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Application Note

Analysis of Polychlorinated Biphenyls

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Note GC 1-73

Summary

Although polychlorinated biphenyls (PCB's) have been widely used for nearly forty years their environmental impact has only been studied since 1966. Because of their unique thermal properties and chemical inertness PCB's may be used as plasticizers, flame retardants, and insulating fluids in natural and synthetic rubber, floor tile, waxes, electrical transformers, factory coolants, printers ink, asphalt and adhesives and resins. With so many and varied uses it is easy to understand how PCB's get into the environment. Unfortunately, because of the chemical inertness, once they are introduced into the ecosystem they are extremely persistent chemicals. A recent chemical spill in an estuary resulted in complete elimination of all the shrimp and drastic reduction in oyster population for three years. Studies [1] on this spill indicate that the natural river flow is only reducing the concentration of the PCB's by 0.35 ppm/year. PCB's are also carried through the food chain and concentrated in fatty tissues of animals and eggs of birds and can slowly build up to lethal doses on long term exposure. The most common PCB's found in the tissues of animals are Aroclor® 1254 and 1260. These are two complex mixtures of PCB's whose total chlorine percentage is indicated in the last two digits of the Aroclor number.

Because the Aroclors are complex blends they present the chromatographer with several unique analysis problems. The chromatograms contain many peaks which span the retention times of the frequently studied chlorinated pesticide; therefore PCB presence constitutes severe interference in presticide residue analysis. Because of the number of peaks quantitation becomes difficult, with the standard methods involving total area of a retention window or the peak height of an intense peak known to be present in the Aroclor blend. Identification of one Aroclor in the presence of another becomes almost impossible because of overlapping compositions, and the high column temperature requirements of analysis make analysis difficult when low temperature electron capture cells are used.

Although a great deal of information has been reported about the analysis of Aroclor blends very little has been reported on the gas chromatography of the individual PCB's [2]. This applications note will report a method for the analysis of aqueous Aroclor samples and will illustrate some separations of mixtures of pure PCB's. By using the Model 5713A Electron Capture Gas Chromatograph higher column temperatures can be used to shorten the analysis time and picogram analysis can routinely be performed.

Experimental

A Hewlett-Packard Model 5713A Gas Chromatograph equipped with a Model 3370B Electronic Digital Integrator was employed in this analysis. The column used throughout this study was a 6 ft x 4mm ID borosilicate glass column packed with 1.5% OV-17/1.95% QF-1 on 80/100 Chromosorb W-HP.

The standard solutions were prepared by weighing the pure PCB's obtained from Analabs, Inc., and dissolving in appropriate volumes with G.C./Spectograde 2, 2, 4-trimethylpentane, (Baker). Solutions of Aroclor 1260 and 1254 were prepared by dissolving weighted amounts of the pure Aroclor in G.C./Spectograde 2, 2, 4-trimethylpentane. The water sample was extracted by taking a 100 ml aliquot of the sample and transfering it into a separatory funnel. Approximately 50 ml of 2, 2, 4-trimethylpentane was then added to the separatory funnel. The funnel was then stoppered and agitated vigorously for 1 minute. After the phases separated the lower aqueous layer was transfered to a second separatory funnel and extracted a second time with another 50 ml portion of 2, 2, 4-trimethylpentane. Both organic layers were combined and the aqueous layer extracted a third time with 50 ml of 2, 2, 4-trimethylpentane. The combined (three) organic layers were then filtered through a 4" funnel plugged with glass wool which had been covered with sodium sulfate. The filtrate was collected in a Kunderna-Danish evaporator concentrator and the volume reduced to 5 ml by heating the apparatus in a 115°C oil bath.

Calibration plots for the individual PCB's were made by averaging the integrated areas of five injections of standard dilutions. The plots were linear for five orders of magnitude. The calibration plots for the Aroclors were made by averaging the peak height for a well resolved intense peak in each blend and were also linear for five orders of magnitude.

Instrumental operating parameters were as follows:

MODEL 5713 GC CONDITIONS

Column Temperature:	225°C
Injection Port:	On column unheated
EC Detector Temperature:	300° C
Attenuation:	As shown (does not apply if integrator recorder presentation is used)
Carrier:	Argon-methane 95-5 60 ml/min
Sample Introduced:	Hewlett-Packard 10 µl syringes, all injections 1.0 µl
Recorder:	Hewlett-Packard 7123A, 1 mv full scale, 0.25"/min chart speed

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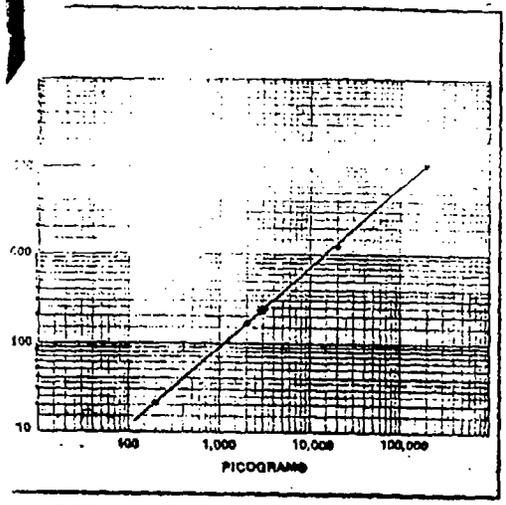
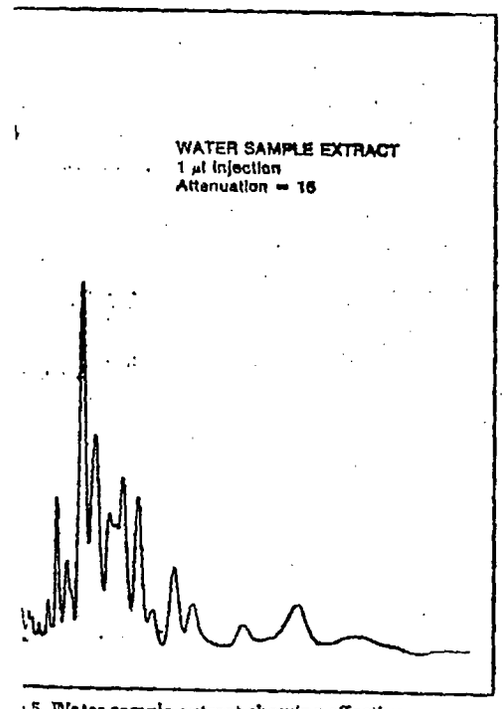


e 4. Calibration plot for Aroclor 1254.



5. Water sample extract showing effective isolation of Aroclor.

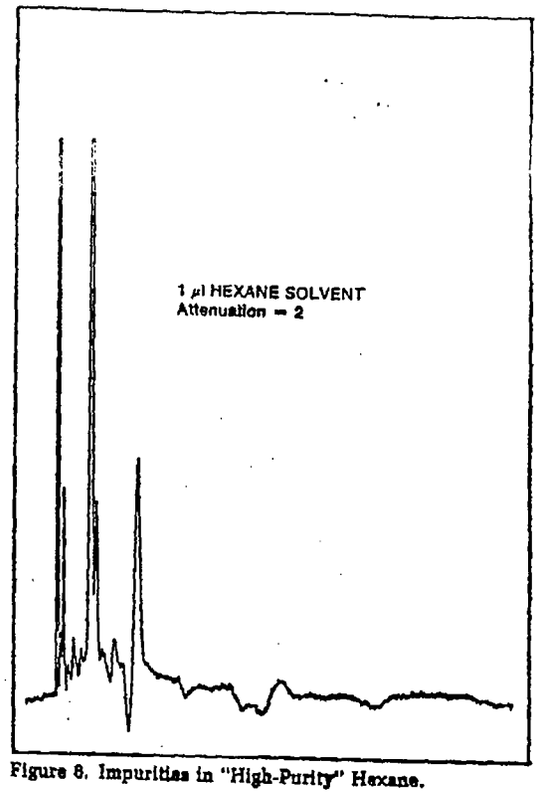


Figure 6. Impurities in "High-Purity" Hexane.

Calculations

The following calculation is used to calculate the concentration of PCB's in the original sample.

Concentration found from calibration plot = 2850 picograms
Volume injected = 1 μl of 5 ml concentrated extract
Original sample volume = 100 ml
1 ppm = 1 mg/1000 ml H_2O

$$\frac{2850 \times 10^{-12}\,g}{10^{-3}\,ml} \times \frac{5\,ml}{100\,ml} \times 1000\,ml/L \times \frac{10^{3}\,mg}{g} = 0.14\ ppm$$

References

[1] Work presented at the Tenth Annual Pesticide Residue Conference, Tallahassee, Florida, July 16, 1973 by J. Forester, EPA Gulf Breeze Laboratory, Sabine Island, Gulf Breeze, Florida.

[2] Zitho V., D. Hutzinger, and S. Safe, Bull. of Environ. Contam. and Tox., Vol. 8, (160) 1971.

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