Designation: D 4947 – 00

Standard Test Method for Chlordane and Heptachlor Residues in Indoor Air[1]

This standard is issued under the fixed designation D 4947; 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 covers the sampling and analysis of indoor atmospheres for residues of chlordane and heptachlor.

1.2 This test method is based upon the collection of chlordane and heptachlor from air onto polyurethane foam (PUF) and analysis by gas chromatography coupled with electron capture detection.

1.3 This test method is applicable to concentrations of chlordane varying from 0.1 to 100 μg/m^3 and heptachlor varying from 0.01 to 80.0 μg/m^3 with sampling periods to collect at least 0.25 m^3 of air. Detection limits will depend upon the conditions of the gas chromatography (GC) and the length of the sampling period.

1.4 *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 regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

D 3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)[2]

D 3687 Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method[2]

D 4185 Practice for Measurement of Metals in Workplace Atmospheres by Atomic Absorption Spectrophotometry[2]

D 4861 Practice for Sampling and Selection of Analytical Techniques for Pesticides and Polychlorinated Biphenyls in Air[2]

E 355 Practice for Gas Chromatography Terms and Relationships[3]

2.2 *EPA Methods:*

Compendium of Methods for the Determination to Toxic Organic Compounds in Ambient Air, EPA 600/R-96/010b[4]

2.3 *Other Documents:*

Indoor Sampling Guidelines for Termiticides[5]

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology D 1356, Practice E 355, and Practice D 4861 for definitions of terms used in this test method.

3.1.2 The term "chlordane" refers to a technical-grade mixture consisting mostly of chlorinated Diels-Alder addition products of cyclopentadiene and hexachlorocyclopentadiene. The mixture consists of 50 or more compounds, 10 of which are major components **(1).**[6] The isomers α-(or *cis*-) and γ-(or *trans*-) chlordane, heptachlor, and *trans*-nonachlor are among these.

3.1.2.1 The terms "chlordane" and "technical" chlordane are used interchangeably.

3.1.3 Heptachlor is a single chemical compound, which may be used alone or in formulations with technical chlordane. It is also a component of technical chlordane.

4. Summary of Practice

4.1 A low-volume (1 to 5 L/min) sampler is used to collect airborne chlordane and heptachlor on a sorbent cartridge containing PUF. The method is taken from Refs. **(2)** through **(4)** and Practice D 4861.

4.2 Chlordane and heptachlor are extracted from the sorbent cartridge with 5 % diethyl ether in hexane and analyzed on a gas chromatograph (GC) equipped with an electron capture detector (ECD).

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from the U.S. Department of Commerce, National Technical Information Service, Port Royal Road, Springfield, VA 22161.

[5] Available from Wood Protection Council, National Institute for Building Sciences, Washington, DC, 1987.

[6] The boldface numbers in parentheses refer to the list of references at the end of this standard.

4.3 Because of the possibility of interfering materials having similar retention times to chlordane and heptachlor peaks, column chromatography or the use of a second chromatographic column of a different type is necessary to obtain accurate identification and quantification. Mass spectrometry may be required for unambiguous determination.

5. Significance and Use

5.1 This test method is intended to be used primarily for non-occupational exposure monitoring in domiciles, public access buildings and offices.

5.2 Chlordane has been used widely as a general insecticide for crops (for example, cotton) and as a termiticide. Heptachlor is a major component of technical chlordane and an insecticide in its own right. Although their use in the United States was discontinued in 1988, residues of the chemicals may remain in indoor air for many years after application.

6. Interferences

6.1 The electron capture detector responds to a wide variety of organic compounds. It is likely that such compounds will be encountered as interferences during GC-ECD analysis. Although mass spectrometry can provide positive identification of chlordane and heptachlor, some laboratories do not possess such instrumentation.

6.2 Technical chlordane is a complex mixture of chemically-related chlorinated compounds, including 8 to 10 % by weight of heptachlor. Similar compounds (for example, DDT and its isomers) can cause difficulty in identifying and quantifying this multiple-component mixture.

6.3 In addition, contaminated glassware and sampling tubes can be a major source of error when attempting to quantitate multiple-component mixtures with an ECD. To minimize this source of error, careful attention to glassware cleaning and sample handling procedures must be followed.

6.4 General approaches that can be followed to minimize interferences are given as follows:

6.4.1 Chlordane and heptachlor can be cleaned up by column chromatography on Florisil[7]. See Ref **(5).**

6.4.2 Chlordane- and heptachlor-containing samples can be cleaned up with sulfuric acid treatment. See Ref **(6).**

7. Apparatus

7.1 *Air Sampler*—Refer to the appropriate section of Practice D 4861 for specifications on air sampling equipment.

7.2 *Equipment and Reagents for Sample Extraction and Concentration*—Refer to the applicable section of Practice D 4861 for required equipment and reagents.

7.3 *Equipment for Analysis*:

7.3.1 Gas chromatograph equipped with a Nickel-63 electron capture detector.

7.3.2 Gas chromatographic columns: A 15-m by 0.53 mm inside diameter bonded, crosslinked (50%-phenyl)-methylpolysiloxane[8] fused-silica capillary column, film thickness 3 µm, for quantitation; and a 30-m by 0.53-mm inside diameter poly(5% -diphenyl-95% dimethylsiloxane) fused-silica capillary column,[9] film thickness 1.5 µm, for confirmation.

7.3.3 Microsyringes, 5-µL volume.

8. Sampling Procedures

8.1 Follow the applicable section of Practice D 4861 for clean-up and proper storage of the PUF sampling plugs.

8.2 At least one assembled sampling cartridge from each batch should be analyzed as a laboratory blank prior to using. The blank level should be <0.10 µg/plug for chlordane, <0.01 µg/plug for heptachlor.

8.3 After the sampling system has been assembled and calibrated as described in Section 9, it can be used to collect air samples as described in 9.5.1 to 9.5.9 of Practice D 4861.

9. Calibration of Pump

9.1 Refer to the applicable Annex in Practice D 3686 or the applicable Annex Practice D 4185 for procedures to calibrate small volume air pumps. See also Practice D 4861.

10. Sample Extraction Procedure

10.1 All samples should be extracted within one week after collection in accordance with the procedures outlined in Section 10 of the Practice D 4861.

10.2 Adjust final volume of sample extract to 1 mL for analysis.

11. Analysis Procedures

11.1 Prepare analytical standard solutions of technical chlordane in pesticide-quality[10] 2,2,4-trimethylpentane ("isooctane"). Analytically pure standards of technical chlordane and heptachlor are available from several commercial sources.

11.2 When not in use, store standard solutions at 4°C or below and protect from light. Replace after six months, or sooner if comparison with check standards indicates a problem.

11.3 Chlordane and heptachlor are responsive to detection by GC/ECD at low concentrations, which will be dependent upon the condition of the chromatograph, columns (see 7.3.2) and detector.

11.4 A gas chromatograph (GC) with dual injector ports and dual electron capture detectors is recommended.

11.5 Set up both the quantitation and confirmatory GC columns in the same GC oven.

11.6 Provide helium carrier gas at a nominal flow rate of 10 mL/min at approximately 170 kPa (25 psig) to each column.

11.7 Set the temperature for both injectors at 235°C and the ECD at 350°C.

11.8 Allow samples and standard solutions to warm to room temperature before analysis.

[7] "Florisil" is a trademark of the Floridin Corp., Tallahassee, FL 32303. It is a natural magnesium silicate; it is available from several commercial sources.

[8] This column is available from several commercial sources under such trade names as OV-17, DB-17, SPB-17, and others.

[9] This column is available from several commercial sources under such trade names as DB-5, SPB-5, RTX-5, HP-5, OV-5, BP-5, and others.

[10] Glass distilled and certified for pesticides analysis by GC/ECD.

11.9 Set column temperature program for 60°C (2 min), then programmed to 140°C at 25°C/min, then to 270°C at 4°C/min.

11.10 Calibrate gas chromatograph by injecting 2 µL aliquots of standard solutions (See Practice D 3687 for technique) in order to establish response factors, linearity of the ECD, dynamic range, and retention time windows.

11.11 Set retention time windows at ±0.10 min for the quantitation primary column and ±0.05 min for the confirmatory column.

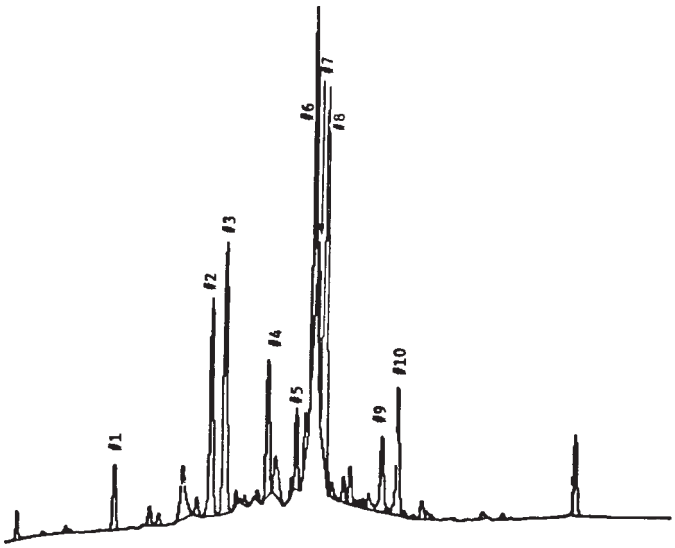
11.12 Typical chromatograms of technical chlordane are shown in Fig. 1 and Fig. 2 for the quantitation and confirmatory columns, respectively. The ten numbered peaks are to be used for identification and quantitation of chlordane in the sample.

11.13 Typical retention times for the two columns are given in Table 1.

11.14 Inject 2 µl of sample extract on quantitation column and obtain a tentative identification of technical chlordane by comparison of chromatographic peaks in the sample with those in the standard in accordance with the flow chart in Fig. 3 and the following steps:

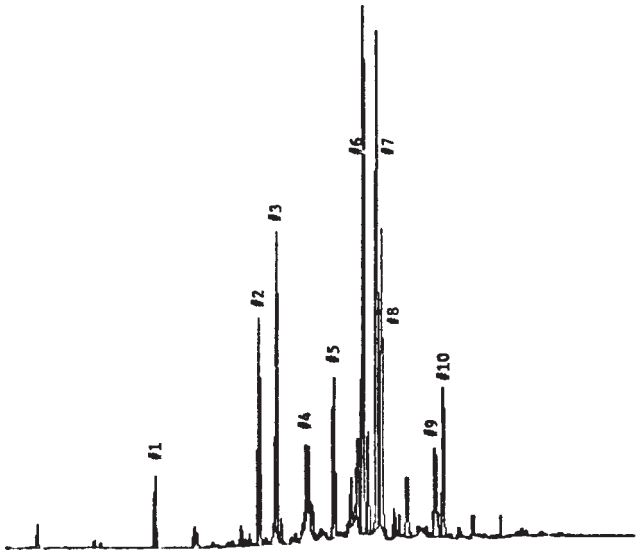
11.14.1 On a worksheet, list the measured retention times (in minutes) and corresponding areas of each chromatographic peak that appears to match any of the ten reference peaks from the standard.

11.14.2 Compare the retention time of each of the ten peaks in the sample chromatogram to the absolute retention time of the respective standard peak using a retention window of ±0.10 min around each standard peak. Draw a line through the retention time and area of all sample peaks that are outside the retention window. However, when a consistent shift is evident in the retention times of many of the sample peaks, the experienced analyst may expand the acceptable retention window in the direction of the shift.



NOTE 1—Refer to Table 1 for typical retention times of peaks.

FIG. 1 Typical Chromatogram of Technical Chlordane on Quantitation Megabore Column



NOTE 1—Refer to Table 1 for typical retention times of peaks.

FIG. 2 Typical Chromatogram of Technical Chlordane on Confirmatory Megabore Column

TABLE 1 Typical Gas Chromatographic Retention Times of Technical Chlordane Components[A]

Quantitation Megabore Column			Confirmatory Megabore Column		
Peak[B]	RT, min	Compound	Peak[C]	RT, min	Compound
1	12.74		1	16.56	
2	18.07		2	22.23	
3	18.80	Heptachlor	3	23.19	Heptachlor
4	21.17		4	24.98	
5	22.75		5	26.37	
6	23.64	γ-Chlordane	6	27.97	γ-Chlordane
7	23.87	*t*-Nonachlor	7	28.72	α-Chlordane
8	24.32	α-Chlordane	8	28.99	*t*-Nonachlor
9	27.51		9	31.97	
10	28.36		10	32.40	

[A] Refer to Section 11 for chromatographic conditions.
[B] Refer to Fig. 1.
[C] Refer to Fig. 2.

11.14.3 If all ten peaks qualify, tentative confirmation is obtained and the sample may be subjected to final confirmation by analysis on the DB-5 column in accordance with 11.15.

11.14.4 When only some of the ten peaks are present in the sample chromatogram, the priority order of peak presence for identification as chlordane is as follows:

11.14.4.1 α- and γ-Chlordane (peaks 8 and 6) (highest priority),

11.14.4.2 Heptachlor component (peak 3),

11.14.4.3 *Trans* -nonachlor (peak 7),

11.14.4.4 Last two components (peaks 9 and 10), and

11.14.4.5 Component immediately preceding heptachlor (peak 2).

11.14.5 If all seven of these peaks listed in 11.14.4 are present in the sample (for example, found to be within its retention window as in 11.14.2), then tentative assignment of technical chlordane is made.

11.14.6 If peaks 2, 3, 6, 8, 9, and 10 are present in the sample, then the tentative assignment of chlordane is made.

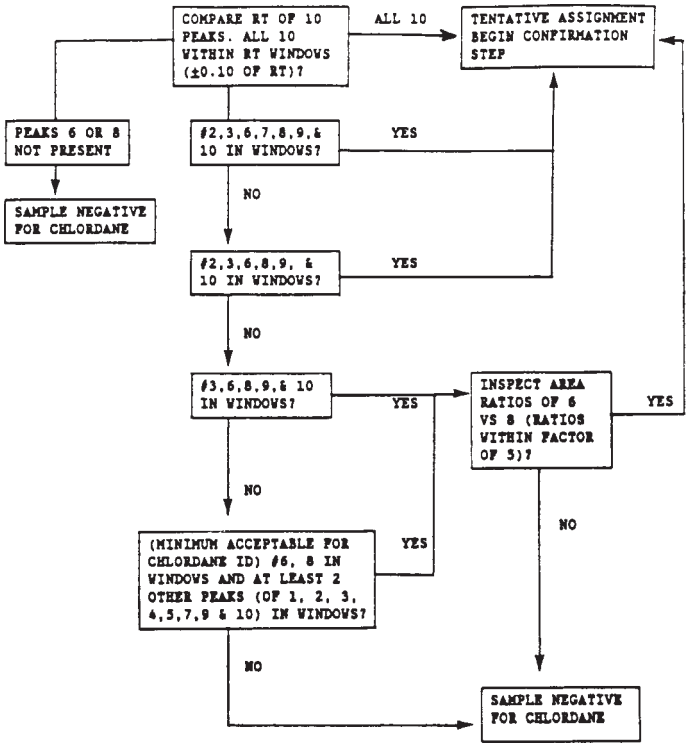


FIG. 3 Flow Chart for Tentative Identification of Chlordane Using the Primary Column

11.14.7 As illustrated in the flow chart in Fig. 3, when the number of peaks within the windows falls to four or five, the area ratios are compared as described.

NOTE 1—Components of similar volatility must be compared (for example, α-chlordane versus γ-chlordane).

11.14.8 When fewer than four of the ten peaks are present, the sample is considered negative for chlordane.

11.14.9 At a minimum, γ- and α-chlordane (peaks 6 and 8) must be present, along with at least two of the other eight peaks (peaks 1, 2, 3, 4, 5, 7, 9, or 10), and the areas of peaks 6 and 8 must be within a factor of 5 in order to consider the sample eligible to confirm for the presence of chlordane (for example, to proceed to 11.15).

11.15 Once tentative confirmation on the quantitation column is obtained, final confirmation is obtained by injecting 2 µL of the sample extract on the confirmatory column (For convenience, the sample may be analyzed simultaneously on both columns). Confirmation is accomplished by following the steps given:

11.15.1 Only the four known chlordane components listed in Table 1 (heptachlor, γ-chlordane, α-chlordane, and *trans*-nonachlor) can be rejected if not confirmed during the confirmation analysis. (Note that the peak corresponding to *trans*-nonachlor, which appears as a trailing shoulder to γ-chlordane on the primary column, is a separate peak on the confirmation column (see Fig. 2)). Conversely, since the identity of the other six primary column peaks on the confirmatory column cannot be positively established, the confirmation analysis cannot be used to reject these unknown components.

11.15.2 On the chlordane identification and quantitation worksheet, list the retention times (in minutes) and areas of the ten reference chlordane component peaks obtained from the confirmatory column analysis of the technical chlordane standard solution.

11.15.3 On the same worksheet, list the retention times and areas of the peaks corresponding to these ten peaks from the sample analysis using the DB-5 confirmation column.

11.15.4 Compare the retention times of peaks 3 (heptachlor), 6 (γ-chlordane), 7 (α-chlordane), and 8 (*trans*-nonachlor) in the confirmation analysis of the sample to the absolute retention times of the respective standard peaks, using a retention window of ±0.05 minutes around each standard peak. Draw a line through the sample retention times and areas for both the confirmation column and the primary quantitation column for any of these four components that are outside the retention window (for example, not present) on the confirmation column. However, when a consistent shift is evident in the retention times of many of the sample peaks, the analyst may expand the acceptable retention window in the direction of the shift. (Remember that the elution sequence of *trans*-nonachlor and α-chlordane are reversed on the OV-17 megabore and DB-5 columns).

11.15.5 If either α-chlordane or γ-chlordane (peaks 6 or 7 on the confirmatory column) is not present within the standard retention time window on the confirmation column, the sample is considered negative for chlordane.

11.15.6 If heptachlor (peak 3) or *trans*-nonachlor (peak 8) is outside its standard retention time window on the confirmation column, cross out its respective retention times and areas for both the confirmation and primary columns.

11.15.7 If α- and γ-chlordane are present on the confirmation column, and if criterion in 11.14.9 remains satisfied after considering the confirmation column, chlordane is confirmed in the sample. Proceed to the chlordane quantitation procedures in Section 12.

12. Calculations

12.1 *Determination of the Concentration of Technical Chlordane*:

12.1.1 Sum the areas of all matching GC peaks from the quantitation column analysis of the sample *except peak 3* (*heptachlor*). Likewise, determine the total areas of the corresponding peaks in the quantitation column analysis of the standard.

12.1.2 Calculate the amount of technical chlordane on the PUF plug using the following equation:

$$C_p = \frac{A_s}{A_p} \times C_s \times V_p \quad (1)$$

where:

C_p = quantity of technical chlordane on the PUF plug, µg,
A_p = total area of the appropriate GC peaks in the sample,
A_s = total area of the GC peaks in the standard,
C_s = concentration of standard, µg/mL, and
V_p = sample volume, mL.

NOTE 2—Injection volumes are excluded from the equation, since the same volume is injected for both the sample extract and standard solution.

12.1.3 If the area of one of the positive sample peaks differs markedly from the predicted value based on the standard (for example, ten times too large), the peak is excluded from the

quantitation. Since many considerations are involved, such exclusions are based on the judgment of the analyst.

12.1.4 Heptachlor should be quantified separately from technical chlordane and its concentration excluded from the determination of technical chlordane concentrations. The confirmatory megabore column should be used, since a commonly-occurring environmental contaminant may coelute with heptachlor on the quantitation column.

12.1.5 To determine whether the sample blank has been contaminated. The level of suspected contamination will exceed 0.10 µg/sample for technical chlordane and 0.01 µg/sample for heptachlor. If the blank has been contaminated, the field samples must be held suspect (See applicable paragraph Practice D 3686).

12.1.6 Correct sample concentrations for extraction recovery, sampling efficiency, and blank contamination according to the procedures given in Section 12 of Practice D 4861.

12.1.7 To determine air concentration, divide the quantity found on the PUF plug by the total volume of air pulled through the plug in accordance with 12.1.6 through 12.1.10 of Practice D 4861.

13. Sensitivity of the Procedure

13.1 Several different parameters involved in both the sampling and analysis steps of this method collectively determine the sensitivity with which chlordane or heptachlor are detected. As the volume of air sampled is increased, the sensitivity of detection increases proportionately within limits set by: (*a*) the retention efficiency for each component trapped on the polyurethane foam plug and (*b*) the background interference associated with the analysis of each component at a given site sampled. The sensitivity of detection of samples recovered by extraction depends on: (*a*) the inherent response of the particular GC/ECD used in the determinative step and (*b*) the extent to which the sample is concentrated for analysis. It is the responsibility of the analyst(s) performing the sampling and analysis steps to adjust parameters so that the required detection limits can be obtained.

14. Precision and Bias

14.1 Precision and bias in this type of analytical proce-dure are dependent upon the precision and bias of the analytical procedure for either chlordane or heptachlor, and the precision and bias of the sampling process.

14.2 Precision determined from collocated 24-h sampling has been demonstrated to be good. Results from 25 paired samples for which chlordane was detected in both samples or at least one sample in the applicable concentration range (see 1.3), showed a median difference of 13 %. Similar results from 23 paired sets of heptachlor samples gave a median difference of 18 %.

14.3 The accuracy of the method as determined by analysis of PUF plugs spiked with technical chlordane and six or seven other, potentially interfering, pesticides showed a bias range from + 6 % to − 16 % from the true values.

14.4 Sample recoveries for individual compounds generally fall within the range of 85 to 110 %, but recoveries ranging from 75 to 125 % are considered acceptable.

15. Keywords

15.1 chlordane; electron capture detection; gas chromatography; heptachlor; indoor air; polyurethane foam

ANNEX

(Mandatory Information)

A1. DETERMINATION OF SAMPLING EFFICIENCY AND FORTIFIED PUF RECOVERY

A1.1 Before using this procedure the user should determine the sampling efficiency for technical chlordane, its primary components and heptachlor. The sampling efficiencies shown in Table A1.1 were determined for approximately 0.25 m^3 of air sampled at 2.0 L/min at about 24°C. These sampling efficiencies were determined in accordance with the procedures found in the Annex of Practice D 4861.

TABLE A1.1 Sampling Efficiencies for Technical Chlordane, Heptachlor, and Chlordane/Heptachlor Combination[A]

Pesticide	Quantity introduced (µg)	Flow Rate L/min	Vol of Air (m^3)	Quantity Recovered (µg)	Sampling Efficiency[B] (%)
Technical Chlordane	7.10	2.0	0.257	6.58	92.7
	7.10	2.0	0.262	6.86	96.6
	7.10	2.0	0.286	6.09	85.7
	7.10	2.0	0.272	6.37	89.7
	7.10	2.0	0.252	6.03	84.9
Heptachlor	4.42	2.0	0.250	4.14	93.7
	4.42	2.0	0.259	3.77	85.4
	4.42	2.0	0.275	3.88	87.8
	4.42	2.0	0.271	3.40	77.0
	4.42	2.0	0.250	3.21	72.7
Chlordane/ Heptachlor[C]	3.55/1.10	2.0	0.318	3.33/1.02	93.9/91.8
	3.55/1.10	2.0	0.253	3.63/1.10	102.2/99.5
	3.55/1.10	2.0	0.261	3.05/0.92	85.9/84.3
	3.55/1.10	2.0	0.251	3.20/0.95	90.1/86.0
	3.55/1.10	2.0	0.340	2.91/0.78	81.9/70.6

[A] Sampling apparatus used was similar to that described in Annex A1 of Practice D 4861.
[B] Impinger rinsed with hexane and chromatographed to determine any remaining residue. If found, the amount was subtracted from total amount added to impinger prior to calculating amount found in PUF.
[C] One mL of hexane containing 3.55 µg analytical grade technical chlordane and 1.10 µg analytical grade heptachlor added to impinger.

APPENDIXES

(Nonmandatory Information)

X1. RECOVERY EFFICIENCY DATA FOR TECHNICAL CHLORDANE AND HEPTACHLOR FROM POLYURETHANE FOAM

X1.1 The data shown in Table X1.1 and Table X1.2 were determined as follows: Known amounts of analytical grade technical chlordane or heptachlor added to untreated PUF plug, allowed to sit for 1.0 h and extracted by analytical procedure. Using the same pipet, an equivalent amount(spike) added to 12.0 mL centrifuge tube and stored at − 20°C until samples analyzed. Recovery based on comparison between amount recovered from PUF plug and spiked tube.

TABLE X1.1 Recoveries of Known Amounts of Technical Chlordane from Polyurethane Foam (PUF) Plugs

Amount Added (µg)	Amount Recovered (µg)	Recovery (%)
0.05	0.048	96.0
0.1	0.085	85.3
0.1	0.099	98.7
0.1	0.066	65.8
0.1	0.087	86.5
0.2	0.189	94.6
0.2	0.196	98.0
0.2	0.185	92.3
0.2	0.198	99.0
0.3	0.262	87.5
0.3	0.256	85.4
0.4	0.333	83.2
0.5	0.431	86.1
0.5	0.481	96.1
1.0	1.223	122.3
1.0	0.880	88.0
1.0	0.894	89.4
1.0	0.902	90.2
1.0	0.871	87.1
1.0	1.232	123.2
1.0	0.771	77.1
2.0	1.728	86.4
2.0	1.794	89.7
2.0	1.762	88.1
2.5	2.253	90.1
3.0	2.493	83.1
3.0	2.820	94.1
4.0	3.484	87.1
5.0	3.670	73.4
6.0	5.130	85.5
10.0	8.861	88.6
10.0	10.723	107.2
15.0	13.410	89.4
20.0	16.602	83.0
25.0	21.100	84.4
25.0	18.331	73.3

TABLE X1.2 Recoveries of Known Amounts of Heptachlor from Polyurethane Foam (PUF) Plugs

Amount Added (µg)	Amount Recovered (µg)	Recovery (%)
0.02	0.018	90.0
0.02	0.018	88.5
0.02	0.018	88.5
0.02	0.019	94.0
0.03	0.027	91.1
0.04	0.034	85.9
0.04	0.036	89.0
0.05	0.038	75.8
0.05	0.046	92.9
0.05	0.033	65.5
0.10	0.095	95.0
0.10	0.076	75.9
0.10	0.085	85.0
0.10	0.081	81.4
0.15	0.145	96.6
0.20	0.177	88.3
0.20	0.187	93.3
0.20	0.180	89.8
0.20	0.195	97.3
0.25	0.261	104.4
0.30	0.274	91.2
0.30	0.284	94.6
0.40	0.350	87.6
0.50	0.565	113.0
0.50	0.467	93.3
0.80	0.836	104.5
0.80	0.707	88.4
1.00	0.956	95.6
1.00	0.862	86.2
2.50	2.234	89.4
3.20	2.881	90.0
4.00	3.256	81.4
8.00	6.887	86.1
10.00	9.314	93.1
12.00	10.441	87.0
16.00	14.383	89.9

X2. STORAGE STABILITIES OF TECHNICAL CHLORDANE AND HEPTACHLOR ON POLYURETHANE FOAM

X2.1 The data shown in Table X2.1 and Table X2.2 were determined by spiking PUF plugs and storing them at room temperature. (Note: Storage at 4°C or below is recommended.) The procedure used was as follows: Approximately 4.0 µg (1.0 mL) of analytical grade technical chlordane added to 12.0 mL tube, diluted to 10.0 mL, and chromatographed on day 0. Same pipet used to fortify untreated PUF plug contained in glass sampling cartridge, placed in foil, and stored for 30 days in the dark. Plugs extracted as described in Section 10.

TABLE X2.1 Storage Stability of Chlordane-Fortified PUF Cartridges Stored at Room Temperature (ca. 24°C) for 30 Days

Sample No.	Amount Added (µg)	Amount Found (µg)	Recovery (%)
1	3.70	3.44	93.0
2	4.44	4.12	92.8
3	4.37	4.43	101.4
4	4.56	4.02	88.1
5	4.53	4.22	93.2
6	4.39	4.06	92.5
7	4.45	3.90	87.6
8	3.96	3.69	93.3
9	3.85	3.62	94.1
10	3.91	3.76	96.2
11	3.82	3.58	93.8
12	4.24	3.90	91.9

TABLE X2.2 Storage Stability of Heptachlor-Fortified PUF Cartridges Stored at Room Temperature (ca. 24°C) for 30 Days

Sample No.	Amount Added (µg)	Amount Found (µg)	Recovery (%)
1	0.31	0.30	96.0
2	0.32	0.31	96.8
3	0.35	0.32	90.8
4	0.32	0.33	103.9
5	0.36	0.32	89.8
6	0.37	0.34	92.8
7	0.35	0.35	100.0
8	0.39	0.34	88.0
9	0.34	0.34	99.1
10	0.31	0.32	102.6
11	0.32	0.31	95.7
12	0.33	0.33	100.0

REFERENCES

(**1**) Sovocool, G., Lewis, R., Harless, R., Wilson, N., and Zehr, R., "Analysis of Technical Chlordane by Gas Chromatography/Mass Spectrometry," *Analytical Chemistry*, Vol 49, 1977, pp. 734–740.

(**2**) Lewis, R. and Macleod, K., "Portable Sampler for Pesticides and Semivolatile Industrial Organic Chemicals in Air," *Analytical Chemistry*, Vol 54, 1982, pp. 310–315.

(**3**) Schattenberg, H. and Camann, D., "A Systematic Procedure for Chlordane Identification in Air," *Proceedings of the 1988 EPA/APCA Symposium on Measurement of Toxic and Related Air Pollutants* , Raleigh, NC, May 1988, pp. 42–50.

(**4**) Leidy, R. B., Wright C. G., Dupree, H. E., Jr., and Sheets, T. J., "Subterranean Termite Control: Chlordane Residues in Soil Surrounding and Air Within Houses," in *Dermal Exposure Related to Pesticide Use*. ACS Symposium Series, No. 273, R. C. Honeycutt, G. Zweig and Nancy Ragsdale, Eds. 1985, pp. 265–277.

(**5**) *Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples*, U.S. Environmental Protection Agency Report No. EPA-600/8/80/038, Research Triangle Park, NC, June 1980 (NTIS No. PB82-208752).

(**6**) Atallah, Y. H., Whitacre, D. M., and Hoo, B. L. "Comparative Volatility of Liquid and Granular Formulations of Chlordane and Heptachlor from Soil," *Bulletin of Environmental Contamination and Toxicology*, Vol 22, 1979, pp. 570–574.