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Quantitative determination of the γ-isomer of BHC* in technical products and formulations

Several papers on the gas chromatography of BHC isomers have been published and quantitative gas–liquid chromatography (GLC) methods have been described by ESSELBORN AND KREBS[1], KANAZAWA *et al.*[2], ODA *et al.*[3], FÜRST *et al.*[4], SUZUKI *et al.*[5], and DAVIS AND JOSEPH[6]. In this paper, a rapid method for the separation of the five main isomers of BHC and the quantitative determination of the γ-isomer by means of a stationary phase widely used in the chromatography of pesticides is described.

Experimental

Method. A 320 cm × 3 mm I.D. column of silanised Pyrex glass packed with 3 % methylphenyl silicone OV-17 on Gas-Chrom Q, 80–100 mesh, was used.

GLC was carried out with a Varian Aerograph Model 1520 gas chromatograph equipped with a flame ionisation detector. The chromatograph was connected to a Leeds and Northrup Model W recorder equipped with an integrator disc. The best operating conditions were: injection port temperature, 240°; column oven temperature, 215°; detector temperature, 240°; flow rates: nitrogen carrier gas, 35 ml/min; hydrogen 30 ml/min; air 400 ml/min; electrometer settings: range, 10; attenuation 32. With these conditions, a column efficiency of about 3200 theoretical plates, referred to the γ-isomer, was obtained.

Quantitative determination of the γ-isomer was carried out by means of the internal standard method. Heptachlor expoxide was found to be the most suitable standard because its peak did not interfere with those of BHC and possible impurities. Retention times relative to aldrin were: α-isomer, 0.50; γ-isomer, 0.64; β-isomer, 0.73; δ-isomer, 0.87; ε-isomer, 0.98; internal standard, 1.47 (Fig. 1).

For various γ-isomer concentrations within a certain range, the linearity of the area ratio of internal standard/γ-isomer was obtained by injecting 5-μl aliquots of solutions containing 600 mg of heptachlor epoxide and 400–800 mg/100 ml of γ-isomer. The plot of ratio values against γ-isomer concentrations was a straight line passing through the origin.

Materials. The internal standard solution was a solution of 3.0 % heptachlor epoxide in acetone. The reference standard solution consisted of 0.30 g of pure γ-isomer in 10.0 ml of internal standard solution, made up to 50 ml in a volumetric flask with carbon disulphide.

Determination. A BHC sample containing about 0.3 g of γ-isomer was weighed into a 50-ml volumetric flask, 10.0 ml of internal standard solution were added, the sample was dissolved and the flask was made up to the mark with carbon disulphide.

Alternately, 5-μl aliquots of this solution and 5 μl of reference standard solution were injected with a Hamilton syringe Model 701 NCH.

Areas were calculated with integrator disc and the γ-isomer content was obtained.

* Abbreviation: BHC = 1,2,3,4,5,6-hexachlorocyclohexane.

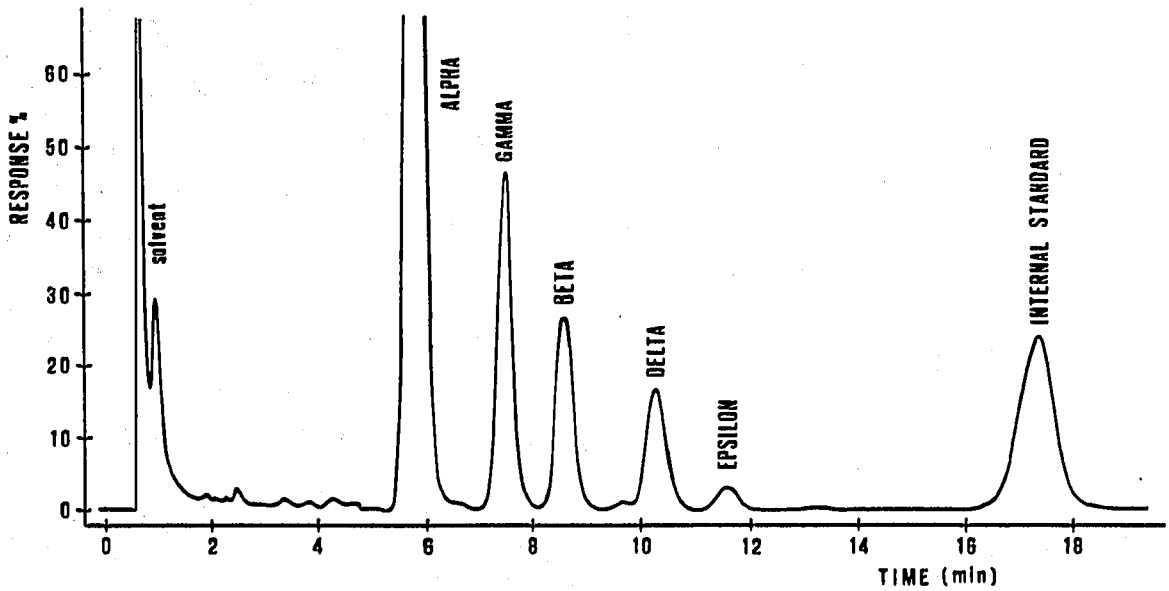

Fig. 1. Gas chromatogram of BHC. Column: OV-17 on 80–100 mesh Gas-Chrom Q, 320 cm × 3 mm I.D. glass, temperature 215°. Internal standard, heptachlor epoxide.

Discussion

By the above procedure, technical BHC samples containing 14–15 %, 46–48 % and 60 % of γ-isomer were analysed and the results were in good agreement with those obtained by partition chromatography. Emulsifiable formulations were determined similarly. Dusts and wettable powders were analysed by GLC, after extraction.

Carbon disulphide solutions were used owing to the poor response of the flame ionisation detector with this solvent; it allowed chromatograms to be obtained with negligible solvent tailing.

For rapid and simple performance, GLC methods can compare advantageously with the official analytical methods[7–9] based on partition chromatography on a silica column and IR spectrophotometric determination of an aliquot containing the γ-isomer.

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