Simultaneous Quantitative Determination of Lindane and DDT by Gas Chromatography

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An argon ionization detector is utilized to determine lindane and DDT quantitatively in the same sample by employing heptachlor as an internal standard. The procedure is fast, accurate, and relatively easy to carry out.

HEPTACHLOR can be determined quantitatively using lindane as an internal standard (*2*). To determine lindane and DDT simultaneously, heptachlor is an ideal internal standard, as its retention time falls between those of lindane and DDT.

Apparatus and Reagents

Gas Chromatograph. Barber-Colman Model 10 equipped with a radium sulfate ionization detector and 5-mv. recorder.

Stationary Phase. General Electric SE-30 silicone gum (2% w./w.) and Dow Corning QF-1-0065 silicone fluid (3% w./w.) on Chromosorb W (80–100 mesh) packed in a borosilicate glass column (6 feet × $^1/_4$ inch, I.D.).

Syringe. A 10-μl. No. 701N Hamilton syringe was used to inject the samples.

Reagents. Benzene (analytical reagent grade) was used as a solvent for heptachlor (analytical reference grade, assaying 99.5%), lindane (technical grade, assaying 99.0%), and DDT (technical grade, assaying 90.0% *p,p'*-isomer).

Procedure

Because of its accuracy, the internal standard technique of Ray (*1*) was employed. Standard solutions of heptachlor, lindane, and DDT were prepared in the concentrations shown in Table I and chromatographed under the following conditions: column temperature, 190° C.; detector temperature, 220° C.; flash heater temperature, 275° C.; argon pressure, 25 p.s.i.; cell voltage, 1000; electrometer gain, 1 × 10^{-7}; sample size, approximately 1 μl. Under these conditions separation is very good (Figure 1).

Table I. Solution Concentrations and Ratio of Peak Heights of Internal Standard and Solutions of Lindane and DDT

(5 mg. of heptachlor per milliliter used)

Lindane, Mg./Ml.	DDT, Mg./Ml.	Heptachlor/ Lindane	Heptachlor/ DDT
1.0	7.5	4.615	2.074
		4.400	2.126
		4.291	2.452
	Av.	4.435	2.217
1.5	10.0	2.642	1.373
		2.643	1.423
		2.655	1.377
	Av.	2.647	1.391
2.0	12.5	1.800	1.039
		1.867	1.033
		1.891	1.040
	Av.	1.853	1.037
2.5	15.0	1.388	0.812
		1.384	0.828
		1.417	0.823
	Av.	1.394	0.821
3.0	17.5	1.104	0.718
		1.138	0.677
		1.139	0.677
	Av.	1.127	0.691
3.5	20.0	0.962	0.584
		0.937	0.566
		0.952	0.601
	Av.	0.950	0.584

Table II. Analysis of Greenfield Rose Dust, HO75

Sample	% Lindane (Theory = 1.00%)	% DDT (Theory = 5.00%)
1	1.05	5.04
2	0.97	4.83
3	0.98	5.03
4	0.99	5.25
5	0.85	4.93
6	1.09	4.98
7	0.97	4.91
8	0.97	4.91

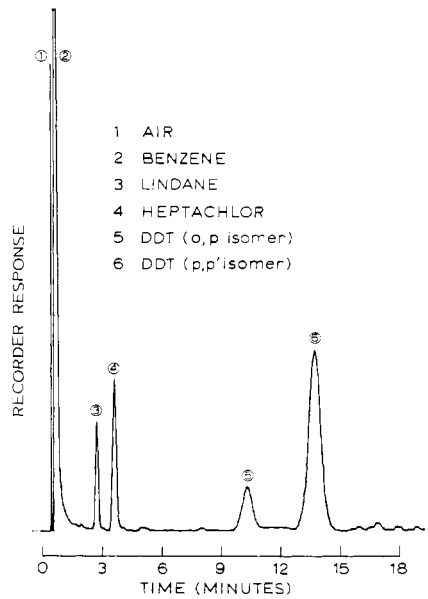

Figure 1. Separation of lindane heptachlor, and DDT

To prepare calibration curves, the log of the ratio of the heptachlor peak height to the lindane and DDT peak heights is plotted against the log of the concentrations (Table I). The resulting curves are straight lines; they may shift slightly from day to day because of changes in operating parameters but are always parallel. A standard curve must be established each day to ensure good quantitation.

To assay Greenfield rose dust, HO75, a 7.0-gram sample is weighed into a Soxhlet extraction thimble and refluxed for 2 hours with chloroform. The extract is then evaporated to dryness with the aid of a rotary evaporator and dissolved in 30.0 ml. of benzene containing 150.0 mg. of heptachlor. Approximately 1 μl. of this solution is injected into the gas chromatograph. After measuring the peak heights and calculating the ratio of heptachlor to lindane and heptachlor to DDT, the concentrations of lindane and DDT are read from the calibration curves. The per cent lindane and DDT is then obtained using the following formulas:

$$\% \text{ lindane} = \text{mg./ml.} \times \frac{30}{7} \times \frac{100}{1000}$$

$$\% \text{ DDT} = \text{mg./ml.} \times \frac{30}{7} \times \frac{100}{1000}$$

Results

Table II shows the results of the analysis of eight lots of rose dust, by the method described.

Precision

To estimate the precision of the method, six samples from a lot of rose dust were independently assayed, giving a relative standard deviation of ±3.62% for lindane and ±1.83% for DDT.

Literature Cited

(1) Ray, N. H., *J. Appl. Chem. London* **4**, 21 (1954).
(2) Wesselman, H. J., Koons, J. R., J. AGR. FOOD CHEM. **11**, 173 (1963).

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