GLC Separation of Heptachlor Epoxide Oxychlordane, α - and γ -Chlordane

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We found that the GLC columns generally used to identify and quantitate organochlorine pesticide residues, such as 3% OV 1, 2% SE 30 plus 6% QF 1, or 2% OV 1 plus 6% OV 210, do not satisfactorily separate all of these compounds: α-chlordane, γ-chlordane, oxychlordane, and heptachlor epoxide. Especially the latter two compounds are generally difficult to resolve in most GLC columns. Polen et al. (1) reported that a column containing OV 17 plus QF 1 at various ratios gives good resolution for oxychlordane and heptachlor epoxide, but the authors stated neither the ratio nor the concentrations of these coatings. We are describing the preparation and performance of a column containing 1.5% OV 17 plus 1.95% OV 210 which separates all four compounds and gives sufficiently sensitive response.

Experimental

Appropriate amounts of OV 17 and OV 210, dissolved in 150 ml of analytical grade ethyl acetate, were mixed with 25 g of Chromosorb W "HP", 80/100 mesh, in a ribbed, round-bottom flask. The slurry was slowly rotated at 70°C, and a stream of nitrogen blown into the flask, until the solvent had evaporated. The packing was then oven-dried for 12 hr at 150°C.

A glass column was silanized prior to packing with 5% dimethyldichlorosilane in toluene. The packing was then added in small increments and settled with a mechanical vibrator. The ends were plugged with silanized glass wool. The column was conditioned for 24 hr at 250°C and 100 ml/min nitrogen flow.

The operating conditions were as follows: gas chromatograph: Micro Tek MT 220 with Ni-63 electron capture detector; column: 1.5% OV 17 plus 1.95% OV 210 on Chromosorb W "HP", 80/100 mesh, 1/4" O.D., 6 ft; detector temperature: 295°C; column temperature: 195°C; inlet temperature: 220°C; nitrogen carrier gas: 48 psig, 100 ml/min without purge; detector potential: 13 1/2 volts; attenuation: 10^2 x 16; operation: on-column injection.

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Results and Discussion

Table I shows the retention times, relative to aldrin, of 11 organochlorine pesticides under the described conditions. Fig. 1 is a chromatogramme of all 11 compounds, Fig. 2 of oxychlordane, heptachlor epoxide, α-chlordane, and γ-chlordane. Since p,p'-DDT is eluted in 15 min, we have found this column to be valuable in routine screening for organochlorine residues and for confirmation of heptachlor epoxide and oxychlordane.

TABLE I

Retention Times of Some Common Organochlorine Pesticides Relative to Aldrin

Pesticide	ng injected	Relative Retention Time
Lindane	0.04	0.66
Heptachlor	0.04	0.81
Aldrin	0.04	1.00
Oxychlordane	0.08	1.44
Heptachlor Epoxide	0.04	1.62
γ-Chlordane	0.08	1.99
α-Chlordane	0.08	1.80
p,p'-DDE	0.08	2.43
Dieldrin	0.08	2.58
p,p'-DDD	0.16	3.90
p,p'-DDT	0.32	4.71

References

1. Polen, P. B. *et al.* Bull. Environ. Contam. Toxicol. 5:521 (1970).

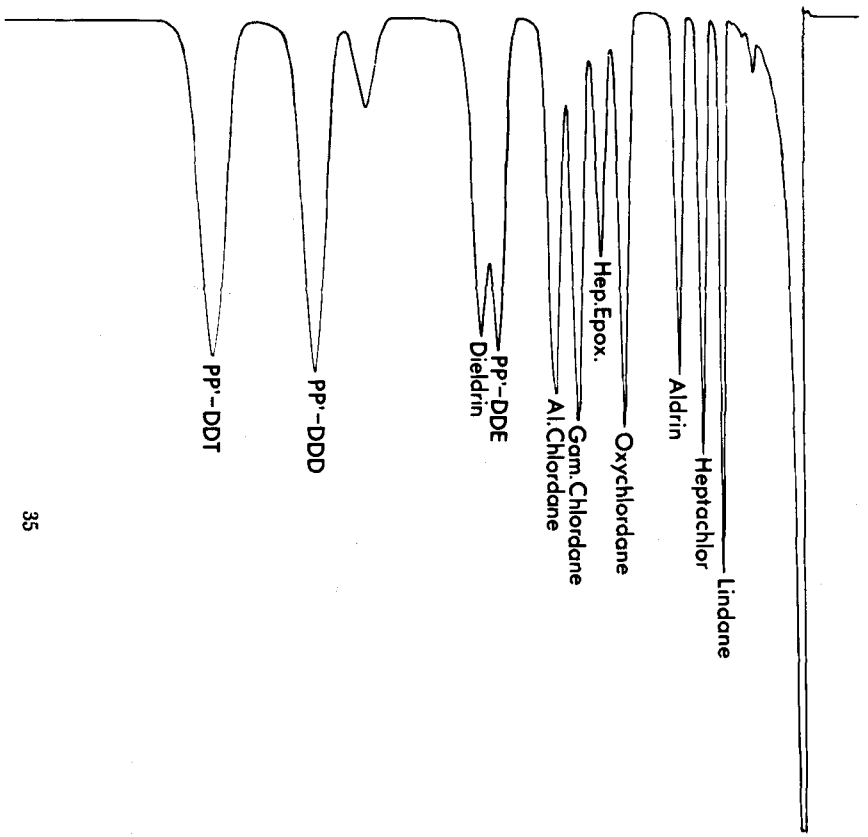

Figure 1: Chromatogramme showing satisfactory separation of 11 organochlorine pesticides.

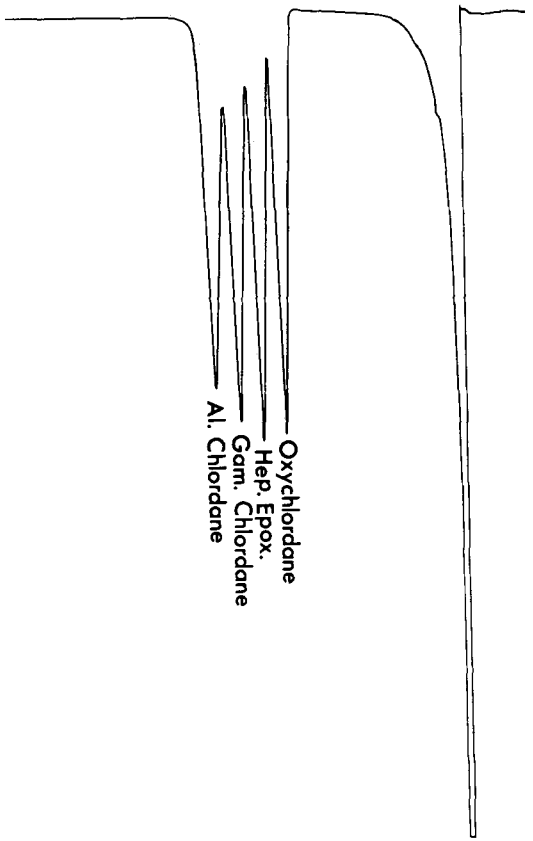

Figure 2: GLC separation of oxychlordane, heptachlor epoxide, α-, and γ-chlordane.