RESIDUES AND TRACE ELEMENTS

Analysis of a Mixture of Polychlorinated Biphenyls and Chlorinated Pesticides in Human Serum by Column Fractionation and Dual-Column Capillary Gas Chromatography with Electron Capture Detection

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An analytical method is presented for precise identification and quantitation of 29 specific polychlorinated biphenyl (PCB) congeners and 15 chlorinated pesticides in human serum. Analyte surrogates PCB 30, PCB 204, 2,2′,4,4′,5,5′-hexabromo-biphenyl, perthane, α-hexachlorocyclohexane, and dichlorobenzophenone were added to each sample. The serum was extracted with an organic solvent and separated by adsorption chromatography into 3 elution fractions for high-resolution gas chromatographic analysis. Each fraction was analyzed by dual-column capillary chromatography followed by electron capture detection. Two capillary columns, DB-5 and DB-1701, with different polarities were used to increase selectivity for each analyte. Quantitation was performed by selecting 2 sets of calibration standard mixtures and 1,2-dichloronaphthalene as an internal standard. Mean recoveries ranged from 39 to 126% for selected analytes and from 31 to 88% for surrogates. Detection limits for specific congeners and pesticides are reported. Typical chromatographic profiles of calibration standard mixtures, as well as a human sample, are illustrated. Verification of each analyte is assessed, and results of analyses of selected human samples and quality control criteria used to ensure data validity also are presented.

Polychlorinated biphenyls (PCBs) and persistent pesticides were widely used in various applications. Although their use has subsided, they still are found in many sample matrixes: blood, milk, meat, fish, water, crops, and soil. Correct identification and quantitation of a blend of PCBs, pesticides, and their metabolites always pose a great challenge to an analytical chemist. Current epidemiologic studies require qualitative and quantitative data on specific analytes so that their presence can be linked to certain toxic health effects.

Commercial PCB products (Aroclor 1016, 1242, 1254, 1260, 1268, etc.) have different levels of chlorination and isomer compositions. Similarly, many commercial preparations contain a variety of pesticide mixtures. However, the composition of analytes in biological specimens differs from that in commercial products because of metabolism, selective absorption, or excretion.

There is no single gas chromatographic (GC) column available that can completely resolve a mixture of chlorinated pesticides (CPs) and all 209 PCB congeners. This paper describes use of adsorption chromatography to extract analytes of interest into 3 elution fractions, dual capillary columns, and pure standards for identification and quantitation of analytes in human serum. It also presents surrogates used to monitor recovery of analytes in each fraction, as well as analyses of selected human serum samples by the method.

Experimental

Reagents and Standards

All solvents were nanograde quality and, except for isooctane, were purchased from Burdick and Jackson, Inc. (Muskegon, MI). Isooctane was obtained from Mallinckrodt Chemical, Inc. (Paris, KY). Analytical standards were prepared from neat materials in nanograde isooctane. 1,2-Dichloronaphthalene (DCN) was used as an internal standard (IS) and was prepared in isooctane. DCN and PCB congeners IUPAC codes [Ballschmiter and Zell (BZ; 1) numbers are given in parentheses if they differ from IUPAC codes] 28, 30, 52, 60, 66, 74, 99, 101, 118, 138, 153, 170, 180, 183, 195, and 204 were purchased from Ultra Scientific (North Kingston, RI). PCBs 56, 146, 172, 178, 189, 193, 199 (201), 203, and 2,2′,4,4′,5,5′-hexabromobiphenyl (HxBrB) were obtained from AccuStandard (New Haven, CT). The following PCBs were obtained from Cambridge Isotope Labs (Woburn, MA): PCB 105, 110, 156, 177, 187, 194, and 206. Pesticide standards (99+% pure) were obtained from the U.S. Environmental Protection Agency Pesticides Repository (Research Triangle Park, NC, or Perrine, FL). The purity and the integrity of each standard were verified in-

Received April 28, 1998. Accepted by JS September 1, 1998.

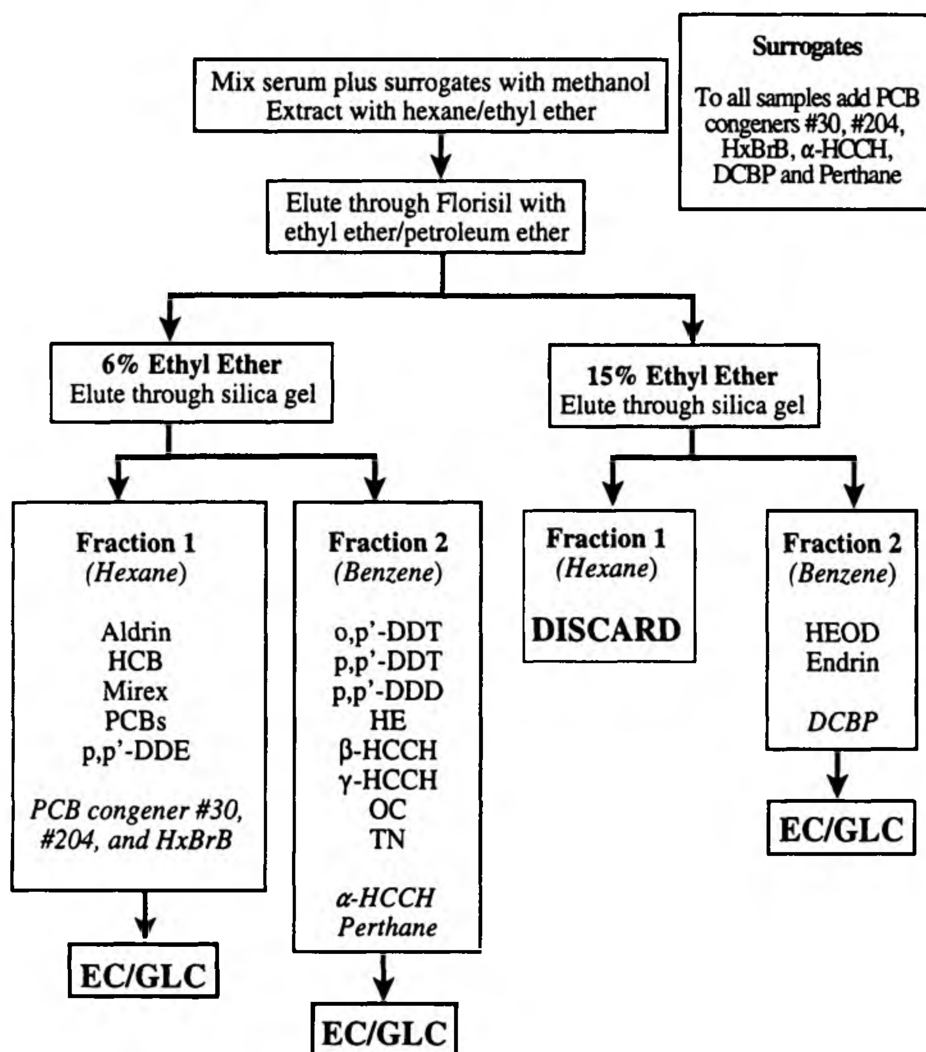

Figure 1. Extraction scheme and analytes eluted in each fraction.

dividually by analyzing on 2 capillary columns of different polarities and comparing retention times (RTs) against published chromatograms (1–4). A "keeper" solution (used to reduce or minimize analyte loss during evaporation of solvents) was made by mixing 1 g paraffin oil (Fisher Scientific, Pittsburgh, PA) in 100 mL hexane.

PCB congeners and pesticides for use as standards were selected on the basis of their detection in human serum as reported in various health studies (5–8). Four levels of calibration standard mixes ranging from 0.25 to 7 ppb for PCBs (standard mix 1) and 4 levels of calibration standard mixes ranging from 0.25 to 10 ppb for pesticides (standard mix 2) were prepared in nanograde isooctane.

Base bovine serum (BBS) to monitor the matrix blank was processed at the Centers for Disease Control and Prevention, National Center for Environmental Health Laboratory, Atlanta, Georgia, and was found to be essentially free of pesticides and PCBs. Bench quality control (QC) samples, also known as goat serum reference pools (SRPs), were prepared by feeding goats technical Aroclor 1016, 1242, 1254, or 1260, allowing the goats to recover for 30 days, exsanguinating them, and obtaining serum for characterization of individual PCB congeners (9). Chlorinated hydrocarbon spike (CHS) consisted of BBS spiked in vitro with a series of persistent chlorinated pesticides at 1.0 ppb (except for *p,p′*-DDT, which was spiked at 2.0 ppb, and *p,p′*-DDE, which was spiked at 10.0 ppb).

Analytical Procedure

A 2 mL portion of sample serum or QC control sample was dispensed in a clean glass test tube and spiked with a surrogate standard containing PCB congeners 30 and 204, HxBrB, α-hexachlorocyclohexane (α-HCCH), dichlorobenzophenone (DCBP), and Perthane. They were mixed well and left overnight in a refrigerator to equilibrate. These surrogates were selected because they normally are not found in human samples, they are recovered in various extraction steps, and their elution

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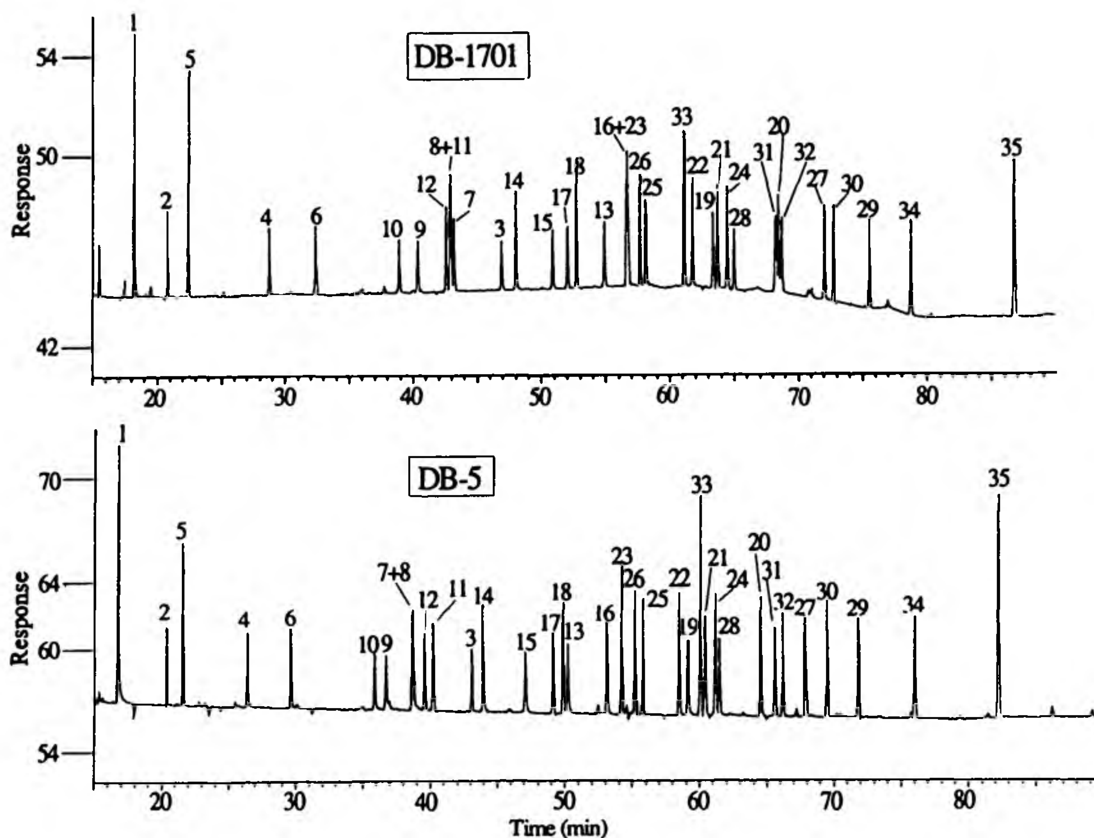

Figure 2. Gas chromatograms of calibration standard mix 1.

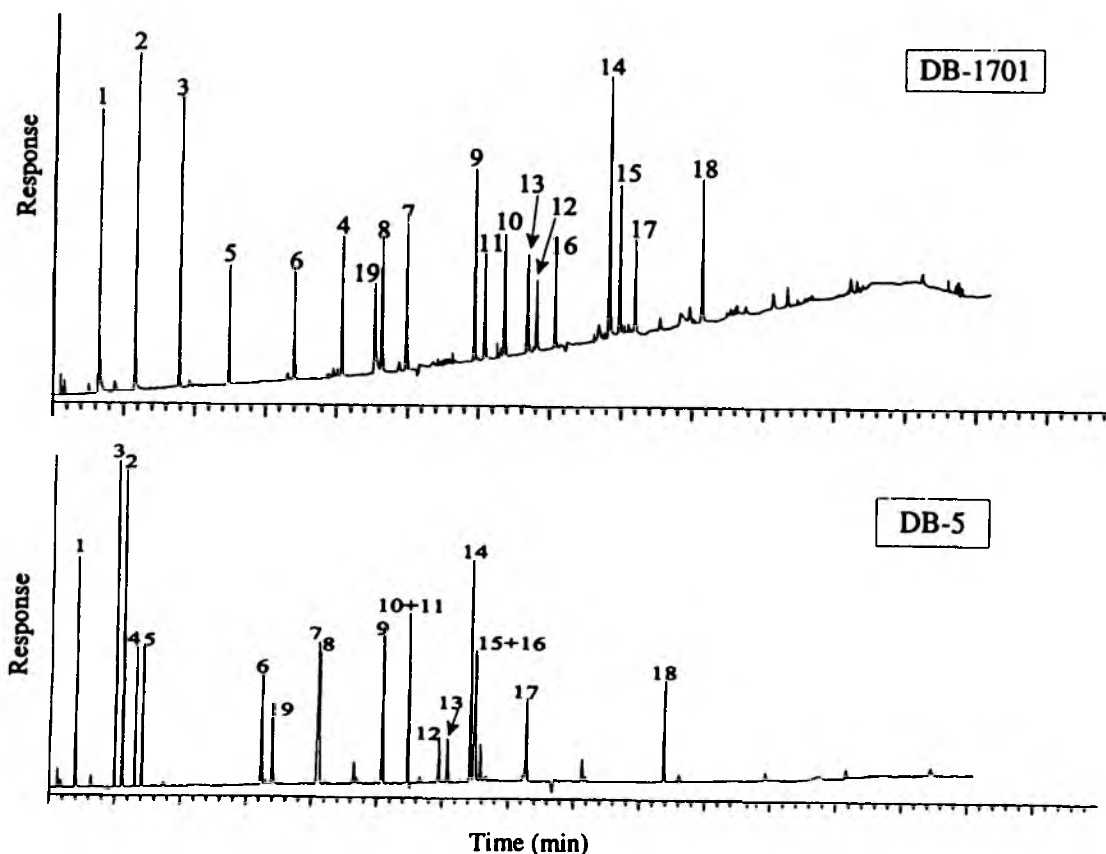

Figure 3. Gas chromatograms of calibration standard mix 2.

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Table 1. Relative retention times of components of calibration standard mix 1[a]

		Retention times relative to DCN with indicated column	
Analyte No.	Analyte identity	DB-5	DB-1701
1	1,2-DCN (IS)	1.000	1.000
2	HCB	1.222	1.148
3	*p,p'*-DDE	2.581	2.568
4	PCB-28	1.585	1.581
5	PCB-30(S)	1.297	1.237
6	PCB-52	1.782	1.780
7	PCB-56	2.324	2.364
8	PCB-60	2.324	2.349
9	PCB-66	2.207	2.212
10	PCB-74	2.157	2.135
11	PCB-99	2.412	2.349
12	PCB-101	2.375	2.332
13	PCB-105	2.999	3.001
14	PCB-110	2.631	2.628
15	PCB-118	2.816	2.783
16	PCB-138	3.169	3.098
17	PCB-146	2.937	2.845
18	PCB-153	2.979	2.883
19	PCB-156	3.522	3.461
20	PCB-170	3.836	3.738
21	PCB-172	3.597	3.479
22	PCB-177	3.485	3.374
23	PCB-178	3.232	3.098
24	PCB-180	3.641	3.521
25	PCB-183	3.324	3.179
26	PCB-187	3.290	3.154
27	PCB-189	4.030	3.934
28	PCB-193	3.659	3.549
29	PCB-194	4.264	4.121
30	PCB-195	4.129	3.971
31	PCB-199 (201)	3.899	3.727
32	PCB-203	3.933	3.753
33	PCB-204 (S)	3.577	3.341
34	PCB-206	4.508	4.295
35	HxBrB (S)	4.878	4.737

[a] The PCBs are numbered according to IUPAC codes; BZ codes are in parentheses. S, surrogate; IS, internal standard.

Table 2. Relative retention times of components of calibration standard mix 2[a]

		Retention times relative to DCN with indicated column	
Analyte No.	Analyte identity	DB-5	DB-1701
1	1,2-DCN (IS)	1.000	1.000
2	HCB	1.223	1.147
3	α-HCCH (S)	1.189	1.327
4	β-HCCH	1.283	1.989
5	γ-HCCH	1.316	1.529
6	Aldrin	1.883	1.795
7	Heptachlor epoxide	2.149	2.249
8	Oxychlordane	2.158	2.151
9	*trans*-Nonachlor	2.457	2.528
10	Dieldrin	2.580	2.660
11	*p,p'*-DDE	2.580	2.569
12	Endrin	2.727	2.791
13	Perthane (S)	2.767	2.753
14	*p,p'*-DDD	2.875	3.085
15	*cis*-Nonachlor	2.895	3.127
16	*o,p'*-DDT	2.895	2.866
17	*p,p'*-DDT	3.140	3.189
18	Mirex	3.791	3.462
19	DCBP (S)	1.935	2.121

[a] S, surrogate; IS, internal standard.

order covers a wide area of the chromatogram. The following samples were added to check the integrity and performance of an analytical run: a reagent blank (RB) or extraction blank to monitor background levels of solvents and reagents, BBS, and bench control samples (SRPs and CHS). Serum was extracted through partition and selective adsorption chromatography according to the procedure described by Burse et al. (10). Briefly, PCBs and pesticides were separated by mixing surrogate-spiked serum with methanol and extracting with hexane–ethyl ether (1 + 1). The extract was passed through a prewashed Florisil column. Trapped analytes were eluted with 15 mL 6% ethyl ether–petroleum ether followed by 15 mL 15% ethyl ether–petroleum ether. Eluates were treated as follows: (*1*) Eluates from 6% ethyl ether–petroleum ether elution were passed through a 4.8% deactivated silica gel column. This silica gel column was eluted initially with hexane to collect PCBs, aldrin, hexachlorobenzene (HCB), mirex, and *p,p'*-DDE (6% fraction 1) and then with benzene to collect other pesticides (6% fraction 2). (*2*) Eluates from the 15% ethyl ether–petroleum ether elution were passed through another 4.8% deactivated silica gel column and eluted first with hexane (15% fraction 1) and then with benzene (15% fraction 2). Fraction 1 was discarded, and fraction 2 was analyzed for the presence of dieldrin and endrin.

This cleanup yields 3 fractions for instrumental analysis. The extraction scheme and analytes eluted in each fraction are outlined in Figure 1. Five drops keeper solution were added to each final eluate, and the sample was evaporated just to dryness in a water bath under a slow stream of nitrogen. The residue from each fraction was reconstituted in 1 mL isooctane containing 12.5 ppb 1,2-DCN as IS. A portion of the reconstituted fraction was transferred to a conical glass sample vial and analyzed on 2 different capillary GC instruments, each equipped with an electron capture detector (ECD).

Instrumentation

Analysis was performed on 60 m × 0.25 mm id × 0.25 μm film thickness fused silica DB-5 and DB-1701 (J&W Scientific, Folsom, CA) capillary columns, each installed in an HP-5890 Series II (Hewlett-Packard, Wilmington, DE) GC equipped with an ECD, HP7673 autosampler, and split/split-

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Table 3. Recovery and LOD of PCB congeners ($n = 7$)

Analyte	Spike level, ng/mL	Average amount detected, ng/mL	Average recovery ± standard deviation, %	LOD, ng/mL
PCB-28	1.03	0.79	76.39 ± 14.26	0.45
PCB-52	0.86	0.61	71.14 ± 12.56	0.33
PCB-56[a]	0.92	0.73	79.77 ± 15.33	0.42
PCB-66	0.93	0.78	83.51 ± 16.20	0.45
PCB-74	1.07	0.85	79.69 ± 14.53	0.45
PCB-99[b]	1.17	0.84	71.89 ± 12.25	0.42
PCB-101	1.03	0.73	71.04 ± 11.41	0.36
PCB-105	0.93	0.77	81.97 ± 15.45	0.42
PCB-110	1.15	0.92	79.68 ± 15.10	0.51
PCB-118	1.00	0.80	80.13 ± 14.35	0.42
PCB-138[b]	0.91	0.70	77.22 ± 13.20	0.36
PCB-146	0.83	0.51	61.50 ± 11.26	0.27
PCB-153	1.32	0.83	62.66 ± 10.42	0.42
PCB-156	0.83	0.65	77.85 ± 14.25	0.36
PCB-170	1.17	0.93	79.82 ± 14.87	0.51
PCB-172	1.04	0.63	60.72 ± 10.25	0.33
PCB-177	0.95	0.76	80.20 ± 14.41	0.42
PCB-178[b]	1.67	0.94	56.25 ± 9.77	0.48
PCB-180	0.96	0.63	65.48 ± 10.65	0.30
PCB-183	1.18	0.65	55.43 ± 9.03	0.33
PCB-187	1.11	0.67	60.63 ± 9.70	0.33
PCB-189	1.14	0.86	75.61 ± 13.36	0.45
PCB-193	0.92	0.65	70.98 ± 14.79	0.36
PCB-194	0.83	0.54	64.73 ± 10.77	0.27
PCB-195	0.88	0.69	78.33 ± 14.04	0.36
PCB-199 (201)[c]	0.83	0.51	61.65 ± 9.92	0.24
PCB-203	0.85	0.42	49.41 ± 7.81	0.21
PCB-206	0.89	0.44	49.21 ± 8.11	0.21

[a] DB-1701 value (all other values represent the average of the 2 columns unless otherwise noted).
[b] DB-5 value (all other values represent the average of the 2 columns unless otherwise noted).
[c] Value in parentheses is BZ number.

Table 4. Recovery and LOD of pesticides ($n = 7$)

Analyte	Spike level, ng/mL	Average amount detected, ng/mL	Average recovery ± standard deviation, %	LOD, ng/mL
HCB	1.07	0.54	50.40 ± 13.87	0.44
γ-HCCH	1.27	0.49	38.80 ± 12.25	0.47
Aldrin	0.91	0.61	66.54 ± 7.93	0.22
β-HCCH	1.59	0.63	39.60 ± 10.40	0.50
Oxychlordane[a]	1.13	0.77	68.83 ± 10.60	0.36
Heptachlor epoxide[a]	1.08	0.58	53.95 ± 6.03	0.20
trans-Nonachlor	1.18	0.88	74.56 ± 8.99	0.32
p,p'-DDE[a]	1.01	0.70	69.59 ± 7.82	0.23
Dieldrin[a]	1.52	1.29	85.22 ± 4.99	0.23
Endrin	1.67	1.74	104.22 ± 5.02	0.25
o,p'-DDT[a]	1.48	0.99	66.99 ± 7.47	0.33
cis-Nonachlor[a]	0.83	0.60	72.20 ± 6.18	0.15
p,p'-DDT	1.69	1.11	65.85 ± 6.35	0.32
Mirex	0.83	1.05	125.77 ± 8.70	0.21

[a] DB-1701 value (all other values represent the average of the 2 columns).

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Table 5. Recoveries of surrogates spiked in base bovine serum (n = 7)

Surrogate	Spike level, ng/mL	Average recovery, %	Standard deviation, %
PCB-30	1.03	62.01	9.58
PCB-204	1.07	69.29	4.85
HxBrB	2.00	72.72	12.05
α-HCCH	1.33	30.76	7.40
Perthane	3.34	56.32	11.12
DCBP	3.34	87.98	9.51

less injector. GC operating parameters for both columns were essentially identical. Injector temperature was kept at 270°C, and detector was kept at 340°C. Helium was used as carrier gas at a constant flow rate of 1.7 mL/min. Makeup gas was 5% methane in argon at a flow rate of 40 mL/min. The initial column temperature was held at 90°C for 4 min and then ramped to 180°C at 18°C/min and held for 1 min. The temperature was increased to 200°C at 0.9°C/min and held for 1 min. Finally the temperature was increased to 270°C at 1.5°C/min. The final temperature was held for an additional 10 min for the DB-1701 column and for an additional 15 min for the DB-5 column. A 2 μL portion of each GC sample was injected by the autosampler in the splitless mode. Before GC analysis of samples, blank DCN (IS) was run on each GC system to ensure that the instrument, syringe, and column were free of contaminants. Both GCs were controlled and data were acquired and processed by a single Turbochrom 4.0 chromatography software data system (PE Nelson, San Jose, CA). Analytical results were exported electronically to a spreadsheet for further data analysis and report preparation.

Chromatograms of calibration standard mix 1 and 2 for each column are presented in Figures 2 and 3. Analytes selected for calibration solutions and their relative RTs for each column are presented in Tables 1 and 2. Each peak was identified by comparing its RT with an authentic standard. First-order calibration curves were generated for each analyte. The area response ratio of the analyte to the IS was regressed on the concentration ratio of the analyte to the IS. If the correlation coefficient (r^2) was 0.990 or greater, the calibration curve of a particular analyte was accepted. Accuracies of calibration standard curves were verified by analyzing all levels of calibration standards and comparing results against their expected values. If results based on calibration standards varied by no more than 10% from expected values, the calibration curves generated for a particular run were accepted for quantitation.

Results and Discussion

Calibration standard mix 1 consisted of 1 IS, 31 PCBs, 1 polybromobiphenyl (PBB) congener, and 2 pesticides. Calibration standard mix 2 contained 1 IS, DCBP, and 17 pesticides. HCB and *p,p'*-DDE were included in calibration standard mix 1 because of their elution in 6% fraction 1 and their frequency of detection in human samples. As shown in Figure 2, a DB-5 column offered the best baseline resolution for

Table 6. Levels of PCB congeners in human serum[a]

PCB congener	No. of samples in which congener was detected	Mean	Median	Range, ng/mL
PCB-28	1	0.21	0.23	ND–0.08
PCB-74	10	0.21	0.17	0.07–0.60
PCB-99	9	0.16	0.16	ND–0.22
PCB-105	2	0.18	0.21	ND–0.08
PCB-118	10	0.41	0.40	0.22–0.62
PCB-138	10	1.32	1.05	0.76–2.59
PCB-146	10	0.30	0.22	0.16–0.64
PCB-153	10	2.05	1.58	0.94–4.14
PCB-156	9	0.18	0.18	ND–0.36
PCB-170	10	0.81	0.65	0.35–1.58
PCB-172	2	0.16	0.17	ND–0.14
PCB-177	9	0.13	0.10	ND–0.25
PCB-178	3	0.19	0.24	ND–0.11
PCB-180	10	1.67	1.34	0.78–3.33
PCB-183	8	0.18	0.17	ND–0.37
PCB-187	10	0.67	0.57	0.29–1.49
PCB-193	7	0.16	0.18	ND–0.25
PCB-194	10	0.22	0.16	0.05–0.43
PCB-199 (201)[b]	9	0.25	0.18	ND–0.52
PCB-203	9	0.21	0.18	ND–0.43
Total PCBs		9.66	7.85	5.84–17.73

[a] Data not corrected for surrogate recovery. ND, not detected; ND = LOD/2.

[b] Value in parentheses is BZ number.

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Table 7. Levels of pesticides in human serum[a]

Pesticide	No. of samples in which a pesticide was detected	Mean	Median	Range, ng/mL
β-HCCH	10	4.01	3.35	1.65–8.68
HCB	10	7.69	7.02	0.63–14.0
Heptachlor epoxide	8	0.15	0.13	ND–0.43
Oxychlordane	8	0.15	0.13	ND–0.40
p,p'-DDE	10	41.63	34.60	7.24–124
p,p'-DDT	10	0.82	0.88	0.29–1.62
trans-Nonachlor	10	0.23	0.21	0.11–0.39

[a] Data not corrected for surrogate recovery. ND, not detected; ND = LOD/2.

Table 8. Recovery of surrogates from human serum (*n* = 10)

Surrogate	Spike level, ng/mL	Mean, %	Median, %	Range, %
PCB-30	1.03	65.00	64.10	53.4–76.7
PCB-204	1.07	80.50	82.70	63.6–87.4
HxBrB	2.00	88.10	87.50	68.5–102.4
α-HCCH	2.00	50.30	53.50	29.5–61.5
Perthane	10.01	93.00	95.00	64.4–107
DCBP	5.01	88.10	91.30	59.3–116

all analytes in calibration standard mix 1 except PCBs 56 and 60. The DB-1701 column gave 2 unresolved peaks: PCB congeners 60 and 99 and PCB congeners 138 and 178. With the present GC conditions, PCB congener 60 could not be resolved by either column. Calibration standard mix 2 contained all pesticides and was best resolved by the DB-1701 column. Heptachlor epoxide and oxychlordane, dieldrin and *p,p'*-dichlorodiphenyldichloroethylene (DDE), and *o,p'*-dichlorodiphenyltrichloroethane (DDT) and *cis*-nonachlor coeluted on the DB-5 column.

Each fraction was analyzed on both columns, and results were produced by processing against calibration curves gener-

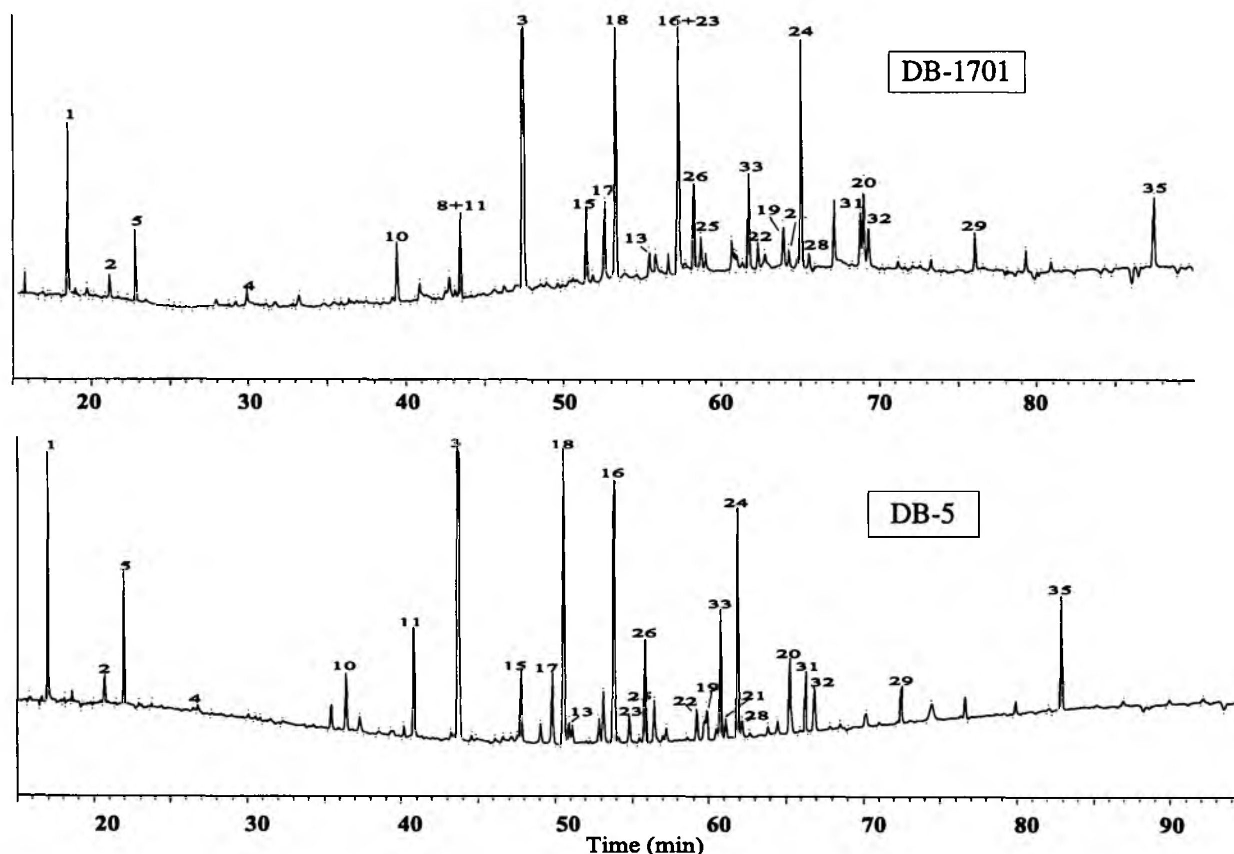

Figure 4. Example of a gas chromatographic pattern of 6% fraction 1 (DB-5 and DB-1701) as found in a selected human serum sample. Peaks identified against calibration standard mix 1.

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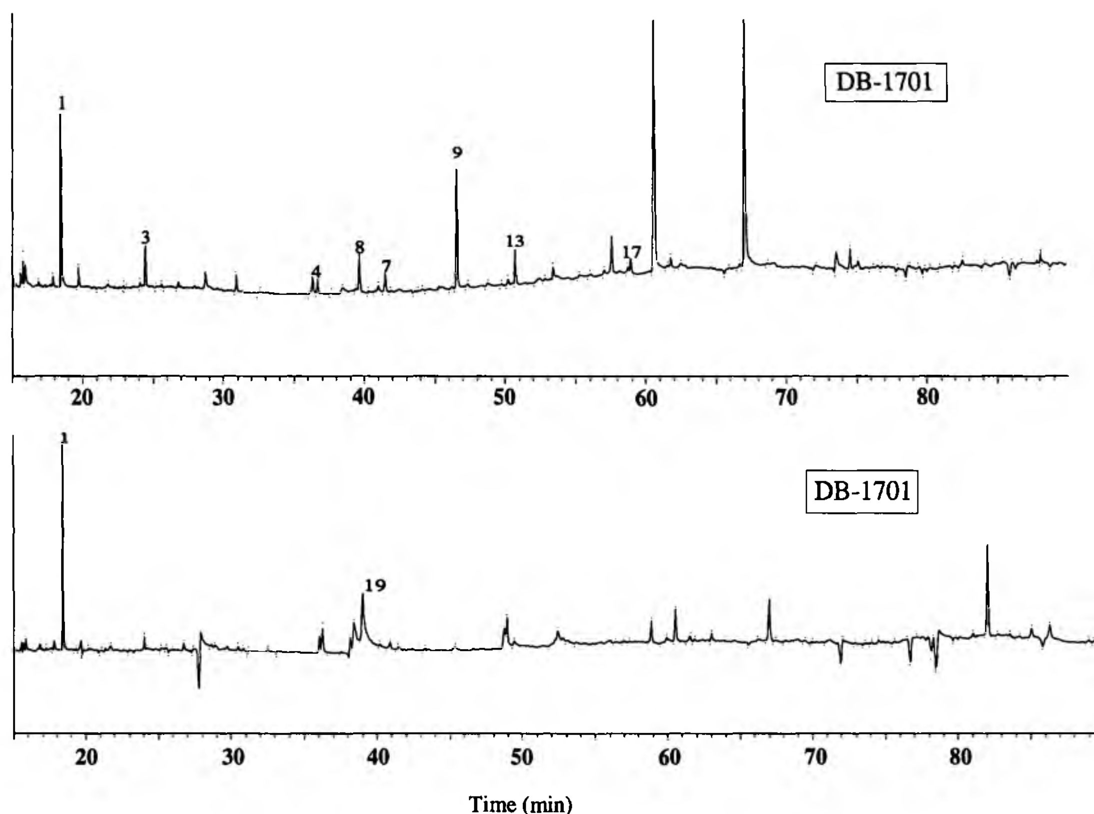

Figure 5. Example of a gas chromatographic pattern of 6% fraction 2 (top) and 15% fraction 2 (bottom) as found in a selected human serum sample. Peaks identified against calibration standard mix 2.

ated by calibration standard mix 1 and mix 2. For reporting purposes, average values of results from the 2 columns were reported if an analyte was well resolved and the results were within 15%. The lower value was taken when results from the 2 columns differed by more than 15%. Results for analytes well resolved by only one column were accepted. Because PCB 60 coelutes on both columns, it was verified by eliminating PCB 99 on the DB-5 column or PCB 56 on the DB-1701 column.

To examine the efficiency of this procedure, we spiked 2 mL BBS (to yield a spike of about 1 ppb of each analyte) with 25 μL of a mixture consisting of PCBs and pesticides (each about 100 ppb), as reported in Tables 1 and 2. BBS was also spiked with a surrogate mix, agitated on a Vortex mixer, allowed to equilibrate overnight in a refrigerator before extraction, and analyzed. This experiment was repeated 7 times. Recoveries were determined by subtracting background levels found in RB or BBS and dividing results by the actual amount spiked. Recoveries of selected analytes and surrogates are presented in Tables 3–5. Recoveries ranged from 49 to 84% for PCBs and from 39 to 126% for pesticides. Surrogate recoveries ranged from 31 to 88%.

Recoveries of analytes and surrogates were compared for variation among batches. The efficiency and the repeatability of the procedure also were monitored by analyzing QC samples (SRPs and CHS) in each run and comparing results against previously reported values. Limits of detection (LODs) were determined from spike and recovery experiments with consideration for normal GC detector response for various analytes. A mixture of analytes estimated at about twice the limit of quantitation (LOQ) was spiked into BBS, and the spiked BBS was cleaned up, and analyzed with a properly maintained GC. Recovery of each analyte was determined. This procedure was repeated 7 times to evaluate standard deviation (S) and to estimate standard deviation at zero concentration (S_0). Because the concentration of spiked analytes was about twice LOQ, S was considered equivalent to S_0. LODs were calculated arbitrarily as $3S_0$ (11; Tables 3 and 4). The results appear to be high. In our experience, about half of these amounts can be detected easily by following this procedure.

To determine how well this method performs with unknown human samples, we analyzed 10 archived serum samples. Tables 6 and 7 summarize amounts of selected PCBs and CPs found in these samples. Half of the LOD was substituted for undetected analytes in calculating the mean and median for each analyte and for total PCBs. Recoveries of surrogates are listed in Table 8. α-HCCH recoveries were low (30–62%); recoveries of other surrogate ranged from 53 to 116%. The GC patterns of the 3 adsorption chromatography fractions found in one of the human serum samples are presented in Figures 4 and 5. The 6% fractions 1 and 2 gave few contaminant peaks, whereas 15% fraction 2 (for detection of dieldrin and endrin) yielded many extraneous peaks that could make quantitation of both dieldrin and DCBP erroneous. Inserted QC materials were identified by the same criteria used for unknown samples. Results of QC samples were reviewed and compared against characterized values to determine the validity of the analytical run.

The procedure is a good routine method for determining a mixture of PCBs and CPs in human serum. It also can be used

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to analyze human plasma for the presence of PCBs and pesticides. In general, however, the sample cleanup consumes a large volume of solvent, is labor intensive, and results in a significantly low sample throughput. Solid-phase extraction (SPE) is being considered as an alternative. Preliminary studies with SPE show that recoveries are lower and that samples with high lipid content pose a problem with solvent elution. Because 3 extracted fractions are obtained for instrumental analysis, a fair number of specific PCBs or pesticides from a sample can be resolved and analyzed on a single GC column without interference from each other.

Conclusions

The procedure was developed to determine the identities and concentrations of components of a mixture of selected PCBs and CPs in human serum. Each sample is spiked with a series of surrogates. Cleanup by adsorption chromatography yields 3 elution fractions. Each fraction is analyzed by GC–ECD on DB-5 and DB-1701 capillary columns for positive confirmation of selected PCB congeners and a group of CPs. The DB-5 column proved better for resolving a mixture of PCBs, whereas the DB-1701 column was more suitable for pesticide analysis. Surrogates and QC samples are introduced to monitor the performance of an analytical run. Recoveries ranged from 39 to 126% for selected analytes and from 31 to 88% for surrogates. Analysis of human serum and QC samples gave consistent results, except for DCBP and dieldrin. α-HCCH recoveries were low (30–62%); other surrogate recoveries ranged from 53 to 116%. The 3 extracted fractions obtained after cleanup procedure and use of 2 sets of calibration standards, permit analysis of a specimen for the presence of a wide range of PCBs and pesticides. Use of dual columns and a number of surrogates provides better resolution and quality assurance. This procedure also may be used to analyze human plasma or denatured serum for the presence of PCBs or CPs.

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