Designation: D6160 – 98 (Reapproved 2008)

Standard Test Method for Determination of Polychlorinated Biphenyls (PCBs) in Waste Materials by Gas Chromatography[1]

This standard is issued under the fixed designation D6160; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method[2] covers a two-tiered analytical approach to PCB screening and quantitation of liquid and solid wastes, such as oils, sludges, aqueous solutions, and other waste matrices.

1.2 Tier I is designed to screen samples rapidly for the presence of PCBs.

1.3 Tier II is used to determine the concentration of PCBs, typically in the range of from 2 to 50 mg/kg. PCB concentrations greater than 50 mg/kg are determined through analysis of sample dilutions.

1.4 This is a pattern recognition approach, which does *not* take into account individual congeners that might occur, such as in reaction by-products. This test method describes the use of Aroclors[3] 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268, as reference standards, but others could also be included. Aroclors 1016 and 1242 have similar capillary gas chromatography (GC) patterns. Interferences or weathering are especially problematic with Aroclors 1016, 1232, and 1242 and may make distinction between the three difficult.

1.5 This test method provides sample clean up and instrumental conditions necessary for the determination of Aroclors. Gas chromatography (GC) using capillary column separation technique and electron capture detector (ECD) are described. Other detectors, such as atomic emission detector (AED) and mass spectrometry (MS), may be used if sufficient performance (for example, sensitivity) is demonstrated. Further details about the use of GC and ECD are provided in Practices E355, E697, and E1510.

1.6 Quantitative results are reported on the dry weights of waste samples.

1.7 Quantification limits will vary depending on the type of waste stream being analyzed.

1.8 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.9 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulator limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography

E203 Test Method for Water Using Volumetric Karl Fischer Titration

E288 Specification for Laboratory Glass Volumetric Flasks

E355 Practice for Gas Chromatography Terms and Relationships

E697 Practice for Use of Electron-Capture Detectors in Gas Chromatography

E969 Specification for Glass Volumetric (Transfer) Pipets

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *U.S. EPA Standards:*

Method 608 Organochlorine Pesticides and PCBs[5]

Method 680 Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry[6]

Method 3620 Florisil Column Clean-Up[7]

Method 3630 Silica Gel Clean-Up[7]

Method 3660 Sulfur Clean-Up[7]

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

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[2] This test method is based largely on EPA 8080 (and the proposed modification for the use of capillary columns, EPA 8081) and EPA Report 600/4–81–045 by Bellar, T. and J. Lichtenberg, reported in 1981. The report is titled, "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," and provides significant support to the protocol in this standard.

[3] Aroclor Standards may be purchased as 1000 µg/mL in *iso*octane. Aroclor is a registered trademark of the Monsanto Company, 800 N. Lindbergh Blvd., St. Louis, MO 63167.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[5] EPA Report 600/4/82–057, Environmental Monitoring and Support Laboratory, Cincinnati, OH.

[6] Alford-Stevens, Ann, et al, Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory Office of Research and Development, USEPA, Cincinnati, OH.

[7] U.S. EPA, "Test Methods for Evaluating Solid Waste," *Physical/Chemical Methods*, SW-846.

Method 8082 Determination of PCB in Water and Soil/Sediment by Gas Chromatography: Capillary Column Technique[7]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Aroclors*, *n*—commercial mixtures of polychlorinated biphenyl congeners marketed and trademarked by Monsanto prior to 1977.

3.1.1.1 *Discussion*—Specific Aroclors are usually designated by a four-digit number, with the first two digits usually designating the number of carbon atoms and the last two digits providing the chlorine content (for example, Aroclor 1260 is 60 % (weight) chlorine).

3.1.2 *congeners*, *n*—compounds related by structural similarities.

3.1.2.1 *Discussion*—All polychlorinated biphenyls (PCBs) share the same C_{12} structure and vary only by the number and position of the chlorine atoms attached to the aromatic rings.

3.1.3 *continuing calibration standard (CCS)*—a known blend or one or more Aroclors at a fixed concentration that is injected into the gas chromatograph to demonstrate the validity of the calibration.

3.1.4 *dry weight*, *n*—concentration of PCBs after factoring out the water content.

3.1.4.1 *Discussion*—This correction assumes that all PCBs originated from nonaqueous sources and any water present has been added subsequently, diluting the original concentration. This correction can be described using the formula:

$$\text{Aroclor (mg/Kg) (dry)} = \frac{\text{Aroclor (mg/Kg) (wet)}}{(100 - \%\ \text{water})/100} \quad (1)$$

3.1.5 *instrument performance standard (IPS)*, *n*—a known low level of an Aroclor in a clean solvent used as a comparator to determine which qualitative (screening) results are of sufficient magnitude to require quantitative analyses.

3.1.6 *surrogate*, *n*—compound or compounds that are similar to analytes of interest in chemical composition, extraction, and chromatography, but that are not normally found at significant levels in the matrices of interest.

3.1.6.1 *Discussion*—Surrogates may be spiked into blanks, standards, samples, or matrix spikes prior to analysis to allow a determination of a quantitative recovery rate. Surrogates are also used to document matrix effects and method control.

3.1.7 *waste material*, *n*—any matter, within the scope of this test method, that is in the process of being recycled or disposed.

4. Summary of Test Method

4.1 The sample is extracted with solvent and the extract is treated to remove interfering substances, if needed. The sample extract is injected into a gas chromatograph. The components are separated as they pass through the capillary column and polychlorinated biphenyl compounds, if present, are detected by an ECD.

NOTE 1—Portions of this test method are similar to EPA Methods 608, 680, and 8082.

4.2 For screening (Tier I), instrument performance is monitored by a 2-µL injection of a standard containing Aroclors 1016 and 1260. For low level work (1 ppm) the instrument is checked with a standard concentration of 0.01 µg/mL (each) and for higher level work (10 ppm), the instrument is checked with a 0.1 µg/mL standard.

4.3 Identification involves a pattern comparison of the chromatograms of an unknown sample with that of a standard obtained under identical instrumental conditions.

4.4 When quantification is required (Tier II), an external standards method (ESTD) is used. The quantitation technique typically requires a comparison of five peaks (minimum of three) between the chromatograms of an unknown sample and that of standard Aroclor obtained under identical conditions. Quantitation of either Aroclors 1016 or 1260 is performed using a five-point calibration of a mixed Aroclor standard containing Aroclors 1016 and 1260. All remaining Aroclors are quantitated from single point calibrations. Calibration is verified daily by comparison of results obtained for analysis of the midpoint calibration standard of Aroclor 1016 and 1260 to the five-point calibration curve. (See Appendix X1 for an example chromatogram and calibration table.)

5. Significance and Use

5.1 This test method provides sufficient PCB data for many regulatory requirements. While the most common regulatory level is 50 ppm (dry weight corrected), lower limits are used in some locations. Since sensitivities will vary for different types of samples, one shall demonstrate a sufficient method detection limit for the matrix of interest.

5.2 This test method differs from Test Method D4059 in that it provides for more sample clean-up options, utilizes a capillary column for better pattern recognition and interference discrimination, and includes both a qualitative screening and a quantitative results option.

6. Interferences

6.1 The ECD has selective sensitivity to alkyl halides, conjugated carbonyls, nitrogen compounds, organometallics, and sulfur. Therefore, the chromatogram obtained for each sample shall be carefully compared to chromatograms of standards to allow proper interpretation.

6.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts or interferences, or both, to standard analysis. All these materials shall be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.

6.3 Interferences from phthalate esters may pose a major problem in Aroclor determinations when using ECD. Phthalates generally appear in the chromatogram as broad late eluting peaks. Since phthalates are commonly used as plasticizers and are easily extracted from plastic, all contact of samples and extracts with plastic should be avoided.

6.4 While general clean-up techniques are provided as part of this test method, some samples may require additional clean-up beyond the scope of this test method before proper instrumental analysis may be performed.

7. Apparatus

7.1 *Gas Chromatograph*, a temperature programmable gas chromatograph suitable for splitless injections; equipped with an ECD.

7.2 *Data System*, a data system capable of measuring peak areas.

7.3 *Regulator (Make-up Gas)*—N_2 or Ar:Methane (95:5); two stage regulator rated at 20 MPa (3000 psi) inlet and 35 to 860 kPa (5 to 125 psi) outlet.

7.4 *Regulator (Carrier Gas)*—H_2, two-stage regulator rated at 20 MPa (3000 psi) inlet and 35 to 860 kPa (5 to 125 psi) outlet.

7.5 *Gas Purifiers*, to remove moisture and particulates. Depending on the levels and types of interferences encountered, these might involve molecular sieves (moisture), activated carbon (organics), or other commercially-available media.

7.6 *Flow Meter*, to measure gas flow. Typical range is from 0.5 to 50 mL/min. ± 0.1 mL/min.

7.7 *Column*, crosslinked 5 % phenyl methyl silicone, 30 m by 0.32 mm id by 0.25 µm film thickness.

7.7.1 It is possible that other columns will provide sufficient separating power, but this shall be demonstrated before use.

7.8 *Analytical Balance*, capable of weighing to 0.0001 g.

7.9 *Volumetric Flasks*, 10, 50, 100, 200 mL, (see Specification E288) Class A with ground-glass stoppers.

7.10 *Vortex Mixer*:

7.11 *Vials*, glass, 20 mL and 40 mL capacity with TFE-fluorocarbon-lined caps.

7.12 *Septum Inserts*—Inserts shall be treated with a silynization reagent before use or after cleaning. (See Annex A2 for possible procedure.) They may be purchased already treated.

7.13 *Volumetric Pipette*, 1, 5, 10 mL (see Specification E969), Class A.

7.14 *Syringe*, 500 µL, mechanical guide.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[8] 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.

8.2 *Acetone*—(**Warning—**Extremely flammable. Vapors may cause flash fire.)

8.3 *Activated Magnesium Silicate (Florisil)*, Pesticide residue (PR) grade (60/100 mesh); store in glass containers with ground glass stoppers or foil lined screw caps.

8.3.1 Just before use, activate each batch at least 4 h at 130°C in a glass container loosely covered with aluminum foil. Alternatively, store the magnesium silicate in an oven at 130°C. Cool the magnesium silicate in a desiccator for 30 min before use.

8.4 *Hexane*—(**Warning—**Extremely flammable. Harmful if inhaled. May produce nerve cell damage. Vapors may cause flash fire.)

8.5 *Isooctane*—(**Warning—**Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.)

8.6 *Methanol*—(**Warning—**Flammable. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled. Cannot be made nonpoisonous.)

8.7 *Silynization Reagent* (for example, 5 % dimethyldichlorosilane in toluene). See Annex A2 for instructions.

8.8 *Sodium Sulfate*, granular, anhydrous (maintained at 130°C for at least 24 h prior to use). Cool the sodium sulfate in a desiccator for 30 min before use.

8.9 *Sulfuric Acid (concentrated)*:

8.10 *Acetone/Hexane*, 10 % acetone/90 % hexane (v/v).

8.11 *Gases*, Hydrogen (zero grade; 99.995 % purity) and nitrogen (zero grade; 99.998 % purity) or argon/methane (95:5; ECD grade).

8.11.1 Care shall be given to ensure purity of the carrier gas. For example, an in-line filter may be required.

8.12 *Aroclor Standards*[3], Aroclor 1016, 1221, 1232, 1242, 1254, 1260, 1262, 1268.

8.13 *Decachlorobiphenyl (DCB) (surrogate) Optional*:

8.13.1 *Surrogate Stock Standard (15 µg/mL) Preparation*—Accurately dilute 1.5 mL of 1000 µg/mL DCB concentrate in 100 mL volumetric flask and fill to the mark with methanol, yielding a 15 µg/mL solution.

8.13.2 *Surrogate Working Standard (1.5 µg/mL) Preparation*—Accurately dilute 10 mL of the 15 µg/mL DCB stock standard in a 100 mL volumetric flask and fill to the mark with methanol, yielding a 1.5 µg/mL working DCB standard.

NOTE 2—Sample preparations will normally use 0.1 mL of this solution. The resulting concentration in the sample extract is 0.005 µg/mL before any further dilutions. The following calculations show this.

$$1.5\ \mu g/mL \times 0.1\ mL = 0.15\ \mu g$$

$$\frac{0.15\ \mu g}{(3.0\ mL\ sample + 27\ mL)} = 0.005\ \mu g/mL \qquad (2)$$

8.14 *Calibration Standards*:

8.14.1 *Intermediate Stock Standard (50 µg/mL)*:

If high level standards (for example, commercially available standards at 2000 to 5000 µg/mL) have been purchased, prepare solutions of 50 µg/mL concentration.

8.14.1.1 The surrogate calibration standard may be added (optional) to the Aroclor 1016/1260 intermediate stock standard at a concentration of 2.5 µg/mL. For preparation of the standard, add 500 µL of 50 µg/mL surrogate to a 10 mL volumetric flask containing 3.0 mL of *iso*octane. Add the Aroclor 1016/1260 standard (5.0 mL at 100 µg/mL) to the flask. Dilute to 10 mL volume with *iso*octane and mix well.

8.14.1.2 To prepare the continuing CCS, dilute 200 µL of the intermediate stock standard to 100 mL.

[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Volume add into the 100 mL flask	Ar-1016/1260 concentration µg/mL	Surrogate concentration µg/mL
200 µL	0.10	0.005

8.14.2 *Instrument Performance Standard (IPS) (Tier I–Screening)*—An *iso*octane solution of Aroclors 1016 and 1260 is prepared at a concentration of 0.01 µg/mL (each) or 0.1 µg/mL (each) (depending on whether the minimum level of interest is 2 µg/mL or 20 µg/mL) from the appropriate stock standard.

8.14.2.1 If the surrogate (decachlorobiphenyl, (DCB)) is used, it shall be added to the IPS to result in a concentration of 0.005 µg/mL.

8.14.2.2 To prepare the IPS along with DCB, add 10 mL of Aroclor 1016/1260 at 0.1 µg/mL and 0.033 mL of DCB at 15 µg/mL into 100 mL volumetric flask. Dilute to 100 mL volume with *iso*octane. Mix well. This yields 0.01 µg/mL IPS and 0.005 µg/mL of DCB.

8.14.2.3 The following additional standards shall be run once (at 0.1 µg/mL) to demonstrate the Aroclor patterns and be mixed if preferred.

Aroclor	Mix with the following:
1268	1221 or 1232 or 1242 or 1248 or 1254
1262	1221 or 1232 or 1242 or 1248
1254	1221

8.14.3 *Individual Working Standards (Tier 2–Quantitation)*—Working standards are typically prepared in *iso*octane at concentrations of 0.02 µg/mL, 0.05 µg/mL, 0.1 µg/mL, 0.3 µg/mL and 0.5 µg/mL for Aroclors 1016 and 1260. All other Aroclors are prepared at the mid level concentration (0.1 µg/mL) for the single point calibration. An alternative calibration range may be used as long as the criteria for linearity of the calibration range is documented.

8.14.3.1 Aroclors 1016 and 1260 shall be a mixed standard. The following additional standards shall be run once (at 0.1 µg/mL) to demonstrate the Aroclor patterns and may be mixed, if preferred.

Aroclor	May be mixed with:
1268	1221 or 1232 or 1242 or 1248 or 1254
1262	1221 or 1232 or 1242 or 1248
1254	1221

8.15 *Quality Control Standards*:

8.15.1 *Calibration Check Standard (CCS) (Tier 2–Quantitation)*—This standard contains 0.1 µg/mL (those who are interested in the 20 mg/Kg level with no compositing, use 0.2 µg/mL each) each of Aroclors 1016 and 1260 in hexane.

8.15.1.1 The surrogate concentration, if used, is 0.005 µg/mL.

8.15.1.2 *Example*—To prepare the CCS along with DCB, add 20 mL of Aroclors 1016/1260 to 0.5 µg/mL and 0.05 mL of DCB at 10 µg/mL into 100 mL volumetric flask. Dilute to 100 mL volume with *iso*octane. Mix well. This yields a 0.1 µg/mL of CSS and 0.005 µg/mL of DCB.

8.15.2 *Matrix Spiking Standard (Tier 2–Quantitation)*—The matrix spiking standard is to contain Aroclor 1268 at a concentration of 50 µg/mL in methanol. Laboratories working at lower calibration ranges will need to dilute this (for example, to 25 µg/mL).

8.16 *Copper Powder*, 200 mesh, 99 % min..

8.17 *Silica Gel*, 100 to 200 mesh.

9. Sampling

9.1 PCBs are hydrophobic compounds. Therefore, when sampling, all organic phases, including bottom sludge beneath aqueous phases, shall be sampled. Given the possible presence of alcohols and glycols, it is typically not acceptable to sample the organic phase only.

9.2 Headspace above stored standards and samples or extracts should be minimized such that the volume is less than 50 %.

9.3 Three mL of sample are required for each determination. No special sample preservation is required other than storage in a closed container with minimal headspace. It is accepted practice to use borosilicate glass containers with TFE-fluorocarbon-lined lids.

10. Preparation of Apparatus

10.1 *General Gas Chromatographic Conditions*—The first temperature profile (12 min run time) is used for Tier I screening method for the presence of Aroclor. The longer second temperature profile (17 min run time) is used for Tier II to quantitate the Aroclors present, but may also be used for Tier I, if desired.

10.1.1 *Rapid Screen Capillary Column Oven Temperature Profile (Tier I, 12 min run time)*:

Initial value	130°C
Initial time	2 min
Program rate	20°C/min
Final value	270°C
Final time	3 min
Carrier gas	hydrogen
Head pressure	depend on DCB RT (approximately 105 KPa (15 psi)) column flow: 3.1-3.2 mL/min
Make-up gas	nitrogen or argon: methane
Make-up gas rate	approximately 65 mL/min.
Splitless mode	
Purge off	0 min
Purge on	1.0 min
Purge vent	2.5 mL/min
Split vent	50 mL/min
Sample injection	2.0 µL
Injector inlet system	250°C
Detector	315°C

10.1.2 *Quantitation Capillary Column Oven Temperature Profile (Tier II, 17 min run time; may also be used for Tier I analysis*:

Initial value	125°C
Initial time	3 min
Level I	
Program rate	12°C/min
Final value	270°C
Final time	2 min
Carrier gas	hydrogen
Head pressure	Depend on DCB RT (approximately, 105 KPa (15 psi))
Column flow	3.1 mL/min (approximately at 270°C)
Make-up gas	nitrogen
Make-up gas rate	approximately 65 mL/min
Splitless mode	
Purge off	0 min
Purge on	1.0 min

Purge rate	50 mL/min
Sample injection	2.0 µL
Injector inlet system	250°C
Detector	315°C

11. Calibration and Standardization

11.1 *Calibration*:

11.1.1 *Tier 1–Screening Method*—Aroclors are multi-peak chemical mixtures that have very unique identification patterns. All Aroclors shall be run individually or in mixtures at 0.1 µg/mL on each channel performing screening to produce reference patterns. It is important to note that some of these patterns have the same constituents and that some Aroclors are quantitated using the same peaks (such as Aroclors 1016 and 1232 or 1242). When screening for Aroclors, a visual determination is made by the following key items:

11.1.1.1 Aroclor pattern—(a) same singlets, doublets, and triplets present in the reference chromatograms, and (b) same relative peak heights between peaks in the sample chromatogram and the reference chromatogram.

11.1.1.2 Retention time shifts should be very consistent between the standard and the sample peaks.

11.1.1.3 All samples in which an Aroclor is detected (using Tier I) require a judgment concerning the amount. The recognized Aroclor pattern shall be compared to the IPS (0.01 µg/mL or 0.1 µg/mL). If the overall level of the suspected Aroclor pattern is equal to or greater than overall level of the IPS pattern, then Tier II analysis may be used to quantitate the sample. If multiple Aroclors are suspected, a Tier II analysis may be run to help resolve the mixture.

11.1.1.4 Recovery control limits for the surrogate are 40 to 150 % recovered. If the recovery is outside of these limits, see Annex A1.

11.1.2 *Tier I Calibration Check*—An instrument performance standard (IPS) at 0.01 µg/mL of Aroclor 1016 and 1260 is used to check the instrument sensitivity *once a day or every 20 samples, whichever is more frequent* (typically laboratories using ten samples compositing shall use the 0.01 µg/mL standard to achieve a detection limit of 5 µg/mL of Aroclor in any individual sample). Sample results will be compared qualitatively with the daily IPS. (See the Calculation section 13).

11.1.2.1 Tabulate the sum of the areas or the data system calculated amount of the five major peaks for each of the Aroclors 1016 ad 1260 in the instrument performance standard. The response shall be within 50 % of the initial response. Initial response shall be established by averaging the response of a minimum of five injections of the instrument performance standard (IPS). If the limit is exceeded, new limits may need to be established.

11.1.2.2 Likewise, the expected response for the surrogate, if used, is established by averaging the areas of DCB in the five initial IPS analyses.

11.1.2.3 The surrogate also may be used for retention time control. It is recommended that column flow be adjusted so DCB elutes between 10.5 to 11.5 min using the 12 min GC program. (This will typically require a column head pressure of 105 to 112 kPa.) (Alternatively, the retention time should be 15 to 16.5 min using the 17 min program.)

11.1.3 *Tier 2–Quantitative Method*—The GC data system must be calibrated for both Aroclors 1016 and 1260, using five peaks for each Aroclor. [For example, when using an integrator, divide the standard amount by the number of peaks being used. Using five peaks on a 0.5 µg/mL standard would assign 0.1 µg/mL to each peak. This will allow for a calibration table to be made, yielding response factors for each peak at the five levels of calibration. Set up a calibration table in the method file of the integrator or data system that is to be used. Calculate an average response factor for each of five peaks for both Aroclors. Calculate the standard deviation of the average response factor for each peak of the Aroclor using the following calculation.

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - X)^2}{n - 1}} \quad (3)$$

where:

S = standard deviation,
X_i = each observed value,
X = the arithmetic mean of observed values, and
n = total number of calibration points.

11.1.3.1 Calculate the percent relative standard deviations (% RSDs) for the response factors of the calibrated peaks for each Aroclor from the formula below. The acceptance criteria for the % RSD for each Aroclor is ≤20 %. If the average % RSD is greater than 20 % for either Aroclor, then linearity over the desired calibration range for that instrument has not been demonstrated.

NOTE 3—The % RSD is 100 % multiplied by the result of Eq 3 (s) divided by the arithmetic mean (X).

11.1.3.2 When samples are to be analyzed, instrument control is verified by analyzing the CCS and the percent difference (% D) is calculated. *The acceptance criteria is within +30 % for each AROCLOR in the CCS (1016 and 1260).*

11.1.3.3 If either Aroclor 1016 or 1260 is out of control for the daily CCS, corrective action shall be taken and a CCS reanalyzed. If corrective action does not correct the problem, then a new five point calibration curve shall be created.
Percent difference (% D)

$$\%\ D = \frac{Amt_I - Amt_C}{Amt_I} \times 100\ \% \quad (4)$$

where:

Amt_I = amount in standard, and
Amt_C = calculated amount from current CCS.

11.1.3.4 Calibration for Aroclors other than Aroclor 1016 and Aroclor 1260 will be performed by analyzing standards at the concentration representing the midpoint of the calibration range selected. For example, if calibration is desired over the range of 0.02 µg/mL to 0.5 µg/mL, then the 0.1 µg/mL standards shall be used for calibration. Therefore, a five point calibration shall be performed for Aroclors 1016 and 1260 and a one-point calibration shall be performed for all remaining Aroclors.

11.1.3.5 After the linearity of the system has been demonstrated, and each of the remaining Aroclors has been analyzed using middle level concentration, recalibration will be required

only when the calibration check standard criteria is met. Old calibration curves may not be used again, other than to review data generated using those calibration curves.

11.2 *Standardization*:

11.2.1 *Surrogate Recovery*—Recovery control limits for the surrogate are 40 to 150 % recovered.

11.2.1.1 If the recovery is outside of these limits, see Annex A1.

11.2.2 *Method Blank*— For every 20 samples or batch, whichever is more frequent, a method blank shall be prepared by processing the extraction solvent (with surrogate, if used) through the same clean-up as that used for the samples. This is to detect possible contamination picked up during the sample clean-up process.

NOTE 4—A batch is the group of samples prepared at the same time. A batch may not exceed 20 samples.

11.2.3 *Calibration Check Standard (CCS) (Tier II only)*—A 0.1 µg/mL standard (or 0.2 µg/mL) obtained from a source separate from the intermediate standard and containing Aroclors 1016 and 1260 is the CCS which is used to verify the validity of the five-point calibration curve. The calculated results for the CCS shall agree with the current calibration curve to within ±30 % percent difference (% D). If the CCS results indicate that the calibration is outside control limits, and routine maintenance does not correct the problem, then the GC/ECD must be recalibrated.

11.2.4 *Matrix Spike (MS) Samples (Tier II only)*—For every batch or 20 samples, whichever is more frequent, a sample requiring Tier II analysis shall be selected in an unbiased manner and spiked with Aroclor 1268. These results shall be documented, with an example shown in Appendix X2.

11.2.4.1 1.0 mL of 50 µg/mL of Aroclor 1268 (25 µg/mL, if working at lower calibration range) is added to the sample chosen for spiking. Matrix spiked sample recovery limits are from 60 to 140 %, providing any Aroclor present in the sample before spiking does not exceed five times the spike level.

$$\% \text{ Recovery} = \frac{\text{Recovered amount}}{\text{Spiked amount}} \times 100\ \% \quad (5)$$

11.2.5 *Matrix Spike Duplicate (MSD) Sample (Tier II only)*—Every batch or 20 samples, whichever is more frequent, precision data is generated using a matrix spike duplicate. Acceptance criteria is 20 % relative percent difference (RPD) for the duplicate analyses.

11.2.5.1 RPD is calculated from the absolute difference between duplicate percent recovery results D_1 and D_2 divided by the mean value of the duplicates.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100\ \% \quad (6)$$

12. Procedure

12.1 *Compositing*—It is common to analyze mixtures of multiple samples, called composites, if a large number of samples are analyzed. This approach is described in Annex A3.

12.2 *Sample Preparation Procedure*:

12.2.1 *Liquid Samples*—Accurately pipette 3.0 mL of sample into a tared 40 mL vial (fitted with a TFE-fluorocarbon-lined cap) and weight. *If the results are calculated by weight* accurately weigh the sample and record the weight. Spike this sample with 100 µL of decachlorobiphenyl surrogate working standard.

12.2.1.1 Add 27 mL acetone/hexane to the vial, producing a 1:10 dilution. Cap it and vortex vigorously for at least 30 s. If the sample is not completely miscible with acetone/hexane, add more acetone to reach a total of approximately 30 mL extract and vortex again. (Alternatively, place capped vial in sonic bath for 5 min.)

12.2.2 *Solid, Semi-solids, Sludge Samples*—Weigh accurately 3.0 g of sample into a 40 mL vial fitted with a TFE-fluorocarbon-lined cap. Spike this sample with 100 µL of decachlorobiphenyl surrogate working standard. Add 30 mL of acetone/hexane to the vial for a 1:10 dilution. Vortex for at least 30 s.

12.2.2.1 If the sample does not totally dissolve, vortex again or place capped vial in sonic bath for 5 min. This shall provide adequate contact whether or not any further dissolution occurs.

12.2.3 *Matrix Spike and Matrix Spike Duplicate Samples*—Add 1.0 mL of spiking solution to the sample just after the addition of the surrogate and prior to the addition of the acetone-hexane solvent.

12.2.4 *Centrifuge*—If sediment is visible, centrifuge the extract to separate out the sediment.

12.3 *Sample Clean-up*—Clean-up is not required for all samples; however, interference problems due to the presence of other chemical species may usually be addressed using the procedures found in Annex A4.

12.4 *Gas Chromatographic Analysis Sequence*—Samples are analyzed in a set referred to as an analysis sequence.

12.4.1 *Tier 1–Screening*:

12.4.1.1 *Standards Sequence (initially and optionally with recalibrations)*—(a) Aroclor 1016/1260, at selected IPS level (5 times) and (b) The following may be mixed as described below and shall be analyzed at 0.1 µg/mL each (for 20 mg/Kg level of interest use 0.2 µg/mL).

Aroclor	1221
Aroclor	1232
Aroclor	1242
Aroclor	1248
Aroclor	1254
Aroclor	1262
Aroclor	1268

12.4.1.2 Some of the standards in 12.4.1.1 may be run as mixed standards:

Aroclor	May be Mixed With
1268	1221 or 1232 or 1242 or 1248 or 1254
1262	1221 or 1232 or 1242 or 1248
1254	1221

12.4.1.3 *A Typical Analysis Sequence*—A typical analysis sequence includes (a) reagent blank (optional), (b) Instrument Performance Standard (IPS) (every 20 samples or every day, whichever is more frequent), (c) method blank, and (d) Samples 1 to 20.

12.4.1.4 Repeat this sequence as long as the system meets the IPS criteria.

12.4.2 *Tier 2–Quantitation*:

12.4.2.1 *Standards Sequence*—The standards sequence includes (a) reagent blank, (b) Aroclor 1016/1260 (5 point

calibration), (c) Aroclor 1268 mid-level standard, (d) Mid level standard of suspected Aroclors if not, 1016 or 1260, and (e) (CCS, five times, to establish DCB response, if DCB is not spiked in 1016/1260 standards.)

12.4.2.2 *A Typical Analysis Sequence*—A typical analysis sequence includes (a) reagent blank (optional), (b) CCS (1016/1260 mid standard), (c) method blank, (d) Samples 1 to 20, (e) matrix spike sample, and (f) matrix spike duplicate.

12.4.2.3 Repeat this sequence as long as the system meets the quality assurance criteria.

12.5 Inject 2 µL of the sample extract into the gas chromatograph using an autosampler or a manual injection.

12.6 Set the data display (printer or video screen) conditions so that a mid point calibration standard shall be full scale on the chromatogram.

12.7 If the results exceed the calibrated range of the system, and quantitation is desired, the extract shall be diluted and reanalyzed within the calibration range.

13. Calculation

13.1 *Screening*—Aroclors are made up of numerous congeners and so the chromatograms are multi-peak. Often the chromatogram of the sample may not exactly match that of the standard due to factors such as environmental exposure, interferences not easily removed by cleanup techniques, and the presence of multiple Aroclors.

13.1.1 Visual determinations are made by comparing the chromatogram with the reference chromatograms. Set the data display conditions so that a 0.1 µg/mL standard is full scale on the chromatogram.

13.1.2 All samples in which an Aroclor is detected require a judgment concerning the amount. The recognized Aroclor pattern shall be compared to the IPS (0.01 µg/mL or 0.1 µg/mL). If the overall level of the suspected Aroclor pattern is equal to or greater than the overall level of the IPS pattern, then Tier II analysis may be used to quantitate the PCBs.

13.1.3 If Aroclor identification is prevented by the presence of interferences, additional sample preparation is required. All composites having such interferences shall be analyzed as individual samples. Individual samples may be diluted prior to analysis, but it must be remembered that the detection limit of the analysis has been changed. Used oil samples shall not be diluted beyond 1:100 during initial screening analysis to meet the regulated level of interest (2 µg/mL).

13.1.4 If PCBs are detected, (when compared to the IPS criteria above) the result is reported Positive. If no PCBs are detected above the IPS level, the result is Negative.

13.1.5 When screening for Aroclors, visual determination is made by the following key items:

13.1.5.1 *Aroclor Pattern*—The Aroclor pattern includes (a) Same singlets, doublets, and triplets present in the reference chromatograms, and (b) Same relative peak heights between peaks in the sample chromatogram and the reference chromatogram.

13.1.5.2 Retention times shall be very consistent between the standard and the sample peaks.

13.2 *Data System Quantitation*—The GC data system shall be calibrated for each Aroclor using a minimum of five peaks (with exception of Aroclor 1221, which uses three peaks) for each Aroclor. For use with integrators, divide the standard amount by the number of peaks being used (for example, using five peaks on a 0.5 µg/mL standard would assign 0.1 µg/mL to each peak.) For some data systems, the total standard amount may be assigned to each peak. This will allow for a calibration table to be made, yielding response factors for each peak.

NOTE 5—Response factors are based on amount/area for some data systems, while response factors are based on area/amount for others.

13.2.1 Quantitation of Aroclor in samples requires selecting five peaks that are free of interferences (minimum of three peaks, if interferences present) in the TIER II analysis, and assigning the appropriate response factor to each peak.

13.2.2 Aroclors 1016/1260 are quantitated using a five point calibration. All other Aroclors use a single point calibration. Samples exceeding the working range shall be diluted prior to analysis so that quantitation is performed within the calibration range.

13.2.3 As an example, the data system shall be set up to provide results in µg/mL. The following equation yields the concentration of Aroclors in mg/kg on a wet weight basis.

$$\text{Aroclor (mg/kg) (wet)} = \frac{\text{Aroclor (µg/mL)} \times \text{dilution volume}}{\text{sample wt (g)}} \times \frac{10\ \text{mL}}{1\ \text{mL}} \tag{7}$$

After determining the water content, using Test Method E203, the concentration of Aroclor in a sample is *corrected for dry weight* of the sample by the following:

$$\text{Aroclor mg/kg (dry)} = \frac{\text{Aroclor g/kg (wet)}}{(100 - \%\ \text{water}) / 100} \tag{8}$$

Water content is usually determined by Test Method E203.

13.3 *Manual Quantitation*—Quantitate Aroclor samples by comparing the area of five sample peaks (minimum of three, if interferences present) to the area of the same peaks from appropriate (mid level) reference standards. Use only those peaks from the sample that are attributed to Aroclors. These peaks shall be present in the chromatogram of reference materials. See Aroclor Calculation Work Sheet (Appendix X2.) for an example of how to perform manual quantitation.

13.3.1 Use the following formulas to calculate the concentration of each of the Aroclor peaks in the sample (wet weight):

$$\text{Aroclor (wet) Peak No. 1 (µg/mL)} = \frac{\text{sample area} \times \text{dilution volume (mL)}}{\text{standard area} \times \text{sample volume (mL)}} \times (\textit{standard concentrate µg/mL}) \times \frac{\textit{dilution volume No. 1}}{\textit{aliquot volume}} \tag{9}$$

13.3.2 This is repeated for each peak used, and the results summed to give the wet concentration.

The result may be converted from µg/mL to µg/g by:

$$\text{Aroclor (µg/g)} = \frac{\text{total Aroclor (µg/mL)}}{\text{specific gravity of sample}} \tag{10}$$

Specific gravity may be the measured value or calculated by (sample weight/3 mL).

Care shall be taken in handling viscous samples as the volumes may not be correct. In those cases the measured sample weight shall be used.

A simplified formula using sample weight is:

$$\text{Aroclor (µg/g, mg/Kg)} = \frac{\text{total Aroclor (µg/mL)} \times \text{dilution volume (mL)} \times \text{additional dilution factor (mL/mL)}}{\text{sample weight (g)}} \quad (11)$$

The concentration of Aroclor in a sample is corrected for dry weight of the sample by the following:

$$\text{Aroclor (mg/Kg) (dry)} = \frac{\text{Aroclor (mg/Kg) (wet)}}{(100 - \%\ \text{water}) / 100} \quad (12)$$

13.4 *Mixed Aroclors*—For routine Tier II samples showing evidence of mixed Aroclors, select a minimum of three peaks lacking significant interference for each identified Aroclor and quantitate. Report the amount for each Aroclor separately.

NOTE 6—This approach will normally overstate the PCB concentration and, thus, is considered to be a conservative approach.

13.4.1 Since mixed Aroclors present special problems in quantitation, it is permissible to prepare individualized mixed standards in an attempt to match the suspected sample concentrations and obtain greatest possible accuracy. This will involve a judgment about what proportion of the different suspected Aroclors to combine to produce the appropriate reference material. A calibration standard is then made using this blend. Use only those peaks from the sample that are attributed to chlorobiphenyls. These peaks shall be present in the reference blend.

14. Precision and Bias [9]

14.1 The precision of this test method was determined by statistical examination of interlaboratory study results. All data was generated using GC/ECD.

NOTE 7—Ten samples in the range of 4.3 to 61.4 mg/kg PCBs with water content in the range 0 to 72 % were analyzed in duplicate at ten different laboratories using ten operators.

14.1.1 *Repeatability*— The difference between two results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only one case in twenty:

$$\text{Repeatability} = 0.16\ (X^{1.1}). \quad (13)$$

where X is the average PCB concentration in mg/kg.

14.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical materials would in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.73\ (X^{1.1}). \quad (14)$$

where X is the average PCB concentration in mg/kg.

14.1.3 Precision estimates for selected values of X are set out in the following Table 1:

14.2 *Bias*—A reliable quantitation of bias was not possible due to the manner in which the samples were prepared and aliquoted. However, the method tends to produce a result that is low. This tendency is mitigated to some extent through the use of a surrogate as described in Section 11.

15. Keywords

15.1 gas chromatography; GC/ECD; PCBs; polychlorinated biphenyls

TABLE 1 Repeatability and Reproducibility

X, mg/kg	Repeatability	Reproducibility
5	0.9	4.3
10	2.0	9.4
20	4.3	20.2
50	11.8	55.5

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1413.

ANNEXES

(Mandatory Information)

A1. POOR RECOVERY TROUBLESHOOTING

A1.1 If the necessary recovery is outside of limits, the following may be useful in identifying the source of the problem:

A1.1.1 Check for proper dilution factor,

A1.1.2 Check for dirty insert and drifting baseline,

A1.1.3 Recheck the recovery calculation,

A1.1.4 Look for major interfering peak,

A1.1.5 Look for sample preparation problems,

A1.1.6 Reanalyze sample, on different channel, if possible, and

A1.1.7 Re-evaluate surrogate standard.

A1.2 If none of the above results in acceptable surrogate recoveries:

A1.2.1 Break composites into individual samples, prepare samples, and reanalyze.

A1.2.2 For individual samples, if the recovery is still outside of the limits after a second preparation and analysis, this demonstrates confirmation of a matrix effect and is reported as such.

A2. SILYNIZATION

A2.1 There are many possible pathways to deactivate glass surfaces. These are basically divided into vapor and liquid methods. Two examples follow:

A2.2 Liquid Silynization

A2.2.1 Prepare 5 % (volume) solution of dimethyldichlorosilane in toluene.

A2.2.2 Place the clean glass parts to be treated in a wide-mouth jar or beaker large enough to allow the solution to cover the parts.

A2.2.3 Heat the solution with the glass part submerged to the boiling point and continue gentle boiling for 30 min.

A2.2.4 Allow the parts to drain and rinse with methanol.

A2.2.5 Oven dry the glass parts at 100°C for at least 20 min.

A2.3 Vapor Silynization

A2.3.1 Place the clean glass parts in a wide-mouth glass jar.

A2.3.2 Add 1 mL of concentrated dimethyldichlorosilane to the jar.

A2.3.3 Screw a lid on the jar and place it in an oven preheated to 50°C for at least 2 h.

A2.3.4 Remove jar and open lid.

A2.3.5 Rinse glass parts with methanol.

A2.3.6 Oven dry the glass parts at 100°C for at least 20 min.

A2.4 Silanized glass parts shall be stored in the oven or in a desiccator with activated desiccant in the bottom until needed.

A3. COMPOSITING

A3.1 It is common to analyze mixtures of multiple samples, called composites, if a large number of samples are analyzed. Positive identification of PCBs within a composite usually then requires that the individual samples making up the composite be reanalyzed individually to identify the source of the PCBs.

A3.1.1 If samples are to be run as a composite, rather than individually, transfer 1 mL of a representative portion of each sample (1 g, if a solid) into a vial by means of disposable pipet. Larger volumes than 1 mL may be used, if required by sample matrix to obtain a representative sample. Mix them well to get a composite of up to ten samples. If using the surrogate, be sure to consider the dilution factor.

A3.1.2 *Compositing of Samples Representing Varying Volumes*

A3.1.2.1 If receipt samples representing varying volumes (for example, multiple partially-filled drums) are to be composited, it is important to ensure that each unit volume (for example, each litre) is equally represented. If preliminary composites have been generated outside of the laboratory, the analyst making the composite will need to understand the volume of material represented by each sample.

A3.1.2.2 The composite is generated by using a pipette to transfer proportional amounts (for example, 1 mL/drum) into a vial. For example, if a sample entering the laboratory represents eight drums, place 8 mL of that sample in the vial. Assuming the 20 drum maximum is reached, place 20 mL of material in the vial. Vortex well.

A3.2 When Aroclor is detected in a composite at a level equal to or above the IPS, all samples included in the composite shall be analyzed individually.

A4. SAMPLE CLEAN-UP

A4.1 Clean-up is not required for all samples; however, the following clean-up procedures will solve most interference problems to obtain analyzable chromatograms. Use the particular clean-up method demonstrated to yield acceptable results, if a lab is familiar with the type of matrix in their samples.

A4.2 *Magnesium Silicate Slurry and Acid Clean-Up (Individual Samples)*—Pipet 1.0 mL of the 1:10 diluted extract to a 20 mL vial containing 9.0 hexane. Further information is provided in EPA Method 3620.

NOTE A4.1—For Composited Sample (see Annex A3)—No further dilution required beyond the initial 1:10, unless sample matrix requires it. Further dilutions may cause detection limits to rise above the level of interest.

A4.2.1 Add 3 mL concentrated sulfuric acid to the solution. Cap it with a TFE-fluorocarbon-lined cap and vortex well. Let it settle to allow phase separation. More than one acid clean-up may be used if the acid layer is discolored after phase separation. Record the number of washings performed.

A4.2.2 Transfer about 8 mL of this solution (free from acid) into another 20 mL vial, containing about 0.25 g magnesium silicate and 0.5 g of anhydrous sodium sulfate. For used oil samples it may be preferable to use 0.25 g anhydrous sodium sulfate and 0.5 g silica gel. Vortex well and allow the magnesium silicate to settle by gently tapping the vial or by centrifuging for about 2 min.

A4.2.3 The final solution after clean-up is transferred into a GC vial.

A4.3 *Silica Gel Slurry Clean-Up*—Follow the procedure for magnesium silicate clean-up, substituting silica gel for magnesium silicate. Samples that have undergone acid clean-up and magnesium silicate slurry and that still display interferences

shall undergo an additional silica gel slurry cleanup. The extract, after acid and magnesium silicate slurry clean-up, is pipetted to a 20–mL vial containing 0.5 g silica gel and vortexed. Further information can be found in EPA Method 3630.

A4.4 *Magnesium Silicate Column Clean-up*:

A4.4.1 Plug a 10 mL disposable pipet with glass wool. Add the equivalent depth of 1 mL anhydrous sodium sulfate. Add the equivalent depth of about 2 mL magnesium silicate. Top off the column with the equivalent depth of 1 mL anhydrous sodium sulfate. Tap the column gently. Optionally, one may use a commercial clean-up cartridge (1000 mg packing).

A4.4.2 Put a container beneath the column to catch the eluate. Wet with 2 to 3 mL hexane. Transfer 1 to 2 mL of the acid cleaned sample to the column.

NOTE A4.2—Do not allow any acid to go into the column.

A4.4.3 Discard the eluate. When the extract level reaches the top of the upper sodium sulfate, add 5 mL more of the extract into the column. Collect the eluate in a GC autosampler vial.

A4.5 *Combined Magnesium Silicate and Silica Gel Column Clean-up*—Prepare a silica gel column using a 10–mL disposable pipet plugged with glass wool. On top of the glass wool place 1 mL silica gel. Add to the column about 1 mL magnesium silicate and then 1 mL anhydrous sodium sulfate. Tap the column gently. Transfer 5 mL of already acid cleaned sample to wet column (see EPA Method 3620)

A4.5.1 Discard the eluate. When the extract level reaches the top of the upper sodium sulfate, add about 3 mL more of the extract. Collect about 2 mL in a GC vial.

A4.6 *Copper Clean-Up*—Elemental sulfur in samples will cause interferences in the GC/ECD analysis. Presence of sulfur will be indicated by a yellow extract color and big interfering peaks. Check sample history for the presence of sulfur. To remove the sulfur, transfer as much as possible of the extract (usually 7–8 mL) into a vial, add 0.5 g powdered copper; seal, and vortex vigorously. Allow the copper sulfide to settle. Remove about 4 mL of the hexane solution and perform an acid wash, if needed, to clear the hexane phase. Further information can be found in EPA Method 3660. (**Warning—** Handle mercury with care. Keep a minimum amount on site.)

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLE CALIBRATION TABLE AND CHROMATOGRAM

X1.1 See Table X1.1 for an example calibration table and chromatogram.

X1.2 Chromatogram

X1.2.1 The chromatogram in Fig. X1.1 includes the retention time in minutes for each major peak in a mixture of Aroclors 1016 and 1260. The five peaks for each Aroclor used for quantitation are clearly marked (for example, 7.295–1016).

X1.2.2 The decachlorobiphenyl surrogate internal standard is also shown and designated as 15.220–DCB.

X1.2.3 This chromatogram was generated using the longer run time described in 10.1.2.

TABLE X1.1 Calibration Table

Retention Time	Peak Name	Average Response	Standard Deviation	Peak % RSD
7.520	1016	4543228	528055	11.62
7.870	1016	2743429	214966	7.84
8.390	1016	6378927	360623	5.65
10.150	1016	1851507	129351	6.99
10.540	1016	1919245	150403	7.84
11.540	1260	4325072	493032	11.40
11.930	1260	6012570	639705	10.64
12.370	1260	5503749	470809	8.57
13.430	1260	5830717	428980	7.36
13.880	1260	3118738	164338	5.27

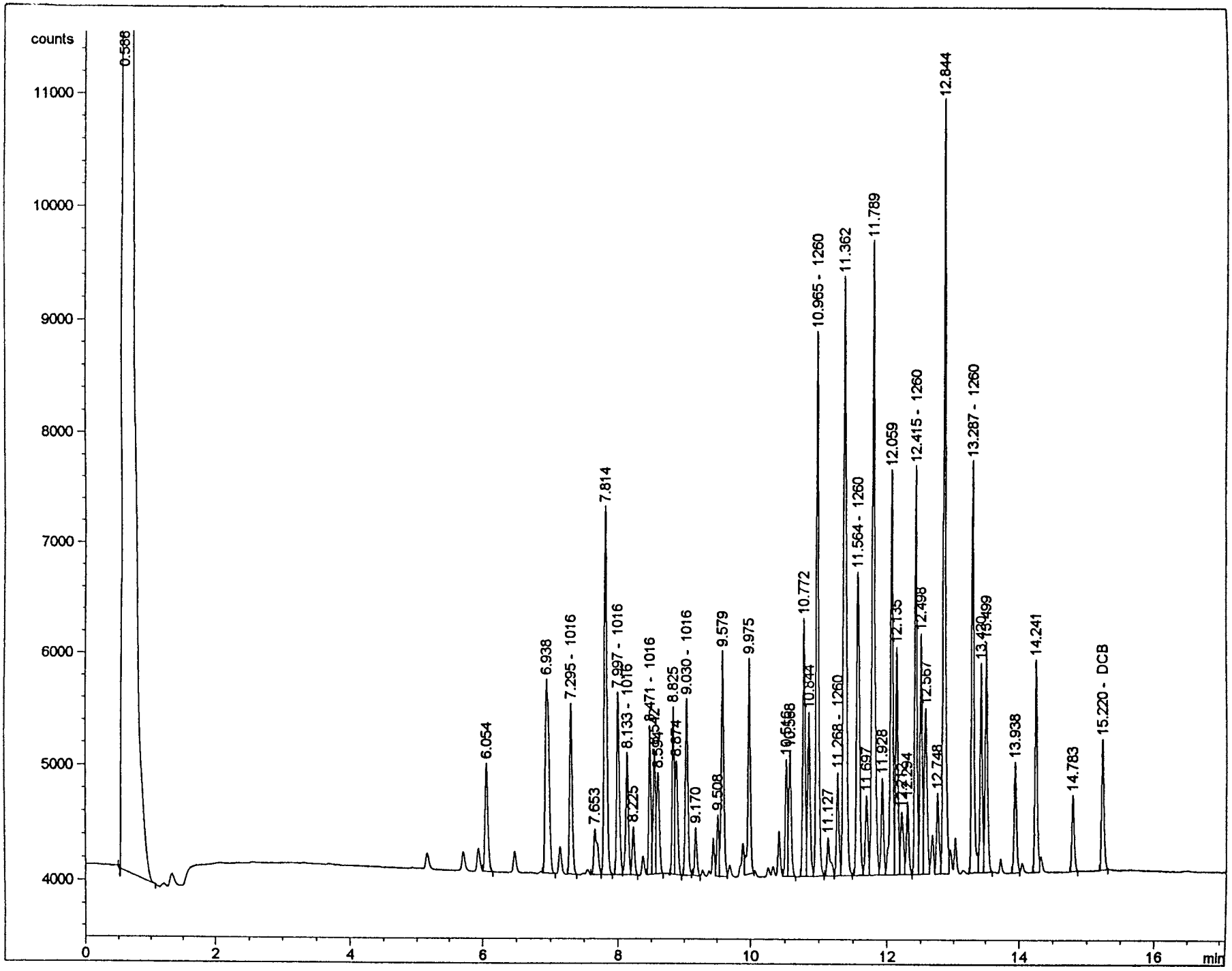

FIG. X1.1 Sample Chromatogram

X2. WORKSHEETS

X2.1 See Fig. X2.1 and Fig. X2.2 for calculation worksheets.

GC IDENTIFICATION: GC#_____ CHANNEL#_____ SAMPLE CONTROL#_____

AROCLOR STANDARD:_____ _____UG/ML
ANALYST/DATE:_____
REVIEWER/DATE:_____

PEAK #	R.T. (MIN)	AREA	PEAK #	R.T. (MIN)	AREA
1.	_____	_____	1	_____	_____
2.	_____	_____	2	_____	_____
3	_____	_____	3	_____	_____
4.	_____	_____	4.	_____	_____
5.	_____	_____	5.	_____	_____
TOTAL		_____	TOTAL		_____

CALCULATION:

$$\text{THEOR. PCB SPK (AR 1268)} = \frac{25\ \text{ug/mL} \times 1.0\ \text{mL}}{31.0\ \text{mL} \times 10\ \text{mL}} = 0.0806\ \text{ug/mL}$$

$$\text{**EXPERIMENTAL RESULT} = \frac{\text{TOTAL SAMPLE AREA}}{\text{TOTAL STANDARD AREA}} \times \text{STANDARD CONC}$$

$$= \frac{\quad}{\quad} \times 0.1\ \text{ug/mL} = \frac{\quad}{\quad}\ \text{ug/mL}$$

$$\%\ \text{RECOVERY OF AR 1268} = \frac{\text{EXPERIMENTAL RESULT}}{\text{THEORETICAL RESULT}} \times 100$$

$$= \frac{\quad}{0.0806\ \text{ug/mL}} \times 100 = \_\_\_\_\_\ \%$$

**AFTER SUBTRACTING BACKGROUND PCB IN THE SAMPLE.

FIG. X2.1 PCB Calculation Work Sheet for Spikes

SAMPLE CONTROL # : _____ LAB # : _____

GC SYSTEM IDENTIFICATION : _____

COLUMN ID : _____

30 m RTX-5 (or DB-5), 0.32 mm ID capillary column,
with 0.25 µm film thickness.

INSTALLED BY / DATE : _____

AROCLOR TYPE : _____ ANALYST / DATE : _____

AROCLOR STD : _____ µg/mL REVIEWER / DATE : _____

	STD PEAK R.T.	STD PEAK AREA	STD AMT/ µg/mL	RF AMT / AREA	SAMPLE PEAK R.T.	SAMPLE PEAK AREA	SAMPLE PEAK AMT (AREA*RF)
1.	_____	_____	_____	_____	_____	_____	_____
2.	_____	_____	_____	_____	_____	_____	_____
3.	_____	_____	_____	_____	_____	_____	_____
4.	_____	_____	_____	_____	_____	_____	_____
5.	_____	_____	_____	_____	_____	_____	_____

Total Aroclor Amount (µg/mL) _____

Calculation: Aroclor Concentration in mg/Kg (µg/g)

$$= \frac{\text{Aroclor Amt (µg/mL) X Dil. Volume mL X Dil. Factor}}{\text{Sample Weight (g)}}$$

$$= \frac{\qquad\qquad}{(\qquad\qquad)}$$

= _____ mg/Kg of Aroclor _____

% Water (By Karl-Fischer / NVR / Specified Other) = _____ %
% Dry Weight = _____ %
Aroclor Concentration Corrected For Dry Weight = _____ mg/Kg

FIG. X2.2 AROCLOR Calculation Work Sheet