

The Determination of Polychlorinated Biphenyls in Selected Household Products

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The occurrence and accumulation of polychlorinated biphenyls (PCBs) in the environment has been well reviewed (AHMED 1976, PEAKALL and LINCER 1970, EDWARDS 1971) and problems associated with quantitative PCB analysis have been discussed (CHAU and SAMPSON 1975, EDER 1976, DEXTER and PAVLOU 1976). Although the production of PCBs has been stopped there is still a potential for environmental contamination from sediments, transformers and recirculation of PCBs from commercial products. A general survey of commercially available materials showed that trace amounts of PCBs were present in some commercial products (REYNOLDS 1977).

As part of our overall concern with organic compounds in drinking water, we have analysed household products which are disposed of via the sewage system. Such materials might contribute PCBs to raw water systems from which potable water supplies are taken. The results of this survey are reported here.

EXPERIMENTAL

The procedures used are adaptations of previously published methods (VILLENEUVE et al. 1973, REYNOLDS 1977). Ten gram sub-samples of paper cut into small pieces, powder and grated bar soap were soxhlet extracted for 4 hr with hexane. Liquid samples (10 g) were diluted with water (40 ml) and gently shaken with hexane (3 x 50 ml). The hexane extracts for each sample were washed twice with an equal volume of water and then dried over anhydrous sodium sulfate. The hexane solution was then concentrated to 2 ml using a rotary evaporator, bath temperature 35-40°C, and analysed by gas chromatography (GC). Those samples which showed peaks with retention times similar to PCBs were further cleaned up by column chromatography using 20 g deactivated Florisil.

The Florisil used was 80-100 mesh, supplied by Applied Science, and was heated at 300°C overnight. Water (2% W/W) was added to the Florisil in a tightly sealed bottle and the mixture shaken well and allowed to equilibrate for 24 hr before use. Each batch of deactivated Florisil was checked by the use of standard solutions of Aroclor 1248.

Prior to transfer of the sample to the column the Florisil was washed with hexane (100 ml). The sample was transferred to the column with a minimum volume of hexane and the PCBs eluted with 100 ml hexane. The eluate was concentrated to ca. 10 ml using a rotary evaporator, bath temperature 35-40°C, and then to dryness using a stream of nitrogen. The residue was then dissolved in a known volume of hexane and analysed by EG-GC and by gas chromatography-mass spectrometry (GC-MS).

The conditions for gas chromatographic analysis were: 1.8 m x 6.2 mm coiled glass column packed with 3% OV-210 on 80/100 mesh Chromosorb W(HP), with nitrogen as carrier gas at a flow rate of 60 ml/min. The oven, injection port, and the electron capture detector temperatures were 150°C, 240°C, 300°C, respectively.

Gas chromatography-mass spectrometry was carried out on a Finnigan 4000 GC-MS coupled to a data system. The gas chromatography conditions were 1.8 m x 2 mm glass column packed with 3% OV-17 on 80-100 mesh Chromosorb 750, with helium as carrier gas at a flow of 20 ml/min. The injection port was at 240°C and the oven temperature was 175°C isothermal. The mass spectrometer was operated in the electron impact mode (70 eV) and data was collected by monitoring selected ions at m/q 186, 222, 256 and 292.

For quantitation by EC-GC the peak heights of 6-8 selected peaks with retention times between 2 and 12 min (Fig. 1B) were compared to peak heights of the corresponding peaks in standard Aroclor 1248 (Fig. 1A). The values obtained for the individual peaks were then averaged to give an overall PCB value for the sample.

For quantitation by GC-MS the total integrated area for 6-8 selected peaks from the ion scans was compared to the total area of the corresponding peaks from standard Aroclor 1248.

Recovery studies on toilet paper samples spiked at 1 ug/g and 10 ug/g with Aroclor 1248 showed that recoveries were greater than 90%.

RESULTS AND DISCUSSION

A selection of brand name household products which are usually disposed of via the sewage system was purchased from local stores. These products included detergents, soap powder, fabric softeners, household cleaners, disinfectants, soap bars and toilet paper. Extracts of these products, after clean-up, were analysed quantitatively for PCBs by EC-GC and GC-MS. Aroclor 1248 was used as the standard PCB for quantitation purposes. The EC-GC peak patterns from the samples (Fig. 1B) did not exactly correspond to those from Aroclor 1248 (Fig. 1A) but the use of mixed standards of other Aroclors did not detectably improve the peak pattern agreement.

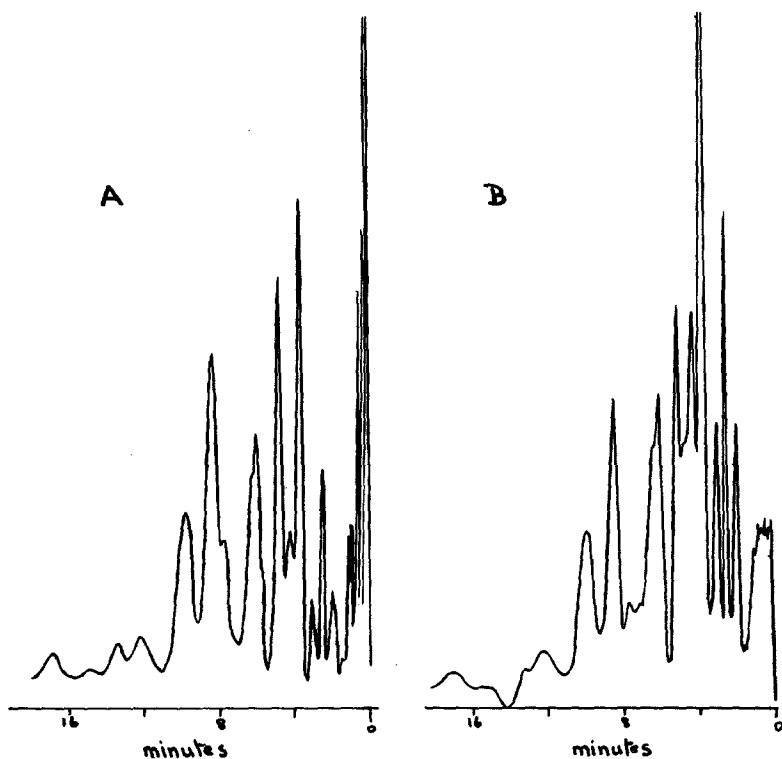


Fig. 1. EC-GC Chromatogram of (A) 0.75 ng of Aroclor 1248. (B) 0.8 ng PCB from toilet paper, Brand C (1.3 ug/g).

The GC-MS selected ion peak patterns for the sample (Fig. 2B) were similar to the peak patterns from standard Aroclor 1248 (Fig. 2A) but some differences are discernable.

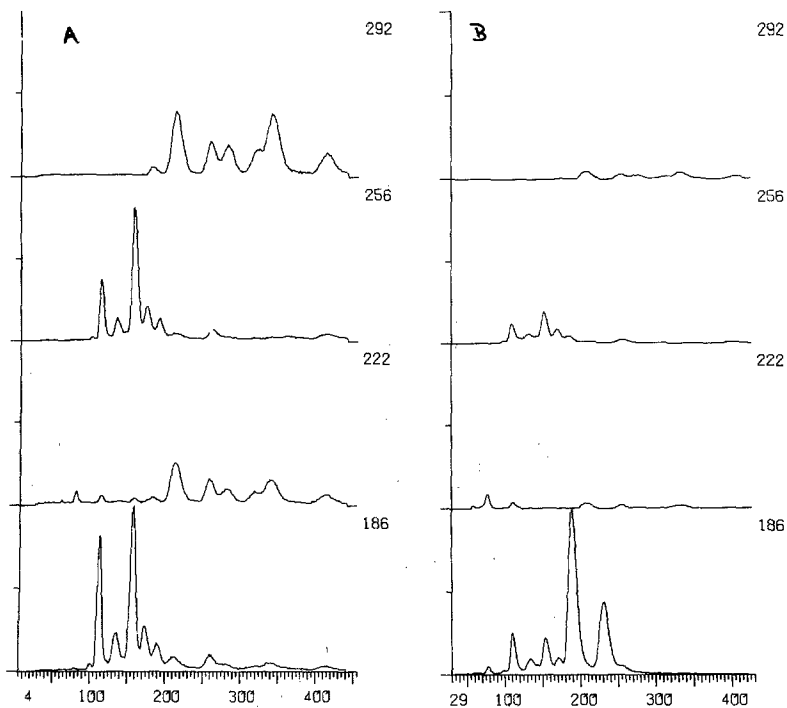


Fig. 2. GC-MS selected ion chromatograms (A) 0.7 ug Aroclor 1248. (B) 0.18 ug PCB from toilet papers, Brand C (1.3 ppm).

Most of the cleaning products contained no detectable levels of PCBs except for one soap and one soap powder (Table 1). The toilet paper samples, however, were almost all positive for PCBs (Table 1) and contained levels of PCBs ranging from trace amounts to as high as 21 ug/g (Table 2).

TABLE 1

Household Products Analysed for PCBs.

Household Product	Number of Brands Analysed	Number of Brands Containing PCBs
Detergent/Soap Powder	4	1
Fabric Softeners	3	0
Cleaners/Disinfectants	7	0
Soap	9	1
Toilet Paper	11	9

TABLE 2

Levels of PCBs Found in Household Products

Product	Levels of PCBs (ug/g)	
	EC-GC	GC-MS
Soap Powder - Brand A	0	0
	0.07	0.03
	0.07	0.04
	0.10	0.04
Soap	0.48	0.35
	0.04	<0.01
	0.05	0.01
	0.08	<0.01
Toilet Paper - Brand A	0.03	0.01
	0.03	0.09
	0.05	0.03
	0.16	0.22
Brand C	1.2	0.92
	1.3	1.2
	1.4	1.3
	1.4	1.5
Brand D	3.8	3.9
	0.04	0.05
	0.05	0.05
	0.05	0.04
Brand E	0.42	0.63
	0.10	0.06
	0.16	0.13
	0.14	0.28
Brand H	0.08	0.07
	0.94	0.55
	21	17

The quantitative values obtained by EC-GC and GC-MS agree very well for higher levels of PCBs but are in poorer agreement for low levels of PCBs (Table 2). This is to be expected since quantitation is less accurate and any interferences present will be more significant at low PCB levels.

Other workers have reported the presence of PCBs in food packaging material (VILLENEUVE et al. 1973) and a variety of paper and paperboard (SHAHIED et al. 1973) but these products are normally disposed of via solid waste treatment procedures. The presence of PCBs in toilet papers indicates a potential source of PCB contamination of surface waters.

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