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Note

Determination of residues of dalapon in soil by gas chromatography of the 1-butyl ester

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Dalapon (2,2-dichloropropionic acid) is used for the control of weed grasses and for total weed control in non-crop situations¹. Several methods for dalapon residue analysis are available. These methods generally require protein precipitation and numerous solvent partitions before either colorimetric² or gas chromatographic^{3,4} measurement. Getzender³ estimates the free acid while Frank and Demint⁴ prepare the methyl ester for electron capture gas chromatography. In the work reported here the 1-butyl ester is employed. Under the chromatographic conditions used, this ester, unlike the acid or the methyl ester, elutes with a retention time that does not coincide with that of any of the co-extractants from the soils studied, thus removing the necessity for elaborate "clean-up" procedures.

MATERIALS AND METHODS

Soil

Soils from two locations were obtained. Table I gives some details of the composition of these soils. Both soils were air dried and passed through a 3-mm sieve prior to fortification with dalapon.

TABLE I
SOME PROPERTIES OF THE TWO SOILS EXTRACTED

	1	2
% organic carbon	1.6	4.1
pH	7.0	5.1
% clay	16	16
% silt	11	16
% sand	73	68
Field capacity (% moisture)	16.6	27.0

Soil fortification

The soils were fortified at 1.0, 0.1 and 0.05 ppm. Aqueous solutions of the sodium salt of dalapon were prepared so that, when sufficient solution was added to

the dry soils to achieve field capacity, the concentration was correct. All samples were prepared in triplicate at all levels and allowed to stand for 24 h before extraction to ensure homogeneity.

Extraction

A methanolic solution of 0.1 *M* sodium hydroxide was used for the extraction. 10 g of soil were shaken with 50 ml of extractant for 1 h on a wrist-action shaker. The extract was allowed to settle for 10 min before filtering through a Whatman No. 42 paper.

Esterification

The method used was that of McKone and Hance⁵ for the esterification of 2,4,5-trichlorophenoxyacetic acid. A 5-ml aliquot of the filtrate contained in a test-tube was evaporated and the residue re-dissolved in 1 ml of 1-butanol, three drops of concentrated sulphuric acid were then added and the tube placed in a boiling water-bath for 30 min. After cooling, 20 ml of water and 5 ml of 2,2,4-trimethylpentane were added. The ester partitioned into the organic phase and 5- μ l aliquots of this layer were injected into the chromatograph.

Chromatography

A Pye 104 gas chromatograph fitted with a ⁶³Ni electron capture detector and a 1.5 m \times 4 mm I.D. glass column was used for estimation of the ester. The conditions employed were as follows: column packing, 2% OV-17 on Chromosorb WHP 80-100 mesh; carrier gas, nitrogen, flow-rate, 40 ml/min; temperatures, column 100°, injector 250°, detector 360°; attenuation, 20 \times 10²; pulse time, 150 μ sec; chart speed, 300 mm/h; injection volume, 5 μ l.

Under these conditions the dalapon butyl ester elutes with a retention time of 1 min 35 sec. Standards were prepared from the sodium salt of dalapon and taken through the butylation procedure. A graph of log peak area vs. log ng of herbicide was linear for standards ranging from 0.05 ng/5 μ l to 0.5 ng/5 μ l.

RESULTS AND DISCUSSION

Table II shows that good recoveries were achieved with both soils at all three levels of fortification. The recorded response for the lowest level (0.05 ppm) was

TABLE II

RECOVERY OF 2,2-DICHLOROPROPIONIC ACID FROM FORTIFIED SOILS

Overall mean recovery: 96.3 \pm 0.65 %.

<i>Soil</i>	<i>Fortification (ppm)</i>	<i>Mean recovery (%)</i>	<i>S.E. of mean (%)</i>
1	1.0	97.6	± 0.67
	0.1	96.0	± 1.41
	0.05	97.4	± 0.59
2	1.0	95.3	± 0.51
	0.1	96.4	± 1.88
	0.05	95.2	± 0.97

approximately twice the background signal and this was taken to be the limit of detection for practical purposes. With some soils it may be possible to obtain a lower limit of detection by using a larger soil sample or taking a bigger aliquot of filtrate for butylation but for the two soils studied here both of these alternatives resulted in an unsatisfactory background response. It was necessary to use standards prepared by the butylation procedure since the yield of dalapon butyl ester, though reproducible, was only 80%.

It is the author's opinion that the results obtained together with the ease and speed of performance make this method more applicable to routine analysis than previous methods.

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