Gas Chromatographic Determination of Zinophos Residue in Soil[1]

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Although a spectrophotofluorometric method of analysis for Zinophos (O,O-diethyl O,2-pyrazinyl phosphorothioate) is already available(3), it seemed probable that a less complex procedure could be developed, capitalizing on the great selectivity for phosphorus compounds of a gas chromatograph equipped with a sodium flame detector(1).

Chromatographic analysis proved feasible and is described below.

Procedure

One hundred fifty grams of soil is placed in a medium porosity 45 x 127 mm Alundum thimble. The thimble is loosely plugged with absorbent cotton to prevent the soil from being splashed out. The soil is extracted overnight in a 200 ml Soxhlet extractor. The extract is evaporated nearly to dryness at

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reduced pressure in a rotary evaporator, transferred to a 10 ml volumetric flask and made up to volume.

A Wilkens Instrument and Research Model 204 Chromatograph fitted with a 1/8" x 5' glass column is used for analysis.

Both 5% Dow 11 on 60/80 mesh Chromosorb W and 5% Dow 200 on 80/90 mesh Anachrom ABS have been used as column packing. Both are satisfactory although the latter appears to give slightly sharper peaks. Column temperatures between 165° and 180° C are suitable with both packings. The carrier gas is prepurified nitrogen at a flow rate of about 30 ml/minute. A sodium flame detector(1) was used for the major part of this work, but a Varian-Aerograph (formerly Wilkens Instrument and Research, Inc.) phosphorus detector has also been used and is well adapted to this procedure. The sodium flame detector gives a limit of detection of about 0.06 p.p.m. Zinophos in the extract. In our hands the Aerograph phosphorus detector's limit is about 0.01 p.p.m. For 150 gram soil samples and a final extract volume of 10 ml as described, the calculated limits of detection in soil are .005 and .0007 p.p.m. respectively.

Discussion

Data on the recovery of Zinophos from several soils are given in Tables I and II. As can be seen from Table 1, recovery is good in all cases. Table II shows that the recovery of Zinophos from soil does not decrease greatly upon frozen storage.

None of the soils used shows any chromatographic components other than those attributable to organophosphorus pesticides.

Getzin(2) has used the sodium flame detector in a similar procedure using acetone instead of hexane and extracting for only one hour. In our experience, this gives lower recoveries at low Zinophos concentrations, possibly due in part to the large quantity of extraneous material in the extract or to the shorter extraction time.

TABLE I

Immediate Recovery of Zinophos From Soil

Sample Weight grams	Spike Rate p.p.m.	Recovery percent	
Felida Silt Loam			
50	0.50	82	
80	0.34	99	
80	0.32	79	
80	0.64	82	
Lauren Sand Loam			
50	0.50	88	
80	0.64	88	
80	0.64	86	
80	0.64	81	
80	1.61	80	
Burntbridge Silt Loam			
50	0.50	94	
50	0.50	88	
Caldwell Silt Loam			
50	0.50	85	
80	0.64	97	
80	0.64	90	
80	0.64	79	
20	0.85	76	
19	0.85	96	
22	0.85	79	Ave. 82%
18	0.85	93	
19	0.85	75	
22	0.85	76	

TABLE I (cont.)

Sample Weight grams	Spike Rate p.p.m.	Recovery percent	
Caldwell Silt Loam (cont.)			
20	4.2	91	
20	4.2	100	
21	4.2	96	Ave. 97%
22	4.2	98	
20	4.2	97	
17	4.2	100	
21	4.1	77	
20	4.1	94	
20	4.1	88	Ave. 92%
20	4.1	95	
21	4.1	100	
21	4.1	103	

TABLE II

Recovery of Zinophos From Caldwell Silt Loam After Storage at -5° to -10° C.

Time of Storage days	Spike Rate p.p.m.	Sample Size grams	Recovery percent
49	5.06	40	92
49	5.06	58	93
114	5.06	50	78
191	3.47	99	78

Acknowledgments

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References

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