Analysis of PCB's in Transformer Oil

Pamela Edwards, Altex Scientific

Detection of part-per-million levels of polychlorinated biphenyls (PCB's) is important because of the health hazards these compounds present. PCB's were used for many years as cooling-lubricants in transformers without knowledge of their toxic effects. The improper handling of PCB's during that time has contributed to a situation where PCB's can be found in waste water, fish products, soil, and in oil which has replaced PCB's in transformers.

The analysis of PCB's in transformer oil depends in part on the limits the U.S. Environmental Protection Agency has established. At the time of this writing, the limits dictate that materials containing 50 ppm PCB's or more are subject to strict disposal regulations. Thus, a sensitive analytical method is necessary to determine the level of PCB's. Several recent articles describing both gas chromatographic (GLC) and liquid chromatographic (HPLC) methods have been published. However, these reported procedures require time consuming sample preparation.[1,2,3] The HPLC method described in this report eliminates the need for tedious sample preparation by using a detector which is specific for chemical species such as PCB's. This method requires only filtration as sample preparation and is sensitive enough to detect 1-2 ppm of PCB's.

Materials and Methods. All HPLC work was done on a Model 332 system, consisting of two 110A pumps, a 420 microprocessor/controller and a 210 injection valve with a 20 μl loop. The detector was a Tracor 965 photoconductivity detector with a zinc lamp. An Ultrasphere ODS (250 mm x 4.6 mm) column was used for analysis. A C-R1A integrator was used to plot and analyze detector output signal.

Acetonitrile (MeCN) and isopropanol (IPA) were obtained from Burdick and Jackson. A mobile phase of 56% IPA, 14% MeCN and 30% water was used for all analyses at a flow rate of 1 ml/min. PCB standards, clean transformer oil, and transformer oils contaminated with PCB's were obtained from industry. Sample preparation consisted of filtering the oil samples through a 0.45 micron filter before injection.

Results and Discussion. A chromatogram of the PCB standard, Aroclor 1242, in MeCN is shown in Figure 1. In Figure 2 a separation of the same standard at the same concentration, 100 ppm, dissolved in clean transformer oil is shown. Because it is the total amount of PCB's which is

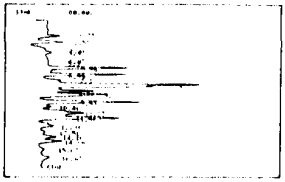

Figure 1. Photoconductivity chromatogram of standard Aroclor 1242 in acetonitrile. Sample volume was 20 μl and total concentration of PCB's was 100 ppm.

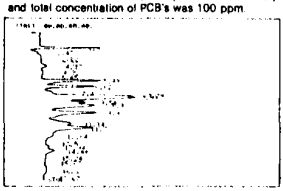

Figure 2. Photoconductivity chromatogram of standard Aroclor 1242 made up in clean transformer oil to a total PCB concentration of 100 ppm.

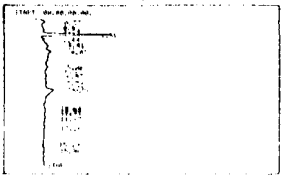

Figure 3. Photoconductivity chromatogram of 20 μl clean transformer oil.

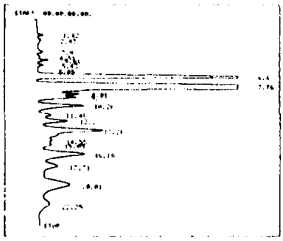

Figure 4. Photoconductivity chromatogram of 20 μl of dirty transformer oil containing more than 100 ppm PCB's.

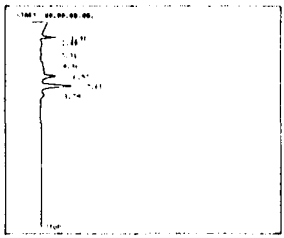

Figure 5. Photoconductivity chromatogram of 20 μl of transformer oil containing small amounts of PCB's.

required, not amounts of individual components, complete separation of all components is not critical. "Fingerprint" chromatograms of PCB's which can be quantified by a grouping system is adequate. It is apparent that injecting the oil directly reduces resolution of individual components but does not prevent quantifying total PCB's. To insure that the transformer oil contained no substances which would show up using the photoconductivity detector, a blank injection was made. No significant peaks are present in the chromatogram of clean oil shown in Figure 3. The solvent front may be due to dissolved gas in the sample.

Several samples of dirty transformer oil were also analyzed. A chromatogram of an oil sample which obviously contains more than 50 ppm PCB's and would be subject to the EPA regulations is shown in Figure 4. Quantification of the PCB's in this sample would have been quite simple using a grouped/external standard technique. A dirty oil which does not contain high levels of PCB's is shown in Figure 5. By comparing this chromatogram to those in Figures 1 and 3, it is clear that the peaks at 6.53 and 7.68 minutes represent small amounts of PCB's.

The photoconductivity detector is also sensitive to nitrosamines, sulfonated organics, and polybrominated biphenyls which are on occasion also found in transformer oils. To eliminate concerns that these compounds might be coeluting with the PCB's, fractions were collected from a dirty oil analysis and were analyzed by GC-MS. The results of the GC-MS indicated the presence of polynuclear aromatics and other organic compounds. None of the latter compounds would have been detected by the photoconductivity detector.

The instrumentation and methods described here for analyzing PCB's in transformer oil have advantages compared to other methods in that very little sample preparation is needed for specific detection with more than adequate sensitivity (1-2 ppm) for EPA regulations.

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