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Superfund

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK FOR ORGANICS ANALYSIS

MULTI-MEDIA, MULTI-CONCENTRATION

OLMO 1.9

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FOR

ORGANICS ANALYSIS
Multi-Media, Multi-Concentration

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STATEMENT OF WORK

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EXHIBIT A

SUMMARY OF REQUIREMENTS

SECTION I

GENERAL REQUIREMENTS

The Contractor shall use proven instruments and techniques to identify and measure the concentrations of volatile, semivolatile, and pesticide compounds listed on the Target Compound List (TCL) in Exhibit C. The Contractor shall employ state-of-the-art GC/MS and/or GC/EC procedures to perform all analyses, including the necessary preparations for analysis.

In Exhibit D, the EPA provides the Contractor with the specific analytical procedures to be used and defines the specific application of these procedures to this contract. For volatiles and semivolatiles, this includes instructions for sample preparation, gas chromatographic screening, mass spectrometric identification, and data evaluation. Specific ions used for searching the mass spectral data for each compound are included. For pesticides, this includes instructions for sample preparation, gas chromatography, confirmation of identification by gas chromatography and/or mass spectrometry, and data evaluation.

The Contractor shall prepare extracts and dilutions of samples. The Contractor shall screen extracts by methods of his choice (soil characterization mandatory; water characterization optional) at an initial extract concentration. Then, based on the screening response, the Contractor shall use the specific analytical methods described in Exhibit D to extract and concentrate samples to achieve the Contract Required Quantitation Limits (CRQL) listed in Exhibit C. Exhibit D lists the analytical methods and starting points to be utilized for each of the target compounds.

During preparation, the Contractor shall fortify all semivolatile and pesticide samples, blanks, matrix spikes, and matrix spike duplicates with the surrogate spiking compounds listed in Exhibit D. The Contractor shall fortify all volatile samples, blanks, matrix spikes, and matrix spike duplicates with the system monitoring compounds listed in Exhibit D. Additionally, all sample semivolatile extracts and aliquots for volatile organics analysis shall be spiked with the internal standard compounds listed in Exhibit D before injection or purging.

Additionally, for each sample analyzed by GC/MS, the Contractor shall conduct mass spectral library searches to determine the possible identity of up to ten (10) volatile components and up to twenty (20) semivolatile components that are neither system monitoring compounds, surrogates, internal standards, or volatile or semivolatile target compounds (see Exhibit C).

Exhibit F contains chain-of-custody and sample documentation requirements which the Contractor must follow in processing samples under this contract, and specifies requirements for written laboratory standard operating procedures.

Sample analysis data, sample documentation and other deliverables shall be reported as specified in Exhibit B. Specifications for reporting data in computer-readable form appear in Exhibit H.

To ensure proper understanding of language utilized in this contract, Exhibit G contains a glossary of terms. When a term is used in the text without explanation, the glossary meaning shall be applicable.

The samples to be analyzed by the Contractor are from known or suspected hazardous waste sites and, potentially, may contain hazardous organic and/or inorganic materials at high concentration levels. The Contractor should be aware of the potential hazards associated with the handling and analyses of these samples. It is the Contractor's responsibility to take all necessary measures to ensure the health and safety of its employees.

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

Prior to accepting any samples from the Agency, the Contractor shall have, in-house, the appropriate standards for all target compounds listed in Exhibit C.

SECTION II

SUMMARY OF REQUIREMENTS

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

A. Task I Receive and Prepare Hazardous Waste Samples.

1. Receive and handle samples under the chain-of-custody procedures described in Exhibit F.

2. Prepare samples as described in Exhibit D. VOA analysis of water or soil samples must be completed within 10 days of VTSR (Validated Time of Sample Receipt). Separatory funnel extractions for pesticides in water samples must be completed within 5 days of VTSR. Sonication extractions for pesticides and/or semivolatiles in soil samples must be completed within 10 days of VTSR. Continuous liquid-liquid extraction for semivolatile samples must be started within 5 days of VTSR.

Extracts of either water or soil samples must be analyzed within 40 days of extraction. This does not release the Contractor from the data turnaround time specified in Exhibit B, Section I.

B. Task II Analysis for Identification of Specific Organic Compounds.

1. Extracts and aliquots prepared in Task I shall be analyzed by GC/EC and GC/MS techniques given in Exhibit D for the target compounds listed in Exhibit C.

2. 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.

C. Task III Qualitative Verification of the Compounds Identified in Task II.

1. The volatile and semivolatile compounds analyzed by GC/MS techniques and initially identified in Task II 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. Two criteria must be satisfied to verify the identifications:

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

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

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 calibration standard must be run on the same 12-hour time period as the sample.

   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 DFTPP or BFB daily instrument performance check requirements specified in Exhibit D. The standard spectra used may be from a laboratory generated library on the same instrument or obtained from the calibration standard run used to obtain reference RRTs. The requirements for qualitative verification by comparison of mass spectra are as follows:

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

   b. The relative intensities of ions specified above must agree within ± 20 percent between the standard and sample spectra.

   c. Ions greater than 10 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. When GC/MS computer data processing programs are used to obtain the sample component spectrum, both the processed and the raw spectra must be evaluated. In Task III, the verification process should favor false positives.

3. If a compound analyzed by GC/MS techniques and initially identified in Task II cannot be verified by all of the criteria in items 1 and 2 above, but in the technical judgement of the mass spectral interpretation specialist the identification is correct, then the Contractor shall report that identification, and proceed with quantification in Task IV.

4. The Pesticide/Aroclor compounds listed in Exhibit C and analyzed by GC/EC techniques shall have their identifications verified by an analyst competent in the interpretation of gas chromatograms. Two criteria must be satisfied to verify the identifications:

a. Elution of the sample component within the retention time window (established by the procedures in Exhibit D) of the standard component analyzed on the same GC column and instrument, as specified in Exhibit D.

b. Analysis of the sample and standard on a second GC column with a stationary phase with retention characteristics dissimilar to that used in a. above, and meeting the same criteria for elution of the sample component and the standard as in a. above.

D. Task IV Quantification of Compounds Verified in Task III.

1. The Contractor shall quantify components analyzed by GC/MS techniques, identified in Task II and verified in Task III 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.

2. The Contractor shall determine response factors for each 12-hour time period of GC/MS analysis and shall include a calibration check of the initial five point calibration as described in Exhibit D.

3. The Contractor shall quantify components analyzed by GC/EC techniques, identified in Task II and verified in Task III by the external standard method stipulated in Exhibit D.

4. The Contractor shall 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.

E. Task V Tentative Identification of Non-target Sample Components.

1. For each analysis of a sample, the Contractor shall conduct mass spectral library searches to determine tentative compound identifications as follows. For each volatile fraction, the Contractor shall conduct a search to determine the possible identity of up to ten (10) organic compounds of greatest concentration which are not system monitoring compounds and are not listed in Exhibit C. For each semivolatile fraction, the Contractor shall conduct a search to determine the possible identification of up to twenty (20) non-surrogate organic compounds of greatest concentration which are not listed in Exhibit C. In performing searches, the most recent release of the NIST/EPA/MSDC mass spectral library must be used. NOTE: Substances with responses less than 10 percent of the nearest internal standard are not required to be searched in this fashion.

2. Only after visual comparison of sample spectra with the spectra from the library searches will the mass spectral interpretation specialist assign a tentative identification. If the compound does not meet the identification criteria of Task III, it shall 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.

3. The Contractor shall not report as semivolatile tentatively identified compounds (TIC) any target compounds from the volatile fraction (i.e., do not report late eluting volatile compounds as TICs in the semivolatile analysis). However, the Contractor may report pesticide target compounds that appear as semivolatile tentatively identified compounds.

F. Task VI Quality Assurance/Quality Control Procedures.

1. All specific quality assurance procedures prescribed in Exhibits D and E shall be strictly adhered to by the Contractor. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibit B, Reporting Requirements and Deliverables, and Exhibit H, Data Dictionary and Format for Data Deliverables in Computer-Readable Format.

2. The Contractor shall establish 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.

3. The Contractor shall perform one spiked sample analysis (matrix spike) and one duplicate spiked sample analysis (matrix spike duplicate) for each group of samples of a similar matrix (for water or soil samples) and concentration level (for volatile and semivolatile soil samples only) for the following, whichever is most frequent:

   - Each Case of field samples received, OR
   - Each 20 samples in a Case, OR
   - Each 14 calendar day period during which field samples in a Case were received (7 calendar day period for 14-day data turnaround contracts) (said period beginning with the receipt of the first sample in that Sample Delivery Group).

Matrix spikes and matrix spike duplicates shall be carried through the entire analytical process from extraction to final GC/MS or GC/EC analysis, including all Contract Performance/Delivery Requirements (see Contract Schedule).

4. The Contractor shall prepare and analyze one laboratory reagent blank (method blank) for each group of samples of a similar matrix (for water or soil samples), extracted by a similar method (separatory funnel, continuous liquid-liquid extraction, or sonication, as specified in Exhibit D), and a similar concentration level (for volatile and semivolatile soil samples only) for the following, whichever is most frequent:

   - o Each Case of field samples received, OR
   - o Each 20 samples in a Case, including matrix spikes and reanalyses, OR
   - o Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which field samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
   - o Whenever samples are extracted.

   Volatile analysis requires one method blank for each 12-hour time period when volatile target compounds are analyzed.

   Semivolatile and pesticide method blanks shall be carried through the entire analytical process from extraction to final GC/MS or GC/EC analysis, including all Contract Performance/Delivery Requirements (see Contract Schedule).

5. The Contractor shall verify instrument performance for each 12-hour time period, to include the following: Decafluorotriphenylphosphine (DFTPP) and/or Bromofluorobenzene (BFB) as applicable, and a specific calibration using standards of defined concentration to monitor response, retention time, and mass spectra.

   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 control or laboratory evaluation samples may be used as grounds for termination of noncompliant Contractors. "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 SOP documentation (Exhibit F).

B. The EPA has provided to the Contractor formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and returning analysis data sheets and submitting computer-readable data on diskette in the format specified in this SOW and within the time specified in the Contract Performance/Delivery Schedule.

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

2. 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.

3. The data reported by the Contractor on the hardcopy data forms and the associated computer-readable data submitted by the Contractor must contain identical information. If during government inspection discrepancies are found, 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 government.

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

1. The Contractor shall have sufficient gas chromatograph/electron capture/data systems (GC/EC/DS) and gas chromatograph/mass spectrometer/data system (GC/MS/DS) capability to meet all the terms and conditions of the Contract. The Contractor shall maintain, at a minimum, all analytical equipment allocated for this contract at the time of contract award.

2. The Contractor's instrument systems shall have the following:

   a. The GC/MS shall be equipped with a glass jet separator when using packed columns.

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

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

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

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

f. The GC/MS shall be equipped with a GC to MS interface capable of extending a fused silica capillary column into the ion source. The column is to be 30 meters long by 0.25 or 0.32 mm inside diameter, bonded DB-5, fused silica, or equivalent.

g. The GC/EC/DS for pesticide analysis shall be equipped with wide bore capillary columns and a suitable detector and data system as described in Exhibit D.

3. The Contractor shall use a magnetic tape storage device capable of recording data and suitable for long-term, off-line storage. The Contractor shall retain all raw GC/MS data acquired under this contract on magnetic tape in appropriate instrument manufacturer's format. The Contractor is required to retain the magnetic tapes with associated hardcopy tape logbook identifying tape contents (see Exhibit E) for 365 days after data submission. During that time, the Contractor shall submit tapes and logbook within 7 days of request from EMSL/LV or the Administrative Project Officer (APO), as specified in the Contract Performance/Delivery Schedule and Exhibit E.

4. The Contractor shall have a computerized MS library search system capable of providing a forward comparison, utilizing the standard spectra contained in the mass spectral library. The most recent release of the NIST/EPA/MSDC mass spectral library, must be used.

   a. The system shall provide a numerical ranking of the standard spectra most closely corresponding to the sample spectra examined.

   b. The data system shall have software capable of removing background signals from spectra.

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

6. The Contractor shall have, in-house, the appropriate standards for all target compounds listed in Exhibit C prior to accepting any samples from the Agency.

D. The Contractor shall have an IBM or IBM-compatible mini-computer or PC capable of recording required sample data on 5.25 inch floppy double-sided double-density 360 K-byte or 1.2 M-byte or a 3.5 inch double-sided, double density 720 K-byte or 1.44 M-byte diskette, in ASCII text file format, and in accordance with the file, record and field specifications listed in Exhibit H.

E. The Contractor shall designate and utilize key personnel to perform the minimum functional requirements necessary to meet the terms and conditions of this contract. The EPA reserves the right to review personnel qualifications and experience. The minimum functional requirements are listed below:

- Project Manager
- GC/MS Laboratory Supervisor
- GC/EC Laboratory Supervisor
- Sample Preparation Laboratory Supervisor
- Quality Assurance Officer
- Systems Manager
- Programmer Analyst
- GC/MS Operator
- Mass Spectral Interpretation Specialist
- GC/EC Operator
- Pesticide Residue Analysis Expert
- Extraction/Concentration Expert
- Sample Custodian
- Data Reporting and Delivery Officer

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

G. The Contractor shall store all samples and unused sample volume at 4°C (±2°C), protected from light, from time of receipt until 60 days after data submission. Samples and unused sample volumes must be stored separately from sample extracts and standards. The Contractor shall preserve all sample extracts after analysis in bottles/vials with Teflon-lined septa and shall maintain stored extracts at 4°C (±2°C). The Contractor is required to retain the sample extracts for 365 days after data submission. During that time, the Contractor shall submit the extracts within seven days of a written request by TPO/APO, as specified in the Contract Performance/Delivery Schedule.

H. The Contractor shall adhere to chain-of-custody procedures described in Exhibit F. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported as the complete SDG file purge (see Exhibit B).

I. Sample shipments to the Contractor's facility will be scheduled and coordinated by the EPA CLP Sample Management Office (SMO) acting on behalf of the Administrative Project Officer. 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.

   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.

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

   A Case consists of one or more 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 within a Case, OR
   - Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the Sample Delivery Group).

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory. Such assignment must be made at the time the samples are received, and may not be made retroactively.

All data (hardcopy and computer-readable format) for all samples in a Sample Delivery Group are due concurrently to all data recipients as stipulated in the Delivery Schedule in Exhibit B, Section I. Data for all samples in a Sample Delivery Group must be submitted together (in one package) in the order specified in Exhibit B. The Sample Delivery Group 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. The SDG number is reported on all data reporting forms.

The SDG Receipt Date is the day the last sample in the SDG is received. Data for all samples in the SDG are due as stipulated in the Delivery Schedule in Exhibit B.

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

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

The Contractor shall submit signed copies of Traffic Reports for all samples in a Sample Delivery Group to SMO within 3 calendar days following receipt of the last sample in the Sample Delivery Group. Traffic Reports shall be submitted in Sample Delivery Group sets (i.e., all Traffic Reports for a Sample Delivery Group shall be clipped together) with an SDG Cover Sheet containing information regarding the Sample Delivery Group, as specified in Exhibit B.

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

M. 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.

N. 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.

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

P. 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 analyses. The Contractor shall not perform MS/MSD analyses on any of the field QC samples.

Q. 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 the MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analyses. 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 analyses. The rationale for the choice of a sample other than the one designated by the Region shall be documented in the SDG narrative.

R. 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 analyses be performed or will require that a reduced volume be used for the MS/MSD analyses. No other changes in the analyses will be permitted. SMO will notify the Contractor of the Region's decision. The Contractor shall document the Region's decision in the SDG narrative.

S. When a Contractor receives <u>only</u> a Performance Evaluation (PE) sample(s), no MS/MSD shall be performed within that SDG.

T. 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.

SECTION III

DETAILED TECHNICAL & MANAGEMENT REQUIREMENTS

As cited in Section II the Contractor shall have the following technical and management capabilities. Note: For those technical functions which require a minimum educational degree and experience, an advanced degree in chemistry or any scientific/engineering discipline, (e.g., Master's or Doctorate) does not substitute for the minimum experience requirements.

Any personnel changes affecting the key personnel as stated in Exhibit A, Section III, Items I and II, the Contractor shall notify in writing the Technical Project Officer and the Administrative Project Officer within 14 days of the personnel change. The Contractor shall provide a detailed resume to the Technical Project Officer, Administrative Project Officer, and EMSL/LV for the replacement personnel within 14 days of the Contractor's assignment of the personnel. The resume shall include position description of titles, education (pertinent to this contract), number of years of experience (pertinent to this contract) month and year hired, previous experience and publications.

I. TECHNICAL CAPABILITY

A. Technical Functions

1. GC/MS Laboratory Supervisor

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

b. Qualifications:

(1) Education:

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

(2) Experience:

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

2. GC/EC Laboratory Supervisor

a. Responsible for all technical efforts of the GC/EC laboratory to meet all terms and conditions of the EPA contract.

b. Qualifications:

(1) Education:

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

(2) Experience:

Minimum of three years of laboratory experience in operating a GC/EC, including at least one year of supervisory experience.

3. Sample Preparation Laboratory Supervisor

a. Responsible for all technical efforts of sample preparations to meet all terms and conditions of the EPA contract.

b. Qualifications:

(1) Education:

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

(2) Experience:

Minimum of three years of laboratory experience in organic sample preparation, including at least one year of supervisory experience.

4. Quality Assurance Officer

a. Responsible for overseeing the quality assurance aspects of the data and reporting directly to upper management to meet all terms and conditions of the EPA contract.

b. Qualifications:

(1) Education:

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

(2) Experience:

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

5. Systems Manager

   a. Responsible for the management and quality control of all computing systems (hardware, software, documentation and procedures), generating, updating, and quality controlling automated deliverables to meet all terms and conditions of the EPA contract.

   b. Qualifications:

      (1) Education:

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

      (2) Experience:

          Minimum of three years experience in data or systems management or programming including one year of experience with the software being utilized for data management and generation of laboratory reports.

6. Programmer Analyst

   a. Responsible for the installation, operation and maintenance of software and programs generating, updating and quality controlling analytical databases and automated deliverables to meet all terms and conditions of the EPA contract.

   b. Qualifications:

      (1) Education:

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

      (2) Experience:

          Minimum of two years experience in systems or applications programming including one year of experience with the software being utilized for data management and generation of laboratory reports.

7. GC/MS Operator Qualifications

One year of experience in operating and maintaining GC/MS/DS and a minimum of a Bachelor's degree in chemistry or a scientific/engineering discipline, or in lieu of the minimum education requirement, three years of experience in operating and maintaining the GC/MS and interpreting GC/MS data.

8. Mass Spectral Interpretation Specialist Qualifications

a. Education:

- o Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
- o Training course(s) in mass spectral interpretation.

b. Experience:

Minimum of two years of experience in mass spectral interpretation.

9. GC/EC Operator Qualifications

One year of experience in operating and maintaining GC/EC and a minimum of a Bachelor's degree in chemistry or a scientific/engineering discipline, or in lieu of the minimum education requirement, three years of experience in operating and maintaining the GC/EC and interpreting GC/EC data.

10. Pesticide Residue Analysis Expert Qualifications

a. Education:

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

b. Experience:

Minimum of two years of experience in operating and maintaining GC and interpreting GC chromatograms.

11. Extraction/Concentration Expert Qualifications

a. Education:

Minimum of High school diploma and a college level course in general chemistry.

b. Experience:

Minimum of one year of experience in extraction/concentration.

12. Technical Staff Redundancy

The bidder shall have a minimum of one (1) chemist available at any one time as a back-up technical person with the following qualifications, to ensure continuous operations to accomplish the required work as specified by EPA contract.

a. Education:

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

b. Experience: Minimum of one year in each of the following areas

- o GC operation and maintenance for volatiles and semivolatiles analyses.
- o Mass spectral interpretation.
- o Extraction.
- o Pesticide/ Aroclors analysis.

B. Facilities

The adequacy of the facilities and equipment is of equal importance as the technical staff to accomplish the required work as specified by the EPA contract.

1. Sample Receipt Area

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

2. Storage Area

Sufficient refrigerator space to maintain unused EPA sample volume for 60 days after data submission and sample extracts for 365 days after data submission. Samples must be stored in an atmosphere demonstrated to be free from all potential contaminants. Volatile samples must be stored in a refrigerator used only for storage of volatile samples from this contract. Samples, sample extracts, and standards must be stored separately to prevent cross contamination. Semivolatile and pesticide/Aroclor standards and extracts must be stored separately from volatile standards and extracts.

3. Sample Preparation Area

Adequate, contamination-free, well-ventilated work space provided with the following:

a. Benches with chemical resistant tops, exhaust hoods. NOTE: Standards must be prepared in a glove box or isolated area.

b. Source of distilled or demineralized organic-free water.

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

C. Instrumentation

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

1. Primary Instrument Requirements for up to 200 Samples/Month Capacity

Fraction	No. of Instrument(s)	Type of Instrument
Volatiles	1	GC/MS/DS with purge and trap device
Semivolatiles	2	GC/MS/DS
Pesticides/ Aroclors	2	GC/EC/DS with dual column

This equates to three GC/MS/DS and 2 GC/EC/DS.

NOTE: For 300-400 Samples/Month Capacity, twice as much instrumentation is needed as is listed in item 1. For 500-600 Samples/Month Capacity, three times as much instrumentation is needed as is listed in item 1. For 700-800 Samples/Month Capacity, four times as much instrumentation is needed as is listed in item 1.

2. Secondary Instrument Requirements for up to 400 Samples/Month Capacity

The Contractor shall have the following instruments in place and operational at any one time as a back-up system;

Quantity	Instruments
One	GC/MS/DS
One	Purge and Trap Device
One	GC/EC/DS

Note: For over 400 samples/month capacity, twice as much instrumentation is needed as listed in item 2.

These instruments must be included in the bidder's inventory of equipment along with those in 1, above.

In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turn-around times.

3. Instrument Specifications

Instrument specifications are described in detail in the Statement of Work (SOW) in the following Exhibits:

- o Purge and trap device Exhibit D
- o GC/MS/DS Exhibit D
- o GC/EC/DS Exhibit D

D. Data Management and Handling

1. Hardware - Contractor shall have an IBM or IBM-compatible mini-computer or PC capable of recording required sample data on 5.25 inch double-sided, double-density 360 K-byte or high density 1.2 M-byte diskettes; or 3.5 inch double-sided, double-density 720 K-byte or 1.44 M-byte diskettes in ASCII text file format and in accordance with the file, record and field specifications listed in SOW, Exhibit H.

Other minimum requirements include:

- o Hard disk of at least 20 M-bytes.
- o Asynchronous, Hayes-compatible modem capable of at least 2,400 baud transmission speed. In addition, MNP level 5 compatibility is recommended.
- o Modem capable of at least 2,400 baud transmission speed which is compatible with the EPA Telecommunications Network.

2. Software - Software, utilized in generating, updating and quality controlling analytical databases and automated deliverables shall have the following additional capabilities:

- o Editing and updating databases.
- o QC of automated deliverables.
- o Controlled access using user ID and file password protection.

3. The Contractor shall also be able to submit reports and data packages as specified in the Statement of Work Exhibit B. To complete this task, the Contractor shall be required to provide space, tables and adequate copy machines to meet the contract requirements.

II. LABORATORY MANAGEMENT CAPABILITY

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

A. Technical Staff

Responsible for all technical efforts for the EPA contract. The Contractor shall have adequate number of technical personnel to meet the requirements of this contract.

B. Project Manager

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

C. Sample Custodian

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

D. Quality Assurance Officer

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

E. Document Control Officer

Responsible for all aspects of data deliverables: organization, packaging, copying, and delivery. Responsible for ensuring that all documents generated are placed in the Complete SDG File for inventory and are delivered to the appropriate EPA Regional personnel or other receiver.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

Table of Contents

SECTION I

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION
(For 35-Day Turnaround Contracts)

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

Item	No. Copies	Delivery Schedule	Distribution (1)	(2)	(3)	(4)
*****A. Standard Operating Procedures	3	60 days after contract award, and as required in Exhibit E.		X	X	X
*B. Sample Traffic Reports	1	3 days after receipt of last sample in Sample Delivery Group (SDG).**	X			
***C. Sample Data Summary Package	1	35 days after receipt of last sample in SDG.	X			
***D. Sample Data Package	2	35 days after receipt of last sample in SDG.	X		X	
E. Complete SDG File *	1	35 days after receipt of last sample in SDG.		X		
*****F. Quality Assurance Plan	3	60 days after contract award, and as required in Exhibit E.		As directed		
***G. Data in Computer-Readable Form	1	35 days after receipt of last sample in SDG.	X			

SECTION I

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION
(For 14-Day Turnaround Contracts)

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

Item	No. Copies	Delivery Schedule	Distribution (1)	(2)	(3)	(4)
*****A. Standard Operating Procedures	3	60 days after contract award, and as required in Exhibit E.		X	X	X
*B. Sample Traffic Reports	1	3 days after receipt of last sample in Sample Delivery Group (SDG).**	X			
***C. Sample Data Summary Package	1	14 days after receipt of last sample in SDG.	X			
***D. Sample Data Package	2	14 days after receipt of last sample in SDG.	X		X	
E. Complete SDG File *	1	14 days after receipt of last sample in SDG.		X		
*****F. Quality Assurance Plan	3	60 days after contract award, and as required in Exhibit E.		As directed		
***G. Data in Computer-Readable Form	1	14 days after receipt of last sample in SDG.	X			

Item	No. Copies	Delivery Schedule	Distribution (1)	(2)	(3)	(4)
H. GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO and/or EMSL/LV.	As Directed			
I. Extracts	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO or SMO.	As Directed			

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

* Also required in the Sample Data Summary Package.

** Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 14 days or less (7 days or less for 14-day data turnaound contracts) and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that all samples have been delivered. (See Exhibit A for further description).

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

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

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

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

Distribution Addresses:

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

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

(2) USEPA REGIONS:

The CLP Sample Management Office, acting on behalf of the Administrative Project Officer, will provide the Contractor with the list of addressees for the ten EPA Regions. SMO will provide the Contractor with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

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

For overnight delivery service, use street address:
944 E. Harmon, Executive Center
Las Vegas, NV 89109
ATTN: Data Audit Staff

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

SECTION II

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

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

All reports and documentation MUST BE as follows:

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

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

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

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

Section III of this Exhibit contains forms instructions to assist the Contractor in accurately providing the Agency with all required data. Section IV contains copies of the required data reporting forms in Agency-specified formats. Data elements with field parameters for reporting data in computer readable form are contained in Exhibit H.

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

A. Quality Assurance Plan and Standard Operating Procedures

See Exhibits E and F for requirements.

B. Sample Traffic Reports

Original Sample Traffic Report page marked "Lab Copy for Return to SMO" with lab receipt information and signed in original Contractor signature, for each sample in the Sample Delivery Group.

Traffic Reports (TRs) shall be submitted in Sample Delivery Group (SDG) sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

The SDG Cover Sheet shall contain the following items:

- Lab name.
- Contract number.
- Sample Analysis Price - full sample price from contract.
- Case Number.
- 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).

In addition, each Traffic Report must be clearly marked with the SDG Number, the sample number of the first sample in the SDG (as described in the following paragraph). This information should be entered below the Lab Receipt Date on the TR. In addition, the TR for the last sample received in the SDG must be clearly marked "SDG - FINAL SAMPLE."

The EPA sample number of the first sample received in the SDG is the SDG number. 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. The SDG number is also reported on all data reporting forms (see Section III, Forms Instruction Guide).

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

C. Sample Data Summary Package

As specified in the Delivery Schedule, one Sample Data Summary Package shall be delivered to SMO concurrently with delivery of other required sample data. The Sample Data Summary Package consists of copies of specified items from the Sample Data Package. These items are listed below and are described under part D, Sample Data 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 Sample Data Summary Package shall contain data for samples in one Sample Delivery Group of the Case, as follows:

1. SDG Narrative

2. By fraction (VOA, SV, PEST) and by sample within each fraction - tabulated target compound results (Form I) and tentatively identified compounds (Form I, TIC) (VOA and SV only)

3. By fraction (VOA, SV, PEST) - surrogate spike analysis results (Form II) by matrix (water and/or soil) and for soil, by concentration (low or medium)

4. By fraction (VOA, SV, PEST) - matrix spike/matrix spike duplicate results (Form III)

5. By fraction (VOA, SV, PEST) - blank data (Form IV) and tabulated results (Form I) including tentatively identified compounds (Form I, TIC) (VOA and SV only)

6. By fraction (VOA, SV only) - internal standard area data (Form VIII)

D. Sample Data Package

The Sample Data Package is divided into the five major units described below. The last three units are each specific to an analytical fraction (volatiles, semivolatiles, 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 analyses of all samples in one Sample Delivery Group, including field samples, 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.

1. SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; Case number; sample numbers in the Sample Delivery Group (SDG), differentiating between initial analyses and re-analyses; SDG number; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.

Whenever data from sample re-analyses are submitted, the Contractor shall state in the SDG Narrative for each re-analysis, whether it considers the re-analysis to be billable, and if so, why.

The Contractor must also include any problems encountered: both technical and administrative, the corrective actions taken, and resolution and an explanation for all flagged edits (i.e., manual edits) on quantitation lists.

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

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

Additionally, the SDG Narrative itself must be signed in original signature by the Laboratory Manager or his designee and dated.

2. Traffic Reports

A copy of the Sample Traffic Reports submitted in Item A for all of the samples in the SDG. The Traffic Reports shall be arranged in increasing EPA sample number order, considering both letters and numbering in ordering samples. Copies of the SDG cover sheet is to be included with the copies of the Traffic Reports.

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

3. Volatiles Data

a. QC Summary

(1) System Monitoring Compound Summary (Form II VOA)

(2) Matrix Spike/Matrix Spike Duplicate Summary (Form III VOA)

(3) Method Blank Summary (Form IV VOA)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank, by instrument.)

(4) GC/MS Instrument Performance Check (Form V VOA)

In chronological order; by instrument.

(5) Internal Standard Area and RT Summary (Form VIII VOA)

In chronological order; by instrument.

b. Sample Data

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

(1) Target Compound Results - Organic Analysis Data Sheet (Form I VOA).

Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C). The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (reference D.1). In the event that the Laboratory Manager cannot validate 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) Tentatively Identified Compounds (Form I VOA-TIC).

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found."

Form I VOA-TIC is the tabulated list of the highest probable match for up to 10 organic compounds that are not system monitoring compounds and are not listed in Exhibit C (TCL). It includes the CAS (Chemical Abstracts Service) Registry Number, tentative identification, and estimated concentration.

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

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

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

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

If automated data system procedures are used for preliminary identification and/or quantification of the target compounds, the complete data system report must 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, must be included in the sample data package in addition to the chromatogram.

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

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

(4) For each sample, by each compound identified, the following shall be included in the data package:

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

(b) Copies of mass spectra of organic compounds not listed in Exhibit C (TCL), Tentatively Identified Compounds, with associated best-match spectra (three best matches), labeled as in (4)(a) above.

c. Standards Data

(1) Initial Calibration Data (Form VI VOA) - in order by instrument, if more than one instrument used.

(a) VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five point) calibration, labeled as in b.(3) above. Spectra are not required.

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

(2) Continuing Calibration (Form VII VOA) - in order by instrument, if more than one instrument used.

(a) VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for all continuing (12 hour) calibrations, labeled as in b.(3) above. Spectra are not required.

(b) When more than one continuing calibration is performed, forms must be in chronological order, within fraction and instrument.

d. Raw QC Data

(1) BFB (for each 12-hour period, for each GC/MS system utilized)

(a) Bar graph spectrum, labeled as in b.(3) above.

(b) Mass listing, labeled as in b.(3) above.

(c) Reconstructed total ion chromatogram (RIC), labeled as in b.(3) above.

(2) Blank Data - in chronological order. NOTE: This order is different from that used for samples.

(a) Tabulated results (Form I VOA).

(b) Tentatively Identified Compounds (Form I VOA-TIC) even if none found.

(c) Reconstructed ion chromatogram(s) and quantitation

report(s) or legible facsimile (GC/MS), labeled as in b.(3) above.

(d) Target compound spectra with lab generated standard, labeled as in b.(4) above. Data systems which are incapable of dual display shall provide spectra in the following order:

- o Raw target compound spectra.
- o Enhanced or background subtracted spectra.
- o Laboratory generated standard spectra.

(e) GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in b.(4) above.

(f) Quantitation/Calculation of Tentatively Identified Compound(s) (TIC) concentrations.

(3) Matrix Spike Data

(a) Tabulated results (Form I VOA) of target compounds. Form I VOA-TIC not required.

(b) Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in b.(4) above. Spectra not required.

(4) Matrix Spike Duplicate Data

(a) Tabulated results (Form I VOA) of target compounds. Form I VOA-TIC not required.

(b) Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in b.(4) above. Spectra not required.

4. Semivolatiles Data

a. QC Summary

(1) Surrogate Percent Recovery Summary.(Form II SV)

(2) Matrix Spike/Matrix Spike Duplicate Summary (Form III SV)

(3) Method Blank Summary (Form IV SV)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

(4) GC/MS Instrument Performance Check (Form V SV)

In chronological order; by instrument.

(5) Internal Standard Area and RT Summary (Form VIII SV)

In chronological order; by instrument.

b. Sample Data

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

(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). The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (reference D.1). In the event that the Laboratory Manager cannot validate 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) Tentatively Identified Compounds (Form I SV-TIC).

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found".

Form I SV-TIC is the tabulated list of the highest probable match for up to 20 of the non-surrogate organic compounds not listed in Exhibit C (TCL). It includes the CAS (Chemical Abstracts Service) Registry Number, tentative identification, and estimated concentration.

(3) Reconstructed total ion chromatograms (RIC) for each sample, sample extract, standard, blank, and spiked sample.

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

- o EPA sample number.
- o Date and time of analysis.

- o GC/MS instrument ID.
- o Lab file ID.

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

If automated data system procedures are used for preliminary identification and/or quantification of the target compounds, the complete data system report must 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, must be included in the sample data package in addition to the chromatogram.

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

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

(4) For each sample, by each compound identified, the following shall be included in the data package:

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

(b) Copies of mass spectra of nonsurrogate organic compounds not listed in Exhibit C (TCL), Tentatively Identified Compounds with associated best-match spectra (three best matches), labeled as in (4)(a) above.

c. Standards Data

(1) Initial Calibration Data (Form VI SV-1, SV-2) - in order by instrument, if more than one instrument used.

(a) Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five point) calibration, labeled as in b.(3) above. Spectra are not required.

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

(2) Continuing Calibration (Form VII SV-1, SV-2) - in order by instrument, if more than one instrument used.

(a) Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for all continuing (12 hour) calibrations, labeled as in b.(3) above. Spectra are not required.

(b) When more than one continuing calibration is performed, forms must be in chronological order, by instrument.

(3) Semivolatile GPC Calibration Data - UV detector traces showing peaks that correspond to the compounds in the semivolatile GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. Do <u>not</u> include Form IX Pest-2, as the compounds used on that form are <u>not</u> appropriate for semivolatile sample extracts.

d. Raw QC Data

(1) DFTPP (for each 12-hour period, for each GC/MS system utilized)

(a) Bar graph spectrum, labeled as in b(3) above.

(b) Mass listing, labeled as in b.(3) above.

(c) Reconstructed total ion chromatogram (RIC), labeled as in b.(3) above.

(2) Blank Data - in chronological order by extraction date. NOTE: This order is different from that used for samples.

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

(b) Tentatively Identified Compounds (Form I SV-TIC) - even if none found.

(c) Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in b(3) above.

(d) Target compound spectra with lab generated standard, labeled as in b(4) above. Data systems which are incapable of dual display shall provide spectra in the following order:

- o Raw target compound spectra.
- o Enhanced or background subtracted spectra.
- o Laboratory generated standard spectra.

(e) GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in b(4) above.

(f) Quantitation/Calculation of Tentatively Identified Compound(s) (TIC) concentrations

(3) Matrix Spike Data

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

(b) Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in b(3) above. Spectra not required.

(4) Matrix Spike Duplicate Data

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

(b) Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in b(3) above. Spectra not required.

5. Pesticide/Aroclor Data

a. QC Summary

(1) Surrogate Percent Recovery Summary (Form II PEST)

(2) Matrix Spike/Matrix Spike Duplicate Summary (Form III PEST)

(3) Method Blank Summary (Form IV PEST)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

b. 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 increasing EPA sample number order, considering both letters and numbers in ordering samples.

(1) Target Compound Results - Organic Analysis Data Sheet (Form I PEST).

Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C). The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (reference D.1). In the event that the Laboratory Manager cannot validate 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) Copies of pesticide chromatograms.

All chromatograms must be labeled with the following information:

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

(3) Copies of pesticide chromatograms from second GC column, labeled as in (2) above.

(4) GC Integration report or data system printout.

(5) Manual work sheets.

(6) If pesticide/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 (TCL) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Compound names must be clearly marked on all spectra. For multicomponent pesticides/Aroclors confirmed by GC/MS, the Contractor shall submit mass spectra of 3 major peaks of multicomponent compounds from samples and standards.

c. Standards Data

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

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

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

(4) 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.

(5) 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.

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

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

(8) Pesticide GPC Calibration (Form IX PEST-2) - all GPC columns, in chronological order by calibration date.

(9) 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.

(10) 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.

(11) Chromatograms and data system printouts are required for all standards including the following:

- o Resolution Check Mixture.
- o Performance Evaluation Mixtures, all.
- o Individual Standard Mixture A, at three concentrations, each initial calibration.
- o Individual Standard Mixture B, at three concentrations, each initial calibration.
- o All multicomponent analytes (Toxaphene and Aroclors), each initial calibration.
- o All mid point concentrations of Individual Standard Mixtures A and B used for calibration verification.
- o Florisil cartridge check solution, all lots.
- o Pesticide GPC Calibration Check Solution, all calibrations relating to samples in the SDG.
- o All multicomponent analyte standards analyzed for confirmation.

(12) A printout of retention times and corresponding peak areas or peak heights must accompany each chromatogram. In addition, all chromatograms are required to be labeled with the following:

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

(13) Pesticide GPC Calibration Data - UV detector traces showing peaks that correspond to the compounds in the pesticide GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak.

d. Raw QC Data

(1) Blank Data - in chronological order, by type of blank (method, instrument, sulfur clean up). NOTE: This order is different from that used for samples.

(a) Tabulated results (Form I PEST).

(b) Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, labeled as in b.(2) above.

(2) Matrix Spike Data

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

(b) Chromatogram(s) and data system printout(s) (GC), labeled as in b.(2) above.

(3) Matrix Spike Duplicate Data

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

(b) Chromatogram(s) and data system printout(s) (GC), labeled as in b.(2) above.

E. Complete SDG File

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

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

1. Original Sample Data Package

2. A completed and signed Document Inventory Sheet (Form DC-2).

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

a. EPA Chain of Custody Record.

b. Airbills.

c. EPA Traffic Reports.

d. Sample Tags (if present) sealed in plastic bags.

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

   a. Form DC-1.

   b. Other receiving forms or copies of receiving logbooks.

   c. SDG Cover Sheet.

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:

   a. Original preparation and analysis forms or copies of preparation and analysis logbook pages.

   b. Internal sample and sample extract transfer chain-of-custody records.

   c. Screening records.

   d. All instrument output, including strip charts from screening activities.

6. All other original SDG-specific documents in the possession of the laboratory, including, but not limited to, the following documents:

   a. Telephone contact logs.

   b. Copies of personal logbook pages.

   c. All hand written case-specific notes.

   d. Any other case-specific documents not covered by the above.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to EPA, as well as copies that are altered in any fashion, are also deliverables to EPA. (Original to the Region, and copies to SMO and EMSL-LV).

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

F. Data in Computer-Readable Form

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

When submitted, diskettes shall be packaged and shipped in such a manner that the diskette(s) cannot be bent or folded, and will not be exposed to extreme heat or cold or any type of electromagnetic radiation. The diskette(s) must be included in the same shipment as the hardcopy data and shall, at a minimum, be enclosed in a diskette mailer. The data shall be recorded in ASCII, text file format, and shall adhere to the file, record and field specifications listed in Exhibit H, Data Dictionary and Format for Data Deliverables in Computer-Readable Format.

G. GC/MS Tapes

See Exhibit E for requirements.

H. 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 Sample Delivery Group (SDG) number. A logbook of stored extracts shall be maintained, listing EPA sample numbers and associated Case and SDG numbers.

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

SECTION III

FORMS INSTRUCTIONS

SECTION III

FORM INSTRUCTION GUIDE

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

A. General Information and Header Information

B. Organic Analysis Data Sheets (Form I, All Fractions)

C. System Monitoring Compound Recovery (Form II VOA)

D. Surrogate Recovery (Form II, SV and PEST)

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

F. Method Blank Summary (Form IV, All Fractions)

G. GC/MS Instrument Performance Check (Form V VOA and SV)

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

I. GC Initial Calibration Data (Form VI PEST-1, PEST-2, PEST-3, PEST-4)

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

K. GC/EC Calibration Verification Summary(Form VII PEST)

L. Internal Standard Area and RT Summary (Form VIII VOA and SV)

M. Pesticide Analytical Sequence (Form VIII PEST)

N. Pesticide Cleanup Procedures (Form IX PEST-1, PEST-2)

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

P. Sample Log-In Sheet (Form DC-1)

Q. Document Inventory Sheet (Form DC-2)

A. General Information and Header Information

The data reporting forms presented in Section IV have been designed in conjunction with the computer-readable data format specified in Exhibit H, Data Dictionary and Format for Data Deliverables in Computer-Readable Format. The specific length of each variable for computer-readable data transmission purposes is given in the Data Dictionary (Exhibit H). Information entered on these forms must not exceed the size of the field given on the form, including such laboratory-generated items as Lab Name and Lab Sample ID.

Note that on the hardcopy forms (Section IV), the space provided for entries is greater in some instances than the length prescribed for the variable as written to diskette (see Exhibit H). Greater space is provided on the hardcopy forms for the sake of visual clarity.

Values must be reported on the hardcopy forms according to the individual form instructions in this Section. For example, results for concentrations of VOA target compounds must be reported to two significant figures if the value is greater than or equal to 10. Values can be written to the diskette file in any format that does not exceed the field specification as given in the record specifications and discussed in "Record Structure", paragraph 5 of Exhibit H.

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

Alphabetic entries made onto 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, do not remove the underscores or vertical bar characters that delineate "boxes" on the forms. The only exception would be those underscores at the bottom of a "box" that are intended as a data entry line (for instance, on Form 2A, line 30, if data must be entered on line 30, it will replace the underscores).

Six pieces of information are common to the header sections of each data reporting form. They are Lab Name, Contract, Lab Code, Case No., SAS No., and SDG No. Except as noted below for SAS No., this information must be entered on every form and must match on every form.

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

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

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

The "Contract" is the number of the EPA contract under which the analyses were performed.

The "SDG No." is the Sample Delivery Group number. The Sample Delivery Group (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.

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

The other information common to most of the forms is the "EPA Sample No.". This number appears either in the upper righthand corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper 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.

All samples, matrix spikes, matrix spike duplicates, blanks, and standards shall be identified 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 following sample suffixes must be used:

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

Form VIII Pest requires that all samples analyzed in a given analytical sequence be listed, regardless of whether or not they are part of the SDG being reported. Therefore, use "ZZZZZ" as the EPA Sample No. for any sample analysis not associated with the SDG being reported.

For blanks and standards, the following identification scheme must be used as the "EPA Sample No."

1. Volatile blanks shall be identified as VBLK##.
2. Semivolatile blanks shall be identified as SBLK##.
3. Pesticide/Aroclor method blanks shall be identified as PBLK##.
4. Pesticide/Aroclor instrument blanks shall be identified as PIBLK##.

The "EPA Sample No." must be unique for each blank within an SDG. Within a fraction, a laboratory must achieve this by replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible identifiers for volatile blanks would be VBLK1, VBLK2, VBLKA1, VBLKB2, VBLK10, VBLKAB, etc.

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

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

STD - Indicates a standard.

- The concentration in ug/L of volatile standards (i.e., 010, 020, 050, 100, and 200) or the amount injected in ng for semivolatile standards (i.e., 020, 050, 080, 120, and 160).

As for the blank identifiers, these designations will have to be concatenated with other information to uniquely identify each standard.

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

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##

The permitted mixture of Aroclor 1016 and Aroclor 1260 shall be entered as AR1660##.

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

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

Several other pieces of information are common to many of the Data Reporting Forms. These include Matrix, Sample wt/vol, Level, Lab Sample ID, and Lab File ID.

For "Matrix", enter "SOIL" for soil/sediment samples, and enter "WATER" for water samples. NOTE: The matrix must be spelled out. Abbreviations such as "S" or "W" shall not be used.

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

For volatiles and semivolatiles, for "Level", enter the determination of concentration level made from the mandatory screening of soils. Enter as "LOW" or "MED", not "L" or "M". All water samples are "LOW" level and shall be entered as such. Note: There is no differentiation between low and medium soil samples for the Pesticide/Aroclor forms, and no level is entered on any of the these forms.

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

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

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

"GC Column" and "ID (mm)" are common to various other forms. These two fields are to be used to identify the stationary phase of the GC column (previously called GC Column ID), and the internal diameter of the GC column in millimeters (mm). For packed columns, convert the ID from inches to millimeters as necessary, and enter in the "ID" field.

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, as "page 1 of 2" and "page 2 of 2". If a second page is not required, number the page "page 1 of 1." NOTE: These forms are fraction-specific, and often matrix-specific within 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 within a fraction-specific and matrix-specific form.

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

B. Organic Analysis Data Sheet (Form I, All Fractions)

1. Form I VOA, Form I SV-1, Form I SV-2, Form I PEST

This form is used for tabulating and reporting sample analysis results for target compounds. If all fractions are not requested to be analyzed, only the pages specifically required must be submitted. If VOA analysis only is requested, Form I VOA and Form I VOA TIC must be submitted. If the pesticide/Aroclor analysis is the only analysis requested, only Form I Pest must be submitted for that sample.

Complete the header information on each page of Form I required, according to the instructions in Part A and as follows:

For soil samples analyzed for volatiles, for "% moisture not dec.", enter the nondecanted percent moisture. This is the only percent moisture determination made for volatiles, as the entire contents of the VOA vial are considered as the sample. For water samples, leave this field blank.

For soil samples analyzed for semivolatiles and pesticides/Aroclors, enter the values for the percent moisture determined during the analysis. In the field "decanted (Y/N)", 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 (i.e., 5%, not 5.3%). Leave these fields blank for Form I for water samples, method blanks, and instrument blanks.

For volatiles, enter the GC column identifier under "GC Column", and the internal diameters in millimeters (mm), to two decimal places, under "ID". For packed columns, convert the ID from inches to millimeters as necessary, and enter in this field.

For pesticides/Aroclors, enter the method of extraction as "SEPF" for separatory funnel, "CONT" for continuous liquid-liquid extraction, or "SONC" for sonication (soils only).

If gel permeation chromatography, "GPC Cleanup", was performed, enter "Y" for yes. Otherwise, enter "N" for no, if GPC was not performed. NOTE: GPC is <u>required</u> for all <u>soil</u> samples analyzed for semivolatiles and pesticides/Aroclors, therefore all soil sample forms will contain "Y" in this field.

For soil samples only, enter pH for semivolatiles and pesticides/Aroclors, reported to 0.1 pH units.

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

"Date Extracted" and "Date Analyzed" should be entered in a similar fashion. When continuous liquid-liquid extraction procedures are used for water samples, enter the date on which the procedure was <u>started</u> for "Date Extracted". If separatory funnel (pesticides only) or sonication procedures are used, enter the date on which the procedure was <u>completed</u>. For pesticide/Aroclor samples, the date of analysis should be the date of the first GC analysis performed. 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.

For volatiles, if a medium soil sample is analyzed, under the "Soil Extract Volume" enter total volume of the methanol extract in microliters. This volume includes any methanol not collected from the filtration of the extract through glass wool, and is typically 10000 uL, i.e., the 10 mL of methanol use for the extraction. If a medium soil sample is analyzed, enter also the volume of the methanol extract added to the reagent water in the purge tube and analyzed, under "Soil Aliquot Volume". Enter this volume in microliters (uL).

For semivolatiles, enter the actual volume of the most concentrated sample extract, in microliters, under "Concentrated Extract Volume". This volume typically will be 1000 uL (water), or 500 uL (water and soil) when GPC was performed. For pesticides/Aroclors, the volume of the most concentrated extract typically will be 10000 uL (water), or 5000 uL (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.

For semivolatiles and pesticides/Aroclors, enter the volume of the sample extract injected into the GC under "Injection Volume". Report this volume in microliters to one decimal place, i.e., 1.0 uL. Note: A 2.0 microliter injection is required for semivolatile analyses. If pesticide/Aroclors are analyzed using two GC columns connected to a single injection port, the "Injection Volume" must be entered as half the volume in the syringe, i.e., assume that the extract injected is evenly divided between the two columns.

If a sample or sample extract has been diluted for analysis, enter the "Dilution Factor" as a single number, not a fraction, such as "100.0," for a 1 to 100 dilution of the sample. Enter 0.1 for a concentration of 10 to 1. If a sample was not diluted, enter "1.0." Report dilution factors to one decimal place.

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

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

Report all pesticide/Aroclor results to two significant figures. The appropriate concentration units, ug/L or ug/kg, must be entered.

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

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

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

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

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution and for percent moisture. For example, 10 U for phenol in water if the sample final volume is the protocol-specified final volume. If a 1 to 10 dilution of extract is necessary, the reported limit is 100 U. For a soil sample, the value must also be adjusted for percent moisture. For example, if the sample had 24% moisture and a 1 to 10 dilution factor, the sample quantitation limit for phenol (330 U) would be corrected to

$$\frac{(330\ U)}{D} \times df \qquad \text{where } D = \frac{100 - \%\ \text{moisture}}{100}$$

and df = dilution factor

For example, at 24% moisture, $D = \frac{100-24}{100} = 0.76$

$$\frac{(330\ U)}{.76} \times 10 = 4300\ U$$ rounded to the appropriate number of significant figures

For semivolatile soil samples, the extract must be concentrated to 0.5 mL, and the sensitivity of the analysis is not compromised by the cleanup procedures. Similarly, pesticide samples subjected to GPC are concentrated to 5.0 mL. Therefore, the CRQL values in Exhibit C will apply to all samples, regardless of cleanup. However, if a sample extract cannot be concentrated to the protocol-specified volume (see Exhibit C), this fact must be accounted for in reporting the sample quantitation limit.

J - Indicates an estimated value. This flag is used under the following circumstances: 1) when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, 2) when the mass spectral and rentention 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, 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. Note: the "J" code is not used and the compound is not reported as being identified for pesticide/Aroclor results less than the CRQL, if the technical judgement of 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.). For example, if the sample quantitation limit is 10 ug/L, but a concentration of 3 ug/L is calculated, report it as 3J. The sample quantitation limit must be adjusted for dilution as discussed for the U flag.

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

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, instead use a laboratory-defined flag, discussed below.

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

E - This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. If one or more compounds have a response greater than full scale, except as noted in Exhibit D, the sample or extract must be diluted and re-analyzed according to the specifications in Exhibit D. All such compounds with a response greater than full scale should have the concentration flagged with an "E" on the Form I for the original analysis. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported on separate copies of Form I. The Form I for the diluted sample shall have the "DL" suffix appended to the sample number. NOTE: For total xylenes, where three isomers are quantified as two peaks, the calibration range of each peak should be considered separately, e.g., a diluted analysis is not required for total xylenes unless the concentration of the peak representing the single isomer exceeds 200 ug/L or the peak representing the two coeluting isomers on that GC column exceeds 400 ug/L. Similarly, if the two 1,2-Dichloroethene isomers coelute, a diluted analysis is not required unless the concentration exceeds 400 ug/L.

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

A - This flag indicates that a TIC is a suspected aldol-condensation product.

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

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

2. Form I VOA-TIC and Form I SV-TIC

Fill in all header information as above.

Report Tentatively Identified Compounds (TICs) including CAS number, compound name, retention time, and the estimated concentration (criteria for reporting TICs are given in Exhibit D, Section IV). Retention time must 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.

Include a Form I VOA-TIC or SV-TIC for every volatile and semivolatile fraction of every sample and method blank analyzed, even if no TICs are found. Total the number of TICs found, including aldol-condensation products (but see below), and enter this number in the "Number TICs found." If none were found, enter "0" (zero). Form I VOA-TIC or SV-TIC must be provided for every analysis, including required dilutions and reanalyses, even if no TICs are found.

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

Peaks that are suspected as aldol-condensation reaction products (i.e., 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 total "Number TICs found," but not counted as part of the 20 most intense non-target semivolatile compounds to be searched.

C. System Monitoring Compound Recovery (Form II VOA)

For volatiles, Form II is used to report the recoveries of the system monitoring compounds added to each volatile sample, blank, matrix spike, and matrix spike duplicate prior to analysis. The system monitoring compounds, previously termed volatile surrogates, 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 in soil samples. Soil sample recoveries are further differentiated by concentration level.

Complete the header information and enter EPA Sample Numbers as described in part A. For volatile soil samples only, specify the "level" as "LOW" or "MED", as on Form I. Do not mix low and medium level samples on one form. Complete one for each level. For each system monitoring compound, 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.

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

If the system monitoring compound(s) are diluted out in any analysis, enter the calculated recovery, or "0" (zero) if the system monitoring compound(s) is not detected, and flag the system monitoring compound(s) recoveries with a "D" in the column under the "#" symbol. Do not include results flagged "D" in the total number of recoveries for each sample outside the QC limits.

Number all pages as described in part A.

D. Surrogate Recovery (Form II, SV and PEST)

Form II is used to report the recoveries of the surrogate compounds added to each semivolatile and pesticide/Aroclor sample, blank, matrix spike, and matrix spike duplicate. For semivolatiles, Form II is matrix-specific as well as fraction-specific, so surrogate recoveries for semivolatile water samples are reported on a different version of Form II than semivolatile soil sample surrogate recoveries.

Complete the header information and enter EPA Sample Numbers as described in part A. For semivolatile soil samples only, specify the "level" as "LOW" or "MED", as on Form I. Do not mix low and medium level samples on one form. Complete one for each level. For each surrogate, report the percent recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.

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

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

The pesticide surrogate recoveries must be reported from both GC columns used for the analyses. Therefore, identify each GC column in the header, entering the stationary phase under "GC Column" (previously called GC Column ID), and the internal diameter (ID) of the column in millimeters under "ID". The assignment of columns as "1" and "2" is left to the discretion of the laboratory if 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" must be consistent across all the reporting forms. If the analysis is not performed by simultaneous injection, then the assignment of GC Column number should be based on the chronological order of the two analyses.

The pesticide surrogate recovery limits are only advisory, but the Contractor must flag those recoveries outside the advisory QC limits or diluted out, nonetheless. The total number outside the QC limits includes all values, regardless of GC column.

Number all pages as described in part A.

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

This form is used to report the results of the analyses of a matrix spike and matrix spike duplicate. The form is matrix-specific for volatiles and semivolatiles.

Complete the header information as instructed in Part A, including the EPA Sample Number for the matrix spike, without the suffixes MS or MSD.

For volatile and semivolatile soil samples, specify "level" as "LOW" or "MED", as on Form I. SDGs containing soil samples at both levels require MS/MSD at each level, therefore, for soils, prepare one form for each level.

All water samples are "LOW". Therefore, there is no MS/MSD for "medium level waters", and none shall be reported.

In the upper box in Form III, under "SPIKE ADDED", enter the calculated concentration in ug/L or ug/Kg (according to the matrix) that results from adding each spiked compound to the aliquot chosen for the matrix spike (MS). 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. Enter the "SAMPLE CONCENTRATION", 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). Under "MS CONCENTRATION", enter the actual concentration of each spike compound detected in the matrix spike aliquot. Calculate the percent recovery of each spike compound in the matrix spike aliquot to the nearest whole percent, according to Exhibit D, and enter under "MS % REC". Flag all

percent recoveries outside the QC limits with an asterisk (*). The asterisk must be placed in the last space of the percent recovery column, under the "#" symbol.

For pesticide/Aroclor matrix spikes and matrix spike duplicates, the concentration used for "MS CONCENTRATION" AND "MSD CONCENTRATION" must 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 is reported on Form I, and both concentrations are 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.

Complete the lower box on Form III in a similar fashion, using the results of the analysis of the matrix spike duplicate (MSD) aliquot. Calculate the relative percent difference (RPD) between the matrix spike recovery and the matrix spike duplicate recovery, and enter this value in the lower box under "% RPD". Report the relative percent difference to the nearest whole percent. 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.

Summarize the values outside the QC limits at the bottom of the page. No further action is required by the laboratory. Performance-based QC limits will be generated and updated from recovery and RPD data.

F. Method Blank Summary (Form IV, All Fractions)

This form summarizes the samples associated with each method blank analysis. A copy of the appropriate Form IV is required for each blank.

Complete the header information on Form IV as described in Part A. The "EPA Sample No." entered in the box at the top of Form IV shall be the same number entered on the Form I for the blank itself.

For volatile blanks, enter the "Instrument ID", "Date Analyzed" "Time Analyzed", "GC Column", "ID", and "Heated Purge (Y/N)". Volatile samples analyzed by the same purging technique, i.e., ambient purge, or heated purge, may be reported together on the same Form IV, if the same method blank applies to those samples. Thus, water samples and medium soil sample may be combined on a single form, if run with a single blank.

For semivolatile blanks, enter the "Instrument ID", "Date Analyzed", "Matrix" and "Level". All water blanks are "LOW". The "Time Analyzed" shall be in military time.

For pesticide/Aroclor blanks, enter the method of extraction as "SEPF" for separatory funnel, "SONC" for sonication, or "CONT" for continuous liquid-liquid extraction.

For pesticide/Aroclor blanks, there is no differentiation between medium and low level soil samples, so no "Level" is entered on this form.

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

If the samples associated with pesticide/Aroclor blank are subjected to sulfur cleanup, then the blank must also be subjected to sulfur cleanup. If sulfur cleanup is employed, enter "Y" in the "Sulfur Cleanup" field, else, enter "N". 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.

Pesticide/Aroclor contaminants must meet the identification criteria in Exhibit D PEST, which requires analysis of the blank on two different GC Columns. Therefore, enter the date, time and instrument ID of both analyses of the blank on the pesticide method blank summary. 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 must be consistent with the information reported on all other pesticide forms. Otherwise, (1) shall be the first analysis, and (2) the second. Identify the GC Column and internal diameter as described previously.

Enter "Lab File ID" only if GC/MS confirmation was attempted. Otherwise, leave blank.

For all three fractions, as appropriate, summarize the samples associated with a given method blank in the table below the header, entering EPA Sample Number and Lab Sample ID. For volatiles, enter the Lab File ID and time of analysis of each sample. For semivolatiles, enter Lab File ID and Date Analyzed. For pesticides/Aroclors, enter the dates of both analyses as Date Analyzed (1) and Date Analyzed (2), as discussed above.

Number all pages as described in part A.

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

This form is used to report the results of GC/MS instrument performance check (previously known as "Tuning") for volatiles and semivolatiles, and to summarize the date and time of analysis of samples, standards, blanks, matrix spikes, and matrix spike duplicates associated with each analysis of the instrument performance check solution.

Complete the header information as in Instruction A. Enter the "Lab

File ID" for the injection containing the instrument performance check solution (BFB for volatiles, DFTPP for semivolatiles). Enter the "Instrument ID". Enter the date and time of injection of the instrument performance check solution. Enter time as military time. For volatiles, indicate the purging method by entering "Y" for heated purge, and "N" for ambient temperature purge, as described previously. Water samples and medium soil sample extracts may be reported on the same Form V if analyzed together, as a single calibration may be applied to both sample types.

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

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

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

In the lower half of the form, list all samples, standards, 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 Part A for specific instructions for identifying standards and blanks. Enter "EPA Sample No.", "Lab Sample ID", "Lab File ID", "Date Analyzed", and "Time Analyzed" for all standards, samples, blanks, matrix spikes, and matrix spike duplicates.

The GC/MS instrument performance check solution must be analyzed again twelve hours from the time of injection of the instrument performance check solution (BFB or DFTPP) listed at the top of the form. In order to meet these requirements, a sample, standard, blank, matrix spike, or matrix spike duplicate must be injected within twelve hours of the injection of the instrument performance check solution.

Number all pages as described in Instruction A.

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

After a GC/MS system has undergone an initial five-point[1] calibration at the specific concentration levels described in Exhibits D and E, and after all initial calibration criteria have been met, the laboratory must complete and submit a Form VI for each volatile or semivolatile target compound initial calibration performed which is relevant to the samples, blanks, matrix spikes, matrix spike duplicates in the SDG, regardless of when that calibration was performed.

Complete all header information as in Part A. Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the initial calibration was performed. Enter "Instrument ID" and the date(s) of the calibration. If the calendar date changes during the calibration procedure, the inclusive dates should be given on Form VI. Enter the injection times of the first and last of the standards analyzed under "Calibration Times".

For volatiles, enter Heated Purge, and GC Column, ID, as on Form V.

Enter the "Lab File ID" 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. For volatiles, report the response factors for the system monitoring compounds in the calibration standards. For semivolatiles, report the response factors for all surrogate compounds in the calibration standards. The laboratory must report the relative standard deviation (%RSD) for <u>all</u> compounds.

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

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[1] 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 nanograms per injection is difficult. If a four-point calibration is performed for these compounds, leave RF20 blank.

In order to be used for the analysis of samples or sample extracts, the volatile and semivolatile initial calibration must meet the acceptance criteria for relative response factors outlined in Exhibits D and E. The compounds for which criteria have been developed for minimum RRF and maximum %RSD are indicated on the form by an "*". All other compounds must meet a minimum RRF of 0.010.

I. GC/EC Initial Calibration Data (Form VI PEST-1, PEST-2, PEST-3, PEST-4)

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

For the three analyses of Individual Standard Mixture A (low point, mid point, and high point), and the three analyses of Individual Standard Mixture B performed on each GC column during an initial calibration, complete one copy of Form VI for <u>each</u> GC column used.

Complete the header information as above. Enter the Instrument ID, GC Column, and ID as described previously. Enter the dates of analysis of the first and last of the six standards on each form under "Date(s) Analyzed". Under "Level (x low)", enter the concentration of the low point, mid point, and high point calibration standards as a multiplier of the low point. Therefore, for the low point, enter "1.0". The concentration of the mid point standard is specified in Exhibit D as four times the low point, therefore, enter "4.0" for "mid". The high point standard must be at least 16 times the low point, but may be higher, if that value lies within the linear range of the instrument, as specified in Exhibit D. Therefore, enter the appropriate multiplier to the high point standard concentration to one decimal place.

For each standard analyzed, enter the retention time of each applicable analyte in minutes and decimal minutes, under the appropriate concentration level. Calculate the mean retention time of each analyte from the three individual mixtures, and report it under "Mean RT". Calculate the retention time window for each analyte using the specifications in Exhibit D, and enter the lower limit of the window under RT Window "From", and the upper limit of the window under "To". 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.

For the six analyses of the Individual Standard Mixtures, the laboratory must also complete the calibration factor data on Form VI PEST-2. In a similar fashion as for the retention time data on Form VI PEST-1, prepare one form for each instrument and GC column used. Enter the concentration level of the standards in the same fashion as on Form VI PEST-1.

Enter the calibration factor for each compound in each of the

standards, and calculate a mean calibration and a percent relative standard deviation (%RSD), and enter on the form. As with surrogate retention times, the calibration factors are only required from Individual Mixture A analyses.

In order to be used for sample analyses, the %RSD of the calibration factors for each single component target compound must be less than or equal to 20.0 percent except as noted in the following. The %RSD of the calibration factors for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (but not surrogates) may exceed the 20.0 percent limit for %RSD, but these compounds must have a %RSD of less than of equal to 30.0 percent. These criteria apply to both GC columns.

For the multicomponent target compounds, the retention times, retention time windows, and calibration factor must be reported in a similar fashion for each single point calibration standard. For each multicomponent compound, the laboratory must select at least three peaks from each analyte, according to the specifications in Exhibit D. The retention 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.

Form VI is used also to report the results of analysis of the Resolution Check Solution that must begin each pesticide/Aroclor initial calibration sequence. The purpose of the Resolution Check Solution is to demonstrate for each initial calibration that the GC columns employed are capable of satisfactorily resolving the most difficult of the target analytes. One copy of Form VI PEST-4 is completed that covers both GC columns.

Complete the header information as described in Instruction A. Using the same assignment of first and second GC columns made for Form IV, enter the GC Column, ID, Instrument ID, and Date and Time Analyzed. Enter the "EPA Sample No." for the Resolution Check Standard. If simultaneous injections on a single GC are used, the EPA Sample No. may be the same for both Resolution Check Standards. If simultaneous injections were not used, use different suffixes to identify the standards.

In the boxes on the form, list each analyte, in retention time order, including both surrogate compounds. Thus, the order of analytes in the two boxes on a copy of 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.

Enter the retention time of each analyte from the analysis under "RT". Calculate the resolution between each pair of analytes according to the definition in Exhibit D PEST. The resolution is calculated as percentage of the height of the smaller of each pair of adjacent peaks. 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. In order for these GC columns to be used for pesticide/Aroclor analyses, the resolution of all pairs of peaks listed on this form must be greater than or equal to 60.0%.

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

For volatiles and semivolatiles, the Continuing Calibration Check form is used to report the calibration of the GC/MS system by the analysis of specific calibration standards. A Continuing Calibration Check form is required for each twelve (12) hour time period for both volatile and semivolatile target compound analyses.

The Contract laboratory must analyze calibration standards and meet all criteria outlined in Exhibits D and E for the minimum RRF and maximum percent difference between initial and continuing calibrations.

Complete all header information as in Instruction A. Enter instrument ID, date and time of continuing calibration, the Lab File ID of the continuing calibration standard, and dates and times of initial calibration (give inclusive dates if initial calibration is performed over more than one date). For volatiles, enter purge method and column as on Forms IV, V and VI. Using the appropriate Initial Calibration (volatile or semivolatile) fill in the average relative response factor (RRF) for each target compound, each system monitoring compound for volatiles, and each surrogate for semivolatiles. Report the relative response factor (RRF50) from the continuing calibration standard analysis. Calculate the Percent Difference (%D) for all compounds.

$$\% \text{ Difference} = \frac{\overline{RRF_i} - RRF_c}{\overline{RRF_i}} \times 100$$

Where

$\overline{RRF_i}$ = Average relative response factor from initial calibration.

RRF_c = Relative response factor from continuing calibration standard.

All semivolatile standards are analyzed at 50 total ng.

K. GC/EC Calibration Verfication Summary (Form VII PEST)

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

Complete the header information on each Form VII required according to the instructions in part A.

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

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

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

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

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

L. Internal Standard Area and RT Summary (Form VIII VOA and SV)

This form is used to summarize the peak areas and retention times of the internal standards added to all volatile and semivolatile samples, 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 must 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.

Complete the header information as in Instruction A. Enter the Lab File ID of the continuing calibration standard, as well as the date and time of analysis of the continuing calibration standard. 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 its Lab File ID and areas in place of those of a continuing calibration standard.

For volatiles, enter purge method and column, as on Forms IV, V, VI, and VII.

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 row labeled "12 HOUR STD". For each internal standard, calculate the upper limit of the area as the area of the particular standard plus 100% of its area (i.e., two times the area in the 12 HOUR STD box), and the lower limit of the area as the area of the internal standard minus 50% of its area (i.e., one half the area in the 12 HOUR STD box). Report these values in the boxes labeled "UPPER LIMIT" and "LOWER LIMIT" respectively. 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).

For each sample, blank, matrix spike, and matrix spike duplicate 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 above, flag that area with an asterisk (*). The asterisk must be placed in the far right hand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the retention time of any internal standard that is outside the limits with an asterisk.

Number all pages as described in Instruction A.

M. Pesticide Analytical Sequence (Form VIII Pest)

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

The laboratory shall complete all the header information as in Part A. Enter dates of analyses for the initial calibration, GC column, ID, and Instrument ID, as on Forms IV, VI, and VII.

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

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

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 Part A.

Form VIII PEST is required for each for each GC system and for each GC column used to analyze target pesticides/Aroclors.

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

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. In this fashion, problems with a lot of cartridges may be tracked across many samples.

Complete the header information on each Form IX required, according to the instructions in Part A.

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

Enter "GC Column" and "ID" for the GC column used to determine the recovery of the analytes in the Florisil cartridge check solution, under "GC Column (1)". If more than one GC column is used, enter the information for a second column under "GC Column (2)", etc., as discussed previously.

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

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

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

Number the Form IX pages as described in Part A.

Form IX PEST-2 summarizes the results of the calibration of the Gel Permeation Chromatography device (GPC) that must be used to process all soil sample extracts for pesticide/Aroclor analyses.

Complete all header information as in Part A. Enter an identifier for the GPC Column, and the date of calibration in the appropriate fields. Enter the two "GC Column" and "ID" fields, as discussed above.

For each of the pesticide matrix spike compounds listed in the box in the upper portion of the form, enter the amount of the spike added to the GPC column in ng, and the amount recovered, also in ng. 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 column under the "#" symbol.

For each sample in the data package that was subjected to GPC under this calibration, enter the EPA Sample No., Lab Sample ID, and the date of both analyses in the lower portion of the form.

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

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

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 copies of Form X are used for single component analytes and multicomponent analytes.

Copies of Form X are required for each sample, blank, matrix spike, and matrix spike duplicate in which target pesticides or Aroclors are detected. If none are detected in a given sample, no copy of Form X is required for that sample.

Complete the header information as in Instruction A. Enter the GC Column, and ID for each of the two columns, one as GC Column (1), the other as (2), as described previously. Enter the Instrument ID associated with each GC column directly below.

For each single component pesticide detected, enter the name of the compound under "ANALYTE" as it appears on Form I. Enter the retention times on each column of the compounds detected in the sample next to the appropriate column designation (1 or 2). Enter the retention time windows on each column from the initial calibration standard. These data must correspond with those on Form VI, and are entered in a similar manner. The lower value is entered under the "FROM" column, the upper value under the "TO" column.

Enter the concentration calculated from each GC column under the column labeled "CONCENTRATION". The units are the same as those used on Form I, ug/L for water samples, and ug/Kg for soil samples. However, do not enter any units on Form X. Calculate the percent difference between the concentrations entered on this form, using the equation below, and report it to a tenth of a percent under "%D".

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

Where,

$Conc_H$ = The higher of the two concentraiton for the target compound in question

$Conc_L$ = The lower of the two concentrations for the target compound in question

Note that using this equation will result in percent difference values that are always positive. The value will also be greater than a value calculated using the higher concentration in the denominator, however, given the likelihood of a positive interference raising the concentration determined on one GC column, this is a conservative approach to comparing the two concentrations.

The lower of the two concentrations is reported on Form I for each pesticide compound. The lower concentration is used because, if present, co-eluting 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.

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

Multicomponent analytes detected in samples are reported on a separate version of Form X. Complete the header information and Instrument 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 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 as described previously, if the mean concentrations from the two GC columns differ by more than 25.0 percent.

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

THE FOLLOWING ARE DOCUMENT CONTROL FORMS
(To be submitted as hardcopy only)

P. Sample Log-In Sheet (Form DC-1)

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

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

Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (i.e., Traffic Reports, Packing Lists), and airbills or airbill stickers in items 3-5 on Form DC-1. Specify if there is an airbill present or an airbill sticker in item 5 on Form DC-1. Record the airbill or sticker number in item 6.

Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking) and presence of absence of sample tags in items 7 and 8 on Form DC-1.

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

If there are no problems observed during receipt, sign and date (include time) Form DC-1, the chain-of-custody record, and Traffic Report, and write the sample numbers on Form DC-1. Record the appropriate sample tags and assigned laboratory numbers if applicable. The log-in date should be recorded at the top of Form DC-1 and the date and time of cooler receipt at the laboratory should be recorded in items 10 and 11. Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of Form DC-1. Sign and date the Sample Transfer block. Cross out unused columns and spaces.

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

Q. Document Inventory Sheet (Form DC-2)

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

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

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

SECTION IV

DATA REPORTING FORMS

1A

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: _____(uL) Soil Aliquot Volume: _____(uL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg)_____	Q
74-87-3	Chloromethane		
74-83-9	Bromomethane		
75-01-4	Vinyl Chloride		
75-00-3	Chloroethane		
75-09-2	Methylene Chloride		
67-64-1	Acetone		
75-15-0	Carbon Disulfide		
75-35-4	1,1-Dichloroethene		
75-34-3	1,1-Dichloroethane		
540-59-0	1,2-Dichloroethene (total)		
67-66-3	Chloroform		
107-06-2	1,2-Dichloroethane		
78-93-3	2-Butanone		
71-55-6	1,1,1-Trichloroethane		
56-23-5	Carbon Tetrachloride		
75-27-4	Bromodichloromethane		
78-87-5	1,2-Dichloropropane		
10061-01-5	cis-1,3-Dichloropropene		
79-01-6	Trichloroethene		
124-48-1	Dibromochloromethane		
79-00-5	1,1,2-Trichloroethane		
71-43-2	Benzene		
10061-02-6	trans-1,3-Dichloropropene		
75-25-2	Bromoform		
108-10-1	4-Methyl-2-Pentanone		
591-78-6	2-Hexanone		
127-18-4	Tetrachloroethene		
79-34-5	1,1,2,2-Tetrachloroethane		
108-88-3	Toluene		
108-90-7	Chlorobenzene		
100-41-4	Ethylbenzene		
100-42-5	Styrene		
1330-20-7	Xylene (total)		

1B

EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

.b Name:_____ Contract:_____

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

.trix: (soil/water)_____ Lab Sample ID: _____

.mple wt/vol: _____(g/mL)____ Lab File ID: _____

:vel: (low/med) _____ Date Received: _____

Moisture: _____ decanted: (Y/N)___ Date Extracted:_____

ıncentrated Extract Volume: _____(uL) Date Analyzed: _____

ıjection Volume: _____(uL) Dilution Factor: _____

'C Cleanup: (Y/N)___ pH:____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg)_____	Q
108-95-2	Phenol		
111-44-4	bis(2-Chloroethyl)ether		
95-57-8	2-Chlorophenol		
541-73-1	1,3-Dichlorobenzene		
106-46-7	1,4-Dichlorobenzene		
95-50-1	1,2-Dichlorobenzene		
95-48-7	2-Methylphenol		
108-60-1	2,2'-oxybis(1-Chloropropane)		
106-44-5	4-Methylphenol		
621-64-7	N-Nitroso-di-n-propylamine		
67-72-1	Hexachloroethane		
98-95-3	Nitrobenzene		
78-59-1	Isophorone		
88-75-5	2-Nitrophenol		
105-67-9	2,4-Dimethylphenol		
111-91-1	bis(2-Chloroethoxy)methane		
120-83-2	2,4-Dichlorophenol		
120-82-1	1,2,4-Trichlorobenzene		
91-20-3	Naphthalene		
106-47-8	4-Chloroaniline		
87-68-3	Hexachlorobutadiene		
59-50-7	4-Chloro-3-methylphenol		
91-57-6	2-Methylnaphthalene		
77-47-4	Hexachlorocyclopentadiene		
88-06-2	2,4,6-Trichlorophenol		
95-95-4	2,4,5-Trichlorophenol		
91-58-7	2-Chloronaphthalene		
88-74-4	2-Nitroaniline		
131-11-3	Dimethylphthalate		
208-96-8	Acenaphthylene		
606-20-2	2,6-Dinitrotoluene		
99-09-2	3-Nitroaniline		
83-32-9	Acenaphthene		

1C

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: _____(uL) Date Analyzed: _____

Injection Volume: _____(uL) Dilution Factor: _____

GPC Cleanup: (Y/N)___ pH:____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/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		
7005-72-3	4-Chlorophenyl-phenylether		
86-73-7	Fluorene		
100-01-6	4-Nitroaniline		
534-52-1	4,6-Dinitro-2-methylphenol		
86-30-6	N-Nitrosodiphenylamine (1)		
101-55-3	4-Bromophenyl-phenylether		
118-74-1	Hexachlorobenzene		
87-86-5	Pentachlorophenol		
85-01-8	Phenanthrene		
120-12-7	Anthracene		
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	Dibenz(a,h)anthracene		
191-24-2	Benzo(g,h,i)perylene		

(1) - Cannot be separated from Diphenylamine

1D

PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

ab Name:_____ Contract:_____

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

atrix: (soil/water)_____ Lab Sample ID: _____

ample wt/vol: _____(g/mL)____ Lab File ID: _____

Moisture: _____ decanted: (Y/N)___ Date Received: _____

xtraction: (SepF/Cont/Sonc) _____ Date Extracted:_____

oncentrated Extract Volume: _____(uL) Date Analyzed: _____

njection Volume: _____(uL) Dilution Factor: _____

PC Cleanup: (Y/N)___ pH:____ Sulfur Cleanup: (Y/N) ___

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg)_____	Q
319-84-6	alpha-BHC		
319-85-7	beta-BHC		
319-86-8	delta-BHC		
58-89-9	gamma-BHC (Lindane)		
76-44-8	Heptachlor		
309-00-2	Aldrin		
1024-57-3	Heptachlor epoxide		
959-98-8	Endosulfan I		
60-57-1	Dieldrin		
72-55-9	4,4'-DDE		
72-20-8	Endrin		
33213-65-9	Endosulfan II		
72-54-8	4,4'-DDD		
1031-07-8	Endosulfan sulfate		
50-29-3	4,4'-DDT		
72-43-5	Methoxychlor		
53494-70-5	Endrin ketone		
7421-36-3	Endrin aldehyde		
5103-71-9	alpha-Chlordane		
5103-74-2	gamma-Chlordane		
8001-35-2	Toxaphene		
12674-11-2	Aroclor-1016		
11104-28-2	Aroclor-1221		
11141-16-5	Aroclor-1232		
53469-21-9	Aroclor-1242		
12672-29-6	Aroclor-1248		
11097-69-1	Aroclor-1254		
11096-82-5	Aroclor-1260		

1E

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: _____(uL) Soil Aliquot Volume: _____(uL)

Number TICs found: _____ CONCENTRATION UNITS: (ug/L or ug/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.					

1F

EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

ıb Name:_____ Contract:_____

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

ıtrix: (soil/water)_____ Lab Sample ID: _____

ımple wt/vol: _____(g/mL)____ Lab File ID: _____

:vel: (low/med) _____ Date Received: _____

Moisture: _____ decanted: (Y/N)___ Date Extracted:_____

›ncentrated Extract Volume: _____(uL) Date Analyzed: _____

ıjection Volume: _____(uL) Dilution Factor: _____

›C Cleanup: (Y/N)___ pH:____

ıumber TICs found: _____ CONCENTRATION UNITS:
(ug/L or ug/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.				

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

D System Monitoring Compound diluted out

2B

SOIL VOLATILE SYSTEM MONITORING COMPOUND RECOVERY

b Name:_____ Contract:_____

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

vel:(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

D System Monitoring Compound diluted out

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

2D

SOIL SEMIVOLATILE SURROGATE RECOVERY

ıb Name:_____ Contract:_____

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

ːvel:(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								

	ADVISORY QC LIMITS
TCX = Tetrachloro-m-xylene	(60-150)
DCB = Decachlorobiphenyl	(60-150)

\# Column to be used to flag recovery values
* Values outside of QC limits
D Surrogate diluted out

2F

SOIL PESTICIDE SURROGATE RECOVERY

ab Name:_____ Contract:_____

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

: 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								

	ADVISORY QC LIMITS
TCX = Tetrachloro-m-xylene	(60-150)
DCB = Decachlorobiphenyl	(60-150)

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

3A

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: _____

_____

3B

SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

ıb Name:_____ Contract:_____

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

.trix 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

)D:_____ out of _____ outside limits
)ike Recovery:_____ out of _____ outside limits

)MMENTS: _____
_____

3C

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
1,4-Dichlorobenzene					36- 97
N-Nitroso-di-n-prop.(1)					41-116
1,2,4-Trichlorobenzene					39- 98
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
1,4-Dichlorobenzene					28	36- 97
N-Nitroso-di-n-prop.(1)					38	41-116
1,2,4-Trichlorobenzene					28	39- 98
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: _____

_____

3D

SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

b Name:_____ Contract:_____

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

trix 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
1,4-Dichlorobenzene					28-104
N-Nitroso-di-n-prop.(1)					41-126
1,2,4-Trichlorobenzene					38-107
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 RPD	QC LIMITS REC.
Phenol					35	26- 90
2-Chlorophenol					50	25-102
1,4-Dichlorobenzene					27	28-104
N-Nitroso-di-n-prop.(1)					38	41-126
1,2,4-Trichlorobenzene					23	38-107
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

)D:_____ out of _____ outside limits
)ike Recovery:_____ out of _____ outside limits

)MMENTS: _____

_____

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 RPD	QC LIMITS REC.
gamma-BHC (Lindane)					15	56-123
Heptachlor					20	40-131
Aldrin					22	40-120
Dieldrin					18	52-126
Endrin					21	56-121
4,4'-DDT					27	38-127

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

* Values outside of QC limits

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

COMMENTS: _____

3F

SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

ıb Name:_____ Contract:_____

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

ıtrix 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

PD:_____ out of _____ outside limits
ɔike Recovery:_____ out of _____ outside limits

OMMENTS: _____
_____

4A

VOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

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

SEMIVOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

ıb Name:_____ Contract:_____

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

ıb File ID: _____ Lab Sample ID: _____

ıstrument ID: _____ Date Extracted:_____

ıtrix: (soil/water) _____ Date Analyzed: _____

ːvel:(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				

ɔMMENTS:

_____

_____

ıge __ of __

4C

PESTICIDE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

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

Lab Sample ID: _____ Lab File ID: _____

Matrix: (soil/water) _____ Extraction: (SepF/Cont/Sonc) _____

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)

b Name:_____ Contract:_____

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

b File ID: _____ BFB Injection Date:_____

strument ID: _____ BFB Injection Time:_____

: Column: _____ ID: _____(mm) Heated Purge: (Y/N) ___

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

(IS 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

ab Name:_____ Contract:_____

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

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

eated Purge: (Y/N) ___ Calibration Times: _____ _____

: Column: _____ ID: _____(mm)

LAB FILE ID: RRF10 =_____ RRF20 =_____
RRF50 =_____ RRF100=_____ RRF200=_____

COMPOUND		RRF10	RRF20	RRF50	RRF100	RRF200	$\overline{RRF}$	% RSD	
Chloromethane									
Bromomethane	*								*
Vinyl Chloride	*								*
Chloroethane									
Methylene Chloride									
Acetone									
Carbon Disulfide									
1,1-Dichloroethene	*								*
1,1-Dichloroethane	*								*
1,2-Dichloroethene (total)									
Chloroform	*								*
1,2-Dichloroethane	*								*
2-Butanone									
1,1,1-Trichloroethane	*								*
Carbon Tetrachloride	*								*
Bromodichloromethane	*								*
1,2-Dichloropropane									
cis-1,3-Dichloropropene	*								*
Trichloroethene	*								*
Dibromochloromethane	*								*
1,1,2-Trichloroethane	*								*
Benzene	*								*
trans-1,3-Dichloropropene	*								*
Bromoform	*								*
4-Methyl-2-Pentanone									
2-Hexanone									
Tetrachloroethene	*								*
1,1,2,2-Tetrachloroethane	*								*
Toluene	*								*
Chlorobenzene	*								*
Ethylbenzene	*								*
Styrene	*								*
Xylene (total)	*								*
Toluene-d8									
Bromofluorobenzene	*								*
1,2-Dichloroethane-d4									

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

6B

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

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

6C

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

ab Name:_____ Contract:_____

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

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

Calibration Times: _____ _____

LAB FILE ID: RRF20 =_____ RRF50 =_____
RRF80 =_____ RRF120=_____ RRF160=_____

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Diethylphthalate							
4-Chlorophenyl-phenylether	*						*
Fluorene	*						*
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine (1)							
4-Bromophenyl-phenylether	*						*
Hexachlorobenzene	*						*
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	*						*
Dibenz(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.

6D

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 LOW	MID	HIGH	MEAN RT	RT WINDOW 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 epoxide, ±0.07 minutes for all other compounds, except ±0.10 minutes for Decachlorobiphenyl.

6E

PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

ab Name:_____ Contract:_____

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

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

: 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.

RSD must be less than or equal 20.0 % for all compounds except the
rrogates, where %RSD must be less than or equal to 30.0%. Up to
o target compounds, but not surrogates, may have %RSD greater than
).0% but less than or equal to 30.0%.

6F

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	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				

* Denotes required peaks

6G

PESTICIDE ANALYTE RESOLUTION SUMMARY

ıb Name:_____ Contract:_____

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

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

'A Sample No. (Standard 1): _____ Lab Sample ID (1): _____

ıte Analyzed (1): _____ Time Analyzed (1): _____

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

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

'A Sample No. (Standard 2): _____ Lab Sample ID (2): _____

ıte Analyzed (2): _____ Time Analyzed (2): _____

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

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

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):_____ _____

Heated Purge: (Y/N) ___ Init. Calib. Times: _____ _____

GC Column: _____ ID: _____(mm)

COMPOUND	$\overline{RRF}$	RRF50	MIN RRF	%D	MAX %D
Chloromethane					
Bromomethane			0.100		25.0
Vinyl Chloride			0.100		25.0
Chloroethane					
Methylene Chloride					
Acetone					
Carbon Disulfide					
1,1-Dichloroethene			0.100		25.0
1,1-Dichloroethane			0.200		25.0
1,2-Dichloroethene (total)					
Chloroform			0.200		25.0
1,2-Dichloroethane			0.100		25.0
2-Butanone					
1,1,1-Trichloroethane			0.100		25.0
Carbon Tetrachloride			0.100		25.0
Bromodichloromethane			0.200		25.0
1,2-Dichloropropane					
cis-1,3-Dichloropropene			0.200		25.0
Trichloroethene			0.300		25.0
Dibromochloromethane			0.100		25.0
1,1,2-Trichloroethane			0.100		25.0
Benzene			0.500		25.0
trans-1,3-Dichloropropene			0.100		25.0
Bromoform			0.100		25.0
4-Methyl-2-Pentanone					
2-Hexanone					
Tetrachloroethene			0.200		25.0
1,1,2,2-Tetrachloroethane			0.500		25.0
Toluene			0.400		25.0
Chlorobenzene			0.500		25.0
Ethylbenzene			0.100		25.0
Styrene			0.300		25.0
Xylene (total)			0.300		25.0
Toluene-d8					
Bromofluorobenzene			0.200		25.0
1,2-Dichloroethane-d4					

All other compounds must meet a minimum RRF of 0.010.

7B

SEMIVOLATILE CONTINUING CALIBRATION CHECK

ıb Name:_____ Contract:_____

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

ıstrument ID: _____ Calibration Date:_____ Time:_____

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

Init. Calib. Times: _____ _____

COMPOUND	$\overline{RRF}$	RRF50	MIN RRF	%D	MAX %D
Phenol			0.800		25.0
bis(2-Chloroethyl)ether			0.700		25.0
2-Chlorophenol			0.800		25.0
1,3-Dichlorobenzene			0.600		25.0
1,4-Dichlorobenzene			0.500		25.0
1,2-Dichlorobenzene			0.400		25.0
2-Methylphenol			0.700		25.0
2,2'-oxybis(1-Chloropropane)					
4-Methylphenol			0.600		25.0
N-Nitroso-di-n-propylamine			0.500		25.0
Hexachloroethane			0.300		25.0
Nitrobenzene			0.200		25.0
Isophorone			0.400		25.0
2-Nitrophenol			0.100		25.0
2,4-Dimethylphenol			0.200		25.0
bis(2-Chloroethoxy)methane			0.300		25.0
2,4-Dichlorophenol			0.200		25.0
1,2,4-Trichlorobenzene			0.200		25.0
Naphthalene			0.700		25.0
4-Chloroaniline					
Hexachlorobutadiene					
4-Chloro-3-methylphenol			0.200		25.0
2-Methylnaphthalene			0.400		25.0
Hexachlorocyclopentadiene					
2,4,6-Trichlorophenol			0.200		25.0
2,4,5-Trichlorophenol			0.200		25.0
2-Chloronaphthalene			0.800		25.0
2-Nitroaniline					
Dimethylphthalate					
Acenaphthylene			1.300		25.0
2,6-Dinitrotoluene			0.200		25.0
3-Nitroaniline					
Acenaphthene			0.800		25.0
2,4-Dinitrophenol					
4-Nitrophenol					
Dibenzofuran			0.800		25.0
2,4-Dinitrotoluene			0.200		25.0

All other compounds must meet a minimum RRF of 0.010.

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):_____ _____

Init. Calib. Times: _____ _____

COMPOUND	$\overline{RRF}$	RRF50	MIN RRF	%D	MAX %D
Diethylphthalate					
4-Chlorophenyl-phenylether			0.400		25.0
Fluorene			0.900		25.0
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine (1)					
4-Bromophenyl-phenylether			0.100		25.0
Hexachlorobenzene			0.100		25.0
Pentachlorophenol			0.050		25.0
Phenanthrene			0.700		25.0
Anthracene			0.700		25.0
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
Dibenz(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.

7D

PESTICIDE CALIBRATION VERIFICATION SUMMARY

ıb Name:_____ Contract:_____

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

: Column: _____ ID: _____(mm) Init. Calib. Date(s):_____ _____

'A Sample No.(PIBLK): _____ Date Analyzed :_____

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

'A Sample No.(PEM): _____ Date Analyzed :_____

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

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

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

ɔmbined % breakdown (1): _____

: LIMITS:

RPD of amounts in PEM must be less than or equal to 25.0%

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

Endrin breakdown must be less than or equal to 20.0%

Combined breakdown must be less than or equal to 30.0%

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.(INDA): _____ 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)	RPD
alpha-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Endosulfan I						
Dieldrin						
Endrin						
4,4'-DDD						
4,4'-DDT						
Methoxychlor						
Tetrachloro-m-xylene						
Decachlorobiphenyl						

EPA Sample No.(INDB): _____ Date Analyzed :_____

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

INDIVIDUAL MIX B COMPOUND	RT	RT WINDOW FROM	RT WINDOW TO	CALC AMOUNT (ng)	NOM AMOUNT (ng)	RPD
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						

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

8A
VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

ıb Name:_____ Contract:_____

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

ıb File ID (Standard): _____ Date Analyzed:_____

ıstrument ID: _____ Time Analyzed:_____

: Column: _____ ID: _____(mm) Heated Purge: (Y/N) ___

	IS1(BCM) AREA #	RT #	IS2(DFB) AREA #	RT #	IS3(CBZ) AREA #	RT #
12 HOUR STD						
UPPER LIMIT						
LOWER LIMIT						
EPA SAMPLE NO.						
)1						
)2						
)3						
)4						
)5						
)6						
)7						
)8						
)9						
10						
.1						
.2						
.3						
.4						
.5						
.6						
.7						
.8						
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.: _____

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

Instrument ID: _____ Time Analyzed:_____

	EPA SAMPLE NO.	IS1(DCB) AREA #	RT #	IS2(NPT) AREA #	RT #	IS3(ANT) AREA #	RT #
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
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 internal standard area values 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.: _____

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

Instrument ID: _____ Time Analyzed:_____

	IS4(PHN) AREA #	RT #	IS5(CRY) AREA #	RT #	IS6(PRY) AREA #	RT #
12 HOUR STD						
UPPER LIMIT						
LOWER LIMIT						
EPA SAMPLE NO.						
01						
02						
03						
04						
05						
06						
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 internal standard area values 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

ıb Name:_____ Contract:_____

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

.orisil Cartridge Lot Number: _____ Date of Analysis: _____

: 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

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

Lab Name:_____ Contract:_____

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

GPC Column: _____ Calibration Date: _____

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

COMPOUND	SPIKE ADDED (ng)	SPIKE RECOVERED (ng)	% REC #	QC. LIMITS REC.
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 values with an asterisk
* Values outside of QC limits

THIS GPC CALIBRATION 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

ıb Name:_____ Contract:_____

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

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

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

: 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					

ıge __ 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 FROM	TO	CONCENTRATION	MEAN CONCENTRATION	%D
______	1						
	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						
______	1						
	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						
______	1						
	2						
	3						
COLUMN 1	4						
	5						
	1						
	2						
	3						
COLUMN 2	4						
	5						

At least 3 peaks 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 List Present/Absent*
5. Airbill Airbill/Sticker Present/Absent*
6. Airbill No.: _____ _____
7. Sample Tags Present/Absent*

   Sample Tag Numbers Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/ Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: _____
11. Time Received: _____

Sample Transfer

Fraction: _____ _____

Area #: _____ _____

By: _____ _____

On: _____ _____

	CORRESPONDING		
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Reviewed By: _____

Date: _____

Logbook No.: _____

Logbook Page No: _____

FORM DC-1

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____
CITY/STATE _____

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

CONTRACT NO. _____
SOW NO. _____

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

		PAGE NOs FROM	TO	CHECK 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)			____	____
	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)			____	____
	RICs and Quan Reports for all Standards			____	____
	Continuing Calibration Data (Form VII VOA)			____	____
	RICs and Quantitation Reports for all Standards			____	____
	d. Raw QC Data				
	BFB	____	____	____	____
	Blank Data	____	____	____	____
	Matrix Spike/Matrix Spike Duplicate Data	____	____	____	____

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

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

	PAGE NOs FROM	TO	CHECK LAB	EPA
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 performed)			___	___
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			___	___
Semivolatile GPC Calibration Data-UV detector traces			___	___
d. Raw QC Data				
DFTPP	___	___	___	___
Blank Data	___	___	___	___
Matrix Spike/Matrix Spike Duplicate Data	___	___	___	___
Pesticides				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II PEST)	___	___	___	___
MS/MSD Duplicate Summary (Form III PEST)	___	___	___	___
Method Blank Summary (Form IV PEST)	___	___	___	___

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

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

	PAGE NOs FROM	TO	CHECK LAB	EPA
6. Pesticides (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 confirmed 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)			____	_____
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			____	_____
Pesticide GPC calibration data - UV detector traces			____	_____
d. Raw QC Data				
Blank Data	____	____	____	_____
Matrix Spike/Matrix Spike Duplicate Data	____	____	____	_____

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

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

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

Comments: _____
_____
_____
_____

leted by: _____ _____ _____
LP Lab) (Signature) (Printed Name/Title) (Date)

ted by: _____ _____ _____
PA) (Signature) (Printed Name/Title) (Date)

FORM DC-2-4

EXHIBIT C

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

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

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

The CRQL values listed on the following pages are based on the analysis of samples according the specifications given in Exhibit D. For each fraction and matrix, a brief synopsis of the sampling handling and analysis steps is given, along with an example calculation for the CRQL value. All CRQL values are rounded to two significant figures. For soil samples, the moisture content of the samples is not considered in these example calculations.

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

Volatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
1. Chloromethane	74-87-3	10	10	1200	(50)
2. Bromomethane	74-83-9	10	10	1200	(50)
3. Vinyl Chloride	75-01-4	10	10	1200	(50)
4. Chloroethane	75-00-3	10	10	1200	(50)
5. Methylene Chloride	75-09-2	10	10	1200	(50)
6. Acetone	67-64-1	10	10	1200	(50)
7. Carbon Disulfide	75-15-0	10	10	1200	(50)
8. 1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9. 1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10. 1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11. Chloroform	67-66-3	10	10	1200	(50)
12. 1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13. 2-Butanone	78-93-3	10	10	1200	(50)
14. 1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15. Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16. Bromodichloromethane	75-27-4	10	10	1200	(50)
17. 1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18. cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19. Trichloroethene	79-01-6	10	10	1200	(50)
20. Dibromochloromethane	124-48-1	10	10	1200	(50)
21. 1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22. Benzene	71-43-2	10	10	1200	(50)
23. trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24. Bromoform	75-25-2	10	10	1200	(50)
25. 4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26. 2-Hexanone	591-78-6	10	10	1200	(50)
27. Tetrachloroethene	127-18-4	10	10	1200	(50)
28. Toluene	108-88-3	10	10	1200	(50)
29. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
30. Chlorobenzene	108-90-7	10	10	1200	(50)
31. Ethyl Benzene	100-41-4	10	10	1200	(50)
32. Styrene	100-42-5	10	10	1200	(50)
33. Xylenes (Total)	1330-20-7	10	10	1200	(50)

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

Note that the CRQL values listed on the preceding page may not be those

specified in previous CLP Statements of Work. These values are set at concentrations in the sample equivalent to the concentration of the lowest calibration standard specified in Exhibit D VOA. Lower quantitation limits may be achievable for water samples by employing the Statement of Work for Low Concentration Water for Organic Analyses.

VOLATILES

Water Samples

A 5 mL volume of water is purged with an inert gas at ambient temperature. The volatiles are trapped on solid sorbents, and desorbed directly onto the GC/MS. For a sample with compound X at the CRQL of 10 ug/L:

(10 ug/L) (5 mL) (10^{-3} L/mL) = 50 x 10^{-3} ug = 50 ng on the GC column

Low Level Soil/Sediment Samples

A 5 g aliquot of the soil/sediment sample is added to a volume of water in a purge tube, heated, and purged with an inert gas. The volatiles are trapped, and later desorbed directly onto the GC/MS. For a sample with compound X at the CRQL of 10 ug/Kg:

(10 ug/Kg) (5 g) (10^{-3} Kg/g) = 50 x 10^{-3} ug = 50 ng on the GC column

Medium Level Soil/Sediment Samples

A 4 g aliquot of soil/sediment is extracted with 10 mL of methanol, and filtered through glass wool. Only 1 mL of the methanol extract is taken for screening and analysis. Based on the results of a GC/FID screen, an aliquot of the methanol extract is added to 5 mL of reagent water and purged at ambient temperature. The largest aliquot of extract considered in Exhibit D is 100 uL. For a sample with compound X at the CRQL of 1200 ug/Kg:

(1200 ug/Kg) (4 g) (10^{-3} Kg/g) = 4800 x 10^{-3} ug = 4800 ng

This material is contained in the 10 mL methanol extract:

(4800 ng)/ 10 mL = 480 ng/mL

Of which, 100 uL are purged from the reagent water.

(480 ng/mL) (100 uL) (10^{-3} mL/uL) = 480 x 10^{-1} ng) 50 ng on the GC column

Note that for both low and medium soil/sediment samples, while it may affect the purging efficiency, the volume of reagent water used in the purging process does not affect the calculations.

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

Semivolatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
34. Phenol	108-95-2	10	330	10000	(20)
35. bis(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)
36. 2-Chlorophenol	95-57-8	10	330	10000	(20)
37. 1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)
38. 1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)
39. 1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)
40. 2-Methylphenol	95-48-7	10	330	10000	(20)
41. 2,2'-oxybis (1-Chloropropane)#	108-60-1	10	330	10000	(20)
42. 4-Methylphenol	106-44-5	10	330	10000	(20)
43. N-Nitroso-di-n-propylamine	621-64-7	10	330	10000	(20)
44. Hexachloroethane	67-72-1	10	330	10000	(20)
45. Nitrobenzene	98-95-3	10	330	10000	(20)
46. Isophorone	78-59-1	10	330	10000	(20)
47. 2-Nitrophenol	88-75-5	10	330	10000	(20)
48. 2,4-Dimethylphenol	105-67-9	10	330	10000	(20)
49. bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)
50 2,4-Dichlorophenol	120-83-2	10	330	10000	(20)
51. 1,2,4-Trichlorobenzene	120-82-1	10	330	10000	(20)
52. Naphthalene	91-20-3	10	330	10000	(20)
53. 4-Chloroaniline	106-47-8	10	330	10000	(20)
54. Hexachlorobutadiene	87-68-3	10	330	10000	(20)
55. 4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)
56. 2-Methylnaphthalene	91-57-6	10	330	10000	(20)
57. Hexachlorocyclopentadiene	77-47-4	10	330	10000	(20)
58. 2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)
59. 2,4,5-Trichlorophenol	95-95-4	25	800	25000	(50)
60. 2-Chloronaphthalene	91-58-7	10	330	10000	(20)
61. 2-Nitroaniline	88-74-4	25	800	25000	(50)
62. Dimethylphthalate	131-11-3	10	330	10000	(20)
63. Acenaphthylene	208-96-8	10	330	10000	(20)
64. 2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
65. 3-Nitroaniline	99-09-2	25	800	25000	(50)
66. Acenaphthene	83-32-9	10	330	10000	(20)
67. 2,4-Dinitrophenol	51-28-5	25	800	25000	(50)
68. 4-Nitrophenol	100-02-7	25	800	25000	(50)

Previously known by the name bis(2-Chloroisopropyl) ether

Semivolatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
69. Dibenzofuran	132-64-9	10	330	10000	(20)
70. 2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71. Diethylphthalate	84-66-2	10	330	10000	(20)
72. 4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73. Fluorene	86-73-7	10	330	10000	(20)
74. 4-Nitroaniline	100-01-6	25	800	25000	(50)
75. 4,6-Dinitro-2-methylphenol	534-52-1	25	800	25000	(50)
76. N-nitrosodiphenylamine	86-30-6	10	330	10000	(20)
77. 4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
78. Hexachlorobenzene	118-74-1	10	330	10000	(20)
79. Pentachlorophenol	87-86-5	25	800	25000	(50)
80. Phenanthrene	85-01-8	10	330	10000	(20)
81. Anthracene	120-12-7	10	330	10000	(20)
82. Carbazole	86-74-8	10	330	10000	(20)
83. Di-n-butylphthalate	84-74-2	10	330	10000	(20)
84. Fluoranthene	206-44-0	10	330	10000	(20)
85. Pyrene	129-00-0	10	330	10000	(20)
86. Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87. 3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88. Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89. Chrysene	218-01-9	10	330	10000	(20)
90. bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	(20)
91. Di-n-octylphthalate	117-84-0	10	330	10000	(20)
92. Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93. Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
94. Benzo(a)pyrene	50-32-8	10	330	10000	(20)
95. Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	(20)
96. Dibenz(a,h)anthracene	53-70-3	10	330	10000	(20)
97. Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

SEMIVOLATILES

Water Samples

A 1 L volume of water is extracted in a continuous liquid-liquid extractor with methylene chloride at a pH of approximately 2. This extract is reduced in volume to 1.0 mL, and a 2 uL volume is injected onto the GC/MS for analysis. For a sample with compound X at the CRQL of 10 ug/L:

(10 ug/L) (1 L) = 10 ug in the original extract

When the extract is concentrated, this material is contained in the 1 mL concentrated extract, of which 2 uL are injected into the instrument:

(10 ug/mL) (2 uL) (10^{-3} mL/uL) = 20 x 10^{-3} ug = 20 ng on the GC column

Low Soil Samples

A 30 g soil sample is extracted three times with methylene chloride/acetone at ambient pH, by sonication. The extract is reduced in volume to 1.0 mL, and a 2 uL volume is injected onto the GC/MS for analysis. For a sample with compound X at the CRQL of 330 ug/Kg:

(330 ug/Kg) (30 g) (10^{-3} Kg/g) = 9900 x 10^{-3} ug = 9.9 ug

When the sample extract is to be subjected to Gel Permeation Chromatography (required) to remove high molecular weight interferences, the volume of the extract is initially reduced to 10 mL. This 10 mL is put through the GPC column, and only 5 mL are collected off the GPC. That 5 mL volume is reduced to 0.5 mL prior to analysis. Therefore:

(9.9 ug/10 mL) (5 mL) = 4.95 ug

This material is contained in the 0.5 mL extract, of which 2 uL are injected into the instrument:

(4.95 ug/0.5 mL) (2 uL) (10^{-3} mL/uL) = 1.98 x 10^{-2} ug) 20 ng on the GC column

Medium Soil Samples

A 1 g soil sample is extracted once with 10 mL of methylene chloride/acetone, which is filtered through glass wool to remove particles of soil. The filtered extract is then subjected to GPC clean up, and only 5 mL of extract are collected after GPC. This extract is reduced in volume to 0.5 mL, of which 2 uL are injected onto the GC/MS. For a sample with compound X at the CRQL of 10,000 ug/Kg:

(10,000 ug/Kg) (1g) (10^{-3} Kg/g) = 10 ug

(continued)

Semivolatiles, Medium Soil, continued -

This material is contained in the 10 mL extract, of which only 5 mL are collected after GPC:

(10 ug) (5 mL/10mL) = 5 ug

The volume of this extract is reduced to 0.5 mL, of which 2 uL are injected into the instrument:

(5 ug/0.5 mL) (2 uL) (10^{-3} mL/uL) = 20 x 10^{-3} ug = 20 ng on the GC column

Eight semivolatile compounds are calibrated using only a four point initial calibration, with the lowest standard at 50 ng. Therefore, the CRQL values for these eight compounds are 2.5 times higher for all matrices and levels.

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

Pesticides/Aroclors	CAS Number	Quantitation Limits* Water ug/L	Soil ug/Kg	On Column (pg)
98. alpha-BHC	319-84-6	0.05	1.7	5
99. beta-BHC	319-85-7	0.05	1.7	5
100. delta-BHC	319-86-8	0.05	1.7	5
101. gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
102. Heptachlor	76-44-8	0.05	1.7	5
103. Aldrin	309-00-2	0.05	1.7	5
104. Heptachlor epoxide	1024-57-3	0.05	1.7	5
105. Endosulfan I	959-98-8	0.05	1.7	5
106. Dieldrin	60-57-1	0.10	3.3	10
107. 4,4'-DDE	72-55-9	0.10	3.3	10
108. Endrin	72-20-8	0.10	3.3	10
109. Endosulfan II	33213-65-9	0.10	3.3	10
110. 4,4'-DDD	72-54-8	0.10	3.3	10
111. Endosulfan sulfate	1031-07-8	0.10	3.3	10
112. 4,4'-DDT	50-29-3	0.10	3.3	10
113. Methoxychlor	72-43-5	0.50	17.0	50
114. Endrin ketone	53494-70-5	0.10	3.3	10
115. Endrin aldehyde	7421-36-3	0.10	3.3	10
116. alpha-Chlordane	5103-71-9	0.05	1.7	5
117. gamma-Chlordane	5103-74-2	0.05	1.7	5
118. Toxaphene	8001-35-2	5.0	170.0	500
119. Aroclor-1016	12674-11-2	1.0	33.0	100
120. Aroclor-1221	11104-28-2	2.0	67.0	200
121. Aroclor-1232	11141-16-5	1.0	33.0	100
122. Aroclor-1242	53469-21-9	1.0	33.0	100
123. Aroclor-1248	12672-29-6	1.0	33.0	100
124. Aroclor-1254	11097-69-1	1.0	33.0	100
125. Aroclor-1260	11096-82-5	1.0	33.0	100

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

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

PESTICIDES/AROCLORS

Water Samples

A 1 L volume of water is extracted three times with methylene chloride or by a continuous liquid-liquid extractor. This extract is reduced in volume to approximately 3-5 mL, and diluted up to 10.0 mL with clean solvent. When Gel Permeation Chromatography is performed, only 5 of the 10 mL of extract are collected after GPC.

Regardless of whether GPC is performed, either 1.0 or 2.0 mL of the 10.0 mL of the original extracts are taken through the remaining clean up steps (Florisil and sulfur removal). The volume taken through Florisil cleanup and the final volume of the extract after the clean up steps depends on the requirements of the autosampler. If the autosampler can handle 1.0 mL final extract volumes, this is the volume taken through Florisil and the final volume. If the autosampler cannot reliably handle 1.0 mL volumes, the volume is 2.0 mL. When using an autosampler, the injection volume may be 1.0 or 2.0 uL. Manual injections must use a 2.0 uL injection volume.

For a sample with compound X at the CRQL of 0.05 ug/L and an autosampler requiring a 1.0 mL volume:

(0.05 ug/L) (1 L) = 0.05 ug in the original extract

This material is contained in the 10.0 mL of extract:

(0.05 ug)/ (10.0 mL) = 0.005 ug/mL

Of which, only 1.0 mL is carried through the remaining clean up steps. For a final extract volume of 1.0 mL and a 1 uL injection volume:

(0.005 ug/mL)(1 uL)(10^{-3} mL/uL) = 5 x 10^{-6} ug = 5 pg on the GC column

Soil Samples

There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides/Aroclors. A 30 g soil sample is extracted three times with methylene chloride/acetone by sonication. The extract is reduced in volume to 10.0 mL and subjected to Gel Permeation Chromatography. After GPC, only 5.0 mL of extract are collected. However, as with the water sample described above, either 1.0 or 2.0 mL of that extract are subjected to the other clean up steps, so no loss of sensitivity results from the use of GPC. From this point on, the soil sample extract is handled in the same fashion as the extract of a water sample. For a sample with compound X at the CRQL of 1.7 ug/Kg:

(1.7 ug/Kg) (30 g) (10^{-3} Kg/g) = 51 x 10^{-3} ug = 51 ng in the original extract

This material is contained in the 10.0 mL of extract:

(51 ng)/ 10 mL = 5.1 ng/mL

(continued)

Pesticides/Aroclors, continued -

Of which, only 1.0 or 2.0 mL are carried through the remaining cleanup steps. For a final extract volume of 1.0 mL and a 1 uL injection volume:

$(5.1\ ng/mL)(1\ uL)(10^{-3}\ mL/uL) = 5.1 \times 10^{-3}\ ng$) 5 pg on the GC column.

For either water or soil samples, if the autosampler used requires a 2.0 mL final volume, the concentration in the 10.0 mL of extract above remains the same.

Using a 2 uL injection volume, twice the total number of picograms are injected onto the GC column. However, because the injection volume must be the same for samples and standards, twice as much material is injected onto the column during calibration, and thus the amount of compound X injected from the sample extract is equivalent to the amount of compound X injected from the calibration standard, regardless of injection volume.

If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2 uL.

EXHIBIT D

ANALYTICAL METHODS
FOR VOLATILES

Table of Contents

SECTION I

INTRODUCTION

The analytical methods that follow are designed to analyze water, sediment and soil from hazardous waste sites for the organic compounds on the Target Compounds List (TCL, see Exhibit C). The methods are based on EPA Method 624 (Purgeables).

The methods are divided into the following sections: sample preparation, screening, and analysis. Sample preparation covers sample storage, sample holding times, and medium level sample extraction. As described in the screening section, a portion of a hexadecane extract may be screened on a gas chromatograph with appropriate detectors to determine the concentration level of organics. The analysis section contains the GC/MS analytical methods for organics. The purge and trap technique, including related sample preparation, is included in the analysis section because GC/MS operation and the purge and trap technique are interrelated.

1. Method for the Determination of Volatile (Purgeable) Organic Compounds

1.1 Scope and Application

This method covers the determination of the target volatile (purgeable) organics as listed in Exhibit C. The contract required quantitation limits are also listed in Exhibit C. The method includes an optional hexadecane screening procedure. The extract is screened on a gas chromatograph/flame ionization detector (GC/FID) to determine the approximate concentration of organic constituents in the sample. The actual analysis is based on a purge and trap gas chromatographic/mass spectrometer (GC/MS) method. For soil/sediment samples, the purge device is heated.

1.2 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 dehydrogenate during storage or analysis.
- Tetrachloroethane and 1,1-Dichloroethane can be degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.
- 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 BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve Bromoform response.

SECTION II

SAMPLE PREPARATION AND STORAGE

PART A - SAMPLE STORAGE AND HOLDING TIMES

1. Procedures for Sample Storage

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 sample data package to the Agency. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants and in a refrigerator used only for storage of purgeable samples received under this contract.

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

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

2. Contract Required Holding Times

Analysis of water and soil/sediment samples must be completed within 10 days of validated time of sample receipt (VTSR).

PART B - PROTOCOLS FOR HEXADECANE EXTRACTION OF VOLATILES FROM WATER AND SOIL/SEDIMENT FOR OPTIONAL SCREENING

1. Summary of Method

1.1 Matrices

1.1.1 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 ug/L
non-halogenated aromatics	40- 50
halogenated methanes	800-1000
halogenated ethanes	400- 500

1.1.2 Soil/sediment - 40 mL of reagent water are added to 10 g (wet weight) of soil 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.

1.2 The hexadecane extraction and screening protocols for purgeables are optional. These protocols are included to aid the analyst in deciding whether a sample is low or medium level. The use of these or other screening protocols could prevent saturation of the purge and trap system and/or the GC/MS system. It is recommended that these or other screening protocols be used, particularly if there is some doubt about the level of organics in a sample. This is especially true in soil/sediment analysis.

2. Limitations

These extraction and preparation procedures were developed for rapid screening of water 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.

3. Interferences

3.1 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. Apparatus and Materials

4.1 Vials and caps - 2 mL for GC auto sampler.

4.2 Volumetric flask - 50 mL with ground glass stopper.

4.3 Pasteur pipets - disposable.

4.4 Centrifuge tube - 50 mL with ground glass stopper or Teflon-lined screw cap.

4.5 Balance - analytical, capable of accurately weighing ± 0.0001 g.

5. Reagents

5.1 Hexadecane and methanol - pesticide residue analysis grade or equivalent.

5.2 Reagent water - defined as water in which an interferent is not observed at the CRQL of each parameter of interest.

5.3 Standard mixture #1 containing benzene, toluene, ethyl benzene and xylene. Standard mixture #2 containing n-nonane and n-dodecane.

5.3.1 Stock standard solutions (1.00 ug/uL) - prepared from pure standard materials or purchased as certified solutions.

5.3.1.1 Prepare stock standard solutions by accurately weighing about 0.0100 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.

5.3.1.2 Transfer the stock standard solutions into multiple Teflon-sealed screw-cap vials. Store, with no head-space, 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 gases or reactive compounds such as styrene must be replaced after two months, or sooner, if comparison with quality control check samples indicates a problem.

5.3.2 Prepare working standards of mixtures #1 and #2 at 100 ng/uL of each compound in methanol. Store these solutions as in 5.3.1.2 above.

6. Sample Extraction

6.1 Water

6.1.1 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 2.0 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.

6.1.2 Transfer approximately 1 mL of the hexadecane layer to a 2.0 mL GC vial. If an emulsion is present after shaking the sample, break it by doing the following:

- o Pulling the emulsion through a small plug of Pyrex glass wool packed in a pipet, or
- o Transferring the emulsion to a centrifuge tube and centrifuging for several minutes.

6.1.3 Add 200 uL each of working standard #1 and #2 to separate 40 mL portions of reagent water in 50 mL volumetric flasks. Follow steps 6.1.1 - 6.1.2, starting with the immediate addition of 2.0 mL of hexadecane.

6.2 Soil/Sediment

6.2.1 Add approximately 10 g of soil (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 one minute. Centrifuge the capped flask briefly. Quickly transfer supernatant water to a 50 mL volumetric flask equipped with a ground-glass stopper.

6.2.2 Follow 6.1, starting with the addition of 2.0 mL of hexadecane.

7. Sample Analysis

The sample is ready for GC/FID screening. Proceed to Section III, Optional Screening of Hexadecane Extracts for Volatiles.

SECTION III

OPTIONAL SCREENING OF HEXADECANE EXTRACTS FOR VOLATILES

1. Summary of Method

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.

2. Apparatus and Materials

2.1 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.

2.1.1 Above-described GC, equipped with flame ionization detector.

2.1.2 GC column - 3 m 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.

3. Reagents

Hexadecane - pesticide residue analysis grade or equivalent.

4. Limitations

4.1 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 extract from the water, and therefore will not be detected by the GC/FID.

4.2 Following are two options for interpreting the GC/FID chromatogram.

4.2.1 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 a water, or whether to use the low or medium level GC/MS purge and trap methods if the sample is a soil/sediment (see Table 1, paragraph 6.2.1.3 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.

4.2.2 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, paragraph 6.2.1.3 for guidance). All purgeables of interest have retention times less than the n-dodecane.

5. Extract Screening

5.1 External standard calibration - Standardize the GC/FID each 12 hr. shift for half scale response. This is done by injecting 1-5 uL of the extracts that contain approximately 10 ng/uL each of mix #1 and mix #2 compounds, as prepared in 5.3, Section II, Part B. Use the GC conditions specified in paragraph 2.1.2.

5.2 Inject the same volume of hexadecane extract as the extracted standard mixture in 5.1. Use the GC conditions specified in 2.1.2.

6. Analytical Decision Point

6.1 Water

6.1.1 Compare the chromatograms of the hexadecane extract of the sample with those of the reagent blank and extract of the standard.

6.1.1.1 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.

6.1.1.2 If peaks are present prior to the n-dodecane and the aromatics are distinguishable, follow Option A (4.2.1).

6.1.1.3 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 $\leq$3% of the n-nonane, analyze a 5 mL water sample by purge and trap GC/MS. If any peaks are $\geq$3% of the n-nonane, measure the peak height or area of the major peak and calculate the dilution factor as follows:

$$\frac{\text{peak area of sample major peak}}{\text{peak area of n-nonane}} \times 50 = \text{dilution factor}$$

The water sample will be diluted using the calculated factor just prior to purge and trap GC/MS analysis.

6.2 Soil/Sediment

6.2.1 Compare the chromatograms of the hexadecane extract of the sample with those of the reagent blank and extract of the standard.

6.2.1.1 If no peaks are noted, other than those also in the reagent blank, analyze a 5 g sample by low level GC/MS.

6.2.1.2 If peaks are present prior to the n-dodecane and the aromatics are distinguishable, follow Option A (4.2.1) and the concentration information in Table 1, paragraph 6.2.1.3, to determine whether to analyze by low or medium level method.

6.2.1.3 If peaks are present prior to the n-dodecane but the aromatics are absent or indistinguishable, and using Option B as follows, calculate a factor using the following formula:

$$\frac{\text{peak area of sample major peak}}{\text{peak area of n-nonane}} = \text{X Factor}$$

Table 1 - Determination of GC/MS Purge & Trap Method

X Factor	Analyze by	Approximate Concentration Range* (ug/kg)
0-1.0	low level method	0-1,000
>1.0	medium level method	>1,000

* This concentration range is based on the response of aromatics to GC/FID. When comparing GC/FID responses, the concentration for halomethanes is 20x higher, and that for haloethanes is 10x higher.

6.3 Sample Analysis

Proceed to Section IV, GC/MS Analysis of Volatiles.

SECTION IV

GC/MS ANALYSIS
OF VOLATILES

1. Summary of Methods

1.1 Water Samples

An inert gas is bubbled through a 5 mL sample contained in a specifically designed purging chamber at ambient temperature. The purgeables 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 purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging.

1.2 Soil/Sediment Samples

1.2.1 Low level - 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 purgeables 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 purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

1.2.2 Medium level - a measured amount of soil is extracted with methanol. A portion of the methanol extract 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 purgeables 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 purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

2. Interferences

2.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Exhibit E. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

2.2 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. A holding blank prepared from reagent water and carried through the holding period and the analysis protocol serves as a check on such contamination. **One holding blank per case must be analyzed. Data must be retained by the laboratory and be made available for inspection during on-site laboratory evaluations.**

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

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

3. Apparatus and Materials

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

3.1 Micro syringes - 25 uL and larger, 0.006 inch ID needle.

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

3.3 Syringe - 5 mL, gas-tight with shut-off valve.

3.4 Balance - analytical, capable of accurately weighing ± 0.0001 g, and a top-loading balance capable of weighing ± 0.1 g

3.5 Glassware

3.5.1 Bottle - 15 mL, screw cap, with Teflon cap liner.

3.5.2 Volumetric flasks - class A with ground-glass stoppers.

3.5.3 Vials - 2 mL for GC autosampler.

3.6 Purge and trap device - consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

3.6.1 The sample purger 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.

3.6.2 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: 15 cm of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh) and 8 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15, or equivalent).

3.6.3 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.

3.6.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph.

3.6.5 A heater or heated bath capable of maintaining the purge device at 40°C ± 1°C is to be used for low level soil analysis, but <u>not</u> for waters or medium level soil analyses.

3.7 GC/MS system

3.7.1 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 paragraph 3.6 and have all required accessories including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. If capillary columns are to be used (see below), the column oven must be cooled to 10°C; therefore, a subambient oven controller is required.

3.7.2 Gas Chromatography Columns

3.7.2.1 Packed Columns - 6 ft long x 0.1 in ID glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent.

NOTE: Capillary columns may be used for analysis of volatiles, as long as the Contractor uses the instrumental parameters in EPA Method 524.2 as guidelines, uses the internal standards and surrogates specified in this contract, and demonstrates that the analysis meets all of the performance and QA/QC criteria contained in this contract.

3.7.2.2 Capillary Columns

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

  OR

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

3.7.3 Mass Spectrometer - must be capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng of p-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet. The instrument conditions required for the acquisition of the BFB mass spectrum are given in paragraph 6.4.3.

NOTE: BFB criteria must be met before any sample extracts are analyzed. Any samples analyzed when BFB criteria have not been met will require reanalysis at no cost to the Agency. To ensure sufficient precision of mass spectral data, the MS scan rate should allow acquisition of at least five spectra while a sample compound elutes from the GC. The 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.

3.7.4 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.

3.7.5 Data system - a computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion 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 most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

3.7.6 Magnetic tape storage device - must be capable of recording data and must be suitable for long-term, off-line storage.

3.7.8 pH paper - wide range.

4. Reagents

4.1 Reagent water - defined as water in which an interferent is not observed at or above the CRQL of the parameters of interest.

4.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

4.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

4.1.3 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.

4.2 Sodium thiosulfate - (ACS) granular.

4.3 Methanol - pesticide quality or equivalent.

5. Standards

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

5.2 Stock Standard Solutions

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

5.2.1 Prepare stock standard solutions by placing about 9.8 mL of methanol into a 10.0 mL ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes, or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.2.2 Add the assayed reference material as described below.

5.2.2.1 If the compound is a liquid, using a 100 uL 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.

5.2.2.2 If the compound is a gas at room temperature, fill a 5 mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The gas will rapidly dissolve in the methanol. This may also be accomplished by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side-arm relief valve and direct a gentle stream of the reference standard into the methanol meniscus.

5.2.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. For non-gaseous and compounds, calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 97 percent or greater, the weight may be used, without correction, to calculate the concentration of the stock standard. If the compound purity is assayed to be less than 97 percent, the weight must be corrected when calculating the concentration of the stock solution. For gaseous compounds, calculate the concentration in micrograms per microliter, using the Ideal Gas Law, taking into account the temperature and pressure conditions within the laboratory.

5.2.4 Prepare fresh stock standards every two months for gases or for reactive compounds such as styrene. All other stock standards for non-gases/non-reactive purgeable compounds must be replaced after six months, or sooner if standard has degraded or evaporated.

5.3 Secondary Dilution Standards

5.3.1 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.

5.3.2 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.

5.4 Working Standards

5.4.1 Instrument Performance Check Solution - p-Bromofluorobenzene (BFB)

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

5.4.2 Calibration Standard Solution

Prepare the working calibration standard solution containing all of the purgeable target compounds in methanol. The recommended concentration of the target compounds is 100 ug/mL. Prepare fresh working calibration standard solutions weekly, or sooner, if solutions have degraded or evaporated.

5.4.3 Internal Standard Spiking Solution

Prepare an internal standard spiking solution containing Bromochloromethane, Chlorobenzene-d_5, and 1,4-Difluorobenzene in methanol at the concentration of 25.0 ug/mL for each internal standard. Add 10 uL of this spiking solution into 5.0 mL of sample or calibration standard for a concentration of 50 ug/L. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

5.4.4 System Monitoring Compound (SMC) Spiking Solution

Prepare a system monitoring compound spiking solution containing Toluene-d_8, p-Bromofluorobenzene, and 1-2-Dichloroethane-d_4 in methanol at a concentration of 25.0 ug/mL. Add 10.0 uL of this spiking solution into 5.0 mL of sample, for a concentration of 50 ug/L. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

5.4.5 Volatile Matrix Standard Spiking Solution

5.4.5.1 Prepare a spiking solution in methanol that contains the following compounds at a concentration of 25.0 ug/mL: 1,1-Dichloroethene, Trichloroethene, Chlorobenzene, Toluene, and Benzene. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

5.4.5.2 Matrix spikes are analyzed in duplicate; therefore, add an aliquot of this solution to each of two portions from one sample chosen for spiking.

5.5 Aqueous Calibration Standard Solutions

5.5.1 Prepare five aqueous initial calibration standard solutions containing all of the purgeable target compounds and system monitoring compounds at the 10, 20, 50, 100, and 200 ug/L levels. Note: These are not the same levels as have been used in previous Statements of Work. It is required that all three Xylene isomers (o-, p-, and m-Xylene) be present in the calibration standards at concentrations of each isomer equal to that of the other target compounds (i.e., 10, 20, 50, 100, and 200 ug/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.

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

5.5.2.1 Volumetric flask - add an appropriate volume of working calibration standard solution to an aliquot of reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcohol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the head of the flask.

5.5.2.2 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 5.0 mL minus the amount of calibration standard to be added. Withdraw the plunger slightly and add an appropriate volume of working calibration standard through the valve bore of the syringe. Close the valve and invert three times.

5.5.3 The 50 ug/L aqueous calibration standard solution is the continuing calibration standard.

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

5.6 Storage of Standards

5.6.1 Store the stock standards in Teflon-sealed screw-cap bottles with zero headspace at -10°C to -20°C. Protect the standards from light. Once one of the bottles containing the stock standard solution has been opened, it may be used for no longer than one week.

5.6.2 Store secondary dilution standards in Teflon-sealed screw-cap bottles with minimal headspace at -10°C to -20°C. Protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to preparing the working calibration standards from them.

5.6.3 Aqueous standards may be stored for up to 24 hours if held in Teflon-sealed screw-cap vials with zero headspace at 4°C. 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.

5.6.4 Purgeable standards must be stored separately from other standards.

6. Instrument Operating Conditions

6.1 Purge and Trap Device

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

Purge Conditions:

Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 ± 0.1 min
Purge Flow Rate:	25-40 mL/min
Purge Temperature:	Ambient (water or medium level soil), required 40°C (low level soil), required

Desorb Conditions:

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

Trap Reconditioning Conditions:

Reconditioning Temperature:	180°C
Reconditioning Time:	7.0 min ± 0.1 min

Before initial use, condition the trap overnight at 180°C by backflushing with at least 20 mL/min flow of inert gas. Vent the trap effluent to the room and not to the analytical column. Prior to daily use, condition the trap by heating at 180°C for 10 minutes while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

6.2 Gas Chromatograph

The following are the recommended GC analytical conditions:

6.2.1 Packed Columns

Carrier Gas:	Helium
Flow Rate:	30 mL/min
Initial Temperature:	45°C
Initial Hold Time:	3 min
Ramp Rate:	8°C/min
Final Temperature:	220°C
Final Hold Time:	15 min
Transfer Line Temperature:	250-300°C

6.2.2 Capillary Columns

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

6.2.3 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.

6.3 Mass Spectrometer

The following are the required mass spectrometer conditions:

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

6.4 The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as 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 (paragraph 5.4.1).

6.4.1 Prior to the analyses of any samples, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing *p*-bromofluorobenzene (BFB).

6.4.2 The analysis of the instrument performance check solution may be performed as follows:

- o As an injection of up to 50 ng of BFB into the GC/MS.
- o By adding 50 ng of BFB to 5.0 ml of reagent water and analyzing the resulting solution as if it were an environmental sample (see section 8 below).

BFB may <u>not</u> be analyzed simultaneously with a calibration standard.

6.4.3 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of BFB. NOTE: All subsequent standards, samples, MS/MSD, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

6.4.4 The analysis of the instrument performance check solution must meet the ion abundance criteria given below.

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 percent that of m/z 95.

6.4.5 The criteria listed above are based on adherence to the acquisition specifications identified in paragraph 6.4.3, and were developed for the specific target compound list associated with this Statement of Work. The criteria are based on performance characteristics of instruments currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for 23 target compounds (see Table 2), are designed to control and monitor instrument performance associated with the requirements of this Statement of Work.

6.4.6 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) and method blank 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 has elapsed according to the system clock.

7. Calibration

7.1 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 a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the purgeable target compounds.

7.2 Assemble a purge and trap device that meets the specification in 3.6. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Daily, prior to use, condition the traps for 10 minutes while backflushing at 180°C with the column at 220°C.

7.3 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in 6.2. Calibrate the purge and trap-GC/MS system using the internal standard technique (7.4).

7.4 Internal standard calibration procedure. The three internal standards are Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d_5, at 50 ug/L at time of purge. Separate initial and continuing calibrations must be performed for water samples, and low level soil samples (unheated purge vs. heated purge). Extracts of medium level soil samples may be analyzed using the calibrations for water samples.

7.4.1 Prepare calibration standards at a minimum of five concentration levels for each target compound and system monitoring compound, as specified in 5.5. Standards may be stored up to 24 hours, following the procedures in paragraph 5.6.3.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in paragraph 5.4.3.

7.4.3 Verify that the GC/MS system meets the instrument performance criteria in paragraph 6.4 by injecting BFB. Analyze each calibration standard, according to paragraph 7.1, adding 10 uL of internal standard spiking solution directly to the syringe.

Tabulate the area response of the characteristic ions in the extracted ion current profile (EICP) against concentration for each compound and internal standard and calculate relative response factors (RRF) for each compound as follows:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (see Table 3)

C_{is} = Concentration of the internal standard

C_x = Concentration of the compound to be measured

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 coelute. On capillary columns, the m- and p-Xylene isomers coelute. 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.

For the cis and trans isomers of 1,2-Dichloroethene which may coelute on packed columns but not on capillary columns, both isomers must be present in the standards. If the two isomers coelute, use the area of the coeluting peak and the total concentration of the two isomers in the standard to determine the relative response factor. If the two isomers do not coelute, sum the areas of the two peaks and the concentrations of the two isomers in the standard to determine the relative response factor.

7.4.4 The average relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of RRF values over the working range of the curve.

$$\%RSD = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

7.4.5 The response factors of the compounds listed below (Table 2) must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to two volatile compounds. However, the RRFs for those two compounds must be greater than or equal to 0.010, and the %RSD of those two compounds must be less than or equal to 40.0% for the initial calibration to be acceptable.

TABLE 2
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION OF VOLATILE ORGANIC COMPOUNDS

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Bromomethane	0.100	20.5	25.0
Vinyl chloride	0.100	20.5	25.0
1,1-Dichloroethene	0.100	20.5	25.0
1,1-Dichloroethane	0.200	20.5	25.0
Chloroform	0.200	20.5	25.0
1,2-Dichloroethane	0.100	20.5	25.0
1,1,1-Trichloroethane	0.100	20.5	25.0
Carbon tetrachloride	0.100	20.5	25.0
Bromodichloromethane	0.200	20.5	25.0
cis-1,3-Dichloropropene	0.200	20.5	25.0
Trichloroethene	0.300	20.5	25.0
Dibromochloromethane	0.100	20.5	25.0
1,1,2-Trichloroethane	0.100	20.5	25.0
Benzene	0.500	20.5	25.0
trans-1,3-Dichloropropene	0.100	20.5	25.0
Bromoform	0.100	20.5	25.0
Tetrachloroethene	0.200	20.5	25.0
1,1,2,2-Tetrachloroethane	0.500	20.5	25.0
Toluene	0.400	20.5	25.0
Chlorobenzene	0.500	20.5	25.0
Ethylbenzene	0.100	20.5	25.0
Styrene	0.300	20.5	25.0
Xylenes (total)	0.300	20.5	25.0
Bromofluorobenzene	0.200	20.5	25.0

7.4.6 The following compounds have no Maximum %RSD, or Maximum %Difference criteria; however, these compounds must meet a minimum RRF criterion of 0.010:

Acetone	1,2-Dichloropropane
2-Butanone	2-Hexanone
Carbon disulfide	Methylene chloride
Chloroethane	4-Methyl-2-pentanone
Chloromethane	Toluene-d_8
1,2-Dichloroethene (total)	1,2-Dichloroethane-d_4

7.4.7 A check of the calibration curve must be performed once every 12 hours (see paragraph 6.4.6 for the definition of the twelve hour time period). Check the relative response factors of those compounds for which RRF values have been established. If these criteria are met, the relative response factors for all compounds are calculated and reported. A percent difference of the daily relative response factor (12 hour) compared to the average relative response factor from the initial curve is

calculated. Calculate the percent difference for each compound and compare with the maximum percent difference criteria listed above. For negative percent difference values, the value must be greater than or equal to -25.0%, but less than 0%. As with the initial calibration, up to two volatile compounds in Table 2 may fail to meet the minimum RRF or maximum %D criteria, but the RRFs of those two compounds must be greater than or equal to 0.010, and the percent differences must be less than or equal to 40.0% for the continuing calibration to be acceptable.

7.4.8 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 0.50 minutes (30 seconds) from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction, and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

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

7.6 If time remains in the 12 hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard, if the initial calibration meets the calibration acceptance criteria above. A method blank is necessary. Quantify all sample results against the initial calibration standard that is the same concentration as the continuing calibration standard (50 ug/L).

7.7 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.

7.8 The concentrations of volatile target compounds in the continuing calibration standard are given in paragraph 5.5.3.

7.9 The response factors for the continuing calibration standard must meet the criteria given in paragraph 7.4.5 prior to the analysis of any blanks or samples.

8. Sample Analysis

8.1 Water Samples

8.1.1 All water samples must be allowed to warm to ambient temperature before analysis.

8.1.2 Prior to the analysis of samples, establish the appropriate GC/MS operating conditions, as outlined in paragraphs 6-6.4.6, analyze the instrument performance check solution (6.4), and calibrate the GC/MS system according to paragraphs 7-7.7.3.

8.1.3 If time remains in the 12-hour period (as described in paragraph 7.6), samples may be analyzed without analysis of a continuing calibration standard.

8.1.4 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 paragraphs 7.7-7.9).

8.1.5 Adjust the purge gas (helium) flow rate to 25-40 mL/min. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly Chloromethane and Bromoform.

8.1.6 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 5.0 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 should 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.

8.1.7 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.

8.1.8 The purgeable organics screening procedure (Section III), if used, will have shown 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.

8.1.8.1 The following procedure will allow for dilutions near the calculated dilution factor from the screening procedure:

8.1.8.1.1 All dilutions are made in volumetric flasks (10 mL to 100 mL).

8.1.8.1.2 Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

8.1.8.1.3 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.

8.1.8.1.4 Inject the proper aliquot from the syringe prepared in paragraph 8.1.6 into the volumetric flask. Aliquots of less than 1 mL increments are prohibited. Dilute the flask to the mark with reagent water. Cap the flask, invert, and shake three times.

8.1.8.1.5 Fill a 5 mL syringe with the diluted sample as in paragraph 8.1.6.

8.1.8.1.6 If this is an intermediate dilution, use it and repeat the above procedure to achieve larger dilutions.

8.1.9 Add 10.0 uL of the system monitoring compound spiking solution (paragraph 5.4.4) and 10.0 uL of the internal standard spiking solution (paragraph 5.4.3) 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 uL of the system monitoring compound spiking solution to 5 mL of sample is equivalent to a concentration of 50 ug/L of each system monitoring compound.

8.1.10 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.

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

8.1.12 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/min for four minutes. If this rapid heating requirement cannot be met, the gas chromatographic column must be used as a secondary trap by cooling it to 30°C (or subambient, if problems persist) instead of the recommended initial temperature of 45°C.

8.1.13 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.

8.1.14 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.

8.1.15 Each analytical run must be checked also for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. The initial calibration requires that the system should not be saturated for high response compounds at 200 ug/L for VOA target compounds. In addition, the system must not be saturated by the two Xylene isomers that coelute on the GC column used for analysis when the coeluting peak represents 400 ug/L, or, for the two 1,2-Dichloroethene isomers that may coelute when the coeluting peak represents 400 ug/L. Secondary ion quantitation is allowed <u>only</u> when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by the analysis of a reagent water blank. If the blank is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank has been analyzed that demonstrates that the system is free of interferences. Once the system is free of interferences, the sample that saturated the detection must be diluted and reanalyzed.

8.1.16 To prepare a matrix spike and matrix spike duplicate for water

samples, add 10 uL of the matrix spike solution (paragraph 5.4.5) to each of the 5 mL aliquots of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike compound. The frequency of MS/MSD analysis is given in paragraph 10.8.

8.1.17 A volatile method blank must be analyzed at least once during every twelve hour time period, on each GC/MS system used for volatile analysis (see paragraph 6.4.6 for the definition of the twelve hour time period).

8.1.17.1 For water samples, a volatile method blank consists of a 5 mL volume of reagent water (paragraph 4.1) spiked with the system monitoring compounds and internal standards, and carried through the analytical procedure.

8.1.17.2 An acceptable volatile method blank for water samples must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Exhibit C) of Methylene chloride, Acetone, and 2-Butanone, and less than or equal to the CRQL of any other volatile target compound.

8.1.17.3 All volatile analyses associated with a blank that does not meet the requirements above, (i.e., a contaminated blank) must be repurged, reanalyzed, and reported at no additional cost to the Agency.

8.1.17.4 The volatile method blank must be analyzed after the calibration standards, to ensure that there is no carryover of material from the standards into samples.

8.1.18 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:

8.1.18.1 Analyze a method blank immediately after the contaminated sample. If an autosampler is used, a method blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The method blanks must meet the technical acceptance criteria for blank analysis (see 8.1.17), or

8.1.18.2 Monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample and that exceeded the limits above. The maximum contamination criteria are as follows: the sample must not contain a concentration above the CRQL for the target

compounds that exceeded the limits in the contaminated sample. If an autosampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample also must meet the maximum contamination criteria.

8.2 Soil/Sediment Samples

Two approaches may be taken to determine whether the low level or medium level method must be followed.

- o Assume the sample is low level and analyze a 5 g sample.
- o Use the X factor calculated from the optional hexadecane screen (Section III, paragraph 6.2.1.3) to determine the appropriate method for analysis.

If peaks are saturated from the analysis of a 5 g sample, a smaller sample size must be analyzed to prevent saturation. However, the smallest sample size permitted is 1 g. If smaller than 1 g sample size is needed to prevent saturation, the medium level method must be used.

8.2.1 Low Level Soil Method

The low level soil 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 method blanks and standards under the same conditions as the samples.

Use 5 grams of sample, or use the X Factor to determine the sample size for purging.

- o If the X Factor is 0 (no peaks noted on the hexadecane screen), analyze a 5 g sample.
- o If the X Factor is between 0 and 1.0, analyze a minimum of a 1 g sample.
- o If the X Factor is >1, use the medium soil method.

8.2.1.1 The GC/MS system should be set up as in paragraphs 7-7.7.3. This should be done prior to the preparation of the sample to avoid loss 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 (7.4 and 7.7), but increase the purge temperature to 40°C.

8.2.1.2 To prepare the reagent water containing the system monitoring compounds and the internal standards, remove the plunger from a 5 mL "Luerlock" 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 5.0 mL. Add 10 uL of the system monitoring compound spiking solution and 10 uL of the internal standard solution to the syringe through the valve.

8.2.1.3 The sample (for volatile organics) consists of 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 the amount determined in paragraph 8.2.1 into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

8.2.1.4 Add the spiked reagent water to the purge device and connect the device to the purge and trap system. NOTE: Prior to the attachment of the purge device, the steps in paragraphs 8.2.1.2 and 8.2.1.3 must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

8.2.1.5 Immediately after weighing the sample, weigh 5-10 g of the 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 sediment.

$$\frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100 = \% \text{ moisture}$$

8.2.1.6 Heat the sample to 40°C ± 1°C and purge the sample for 11.0 ± 0.1 minutes.

8.2.1.7 Proceed with the analysis as outlined in paragraphs 8.1.11 - 8.1.14. Requirements for dilution of samples are given in paragraphs 8.2 and 10.7.

8.2.1.8 To prepare a matrix spike and matrix spike duplicate for low level soils/sediment, add 10 uL of the matrix spike solution (5.4.4) to the 5 mL of water added to each of the two aliquots of the soil from the sample chosen for spiking (paragraph 8.2.1.2). The concentration for a 5 g sample would be equivalent to 50 ug/kg of each matrix spike compound. The frequency of MS/MSD analysis is given in paragraph 10.8.

8.2.1.9 A volatile method blank must be analyzed at least once during every twelve hour time period, on each GC/MS system used for volatile analysis (see paragraph 6.4.6 for the definition of the twelve hour time period).

8.2.1.9.1 For low level soil/sediment samples, a volatile method blank consists of a 5 g of a purified solid matrix added to reagent water, spiked with the system monitoring compounds and internal standards, and carried through the analytical procedure.

8.2.1.9.2 An acceptable volatile method blank for low level soil samples must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Exhibit C) of Methylene chloride, Acetone, and 2-Butanone, and less than or equal to the CRQL of any other volatile target compound.

8.2.1.9.3 <u>All</u> volatile analyses associated with a blank that does <u>not</u> meet the requirements above, (i.e., a contaminated blank) <u>must</u> be repurged, reanalyzed,and reported at no additional cost to the Agency.

8.2.1.9.4 The volatile method blank <u>must</u> be analyzed <u>after</u> the calibration standards, to ensure that there is no carryover of material from the standards into samples.

8.2.1.10 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:

8.2.1.10.1 Analyze a method blank immediately after the contaminated sample. If an autosampler is used, a method blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The method blanks must meet the technical acceptance criteria for blank analysis (see 8.2.1.9), or

8.2.1.10.2 Monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample and that exceeded the limits above. The maximum contamination criteria are as follows: the sample must not contain a concentration above the CRQL for the target compounds that exceeded the limits in the contaminated sample. If an autosampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample also must meet the maximum contamination criteria.

8.2.2 Medium Level Soil Method

The medium level soil 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. All samples with an X Factor >1.0 must be analyzed by the medium level method. If saturated peaks occurred, or would occur, when a 1 g sample was analyzed, the medium level method must be used.

8.2.2.1 The GC/MS system should be set up as in paragraphs 7-7.7.3. 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 <u>ambient</u> temperature, the instrument performance check, initial calibration, and continuing calibration for water samples may be used for analyses of medium soil sample extracts.

8.2.2.2 The sample (for volatile organics) consists of 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. Note and record the actual weight to the nearest 0.1 g. Determine the percent moisture as in paragraph 8.2.1.5.

8.2.2.3 Quickly add 9.0 mL of methanol to the vial. Then add 1.0 mL of the system monitoring compound spiking solution to the vial. Cap and shake for 2 minutes. NOTE: The steps in paragraphs 8.2.2.2 and 8.2.2.3 must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

8.2.2.4 Using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be discarded. Transfer approximately 1 mL of the reagent methanol to a GC vial for use as the method blank for each Case, SDG, or day on which medium soil sample extractions are performed, whichever is most frequent. These extracts may be stored in the dark at 4°C (±2°C) prior to analysis.

8.2.2.5 The following table 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 was followed, use the X factor (Option B) or the estimated concentration (Option A) to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low level analysis to determine the appropriate volume. If the sample was submitted as a medium level sample, start with 100 uL.

All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of linear range of the curve.

X Factor	Estimated Concentration Range[1] ug/kg	Take this Volume of Methanol Extract[2] uL
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 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 uL added to the syringe.

3 Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

8.2.2.6 Remove the plunger from a 5 mL "Luerlock" 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 uL of the internal standard solution. Also add the volume of methanol extract determined in paragraph 8.2.2.5 and a volume of clean methanol to total 100 uL (excluding methanol in standards).

8.2.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

8.2.2.8 Proceed with the analysis as outlined in paragraph 8. Analyze all method blanks on the same instrument as the samples. Requirements for dilution of samples are given in paragraphs 8.2 and 10.7.

8.2.2.9 To prepare a matrix spike and matrix spike duplicate for the medium level soil/sediment samples, add 8.0 mL of methanol, 1.0 mL of the system monitoring compound spiking solution, and 1.0 mL of matrix spike solution (paragraph 5.4.4) as in paragraph 8.2.2.3, to each of the two aliquots of the soil sample chosen for spiking. This results in a 6,200 ug/kg concentration of each matrix spike compound when added to a 4 g sample. Add a 100 uL aliquot of this extract to 5 mL of water for purging (as per paragraph 8.2.2.6). The frequency of MS/MSD analysis is given in paragraph 10.8.

8.2.2.10. A volatile method blank must be analyzed at least once during every twelve hour time period, on each GC/MS system used for volatile analysis (see paragraph 6.4.6 for the definition of the twelve hour time period).

8.2.2.10.1 For medium level soil/sediment samples, a volatile method blank consists of a 4 g of a purified solid matrix spiked with the system monitoring compounds, extracted with methanol, and carried through the analytical procedure.

8.2.2.10.2 An acceptable volatile method blank for medium level soil/sediment samples must contain less than or equal five times (5x) the Contract Required Quantitation Limit (CRQL, see Exhibit C) of Methylene chloride, Acetone, and 2-Butanone, and less than or equal ot the CRQL of any other volatile target compound.

8.2.2.10.3 All volatile analyses associated with a blank that does not meet the requirements above, (i.e. a contaminated blank) must be repurged, reanalyzed,and reported at no additional cost to the Agency.

8.2.2.10.4 The volatile method blank must be analyzed after the calibration standards, to ensure that there is no carryover of material from the standards into samples.

8.2.2.11 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:

8.2.2.11.1 Analyze a method blank immediately after the contaminated sample. If an autosampler is used, a method blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The method blanks must meet the technical acceptance criteria for blank analysis (see 8.2.2.10), or

8.2.2.11.2 Monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample and that exceeded the limits above. The maximum contamination criteria are as follows: the sample must not contain a concentration above the CRQL for the target compounds that exceeded the limits in the contaminated sample. If an autosampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample also must meet the maximum contamination criteria.

9. Qualitative Analysis

9.1 The compounds listed in the Target Compound List (TCL), Exhibit C, shall be identified by an analyst competent in the interpretation of mass spectra (see Exhibit A, Section III) 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.

9.1.1 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 ug/L standard. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

9.1.2 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.

9.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

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

9.1.3.2 The relative intensities of ions specified in paragraph 9.1.3.1 must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent).

9.1.3.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Task III, 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."

9.1.4 If a compound cannot be verified by all of the criteria in paragraph 9.1.3.3, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantification in paragraph 10.

9.2 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the most recent release of the NIST/EPA/MSDC mass spectral library, shall be used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

9.2.1 Up to 10 organic compounds of greatest apparent concentration not listed in Exhibit C for the purgeable organic fraction, excluding the system monitoring compounds, shall be tentatively identified via a forward search of the NIST/EPA/MSDC Library (substances with responses less than 10% of the internal standard are not required to be searched in this fashion). Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

9.2.2 Guidelines for making tentative identification:

9.2.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

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

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

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

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

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

10. Quantitative Analysis

10.1 Target components identified shall be quantified by the internal standard method. The internal standard used shall be that which is assigned in Table 5 of this Section. The EICP area of the characteristic ions of analytes listed in Tables 3 and 4 in this Section are used.

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 initializing and dating the changes made to the report.

10.2 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. 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 ug/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. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

10.2.1 If after re-analysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with EICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

10.2.2 If the re-analysis of the sample does not solve the problem, i.e., the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the SDG Narrative all inspection and corrective actions taken.

10.3 The relative response factor (RRF) from the continuing calibration standard is used to calculate the concentration in the sample. Use the relative response factor as determined in paragraph 7.4.3 and the equations below. When target compounds are below contract required quantitation limits (CRQL), but the spectra meet the identification criteria, report the concentration with a "J." For example, if CRQL is 10 ug/L and concentration of 3 ug/L is calculated, report as "3J."

Water

$$\text{Concentration} \quad \text{ug/L} = \frac{(A_x)(I_s)(Df)}{(A_{is})(RRF)(V_o)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (see Table 3)

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 2.5 mL of sample is diluted to 5.0 mL with reagent water and purged, Df = 5.0 mL/2.5 mL = 2.0. If no dilution is performed, Df = 1.0.

Low Soil

Concentration (Dry weight basis) ug/Kg = $\frac{(A_x)(I_s)}{(A_{is})(RRF)(W_s)(D)}$

Where,

A_x, I_s, A_{is} are as given for water.

RRF = Relative response factor from the heated purge of the calibration standard.

D = $\frac{100 - \%\ moisture}{100}$

W_s = Weight of sample added to the purge tube, in grams (g)

Medium Soil

Concentration (Dry weight basis) ug/Kg = $\frac{(A_x)(I_s)(V_t)(1000)(Df)}{(A_{is})(RRF)(V_a)(W_s)(D)}$

Where,

A_x, A_{is}, I_s are as given for water above.

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 10.0 mL, even though only 1.0 mL is transferred to the vial in paragraph 8.2.2.4.

V_a = Volume of the aliquot of the methanol extract in microliters (uL) added to reagent water for purging

W_s = Weight of soil extracted, in grams (g)

D = $\frac{100 - \%\ moisture}{100}$

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as the ratio of the number of microliters (uL) of methanol added to the reagent water for purging i.e., V_a above, to the number of microliters of the methanol extract of the sample contained in that volume V_a. The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the methanol extract. Dilution of the extract is required when the "X" factor (paragraph 8.2.2.5) is $\geq$ 12.5.

The factor of 1,000 in the numerator converts the value of V_t from mL to uL.

10.4 An estimated concentration for non-target components tentatively identified shall be determined by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used.

The formula for calculating concentrations is the same as in paragraph 10.3. 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 should be calculated for all tentatively identified compounds as well as those identified as unknowns.

10.5 Xylenes (o-, m-, and p- isomers) are to be reported as Xylenes (total). Because the o- and p-Xylene isomers coelute on packed columns, and the m- and p-Xylene isomers coelute on capillary columns, special attention must be given to the quantitation of the Xylenes. The relative response factor (RRF) determined in paragraph 7.4 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 paragraph 7.4. The areas of the two peaks may be summmed, 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.

10.6 The cis and trans steriosomers of 1,2-Dichloroethene are to be reported as 1,2-Dichloroethene (total). If the two isomers coelute on the GC column used for analysis, use the area of the coeluting peaks and the RRF determined in 7.4 to determine the concentration. If the isomers do not coelute, use the single RRF values determined in 7.4 to determine the concentration. 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 both the cis and trans isomers of the 1,2-Dichloroethene be present in the initial and continuing calibration standards.

10.7 If the on-column concentration of any 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 below.

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

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

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

10.7.4 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the SDG Narrative.

10.7.5 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 ug/L or the peak representing the two coeluting isomers on the GC column exceeds 400 ug/L. Similarly, if the cis and trans isomers of 1,2-Dichloroethene coelute, a diluted analysis is not required unless the concentration of the coeluting peak exceeds 400 ug/L. If the two isomers do not coelute, a diluted analysis is not required unless the concentration of either peak exceeds 200 ug/L.

10.8 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 6), and report on appropriate form.

10.8.1 Calculate the concentrations of the system monitoring compounds using the same equations as used for target compounds. Calculate the recovery of each system monitoring compound as follows:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

10.8.2 If the recovery of any one system monitoring compound is not within limits, the following are required:

- Check to be sure that there are no errors in calculations, formulation of the system monitoring compound spiking solutions, and internal standards. Also check instrument performance.
- Reanalyze the sample if none of the above steps reveal a problem.
- If an undiluted analysis with acceptable monitoring compound recoveries is being submitted, do not reanalyze diluted samples if the system monitoring compound recoveries are outside the limits.

- o Never reanalyze the matrix spike or matrix spike duplicate (MS/MSD), even if the system monitoring compound recoveries are outside the limits.
- o If the sample associated with the matrix spike and matrix spike duplicate does not meet specifications, it should be reanalyzed only if the MS/MSD system monitoring compound recoveries are within the limits. If the sample and associated MS/MSD show the same pattern (i.e., outside the limits), then the sample does not require reanalysis and a reanalysis must not be submitted. Document in the narrative the similarity in recoveries of the system monitoring compounds in the sample and associated MS/MSD.

10.8.3 If the reanalysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analysis with system monitoring compound recoveries within the limits. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

10.8.4 If the reanalysis of the sample does not solve the problem (i.e., the system monitoring compound recoveries are outside the limits for both analyses), then submit the data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes supplied in Exhibit B.

10.8.5 For medium level soil analyses, involving methanol extraction, the treatment of system monitoring compound recoveries is similar to that for semivolatile surrogate recoveries. If any system monitoring compound recovery is outside the limits, reanalyze the methanol extract first, to determine if the problem was with the analysis. If reanalysis of the extract does not solve the problem, then reextract the medium soil sample and analyze the second extract. Follow paragraphs 10.8.3 and 10.8.4 when determining which analyses to submit.

10.8.6 If the recovery of any one system monitoring compound in a method blank is outside the limits, then the method and all associated samples must be reanalyzed at no additional cost to the Agency.

10.9 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:

- o Each Case of field samples received, OR
- o Each 20 field samples in a Case, OR
- o Each group of field samples of a similar concentration level (soils only), OR

- o Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which field samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group).

10.9.1 Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds. Calculate the recovery of each matrix spike compound as follows:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiked sample result
SR = Sample result
SA = Spike added

10.9.2 Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \frac{|MSR - MSDR|}{(1/2)(MSR+MSDR)} \times 100$$

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.

10.9.3 The limits for matrix spike compound recovery and RPD are given in Table 7. 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.

10.10 Determine the concentrations of any target compounds detected in the volatile method blank, using the equations in paragraph 10.3. The method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL) of the volatile target compounds in Exhibit C, except Methylene chloride, Acetone, and 2-Butanone, which must be less than or equal to five times (5x) the CRQL. For soil/sediment method blanks, CRQL value must be adjusted for percent moisture (see Exhibit B).

If a laboratory method blank exceeds these criteria, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be reextracted/repurged and reanalyzed at no additional cost to the Agency. The Laboratory Manager, or his designee, must address problems and solutions in the SDG Narrative (Exhibit B).

TABLE 3
CHARACTERISTIC IONS FOR
SYSTEM MONITORING COMPOUNDS AND
INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
SYSTEM MONITORING COMPOUNDS		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d-$_4$	65	102
Toluene-d-$_8$	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d-$_5$	117	82, 119

TABLE 4
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Analyte	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43**	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	-
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.

** m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

TABLE 5
VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS
AND SYSTEM MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2-Dichloropropane	1,1,2,2-Tetrachloroethane
Methylene Chloride	trans-1,3-Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1-Dichloroethene	1,1,2-Trichloroethane	Styrene
1,1-Dichloroethane	Benzene	Xylene (total)
1,2-Dichloroethene(tot.)	cis-1,3-Dichloropropene	Bromofluorobenzene (smc)
Chloroform	Bromoform	Toluene-d_8 (smc)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d_4 (smc)		

(smc) = system monitoring compound

EXHIBIT D

ANALYTICAL METHODS
FOR SEMIVOLATILES

Table of Contents

SECTION I

INTRODUCTION

The analytical methods that follow are designed to analyze water, soil and sediment from hazardous waste sites for the organic compounds on the Target Compound List (TCL, see Exhibit C). The methods are based on EPA Method 625 (Bases/Neutrals and Acids).

The methods are divided into the following sections: sample preparation, screening, and analysis. Sample preparation covers sample extraction and cleanup techniques. As described in the screening section, a portion of the extracts may be screened on a gas chromatograph with appropriate detectors to determine the concentration level of organics. The analysis section contains the GC/MS analytical methods for organics.

1. Method for the Determination of Extractable Semivolatile Organic Compounds.

1.1 Scope and Application

This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. These target compounds and the contract required quantitation limits are listed in Exhibit C.

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

- Dichlorobenzidine and 4-Chloroaniline can be subject to oxidative losses during solvent concentration.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatograph inlet forming diphenylamine and, consequently, may be detected as diphenylamine.

1.2 The method involves solvent extraction of the matrix sample, characterization to determine the appropriate analytical protocol to be used, and GC/MS analysis to determine semivolatile organic compounds present in the sample.

SECTION II

SAMPLE PREPARATION AND STORAGE

PART A - SAMPLE STORAGE AND HOLDING TIMES

1. Procedures for Sample Storage

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

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

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

2. Procedure for Sample Extract Storage

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

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

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

3. Contract Required Holding Times

Extraction of water samples by continuous liquid-liquid procedures shall be started within 5 days of VTSR (Validated Time of Sample Receipt). Extraction of soil/sediment samples by sonication procedures shall be completed within 10 days of VTSR. NOTE: Separatory funnel extraction procedures are not permitted.

Extracts of either water or soil/sediment samples must be analyzed within 40 days following extraction.

PART B - SAMPLE PREPARATION FOR EXTRACTABLE SEMIVOLATILES (BNA) IN WATER

1. Summary of Sample Preparation Method

1.1 A one liter aliquot of sample is acidified to pH 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. Separatory funnel extraction is NOT permitted. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

2. Interferences

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source.

3. Apparatus and Materials

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

3.1 Glassware (brand names and catalog numbers are included for illustration purposes only).

3.1.1 Continuous liquid-liquid extractors - equipped with Teflon or glass connnecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

3.1.2 Drying column - 19 mm ID chromatographic column with coarse frit (substitution of a small pad of Pyrex glass wool for the frit will prevent cross contamination of sample extracts).

3.1.3 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stoppers are used to prevent evaporation of extracts.

3.1.4 Evaporative flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

3.1.5 Snyder column - Kuderna-Danish, three-ball macro (Kontes K-503000-0121 or equivalent).

3.1.6 Snyder column - Kuderna-Danish, two-ball micro (Kontes K569001-0219 or equivalent).

3.1.7 Vials - amber glass, 2 mL capacity with Teflon-lined screw cap.

3.1.8 Syringes - 0.2 mL, 0.5 mL, and 5 mL volumes.

3.2 Silicon carbide boiling chips - approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

3.3 Water bath - heated, with concentric ring cover, capable of temperature control (± 2°C). The bath should be used in a hood.

3.4 Balance - analytical, capable of accurately weighing ± 0.0001 g.

3.5 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C. The N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent), is suitable.

4. Reagents

4.1 Reagent water - defined as water in which an interferent is not observed at or above the CRQL of each parameter of interest.

4.2 Sodium thiosulfate - (ACS) granular.

4.3 Sulfuric acid solution (1+1) - slowly add 50 mL of H_2SO_4 (sp gr 1.84) to 50 mL of reagent water.

4.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

4.5 Sodium sulfate - (ACS) powdered, anhydrous. Purify by heating at 400°C for four hours in a shallow tray, cool in a desiccator and store in a glass bottle (Baker anhydrous powder, catalog #73898, or equivalent).

4.6 Surrogate standard spiking solution.

4.6.1 Surrogate standards are added to all samples and calibration solutions; the compounds specified for this purpose are Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, 2-Chlorophenol-d_4, and 1,2-Dichlorobenzene-d_4. Additional surrogates may be added at the laboratory's discretion.

4.6.2 Prepare a surrogate standard spiking solution that contains Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, and 1,2-Dichlorobenzene-d_4 at a concentration of 100 ug/mL; Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, and 2-Chlorophenol-d_4 at a concentration of 150 ug/mL. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

4.7 BNA Matrix standard spiking solution - the matrix spike solution consists of the following:

Bases/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Prepare a spiking solution that contains each of the base/neutral compounds above at 100 ug/1.0 mL in methanol and the acid compounds at 150 ug/1.0 ml in methanol. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

5. Water Sample Extraction

5.1 Continuous liquid-liquid extraction is used to extract the samples.

5.1.1 Add methylene chloride to the bottom of the extractor and fill it to a depth of at least 1 inch above the bottom side arm.

5.1.2 Using a 1 liter graduated cylinder, measure out a 1.0 liter sample aliquot. Transfer the 1 liter sample aliquot to the continuous extractor. Pipet 0.5 mL of surrogate standard spiking solution into the sample and mix well. Check the pH of the sample with wide range pH paper and adjust the pH to 2.0 with 1:1 H_2SO_4.

5.1.3 Following the procedures in 5.1.1 and 5.1.2 above, prepare two additional 1.0 Liter aliquots of the sample chosen for spiking. Add 0.5 mL of the BNA Matrix Spiking Solution to each of the additional aliquots. The frequency of MS/MSD analysis is given in Section IV, paragraph 8.6.

5.1.4 Add 500 mL of methylene chloride to the distilling flask. Add sufficient reagent water to ensure proper operation. Extract for 18 hours. Allow to cool, then detach the distilling flask, and label the flask.

5.1.5 Prepare a method blank with each group of water samples extracted. For semivolatile analyses, a method blank for water samples consists of a 1 L volume of reagent water (see paragraph 4.1), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in Section IV, paragraph 8.7

5.2 Concentrating the Extracts

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

5.2.2 Transfer the extract by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate, and collect the extract in a K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

5.2.3 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60°C to 80°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will chatter actively, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5 mL syringe is recommended for this operation.

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

5.2.4.1 Micro Snyder Column Technique

Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Pre-wet the Snyder column by adding about 0.5 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60°C to 80°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will chatter actively, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the

Snyder column and rinse its flask and its lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL with methylene chloride. Transfer the extract to a Teflon-sealed screw-cap bottle, label the bottle, and store at 4°C (±2°C).

5.2.4.2 Nitrogen Evaporation Technique (taken from ASTM Method D3086)

Place the concentrator tube with an open micro Snyder attached in a warm water bath (30°C to 35°C) and evaporate the solvent volume to just below 1 mL by blowing a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon) above the extract. CAUTION: Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or Teflon tubing. The internal wall of the concentrator tube must be rinsed down several times with methylene chloride during the operation and the final volume brought to 1.0 mL with methylene chloride. During evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry. Transfer the extract to a Teflon-sealed screw-cap bottle, label the bottle and store at 4°C (±2°C).

6. The sample extracts are ready for GC/MS analysis. Proceed to Section IV, GC/MS Analysis of Semivolatiles. If high concentrations are suspected (e.g., highly colored extracts), the optional GC/FID screen in Section III is recommended.

PART C - SAMPLE PREPARATION FOR EXTRACTABLE SEMIVOLATILES (BNA) IN SOIL/SEDIMENT

It is mandatory that all soil/sediment samples be characterized as to concentration level so that the appropriate analytical protocol is chosen to ensure proper quantitation limits for the sample. Note that the terms "low level" and "medium level" are not used here as a judgement of degree of contamination but rather as a description of the concentration ranges that are encompassed by the "low" and "medium" level procedures.

The laboratory is at liberty to determine the method of characterization. The following two screening methods may be used for soil/sediment sample characterization:

- Screen an aliquot from the "low level" 30 g extract or an aliquot from the "medium level" 1 g extract.
- Screen using either GC/FID or GC/MS as the screening instrument.

The concentration ranges covered by these two procedures may be considered to be approximately 330 ug/kg - 10,000 ug/kg for the low level analysis and >10,000 ug/kg for medium level analysis for semivolatile extractables.

Screen from the Medium Level Method

Take 5.0 mL from the 10.0 mL total extract and concentrate to 1.0 mL and screen. If the sample concentration is >10,000 ug/kg, proceed with GC/MS analysis of the organics. If the sample concentration is <10,000 ug/kg, discard the medium level extract and follow the low level method.

Screen from the Low Level Method

Take 5.0 mL from the 300 mL (approximate) total extract from the 30 g sample and concentrate to 1.0 mL and screen. If the original sample concentration is >10,000 ug/kg, discard the 30 g extract and follow the medium level methods for organics, using medium level surrogates. If the sample concentration is <10,000 ug/kg, proceed with concentration and the remainder of the low level method.

Mandatory GPC Clean Up

Regardless of the concentration level, all soil/sediment sample extracts must be subjected to clean up by Gel Permeation Chromatography (GPC). Because the effectiveness of GPC can be adversely affected by the amount of material loaded onto the GPC column, it may be advisable to screen the sample extracts described here prior to employing GPC.

1. Medium Level Preparation for Screening and Analysis of Semivolatiles

1.1 Scope and Application

This procedure is designed for the preparation of soil/sediment samples which may contain organic chemicals at a level greater than 10,000 ug/kg.

1.1.1. The extracts and sample aliquots prepared using this method are screened by GC/MS or FID, using capillary columns for semivolatile priority pollutants, and related organic chemicals. The results of these screens will determine whether sufficient quantities of pollutants are present to warrant analysis by the medium protocol.

1.1.2 If the screenings indicate no detectable pollutants at the lower limits of quantitation, the sample should be prepared by the low level protocol in Section II, Part C, beginning at paragraph 2.

1.2 Summary of Method

1.2.1. Approximately 1 g portions of soil/sediment are transferred to vials and extracted with methylene chloride. The methylene chloride extract is screened for extractable organics by GC/FID or GC/MS.

1.2.2 If organic compounds are detected by the screen, the methylene chloride extract is subjected to GPC clean up and analyzed by GC/MS for extractable organics.

1.2.3 If no organic compounds are detected by the medium level screen, then a low level sample preparation is required.

1.3 Interferences

1.3.1. 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 routinely must be 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.

1.4 Limitations

1.4.1. The procedure is designed to allow quantitation limits for screening purposes as low as 10,000 ug/kg for extractable organics. For analysis purposes, the quantitation limits are 10,000 ug/kg for extractable organics. If peaks are present based on the GC/FID screen, the sample is determined to require a medium level analysis by GC/MS. Some samples may contain

high concentrations of chemicals that interfere with the analysis of other components at lower levels; the quantitation limits in those cases may be significantly higher.

1.4.2 These extraction and preparation procedures were developed for rapid and safe handling of high concentration hazardous waste samples. The design of the methods thus does not stress efficient recoveries or low limits of quantitation of all components. Rather, the procedures were designed to screen, at moderate recovery and sufficient sensitivity, 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.

1.5 Reagents

1.5.1. Sodium Sulfate - anhydrous powdered reagent grade, heated at 400°C for four hours, cooled in a desiccator, and stored in a glass bottle (Baker anhydrous powder, catalog # 73898 or equivalent).

1.5.2 Acetone, Methanol, Methylene chloride - pesticide residue analysis grade or equivalent.

1.5.3 Base/Neutral and Acid Surrogate Spiking Solution

Surrogate standards are added to all samples and calibration solutions. The compounds specified are Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, 2-Chlorophenol-d_4, and 1,2-Dichlorobenzene-d_4. Prepare a surrogate standard spiking solution that contains Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, and 1,2-Dichlorobenzene-d_4 at a concentration of 100 ug/mL; Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, and 2-Chlorophenol-d_4 at a concentration of 150 ug/mL. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

1.5.4 Base/Neutral and Acid Matrix Spiking solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 100 ug/mL for base/neutrals and 150 ug/mL for acids. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

Bases/ Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

1.6 Equipment

1.6.1. Glass scintillation vials - at least 20 mL, with screw cap and teflon or aluminum foil liner.

1.6.2 Spatula - stainless steel or Teflon.

1.6.3 Balance - capable of weighing 100 g to ± 0.01 g.

1.6.4 Vials and caps - 2 mL for GC auto sampler.

1.6.5 Disposable pipets - Pasteur; glass wool rinsed with methylene chloride.

1.6.6 Concentrator tubes - 15 mL.

1.6.7 Ultrasonic cell disruptor - Heat Systems, Ultrasonics, Inc., Model W-385 SONICATOR (475 Watt with pulsing capability, No. 200, 1/2 inch tapped disruptor horn and No. 419, 1/8 inch standard tapered MICROTIP probe), or equivalent device with a minimum of 375 Watt output capability. NOTE: In order to ensure that sufficient energy is transferred to the sample during extraction, the MICROTIP probe must be replaced if the tip begins to erode. Erosion of the tip is evidenced by a rough surface.

1.6.8 Sonabox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.

1.6.9 Test tube rack.

1.6.10 Oven - drying.

1.6.11 Desiccator.

1.6.12 Crucibles - porcelain.

1.6.13 Syringes - 0.5 mL volume.

1.7 Medium Level Sample Preparation.

1.7.1. Transfer the sample container into a fume hood. Decant and discard any water layer on a sediment sample. Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks. Transfer

approximately 1 g (record weight to the nearest 0.1 g) of sample to a 20-mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before of sample tak sample to avoid any cross-contamination.

1.7.1.1 Transfer 50 g of soil/sediment to a 100 mL beaker. Add 50 mL of water and stir for 1 hour. Determine pH of sample with glass electrode and pH meter while stirring. Report pH value on appropriate data sheets. If the pH of the soil is greater than 11 or less than 5, contact the Technical Project Officer cited in the contract for instructions on how to handle the sample. Document the instructions in the SDG Narrative. Discard this portion of sample. NOTE: If limited sample volume is received, use 5 g of soil and 5 mL of water for the pH determination. Note this in the SDG Narrative.

1.7.2 Immediately after weighing the sample for extraction, weigh 5-10 g of the 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 sediment.

$$\frac{\text{g of sample - g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

1.7.3 Add 2.0 g of anhydrous powdered sodium sulfate to the sample in the 20 mL vial from paragraph 1.7.1 and mix well.

1.7.4 Surrogates are added to all samples, spikes, and blanks. Add 0.5 mL of surrogate spiking solution to sample mixture.

1.7.5 Add 0.5 mL of matrix standard spiking solution to each of two 1 g portions from the sample chosen for spiking. The frequency of MS/MSD analysis is given in Section IV, paragraph 8.6.

1.7.6 Immediately add 9.5 mL of methylene chloride to the sample and disrupt the sample with the 1/8 inch tapered MICROTIP ultrasonic probe for 2 minutes at output control setting 5, in continuous mode (if using a sonicator other than Models W-375 or W-385, contact the Project Officer for appropriate output settings). Before extraction, make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula or, very carefully, with the tip of the unenergized probe.

Add only 9.0 mL of methylene chloride to the matrix spike samples to achieve a final volume of 10 mL.

1.7.7 Prepare a method blank with each group of medium soil/sediment samples extracted. For semivolatile analyses, a method blank for medium soil/sediment samples consists of 1 g of sodium sulfate (see paragraph 1.5.1), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in Section IV, paragraph 8.7

1.7.8 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect at least 8.0 mL in a concentrator tube.

1.7.9 If the extract is to be screened prior to GPC, concentrate 5.0 mL of the extract collected in paragraph 1.7.7 to 1.0 mL using the nitrogen evaporation technique described in paragraph 3.6.2. Transfer the concentrate to an autosampler vial for GC/FID or GC/MS for screening. The quantitation limits for the screening procedure in Section III are approximately 10,000 ug/Kg.

1.7.10 If the extract is to be cleaned up using GPC without screening, take at least 8.0 mL of the extract in paragraph 1.7.7 and proceed to paragraph 3 of this section. Following GPC, the 5.0 mL of extract collected must be concentrated to 0.5 mL by the nitrogen evaporation technique described in paragraph 3.6.2, and screened according to the procedures in Section III. In this case, the quantitation limits for the screening procedures in Section III are approximately 20,000 ug/Kg.

2. Low Level Preparation for Screening and Analysis of Semivolatiles

2.1 Summary of Method

A 30 gram portion of sediment is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone using an ultrasonic probe. If the optional low level screen is used, a portion of this dilute extract is concentrated fivefold and is screened by GC/FID or GC/MS. If peaks are present at greater than 10,000 ug/kg, discard the extract and prepare the sample by the medium level method. If no peaks are present at greater than 10,000 ug/kg, the entire extract is concentrated, subjected to GPC clean up, and analyzed by GC/MS for extractable organics.

2.2 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 routinely must be 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.

2.3 Apparatus and Materials

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

2.3.1 Apparatus for determining percent moisture

2.3.1.1 Oven - drying.

2.3.1.2 Desiccator.

2.3.1.3 Crucibles - porcelain.

2.3.2 Disposable Pasteur glass pipets - 1 mL.

2.3.3 Ultrasonic cell disruptor, Heat Systems, Ultrasonics, Inc., Model W-385 SONICATOR (475 Watt with pulsing capability, No. 305, 3/4 inch tapped high gain "Q" disruptor horn, or No. 208, 3/4 inch standard solid disruptor horn), or equivalent device with a minimum of 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.

Sonabox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.

2.3.4 Beakers - 400 mL.

2.3.5 Vacuum filtration apparatus.

2.3.5.1 Buchner funnel.

2.3.5.2 Filter paper - Whatman No. 41 or equivalent.

2.3.6 Kuderna-Danish (K-D) apparatus.

2.3.6.1 Concentrator tube - 10 mL, graduated (Kontes K-570040-1025 or equivalent).

2.3.6.2 Evaporative flask - 500 mL (Kontes K-570001-0500 or equivalent).

2.3.6.3 Snyder column - three-ball macro (Kontes K-503000-0121 or equivalent).

2.3.6.4 Snyder column - two-ball micro (Kontes K-569001-0219 or equivalent).

2.3.7 Silicon carbide boiling chips - approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

2.3.8 Water bath - heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

2.3.9 Balance - capable of accurately weighing ± 0.01 g.

2.3.10 Vials and caps - 2 mL for GC auto sampler.

2.3.11 Balance - analytical, capable of accurately weighing ± 0.0001g.

2.3.12 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C. (N-Evap by Organomation Associates, Inc., South Berlin, MA, or equivalent).

2.3.13 Pyrex glass wool.

2.3.14 Pasteur pipets - disposable.

2.3.15 Syringes - 0.5 mL volume.

2.4 Reagents

2.4.1 Sodium Sulfate - anhydrous powdered reagent grade, heated at 400°C for four hours, cooled in a desiccator, and stored in a glass bottle (Baker anhydrous powder, catalog #73898 or equivalent).

2.4.2 Methylene chloride, methanol, acetone, isooctane, 2-propanol, and benzene - pesticide residue analysis grade or equivalent.

2.4.3 Reagent water - defined as water in which an interferent is not observed at or above the CRQL of each parameter of interest.

2.4.4 Sodium Sulfite - reagent grade.

2.4.5 Base/Neutral and Acid Surrogate Spiking Solution

Surrogate standards are added to all samples and calibration solutions. The compounds specified are Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, 2-Chlorophenol-d_4, and 1,2-Dichlorobenzene-d_4. Prepare a surrogate standard spiking solution that contains Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, and 1,2-Dichlorobenzene-d_4 at a concentration of 100 ug/mL; Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, and 2-Chlorophenol-d_4 at a concentration of 150 ug/mL. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

2.4.6 Base/Neutral and Acid Matrix Spiking solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 100 ug/mL for base/neutrals and 150 ug/mL for acids. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

Bases/ Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

2.5 Low Level Sample Preparation

2.5.1 Decant and discard any water layer on a sediment sample. Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

Transfer 50 g of soil/sediment to 100 mL beaker. Add 50 mL of water and stir for 1 hour. Determine pH of sample with glass electrode and pH meter while stirring. Report pH value on appropriate data sheets. If the pH of the soil is greater than 11 or less than 5, contact the Technical Project Officer cited in the contract for instructions on how to handle the sample. Document the instructions in the SDG Narrative. Discard this portion of sample. NOTE: If limited sample volume is received, use 5 g of soil and 5 mL of water for the pH determination. Note this in the SDG Narrative.

2.5.2 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample to the nearest 0.1 g into a 400-mL beaker and add 60 g of anhydrous powdered sodium sulfate. Mix well. The sample should have a sandy texture at this point. Immediately, add 100 mL of 1:1 methylene chloride-acetone to the sample, then add the surrogates according to paragraph 2.5.2.3.

2.5.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the 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 sediment.

$$\frac{\text{g of sample - g of dry sample}}{\text{g of sample}} \times 100 = \text{\% moisture}$$

2.5.2.2 Weigh out two 30 g (record weight to nearest 0.1 g) portions for use as matrix and matrix spike duplicates according to paragraph 2.5.2. Add 0.5 mL of the BNA matrix spike solution to each of two portions. The frequency of MS/MSD analysis is given in Section IV, paragraph 8.6.

2.5.2.3 Add 0.5 mL of base/neutral and acid surrogate standard to the sample and each of the aliquots in 2.5.2.2.

2.5.2.4 Prepare a method blank with each group of low soil/sediment samples extracted. For semivolatile analyses, a method blank for low soil/sediment samples consists of 30 g of sodium sulfate (see paragraph 2.4.1), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in Section IV, paragraph 8.7

2.5.3 Place the bottom surface of the tip of the 3/4 inch disruptor horn about 1/2 inch below the surface of the solvent but above the sediment layer.

2.5.4 Sonicate for 1 1/2 minutes with the W-385 (or 3 minutes with the W-375), using No. 208, 3/4 inch standard disruptor horn with output control knob set at 10 (or No. 305, 3/4 inch tapped high gain "Q" disruptor horn at 5) and mode switch on "1 sec. pulse" and % duty cycle knob set at 50% (if using a sonicator other than Models W-375 or W-385, contact the Project Officer for appropriate output settings). Do NOT use MICROTIP probe.

2.5.5 Decant and filter extracts through Whatman #41 filter paper using vacuum filtration or centrifuge and decant extraction solvent.

2.5.6 Repeat the extraction two more times with 2 additional 100 mL portions of 1:1 methylene chloride-acetone. Before each extraction, make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula or, very carefully, with the tip of the unenergized probe. Decant the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with 1:1 methylene chloride-acetone.

2.5.6.1 If the sample is to be screened from the low level method prior to GPC, take 5.0 mL and concentrate to 1.0 mL following paragraph 3.6.1 or 3.6.2, but note that the final volume for screening is 1.0 mL, not 0.5 mL. Screen the extract as per Section III, paragraph 1., "Screening of Extractable Organic Extracts."

2.5.6.2 After GC/FID or GC/MS screening, transfer the remainder of the 1 mL back to the total extract from paragraph 2.5.6. CAUTION: To minimize sample loss, autosamplers which pre-flush samples through the syringe should not be used.

2.6 Concentration and Solvent Exchange

2.6.1 Low level soil/sediment samples prepared by the procedures in paragraph 2.5 will result in extracts containing a mixture of acetone and methylene chloride. Because all soil/sediment sample extracts must be subjected to GPC clean up prior to analysis, the majority of the acetone must be removed from the extract, otherwise it will have adverse effects on the GPC column. To remove the acetone from the sample extract, follow the steps in 2.6.2-2.6.4.

2.6.2 Transfer the extract to a Kuderna-Danish (K-D) concentrator consisting of a 10 mL concentrator tube and a 500 mL evaporative flask. Other concentration devices or techniques may be used if equivalency is demonstrated for all extractable compounds listed in Exhibit C.

2.6.3 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Do not allow the evaporator to go dry.

2.6.4 Dilute the extract to 10.0 mL with methylene chloride, and proceed with GPC clean up (see paragraph 3).

3. Extract Cleanup by Gel Permeation Chromatography

Gel permeation chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of 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. A cross-linked divinyl benzenestyrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

GPC is required for all soil/sediment samples, regardless of concentration level, for the elimination of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds from the sample extract. GPC is appropriate for both polar and non-polar analytes, therefore, it can be used effectively to clean up extracts containing a broad range of analytes.

Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue ultimately will reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol especially is susceptible to this problem.

In the event that the Laboratory fails to appropriately employ GPC clean-up procedures, the Agency will require the clean up and reanalysis of all affected samples or sample extracts at no additional cost to the Agency.

3.1 Apparatus and Materials

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

3.1.1 Gel permeation chromatography (GPC) cleanup device. NOTE: GPC cleanup is required for all soil/sediment extracts.

Gel permeation chromatography system - GPC Autoprep Model 1002 A or B (Analytical Biochemical Laboratories, Inc., or equivalent) Systems that perform very satisfactorily also 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 paragraph 3.4.

3.1.1.1 Chromatographic column - 700 mm x 25 mm i.d. 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 so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

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

3.1.1.3 Bio Beads (S-X3) - 200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent). An additional 5 gm 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 also can pass through the column screens and damage the valve.

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

3.1.1.5 Strip chart recorder, recording integrator or laboratory data system.

3.1.1.6 Syringe - 10-mL with Luerlok fitting.

3.1.1.7 Syringe filter assembly, disposable - Bio-Rad "Prep Disc" sample filter assembly #343-0005, 25 mm, and 5 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

3.1.1.8 A description of a manual system assembled from parts can be found in Wise, R.H., Bishop, D.F., Williams, R.T. & Austern, B.M. "Gel Permeation Chromatography in the GC/MS Analysis of Organics in Sludges" U.S. EPA, Municipal Environmental Research Laboratory, Cincinnati, Ohio, 45268.

3.2 Reagents

3.2.1 GPC Calibration Solution - prepare a calibration solution in methylene chloride containing the following analytes (in elution order):

Compound	mg/mL
corn oil	25.0
bis(2-ethylhexyl)phthalate	1.0
methoxychlor	0.2
perylene	0.02
sulfur (optional)	0.08

NOTE: If used, sulfur is not very soluble in methylene chloride, however, 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.

Store the calibration solution in an amber glass bottle with a Teflon-lined screw-cap at 4°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 6 months, or more frequently if necessary.

3.3 Column Preparation

3.3.1 Weigh out 70 gm of Bio Beads (SX-3). Transfer them to a quart 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 gm of Bio Beads in a 125 mL bottle or a beaker, using 25 mL of methylene chloride.

3.3.2 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).

3.3.3 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.

3.3.4 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.

3.3.5 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.

3.3.6 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 paragraph 3.3.5 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.

3.3.7 Push the plunger until it meets the gel, then compress the column bed about four centimeters.

3.3.8 Pack the optional 5 cm column with approximately 5 gm of preswelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

3.3.9 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 10/1000" ID x 2". Pump methylene chloride through the column at a rate of 5 mL/min for one hour.

3.3.10 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 paragraph 3.3.9) in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect 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.

3.3.11 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.

3.4 Calibration of the GPC Column

3.4.1 Using a 10 mL syringe, load sample loop #1 with calibration solution (paragraph 3.2). With the ABC automated system, the 5 mL sample loop requires a minimum of 8 mL of the calibration solution. Use a firm, continuous pressure to push the sample onto the loop. Switch the valve so that GPC flow is through the UV flow-through cell.

3.4.2 Inject the calibration solution and obtain a UV trace showing a discrete peak for each component. Adjust the detector and/or recorder sensitivity to produce a UV trace that meets the following requirements. Differences between manufacturer's cell volumes and detector sensitivities may require a dilution of the calibration solution to achieve similar results. An analytical flow-through detector cell will require a much less concentrated solution than the semi-prep cell and, therefore, the analytical cell is <u>not</u> acceptable for use.

- o Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- o Corn oil and phthalate peaks must exhibit >85% resolution.
- o Phthalate and methoxychlor peaks must exhibit >85% resolution.
- o Methoxychlor and perylene peaks must exhibit >85% resolution.
- o Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

3.4.3 Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis(2-ethylhexyl)phthalate and after the elution of

the corn oil. Stop eluate collection shortly after the elution of perylene. Collection should be stopped before sulfur elutes. Use a "wash" time of 10 minutes after the elution of sulfur. Each laboratory is required to establish its specific time sequences.

3.4.4 Verify the flow rate by collecting column eluate for 10 minutes in a graduated cylinder and measure the volume, which should be 45-55 mL (4.5-5.5 mL/min). If the flow rate is outside of this range, corrective action must be taken, as described above. Once the flow rate is within the range of 4.5-5.5 mL/min, record the column pressure (should be 6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared. A UV trace that does not meet the criteria in paragraph 3.4.2 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.

3.4.5 Reinject the calibration solution after appropriate collect and dump cycles have been set, and the solvent flow and column pressure have been established.

3.4.5.1 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.

3.4.5.2 The retention times for bis(2-ethylhexyl)phthalate and perylene must not vary more than $\pm$5% between calibrations. If the retention time shift is >5%, 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.

3.4.6 Analyze a GPC blank by loading 5 mL of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by GC/MS. If the blank exceeds one half the CRQL of any analyte, 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.

3.5 Sample Extract Cleanup

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 outgassing of the methylene chloride is 22°C.

3.5.1 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 a 1:1 glycerol:water solution must be diluted and loaded into several loops. Similarly, extracts containing more than 500 mg of nonvolatile residue per 5 mL of extract must be diluted and loaded into several loops. The nonvolatile residue may be determined by evaporating a 100 uL aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.

3.5.2 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.

3.5.3 Attach the syringe to the turn lock on the injection port. Use firm, continuous pressure to push the sample onto the 5-mL sample loop. If the sample is difficult to load, some part of the system may be blocked. Take appropriate corrective action. If the back pressure is normal (6-10 psi) the blockage is probably in the valve. Blockage may be flushed out of the valve by reversing the inlet and outlet tubes and pumping solvent through the tubes (this should be done before sample loading).

NOTE: Approximately 2 mL of the extract remains in the lines between the injection port and the sample loop; excess sample also passes through the sample loop to waste.

3.5.4 After loading a loop, and before removing the syringe from the injection port, index the GPC to the next loop. This will prevent loss of sample caused by unequal pressure in the loops.

3.5.5 After loading each sample loop, wash the loading port with methylene chloride in a PTFE wash bottle to minimize cross contamination. Inject approximately 10 mL of methylene chloride to rinse the common tubes.

3.5.6 After loading all the sample loops, index the GPC to the OO position, switch to the "RUN" mode and start the automated sequence. Process each sample using the collect and dump cycle times established in 3.4.

3.5.7 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:

- o Change in solvent flow rate, caused by channeling in the column or changes in column pressure.
- o 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.
- o Leaks in the system or significant variances in room temperature.

3.5.8 Concentrate the extract as per paragraphs 3.6.1 or 3.6.2.

3.5.9 Calibrate the GPC at least once per week, following the procedure outlined in 3.4. The UV trace must meet the requirements in paragraph 3.4.2. In addition, the retention times of the calibration compounds must be within ±5% of their retention times in the previous calibration. A copy of the UV trace of the calibration solution must be submitted with the data for the associated samples.

3.5.10 If the requirements in paragraphs 3.4.2 and 3.5.9 cannot be met, the column may be cleaned by processing several 5 mL volumes of butyl chloride throught the system. Butyl chloride removes the discoloration and particles that may have precipitated out of the methylene chloride extracts. If a guard column is being used. replace it with a new one. This may correct the problem. If column maintenance does not restore the performance of the column, the column must be repacked with new packing and recalibrated.

3.6 Final Concentration of Extract

3.6.1 Transfer the sample extract to a K-D evaporator, attach the micro-Snyder column to the concentrator tube and add a silicon carbide boiling chip to the concentrator tube. Pre-wet the Snyder column with 0.5 mL of methylene chloride. Place the K-D apparatus on the hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water

temperature as required to complete the concentration in 5 to 10 minutes. When the apparent volume of the liquid reaches 0.4 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 the lower joint into the concentrator tube with 0.1 mL of methylene chloride. Adjust the final volume to 0.5 mL with methylene chloride. Concentrating the extract to 0.5 mL will result in no loss of sensitivity despite the volume of extract (5 mL) not recovered after GPC.

3.6.2 Nitrogen evaporation technique (taken from ASTM Method D 3086)

The following method may be used for final concentration of the semivolatile extract instead of the procedures in paragraph 3.6.1. Place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume to below 0.5 mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). CAUTION: New plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

The internal wall of the tube must be rinsed down several times with methylene chloride during the operation. During evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry. Concentrating the extract to 0.5 mL will result in no loss of sensitivity despite the volume of extract (5 mL) not recovered after GPC.

3.7 Store all extracts at 4°C (±2°C) in the dark in Teflon-sealed containers.

3.8 If the extract was not screened prior to GPC, proceed to Section III for the screening procedures. If the extract was screened prior to GPC, proceed with the GC/MS analysis in Section IV.

SECTION III

SCREENING OF SEMIVOLATILE ORGANIC EXTRACTS

1. Summary of Method

1.1 The solvent extracts of water and soil/sediment are screened on a gas chromatograph/flame ionization detector (GC/FID) using a fused silica capillary column (FSCC). For water samples, the results of the screen may be used to determine an appropriate dilution factor for the GC/MS analysis of the sample extract. For soil/sedimant samples, the results of the screen are used to determine which of the two sample preparation procedures (low or medium) is required, and to determine an appropriate dilution factor for GC/MS analysis. The results of the screen may be used also to assist the analyst in performing Gel Permeation Chromatography (GPC) clean up procedures on extracts of either water or soil/sediment samples.

2. Apparatus and Materials

2.1 Gas chromatograph - an analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port must be designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

2.1.1 Above GC equipped with flame ionization detector.

2.1.2 GC column - 30 m x 0.32 mm, 1 micron film thickness, silicone coated, fused silica capillary column (J & W Scientific DB-5 or equivalent).

3. Reagents

3.1 Methylene chloride - pesticide residue analysis grade or equivalent.

3.2 GC calibration standard - prepare a standard solution containing phenol, phenanthrene and di-n-octylphthalate.

3.2.1 Stock standard solutions (1.00 ug/uL) - Stock standard solutions can be prepared from pure standard materials or purchased solutions.

3.2.1.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality methylene chloride and dilute to volume in a 10 mL volumetric flask. Larger volumes may be used at the convenience of the analyst. If compound purity is assayed at 97% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source (see Exhibit E).

3.2.1.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store 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. Stock standard solutions must be replaced after six months, or sooner, if comparison with quality control check samples indicates a problem. Standards prepared from gases or reactive compounds such as styrene must be replaced after two months, or sooner, if comparison with quality control check samples indicates a problem.

3.2.2 Prepare a working standard mixture of the three compounds in methylene chloride. The concentration must be such that the volume injected equals 50 ng of each compound. The storage and stability requirements are the same as specified in paragraph 3.2.1.2.

4. GC Calibration

4.1 At the beginning of each 12 hour shift, inject the GC calibration standard. The following criteria must be met:

4.1.1 The GC must be standardized for half scale response from 50 ng of phenanthrene.

4.1.2 The GC must adequately separate phenol from the solvent front.

4.1.3 A minimum of quarter scale response for 50 ng of di-n-octylphthalate must be exhibited.

5. GC/FID Screening

5.1 Suggested GC operating conditions are as follows:

- Initial Column Temperature Hold - 50°C for 4 minutes.
- Column Temperature Program - 50-280°C at 8 degrees/min.
- Final Column Temperature Hold - 280°C for 8 minutes.
- Injector - Grob-type, splitless.
- Sample Volume - 1-2 uL.
- Carrier Gas - Helium at 30 mL/sec.

5.2 Inject the GC calibration standard and ensure the criteria specified in 4 are met before injecting samples. Estimate the response for 10 ng of phenanthrene.

5.3 Inject the appropriate extracts from Section II, including blanks.

6. Interpretation of Chromatograms

6.1 Water

6.1.1 If no sample peaks are detected, or all are less than full scale deflection, the undiluted extract is analyzed on GC/MS.

6.1.2 If any sample peaks are greater than full scale deflection, calculate the dilution necessary to reduce the major peaks to between half and full scale deflection. Use this dilution factor to dilute the extract for GC/MS analysis.

6.2 Soil/Sediment

6.2.1 If no sample peaks from the extract (from low or medium level preparation) are detected, or all are less than 10% full scale deflection, the sample must be prepared by the low level protocol, Section II, Part C, beginning at 2.

6.2.2 Peaks are detected at greater than 10% full scale deflection and less than or equal to full scale deflection.

6.2.2.1 If the screen is from the medium level extract, proceed with GC/MS analysis of this extract with appropriate dilution if necessary.

6.2.2.2 If screen is from the low level extract, discard extract and prepare sample by medium level method for GC/MS analysis.

6.2.3 Peaks are detected at greater than full scale deflection.

6.2.3.1 If the screen is from the medium level preparation, calculate the dilution necessary to reduce the major peaks to between half and full scale deflection. Use this dilution factor to dilute the extract. This dilution is analyzed by GC/MS for extractable organics.

6.2.3.2 If the screen is from the low level preparation, discard the extract and prepare a sample by the medium level method for GC/MS analysis.

7. GC/MS Analysis

7.1 Use the information from paragraph 6 (Interpretation of Chromatograms) to perform the GC/MS analysis, beginning Section IV, GC/MS Analysis of Semivolatiles.

7.2 The information from paragraph 6 may be usefull also in processing sample extracts through GPC cleanup.

NOTE: The choice of screening sample extracts before or after GPC cleanup is left to the laboratory.

SECTION IV

GC/MS ANALYSIS OF SEMIVOLATILES

1. Summary of Method

This method is to be used for the GC/MS analysis of semivolatiles screened by Section III protocols and for confirmation of pesticides/Aroclors identified by GC/EC, if concentrations permit.

2. Apparatus and Materials

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

2.1 Gas chromatograph/mass spectrometer system.

2.1.1 Gas chromatograph - an analytical system complete with a temperature programmable gas chromatograph suitable for splitless injection and all required accessories including syringes, analytical columns and gases.

2.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm) bonded-phase silicone coated fused silica capillary column (J&W Scientific DB-5 or equivalent). A film thickness of 1.0 micron is recommended because of its larger capacity. A film thickness of 0.25 micron may be used.

2.1.3 Mass Spectrometer - capable of scanning from 35 to 500 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the instrument performance criteria in Table 1 when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet. The instrument conditions required for the acquisition of the DFTPP mass spectrum are given in paragraph 4.3.4. NOTE: DFTPP criteria must be met before any sample extracts are analyzed. Any samples analyzed when DFTPP criteria have not been met will require reanalysis at no cost to the Agency.

2.1.4 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 acceptable performance criteria (Exhibit E), may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

2.1.5 Data system - a computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specific 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 be available that allows integrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows comparing sample spectra against reference spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

2.1.6 Syringes - 2 uL and 10 uL volumes.

3. Reagents

3.1 Internal standards - 1,4 Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}.

An internal standard solution can be prepared by dissolving 100 mg of each compound in 50 mL of methylene chloride. It may be necessary to use 5 to 10 percent benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. The resulting solution will contain each standard at a concentration of 2000 ng/uL. A 10 uL portion of this solution should be added to each 1 mL of sample extract. This will result in 40 ng of each internal standard in the 2 uL volume of extract injected into the GC/MS.

3.2 Prepare calibration standards at a minimum of five concentration levels (20, 50, 80, 120, and 160 total ng per 2 uL). Each calibration standard should contain each compound of interest and each surrogate. 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 require only a four-point initial calibration at 50, 80, 120, and 160 total ng, since detection at less that 50 ng per injection is difficult. Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at -10°C to -20°C in screw-cap amber bottles with teflon liners. Fresh standards should be prepared every twelve months at a minimum. The continuing calibration standard (50 ng) should be prepared weekly and stored at 4°C (±2°C).

In order to facilitate the confirmation of pesticides and Aroclors from the semivolatile library search data (see Exhibit D PEST, paragraph 17), the laboratory may wish to include the pesticide/Aroclor target compounds listed in Exhibit C in the semivolatile continuing calibration standard. The laboratory may add any or all of these compounds to the semivolatile continuing calibration standard, but at a concentation of 10 ng/uL or less. If added to this GC/MS standard, these additional analytes are <u>not</u> reported on the semivolatile calibration form (Form VII), but must be included in the quantitation report for the continuing calibration standard. As only a single point calibration would be performed, no %RSD or percent difference criteria would apply to these additional analytes.

3.3 Instrument performance check solution - prepare a solution of decafluorotriphenylphosphine (DFTPP), such that a 2 uL injection will contain 50 ng of DFTPP. The DFTPP also be included in the calibration standards at this level.

4. Instrument Operating Conditions

4.1 Gas Chromatograph

The following are the recommended GC analytical conditions:

Initial Column Temperature Hold	–	40°C for 4 minutes
Column Temperature Program	–	40-270°C at 10 degrees/min.
Final Column Temperature Hold	–	270°C for 10 minutes
Injector Temperature	–	250-300°C
Transfer Line Temperature	–	250-300°C
Source Temperature	–	according to manufacturer's specifications
Injector	–	Grob-type, splitless
Sample Volume	–	2 uL
Carrier Gas	–	Helium at 30 mL/sec

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.

4.2 Mass Spectrometer

The following are the required mass spectrometer analytical conditions:

Electron Energy - 70 volts (nominal)

Mass Range - 35 to 500 amu

Scan Time - not to exceed 1 second per scan

4.3 The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as 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 (paragraph 3.3).

4.3.1 Prior to the analysis of any samples, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing decafluorotriphenylphosphine (DFTPP).

4.3.2 The analysis of the instrument performance check solution may be performed as follows:

- o As an injection of up to 50 ng of DFTPP into the GC/MS
- o By adding 50 ng of DFTPP to the calibration standards (paragraph 3.2) and analyzing the calibration standard.

4.3.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given below.

TABLE 1
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA FOR QUADRAPOLE MASS SPECTROMETERS

Mass	Ion Abundance Criteria
51	30.0 - 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Present
70	Less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see note)
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	Greater than 0.75 percent of mass 198
441	Present but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

4.3.4 The abundance criteria listed above must be met for a 50 ng injection of DFTPP. The mass spectrum of DFTPP must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of DFTPP. Note: All subsequent standards, samples, MS/MSD, and blanks associated with a DFTPP analysis must use identical mass spectrometer instrument conditions.

4.3.5 The criteria above are based on adherence to the acquisition specifications identified in paragraph 4.3.4. The criteria are based on performance characteristics of instruments currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for 54 target compounds (see Table 2), are designed to control and monitor instrument performance associated with the requirements of this Statement of Work.

4.3.6 The instrument performance check solution must be analyzed once at the beginning of each 12-hour period during which samples or standards are analyzed.

The twelve (12) hour time period for a GC/MS system instrument performance check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after twelve (12) hours has elapsed according to the system clock.

5. Calibration

5.1 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 a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target compounds.

5.2 The internal standards are added to all calibration standards and all sample extracts (including blanks, matrix spikes, and matrix spike duplicates) just prior to analysis by GC/MS. A 10 uL aliquot of the internal standard solution should be added to a 1 mL aliquot of calibration standards. The internal standards specified in paragraph 3.1 should permit most of the semivolatile target compounds to have relative retention times of 0.80 to 1.20, using the assignments of internal standards to target compounds given in Table 2.

5.3 The quantitation ions for each internal standard are given in Table 3. Use the primary ion listed in Table 3 for quantitation, unless interferences are present. If interferences prevent the use of the primary ion for a given internal standard, use the secondary ion(s) listed in Table 3.

5.4 Prepare calibration standards at a minimum of five concentration levels for each target compound and surrogate, as specified in paragraph 3.2. Analyze 2 uL of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound including the surrogate compounds. A 2 uL injection is required. Calculate relative response factors (RRF) for each compound using Equation 1.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured (see Table 4)

A_{is} = Area of the characteristic ion for the specific internal standard (see Table 3)

C_{is} = Concentration of the internal standard (ng/uL)

C_x = Concentration of the compound to be measured (ng/uL)

5.5 The average relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values for the initial calibration using the following equation:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x - \bar{x})^2}{n-1} \right|^{1/2}$$

Where

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

TABLE 2

SEMIVOLATILE INTERNAL STANDARDS WI[illegible] CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}	Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
Phenol	Nitrobenzene	Hexachlorocyclo-pentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl-phthalate
bis(2-Chloroethyl) ether	Isophorone	2,4,6-Trichloro-phenol	[illegible]-nitrosodi-phenylamine	Butylbenzyl phthalate	Benzo(b)fluor-anthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichloro-phenol	4-Bromophenyl phenyl ether	3,3'-Dichloro-benzidine	Benzo(k)fluor-anthene
1,3-Dichlorobenzene	2,4-Dimethyl-phenol	2-Chloronaphthalene	Hexachloro-benzene	Benzo(a)-anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	bis(2-Chloro-ethoxy)methane	2-Nitroaniline	Pentachloro-phenol	bis(2-Ethyl-hexyl)phthalate	Indeno(1,2,3-cd)-pyrene
1,2-Dichlorobenzene	2,4-Dichloro-phenol	Dimethyl Phthalate	Phenanthrene	Chrysene	Dibenz(a,h)-anthracene
2-Methylphenol	1,2,4-Trichloro-benzene	Acenaphthylene	Carbazole	Terphenyl-d_{14} (surr)	Benzo(g,h,i)-perylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene		
4-Methylphenol	4-Chloroaniline	Acenaphthene	Di-n-butyl-phthalate		
N-Nitroso-Di-n-propylamine	Hexachloro-butadiene	2,4-Dinitrophenol	Fluoranthene		
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol			
2-Fluorophenol (surr)	2-Methylnaphth-alene	Dibenzofuran			
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene			
2-Chlorobenzene-d_4 (surr)		2,6-Dinitrotoluene			
1,2-Dichlorobenzene-d_4 (surr)		Diethyl phthalate			
		4-Chlorophenyl phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromo-phenol (surr)			

surr = surrogate compound

TABLE 3
CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR SEMIVOLATILE COMPOUNDS

INTERNAL STANDARDS	Primary Ion	Secondary Ions
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenapthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

TABLE 4
CHARACTERISTIC IONS FOR SEMIVOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
2,2'-oxybis(1-Chloropropane)	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121

(continued)

TABLE 4 (continued)
CHARACTERISTIC IONS FOR SEMIVOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Carbazole	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-Octyl phthalate	149	—
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
SURROGATES		
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d_5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
2-Chlorophenol-d_4	132	68, 134
1,2-Dichlorobenzene-d_4	152	115, 150

5.6 Response factor criteria have been established for the calibration of the semivolatile target compounds and semivolatile surrogate compounds.

5.6.1 The response factors of the compounds listed in Table 5 must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to four semivolatile target and surrogate compounds. However, the RRFs for those four compounds must be greater than 0.010, and the %RSD of those four compounds must be less than or equal to 40.0% for the initial calibration to be acceptable.

TABLE 5
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF SEMIVOLATILE TARGET COMPOUNDS

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Phenol	0.800	20.5	25.0
bis(-2-Chloroethyl)ether	0.700	20.5	25.0
2-Chlorophenol	0.800	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
2-Methylphenol	0.700	20.5	25.0
4-Methylphenol	0.600	20.5	25.0
N-Nitroso-Di-propylamine	0.500	20.5	25.0
Hexachloroethane	0.300	20.5	25.0
Nitrobenzene	0.200	20.5	25.0
Isophorone	0.400	20.5	25.0
2-Nitrophenol	0.100	20.5	25.0
2,4-Dimethylphenol	0.200	20.5	25.0
bis(-2-Chloroethoxy)methane	0.300	20.5	25.0
2,4-Dichlorophenol	0.200	20.5	25.0
1,2,4-Trichlorobenzene	0.200	20.5	25.0
Naphthalene	0.700	20.5	25.0
4-Chloro-3-methylphenol	0.200	20.5	25.0
2-Methylnaphthalene	0.400	20.5	25.0
2,4,6-Trichlorophenol	0.200	20.5	25.0
2,4,5-Trichlorophenol	0.200	20.5	25.0
2-Chloronaphthalene	0.800	20.5	25.0
Acenaphthylene	1.300	20.5	25.0
2,6-Dinitrotoluene	0.200	20.5	25.0
Acenaphthene	0.800	20.5	25.0
Dibenzofuran	0.800	20.5	25.0
2,4-Dinitrotoluene	0.200	20.5	25.0
4-Chlorophenyl-phenylether	0.400	20.5	25.0
Fluorene	0.900	20.5	25.0
4-Bromophenyl-phenylether	0.100	20.5	25.0
Hexachlorobenzene	0.100	20.5	25.0
Pentachlorophenol	0.050	20.5	25.0

(continued)

TABLE 5 (continued)
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING
CALIBRATION OF SEMIVOLATILE TARGET COMPOUNDS

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Phenanthrene	0.700	20.5	25.0
Anthracene	0.700	20.5	25.0
Fluoranthene	0.600	20.5	25.0
Pyrene	0.600	20.5	25.0
Benzo(a)anthracene	0.800	20.5	25.0
Chrysene	0.700	20.5	25.0
Benzo(b)fluoranthene	0.700	20.5	25.0
Benzo(k)fluoranthene	0.700	20.5	25.0
Benzo(a)pyrene	0.700	20.5	25.0
Indeno(1,2,3-cd)pyrene	0.500	20.5	25.0
Dibenzo(a,h)anthracene	0.400	20.5	25.0
Benzo(g,h,i)perylene	0.500	20.5	25.0
Nitrobenzene-d_5	0.200	20.5	25.0
2-Fluorobiphenyl	0.700	20.5	25.0
Terphenyl-d_{14}	0.500	20.5	25.0
Phenol-d_5	0.800	20.5	25.0
2-Fluorophenol	0.600	20.5	25.0
2-Chlorophenol-d_4	0.800	20.5	25.0
1,2-Dichlorobenzene-d_4	0.400	20.5	25.0

5.6.2 The following compounds have no Maximum %RSD, or Maximum %Difference criteria; however, these compounds must meet a minimum RRF criterion of 0.010:

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

5.7 A check of the calibration curve must be performed once every 12 hours (see paragraph 4.3.6 for the definition of the twelve-hour time period). Check the relative response factors of those compounds for which RRF values have been established. If these criteria are met, the relative response factors for all compounds are calculated and reported. A percent difference of the daily relative response factor (12 hour) compared to the average relative response factor from the initial curve is calculated. Calculate the percent difference for each compound and compare with the maximum percent difference criteria listed above. For negative percent difference values, the value must be greater than or equal to -25.0%, but less than 0%.

As with the initial calibration, up to four semivolatile target compounds in Table 5 may fail to meet the minimum RRF or maximum %D criteria, but the RRFs of those four compounds must be greater than or equal to 0.010, and the percent differences must be less than or equal to 40.0% for the continuing calibration to be acceptable.

5.8 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 0.50 minutes (30 seconds) from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction, and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is necessary.

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

5.10 If time remains in the 12 hour time period after meeting the 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 acceptance criteria. Quantify all sample results against the initial calibration standard that is the same concentration as the continuing calibration standard (50ng/2uL).

5.11 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. The DFTPP may be included in the continuing calibration standard.

5.12 If the injection of the instrument performance check solution meets the criteria in Table 1, calculate the response factors for the continuing calibration standard and the percent difference of the response factors from the mean response factors in the initial calibration.

5.13 The response factors from the continuing calibration standard must meet the criteria in Table 5 prior to the analysis of any blanks or samples.

6. Sample Analysis

6.1 Sample extracts may be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, and continuing calibration requirements above. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

6.2 Internal standard solution is added to each sample extract. Add 10 uL of internal standard solution to each accurately measured 1.0 mL of water sample extract. For soil samples and water samples subjected to GPC, add 5 uL of internal standard solution to each accurately measured 0.5 mL of sample extract. This will result in a concentration of 20 ng/uL of each internal standard.

6.3 Make any extract dilution indicated by characterization prior to the addition of internal standards. If any further dilutions of water or soil/sediment extracts are made, additional internal standards must be added to maintain the required 40 ng (20 ng/uL) of each internal standard in the extract volume.

6.4 Inject 2 uL of the sample extract into the GC/MS. This 2 uL volume must contain 40 ng of each internal standard.

7. Qualitative Analysis

7.1 The compounds listed in the Target Compound List (TCL), Exhibit C, shall be identified by an analyst competent in the interpretation of mass spectra (see Exhibit A, Section III) 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:

- o Elution of the sample component at the GC relative retention time as the standard component.
- o Correspondence of the sample component and standard component mass spectra.

7.1.1 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 samples analyzed during the same 12-hour time period as the initial calibration standards, compare the sample retention times to those from the 50 ng calibration standard. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

7.1.2 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 DFTPP daily instrument performance requirements. These standard spectra may be obtained from the run used to obtain reference RRTs.

7.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

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

7.1.3.2 The relative intensities of ions specified in 7.1.3.1 must agree within ±20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

7.1.3.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Task III, 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 "J", e.g., "3J."

7.1.4 If a compound cannot be verified by all of the criteria in 7.1.3, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantification in paragraph 8.

7.2 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the most recent release of the NIST/EPA/MSDC mass spectral library, shall be used.

7.2.1 Up to 20 nonsurrogate organic compounds of greatest apparent concentration not listed in Exhibit C for the semivolatile fraction shall be identified tentatively via a forward search of the NIST/EPA/MSDC mass spectral library. Substances with responses less than 10% of the nearest internal standard are not required to be searched in this fashion. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. NOTE: Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Peaks that are suspected as aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be searched and reported but not counted as part of the 20 most intense non-target semivolatile compounds.

7.2.2 Guidelines for making tentative identification.

7.2.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

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

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

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

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

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

8. Quantitation

8.1 Target components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte (see Table 2). The EICP area of characteristic ions of analytes listed in Table 4 are used for quantitation. 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 initializing and dating the changes made to the report.

Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 0.50 minutes (30 seconds) from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. For samples analyzed during the same 12-hour time period as the initial calibration standards, compare the internal standard responses and retention times to those of the 50 ng 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. The criteria are described in detail in the instructions for Form VIII, Internal Standard Area Summary. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required. The samples or standards with EICP areas outside the limits must be re-analyzed, and treated according to paragraphs 8.1.1 and 8.1.2 below. If corrections are made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that does meet the EICP criteria. After corrections are made, the re-analysis of samples analyzed while the system was malfunctioning is required.

8.1.1 If after re-analysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with EICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

8.1.2 If the re-analysis of the sample does not solve the problem, i.e., the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the SDG Narrative all inspection and corrective actions taken.

8.1.3 Do not re-analyze MS/MSD samples that do not meet the EICP area limits.

8.2 The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. For samples analyzed during the same 12-hour time period as the initial calibration standards, use the RRF values from the 50 ng calibration standard. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion.

When target compounds are below contract required quantitation limits (CRQL) but the spectrum meets the identification criteria, report the concentration with a "J." For example, if CRQL is 10 ug/L and concentration of 3 ug/L is calculated, report as "3J." Calculate the concentration in the sample using the relative response factor (RRF) as determined in 5.4 and the following equation:

Water

$$\text{Concentration} \quad \text{ug/L} = \frac{(A_x)(I_s)(V_t)(Df)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where,

A_x - Area of the characteristic ion for the compound to be measured

A_{is} - Area of the characteristic ion for the internal standard

I_s - Amount of internal standard injected in nanograms (ng)

V_o - Volume of water extracted in milliliters (mL)

V_i - Volume of extract injected in microliters (uL)

V_t - Volume of the concentrated extract in microliters (uL)

Df - Dilution Factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

Soil/Sediment

$$\text{Concentration (Dry weight basis)} \quad \text{ug/kg} = \frac{(A_x)(I_s)(V_t)(Df)(2.0)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where,

A_x, I_s, A_{is} are as given for water, above.

V_t - Volume of the concentrated extract in microliters (uL)

V_i - Volume of extract injected in microliters (uL)

$D = \frac{100 - \%\ \text{moisture}}{100}$

W_s - Weight of sample extracted in grams (g)

Df - Dilution Factor. The dilution factor for analysis of soil samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + mL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator 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 0.5 mL, rather than 1.0 mL for water samples not subjected to GPC, maintains the sensitivity of the soil method comparable to that of the water method, but correction of the numerical result is still required.

8.3 An estimated concentration for non-target components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used. The formula for calculating concentrations is the same as in paragraph 8.2. 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 should be calculated for all tentatively identified compounds as well as those identified as unknowns.

8.4 If the on-column concentration of any compound in any sample exceeds the initial calibration range, that sample extract must be diluted, the internal standard concentration readjusted, and the sample extract reanalyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

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

8.4.2 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.

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

8.4.4 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the SDG Narrative.

8.5 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits (see Table 6) and report on appropriate form.

8.5.1 Calculate the concentrations of the surrogate compounds using the same equations as used for the target compounds. Calculate the recovery of each surrogate as follows:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

8.5.2 Determine if the sample surrogate recovery meets specifications as follows:

- o The eight semivolatile surrogates can be divided into three groups: base/neutral compounds (Nitrobenzene-d_5, 2-Fluorobiphenyl, and Terphenyl-d_{14}); acid compounds (Phenol-d_5, 2-Fluorophenol, and 2,4,6-Tribromophenol); and compounds with advisory QC limits (2-Chlorophenol-d_4 and 1,2-Dichlorobenzene-d_4.
- o If a single surrogate recovery from any group is not within the contract windows, the sample does not require reanalysis or re-extraction.
- o If a single surrogate recovery from the base/neutral group and a single surrogate recovery from the acid group are not within the contract windows, the sample does not require reanalysis or re-extraction.
- o Do not reanalyze or re-extract if only surrogates with advisory QC limits are not within the contract windows.

8.5.3 If the sample surrogate recovery does not meet specifications (i.e., if two base/neutral or two acid surrogates are out of limits or if recovery of any one base/neutral or acid surrogate is below 10%), the following are required:

- o Check to be sure that there are no errors in calculations, surrogate solutions, and internal standards. Also check instrument performance.
- o Reanalyze the sample if none of the above reveal a problem.
- o If surrogate recoveries in a blank do not meet specifications, the blank may be reanalyzed alone.
- o Do not reanalyze dilutions if surrogate recoveries are outside the limits.
- o Never reanalyze the matrix spike or matrix spike duplicate (MS/MSD), even if surrogate recoveries are outside the limits.
- o If the sample associated with the matrix spike and matrix spike duplicate does not meet specifications, it should be reanalyzed only if the MS/MSD surrogate recoveries are within the limits. If the sample and associated MS/MSD show the same pattern (i.e., outside the limits), then the sample does not require reanalysis and a re-analysis must not be submitted.

  Document in the narrative the similarity in surrogate recoveries.

8.5.4 If the reanalysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analysis with surrogate spike recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

8.5.5 If none of the steps in paragraph 8.5.3 or 8.5.4 solves the problem, then, except as noted below, re-extract and reanalyze the sample. If the re-extraction and reanalysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analysis with surrogate recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

- o If surrogate recoveries in a blank do not meet specifications even after reanalysis, all of the samples associated with that blank must be re-extracted along with the blank. The blank is intended to detect contamination in samples processed at the same time.
- o Do not re-extract diluted samples if surrogate recoveries are outside the limits.
- o Never re-extract the matrix spike or matrix spike duplicate (MS/MSD), even if surrogate recoveries are outside the limits.
- o If the sample associated with the matrix spike and matrix spike duplicate does not meet specifications after reanalysis, it should be reextracted only if the reanalysis surrogate recoveries are not within the limits and MS/MSD surrogate recoveries are within the limits. If the sample and associated MS/MSD show the same pattern (i.e., outside the limits), then the sample does not require reanalysis and a reanalysis must not be submitted.

  Document in the narrative the similarity in surrogate recoveries.

8.5.6 If the re-extraction and reanalysis of the sample does not solve the problem (i.e., the surrogate recoveries are outside the contract limits for both analyses), then submit the surrogate recovery data and sample analysis data from the initial analysis of both sample extracts (e.g., the first analysis of both extracts of the sample). Distinguish between the initial analysis and the analysis of the re-extracted sample on all data deliverables, using the sample suffixes supplied in Exhibit B.

TABLE 6
SURROGATE RECOVERY LIMITS

Compound	%Recovery Water	%Recovery Soil	
Nitrobenzene-d_5	35-114	23-120	
2-Fluorobiphenyl	43-116	30-115	
Terphenyl-d_{14}	33-141	18-137	
Phenol-d_5	10-110	24-113	
2-Fluorophenol	21-110	25-121	
2,4,6-Tribromophenol	10-123	19-122	
2-Chlorophenol-d_4	33-110	20-130	(advisory)
1,2-Dichlorobenzene-d_4	16-110	20-130	(advisory)

8.6. 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 Case of field samples received, OR
- Each 20 field samples in a Case, OR
- Each group of field samples of a similar concentration level (soils only), OR
- Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which field samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group).

Calculate the recovery of each matrix spike compound in the matrix spike and matrix spike duplicate and report on appropriate form.

8.6.1. Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds. Calculate the recovery of each matrix spike compound as follows:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike sample result
SR = Sample result
SA = Spike added

8.6.2 Calculate the relative percent difference of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \frac{|MSR - MSDR|}{(1/2)(MSR+MSDR)} \times 100$$

Where,

RPD = Relative Percent Difference

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.

8.6.3 The limits for matrix spike compound recovery and RPD are given in Table 7. 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.

TABLE 7
MATRIX SPIKE RECOVERY AND
RELATIVE PERCENT DIFFERENCE LIMITS

Compound	%Recovery Water	RPD Water	%Recovery Soil	RPD Soil
Phenol	12-110	42	26- 90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36- 97	28	28-104	27
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39- 98	28	38-107	23
4-Chloro-3-methylphenol	23- 97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10- 80	50	11-114	50
2,4-Dinitrotoluene	24- 96	38	28- 89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

8.7 Method blank analysis must be performed once for the following, on each GC/MS system used to analyze samples, whichever is most frequent:

- Each Case, OR
- Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which samples in a Case are received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
- Each 20 samples in a Case, including matrix spikes and reanalyses, that are of similar matrix (water or soil) or similar concentration (soil only), OR
- Whenever samples are extracted by the same procedure (continuous liquid-liquid extraction or sonication).

Determine the concentrations of any target compounds detected in the semivolatile method blank, using the equations in paragraph 8.2. The method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL) of the semivolatile target compounds in Exhibit C, except the phthalate esters, which must be less than or equal to five times (5x) the CRQL. For soil/sediment method blanks, CRQL value must be adjusted for percent moisture (see Exhibit B).

If a laboratory method blank exceeds these criteria, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be re-extracted and re-analyzed at no additional cost to the Agency. The Laboratory Manager, or his designee, must address problems and solutions in the SDG Narrative (Exhibit B).

9. GC/MS Confirmation of Pesticides and Aroclors

The requirements for GC/MS confirmation of pesticides and Aroclors are given in paragraph 17 of Exhibit D PEST. When performed, the characteristic ions to be used for these analytes are given in Table 8. Also see paragraph 3.2 of this section regarding the inclusion of these analytes in the semivolatile continuing calibration standard.

TABLE 8

CHARACTERISTIC IONS FOR PESTICIDES/AROCLORS

Parameter	Primary Ion	Secondary Ion(s)
alpha-BHC	183	181, 109
beta-BHC	181	183, 109
delta-BHC	183	181, 109
gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endrin ketone	317	67, 319
Endrin aldehyde	67	250, 345
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Endosulfan sulfate	272	387, 422
4,4'-DDT	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Aroclor-1016	222	260, 292
Aroclor-1221	190	222, 260
Aroclor-1232	190	222, 260
Aroclor-1242	222	256, 292
Aroclor-1248	292	362, 326
Aroclor-1254	292	362, 326
Aroclor-1260	360	362, 394

EXHIBIT D

ANALYTICAL METHODS
FOR PESTICIDES/AROCLORS

EXHIBIT D

Table of Contents

SECTION I

INTRODUCTION

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 (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. The method is based on EPA Method 608.

The method is divided into three sections: Introduction, Sample Preparation, and Analysis. Sample preparation covers sample extraction and cleanup techniques. The analysis section contains the specific GC/EC analytical methods for pesticides and Aroclors.

1. Summary of the Method

1.1 Continuous liquid-liquid or separatory funnel extraction procedures are employed for aqueous samples. Sonication extraction is required for soil/sediment samples (Section II, beginning at 6.2). The method specifies GPC, adsorption column cleanup, and sulfur cleanup techniques (Section II, beginning at 7).

1.2 The chlorinated pesticides and Aroclors listed in Exhibit C are determined by a two-column GC/EC technique.

1.3 Sample extracts, standards, and blanks must be analyzed within an analytical sequence as defined in Section III. GC/EC analysis begins with an initial demonstration of instrument performance and the calibration of all pesticides and Aroclors. Acceptable initial calibration is defined in Section III, beginning at 6. Initial calibration must be repeated whenever the calibration verification stipulated in Section III, 7, fails or when major instrument maintenance or modification is performed.

1.4 An instrument blank and a Performance Evaluation Mixture are analyzed no less than once in every 12 hour analytical sequence in order to monitor retention times, calibration factors, and column performance. Data can be collected only as long as the results for the Performance Evaluation Mixtures and instrument blanks fall within the limits defined in Section III, 7. If two consecutive unacceptable Performance Evaluation Mixtures are run, all extracts run since the previous acceptable Performance Evaluation Mixture must be reanalyzed. Additional Performance Evaluation Mixtures and blanks are recommended when highly contaminated samples are suspected.

1.5 Calibration and analysis sequence specifications of the GC/EC method apply independently to both GC columns.

1.6 Matrix spike and a matrix spike duplicate analyses must be prepared and analyzed at least once for each matrix type or once per Sample Delivery Group (SDG), whichever is most frequent.

1.7 Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, and matrix spike duplicates.

1.8 A single component pesticide is identified if a peak is detected within its appropriate retention time window on each of two columns. Toxaphene and Aroclors are identified primarily by pattern recognition, but RTs of three to five major peaks must also be taken into consideration. Guidance on quantitation of Aroclors is given in Section III, paragraph 13.

1.9 Standards for all tentatively identified Aroclors must be run within 72 hours of the sample analysis in which they were observed. These standards are used to verify identification only; quantitation is based on the standard analyzed during initial calibration.

1.10 Quantitative analysis of pesticides/Aroclors must be accomplished by the external standard method. Three-point calibration curves for single component analytes and the surrogates must be generated during the initial calibration. A linear response range must be demonstrated from the CRQL to a high point at least 16 times greater than the CRQL. Single-point calibrations for multicomponent analytes are sufficient for quantitation by this method.

1.11 The ECD response for single component analytes must be within the three-point calibration range in order for quantitative measurements to be made. The ECD response for the Aroclors/toxaphene must not be larger than the response for the high point calibration analysis of the single component analytes. The extracts must be diluted if the ECD response exceeds the calibration range. Quantitation must be performed and reported for both GC columns.

1.12 Absolute retention times (RTs) are used for the identification of pesticides/Aroclors. The absolute retention time window is calculated during initial calibration from the mean RT of the standard, using the retention time window specifications in Section III, paragraph 8.4.

1.13 The surrogates, 2,4,5,6-Tetrachloro-m-xylene and decachlorobiphenyl, must be added to all samples, blanks, matrix spikes, and matrix spike duplicates prior to extraction. The retention time of both surrogates must fall within the retention time windows for an analysis to be acceptable. The surrogate recoveries will be determined in all of these samples and will be reported to the Agency as a measure of method performance.

1.14 The criteria in Section III, paragraph 14, are used to determine whether an analysis is complete or whether additional cleanup, dilution, or reextraction is required.

1.15 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.

SECTION II

SAMPLE PREPARATION AND STORAGE

SECTION II

PART A - SAMPLE STORAGE AND HOLDING TIMES.

1. Procedures for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±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.

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

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

2. Procedure for Sample Extract Storage

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

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

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

3. Contract Required Holding Times

The extraction of 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.

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

PART B - SAMPLE PREPARATION FOR EXTRACTABLE PESTICIDES AND AROCLORS

1. Summary of Sample Preparation Methods

1.1 Water Samples

A 1-L volume of sample is spiked with the surrogate solution and is extracted with methylene chloride by using a separatory funnel or a continuous extractor. The methylene chloride extract is dried and concentrated (5.3). The extract is then 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), exchanged to hexane, cleaned up by Florisil cartridge, and adjusted to a final volume of 1.0 mL or 2.0 mL as described beginning at paragraph 7.2.

1.2 Soil/sediment Samples

A 30 g aliquot of sample is spiked with the surrogate solution and then mixed with sodium sulfate and extracted with a 1:1 acetone/methylene chloride solvent mixture by sonication. The extract is then filtered, dried, concentrated by K-D, and the solvent exchanged into methylene chloride (6.3). The extract is then cleaned up by GPC (mandatory), exchanged to hexane, cleaned up by Florisil cartridge, and adjusted to a final volume of 1.0 or 2.0 mL (7.2).

2. Interferences

2.1 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 blanks. 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.

2.2 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.

3. Apparatus and Materials

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

3.1 Continuous liquid-liquid extractors - with Teflon or glass connecting lines for use with methylene chloride, (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NH, P/N 6841-10 or equivalent).

3.2 Separatory funnel - 2000 mL with Teflon stopcock.

3.3 Apparatus for determining percent moisture

3.3.1 Oven - drying.

3.3.2 Desiccator.

3.3.3 Crucibles - porcelain (optional).

3.3.4 Aluminum weighing pans (optional).

3.4 Sonic cell disruptor - Heat Systems, Ultrasonics, Inc., Model W-385 (475 watt with pulsing capability, No. 207 3/4-inch tapped 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.

3.5 Sonabox (or equivalent) for use with disruptor to decrease noise level.

3.6 Beakers - 400-mL.

3.7 Kuderna-Danish (K-D) apparatus.

3.7.1 Concentrator tube - 10-mL, graduated (Kontes K-570040-1029, or equivalent).

3.7.2 Evaporative flask - 500-mL (Kontes K-470001-0500, or equivalent).

3.7.3 Snyder column - three-ball macro (Kontes K-503000-0121, or equivalent).

3.8 Funnels and Filter Paper.

3.8.1 Powder funnels - 10-cm diameter (optional), for filtration/drying.

3.8.2 Buchner funnels - 9-cm diameter, for filtration (optional).

3.8.3 Filter paper - No. 41 Whatman (or equivalent), 9-cm circles (optional).

3.9 Boiling chips.

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

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

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

3.11 Top loading balance - capable of weighing accurately to ± 0.01 g.

3.12 Balance - analytical, capable of weighing accurately to ± 0.0001 g. The balance must be calibrated with class S weights once per each 12-hour workshift. The balances must also be checked annually by a certified technician.

3.13 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).

3.14 Vials and caps - 2-mL for GC auto sampler.

3.15 Gel permeation chromatography (GPC) cleanup device. NOTE: GPC cleanup is required for all extracts for all soils and for water extracts containing higher molecular weight contaminants that interfere with the analyses of the target compounds (see paragraph 7.1.1).

Gel permeation chromatography system - GPC Autoprep Model 1002 A or B, Analytical Biochemical Laboratories, Inc., or equivalent. Systems that perform very satisfactorily also 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 7.1.3.

3.15.1 Chromatographic column - 700 mm x 25 mm i.d. 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 so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

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

3.15.3 Bio Beads (S-X3) - 200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent). An additional 5 gm 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 also can pass through the column screens and damage the valve.

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

3.15.5 Strip chart recorder, recording integrator or laboratory data system.

3.15.6 Syringe - 10-mL with Luerlok fitting.

3.15.7 Syringe filter assembly, disposable - Bio-Rad "Prep Disc" sample filter assembly #343-0005, 25 mm, and 5 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

3.15.8 A description of a manual system assembled from parts can be found in Wise, R.H., Bishop, D.F., Williams, R.T. & Austern, B.M. "Gel Permeation Chromatography in the GC/MS Analysis of Organics in Sludges" U.S. EPA, Municipal Environmental Research Laboratory, Cincinnati, Ohio, 45268.

3.15.9 Glass bottle - 1 liter volume, for use in preparation of Bio beads for packing into column.

3.16 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).

3.17 Vacuum system for eluting multiple cleanup cartridges.

3.17.1 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.

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

3.17.3 Vacuum pressure gauge.

3.17.4 Rack for holding 10-mL volumetric flasks in the manifold.

3.18 Pyrex glass wool - rinsed with methylene chloride and dried before use.

3.19 Bottle or test tube - 20-mL with Teflon-lined screw cap for sulfur removal.

3.20 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.)

3.21 Glass vials - minimum of 20-mL, with screw cap and Teflon or aluminum foil liner.

3.22 Spatula - stainless steel or Teflon.

3.23 pH Paper - wide range, (Hydrion Papers, Microessential Laboratory, Brooklyn, NY, or equivalent).

3.24 Pipet - Volumetric 1.00-mL or 2.00-mL (optional).

3.25 Syringe - 1.00-mL or 2.00-mL (optional).

3.26 Flask - Volumetric 10.00-mL.

3.27 Flask - Volumetric 1.00-mL or 2.00-mL (optional).

3.28 Vials - 10-mL, with screw cap and Teflon liner (optional).

3.29 Tube - centrifuge, 12- to 15-mL with 19-mm ground glass joint (optional).

3.30 Snyder Column - micro two or three ball with a 19-mm ground glass joint.

3.31 Centrifuge - table top (optional).

3.32 Vortex mixer - Genie, Model 550-6, Scientific Industrial, Inc., Bohemia, NY, or equivalent.

3.33 pH Meter with a combination glass electrode.

3.34 Magnetic stirrer motor - Model PC353, Corning Co., Corning, NY, or equivalent.

3.35 Magnetic stir bar - Teflon coated, at least 4 cm long.

3.36 Graduated cylinder - 1 L capacity.

4. Reagents

4.1 Sodium sulfate - granular-anhydrous reagent grade, heated at 400°C for 4 hours, or at 120°C for 16 hours, cooled in a desiccator, and stored in a glass bottle. Each lot must be extracted with hexane and analyzed by GC/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.

4.2 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.

4.3 Mercury - triple distilled, for sulfur clean-up.

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

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

4.6 Concentrated sulfuric acid - 18 N.

4.7 Reagent water - defined as a water in which no interferent 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.

4.8 Ten percent acetone in hexane (v/v) - prepare by adding 10.0 mL of acetone to 90.0 mL of hexane. NOTE: Prepare this mixture accurately or the results from the Florisil cartridge cleanup will be adversely affected. Water in the acetone also will adversely affect Florisil performance.

4.9 Standards

4.9.1 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.

4.9.2 Stock standard solutions (1.00 ug/uL) - can be prepared from pure standard materials or purchased as certified solutions.

4.9.2.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in toluene, dilute to volume in a 10-mL volumetric flask with toluene or acetone.

4.9.2.2 Transfer the stock standard solutions into a bottle/vial with Teflon-lined cap or septa. Store at 4°C (±2°C) and protect from light. Stock standard solutions must be replaced after six months or sooner, if comparison with check standards indicates a problem.

4.9.3 GPC calibration solution - prepare a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
corn oil	25
bis-2-ethylhexyl phthalate	1.0
methoxychlor	0.2
perylene	0.02
sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride, however, 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.

Store the calibration solution in an amber glass bottle with a Teflon lined screw-cap at 4°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.

4.9.4 Surrogate 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 ug/mL of each of the two compounds in acetone. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner, if comparison with quality control check samples indicates a problem. CAUTION: Analysts must allow all spiking solutions to equilibrate to room temperature before use.

4.9.5 Pesticide matrix spiking solution - prepare a spiking solution in acetone or methanol that contains the following pesticides in the concentrations specified:

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

The solution must be prepared every six months, or sooner if the solution has degraded or concentrated.

4.9.6 Florisil cartridge check solution.

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

4.9.7 Store all standard solutions in amber glass bottles or vials with a teflon-lined screw cap at 4°C (±2°C) and protect from light.

5. 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.

5.1 Separatory Funnel Extraction

5.1.1 Measure out each 1.0 L sample aliquot in a separate graduated cylinder. Measure and record the pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or conc. sulfuric acid, if required. Samples requiring pH adjustment must be noted in the SDG Narrative. Place the sample into a 2-L separatory funnel.

5.1.2 For each sample selected for matrix spike and matrix spike duplicate analyses, measure out two additional 1-L portions and transfer those portions into separate funnels. Adjust the pH of each, if required, and fortify each with 1.0 mL of matrix spike solution before continuing the extraction. The frequency of MS/MSD analysis is given in Section III, paragraph 16.

5.1.3 Using a syringe or a volumetric pipet, add 1.0 mL of the surrogate solution to all water samples, matrix spikes, and blanks.

5.1.4 Add 60 mL methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure. 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.

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

5.1.6 Prepare a method blank with each group of water samples extracted. For pesticide/Aroclor analyses, a method blank for water samples consists of a 1 L volume of reagent water (see paragraph 4.7), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in Section III, paragraph 15.

5.2 Continuous Liquid-Liquid Extraction

5.2.1 Add methylene chloride (100 to 250 mL) to the bottom of the extractor and fill it to a depth of at least one inch above the bottom sidearm.

5.2.2 Measure out each 1.0 L sample aliquot in a separate graduated cylinder. Measure and record the pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring pH adjustment must be noted in the SDG Narrative. Place the sample into the continuous extractor.

5.2.3 With some samples it may be necessary to place a layer of glass wool between the methylene chloride and the water layers in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

5.2.4 For each sample selected for matrix spike and matrix spike duplicate analyses, measure out two additional 1-L portions and transfer those portions into separate funnels. Adjust the pH of each, if required, and fortify each with 1.0 mL of matrix spike solution before continuing the extraction. The frequency of MS/MSD analysis is given in Section III, paragraph 16.

5.2.5 Using a syringe or a volumetric pipet, add 1.0 mL of the surrogate solution to all water samples, matrix spikes, and blanks.

5.2.6 Adjust the level of methylene chloride in the extractor so that the bottom sidearm is half filled with solvent.

5.2.7 Add sufficient methylene chloride to the distilling flask to ensure proper solvent cycling during operation and extract the solution for 18 hours. Allow to cool, then detach the distillation flask and label.

5.2.8 Prepare a method blank with each group of water samples extracted. For pesticide/Aroclor analyses, a method blank for water samples consists of a 1 L volume of reagent water (see paragraph 4.7), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in Section III, paragraph 15.

5.3 Extract Drying and Concentration

5.3.1 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.

5.3.2 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.

5.3.3 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60° - 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 3 to 5 mL, remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

5.3.4 If no GPC cleanup is required, proceed with the hexane exchange described in paragraph 7.2. If GPC cleanup is to be used, remove the Snyder column, rinse the flask and its lower joint and collect the rinsate in the concentrator tube, adjust the volume to 10.0 mL with methylene chloride. Proceed to 7.1.

6. Extraction of Soil/Sediment Samples

6.1 Sample Preparation

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

6.1.2 pH Determination - 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 glass electrode and the pH meter while the sample is

stirred. Report pH value on the appropriate data sheet. If the pH of the soil is > 9 or < 5, document any subsequent problems in 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.

NOTE: If insufficient volume of soil is received, use 5 g of soil and 5 mL of water for the pH determination and note in the SDG Narrative.

6.1.3 Percent Moisture - Weigh 5 to 10 g of the 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 1 below. Concentrations of individual analytes will be reported relative to the dry weight of sediment. CAUTION: Gases volatilized from some soil/sediment samples may require that this drying procedure be carried out in a hood.

$$\text{Percent Moisture} = \frac{\text{Wt of Sample - Wt of Dry Sample}}{\text{Wt of sample}} \times 100 \qquad \text{EQ. 1}$$

6.2 Extraction with Sonication

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

6.2.2 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).

6.2.3 For a sample to be used for matrix spike and matrix spike duplicate analysis, weigh out two additional 30 g (record weight to nearest 0.1 g) portions of sample and add 1.0 mL of the pesticide matrix spike solution to each soil aliquot. The frequency of MS/MSD analysis is given in Section III, paragraph 16.

6.2.4 Add 2.0 mL of surrogate solution to all soil samples, matrix spikes, and blanks by using a volumetric pipet or a syringe. Mix the solution 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 samples than to water samples because of the increased likelihood that the soil sample extracts will require dilution).

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

6.2.6 Place the bottom surface of the sonicator probe about 1/2 inch below the surface of the solvent but above the sediment layer.

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

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

6.2.8.1 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 evaporation (K-D) flask.

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

6.2.9 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 in paragraph 6.2.8. 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.

6.2.10 Prepare a method blank with each group of soil/sediment samples extracted. For pesticide/Aroclor analyses, a method blank for soil/sediment samples consists of 30 g of sodium sulfate (see paragraph 4.1), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in Section III, paragraph 15.

6.3 Soil Extract Concentration

6.3.1 Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 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. Reduce the volume of liquid to less than 10 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.

6.3.2 In order to remove most of the acetone, it is absolutely necessary to further reduce the volume of all soil/sediment extracts to 1.0 mL. This is best accomplished using the nitrogen evaporation technique (7.3.2). 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.

6.3.3 Adjust the extract volume to 10.0 mL with methylene chloride. Proceed to 7, below, for mandatory GPC and Florisil cartridge cleanup of soil extracts.

7. Extract Cleanup

There are three cleanup procedures specified in this method: GPC, Florisil cartridge, and sulfur cleanup. GPC must be performed for all soil 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.

7.1 Extract Cleanup by Gel Permeation Chromatography (GPC)

7.1.1. GPC cleanup is mandatory 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. Gel permeation chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of 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. A cross-linked divinyl benzenestyrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

GPC is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds. GPC is appropriate for both polar and non-polar analytes, therefore, it can be used effectively to clean up extracts containing a broad range of analytes.

Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue ultimately will reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol especially is susceptible to this problem. GPC system performance must be validated at least once every seven calendar days by demonstrating 80-110 percent recovery of the pesticide matrix spike mixture and examining the pattern of peaks from an Aroclor 1016/1260 mixture.

7.1.2 GPC Column Preparation

7.1.2.1 Weigh out 70 gm of Bio Beads (SX-3). Transfer them to a 1 liter 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 gm of Bio Beads in a 125 mL bottle or a beaker, using 25 mL of methylene chloride.

7.1.2.2 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).

7.1.2.3 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.

7.1.2.4 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.

7.1.2.5 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.

7.1.2.6 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 paragraph 7.1.2.5 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.

7.1.2.7 Push the plunger until it meets the gel, then compress the column bed about four centimeters.

7.1.2.8 Pack the optional 5 cm column with approximately 5 gm of preswelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

7.1.2.9 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 10/1000" ID x 2". Pump methylene chloride through the column at a rate of 5 mL/min for one hour.

7.1.2.10 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 paragraph 7.1.2.9) in the outlet tube from the UV

detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect 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.

7.1.2.11 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 7.1.4.

7.1.2.12 The GPC calibration procedure is based on monitoring the elution of standards with a UV detector connected to the GPC column. Care must be taken to account for any difference in volume (elution time) between the GC column and the detector and between the GPC column and the collection vial.

NOTE: The UV detector calibration procedure described in 7.1.3 is to be used 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.

7.1.3 Calibration of the GPC Column

7.1.3.1 Using a 10 mL syringe, load sample loop #1 with calibration solution (paragraph 4.9.3). With the ABC automated system, the 5 mL sample loop requires a minimum of 8 mL of the calibration solution. Use a firm, continuous pressure to push the sample onto the loop. Switch the valve so that GPC flow is through the UV flow-through cell.

7.1.3.2 Inject the calibration solution and obtain a UV trace showing a discrete peak for each component. Adjust the detector and/or recorder sensitivity to produce a UV trace that meets the following requirements. Differences between manufacturer's cell volumes and detector sensitivities may require a dilution of the calibration solution to achieve similar results. An analytical flow-through detector cell will require a much less concentrated solution than the semi-prep cell and, therefore, the analytical cell is not acceptable for use.

- o Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- o Corn oil and phthalate peaks must exhibit >85% resolution.
- o Phthalate and methoxychlor peaks must exhibit >85% resolution.
- o Methoxychlor and perylene peaks must exhibit >85% resolution.
- o Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

7.1.3.3 Determine the elution times for the phthalate, methoxychlor, and perylene. Phthalate will elute first, perylene, last.

7.1.3.4 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.

7.1.3.5 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.

7.1.3.6 Verify the flow rate by collecting column eluate for 10 minutes in a graduated cylinder and measure the volume, which should be 45-55 mL (4.5-5.5 mL/min). If the flow rate is outside of this range, corrective action must be taken to achieve this flow rate. Once the flow rate is within the range of 4.5-5.5 mL/min, record the column pressure (should be 6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared. A UV trace that does not

meet the criteria in paragraph 7.1.3.2 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.

7.1.3.7 Reinject the calibration solution after appropriate collect and dump cycles have been set, and the solvent flow and column pressure have been established.

7.1.3.7.1 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.

7.1.3.7.2 The retention times for bis(2-ethylhexyl)phthalate and perylene must not vary more than ±5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by the following:

- o Poor laboratory temperature control or system leaks.
- o An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight.
- o Excessive laboratory temperatures causing outgassing of the methylene chloride.

7.1.3.8 Analyze a GPC blank by loading 5 mL of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Exchange the solvent to hexane and analyze the concentrate by GC/EC. If the blank exceeds one half the CRQL of any analyte, assuming that the blank represents the extract from a one liter water sample, 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.

7.1.4 GPC Calibration Check

No Florisil cleanup is used in the GPC calibration check.

7.1.4.1 At least once every 7 days, the calibration of the GPC must be verified with two check mixtures. The first mixture is prepared by concentrating 2.0 mL of the matrix spiking solution (paragraph 4.9.5) to less than 1 mL under a stream of nitrogen (7.3.2), and adjusting the final volume to 10.0 mL with methylene chloride. The second mixture is prepared with 2 ug of Aroclor 1016 and 2 ug of Aroclor 1260 in a final volume of 10.0 mL methylene chloride.

7.1.4.2 Load the first 5.0 mL sample loop by using a 10 mL syringe containing 8 mL of the diluted pesticide matrix spike solution (paragraph 7.1.4.1). 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 by the UV detector calibration procedure (Paragraph 7.1.3).

7.1.4.3 The collected GPC calibration 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 (described in 7.3.2). After cooling, the solvent is exchanged to hexane according to the instruction in 7.2. The final volume is adjusted to 10.0 mL, and the sample is analyzed by GC according to the procedures in Section III. The analysis must be performed on at least one of the GC columns used for samples analysis.

7.1.4.4 The pattern of the Aroclor quantitation peaks and the recovery of each single component analyte must be determined for evaluation and reporting purposes. If the recovery of each of the single component analytes is 80 to 110 percent and if the Aroclor pattern is the same as with previously run standards, then the analyst may continue to use the column. If recoveries are out of the acceptance window or if changes in the relative peak heights of the patterns of the Aroclor are observed, the column must be replaced and recalibrated according to the instructions in 7.1.3.

7.1.4.5 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. In many cases, the SX-3 Bio Beads may be used for several months as long as the column calibration and flow rate remain constant.

7.1.5 Daily UV calibration check (optional)

The calibration of the GPC may be monitored daily by use of the UV-GPC calibration solution (paragraph 4.9.3) and the UV Detector Calibration Procedure (7.1.3). The UV detector should be used to monitor the elution times for the phthalate, methoxychlor, and perylene, in that order. The precalibrated 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.

7.1.6 Sample Extract Cleanup

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 outgassing of the methylene chloride is 22°C.

7.1.6.1 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 a 1:1 glycerol:water solution must be diluted and loaded into several loops. Similarly, extracts containing more than 500 mg of nonvolatile residue per 5 mL of extract must be diluted and loaded into several loops. The nonvolatile residue may be determined by evaporating a 100 uL aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.

7.1.6.2 Particles greater than 5 micron 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. INTRODUCTION OF PARTICULATES OR GLASS WOOL INTO THE GPC SWITCHING VALVES MAY REQUIRE FACTORY REPAIR OF THE APPARATUS.

7.1.6.3 Prior to loading samples, put the GPC into the "LOAD" mode, set the instrument terminal for the number of loops to be loaded, and set the "DUMP", "COLLECT", and "WASH" times for the values determined by the calibration procedure described in 7.1.3.

7.1.6.4 Attach the syringe to the turn lock on the injection port. Use firm, continuous pressure to push the sample onto the 5-mL sample loop. If the sample is difficult to load, some part of the system may be blocked. Take appropriate corrective action. If the back pressure is normal (6-10 psi) the blockage is probably in the valve. Blockage may be flushed out of the valve by reversing the inlet and outlet tubes and pumping solvent through the tubes (this should be done before sample loading).

NOTE: Approximately 2 mL of the extract remains in the lines between the injection port and the sample loop; excess sample also passes through the sample loop to waste.

7.1.6.5 After loading a loop, and before removing the syringe from the injection port, index the GPC to the next loop. This will prevent loss of sample caused by unequal pressure in the loops.

7.1.6.6 After loading each sample loop, wash the loading port with methylene chloride in a PTFE wash bottle to minimize cross contamination. Inject approximately 10 mL of methylene chloride to rinse the common tubes.

7.1.6.7 After loading all the sample loops, index the GPC to the OO position, switch to the "RUN" mode and start the automated sequence. Process each sample using the collect and dump cycle times established in 7.1.3.

7.1.6.8 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:

- o Change in solvent flow rate, caused by channeling in the column or changes in column pressure.
- o 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.
- o Leaks in the system or significant variances in room temperature.

7.1.6.9 After the appropriate GPC fraction has been collected for each sample, the solvent must be exchanged to hexane as described in 7.2.

7.1.6.10 Any samples that were loaded into two or more loops must be recombined before proceeding to 7.2.

7.2 Solvent exchange into hexane

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

7.2.1 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. 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.

7.2.2 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.

7.2.3 For samples which have <u>not</u> been subjected to GPC cleanup, adjust the volume of the hexane extract to 10.0 mL. For samples which <u>have</u> been subjected to GPC cleanup, concentrate the hexane extract to 5.0 mL using nitrogen evaporation, as described in 7.3.2. Proceed to 7.3 for Florisil cartridge cleanup.

7.3 Florisil Cartridge Procedure

Florisil cartridge cleanup is required for all extracts. Cleanup significantly reduces matrix interferences caused by polar compounds.

7.3.1 Cartridge Performance Check - every lot number of Florisil cartridges must be tested by the following procedure before they are used for sample cleanup. Add 0.5 mL of 2,4,5-trichlorophenol solution (0.1 ug/mL in acetone) and 0.5 mL of Standard Mixture A, midpoint concentration (Section III, paragraph 3.3) to 4 mL of hexane. Reduce the final volume to 0.5 mL using nitrogen (paragraph 7.3.2). Place the mixture onto the top of a washed Florisil cartridge (paragraph 7.3.4.4), and elute it with 9 mL of hexane/acetone [(90:10)(V/V)]. Use two additional 1-mL hexane rinses to ensure quantitative transfer of standard from the cartridge. Reduce the final volume to 1.0 mL using nitrogen (7.3.2) and analyze the solution by GC/EC using at least one of the GC columns specified for sample analyses. The recovery of each analyte must be determined for evaluation and reporting purposes. Calculate the percent recovery using the equation below. 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%, and if no peaks interfering with the target analytes are detected.

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} \times 100 \qquad \text{EQ. 1}$$

Where,

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample/blank

7.3.2 Nitrogen evaporation technique (taken from ASTM Method D 3086)

7.3.2.1 Place the concentrator tube with an open mini-Snyder column attached in a heating bath (30 to 35°C) and evaporate the solvent 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.

7.3.2.2 New 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.

7.3.3 Florisil cartridge cleanup

7.3.3.1 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.

7.3.3.2 Place one Florisil cartridge into the vacuum manifold for each sample extract.

7.3.3.3 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 1.0 mL of sample extract, then a 500-mg cartridge is used and the required final volume is 1.0 mL. If the autosampler requires more sample, prepare 2.0 mL of sample extract using a 1-g cartridge. Manual injection requires only a 1.0 mL final extract volume and a 500-mg cartridge.

7.3.3.4 Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge in the vacuum manifold, by pulling a vacuum, and by passing at least 5 mL of the hexane/acetone solution through the cartridge. While the cartridges are being washed, adjust the vacuum applied to each cartridge so that the flow rate through each cartridge is approximately equal. DO NOT ALLOW THE CARTRIDGES TO GO DRY AFTER THEY HAVE BEEN WASHED.

7.3.3.5 After the cartridges in the manifold are washed, the vacuum is released, and a rack containing labeled 10-mL volumetric flasks is placed inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside of the appropriate volumetric flask as the manifold top is replaced.

7.3.3.6 After the volumetric flasks are in place, vacuum to the manifold is restored, and a volume of extract equal to the required final volume (1.0 or 2.0 mL) from each sample, blank or matrix spike extract is transferred to the top frit of the appropriate Florisil cartridge.

7.3.3.7 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.

7.3.3.8 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.

7.3.3.9 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.

7.3.3.10 Concentrate the extract to 1.0 or 2.0 mL as required in paragraph 7.3.4.3 by using either nitrogen blowdown (7.3.2) or a micro-Snyder column. Measure the final volume with a syringe or by transferring the extract to a volumetric flask.

7.3.3.11 Sulfur contamination will cause a rise in the baseline of the chromatogram that may interfere with the analyses of the later eluting pesticides. If crystals of sulfur are evident or if the presence of sulfur is suspected, proceed to 7.4. Sample analyses showing the presence of sulfur are not acceptable and must be cleaned up and reanalyzed.

7.3.3.12 If sulfur is not present, transfer the sample to a GC vial and label the vial. The extract is ready for GC/EC analysis. Proceed to Section III. Store the extracts at 4°C (±2°C)in the dark.

7.4 Sulfur Removal

Sulfur can be removed by one of two methods, according to laboratory preference. Interference which is due to sulfur is not acceptable. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the 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.

7.4.1 If only part of a set of samples requires sulfur cleanup, then two method blanks are required for that set: one that is shaken with mercury or copper, and one that is not.

Sulfur cleanup blank - add 1.0 mL of surrogate to 10 mL of hexane in a clean centrifuge tube or 10-mL vial. Concentrate the solution to 2.0 mL by using either nitrogen evaporation or a micro Snyder column. The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. Measure the volume with a syringe or by transferring the solution to a volumetric flask. Proceed with the sulfur removal using the same technique (mercury or copper) as the samples associated with the blank.

7.4.2 Mercury technique

Add one to three drops of mercury to each hexane extract in a clean vial. Tighten the top on the vial and agitate the sample for 30 seconds. Filter or centrifuge the extract. Pipet the extract to another vial and leave all solid precipitate and liquid mercury. If the mercury appears shiny, proceed to Section III and analyze the extract. If the mercury turns black, repeat sulfur removal as necessary. CAUTION: Waste containing mercury should be segregated and disposed of properly.

NOTE: Mercury is a highly toxic metal and therefore must be used with great care. Prior to using mercury, it is recommended that the analyst become acquainted with proper handling and cleanup techniques associated with this metal.

7.4.3 Copper technique

Add approximately 2 g of cleaned copper powder to the extract in the centrifuge or concentrator tube. (2 g will fill the tube to about the 0.5 mL mark). Mix the copper and extract for at least 1 minute on a mechanical shaker. Separate the extract from the copper powder by drawing off the extract with a disposable pipet, and transfer the extract to a clean vial. The extract transferred to the vial still represents the 2.0 mL final volume. The separation of the extract from the copper powder is necessary to prevent degradation of the pesticides. If the copper appears bright, proceed to Section III and analyze the extract. If the copper changes color, repeat the sulfur removal procedure as necessary.

SECTION III

GC/EC ANALYSIS OF PESTICIDES AND AROCLORS

SECTION III

GC/EC ANALYSIS OF PESTICIDES AND AROCLORS

1. Summary of GC/EC Analysis

1.1 The analysis of samples is accomplished by using a wide-bore (0.53 mm ID) fused silica capillary column.

1.2 Sample extracts, standards, and blanks must be analyzed within an analytical sequence as defined in 5. GC/EC analysis begins with an initial demonstration of instrument performance and the calibration of all pesticides and Aroclors. Acceptable initial calibration is defined in paragraph 6. Initial calibration must be repeated whenever the calibration verification stipulated in paragraph 7 fails, or when major instrument maintenance or modification is performed.

1.3 An instrument blank, a Performance Evaluation Mixture, and a second instrument blank and the midpoint concentration of Individual Standard Mixtures A and B are analyzed no less than once in every 12 hour analytical sequence in order to monitor retention times, calibration factors, and column performance. Data can be collected only as long as the results for these standards and instrument blanks fall within the limits defined in paragraph 7. If two consecutive unacceptable standards are run, all extracts run since the previous acceptable standard must be reanalyzed. Additional standards and blanks are recommended when highly contaminated samples are suspected.

1.4 Calibration and run sequence specifications of the GC/EC method apply independently to both GC columns.

1.5 Matrix spike and a matrix spike duplicate analyses must be prepared and analyzed at least once for each matrix type or once per Sample Delivery Group (SDG), whichever is most frequent.

1.6 Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, and matrix spike duplicates.

1.7 A single component pesticide is identified if a peak is detected within its appropriate retention time window on each of two columns. Toxaphene and Aroclors are identified primarily by pattern recognition, but RTs of three to five major peaks must also be taken into consideration. Guidance on quantitation of Aroclors is given beginning at 13.9.

1.8 Standards for all tentatively identified Aroclors must be run within 72 hours of the sample analysis in which they were observed. These standards are used to verify identification only; quantitation is based on the standard analyzed during initial calibration.

1.9 Quantitative analysis of pesticides/Aroclors must be accomplished by the external standard method. Three-point calibration curves for single component analytes and the surrogates must be generated during

the initial calibration. A linear response range must be demonstrated from the CRQL to a high point at least 16 times greater than the CRQL. Single-point calibrations for multicomponent analytes are sufficient for quantitation by this method.

1.10 The ECD response for single component analytes must be within the three-point calibration range in order for quantitative measurements to be made. The ECD response for the Aroclors/toxaphene must not be larger than the response for the high point calibration analysis of the single component analytes. The extracts must be diluted if the ECD response exceeds the calibration range. Quantitation must be performed and reported for both GC columns.

1.11 Absolute retention times (RTs) are used for the identification of pesticides/Aroclors. The absolute retention time window is calculated during initial calibration from the RT of the standard, using the retention time window specifications in Paragraph 8.4.

2. Gas Chromatograph/Election Capture Detector (GC/EC)

2.1 Gas Chromatograph

2.1.1 The gas chromatograph (GC) system must adequately regulate temperature in order to give a reproducible temperature program and have a flow controller that maintains a constant column flow rate throughout temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases.

2.1.2 Gas chromatographs that are available from some manufacturers may have difficulty in meeting certain method QC requirements because of Endrin and DDT breakdown in the injector. This problem can be minimized by operating the injector at 200-205°C, using a Pyrex (not quartz) methyl silicone deactivated injector liner, and deactivating any metal parts in the injector with dichlorodimethyl silane. In some cases, using a 0.25 inch packed column injector converted for use with 0.53 mm capillary columns works better than a Grob-type injector. If a Grob-type injector is used, a 4 mm liner may be required to meet breakdown criteria.

2.2 Gas Chromatograph Columns

2.2.1 Two wide-bore (0.53 mm ID) fused silica GC columns are required. A separate detector is required for each column. The specified analytical columns are a DB-1701, 30 m x 0.53 mm ID, 1.0 um film thickness, (J&W Scientific, Folsom, CA, or equivalent), and a DB-608, 30 m x 0.53 mm ID, 0.5 to 1.0 um film thickness (J&W Scientific, or equivalent). Equivalent columns may be employed if they meet the requirements for resolution, initial calibration, and calibration verification listed in this section.

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

2.3 The carrier gas for routine applications is helium. Laboratories may choose to use hydrogen as a carrier gas, but they must clearly identify its use in the SDG Narrative and on all divider pages preceding raw chromatographic data in submissions to 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.

2.4 Electron Capture Detector - the make-up gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. The linearity of the response 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. 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.

2.5 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.

3. Calibration Standards

3.1 Resolution Check Mixture - prepare the mixture of pesticides in hexane or iso-octane at the concentrations listed below. The mixture must be prepared every six months, or sooner if the solution has degraded or concentrated.

gamma-Chlordane	10.0 ng/mL	Endrin ketone	20.0 ng/mL
Endosulfan I	10.0 ng/mL	Methoxychlor	100.0 ng/mL
p,p'-DDE	20.0 ng/mL	Tetrachloro-m-xylene	20.0 ng/mL
Dieldrin	20.0 ng/mL	Decachlorobiphenyl	20.0 ng/mL
Endosulfan sulfate	20.0 ng/mL		

3.2 Performance Evaluation Mixture (PEM) - prepare the PEM in hexane or iso-octane at the concentration levels listed below. The PEM must be prepared weekly, or more often if the solution has degraded or concentrated.

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

3.3 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 concentration is valid. The solution must be prepared every six months, or sooner if the solution has degraded or concentrated.

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
p,p'-DDD	10.0 ng/mL
p,p'-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 expoxide	5.0 ng/mL
alpha-Chlordane	5.0 ng/mL
gamma-Chlordane	5.0 ng/mL
p,p'-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

3.4 Multicomponent Standards - Toxaphene and Aroclor standards must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture. The calibration standards for the Aroclors must be prepared at concentrations of 100 ng/mL, except for Aroclor 1221 which must be prepared at 200 ng/mL. Toxaphene must be prepared at 500 ng/mL. All multicomponent standards must contain the surrogates at 20.0 ng/mL. The Aroclor and Toxaphene solutions must be prepared in hexane or iso-octane. Each solution must be prepared every six months, or sooner if the solution has degraded or evaporated.

4. Gas Chromatograph Operating Conditions

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

Carrier Gas:	Helium (Hydrogen may be used, see 2.3)
Column Flow:	5 mL/min
Make-up Gas:	P-5/P-10 or N_2 (required)
Injector Temperature:	$\geq$ 200°C (see 4.2)
Injection:	On-column
Injection Volume:	1 or 2 uL (see 4.1)
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	1/2 min
Temperature Ramp:	5°C to 6°C/min
Final Temperature:	275°C
Final Hold Time:	Until after Decachlorobiphenyl has eluted (approximately 10 minutes)

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

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.

4.1 Manual injections must be 2.0 uL. Auto injectors may use 1.0 uL volumes. The same injection volume must be used for all standards, blanks, and samples.

4.2 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.

5. Analysis Sequence for Standards and Samples

5.1 All acceptable samples must be analyzed within a valid analysis sequence as given below.

Time	Injection #	Material Injected
	1 - 15	First 15 steps of the Initial Calibration
0 hr.	16	Instrument Blank at end of Initial Calibration
	17	PEM at end of Initial Calibration
	18	First Sample
	o	
	o	Subsequent Samples
	o	
12 hr.	o	Last sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd injection	Performance Evaluation Mixture
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd and 3rd injections past 12 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
	etc.	

NOTE: The first 12 hours are counted from the injection #16 (the Instrument Blank at the end of the initial calibration sequence), not from injection #1. Samples may be injected until 12:00 hours have elapsed. All subsequent 12-hour periods are timed from the injection

of the instrument blank that bracketts the front end of the samples. Because the 12-hour time period is timed from injection of the instrument blank until the injection of the last sample, each 12-hour period may be separated by the length of one chromatographic run, that of the analysis of the last sample. While the 12-hour period may not be exceeded, the laboratory may run instrument blanks and standards more frequently, for instance to accomodate staff working on 8-hour shifts.

5.2 Before any samples are analyzed, it is necessary for the Contractor to complete an acceptable initial calibration sequence (see paragraph 6).

5.3 After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks, Performance Evaluation Mixtures, and Individual Standard Mixtures A and B are analyzed at the required frequency (see paragraph 7). 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 paragraph 7 in order to continue the run sequence.

5.4 An analysis sequence must also include all required matrix spike/matrix spike duplicate analyses and method blanks, but the Contractor may decide at what point in the sequence they are to be analyzed.

5.5 A standard of any identified Aroclor must be run within 72 hours of its detection in a sample chromatogram.

6. Initial Calibration

6.1 Initial Calibration Sequence

6.1.1 Before any samples are analyzed, it is necessary for the Contractor to complete the initial calibration sequence given below.

NOTE: Steps 16 and 17 are used as part of the calibration verification as well (see paragraph 7).

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
6. 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

6.1.2 Samples may be analyzed only after the initial calibration acceptance criteria (6.2) are met. Otherwise, the analytical system is not functioning adequately for use with this protocol.

6.1.3 The initial calibration may continue to be used as long as the analytical system remains under control. The proof that the analytical system is under control is provided by the analyses of the Performance Evaluation Mixtures. If those analyses do not meet the criteria described in paragraph 7, appropriate corrective action must be taken, and the initial calibration sequence must be repeated. The calibration sequence must also be repeated if any major change in instrument hardware or instrument parameters is made (e.g., if a new column is installed or if the detector temperature is changed).

6.2 Initial Calibration Acceptance Criteria (apply to each GC column independently)

6.2.1 The initial calibration sequence must be analyzed in the order listed in paragraph 6.1 using the optimized GC/EC operating conditions described in paragraph 4. The standards must be prepared according to paragraph 3. Calculate the calibration factors and retention times according to paragraphs 8-10.

6.2.2 The resolution criterion is that the depth of the valley between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0% of the height of the shorter peak. The poorest resolution on the DB-608 column probably will be between DDE and Dieldrin, between Methoxychlor and Endrin ketone and between Endosulfan I and gamma-Chlordane. On the DB-1701 column, resolution difficulties most frequently occur between Endosulfan I and gamma-Chlordane, and between Methoxychlor and Endosulfan sulfate.

6.2.3 The breakdown of DDT and Endrin in both of the Performance Evaluation Mixtures must be less than 20.0 percent, and the combined breakdown of DDT and Endrin must be less than 30.0 percent where,

EQ.2

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

EQ.3

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

EQ.4

Combined % Breakdown = %Breakdown DDT + %Breakdown Endrin

6.2.4 All peaks in both of the Performance Evaluation Mixtures must be 100 percent resolved on both columns.

6.2.5 The absolute retention times of each of the single component pesticides and surrogates in both of the PEMs must be within the retention time windows determined from the three-point initial calibration, in paragraph 8.4.

6.2.6 The relative percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in both of the PEMs must be less than or equivalent to 25.0 percent, using equation 5.

6.2.7 At least one chromatogram from each of the two Individual Standard Mixtures A and B, run during the initial calibration, must yield peaks that give recorder deflections of 50 to 100 percent of full scale.

6.2.8 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.

6.2.9 The % RSD of the calibration factors for each single component target compound must be less than or equal to 20.0 percent, except as noted below. 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, but those compounds must have a % RSD of less than or equal to 30.0 percent.

$$\%\text{RSD} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

6.3 Corrective Action

6.3.1 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.

6.3.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. In the case of light 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 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.

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

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

6.3.5 Initial calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed. Any samples or required blanks analyzed after the initial calibration criteria have not been met will require reanalysis at no additional cost to the Agency.

7. Calibration Verification

7.1 Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid point concentration of Individual Standard Mixtures A and B constitute the continuing

calibration. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

7.2 An instrument blank and the Performance Evaluation Mixture must bracket one end of a 12-hour period during which sample data are collected, and a second instrument blank and the the mid point concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period.

7.3 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 paragraph 5.1). 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).

7.4 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 paragraphs 7.8-7.14. 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 paragraphs 7.8-7.14. The 12-hour time period begins with the injection of the instrument blank. Standards (PEM or Individual Standard Mixtures), samples and required blanks may be injected for 12:00 hours from the time of injection of the instrument blank.

7.5 If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an acceptable instrument blank and PEM must be analyzed in order to start a new sequence. This requirement applies even if no analyses were performed since that standard(s) was injected.

7.6 After a break in sample analyses, the laboratory may only resume the analysis of samples using the current initial calibration for quantitation by analyzing an acceptable instrument blank and a PEM.

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

7.8 Analysts are cautioned that running an instrument blank and a performance evaluation mixture 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 performance evaluation mixtures more often to avoid discarding data.

7.9 The requirements for running the instrument blanks, Performance Evaluation Mixture, and Individual Standard Mixtures A and B are waived when no samples, method blanks, or matrix spikes are run during that 12-hour period. After a break in sample analysis, a laboratory may resume the analysis of samples, method blanks, and matrix spikes and may use the current initial calibration for quantitation only after an acceptable PEM is run (paragraphs 7.2 - 7.6). If a successful PEM cannot be run after an interruption, an acceptable initial calibration must be run before sample data may be collected. All acceptable sample analyses must be bracketed by acceptable performance evaluation mixtures and instrument blanks.

7.10 Technical Acceptance Criteria (apply to each GC column independently)

7.10.1 All single component pesticides and surrogates in the Performance Evaluation Mixtures used to demonstrate continuing calibration must be 100 percent resolved. The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

7.10.2 The absolute retention time for each of the single component pesticides and surrogates in the PEMs and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be within the retention time window determined from the three-point initial calibration in paragraph 8.4.

7.10.3 The relative percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEM and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be less than or equal to 25.0 percent, using Equation 5.

$$RPD = \frac{|C_{nom} - C_{calc}|}{C_{nom}} \times 100 \qquad \text{EQ. 5}$$

C_{nom} = true concentration of each analyte

C_{calc} = calculated concentration of each analyte from the analyses of the standard

Note: The vertical bars in the equation indicate the absolute value, hence RPD is always a positive number.

7.10.4 The percent breakdown of DDT and Endrin in the PEM must be less than or equal to 20.0 percent on both columns. The combined breakdown of DDT and Endrin must be less than or equal to 30.0 percent on both columns.

7.10.5 All instrument blanks must meet the acceptance criteria in paragraph 15.3.

7.11 Corrective Action

7.11.1 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.

7.11.2 Major corrective actions such as replacing the GC column or baking out the detector will require that a new initial calibration be performed and meets the technical acceptance criteria in 6.2.

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

7.11.4 If the analysis of the standard and instrument blank in 7.11.3 fail any of the technical acceptance criteria, a new initial calibration must be performed.

8. Determination of Absolute Retention Times.

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

8.2 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 Mixture A during the initial calibration and the mean RT is calculated as the average of the three values.

8.3 A retention time window is calculated for each single component analyte and surrogate by using the list in paragraph 8.4. Windows are centered around the mean absolute retention time for the analyte established during the initial calibrations.

8.4 Retention time windows for single and multicomponent analytes and surrogates.

Compound	Retention Time Window in Minutes
alpha-BHC	± 0.05
beta-BHC	± 0.05
gamma-BHC	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
Aldrin	± 0.05
alpha-Chlorodane	± 0.07
gamma-Chlorodane	± 0.07
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
DDD	± 0.07
DDE	± 0.07
DDT	± 0.07
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

8.5 For each multicomponent analyte, the RTs for three to five peaks are calculated from the initial calibration standard analysis. An RT window of ±0.07 minutes is used for all multicomponent analyte peaks.

8.6 Analytes are identified when peaks are observed in the RT window for the compound on both GC columns.

9. Calibration Factors for Single Component Pesticides.

9.1 During the initial calibration sequence, the Contractor must establish the magnitude of the linear ECD response range for each single component pesticide and surrogate on each column and for each GC system. This is accomplished by analyzing the Individual Standard Mixtures A and B at three concentrations during the initial calibration sequence in paragraph 6.

9.2 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.

9.2.1 Calculate the calibration factor for each single component pesticide and surrogate over the initial calibration range using Equation 6. The calibration factors for the surrogates are calculated from the three analyses of Individual Standard Mixture A only.

9.2.2 Calculate the mean and the %RSD of the calibration factors for each single component pesticide and surrogate over the initial calibration range using Equations 7 and 8.

$$CF = \frac{\text{Peak Area (or Height) of the Standard}}{\text{Mass Injected (ng)}} \qquad \text{EQ. 6}$$

$$\overline{CF} = \sum_{i=1}^{n} \frac{CF_i}{n} \qquad \text{EQ. 7}$$

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\overline{CF}} \times 100 \qquad \text{EQ. 8}$$

CF = Calibration factor

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

9.2.3 The linearity of the calibration is considered acceptable when the % RSD of the three point calibration is less than 20.0 percent except as noted in the following.

The % RSD of 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., but those compounds must have a % RSD of less than or equal to 30.0 percent.

9.2.4 If the linearity requirements listed above are met, the calibration factor from the mid point concentration standard is used for quantitation of each single component pesticide.

9.3 Sample analysis may not proceed until a satisfactory calibration has been demonstrated.

10. Calibration Factors for Toxaphene and Aroclors

10.1 Toxaphene and Aroclors require only a single-point calibration and they present special analytical difficulties. Because of the alteration of these materials in the environment, it is probable that samples which contain multicomponent analytes will give patterns similar to, but not identical with, those of the standards.

10.2 A set of three to five major peaks is selected for each multicomponent analyte. Retention times (see 8.4) and calibration factors are determined from the initial calibration analysis for each peak. Guidance for the choice of which peaks to use is given in paragraph 13.9

11. Acceptance Criteria for Chromatograms of Calibration Standards

The identification of single component pesticides by gas chromatographic methods is based primarily on rentention 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.

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

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

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

11.4 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.

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

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

12. Sample Analysis

12.1 Unless ambient temperature on-column injection is used (see paragraph 4.2), the injector must be heated to at least 200°C. The optimized gas chromatographic conditions from paragraph 4 must be used.

12.2 The injection must be made on-column by using either automatic or manual injection. If autoinjectors are used, 1.0 uL injection volumes may be used. Manual injections shall use at least 2.0 uL injection volumes. The same injection volume must be used for all standards, samples, and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2 uL. However, the same injection volume must be used for all analyses.

12.3 Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, and matrix spike duplicates.

12.4 The requirements for the analysis sequence apply to both GC columns and for all instruments used for these analyses.

12.5 The laboratory will identify and quantitate analyte peaks based on RT and calibration factor established during the initial calibration sequence, as long as an acceptable calibration verification (see paragraph 7) is performed every 12 hours.

12.6 The protocol is intended to achieve the quantitation limits shown in Exhibit C whenever possible. If sample chromatograms have interfering peaks, a high baseline, or off-scale peaks, then those samples must be reanalyzed following dilution, further cleanup, or reextraction. Samples which cannot be made to meet the given specifications after one reextraction and three-step cleanup (GPC, Florisil, and sulfur removal) are reported in the SDG Narrative and do not require further analysis. No limit is placed on the number of reextractions of samples that may be required because of contaminated method blanks.

12.7 The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined below). 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 extract must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

12.8 If the Contractor has reason to believe that diluting the final extracts will be necessary, an undiluted run may not be required. If an acceptable chromatogram (as defined below) is achieved with the diluted extract, an additional extract 10 times the concentration of the dilute sample must be injected and reported with the sample data.

12.9 No target analyte concentrations may exceed the upper limit of the initial calibration.

12.10 A standard for any identified multicomponent analyte must be analyzed during a valid analytical sequence on the same instrument, within 72 hours of its detection in a sample.

12.11 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.

12.11.1 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.

12.11.2 Chromatograms must display single component pesticides detected in the sample at less than full scale.

12.11.3 Chromatograms must display the largest peak of any multicomponent analyte detected in the sample at less than full scale.

12.11.4 If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale.

12.11.5 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.

12.11.6 For any sample, 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.

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

12.11.8 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.

13. Quantitation of Analytes

13.1 Quantitation of target analytes and surrogates must be performed and reported on both columns.

13.2 Analytes must be quantitated with an electronic integrator or with a laboratory data system. The analyst can use either peak height or peak area as the basis for quantitation. The use of an electronic integrator or a laboratory data system is required.

13.3 The chromatograms of all samples must be reviewed by a qualified pesticide analyst before they are reported.

13.4 In order to be quantitated, the detector response (peak area or peak height) of all of the single component analytes must lie between the response of the low and high concentrations in the initial calibration. If the analytes are detected below the CRQL, they are reported as present below the CRQL, and flagged according to the instructions in Exhibit B. If they are detected at a level greater than the high calibration point, the sample must be diluted either to a maximum of 1:100,000 or until the response is within the linear range established during calibration. Guidance in performing dilutions and exceptions to this requirement are given below.

13.4.1 If the response is still above the high calibration point after the dilution of 1:100,000, the Contractor shall contact the SMO immediately.

13.4.2 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.

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

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

13.4.5 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis and note the problem in the SDG Narrative.

13.5 The concentrations of the single component pesticides are calculated separately for both GC columns by using the following equations:

13.5.1 Water

$$\text{Concentration ug/L} = \frac{(A_x)(V_t)(Df)}{(CF)(V_o)(V_i)} \qquad \text{EQ. 9}$$

Where

A_x = Area of the peak for the compound to be measured

CF = Calibration factor for the mid point concentration external standard (area per ng)

V_o = Volume of water extracted in milliliters (mL)

V_i = Volume of extract injected in microliters (uL) (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto to each column.)

V_t = Volume of the concentrated extract in microliters (uL) (this volume must be 10000 uL, see Section II, 7.2.3)

Df = Dilution Factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

If GPC is performed on a water sample extract, V_t becomes 5000 uL, and a factor of 2 must be added to the numerator, as described below for soil/sediment samples.

13.5.2 Soil/Sediment

$$\text{Concentration (Dry weight basis) ug/Kg} = \frac{(A_x)(V_t)(Df)(2.0)}{(CF)(V_i)(W_s)(D)} \qquad \text{EQ. 10}$$

Where

A_x and CF are as given for water, above.

V_t = Volume of the concentrated extract in microliters (uL) (this volume must be 5000 uL, see Section II, 7.2.3)

V_i = Volume of extract injected in microliters (uL) (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto to 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 samples by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator 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 (see Section II, 7.2.3), maintains the sensitivity of the soil method comparable to that of the water method, but correction of the numerical result is still required.

13.5.3 Note that the calibration factors used for the quantitation of the single component pesticides are the calibration factors from the mid point concentration standard for each analyte.

13.5.4 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 11.

EQ. 11

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

Where,

$Conc_H$ - The higher of the two concentrations for the target compound in question

$Conc_L$ - The lower of the two concentrations for the target compound in question

Note that using this equation will result in percent difference values that are always positive. The value will also be greater than a value calculated using the higher concentration in the denominator, however, given the likelihood of a positive interference raising the concentration determined on one GC column, this is a conservative approach to comparing the two concentrations.

3.6 The concentrations of the surrogates are calculated separately for both GC columns in a similar manner as the other analytes, using Equations 9 and 10. Use the calibration factors from the midpoint concentration of Individual Standard Mixture A. The recoveries of the surrogates are calculated for both GC columns acccording to Equation 12.

$$\text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100 \qquad \text{EQ. 12}$$

Where,

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample/blank

The limits for the recovery of the surrogates are 60-150 percent for both surrogate compounds. As these limits are only advisory, no further action is required by the laboratory is required, 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).

13.7 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 10^6 times the CRQL, the Contractor shall contact the SMO immediately.

13.8 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 9 and 10, 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 three to five major peaks is determined on both columns. The following table lists the number of potential quantitation peaks for each Aroclor and Toxaphene.

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

13.9 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 X, and the two mean concentrations are compared using Equation 11.

13.10 The choice of the peaks used for multicomponent quantitation and the recognition of those peaks may be complicated by the environmental alteration of the Toxaphene or Aroclors, and by the presence of coeluting analytes or matrix interferences, or both.

13.11 If more than one multicomponent analyte is observed in a sample, the Contractor must choose separate peaks to quantitate the different multicomponent analytes. A peak common to both analytes present in the sample must not be used to quantitate either compound.

14. Sample Data Acceptance Criteria

14.1 The requirements below apply to both columns, and quantitation must be performed on both GC columns and reported.

14.2 All samples must be be analyzed as part of a valid analysis sequence (paragraph 5). They must be bracketed by acceptable instrument blanks (paragraph 15.3), acceptable Performance Evaluation Mixtures, and acceptable Individual Standard Mixtures A and B (paragraph 7) that were analyzed at the required frequency.

14.3 The retention times for both of the surrogates must be within the retention time windows as calculated in paragraph 8 for both GC columns.

14.4 Reportable data for a sample must include a chromatogram in which a baseline returns to below 50 percent of full scale before the elution time of alpha-BHC, and to below 25 percent of full scale after alpha-BHC and before decachlorobiphenyl.

14.5 If dilution has been applied and if no peaks are detected above 25 percent of full scale, analysis of a more concentrated sample is required.

14.6 Reportable sample data must include chromatogram(s) which meet the criteria in paragraph 12.11.

15. Blanks

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.

15.1 Method blank

15.1.1 Method blanks are spiked with the surrogate solution, extracted, cleaned up, and analyzed by following the same procedure that is used with the samples. A water method blank is one liter of reagent water treated as the water sample aliquot. A soil method blank is 30 g of sodium sulfate treated as the soil sample aliquot.

Method blank analysis must be performed once for the following, whichever is most frequent, and analyzed on each GC/EC system used to analyze samples:

- o Each Case, OR
- o Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which samples in a Case are received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
- o Each 20 samples in a Case, including matrix spikes and reanalyses, that are of similar matrix (water or soil), OR
- o Whenever samples are extracted by the same procedure (separatory funnel, continuous liquid-liquid extraction, or sonication).

15.1.2 In order to be acceptable, a method blank analysis cannot contain any of the analytes listed in Exhibit C at greater than the CRQL. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence mean retention time for both tetrachloro-m-xylene and decachlorobiphenyl.

15.1.3 All samples associated with an unacceptable method blank (see Form IV) must be reextracted and reanalyzed at no additional cost to the Agency.

15.2 Sulfur Cleanup Blank.

15.2.1 The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and passed through the sulfur cleanup procedure (see Section II, paragraph 7.4).

15.2.2 The sulfur cleanup blank is prepared when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur cleanup blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then the method blank must be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

15.2.3 In order to be acceptable, a sulfur blank analysis cannot contain any of the analytes listed in Exhibit C at greater than the CRQL, assuming that the material in the sulfur blank resulted from the extraction of a 1 L water sample. Calculate the concentration of each analyte using the equation in paragraph 13.5.1. Compare the results to the CRQL values for water samples in Exhibit C. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence mean retention time for both tetrachloro-m-xylene and decachlorobiphenyl.

15.2.4 All samples associated with an unacceptable sulfur blank (see Form IV) must be reextracted and reanalyzed at no additional cost to the Agency.

15.3 Instrument blank

15.3.1 An instrument blank is a hexane or iso-octane solution containing 20.0 ng/mL of tetrachloro-m-xylene and decachlorobiphenyl.

15.3.2. The first analysis in a 12-hour analysis sequence must be an instrument blank. All acceptable samples analyses are to be bracketed by acceptable instrument blanks, as described in paragraph 5.1.

15.3.3 An acceptable instrument blank must be analyzed within a 12-hour analysis sequence and must demonstrate that no analyte in Exhibit C is detected at greater than 0.5 times the CRQL and that the surrogate retention times are within the retention time windows. 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 paragraph 13.5.1. Compare the results to <u>one-half</u> the CRQL values for water samples in Exhibit C.

15.3.4 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 run after the last acceptable instrument blank must be reinjected during a valid run sequence at no additional cost to the Agency and must be reported.

15.3.5 Analysts are cautioned that running an instrument blank 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 more often to avoid

discarding data.

16. Matrix Spike/Matrix Spike Duplicate

16.1 A matrix spike and matrix spike duplicate must be extracted and analyzed at least once with every 20 samples of each matrix. NOTE: There is no differentiation between "low" and "medium" soil samples in this method. Therefore only one soil MS/MSD is to be submitted per SDG.

16.2 The surrogate retention times must be within the retention time windows specified.

16.3 The percent recoveries and the relative percent difference between the recoveries of each of the 6 compounds in the matrix spike samples will be calculated and reported by using the following equations:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100 \qquad \text{Eq. 12}$$

Where,

SSR = Spike sample result
SR = Sample result
SA = Spike added

$$RPD = \frac{|MSR - MSDR|}{1/2(MSR + MSDR)} \times 100 \qquad \text{Eq. 13}$$

Where,

RPD = Relative percent difference
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.

16.4 The Contractor shall report matrix spike and matrix spike duplicate recoveries and percent difference values with the analytical results (see Exhibit B). The limits for matrix spike compound recovery and RPD are given below. 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.

MATRIX SPIKE RECOVERY AND
RELATIVE PERCENT DIFFERENCE LIMITS

Compound	%Recovery Water	RPD Water	%Reco Soil	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

17. GC/MS Confirmation of Pesticides and Aroclors

17.1 Any pesticide or Aroclor analyte listed in Ex. 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 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.

17.1.1 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)
- A second analysis of the the semivolatile extract
- Analysis of the pesticide/Aroclor extract, following any solvent exchange and concentration steps that may be necessary.

17.1.2 The semivolatile GC/MS analysis procedures outlined in Ex. D SV are based on the injection into the instrument of approximately 20 ng of a target compound in a 2 uL volume. The semivolatile CRQL values in Ex. C are based on the sample concentration that corresponds to an extract concentration of 10 ng/uL 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/uL in extract will depend on the sample matrix.

- For water samples, 20 ng/ 2 uL corresponds to a sample concentration of 10 ug/L.
- For soil samples prepared according to the semivolatile low level soil method (i. e. 30 g of soil), the corresponding sample concentration is 330 ug/Kg.
- For soil samples prepared according to the

semivolatile medium level soil method (i. e. 1 g of soil), the corresponding sample concentration is 10,000 ug/Kg.

Therefore, based on the values given above, any pesticide sample in which the sample concentration is greater than or equal to an extract concentration of 10 ng/uL should enable the laboratory to confirm the pesticide/Aroclor by GC/MS analysis of the semivolatile extract.

7.1.3 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 no greater than 10 ng/uL.

17.1.3.1 To facilitate the confirmation of the pesticide/Aroclor 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/uL or less. 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 <u>not</u> 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.

17.1.3.2 The laboratory is advised that library search results from the NIST/EPA/MSDC 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.

17.1.4 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 paragraph 17.1.3, etc., or it must be analyzed along with separate reference standards for the analytes to be confirmed.

17.1.5 If the analyte cannot be confirmed by either the procedures in paragraphs 17.1.3 or 17.1.4, 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 paragraph 17.1.4, analysis of a reference standard is required if the GC/MS continuing calibration standard does not contain the analyte to be confirmed.

17.1.6 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.

17.2 If the identification of the analyte can not 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 result 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 Narrative.

17.3 For GC/MS confirmation of single component analytes, the required deliverables are copies of the library search results (three 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.

17.4 The purpose of GC/MS analysis is for confirmation of identification, not quantitation. Therefore, the concentrations of all pesticides/Aroclors shall be based on the GC/EC results. The exception noted in paragraph 17.2 applies only to analytes that cannot be confirmed at a concentration above that of the reference standard.

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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OVERVIEW

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, to ensure 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.

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.

The purpose of this Exhibit is to describe the overall quality assurance/quality control operations and the processes by which the Program meets the QA/QC objective 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 the 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.

SECTION I

INTRODUCTION

Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the quality control procedures and criteria incorporated into the methods. The methods in this contract have been validated on samples typical of those received by the laboratories in the Contract Laboratory Program (CLP). However the validation of these methods does not guarantee that they perform equally well for all sample matrices encountered. Inaccuracies can also result from causes other than unanticipated matrix effects, such as sampling artifacts, equipment malfunctions, and operator error. Therefore, the quality control component of each method is indispensable.

The data acquired from quality control 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, it gives an overview of the activities required in an integrated program to generate data of known and documented quality required to meet defined objectives.

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, NEIC, and EMSL/LV. Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Laboratory evaluation samples, magnetic tape audits, and data package audits provide an external QA reference for the program. A laboratory on-site evaluation system is also part of the external QA monitoring. A feedback loop provides the results of the various review functions to the contract laboratories through direct communications with the Technical Project Officers and Administrative Project Officers.

This Exhibit is not a guide to constructing quality assurance project plans, quality control systems, or a quality assurance organization. It is, however, an explanation of the quality control and quality assurance 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.

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:

- A written Quality Assurance Plan, the elements of which are designated in Section II.
- Written preparation of and adherence to QA/QC Standard Operating Procedures (SOPs) as described in Section III.
- Adherence to the analytical methods and associated QC requirements specified in the contract.
- Verification of analytical standard 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 tapes and applicable documentation for tape audits.
- Participation in On-Site Laboratory Evaluations, including adherence to corrective action procedures.
- Submission of all original documentation generated during sample analyses for Agency review.

SECTION II

QUALITY ASSURANCE PLAN

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.

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 useability.
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility .
- Detect problems through data assessment and establishes corrective action procedures which keep the analytical process reliable.
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

The QAP must 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, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during On-Site Laboratory evaluation. Additional information relevant to the preparation of a QAP can be found in EPA and ASTM publications.

Elements of a Quality Assurance Plan

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. Resumes
      b. Education and Experience Pertinent to This Contract
      c. Training Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives
2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy
2. Samples 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 SOPs.
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

Updating and Submission of the QAP:

Within 60 Days of contract award:

During the contract solicitation process, the Contractor was required to submit their QAP to EMSL/LV and NEIC. Within sixty (60) days after contract award, the Contractor shall send a revised QAP, fully compliant with the requirements of this contract, to the Technical Project Officer, EMSL/LV and NEIC. The revised QAP will become the official QAP under the contract. The revised QAP must include:

1) Changes resulting from A) The Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures and B) The Contractor's implementation of the requirements of the contract; and,

2) 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

Subsequent submissions:

During the term of contract, the Contractor shall amend the QAP when the following circumstances occur:

1) The Agency modifies the contract,

2) The Agency notifies the Contractor of deficiencies in the QAP document

3) The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,

4) The Contractor identifies deficiencies resulting from their internal review of their QAP document,

5) The Contractor's organization, personnel, facility, equipment, policy or procedures change,

6) The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy or procedures changes.

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 must be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended section pages must have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the current QAP document. The Contractor shall archive all amendments to the QAP document for future reference by the Agency.

The Contractor shall send a copy of the current QAP document within 14 days of a request by the Technical Project Officer or Administrative Project Officers to the designated recipients.

Corrective Action:

If a Contractor fails to adhere to the requirements listed in Section II, a 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, GC/MS tape audit, data package audit, an On-Site laboratory evaluation , remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION III

STANDARD OPERATING PROCEDURES

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document 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.

SOPs prepared by the Contractor must be functional: i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs, as presented to the Agency, must reflect activities as they are currently performed in the laboratory. In addition, all SOPs must be:

- Consistent with current EPA regulations, guidelines, and the CLP contract's requirements.
- Consistent with instruments manufacturers's specific instruction manuals.
- Available to the EPA during an On-Site Laboratory Evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During On-Site Laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs.
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Capable of demonstrating the validity of data reported by the Contractor and explain the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.
- Available at specific work stations as appropriate
- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

SOP 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 must 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
- References

SOPS REQUIRED:

The following SOPs are required by the Agency:

1. Evidentiary SOP

   Evidentiary SOPs for required chain-of-custody and document control are discussed in Exhibit F, "Specification for Written Standard Operating Procedures"

2. Sample Receipt and Storage
   a. Sample receipt and identification logbooks
   b. Refrigerator temperature logbooks
   c. Extract storage logbooks
   d. Security precautions
3. Sample preparation
   a. Reagent purity check procedures and documentation
   b. Extraction procedures
   c. Extraction bench sheets
   d. Extraction logbook maintenance
4. Glassware cleaning
5. Calibration (Balances, GPC)
   a. Procedures
   b. Frequency requirements
   c. Preventative maintenance schedule and procedures
   d. Acceptance criteria and corrective actions

e. Logbook maintenance authorization

6. Analytical procedures (for each analytical system)
   a. Instrument performance specifications
   b. Instrument operating procedures
   c. Data acquisition system operation
   d. Procedures when automatic quantitation algorithms are overridden
   e. QC required parameters
   f. Analytical run/injection logbooks
   g. Instrument error and editing flag descriptions and resulting corrective actions

7. Maintenance activities (for each analytical system)
   a. Preventative maintenance schedule and procedures
   b. Corrective maintenance determinants and procedures
   c. Maintenance authorization

8. Analytical standards
   a. Standard coding/identification and inventory system
   b. Standards preparation logbook(s)
   c. Standard preparation procedures
   d. Procedures for equivalency/traceability analyses and documentation
   e. Purity logbook (primary standards and solvents)
   f. Storage, replacement, and labelling requirements
   g. QC and corrective action measures

9. Data reduction procedures
   a. Data processing systems operation
   b. Outlier identification methods
   c. Identification of data requiring corrective action
   d. Procedures for format and/or forms for each operation

10. Documentation policy/procedures
   a. Laboratory/analyst's notebook policy, including review policy
   b. Complete SDG File contents
   c. Complete SDG File organization and assembly procedures, including review policy
   d. Document inventory procedures, including review policy

11. Data validation/self inspection procedures
   a. Data flow and chain-of-command for data review
   b. Procedures for measuring precision and accuracy

c. Evaluation parameters for identifying systematic errors

d. Procedures to assure that hardcopy and diskette deliverables are complete and compliant with the requirements in SOW Exhibits B and H.

e. Procedures to assure that hardcopy deliverables are in agreement with their comparable diskette deliverables.

f. Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal laboratory evaluation samples, etc.).

g. Frequency and type of internal audits (eg., random, quarterly, spot checks, perceived trouble areas).

h. Demonstration of problem identification-corrective actions and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).

i. Documentation of audit reports, (internal and external), response, corrective action, etc.

12. Data management and handling

a. Procedures for controlling and estimating data entry errors.

b. Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

c. Lifecycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

d. Database security, backup and archival procedures including recovery from system failures.

e. System maintenance procedures and response time.

f. Individuals(s) responsible for system operation, maintenance, data integrity and security.

g. Specifications for staff training procedures.

SOPS DELIVERY REQUIREMENTS:

Updating and submission of SOPs:

Within 60 days of contract award:

During the contract solicitation process, the Contractor was required to submit their SOPs to EMSL/LV and NEIC. Within sixty (60) days after contract award, the Contractor shall send a complete revised set of SOPs, fully compliant with the requirements of this contract, to the Technical Project Officer, EMSL/LV and NEIC. The revised SOPs will become the official SOPs under the contract. The revised SOPs must include:

1) Changes resulting from A) the Contractor's internal review of their procedures and B) the Contractor's implementation of the requirements of the contract;

2) 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.

Subsequent Submissions:

During the term of contract, the Contractor shall amend the SOPs when the following circumstances occur:

1) The Agency modifies the contract,

2) The Agency notifies the Contractor of deficiencies in their SOPs documentation

3) The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,

4) The Contractor's procedures change,

5) The Contractor identifies deficiencies resulting from the internal review of their SOPs documentation, or

6) The Contractor identifies deficiencies resulting form the internal review of their procedures.

The SOPs must be amended or new SOPs must 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 must 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 must have the date on which the changes were implemented.

When the SOPs are amended or new SOPs are written, the Contractor shall document in a letter the reasons for the changes, and submit the amended SOPs or new SOPs to the Technical Project Officer, EMSL/LV (quality assurance/technical SOPs) and NEIC (evidentiary SOPs). The Contractor shall send the letter and the amended sections of the SOPs or new SOPs within 14 days of the change. An alternate delivery schedule for the submittal of the letter and amended/new SOPs 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 TPO and/or APO.

The Contractor shall send a complete set of current SOPs within 14 days of a request by the Technical Project Officer or Administrative Project Officer to the recipients he/she designates.

Corrective action:

If a Contractor fails to adhere to the requirements listed in Exhibit E, Section III, a 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, GC/MS tape audit, data package audit, On-Site laboratory evaluation, remedial laboratory evaluation sample, and/or contract sanction, such as a Cure Notice.

SECTION IV VOA
VOLATILE QA/QC REQUIREMENTS

INTRODUCTION

Sections II and III of this exhibit outline the requirements for the quality assurance program that each laboratory must establish under this contract. The purpose of Section III is to outline the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of the volatile organic target compounds listed in Exhibit C using the procedures in Exhibit D VOA for water and soil/sediment samples. This section is not intended as a comprehensive quality control document, but rather as a guide to the specific QC operations that must be considered for volatile analyses. At a minimum, the laboratory is expected to address these operations in preparing the quality assurance plan and QA/QC Standard Operating Procedures discussed in Section II.

These operations include the following:

- o GC/MS Mass Calibration and Ion Abundance Patterns
- o GC/MS Initial and Continuing Calibration
- o Stability of Internal Standard Responses and Retention Times
- o Method Blank Analysis
- o System Monitoring Compound Recoveries
- o Matrix Spike and Matrix Spike Duplicate Analyses
- o Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates

Not discussed in this section are the requirements for quality assurance of the data reporting aspects of volatile analyses which are described in general terms in Section II and III of this exhibit.

1. GC/MS Mass Calibration and Ion Abundance Patterns

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC/MS system meets the instrument performance criteria specified in Exhibit D VOA, Section IV, paragraph 6. The purpose of this instrument performance check is to assure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of Bromofluorobenzene (BFB).

1.1 The required frequency of BFB analysis (once every 12 hours on each GC/MS system) is described in detail in Ex. D VOA, Section IV, paragraph 6.4.

1.2 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Table 1, Ex. D VOA, Section IV, paragraph 6.4.4.

1.3 The documentation includes Form V VOA, and a mass listing and bar graph spectrum of each BFB analysis.

2. GC/MS Initial Calibration for Target Compounds and System Monitoring Compounds

Prior to the analysis of samples and required blanks and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound and system monitoring compound standards.

2.1 The concentrations of the initial calibration standards for volatile target compounds and system monitoring compounds are 10, 20, 50, 100, and 200 ug/L, as described in Ex. D VOA, Section IV, paragraph 5.5.

2.2 The standards are to be analyzed according to the procedures given in Ex. D VOA, Section IV, paragraph 7, and at the frequency given in that paragraph.

2.3 The relative response factors (RRFs) are determined according to the procedures in Ex. D VOA, Section IV, paragraph 7.4, using the assignment of internal standard to target compounds and system monitoring compounds given in Ex. D VOA, Section IV, paragraph 7.4, and Table 5.

2.4 The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors of each target compound and system monitoring compound. The minimum RRF of each compound at each concentration level in the initial calibration and the percent relative standard deviation (%RSD) across all five points must meet the criteria given Ex. D VOA, Section IV, paragraphs 7.4.5 and 7.4.6, and Table 2. Allowance is made for any two volatile compounds that fail to meet these criteria. The minimum RRFs of those two compounds must be greater than or equal to 0.010, and the %RSD must be less than or equal to 40.0% for the initial calibration to be acceptable.

2.5 The documentation includes Form VI VOA, a GC/MS data system printout for the analysis of each volatile calibration standard.

3. GC/MS Continuing Calibration for Target Compounds and System Monitoring Compounds.

Once the GC/MS system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

3.1 The concentration of the continuing calibration standard for volatile target compounds and system monitoring compounds is 50 ug/L, as described in Ex. D VOA, Section IV, paragraph 5.5.3.

3.2 The standard is to be analyzed according to the procedures given in Ex. D VOA, Section IV, paragraph 7, and at the frequency given in that paragraph.

3.3 The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum RRF of each compound in the continuing calibration and the percent difference must meet the criteria given Ex. D VOA, Section IV, paragraphs 7.4.5, 7.4.6 and 7.4.7, and Table 2. Allowance is made for any two volatile compounds that fail to meet these criteria. The minimum RRFs of those two compounds must be greater than or equal to 0.010, and the percent difference must be less than or equal to 40.0% for the continuing calibration to be acceptable.

3.4 The documentation includes Form VII VOA, a GC/MS data system printout for the analysis of the volatile calibration standard.

4. Internal Standard Responses and Retention Times

The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results, because the quantitative determination of volatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

4.1 The specific compounds used as internal standards are given in Ex. D VOA, Section IV, paragraph 5.4.3. The concentration of each internal standard in the aliquot of the sample analyzed by GC/MS must be 50 ug/L at the time of purging.

4.2 The retention time and the extracted ion current profile (EICP) of each internal standard must be monitored for all analyses.

4.3 The area response of each internal standard from the EICP and the retention time of the internal standard are evaluated for stability, according to the procedures in Ex. D VOA, Section IV, paragraph 10.2. The area of the internal standard in a sample must not vary by more than a factor of 2 (i.e. -50% to +100%) from the area of the same internal standard in the associated continuing calibration standard. Likewise, the retention time of an internal standard must be within $\pm$ 0.50 minutes (30 seconds) of its retention time in the continuing calibration standard (see Ex.D VOA, Section IV, paragraph 10.2).

4.4 Requirements for reanalysis of samples when internal standards do not meet specifications are given in Ex. D VOA, Section IV, paragraph 10.2.

4.5 The documentation includes Form VIII VOA, and the GC/MS data system printout for the analysis of each sample, blank, matrix spike, matrix spike duplicate, and standard.

5. Method Blank Analysis

A method blank is a volume of a clean reference matrix (deionized distilled 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.

5.1 For volatile analysis, a method blank must be analyzed once every 12 hours on each GC/MS system, as described in detail in Ex. D VOA, Section IV, paragraphs 8.1.17, 8.2.1.9, and 8.2.2.10.

5.2 For the purposes of this protocol, an acceptable method blank must meet the criteria in paragraphs 5.2.1. and 5.2.2 below.

5.2.1 A method blank for volatile analysis must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Exhibit C) of Methylene chloride, Acetone, and 2-Butanone.

5.2.2 For all other target compounds, the method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL, see Exhibit C) of any single target compound.

5.3 If a method blank exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reanalysis of associated samples are given in Ex. D VOA, Section IV, paragraph 10.10.

5.4 The documentation includes Form I VOA for the blank analysis, Form IV VOA, associating the samples and the blank, and a GC/MS data system printout for the analysis of the method blank.

6. System Monitoring Compound Recoveries

The recoveries of the three system monitoring compounds are calculated from the analysis of each sample, blank, matrix spike and matrix spike duplicate. The purpose of the system monitoring compounds is to evaluate the performance of the entire purge and trap-gas chromatograph-mass spectrometer system. Poor purging efficiency, leaks, and cold spots in transfer lines are only a few of the potential causes of poor recovery of these compounds.

6.1 The system monitoring compounds are added to each sample, blank, matrix spike, and matrix spike duplicate prior to purging or extraction (medium soils only), at the concentration described in Ex. D VOA, Section IV, paragraph 5.4.4.

6.2 The recoveries of the system monitoring compounds are calculated according to the procedures in Ex. D VOA, Section IV, paragraph 10.8.1.

6.3 The recoveries must be within the quality control limits given in Ex. D VOA,. Section IV, Table 6. If the recovery of any one system monitoring compound is outside these limits, the Contractor must follow the steps outlined in Ex. D VOA, Section IV, paragraphs 10.8.2 to 10.8.6.

6.4 The documentation includes Form II VOA, and a GC/MS data system printout for the analysis of each sample, blank, matrix spike, and matrix spike duplicate.

7. Matrix Spike and Matrix Spike Duplicate Analysis

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.

7.1 The frequency of matrix spike and matrix spike duplicate (MS/MSD) analysis is described in Ex. D VOA, Section IV, paragraph 10.9.

7.2 The recoveries of the matrix spike compounds are calculated according to the procedures in Ex. D VOA, Section IV, paragraph 10.9.1. The relative percent difference between the results for each spiked analyte of the matrix spike and the matrix spike duplicate are calculated according to the procedures in Ex. D VOA, Section IV, paragraph 10.9.2.

7.3 The quality control limits for recovery and relative percent difference are given in Ex. D VOA, Section IV, Table 7. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

7.4 The documentation includes Form I VOA for both the MS and MSD analyses, Form III VOA, and a GC/MS printout for each analysis.

8. Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates

If the on-column concentration of any sample exceeds the initial calibration range, that sample must be diluted and reanalyzed, as described in Ex. D VOA, Section IV, paragraph 10.7. Guidance in performing dilutions and <u>exceptions</u> are given in that paragraph, and reiterated here.

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

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

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

8.4 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the SDG Narrative.

8.5. 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 either peak separately exceeds 200 ug/L.

SECTION IV SV
SEMIVOLATILE QA/QC REQUIREMENTS

INTRODUCTION

Sections II and III of this exhibit outline the requirements for the quality assurance program that each laboratory must establish under this contract. The purpose of Section III is to outline the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of the semivolatile organic target compounds listed in Exhibit C using the procedures in Exhibit D SV for water and soil/sediment samples. This section is not intended as a comprehensive quality control document, but rather as a guide to the specific QC operations that must be considered for semivolatile analyses. At a minimum, the laboratory is expected to address these operations in preparing the quality assurance plan and QA/QC Standard Operating Procedures discussed in Section II.

These operations include the following:

- o GC/MS Mass Calibration and Ion Abundance Patterns
- o GC/MS Initial and Continuing Calibration
- o Stability of Internal Standard Responses and Retention Times
- o Method Blank Analysis
- o Surrogate Recoveries
- o Matrix Spike and Matrix Spike Duplicate Analyses
- o Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates

Not discussed in this section are the requirements for quality assurance of the data reporting aspects of semivolatile analyses which are described in general terms in Section II and III of this exhibit.

1. GC/MS Mass Calibration and Ion Abundance Patterns

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC/MS system meets the instrument performance criteria specified in Exhibit D SV, Section IV, paragraph 4. The purpose of this instrument performance check is to assure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of Decafluorotriphenyl phosphine (DFTPP).

1.1 The required frequency of DFTPP analysis (once every 12 hours on each GC/MS system) is described in detail in Ex. D SV, Section IV, paragraph 4.3.6.

1.2 The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Table 1, Ex. D SV, Section IV, paragraph 4.3.3.

1.3 The documentation includes Form V SV, and a mass listing and bar graph spectrum of each DFTPP analysis.

2. GC/MS Initial Calibration for Target Compounds and Surrogates.

Prior to the analysis of samples and required blanks, and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound and surrogate standards.

2.1 The levels of the initial calibration standards for semivolatile target compounds and surrogates are 20, 50, 80, 120, and 160 ng, in a 2 uL injection volume, as described in Ex. D SV, Section IV, paragraph 3.2.

2.2 The standards are to be analyzed according to the procedures given in Ex. D SV, Section IV, paragraph 5, and at the frequency given in that paragraph.

2.3 The relative response factors (RRFs) are determined according to the procedures in Ex. D SV, Section IV, paragraph 5.4, using the assignment of internal standard to target compounds and surrogates given in Ex. D SV, Section IV, paragraph 5.4, and Tables 3 and 4.

2.4 The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors of each target compound and surrogate. The minimum RRF of each compound at each concentration level in the initial calibration and the percent relative standard deviation (%RSD) across all five points must meet the criteria given Ex. D SV, Section IV, paragraph 5.6, and Table 5. Allowance is made for any four semivolatile compounds that fail to meet these criteria. The minimum RRFs of those four compounds must be greater than or equal to 0.010, and the %RSD must be less than or equal to 40.0% for the initial calibration to be acceptable.

2.5 The documentation includes Form VI SV, a GC/MS data system printout for the analysis of each semivolatile calibration standard.

3. GC/MS Continuing Calibration for Target Compounds and Surrogates.

Once the GC/MS system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

3.1 The level of the continuing calibration standard for semivolatile target compounds and surrogates is 50 ng, in a 2 uL injection volume, as described in Ex. D SV, Section IV, paragraph 3.2.

3.2 The standard is to be analyzed according to the procedures given in Ex. D SV, Section IV, paragraph 5, and at the

frequency given in that paragraph.

3.3 The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum RRF of each compound in the continuing calibration and the percent difference must meet the criteria given Ex. D SV, Section IV, paragraphs 5.6, 5.7, and Table 5. Allowance is made for any four semivolatile compounds that fail to meet these criteria. The minimum RRFs of those four compounds must be greater than or equal to 0.010, and the %D must be less than or equal to 40.0% for the continuing calibration to be acceptable.

3.4 The documentation includes Form VII SV, a GC/MS data system printout for the analysis of the semivolatile calibration standard.

4. Internal Standard Responses and Retention Times

The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results because the quantitative determination of semivolatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

4.1 The specific compounds used as internal standards are given in Ex. D SV, Section IV, paragraph 3.1. The amount of each internal standard in the injection volume (2 uL) of the sample extract analyzed by GC/MS must be 40 ng (20 ng/uL).

4.2 The retention time and the extracted ion current profile (EICP) of each internal standard must be monitored for all analyses.

4.3 The area response of each internal standard from the EICP and the retention time of the internal standard are evaluated for stability, according to the procedures in Ex. D SV, Section IV, paragraph 8.1. The area of the internal standard in a sample must not vary by more than a factor of 2 (i.e. -50% to +100%) from the area of the same internal standard in the associated continuing calibration standard. Likewise, the retention time of an internal standard must be within $\pm$ 0.50 minutes (30 seconds) of its retention time in the continuing calibration standard (see Ex.D SV, Section IV, paragraph 8.1).

4.4 Requirements for reanalysis of samples when internal standards do not meet specifications are given in Ex. D SV, Section IV, paragraph 8.1.

4.5 The documentation includes Form VIII SV, and the GC/MS data system printout for the analysis of each sample, blank, matrix spike, matrix spike duplicate, and standard.

5. Method Blank Analysis

A method blank is a volume of a clean reference matrix (deionized distilled water for water samples, or a 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.

5.1 For semivolatile analysis, one method blank must be extracted with each group of samples of a similar matrix and concentration level (soils only), as described in Ex. D SV, Section IV, paragraph 8.7.

5.2 For the purposes of this protocol, an acceptable method blank must meet the criteria in paragraphs 5.2.1. and 5.2.2 below.

5.2.1 A method blank for semivolatile analysis must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Ex. C) of the phthalate esters listed in Ex. C.

5.2.2 For all other target compounds, the method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL, see Exhibit C) of any single target compound.

5.3 If a method blank exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reextraction and reanalysis of associated samples are given in Ex. D SV, Section IV, paragraph 8.7.

5.4 The documentation includes Form I SV for the blank analysis, Form IV SV, associating the samples and the blank, and a GC/MS data system printout for the analysis of the method blank.

6. Surrogate Recoveries

The recoveries of the eight surrogates are calculated from the analysis of each sample, blank, matrix spike and matrix spike duplicate. The purpose of the surrogates is to evaluate the preparation and analysis of samples.

6.1 The surrogates are added to each sample, blank, matrix spike, and matrix spike duplicate prior to extraction, at the concentrations described in Ex. D SV, Section II, Part B paragraph 4.6 and Part C paragraph 1.5.3 and 2.4.5.

6.2 The recoveries of the surrogates are calculated according to the procedures in Ex. D SV, Section IV, paragraph 8.5.1.

6.3 The recoveries must be within the quality control limits given in Ex. D SV, Section IV, Table 6. If the recovery of any surrogate is outside these limits, the Contractor must follow the steps outlined in E. D SV, Section IV, paragraphs 8.5.2 to 8.5.6 to determine whether or not reextraction and/or reanalysis is required.

6.4 The documentation includes Form II SV, and a GC/MS data system printout for the analysis of each sample, blank, matrix spike, and matrix spike duplicate.

7. Matrix Spike and Matrix Spike Duplicate Analysis

In order to evaluate the effects of the sample matrix on the methods used for semivolatile analyses, the Agency has prescribed a mixture of semivolatile target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

7.1 The frequency of matrix spike and matrix spike duplicate (MS/MSD) analysis is described in Ex. D SV, Section IV, paragraph 8.6.

7.2 The recoveries of the matrix spike compounds are calculated according to the procedures in Ex. D SV, Section IV, paragraph 8.6.1. The relative percent difference between the results for each spiked analyte of the matrix spike and the matrix spike duplicate is calculated according to the procedures in Ex. D SV, Section IV, paragraph 8.6.2.

7.3 The quality control limits for recovery and relative percent difference are given in Ex. D SV, Section IV, Table 7. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

7.4 The documentation includes Form I SV for both the MS and MSD analyses, Form III SV, and a GC/MS printout for each analysis.

8. Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates

If the on-column concentration of any sample exceeds the initial calibration range, that sample must be diluted and reanalyzed, as described in Ex. D SV, Section IV, paragraph 8.4. Guidance in performing dilutions and <u>exceptions</u> are given in that paragraph, and reiterated here.

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

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

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

8.4 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the SDG Narrative.

SECTION IV PEST
PESTICIDE/AROCLOR QA/QC REQUIREMENTS

INTRODUCTION

Sections II and III of this exhibit outline the requirements for the quality assurance program that each laboratory must establish under this contract. The purpose of Section III is to outline the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of the pesticide/Aroclor target compounds listed in Exhibit C using the procedures in Exhibit D PEST for water and soil/sediment samples. This section is not intended as a comprehensive quality control document, but rather as a guide to the specific QC operations that must be considered for pesticide/Aroclor analyses. At a minimum, the laboratory is expected to address these operations in preparing the quality assurance plan and QA/QC Standard Operating Procedures discussed in Section II.

These operations include the following:

- o GC Column Resolution
- o GC/EC Initial and Continuing Calibration
- o Determination of Retention Times and Retention Time Windows
- o Analytical Sequence
- o Blank Analyses
- o Surrogate Recoveries
- o Matrix Spike and Matrix Spike Duplicate Analyses
- o Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates

Not discussed in this section are the requirements for quality assurance of the data reporting aspects of pesticide/Aroclor analyses which are described in general terms in Section II and III of this exhibit.

1. GC Column Resolution

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC column meets the analyte resolution criteria specified in Exhibit D PEST, Section III, paragraph 6.2.2. The purpose of this resolution check is to demonstrate that at the time of the initial calibration, the GC column is capable of chromatographically resolving the target compounds. This is accomplished through the analysis of the Resolution Check Mixture (see Ex. D, Section III, paragraph 3.1), which contains the nine target compounds that are most difficult to resolve.

1.1 The Resolution Check Mixture must be analyzed at the beginning of every initial calibration sequence, on each GC column and instrument used for analysis.

1.2 Additional resolution criteria apply to the target compounds in the standards used for initial calibration and calibration verification, as described in Ex. D Section III, paragraphs 6.2.4, 6.2.10, and 7.10.

1.3 The documentation includes Form VI PEST-4, chromatograms and data system printouts for the analysis of the Resolution Check Mixture on each GC column and instrument used for analysis.

2. GC/EC Initial Calibration for Target Compounds and Surrogates.

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

2.1 The concentrations of the low point initial calibration standards for single component pesticide target compounds and surrogates are described in Ex. D PEST, Section III, paragraph 3.3. The concentration of the mid point initial calibration standards is specified in Ex. D, Section III, paragraph 3.3 as 4 times the low point concentration. The concentration of the high point initial calibration standard must be at least 16 times the low point concentration, and may be higher as described in Ex. D PEST, Section III, paragraph 3.3.

2.2 The standards are to be analyzed according to the procedures given in Ex. D PEST, Section III, using the GC operating conditions in paragraphs 4 and 6, and at the frequency given in paragraph 6.1.

2.3 The calibration factors are determined according to the procedures in Ex. D PEST, Section III, paragraphs 9 and 10.

2.4 The initial calibration of the GC/EC is evaluated on the basis of the stability of the calibrations factors and retention times of each target compound and surrogate, described in Ex. D PEST, Section III, paragraphs 6.2.5 to 6.2.9.

2.5 The calibration is also evaluated on the basis of the extent of breakdown of two target compounds, Endrin and 4,4'-DDT, as described in Ex. D PEST, Section III, paragraph 6.2.3.

2.6 The documentation includes Form VI PEST, chromatograms and data system printouts of all standards for the pesticide/Aroclor calibration standards.

3. GC/EC Continuing Calibration for Target compounds and Surrogates.

Once the GC/EC system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC column and instrument used for analysis. The calibration is verified through the analysis of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid point concentrations of Individual Standard Mixtures A and B.

3.1 The concentrations of the PEM and Individual Standard Mixtures used for continuing calibration are given in Ex. D PEST, Section III, paragraphs 3.2 and 3.3.

3.2 The instrument blank is described in Ex. D PEST, Section III, paragraph 15.3.

3.3 The instrument blank and the standards must be analyzed once every twelve hours according to the procedures in Ex. D PEST, Section III, paragraph 5, bracketing the sample analyses, as described in Ex. D PEST, Section III, paragraph 7.

3.4 The continuing calibration is evaluated on the basis of the stability of the retention times of the target compounds in the standards.

3.5 The continuing calibration is evaluated on the basis of the stability of the instrument response to the target compounds in the PEM, as judged by the reproducibility of the determinations of the concentrations of these compounds in the standard, as described in Ex. D PEST, Section III, paragraph 7.10.

3.6 The continuing calibration is evaluated on the basis of the extent of breakdown of two target compounds in the PEM, Endrin and 4,4'-DDT, as described in Ex. D PEST, Section III, paragraph 7.10.

3.7 The continuing calibration is evaluated on the basis of the levels of contamination that are found in the instrument blank, as described in Ex. D PEST, Section III paragraph 15.3.

3.8 The documentation includes Form VII PEST, Form VIII PEST, chromatograms and data system printouts for all standards and instrument blanks analyzed.

4. Determination of Retention Times and Retention Time Windows

The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The identification of multicomponent analytes is based primarily on recognition of patterns of retention times displayed on a chromatogram. Therefore, the determination of retention times and retention time windows is crucial to the provision of valid data for these target compounds.

4.1 The identification of all target compounds analyzed by the procedures described in Ex. D PEST is based on the use of absolute retention time. The mean retention time of each target compound, or each peak in a multicomponent target compound, is determined from the initial calibration standards, according to the procedures outlined in Ex. D PEST. Section III, paragraph 8.

4.2 The retention time window of each target compound peak is determined as described in Ex. D, Section III, paragraph 8.4.

4.3 The retention time shifts of the surrogates are used to evaluate the stability of the gas chromatographic system during analysis of samples and standards. The retention time of the surrogates must be within the retention time windows determined during the initial calibration (Ex. D PEST, Section III, paragraphs 7.10 and 8.4.

4.4 The documentation includes Form VI PEST, Form VII PEST, Form VIII PEST, chromatograms and data system printouts for all standards for the Pesticide/Aroclor initial and continuing calibrations, on each instrument and GC column used for analysis.

5. Analytical Sequence

The standards and samples analyzed according to the procedures in Ex. D PEST Section III must be analyzed in a sequence described in paragraphs 5 and 6. This sequence includes requirements that apply to the initial and continuing calibrations, as well as to the analysis of samples. The documentation includes Form VIII PEST.

6. Blank Analysis

Two types of blanks are required for analyses using the procedures in Ex. D PEST. They are method blanks and instrument blanks. A third type of blank, a sulfur clean up blank, may be required.

6.1 A method blank is a volume of a clean reference matrix (deionized distilled 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.

6.1.1 The frequency of method blank extraction is described in Ex. D PEST, Section III, paragraph 15.1.1.

6.1.2 The method blank must be analyzed on each GC column and instrument used for the analysis of associated samples.

6.1.3 For the purposes of this protocol, an acceptable method blank must meet the criteria in Ex. D PEST, Section, III, paragraph 15.1.2.

6.2 The 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.

6.2.1 The frequency of instrument blank analysis is part of the initial and continuing calibration requirements described in Ex. D, Section III, paragraphs 5,6, and 7.

6.2.2 For the purposes of this protocol, an acceptable instrument blank must meet the criteria in Ex. D PEST, Section, III, paragraph 15.3.3.

6.3 The sulfur clean up blank is a volume of clean solvent spiked with the surrogates and carried through the sulfur clean up and analysis steps. The purpose of the sulfur clean up blank is to determine the levels of contamination associated with the separate sulfur clean up steps.

6.3.1 The sulfur clean up blank is only required when all the samples associated with a particular method blank are <u>not</u> subjected to sulfur clean up, as described in Ex. D PEST, Section III, paragraph 15.2.2.

6.3.2 The sulfur clean up blank must be analyzed on all GC column and instruments used for analysis of samples that received sulfur clean up.

6.3.3 For the purposes of this protocol, an acceptable sulfur clean up blank must meet the criteria in Ex. D PEST, Section, III, paragraph 15.2.3.

6.4 If a method blank exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reextraction and reanalysis of associated samples are given in Ex. D PEST, Section III, paragraph 15.1.3.

6.5 If an instrument blank exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reanalysis of associated samples are given in Ex. D PEST, Section III, paragraph 15.3.4.

6.6 If a sulfur clean up blank exceeds the limits for contamination above, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for reextraction and reanalysis of associated samples are given in Ex. D PEST, Section III, paragraph 15.2.4.

6.7 The documentation includes Form I PEST for the analysis of each type of blank; Form IV PEST, associating the samples and the method and sulfur clean up blank; Form VIII PEST, associating the samples and the instrument blanks; and chromatograms and GC/EC data system printouts for the analysis of each blank.

7. Surrogate Recoveries

The recoveries of the two surrogates are calculated from the analysis on each GC column of each sample, blank, matrix spike and matrix spike duplicate. The purpose of the surrogates is to evaluate the preparation and analysis of samples.

7.1 The surrogates are added to each sample, blank, matrix spike, and matrix spike duplicate prior to extraction, at the concentrations described in Ex. D PEST, Section II, paragraph 4.9.4.

7.2 The recoveries of the surrogates are calculated according to the procedures in Ex. D PEST, Section III, paragraph 13.6.

7.3 The quality control limits for surrogate recovery, given in Ex. D PEST,. Section III, paragraph 13.6, are 60-150 percent. These limits are only advisory, and no further action by the laboratory is required if the limits are exceeded, however, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory, and any result in questions from the Agency.

7.4 The documentation includes Form II PEST, a chromatogram and a

GC/EC data system printout for the analysis of each sample, blank, matrix spike, and matrix spike duplicate.

8. Matrix Spike and Matrix Spike Duplicate Analysis

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.

8.1 The frequency of matrix spike and matrix spike duplicate (MS/MSD) analysis is described in Ex. D PEST, Section III, paragraph 16.1.

8.2 The recoveries of the matrix spike compounds are calculated according to the procedures in Ex. D PEST, Section III, paragraph 16.3. The relative percent difference for each spiked analyte between the results of the matrix spike and the matrix spike duplicate are calculated according to the procedures in Ex. D PEST, Section III, paragraph 16.3.

8.3 The quality control limits for recovery and relative percent difference are given in Ex. D PEST, Section III, paragraph 16.4. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

8.4 The documentation includes Form I PEST for both the MS and MSD analyses, Form III PEST, and chromatograms and a GC/EC data system printout for each analysis.

9. Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates

If the on-column concentration of any sample exceeds the initial calibration range, that sample must be diluted and reanalyzed, as described in Ex. D PEST, Section III, paragraph 13.4. Guidance in performing dilutions and exceptions are given in that paragraph, and reiterated here.

9.1 If the response is still above the high calibration point after the dilution of 1:100,000, the Contractor shall contact the SMO immediately.

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

9.3 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.

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

9.5 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis and note the problem in the SDG Narrative.

SECTION V

ANALYTICAL STANDARDS REQUIREMENTS

Overview

The U.S. Environmental Protection Agency will not supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories will be required to prepare from neat materials or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

A. Preparation of Chemical Standards from the Neat High Purity Bulk Material

A laboratory may prepare their chemical standards from neat materials. Commercial sources for neat chemical standards pertaining to compounds listed on the Compound Target List are given in the Appendix C of the "Quality Assurance Materials Bank: Analytical Reference Standards" Seventh Edition, January 1988. Laboratories should obtain the highest purity possible when purchasing neat chemical standards; standards purchased at less than 97% purity must be documented as to why a higher purity could not be obtained.

1. Neat chemical standards must be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of neat chemicals is essential in order to safeguard them from decomposition.

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 contract laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use either differential scanning calorimetry, gas chromatography with flame ionization detection, high performance liquid chromatography, infrared spectrometry, or other appropriate techniques. Use of two or more independant methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

Equation 1

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

where "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

3. Mis-identification of compounds occasionally occurs and it is

possible that a mislabeled compound may be received from a chemical supply house. It is the contract laboratory's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards be correctly identified. Identification confirmation, when performed, should use , gas chromatographic/mass spectrometry analysis on at least two different analytical columns, or other appropriate techniques.

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

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

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

B. Purchase of chemical standards already in solution

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

1. Laboratories must maintain the following documentation to verify the integrity of the standard solutions they purchase:

   a. mass spectral identification confirmation of the neat material

   b. purity confirmation of the neat material

   c. chromatographic and quantitative documentation that the solution standard was QC checked according to the following section

2. The Contractor must purchase standards for which the quality is demonstrated statistically and analytically by a method of the supplier's choice. One way this can be demonstrated is to prepare and analyze three solutions; a high standard, a low standard, and a standard at the target concentration (see parts a and b below). The supplier must then demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the Student's t-test in part "d". If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test in part e. Thus the standard is certified to be within 10 percent of the target concentration.

   If the procedure above is used, the supplier must document that the following have been achieved:

   a. Two solutions of identical concentration must be prepared independently from neat materials. An aliquot of the first solution must be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration ten percent greater than the target standard. This is called the "high standard". One further aliquot is taken from the second solution and diluted to a concentration 10 percent less that the target standard. This is called the "low standard".

   b. Six replicate analyses of each standard (a total of 18 analyses) must be performed in the following sequence: low standard, target, high standard, low standard, target standard, high standard, ...

   c. The mean and variance of the six results for each solution must be calculated.

   Equation 2

   $$\text{MEAN} = (Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6)/6$$

   Equation 3

   $$\text{VARIANCE} = (Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2 + Y_5^2 + Y_6^2 - (6*\text{MEAN})^2)/5$$

   The values Y_1, Y_2, Y_3, ..., represent the results of the six analyses of each standard. The means of the low, target, and high standards are designated M_1, M_2, and M_3, respectively. The variances of the low, target, and high standards are designated V_1, V_2, and V_3, respectively. Additionally, a pooled variance, V_p, is calculated.

   Equation 4

   $$V_p = (V_1/(0.81) + V_2 + V_3/(1.21))/3$$

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

d. The test statistic must be calculated:

Equation 5
$$\text{TEST STATISTIC} = |(M_3 /1.1) - (M_1 /0.9)|/(V_p /3)^{0.5}$$

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

e. The test statistic must be calculated:

Equation 6
$$\text{TEST STATISTIC} = |M_2 - (M_1 /1.8) - (M_3 /2.2)|/(V_p /4)^{0.5}$$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

f. The 95 percent confidence intervals for the mean result of each standard must be calculated:

Equation 7
$$\text{Interval for Low Standard} = M_1 \pm (2.13)(V_p /6)^{0.5}$$

Equation 8
$$\text{Interval for Target Standard} = M_2 \pm (2.13)(V_p /6)^{0.5}$$

Equation 9
$$\text{Interval for High Standard} = M_3 \pm (2.13)(V_p /6)^{0.5}$$

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

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

C. 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.

D. Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of each laboratory to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra,

etc, whether produced by the laboratory or purchased from chemical supply houses, must be maintained by the laboratory and may be subject to review during On-Site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the laboratories for a period of one year.

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.

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, Administrative Project Officer, and EMSL-LV, 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 TPO and/or APO.

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 Exhibit E, Section III.

If the Contractor fails to adhere to the requirements listed in Section V, a 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, GC/MS tape audit, data package audit, an On-Site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION VI

CONTRACT COMPLIANCE SCREENING

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.

CCS is performed by the Sample Management Office (SMO) under the direction of the EPA. To assure a uniform review, a set of standardized procedures have been developed to evaluate the sample data package submitted by a Contractor against the technical and completeness requirements of the contract.

CCS results are mailed to the Contractor and all other data recipients. The Contractor has a period of time to correct deficiencies. The Contractor must send all corrections to the Regional Client, EMSL/LV, and SMO.

CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

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 alterante 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 disprove 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 will not grant an extension for greater than 14 days for the Contractor's response to the CCS trend report.

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 Exhibit E, Section III.

If the Contractor fails to adhere to the requirements listed in Section VI, 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, GC/MS tape audit, data package audit, an On-Site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION VII

REGIONAL DATA REVIEW

Contract laboratory data are generated to meet the specific needs of the Regions. In order to verify the useability of data for the intended purpose, each Region reviews data from the perspective of end-user, based upon functional aspects of data quality. General guidelines for data review have been developed jointly by the Region and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problems under investigation and the Regional response appropriate to the specific circumstances.

Regional data reviews, relating useability of the data to a specific site, are part of the collective assessment process. They complement the review done at the Sample Management Office, which is designed to identify contractual discrepancies and the review done at EMSL/LV which is designed to evaluate Contractor and method performance. These individual evaluations are integrated into a collective review that is necessary for program and laboratory administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

SECTION VIII

LABORATORY EVALUATION SAMPLES

Although intralaboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in interlaboratory comparison studies conducted by the EPA. Results from the analysis of these laboratory evaluation samples, also referred to as performance evaluation (PE) samples, will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

Sample sets may be provided to participating Contractors as frequently as on an SDG-by-SDG basis as a recognizable QC sample of known composition; as a recognizable QC sample of unknown composition; or not recognizable as a QC material. The laboratory evaluation samples may be sent either by the Regional client or the National Program Office, and may be used for contract action.

Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

At a minimum, the results are evaluated for compound identification, quantification, and sample contamination. Confidence intervals for the quantification of target compounds are based on reported values using population statistics. EPA may adjust the scores on any given laboraotry evaluation sample to compensate for unanticipated difficulties with a particular sample. Normally, a fraction of the compounds spiked into the sample are not specifically listed in the contract. Contractors are required to use the NIST/EPA/MSDC mass spectral library to tentatively identify a maximum number of non-target compounds in each fraction that are present above a minimal response. Tentative identification of these compounds, based on contractually described spectral interpretation procedures, is evaluated and integrated into the evaluation process.

A Contractor's results on the laboratory evaluation samples will determine the Contractor's performance as follows:

1. Acceptable, No Response Required (Score greater than or equal to 90 percent):

   Data meets most or all of the scoring criteria. No response is required.

2. Acceptable, Response Explaining Deficiency(ies) Required (Score greater than or equal to 75 percent but less than 90 percent):

   Deficiencies exist in the Contractor's performance.

Within 14 days of receipt of notification from EPA, the Contractor shall describe the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a letter to the Administrative Project Officer, the Technical Project Officer and EMSL/LV.

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 of 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 laboratory evaluation sample report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

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 Exhibit E, Section III.

3. Unacceptable Performance, Response Explaining Deficiency(ies) 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.

Within 14 days of receipt of notification from EPA, the Contractor shall describe the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a letter to the Administrative Project Officer, the Technical Project Officer and EMSL/LV.

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 of 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 laboratory evaluation sample report.

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 Exhibit E, Section III.

The Contractor shall be notified by the Technical Project Officer or Administrative Project Officer concerning the remedy for their unacceptable performance. A Contractor may expect, but the Agency is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, an On-Site laboratory evaluation, GC/MS tape audit, data package audit, remedial laboratory evaluation sample, and/or a contract sanction, such as a Cure Notice.

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

If the Contractor fails to adhere to the requirements listed in Section VIII, a 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, GC/MS tape audit, data package audit, a remedial laboratory evaluation sample and/or contract sanctions, such as a Cure Notice.

SECTION IX

GC/MS TAPE AUDITS

Periodically, EPA requests from Contractors the GC/MS magnetic tapes corresponding to a specific case in order to accomplish tape audits. Generally, tape submissions and audits are requested for the following reasons:

- Program overview
- Indication of data quality problems from EMSL/LV, SMO, or Regional data reviews
- Support for On-Site audits
- Specific Regional requests

Depending upon the reason for an audit, the tapes from a recent case, a specific case, or a laboratory evaluation sample may be requested. Tape audits provide a mechanism to assess adherence to contractual requirements and to ensure the consistency of data reported on the hardcopy/floppy diskettes with that generated on the GC/MS tapes. This function provides external monitoring of Program QC requirements and checks adherence of the Contractor to internal QA procedures. In addition, tape audits enable EPA to evaluate the utility, precision, and accuracy of the analytical methods.

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

The Contractor is required to retain the GC/MS tapes for 365 days after data submission.

When submitting GC/MS tapes to the Agency, the following materials must be delivered in response to the request:

1. All associated raw data files for samples, blanks, matrix spikes, matrix spike duplicates, initial and continuing calibration standards, and instrument performance check solutions (BFB and DFTPP).

2. All processed data files and quantitation output files associated with the raw data files described above.

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

4. All laboratory-generated mass spectral library files (NIST/EPA/MSDC library not required).

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 must include EPA Sample Numbers and Lab File identifiers for all samples, blanks, and standards, identified by Case and SDG.

The laboratory must also provide a statement attesting to the completeness of the GC/MS data tape submission, signed and dated by the Laboratory Manager. This statement must be part of a cover sheet that includes the following information relevant to the data tape submission:

1. Laboratory name

2. Date of submission

3. Case Number

4. SDG Number

5. GC/MS make and model number

6. Software version

7. Disk drive type (e.g. CDC, PRIAM, etc.)

8. File transfer method (e.g. DSD, DTD, FTP, Aquarius, etc.)

9. Names and telephone numbers of two laboratory contacts for further information regarding the submission.

Submission of the GC/MS tape:

Upon request of the Administrative Project Officer or EMSL/LV, the Contractor shall send the required GC/MS tapes and all necessary documentation to EMSL/LV within seven 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 tape. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

Responding to the GC/MS tape audit report:

After completion of the GC/MS tape audit, the Agency may send a copy of the GC/MS tape audit report to the Contractor or may discuss the GC/MS tape audit report on an On-Site laboratory evaluation. In a detailed letter to the Technical Project Officer, Administrative Project Officer, and EMSL/LV, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the GC/MS tape audit report within 14 days of receipt of the report. 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 tape report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

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 Exhibit E, Section III.

Corrective actions

If the Contractor fails to adhere to the requirements listed in Section IX, 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, GC/MS tape audit, data package audit, remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION X

DATA PACKAGE AUDITS

Data package audits are performed by the Agency for program overview and specific Regional concerns. Standardized procedures have been established to assure uniformity of the auditing process. Data packages are periodically selected from recently received cases. They are evaluated for the technical quality of hardcopy raw data, quality assurance, and the adherence to contractual requirements. This function provides external monitoring of program QC requirements.

Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance. It provides the Agency with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. A thorough review of the raw data is completed including: a check of instrument printouts, quantitations 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 laboratory personnel involved with the Case, and a review of all current SOPs on file.

Responding to the data package audit report:

After completion of 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 on an On-Site laboratory evaluation. In a detailed letter to the Technical Project Officer, Administrative Project Officer, and EMSL/LV, 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. 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 TPO and/or APO.

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 Exhibit E, Section III.

Corrective Actions

If the Contractor fails to adhere to the requirements listed in Section X, 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, GC/MS tape audit, data package audit, remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION XI

ON-SITE LABORATORY EVALUATIONS

At a frequency dictated by a contract laboratory's performance, the Administrative Project Officer, Technical Project Officer or their authorized representative will conduct an On-Site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: Quality Assurance Evaluation, and an Evidentiary Audit.

A. 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. The Contractor should expect that items to be monitored will include, but not be limited to the following items.
- Size and appearance of the facility
- Quantity, age, availability, scheduled maintenance and performance of instrumentation
- Availability, appropriateness, and utilization of the QAP and SOPs
- Staff qualifications, experience, and personnel training programs
- Reagents, standards, and sample storage facilities
- Standard preparation logbooks and raw data
- Bench sheets and analytical logbook maintenance and review
- Review of the Contractor's sample analysis/data package inspection/data management procedures

Prior to an On-Site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous On-Site reports, laboratory evaluation sample scores, Regional review of data, Regional QA materials, GC/MS tape audit reports, data audit reports, results of CCS, and date trend reports.

B. Evidentiary Audit

Evidence auditors conduct an On-Site laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. The evidence audit is comprised of the following three activities:

1. Procedural Audit

The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis) and analytical project file organization and assembly.

2. Written SOPs Audit

The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis) and analytical project file organization and assembly.

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:

- o The accuracy of the document inventory
- o The completeness of the file
- o The adequacy and accuracy of the document numbering system
- o Traceability of sample activity
- o Identification of activity recorded on the documents
- o Error correction methods

C. Discussion of the On-Site Team's Findings

The quality assurance and evidentiary auditors discuss their findings with the Administrative Project Officer/Technical Project Officer prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

D. Corrective Action Reports For Follow-Through to Quality Assurance and Evidentiary Audit Reports

On-site laboratory evaluation:

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, Administrative Project Officer, and EMSL/LV (response to quality assurance/technical report) and NEIC (response to the evidentiary

report), within 14 days of receipt of the report. 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 TPO and/or APO.

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 Exhibit E, Section III.

Corrective actions

If the Contractor fails to adhere to the requirements listed in Section XI, 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, GC/MS tape audit, data package audit, a remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION XII

QUALITY ASSURANCE AND DATA TREND ANALYSIS

Data submitted by laboratories are subject to review from several aspects: compliance with contract-required QC, useability, and full data package evaluation. Problems resulting from any of these reviews may determine the need for a GC/MS tape audit, an On-Site laboratory evaluation and/or a remedial laboratory evaluation sample. In addition, QC prescribed in the methods provides information that is continually used by the Agency to assess sample data quality, Contractor data quality and Program data quality via data trend analysis. Trend analysis is accomplished by entering data into a computerized data base. Statistical reports that evaluate specific anomalies or disclose trends in many areas, including the following, are generated from this data base:

- Surrogate Spike Recovery
- Laboratory Evaluation Sample
- Blanks
- GC/MS Instrument Performance Checks (BFB and DFTPP)
- Initial and Continuing Calibration Data
- Other QC and Method Parameters

Program-wide statistical results are used to rank laboratories in order to observe the relative performance of each Contractor using a given protocol against its peers. The reports are also used to identify trends within laboratories. The results of many of these trends analyses are included in overall evaluation of a Contractor's performance, and are reviewed to determine if corrective action or an On-Site laboratory evaluation is indicated in order to meet the QA/QC requirements of the contract.

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.

As a further benefit to the Program, the data base provides the information needed to establish performance-based criteria in updated analytical protocols, where advisory criteria has been previously used. The vast empirical data set produced by contract laboratories is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of quality control and performance criteria specifications of what is routinely achievable and expected of environmental chemistry laboratories in mass production analysis of environmental samples. This, in turn, assists the Agency in meeting its objectives of obtaining data of known and documented quality.

SECTION XIII

DATA MANAGEMENT

Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage and security of computer readable data and files. These procedures should be in written form and contain a clear definition for all databases and files used to generate or resubmit deliverables. Key areas of concern include: system organization (including personnel and security), documentation operations, traceability and quality control.

Data manually entered from hard-copy must be quality controlled and the error rates estimated. Systems should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors. In addition, data entry error rates must be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

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

- Justification or rationale for the change.
- Initials of the person making the change or changes. Data changes must be implemented and reviewed by a person or group independant of the source generating the deliverable.
- Change documentation must be retained according to the schedule of the original deliverable.
- Resubmitted diskettes or other deliverables must be reinspected as a part of the laboratories' internal inspection process prior to resubmission. The entire deliverable, not just the changes, must be inspected.
- The Laboratory Manager must approve changes to originally submitted deliverables.
- Documentation of data changes may be requested by laboratory auditors.

Lifecycle management procedures must be applied to computer software systems developed by the laboratory to be used to generate and edit contract deliverables. Such systems must be thoroughly tested and documented prior to utilization.

- A software test and acceptance plan including test requirements, test results and acceptance criteria must be developed, followed, and available in written form.
- System changes must not be made directly to production systems generating deliverables. Changes must be made first to a development system and tested prior to implementation.

- Each version of the production system will be given an identification number, date of installation, date of last operation and archived.
- System and operations documentation must be developed and maintained for each system. Documentation must include a users manual and an operations and maintenance manual.

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

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating and quality control.
- Data and system security, backup and archiving.

EXHIBIT F

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

1. SAMPLE CHAIN-OF-CUSTODY

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 accomplish this, Contractors are required to develop and implement the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures.

1.1 Sample Identification

To assure traceability of the samples while in possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

Each sample and sample preparation container shall be labeled with the EPA number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA number.

1.2 Chain-of-Custody Procedures

Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented. A sample is under custody if:

- It is in your possession, or
- It is in your view after being in your possession, or
- It was in your possession and you locked it up, or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

1.3 Sample Receiving Procedures

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available.

1.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

- o Airbills or airbill stickers
- o Custody seals
- o EPA custody records
- o EPA traffic reports or SAS packing lists
- o Sample tags

1.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

1.3.7 The Contractor shall contact the Sample Management Office (SMO) to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

1.3.8 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

1.3.9 The following information shall be recorded on Form DC-1 (See Exhibit B) by the sample custodian or his/her representative as samples are received and inspected:

- o Condition of the shipping container
- o Presence or absence and condition of custody seals on shipping and/or sample containers
- o Custody seal numbers, when present
- o Condition of the sample bottles
- o Presence or absence of airbills or airbill stickers
- o Airbill or airbill sticker numbers
- o Presence or absence of EPA custody records
- o Presence or absence of EPA traffic reports or SAS packing lists
- o Presence or absence of sample tags
- o Sample tag identification numbers cross-referenced to the EPA sample numbers
- o Verification of agreement or non-agreement of information recorded on shipping documents and sample containers
- o Problems or discrepancies

1.4 Sample Tracking Procedures

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis.

2. DOCUMENT CONTROL PROCEDURES

The goal of the laboratory document control program is to assure that all documents for a specified Sample Delivery Group (SDG) will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to EPA or are available upon request from EPA prior to the delivery schedule.

2.1 Preprinted Laboratory Forms and Logbooks

2.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete sample delivery group file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG is compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents which are directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments.

Because the laboratory must provide copies of the instrument run logs to EPA, the laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

2.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable.

All notations shall be recorded in ink.

Unused portions of documents shall be "z'd" out.

2.2 Consistency of Documentation

The Contractor shall assign a document control officer responsible for the organization and assembly of the CSF.

All copies of laboratory documents shall be complete and legible.

Original documents which include information relating to more than one SDG shall be filed in the CSF of the lowest SDG number. The copy(s) shall be placed in the other CSF(s) and the Contractor shall record the following information on the copy(s) in red ink:

"COPY

ORIGINAL IS FILED IN CSF _____"

The Contractor shall sign and date this addition to the copy(s).

Before releasing analytical results, the document control officer shall assemble and cross-check the information on samples tags, custody records, lab bench sheets, personal and instrument logs, and other relevant deliverables to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the CSF.

2.3 Document Numbering and Inventory Procedure

In order to provide document accountability of the completed analysis records, each item in the CSF shall be inventoried and assigned a serialized number as described in Exhibit B).

All documents relevant to each sample delivery group, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc. shall be inventoried.

The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the appropriate EPA region or other receiver as designated by EPA. The DCO shall place the sample tags in plastic bags in the file.

2.4 Storage of EPA Files

The Contractor shall maintain EPA laboratory documents in a secure location.

2.5 Shipment of Deliverables

The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used.

3. SPECIFICATIONS FOR WRITTEN STANDARD OPERATING PROCEDURES

The Contractor shall have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, sample tracking, and assembly of completed data. An SOP is defined as a written narrative stepwise description of laboratory operating procedures including examples of laboratory documents. The SOPs shall accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits.

3.1 The Contractor shall have written SOPs describing the sample custodian's duties and responsibilities.

3.2 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.2.1 Presence or absence of EPA chain-of-custody forms

3.2.2 Presence or absence of airbills or airbill stickers

3.2.3 Presence or absence of traffic reports or SAS packing lists

3.2.4 Presence or absence of custody seals on shipping and/or sample containers and their condition

3.2.5 Custody seal numbers, when present

3.2.6 Airbill or airbill sticker numbers

3.2.7 Presence or absence of sample tags

3.2.8 Sample tag ID numbers

3.2.9 Condition of the shipping container

3.2.10 Condition of the sample bottles

3.2.11 Verification of agreement or non-agreement of information on receiving documents and sample containers

3.2.12 Resolution of problems or discrepancies with the SMO

3.2.13 An explanation of any terms used by the laboratory to describe sample condition upon receipt (e.g., good, fine, OK)

3.3 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and shall include a description of the document used to cross-reference the unique laboratory identifier to the EPA sample number.

If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

3.4 The Contractor shall have written SOPs describing all storage areas for samples in the laboratory. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.5 The Contractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.

3.6 The Contractor shall have written SOPs describing the method by which the laboratory maintains the security of any areas identified as secure.

3.7 The Contractor shall have written SOPs for tracking the work performed on any particular samples. The tracking SOP shall include:

- A description of the documents used to record sample receipt, sample storage, sample transfers, sample preparations, and

sample analyses.

- o A description of the documents used to record calibration and QA/QC laboratory work.
- o Examples of document formats and laboratory documents used in the sample receipt, sample storage, sample transfer, and sample analyses.
- o A narrative step-wise description of how documents are used to track samples.

3.8 The Contractor shall have written SOPs for organization and assembly of all documents relating to each SDG. Documents shall be filed on a sample delivery group-specific basis. The procedures shall ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the SDG are compiled in one location for submission to EPA. The written SOPs shall include:

- o A description of the numbering and inventory method.
- o A description of the method used by the laboratory to verify consistency and completeness of the CSF.
- o Procedures for the shipment of deliverables packages using custody seals.

4. HANDLING OF CONFIDENTIAL INFORMATION

A Contractor conducting work under this contract may receive EPA-designated confidential information from the agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

4.1 All confidential documents shall be under the supervision of a designated document control officer (DCO).

4.2 Confidential Information

Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO will log these documents into a Confidential Inventory Log. The information will then be available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Technical and Administrative Project Officer. The DCO will enter all copies into the document control system described above. In addition, this information may not be disposed of except upon approval by the EPA project officer. The DCO shall remove and retain the cover page of any confidential

information disposed of for one year and shall keep a record on the disposition in the Confidential Inventory Log.

EXHIBIT G

GLOSSARY OF TERMS

GLOSSARY OF TERMS

ALIQUOT - a measured portion of a sample taken for 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 - see Method Blank

4-BROMOFLUOROBENZENE (BFB) - compound chosen to establish mass spectral instrument performance for volatile analyses.

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, not on the basis of information entered on the Traffic Report by the sampler.

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the calibration of the GC/MS 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.

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.

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.

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOAs), 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.

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 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 (previously termed reagent blank) - an analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through 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 below).

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.

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.

REAGENT WATER - water in which an interferent 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 above).

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where

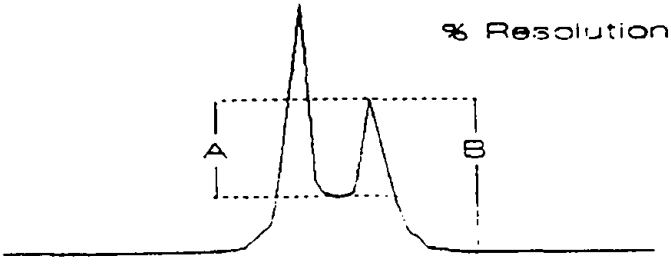
A = area of the characteristic ion measured

C = concentration

is = internal standard

x = analyte of interest

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, multipled by 100.



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 14 calendar days (7 calendar days for 14-day data turnaround contracts). Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 field samples within a Case; or
- Each 14-day calendar period (7-day calendar period for 14-day data turnaround contracts) during which field samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.

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

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.

STANDARD ANALYSIS - an analytical determination made with known quantities of target compounds; used to determine response factors.

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 labelled 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. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

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

TRAFFIC REPORT (TR) - an EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which 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 0.32 mm. Columns with lesser diameters are classified as narrow bore capillaries.

EXHIBIT H

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

AGENCY STANDARD IMPLEMENTATION
FOR ORGANICS OLM01.0
(This document will replace existing formats as of October 1991)

1. 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 with the delimiter as place holder.

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 must not be entered into any numeric field.

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, decimal, 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 EPA Rounding Rules) to fit into the field. The rounding must maintain the greatest significance possible providing the field length limitation. In addition, the rounded number that appears on the form, and therefore the field in 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. Field lengths should only be as long as necessary to contain the data; packing with blanks is not allowed.

2. 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 must contain "carriage return" and "line feed", respectively.

2.2 This implementation consists of eleven 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, semivolatiles, and each column analysis for pesticides (minimum of four Type 10 series for VOA/SV/PEST SDG). The 20 series records are used to link samples within an SDG to the corresponding calibrations, blanks, and so on for screening purposes. 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. Production Runs

A production run represents a "group" or "batch" of samples that are processed in a continuous sequence under relatively stable conditions. Specifically:

Calibration - All samples in a run use the same initial calibration data.

Method number - Constant.

Instrument conditions - Constant throughout a run. Results obtained on different instruments cannot be combined in one run.

Analyses from each fraction consist of separate production runs, and are reported in separate files. There will be a separate production run for each 72-hour sequence for pesticides for each GC column utilized. Thus, a full three fraction analysis will consist of a minimum of four production runs, and could consist of more.

EXAMPLE OF THE SEQUENCE OF RECORD TYPES IN A FILE

```
10        Contains Run Header information
     11        Contains additional run-wide information if required.
     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 TICs if necessary.
               36        Reports any instrumental data necessary.
          30        Values for the next analyte or parameter being measured.
                         Additional data may vary for each parameter, and records
               32        may occur in any order.  Multiple occurrences of the
               33        same record type, however, must be consecutive.
               36
          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 next
     21        sample or other group of data.
     22
          30
               32
               33
               36
          30
               32
               33
               36
                         etc.
     20
     21
          30
               32
               33
               36
                         etc.
```

4. Record Sequence

4.1 The sequence of records for Agency Standard files is as follows: A Run Header (type 10) record must 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, 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 or standard in the sample. The type 20 record holds a count for the number of method analytes being determined, and includes all target compounds, surrogates, 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 must occur in the order of sample analysis. In addition, a type 20 record is used as a header for any additional run-wide data that must be reported for each method analyte (such as mean response factors). Unique identifiers given in Section 10 are used in place of "QC codes" to indicate the type of data that follows. Type 30 records for each analyte must occur in the order specified on hardcopy deliverable Form 6.

4.3 Type 90 comment records may be defined to occupy any position except before the type 10 (header) record.

5. 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 within file	00000-99999, numbered sequentially
	4	Record checksum	Four hexadecimal digits(*)
	2	Must contain CR and LF	

(*) The checksum is the sum of the ASCII representation of the data on the record up to the Record Sequence Number plus the checksum of the previous record. The sum is taken modulo 65536 (2^{16}) and represented as four hexadecimal digits.

6. 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 YY MM DD, and times as HH MM. All hours must be given as 00 to 23 using a 24 hour-clock and must be local time.

7. 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 may not be split between files.

8. Deliverable

8.1 The file must be submitted on 5-1/4 inch floppy diskette(s), which may be either double-sided, double density, 360 K-byte or high capacity 1.2 M-byte diskette(s). IBM-compatible, 3.5 inch double-sided, double density 720 K-byte or high density 1.44 M-byte diskettes may also be submitted. The diskettes must be formatted and recorded using MS-DOS Operating System. The diskettes must contain all information relevant to one and only one SDG, and must accompany the hardcopy package for the SDG submitted to the Sample Management Office (see Exhibit B).

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 must start with a type 10 record, and the multiple type 10 records for each file of the same production run must be identical. Do not split the data from a single sample onto multiple diskettes.

8.2 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 on Exhibit H; when reporting 'calculated amounts' on Form 7D, hardcopy specified maximum length of 8 and so, a maximum length of 8 should be used. Blank or unused records must not be included on the diskettes. If the information submitted in the hardcopy data package forms is changed, the information in the diskette file must be changed accordingly, and a complete diskette containing all the information for the SDG must be resubmitted along with the hard copy at no additional cost to the EPA.

8.3 Each diskette must be identified with an external label containing (in this order) the following information:

Disk Density
File Name(s)
Laboratory Name (optional)
Laboratory Code
Case Number
SAS Number (where applicable)

The format for the File Name must be XXXXX.001 to XXXXX.099

where XXXXX is the SDG identifier, 0 designates organics, and 01 through 99 the file number.

Dimensions of the label must be in the range 4-3/4" to 5" long by 1-1/4" to 1-1/2" wide for 5-1/4 inch floppy diskette; and 2" to 2-1/4" long by 2-1/8" to 2-3/8" wide for 3.5 inch IBM-compatible diskette.

9. Record Listing

Following is a listing of every record type required to report data from a single SDG.

PRODUCTION RUN HEADER RECORD (Type 10)

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	RECORD TYPE	"10"
6	Delimiters	||||||
5	MEASUREMENT TYPE	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 or GC.

2 OLM01.0V For Volatiles; OLM01.0B For Semivolatiles; OLM01.0P For Pesticides.

CHROMATOGRAPHY RECORD (TYPE 11)

Use: To describe chromatograph condition. Must be present for volatiles and pesticides. Is optional for semivolatiles.

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	RECORD TYPE	"11"
1	Delimiter	|
10	GC COLUMN	Character
2	Delimiters	||
4	GC COLUMN ID	Numeric (mm)
11	Delimiters	|||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

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[2]
1	Delimiter	|
5	CASE NUMBER	Numeric
1	Delimiter	|
6	SDG NO.	Character
1	Delimiter	|
2	YEAR ANALYZED	YY
1	Delimiter	|
2	MONTH ANALYZED	MM
1	Delimiter	|
2	DAY ANALYZED	DD
1	Delimiter	|
2	HOUR ANALYZED	HH
1	Delimiter	|
2	MINUTE ANALYZED	MM
2	Delimiters	||
2	SAMPLE WT/VOL UNITS	"G"/"ML"[3]
1	Delimiter	|
5	SAMPLE WT/VOL	Numeric
1	Delimiter	|
3	ANALYTE COUNT	Numeric[4]
3	Delimiters	|||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

---

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, and "SRN" for dilutions.

3 Sample WT/VOL is the volume in mililiters for liquid and the wet weight in grams for solids. The sample units code indicates which units are in use for the current sample. Leave zero or blank if not applicable. Sample WT/VOL includes purge volume.

4 1-3 decimal digits; Counts all analytes including surrogates, internal standards, and all peaks for multi-component pesticides. For calibrations, count also DFTPP/BFB if mixed in injection.

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
1	Delimiter	|
1	LEVEL	"L"/"M"[1]
2	Delimiters	||
1	EXTRACTION	S/C/N[2]
2	Delimeters	||
6	SAS NUMBER	Character
1	Delimiter	|
14	LAB FILE/SAMPLE ID	Character[3]
1	Delimiter	|
2	YEAR EXTRACTED	YY
1	Delimiter	|
2	MONTH EXTRACTED	MM
1	Delimiter	|
2	DAY EXTRACTED	DD
2	Delimiters	||
2	YEAR RECEIVED	YY
1	Delimiter	|
2	MONTH RECEIVED	MM
1	Delimiter	|
2	DAY RECEIVED	DD
2	Delimiters	||
8	INJECTION/ALIQUOT VOLUME	Numeric[4]
2	Delimiters	||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

---

1 "L" for low level samples and "M" for medium level samples for volatile and semivolatile analyses. Leave blank for calibrations and tunes.

2 "S" for separatory funnel, "C" for continuous liq-liq and "N" for sonication.

3 Lab file ID for Volatile, Semivolatile; Lab sample ID for Pesticides in same format as on forms.

4 Injection volume for BNAs and PESTs; Soil Aliquot Volume for VOA.

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	|
2	CALIBRATION YEAR	YY[1]
1	Delimiter	|
2	CALIBRATION MONTH	MM
1	Delimiter	|
2	CALIBRATION DAY	DD
1	Delimiter	|
2	CALIBRATION HOUR	HH
1	Delimiter	|
2	CALIBRATION MINUTE	MM
1	Delimiter	|
14	CALIBRATION FILE ID	Character[2]
1	Delimiter	|
4	PH	Numeric
1	Delimiter	|
5	PERCENT MOISTURE	Numeric
1	Delimeter	|
1	DECANTED	"Y" or "N"
1	Delimeter	|
8	EXTRACT VOLUME	Numeric[3]
1	Delimiter	|
8	DILUTION FACTOR	Numeric[4]
3	Delimiters	|||
5	LEVEL	Numeric[5]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

1 For average, use the date and time average was calculated.

2 If "AVERAGE" is entered, then Form 8 will be constructed using the initial calibration with a QC code of "CLD". This field must match Lab Field/Sample ID on Type 21 for the associated calibration.

3 Use the initial extract volume adjusted (multiplied) by all contract-mandated dilutions that are to be excluded from the dilution factor. Soil extract volume for VOA; Conc. Extract volume for BNA and Pest. (In microliters)

4 Dilution factor of sample analyzed (omit contract-mandated dilutions).

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 = up to 10.0, High point = 30.0 to 100.0.

ASSOCIATED INJECTION AND COUNTER RECORD (TYPE 23)

Use: Continuation of Type 20. Used to identify associated blanks and tunes, and to count the number of surrogates and spikes outside of the QC limits and the number of TIC compounds. Used for Forms 3,4, and 5.

Position: Follows the Type 20, 21, and 22 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	RECORD TYPE	"23"
1	Delimiter	|
1	TUNE LABEL	"P" or blank
1	Delimiter	|
2	INJECTION YEAR	YY
1	Delimiter	|
2	INJECTION MONTH	MM
1	Delimiter	|
2	INJECTION DAY	DD
1	Delimiter	|
2	INJECTION HOUR	HH
1	Delimiter	|
2	INJECTION MINUTE	MM
1	Delimiter	|
14	DFTPP/BFB LAB FILE ID	Character
1	Delimiter	|
2	INSTRUMENT BLANK LABEL	"IB" or blank
1	Delimiter	|
2	BLANK INJECTION YEAR	YY
1	Delimiter	|
2	BLANK INJECTION MONTH	MM
1	Delimiter	|
2	BLANK INJECTION DAY	DD
1	Delimiter	|
2	BLANK INJECTION HOUR	HH
1	Delimiter	|
2	BLANK INJECTION MINUTE	MM
1	Delimiter	|
14	INSTRUMENT BLANK LAB FILE/SAMPLE ID	Character
4	Delimiters	||||
2	METHOD BLANK LABEL	"MB"
1	Delimiter	|
2	BLANK INJECTION YEAR	YY
1	Delimiter	|
2	BLANK INJECTION MONTH	MM
1	Delimiter	|
2	BLANK INJECTION DAY	DD
1	Delimiter	|
2	BLANK INJECTION HOUR	HH
1	Delimiter	|
2	BLANK INJECTION MINUTES	MM
1	Delimiter	|
14	METHOD BLANK LAB FILE/SAMPLE ID	CHARACTER
1	Delimiter	|

ASSOCIATED INJECTION AND COUNTER RECORD (TYPE 23) CONT:

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
1	SURROGATE RECOVERY LABEL	"P" for % recoveries
1	Delimiter	|
2	SURROGATE RECOVERIES OUT	Numeric
1	Delimiter	|
1	TIC LABEL	T" for TICS
1	Delimiter	|
2	NO. OF TICS	Numeric
1	Delimiter	|
1	SPIKE RECOVERY LABEL	"S" for Spikes
1	Delimiter	|
2	SPIKE RECOVERIES OUT	Numeric
1	Delimiter	|
1	RPD LABEL	"R" for RPD[1]
1	Delimiter	|
2	RPD OUT	Numeric
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

1 "R" for Matrix Spike/Duplicate Recovery Relative Percent Differences.

SAMPLE CLEANUP RECORD (TYPE 27)

Use: Continuation of Type 20. Used to identify sample/blank cleanup procedures and QC results. Used for Form 9.

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 CLEAN-UP TYPE	"G" for GPC
1	Delimiter	|
2	GPC CALIBRATION CHECK YEAR	YY
1	Delimiter	|
2	GPC CALIBRATION CHECK MONTH	MM
1	Delimiter	|
2	GPC CALIBRATION CHECK DAY	DD
1	Delimiter	|
2	GPC CALIBRATION CHECK HOUR	HH
1	Delimiter	|
2	GPC CALIBRATION CHECK MINUTE	MM
1	Delimiter	|
14	GPC Data Descriptor	Character[1]
1	Delimiter	|
1	SECOND CLEAN-UP TYPE	"F" or blank
1	Delimiter	|
2	FLORISIL LOT CHECK YEAR	YY
1	Delimiter	|
2	FLORISIL LOT CHECK MONTH	MM
1	Delimiter	|
2	FLORISIL LOT CHECK DAY	DD
1	Delimiter	|
2	FLORISIL LOT CHECK HOUR	HH
1	Delimiter	|
2	FLORISIL LOT CHECK MINUTE	MM
1	Delimiter	|
14	FLORISIL DATA DESCRIPTOR	Character[2]
1	Delimiter	|
1	SULFUR CLEAN-UP	Y/N
1	Delimiter	|
2	SULFUR BLANK LABEL	"SB"
1	Delimiter	|
2	BLANK INJECTION YEAR	YY
1	Delimiter	|
2	BLANK INJECTION MONTH	MM
1	Delimiter	|

1 Lab Sample ID for GPC (GPC Column). Format is "GPC" followed by unique identifier.

2 Lab Sample ID for Florisil lot check. Format is "FLO" followed by florisil cartridge lot number.

SAMPLE CLEANUP RECORD (TYPE 27) Cont.

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
1	Delimiter	|
2	BLANK INJECTION DAY	DD
1	Delimiter	|
2	BLANK INJECTION HOUR	HH
1	Delimiter	|
2	BLANK INJECTION MINUTE	MM
1	Delimiter	|
14	SULFUR BLANK LABORATORY FILE/SAMPLE ID	Character
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

RESULTS DATA RECORD (TYPE 30)

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	RECORD TYPE	"30"
1	Delimiter	|
1	ANALYTE LABEL	"C" for Cas Number
1	Delimiter	|
9	CAS NUMBER	Numeric
1	Delimiter	|
9	INTERNAL STD. CAS NUMBER	Numeric
1	Delimiter	|
5	CONCENTRATION UNITS	Character
1	Delimiter	|
3	RESULT QUALIFIER	Character[1]
1	Delimiter	|
13	RESULTS	Numeric
1	Delimiter	|
5	FLAGS	Character[2]
1	Delimiter	|
1	AMOUNT ADDED LABEL	"A" for Amt. added[3]
1	Delimiter	|
13	AMOUNT ADDED	Numeric
1	Delimiter	|
1	CRQL LABEL	"U" for "undetected" or blank |
1	Delimiter	|
13	CRQL	Numeric
1	Delimiter	|
1	RSD LABEL	"R" for RSD
1	Delimiter	|
5	RSD VALUE	Numeric[4]
1	Delimiter	|
1	MS/MSD REC LABEL	"P" for % recovery
1	Delimiter	|
5	MS % RECOVERY	Numeric
1	Delimiter	|
5	MSD % RECOVERY	Numeric
1	Delimiter	|
1	RPD LABEL	"D"
1	Delimiter	|
5	RPD VALUE	Numeric[5] |
1	Delimiter	|
1	SURR/SPIKE RECOVERY LABEL	"S" for % recovery
1	Delimiter	|
5	SURR/SPIKE RECOVERY	% Recovery[6]
1	Delimiter	|
1	MEAN CONCENTRATION LABEL	"M" for Mean conc.
1	Delimiter	|
13	MEAN CONCENTRATION	Numeric[7]
1	Delimiter	|
1	PERCENT DIFFERENCE LABEL	"F" or "P"[8]
1	Delimiter	|

RESULTS DATA RECORD (TYPE 30) CONT:

5	PERCENT DIFFERENCE	Numeric
1	Delimiter	|
1	INTERNAL STANDARD AREA LABEL	"I" for IS Area
1	Delimiter	|
13	INTERNAL STANDARD AREA	Numeric
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

1 For Type 20 for calibration summary (MNC), use "AVG" for average RRFs and Mean Calibration Factors.

2 A maximum of five flags (D,E,J,B,A,P or N) with no space between the flags can be reported, each representing a qualification of the result as described in Exhibit B.

3 Also Nominal Amount for Pest Form (7D-7E).

4 "R" for % Resolution (Form 6G) or for RSD of Response factors under Calibration summary (MNC) Type 20.

5 RPD for MS/MSD recoveries, or for Pest. Calibration Verification (Form 7D/7E).

6 Surrogate or Spike (Forms 2, Form 9A/9B) recovery.

7 Mean Concentration for Multicomponent analytes detected in Pesticide analyses.

8 "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/BNA analyses.

AUXILIARY DATA RECORD (TYPE 32)

Use: Used to report scan number and retention time (in minutes) for Internal Standards and for TIC compounds. 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	|
1	RT WINDOW UPPER LIMIT	Numeric
2	Delimiters	||
2	THIRD LIMIT LABEL	"PB" for % breakdown
1	Delimiter	|
5	% BREAKDOWN	Numeric (DDT/ENDRIN)
1	Delimiter	|
5	COMBINED % BREAKDOWN	Numeric
2	Delimiters	||
1	PEAK	1 THROUGH 5[1]
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

1 Peaks 1, 2, and 3 are mandatory, peaks 4 and 5 are optional. Types 30 and 31 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.

NAME RECORD (Type 33)

Use: To carry an analyte name for TIC compounds

Position: Follows Type 30 for TIC compounds.

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

INSTRUMENTAL DATA READOUT RECORD (TYPE 36)

Use: To describe DFTPP/BFB percent abundances for Form 5.

Position: Follows Type 30 for internal standards and 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
2	Delimiters	||
5	FIRST PERCENT ABUNDANCE	Numeric
1	Delimiter	|
3	SECOND MASS	Numeric
1	Delimiter	|
5	SECOND PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 69, BNA only
1	Delimiter	|
3	THIRD MASS	Numeric
1	Delimiter	|
5	THIRD PERCENT ABUNDANCE	Numeric
2	Delimiters	||
3	FOURTH MASS	Numeric
1	Delimiter	|
5	FOURTH PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 69, BNA only
1	Delimiter	|
3	FIFTH MASS	Numeric
1	Delimiter	|
5	FIFTH PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 174, VOA only
1	Delimiter	|
3	SIXTH MASS	Numeric
1	Delimiter	|
5	SIXTH PERCENT ABUNDANCE	Numeric
2	Delimiters	||
3	SEVENTH MASS	Numeric
1	Delimiter	|
5	SEVENTH PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 174, VOA only
1	Delimiter	|
3	EIGHTH MASS	Numeric
1	Delimiter	|
5	EIGHTH PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 174, VOA only

INSTRUMENTAL DATA READOUT RECORD (TYPE 36) CONT:

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
1	Delimiter	|
3	NINTH MASS	Numeric
1	Delimiter	|
5	NINTH PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 176, VOA only
1	Delimiter	|
3	TENTH MASS	Numeric
1	Delimiter	|
5	TENTH PERCENT ABUNDANCE	Numeric
2	Delimiters	||
3	ELEVENTH MASS	Numeric
1	Delimiter	|
5	ELEVENTH PERCENT ABUNDANCE	Numeric
2	Delimiters	||
3	TWELFTH MASS	Numeric
1	Delimiter	|
5	TWELFTH PERCENT ABUNDANCE	Numeric
2	Delimiters	||
3	THIRTEENTH MASS	Numeric
2	Delimiters	||
5	THIRTEENTH PERCENT ABUNDANCE	Numeric
1	Delimiter	|
5	PERCENT MASS	% mass of 442, BNA only
1	Delimiter	|
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

COMMENT RECORD (Type 90)

Use: To provide for Operator-Entered Comments.

Position: May occur anywhere

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. Definitions of Various Codes Used in Agency Standard Records

10.1 Quality Control and Related Codes (QCC) in Type 20 Records

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)
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 preceed LF2
LF2	LABORATORY SPIKED SAMPLE - FINAL - SECOND MEMBER	The "Matrix Spike Duplicate" (See Exhibit G)
LPC	LABORATORY PERFORMANCE CHECK SOLUTION	A solution of DFTPP (BNA) 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.
GPC	GPC CHECK SOLUTION	A solution of pesticides used to check recovery from each new GPC calibration.
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) rather than concentrations 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 CHECK CALIBRATION	The continuing calibration for GC/MS (See Exhibit G).
CLE	CONTINUING PERFORMANCE CHECK	The subsequent Individual Standard Mixes (A,B) and Performance Evaluation Mixture 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]

blank	Sample, not associated with any quality control item.

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. If the data apply to only a portion of the samples in the run, they should 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 RSD's 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, Source)

Medium	Code
All Media, Specific Medium not Applicable. Use for Calibrations, Tunes, etc	0
Water	1

10.3 List of Sample and Result Qualifiers

Definition: A sample qualifier (also called a non-numeric result) consists of 3 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
REX	RE-PREPARED	The indicated analysis results were generated from a re-extraction of the same sample
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)
SRS	SECONDARY DILUTION	The indicated analysis results were generated from a secondary dilution of the same sample (DL2 SUFFIX - Pesticides)

10.3.2 Result Qualifiers in Type 30 Records

A result qualifier (also called a non-numeric result) consists of 3 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.

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. Identifies analytes whose Internal Standard is not found.
LTL	LESS THAN LOWER CALIBRATION LIMIT	Actual value is known to be less than the lower calibration range due to dilution. (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 rejected by the laboratory.
STD	INTERNAL STANDARD	The indicated compound is an internal standard. There is no analysis result to report.
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)
MSP	PERCENT RECOVERY	The following value represents the percent recovery for the "MS" sample. The remaining two values give the "MSD" percent recovery and the Percent RPD.
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 TIC's (Form 1-E or 1-F "A" Flag).
NRP	NON-REPRODUCIBLE	Results of two or more injections are not comparable (Form ID "p" flag). e.g., Aroclor target analyte with greater than 25% difference between column analyses.
PRE	PRESUMPTIVE PRESENCE	Presumptive evidence of presence of material for TIC (Form 1-E or 1-F "N" flag).

11. Format of Records for Specific Uses

11.1 Format of the SAMPLE HEADER DATA RECORD (Type 20) for Mean Response Factors

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"20"	Record Type
3	Delimiters	|||
1	"0"	All matrices
1	Delimiter	|
3	"MNC"	Identifies Mean Response Factors
2	Delimiters	||
5	Case Number	Numeric
1	Delimiter	|
6	SDG No.	Character
9	Delimiters	|||||||||
3	Analyte Count	Numeric
3	Delimiters	|||
5	Record Sequence No.	Numeric
4	Checksum	Character

11.2 Format of the RESULTS DATA RECORD (Type 30) for Mean Relative Response Factors

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"30"	Record Type
1	Delimiter	|
1	"C"	"C" for CAS Number
1	Delimiter	|
9	CAS NUMBER	Numeric
1	Delimiter	|
9	INTERNAL STANDARD CAS NUMBER	Numeric
3	Delimiters	|||
3	"AVG"	Indicates Average Value
1	Delimiter	|
6	MEAN RESPONSE FACTOR	Right Justified
6	Delimiters	||||||
1	"R"	Indicates Percent RSD
1	Delimiter	|
5	PERCENT RSD	Numeric
14	Delimiters	||||||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

11.3 Format of the SAMPLE HEADER DATA RECORD (Type 20) for Matrix Spike Duplicates

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	Record Type	"20"
2	Delimiters	||
12	EPA Sample Number	As is exactly on the Form
1	Delimiter	|
1	Matrix	"H"
1	Delimiter	|
3	QC Code	LF2
2	Delimiters	||
5	Case Number	Numeric
1	Delimiter	|
6	SDG No.	Character
1	Delimiter	|
2	Year Analyzed	YY
1	Delimiter	|
2	Month Analyzed	MM
1	Delimiter	|
2	Day Analyzed	DD
1	Delimiter	|
2	Hour Analyzed	HH
1	Delimiter	|
2	Minute Analyzed	MM
2	Delimiters	||
1	Sample wt/vol unit	"G"
1	Delimiter	|
5	Sample wt/vol	Numeric
1	Delimiter	|
3	Analyte Count	Numeric
3	Delimiters	|||
5	Record Sequence Number	Numeric
4	Checksum	Character

11.4 Format of the COUNTER RECORD (Type 23) for Matrix Spike Duplicates (for VOA and BNA)

Position: Follows the type 20 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	Record Type	"23"
2	Delimiters	||
2	Injection Year	YY
1	Delimiter	|
2	Injection Month	MM
1	Delimiter	|
2	Injection Day	DD
1	Delimiter	|
2	Injection Hours	HH
1	Delimiter	|

2	Injection Minutes	MM
1	Delimiter	|
14	DFTPP Lab File ID	Character
11	Delimiters	|||||||||||
2	Method Blank Label	"MB"
1	Delimiter	|
2	Blank Injection Year	YY
1	Delimiter	|
2	Blank Injection Month	MM
1	Delimiter	|
2	Blank Injection Day	DD
1	Delimiter	|
2	Blank Injection Hour	HH
1	Delimiter	|
2	Blank Injection Minute	MM
1	Delimiter	|
14	Method Blank Lab File ID	Character
1	Delimiter	|
1	Surrogate Recovery Label	"P"
1	Delimiter	|
2	Surrogate Recovery Out	Numeric
3	Delimiters	|||
1	Spike Recovery Label	"S"
1	Delimiter	|
2	Spike Recovery Out	Numeric
1	Delimiter	|
1	RPD Label	"R"
1	Delimiter	|
2	RPD Out	Numeric
1	Delimiter	|
5	Record Sequence Number	Numeric
4	Checksum	Character

(Type 23) for Matrix Spike Duplicates (for Pesticide)

Position: Follows the type 20 to which it applies.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	Record Type	"23"
8	Delimiters	||||||||
2	Instrument Blank Label	"IB"
1	Delimiter	|
2	Blank Injection Year	YY
1	Delimiter	|
2	Blank Injection Month	MM
1	Delimiter	|
2	Blank Injection Day	DD
1	Delimiter	|
2	Blank Injection Hour	HH
1	Delimiter	|
2	Blank Injection Minute	MM
1	Delimiter	|
14	Instrument Blank Lab File ID	Character
4	Delimiters	||||
2	Method Blank Label	"MB"
1	Delimiter	|
2	Blank Injection Year	YY
1	Delimiter	|
2	Blank Injection Month	MM
1	Delimiter	|
2	Blank Injection Day	DD
1	Delimiter	|
2	Blank Injection Hour	HH
1	Delimiter	|
2	Blank Injection Minute	MM
1	Delimiter	|
14	Method Blank Lab File ID	Character
1	Delimiter	|
1	Surrogate Recovery Label	"P"
1	Delimiter	|
2	Surrogate Recovery Out	Numeric
3	Delimiters	|||
1	Spike Recovery Label	"S"
1	Delimiter	|
2	Spike Recovery Out	Numeric
1	Delimiter	|
1	RPD Label	"R"
1	Delimiter	|
1	RPD Out	Numeric
1	Delimiter	|
5	Record Sequence Number	Numeric
4	Checksum	Character

11.5 Format of the RESULTS DATA RECORD (Type 30) for Matrix Spike Duplicates

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	Record Type	"30"
1	Delimiter	|
1	Analyte Label	"C" for CAS No.
1	Delimiter	|
9	CAS Number	Numeric
1	Delimiter	|
9	Internal Standard CAS Number	Numeric
1	Delimiter	|
2	Concentration Unit	"NG"
1	Delimiter	|
3	Result Qualifier	STD
1	Delimiter	|
13	Results	Numeric
2	Delimiters	||
1	Amount Added Label	A
1	Delimiter	|
13	Amount Added	Numeric
1	Delimiter	|
1	CRQL Label	"U"
3	Delimiter	|||
1	MS/MSD Recovery Label	"P"
1	Delimiter	|
5	MS/MSD % Recovery	Numeric
2	Delimiters	||
1	RPD Label	"D"
1	Delimiter	|
1	Surrogate Recovery Label	"S"
1	Delimiter	|
5	Surrogate Recovery	% Recovery
1	Delimiter	|
1	Mean Concentration Label	"M"
1	Delimiter	|
1	Percent Difference Label	"P"
1	Delimiter	|
5	Percent Difference	Numeric
1	Delimiter	|
1	Internal Standard Area Label	"I"
1	Delimiter	|
13	Internal Standard Area	Numeric
1	Delimiter	|
5	Record Sequence Number	Numeric
4	Checksum	Character

11.6 Format of the Sample Header Data Record (Type 20) for Performance Evaluation Mixture

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"20"	Record Type
2	Delimiters	||
5	PEM ##	Sample I.D.
1	Delimiter	|
1	"0"	All matrices
1	Delimiter	|
3	"CLE"	Indicates Continuing Performance Check (Pesticide Standard)
2	Delimiters	||
5	CASE NUMBER	Numeric
1	Delimiter	|
5	SDG NO.	Character
1	Delimiter	|
2	YEAR OF INSTRUMENTAL ANALYSIS	YY
1	Delimiter	|
2	MONTH OF ANALYSIS	MM
1	Delimiter	|
2	DAY OF ANALYSIS	DD
1	Delimiter	|
2	HOUR OF INSTRUMENTAL ANALYSIS	HH
1	Delimiter	|
2	MINUTES OF ANALYSIS	MM
3	Delimiters	|||
3	ANALYTE COUNT	Numeric
3	Delimiters	|||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

11.7 Format of the Auxiliary Data Record (Type 32) for Pesticide Calibration Verification Summary (Percent Breakdown Data for 4,4'-DDT from Form 7D).

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"32"	Record Type
3	Delimiters	|||
2	"RT"	Retention time label.
1	Delimiter	|
5	RT Value	Numeric
1	Delimiter	|
3	"RTF"	First limit value
1	Delimiter	|
5	RT Window Lower Limit	Numeric
1	Delimiter	|
3	"RTT"	Second limit label
1	Delimiter	|
5	RT Window Upper Limit	Numeric
3	Delimiters	|||
2	"PB"	% Breakdown Label
1	Delimiter	|
5	% Breakdown	Numeric (for 4,4'-DDT).
4	Delimiters	||||
5	Record Sequence No.	Numeric
4	Checksum	Character

11.8 Format of the Sample Header Data Records (Type 20-30) for Continuing Checks (GC/MS Methods) Format

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"20"	Record Type
2	Delimiters	||
12	EPA Sample I.D.	e.g., VSTD050 From Exhibit B
1	Delimiter	|
1	"0"	All matrices
1	Delimiter	|
3	"CLC"	Indicates Continuing Check
1	Delimiter	|
3	Sample Qualifier	See Section 7.3
1	Delimiter	|
5	Case Number	Optional
1	Delimiter	|
5	SDG No.	Character
1	Delimiter	|
2	Year of Instrumental Analysis	YY
1	Delimiter	|
2	Month of Analysis	MM
1	Delimiter	|
2	Day of Analysis	DD
1	Delimiter	|
2	Hour of Instrumental Analysis	HH
1	Delimiter	|
2	Minute of Analysis	MM
3	Delimiters	|||
3	Analyte Count	Numeric
3	Delimiters	|||
5	Record Sequence No.	Numeric
4	Checksum	Character

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"21"	Record Type
1	Delimiter	|
1	"Y" or "N"	"Y" for heated; "N" for non-heated
5	Delimiters	|||||
6	SAS NUMBER	Leave Delimiter if none
1	Delimiter	|
14	LAB FILE I.D.	Character
11	Delimiters	|||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"23"	Record Type
1	Delimiter	|
1	"P"	Labels data as "tune" data
1	Delimiter	|
2	INJECTION YEAR	Date of associated DFTPP/BFB Injection
1	Delimiter	|
2	INJECTION MONTH	MM
1	Delimiter	|
2	INJECTION DAY	DD
1	Delimiter	|
2	INJECTION HOUR	HH
1	Delimiter	|
2	INJECTION MINUTES	MM
1	Delimiter	|
14	DFTPP/BFB LAB FILE ID	From Instrument data system
26	Delimiters	||||||||||||||||||||||||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character

MAXIMUM LENGTH	CONTENTS	FORMAT/ CONTENTS
2	"30"	Record Type
1	Delimiter	|
1	"C"	
1	Delimiter	|
9	CAS NUMBER	Numeric
1	Delimiter	|
9	CAS NUMBER INTERNAL STD. UTILIZED	Numeric
1	Delimiter	|
5	UNITS OF MEASURE	Character
1	Delimiter	|
3	RESULT QUALIFIER	
1	Delimiter	|
13	ANALYTICAL RESULT	Numeric
11	Delimiters	|||||||||||
1	"D"	Identifies Percent Difference
7	Delimiters	|||||||
13	RF PERCENT DIFFERENCE	Numeric
4	Delimiters	||||
5	RECORD SEQUENCE NO.	Numeric
4	CHECKSUM	Character