Thin Layer Chromatography of Metaldehyde

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While studying metaldehyde residues on food crops it became necessary to develop a rapid procedure for separation and detection of the compound in plant extracts. Thin layer chromatography proved to be an effective method and the following procedure was developed.

Absorbents. Adsorbosil-1 was applied to glass plates in layers 250 microns thick using conventional techniques. The plates were air dried and used without activation.

Sample Preparation. Head lettuce samples weighing 100 g. were placed in a blender and 1.0 mg. of metaldehyde was added directly to the lettuce to correspond to a 10 ppm. concentration on lettuce. Chloroform (200 ml., technical grade redistilled) was added and the mixture blended for three minutes at high speed. The resulting slurry was vacuum filtered through Whatman #1 filter paper and the aqueous phase separated from the chloroform layer in a separatory funnel. Ten grams of Nuchar Attaclay was added to the chloroform extract for color removal of the pigmented extracts. After stirring for five minutes the slurry was vacuum filtered and the decoloration step repeated. The clear extract was evaporated to dryness and taken up in 5 ml. of chloroform

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for spotting on thin layer plates.

Development. Four chromatographic solvent systems were developed which gave good separation of metaldehyde from plant constituents remaining in the final extract and which produced color upon application of the spray reagents.

TABLE

Rf values for metaldehyde and plant constituents in various solvent systems

Solvent System	Rf		
	Metaldehyde Red[a]	Plant Constituent 1 Green[a]	Plant Constituent 2 Orange[a]
Chloroform (Aged 1 week)[b]	0.34	0.27	0.22
Acetone-benzene (20:80)	0.77	< 0.69	< 0.69
Ethyl acetate	0.74	0.81	0.69
Methanol-toluene (20:80)	0.55	0.67	0.26

[a] Spot color
[b] Fresh chloroform gives Rf of 0.56 for metaldehyde.

Visualization. Metaldehyde was located after development by spraying the plates heavily with 5% guiacol in chloroform followed by spraying with hot (90°C) concentrated sulfuric acid (1) in a well ventilated and plastic protected hood. As little as 0.1 microgram of metaldehyde can be visualized by this method using stock solutions. With plant extracts the lower limit of sensitivity would approach 1 ppm. metaldehyde on food crops.

REFERENCE

1. BRUYERE, P., Bull. Soc. Chim. Biol. 8, 462-463 (1926).