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PCBs in Transformer Oils

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Due to the suspected carcinogenic nature of polychlorinated biphenyls (PCBs), a high degree of interest in the analysis and identification of these compounds in transformer oil has developed. The Federal Drug Administration (FDA), Environmental Protection Agency (EPA), and the United States Department of Agriculture (USDA) have proposed rules calling for removal of most electrical transformers and capacitors containing PCBs from food and feed processing plants.

This paper reports the identification of Aroclors 1242, 1254, and 1260 in transformer oil samples brought to this laboratory for analysis.

EXPERIMENTAL

Apparatus. A gas chromatograph equipped with a Ni^{63} electron capture detector was used with a 1.8 x 4 mm i.d. glass column packed with 1.5% OV-17/1.95% OV-210 coated on 80/100 mesh Gas Chrom Q. Column, inlet, and detector temperatures were 200, 225, and 275°, respectively; nitrogen carrier gas flow was 60 mL/min.

Reagents and Materials. Solvents - hexane, actonitrile, pesticide grade (Fisher Scientific).

Florisil - 60-100 mesh, PR grade, used as received.

Analytical PCB Standards - Supplied by U.S. EPA, HERL, ETD, RTP.

Sodium Sulfate - anhydrous granular.

Chromatographic Columns - 25 mm o.d. x 300 mm long, with Teflon stopcocks, with coarse fritted glass plates and 300 mL reservoirs.

Procedure. The procedure followed was based primarily on the method found in the Pesticide Analytical Manual (U.S. HEW 1971) with a few modifications (U.S. EPA 1980).

Weigh 2 g of transformer oil into a test tube and add 5 ml of hexane. Tightly stopper the tube with a Teflon-lined screw cap. Place tube on rotator and rotate for 15 min. Prepare a chromatographic column (25 mm o.d. x 300 mm long, with Teflon stopcock, with coarse fritted glass plate) that contains ½ inch sodium sulfate, then 3 inches of RP Grade Florisil (60-100 mesh, used as received) topped with 1 inch of sodium sulfate. Transfer sample

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to dry Florisil column. Allow sample to sink into Florisil, applying slight vacuum to tip of column to remove solvent. Rinse the test tube with two 2 mL portions of hexane and remove all solvent from column with vacuum. Place 1 L separatory funnel under the column to receive eluate and eluate column with 70 mL, 10% H_2O/CH_3CN eluting solvent. Add 100 mL hexane to funnel and shake. Add 10 mL saturated NaCl solution, 300 mL H_2O and shake. Let separate, discard aqueous layer and rinse solvent layer with two 100 mL portions H_2O. Discard washings, transfer solvent layer to chromatographic column (as before) containing 4 inches of just sodium sulfate and elute into a 250 mL concentrator flask and concentrate to approximately 5 mL on a rotovap. Quantitatively transfer to a 13 mL graduated concentrator tube and dilute as needed for gas chromatography.

RESULTS AND DISCUSSION

One source of entry of PCBs into the environment may be from the improper handling and disposal of PCB contaminated waste oil from oil or discarded transformers. PCBs have been used as transformer cooling liquids, capacitor dielectric fluids and heat transfer and hydraulic liquids. In 1979, the EPA banned the manufacture and use of PCBs because of their adverse environmental effects. Transformers containing oil with concentrations of PCBs in excess of 500 ppm are classified as PCB transformers; those which contain between 50 and 500 ppm PCBs are considered to be PCB contaminated; and those which contain less than 50 ppm PCB are categorized as non-PCB transformers (OGATTA et al. 1980).

The concentration and type of Aroclor found in samples of transformer oils analyzed in this lab are shown in Table 1. Samples 1 and 2 are classified as PCB transformers; samples 3 through 8 are considered to be PCB-contaminated; and the remaining samples which contain less than 50 ppm PCB are categorized as non-PCB transformers. Sample 1, which contained approximately 450,000 ppm PCB as Aroclor 1260 required a one to ten million dilution in order to be analyzed by electron capture GC.

Figures 1, 2, and 3 depict the electron capture gas chromatograms of analytical standards of Aroclors 1242, 1254, and 1260 respectively. The relative retention times (relative to aldrin) of their corresponding peaks are written on the figures. The number of peaks and the characteristic peak patterns associated with each Aroclor can act as a "fingerprint" identification when analyzing samples containing unknown PCB (KIRSHEN 1981). The reagent blank associated with the analytical procedure is shown in Figure 4. It is obvious from the chromatogram that the blank contains no peaks which would interfere with the qualitative or quantitative analysis of PCBs. Figure 5 represents transformer oil No. 2 which was submitted to the laboratory for PCB analysis. A comparison of the characteristic peak pattern and relative retention times of the standard Aroclors with that of the unknown transformer oil, identifies the PCB present as Aroclor 1260.

TABLE 1

Concentration of PCBs found in samples of transformer oils (ppm).

Sample I.D.	Concentration	Aroclor
1	450000	1260
2	5500	1260
3	480	1242
4	220	1242
5*	160	1242
	30	1254
6	98	1260
7	76	1260
8	46	1260
9	28	1260
10	18	1260
11	16	1254
12	8	1260
13	5	1260
14	4	1254
15	2	1242
16	2	1260
17	<1	1260
18	<1	1260
19	<1	1254
20	<1	1242
21	<1	1242
22	<1	1242
23	<1	1242
24	0	**
25	0	**
26	0	**
27	0	**
28	0	**
29	0	**
30	0	

*Sample 5 was a mixture of two Aroclors.
**No PCB residue found at a detection limit of 1 ppm.

Two different dilutions of transformer oil No. 5 (top, 1 to 100; bottom, 1 to 1000) are depicted in Figure 6. This figure is an illustration of what a transformer oil resembles when it is a mixture of two Aroclors. Comparing the peak patterns and relative retention times of the unknown oil against standard Aroclors, identifies the two PCBs present as 1242 and 1254.

Finally, Table 2 lists the percent recovery of known amounts of Aroclors added to oil samples prior to analysis. One mL of 10μg/mL Aroclor was added to 2 gm of oil and then the fortified sample was subjected to the entire analytical procedure.

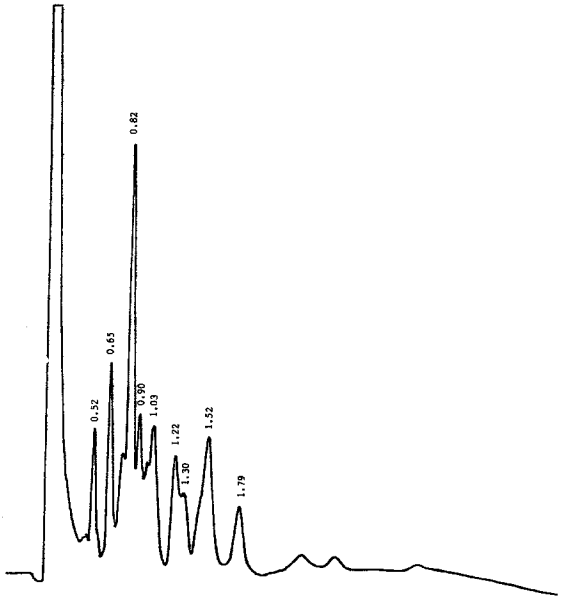

Figure 1. Electron capture gas chromatogram of an Aroclor 1242 analytical standard.

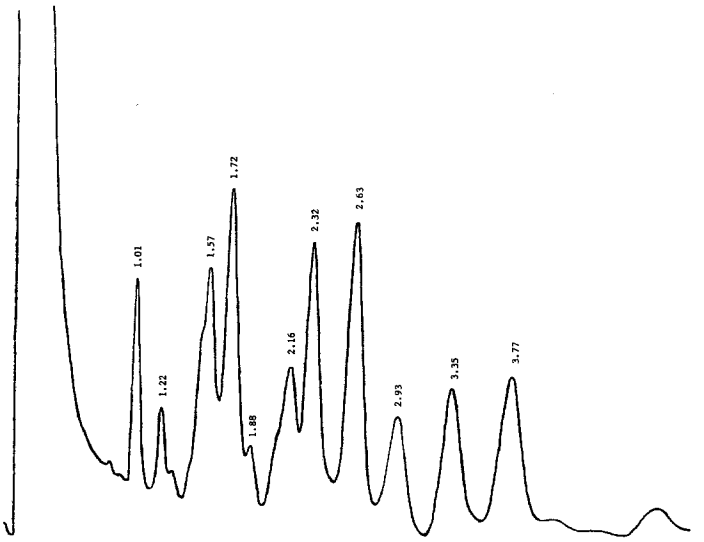

Figure 2. Electron capture gas chromatogram of an Aroclor 1254 analytical standard.

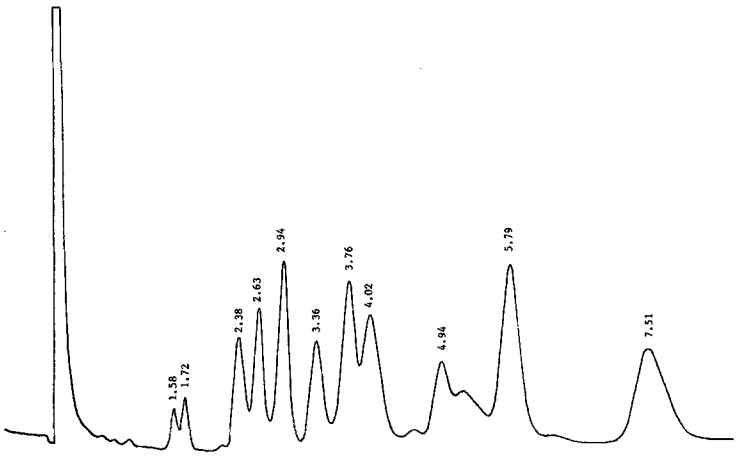

Figure 3. Electron capture gas chromatogram of an Aroclor 1260 analytical standard.

Figure 4. Electron capture gas chromatogram of the reagent blank.

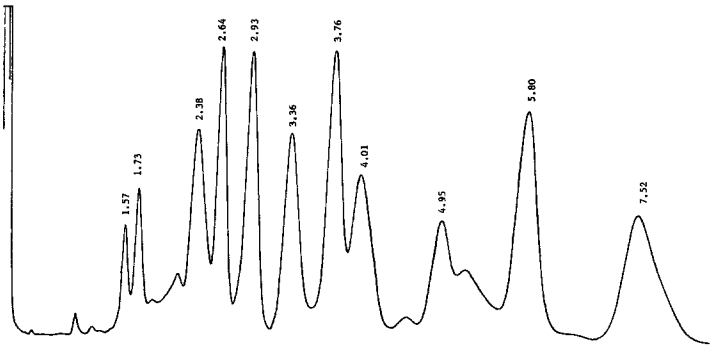

Figure 5. Electron capture gas chromatogram of transformer oil No. 2.

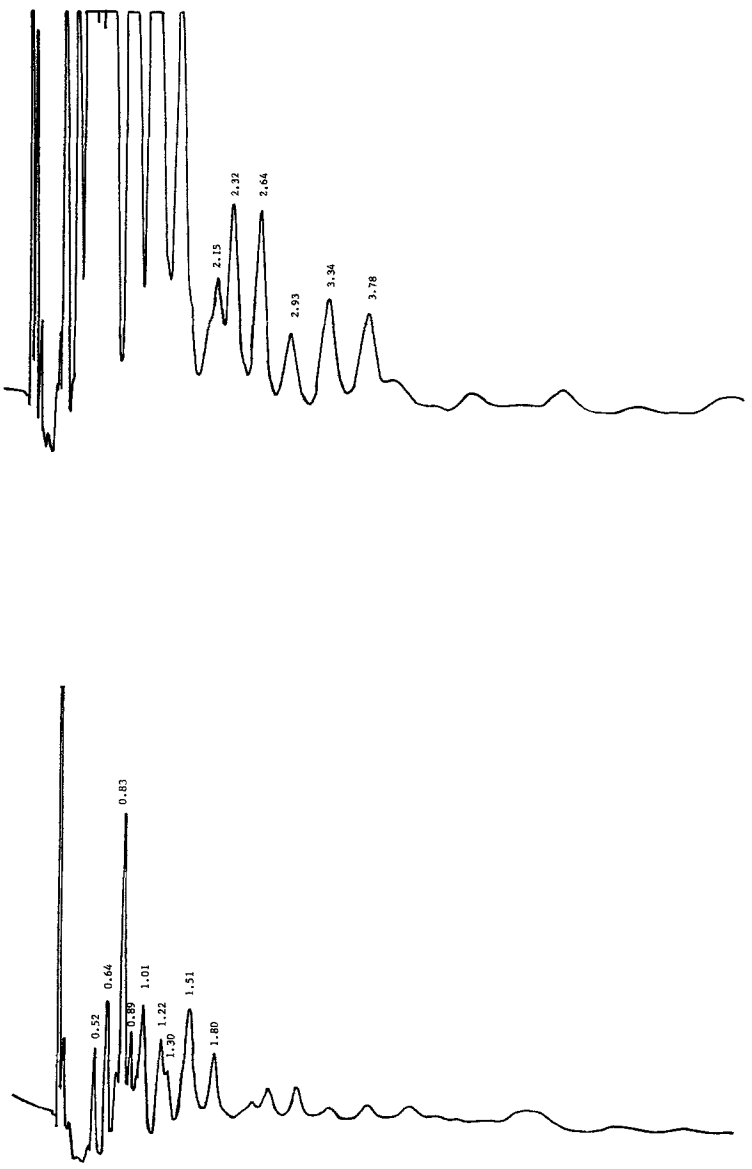

Figure 6. Top, electron capture gas chromatogram of the 1 to 100 dilution of transformer oil No. 5; bottom, electron capture gas chromatogram of the 1 to 1000 dilution of transformer oil No. 5.

TABLE 2

Percent recovery data

Fortified Sample	Aroclor	% Recovery
1	1242	71
2	1242	85
3	1254	88
4	1260	77
5	1260	80

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