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Note

Qualitative analysis of carboxylic acids by partition thin-layer chromatography

CHRISTOPHER JOHN LUPTON

Textile Research Center, Texas Tech University, P.O. Box 4150, Lubbock, Texas 79409 (U.S.A.)

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Adsorption thin-layer chromatography has been used in the past for resolution of mixtures of mono- (C_1-C_6) and di- (C_2-C_6) carboxylic acids. Solvents incorporating aqueous ammonia have been recommended for elution on layers of silica gel^{1,2}. However, solvents containing ammonia must be used with great care. If the concentration of ammonia is slightly high, the acids remain close to the origin; if it is too low, the acids "streak" towards the solvent front with a subsequent loss of resolution. It is, therefore, necessary to standardize the aqueous ammonia before use. On storage, the effectiveness of the eluent may change, owing to reaction of ammonia with other components of the solvent mixture.

Normal partition chromatography has been found to be a more reliable method for resolving mono- and dicarboxylic acids.

EXPERIMENTAL AND RESULTS

Chromatography of monocarboxylic acids

Silica gel G (Merck) films were impregnated with a 10% aqueous solution of ethylene glycol and allowed to dry at room temperature for 24 h. Samples (10 μ g each) of monocarboxylic acids (C_1-C_6) were placed on the substrate and were chromatographed using the two-development technique, light petroleum (b.p. 40-60°)-acetone (2:1) 95% saturated with ethylene glycol being the eluent. The acids were detected with methyl red reagent (Fig.1). This chromatographic system suffers from the disadvantage that a portion of the free acids was always irreversibly adsorbed at the origin of the chromatogram, making quantitative analysis difficult. Although methyl red was the best of the reagents tested, its chromogenic power is not good enough for quantitative analysis.

Chromatography of dicarboxylic acids

Silica gel G films were impregnated with polyethylene glycol 400 from 10% aqueous solution. Samples (10 μ g each) of diacids (C_2-C_6) were applied to the prepared substrate and chromatographed using diethyl ether-98% formic acid (50:1) 95% saturated with polyethylene glycol 400 as the eluent. The resulting chromatogram was dried at 110° for 5 min and the acids were detected with bromocresol green reagent (Fig. 2).

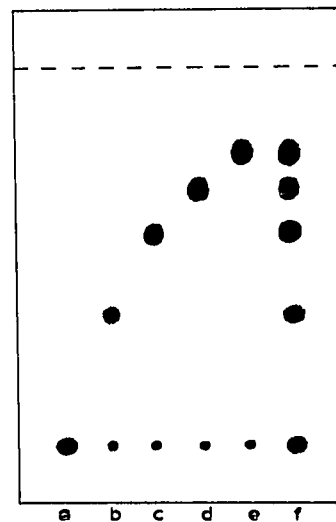
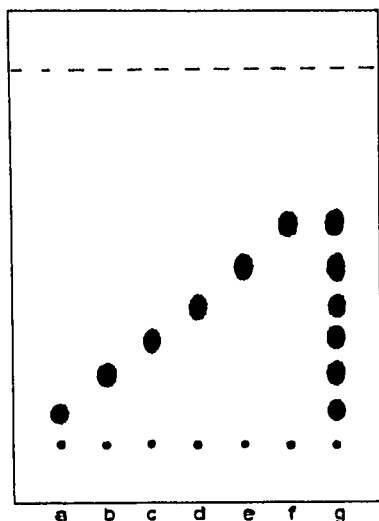


Fig. 1. Chromatogram of monocarboxylic acids. Solvent: light petroleum (b.p. 40–60°)–acetone (2:1), 95% saturated with ethylene glycol. Substrate: silica gel G impregnated with a 10% aqueous solution of ethylene glycol. Developments: two, each to a distance of 15 cm. Spray: Methyl red reagent. (a) Formic acid (10 μ g); (b) acetic acid (10 μ g); (c) propionic acid (10 μ g); (d) butyric acid (10 μ g); (e) valeric acid (10 μ g); (f) caproic acid (10 μ g); (g) mixture of a–f (10 μ g each).

Fig. 2. Chromatogram of dicarboxylic acids. Solvent: diethyl ether–98% formic acid (50:1), 95% saturated with polyethylene glycol 400. Substrate: silica gel G impregnated with a 10% aqueous solution of polyethylene glycol 400. Spray: bromocresol green reagent. (a) Oxalic acid (10 μ g); (b) malonic acid (10 μ g); (c) succinic acid (10 μ g); (d) glutaric acid (10 μ g); (e) adipic acid (10 μ g); (f) mixture of a–c (10 μ g each).

Two thin-layer chromatographic systems have been developed for the qualitative analysis of mixtures of carboxylic acids; neither system suffers from the disadvantages normally associated with eluents containing aqueous ammonia.

REFERENCES

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