



Project Summary

Single-Laboratory Evaluation of Method 8080 — Organochlorine Pesticides and PCBs

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Method 8080 was developed for the determination of certain organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) in liquids and solids. Liquid samples are extracted according to Method 3510 (separatory funnel) or Method 3520 (continuous liquid-liquid extractor) and solid samples according to Method 3540 (Soxhlet extraction) or Method 3550 (sonication). The extracts are concentrated, fractionated on Florisil and the fractions analyzed by gas chromatography on packed columns.

EPA Method 8080, as published in the Second Edition of "Test Methods for Evaluating Solid Waste," Office of Solid Waste Manual SW-846, has been evaluated in a single-laboratory study. The Florisil cleanup procedure recommended in Method 8080 does not separate the OCPs from the PCBs. Consequently, the gas chromatographic analysis of the OCPs on the packed columns specified in the method may result in false identifications or in no identifications at all when PCBs are present. Toxaphene and chlordane pose special problems because of their multi-peak responses. Silica gel was therefore substituted for Florisil, and capillary columns for the packed columns. Furthermore, a sulfur cleanup procedure was incorporated in the method.

The Method 8080 protocol was revised accordingly and was evaluated with extracts of environmental samples spiked with the substances of interest at known concentrations. The precision and accuracy results indicate that the revised Method 8080 can be reliably

applied to the determination of OCPs and PCBs in liquid and solid matrices. The method detection limits for liquid matrices range from 0.02 to 0.09 $\mu\text{g}/\text{L}$ for the OCPs and from 0.5 to 0.9 $\mu\text{g}/\text{L}$ for PCBs. The method detection limits for solid matrices range from 1 to 6 $\mu\text{g}/\text{Kg}$ for the OCPs and from 60 to 70 $\mu\text{g}/\text{Kg}$ for PCBs.

The revised protocol is included in this report as an appendix. Also included as an appendix is an extensive literature review covering analytical methods for the determination of OCPs and PCBs in water, soil, sediment and sludge samples.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Las Vegas, NV, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The determination of organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) in environmental samples by gas chromatography (GC) with electron capture detection and by mass spectrometry has been recommended. Electron capture detection is preferred over mass spectrometry because the former is two to three orders of magnitude more sensitive than the latter. Since PCBs are extracted along with the OCPs and since they interfere with the determination of the OCPs whenever electron capture detectors are used, their presence in the extracts together with the OCPs

needs to be minimized. Therefore, several cleanup techniques based on Florisil, alumina, and silica gel chromatography have been developed.

EPA Method 8080, as published in the document "Test Methods for Evaluating Solid Waste," Office of Solid Waste Manual SW-846 (1), provides sample extract cleanup and GC conditions for the determination of the OCPs and the PCBs listed in Table 1 in a variety of environmental matrices including ground water, liquids, and solids. Following solvent extraction of liquid samples in a separatory funnel (Method 3510) or in a continuous liquid-liquid extractor (Method 3520) and of solid samples in a Soxhlet extractor (Method 3540) or with a sonicator (Method 3550), the extracts are cleaned up by Florisil chromatography. Elution of the compounds from the Florisil column is performed with 6, 15, and 50 percent ethyl ether in hexane. All compounds listed in Table 1 except six OCPs elute in Fraction I (6 percent ether in hexane). Of those six pesticides, four (dieldrin, endosulfan I, endrin, and endrin aldehyde) elute in Fraction II (15 percent ether in hexane), and two (endosulfan II and endosulfan sulfate) elute in Fraction III (50 percent ether in hexane). Endrin aldehyde was also reported in Fraction III. There is no mention in Method 8080 of

possible overlapping of compounds between fractions and of the reproducibility of the elution pattern.

Acurex, under contract to the EMSL-LV, conducted an evaluation and improvement study of Method 8080. In the first phase of this study, Method 8080, as written, was evaluated to: (a) determine the recoveries of the OCPs and PCBs listed in Table 1 in the absence of matrix interferences, (b) determine the extent of overlapping of compounds between fractions, and (c) determine the efficiency of the Florisil cleanup scheme with real samples. Also, the GC determination of the OCPs and PCBs using packed and capillary columns was evaluated, and a literature review of the analytical methodologies for the determination of the compounds listed in Method 8080 was conducted. Because of the complex nature of the PCB formulations, only Aroclor 1016 and Aroclor 1260 were used throughout this study.

In the second phase of this study, the focus was on developing a fractionation procedure to separate the PCBs from the bulk of the OCPs, and, at the same time, to remove interfering compounds coextracted with the OCPs and PCBs. Several extract cleanup procedures based on silica, alumina, silica gel/Celite, and Florisil/charcoal chromatography were

investigated. Furthermore, a capillary GC method was developed, and a method for sulfur removal was tested and incorporated. Upon completion of this phase, a revised protocol was prepared.

The analytical scheme given in the revised Method 8080 protocol employs silica gel fractionation (silica gel deactivated with 3.3 percent water). Three fractions are collected: Fraction I eluted with 80 mL hexane, Fraction II eluted with 50 mL hexane, and Fraction III eluted with 15 mL methylene chloride. The determination of the OCPs and PCBs which are recovered in these three fractions is performed by GC on fused silica capillary columns and with electron capture detection.

The revised protocol was evaluated in Phase III with extracts of environmental samples spiked with the substances of interest at known concentrations. The evaluation studies were conducted at three concentrations, each in triplicate. The precision and accuracy results indicated that the revised Method 8080 could be reliably applied to the determination of the OCPs and PCBs in liquid and solid matrices. A method detection limit determination was performed for both the aqueous and the solid matrices.

Experimental

Materials and Reagents

The materials and reagents were those specified in Method 8080, as applicable. All solvents and reagents used were pesticide grade or analytical grade. The two capillary GC columns used in the second and third phase of this study were a 30 m x 0.25 mm ID DB-5 fused-silica capillary column (J & W Scientific Inc., Folsom, California) with a 0.25- μ m film thickness, and a 30 m x 0.25 mm ID SPB-608 fused-silica capillary column (Supelco Inc., Bellefonte, Pennsylvania) with a 0.25- μ m film thickness. The DB-5 column was held for 2 min at 100°C, heated at 15°C/min from 100°C to 160°C, and finally heated at 5°C/min from 160°C to 270°C. The SPB-608 column was held for 2 min at 160°C, heated at 5°C/min from 160°C to 290°C and held 1 min at 290°C. The gas chromatograph was equipped with a constant current pulsed frequency electron capture detector and a data system. A Varian 8000 autosampler was used; the injection volume was 2 μ L.

Samples and Sample Extract Preparation

The aqueous samples used in this study

Table 1. Compounds Listed in EPA Method 8080

Parameter ^a	Storet No.	CAS No.
alpha-BHC	39337	319-84-6
beta-BHC	39338	319-85-7
gamma-BHC (Lindane)	39340	58-89-9
delta-BHC	34259	319-86-8
Heptachlor	39410	76-44-8
Aldrin	39330	309-00-2
Heptachlor epoxide	39420	1024-57-3
gamma-Chlordane	39350	57-74-9
Endosulfan I	34361	959-98-8
4,4'-DDE	39320	72-55-9
Dieldrin	39380	60-57-1
Endrin	39390	72-20-8
Endosulfan II	34356	33212-65-9
4,4'-DDD	39310	72-54-8
Endrin aldehyde	34366	7421-93-4
Endosulfan sulfate	34351	1031-07-8
4,4'-DDT	39300	50-29-3
4,4'-Methoxychlor	NA	72-43-5
Toxaphene	39400	8001-35-2
Aroclor 1016	34671	12674-11-2
Aroclor 1221	39488	1104-28-2
Aroclor 1232	39492	11141-16-5
Aroclor 1242	39496	53469-21-9
Aroclor 1248	39500	12672-29-6
Aroclor 1254	39504	11097-69-1
Aroclor 1260	39508	11096-82-5

NA — Storet number not available.

^a Kepone is included in Method 8080 in the second edition of SW-846 but is not included in Method 8080 in the third edition.

included distilled water, an aqueous waste (obtained from a pesticide waste storage facility) with a high concentration of organics (Liquid Waste 1), and an aqueous waste (obtained from a pesticide manufacturing plant) high in total dissolved solids (50 g/L) and various organic solvents (>9.5 percent). The solid matrices used were NBS SRM-1645 (River Sediment), a sandy loam soil from Soils Inc., Puyallup, Washington, with a total organics content of approximately 1300 mg/Kg, and a waste consisting of activated charcoal mixed with a polymeric material. Except for the evaluation of Method 3520 and 3540, the aqueous samples were extracted in a separatory funnel with methylene chloride, and the soil and sediment samples were extracted with hexane/acetone by sonication. The extract solvents were exchanged for hexane, and the extract concentrates were spiked with the OCPs and PCBs.

Sample Extract Cleanup

The Florisil cleanup was performed as specified in Method 8080. The silica gel cleanup procedure was performed according to Biddleman et al. (2), with slight modifications. Cleanup on alumina was similar to the procedure specified in the EPA Superfund Contract Laboratory Program (3), the Florisil/charcoal cleanup procedure was performed as described by Berg et al. (4), and the silical gel/Celite cleanup according to the procedure of Armour and Burke (5). The sulfur removal procedure evaluated and adopted was that described by Jensen et al. (6) with tetrabutylammonium sulfite as the active reagent.

Results and Discussion

Sample Extraction

A brief evaluation of the four extraction procedures recommended in Method 8080 showed, with the samples tested, the following results:

- The efficiencies of Methods 3510 (separatory funnel) and 3520 (continuous liquid/liquid extraction) for the extraction of aqueous samples were approximately equal. Method 3510 was then used for the extraction of all aqueous samples.
- Method 3550 (sonication) for solids showed a better precision than Method 3540 (Soxhlet extraction); the accuracies were similar for both methods. Method 3550 was then used for the extraction of all solid samples.

Florisil Fractionation

Florisil fractionation was performed as recommended in Method 8080. Separate experiments were run in duplicate for PCBs, toxaphene, technical chlordane, OCP group A (gamma-BHC, heptachlor, aldrin, heptachlor epoxide, endosulfan I, dieldrin, endosulfan II, 4,4'-DDT, and endrin aldehyde), and OCP group B (alpha-BHC, beta-BHC, delta-BHC, 4,4'-DDE, endrin, 4,4'-DDD, endosulfan sulfate, and 4,4'-methoxychlor). The experimental results are presented in Table 2.

The overall recoveries are quantitative, and the agreement between the duplicate experiments is in most cases excellent. However, discrepancies have been found between our data and the recovery data listed in Method 8080. But regardless of the reproducibility of the fractionation, it is apparent that the Florisil fractionation method is not suitable for samples that contain both OCPs and PCBs. PCBs appear in the same fraction as the bulk of the OCPs, but these two types of compounds need to be separated from each other to a larger extent to avoid cross-interference. To exemplify this on a real sample, we extracted a liquid waste and spiked the extract with known amounts of OCPs, Aroclor 1016 and Aroclor 1260. Because of the complexity of this sample matrix, we chose to analyze the fractions on a DB-5 capillary column; however, even then we were not able to find the spiking compounds. In view of these results, we

eliminated the Florisil fractionation scheme from further evaluation.

Silical Gel Fractionation

The silica gel fractionations were performed in triplicate at two concentration levels. Technical chlordane and toxaphene fractionations were performed separately. The distribution and percent recoveries of the OCPs, Aroclor 1016, Aroclor 1260, technical chlordane, and toxaphene are presented in Table 3. The distribution patterns of the OCPs and PCBs in the three silica gel fractions were quite reproducible. Compounds found to elute in Fraction I (80 mL hexane) include: heptachlor, aldrin, 4,4'-DDE, chlordane (partially), and the PCBs. Almost all the other OCPs elute in Fraction III. Total recoveries were greater than 70 percent, except for technical chlordane at concentration 1, with most values ranging from 80 to 110 percent.

The evaluation of the silica gel fractionation scheme with real sample extracts (e.g., liquid waste, NBS SRM-1645, sandy loam, etc.) gave comparable results. Only a few compounds (4,4'-DDD, alpha-BHC, gamma-chlordane) were split between fractions when the real samples were fractionated.

These results show that Fraction II and III may be combined before concentration and analysis when the sample matrices are relatively simple. However, when samples with complex matrices have to

Table 2. Results of the Florisil Fractionation Study

	Spike level (µg)	Recovery (%) ^a			
		Fraction I	Fraction II	Fraction III	Total
alpha-BHC	0.5	94; 92	9.7; 8.7		104; 101
beta-BHC	0.5	86; 83	10 ; 0		96; 83
gamma-BHC	0.5	76; 77	8.5; 8.1	1.5; <1.0	86; 85
delta-BHC	0.5	70; 60	31 ; 27		101; 87
Heptachlor	0.5	75; 78	5.3; 5.8		80; 84
Aldrin	0.5	78; 80	6.0; 6.6	2.8; 3.8	87; 90
Heptachlor epoxide	0.5	79; 80	15 ; 15		94; 95
Endosulfan I	0.5	59; 64	29 ; 30		88; 94
4,4'-DDT	1.0	95; 95	8.7; 6.7		104; 102
Dieldrin	1.0	20; 24	71 ; 70		91; 94
Endrin	1.0	37; 43	64 ; 51		101; 94
Endosulfan II	1.0		60 ; 79	30 ; 15	90; 94
4,4'-DDD	1.0	93; 90	11 ; 10		104; 100
Endrin aldehyde	1.0		80 ; 90	11 ; 4.4	91; 94
Endosulfan sulfate	1.0		23 ; 23	57 ; 51	80; 74
4,4'-DDE	1.0	97; 93	9.6; 8.6		107; 102
4,4'-Methoxychlor	5.0	87; 84	29 ; 26		116; 110
Aroclor 1016	10.0	86; 76	4.6; 4.4		91; 80
Aroclor 1260	10.0	91; 78	10 ; 9.0		101; 87
Technical chlordane	5.0	93; 94	9.6; 7.6		103; 102
Toxaphene	10.0	105; 108			105; 108

^a The number of determinations is two.

Table 3. Distribution and Percent Recoveries of Organochlorine Pesticides and PCBs in Silica Gel Column Fractions^{a,b,c,d,e}

Compound	Fraction I		Fraction II		Fraction III		Total recovery	
	Conc. 1	Conc. 2	Conc. 1	Conc. 2	Conc. 1	Conc. 2	Conc. 1	Conc. 2
alpha-BHC					82 (1.7)	74 (8.0)	82 (1.7)	74 (8.0)
beta-BHC					107 (2.1)	98 (12.5)	107 (2.1)	98 (12.5)
gamma-BHC					91 (3.6)	85 (10.7)	91 (3.6)	85 (10.7)
delta-BHC					92 (3.5)	83 (10.6)	92 (3.5)	83 (10.6)
Heptachlor	109 (4.1)	118 (8.7)					109 (4.1)	118 (8.7)
Aldrin	97 (5.6)	104 (1.6)					97 (5.6)	104 (1.6)
Heptachlor epoxide					95 (4.7)	88 (10.2)	95 (4.7)	88 (10.2)
Endosulfan I					95 (5.1)	87 (10.2)	95 (5.1)	87 (10.2)
4,4'-DDE	86 (5.4)	94 (2.8)					86 (5.4)	94 (2.8)
Dieldrin					96 (6.0)	87 (10.6)	96 (6.0)	87 (10.6)
Endrin					85 (10.5)	71 (12.3)	85 (10.5)	71 (12.3)
Endosulfan II					97 (4.4)	86 (10.4)	97 (4.4)	86 (10.4)
4,4'-DDD					102 (4.6)	92 (10.2)	102 (4.6)	92 (10.2)
Endrin aldehyde					81 (1.9)	76 (9.5)	81 (1.9)	76 (9.5)
Endosulfan sulfate					93 (4.9)	82 (9.2)	93 (4.9)	82 (9.2)
4,4'-DDT			86 (13.4)	73 (9.1)	15 (18.7)	8.7 (15.0)	101 (5.3)	82 (23.7)
4,4'-Methoxychlor					99 (9.9)	82 (10.7)	99 (9.9)	82 (10.7)
Aroclor 1016	86 (4.0)	87 (6.1)					86 (4.0)	87 (6.1)
Aroclor 1260	91 (4.1)	95 (5.0)					91 (4.1)	95 (5.0)
Technical chlordane	14 (5.5)	22 (5.3)	19 (6.8)	39 (3.6)	29 (5.0)	37 (5.1)	62 (3.3)	98 (1.9)
Toxaphene			15 (2.4)	17 (1.4)	73 (9.4)	84 (10.7)	88 (12.0)	101 (10.1)

^a Eluant composition: Fraction I — 80 mL hexane; Fraction II — 50 mL hexane; Fraction III — 15 mL methylene chloride.

^b Concentration 1 is 0.5 µg per column for BHCs, heptachlor, aldrin, heptachlor epoxide, endosulfan I; 1.0 µg per column for dieldrin, endosulfan II, 4,4'-DDT, endrin aldehyde, 4,4'-DDD, 4,4'-DDE, endrin, and endosulfan sulfate; 5 µg per column for 4,4'-methoxychlor and technical chlordane; 10 µg per column for toxaphene, Aroclor 1016, and Aroclor 1260.

^c For concentration 2 the amounts spiked are 10 times those of concentration 1.

^d The values listed represent the average recoveries from three determinations; the numbers in parentheses are the standard deviations. The recovery cut-off point is 5 percent.

^e Data obtained with standards, as indicated in footnotes b and c, dissolved in 2 mL hexane.

be extracted, especially matrices containing organic solvents, more crossover between fractions may occur. In such cases it is more advantageous to analyze the three fractions separately.

Fractionation on Alumina, Florisil/Charcoal, and Silica Gel/Cellite

It was found in a series of experiments that none of these procedures was superior to the relatively simple silica gel fractionation.

Sulfur Removal

Elemental sulfur, which may be present in extracts from sediments and from some industrial samples, gives GC peaks which mask the region of aldrin, BHCs, heptachlor and heptachlor epoxide when the analysis is performed on the 1.5 percent OV-17/1.95 percent OV-210 on Chromosorb-WHP column or on the 30 m DB-5 fused-silica capillary column. The procedure of Jensen et al. (6) was used on five sample extracts fortified with the OCPs and PCBs to determine if removal of sulfur is affected by matrix interferences and if the OCP and PCB recoveries are acceptable (>80 percent) when this method is used. In addition to the real

sample extracts, three pesticide standards were reacted with the tetrabutylammonium sulfite reagent to determine compound recovery in the absence of matrix interferences. The results showed that sulfur was removed quantitatively, regardless of the matrix, and that the recoveries were acceptable except for the endrin aldehyde recovery which was only about 10 percent. This procedure was therefore incorporated into the revised method protocol.

Capillary Gas Chromatography

The gas chromatographic retention times of 18 OCPs on the DB-5 and the SPD-608 fused-silica capillary columns are presented in Table 4. Toxaphene is not included because of its multiplex response. Aroclor mixtures have been analyzed individually on the DB-5 column; the retention times of the individual chlorinated biphenyls in these mixtures are included in the full report.

Those OCPs that elute on the DB-5 column at the same retention times as some of the components of the Aroclor mixtures are identified in Table 4. Of the six OCP peaks that overlap with PCB component peaks, only heptachlor and gamma-chlordane are of concern because

the other four compounds are separated from the PCBs during the silica gel chromatography step. The heptachlor peak overlaps with a PCB peak eluting at 15.93 (present in six of the seven PCB mixtures), and the gamma-chlordane peak overlaps with a PCB peak eluting at 19.5 minutes (present in four of the seven PCB mixtures).

Method Performance

Method performance, as used here includes the method precision and accuracy and the method detection limit. To determine method precision and accuracy clean hexane and extracts of environmental samples (Liquid Waste 1, NBS River Sediment SRM-1645, and sandy loam soil) were spiked with the OCPs listed in Table 1 (except toxaphene), with Aroclor 1016 and with Aroclor 1260 at three concentrations (0.25 ng/µL, 0.5 ng/µL, and 2.5 ng/µL for the OCPs and 2.5 ng/µL, 5.0 ng/µL and 25 ng/µL for PCBs) and were processed through the method. The average recoveries of triplicate determinations (method accuracy and the relative standard deviations; (method precision) are presented in Table 5.

Of all recovery determinations in Table

5, 77 percent fall within the range 85 to 132 percent. An additional 5.2 percent are below 70 percent, and 18 percent are between 71 and 84 percent. Because of interferants, delta BHC, endosulfan II, and 4,4'-DDD could not be determined in the liquid waste when spiked at concentrations 1 and 2, endrin aldehyde

could not be determined in the liquid waste at any of the three spike levels, and 4,4'-methoxychlor could not be determined in the NBS SRM-1645 when spiked at concentration 1.

Of all relative standard deviations in Table 5, 58 percent fall below 10 percent, 36 percent are between 11 percent and

20 percent, and only 6 percent are above 21 percent.

There seem to be no patterns between the recovery and the concentration of the OCPs and PCBs or the matrix. When the percent recovery of each compound was plotted as a function of matrix for each of the three concentrations, no trend could be found.

The method detection limits (MDL) were determined for both distilled water and sandy loam soil from the standard deviations (SD) of 7 replicate measurements (MDL equals 3 times the SD); they represent the minimum concentrations that can be measured and reported with 99 percent confidence. The MDLs for water samples ranged from 0.02 to 0.09 µg/L for OCPs and from 0.5 to 0.9 µg/L for PCBs, and those for soil samples ranged from 1 to 6 µg/Kg for OCPs and from 60 to 70 µg/Kg for PCBs. It should be kept in mind that these values are representative of clean sample matrices. For complex matrices, the MDLs may be higher.

Conclusions

A revision of Method 8080 for the determination of the organochlorine pesticides and PCBs was necessary because the Florisil procedure does not allow separation of the organochlorine pesticides from the multi-component PCB mixture (except for endosulfans and endrin aldehyde) and because the packed

Table 4. Summary of Retention Times for the Organochlorine Pesticides^a

Compound No.	Compound Name	Retention time (min)	
		DB-5 ^b	SPB-608 ^c
1	alpha-BHC	12.29 ± 0.010	9.46
2	beta-BHC	13.13 ± 0.009	11.33
3	gamma-BHC	13.37 ± 0.011	10.97
4	delta-BHC	14.14 ± 0.011	12.73
5	Heptachlor ^d	15.91 ± 0.008	12.46
6	Aldrin	17.16 ± 0.009	13.76
7	Heptachlor epoxide	18.60 ± 0.009	15.98
8	gamma-Chlordane ^d	19.48 ± 0.012	16.70
9	Endosulfan I ^d	19.94 ± 0.010	17.40
10	4,4'-DDE	20.83 ± 0.008	18.36
11	Dieldrin ^d	20.91 ± 0.008	18.60
12	Endrin	21.71 ± 0.007	19.96
13	Endosulfan II ^d	22.05 ± 0.006	20.69
14	4,4'-DDD	22.38 ± 0.008	20.53
15	Endrin aldehyde ^d	22.75 ± 0.007	21.90
16	Endosulfan sulfate	23.64 ± 0.008	22.54
17	4,4'-DDT	23.79 ± 0.008	21.72
18	4,4'-Methoxychlor	25.94 ± 0.007	24.90

^a Toxaphene and PCBs are not included because of their multippeak response.

^b The values given for the DB-5 column are average retention time ± standard deviation of 10 replicate determinations.

^c Single determinations.

^d Coeluting with Aroclor components on the DB-5 column.

Table 5. Recoveries (in Percent) of the Method 8080 Compounds from Spike Extracts of Environmental Samples.

Compound	Hexane ^a			Liquid waste extract ^a		
	Conc. 1	Conc. 2	Conc. 3	Conc. 1	Conc. 2	Conc. 3
alpha-BHC	83 ± 16 (19)	106 ± 6.8 (6.4)	91 ± 4.6 (5.1)	96 ± 7.0 (7.3)	97 ± 3.5 (3.6)	79 ± 10 (13)
beta-BHC	86 ± 15 (17)	110 ± 10 (9.5)	98 ± 2.0 (2.1)	92 ± 10 (11)	100 ± 4.0 (4.0)	90 ± 3.1 (3.4)
gamma-BHC	85 ± 15 (18)	108 ± 11 (10)	99 ± 2.3 (2.3)	91 ± 10 (11)	100 ± 5.5 (5.5)	90 ± 4.0 (4.4)
delta-BHC	87 ± 12 (14)	109 ± 12 (11)	97 ± 1.6 (1.6)	^b	^b	90 ± 11 (8.8)
Heptachlor	79 ± 11 (13)	94 ± 9.5 (10)	83 ± 6.6 (7.9)	89 ± 14 (15)	94 ± 10 (11)	90 ± 11 (12)
Aldrin	88 ± 11 (13)	107 ± 9.5 (8.9)	89 ± 4.2 (4.7)	96 ± 8.9 (9.3)	98 ± 9.4 (9.6)	92 ± 9.2 (10)
Heptachlor epoxide	94 ± 6.7 (7.1)	109 ± 14 (13)	100 ± 23 (23)	111 ± 17 (15)	109 ± 14 (13)	89 ± 4.1 (4.6)
gamma-Chlordane	94 ± 13 (14)	110 ± 11 (10)	91 ± 1.2 (1.3)	100 ± 8.5 (8.5)	103 ± 2.5 (2.4)	95 ± 8.0 (8.4)
Endosulfan I	89 ± 12 (14)	108 ± 13 (12)	99 ± 2.3 (2.3)	95 ± 6.5 (6.8)	100 ± 12 (12)	88 ± 3.8 (4.3)
4,4'-DDE	92 ± 13 (14)	107 ± 15 (14)	89 ± 4.5 (5.0)	119 ± 11 (8.9)	113 ± 2.5 (2.2)	95 ± 16 (17)
Dieldrin	89 ± 12 (14)	112 ± 13 (12)	102 ± 1.5 (1.5)	88 ± 3.2 (3.6)	86 ± 9.2 (11)	82 ± 4.3 (5.3)
Endrin	66 ± 11 (17)	65 ± 10 (16)	64 ± 8.3 (13)	101 ± 5.9 (5.8)	90 ± 10 (11)	65 ± 3.1 (4.7)
Endosulfan II	86 ± 7.9 (9.2)	111 ± 14 (13)	101 ± 0.6 (0.6)	^b	^b	79 ± 7.1 (9.0)
4,4'-DDD	89 ± 12 (14)	110 ± 9.8 (8.9)	97 ± 1.7 (1.8)	^b	^b	76 ± 16 (21)
Endrin aldehyde	83 ± 8.3 (10)	102 ± 19 (19)	95 ± 3.0 (3.2)	^b	^b	^b
Endosulfan sulfate	91 ± 5.2 (5.7)	112 ± 21 (19)	104 ± 2.5 (2.4)	132 ± 17 (13)	127 ± 22 (17)	83 ± 4.0 (4.8)
4,4'-DDT	74 ± 19 (26)	88 ± 13 (15)	73 ± 5.8 (8.0)	101 ± 23 (23)	83 ± 11 (13)	88 ± 18 (21)
4,4'-Methoxychlor	98 ± 2.6 (2.7)	104 ± 18 (17)	104 ± 3.2 (3.1)	49 ± 14 (29)	58 ± 9.3 (16)	75 ± 4.6 (6.1)
Aroclor 1016	94 ± 14 (15)	93 ± 6.5 (7.0)	93 ± 2.0 (2.2)	114 ± 6.0 (5.3)	122 ± 10 (8.3)	118 ± 9.8 (8.3)
Aroclor 1260	92 ± 12 (13)	87 ± 15 (17)	78 ± 4.0 (5.1)	99 ± 4.6 (4.6)	102 ± 4.7 (4.6)	100 ± 18 (18)
Concentration (ng/µL extract)	0.25	0.5	2.5	0.25	0.5	2.5
Number of determinations	3	3	3	3	3	3

^a Percent recovery ± standard deviation (triplicate determinations).

^b Unable to determine recovery because of interference.

Table 5. (Continued)

Compound	NBS SRM-1645 extract ^a			Sandy loam soil extract ^a		
	Conc. 1	Conc. 2	Conc. 3	Conc. 1	Conc. 2	Conc. 3
alpha-BHC	73 ± 2.1 (2.9)	75 ± 6.0 (8.0)	76 ± 5.6 (7.3)	86 ± 9.5 (11)	87 ± 4.9 (5.7)	89 ± 2.5 (2.8)
beta-BHC	88 ± 4.1 (4.7)	94 ± 3.0 (3.2)	92 ± 7.1 (7.7)	94 ± 8.4 (8.9)	90 ± 4.1 (4.5)	93 ± 5.0 (5.4)
gamma-BHC	83 ± 3.0 (3.6)	89 ± 4.1 (4.6)	93 ± 8.1 (8.7)	92 ± 11 (12)	91 ± 3.0 (3.3)	95 ± 4.6 (4.8)
delta-BHC	85 ± 4.6 (5.4)	92 ± 5.2 (5.6)	94 ± 8.7 (9.3)	94 ± 12 (13)	89 ± 3.6 (4.1)	93 ± 6.1 (6.5)
Heptachlor	53 ± 10 (19)	70 ± 7.7 (11)	88 ± 4.1 (4.7)	89 ± 9.6 (11)	83 ± 8.7 (11)	79 ± 17 (21)
Aldrin	69 ± 3.6 (5.2)	65 ± 4.6 (7.1)	72 ± 1.0 (1.4)	99 ± 4.4 (4.4)	88 ± 2.0 (2.3)	82 ± 12 (15)
Heptachlor epoxide	91 ± 4.9 (5.4)	91 ± 5.7 (6.3)	93 ± 8.6 (9.2)	96 ± 11 (11)	90 ± 4.6 (5.1)	94 ± 6.6 (7.0)
gamma-Chlordane	77 ± 5.3 (6.9)	81 ± 4.9 (6.1)	85 ± 1.0 (1.2)	100 ± 8.3 (8.3)	93 ± 3.0 (3.2)	87 ± 11 (13)
Endosulfan I	85 ± 5.5 (6.5)	88 ± 5.1 (5.8)	91 ± 9.1 (10)	95 ± 10 (11)	89 ± 5.3 (5.9)	93 ± 7.5 (8.1)
4,4'-DDE	75 ± 5.3 (7.1)	76 ± 7.1 (9.3)	84 ± 1.0 (1.2)	105 ± 14 (13)	93 ± 5.6 (6.0)	82 ± 11 (13)
Dieldrin	92 ± 8.6 (9.4)	85 ± 9.4 (11)	94 ± 10 (11)	113 ± 12 (11)	99 ± 4.6 (4.6)	93 ± 8.2 (8.8)
Endrin	100 ± 9.5 (9.5)	87 ± 6.4 (7.3)	76 ± 9.9 (13)	74 ± 5.6 (7.5)	60 ± 7.6 (13)	47 ± 11 (24)
Endosulfan II	80 ± 7.4 (9.2)	81 ± 4.5 (5.5)	91 ± 12 (13)	97 ± 14 (14)	86 ± 6.7 (7.8)	89 ± 11 (12)
4,4'-DDD	106 ± 6.4 (6.0)	85 ± 3.1 (3.6)	90 ± 7.2 (8.0)	103 ± 9.6 (9.3)	88 ± 7.8 (8.9)	90 ± 7.8 (8.6)
Endrin aldehyde	70 ± 5.7 (8.2)	71 ± 9.2 (13)	88 ± 12 (14)	86 ± 11 (13)	82 ± 7.6 (9.3)	73 ± 20 (28)
Endosulfan sulfate	75 ± 8.7 (12)	86 ± 5.0 (5.8)	72 ± 11 (15)	112 ± 19 (17)	91 ± 13 (14)	88 ± 11 (13)
4,4'-DDT	54 ± 13 (24)	61 ± 7.9 (13)	76 ± 2.5 (3.3)	107 ± 25 (23)	83 ± 5.3 (6.5)	64 ± 14 (21)
4,4'-Methoxychlor	^b	99 ± 17 (17)	92 ± 17 (19)	91 ± 14 (15)	89 ± 9.6 (11)	86 ± 14 (16)
Aroclor 1016	104 ± 9.0 (8.7)	104 ± 2.5 (2.4)	102 ± 4.6 (4.5)	90 ± 15 (17)	92 ± 4.5 (4.9)	85 ± 10 (12)
Aroclor 1260	92 ± 9.5 (10)	95 ± 7.5 (7.9)	91 ± 4.0 (4.4)	99 ± 6.8 (6.9)	89 ± 8.9 (10)	85 ± 13 (15)
Concentration (ng/μL extract)	0.25	0.5	2.5	0.25	0.5	2.5
Number of determinations	3	3	3	3	3	3

^a Percent recovery ± standard deviation (triplicate determinations).

^b Unable to determine recovery because of interference.

columns do not have enough resolving power to handle complex environmental samples.

The revised Method 8080 presented in Appendix B has been evaluated in a single laboratory with some relevant liquid and solid wastes. The utilization of silica gel fractionation and capillary column analysis was found to be appropriate. When silica gel fractionation was used, three fractions were collected. The silica gel procedure is tedious and does account for a major part of the analysis time. However, we have demonstrated that the method precision is better than ±20 percent for all compounds, and the accuracy is greater than 60 percent when standards are processed through the silica gel procedure. Fraction III may be com-

bined with Fraction II when the matrix is not very complex, and thus the number of analyses per sample may be reduced. The use of a second capillary column as a confirmatory column is recommended.

Toxaphene, if present in the sample at concentrations 10 times as high as the OCPs, is likely to cause problems in the determination of the OCPs and PCBs since it does not elute from the silica gel column in a narrow band. Other analytical techniques (e.g., chemical ionization mass spectrometry) should be considered for the determination of toxaphene.

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The complete report, entitled "Single-Laboratory Evaluation of Method 8080—Organochlorine Pesticides and PCBs," (Order No. PB 87-232 591/AS; Cost: \$24.95, subject to change) will be available only from:

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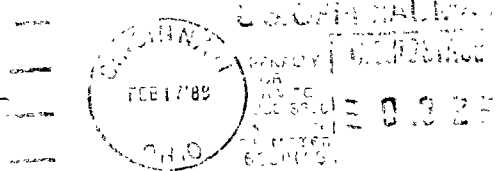
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