

are indicated. To obtain this separation, the solvent system proportions were altered in the direction of greater polarity (chloroform-acetic acid-water (10:79:11, v/v)); four developments were conducted.

Research Laboratories, Continental Baking Co.,
Rye, N.Y. (U.S.A.)

V. A. DE STEFANIS
J. G. PONTE, Jr.

- 1 D. W. VOMHOFF AND T. C. TUCKER, *J. Chromatog.*, 17 (1965) 300.
- 2 YU. S. OVODOV, E. V. EVTUSHENKO, V. E. VASKOVSKY, R. G. OVODOVA AND T. F. SOLOV'EVA, *J. Chromatog.*, 26 (1967) III.
- 3 J. P. MARAIS, *J. Chromatog.*, 27 (1967) 321.
- 4 E. STAHL AND U. KALTENBACH, *J. Chromatog.*, 5 (1961) 351.
- 5 C. E. WEILL AND P. HANKE, *Anal. Chem.*, 34 (1962) 1736.
- 6 V. PREY, H. SCHERZ AND E. BANCHER, *Mikrochim. Acta*, (1963) 567.
- 7 G. W. HAY, B. A. LEWIS AND F. SMITH, *J. Chromatog.*, 11 (1963) 479.
- 8 H. JACIN AND A. R. MISHKIN, *J. Chromatog.*, 18 (1965) 170.
- 9 P. G. PIFFERI, *Anal. Chem.*, 37 (1965) 925.
- 10 M. L. WOLFROM, R. M. DE LEDERKREMER AND G. SCHWAB, *J. Chromatog.*, 22 (1966) 474.
- 11 E. J. SHELLARD AND G. H. JOLLIFFE, *J. Chromatog.*, 24 (1966) 76.
- 12 E. GAROFALO, *Minerva Pediat.*, 18 (1966) 3.
- 13 A. LOMBARD, *J. Chromatog.*, 26 (1967) 283.
- 14 A. AFFONSO, *J. Chromatog.*, 27 (1967) 324.
- 15 I. SMITH, *Chromatographic and Electrophoretic Techniques, Vol. I*, Heinemann, London, 1962, p. 251.
- 16 C. N. HUBER, H. SCOBELL AND HAN TAI, *Cereal Chem.*, 43 (1966) 342.

Received November 30th, 1967

J. Chromatog., 34 (1968) 116-120

CHROM. 3305

A rapid method for the determination of *p,p'*-DDT to *p,p'*-DDE ratios in fish

We wish to describe a more rapid method of analyzing for the presence of DDT in fish than the commonly used method of MILLS, ONLEY AND GAITHER¹. The following procedure was developed as the result of a need to routinely and rapidly determine the ratio of *p,p'*-DDT to its metabolites and although the recoveries of the pesticides from the tissues were not as high as those obtained using the above method, it was found to be satisfactory for obtaining the ratios of *p,p'*-DDT to *p,p'*-DDE.

Tissue samples ranging from 0.005 to 0.1 g were digested in 2-4 ml of formic acid and maintained at 60° for 1.5-2 h followed by extraction four times with hexane. The combined extracts were evaporated to dryness and the residue was washed with three 2 ml portions of acetonitrile. A small amount of alumina (20 mg, 80-200 mesh) was added and the sample was centrifuged. After evaporating the supernatant to dryness, the residue was dissolved in a small amount of ethyl acetate and spotted on a plate coated with aluminum oxide using the procedure described by KOVACS².

Since our samples contained ¹⁴C-labeled DDT and its corresponding metabolites, DDE and DDD, a radio chromatogram scanner was used to detect the amounts

J. Chromatog., 34 (1968) 120-121

of the radioactive compounds. Although the use of heptane as an elution solvent² for the separation of *p,p'*-DDT from *p,p'*-DDE was good enough for most tissues, their separation in some tissues was not sufficient to allow the recorder pen of the scanner to return to the base line between the two compounds. However, substitution of low-boiling (30–60°) petroleum ether for heptane provided better separation as evidenced by Table I.

TABLE I

R_{DDD} VALUES OF *p,p'*-DDT AND *p,p'*-DDE IN DIFFERENT ELUENTS^a

<i>Tissue</i>	<i>Pesticide</i>	<i>Heptane</i>	<i>Petroleum ether</i>
Skin	DDT	1.9	1.9
	DDE	2.6	2.9
Muscle	DDT	2.0	2.0
	DDE	3.1	3.3
Testes	DDT	1.7	1.7
	DDE	2.2	2.4
Kidney	DDT	1.2	1.3
	DDE	1.4	1.8
Ovary	DDT	1.7	1.7
	DDE	2.2	2.2

^a Eluted for 16.5 cm, other conditions remaining constant for both eluents.

Although the difference in the R_{DDD} values is small, it was sufficient to allow the pen to return to the baseline, thereby giving two distinct peaks with more accurately measurable areas than those in which an overlap occurs.

*Southeast Water Laboratory,
Athens, Ga. 3069 (U.S.A.)*

DORIS L. FORT

¹ P. A. MILLS, J. H. ONLEY AND R. A. GAITHER, *J. Assoc. Offic. Agr. Chemists*, 46 (1963) 186.
² M. F. KOVACS, *J. Assoc. Offic. Agr. Chemists*, 46 (1963) 884.

Received November 10th, 1967

J. Chromatog., 34 (1968) 120–121