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The examination of mixtures of Captan and Phaltan* by gas chromatography**

The fungicides Captan [N-(trichloromethylthio)-4-cyclohexene-1,2-dicarboximide] and Phaltan [N-trichloromethylthiophthalimide] are used extensively in the treatment of foliar, soil, and seed-borne diseases. Available methods for the determination of residues of these compounds in crops have been reviewed by OSPENSON *et al.*¹, and this review has been recently supplemented by the description of gas chromatographic²⁻⁵ and thin-layer chromatographic⁶⁻⁷ techniques for Captan. However, the similar chemical characteristics of Captan and Phaltan have made it difficult to analyze mixtures of these compounds except by tedious procedures of separation of the materials on silicic acid¹ or florisol columns⁴ prior to analysis by the resorcinol colorimetric or by the gas chromatographic methods that have been recommended.

The stationary liquid phases, Dow 11, DC-200, OV-1, OV-17, QF-1, and SE-30, in the amounts and under the conditions commonly used for pesticide residue analysis by gas chromatography, will not resolve mixtures of Captan and Phaltan on the gas chromatograph column. During the process of screening other liquid phases for possible application to this problem, it was observed that columns which contained GE XE-60 (nitrile) silicone gum effectively separated Captan and Phaltan and produced two distinct peaks on the gas chromatogram. The procedure is described below.

Materials and methods

Gas chromatographs. Aerograph, Model 204-B, electron capture detector (250 mC); column temperature 195°, injection temperature 200°, detector temperature 200°; range 10, attenuation 2; nitrogen carrier gas, flow rate 30 ml/min.

F & M, Model 810, electron capture detector (200 mC); column temperature 200°, injection temperature 215°, detector temperature 200°; pulse interval 50 μ sec, range 10, attenuation 32; argon-methane (90:10) carrier gas, flow rate 75 ml/min.

Gas chromatographic columns. The silicone GE XE-60 (nitrile) was dispersed in acetone in amounts that would provide column supports containing 3% or 5% GE XE-60 on a wt/wt basis (liquid phase/solid support). The solid support, Chromosorb W, high performance grade, acid washed (AW) and treated with dimethyldichlorosilane (DMCS), was added to the acetone-GE XE-60 dispersion, mixed thoroughly, and the acetone was removed by evaporation on a steam bath. The dried support material was packed into 1/8 in. \times 5 ft. borosilicate glass columns, for use in the Aerograph gas chromatograph, and into 1/4 in. \times 4 ft. columns, for use in the F & M instrument. The material was retained in the columns with plugs of silanized glass wool. All columns were conditioned for at least 18 h prior to use at the temperature (195-200°) used for the analysis of the compounds.

Captan, 92% assay, and Phaltan (Folpet), 88% assay, were supplied through the courtesy of the Chevron Chemical Company, Richmond, Calif. Solutions of the

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up to 5.0 ng for similar samples applied to the F & M instrument. The data indicated that a 3 % silicone GE XE-60 column would be preferable, because of the decreased retention time characteristics and because of minimal "bleed" problems after about 150 h of column conditioning.

Preliminary studies have been made with this gas chromatographic procedure, in which fresh papayas were fortified with Captan and Phaltan at the 1.0 p.p.m. level. Cleanup of the benzene extract of the papaya consisted of a 5-minute contact of the extract with Nuchar C-190 carbon (3 g carbon per 100 g whole fresh fruit). Papaya plant extractives did not affect the pattern of the Captan and Phaltan curves chromatographed on the GE XE-60 column, and recoveries of the fungicides from the extracts ranged from 85–95 %.

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Gas-liquid chromatography of methylated D-galactose derivatives

Gas-liquid chromatography (GLC) of methylated methyl glycosides, and GLC of methyl-D-galactosides^{1–3} in particular, is widely used for structural polysaccharide investigation by methylation procedure^{4, 5}. This paper describes the further investigations of the experimental conditions of GLC-separations of methylated methyl-D-galactosides.

Experimental

Apparatus. The analysis was carried out using a Pye Argon Chromatograph. Experimental conditions are given in Table I.

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