CHROM. 3860

Interference from extraction thimbles in the gas chromatographic analysis of insecticides

In this laboratory, organochlorine insecticide residues in animal tissues are analyzed by means of a gas chromatograph equipped with an electron capture detector. The insecticide residues are extracted from the tissues by Soxhlet extractors using Whatman thimbles, 60 × 180 mm, and petroleum ether. It was found that materials, being coextracted from the thimbles, could interfere with the GC analysis of γ-BHC, heptachlor, aldrin, heptachlor epoxide, dieldrin, and p,p'-DDE. These interfering compounds remained after an acetonitrile–hexane partitioning and a Florisil column cleanup of the tissue extracts. A method was developed to remove the interfering compounds.

Procedure

The thimble was inverted in the Soxhlet and a pin hole was made in the bottom of the thimble to prevent air from being trapped. The thimble was preextracted for 7 h with methylene chloride and air dried overnight. To test the efficiency of the cleanup, the thimble was extracted with petroleum ether, the solvent was concentrated to 10 ml and 5 μl was injected into the gas chromatograph. This dilution is similar to that employed for residue analysis. The GC was equipped with an electron capture detector operated at 210° and 1 × 10^{-9} A, 6 ft. × $\frac{1}{4}$ in. glass column packed with 3% OV-17 on 100/120 mesh Gas Chrom Q at 190° and a N_2 flow rate of 100 ml/min.

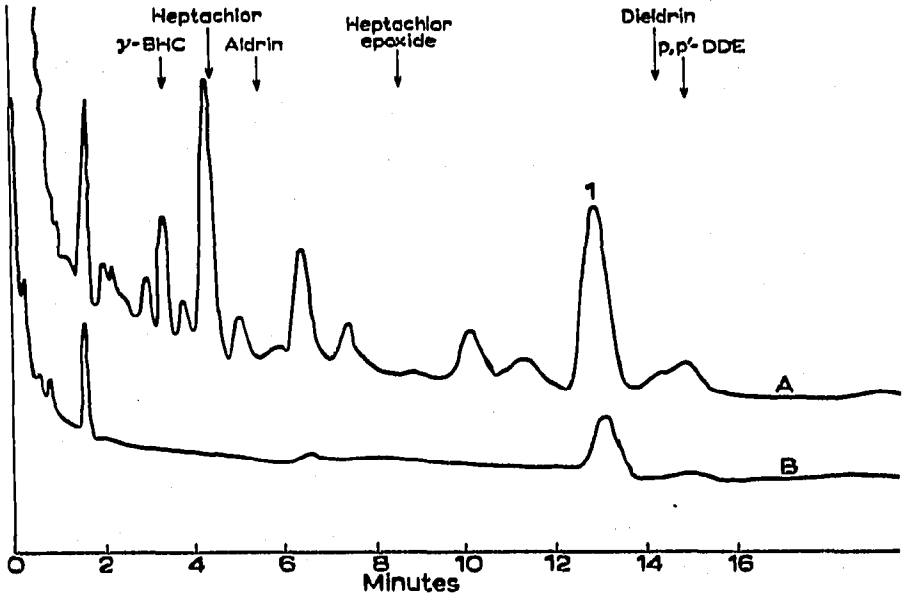

Fig. 1. Chromatogram (A) of a petroleum ether extract of one extraction thimble. Chromatogram (B) of a petroleum ether extract of a thimble that had been previously extracted for 7 h with methylene chloride.

Results

In Fig. 1 the petroleum ether extract from a thimble that had been preextracted with methylene chloride (chromatogram B) is compared with the extract from a thimble that had not been preextracted (chromatogram A). The arrows indicate the retention times of insecticides that could be misidentified due to the interfering materials. The response of unknown peak No. 1 is equivalent to 3 ng of dieldrin.

Thimbles also were preextracted with petroleum ether, water, methanol, ethyl ether, acetone, and acetonitrile; however, methylene chloride was the most efficient.

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Separation and estimation of collagen amino acids by programmed analysis

A rapid procedure for quantitative amino acid analysis of collagen or gelatin hydrolysates poses some problems. This protein contains 3- and 4-hydroxyprolines, usually in very different amounts, which have to be separated from each other and from aspartic acid. Another problem is the satisfactory resolution of glycine and alanine peaks, which is difficult due to unusually high quantities of glycine (one third of all amino acids). Furthermore, a good separation of only small amounts of hydroxylysine, histidine, methionine, tyrosine and occasionally methionine-sulfoxides and homoserine from neighboring peaks is required. The latter amino acid is a reaction product of methionine derived from cyanogen bromide treatment[1].

We wish to report results obtained with a rapid method for separating acidic and neutral amino acids of collagen hydrolysates. The equipment used was a "BC-200" analyzer, manufactured by Bio-Cal Instrument, Gräfelfing, Munich (Germany). This analyzer works on the basis of the approved principle described by SPACKMAN, STEIN AND MOORE[2]. It is designed for a programmed step-wise elution. A programming unit controlling five buffer systems and four different temperatures by timing devices allows the operator the choice of many separation programs that are variable over a wide range.

Our experience in separating collagen amino acids on columns of Aminex A-6 (Bio-Rad Laboratories, Richmond, Calif., U.S.A.) is summarized in Table I. A program (A) is given which takes into account all the parameters outlined in the introduction. The time required, including regeneration and equilibration of the column, is 175 min. A second program (B) is used in cases where an accurate analysis of all