



# **Environmental Technology Verification Program**

## **APPENDICES to Verification Test Plan**

### **Evaluation of Field Polychlorinated Biphenyl (PCB) Detection Technologies**

**ornl**

Oak Ridge National Laboratory

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# APPENDIX A

## Soil Sample Collection Procedures April 1997

# **R&D Soil Sample Collection Plan**

**Polychlorinated Biphenyl (PCB) Field Analytical Technology Demonstration**

**A Plan for the Pre-Demonstration Sampling Activities**

Prepared By:

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Lockheed Martin Energy Research Corporation  
Oak Ridge, TN 37831-6120



## Executive Summary

The purpose of this document is to describe the sample collection activities to occur at the Lockheed Martin Energy Systems' K-25 site in Oak Ridge, TN. The objective is to collect research and development (R&D) soil samples for evaluation during a polychlorinated biphenyl (PCB) field analytical technology demonstration to occur at Oak Ridge National Laboratory (ORNL). ORNL is responsible for the coordination and technical oversight for the demonstration.

A total of thirty-eight R&D soil samples, originally from the Oak Ridge Reservation and Portsmouth sites, will be collected and analyzed in duplicate. A summary of the activities is as follows:

1. A waste container will be located and opened by K-25 Operations group.
2. Health Physics (HP) technicians will survey the container to ensure appropriate personal protective equipment is being utilized.
3. K-25 Sampling group will collect a R&D soil sample from the top of the waste container. The R&D soil sample will be sieved to remove large debris. The debris will be dumped back into the waste container. The R&D soil sample will then be homogenized by gentle mixing. The R&D soil sample will be transferred to a 5-gallon container that is lined with a plastic bag. The sample volume collected will be approximately half of a 5-gallon container, which is calculated to be approximately 12 kg of soil. The 5-gallon container will be labeled using the procedure described below.
4. The K-25 Sampling Group will then collect two analytical samples from the R&D soil sample. The sample volume collected will be enough to fill a 40 mL VOA vial.
5. The two analytical samples will be checked for radiological activity and labeled as described below.
6. Steps 1-5 will be performed for all of the drums listed in Table 2.
7. All analytical samples will be stored in a secure location until the Grand Junction group is prepared to analyze them. The waste containers and R&D soil samples will be left in the staging area until final disposition is determined by ORNL.
8. When appropriate, the analytical samples will be transferred to the ORNL Grand Junction group for preliminary characterization by gas chromatography.
9. Based on the analytical characterization, ORNL will determine if the R&D soil samples should be used in the demonstration. If so, the R&D soil sample will be retained for transfer to ORNL by Analytical Services Organization's Sample Management office. If the R&D soil sample is deemed inappropriate by ORNL for evaluation in the demonstration, the R&D soil sample will be returned to the waste container.
10. The final weights of the waste containers will be determined by K-25 Operations and Sampling groups and recorded before returning the containers to their original storage locations.
11. Careful notes will be recorded to document all activities.

## Key Personnel

Table 1 describes the key personnel involved in the sample collection activities.

**TABLE 1 - Key Personnel**

<b>Name/Contact information</b>	<b>Organization</b>	<b>Role</b>
W. T. Wright phone: (423) 574-8214 pager: (423) 971-9255	EET Corporation	K-25 Site Coordinator for this project
Mike Shelton phone: (423) 576-1895 pager: (423) 873-7684	Lockheed Martin Energy Systems, Inc.	K-25 Operations Group Lead
Bill Ghormley phone: (423) 574-8160 pager: (423) 873-6560	Lockheed Martin Energy Systems, Inc.	K-25 Sampling Group Lead
Frank Gardner phone: (970) 248-6238 email: fgg@ornl.gov	Oak Ridge National Laboratory, Grand Junction, CO	Grand Junction Analytical Group Leader
Charles K. Bayne phone: (423) 574-3134 email: bayneck@ornl.gov	Oak Ridge National Laboratory	Chief Statistician, PCB Field Analytical Technology Demonstration
Amy Dindal phone: (423) 574-4863 email: dindalab@ornl.gov	Oak Ridge National Laboratory	Technical Lead, PCB Field Analytical Technology Demonstration

**Responsibilities**

ORNL will be responsible for:

- Overall coordination and decision-making for the sample collection and analytical activities
- Selecting R&D soil samples to be collected
- Acquiring of materials and/or services not provided by K-25 personnel
- Supplying K-25 Sampling group with labels for R&D and analytical soil samples
- Assisting in disposal of waste generated by Grand Junction group
- Assisting in the transfer of R&D samples to ORNL
- Assisting K-25 Site Coordinator, as necessary

K-25 Site Coordinator for this project will be responsible for:

- Completing appropriate paperwork
- Assisting in the selection of samples to be collected
- Instructing the Operations and Sampling groups through proper chains-of-command
- Supplying and assisting Grand Junction group with laboratory space
- Site access for Grand Junction and ORNL personnel
- Assisting in the transfer of R&D samples to ORNL
- Assisting in disposal of waste generated by Grand Junction group
- Assisting ORNL, as necessary

K-25 Operations Group Lead will be responsible for:

- Supplying the appropriate personnel and equipment to accomplish the activity
- Locating the requested waste containers
- Moving the waste container to a location where a R&D soil sample can be acquired

- Opening the container
- Requesting and acquiring health physics (HP) support
- Determining final weight of the waste container
- Completing appropriate paperwork

K-25 Sampling Group Lead will be responsible for:

- Supplying the appropriate personnel and equipment to accomplish the activity
- Acquiring approximately 12 kg (half of a 5-gal container) R&D soil sample from the waste container
- Sieving and homogenizing the R&D soil sample
- Transferring R&D sample to (plastic bag-lined) 5-gallon container (container to be supplied by K-25)
- Labeling of the R&D soil sample (see description below)
- Obtaining and labeling (see description below) two analytical samples from the R&D soil sample (each analytical sample consists of one full 40 mL VOA vial; vial to be supplied by K-25)
- Storing the analytical samples in a secure location until ready for analysis
- Completing proper chain-of-custody documentation, if appropriate
- Delivering analytical samples to Grand Junction group for analysis
- Returning R&D soil sample to the waste container, as directed by ORNL
- Transferring R&D soil samples that are wanted by ORNL to Analytical Services Organization, Sample Management Office (Angie McGee) for shipment to ORNL

Grand Junction Analytical Group will be responsible for:

- Supplying the appropriate personnel and equipment to accomplish the activity
- Analyzing analytical samples by gas chromatography and reporting results to ORNL
- Labeling (see description below) the R&D soil samples to describe analytical results
- Properly segregating waste generated by the analytical process and assisting in disposal

### **R&D Soil Samples to be Collected**

Table 2 describes the R&D soil samples to be collected by K-25.

### **R&D Soil Sample Label**

Upon collection, K-25 Sampling group will label the R&D soil samples with appropriate PCB labels and with a label provided by ORNL. K-25 will fill-in the following information regarding the R&D soil sample and adhere the label to the 5-gallon container:

---

Sampling Date: \_\_\_\_\_  
 Sampling Time: \_\_\_\_\_  
 Sampler Name: \_\_\_\_\_  
 RFD #: \_\_\_\_\_  
 Drum #: \_\_\_\_\_  
 HP Checked? Yes No  
 Rad Results: \_\_\_\_\_  
 R&D Soil Sample ID: xxxxxxyyyyyz

---

where xxxxxx = sampling date, yyyyy = original waste container ID, z = P if it's a Portsmouth soil and O if it's an Oak Ridge Reservation soil

### **Analytical Sample Labels**

Each analytical sample obtained from a R&D soil sample will be labeled by the K-25 Sampling Group using the following:

---

Sampling Date: \_\_\_\_\_  
Sampling Time: \_\_\_\_\_  
Sampler Name: \_\_\_\_\_  
Drum #: \_\_\_\_\_  
Analytical Sample ID: xxxxxyyyyzAn

---

where xxxxyyyyz is the R&D Soil Sample ID and n is the number of aliquot taken (1 would be the first aliquot taken, etc.)

A second label will be adhered to the R&D soil sample container after the analytical results have been obtained. This label will also be provided by ORNL, to be completed by the Grand Junction group.

---

R&D Soil Sample ID: \_\_\_\_\_  
Analysis Date: \_\_\_\_\_  
Analysis Time: \_\_\_\_\_  
Analyst Name: \_\_\_\_\_  
Mean PCB level: \_\_\_\_\_ ppm  
# sample analyzed: \_\_\_\_\_  
# replicates of each sample: \_\_\_\_\_

---

### **R&D Sample Transfer to ORNL**

Upon completion of the sample collection activities, Analytical Services Organization's Sample Management office will transfer the R&D soil samples to ORNL.

**Table 2 - R&D Soil Samples to be Collected**

Oak Ridge Reservation Soil

"PCB Level"	RFD #	Cont. #	Barcode #	Cont. Type	PCB Conc.	PCB Date	Current Loc.	Area	Row	Level
0.5 ppm	40022	1	K25C9301173	B-25 box	< 0.52 ppm	7/12/91	K-1065E	D3L	6	BOT
	40022	2	K25C9301171	B-25 box	< 0.44 ppm	7/12/91	K-1065E	D3L	6	BOT
	40022	3	K25C9301169	B-25 box	< 0.47 ppm	7/12/91	K-1065E	D3L	6	TOP
	40022	4	K25C9301170	B-25 box	< 0.63 ppm	7/12/91	K-1065E	D3L	6	MID
2 ppm	134555	1	K25C9505076	B-25 box	approx. 11 ppm	8/18/95	K-1065E	D3L	5	TOP
	134555	2	K25C9507505	85 gal drum	approx. 11 ppm	8/18/95	K-1065E	D2L	18	BOT
	134555	3	K25C9507506	85 gal drum	approx. 11 ppm	8/18/95	K-1065E	D2L	18	BOT
25 ppm	use anything that's close from the other ORR samples									
50 ppm	41554	44	K25C9319688	55 gal drum	>50 ppm	9/6/85	K-303-3	A2L	13	BOT
	41554	38	K25C9319695	55 gal drum	>50 ppm	9/6/85	K-303-3	A2L	12	BOT
	41554	60	K25C9319700	55 gal drum	>50 ppm	9/6/85	K-303-3	A2L	13	BOT
	41554	43	K25C9319689	55 gal drum	>50 ppm	8/28/85	K-303-3	A2L	13	BOT
	41554	39	K25C9319608	55 gal drum	>50 ppm	9/6/85	K-303-3	A2L	8	MID
500 ppm	41554	97	K25C9319832	55 gal drum	360 ppm	9/6/86	K-303-3	A2L	12	MID
	41554	98	K25C9405260	85 gal drum	360 ppm	8/12/86	K-303-3	A3L	12	MID
	41554	80	K25C9319634	55 gal drum	360 ppm	5/13/88	K-303-3	A2L	8	BOT
	41554	82	K25C9319635	55 gal drum	360 ppm	5/14/86	K-303-3	A1L	2	BOT
	41554	79	K25C9405269	85 gal drum	360 ppm	3/3/86	K-303-3	A2L	3	BOT

Portsmouth Soil Samples

"PCB Level"	RFD #	Cont. #	Barcode #	Cont. Type	PCB Conc.	PCB Date	Current Loc.	Area	Row	Level
0.5 ppm	7515	10171	K25C9403774	85 gal drum	0.6 ppm	8/23/86	K-303-3	A1R	6	MID
	7515	283	K25C9327174	110 gal drum	1.1 ppm	8/23/86	K-33 TSCA	DF	4	BOT
	7515	4096	K25C9325078	110 gal drum	1.8 ppm	8/23/86	K-303-4	D1L	6	MID
	7515	2309	K25C9325498	110 gal drum	1.9 ppm	8/23/86	K-33 TSCA	DF	4	BOT
	7515	3281	K25C9323899	110 gal drum	1.9 ppm	8/23/86	K-303-4	D1R	11	MID
2 ppm	7515	2403	K25C9324787	110 gal drum	2.2 ppm	8/23/86	K-303-4	D1L	4	MID
	7515	2528	K25C9322593	110 gal drum	2.3 ppm	8/23/86	K-303-4	D1R	10	MID
	7515	2813	K25C9323026	110 gal drum	2.5 ppm	8/23/86	K-303-4	D2L	1	BOT
	7515	4007	K25C9324628	110 gal drum	2.7 ppm	8/23/86	K-303-4	D1L	5	MID
	7515	10221	K25C9403839	85 gal drum	2.7 ppm	8/23/86	K-303-3	A1R	6	MID
25 ppm	7515	3039	K25C9324108	110 gal drum	23.0 ppm	8/23/86	K-303-4	D2L	1	BOT
	7515	1029	K25C9326047	110 gal drum	25.0 ppm	8/23/86	K-33 TSCA	DF	4	BOT
	7515	1898	K25C9326023	110 gal drum	25.0 ppm	8/23/86	K-33 TSCA	DF	4	BOT
	7515	940	K25C9322841	110 gal drum	26.0 ppm	8/23/86	K-303-4	D1R	3	BOT
	7515	3882	K25C9324575	110 gal drum	27.0 ppm	8/23/86	K-303-4	D1L	11	MID
50 ppm	7515	2862	K25C9322800	110 gal drum	42.0 ppm	8/23/86	K-303-4	D2L	6	BOT
	7515	538	K25C9326175	110 gal drum	60.0 ppm	8/23/86	K-33 TSCA	DF	6	MID
	7515	1096	K25C9325466	110 gal drum	54.0 ppm	8/23/86	K-33 TSCA	DA	3	BOT
500 ppm	7515	858	K25C9324788	110 gal drum	14.0-190.0 ppm	8/23/86	K-303-4	D1L	12	MID
	7515	657	K25C9323731	110 gal drum	300 ppm	8/23/86	K-303-4	D2R	12	MID
	7515	1915	K25C9325586	110 gal drum	290 ppm	8/23/86	K-33 TSCA	DA	1	BOT

## APPENDIX B

In-Field Support Laboratory <sup>+</sup>

ORNL-Grand Junction, CO  
Analytical Procedures

<sup>+</sup> The following procedures are general for the analysis of organic compounds. The following are method-specific comments regarding the analysis of PCBs:

- 1) Analytical column: HP5 (30 m x 0.53 mm ID x 0.88  $\mu$ m)
- 2) Oven temperature: 170 °C (hold 2 min) ramped at 4 ° C/min to 275 °C (hold 5 min)
- 3) Calibration standard solvent - hexane
- 4) Solvent extraction procedure: 1 mL deionized water, 4 mL methanol, 5 mL hexane

<b>CAK RIDGE NATIONAL LABORATORY</b> Health Sciences Research Division	<b>PROCEDURE</b>  Environmental Technology Section	Number: TE-160 Page: 1 of 6 Revision: 0 Date: 08/04/95
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**FIELD OPERATION OF THE HEWLETT PACKARD 5890 SERIES II  
GAS CHROMATOGRAPH**

**1.0 PURPOSE**

This procedure describes the use of the Hewlett Packard (HP) 5890 Series II gas chromatograph (GC) for analysis of environmental pollutants.

**2.0 APPLICABILITY**

This procedure applies to the HP 5890 Series II gas chromatograph (GC) which is equipped with a packed injector and a split/splitless injector. The GC includes two detectors: an electron capture detector (ECD) and a photoionization detector (PID). The unit is configured for two 30-m, 60-m, or 120-m megabore capillary columns (0.53-mm I.D.).

**3.0 OTHER DOCUMENTS**

**3.1 REFERENCES**

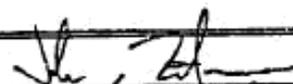
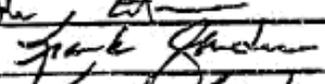
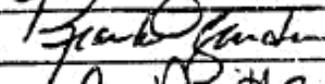
- 3.1.1 Hewlett Packard 5890 Series II Gas Chromatograph operating manual.
- 3.1.2 HP 3396 Series II Integrator operating manual.
- 3.1.3 Procedure TE-169, Using the Chrom Perfect Gas Chromatograph Software.

**3.2 EXHIBITS**

- 3.2.1 Exhibit 1: Example of Chromatogram and Report

**4.0 EQUIPMENT AND MATERIALS**

**4.1 EQUIPMENT**

APPROVALS	
Author 	Date 9/15/95
Supervisor 	Date 10-9-95
Group Leader 	Date 10-9-95
Section Head 	Date 10/16/95

- 4.1.1 HP 5890 Series II gas chromatograph
- 4.1.2 HP 3396 Series II integrator
- 4.1.3 Carrier gas bottles
- 4.1.4 In-line oxygen trap
- 4.1.5 Moisture trap
- 4.1.6 Forceps
- 4.1.7 Set of box and open-end wrenches
- 4.1.8 Personal computer (PC) when using Chrom Perfect software

## **4.2 MATERIALS**

- 4.2.1 Liquid leak detector
- 4.2.2 Non-liquid leak detector
- 4.2.3 Capillary column
- 4.2.4 Septums

## **5.0 RESPONSIBILITY**

- 5.1 It is the responsibility of the operator of the HP 5890 Series II gas chromatograph to follow this procedure.

## **6.0 DEFINITIONS**

- 6.1 **Carrier gas:** gas that serves to transport the standard or sample through the column.
- 6.2 **Chromatogram:** a graphic display, in the form of peaks, of the detector's response to a given standard or sample.
- 6.3 **Electron capture detector (ECD):** a GC detector that measures a decrease in electrical signal rather than an increase in electrical current. The loss of current is proportional to the amount of compound present. The ECD is virtually insensitive to hydrocarbons. It is especially valuable for the analysis of pesticides, PCBs, and chlorinated solvents.
- 6.4 **Photoionization detector (PID):** a GC detector in which the GC column effluent gas stream is subjected to ultraviolet radiation energetic enough to ionize many organic compounds in the gas stream. Since each organic molecule has a characteristic ionization potential (the energy required to remove an electron from a molecule), the PID can be tuned to detect certain classes of organic compounds.
- 6.5 **Retention time:** the elapsed time from the injection of a standard or sample (when START is activated) until detection.

## 7.0 PROCEDURE

7.1 Start up the GC following the steps below:

7.1.1 Connect the GC to the carrier gas bottles.

Note: Nitrogen, if used with the ECD, must be at least 99.9995% hydrocarbon-free.

7.1.2 Install the moisture trap and in-line oxygen trap as close to the injector as possible to ensure carrier gas purity.

7.1.3 Install the column. See the GC operating manual, Reference 3.1.1, for installation instructions.

7.1.4 Test all connections for leaks.

a. Use a liquid leak detector such as Snoop™ on the connections to the oxygen trap.

b. Use a non-liquid leak detector on all gas-line and column connections past the oxygen trap.

7.1.5 Set the desired column flow rate and the total flow output, referring to the operating manual, Reference 3.1.1.

Note: Recommended column flow rates are between 5 and 15 mL/min but may be outside this range depending on the type of analysis performed. When using the ECD detector, total flow must be greater than 50 mL/min.

7.1.6 If using a new column, condition the column according to the manufacturer's instructions.

7.1.7 Enter the oven ramping program and the injector and detector temperatures, referring to the operating manual.

Example: for the VOCOL column with the ECD detector enter:

ECD temperature: 250° C

Injector temperature: 175° C

Program: 35° C for 4.00 min

Ramp at 4.0° C/min to 100° C

Hold at 100° C for 1.00 min

Ramp at 20° C/min to 170° C

Hold at 170° C for 12.00 min

Note: The final temperature and hold time ensure that the column is free of residual contamination.

- 7.1.8 Turn on the detector injector and oven and allow the GC to stabilize at operating temperatures.

Note: Stabilization time will depend on how long the GC has been turned off. A recommended baseline signal of the ECD is less than 20 mV.

- a. Referring to the operating manual, troubleshoot the instrument if the baseline is unstable.

- 7.1.9 Set up the HP 3396 Integrator.

- a. Connect the two INET cables from the back of the integrator to the instrument block on top of the GC.
- b. Set the current date and time.
- c. If using the Chrom Perfect software with the integrator and PC, see Procedure TE-169.

- 7.2 Calibrate the GC. Refer to the specific method to be used for calibration procedures.

- 7.3 Analyze samples. Refer to the specific method to be used for analysis procedures.

- 7.4 When contamination of the column or detector(s) is indicated by an upward drift in the signal level output, bake out either or both of these with the following steps:

7.4.1 Increase the column temperature to 190°C to drive off residual contamination within the column.

7.4.2 Increase the temperature of the ECD to 275°C and the PID temperature to 225°C to purge contaminants from the detectors.

- 7.5 Replace the septum at the end of the day in order for the GC to stabilize overnight.

7.5.1 Turn off the detectors.

7.5.2 Lower the injector and detector temperatures to 35°C.

- 7.5.3 Unscrew the septum retaining nut and remove the septum using forceps or a clean syringe.
- 7.5.4 Quickly install a new septum and replace the retaining nut. **DO NOT OVERTIGHTEN THE RETAINING NUT.**
- 7.6 Turn off the instrument following these steps:
  - 7.6.1 Lower the oven, injector, and detector temperatures to 35° C.
  - 7.6.2 Turn off the detectors.
  - 7.6.3 Turn off the oven.
  - 7.6.4 Turn off the GC.

- 7.5.3 Unscrew the septum retaining nut and remove the septum using forceps or a clean syringe.
- 7.5.4 Quickly install a new septum and replace the retaining nut. **DO NOT OVERTIGHTEN THE RETAINING NUT.**
- 7.6 Turn off the instrument following these steps:
  - 7.6.1 Lower the oven, injector, and detector temperatures to 35° C.
  - 7.6.2 Turn off the detectors.
  - 7.6.3 Turn off the oven.
  - 7.6.4 Turn off the GC.

<b>OAK RIDGE NATIONAL LABORATORY</b> Health Sciences Research Division	<b>PROCEDURE</b>  Environmental Technology Section	Number: TE-164 Page: 1 of 4 Revision: 0 Date: 08/04/95
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**PREPARATION OF CALIBRATION STANDARDS FOR GC ANALYSIS OF VOCs**

**1.0 PURPOSE**

This procedure describes the preparation of calibration standards for analysis of samples for volatile organic compounds (VOCs) using a gas chromatograph (GC).

**2.0 APPLICABILITY**

This procedure applies to the Hewlett Packard (HP) 5890 Series II GC and Photovac 10S50 GC for VOC analysis using the headspace method for water and the solvent extraction method for water and soil.

**3.0 OTHER DOCUMENTS**

**3.1 REFERENCES**

3.1.1 *Test Methods for Evaluating Solid Waste, Methods 8010 and 8020.* SW-846. U. S. Environmental Protection Agency.

**4.0 EQUIPMENT AND MATERIALS**

**4.1 EQUIPMENT**

- 4.1.1 Top-loading balance accurate to 0.01 g
- 4.1.2 Assorted volumetric flasks, 10 to 1000 mL
- 4.1.3 Graduated cylinder

**4.2 MATERIALS**

- 4.2.1 Syringes (10 µL, 25 µL, 50 µL, 100 µL, 250 µL)
- 4.2.2 15-mL vials
- 4.2.3 Pipettes (10-100 µL, 50-250 µL, 100-1000 µL) with disposable tips

APPROVALS	
Author <i>[Signature]</i>	Date 8/30/95
Supervisor <i>[Signature]</i>	Date 9-12-95
Group Leader <i>[Signature]</i>	Date 9-12-95
Section Head <i>[Signature]</i>	Date 9/15/95

- 4.2.4 40-mL VOA vials
- 4.2.5 GC capillary-grade solvent, usually methanol or hexane
- 4.2.6 Solvent and water dispensers
- 4.2.7 Certified standards
- 4.2.8 Deionized (DI) water

## 5.0 RESPONSIBILITY

- 5.1 It is the responsibility of the field laboratory analyst to follow this procedure.

## 6.0 DEFINITIONS

- 6.1 **Calibration Standards:** known concentrations of analytes of interest used to establish a calibration curve from which unknown concentrations in a sample may be determined.
- 6.2 **Solvent:** a liquid used for extracting chemical contaminants from soil or water. Also used as a matrix for calibration standards and as a preservative for VOCs.

## 7.0 PROCEDURE

- 7.1 Prepare calibration standards in water for headspace method.

- 7.1.1 Calculate the amount of prepared standard needed to add to a volume of water to obtain the desired concentration.

Note: At least three calibration concentrations are needed in order to generate a calibration curve. For quantitative results, a five point calibration is needed. Normal concentrations for VOCs in commercially available standards are 200 µg/mL (200 ppm) but may vary depending upon the manufacturer. Check the certification sheet attached to the standard ampule for the correct concentration.

$$\frac{M_1}{V_1} = \frac{M_2}{V_2}$$

where

$M_1$  = mass of analyte to be added, µg

$V_1$  = volume of diluent, mL

$\frac{M_2}{V_2}$  = desired concentration of standard, µg/mL

Example: A concentration of 500 ppb (500  $\mu\text{g}/1000\text{ mL}$ ) in 100 mL of diluent is needed:

$$\frac{M_1}{100\text{ mL}} = \frac{500\ \mu\text{g}}{1000\text{ mL}}$$

$$M_1 = 50\ \mu\text{g of analyte needed}$$

Using prepared stock standard of 200 ppm (200  $\mu\text{g}/1\text{ mL}$ ),  
 $50\ \mu\text{g} \times 1\text{ mL}/200\ \mu\text{g} = 0.25\text{ mL} = 250\ \mu\text{L}$ .  
Therefore, 250  $\mu\text{L}$  of prepared stock standard is needed.

7.1.2 Fill a 100-mL flask with DI water.

7.1.3 Using a clean syringe, remove the same amount of DI water from the flask that will be injected with the prepared standards. For example, if 25  $\mu\text{L}$  of prepared standard is needed to obtain the desired concentration, remove 25  $\mu\text{L}$  of water from the flask prior to adding the standards.

7.1.4 Using a syringe dedicated for calibration standards, withdraw the desired amount of prepared standards from the ampule and transfer it to the flask.

Note: The end of the needle of the syringe must be below the surface of the water. Do not contact the sides of the flask.

7.1.5 Mix by inverting the flask three times.

7.1.6 Transfer the contents of the flask to 40-mL VOA vials leaving zero headspace.

7.1.7 Cap the vials and refrigerate until needed.

Note: The aqueous standards can be stored up to 24 hours.

7.2 Prepare calibration standards in solvent for direct injection method.

7.2.1 Calculate the amount of prepared standard needed to add to a volume of solvent to obtain the desired concentration.

Note: The calculation is the same as outlined in 7.1.1. but the volume of solvent will be substantially less than that of water. Normally, 10 or 20 mL of solvent is used.

**7.2.2** Add the desired volume of solvent to a 15 or 40-mL vial using a graduated cylinder.

- a. Weigh the amount of solvent added to the vial and multiply by the density of the solvent in order to ensure that the correct amount has been added.

**Example:** Tare weigh an empty 15-mL vial. Add 10 mL of solvent to the vial using a graduated cylinder or dispenser. Re-weigh the vial. Assuming hexane is being used as the solvent, the density of hexane at 20° C is 0.66. Therefore, the weight of 10 mL is 6.60 g (assuming a room temperature of 20° C).

- b. In order to reduce evaporation of the solvent during the standard preparation, place about 100 mL of solvent into a volumetric flask.
- c. Cap the flask and store refrigerated until the solvent is cold, about 2 hours.
- d. Use this solvent for preparing the standards.

**7.2.3** Follow instructions in 7.1.3 and 7.1.4.

**7.2.4** Cap the vials. Store refrigerated until needed.

**Note:** Calibration standards prepared in a solvent matrix are more stable than aqueous mixtures. Tightly capped and refrigerated, the standards are usable for up to two weeks.

**7.2.5** See Reference 3.1.1 for further information.

<b>OAK RIDGE NATIONAL LABORATORY</b> Health Sciences Research Division	<b>PROCEDURE</b>  Environmental Technology Section	Number: TE-165 Page: 1 of 11 Revision: 0 Date: 08/03/95
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## GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION

### 1.0 PURPOSE

This procedure describes the analysis of soil and water samples in the field using a gas chromatograph (GC) and solvent extraction.

### 2.0 APPLICABILITY

This procedure applies to the Hewlett Packard (HP) 5890 Series II GC, the HP 7673A autosampler, and the HP 3396 integrator. The GC is equipped with a 30-m capillary column, two injector ports (packed and split-splitless), and two detectors configured for parallel operation [the electron capture detector (ECD) and the photoionization detector (PID)]. The autosampler is mounted over injector B, the split-splitless injector, and is controlled via a sequence file on the integrator. Chromatograms are generated using Chrom Perfect software.

### 3.0 OTHER DOCUMENTS

#### 3.1 REFERENCES

- 3.1.1 HP 5890 Series II GC operating manual
- 3.1.2 HP 7673A autosampler operating manual
- 3.1.3 Chrom Perfect operating manual
- 3.1.4 HP 3396 Series II integrator operating manual
- 3.1.5 Procedure TE-160, Field Operation of the Hewlett Packard 5890 Series II Gas Chromatograph
- 3.1.6 Procedure TE-164, Preparation of Calibration Standards for GC Analysis of VOCs
- 3.1.7 Procedure TE-169, Using the Chrom-Perfect Gas Chromatograph Software

APPROVALS	
Author <i>[Signature]</i>	Date 9/8/95
Supervisor <i>[Signature]</i>	Date 9/14/95
Group Leader <i>[Signature]</i>	Date 9/14/95
Section Head <i>[Signature]</i>	Date 9/14/95

## **3.2 EXHIBITS**

### **3.2.1 Example of Chromatogram and Report**

## **4.0 EQUIPMENT AND MATERIALS**

### **4.1 EQUIPMENT**

- 4.1.1 HP 5890 Series II GC
- 4.1.2 HP 3396 Series II integrator
- 4.1.3 HP 7673A autosampler
- 4.1.4 GC logbook
- 4.1.5 Top-loading balance accurate to 0.01 g

### **4.2 MATERIALS**

- 4.2.1 Gas-tight syringes (manual liquid and autosampler liquid)
- 4.2.2 Autosampler vials
- 4.2.3 40-mL VOA sample vials
- 4.2.4 15-mL dilution vials
- 4.2.5 Pipettes (10-100  $\mu$ L, 50-250  $\mu$ L, 100-1000  $\mu$ L) with disposable tips
- 4.2.6 Capillary GC-grade solvent, usually methanol or hexane
- 4.2.7 0.53  $\times$  30-m megabore capillary column
- 4.2.8 Solvent and water dispensers
- 4.2.9 Deionized (DI) water
- 4.2.10 Waterproof labels
- 4.2.11 5 and 10-mL disposable syringes
- 4.2.12 Certified calibration standard

## **5.0 RESPONSIBILITY**

- 5.1 It is the responsibility of the operator of the HP 5890 Series II GC to follow this procedure.

## **6.0 DEFINITIONS**

- 6.1 **Calibration standards:** known concentrations of analytes of interest used to establish a calibration curve from which unknown concentrations in a sample may be determined.
- 6.2 **Calibration verification:** a QC sample analyzed once per day, preferably before analyzing a set of samples.
- 6.3 **Chromatogram:** a graphic display, in the form of peaks, of the detector's response to a given standard or sample.

- 6.4 **Matrix spike:** a QC sample analyzed once per day.
- 6.5 **Quality check:** a QC sample analyzed once per day.
- 6.6 **Reagent blank:** a quality control (QC) sample analyzed a minimum of once every 20 samples and prior to initial calibration or daily calibration verification. This blank serves to verify that the solvent used is free of contaminants. It also verifies that the GC is free of contaminants and carry-over.
- 6.7 **Replicate sample:** a QC sample analyzed once every 20 samples.
- 6.8 **Solvent:** a liquid used for extracting chemical contaminants from soil or water. Also used as a matrix for calibration standards and as a preservative for volatile organic compounds.

## 7.0 PROCEDURE

- 7.1 Prepare soil sample vials before going into the field.
  - 7.1.1 Calibrate the solvent dispenser before using.
    - a. Adjust the dispenser to 5.0 mL
    - b. Tare a 40-mL VOA vial on the balance.
    - c. Dispense 20 to 30 mL of solvent into the tared vial.
    - d. Weigh the vial and calculate the volume by multiplying the weight of the solvent by the density of the solvent.
    - e. Adjust the dispenser accordingly.
  - 7.1.2 Using the water dispenser, dispense 5 mL of DI water into 40-mL VOA vials.
  - 7.1.3 Using the solvent dispenser, dispense 5 mL of capillary GC-grade solvent into the same vials.
  - 7.1.4 Immediately cap the vials.
  - 7.1.5 Attach waterproof labels to the 40-mL vials.
  - 7.1.6 Weigh each vial to the nearest 0.01 g on the top-loading balance and record the weight on the label.

- 7.1.7 Store the vials in a refrigerator or cooler containing Blue Ice until needed.
- 7.2 Set up the GC as outlined in Procedure TE-160.
- 7.3 Set up the HP 7673A autosampler.
- 7.3.1 Attach the autosampler to the GC following procedures outlined in the HP 7673A autosampler operating manual, Reference 3.1.2.
- 7.3.2 Connect the INET cables between the GC, the integrator, and the autosampler controller as outlined in the autosampler operating manual.
- 7.3.3 Attach a 5 or 10- $\mu$ L autosampler syringe to the tower of the autosampler as outlined in the autosampler operating manual.
- 7.3.4 Fill solvent wash vials A and B with solvent.
- 7.3.5 Place the vials in the respective holders on the autosampler turret.
- 7.3.6 Place empty solvent waste vials A and B in the respective holders on the turret.
- 7.4 Calibrate the GC.
- 7.4.1 Prepare calibration standards following Procedure TE-164.
- a. Prepare a minimum of three calibration standards at concentrations at the low, middle, and high range of expected concentrations of the samples.
- Note: It is recommended that a 5- or 6-point calibration curve be used with concentrations of 10, 50, 100, 250, 500, and 750 ppb. This range of standards establishes a good linear calibration curve. It has been found that concentrations above 750 ppb are beyond the linear range of the instrument.
- 7.4.2 Transfer 1 mL of the calibration standards to the autosampler vials using a pipette.
- a. Use a new pipette tip for each transfer.

- b. Mark the concentrations on the autosampler vials and place them in the autosampler tray.

7.4.3 Set up the options of the autosampler using the integrator.

Note: The integrator controls the autosampler through a sequence file. Commands contained in the file are sent to the autosampler controller via INET cables connecting the integrator, autosampler, and GC.

- a. Press SHIFT and SEQ simultaneously.
- b. The integrator responds with:  
PREP SEQ  
ALS INFORMATION  
INET SAMPLER CONTROL {Y\*/N}  
Press Y and return.
- c. Continue through the list of prompts, referring to the autosampler operating manual for details.

7.4.4 Set up the Chrom Perfect sequence file, referring to the Chrom Perfect operating manual (Reference 3.1.3) for sequence file directions.

Note: This file is used to control the sample information listed on the chromatograms and should not be confused with the sequence file generated on the integrator to control the autosampler.

7.4.5 Analyze the calibration standards.

- a. On the integrator, press SEQ then START. The GC, integrator, autosampler, and Chrom Perfect software will be activated together.

7.4.6 At the end of the calibration, generate a calibration file using data obtained from the standards analysis (see Reference 3.1.7).

- a. Determine that the resulting calibration curve has a linear correlation coefficient greater than 0.98 (this value will vary depending upon the response of the instrument to the chemical component).

- b. If the correlation coefficient is less than 0.98, prepare new standards and re-calibrate.

7.4.7 Record the results of the calibration standards analysis in the GC logbook.

- a. Include the area and concentration for each component and the amount injected into the GC.

7.5 Analyze the soil samples.

7.5.1 Ensure that the amount of sample injected is equal to that of the calibration standards, under normal circumstances 1  $\mu$ L.

Note: Samples that are suspected of being highly contaminated must be diluted in order to remain within the linear range of the GC. Hand-held PID measurements taken in the field at the time of sampling may be useful in determining a good dilution factor. If in doubt, it is better to over-dilute.

- a. For a 1:10 dilution, dispense 4.5 mL of solvent using the solvent dispenser into a 15-mL vial. Using a pipette, withdraw 500  $\mu$ L of solvent, the upper liquid layer, from the sample vial and transfer to the 15-mL vial.
- b. For a 1:100 dilution, dispense 4.5 mL of solvent into a 15-mL vial. Withdraw 500  $\mu$ L of the 1:10 diluted sample and transfer to the 15-mL vial.
- c. For a 1:1000 dilution, follow this same procedure using the 1:100 diluted sample.

7.5.2 Transfer 1 mL of the sample (diluted if necessary) into an autosampler vial using a pipette.

7.5.3 Analyze the sample by repeating steps 7.3.3 through 7.3.5 above.

Note: The amount of sample injected into the GC must be equal to the amount of calibration standard injected during calibration.

7.5.4 On the integrator, press SEQ then START.

7.5.5 Examine the chromatograms. If the area counts of the contaminant peaks do not fall within the calibrated range of the GC, reanalyze the sample at a different dilution.

**Example:** The GC is calibrated for trichloroethene (TCE) using 50, 100, 250, 500, and 750 ppb concentration calibration standards. The detector response in area counts for these concentrations are 4779, 9184, 21,063, 38,997, and 56,155 respectively. The results of a sample analysis show a TCE concentration of 5000 ppb with a detector response in area counts of 340,000. The area counts exceed the area counts (56,155) of the highest calibration standard, 750 ppb. The sample is diluted 1:10 and reanalyzed. Results of this analysis show a TCE concentration of 5025 ppb, with area counts of 39,250, which falls within the calibrated range of the GC. The GC software will automatically calculate the correct concentration for the specified dilution factor.

7.5.6 Record the concentrations and area counts for the target analytes in the GC logbook.

7.5.7 Calculate the concentrations in  $\mu\text{g}/\text{kg}$ :

$$C_w = \frac{M_a}{M_s} \times \frac{0.001 \text{ g}}{1 \text{ kg}}$$

where

$C_w$  = concentration of analyte in soil sample by weight,  $\mu\text{g}/\text{kg}$

$M_a$  = mass of analyte,  $C_v \times V_d$ ,  $\mu\text{g}$

$M_s$  = mass of soil sample, g

$C_v$  = concentration of analyte,  $\mu\text{g}/1000 \text{ mL}$  (this is the concentration reported by the GC in ppb, 1 ppb = 1  $\mu\text{g}/1000 \text{ mL}$ )

$V_d$  = volume of solvent, mL

**Example:** GC analysis of a 5.75 g soil sample in 5 mL of hexane indicates a concentration of 500 ppb.

$$C_v = 500 \text{ ppb} = 500 \mu\text{g}/1000 \text{ mL}$$

$$V_d = 5 \text{ mL}$$

$$M_a = \frac{(500 \mu\text{g})(5 \text{ mL})}{1000 \text{ mL}} = 2.5 \mu\text{g}$$

$$C_w = \frac{2.5 \mu\text{g} \times 0.001 \text{ g}}{5.75 \text{ g} \quad 1 \text{ kg}} = 434.7 \mu\text{g}/\text{kg}$$

7.6 Analyze the water samples.

7.6.1 Dispense 5 mL of solvent into a 40-mL VOA vial using the solvent dispenser.

- 7.6.2 Transfer 5 mL of sample to the 40-mL VOA vial using a 5- or 10-mL disposable syringe.
- 7.6.3 Shake the vial. Allow the solvent and water to separate.
- 7.6.4 Transfer 1 mL of solvent to an autosampler vial.
- 7.6.5 Analyze the sample by repeating steps 7.3.3 through 7.3.5 and 7.5.4 through 7.5.5.
- 7.6.6 On the integrator, press SEQ then START.
- 7.6.7 Calculate the concentrations in  $\mu\text{g/L}$

$$C_{ww} = \frac{M_a \times 1000 \text{ mL}}{V_s \times 1 \text{ L}}$$

where

- $C_{ww}$  = concentration of analyte by weight/volume,  $\mu\text{g/L}$   
 $M_a$  = mass of analyte,  $C_v \times V_s$ , g  
 $V_s$  = volume of sample, mL  
 $C_v$  = concentration of analyte,  $\mu\text{g}/1000 \text{ mL}$  (this is the concentration reported by the GC in ppb, 1 ppb = 1  $\mu\text{g}/1000 \text{ mL}$ )  
 $V_s$  = volume of solvent, mL

## 8.0 QUALITY CONTROL

- 8.1 Analyze a reagent blank.
- 8.1.1 Dispense 5 to 10 mL of solvent into a 40-mL VOA vial using the solvent dispenser.
- 8.1.2 Transfer 1 mL of solvent from the vial into an autosampler vial using a pipette.
- 8.1.3 Analyze the blank by repeating steps 7.3.3 through 7.3.5.
- 8.1.4 On the integrator, press SEQ then START.
- 8.1.5 Examine the resulting chromatogram and record any observations in the GC logbook.
- 8.2 Perform a calibration verification.

8.2.1 Prepare and analyze initial calibration standards as outlined in 7.4.

8.2.2 Calculate the calibration factor:

$$\text{Calibration factor} = \frac{\text{total area of peak}}{\text{concentration of standard}}$$

8.2.3 Calculate the percent difference:

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where

$R_1$  = calibration factor from initial calibration

$R_2$  = calibration factor from succeeding analysis

8.2.4 Verify that the difference is  $\pm 20\%$  of the initial calibration. If it is not, recalibrate the GC.

8.3 Perform a quality check once per day.

8.3.1 Prepare the quality check sample.

- a. Obtain certified standard. Do not use the same certified standard used in the preparation of the calibration standards.
- b. Following procedure TE-164, prepare one or more quality check samples of concentrations at or near the mid-point of the calibration curve (200 to 600 ppb).

8.3.2 Transfer 1 mL of the quality check sample to an autosampler vial using a pipette.

8.3.3 Analyze the sample.

8.3.4 Verify that the concentration reported by the analysis is within  $\pm 20\%$  of the calculated concentration (see 8.2.2).

8.4 Analyze a replicate sample once every 20 samples.

8.4.1 Using a pipette, transfer 1 mL of the sample extract into an autosampler vial.

8.4.2 Repeat the above procedure. Label the second sample as the replicate.

8.4.3 Analyze both samples. Verify that the results are within  $\pm 20\%$  (see 8.2.2).

8.5 Perform a matrix spike once per day.

8.5.1 Using a pipette and either a calibration standard or quality check sample, transfer 1 to 5 mL into a previously analyzed soil sample.

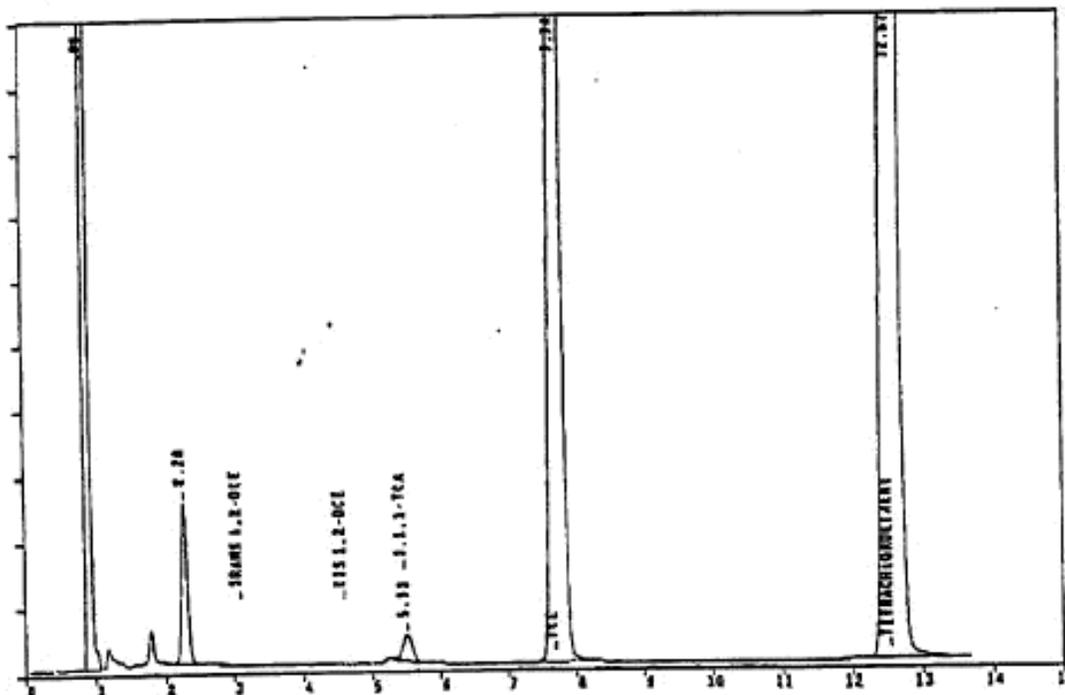
8.5.2 Analyze the sample.

8.5.3 Determine the calculated amount of analyte contained in the matrix spike.

8.5.4 Verify that the reported concentration of the matrix spike sample is within  $\pm 20\%$  of the calculated concentration.

Exhibit 1. Example of Chromatogram and Report

File=C:\CP\DATA1\TUCSII.62R Sample name=SV129 Date printed= 12-11-1993 Time= 11:41:23  
0.00 to 15.00 min. Low Y = -0.08749 mv High Y = 0.41251 mv Span = 0.50000 mv



C:\CP\DATA1\Q78EA97B.BNC  
Data file = C:\CP\DATA1\TUCSII.62R  
Date stamp = 12/11/93 Time = 11:41:16  
Sample name = SV129  
Collected on DEC 11, 1993 11:26:50 from port # 1  
Operator = J.L. ZUTMAN  
Reference file name = Q78EA97Bb#66  
Instrument = HP5890  
Method name = C:\CP\DATA1\TUCIIAIR.MET version # 3  
Date method last modified = 12/08/93 Time = 16:41:38  
Calibration file = C:\CP\DATA1\TUCIIAIR.CAL version # 10  
Date cal file last modified = 12/08/93 Time = 17:34:38  
Run time = 13.67 minutes Area reject = 100  
Amount injected = 1 Dilution Factor = 1  
Sample Weight = 1 Internal Standard Amount = 0  
Sampling rate = 10 per second  
Peak detect threshold = 0 Starting peak width = .04 minutes  
Chrom-Perfect Software Serial # 12948 Version = 5.05 For Martin Marietta  
Today's date = 12-11-1993 Time = 11:41:47

PK	Ret Time	Name	Amount	Amount %	Area	Area %	Type	Width	Height	Height %
1	0.888		0.0000	0.0000%	19,654.3	26.316%	BB	0.042	7,861.79	57.9427%
2	2.280		0.0000	0.0000%	701.5	0.939%	BB	0.096	122.10	0.8999%
3	5.505	1,1,1-TCA	11.7708	0.4479%	183.0	0.245%	BB	0.150	20.31	0.1497%
4	7.737	TCE	1,204.8097	45.8427%	12,558.5	16.815%	BB	0.154	1,356.37	9.9966%
5	12.575	TETRACHLOROETHENE	1,411.5570	53.7094%	41,589.7	55.685%	BB	0.165	4,207.65	31.0111%
Total area = 74686.94			Total amount = 2628.137		sample units = ug/l		Total height = 13568.23			

## APPENDIX C

### Oak Ridge Sample Management Office Procedures

COMMERCIAL LABORATORY SELECTION PROCESS FOR THE ANALYTICAL SUPPORT AGREEMENT	LMES-ASO-AP-203, REV. 0
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U.S. DEPARTMENT OF ENERGY OAK RIDGE OPERATIONS ENVIRONMENTAL INFORMATION MANAGEMENT ORGANIZATION - VOLUME II ADMINISTRATIVE PROCEDURE		LMES-ASO-AP-203			
		APPROVAL:			
		PAGE 1 OF 6      REVISION 0			
		NEXT REVISION:			
QA MANAGER	Date	EIMO BRANCH CHIEF	Date	ERD DIRECTOR	Date

**TITLE:      COMMERCIAL LABORATORY SELECTION PROCESS FOR THE ANALYTICAL SUPPORT AGREEMENT**

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**1.0      PURPOSE**

This procedure describes the process for selecting, adding and expelling commercial laboratories to the LMES Pricing Agreement (PA).

**2.0      SCOPE**

This procedure is applicable to all prime contractors to the Department of Energy Oak Ridge Operations (DOE-ORO) and all subcontractors to DOE prime contractors.

**3.0      ACRONYMS**

- DOE-ORO    Department of Energy Oak Ridge Operations
- EIMO        (DOE) Environmental Information Management Organization
- FAR         Federal Acquisition Regulations
- IPIP        Integrated Performance Indicator Program
- LMES        Lockheed Martin Energy Systems
- LSB         Laboratory Selection Board
- PA          Pricing Agreement
- SMO        Sample Management Office

**4.0      DEFINITIONS**

- 4.1        Analytical Support Agreement - A set of terms and conditions agreed to by commercial laboratories and LMES to provide analytical services to environmental restoration, waste management, research and compliance groups. Laboratories are accepted into the analytical support agreement pending satisfaction of specific requirements and the laboratory's acceptance

of a LMES purchase order. The analytical support agreement is also known as the Pricing Agreement (PA).

4.2 Laboratory Selection Board - A committee comprised of the Oak Ridge Sample Management Office (SMO) Operations Manager, and appointees from all prime contractors conducting business with the SMO. The LSB evaluates the current laboratory list and determines the need for additional capacity or unique capability and makes recommendations for laboratory addition or expulsion to the SMO Manager and EIMO Branch Chief who confirms the need for assessment of penalties using the results of the IPIP and audit program.

4.3 Integrated Performance Indicator Program -

## **5.0 RESPONSIBILITIES**

### **5.1 SMO Operations Manager**

5.1.1 Monitors the current laboratory list and incoming project requirements and project needs for additional capacity or the need to reduce existing capacity.

5.1.2 Serves as a member and Chairman of the Laboratory Selection Board. Convenes the Laboratory Selection Board as needed. Documents the selection criteria; and documents the recommendations of the Laboratory Selection Board.

### **5.2 SMO Manager**

5.2.1 Makes final decision along with EIMO Branch Chief on recommendation by Laboratory Selection Board.

### **5.3 EIMO Branch Chief**

5.3.1 Makes final decision along with SMO Manager on recommendation by Laboratory Selection Board.

### **5.4 Laboratory Selection Board**

5.4.1 Ranks commercial laboratories that have submitted requests to join the Pricing Agreement. Recommends additions and expulsions of commercial laboratories to the SMO Manager based on this ranking.

### **5.5 LMES Subcontract Administrator**

5.5.1 Completes the Terms and Conditions document, interfaces with commercial laboratories to request pre-pricing.

## 6.0 PROCEDURE

### 6.1 Commercial Laboratories Application and Recertification

6.1.1 All **commercial laboratories** wishing to participate in the LMES Pricing Agreement will contact the SMO Operations Manager and provide the documents listed below:

1. Certificate of D & D insurance;
2. Certification of financial status;
3. Radiological license per Terms and Conditions;
4. Acceptable Laboratory Quality Assurance Plan;
5. Evidence of satisfactory performance in a nationally recognized Performance Evaluation Program for 1 year per Terms and Conditions;
6. Waste Management Plan per Terms and Conditions;
7. Chemical Hygiene Plan per Terms and Conditions;
8. Radiological Inventory Plan per Terms and Conditions;
9. Completed, signed and returned the LMES Pricing Agreement.

6.1.2 The **SMO Operations Manager** will assign a staff member to make the review and respond to the laboratory for (1) acceptance of the documents, or (2) request for additional information or (3) reason for rejection of submitted documents. The staff member will approve the content and specifications of the documents and the **SMO Manager** will concur.

6.1.3 The status of applicant laboratories will be either (1) approved for Pricing Agreement selection or (2) awaiting approval for Pricing Agreement selection. "Awaiting approval" status indicates the application has been received and the documents have not been reviewed, incomplete, insufficient, or inadequate. "Approved" status indicates that all prerequisite requirements have been submitted and are acceptable.

6.1.4 Annually, each **laboratory** that has been approved for Pricing Agreement selection for the previous year will be requested to resubmit their request for participation and verify the status of all the prerequisites. (Appendix A)

### 6.2 Selection and Addition of Laboratories to the Pricing Agreement List

6.2.1 The **SMO Operations Manager** will convene the **Laboratory Selection Board** as deemed appropriate to consider additions to the laboratory list on the following conditions.

- a. Recent solicitations of laboratories did not have sufficient quotations, leading to the conclusion there is inadequate number of participants.

- b. Either the **SMO Manager** or **EIMO Branch Chief** makes a formal request to add capacity to the laboratory list.
- 6.2.3 Upon convening the Laboratory Selection Board, the **SMO Operations Manager** will call for the selection criteria to be established taking into consideration, at a minimum, SMO needs, quality, services, personnel, facilities, pricing, experience, current certifications. Each criterion will be assigned a ranking factor before selection begins.
- 6.2.4 The **Laboratory Selection Board** will assign a score for each of the pre-approved applicant laboratories on each criterion and will calculate an overall score.
- 6.2.5 The **Laboratory Selection Board** will recommend the laboratory with the highest overall score to the **SMO Manager** and **EIMO Branch Chief** for concurrence and approval.
- 6.2.6 Once concurrence is gained the **EIMO** will arrange for an on-site prequalification audit.
- 6.2.7 If the laboratory passes the prequalification audit, then the **SMO Operations Manager** will sign the required Federal Acquisition Regulations (FAR) forms and will send them to the **LMES Subcontract Administrator** who will complete the Terms and Conditions document, and notify the laboratory to request pre-pricing.
- 6.2.8 The **SMO Operations Manager** will ensure the laboratory is added to the approved laboratory list and added to the TRACKER data base.
- 6.3 Expulsion of Laboratories from the Pricing Agreement List**
- 6.3.1 If conditions exist to justify expulsion, the **LSB** may recommend expulsion of a laboratory from the Pricing Agreement. The **LSB** may consider IPIP results, facilities personnel changes, audit reports, monthly progress reports and other relevant documentation.
- 6.3.2 The **SMO Manager** and **EIMO Branch Chief** must concur to expel any laboratory on the Pricing Agreement list upon recommendation of the **Laboratory Selection Board**.
- 6.3.3 Upon recommendation by the **LSB** and concurrence by the **SMO Manager** and **EIMO Branch Chief**, **LMES** will remove a laboratory from the Pricing Agreement.
- 7.0 REPORTS/RECORDS**
- 7.1 The minutes and recommendations of Laboratory Selection Board will be maintained as record copies in the SMO Operations Office.

## **8.0 REFERENCED DOCUMENTS**

- 8.1 Federal Acquisition Regulations
- 8.2 LMES Pricing Agreement
- 8.3 Department on Energy Acquisition Regulations
- 8.4 Laboratory Auditing DOE-ORO-EIMO-AP-202

## **9.0 APPENDIXES**

Appendix A Oak Ridge Sample Management Operations annual recertification of application for addition to Pricing Agreement.

Appendix A

**Oak Ridge Sample Management Operations**

**Annual Recertification of Applicant for Addition to Analytical Support Agreement**

The undersigned laboratory recertifies an interest in participating in the Oak Ridge Sample Management Office Analytical Support Agreement. The laboratory has not changed significantly in key management, facilities, financial status, ability to perform the previously indicated analyses or the ability to receive, analyze, report data, track radiological inventory, and dispose of radiological waste, from the previously submitted documents. Furthermore, the laboratory understands that the application and signing of the Lockheed Martin Energy Systems, Inc. Analytical Support Agreement does not represent an obligation or commitment of Lockheed Martin Energy Systems, Inc. to enter into the Agreement. This recertification simply indicates the intention of the laboratory to participate in the Analytical Support Agreement if selected.

Signature: \_\_\_\_\_

Please Print Name: \_\_\_\_\_

Laboratory: \_\_\_\_\_

Date: \_\_\_\_\_

## **TITLE: CONTRACTING OF ANALYTICAL WORK TO COMMERCIAL LABORATORIES**

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### **1.0 PURPOSE**

This procedure defines the methodology used by Oak Ridge Sample Management Office (SMO) personnel in processing statements of work (SOWs), processing purchase requisitions, and selecting commercial analytical laboratories. These activities for the procurement of commercial laboratory services are to support projects sponsored by the Department of Energy (DOE) Oak Ridge Operations Office. The procedure serves to ensure that as an operation of a DOE contractor, LMES SMO maintains an optimum level of technical and administrative oversight on each project, and SMO commercial procurement activities comply with federal acquisition laws and LMES procurement policy.

### **2.0 SCOPE**

This procedure is applicable to all SMO personnel tasked to procure commercial analytical laboratory services in support of DOE Oak Ridge Operations.

### **3.0 ACRONYMS**

ASO	Analytical Services Organization
DOE-ORO	Department of Energy Oak Ridge Operations
CLP	Contract Laboratory Program
CWA	Clean Water Act
EIMO	(DOE) Environmental Information Management Organization
LMES	Lockheed Martin Energy Systems
NPDES	National Pollutant Discharge Elimination System
PE	Performance Evaluation
RCRA	Resource Conservation and Recovery Act
SMO	Sample Management Office
SOW	Statement of Work
WAC	Waste Acceptance Criteria

### **4.0 DEFINITIONS**

- 4.1 Administrative Procedures - written procedures to explain nonanalytical, administrative operations or tasks.
- 4.2 Customer - the project or program manager, engineer, or other individual responsible for a DOE/ORO-funded task who obtains associated analytical services through the SMO.

- 4.3 Analytical Support Agreement - An agreement between approved commercial laboratories and LMES procurement. The agreement defines the technical and administrative terms and conditions of providing analytical laboratory services.
- 4.4 SMO Customer Representative - a staff member of the SMO Operations Office who supplies pricing and other key information to the customer, assures the correctness and completeness of SOWs, brokers commercial analytical work, and serves as liaison between the Customer and the laboratory.

## **5.0 RESPONSIBILITIES**

### **5.1 SMO Customer Representative**

- 5.1.1 Identify all the cost, quality, and timeliness elements in a project deliverable.
- 5.1.2 Ensure all potential awardees are in compliance with those quality standards defined in the SOW and relevant sections of the current terms and conditions of the LMES Analytical Support Agreement.

### **5.1 SMO Customer Representative (Cont.)**

- 5.1.3 Ensure the technical and business details of each SOW are complete and accurate prior to issuance of a commercial laboratory solicitation for support.
- 5.1.4 Ensure those Analytical Support Agreement laboratories designated as Small Business, Small Disadvantaged Business, and 8(a) Business under DOE regulation 970.7104-12 are included in all solicitations.
- 5.1.5 Maintain clear, concise, current records to document all technical and business activities associated with each SOW as per SMO requirements in SMO procedure LMES-ASO-AP-209.
- 5.1.6 Ensure the analytical requirements are consistent with applicable regulatory requires (e.g., NPDES, RCRA, CLP, WAC, CWA).

### **5.2 LMES Subcontract Administrator**

- 5.2.1 Ensure the SMO solicitation and award process complies with federal acquisition and DOE regulations and LMES procurement policy.
- 5.2.2 Document the level of competitiveness associated with the SMO solicitation process.
- 5.2.3 Monitor all SMO procurement activities.

5.2.4 Reviews price comparisons

### 5.3 SMO Operations Manager

5.3.1 Supervise all SMO internal procurement activities.

5.3.2 Ensure the availability of all SMO job related resources.

5.3.3 Provide all pertinent information which ensures proper SMO interfaces within the EIMO organization.

5.3.4 Manage all SMO technical related activities.

5.3.5 Ensure proper training of SMO operation personnel.

### 5.4 SMO Customer

5.4.1 Initiates SOWs in TRACKER

5.4.2 Is available for laboratory readiness review

5.4.3 Contacts SMO Customer Representative with scope or schedule changes to the SOW

## 6.0 PROCEDURE

### 6.1 Processing an SMO Statement of Work (SOW)

6.1.1 The **Customer** submits an SOW to the SMO Operations Office via the TRACKER system. TRACKER automatically sends an electronic mail message to all affected SMO Customer Representatives notifying them of the submission. The **Customer** may request that the SMO Customer Representative initiate the SOW.

6.1.2 The **SMO Customer Representative** ensures each SOW includes the following technical and business information and that this information is reviewed relative to the project description using governmental regulatory and WAC documents for guidance:

ID number	Matrix
Date	Deliverables
Project Description	Analyte
Project Manager	Proponent of the method
Project Finance Officer	Required quantitative limits
Turnaround Time	

- 6.1.3 If a technical irregularity exists within the SOW, the **SMO Customer Representative** consults the **Customer** immediately for explanation and resolution.
- 6.1.4 The **SMO Customer Representative** splits the SOW into functional subsets, such as nonradiological analyses, radiological analyses, dioxins/furans, and physical measurements, to allow for a broader competitive base. This is done if the Customer does not object.
- 6.1.5 The SMO Customer Representative faxes the SOW to the **Customer** for signature. After the **Customer** returns the signed copy to the SMO office, the files it in the folder which has been prepared by the department secretary.

## 6.2 Procurement of Analytical Services

To procure analytical services, the **SMO Customer Representative** follows these steps:

- 6.2.1 Obtain an estimate of the value of each section of the finalized SOW using the TRACKER “pricing” module.

- 6.2.2 Process SOW sections which have an estimated value below \$100,000.00 as follows:

If the SOW has **an estimated valued of less than \$25,000.00** and the matrix/method are conventional, award the work to one of the Analytical Support Agreement laboratories based on capability, capacity, and the lowest listed price without solicitation of bids, then proceed to 6.2.3.10. The price must be current and taken from the TRACKER pricing module Comparison of Offers spreadsheet.

- 6.2.3 If the SOW has an estimated value of **greater than \$100,000**, contact the LMES Subcontract Administrator to review the SOW pricing.

- 6.2.4 When the SOW has **an estimated value between \$25,000.00 and \$100,000**, or the requirements do not fit the Analytical Support Agreement, issue a solicitation for spot quotation as follows:

- 6.2.4.1 Prepare a cover letter (Appendix B) and the SMO spot quote form (Appendix C) requesting pricing for project-specific requirements. Include the following information:

- Analytical method
- Data deliverable categories
- Electronic media deliverable
- Turnaround time

- 6.2.4.2 Send the price comparisons to the **LMES Subcontract Administrator** for review.

- 6.2.4.3 Fax the cover letter, spot quote, and a copy of the SOW to each of those competitive Analytical Support Agreement laboratories. Retain the fax confirmation in the SOW folder.
- 6.2.4.4 Allow three to four days for all solicitation responses. Enter each laboratory's spot quotation response into the TRACKER pricing module data base. Include all information concerning request for alternative methods, reporting modifications, no-bids, etc., in the "NOTES" section of the database. Place the actual spot quotations in the SOW folder.
- 6.2.4.5 From the TRACKER reports module, print the project Pricing Report associated with the SOW as described in the TRACKER User Manual.
- 6.2.4.6 Consult with the SMO technical assessment staff on responses requesting deviations, alternatives, or modifications. Document the acceptability or unacceptability of each request, and inform the Customer of the evaluation.
- 6.2.4.7 Request a written agreement with, or rejection of, the evaluation from the Customer. The Customer makes the final decision about acceptability.
- 6.2.4.8 If request for deviations, alternatives, or modifications are not acceptable, consider the requesting laboratory nonresponsive to the spot quotation, and eliminate it as a potential awardee. If the requests are acceptable, extend a "Best and Final" opportunity for rebid, inclusive of the acceptances, to each laboratory participating in the original spot quotation. Revise the original request for spot quotation to reflect the acceptable deviation, and resubmit it to the laboratories for quotation. Follow steps 6.2.4.1 through 6.2.4.3. After the "Best and Final" notifications are issued, no further deviations from the SOW by the laboratory are allowed.
- 6.2.4.9 Enter the final spot quotations into the TRACKER "pricing" module as described in step 6.2.4.3 and a comparison of offers is printed as indicated in step 6.2.4.4.
- 6.2.4.10 Select an awardee based on capability and bid price. TRACKER will suggest an awardee, which can be overridden based on the information available.
- 6.2.4.11 The **SMO secretary** prepares and faxes a Letter of Placement to the project manager, and a Quotation Offer Sheet to the awarded laboratory informing each of the SMO intent to contract. The Quotation Offer Sheet includes direction to the laboratory to fax its response of acceptance.

**NOTE:** If the intended awardee does not respond, or does not confirm its bid, offer the work to the next lowest bidder and repeat step 6.2.3.10.

### **6.3 Processing an LMES Purchase Requisition:**

To process an LMES purchase requisition to cover the SOW, the **SMO Customer Representative** follows these steps:

- 6.3.1 Prepare a purchase requisition, form UCN-14715, making sure to include the following information:

- Requisition number
- Date prepared
- Urgency
- Vendor name (Awardee)
- Account number ( project charge number)
- Deliver to Operations Manager
- Noun = Sample Analysis
- Quantity = Estimated unit price (comparison of offers price + 10%)
- Description of the project

- 6.3.1 Prepare a purchase requisition, form UCN-14715, making sure to include the following information (Cont.):

- Requester (**SMO Customer Representative**) signature
- Requester (**SMO Customer Representative**) badge number
- Phone (**SMO Customer Representative**)
- Authorized signature (**SMO Operations Manager**)
- Approval employee number (**SMO Operations Manager**)

- 6.3.2 Submit the purchase requisition to the **SMO Operations Manager**.

- 6.3.3 Fax the signed purchase requisition to the project finance officer (listed in the SOW) for signature and request it be faxed to the SMO Operations Office upon completion.

- 6.3.4 Fax the signed purchase requisition to the SMO finance officer for signature and request it be faxed to the SMO Operations Office upon completion.

- 6.3.5 Fax the signed purchase requisition to material control for entry into the purchasing department computer data base and issuance of a purchase order.

- 6.3.6 Ensure a copy of the signed (both finance officers) purchase requisition, and the fax logs are filed in the SOW folder.

- 6.4 Prior to sample shipment, establish a conference call for a laboratory readiness review (on-site readiness review is optional) and include the **Customer**, the laboratory representative(s), and the **SMO technical staff** to ensure agreement among all parties on the project requirements and laboratory needs. Document each expressed concern and accepted resolution, and file the conference notes in the associated SOW folder.

## **7.0 REPORTS/RECORDS**

- 7.1 Statement of Work (TRACKER-generated)
- 7.2 Purchase Requisition Form UCN-14715
- 7.3 Comparison of Offers (TRACKER-generated)
- 7.4 Project FAX logs

## **8.0 REFERENCE DOCUMENTS**

- 8.1 Lockheed Martin Energy Systems Analytical Support Agreement and Terms and Conditions, LMES Procurement Division, Environmental Services Group. September 9, 1996.
- 8.2 K/DSRD-3020/D Oak Ridge Sample Management Office TRACKER User's Manual for SMO Users
- 8.3 K/DSRD-3022/D Oak Ridge Sample Management Office TRACKER User's Manual for Customers
- 8.4 Sample Management Office Records Management. LMES-ASO-AP-209

## **9.0 APPENDIXES**

- Appendix A: SOW format
- Appendix B: Spot Quote letter
- Appendix C: Spot Quote form
- Appendix D: Comparison of Offers form
- Appendix E: Spot Quote Offer
- Appendix F: Offer Letter for Pricing Agreement Awards

CONTRACTING OF ANALYTICAL WORK TO  
COMMERCIAL LABORATORIES

LMES-ASO-AP-210, REV. 0

SOW:  
REV:  
SOW GROUP:

OAK RIDGE SAMPLE MANAGEMENT OFFICE  
ANALYTICAL STATEMENT OF WORK

<b>PROJECT DESCRIPTION:</b>	
<b>PROJECT NUMBER:</b>	<b>PROJECT MANAGER:</b> <b>TELEPHONE:</b> <b>FAX:</b> <b>ADDRESS:</b>
<b>FINANCE OFFICER:</b> <b>TELEPHONE:</b> <b>FAX NO.:</b> <b>ALTERNATE:</b>	<b>CHARGE NUMBER:</b> <b>ADS:</b> <b>B&amp;R NO:</b> <b>FUNDING SOURCE:</b>
<b>SAMPLE START DATE:</b>	
<b>SAMPLE COMPLETION DATE:</b>	
<b>SAMPLING EVENTS:</b>	
<b>SAMPLE QUANTITY:</b>	
<b>SAMPLE DISPOSAL:</b>	<b>REQUIRED ARCHIVAL:</b> _____ <b>Months</b>
<b>SUSPECTED HAZARDS:</b>	<b>TSCA REGULATED:</b> (PCBs > 50 ppm)
<b>ISOTOPES OF CONCERN:</b> <b>ESTIMATED LEVEL OF RADIOACTIVITY:</b>	
<b>SHIPPING RECEIPT:</b> The project manager is to fax a copy of the Sample Shipping Receipt to the OR SMO and the Project Manager in addition to a copy of the Chain of Custody with each shipment to include SOW.	

SOW:

(All turnaround times are in calendar days.)

QTY	MATRIX TYPE	PROONENT OF METHOD	METHOD NUMBER	ANALYTE	REQ DET LIMIT	TURN TIME
1	WATER	EPA	200.7	All Analytes	PER METHOD	30
410	WATER	EPA	245.1	Mercury	PER METHOD	30
1	WATER	EPA	300.0	All Analytes	PER METHOD	30
2	WATER	EPA	Technetium 99	Technetium-99	10 pCi/L	30

SOW:

**QUALITY CONTROL REQUIREMENTS / DATA DELIVERABLE:**

<u>METHODOLOGY</u>	<u>QC REQUIREMENTS</u>	<u>DATA DELIVERABLE</u>
METALS	PER APPROPRIATE AMS	FORMS ONLY
RADIOLOGICAL	PER APPROPRIATE AMS	FORMS PLUS RAW DATA
WET CHEM	PER APPROPRIATE AMS	FORMS ONLY
OTHER	SEE COMMENT/ATTACH.	FORMS ONLY

QC COMMENTS:

DATA DELIVERABLE COMMENTS:

**ANALYTICAL BATCH REQUIREMENTS:**

**REPORTING REQUIREMENTS: (Report results formally to:)**

Original: \_\_\_\_\_

Copy:

James A. Ealy  
Lockheed Martin Energy Systems, Inc.  
Post Office Box 2003  
Blair Road  
Oak Ridge, TN 37831-7169

**ATTACHMENTS:**

\_\_\_\_\_  
Project Manager

\_\_\_\_\_  
Date

\_\_\_\_\_  
Approving Manger (Optional)

\_\_\_\_\_  
Date

\_\_\_\_\_  
SMO Representative

\_\_\_\_\_  
Date

**SAMPLE RECEIPT CONFIRMATION:** The laboratory is to fax a copy of the Chain-of-Custody to the OR SMO and the project manager with any remarks, signature, date, and time with each shipment.

**Lockheed Martin Energy Systems**

James A. Ealy  
Sample Management Office  
Post Office Box 2003      Oak Ridge, Tennessee 37831 - 7169  
Telephone: 423-576-2724      Facsimile: 423-574-9433

July 25, 2000

FIELD(Title) FIELD(FirstName) FIELD(LastName)  
FIELD(Company)  
FIELD(Address1)  
FIELD(Address2)  
FIELD(City), FIELD(State) FIELD(PostalCode)

Dear FIELD(Title) FIELD(LastName):

**SPOT QUOTATION OF  
SOW-**

The Sample Management Office is seeking spot quotations from approved Pricing Agreement laboratories for the above referenced project. The spot quotations are specific only for the above project and your response and pricing is not constrained by the previous prices submitted under the Pricing Agreement. All terms and conditions shall apply except as modified in the statement of work. The award will be made on an "all or none" basis.

This project has requirements that are not covered in the Pricing Agreement. This invitation is being sent to all non-rad approved Pricing Agreement laboratories. Pricing for this spot quotation **will not** be added to the Pricing Agreement as this is a project-specific spot quotation. The pricing submitted should consider the entire project requirements as to methods, required quantitation limits, quality control, turnaround times, and data deliverables.

SOW- is enclosed for your evaluation along with a consolidation of SOW- . Your quotation should include a price per sample per the enclosure. The number of samples indicated in the enclosure is an estimate and may not represent the actual number of samples submitted. However, the award will be based on the extension of the estimated number of samples multiplied by the quoted price. As always, the award will be offered to the laboratory submitting the overall lowest quote that is technically acceptable for the specific project. The laboratory must confirm or reject the offer without changing any pricing.

All responses which are to be considered for this project must be received in this office by **3:30 p.m. EST**

**on** . As always, I am available to answer any question you may have on this project (423-576-2724). Thank you for your continued cooperation and responsiveness.

Sincerely,

James A. Ealy  
Operations Manager  
Sample Management Office

JAE:cds  
Enclosures  
By Fax  
cc: File-SMO-RC

Attachment

**SPOT QUOTATION FOR SOW-**

**Lab:** \_\_\_\_\_

**Date:** \_\_\_\_\_

Synopsis of Work:

Estimated No. Samples	Matrix	Parameter	Method	Price/ Sample

**TOTAL AMOUNT FOR PROJECT: \$** \_\_\_\_\_

**Samples to be Shipped:**

**Holding Time:**

**TAT:**

**Data Deliverables:**

**Electronic Data Deliverable:**

**QC Requirements:**

**Estimated Level of Radioactivity:**

TNVT - to host smo1.dsr.d.onl.gov

Session Edit Commands Settings Script Help

SMD - COMPARISON OF OFFERS  
SPOT QUOTE

SOW: 0000

LABORATORY:

Matrix	Proponent	Method	Quantity	Cost

COMMENT:  Total Cost: \$.00

Add/Change(1) Delete(2) Clear(0) Previous(F3)

12:07:33

**Lockheed Martin Energy Systems**

James A. Ealy  
Sample Management Office  
Post Office Box 2003  
Telephone: 423-576-2724

Oak Ridge, Tennessee 37831 - 7169  
Facsimile: 423-574-9433



July 25, 2000

Dear :

**SOW-**

The Sample Management Office (SMO), on the behalf of Lockheed Martin Energy Systems, Inc. (the ACompany@), is pleased to inform you that it is the intent of the Company to award to you the subject statement of work based on your spot quotation for the project. Project work shall not begin until a subcontract/purchase order has been issued by the Company. Please confirm the project price of \$ \_\_\_\_\_, on the estimated number of samples to be submitted and your review of the complete statement of work. Please note that your laboratory was provided a copy of the statement of work (SOW) prior to receiving your quotation. The SMO recognizes that the actual number of samples submitted may vary from the estimate and will approve remittance based on your quoted unit price and the actual number of samples submitted. As previously stated, the terms and conditions of the Pricing Agreement remain in effect except for the pricing received as a project specific quotation.

The SMO would like to receive your acceptance confirming the pricing or declination of this SOW within 24 hours of receipt of this fax. As always, I am available to answer any technical questions related to this project.

The SMO appreciates the continued cooperation and support of your laboratory. Please contact me at 423-576-2724, if you need additional information.

Sincerely,

James A. Ealy  
Operations Manager  
Sample Management Office

Approved By: \_\_\_\_\_  
Subcontract Administrator

Date: \_\_\_\_\_

JAE:cds  
By Fax (1 page)  
cc: File-SMO-RC

**Lockheed Martin Energy Systems**

James A. Ealy  
Sample Management Office  
Post Office Box 2003  
Telephone: 423-576-2724

Oak Ridge, Tennessee 37831 - 7169  
Facsimile: 423-574-9433



July 25, 2000

Dear :

**SOW-**

The Sample Management Office (SMO), on the behalf of Lockheed Martin Energy Systems, Inc. (the "Company") is pleased to inform you that it is the intent of the Company to award to you the subject statement of work based on a comparison of all pricing of all laboratories participating in the Pricing Agreement with Lockheed Martin Energy Systems, Inc. The comparison was made on the complete requirement to include matrix, method, turnaround time, data deliverables, and electronic data deliverables.

Following is our assessment of the subject statement of work:

MATRIX	
METHOD	
UNIT PRICE	\$
DATA DELIVERABLE	
MULTIPLIER FOR TAT DELIVERABLE	
ELECTRONIC DATA DELIVERABLE	
TOTAL NUMBER OF SAMPLES	
TOTAL ESTIMATED PROJECT PRICE	

As previously agreed, the terms and conditions in the Pricing Agreement will remain in effect. Additionally, the SMO recognizes that the actual number of samples submitted is an estimate and the SMO will approve remittance based on the unit price and number of samples submitted.

The SMO would like to receive your acceptance confirming the pricing or declination of this SOW within 24 hours of receipt of this fax. As always, I am available to answer any questions on this project.

Sincerely,

James A. Ealy  
Operations Manager  
Sample Management Office

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_  
Subcontract Administrator

By Fax  
cc: File-SMO-RC

## APPENDIX D

### Reference Laboratory Analytical Procedures for PCB Soil Analyses

LAS Laboratories  
Las Vegas, NV



**DETERMINATION OF ORGANOCHLORINE  
PESTICIDES AND PCBS BY  
METHODS 8080/8080A/8081/608**

**LAL-91-SOP-0101**

*Jon Humes* *D. L. L. L.* 9/11/96  
Prepared by Date

*David Callahan* 9-11-96  
Reviewed by Supervisor Date

*James L. Aensour* 9/11/96  
Reviewed by QA Date

*Nathan J. Dunn* 9/12/96  
LAS Health and Safety Officer Date

*Ch. W. H.* 9-12-96  
Laboratory Director Date

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## 1.0 PURPOSE

The purpose of this SOP is to describe the procedures necessary to determine Organochlorine Pesticides and Polychlorinated Biphenyls PCBs (Aroclors) according to the following methodologies:

SW-846 EPA Method 8080 with Capillary Chromatography  
SW-846 EPA Method 8080A with Capillary Chromatography  
SW-846 EPA Method 8081  
EPA Method 608

The SOP also includes information obtained from instrument manuals and practical laboratory experience. It is to be used closely in conjunction with the specific analytical methods listed above as well as applicable project-specific documents.

In this SOP, where different procedures are required for method compliance with a specific method listed above, those variations are described. This is most evident in the calibration discussions in this SOP.

## 2.0 SCOPE AND APPLICATION

- 2.1 This procedure is a gas chromatographic (GC) method which utilizes dual columns and dual electron capture detectors (ECDs). The method must first be preceded by appropriate sample extraction and cleanup procedures.
- 2.2 This procedure is to be used by experienced LAS GC-ECD personnel as a guide for the determination of Organochlorine Pesticides and PCBs according to EPA Method 8080/8080A/8081/608 using a Hewlett Packard Model 5890 Series II Gas Chromatograph equipped with dual columns and dual electron capture detectors.
- 2.3 LAS Default Target Analyte List (TAL) and Additional Target Analytes for Methods 8080, 8080A, 8081, and 608 are provided on the following page. The LAS Default TAL is the same for 8080, 8080A, 8081, and 608. Extended or Reduced TALs are provided on a sample/project specific basis.
- 2.3 Required detection limits for this procedure are compound dependent and vary with extraction efficiency and concentration. The applicable concentration range for this procedure is compound and matrix dependent. Method Detection Limits (MDLs) and Practical Quantitation Limits (PQLs) or Reporting Detection Limits (RDLs) are determined in accordance with LAS policy. MDL/PQL Tables are published by the LAS Quality Assurance Department (QAD). At LAS the terms PQL and RDL are essentially used interchangeably.
- 2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing response and retention times. Each identified component is quantified by relating the response produced by the compound in the sample to the response produced by an external standard.

## Organochlorine Pesticide/PCB Target Analyte Lists (TALs)

Default Single Component Pesticide Analytes	Standard Mix
alpha-BHC	Mix A
beta-BHC	Mix B
gamma-BHC	Mix A
delta-BHC	Mix B
Heptachlor	Mix A
Aldrin	Mix B
Heptachlor Epoxide	Mix B
gamma-Chlordane	Mix B
Endosulfan I	Mix A
alpha-Chlordane	Mix B
p,p'-DDE	Mix B
p,p'-DDT	Mix A
Dieldrin	Mix A
Endrin	Mix A
Endosulfan II	Mix B
p,p'-DDD	Mix A
Endrin Aldehyde	Mix B
Endosulfan Sulfate	Mix B
Methoxychlor	Mix A

### Default Multicomponent Pesticide Analytes

Toxaphene  
Chlordane (Technical)

### Default Aroclor Analytes

PCB 1016  
PCB 1221  
PCB 1232  
PCB 1242  
PCB 1248  
PCB 1254  
PCB 1260

### Additional F039/APP IX/UTS Analytes

Isodrin  
Kepone

### Additional F039/UTS Analytes

o,p-DDD  
o,p-DDE  
o,p-DDT

### 3.0 SUMMARY OF METHOD

For methylene chloride-immiscible liquid samples (waters, wastewaters, other aqueous liquids), a measured volume of sample is solvent extracted with methylene chloride by separatory funnel or continuous liquid-liquid extractor according to LAS-93-SOP-257, Extraction of Organochlorine Pesticides and PCBs for Methods 8080/8080A/8081/608. For methylene chloride-miscible liquid samples (oils, other organic liquids), a measured mass (viscous samples) or volume (low viscosity samples) of sample is diluted with methylene chloride according to LAS-93-SOP-257. For samples subsampled by mass, it is necessary to determine the density if reporting in liquid units. For methylene chloride/acetone-insoluble solid samples (soils, sludges, other inorganic substances), a measured mass of sample is extracted with methylene chloride/acetone by sonication or Soxhlet extraction according to LAS-93-SOP-257. For methylene chloride/acetone-soluble solid samples (organic sludges, other organic solids), a measured mass of sample is dissolved in methylene chloride or hexane according to LAS-93-SOP-257. Extracts may be subjected to Gel Permeation Chromatography (GPC), florisil, and/or acid cleanup as necessary. All extracts not in hexane are solvent exchanged to hexane during final concentration.

Extracts are analyzed on a gas chromatograph equipped with two dissimilar capillary columns and dual Electron Capture Detectors (ECD) to determine the amount of each separated compound. Single component pesticides and surrogates are identified in the samples by analyzing standards under the same conditions used for the samples, establishing the specific elution order, comparing the retention times, and confirming by the second chromatographic column or, rarely, by GC/MS. Multicomponent target analytes are identified by comparing the pattern and retention times of their respective standards to the samples. Quantitative results are determined using external standards to establish response factors for each target compound and surrogate during the initial calibration by integration of peak area. Each identified target compound and surrogate is quantified by comparing the response for the target compound in the sample to the external standard, while taking into account the sample volume or weight and any sample dilutions or concentrations. Multicomponent quantitations are performed by selecting representative peaks (usually five), quantitating the peaks as above, and then averaging the results to obtain a value for the multicomponent compound.

## 4.0 SAFETY

- 4.1 Personnel will adhere to the policies in the LAS Environmental Safety and Health Operations Manual. Personnel who have been trained as per this SOP have read the Material Safety Data Sheets (MSDS) for all materials used and are familiar with the contents and hazards listed on the MSDS.
- 4.2 The minimum level of hand protection when handling samples or hazardous chemicals is Nitrile™ gloves.
- 4.3 Care should be taken when performing repair or routine maintenance on the instrument. The injector ports, oven, and detectors are kept at elevated temperatures. When working with these parts of the instrument, gloves or hot pads should be used.
- 4.4 Careful note should be taken of high voltage when performing maintenance or repair procedures. Instrument manuals should be closely followed to minimize risk of electric shock.
- 4.5 Spent solvent will be collected in 10 liter poly carboys. Vials of extracts will be collected in 10 liter poly pails. These waste containers will have a red "Hazardous Waste" label. The waste code 001 will be written on the waste container.

## 5.0 SAMPLE STORAGE

- 5.1 All samples must be iced or refrigerated at  $4 \pm 2$  °C from the time of receipt until the time of extraction.
- 5.2 All sample extracts should be stored in a Teflon-lined screw-cap vial at a temperature of  $4 \pm 2$  °C and kept isolated from standards.
- 5.3 Prior to analysis, the sample extract should be brought to ambient temperature.
- 5.4 The extracts must be analyzed within 40 days of the beginning of extraction.
- 5.5 After analysis, sample extracts and sample splits will be stored for 60 days.

## 6.0 INTERFERENCES

- 6.1 Analysis of method and instrument blanks provide information about the presence of contaminants. When interfering peaks are noted in blanks, the analyst should identify and eliminate the source of contamination as subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks results in what the laboratory feels is a false positive for a sample, this should be fully explained in Analyst Notes accompanying the data.
- 6.2 Interfering contamination may occur when a sample of low concentration is analyzed immediately after a sample of high concentration. The preventative technique involves rinsing of the associated syringes with hexane and methanol. Successive instrument blanks are to be run until traces of carryover have been eliminated. In conjunction with this technique, cleaning of the injection port, baking the analytical column, and baking the detectors at high temperature is also strongly suggested. In extreme situations, the whole chromatographic system may require dismantling and cleaning. Screening of extracts suspected to contain high levels of target analytes or interferences at dilutions can be employed. If there is a question as to whether a sample needs to be diluted, it is always better to dilute first and eliminate the risk of instrument downtime than to analyze a high level sample and risk cross contamination of a successive sample.
- 6.3 Endrin and DDT degradation has been a problem with systems which are not kept clean. Regular changing of the injection liner and/or maintenance of the chromatographic system is imperative.
- 6.4 Phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large late-eluting peaks in the chromatogram. They are used as plasticizers and, therefore, plastic tubing, etc. must be avoided in the laboratory.
- 6.5 Hydrocarbons and other matrix interferences can make identification and quantitation of multicomponent analytes difficult. Acid cleanup of extracts can eliminate these interferences for PCB only determinations. The acid cleanup eliminate TAL pesticides and many interferences from the extract. In the case where only a PCB analysis is requested, the extraction procedure used will incorporate the acid cleanup (see LAS-93-SOP-257).

## 7.0 APPARATUS AND MATERIALS

- 7.1 Micro syringes: 10  $\mu\text{L}$ , 25  $\mu\text{L}$ , 50  $\mu\text{L}$ , 100  $\mu\text{L}$ , 250  $\mu\text{L}$ , 500  $\mu\text{L}$ , and 1000  $\mu\text{L}$ .
- 7.2 100 mL reagent bottles with screw top Teflon lined caps
- 7.3 2 mL glass crimp top vials, Teflon-lined crimp top caps, and crimper.
- 7.4 Disposable pasteur pipettes and bulbs.
- 7.5 GC System
- 7.5.1 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming capillary columns from 30°C to 350°C, have a flow controller which maintains a constant column flow rate or pressure throughout temperature program operation, be equipped with a dual Ni63 electron capture detectors, and possess an autosampler for injection of the sample. It is to be interfaced with a computer capable of acquiring and processing data using the Dionex AI-450 chromatography system. All GC carrier gas lines are to be of copper or stainless steel.
- 7.5.2 Analytical Columns
- 7.5.2.1 GC column 1 - 30 m x 0.53 mm ID RTX-1701 (Restek #12040) capillary column with 0.5  $\mu\text{m}$  film thickness.
- 7.5.2.2 GC column 2 - 30 m x 0.53 mm ID RTx-5 (Restek #10240) capillary column with 0.5  $\mu\text{m}$  film thickness.
- 7.5.3 Make-up Gas - To maximize ECD efficiency and analytical column efficiency, make-up gas must be used. The most efficient flow rates for maximum resolution by the analytical columns are between 5 and 10 mL/min. The most efficient flow rate for the ECD is approximately 50 mL/min. Make-up gas, introduced via a separate mass flow controller before the detector, should be set to a flow rate which allows maximum efficiency of both the analytical column and the ECD (40-60 mL/min).
- 7.5.4 Injection Port - For the Hewlett-Packard 5890 GC, an on-column injection port is recommended. A split/splitless injection port can be used, however,

this injector is prone to Endrin and DDT breakdown. Therefore, the special liner and gold washer developed by Hewlett Packard for use in pesticide analysis must be used. Injection port temperature must be 200°C.

- 7.5.5 Electron capture detector operated at 300 °C.
- 7.5.6 Data System - The data system is the Dionex AI-450 Chromatography system which runs under Windows. It must be capable of receiving input from the detector (ECD), allow integration of any peak, and allow continuous acquisition and storage on machine readable media of all response obtained throughout the duration of the chromatographic program.

### 8.0 REAGENTS AND STANDARDS

8.1 Hexane - Pesticide quality or equivalent and demonstrated to be free of analytes. Solvent purity is checked by the vendor. Data from these analyses are kept on file in Extraction Lab 124.

8.2 A Performance Evaluation Mix (PEM) must be prepared by diluting 100  $\mu$ L of Ultra's Performance Evaluation Mix (Catalog #CLP-250) to 100 mL in hexane.

Pesticide Stock Standard Solutions are certified solutions purchased from Ultra called Custom Pesticide Standard Mix A (Catalog #LOC-053 or equivalent) and Custom Pesticide Standard Mix B (Catalog #CUS1686 or equivalent). Prepare calibration standard solutions in hexane using the recipe in the summary below.

Standard	Mix A Conc. $\mu$ g/mL	Aliquot ( $\mu$ L)	Final Vol (mL)	Final Conc. $\mu$ g/mL
XLow A	50-100	10	100	0.005-0.01
Low A	50-100	40	100	0.02-0.04
Mid A	50-100	120	100	0.06-0.12
High A	50-100	160	100	0.08-0.16
XHigh A	50-100	320	100	0.16-0.32

Standard	Mix B Conc. $\mu$ g/mL	Aliquot ( $\mu$ L)	Final Vol (mL)	Final Conc. $\mu$ g/mL
XLow B	50-100	10	100	0.005-0.01
Low B	50-100	40	100	0.02-0.04
Mid B	50-100	120	100	0.06-0.12
High B	50-100	160	100	0.08-0.16
XHigh B	50-100	320	100	0.16-0.32

The TAL Table in Section 2 of this SOP indicates which standard mix each individual pesticide is in.

The multicomponent pesticide standards are prepared at a concentration which represents the reporting detection limit. These standards are used as single point screening mixtures in order to identify any multicomponent patterns which may be present in the samples. The multicomponent standards are prepared according to the following summary using Ultra standards or equivalent. Surrogate is added using Ultra Pesticide Surrogate Mix (Catalog # ISM-320) at 200  $\mu\text{g}/\text{mL}$  or equivalent such that both surrogates are at 0.02  $\mu\text{g}/\text{mL}$ .

Compound	Conc. ( $\mu\text{g}/\text{mL}$ )	Ultra Cat#	Aroclor Aliquot Vol ( $\mu\text{L}$ )	Surrogate Aliquot Vol ( $\mu\text{L}$ )	Final Vol. (mL)	Aroclor Conc. $\mu\text{g}/\text{mL}$
Aroclor 1016/1260	1000	LOC-030	10	10	100	0.1
Aroclor 1221	100	PP-291	100	5	50	0.2
Aroclor 1232	100	PP-301	50	5	50	0.1
Aroclor 1242	100	PP-311	50	5	50	0.1
Aroclor 1248	100	PP-341	50	5	50	0.1
Aroclor 1254	100	PP-351	50	5	50	0.1
Aroclor 1260	100	PP-361	50	5	50	0.1
Toxaphene	100	PP-271	250	5	50	0.5
Technical Chlordane	100	PP-151	50	5	50	0.1

NOTE: Because of the toxicity of some of the compounds, primary dilutions of these materials should be prepared in fume hoods.

- 8.2.1 Fill the appropriate volumetric flask with hexane leaving enough room for the addition of the standard materials. Allow the flask to stand, unstoppered, until all solvent wetted surfaces have dried.
- 8.2.2 Add the stock material using a hexane rinsed syringe of appropriate size for the amount being added. The liquid is introduced just below the surface of the solvent without contacting the neck of the flask. After depressing the plunger, the syringe is immediately withdrawn. The syringe is again thoroughly rinsed with hexane before the next aliquot of standard is added.
- 8.2.3 Continue this process until all analytes and surrogates have been added. Invert the flask several times to distribute the analytes in the solvent.

Calculate the concentration in micrograms per milliliter. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

- 8.2.4 Transfer the standard solution to a Teflon-sealed screw-cap bottle, store at  $4 \pm 2$  °C, and protect from light. Standards are not to be stored in the same refrigerator as samples which are awaiting analysis.
- 8.2.5 Prepare fresh calibration standards every six months or sooner if comparison check standards indicate a problem. Standards must be monitored closely by comparison to the initial calibration curve and by comparison to GC check standards. It may be necessary to replace the standards more frequently if either check exceeds a 25% difference.
- 8.2.6 Single component and multi-component calibration standards are prepared at concentrations which allow the solutions to be used for CLP 3/90 analyses in addition to the 8080/8080A/8081/608 analyses.

**NOTE:** Documentation for the receipt and preparation of all standards must follow LAL-90-SOP-0005, Standards Traceability in the Organic and Inorganic Sections.

- 8.3 Traceability - Each standard prepared is to be recorded in a separate GC Standards Preparation Logbook with the following information recorded for each entry:

LAS Standard ID Number of the Prepared Standard  
Standard Name/Description of the Prepared Standard  
Expiration Date of the Prepared Standard  
LAS Standard ID Number(s) of the Parent Standard(s)  
Compound/Element/Description(s) of the Parent Standard(s)  
Lot Number(s) of the Parent Standard(s)  
Supplier(s) of the Parent Standard(s)  
Concentration(s) of the Parent Standard(s)  
Aliquot(s) of the Parent Standard(s) an be included.  
Final Volume of the Prepared Standard  
Final Concentration(s) of the Prepared Standard  
Solvent and Solvent Lot Number of the Prepared Standard  
Preparation Date  
Signature of the Preparer

- 8.4 **Labelling Prepared Standards** - Each standard prepared at LAS is to have a label permanently attached to the bottle which contains the following information:

LAS Standard ID Number  
Standard Name  
Concentration (if feasible)  
Solvent/Matrix/Acid  
Expiration Date  
Preparation Date  
Initials of the Preparer

- 8.5 **Standards Expiration** - Ampulated standards received from vendors expire on the manufacturer's expiration date. If no manufacturer's expiration date is provided, an expiration date two years from the date of manufacture will be assigned. Standards prepared at LAS from these parent materials will be assigned an expiration date six months from the date of preparation or the earliest expiration date of its parent standards, whichever is shorter.

## 9.0 PROCEDURE

### 9.1 Instrument operating conditions.

#### 9.1.1 Gas Chromatograph

The following are the recommended GC analytical conditions for the capillary columns in 7.1.4.2:

Carrier Gas:	Hydrogen
Flow Rate:	5-10 mL/min. (approx. 10 psi)
Initial Temperature:	145 °C
Initial Hold Time:	1 min.
Ramp Rate:	3.5 °C/min
Final Temperature:	236 °C
Final Hold Time:	3.5 min.
Ramp Rate A:	4 °C/min
Final Hold Time A:	250 °C
Ramp Rate B:	30 °C/min
Final Hold Time A:	270 °C

#### 9.1.2 ECD

The following are the conditions for the detector:

Detector Temperature:	300 °C
Make-up flow Rate: (P10)	40-90 mL/min Argon/10% Methane

### 9.2 Calibration

Prior to the injection of any standards or samples, the Performance Evaluation Mix must be injected in order to check the system for Endrin and DDT breakdown. The degradation compounds of Endrin (Endrin Aldehyde and Endrin Ketone) and DDT (DDE and DDD) are observed and areas recorded. The following formulas are used to calculate the breakdown percentages. For 8080A and 8081 only, the performance evaluation mix should be injected at the beginning of every 12 hour shift after the initial calibration to confirm that the system is operating within control.

The following equations should be used to calculate the percent breakdown for DDT and Endrin.

$$\text{DDT breakdown \%} = \frac{\text{DDE area} + \text{DDD area}}{\text{DDT area} + \text{DDE area} + \text{DDD area}} \times 100\%$$

$$\text{End breakdown \%} = \frac{\text{End Ald area} + \text{End Ket area}}{\text{End area} + \text{End Ald area} + \text{End Ket area}} \times 100\%$$

When performing 8080 or 608, analysis should not proceed if the breakdown exceeds 20% for either Endrin or DDT or 30% combined. When performing 8080A or 8081, analysis should not proceed if breakdown exceeds 15% for either Endrin or DDT. See Section 10 for corrective action.

- 9.2.1 The standards required for the calibration are pipetted directly out of the reagent bottles in which the standards are stored using a fresh Pasteur pipette for each standard. They are placed into 2 mL vial crimp top vials and labelled. The vials are then allowed to warm to ambient temperature before analysis. Hexane instrument blank vials should also be prepared at this time.
- 9.2.2 A calibration curve is created using the following procedure. Inject each calibration standard using the same analytical conditions which will be used for the analysis of samples. To eliminate questions of carryover from one standard concentration to the next, analyze in order from the lowest concentration to the highest. After analysis, verify correct integration of all the target analyte peaks and plot the area against the concentration of the compound injected using the Dionex software. The coefficient of determination ( $r^2$ ) is used to check the linearity of the curve and must be 0.99 or greater. The preferred plotting method is quadratic forced through zero. Second order equations most accurately describe ECD responses in most cases. When required by specific projects/clients, linear (first order) equations are used. In special case, higher order equations or equations not forced through zero can be used. Consult the GC Technical Lead or Organic Section Supervisor if special equations are warranted. See Section 10 for corrective action.

When performing methods 8080, 8080A or 8081, a five point calibration is required for all compounds of interest. When performing method 808, a minimum of three calibration points is required. It is not practical to inject all multiresponse components for each calibration. Therefore, a five point calibration is performed for all pesticides and each multicomponent compound is injected at one concentration for screening purposes. If a multicomponent compound is identified in a sample, a five point calibration for that compound is injected and the sample reanalyzed.

- 9.2.3 Initial calibrations must be performed whenever substantial changes have been made to the GC system (i.e., detector cleaning, flow rate change, column removal or replacement) or if continuing calibration criteria have not been met. A typical calibration sequence appears below.

Instrument Blank  
Performance Evaluation Mix  
Aroclor 1221  
Aroclor 1232  
Aroclor 1242  
Aroclor 1248  
Aroclor 1254  
Aroclor 1016\1260 (A five point can be analyzed for this Aroclor)  
Toxaphene  
Technical Chlordane  
Mix A XLOW  
Mix B XLOW  
Mix A LOW  
Mix B LOW  
Mix A MID  
Mix B MID  
Mix A HIGH  
Mix B HIGH  
Mix A XHIGH  
Mix B XHIGH  
Instrument Blank

- 9.2.4 Continuing Calibration

- 9.2.4.1 The calibration curve for each compound must be verified at the beginning and end of every ten samples (excluding instrument blanks, matrix spikes, and duplicate spikes) or prior to sample analysis if the sequence was

interrupted. This is done by the analysis of one or more continuing calibration standards usually at the mid-range concentration. A continuing calibration is valid if the percent differences for the calibrated compounds are less than 15%. See Section 10 for corrective action if the percent difference is greater than 15%. The following equation is used to calculate percent difference:

$$\% \text{ Difference} = \frac{R_i - R_c}{R_i} \times 100\%$$

where,

$R_i$  = True concentration of the analyzed standard  
 $R_c$  = Measured concentration of the continuing calibration

- 9.2.4.2 When performing 8080 or 608, a group of samples are valid if the continuing calibration preceding them meets criteria. When performing 8080A or 8081, a group of samples is valid if both bracketing continuings are valid. See Section 10 for corrective action procedures.
- 9.2.5 For those clients requesting that an initial calibration verification be analyzed, a quality control check standard from a different lot of standard shall be prepared and analyzed after the initial calibration.
- 9.2.6 Retention time windows are set in the software by using a retention time window centered on the highest calibration standard's retention time. The width of this window is typically 0.1-0.2 minutes which biases the quantitation report toward false positives. The judgement of the analyst must weigh heavily in the interpretation of the chromatograms and, the retention time window is only one piece of information used to identify peaks. See Section 10 for corrective action.
- 9.3 Sample Analysis
- 9.3.1 Sample extracts may only be analyzed after the GC system has met either the initial calibration criteria or the criteria for continuing calibration as outlined above. The extracted samples are pipetted into crimp top vials and allowed to warm to ambient temperature before analysis. The same conditions must be used for the analysis of samples as were used for calibration.

- 9.3.2 Samples are analyzed in a set referred to as an analytical batch. Unless immediately following an initial calibration, the sequence begins with an instrument blank followed by a continuing calibration (mid-range) standard. If there is contamination in the blank where an analyte is present at or above the method detection limit (MDL), another blank is to be run. If contamination is consistent, every effort is to be made to eliminate the source of contamination before continuing the run. If the continuing calibration standard meets the 15.0% difference criteria for all compounds, method blanks (extraction blanks) are then analyzed followed by the samples and dilutions thereof. If a sample is suspected to be of high concentration, then a dilution should be analyzed prior to analyzing the undiluted sample. If the sample is a designated matrix spike, no dilution is required for the matrix spike and duplicate, but the unspiked sample should be diluted to within the calibration range of the instrument. Whenever possible, matrix spikes and duplicates are to be run consecutively with the sample in the same analysis sequence.
- 9.3.3 The analytical batch can include no more than ten samples (excluding instrument blanks, continuing calibration standards, matrix spikes, duplicate matrix spikes, LCSs, and dilutions). At the end of the sequence, another continuing calibration standard must be analyzed to conclude the batch. When performing 8080A or 8081, continuing calibration standards and performance evaluation standards must be analyzed at least every 12 hours.
- 9.3.7 For automated runs, complete loading of all of the samples to be analyzed. A typical sequence of standards, samples, and QC appears below.

**608/8080 Sequence**

Instrument Blank  
CCV  
MB  
LCS  
Ten samples  
Matrix Spike  
Matrix Spike Dup.  
Instrument Blank  
CCV  
CCV

**8080A/8081 Sequence**

Instrument Blank  
CCV  
PEM  
MB  
LCS  
Ten Samples  
Matrix Spike  
Matrix Spike Dup.  
Instrument Blank  
CCV (every 10 samples or 12 hours)  
CCV (every 10 samples or 12 hours)  
PEM (every 12 hours)

## 9.4 Data Interpretation

### 9.4.1 Qualitative Analysis

9.4.1.2 Single component analytes are identified by comparison of retention time and peak shape of the sample with that of a standard at similar concentration. Elution order is established for each particular analytical column by analyzing each compound separately. Tentative identification of an analyte occurs when a peak from a sample extract falls within an established retention time window. Retention time windows in the software are weighted toward false positives and are normally set from 0.1-0.2 minutes of the retention time, however, they are narrowed if the windows would overlap or widened, if necessary. This method of identification forces the analyst to carefully consider all positives and have a reason for reporting or not reporting the result. The experience of the analyst must weigh heavily in the interpretation of chromatograms.

9.4.1.3 Multicomponent analytes are identified by comparing the peak pattern and the retention time range of the multicomponent standards with peak pattern of the sample. Peak height ratios, peak shapes, and general similarity in detector response to the standard and sample are considered when identifying a multicomponent analyte. Single point standards for AR1221, AR1232, AR1242, AR1248, AR1254, AR1016, AR1260, Toxaphene, and Technical Chlordane are analyzed in every initial calibration sequence to provide the analyst a standard for comparison. If a multicomponent pesticide is detected, a five point calibration is generated and the sample is re-analyzed. Samples which appear to contain Aroclors should be examined carefully when attempting to make an identification. Peak weathering and mixtures of multicomponent compounds may distort the peak pattern and relative peak ratios.

9.4.1.4 The multicomponent compound technical chlordane is a mixture of many different components including alpha-chlordane, gamma-chlordane, heptachlor, and heptachlor epoxide. These components are part of the single component analyte list. Depending upon the sample history, any number of components may be present. In cases where the technical chlordane pattern can be identified, quantitation of technical chlordane should be done. Additional chlordane components will be identified as single component pesticides. In this situation, both technical chlordane and the components present should be reported.

9.4.1.5 Confirmation of target analytes is done during analysis on a secondary GC column. If necessary it can be done by GC/MS (if concentration levels permit). The GC/MS operating conditions and procedures for analysis are those specified in Method 8270. Confirmation may not be necessary if the composition of the sample is well established by prior analysis.

#### 9.4.2 Quantitative Analysis

9.4.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated area of the compound specific peak(s). The quantitation of a given analyte is based on the calibration curve established by the initial calibration curve - not the continuing calibration.

9.4.2.2 The software calculates the concentration of each analyte in the extract by plotting the area of the peak against the calibration curve established by the initial calibration. This extract concentration must then be related to the sample concentration by using the following equations:

#### Water Samples

$$C_s \text{ in } \mu\text{g/L} = \frac{\text{Extract Volume in milliliters} \times C_x}{\text{Sample Volume in liters}}$$

#### Soil Samples

$$C_s \text{ in } \mu\text{g/kg} = \frac{\text{Extract Volume in milliliters} \times C_x}{\text{Sample Weight in kg} \times \text{percent solids as decimal}}$$

where,  $C_s$  is the sample concentration  
 $C_x$  is the extract concentration in  $\mu\text{g/mL}$

9.4.2.3 Multicomponent analytes are quantitated by choosing five prominent congeners, quantitating each against the five point calibration and then averaging the concentration. Specific peaks can be discarded if poor quantitation is obtained, however, three peaks should be considered the minimum required for quantitation.

9.5 If the on-column concentration of any target analyte exceeds the initial calibration range, that sample must be diluted. Utilize the results of the

original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range. The dilution factor chosen should keep the response of the largest peak in the upper half of the initial calibration range of the instrument for that compound.

- 9.5.1 Identified target compounds will be reported if they are determined to have a concentration greater than the laboratory reporting detection limit (RDL). Positively identified target compounds with concentrations less than the RDL but greater than the method detection limit will be reported and flagged with a "J" qualifier when requested by a client.
- 9.5.2 Do not dilute matrix spike/duplicate matrix spike (MS/MSD) samples solely to get spiked analytes within the calibration range. The spiking level of each compound in the matrix spike solution should not require the dilution of the MS/MSD samples.

## 10.0 QC REQUIREMENTS AND CORRECTIVE ACTIONS

### 10.0 INSTRUMENT BLANK

#### 10.1 QC Requirement

The instrument blank must contain less than or equal to the method detection limit (MDL) for that analyte.

#### 10.1.2 Corrective Action

If the instrument blank contains target analytes higher than the MDL, the data must be judged for validity and corrective action initiated which may include the following:

Correct any integration problems encountered by the software.

Proceed with the analysis and flag the samples with a "B" qualifier if the analyte is detected.

Reanalyze the instrument blank.

Perform instrument maintenance and reanalyze the instrument blank.

#### 10.1.3 Documentation

Documentation must include quantitation report(s) and chromatogram(s). Any instrument maintenance must be documented in the maintenance log.

### 10.2 INITIAL CALIBRATION

#### 10.2.1 QC Requirement

A Pesticide Performance Evaluation Mix must be injected prior to the initial calibration. When using 8080 or 608, breakdown must be less than 20% for Endrin and DDT individually or 30% combined. When using method 8080A or 8081, breakdown cannot exceed 15% for either Endrin or DDT.

10.2.2 Corrective Action

If the PEM does not meet the criteria, the data must be judged for validity and corrective action initiated which may include the following:

Check for errors.

Correct any integration problems encountered by the software.

Proceed with the analysis and reanalyze any samples which contain analytes which did not meet the criteria.

Proceed with the analysis and document in the case narrative samples which were analyzed under an initial calibration which did not meet criteria.

Reanalyze some or all of the initial calibration standards.

Prepare new calibration standards and recalibrate the instrument.

Perform instrument maintenance and reanalyze the initial calibration standards and samples, if applicable.

10.2.3 QC Requirement

The calibration equations for each analyte must meet the 0.99  $r^2$  calibration criteria and the points must visually correlate, or the client specific criteria as applicable.

10.2.4 Corrective Action

If a compound does not meet the criteria, the data must be judged for validity and corrective action initiated which may include the following:

Check for errors.

Correct any integration problems encountered by the software.

Proceed with the analysis and reanalyze any samples which contain analytes which did not meet the criteria.

Proceed with the analysis and document in the case narrative samples which were analyzed under an initial calibration which did not meet criteria.

Reanalyze some or all of the initial calibration standards.

Prepare new calibration standards and recalibrate the instrument.

Perform instrument maintenance and reanalyze the initial calibration standards.

#### 10.2.5 Documentation

Documentation must include quantitation report(s) and chromatogram(s), calibration plot(s), and method printout(s). Any instrument maintenance must be documented in the maintenance log.

#### 10.6 QUALITY CONTROL CHECK STANDARD (QCCS)

##### 10.6.1 QC Requirement

In order to check the validity of the initial calibration, an independent standard is analyzed when requested by a client. The QCCS is made from a standard produced by a different manufacturer or, if a second manufacturer is unavailable, the QCCS is made by a different individual. The QCCS should meet the same criteria as a continuing calibration standard or the client specific criteria as applicable.

##### 10.6.2 Corrective Action

If the QCCS criteria cannot be met for any one analyte, the data must be judged for validity and corrective action initiated which may include the following:

Reanalyze the same QCCS solution.

Correct any integration problems encountered by the software.

Prepare and analyze a new QCCS solution.

Perform instrument maintenance and reanalyze the QCCS. If maintenance

is performed which changes the analytical conditions, a new initial calibration must be analyzed and the QCCS reanalyzed.

Proceed with the analysis and reanalyze any samples which contain analytes which did not meet the criteria.

Proceed with the analysis and document in the case narrative samples which were analyzed under an initial calibration which did not meet criteria.

10.6.3 Documentation

Documentation must include quantitation report(s) and chromatogram(s). Any instrument maintenance must be documented in the maintenance log.

10.7 METHOD BLANK

10.7.1 QC Requirement

The method blank must contain less than or equal to the method detection limit (MDL) for that analyte.

10.7.2 Corrective Action

If the method blank contains target analytes higher than the MDL, the data must be judged for validity and corrective action initiated which may include the following:

Correct any integration problems encountered by the software.

Proceed with the analysis and flag the samples with a "B" qualifier if the analyte is detected.

Reextract and reanalyze all the samples in the QC batch

Reanalyze the method blank.

Perform instrument maintenance and reanalyze the method blank and/or samples.

10.7.3 Documentation

Documentation must include quantitation report(s) and chromatogram(s). Any instrument maintenance must be documented in the maintenance log.

## 10.8 LABORATORY CONTROL SAMPLES (LCS\LABORATORY FORTIFIED BLANK)

### 10.8.1 QC Requirement

The recovery limits for each matrix spike compound are outlined in the method, specified by the client, or have been generated in house. Calculate the percent recovery using the following equation:

$$\% R = \frac{\text{Concentration(or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\%$$

### 10.8.2 Corrective Action

If the recovery for any compound is not within the limits outlined above, the data must be judged for validity and corrective action initiated which may include:

Report the analysis, flag the out of criteria data, and note in the case narrative.

Reanalyze the samples and QC and report the best (or both) analysis, flag the out of criteria data, and note in the narrative.

Reextract and reanalyze the samples and QC.

### 10.8.3 Documentation

Documentation must include quantitation report(s) and chromatogram(s). Recoveries are documented on the LCS summary form.

## 10.9 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE (MS/MSD) ANALYSES

### 10.9.1 QC Requirement

The recovery limits for each matrix spike compound are outlined in the method, specified by the client, or have been generated in house.

Calculate the percent recovery using the following equation:

$$\% R = \frac{\text{Concentration(or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\%$$

The duplicate spike data provides an estimate of analytical precision for each spiked analyte. The precision estimate is calculated as Relative Percent Difference (RPD) using the following equation:

$$RPD = \frac{|(MS \%R - MSD \%R)|}{(1/2)(MS \%R + MSD \%R)} \times 100\%$$

#### 10.9.2 Corrective Action

If the recovery or the relative percent difference for any compound is not within the limits outlined above, the data must be judged for validity and corrective action initiated which may include:

Report the analysis, flag the out of criteria data, and note in the case narrative.

Reanalyze the sample, MS, and MSD, report the best analysis, flag the out of criteria data, and note in the narrative.

Reextract and reanalyze the sample, MS, and MSD.

#### 10.9.3 Documentation

Documentation must include quantitation report(s) and chromatogram(s). Recoveries and RPD's are documented on the MS/MSD summary form.

### 10.10 CONTINUING CALIBRATION

#### 10.10.1 QC Requirement

The retention time for any analyte in the continuing calibration standard should not fall outside the window established during either the initial calibration or a previous continuing calibration standard.

#### 10.10.2 Corrective Action

If the retention time for any analyte in a continuing calibration falls outside the retention time windows, the data must be judged for validity and corrective action initiated which may include:

Observation of the retention times of the surrogates/spikes in order to properly identify target analytes present in the samples.

Updating of the retention time window width/midpoint to the last continuing calibration standard.

Reanalysis of the samples.

Reanalysis of the continuing calibration standard.

Perform instrument maintenance and reanalyze the samples. If maintenance is performed which changes the analytical conditions, a new initial calibration must be analyzed before the samples are reanalyzed.

Proceed with the analysis, report the data, and note any deviations in the case narrative.

#### 10.10.3 QC Requirement

The concentration of each analyte determined during analysis should not vary by more than 15% from the theoretical concentration. When using 8080 or 608, samples are valid when the continuings analyzed prior meet the 15% difference criteria. When using 8080A or 8081, samples are only valid when both bracketing continuings meet the 15% difference criteria.

#### 10.10.4 Corrective Action

##### 8080 or 608

If the concentration for any analyte in a continuing calibration falls outside the 15% difference, the data must be judged for validity and corrective action initiated which may include:

Reanalysis of the samples.

Reanalysis of the continuing calibration standard.

Perform instrument maintenance and reanalyze the samples. If maintenance is performed which changes the analytical conditions, a new initial calibration must be analyzed before the samples are reanalyzed.

Proceed with the analysis, report the data, and note any deviations in the case narrative.

#### **8080A or 8081**

If the concentration for any analyte in an ending continuing calibration falls outside the 15% difference, the associated samples must be re-analyzed.

Reanalysis of the continuing calibration standard.

Perform instrument maintenance and reanalyze the samples. If maintenance is performed which changes the analytical conditions, a new initial calibration must be analyzed before the samples are reanalyzed.

If upon re-analysis of the samples, the end continuing fails again, the best analysis of the samples will be reported.

### 10.11 DOCUMENTATION

The documentation of retention times and windows must include the method printout for the initial calibration. Any adjustment of the retention time width/midpoint is noted on the raw data.

Document all inspection and corrective actions, if any, in the injection or maintenance logbook.

The documentation must include reports and chromatograms for each calibration and associated samples, blanks, matrix spike, and matrix spike duplicate.

Document all inspection and corrective actions, if any, in the injection or maintenance logbook and provide a copy in the data package to be submitted to the document control.

10.12 SURROGATE SPIKE

10.12.1 QC Requirement

The recovery limits for each surrogate spike compound are outlined in the method, specified by the client, or have been generated in house. Calculate the percent recovery using the following equation:

$$\% R = \frac{\text{Concentration(or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\%$$

10.12.2 Corrective Action

If the recovery for any compound is not within the limits outlined above, the data must be judged for validity and corrective action initiated which may include:

Check the recoveries in the LCS and MB. If the recoveries meet criteria, report the analysis, flag the out of criteria data, and note in the case narrative as attributable to matrix, if necessary.

Reanalyze the sample(s) and report the best analysis or both analyses, flag the out of criteria data, and note in the narrative, if necessary.

Reextract and reanalyze the sample.

10.12.3 Documentation

Documentation must include quantitation report(s) and chromatogram(s).

10.13 DILUTION OF SAMPLES, MATRIX SPIKES, AND MATRIX SPIKE DUPLICATES

10.13.1 QC Requirement

Target analytes are to be quantitated within the calibration curve.

### 10.13.2 Corrective Action

If the on-column concentration of any compound in any sample exceeds the upper limit of the initial calibration, the data must be judged for validity and corrective action initiated which may include:

Dilute and reanalyze the sample.

Report the analysis and flag the analyte concentration with an "E" qualifier.

### 10.13.3 Documentation

All dilutions must be noted on the raw data and the reporting forms.

## 10.14 ADDITIONAL DOCUMENTATION REQUIREMENTS

### Sample Analysis

Documentation for sample analysis must include data system printouts, chromatograms, surrogate spike recoveries, and matrix spike recoveries. All data pertinent to this analysis must be recorded in the appropriate logbooks.

The reference name of analytical batches of data on forms and in the data archives has been standardized in order to facilitate retrieval of the data and to match LCS's, Blank's, and MS/MSD's with the appropriate samples. The convention followed is: date of calibration-analysis number-instrument letter-sequential number, ie. 041994-801020-N-1. The sequential number increments after each continuing calibration. The initial calibration is placed in a separate folder ie., 051494-801020-N-init. ie. 051494-801020-N-1. For sequences which last more than the 99 sample limit imposed by the Dionex software, change the calibration date to the date of the new schedule and begin the sequential number at 1. A typical naming sequence follows:

**051994-801020-N-1**

Instrument Blank  
2  $\mu\text{g/L}$  Calibration standard  
5  $\mu\text{g/L}$  Calibration standard  
10  $\mu\text{g/L}$  Calibration standard  
15  $\mu\text{g/L}$  Calibration standard  
20  $\mu\text{g/L}$  Calibration standard  
30  $\mu\text{g/L}$  Calibration standard  
Instrument Blank  
LCS  
Ten samples  
Matrix spike  
Matrix spike duplicate

---

**051991-801020-N-2**

CCV (two may be placed in the autosampler for unattended operation)  
Instrument Blank  
LCS  
Ten samples  
Matrix spike  
Matrix spike duplicate  
CCV (two may be placed in the autosampler for unattended operation)

---

**051994-801020-N-3**

CCV (two may be placed in the autosampler for unattended operation)  
Instrument Blank  
LCS  
Ten samples  
Matrix spike  
Matrix spike duplicate  
CCV

## 11.0 REFERENCES

- 11.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Third Edition, Method 8080, "Organochlorine Pesticides and PCBs", September 1986.
- 11.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Third Edition, Method 8000, "Gas Chromatography", September 1986.
- 11.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Final Update II, Method 8080A, "Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography", September 1994.
- 11.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Final Update II, Method 8081, "Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique", September 1994.
- 11.5 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Final Update I, Method 8000A, "Gas Chromatography", July 1992.
- 11.6 Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, USEPA 600 Series 600/4-84-061, 40CFR Pt. 136 App. A, Method 608, "Organochlorine Pesticides and PCBs", June 1984.

# APPENDIX E

## Reference Laboratory Analytical Procedures for PCB Oil Analyses

United Power Services Inc.  
Nashville, TN



## Test Method

# The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils

Thomas A. Bellar and James J. Lichtenberg

### 1. Scope

1.1 This is the EPA preferred method for the determination of polychlorinated biphenyls (PCBs) in waste oils according to PCB regulations.<sup>1</sup> This gas chromatographic (GC) procedure is applicable to the determination of commercial mixtures of PCBs in transformer fluids and certain other hydrocarbon-based waste oils. The method can be used to analyze waste oils for individual PCB isomers or complex mixtures of chlorinated biphenyls from monochlorobiphenyl through decachlorobiphenyl only if the isomers have been previously identified by other methods<sup>2</sup> or by knowledge of the sample history.

1.2 The detection limits are dependent upon the complexity of the sample matrix and the ability of the analyst to properly maintain the analytical system. Using a carefully optimized instrument, this method has been shown to be useful for the determination of commercial PCB mixtures spiked into transformer fluid over a range of 5.0 to 500 mg/kg. Based upon a statistical calculation at 5 mg/kg for a simple oil matrix, the method detection limit for Aroclors 1221, 1242, 1254, and 1260 is 1 mg/kg. The method detection limit (MDL) is defined as the

minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Prior to sample analysis, each analyst must demonstrate the ability to generate acceptable results with this method by following the procedures described in Section 10.2.

### 2. Summary

2.1 The sample is diluted on a weight/volume basis so that the concentration of each PCB isomer is within capability of the GC system (0.01 to 10 ng/ $\mu$ L).

2.2 The diluted sample is then injected into a gas chromatograph for separation of the PCB isomers. Measurement is accomplished with a halogen-specific detector which maximizes baseline stability and minimizes interferences normally encountered with other detectors. The electron capture detector (ECD) can normally be substituted for the halogen-specific detector when samples contain dichloro through decachlorobiphenyl isomers (Aroclors 1016, 1232, 1242, 1248, 1254, 1260,

1262 and 1268) or when the sample matrix does not interfere with the PCBs. Several cleanup techniques are provided for samples containing interferences. A mass spectrometer operating in the selected ion monitoring mode of data acquisition may also be used as the GC detector when PCB levels are sufficiently high and the PCB m/z ranges are free from interference. Interferences may occur in some waste oil samples even after exhaustive cleanup.

**2.3** The concentration of the PCBs are calculated on a mg/kg basis, using commercial mixtures of PCBs as standards. The analysis time, not including data reduction, is approximately 35 min/sample.

### 3. Interferences

**3.1** Qualitative misidentifications are always a potential problem in GC analysis. The use of a halogen-specific detector and the analyst's skill in recognizing chromatographic patterns of commercial PCB mixtures minimizes this possibility.

**3.2** Whenever analyzed samples do not provide chromatographic patterns nearly identical to the standards prepared from commercial PCBs, the analyst must confirm the presence of PCBs by one of three ways: by analysis after column cleanup; by analysis on dissimilar GC columns; or, by gas chromatography/mass spectrometry (GC/MS).

**3.3** During the development and testing of this method, certain analytical parameters and equipment designs were found to affect the validity of the analytical results. Proper use of the method requires that such parameters or designs are to be used as specified. These items are identified in the text by the word "must." Anyone wishing to deviate from the method in areas so identified must demonstrate that the deviation does not affect the validity of the data and alternative test procedure approval must be obtained through the USEPA, Environmental Monitoring and Support Laboratory, Equivalency Program, 26 W. St. Clair Street, Cincinnati, Ohio 45268.<sup>3</sup> An experienced analyst may make modifications to parameters or equipment identified by the term "recommended." Each time such modifications are made to the method, the analyst must repeat the procedure in Section 10.2. In this case, formal approval is not required, but the

documented data from Section 10.2 must be on file as part of the overall quality assurance program

**3.4** Samples which are diluted at a ratio of 100:1 and are analyzed by electron capture GC, consistently produce results that are 10 to 20% lower than the true value (See Section 12). This is due to quenching of the detector response by high boiling hydrocarbons coeluting with the PCBs. The degree of error is matrix dependent and is not predictable for samples of unknown origin. As the PCB concentration approaches 20% of a control level, for example: 50 mg/kg, the analyst must routinely reanalyze a duplicate spiked sample to determine the actual recovery. The duplicate or diluted sample is spiked at two times the electron capture observed value and reanalyzed according to Section 10.2. The results are corrected accordingly.

### 4. Apparatus

**4.1** Gas Chromatograph—The gas chromatograph should be equipped with on-column 1/4-inch injectors. The oven must be large enough to accept a 1/4" OD 2-meter coiled glass column. If halogen-specific detectors are used, then the column oven should have programming capabilities.

#### 4.2 Gas Chromatographic Detector

**4.2.1** A halogen-specific detector is used to eliminate interferences causing misidentifications or false-positive values due to non-organohalides which commonly coelute with the PCBs.

**4.2.1.1** Electrolytic conductivity detector — the Hall electrolytic conductivity detector, Model 700-A (HED), available from Tracor, Inc., has been found to provide the sensitivity and stability needed for the current PCB Regulations.<sup>1</sup>

**4.2.1.2** Other halogen-specific detectors, including older model electrolytic conductivity detectors and microcoulometric titration, may be used. However, the stability, sensitivity, and response time of these detectors may raise the MDL and adversely affect peak resolution. Each system must be shown to be operating within requirements of the PCB regulations by collecting single laboratory accuracy and precision data and MDL on simple spiked samples, as described in Section 10.2.

**4.2.2** Semi-specific detectors, such as ECD, may be substituted when sample chromatographic patterns closely match those of the standards. Acid cleanup (See Section 8.1) or Florisil slurry cleanup (See Section 8.7) should be incorporated routinely when the ECD is used. See Section 3.4 for additional quality control procedures for ECD.

**4.2.3** Quantitative GC. MS techniques can be used. The recommended approach is selected ion monitoring, but the GC/MS data system must have a program that supports this method of data acquisition. The program must be capable of monitoring a minimum of eight ions, and it is desirable for the system to have the ability to change the ions monitored as a function of time. For PCB measurements, several sets of ions may be used, depending on the objectives of the study and the data system capabilities. The alternatives are as follows:

**4.2.3.1** Single ions for high sensitivity: 154, 188, 222, 256, 292, 326, 360, 394

**4.2.3.2** Short mass ranges which may give enhanced sensitivity, depending on the data system capabilities: 154-156, 188-192, 222-226, 256-260, 290-295, 322-328, 356-364, 390-398.

**4.2.3.3** Single ions giving decreased sensitivity but are selective for levels of chlorination: 190, 224, 260, 294, 330, 362, 394.

**4.2.3.4** The data system must have the capability of integrating an abundance of the selected ions between specified limits and relating integrated abundances to concentrations, using the calibration procedures described in this method.

#### 4.3 Gas Chromatographic Columns

**4.3.1** The GC columns and conditions listed below are recommended for the analysis of PCB mixtures in oil. If these columns and conditions are not adequate, the analyst may vary the column parameters to improve separations. The columns and conditions selected must be capable of adequately resolving the PCBs in the various Aroclor mixtures so that each Aroclor is identifiable through isomer pattern recognition. (See Figures 1 through 6 to establish this.) To properly use the calculation procedure described in Section 11.5, the analyst must use the methyl silicone liquid phase column,

described in Section 4.3.2. Capillary columns and their associated specialized injection techniques are acceptable alternatives; however, due to problems associated with the use of capillary columns the analyst must demonstrate that the entire system will produce acceptable results by performing the operations described in Section 10.2.

**4.3.2 Recommended primary analytical column:** Glass, 1/4-inch O.D. (2-mm I.D.), 6-ft. (180 cm) long, packed with Gas-Chrom Q 100/120 mesh coated with 3% OV-1.

**Carrier gas:** 40 to 60 mL/min (helium, nitrogen or mixtures of methane in argon, as recommended by the manufacturer of the detector).

**Temperature Program:** 120°C isothermal for 2 minutes, 6°/min to 220°C and hold until all compounds elute. Figure 7 shows a chromatogram of the PCB locator mixture (See Section 5.8) analyzed under these conditions. Each PCB peak has been identified by assigning the same relative retention times determined in the isothermal runs (Figures 1 through 6).

**Isothermal Operation:** Aroclor 1221, 1232, or Cl<sub>1</sub> through Cl<sub>4</sub> isomers — recommended range 140 to 150°C  
Aroclor 1016, 1242, 1248, 1254, 1260, 1262, 1268, or Cl<sub>3</sub> through Cl<sub>10</sub> isomers — recommended range 170 to 200°C

**4.3.3 Recommended confirmatory column:** Glass tubing, 1/4-inch O.D. (2-mm I.D.), 6-ft. (180 cm) long, packed with Gas-Chrom Q 100/120 mesh coated with 1.5% OV-17 + 1.95% OV-210.

**Carrier gas:** 40 to 60 mL/min (helium, nitrogen or mixtures of methane in argon, as recommended by the manufacturer of the detector).

**Column temperatures:** Aroclor 1221, 1232, or Cl<sub>1</sub> through Cl<sub>4</sub> isomers recommended range — 170 to 180°C.  
Aroclor 1016, 1242, 1248, 1254, 1260, 1268, or Cl<sub>3</sub> through Cl<sub>10</sub> isomers 200°C.

**4.4 Volumetric flasks** — 10, 100, 200, and 250-mL.

**4.5 Pipets** — 0.10, 1.0, and 5.0 mL Mohr delivery (for viscous oils cut off tip of pipet).

**4.6 Micro syringes** — 10.0 μL

**4.7 Sample containers** — 20 mL or larger screw-cap bottles with Teflon-faced cap liners. (Aluminum foil cap liners can be used for non-corrosive samples.)

**4.8 Chromatographic column** — Chromaflex, 400-mm long x 19-mm I.D. (Kontes K-420540-9011 or equivalent).

**4.9 Gel Permeation Chromatograph** — GPC Autoprep 1002 or equivalent, available from Analytical Bio Chemistry Laboratories, Inc.

**4.10 Balance** — Analytical, capable of weighing 99 g with a sensitivity of ± 0.0001 g.

**4.11 Kuderna-Danish (K-D) Evaporative Concentrator Apparatus**

**4.11.1 Concentrator tube** — 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of solvent.

**4.11.2 Evaporative flask** — 500 mL (Kontes K-57001-0500 or equivalent). Attach to concentrator tube with springs (Kontes K-662750-0012 or equivalent).

**4.11.3 Snyder column** — Three-ball macro (Kontes K503000-0121 or equivalent).

## 5. Reagents and Materials

### 5.1 Reagent safety precautions

**5.1.1** The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration regulations regarding the safe handling of the chemical specified in this method. A reference file of material data-handling sheets should also be made available to all personnel involved in the chemical analysis.

**5.1.2** PCBs have been tentatively classified as known or suspected, human or mammalian carcinogens. Primary standards of these toxic compounds should be prepared in a hood.

**5.1.3** Diethyl ether should be monitored regularly to determine the peroxide content. Under no circumstances should diethyl ether be used with a peroxide content in excess of 50 ppm as an explosion could result. Peroxide test strips manufactured by EM Laboratories (available from Scientific Products Co., Cat. No. P1126-8 and other suppliers) are

recommended for this test. Procedures for removal of peroxides from diethyl ether are included in the instructions supplied with the peroxide test kit.

**5.2** Hexane (mixed hexanes), isooctane, acetonitrile, methylene chloride, cyclohexane, and diethyl ether of pesticide grade.

### 5.3 Recommended Column Packings

**5.3.1** Gas Chrom Q 100, 120 mesh coated with 3% OV-1.

**5.3.2** Gas Chrom Q 100/120 mesh coated with 1.5% OV-17 + 1.95% OV-210.

### 5.4 Standards

**5.4.1** Aroclors 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, 1268. Primary dilutions of various Aroclors are available from USEPA, Environmental Monitoring and Support Laboratory, Quality Assurance Branch, 26 W. St. Clair Street, Cincinnati, Ohio 45268.

**5.4.2** 2-Chlorobiphenyl, 3-chlorobiphenyl, and decachlorobiphenyl.

**5.4.3** Pure, individual PCBs, as identified in the sample by mass spectrometry or indicated by retention data.

**5.4.4** Alumina (Fisher A540 or equivalent).

**5.4.5** Silica gel (Davison Grade 950 or equivalent)

**5.4.6** Florisil (PR grade or equivalent).

**5.4.7** Sulfuric acid A.C.S.

**5.4.8** Quality Control Check Sample — Certified Samples of PCBs in oil matrices are available from USEPA, Environmental Monitoring and Support Laboratory, Quality Assurance Branch, 26 W. St. Clair Street, Cincinnati, Ohio 45268.

**5.5 Standard Stock Solutions** — Prepare primary dilutions of each of the Aroclors or individual PCBs by weighing approximately 0.01 g of material within ± 0.0001 g. Dissolve and dilute to 10.0 mL with isooctane or hexane. Calculate the concentration in μg/μL. Store the primary dilutions at 4°C in 10- to 15-mL narrow-mouth, screw-cap bottles with Teflon cap liners. Primary dilutions are stable indefinitely if the seals are maintained. The validity of inhouse-generated or stored primary and secondary dilutions must be verified on a quarterly basis by analyzing Environmental Monitoring and Support Laboratory-Cincinnati-Quality

Control Check Samples or certified PCB standards.

**5.6 Working Standards** — Prepare working standards similar in PCB composition and concentration to the samples by mixing and diluting the individual standard stock solutions. Dilute the mixture to volume with pesticide quality hexane. Calculate the concentration in  $\text{ng}/\mu\text{L}$  as the individual Aroclors (Section 11.4) or as the individual PCBs (Section 11.5). Store dilutions at  $4^\circ\text{C}$  in 10- to 15-mL narrow-mouth, screw-cap bottles with Teflon cap liners. If the seals are maintained, these secondary dilutions can be stored indefinitely. (See Section 5.5.)

**5.7 Laboratory control standard (LCS)** — Prepare a LCS by spiking a PCB-free oil typical of the matrix normally analyzed, such as a transformer oil, at  $50.0 \text{ mg}/\text{kg}$  with a PCB mixture typical of those normally found in the samples, such as Aroclor 1260 at  $50.0 \text{ mg}/\text{kg}$ .

**5.8 PCB Locator Mixture** — Prepare a PCB locator mixture containing  $0.1 \text{ ng}/\mu\text{L}$  of 2-chlorobiphenyl,  $0.1 \text{ ng}/\mu\text{L}$  3-chlorobiphenyl,  $0.5 \text{ ng}/\mu\text{L}$  Aroclor 1242,  $0.5 \text{ ng}/\mu\text{L}$  Aroclor 1260, and  $0.2 \text{ ng}/\mu\text{L}$  Aroclor 1268 in hexane ( $0.1 \text{ ng}/\mu\text{L}$  of decachlorobiphenyl can be substituted for Aroclor 1268). Use the chromatogram generated by the PCB locator mixture to help identify the retention times of the various PCB isomers commonly found in commercial PCB mixtures.

## 6. Sample Collection and Handling

**6.1 Sample containers** should have a volume of 20 mL or more and have Teflon or foil-lined screw caps.

### 6.2 Sample Bottle Preparation

**6.2.1** Wash all sample bottles and seals in detergent solution. Rinse first with tap water and then with distilled water. Allow the bottles and seals to drain dry in a contaminant-free area. Then rinse seals with pesticide-grade hexane and allow to air dry.

**6.2.2** Heat sample bottles to  $400^\circ\text{C}$  for 15 to 20 minutes or rinse with pesticide-grade acetone or hexane and allow to air dry.

**6.2.3** Store the clean bottles inverted or sealed until use.

**6.2.4** Sample bottles can be reused. Prior to reuse, rinse the bottles and seals three times with hexane, allow to air dry, and then proceed to Section 6.2.1.

**6.3 Sample Preservation** — The samples should be stored in a cool, dry, dark area until analysis. Storage times in excess of four weeks are not recommended for unknown or undefined sample matrices.

### 6.4 Sample Collection

**6.4.1** Fill a large container, such as a 500-mL beaker, from a representative area of the sample source. If practical, mix the sample source prior to sampling.

**6.4.2** Fill a minimum of two 20-mL sample bottles (Field Sample 1 (FS1) and Field Sample 2 (FS2)) approximately 80% full from the sampling container.

**6.4.3** Repeat Sections 6.4.1 and 6.4.2 if there is a need to monitor sampling precision, as described in Section 10.6.

## 7. Procedure

**7.1** The approximate PCB concentration of the sample may be determined by X-ray fluorescence (total halogen measurement), microcoulometry (total halogen measurement), density measurements, or by analyzing a very dilute mixture of the sample (10,000:1) according to Section 7.4.

**7.2** For samples in the 0- to  $100\text{-mg}/\text{kg}$  range, dilute at the rate of 100:1 in hexane.

**7.2.1** Pipet 1.0 mL of sample into a 100-mL volumetric flask, using a 1.0-mL Mohr pipet. For viscous samples, cut the capillary tip off the pipet. Dilute to volume with hexane. Stopper and mix.

**7.2.2** Using the same pipet as in Section 7.2.1, deliver 1.0 mL of sample into a tared 10-mL beaker weighed to  $\pm .001 \text{ g}$ . Reweigh the beaker to  $\pm .001 \text{ g}$  to determine the weight of sample used in 7.2.1.

**7.2.3** As an alternative to Sections 7.2.1 and 7.2.2, weigh approximately 1 g to  $\pm .001 \text{ g}$  of sample in a 100-mL volumetric flask and dilute to volume with hexane.

**7.2.4** Analyze the diluted sample according to Section 7.4 or store the diluted sample in a narrow-mouth bottle with a Teflon-lined screw cap.

**7.3** For samples above  $100 \text{ mg}/\text{kg}$  in concentration, dilute at a rate of 1000:1 in hexane.

**7.3.1** Pipet 0.10 mL of sample into a 100-mL volumetric flask, using a 0.10 mL-Mohr pipet. Dilute to volume with hexane, stopper and mix.

**7.3.2** Using the same pipet as in Section 7.3.1, deliver 0.10 mL of sample into a tared 10-mL beaker to  $\pm .0001 \text{ g}$ . Reweigh the beaker to determine the weight of sample used in Section 7.3.1.

**7.3.3** As an alternative to Sections 7.3.1 and 7.3.2, weigh approximately 0.1 g to  $\pm .0001 \text{ g}$  of sample and in a 100 mL volumetric flask. Dilute to volume with hexane.

**7.3.4** Analyze the diluted sample according to Section 7.4 or store in a narrow-mouth bottle with a Teflon-lined screw cap.

**7.3.5** If the concentration of PCBs is still too high for the chromatographic system, prepare secondary dilutions from Sections 7.3.1 or 7.3.3 until acceptable levels are obtained.

**7.4** Analyze the sample by injecting the hexane mixture into the gas chromatograph, using auto injectors or the solvent flush technique.<sup>4</sup>

**7.4.1** Recommended injection volumes: Halogen-specific detector — 4 to  $5 \mu\text{L}$ , ECD 2 to  $3 \mu\text{L}$ . Smaller volumes may be injected when auto injectors are used if the resulting MDL are acceptable.

*Note:* When semi-specific detectors are used, cleanup techniques (See Section 4.2.2) should be routinely incorporated into the analysis scheme prior to injection.

**7.5** If the resulting chromatogram shows evidence of column flooding or nonlinear detector responses, further dilute the sample according to Section 7.3.5.

**7.6** Determine whether or not PCBs are present in the sample by comparing the sample chromatogram to that of the PCB locator mixture, Section 5.8.

**7.6.1** If a series of peaks in the sample match some of the retention times of PCBs in the PCB locator mixture, attempt to identify the source by comparing chromatograms of each standard prepared from commercial mixtures of PCBs (See Section 5.6). Proceed to Section 11.4 if the source of PCBs is identified.

**7.6.2** If the sample contains a complex mixture of PCBs, proceed to Section 11.5.

**7.6.3** If a dilution ratio of 1000:1 (Section 7.3) or higher was analyzed and no measurable PCB peaks were detected, analyze an aliquot of sample diluted to 100:1.

7.6.4 If several PCB interference problems are encountered or if PCB ratios do not match standards, proceed to Section 8. Use alternate columns or use GC-MS<sup>2</sup> to verify whether or not the nonrepresentative patterns are due to PCBs.

## 8. Cleanup

Several tested cleanup techniques are described. Depending upon the complexity of the sample, one or all of the techniques may be required to resolve the PCBs from interferences.

### 8.1 Acid Cleanup

**8.1.1** Place 5.0 mL of concentrated sulfuric acid into a 40-mL narrow-mouth screw-cap bottle. Add 10.0 mL of the diluted sample. Seal the bottle with a Teflon-lined screw-cap and shake for one minute.

**8.1.2** Allow the phases to separate, transfer the sample (upper phase) to a clean narrow-mouth screw-cap bottle. Seal with a Teflon-lined cap.

**8.1.3** Analyze according to Section 7.4.

**8.1.4** If the sample is highly contaminated, a second or third acid cleanup may be employed.

*Note:* This cleanup technique was tested over a 6-month period, using both electron capture and electrolytic conductivity detectors. Care was taken to exclude any samples that formed an emulsion with the acid. The sample was withdrawn well above the sample-acid interface. Under these conditions, no adverse effects associated with column performance and detector sensitivity to PCBs were noted. This cleanup technique could adversely affect the chromatographic column performance for samples containing analytes other than PCBs.

### 8.2 Florisil Column Cleanup

**8.2.1** Variances between batches of Florisil may affect the elution volume of the various PCBs. For this reason, the volume of solvent required to completely elute all of the PCBs must be verified by the analyst. The weight of Florisil can then be adjusted accordingly.

**8.2.2** Place a 20.0-g charge of Florisil, activated at 130°C, into a Chromaflex column. Settle the Florisil by tapping the column. Add about 1 cm of anhydrous sodium sulfate to the top of the Florisil.

Pre-elute the column with 70 to 80 mL of hexane. Just before the exposure of the sodium sulfate layer to air, stop the flow. Discard the eluate.

**8.2.3** Add 2.0 mL of the undiluted sample to the column with a 2-mL Mohr pipet. For viscous samples, cut the capillary tip off the pipet. Add 225 mL of hexane to the column. Carefully wash down the inner wall of the column with a small amount of the hexane prior to adding the total volume. Collect and discard the first 25.0 mL.

**8.2.4** Collect exactly 200 mL of hexane eluate in a 200-mL volumetric flask. All the PCBs must be in this fraction.

**8.2.5** Using the same pipet as in Section 8.2.2, deliver 2.0 mL of sample into a tared 10-mL beaker weighed to  $\pm 0.001$  g. Reweigh the beaker to determine the weight of the sample diluted to 200 mL.

**8.2.6** Analyze the sample according to Section 7.4.

### 8.3 Alumina Column Cleanup

**8.3.1** Adjust the activity of the alumina by heating to 200°C for 2 to 4 hours. When cool, add 3% water (weight:weight) and mix until uniform. Store in a tightly sealed bottle. Allow the alumina to equilibrate at least 30 minutes before use. Adjust activity weekly.

**8.3.2** Variances between batches of alumina may affect the elution volume of the various PCBs. For this reason, the volume of solvent required to completely elute all of the PCBs must be verified by the analyst. The weight of alumina can then be adjusted accordingly.

**8.3.3** Place a 50.0-g charge of alumina into a Chromaflex column. Settle the alumina by tapping. Add about 1 cm of anhydrous sodium sulfate to the top of the alumina. Pre-elute the column with 70 to 80 mL of hexane. Just before exposing the sodium sulfate layer to air, stop the flow. Discard the eluate.

**8.3.4** Add 2.5 mL of the undiluted sample to the column with a 5-mL Mohr pipet. For viscous samples, cut the capillary end off the pipet. Add 300 mL of hexane to the column. Carefully wash down the inner walls of the column with a small volume of hexane prior to adding the total volume. Collect and discard the 0- to 50-mL fraction.

**8.3.5** Collect exactly 250 mL of the hexane in a 250-mL volumetric flask. All the PCBs must be in this fraction.

**8.3.6** Using the same pipet as in Section 8.3.4, deliver 2.5 mL of sample into a tared 10-mL beaker ( $\pm 0.001$  g). Reweigh the beaker to determine weight of sample diluted to 250 mL.

**8.3.7** Analyze the sample according to Section 7.4.

### 8.4 Silica Gel Column Cleanup

**8.4.1** Activate silica gel at 135°C overnight.

**8.4.2** Variances between batches of silica gel may affect the elution volume of the various PCBs. For this reason, the volume of solvent required to completely elute all of the PCBs must be verified by the analyst. The weight of silica gel can then be adjusted accordingly.

**8.4.3** Place a 25-g charge of activated silica gel into a Chromaflex column. Settle the silica gel by tapping the column. Add about 1 cm of anhydrous sodium sulfate to the top of the silica gel.

**8.4.4** Pre-elute the column with about 70 to 80 mL of hexane. Just before exposing the sodium sulfate layer to air, stop the flow. Discard the eluate.

**8.4.5** Add 2.0 mL of the undiluted sample to the column with a 2-mL Mohr pipet. For viscous samples, cut the capillary tip off the pipet.

**8.4.6** Wash down the inner wall of the column with 5 mL of hexane.

**8.4.7** Elute the PCBs with 195 mL of 10% diethyl ether in hexane (volume:volume).

**8.4.8** Collect exactly 200 mL of the eluate in a 200-mL volumetric flask. All the PCBs must be in this fraction.

**8.4.9** Using the same pipet as in Section 8.4.5, deliver 2.0 mL of sample into a tared 10-mL beaker ( $\pm 0.001$  g). Reweigh to determine the weight of sample diluted to 200 mL.

**8.4.10** Analyze the sample according to Section 7.4.

### 8.5 Gel Permeation Cleanup

**8.5.1** Set up and calibrate the gel permeation chromatograph with an SX-3 column according to the instrument manufacturer's instruction manual. Use 15% methylene chloride in cyclohexane (volume:volume) as the mobile phase.

**8.5.2** Place 1.0 mL of sample into a 100-mL volumetric flask, using a 1-mL Mohr pipet. For viscous samples, cut the capillary tip off the pipet.

**8.5.3** Dilute the sample to volume, using 15% methylene chloride in cyclohexane (volume:volume).

**8.5.4** Using the same pipet as in Section 8.5.2, deliver 1.0 mL of sample into a tared 10-mL beaker ( $\pm 0.001$  g). Reweigh the beaker ( $\pm 0.001$  g) to determine the weight of sample used in Section 8.5.2.

**8.5.5** As an alternative to Sections 8.5.2 and 8.5.3, weigh approximately 1 g ( $\pm 0.001$  g) of sample and dilute to 100.0 mL in 15% methylene chloride in cyclohexane (volume:volume).

**8.5.6** Inject 5.0 mL of the diluted sample into the instrument. Collect the fraction containing the  $Cl_1$  through  $Cl_{10}$  PCBs (see instruction manual, Section 8.5.1) in a K-D flask equipped with a 10-mL ampul.

**8.5.7** Concentrate the Section 8.5.4 fraction down to less than 5 mL, using K-D evaporative concentration techniques.

**8.5.8** Dilute to 5.0 mL with hexane, then analyze according to Section 7.4. Be sure to use 100 mL as the dilution volume for the final calculation.

## **8.6 Acetonitrile Partition**

**8.6.1** Place 10.0 mL of the previously diluted sample into a 125-mL separatory funnel. Add 5.0 mL of hexane. Extract the sample four times by shaking vigorously for one minute with 30-mL portions of hexane-saturated acetonitrile.

**8.6.2** Transfer and combine the acetonitrile phases to a 1-L separatory funnel and add 650 mL of distilled water and 40 mL of saturated sodium chloride solution. Mix thoroughly for 30 to 35 seconds. Extract with two 100-mL portions of hexane by vigorously shaking about 15 seconds.

**8.6.3** Combine the hexane extracts in a 1-L separatory funnel and wash with two 100-mL portions of distilled water. Discard the water layer and pour the hexane layer through a column (Section 4.8) packed with 3 to 4 inches of anhydrous sodium sulfate. Drain the column into a 500-mL K-D flask equipped with a 10-mL ampul. Rinse the separatory funnel and column with three 10-mL portions of hexane.

**8.6.4** Concentrate the extracts to 6 to 10 mL in the K-D evaporator in a hot water bath, then adjust the volume to 10.0 mL. Be sure to use the correct dilution volume (See Section 8.6.1) for the final calculation.

**8.6.5** Analyze according to Section 7.4.

## **8.7 Florisil Slurry Cleanup**

**8.7.1** Place 10 mL of the diluted sample into a 20-mL narrow-mouth screw-cap container. Add 0.25 g of Florisil Seal with a Teflon-lined screw-cap and shake for one minute.

**8.7.2** Allow the Florisil to settle then decant the treated solution into a second container. Analyze according to Section 7.4.

## **9. Calibration**

**9.1 Single Point Calibrations** — Prepare calibration standards from standard stock solutions in hexane that are close to the unknown in composition and in concentration. If when using an electrolytic conductivity detector the sample response is in the low level nonlinear detection area, the calibration point must then be within 20% of the sample. The ECD must be operated only within its linear response range.

**9.2** As an alternative to Section 9.1, prepare a calibration curve for each Aroclor or PCB detected in the sample. The standard curve must contain at least three points, two of which must bracket the sample concentration. When using an electrolytic conductivity detector, if the sample response is in a low level nonlinear area of the calibration curve, two of the calibration points must be within 20% of the unknown. The calibration curve must be checked daily, using the LCS, Section 5.7. If the calibration curve is not within 15% of the LCS, recalibrate the instrument. If an ECD is used then it will be necessary to correct the LCS value for recovery (See Section 3.4). Use the recovery value determined the same day the calibration curve was generated. The correct value must be within 15% of the spike value, otherwise the instrument must be recalibrated.

## **10. Precision and Accuracy**

**10.1** Each laboratory using this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. *After January 1, 1983*, ongoing performance checks must be

compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

**10.1.1** Before performing any analyses the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established, as described in Section 10.2.

**10.1.2** In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 10.2.

**10.1.3** The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 10.4.

**10.2** To establish the ability to generate acceptable accuracy and precision in the use of this method, the analyst must perform the following operations.

**10.2.1** For each commercial PCB mixture or individual PCB isomer normally measured, prepare a PCB spiking concentrate, in isooctane within the range of 40 to 60 mg/mL.

**10.2.2** Using a microsyringe, add 100  $\mu$ L of the PCB concentrate to each of a minimum of four 100 g aliquots of PCB-free oil. A representative waste oil may be used in place of the clean oil, but one or more additional aliquots must be analyzed to determine the PCB background level, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 7.

**10.2.3** Calculate the average percent recovery, (R), and the relative standard deviation (s) of the concentration found. Waste oil background corrections must be made before R calculations are performed.

**10.2.4** Using the appropriate data from Tables 1, 2, and 3, determine the recovery and single operator precision expected for the method and compare these results to the values calculated in Section 10.2.3. If the data are not comparable, the analyst must review and remedy potential problem areas and repeat the test.

10.2.5 After January 1, 1983, the values for R and s must meet method performance criteria provided by the USEPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, before any samples may be analyzed.

10.3 The analyst must calculate method performance of the laboratory for each spike concentration and parameter being measured.

10.3.1 Calculate upper and lower control limits for method performance:

$$\begin{aligned}\text{Upper Control Limit (UCL)} &= R + 3s \\ \text{Lower Control Limit (LCL)} &= R - 3s\end{aligned}$$

where R and s are calculated as in Section 10.2.3. The UCL and LCL can be used to construct control charts<sup>5</sup> that are useful in observing trends in performance. After January 1, 1983, the control limits above must be replaced by method performance criteria provided by the USEPA.

10.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for waste oil samples. An accuracy statement for the method is defined as  $R \pm s$ . The accuracy statement should be developed by the analysis of 4 aliquots of waste oil, as described in Section 10.2.2, followed by the calculation of R and s. Alternately, the analyst may use four waste oil data points gathered through the requirement for continuing quality control in Section 10.4. The accuracy statements should be updated regularly.

10.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed, as described in Section 10.2.2, at two times the background level. If the recovery for a particular parameter does not fall within the control limits for method performance, the results reported for the parameter in all samples processed as part of the same set must be qualified, as described in Section 11.9. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

10.5 Before processing any samples, the analyst should demonstrate through the analysis of a PCB-free oil sample, that all glassware and reagents are free of

interferences. Each time a set of samples is analyzed or there is a change in reagents, a laboratory reagent blank should be processed as a safeguard against contamination.

10.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The most productive, specific practices depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists regarding the identification of a peak on the chromatogram, confirmatory techniques such as GC with a dissimilar column, specific element detector, or MS must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

10.7 Analyze the LCS, Section 5.7, daily before any samples are analyzed. Instrument status checks, calibration curve validation and long-term precision are obtained from these data. In addition, response data obtained from the LCS can be used to estimate the concentration of the unknowns. From this information, the appropriate standard dilutions can be determined for single-point calibrations.

10.8 Analyze on a quarterly basis a Quality Control Sample (Section 5.4.8.) of PCBs in oil or whenever new standard dilutions are prepared.

10.8.1 The results of the Quality Control Sample should agree within 15% of the true value. If they do not, the analyst must check each step in the standard preparation procedure to resolve the problem.

## 11. Calculations

11.1 Locate each PCB in the sample chromatogram by comparing the retention time of the suspect peak to the retention data gathered from analyzing standards and interference-free Quality Control Samples. The width of the retention time window used to make identifications should be based upon measurement of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for each PCB can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.2 If the response for any PCB peak exceeds the working range of the system, dilute according to Section 7.3.5.

11.3 If accurate measurement of the peaks in the PCB elution area of the chromatogram is prevented by the presence of interferences, further cleanup is required.

11.4 If the parent Aroclors or PCBs are identified in the sample, calibrate according to Section 9. The concentration of the PCBs in the sample is calculated by comparing the sum of the responses for each PCB in the standard to the sum of all of the PCBs in the sample. This is particularly important as sample concentrations approach within 20% of 50 mg/kg or any other EPA-regulated concentration. If calculations are based upon a single PCB peak or upon a small percentage of the total PCB peaks, serious errors may result. Peaks comprising less than 50% of the total can be disregarded only if (1) interference problems persist after cleanup, (2) the source of PCBs is obvious, or (3) the concentration of PCBs is not within  $\pm 20\%$  of an EPA-controlled value such as 50 mg/kg.

11.4.1 Measure the peak height or peak area of each peak identified as a PCB (Section 11.1) in both the sample and the standard.

11.4.2 Use the following formula to calculate the concentration of PCBs in the sample:

$$\text{Concentration mg/kg} = \frac{B \times V_i}{A \times W}$$

where:

$$A = \frac{\text{Sum of standard Peak Heights (areas)}}{\text{ng of standard injected}} = \text{mm/ng}$$

$$B = \frac{\text{Sum of sample Peak Heights (areas)}}{\mu\text{L injected}} = \text{mm}/\mu\text{L}$$

$V_i$  = dilution volume of sample in mL

W = weight of the sample in grams

11.5 If the parent Aroclors or source of PCBs is not apparent, calculate the concentration according to the procedure of Webb and McCall.<sup>6</sup> The concentration of the PCBs in each peak is determined individually then added together to determine the total PCB content of the sample. Each PCB identified in the

sample must be included in these calculations.

**11.5.1** Small variations between Aroclor batches make it necessary to obtain standards prepared from a specific source of Aroclors. Primary dilutions of these reference Aroclors will be available in 1981 from the USEPA, Environmental Monitoring and Support Laboratory, Quality Assurance Branch, Cincinnati, Ohio 45268.

**11.5.2** Analyze a standard mixture of Aroclors 1242, 1254, and 1260 under the conditions shown in Figures 3, 5, and 6. Analyze the sample under the same conditions. Compare the resulting standard chromatograms to those shown in Figures 3, 5, and 6. Each PCB peak must be resolved as well or better than those shown in the figures. Determine the relative retention time (RRT) of each peak in the standards with respect to p,p'-DDE or assign the RRT shown in the figures to the corresponding peak in the standard. Identify the RRT of each PCB in the sample by comparing the sample chromatogram to the standard chromatograms.

**11.5.3** Identify the most likely Aroclors present in the sample, using the Identification Flow Chart, Figure 8.

**11.5.4** Analyze standards according to Section 9, using the appropriate Aroclors.

**11.5.5** Determine the instrument response factor (A) for each individual PCB, using the following formula:

$$A = \frac{\text{Peak Height (area)}}{\text{Ng, x mean weight \%}} \times 100$$

where:

Ng = Ng of Aroclor standard injected (mean weight percent is obtained from Tables 4 through 9).

**11.5.6** Calculate the concentration of each PCB in the sample, using the following formula:

$$\text{Concentration mg/kg} = \frac{B \times V_1}{A \times W}$$

where:

A = Response factor from 11.5.5

$$B = \frac{\text{Peak Height (areas) of sample mm}/\mu\text{L}}{\mu\text{L injected}}$$

V<sub>1</sub> = dilution volume of sample in mL

W = weight of sample in grams

The concentration of each PCB must be calculated and added together to obtain the total amount of PCBs present.

**11.6** Report all data in mg/kg.

**11.7** Round off all data to two significant figures.

**11.8** Add all Aroclors and report what was used as the standard. For example, 57 mg/kg measured as Aroclor 1260 or 57 mg/kg measured as Aroclors 1242 and 1260.

**11.9** Data for the affected parameters of samples processed as part of a set where the laboratory spiked sample recovery falls outside the control limits in Section 10.4 must be labeled as suspect.

**11.10** Determine the actual recovery for electron capture analyses of each sample in the uncorrected 40- to 50-mg/kg concentration range (See Section 3.4). Report the corrected value and the recovery.

## 12. Precision and Accuracy

**12.1** The data shown in Tables 1 through 3 were generated using the recommended procedures described in this method to analyze both spiked and nonspiked oil samples of varying degrees of complexity. Data for both the HED and ECD were generated by the USEPA, Environmental Monitoring and Support Laboratory, Physical and Chemical Methods Branch, Cincinnati, Ohio 45268.

## References

1. Federal Register, 40 CFR, Part 761, July 1, 1981.
2. Eichelberger, J. W., L. E. Harris, and W. L. Budde. *Anal. Chem.*, **46**, 227 (1974).
3. Federal Register, 40 CFR, Sections 136.4 and 136.5, July 1, 1981.
4. White, L. D., et al., *AIHA Journal*, **31**, 22S, (1970).
5. Handbook of Analytical Quality Control in Water and Wastewater Laboratories. EPA-600/4-79-019

USEPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

6. Webb, R. G. and A. C. McCall. *J. Chrom. Sci.*, **11**, 366 (1973).

**Table 1.** Accuracy and precision using spiked motor oil<sup>1</sup>

Dilution Ratio	Detector	Method Cleanup	Spike mg/kg	Aroclor Spiked	Avg. Conc. Found mg/kg	(Precision) Rel. Std. Deviation %	(Accuracy) Percent Recovered	Number of Dilutions
100:1	HED	None	30.3	1242	28.2	4.2	93.1	5
100:1	ECD	None	30.3	1242	26.7 <sup>1</sup>	5.7	88.1	3
100:1	HED	None	31.1	1260	27.2	2.0	87.5	5
100:1	ECD	None	31.1	1260	23.9	2.2	76.8	3
100:1	HED	8.1	30.3	1242	28.4	11.5	93.7	3
"	ECD	8.1	30.3	1242	25.4 <sup>1</sup>	6.1	83.8	3
"	HED	8.1	31.1	1260	28.1	8.0	90.3	3
"	ECD	8.1	31.1	1260	24.3	7.8	78.1	3
"	HED	8.2	30.3	1242	30.7	2.4	101.	4
"	ECD	8.2	30.3	1242	27.3 <sup>1</sup>	10.2	90.1	4
"	HED	8.2	31.1	1260	30.9	3.6	99.4	4
"	ECD	8.2	31.1	1260	31.0	8.6	99.7	4
"	HED	8.3	30.3	1242	30.3	8.6	100.	3
"	ECD	8.3	30.3	1242	28.9 <sup>1</sup>	5.0	95.4	3
"	HED	8.3	31.1	1260	29.8	4.7	95.8	3
"	ECD	8.3	31.1	1260	30.8	6.5	99.0	3
"	HED	8.4	30.3	1242	29.4	5.8	97.0	3
"	ECD	8.4	30.3	1242	26.4 <sup>1</sup>	5.3	87.1	3
"	HED	8.4	31.1	1260	29.4	5.2	94.5	3
"	ECD	8.4	31.1	1260	23.6	4.5	105.	3
"	HED	8.5	30.3	1242	31.9	8.5	75.9	3
"	ECD	8.5	30.3	1242	23.4 <sup>1</sup>	3.0	77.2	2
"	HED	8.5	31.1	1260	33.6	9.2	108.	3
"	ECD	8.5	31.1	1260	30.9	5.5	99.4	3
"	HED	8.6	30.3	1242	34.4	3.8	107.	4
"	ECD	8.6	31.1	1242	23.4 <sup>1</sup>	4.4	77.2	4
"	HED	8.6	30.3	1260	29.1	4.2	96.7	4
"	ECD	8.6	31.1	1260	27.0	4.6	86.7	4

<sup>1</sup> Severe interference problems in elution area of 1242. Measurement based upon only 3 of the 10 normally resolved major peaks. Cleanup technique. Sections 8.1, 8.2, 8.3, 8.4, 8.5, and 8.6 did not improve the quality of the 1242 chromatogram. If this were an unknown sample, it would be impossible to qualitatively identify the presence of Aroclor 1242 using ECD. The HED provided an interference-free chromatogram.

**Table 2.** Accuracy and precision using waste transformer fluids

Sample	Dilution Ratio	Detector	Method Cleanup	1260 Spike mg/kg	Avg.(D) Conc. Found	(Precision) Rel. Std. Deviation %	(Accuracy) Percent Recovered	Number of Dilutions
A	100:1	ECD	None	--	22.6	3.6	--	7 <sup>2</sup>
A	"	HED	None	--	27.0	1.7	--	7 <sup>2</sup>
A	"	ECD	8.1	--	22.8	2.5	--	7 <sup>2</sup>
A	"	HED	8.1	--	29.7	1.4	--	7
A	"	ECD	8.2	--	22.4	1.0	--	3 <sup>2</sup>
A	"	HED	8.2	--	28.2	2.2	--	3 <sup>2</sup>
A	"	ECD	8.3	--	22.7	1.3	--	3 <sup>2</sup>
A	"	HED	8.3	--	27.8	2.8	--	3 <sup>2</sup>
A	"	ECD	8.4	--	20.9	--	--	1
A	"	HED	8.4	--	30.2	--	--	1
A	"	ECD	8.5	--	23.8	0.3	--	7 <sup>2</sup>
A	"	HED	8.5	--	28.6	4.1	--	7 <sup>2</sup>
A	"	ECD	None	27.0	45.0	3.3	91	7
A	"	HED	None	27.0	55.2	1.5	102	7 <sup>2</sup>
B	1000:1	ECD	None	--	452	0.8	--	7 <sup>2</sup>
B	"	HED	"	--	471	1.2	--	7
B	"	ECD	"	455	875	0.5	96	7 <sup>2</sup>
B	"	HED	"	455	916	2.0	99	7 <sup>2</sup>
C	1000:1	ECD	None	--	284	1.2	--	7
C	"	HED	"	--	300	1.4	--	7
C	"	ECD	"	300	607	3.6	104	7 <sup>2</sup>
C	"	HED	"	300	686	3.9	114	7

<sup>1</sup> A - dark waste oil  
 B - black waste oil with suspended solids  
 C - clear waste oil  
 D - all samples contained Aroclor 1260  
<sup>2</sup> Duplicate analyses made at each dilution

**Table 3.** Accuracy and precision and limit of detection data results of analyses of Shell transformer fluid spiked with PCBs at 5.0 and 27 mg/kg

Electron Capture Detector (100:1 dilution)						
Aroclor	Spike (mg/kg)	Number of Analyses	Avg. (mg/kg)	Standard Deviation	Percent Recovery	MDL <sup>1</sup> (mg/kg)
1221	5.0	7	7.5	0.43	150	1.4
1242	5.0	14	3.8	0.18	76	0.5
1254	5.0	7	4.1	0.08	82	0.2
1260	5.0	14	4.7	0.18	94	0.5

Electrolytic Conductivity Detector (100:1 dilution)						
Aroclor	Spike (mg/kg)	Number of Analyses	Avg. (mg/kg)	Standard Deviation	Percent Recovery	MDL <sup>1</sup> (mg/kg)
1221	5.0	6	7.5	0.23	150	0.7
1242	5.0	7	5.9	0.17	118	0.5
1254	5.0	6	5.8	0.16	116	0.5
1260	5.0	7	5.4	0.10	108	0.3

Shell Transformer Oil - 27 ppm Aroclor 1260 (100:1 dilution)					
Detector	Spike (mg/kg)	Number of Analyses	Avg. (mg/kg)	Rel. Std. Deviation, %	Percent Recovery
ECD	27	14	24.0	.70	89
HED	27	7	28.3	2.1	105

<sup>1</sup> MDL = Method Detection Limit at 99% confidence that the value is not zero.  
 Note: At these values it would be impossible to identify Aroclor patterns with any degree of confidence. 1 mg/kg appears to be a reasonable MDL.

$$MDL = t_{(n-1, .99)} (S)$$

where:  
 MDL = the method detection limit  
 $t_{(n-1, .99)}$  = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.  
 S = standard deviation of the replicate analyses

**Table 4.** Composition of Aroclor 1221

RRT <sup>2</sup>	Mean Weight Percent	Relative Std. Dev. <sup>3</sup>	Number of Chlorines <sup>4</sup>
11	31.8	15.8	1
14	19.3	9.1	1
16	10.1	9.7	2
19	2.8	9.7	2
21	20.8	9.3	2
28	5.4	13.9	2
32	1.4	30.1	2
37	1.7	48.8	3
40			3
Total		93.3	

<sup>1</sup> Data obtained from Webb and McCall.<sup>5</sup>  
<sup>2</sup> Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.  
<sup>3</sup> Relative standard deviation of 17 analyses (as percentages of the mean of the results).  
<sup>4</sup> From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

**Table 5.** Composition of Aroclor 1232<sup>1</sup>

RRT <sup>2</sup>	Mean Weight Percent	Relative Std. Dev. <sup>3</sup>	Number of Chlorines <sup>4</sup>
11	16.2	3.4	1
14	9.9	2.5	1
16	7.1	6.8	2
[20	17.8	2.4	2
21			2
28	9.6	3.4	2] 40%
			3] 60%
32	3.9	4.7	3
37	6.8	2.5	3
40	6.4	2.7	3
47	4.2	4.1	4
54	3.4	3.4	3] 33%
			4] 67%
58	2.6	3.7	4
70	4.6	3.1	4] 90%
			5] 10%
78	1.7	7.5	4
Total	94.2		

<sup>1</sup> Data obtained from Webb and McCall.<sup>6</sup>

<sup>2</sup> Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.

<sup>3</sup> Relative standard deviation of four analyses (as percentages of the mean of the results).

<sup>4</sup> From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

**Table 6.** Composition of Aroclor 1242<sup>1</sup>

RRT <sup>2</sup>	Mean Weight Percent	Relative Std. Dev. <sup>3</sup>	Number of Chlorines <sup>4</sup>
11	1.1	35.7	1
16	2.9	4.2	2
21	11.3	3.0	2
28	11.0	5.0	2] 25%
			3] 75%
32	6.1	4.7	3
37	11.5	5.7	3
40	11.1	6.2	3
47	8.8	4.3	4
54	6.8	2.9	3] 33%
			4] 67%
58	5.6	3.3	4
70	10.3	2.8	4] 90%
			5] 10%
78	3.6	4.2	4
84	2.7	9.7	5
98	1.5	9.4	5
104	2.3	16.4	5
125	1.6	20.4	5] 85%
			6] 15%
146	1.0	19.9	5] 75%
			6] 25%
Total	98.5		

<sup>1</sup> Data obtained from Webb and McCall.<sup>6</sup>

<sup>2</sup> Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

<sup>3</sup> Relative standard deviation of six analyses (as percentages of the mean of the results).

<sup>4</sup> From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

**Table 7.** Composition of Aroclor 1248<sup>1</sup>

RRT <sup>2</sup>	Mean Weight Percent	Relative Std. Dev. <sup>3</sup>	Number of Chlorines <sup>4</sup>
21	1.2	23.9	2
28	5.2	3.3	3
32	3.2	3.8	3
47	8.3	3.6	3
40	8.3	3.9	3 } 85%
			4 } 15%
47	15.6	1.1	4
54	9.7	6.0	3 } 10%
			4 } 90%
58	9.3	5.8	4
70	19.0	1.4	4 } 80%
			5 } 20%
78	6.6	2.7	4
84	4.9	2.6	5
98	3.2	3.2	5
104	3.3	3.6	4 } 10%
			5 } 90%
112	1.2	6.6	5
125	2.6	5.9	5 } 90%
			6 } 10%
146	1.5	10.0	5 } 85%
			6 } 15%
<b>Total</b>	<b>103.1</b>		

<sup>1</sup> Data obtained from Webb and McCall.<sup>6</sup>

<sup>2</sup> Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

<sup>3</sup> Relative standard deviation of six analyses (as percentages of the mean of the results).

<sup>4</sup> From GC/MS data. Peaks containing mixtures of isomers of different numbers are bracketed.

**Table 8.** Composition of Aroclor 1254<sup>1</sup>

RRT <sup>2</sup>	Mean Weight Percent	Relative Std. Dev. <sup>3</sup>	Number of Chlorines <sup>4</sup>
47	6.2	3.7	4
54	2.9	2.6	4
58	1.4	2.8	4
70	13.2	2.7	4 } 25%
			5 } 75%
84	17.3	1.9	5
98	7.5	5.3	5
104	13.6	3.8	5
125	15.0	2.4	5 } 70%
			6 } 30%
146	10.4	2.7	5 } 30%
			6 } 70%
160	1.3	8.4	6
174	8.4	5.5	6
160	1.3	8.4	6
174	8.4	5.5	6
203	1.8	18.6	6
232	1.0	26.1	7
<b>Total</b>	<b>100.0</b>		

<sup>1</sup> Data obtained from Webb and McCall.<sup>6</sup>

<sup>2</sup> Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

<sup>3</sup> Relative standard deviation of six analyses (as percentages of the mean of the results).

<sup>4</sup> From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

**Table 9.** Composition of Aroclor 1260<sup>a</sup>

RRT	Mean Weight Percent	Relative Std. Dev.	Number of Chlorines <sup>b</sup>
72	2.7	5.3	5
84	4.7	1.6	5
[98	3.8	3.5	5
104			5
117	3.3	6.7	6
125	12.3	3.3	5
146	14.1	3.6	6
160	4.9	2.2	6
174	12.4	2.7	6
203	9.3	4.0	6
[232	9.8	3.4	6
244			7
280	11.0	2.4	7
332	4.2	5.0	7
372	4.0	8.6	8
448	.6	25.3	8
528	1.5	10.2	8
<b>Total</b>	<b>98.6</b>		

<sup>a</sup> Data obtained from Webb and McCall.<sup>5</sup>

<sup>b</sup> Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

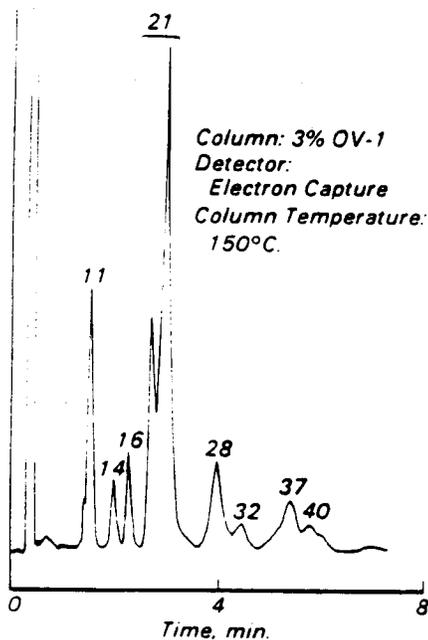
Overlapping peaks that are quantitated as one peak are bracketed.

<sup>3</sup> Relative standard deviation of six analyses (as percentages of the mean of the results).

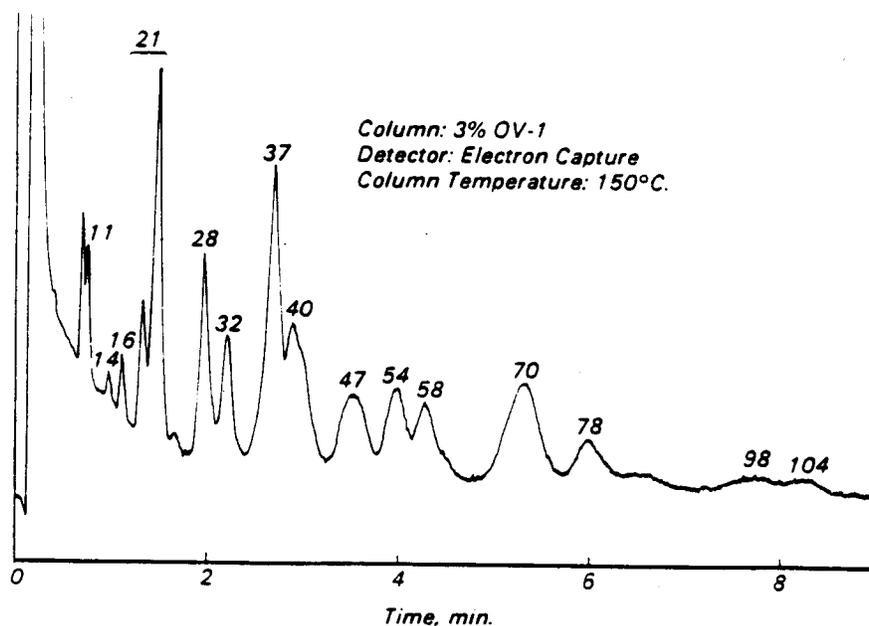
<sup>4</sup> From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

<sup>5</sup> Composition determined at the center of peak 104.

<sup>6</sup> Composition determined at the center of peak 232.



**Figure 1.** Gas chromatogram of Aroclor 1221.



**Figure 2.** Gas chromatogram of Aroclor 1232.

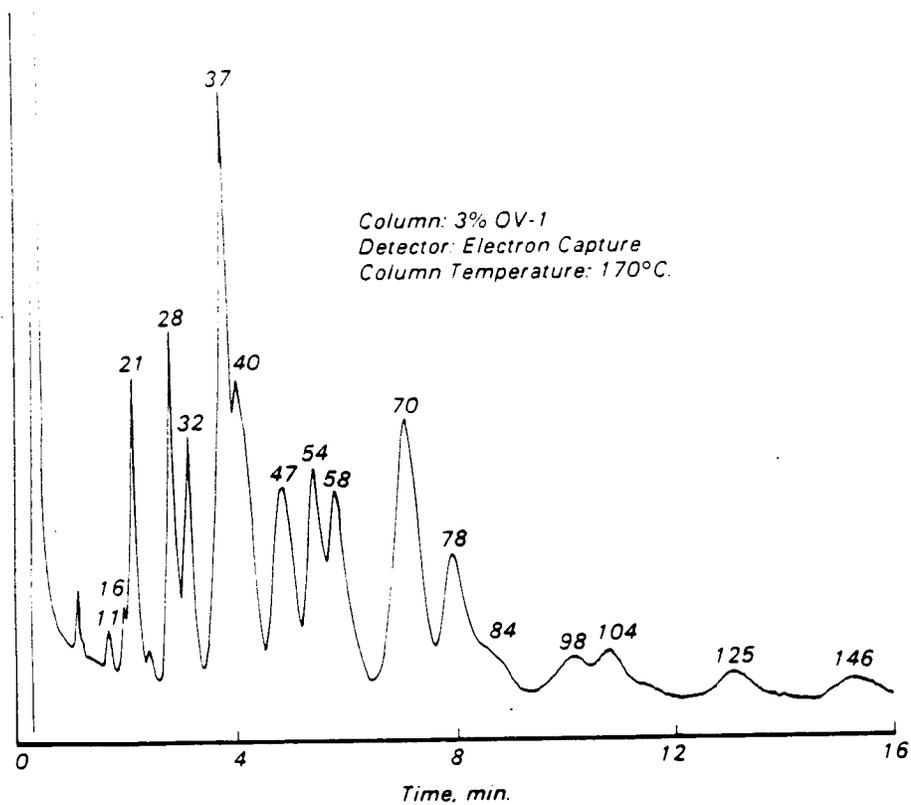


Figure 3. Gas chromatogram of Aroclor 1242.

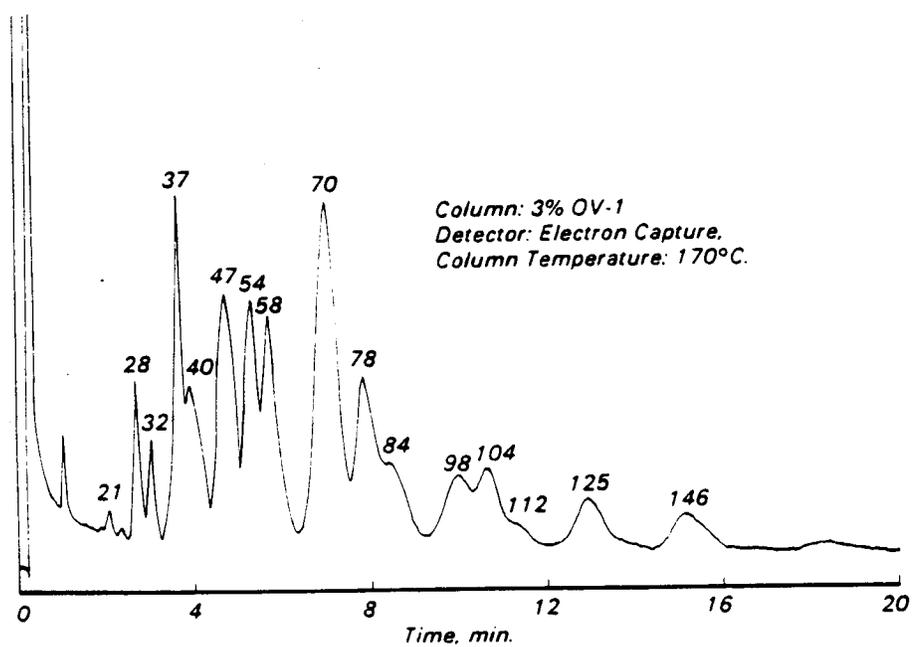


Figure 4. Gas chromatogram of Aroclor 1248.

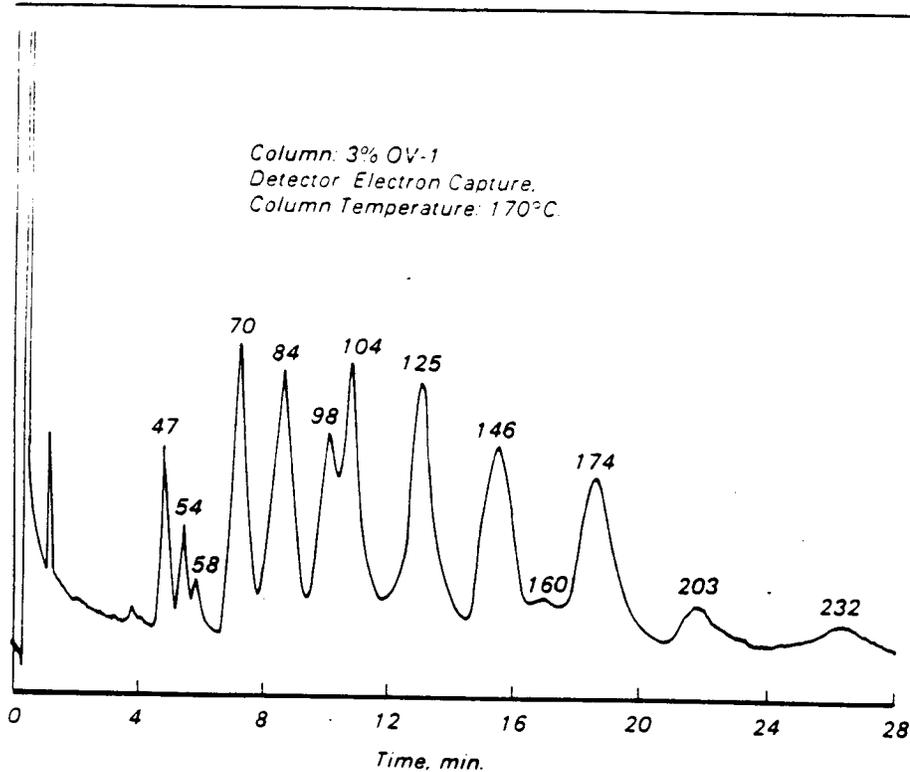


Figure 5. Gas chromatogram of Aroclor 1254.

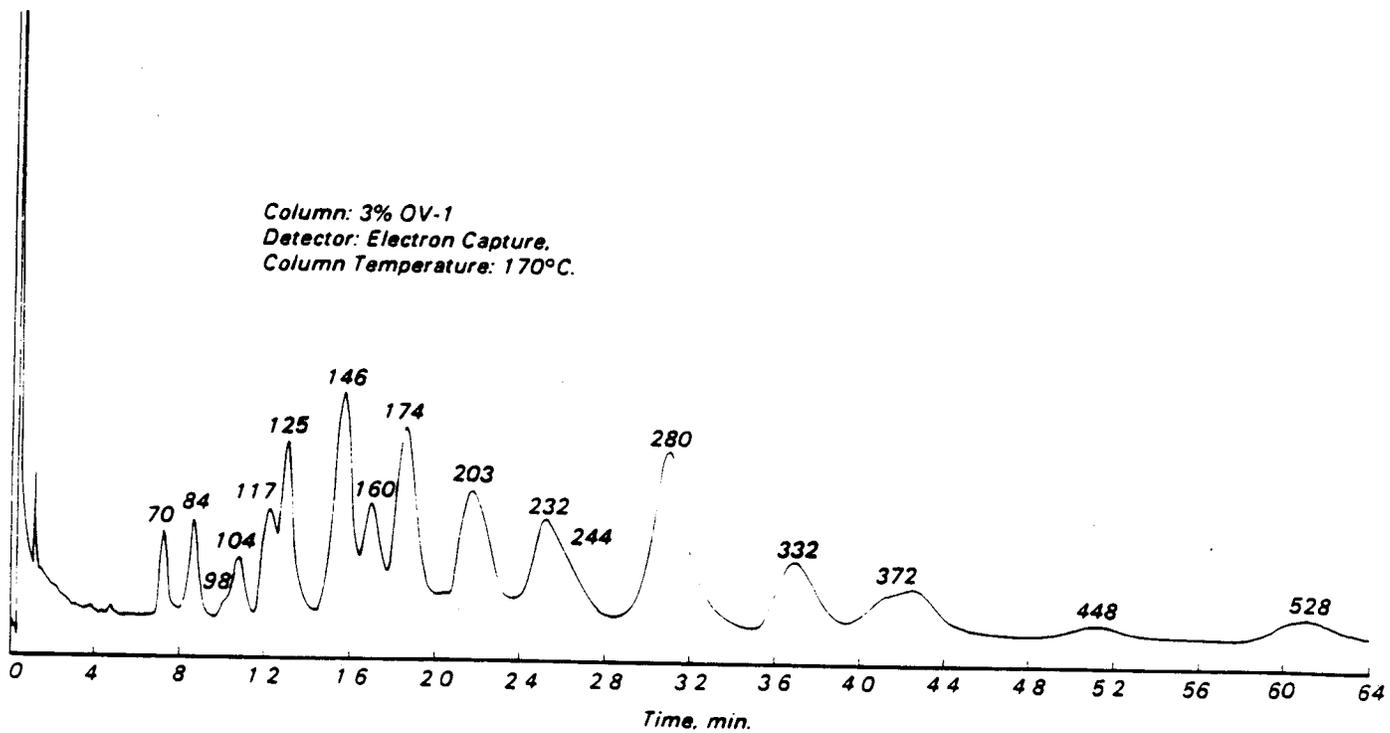


Figure 6. Gas chromatogram of Aroclor 1260.

Column: 3% OV-1  
Detector: Hall 700-A  
Program: 120°C. -6°/Minute to 220°C.

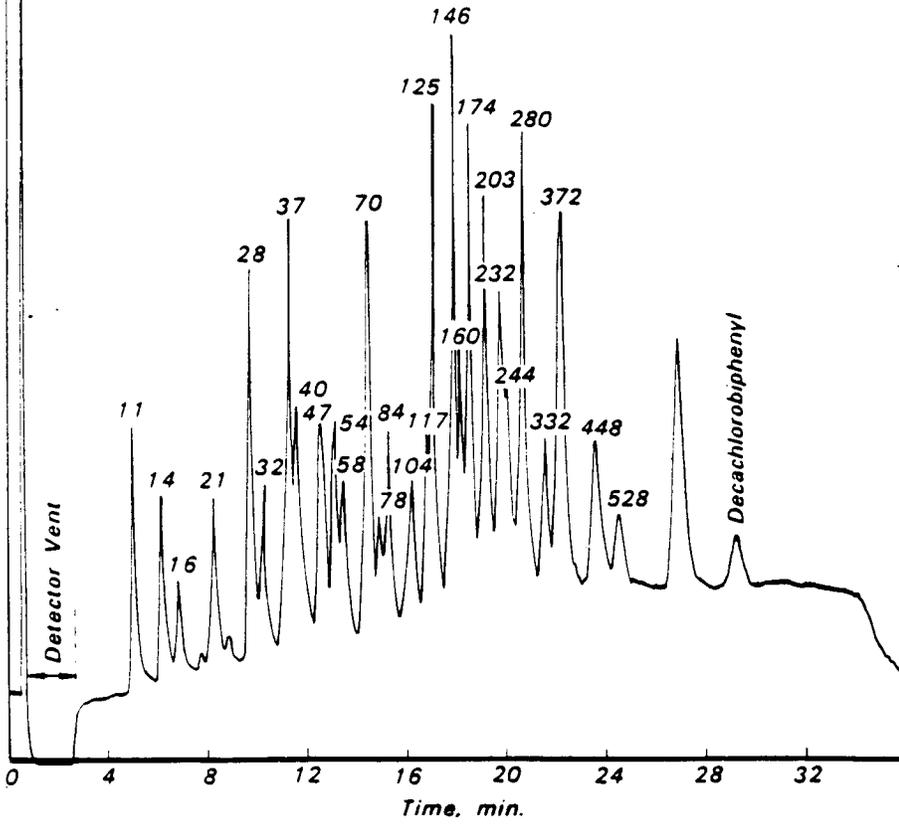


Figure 7. Gas chromatogram of PCB locator mixture.

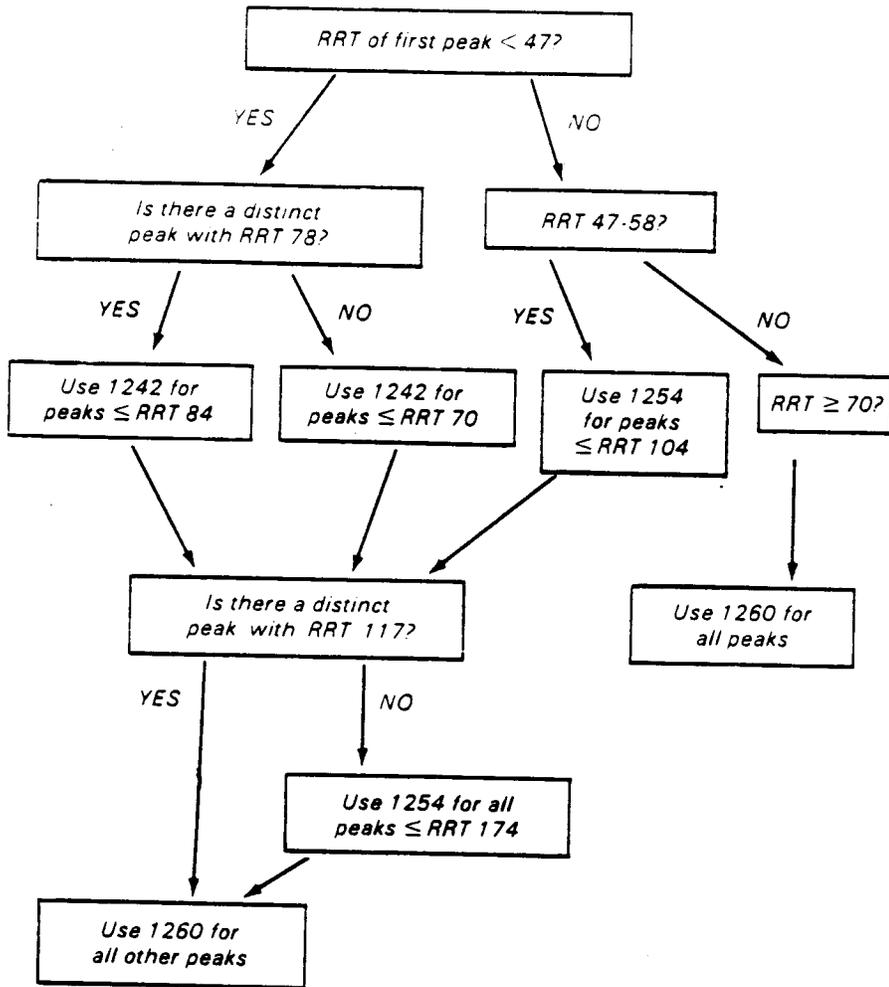


Figure 8. Chromatogram division flowchart.