If the sample size is in the milligram region, then the material must be vacuum transferred. If the sample size is in the gram region, a Teflon tube can be inserted down the gas outlet tube of the collector, and the sample removed with a syringe or bulb. The collector can then be rinsed clean with appropriate solvents, dried, and thus be ready for another collection.

Trapping yield data have been obtained with ethyl caproate and limonene. In the 1-gram range, yields of 90–95 % were obtained with ice as the coolant. Yields slightly higher than 95 % were obtained with dry ice as the coolant.

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The isolation of DDT, parathion and lindane from biological fatty materials by liquid–liquid partition chromatography

The isolation of organic insecticides from biological fatty matter constitutes a fundamental step for their ultimate quantitative estimation. While studying the applicability of some of the methods described in the literature, the work of JONES AND RIDDICK[1] and that of BURCKFIELD AND STORRS[2] stimulated us to develop liquid–liquid partition chromatographic columns for the isolation of the above-mentioned insecticides.

JONES AND RIDDICK isolated several insecticides by partitioning them between acetonitrile and *n*-hexane. BURCKFIELD AND STORRS replaced acetonitrile by N,N-dimethyl-formamide. By using these solvent pairs in chromatographic columns we succeeded in recovering milligram quantities of DDT, parathion and lindane for more than 90 % out of their solutions in concentrated insect extractives or in peanut oil. One of the advantages of these columns is the possibility of using them more than once.

Application of this method to microgram quantities, often occurring in biological material, could not at the moment be checked owing to the lack of a detection method of adequate sensitivity.

Methods for isolating the insecticides

(1) *Isolation of DDT.* Celite 545 (JOHNS MANVILLE), from which the fines have been slibbed off, is washed with concentrated hydrochloric acid and then with distilled water and dried. Of this material 5 g are mixed thoroughly with 2 g of dimethyl-

formamide by shaking in a stoppered flask until a homogeneous slightly damp powder is obtained. *n*-Hexane saturated with dimethylformamide is added until a smooth slurry is formed. Then the slurry is brought in small portions into a chromatographic tube (length 20 cm, inner diameter 1 cm) which is plugged with glasswool at the constricted end and which contains about 5 ml *n*-hexane saturated with dimethylformamide.

The contents of the tube are packed down slightly with a glass tamping rod after each addition. A loose packing of the column is of crucial importance for obtaining a good separation. After preparation of the column the excess of liquid is drained off until a small volume remains above the kieselguhr.

The concentrated insect extractives are dissolved in 5 ml of *n*-hexane saturated with dimethylformamide. This solution is poured quantitatively into the column, taking care not to disturb the surface of the kieselguhr, whereupon elution is started.

The elution rate should not be more than 5 ml in about 2 min. The fatty materials are eluted with the first 20 ml of hexane. Then about 10 ml almost pure solvent are collected, after which the insecticide appears with the following 20 ml of eluents.

(2) *Isolation of lindane.* The column used for the isolation of lindane is prepared, in the same way as described for DDT, from 3 g of kieselguhr and 1.2 g of dimethylformamide. A lower boiling solvent, *viz.*, *n*-pentane may be used instead of *n*-hexane in order to minimize losses during evaporation of the solvent. The lindane appears in the eluate when 30 ml of the solvent have percolated through the column.

(3) *Isolation of parathion.* The column employed for the isolation of parathion differs from those used for DDT and lindane. It consists of 3 g of cellulose powder (Whatman No. 1)[3], and 1.2 g of acetonitrile as the stationary phase; *n*-hexane saturated with acetonitrile is used as the mobile phase. Packing and elution are carried out as described above.

The retention volume of parathion is also 30 ml.

Recovery tests

These were carried out by evaporating the solvent of the eluate fractions, after which the residues obtained were weighed and then analyzed quantitatively by means of gas–liquid chromatography. A J. H. BECKER Gaschromatograph (Delft, the Netherlands) was used with katharometer detection. The column employed was a coiled copper tube (length 50 cm, inner diameter 0.4 cm) filled with 1.6 g of Embacel (May and Baker) impregnated with Apiezon L (10:1 weight ratio).

Hydrogen was used as the carrier gas. The working conditions were as follows:

	Parathion	*Lindane*	*DDT*
Column temperature	220°	220°	230°
Gas flow rate	60 ml/min	60 ml/min	120 ml/min
Retention volume	174 ml	123 ml	744 ml

Under these conditions the smallest amount of the insecticides that could be detected was about 50 γ. Aiming at a greater sensitivity we applied the combustion method of BEERTHUIS *et al.*[4] with helium as the carrier gas.

In this method the components leaving the G.L.C. column are burned over copper oxide at 1000°, after which the carbon dioxide formed is detected by the katharometer. In this way the smallest detectable amount was lowered to 2 γ.

This sensitivity, however, does not yet allow the determination of the minute amounts of the insecticides often present in biological material. This analysis requires application of ionisation detectors as developed by LOVELOCK AND MCWILLIAM[5,6]. Then quantities as small as 0.01 γ can be estimated.

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Bestimmung des Methylbromids und Äthylbromids nebeneinander

Äthylbromid wird weitgehend in der organischen Industrie verwendet. Es wurde von uns bei einer Gelegenheit verlangt es zu prüfen, ob und wieviel Methylbromid darin enthalten ist.

Obwohl in der Literatur viele und die verschiedensten Methoden zur Äthylbromid- bzw. Methylbromidbestimmung vorliegen[1–12], fanden wir keine Beschreibung einer Methode, welche die Bestimmung dieser Verbindungen nebeneinander behandelt. Inbetracht der physikalischen Eigenschaften dieser Verbindungen (Tabelle I) nahmen

TABELLE I

Formel	*Molgewicht*	*Schmelzpunkt °C*	*Siedepunkt °C*	*Dampfdruck Torr (20°C)*
CH_3Br	94.95	— 93.66	+ 3.56	1250.0
CH_3CH_2Br	108.98	—119	+38.0	386.0

wir es vor, ihre Trennung bzw. Bestimmung mittels Gaschromatographie zu versuchen.