Journal of Chromatography, 189 (1980) 425–427
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Gas chromatographic method for the analysis of polychlorinated biphenyls in transformer oil*

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(Received September 25th, 1979)

Polychlorinated biphenyls (PCBs) are widely distributed in the environment and are ubiquitously present in birds, fish, mammals, soils and water. One source of entry of PCBs into the environment may be from the improper handling and disposal of PCB-contaminated waste oil from old or discarded transformers[1]. New rules under the provisions of the U.S. Toxic Substances Control Act[2], to protect the environment from further contamination by PCBs, have been implemented by the U.S. Environmental Protection Agency[3–5]. The rules prescribe marking and disposal requirements for PCBs and PCB items that contain 50 ppm or greater PCBs. Transformers with dielectric fluid containing PCBs in excess of 500 ppm are classified as PCB transformers and are subject to marking and disposal regulations; those which contain between 50 and 500 ppm PCBs are categorized as PCB-contaminated transformers and are exempt from the marking requirement only. Transformers whose dielectric fluid contain less than 50 ppm PCB are classified as non-PCB transformers and a disposal rule applies only if it contains a detectable concentration of PCB. To properly label and dispose of these oils, an analytical procedure was required for the routine testing of the oils for PCBs. Methods available[6–11] for PCB analysis were examined, but problems were encountered with time-consuming and inadequate cleanup procedures. Therefore, a simple analytical method was developed and is the substance of this report.

This method for the routine analysis of PCB residues in transformer oil, with a sensitivity of 1 ppm, utilizes silica gel to isolate the PCBs from the oil. Other adsorbents, such as Florisil, charcoal, and alumina produced less satisfactory separations.

MATERIALS AND METHODS

Woelm silica gel, 70–150 mesh, obtained from ICN Pharmaceuticals, Cleveland, Ohio, U.S.A., was heated overnight at 130° before use. Sodium sulfate was anhydrous, granular, and reagent grade. Reagent grade *n*-heptane was redistilled in an all-glass apparatus.

* Journal Series No. 2438 of the Hawaii Agricultural Experiment Station.

MONS 002717

A Hewlett-Packard Model 5750 gas chromatograph equipped with a tritium electron capture detector was used for the analysis under the following conditions: 4 ft. (1.22 m) × 4 mm I.D. borosilicate glass column containing 3% SE-30 silicone on high-performance grade Chromosorb W (80–100 mesh); column temperature 200°, injector port temperature 210°, detector temperature 208°; carrier gas was argon–methane (90:10) at a flow-rate of 60 ml/min. PCBs (Aroclors®) 1248, 1254, and 1260 were kindly furnished by Monsanto, St. Louis, Mo., U.S.A. Solutions of the PCBs for gas chromatography (GC) were prepared in heptane and contained 1–5 ng/μl.

A chromatographic column (250 × 12 mm I.D. with a 125-ml reservoir, Cat. No. K-422250), obtained from Kontes, Vineland, N.J., U.S.A., was prepared by first inserting a plug of glass wool followed by the addition of 1 cm sodium sulfate, 10 cm hot silica gel, and topped with 1 cm sodium sulfate. The column was pre-wet with 50 ml heptane. A 1-ml volume of oil solution (equiv. 0.25 g sample), prepared by diluting 2.5 g of transformer oil to a 10-ml volume with heptane, was added directly onto the surface of the sodium sulfate in the column. The walls of the column were rinsed with 1–2 ml heptane. At the moment the heptane rinse entered the surface of the sodium sulfate, 75 ml heptane was added to the column and the flow-rate was adjusted to 1 ml/min. The first 20 ml of eluate, which contained the non-polar oil components, was collected in a 25-ml graduated cylinder and discarded. The next 50 ml of eluate, which contained the PCBs, was collected in a 50-ml graduated cylinder, transferred to a 100-ml round bottom flask and evaporated to dryness on a Rinco vacuum evaporator at a water-bath temperature of 60°. The residue was transferred with heptane to a 2-ml volumetric flask, made to volume, and 5 μl was injected into the gas chromatograph. The PCBs were quantitated by comparison of peak heights in the unknown with those of commercial PCB preparations.

With this method, approximately 90% of the oil was eluted from the cleanup column in the first 20 ml of heptane. All of the Aroclors 1254 and 1260 and a major portion of 1248 were eluted in the next 40 ml; an additional 10 ml was required to completely elute the more polar components of Aroclor 1248. Three different commercial batches of Woelm silica gel were used and the required eluting volumes for each batch were the same, which indicated a uniform product.

The GC patterns of the PCBs observed during the analyses of 138 transformer oil samples resembled those of the Aroclors 1248, 1254, and 1260. Of the 56 samples which contained ≥5 ppm, Aroclor 1260 was observed in 43 samples, Aroclors 1254 and 1248 in 5 samples each, and 3 samples had PCB patterns which indicated a mixture of Aroclors 1248 and 1260. Eighty-two samples contained less than 5 ppm PCBs, 14 samples had amounts ranging from 50 to 500 ppm, and 8 contained more than 500 ppm. The highest amount of residue found in one sample, was 5020 ppm of Aroclor 1260.

The recovery of the individual Aroclors from oil samples fortified at levels ranging from 5 to 2000 ppm averaged 95%. The limit of detectability was 1 ppm.

The method described here is simple and requires less than 2 h to cleanup a series of 8 samples.

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MONS 002718

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MONS 002719