CHROM. 3968

Determination and separation of some uracil herbicides by thin-layer chromatography

Substituted uracil herbicides were first introduced by the Du Pont de Nemours Co. in 1962. Three more recent members of this group are 3-butyl-6-methyluracil, bromacil (5-bromo-3-*sec.*-butyl-6-methyluracil) and terbacil (5-chloro-3-*tert.*-butyl-6-methyluracil). The latter two are particularly useful in the control of annual and certain perennial weeds in orchards, alfalfa and sugar cane. Methods for the determination of these herbicides involve primarily gas chromatography[1–3] and a thin-layer method for the determination of bromacil[4]. It is the purpose of this paper to report a convenient two-dimensional thin-layer procedure for the determination and separation of the three herbicides. This method complements gas chromatographic procedures and is particularly useful if proof of identification of results obtained by gas chromatography is required.

Experimental

Samples of 3-butyl-6-methyluracil, bromacil and terbacil were supplied by the Du Pont de Nemours Co. Thin-layer sheets (Eastman K301R2) were used and the solvent systems were: (1) benzene–hexane–acetone (5:2:1) and (2) chloroform–acetone (9:1). The chromogenic reagent used was Brilliant Green (0.5 % sol. in acetone) followed by bromination, which produced pink coloured spots.

Results and discussion

The results of the two-dimensional thin-layer separation are shown in Fig. 1 and R_F values for the individual compounds are given in Table I. The spots were clearly visible after application of the Brilliant Green spray followed by treatment with bromine vapour. The limit of detectability for this method is between 0.5 μg and 1.0 μg of each herbicide and it was found that the two-dimensional technique offers greater accuracy and better separation than development in one direction only. Silver

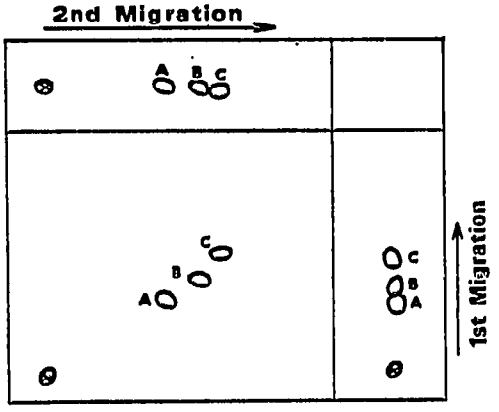

Fig. 1. Two-dimensional thin-layer chromatogram of 3-butyl-6-methyluracil (A), 5-bromo-3-*sec.*-butyl-6-methyluracil (bromacil) (B), and 5-chloro-3-*tert.*-butyl-6-methyluracil (terbacil) (C). Solvent systems: (1) benzene–hexane–acetone (5:2:1); (2) chloroform–acetone (9:1).

TABLE I

APPROXIMATE R_F VALUES

Compounds	*Solvent No. 1*	*Solvent No. 2*
3-Butyl-6-methyluracil (A)	0.21	0.30
5-Bromo-3-*sec.*-butyl-6-methyluracil (bromacil) (B)	0.25	0.38
5-Chloro-3-*tert.*-butyl-6-methyluracil (terbacil) (C)	0.34	0.42

nitrate reagent can also be used to make spots of bromacil[4] and terbacil visible. However, it cannot be used for the detection of 3-butyl-6-methyluracil.

The applicability of the method described was tested by analysing known amounts of terbacil, bromacil and 3-butyl-6-methyluracil that had been added to soil samples. In a typical run, 100 μg of each of the three herbicides was added to 100 g of soil. The soil was then extracted with 1.5 % NaOH solution as prescribed by the method of JOLLIFFE *et al.*[3], and the final acetone solution evaporated to 1.0 ml in a Kuderna–Danish concentrator. Of this solution 10 μl was then applied to the thin-layer sheet. After the sheet had been developed for the required distance the Brilliant Green spray was applied followed by bromination, which produced well defined pink spots. Based on a 100 g sample and a final solution volume of 10 ml this method is sensitive in soil to 0.1 p.p.m.

Canada Department of Agriculture, Research Station, Harrow, Ont. (Canada)

F. G. VON STRYK
G. F. ZAJACZ

1 H. L. PEASE, *J. Agr. Food Chem.*, 16 (1968) 54.
2 W. H. GUTEMANN AND D. J. LISK, *J. Assoc. Offic. Anal. Chemists*, 51 (1968) 688.
3 V. A. JOLLIFFE, B. A. DAY, L. S. JORDAN AND J. D. MANN, *J. Agr. Food Chem.*, 15 (1967) 174.
4 D. J. HAMILTON, *J. Agr. Food Chem.*, 16 (1968) 142.

Received January 17th, 1969