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Determination of bromacil by gas chromatography*

Available gas chromatographic procedures for the analysis of residues of the herbicide bromacil (5-bromo-3-*sec.*-butyl-6-methyluracil) imply the need for a microcoulometric titrating system coupled with a temperature-programming module[1,2] and/or short (13–24 in. long) gas chromatography columns[1,3] with the optional use of an electron-capture detector. Without doubt, some laboratories are not equipped with the microcoulometric system, and under ordinary usage conditions the average length of a gas chromatography column varies from 4 ft. to 6 ft. with a column diameter in the range of $\frac{1}{8}$ to $\frac{1}{4}$ in.

Bromacil residues in plant material, soil, and water can be determined with a conventional gas chromatography column and an ordinary type of gas chromatograph equipped with an electron-capture detector as discussed below.

Materials and methods

Gas chromatograph. Aerograph, Model 204-B, tritium foil electron-capture detector (250 mC); column temperature 200°, injection port temperature 200°, detector temperature 200°; range 10, attenuation 2; nitrogen carrier gas flow rate 30 ml/min.

Gas chromatography column. Gas-Chrom Q, 80–100 mesh, coated with a mixture of 3% QF-1 fluorosilicone (FS 1265), 10,000 cS, and 2% DC-200 silicone, 12,500 cS, packed in a $\frac{1}{8}$ in. O.D. × 5 ft. spiral borosilicate glass column; the column should be conditioned at 225° for at least a 24-hour period before it is used.

Bromacil, recrystallized, was furnished through the courtesy of E. I. du Pont de Nemours and Co., Inc., Wilmington, Dela. A solution of bromacil in ethyl acetate (Mallinckrodt, Nanograde), 0.2 ng/μl, gave a linear detector response within the examined range of 0.2–1.2 ng (see Fig. 1).

Water samples containing suspended soil particles, soil, and rice plant seedlings were used in this study. The preparation of the samples included the extraction and

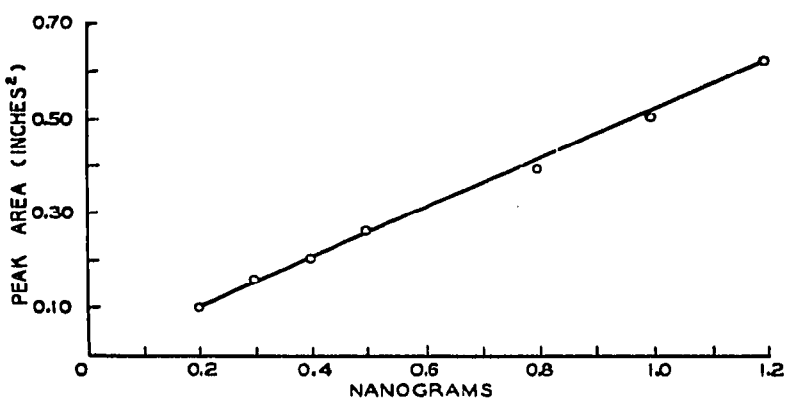

Fig. 1. Linearity curve for bromacil.

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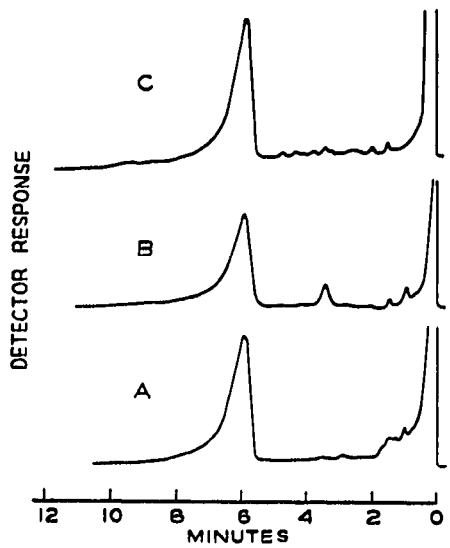

Fig. 2. Analysis of bromacil residues from 100-g samples of soil and 125-ml samples of water. Curve A: bromacil standard, 1 ng. Curve B: water sample, 1 μl of 10 ml ethyl acetate solution (10 ml ethyl acetate solution equivalent to 125 ml original water sample); water contained 0.06 p.p.m. bromacil. Curve C: soil sample, 3 μl of 10 ml ethyl acetate solution (10 ml ethyl acetate solution equivalent to 100 g soil sample); soil contained 0.04 p.p.m. bromacil.

cleanup procedures of PEASE[1,2] and JOLLIFFE *et al.*[3]; the final cleanup step for all samples was accomplished on a Florisil column instead of by the charcoal cleanup procedure suggested by JOLLIFFE *et al.*[3]. Because of the presence of soil particles in the water samples, sufficient sodium hydroxide (previously dissolved in a minimum amount of distilled water) was added to each water sample to make a 1% sodium hydroxide concentration, to insure efficient extraction of the bromacil residue from the samples[3]. The final extracts were made to suitable volumes with ethyl acetate (Mallinckrodt, Nanograde) for analysis by gas chromatography.

Representative gas chromatography curves in Fig. 2 illustrate the results of the analysis of bromacil residues from 100-g samples of soil and 125-ml samples of water. Samples fortified with bromacil gave recovery values of 85–89%. The limit of detectability, under the conditions used, was 0.005 p.p.m.

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