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Article

Determination of Polychlorinated Biphenyls and Organochlorine Pesticides in Human Serum by Gas Chromatography with Micro-Electron Capture Detector

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A method for determination of concentrations of polychlorinated biphenyl congeners (PCB-28, 52, 101, 118, 138, 153, 156, and 187) and organochlorine pesticides (hexachlorobenzene, alpha-hexachlorocyclohexane, beta-hexachlorocyclohexane, gamma-hexachlorocyclohexane, delta-hexachlorocyclohexane, p,p′-dichlorodiphenyl dichloroethylene, o,p′-dichlorodiphenyl trichloroethane, p,p′-dichlorodiphenyl dichloroethane, p,p′-dichlorodiphenyl trichloroethane, alpha-chlordane, gamma-chlordane, heptachlor, heptachlor epoxide, and aldrin) in human serum is developed. Recovery is assessed with artificial serum, in which PCBs and OCPs could not be detected. The method is then confirmed with pooled human serum. Experiments are performed by adding two concentrations of analytes (0.5 μg/L and 1.0 μg/L) to both matrices. The sample pretreatment process involves denaturing with a mixture of water–1-propanol (v:v, 85:15), extraction with a C–18 cartridge, and cleanup with an Alumina B cartridge. This process required about 2 mL of serum. The limit of detection ranged from 0.05–0.35 μg/L for all the analytes. Recovery of analytes at low and high spiking concentrations varied from 63–122% and 61–124% for artificial serum and pooled human serum, respectively. Relative standard deviation was lower than 16% and 18% for artificial serum and pooled human serum, respectively. Stability of the method, expressed as relative standard deviation, was lower than 14%. The method has been applied in epidemiological research.

Introduction

Polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) are classified as persistent organic pollutants (POPs), which are highly lipid-soluble and persistent in the environment (1). They can accumulate in the fatty tissue of humans after exposure from the food chain. Some of the POPs have been identified as endocrine disruptors, altering normal function of the endocrine and reproductive systems, which may have adverse effects on human health, such as cancer, birth defects, learning disabilities, and immunological, behavioral, neurological or reproductive disturbances (2).

Determination of concentrations of PCBs and OCPs in biological samples, such as serum or milk, requires a quantitative approach for assessing exposure risk in epidemiological studies. Conventional methods for determination of concentrations of PCBs and OCPs in serum involve liquid–liquid extraction (LLE) (3–5) or solid-phase extraction (SPE), followed by clean-up steps with silica gel clean-up cartridges (4–6) or an Alumina B cartridge (Al-B cartridge) (7). Extracts are then analyzed by gas chromatography coupled with electron capture detection (GC–ECD) (8), GC–mass spectrometry (MS) (6), or GC–high resolution mass spectrometry (HRMS) (9). Methods based on LLE require many reagents and are often complex and time-consuming, which make them less than optimal for the number of samples in epidemiological research (10, 11). In contrast, SPE methods use less solvents and are generally simple, making them more appropriate for use in epidemiological research. However, the main disadvantage of most SPE methods is that they require large amounts of serum, usually about 5–10 mL (10), which is very difficult to obtain, especially from children. Thus, it is very important to develop a method that requires less serum.

This paper describes a method for determination of concentrations of selected PCBs and OCPs in human serum and required only 2 mL of serum. For sample pretreatment, a mixture of water-1-propanol (v:v = 85:15) was chosen as protein denaturant, and analytes were extracted using a C-18 cartridge and cleaned-up with a SampliQ Alumina B cartridge (Al-B cartridge). Analytical work was performed using GC–μECD. To decrease interference from residual PCBs and OCPs in human serum, artificial serum was initially chosen as a matrix to check the accuracy and precision of the method. Later, pooled human serum was chosen for confirmation of the method. Measurements were performed by spiking samples with two concentrations of PCBs and OCPs (0.5 μg/L and 1 μg/L) in both artificial serum and pooled human serum.

Materials and Methods

Reagents and materials

Analytes that were measured included hexachlorobenzene (HCB), alpha-hexachlorocyclohexane (α-HCH), beta-hexachlorocyclohexane (β-HCH), gamma-hexachlorocyclohexane (γ-HCH), delta-hexachlorocyclohexane (δ-HCH), p,p′-dichlorodiphenyl dichloroethylene (p,p′-DDE), o,p′-dichlorodiphenyl trichloroethane (o,p′-DDT), p,p′-dichlorodiphenyl dichloroethane (p,p′-DDD), p,p'-dichlorodiphenyl trichloroethane (p,p'-DDT), alpha-chlordane (α-chlordane), gamma-chlordane (γ-chlordane), heptachlor, heptachlor epoxide, aldrin, and PCBs (IUPAC NO. 28, 52, 101, 118, 138, 153, 156, 187, and 198). PCB-198 was used as an internal standard. Standard solutions of OCPs and PCBs were all 10 μg/mL dissolved in iso-octane and stored at 4°C until used. They were purchased from AccuStandard, Inc. (New Haven, CT). Before use, mixtures of standard solutions containing all analytes

were prepared in iso-octane at concentrations of 200 μg/L and stored at 4°C.

All solvents (methanol, hexane, dichloromethane, acetone, iso-octane, *n*-propanol) were HPLC grade or better and purchased from Merck Co. (Darmstadt, Germany), except dichloromethane, which was from Tedia Co. (Fairfield, OH). All equipment and materials used in the process of serum pretreatment were purchased from Agilent Technologies (Wilmington, DE).

Serum samples

Artificial serum used for method development was purchased from Biopanda Diagnostics (Newtownards, UK). Real human serum used to confirm the method was obtained from the clinical lab at XinHua Hospital, which was affiliated with Shanghai Jiao Tong University School of Medicine. Blood was extracted by venipuncture and collected in vacutainer blood tubes. Serum was separated by centrifugation at 4,500 rpm for 10 min and pooled together. The pooled serum was then transferred into 40 mL glass bottles equipped with Teflon screw caps (Agilent Technologies, Wilmington, DE), which were washed with acetone and heated at 300°C overnight before use, and then kept frozen at −20°C until analyzed.

Cord blood serum, to which the method was applied, was collected from healthy pregnant women at delivery. They were participants in a cross-sectional study aimed at investigating exposure levels of PCBs and OCPs in cord serum. Informed consent was obtained from all subjects. Cord blood serum was separated by centrifugation and frozen at −80°C until used.

Sample pretreatment procedure

Before serum pretreatment, spiking solutions including all analytes and the internal standard solution were each prepared in acetone at a final concentration of 100 mg/L. Recovery experiments were performed by spiking with two concentrations of analytes: 0.5 μg/l and 1 μg/l.

Serum (2 mL), containing 10 μl or 20 μl spiking solution of each analyte, and 20 μl internal standard solution, was sequentially added to 4 mL vials. After that, 2 mL mixture of water-1-propanol (v:v, 85:15) was added into the mixture. Vials were sealed with Teflon screw caps. Then the spiked samples were equilibrated in an ultrasonic bath for 20 min. Meanwhile, C-18 cartridges were conditioned with 3 mL methanol and 3 mL × 2 mixture of water−1-propanol (v:v, 85:15). Afterwards, spiked samples were loaded onto C-18 cartridges, the vials were washed with 1 mL × 3 mixture of water−1-propanol (v:v, 85:15), and the mixtures of water−1-propanol were loaded onto C-18 cartridges. To wash out interfering materials in C-18 cartridges, C-18 cartridges were washed with 10 mL deionized water. They were then centrifuged for 30 min at 4,500 rpm and dried in ambient air for 1 h. SampliQ Alumina B cartridges (Al-B cartridges) were washed with a 2 mL mixture of hexane and dichloromethane (v:v, 50:50) and were attached to the lower end of the C-18 cartridges. PCBs and OCPs were eluted with 8 mL mixture of hexane− dichloromethane (v:v, 80:20). The eluate was concentrated to approximately 500 μL under a gentle nitrogen stream and then transferred to a tube that was inserted into a 2 mL autosampler vial that contained 100 μL iso-octane. Finally, the extracts were concentrated to 100 μL.

Analytical conditions

Analytical work was performed using GC−μECD (7890A; Agilent Technologies, Wilmington, DE). The extract (1 μL) was auto-injected using the splitless injection mode. Nitrogen (>99.999% purity) was used as the carrier gas, and the average velocity was 1.5 mL/min. Injector and detector temperatures were set at 280°C and 320°C, respectively. Nitrogen was used as the make-up gas with a velocity of 60 mL/min. Chromatographic separation was accomplished using a DB-1701 column (60 m × 0.25 mm × 0.25 um) (Agilent Technologies, Wilmington, DE). The temperature program was: initial temperature, 80°C; increase of 10°C/min to 180°C; increase of 2°C/min to 220°C; maintain at 220°C for 3 min; increase of 12°C/min to 250°C; decrease of 15°C/min to 200°C; increase of 4°C/min to 250°C; and maintain at 250°C for 17 min.

The linearity of μECD was determined by using standard solutions of the analytes at 1, 5, 10, 20, and 100 μg/L in iso-octane. The correlation coefficient was $R^2 \geqq 0.999$. Limit of detection (LOD) was evaluated from the ratio of signal-to-noise. The peak of each analyte should be at least three times higher than the peak of the noise. Results were obtained by subtraction between the detected value of samples and blanks. To perform the statistical analyses, the concentrations below LOD were set at half of the LOD.

Results and Discussion

Method development using artificial serum

Potential interference by residual PCBs and OCPs in human serum is an important factor that must be considered in the process of method development. The food chain is the main pathway of entry for PCBs and OCPs into the human body. Therefore, the general population may have residual pollutants in their serum, even without occupational exposure. In this experiment, artificial serum was chosen as a matrix to reduce the interference by PCBs and OCPs in human serum and as a blank for initial measurements in human serum. Neither PCBs nor OCPs were detected in artificial serum. Except for PCBs and OCPs, other components in artificial serum were similar to real human serum, which makes it appropriate for method development without interference from background materials.

A pretreatment process for samples, which included extraction and clean-up steps, was needed because of the complexity of serum. A mixture of water−1-propanol was chosen as a protein denaturant. The water−1-propanol mixture prevented protein co-precipitation with analytes. During sample processing and analysis, the extraction cartridge was not clogged, as the spiked serum sample was able to pass through the C-18 cartridge in a dropwise manner. In previously published studies, many solvents have been used as protein denaturants, such as mixtures of water−1-propanol (12), formic acid and acetonitrile (13, 14), urea (15), and 5% Na_2SO_4 and acetonitrile (5). Besides the water−1-propanol mixture, other solvents were also tried as potential protein denaturants; however, these yielded unsatisfactory results. For example, formic acid caused excessive precipitation and clogging of the extraction cartridge. To improve pretreatment efficiency, precipitates were washed three times and removed by centrifugation. The results were still unsatisfactory. Furthermore, addition of these

extra steps complicated the process and wasted time. Similar to results with formic acid, results using the mixture of 5% Na_2SO_4 and acetonitrile were also unsatisfactory. It made human serum viscous, which blocked the C-18 cartridge when the mixture passed through. Other studies also encountered this problem and in those studies the authors suggested that serum should be handled within 1 h after acidification (13). In reality, however, this was very difficult to accomplish.

A cleanup step was needed to decrease interference from other components because of the complexities of human serum. Besides PCBs and OCPs, human serum contains other components, such as protein, lipids, fatty acids and inorganic salts, which could influence the final results. After the process of protein denaturation and extraction by the C-18 cartridge, most of the protein was removed and inorganic salts were washed out with water. Lipids, however, were still present. Lipids, which have high boiling points, readily adhere to the chromatographic column, thereby decreasing the service life of the column. Another important factor taken into consideration was that some fatty acids have similar retention times as many of the analytes and could interfere with their detection (13).

In this study, the Al-B cartridge was chosen to cleanup the samples, thereby minimizing interference. Use of the Al-B column made the basic line cleaner and the LOD lower. In a previously published work, a silica gel clean-up cartridge was used (5, 6, 12). It removed lipids and other sources of interference and had been recognized as a good way to remove lipids. However, it also had some disadvantages. Some OCPs, such as heptachlor epoxide, o,p′-DDT, α-endosulfan, aldrin, endrin, diedrin, and methoxychlor, are sensitive to acid and could be degraded when they came in contact with the acidified silica gel clean-up cartridge (5). Furthermore, the silica gel clean-up cartridge needed to be freshly prepared every time, and addition of silica-sulfuric acid with stirring was necessary to get good homogenization. In reality, however, it was very difficult to assure adequate homogenization among batches.

The recovery and RSD are shown in Table I. Recovery of analytes ranged from 64–112% and 61–124% at low and high spiking concentrations, respectively. RSD values were below 16% in both experiments.

Table I
Recovery and RSD of Artificial Serum Samples ($n = 6$)*

	0.5 μg/L		1 μg/L	
Analytes	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
HCB	83	6	96	12
α-HCH	64	7	96	3
β-HCH	87	5	111	12
γ-HCH	89	5	105	7
δ-HCH	87	6	61	5
p, p′-DDE	97	5	98	8
o, p′-DDT	103	6	112	6
p, p′-DDD	88	6	79	13
p, p′-DDT	77	7	111	3
α-chlordane	106	2	112	2
γ-chlordane	95	5	89	3
heptachlor	66	5	67	7
heptachlor epoxide	99	7	66	0
aldrin	74	8	89	9
PCB-28	91	9	88	4
PCB-52	102	16	88	4
PCB-101	95	4	124	12
PCB-118	112	3	116	3
PCB-138	97	7	85	7
PCB-153	79	4	92	6
PCB-156	97	7	102	4
PCB-187	86	8	96	6

*Note: hexachlorobenzene (HCB), alpha-hexachlorocyclohexane (α-HCH), beta-hexachlorocyclohexane (β-HCH), gamma-hexachlorocyclohexane (γ-HCH), delta-hexachlorocyclohexane (δ-HCH), p,p′-dichlorodiphenyl dichloroethylene (p,p′-DDE), o,p′-dichlorodiphenyl trichloroethane (o,p′-DDT), p,p′-dichlorodiphenyl dichloroethane (p,p′-DDD), p,p′-dichlorodiphenyl trichloroethane (p,p′-DDT), alpha-chlordane (α-chlordane), gamma–chlordane (γ-chlordane), polychlorinated biphenyls (PCBs).

Method confirmation with pooled serum and application to cord blood serum

After the method was developed using artificial serum (Figure 1), it was confirmed by analyses of pooled human

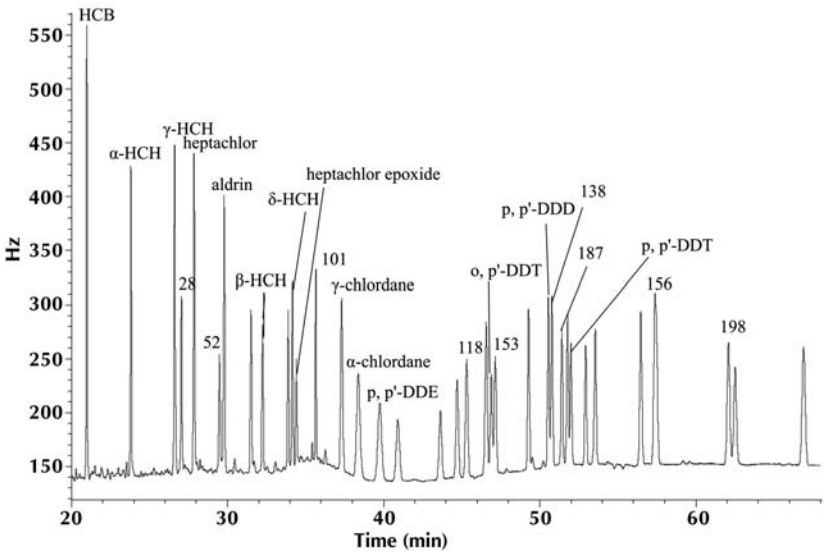

Figure 1. Chromatograms of spiked artificial serum at 1μg/L.

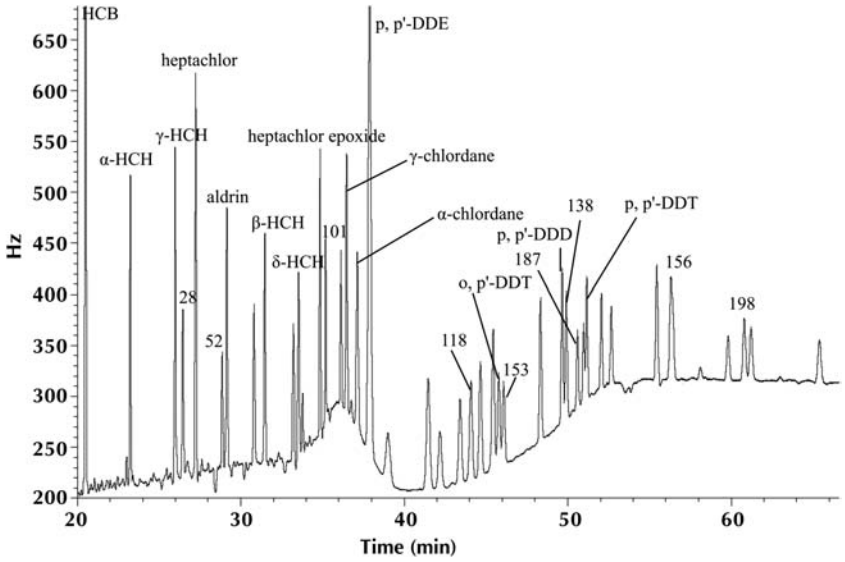

Figure 2. Chromatograms of spiked pooled human serum at 1μg/L.

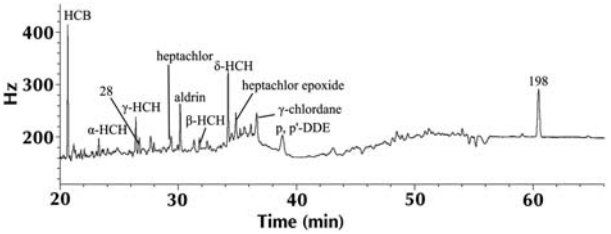

Figure 3. Chromatograms of human cord blood serum.

Table II
Recovery and RSD of the Pooled Human Serum Samples ($n = 6$)

	0.5 μg/L		1 μg/L	
Analytes	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
HCB	84	6	86	5
α-HCH	68	2	76	5
β-HCH	107	9	96	14
γ-HCH	78	7	90	2
δ-HCH	101	8	98	5
p,p′-DDE	108	7	97	16
o, p′-DDT	97	17	90	12
p, p′-DDD	80	3	77	2
p,p′-DDT	104	9	112	3
α-chlordane	103	5	88	8
γ-chlordane	111	12	74	7
heptachlor	80	9	73	10
heptachlor epoxide	88	9	64	4
aldrin	63	3	66	12
PCB-28	99	7	97	9
PCB-52	70	12	93	9
PCB-101	85	1	84	15
PCB-118	81	4	88	13
PCB-138	122	7	94	8
PCB-153	68	4	80	10
PCB-156	111	18	105	9
PCB-187	97	13	78	15

serum (Figures 2 and 3). The pretreatment process was similar as with artificial serum.

The recovery experiment was performed at both low (0.5 μg/L) and high (1 μg/L) concentrations of analyte spiking, and the results are shown in Table II. Recovery of analytes ranged from 63–122% and 64–112% at low and high concentrations, respectively. RSD values were below 17% in both experiments. The analytical method developed in this study was applied to an epidemiological study in Shanghai that was aimed at determining exposure levels of PCBs and OCPs by measurements in cord serum. Results of measurements of 294 serum samples are shown in Table III. Because the distribution of most of the results was not normal, statistical results are listed as a median and range. OCPs, p,p'-DDE, HCB, and β-HCH were found in cord serum as the major pollutants, and the percentage of each above the LOD was about 98%, 91%, and 77%, respectively. For PCBs, the percentages above LOD were lower than those for OCPs. These results were similar to previously published research (16, 17).

Table III
Concentrations of the PCBs and OCPs in Human Cord Blood Serum ($n = 294$)

Analyte	Median (μg/L)	Range (μg/L)	>MDL (n)	>MDL (%)
HCB	0.570	ND–2.309	268	91
α-HCH	0.045	ND–0.503	85	29
β-HCH	0.044	ND–6.812	225	77
γ-HCH	0.499	ND–1.844	160	54
δ-HCH	0.030	ND–2.719	132	45
p,p′-DDE	1.710	ND–23.014	288	98
o, p′;-DDT	0.085	ND–0.529	24	8
p, p′-DDD	0.035	ND–2.599	107	36
p,p′-DDT	0.045	ND–1.617	131	45
α-chlordane	0.060	ND–3.890	71	24
γ-chlordane	0.045	ND–3.819	40	14
heptachlor	0.727	ND–1.337	157	53
heptachlor epoxide	0.030	ND–1.548	67	23
aldrin	0.110	ND–2.665	73	25
PCB-28	0.095	ND–6.678	144	49
PCB-52	0.125	ND–10.196	146	50
PCB-101	0.035	ND–1.495	51	17
PCB-118	0.050	ND–0.334	18	6
PCB-138	0.035	ND–0.525	45	15
PCB-153	0.175	ND–1.200	5	2
PCB-156	0.015	ND–0.249	5	2
PCB-187	0.060	ND–0.813	3	1

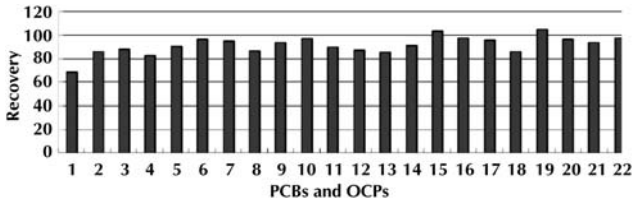

Figure 4. Recovery for method to determine concentrations of PCBs and OCPs calculated from human serum sample fortified with 0.5 μg/L of each analytes.

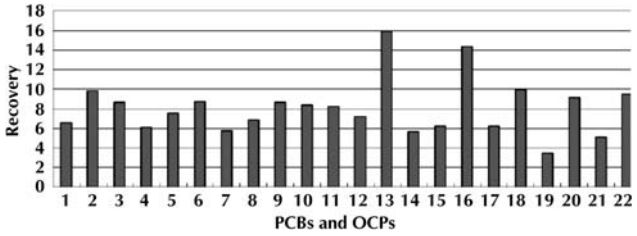

Figure 5. Precision of method for determination of concentrations of PCBs and OCPs calculated from human serum samples fortified with 0.5 μg/l of each analytes, expressed as RSD%.

Method evaluation and quality control

Linearity of μECD was established by using standard solutions of analytes at 1, 5, 10, 20, and 100 μg/L in iso-octane. Coefficients of correlation for the calibration curves were $R^2 \geq 0.999$. LOD, which was evaluated from measurements that were at least three times higher than the blank, was 0.05–0.17 μg/L for OCPs and 0.07–0.35 μg/L for PCBs. The LOD of the analytical methods developed varied widely. For monitoring the general population, the LOD should be ≤ 1 μg/L (18, 19). Using the present method, LOD values for all analytes were < 1 μg/L.

For evaluation of recovery and precision, each sample batch included a pooled human serum sample spiked with 0.5 μg/L of each analyte. Average recoveries of analytes are shown in Figure 4. The precision of the method, expressed as RSD, was lower than 10% for all the analytes, except heptachlor epoxide (16%) and PCB-52 (14%) (Figure 5).

Conclusions

A new solid-phase extraction method was developed to measure PCBs and OCPs in human serum. First, a mixture of water–1-propanol was chosen as a protein denaturant to separate analytes and protein; use of this mixture did not lead to co-precipitation. Second, analytes were extracted with C-18 cartridges and clean-up was performed using a Al-B cartridge. Third, analytical work was accomplished using GC–μECD.

Compared with other studies, artificial serum was initially chosen as the matrix to check the method and to minimize interference from residual PCBs and OCPs. Pooled human serum was then chosen to confirm the method. One limitation of our study was the absence of comparisons between different laboratories. Nonetheless, results showed good recovery and RSD for most of the analytes. LOD met the requirements for biomonitoring of the general population.

Acknowledgements

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