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Superfund

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK FOR ORGANICS ANALYSIS

MULTI-MEDIA, HIGH CONCENTRATION

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USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK
FOR
ORGANICS ANALYSIS

Multi-Media, High-Concentration

SOW No. Rev. 9/88
including Rev. 4/89

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 and extractable compounds listed on the Target Compound List (TCL) in Exhibit C. The Contractor shall employ state-of-the-art GC/MS and GC procedures to perform all analyses, including all 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. 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.

The Contractor shall separate multiphase samples into single phase units and prepare extracts and dilutions of samples. The Contractor shall screen extracts 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 achieved for each of the TCL compounds.

During preparation, the Contractor shall fortify all single phase units, blanks, and control matrix spikes with the surrogate spiking compounds listed in Exhibit E. Aliquots for volatile organics analysis shall be spiked with the internal standard compounds listed in Exhibit E before purging.

Additionally, for each single phase unit analyzed by GC/MS, the Contractor shall conduct mass spectral library searches to determine the possible identity of up to ten (10) nonsurrogate volatile components and up to twenty (20) nonsurrogate extractable components that are not on the Target Compound List (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.

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.

SECTION II

SPECIFIC REQUIREMENTS

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

Task I: Receive and Prepare Hazardous Waste Samples.

1. Receive and handle samples under the chain-of-custody procedures described in Exhibit F. Documentation, as described therein, shall be required to show that all procedures are being strictly followed.

2. Prepare samples as described in Exhibit D. Samples are separated into single phase units (if required) and screened to determine the proper dilution for GC/MS analysis.

   Extracts must be analyzed within 40 days of VTSR.

Task II: Analysis for Identity of Specific Organic Compounds.

1. Extracts and aliquots prepared in Task I shall be analyzed by GC 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.

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

1. The 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. 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, and

   b. Correspondence of the sample component and standard component mass spectra. This procedure requires the use of multiple internal standards.

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 tuning requirements of Tables 1.1 and 1.2 in Exhibit E. The standard spectra used may be from a laboratory generated library 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 in (1) must agree within plus or minus 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 Toxaphene and the 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 E) of the standard component analyzed on the same GC column and instrument, as part of the same analytical sequence specified in Exhibit D ARO.

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.

Task IV: Quantification of Compounds Verified in Task III.

1. The Contractor shall quantify components analyzed by GC/MS techniques and 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 E, Part 2, Tables 2.1 or 2.2.

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 calibration as described in Exhibit E.

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

4. The Contractor shall perform an initial three-point calibration, verify its linearity, determine the degradation of labile components, and determine calibration factors for all standards analyzed by GC/EC techniques as part of an analytical sequence, as described in Exhibit D ARO and Exhibit E.

Task V: Tentative Identification of Non-TCL Sample Components.

1. For each 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 the ten (10) nonsurrogate organic compounds of greatest concentration which are not listed in Exhibit C. For each extractable fraction, the Contractor shall conduct a search to determine the possible identification of the (20) nonsurrogate organic compounds of greatest concentration which are not listed in Exhibit C. In performing searches, the 1985 (or most recent) release of the National Bureau of Standards library (containing 42,261 spectra) 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.

   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.

   The Contractor shall not report as tentatively identified compounds (TIC) any TCL compounds from another analytical fraction (i.e., do not report late eluting volatile compounds as TICs in the semivolatile analysis).

Task VI: Quality Assurance/Quality Control Procedures.

1. All specific quality assurance procedures prescribed in Exhibit 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. Single phase units, method blanks and control matrix spikes shall be carried through the entire analytical process from extraction, GC screen, to final GC/MS analysis, including all data reporting requirements and magnetic tape data storage.

2. The Contractor shall perform one control matrix spike sample analysis for each case received, or for each 20 single phase units, or each 14 calendar day period during which single phase units in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group) whichever is most frequent.

   Samples, blanks and control matrix spikes shall be carried through the entire analytical process from extraction to final GC/MS analysis, including all Contract Performance/Delivery Requirements (see Contract Schedule).

3. The Contractor shall prepare and analyze one laboratory reagent blank (method blank) for each group once for:

   - each Case of single phase units received, OR
   - each 20 single phase units in a Case, OR
   - each 14 calendar day period during which single phase units in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
   - whenever samples are extracted,

   whichever is most frequent

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

   Extractable 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).

4. The Contractor shall perform instrument calibration (by "hardware tune") for each 12-hour time period, to include: 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 Performance Evaluation check samples submitted to the laboratory by EPA. The results of comparison studies are due within

40 calendar days of receipt of the samples. The results of all such control or PE check samples may be used as grounds for termination of noncompliant contractors.

B. EPA has provided to the Contractor formats for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in this 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

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

1. The Contractor shall have sufficient gas chromatograph (GC) and gas chromatograph/mass spectrometer/data system (GC/MS/DS) capability to meet all the terms and conditions of the Contract. Instrument requirements are defined in Section III, Detailed Technical & Management Requirements. 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. The GC/MS shall be equipped with a split/splitless injector and 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.

f. The GC for Aroclor analysis shall be equipped with dual wide bore (>0.53 mm) capillary columns and a suitable detector as described in Exhibit D. The instrument must be capable of operating with a temperature program.

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 B) for 365 days after data submission. During that time, the Contractor shall submit tapes and logbook within 7 days of request, as specified in the Contract Performance/Delivery Schedule.

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 1985 (or most recent) release of the National Bureau of Standards library (containing 42,261 spectra) 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.

D. The minimum functional requirements necessary to meet the terms and conditions of this contract are listed below. The Contractor shall designate and utilize key personnel to perform these functions. The EPA reserves the right to review personnel qualifications and experience. See Section III, Detailed Technical & Management Requirements.

- GC/MS/DS operation.
- Mass spectral interpretation.
- Sample extraction and concentration.
- Purge and trap volatile organic compounds analysis.
- Pesticide residue analysis of organochlorine pesticides and PCBs, including clean-up procedures.
- Quality assurance/quality control
- Sample receipt, storage, and tracking, including chain-of-custody procedures.

E. The Contractor shall respond in a timely manner to requests from data recipients for additional information or explanations that result from the Government's inspection activities.

F. 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 single phase unit extracts within 7 days after request, as specified in the Contract Performance/Delivery Schedule.

G. 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 Case file purge (see Exhibit B).

H. 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 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.

I. 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, <u>whichever is most frequent</u>:

- each Case of single phase units received, OR
- each 20 single phase units within a Case, OR
- each 14 calendar day period during which single phase units in a Case are received (said period beginning with the receipt of the first sample in the Sample Delivery Group).

Data for all samples in a Sample Delivery Group are due concurrently 40 days after receipt of the last sample received in the Sample Delivery Group. 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 40 days following this date.

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.

J. 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 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.

K. 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.

L. Samples will routinely be shipped 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.

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

SECTION III

DETAILED TECHNICAL & MANAGEMENT REQUIREMENTS

As cited in Section II, Task VI, the Contractor shall have the following technical and management capabilities:

A. TECHNICAL CAPABILITY

1. Technical Functions

a. GC/MS Laboratory Supervisor

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

(2) Qualifications:

(a) Education:

Minimum of Bachelor's degree in chemistry or any physical science.

(b) Experience:

Minimum of three years of laboratory experience, including at least one year of supervisory experience.

b. GC/MS Operator Qualifications

(1) Education:

Minimum of Bachelor's degree in chemistry or any physical science.

(2) Experience:

One year of experience in operating and maintaining GC/MS/DS with degree in chemistry or a physical science, or three years of experience in operating and maintaining GC/MS/DS.

c. Mass Spectral Interpretation Specialist Qualifications

(1) Education:

- Minimum of Bachelor's degree in chemistry or any physical science.
- Training course(s) in mass spectral interpretation.

(2) Experience:

Minimum of two years of experience.

d. GC Laboratory Supervisor

(1) Responsible for all technical efforts of the GC laboratory.

(2) Qualifications:

(a) Education:

Minimum of Bachelor's degree in chemistry or any physical science.

(b) Experience:

Minimum of three years of laboratory experience, including at least one year of supervisory experience.

e. Pesticide Residue Analysis Expert Qualifications

(1) Education:

Minimum of Bachelor's degree in chemistry or any physical science.

(2) Experience:

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

f. Sample Preparation Laboratory Supervisor

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

(2) Qualifications:

(a) Education:

Minimum of Bachelor's degree in chemistry or any physical science.

(b) Experience:

Minimum of three years of laboratory experience, including at least one year of supervisory experience.

g. Extraction/Concentration Expert Qualifications

(1) Education:

Minimum of High school diploma and knowledge of general chemistry.

(2) Experience:

Minimum of one year of experience.

h. 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.

(1) Education:

Minimum of Bachelor's degree in chemistry or any physical science.

(2) Experience: Minimum of one year in each of the following areas -

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

2. 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.

a. Sample Receipt Area

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

b. 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. NOTE: Volatiles, semivolatiles, extracts, and standards must each be stored separately.

c. Sample Preparation Area

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

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

(2) Source of distilled or demineralized organic-free water.

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

3. Instrumentation

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

a. Primary Instrument Requirements

(1) 60 Phase Units/Month Capacity

Purpose	Fraction	No. of Instrument(s)	Type of Instrument
Analysis	Volatiles	1	GC/MS/DS with purge and trap device
	Extractables	1	GC/MS/DS
	Aroclors/ Toxaphene	1	GC/EC with dual column
GPC Cleanup	Extractables	1	GPC with UV detector
Screening		1	GC/FID

Note: For contracts of two (2) bid lots or more:

- Minimum of three (3) GC/MS/DS and three (3) GC systems are required at the time of on-site laboratory evaluation.
- An additional one (1) GC/MS/DS and one (1) GC system with dual detectors are required as a back-up system at the time of on-site laboratory evaluation.

b. Secondary Instrument Requirements

(1) 60 Phase Units/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 with dual detectors (FID and EC)

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 turnaround times.

c. 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	Exhibits A and D
o GC	Exhibit D

4. Data Handling and Packaging

The Contractor shall 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:

a. Provide space, tables and copy machines to meet the contract requirements.

b. Designate personnel.

B. 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 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:

1. Technical Staff

   Responsible for all technical efforts for the EPA contract.

2. Project Manager

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

3. Sample Custodian

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

4. Quality Assurance Officer

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

5. Data Reporting and Delivery Officer

   Responsible for all aspects of data deliverables: organization, packaging, copying, and delivery.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

EXHIBIT B
REPORTING AND DELIVERABLES REQUIREMENTS

SECTION I

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

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

Item	No. Copies	Delivery Schedule	Distribution (1)	(2)
A. Contract Start-Up Plan	2	7 Days after contract award	X	X
B. Updated SOPs	1	120 days after contract award	X	

Item	No. Copies	Delivery Schedule	Distribution (3)	(4)	(5)	(6)
**C. Sample Traffic Reports	1	3 days after receipt of last sample in Sample Delivery Group (SDG)****	X			
***D. Sample Data Summary Package last sample in SDG	1	35 days after receipt of	X			
***E. Sample Data Package	3	35 days after receipt of last sample in SDG	X	X	X	
F. GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by PO and/or EMSL/LV.	As Directed			

Item	No. Copies	Delivery Schedule	Distribution (3)	(4)	(5)	(6)
G. Extracts	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by PO or SMO		As Directed		
H. Complete Case File Purge	1 Pkg	Submit 180 days after data submission or 7 days after receipt of written request by PO or SMO.				X

* Contractor must be prepared to receive samples within 30 days of contract award. NOTE: EPA cannot guarantee exact adherence to start-up plan that is agreed upon by the PO and Contractor, but will attempt to meet it as close as possible.

** Also required in the Sample Data Package.

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

**** Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 14 days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. (See SOW Exhibit A, paragraph J., for further description).

NOTE: As specified in the Contract Schedule (G.6 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 analytical data.

Distribution Addresses:

(1) USEPA Analytical Operations Branch (WH 548A)
401 M Street, SW
Washington, DC 20460
ATTN: (Project Officer's Name)

(2) USEPA
Contracts Management Division (MD-33)
Administration Building Lobby, Alexander Drive
Research Triangle Park, NC 27711
ATTN: (Contract Officer's Name)

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

For overnight delivery service, use street address:
209 Madison Street, Suite 200
Alexandria, VA 22314

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

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

(5) USEPA REGIONS:

The CLP Sample Management Office acting on behalf of the 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.

(6) NEIC, Contractor Evidence Audit Team
12600 West Colfax, Suite 310
Lakewood, Colorado 80215

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.1). The required content and form of each deliverable is described in this Exhibit.

All reports and documentation MUST BE:

- o Legible,
- o Clearly labeled and completed in accordance with instructions in this Exhibit,
- o Arranged in the order specified in this Section, and
- o Paginated.

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 PO/DPO action, 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 copies of the required data reporting forms in Agency-specified formats, along with instructions to assist the Contractor in accurately providing the Agency all required data.

Descriptions of the requirements for each deliverable item cited in the Contract Performance/Delivery Schedule (Contract Schedule, Section F.1) are specified in parts A-G 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.

Examples of specific data deliverables not included herein may be obtained by submitting a written request to the EPA Project Officer, stating the information requested, and signed by the Laboratory Manager.

A. Contract Start-Up Plan

The Contractor shall submit a contract start-up plan for EPA approval as specified in the Contract Performance/Delivery Schedule. The plan shall set forth the Contractor's proposed schedule for receiving samples starting with the 30th calendar day after award and ending with the date the Contractor is capable of receiving the full monthly sample allotment stipulated in the Contract. The Project Officer will review the contract start-up plan within 7 days of submission and will notify the Contractor of the plan's status.

NOTE: The Contractor shall be required to receive samples within 30 days of contract award. EPA can't guarantee exact adherence to start-up plan that is agreed upon by the PO and Contractor, but will attempt to meet it as close as possible.

B. Updated SOPs

The Contractor shall submit updated copies of all required Standard Operating Procedures (SOPs) that were submitted with the prebid Performance Evaluation sample results. The updated SOPs must address any and all issues of laboratory performance and operation identified through the review of the Performance Evaluation sample data and the evaluation of Bidder-Supplied Documentation.

The Contractor must supply SOPs for :

1. Sample receipt and logging.
2. Sample and extract storage.
3. Preventing sample contamination.
4. Security for laboratory and samples.
5. Traceability/Equivalency of standards.
6. Maintaining instrument records and logbooks.
7. Sample analysis and data control systems.
8. Glassware cleaning.
9. Technical and managerial review of laboratory operation and data package preparation.
10. Internal review of contractually-required quality assurance and quality control data for each individual data package.
11. Sample analysis, data handling and reporting.
12. Chain-of-custody.
13. Document control, including case file preparation.

C. 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, all the samples on one multi-sample TR may not be necessarily 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.

D. 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 described under part C, 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. 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. Case Narrative

2. By fraction (HCV, HCE, and HCA,) and by phase unit within each fraction - tabulated target compound results (Form I) and tentatively identified compounds (Form I, TIC) (HCV, HCE only).

3. By fraction (HCV, HCE, HCA) - surrogate spike analysis results (Form II).

4. By fraction (HCV, HCE, HCA) - control matrix spike results (Form III)

5. By fraction (HCV, HCE, HCA) - blank data (Form IV) and tabulated results (Form I) including tentatively identified compounds (Form I, TIC)(HCV, HCE only).

E. 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, extractables, 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, and control matrix spikes.

1. Case Narrative

   This document shall be clearly labeled "Case Narrative" and shall contain: laboratory name; Case number; sample numbers in the Sample Delivery Group (SDG) and the phase units analyzed from each sample, 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 Case 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.

The Case 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 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 Case 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 are 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. High Concentration Volatiles Data

a. QC Summary

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

(2) Control Matrix Spike Summary (Form III HCV)

(3) Method Blank Summary (Form IV HCV)

(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 Tuning and Mass Calibration (Form V HCV)

BFB in chronological order; by instrument.

b. Sample Data

Sample data shall be arranged in packets with the High Concentration Volatile Analysis Data Sheet (Form I HCV, including Form I HCV-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. Phase units within a sample should be placed in order by phase unit suffix (i.e. AB123-11, AB123-12, etc.)

(1) TCL Results - High Concentration Volatile Analysis Data Sheet (Form I HCV).

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 Case Narrative (reference C.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 Case Narrative.

On Form I, the appropriate concentration units shall be mg/kg. No other units are acceptable. NOTE: Report analytical results to one significant figure if the value is less than 10; to two significant figures above 10.

(2) Tentatively Identified Compounds (Form I 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 HCV-TIC is the tabulated list of the highest probable match for up to 10 of the nonsurrogate organic compounds not listed in Exhibit C (TCL), including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentration. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. NOTE: The laboratory must be consistent (i.e., use peak height for all comparisons or use total area count for all comparisons).

(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 standard and surrogate spiking 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 Compound List (TCL) 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 of identified TCL 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

(4) For each sample, by each compound identified:

(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 TCL 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 HCV) - in order by instrument, if more than one instrument used.

(a) HCV 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 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 HCV) - in order by instrument, if more than one instrument used.

(a) HCV 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.

(3) Internal Standard Area Summary (Form VIII HCV) - in order by instrument, if more than one instrument used.

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

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

(a) Tabulated results (Form I HCV).

(b) Tentatively Identified Compounds (Form I HCV-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) TCL spectra with lab generated standard, labeled as in b.(4) above. Data systems which are incapable of dual display shall provide spectra in order:

- o Raw TCL compound spectra
- o Enhanced or background subtracted spectra
- o Laboratory generated TCL 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) Control Matrix Spike Data

(a) Tabulated results (Form I HCV) of nonspiked TCL compounds. Form I HCV-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. Extractables Data

a. QC Summary

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

(2) Control Matrix Spike Summary (Form III HCE)

(3) Method Blank Summary (Form IV HCE)

(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 Tuning and Mass Calibration (Form V HCE)

DFTPP in chronological order; by instrument.

b. Sample Data

Sample data shall be arranged in packets with the High Concentration Extractable Analysis Data Sheet (Form I HCE, including Form I HCE-TIC), followed by the raw data for extractable samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers in ordering samples. Phase units within a sample should be placed in order by phase unit suffix (i.e., AB123-11, AB123-12, etc.).

(1) TCL Results - High Concentration Extractable Analysis Data Sheet (Form I HCE-1, HCE-2, HCE-3).

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 Case Narrative (reference C.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 Case Narrative.

On Form I, the appropriate concentration units shall be mg/kg. No other units are acceptable. NOTE: Report analytical results to one significant figure if the value is less than 10; to two significant figures above 10.

(2) Tentatively Identified Compounds (Form I HCE-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 HCE-TIC is the tabulated list of the highest probable match for up to 20 of the nonsurrogate organic compounds not listed in Exhibit C (TCL), including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentration. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. NOTE: The laboratory must be consistent (i.e., use peak height for all comparisons or use total area count for all comparisons).

(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 standard and surrogate spiking 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 Compound List (TCL) 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 of identified TCL 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

(4) For each sample, by each compound identified:

(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 TCL 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 HCE-1, HCE-2, HCE-3) - in order by instrument, if more than one instrument used.

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

(b) All initial calibration data 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 HCE-1, HCE-2, HCE-3) - in order by instrument, if more than one instrument used.

(a) Extractable 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) Internal Standard Area Summary (Form VIII HCE-1, HCE-2) - in order by instrument, if more than one instrument used.

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

(4) GPC Calibration (Form IX HCE) - in order by calibration date.

(a) Copies of UV traces for each GPC calibration performed labeled with date and time of the calibration, and labeling each peak in the calibration standard with the name of the compound and its retention time.

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.

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

(a) Tabulated results (Form I HCE-1, HCE-2, HCE-3)

(b) Tentatively Identified Compounds (Form I HCE-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) TCL spectra with lab generated standard, labeled as in b.(4) above. Data systems which are incapable of dual display shall provide spectra in order:

- o Raw TCL compound spectra
- o Enhanced or background subtracted spectra
- o Laboratory generated TCL 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) Control Matrix Spike Data

(a) Tabulated results (Form I) of nonspiked TCL compounds. Form 1 HCE-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. Aroclor/Toxaphene Data

a. QC Summary

(1) High Concentration Aroclor Surrogate Recovery Summary (Form II HCA)

(2) High Concentration Aroclor Control Matrix Spike Recovery Summary (Form III HCA)

(3) High Concentration Aroclor Method Blank Summary (Form IV HCA-1)

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

(4) High Concentration Aroclor Instrument Blank Summary (Form IV HCA-2)

(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 High Concentration Aroclor Analysis Data Sheet (Form I HCA), followed by the raw data for samples. These sample packets

should then be placed in increasing EPA sample number order, considering both letters and numbers in ordering samples.

(1) TCL Results - High Concentration Aroclor Analysis Data Sheet (Form I HCA).

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 Case Narrative (reference C.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 Case Narrative.

On Form I, the appropriate concentration units shall be mg/kg. No other units are acceptable.

NOTE: Report analytical results to two significant figures for all Aroclor/Toxaphene samples.

(2) Copies of Aroclor/Toxaphene analysis 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)
- 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 Aroclor/Toxaphene analysis chromatograms from second GC column confirmation. Chromatograms to be labeled as in (2) above.

(4) Manual work sheets.

c. Standards Data

(1) Form VI HCA-1 and HCA-2 - High Concentration Initial Calibration of Multicomponent Analytes (all GC columns)

(2) Form VII HCA - High Concentration Continuing Calibration of Multicomponent Analytes (all GC columns)

(3) Form VIII HCA - High Concentration Analytical Sequence (all GC columns)

(4) Form IX HCA - High Concentration Single Component Pesticide Retention Times (all GC columns)

(5) Form X HCA - High Concentration Aroclor Identification Summary (only required for positive results)

(6) Form XI HCA - High Concentration Aroclor Diol Cartridge Check (all lot numbers used)

(7) Aroclor/Toxaphene standard chromatograms and data system printouts for <u>all</u> standards to include:

- o All Aroclors and Toxaphene
- o All quantitation standards
- o Diol cartridge check standard for each lot of cartridges used
- o A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

(a) All chromatograms are required to have the following:

- 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 Label the chromatogram for multicomponent standards. (i.e., Aroclor 1242, Toxaphene)
- o List total ng injected for each standard.
- o A printout of retention times and corresponding peak areas must accompany each chromatogram.
- o Date and time of injection.
- o GC column identification (by stationary phase).
- o GC instrument identification.

d. Raw QC Data

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

(a) Tabulated results (Form I HCA).

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

(a) Tabulated results (Form I HCA).

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

(3) Control Matrix Spike Data

(a) Tabulated results (Form I HCA) of nonspike TCL compounds.

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

F. GC/MS Tapes

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, control matrix spikes, 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, and control matrix spikes. 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. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the Project Officer or the Sample Management Office.

G. 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 Project Officer or the Sample Management Office.

H. Complete Case File Purge

(Formerly, Document Control and Chain-of-Custody Package).

The complete case file purge includes all laboratory records received or generated for a specific Case that have not been previously submitted to EPA as a deliverable. These items include but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence, and the document inventory (see Exhibit F).

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, or extractable). 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 high concentration volatile analysis only, provide only HCV forms. There are three pages relating to the extractable fraction for Forms I, VI, VII, and VIII. Whenever extractables are analyzed and one of the above named forms is required, all three pages (HCE-1, HCE-2, and HCE-3) must be submitted. In addition to the forms for high concentration volatiles and high concentration extractable, forms are provided for the Aroclor-specific GC/EC analysis of high concentration samples (HCA). These instructions are arranged in the following order:

A. General Information and Header Information

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

C. Surrogate Recovery (Form II, All Fractions)

D. Control Matrix Spike Recovery (Form III, All Fractions)

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

F. GC/MS Tuning and Mass Calibration (Form V HCV, Form V HCE)

G. Initial Calibration Data (Form VI HCV, Form VI HCE, Form VI HCA)

H. Continuing Calibration Data (Form VII HCV, Form VII HCE, Form VII HCA)

I. Internal Standard Area Summary (Form VIII HCV, Form VIII HCE) and Analytical Sequence (Form VIII HCA)

J. GPC Calibration (Form IX HCE) and Pesticide Retention Times (Form IX HCA)

K. Aroclor Identification Summary (Form X HCA)

L. Diol Cartridge Check (Form XI HCA)

A. General Information and Header Information

Values must be reported on the forms according to the individual form instructions in this Section. For example, results for concentrations of HCV TCL compounds must be reported to two significant figures if the value is greater than or equal to 10.

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 Project Officer. The names of the various fields and compounds (i.e., "Lab Code," "Chloromethane") must appear as they do on the forms in the contract. For items appearing on the uncompleted forms (Section IV), the use of uppercase and lowercase letters is optional.

Do not remove the underscores or vertical bar characters that delineate "boxes" on the forms. The only exception would be those underscores at the bottom of a "box" that are intended as a data entry line (for instance, see Form 2HA, 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. 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.

The "Lab Code" is an alphabetical abbreviation of up to 6 letters, assigned by EPA, to identify the laboratory and aid in data processing. This lab code shall be assigned by EPA at the time a contract is awarded, and shall not be modified by the contractor, except at the direction of EPA.

The "Case No." is the EPA-assigned Case number (up to 5 digits) 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 right-hand corner of the form, or as the left column of a table summarizing data from a number of samples. When "EPA Sample No." is entered into the triple-spaced box in the upper right-hand corner of Form I, it should be entered on the middle line of the three lines that comprise the box.

All samples, control matrix spikes, blanks and standards shall be identified with an EPA Sample Number. Because a sample may comprise a number of single-phase units, the use of sample number suffixes is necessary to differentiate between phase units. The following system of suffixes must be used:

XXXXX - EPA sample number

XXXXX-11 - first phase unit

XXXXX-12 - second phase unit

through

XXXXX-19 - ninth phase unit

XXXXX-YYRE - reanalysis of phase unit YY

XXXXX-YYDL - phase unit YY analyzed at a secondary dilution.

CMS - Control Matrix Spike

Note: If more than nine phases are separated from a single sample, contact SMO for instructions on numbering the phases.

Use of the suffixes "RE" and/or "DL" assumes that data are also being submitted for an analysis of a phase unit that represents the "original" analysis of the phase unit. If only one set of data are being submitted, do not use the suffix "RE" or "DL".

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. Extractable blanks shall be identified as EBLK##.

3. Aroclor method blanks shall be identified as ABLK##.

4. Aroclor instrument blanks shall be identified as IBLK##.

   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, VBLK10, VBLKAB, etc.

5. Volatile and extractable standards shall be identified as:

   FSTD###, where:

   F = fraction (V for volatiles; E for extractable).

   ### = the concentration in ug/L of volatile standards (i.e., 20, 50, 100, 150, 200)

   or

   the amount injected in ng for extractable standards (i.e. 50, 80, 160). For PCB standards, the amounts injected would be 10, 30, 50 or 20, 60, 100 ng. If PCB standards are coinjected in different amounts, use the lower amount (i.e., ESTD10 for coinjection of 10 and 20 ng of PCBs)

6. Aroclor standards shall be identified as specified in the instructions for Form VIII.

Several other pieces of information are common to many of the Data Reporting Forms. These include: Phase type, Phase weight, and Lab Sample ID.

For the purposes of this contract, there are only three possible phase types. They are:

Solids
Water Immiscible Liquids
Water Miscible Liquids

For "Phase Type", enter "SOLID" for solid phase units. Enter "WIL" for water immiscible liquids, and enter "WML" for water miscible liquids. Water samples are considered as water miscible liquids, and would be entered as "WML", not "WATER".

For "Phase weight" enter the number of grams of phase unit used.

"Lab Sample ID" is an optional laboratory-generated internal identifier.

"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.

Forms II, IV, V, and VIII contain a field labeled "page _ of _" in the bottom left-hand corner. If the number of entries required on any of these forms exceeds the available space, continue entries on another copy of the same fraction-specific form, duplicating all header information. If a second page is required, number them consecutively, as "page 1 of 2" and "page 2 of 2". If a second page is not required, number the page "page 1 of 1."

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)

1. Form I HCV, Form I HCE-1, Form I HCE-2, Form I HCE-3, Form I HCA

This form is used for tabulating and reporting sample analysis results for Target Compound List (TCL) compounds. If all fractions are not requested to be analyzed, only the pages specifically required must be submitted. If HCV analysis only is requested, Form I HCV and Form I HCV TIC must be submitted. If the Aroclor-specific analysis is the only analysis requested, only Form I HCA 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:

Enter pH for Extractables and 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 Separated", "Date Extracted" and "Date Analyzed" should be entered in a similar fashion. For Aroclor-specific samples, the date of analysis should be the date of the first GC analysis performed.

For all fractions, enter the final volume in milliliters of the sample extract under "Final Extract Volume". Report volume to one tenth of a milliliter (i.e. 10.0, not 10).

For volatiles, enter the number of microliters of the extract that was added to the reagent water under "Aliquot Volume".

For extractable, enter the number of microliters of the extract that is injected under "Injection Volume".

For all three fractions, enter under "Conversion Factor" the value used to convert your raw data into the concentration values in mg/Kg reported on Form I for all detected compounds. Note: this conversion factor must incorporate the phase weight, the final extract volume, the injection or aliquot volume, the use of GPC clean-up procedures, and any dilution of the extract or sample that is required. The units associated with this factor will vary, depending on the units given in the raw data. Therefore, no units for the conversion factor are to be reported on Form I.

If samples for Aroclor/Toxaphene analysis were subjected to sulfur clean-up procedures, enter "Y" under "Sulfur Clean-up (Y/N)." If no sulfur clean-up was performed enter "N".

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

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

Report all Aroclor-specific results to two significant figures.

The concentration units are mg/Kg for all phase types and fractions.

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 Case Narrative.

For reporting results to the USEPA, the following contract specific qualifiers are to be used. The eight 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 eight 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. For example, 20 U for phenol 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 200 U.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral or GC/EC data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero. For example, if the sample quantitation limit is 10 mg/Kg, but a concentration of 3 mg/Kg is calculated, report it as 3J. The sample quantitation limit must be adjusted for dilution as discussed for the U flag.

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 TCL compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. This flag will not apply to Aroclors analyzed by GC/EC methods. If one or more compounds have a response greater than full scale, the 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 Forms I. The Form I for the diluted sample shall have the "DL" suffix appended to the sample number.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample 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.

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

N - This flag identifies Aroclor or Toxaphene compounds where one or more of the peaks used for quantitation are more than two times the width of the corresponding peaks in the highest concentration calibration standard. It indicates an uncertainty in the quantitation for the compound other than those discussed under the "J" flag.

X - Other specific flags and footnotes may be required to properly define the results. In order to limit the number of laboratory-defined flags and not use such flags as may be part of the Agency's data review processes, the laboratory-defined flags are restricted to the three letters "X", "Y", and "Z". If used, they must be fully described and such description attached to the Sample Data Summary Package and the Case Narrative. If more than one 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 combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are also detected in the sample.

If analyses at two different dilution factors are required (see Exhibit D), follow the data reporting instructions given in Exhibit D and with the "D" and "E" flags above.

2. Form I HCV-TIC and Form I HCE-TIC

Fill in all header information as above.

Report Tentatively Identified Compounds (TIC) 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 HCV-TIC or HCE-TIC for every volatile and extractable 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).

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-TCL extractable compounds to be searched. NOTE: The name of the first of these aldol-condensation reaction products will have to be truncated to 28 characters (i.e., "4-methyl-4-hydroxy-2-pentano").

C. Surrogate Recovery (Form II)

Form II is used to report the recoveries of the surrogate compounds added to each sample, blank, and control matrix spike. Form II is fraction-specific.

Complete the header information and enter EPA Sample Numbers as described in part A. For each surrogate, report the percent recovery 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 right-hand column, total the number of surrogate recoveries outside the QC limits for each sample. If no surrogates were outside the limits, enter "0".

If 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.

On Form II HCA, for Aroclor-specific analyses by GC/EC, report the surrogate recoveries from both GC columns, identifying each column by

stationary phase in the fields labelled "GC Column ID (1)" and "GC Column ID (2)".

If an interference prevents the quantitation of one of the two Aroclor surrogates, enter that value as "0", flag it in column under the "#" symbol, and describe the problem in the Case Narrative.

Number all pages as described in part A.

D. Control Matrix Spike Recovery (Form III)

This form is used to report the results of the analyses of the control matrix spike.

Complete the header information as instructed in Part A.

In the box on Form III, under "SPIKE ADDED", enter the calculated concentration in mg/Kg that results from adding each spiked compound to the aliquot chosen for the control matrix spike (CMS). For instance, for extractable compounds, if 100 ug of spike are added to 1 g of sample, the resulting concentration is 100 mg/Kg. Under "CMS CONCENTRATION", enter the actual concentration of each spike compound detected in the control matrix spike aliquot. Calculate the percent recovery of each spike compound in the control matrix spike to the nearest whole percent, according to Exhibit E, and enter under "CMS % 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.

On Form III HCA, there are no QC limits on recovery at this time.

For volatiles and extractables, 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 data.

E. Method Blank Summary (Form IV)

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.

For volatile and extractable blanks, enter the "Instrument ID", "Date Analyzed", and "Time Analyzed". The "Time Analyzed" shall be in military time.

For extractable blanks and Aroclor method blanks, enter the date of extraction of the blank.

If some of the samples associated with a given method blank are subjected to sulfur clean-up procedures and some samples are not, then two method blanks are required, one subjected to sulfur clean-up and one not. (See Exhibit D for suggestions regarding the aliquotting of method

blank extracts). If the method blank has been subjected to sulfur clean-up, then enter "Y" under "Sulfur Clean-up (Y/N)," and list in the table only those samples associated with that blank that have also undergone sulfur clean-up. Complete a separate Form IV HCA-1 for the aliquot of the method that did not undergo sulfur clean-up, listing the associated samples that also did not undergo sulfur clean-up. NOTE: The aliquotting of the method blank will require that different blank identifiers be used in place of the EPA Sample Number for each blank on all deliverables. (See Section A above).

Aroclor/Toxaphene contaminants must meet the identification criteria in Exhibit D ARO, which requires analysis of the blank on two different GC Columns. Therefore, enter the date, time and instrument ID of both analyses 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 Form X. Otherwise (1) shall be the first analysis, and (2) the second. Identify both GC columns by stationary phase under "GC Column ID".

Aroclor/Toxaphene analyses also require the analysis of an instrument blank at specified points in the analytical sequence. For each instrument blank that is associated with phase units in a data package, complete a copy of Form IV HCA-2, and summarize the samples associated with that instrument blank, as described below. NOTE: The samples associated with an instrument blank may not be the same as those associated with a method blank. Samples associated with an instrument blank are those that were analyzed in the 12-hour period prior to the instrument blank in question, and after the previous acceptable instrument 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 extractables, enter Lab File ID and the date and time of analysis of each sample.

Number all pages as described in part A.

F. GC/MS Tuning and Mass Calibration (Form V)

This form is used to report the results of GC/MS tuning for volatiles and extractable, and to summarize the date and time of analysis of samples, standards, blanks, and control matrix spikes associated with each GC/MS tune.

Complete the header information as in part A. Enter the "Lab File ID" for the injection containing the GC/MS tuning compound (BFB for volatiles, DFTPP for extractable). Enter the "Instrument ID". Enter the date and time of injection of the tuning compound. Enter time as military time.

For each ion listed on the form, enter the percent relative abundance in the right-hand column. Report relative abundances to the number of

significant figures given for each ion in the ion abundance criteria column.

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, and control matrix spikes analyzed under that tune 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, and control matrix spikes.

The GC/MS tune expires twelve hours from the time of injection of the tuning compound (BFB or DFTPP) listed at the top of the form. In order to meet the tuning requirements, a sample, standard, blank, or control matrix spike must be injected within twelve hours of the injection of the tuning compound.

Number all pages as described in part A.

G. Initial Calibration Data (Form VI)

After a GC/MS system has undergone an initial calibration at the specific concentration levels described in Exhibit E, and after all initial calibration criteria have been met, the laboratory must complete and submit a Form VI for each volatile or extractable TCL initial calibration performed which is relevant to the samples, blanks, or control matrix spikes in the SDG, regardless of when that calibration was performed. A five-point initial calibration is required for volatiles. A three-point calibration is required for extractables.

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 "Lab File ID" for each of the calibration standards injected. Complete the response factor data for the calibration points, and then calculate and report the average relative response factor (RRF) for all TCL and surrogate compounds. The laboratory must report the %RSD for all compounds. All CCC compounds must have a %RSD of less than or equal to 30.0 percent. All HCV SPCC compounds must have a minimum average relative response factor (RRF) of 0.300 (0.250 for Bromoform). All extractable SPCC compounds must have a minimum average relative response factor (RRF) of 0.050.

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

where: %RSD = Relative Standard Deviation

SD = Standard Deviation of initial response factors (per compound)

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \bar{x})^2}{n-1}}$$

$\bar{x}$ = mean of initial response factors (per compound)

The PCBs listed on Form VI HCE-3 are injected in different amounts than the other extractable standards, and the octa-decachlorinated biphenyls are injected at twice the amount as the lower levels of chlorination. Enter the two sets of Lab File IDs for the injections of the PCBs in the appropriate spaces on the lower half of this form. If the PCBs are injected along with the other standards, enter the same Lab File IDs as at the top of the form. If the PCBs are all injected in one standard regardless of level of chlorination, but separate from the other extractable standards, enter one set of Lab File IDs for the extractable standards, and enter a second set of Lab File IDs for the PCBs, using the same Lab File ID for RRF10 as for RRF20, etc.

The initial calibration of the Aroclors and Toxaphene is reported on Form VI HCA-1 and HCA-2. Complete all header information as in part A. For each GC column used for Aroclor/Toxaphene analyses, complete a copy of Form VI HCA-1 and HCA-2, identifying the stationary phase of the GC Column under "GC Column ID". Each analyte requires a three-point calibration, therefore, for each GC Column, there will be three pages of Form VI HCA-1. The "Date(s) Analyzed" at the top of the form must be the inclusive dates of analysis for all 24 of the standards in the initial calibration.

For each injection of each standard, enter the date and time of injection, and the amount injected in nanograms.

The calibration of these multicomponent analytes requires the use of at least three chromatographic peaks. Two additional peaks may be reported as well to allow for coeluting interferences. For each peak used, enter the retention time in minutes and decimal minutes (<u>not</u> minutes and seconds), and the calibration factor for each peak.

Calculate a mean of the three retention times for each peak of each analyte, and enter under "MEAN RT". Calculate a mean deviation at the three retentions for each peak, according to the formula in Ex. D. ARO, and report this value under "MD RT".

Calculate a mean and relative standard deviation of the three calibration factors for each peak in each analyte according to Ex. D ARO, and enter them on Form VI HCA-2 under "Mean Cal. Factor" and "%RSD".

If the mean and %RSD do not meet the specifications in Ex. D. ARO, the laboratory may use a regression line through all three calibration points, or a two segment regression line (see Ex. D. ARO). If the mean and %RSD meet the specifications in Ex. D. ARO, leave blank the regression coefficients and intercept fields.

If a single correlation line is used, enter the mean, %RSD, and the regression coefficient "r (1)" and the intercept "INTCPT (1)".

If a two segment regression line is used, enter both correlation coefficients and intercepts "r (1)"and "r (2)", etc. Also enter the mean RT and %RSD.

H. Continuing Calibration Data (Form VII)

1. Form VII HCV, HCE-1, HCE-2, and HCE-3

The Continuing Calibration Data Form is used to verify the calibration of the GC/MS system by the analysis of specific calibration standards. A Continuing Calibration Data Form is required for each twelve (12) hour time period for both volatile and extractable TCL compound analyses.

The Contractor laboratory must analyze calibration standards and meet all criteria outlined in Exhibit E. After meeting specific criteria for both SPCC and CCC compounds, a Continuing Calibration Data Form must be completed and submitted.

Complete all header information as in part A. Enter instrument ID, date and time of continuing calibration, the Lab File ID of the continuing calibration standard, and date of initial calibration (give inclusive dates if initial calibration is performed over more than one date). Using the appropriate Initial Calibration (Volatile or extractable) fill in the average relative response factor (RRF) for each TCL compound. Report the relative response factor from the continuing calibration standard analysis. Calculate the Percent Difference (%D) for all compounds. For CCC compounds, ensure that the %D is less than or equal to 25.0 percent. After this criterion has been met, report the Percent Difference for all TCL and surrogate 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 continuing calibration standards for extractables (except PCBs) are analyzed at 80 total ng. The PCB standards are analyzed at 30 total ng for mono-heptachlorinated biphenyls, and 60 total ng for octa-decachlorinated biphenyls. As on Form VI, if these standards are co-injected, enter the same Lab File ID in both locations on the form.

2. Form VII HCA

The calibration of multicomponent analytes is verified by a comparison of the initial calibration factors to those obtained by the periodic analysis of Performance Evaluation Mixtures (PEM).

Complete the header information as in Part A. The GC Column ID must match that on the corresponding Form VI HCA. For each PEM injected, record the date and time of analysis on Form VII, along with amount injected in nanograms. For each of the three required peaks, and any additional peaks, enter the retention time of the peak, in minutes and decimal minutes. For each peak, calculate the percent difference between the retention time of each peak in the PEM and the mean retention time of that peak on Form VI HCA-2, according to the formula above. Report this value under "%D" on Form VII HCA.

Enter initial calibration factor for each peak from Form VI HCA under "Initial Cal. Factor". Enter the calibration factor from the injection listed on Form VII HCA under "Continuing Cal. Factor." Calculate the relative percent difference according to Ex. D. ARO, and enter "RPD".

Number all pages as part A.

I. Form VIII

1. Internal Standard Area Summary (Form VIII HCV and HCE)

This form is used to summarize the peak areas of the internal standards added to all volatile and extractable samples, blanks, and control matrix spikes. 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 tune as an initial calibration.

Complete the header information as in part 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 GC/MS tune 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 80 ng initial calibration standard for extractables. 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.

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

For each sample, blank, and control matrix spike 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.

Number all pages as described in part A.

2. Analytical Sequence (Form VIII HCA)

This form is used to record the sequence of analysis of samples and standards for all Aroclor/Toxaphene analyses. Data must be provided for the initial calibration standards as well as the samples in the SDG. If samples in the SDG being reported were analyzed immediately after the initial calibration, list them immediately following the calibration standards. If an older calibration is being used, list the initial calibration standards on one Form VIII HCA, and the samples on separate forms, (as with Form V for volatiles or extractables).

For each Form VIII HCA, complete all header information as in part A. Enter the stationary phase of the GC column under "GC Column ID". Begin each Form VIII HCA with calibration standards or performance evaluation mixtures associated with the samples.

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

Name	EPA Sample Number
Toxaphene	TOXAPH
Aroclor 1016	AR1016
Aroclor 1221	AR1221
Aroclor 1232	AR1232
Aroclor 1242	AR1242
Aroclor 1248	AR1248
Aroclor 1254	AR1254
Aroclor 1260	AR1260

If Aroclor 1016 and Aroclor 1260 are combined into one standard mixture, use AR1660 as the "EPA Sample Number".

Instrument Blanks must be identified as "IBLK##", where "##" may be any combination of numbers or letters needed to distinguish between various instrument blanks in one SDG.

For each sample, standard, control matrix spike, and blank, enter the EPA sample number, lab sample ID, date and time of analysis.

Every sample, standard, control matrix spike, method blank, and instrument blank must contain the surrogates Tetrachloro-meta-Xylene (TMX) and Decachlorobiphenyl (DEC) at the level specified in Ex. D. ARO. Calculate the retention time shift for each of the surrogates on both GC columns according to the following formula, and report under the appropriate column for %D on Form VIII HCA.

$$\% \text{ Difference} = \frac{\overline{RT} - RT_s}{\overline{RT}}$$

where:

RT = Mean retention time from initial calibration of the surrogate

RT_s = Retention time of the sample of interest

The %D for the surrogates in every sample, standard, control matrix spike, and blank must be less than or equal to 0.5%, as calculated above. Flag each retention time shift outside the QC limits with an asterisk in the last column, under the "*". If the surrogates have been diluted out, and no %D can be calculated, enter "dil" under "%D", and describe in the Case Narrative.

Number all pages according to part A.

J. Form IX

1. Extractable GPC Calibration (Form IX HCE)

This form reports the results of the calibration of the gel permeation chromatographic apparatus (GPC) used to clean-up sample extracts. It reports the retention times of the four GPC calibration compounds, and lists the phase units associated with that calibration.

Complete the header information as in Part A. Enter the laboratory's GPC column identifier under "GPC Column ID", and the date of the GPC calibration.

For each of the four GPC calibration compounds, enter the retention time of that compound during the initial calibration under "INITIAL CALIB. RT". Enter the retention time of each calibration compound from the calibration verification standard

under "CALIB. VERIF. RT". Calculate the percent difference in the two retention times according to the formula for the surrogates above, and enter under "%D". There are no QC limits on percent difference for Polystrene or Pentachlorophenol. The QC limit on the other two GPC calibration compounds is 5%.

In the lower portion of the form, list the phase units which are associated with this GPC calibration. Enter the EPA Sample No., including the phase unit suffix, and the Lab Sample ID, Lab File ID, and the date and time analyzed by GC/MS.

Include a copy of Form IX HCE for each GPC calibration performed that is associated with samples in a data package. Number all pages as in Part A.

2. Pesticide Retention Times (Form IX HCA)

This form reports the retention times and retention time windows of the single component pesticide compounds. While these pesticides are <u>not</u> target compounds by the GC/EC methods used for Aroclors and Toxaphene, their retention times are reported with the Aroclor/Toxaphene data to ensure that peaks for pesticide compounds are not misidentified as Aroclor or Toxaphene peaks.

The retention times of the single compound pesticides must be determined at least once per initial calibration on each instrument and each GC column used for sample analysis. These data must be submitted with each group of samples analyzed under that initial calibration.

Retention time windows are determined as a plus/minus percentage of the retention time. For the four BHC compounds and Heptachlor, the retention time window is ±1.5% of the retention time. The retention time windows of the remaining 15 compounds are ±1.0% of the retention time.

Complete the header information as in Part A. Enter the GC Column ID by stationary phase. This GC Column ID must match that on Forms II, VII, and VIII HCA. Enter the inclusive dates of analysis under "Date(s) of Analysis".

Enter the retention time of each pesticide under "RT" in minutes and <u>decimal</u> minutes, not minutes and seconds. Calculate the retention time windows according to the percentages above, and report as a part of from/to values, <u>not</u> as a plus/minus percentage.

No Form X HCA is required for the analyses of these single component pesticides.

Number all pages as in Part A.

K. Aroclor/Toxaphene Identification Summary (Form X)

This form summarizes the tentative and confirmed identity of all TCL Aroclors/Toxaphene detected in a given sample. It reports the retention times of each quantitation peak in the compound on both columns on which it was analyzed, as well as the retention time windows of the same peaks in the standard for that compound on both of these columns. One copy of Form X is required for each sample, control matrix spike, or blank in which Aroclors/Toxaphene 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 Part A. Enter the GC Column ID (By stationary phase) for each of the two columns, one as GC Column (1), the other as (2). Enter the Instrument ID associated with each GC column directly below.

For each Aroclor/Toxaphene detected, enter the name of the target compound on the line under "COMPOUND". For each of the quantification peaks selected for that compound (at least 3 peaks are required), enter the retention time of that peak on each GC column in the appropriate box under "RT" in <u>decimal</u> minutes. Enter the retention time windows of the same peaks in the standard for that compound. The retention time window of the standard is defined as ±1.0% of the mean retention time of the peak determined in the initial calibration. The lower value is entered under the "FROM" column, the upper value under the "TO" column.

Calculate the concentration of each Aroclor or Toxaphene peak reported on Form X from the calibration factors on each of the two columns used. Enter the concentration under the "CONCENTRATION" column. Calculate a mean of the 3-5 concentrations for each analyte on each column that result, and enter this value under the "MEAN CONCENTRATION" column. Note: There will be one mean concentration per compound in each column. Report the <u>lower</u> of the two mean concentrations on Form I HCA.

Calculate the percent difference between the two mean concentrations, according to the formula below, and enter this value under the "%D" column. If the percent difference is greater than 25% of the lower value, flag the result on Form I with one of the optional flags "X,Y,or Z". Define your use of the flag in the Case Narrative.

$$\%\ D = \frac{C_H - C_L}{C_L}$$

where:

C_L = lower mean concentration (based on 3-5 peaks)

C_H = higher mean concentration (based on 3-5 peaks)

If more 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 Part A.

L. Diol Cartridge Check (Form XI)

This form is used to report the results of the Diol cartridge check. The amount of the spike compound passed through the cartridge and removed from the eluent must meet the QC limits listed on the form before that lot of Diol cartridges may be used for extract clean-up. The form also lists all the phase units in the data package which were cleaned using Diol cartridges from this lot.

Complete all header information as in Part A. Enter the lot number of the Diol cartridges, and the date of the analysis of the cartridge check solution.

Enter the amount of Aroclor 1254 spiked in the solution in nanograms and the amount recovered under "SPIKE ADDED" and "SPIKE RECOVERY" respectively.

Calculate a percent recovery according to the formula in Ex. D ARO, and enter under "% REC". This value must be within the QC limits of 80-110%.

In the lower portion of the form, enter the EPA Sample No., including phase unit suffix, of each phase unit which was cleaned using a cartridge from this lot. If there is a Lab Sample ID, enter it here as well. If more than 25 phase units used cartridges from the same lot, duplicate the header information, and continue on another copy of Form XI, numbering the pages as in Part A.

SECTION IV

DATA REPORTING FORMS

1HA

HIGH CONCENTRATION VOLATILE ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Lab File ID: _____

Final Extract Volume: _____ (mL) Date Received: _____

Aliquot Volume: _____ (uL) Date Separated: _____

Conversion Factor: _____ Date Extracted: _____

Date Analyzed: _____

CAS NO.	COMPOUND	CONCENTRATION (mg/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		
108-05-4	Vinyl Acetate		
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)		

1HB

HIGH CONCENTRATION EXTRACTABLE ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name:_____ Contract:_____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Lab File ID: _____

Final Extract Volume: _____ (mL) Date Received: _____

Injection Volume: _____ (uL) Date Separated:_____

Conversion Factor: _____ Date Extracted:_____

pH: ___ Date Analyzed: _____

CAS NO.	COMPOUND	CONCENTRATION (mg/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		
100-51-6	Benzyl alcohol		
95-50-1	1,2-Dichlorobenzene		
95-48-7	2-Methylphenol		
108-60-1	bis(2-Chloroisopropyl)ether		
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		
65-85-0	Benzoic acid		
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		

1HC

HIGH CONCENTRATION EXTRACTABLE ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name:_____ Contract:_____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Lab File ID: _____

Final Extract Volume: _____ (mL) Date Received: _____

Injection Volume: _____ (uL) Date Separated: _____

Conversion Factor: _____ Date Extracted: _____

pH: ___ Date Analyzed: _____

CAS NO.	COMPOUND	CONCENTRATION (mg/Kg)	Q
99-09-2	3-Nitroaniline		
83-32-9	Acenaphthene		
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		
319-84-6	alpha-BHC		
118-74-1	Hexachlorobenzene		
319-85-7	beta-BHC		
87-86-5	Pentachlorophenol		
58-89-9	gamma-BHC (Lindane)		
85-01-8	Phenanthrene		
120-12-7	Anthracene		
319-86-8	delta-BHC		
76-44-8	Heptachlor		
309-00-2	Aldrin		
84-74-2	Di-n-butylphthalate		
206-44-0	Fluoranthene		
1024-57-3	Heptachlor epoxide		
27323-18-8	Monochlorobiphenyl		
2051-60-7	Dichlorobiphenyl		
2051-61-8	Trichlorobiphenyl		
2051-62-9	Tetrachlorobiphenyl		
129-00-0	Pyrene		
5103-74-2	gamma-Chlordane		

(1) - Cannot be separated from Diphenylamine

1HD

HIGH CONCENTRATION EXTRACTABLE ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name:_____ Contract:_____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Lab File ID: _____

Final Extract Volume: _____ (mL) Date Received: _____

Injection Volume: _____ (uL) Date Separated:_____

Conversion Factor: _____ Date Extracted:_____

pH: ___ Date Analyzed: _____

CAS NO.	COMPOUND	CONCENTRATION (mg/Kg)	Q
959-98-8	Endosulfan I		
5103-71-9	alpha-Chlordane		
25429-29-2	Pentachlorobiphenyl		
72-55-9	4,4'-DDE		
60-57-1	Dieldrin		
26601-64-9	Hexachlorobiphenyl		
72-20-8	Endrin		
33213-65-9	Endosulfan II		
72-54-8	4,4'-DDD		
28655-71-2	Heptachlorobiphenyl		
85-68-7	Butylbenzylphthalate		
1031-07-8	Endosulfan sulfate		
50-29-3	4,4'-DDT		
53494-70-5	Endrin ketone		
56-55-3	Benzo(a)anthracene		
72-43-5	Methoxychlor		
218-01-9	Chrysene		
55722-26-4	Octachlorobiphenyl		
91-94-1	3,3'-Dichlorobenzidine		
117-81-7	bis(2-Ethylhexyl)phthalate		
53742-07-7	Nonachlorobiphenyl		
2051-24-3	Decachlorobiphenyl		
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		

1HE

EPA SAMPLE NO.

HIGH CONCENTRATION VOLATILE ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name:_____ Contract:_____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Lab File ID: _____

Final Extract Volume: _____ (mL) Date Received: _____

Aliquot Volume: _____ (uL) Date Separated:_____

Conversion Factor: _____ Date Analyzed: _____

Number TICs found: _____

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC. (mg/Kg)	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.					

1HF

EPA SAMPLE NO.

HIGH CONCENTRATION EXTRACTABLE ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name: _____ Contract: _____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Lab File ID: _____

Final Extract Volume: _____ (mL) Date Received: _____

Injection Volume: _____ (uL) Date Separated: _____

Conversion Factor: _____ Date Extracted: _____

pH: ___ Date Analyzed: _____

Number TICs found: _____

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC. (mg/Kg)	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.					

1HG

HIGH CONCENTRATION AROCLOR ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name:_____ Contract:_____

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

Phase Type: _____ Lab Sample ID: _____

Phase weight: _____ (g) Date Received: _____

Final Extract Volume: _____ (mL) Date Separated:_____

Injection Volume: _____ (uL) Date Extracted:_____

Conversion Factor: _____ Date Analyzed: _____

pH: ___ Sulfur Clean-up: ____ (Y/N)

CAS NO.	COMPOUND	CONCENTRATION (mg/Kg)	Q
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		

2HA

HIGH CONCENTRATION VOLATILE SURROGATE RECOVERY

Lab Name:_____ Contract:_____

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

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (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
S1 (TOL) = Toluene-d8	(50-160)
S2 (BFB) = Bromofluorobenzene	(50-160)
S3 (DCE) = 1,2-Dichloroethane-d4	(50-160)

Column to be used to flag recovery values

D Surrogates diluted out

Method blanks are required to meet the QC limits. For samples and control matrix spikes, the QC limits are advisory.

2HB

HIGH CONCENTRATION EXTRACTABLE 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) #	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
S1 (NBZ) = Nitrobenzene-d5	(20-140)
S2 (FBP) = 2-Fluorobiphenyl	(20-140)
S3 (TPH) = Terphenyl-d14	(20-150)
S4 (PHL) = Phenol-d5	(20-140)
S5 (2FP) = 2-Fluorophenol	(20-140)
S6 (TBP) = 2,4,6-Tribromophenol	(10-140)

Column to be used to flag recovery values

D Surrogates diluted out

Method blanks are required to meet the QC limits. For samples and control matrix spikes, the QC limits are advisory

2HC

HIGH CONCENTRATION AROCLOR SURROGATE RECOVERY

Lab Name:_____ Contract:_____

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

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

	EPA SAMPLE NO.	COL.1 S1 (TMX) #	COL. 2 S1 (TMX) #	COL.1 S2 (DEC) #	COL. 2 S2 (DEC) #
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 (TMX) = Tetrachloro-meta-Xylene	(40-120)
S2 (DEC) = Decachlorobiphenyl	(40-120)

Column to be used to flag recovery values

D Surrogate diluted out

3HA

HIGH CONCENTRATION VOLATILE CONTROL MATRIX SPIKE RECOVERY

Lab Name:_____ Contract:_____

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

COMPOUND	SPIKE ADDED (mg/Kg)	CMS CONCENTRATION (mg/Kg)	CMS % REC #	QC. LIMITS REC.
1,1-Dichloroethene				60-150
Trichloroethene				60-150
Benzene				60-150
Toluene				60-150
Chlorobenzene				60-150

Column to be used to flag recovery values with an asterisk

* Values outside of QC limits

Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

_____

3HB

HIGH CONCENTRATION EXTRACTABLE CONTROL MATRIX SPIKE RECOVERY

Lab Name:_____ Contract:_____

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

COMPOUND	SPIKE ADDED (mg/Kg)	CMS CONCENTRATION (mg/Kg)	CMS % REC #	QC. LIMITS REC.
Phenol				10-120
2-Chlorophenol				10-120
1,4-Dichlorobenzene				30-140
N-Nitroso-di-n-prop.(1)				30-140
1,2,4-Trichlorobenzene				30-140
4-Chloro-3-methylphenol				10-120
Acenaphthene				30-140
4-Nitrophenol				10-120
2,4-Dinitrotoluene				30-140
Pentachlorophenol				10-120
Heptachlor				30-140
Pyrene				30-140
Dieldrin				30-140

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

Column to be used to flag recovery values with an asterisk

* Values outside of QC limits

Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

_____

3HC

HIGH CONCENTRATION AROCLOR CONTROL MATRIX SPIKE RECOVERY

Lab Name:_____ Contract:_____

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

COMPOUND	SPIKE ADDED (mg/Kg)	CMS CONCENTRATION (mg/Kg)	CMS % REC	QC. LIMITS REC.
Aroclor 1254				none

COMMENTS: _____

_____

4HA

HIGH CONCENTRATION VOLATILE METHOD BLANK SUMMARY

Lab Name:_____ Contract:_____

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

Lab Sample ID: _____ Date Analyzed: _____

Lab File ID: _____ Time Analyzed: _____

Instrument ID: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND CMS:

	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: _____

_____

4HB

HIGH CONCENTRATION EXTRACTABLE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

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

Lab Sample ID: _____ Date Extracted: _____

Lab File ID: _____ Date Analyzed: _____

Instrument ID: _____ Time Analyzed: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND CMS:

	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					
23					
24					
25					
26					
27					
28					
29					
30					

COMMENTS: _____

4HC

HIGH CONCENTRATION AROCLOR METHOD BLANK SUMMARY

Lab Name:_____ Contract:_____

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

Lab Sample ID: _____ Date Extracted: _____

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

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

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

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

Sulfur Clean-up: ____ (Y/N)

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND CMS:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	TIME ANALYZED 1	DATE ANALYZED 2	TIME 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						
27						
28						
29						
30						

COMMENTS: _____

_____

4HD

HIGH CONCENTRATION AROCLOR INSTRUMENT BLANK SUMMARY

Lab Name:_____ Contract:_____

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

Lab Sample ID: _____ Date Prepared: _____

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

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

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

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

THIS INSTRUMENT BLANK APPLIES TO THE FOLLOWING SAMPLES AND CMS:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	TIME ANALYZED 1	DATE ANALYZED 2	TIME 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						
27						
28						
29						
30						

COMMENTS: _____

_____

5HA

HIGH CONCENTRATION VOLATILE GC/MS TUNING AND MASS CALIBRATION - BROMOFLUOROBENZENE (BFB)

Lab Name:_____ Contract:_____

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

Lab File ID: _____ BFB Injection Date:_____

Instrument ID: _____ BFB Injection Time:_____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 60.0% 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	Greater than 50.0% of mass 95	
175	5.0 - 9.0% of mass 174	( )1
176	Greater than 95.0%, but less than 101.0% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

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

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, CMS, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
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17					
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20					
21					
22					

5HB

HIGH CONCENTRATION EXTRACTABLE GC/MS TUNING AND MASS CALIBRATION - 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 - 60.0% 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	40.0 - 60.0% 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 1.00% of mass 198	
441	Present, but less than mass 443	
442	Greater than 40.0% of mass 198	
443	17.0 - 23.0% of mass 442	( )2

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

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, CMS, 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					

6HA

HIGH CONCENTRATION VOLATILE INITIAL CALIBRATION DATA

Lab Name:_____ Contract:_____

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

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

Min $\overline{RRF}$ for SPCC(#) = 0.300 (0.250 for Bromoform) Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF20 =_____ RRF50 =_____

RRF100=_____ RRF150=_____ RRF200=_____

COMPOUND	RRF20	RRF50	RRF100	RRF150	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							
Vinyl Acetate							
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							

6HB

HIGH CONCENTRATION EXTRACTABLE INITIAL CALIBRATION DATA

Lab Name:_____ Contract:_____

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

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

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

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

COMPOUND	RRF50	RRF80	RRF160	$\overline{RRF}$	% RSD
Phenol	*				*
bis(2-Chloroethyl)ether					
2-Chlorophenol					
1,3-Dichlorobenzene					
1,4-Dichlorobenzene	*				*
Benzyl alcohol					
1,2-Dichlorobenzene					
2-Methylphenol					
bis(2-Chloroisopropyl)ether					
4-Methylphenol					
N-Nitroso-di-n-propylamine	#				#
Hexachloroethane					
Nitrobenzene					
Isophorone					
2-Nitrophenol	*				*
2,4-Dimethylphenol					
Benzoic acid					
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	#				#

6HC

HIGH CONCENTRATION EXTRACTABLE INITIAL CALIBRATION DATA

Lab Name:_____ Contract:_____

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

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

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

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

COMPOUND	RRF50	RRF80	RRF160	$\overline{RRF}$	% RSD
Dibenzofuran					
2,4-Dinitrotoluene					
Diethylphthalate					
4-Chlorophenyl-phenylether					
Fluorene					
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine (1)	*				*
4-Bromophenyl-phenylether					
alpha-BHC					
Hexachlorobenzene					
beta-BHC					
Pentachlorophenol	*				*
gamma-BHC (Lindane)					
Phenanthrene					
Anthracene					
delta-BHC					
Heptachlor					
Aldrin					
Di-n-butylphthalate					
Fluoranthene	*				*
Heptachlor epoxide					
Pyrene					
gamma-Chlordane					
Endosulfan I					
alpha-Chlordane					
4,4'-DDE					
Dieldrin					
Endrin					
Endosulfan II					
4,4'-DDD					
Butylbenzylphthalate					
Endosulfan sulfate					
4,4'-DDT					
Endrin ketone					
Benzo(a)anthracene					
Methoxychlor					

(1) Cannot be separated from Diphenylamine

6HD

HIGH CONCENTRATION EXTRACTABLE INITIAL CALIBRATION DATA

Lab Name:_____ Contract:_____

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

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

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0

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

COMPOUND	RRF50	RRF80	RRF160	$\overline{RRF}$	% RSD
Chrysene					
3,3'-Dichlorobenzidine					
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					

LAB FILE ID: RRF10 =_____ RRF30 =_____
RRF50 =_____

	RRF10	RRF30	RRF50	$\overline{RRF}$	% RSD
Monochlorobiphenyl					
Dichlorobiphenyl					
Trichlorobiphenyl					
Tetrachlorobiphenyl					
Pentachlorobiphenyl					
Hexachlorobiphenyl					
Heptachlorobiphenyl					

LAB FILE ID: RRF20 =_____ RRF60 =_____
RRF100=_____

	RRF20	RRF60	RRF100	$\overline{RRF}$	% RSD
Octachlorobiphenyl					
Nonachlorobiphenyl					
Decachlorobiphenyl					

6HE

HIGH CONCENTRATION INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name:_____ Contract:_____

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

Instrument ID: _____ GC Column ID: _____

COMPOUND	AMOUNT (ng)	PEAK	RT	CALIBRATION FACTOR
Toxaphene		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1016		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1221		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1232		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1242		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1248		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1254		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		
Aroclor 1260		*1		
Date: _____		*2		
		*3		
Time: _____		4		
		5		

* Denotes required peaks

6HF

HIGH CONCENTRATION INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name:_____ Contract:_____

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

Instrument ID: _____ GC Column ID: _____

Date(s) Analyzed: _____ _____

COMPOUND	PEAK	MEAN RT	MEAN CAL. FACTOR	% RSD	r (1)	INTCP (1)	r (2)	INTCP (2)
Toxaphene	*1							
	*2							
	*3							
	4							
	5							
Arolcor 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

7HA

HIGH CONCENTRATION 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):_____ _____

Min RRF50 for SPCC(#) = 0.300 (0.250 for Bromoform) Max %D for CCC(*) = 25.0%

COMPOUND	$\overline{RRF}$	RRF50	%D
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			
Vinyl Acetate			
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			

7HB

HIGH CONCENTRATION EXTRACTABLE 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):_____ _____

Min RRF80 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.0%

COMPOUND		$\overline{RRF}$	RRF80	%D	
Phenol	*				*
bis(2-Chloroethyl)ether					
2-Chlorophenol					
1,3-Dichlorobenzene					
1,4-Dichlorobenzene	*				*
Benzyl alcohol					
1,2-Dichlorobenzene					
2-Methylphenol					
bis(2-Chloroisopropyl)ether					
4-Methylphenol					
N-Nitroso-di-n-propylamine	#				#
Hexachloroethane					
Nitrobenzene					
Isophorone					
2-Nitrophenol	*				*
2,4-Dimethylphenol					
Benzoic acid					
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	#				#

7HC

HIGH CONCENTRATION EXTRACTABLE 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):_____ _____

Min RRF80 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.0%

COMPOUND	$\overline{RRF}$	RRF80	%D
Dibenzofuran			
2,4-Dinitrotoluene			
Diethylphthalate			
4-Chlorophenyl-phenylether			
Fluorene			
4-Nitroaniline			
4,6-Dinitro-2-methylphenol			
N-Nitrosodiphenylamine (1) *			*
4-Bromophenyl-phenylether			
alpha-BHC			
Hexachlorobenzene			
beta-BHC			
Pentachlorophenol *			*
gamma-BHC (Lindane)			
Phenanthrene			
Anthracene			
delta-BHC			
Heptachlor			
Aldrin			
Di-n-butylphthalate			
Fluoranthene *			*
Heptachlor epoxide			
Pyrene			
gamma-Chlordane			
Endosulfan I			
alpha-Chlordane			
4,4'-DDE			
Dieldrin			
Endrin			
Endosulfan II			
4,4'-DDD			
Butylbenzylphthalate			
Endosulfan sulfate			
4,4'-DDT			
Endrin ketone			
Benzo(a)anthracene			
Methoxychlor			

(1) Cannot be separated from Diphenylamine

7HD

HIGH CONCENTRATION EXTRACTABLE 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):_____ _____

Min RRF80 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.0%

COMPOUND	$\overline{RRF}$	RRF80	%D
Chrysene			
3,3'-Dichlorobenzidine			
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			

LAB FILE ID: RRF10 = _____	$\overline{RRF}$	RRF30	%D
Monochlorobiphenyl			
Dichlorobiphenyl			
Trichlorobiphenyl			
Tetrachlorobiphenyl			
Pentachlorobiphenyl			
Hexachlorobiphenyl			
Heptachlorobiphenyl			

LAB FILE ID: RRF20 = _____	$\overline{RRF}$	RRF60	%D
Octachlorobiphenyl			
Nonachlorobiphenyl			
Decachlorobiphenyl			

7HE

HIGH CONCENTRATION CONTINUING CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name:_____ Contract:_____

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

Instrument ID: _____ GC Column ID: _____

COMPOUND	AMOUNT (ng)	PEAK	RT	%D	INITIAL CAL. FACTOR	CONTINUING CAL. FACTOR	RPD
Toxaphene		*1					
Date:		*2					
		*3					
Time:		4					
		5					
Aroclor 1016		*1					
Date:		*2					
		*3					
Time:		4					
		5					
Aroclor 1248		*1					
Date:		*2					
		*3					
Time:		4					
		5					
Aroclor 1254		*1					
Date:		*2					
		*3					
Time:		4					
		5					
Aroclor 1260		*1					
Date:		*2					
		*3					
Time:		4					
		5					

* Denotes required peaks

8HA

HIGH CONCENTRATION VOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name:_____ Contract:_____

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

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

Instrument ID: _____ Time Analyzed:_____

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

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (CBZ) = Chlorobenzene

UPPER LIMIT = + 100% of internal standard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

8HB

HIGH CONCENTRATION EXTRACTABLE INTERNAL STANDARD AREA SUMMARY

Lab Name:_____ Contract:_____

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

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

Instrument ID: _____ Time Analyzed:_____

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

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

UPPER LIMIT = + 100% of internal standard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

8HC

HIGH CONCENTRATION EXTRACTABLE INTERNAL STANDARD AREA 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

UPPER LIMIT = + 100%
of internal standard area.
LOWER LIMIT = - 50%
of internal standard area.

Column used to flag internal standard area values with an asterisk

8HD

HIGH CONCENTRATION ANALYTICAL SEQUENCE

Lab Name:_____ Contract:_____

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

Instrument ID: _____ GC Column ID: _____

Date(s) Analyzed: _____ _____

THE ANALYTICAL SEQUENCE OF INITIAL CALIBRATION STANDARDS, PERFORMANCE EVALUATION MIXES, INSTRUMENT BLANKS, METHOD BLANKS, SAMPLES, AND CONTROL MATRIX SPIKES IS GIVEN BELOW.

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED	%D TMZ #	%D DEC #
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						
33						
34						
35						
36						

Column used to flag values outside of QC limits (± 0.5%).

9HA

HIGH CONCENTRATION EXTRACTABLE GPC CALIBRATION

Lab Name:_____ Contract:_____

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

GPC Column ID: _____ Calibration Date: _____

COMPOUND	INITIAL CALIB. RT	CALIB. VERIF. RT	% D	QC LIMITS
Polystyrene				
Bis(2-ethylhexyl)phthalate				5%
Pentachlorophenol				
Perylene				5%

THIS GPC CALIBRATION APPLIES TO THE FOLLOWING SAMPLES, AND CMS:

	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					

COMMENTS: _____

_____

9HB

HIGH CONCENTRATION SINGLE COMPONENT PESTICIDE RETENTION TIMES

Lab Name:_____ Contract:_____

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

Instrument ID: _____ GC Column ID: _____

Date(s) Analyzed: _____ _____

COMPOUND	RT	RT WINDOW FROM	RT WINDOW TO
alpha-BHC			
beta-BHC			
delta-BHC			
gamma-BHC			
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			

Retention times of single component pesticides must be determined at least once per initial calibration on each instrument and column used for the analysis of Aroclors and Toxaphene. See the Forms instructions for the calculation of retention time windows. These data must be submitted with the data package for each group of samples analyzed under that initial calibration.

COMMENTS: _____

_____

10H

HIGH CONCENTRATION AROCLOR IDENTIFICATION SUMMARY

EPA SAMPLE NO.

Lab Name: ____________________ Contract: __________

Lab Code: ________ Case No.: ______ SAS No.: ______ SDG No.: ______

Instrument ID (1): __________ Instrument ID (2): __________

GC Column ID (1): __________ GC Column ID (2): __________

Lab Sample ID (1): __________ Lab Sample ID (2): __________

COMPOUND	PEAK	RT	RT WINDOW OF STANDARD 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						

* Denotes required peaks. Report the lower mean concentration on Form I.
If %D >25%, flag the value on Form I according to the instructions in Ex. B.

page __ of __

11H

HIGH CONCENTRATION AROCLOR DIOL CARTRIDGE CHECK

Lab Name:_____ Contract:_____

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

Diol Cartridge Lot Number: _____ Date of Analysis: _____

COMPOUND	SPIKE ADDED (ng)	SPIKE RECOVERY (ng)	% REC	QC. LIMITS
Aroclor 1254				80-110

THIS LOT OF DIOL CARTRIDGES WAS USED FOR THE FOLLOWING SAMPLES AND CMS:

	EPA SAMPLE NO.	LAB SAMPLE ID
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		

COMMENTS: _____

_____

EXHIBIT C

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

Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL)*

	Volatiles	CAS Number	Quantitation Limits (mg/Kg)
1.	Chloromethane	74-87-3	5.0
2.	Bromomethane	74-83-9	5.0
3.	Vinyl Chloride	75-01-4	5.0
4.	Chloroethane	75-00-3	5.0
5.	Methylene Chloride	75-09-2	2.5
6.	Acetone	67-64-1	5.0
7.	Carbon Disulfide	75-15-0	2.5
8.	1,1-Dichloroethene	75-35-4	2.5
9.	1,1-Dichloroethane	75-34-3	2.5
10.	1,2-Dichloroethene (total)	540-59-0	2.5
11.	Chloroform	67-66-3	2.5
12.	1,2-Dichloroethane	107-06-2	2.5
13.	2-Butanone	78-93-3	5.0
14.	1.1,1-Trichloroethane	71-55-6	2.5
15.	Carbon Tetrachloride	56-23-5	2.5
16.	Vinyl Acetate	108-05-4	5.0
17.	Bromodichloromethane	75-27-4	2.5
18.	1,2-Dichloropropane	78-87-5	2.5
19.	cis-1,3-Dichloropropene	10061-01-5	2.5
20.	Trichloroethene	79-01-6	2.5
21.	Dibromochloromethane	124-48-1	2.5
22.	1,1,2-Trichloroethane	79-00-5	2.5
23.	Benzene	71-43-2	2.5
24.	trans-1,3-Dichloropropene	10061-02-6	2.5
25.	Bromoform	75-25-2	2.5
26.	4-Methyl-2-pentanone	108-10-1	5.0
27.	2-Hexanone	591-78-6	5.0
28.	Tetrachloroethene	127-18-4	2.5
29.	1,1,2,2-Tetrachloroethane	79-34-5	2.5
30.	Toluene	108-88-3	2.5
31.	Chlorobenzene	108-90-7	2.5
32.	Ethylbenzene	100-41-4	2.5
33.	Styrene	100-42-5	2.5
34.	Xylene (Total)	1330-20-7	2.5

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

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

	Extractables	CAS Number	Quantitation Limits (mg/Kg)
35.	Phenol	108-95-2	20
36.	bis(2-Chloroethyl) ether	111-44-4	20
37.	2-Chlorophenol	95-57-8	20
38.	1,3-Dichlorobenzene	541-73-1	20
39.	1,4-Dichlorobenzene	106-46-7	20
40.	Benzyl alcohol	100-51-6	20
41.	1,2-Dichlorobenzene	95-50-1	20
42.	2-Methylphenol	95-48-7	20
43.	bis(2-Chloroisopropyl) ether	108-60-1	20
44.	4-Methylphenol	106-44-5	20
45.	N-Nitroso-di-n-dipropylamine	621-64-7	20
46.	Hexachloroethane	67-72-1	20
47.	Nitrobenzene	98-95-3	20
48.	Isophorone	78-59-1	20
49.	2-Nitrophenol	88-75-5	20
50.	2,4-Dimethylphenol	105-67-9	20
51.	Benzoic acid	65-85-0	100
52.	bis(2-Chloroethoxy) methane	111-91-1	20
53	2,4-Dichlorophenol	120-83-2	20
54.	1,2,4-Trichlorobenzene	120-82-1	20
55.	Naphthalene	91-20-3	20
56.	4-Chloroaniline	106-47-8	20
57.	Hexachlorobutadiene	87-68-3	20
58.	4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	20
59.	2-Methylnaphthalene	91-57-6	20
60.	Hexachlorocyclopentadiene	77-47-4	20
61.	2,4,6-Trichlorophenol	88-06-2	20
62.	2,4,5-Trichlorophenol	95-95-4	100
63.	2-Chloronaphthalene	91-58-7	20
64.	2-Nitroaniline	88-74-4	100
65.	Dimethylphthalate	131-11-3	20
66.	Acenaphthylene	208-96-8	20
67.	2,6-Dinitrotoluene	606-20-2	20
68.	3-Nitroaniline	99-09-2	100

(continued)

	Extractables	CAS Number	Quantitation Limits (mg/Kg)
69.	Acenaphthene	83-32-9	20
70.	2,4-Dinitrophenol	51-28-5	100
71.	4-Nitrophenol	100-02-7	100
72.	Dibenzofuran	132-64-9	20
73.	2,4-Dinitrotoluene	121-14-2	20
74.	Diethylphthalate	84-66-2	20
75.	4-Chlorophenyl-phenylether	7005-72-3	20
76.	Fluorene	86-73-7	20
77.	4-Nitroaniline	100-01-6	100
78.	4,6-Dinitro-2-methylphenol	534-52-1	100
79.	N-nitrosodiphenylamine (1)	86-30-6	20
80.	4-Bromophenyl-phenylether	101-55-3	20
81.	alpha-BHC	319-84-6	20
82.	Hexachlorobenzene	118-74-1	20
83.	beta-BHC	319-85-7	20
84.	Pentachlorophenol	87-86-5	100
85.	gamma-BHC (Lindane)	58-89-9	20
86.	Phenanthrene	85-01-8	20
87.	Anthracene	120-12-7	20
88.	delta-BHC	319-86-8	20
89.	Heptachlor	76-44-8	20
90.	Aldrin	309-00-2	20
91.	Di-n-butylphthalate	84-74-2	20
92.	Fluoranthene	206-44-0	20
93.	Heptachlor epoxide	1024-57-3	20
94.	Monochlorobiphenyl	27323-18-8	100
95.	Dichlorobiphenyl	2051-60-7	100
96.	Trichlorobiphenyl	2051-61-8	100
97.	Tetrachlorobiphenyl	2051-62-9	100
98.	Pyrene	129-00-0	20
99.	gamma-Chlordane	5103-74-2	20
100.	Endosulfan I	959-98-8	20
101.	alpha-Chlordane	5103-71-9	20
102.	4,4'-DDE	72-55-9	20
103.	Dieldrin	60-57-1	20
104.	Hexachlorobiphenyl	26601-64-9	100
105.	Pentachlorobiphenyl	25429-29-2	100

(continued)

Extractables	CAS Number	Quantitation Limits (mg/Kg)
106. Endrin	72-20-8	20
107. Endosulfan II	33213-65-9	20
108. 4,4'-DDD	72-54-8	20
109. Heptachlorobiphenyl	28655-71-2	100
110. Butylbenzylphthalate	85-68-7	20
111. Endosulfan sulfate	1031-07-8	20
112. 4,4'-DDT	50-29-3	20
113. Endrin ketone	53494-70-5	20
114. Benzo(a)anthracene	56-55-3	20
115. Methoxychlor	72-43-5	20
116. Chrysene	218-01-9	20
117. Octachlorobiphenyl	55722-26-4	200
118. 3,3'-Dichlorobenzidine	91-94-1	40
119. bis(2-Ethylhexyl)phthalate	117-81-7	20
120. Nonachlorobiphenyl	53742-07-7	200
121. Decachlorobiphenyl	2051-24-3	200
122. Di-n-octylphthalate	117-84-0	20
123. Benzo(b)fluoranthene	205-99-2	20
124. Benzo(k)fluoranthene	207-08-9	20
125. Benzo(a)pyrene	50-32-8	20
126. Indeno(1,2,3-cd)pyrene	193-39-5	20
127. Dibenz(a,h)anthracene	53-70-3	20
128. Benzo(g,h,i)perylene	191-24-2	20

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

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Aroclor-Specific/Toxaphene by GC/EC Method	CAS Number	Quantitation Limits (mg/Kg)
129. Toxaphene	8001-35-2	50
130. Aroclor 1016	12674-11-2	10
131. Aroclor 1221	11104-28-2	10
132. Aroclor 1232	11141-16-5	10
133. Aroclor 1242	53469-21-9	10
134. Aroclor 1248	12672-29-6	10
135. Aroclor 1254	11097-69-1	10
136. Aroclor 1260	11096-82-5	10

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

EXHIBIT D

ANALYTICAL PROCEDURES FOR

HIGH CONCENTRATION VOLATILE ORGANIC WASTE SAMPLES

TABLE OF CONTENTS

SECTION I

INTRODUCTION

The samples received for high concentration analysis will consist of three types of phases: solids, water immiscible liquids, and water miscible liquids. It is also possible to receive samples that have multiple phases such as soil, water and oil, in the same sample jar. Because of this possibility the samples are to be "phase separated" into their individual phases. The weight of each phase and the phase type are to be recorded on appropriate data sheets and reported with the sample.

The phase separation techniques employed will vary according to the types of sample received. Since it is impossible to know the number and types of phases that will be present in a sample, the choice of phase separation techniques is left to the discretion of the analyst. Various techniques can be employed to separate the phases. These include pipetting off liquid phases (decanting should not be done), centrifuging to remove suspended solids, use of spatulas to remove solids (wooden tongue depressors work well). Whenever possible, phase separation operations should be done with disposable glassware. This eliminates the problem of cleaning contaminated glassware. The phases should be separated into glass containers with teflon-lined screw caps. This allows for storage and handling of the waste in a safe manner. Under no circumstances are samples to be homogenized to eliminate separate phases.

Each individual phase is then taken through the procedures as a sub-sample. The results of the analyses are to be reported for each phase of the sample.

The analyical methods that follow are designed to analyze the organic compounds on the Target Compound List (TCL) (See Exhibit C). The methods are divided into the following sections: sample preparation, screening and analysis. Sample preparation covers sample storage. The analysis section contains the GC/MS analytical methods for volatile 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) High Concentration Organic Compounds

1.1 Scope and Application

These procedures are designed for the preparation of waste samples which may contain organic chemicals at a level greater than 20 mg/kg.

The extracts and sample aliquots prepared using these methods are screened by gas chromatograph/flame ionization detector (GC/FID) for volatile organic chemicals. The results of these screens will determine the dilution required for gas chromatograph/mass spectrometer (GC/MS) analysis. The analytical scheme is summarized in flowchart form (Figure D1).

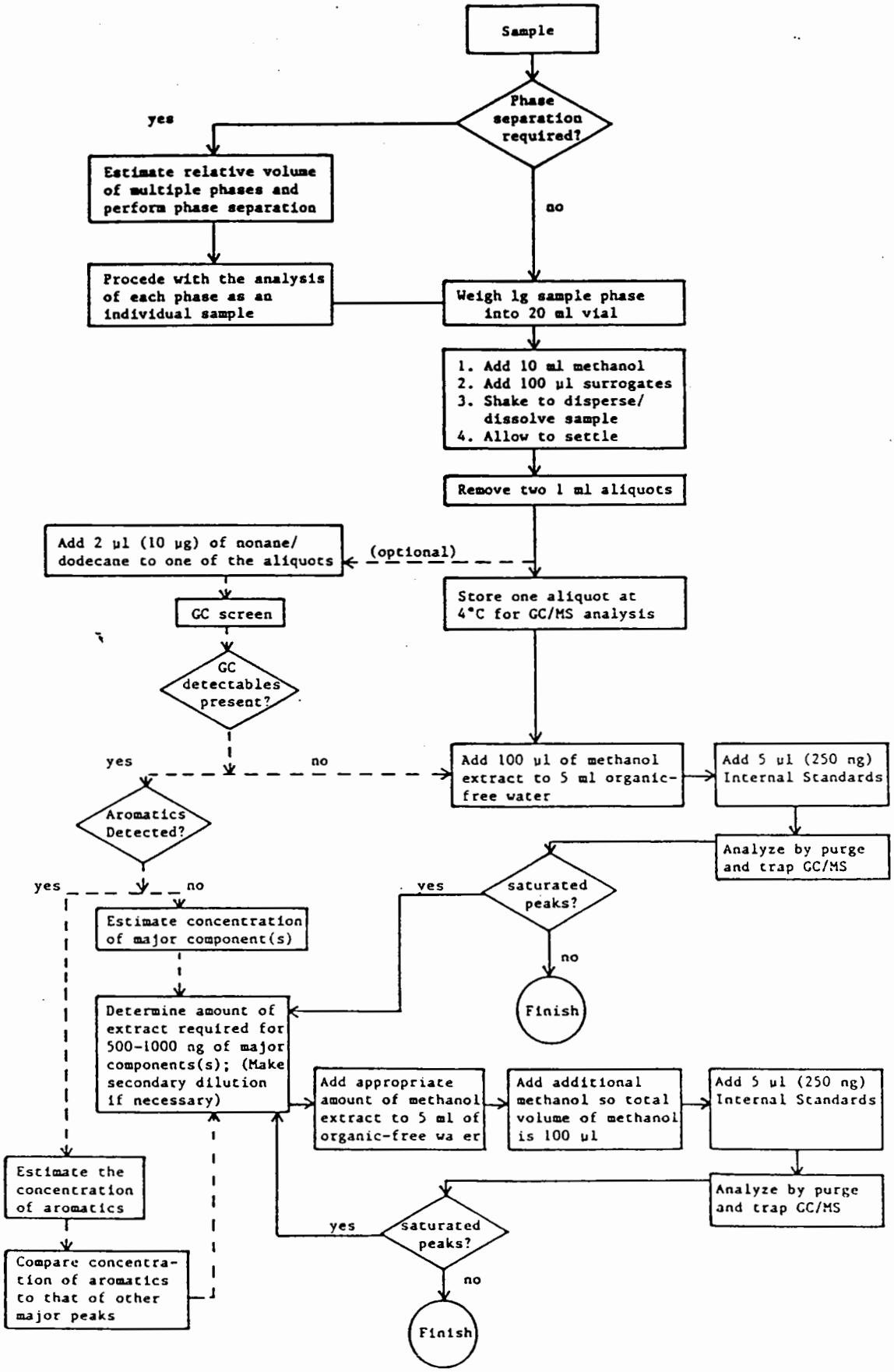

FIGURE D1 - FLOWCHART FOR ANALYSIS OF HIGH CONCENTRATION VOLATILE ORGANICS

SECTION II

SAMPLE STORAGE AND PREPARATION

PART A - SAMPLE STORAGE

Procedures for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until analysis or extraction.

PART B - HIGH LEVEL METHODS FOR SCREENING AND ANALYSIS OF VOLATILE ORGANICS

1. Summary of Methods

1.1 Samples received for high concentration analysis will consist of three types of phases: solids, water immiscible liquids, and water miscible liquids. It is possible to receive samples that will be multiple phase, such as soil, water and oil in the same sample container. Multi-phase samples are to be "phase-separated" into single phase units.

1.2 One gram aliquots of the samples are transferred to vials and extracted with methanol. The methanol extracts are screened for volatile organics by GC/FID.

1.3 If organic compounds are not detected by the screen, then a 100 uL aliquot of the methanol extract is analyzed by purge and trap GC/MS for volatile organics. If compounds are detected by the screen, the screening data are used to determine the amount of methanol extract appropriate for GC/MS analysis.

2. Limitations

2.1 The procedure is designed to allow detection limits for screening purposes as low as 0.5 - 1 mg/kg for volatile organics. If peaks are present based on the GC/FID screens, a dilution of the methanol extract prior to GC/MS analysis is required. Some samples may contain high concentrations of chemicals that interfere with the analysis of other components at lower levels; the detection limits in those cases may be significantly higher.

2.2 These extraction and preparation procedures were developed for rapid and safe handling of high concentration hazardous waste samples. The design of the methods does not stress efficient recoveries or low limits of detection for all components. Rather, the procedures were designed for moderate recovery and sufficient sensitivity of a broad spectrum of volatile organic chemicals. The results of the analyses thus may reflect only a minimum of the amount of pollutants actually present in some samples.

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 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, depending upon the nature and diversity of the samples.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling.

4. Apparatus and Equipment

4.1 Analytical balance capable of accurately weighing 0.0001 g and a top-loading balance capable of weighing 0.1 g.

4.2 Glassware

Bottles - 15 mL, screw cap, with teflon cap liner.

Volumetric flasks - Class A with ground glass stoppers.

Vials - 2 mL for GC autosampler.

5. Reagent Specifications

5.1 Reagent water is defined as water in which an interferent is not observed at the minimum detection limit of the parameters of interest.

5.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing activated carbon (Calgon Corp., Filtrasorb-300, or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

5.1.3 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 1 hour. While still hot, transfer the water to a narrow-mouth, screw-cap bottle and seal with a teflon-lined septum and cap or maintain the water under a continuous purge of inert gas.

5.2 Methanol - Pesticide residue analysis grade, or equivalent. Methanol must be demonstrated to be free from purgeable interferences.

5.3 Preparation of Spiking Standards and Analytical Standards

5.3.1 Stock standard solutions may be prepared from pure standard materials or purchased as certified (compound purity of 96% or greater) solutions. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate.

5.3.2 Great care must be taken to maintain the integrity of all standard solutions. All standard solutions should be stored at -10°C to -20°C in amber bottles with teflon liners in the

screw caps. For storage of calibration standards, bottle caps with syringe valves are recommended.

5.3.3 Place about 9.8 mL of methanol into a 10.0 mL tared, 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 0.0001 g.

5.3.4 Add the assayed reference material as described below.

5.3.4.1 Liquids - 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.

5.3.4.2 Gases - To prepare standards for any of the four halocarbons that boil below 30°C (i.e., bromomethane, chloroethane, chloromethane, and vinyl chloride), 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 heavy gas rapidly dissolves in the methanol.

5.3.5 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration from the net gain in weight.

5.3.6 Transfer the stock standard solution into a teflon-sealed, screw-cap bottle. Store with minimal headspace at -10°C to -20°C and protect from light.

5.3.7 Prepare fresh stock standards every two months for the four gases. All other standards must be replaced after six months, or sooner if comparison with check standards indicate a problem.

5.3.8 Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singularly or mixed together. (See GC/MS Calibration in Exhibit E.) Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.4 Surrogate Standard Spiking Solution - Prepare stock standard solutions for toluene-d_8, p-bromofluorobenzene (BFB), and 1,2-dichloroethane-d_4 in methanol. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 250 ug/mL in methanol.

5.5 Purgeable Organic Control Matrix Spiking Solution - Prepare in methanol a spiking solution that contains the following compounds at a concentration of 250 ug/mL: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

5.6 Screening Standards - Prepare standard mixture #1 containing benzene, toluene, ethylbenzene, and xylene in methanol. Prepare standard mixture #2 containing n-nonane and n-dodecane in methanol.

5.6.1 Stock standard solutions (1.00 mg/mL) can be prepared from pure standard materials or purchased as certified solutions.

5.6.1.1 Prepare stock standard solutions by accurately weighing approximately 0.0100 grams of pure material. Dissolve the material in methanol; dilute to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. Compound purity should be certified at 96% or greater.

5.6.1.2 Transfer the stock standard solutions into teflon-sealed, screw-cap bottles. Store with minimal headspace 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.

5.6.2 Prepare working standards of mixtures #1 and #2 at 10 ng/uL of each compound in methanol. Prepare spiking mixture of #2 at 5000 μg/mL in methanol. The spiking mixture is added to each screening extract.

5.7 Internal Standard Spiking Solution - Prepare stock standard solutions for bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5 in methanol. Prepare an internal standard spiking solution from these stock standards at a concentration of 50 ug/mL in methanol. This 50 ug/mL solution is the working standard solution.

5.8 p-bromofluorobenzene (BFB) Standard - Prepare a 25 ug/mL solution of BFB in methanol.

5.9 Calibration Standards - Prepare a stock standard solution(s) for the 34 volatile target compounds listed in Exhibit C. Prepare working standard solutions from these stock standards at a concentration of 50 ug/mL in methanol. The working standard(s) will be used to prepare calibration standards at five specified concentrations as described in Section IV, 6.3.1. Prepare fresh working standards weekly for the four gases. All other working standards must be replaced after one month.

6. Phase Separation

6.1 The samples received for high concentration analysis will consist of three phase types:

Water miscible liquid
Water immiscible liquid
Solid

It is possible to receive samples that contain multiple phases such as water, oil, and soil in the same sample jar. Because of this possibility, the samples are to be "phase separated" into individual phases.

6.2 Each individual phase is taken through the procedures as a subsample. Report analytical results for each sample phase.

6.3 Do not analyze any phase that represents less than 10% of the total sample volume.

6.4 In the following procedures, where applicable, references to "samples" explicitly mean "single phase units".

7. Sample Preparation for Volatile Organics

The high concentration method is based on extracting the sample with methanol. A portion of the extract is used for a screen on the GC/FID. From the results of the screen, an aliquot of the methanol extract is added to reagent water containing the internal standards. This is purged at ambient temperature.

7.1 Weigh 1 g of each sample phase into separate tared 15 mL vials using a top loading balance. Record the actual weight to the nearest 0.1 g.

7.2 Weigh 1.0 g of corn oil into a tared 15 mL vial for use as the control matrix spike.

7.3 Quickly add 10 mL of methanol to all vials, then add 100 uL of surrogate spiking solution (5.4). For the control matrix spike, add 10 mL of methanol, 100 uL of surrogate spiking solution, and 100 uL of matrix spiking solution (5.5).

7.4 Prepare a method blank by adding 100 uL of surrogate spiking solution to 10 mL of methanol in a 15 mL vial.

7.5 Cap all vials and shake for 2 minutes. (Note: Steps 7.1 through 7.4 must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.)

7.6 In order to enhance sample extraction with methanol, the sample may be agitated in cold water in an ultrasonic bath for 2-3 minutes.

7.7 Transfer for storage 1 mL of extract to a GC vial using a disposable pipet. Transfer an additional 1 mL of extract to a GC vial for use as a screening extract. These extracts may be stored in the dark at 4°C prior to analysis. Mark the level of liquid in the vial to check for evaporation of the extract. Add 2 uL of 5000 ug/mL #2 spiking mixture (n-nonane and n-dodecane) (5.6.2) to each screening extract. The sample is now ready for screening.

SECTION III

SCREENING OF METHANOL EXTRACTS FOR VOLATILES

1. Summary of Method

The methanol extracts of the samples are screened on a gas chromatograph/flame ionization detector (GC/FID). The results of the screen will determine at what dilution level the volatile organics extracts are to be analyzed by GC/MS.

2. Apparatus and Materials

2.1 Gas Chromatograph - A gas chromatograph suitable for on-column injection, and all required accessories including syringes, analytical columns, gases, flame ionization detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

2.2 GC Column - 3 m x 2 mm ID glass column packed with 10% SP-2100 on 100-120 mesh Supelcoport (or equivalent). The column temperature should be programmed from 55°C to 280°C at 16°C/minute and held at 280°C for 10 minutes. (Other chromatographic conditions may also provide acceptable results.)

3. Reagents

3.1 Methanol - Pesticide residue analysis grade, or equivalent. Methanol must be demonstrated to be free from purgeable interferences.

3.2 Screening Standards - Prepare standard mixture #1 containing benzene: toluene, ethylbenzene, and xylene in methanol. Prepare standard mixture #2 containing n-nonane and n-dodecane in methanol.

3.2.1 Stock standard solutions (1.00 mg/mL) can be prepared from pure standard materials or purchased as certified solutions.

3.2.1.1 Prepare stock standard solutions by accurately weighing approximately 0.0100 grams of pure material. Dissolve the material in methanol; dilute to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. Compound purity should be certified at 96% or greater.

3.2.1.2 Transfer the stock standard solutions into Teflon-sealed, screw-cap bottles. Store with minimal headspace 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.

3.2.2 Prepare working standards of mixtures #1 and #2 at 10 ng/uL of each compound in methanol.

Screening Standard #1	Screening Standard #2
Benzene	n-nonane
Toluene	n-dodecane
Ethylbenzene	
Xylene	

4. Limitations

The flame ionization detector varies considerably in sensitivity when comparing aromatics and halogenated methanes and ethanes. Halomethanes are approximately 20 times less sensitive than aromatics and haloethanes are approximately 10 times less sensitive.

5. Extract Screening

5.1 External Standard Calibration - Standardize the GC/FID each 12 hour shift for half scale response when injecting 1-5 uL of the screening standards, mix #1 (aromatics) and the mix #2 (n-nonane, n-dodecane), at the 10ng/μL concentration.

5.2 Inject the same volume of methanol sample extract as the standard mixture.

6. Interpretation of Screening Results

6.1 Compare the methanol sample extract chromatogram with the method blank and standard chromatograms.

6.1.1 If no peaks are noted, other than those also in the method blank, analyze 100 uL of the sample by GC/MS. (See Table 1)

6.1.2 If peaks are present prior to the n-dodecane, and the aromatics are distinguishable, follow Option A below to determine the dilution needed to analyze by GC/MS.

6.1.3 If peaks are present prior to the n-dodecane, but the aromatics are absent or indistinguishable, use Options B or C below. Calculate a factor using Equation D1:

Equation D1

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

See Table 1 to determine the dilution needed to analyze by GC/MS.

6.2 Following are three options for interpreting the GC/FID chromatogram.

6.2.1 Option A - Use standard mixture #1 as an external standard to calculate an approximate concentration of the aromatics in the sample. If aromatics appear to be the most concentrated materials in the sample, use the screening information to determine the proper dilution for purge and trap. Use a volume of methanol to give 500 - 1000 ng of the most concentrated aromatics. This should be the best approach; however, the aromatics may be absent or obscured by higher concentrations of other purgeables. If this is the case, Options B or C may be more suitable.

6.2.2 Option B - Use standard mixture #2 as an internal standard to calculate the factor in Equation D1. Use this factor and Table 1 to determine a dilution for purge and trap. All purgeables of interest have retention times less than n-dodecane.

6.2.3 Option C - Use standard mixture #2 as an internal standard to estimate the concentration of the major peaks. Calculate a volume of methanol that gives 500 - 1000 ng of the most concentrated constituents. Use that volume of methanol for purge and trap analysis.

6.2.4 If the screening results indicate that less than 5 uL of extract should be used for GC/MS analysis, prepare a secondary dilution in methanol and use the secondary dilution for GC/MS analysis.

6.2.5 NOTE: The screening procedure can also provide information regarding certain late-eluting compounds (e.g., naphthalene) which may complicate subsequent GC/MS analyses. Calculate the appropriate dilution factor for the concentrations exceeding the table.

TABLE 1 - DETERMINATION OF GC/MS PURGE AND TRAP DILUTION LEVEL

X Factor	Estimated Concentration Range (mg/kg)*	Volume of Methanol Extract (uL)**
0 - 5	0 - 10	100
0.5 - 10	1 - 20	50
2.5 - 50	5 - 100	10
12.5 - 250	25 - 500	100 of 1/50 dilution***

* Actual concentration ranges could be 10 to 20 times higher than this if the compounds are halogenated and the estimates are from a GC/FID.

** The volume of methanol extract added to the 5 mL of water being purged. Add additional PURE methanol to bring the total volume of methanol to 100 uL.

*** Concentrations at this and higher levels require an initial dilution of the extract in methanol before removing an aliquot for analysis by GC/MS.

SECTION IV

GC/MS ANALYSIS OF VOLATILE ORGANICS

1. Summary of Method

A portion of the methanol extract, prepared as described in the preceeding sections, 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 method blanks. Teflon tubing, thread sealants, and flow controllers should be used in the purging device.

2.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons, acetone, and methylene chloride) through the septum seal into the sample during storage and handling. Blanks prepared from methanol and carried through the holding period and the analysis protocol may serve as a check on such contamination.

2.3 Contamination by carry-over can occur whenever highly concentrated and minimally concentrated samples are sequentially analyzed. To reduce carry-over, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever a highly concentrated sample is encountered, it should 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 levels of purgeables , it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. 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 Contamination can also occur when samples contain certain late-eluting compounds (e.g., naphthalene). The screening results should provide an indication of the presence of such contamination. When contamination due to late-eluting compounds is suspected, a prolonged bakeout of the GC column and the trap is recommended.

3. Apparatus, Materials, and Equipment

3.1 Micro syringes - 10 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 Analytical balance, capable of accurately weighing 0.0001 g. and a top-loading balance capable of weighing 0.1 g.

3.5 Glassware

3.5.1 Bottles - 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. The purge and trap device consists of three separate pieces of equipment; the sample purger, the trap, and the trap heater. 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 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. The sample purger, illustrated in Figure D2, meets these design criteria. Alternate sample purge devices may be utilized provided equivalent performance is demonstrated.

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). The minimum specifications for the trap are illustrated in Figure D3.

3.6.3 The trap heater 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. The heater design, illustrated in Figure D3, meets these criteria.

3.6.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures D4 and D5.

3.7 GC/MS System

3.7.1 Gas Chromatograph - A temperature programmable gas chromatograph suitable for on-column injection, and all

required accessories including syringes, analytical columns, and gases.

3.7.2 Column - 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.

3.7.3 Mass Spectrometer - Capable of scanning from 35 to 260 amu every seven seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria in Table 2 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

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 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.

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.

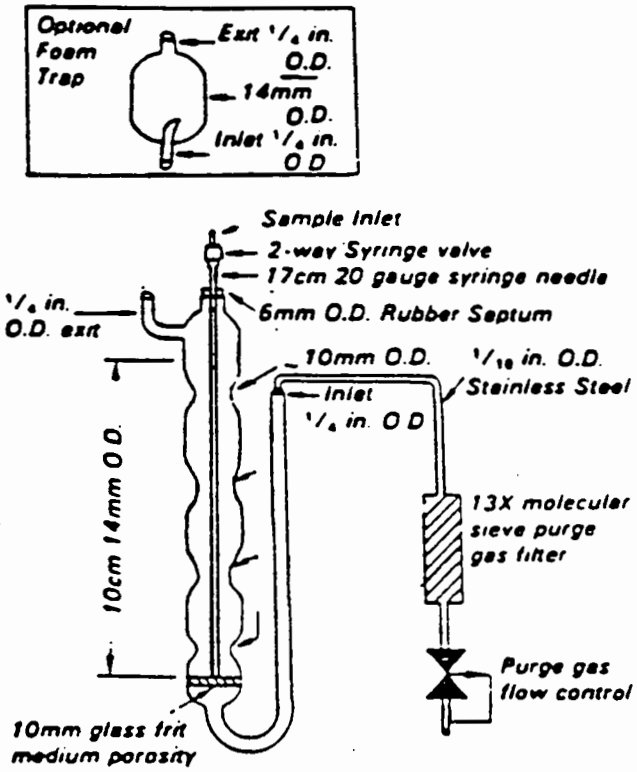

FIGURE D2 - PURGING DEVICE

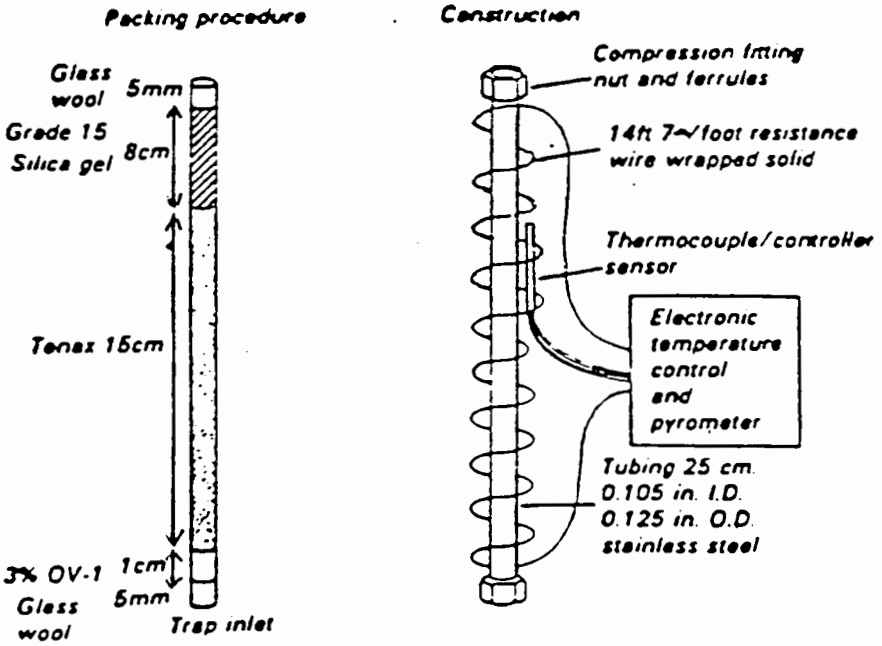

FIGURE D3 - TRAP PACKINGS AND CONSTRUCTION TO INCLUDE DESORB CAPABILITY

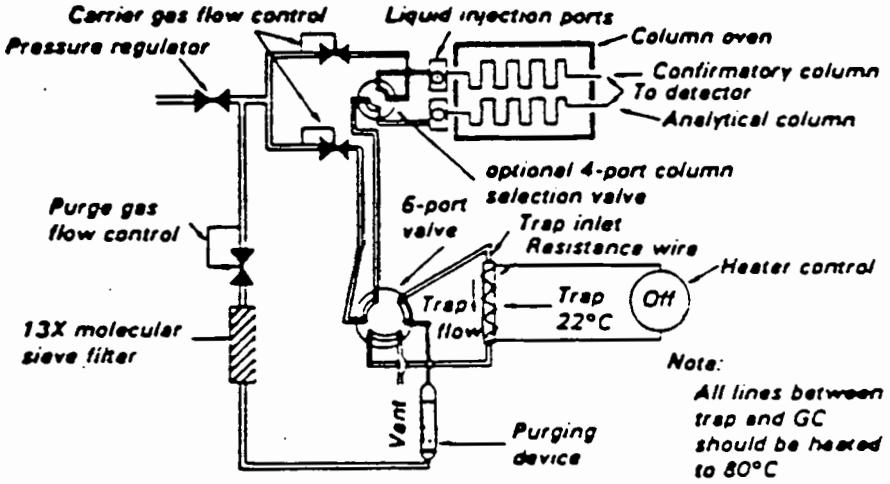

FIGURE D4 - SCHEMATIC OF PURGE AND TRAP DEVICE - PURGE MODE

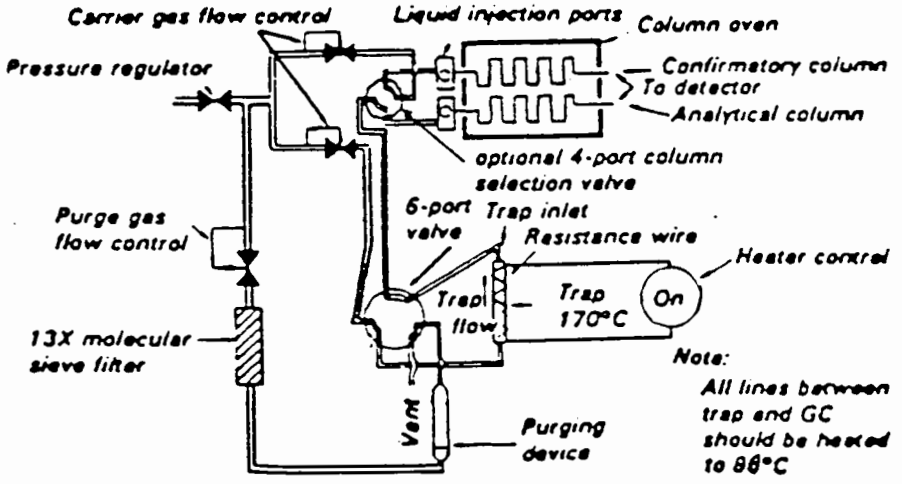

FIGURE D5 - SCHEMATIC OF PURGE AND TRAP DEVICE - DESORB MODE

4. Reagents

4.1 Reagent water is defined as water in which an interferent is not observed at the minimum detection limit of the parameters of interest.

4.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing 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 1 hour. While still hot, transfer the water to a narrow-mouth, screw-cap bottle and seal with a Teflon-lined septum and cap or maintain the water under a continuous purge of inert gas.

4.2 Methanol - Pesticide residue analysis grade, or equivalent. Methanol must be demonstrated to be free from purgeable interferences.

4.3 Preparation of Spiking Standards and Analytical Standards.

4.3.1 Stock standard solutions may be prepared from pure standard materials or purchased as certified (compound purity of 96% or greater) solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

4.3.2 Great care must be taken to maintain the integrity of all standard solutions. All standard solutions should be stored at -10°C to -20°C in amber bottles with teflon liners in the screw caps. For storage of calibration standards, bottle caps with syringe valves are recommended.

4.3.3 Place about 9.8 mL of methanol into a 10.0 mL tared, 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 0.0001 g.

4.3.4 Add the assayed reference material as described below.

4.3.4.1 Liquids - 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.

4.3.4.2 Gases - To prepare standards for any of the four halocarbons that boil below 30°C (i.e., bromomethane, chloroethane, chloromethane, and vinyl chloride), 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 heavy gas rapidly dissolves in the methanol.

4.3.5 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration from the net gain in weight.

4.3.6 Transfer the stock standard solution into a Teflon-sealed, screw-cap bottle. Store with minimal headspace at -10°C to -20°C and protect from light.

4.3.7 Prepare fresh stock standards every two months for the four gases. All other standards must be replaced after six months, or sooner if comparison with check standards indicate a problem.

4.3.8 Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singularly or mixed together. (See GC/MS Calibration in Exhibit E.) Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

4.4 Surrogate Standard Spiking Solution - Prepare stock standard solutions for toluene-d_8, p-bromofluorobenzene (BFB), and 1,2-dichloroethane-d_4 in methanol. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 250 ug/mL in methanol.

4.5 Purgeable Organic Control Matrix Spiking Solution - Prepare in methanol a spiking solution that contains the following compounds at a concentration of 250 ug/mL: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

4.6 Internal Standard Spiking Solution - Prepare stock standard solutions for bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5 in methanol. Prepare an internal standard spiking solution from these stock standards at a concentration of 50 ug/mL in methanol. This 50 ug/mL solution is the working standard solution.

4.7 P-bromofluorobenzene (BFB) Standard - Prepare a 25 ug/mL solution of BFB in methanol.

4.8 Calibration Standards - Prepare a stock standard solution(s) for the 34 target compounds listed in Exhibit C. Prepare working standard solutions from these stock standards at a concentration of 50 ug/mL

in methanol. The working standard(s) will be used to prepare calibration standards at five specified concentrations as described in 6.3.1. Prepare fresh working standards weekly for the four gases. All other working standards must be replaced after one month.

5. Instrument Operating Conditions

5.1 The following are recommended purge and trap conditions:

Purge Flow:	25-40 mL per minute.
Desorb Flow:	20-60 mL per minute.
Purge:	11 minutes with trap temperature of 26°C or less.
Desorb:	4 minutes at 180°C.
Bake:	12 - 15 minutes at 180°C.

5.2 The following are recommended operating conditions for the gas chromatograph:

Column:	Carbopack B (60/80 mesh) with 1% SP-1000 packed in a 6 foot by 2 mm ID glass column.
Carrier:	Helium - 30 mL per minute.
Temperature Program:	45°C for 3 minutes, program at 8°C per minute to 220°C and hold for 15 minutes.

5.3 Establish the following operating conditions for the mass spectrometer:

Electron Energy:	70 Volts (nominal)
Mass Range:	35 - 260
Scan Time:	To give at least 5 scans per peak, but not to exceed 3 seconds per scan.

6. Calibration

6.1 Assemble a purge and trap device that meets the specification in paragraph 3.6. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180°C with the column at 220°C.

6.2 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 paragraph 5.2. Calibrate the purge and trap-GC/MS system using the internal standard technique described below.

6.3 Internal Standard Calibration Procedure. The three internal standards are: bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5.

6.3.1 From working standards in methanol, prepare calibration standards at the following five concentration levels for each target parameter: 20, 50, 100, 150, and 200 ug/L in reagent water (corresponding to 10, 25, 50, 75, and 100 mg/kg in a 1 g sample). Add additional methanol so that the total amount of methanol is 100 uL per 5 mL of aqueous standards. Aqueous standards may be stored up to 24 hours, if held in sealed vials with zero headspace. If not so stored, they must be discarded after an hour unless they are set up to be purged by an autosampler. When using an autosampler, the standards may be kept up to 12 hours in purge tubes connected via the autosampler to the purge and trap device.

6.3.2 Add 5 uL of the 50 ug/mL internal standard spiking solution (4.6) to each 5 mL standard, blank, and sample.

6.3.3 After the ion abundance criteria for BFB (Table 2) are met, analyze each calibration standard (6.1.3). Tabulate the area response of the characteristic ions against the concentration for each compound and internal standard. Calculate relative response factors (RRF) for each compound using Equation D2. The characteristic ions and internal standards for the volatile compounds are shown in Table 3.

TABLE 2 - BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of mass 95
75	30.0 - 60.0 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95
173	Less than 2.00 percent of mass 95
174	Greater than 50.0 percent of mass 95
175	5.0 - 9.0 percent of mass 174
176	Greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

NOTE 1: BFB criteria MUST be met before any samples, blanks, or standards are analyzed. Any samples analyzed when tuning criteria have not been met will require reanalysis at no additional cost to the Agency.

NOTE 2: Whenever the laboratory takes corrective action which may change or affect the tuning criteria for BFB (e.g., ion source cleaning or repair, etc.), the tune must be verified irrespective of the 12-hour tuning requirements.

NOTE 3: The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of compliant tune. The time period ends after twelve (12) hours has elapsed according to the system clock.

$$\text{Equation D2} \quad RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard from Table 3.

C_{is} = Concentration of the internal standard.

C_x = Concentration of the compound to be measured.

TABLE 3 - CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)	Internal Standard
SURROGATE STANDARDS			
4-Bromofluorobenzene	95	174, 176	3
1,2-Dichloroethane-d_4	65	102	1
Toluene-d_8	98	70, 100	3
INTERNAL STANDARDS			
Bromochloromethane (1)	128	49, 130, 51	1
1,4-Difluorobenzene (2)	114	63, 88	2
Chlorobenzene-d_5 (3)	117	82, 119	3
TARGET COMPOUNDS			
Chloromethane	50	52	1
Bromomethane	94	96	1
Vinyl Chloride	62	64	1
Chloroethane	64	66	1
Methylene Chloride	84	49, 51, 86	1
Acetone	43	58	1
Carbon Disulfide	76	78	1
1,1-Dichloroethene	96	61, 98	1
1,1-Dichloroethane	63	65, 83, 85, 98, 100	1
1,2-Dichloroethene	96	61, 98	1
Chloroform	83	85	1
1,2-Dichloroethane	62	64, 100, 98	1
2-Butanone	72	57	1
1,1,1-Trichloroethane	97	99, 117, 119	2
Carbon Tetrachloride	117	119, 121	2
Vinyl Acetate	43	86	2
Bromodichloromethane	83	85, 129	2
1,2-Dichloropropane	63	65, 114	2
trans-1,3-Dichloropropene	75	77	2
Trichloroethene	130	95, 97, 132	2
Dibromochloromethane	129	208, 206	2
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134	2
Benzene	78	----	2
cis-1,3-Dichloropropene	75	77	2
Bromoform	173	171, 175, 250, 252, 254, 256	2
2-Hexanone	43	58, 57, 100	3
4-Methyl-2-Pentanone	43	58, 100	3
Tetrachloroethene	164	129, 131, 166	3
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166	3
Toluene	92	91	3
Chlorobenzene	112	114	3
Ethylbenzene	106	91	3
Styrene	104	78, 103	3
Total Xylenes	106	91	3

6.3.4 The average relative response factor ($\bar{RRF}$) must be calculated for all compounds. A system performance check must be made before this calibration curve is used. The following five System Performance Check Compounds (SPCC) are checked for a minimum average response factor: chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum average response factor for bromoform is 0.250; the minimum average response factor for all other SPCCs is 0.300.

The following five Calibration Check Compounds (CCC) are used to evaluate the curve: vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, and ethylbenzene. Calculate the Percent Relative Standard Deviation (%RSD) (Exhibit E, Section III, Equation 2.2) of RRF values over the working range of the curve. A maximum %RSD of 30.0% for each CCC must be met before the curve is valid.

6.3.5 Once each 12 hours (beginning with the injection of the tuning compound), after demonstrating the key ion abundance criteria for BFB (Table 2), a continuing calibration check must be performed. Analyze the 50 ug/L standard, using the same conditions as those used for the initial calibration.

6.3.5.1 Calculate the response factors as in 6.3.3. Check the minimum response factors for the system performance check compounds (SPCC). The minimum response factor for bromoform is 0.250; the minimum response factor for all other SPCCs is 0.300.

6.3.5.2 Calculate the response factors for the calibration check compounds (CCC) and compare against those determined in the initial calibration. Calculate the percent difference (%D) (Exhibit E, Section III, Equation 2.3). The maximum %D for CCCs is 25.0%.

6.3.5.3 After verifying that the continuing calibration meets the criteria for SPCCs and CCCs, calculate the relative response factors for the remaining compounds.

7. Sample Analysis

An aliquot of the methanol extract is added to reagent water containing the internal standards. This is purged at ambient temperature and analyzed by GC/MS.

7.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.2 Adjust the purge gas (helium) flow rate to 25-40 (±3) mL per minute. Variations from this flow rate may be necessary to achieve better

purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

7.3 After achieving the key ion abundance criteria for BFB (Table 2), perform the daily system calibration, as described in 6.3.5.

7.4 The purgeable organics screening procedure will show the approximate concentrations of major sample components. If a dilution of the sample is indicated, this dilution shall be made just prior to GC/MS analysis of the sample.

7.5 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 5 uL of the internal standard solution. Add the volume of methanol extract determined from the screening procedure and a volume of pure methanol to total 100 uL (excluding methanol in the standards).

7.6 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.

7.7 Proceed with the analysis as outlined. Analyze all method blanks on the same instrument as the samples.

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

7.9 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 4 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.

7.10 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 carry-over of pollutant compounds. Baking out the purging chamber to remove traces of possible contamination is highly recommended.

7.11 After desorbing the sample for 4 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 7 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.

7.12 If the initial analysis of a sample or a dilution of a sample has concentration of target compounds that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until an acceptable blank can be analyzed that is free of interferences. (Exhibit E, Part III)

7.13 All dilutions must be made such that major constituents are not saturated but give a response greater than that of the nearest internal standard.

7.14 Internal standard responses and retention times in all samples and standards must be evaluated immediately after or during 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. If the extracted ion current profile (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. Internal Standard Areas and Retention Times are reported on Form VIII HCV, as described in Exhibit B.

8. Qualitative Analysis

8.1 Criteria for Verification of Target Compound Identifications

8.1.1 The target compounds listed in Exhibit C shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound.

8.1.2 Two criteria must be satisfied to verify the identifications:

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

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

8.1.3 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units (or ±5 seconds) of the RRT of the standard component. For reference, the calibration 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.

8.1.4 For comparison of standard and sample component mass spectra, mass spectra obtained on the contractor's GC/ MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the contractor's GC/MS meets the daily tuning requirements for BFB (Table 2). These standard spectra may be obtained from the daily calibration standard or from the user-created mass spectral library. The requirements for qualitative verification by comparison of mass spectra are as follows:

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

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

8.1.4.3 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. Both raw and background subtracted spectra must be evaluated. 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."

8.1.5 If a compound cannot be verified by all of the criteria in 8.1.4.3, 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 9.

8.2 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the most recent available version of the NBS Mass Spectral Library shall be used. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

8.2.1 Up to 10 nonsurrogate organic compounds of greatest apparent concentration not listed in Exhibit C shall be tentatively identified via a forward search of the NBS Mass Spectral 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.

8.2.2 Guidelines for making tentative identification are as follows:

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

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

8.2.2.3 The molecular ion of the reference spectrum must be present in sample spectrum.

8.2.2.4 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.

8.2.2.5 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.

8.2.3 If in the opinion of the mass 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 aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

9. Quantitative Analysis

9.1 Target compounds identified shall be quantified by the internal standard method. The internal standard used shall be the one assigned in Table 2.1, Volatile Internal Standards with Corresponding Target Analytes Assigned for Quantitation (Exhibit E). The EICP area of the characteristic ions of analytes listed in Table 3 is used. The relative response factor (RRF) from the daily calibration standard analysis is used to calculate the concentration in the sample. Use the relative response factor as determined in paragraph 6.3.3 and calculate the analyte concentration in the sample using Equation D3.

Xylenes (o, m, and p isomers) are to be reported as total xylenes. Since o- and p-xylene overlap, the two xylene peaks (o/p and m) must be added together to give the total concentration of all xylene isomers.

Equation D3

$$\text{Sample Concentration (mg/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard from Table 3.

I_s = Amount of internal standard added in nanograms (ng).

V_t = Volume of total extract (uL).

V_i = Volume of extract added (uL) for purging.

W_s = Weight of sample extracted (mg).

9.2 An estimated concentration for non-target compounds tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences must be used.

Equation D3 is used to calculate concentrations. 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 value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

9.3 Use Equation D4 to calculate surrogate standard recoveries on all samples, blanks and spikes. Determine if recovery is within specified limits.

Equation D4

$$\text{Percent Surrogate Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample

9.4 If recovery is not within specified limits, the following is required:

9.4.1 Check to be sure there are no errors in calculations, surrogate solutions, and internal standards. Also, check instrument performance.

9.4.2 Recalculate the sample data if any of the above checks reveal a problem.

9.4.3 If surrogates cannot be detected due to dilution factors, this requirement need not be met. (Report the value as DL.)

NOTE: Surrogate recovery limits are MANDATORY for method blanks. However, surrogate recoveries for high concentration samples and control matrix spikes are ADVISORY at this time. Reanalysis of samples and control matrix spikes is not required if surrogate recoveries are outside of contractually specified limits. However, the analyst must verify that the deviation is not a result of laboratory error.

EXHIBIT D

ANALYTICAL PROCEDURES FOR HIGH CONCENTRATION EXTRACTABLE ORGANIC WASTE SAMPLES

Rev. 9/88

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SECTION I

INTRODUCTION

The samples received for high concentration analysis will consist of three types of phases: solids, water immiscible liquids, and water miscible liquids. It is also possible to receive samples that will be multiple phase such as soil, water and oil in the same sample jar. Because of this possibility, the samples are to be "phase separated" into their individual phases. The weight of the aliquot of each phase analyzed, and the phase type are recorded on appropriate data sheets and reported with the sample data.

The phase separation techniques employed will vary according to the types of samples received. Since it is impossible to know the number and types of phases that will be present in a sample, the choice of phase separation techniques is left to the discretion of the analyst. Various techniques can be employed to separate the phases. These include pipetting off liquid phases (decanting should not be done), centrifuging to remove suspended solids, use of spatulas to remove solids (wooden tongue depressors work well). All operations should be done with disposable phthalate-free labware. This eliminates the problem of cleaning contaminated glassware. The separate phases should be stored into glass containers with teflon-lined screw caps. This allows for storage and handling of the waste in a safe manner. Under no circumstances are samples to be homogenized to eliminate separate phases.

Each individual phase is treated as a subsample, and is taken through the separate GC/MS and GC/ECD procedures as a sub-sample. The results of each of the analyses are to be reported for each phase of the sample.

The analytical methods that follow are designed to analyze the organic compounds on the Target Compound List (TCL) (See Exhibit C). The methods are divided into the following sections: sample preparation, screening, and analysis. Sample preparation covers sample storage.

1. Method For The Determination of Extractable Organic Compounds

1.1 Scope and Application

These procedures are designed for the preparation of waste samples which may contain organic chemicals at a level greater than 20 mg/kg.

1.1.1 The extracts prepared using these methods are screened by GC/FID for Base/Neutrals, Acids and Pesticides/PCBs target compounds. The results of these screens will determine whether sufficient quantities of pollutants are present to warrant analysis by the high or low/medium protocol.

SECTION II

SAMPLE STORAGE AND PREPARATION

PART A - SAMPLE STORAGE

1. Procedures for Sample Storage

1.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until extraction and analysis.

1.2 After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C (±2°C) for the periods specified in the contract schedule.

PART B - SAMPLE PREPARATION FOR EXTRACTABLES

1. Summary of Method

Approximately 1.0 g portions of the single phase unit are transferred to vials and extracted with methylene chloride and are cleaned-up using GPC. The methylene chloride extract, prepared using Gel Permeation Chromatography may be prepared in methylene chloride or a 1:1 mixture of methylene chloride/butyl chloride, at the option of the laboratory, prior to GPC cleanup. Following GPC cleanup, a 1 mL aliquot of the collected fraction is screened for target compounds by GC/FID (See Section 9). If organic compounds are detected by the high level screen, the methylene chloride extract is analyzed by GC/MS for the extractable target compounds listed in Exhibit C. If organic compounds are not detected by the high level screen, contact SMO immediately.

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 must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the samples.

3. Limitations

3.1 The extraction procedure is designed to allow detection limits for screening purposes as low as 20 mg/kg for Base/Neutrals, Acids, chlorinated pesticides and individual PCB congeners. For analysis purposes, the detection limits for Base/Neutrals, Acids, chlorinated pesticides and individual PCB congeners range from 20-200 mg/Kg (see Ex. C). The pesticides and PCBs tend to have low responses when using a GC/FID detector. Peaks that are present where pesticides and PCBs may elute must be viewed with extra caution; the concentration may be higher than the response would indicate. If peaks are present in the GC/FID screens, high level analysis by GC/MS is required. Some samples may contain high concentrations of chemicals that interfere with the analysis of other components at lower levels.

3.2 These extraction and preparation procedures were developed for rapid and safe handling of high concentration hazardous waste samples. The design of the methods does not stress efficient recoveries or low limits of detection 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 the sample.

4. Reagents

4.1 Sodium Sulfate - Powdered anhydrous and reagent grade, heated at 400°C for four hours, cooled in a desiccator, and stored in a glass bottle. Baker anhydrous powder, catalog number 73898 or equivalent.

4.2 Methylene chloride - Pesticide residue analysis grade, or equivalent.

4.3 Methanol - Pesticide residue analysis grade, or equivalent.

4.4 Toluene - Pesticide residue analysis grade, or equivalent.

4.5 Benzene - Pesticide residue analysis grade, or equivalent.

4.6 Butyl chloride (1-chlorobutanol) - Analytical reagent grade, or equivalent (optional).

4.7 Base/Neutral and Acid Surrogate Standard Spiking Solution

Prepare a solution in methanol containing the following compounds at a concentration of 1000 ug/mL for base/neutral surrogates, and 2000 ug/mL for the acid surrogate standards:

Base/Neutrals	Acids
nitrobenzene-d_5	phenol-d_5
p-terphenyl-d_{14}	2,4,6-tribromophenol
2-fluorobiphenyl	2-fluorophenol

4.8 Base/Neutral and Acid Control Matrix Spiking Solution.

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 1000 ug/mL for base/neutrals and 2000 ug/mL for acids:

Base/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	

4.9 Pesticide/PCB Control Matrix Spiking Solution.

Prepare a spiking solution in methanol that contains the following pesticides in the concentrations specified:

Pesticide	ug/mL
heptachlor	1,000
dieldrin	1,000

4.10 Corn oil.

4.11 Purified solid matrix, supplied by EMSL/LV upon availability.

5. Apparatus and Materials

5.1 Glass vials, at least 20 mL, with screw cap and teflon or aluminum foil liner.

5.2 Spatula - Stainless steel or teflon.

5.3 Balance capable of weighing 100 g to the nearest 0.01 g.

5.4 Vials and caps, 2 mL for GC auto sampler.

5.5 Disposable Pasteur pipets, packed with glass wool rinsed with methylene chloride.

5.6 15-mL.concentrator tubes.

5.7 Ultrasonic cell disruptor, Heat Systems Ultrasonics, Inc., Model W-385 SONICATOR (475 Watt with pulsing capability, No. 200 1/2 inch tapped disruption horn, 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.

5.8 Sonabox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.

5.9 Test tube rack.

5.10 Pyrex glass wool.

5.11 pH paper range 1-14. (Fischer Part No. A979 wide range test ribbons or equivalent).

6. Sample Preparation.

6.1 Transfer approximately 1 g of each phase (record weight to the nearest 0.1 g) of the sample to separate 20 mL vials. Wipe the mouth of the

vial with a tissue before weighing to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross contamination.

6.1.1 Prepare a 1.0 g control matrix consisting of corn oil.

6.1.2 Prepare a 1.0 g method blank consisting of purified solid matrix. (May be available through EMSL/LV).

6.1.3 Determine pH of sample with pH paper. (Add 1 mL of water to 1 g of soil or water immiscible liquid and shake. Decant water portion and measure pH of water with pH paper.)

6.2 Add 100 uL surrogate spiking solution to each sample, blank and the control matrix.

6.3 Add 100 uL of each control matrix standard spiking solution to the control matrix.

6.4 Add 2.0 g of powdered anhydrous sodium sulfate to sample.

6.5 Immediately add 10 mL of methylene chloride to the sample.

6.6 Disrupt the sample by ultrasonic probe for 2 minutes at 100 watts power.

6.7 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect a minimum of 8.0 mL of the extract in a concentrator tube.

6.8 Proceed to extract clean-up by GPC, Section 7.

7. Apparatus and Materials for Gel Permeation Chromatography (GPC) Clean Up.

7.1 Beakers, 400 mL.

7.2 Kuderna-Danish (K-D) apparatus.

7.2.1 Concentrator tube - 10 mL, graduated (Kontes K-570040-1029 or equivalent).

7.2.2 Evaporative flask - 500 mL (Kontes K-570001-0500 or equivalent).

7.2.3 Snyder column - three-ball macro (Kontes K-503000-0121 or equivalent).

7.2.4 Snyder column - two ball micro (Kontes K-569001-0219 or equivalent).

7.3 Silicon carbide boiling chips - approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

7.4 Water bath - heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

7.5 Top loading balance, capable of accurately weighing 0.01 g.

7.6 Balance - Analytical, capable of accurately weighing 0.0001 g.

7.7 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.

7.8 0.45 micron teflon membrane filter, stainless steel and/or teflon filter holder with a 13 to 25 diameter (Millipore, Schleicher and Schuell or equivalent).

7.9 Syringes, 10 mL glass with luer lock tip.

7.10 Gel permeation chromatography cleanup device. (Automated system.)

7.10.1 Gel permeation chromatograph (GPC) Analytical Biochemical Labs, Inc., Columbia, MO, GPC Autoprep 1002 or equivalent.

7.10.2 25 mm ID X 700 mm glass column.

7.10.3 Optional 5 cm guard column (Supelco 5-8319 or equivalent) with appropriate fittings to connect to the inlet side of the analytical column.

7.10.4 70 g 200-400 mesh Bio Beads (S-X3), Bio Rad Laboratories, Richmond, CA Catalog 152-2750 or equivalent (an additional 5 g of Bio Beads is required if the optional guard column is employed).

7.10.5 Fixed wavelength ultraviolet detector (254 nm) with a semi-prep flow-through cell.

7.10.6 Strip chart recorder.

8. Reagents

8.1 Methylene chloride, pesticide residue analysis grade, or equivalent.

8.1.1 Some brands of methylene chloride may contain unacceptably high levels of acid (HCl). Check the pH by shaking equal portions of methylene chloride and water, then check the pH of the water layer.

If the pH of the methylene chloride is less than or equal to 5, filter the solvent through a 2 in. X 15 in. glass column containing activated basic alumina. This column should be sufficient for processing approximately 20-30 liters of solvent. Alternatively a different source of methylene chloride should be found.

8.2 Cyclohexane, pesticide residue analysis grade, or equivalent.

8.3 GPC solution

8.3.1 Calibration solution prepared in methylene chloride containing the following analytes (in order of elution):

	mg/mL	
polystyrene (MW 300,000)	2.8	(Optional)
corn oil	60.0	
bis(2-ethylhexyl)phthalate	3.0	
pentachlorophenol	0.3	
perylene	0.02	

8.3.2 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. Allow the calibration solution to stand at room temperature until the corn oil dissolves.) Replace the calibration standard solution as necessary or a minimum of every 3 months.

9. Extract Cleanup

The GC screen may be performed before the GPC column cleanup. The results from the screen will indicate how much sample should be loaded onto the GPC column so as not to contaminate the column and, at the same time, have enough extract for GC/MS analysis. This will preserve the life of the GPC column since these columns are easily contaminated by high concentration samples.

9.1 GPC Setup and Calibration

9.1.1 Column Preparation

9.1.1.1 Weight 70.0 g (75.0 g if using optional guard column) of Bio Beads (S-X3) into a 400 mL beaker.

9.1.1.2 Add an excess quantity of a 1:3 (v/v mixture) of methylene chloride/cyclohexane to ensure that the beads will be completely submerged during the swelling process. Cover the beaker to prevent solvent evaporation and allow the beads to swell for a minimum of 16 hours.

9.1.1.3 Prepare a slurry of approximately 50% swelled beads and 50% solvent. 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 to the column end as possible (approximately 2.5 cm from the end).

9.1.1.4 Ensure that the outlet stopcock is closed. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing. Swirl the bead/solvent slurry to get a homogeneous mixture, and quickly and consistently pour the swelled slurry into the glass column. Never allow the beads to settle completely while pouring the column. A glass rod can be used to facilitate pouring the mixture down the side of the column and to help minimize bubble formation. Use additional solvent to rinse the beaker and transfer all of the beads to the column.

9.1.1.5 When the transfer solvent has drained into the column bed, the inlet bed support plunger can be used to temporarily compress the gel bed. Do not tighten the column/plunger seal if beads are caught between it and the glass column. Remove the plunger and rinse it and the sides of the glass column with minimal solvent. A clean paper towel can be used to remove any remaining beads.

9.1.1.6 Insert the inlet bed support bed plunger, compress the gel bed approximately 1 cm and tighten.

9.1.1.7 Pack the optional 5 cm guard column with approximately 2-3 g of the remaining pre-swelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

9.1.1.8 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. Pump methylene chloride through the column at a rate of 5 mL/min for 1 hour. While the solvent in the column is being changed from methylene chloride/cyclohexane to methylene chloride only, the gel may pack down or undergo additional swelling, which will change the pressure.

9.1.1.9 After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. While still pumping methylene chloride through the column, adjust the inlet bed support plunger until approximately 6-10 psi backpressure is achieved.

9.1.1.10 When the GPC column is not used for several days, evaporation of methylene chloride may cause column drying, which can cause stratification or channeling in the gel. To prevent this drying, connect the column outlet line to the column inlet. This will

allow a continuous recycling of the solvent when the system is not in use. 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 same as when the column was new.

9.1.2 Calibration of the GPC column

9.1.2.1 Using a 10 mL syringe, load sample loop #1 with calibration solution (8.3.1). 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.

9.1.2.2 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 similar to Figure 1 that meets the following requirements. Differences between manufacturer's cell volumes 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.

UV Trace Requirements:

- Peaks must be observed for all compounds in the calibration solution.
- Perylene peaks must not be saturated and must exhibit baseline resolution.
- Corn oil and phthalate peaks must exhibit baseline resolution.

9.1.2.3 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 (approximately 30 minutes) and after the elution of the corn oil (approximately 20 minutes). Stop eluate collection shortly after the elution of perylene (approximately 50 minutes). Collection should be stopped before sulfur would elute, if it were present (approximately 55 minutes). Each laboratory is required to establish its specific time sequences. The times provided are for <u>general guidance only</u>.

NOTE: The collect and dump times must be adjusted to compensate for the difference in volume of the lines between the UV detector cell and the collection flask.

9.1.2.6 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, as described above. Once the flow rate is within the range of 4.5-5.5 mL/min, record the column pressure and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored.

9.1.2.7 Reinject the calibration solution after appropriate collect and dump cycles have been set and the solvent flow and column pressure have been established.

9.1.2.7.1 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.

9.1.2.7.2 The retention times for bis(2-ethylhexyl)phthalate and perylene must not vary more than plus or minus 5% between calibrations. If the retention time shift is greater than 5%, take corrective action.

9.2 GPC Extract Cleanup

9.2.1 Studies have demonstrated that the recovery of certain aromatic compounds is improved when samples are introduced into the GPC in a 1:1 solvent mixture of methylene chloride/butyl chloride. If it is used, reduce the 8.0 mL sample volume to 3.5-4.0 mL under a stream of dry nitrogen (Paragraph 8.4.3). Reconstitute to the 8.0 mL volume with butyl chloride. Thoroughly mix the sample before proceeding.

9.2.2 Pre-filter each sample extract through a 0.45 micron PTFE filter (Millipore, Schleicher and Schuell or equivalent). Samples that contain suspended particles must be centrifuged prior to filtration. Clean the filter holder assembly between samples.

9.2.3 Using a 10 mL syringe, load the nominal 5 mL sample loop.

CAUTION: 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.

Attach the syringe to the turn lock on the injection port. Use firm, continuous pressure to push the sample onto the loop. If the sample is difficult to load, some part of the system may be blocked. Take appropriate corrective action.

9.2.4 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.

9.2.5 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.

9.2.6 Column overloading.

9.2.6.1 Column overloading can occur when too much material is loaded in a sample loop.

9.2.6.1.1 For highly contaminated samples, dilute the extract and process in more than one sample loop. An example dilution procedure is to mix 10 mL of sample extract with 10 mL of methylene chloride or 1:1 butyl chloride/methylene chloride, shake well to thoroughly mix, and load into two sample loops.

9.2.6.1.2 After GPC cleanup, combine the collected fractions and treat as a single sample. Therefore, no additional dilution factor will be required when a sample extract is diluted and divided for GPC cleanup.

9.2.7 After loading all the sample loops, index the GPC to the 00 position, switch to the "run" mode and start the automated sequence.

9.2.8 Process each sample using the collect and dump cycle times established in 9.1.2. Process calibration standards, control matrix (GPC blank), samples and method blanks in the following order:

Loop 1 Calibration Standard
Loop 2 Control Matrix (corn oil)
Loop 3-22 Samples
Loop 23 Calibration Standard (Verification)

NOTE: GPC cleanup sequence must end with a calibration standard verification regardless of the number of samples processed.

9.2.9 Collect each sample in a 250 mL Erlenmeyer flask, covered with aluminum foil to reduce solvent evaporation. Monitor sample volumes collected. Do not concentrate the processed GPC extract at this point. Changes in sample volumes collected may indicate one or more of the following problems:

9.2.9.1 Change in solvent flow rate, caused by channeling in the column or changes in column pressure.

9.2.9.2 Increase in column operating pressure due to the absorbtion of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

9.2.9.3 Leaks in the system or significant variances in room temperature.

9.2.10 Evaluation of calibration standards.

9.2.10.1 Evaluate the retention times for bis(2-ethylhexyl)phthalate and perylene in each daily calibration standard UV trace. The retention time for either compound cannot exceed a 5% retention time shift when compared to the retention time established in the initial calibration (loop 1).

9.2.10.2 Corrective action must be taken before sample cleanup can proceed. All samples processed prior to an unacceptable calibration verification must be identified in the case narrative.

9.2.10.3 Acceptable GPC performance is demonstrated by the successful analysis of the calibration standard solution. Corrective actions may include those listed in 9.1.1 and 9.1.2 above or may require repacking and recalibration of the column.

9.3 Transfer a 1.0 mL aliquot of the processed extract to a GC vial. Do not discard the remaining extract until the GC/FID screen has been performed. Proceed to GC/FID screen, Section III.

9.3.1 For the control matrix sample and method blank, concentrate the extract (9.4).

9.3.2 Store all extracts at 4°C in the dark in teflon-sealed containers until all analyses are performed.

9.4 If no sample peaks are detected or all are less than 10% full scale deflection during the GC/FID screen, the sample must be concentrated. The control matrix samples and the method blanks are also concentrated.

9.4.1 Transfer the extract (9.2.9) 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 and pesticide compounds listed in Exhibit C.

9.4.2 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 (80° to 90°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, and make up to 10 mL volume with methylene chloride.

9.4.3 Nitrogen blowdown technique (taken from ASTM Method D 3086).

The following method must be used for final concentration of the extracts. Place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume to just below 1 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, and the final volume brought to 1.0 mL with methylene chloride. During evaporation, the tube solvent level must be just above the water level of the bath. The extract must never be allowed to become dry.

9.5 Store all extracts at 4°C in the dark in teflon-sealed containers until all analyses are performed.

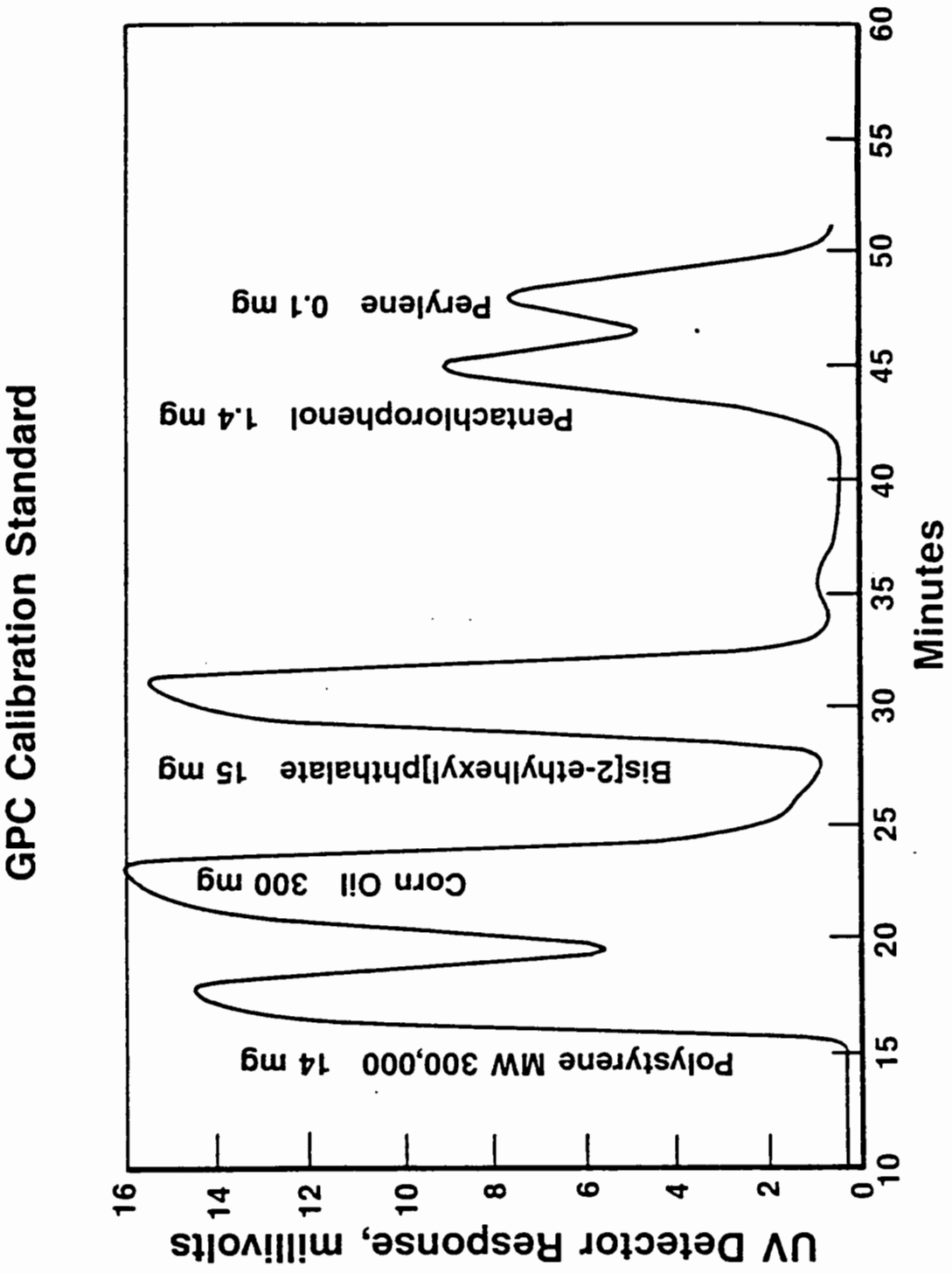

Figure 1. UV Detector Trace Produced by GPC Calibration Solution.

SECTION III

GC/FID SCREENING FOR EXTRACTABLE TARGET COMPOUNDS

1. Summary of Method

The solvent extracts of the single phase units are screened on a gas chromatograph/flame ionization detector (GC/FID) using a fused silica capillary column (FSCC). The results of the screen will determine the volume of extract taken for GC/MS analysis.

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 splitless injection using capillary columns.

2.1.1 Above GC equipped with a 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 stock standard solution containing phenol, phenanthrene, and di-n-octylphthalate in methylene chloride.

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 96% 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.

3.2.1.2 Transfer the stock standard solutions into teflon-sealed screw-cap bottles. Stored 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.

3.2.1.3 Prepare a working standard of the GC calibration standard 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 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 Standardized for 50% full scale response for 50 ng of phenanthrene.

4.1.2 Adequately separates phenol from the solvent front.

4.1.3 Minimum of 25% full scale response for 50 ng of di-n-octylphthalate.

5. GC/FID Screening

5.1 Suggested GC operating conditions:

Initial Column Temperature Hold - 50°C for 4 minutes.

Column Temperature Program - 50° to 280°C at 8 degrees per minute.

Final Column Temperature Hold -280°C for 8 minutes.

Injector - Grob-type; splitless.

Sample Volume - 1 uL to 2 uL.

Carrier Gas - Helium at 30 cm per sec.

5.2 Inject the GC calibration standard and ensure the criteria specified in 4.1 are met before injecting samples. Estimate the response for 10 ng of phenanthrene.

5.3 Inject the extracts of all single phase units to be screened, including blanks (Section II, paragraphs 9.3 and 9.4).

6. Interpretation of Chromatograms

6.1 If no sample peaks from the extract are detected or all are less than 10% full scale deflection, the sample must be concentrated as per Section II (9.4), rescreened, and analyzed by GC/MS. If no peaks are detected or all are less than 10% full scale deflection after concentration, contact the Sample Management Office for instructions.

6.2 If peaks are detected at greater than 10% deflection and less than or equal to 100% full scale deflection, proceed with GC/MS analysis of this extract with appropriate dilution if necessary.

6.3 If peaks are detected at greater than 100% full scale deflection, calculate the dilution necessary to reduce the major peaks to between 50% and 100% full scale deflection. Use this dilution factor to dilute the extract for GC/MS analysis.

SECTION IV

GC/MS ANALYSIS OF EXTRACTABLE TARGET COMPOUNDS

1. Summary of Method

This method is to be used for the GC/MS analysis of extractable extracts screened by Section III protocols.

2. Apparatus and Materials

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 510 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 required criteria when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet (Exhibit E, Table E-1).

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 additional cost to the Government.

2.1.4 Data system - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a 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 also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

3. Reagents

3.1 Internal standards - 1,4 dichlorobenzene-d_4, napthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, perylene-d_{12}. An internal standard solution can be prepared by dissolving 200 mg of each compound in 50 mL of methylene chloride. It may be necessary to use 5% to 10% 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

4000 ng/uL. A 10 uL portion of this solution should be added to each 1 mL of sample extract. This will give a concentration of 40 ng/uL of each constituent.

3.2 Target Compound Calibration Standards

3.2.1 Prepare calibration standards at a minimum of three concentration levels. Each calibration standard shall contain each compound of interest and each surrogate standard. See GC/MS calibration in Exhibit E (Section 2.1.1) for calibration standard concentration.

4. Calibration

4.1 Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standard solutions be stored at 4°C or less in screw cap amber bottles with teflon liners. Fresh standards should be prepared every six months at a minimum.

4.2 Each GC/MS system must be hardware tuned to meet the criteria listed in Table E-1 in Exhibit E for a 50 ng injection of decafluorotriphenylphosphine (DFTPP). No sample analyses can begin until all these criteria are met. This criteria must be demonstrated each 12 hour shift. DFTPP has to be injected to meet this criterion. Post-acquisition manipulation of abundances is not acceptable.

4.3 The internal standards selected in paragraph 3.1 should permit most components of interest in a chromatogram to have retention times of 0.80 to 1.20 relative to the internal standards (see instructions for Form VI, Initial Calibration Data). Use the base peak ion from the specific internal standard as the primary ion for quantification, found in Table D-5. If interferences are noted, use the next most intense ion as the secondary ion, i.e. For 1,4-dichlorobenzene-d_4 use m/z 115 for quantification.

4.3.1 The internal standards are added to all sample extracts just prior to analysis by GC/MS and to all calibration standards. A 10 uL aliquot of the internal standard solution should be added to a 1 mL aliquot of calibration standards.

4.4 Target Compound Relative Response Factors

4.4.1 Analyze 1 to 2 uL of each calibration standard and tabulate the area of the primary characteristic ion (Table D-1) against concentration for each compound including the surrogate compounds. Calculate relative response factors (RRF) for each compound using Equation 1.

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.

A_{is} - Area of the characteristic ion for the specific internal standard from Exhibit E.

C_{is} - Concentration of the internal standard (ng/uL).

C_x - Concentration of the compound to be measured (ng/uL).

4.4.2 The average relative response factor (RRF) should be calculated for all compounds. A system performance check must be made before this calibration curve is used. Four system performance check compounds (SPCCs) are checked for a minimum average response factor. These compounds are N-nitroso-di-n propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, 4-nitrophenol. The minimum acceptable average relative response factor for extractable System Performance Check Compounds is 0.050.

4.4.3 A maximum % Relative Standard Deviation (% RSD) of 30.0% for the thirteen Calibration Check Compounds (CCC) must be met for the calibration curve to be valid (see Exhibit E, Section III, 2.3.2).

4.4.4 A check of the calibration curve must be performed once every 12 hours during analysis. These criteria are described in detail in the instructions for Form VII HCE. The minimum response factor for the system performance check compounds is 0.050. If this criteria is met, the response factor of all compounds is calculated. A percent difference of the daily response factor (12 hour) compared to the average response factor from the initial curve is calculated. A maximum percent difference of 25.0% is allowed for each compound flagged as "CCC" on Form VII. Only after both these criteria are met can sample analysis begin.

4.5 Internal standard responses and retention times in all samples must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds, the chromatographic system must be inspected for malfunction and corrections made as required. If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%), from the latest daily (12 hour) calibration standard, 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.

Retention times and EICP areas are reported on Form VIII.

5. GC/MS Analysis

5.1 The following instrumental parameters are required for all performance tests and for all sample analyses:

Electron Energy	- 70 volts (nominal)
Mass Range	- 35 to 510 amu
Scan Time	- 1 second per scan

5.2 Internal standard solution is added to each sample extract. Add 10 uL of internal standard solution to 1.0 mL (accurately measured) of sample extract.

NOTE: Make appropriate extract dilutions as indicated by the screening procedure prior to the addition of internal standards. If any further dilutions of the extracts are made, additional internal standards must be added to maintain the required 40 ng/uL of each constituent in the extract volume. If any compound saturates the detector, the extract must be diluted and reanalyzed.

Analyze an aliquot of the 1.0 mL extract by GC/MS using a bonded phase silicone coated fused silica capillary column. The recommended GC operating conditions to be used are as follows:

Initial Column Temperature Hold	- 30°C for 4 minutes
Column Temperature Program	- 30° to 300°C at 8 degrees per minute
Final Column Temperature Hold	- 300°C for 10 minutes
Injector Temperature	- 250° to 300°C
Transfer Line Temperature	- 250° to 300°C
Source Temperature	- According to manufacturer's specifications
Injector	- Grob Type, Splitless
Injection Volume	- 1 to 2 uL
Carrier Gas	- Helium at 30 cm^3/min

6. Qualitative Analysis

6.1 Single Component Extractable Target Compounds

6.1.1 The single component target compounds, listed in Exhibit C, shall be identified 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.

6.1.2 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.05 of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. The RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

6.1.3 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the Contractor's GC/MS meets the DFTPP daily tuning requirements. These standard spectra may be obtained from the run used to obtain reference RRTs.

6.1.3.1 The requirements for qualitative verification by comparison of mass spectra are as follows:

(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.

(2) The relative intensities of ions specified in (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%.)

(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. The verification process should favor false positives.

6.2 PCB Congener Analysis

6.2.1 PCB's are identified and measured by level of chlorination. Concentration is measured for each PCB isomer group.

The ten individual PCB congeners listed in Table D-2 are used as concentration calibration compounds for PCB determinations. One isomer at each level of chlorination is used as the concentration calibration standard for all isomers at that level of chlorination.

6.2.2 Identification and measurement

Special software can be used for automated identification and measurement of PCBs. Unprocessed GC/MS data are handled without human interaction with the software operationg on the dedicated computer. A concentration for each PCB isomer group is calculated automatically.

Examine each PCB candidate spectrum after background correction routines have been applied. Verify the absence of any ions with mass greater than the highest mass possible for the compound of concern. (Ions in PCB M+ ion clusters are shown in Table D-3).

6.2.2.1 For all PCB candidates, confirm the presence of an (M+70)+ ion cluster by examining ICPs or spectra for at least one of the most intense ions in the appropriate ion cluster.

6.2.2.2 For Cl_3-Cl_7 isomer groups, examine the extracted ion current profiles (EICPs) or spectra for intense (M+70)+ ions that would indicate a coeluting PCB containing two additional chlorines.

6.2.2.3 For Cl_2-Cl_8-PCB candidates, examine ICPs or spectra for intense (M+35)+ ions that would indicate a coeluting PCB containing one additional chlorine. This coelution causes interferences because of the natural abundance of ^{13}C. (This interference will be small and can be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of another PCB containing one more chlorine).

6.2.2.4 Use ICP data to calculate the ratio of the measured peak areas of the quantitation ion and confirmation ion(s), and compare to the acceptable ratio (Table D-3). If acceptable ratios are not obtained, a coeluting or partially coeluting compound may be interfering.

6.2.2.5 Quantitation and confirmation ions for each PCB isomer group must maximize within ± 1 scan of each other.

6.2.2.6 The integrated ion current for each quantitation and confirmation ion must be at least three times background noise and must not have saturated the detector.

6.2.2.7 For each PCB isomer group candidate, the ratio of the quantitation ion area to the confirmation ion

area must be within limits shown in Table D-3; at least one ion in the (M-70)+ ion cluster must be present.

6.3 A library search shall be executed for non-target compound sample components for the purpose of tentative identification. For this purpose, the most recent available version of the EPA/NBS Mass Spectral Library should be used.

6.3.1 Up to 20 nonsurrogate organic compounds of greatest apparent concentration not listed in Exhibit C for the extractable fraction shall be tentatively identified via a forward search of the EPA/NBS 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. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

6.3.2 Guidelines for making tentative identification:

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant) should be present in the sample spectrum.

(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%.

(3) Molecular ions present in reference spectrum should be present in sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

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

6.3.3 If in the opinion of the mass 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.

7. Quantitation

7.1 Target Compound Quantitation

7.1.1 The target compounds components identified shall be quantified by the internal standard method. The internal standard used shall be the one assigned in Table E-3, in Exhibit E. The EICP area of characteristic ions of analytes listed in Tables D-1, D-3, and D-5 are used. The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless a response factor is calculated using the secondary ion.

7.1.1.1 Calculate the concentration in the sample using the relative response factor (RRF) as determined in paragraph 4.4 and the following equation:

$$\text{Concentration mg/kg} = \frac{(A_x)\ (I_s)\ (V_t)}{(A_{is})\ (RRF)\ (V_i)\ (W_s)\ (1000)}$$

Where:

V_t = Volume of total extract (uL)

V_i = Volume of extract injected (uL)

W_s = Weight of sample extracted (grams)

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 (ng)

NOTE: Special software may be used for automated identification and measurement of PCBs. Unprocessed GC/MS data are handled without human interaction with the software operating on the dedicated computer. A concentration for each PCB isomer group is calculated automatically.

7.2 An estimated concentration for non-target compounds components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free-of-interferences shall be used.

7.2.1 The formula for calculating concentrations is the same as in paragraph 7.1.1.1. Total area counts 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 value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

7.3 Calculate surrogate standard recovery on all samples, control matrix spikes, and method blanks. Determine if recovery is within the suggested limits and report on appropriate form.

7.3.1 If recovery is not within the suggested limits for a sample, the following is required:

7.3.1.1 Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

7.3.1.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

7.3.1.3 If surrogates cannot be detected due to dilution factors, the requirement in 7.3.1.2 need not be met.

7.3.2 Method Blank Surrogate Recoveries

If one or more surrogates are outside the contract required limits (listed in Table E-5), the laboratory must take the following actions:

7.3.2.1 Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

7.3.2.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

7.3.2.3 If the above measures fail to correct the problem, the analytical system must be considered out of control. The method blank and all associated single phase units, including control matrix spikes must be re-extracted and reanalyzed at no additional cost to the agency.

TABLE D-1. Characteristic Ions for Extractable Target Compounds

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(-2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
Benzyl Alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
bis(2-chloroisopropyl)ether	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
Benzoic Acid	122	105, 77
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
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

TABLE D-1. (Continued)

Parameter	Primary Ion	Secondary Ion(s)
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octyl phthalate	149	-
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a, h)anthracene	278	139, 279
Benzo(g, h, i)perylene	276	138, 277
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
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	373	375, 377
Chlordane-gamma	373	375, 377
Endrin Ketone	317	67, 319

TABLE D-2. PCB Congeners Used as Calibration Standards

PCB Isomer Group	IUPAC Congener Number	Chlorine Substitution of Congener in Calibration Standard
Concentration Calibration Standard		
Monochlorobiphenyl	1	2
Dichlorobiphenyl	5	2,3
Trichlorobiphenyl	29	2,4,5
Tetrachlorobiphenyl	50	2,2',4,6
Pentachlorobiphenyl	87	2,2',3,4,5'
Hexachlorobiphenyl	154	2,2',4,4',5,6'
Heptachlorobiphenyl	188	2,2',3,4',5,6,6'
Octachlorobiphenyl	200	2,2',3,3',4,5',6,6'
Nonachlorobiphenyl	207	2,2',3,3',4,4',5,6,6'
Decachlorobiphenyl	209	2,2',3,3',4,4',5,5',6,6'

TABLE D-3. Quantitation, Confirmation, and Interference Check Ions and PCBs and Internal Standards

Analyte/ IS	Nom. MW	Quant. Ion	Confirm. Ion	Expected Ratio[a]	Accept. Ratio[a]	M-70 Confirm. Ion	Interference Check Ions M+70	Interference Check Ions M+35
PCB Isomer Group								
Cl_1	188	188	190	3.0	2.5-3.5	152[b]	256	222
Cl_2	222	222	224	1.5	1.3-1.7	152	292	256
Cl_3	256	256	258	1.0	0.8-1.2	186	326	290
Cl_4	290	292	290	1.3	1.1-1.5	220	360	326
Cl_5	324	326	324	1.6	1.4-1.8	254	394	360
Cl_6	358	360	362	1.2	1.0-1.4	288	430	394
Cl_7	392	394	396	1.0	0.8-1.2	322	464	430
Cl_8	426	430	428	1.1	0.9-1.3	356	498	464
Cl_9	460	464	466	1.3	1.1-1.5	390	-	498
Cl_{10}	494	498	500	1.1	0.9-1.3	424	-	-
Internal Standard								
Chrysene-d_{12}	240	240	241	5.1	4.3-5.9	-	-	-

[a] Ratio of quantitation ion to confirmation ion.

[b] Monodichlorobiphenyls lost HCl to produce an ion at m/z 152.

TABLE D-4.
Known Relative Abundances of Ions in PCB Molecular Ion Clusters

m/z	Relative Intensity
Monochlorobiphenyls	
188	100
189	13.5
190	33.4
192	4.41
Dichlorobiphenyls	
222	100
223	13.5
224	66.0
225	8.82
226	11.2
227	1.44
Trichlorobiphenyls	
256	100
257	13.5
258	98.6
259	13.2
260	32.7
261	4.31
262	3.73
263	0.47
Tetrachlorobiphenyls	
290	76.2
291	10.3
292	100
293	13.4
294	49.4
295	6.57
296	11.0
297	1.43
298	0.95
Pentachlorobiphenyls	
324	61.0
325	8.26
326	100
327	13.5
328	65.7
329	8.78
330	21.7
331	2.86
332	3.62
333	0.47
334	0.25
Hexachlorobiphenyls	
358	50.9
359	6.89
360	100
361	13.5
362	82.0
363	11.0
364	36.0
365	4.77
366	8.92
367	1.17
368	1.20
369	0.15
Heptachlorobiphenyls	
392	43.7
393	5.91
394	100
395	13.5
396	98.3
397	13.2
398	53.8
399	7.16
400	17.7
401	2.34
402	3.52
403	0.46
404	0.40
Octachlorobiphenyls	
426	33.4
427	4.51
428	87.3
429	11.8
430	100
431	13.4
432	65.6
433	8.76
434	26.9
435	3.57
436	7.10
437	0.93
438	1.18
439	0.15
440	0.11
Nonachlorobiphenyls	
460	26.0
461	3.51
462	76.4
463	10.3
464	100
465	13.4
466	76.4
467	10.2
468	37.6
469	5.00
470	12.4
471	1.63
472	2.72
473	0.35
474	0.39
Decachlorobiphenyls	
494	20.8
495	2.81
496	68.0
497	9.17
498	100
499	13.4
500	87.3
501	11.7
502	50.0
503	6.67
504	19.7
505	2.61
506	5.40
507	0.71
508	1.02
509	0.13

Source: J.W. Rote and W.J. Morris, J. Assoc. Anal. Chem. 56, 188, 1973.

TABLE D-5. Characteristic Ions for Surrogates and Internal Standards for Extractable Target Compounds

Surrogates	Primary Ion	Secondary Ion(s)
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-d_{14}	244	122, 212
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenapthene-d_8	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

EXHIBIT D

GC/ECD ANALYSES OF AROCLORS AND TOXAPHENE

1. Summary of Method

1.1 The analytical method that follows is designed to determine the concentration of Aroclors and Toxaphene (Target Compound List [Exhibit C]) in high concentration samples. The method can be used for analyte concentrations from the contract required quantitation limits (CRQL) to one million times the CRQL in medium and high level waste matrices.

1.2 The Aroclors and Toxaphene listed in Exhibit C are determined by a two-column GC/ECD technique.

1.3 This method specifies treatment of extracts with sulfuric acid and potassium permanganate.

1.4 GC/ECD analysis begins with initial demonstration of instrument performance and calibration of all Aroclors and Toxaphene. Acceptable initial calibration is defined in Section 6.3. This must be repeated whenever the required 12-hour performance evaluation test of Section 6.4.4 fails or when major instrument maintenance or modification is performed.

1.5 Sample extracts must be analyzed within a run sequence as defined in Section 6.4. At a minimum, the sequence consists of an initial calibration check using Aroclor standards, method blank analysis, sample extract analysis and periodic evaluation mixtures and instrument blanks. (NOTE: Data can only be collected as long as the results for the evaluation mixture and instrument blank fall within the limits defined in Sections 6.4.3 and 6.4.4. If two consecutive unacceptable evaluation standards are analyzed, all extracts with analytes present at >CRQL which have been run since the previous acceptable evaluation standard must be reanalyzed.) Additional evaluation mixtures and blanks are recommended when highly contaminated samples are suspected.

1.6 Calibration and run sequence specifications for the GC method apply separately to both columns.

1.7 One control matrix spike analysis must be run for every 20 single phase units in a Case, or once per Case, whichever is more frequent.

1.8 Absolute retention times (RT's) are used for identification of Aroclors and Toxaphene.

1.9 The absolute retention time window is calculated from the most recent standard as $\pm$1.0 percent of the RT of the standard.

1.10 Aroclors and Toxaphene are identified primarily by pattern recognition, but RT's of three to five major peaks must also be taken into consideration. Guidance on analysis of Aroclors and Toxaphene is given in Section 6.7.

1.11 Quantitative analysis of Aroclors and Toxaphene must be accomplished by the external standard method as described in Section 6.8. Three point calibration curves for Aroclors and Toxaphene must be generated during the initial calibration phase. A linear response range must be

demonstrated from the CRQL to a high point at least 30 times greater than the CRQL.

1.12 Quantitative measurements are made from extracts which have been diluted such that ECD response is within the established linear range determined by the three-point calibration curve. Quantitation must be performed and reported for both GC columns.

1.13 The surrogates, Tetrachloro-meta-xylene and Decachlorobiphenyl, must be added to all samples, blanks, and control matrix spikes analyzed by GC/ECD prior to extraction. The recovery the surrogates will be determined in all of these samples and reported to the EPA as a measure of method performance. The retention time shift of the surrogates in any standard, sample, control matrix spike, or blank may not excede 0.5%.

1.14 Section 6.6 gives criteria which determine whether an analysis is complete or whether additional cleanup or dilution is required.

1.15 All samples must be protected from light and refrigerated at 4°C from the time of receipt until extraction.

2. Apparatus and Materials

2.1 Kuderna-Danish (K-D) apparatus.

2.1.1 Concentrator tube, 10 mL, graduated (Kontes K- 570040-1029, or equivalent).

2.1.2 Evaporative flask, 500 mL (Kontes K-470001-0500, or equivalent).

2.1.3 Snyder column, three-ball macro (Kontes K-503000-0121, or equivalent).

2.2 Boiling chips.

2.2.1 Silicon carbide boiling chips (optional), approximately 10-40 mesh. Heat to 400°C for 30 minutes or solvent rinse before use.

2.2.2 Teflon boiling chips (optional). Solvent rinse before use.

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

2.4 Top loading balance, capable of accurately weighing to ±0.01 g.

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

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

2.7 Vials and caps, 1 or 2 mL for GC auto sampler.

2.8 Vacuum system for eluting multiple cleanup cartridges.

2.8.1 Vac Elute Manifold (Analytichem International, Harbor City, J.T. Baker or Supelco) or equivalent.

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

2.8.3 Vacuum pressure gauge.

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

2.9 Glass vials, at least 20 mL, with screw cap and teflon or inert plastic liner for sample extraction and for sulfuric acid and permanganate treatment.

2.10 Spatula, stainless steel or teflon.

2.11 Pipet, Volumetric 1.00 mL or 2.00 mL (optional).

2.12 Syringe, 1.00 mL or 2.00 mL (optional).

2.13 Flask, Volumetric 10.00 mL.

2.14 Flask, Volumetric 1.00 mL or 2.00 mL (optional).

2.15 Vials, 10 mL, with screw cap and teflon liner (optional).

2.16 Tube, centrifuge, 12 to 15 mL with 19 mm ground glass joint, (optional).

2.17 Snyder Column, micro two or three ball with a 19 mm ground glass joint.

2.18 Centrifuge, table top (optional)

2.19 Gas chromatographic system, including a 0.25 inch injector and an electron capture detector. The GC must be equipped with an integrator or data system rather than a strip chart recorder. Detector makeup gas is required for capillary analysis.

2.19.1 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 DB-1701, 30 m x 0.53 mm ID, 1.0 to 1.5 um film thickness, J&W Scientific, Folsom, CA and a DB-608 or SPB-608, 30 m x 0.53 mm ID, 0.8 to 1.5 um film thickness (or equivalent) from J&W Scientific or Supelco, Inc., Bellefonte, PA. Equivalent columns may be employed if they meet the requirements in 2.19.3.

2.19.2 Columns are mounted in 1/4 inch injector ports using glass adapters available from a variety of commercial sources (J&W Scientific, Supelco, Inc., Hewlett-Packard, Varian, Inc., Perkin Elmer).

2.19.3 Column equivalence is demonstrated by running the calibration standards mixtures described in Section 6.3.1. Each equivalent column must be calibrated according to the procedures described in Section 6.3 and satisfy all of the acceptance criteria described therein (if an equivalent column is used, it must be described in the Case Narrative).

2.19.4 The carrier gas must be helium.

2.19.5 Because the column flow for wide bore capillary systems is 5 mL/minute which is slower than for packed column systems, it is necessary to have precise control of the carrier gas flow and to supply makeup gas to the detector. The makeup gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification.

2.20 Vortex mixer, Genie, Model 550-6, Scientific Industrial, Inc., Bohemia, NY (or equivalent).

2.21 Disposable Pasteur pipets, packed with glass wool rinsed with hexane.

2.22 Ultrasonic cell disruptor, Heat Systems Ultrasonics, Inc., Model W-385 SONICATOR (475 Watt with pulsing capability, 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.

2.23 Sonabox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.

3. Reagents

3.1 Hexane, acetone, iso-octane (optional), and methanol (optional) solvents of pesticide residue analysis grade or equivalent. It is recommended that each lot of solvent be analyzed to demonstrate that it is free of interference before use.

3.2 Primary Aroclor standards will be obtained from the EPA Quality Assurance Materials Bank, Pesticides and Industrial Chemicals Repository, Research Triangle Park, NC, if available. Commercial standards must be used for all working solutions after they have been shown to be the proper material and at least 95 percent pure by comparison to primary standards.

3.3 Mercury (optional).

3.4 Copper powder (optional), bright and non-oxidized.

3.5 Concentrated sulfuric acid (Sp. gr. 1.84).

3.6 Potassium permanganate solution (5 percent w/v). Slowly add 100 mL water to 5 g of potassium permanganate in a Pyrex vessel.

3.7 Diol bonded silica 500-mg, cartridges with stainless steel frits Catalog No. 614313, Analytichem, 24201 Frampton Ave., Harbor City, CA (or equivalent).

3.8 Ten percent acetone in hexane (v/v). Prepare the mixture by adding 10 mL of acetone to 90.0 mL of hexane. NOTE: Prepare this mixture accurately or the results from the Diol cartridge cleanup will be adversely affected.

3.9 Surrogate Standard Solution.

The surrogates, tetrachloro-meta-xylene and decachlorobiphenyl, are added to all samples, the control matrix spike, and blanks. Prepare a surrogate standard spiking solution of 1.0 ug/mL of each of the surrogates in acetone. 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.

3.10 Aroclor control matrix spike solution. Prepare a spiking solution in acetone or methanol that contains 25 ug/mL of Aroclor 1254.

3.11 Performance evaluation standards are a series of 12 mixtures each containing the surrogates and one other analyte, at three concentration levels (low, medium, and high). The other analytes are specified in 6.4.4.1 (note that the mixture of Aroclors 1016 and 1260 is considered as a single analyte). The concentration level of the low level standard is given in 6.3.3. The medium level standards are 10 times the low level concentrations in 6.3.3, and the high level standards are 30 times the low level concentrations.

4. Sample Preparation For GC/ECD Analyses

4.1 High concentration samples are initially separated into individual phases. An aliquot of 500 mg of each phase is transferred to a separate 20 mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. To avoid cross contamination, cap the vial before proceding to the next phase unit.

4.1.1 Add 1.0 mL of the surrogate spiking solution to each vial.

4.1.2 Add 1.0 g of anhydrous sodium sulfate to each vial, and mix with a clean spatula.

4.1.3 Add 9.0 mL of hexane to each vial, and sonicate each vial for 2 minutes at 100 watts power. For control matrix spike, add only 8.0 mL of hexane (see 4.3 below).

4.1.4 Filter the extract through a disposable pipet loosely packed with glass wool. Collect at least 7 mL of extract in a clean vial.

4.1.5 Proceed to paragraph 5.1 for extract cleanup procedures.

4.2 Control matrix spikes, blanks and all single phase units are spiked with the surrogate solution. The control matrix spike and the blanks are subjected to the same extraction, cleanup, and dilution procedures as the samples.

4.3 Once per Case, or for every group of 20 single phase units (whichever is more frequent), a control matrix spike sample is prepared with 1.0 mL of the Aroclor 1254 spiking solution and 500 mg of corn oil. The control matrix spike must be extracted, cleaned up, and analyzed in the same fashion as all other single phase units (see 4.1 above). Note: Add only 8.0 mL of hexane to the vial during the extraction of the control matrix spike.

4.4 Interferences

4.4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware; these contaminants lead to discrete artifacts, and/or elevated baselines, in gas chromatograms. All of these materials should be routinely demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences by phthalate esters can pose a major problem in Aroclor analysis when using the electron capture detector. Common flexible plastics contain varying amounts of phthalate esters which are easily extracted during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.4.2 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 of the site being sampled. The cleanup procedures in Section 5 must be used to remove such interferences in order to achieve the contract required quantitation limits.

5. Extract Cleanup For GC/ECD Analyses

5.1 Requirements

5.1.1 This procedure is only suitable for Aroclors and Toxaphene. Because the cleanup involves the use of sulfuric acid and potassium permanganate, the final solutions are not suitable for pesticide analysis.

5.1.2 Diol cartridge cleanup is required for all samples, control matrix spikes and blanks. This procedure removes polar organic molecules such as phenols. Each lot number of Diol cartridges must pass a cartridge performance check.

5.1.3 Sulfur can be removed by one of two methods according to laboratory preference (Section 5.6).

5.1.4 Blanks and control matrix spikes must be subjected to the same cleanup as the field samples.

5.1.5 It is required that all the waste aliquots be diluted with hexane before initiating the following treatments.

5.1.6 No GPC cleanup is used in the preparation of samples for GC/ECD analysis under this protocol.

5.2 Sulfuric Acid Cleanup

5.2.1 Using a syringe or a volumetric pipet, transfer 2.0 mL of the hexane solution to a 10 mL vial and carefully add 5 mL of conc. sulfuric acid. This procedure must always be done in a fume hood. NOTE: If the remaining extract from the method blank is saved at this point, it may be used in paragraph 5.6.3.2 to prepare a sulfur blank.

5.2.2 CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

5.2.3 Cap the vial tightly and vortex for one minute. A vortex must be visible in the vial.

5.2.4 CAUTION: Stop the vortexing immediately if the vial leaks, AVOID CONTACTING THE SOLUTION WITH BARE SKIN, SULFURIC ACID WILL BURN.

5.2.5 Allow the phases to separate for at least one minute. Examine the top (hexane) layer, it should not be highly colored nor should it have a visible emulsion or cloudiness.

5.2.6 If a clean phase separation is achieved proceed to Step 5.2.10.

5.2.7 If the hexane layer is colored or the emulsion persists for several minutes, remove the sulfuric acid layer from the vial via a glass pipette and dispose of it properly. Add another 5 mL of clean sulfuric acid.

5.2.8 Note: Do not remove any hexane at this stage of the procedure.

5.2.9 Vortex the sample and allow the phases to separate as described previously.

5.2.10 Transfer the hexane layer to a clean 10 mL vial.

5.2.11 Add an additional 1 mL of hexane to the sulfuric acid layer, cap the vial securely and shake. This second extraction is done to ensure quantitative transfer of all analytes.

5.2.12 Remove the second hexane layer and combine with the hexane from Step 5.2.10.

5.3 Permanganate Cleanup

5.3.1 Add 5 mL of the five percent aqueous potassium permanganate solution to the combined hexane fractions from 5.2.12.

5.3.2 CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

5.3.3 Cap the vial tightly and vortex for one minute. A vortex must be visible in the vial.

5.3.4 CAUTION: Stop the vortexing immediately if the vial leaks, AVOID CONTACTING THE SOLUTION WITH BARE SKIN, POTASSIUM PERMANGANATE WILL BURN.

5.3.5 Allow the phases to separate for at least one minute. Examine the top (hexane) layer, it should not be highly colored nor should it have a visible emulsion or cloudiness.

5.3.6 If a clean phase separation is achieved, proceed to Step 5.3.10.

5.3.7 If the hexane layer is colored or the emulsion persists for several minutes, remove the permanganate solution from the vial via a glass pipette and dispose of it properly. Add another 5 mL of the clean aqueous permanganate solution.

5.3.8 NOTE: Do not remove any hexane at this stage of the procedure.

5.3.9 Vortex the sample and allow the phases to separate.

5.3.10 Transfer the hexane layer to a clean 10-mL vial.

5.3.11 Add an additional 1 mL of hexane to the permanganate layer, cap the vial securely and shake. This second extraction is done to ensure quantitative transfer of all Aroclors and Toxaphene.

5.3.12 Remove the second hexane layer and combine with the hexane from Step 5.3.10.

5.4 Final Preparation

5.4.1 Reduce the volume of the combined hexane layers to 1.0 mL under a stream of dry nitrogen.

5.4.1.1 Nitrogen Blowdown Technique (Taken from ASTM Method D 3086).

5.4.1.1.1 Place the concentrator tube in a heating bath (30-35°C) and evaporate the solvent to the final volume using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). The extract must never be allowed to become dry.

5.4.1.1.2 CAUTION: 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 then dried prior to use.

5.4.2 Prepare the extracts using the Diol cartridge cleanup as described below.

5.5 Diol Cartridge Procedure

5.5.1 Cartridge Performance Check

Each lot number of Diol cartridges must be tested by the following procedure before it is used for sample cleanup. Add 1.0 mL of the control matrix spike solution to 4 mL of hexane. Place a 1.0 mL aliquot of the diluted solution onto the top of a prewashed Diol cartridge, and elute it with 9 mL of hexane/acetone [90:10(V/V)]. Adjust the final volume to 10.0 mL and analyze by GC/ECD. The recovery of Aroclor 1254 must be determined for evaluation and reporting purposes. The lot of Diol cartridges is acceptable if the Aroclor is recovered at 80 to 110 percent.

5.5.2 Diol Cartridge Cleanup

5.5.2.1 Attach the Vac Elute vacuum manifold to a water aspirator or 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.

5.5.2.2 A 500 mg Diol cartridge is selected for each hexane solution of waste and placed into the vacuum manifold.

5.5.2.3 Prior to cleanup of the hexane solutions of waste, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge in the vacuum manifold, pulling a vacuum and passing 5 mL of the hexane/acetone solution through the cartridge.

5.5.2.4 After the cartridges in the manifold are washed, the vacuum is released and a rack containing labeled 10 mL volumetric flasks is placed inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside of the appropriate volumetric flask as the manifold top is replaced.

5.5.2.5 After the volumetric flasks are in place, vacuum to the manifold is restored and 1 mL (the entire volume) from each sample, blank, or control matrix spike solution is transferred to the top frit of the appropriate Diol cartridge.

5.5.2.6 The Aroclors and Toxaphene in the solution concentrates are then eluted through the column with 9 mL of hexane/acetone (90:10) and collected into the 10 mL volumetric flasks held in the rack inside of the vacuum manifold.

5.5.2.7 Transfer the eluate in each volumetric flask to a clean centrifuge tube or 10 mL vial. Use two additional 1 mL hexane rinses of the flask to ensure quantitative transfer of the cartridge eluate.

5.5.2.8 Adjust the extract volume to 10.0 mL with hexane

5.5.2.9 If crystals of sulfur are evident or the presence of sulfur is suspected, proceed to Section 5.6.

5.5.2.10 If sulfur is not expected to be a problem, transfer the 1 mL of solution to a GC vial and label the vial. (Some autosamplers require 1 mL solvent volumes, others require 2 mL.) The solution is ready for the GC/ECD analysis detailed in Section 6. Store the extracts at 4°C in the dark until analyses are performed.

5.6 Sulfur Removal

5.6.1 Two options are available for the removal of sulfur from samples. The mercury technique appears to be the most reliable, but requires the use of small volumes of mercury in the laboratory.

5.6.2 CAUTION: Mercury containing waste should be segregated and disposed of properly.

5.6.3 Mercury Technique

5.6.3.1 Add 1 to 3 drops of mercury to 1.0 mL of each hexane solution in a clean vial. Tighten the top on the vial and agitate it for 30 seconds. Filter or centrifuge and decant the solution to remove all

solid precipitates and liquid mercury. Proceed to Section 6 for GC/ECD analysis if the mercury appears shiny. If the mercury turns black, repeat the process as necessary until it remains shiny. Dispose of the mercury waste properly.

5.6.3.2 If only a partial set of the hexane concentrates require sulfur cleanup, an additional reagent blank of hexane and mercury (or copper) is required. This additional blank may be prepared from the remaining blank extract in paragraph 4.14.

5.6.4 Copper Technique

5.6.4.1 Bright (non-oxidized) granular copper (one to three granules) can be used in place of mercury in the procedure described in Section 5.6.3. If the copper appears shiny, proceed to Section 6 for GC/ECD analysis. If the copper changes color, repeat sulfur removal as necessary.

6. GC/ECD Analysis For Aroclors

6.1 Summary

6.1.1 This GC/ECD method is used for the analysis of the Aroclors and Toxaphene only from Exhibit C. Although this method is similar to GC/ECD methods for pesticides, this method is only appropriate for Aroclors and Toxaphene.

6.1.2 1.0 mL of the surrogate spiking solution is added to all single phase units, control matrix spikes and blanks prior to extraction and surrogate recoveries will be reported with sample data. The retention time shift of the surrogates in any standard, sample, control matrix spike, or blank may not excede 0.5%.

6.1.3 Control matrix spikes are required for this method and are prepared from the Aroclor 1254 control matrix spike solution.

6.1.4 Quantitation of Aroclors and Toxaphene is done by external standard techniques using three-point curves generated during an initial calibration sequence. Quantitation is based on comparison of three to five sample peaks with the corresponding peaks in the standard.

6.1.5 The absolute retention times of single component organochlorine pesticides are determined as part of this method and reported with the sample data in order to prevent misidentification of these compounds as constituent peaks of Aroclors or Toxaphene.

6.1.6 Sample data are collected after an initial calibration sequence is run. The RT's, calibration factors, and column performance are monitored no less than once every 12 hours with an

instrument blank and with a standard. Data can be collected only as long as all evaluation criteria given in Section 6.4.4 are met. In all GC runs, the injector must be heated to at least 205°C. The injection must be made on column, using either automatic or manual injection. If autoinjectors are used, 1.0 uL injection volumes may be used. Manual injections must use at least 2.0 uL injection volumes.

6.1.7 The carrier gas must be helium.

6.1.8 The analysis of samples is accomplished by using two wide bore (0.53 mm ID) fused silica GC columns. A separate detector is required for each column. The specified analytical columns are DB-1701, 30 m x 0.53 mm ID, 1.0 to 1.5 um film thickness, J&W Scientific, Folsom, CA and a DB-608 or SPB-608, 30 m x 0.53 mm ID, 0.8 to 1.5 um film thickness (or equivalent) from J&W Scientific or Supelco, Inc., Bellefonte, PA. Equivalent columns may be employed if they meet the requirements in 2.19.3.

6.1.9 Analysis of a sample on both columns is only required when one column gives at least one positive result. Peak identification and quantitation must be reported separately for both columns if any Aroclors or Toxaphene are detected in a sample.

6.1.10 Wide bore capillary columns are installed in standard 0.25 inch packed column injector and detector ports by use of suitable glass adapters and ferrules. Because the column flow used is 5 mL/minute of helium, it is necessary to have precise carrier gas flow and to supply makeup gas to the detector.

6.1.11 Electron capture detectors must be plumbed with P-5, P-10 (argon/methane) or nitrogen as a detector makeup gas according to the instrument specification.

6.1.12 The temperature program for GC analysis is:

T_i	150°C
Initial time	1/2 minute
Temperature ramp	5°/minute to 275°C
Final hold	10 minutes

NOTE: It may be necessary to adjust this temperature program for individual gas chromatographs. It is a requirement for this method that the oven(s) regulate temperature.

6.1.13 All calibration and run sequence requirements of the following sections apply independently to <u>both</u> the specified columns.

6.2 Calibration Standards

6.2.1 The resolution check standard must be run after the first blank in the calibration sequence in order to demonstrate the ability of the GC column to resolve certain pesticide compounds. The

resolution check standard must contain the following seven compounds each with a concentration of 50 ng/mL:

Methoxychlor
Endrin ketone
p,p'-DDE
Dieldrin
Endosulfan sulfate
Endosulfan I
gamma-Chlordane

6.2.2 The resolution criterion is that the height of the valley between two adjacent peaks in the mixture must not be greater than 60% of the height of the shorter peak. Experience to date suggests that the poorest resolution on the DB-608 column will be between DDE and Dieldrin and between Methoxychlor and Endrin ketone. On the DB-1701 column, resolution difficulties should be expected between Endosulfan I and gamma-Chlordane and between Methoxychlor and Endosulfan sulfate.

6.2.3 Each Aroclor must be analyzed at the three concentrations given in 6.3.1 and 6.3-6.5 in order to establish the response factors for the quantitation peaks for each Aroclor and to demonstrate detector linearity.

The high point concentration defines the upper end of the concentration range for which the calibration is valid.

6.2.4 Aroclor and Toxaphene standards must be prepared individually, except for Aroclor 1260 and Aroclor 1016, which can be combined in one standard mixture.

6.2.5 The performance of the GC system is monitored by the use of a standard and an instrument blank, both of which must be run no less than once every 12 hours (see Section 6.4.4 for the one exception to this requirement). The instrument blank is a hexane solution containing 20 ng/mL of each of the surrogates.

6.3 Initial Calibration

6.3.1 All columns in all GC systems used to collect data using this method must be calibrated with the following sequence of standards (1) during the initial set up and (2) whenever corrective action is required because a 12-hour check has shown the instrument to be out of control (Section 6.4.4).

Injection No.	Concentration	Aroclor	No. of Potential Quantitation Peaks
1	Blank	–	–
2	Resolution Check	–	–
3	Low	1221	4
4	Medium	1221	4
5	High	1221	4
6	Low	1232	4
7	Medium	1232	4
8	High	1232	4
9	Low	1242	5
10	Medium	1242	5
11	High	1242	5
12	Low	1248	5
13	Medium	1248	5
14	High	1248	5
15	Low	1254	5
16	Medium	1254	5
17	High	1254	5
18	Low	1016/1260	5/5
19	Medium	1016/1260	5/5
20	High	1016/1260	5/5
21	Low	Toxaphene	4
22	Medium	Toxaphene	4
23	High	Toxaphene	4
24	Low	Pesticide A	–
25	Low	Pesticide B	–
26	Blank	–	–

6.3.2 Select four or five major peaks from each Aroclor and four major peaks from Toxaphene as potential quantitation peaks. The same peaks must be used throughout the run sequence, including: initial calibration, 12-hour performance checks, sample quantitation, and control matrix spike analyses.

6.3.3 The low-point concentration calibration standards for the method analytes are:

LOW POINT AROCLOR CALIBRATION SOLUTIONS

Compound	CAS Number	Low Concentration
1. Aroclor 1016	12674-11-2	50 ng/mL
Aroclor 1260	11096-82-5	50 ng/mL
2. Aroclor 1221	11104-28-2	50 ng/mL
3. Aroclor 1232	11141-16-5	50 ng/mL
4. Aroclor 1242	53469-21-9	50 ng/mL
5. Aroclor 1248	12672-29-6	50 ng/mL
6. Aroclor 1254	11097-69-1	50 ng/mL
7. Toxaphene	8001-35-2	500 ng/mL
8. Tetrachloro-meta-xylene	877-09-8	20 ng/mL
9. Decaclorobiphenyl	2051-24-3	20 ng/mL

6.3.4 The mid-point concentration of each standard is prepared at 10 times the concentrations given in 6.3.3. The surrogates must be added at 200 ng/mL to all mid-point calibration solutions.

6.3.5 The high point concentration is prepared at 30-100 times the concentrations given in Section 6.3.3. The surrogates must be added at at least 600 ng/mL to all high point calibration solutions.

6.3.6 Determine the absolute retention times (RT) for the selected major peaks for each Aroclor and Toxaphene as well as the relative mean deviation (RMD) for each peak,

$$RMD = \frac{100\%}{3} \sum_{i=1}^{3} \left| RT_i - \overline{RT} \right| / \overline{RT}$$

where $\overline{RT}$ is the mean RT for a specific peak in a particular Aroclor and Toxaphene. The RMD for each Aroclor and Toxaphene must be less than 0.5 percent before analytical results can be reported.

6.3.7 Only three peaks are required for sample quantitation, however, the peaks chosen must not co-elute with matrix interference. Therefore, linearity of response is required for each of the four or five potential quantitation peaks selected during the initial calibration in order to establish system performance.

6.3.8 Three-point instrument calibration is required for each potential quantitation peak of each Aroclor and Toxaphene. The laboratory has three choices on how to establish a three point calibration. Only one of the three calibration methods can be used to quantitate samples in any single run sequence. Laboratories may not mix calibration techniques for samples quantitated using a single initial calibration.

6.3.8.1 The laboratory can use a mean calibration factor (CF) determined from the three concentrations, <u>but only</u> if the % RSD for the three points is < 15.0 percent.

$$\overline{CF} = \frac{1}{3} \sum_{i=1}^{3} \frac{\text{Response of Selected Peaks in Standard}}{\text{Mass Injected (ng)}}$$

$$\%RSD = \frac{SD}{\overline{CF}} \times 100$$

$$\text{Where SD} = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \qquad \text{and } n=3$$

6.3.8.2 The laboratory can use a calibration line drawn through all three calibration points if the value for r (correlation coefficient from the linear regression calculation) is >0.975 and if the zero concentration intercept is <0.20.

$$\text{Where } r = \frac{(1/n) \sum_{i=1}^{n} (x_i - \bar{x})(y_i - \bar{y})}{[(1/n) \Sigma (x_i - \bar{x})^2]^{1/2}[(1/n) \Sigma (y_i - \bar{y})^2]^{1/2}}$$

6.3.8.3 Laboratories with electronic integrators or data systems that automatically calculate calibration curves as line segments between calibration points may use two line segment calibration curves for each quantitation peak of the Aroclor or Toxaphene. This technique may be used only if r (the correlation coefficient from the linear regression calculation) is >0.975 for all three points for each peak and if the zero concentration intercept is <0.20 times the low point response for each Aroclor or Toxaphene peak quantitated.

6.3.9 Mixtures of single component pesticides are injected as part of the calibration sequence to establish the RT of individual pesticides because they are potential method interferences. Calibration factors are not calculated for the individual pesticide standards.

6.3.10 Single Component Pesticide Mixtures

Individual Standard Mix A	Concentration (ng/mL)	Individual Standard Mix B	Concentration (ng/mL)
alpha-BHC	25.0	beta-BHC	25.0
Heptachlor	25.0	delta-BHC	25.0
gamma-BHC	25.0	Aldrin	25.0
Endosulfan I	25.0	Heptachlor epoxide	25.0
Dieldrin	50.0	alpha-Chlordane	25.0
Endrin	50.0	gamma-Chlordane	25.0
p,p'-DDD	50.0	p,p'-DDE	50.0
p,p'-DDT	50.0	Endosulfan sulfate	50.0
Methoxychlor	50.0	Endrin aldehyde	50.0
Tetrachloro-meta-xylene	20.0	Endrin Ketone	50.0
Decachlorobiphenyl	20.0	Endosulfan II	50.0
		Tetrachloro-meta-xylene	20.0
		Decachlorobiphenyl	20.0

6.3.11 The average retention times of the surrogates must be calculated using all 26 injections listed in 6.3.1. This value is used to establish the acceptance criteria for all subsequent injections.

$$\overline{RT}\ (\text{surrogate}) = \frac{\sum_{26} RT\ (\text{surrogate})}{26}$$

6.3.12 The mean response for each surrogate in the low-point calibration analysis is used as the surrogate calibration factor (CF).

6.3.13 Sample analysis may not proceed until a satisfactory calibration has been demonstrated.

6.3.14 The equivalence of GC columns other than those specified in 2.19.1 may be demonstrated by:

- o Successful initial calibration of the gas chromatographic system including meeting the requirements in 6.3.6 and 6.3.8.
- o Meeting the resolution criterion in 6.2.2.
- o Achieving baseline resolution of each of the components in Individual Mix A from one another and baseline resolution of each of the components in Individual Mix B from one another.

6.4 Sample Analysis Run Sequence

6.4.1 Summary

6.4.1.1 Extracts of single phase units are analyzed as part of a run sequence that includes both instrument blanks and performance evaluation standards, which provide both calibration verification and instrument performance evaluation. Both an instrument blank and a standard must be analyzed successfully on each column no less than once in every 12 hours that sample data are collected. In addition, the laboratory must analyze at least one method blank for each Case, or once per 20 single phase units, whichever is more frequent. Acceptance criteria for the instrument blanks (Section 6.4.3), method blanks (Section 6.4.2), and for the performance evaluation standards (Section 6.4.4) are given below.

6.4.1.2 The laboratory may identify and quantitate analyte peaks based on data collected during the initial calibration as long as an acceptable instrument blank and an acceptable evaluation mixture are analyzed every 12 hours. The requirements for the run sequence apply to <u>both</u> columns in all instruments used in these analyses.

6.4.1.3 Example Run Sequence

Note: The 12 hours are counted from the injection of the sample in step 27, not from step 1.

Time	Injection #	Material Injected
	1	Instrument Blank
	2	Resolution Check
	3 - 26	Initial Calibration Standards and Blanks
0 hr.	27	First Sample
	o	
	o	Subsequent
	o	Samples
	o	
12 hr.	o	Instrument Blank
	1st injection past 12 hr.	Evaluation Mixture
	2nd injection	Sample
	o	
	o	Subsequent
	o	Samples
	o	
	o	
24 hr.	o	Instrument Blank
	1st injection past 24 hr.	Evaluation Mixture
	2nd injection	Sample
	o	
	o	

6.4.1.4 The run continues until an unacceptable instrument blank or standard is analyzed. This example run sequence shows only the minimum number of instrument blanks and evaluation mixtures necessary to meet the requirements. More instrument blanks and evaluations may be run at the discretion of the laboratory, but these must satisfy the criteria presented in Section 6.4.3 and 6.4.4.

6.4.1.5 A run sequence must include all required control matrix spike analyses and method blanks, but each laboratory may decide at what point in the run sequence they are analyzed.

6.4.2 Method Blanks

The method blank consists of 500 mg of corn oil spiked with the surrogates at 20 ng/mL that is subjected to the same cleanup as a sample, in order to check for system contamination. None of the Aroclors or Toxaphene listed in Exhibit C may be present at greater than the CRQL in the analysis of a method blank. If any of the Aroclors or Toxaphene are present at >CRQL, data

collection must be stopped, and the data for all single phase units analyzed since the last acceptable blank are considered suspect. Therefore all single phase units with Aroclors and Toxaphene detected at levels >CRQL that were prepared during the same 12-hour shift as a contaminated method blank must be reextracted and reanalyzed at no additional expense to the Agency. At least one method blank must be included for every 20 single phase units.

6.4.3 Instrument Blank

An instrument blank is a hexane solution containing 20 ng/mL of the surrogates. An acceptable instrument blank analysis must demonstrate that no Aroclor or Toxaphene can be detected at greater than 0.5 times the CRQL. If Aroclors or Toxaphene are detected at greater than half the CRQL, all data collection must be stopped and corrective action taken. Data for samples with analytes detected at >CRQL analyzed between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be analyzed before additional data are collected. After an acceptable instrument blank is analyzed, all single phase units with Aroclors or Toxaphene detected at levels >CRQL that were analyzed after the previous acceptable instrument blank must be reinjected during a valid run sequence and reported at no additional expense to the Agency.

6.4.4 Performance Evaluation Standards

6.4.4.1 The performance evaluation standards injected at 12 hour intervals will be rotated so as to include the common Aroclors and Toxaphene at low, medium, and high concentrations according to the following schedule:

NOTE: The time clock starts <u>after</u> the completion of the initial calibration as in 6.14.3. The performance evaluation mixture is injected following an instrument blank, as in 6.4.13.

Concentration	Compound	Hour
low	Toxaphene	12
low	1248	24
low	1254	36
low	1016/1260	48
medium	Toxaphene	60
medium	1248	72
medium	1254	84
medium	1016/1260	96
high	Toxaphene	108
high	1248	120
high	1254	132
high	1016/1260	144
Repeat		

6.4.4.2 For each of the four or five potential quantitation peaks (Section 6.3.1), the RT in the standard must be within the retention time window of ±1.0 percent of the mean RT calculated during the initial calibration.

6.4.4.3 For each of the four or five potential quantitation peaks (Section 6.3.1), the response (area or height) must be within 20.0 percent (±20.0 RPD) of the mean response obtained during the initial calibration.

$$RPD = \frac{\overline{CF} - CF_E}{\overline{CF}} \times 100$$

Where:

$\overline{CF}$ = Average calibration factor from initial calibration (Equation 6.2).

CF_E = Calibration factor from current evaluation mixture.

6.4.4.4 The retention time shift of the surrogates must be within 0.5 percent of the average retention time established during the initial calibration.

6.4.4.5 If a performance evaluation standard does not meet the criteria listed above, it must be reinjected immediately. If the second injection also does not meet the criteria, all data collection must be stopped. Appropriate corrective action must be taken and a new initial calibration sequence must be analyzed before more sample data are collected. Data collected after the last acceptable evaluation mixture are considered suspect and all extracts with analytes present at > CRQL must be reinjected and reported at no additional expense to the Agency.

6.4.4.6 Analysts are cautioned that running an instrument blank and an evaluation mixture once every 12 hours is the <u>minimum</u> contract requirement. Highly complex samples or unstable GC equipment may cause peaks from one injection to be carried over into the next. It may be necessary to analyze instrument blanks and evaluation mixtures more often in order to avoid discarding data.

6.4.4.7 The requirement for running the 12 hour instrument blanks and evaluation standards is waived when no sample or control matrix spikes are analyzed during the 12-hour period. After a break in sample data analysis, a laboratory may resume the analysis of

samples and control matrix spikes using the current initial calibration after an acceptable performance evaluation standard is analyzed. If a successful continuing calibration cannot be demonstrated after an interruption, an acceptable initial calibration must be run before sample data can be collected.

NOTE: This section does not affect the requirement that all acceptable sample analyses must be bracketed by acceptable evaluation mixtures.

6.4.5 Control Matrix Spike

6.4.5.1 A control matrix spike must be analyzed once per Case, or once for every 20 single phase units, whichever is more frequent.

The percent recovery of each of the five Aroclor 1254 peaks is calculated using the following equation:

$$\text{Matrix Spike Recovery} = \frac{SSR-SR}{SA} \times 100$$

Where:

SSR = Spike Sample Recovery
SR = Sample Result
SA = Spike Added

These results are reported per Aroclor 1254 peak (5 total).

6.4.5.2 Control matrix spike recoveries will be reported by the Contractor. (See Exhibit B.)

6.5 Sample Analyses

6.5.1 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 samples must be reanalyzed following dilution, or another aliquot of the original dilution should be cleaned up and analyzed. Samples which cannot be made to meet specifications given in this section after the second full cleanup (sulfuric acid, permanganate, Diol and mercury sulfur removal) are reported as intractable in the Case Narrative and do not require further analysis. No limit is placed on the number of repeat full cleanups of samples that may be required because of contaminated method blanks.

6.5.2 The sample must be analyzed at the most concentrated level consistent with achieving satisfactory chromatography. If dilution is employed solely to bring a peak within the

calibration range or to produce an on-scale Aroclor or Toxaphene pattern, the results for both a more and a less concentrated extract must be reported. The resulting changes in quantitation limits and surrogate recovery must also be reported for the dilute samples.

6.5.3 If the laboratory 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 in 6.6) is achieved with the diluted cleaned sample extract, an additional analysis at 10 times the concentration of the dilution must be injected and reported with the sample data.

6.5.4 The peak response of sample peaks in diluted cleaned samples must be >25 percent of full scale to allow visual pattern recognition of the Aroclors and Toxaphene.

6.5.5 An on-scale chromatogram(s) of all selected Aroclor quantitation peaks must be presented with the sample data.

6.5.6 Chromatographic data may be replotted electronically in order to produce an on-scale chromatogram, except when the off-scale sample peaks are larger than the high point calibration peaks. In that case, the samples must be diluted and reinjected.

6.5.7 The peak response of sample peaks on the replotted chromatogram must be >25 percent of full scale to allow visual pattern recognition of the Aroclors or Toxaphene.

6.6 Data Acceptance Criteria

6.6.1 Reportable data for a sample must include a chromatogram with a baseline which returns to below 50 percent of full scale before the elution time of Aroclor 1221 and to below 25 percent of full scale after Aroclor 1221 and before the elution time of Decachlorobiphenyl.

6.6.2 If dilution has been applied and no peaks are detected above 25 percent of full scale, analysis of a more concentrated sample is required.

6.6.3 Reportable sample data must include chromatogram(s) with all detected Aroclor or Toxaphene peaks in the linear range of the quantitation determined by the initial calibration.

6.6.4 NOTE: If more than one chromatogram is required to satisfy the criteria for a sample, the results of all chromatograms must be reported. These requirements apply to both columns.

6.6.5 Peaks used for quantitation that are more than two times the width of the high concentration calibration peaks for that analyte must be reported with an appropriate data flag. (See Exhibit B.)

6.6.6 The retention time shift of the surrogates in any standard, sample, control matrix spike, performance evaluation standard, or blank may not excede 0.5%.

6.7 Identification Of Aroclors And Toxaphene

6.7.1 Aroclors and Toxaphene present special analytical difficulties. Because of the alteration of these materials in the environment, it is probable that samples which contain Aroclors or Toxaphene will give similar but not identical patterns as Aroclor or Toxaphene standards. Thus, identification requires visual inspection of an on-scale pattern. The pattern may be brought on-scale either by diluting the sample and reinjecting, or by replotting the chromatograph stored in a laboratory data system.

6.7.2 The choice of the peaks used for quantitation and recognition of those peaks may be complicated by the environmental alteration of the Aroclors and Toxaphene and by the presence of coeluting analytes and/or matrix interferences.

6.7.3 The numbers of potential quantitation peaks are listed in 6.3.1.

6.7.4 The more highly chlorinated components of the Aroclors and Toxaphene are more stable in the environment. Therefore, the analyst should emphasize the later eluting peaks of a pattern in identifying analytes when weathered Aroclors or Toxaphene are detected.

6.8 Quantitation Of Analytes

6.8.1 Analytes may be quantitated using either a modern electronic integrator or a laboratory data system. The analyst may use either peak height or peak area as the basis for quantitation, however the use of area versus height must be consistent between samples. The use of an electronic integrator or a laboratory data system is <u>required</u>.

6.8.2 The chromatograms of all samples must be reviewed by a qualified Pesticide/PCB analyst before they are reported.

6.8.3 Using an electronic integrator, one of three calibration techniques may be employed (6.3.5). It is the responsibility of the analyst to set the integration parameters such that off-scale chromatograms are within the dynamic range of the instrument. The analyst should also check for data flags generated by the instrument that indicate improper quantitation of peaks prior to reporting data to the EPA.

6.8.4 In order to be quantitated, the detector response (peak area or peak height) of all analytes must lie between the responses of the low and high concentrations in the initial calibration. If the analytes are detected below the CRQL, they are reported

with the appropriate flags (See 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.

6.8.6 The analyst must select 3-5 quantitation peaks for each detected Aroclor or Toxaphene that do not co-chromatograph with matrix interferences and determine the concentration using each peak separately. Concentrations are calculated using the following equations:

6.8.7 Samples:

$$\text{Concentration mg/Kg} = \frac{(A_x)(V_t)}{(CF)(V_i)(W_x)(1000)}$$

Where:

A_x = Response for the parameter to be measured (height or area).

CF = Calibration factor for the external standard (Section 6.3.5).

V_t = Volume of total extract (uL) (take into account any extra dilution).

V_i = Volume of extract injected (uL).

W_x = Weight of waste diluted (mL).

6.8.8 The laboratory will quantitate each of the selected Aroclor or Toxaphene peaks individually, and determine an average concentration from all of the selected peaks. Quantitation is performed on both columns; the lower value is reported on Form I. See Exhibit B for instructions on completing Form I and Form X.

6.8.9 Detected Aroclors and Toxaphene must be reported as: (1) a concentration between the CRQL and 10^6 x CRQL, or (2) as an estimated value below the CRQL. (See Exhibit B.)

6.8.10 If more than one Aroclor is observed in a sample or if an Aroclor and Toxaphene are observed in the same sample, the laboratory must choose separate congener peaks to quantitate the different analytes. A peak common to both analytes present in the sample must not be used to quantitate both analytes.

6.8.11 The concentrations of the surrogates are calculated using the equation in 6.8.7, where the mean response of the surrogate established during the initial calibration serves as the surrogate calibration factor (CF).

6.8.12 The retention time shift of the surrogates in any standard, sample, control matrix spike, or blank may not excede 0.5%.

EXHIBIT E

Quality Assurance/Quality Control Requirements

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QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

SECTION I

INTRODUCTION AND SCOPE

The Quality Assurance/Quality Control (QA/QC) procedures defined herein must be used by the Contractor when performing analyses according to the methods specified in Exhibit D. This exhibit summarizes the QA/QC procedures and criteria that are mandatory for the performance of the Contract.

The purpose of this document is to provide a uniform set of procedures for the analysis of high-concentration organic samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation.

SECTION II

QA/QC STANDARD OPERATING PROCEDURES

1. General QA/QC Consideration

The Contractor shall have a written QA/QC SOP (Standard Operating Procedure) which describes the inhouse procedures that he employs to guarantee, to the extent possible, the quality of all analysis activities. It should describe the quality assurance and the quality control procedures used during the analysis. Each Contractor should prepare his own SOPs to suit the needs of his organization as he has best determined. The QA/QC SOP should contain the essential elements described in this section.

2. Elements of a QA/QC SOP

2.1 All routine laboratory tasks should have written QA/QC Standard Operating Procedures. Standard Operating Procedures should be detailed documents describing who does what, when, where, how, and why. They shall be sufficiently complete and detailed to ensure:

2.1.1 Data of known quality and integrity are generated.

2.1.2 The loss of data due to out of control conditions is minimized.

2.2 Standard Operating Procedures shall be:

2.2.1 Adequate to establish the traceability of standards, instrumentation, samples, and environmental data.

2.2.2 Simple, so a user with basic education, experience and/or training can properly use them.

2.2.3 Complete enough so the user follows the directions in a stepwise manner.

2.2.4 Consistent with sound scientific principles.

2.2.5 Consistent with current EPA regulations, guidelines, and contract requirements.

2.2.6 Consistent with the instrument manufacturer's specific instruction manuals.

2.3 Standard Operating Procedures will also provide for documentation sufficiently complete to:

2.3.1 Record the performance of all tasks and their results.

2.3.2 Explain the cause of missing data.

2.3.3 Demonstrate the validation of data each time they are recorded, calculated, or transcribed.

2.4 To accomplish these objectives, Standard Operating Procedures should address the major elements upon which the final quality of the contractors work depends. In the following descriptions these six major areas have been divided into subelements, where applicable. These elements include but are not limited to:

2.4.1 Organization and personnel,

2.4.2. Facilities and equipment,

2.4.3. Analytical methodology,

2.4.4 Sample custody procedures,

2.4.5 Quality control, and

2.4.6 Data handling.

3. Organization and Personnel

3.1 QA Policy and Objectives - Each organization should have a written quality assurance policy that should be made known to all organization personnel. Objectives should be established to produce data that meet contract requirements in terms of completeness, precision, accuracy, representativeness, documentation, and comparability. The SOP should require the preparation of a specific QA plan for the analysis.

3.2 QA Organization - The organization and management of the QA function should be described in the Contractor's SOP. Reporting relationships and responsibilities should be clearly defined. A QA Coordinator or Supervisor should be appointed and his responsibilities established. A description of the QC paperwork flow should be available. There should be a clear designation of those who are authorized to approve data and results. Responsibilities for taking corrective action should be assigned to appropriate management personnel.

3.3 Personnel Training - It is highly desirable that there be a training program for employees. This system should include motivation toward producing data of acceptable quality and should involve "practice work" by the new employee. The quality of this work can be immediately verified and discussed by the supervisor, with appropriate corrective action taken.

3.4 Document Control and Revisions - The SOP should include a system for documenting:

3.4.1 Calibration procedures,

3.4.2 Analytical procedures,

3.4.3 Computational procedures,

3.4.4 Quality control procedures,

3.4.5 Bench data,

3.4.6 Operating procedures, or any changes to these procedures, and

3.4.7 Laboratory notebook policy.

3.5 Procedures making revisions to technical procedure or documents must be clearly defined, with the lines of authority indicated. Procedural revisions should be written and distributed to all affected individuals, thus ensuring implementation of changes.

4. Facilities and Equipment

4.1 Procurement and Inventory Procedures - Purchasing guidelines for all equipment and reagents having an effect on data quality should be well defined and documented. Similarly, performance specifications should be documented for all items of equipment having an effect on data quality. Once any item which is critical to the analysis such as an *in situ* instrument, or reagent is received and accepted by the organization, documentation should be retained of the type, age, and acceptance status of the item. Reagents should be dated upon receipt in order to establish their order of use and to minimize the possibility of exceeding their useful shelf life.

4.2 Preventive Maintenance - Preventive maintenance procedures should be clearly defined and written for each measurement system and required support equipment. When maintenance activity is necessary, it should be documented on standard forms maintained in logbooks. A history of the maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory.

5. Analytical Methodology

5.1 Calibration and Operating Procedures - Calibration is the process of establishing the relationship of a measurement system output to a known stimulus. In essence, calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration SOP should include provisions for documentation of frequency, conditions, standards, and records reflecting the calibration history of a measurement system.

5.1.1 The accuracy of the calibration standards is an important point to consider since all data will be in reference to the standards used, An SOP for verifying the accuracy of all working standards against primary grade standards should be routinely followed.

5.2 Feedback and Corrective Action - The SOP should specify the corrective action that is to be taken when an analytical or sampling error is discovered or the analytical system is determined to be out of control. The SOP should require documentation of the corrective action and notification of the analyst of the error and correct procedures.

6. Sample Custody

6.1 Sample custody is a part of any good laboratory or field operation. Where samples may be needed for legal purposes, "chain of custody" procedures, as defined in Exhibit F must be used. However, at a minimum, the following sample custody procedures should be addressed in the QA/QC SOP.

6.2 Chain of Custody in Laboratory Operations

6.2.1 Identification of responsible party to act as sample custodian at the laboratory facility authorized to sign for incoming field samples, obtain documents of shipment (e.g., bill of lading number or mail receipt), and verify the data entered onto the sample custody records.

6.2.2 Provision for a laboratory sample custody log consisting of serially numbered standard lab tracking report sheets.

6.2.3 Specification of laboratory sample custody procedures for sample handling, storage and dispersement of analysis.

7. Quality Control

7.1 Quality Control Procedures - The quality control procedures used during analysis should be described and must conform to those described in Exhibit E. The quality control checks routinely performed during sample analysis include reagent blank analysis to establish analyte levels, control matrix spike and blank sample analysis to determine analytical accuracy. The frequency of these quality assurance checks are defined in the contract. Limits of acceptance or rejection are also defined for analysis and control charts should be used. Confirmation procedures should be described in the SOP.

7.2 Control Checks and Internal Audits - A good SOP will make provision for and describe control checks and internal audits by the Contractor. Several approaches are used for control checks. These include:

7.2.1 Reference Material Analysis - Analytical reference materials are available from several commercial and government sources, or they may be prepared inhouse. The chemical analysis of these materials has been well established. Such materials can be analyzed alongside routine samples and the results used to check the accuracy of analytical procedures.

7.2.2 Blank Analysis - The procedures and the frequency of blank analyses are defined in the contract.

7.2.3 Control Matrix Spike Analysis - The procedures and the frequency of matrix spike analyses are defined in the contract.

7.2.4 Internal Audits - Internal audits should be periodically conducted to evaluate the functioning of the QA SOP. This involves an independent check of the performance of the laboratory analysts to determine if prescribed procedures are closely followed.

8. Data Handling

8.1 Data Handling, Reporting, and Recordkeeping - Data handling, reporting, and recordkeeping procedures should be described. Data handling and reporting includes all procedures used to record data on standard forms, and in laboratory notebooks. The reporting format for different types of bench data should be described and the forms provided. The contents of notebooks should be specified.

8.1.1 Recordkeeping of this type serves at least two useful functions: (1) it makes possible the reanalysis of a set of data at a future time, and (2) it may be used in support of the experimental conclusions if various aspects of the analysis are called into question.

8.2 Data Validation - Data validation procedures, defined ideally as a set of computerized and manual checks applied at various appropriate levels of the measurement process, should be in written form and clearly defined for all measurement systems.

8.2.1 Criteria for data validation must be documented and include limits on:

8.2.1.1 Operational parameters such as GC conditions;

8.2.1.2 Calibration data;

8.2.1.3 Special checks unique to each measurement, e.g., successive values/averages;

8.2.1.4 Statistical tests, e.g. outliers; and

8.2.1.5 Manual checks such as hand calculations.

8.2.2 The limits defined in the contract ensure a high probability of detecting invalid data for either all or the majority of activities. (GC operating conditions, analytical precision, etc. should be recorded on standard forms in a logbook.)

SECTION III

VOLATILES QA/QC REQUIREMENTS

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This section outlines the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of volatile organic target compounds in waste samples. These QC operations are as follows:

- o Documentation of GC/MS Mass Calibration and Abundance Pattern
- o Documentation of GC/MS Response Factor Stability
- o Internal Standard Response and Retention Time Monitoring
- o Method Blank Analysis
- o Surrogate Spike Response Monitoring
- o Control Matrix Spike Analysis

PART 1 - TUNING AND GC/MS MASS CALIBRATION

1. Summary

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

Definition: The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant tune. The time period ends after twelve (12.0) hours has elapsed according to the system clock.

1.1 p-Bromofluorobenzene (BFB)

1.1.1 Each GC/MS system used for the analysis of volatile target compounds must be hardware tuned to meet the abundance criteria listed in Table 1.1 for a maximum of a 50 nanogram injection of BFB. Alternately, add 50 ng of BFB solution to 5.0 mL of reagent water and analyze according to Exhibit D VOA, Section IV. This criterion must be demonstrated daily or for each twelve (12) hour time period, whichever is more frequent. If required, background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction actions that result in spectral distortions for the purpose of meeting the contract specifications are unacceptable.

NOTE: All instrument conditions must be identical to those used in sample analysis, except that a different temperature program will be used.

1.1.2 BFB criteria MUST be met before any standards, samples or blanks are analyzed. Any samples analyzed when tuning criteria have not been met may require reanalysis at no additional cost to the Agency.

1.1.3 Whenever the Contractor takes corrective action which may change or affect the tuning criteria for BFB (e.g., ion source cleaning or repair, etc.), the tune must be verified irrespective of the 12-hour tuning requirements.

TABLE 1.1 BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of mass 95
75	30.0 - 60.0 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95
173	Less than 2.00 percent of mass 95
174	Greater than 50.0 percent of mass 95
175	5.0 - 9.0 percent of mass 174
176	Greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

1.2 Documentation

The Contractor shall provide documentation of the calibration in the form of a bar graph spectrum and as a mass listing.

1.2.1 The Contractor shall complete a Form V (GC/MS Tuning and Mass Calibration) each time an analytical system is tuned. In addition, all standards, samples, control matrix spikes and blanks analyzed during a particular tune must be summarized in chronological order on the bottom of the appropriate Form V. Detailed instructions for the completion of Form V are in Exhibit B, Section III.

PART 2 - CALIBRATION OF THE GC/MS SYSTEM

2. Summary

Prior to the analysis of samples, method blanks or control matrix spikes, and after tuning criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound standards. Once the system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

2.1. Prepare calibration standards as described in Exhibit D VOA, Section IV, to yield the following specific concentrations:

2.1.1 Initial calibration of volatile target compounds is required at 20, 50, 100, 150 and 200 ug/L in water. (This corresponds to 10, 25, 50, 75, and 100 mg/kg in a 1 g sample.) Add working standards of the target compounds (in methanol) to reagent water. Add additional methanol so that the total volume of methanol is 100 uL per 5 mL of aqueous standard. Utilizing the analytical protocol specified in Exhibit D, this will result in 100-1000 total ng analyzed. If a standard analyte saturates at the 200 ug/L concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity of equal to or greater than 5 ug/L (in water), the Contractor must document it in the Case Narrative, and proceed with a four-point initial calibration for that specific analyte.

2.2 The USEPA plans to develop performance-based criteria for response factor data acquired during this program. To accomplish this goal, the Agency has specified the concentration levels for initial calibration and has also specified the specific internal standard to be used on a compound-by-compound basis for quantitation (see Table 2.1). Establishment of standard calibration procedures is necessary and deviations by Contractors will not be allowed.

2.3 Analyze each calibration standard and tabulate the area of the primary characteristic ion (Exhibit D VOA, Table 3) against concentration for each compound including all contract required surrogate compounds. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late eluting compounds usually will have much better agreement.

Using Table 2.1 and Equation 2.1, calculate the relative response factors (RRF) for each compound at each concentration level.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \qquad \text{Equation 2.1}$$

where,

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standards from Table 3 in Exhibit D.

C_{is} = Concentration of the internal standard (ng/uL).

C_x = Concentration of the compound to be measured (ng/uL).

TABLE 2.1 VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET ANALYTES 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	Vinyl Acetate	Tetrachloroethene
Chloroethane	Bromodichloromethane	1,1,2,2-Tetrachloroethane
Methylene Chloride	1,2-Dichloropropane	Toluene
Acetone	trans-1,3-Dichloropropene	Chlorobenzene
Carbon Disulfide	Trichloroethene	Ethylbenzene
1,1-Dichloroethene	Dibromochloromethane	Styrene
1,1-Dichloroethane	1,1,2-Trichloroethane	Total Xylenes
1,2-Dichloroethene (total)	Benzene	Bromofluorobenzene (surr)
Chloroform	cis-1,3-Dichloropropene	
1,2-Dichloroethane	Bromoform	Toluene-d_8 (surr)
2-Butanone		
1,2-Dichloroethane-d_4 (surr)		

(surr) = surrogate compound

2.3.1 Using the average relative response factors (RRF) from the initial calibration, calculate the percent relative standard deviations (%RSD) for compounds labeled on Form VI as Calibration Check Compounds (CCC) and shown in Table 2.2 (see 2.6.2) using Equation 2.2 below. The calibration check compounds for volatiles are: vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, and ethylbenzene.

$$\%RSD = \frac{SD}{\bar{X}} \times 100 \qquad \text{Equation 2.2}$$

where,

RSD = Relative Standard Deviation

SD = Standard Deviation of 5 initial response factors (per compound)

where, $SD = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{X})^2}{n-1}}$

x = mean of 5 initial response factors (per compound)

The %RSD for each individual Calibration Check Compound must be less than or equal to 30.0 percent. This criterion must be met for the initial calibration to be valid.

2.4 A system performance check must be performed to ensure that minimum average relative response factors are met before the calibration curve is used.

2.4.1 For volatiles, the five System Performance Check Compounds (SPCC) are: chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average relative response factor (RRF) for these compounds is 0.300 (0.250 for Bromoform). These compounds typically have RRFs of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. For instance:

- o Chloromethane - this compound is the most likely compound to be lost if the purge flow is too fast.
- o Bromoform - this compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.
- o Tetrachloroethane and 1,1-Dichloroethane - These compounds can be deteriorated by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

2.4.2 The initial calibration is valid only after the criteria for the %RSD for the CCC and the minimum average RRF for the SPCC have both been met. Only after both of these criteria are met can sample analysis begin.

2.5 Documentation

Once the initial calibration is validated, calculate and report the average relative response factor (RRF) and percent relative standard deviation (%RSD) for all target compounds. The Contractor shall

complete and submit Form V (the GC/MS tune for the initial calibration) and Form VI (Initial Calibration Data) for each instrument used to analyze samples under this protocol. Detailed instructions for completion of Forms V and VI are found in Exhibit B, Section III.

2.6 Continuing Calibration

A calibration standard(s) containing all volatile target compounds, including all required surrogates, must be performed each twelve (12) hours during analysis (see definition of twelve-hour time period, paragraph 1 of this Section). Compare the relative response factor data from the standards run each twelve hours with the average relative response factor from the initial calibration for a specific instrument. A system performance check must be made each twelve hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration (Form VI). If the minimum relative response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins.

2.6.1 Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatography system. This check must be met before analysis begins. The minimum relative response factor (RRF) for volatile System Performance Check Compounds (SPCC) is 0.300 (0.250 for Bromoform).

2.6.2 Calibration Check Compounds (CCC)

After the system performance check is met, Calibration Check Compounds (CCC) listed in Table 2.2 are used to check the validity of the initial calibration. Calculate the percent difference using Equation 2.3.

$$\% \text{ Difference} = \frac{\overline{RRF}_I - RRF_c}{\overline{RRF}_I} \times 100 \qquad \text{Equation 2.3}$$

where,

$\overline{RRF}_I$ = average relative response factor from initial calibration.

RRF_c = relative response factor from current verification check standard.

2.6.2.1 If the percent difference for any compound is greater than 20%, the Contractor should consider this a warning limit. If the percent difference for each CCC is less than or equal to 25.0%, the initial calibration is assumed to be valid. If

the criteria are not met (>25.0% difference), for any one calibration check compound, corrective action MUST be taken. Problems similar to those listed under SPCC could affect these criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five point calibration MUST be generated. These criteria MUST be met before sample analysis begins.

TABLE 2.2 VOLATILE CALIBRATION CHECK COMPOUNDS (CCC)

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

2.6.3 Concentration Levels for Continuing Calibration Check

The USEPA plans to evaluate the long term stability of relative response factors during this program. Standardization among contract laboratories is necessary to reach these long term goals. Along with contract specified concentrations for initial calibration, the USEPA is requiring specific concentrations for each continuing calibration standard(s).

2.6.3.1 The concentration for each volatile target compound in the continuing calibration standard(s) is 50 ug/L.

2.7 Documentation

The contractor shall complete and submit a Form VII for each GC/MS system utilized for each twelve hour time period. Calculate and report the relative response factor and percent difference (%D) for all compounds. The percent difference (%D) for each CCC compound must be less than or equal to 25.0 percent. Ensure that the minimum RRF for volatile SPCCs is 0.300 (0.250 for bromoform). Additional instructions for completing Form VII are found in Exhibit B, Section III.

PART 3 - METHOD BLANK ANALYSIS

3. Summary

The method blank is prepared by adding 100 μL of surrogate to 10 mL of methanol. A volume of the methanol is added to 5 mL of reagent

water and carried through the entire analytical scheme. The method blank volume must be approximately equal to the sample volumes being processed.

3.1 Method blank analysis must be performed at the following frequency:

3.1.1 For the analysis of volatile target compounds, a method blank analysis must be performed once for each twelve hour time period. The twelve hour period begins with the injection of BFB. The method blank must be analyzed after the calibration standard(s).

3.2 It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be minimized.

3.2.1 For the purposes of this protocol, an acceptable laboratory method blank should meet the criteria of paragraphs 3.2.1.1 and 3.2.1.2.

3.2.1.1 A method blank for volatile analysis must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL from Exhibit C) of methylene chloride, acetone, 2-butanone, and toluene.

3.2.1.2 For all other target compounds not listed above, the method blank must contain less than or equal to the Contract Required Quantitation Limit of any single target analyte.

3.2.2 If a laboratory method blank exceeds this criterion, 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 Case Narrative (Exhibit B).

3.3 Documentation

The Contractor shall report results of method blank analysis using the Organic Analysis Data Sheet (Form I) and the form for tentatively identified compounds (Form I, TIC). In addition, the samples associated with each method blank must be summarized on Form IV (Method Blank Summary). Detailed instructions for the completion of these forms can be found in Exhibit B, Section III.

3.3.1 The Contractor shall report ALL sample concentration data as UNCORRECTED for blanks.

PART 4 - SURROGATE SPIKE (SS) ANALYSIS

4. Summary

Surrogate standard determinations are performed on all samples and blanks. All blanks are fortified with surrogate spiking compounds before extraction in order to monitor preparation and analysis of samples.

4.1 Each sample, blank, and control matrix spike must be fortified with the surrogate compounds (shown in Table 4.1) prior to extraction and purging. Performance based criteria are generated from laboratory results. Therefore, deviations from the spiking protocol will not be permitted.

TABLE 4.1 SURROGATE SPIKING LEVELS AND RECOVERY LIMITS

Compound	Concentration*	Recovery Limit(%)**
Toluene-d_8	50 ug	50 - 160
4-Bromofluorobenzene	50 ug	50 - 160
1,2-Dichloroethane-d_4	50 ug	50 - 160

*In sample extract at the time of injection (before any optional dilutions).

**These limits are mandatory for method blanks and advisory for samples and control matrix spikes. They are not used to determine if a sample or a control matrix spike should be reanalyzed.

4.2 Surrogate spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the contract required recovery limits listed in Table 4.1.

4.3 Treatment of surrogate spike recovery information shall be according to paragraphs 4.3.1 and 4.3.2.

4.3.1 Method Blank Surrogate Spike Recovery

The laboratory must take actions listed below if recovery of any one surrogate compound in the volatiles fraction of the method blank is outside of the contract required surrogate spike recovery limits (Table 4.1).

4.3.1.1 Check calculations to ensure there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc; also, check instrument performance.

4.3.1.2 Recalculate or re-inject/repurge the blank if steps in 4.3.1.1 indicate this may produce compliant surrogate recoveries.

4.3.1.3 Re-extract and re-analyze the blank and any associated samples.

4.3.1.4 If the measures listed in 4.3.1.1 thru 4.3.1.3 fail to correct the problem, the analytical system must be considered out of control. The problem MUST be corrected before continuing.

This may mean recalibrating the instrumentation but it may also mean more extensive action. The specific corrective action is left up to the GC/MS operator. When surrogate recovery(ies) in the blank is outside of contract required windows, all samples associated with that blank MUST be reanalyzed at no additional cost to the Agency.

4.3.2 Sample Surrogate Spike Recovery

When the recovery of any one surrogate compound in the volatiles fraction of the sample or control matrix spike is outside of the contract surrogate spike recovery limits (Table 4.1), it is the responsibility of the Contractor to establish that the deviation is not due to laboratory problems. The surrogate spike recovery windows for high concentration samples and control matrix spikes are advisory at this time.

4.4 Documentation

The Contractor is required to report surrogate recovery data for the following:

- Method Blank Analyses
- Sample Analyses
- Control Matrix Spike Analyses

The surrogate spike recovery data are summarized on the Surrogate Spike Percent Recovery Summary (Form II). Detailed instructions for the completion of Form II are in Exhibit B, Section III.

PART 5 - CONTROL MATRIX SPIKE ANALYSIS

5. Summary

In order to evaluate the efficiency of the analytical methodology, the USEPA has developed standards to be used for control matrix spike analysis. These compounds are subject to change depending upon availability and suitability for use as control matrix spikes.

5.1 The Contractor shall perform one control matrix spike sample analysis for each Case received, or for each 20 single phase units, or each 14 calendar day period during which single phase units in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group) whichever is most frequent.

5.2 Use the following compounds to prepare control matrix spiking solutions (Exhibit D, Section II, 5.5.1), which require that a uniform amount of control matrix spiking solution be added to a control matrix aliquot prior to extraction. The spiking solution contains the following compounds: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

Analyze the control matrix spike and calculate the individual component recoveries using Equation 5.1.

$$\text{Control Matrix Spike Percent Recovery} = \frac{CR}{SA} \times 100 \qquad \text{Equation 5.1}$$

where,

CR = Control matrix spike concentration

SA = Concentration of spike added from spiking mix

5.3 Documentation

Matrix spike recovery limits are 60% - 150% for all of the volatile control matrix spike compounds. These limits are for advisory purposes only. (They should not be used to determine reanalysis of a control matrix spike.) When sufficient multi-laboratory data become available, standard recovery limits will be calculated.

The control matrix spike percent recoveries shall be summarized on Form III. These values will be used by EPA to establish performance based QC recovery limits. Complete instructions for the completion of Form III can be found in Exhibit B, Section III.

PART 6 - SAMPLE ANALYSIS

6. Summary

6.1 Samples can be analyzed upon successful completion of the initial calibration analysis. When twelve (12) hours have elapsed since the initial calibration was completed, it is necessary to conduct an

instrument tune and continuing calibration analysis. Any major system maintenance, such as a source cleaning or installation of a new column, will necessitate a retune and recalibration (See Initial Calibration, Part 3).

6.1.1 Internal Standards Evaluation

Internal standard response and retention times in all samples must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds, the chromatographic system must be inspected for malfunctions and corrections made as required. If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%), from the latest daily (12 hour time period) calibration standard, the mass spectrometric 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 single phase unit, method blank and control matrix. The criteria are described in detail in the instructions for Form VIII, High Concentration Internal Area Summary (See Exhibit B). Breaking off 1 foot of the column or cleaning the injector sleeve will often improve high end sensitivity for the late eluting compounds.

Poor injection technique can also lead to variable IS ratios. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

6.1.1.1 If after reanalysis, 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, only submit data from the analysis with EICP's with the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

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

6.1.2 Each analytical run must also be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that

compound. The initial method calibration (Part 2) requires that the system should not be saturated for high response compounds at 200 ug/L for VOA target compounds.

6.1.2.1 If any compound in any sample exceeds the initial calibration range, that sample must be diluted, the internal standard concentration readjusted, and the sample re-injected, as described in specific methodologies in Exhibit D VOA. Secondary ion quantitation is only allowed when there are sample matrix interferences with the primary ion.

6.1.2.2 If the dilution of the sample extract causes any compound detected in the first analysis to be undetectable in the second analysis, then the results of both analyses shall be reported on separate Forms (I), according to instructions in Exhibit B.

6.1.3 Qualitative Analysis

The target compounds listed in Exhibit C, shall be identified 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. 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 (Exhibit D).

6.1.3.1 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 on the same shift as the sample.

6.1.3.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. The BFB tuning requirements listed in Part 1 of this Section must be met on that same GC/MS.

6.1.3.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

6.1.3.3.1 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.

6.1.3.3.2 The relative intensities of ions specified in the above paragraph must agree within ±20% between the standard and sample spectra.

6.1.3.3.3 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. 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. The verification process should favor false positives.

6.1.3.4 If a compound cannot be verified by all of the criteria in 6.1.3.3, but in the technical judgement of the mass spectral interpretation specialist the identification is correct, the Contractor shall report the identification and proceed with the quantitation.

6.1.3.5 A library search shall be executed for nonsurrogate and non-target sample components for the purpose of tentative identification. For this purpose, the 1985 or most recent available version of the National Bureau of Standards Mass Spectral Library, containing 42,261 spectra should be used.

6.1.4 Quantitation

6.1.4.1 Target compounds identified shall be quantitated by the internal standard method. The internal standards used shall be the ones assigned in Table 2.1 of this Section. The EICP area of characteristic ions of target analytes are used (Exhibit D).

6.1.4.2 An estimated concentration for non-target compounds tentatively identified shall be quantitated by the internal standard method. For quantification, the nearest internal standard free of interferences must be used.

6.1.4.3 Calculate surrogate standard recovery for all surrogate compounds, on all single phase units, method blanks, and control matrix spikes. If recovery is within contractual or advisory limits, report on Form II. If recovery is outside contractual limits for the method blank, take specific steps listed in Section 4.3, Surrogate Spike Recoveries.

6.1.4.4 Calculate control matrix spike present recovery for all spiked compounds. Report results on Form III.

6.1.5 Reporting and Deliverables

Refer to Exhibit B of this document for specific details on contract deliverables and reporting formats. Exhibit B contains specific instructions for completing all required forms, as well as a detailed itemization of reporting and deliverables requirements.

SECTION III

EXTRACTABLES QA/QC REQUIREMENTS

TABLE OF CONTENTS

This Section outlines the minimum Quality Control (QC) operations necessary to satisfy the analytical requirements associated with the determination of high concentration extractable target compounds in waste samples. These QC operations are as follows:

- o Documentation of GC/MS Mass Calibration and Abundance Pattern
- o Documentation of GC/MS Response Factor Stability
- o Internal Standard Response and Retention Time Monitoring
- o Method Blank Analysis
- o Surrogate Spike Response Monitoring
- o Control Matrix Spike Analysis

Part 1 - Tuning and GC/MS Mass Calibration

1. Summary

Prior to initiating any on going data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of Decafluorotriphenylphosphine (DFTPP). The ion abundance criteria for each calibration compound MUST be met before any single phase units, method blanks, or control matrix spikes can be analyzed.

1.1 Decafluorotriphenylphosphine (DFTPP)

1.1.1 Each GC/MS system used for the analysis of semi-volatile or pesticide target compounds must be hardware tuned to meet the abundance criteria listed in Table E-1 for a 50 ng injection of decafluorotriphenylphosphine (DFTPP). DFTPP may be analyzed separately or as part of the calibration standard. The criteria must be demonstrated daily or for each twelve (12) hour period, whichever is more frequent. DFTPP must be injected to meet this criterion. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the objectives of Quality Assurance and are unacceptable.

NOTE: All instrument conditions must be identical to those in sample analyses, except that a different (faster) temperature program may be used.

1.1.2 Whenever the Contractor takes corrective action which may change or affect the tuning criteria of DFTPP (e.g., ion source cleaning or repair, etc.), the tune must be verified irrespective of the twelve (12) hour tuning requirements.

1.1.3 Any samples analyzed when tuning criteria have not been met will require reanalysis at no additional cost to the Agency. The twelve (12.0) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration

criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of compliant tune. The time period ends after twelve (12) hours has elapsed according to the GC/MS system clock.

1.2 Documentation

Documentation of the calibration must be provided in the form of a bar graph plot and as a mass listing.

1.2.1 The Contractor shall complete a Form V (High Concentration Extractable GC/MS Tuning and Mass Calibration) each time an analytical system is tuned. In addition, all single phase units, standards, method blanks, and control matrix spikes analyzed during a particular tune must be summarized on the bottom of the appropriate Form V. Detailed instructions for the completion of Form V are found in Exhibit B, Section III.

TABLE E-1. DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30.0 to 60.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Mass 69 relative abundance
70	Less than 2.0 percent of mass 69
127	40.0 to 60.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance
199	5.0 to 9.0 percent of mass 198
275	10.0 to 30.0 percent of mass 198
365	Greater than 1.00 percent of mass 198
441	Present but less than mass 443
442	Greater than 40.0 percent of mass 198
443	17.0 to 23.0 percent of mass 442

Part 2 - Calibration of the GC/MS System

2. Summary

Prior to the analysis of samples, method blanks, or control matrix spikes, and after tuning criteria have been met, the GC/MS system must be initially calibrated at a minimum of three concentrations to

determine the linearity of response utilizing target compound standards. Once the system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

2.1 Prepare calibration standards as described in Exhibit D, Section 4, to yield the following specific concentrations:

2.1.1 Extractable Target Compounds

Initial calibration of <u>all</u> extractable target compounds except PCBs are required at 50, 80 and 160 total nanograms. Initial calibration of PCB homolog: one solution containing mono- through heptachlorobiphenyl are required at 10, 30 and 50 total nanograms. Octa-, nona- and decachlorobiphenyl are required at 20, 60 and 100 total nanograms (due to their low responses).

2.2 The USEPA plans to develop performance based criteria for response factor data acquired during this program. To accomplish this goal, the Agency has specified both the concentration levels for initial calibration and has also specified the internal standard to be used on a compound by compound basis for quantitation. Establishment of standard calibration procedures is necessary and deviations by contractor laboratories will not be allowed.

2.3 Analysis of Calibration Standard

2.3.1 Analyze each calibration standard and tabulate the area of the primary characteristic ion (Exhibit D, Table 1) against concentration for each compound including all contract required surrogate compounds. The relative retention times of each compound in each calibration run should agree within ±0.05 RRT. Late eluting compounds usually will have much better agreement. Using Table E-3, calculate the relative response factors (RRF) for each target compound at each concentration level using Equation 3.1.

Equation 3.1

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specified internal standards from Table E-3.

C_{is} = Concentration of the internal standard (ng/uL).

C_x = Concentration of the compound to be measured (ng/uL).

2.3.2 Using the relative response factors (RRF) from the initial calibration, calculate the percent relative standard deviations (% RSD) for compounds labeled as Calibration Check Compounds using Equation 3.2.

Equation 3.2

$$\% \text{ RSD} = \frac{SD}{\bar{x}} \times 100$$

Where:

RSD = Relative Standard Deviation.

SD = Standard Deviation of 3 Initial Relative Response Factors (Per Compound).

Where:

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \bar{X})^2}{n-1}}$$

$\bar{x}$ = Mean of 3 Initial Relative Response Factors (Per Compound).

The % RSD for each individual Calibration Check Compound must be less than 30.0 percent. This criteria must be met for the initial calibration to be valid.

2.4 A system performance check must be performed to ensure minimum average relative response factors are met before the calibration curve is used.

2.4.1 For extractables, the System Performance Check Compounds (SPCC's) are:

N-Nitroso-di-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4-Nitrophenol. The minimum acceptable average relative response factor (RRF) for these compounds is 0.050. These compounds (SPCC's) typically have very low RRF's (0.1-0.2) and tend to decrease in response as the chromatographic system or the standard material begin to deteriorate. These compounds are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

2.4.2 The initial calibration is valid only after both the % RSD for CCC compounds and the minimum RF for SPCC have been met. Only after both these criteria are met can sample analysis begin.

2.5 Documentation

Once the initial calibration is validated, calculate and report the average relative response factor (RRF) and percent relative standard deviation (% RSD) for all target compounds. The laboratory is required to submit a Form VI (Initial Calibration Data) for each instrument used to analyze samples under this protocol. Detailed instructions for completion of Form VI are found in Exhibit B, Section III.

2.6 Continuing Calibration

The analysis of a calibration standard(s) containing all required target compounds, including all required surrogates, must be performed each twelve (12) hours during analysis. Compare the response factor data from the standards each twelve hours with the average relative response factor from the initial calibration for a specific instrument. A system performance check must be made each twelve (12) hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration (Form VI). If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins.

2.6.1 Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatography system. This check must be met before analysis begins. The minimum relative response factor (RRF) for extractable System Performance Check Compounds (SPCC) is 0.050.

2.6.2 Calibration Check Compounds (CCC)

After the system performance check is met, Calibration Check Compounds listed in Table E-2 are used to check the validity of the initial calibration. Calculate the percent difference using Equation 3.3.

Equation 3.3

$$\% \text{ Difference} = \frac{\overline{RRF}_I - RRF_c}{RRF_I} \times 100$$

Where:

$\overline{RRF}_I$ = Average Relative Response Factor from Initial Calibration.

RRF_c = Relative Response Factor from Current Verification Check Standard.

2.6.2.1 If the percent difference for any compound is greater than 20.0%, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25.0%, the initial calibration is assumed to be valid. If the criteria are not met (greater than 25.0% difference), for any one calibration check compound, corrective action MUST be taken. Problems similar to those listed under SPCC could affect this criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five point calibration MUST be generated. This criteria MUST be met before sample analysis begins.

TABLE E-2. Extractable Calibration Check Compounds

Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

2.6.3 Concentration Levels for Continuing Calibration Check

The USEPA plans to evaluate the long term stability of response factors during this program. Standardization among contract laboratories is necessary to reach these long term goals. Along with contract specified concentrations for initial calibration, the USEPA is requiring specific concentrations for each calibration standard(s).

2.6.3.1 The concentration for each extractable target compound except PCBs in the continuing calibration standard(s) is 80 total nanograms. For the continuing calibration of the PCBs, one solution containing mono-through heptachlorobiphenyl is required at 30 total nanograms, octa-, nano-, and decachlorobiphenyl are required at 60 total nanograms for the continuing calibration solution.

2.7 Documentation

The laboratory is required to complete and submit a Form VII for each GC/MS system utilized for each twelve (12) hour time period. Calculate and report the response factor and percent difference (% D) for all compounds. Ensure the minimum RF for SPCC's is 0.050. The percent difference (% D) for each CCC

compound must be less than 25.0 percent. Additional instructions for completing Form VII are found in Exhibit B, Deliverables, Section III.

TABLE E-3. Standards With Corresponding Extractable Target Compounds Analytes Assigned For Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	Benzoic acid	2-Nitroaniline
Benzyl alcohol	bis(2-Chloroethoxy)methane	Dimethyl phthalate
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene
2-Methylphenol	1,2,4-Trichlorobenzene	3-Nitroaniline
bis(2-Chloroisopropyl)ether	Naphthalene	Acenaphthene
4-Methylphenol	4-Chloroaniline	2,4-Dinitrophenol
N-Nitroso-di-n-propylamine	Hexachlorobutadiene	4-Nitrophenol
Hexachloroethane	4-Chloro-3-methylphenol	Dibenzofuran
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene
Phenol-d5 (surr)	Nitrobenzene-d5 (surr)	2,6-Dinitrotoluene
		Diethyl phthalate
		4-Chlorophenyl-phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr)
		2,4,6-Tribromophenol (surr)

surr = surrogate compound

TABLE E-3. (Continued)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
N-nitrosodiphenylamine	Butylbenzyl phthalate	Benzo(b)fluoranthene
1,2-Diphenylhydrazine	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Benzo(a)anthracene	Benzo(a)pyrene
Hexachlorobenzene	bis(2-ethylhexyl)phthalate	Indeno(1,2,3-cd) Pyrene
Pentachlorophenol	Chrysene	Dibenz(a,h)anthracene
Phenanthrene	Terphenyl-d14 (surr)	Benzo(g,h,i)perylene
Anthracene	gamma-Chlordane	
Di-n-butyl phthalate	Endosulfan I	
Fluoranthene	Alpha-Chlordane	
alpha-BHC	Dieldrin	
beta-BHC	4,4'-DDE	
gamma-BHC	Endrin	
delta-BHC	Endosulfan II	
Heptachlor	4,4'-DDD	
Aldrin	Endosulfan sulfate	
Heptaclor epoxide	4,4'-DDT	
Endrin ketone	Methoxychlor	
	Monochlorobiphenyl	
	Dichlorobiphenyl	
	Trichlorobiphenyl	
	Tetrachlorobiphenyl	
	Pentachlorobiphenyl	
	Hexachlorobiphenyl	
	Heptachlorobiphenyl	
	Octachlorobiphenyl	
	Nonachlorobiphenyl	
	Decachlorobiphenyl	

surr = surrogate compound

Part 3 - Method Blank Analysis Summary

3. Summary

A method blank is an aliquot of the reagent(s) that is carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank weight must be approximately equal to the sample weights being processed.

3.1 Method blank analysis must be performed at the following frequency:

3.1.1 For the analysis of extractable target compounds, a method blank analysis must be performed for each case received, or for each 20 single phase units, or whenever single phase units are extracted, whichever is most frequent. The method blank associated with a specific set or group of single phase units must be analyzed on each GC/MS or GC system used to analyze that specific group or set.

3.1.2 It is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be minimized.

3.1.2.1 For the purposes of this protocol, an acceptable laboratory method blank must meet the criteria of paragraphs 3.1.2.1.1 and 3.1.2.1.2.

3.1.2.1.1 A method blank for extractable analysis must contain less than two times (2X) the Contract Required Quantitation Limit (CRQL from Exhibit C) of phthalate esters in the TCL.

3.1.2.1.2 For all other extractable target compounds not listed above, the method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL) of any target analyte.

3.2.2 If a laboratory method blank exceeds criteria, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken before further sample analysis proceeds. All single phase units processed with a method blank that is out of control (i.e., contaminated) must be reextracted and reanalyzed at no additional cost to the Agency. The Laboratory Manager, or his designate, must address problems and solutions in the Case Narrative (Exhibit B).

3.3 Documentation

Results of extractable method blank analysis shall be reported using the High Concentration Extractable Analysis Data Sheet (Form I), and the tentatively identified compounds (Form I, TIC). In addition, the single phase units associated with each method blank must be summarized on Form IV (High Concentration Extractable Method Blank Summary). Specific instructions for the completion of these forms can be found in Exhibit B (Reporting and Deliverables), Section III.

3.3.1 The Contractor will report ALL sample concentration data as UNCORRECTED for blanks. It shall be the responsibility of the EPA evaluator, and/or data auditor, to correct analyte concentrations for concentrations detected in the method blank(s). It is the Contractor's responsibility to ensure the proper number of method blanks are analyzed and the data properly reported.

Part 4 - Surrogate (SS) Analysis

4. Summary

Surrogate standard determinations are performed on all single phase units, method blanks, and control matrix spikes. ALL single phase units, method blanks, and control matrix spikes are fortified with surrogate spiking compounds before extraction in order to monitor preparation and analysis of samples.

4.1 Each single phase unit (including control matrix spike and method blank) is spiked with surrogate compounds prior to extraction. The surrogate spiking compounds shown in Table E-4 are used to fortify each single phase unit with the proper concentrations. Performance based criteria are generated from laboratory results. Therefore deviations from spiking protocol will not be permitted.

TABLE E-4. Extractable Surrogate Spiking Compounds

Compound	Amount in Sample Extract (before any optional dilutions)
Nitrobenzene-d_5	50 ug
2-Fluorobiphenyl	50 ug
p-Terphenyl-d_{14}	50 ug
Phenol-d_5	100 ug
2-Fluorophenol	100 ug
2,4,6-Tribromophenol	100 ug

4.2 Surrogate recovery must be evaluated for acceptance by determining whether the concentration (measured as percent recovery) falls inside the advisory recovery limits listed in Table E-5.

4.3 Surrogate spike recovery information is treated according to paragraphs 4.3.1 through 4.3.1.2.

4.3.1 Method Blank Surrogate Recovery

When the surrogate recovery of any one extractable surrogate compound is outside of the contract required surrogate recovery limits (listed in Table E-5) for a method blank, the laboratory must take the following actions:

4.3.1.1 Check calculations to assure there are no errors. Check internal standard and surrogate spiking solutions for degradation, contamination, etc. Also, check instrument performance.

4.3.1.2 Recalculate and/or reinject the extract if steps in 4.3.1.1 reveal the cause of the non-compliant surrogate recoveries.

4.3.1.3 If the measures listed above fail to correct the problem, the analytical system must be considered out of control. The problem MUST be corrected before continuing. This may mean recalibration of the instrumentation but it may also mean more extensive action. The specific corrective action is left up to the GC/MS operator. The method blank and all associated single phase units, including any control matrix spikes must be re-extracted and re-analyzed at no additional cost to the Agency.

TABLE E-5. Surrogate Recovery Limits*

Surrogate Compound	% Recovery
Nitrobenzene-d_5	20-140
2-Fluorobiphenyl	20-140
p-Terphenyl-d_{14}	20-150
Phenol-d_5	20-140
2-Fluorophenol	20-140
2,4,6-Tribromophenol	10-140

* Mandatory for method blanks. Advisory for single phase units and control matrix spikes.

4.3.2 Sample Surrogate Recovery

When the surrogate recovery of any one extractable surrogate compound is outside of the advisory recovery limits (listed in Table E-5) for any single phase unit (including the control matrix spike(s)), the laboratory must take the following actions:

4.3.2.1 Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

4.3.2.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

4.3.2.3 If surrogates cannot be detected due to dilution factors, this requirement (4.3.2.1) need not be met, report the value(s) as (DL) as described in Exhibit B.

4.4 Documentation

The Contractor is required to report surrogate recovery data for the following:

- Method Blanks
- Control Matrix Spikes
- ALL Single Phase Units

The surrogate spike recovery data is summarized on the High Concentration Extractable Surrogate Spike Percent Recovery Summary (Form II). Complete instructions for the completion of Form II can be found in Exhibit B, Section III.

Part 5 - Control Matrix Spike

5. Summary

In order to evaluate the efficiency of the analytical methodology, the USEPA has developed the standard mixtures listed in Table E-6 to be used for control matrix spike analysis. These compounds are subject to change depending upon availability and suitability for use as control matrix spikes. The control matrix spike analysis is not to be misinterpreted as a matrix spike or matrix spike duplicate analysis.

5.1 Frequency of Analysis

The Contractor shall perform one contol matrix spike sample analysis for each Case received, or for each 20 single phase units, or each 14 calendar day period during which single phase units in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group) whichever is most frequent.

5.2 Use the compounds listed in Table E-6 to prepare control matrix spiking solutions according to protocols described in Exhibit D, Section II, 4.8. The analytical protocols in Exhibit D, require that a uniform amount of control matrix spiking solution be added to the control matrix prior to extraction.

5.3 Individual component recoveries of the control matrix spike are calculated using Equation 5.1.

$$\text{Matrix Spike Percent Recovery} = \frac{SSR - SR}{SA} \times 100 \qquad \text{Equation 5.1}$$

Where:

SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from Spiking Mix

TABLE E-6.Control Matrix Spiking Solutions*

Base/Neutrals	Acids	Pesticides
1,4-Dichlorobenzene	Phenol	Heptachlor
N-Nitroso-di-n-propylamine	2-Chlorophenol	Dieldrin
1,2,4-Trichlorobenzene	4-Chloro-3-methylphenol	
Acenaphthene	4-Nitrophenol	
2,4-Dinitrotoluene	Pentachlorophenol	
Pyrene		

*Base/Neutrals and Acids are combined in one solution. The pesticides are prepared in a separate solution.

5.4 Documentation

The control matrix spike percent recoveries shall be summarized on Form III. These values will be used by EPA to periodically update existing performance based QC recovery limits.

See Exhibit B, Deliverables, Section III, for complete instructions on the completion of Form III.

TABLE E-7.Control Matrix Spike Recovery Limits*

Compound Class	Matrix Spike Compound	% Recovery*
BN	N-Nitroso-di-n-propylamine	30-140
BN	1,4-Dichlorobenzene	30-140
BN	1,2,4-Trichlorobenzene	30-140
BN	Acenaphthene	30-140
BN	2,4-Dinitrotoluene	30-140
BN	Pyrene	30-140
Acid	Phenol	10-120
Acid	2-Chlorophenol	10-120
Acid	4-Chloro-3-methylphenol	10-120
Acid	4-Nitrophenol	10-120
Acid	Pentachlorophenol	10-120
Pesticide	Heptachlor	30-140
Pesticide	Dieldrin	30-140

* These limits are for advisory purposes only. They are not to be used to determine if a sample should be reanalyzed. When sufficient data are available, standard limits will be calculated.

Part 6 - Quality Control

6. Summary

6.1 Samples can be analyzed upon successful completion of the initial QC activities. When twelve (12) hours have elapsed since the initial QC was completed, it is necessary to conduct an instrument tune and calibration check analysis (described in Part 1 of this Exhibit). Any major system maintenance, such as a source cleaning or installation of a new column, will necessitate a retune and recalibration (See Initial Calibration, Section III, Part 2).

6.1.1 Internal Standards Evaluation

Internal standard response and retention times in all samples must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds, the chromatographic system must be inspected for malfunctions and corrections made as required. If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%), from the latest daily (12 hour time period) calibration standard, the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each single phase unit, method blank, and control matrix. The criteria are described in detail in the instructions for Form VIII, High Concentration Internal Area Summary (See Exhibit B). Breaking off 1 foot of the column or cleaning the injector sleeve will often improve high end sensitivity for the late eluting compounds.

Poor injection technique can also lead to variable IS ratios. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

6.1.1.1 If, after reanalysis, 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, only submit data from the analysis with EICP's within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

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

6.1.2 Each analytical run must also be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. The initial method calibration (Part 2) requires that the system should not be saturated for high response compounds at 160 nanograms.

6.1.2.1 If any compound in any sample exceeds the initial calibration range, that sample must be diluted, the internal standard concentration re-adjusted, and the sample re-injected, as described in Exhibit D, Section III, 5.2. Secondary ion quantitation is only allowed when there are sample matrix interferences with the primary ion.

6.1.2.2 If the dilution of the sample extract causes any compound detected in the first analysis to be undetectable in the second analysis, then the results of both analyses shall be reported on Separate Forms I, according to instructions in Exhibit B.

6.1.3 Qualitative Analysis

The target compounds listed in Exhibit C, shall be identified 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. 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 (Exhibit D).

6.1.3.1 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.05 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample.

6.1.3.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. The DFTPP tuning requirements listed in Exhibit E, Part 1 must be met on the Contractor's same GC/MS.

6.1.3.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

6.1.3.3.1 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 same spectrum.

6.1.3.3.2 The relative intensities of ions specified in the above paragraph must agree within ±20% between the standard and sample spectra.

6.1.3.3.3 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. 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. The verification process should favor false positives (Exhibit D).

6.1.3.4 A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the most recent available version of the EPA/NBS Mass Spectral Library should be used.

6.1.4 Quantitation

6.1.4.1 Target components identified shall be quantitated by the internal standard method. The internal standard used shall be the ones assigned in Table E-3, Exhibit E. The EICP area of characteristic ions of target analytes are used (Exhibit D tables D-1, D-5).

6.1.4.2 An estimated concentration for non-target components tentatively identified shall be quantitated by the internal standard method. For quantification, the nearest internal standard free of interferences must be used.

6.1.4.3 Calculate surrogate standard recovery for all surrogate compounds, on all single phase units, method blanks, and control matrix spikes. If recovery is within contractual or advisory limits, report on Form II (See Exhibit B). If recovery is outside contractual limits for the method blank, take specific steps listed in Exhibit E, Surrogate Spike Recoveries.

6.1.4.4 Calculate control matrix spike percent recovery for all spiked compounds. Report results on Form III.

6.1.5 Reporting and Deliverables

Refer to Exhibit B of this document for specific details on contract deliverables and reporting formats. Exhibit B contains specific instructions for completing all required Forms, as well as a detailed itemization of reporting and deliverables requirements.

EXHIBIT E

AROCLORS/TOXAPHENE QA/QC REQUIREMENTS

1. Summary

This section outlines the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of Aroclors and Toxaphene in high concentration samples using the analytical method described in Exhibit D. The QC operations described below apply to analyses performed on both GC columns of all instruments.

Initial QC requirements:

- o Determination of potential quantitation peaks for each analyte and determination of their Absolute Retention Times (RT) and calibration factors.

Periodic QC requirements:

- o Continued monitoring of RTs and calibration factors.
- o Instrument blank analysis.
- o Method blank analysis.
- o Control matrix spike analysis.

Additional QC requirements:

- o Surrogate recoveries are reported for all samples, blanks, and control matrix spike analyses.
- o Retention time shifts of the surrogates are reported for the analyses of all standards, samples, control matrix spikes and blanks.

These QC operations are designed to facilitate comparison of analytical data from different laboratories. These requirements do not release the analytical laboratory from maintaining their own checks on the performance of their instruments. These checks may include, but are not limited to, determining detector standing current, monitoring the number of theoretical plates per column and establishing the limits of quantitation for each of the analytes.

2. DEFINITIONS

2.1 Instrument Blank

The instrument blank is a solution containing the surrogates Tetrachloro-meta-xylene and Decachlorobiphenyl (20 ng/mL) in hexane. This solution must be injected no less than once every 12 hours on each GC column used in order to demonstrate that none of the Aroclors or Toxaphene listed in the Exhibit C are detected at $\geq$ 0.5 the contract required quantitation limit (CRQL).

2.2 Method Blank

A method blank is 500 mg of corn oil spiked with the surrogates (20 ng/mL) that is carried through the entire analytical scheme given in Exhibit D. An acceptable method blank is required for each full or

partial set of 20 samples analyzed in a sample delivery group (SDG). An acceptable method blank has none of the Aroclors or Toxaphene listed in Exhibit C at > CRQL.

2.3 Control Matrix Spike

A control matrix spike is prepared by spiking an aliquot of corn oil with the Aroclor 1254 spiking solution (25 ug/mL). The Contractor shall perform one control matrix spike sample analysis for each Case received, or for each 20 single phase units, or each 14 calendar day period during which single phase units in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group) whichever is most frequent.

3. INITIAL CALIBRATION

3.1 Prior to analysis of samples, it is necessary to run a successful calibration sequence using this sequence of injections:

TABLE E.1. INITIAL CALIBRATION

Injection No.	Concentration	Aroclor	No. of Potential Quantitation Peaks
1	Blank	–	–
2	Resolution Check	–	–
3	Low	1221	4
4	Medium	1221	4
5	High	1221	4
6	Low	1232	4
7	Medium	1232	4
8	High	1232	4
9	Low	1242	5
10	Medium	1242	5
11	High	1242	5
12	Low	1248	5
13	Medium	1248	5
14	High	1248	5
15	Low	1254	5
16	Medium	1254	5
17	High	1254	5
18	Low	1016/1260	5/5
19	Medium	1016/1260	5/5
20	High	1016/1260	5/5
21	Low	Toxaphene	4
22	Medium	Toxaphene	4
23	High	Toxaphene	4
24	Low	Pesticide A	–
25	Low	Pesticide B	–
26	Blank	–	–

This sequence will establish potential quantitation peaks and their retention times. It will also establish a linear response calibration for each analyte peak.

3.2 Each Aroclor and Toxaphene calibration standard must be run at three concentrations. The analytes and the concentrations required for the low point are given in Table E.2. The midpoint concentrations are 10 times the low point concentration, and the high point concentration is selected by the laboratory in the concentration range between 30 to 100 times the low point concentration. It is recommended that a concentration 100 times the CRQL be used for the high point calibration, provided that it lies within the linear range of the detector. The high point calibration point defines the upper end of the concentration range for which the calibration is valid.

TABLE E.2. LOW POINT AROCLOR CALIBRATION SOLUTIONS

Compound	CAS Number	Low Concentration
1. Aroclor 1016	12674-11-2	50 ng/mL
Aroclor 1260	11096-82-5	50 ng/mL
2. Aroclor 1221	11104-28-2	50 ng/mL
3. Aroclor 1232	11141-16-5	50 ng/mL
4. Aroclor 1242	53469-21-9	50 ng/mL
5. Aroclor 1248	12672-29-6	50 ng/mL
6. Aroclor 1254	11097-69-1	50 ng/mL
7. Toxaphene	8001-35-2	500 ng/mL
8. Tetrachloro-meta-xylene	877-09-8	20 ng/mL
9. Decaclorobiphenyl	2051-24-3	20 ng/mL

3.3 An on-scale chromatogram must be presented for each calibration run.

3.4 During the initial calibration sequence absolute retention times (RT) are determined for four or five major peaks of each Aroclor and Toxaphene.

3.5 If the mean deviation (M.D.) of the absolute retention time for the three calibration measurements for any peak exceeds 0.5 percent of the mean absolute retention time (RT) for that peak, the analytical system is out of control and corrective action must be taken before collecting any data.

$$M.D. = \frac{1}{3} \sum_{i=1}^{3} |RT_i - \overline{RT}| \qquad (3.1)$$

RT_i = Absolute retention time of quantitation peak.

$\overline{RT}$ = Mean absolute retention time of the quantitation peak.

3.6 A retention time window of ± 1.0 percent of the RT established during the initial calibration is calculated for each quantitation peak.

3.7 Three-point instrument calibration is required for each potential quantitation peak. The laboratory has three choices on how to establish three-point calibration (sections 3.7.1 - 3.7.3). Only one

of the three calibration methods may be used to quantitate samples in any single run sequence. Therefore, once a system is calibrated, laboratories may not change calibration techniques for samples until a new initial calibration is run.

3.7.1 The laboratory may use a mean calibration factor (CF) determined from the three concentrations but only if the % RSD for the three points is <15 percent.

$$\overline{CF} = \frac{1}{3} \sum_{i=1}^{n} \frac{\text{Response of Peak Area (or Height) of the Standard}}{\text{Mass Injected (ng)}} \qquad (3.2)$$

$$\%RSD = \frac{SD}{\overline{CF}} \times 100 \qquad (3.3)$$

$$\text{Where } SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \quad \text{and } n=3 \qquad (3.4)$$

3.7.2 The laboratory may use a calibration line drawn through all three calibration points if the value for r (the correlation coefficient from the linear regression calculation) is >0.975 and if the zero concentration intercept is <0.20 times the low point response for each analyte quantitated.

3.7.3 Laboratories with electronic integrators or data systems that automatically calculate calibration curves as line segments between calibration points may use two line segment calibration curves for each quantitation peak of the Aroclor or Toxaphene. This technique may be used only if r (the correlation coefficient from the linear regression calculation) is >0.975 for all three points for each peak and if the zero concentration intercept is <0.20 times the low point response for each Aroclor or Toxaphene peak quantitated.

3.8 Because they are potential method interferences, mixtures of single component pesticides are injected as part of the calibration sequence to establish the RT of individual pesticides. Calibration factors are not calculated for the individual pesticide standards.

3.9 TABLE E.3. PESTICIDE MIXTURES

Individual Standard Mix A	Concentration (ng/mL)	Individual Standard Mix B	Concentration (ng/mL)
alpha-BHC	25.0	beta-BHC	25.0
Heptachlor	25.0	delta-BHC	25.0
gamma-BHC	25.0	Aldrin	25.0
Endosulfan I	25.0	Heptachlor epoxide	25.0
Dieldrin	50.0	alpha-Chlordane	25.0
Endrin	50.0	gamma-Chlordane	25.0
p,p'-DDD	50.0	p,p'-DDE	50.0
p,p'-DDT	50.0	Endosulfan sulfate	50.0
Methoxychlor	50.0	Endrin aldehyde	50.0
Tetrachloro-meta-xylene	20.0	Tetrachloro-meta-xylene	20.0
Decachlorobiphenyl	20.0	Decachlorobiphenyl	20.0
		Endrin Ketone	50.0
		Endosulfan II	50.0

3.10 Sample analysis may not proceed until a satisfactory calibration has been demonstrated.

3.11 The more highly chlorinated Aroclor and Toxaphene components are more stable in the environment. Therefore, the analyst should emphasize the later eluting peaks of a pattern in identifying and quantitating weathered Aroclors and Toxaphene

3.12 If more than one Aroclor is observed in a sample, or if an Aroclor and Toxaphene occur in the same sample, the laboratory must choose separate peaks to quantitate the different analytes. A peak common to both analytes present in the sample must <u>not</u> be used to quantitate both Aroclors.

3.13 The surrogates must be added to all calibration standards. The retention time shifts of the surrogates may not excede 0.5% for the analysis of any calibration standard.

3.14 Documentation for Initial Calibration

3.14.1 Documentation of the RT and calibration factors for potential quantitation peaks on both columns must be provided in tabular form. Form VI will be used to report the RT of each potential quantitation peak at each of the three concentrations. The average RT and the relative mean deviation of the three measurements shall also be reported. Form VI will be used to report the CF for each peak at each concentration injected, the concentrations injected, the average CF and the relative standard deviation of the calibration factors at each of the three concentrations.

3.14.2 Documentation of sequence of calibration standards and their retention time shifts must be provided in tabular form on Form VIII.

3.14.3 Documentation of the RT for pesticides on both columns must be provided in tabular form using Form IX.

3.15 Copies of Forms VI, VIII, and IX, as well as instructions for completion of those forms, are presented in Exhibit B of this document. All chromatograms, as well as integration reports or data system printouts for calibration analyses must be submitted in hard copy with the data package.

4. CONTINUED GC PERFORMANCE EVALUATION

4.1 Summary

4.1.1 The performance of the GC must be monitored every 12 hours by running an instrument blank (Section 2.1) and an evaluation standard. The evaluation standard is used to verify that the RT's and CF's of the analyte quantitation peaks have not changed since the initial calibration.

4.1.2 Analysts are cautioned that analyzing an instrument blank and an evaluation mixture once every 12 hours is the minimum contract requirement. Highly complex samples or unstable GC equipment may cause peaks from one injection to be carried over to the next. It may be necessary to analyze these more often to avoid discarding data.

4.1.3 The requirement for running the 12 hour instrument blanks and evaluation mixture is waived when no samples or spike control matrix analyses are run during that 12-hour period. After a break in sample data analysis, a laboratory can resume the analysis of samples and control matrix spikes using the current initial calibration only after an acceptable evaluation mixture is analyzed (3.2). If a successful evaluation mixture cannot be analyzed after an interruption, an acceptable initial calibration must be run before sample data can be collected.

NOTE: This section does not affect the requirement that all acceptable sample analyses must be bracketed by acceptable evaluation mixtures.

4.2 Performance Evaluation Standard

4.2.1 A performance evaluation standard must be run at least once in every 12 hours. If that analysis does not meet the criteria below, a second injection of the same standard at the same concentration must be made immediately.

4.2.2 The performance evaluation standards injected at 12 hour (or less) intervals will be rotated so as to include the Toxaphene and common Aroclors at low, medium, and high concentrations according to the following schedule:

TABLE E.4. 12 HOUR EVALUATIONS

Concentration	Compound	Hour
low	Toxaphene	12
low	1248	24
low	1254	36
low	1016/1260	48
medium	Toxaphene	60
medium	1248	72
medium	1254	84
medium	1016/1260	96
high	Toxaphene	108
high	1248	120
high	1254	132
high	1016/1260	144
Repeat		

4.2.3 For each of the four or five potential quantitation peaks, the RT in the standard must be within the retention time window of ±1.0 percent of the mean RT calculated during the initial calibration.

4.2.4 For each of the four or five potential quantitation peaks, the response (area or height) must be within 20.0 percent (±20.0 RPD) of the mean response obtained during the initial calibration.

$$RPD = \frac{\overline{CF} - CF_E}{\overline{CF}} \times 100 \qquad (4.1)$$

$\overline{CF}$ = Average calibration factor from initial calibration (Equation 3.2).

CF_E = Calibration factor from current evaluation mixture.

4.2.5 The retention time shifts of the surrogates in the evaluation standard must not excede 0.5%.

4.2.6 If the performance evaluation mixture does not meet all of the criteria listed above, the GC system is out of control and appropriate corrective action must then be taken before additional data are collected. After corrections have been made, the initial calibration must be run successfully. All samples analyzed since the previous acceptable evaluation must be reinjected at no additional cost to the Agency. The laboratory manager, or his designate, must address problems and solutions in the narrative.

4.3 Documentation

4.3.1 Documentation of an acceptable performance evaluation mixture analysis on each column shall be submitted on Form VII for each 12 hours of analysis. In addition, each analysis of a performance evaluation mixture must be listed on the analytical sequence given on Form VIII regardless of whether or not it was acceptable.

4.3.2 A hard copy of all chromatograms of performance evaluation analyses, as well as integration reports or data system printouts, must be submitted with the data package.

4.4 Instructions for completing the data reporting forms are contained in Exhibit B.

5. BLANK ANALYSIS

5.1 Two types of blanks are required as part of this protocol. Method blanks, which provide a measure of total laboratory contamination, and instrument blanks, which provide a measure of instrument contamination and sample carry over between injections.

5.2 Method Blank

5.2.1 A method blank of corn oil (500 mg) must be extracted and cleaned up using the protocols given in Exhibit D.

5.2.2 A method blank analysis must be performed for each twenty single phase units in each sample delivery group. It is suggested that blanks be run more often whenever particularly dirty samples are analyzed.

5.2.3 If only a partial set of samples require sulfur cleanup, then two method blanks are required, [one that is shaken with mercury (or copper) and one that is not].

5.2.4 The method blanks must be injected directly after a sample in the run sequence. They may not be run immediately after an instrument blank.

5.2.5 The method blank may not contain more than the contract required quantitation limit of any Aroclor or Toxaphene quantitation peak. If a method blank exceeds the criterion, the analytical system is out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken. All samples processed with a method blank that is out of control (i.e., contaminated) must be re-extracted, cleaned up again, and reanalyzed at no additional cost to the Agency. The laboratory manager, or his designate, must address problems and solutions in the narrative (Exhibit B).

5.2.6 The retention time shifts of the surrogates in the method blank must not excede 0.5%.

5.3 Instrument Blank

5.3.1 An instrument blank is a hexane solution containing 20 ng/mL of both of the surrogates. An acceptable instrument blank must be run at least once every 12 hours. An instrument blank must be run immediately prior to the performance evaluation mixture in the run sequence (see Exhibit D). To avoid the necessity of reanalysis, it is recommended that additional blanks be analyzed whenever particularly dirty samples are analyzed.

5.3.2 An acceptable instrument blank analysis must demonstrate that no potential quantitation peak of an Aroclor or Toxaphene is detected at greater than 0.5 times the CRQL. If quantitation peaks identified during the initial calibration are detected at greater than half the CRQL, all data collection must be stopped and corrective action taken. Data for samples with analytes detected at >CRQL which were analyzed between the last acceptable instrument blank and the unacceptable blank must be considered suspect. An acceptable instrument blank must be run before additional data is collected. After an acceptable instrument blank is run, all samples with Aroclors or Toxaphene detected at levels >CRQL analyzed after the last acceptable instrument blank must be reinjected during a valid run sequence and reported at no expense to the Agency.

5.3.3 If an instrument blank exceeds the 0.5 x CRQL criteria, the Contractor must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken. The laboratory manager, or his designate, must address problems and solutions in the narrative (Exhibit B).

5.3.4 The retention time shifts of the surrogates in the instrument blank must not excede 0.5%.

5.4 Documentation

5.4.1 Results of method blank analysis must be reported using Form I (Aroclor/Toxaphene Analysis Data Sheet). In addition, the samples associated with each method blank must be summarized on Form IV. Specific instructions for the completion of these forms can be found in Exhibit B.

5.4.2 Results of instrument blank analyses must be reported using Form I. In addition, the samples associated with each instrument blank must be summarized on Form IV. Specific instructions for the completion of these forms can be found in Exhibit B.

5.4.3 In addition, each analysis of a method blank or instrument blank must be reported on Form VIII for each analytical sequence.

5.4.4 Chromatograms, as well as integration reports or data system printouts for calibration analyses must be submitted in hard copy with the data package.

5.4.5 The Contractor will report ALL sample concentration data UNCORRECTED for blanks. It shall be the responsibility of the EPA evaluator, or data auditor, to correct analyte concentrations for concentrations detected in the blank(s). It is the Contractor's responsibility to ensure that the proper number of blanks are analyzed and that the data are properly reported.

6. CONTROL MATRIX SPIKE ANALYSIS

6.1 Control Matrix Spike Requirements

6.1.1 In order to evaluate the effect of the sample matrix upon the analytical methodology, the EPA has specified that a solution containing Aroclor 1254 at 25 ug/mL) be used to spike a control matrix of corn oil (500 mg).

6.1.2 A control matrix spike must be analyzed once for each Case, or for every 20 single phase units.

6.2 Calculations

6.2.1 The recovery of each Aroclor 1254 in the control matrix spike is calculated using Equation 6.1.

$$\text{Control Matrix Spike Percent Recovery} = \frac{SSR}{SA} \times 100 \qquad (6.1)$$

Where:

SSR = Spike Sample Results
SA = Spike Added from Spiking Mix

6.3 Documentation

6.3.1 The concentration of nonspike target compounds in the control matrix spike must be reported on Form I. The quantitation of each Aroclor 1254 peak on both columns must be reported on Form X. The recovery of Aroclor 1254 must be reported on Form III. Specific instructions on the completion of these forms may be found in Exhibit B.

6.3.2 Chromatograms as well as integration reports or data system printouts of control matrix spike analyses, will be submitted in hard copy with the data package.

7. SAMPLE CLEANUP

7.1. Summary

7.1.1 The cleanup of sample requires Diol cartridges for all samples.

7.1.2 Every lot number of Diol cartridges must be tested by the following procedure before they are used for sample cleanup. Add 0.5 mL of Aroclor, midpoint concentration (described in Exhibit D, Section 5.5.1) to 4 mL of hexane, then reduce the final volume to 0.5 mL using nitrogen (Exhibit D, Section 5.4). Place the mixture onto the top of a prewashed Diol cartridge, and elute it with 9 mL of hexane/acetone [(90:10)(V/V)]. Adjust the final volume to 10.0 mL and analyze by GC/ECD.

7.1.3 The recovery of Aroclor 1254 must be determined for evaluation and reporting purposes. The lot of Diol cartridges is acceptable if the Aroclor is recovered at 80 to 110 percent.

7.4 Documentation

7.4.1 Documentation of the Diol cartridge performance will be provided on Form XI by reporting the recovery of Aroclor 1254.

8. SAMPLE ANALYSIS SUMMARY

8.1 General

8.1.1 This section DOES NOT replace or supersede the specific analytical methods or QA/QC activities described in previous sections. The intent of this subsection is to provide the contractor laboratory with a BRIEF summary of QC activities involved with sample analysis to help the contractor laboratory meet specific reporting and deliverables required by this contract.

8.1.2 Samples may only be analyzed upon successful completion of the initial QC activities. The laboratory must run an evaluation mixture and an instrument blank (described in Section 2.1) at least every 12 hours. The laboratory may continue to quantitate data using the initial calibration until an instrument blank shows greater than half the contract minimum level of any Aroclor or Toxaphene, an unacceptable evaluation mixture is analyzed, or the calibration factor of a multicomponent analyte changes by more than 20 percent. Any major system maintenance, such as installation of a new column or, changing or cleaning the detector will also necessitate reanalysis of the initial calibration sequence.

8.1.3 All acceptable data must be bracketed with an acceptable instrument blank and an acceptable evaluation mixture. Any samples analyzed which do not meet this criteria must be reinjected and reported at no expense to the Agency.

8.1.4 At least one acceptable evaluation mixture must be run every 12 hours. If an unacceptable evaluation run is made, the laboratory must run a second evaluation mixture immediately. If the results of two successive evaluation runs do not meet the specifications of Section 4.2, then all analyses since the last acceptable evaluation mixture are not valid. They must be reinjected after the GC is inspected for malfunction and corrections made as appropriate. Loss of sensitivity or resolution could result from a damaged septum or column. High detector standing current (background) could result from contaminated carrier gas or from a dirty detector.

8.1.5 An acceptable instrument blank analysis must demonstrate that no potential quantitation peak of an Aroclor or Toxaphene is detected at greater than 0.5 times the CRQL. If quantitation peaks identified during the initial calibration are detected at greater than half the CRQL, all data collection must be stopped and corrective action taken. Data for samples with analytes detected at >CRQL which were analyzed between the last acceptable instrument blank and the unacceptable blank must be considered suspect. An acceptable instrument blank must be run before additional data is collected. After an acceptable instrument blank is run, all samples with Aroclors or Toxaphene detected at levels >CRQL analyzed after the last acceptable instrument blank must be reinjected during a valid run sequence and reported at no expense to the Agency.

8.1.6 At least one method blank must be analyzed for each 20 single phase units analyzed. If any Aroclor or Toxaphene is detected, in a method blank, at more than the CRQL, all sample data collection must stop. The laboratory must then immediately run an instrument blank to demonstrate that the instrument is not contaminated. When an acceptable instrument blank has been analyzed, the same method blank must be reinjected. If the method blank is unacceptable after the second injection, all single phase units that were analyzed since the last acceptable method blank must be re-extracted, cleaned up again and analyzed at no expense to the Agency.

8.2 Sample Dilution

8.2.1 The protocol is intended to achieve the quantitation limits shown in Exhibit C whenever possible. Whenever sample chromatograms have interfering peaks, a high baseline, or off-scale peaks, the samples must be reanalyzed following further dilution. Samples which cannot be made to meet the specifications given in this section after dilution and cleanup (sulfuric acid, permanganate, Diol cartridge and mercury removal) are discussed in detail in the Case Narrative and do not require further analysis. No limit is placed on the number of re-extractions and cleanups of samples that may be required because of contaminated method blanks.

8.2.2 The sample must be analyzed at the most concentrated level consistent with achieving satisfactory chromatography (defined below). If dilution is employed solely to bring a peak within the calibration range or produce an on-scale chromatogram for the multicomponent analyte, the results for both a more and a less concentrated dilution must be reported. The resulting changes in quantitation limits and surrogate recoveries must also be reported for the dilute samples.

8.2.3 If the laboratory has reason to believe that diluting the final volume will be necessary, an undiluted run may not be required. If an acceptable chromatogram (as defined in Section 5.) is achieved with the diluted final volume, an additional analysis at 10 times the concentration of the diluted material must be injected and reported with the sample data.

8.2.4 The response of peaks in the sample must be >25 percent of full scale to allow visual pattern recognition of multicomponent analytes.

8.2.5 An on-scale chromatogram(s) of all identified peaks must be presented with the sample data.

8.2.6 Chromatographic data may be replotted electronically in order to get an on-scale chromatogram, except when the off-scale sample peaks are larger than the high point calibration peaks. In that case, the samples must be diluted and reinjected.

8.2.7 The peak response of sample peaks on the replotted chromatogram must be >25 percent of full scale to allow visual pattern recognition of multicomponent analytes.

8.3 Data Acceptance Criteria

8.3.1 Reportable data for a sample must include a chromatogram with a baseline which returns to below 50 percent of full scale before the elution time of Aroclor 1221 and to below 25 percent of full scale after Aroclor 1221 and before the elution time of Decachlorobiphenyl.

8.3.2 If dilution has been applied and no peaks are detected above 25 percent of full scale, analysis of a more concentrated extract is required.

8.3.3 Reportable sample data must include chromatogram(s) with all Aroclor and Toxaphene quantitation peaks in the linear range of the quantitation curve determined by the initial calibration.

8.3.4 Reportable sample data must include chromatogram(s) with all Aroclor and Toxaphene peaks on-scale.

NOTE: If more than one chromatogram is required to satisfy the criteria for a sample, the results of all chromatograms must be reported. These requirements apply to both columns.

8.3.5 The retention time shifts for the surrogates in any sample must not excede 0.5%. If the surrogates have been diluted out, so that no retention time shift can be calculated, report the percent difference as "dil" (see Forms Instructions), and explain in the Case Narrative.

8.4 Quantitation of Analyses

8.4.1 Analytes can be quantitated using either a modern 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.

8.4.2 The chromatograms of all samples must be reviewed by a qualified pesticide/PCB analyst before they are reported.

8.4.3 Using an electronic integrator, one of three calibration techniques may be employed (Section 3.7). It is the responsibility of the analyst to set the integration parameters such that off-scale chromatograms are within the dynamic range of the instrument. The analyst should also check for data flags generated by the instrument that indicate improper quantitation of peaks prior to reporting data to the EPA.

8.4.4 In order to be quantitated, the detector response (peak area or peak height) of all analytes must lie between the responses of the low and high concentrations in the initial calibration. If the analytes are detected below the CRQL, they are reported with the appropriate flags (See 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.

8.4.5 The concentration of the Aroclor and Toxaphene quantitation peaks are calculated using the following equations:

$$\text{Concentration ug/L} = \frac{(A_x)(V_t)}{(\overline{CF})(V_i)(W_x)} \qquad (8.1)$$

Where:

A_x = Response for the peak to be measured.
$\overline{CF}$ = Calibration factor from the initial calibration.
V_t = Volume of total waste dilution (uL) (take into account any dilution).
V_i = Volume of waste dilution injected (uL).
W_x = Weight of waste diluted (gm).

8.4.6 The the recoveries of the surrogates are calculated using the following equations:

$$\text{Percent Recovery} = \frac{SA - SR}{SA} \times 100\% \qquad (8.2)$$

SA = Concentration of surrogate added

SR = Concentration of surrogate recovered

8.4.7 The laboratory will quantitate each of the selected Aroclor or Toxaphene peaks individually, and determine an average concentration from all of the selected peaks. Quantitation is performed on both columns, and the lower value is reported on Form I. See Exhibit B for instructions on completing Form I and Form X.

8.4.8 Detected Aroclors and Toxaphene must be reported as: (1) a concentration between the CRQL and 10^6 x CRQL, or (2) as an estimated value below the CRQL. (See Exhibit B.)

8.4.9 The choice of the peaks used for Aroclor or Toxaphene quantitation and recognition of those peaks may be complicated by the environmental alteration of the Aroclor or Toxaphene, and by the presence of coeluting analytes or matrix interferences.

8.4.10 The more highly chlorinated components of the Aroclors and Toxaphene are more stable in the environment. Therefore, the analyst should emphasize the later eluting peaks of a pattern when identifying and quantitating weathered Aroclors or Toxaphene.

8.4.11 If more than one Aroclor is observed in a sample or if an Aroclor and Toxaphene are both observed in a sample the laboratory must choose separate quantitation peaks for the different Aroclors and Toxaphene. A peak common to both analytes in the sample must <u>not</u> be used to quantitate both compounds.

8.5 Documentation

8.5.1 Refer to Exhibit B of this document for specific details on contract deliverable and reporting formats. Exhibit B contains specific instructions for completing all required forms, as well as detailed itemization of deliverables and reporting requirements.

9. Surrogates

9.1 Requirements

9.1.1 Tetrachloro-meta-xylene and Decachlorobiphenyl must be added to each sample, control matrix spike, and blank analyzed as part of this protocol.

9.1.2 The recoveries of the surrogates are calculated using equation 8.2.

9.2 Documentation

9.2.1 The recoveries of the surrogates are reported for each sample, diluted sample, and control matrix spike on Form II.

9.2.2 Complete instructions for the completion of Form II are given in Exhibit B.

10. GC Maintenance

10.1 Laboratory Responsibility

10.1.1 It is critical that the gas chromatographs used for these analyses be maintained properly and that all manufacturers' recommendation be followed. Although not addressed in this document, it is expected that each analytical laboratory will maintain performance checks on their instruments, including monitoring detector standing current and monitoring the number of theoretical plates per column.

10.2 Suggested Maintenance

10.2.1 GC Columns - When degradation in column performance is observed, it is usually the result of a build up of nonvolatiles on the head of the column. These can best be removed by cutting off 1-2 feet of the injector end of the column. The analyst must cool the column(s) to room temperature before exposing them to air or the column(s) will oxidize and lose some ability to resolve analytes.

10.2.2 Septa - must be replaced regularly to prevent degradation of chromatographic performance and destruction of the columns. They must be replaced at least once per day whenever data are collected.

10.2.3 Carrier Gas - should be high purity, oxygen, and water free. The use of in-line water and oxygen traps is recommended even if ultra-high purity gas is used.

10.3.4 Electron Capture Detector - The condition of the detector can be monitored by its standing current. A high standing current usually indicates contaminated carrier gas, a high column bleed rate, or a dirty detector. Changing the gas filters, oxygen traps, or the column may correct the problem. If it does not, raise the detector temperature overnight to bake out the detector.

10.2.5 NOTE: Do not collect data while cleaning the detector.

10.2.6 More serious contamination will require steam cleaning the detector or detector repair by the manufacturer. Steam cleaning or detector repair will necessitate recalibration of the instrument.

10.3 Documentation

10.3.1 All major instrument maintenance or repair will be recorded, dated, and initialed in an instrument log that will be made available to auditors during on-site inspections.

11. Solvents and Reagents

11.1 Aliquots of 100 mL of all lots of solvents used for the analysis of samples by this method will be concentrated, exchanged to a final volume of 1.0 mL of hexane and analyzed by GC/ECD. The solvent lot is acceptable only if no analyte is detected at >0.5 CRQL.

11.2 Commercial standards must be diluted and analyzed by GC/ECD to demonstrate that they give acceptable retention times and that they give a response between 95 to 105 percent of EPA repository standards.

11.3 Documentation

11.3.1 Chromatograms for all lots of solvents, and commercial standards must be on file at the laboratory. They are not a contract deliverable.

SECTION IV

ANALYTICAL STANDARDS

A. The Environmental Protection Agency's Quality Assurance Materials Bank will supply primary standards (calibration standards, surrogate standards, control matrix spiking standards, and internal standards), contingent upon their availability, for traceability and quantitative verification of Contractor standards. It is emphasized that these primary standards are for traceability only. There are insufficient quantities to have these available to serve as working standards. The Contractor laboratory is responsible for preparing its own working standards from commercial sources.

B. Caution should be exercised in the mixing of these standards, particularly the multicomponent standards. Chemical reactions such as acid/base reactions, Schiff base formations (reactions of aldehydes and ketones with primary amines), hydrolysis, isotopic exchange, and others may occur.

C. EPA contract laboratories can call or write directly to the QAMB (address and phone number on the following request form) to obtain reference standards. Standards will be provided based on the reasonableness of the request and their availability. Any request from a commercial laboratory that is not currently under contract to EPA will be denied.

D. Upon award of a contract, a list of available standards will be provided by EMSL-LV upon request.

QUALITY ASSURANCE MATERIALS BANK
REQUEST FOR REFERENCE STANDARDS

MAIL TO: U.S. Environmental Protection Agency
Quality Assurance Materials Bank (MD-8)
Research Triangle Park, NC 27711 USA

Date _____

Telephone:
Requests ONLY: (919) 541-4019
(FTS) 629-4019
Information: (702) 545-2690
(FTS) 545-2690
Technical Assistance: (919) 541-3951
(FTS) 629-3951

Date Request Received _____
Date of Shipment _____
Laboratory Code Number _____
Request Number _____
Verified _____

THIS BLOCK FOR AGENCY USE ONLY

The following reference standards are required for our program:

Number Required	Standard Code Number	Compound(s)	Solvent	Purity	Concentration (µg/ml)

Please complete this form in full, PRINTING OR TYPING name and address. Use block one if possible. Use back of sheet to complete list if necessary. Do cover letter if needed.

Name and Address of Laboratory: _____

_____

_____

_____

_____
Requester's Name

IMPORTANT:

I certify that the calibrated sample(s) requested are to be used only in this laboratory by qualified personnel for the purpose of developing or checking analytical procedures and/or for the calibration of instrumentation.

_____
Date

_____
Signature

Request for Reference Standards

SECTION V

LABORATORY EVALUATION PROCEDURES

This section outlines the procedures which will be used by the Project Officer or his/her authorized representative during the contract period of performance to conduct laboratory audits to determine the Contractor's continuing ability to meet the terms and conditions of this contract. The evaluation process incorporates two major steps: 1) evaluation of laboratory performance, and 2) on-site inspection of the laboratory to verify continuity of personnel, instrumentation and quality control requirements of the contract. The following is a description of these two steps.

Part 1 - Evaluation of Laboratory Performance

1. Performance Evaluation Sample Analysis

1.1 A Performance Evaluation (PE) sample set may be sent to a participating laboratory on a quarterly basis to verify the laboratory's continuing ability to produce acceptable analytical results. These samples will be provided either as single blind (recognizable as a PE material and of unknown composition) or double blind (not recognizable as a PE material and of unknown composition).

If received as a single blind, the contractor is required to submit PE sample data in a separate SDG package in accordance with Delivery Schedule requirements for sample data. PE samples received as double blind would be treated as routine samples and data would be submitted in the SDG deliverables package per normal procedure.

1.2 When the PE data are received, results will be scored routinely for identification and quantitation. Results of these scorings will be provided to the Contractor. The government may adjust the scores on any given PE sample to compensate for unanticipated difficulties with a particular sample.

1.3 If a laboratory performs unacceptably, the laboratory will be immediately notified by the Project Officer. A laboratory so notified may expect, but the government is not limited to, the following actions: a site visit, a full data audit, and/or laboratory analysis of a second PE sample. Failure by the laboratory to take corrective actions and/or failure of two successive PE sample analyses will require that the laboratory discontinue analysis of samples until such time as the Project Officer has determined that the laboratory may resume analyses.

2. Organic Data Audit

2.1 Organic data audits are conducted on CLP Contractor's Reporting and Deliverables packages by EMSL/LV. The organic data audit provides the Agency with an in-depth inspection and evaluation of the Case data packages with regard to achieving QA/QC acceptability.

Part 2 - On-Site Laboratory Evaluation

2. The on-site laboratory evaluation helps to ensure that all the necessary quality control is being applied by the Contractor in order to deliver a quality product.

2.1 Quality assurance evaluations allow the evaluators to determine that:

2.1.1 The organization and personnel are qualified to perform assigned tasks,

2.1.2 Adequate facilities and equipment are available,

2.1.3 Complete documentation, including chain-of-custody of samples is being implemented,

2.1.4 Proper analytical methodology is being used,

2.1.5 Adequate analytical Quality Control, including reference samples, control charts, and documented corrective action measures, is being provided, and

2.1.6 Acceptable data handling and documentation techniques are being used.

2.2 The on-site visit also serves as a mechanism for discussing weaknesses identified through the Performance Evaluation sample analysis or through Contract Compliance Screening or other review of data deliverables. Lastly, the on-site visit allows the evaluation team to determine if the Contractor has implemented the recommended and/or required corrective actions, with respect to quality assurance, made during the previous on-site visit.

EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL,

AND STANDARD OPERATING PROCEDURES

Rev. 9/88

1. Sample Chain-of-Custody

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous waste investigations is that samples and data may be used as evidence in EPA enforcement proceedings. To satisfy enforcement uses of the data, the following chain-of-custody procedures have been established.

1.1 Sample Identification

To assure traceability of samples while in possession of the Contractor, a method for sample identification shall be developed and documented in laboratory Standard Operating Procedures (SOPs) (see Section 3). Each sample or sample preparation container shall be labeled with a unique number identifier (or the SMO number). This identifier shall be cross-referenced to the sample tag number and the SMO number. There shall be a written description of the method of assigning this identifier and attaching it to the sample container included in the laboratory SOPs.

1.2.1 A sample is under custody if:

1.2.1.1 It is in your actual possession,

1.2.1.2 It is in your view after being in your physical possession,

1.2.1.3 It was in your possession and then you locked or sealed it up to prevent tampering, or

1.2.1.4 It is in a secure area.

1.2.2 Upon receipt of the samples in custody, the Contractor shall inspect the shipping container and sample bottles and shall document receiving information as specified in Section 3.2. The sample custodian or a designated representative shall sign and date all appropriate receiving documents at the time of receipt (i.e., EPA chain-of-custody forms, traffic reports, airbills, etc.). The Contractor shall contact SMO if documents are absent, information on receiving documents does not agree, custody seals are not intact, or the sample is not in good condition. The Contractor shall document resolution of any discrepancies, and this documentation shall become a part of the permanent case file.

1.2.3 Once samples have been accepted by the laboratory, checked, and logged in, they must be maintained in accordance with custody and security requirements specified in 3.3.

2. Document Control Procedures

The goal of the laboratory document control program is to assure that all documents for a specified case 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 Data Sheets and Logbooks

Preprinted data sheets shall contain the name of the laboratory and be dated and signed by the analyst or individual performing the work. All documents produced by the laboratory 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 case file. For that reason, all observations and results recorded by the laboratory but not on preprinted data sheets are entered into permanent laboratory logbooks. The person responsible for the work shall sign and date each entry and/or page in the logbook. When all data from a case is compiled, copies of all EPA case-related logbook entries shall be included in the documentation package. Analysts' logbook entries must be in chronological order and shall include only one case per page. Instrument run logs shall be maintained so as to enable a reconstruction of the run sequences 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 SMO sample identification numbers in the logs for sample ID rather than government agency or commercial client names.

Using laboratory or SMO sample IDs only in the run sequences will assist the laboratory in preserving the confidentiality of commercial clients.

2.2 Error Correction Procedure

All documentation in logbooks and other documents shall be in ink. If an error is made, corrections shall be made by crossing a line through the error and entering the correct information. Changes shall be dated and initialed. No information shall be obliterated or rendered unreadable.

2.3 Consistency of Documentation

Before releasing analytical results, the laboratory shall assemble and cross-check the information on sample tags, custody records, lab bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or case is consistent throughout the case file.

2.4 Document Numbering and Inventory Procedure

In order to provide document accountability of the completed analysis records, each item in a case shall be inventoried and assigned a serialized number and identifier associating it to the case and Region.

Case # - Region - Serialized number (For example: 75-2-0240)

The number of pages of each item must be accounted for if each page is not individually numbered. All documents relevant to each case, including logbook pages, bench sheets, mass spectra, chromatograms, custody records, library search results, etc., shall be inventoried. The laboratory shall be responsible for ensuring that all documents generated are placed in the file for inventory and are delivered to EPA. Figure 1 is an example of a document inventory.

2.5 Shipping Data Packages and Case Files

The Contractor shall have written procedures to document shipment of deliverables packages to the recipients. Case File Purge shipments require custody seals on the container(s) placed such that it cannot be opened without damaging or breaking the seal. The Contractor shall also document what was sent, to whom, the date, and the method (carrier) used.

3. Standard Operating Procedures

The Contractor must have written standard operating procedures (SOPs) for (1) receipt of samples, (2) maintenance of custody, (3) sample storage, (4) tracking the analysis of samples, and (5) assembly of completed data.

An SOP is defined as a written narrative step-wise description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits.

3.1 The Contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.2 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:

- o Presence or absence of EPA chain-of-custody forms
- o Presence or absence of airbills
- o Presence or absence of EPA Traffic Reports or SAS packing lists
- o Presence or absence of custody seals on shipping and/or sample containers and their condition
- o Presence or absence of sample tags
- o Sample tag ID numbers if not recorded on the chain-of-custody record(s) or packing list(s)
- o Condition of the shipping container
- o Condition of the sample bottles
- o Verification of agreement or nonagreement of information on receiving documents
- o Resolution of problems or discrepancies with the Sample Management Office

3.3 The Contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.4 The Contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

3.4.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.4.2 A description of the documentation used to record instrument calibration and other QA/QC activities.

3.4.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.5 The Contractor shall have written SOPs for organization and assembly of all documents relating to each EPA case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other

written documents having reference to the Case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure.

3.6 The Contractor shall have written SOPs for laboratory safety.

3.7 The Contractor shall have written SOPs for cleaning of glassware used in preparing and analyzing samples under this contract.

3.8 The Contractor shall have SOPs for traceability of standards used in sample analysis QA/QC.

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 logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Contracting Officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA Contracting Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

Figure 1

Example

DOCUMENT INVENTORY

Document Control #*	Document Type	# Pages
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	50
232-2-0005	SMO Organics Traffic Reports	10
232-2-0006	GC/MS spectra for sample B0310	20
232-2-0007	GC/MS spectra for sample B0311	20
232-2-0008	GC/MS spectra for sample B0319	20
232-2-0009	Analyst's logbook pages	6
232-2-0010	GC/MS library search worksheets	15
232-2-0011	GC instrument log pages	5
232-2-0012	GC/MS QC data sheets	4
etc.	etc.	etc.

*This number is to be recorded on each set of documents.

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.

AROCLOR - trade name (Monsanto) for a series of commercial polychlorinated biphenyls and polychlorinated terphenyl mixtures marketed in the United States.

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 tuning performance for volatile analyses. BFB tuning criteria must be met before GC/MS analysis can begin. (BFB is also used as a surrogate.)

CALIBRATION CHECK COMPOUNDS (CCC) - target compounds used to evaluate the calibration stability (precision) of the GC/MS system. Maximum percent deviations of the CCCs are defined in the protocol.

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.

CONFIRMATION ANALYSIS - see Primary Analysis.

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the calibration of the purge and trap-GC/MS system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction.

CONTROL MATRIX SPIKE - corn oil fortified with known quantities of specified compounds analyzed to measure the recovery (accuracy) of the entire analytical method.

DAY - unless otherwise specified, day shall mean calendar day.

DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) - compound chosen to establish mass spectral tuning performance for extractable analysis. DFTPP tuning criteria must be met before GC/MS analysis can begin.

EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include Base/Neutrals, Acids and Pesticide/PCB compounds.

HOMOLOG - one of the 10 levels of chlorination of PCBs ($C_{12}H_9Cl$ through $C_{12}Cl_{10}$) or other group of compounds, varying by systematic addition of substituent.

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 to the target compounds.

INTERNAL STANDARDS - compounds added to every standard, blank, control matrix spike and sample extract 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.

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 contamination.

NARRATIVE (Case Narrative) - first portion of the data package which includes laboratory name, contract number, 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 Case Narrative specifications are included in Exhibit B.

PERCENT MOISTURE - an approximation of the amount of water in a solid phase 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 105°C, including water. Percent moisture is determined from decanted samples and from samples that are not decanted.

PHASE - describes the physical state(s) of the sample. Three "phase designatores" are used: Solid; Water miscible liquid; Water immiscible liquid. A sample may contain multiple phases.

PCB CONGENER - one of 209 PCBs at any level of chlorination.

PCB ISOMER - any PCB or other compound which has the same molecular formula, but different positional substitutions. (2,2' dichlorobiphenyl and 2,3 dichlorobiphenyl are isomeric.)

POLYCHLORINATED BIPHENYLS (PCBS) - a class of 209 discrete chemical compounds in which one to ten chlorine atoms are attached to biphenyl; i.e., monochlorobiphenyl through decachlorobiphenyl with the formula $C_{12}H_{10-n}CL_n$ where n = 1-10.

PRIMARY ANALYSIS - one of two types of pesticide/PCB analysis by GC/EC techniques, the other being the Confirmation Analysis. If the two analyses are run at separate times, the Primary Analysis is the first analysis

chronologically, and is used to establish the tentative identification of any pesticides/PCBs detected. The identification is then confirmed in the confirmation analysis. If the two analyses are simultaneous, either may be considered the Primary Analysis.

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 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.

RECOVERY - a determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

$$\%Rec = \frac{\text{measured value}}{\text{known value}} \times 100\%$$

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where A = area of the characteristic ion measured

C = concentration

is = internal standard

x = analyte of interest

RESOLUTION - also termed separation, the separation between peaks on a chromatograms, calculated by dividing the height 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. A sample may contain more than one phase.

SAMPLE DELIVERY GROUP (SDG) - a unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer single phase units within a Case, received over a period of up to 14 calendar days. Data from all single phase units in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 single phase units within a Case; or
- Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

SAMPLE NUMBER (EPA Sample Number) - a unique identification number designated by EPA for each sample. The EPA sample number appears on the sample Traffic Report which documents information on that sample.

SINGLE PHASE UNIT - a subsample consisting of a single phase. Multiple phase samples are phase separated into single phase units. According to the protocol, single phase units are analyzed and reported as discrete samples.

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 and thereby calibrate the mass spectrometer.

SURROGATES (Surrogate Standard) - compounds added to every blank, sample, control matrix spike, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated or isotopically labeled compounds not expected to be detected in environmental media.

SYSTEM PERFORMANCE CHECK COMPOUNDS (SPCC) - target compounds designated to monitor purge and trap/chromatographic performance, sensitivity and compound instability or degradation on active sites. Minimum response factor criteria for the SPCCs are defined in the protocol.

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 or surrogate standards. 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 tuning and standards calibration (initial or continuing calibration) begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of compliant tune. The time period ends after 12 hours has elapsed according to the system clock.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - the date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report.

VOLATILE COMPOUNDS - 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.