

Analysis of Aldrin in the Presence of Sulfur by Electron Capture Gas Chromatography

by

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Elemental sulfur in extracts of plant and animal origin (OSADCHUK and WANLESS 1968), sewage sludge (MILLSON 1970), and soil (PEARSON et al. 1967, FRAZIER et al. 1970) often interferes with electron capture GLC analysis of chlorinated insecticides, particularly aldrin. Aldrin analysis in the presence of sulfur has been done by oxidizing aldrin to dieldrin (OSADCHUK and WANLESS 1968), by removing sulfur with copper-aluminum alloy (SCHUTZMANN et al. 1971), and mercury (GOERLITZ and LOW 1971), and by direct electron capture GLC using OV-17 as the liquid phase (LESTER and SMILEY 1972). The third method is the least time-consuming. The present report describes an improved liquid phase for this direct GLC analysis.

Elemental sulfur gives rise to three components under several GLC conditions (PEARSON et al. 1967, OSADCHUK and WANLESS 1968, SCHUTZMANN et al. 1971, STRUBLE 1972). On OV-17 liquid phase, one of the minor sulfur components had a retention time similar to that of aldrin and the main peak was not completely resolved from dieldrin and DDE (Table 1). The use of Apiezon L liquid phase for this analysis was much more satisfactory. Sulfur was not detectable on Apiezon L until the column became saturated, which required the injection of about 300 ng of sulfur. Once the column was saturated, sulfur appeared as one peak at 22.8 min, which interfered with the analysis of DDE (Table 1). To free the column of sulfur, the column was allowed to remain at operating temperature overnight. A detector bypass (STRUBLE 1970) was used to prevent sulfur from collecting on the detector foil. Solutions to be analyzed could contain up to 2 ppm sulfur without interfering with insecticide analysis, provided the total number of injections (5 μ l) per 8-hr period did not exceed 30.

Apiezon N and M liquid phases gave similar results but were saturated by smaller quantities of sulfur.

A Varian Model 600D gas chromatograph was used with a tritium ECD and a 2 mm ID glass column with a glass injector liner. A column (102 cm) packed with 3% OV-17 on 60/80 mesh, AW-DMCS treated, Chromosorb W was used with a nitrogen (carrier) flow rate of 66 ml/min, injector and column temperatures of 195 and 190°C, and an attenuation of 16×10^{-9} amp for a full scale deflection of 25 cm. Apiezon L (10%) was used on the same solid support in an 84 cm column and with a nitrogen (carrier) flow rate of 46 ml/min, injector and column temperatures of 200 and 190°C, and an attenuation of 8×10^{-9} amp.

TABLE 1

The GLC analysis of chlorinated insecticides in the presence of elemental sulfur

Compound	Injected (ng)	Liquid phases			
		OV-17		Apiezon L	
		R _t (min)	Pk. ht. (cm)	R _t (min)	Pk. ht. (cm)
Sulfur ^a	3	1.2	13.7	-	-
		3.5	3.4	-	-
		8.9	18.1	22.8	Variable
Lindane	0.1	2.4	10.2	4.4	13.3
Heptachlor	0.5	3.1	32.0	6.2	4.0
Aldrin	0.1	3.9	5.8	8.4	8.7
Heptachlor epoxide	0.4	6.2	1.5	10.4	2.7
Dieldrin	0.1	10.5	10.0 ^b	17.3	3.2
p,p'-DDE	0.4	10.5	10.0 ^b	22.0	14.7
p,p'-DDD	0.5	17.0	3.5	33.0	10.0 ^b
p,p'-DDT	1.0	21.0	5.0	33.0	10.0 ^b

^a Sulfur, USP grade, was recrystallized from xylene-ethanol to a constant m.p. 117-118°C (uncorrected).

^b Peak height is for the total of two unresolved compounds.

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