times are within the acceptance criteria, then the problem was within the Contractor's control. Therefore, submit only data from the reanalysis when the system monitoring compounds relative retention times and the internal standard compounds retention times are within the acceptance limits.

- If the system monitoring compounds relative retention times or the internal standard compounds retention times are outside the acceptance criteria in the reanalysis, then submit data from both analyses. Distinguish between the initial analysis and the reanalysis on all deliverables, using the suffixes in Exhibit B.

12.0 QUALITY CONTROL

12.1 Blank Analyses

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

12.1.1.1 METHOD BLANK - a volume of a clean reference matrix (reagent water for water samples or a purified solid matrix for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.1.2 STORAGE BLANK - upon receipt of the first samples in an SDG, two 40.0 mL screw-cap volatile vials with a PTFE-faced silicone septum are filled with reagent water (80 mL total). The vials are stored with the samples in the SDG under the same conditions. After all samples in the SDG have been analyzed, the storage blank is analyzed. The storage blank indicates whether contamination may have occurred during storage of samples.

12.1.1.3 INSTRUMENT BLANK - a 5.0 mL aliquot of reagent water that is carried through the entire analytical procedure. Instrument blanks are analyzed after a sample/dilution which contains a target compound exceeding the initial calibration range. The results from the instrument blank analysis indicate whether there is contamination from a previous sample.

12.1.2 Frequency of Blank Analyses

12.1.2.1 The method blank **must** be analyzed at least once during every 12-hour time period on each GC/MS system used for volatile analysis (see Section 9.2.2 for the definition of the 12-hour time period).

12.1.2.2 The method blank **must** be analyzed after the continuing calibration and before any samples, including matrix spike/matrix spike duplicates, dilutions, or storage blanks are analyzed. The method blank must be analyzed after the initial calibration sequence if samples are analyzed before the 12-hour period expires. A method blank must be analyzed in each 12-hour time period in which samples, including dilutions, matrix spikes/matrix spike duplicates, and storage blanks are analyzed.

12.1.2.3 A minimum of one storage blank must be analyzed per SDG after all samples for that SDG have been analyzed.

12.1.2.4 The Contractor must demonstrate that there is no carryover from contaminated samples before data from subsequent analyses may be used. Samples/dilutions may contain target compounds at levels exceeding the initial calibration range. An instrument blank must be analyzed after the sample that exceeds the calibration range (also in the same purge inlet if an autosampler is used) or a sample

that meets the maximum contamination criteria in Section 11.3.8 must be analyzed. For these purposes, if the instrument blank meets the technical acceptance criteria for blank analyses 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., contaminated), the system must be decontaminated. Until an instrument blank meets the blank technical acceptance criteria or a sample meets the maximum contamination criteria in Section 11.3.8, any samples analyzed since the original contaminated sample will require reanalysis at no additional cost to the Agency. NOTE: Only the instrument blank which demonstrates that there was no carryover from the previous sample or the instrument blank that demonstrates that the system is clean (Section 12.1.4) needs to be reported. Instrument blanks analyzed during the instrument decontamination process which exceed the requirements listed in Section 12.1.4 do not need to be reported.

12.1.3 Procedure for Blank Analyses

12.1.3.1 For water samples, a volatile method blank consists of a 5 mL volume of reagent water (Section 7.1.1) spiked with 10 µL of the system monitoring compound spiking solution (Section 7.2.4.1) and 10 µL of the internal standard spiking solution (Section 7.2.4.3) and carried through the analytical procedure.

12.1.3.2 For low level soil/sediment samples, a volatile method blank consists of 5 g of a purified solid matrix added to 5 mL of reagent water that has been spiked with 10 µL each of the system monitoring compound spiking solution and the internal standard spiking solution. This method blank is then carried through the analytical procedure.

12.1.3.3 For medium level soil/sediment samples, a volatile method blank consists of 4 g of a purified solid matrix added to 10 mL of methanol and extracted for two minutes. A 100 µL aliquot of the methanol is added to reagent water and spiked with 10 µL of the internal standard spiking solution and 10 µL of the system monitoring compound spiking solution and taken through the analytical procedure.

12.1.3.4 Storage/instrument blanks consist of a 5 mL volume of reagent water (Section 7.1.1) spiked with 10 µL of the system monitoring compound spiking solution (Section 7.2.4.1) and 10 µL of the internal standard spiking solution (Section 7.2.4.3) and carried through the analytical procedure.

12.1.3.5 A storage blank shall be analyzed and reported as a water sample unless the SDG contains only soil samples. If an SDG contains only soil samples, the storage blank may be analyzed and reported as a soil sample.

12.1.3.6 Identify and quantitate analytes according to Section 11.0.

12.1.4 Technical Acceptance Criteria for Blank Analyses

12.1.4.1 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 12.1.2.

12.1.4.2 A storage blank shall be analyzed and reported as a water sample unless the SDG contains only soil samples. If an SDG contains only soil samples, the storage blank may be analyzed and reported as a soil sample.

12.1.4.3 The percent recovery of each of the system monitoring compounds in a blank must be within the acceptance windows in Table 7.

12.1.4.4 The EICP area for each of the internal standards in a blank must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis.

12.1.4.5 The retention time shift for each of the internal standards in a blank must be within ±0.50 minutes (30 seconds) of its retention time in the most recent continuing calibration standard analysis.

12.1.4.6 The concentration of each target compound found in the blank must be less than its CRQL listed in Exhibit C (Volatiles), except for methylene chloride *and cyclohexane* which must be less than 2.5 times *their respective* CRQL*s*, and acetone and 2-butanone, which must be less than 5 times *their respective* CRQL*s*.

12.1.5 Corrective Action for Blank Analyses

12.1.5.1 It is the Contractor's responsibility to ensure that method interferences caused by the contaminants in solvent, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms, be eliminated. If a Contractor's blanks exceed the criteria in Section 12.1.4.6, 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 analysis proceeds.

12.1.5.2 Any method blank or instrument blank that fails to meet the technical acceptance criteria for blank analyses must be reanalyzed at no additional cost to the Agency. Furthermore, all samples, including MS/MSD samples, processed within the 12-hour period with a method blank or instrument blank that does not meet the technical acceptance criteria for blanks will require reanalysis at no additional cost to the Agency.

12.1.5.3 If the storage blank does not meet the technical acceptance criteria for blank analyses in Sections 12.1.4.1 through 12.1.4.5, correct system problems and reanalyze the storage blank. If the storage blank does not meet the criteria in Section 12.1.4.6, reanalyze the storage blank to determine whether the contamination occurred during storage or during the analysis. If, upon reanalysis, the storage blank meets the criteria in Section 12.1.4.6, the problem occurred

during the analysis and the reanalyzed storage blank results must be reported. If upon reanalysis, the storage blank did not meet the criteria in Section 12.1.4.6, the problem occurred during storage. The laboratory manager or his/her designee must address the problem in the SDG Narrative and discuss the corrective actions implemented to prevent future occurrences. NOTE: A copy of the storage blank data must also be retained by the Contractor and be made available for inspection during on-site laboratory evaluations.

12.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

12.2.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the methods used for volatile analyses, the Agency has prescribed a mixture of volatile target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

12.2.2 Frequency of MS/MSD

12.2.2.1 A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix for the following, whichever is most frequent:

- Each SDG, or
- Each matrix within an SDG, or
- Each group of samples of a similar concentration level (soils only).

MS/MSD samples shall be analyzed unless otherwise specified on the Traffic Report (TR). If no MS/MSD samples are specified on the TR, the Contractor shall contact SMO to confirm that MS/MSD analyses are not required.

12.2.2.2 As a part of the Agency's QA/QC program, water rinsate samples and/or field/trip blanks (field QC) may accompany soil/sediment samples and/or water samples that are delivered to a laboratory for analysis. The Contractor shall not perform MS/MSD analysis on any of the field QC samples.

12.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample, less than the required amount, remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify the Region (through SMO) that insufficient sample was received and identify the EPA sample selected for the MS/MSD analysis. The rationale for the choice of a sample other than the one designated by the Region shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample remaining in any of the samples in an SDG to perform an MS/MSD, then the Contractor shall immediately

contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no MS/MSD is required, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the resolution. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the Region has requested MS/MSD analysis at a greater frequency than required by the contract, the Contractor shall contact SMO. SMO will contact the Region to determine which samples should have an MS/MSD performed on them. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative. *If this procedure is not followed, the Contractor will not be paid for MS/MSD analysis performed at a greater frequency than required by the contract.*

12.2.2.6 When a Contractor receives **only** a Performance Evaluation (PE) sample(s), no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the MS/MSD when the Region did not designate samples to be used for this purpose. If the PE sample is received as an ampulated standard extract, the ampulated PE sample is not considered to be another matrix type.

12.2.3 Procedure for Preparing MS/MSD

12.2.3.1 Water

To prepare a matrix spike and matrix spike duplicate for water samples, add 10 µL of the matrix spike solution (Section 7.2.4.2) to each of the 5 mL aliquots of the sample chosen for spiking. Process samples according to Sections 10.1.3.7 through 10.1.3.12. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike compound.

12.2.3.2 Soil - Low Level

To prepare a matrix spike and matrix spike duplicate for low level soil/sediment samples, add 10 µL of the matrix spike solution to the 5 mL of spiked reagent water added to each of the two aliquots of the soil/sediment from the sample chosen for spiking. Process samples according to Sections *10.1.4.8* through *10.1.4.13*. The concentration for a 5 g sample should be equivalent to 50 µg/kg of each matrix spike compound.

12.2.3.3 Soil - Medium Level

12.2.3.3.1 To prepare a matrix spike and matrix spike duplicate for medium level soil/sediment samples, add 9 mL of methanol and 1 mL of matrix spike solution to each of the two aliquots of the soil/sediment sample chosen for spiking. Process samples according to Sections 10.1.5.6 through 10.1.5.10. This results in a 6,200 µg/kg concentration of each matrix spike compound when added to a 4 g sample. Add a 100 µL aliquot of this

extract to 5 mL of water for purging (as per Sections 10.1.5.8 through 10.1.5.9).

12.2.3.3.2 NOTE: Before performing an MS/MSD analysis, analyze the sample used for MS/MSD. If the sample analysis required dilution, the aliquots for the MS/MSD *shall* be prepared at the same dilution as the least diluted analysis for which the sample results will be reported to the Agency. Sample dilutions must be performed in accordance with Section 10.1.6. Do **not** further dilute MS/MSD samples to get **either** spiked **or** non-spiked analytes within calibration range.

12.2.4 Calculations for MS/MSD

12.2.4.1 Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds (Equations 5, 6, and 7). Calculate the recovery of each matrix spike compound as follows:

EQ. 12

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiked sample result
SR = Sample result
SA = Spike added

12.2.4.2 Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

EQ. 13

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where,

MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

12.2.5 Technical Acceptance Criteria for MS/MSD

12.2.5.1 All MS/MSDs must be analyzed on a GC/MS system meeting the BFB, initial calibration and continuing calibration technical acceptance

criteria, the blank technical acceptance criteria, and at the frequency described in Section 12.2.2.

12.2.5.2 The MS/MSD must be analyzed within the contract holding time.

12.2.5.3 The retention time shift for each of the internal standards in the MS/MSD must be within ±0.50 minutes (30 seconds) of its retention time and the most recent continuing calibration standard analysis.

12.2.5.4 The limits for matrix spike compound recovery and RPD are given in Table 8. As these limits are only advisory, no further action by the laboratory is required. However, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questions from the Agency.

12.2.6 Corrective Action for MS/MSD

Any MS/MSD that does not meet the technical acceptance criteria for MS/MSD must be reanalyzed at no additional cost to the Agency.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington D.C., 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult "The Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.2.

16.0 REFERENCES

Not applicable.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Table 1

BFB Key Ions and Ion 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 120.0 percent that of m/z 95.

Table 2

Characteristic Ions for Volatile Target Compounds

Analyte	Primary Quantitation Ion	Secondary Ion(s)
Dichlorodifluoromethane	*85*	*87*
Chloromethane	50	52
Vinyl chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	*101*	*103*
1,1-Dichloroethene	96	61, 98
1,1,2-Trichloro-1,2,2-trifluoroethane	*101*	*85, 151*
Acetone	43	58
Carbon disulfide	76	78
Methyl Acetate	*43*	*74*
Methylene chloride	84	49, 51, 86
trans-1,2-Dichloroethene	*96*	*61, 98*
tert-Butyl Methyl Ether	*73*	*43, 57*
1,1-Dichloroethane	63	65, 83, 85, 98, 100
cis-1,2-Dichloroethene	*96*	*61, 98*
2-Butanone	43*	57
Chloroform	83	85
1,1,1-Trichloroethane	97	99, 117, 119
Cyclohexane	*56*	*69, 84*
Carbon Tetrachloride	117	119, 121
Benzene	78	-
1,2-Dichloroethane	62	64, 100, 98
Trichloroethene	130	95, 97, 132
Methylcyclohexane	*83*	*55, 98*
1,2-Dichloropropane	63	65, 114
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58, 100
Toluene	91	92
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Tetrachloroethene	164	129, 131, 166
2-Hexanone	43	58, 57, 100
Dibromochloromethane	129	208, 206
1,2-Dibromoethane	*107*	*109*
Chlorobenzene	112	114
Ethylbenzene	106	91
Xylene (total)	106	91
Styrene	104	78, 103
Bromoform	173	171, 175, 250, 252, 254, 256

*m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

Table 2 (Con't)

Characteristic Ions for Volatile Target Compounds

Analyte	Primary Quantitation Ion	Secondary Ion(s)
Isopropylbenzene	*105*	*120, 77*
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,3-Dichlorobenzene	*146*	*111, 75*
1,4-Dichlorobenzene	*146*	*111, 75*
1,2-Dichlorobenzene	*146*	*111, 75*
1,2-Dibromo-3-Chloropropane	*75*	*157, 155*
1,2,4-Trichlorobenzene	*180*	*182, 145*

Table 3

Volatile Internal Standards with Corresponding Target Compounds and System Monitoring Compounds Assigned for Quantitation

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d5
Dichlorodifluoromethane	1,1,1-Trichloroethane	4-Methyl-2-pentanone
Chloromethane	*Cyclohexane*	Toluene
Vinyl Chloride	Carbon Tetrachloride	Tetrachloroethene
Bromomethane	Benzene	2-Hexanone
Chloroethane	Trichloroethene	*1,2-Dibromoethane*
Trichlorofluoromethane	*Methylcyclohexane*	Chlorobenzene
1,1-Dichloroethene	1,2-Dichloropropane	Ethylbenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Bromodichloromethane	Xylene (total)
Acetone	cis-1,3-Dichloropropene	Styrene
Carbon Disulfide	trans-1,3-Dichloropropene	*Isopropylbenzene*
Methyl Acetate	1,1,2-Trichloroethane	1,1,2,2-Tetrachloroethane
Methylene Chloride	Dibromochloromethane	*1,3-Dichlorobenzene*
trans-1,2-Dichloroethene	Bromoform	*1,4-Dichlorobenzene*
tert-Butyl Methyl Ether		*1,2-Dichlorobenzene*
1,1-Dichloroethane		*1,2-Dibromo-3-chloropropane*
cis-1,2-Dichloroethene		*1,2,4-Trichlorobenzene*
2-Butanone		Toluene-d8 (SMC)
Chloroform		4-Bromofluorobenzene (SMC)
1,2-Dichloroethane		
1,2-Dichloroethane-d4 (SMC)		

(SMC) = system monitoring compound

Table 4

Characteristic Ions for System Monitoring Compounds and Internal Standards for Volatile Organic Compounds with CAS Numbers

Compound	Primary Quantitation Ion	Secondary Ion(s)	CAS Number
	SYSTEM MONITORING COMPOUNDS		
4-Bromofluorobenzene	95	174, 176	460-00-4
1,2-Dichloroethane-d4	65	102	17060-07-0
Toluene-d8	98	70, 100	2037-26-5
	INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51	74-97-5
1,4-Difluorobenzene	114	63, 88	540-36-3
Chlorobenzene-d5	117	82, 119	3114-55-4

Table 5

Relative Response Factor Criteria for Initial and Continuing Calibration of Volatile Organic Compounds

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Dichlorodifluoromethane	*0.010*	*none*	*none*
Chloromethane	0.010	none	none
Vinyl chloride	0.100	20.5	±25.0
Bromomethane	0.100	20.5	±25.0
Chloroethane	0.010	none	none
Trichlorofluoromethane	*0.010*	*none*	*none*
1,1-Dichloroethene	0.100	20.5	±25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	*0.010*	*none*	*none*
Acetone	0.010	none	none
Carbon disulfide	0.010	none	none
Methyl Acetate	*0.010*	*none*	*none*
Methylene chloride	0.010	none	none
trans-1,2-Dichloroethene	*0.010*	*none*	*none*
Methyl tert-Butyl Ether	*0.010*	*none*	*none*
1,1-Dichloroethane	0.200	20.5	±25.0
cis-1,2-Dichloroethene	*0.010*	*none*	*none*
2-Butanone	0.010	none	none
Chloroform	0.200	20.5	±25.0
1,1,1-Trichloroethane	0.100	20.5	±25.0
Cyclohexane	*0.010*	*none*	*none*
Carbon tetrachloride	0.100	20.5	±25.0
Benzene	0.500	20.5	±25.0
1,2-Dichloroethane	0.100	20.5	±25.0
Trichloroethene	0.300	20.5	±25.0
Methylcyclohexane	*0.010*	*none*	*none*
1,2-Dichloropropane	0.010	none	none
Bromodichloromethane	0.200	20.5	±25.0
cis-1,3-Dichloropropene	0.200	20.5	±25.0
4-Methyl-2-pentanone	0.010	none	none
Toluene	0.400	20.5	±25.0
trans-1,3-Dichloropropene	0.100	20.5	±25.0
1,1,2-Trichloroethane	0.100	20.5	±25.0
Tetrachloroethene	0.200	20.5	±25.0
2-Hexanone	0.010	none	none
Dibromochloromethane	0.100	20.5	±25.0
1,2-Dibromoethane	*0.010*	*none*	*none*
Chlorobenzene	0.500	20.5	±25.0
Ethylbenzene	0.100	20.5	±25.0
Xylene (total)	0.300	20.5	±25.0
Styrene	0.300	20.5	±25.0
Bromoform	0.100	20.5	±25.0

Table 5 (Con't)

Relative Response Factor Criteria for Initial and Continuing Calibration of Volatile Organic Compounds

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Isopropylbenzene	*0.010*	*none*	*none*
1,1,2,2-Tetrachloroethane	0.300	20.5	±25.0
1,3-Dichlorobenzene	*0.600*	*20.5*	*±25.0*
1,4-Dichlorobenzene	*0.500*	*20.5*	*±25.0*
1,2-Dichlorobenzene	*0.400*	*20.5*	*±25.0*
1,2-Dibromo-3-chloropropane	*0.010*	*none*	*none*
1,2,4-Trichlorobenzene	*0.200*	*none*	*±25.0*
SYSTEM MONITORING COMPOUNDS			
Bromofluorobenzene	0.200	20.5	±25.0
Toluene-d8	0.010	none	none
1,2-Dichloroethane-d4	0.010	none	none

Table 6

The "X" Factor Table

X Factor	Estimated Concentration Range[1] (µg/kg)	Take This Volume of Methanol Extract[2] (µL)
0.25 - 5.0	500 - 10,000	100
0.5 - 10.0	1000 - 20,000	50
2.5 - 50.0	5000 - 100,000	10
12.5 - 250	25,000 - 500,000	100 of 1/50 dilution[3]

Calculate appropriate dilution factor for concentrations exceeding those in the table.

[1] Actual concentration ranges could be 10 to 20 times higher than this if the compounds are halogenated and the estimates are from GC/FID.

[2] The volume of methanol added to the 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 µL added to the syringe.

[3] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

Table 7

System Monitoring Compound Recovery Limits

Compound	% Recovery Water	% Recovery Soil
Toluene-d8	88-110	84-138
Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d4	76-114	70-121

Table 8

Matrix Spike Recovery and
Relative Percent Difference Limits

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

APPENDIX A - SCREENING OF HEXADECANE EXTRACTS FOR VOLATILES

1.0 SCOPE AND APPLICATION

1.1 The hexadecane extraction and screening methods for purgeables described in this section are designed to aid the analyst in deciding whether a soil sample is low or medium level in order to prevent saturation of the purge and trap system and/or the GC/MS system. These or other screening methods should be used, particularly if there is some doubt about the level of organics in a sample. This is especially true in soil/sediment analysis. Water samples may also be screened to determine an appropriate dilution factor for analysis.

1.2 These extractions and preparation procedures were developed for rapid screening of water and soil/sediment samples from hazardous waste sites. The design of the methods thus does not stress efficient recoveries or low limits of quantitation. Rather, the procedures were designed to screen at moderate recovery and sufficient sensitivity for a broad spectrum of organic chemicals. The results of the analyses thus may reflect only a minimum of the amount actually present in some samples. This is especially true if water soluble solvents are present.

2.0 SUMMARY OF METHODS

2.1 Sample Preparation

2.1.1 Water

A 40 mL aliquot of sample is extracted with 2 mL of hexadecane. This provides a minimum quantitation limit (MQL) as follows:

Compounds	MQL (µg/L)
non-halogenated aromatics	40-50
halogenated methanes	80-1000
halogenated ethanes	400-500

2.1.2 Soil/Sediment

40 mL of reagent water are added to 10 g (wet weight) of soil/sediment and shaken. The water phase is in turn extracted with 2 mL of hexadecane. This provides a minimum quantitation limit of approximately four times higher than those listed for water.

2.2 GC/FID Screening

The hexadecane extracts of water and soil/sediment are screened on a gas chromatograph/flame ionization detector (GC/FID). The results of the screen will determine if volatile organics are to be analyzed by low or medium level GC/MS procedures if the sample is a soil/sediment, or to determine the appropriate dilution factor if the sample is water. Note: The flame ionization detector varies considerably in sensitivity when comparing aromatics and halogenated methanes and ethanes. Halomethanes are approximately 20x less sensitive than aromatics and haloethanes are

approximately 10x less sensitive than aromatics. Low molecular weight, water soluble solvents (e.g., alcohols and ketones) will not be extracted from the water, and therefore will not be detected by the GC/FID.

3.0 INTERFERENCES

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent 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 depending upon the nature and diversity of the site being sampled.

4.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses. Use all reagents in fume hoods whenever possible. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

5.0 EQUIPMENT AND SUPPLIES

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

5.1 Glassware

5.1.1 Syringes - 0.5 mL

5.1.2 Vials and Caps - 2 mL capacity for GC autosampler

5.1.3 Pasteur Pipets - disposable

5.1.4 Centrifuge Tube - 50 mL with ground glass stopper or Teflon-lined screw cap.

5.1.5 Volumetric Flask - 50 mL with ground glass stopper.

5.2 Balance - analytical, capable of accurately weighing ±0.0001 g.

5.3 Pyrex Glass Wool

5.4 Balances - analytical, capable of accurately weighing ±0.0001 g, and a top-loading balance capable of weighing 100 g ±0.01 g. The balances must be calibrated with class S weights or known reference weights once per each 12-hour work shift. The balances must be calibrated with class S weights at a minimum of once per month. The balances must also be annually checked by a certified technician.

5.5 Centrifuge

5.6 Gas Chromatograph/Mass Spectrometer (GC/MS) System

5.6.1 Gas Chromatograph - an analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.2 Gas Chromatography Column - 30 m (or longer) x 2 mm ID glass column packed with 10% OV-101 on 100-120 mesh chromosorb W-HP (or equivalent). The column temperature should be programmed from 80 °C to 280 °C at 16 C°/min and held at 280 °C for 10 minutes.

5.6.3 Flame Ionization Detector

6.0 REAGENTS AND STANDARDS

6.1 Reagents

6.1.1 Reagent Water - defined as water in which an interferant is not observed at the CRQL of each analyte of interest.

6.1.2 Hexadecane and Methanol - pesticide residue analysis grade or equivalent.

6.2 Standards

6.2.1 Introduction

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.

6.2.2 Stock Standard Solutions

6.2.2.1 Stock standard solutions (1 µg/µL) can be prepared from pure standard materials or purchased as certified solutions.

6.2.2.2 Prepare stock standard solutions by accurately weighing about 0.01 g of pure material. Dissolve the material in methanol and dilute to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 97% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

6.2.3 Working Standard Solutions

6.2.3.1 Standard Mixture #1

Prepare a working standard mixture containing benzene, toluene, ethylbenzene, and xylene at 100 ng/µL of each compound in methanol.

6.2.3.2 Standard Mixture #2

Prepare a working standard mixture containing n-nonane. And n-dodecane at 100 ng/µL of each compound in methanol.

6.2.4 Storage of Standards

Transfer all standard solutions into multiple Teflon-sealed screw-cap vials. Store, with no head-apace, at -10 °C to -20 °C, and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. These solutions must be replaced after six months, or sooner, if comparison with quality control check samples indicates a problem. Standards prepared from gasses or reactive compounds such as styrene must be replaced after two months, or sooner, if comparison with quality control check samples indicates a problem.

7.0 QUALITY CONTROL

7.1 Method Blank

7.1.1 Summary

A method blank is a volume of clean reagent water taken through the extraction and screening procedure. The volume of reagent water used must be approximately equal to the volume of associated samples. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

7.1.2 Frequency

One method blank must be extracted and analyzed on each GC/FID system used to screen samples for the following, whichever is most frequent.

- Each SDG, or

- Each 20 samples in *an* SDG, excluding matrix spikes/matrix spike duplicates, or

- When samples are extracted.

7.1.3 Procedure

For screening of volatile organics, a method blank consists of a 40 mL volume of reagent water extracted with 2 mL of hexadecane. The hexadecane extract is then screened on a GC/FID system.

8.0 CALIBRATION AND STANDARDIZATION

8.1 GC/FID Operating Conditions

Refer to Section 5.5.2 for recommended column temperature program.

8.2 GC Calibration

8.2.1 Summary

Prior to sample analysis each GC/FID system must be standardized for half scale response.

8.2.2 Frequency

Each GC/FID system must be calibrated at the beginning of each 12-hour shift.

8.2.3 Procedure

8.2.3.1 Add 200 µL of each of working standard mixtures #1 and #2 (Section 6.2.3) to separate 40 mL portions of reagent water in 50 mL volumetric flasks. Immediately add 2 mL of hexadecane, cap the flask, and shake vigorously for 1 minute. Let phases separate. Open the flask and add sufficient reagent water to bring the hexadecane layer into the neck of the flask. Transfer approximately 1 mL of the hexadecane layer to a 2 mL GC vial.

8.2.3.2 Inject 1-2 µL of the extracts that contain approximately 10 ng/µL each of standard mixture #1 and standard mixture #2 compounds.

9.0 PROCEDURE

9.1 Sample Preparation

9.1.1 Water

9.1.1.1 Allow the contents of the 40 mL sample vial to come to room temperature. Quickly transfer the contents of the 40 mL sample vial to a 50 mL volumetric flask. Immediately add 2 mL of hexadecane, cap the flask, and shake vigorously for 1 minute. Let phases separate. Open the flask and add sufficient reagent water to bring the hexadecane layer into the neck of the flask.

9.1.1.2 Transfer approximately 1 mL of the hexadecane layer to a 2 mL GC vial. If an emulsion is present after shaking the sample, break it by doing the following:

- Pulling the emulsion through a small plug of Pyrex glass wool packed in a pipet, or
- Transferring the emulsion to a centrifuge tube and centrifuging for several minutes.

9.1.2 Soil/Sediment

Add approximately 10 g of soil/sediment (wet weight) to 40 mL of reagent water in a 50 mL centrifuge tube with a ground glass stopper or Teflon-lined cap. Cap and shake vigorously for 1 minute. Centrifuge the capped flask briefly. Quickly transfer supernatant water to a 50 mL volumetric flask equipped with a ground-glass stopper. Follow Section 9.1.1 starting with the addition of 2 mL of hexadecane.

9.2 GC/FID Analysis

Inject the same volume of sample hexadecane extract as the extracted standard mixture in Section 8.2.3.

9.2.1 GC/FID Chromatogram Interpretation -- Following are two options for interpreting the GC/FID Chromatograms.

9.2.1.1 Option A is to use standard mixture #1 containing the aromatics to calculate an approximate concentration of the aromatics in the sample. Use this information to determine the proper dilution for purge and trap if the sample is water, or whether to use the low or medium level GC/MS purge and trap methods if the sample is soil/sediment (see Table 1, Section 9.3 for guidance). This should be the best approach; however, the aromatics may be absent or obscured by higher concentrations of other purgeables. In these cases, Option B may be the best approach.

9.2.1.2 Option B is to use standard mixture #2 containing n-nonane and n-dodecane to calculate a factor. Use the factor to calculate a dilution for purge and trap of a water sample or to determine whether to use the low or medium level GC/MS purge and trap methods for soil/sediment samples (see Table 1, Section 9.3 for guidance). All purgeables of interest have retention times less than the n-dodecane.

9.3 Analytical Decision Point

9.3.1 Water

9.3.1.1 Compare the chromatograms of the hexadecane extract of the sample with those of the reagent blank and extract of the standard.

9.3.1.2 If no peaks are noted, other than those also in the reagent blank, analyze a 5 mL water sample by purge and trap GC/MS.

9.3.1.3 If peaks are present prior to the n-dodecane and the aromatics are distinguishable, follow Option A (Section 9.2.1).

9.3.1.4 If peaks are present prior to the n-dodecane but the aromatics are absent or indistinguishable, use option B as follows: if all peaks are ≤3% of the n-nonane, analyze a 5 mL water sample by purge and trap GC/MS. If any peaks are ≥3%of the n-nonane, measure the peak height or area of the major peak and calculate the dilution factor as follows:

$$\text{Dilution Factor} = \frac{\text{Peak area of sample major peak}}{\text{Peak area of n-nonane}} \times 50$$

The water sample will be diluted using the calculated factor just prior to purge and trap GC/MS analysis.

9.3.2 Soil/Sediment

9.3.2.1 Compare the chromatograms of the hexadecane extract of the sample with those of the reagent blank and extract of the standard.

9.3.2.2 If no peaks are noted, other than those also in the reagent blank, analyze a 5 g sample by low level GC/MS.

9.3.2.3 If peaks are present prior to the n-dodecane and the aromatics are distinguishable, follow Option A (Section 9.2.1) and the concentration information in Table 1, to determine whether to analyze by low or medium level method.

9.3.2.4 If peaks are present prior to the n-nonane but the aromatics are absent or indistinguishable, and using Option B as follows, calculate a factor using the following formula:

$$\text{X Factor} = \frac{\text{Peak area of sample major peak}}{\text{Peak area of n-nonane}}$$

Table 1
Determination of GC/MS Purge and Trap Method

X Factor	Analyze by	Approximate Concentration Range* (µg/Kg)
0-1.0	low level method	0-1,200
> 1.0	medium level method	>1,200

* This concentration range is based on the response of aromatics to GC/FID. When comparing GC/FID responses, the concentration for halomethanes is 20 times higher, and that for haloethanes is 10 times higher.

APPENDIX B - MODIFIED SW-846 METHOD 5035 FOR VOLATILES IN LOW LEVEL SOILS

1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze low level sediment and soil samples from hazardous waste sites for the volatile organic compounds on the Target Compound List (TCL, see Exhibit C). The method includes sample preparation, screening to determine the approximate concentration of organic constituents in the sample, and the actual analysis which is based on a closed-system purge and trap gas chromatograph/mass spectrometer (GC/MS) method.

2.0 SUMMARY OF METHOD

2.1 Low level volatile organic compounds are determined by analyzing approximately 5 g of sample, in a pre-weighed vial with a septum-sealed screw-cap (see Section 5.0) that already contains a stirring bar and a sodium bisulfate preservative solution. Note: The sodium bisulfate preservative and the stirring bar may be omitted under certain circumstances (see Sections 9.3.2 and 9.3.8). The entire vial is placed into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged through a sorbent trap using an inert gas combined with agitation of the sample. When purging is complete, the trap is heated and backflushed with helium to desorb the purgeable compounds onto a gas chromatograph column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

2.2 The sample introduction technique in Section 2.1 is not applicable to all samples. If sample screening indicates that the soil/sediment sample should be analyzed as a medium level sample, the Contractor shall follow the procedure described in Exhibit D-VOA Section 10.1.5 for medium level soil/sediment samples.

3.0 INTERFERENCES

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

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling.

3.3 Contamination by carryover can occur whenever medium level and low level samples are sequentially analyzed. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device. The trap and other parts of the system are also subjected to

contamination; therefore, frequent bakeout and purging of the entire system may be required.

3.4 *The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.*

4.0 *SAFETY*

4.1 *The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.*

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

5.0 *EQUIPMENT AND SUPPLIES*

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

5.1 *Sample Containers*

The specific sample containers required will depend on the purge-and-trap system to be employed. Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water, and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. The Contractor shall consult the purge-and-trap system manufacturer's

instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices.

5.2 Glassware

5.2.1 Syringes - 25 mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used). 5.0, 1.0, and 0.5 mL syringes gas-tight with shut-off valve.

5.2.2 Syringe valve - 2-way with Luer ends.

5.2.3 Micro syringes - 25 µL with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent). 10 and 100 µL.

5.2.4 60-mL, septum-sealed glass vials to collect samples for screening, dry weight determination.

5.2.5 40-mL, screw-cap, PTFE lined, septum-sealed glass vials. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

5.2.6 Volumetric flasks - Class A, 10-mL and 100-mL, with ground glass stoppers.

5.2.7 Disposable Pasteur pipettes.

5.3 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

5.4 Balances - analytical, capable of accurately weighing ± 0.0001 g, and a top-loading balance capable of weighing 100 g ± 0.01 g. The balances must be calibrated with class S weights or known reference weights once per each 12-hour work shift. The balances must be calibrated with class S weights at a minimum of once per month. The balances must also be annually checked by a certified technician.

5.5 Purge and Trap Device - consists of a unit that automatically adds water, SMCs, and internal standards to a hermetically sealed vial containing the sample, purges the volatile compounds using an inert gas stream while agitating the contents of the vial, and also traps the released volatile compounds for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

5.5.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5 g soil/sediment sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during

purging, (e.g., using a magnetic stirring bar, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed volatile compounds to the gas chromatograph.

5.5.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. Starting from the inlet, the trap must contain equal amounts of the absorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60) mesh (Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap.

- *2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent)*
- *Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent*
- *Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen*

Trapping materials other than those listed above may also be used, provided that they meet the specifications listed in Exhibit D-VOA Sections 6.4.2 and 6.4.4.

5.5.3 The desorber for the trap must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 220 °C during bakeout mode.

5.6 Gas Chromatograph/Mass Spectrometer (GC/MS) System

5.6.1 Gas chromatograph/mass spectrometer system specifications and requirements are described in Exhibit D-VOA Section 6.6.

6.0 REAGENTS AND STANDARDS

6.1 Reagents

6.1.1 Reagent water - defined as water in which an interferant is not observed at or above the CRQL of the analytes of interest. 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).

6.1.1.1 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.1.2 Reagent water may also 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 and seal with a Teflon-lined septum and cap.

6.1.2 Methanol - pesticide quality or equivalent.

6.1.3 Sodium bisulfate - ACS reagent grade or equivalent.

6.2 Standards

6.2.1 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.

6.2.2 The Contractor shall follow the procedures described in Exhibit D-VOA Section 7.2 for preparing stock standards, secondary dilutions, and all working standard solutions.

6.3 Storage of Standard Solutions

6.3.1 The Contractor shall follow the procedures described in Exhibit D-VOA Section 7.3 for storage of all standard solutions.

6.3.2 The Contractor is responsible for maintaining the integrity of standard solutions and verifying prior to use. This means that standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in the solution.

7.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

7.1 Sample Collection and Preservation

7.1.1 Soil/sediment samples should be collected in field core sampling/storage containers (i.e., EnCore™ or equivalent) and 60 mL septum-sealed glass vials in sufficient quantity to perform the analysis. The field core sampling/storage containers should contain approximately 5 g of sample each. The Contractor shall transfer the contents of the field core sampling container immediately upon receipt into the closed-system sample vial prepared as described in Section 9.3 below and record the date and time of transfer. The specific requirements for site sample collection are outlined by the Region. If soil/sediment samples are received in pre-prepared closed-system purge-and-trap sample vials as described in Section 9.3, then the Contractor shall proceed to Section 9.3.9 and determine final sample weight.

7.1.2 All samples must be iced or refrigerated at 4 °C (±2 °C) from the time of collection until analysis.

7.2 Procedure for Sample Storage

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

7.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants and in a refrigerator used only for storage of volatile samples.

7.2.3 All volatile samples in an SDG must be stored together in the same refrigerator.

7.2.4 Storage blanks shall be stored with samples until all samples are analyzed.

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

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

7.3 Contract Required Holding Times

Analysis of soil/sediment samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR). As part of the Agency's QA program, the Agency may provide Performance Evaluation (PE) samples as standard extracts which the Contractor is required to prepare per the instructions provided by the Agency. PE samples must be prepared and analyzed concurrently with the samples in the SDG. The contract required 10 day holding time does not apply to PE samples received as standard extracts.

8.0 CALIBRATION AND STANDARDIZATION

8.1 Purge and Trap

8.1.1 Assemble a purge-and-trap device that meets the specification in Section 5.5 and that is connected to a gas chromatograph/mass spectrometer system.

8.1.2 Before initial use, condition the trap overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, condition the trap for 10 min at 180°C 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.

8.1.3 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of reagent water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer. Once established, the same purge and trap conditions must be used for the analysis of all standards, samples, and blanks.

8.2 Gas Chromatograph/Mass Spectrometer

The Contractor shall follow the instrument conditions described in Exhibit D-VOA Sections 9.1.2 and 9.1.3.

8.3 GC/MS Calibration (Tuning) and Ion Abundance

The Contractor shall follow the procedure described in Exhibit D-VOA Section 9.2. All technical acceptance criteria for the GC/MS performance check shall be met before any standards or samples, including MS/MSDs or required blanks, are analyzed. Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis at no additional cost to the Agency.

8.4 Initial Calibration

The Contractor shall follow the procedure described in Exhibit D-VOA Section 9.3. However, the volume of reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before sample addition plus the reagent water added by the instrument). The calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., approximately 1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the system monitoring compounds (SMCs) using standards at five concentrations, it may be necessary to disable the automatic addition of SMCs to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

All technical acceptance criteria for GC/MS initial calibration specified in Exhibit D-VOA Section 9.3.5 shall be met prior to the analysis of any samples, including MS/MSDs or required blanks. Any samples or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the Agency.

8.5 Continuing Calibration

The Contractor shall follow the procedure for continuing calibration described in Exhibit D-VOA Section 9.4. However, the continuing calibration standard shall be prepared in the same manner as the initial calibration standard of the same concentration as specified in Section 8.4 above (i.e., addition of the sodium bisulfate preservative).

All technical acceptance criteria for continuing calibration specified in Exhibit D-VOA Section 9.4.5 shall be met prior to the analysis of any samples, including MS/MSDs or required blanks. Any samples or required blanks analyzed when continuing calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the Agency.

9.0 PROCEDURE

9.1 The Contractor must determine whether a soil/sediment sample should be analyzed by the low or medium method. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system thereby requiring extensive cleanup and instrument maintenance. The Contractor may follow one of the screening procedures identified in Exhibit D-VOA Section 10.1.4.2. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample. If, based on the screening results, medium level analysis is required, the Contractor shall follow the procedure in Exhibit D-VOA Section 10.1.5. If the Contractor received a pre-weighed sample preserved in methanol (see Section 7.1.1), this sample shall be utilized for the medium level analysis. It is the responsibility of the Contractor to analyze the sample at the correct level.

9.2 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact SMO to notify them of the problem. SMO will contact the Region for instructions. The Region will either require that no sample analyses be performed or will require that a reduced volume be used for the sample analysis.

9.3 Sample Preparation

9.3.1 The following steps apply to the preparation of vials used for the analysis of low level soil/sediment samples by the closed-system purge-and-trap equipment described in this method.

9.3.2 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

9.3.3 Add approximately 1 g of sodium bisulfate preservative to each vial. If samples significantly smaller or larger than 5 g are to be used, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤ 2.

9.3.4 Add 5 mL of reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target compounds.

9.3.5 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted vials are used, seal both ends as recommended by the manufacturer.

9.3.6 Affix a label to each vial and weigh the prepared vial to the nearest 0.01 g. Record the tare weight.

9.3.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, system monitoring compounds, matrix spikes and internal standards should only be added to the vials after the sample has been

added to the vial. The standards should be introduced either manually by puncturing the septum with a small-gauge needle or automatically by the purge-and-trap system just prior to analysis.

9.3.8 Using the sample collection device, transfer the contents (approximately 5 g) into the sample vial containing the preservative solution. This sample transfer must be performed rapidly to minimize loss of volatile compounds. Quickly brush any soil off the vial and immediately seal the vial with the septum and screw-cap. The soil vial is hermetically sealed and must remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. Record the date and time of sample transfer onto the pre-prepared vials and submit with the data package.

NOTE: Soil samples that contain carbonate minerals may effervesce upon contact with the acidic preservative solution in the sample vial. Therefore, if samples are known or suspected to contain high levels of carbonates, a test sample (from the 60 mL glass vial) should be added to a clean vial and checked for effervescence. If a rapid or vigorous reaction occurs, the Contractor may discard the test sample and proceed with sample preparation by transferring the contents of the field core sampling/storage container into a clean vial that does not contain the preservative.

9.3.9 Weigh the vial and contents to the nearest 0.01 g and record this weight. Sample weight is determined by subtracting the sample vial tared weight determined above from this final weight.

9.4 Sample Purge-and-Trap

9.4.1 Prior to sample purge, all soil/sediment samples must be allowed to warm to ambient temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instruction.

9.4.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of reagent water, 10 uL of the internal standard spiking solution (Exhibit D-VOA Section 7.2.4.3), and 10 uL of the system monitoring compound spiking solution (Exhibit D-VOA Section 7.2.4.1). ***All samples, including MS and MSD, standards, and blanks, within an SDG must have the same amount of reagent water added. Do not increase/change the amount of system monitoring compound and internal standard solution added.*** *Prior to purging, heat the sample vial to 40 °C for 1.5 minutes, or as described by the manufacturer.*

9.4.3 Purge the sample with helium or another inert gas at a flow rate of 20 to 40 mL/minute for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

9.4.4 If a non-cryogenic interface is to be utilized, place the purge-and-trap system in the desorb mode after the 11-minute purge, and preheat the trap to 180 °C without a flow of desorption gas. Start the flow

of desorption gas at 10 mL/minute for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

9.4.5 If a cryogenic interface is to be utilized, place the purge-and-trap system in the desorb mode after the 11-minute purge, making sure that the cryogenic interface is at -150 °C or lower, and rapidly heat the trap to 180 °C while backflushing with an inert gas at 4 mL/minute for about 5 minutes. At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250 °C. Begin the temperature program of the gas chromatograph and start the data acquisition.

9.4.6 After desorbing the sample for 4 to 5 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 180 °C. After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

9.5 Sample Dilutions

If the on column concentration of any target compound exceeds the initial calibration range from the analysis of 5 g sample, a smaller sample size must be analyzed utilizing the procedure and methodology described in Exhibit D-VOA. Guidance in performing dilutions and exceptions to this requirement are given in Sections 10.1.6.2 through 10.1.6.10 of Exhibit D-VOA.

9.6 Percent Moisture Determination

It is highly recommended that the percent moisture determination only be made after the analyst has determined that no sample aliquots will be taken from the 60 mL vial for further analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. The Contractor shall follow the procedure described in Exhibit D-VOA Section 10.3 for determining percent moisture of samples.

10.0 DATA ANALYSIS AND CALCULATIONS

The Contractor shall perform qualitative and quantitative analysis for the target and non-target compounds following the procedures described in Exhibit D-VOA Section 11.0. All technical acceptance criteria for sample analysis described in Exhibit D-VOA Section 11.3 shall be met or the corrective action for sample analysis described in Section 11.4 of Exhibit D-VOA shall be followed.

11.0 QUALITY CONTROL

11.1 Blank Analyses

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

11.1.1.1 METHOD BLANK - a volume of purified solid matrix (prepared as described in Sections 9.3.2 through 9.3.5) and carried through the entire analytical procedure. The weight of the purified solid matrix must be approximately equal to the weight of samples

associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

11.1.1.2 STORAGE BLANK - upon receipt of the first samples in an SDG, two of the sample vials to be used for the closed-system purge-and-trap analysis (prepared as described in Sections 9.3.2 through 9.3.5) are filled with reagent water. The vials are stored with the samples in the SDG under the same conditions. After all samples in the SDG have been analyzed, the storage blank is analyzed. The storage blank indicates whether contamination may have occurred during storage of samples.

11.1.1.3 INSTRUMENT BLANK - a 5.0 mL aliquot of reagent water that is added to the sample vial (prepared as described in Sections 9.3.2 through 9.3.5) and carried through the entire analytical procedure. Instrument blanks are analyzed after a sample which contains a target compound exceeding the initial calibration range. The results from the instrument blank analysis indicate whether there is contamination from a previous sample.

11.1.2 Frequency of Blank Analyses

11.1.2.1 The method blank ***must*** *be analyzed at least once during every 12-hour time period on each GC/MS system used for volatile analysis (see Section 9.2.2 of Exhibit D-VOA for the definition of the 12-hour time period).*

11.1.2.2 The method blank ***must*** *be analyzed after the continuing calibration and before any samples, including matrix spike/matrix spike duplicates, or storage blanks are analyzed. The method blank must be analyzed after the initial calibration sequence if samples are analyzed before the 12-hour period expires. A method blank must be analyzed in each 12-hour time period in which samples, including matrix spikes/matrix spike duplicates, and storage blanks are analyzed.*

11.1.2.3 A minimum of one storage blank must be analyzed per SDG after all samples for that SDG have been analyzed.

11.1.2.4 The Contractor must demonstrate that there is no carryover from contaminated samples before data from subsequent analyses may be used. Samples may contain target compounds at levels exceeding the initial calibration range. An instrument blank must be analyzed after the sample that exceeds the calibration range (also in the same purge inlet if an autosampler is used) or a sample that meets the maximum contamination criteria in Section 11.3.8 of Exhibit D-VOA must be analyzed. For these purposes, if the instrument blank meets the technical acceptance criteria for blank analyses 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., contaminated), the system must be decontaminated. Until an instrument blank meets the blank technical acceptance criteria or a sample meets the maximum contamination criteria in Section 11.3.8 of Exhibit D-

VOA, any samples analyzed since the original contaminated sample will require reanalysis at no additional cost to the Agency.

NOTE: Only the instrument blank which demonstrates that there was no carryover from the previous sample or the instrument blank that demonstrates that the system is clean (Section 12.1.4 Exhibit D-VOA) needs to be reported. Instrument blanks analyzed during the instrument decontamination process which exceed the requirements listed in Exhibit D-VOA Section 12.1.4 do not need to be reported.

11.1.3 Procedure for Blank Analyses

11.1.3.1 Method blanks shall be analyzed in the same manner as the associated samples, following the procedure described in Section 9.4.

11.1.3.2 Storage/instrument blanks shall be analyzed in the same manner as the associated samples following the procedure outlined in section 9.4.

11.1.3.3 A storage blank may be analyzed and reported as a soil sample if the SDG contains only soil samples.

11.1.3.4 Identify and quantitate analytes according to Section 11.0 of Exhibit D-VOA.

11.1.4 Technical Acceptance Criteria for Blank Analyses

11.1.4.1 All technical acceptance criteria for blank analyses described in Exhibit D-VOA Section 12.1.4 shall be met or corrective action for blank analyses described in Exhibit D-VOA Section 12.1.5 shall be followed.

11.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

11.2.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the methods used for volatile analyses, the Agency has prescribed a mixture of volatile target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

11.2.2 Frequency of MS/MSD

11.2.2.1 A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix for the following, whichever is most frequent:

- *Each SDG, or*
- *Each matrix within an SDG, or*
- *Each group of samples of a similar concentration level (soils only).*

MS/MSD samples shall be analyzed unless otherwise specified on the Traffic Report (TR). If no MS/MSD samples are specified on the TR, the Contractor shall contact SMO to confirm that MS/MSD analyses are not required.

11.2.2.2 As a part of the Agency's QA/QC program, water rinsate samples and/or field/trip blanks (field QC) may accompany soil/sediment samples that are delivered to a laboratory for analysis. The Contractor shall not perform MS/MSD analysis on any of the field QC samples.

11.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample, less than the required amount, remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify the Region (through SMO) that insufficient sample was received and identify the EPA sample selected for the MS/MSD analysis. The rationale for the choice of a sample other than the one designated by the Region shall be documented in the SDG Narrative.

11.2.2.4 If there is insufficient sample remaining for any of the samples in an SDG to perform an MS/MSD, then the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no MS/MSD is required, or specify an alternative means of performing the MS/MSD analysis. SMO will notify the Contractor of the resolution. The Contractor shall document the decision in the SDG Narrative.

11.2.2.5 If it appears that the Region has requested MS/MSD analysis at a greater frequency than required by the contract, the Contractor shall contact SMO. SMO will contact the Region to determine which samples should have an MS/MSD performed on them. SMO will notify the Contractor of the Region's decision. The Contractor shall document the decision in the SDG Narrative. If this procedure is not followed, the Contractor will not be paid for the MS/MSD analysis performed at a greater frequency than required by the contract.

11.2.2.6 When a Contractor receives ***only*** *a Performance Evaluation (PE) sample(s), no MS/MSD shall be performed within that SDG.*

11.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the MS/MSD when the Region did not designate samples to be used for this purpose.

11.2.3 Procedure for Preparing MS/MSD

11.2.3.1 To prepare a matrix spike and matrix spike duplicate for low level soil/sediment samples, follow the procedure outlined in Section 9.3. Add 10 µL of the matrix spike solution (Exhibit D-VOA Section 7.2.4.2) either manually by puncturing the septum with a small-gauge needle or automatically by the purge-and-trap system just prior to analysis. Analyze the matrix spike and matrix spike

duplicate samples by the procedure described in Section 9.4. Do ***not*** *further dilute MS/MSD samples to get* ***either*** *spiked* ***or*** *non-spiked analytes within calibration range.*

11.2.4 Calculations for MS/MSD

The Contractor shall calculate the concentrations of the matrix spike compounds in the matrix spike and matrix spike duplicate samples using the same equation as used for target compounds (Equation 6) Exhibit D-VOA Section 11.2.1.3. The recovery of each matrix spike compound in the matrix spike and matrix spike duplicate samples and the relative percent difference (RPD) of the recoveries shall be calculated as specified in Exhibit D-VOA Section 12.2.4.

11.2.5 Technical Acceptance Criteria for MS/MSD

All technical acceptance criteria for MS/MSD specified in Exhibit D-VOA Section 12.2.5 must be met or corrective action for MS/MSD in Exhibit D-VOA Section 12.2.6 shall be followed.

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND REQUIREMENTS

Exhibit E - Quality Assurance/Quality Control Procedures and Requirements

Table of Contents

1.0 OVERVIEW

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

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

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

2.0 INTRODUCTION

2.1 Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the QC procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories in the CLP. However, the validation of these methods does not guarantee that they perform equally well for all sample matrices encountered. Inaccuracies can also result from causes other than unanticipated matrix effects, such as sampling artifacts, equipment malfunctions, and operator error. Therefore, the quality control component of each method is indispensable.

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

2.3 The necessary components of a complete QA/QC program include internal QC criteria that demonstrate acceptable levels of performance, as determined by QA review. External review of data and procedures is accomplished by the monitoring activities of the National Program Office, Regional data users, Sample Management Office (SMO), and *the Quality Assurance Technical Support (QATS) Laboratory*. Each external review accomplishes a different purpose. These reviews are described in specific sections of this exhibit. Laboratory evaluation samples, GC/MS *and GC/EC* tape audits, and data packages provide an external QA reference for the program. A Contractor on-site evaluation system is also part of the external QA monitoring. A feedback loop provides the results of the various review functions to the Contractors through direct communications with the Technical Project Officers (TPOs) and Administrative Project Officers (APOs).

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

2.5 In order to assure that the product delivered by the Contractor meets the requirements of the contract, and to improve interlaboratory data comparison, the Agency requires the following from the Contractor:

- Preparation of and adherence to a written quality assurance plan, the elements of which are designated in Section 3,

- Preparation of and adherence to QA/QC standard operating procedures as described in Section 4,

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

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

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

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

- Submission, upon request, of GC/MS *and/or GC/EC* tapes and applicable documentation for tape audits, including a copy of the sample data package,

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

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

3.0 *QUALITY ASSURANCE PLAN*

3.1 Introduction. The Contractor shall establish a quality assurance program with the objective of providing sound analytical chemical measurements. This program shall incorporate the quality control procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

3.1.1 As evidence of such a program, the Contractor shall prepare a written quality assurance plan (QAP) which describes the procedures that are implemented to achieve the following:

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

3.1.2 The QAP shall present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, standard operating procedures pertaining to each element shall be included or referenced as part of the QAP. The QAP shall be paginated consecutively in ascending order. The QAP shall be available during on-site laboratory evaluations. Additional information relevant to the preparation of a QAP can be found in Agency and American Society for Testing and Materials publications.

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

A. Organization and Personnel

1. QA Policy and Objectives
2. QA Management
   a. Organization
   b. Assignment of QC and QA Responsibilities
   c. Reporting Relationships
   d. QA Document Control Procedures
   e. QA Program Assessment Procedures

3. Personnel

   a. Résumés

   b. Education and Experience

   c. Training Progress

B. Facilities and Equipment

   1. Instrumentation and Backup Alternatives

   2. Maintenance Activities and Schedules

C. Document Control

   1. Contractor Notebook Policy

   2. Sample Tracking/Custody Procedures

   3. Logbook Maintenance and Archiving Procedures

   4. Case File Organization, Preparation, and Review Procedures

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

   6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

   1. Calibration Procedures and Frequency

   2. Sample Preparation/Extraction Procedures

   3. Sample Analysis Procedures

   4. Standards Preparation Procedures

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

E. Data Generation

   1. Data Collection Procedures

   2. Data Reduction Procedures

   3. Data Validation Procedures

   4. Data Reporting and Authorization Procedures

F. Quality Control

1. Solvent, Reagent, and Adsorbent Check Analysis

2. Reference Material Analysis

3. Internal Quality Control Checks

4. Corrective Action and Determination of QC Limit Procedures

5. Responsibility Designation

G. Quality Assurance

1. Data Quality Assurance

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures

5. Quality Assurance Reporting Procedures

6. Responsibility Designation

3.3 Updating and Submitting the Quality Assurance Plan

3.3.1 Initial Submission. During the contract solicitation process, the Contractor is required to submit their QAP to the *Contracting* Officer. Within 60 days after contract award, the Contractor shall revise the QAP to be in full compliance with the requirements of this contract. The Contractor shall maintain the QAP on file at the Contractor's facility for the term of the contract. The revised QAP will become the official QAP under the contract and may be used during legal proceedings. Both the initial QAP submission and the revised QAP shall be paginated consecutively in ascending order. The revised QAP shall include:

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

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

3.3.1.1 The Contractor shall send a copy of the *latest version of the* QAP within 7 days of a request from a Technical Project Officer or Administrative Project Officer. The Agency requestor will designate the recipients.

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

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

3.3.2.1 The Contractor shall amend the QAP within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility. When the QAP is amended, all changes in the QAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the *latest version of the* QAP. The Contractor shall archive all amendments to the QAP for future reference by the Agency.

3.3.2.2 The Contractor shall send a copy of the *latest version of the* QAP within 7 days of a request from a Technical Project Officer or Administrative Project Officer. The Agency requestor will designate the recipients.

3.4 Corrective Actions. If the Contractor fails to adhere to the requirements listed in Section 3, the Contractor may expect, but the Agency is not limited to, the following actions: reduction of numbers of samples sent under this contract, suspension of sample shipment to the Contractor, a GC/MS *and/or GC/EC* tape audit, a data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions.

4.0 STANDARD OPERATING PROCEDURES

4.1 Introduction. In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of standard operating procedures (SOPs). As defined by EPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks.

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

4.1.2 All SOPs shall reflect activities as they are currently performed by the Contractor. In addition, all SOPs shall be:

- Consistent with current Agency regulations, guidelines, and the CLP contract's requirements.
- Consistent with instrument manufacturers' specific instruction manuals.
- Available to the Agency during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site evaluations, Contractor personnel may be asked to demonstrate the application of the SOPs.
- Available to the designated recipients within 7 days, upon request by the Technical Project Officer or Administrative Project Officer.
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Capable of demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.
- Available at specific work stations as appropriate.

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

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

- Title page,
- Scope and application,
- Definitions,
- Procedures,
- QC limits,
- Corrective action procedures, including procedures for secondary review of information being generated,
- Documentation description and example forms,
- Miscellaneous notes and precautions, and
- References.

4.3 Requirements. The Contractor shall maintain the following SOPs.

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

4.3.2 Sample Receipt and Storage

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

4.3.3 Sample Preparation

- Reagent purity check procedures and documentation
- Extraction procedures
- Extraction bench sheets
- Extraction logbook maintenance

4.3.4 Glassware Cleaning

4.3.5 Calibration (Balances)

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

4.3.6 Analytical Procedures (for each Analytical System, including GPC)

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

4.3.7 Maintenance Activities (for each Analytical System, including GPC)

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

4.3.8 Analytical Standards

- Standard coding/identification and inventory system
- Standards preparation logbook(s)
- Standards preparation procedures
- Procedures for equivalency/traceability analyses and documentation
- Purity logbook (primary standards and solvents)
- Storage, replacement, and labeling requirements
- QC and corrective action measures

4.3.9 Data Reduction Procedures

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

4.3.10 Documentation Policy/Procedures

- Contractor/analysts' notebook policy, including review policy
- Complete SDG File contents
- Complete SDG File organization and assembly procedures, including review policy
- Document inventory procedures, including review policy

4.3.11 Data Validation/Self-Inspection Procedures

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

4.3.12 Data Management and Handling

- Procedures for controlling and estimating data entry errors
- Procedures for reviewing changes to data and deliverables and ensuring traceability of updates
- Life cycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems
- Database security, backup and archival procedures including recovery from system failures
- System maintenance procedures and response time
- Individuals(s) responsible for system operation, maintenance, data integrity and security
- Specifications for staff training procedures
- Storage, retrieval, and verification of the completeness and readability of GC/MS and GC/EC files transferred to magnetic media

4.4 Submitting and Updating SOPs

4.4.1 Initial Submission. During the contract solicitation process, the Contractor is required to submit their SOPs to the Contracting Officer. Within 60 days after contract award, the Contractor shall prepare and maintain on file, at their facility, a complete, revised set of SOPs fully compliant with the requirements of this contract. The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings. Both the initial submission of SOPs and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

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

4.4.1.1 The Contractor shall send a complete set of the latest version of SOPs or individually requested SOPs within 7 days of a request from a Technical Project Officer or Administrative Project Officer. The Agency requestor will designate the recipients.

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

- The Agency modifies the contract,
- The Agency notifies the Contractor of deficiencies in their SOPs,
- The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,
- The Contractor's procedures change,
- The Contractor identifies deficiencies resulting from the internal review of their SOPs documentation, or
- The Contractor identifies deficiencies resulting from the internal review of their procedures.

4.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended/new SOPs shall have the date on which the changes were implemented.

4.4.2.2 When existing SOPs are amended or new SOPs are written, the Contractor shall document the reason(s) for the change, and maintain the amended or new SOPs on file at the laboratory facility. Documentation of the reason(s) for the changes shall be maintained on file with the amended SOPs or new SOPs.

4.4.2.3 The Contractor shall send a complete set of *the latest version of* SOPs or individually requested SOPs within 7 days of a request from a Technical Project Officer or Administrative Project Officer. The Agency requestor will designate the recipients.

4.4.2.4 Documentation of the reason(s) for changes to the SOPs shall also be submitted with the SOPs. An alternate delivery schedule for submitting the amended/new SOPs and their documentation may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 30 days for amending/writing new SOPs. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs. The Contractor shall proceed and not assume

that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

4.5 Corrective Actions. If the Contractor fails to adhere to the requirements listed in Section 4, the Contractor may expect, but the Agency is not limited to, the following action: reduction of number of samples sent under this contract, suspension of sample shipment to the Contractor, a GC/MS *and/or GC/EC* tape audit, a data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions.

5.0 ANALYTICAL STANDARDS REQUIREMENTS

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

5.2 Preparation of Chemical Standards from the Neat High Purity Bulk Material. A Contractor may prepare their chemical standards from neat materials. Contractors shall obtain the highest purity possible when purchasing neat chemical standards; when standards are purchased at less than 97% purity, the Contractor shall document the reason why a higher purity could not be obtained.

5.2.1 Neat chemical standards shall be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of neat chemicals is essential in order to safeguard them from decomposition.

5.2.2 The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the Contractor's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use either differential scanning calorimetry, gas chromatography with flame ionization detection, high performance liquid chromatography, infrared spectrometry, or other appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

EQ. 1

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

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

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

5.2.4 Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. It is the Contractor's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards are correctly identified.

Identification confirmation, when performed, shall use gas chromatography/mass spectrometry analysis on at least two different analytical columns, or other appropriate techniques.

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

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

5.2.7 Log notebooks shall be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person. All solution standards shall be refrigerated when not in use. All solution standards shall be clearly labeled as to the identity of the compound or compounds, concentration, date prepared, solvent, and initials of the preparer.

5.3 Purchase of Chemical Standards Already in Solution. Solutions of analytical reference standards can be purchased by Contractors provided they meet the following criteria.

5.3.1 Contractors shall maintain the following documentation to verify the integrity of the standard solutions they purchase:

- Mass spectral identification confirmation of the solution,
- Purity confirmation of the solution, and
- Chromatographic and quantitative documentation that the solution standard was QC checked according to the following section.

5.3.2 The Contractor shall purchase standards for which the quality is demonstrated statistically and analytically. One way this may be demonstrated is to prepare and analyze three solutions, a high standard, a low standard, and a standard at the target concentration (see Sections 5.3.2.1 and 5.3.2.2). The Contractor shall have documentation to demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the Student's t-test in Section 5.3.2.4. If this is achieved, the Contractor shall then

demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test in Section 5.3.2.5. The standard is certified to be within 10% of the target concentration using the equations in Section 5.3.2.6. If this procedure is used, the Contractor shall document that the following have been achieved.

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

5.3.2.2 Six replicate analyses of each standard (a total of 18 analyses) shall be performed in the following sequence: low standard, target, high standard, low standard, target standard, high standard,

5.3.2.3 The mean and variance of the six results for each solution shall be calculated.

EQ. 2

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

EQ. 3

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

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

EQ. 4

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

If the square root of V_p is less than 1% of M_2, then $M_2^2/10,000$ shall be used as the value of V_p in all subsequent calculations.

5.3.2.4 The test statistic shall be calculated.

EQ. 5

$$Test\ Statistic = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\sqrt{\frac{V_p}{3}}}$$

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

5.3.2.5 The test statistic shall be calculated.

EQ. 6

$$Test\ Statistic = \frac{\left| M_2 - \frac{M_1}{1.8} - \frac{M_3}{2.2} \right|}{\sqrt{\frac{V_p}{4}}}$$

If the test statistic exceeds 2.13, then the target standard concentration has not been demonstrated to be the midway between the high and low standards. In such a case, the standards are not acceptable.

5.3.2.6 The 95% confidence intervals for the mean result of each standard shall be calculated.

EQ. 7

$$Interval\ for\ Low\ Standard = M_1 \pm 2.13 \sqrt{\frac{V_p}{6}}$$

EQ. 8

$$Interval\ for\ Target\ Standard = M_2 \pm 2.13 \sqrt{\frac{V_p}{6}}$$

EQ. 9

$$Interval\ for\ High\ Standard = M_3 \pm 2.13\sqrt{\frac{V_p}{6}}$$

5.3.2.6.1 These intervals shall not overlap. If overlap is observed, the ability to discriminate the 10% difference in concentrations has not been demonstrated. In such a case, the standards are not acceptable.

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

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

5.5 Documentation of the Verification and Preparation of Chemical Standards. It is the responsibility of each Contractor to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed.

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

5.5.2 Upon request by the Technical Project Officer or Administrative Project Officer, the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of the receipt of request to the recipients he/she designates.

5.5.3 The Agency may generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards or may discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer and the Administrative Project Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an

alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the standards documentation report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

5.5.4 If new SOPs are required to be written or SOPs are required to be amended because of deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Section 4.

5.6 Corrective Actions. If the Contractor fails to adhere to the requirements listed in Section 5, the Contractor may expect, but the Agency is not limited to, the following actions: reduction of number of samples sent under the contract, suspension of sample shipment to Contractor, a GC/MS *and/or GC/EC* tape audit, a data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions.

6.0 DETERMINATION OF METHOD EQUIVALENCY FOR ALTERNATIVE EXTRACTION PROCEDURES

If the Contractor wishes to use one or both of the alternative extraction procedures described in Section 1.3 Exhibits D SVOA and D PEST under Scope and Application, the Contractor must develop and implement SOPs for performing the alternative extractions in accordance to Exhibit E Section 4.0. In addition, the Contractor shall maintain documentation, including raw data, to demonstrate the equivalence of the alternative extraction procedures to those specified in Sections 10.1.4.4, Exhibit D SVOA and 10.1.5.3, Exhibit D PEST. The required documentation for demonstrating extraction equivalence include an Initial Precision Recovery study as described below.

6.1 Initial Precision Recovery (IPR) Study

6.1.1 For the semivolatile fraction, the Contractor shall spike four (4) solid samples (e.g., anhydrous sodium sulfate) with all the target compounds at concentrations equal to three (3) times the Contract Required Quantitation Limits (CRQL) listed in Exhibit C under semivolatiles. For pesticides/Aroclors, the Contractor shall spike four (4) solid samples with the single component target compounds and an additional four (4) solid samples with Aroclor 1254 only at concentrations equal to three (3) times the CRQLs listed in Exhibit C under pesticides/Aroclors. Each sample must contain the appropriate surrogates at the concentrations specified in Section 10.1.4.4, Exhibit D SVOA or Section 10.1.5.3.3, Exhibit D PEST.

6.1.2 The Contractor shall achieve the following recovery limits for the matrix spike compounds in each of the four replicates of the IPR study.

Recovery Limits for Matrix Spike Compounds

Semivolatiles

Compound	*Recovery Limits*
Phenol	*26-90*
2-Chlorophenol	*25-102*
N-Nitroso-di-n-propylamine	*41-126*
4-Chloro-3-methylphenol	*26-103*
Acenaphthene	*31-137*
4-Nitrophenol	*11-114*
2,4-Dinitrotoluene	*28-89*
Pentachlorophenol	*17-109*
Pyrene	*35-142*

Pesticides

Compound	*Recovery Limits*
Gamma-BHC (Lindane)	*46-127*
Heptachlor	*35-130*
Aldrin	*34-132*
Dieldrin	*31-134*
Endrin	*42-139*
4,4'-DDT	*23-134*

6.1.3 The advisory limits for the mean percent recoveries (%R) of all other target compounds in the IPR study is 75% to 125% of the spiked amount.

6.1.4 The advisory limits for the % Relative Standard Deviation (%RSD) of the IPR recoveries for each compound is 25% and shall not exceed 50%.

6.2 Analytical Protocol Required

6.2.1 The Contractor shall extract all IPR samples using SW-846 Methods 3541 (Revision 0, September 1994) and 3545 (Revision 0, December 1996) modified where appropriate to achieve the requirements of this SOW (i.e., CRQLs and all technical acceptance criteria). All modifications to the extraction procedure (e.g., use of methylene chloride/acetone (1:1, v/v) for pesticide extraction) shall be adequately documented and submitted with the data package.

6.2.2 The Contractor shall follow the sample cleanup procedures described in Exhibit D SVOA for semivolatiles and Exhibit D PEST for pesticides/Aroclors.

6.2.3 The Contractor shall analyze the sample extracts for the IPR study following the procedures described in Section 10.6, Exhibit D SVOA for semivolatile compounds and Section 10.2, Exhibit D PEST for pesticides/Aroclors compounds.

6.3 Quantitation Limits/Quality Control Requirements

6.3.1 The Contractor shall achieve the CRQLs specified in Exhibit C under semivolatiles and pesticides/Aroclors.

6.3.2 The Contractor shall follow all QC requirements outlined in Exhibit D SVOA and Exhibit D PEST including frequency of method blanks, instrument blanks, instrument performance checks, initial and continuing calibrations or calibration verifications, internal standards, and surrogates.

6.3.3 All technical acceptance criteria for sample analysis, method blank, and instrument blank analyses described in Exhibit D SVOA and Exhibit D PEST shall be met.

6.3.4 All semivolatile surrogate recoveries shall be within the limits specified in Table 7, Exhibit D SVOA.

6.3.5 The advisory limits for the recovery of pesticide surrogates are 30% to 150%.

6.4 Data Deliverable Requirements

6.4.1 The Contractor shall submit data packages containing all documentation formatted as required in Exhibits B and H (including, but not limited to, SDG Narrative, appropriate summary forms, and raw data). Each IPR replicate shall be reported as a separate sample (i.e., field sample) on Form I. All tuning data, initial calibration data, continuing calibration data, and associated blanks with their raw data must be included in the data package. The Contractor shall include the source of the blank solid samples used for the IPR study in the data deliverables.

6.4.2 The Contractor shall include in the SDG Narrative a discussion of any modifications to the extraction procedures and any problems encountered along with the resolutions. The Contractor shall provide an explanation in the SDG Narrative for any of the target compound recoveries that fall outside the advisory limits. A summary of the IPR results with all calculations must also be included in the SDG Narrative.

6.4.3 Simultaneous delivery of the complete Method Equivalency Data Package shall be made to the following recipients:

EPA: Data Package will be delivered to the laboratory's Administrative Project Officer (APO).

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

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

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

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

7.0 CONTRACT COMPLIANCE SCREENING

7.1 Contract compliance screening (CCS) is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the sample data package delivered to the Agency.

7.2 CCS is performed by the Sample Management Office (SMO) under the direction of the Agency. To assure a uniform review, a set of standardized procedures has been developed to evaluate the sample data package submitted by a Contractor against the technical and completeness requirements of the contract. The government reserves the right to add and/or delete individual checks. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

7.3 CCS results are mailed to the Contractor and all other data recipients. The Contractor has a period of time to correct deficiencies. The Contractor shall send all corrections to the Regional client and SMO.

7.4 The Agency may generate a CCS trend report which summarizes CCS results over a given period of time. The Agency may send the CCS trend report or discuss the CCS trend report during an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer and Administrative Project Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response to the CCS trend report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

7.5 If new SOPs are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Section 4.

7.6 If the Contractor fails to adhere to the requirements listed in Section 7, the Contractor may expect, but the Agency is not limited to, the following actions: reduction of number of samples sent under the contract, suspension of sample shipment to the Contractor, a GC/MS and/or GC/EC tape audit, a data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions.

8.0 REGIONAL DATA REVIEW

8.1 Contractor data are generated to meet the specific needs of the EPA Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end user, based upon functional aspects of data quality. General guidelines for data review have been developed jointly by the Regions and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problems under investigation and the Regional response appropriate to the specific circumstances.

8.2 Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review done at the Sample Management Office, which is designed to identify contractual discrepancies, and the review done by the Program Office, which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for Program and Contractor administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

9.0 PROFICIENCY TESTING

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor must participate in EPA's Proficiency Testing Program. EPA's Proficiency Testing Program involves the analysis of case specific Performance Evaluation (PE) samples and the participation in interlaboratory Quarterly Blind (QB) Audits. The Contractor's analytical PE samples and QB results will be used by EPA to assess and verify the Contractor's continuing ability to produce acceptable analytical data in accordance with the contractual requirements.

9.1 Performance Evaluation Samples

9.1.1 The Performance Evaluation sample(s) may be scheduled with the Contractor as frequently as on an SDG-by-SDG basis. The PE samples may be sent either by the Regional Client or the National Program Office. PE samples will assist EPA in monitoring Contractor performance.

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

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

9.1.4 In addition to PE sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PE sample. When PE sample results are received by EPA, the PE sample results will be evaluated for correct analytical identification and quantitation. EPA will notify the Contractor of unacceptable performance. EPA reserves the right to adjust the PE sample acceptance windows in order to compensate for any unanticipated difficulties with a particular PE sample.

9.1.5 The Contractor shall demonstrate acceptable analytical performance for both identification and quantitation of PE sample analytes. For unacceptable PE sample performance, EPA may take, but is not limited to the following actions: reduce value or rejection of data for the samples, SDG, or Case impacted; Show Cause and/or Cure Notice; reduction in the number of samples shipped to the laboratory;

suspension of sample shipment; an on-site laboratory inspection; a full data package audit; and/or require the laboratory to analyze a Remedial QB.

9.2 Quarterly Blind Audits

9.2.1 Quarterly Blind (QB) Audits may be scheduled concurrently with all contract laboratories up to a frequency of four times a year. A Quarterly Blind Audit is a unique analytical case containing only Performance Evaluation samples (i.e., referred to as Quarterly Blind (QB) samples). The QB samples will be scheduled by the National Program Office through the CLASS Contractor. QB samples will assist EPA in monitoring Contractor performance.

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

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

9.2.4 In addition to QB sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes included in each QB sample. When QB sample results are received by EPA, the QB sample results will be scored for correct analytical identification and quantitation. The QB sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte. EPA will notify the Contractor of unacceptable performance. EPA reserves the right to adjust the PE sample acceptance windows in order to compensate for any unanticipated difficulties with a particular PE sample. The Contractor's QB sample performance will be assessed into one of the following three categories:

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

9.2.4.2 ***Acceptable, Response Explaining Deficiencies Required****: Score greater than 75 percent, but less than 90 percent. Deficiencies exist in the Contractor's performance. Corrective action response required.*

9.2.4.3 ***Unacceptable Performance, Response Explaining Deficiencies Required:*** *Score less than 75 percent. Deficiencies exist in the Contractor's performance to the extent that the National Program Office has determined that the Contractor has not demonstrated the capability to meet the contract requirements. Corrective action response required.*

9.2.5 *In the case of Section 9.2.4.2 or 9.2.4.3, the Contractor shall describe the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a corrective action letter to the Administrative Project Officer, the Technical Project Officer, and the CLP Quality Assurance Coordinator within 14 days of receipt of notification from the Agency.*

9.2.5.1 *An alternate delivery schedule for the corrective action letter may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and Contracting Officer why the laboratory is unable to meet the original delivery schedule listed in Section 9.2.5. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's corrective action letter. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer or Administrative Project Officer.*

9.2.6 *In the case of Section 9.2.4.2 or 9.2.4.3, if new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 4.*

9.2.7 *For unacceptable QB sample performance (Section 9.2.4.3), the EPA may take, but is not limited to the following actions: reduction in the number of samples shipped to the laboratory; suspension of sample shipment; an on-site laboratory inspection; a full data package audit; and/or require the laboratory to analyze a Remedial QB sample; and/or contract sanctions.*

9.2.8 *A Remedial QB Audit is a unique analytical case containing only QB samples. A Remedial QB Audit may be scheduled by the National Program Office with the Contractor(s) for any of the following reasons: unacceptable PE sample performance, unacceptable QB sample performance, and/or major change in the laboratory (e.g., relocation, new owner, or high turn-over of key personnel). Sections 9.2.2 through 9.2.7 apply to the Remedial QB Audit process.*

9.2.9 *If the Contractor fails to adhere to the requirements listed in Section 9, the Contractor may expect, but the Agency is not limited to, the following actions: reduction in the number of samples sent under the contract; suspension of sample shipment to the Contractor; a full data package audit; an on-site laboratory inspection; a Remedial QB sample; and/or contract sanctions.*

10.0 GC/MS *AND GC/EC* TAPE AUDITS

10.1 Overview. Periodically, the Agency requests the GC/MS *and GC/EC* magnetic tapes from Contractors for a specific Case in order to accomplish tape audits. Generally, tape submissions and audits are requested for the following reasons.

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

10.1.1 Depending upon the reason for an audit, the tapes from a recent Case, a specific Case, or a laboratory evaluation sample may be requested. Tape audits provide a mechanism to assess adherence to contractual requirements and to ensure the consistency of data reported on the hardcopy/*electronic deliverables* with that generated on the GC/MS *and GC/EC* tapes. This function provides external monitoring of Program QC requirements and checks adherence of the Contractor to internal QA procedures. In addition, tape audits enable the Agency to evaluate the utility, precision, and accuracy of the analytical methods.

10.1.2 The Contractor shall store <u>all</u> raw and processed GC/MS *and GC/EC* data on magnetic tape, in appropriate instrument manufacturer's format, *uncompressed, and with no security codes*. This tape shall include data for samples, *all QC samples*, blanks, matrix spikes, matrix spike duplicates, initial calibrations, continuing calibrations, *calibration verification standards, including resolution check samples and performance evaluation mixtures, GPC single component and multicomponent and Florisil cartridge check samples and associated calibrations*, and instrument performance check *solutions* (BFB and DFTPP) as well as all Contractor-generated spectral libraries and quantitation reports required to generate the data package. The Contractor shall maintain a written reference logbook of tape files of the EPA sample number, calibration data, standards, blanks, matrix spikes, and matrix spike duplicates. The logbook shall include EPA sample numbers and standard and blank Ids, identified by Case and Sample Delivery Group.

10.1.3 The Contractor is required to retain the GC/MS *and GC/EC* tapes for 365 days after submission of the reconciled Complete SDG File. When submitting GC/MS *and GC/EC* tapes to the Agency, the following materials shall be delivered in response to the request.

10.1.3.1 All associated raw data files for samples, all QC samples, blanks, matrix spikes, matrix spike duplicates, initial calibrations, continuing calibrations, *calibration verification standards, including resolution check samples and performance evaluation mixtures, GPC single component and multicomponent Florisil cartridge check samples and associated calibrations*, and instrument performance check solutions (BFB and DFTPP).

10.1.3.2 All processed data files and quantitation output files associated with the raw data files described in Section *10*.1.3.1.

10.1.3.3 All associated identifications and calculation files *(method files)* used to generate the data submitted in the data package.

10.1.3.4 All Contractor-generated mass spectral library files (NIST/EPA/NIH and/or Wiley, or equivalent, library not required).

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

10.1.3.6 **A directory of *all* files on each tape, *including all subdirectories and the files contained therein.***

10.1.3.7 A copy of the completed sample data package.

10.1.3.8 A statement attesting to the completeness of the GC/MS *and GC/EC* data tape submission, signed and dated by the Contractor's laboratory manager. The Contractor shall also provide a statement attesting that the data reported have not been altered in any way. These statements shall be part of a cover sheet that includes the following information relevant to the data tape submission:

- Contractor name,
- Date of submission,
- Case number,
- SDG number,
- GC/MS *and GC/EC* make and model number,
- Software version,
- Disk drive type (e.g., CDC, PRIAM, etc.),
- File transfer method (e.g., DSD, DTD, FTP, Aquarius, etc.), and
- *Data System Computer,*
- *System Operating Software,*
- *Data System Network,*
- *Tape Backup Software,*
- *Tape Backup Hardware,*

- *Data Analysis Software,*
- *Fraction, and*
- *Volume of data (in Mb) backed up on each tape*
- Names and telephone numbers of two Contractor contacts for further information regarding the submission.

10.2 Submission of the GC/MS *and GC/EC Tapes.* Upon request of the Administrative Project Officer, the Contractor shall send the required GC/MS *and/or GC/EC* tapes and all necessary documentation to the EPA designated recipient (e.g., QATS) within seven (7) days of notification. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than seven days for submission of the GC/MS *and/or GC/EC* tape. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

NOTE: The GC/MS *and GC/EC* tapes shall be shipped according to the procedures in Exhibit F.

10.3 Responding to the GC/MS *and GC/EC* Tape Audit Report. After completion of the GC/MS *and GC/EC* tape audit, the Agency may send a copy of the GC/MS *and GC/EC* tape audit report to the Contractor or may discuss the GC/MS *and GC/EC* tape audit report at an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer and Administrative Project Officer, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the GC/MS *and GC/EC* tape audit report within 14 days of receipt of the report.

10.3.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the GC/MS *and GC/EC* tape report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

10.3.2 If new SOPs are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Section 4.

10.3.3 Maintenance of the Magnetic Tape Storage Device

10.3.3.1 The Contractor shall certify that the tape head alignment on the magnetic tape storage device is in compliance with the ANSI standards for nine track magnetic tapes. If the Contractor does not have documentation of alignment within the last 12 months, the Contractor must perform or have performed the manufacturer's documented head alignment procedure within 60 days of contract award. This is generally performed with a "skew" tape, certified to be in conformance with ANSI standards. The alignment must be performed by qualified personnel. The tape head alignment must be performed at a minimum once every 12 months or when there is evidence that the tape head may be out of alignment.

10.3.3.2 The tape system, including recording head, must be in conformance with the manufacturer's physical and electrical standards. Alignment of the remaining components of the tape system such as the retracting arms, must be performed at intervals not to exceed 24 months. If the Contractor cannot demonstrate that the remaining components of the tape system are in alignment, then the Contractor must perform or have performed the manufacturer's recommended alignment procedure.

10.3.4 Record of Maintenance of the Magnetic Tape Storage Device.

Documentation of maintenance, alignment, and repair procedures must be kept in an instrument maintenance log book for each tape device and data system. Also include any local area network components that provide a means for the transmission of data to or from the instrument data system and the tape system. Maintenance entries must include serial number, property number (if applicable), data and time of repair, name of person performing maintenance, problem description, problem resolution, date and time of failure (if applicable), and date and time placed back in service. Copies of repairs shall be kept in the maintenance documentation. Documentation of 1) data system, and 2) tape system maintenance and alignments, for the last 24 months must be made available upon written request of the Technical Project Officer or *Administrative Project Officer* or during a laboratory on-site evaluation. The Contractor shall always submit a GC/MS *and GC/EC* tape from a tape system in conformance with the manufacturer's physical and electrical standards and alignment according to manufacturer's procedures.

10.4 Corrective Actions. If the Contractor fails to adhere to the requirements listed in Section *10*, the Contractor may expect, but the Agency is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, a GC/MS *and/or*

GC/EC tape audit, a data package audit, a remedial laboratory evaluation sample, and/or contract sanctions.

11.0 DATA PACKAGE AUDITS

11.1 Overview. Data package audits are performed by the Agency for program overview and specific Regional concerns and to assess the technical quality of the data and evaluate overall Contractor performance. They provide the Agency with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. Data packages are periodically selected from recently received Cases. They are evaluated for the technical quality of hardcopy raw data, quality assurance, and adherence to contractual requirements. A thorough review of the raw data is completed, including: a check of instrument printouts, quantitation reports, chromatograms, spectra, library searches and other documentation for deviations from the contractual requirements, a check for transcription and calculation errors, a review of the qualifications of the Contractor personnel involved with the Case, and a review of *the latest version of all* SOPs on file. Standardized procedures have been established to assure uniformity of the auditing process.

11.2 Responding to the Data Package Audit Report. After completing the data package audit, the Agency may send a copy of the data package audit report to the Contractor or may discuss the data package audit report at an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer and the Administrative Project Officer, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report.

11.2.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer, why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the data package report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

11.2.2 If new SOPs are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Section 4.

11.3 Corrective Actions. If the Contractor fails to adhere to the requirements listed in Section *11*, the Contractor may expect, but the Agency is not limited to, the following actions: reduction in the numbers of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, a GC/MS *and/or GC/EC* tape audit, a data package audit, a remedial laboratory evaluation sample, and/or contract sanctions.

12.0 ON-SITE LABORATORY EVALUATIONS

12.1 Overview. At a frequency dictated by a Contractor's performance, the Administrative Project Officer, Technical Project Officer, or the Contracting Officer will conduct an on-site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: a quality assurance evaluation and an evidentiary audit.

12.2 Quality Assurance On-Site Evaluation. Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures.

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

- Size and appearance of the facility,
- Quantity, age, availability, scheduled maintenance, and performance of instrumentation,
- Availability, appropriateness, and utilization of the QAP and SOPs,
- Staff qualifications and experience, and personnel training programs,
- Reagents, standards, and sample storage facilities,
- Standard preparation logbooks and raw data,
- Bench sheets and analytical logbook maintenance and review, and
- Review of the Contractor's sample analysis/data package inspection/data management procedures.

12.2.2 Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous on-site reports, laboratory evaluation sample scores, Regional review of data, Regional QA materials, GC/MS and GC/EC tape audit reports, data audit reports, results of CCS, and data trend reports.

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

12.3.1 Procedural Audit. The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following Contractor operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

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

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

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

12.4 Discussion of the On-Site Team's Findings. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

12.5 Corrective Action Reports for Follow-Through to Quality Assurance and Evidentiary Audit Reports. Following an on-site laboratory evaluation, quality assurance and/or evidentiary audit reports which discuss deficiencies found during the on-site evaluation may be sent to the Contractor. In a detailed letter, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies discussed during the on-site evaluation and discussed in the report(s) to the Technical Project Officer and the Administrative Project Officer within 14 days of receipt of the report.

12.5.1 An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an

extension for greater than 14 days for the Contractor's response letter to the quality assurance and evidentiary audit report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or Administrative Project Officer.

12.5.2 If new SOPs are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Section 4.

12.6 Corrective Actions. If the Contractor fails to adhere to the requirements listed in Section *12*, the Contractor may expect, but the Agency is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, a GC/MS *and/or GC/EC* tape audit, a data package audit, a remedial laboratory evaluation sample, and/or contract sanctions.

13.0 QUALITY ASSURANCE AND DATA TREND ANALYSIS

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

- Surrogate spike recovery,
- Laboratory evaluation sample results,
- Blanks,
- GC/MS instrument performance checks (BFB and DFTPP),
- Initial and continuing calibration data, and
- Other QC and method parameters.

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

13.3 As a further benefit to the Program, the database provides the information needed to establish performance-based criteria in updated analytical protocols, where advisory criteria have been previously used. The vast empirical data set produced by Contractors is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of quality control and performance criteria specifications of what is routinely achievable and expected of environmental chemistry Contractors engaged in mass production analysis of environmental samples. This, in turn, assists the Agency in meeting its objectives of obtaining data of known and documented quality.

14.0 DATA MANAGEMENT

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

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

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

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

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

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

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

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

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

14.4.5 This documentation shall be available for on-site review and/or upon written request by the *Technical Project Officer* or *Administrative Project Officer*.

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

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

EXHIBIT F

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

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

Table of Contents

1.0 INTRODUCTION

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

1.2 The purposes of the evidence requirements include:

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

2.0 STANDARD OPERATING PROCEDURES

The Contractor shall implement the following standard operating procedures for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and complete sample delivery group file organization and assembly to ensure accountability of EPA sample chain-of-custody as well as control of all EPA sample-related records.

2.1 Sample Receiving

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

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

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

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

2.1.5 The sample custodian or his/her representative shall verify and record on Form DC-1 the presence or absence of the following documents accompanying the sample shipment:

- Custody seals,
- Chain-of-custody records,
- Traffic reports or packing lists,
- Airbills or airbill stickers, and
- Sample tags.

2.1.6 The sample custodian or his/her representative shall verify and record on Form DC-1 the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.

2.1.7 The sample custodian or his/her representative shall record the following information on Form DC-1 as samples are received and inspected:

- Custody seal numbers when present,
- Airbill or airbill sticker numbers,
- Sample tags listed/not listed on chain-of-custody records,
- *Cooler temperature,*

- Date of receipt,
- Time of receipt,
- EPA sample numbers,
- Sample tag numbers,
- Assigned laboratory numbers,
- Samples delivered by hand, and
- Problems and discrepancies.

2.1.8 The sample custodian or his/her representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (for example, chain-of-custody records, traffic reports or packing lists, and airbills). Note: Initials are not acceptable.

2.1.9 The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to, absent documents, conflicting information, absent or broken custody seals, *absent temperature indicator bottle,* and unsatisfactory sample condition (for example, leaking sample container).

2.1.10 The Contractor shall record resolution of problems and discrepancies by SMO.

2.2 Sample Identification

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

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

2.3 Sample Security

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

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

2.3.2 The Contractor shall demonstrate security of designated secure areas.

2.4 Sample Storage

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

2.5 Sample Tracking and Document Control

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

2.5.2 Titles which identify the activities recorded shall be printed on each page of all laboratory documents. (Activities include, but are not limited to, sample receipt, sample storage, sample preparation, and sample analysis.) When a document is a record of analysis, the instrument type and parameter group (for example, GC/MS-VOA) shall be included in the title.

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

2.5.4 Reviewers' signatures shall be identified on laboratory documents when reviews are conducted. Note: Individuals recording review comments on computer-generated raw data are not required to be identified unless the written comments address data validity.

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

2.5.6 Each laboratory document entry shall be dated with the month/day/year (for example, 01/01/90) and signed (or initialed) by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

2.5.7 Notations on laboratory documents shall be recorded in ink.

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

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

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

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

2.5.12 Logbook entries shall be in chronological order.

2.5.13 Logbook entries shall include only one Sample Delivery Group (SDG) per page, except in the events where the SDGs "share" QC samples (for example, instrument run logs and extraction logs).

2.5.14 Information inserted into laboratory documents shall be affixed permanently in place. The individual responsible for inserting

information shall sign and date across the insert and logbook page at the time information is inserted.

2.5.15 The Contractor shall document disposal or retention of EPA samples, remaining portions of samples, and prepared samples.

2.6 Computer-Resident Sample Data Control

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

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

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

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

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

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

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

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

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

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

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

2.7 Complete Sample Delivery Group File (CSF) Organization and Assembly

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

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

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

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

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

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

- logbook pages,
- benchsheets,
- mass spectra,
- chromatograms,
- screening records,
- preparation records,
- re-preparation records,
- analytical records,
- re-analysis records,
- records of failed or attempted analysis,
- custody records,
- sample tracking records,
- raw data summaries,
- computer printouts,
- correspondence,
- FAX originals,
- library search results, and
- other.

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

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

2.7.9 Original documents which include information relating to more than one SDG (for example, chain-of-custody records, traffic reports, calibration logs) shall be filed in the CSF of the lowest SDG number, and copies of these originals shall be placed in the other CSF(s). The document control officer or his/her representative shall record the following statement on the copies in dark ink:

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF _____

_____
Signature

_____
Date

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

2.7.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall

be recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

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

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

2.7.14 Shipments of deliverable packages, including resubmittals, shall be sealed with custody seals by the document control officer or his/her representative in a manner such that opening the packages would break the seals.

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

3.0 WRITTEN STANDARD OPERATING PROCEDURES (SOPS)

The Contractor shall develop and implement the following written SOPs for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and CSF file organization and assembly to ensure accountability for EPA sample chain-of-custody and control of all EPA sample-related records.

3.1 Sample Receiving

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

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

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

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

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

- Custody seals,
- Chain-of-custody records,
- Traffic reports or packing lists,
- Airbills or airbill stickers, and
- Sample tags.

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

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

- Custody seal numbers when present,
- Airbill or airbill sticker numbers,
- Sample tag numbers listed/not listed on chain-of-custody records,
- *Cooler temperature,*

- Date of receipt,
- Time of receipt,
- EPA sample numbers,
- Sample tag numbers,
- Assigned laboratory numbers,
- Samples delivered by hand, and
- Problems and discrepancies.

3.1.2.6 All accompanying forms are signed, dated, and the time is recorded, when applicable, at the time of sample receipt (for example, chain-of-custody records, traffic reports or packing lists, and airbills) by the sample custodian or his/her representative.

3.1.2.7 SMO is contacted to resolve problems and discrepancies including, but not limited to, absent documents, conflicting information, absent or broken custody seals, *absent temperature indicator bottle*, and unsatisfactory sample condition (for example, leaking sample container).

3.1.2.8 The resolution of problems and discrepancies by SMO is recorded.

3.2 Sample Identification

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

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

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

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

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

3.3 Sample Security

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

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

3.3.2.1 Procedures which ensure the following:

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

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

3.4 Sample Storage

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

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

3.5 Sample Tracking and Document Control

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

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

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

3.5.2.2 Procedures which ensure the following:

- All activities performed on EPA samples are recorded;
- Titles which identify the activities recorded are printed on each page of all laboratory documents;
- Information recorded in columns is identified with column headings;
- Reviewers' signatures are identified on laboratory documents;
- The laboratory name is included on preprinted laboratory documents;
- Laboratory document entries are signed and dated with the

month/day/year (for example, 01/01/90);

- Entries on all laboratory documents are recorded in ink;
- Corrections and additions to laboratory documents are made by drawing single lines through the errors, entering the correct information, and initialing and dating the new information;
- Unused portions of laboratory documents are lined-out;
- Pages in bound and unbound logbooks are sequentially numbered;
- Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
- Logbook entries are recorded in chronological order;
- Entries are recorded for only one SDG on a page, except in the events where SDGs "share" quality control (QC) samples (for example, instrument run logs and extraction logs);
- Information inserted in laboratory documents is affixed permanently, signed, and dated across the insert; and
- The retention or disposal of EPA samples, remaining portions of samples, and prepared samples is documented.

3.6 Computer-Resident Sample Data Control

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

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

3.6.2.1 Procedures which ensure the following:

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

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

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

3.7 CSF Organization and Assembly

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

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

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

EXHIBIT G

GLOSSARY OF TERMS

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

ANALYSIS DATE/TIME - the date and military time of the injection of the sample, standard, or blank into the GC/MS or GC system.

BAR GRAPH SPECTRUM - a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

BREAKDOWN - a measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses. It is also used in the VOA fraction as a system monitoring compound (SMC).

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

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

CHARACTERIZATION - a determination of the approximate concentration range of compounds of interest used to choose the appropriate analytical protocol.

CONCENTRATION LEVEL (low or medium) - characterization of soil samples or sample fractions as low concentration or medium concentration is made on the basis of the laboratory's preliminary screen, <u>not</u> on the basis of information entered on the Traffic Report by the sampler.

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

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

(CLASS) CONTRACT LABORATORY ANALYTICAL SERVICES SUPPORT - contract that operates the Sample Management Office (SMO) and is awarded and administered by the EPA.

DATE - MM/DD/YY - where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YY = 94, 95, 96, 97, etc.

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

DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) - compound chosen to establish mass spectral instrument performance for semivolatile analysis.

EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil), volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture (EC) detector. In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

GEL PERMEATION CHROMATOGRAPHY (GPC) - a size-exclusion chromatographic technique that is used as a cleanup procedure for removing large organic molecules, particularly naturally occurring macro-molecules such as lipids, polymers, viruses, etc.

IN-HOUSE - at the Contractor's facility.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

INTEGRATION SCAN RANGE - the scan number of the scan at the beginning of the area of integration to the scan number at the end of the area of integration. Performed in accordance with Exhibit D VOA, Sections 11.2.1.9 and 11.2.1.10 and Exhibit D SVOA, Sections 11.2.1.2 and 11.2.1.3.

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

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

INSTRUMENT BLANK - a blank designed to determine the level of contamination associated with the analytical instruments.

INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc. Exhibit D provides guidance for addressing this situation.

LABORATORY - synonymous with Contractor as used herein.

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

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

MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

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

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

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

PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

PERFORMANCE EVALUATION MIXTURE - a calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PRIMARY QUANTITATION ION - a contract specified ion used to quantitate a target analyte.

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

PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

PURGEABLES - volatile compounds.

QUALITY ASSURANCE TECHNICAL SUPPORT (QATS) LABORATORY - a contractor operated facility operated under the QATS contract, awarded and administered by the EPA.

REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

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

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A = area of the characteristic ion measured
C = concentration, or amount (mass)
is = internal standard
x = analyte of interest

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

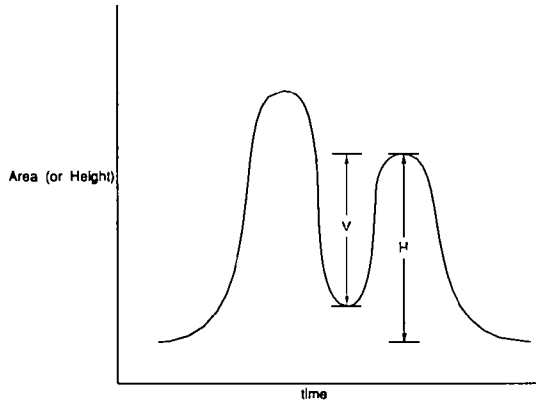
$$RRT = \frac{RT_c}{RT_{is}}$$

Where,

RT_c = Retention time for the semivolatile target or surrogate compound in continuing calibration.
RT_{is}= Retention time for the internal standard in calibration standard or in a sample.

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

RESOLUTION - also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.



For pesticide analysis the X-axis shall be displayed such that a data reviewer can calculate the % Resolution.

RESOLUTION CHECK MIXTURE - a solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

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

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

SAMPLE DELIVERY GROUP (SDG) - a unit within a single Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a Case, received over a period of up to 7 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- All samples within a Case; or
- Every set of 20 field samples *(excluding PE samples)* within a Case; or
- All samples received within 7 *calendar days, excluding Sundays and Government holidays. However, PE samples received within a Case shall be assigned to an SDG containing field samples for the Case.*

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

SAMPLE MANAGEMENT OFFICE (SMO) - a contractor operated facility operated by the CLASS contract, awarded and administered by the EPA.

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

SECONDARY QUANTITATION ION - contract specified ion(s) to be used in quantitation of target analytes when interferences prevent the use of the primary quantitation ion.

SEMIVOLATILE COMPOUNDS - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

SOIL - used herein synonymously with soil/sediment and sediment.

SONIC CELL DISRUPTOR (SONICATOR) - a device that uses the energy from controlled ultrasound applications to mix, disperse, and dissolve organic materials from a given matrix.

STANDARD ANALYSIS - an analytical determination made with known quantities of target compounds; used to determine response factors.

STORAGE BLANK - reagent water (two 40.0 mL aliquots) stored with samples in an SDG. It is analyzed after all samples in that SDG have been analyzed; and is used to determine the level of contamination acquired during storage.

SULFUR BLANK - a modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, then the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur blank is required.

SURROGATES (Surrogate Standard) - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

SYSTEM MONITORING COMPOUNDS - compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard for volatile analysis, and used to evaluate the performance of the entire purge and trap-gas chromatograph-mass spectrometer system. These compounds are brominated or deuterated compounds not expected to be detected in environmental media.

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

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. *TICs must have peak areas or heights* greater than 10% of the peak areas or heights of nearest internal standard. *TICs must be* subjected to mass spectral library searches *and be deemed acceptable by a mass spectral interpretation specialist*.

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

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

TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the twelve hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

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

VOLATILE COMPOUNDS - compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

WIDE BORE CAPILLARY COLUMN - a gas chromatographic column with an internal diameter (ID) that is greater than or equal to 0.53 mm. Columns with lesser diameters are classified as narrow bore capillary columns.

EXHIBIT H

AGENCY STANDARD IMPLEMENTATION

Exhibit H - Agency Standard Implementation

Table of Contents

1.0 FORMAT CHARACTERISTICS

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

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

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

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

1.5 The CLP is currently developing a data delivery strategy that may be

used as an alternative to the requirements stated in Exhibit H. This strategy's intent is to provide a neutral data delivery structure to the Contractor that will further facilitate the exchange of analytical information generated under this analytical protocol. The proposed strategy is intended to accommodate laboratories that generate data transmission files under multiple data formats. Upon implementation of this alternate electronic data delivery strategy by the CLP and prior to submission of data in alternate format(s), the Contractor must first demonstrate its ability to provide electronic data as stated in this Exhibit H and obtain written permission from the CLP for the submission of data in alternate format(s). The Contractor will receive a written response to its request within 90 calendar days. However, until the implementation of this alternate electronic data delivery strategy by the CLP, all electronic data deliverables must be provided as specified in this Exhibit H.

2.0 RECORD TYPES

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

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

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

2.3 A separate run header is used for volatiles (VOA), semivolatiles (SV), and for each column analysis for pesticides (PEST) (minimum of four type 10 series for VOA/SV/PEST SDG). The 20 series records contain sample characteristics and link samples within an SDG to the corresponding calibrations, blanks, and other QCs. The 30 series records contain the actual analytical results by analyte within each sample. The 10, 20, and 30 records are associated with each other by their position in the file (i.e., 30 series records follow the corresponding 20 series, which in turn follow the 10 series run header records).

3.0 PRODUCTION RUNS

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

3.1.1 Calibration - All samples in a run use the same initial calibration data.

3.1.2 Method number - Constant throughout a run.

3.1.3 Instrument conditions - Constant throughout a run.

3.2 Each instrumental analysis consists of a separate production run and is reported in a separate file. There will be a separate production run for each of the two pesticide GC columns utilized. Thus, a full three fraction analysis will consist of a minimum of four production runs.

3.3 Example of the Sequence of Record Types in a File[1]

```
10              Contains Run Header information.
    11          Contains additional run-wide information.
    20          Occurs once for each sample, calibration, mean response
                factor, matrix spike duplicate result, etc.  Acts as a
                header.
    21
    22          Contains additional information for samples.
    23
    27
        30      Occurs once for each final analytical result.  Reports
                the value being determined as defined by the type 20.
            32  Reports any auxiliary data necessary.
            33  Reports compound names for tentatively identified
                compounds (TICs) if necessary.
            36  Reports any instrumental data necessary.
        30      Values for the next analyte or parameter being measured.
            32  Additional data may vary for each parameter, and may
            33  occur in any order.  Multiple occurrences of the same
            36  record type, however, must be consecutive.
        30      Continues for as many as are necessary.
            32
            33
            36
        30
            32
            33
            36
    20          Next Sample Header record.  The following applies to the
    21          next sample or other group of data.
    22
        30
            32
            33
            36
        30
            32
            33
            36
                etc.
    20
    21
        30
            32
            33
            36
                etc.
```

---

1 Appendix A provides a detailed set of examples for the use of the different record types, and their relationship to other record types.

4.0 RECORD SEQUENCE

4.1 The sequence of records for Agency Standard files is as follows: A Run Header (type 10) record shall be present once and once only (per file) as the first record in a file. Therefore, a complete VOA/SV/PEST SDG will consist of several files.

4.2 Each environmental sample, calibration standard, or quality control sample is represented by a group composed of type 20, 21, 22, 23, and 27 records, that hold sample level identifying information, followed by type 30, 32, 33, and 36 records for each method analyte including surrogates, system monitoring compounds, and internal standards in the sample. The type 20 record holds a count for the number of method analytes being determined and includes all target compounds, surrogates, system monitoring compounds, and internal standards plus each peak of the multi-component pesticides (do not include TICs in this count). A separate field on the type 23 record contains the number of TICs found. Type 20 records shall occur in the order of sample analysis. In addition, a type 20 record with a QC code "MNC", followed by a type 30 record for each method analyte (reporting values such as mean response factors) will appear after the type 10 or type 11 record and before the type 20 record that initiates the analytical sequence. Similarly, for pesticide runs, a type 20 record with a QC code "GPC" for GPC recovery, followed by type 30 records for each of the method analytes spiked; and a type 20 record with a QC code "FLO" for Florisil recovery, followed by type 30 records for each of the method analytes (and the two surrogates) included in the Florisil check will appear before the type 20 record that initiates the analytical sequence.

4.3 Type 90 comment records may be defined to occupy any position after the type 10 (header) record.

5.0 FILE/RECORD INTEGRITY

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

Record Position	Field Length	Field Contents	Remarks
First Field	2	Record type	"10" or as appropriate
Last Field	5	Record sequence number	00001-99999, numbered within file sequentially
	4	Record checksum[1]	Four hexadecimal digits
	2	Must contain CR and LF	

6.0 DATES AND TIMES

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

7.0 MULTIPLE VOLUME DATA

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

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

8.0 DELIVERABLE

8.1 The file shall be submitted on IBM-compatible, 3.5 inch high density 1.44 M-byte diskettes. The diskettes shall be formatted and recorded using MS-DOS Operating System. The diskettes shall contain all information relevant to one and only one SDG. *An alternative means of electronic transmission may be utilized if approved in advance by the EPA.*

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

8.3 Information on the diskette **must correspond** to information submitted in the hardcopy raw data package and on the hardcopy raw data package forms. For example, type 30 results field specifies maximum length of 13. When reporting CRQLs or results on Form 1, maximum length is 13 as is specified in this exhibit; when reporting 'calculated amounts' on Form 7D, hardcopy specified maximum length is 8. Unused records shall not be included on the diskettes. If the information submitted in the hardcopy data package forms is changed, the information in the *electronic file (e.g., diskette)* shall be changed accordingly, and a complete *electronic deliverable* containing all the information for the SDG shall be resubmitted along with the hardcopy at no additional cost to the EPA.

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

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

8.5 The format for File Name shall be XXXXX.O01 to XXXXX.O99. Where XXXXX is the SDG identifier, O designates Organics, and 01 through 99 is the file number.

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

8.7 Section 9.0 (Record Listing) provides information for the usage of each of the record types. Where specified, labels indicate the nature of the value(s) that follow on that record. If the value(s) will not be reported, the label shall be omitted.

8.7.1 A record type 30 for each TCL compound, surrogate, system monitoring compound, and internal standard quantitated for shall be reported. If the TCL is not detected, the 'U' qualifier in the appropriate field shall be indicative of that.

8.7.2 For multicomponent analytes (Aroclors/toxaphene), if the multicomponent analyte is detected, a record type 30 and 32 shall be reported for each peak identified.

9.0 RECORD LISTING

The following lists every record type required to report data from a single SDG.

9.1 Production Run Header Record (Type 10)

Use: Each production run will start with a record type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"10"
6	Delimiters	||||||
5	INSTRUMENT/DETECTOR	Character[1]
1	Delimiter	|
8	METHOD NUMBER	Character[2]
2	Delimiters	||
6	LAB CODE	Character
4	Delimiters	||||
11	CONTRACT NUMBER	Character
1	Delimiter	|
10	INSTRUMENT ID	Character
2	Delimiters	||
25	LABORATORY NAME	Character
2	Delimiters	||
5	RECORD SEQUENCE NUMBER	Numeric
4	CHECKSUM	Character

1 General descriptor (GC/MS for VOA/SVOA analysis or GC for pesticide analysis on GC/EC).

2 *OLM04.1V* For Volatiles; *OLM04.1B* for semivolatiles; *OLM04.1P* for pesticides. (O for Organic, L for Low, M for Medium, zero *four* for document number, zero V for volatiles, zero B for semivolatiles, zero P for pesticides.)

9.2 Chromatography Record (Type 11)

Use: To describe chromatograph condition. Must be present once for each production run immediately following the record type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"11"
1	Delimiter	|
10	GC COLUMN IDENTIFICATION	Character
2	Delimiters	||
4	GC COLUMN ID[1]	Numeric (mm)
11	Delimiters	|||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

1 Internal Diameter of the GC column used.

9.3 Sample Header Data Record (Type 20)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"20"
2	Delimiters	||
12	EPA SAMPLE NUMBER	As is exactly on the hardcopy form
1	Delimiter	|
1	MATRIX	CHARACTER[1]
1	Delimiter	|
3	QC CODE	Character (See Section 10)
1	Delimiter	|
3	SAMPLE QUALIFIER	RIN/REX/REJ/SRN/blank[2]
1	Delimiter	|
5	CASE NUMBER	Numeric
1	Delimiter	|
6	SDG NO.	Character
1	Delimiter	|
4	SAMPLE/BLANK/STANDARDS YEAR ANALYZED	YYYY
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS MONTH ANALYZED	MM
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS DAY ANALYZED	DD
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS HOUR ANALYZED	HH
1	Delimiter	|
2	SAMPLE/BLANK/STANDARDS MINUTE ANALYZED	MM
2	Delimiters	||
2	SAMPLE WT/VOL UNITS	"G"/"ML"/blank[3]
1	Delimiter	|
5	SAMPLE WT/VOL	Numeric[4]

1 "0" if not applicable (calibration, tune, etc.); "1" for water; "H" for soil.

2 "RIN" for reinjection; "REX" for re-extractions; "REJ" for rejected samples; "SRN" for dilutions; and leave blank (empty field with zero length) when none of the previous conditions apply. In case of multiple operations on a sample, the final operation will be indicated (e.g., reinjection of a dilution; AAA12DLRE would have a QC Code of "RIN").

3 Sample WT/VOL unit is mL (milliliters) for liquids and G (grams) for solids. The sample units code indicates which units are in use for the current sample. Leave blank (zero length) if not applicable.

4 Sample WT/VOL is the volume in milliliters for liquid or the wet weight in grams for solids. Sample WT/VOL includes the purge volume.

Sample Header Data Record (Type 20) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
3	ANALYTE COUNT	Numeric[5]
3	Delimiters	|||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

5 1-3 decimal digits. Counts TCL analytes, surrogates, system monitoring compounds (SMC), internal standards, and all peaks reported for multi-component PCBs. Do not include the count for TICs in this field. For calibrations, also count DFTPP, if included in calibration solution.

9.4 Sample Header Data Record (Type 21)

Use: Continuation of Type 20.
Position: Follows the Type 20 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"21"
1	Delimiter	|
1	PURGE	"N" for not heated; "Y" for heated; blank if SV or PEST
1	Delimiter	|
1	LEVEL	"L"/"M"/blank[1]
2	Delimiters	||
1	EXTRACTION	S/C/*H*/N/*X*/*P*/*T*/blank (for *all other* volatile *samples*)[2]
2	Delimiters	||
6	SAS NUMBER	Character
1	Delimiter	|
14	LAB FILE/SAMPLE ID	Character[3]
1	Delimiter	|
4	YEAR EXTRACTED	*YYYY*/blank (for volatiles)
1	Delimiter	|
2	MONTH EXTRACTED	MM/blank (for volatiles)
1	Delimiter	|
2	DAY EXTRACTED	DD/blank (for volatiles)
2	Delimiters	||
4	YEAR RECEIVED	*YYYY*/blank (for standards, tunes, and blanks)
1	Delimiter	|
2	MONTH RECEIVED	MM/blank (for standards, tunes, and blanks)
1	Delimiter	|
2	DAY RECEIVED	DD/blank (for standards, tunes, and blanks)
2	Delimiters	||

---

1 "L" for low level samples and "M" for medium level samples for volatile and semivolatile analyses. Leave blank for pesticides, all calibrations, and all tunes.

2 "S" for separatory funnel; "C" for continuous liq-liq *without hydrophobic membrane; "H" for continuous liq-liq with hydrophobic membrane*; "N" for sonication; *"X" for automated soxhlet; "P" for pressurized fluid; "T" for volatile low level soils by the Modified SW-846 Method 5035;* blank (zero length field) for *all other* volatile *samples*.

3 Lab File ID for volatile and semivolatile analyses. Lab Sample ID for pesticides in same format as on forms.

Sample Header Data Record (Type 21) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
8	INJECTION/ALIQUOT VOLUME	Numeric/blank (for low level VOA)[4]
2	Delimiters	||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

4 Injection volume, in uL, for SVOAs and PESTs; Soil Aliquot Volume for medium level VOA.

9.5 Sample Condition Record (Type 22)

Use: Continuation of type 20. Used to describe additional Sample Conditions.
Position: Follows the type 20 and 21 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"22"
1	Delimiter	|
4	CALIBRATION YEAR	YYYY/blank (for PEST)[1]
1	Delimiter	|
2	CALIBRATION MONTH	MM/blank (for PEST)
1	Delimiter	|
2	CALIBRATION DAY	DD/blank (for PEST)
1	Delimiter	|
2	CALIBRATION HOUR	HH/blank (for PEST)
1	Delimiter	|
2	CALIBRATION MINUTE	MM/blank (for PEST)
1	Delimiter	|
14	CALIBRATION FILE ID	Character/blank (for PEST)[2]
1	Delimiter	|
4	PH	Numeric/blank (for aqueous samples and volatiles)
1	Delimiter	|
5	PERCENT MOISTURE	Numeric
1	Delimiter	|
1	DECANTED	"Y"/"N"/blank (for volatiles)
1	Delimiter	|
8	EXTRACT VOLUME	Numeric/blank (for low level VOA)[3]
1	Delimiter	|
8	DILUTION FACTOR	Numeric[4]
3	Delimiters	|||

---

1 For volatiles and semivolatiles, enter the date and time of analysis of the most recent 50 ug/L (VOAs) or the 50 ng (SVOAs) standard run prior to the sample reported in the associated type 20 record. Leave blank for pesticides.

2 Lab File ID of standard specified in 1 above (volatiles/semivolatiles only). This field must match the Lab File ID on Type 21 for the associated calibration (VSTD050/SSTD050). Leave blank for pesticides.

3 Enter the Soil Extract Volume for medium level VOA, and Concentrated Extract Volume for all SVOA and PEST. The value should be reported in microliters.

4 Dilution factor of sample analyzed (omit contract-mandated dilutions).

Sample Condition Record (Type 22) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
5	LEVEL	Numeric/blank (for VOA/SV)[5]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

5 Concentration level of Pesticide Individual Mix A and B standards. Concentration of low point, mid point and high point calibration standards as a multiplier of low point. Low point = 1.0; Mid point = 4.0; High point ≥ 16.0.

9.6 Associated Injection and Counter Record (Type 23)

Use: Continuation of type 20. Used to identify associated blanks and tunes, and the number of surrogates/SMCs and spikes outside of the QC limits and the number of TICs.

Position: Follows the type 20, 21, and 22 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"23"
1	Delimiter	|
1	INSTRUMENT PERFORMANCE CHECK (IPC/TUNE) LABEL	"P" (for BFB and DFTPP IPC) or blank (for pesticides)
1	Delimiter	|
4	IPC/TUNE INJECTION YEAR	YYYY/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION MONTH	MM/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION DAY	DD/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION HOUR	HH/blank (for PEST)
1	Delimiter	|
2	IPC/TUNE INJECTION MINUTE	MM/blank (for PEST)
1	Delimiter	|
14	DFTPP/BFB LAB FILE ID	Character/blank (for PEST)
1	Delimiter	|
2	VOLATILE STORAGE BLANK LABEL	"HB" (for VOA) or blank (for SV and PEST)
1	Delimiter	|
4	STORAGE BLANK INJECTION YEAR	YYYY/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION MONTH	MM/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION DAY	DD/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION HOUR	HH/blank (for SV and PEST)
1	Delimiter	|
2	STORAGE BLANK INJECTION MINUTE	MM/blank (for SV and PEST)
1	Delimiter	|
14	STORAGE BLANK LAB FILE ID (VOA ONLY)	Character
4	Delimiters	||||

Associated Injection and Counter Record (Type 23) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	METHOD BLANK LABEL	"MB"/blank (for standard, tune, and method blanks)
1	Delimiter	|
4	METHOD BLANK INJECTION YEAR	YYYY/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION MONTH	MM/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION DAY	DD/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION HOUR	HH/blank (for standard, tune, and method blanks)
1	Delimiter	|
2	METHOD BLANK INJECTION MINUTES	MM/blank (for standard, tune, and method blanks)
1	Delimiter	|
14	METHOD BLANK LAB FILE (for VOA and SV)/SAMPLE ID (for PEST)	CHARACTER
1	Delimiter	|
1	SURROGATE (for SV and PEST)/SMC (for VOA) RECOVERY LABEL	"P" for % recoveries/blank (for STD/IPC)
1	Delimiter	|
2	SURROGATE (for SV and PEST)/SMC (for VOA) RECOVERIES OUT	Numeric[1]
1	Delimiter	|
1	TIC LABEL	"T" (for VOA and SV TICs)/blank (for PEST)
1	Delimiter	|
2	NO. OF TICS	Numeric
1	Delimiter	|
1	SPIKE RECOVERY LABEL	"S" for Matrix Spikes and Matrix Spike Duplicates/blank for anything else
1	Delimiter	|

1 This will be the number of surrogate (for SV or PEST) or SMC (for VOA) recoveries outside QC limits for a specific column. It should not be cumulative of the two columns for pesticides.

Associated Injection and Counter Record (Type 23) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	SPIKE RECOVERIES OUT	Numeric/blank[2]
1	Delimiter	|
1	RPD LABEL	"R" for RPD/blank[3]
1	Delimiter	|
2	RPD OUT	Numeric
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

2 Enter the number of spike recoveries out. Enter "0"(zero) if none of the spike recoveries are outside of the QC limit.

3 "R" for Matrix Spike/Matrix Spike Duplicate Recovery Relative Percent Differences. Leave blank for all other samples (only report for MS/MSD)

9.7 Sample Cleanup Record (Type 27)

Use: Continuation of type 20. Used to identify sample/blank cleanup procedures and QC results.
Position: Follows type 20, 21, 22, and 23 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"27"
1	Delimiter	|
1	FIRST CLEANUP TYPE	"G" for GPC/blank (for VOA)[1]
1	Delimiter	|
4	GPC CALIBRATION CHECK YEAR	YYYY/blank (for VOA)
1	Delimiter	|
2	GPC CALIBRATION CHECK MONTH	MM/blank (for VOA)
1	Delimiter	|
2	GPC CALIBRATION CHECK DAY	DD/blank (for VOA)
1	Delimiter	|
2	GPC CALIBRATION CHECK HOUR	HH/blank (for VOA)
1	Delimiter	|
2	GPC CALIBRATION CHECK MINUTE	MM/blank (for VOA)
1	Delimiter	|
14	GPC Data Descriptor	Character/blank (for VOA and SV)[2]
1	Delimiter	|
1	FLORISIL CLEANUP TYPE	"F" (for PEST) or blank (for VOA and SV)
1	Delimiter	|
4	FLORISIL LOT CHECK YEAR	YYYY/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK MONTH	MM/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK DAY	DD/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK HOUR	HH/blank (for VOA and SV)
1	Delimiter	|
2	FLORISIL LOT CHECK MINUTE	MM/blank (for VOA and SV)
1	Delimiter	|
14	FLORISIL DATA DESCRIPTOR	Character[3]

1 "G" indicates that GPC was performed. If GPC was not performed, leave the field blank.

2 Lab Sample ID of associated GPC. This is a unique identifier assigned to the spike recovery results for a specific GPC calibration check for pesticides. Leave blank for volatiles and semivolatiles.

3 Lab Sample ID of associate Florisil lot check. This is a unique identifier assigned to a lot of Florisil cartridges.

Sample Cleanup Record (Type 27) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
1	SULFUR CLEANUP	Y/N (for PEST)/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK LABEL	"SB"/blank (if no separate sulfur blank was prepared for pesticides; also blank for VOA and SV)
1	Delimiter	|
4	SULFUR BLANK INJECTION YEAR	YYYY/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION MONTH	MM/blank (for VOA and SV)
2	Delimiters	||
2	SULFUR BLANK INJECTION DAY	DD/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION HOUR	HH/blank (for VOA and SV)
1	Delimiter	|
2	SULFUR BLANK INJECTION MINUTE	MM/blank (for VOA and SV)
1	Delimiter	|
14	SULFUR BLANK LABORATORY/ SAMPLE ID	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.8 Results Data Record (Type 30)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"30"
1	Delimiter	|
1	ANALYTE LABEL	"C" for CAS Number (blank for unknown TICs)
1	Delimiter	|
9	CAS NUMBER	Numeric (for TCL, surrogates, DFTPP, BFB, SMC, internal standards, and identified TICs)
1	Delimiter	|
9	INTERNAL STD. CAS NUMBER	Numeric
1	Delimiter	|
5	CONCENTRATION UNITS	Character "ug/L" (aqueous); "ug/Kg" (soil); "ng" (amount added)
1	Delimiter	|
3	RESULT QUALIFIER	Character[1,2]
1	Delimiter	|
13	RESULTS	Numeric[3]
1	Delimiter	|
5	FLAGS	Character[4]
1	Delimiter	|
1	AMOUNT ADDED LABEL	"A" for Amt. added[5]
1	Delimiter	|
13	AMOUNT ADDED	Numeric
1	Delimiter	|

1 When a Type 20 Record is used for calibration summary (MNC), the associated Type 30 Record uses "AVG" for average RRFs and Mean Calibration Factors. See Exhibit H Section 10.3.2.

2 For pesticide sample analysis, if an analyte is detected in only one of the two column analyses, report the analyte as "not detected" in both runs. Report result qualifier, for each column, as BDL. See Section 10.3.2 for result qualifiers.

3 Leave this field blank *only when* reporting non-detects.

4 A maximum of five flags (D,E,J,B,A,P,C,X,Y,Z, or N) with no space between the flags can be reported, each representing a qualification of the result as described in Exhibit B. For surrogates, the "D" flag will indicate surrogates diluted out.

5 For Matrix Spike/Matrix Spike Duplicate analysis, surrogate, SMC for VOA, SV, and PEST (Form 3s). Nominal Amount for Pesticides (Form *7E/7F*). Spike added for florisil and GPC (Form 9A/9B).

Results Data Record (Type 30) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	CRQL LABEL	"U" for "undetected" or blank when analyte is detected
1	Delimiter	|
13	CRQL	Numeric
1	Delimiter	|
1	RSD LABEL	"R" for % Resolution/RSD[6]
1	Delimiter	|
5	RSD VALUE	Numeric
1	Delimiter	|
1	MS/MSD REC LABEL	"P" for % recovery [MS/MSD]/blank (for sample [except MS/MSD] standard, tune, blanks, calibration)
1	Delimiter	|
5	MS % RECOVERY	Numeric/blank (for everything except MS)
1	Delimiter	|
5	MSD % RECOVERY	Numeric/blank (for everything except MSD)
1	Delimiter	|
1	RPD LABEL	"D" for MS/MSD or for pesticide calibration verification (%D)/blank
1	Delimiter	|
5	RPD VALUE	Numeric/blank[7]
1	Delimiter	|
1	SURR/SPIKE RECOVERY LABEL	"S" for % recovery/blank (for non-surrogate/SMC and non-spike analytes
1	Delimiter	|
5	SURR/SPIKE RECOVERY	% Recovery/blank[8]

6 "R" for % Resolution (Forms *6H, 6I, 6J, and 6K*) or for RSD of Response factors under Calibration summary (MNC) Type 20. (Blank for VOA and SV fractions.)

7 RPD for MS/MSD recoveries, or %D for pesticides. Calibration Verification (Form *7E/7F*). Otherwise, leave blank.

8 Surrogate (for SV and PEST)/SMC (for VOA) or Spike (Forms 2, Form 9A/9B) recovery. Leave blank for non-surrogate and non-spike analytes.

Results Data Record (Type 30) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
1	MEAN CONCENTRATION LABEL	"M" for Mean conc. (for multicomponent PEST only)/blank (for VOA and SV)
1	Delimiter	|
13	MEAN CONCENTRATION	Numeric (for PEST)/blank (for VOA and SV)[9]
1	Delimiter	|
1	PERCENT DIFFERENCE LABEL	"F" or "P" (PEST)/blank (for VOA and SV field sample analysis)[10]
1	Delimiter	|
5	PERCENT DIFFERENCE	Numeric
1	Delimiter	|
1	INTERNAL STANDARD AREA LABEL	"I" for IS Area (for VOA and SV)/blank (for PEST)
1	Delimiter	|
13	INTERNAL STANDARD AREA	Numeric (for VOA and SV)/blank (for PEST)
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9 Mean Concentration for Multicomponent analytes detected in pesticide analyses.

10 "P" for Percent Difference between concentrations from two columns in pesticide analyses, or "F" for Percent Difference between average RRF (initial calibration) and RRF50 (continuing calibration) in VOA/SVOA analyses. Leave blank for volatile and semivolatile sample, blank, and tune analysis.

9.9 Auxiliary Data Record (Type 32)

Use: Used to report retention time (in minutes) for Internal Standards and for TICs (for Volatiles and Semivolatiles). Used to report retention time data and percent breakdown (for pesticides).

Position: Follows type 30. (Record will only be required as specified above.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"32"
3	Delimiters	|||
2	RETENTION TIME LABEL	"RT"
1	Delimiter	|
5	RETENTION TIME	Numeric
1	Delimiter	|
3	FIRST LIMIT LABEL	"RTF"
1	Delimiter	|
5	RT WINDOW LOWER LIMIT	Numeric
1	Delimiter	|
3	SECOND LIMIT LABEL	"RTT"
1	Delimiter	|
5	RT WINDOW UPPER LIMIT	Numeric
2	Delimiters	||
2	% BREAKDOWN LABEL	"PB" for % breakdown/blank (for VOA and SV)
1	Delimiter	|
5	% BREAKDOWN	Numeric (DDT/ENDRIN)/blank (for VOA and SV)
1	Delimiter	|
5	COMBINED % BREAKDOWN	Numeric/blank (for VOA and SV)[1]
2	Delimiters	||
1	PEAK	1 THROUGH 5 (for pesticide multicomponent compounds)/blank (for VOA and SV)[2]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

---

1 The combined %breakdown will be reported on both the record type 32s (for DDT and Endrin).

2 For positively identified compounds, a minimum of 3 peaks and a maximum of 5 peaks are allowed. Types 30 and 32 will be repeated for each peak that is reported (a minimum of three, a maximum of five times). This is for multicomponent analytes in pesticide analyses.

9.10 Name Record (Type 33)

Use: This record type is used for volatile and semivolatile analyses only to carry an analyte name for TICs. This record is not used for pesticide analysis.

Position: Follows types 30 and 32 for TICs.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"33"
1	Delimiter	|
67	NAME OF COMPOUND	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.11 Instrumental Data Readout Record (Type 36)

Use: This record type is only used for volatile and semivolatile analyses to describe DFTPP/BFB percent abundances. This record is not used for pesticide analysis.

Position: Follows type 30 for DFTPP/BFB data.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"36"
1	Delimiter	|
1	MASS LABEL	"M"
3	Delimiters	|||
3	FIRST MASS (DFTPP/BFB)	Numeric (DFTPP for SV or BFB for VOA)
2	Delimiters	||
5	FIRST PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
3	SECOND MASS	Numeric
1	Delimiter	|
5	SECOND PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 69	Numeric, DFTPP only/blank (for VOA)
1	Delimiter	|
3	THIRD MASS	Numeric
1	Delimiter	|
5	THIRD PERCENT RELATIVE ABUNDANCE	Numeric
2	Delimiters	||
3	FOURTH MASS	Numeric
1	Delimiter	|
5	FOURTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 69	Numeric, DFTPP only/blank (for VOA)
1	Delimiter	|
3	FIFTH MASS	Numeric
1	Delimiter	|
5	FIFTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	SIXTH MASS	Numeric
1	Delimiter	|

Instrumental Data Readout Record (Type 36) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
5	SIXTH PERCENT RELATIVE ABUNDANCE	Numeric
2	Delimiters	||
3	SEVENTH MASS	Numeric
1	Delimiter	|
5	SEVENTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	EIGHTH MASS	Numeric
1	Delimiter	|
5	EIGHTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 174	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	NINTH MASS	Numeric
1	Delimiter	|
5	NINTH PERCENT RELATIVE ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS OF 176	Numeric, BFB only/blank (for SV)
1	Delimiter	|
3	TENTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	TENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	ELEVENTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	ELEVENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	TWELFTH MASS	Numeric/blank (for VOA)
1	Delimiter	|
5	TWELFTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
2	Delimiters	||
3	THIRTEENTH MASS	Numeric/blank (for VOA)
2	Delimiters	||

Instrumental Data Readout Record (Type 36) (Cont.)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
5	THIRTEENTH PERCENT RELATIVE ABUNDANCE	Numeric/blank (for VOA)
1	Delimiter	|
5	PERCENT MASS OF 442	Numeric, DFTPP only (blank for VOA)
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

9.12 Comment Record (Type 90)

Use: To provide for operator-entered comments.
Position: May occur anywhere in the file after the type 10 record.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"90"
1	Delimiter	|
67	ANY COMMENT	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

10.0 DEFINITIONS OF VARIOUS CODES USED IN AGENCY STANDARD RECORDS

10.1 Quality Control and Related Codes (QCC) in Type 20 Records

10.1.1 Note: These codes appear in the QC code fields of type 20 records. They are used to indicate the type of data that is being reported.

QCC	Name	Definition
LRB	LABORATORY (REAGENT) BLANK	The "Method Blank" (see Exhibit G).
LIB	LABORATORY INSTRUMENT BLANK	The "Instrument Blank".
LSB	LABORATORY SULFUR BLANK	If different from "Method Blank" (pesticides).
LHB	LABORATORY STORAGE BLANK	The storage blank (volatiles).
FRB	*FIELD BLANK*	*This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.*
FRM	*FIELD REFERENCE SAMPLE*	*This is any sample that is submitted for a Case and is identified as a Performance Evaluation (PE) sample.*
LSD	LABORATORY SPIKE DUPLICATE BACKGROUND (ORIGINAL) VALUES	An environmental sample which is analyzed according to the analytical method, and subsequently used for the matrix spike and the matrix spike duplicate (see Exhibit G).
LF1	LABORATORY SPIKED SAMPLE - FINAL - FIRST MEMBER	The "Matrix Spike" (see Exhibit G); must precede LF2.
LF2	LABORATORY SPIKED SAMPLE - FINAL - SECOND MEMBER	The "Matrix Spike Duplicate" (see Exhibit G).
LPC	LABORATORY PERFORMANCE CHECK SOLUTION	A solution of DFTPP (SVOA) or BFB (VOA) or method analytes (PEST/PCB) used to evaluate the performance of an instrument with respect to a defined set of criteria (Tune or Resolution Check Sample) (see Exhibit G).
FLO	FLORISIL CHECK SOLUTION	A solution of pesticides used to check recovery from each lot of Florisil cartridges. These recovery results will be provided in every production run where associated samples are analyzed.

GPC	GPC CHECK SOLUTION	A solution of pesticides used to check recovery from each new GPC calibration. These recovery results will be provided in every production run where associated samples are analyzed.
CLM	INITIAL CALIBRATION MULTI-POINT	The Initial Calibration for GC/MS (see Exhibit G), or the Initial Individual Standard Mixes (A, B) for pesticides (see Exhibit D PEST). Response factors (GC/MS) or Calibration Factors (pesticides) will be reported on the following type 30 records.
CLS	INITIAL CALIBRATION SINGLE POINT	The Initial Toxaphene/Aroclor Mixes used to determine all calibration factors (see Exhibit D PEST).
CLC	CONTINUING CALIBRATION CHECK	The continuing calibration (*VSTD050/SSTD050*) for GC/MS.
CLE	CONTINUING PERFORMANCE CHECK	The subsequent Individual Standard Mixes (A,B), Performance Evaluation Mixture, and for subsequent injections of Toxaphene/Aroclor mixes for pesticides (see Exhibit D PEST).
CLD	DUAL PURPOSE CALIBRATION	A calibration solution as above used both as an initial calibration (CLM) and a continuing check (CLC). (50 level initial calibration if needed for Form 8.)

10.1.2 The following QCC values are used on type 20 records which act as a header, and indicate that additional (usually calculated) analyte specific data will be present on type 30 (and following type) records. Usually, these data will apply to an entire production run, in which case they will appear immediately following the type 10 record or type 11 record if present. If the data apply to only a portion of the samples in the run, they shall be placed immediately preceding the samples to which they apply. Much of the rest of the information in the type 20 record may be blank, indicating that these data do not apply to these results.

MNC	MEAN VALUES FROM CALIBRATIONS	The data following represent mean values and percent RSDs from the initial calibration (GC/MS) or the mean calibration factors, mean retention times and retention time windows (pesticides).

10.2 Codes For Sample Medium (Matrix, Sources)

Medium	Code
All Media, Specific Medium not Applicable. Use for Calibrations, Tunes, etc.	0 (zero)
Water	1
Soil	H

10.3 List of Sample and Result Qualifiers

Definition: A sample qualifier consists of three characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, or (b) produced a numeric result for an entire sample but it is qualified in some respect relating to the type or validity of the result.

10.3.1 Sample Qualifiers

Qualifier	Full Name	Definition
RIN	RE-ANALYZED	The indicated analysis results were generated from a re-injection of the same sample extract or aliquot (RE SUFFIX).
REX	RE-PREPARED	The indicated analysis results were generated from a re-extraction of the same sample (RE SUFFIX).
REJ	REJECTED	The results for the entire sample analysis have been rejected for an unspecified reason by the laboratory. For initial calibration data, these data were not utilized in the calculation of the mean.
SRN	DILUTED	The indicated analysis results were generated from a dilution of the same sample (DL SUFFIX).

10.3.2 Result Qualifiers in Type 30 Records

A result qualifier consists of three characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, or (b) produced a numeric result for a single analyte but it is qualified in some respect relating to the type or validity of the result. This qualifier is complementary to the flags field on a type 30 record. A TIC **must** have either a TIE, TFB, ALC, or PRE result qualifier.

BDL	BELOW DETECTABLE LIMITS	Indicates compound was analyzed for but not detected (Form 1 "U" Flag).
NAR	NO ANALYSIS RESULT	There is no analysis result required for this subject parameter.

AVG	AVERAGE VALUE	Average value -- used to report a range of values (e.g., relative response factors).
CBC	CANNOT BE CALCULATED	The analysis result cannot be calculated because an operand value is qualified (e.g., identifies analytes whose internal standard is not found) (Form 1 "X" Flag).
LTL	LESS THAN LOWER CALIBRATION LIMIT	Analysis result is from a diluted sample (DL suffix) and may be less accurate than the result from an undiluted sample (Form 1 "D" Flag).
GTL	GREATER THAN UPPER CALIBRATION LIMIT	Actual value is known to be greater than the upper calibration range (Form 1 "E" Flag).
LLS	LESS THAN LOWER STANDARD	The analysis result is less than the sample quantitation limit (Form 1 "J" Flag).
TIE	TENTATIVELY IDENTIFIED ESTIMATED VALUE	The indicated analyte is a tentatively identified analyte; its concentration has been estimated (Form 1-E or 1-F "J" Flag).
REJ	REJECTED	Results for the analyte are rejected by the laboratory.
STD	INTERNAL STANDARD	The indicated compound is an internal standard.
STB	INTERNAL STANDARD BELOW DETECTION LIMITS	A combination of "STD" and "BDL".
FBK	FOUND IN BLANK	The indicated compound was found in the associated method blank (LRB) as well as the sample (Form 1 "B" Flag).
TFB	TENTATIVELY IDENTIFIED AND FOUND IN BLANK	A Combination of "TIE" and "FBK" (Form 1-E or 1-F "B" Flag).
ALC	ALDOL CONDENSATION	Labels a suspected Aldol Condensation-product for TICs (Form 1-F "A" Flag).
NRP	NON-REPRODUCIBLE	Results of two or more injections are not comparable (Form 1-D "P" flag), e.g., Aroclor target analyte with greater than 25% difference between mean concentrations of the two column analyses.
PRE	PRESUMPTIVE PRESENCE	Presumptive evidence of presence of material for TIC (Form 1-E or 1-F "N" Flag).

1A

EPA SAMPLE NO.

VOLATILE ORGANICS ANALYSIS DATA SHEET

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)_____ Lab File ID: _____

Level: (low/med)_____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

Soil Extract Volume: _____(µL) Soil Aliquot Volume: _____(µL)

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

VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)_____ Lab File ID: _____

Level: (low/med)_____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

Soil Extract Volume: _____(µL) Soil Aliquot Volume: _____(µL)

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

EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: _____ Decanted:(Y/N) _____ Date Extracted: _____

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

Injection Volume:_____(µL) Dilution Factor:_____

GPC Cleanup: (Y/N)_____ pH:____ Extraction: (Type)_____

CONCENTRATION UNITS:
(µg/L or µg/Kg)_____

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

EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID:_____

Sample wt/vol: _____(g/mL)_____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: _____ Decanted: (Y/N)_____ Date Extracted: _____

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

Injection Volume: _____(µL) Dilution Factor: _____

GPC Cleanup: (Y/N)_____ pH: _____ Extraction: (Type)_____

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

(1) Cannot be separated from Diphenylamine

1E

PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)_____ Lab File ID: _____

% Moisture: _____ Decanted: (Y/N)____ Date Received: _____

Extraction: (Type) _____ Date Extracted: _____

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

Injection Volume: _____(µL) Dilution Factor: _____

GPC Cleanup: (Y/N)_____ pH: _____ Sulfur Cleanup: (Y/N) _____

CONCENTRATION UNITS:
(µg/L or µg/Kg)_____

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

1F

EPA SAMPLE NO.

VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)_____ Lab File ID: _____

Level: (low/med)_____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

GC Column: _____ ID: _____(mm) Dilution Factor: _____

Soil Extract Volume: _____(µL) Soil Aliquot Volume: _____(µL)

Number TICs found: _____ CONCENTRATION UNITS:
(µg/L or µg/Kg)_____

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	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.				

EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name: _____ Contract: _____

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

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)_____ Lab File ID: _____

Level: (low/med)_____ Date Received: _____

% Moisture: _____ Decanted: (Y/N)___ Date Extracted: _____

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

Injection Volume: _____(μL) Dilution Factor: _____

GPC Cleanup: (Y/N)_____ pH: ____ Extraction: (Type)_____

Number TICS found: _____ CONCENTRATION UNITS:
(μg/L or μg/Kg)_____

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

2A

WATER VOLATILE SYSTEM MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

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

	EPA SAMPLE NO.	SMC1 (TOL) #	SMC2 (BFB) #	SMC3 (DCE) #	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
SMC1 (TOL) = Toluene-d8	(88-110)
SMC2 (BFB) = Bromofluorobenzene	(86-115)
SMC3 (DCE) = 1,2-Dichloroethane-d4	(76-114)

Column to be used to flag recovery values

* Values outside of contract required QC limits

2B

SOIL VOLATILE SYSTEM MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

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

Level: (low/med)_____

	EPA SAMPLE NO.	SMC1 (TOL) #	SMC2 (BFB) #	SMC3 (DCE) #	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
SMC1 (TOL) = Toluene-d8	(84-138)
SMC2 (BFB) = Bromofluorobenzene	(59-113)
SMC3 (DCE) = 1,2-Dichloroethane-d4	(70-121)

Column to be used to flag recovery values

* Values outside of contract required QC limits

page _____ of _____

2C

WATER SEMIVOLATILE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

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

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	S7 (2CP) #	S8 (DCB) #	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	
S1 (NBZ) = Nitrobenzene-d5	(35-114)	
S2 (FBP) = 2-Fluorobiphenyl	(43-116)	
S3 (TPH) = Terphenyl-d14	(33-141)	
S4 (PHL) = Phenol-d5	(10-110)	
S5 (2FP) = 2-Fluorophenol	(21-110)	
S6 (TBP) = 2,4,6-Tribromophenol	(10-123)	
S7 (2CP) = 2-Chlorophenol-d4	(33-110)	(advisory)
S8 (DCB) = 1,2-Dichlorobenzene-d4	(16-110)	(advisory)

Column to be used to flag recovery values

* Values outside of contract required QC limits

D Surrogate diluted out

SOIL SEMIVOLATILE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

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

Level: (low/med)_____

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	S7 (2CP) #	S8 (DCB) #	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	
S1 (NBZ) = Nitrobenzene-d5	(23-120)	
S2 (FBP) = 2-Fluorobiphenyl	(30-115)	
S3 (TPH) = Terphenyl-d14	(18-137)	
S4 (PHL) = Phenol-d5	(24-113)	
S5 (2FP) = 2-Fluorophenol	(25-121)	
S6 (TBP) = 2,4,6-Tribromophenol	(19-122)	
S7 (2CP) = 2-Chlorophenol-d4	(20-130)	(advisory)
S8 (DCB) = 1,2-Dichlorobenzene-d4	(20-130)	(advisory)

\# Column to be used to flag recovery values

* Values outside of contract required QC limits

D Surrogate diluted out

2E

WATER PESTICIDE SURROGATE RECOVERY

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

\# Column to be used to flag recovery values

* Values outside of QC limits

D Surrogate diluted out

2F

SOIL PESTICIDE SURROGATE RECOVERY

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

\# Column to be used to flag recovery values

* Values outside of QC limits

D Surrogate diluted out

WATER VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

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

Matrix Spike - EPA Sample No.: _____

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

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

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

* Values outside of QC limits

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

COMMENTS: _____

_____

SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

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

Matrix Spike - EPA Sample No.: _____ Level:(low/med)_____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
1,1-Dichloroethene					59-172
Trichloroethene					62-137
Benzene					66-142
Toluene					59-139
Chlorobenzene					60-133

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
1,1-Dichloroethene					22	59-172
Trichloroethene					24	62-137
Benzene					21	66-142
Toluene					21	59-139
Chlorobenzene					21	60-133

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

* Values outside of QC limits

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

COMMENTS: _____

_____

WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

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

Matrix Spike - EPA Sample No.: _____

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

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

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

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

* Values outside of QC limits

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

COMMENTS: _____
_____

SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

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

Matrix Spike - EPA Sample No.: _____ Level: (low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
Phenol					26-90
2-Chlorophenol					25-102
N-Nitroso-di-n-prop.(1)					41-126
4-Chloro-3-methylphenol					26-103
Acenaphthene					31-137
4-Nitrophenol					11-114
2,4-Dinitrotoluene					28-89
Pentachlorophenol					17-109
Pyrene					35-142

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS PRD	QC LIMITS REC.
Phenol					35	26-90
2-Chlorophenol					50	25-102
N-Nitroso-di-n-prop.(1)					38	41-126
4-Chloro-3-methylphenol					33	26-103
Acenaphthene					19	31-137
4-Nitrophenol					50	11-114
2,4-Dinitrotoluene					47	28-89
Pentachlorophenol					47	17-109
Pyrene					36	35-142

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

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

* Values outside of QC limits

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

COMMENTS: _____

_____

3E

WATER PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

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

Matrix Spike - EPA Sample No.: _____

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

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

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

* Values outside of QC limits

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

COMMENTS: _____

_____

3F

SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

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

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
gamma-BHC (Lindane)					46-127
Heptachlor					35-130
Aldrin					34-132
Dieldrin					31-134
Endrin					42-139
4,4'-DDT					23-134

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
gamma-BHC (Lindane)					50	46-127
Heptachlor					31	35-130
Aldrin					43	34-132
Dieldrin					38	31-134
Endrin					45	42-139
4,4'-DDT					50	23-134

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

* Values outside of QC limits

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

COMMENTS: _____

_____

4A

EPA SAMPLE NO.

VOLATILE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

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

Lab File ID: _____ Lab Sample ID: _____

Date Analyzed: _____ Time Analyzed: _____

GC Column: _____ ID: _____(mm) Heated Purge: (Y/N) ____

Instrument ID: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS, AND MSD:

	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: _____

_____

4B

EPA SAMPLE NO.

SEMIVOLATILE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

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

Lab File ID: _____ Lab Sample ID: _____

Instrument ID: _____ Date Extracted: _____

Matrix: (soil/water) _____ Date Analyzed: _____

Level: (low/med) _____ Time Analyzed: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS, AND MSD:

	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: _____

_____

4C

EPA SAMPLE NO.

PESTICIDE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

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

Lab Sample ID: _____ Lab File ID: _____

Matrix: (soil/water) _____ Extraction: (Type) _____

Sulfur Cleanup: (Y/N)_____ Date Extracted: _____

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)

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS, AND MSD:

	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: _____

_____

5A

VOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
BROMOFLUOROBENZENE (BFB)

Lab 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 CHECK APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, 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					

5B

SEMIVOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab 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 CHECK APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, 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					

6A

VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

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

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

Heated Purge: (Y/N) _____ Calibration Times: _____ _____

GC Column: _____ ID: _____(mm)

LAB FILE ID: RRF10 = _____ RRF20 = _____

RRF50 = _____ RRF100 = _____ RRF200 = _____

COMPOUND	RRF10	RRF20	RRF50	RRF100	RRF200	$\overline{RRF}$	% RSD
Dichlorodifluoromethane							
Chloromethane							
Vinyl Chloride *							*
Bromomethane *							*
Chloroethane							
Trichlorofluoromethane							
1,1-Dichloroethene *							*
1,1,2-Trichloro-1,2,2-trifluoroethane							
Acetone							
Carbon Disulfide							
Methyl Acetate							
Methylene Chloride							
trans-1,2-Dichloroethene							
tert-Butyl Methyl Ether							
1,1-Dichloroethane *							*
cis-1,2-Dichloroethene							
2-Butanone							
Chloroform *							*
1,1,1-Trichloroethane *							*
Cyclohexane							
Carbon Tetrachloride *							*
Benzene *							*
1,2-Dichloroethane *							*
Trichloroethene *							*
Methylcyclohexane							

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

All other compounds must meet a minimum RRF of 0.010.

VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

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

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

Heated Purge: (Y/N) _____ Calibration Times: _____ _____

GC Column: _____ ID: _____(mm)

LAB FILE ID: RRF10 = _____ RRF20 = _____

RRF50 = _____ RRF100 = _____ RRF200 = _____

COMPOUND	RRF10	RRF20	RRF50	RRF100	RRF200	$\overline{RRF}$	% RSD
1,2-Dichloropropane							
Bromodichloromethane *							*
cis-1,3-Dichloropropene *							*
4-Methyl-2-pentanone							
Toluene *							*
trans-1,3-Dichloropropene *							*
1,1,2-Trichloroethane *							*
Tetrachloroethene *							*
2-Hexanone							
Dibromochloromethane *							*
1,2-Dibromoethane							
Chlorobenzene *							*
Ethylbenzene *							*
Xylene (total) *							*
Styrene *							*
Bromoform *							*
Isopropylbenzene							
1,1,2,2-Tetrachloroethane *							*
1,3-Dichlorobenzene *							*
1,4-Dichlorobenzene *							*
1,2-Dichlorobenzene *							*
1,2-Dibromo-3-chloropropane *							*
1,2,4-Trichlorobenzene							
Toluene-d8							
Bromoflurobenzene *							*
1,2-Dichloroethane-d4							

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

6C

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

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

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

Calibration Times: _____ _____

LAB FILE ID: RRF20 = _____ RRF50 = _____

RRF80 = _____ RRF120 = _____ RRF160 = _____

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Benzaldehyde							
Phenol *							*
bis-(2-Chloroethyl)ether *							*
2-Chlorophenol *							*
2-Methylphenol *							*
2,2'-oxybis(1-Chloropropane)							
Acetophenone							
4-Methylphenol *							*
N-Nitroso-di-n-propylamine *							*
Hexachloroethane *							*
Nitrobenzene *							*
Isophorone *							*
2-Nitrophenol *							*
2,4-Dimethylphenol *							*
bis(2-Chloroethoxy)methane *							*
2,4-Dichlorophenol *							*
Naphthalene *							*
4-Chloroaniline							
Hexachlorobutadiene							
Caprolactam							
4-Chloro-3-methylphenol *							*
2-Methylnaphthalene *							*
Hexachlorocyclopentadiene							
2,4,6-Trichlorophenol *							*
2,4,5-Trichlorophenol *							*
1,1'-Biphenyl							
2-Chloronaphthalene *							*
2-Nitroaniline							
Dimethylphthalate							
2,6-Dinitrotoluene *							*
Acenaphthylene *							*
3-Nitroaniline							
Acenaphthene *							*
2,4-Dinitrophenol							
4-Nitrophenol							
Dibenzofuran *							*

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

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

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

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

Calibration Times: _____ _____

LAB FILE ID: RRF20 = _____ RRF50 = _____

RRF80 = _____ RRF120 = _____ RRF160 = _____

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
2,4-Dinitrotoluene *							*
Diethylphthalate							
Fluorene *							*
4-Chlorophenyl-phenylether *							*
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine (1)							
4-Bromophenyl-phenylether *							*
Hexachlorobenzene *							*
Atrazine							
Pentachlorophenol *							*
Phenanthrene *							*
Anthracene *							*
Carbazole							
Di-n-butylphthalate							
Fluoranthene *							*
Pyrene *							*
Butylbenzylphthalate							
3,3'-Dichlorobenzidine							
Benzo(a)anthracene *							*
Chrysene *							*
bis(2-Ethylhexyl)phthalate							
Di-n-octylphthalate							
Benzo(b)fluoranthene *							*
Benzo(k)fluoranthene *							*
Benzo(a)pyrene *							*
Indeno(1,2,3-cd)pyrene *							*
Dibenzo(a,h)anthracene *							*
Benzo(g,h,i)perylene *							*
Nitrobenzene-d5							
2-Fluorobiphenyl *							*
Terphenyl-d14 *							*
Phenol-d5 *							*
2-Fluorophenol *							*
2,4,6-Tribromophenol							
2-Chlorophenol-d4 *							*
1,2-Dichlorobenzene-d4 *							*

(1) Cannot be separated from Diphenylamine

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

PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab 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 Standard Mix A analyses.

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

6F

PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

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

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

6G

PESTICIDE INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name: _____ Contract: _____

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

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

GC Column: _____ ID: _____(mm)

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

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

6H

PESTICIDE ANALYTE RESOLUTION SUMMARY

Lab Name: _____ Contract: _____

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

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

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

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

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

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

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

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

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

PERFORMANCE EVALUATION MIXTURE (PEM)

Lab Name: _____ Contract: _____

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

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

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

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

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

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

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

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

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

6J

INDIVIDUAL STANDARD MIXTURE A

Lab Name: _____ Contract: _____

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

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

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

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

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

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

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

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

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

6K

INDIVIDUAL STANDARD MIXTURE B

Lab Name: _____ Contract: _____

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

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

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

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

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

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

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

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

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

7A
VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Calibration Date: _____ Time: _____

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

EPA Sample No.(VSTD050##): _____ Init. Calib. Times: _____ _____

Heated Purge: (Y/N)_____

GC Column: _____ ID: _____(mm)

COMPOUND	RRF	RRF50	MIN RRF	%D	MAX %D
Dichlorodifluoromethane					
Chloromethane					
Vinyl Chloride			0.100		25.0
Bromomethane			0.100		25.0
Chloroethane					
Trichlorofluoromethane					
1,1-Dichloroethene			0.100		25.0
1,1,2-Trichloro-1,2,2-trifluoroethane					
Acetone					
Carbon Disulfide					
Methyl Acetate					
Methylene Chloride					
trans-1,2-Dichloroethene					
tert-Butyl Methyl Ether					
1,1-Dichloroethane			0.200		25.0
cis-1,2-Dichloroethene					
2-Butanone					
Chloroform			0.200		25.0
1,1,1-Trichloroethane			0.100		25.0
Cyclohexane					
Carbon Tetrachloride			0.100		25.0
Benzene			0.500		25.0
1,2-Dichloroethane			0.100		25.0
Trichloroethene			0.300		25.0
Methylcyclohexane					

All other compounds must meet a minimum RRF of 0.010.

VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Calibration Date: _____ Time: _____

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

EPA Sample No.(VSTD050##): _____ Init. Calib. Times: _____ _____

Heated Purge: (Y/N) _____

GC Column:_____ID:_____(mm)

COMPOUND	$\overline{RRF}$	RRF50	MIN RRF	%D	MAX %D
1,2-Dichloropropane					
Bromodichloromethane			0.200		25.0
cis-1,3-Dichloropropene			0.200		25.0
4-Methyl-2-pentanone					
Toluene			0.400		25.0
trans-1,3-Dichloropropene			0.100		25.0
1,1,2-Trichloroethane			0.100		25.0
Tetrachloroethene			0.200		25.0
2-Hexanone					
Dibromochloromethane			0.100		25.0
1,2-Dibromoethane					
Chlorobenzene			0.500		25.0
Ethylbenzene			0.100		25.0
Xylene (total)			0.300		25.0
Styrene			0.300		25.0
Bromoform			0.100		25.0
Isopropylbenzene					
1,1,2,2-Tetrachloroethane			0.300		25.0
1,3-Dichlorobenzene			0.600		25.0
1,4-Dichlorobenzene			0.500		25.0
1,2-Dichlorobenzene			0.400		25.0
1,2-Dibromo-3-chloropropane					
1,2,4-Trichlorobenzene			0.200		25.0
Toluene-d8					
Bromoflurobenzene			0.200		25.0
1,2-Dichloroethane-d4					

All other compounds must meet a minimum RRF of 0.010.

7C

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Calibration Date: _____ Time: _____

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

EPA Sample No.(SSTD050##): _____ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(mm)

COMPOUND	$\overline{RRF}$	RRF50	MIN RRF	%D	MAX %D
Benzaldehyde					
Phenol			0.800		25.0
bis-(2-Chloroethyl)ether			0.700		25.0
2-Chlorophenol			0.800		25.0
2-Methylphenol			0.700		25.0
2,2'-oxybis(1-Chloropropane)					
Acetophenone					
4-Methylphenol			0.600		25.0
N-Nitroso-di-n-propylamine			0.500		25.0
Hexachloroethane			0.300		25.0
Nitrobenzene			0.200		25.0
Isophorone			0.400		25.0
2-Nitrophenol			0.100		25.0
2,4-Dimethylphenol			0.200		25.0
bis(2-Chloroethoxy)methane			0.300		25.0
2,4-Dichlorophenol			0.200		25.0
Naphthalene			0.700		25.0
4-Chloroaniline					
Hexachlorobutadiene					
Caprolactam					
4-Chloro-3-methylphenol			0.200		25.0
2-Methylnaphthalene			0.400		25.0
Hexachlorocyclopentadiene					
2,4,6-Trichlorophenol			0.200		25.0
2,4,5-Trichlorophenol			0.200		25.0
1,1'-Biphenyl					
2-Chloronaphthalene			0.800		25.0
2-Nitroaniline					
Dimethylphthalate					
2,6-Dinitrotoluene			0.200		25.0
Acenaphthylene			0.900		25.0
3-Nitroaniline					
Acenaphthene			0.900		25.0
2,4-Dinitrophenol					
4-Nitrophenol					
Dibenzofuran			0.800		25.0

All other compounds must meet a minimum RRF of 0.010.

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Calibration Date: _____ Time: _____

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

EPA Sample No.(SSTD050##): _____ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(mm)

COMPOUND	$\overline{RRF}$	RRF50	MIN RRF	%D	MAX %D
2,4-Dinitrotoluene			0.200		25.0
Diethylphthalate					
Fluorene			0.900		25.0
4-Chlorophenyl-phenylether			0.400		25.0
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine (1)					
4-Bromophenyl-phenylether			0.100		25.0
Hexachlorobenzene			0.100		25.0
Atrazine					
Pentachlorophenol			0.050		25.0
Phenanthrene			0.700		25.0
Anthracene			0.700		25.0
Carbazole					
Di-n-butylphthalate					
Fluoranthene			0.600		25.0
Pyrene			0.600		25.0
Butylbenzylphthalate					
3,3'-Dichlorobenzidine					
Benzo(a)anthracene			0.800		25.0
Chrysene			0.700		25.0
bis(2-Ethylhexyl)phthalate					
Di-n-octylphthalate					
Benzo(b)fluoranthene			0.700		25.0
Benzo(k)fluoranthene			0.700		25.0
Benzo(a)pyrene			0.700		25.0
Indeno(1,2,3-cd)pyrene			0.500		25.0
Dibenzo(a,h)anthracene			0.400		25.0
Benzo(g,h,i)perylene			0.500		25.0
Nitrobenzene-d5			0.200		25.0
2-Fluorobiphenyl			0.700		25.0
Terphenyl-d14			0.500		25.0
Phenol-d5			0.800		25.0
2-Fluorophenol			0.600		25.0
2,4,6-Tribromophenol					
2-Chlorophenol-d4			0.800		25.0
1,2-Dichlorobenzene-d4			0.400		25.0

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

7E

PESTICIDE CALIBRATION VERIFICATION SUMMARY

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

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

Combined % Breakdown (1): _____

7F

PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab 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. (INDAM##): _____ Date Analyzed: _____

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

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

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

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

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

VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: _____ Contract: _____

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

EPA Sample No.(VSTD050##): _____ Date Analyzed: _____

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

Instrument ID: _____ Heated Purge: (Y/N)_____

GC Column: _____ID: _____(mm)

	IS1 (BCM) AREA	#	RT	#	IS2 (DFB) AREA	#	RT	#	IS3 (CBZ) AREA	#	RT	#
12 HOUR STD												
UPPER LIMIT												
LOWER LIMIT												
EPA SAMPLE												
01												
02												
03												
04												
05												
06												
07												
08												
09												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (CBZ) = Chlorobenzene-d5

AREA UPPER LIMIT = + 100% of internal standard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

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

8B

SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: _____ Contract: _____

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

EPA Sample No.(SSTD050##): _____ Date Analyzed: _____

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

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

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

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

AREA UPPER LIMIT = + 100% of internal standard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

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

8C

SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: _____ Contract: _____

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

EPA Sample No.(SSTD050##): _____ Date Analyzed: _____

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

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

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

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

AREA UPPER LIMIT = + 100% of internal standard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

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

8D

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						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						

QC LIMITS

TCX = Tetrachloro-m-xylene (± 0.05 MINUTES)
DCB = Decachlorobiphenyl (± 0.10 MINUTES)

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

9A

PESTICIDE FLORISIL CARTRIDGE CHECK

Lab Name: _____ Contract: _____

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

Florisil Cartridge Lot Number: _____ Date of Analysis: _____

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

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

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

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

	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				

9B

PESTICIDE GPC CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

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

GPC Column: _____ Calibration Verification Date: _____

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

COMPOUND	SPIKE ADDED (ng)	SPIKE RECOVERED (ng)	% REC #	QC LIMITS
gamma-BHC (Lindane)				80-110
Heptachlor				80-110
Aldrin				80-110
Dieldrin				80-110
Endrin				80-110
4,4'-DDT				80-110

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

THIS GPC CALIBRATION VERIFICATION APPLIES TO THE FOLLOWING SAMPLES, BLANKS, MS, AND MSD:

	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				

10A

EPA SAMPLE NO.

PESTICIDE IDENTIFICATION SUMMARY
FOR SINGLE COMPONENT ANALYTES

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

page _____ of _____

10B

PESTICIDE IDENTIFICATION SUMMARY FOR MULTICOMPONENT ANALYTES

EPA SAMPLE NO.

Lab 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			
	1						
________	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						
	1						
________	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						
	1						
________	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						

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

page ______ of ______

SAMPLE LOG-IN SHEET

Lab Name		Page __ of __
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	Sample Delivery Group No.	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 Lists — 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. Cooler Temperature ____________
10. Does information on custody records, traffic reports, and sample tags agree? — Yes/No*
11. Date Received at Lab ____________
12. Time Received ____________

EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.

Sample Transfer

Fraction	Fraction
Area #	Area #
By	By
On	On

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

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.

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

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

_____ _____ SAS NO. _____

	PAGE NOs FROM	PAGE NOs TO	CHECK LAB	CHECK EPA
5. **Semivolatiles Data**				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II SV)				
MS/MSD Summary (Form III SV)				
Method Blank Summary (Form IV SV)				
GC/MS Instrument Performance Check (Form V SV)				
Internal Standard Area and RT Summary (Form VIII SV)				
b. Sample Data				
TCL Results - (Form I SV-1, SV-2)				
Tentatively Identified Compounds (Form I SV-TIC)				
Reconstructed total ion chromatograms (RIC) for each sample				
For each sample:				
Raw Spectra and background-subtracted mass spectra of target compounds				
Quantitation reports				
Mass Spectra of TICs with three best library matches				
GPC chromatograms (if GPC is required)				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI SV-1, SV-2)				
RICs and Quan Reports for all Standards				
Continuing Calibration Data (Form VII SV-1, SV-2)				
RICs and Quantitation Reports for all Standards				
d. Raw QC Data				
DFTPP				
Blank Data				
Matrix Spike/Matrix Spike Duplicate Data				
e. Raw GPC Data				
6. **Pesticides Data**				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II PEST)				
MS/MSD Duplicate Summary (Form III PEST)				
Method Blank Summary (Form IV PEST)				

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

_____ _____ SAS NO. _____

	PAGE NOs FROM	PAGE NOs TO	CHECK LAB	CHECK EPA
6. **Pesticides Data** (Cont.)				
b. Sample Data	_____	_____		
TCL Results - Organic Analysis Data Sheet (Form I PEST)			_____	_____
Chromatograms (Primary Column)			_____	_____
Chromatograms from second GC column confirmation			_____	_____
GC Integration report or data system printout			_____	_____
Manual work sheets			_____	_____
For pesticides/Aroclors by GC/MS, Copies of raw spectra and copies of background-subtracted mass spectra of target compounds (samples & standards)			_____	_____
c. Standards Data	_____	_____		
Initial Calibration of Single Component Analytes (Form VI PEST-1 and PEST-2)			_____	_____
Initial Calibration of Multicomponent Analytes (Form VI PEST-3)			_____	_____
Analyte Resolution Summary (Form VI PEST-4)			_____	_____
Performance Evaluation Mixture (Form VI PEST-5)			_____	_____
Individual Standard Mixture A (FORM VI PEST-6)			_____	_____
Individual Standard Mixture B (FORM VI PEST-7)			_____	_____
Calibration Verification Summary (Form VII PEST-1)			_____	_____
Calibration Verification Summary (Form VII PEST-2)			_____	_____
Analytical Sequence (Form VIII PEST)			_____	_____
Florisil Cartridge Check (Form IX PEST-1)			_____	_____
Pesticide GPC Calibration (Form IX PEST-2)			_____	_____
Pesticide Identification Summary for Single Component Analytes (Form X PEST-1)			_____	_____
Pesticide Identification Summary for Multicomponent Analytes (Form X PEST-2)			_____	_____
Chromatograms and data system printouts A printout of retention times and corresponding peak areas or peak heights			_____	_____
d. Raw QC Data				
Blank Data	_____	_____	_____	_____
Matrix Spke/Matrix Spike Duplicate Data	_____	_____	_____	_____

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

_____ _____ SAS NO. _____

	PAGE NOs FROM	TO	CHECK LAB	EPA
6. **Pesticides Data** (Cont.)				
e. Raw GPC Data				
f. Raw Florisil Data				
7. **Miscellaneous Data**				
Original preparation and analysis forms or copies of preparation and analysis logbook pages				
Internal sample and sample extract transfer chain-of-custody records				
Screening records				
All instrument output, including strip charts from screening activities (describe or list)				
_____				
_____				
8. **EPA Shipping/Receiving Documents**				
Airbills (No. of shipments _____)				
Chain-of-Custody Records				
Sample Tags				
Sample Log-in Sheet (Lab & DC1)				
Miscellaneous Shipping/Receiving Records (describe or list)				
_____				
_____				
9. **Internal Lab Sample Transfer Records and Tracking Sheets** (describe or list)				
_____				
_____				
10. **Other Records** (describe or list)				
Telephone Communication Log				
_____				
_____				

11. **Comments:** _____

_____

_____

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (cont.)

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

_____ _____ SAS NO. _____

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