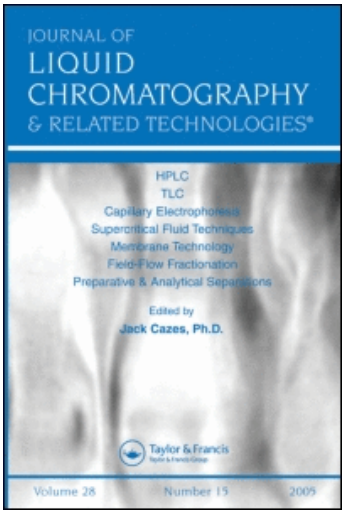

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Determination of Dimethoate in Human Blood by Impregnated Silica Plates and Gas Chromatography

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DETERMINATION OF DIMETHOATE IN HUMAN BLOOD BY IMPREGNATED SILICA PLATES AND GAS CHROMATOGRAPHY

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ABSTRACT

A method for the determination of Dimethoate in human blood is presented here. After extraction with acetone-hexane (1+9 V/V) dimethoate is separated on p-cresol impregnated thin silica plates as well as by Gas Chromatography using nitrogen phosphorous detector. The recovery of dimethoate added to human blood was quite satisfactory.

INTRODUCTION

Dimethoate (0,0-dimethyl-S-(N-methyl carbomoyl methyl) phosphorodithioate is a organophosphorous and systemic insecticide effective against a broad range of insects on a wide range of crops. It is a polar compound and stable in aqueous media at acidic or neutral pH [1].

A number of methods for the simutaneous determination of organophosphorous pesticides in environmental samples or food stuffs have been proposed by many worker in the past [2-4]. However, these methods often give poor results for dimethoate.

The use of impregnants for improving TLC separation of organophosphorous pesticides has recently been made [5]. In this paper authors have separated these pesticides on p-cresol as well as on zinc acelate impregnated silica plates and found that p-cresol impregnated plate gave the best results. In the present paper the author separated out dimethoate from human blood on p-cresol impregnated silica plates and then quantitatively confirmed by Gas Chromatography. This method is well suited for application in routine forensic and clinical investigation owing to its ease and rapidity.

EXPERIMENTAL

Materials and Methods

Dimethoate (99.5%) was a gift from Rallis India Ltd., Bangalore. All chemicals used here were analytical reagent grade. Fresh heparinized blood from healthy volunteeras was used as sample. Dimethoate was used as stock solution at 1 mg/ml in acetone for TLC. Stock solution was diluted 1:100 with water for Gas Chrmatography before use.

For TLC glass plates (20x20 cm) (thickness 0.5 mm) were coated with a mixture of silica gel(50g) and 0.5% p-cresol (W/V) in water (100ml) and then activated to $60^{\circ}\pm1^{\circ}C$ for 24 h.

A packard model 433 Gas Chromatography with a nitrogen phosphorous detector was used. The column was a 25m fused silica capillary column coated with 5% phenyl methyl silica (SE-54). Temperature programming : 0.7 min at $75^{\circ}C$ followed by $8^{\circ}C$/min to $225^{\circ}C$ and $20^{\circ}C$/min to $275^{\circ}C$.

Extraction

Replicate 2 ml blood samples or control blood samples spiked with a range of standard quantities of Dimethoate was mixed throughly and left at

$4^{o}C$ for 4 hrs. to ensure equilibration. Then this sample was transferred in a centrifuge tube, 10 ml of water and 30 ml of acetone-hexane (1+9V/V) were added. The tube was stoppered, Shaken vigorously for 5 min and the two phases were separated by centrifugation at 3000 rev min^{-1} for 10 min. The upper phase was removed and the lower phase was re-extracted with 30 ml of acetone-hexane (1+9V/V). The combined extract was washed once with 30 ml of saturated solution of sodium sulphate for 30 min by vigarous shaking and then the aqueous phase was discarded. Now the extract (in 500 ml conical flask) was placed in a water bath at 35-$40^{o}C$ and evoporate the solvent to dryness, using rotary evaporator. Dissolve the residue in 1.0 ml of acetone.

TLC (Thin Layer Chromatography)

The dissolved residue (1.0 ml acetone) spotted by a micropipette on the activated impregnated silica plates along with the plain silica plates. The spots were allowed to airdry and then subjected to development in a solvent-system n-hexane-xylene-ethyl acetate-acetone (50:15:5:18). The plates were developed to a length of 10 cm. Then exposed in iodine vapours. Dark yellow spots appeared on plates.

Gas Chromatography

A 100 ul aliquot of the acetone extract was injected into the gas chromatography and peak areas were determined from height and width at half-height. Samples peak area was compared to a working curve determined from a series of standard solution of dimethoate.

RESULT AND DISCUSSION

The result obtained on 0.5% p-cresol impregnated plate are shown in (Table 1). The hR_f values from the table 1 shows that dimethoate extracted

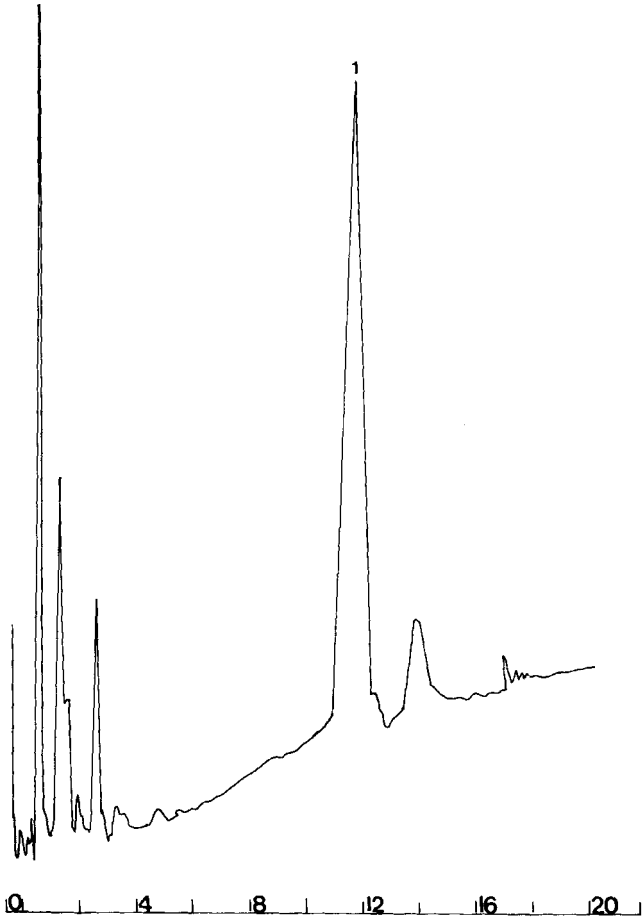

FIGURE 1

Table 1 : hR_f VALUES ON SILICA GEL IMPREGNATED WITH p-CRESOL

Solvent System - n-hexane - xylene - ethyl - acelate - acetone (50:15:5:18)				
Pesticide	on plain silica plate	hR_f on 0.5% W/V p-cresol impregnated plates		
		Std. Dimethoate (Sdn)	Dimethoate from Rat blood	Detection limit (ug)
Dimethoate	54 st	51	50	0.5

on plain silica plate - 10cm run in 35 mints.
on p-cresol impregnated plate - 10cm run in 20 mints.

from blood got suitably separated on p-cresol impregnated silica plates. No tailing was observed and the spots become compact whereas on plain silica plate tailing was observed and the separation was not so clear. The detection limit was 0.5 ug.

The results obtained by gas chromatography are shown in (fig. 1.). Dimethoate extracted from blood had retention time of 11.5 min. Analysis of quadruplicate samples spiked with dimethoate over the range 5-400 ng/ml gave a coefficient of variation of 5.5%, being essentially constant over the whole concentration range. Recovery of dimethoate which was also independent of concentration was 75.6±0.91. s.d.(n=6).

REFERENCES

1. Worthing, C.R. (Ed.). The pesticide manual (The British Crop protection Council, Croydom, 1983), 7th ed., p. 488.

2. Ault, J.A., Schofield, C.M., Johnson, L.D. and Waltz, R.H., J.Agric. Food Chem. 27, 825, 1979.

3. Ambrus, A., Hargitai, E., Karoly, G., Fulop, A. and Lantos, J., J.Assoc. off Anal. Chem. 64, 743, 1981.

4. Lebel, G.L., Williams, D.T., Griffith. and Benoit, F.M., J.Assoc. off. Anal. Chem. 62, 241, 1979.

5. Srivastava, S.P., Reena, Analytical letters. 15(A1), 39-46, 1981.