



Infrastructure, environment, buildings

Mr. Mike Boudloche, Trustee
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Subject:

Additional COPC Assessment Work Plan – Revised September 11, 2012
Former Encycle/ASARCO Facility
5500 Up River Road
Corpus Christi, Texas
TCEQ SWR No. 30003; EPA ID No. TXD008117186

Dear Mr. Boudloche:

As requested in Item No. 3 of the March 9, 2012 Texas Commission on Environmental Quality (TCEQ) letter regarding the March 2012 Soil Remediation Bid Package for the former Encycle/ASARCO facility in Corpus Christi (Site), provided herein is a work plan to collect and analyze additional soil samples from the proposed soil excavation areas prior to initiation of soil excavation activities. As detailed in the May 27, 2005 report entitled “Corrective Measures Study and Corrective Measures Implementation Work Plan”, hundreds of soil samples have already been collected at the Site for analyses of constituents of concern (COCs). The COCs for the Site, which were determined prior to initiation of the RCRA Facility Investigation (RFI), are shown in Attachment 1.

Date:

September 11, 2012

Contact:

Ken Brandner

Phone:

(361) 883-1353

This work plan provides soil sample collection and analyses procedures for the following constituents of potential concern (COPCs):

- Asbestos
- Polychlorinated biphenyls (PCBs)
- Semi-volatile organic compounds (SVOCs)

These COPCs were not manufactured at the Site. However, some of the building construction materials at the Site, including galbestos coating on concrete columns and corrugated metal panels, contained these COPCs. Therefore, soil samples will be collected from the proposed soil excavation areas for analyses of these COPCs. It should be noted that offsite sources of these COPCs may also be present in buildings and structures in the surrounding area, including but not limited to asbestos in offsite buildings older than 1980 (i.e., floor tiles, ceiling tiles, air duct joint compound, drywall joint compound, transite wall panels, roofing felts, etc.), PCBs in offsite transformers older than 1980, and SVOCs in offsite tar or asphalt roofing materials, asphalt roads and parking lots, and underground pipeline cathodic protection coatings. SVOCs, which are components of crude oil, are also actively loaded, unloaded, and processed in large quantities at several nearby refineries.

SOIL SAMPLE COLLECTION PROCEDURES

General

As detailed in the March 16, 2012 “Soil Remediation Bid Documents”, the upcoming soil remediation project will include excavation of soils in the following nine areas:

- Boneyard (Meaney Tract);
- Waste Pile (Meaney Tract);
- NE of 01 Landfill (Encycle Northern Tract);
- Railroad Tracks (Encycle Southern Tract);
- Road Leading to the West of Building C (Encycle Southern Tract);
- Reactor Clarifier (Encycle Southern Tract);
- Building C – Lettered Bins (Encycle Southern Tract);
- Storm Sewer System (Encycle Southern Tract); and
- West Cell House (Encycle Southern Tract).

Soil samples will be collected within and adjacent to the proposed excavation areas as requested in the March 9, 2012 TCEQ letter. Proposed soil sample locations are shown on Figures 1 and 2. The proposed soil samples are located in areas where intact concrete is not present because the COPCs would potentially have been surface releases from the building materials. Therefore, in areas where intact concrete is present (inside the Lettered Bins Building, the concrete driveway east of the former West Cell House), the proposed soil sample locations are in unpaved areas adjacent to the intact concrete.

Soil Sample Collection Methodology

As shown on Figures 1 and 2, a total of 36 soil borings will be installed, including 34 soil borings within and adjacent to the proposed soil excavation areas, and two soil borings adjacent to the former pad-mounted transformer (which had a “non-PCB” label) directly south of the former Power House. The soil sample locations will be sprayed/misted with distilled water immediately prior to soil sample collection for dust suppression purposes. The soil borings will be installed using a stainless steel hand auger. Two soil samples will be collected from each soil boring: (1) a 0-0.5 foot depth soil sample, and (2) a 0.5-1.0 foot depth soil sample.

The soil samples will be transferred from the hand auger directly into laboratory-provided widemouth glass jars with Teflon-lined lids. The sample jars for PCB and SVOC analyses will be securely capped, labeled, placed into coolers with ice, and delivered with chain of custody documentation to a NELAC-accredited analytical laboratory (Test America). The samples for asbestos analysis will be collected by a Texas Department of State Health Services (TDSHS)-licensed asbestos inspector, and the sample jars will be securely capped, labeled, placed into zip-lock plastic bags, and delivered with chain of custody documentation to a TDSHS-licensed

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asbestos laboratory (J3 Resources, Inc.; TDSHS Lic. #300273). Following soil sample collection, the soil borings will be backfilled using bentonite hydrated with distilled water.

Field Documentation

A bound weatherproof notebook will be maintained by the field sampling personnel to document field activities conducted during soil sample collection. The notebook will provide documentation of the sampling locations and sampling procedures. All entries will be completed with an indelible ink pen or indelible marker and will be signed and dated. The field notebook documentation will include the following:

- Site location;
- Date of the sampling event;
- Weather conditions at the time of sampling;
- Sample location and identification;
- Description of sample;
- Sample collection procedures;
- Sample preservation, handling, and transport procedures;
- Names of sampling personnel;
- Time of sample collection; and
- Sample equipment cleaning procedures.

Upon completion of field activities, the field notebook will be relinquished to the project manager, who will maintain the notebook as a permanent record in the project file.

Sample Labeling, Preservation, and Shipment

The following sample labeling, preservation, and shipment procedures will be implemented during field sampling activities.

Sample Labeling

All sample labels will be completed in waterproof ink and attached to the appropriate sample containers. Each sample label will be completed with the facility name; location; the date and time of sample collection; a unique sample identification number; sample type (composite, grab); sample container preservative; the requested analyses; and the initials of the person collecting the sample. An example sample label is provided in Attachment 2.

Sample Preservation

Appropriate preservatives will be provided by the laboratory prior to taking the sample containers to the sampling locations. When each sample is collected, the sample container will be labeled, then placed into an ice chest (cooler). Samples for

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SVOC and PCB analyses will be cooled to an approximate temperature of 6 degrees C or lower using ice. Note the analytical laboratory for **SVOC** and PCB analyses (Test America, 1733 South Padre Island Drive, Corpus Christi, Texas 78408) is located within two miles of the Encycle facility. Therefore, samples collected immediately prior to transport to this nearby analytical laboratory may not have cooled to a temperature less than 6 degrees C due to the short travel time and distance.

Sample Shipment

Samples for **SVOC** and PCB analyses will be delivered to the analytical laboratory (Test America, 1733 South Padre Island Drive, Corpus Christi, Texas 78408) directly by the field sampling team each day of sample collection. The samples for asbestos analysis will be shipped via Federal Express overnight service to the asbestos laboratory (J3 Resources, 6110 W. 34th Street, Houston, Texas). The sample coolers will be accompanied by a chain of custody records completed as described in the following section.

Sample Custody

Sample custody will be documented through the use of chain of custody records and custody seals. Implementation of detailed sample custody procedures will enable the project personnel to document sample shipments from the Site to receipt by the laboratories.

Custody seals will be signed and dated by the field personnel when preparing samples for delivery to the analytical laboratory. The custody seal will be placed on each sample cooler in a location that will preclude opening the sample cooler without breaking the custody seal. The custody seal shall remain intact until the samples are received by the analytical laboratory. Laboratory personnel will inspect each sample cooler upon receipt. If the custody seal is broken, the project manager will be notified and appropriate measures will be implemented to investigate the potential impact on the associated samples. An example custody seal is provided in Attachment 3.

Each time samples are prepared for transport to the laboratories, a chain of custody record will be completed and will accompany the samples. Whenever custody of the samples is transferred, the individuals relinquishing sample custody will retain one carbon copy of the record. The original record will accompany the samples to the laboratory and, upon laboratory receipt, will be returned to the project manager to document that the custody chain was unbroken. The chain of custody record will be completed with the following information:

- Project identification and location;
- Sample identification numbers;
- Signature of sample collector(s);

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- Date and time of sample collection;
- Sample matrix;
- Number and type of sample containers;
- Analyses requested and analytical methods;
- Sample preservation;
- Remarks;
- Signatures of the individuals relinquishing and assuming custody; and
- Date(s) and time(s) when sample custody was relinquished/assumed.

An example chain of custody record is provided in Attachment 4.

Sample Equipment Decontamination

The soil sampling equipment will be cleaned prior to collection of each sample using a non-phosphate detergent wash, potable water rinse, and distilled water final rinse. The equipment decontamination water will be placed into a labeled polyethylene or steel container, then placed into the Lettered Bins Building. A sample of the decontamination water will be collected for analyses of PCBs (EPA Method 8082A), **SVOCs** (EPA Method 8270), and the COCs listed on Table 1 (metals analyses using EPA Method 6010/6020/7470; cyanide analysis using EPA Method 335) for waste evaluation purposes. The equipment decontamination water will be disposed of at an authorized facility, pending disposal facility approval. A manifest will accompany the decontamination water shipment to the disposal facility.

Field Quality Assurance/Quality Control Samples

Trip blank, equipment blank, and duplicate samples will be collected and submitted for analyses of PCBs and **SVOCs** to ensure that sample collection and handling procedures are properly implemented. The analytical data for the field quality assurance/quality control (QA/QC) will be evaluated to determine if the soil sample results may have been influenced by outside factors not related to the actual quality of the source media.

Trip Blanks

Trip blank samples will be prepared to ensure that sample shipment procedures do not impact the quality of the samples collected. Distilled or deionized water will be placed into appropriate sample containers by the analytical laboratory. One set of trip blanks will be prepared for each day that samples are collected for laboratory analyses. The trip blanks will be placed into the same sample cooler as the sample containers delivered to the Site. The trip blanks will remain unopened in the sample cooler during field sample collection activities, and will accompany the samples during shipment back to the analytical laboratory. The trip blanks will be analyzed for **SVOCs** and PCBs.

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Equipment Blanks

Equipment blank samples will be collected during sampling events when field equipment is used to collect samples from more than one sample location (i.e., stainless steel hand auger). The equipment blanks will be collected by taking distilled or deionized water and placing it in contact with the field sampling equipment after the equipment has been cleaned using normal cleaning procedures as described above in the “Soil Sample Collection Methodology” section. The distilled or deionized water will be poured over the sampling equipment and collected directly into the appropriate sample containers. The equipment blanks will be analyzed for SVOCs and PCBs. Equipment blanks will be prepared and analyzed at a frequency of one per 20 samples collected using field equipment, with a minimum of one equipment blank sample per day that samples are collected using field equipment.

Duplicate Samples

Duplicate samples will be collected to ensure that sample collection and handling procedures are implemented consistently. Each duplicate sample will consist of a representative aliquot of the sample being collected. The duplicate samples will be placed into a second container set identical to the initial container set and analyzed for the same parameters as the initial sample. The resulting data can be compared to evaluate sample matrix homogeneity and general laboratory reproducibility. Duplicate samples will be collected and analyzed at a frequency of one per 20 samples, with a minimum of one duplicate sample per day that samples are collected for laboratory analyses.

SOIL SAMPLE ANALYSES PROCEDURES

General

As discussed above, the COPCs were not manufactured at the Site, but some of the building construction materials at the Site (as well as offsite sources) contain these COPCs. Therefore, because potential COPC releases would have been surface releases, the proposed surface (0-0.5 foot depth) soil samples will be analyzed initially. The deeper (0.5-1.0 foot depth) soil samples from each soil boring will be held at the analytical laboratories, and analyzed for the COPCs, if any, detected in the corresponding surface soil sample.

All 36 surface soil samples, located as shown on Figures 1 and 2, will be analyzed for asbestos. All of the surface soil samples will be analyzed for PCBs except the soil samples within and around the Meaney Tract Waste Pile (7 sample locations) and O1 Landfill (2 sample locations) because no galbestos sources were historically located in those areas. SVOC analyses will be performed on the surface soil samples from the following areas: Meaney Tract Boneyard (6 sample locations), Lettered Bins Building (8 sample locations), West Cell House (2 sample locations), and the samples east and west of the West Cell House within the storm sewer system (2

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sample locations). SVOC analyses will not be conducted on the soil samples collected within or around the Meaney Tract Waste Pile, Power House, and 01 Landfill areas because no galbestos sources were historically located in those areas.

The analytical laboratory for this project will be instructed to complete the laboratory analyses of the surface (0-0.5 foot depth) soil samples such that analyses of the deeper soil samples, if COPCs are detected in the corresponding surface sample, can be completed prior to exceeding the allowable holding times specified by the analytical method. If the concentration of asbestos, PCBs, or SVOCs in a surface soil sample exceeds the sample detection limit (SDL), the deeper soil sample at that location will be analyzed for the constituents detected in the corresponding surface sample, including the full suite for SVOCs if detected in the surface sample. Note that constituents detected at low concentrations less than the sample quantitation limit (SQL) are estimated concentrations that are typically flagged with a qualifier (i.e., “J” flag) to indicate the result is estimated.

Test America will be utilized as the analytical laboratory to conduct the SVOC and PCB analyses, and J3 Resources will be utilized as the analytical laboratory to conduct the asbestos analysis. The analytical laboratories shall adhere to the laboratory quality assurance/quality control procedures described herein to ensure the reliability of the data. The following subsections specify the minimum QA/QC procedures with which the laboratories shall comply.

Analytical Procedures

The soil samples will be analyzed for SVOCs using EPA Method 8270 and PCBs using EPA Method 8082A. Analysis of the samples will be performed by the analytical laboratory in accordance with the protocols and QA procedures specified in “Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846).” The EPA Method 8270 and 8082A laboratory methodologies are provided in Attachments 5 and 6, respectively, and the PCB analysis will include a toluene extraction and analysis of Aroclor 1268.

The soil samples for asbestos analysis will be prepared at the asbestos laboratory using Method CARB 435, and then analyzed for asbestos using polarized light microscopy (PLM). If asbestos is detected in any of the soil samples at a concentration less than 10% using PLM, the asbestos concentrations will be confirmed using the Point Count Method. The laboratory sample detection limit (SDL) for asbestos in soil is 0.25%. Additional information regarding laboratory analysis of asbestos in soil is provided in Attachment 7.

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Laboratory QA/QC Samples

Internal laboratory QA/QC samples including blanks, sample spikes, and duplicates will be prepared and analyzed along with each batch of samples submitted for SVOC and PCB analyses. A description of the laboratory QA/QC samples is provided below.

Method Blanks

A method blank will be prepared and analyzed at a frequency not to exceed one per 20 samples. Each method blank will be carried through the entire sample preparation and analytical process. The detection of contaminants in a method blank is indicative of analytical equipment or procedural contamination.

Matrix Spikes

A matrix spike will be prepared and analyzed at a frequency not to exceed one per 20 samples. The matrix spike will assess the ability of the method to recover a known quantity of a constituent from a typical sample matrix. Matrix spikes are prepared by “spiking” a sample with a known quantity of the analyte. A low matrix spike recovery indicates that sample matrix interference may be inhibiting the ability of the analytical procedure to recover the constituent of concern.

Matrix Spike Duplicates

A matrix spike duplicate will also be prepared and analyzed at a frequency not to exceed one per 20 samples. The analytical data reported from the matrix spike duplicate will be compared to the data reported for the matrix spike so that the precision of the analytical method may be evaluated.

Sample Holding Times

All samples will be analyzed within the allowable holding time as specified by the analytical method. The allowable holding time for EPA Method 8082A is 14 days, and the allowable holding time for EPA Method 8270 is 14 days for extraction and 40 days for analysis.

Sample Quantitation Limits

The sample detection limit (SDL) for PCB analysis using EPA Method 8082A is approximately 0.33 milligrams per kilogram (mg/kg). The SDLs for SVOC analysis using EPA Method 8270 vary by compound and are approximately 0.66 mg/kg as detailed in Attachment 5. The SDL for asbestos analysis is approximately 0.25%. The sample quantitation limits (SQLs) are the SDLs adjusted to reflect sample-specific actions, such as dilution, and takes into account sample characteristics, sample preparation, and analytical adjustments. Sample characteristics that may

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affect the SQL include the moisture content in the sample (for solids), the matrix of the sample, and/or the concentration of constituents in the sample. Because the SQL is sample-specific, the SQL in one sample may be higher than, lower than, or equal to the SQL value for the same constituent in another sample.

Proper application of the analytical method includes the use of instrument calibration that brackets the values reported. When the concentration of a compound in a sample exceeds the calibration range, the laboratory may dilute the sample. Since the SQL is a function of the **SDL**, this dilution raises the SQL to a value equal to the **SDL** multiplied by the dilution factor.

COPC ASSESSMENT WORK PLAN OBJECTIVE

The objective of this COPC assessment is to determine if COPC concentrations in the areas of investigation exceed assessment levels, and if so, determine if the impacted soils can be addressed through CMS/CMI implementation. A report summarizing the analytical results with conclusions and recommendations for subsequent environmental activities will be submitted for TCEQ review.

PCBs

General

The soils to be excavated during the upcoming soil remediation project that meet the Target Soil Stabilization Treatment Limits shown on Table 3 of the March 16, 2012 "Soil Remediation Bid Documents" are planned to be disposed of at the Republic Services El Centro Landfill in Robstown, Texas. The El Centro Landfill is a commercial Class 2 non-hazardous landfill authorized to accept soils that contain PCB concentrations less than 50 mg/kg. Therefore, if the PCB concentrations in any of the soil samples collected during the COPC assessment are 50 mg/kg or higher, soils containing ≥ 50 mg/kg PCBs will be disposed of at another offsite disposal facility authorized to accept soils containing ≥ 50 mg/kg PCBs.

PCB Assessment and Remediation Levels – Areas with PCBs Sources <50 mg/kg

Soil samples are being collected for PCB analyses as part of this Additional COPC Assessment Work Plan, as stated on the first page of the TCEQ June 15, 2012 letter, to determine whether unacceptable levels of the COPCs are present potentially deposited by the building demolition activities and the deteriorative condition of galbestos siding/roofing/mastic/adhesives at former buildings. Samples of the galbestos were previously collected by the EPA from the buildings now demolished, and the galbestos concentrations were all less than 50 parts per million (ppm). Samples of the galbestos were also previously collected by the TCEQ from the buildings now demolished, and the galbestos concentrations were all less than 50 ppm. In addition, a comprehensive work plan for galbestos sampling of the buildings now demolished was provided to TCEQ and EPA in a September 28, 2011 work plan entitled "Galbestos Sample Collection and Analysis Plan" that was approved by the EPA in a letter dated October 5, 2011. The PCB concentrations in all of the

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galbestos samples collected from the buildings now demolished were all less than 50 ppm. Also, a former leaking transformer that had been located directly southwest of the Power House was a non-PCB transformer, as documented by the “No PCBs” label on the transformer and the 2004 Encycle PCB Transformer Inventory (this transformer was not listed on the inventory).

The April 14, 2004 BLRA, the August 27, 2004 BLRA Addendum, and the May 27, 2005 Corrective Measures Study and Corrective Measures Implementation Work Plan did not include PRG calculations for PCBs. However, the Corrective Measures Study and Corrective Measures Implementation Work Plan includes institutional controls for the Site including a water well ban, thereby eliminating the groundwater ingestion exposure pathway. The commercial/industrial Soil/Air and Ingestion Standard (SAI-Ind) for PCBs in soil (10 mg/kg) is provided in 30 TAC §335.568. The Texas Risk Reduction Program (TRRP) combined inhalation, ingestion and dermal contact exposure pathway ($T^{ot}Soil_{Comb}$) for PCBs in commercial/industrial soil (7.7 mg/kg) is provided in 30 TAC §350. Therefore the assessment and remediation level for PCBs in soils in areas of the Site where the source PCB concentrations are less than 50 ppm (demolished galbestos-containing buildings and former leaking non-PCB transformer) will be conservatively set at 7.7 mg/kg.

Assessment and Remediation Levels – Areas with PCBs Sources ≥ 50 mg/kg

The September 28, 2011 work plan for galbestos sampling of the buildings now demolished entitled “Galbestos Sample Collection and Analysis Plan” also included collection and analysis of galbestos samples from the Lettered Bins Building, which is not planned to be demolished until after the soil remediation project has been completed. Several galbestos samples from the Lettered Bins Building contained PCB concentrations >50 ppm. Therefore the assessment and remediation level for PCBs in soil within and around the Lettered Bins Building (8 sample locations – see Figure 2) will be 1 ppm (40 CFR §761.61).

SVOCs

The April 14, 2004 BLRA, the August 27, 2004 BLRA Addendum, and the May 27, 2005 Corrective Measures Study and Corrective Measures Implementation Work Plan did not include PRG calculations for SVOCs. However, the Corrective Measures Study and Corrective Measures Implementation Work Plan includes institutional controls for the Site including a water well ban, thereby eliminating the groundwater ingestion exposure pathway. The SAI-Ind for SVOC compounds in soil, provided in 30 TAC §335.568, shall be the assessment level for SVOCs.

Asbestos

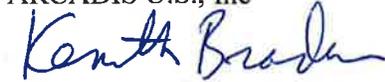
Asbestos-containing material (ACM) is defined as any material or product which contains more than 1 percent asbestos (40 CFR §763.83; 25 TAC §295.32). The EI

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Centro Landfill is authorized to accept non-friable asbestos. The laboratory SDL for asbestos is 0.25%, which shall be the assessment level for asbestos.

Sincerely,

ARCADIS U.S., Inc



Kenneth Brandner, P.E., P.G.
Geological Engineer



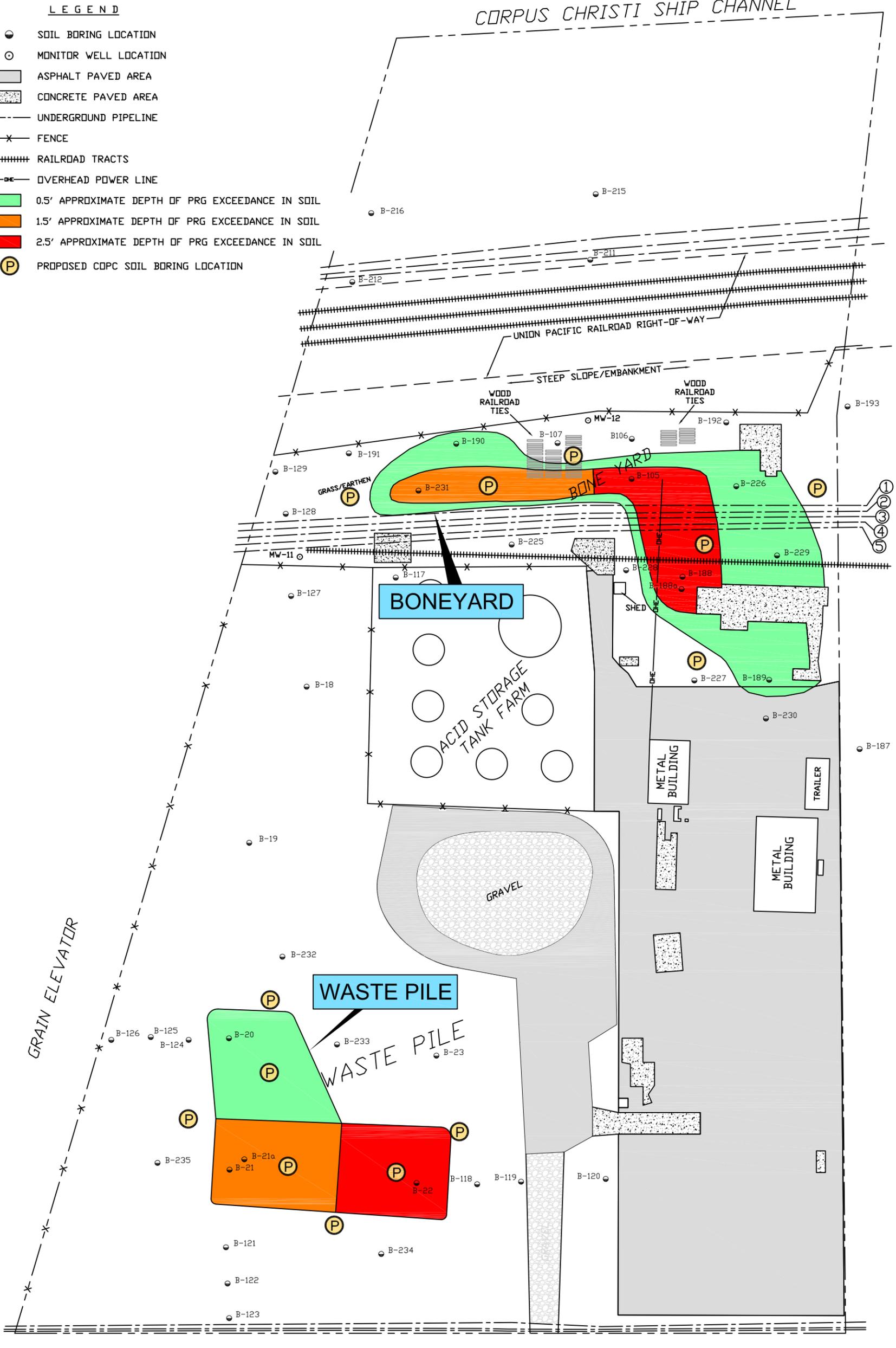
Attachments

- Figure 1: Meaney Tract Proposed COPC Soil Boring Locations
- Figure 2: Encycle Northern and Southern Tracts Proposed COPC Soil Boring Locations
- Attachment 1: Constituents of Concern
- Attachment 2: Sample Label
- Attachment 3: Custody Seal
- Attachment 4: Chain of Custody Record
- Attachment 5: EPA Method 8270
- Attachment 6: EPA Method 8082A
- Attachment 7: Analysis of Asbestos in Soil

LEGEND

- SOIL BORING LOCATION
- MONITOR WELL LOCATION
- ▒ ASPHALT PAVED AREA
- ▒ CONCRETE PAVED AREA
- UNDERGROUND PIPELINE
- x- FENCE
- ||||| RAILROAD TRACTS
- +— OVERHEAD POWER LINE
- 0.5' APPROXIMATE DEPTH OF PRG EXCEEDANCE IN SOIL
- 1.5' APPROXIMATE DEPTH OF PRG EXCEEDANCE IN SOIL
- 2.5' APPROXIMATE DEPTH OF PRG EXCEEDANCE IN SOIL
- Ⓟ PROPOSED CDPC SOIL BORING LOCATION

CORPUS CHRISTI SHIP CHANNEL



ENCYCLE / TEXAS

UP RIVER ROAD

NOTES:

- 1) BASE MAP SURVEYED BY REGISTERED PUBLIC SURVEYOR (SHINER, MOSLEY AND ASSOCIATES, INC.) DURING SEPTEMBER 2004.
- 2) EXTENT AND DEPTH OF AFFECTED SOILS EXCEEDING PRGs TAKEN FROM SOIL SAMPLE ANALYTICAL RESULTS SHOWN ON CMS TABLE 1. SEE CMS TABLE 4 FOR SUMMARY OF CONSTITUENTS IN SOIL EXCEEDING PRGs ON THE MEANEY TRACT.



0 100'



<p>SCALE VERIFICATION</p> <p>THIS BAR REPRESENTS ONE INCH ON THE ORIGINAL DRAWING.</p> <p>USE TO VERIFY FIGURE REPRODUCTION SCALE</p>	PROJECT NO.: CC001231.0001	FILE NO.:
	DRAWING: PRDP CDPC SB	PLOT SIZE: 11x50"
	DRAFTED BY: DPL	DATE: 4/28/12
	CHECKED BY: K. BRANDNER	DATE: 4/28/12
	APPROVED BY: K. BRANDNER	DATE: 4/28/12

MEANEY TRACT
 PROPOSED CDPC SOIL BORING LOCATIONS
 IN AREA OF AFFECTED SOILS TO BE EXCAVATED
 UPDATED AUGUST 2012
 ENCYCLE/TEXAS, INC.
 CORPUS CHRISTI, TEXAS

FIGURE
 1

ATTACHMENT 1

**CONSTITUENTS OF CONCERN, ENCYCLE/TEXAS, INC.
(taken from Table 2 of the "RCRA Facility Investigation
Work Plan" dated January 31, 2000)**

Table 2. Constituents of Concern, Encycle/Texas, Inc., Corpus Christi, Texas

Constituent
Antimony
Arsenic
Barium
Bismuth
Cadmium
Chromium
Cobalt
Copper
Cyanide
Lead
Manganese
Mercury
Nickel
Selenium
Silver
Thallium
Tin
Vanadium
Zinc

ATTACHMENT 2

SAMPLE LABEL

TestAmerica Laboratories
 TestAmerica Corpus Christi
 1733 N. Padre Island Dr. Corpus Christi, TX 78401

Company Name			
Sample ID	Sample Date	Sample Time	
Job No.	Composite	Depth	
	Grain		
Misc. ID Info.			
Preservatives:			
None	HClO ₂	HCL	Filtered
H ₂ O ₂	NaOH	Other	Unfiltered
Analyze Requested Comments			

ATTACHMENT 3

CUSTODY SEAL

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
682202

Custody Seal

DATE

SIGNATURE

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
682202

ATTACHMENT 4

CHAIN OF CUSTODY RECORD

ID#: _____

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ____ of ____

Lab Work Order # _____

<p>Send Results to:</p> <p>Contact & Company Name _____ Telephone _____</p> <p>Address _____ Fax _____</p> <p>City _____ State _____ Zip _____ Email Address _____</p> <p>Project Name/Location (City, State) _____ Project # _____</p> <p>Sampler's Printed Name _____ Sampler's Signature _____</p>		<p>Preservative</p> <p>Filled (✓) _____</p> <p># of Containers _____</p> <p>Container Information _____</p>	<p>PARAMETER ANALYSIS & METHOD</p>	<p>Keys</p> <p>Preservation Key:</p> <p>A. H₂SO₄ B. HCl C. HNO₃ D. NaOH E. None F. Other: _____</p> <p>Container Information Key:</p> <p>1. 40 ml Vial 2. 1 L Amber 3. 250 ml Plastic 4. 500 ml Plastic 5. Encore 6. 2 oz. Glass 7. 4 oz. Glass 8. 8 oz. Glass 9. Other: _____ 10. Other: _____</p> <p>Matrix Key:</p> <p>SE - Sediment NL - NAPL/Oil SO - Soil SL - Sludge SW - Sample Wipe W - Water A - Air T - Tissue Other: _____</p>
<p>Sample ID</p>	<p>Collection</p> <p>Date _____ Time _____</p>	<p>Type (✓)</p> <p>Comp _____ Grab _____</p>	<p>Matrix</p>	
<p>REMARKS</p>				
<p><input type="checkbox"/> Special QA/QC Instructions (✓):</p>				
<p>Laboratory Information and Receipt</p> <p>Lab Name _____</p> <p><input type="checkbox"/> Cooler packed with ice (✓) <input type="checkbox"/> Intact <input type="checkbox"/> Not Intact</p> <p>Specify Turnaround Requirements _____</p> <p>Shipping Tracking # _____</p>		<p>Relinquished By</p> <p>Printed Name _____</p> <p>Signature _____</p> <p>Firm _____</p> <p>Date/Time _____</p>	<p>Received By</p> <p>Printed Name _____</p> <p>Signature _____</p> <p>Firm/Counter _____</p> <p>Date/Time _____</p>	<p>Relinquished By</p> <p>Printed Name _____</p> <p>Signature _____</p> <p>Firm _____</p> <p>Date/Time _____</p>

ATTACHMENT 5

EPA METHOD 8270

TestAmerica Laboratories, Inc. Method 8270C Soil MDLs, Texas (Current Limits September 2012)

Analyte Order	Analytes	CAS #	Limits	Units	Limit Type	Activation Date	Analyte Type
0	Acenaphthene	83-32-9	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
10	1,4-Dichlorobenzene-d4	3855-82-1		ug/Kg	MDL	2011-5-10 9:27 AM	Internal Standard
20	Naphthalene-d8	1146-65-2		ug/Kg	MDL	2011-5-10 9:27 AM	Internal Standard
30	Acenaphthene-d10	15067-26-2		ug/Kg	MDL	2011-5-10 9:27 AM	Internal Standard
40	Phenanthrene-d10	1517-22-2		ug/Kg	MDL	2011-5-10 9:27 AM	Internal Standard
50	Chrysene-d12	1719-03-5		ug/Kg	MDL	2011-5-10 9:27 AM	Internal Standard
60	Perylene-d12	1520-96-3		ug/Kg	MDL	2011-5-10 9:27 AM	Internal Standard
70	Acenaphthylene	208-96-8	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
80	Acetophenone	98-86-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
110	Aniline	62-53-3	47.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
120	Anthracene	120-12-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
140	Benzidine	92-87-5	333	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
150	Benzofluranthene	56-55-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
160	Benzofluoranthene	205-99-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
170	Benzofluoranthene	205-82-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
180	Benzofluoranthene	207-08-9	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
190	Benzofluoranthene	191-24-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
200	Benzofluoranthene	50-32-8	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
210	Benzofluoranthene	100-51-6	24.5	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
220	Butyl benzyl phthalate	85-68-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
230	Bis(2-chloroethoxy)methane	111-91-1	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
240	Bis(2-chloroethyl)ether	111-44-4	37.4	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
260	Di(2-ethylhexyl)adipate	103-23-1	19.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
270	Benz(c)acridine	225-51-4	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
280	Bis(2-ethylhexyl) phthalate	117-81-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
290	4-Bromophenyl phenyl ether	101-55-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
300	Carbazole	86-74-8	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter

TestAmerica Laboratories, Inc. Method 8270C Soil MDLs, Corpus Christi, Texas (Current Limits September 2012)

Analyte Order	Analytes	CAS #	Limits	Units	Limit Type	Activation Date	Analyte Type
310	4-Chloroaniline	106-47-8	46.6	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
330	2-Chloronaphthalene	91-58-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
340	4-Chlorophenyl phenyl ether	7005-72-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
350	Chrysene	218-01-9	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
390	Dibenz[a,h]acridine	226-36-8	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
400	Dibenz[a,j]acridine	224-42-0	22.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
410	Dibenz(a,h)anthracene	53-70-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
420	7H-Dibenzo[c,g]carbazole	194-59-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
430	Dibenzo[a,e]pyrene	192-65-4	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
440	Dibenzo[a,h]pyrene	189-64-0	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
450	1,2,7,8-Dibenzopyrene	189-55-9	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
460	Dibenzofuran	132-64-9	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
470	1,2-Dichlorobenzene	95-50-1	52.1	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
480	1,3-Dichlorobenzene	541-73-1	43.8	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
490	1,4-Dichlorobenzene	106-46-7	45.8	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
500	3,3'-Dichlorobenzidine	91-94-1	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
520	Diethyl phthalate	84-66-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
550	7,12-Dimethylbenz(a)anthracene	57-97-6	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
570	Dimethyl phthalate	131-11-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
580	Di-n-butyl phthalate	84-74-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
590	Di-n-octyl phthalate	117-84-0	18.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
620	2,4-Dinitrotoluene	121-14-2	21.1	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
630	2,6-Dinitrotoluene	606-20-2	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
650	1,1'-Biphenyl	92-52-4	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
710	Fluoranthene	206-44-0	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
720	Fluorene	86-73-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
740	Hexachlorobenzene	118-74-1	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter

TestAmerica Laboratories, Inc. Method 8270C Soil MDLs, Corpus Christi, Texas (Current Limits September 2012)

Analyte Order	Analytes	CAS #	Limits	Units	Limit Type	Activation Date	Analyte Type
750	Hexachlorobutadiene	87-68-3	44.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
760	Hexachlorocyclopentadiene	77-47-4	100.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
770	Hexachloroethane	67-72-1	50.1	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
790	Hexachloropropene	1888-71-7	47.3	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
810	Indene	95-13-6	44.2	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
820	Indeno[1,2,3-cd]pyrene	193-39-5	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
840	Isophorone	78-59-1	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
880	3-Methylcholanthrene	56-49-5	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
910	6-Methylchrysene	1705-85-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
930	1-Methylnaphthalene	90-12-0	31.8	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
940	2-Methylnaphthalene	91-57-6	31.1	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
950	2-Nitroaniline	88-74-4	22.2	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
960	3-Nitroaniline	99-09-2	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
970	4-Nitroaniline	100-01-6	28.1	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
980	Naphthalene	91-20-3	41.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1020	Nitrobenzene	98-95-3	36.4	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1040	N-Nitrosodi-n-butylamine	924-16-3	19.8	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1050	N-Nitrosodiethylamine	55-18-5	26.4	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1060	N-Nitrosodimethylamine	62-75-9	30.2	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1090	N-Nitrosodi-n-propylamine	621-64-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1100	N-Nitrosodiphenylamine	86-30-6	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1150	Pentachlorobenzene	608-93-5	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1160	Pentachloroethane	76-01-7	51.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1170	Pentachloronitrobenzene	82-68-8	330.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1190	Phenanthrene	85-01-8	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1250	Pyrene	129-00-0	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1260	Pyridine	110-86-1	33.4	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter

TestAmerica Laboratories, Inc. Method 8270C Soil MDLs, Texas (Current Limits September 2012)

Analyte Order	Analytes	CAS #	Limits	Units	Limit Type	Activation Date	Analyte Type
1270	Quinoline	91-22-5	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1310	1,2,4,5-Tetrachlorobenzene	95-94-3	38.4	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1330	2-Toluidine	95-53-4	330.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1340	1,2,4-Trichlorobenzene	120-82-1	45.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1370	Benzoic acid	65-85-0	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1380	4-Chloro-3-methylphenol	59-50-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1390	2-Chlorophenol	95-57-8	27.8	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1400	2,4-Dichlorophenol	120-83-2	22.8	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1410	2,6-Dichlorophenol	87-65-0	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1420	2,4-Dimethylphenol	105-67-9	20.4	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1430	2,4-Dinitrophenol	51-28-5	100	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1440	4,6-Dinitro-2-methylphenol	534-52-1	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1450	2-Methylphenol	95-48-7	33.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1460	3 & 4 Methylphenol	15831-10-4	50.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1470	2-Nitrophenol	88-75-5	17.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1480	4-Nitrophenol	100-02-7	30.5	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1490	Pentachlorophenol	87-86-5	100.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1500	Phenol	108-95-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1510	Phenylmercaptan	108-98-5	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1520	2,4,6-Trichlorophenol	88-06-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1530	2,3,4,6-Tetrachlorophenol	58-90-2	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1540	2,4,5-Trichlorophenol	95-95-4	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1580	Nitrobenzene-d5	4165-60-0		ug/Kg	MDL	2011-5-10 9:27 AM	Surrogate Cpnd
1590	2-Fluorobiphenyl	321-60-8		ug/Kg	MDL	2011-5-10 9:27 AM	Surrogate Cpnd
1600	Terphenyl-d14	1718-51-0		ug/Kg	MDL	2011-5-10 9:27 AM	Surrogate Cpnd
1610	Phenol-d5	4165-62-2		ug/Kg	MDL	2011-5-10 9:27 AM	Surrogate Cpnd
1620	2-Fluorophenol	367-12-4		ug/Kg	MDL	2011-5-10 9:27 AM	Surrogate Cpnd

TestAmerica Laboratories, Inc. Method 8270C Soil MDLs, Texas (Current Limits September 2012)

Analyte Order	Analytes	CAS #	Limits	Units	Limit Type	Activation Date	Analyte Type
1630	2,4,6-Tribromophenol	118-79-6		ug/Kg	MDL	2011-5-10 9:27 AM	Surrogate Cpnd
1650	1,3-Dimethylnaphthalene	575-41-7	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1670	Sulfolane	126-33-0	22.9	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1680	Azobenzene	103-33-3	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1690	Tentatively Identified Compound	STL00231		ug/Kg	MDL	2011-5-10 9:27 AM	TIC (Tentatively Identified Compound)
1740	1,3-Dichloro-2-propanol	96-23-1	16.7	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1770	2,2'-oxybis[1-chloropropane]	108-60-1	41.0	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1780	DFTPP	5074-71-5		ug/Kg	MDL	2011-5-10 9:27 AM	Tune Analyte
1820	4,4'-DDD	72-54-8		ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1830	4,4'-DDE	72-55-9		ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1840	4,4'-DDT	50-29-3		ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1850	2,4'-DDD	53-19-0		ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1860	1,4-Dioxane	123-91-1	167	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter
1880	Sulfur	7704-34-9	670	ug/Kg	MDL	2011-5-10 9:27 AM	Analyte/Parameter

METHOD 8270C

SEMIVOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

Compounds	CAS No ^a	Appropriate Preparation Techniques ^b				
		3510	3520	3540/ 3541	3550	3580
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthene-d ₁₀ (IS)		X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea	591-08-2	LR	ND	ND	ND	LR
Aldrin	309-00-2	X	X	X	X	X
2-Aminoanthraquinone	117-79-3	X	ND	ND	ND	X
Aminoazobenzene	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcarbazole	132-32-1	X	X	ND	ND	ND
Anilazine	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite	140-57-8	HS(43)	ND	ND	ND	X
Aroclor 1016	12674-11-2	X	X	X	X	X
Aroclor 1221	11104-28-2	X	X	X	X	X
Aroclor 1232	11141-16-5	X	X	X	X	X
Aroclor 1242	53469-21-9	X	X	X	X	X
Aroclor 1248	12672-29-6	X	X	X	X	X
Aroclor 1254	11097-69-1	X	X	X	X	X
Aroclor 1260	11096-82-5	X	X	X	X	X
Azinphos-methyl	86-50-0	HS(62)	ND	ND	ND	X
Barban	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP
Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X
Benzo(k)fluoranthene	207-08-9	X	X	X	X	X
Benzo(g,h,i)perylene	191-24-2	X	X	X	X	X
Benzo(a)pyrene	50-32-8	X	X	X	X	X

Appropriate Preparation Techniques^b

Compounds	CAS No ^a	Appropriate Preparation Techniques ^b				
		3510	3520	3540/ 3541	3550	3580
p-Benzoquinone	106-51-4	OE	ND	ND	ND	X
Benzyl alcohol	100-51-6	X	X	ND	X	X
α-BHC	319-84-6	X	X	X	X	X
β-BHC	319-85-7	X	X	X	X	X
δ-BHC	319-86-8	X	X	X	X	X
γ-BHC (Lindane)	58-89-9	X	X	X	X	X
Bis(2-chloroethoxy)methane	111-91-1	X	X	X	X	X
Bis(2-chloroethyl) ether	111-44-4	X	X	X	X	X
Bis(2-chloroisopropyl) ether	108-60-1	X	X	X	X	X
Bis(2-ethylhexyl) phthalate	117-81-7	X	X	X	X	X
4-Bromophenyl phenyl ether	101-55-3	X	X	X	X	X
Bromoxynil	1689-84-5	X	ND	ND	ND	X
Butyl benzyl phthalate	85-68-7	X	X	X	X	X
Captafol	2425-06-1	HS(55)	ND	ND	ND	X
Captan	133-06-2	HS(40)	ND	ND	ND	X
Carbaryl	63-25-2	X	ND	ND	ND	X
Carbofuran	1563-66-2	X	ND	ND	ND	X
Carbophenothion	786-19-6	X	ND	ND	ND	X
Chlordane (NOS)	57-74-9	X	X	X	X	X
Chlorfenvinphos	470-90-6	X	ND	ND	ND	X
4-Chloroaniline	106-47-8	X	ND	ND	ND	X
Chlorobenzilate	510-15-6	X	ND	ND	ND	X
5-Chloro-2-methylaniline	95-79-4	X	ND	ND	ND	X
4-Chloro-3-methylphenol	59-50-7	X	X	X	X	X
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	X	ND	ND	ND	X
1-Chloronaphthalene	90-13-1	X	X	X	X	X
2-Chloronaphthalene	91-58-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X
4-Chloro-1,2-phenylenediamine	95-83-0	X	X	ND	ND	ND
4-Chloro-1,3-phenylenediamine	5131-60-2	X	X	ND	ND	ND
4-Chlorophenyl phenyl ether	7005-72-3	X	X	X	X	X
Chrysene	218-01-9	X	X	X	X	X
Chrysene-d ₁₂ (IS)		X	X	X	X	X
Coumaphos	56-72-4	X	ND	ND	ND	X
p-Cresidine	120-71-8	X	ND	ND	ND	X
Crotoxyphos	7700-17-6	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitro-phenol	131-89-5	X	ND	ND	ND	LR
4,4'-DDD	72-54-8	X	X	X	X	X
4,4'-DDE	72-55-9	X	X	X	X	X
4,4'-DDT	50-29-3	X	X	X	X	X
Demeton-O	298-03-3	HS(68)	ND	ND	ND	X
Demeton-S	126-75-0	X	ND	ND	ND	X
Diallate (cis or trans)	2303-16-4	X	ND	ND	ND	X

Appropriate Preparation Techniques^b

Compounds	CAS No ^a	Appropriate Preparation Techniques ^b				
		3510	3520	3540/ 3541	3550	3580
2,4-Diaminotoluene	95-80-7	DC,OE(42)	ND	ND	ND	X
Dibenz(a,j)acridine	224-42-0	X	ND	ND	ND	X
Dibenz(a,h)anthracene	53-70-3	X	X	X	X	X
Dibenzofuran	132-64-9	X	X	ND	X	X
Dibenzo(a,e)pyrene	192-65-4	ND	ND	ND	ND	X
1,2-Dibromo-3-chloropropane	96-12-8	X	X	ND	ND	ND
Di-n-butyl phthalate	84-74-2	X	X	X	X	X
Dichlone	117-80-6	OE	ND	ND	ND	X
1,2-Dichlorobenzene	95-50-1	X	X	X	X	X
1,3-Dichlorobenzene	541-73-1	X	X	X	X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X	X
1,4-Dichlorobenzene-d ₄ (IS)		X	X	X	X	X
3,3'-Dichlorobenzidine	91-94-1	X	X	X	X	X
2,4-Dichlorophenol	120-83-2	X	X	X	X	X
2,6-Dichlorophenol	87-65-0	X	ND	ND	ND	X
Dichlorovos	62-73-7	X	ND	ND	ND	X
Dicrotophos	141-66-2	X	ND	ND	ND	X
Dieldrin	60-57-1	X	X	X	X	X
Diethyl phthalate	84-66-2	X	X	X	X	X
Diethylstilbestrol	56-53-1	AW,OS(67)	ND	ND	ND	X
Diethyl sulfate	64-67-5	LR	ND	ND	ND	LR
Dihydrosaffrole	56312-13-1	ND	ND	ND	ND	ND
Dimethoate	60-51-5	HE,HS(31)	ND	ND	ND	X
3,3'-Dimethoxybenzidine	119-90-4	X	ND	ND	ND	LR
Dimethylaminoazobenzene	60-11-7	X	ND	ND	ND	X
7,12-Dimethylbenz(a)-anthracene	57-97-6	CP(45)	ND	ND	ND	CP
3,3'-Dimethylbenzidine	119-93-7	X	ND	ND	ND	X
α,α-Dimethylphenethylamine	122-09-8	ND	ND	ND	ND	X
2,4-Dimethylphenol	105-67-9	X	X	X	X	X
Dimethyl phthalate	131-11-3	X	X	X	X	X
1,2-Dinitrobenzene	528-29-0	X	ND	ND	ND	X
1,3-Dinitrobenzene	99-65-0	X	ND	ND	ND	X
1,4-Dinitrobenzene	100-25-4	HE(14)	ND	ND	ND	X
4,6-Dinitro-2-methylphenol	534-52-1	X	X	X	X	X
2,4-Dinitrophenol	51-28-5	X	X	X	X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X	X	X	X	X
Dinocap	39300-45-3	CP,HS(28)	ND	ND	ND	CP
Dinoseb	88-85-7	X	ND	ND	ND	X
Dioxathion	78-34-2	ND	ND	ND	ND	ND
Diphenylamine	122-39-4	X	X	X	X	X
5,5-Diphenylhydantoin	57-41-0	X	ND	ND	ND	X
1,2-Diphenylhydrazine	122-66-7	X	X	X	X	X

Appropriate Preparation Techniques^b

Compounds	CAS No ^a	Appropriate Preparation Techniques ^b				
		3510	3520	3540/ 3541	3550	3580
Di-n-octyl phthalate	117-84-0	X	X	X	X	X
Disulfoton	298-04-4	X	ND	ND	ND	X
Endosulfan I	959-98-8	X	X	X	X	X
Endosulfan II	33213-65-9	X	X	X	X	X
Endosulfan sulfate	1031-07-8	X	X	X	X	X
Endrin	72-20-8	X	X	X	X	X
Endrin aldehyde	7421-93-4	X	X	X	X	X
Endrin ketone	53494-70-5	X	X	ND	X	X
EPN	2104-64-5	X	ND	ND	ND	X
Ethion	563-12-2	X	ND	ND	ND	X
Ethyl carbamate	51-79-6	DC(28)	ND	ND	ND	X
Ethyl methanesulfonate	62-50-0	X	ND	ND	ND	X
Famphur	52-85-7	X	ND	ND	ND	X
Fensulfothion	115-90-2	X	ND	ND	ND	X
Fenthion	55-38-9	X	ND	ND	ND	X
Fluchloralin	33245-39-5	X	ND	ND	ND	X
Fluoranthene	206-44-0	X	X	X	X	X
Fluorene	86-73-7	X	X	X	X	X
2-Fluorobiphenyl (surr)	321-60-8	X	X	X	X	X
2-Fluorophenol (surr)	367-12-4	X	X	X	X	X
Heptachlor	76-44-8	X	X	X	X	X
Heptachlor epoxide	1024-57-3	X	X	X	X	X
Hexachlorobenzene	118-74-1	X	X	X	X	X
Hexachlorobutadiene	87-68-3	X	X	X	X	X
Hexachlorocyclopentadiene	77-47-4	X	X	X	X	X
Hexachloroethane	67-72-1	X	X	X	X	X
Hexachlorophene	70-30-4	AW,CP(62)	ND	ND	ND	CP
Hexachloropropene	1888-71-7	X	ND	ND	ND	X
Hexamethylphosphoramide	680-31-9	X	ND	ND	ND	X
Hydroquinone	123-31-9	ND	ND	ND	ND	X
Indeno(1,2,3-cd)pyrene	193-39-5	X	X	X	X	X
Isodrin	465-73-6	X	ND	ND	ND	X
Isophorone	78-59-1	X	X	X	X	X
Isosafrole	120-58-1	DC(46)	ND	ND	ND	X
Kepone	143-50-0	X	ND	ND	ND	X
Leptophos	21609-90-5	X	ND	ND	ND	X
Malathion	121-75-5	HS(5)	ND	ND	ND	X
Maleic anhydride	108-31-6	HE	ND	ND	ND	X
Mestranol	72-33-3	X	ND	ND	ND	X
Methapyrilene	91-80-5	X	ND	ND	ND	X
Methoxychlor	72-43-5	X	ND	ND	ND	X
3-Methylcholanthrene	56-49-5	X	ND	ND	ND	X
4,4'-Methylenebis (2-chloroaniline)	101-14-4	OE,OS(0)	ND	ND	ND	LR

Appropriate Preparation Techniques^b

Compounds	CAS No ^a	3510	3520	3540/ 3541	3550	3580
4,4'-Methylenebis (N,N-dimethylaniline)	101-61-1	X	X	ND	ND	ND
Methyl methanesulfonate	66-27-3	X	ND	ND	ND	X
2-Methylnaphthalene	91-57-6	X	X	ND	X	X
Methyl parathion	298-00-0	X	ND	ND	ND	X
2-Methylphenol	95-48-7	X	ND	ND	ND	X
3-Methylphenol	108-39-4	X	ND	ND	ND	X
4-Methylphenol	106-44-5	X	ND	ND	ND	X
Mevinphos	7786-34-7	X	ND	ND	ND	X
Mexacarbate	315-18-4	HE,HS(68)	ND	ND	ND	X
Mirex	2385-85-5	X	ND	ND	ND	X
Monocrotophos	6923-22-4	HE	ND	ND	ND	X
Naled	300-76-5	X	ND	ND	ND	X
Naphthalene	91-20-3	X	X	X	X	X
Naphthalene-d ₈ (IS)		X	X	X	X	X
1,4-Naphthoquinone	130-15-4	X	ND	ND	ND	X
1-Naphthylamine	134-32-7	OS(44)	ND	ND	ND	X
2-Naphthylamine	91-59-8	X	ND	ND	ND	X
Nicotine	54-11-5	DE(67)	ND	ND	ND	X
5-Nitroacenaphthene	602-87-9	X	ND	ND	ND	X
2-Nitroaniline	88-74-4	X	X	ND	X	X
3-Nitroaniline	99-09-2	X	X	ND	X	X
4-Nitroaniline	100-01-6	X	X	ND	X	X
5-Nitro-o-anisidine	99-59-2	X	ND	ND	ND	X
Nitrobenzene	98-95-3	X	X	X	X	X
Nitrobenzene-d ₅ (surr)		X	X	X	X	X
4-Nitrobiphenyl	92-93-3	X	ND	ND	ND	X
Nitrofen	1836-75-5	X	ND	ND	ND	X
2-Nitrophenol	88-75-5	X	X	X	X	X
4-Nitrophenol	100-02-7	X	X	X	X	X
5-Nitro-o-toluidine	99-55-8	X	X	ND	ND	X
Nitroquinoline-1-oxide	56-57-5	X	ND	ND	ND	X
N-Nitrosodi-n-butylamine	924-16-3	X	ND	ND	ND	X
N-Nitrosodiethylamine	55-18-5	X	ND	ND	ND	X
N-Nitrosodimethylamine	62-75-9	X	X	X	X	X
N-Nitrosomethylethylamine	10595-95-6	X	ND	ND	ND	X
N-Nitrosodiphenylamine	86-30-6	X	X	X	X	X
N-Nitrosodi-n-propylamine	621-64-7	X	X	X	X	X
N-Nitrosomorpholine	59-89-2	ND	ND	ND	ND	X
N-Nitrosopiperidine	100-75-4	X	ND	ND	ND	X
N-Nitrosopyrrolidine	930-55-2	X	ND	ND	ND	X
Octamethyl pyrophosphoramidate	152-16-9	LR	ND	ND	ND	LR
4,4'-Oxydianiline	101-80-4	X	ND	ND	ND	X

Appropriate Preparation Techniques^b

Compounds	CAS No ^a	3540/				
		3510	3520	3541	3550	3580
Parathion	56-38-2	X	X	ND	ND	X
Pentachlorobenzene	608-93-5	X	ND	ND	ND	X
Pentachloronitrobenzene	82-68-8	X	ND	ND	ND	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Perylene-d ₁₂ (IS)		X	X	X	X	X
Phenacetin	62-44-2	X	ND	ND	ND	X
Phenanthrene	85-01-8	X	X	X	X	X
Phenanthrene-d ₁₀ (IS)		X	X	X	X	X
Phenobarbital	50-06-6	X	ND	ND	ND	X
Phenol	108-95-2	DC(28)	X	X	X	X
Phenol-d ₆ (surr)		DC(28)	X	X	X	X
1,4-Phenylenediamine	106-50-3	X	ND	ND	ND	X
Phorate	298-02-2	X	ND	ND	ND	X
Phosalone	2310-17-0	HS(65)	ND	ND	ND	X
Phosmet	732-11-6	HS(15)	ND	ND	ND	X
Phosphamidon	13171-21-6	HE(63)	ND	ND	ND	X
Phthalic anhydride	85-44-9	CP,HE(1)	ND	ND	ND	CP
2-Picoline (2-Methylpyridine)	109-06-8	X	X	ND	ND	ND
Piperonyl sulfoxide	120-62-7	X	ND	ND	ND	X
Pronamide	23950-58-5	X	ND	ND	ND	X
Propylthiouracil	51-52-5	LR	ND	ND	ND	LR
Pyrene	129-00-0	X	X	X	X	X
Pyridine	110-86-1	ND	ND	ND	ND	ND
Resorcinol	108-46-3	DC,OE(10)	ND	ND	ND	X
Safrole	94-59-7	X	ND	ND	ND	X
Strychnine	57-24-9	AW,OS(55)	ND	ND	ND	X
Sulfallate	95-06-7	X	ND	ND	ND	X
Terbufos	13071-79-9	X	ND	ND	ND	X
Terphenyl-d ₁₄ (surr)	1718-51-0	X	X	ND	X	X
1,2,4,5-Tetrachlorobenzene	95-94-3	X	ND	ND	ND	X
2,3,4,6-Tetrachlorophenol	58-90-2	X	ND	ND	ND	X
Tetrachlorvinphos	961-11-5	X	ND	ND	ND	X
Tetraethyl dithiopyrophosphate	3689-24-5	X	X	ND	ND	ND
Tetraethyl pyrophosphate	107-49-3	X	ND	ND	ND	X
Thionazine	297-97-2	X	ND	ND	ND	X
Thiophenol (Benzenethiol)	108-98-5	X	ND	ND	ND	X
Toluene diisocyanate	584-84-9	HE(6)	ND	ND	ND	X
o-Toluidine	95-53-4	X	ND	ND	ND	X
Toxaphene	8001-35-2	X	X	X	X	X
2,4,6-Tribromophenol (surr)	118-79-6	X	X	X	X	X
1,2,4-Trichlorobenzene	120-82-1	X	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	ND	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X
Trifluralin	1582-09-8	X	ND	ND	ND	X

Compounds	CAS No ^a	Appropriate Preparation Techniques ^b				
		3510	3520	3540/ 3541	3550	3580
2,4,5-Trimethylaniline	137-17-7	X	ND	ND	ND	X
Trimethyl phosphate	512-56-1	HE(60)	ND	ND	ND	X
1,3,5-Trinitrobenzene	99-35-4	X	ND	ND	ND	X
Tris(2,3-dibromopropyl) phosphate	126-72-7	X	ND	ND	ND	LR
Tri-p-tolyl phosphate	78-32-0	X	ND	ND	ND	X
O,O,O-Triethyl phosphorothioate	126-68-1	X	ND	ND	ND	X

^a Chemical Abstract Service Registry Number

^b See Sec. 1.2 for other acceptable preparation methods.

KEY TO ANALYTE LIST

- IS = This compound may be used as an internal standard.
surr = This compound may be used as a surrogate.
AW = Adsorption to walls of glassware during extraction and storage.
CP = Nonreproducible chromatographic performance.
DC = Unfavorable distribution coefficient (number in parenthesis is percent recovery).
HE = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
HS = Hydrolysis during storage (number in parenthesis is percent stability).
LR = Low response.
ND = Not determined.
OE = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
OS = Oxidation during storage (number in parenthesis is percent stability).
X = Greater than 70 percent recovery by this technique.

1.2 In addition to the sample preparation methods listed in the above analyte list, Method 3542 describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 11 contains surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid extraction of solids for PAHs (see Tables 13, 14, and 15 for performance data).

1.3 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated.

In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

1.4 The following compounds may require special treatment when being determined by this method:

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices, α -BHC, γ -BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.4.6 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4.7 Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4.8 Toluene diisocyanate rapidly hydrolyses in water (half-life of less than 30 min.). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.9 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660 $\mu\text{g}/\text{kg}$ (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 $\mu\text{g}/\text{L}$ for ground water samples (see Table 2). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm ID) 1 μ m film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 Mass spectrometer

4.1.3.1 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 3 when 1 μ L of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.1.6 Guard column (optional) - (J&W Deactivated Fused Silica, 0.25 mm ID x 6 m, or equivalent) between the injection port and the analytical column joined with column joiners (Hewlett-Packard Catalog No. 5062-3556, or equivalent).

4.2 Syringe - 10- μ L.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Balance - Analytical, capable of weighing 0.0001 g.

4.5 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Stock standard solutions (1000 mg/L) - Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.2 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

5.3.4 It is recommended that nitrosamine compounds be placed together in a separate calibration mix and not combined with other calibration mixes. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.3.5 Mixes with hydrochloride salts may contain hydrochloric acid, which can cause analytical difficulties. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.4 Internal standard solutions - The internal standards recommended are 1,4-dichlorobenzene-d₄, naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂, and perylene-d₁₂ (see Table 5). Other compounds may be used as internal standards as long as the requirements given in Sec. 7.3.2 are met.

5.4.1 Dissolve 0.200 g of each compound with a small volume of carbon disulfide. Transfer to a 50 mL volumetric flask and dilute to volume with methylene chloride so that the final solvent is approximately 20% carbon disulfide. Most of the compounds are also soluble in small volumes of methanol, acetone, or toluene, except for perylene-d₁₂. The resulting solution will contain each standard at a concentration of 4,000 ng/μL. Each 1 mL sample extract undergoing analysis should be spiked with 10 μL of the internal standard solution, resulting in a concentration of 40 ng/μL of each internal standard. Store at -10°C or less when not in use. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.4.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute internal standard solution may be required. Area counts of the internal standard peaks should be between 50-200% of the area of the target analytes in the mid-point calibration analysis.

5.5 GC/MS tuning standard - A methylene chloride solution containing 50 ng/μL of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/μL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at -10°C or less when not in use. If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute tuning solution may be necessary. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five different concentrations. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method.

5.6.1 It is the intent of EPA that all target analytes for a particular analysis be included in the calibration standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.6.2 Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the internal standard solution prior to analysis. All standards should be stored at -10°C or less, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared weekly and stored at 4°C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.7 Surrogate standards - The recommended surrogates are phenol-d₆, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d₅, 2-fluorobiphenyl, and p-terphenyl-d₁₄. See Method 3500 for instructions on preparing the surrogate solutions.

5.7.1 Surrogate Standard Check: Determine what the appropriate concentration should be for the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

NOTE: Method 3561 (SFE Extraction of PAHs) recommends the use of bromobenzene and p-quaterphenyl to better cover the range of PAHs listed in the method.

5.7.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute surrogate solution may be necessary.

5.8 Matrix spike and laboratory control standards - See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS).

5.8.1 Matrix Spike Check: Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery. It is recommended that this check be done whenever a new matrix spiking solution is prepared.

5.8.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute matrix and LCS spiking solution may be necessary.

5.8.3 Some projects may require the spiking of the specific compounds of interest, since the spiking compounds listed in Method 3500 would not be representative of the compounds of interest required for the project. When this occurs, the matrix and LCS spiking

standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - All solvents should be pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Store the sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis.

<u>Matrix</u>	<u>Methods</u>
Air	3542
Water	3510, 3520, 3535
Soil/sediment	3540, 3541, 3545, 3550, 3560, 3561
Waste	3540, 3541, 3545, 3550, 3560, 3561, 3580

7.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10- μ L syringe may be appropriate. The detection limit is very high (approximately 10,000 μ g/L). Therefore, it is only permitted where concentrations in excess of 10,000 μ g/L are expected.

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

<u>Analytes of interest</u>	<u>Methods</u>
Aniline & aniline derivatives	3620
Phenols	3630, 3640, 8041 ^a
Phthalate esters	3610, 3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides & PCBs	3610, 3620, 3630, 3660, 3665
Nitroaromatics and cyclic ketones	3620, 3640
Polynuclear aromatic hydrocarbons	3611, 3630, 3640
Haloethers	3620, 3640
Chlorinated hydrocarbons	3620, 3640
Organophosphorus pesticides	3620

<u>Analytes of interest</u>	<u>Methods</u>
Petroleum waste	3611, 3650
All base, neutral, and acid priority pollutants	3640

^a Method 8041 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered on GC/FID.

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

Mass range:	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	40°C, hold for 4 minutes
Temperature program:	40-270°C at 10°C/min
Final temperature:	270°C, hold until benzo[g,h,i]perylene elutes
Injector temperature:	250-300°C
Transfer line temperature:	250-300°C
Source temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Injection volume:	1-2 µL
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

7.3.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach has been shown to be useful: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

7.3.1.2 Use the DFTPP mass intensity criteria in Table 3 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

7.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD

should not exceed 20%. (See Sec. 8.0 of Method 8081 for the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

7.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column. The use of a guard column (Sec. 4.1.6) between the injection port and the analytical column may help prolong analytical column performance.

7.3.2 The internal standards selected in Sec. 5.4 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d₄, use 152 m/z for quantitation).

7.3.3 Analyze 1-2 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 5.6 and Method 8000). The injection volume must be the same for all standards and sample extracts. Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

Calculate response factors (RFs) for each target analyte relative to one of the internal standards as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

- A_s = Peak area (or height) of the analyte or surrogate.
- A_{is} = Peak area (or height) of the internal standard.
- C_s = Concentration of the analyte or surrogate, in µg/L.
- C_{is} = Concentration of the internal standard, in µg/L.

7.3.4 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

7.3.4.3 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Section 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual CCC (see Table 4) must be less than or equal to 30%.

$$\text{mean RF} = \overline{\text{RF}} = \frac{\sum_{i=1}^n \text{RF}_i}{n} \qquad \text{SD} = \sqrt{\frac{\sum_{i=1}^n (\text{RF}_i - \overline{\text{RF}})^2}{n-1}}$$

$$\text{RSD} = \frac{\text{SD}}{\overline{\text{RF}}} \times 100$$

7.3.5.3 If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Sec. 7.3.

7.3.5.4 If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000.

7.3.6 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

7.3.7 Linearity of target analytes - If the RSD of any target analytes is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation (Sec. 7.6.2).

7.3.7.1 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 in Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 designates a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.7.2 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000B for method blank performance criteria.

7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Table 4 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference for each CCC is less than or equal to 20%, then the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project,

analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

- (1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 If the RSD of a compound's response factor is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data (Sec. 7.3.5). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.7.5 Quantitation of multicomponent compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/ μ L in the concentrated sample extract.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed, a septum is changed), see the guidance in Sec 8.2 of Method 8000 regarding whether recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations ranging from 5 to 1,300 µg/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7. These values are presented as guidance only and are not intended as absolute acceptance criteria. Laboratories should generate their own acceptance criteria for capillary column method performance. (See Method 8000.)

9.2 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of GC performance. These recoveries (using Method 3510 extraction) are presented in Table 8.

9.3 Method performance data (using Method 3541 Automated Soxhlet extraction) are presented in Table 9. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately 1 hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three determinations were performed and each extract was analyzed by gas chromatography/ mass spectrometry following Method 8270. The low recovery of the more volatile compounds is probably due to volatilization losses during equilibration. These data are listed in Table 10 and were taken from Reference 7.

9.4 Surrogate precision and accuracy data are presented in Table 11 from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by Method 8270.

9.5 Single laboratory precision and bias data (using Method 3545 Accelerated Solvent Extraction) for semivolatile organic compounds are presented in Table 12. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (250, 2500, and 12,500 µg/kg) on three types of soil (clay, loam and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by Perstorp Environmental Soxtec™ (automated Soxhlet). The data presented in Table 12 represents seven replicate extractions and analyses for each individual sample and were taken from reference 9. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam 98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low-101.2%, mid-97.2% and high-99.2%.

9.6 Single laboratory precision and bias data (using Method 3561 SFE Extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (one, EC-1, a lake sediment from Environment Canada and the other, HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally-contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions range from 85 to 148% (overall average of 100%) based on the certified value (or a Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions range from 73 to 133% (overall average of 92%) based on the certified value for the marine sediment. The data are found in Tables 13 and 14 and were taken from Reference 10.

9.7 Single laboratory precision and accuracy data (using Method 3561 SFE Extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recoveries from four replicate extractions range from 60 to 122% (overall average of 89%) based on the certified value. Following are the instrument conditions that were utilized to extract a 3.4 g sample: Pressure - 300 atm; Time - 60 min.; Extraction fluid - CO₂; Modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80°C; Restrictor temperature - 120°C; and, Trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 15 and were taken from Reference 11.

10.0 REFERENCES

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TABLE 1
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75 ^a	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d ₄ (IS)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	44,45,74
Thiophenol (Benzenethiol)	7.42	110	66,109,84
Methyl methanesulfonate	7.48	80	79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	79,95,109,140
Ethyl methanesulfonate	9.62	79	109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d ₈ (IS)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	155,127,81,109
Diethyl sulfate	11.37	139	45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	108,77,79,90
Hexachloropropene	12.45	213	211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosopyrrolidine	12.65	100	41,42,68,69
Acetophenone	12.67	105	71,51,120
4-Methylphenol	12.82	107	108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	107,77,51,79
3-Methylphenol	12.93	107	108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,55,56,41
1,4-Phenylenediamine	13.62	108	80,53,54,52
1-Chloronaphthalene	13.65 ^a	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	76,50,148
o-Anisidine	15.00	108	80,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d ₁₀ (IS)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	164,126,98,63
4-Chloroaniline	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00 ^a	143	115,116
1,4-Naphthoquinone	16.23	158	104,102,76,50,130
p-Cresidine	16.45	122	94,137,77,93
Dichlorovos	16.48	109	185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	135,134,91,77
N-Nitrosodi-n-butylamine	16.73	84	57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethyl phosphoramidate	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54 ^a	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro- <i>o</i> -toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d ₁₀ (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro- <i>o</i> -anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di- <i>n</i> -butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosaffrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramidate	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
α,α -Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d ₁₂ (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyrilene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxypfos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate ^b	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d ₁₂ (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
α -BHC	--	183	181,109
β -BHC	--	181	183,109
δ -BHC	--	183	181,109
γ -BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d ₅ (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d ₆ (surr)	--	99	42,71
Terphenyl-d ₁₄ (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard

surr = surrogate

^aEstimated retention times

^bSubstitute for the non-specific mixture, tricresyl phosphate

TABLE 2

ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

Compound	Estimated Quantitation Limits ^a	
	Ground water µg/L	Low Soil/Sediment ^b µg/kg
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-Bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits ^a	
	Ground water µg/L	Low Soil/Sediment ^b µg/kg
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butyl phthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phthalate	10	660
Diethylstilbestrol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
a,a-Dimethylphenethylamine	ND	ND
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits ^a	
	Ground water µg/L	Low Soil/Sediment ^b µg/kg
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl) phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethylphosphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydride	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
4,4'-Methylenebis(2-chloroaniline)	NA	ND
Methyl methanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits ^a	
	Ground water µg/L	Low Soil/Sediment ^b µg/kg
4-Methylphenol	10	660
Mevinphos	10	ND
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro- <i>o</i> -anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro- <i>o</i> -toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodi- <i>n</i> -butylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di- <i>n</i> -propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND
Octamethyl pyrophosphoramidate	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits ^a	
	Ground water µg/L	Low Soil/Sediment ^b µg/kg
Phthalic anhydride	100	ND
2-Picoline	ND	ND
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND
O,O,O-Triethyl phosphorothioate	NT	ND

^a Sample EQLs are highly matrix-dependent. The EQLs listed here are provided for guidance and may not always be achievable.

^b EQLs listed for soil/sediment are based on wet weight. Normally, data are reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined

NA = Not Applicable

NT = Not Tested

Other Matrices

Factor^c

High-concentration soil and sludges by ultrasonic extractor

7.5

Non-water miscible waste

75

^cEQL = (EQL for Low Soil/Sediment given above in Table 2) x (Factor)

TABLE 3
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA^{a,b}

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

^a Data taken from Reference 3.

^b Alternate tuning criteria may be used, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 4
CALIBRATION CHECK COMPOUNDS (CCC)

<u>Base/Neutral Fraction</u>	<u>Acid Fraction</u>
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES
ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethylphenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d ₈ (surr)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d ₈ (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

TABLE 5
(continued)

Phenanthrene-d ₁₀	Chrysene-d ₁₂	Perylene-d ₁₂
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethylaminoazobenzene	
Hexachlorobenzene	Pyrene	
N-Nitrosodiphenylamine	Terphenyl-d ₁₄ (surr)	
Pentachlorophenol	7,12-Dimethylbenz(a)anthracene	
Pentachloronitrobenzene	Di-n-octyl phthalate	
Phenacetin	Indeno(1,2,3-cd)pyrene	
Phenanthrene	3-Methylcholanthrene	
Pronamide		

(surr) = surrogate

TABLE 6
MULTILABORATORY PERFORMANCE DATA^a

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for \bar{x} (µg/L)	Range p, p _s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192

TABLE 6
(continued)

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for \bar{x} (µg/L)	Range p, p _s (%)
Heptachlor epoxide	100	54.7	70.9-109.4	26-155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L

\bar{x} = Average recovery for four recovery measurements, in µg/L

p, p_s = Measured percent recovery

D = Detected; result must be greater than zero

^a Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION^a

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s _r ' (µg/L)	Overall precision, S' (µg/L)
Acenaphthene	0.96C+0.19	0.15 \bar{x} -0.12	0.21 \bar{x} -0.67
Acenaphthylene	0.89C+0.74	0.24 \bar{x} -1.06	0.26 \bar{x} -0.54
Aldrin	0.78C+1.66	0.27 \bar{x} -1.28	0.43 \bar{x} +1.13
Anthracene	0.80C+0.68	0.21 \bar{x} -0.32	0.27 \bar{x} -0.64
Benz(a)anthracene	0.88C-0.60	0.15 \bar{x} +0.93	0.26 \bar{x} -0.21
Benzo(b)fluoranthene	0.93C-1.80	0.22 \bar{x} +0.43	0.29 \bar{x} +0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19 \bar{x} +1.03	0.35 \bar{x} +0.40
Benzo(a)pyrene	0.90C-0.13	0.22 \bar{x} +0.48	0.32 \bar{x} +1.35
Benzo(g,h,i)perylene	0.98C-0.86	0.29 \bar{x} +2.40	0.51 \bar{x} -0.44
Benzyl butyl phthalate	0.66C-1.68	0.18 \bar{x} +0.94	0.53 \bar{x} +0.92
β-BHC	0.87C-0.94	0.20 \bar{x} -0.58	0.30 \bar{x} +1.94
δ-BHC	0.29C-1.09	0.34 \bar{x} +0.86	0.93 \bar{x} -0.17
Bis(2-chloroethyl) ether	0.86C-1.54	0.35 \bar{x} -0.99	0.35 \bar{x} +0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16 \bar{x} +1.34	0.26 \bar{x} +2.01
Bis(2-chloroisopropyl) ether	1.03C-2.31	0.24 \bar{x} +0.28	0.25 \bar{x} +1.04
Bis(2-ethylhexyl) phthalate	0.84C-1.18	0.26 \bar{x} +0.73	0.36 \bar{x} +0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13 \bar{x} +0.66	0.16 \bar{x} +0.66
2-Chloronaphthalene	0.89C+0.01	0.07 \bar{x} +0.52	0.13 \bar{x} +0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20 \bar{x} -0.94	0.30 \bar{x} -0.46
Chrysene	0.93C-1.00	0.28 \bar{x} +0.13	0.33 \bar{x} -0.09
4,4'-DDD	0.56C-0.40	0.29 \bar{x} -0.32	0.66 \bar{x} -0.96
4,4'-DDE	0.70C-0.54	0.26 \bar{x} -1.17	0.39 \bar{x} -1.04
4,4'-DDT	0.79C-3.28	0.42 \bar{x} +0.19	0.65 \bar{x} -0.58
Dibenzo(a,h)anthracene	0.88C+4.72	0.30 \bar{x} +8.51	0.59 \bar{x} +0.25
Di-n-butyl phthalate	0.59C+0.71	0.13 \bar{x} +1.16	0.39 \bar{x} +0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20 \bar{x} +0.47	0.24 \bar{x} +0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25 \bar{x} +0.68	0.41 \bar{x} +0.11
1,4-Dichlorobenzene	0.73C-1.47	0.24 \bar{x} +0.23	0.29 \bar{x} +0.36
3,3'-Dichlorobenzidine	1.23C-12.65	0.28 \bar{x} +7.33	0.47 \bar{x} +3.45
Dieldrin	0.82C-0.16	0.20 \bar{x} -0.16	0.26 \bar{x} -0.07
Diethyl phthalate	0.43C+1.00	0.28 \bar{x} +1.44	0.52 \bar{x} +0.22
Dimethyl phthalate	0.20C+1.03	0.54 \bar{x} +0.19	1.05 \bar{x} -0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12 \bar{x} +1.06	0.21 \bar{x} +1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14 \bar{x} +1.26	0.19 \bar{x} +0.35
Di-n-octyl phthalate	0.76C-0.79	0.21 \bar{x} +1.19	0.37 \bar{x} +1.19
Endosulfan sulfate	0.39C+0.41	0.12 \bar{x} +2.47	0.63 \bar{x} -1.03
Endrin aldehyde	0.76C-3.86	0.18 \bar{x} +3.91	0.73 \bar{x} -0.62
Fluoranthene	0.81C+1.10	0.22 \bar{x} -0.73	0.28 \bar{x} -0.60
Fluorene	0.90C-0.00	0.12 \bar{x} +0.26	0.13 \bar{x} +0.61
Heptachlor	0.87C-2.97	0.24 \bar{x} -0.56	0.50 \bar{x} -0.23
Heptachlor epoxide	0.92C-1.87	0.33 \bar{x} -0.46	0.28 \bar{x} +0.64

TABLE 7
(continued)

Compound	Accuracy, as recovery, x' ($\mu\text{g/L}$)	Single analyst precision, s_r' ($\mu\text{g/L}$)	Overall precision, S' ($\mu\text{g/L}$)
Hexachlorobenzene	0.74C+0.66	0.18 \bar{x} -0.10	0.43 \bar{x} -0.52
Hexachlorobutadiene	0.71C-1.01	0.19 \bar{x} +0.92	0.26 \bar{x} +0.49
Hexachloroethane	0.73C-0.83	0.17 \bar{x} +0.67	0.17 \bar{x} +0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29 \bar{x} +1.46	0.50 \bar{x} -0.44
Isophorone	1.12C+1.41	0.27 \bar{x} +0.77	0.33 \bar{x} +0.26
Naphthalene	0.76C+1.58	0.21 \bar{x} -0.41	0.30 \bar{x} -0.68
Nitrobenzene	1.09C-3.05	0.19 \bar{x} +0.92	0.27 \bar{x} +0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27 \bar{x} +0.68	0.44 \bar{x} +0.47
Aroclor 1260	0.81C-10.86	0.35 \bar{x} +3.61	0.43 \bar{x} +1.82
Phenanthrene	0.87C+0.06	0.12 \bar{x} +0.57	0.15 \bar{x} +0.25
Pyrene	0.84C-0.16	0.16 \bar{x} +0.06	0.15 \bar{x} +0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15 \bar{x} +0.85	0.21 \bar{x} +0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23 \bar{x} +0.75	0.29 \bar{x} +1.31
2-Chlorophenol	0.78C+0.29	0.18 \bar{x} +1.46	0.28 \bar{x} +0.97
2,4-Dichlorophenol	0.87C-0.13	0.15 \bar{x} +1.25	0.21 \bar{x} +1.28
2,4-Dimethylphenol	0.71C+4.41	0.16 \bar{x} +1.21	0.22 \bar{x} +1.31
2,4-Dinitrophenol	0.81C-18.04	0.38 \bar{x} +2.36	0.42 \bar{x} +26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10 \bar{x} +42.29	0.26 \bar{x} +23.10
2-Nitrophenol	0.07C-1.15	0.16 \bar{x} +1.94	0.27 \bar{x} +2.60
4-Nitrophenol	0.61C-1.22	0.38 \bar{x} +2.57	0.44 \bar{x} +3.24
Pentachlorophenol	0.93C+1.99	0.24 \bar{x} +3.03	0.30 \bar{x} +4.33
Phenol	0.43C+1.26	0.26 \bar{x} +0.73	0.35 \bar{x} +0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16 \bar{x} +2.22	0.22 \bar{x} +1.81

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in $\mu\text{g/L}$.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of \bar{x} , in $\mu\text{g/L}$.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of \bar{x} , in $\mu\text{g/L}$.

C = True value for the concentration, in $\mu\text{g/L}$.

\bar{x} = Average recovery found for measurements of samples containing a concentration of C, in $\mu\text{g/L}$.

^a Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 9

MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE ORGANICS FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET EXTRACTION (METHOD 3541) WITH HEXANE-ACETONE (1:1)^a

Compound	Clay Soil		Topsoil	
	Mean Recovery	RSD	Mean Recovery	RSD
1,3-Dichlorobenzene	0	--	0	--
1,2-Dichlorobenzene	0	--	0	--
Nitrobenzene	0	--	0	--
Benzal chloride	0	--	0	--
Benzotrifluoride	0	--	0	--
4-Chloro-2-nitrotoluene	0	--	0	--
Hexachlorocyclopentadiene	4.1	15	7.8	23
2,4-Dichloronitrobenzene	35.2	7.6	21.2	15
3,4-Dichloronitrobenzene	34.9	15	20.4	11
Pentachlorobenzene	13.7	7.3	14.8	13
2,3,4,5-Tetrachloronitrobenzene	55.9	6.7	50.4	6.0
Benefin	62.6	4.8	62.7	2.9
alpha-BHC	58.2	7.3	54.8	4.8
Hexachlorobenzene	26.9	13	25.1	5.7
delta-BHC	95.8	4.6	99.2	1.3
Heptachlor	46.9	9.2	49.1	6.3
Aldrin	97.7	12	102	7.4
Isopropalin	102	4.3	105	2.3
Heptachlor epoxide	90.4	4.4	93.6	2.4
trans-Chlordane	90.1	4.5	95.0	2.3
Endosulfan I	96.3	4.4	101	2.2
Dieldrin	129	4.7	104	1.9
2,5-Dichlorophenyl-4-nitrophenyl ether	110	4.1	112	2.1
Endrin	102	4.5	106	3.7
Endosulfan II	104	4.1	105	0.4
p,p'-DDT	134	2.1	111	2.0
2,3,6-Trichlorophenyl-4'-nitrophenyl ether	110	4.8	110	2.8
2,3,4-Trichlorophenyl-4'-nitrophenyl ether	112	4.4	112	3.3
Mirex	104	5.3	108	2.2

^a The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4-nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.

TABLE 10

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION
OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY
AUTOMATED SOXHLET (METHOD 3541)^a

Compound	Mean Recovery	RSD
Phenol	47.8	5.6
Bis(2-chloroethyl)ether	25.4	13
2-Chlorophenol	42.7	4.3
Benzyl alcohol	55.9	7.2
2-Methylphenol	17.6	6.6
Bis(2-chloroisopropyl)ether	15.0	15
4-Methylphenol	23.4	6.7
N-Nitroso-di-n-propylamine	41.4	6.2
Nitrobenzene	28.2	7.7
Isophorone	56.1	4.2
2-Nitrophenol	36.0	6.5
2,4-Dimethylphenol	50.1	5.7
Benzoic acid	40.6	7.7
Bis(2-chloroethoxy)methane	44.1	3.0
2,4-Dichlorophenol	55.6	4.6
1,2,4-Trichlorobenzene	18.1	31
Naphthalene	26.2	15
4-Chloroaniline	55.7	12
4-Chloro-3-methylphenol	65.1	5.1
2-Methylnaphthalene	47.0	8.6
Hexachlorocyclopentadiene	19.3	19
2,4,6-Trichlorophenol	70.2	6.3
2,4,5-Trichlorophenol	26.8	2.9
2-Chloronaphthalene	61.2	6.0
2-Nitroaniline	73.8	6.0
Dimethyl phthalate	74.6	5.2
Acenaphthylene	71.6	5.7
3-Nitroaniline	77.6	5.3
Acenaphthene	79.2	4.0
2,4-Dinitrophenol	91.9	8.9
4-Nitrophenol	62.9	16
Dibenzofuran	82.1	5.9
2,4-Dinitrotoluene	84.2	5.4
2,6-Dinitrotoluene	68.3	5.8
Diethyl phthalate	74.9	5.4
4-Chlorophenyl-phenyl ether	67.2	3.2
Fluorene	82.1	3.4
4-Nitroaniline	79.0	7.9

TABLE 10
(continued)

Compound	Mean Recovery	RSD
4,6-Dinitro-2-methylphenol	63.4	6.8
N-Nitrosodiphenylamine	77.0	3.4
4-Bromophenyl-phenyl ether	62.4	3.0
Hexachlorobenzene	72.6	3.7
Pentachlorophenol	62.7	6.1
Phenanthrene	83.9	5.4
Anthracene	96.3	3.9
Di-n-butyl phthalate	78.3	40
Fluoranthene	87.7	6.9
Pyrene	102	0.8
Butyl benzyl phthalate	66.3	5.2
3,3'-Dichlorobenzidine	25.2	11
Benzo(a)anthracene	73.4	3.8
Bis(2-ethylhexyl) phthalate	77.2	4.8
Chrysene	76.2	4.4
Di-n-octyl phthalate	83.1	4.8
Benzo(b)fluoranthene	82.7	5.0
Benzo(k)fluoranthene	71.7	4.1
Benzo(a)pyrene	71.7	4.1
Indeno(1,2,3-cd)pyrene	72.2	4.3
Dibenzo(a,h)anthracene	66.7	6.3
Benzo(g,h,i)perylene	63.9	8.0
1,2-Dichlorobenzene	0	--
1,3-Dichlorobenzene	0	--
1,4-Dichlorobenzene	0	--
Hexachloroethane	0	--
Hexachlorobutadiene	0	--

^a Number of determinations was three. The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil; the spike concentration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spiking.

Data taken from Reference 7.

TABLE 12

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Phenol	93.3	78.7	135.9	73.9	82.8	124.6	108.8	130.6	89.7	102.0
Bis(2-chloroethyl) ether	102.1	85.1	109.1	96.0	88.0	103.6	122.3	119.9	90.8	101.9
2-Chlorophenol	100.8	82.6	115.0	93.8	88.9	111.1	115.0	115.3	91.9	101.6
1,3-Dichlorobenzene	127.7	129.7	110.0	*364.2	129.9	119.0	*241.3	*163.7	107.1	120.6
1,4-Dichlorobenzene	127.9	127.0	110.5	*365.9	127.8	116.4	*309.6	*164.1	105.8	119.2
1,2-Dichlorobenzene	116.8	115.8	101.3	*159.2	113.4	105.5	*189.3	134.0	100.4	112.5
2-Methylphenol	98.9	82.1	119.7	87.6	89.4	111.0	133.2	128.0	92.1	104.7
Bis(2-chloroisopropyl)ether	109.4	71.5	108.0	81.8	81.0	88.6	118.1	148.3	94.8	100.2
o-Toluidine	100.0	89.7	117.2	100.0	*152.5	120.3	100.0	*199.5	102.7	110.3
N-Nitroso-di-n-propylamine	103.0	79.1	107.7	83.9	88.1	96.2	109.9	123.3	91.4	98.1
Hexachloroethane	97.1	125.1	111.0	*245.4	117.1	128.1	*566.7	147.9	103.7	118.6
Nitrobenzene	104.8	82.4	106.6	86.8	84.6	101.7	119.7	122.1	93.3	100.2
Isophorone	100.0	86.4	98.2	87.1	87.5	109.7	135.5	118.4	92.7	101.7
2,4-Dimethylphenol	100.0	104.5	140.0	100.0	114.4	123.1	100.0	*180.6	96.3	109.8
2-Nitrophenol	80.7	80.5	107.9	91.4	86.7	103.2	122.1	107.1	87.0	96.3
Bis(chloroethoxy)methane	94.4	80.6	94.7	86.5	84.4	99.6	130.6	110.7	93.2	97.2
2,4-Dichlorophenol	88.9	87.8	111.4	85.9	87.6	103.5	123.3	107.0	92.1	98.6
1,2,4-Trichlorobenzene	98.0	97.8	98.8	123.0	93.7	94.5	137.0	99.4	95.3	104.2
Naphthalene	101.7	97.2	123.6	113.2	102.9	129.5	*174.5	114.0	89.8	106.1
4-Chloroaniline	100.0	*150.2	*162.4	100.0	125.5	*263.6	100.0	*250.8	114.9	108.1
Hexachlorobutadiene	101.1	98.7	102.2	124.1	90.3	98.0	134.9	96.1	96.8	104.7
4-Chloro-3-methylphenol	90.4	80.2	114.7	79.0	85.2	109.8	131.6	116.2	90.1	99.7
2-Methylnaphthalene	93.2	89.9	94.6	104.1	92.2	105.9	146.2	99.1	93.3	102.1
Hexachlorocyclopentadiene	100.0	100.0	0.0	100.0	100.0	6.8	100.0	100.0	*238.3	75.8
2,4,6-Trichlorophenol	94.6	90.0	112.0	84.2	91.2	103.6	101.6	95.9	89.8	95.9
2,4,5-Trichlorophenol	84.4	91.9	109.6	96.1	80.7	103.6	108.9	83.9	87.9	94.1
2-Chloronaphthalene	100.0	91.3	93.6	97.6	93.4	98.3	106.8	93.0	92.0	96.2
2-Nitroaniline	90.0	83.4	97.4	71.3	88.4	89.9	112.1	113.3	87.7	92.6
2,6-Dinitrotoluene	83.1	90.6	91.6	86.4	90.6	90.3	104.3	84.7	90.9	90.3
Acenaphthylene	104.9	95.9	100.5	99.0	97.9	108.8	118.5	97.8	92.0	101.7
3-Nitroaniline	*224.0	115.6	97.6	100.0	111.8	107.8	0.0	111.7	99.0	92.9
Acenaphthene	102.1	92.6	97.6	97.2	96.9	104.4	114.2	92.0	89.0	98.4
4-Nitrophenol	0.0	93.2	121.5	18.1	87.1	116.6	69.1	90.5	84.5	75.6
2,4-Dinitrotoluene	73.9	91.9	100.2	84.7	93.8	98.9	100.9	84.3	87.3	90.7

TABLE 12 (cont.)

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Dibenzofuran	89.5	91.7	109.3	98.5	92.2	111.4	113.8	92.7	90.4	98.8
4-Chlorophenyl phenyl ether	83.0	94.5	98.7	95.7	94.3	94.2	111.4	87.7	90.3	94.4
Fluorene	85.2	94.9	89.2	102.0	95.5	93.8	121.3	85.7	90.9	95.4
4-Nitroaniline	77.8	114.8	94.5	129.6	103.6	95.4	*154.1	89.3	87.5	99.1
N-Nitrosodiphenylamine	82.6	96.7	93.8	92.9	93.4	116.4	97.5	110.9	86.7	96.8
4-Bromophenyl phenyl ether	85.6	92.9	92.8	91.1	107.6	89.4	118.0	97.5	87.1	95.8
Hexachlorobenzene	95.4	91.7	92.3	95.4	93.6	83.7	106.8	94.3	90.0	93.7
Pentachlorophenol	68.2	85.9	107.7	53.2	89.8	88.1	96.6	59.8	81.3	81.2
Phenanthrene	92.1	93.7	93.3	100.0	97.8	113.3	124.4	101.0	89.9	100.6
Anthracene	101.6	95.0	93.5	92.5	101.8	118.4	123.0	94.5	90.6	101.2
Carbazole	94.4	99.3	96.6	105.5	96.7	111.4	115.7	83.2	88.9	99.1
Fluoranthene	109.9	101.4	94.3	111.6	96.6	109.6	123.2	85.4	92.7	102.7
Pyrene	106.5	105.8	107.6	116.7	90.7	127.5	103.4	95.5	93.2	105.2
3,3'-Dichlorobenzidine	100.0	*492.3	131.4	100.0	*217.6	*167.6	100.0	*748.8	100.0	116.5
Benzo(a)anthracene	98.1	107.0	98.4	119.3	98.6	104.0	105.0	93.4	89.3	101.5
Chrysene	100.0	108.5	100.2	116.8	93.0	117.0	106.7	93.6	90.2	102.9
Benzo(b)fluoranthene	106.6	109.9	75.6	121.7	100.7	93.9	106.9	81.9	93.6	99.0
Benzo(k)fluoranthene	102.4	105.2	88.4	125.5	99.4	95.1	144.7	89.2	78.1	103.1
Benzo(a)pyrene	107.9	105.5	80.8	122.3	97.7	104.6	101.7	86.2	92.0	99.9
Indeno(1,2,3-cd)pyrene	95.1	105.7	93.8	126.0	105.2	90.4	133.6	82.6	91.9	102.7
Dibenz(a,h)anthracene	85.0	102.6	82.0	118.8	100.7	91.9	142.3	71.0	93.1	98.6
Benzo(g,h,i)perylene	98.0	0.0	81.2	0.0	33.6	78.6	128.7	83.0	94.2	66.4
Average	95.1	94.3	101.0	95.5	96.5	104.1	113.0	100.9	92.5	

* Values greater than 150% were not used to determine the averages, but the 0% values were used.

TABLE 13

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value ^a (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	(27.9) ^b	41.3 ± 3.6	(148)	8.7
Acenaphthylene	(0.8)	0.9 ± 0.1	(112)	11.1
Acenaphthene	(0.2)	0.2 ± 0.01	(100)	0.05
Fluorene	(15.3)	15.6 ± 1.8	(102)	11.5
Phenanthrene	15.8 ± 1.2	16.1 ± 1.8	102	11.2
Anthracene	(1.3)	1.1 ± 0.2	(88)	18.2
Fluoranthene	23.2 ± 2.0	24.1 ± 2.1	104	8.7
Pyrene	16.7 ± 2.0	17.2 ± 1.9	103	11.0
Benz(a)anthracene	8.7 ± 0.8	8.8 ± 1.0	101	11.4
Chrysene	(9.2)	7.9 ± 0.9	(86)	11.4
Benzo(b)fluoranthene	7.9 ± 0.9	8.5 ± 1.1	108	12.9
Benzo(k)fluoranthene	4.4 ± 0.5	4.1 ± 0.5	91	12.2
Benzo(a)pyrene	5.3 ± 0.7	5.1 ± 0.6	96	11.8
Indeno(1,2,3-cd)pyrene	5.7 ± 0.6	5.2 ± 0.6	91	11.5
Benzo(g,h,i)perylene	4.9 ± 0.7	4.3 ± 0.5	88	11.6
Dibenz(a,h)anthracene	(1.3)	1.1 ± 0.2	(85)	18.2

^a Relative standard deviations for the SFE values are based on six replicate extractions.

^b Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 14

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHS FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value ^a (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	9.0 ± 0.7	7.4 ± 0.6	82	8.1
Acenaphthylene	0.3 ± 0.1	0.4 ± 0.1	133	25.0
Acenaphthene	4.5 ± 1.5	3.3 ± 0.3	73	9.0
Fluorene	13.6 ± 3.1	10.4 ± 1.3	77	12.5
Phenanthrene	85.0 ± 20.0	86.2 ± 9.5	101	11.0
Anthracene	13.4 ± 0.5	12.1 ± 1.5	90	12.4
Fluoranthene	60.0 ± 9.0	54.0 ± 6.1	90	11.3
Pyrene	39.0 ± 9.0	32.7 ± 3.7	84	11.3
Benzo(a)anthracene	14.6 ± 2.0	12.1 ± 1.3	83	10.7
Chrysene	14.1 ± 2.0	12.0 ± 1.3	85	10.8
Benzo(b)fluoranthene	7.7 ± 1.2	8.4 ± 0.9	109	10.7
Benzo(k)fluoranthene	2.8 ± 2.0	3.2 ± 0.5	114	15.6
Benzo(a)pyrene	7.4 ± 3.6	6.6 ± 0.8	89	12.1
Indeno(1,2,3-cd)pyrene	5.0 ± 2.0	4.5 ± 0.6	90	13.3
Benzo(g,h,i)perylene	5.4 ± 1.3	4.4 ± 0.6	82	13.6
Dibenz(a,h)anthracene	1.3 ± 0.5	1.1 ± 0.3	85	27.3

^a Relative standard deviations for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 15

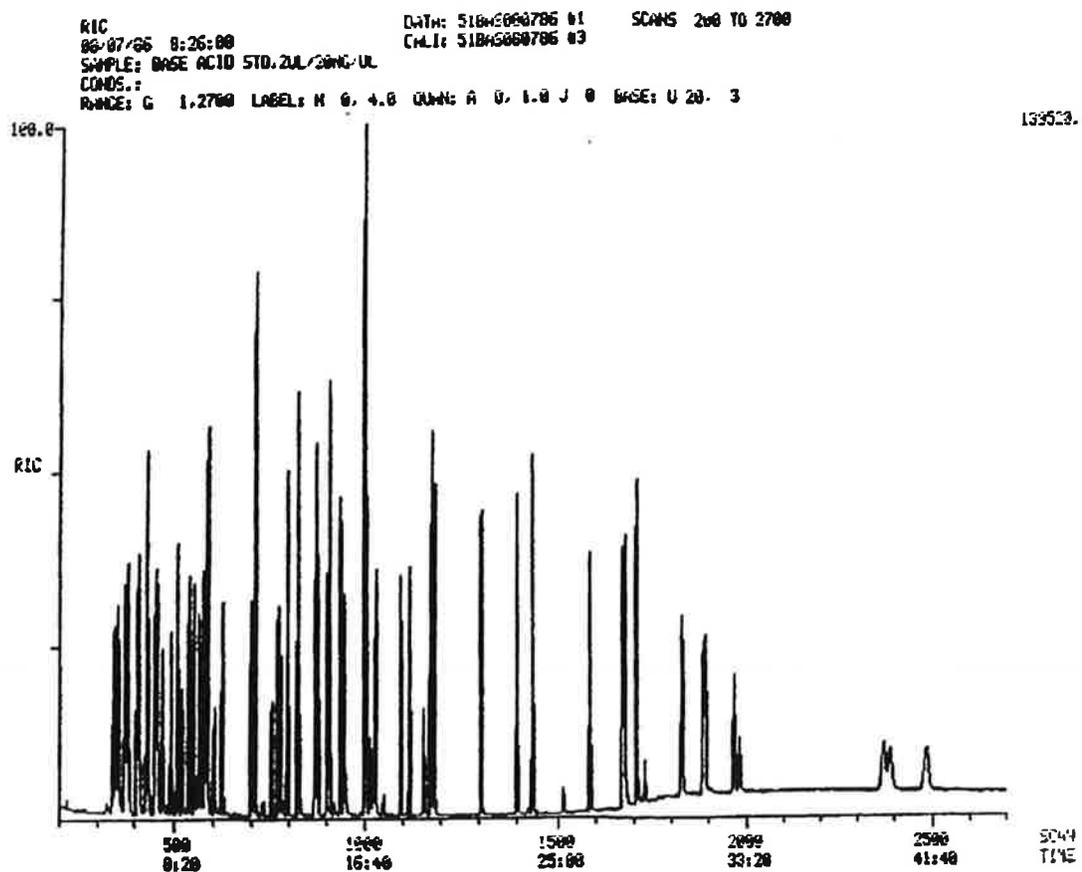
SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs
FROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561
(SFE - LIQUID TRAP)

Compound	Certified Value (mg/kg)		SFE Value ^a (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	32.4	± 8.2	29.55	91	10.5
2-Methylnaphthalene	62.1	± 11.5	76.13	122	2.0
Acenaphthene	632	± 105	577.28	91	2.9
Dibenzofuran	307	± 49	302.25	98	4.1
Fluorene	492	± 78	427.15	87	3.0
Phenanthrene	1618	± 340	1278.03	79	3.4
Anthracene	422	± 49	400.80	95	2.6
Fluoranthene	1280	± 220	1019.13	80	4.5
Pyrene	1033	± 285	911.82	88	3.1
Benz(a)anthracene	252	± 38	225.50	89	4.8
Chrysene	297	± 26	283.00	95	3.8
Benzo(b)fluoranthene + Benzo(k)fluoranthene	153	± 22	130.88	86	10.7
Benzo(a)pyrene	97.2	± 17.1	58.28	60	6.5

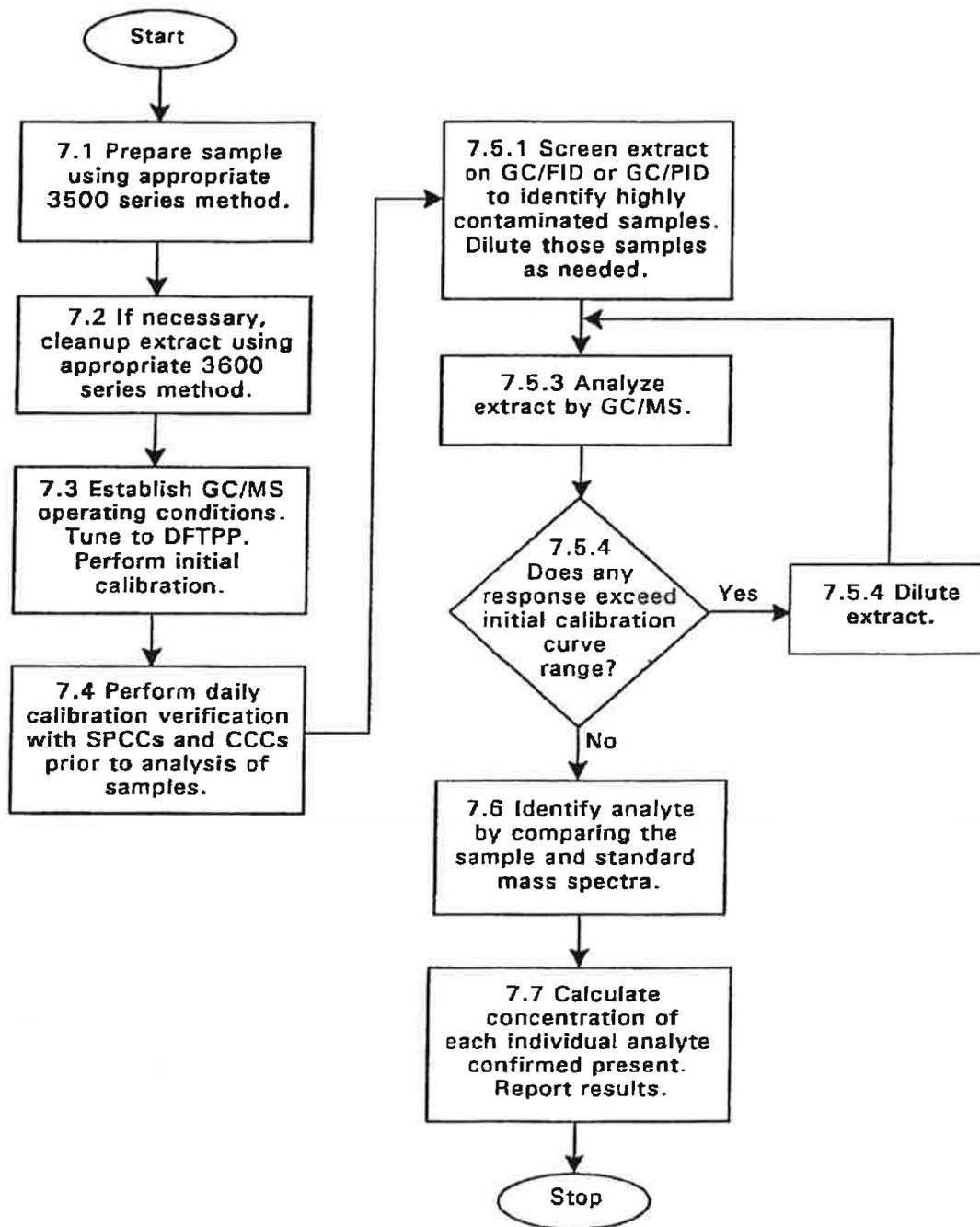
^a Relative standard deviations for the SFE values are based on four replicate extractions.

Data are taken from Reference 11.

FIGURE 1
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD



METHOD 8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS
SPECTROMETRY (GC/MS)



ATTACHMENT 6

EPA METHOD 8082A

METHOD 8082A

POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be followed by individuals formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method may be used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors or as individual PCB congeners in extracts from solid, tissue, and aqueous matrices, using open-tubular, capillary columns with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). The Aroclors and PCB congeners listed below have been determined by this method, using either a single- or dual column analysis system, and this method may be appropriate for additional congeners and Aroclors (see Sec. 1.4). The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

Compound	CAS Registry No. ^a	IUPAC #
Aroclor 1016	12674-11-2	-
Aroclor 1221	11104-28-2	-
Aroclor 1232	11141-16-5	-
Aroclor 1242	53469-21-9	-
Aroclor 1248	12672-29-6	-
Aroclor 1254	11097-69-1	-
Aroclor 1260	11096-82-5	-
2-Chlorobiphenyl	2051-60-7	1
2,3-Dichlorobiphenyl	16605-91-7	5
2,2',5-Trichlorobiphenyl	37680-65-2	18
2,4',5-Trichlorobiphenyl	16606-02-3	31
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141

Compound	CAS Registry No. ^a	IUPAC #
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206

^aChemical Abstract Service Registry No.

1.2 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns compared to those of Aroclor standards.

1.3 The seven Aroclors listed in Sec. 1.1 are those that are commonly specified in EPA regulations. The quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment. Therefore, this method provides procedures for the determination of a selected group of the 209 possible PCB congeners, as another means to measure the concentrations of weathered Aroclors. The 19 PCB congeners listed above have been tested by this method and were chosen for testing because many of them represent congeners specific to the common Aroclor formulations (see Table 6). These 19 PCB congeners do not represent the co-planar PCBs or the other PCBs of greatest toxicological significance. The analytical procedures for these 19 congeners may be appropriate for the analysis of other congeners not specifically included in this method and may be used as a template for the development of such a procedure. However, all 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either document the resolution of the congeners in question, or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

1.4 The PCB congener approach potentially affords greater quantitative accuracy when PCBs are known to be present. As a result, this method may be used to determine Aroclors, some PCB congeners, or "total PCBs," depending on regulatory requirements and project needs. The congener method is of particular value in determining weathered Aroclors. However, analysts should use caution when using the congener method when regulatory requirements are based on Aroclor concentrations. Also, this method is not appropriate as currently written for the determination of the co-planar PCB congeners at the very low (sub part per trillion) concentrations sometimes needed for risk assessment purposes.

1.5 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS (e.g., Method 8270) is also recommended as a confirmation technique, if sensitivity permits (also see Sec. 11.11 of this method). GC/AED may also be used as a confirmation technique, if sensitivity permits (see Method 8085).

1.6 This method includes a dual-column option that describes a hardware configuration in which two GC columns are connected to a single injection port and to two separate detectors. The option allows one injection to be used for dual-column simultaneous analysis.

1.7 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.). Example chromatograms and GC conditions are provided as guidance.

1.8 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.9 Use of this method is restricted to use by, or under the supervision of, personnel appropriately experienced and trained in the use of gas chromatographs (GCs) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using the appropriate matrix-specific sample extraction technique.

2.1.1 Aqueous samples may be extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), Method 3535 (solid-phase extraction), or other appropriate technique or solvents.

2.1.2 Solid samples may be extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3546 (microwave extraction), Method 3550 (ultrasonic extraction), Method 3562 (supercritical fluid extraction), or other appropriate technique or solvents.

2.1.3 Tissue samples may be extracted using Method 3562 (supercritical fluid extraction), or other appropriate technique. The extraction techniques for other solid matrices (see Sec. 2.1.2) may be appropriate for tissue samples.

2.2 Extracts for PCB analysis may be subjected to a sequential sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, this method is not applicable to the analysis of those compounds. Instead, use Method 8081.

2.3 After cleanup, the extract is analyzed by injecting a measured aliquot into a gas chromatograph equipped with either a narrow- or wide-bore fused-silica capillary column and either an electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).

2.4 The chromatographic data may be used to determine the seven Aroclors in Sec. 1.1, selected individual PCB congeners, or total PCBs (see Secs. 11.8 and 11.9).

3.0 DEFINITIONS

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. Also refer to Methods 3500, 3600, and 8000 for a discussion of interferences.

4.2 Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories, as follows:

4.2.1 Contaminated solvents, reagents, or sample processing hardware.

4.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.2.3 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD).

NOTE: A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

4.2.4 Coelution of related analytes – All 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either

document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

4.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.3.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.3.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.3.3 These materials can be removed prior to analysis using Method 3665 (sulfuric acid/permanganate cleanup).

4.4 Cross-contamination of clean glassware can routinely occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned.

4.4.1 Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware, and dry it in an oven at 130 °C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

CAUTION: Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

4.4.2 Other appropriate glassware cleaning procedures may be employed, such as using a muffle furnace at 430 °C for at least 30 min. However, analysts are advised not to place volumetric glassware in a muffle furnace, since the heat will burn off the markings on the glassware and may warp the glassware, changing its volume.

4.5 Sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur contamination should be expected with sediment samples. Sulfur can be removed through the use of Method 3660.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 Gas chromatograph -- An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all necessary accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system. Electrolytic conductivity detectors (ELCDs) may also be employed if appropriate for project needs. If the dual-column option is employed, the gas chromatograph must be equipped with two separate detectors.

6.2 GC columns

This method describes procedures for both single-column and dual-column analyses. The single-column approach involves one analysis to determine that a compound is present, followed by a second analysis to confirm the identity of the compound (Sec. 11.11 describes how GC/MS confirmation techniques may be employed). The single-column approach may employ either narrow-bore (≤ 0.32 -mm ID) columns or wide-bore (0.53-mm ID) columns. The dual-column approach generally employs a single injection that is split between two columns that are mounted in a single gas chromatograph. The dual-column approach generally employs wide-bore (0.53-mm ID) columns, but columns of other diameters may be employed if the analyst can demonstrate and document acceptable performance for the intended application. A third alternative is to employ dual columns mounted in a single GC, but with each column connected to a separate injector and a separate detector.

The columns listed in this section were the columns used in developing the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.2.1 Narrow-bore columns for single-column analysis (use both columns to confirm compound identifications unless another confirmation technique such as GC/MS is employed). Narrow-bore columns should be installed in split/splitless (Grob-type) injectors.

6.2.1.1 30-m x 0.25-mm or 0.32-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1- μ m film thickness.

6.2.1.2 30-m x 0.25-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent), 2.5 μ m coating thickness, 1- μ m film thickness.

6.2.2 Wide-bore columns for single-column analysis (use two of the three columns listed to confirm compound identifications unless another confirmation technique

such as GC/MS is employed). Wide-bore columns should be installed in 1/4-inch injectors, with deactivated liners designed specifically for use with these columns.

6.2.2.1 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5- μ m or 0.83- μ m film thickness.

6.2.2.2 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0- μ m film thickness.

6.2.2.3 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5- μ m film thickness.

6.2.3 Wide-bore columns for dual-column analysis – The three pairs of recommended columns are listed below.

6.2.3.1 Column pair 1

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5- μ m film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0- μ m film thickness.

Column pair 1 is mounted in a press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733) or a Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.

NOTE: When connecting columns to a press-fit Y-shaped connector, a better seal may be achieved by first soaking the ends of the capillary columns in alcohol for about 10 sec to soften the polyimide coating.

6.2.3.2 Column pair 2

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 0.83- μ m film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0- μ m film thickness.

Column pair 2 is mounted in an 8-in. deactivated glass injection tee (Supelco, Catalog No. 2-3665M), or equivalent.

6.2.3.3 Column pair 3

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5- μ m film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (HP-608, DB-608, SPB-608, RTx-35, or equivalent), 0.5- μ m film thickness.

Column pair 3 is mounted in separate injectors and separate detectors.

6.3 Column rinsing kit -- Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000), or equivalent.

6.4 Volumetric flasks -- 10-mL and 25-mL, for preparation of standards.

6.5 Analytical balance, capable of weighing to 0.0001 g.

7.0 REAGENTS AND STANDARDS.

7.1 Reagent-grade or pesticide-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at ≤ 6 °C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot should be stored in individual small vials. All stock standard solutions must be replaced after one year, or sooner if routine QC (see Sec. 9.0) indicates a problem. All other standard solutions must be replaced after six months, or sooner if routine QC (see Sec. 9.0) indicates a problem.

7.2 Solvents used in the extraction and cleanup procedures (appropriate 3500 and 3600 series methods) include *n*-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane) and the solvents must be exchanged to *n*-hexane or isooctane prior to analysis. Therefore, *n*-hexane and isooctane will be required in this procedure. All solvents should be pesticide grade in quality or equivalent, and each lot of solvent should be determined to be free of phthalates.

7.3 The following solvents may be necessary for the preparation of standards. All solvent lots must be pesticide grade in quality or equivalent and should be determined to be free of phthalates.

7.3.1 Acetone, $(\text{CH}_3)_2\text{CO}$

7.3.2 Toluene, $\text{C}_6\text{H}_5\text{CH}_3$

7.4 Organic-free reagent water -- All references to water in this method refer to organic-free reagent water as defined in Chapter One.

7.5 Standard solutions

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards.

7.6 Stock standard solutions (1000 mg/L) -- May be prepared from pure standard materials or can be purchased as certified solutions.

7.6.1 Prepare stock standard solutions by accurately weighing 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution.

7.6.2 Commercially-prepared stock standard solutions may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.7 Calibration standards for Aroclors

7.7.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector. See Method 8000 for additional information regarding the preparation of calibration standards.

7.7.2 Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in Sec. 7.7.1 have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen (see Sec. 11.4). Prepare a standard for each of the other Aroclors. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed at the discretion of the analyst based on project requirements.

7.7.3 Other standards (e.g., other Aroclors) and other calibration approaches (e.g., non-linear calibration for individual Aroclors) may be employed to meet project needs. When the nature of the PCB contamination is already known, use standards of those particular Aroclors. See Method 8000 for information on non-linear calibration approaches.

7.8 Calibration standards for PCB congeners

7.8.1 If results are to be determined for individual PCB congeners, then standards for the pure congeners must be prepared. The table in Sec. 1.1 lists 19 PCB congeners that have been tested by this method along with the IUPAC numbers designating these congeners. This procedure may be appropriate for other congeners as well, but the analyst must either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

7.8.2 Stock standards may be prepared in a fashion similar to that described for the Aroclor standards, or may be purchased as commercially-prepared solutions. Stock standards should be used to prepare a minimum of five concentrations by dilution of the stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector.

7.9 Internal standard

7.9.1 When PCB congeners are to be determined, the use of an internal standard is highly recommended. Decachlorobiphenyl may be used as an internal standard, added to each sample extract prior to analysis, and included in each of the initial calibration standards.

7.9.2 When PCBs are to be determined as Aroclors, an internal standard is typically not used, and decachlorobiphenyl is employed as a surrogate (see Sec. 7.10).

7.9.3 When decachlorobiphenyl is an analyte of interest, as in some PCB congener analyses, see Sec. 7.10.3.

7.10 Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. The choice of surrogate compounds will depend on analysis mode chosen, e.g., Aroclors or congeners. The following compounds are recommended as surrogates. Other surrogates may be used, provided that the analyst can demonstrate and document performance appropriate for the data quality needs of the particular application.

7.10.1 When PCBs are to be determined as Aroclors, decachlorobiphenyl may be used as a surrogate, and is added to each sample prior to extraction. Prepare a solution of decachlorobiphenyl in acetone. The recommended spiking solution concentration is 5 mg/L. Tetrachloro-*m*-xylene also may be used as a surrogate for Aroclor analysis. If used, the recommended spiking solution concentration is 5 mg/L in acetone. (Other surrogate concentrations may be used, as appropriate for the intended application.)

7.10.2 When PCB congeners are to be determined, decachlorobiphenyl is recommended for use as an internal standard, and therefore it cannot also be used as a surrogate. Tetrachloro-*m*-xylene may be used as a surrogate for PCB congener analysis. The recommended spiking solution concentration is 5 mg/L in acetone. (Other surrogate concentrations may be used, as appropriate for the intended application.)

7.10.3 If decachlorobiphenyl is a target congener for the analysis, 2,2',4,4',5,5'-hexabromobiphenyl may be used as an internal standard or a surrogate.

7.11 DDT analog standard -- Used to determine if the commonly found DDT analogs (DDT, DDE, and DDD) elute at the same retention times as any of the target analytes (congeners or Aroclors). A single standard containing all three compounds should be sufficient. The concentration of the standard is left to the judgement of the analyst.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes."

8.2 Extracts should be stored under refrigeration in the dark and should be analyzed within 40 days of extraction.

NOTE: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

9.3 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples.

9.3.1 Include a calibration standard after each group of 20 samples (it is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. Thus, injections of method blank extracts, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. The response factors for the calibration should be within ± 20 percent of the initial calibration (see Sec. 11.6.2). When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

9.3.2 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during initial calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed. The retention times of the internal standards must also be evaluated. A retention time shift of >30 sec necessitates reanalysis of the affected sample.

9.4 Initial demonstration of proficiency

9.4.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.4.2 It is suggested that the QC reference sample concentrate (as discussed in Methods 8000 and Method 3500) contain PCBs as Aroclors at 10-50 mg/L in the concentrate for water samples, or PCBs as congeners at the same concentrations. A 1-mL volume of this concentrate spiked into 1 L of reagent water will result in a sample concentration of 10-50 µg/L. If Aroclors are not expected in samples from a particular source, then prepare the QC reference samples with a mixture of Aroclors 1016 and 1260. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for the QC reference sample. See Method 8000 for additional information on how to accomplish this demonstration. Other concentrations may be used, as appropriate for the intended application.

9.4.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Method 8000 for procedures for evaluating method performance.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6 Sample quality control for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample when surrogates are used. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike

duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for spiking. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD.

9.6.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample quality control procedures for preparation and analysis. In-house acceptance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.7 Surrogate recoveries

If surrogates are used, the laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

9.8 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

See Sec. 11.0 for information on calibration and standardization.

11.0 PROCEDURE

11.1 Sample extraction

11.1.1 Refer to Chapter Two and Method 3500 for guidance in choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510), a continuous liquid-liquid extractor (Method 3520), solid-phase extraction (Method 3535), or other appropriate technique. Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using one of the Soxhlet extraction methods (Method 3540 or 3541), pressurized fluid extraction (Method 3545), microwave extraction (Method 3546),

ultrasonic extraction (Method 3550), supercritical fluid extraction (Method 3562), or other appropriate technique or solvents. Tissue samples are extracted using supercritical fluid extraction (Method 3562) or other appropriate technique.

NOTE: The use of hexane-acetone generally reduces the amount of interferences that are extracted and improves signal-to-noise.

The choice of extraction solvent and procedure will depend on the analytes of interest. No single solvent or extraction procedure is universally applicable to all analyte groups and sample matrices. The analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest, for any solvent system and extraction procedure employed, *including* those specifically listed in this method. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Each new sample type must be spiked with the compounds of interest to determine the percent recovery. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

11.1.2 Reference materials, field-contaminated samples, or spiked samples should be used to verify the applicability of the selected extraction technique to each new sample type. Such samples should contain or be spiked with the compounds of interest in order to determine the percent recovery and the limit of detection for that sample type (see Chapter One). When other materials are not available and spiked samples are used, they should be spiked with the analytes of interest, either specific Aroclors or PCB congeners. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture may be an appropriate choice for spiking. See Methods 3500 and 8000 for guidance on demonstration of initial method proficiency as well as guidance on matrix spikes for routine sample analysis.

11.1.3 The extraction techniques for solids may be applicable to wipe samples and other sample matrices not addressed in Sec. 11.1.1. The analysis of oil samples may need special sample preparation procedures that are not described here. Analysts should follow the steps described in Sec. 11.1.2 to verify the applicability of the sample preparation and extraction techniques for matrices such as wipes and oils.

11.2 Extract cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to Methods 3600, 3660 and 3665 for general guidance on extract cleanup.

11.3 GC conditions

This method allows the analyst to choose between a single-column or a dual-column configuration in the injector port. The columns listed in this section were the columns used to develop the method performance data. Listing these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Wide-bore or narrow-bore columns may be used with either option. Laboratories may use either the columns listed in this method or other capillary columns or columns of other dimensions, provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

must contain the internal standard (see Sec. 7.9) at the same concentration as the sample extracts. When PCBs are to be determined as Aroclors, external standard calibration is generally used.

NOTE: Because of the sensitivity of the electron capture detector, always clean the injection port and column prior to performing the initial calibration.

11.4.2 When PCBs are to be quantitatively determined as congeners, an initial multi-point calibration must be performed that includes standards for all the target analytes (congeners). See Method 8000 for details on calibration options.

11.4.3 When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts, described below.

11.4.3.1 As noted in Sec. 7.7.1, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial multi-point calibration is performed using the mixture of Aroclors 1016 and 1260 described in Sec. 7.7.1. See Method 8000 for guidance on the use of linear and non-linear calibrations.

11.4.3.2 Standards of the other five Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture in Sec. 11.4.3.1 has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards in Sec. 11.4.3.1. For non-linear calibrations, see Sec. 11.4.3.3.

11.4.3.3 In situations where only a few Aroclors are of interest for a specific project, the analyst may employ a multi-point initial calibration of each of the Aroclors of interest (e.g., five standards of Aroclor 1232 if this Aroclor is of concern and linear calibration is employed) and not use the 1016/1260 mixture described in Sec. 11.4.3.1 or the pattern recognition standards described in 11.4.3.2. When non-linear calibration models are employed, more than five standards of each Aroclor of interest will be needed to adequately describe the detector response (see Method 8000).

11.4.4 Establish the GC operating conditions appropriate for the configuration (single-column or dual column, Sec. 11.3), using Tables 1 or 2 as guidance. Optimize the instrumental conditions for resolution of the target compounds and sensitivity. A final temperature of between 240 °C and 275 °C may be needed to elute decachlorobiphenyl. The use of injector pressure programming will improve the chromatography of late eluting peaks.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

11.4.5 A 2- μ L injection of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.

11.4.6 Record the peak area (or height) for each congener or each characteristic Aroclor peak to be used for quantitation.

11.4.6.1 A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.

11.4.6.2 Late-eluting Aroclor peaks are generally the most stable in the environment. Table 5 lists diagnostic peaks in each Aroclor, along with example retention times on two GC columns suitable for single-column analysis. Table 6 lists 13 specific PCB congeners found in Aroclor mixtures. Table 7 lists PCB congeners with example retention times on a DB-5 wide-bore GC column. Use these tables as guidance in choosing the appropriate peaks. Each laboratory must determine retention times and retention time windows for their specific application of the method.

11.4.7 When determining PCB congeners by the internal standard procedure, calculate the response factor (RF) for each congener in the calibration standards relative to the internal standard, decachlorobiphenyl, using the equation that follows.

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate, in μ g/L.

C_{is} = Concentration of the internal standard, in μ g/L.

11.4.8 When determining PCBs as Aroclors by the external standard technique, calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards (from either Sec. 11.4.3.1 or 11.4.3.2) using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Using the equation above, a calibration factor will be determined for each characteristic peak, using the total mass of the Aroclor injected. These individual calibration factors are used to quantitate sample results by applying the factor for each individual peak to the area of that peak, as described in Sec. 11.9.

For a five-point calibration, five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture, e.g., there will be at least 25 separate calibration factors for the mixture. The single standard for each of the other Aroclors (see Sec. 11.4.3.1) will generate at least three calibration factors, one for each selected peak.

If a non-linear calibration model is employed, as described in Method 8000, then additional standards containing each Aroclor of interest will be employed, with a corresponding increase in the total number of calibration factors.

11.4.9 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration, if a linear calibration model is used. This involves the calculation of the mean response or calibration factor, the standard deviation, and the relative standard deviation (RSD) for each congener or Aroclor peak.

When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the linear calibration models must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors (see Sec. 11.4.3.3), use the calibration factors from those standards to evaluate linearity.

See Method 8000 for the specifics of the evaluation of the linearity of the calibration and guidance on performing non-linear calibrations. In general, non-linear calibrations also will consider each characteristic Aroclor peak separately.

11.5 Retention time windows

Absolute retention times are generally used for compound identification. When absolute retention times are used, retention time windows are crucial to the identification of target compounds, and should be established by one of the approaches described in Method 8000. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. Analysts should consult Method 8000 for the details of establishing retention time windows. Other approaches to compound identification may be employed, provided that the analyst can demonstrate and document that the approaches are appropriate for the intended application. When PCBs are determined as congeners by an internal standard technique, absolute retention times may be used in conjunction with relative retention times (relative to the internal standard).

When conducting either Aroclor or congener analysis, it is important to determine that common single-component pesticides such as DDT, DDD, and DDE do not elute at the same retention times as the target congeners. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. Therefore, in conjunction with determining the retention time windows of the congeners, the analyst should analyze a standard containing the DDT analogs. This standard need only be analyzed when the retention time

windows are determined. It is not considered part of the routine initial calibration or calibration verification steps in the method, nor are there any performance criteria associated with the analysis of this standard.

If Aroclor analysis is performed and any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for use in quantitation (see Sec. 11.4.6), then the analyst must either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog. If PCB congener analysis is performed and any of the DDT analogs elute at the same retention time as a PCB congener of interest, then the analyst must adjust the GC conditions to achieve better resolution.

11.6 Gas chromatographic analysis of sample extracts

11.6.1 The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

11.6.2 Verify calibration at least once each 12-hr shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended to minimize the number of samples requiring reinjection when QC limits are exceeded) and at the end of the analysis sequence. For Aroclor analyses, the calibration verification standard should be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

11.6.2.1 The calibration factor for each analyte calculated from the calibration verification standard (CF_v) should not exceed a difference of more than ± 20 percent when compared to the mean calibration factor from the initial calibration curve. If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

11.6.2.2 When internal standard calibration is used for PCB congeners, the response factor calculated from the calibration verification standard (RF_v) should not exceed a ± 20 percent difference when compared to the mean response factor from the initial calibration. If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{\overline{RF} - RF_v}{\overline{RF}} \times 100$$

11.6.2.3 If the calibration does not meet the $\pm 20\%$ limit on the basis of each compound, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within $\pm 20\%$, then a new initial calibration must be prepared. See Sec. 11.6.6 for a discussion on the effects of a failing calibration verification standard on sample results.

11.6.3 Inject a measured aliquot of the concentrated sample extract. A 2- μL aliquot is suggested, however, other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume should be used for both the calibration standards and the sample extracts, unless the analyst can demonstrate acceptable performance using different volumes or conditions. Record the volume injected and the resulting peak size in area units.

11.6.4 Qualitative identifications of target analytes are made by examination of the sample chromatograms, as described in Sec. 11.7.

11.6.5 Quantitative results are determined for each identified analyte (Aroclors or congeners), using the procedures described in Secs. 11.8 and 11.9 for either the internal or the external calibration procedure (Method 8000). If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area when overlapping peaks cause errors in area integration.

11.6.6 Each sample analysis employing external standard calibration must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hr analytical shift), or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration verification criteria in Sec. 11.6.2.

Multi-level standards (mixtures or multi-component analytes) are highly recommended to ensure that detector response remains stable for all analytes over the calibration range.

When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be evaluated to prevent misquantitations and possible false negative results, and reinjection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis.

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., $>20\%$, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, since the verification standard has demonstrated that the analyte would have been detected if it were present. In contrast, if an analyte

above the QC limits was detected in a sample extract, then reinjection is necessary to ensure accurate quantitation.

If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then reinjection is necessary. The purpose of this reinjection is to ensure that the analyte could be detected, if present, despite the change in the detector response, e.g., to protect against a false negative result.

11.6.7 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

11.6.8 The use of internal standard calibration techniques does not require that all sample results be bracketed with calibration verification standards. However, when internal standard calibration is used, the retention times of the internal standards and the area responses of the internal standards should be checked for each analysis. Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard and/or changes in internal standard areas of more than -50 to +100% are cause for concern and must be investigated.

11.6.9 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

11.6.10 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

11.6.11 If compound identification or quantitation is precluded due to interferences (e.g., broad, rounded peaks or ill-defined baselines are present), corrective action is warranted. Cleanup of the extract or replacement of the capillary column or detector may be necessary. The analyst may begin by rerunning the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

11.7 Qualitative identification

The identification of PCBs as either Aroclors or congeners using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. See Method 8000 for information on the establishment of retention time windows.

Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Confirmation is necessary when the sample composition is not well characterized. See Method 8000 for information on confirmation of tentative identifications. See Sec. 11.11 of this procedure for information on the use of GC/MS as a confirmation technique.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

11.7.1 When simultaneous analyses are performed from a single injection (the dual-column GC configuration described in Sec. 11.3), it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

11.7.2 The results of a single column/single injection analysis may be confirmed, if necessary, on a second, dissimilar, GC column. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis. That standard may be either the individual congeners, individual Aroclor or the Aroclor 1016/1260 mixture.

11.7.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. This approach should not be attempted for samples from unknown or unfamiliar sources or for samples that appear to contain mixtures of Aroclors. In order to employ this approach, the analyst must document:

- The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard.
- The absence of major peaks representing any other Aroclor.
- The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, generator knowledge, etc.).

This information should either be provided to the data user or maintained by the laboratory.

11.7.4 See Sec. 11.11 for information on GC/MS confirmation.

11.8 Quantitation of PCBs as congeners

11.8.1 The quantitation of PCB congeners is accomplished by the comparison of the sample chromatogram to those of the PCB congener standards, using the internal standard technique (see Method 8000). Calculate the concentration of each congener.

11.8.2 Depending on project requirements, the PCB congener results may be reported as congeners, or may be summed and reported as total PCBs. The analyst should use caution when using the congener method for quantitation when regulatory requirements are based on Aroclor concentrations. See Sec. 11.9.3.

11.8.3 The analytical procedures for these 19 congeners may be appropriate for the analysis of other congeners not specifically included in this method and may be used

as a template for the development of such a procedure. However, all 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

11.9 Quantitation of PCBs as Aroclors

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

11.9.1 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

11.9.2 Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen in Sec. 11.4.6.1. and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. Non-linear calibration may result in different models for each selected peak. A concentration is determined using each of the characteristic peaks, using the individual calibration factor calculated for that peak in Sec. 11.4.8, and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

11.9.3 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. If the purpose of the analysis is not regulatory compliance monitoring on the basis of Aroclor concentrations, then it may be more appropriate to perform the analyses using the PCB congener approach described in this method. If results in terms of Aroclors are required, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

11.10 Confirmation

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is necessary when the sample composition is not well characterized. Confirmatory techniques such as gas chromatography with a dissimilar column or a mass spectrometer should be used. See Method 8000 for information on confirmation of tentative identifications.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

When the dual-column approach is employed, the target phenols are identified and confirmed when they meet the identification criteria on both columns.

11.11 GC/MS confirmation

GC/MS confirmation may be used in conjunction with either single- or dual-column analysis if the concentration is sufficient for detection by GC/MS.

11.11.1 Full-scan quadrupole GC/MS will normally require a higher concentration of the analyte of interest than full-scan ion trap or selected ion monitoring techniques. The concentrations will be instrument-dependent, but values for full-scan quadrupole GC/MS may be as high as 10 ng/μL in the final extract, while ion trap or SIM may only be a concentration of 1 ng/μL.

11.11.2 The GC/MS must be calibrated for the target analytes when it is used for quantitative analysis. If GC/MS is used only for confirmation of the identification of the target analytes, then the analyst must demonstrate that those PCBs identified by GC/ECD can be confirmed by GC/MS. This demonstration may be accomplished by analyzing a single-point standard containing the analytes of interest at or below the concentrations reported in the GC/ECD analysis. When using SIM techniques, the ions and retention times should be characteristic of the Aroclors to be confirmed.

11.11.3 GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the extract of the associated blank.

11.12 GC/AED confirmation by Method 8085 may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/AED.

11.13 Chromatographic system maintenance as corrective action

When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

11.13.1 Splitter connections

For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters (up to 30 cm) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

11.13.2 Metal injector body

Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

11.13.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the entire inside of the injector port with acetone and then rinse it with toluene, catching the rinsate in the beaker.

11.13.2.2 Consult the manufacturer's instructions regarding deactivating the injector port body. Glass injection port liners may need deactivation with a silanizing solution containing dimethyldichlorosilane. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

11.13.3 Column rinsing

Rinse the column with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases may be the only solvent necessary. Fill the column with methylene chloride and allow it to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. Afterwards, flush the column with fresh methylene chloride, drain the column, and dry it at room temperature with a stream of ultrapure nitrogen.

12.0 DATA ANALYSIS AND CALCULATIONS

See Secs. 11.6 through 11.9 for information regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used. Table 8 provides single laboratory recovery data for Aroclors spiked into clay and soil and extracted with automated Soxhlet. Table 9 provides multiple laboratory data on the precision and accuracy for Aroclors spiked into soil and extracted by automated Soxhlet. These data are provided for guidance purposes only.

13.3 During method performance studies, the concentrations determined as Aroclors were higher than those obtained using the congener method for the limited set of congeners listed in Sec. 1.1. In certain soils, interference prevented the measurement of congener 66. Recoveries of congeners from environmental reference materials ranged from 51 - 66% of the certified Aroclor values, illustrating the potential difficulties in using congener analysis to demonstrate compliance with Aroclor-based regulatory limits. These data are provided for guidance purposes only.

13.4 Tables 10 and 11 contain laboratory performance data for several PCB congeners using supercritical fluid extraction (Method 3562) on an HP 7680 to extract solid samples, including soils, sewage sludge, and fish tissue. Seven replicate extractions were performed on each sample. The method was performed using a variable restrictor and solid trapping material (Florisil). These data are provided for guidance purposes only. Sample analysis was performed by GC/ECD. The following solid samples were used for this study:

13.4.1 Two field-contaminated certified reference materials were extracted by a single laboratory. One of the materials (EC-5) was a lake sediment from Environment Canada. The other material (EC-1) was soil from a dump site and was provided by the National Science and Engineering Research Council of Canada. The average recoveries for EC-5 are based on the certified value for that sample. The average recoveries for EC-1 are based on the certified value of the samples or a Soxhlet value, if a certified value was unavailable for a specific analyte. These data are provided for guidance purposes only.

13.4.2 Four certified reference materials were extracted by two independent laboratories. The materials included a marine sediment from NIST (SRM 1941), a fish tissue from NIST (SRM 2974), a sewage sludge from BCR European Union (CRM 392), and a soil sample from BCR European Union (CRM 481). The average recoveries were based on the certified value of the samples or a Soxhlet value, if a certified value was unavailable for a specific analyte. These data are provided for guidance purposes only.

13.4.3 A weathered sediment sample from Michigan (Saginaw Bay) was extracted by a single laboratory. Soxhlet extractions were carried out on this sample and the SFE recovery is relative to that for each congener. The average recoveries were based on the certified value of the samples. Additional data are shown in the tables for some congeners for which no certified values were available. These data are provided for guidance purposes only.

13.5 Tables 12 through 14 contain single laboratory recovery data for Aroclor 1254 using solid-phase extraction (Method 3535). Recovery data at 2, 10, and 100 µg/L are presented. Results represent three replicate solid-phase extractions of spiked wastewaters. Two different wastewaters from each wastewater type were spiked. All of the extractions were performed using 90-mm C₁₈ disks. These data are provided for guidance purposes only.

13.6 Single-laboratory data were developed for PCBs extracted by pressurized fluid extraction (Method 3545) from sewage sludge, a river sediment standard reference material (SRM 1939), and a certified soil reference material (CRM911-050). Certified values were available for five PCB congeners for the sewage sludge and for four congeners in SRM 1939. The soil reference material was certified for Aroclor 1254. All pressurized fluid extractions were conducted using hexane:acetone (1:1), at 100 °C, 1300-1500 psi, and a 5-min static extraction. Extracts were analyzed by GC/ECD. The data are presented in Tables 15 through 17 and are reported in detail in Reference 13. These data are provided for guidance purposes only.

13.7 Single-laboratory accuracy data were obtained for PCBs extracted by microwave extraction (Method 3546) from three reference materials, EC-1, EC-2, and EC-3, from Environment Canada. Natural soils, glass fiber, and sand samples were also used as matrices that were spiked with PCBs. Concentrations varied between 0.2 and 10 µg/g (total PCBs). All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by GC/ECD. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are presented in Tables 18 through 20 and are reported in detail in Reference 14. These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of

environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society, Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, DC, 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

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12. C. Markell, "3M Data Submission to EPA," letter to B. Lesnik, June 27, 1995.
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17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

ATTACHMENT 7

ANALYSIS OF ASBESTOS IN SOIL

National Institute for Occupational Safety and Health (NIOSH) Method 9002 is both a qualitative and semi-quantitative method for the determination of asbestos in bulk samples by PLM. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience. The method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope (see TEM).

TRANSMISSION ELECTRON MICROSCOPY METHODS

TEM – EPA 600/R-93/116 – VE by TEM after Gravimetric Reduction (< 1% LOD)



A useful method originally referred to as the "Chatfield Method" that improves both the accuracy and precision of analysis of Non-friable Organically Bound samples. Bulk samples containing dense matrices like floor tiles, mastics and roofing materials are considered Problem Samples. If present, asbestos fibers are tightly bound to the matrix material and not easily isolated and detected by light microscopy often resulting in a false negative. Additional prepping procedures (NOB Preparation) include gravimetric reduction by ashing the samples several hours in a muffle furnace at high temperatures, followed by dissolution of the remaining residue with Hydrochloric Acid. The final residue is then analyzed by TEM for the presence of asbestos. TEM is capable of detecting asbestos fibers too small to resolve by optical microscopy (PLM) such as those typically found in Vinyl Asbestos Tile (VAT).

Drop Mount - Qualitative (presence/absence) Analysis

This method quickly checks for asbestos content in bulk samples. A subsample of a material is ground in a mortar and pestle, sonicated in particle-free de-ionized water and placed on a carbon coated grid. The grid is then analyzed by TEM for the presence of asbestos.

TEM – EPA 600/J-93/167 – Asbestos in Carpet Samples

A small piece of carpet (typically 10 by 10 cm) is analyzed by TEM to determine if asbestos fibers are present. Essentially, the carpet sample is sonicated in particle free water to release the fibers and the resulting liquid is filtered and analyzed by TEM. This destructive method has a much greater sensitivity for asbestos bound up in carpet fibers than the sampling protocol outlined in the ASTM D5755 "Microvac" method.

ASBESTOS IN WATER

TRANSMISSION ELECTRON MICROSCOPY METHODS

TEM – Drinking Water – EPA Method 100.2 (7 MFL LOD)

EPA 100.1 and its successor, EPA 100.2 are TEM methods for the determination of the presence and quantitation of asbestos structures in drinking water samples. EPA 100.1/100.2 allows for the quantitation of asbestos structures >10 µm in length. Results are expressed in millions of fibers >10 µm per liter (MFL). An analytical sensitivity of at least 0.2 MFL is required. Asbestos fibers < 10 µm in length are not included in the final calculation, but can be documented upon request.

TEM – Waste Water/Solvents – EPA Method 100.2

The same methodology is utilized for the analysis of waste water samples. The difference results in sensitivities often not achieving the 0.2 MFL required by EPA 100.2. This decrease in sensitivity is typically due to excessive levels of debris or turbidity in the samples. These samples also typically require additional prepping procedures to achieve a "readable" result.



ASBESTOS IN SETTLED DUST

TRANSMISSION ELECTRON MICROSCOPY METHODS

TEM – ASTM D5755-03 Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading

This method utilizes air cassettes as a micro-vacuum sampling device to gather dust from a carefully measured surface. Typically 100 cm² is sampled. The samples are then analyzed by TEM to identify asbestos. The result is an estimate of the surface concentration of asbestos structures reported in asbestos structures per square centimeter. NOTE: There are no accepted "pass/fail" criteria for this method. This method is best employed to compare effectiveness of pre- and post- asbestos abatement activities.

TEM – ASTM 5756-02 Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration

Same as ASTM D5755 method but this method provides results in mass of asbestos per unit area of sample surface, OR mass of asbestos per mass of sampled dust.

TEM – ASTM D6480-05 Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy

A method to identify asbestos in dust samples wiped (as opposed to vacuumed) from surfaces and determine the concentration of asbestos structures in number of asbestos structures per unit area. This method can also be conducted on a qualitative (presence/absence) basis if the area sampled is unknown. Contact J3 for information on the proper types of wipes required by this method.



ASBESTOS IN SOIL AND ROCK

TRANSMISSION ELECTRON MICROSCOPY

TEM – CARB 435 – Determination of Asbestos Content of Serpentine Aggregate with TEM Confirmation

This method involves a fine milling of samples (typically aggregate, but often soils) to create a completely homogenous mixture. This maximizes the possibility of detection and increases the accuracy of quantification. The analysis can be performed by Polarized Light Microscopy (PLM) or Transmission Electron Microscopy (TEM). The TEM analysis is used for a more definitive analysis (confirmation of None Detected by PLM) with sensitivities down to 0.001% by weight.

TEM – J3 In-House Sieve Method plus TEM Confirmation

A sieve method that utilizes a combination of ovens, mills and sieves to best prepare a homogenous subsample to be analyzed by either PLM or TEM depending on the soil sample and type and concentration of asbestos in the sample.

POLARIZED LIGHT MICROSCOPY

PLM – CARB Method 435 Determination of Asbestos Content of Serpentine Aggregate (Rapid Screen Only)

This method involves a fine milling of the sample (typically aggregate) to create a completely homogenous mixture. This maximizes the possibility of detection and increases the accuracy of quantification. The analysis can be performed by Polarized Light Microscopy (PLM) or Transmission Electron Microscopy (TEM) utilizing the widely recognized EPA/600/R-93/118 method. The PLM method uses a 400 Point Count (0.25%) or 1,000 Point Count (0.1%).

PLM – J3 In-House Sieve Method (Quantitative)

A sieve method that utilizes a combination of ovens, mills and sieves to best prepare a homogenous subsample to be analyzed by either PLM or TEM depending on the concentration of asbestos in the sample.

PLM – EPA Region I Screening Protocol (Qualitative)

This comparably low tech screening method was developed in 1994 and is considered to provide semi-quantitative results at best. Good for rapid detection of asbestos in a large number of samples needing rapid turnaround times.



ASBESTOS IN VERMICULITE

POLARIZED LIGHT MICROSCOPY

PLM – EPA 600/R-04/004 – PLM (Rapid Screen Only - Cincinnati Method)

This method provides a procedure for the rapid characterization of fibrous amphibole constituents of vermiculite attic insulation. Vermiculite from Zonolite Mountain near Libby, MT is likely to contain fibrous amphiboles and was widely used as attic insulation or sound dampening material throughout the continental U.S. TEM confirmation of samples determined to be negative by PLM is recommended.

TRANSMISSION ELECTRON MICROSCOPY

TEM – EPA 600/R-04/004 – PLM Rapid Screen + TEM (Full Cincinnati Method)

This method provides a procedure for the characterization of fibrous amphibole constituents of vermiculite attic insulation. Vermiculite from Zonolite Mountain near Libby, MT is likely to contain fibrous amphiboles and was widely used as attic insulation or sound dampening material throughout the continental U.S.

ASBESTOS IN LUNG AND OTHER TISSUE/TISSUE FIBER BURDEN ANALYSIS

TRANSMISSION ELECTRON MICROSCOPY

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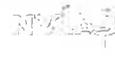
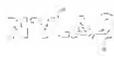
This method focuses on identification and quantification of asbestos fibers and ferruginous bodies in human tissue samples for litigation support. Please contact J3 Resources, Inc. for details.



NON-ASBESTOS FIBEROUS MINERAL QUALIFICATIONS

TRANSMISSION ELECTRON MICROSCOPY

J3 Resources, Inc. can identify a wide list of natural and manmade fibers by TEM including Refractory Ceramic Fibers (RCF's); Talc; Man Made Mineral/Vitreous Fibers (MMMF's/MMVF's); Erionite.





TEXAS DEPARTMENT OF STATE HEALTH SERVICES

J3 RESOURCES INC

is certified to perform as a

**Asbestos Laboratory
PCM, PLM, TEM**

in the State of Texas within the purview of Texas Occupations Code, chapter 1954, so long as this license is not suspended or revoked and is renewed according to the rules adopted by the Texas Board of Health.

A handwritten signature in cursive script, appearing to read "David Lahey MD".

DAVID LAKEY, M.D.
COMMISSIONER OF HEALTH

License Number: 300273

Control Number: 95782

Expiration Date: 3/15/2014

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California Environmental Protection Agency

 **Air Resources Board**

Method 435

**Determination of Asbestos
Content of Serpentine Aggregate**

Adopted: June 6, 1991

Method 435

Determination of Asbestos Content of Serpentine Aggregate

1 PRINCIPLE AND APPLICABILITY

1.1 Principle.

Asbestos fibers may be released from serpentine rock formations and are determined by microscopic techniques. The results are very sensitive to sampling procedures. The analytical results are reported in percent asbestos fibers which is the percent number of asbestos fibers contained in 400 randomly chosen particles of a bulk sample. Since the homogeneity of the material is unknown, the uncertainty in the sampling cannot be defined. The uncertainty of the analytical technique is two percent if twenty asbestos fibers are counted in a sample of 400 particles. The derivation of this uncertainty value is explained in Section 7.4.

1.2 Applicability.

This method is applicable to determining asbestos content of serpentine aggregate in storage piles, on conveyor belts, and on surfaces such as roads, shoulders, and parking lots.

2 DEFINITIONS

2.1 Bulk Sample

A sample of bulk material.

2.2 Grab Sample

A sample taken from a volume of material.

2.3 Composite Sample

A mixture or blend of material from more than one grab sample.

2.4 Serpentine

Serpentinite, serpentine rock or serpentine material.

2.5 Executive Officer

The term Executive Officer as used in this method shall mean the Executive Officer of the Air Resources Board (ARB) or Air Pollution Control Officer/Executive Officer of a local air pollution control district/air quality management district.

3 APPLICABLE SOURCES

This method can be used to obtain bulk material samples from three types of sources:

1. Serpentine aggregate storage piles,
2. Serpentine aggregate conveyor belts
3. Serpentine aggregate covered surfaces.

4 SAMPLING APPARATUS

4.1 Serpentine Aggregate Storage Piles.

Tube insertion often provides the simplest method of aggregate material investigation and sampling. Insertion tubes shall be adequate to provide a relatively rapid continuous penetration force.

- 4.1.1 Thin-walled tubes should be manufactured as shown in Figure 1. The tube should have an outside diameter between 2 to 5 inches and be made of metal or plastic having adequate strength for penetration into aggregate piles. These tubes shall be clean and free of surface irregularities including projecting weld seams. Further information on these tubes can be found in Table 1 and ASTM D 1587-83, which is incorporated herein by reference.
- 4.1.2 The insertion tube can be made out of commercially available two inch PVC Schedule 40 pipe. Further information on the tube can be found in Table 2.
- 4.1.3 A round point shovel may be used.
- 4.2 Serpentine Aggregate Conveyor Belts.
 - 4.2.1 Sampling of aggregate off a conveyor belt requires a hand trowel, a small brush, and a dust pan.
 - 4.2.2 Two templates as shown in Figure 2 are needed to isolate material on the conveyor belt.
 - 4.2.3 An automated belt sampler may be used.

4.3 Serpentine Aggregate Covered Surfaces.

A shovel, a hand or machine-operated auger or other suitable equipment can be used to collect samples of aggregate materials on covered surfaces.

4.3.1 Hand-Operated Augers.

4.3.1.1 Helical Augers-Small lightweight augers such as spiral-type augers and ship-type augers may be used. A description of these augers can be found in ASTM D1452-80, which is incorporated herein by reference.

4.3.1.2 Orchard barrel and open spiral-type tubular augers may be used to collect samples. These augers range in size from 1.5 through 8 inches, and have the common characteristic of appearing essentially tubular when viewed from the digging end. Further description of these auger types can be found in ASTM D1452-80.

4.3.1.3 Clam Shell or Iwan-Type post-hole augers may be used to collect samples from surfaces generally 2 through 8 inches in diameter and have a common mean of blocking the escape of soil from the auger. Further description of these augers can be found in ASTM D1452-80.

4.3.2 Machine-Operated Augers

Machine-Operated Augers such as helical augers and stinger augers may be used. These augers are normally operated by heavy-duty, high-torque machines, designed for heavy construction work. Further description of these augers can be found in ASTM D1452-80.

4.3.3 A round point shovel can also be used to obtain a sample of aggregate covered surface material.

5 SAMPLING

The sampling procedure has been developed to provide an unbiased collection of bulk samples. A sampling plan, including a description of how the grab samples will be randomly collected and the number of samples to be collected, shall be developed. Prior to conducting any sampling the sampling plan shall be submitted to the Executive Officer for approval, if the sampling is conducted for determining compliance with a rule or regulation. The amount of composite 200 mesh material, as described below, shall be sufficient to provide sample to the source or Executive Officer, if requested, and a sample to be archived for future use.

A single test as described below shall cover:

- a) 1000 tons of aggregate for piles and conveyor belts, or
- b) one acre aggregate covered surface, or
- c) one mile of aggregate covered road, or

d) two acres or two miles of dual aggregate covered shoulders.

Exposure to airborne asbestos fibers is a health hazard. Asbestos has been listed by the Governor as causing cancer and identified by the Air Resources Board as a toxic air contaminant. Serpentine aggregate may contain asbestos. Bulk samples collected can contain friable asbestos fibers and may release fibers during sampling, handling or crushing steps. Adequate safety precautions should be followed to minimize the inhalation of asbestos fibers. Crushing should be carried out in a ventilated hood with continuous airflow (negative pressure) exhausting through an HEPA filter. Handling of samples without these precautions may result in the inhalation of airborne asbestos fibers.

5.1 Serpentine Aggregate Storage Piles.

Serpentine aggregate storage piles typically have a conical or a triangular prism shape. The aggregate is introduced at the top of the pile and is allowed to flow over the side. This action, called sloughing, causes a size segregation to occur with the finer material deposited towards the top of the pile.

The locations where grab samples will be taken are randomly chosen over the surface of the pile. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling personnel plan. For 1000 tons of product, a grab sample shall be taken at a minimum of three randomly chosen sampling locations. A minimum of three grab samples shall be taken even if the product pile contains less than 1000 tons of material. The slough is raked or shoveled away from the sampling location. A sampling apparatus is inserted one foot into the pile and the material is removed and is placed in an appropriate sized sampling container. Some of the possible sampling apparatus is discussed in Section 4.1. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Braun mill or equivalent to produce a material of which the majority shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label shall contain all the information described in Section 6 (except item 4).

5.2 Serpentine Aggregate Conveyor Belts.

Serpentine aggregate is transported from the rock crushing plant to a product stacking belt and finally to a storage pile or to a waiting truck for delivery to a buyer.

The grab samples shall be taken from the product stacking belt or if this is not possible then at the first transfer point before the stockpile. The grab samples shall be collected by stopping the belt a minimum of three times or using an automated sampler. The method of randomly choosing the sampling locations and intervals is left up to sampling personnel

but must follow the procedure specified in the sampling plan. For 1000 tons of product, a grab sample is taken at a minimum of three randomly selected intervals. A minimum of three samples shall be taken even if the generated product is less than 1000 tons. Each time the belt is stopped to take a grab sample, templates, as shown in Figure 2, are placed a minimum of six inches apart to isolate the material on the belt. The material within the templates is removed with a small shovel or with a brush and a dust pan for the finer material and is placed in an appropriate sized sampling container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Bruan mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

5.3 Serpentine Aggregate Covered Surfaces.

5.3.1 Serpentine Aggregate Covered Roads

A serpentine aggregate-covered road shall be characterized by taking grab samples from a minimum of three randomly chosen locations per mile of road. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling plan. A minimum of three samples shall be taken even if the road is less than one mile long. Section 4.3 describes some of the possible sampling apparatus used to collect the grab samples. Grab samples shall not contain underlying soils. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Bruan mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

5.3.2 Serpentine Aggregate Covered Areas

A serpentine aggregate-covered play yard or parking lot shall be characterized by taking grab samples from a minimum of three randomly chosen locations per acre. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling plan. A minimum of three samples shall be taken even if the road is less than one mile long. Section 4.3 describes some of the possible sampling apparatus used to collect the grab samples. Grab samples shall not contain underlying soils. Each of the grab samples shall be

placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Bruan mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

5.3.3 Serpentine Aggregate Covered Road Shoulders

The sampling procedure specified in Section 5.3.1 or 5.3.2 shall be used for road shoulders covered with serpentine aggregate. The only difference is that a minimum of three grab samples shall be taken over a length of two miles of shoulder or over an area of two acres of shoulder surface. The word shoulder is meant to imply shoulders on both sides of the road. For serpentine aggregated covered shoulders, the sampling plan specified in Section 5 shall indicate whether the samples are collected on a two mile or two acre basis.

6 SAMPLING LOG

A sample log must be kept showing:

- 1) A unique sample number.
- 2) Facility name.
- 3) Facility address or location where sample is taken.
- 4) A rough sketch, video tape, or photograph of the specific sampling locations.
- 5) Date and time of sampling.
- 6) Name of person performing sampling.

7 ANALYTICAL PROCEDURES

7.1 Principle and Applicability.

Samples of serpentine aggregate taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.^{2,3} A light microscope equipped with two polarizing filters coupled with dispersion staining is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and color are also observed. A retardation plate is placed in the polarized light path for

determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (cross polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting³ and the interpretation of point count data⁴ is available.

This method is applicable to all bulk samples of serpentine aggregate submitted for identification and quantification of asbestos components.

7.2 Range.

The analytical method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is 0.25 percent.

7.3 Interferences.

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos content. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in the identification.

7.4 Analytical Uncertainty.

The uncertainty method is two percent if twenty asbestos fibers are counted in a sample of 400 particles. The uncertainty of the analytical method may be assessed by a 95% confidence interval for the true percentage of asbestos fibers in the rock. The number of asbestos fibers in the sample is assumed to have a binomial distribution. If twenty asbestos fibers are found in a sample of 400 particles, a one-sided confidence interval for the true percentage has an upper bound of seven percent or an analytical uncertainty of two percent.¹¹ The confidence interval used here is an "exact" interval computed directly from the binomial distribution.

7.5 Apparatus.

7.5.1 Microscope. A low-power binocular microscope, preferable stereoscopic, is used to examine the bulk sample as received.

- * Microscope: binocular, 10-45X
- * Light Source: incandescent, fluorescent, halogen or fiber optic
- * Forceps, Dissecting Needles, and Probes
- * Glassine Paper, Clean Glass Plate, or Petri dish

- * Compound Microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris
- * Polarized Light Microscope: described above
- * Objective Lenses: 10X
- * Dispersion Staining Objective Lens: 10X
- * Ocular Lens: 10X
- * Eyepiece Reticule: 25 point or 100 point Chalkley Point Array or cross-hair
- * Compensator Plate: 550 millimicron retardation
- * First Order Red I Compensator: 530 nanometers

7.6 Reagents.

Refractive Index Liquids: 1.490 - 1.570, 1.590 - 1.720 in increments of 0.002 or 0.004.

Refractive Index Liquids for Dispersion Staining: High-dispersion series, 1.550, 1.605, 1.630 (optical).

UICC Asbestos Reference Sample Set: Available from UICC MRC Pneumoconiosis Unit, Lisindough Hospital Penarth, Glamorgan CF6 1xw, UK and commercial distributors.

Tremolite-asbestos: Available from J. T. Baker.

Actinolite-asbestos: Available from J. T. Baker.

Chrysotile, Amosite, and Crocidolite is available from the National Institute of Standards and Technology.

Anthrophyllite, Tremolite, Actinolite will be available from the National Institute of Standards and Technology during the first quarter of 1990.

8 PROCEDURES

Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All samples and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure) exhausting through an HEPA filter. Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

8.1 Sample Preparation.

An aliquot of bulk material is removed from the one pint sample container. The aliquot is spread out on a glass slide. A drop of staining solution with appropriate refractive index is added to the aliquot. A cover slide is placed on top of the sample slide.

The first preparation should use the refractive index solution for Chrysotile. If during the identification phase other asbestiforms are suspected to be present in the sample, due to their morphology, then additional analyses shall be performed with the appropriate solutions. Report the percentages of each asbestiform and combine percentages to determine total asbestos concentrations.

8.2 Fiber Identification.

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology (3 to 1 minimum aspect ratio)
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 3 lists the above properties for commercial asbestos fibers. Natural variations in the conditions under which deposits of asbestiform minerals are formed will occasionally produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Becke line test or dispersion staining shall be used to identify asbestos fibers. Central stop dispersion staining colors are presented in Table 4. Available high-dispersion (HD) liquids should be used.

8.3 Quantification of Asbestos Content.

Asbestos quantification is performed by a point-counting procedure. An ocular reticle (point array) or cross-hair is used to visually superimpose points on the microscope field of view. The point counting rules are as follows:

1. Record the number of points positioned directly above each particle or fiber.
2. Record only one point if two points are positioned over same particle or fiber.
3. Record the number of points positioned on the edge of a particle or fiber.
4. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories.
5. If a test point lies over an ambiguous structure, no particle or fiber is recorded. Examples of "ambiguous" structures are:
 - a) fibers whose dispersion colors are difficult to see
 - b) structures too small to categorize.
6. A fiber mat or bundle is counted as one fiber.

For the purpose of the method, "asbestos fibers" are defined as mineral fibers having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 3.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forceps samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25 - 50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

a reticle with 100 points (Chalkley Point Array) and counting 25 points in at least two randomly selected fields.

or

a reticle with 25 points (Chalkley Point Array) and counting at least two randomly selected fields.

or

a reticle with a standard cross-hair and counting at least 50 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X. Confirmation of the quantitation result by a second analyst on 10 percent of the analyzed samples should be used as standard quality control procedure. All optical properties in Section 8.2 shall be determined to positively identify asbestos.

EXCEPTION I

If the sample is suspected of containing no asbestos a visual technique can be used to report that the sample does not contain asbestos. The rules are as follows:

1. Prepare three slides as described in Section 8.3.
2. View 10 fields per preparation. Identify all fibers.
3. If all fibers are nonasbestos, report no asbestos were found and that visual technique was used.
4. If one fiber is determined to be asbestos, discontinue the visual method and perform the point counting technique as described above.

EXCEPTION II

If the sample is suspected to have an asbestos content in excess of ten percent, a visual technique can be used to report that the sample contains greater than ten percent asbestos. The standard operating procedure of the visual technique allowed in the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program, Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTIR 88-3879 dated October 1988, which is incorporated herein by reference, shall be followed.

9 CALCULATIONS

The percent asbestos is calculated as follows:

$$\% \text{ asbestos} = \left(\frac{a}{n} \right) 100\%$$

Where:

- a = number of asbestos counts
- n = number of nonempty points counted (400)
- If a = 0, report "No asbestos detected."
- If a > 0, report the calculated value to the nearest 0.25%

If "no asbestos detected" is reported by the point counting technique, the analyst may report the observation of asbestos fibers in the non-counted portions of the sample.

10 ALTERNATIVE METHODS

10.1 Alternative Sampling Methods.

Alternative sampling methods may be used as long as they are substantially equivalent to the sampling methods discussed in Section 5 and approved by the Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

10.2 Analytical Methods.

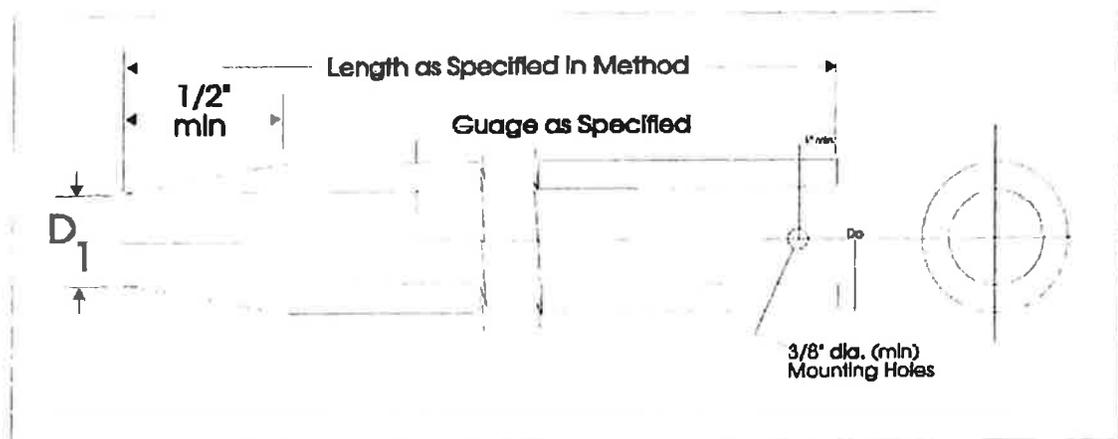
An alternative analytical method may be used as long as it produces results substantially equivalent to the results produced by the point counting method and approved by the Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

11 REFERENCES

- 11.1 G. S. Koch, Jr., R. F. Link. Statistical Analysis of Geological Data. New York. Dover Publications, Inc. December 1985.
- 11.2 Paul F. Kerr. Optical Mineralogy, 4th ed. New York. McGraw-Hill. 1977.
- 11.3 E.M. Chamot and C. W. Mason. Handbook of Chemical Microscopy, Volume One, 3rd ed. New York. John Wiley & Sons. 1958.

- 11.4 F. Chayes. *Petrographic Model Analysis: An Elementary statistical Appraisal*. New York. John Wiley & Sons. 1958.
- 11.5 E. P. Brantly, Jr., K. W. Gold, I. E. Myers, and D. E. Lentzen. *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method*. U. S. Environmental Protection Agency. October 1981.
- 11.6 U. S. Environmental Protection Agency. *Asbestos-Containing Materials in School Buildings: A Guidance Document, Parts 1 and 2* EPA/OTS No. C00090m Narcg. 1979.
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- 11.8 D. H. Taylor and J. S. Bloom. *Hexametaphosphate Pretreatment of Insulation Samples for Identification of Fibrous Constituents*. *Microscope*, 28. 1980.
- 11.9 W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg. *Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization*. U. S. Bureau of Mines Information Circular 8751. 1977.
- 11.10 Walter C. McCrone. *Asbestos Particle Atlas*. Ann Arbor. Ann Arbor Science Publishers. June 1980.
- 11.11 John Moore. *Biostatistician*. Personnel Communication. February 8, 1990.

Figure 1
Thin Wall Tube for Sampling



- Note 1** Minimum of two mounting holes on opposite sides for 2 to 3 inch diameter sampler.
- Note 2** Minimum of four mounting holes spaced a 90° for samplers 4 inch diameter and larger.
- Note 3** Tube held with hardened screws.
- Note 4** Two inch outside-diameter tubes are specified with an 18-gauge wall thickness to comply with area ratio criteria accepted for "undisturbed samples." Users are advised that such tubing is difficult to locate and can be extremely expensive in small quantities. Sixteen-gauge tubes are generally readily available.

Table 1
Suitable Thin Walled Steel Sample Tube^A

OUTSIDE DIAMETER:			
	2	3	5
inches			
millimeters	50.8	76.2	127
WALL THICKNESS:			
	18	16	11
Bwg			
inches	0.049	0.065	0.120
millimeters	1.24	1.65	3.05
TUBE LENGTH:			
	36	36	54
inches			
meters	0.91	0.91	1.45
CLEARANCE RATIO, %	1	1	1

^A The three diameters recommended in Table 1 are indicated for purposes of standardization, and are not intended to indicate that sampling tubes of intermediate or larger diameters are not acceptable. Lengths of tubes shown are illustrative. Proper lengths to be determined as suited to field conditions.

Table 2
Dimensional Tolerances for Thin Walled Tubes

Nominal Tube Diameters from Table 1 ^A Tolerances, inches			
Size Outside Diameter	2	3	4
Outside Diameter	+0.007 -0.000	+0.010 -0.000	+0.015 -0.000
Inside Diameter	+0.000 -0.007	+0.000 -0.010	+0.000 -0.015
Wall Thickness	+0.007	+0.010	+0.015
Ovality	0.015	0.020	0.030
Straightness	0.030/ft	0.030/ft	0.030/ft

^A Intermediate or larger diameters should be proportional. Tolerances shown are essentially standard commercial manufacturing tolerances for seamless steel mechanical tubing. Specify only two of the first three tolerances; O. D. and I. D. or O. D. and Wall, or I. D. and Wall.

Figure 2

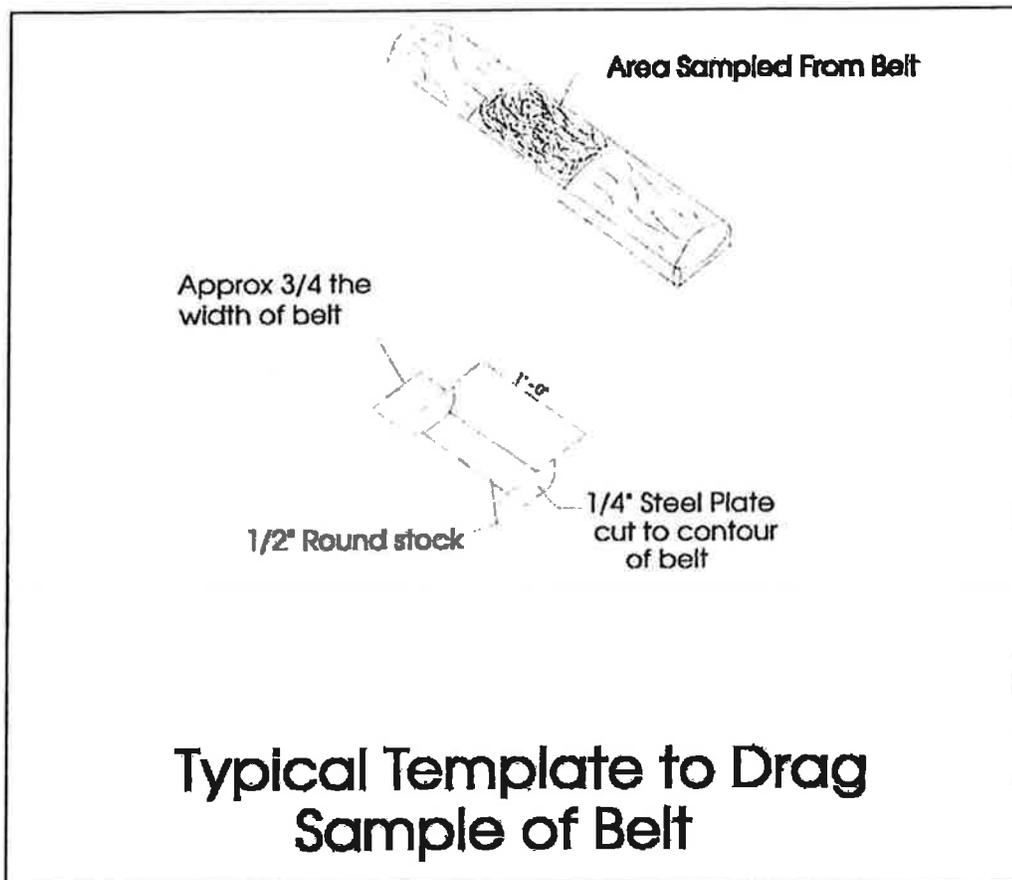


Table 3

Optical Properties of Asbestos Fibers

Mineral	Morphology ^a , color	Refractive Indices ^b		Birefringence	Extinction	Sign of Elongation
		alpha	gamma			
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have spayed ends and "kinks." Aspect ratio typically >10:1. Colorless ^c , nonpleochroic.	1.493 - 1.560	1.517 - 1.562 ^f (normally 1.556)	0.002 - 0.014	to fiber length	+
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635 - 1.696	1.655 - 1.729 ^f (normally 1.696 - 1.710)	0.020 - 0.33	to fiber length	+
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654 - 1.701	1.668 - 1.717 ^e (normally close to 1.700)	0.014 - 0.016	to fiber length	-
Anthophyllite-asbestos	Straight fibers and fiber bundles showing spayed ends. Colorless to light brown. pleochroic absent.	1.596 - 1.652	1.615 - 1.676 ^f	0.019 - 0.024	to fiber length	+
Tremolite-actinolite-asbestos	Straight and curved fibers _d and fiber bundles. Large bundles show spayed ends. Tremolite is colorless and actinolite is green. Weakly to moderately pleochroic.	1.599 - 1.668	1.622 - 1.688 ^f	0.023 - 0.020	to fiber length	+

^a From Reference 6; colors cited are seen by observation with plane polarized light.

^b From Reference 7 and 9.

^c Fibers subjected to heating may be brownish.

^d Fibers defined as having aspect ratio >3:1.

^e ⊥ to fiber length.

^f || to fiber length.

Table 4
Central Stop Dispersion Staining Colors^a

<u>Mineral</u>	<u>RI Liquid</u>	<u>nu </u>	<u>nu </u>
Chrysotile	1.550HD	blue	blue-magenta
Amosite	1.680	blue-magenta to pale blue	golden-yellow
	1.550HD	yellow to white	yellow to white
Crocidolite ^b	1.700	red-magenta	blue-magenta
	1.550HD	yellow to white	yellow to white
Anthophyllite	1.605HD	blue	gold to gold-magenta
Tremolite	1.605HD^o	pale blue	yellow
Actinolite	1.630HD	gold-magenta to blue	gold
	1.630HD ^c	magenta	golden-yellow

- ^a From Reference 11.10.
- ^b Blue absorption color.
- ^c Oblique extinction view.