ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Method 8081 may be used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). The compounds listed below may be determined by either a single- or dual-column analysis system.

Compound	CAS Registry No.
Aldrin	309-00-2
"-BHC	319-84-6
$-BHC	319-85-7
(-BHC (Lindane)	58-89-9
*-BHC	319-86-8
"-Chlordane	5103-71-9
(-Chlordane	5103-74-2
Chlordane - not otherwise specified (n.o.s.)	57-74-9
Chlorobenzilate	510-15-6
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Diallate	2303-16-4
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
Isodrin	465-73-6
Methoxychlor	72-43-5
Toxaphene	8001-35-2

1.2 Method 8081 no longer includes PCBs as Aroclors in the list of target analytes. The analysis of PCBs should be undertaken using Method 8082, which includes specific cleanup and quantitation procedures designed for PCB analysis. This change was made to obtain PCB data of better quality and to eliminate the complications inherent in a combined organochlorine pesticide and PCB method. Therefore, if the presence of PCBs is suspected, use Method 8082 for PCB analyses, and this method (Method 8081) for the organochlorine pesticides. If there is no information on the likely presence of PCBs, either employ a PCB-specific screening procedure such as an immunoassay (e.g., Method 4020), or split the sample extract *prior to* any cleanup steps, and process part of the extract for organochlorine pesticide analysis and the other portion for PCB analysis using Method 8082.

1.3 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.).

1.4 Although performance data are presented for many of the target analytes, it is unlikely that all of them could be determined in a single analysis. The chemical and chromatographic behaviors of many of these chemicals can result in coelution of some target analytes. Several cleanup/fractionation schemes are provided in this method and in Method 3600.

1.5 Several multi-component mixtures (i.e., chlordane and toxaphene) are listed as target analytes. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" multi-component mixtures that may have significant differences in peak patterns than those of standards.

1.6 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 Method 8270 is also recommended as a confirmation technique, if sensitivity permits (see Sec. 7.7). GC/AED may also be used as a confirmation technique, if sensitivity permits (See Method 8085).

1.7 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.

1.8 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, 5000, 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, Sec. 2.1, 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 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.10 Extracts suitable for analysis by this method may also be analyzed for organophosphorus pesticides (Method 8141). Some extracts may also be suitable for triazine herbicide analysis, if low recoveries (normally samples taken for triazine analysis must be preserved) are not a problem.

1.11 The following compounds may also be determined using this method. They have been grouped separately from the compounds in Sec. 1.1 because they have not been as extensively validated by EPA. If these compounds are to be determined using this procedure, the analyst is advised that additional efforts may be needed in order to optimize the instrument operating conditions and to demonstrate acceptable method performance.

Compound	CAS Registry No.
Alachlor	15972-60-8
Captafol	2425-06-1
Carbophenthion	786-19-6
Chloroneb	2675-77-6
Chloropropylate	5836-10-2
Chlorothalonil	1897-45-6
Dacthal (DCPA)	1861-32-1
Dichlone	117-80-6
Dichloran	99-30-9
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Mirex	2385-85-5
Nitrofen	1836-75-5
Pentachloronitrobenzene (PCNB)	82-68-8
Permethrin (*cis* + *trans*)	52645-53-1

Compound	CAS Registry No.
Perthane	72-56-0
Propachlor	1918-16-7
Strobane	8001-50-1
trans-Nonachlor	39765-80-5
Trifluralin	1582-09-8

1.12 Kepone extracted from samples or in standards exposed to water or methanol may produce peaks with broad tails that elute later than the standard by up to 1 minute. This shift is presumably the result of the formation of a hemi-acetal from the ketone functionality and may seriously affect the ability to identify this compound on the basis of its retention time. As a result, Method 8081 is not recommended for determining Kepone. Method 8270 may be more appropriate for the analysis of Kepone.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 to 30 g for solids) 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.

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.

2.2 A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Suggested cleanups include alumina (Method 3610), Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660).

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

3.0 INTERFERENCES

3.1 Refer to Methods 3500 (Sec. 3.0, in particular), 3600, and 8000 for a discussion of interferences.

3.2 Sources of interference in this method can be grouped into three broad categories.

3.2.1 Contaminated solvents, reagents, or sample processing hardware.

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

3.2.3 Compounds extracted from the sample matrix to which the detector will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. 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.

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

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

3.3.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

3.3.3 Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.3.4 These materials may be removed prior to analysis using Method 3640 (Gel Permeation Cleanup) or Method 3630 (Silica Gel Cleanup).

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

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 130EC for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment. (Other appropriate glassware cleaning procedures may be employed).

3.5 The presence of sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur. Since the recovery of endrin aldehyde is drastically reduced when using the TBA procedure in Method 3660, this compound must be determined prior to sulfur cleanup when it is an analyte of interest and the TBA procedure is to be used for cleanup. Endrin aldehyde is not affected by the copper powder, so endrin aldehyde can be determined after the removal of sulfur using the copper powder technique in Method 3660. However, as indicated in Method 3660, the use of copper powder may adversely affect the recoveries of other potential analytes of interest, including some organochlorine compounds and many organophosphorous compounds.

3.6 Waxes, lipids, and other high molecular weight materials can be removed by gel permeation chromatography (GPC) cleanup (Method 3640).

3.7 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain coeluting organophosphorus pesticides are eliminated using Method 3640 (GPC - pesticide option). Coeluting chlorophenols may be eliminated by using Method 3630 (silica gel), Method 3620 (Florisil), or Method 3610 (alumina). Polychlorinated biphenyls (PCBs) also may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as chlordane, toxaphene, and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult Methods 3620 and 3630 for techniques that may be used to separate the pesticides from the PCBs.

3.8 Coelution among the many target analytes in this method can cause interference problems. The following target analytes may coelute on the GC columns listed, when using the single-column analysis scheme:

DB 608	Trifluralin/diallate isomers
	PCNB/dichlone/Isodrin
DB 1701	Captafol/mirex
	Methoxychlor/endosulfan sulfate

3.9 The following compounds may coelute using the dual-column analysis scheme. In general, the DB-5 column resolves fewer compounds than the DB-1701.

DB-5	Permethrin/heptachlor epoxide
	Endosulfan I/"-chlordane
	Perthane/endrin
	Endosulfan II/chloropropylate/chlorobenzilate
	4,4'-DDT/endosulfan sulfate
	Methoxychlor/dicofol
DB-1701	Chlorothalonil/$-BHC
	*-BHC/DCPA/permethrin
	"-Chlordane/*trans*-nonachlor

Nitrofen, dichlone, carbophenothion, and dichloran exhibit extensive peak tailing on both columns. Simazine and atrazine give poor responses on the ECD detector. Triazine compounds should be analyzed using Method 8141 (NPD option).

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required 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 detectors.

4.2 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. 7.7 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 to develop the method performance data presented in Sec. 9. Listing these columns in this method is not intended to exclude the use of other columns that may be developed. Laboratories may use other capillary columns or columns of other dimensions, provided that they document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that meet the data quality needs of the intended application.

4.2.1 Narrow-bore columns for single-column analysis (use both columns to confirm compound identifications unless another confirmation technique such as GC/MS is employed).

4.2.1.1 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-µm film thickness.

4.2.1.2 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 µm coating thickness, 1-µm film thickness.

4.2.1.3 Narrow-bore columns should be installed in split/splitless (Grob-type) injectors.

4.2.2 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.

4.2.2.1 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-µm or 0.83-µm film thickness.

4.2.2.2 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

4.2.2.3 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 95 percent dimethyl - 5 percent diphenyl polysiloxane (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

4.2.3 Wide-bore columns for dual-column analysis - the two pairs of recommended columns are listed below.

4.2.3.1 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-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0-µm 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 seconds to soften the polyimide coating.

4.2.3.2 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-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

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

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

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

5.0 REAGENTS

5.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall 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.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 EC in polytetrafluoroethylene (PTFE)-sealed containers, in the dark. When a lot of standards

is prepared, it is recommended that aliquots of that lot be stored in individual small vials. All stock standard solutions must be replaced after one year, or sooner if routine QC tests (see Sec. 8.0) indicate a problem. All other standard solutions must be replaced after six months, or sooner if routine QC (see Sec. 8.0) indicates a problem.

5.2 Solvents used in the extraction and cleanup procedures (see appropriate 3500 and 3600 series methods) include *n*-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane) and they 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 quality or equivalent, and each lot of solvent should be determined to be free of phthalates.

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

5.3.1 Acetone, $(CH_3)_2CO$

5.3.2 Toluene, $C_6H_5CH_3$

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

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

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

5.5.2 $-BHC, dieldrin, and some other standards may not be adequately soluble in isooctane. A small amount of acetone or toluene should be used to dissolve these compounds during the preparation of the stock standard solutions.

5.6 Composite stock standard - May be prepared from individual stock solutions.

5.6.1 For composite stock standards containing less than 25 components, take exactly 1 mL of each individual stock solution at a concentration of 1000 mg/L, add solvent, and mix the solutions in a 25-mL volumetric flask. For example, for a composite containing 20 individual standards, the resulting concentration of each component in the mixture, after the volume is adjusted to 25 mL, will be 1 mg/25 mL. This composite solution can be further diluted to obtain the desired concentrations.

5.6.2 For composite stock standards containing more than 25 components, use volumetric flasks of the appropriate volume (e.g., 50 mL, 100 mL), and follow the procedure described above.

5.7 Calibration standards should be prepared at a minimum of five different concentrations by dilution of the composite 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.

5.7.1 Although all single component analytes can be resolved on a new 35 percent phenyl methyl silicone column (e.g., DB-608), two calibration mixtures should be prepared for the single component analytes of this method. This procedure is established to minimize potential resolution and quantitation problems on confirmation columns or on older 35 percent phenyl methyl silicone (e.g. DB-608) columns and to allow determination of endrin and DDT breakdown for instrument quality control (Sec. 8.0).

5.7.2 Separate calibration standards are required for each multi-component target analyte (e.g., toxaphene and chlordane). Analysts should evaluate the specific toxaphene standard carefully. Some toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results.

5.8 Internal standard (optional)

5.8.1 Pentachloronitrobenzene is suggested as an internal standard for the single-column analysis, when it is not considered to be a target analyte. 1-Bromo-2-nitrobenzene may also be used. Prepare a solution of 5000 mg/L (5000 ng/µL) of pentachloronitrobenzene or 1-bromo-2-nitrobenzene. Spike 10 µL of this solution into each 1 mL of sample extract.

5.8.2 1-Bromo-2-nitrobenzene is suggested as an internal standard for the dual-column analysis. Prepare a solution of 5000 mg/L (5000 ng/µL) of 1-bromo-2-nitrobenzene. Spike 10 µL of this solution into each 1 mL of sample extract.

5.9 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 following compounds are recommended as possible surrogates.

5.9.1 Decachlorobiphenyl and tetrachloro-*m*-xylene have been found to be a useful pair of surrogates for both the single-column and dual-column configurations. Method 3500, Sec. 5.0, describes the procedures for preparing these surrogates.

5.9.2 4-Chloro-3-nitrobenzotrifluoride may also be useful as a surrogate if the chromatographic conditions of the dual-column configuration cannot be adjusted to preclude coelution of a target analyte with either of the surrogates in Sec. 5.8.1. However, this compound elutes early in the chromatographic run and may be subject to other interference problems. A recommended concentration for this surrogate is 500 ng/µL. Use a spiking volume of 100 µL for a 1-L aqueous sample.

5.9.3 Store surrogate spiking solutions at 4EC in PTFE-sealed containers in the dark.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Four, Sec. 4.1, for sample collection and preservation instructions.

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

7.0 PROCEDURE

7.1 Sample extraction

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), or other appropriate technique. Solid samples may also be extracted using supercritical fluid extraction (Method 3562).

NOTE: Hexane-acetone (1:1) may be more effective as an extraction solvent for organochlorine pesticides in some environmental and waste matrices than is methylene chloride-acetone (1:1). Relative to the methylene chloride-acetone mixture, the use of hexane-acetone generally reduces the amount of interferences that are extracted and improves the signal-to-noise ratio.

The choice of extraction solvent will depend on the analytes of interest. No single solvent is universally applicable to all analyte groups. The analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest, for any solvent system employed, *including* those specifically listed in this method. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Sec. 8.2 of 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.

7.2 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. General guidance for sample extract cleanup is provided in this section and in Method 3600.

7.2.1 If a sample is of biological origin, or contains high molecular weight materials, the use of Method 3640 (GPC - pesticide option) is recommended. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel, or Florisil) may also be required following the GPC cleanup.

7.2.2 Method 3610 (alumina) may be used to remove phthalate esters.

7.2.3 Method 3620 (Florisil) may be used to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

7.2.4 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

7.2.5 Sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in Method 3660.

7.3 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 may be developed. Wide-bore or narrow-bore columns may be used with either option. Laboratories may use other capillary columns or columns of other dimensions, provided that they document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that meet the data quality needs of the intended application.

7.3.1 Single-column analysis

This capillary GC/ECD method allows the analyst the option of using 0.25-0.32-mm ID capillary columns (narrow-bore) or 0.53-mm ID capillary columns (wide-bore). Performance data are provided for both options. Figures 1 - 6 provide example chromatograms. Columns with diameters other than these may be used provided that the analyst can demonstrate the ability to generate data that meets the data quality requirements for the intended application.

7.3.1.1 Narrow-bore columns generally provided greater chromatographic resolution than wide-bore columns, although narrow-bore columns have a lower sample capacity. As a result, narrow-bore columns may be more suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53-mm ID) may be more suitable for more complex environmental and waste matrices. However, the choice of the appropriate column diameter is left to the analyst.

7.3.1.2 Table 1 lists example retention times for the target analytes using wide-bore capillary columns. Table 2 lists example retention times for the target analytes using narrow-bore capillary columns.

7.3.1.3 Table 4 lists suggested GC operating conditions for the single-column method of analysis.

7.3.2 Dual-column analysis

The dual-column/dual-detector approach recommends the use of two 30-m x 0.53-mm ID fused-silica open-tubular columns of different polarities, thus, different selectivities towards the target analytes. The columns are connected to an injection tee and separate electron capture detectors. However, the choice of the appropriate column dimensions is left to the analyst.

7.3.2.1 Example retention times for the organochlorine analytes on dual-columns are provided in Table 6. The suggested GC operating conditions for the compounds in Table 6 are given in Table 7.

7.3.2.2 Multi-component mixtures of toxaphene and Strobane were analyzed separately (Figures 5 and 6) using the operating conditions in Table 7.

7.3.2.3 Figure 6 is an example chromatogram for a mixture of organochlorine pesticides. The retention times of the individual components detected in these mixtures are given in Table 6, and are provided as examples.

7.3.2.4 Suggested operating conditions for a more heavily loaded DB-5/DB-1701 pair are given in Table 8. This column pair was used for the detection of multi-component organochlorine compounds.

7.3.2.5 Suggested operating conditions for a DB-5/DB-1701 column pair with thinner films, a different type of splitter, and a slower temperature programming rate are provided in Table 7. These conditions gave better peak shapes for nitrofen and dicofol. Table 6 lists the retention times for the compounds on this column pair.

7.4 Calibration

7.4.1 Prepare calibration standards using the procedures in Sec. 5.0. Refer to Method 8000 (Sec. 7.0) for proper calibration techniques for both initial calibration and calibration verification. The procedure for either internal or external calibration may be used. In most cases, external standard calibration is used with Method 8081 because of the sensitivity of the electron capture detector and the probability of the internal standard being affected by interferences. Because several of the pesticides may coelute on any single column, analysts should use two calibration mixtures (see Sec. 3.8). The specific mixture should be selected to minimize the problem of peak overlap.

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

7.4.1.1 Unless otherwise necessary for a specific project, the analysis of the multi-component analytes employs a single-point calibration. A single calibration standard near the mid-point of the expected calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column. The calibration standard may be at a lower concentration than the mid-point of the expected range, if appropriate for the project.

7.4.1.2 For calibration verification (each 12-hour shift) all target analytes required in the project plan must be injected.

7.4.2 Establish the GC operating conditions appropriate for the configuration (single-column or dual column, see Sec. 7.3) using Tables 4, 5, 7, or 8, as guidance. Optimize the instrumental conditions for resolution of the target analytes and sensitivity. An initial oven temperature of #140 -150EC may be required to resolve the four BHC isomers. A final temperature of 240 - 270EC may be required 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.

7.4.3 A 2-µL injection volume 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.

7.4.4 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column should be primed (or deactivated) by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

CAUTION: Several analytes, including aldrin, may be observed in the injection just following this system priming because of carry-over. Always run an acceptable blank prior to running any standards or samples.

7.4.5 Calibration factors

When external standard calibration is employed, calculate the calibration factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, using the formulae below. If internal standard calibration is employed, refer to Method 8000 for the calculation of response factors.

7.4.5.1 Calculate the calibration factor for each analyte at each concentration as:

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

7.4.5.2 Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where n is the number of standards analyzed.

7.4.5.3 Calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{CF}} \times 100$$

If the RSD for each analyte is # 20%, then the response of the instrument is considered linear and the mean calibration factor may be used to quantitate sample results. If the RSD is greater than 20%, the analyst should consult Method 8000 for other calibration options, which may include: the grand mean RSD approach, a linear calibration not through the origin, or a non-linear calibration model (e.g., a polynomial equation).

7.4.6 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 they are appropriate for the intended application.

7.4.6.1 Before establishing the retention time windows, make sure the gas chromatographic system is operating within optimum conditions.

7.4.6.2 The widths of the retention time windows are defined as described in Method 8000. However, the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

7.5 Gas chromatographic analysis of sample extracts

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

7.5.2 Verify calibration at least once each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. Analysts should alternate the use of high and low concentration mixtures of single-component analytes and multi-component analytes for calibration verification. 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 re-injection when QC limits are

exceeded) and at the end of the analysis sequence. See Sec. 8.4.4 for additional guidance on the frequency of the standard injections.

7.5.2.1 The calibration factor for each analyte should not exceed a ± 15 percent difference from the mean calibration factor calculated for 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 Sec. 7 of Method 8000 for the specific details of calibration verification.

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

7.5.2.2 If the calibration factor for any analyte exceeds a ±15 percent difference, use the approach described in Sec. 7 of Method 8000 to calculate the average percent difference across all analytes. If the average of the responses for all analytes is within ±15%, then the calibration has been verified. However, the conditions in Sec. 7 of Method 8000 also apply, e.g., the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the ±15% limit.

7.5.2.3 If the calibration does not meet the ±15% limit (either on the basis of each compound or the average across all compounds), 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 ±15%, then a new initial calibration must be prepared. The effects of a failing calibration verification standard on sample results are discussed in Sec. 7.5.7.

7.5.3 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in Sec. 7.4.6. Each analyte in each subsequent standard run during the 12-hour period must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established. As noted in Sec. 7.4.6, other approaches to compound identification may be employed, provided that the analyst can demonstrate and document that they are appropriate for the intended application.

7.5.4 Inject a measured aliquot of the concentrated sample extract. A 2-µL aliquot is suggested, however 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.

7.5.5 Tentative identification of an analyte (either single-component or multi-component) 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. See Method 8000 for information on confirmation of tentative identifications. See Sec. 7.7 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 Sec. 7 of Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

7.5.6 When using the external calibration procedure (Method 8000), determine the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes, as follows. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined.

7.5.6.1 For aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (µL).

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

&& = Mean calibration factor from the initial calibration (area/ng).

V_i = Volume of the extract injected (µL). The injection volume for samples and calibration standards should be the same, unless the analyst can demonstrate acceptable performance using different volumes or conditions.

V_s = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.

7.5.6.2 For non-aqueous samples

$$\text{Concentration } (\mu g/kg) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(W_s)}$$

where A_x, V_t, D, **&&**, and V_i are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.

7.5.6.3 See Method 8000 for the equation used for internal standard quantitation.

7.5.6.4 If the responses exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.5.6.5 If partially overlapping or coeluting peaks are found, change GC columns or try GC/MS quantitation (see Sec. 8.0 and Method 8270).

7.5.7 Each sample analysis employing external standard calibration must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration verification criteria in Sec. 7.5.2.

Although analysis of a single mid-concentration standard (standard mixture or multi-component analyte) will satisfy the minimum requirements, analysts are urged to use different calibration verification standards during organochlorine pesticide analyses. Also, multi-level standards (mixtures or multi-component analytes) are highly recommended to ensure that the detector response remains stable for all the 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 last 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., >15%, 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, as the verification standard has demonstrated that the analyte would have been detected were it 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 15% below the initial calibration response, then reinjection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present (i.e., a false negative result).

7.5.8 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 and/or quantitative QC criteria are exceeded.

7.5.9 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 >30 seconds 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.

7.5.10 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.

7.5.11 Use the calibration standards analyzed during the sequence to evaluate retention time stability. Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

7.5.12 The identification of mixtures (i.e., chlordane and toxaphene) is not based on a single peak, but rather on the characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks. Quantitation is based on the areas of the characteristic peaks as compared to the areas of the corresponding peaks at the same retention times in the calibration standard, using either internal or external calibration procedures. See Method 8000 for information on confirmation of tentative identifications. See Sec. 7.7 of this procedure for information on the use of GC/MS as a confirmation technique.

7.5.13 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines), cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun 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.

7.6 Quantitation of multi-component analytes - Multi-component analytes present problems in measurement. Suggestions are offered in the following sections for handling toxaphene, Strobane, chlordane, BHC, and DDT.

7.6.1 Toxaphene and Strobane - Toxaphene is manufactured by the chlorination of camphenes, whereas Strobane results from the chlorination of a mixture of camphenes and pinenes. Quantitation of toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate toxaphene from GC/ECD results:

7.6.1.1. Adjust the sample size so that the major toxaphene peaks are 10-70% of full-scale deflection (FSD).

7.6.1.2 Inject a toxaphene standard that is estimated to be within ± 10 ng of the sample amount.

7.6.1.3 Quantitate toxaphene using the total area of the toxaphene pattern or using 4 to 6 major peaks.

7.6.1.3.1 While toxaphene contains a large number of compounds that will produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. This unresolved complex mixture results in the "hump" in the chromatogram that is characteristic of this mixture. Although the resolved peaks are important for the identification of the mixture, the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

7.6.1.3.2 To measure total area, construct the baseline of toxaphene in the sample chromatogram between the retention times of the first and last eluting toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

7.6.1.3.3 Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. A collaborative study of a series of toxaphene residues evaluated several approaches to quantitation of this compound, including the use of the total area of the peaks in the toxaphene chromatogram and the use of a subset of 4 to 6 peaks. That study indicated that the use of 4 to 6 peaks provides results that agree well with the total peak area approach and may avoid difficulties when interferences with toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT. Whichever approach is employed should be documented and available to the data user, if necessary.

7.6.1.3.4 When toxaphene is determined using the 4 to 6 peaks approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionally larger or smaller in the sample compared to the standard.

7.6.1.3.5 The heights or areas of the 4 to 6 peaks that are selected should be summed together and used to determine the toxaphene concentration. Alternatively, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for the sample.

7.6.2 Chlordane - Technical chlordane is a mixture of at least 11 major components and 30 or more minor components that have been used to prepare specific pesticide formulations. The nomenclature of the various forms of chlordane has been the subject of some confusion in both Agency methods and the open literature for some time. The CAS number for technical chlordane is properly given as 12789-03-6. The two most prevalent

major components of technical chlordane are (-chlordane (or *trans*-chlordane, CAS number 5103-74-2) and "-chlordane (or *cis*-chlordane, CAS number 5103-71-9). The structure represented by (-chlordane has occasionally been referred to by the name $-chlordane, and a separate CAS number of 5566-34-7 has been assigned by CAS to that designation. For the purposes of the RCRA program, the name $-chlordane is not generally used, and this isomer is known as (-chlordane (CAS number 5103-74-2).

The exact percentages of "-chlordane and (-chlordane in the technical material are not completely defined, and are not consistent from batch to batch. Moreover, changes may occur when the technical material is used to prepare specific pesticide formulations. The approach used for evaluating and reporting chlordane results will often depend on the end use of the results and the analyst's skill in interpreting this multicomponent pesticide residue. The following sections discuss three specific options: reporting technical chlordane (CAS number 12789-03-6), reporting chlordane (not otherwise specified, or n.o.s., CAS number 57-74-9), and reporting the individual chlordane components that can be identified under their individual CAS numbers.

7.6.2.1 When the GC pattern of the residue resembles that of technical chlordane, the analyst may quantitate chlordane residues by comparing the total area of the chlordane chromatogram using three to five major peaks or the total area. If the heptachlor epoxide peak is relatively small, include it as part of the total chlordane area for calculation of the residue. If heptachlor and/or heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected chlordane area.

NOTE: Octachloro epoxide, a metabolite of chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GC column.

To measure the total area of the chlordane chromatogram, inject an amount of a technical chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms. Construct the baseline of technical chlordane in the standard chromatogram between the retention times of the first and last eluting toxaphene components. Use this area and the mass of technical chlordane in the standard to calculate a calibration factor. Construct a similar baseline in the sample chromatogram, measure the area, and use the calibration factor to calculate the concentration in the sample.

7.6.2.2 The GC pattern of a chlordane residue in a sample may differ considerably from that of the technical chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the pesticide active ingredient technical chlordane. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable chlordane components as “chlordane (n.o.s.)” under the CAS number 57-74-9.

7.6.2.3 The third option is to quantitate the peaks of "-chlordane, (-chlordane, and heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers.

7.6.2.4 To measure the total area of the chlordane chromatogram, inject an amount of a technical chlordane standard which will produce a chromatogram in

which the major peaks are approximately the same size as those in the sample chromatograms.

7.6.3 Hexachlorocyclohexane - Hexachlorocyclohexane is also known as BHC, from the former name, benzene hexachloride. Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor. It consists of a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes. Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. Quantitate each isomer (" , $, (, and *) separately against a standard of the respective pure isomer.

7.6.4 DDT - Technical DDT consists primarily of a mixture of 4,4'-DDT (approximately 75%) and 2,4'-DDT (approximately 25%). As DDT weathers, 4,4'-DDE, 2,4'-DDE, 4,4'-DDD, and 2,4'-DDD are formed. Since the 4,4'-isomers of DDT, DDE, and DDD predominate in the environment, these are the isomers normally regulated by EPA. Therefore, sample extracts should be quantitated against standards of the respective pure isomers of 4,4'-DDT, 4,4'-DDE, and 4,4'-DDD.

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

7.7.1 Full-scan GC/MS will normally require a concentration of approximately 10 ng/µL in the final extract for each single-component compound. Ion trap or selected ion monitoring will normally require a concentration of approximately 1 ng/µL.

7.7.2 The GC/MS must be calibrated for the specific target pesticides 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 pesticides 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/EC analysis.

7.7.3 GC/MS is not recommended for confirmation when concentrations are below 1 ng/µL in the extract, unless a more sensitive mass spectrometer is employed.

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

7.7.5 If a base/neutral/acid extraction of an aqueous sample was performed for an analysis of semivolatile organics (e.g., Method 8270), then that extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are *not* detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

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

7.8.1 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.

7.8.2 GC injector ports can be of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT break down to endrin aldehyde, endrin ketone, DDD, or DDE. When such breakdown is observed, clean and deactivate the injector port, break off at least 30 cm of the column and remount it. Check the injector temperature and lower it to 205EC, if required. Endrin and DDT breakdown are less of a problem when ambient on-column injectors are used.

7.8.3 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.

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

7.8.3.2 Prepare a solution of a deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. 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.

7.8.4 Column rinsing - The column should be rinsed 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 required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation techniques can be found in Method 3500. If an extract cleanup procedure was performed, refer to Method 3600 for the appropriate quality control procedures. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0, and include evaluation of retention time windows, calibration verification, and chromatographic analysis of samples.

8.2.1 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 verification standard should be within ±15% of the initial calibration (see Sec. 7.5.2). When this calibration verification standard falls out of this acceptance window, the laboratory should stop analyses and take corrective action.

8.2.2 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 seconds requires reanalysis of the affected sample.

8.2.3 DDT and endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and endrin. Presence of 4,4'-DDE, 4,4'-DDD, endrin ketone or endrin indicates breakdown. If degradation of either DDT or endrin exceeds 15%, take corrective action before proceeding with calibration. Unless otherwise specified in an approved project plan, this test should be performed even when DDT and endrin are not target analytes for a given project, as a test of the inertness of the analytical system.

8.2.3.1 Calculate percent breakdown as follows:

$$\%\ \text{breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD \% DDE)}}{\text{sum of all peak areas (DDT \% DDE \% DDD)}} \times 100$$

$$\%\ \text{breakdown of endrin} = \frac{\text{sum of degradation peak areas (aldehyde \% ketone)}}{\text{sum of all peak areas (endrin \% aldehyde \% ketone)}} \times 100$$

8.2.3.2 The breakdown of DDT and endrin should be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound (Sec. 7.8.2).

8.2.4 Whenever silica gel (Method 3630) or Florisil (Method 3620) cleanups are used, the analyst must demonstrate that the fractionation scheme is reproducible. Batch to batch variation in the composition of the silica gel or Florisil or overloading the column may

cause a change in the distribution patterns of the organochlorine pesticides. When compounds are found in two fractions, add the concentrations found in the fractions, and correct for any additional dilution.

8.3 Initial demonstration of proficiency

8.3.1 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. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

8.3.2 It is suggested that the quality control (QC) reference sample concentrate (as discussed in Sec. 8.0 of Methods 8000 and 3500) contain each analyte of interest at 10 mg/L in the concentrate. A 1-mL spike of this concentrate into 1 L of reagent water will yield a sample concentration of 10 µg/L. If this method is to be used for analysis of chlordane or toxaphene only, the QC reference sample concentrate should contain the most representative multi-component mixture at a suggested concentration of 50 mg/L in acetone. See Method 8000, Sec. 8.0 for additional information on how to accomplish this demonstration. Other concentrations may be used, as appropriate for the intended application.

8.3.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

8.4 Sample quality control for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision and accuracy). At a minimum, this includes the analysis of QC samples including a method blank and a laboratory control sample (LCS) in each analytical batch, the addition of surrogates to each field sample and QC sample, and routine analyses of matrix spike and matrix spike duplicate aliquots.

8.4.1 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.

8.4.2 In-house method performance criteria should be developed using the guidance found in Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

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 indicates

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.

8.5 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. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

8.6 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 The method detection limit (MDL) is defined in Chapter One. Each laboratory should develop its own matrix-specific MDLs, if necessary, using the guidance found in Chapter One. Estimated quantitation limits (EQLs) may be determined using the factors in Table 3.

9.2 The chromatographic separations in this method have been tested in a single laboratory by using clean hexane and liquid and solid waste extracts that were spiked with the test compounds at three concentrations. Single-operator precision, overall precision, and method accuracy were found to be related to the concentration of the compound and the type of matrix.

9.3 The accuracy and precision that can be achieved with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

9.4 Tables 9 and 10 contain precision (as %RSD) and accuracy (as % recovery) data generated for sewage sludge and dichloroethane stillbottoms. Table 11 contains recovery data for a clay soil, taken from Reference 10. The spiking concentration for the clay soil was 500 µg/kg. The spiking solution was mixed into the soil and then immediately transferred to the extraction device and immersed in the extraction solvent. The spiked sample was then extracted by Method 3541 (Automated Soxhlet). The data represent a single determination. Analysis was by capillary column gas chromatography/electron capture detector.

9.5 Table 12 contains single-laboratory precision and accuracy data for solid-phase extraction of TCLP buffer solutions spiked at two levels and extracted using Method 3535.

9.6 Table 13 contains multiple-laboratory data for solid-phase extraction of spiked TCLP soil leachates extracted using Method 3535.

9.7 Table 14 contains single-laboratory data on groundwater and wastewater samples extracted by solid-phase extraction, using Method 3535.

9.8 Tables 15 and 16 contain single-laboratory performance data using supercritical fluid extraction (Method 3562). Samples were analyzed by GC/ELCD. The method was performed using a variable restrictor and solid trapping material (octadecyl silane [ODS]). Three different soil samples were spiked at 5 and 250 µg/kg. Soil 1 (Delphi) is described as loamy sand, with 2.4% clay, 94% sand, 0.9% organic matter, 3.4% silt, and 0.1% moisture. Soil 2 (McCarthy) is described as sandy-loam, with 11% clay, 56% sand, 22% organic matter, 33% silt, and 8.7% moisture. Soil 3 (Auburn) is described as clay loam, with 32% clay, 21% sand, 5.4% organic matter, 46% silt, and 2.2% moisture. Seven replicate extractions were made of each soil at the two concentrations.

9.9 Tables 17 to 19 contain single-laboratory accuracy data for chlorinated pesticides extracted by pressurized fluid extraction (Method 3545) from clay, loam, and sand samples spiked by a commercial supplier at three certified concentrations (low, medium, and high). Samples of 10 to14 g were extracted with hexane:acetone (1:1), at 100EC and 2000 psi, using a 5-minute heating time and a 5-minute static extraction. Extract volumes were 13 to 15 mL, and were adjusted prior to GC/EC analysis to match the linear range of the instrumentation. The data are taken from Reference 14, where the PFE results were presented as the percent of the results from an automated Soxhlet (Method 3541) extraction, which were in turn reported as a percent of the certified values.

9.10 Tables 20 and 21 contain single-laboratory accuracy data for chlorinated pesticides extracted from natural soils, glass-fiber, and sand matrices, using microwave extraction (Method 3546). Concentrations of each target analyte ranged between 0.5 to 10 µg/g. Four real-world split samples contaminated with pesticides and creosotes were also used (obtained from US EPA ERT, Edison, NJ). The latter were extracted by an independent laboratory using standard Soxhlet procedures and results compared to those obtained with this procedure. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by Method 8081. Method blanks and five spiked replicates were included. Work was also carried out to assess the level of degradation of thermally labile pesticides and it was found that no significant degradation takes place under the procedure described herein. The data are taken from Reference 15.

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TABLE 1

EXAMPLE GAS CHROMATOGRAPHIC RETENTION TIMES FOR THE ORGANOCHLORINE PESTICIDES USING WIDE-BORE CAPILLARY COLUMNS
SINGLE-COLUMN METHOD OF ANALYSIS

	Retention Time (min)	
Compound	DB-608[a]	DB-1701[a]
Aldrin	11.84	12.50
"-BHC	8.14	9.46
$-BHC	9.86	13.58
*-BHC	11.20	14.39
(-BHC (Lindane)	9.52	10.84
"-Chlordane	15.24	16.48
(-Chlordane	14.63	16.20
4,4'-DDD	18.43	19.56
4,4'-DDE	16.34	16.76
4,4'-DDT	19.48	20.10
Dieldrin	16.41	17.32
Endosulfan I	15.25	15.96
Endosulfan II	18.45	19.72
Endosulfan sulfate	20.21	22.36
Endrin	17.80	18.06
Endrin aldehyde	19.72	21.18
Heptachlor	10.66	11.56
Heptachlor epoxide	13.97	15.03
Methoxychlor	22.80	22.34
Toxaphene	MR	MR

MR = Multiple response compound.
[a] See Table 5 for GC operating conditions used for these analyses.

All data are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 2

EXAMPLE GAS CHROMATOGRAPHIC RETENTION TIMES FOR THE ORGANOCHLORINE PESTICIDES USING NARROW-BORE CAPILLARY COLUMNS SINGLE-COLUMN METHOD OF ANALYSIS

	Retention Time (min)	
Compound	DB-608[a]	DB-5[a]
Aldrin	14.51	14.70
"-BHC	11.43	10.94
$-BHC	12.59	11.51
*-BHC	13.69	12.20
(-BHC (Lindane)	12.46	11.71
"-Chlordane	NA	NA
(-Chlordane	17.34	17.02
4,4'-DDD	21.67	20.11
4,4'-DDE	19.09	18.30
4,4'-DDT	23.13	21.84
Dieldrin	19.67	18.74
Endosulfan I	18.27	17.62
Endosulfan II	22.17	20.11
Endosulfan sulfate	24.45	21.84
Endrin	21.37	19.73
Endrin aldehyde	23.78	20.85
Heptachlor	13.41	13.59
Heptachlor epoxide	16.62	16.05
Methoxychlor	28.65	24.43
Toxaphene	MR	MR

NA = Data not available.
MR = Multiple response compound.
[a] See Table 4 for GC operating conditions.

All data are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 3

FACTORS FOR DETERMINATION OF ESTIMATED QUANTITATION LIMITS[a] (EQLs)
FOR VARIOUS MATRICES

Matrix	Factor
Ground water	10
Low-concentration soil by sonication with GPC cleanup	670
High-concentration soil and sludges by sonication	10,000
Non-water miscible waste	100,000

[a] Laboratories may estimate the quantitation limits of the target analytes in environmental and waste media by generating MDLs in organic-free reagent water and using the following equation (see Sec. 5.0 of Chapter One for information on generating MDL data):

EQL ' [MDL in water] × [factor in this table]

For nonaqueous samples, the factor is on a wet-weight basis. Sample EQLs are highly matrix-dependent. EQLs determined using these factors are provided as guidance and may not always be achievable.

TABLE 4

SUGGESTED GC OPERATING CONDITIONS FOR ORGANOCHLORINE COMPOUNDS
SINGLE-COLUMN ANALYSIS USING NARROW-BORE COLUMNS

Column 1 - 30-m x 0.25 or 0.32-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1-µm film thickness.	
Carrier gas	Helium
Carrier gas pressure	16 psi
Injector temperature	225EC
Detector temperature	300EC
Initial temperature	100EC, hold 2 minutes
Temperature program	100EC to 160EC at 15EC/min, followed by 160EC to 270EC at 5EC/min
Final temperature	270EC

Column 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), 1-µm film thickness.	
Carrier gas	Nitrogen
Carrier gas pressure	20 psi
Injector temperature	225EC
Detector temperature	300EC
Initial temperature	160EC, hold 2 minutes
Temperature program	160EC to 290EC at 5EC/min
Final temperature	290EC, hold 1 min

TABLE 5

SUGGESTED GC OPERATING CONDITIONS FOR ORGANOCHLORINE COMPOUNDS
SINGLE-COLUMN ANALYSIS USING WIDE-BORE COLUMNS

Column 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-µm or 0.83-µm film thickness.

Column 2 - 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

Both Column 1 and Column 2 use the same GC operating conditions.

Carrier gas	Helium
Carrier gas flow rate	5-7 mL/minute
Makeup gas	argon/methane (P-5 or P-10) or nitrogen
Makeup gas flow rate	30 mL/min
Injector temperature	250EC
Detector temperature	290EC
Initial temperature	150EC, hold 0.5 minute
Temperature program	150EC to 270EC at 5EC/min
Final temperature	270EC, hold 10 min

Column 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-µm film thickness.

Carrier gas	Helium
Carrier gas flow rate	6 mL/minute
Makeup gas	argon/methane (P-5 or P-10) or nitrogen
Makeup gas flow rate	30 mL/min
Injector temperature	205EC
Detector temperature	290EC
Initial temperature	140EC, hold 2 min
Temperature program	140EC to 240EC at 10EC/min, hold 5 minutes at 240EC, 240EC to 265EC at 5EC/min
Final temperature	265EC, hold 18 min

TABLE 6

EXAMPLE RETENTION TIMES OF THE ORGANOCHLORINE PESTICIDES[a]
DUAL-COLUMN METHOD OF ANALYSIS

Compound	DB-5 RT (min)	DB-1701 RT (min)
DBCP	2.14	2.84
Hexachlorocyclopentadiene	4.49	4.88
Etridiazole	6.38	8.42
Chloroneb	7.46	10.60
Hexachlorobenzene	12.79	14.58
Diallate	12.35	15.07
Propachlor	9.96	15.43
Trifluralin	11.87	16.26
"-BHC	12.35	17.42
PCNB	14.47	18.20
(-BHC	14.14	20.00
Heptachlor	18.34	21.16
Aldrin	20.37	22.78
Alachlor	18.58	24.18
Chlorothalonil	15.81	24.42
Alachlor	18.58	24.18
$-BHC	13.80	25.04
Isodrin	22.08	25.29
DCPA	21.38	26.11
*-BHC	15.49	26.37
Heptachlor epoxide	22.83	27.31
Endosulfan-I	25.00	28.88
(-Chlordane	24.29	29.32
"-Chlordane	25.25	29.82
trans-Nonachlor	25.58	30.01
4,4'-DDE	26.80	30.40
Dieldrin	26.60	31.20
Perthane	28.45	32.18
Endrin	27.86	32.44
Chloropropylate	28.92	34.14
Chlorobenzilate	28.92	34.42
Nitrofen	27.86	34.42

TABLE 6
(continued)

Compound	DB-5 RT (min)	DB-1701 RT (min)
4,4'-DDD	29.32	35.32
Endosulfan II	28.45	35.51
4,4'-DDT	31.62	36.30
Endrin aldehyde	29.63	38.08
Mirex	37.15	38.79
Endosulfan sulfate	31.62	40.05
Methoxychlor	35.33	40.31
Captafol	32.65	41.42
Endrin ketone	33.79	42.26
Permethrin	41.50	45.81
Kepone	31.10	[b]
Dicofol	35.33	[b]
Dichlone	15.17	[b]
","´-Dibromo-*m*-xylene	9.17	11.51
2-Bromobiphenyl	8.54	12.49

[a] See Table 7 for GC operating conditions.

[b] Not detected at 2 ng per injection.

All data are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 7

SUGGESTED GC OPERATING CONDITIONS FOR ORGANOCHLORINE PESTICIDES
FOR DUAL-COLUMN METHOD OF ANALYSIS
LOW TEMPERATURE, THIN FILM

Column 1:	DB-1701 or equivalent 30-m x 0.53-mm ID 1.0-µm film thickness
Column 2:	DB-5 or equivalent 30-m x 0.53-mm ID 0.83-µm film thickness
Carrier gas	Helium
Carrier gas flow rate	6 mL/minute
Makeup gas	Nitrogen
Makeup gas flow rate	20 mL/min
Injector temperature	250EC
Detector temperature	320EC
Initial temperature	140EC, hold 2 minutes
Temperature program	140EC to 270EC at 2.8EC/min
Final temperature	270EC, hold 1 minute

TABLE 8

SUGGESTED GC OPERATING CONDITIONS FOR ORGANOCHLORINE PESTICIDES
FOR THE DUAL-COLUMN METHOD OF ANALYSIS
HIGH TEMPERATURE, THICK FILM

Column 1:	DB-1701 or equivalent 30-m x 0.53-mm ID 1.0-µm film thickness
Column 2:	DB-5 or equivalent 30-m x 0.53-mm ID 1.5-µm film thickness
Carrier gas:	Helium
Carrier gas flow rate:	6 mL/minute
Makeup gas:	Nitrogen
Makeup gas flow rate:	20 (mL/min)
Injector temperature:	250EC
Detector temperature:	320EC
Initial temperature:	150EC, hold 0.5 min
Temperature program:	150EC to 190EC at 12EC/min, hold 2 min190EC to 275EC at 4EC/min
Final temperature	275EC, hold 10 min

TABLE 9

ANALYTE RECOVERY FROM SEWAGE SLUDGE

	Ultrasonic Extraction		Soxhlet	
Compound	%Recovery	%RSD	%Recovery	%RSD
Hexachloroethane	80	7	79	1
2-Chloronapthalene	50	56	67	8
4-Bromodiphenyl ether	118	4	nd	nd
"-BHC	88	25	265	18
(-BHC	55	9	155	29
Heptachlor	60	13	469	294
Aldrin	92	33	875	734
$-BHC	351	71	150	260
*-BHC	51	11	57	2
Heptachlor epoxide	54	11	70	3
Endosulfan I	52	11	70	4
(-Chlordane	50	9	65	1
"-Chlordane	49	8	66	0
DDE	52	11	74	1
Dieldrin	89	19	327	7
Endrin	56	10	92	15
Endosulfan II	52	10	88	11
DDT	57	10	95	17
Endrin aldehyde	45	6	42	10
DDD	57	11	99	8
Tetrachloro-*m*-xylene	71	19	82	1
Decachlorobiphenyl	26	23	28	48

nd = Not detected
Concentration spiked in the sample: 500-1000 ng/g, three replicates analyses.
Soxhlet extraction by Method 3540 with methylene chloride.
Ultrasonic extraction by Method 3550 with methylene chloride/acetone (1:1).
Cleanup by Method 3640.
GC column: DB-608, 30-m x 0.53-mm ID.

TABLE 10

ANALYTE RECOVERY FROM DICHLOROETHANE STILLBOTTOMS

	Ultrasonic Extraction		Soxhlet	
Compound	%Recovery	%RSD	%Recovery	%RSD
Hexachloroethane	70	2	50	30
2-Chloronapthalene	59	3	35	35
4-Bromodiphenyl ether	159	14	128	137
"-BHC	55	7	47	25
(-BHC	43	6	30	30
Heptachlor	48	6	55	18
Aldrin	48	5	200	258
$-BHC	51	7	75	42
*-BHC	43	4	119	129
Heptachlor epoxide	47	6	66	34
Endosulfan I	47	4	41	18
(-Chlordane	48	5	47	13
"-Chlordane	45	5	37	21
DDE	45	4	70	40
Dieldrin	45	5	58	24
Endrin	50	6	41	23
Endosulfan II	49	5	46	17
DDT	49	4	40	29
Endrin aldehyde	40	4	29	20
DDD	48	5	35	21
Tetrachloro-*m*-xylene	49	2	176	211
Decachlorobiphenyl	17	29	104	93

Concentration spiked in the sample: 500-1000 ng/g, three replicates analyses.
Soxhlet extraction by Method 3540 with methylene chloride.
Ultrasonic extraction by Method 3550 with methylene chloride/acetone (1:1).
Cleanup by Method 3640.
GC column: DB-608, 30-m x 0.53-mm ID.

TABLE 11

SINGLE-LABORATORY ACCURACY DATA FOR THE EXTRACTION OF ORGANOCHLORINE PESTICIDES FROM SPIKED CLAY SOIL BY METHOD 3541 (AUTOMATED SOXHLET)[a]

	Percent Recovery	
Compound	DB-5	DB-1701
"-BHC	89	94
$-BHC	86	ND
Heptachlor	94	95
Aldrin	ND	92
Heptachlor epoxide	97	97
(-Chlordane	94	95
Endosulfan I	92	92
Dieldrin	ND	113
Endrin	111	104
Endosulfan II	104	104
4,4'-DDT	ND	ND
Mirex	108	102

[a] The operating conditions for the automated Soxhlet were:

Immersion time 45 min
Extraction time 45 min
10-g sample size
Extraction solvent 1:1 acetone/hexane
No equilibration time following spiking.

ND = Not able to determine because of interference.

All compounds were spiked at 500 µg/kg.

Data are taken from Reference 10.

TABLE 12

SINGLE-LABORATORY RECOVERY DATA FOR SOLID-PHASE EXTRACTION OF ORGANOCHLORINE PESTICIDES FROM TCLP BUFFERS SPIKED AT TWO LEVELS

Compound	Spike Level (µg/L)	Buffer 1 (pH = 2.886)		Buffer 2 (pH = 4.937)	
		Recovery (%)	RSD	Recovery (%)	RSD
Low Level Spike					
Toxaphene	250	86	13	77	17
Chlordane	15	88	7	95	6
(-BHC (Lindane)	200	115	7	98	5
Heptachlor	4	95	11	77	23
Heptachlor epoxide	4	107	9	104	12
Endrin	10	89	5	100	6
Methoxychlor	5000	97	8	95	6
High Level Spike					
Toxaphene	1000	106	7	85	15
Chlordane	60	116	12	107	12
(-BHC (Lindane)	800	109	19	112	5
Heptachlor	16	113*	18*	93	3
Heptachlor epoxide	16	82	17	91	7
Endrin	40	84	19	82	4
Methoxychlor	20,000	100	4	87	8

Results were from seven replicate spiked buffer samples, except where noted with *, which indicates that only three replicates were analyzed.

TABLE 13

RECOVERY DATA FROM THREE LABORATORIES FOR SOLID-PHASE EXTRACTION OF ORGANOCHLORINE PESTICIDES FROM SPIKED TCLP LEACHATES FROM SOIL SAMPLES

Compound	Spike Level (µg/L)*	Lab 1			Lab 2			Lab 3		
		%R	RSD	n	%R	RSD	n	%R	RSD	n
Buffer 1 pH = 2.886										
Toxaphene	500	75	25	7	95.4	2.4	3	86.0	4.3	3
Chlordane	30	80	15	7	57.8	12.0	3	73.8	0.9	3
(-BHC (Lindane)	400	104	11	7	99.3	0.6	3	86.6	6.4	3
Heptachlor	8	88	13	7	70.8	20.4	3	88.0	9.1	3
Heptachlor epoxide	8	92	13	7	108.7	6.9	3	75.0	2.8	3
Endrin	20	106	12	7	110	0	3	78.3	4.6	3
Methoxychlor	10,000	107	12	7	86.7	2.2	3	84.8	8.5	3
Buffer 2 pH = 4.937										
Toxaphene	500	87	9	7	98	4.1	3	88.8	4.1	3
Chlordane	30	91	8	7	66.7	5.0	3	73.7	11.5	3
(-BHC (Lindane)	400	74	20	7	102.7	2.2	3	89.3	3.1	3
Heptachlor	8	71	21	7	62.5	20	3	85.0	1.5	3
Heptachlor epoxide	8	118	1	3	113	0	3	81.3	2.7	3
Endrin	20	124	7	3	111.7	2.6	3	83.0	3.4	3
Methoxychlor	10,000	73	22	7	88.8	2.7	3	89.6	2.7	3

* 250-mL aliquots of leachate were spiked by Labs 2 and 3 at the levels shown. Lab 1 spiked at one-half these levels.

TABLE 14

SINGLE-LABORATORY ACCURACY AND PRECISION DATA FOR SOLID-PHASE EXTRACTION BY METHOD 3535[1]

	Bias (%)				Precision (%)				MDL	
Compound	Ground water (low)	Ground water (high)	Waste water (low)	Waste water (high)	Ground water (low)	Ground water (high)	Waste water (low)	Waste water (high)	Ground water (µg/L)	Waste water (µg/L)
Aldrin	37.3	93.5	79.3	94.0	23.7	5.5	6.7	3.4	1.4	0.83
$-BHC	89.2	107.8	79.7	82.3	6.5	2.5	1.6	4.2	0.91	0.20
*-BHC	106.2	86.0	88.9	83.4	5.6	2.4	2.5	4.2	0.93	0.35
"-Chlordane	75.4	112.3	78.9	89.5	12.8	2.7	4.7	2.4	1.5	0.58
(-Chlordane	70.7	98.9	79.9	93.9	15.8	2.7	4.6	2.9	1.8	0.58
Dieldrin	83.4	96.1	81.2	93.3	7.1	2.3	3.8	3.6	0.9	0.49
Endosulfan I	79.6	99.1	79.6	87.9	10.6	2.3	4.1	3.8	1.3	0.51
Endosulfan II	94.5	101.6	82.7	93.5	5.8	2.8	4.2	4.1	0.9	0.54
Endrin	88.3	98.4	85.1	89.6	6.2	2.3	3.1	2.9	1.7	0.82
Endrin aldehyde	87.5	99.9	69.0	80.2	6.0	4.0	3.3	5.9	0.8	0.36
Heptachlor	43.1	95.4	71.8	78.6	19.2	3.9	5.0	2.8	1.3	0.56
Heptachlor epoxide	76.4	97.6	75.3	83.4	12.1	2.4	2.9	3.3	1.5	0.34
Lindane	81.3	115.2	82.1	85.3	11.1	3.2	2.4	3.1	1.4	0.32
4,4'-DDE	80.3	96.0	85.1	97.9	8.3	2.5	4.4	2.4	1.0	0.59
4,4'-DDT	86.6	105.4	105	111	4.4	2.7	4.3	4.7	0.6	0.71
4,4'-TDE (DDD)	90.5	101.1	74.9	79.6	4.8	2.4	4.6	2.9	1.4	0.85

[1]All results determined from seven replicates of each sample type. Two spiking levels were used. "Low" samples were spiked at 5-10 µg/L for each analyte, while "high" samples were spiked at 250 - 500 µg/L. MDL values were determined from the "low" samples without further consideration of the spiking level, and are provided for illustrative purposes only.

TABLE 15

RECOVERY (BIAS) OF ORGANOCHLORINE PESTICIDES USING SFE METHOD 3562
(Seven replicates)

Compound	Delphi[a] 250 ug/kg	Delphi[a] 5 ug/kg	McCarthy[b] 250 ug/kg	McCarthy[b] 5 ug/kg	Auburn[c] 250 ug/kg	Auburn[c] 5 ug/kg	Mean Recovery
(-BHC	102.6	66.4	80.7	82.7	86.0	86.1	84.1
$-BHC	101.9	73.0	86.1	85.1	87.4	86.3	86.6
Heptachlor	101.3	61.6	78.0	79.1	83.3	80.4	80.6
*-BHC	120.9	82.3	90.4	89.6	92.9	89.4	94.2
Aldrin	56.7	28.7	52.1	77.1	42.1	74.6	55.2
Heptachlor epoxide	102.3	71.9	87.1	87.4	89.6	91.1	88.2
"-Chlordane	106.4	87.1	88.1	105.9	91.7	97.1	96.1
4,4'DDE	110.9	75.7	88.4	118.7	83.6	110.9	98.0
Dieldrin	106.9	80.4	88.1	140.8	90.6	80.1	97.8
Endrin	211.0	87.0	111.7	98.7	90.5	87.6	114.4
4,4'-DDD	93.0	80.4	85.0	88.1	83.7	90.4	86.8
Endosulfan II	105.6	89.9	92.1	88.6	87.7	92.9	92.5
4,4'-DDT	126.7	81.3	110.9	199.7	83.6	124.3	121.1
Endrin aldehyde	64.3	74.0	63.0	86.7	21.0	38.3	37.9
Matrix Mean Recovery	107.9	74.3	85.9	102.0	79.8	87.8	89.5

[a] Delphi: Loamy sand soil
[b] McCarthy: Sandy loamy-organic rich soil
[c] Auburn: Clay-loamy soil

TABLE 16

RELATIVE STANDARD DEVIATION (PRECISION) OF ORGANOCHLORINE PESTICIDES USING SFE METHOD 3562
(Seven replicates)

Compound	Delphi[a] 250 ug/kg	Delphi[a] 5 ug/kg	McCarthy[b] 250 ug/kg	McCarthy[b] 5 ug/kg	Auburn[c] 250 ug/kg	Auburn[c] 5 ug/kg	Mean
(-BHC	3.9	3.3	3.3	6.5	4.0	1.6	3.7
$-BHC	6.5	3.0	3.0	4.3	4.6	2.0	3.9
Heptachlor	4.4	2.1	4.3	5.0	4.4	2.6	3.8
*-BHC	5.3	3.1	3.3	7.1	4.1	3.5	4.4
Aldrin	2.9	5.5	2.8	4.6	1.6	1.9	3.2
Heptachlor epoxide	3.0	2.7	3.6	4.3	4.7	4.2	3.8
"-Chlordane	3.6	5.7	4.8	13.8	4.2	2.5	5.8
4,4'DDE	5.2	15.3	4.8	4.2	7.7	3.4	6.8
Dieldrin	4.3	4.5	2.9	23.9	5.0	3.1	7.3
Endrin	7.2	6.0	4.5	6.0	4.3	10.5	6.4
4,4'-DDD	6.9	3.1	3.7	3.5	4.3	7.4	4.8
Endosulfan II	5.1	4.7	3.2	3.3	5.5	4.6	4.4
4,4'-DDT	12.5	6.2	6.6	5.9	4.9	3.4	6.6
Endrin aldehyde	3.9	7.5	4.7	11.6	1.9	26.0	9.3
Matrix Mean Recovery	5.3	5.2	4.0	7.4	4.4	5.5	5.3

[a] Delphi: Loamy sand soil
[b] McCarthy: Sandy loamy-organic rich soil
[c] Auburn: Clay-loamy soil

TABLE 17

SINGLE-LABORATORY ORGANOCHLORINE PESTICIDES DATA FROM THREE SOIL MATRICES SPIKED AT 5 TO 10 PPB AND EXTRACTED USING METHOD 3545 (PRESSURIZED FLUID EXTRACTION)

		PFE Recovery and Precision					
	Certified Value	Clay		Loam		Sand	
Compound	(µg/L)	% Rec.	RSD	% Rec	RSD	% Rec	RSD
Aldrin	5.2	65	10	60	6	71	11
"-BHC	5.0	52	7	50	10	60	12
$-BHC	5.0	84	6	76	5	92	11
*-BHC	5.0	100	7	96	4	104	11
(-BHC (Lindane)	5.0	6	6	62	8	74	12
"-Chlordane	4.9	9	7	86	4	84	11
(-Chlordane	4.9	9	8	82	5	86	11
4,4'-DDD	10.1	8	8	84	5	87	10
4,4'-DDE	4.9	10	7	90	5	96	12
4,4'-DDT	4.8	90	7	67	6	88	17
Dieldrin	5.0	94	8	84	4	88	11
Endosulfan I	5.0	88	8	80	5	78	11
Endosulfan II	5.0	88	7	84	4	86	10
Endosulfan sulfate	9.5	93	7	87	3	88	11
Endrin	9.8	84	9	81	5	85	11
Endrin aldehyde	5.0	86	7	76	4	64	15
Endrin ketone	9.7	100	6	90	4	94	11
Heptachlor	5.0	70	8	64	8	76	12
Heptachlor epoxide	5.0	78	8	76	5	82	11
Methoxychlor	5.0	82	7	62	6	80	17

Seven replicate extractions were performed using 14-g samples of spiked soil from a commercial supplier. Hexane:acetone (1:1) was used as the extraction solvent, at 100EC and 2000 psi, using a 5-minute heating time and a 5-minute static extraction.

Data are adapted from Reference 14.

TABLE 18

SINGLE-LABORATORY ORGANOCHLORINE PESTICIDES DATA FROM THREE SOIL MATRICES SPIKED AT 50 TO 100 PPB AND EXTRACTED USING METHOD 3545 (PRESSURIZED FLUID EXTRACTION)

		PFE Recovery and Precision					
	Certified Value	Clay		Loam		Sand	
Compound	(µg/L)	% Rec.	RSD	% Rec	RSD	% Rec	RSD
Aldrin	51.5	77	5	77	11	72	9
"-BHC	49.5	64	6	67	11	62	9
$-BHC	49.5	79	3	81	9	77	7
*-BHC	50.0	85	4	88	9	83	7
(-BHC (Lindane)	49.5	74	5	77	11	73	9
"-Chlordane	48.6	87	4	85	9	81	8
(-Chlordane	49.1	87	3	85	9	82	8
4,4'-DDD	101.0	90	3	90	7	87	8
4,4'-DDE	49.1	95	4	93	8	90	8
4,4'-DDT	48.4	79	7	73	13	75	15
Dieldrin	49.6	94	4	88	8	85	8
Endosulfan I	49.8	85	3	82	9	80	8
Endosulfan II	49.7	94	4	91	7	88	8
Endosulfan sulfate	94.7	93	4	89	7	87	8
Endrin	98.0	86	3	83	8	81	8
Endrin aldehyde	49.5	78	4	75	7	75	8
Endrin ketone	97.2	95	4	91	8	85	8
Heptachlor	49.5	70	5	72	11	68	10
Heptachlor epoxide	49.8	89	3	84	9	80	8
Methoxychlor	49.6	79	8	74	12	74	14

Seven replicate extractions were performed using 10-g samples of spiked soil from a commercial supplier. Hexane:acetone (1:1) was used as the extraction solvent, at 100EC and 2000 psi, using a 5-minute heating time and a 5-minute static extraction.

Data are adapted from Reference 14.

TABLE 19

SINGLE-LABORATORY ORGANOCHLORINE PESTICIDES DATA FROM THREE SOIL MATRICES SPIKED AT 250 TO 500 PPB AND EXTRACTED USING METHOD 3545 (PRESSURIZED FLUID EXTRACTION)

		PFE Recovery and Precision					
	Certified Value	Clay		Loam		Sand	
Compound	(µg/L)	% Rec.	RSD	% Rec	RSD	% Rec	RSD
Aldrin	257	66	3	71	11	60	16
"-BHC	247	54	5	59	11	51	14
$-BHC	247	67	2	72	9	63	13
*-BHC	250	70	2	76	10	66	13
(-BHC (Lindane)	247	64	4	69	11	60	14
"-Chlordane	243	72	2	76	9	65	15
(-Chlordane	245	73	2	76	9	65	15
4,4'-DDD	507	72	3	76	8	66	14
4,4'-DDE	246	83	3	87	9	75	14
4,4'-DDT	242	77	6	82	10	71	20
Dieldrin	248	78	2	81	9	70	15
Endosulfan I	249	71	2	74	9	64	15
Endosulfan II	249	82	2	84	8	72	15
Endosulfan sulfate	474	81	4	83	8	72	17
Endrin	490	70	3	72	8	63	15
Endrin aldehyde	247	71	3	74	8	64	17
Endrin ketone	486	92	3	94	8	81	16
Heptachlor	247	63	3	68	11	58	16
Heptachlor epoxide	249	70	2	75	9	64	15
Methoxychlor	248	81	7	85	9	74	19

Seven replicate extractions were performed using 10-g samples of spiked soil from a commercial supplier. Hexane:acetone (1:1) was used as the extraction solvent, at 100EC and 2000 psi, using a 5-minute heating time and a 5-minute static extraction.

Data are adapted from Reference 14.

TABLE 20

SINGLE-LABORATORY ORGANOCHLORINE PESTICIDES DATA FROM A REAL-WORLD SOIL MATRIX SPIKED AT THE 500 PPB LEVEL AND EXTRACTED USING METHOD 3546 (MICROWAVE EXTRACTION)

Compound	Recovery (%)	RSD (%)
"-BHC	96	3
$-BHC	126	8
(-BHC	103	4
*-BHC	115	5
Heptachlor	131	5
Aldrin	103	2
Heptachlor epoxide	126	9
Endosulfan I	122	5
DDE + Dieldrin	118	4
Endrin	155	12
Endosulfan II	116	5
DDD	95	7
Endosulfan aldehyde	103	9
Endosulfan sulfate	122	5
DDT	118	5
Methoxychlor	119	6

n = 3

DDE and dieldrin are reported as the sum of the two compounds since they were not resolved by chromatography.

Data are taken from Reference 15.

TABLE 21

SINGLE-LABORATORY COMPARISON OF METHOD 3546 (MICROWAVE EXTRACTION) AND METHOD 3540 (SOXHLET EXTRACTION) OF ORGANOCHLORINE PESTICIDES FROM A REAL-WORLD CONTAMINATED SOIL

	Microwave Extraction Results				
Compound	Average Concentration (µg/kg)	Standard Deviation (µg/kg)	RSD (%)	n	Soxhlet Result (µg/kg)
DDE + dieldrin	3,400	340	10	3	7,100
Endrin	22,000	2,300	11	3	22,000
*DDD	40,000	5,800	14	3	45,000
*DDT	63,000	8,400	13	3	62,000
*Methoxychlor	17,000	2,000	12	3	16,000
"-Chlordane	730	100	13	3	750
(-Chlordane	720	90	12	3	910

* Sample extracts were diluted 1:5 for these compounds.

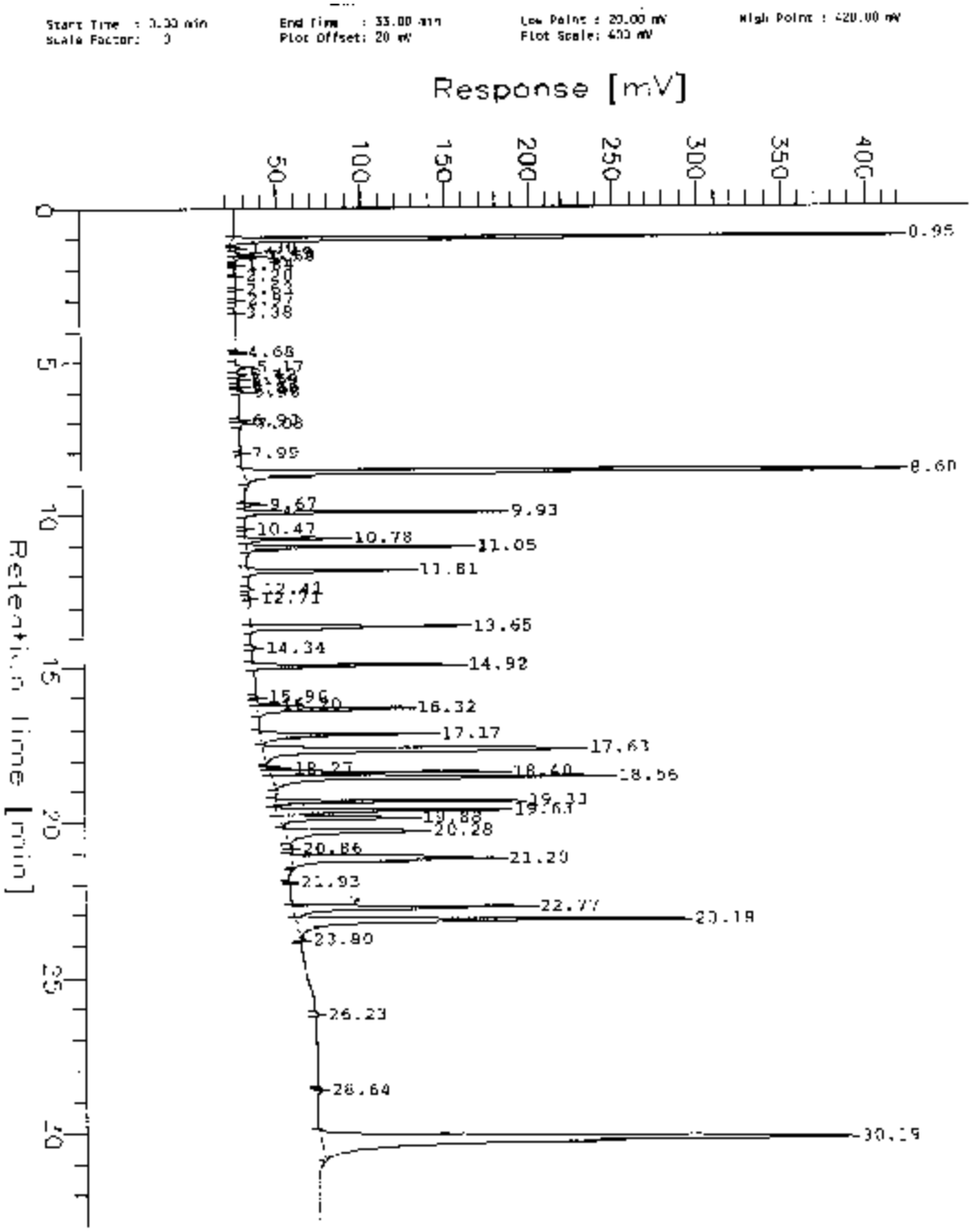
Soil samples obtained from the US EPA Emergency Response Center archive bank through their contract laboratory, REAC (Edison, NJ). The single Soxhlet extraction was performed by REAC three years earlier and the long storage period is believed to account for the low DDE + dieldrin recovery in the present study.

DDE and dieldrin are reported as the sum of the compounds since they were not resolved by chromatography

Data are taken from Reference 15.

FIGURE 1

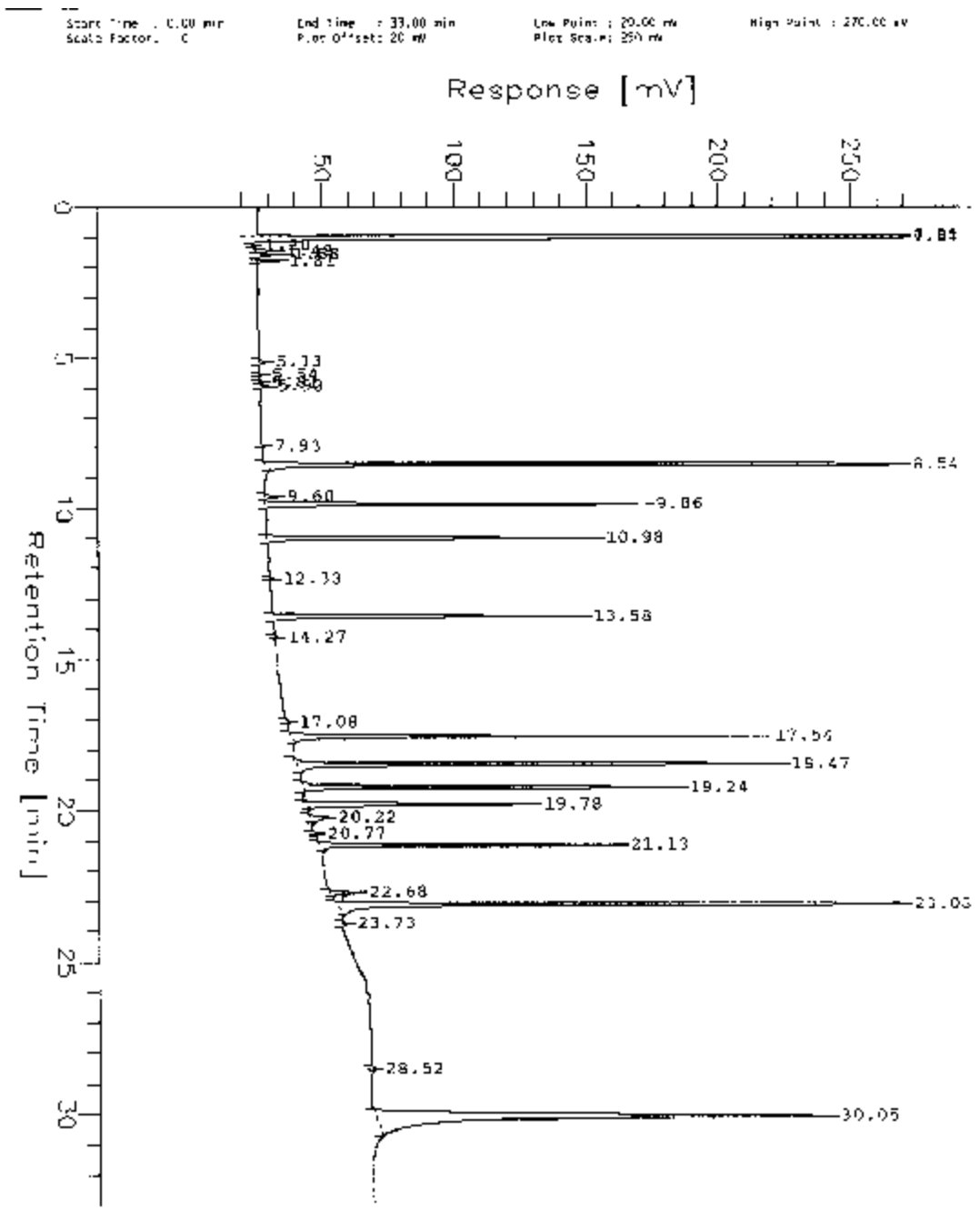
EXAMPLE GAS CHROMATOGRAM OF
THE MIXED ORGANOCHLORINE PESTICIDE STANDARD



Column: 30-m x 0.25-mm ID, DB-5
Temperature program: 100EC (hold 2 minutes) to 160EC at 15EC/min, then at 5EC/min to 270EC; carrier He at 16 psi

FIGURE 2

EXAMPLE GAS CHROMATOGRAM OF INDIVIDUAL ORGANOCHLORINE PESTICIDE STANDARD MIX A

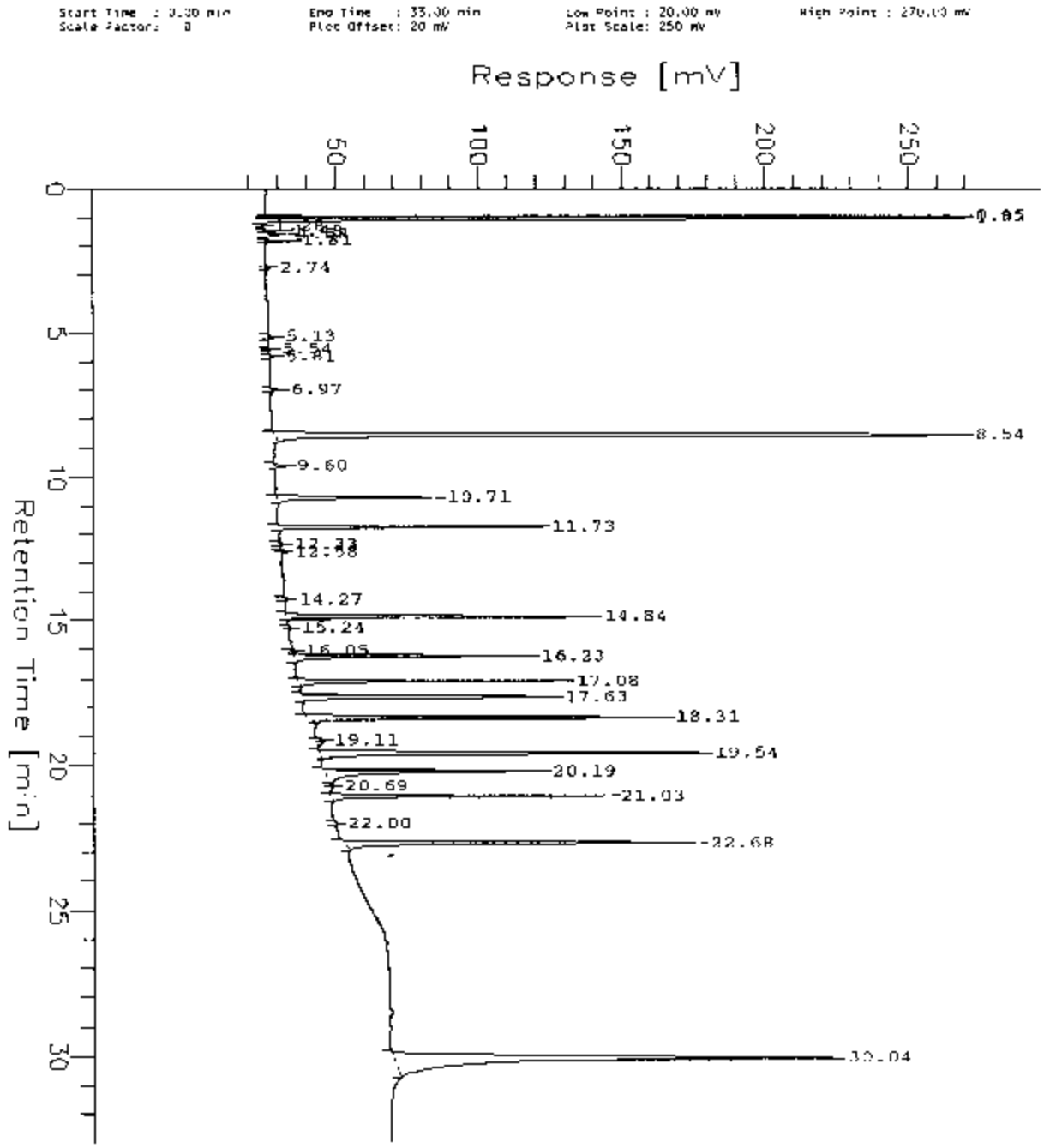


Column: 30-m x 0.25-mm ID, DB-5

Temperature program: 100EC (hold 2 minutes) to 160EC at 15EC/min, then at 5EC/min to 270EC; carrier He at 16 psi.

FIGURE 3

EXAMPLE GAS CHROMATOGRAM OF INDIVIDUAL ORGANOCHLORINE PESTICIDE STANDARD MIX B

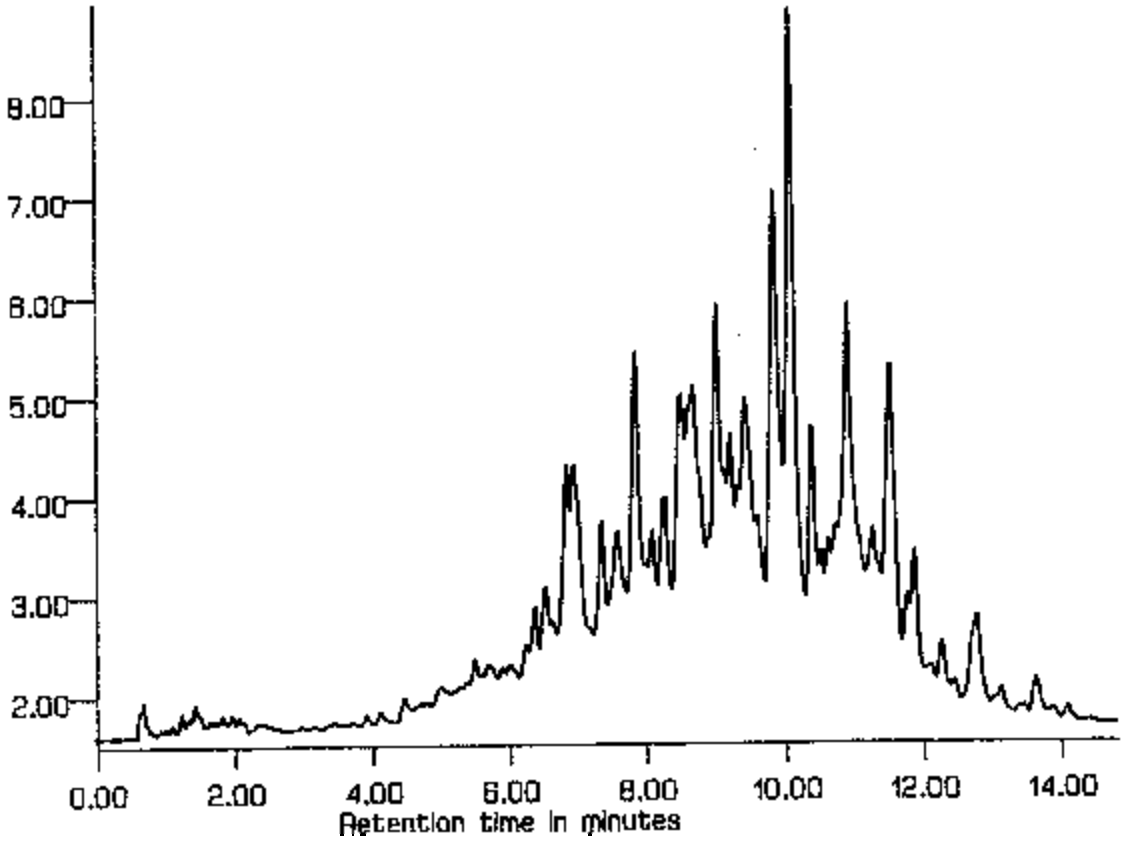


Column: 30-m x 0.25-mm ID, DB-5

Temperature program: 100EC (hold 2 minutes) to 160EC at 15EC/min, then at 5EC/min to 270EC; carrier He at 16 psi.

FIGURE 4

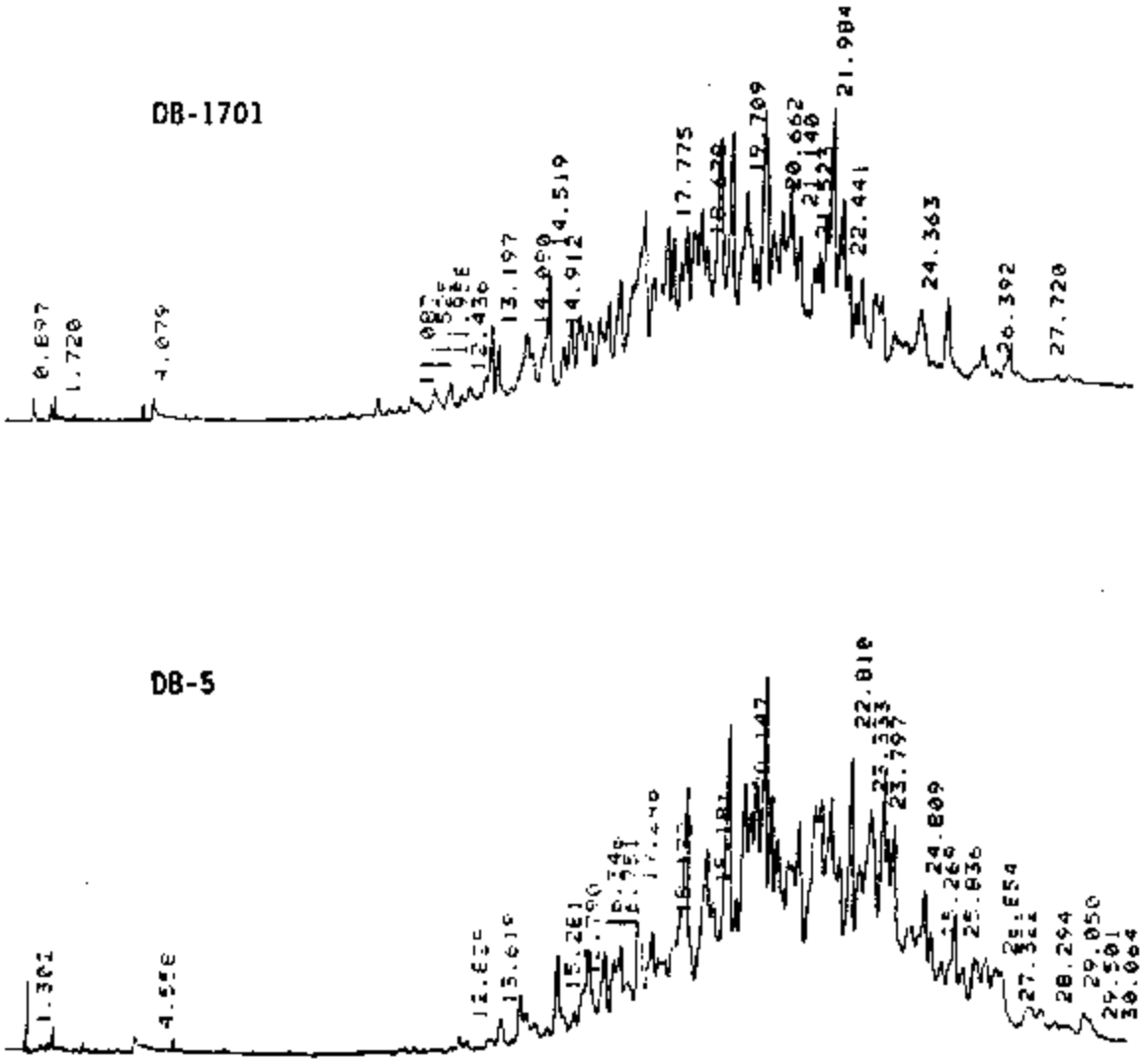
EXAMPLE GAS CHROMATOGRAM OF TOXAPHENE



Toxaphene analyzed on an SPB-608 fused-silica open-tubular column. The GC operating conditions were as follows: 30-m x 0.53-mm ID SPB-608. Temperature program: 200EC (2 min hold) to 290EC at 6EC/min.

FIGURE 5

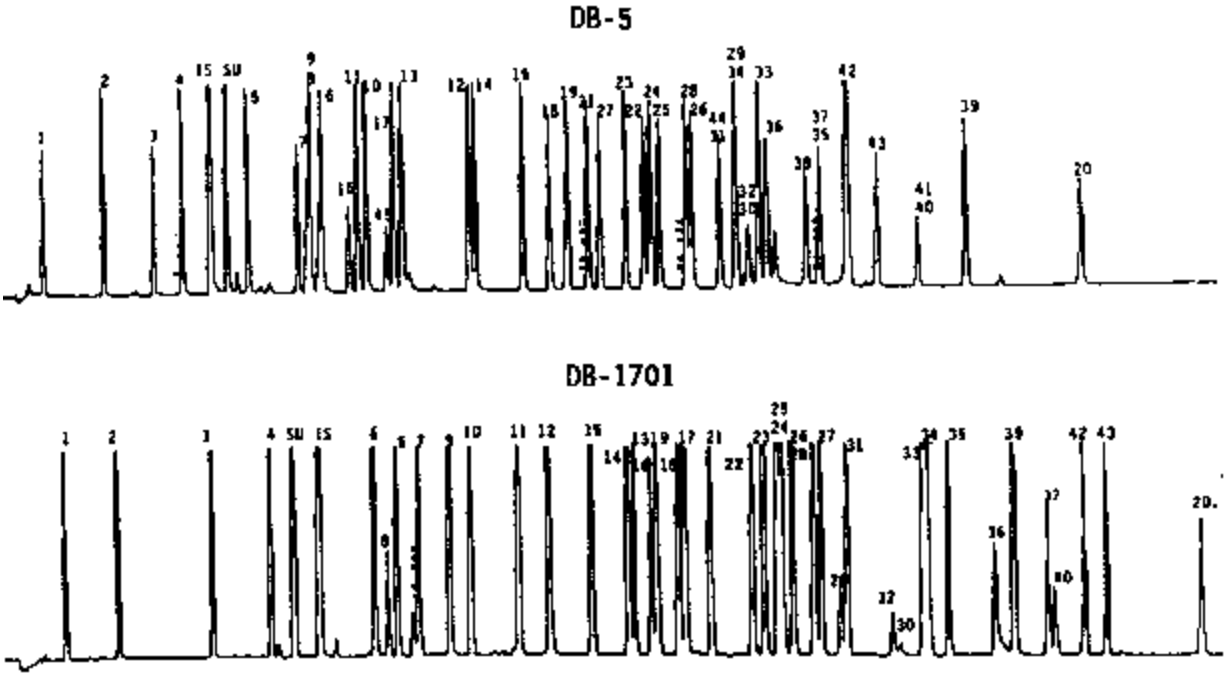
EXAMPLE GAS CHROMATOGRAM OF STROBANE



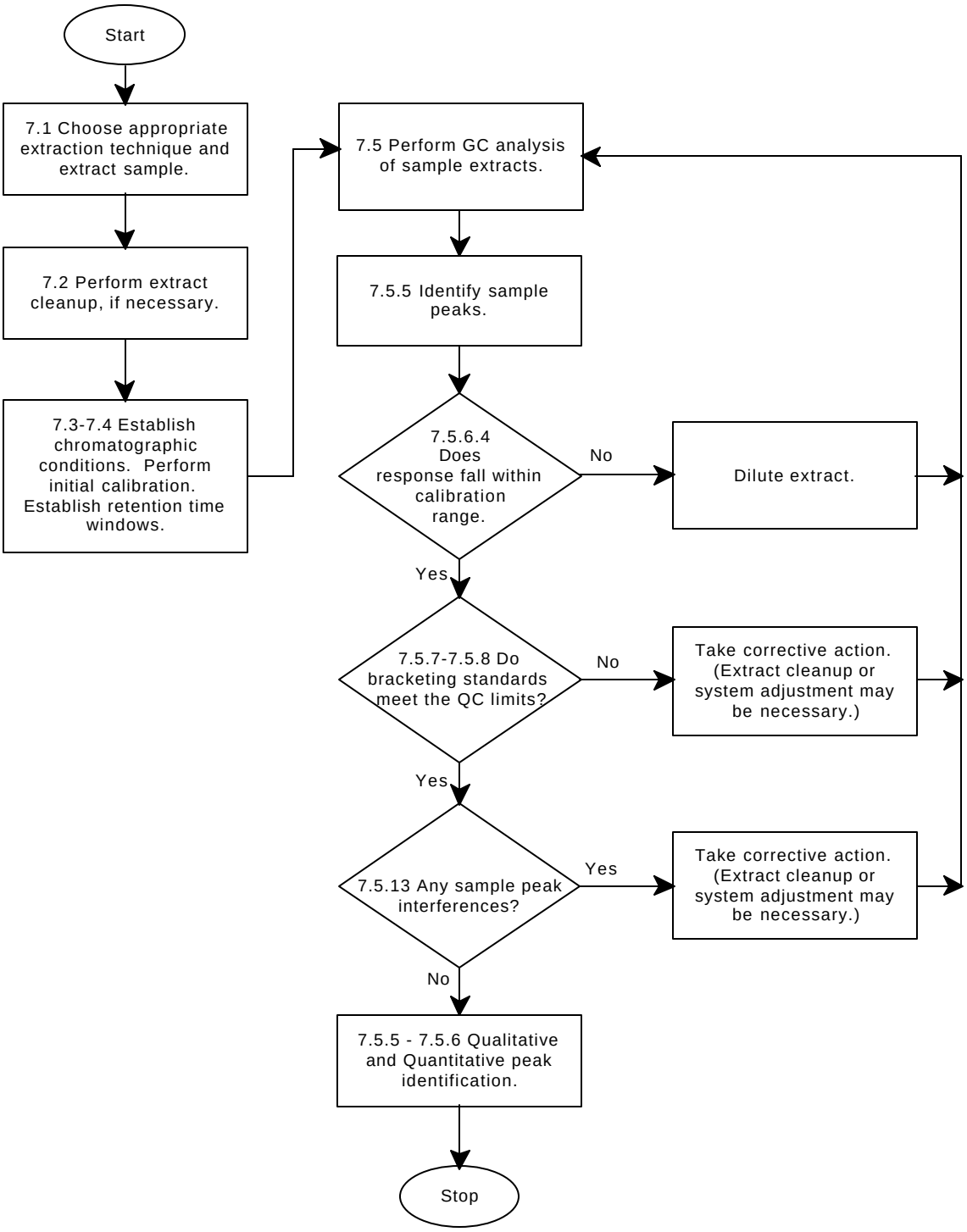
Strobane analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30-m x 0.53-mm ID DB-5 (1.5-µm film thickness) and 30-m x 0.53-mm ID DB-1701 (1.0-µm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150EC (0.5 min hold) to 190EC (2 min hold) at 12EC/min then to 275EC (10 min hold) at 4EC/min.

FIGURE 6

EXAMPLE GAS CHROMATOGRAM OF ORGANOCHLORINE PESTICIDES



Organochlorine pesticides analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30-m x 0.53-mm ID DB-5 (0.83-µm film thickness) and 30-m x 0.53-mm ID DB-1701 (1.0-µm film thickness) connected to an 8-in. injection tee (Supelco Inc.). Temperature program: 140EC (2 min hold) to 270EC (1 min hold) at 2.8EC/min.


Start
7.1 Choose appropriate extraction technique and extract sample.
7.2 Perform extract cleanup, if necessary.
7.3-7.4 Establish chromatographic conditions. Perform initial calibration. Establish retention time windows.
7.5 Perform GC analysis of sample extracts.
7.5.5 Identify sample peaks.
7.5.6.4 Does response fall within calibration range.
No
Dilute extract.
Yes
7.5.7-7.5.8 Do bracketing standards meet the QC limits?
No
Take corrective action. (Extract cleanup or system adjustment may be necessary.)
Yes
7.5.13 Any sample peak interferences?
Yes
Take corrective action. (Extract cleanup or system adjustment may be necessary.)
No
7.5.5 - 7.5.6 Qualitative and Quantitative peak identification.
Stop