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Thin-layer chromatography of epoxy plasticizers

It has been reported[1] that plasticizers lend themselves satisfactorily to thin-layer chromatography (TLC) analysis using Silica Gel G plates with high moisture content. The developing solvent used was benzene–methylene chloride (2:1). However, it was hard to differentiate between an epoxidized tallate, an epoxidized soya oil or an epoxidized linseed oil. To improve the results, HUBER AND WIMMER[2] suggested a mixture consisting of petroleum ether (boiling range 40–80°)–ethyl ether–glacial acetic acid (80:20:1). However, this too was not satisfactory in accomplishing complete separation. Epoxy plasticizers are based on epoxidized triglycerides (soybean oil, linseed oil) or alkyl stearate[1]. Only the latter, however, can be detected by gas chromatography, since triglycerides cannot be evaporated without decomposing them[1]. Our studies have revealed that epoxidized triglycerides lend themselves satisfactorily to analysis by TLC on Silica Gel G plates of 500 μ thickness.

In our studies, we have found that the best developing solvent system to distinguish between different types of epoxidized plasticizers (soya from linseed or tallate types) was: hexane–ethyl ether–glacial acetic acid (70:40:3). This solvent system also differentiated oils from primary plasticizers such as adipates, phthalates, and from the phosphate type plasticizers. The R_F value of the epoxy plasticizer was much smaller than the primary and phosphate plasticizers, so one can easily distinguish between the different types.

Experimental

Plates coated with 500 μ thick Silica Gel G (E. Merck A.G.) are spotted with 5 μl of 0.5% w/w of known plasticizers in methylene chloride. The plate is developed in a solvent mixture of hexane–ethyl ether–glacial acetic acid (70:40:3), all ACS reagent grade. The traveling distance of the solvent front should be 15 cm. After the marked end point has been reached, the plate is dried for 15 min at 105°. Visualization is accomplished with one of the following spraying reagents: (1) Concentrated H_2SO_4 or chromic acid–concentrated H_2SO_4 followed by charring in the oven at 125–130°. (2) 10 g of $SbCl_5$ (5 ml of $SbCl_5$) in 40 ml carbon tetrachloride—heat for 30 min in an air oven at 100°. (3) 20% alcoholic solution of phosphomolybdic acid, followed by heating for 20 min at 130–140°. Upon cooling of the plate, immerse in a chamber saturated with ammonium hydroxide for a few seconds.

The above three spraying reagents have been found to be best suited for this type of work.

Results and discussion

Fig. 1 shows the results obtained. It is of interest to observe that this simple technique can differentiate between an epoxy type oil of the soya family, the tallate family and that of the linseed oil family. The 20% phosphomolybdic acid spraying reagent gives a better visualization of the spots than the concentrated H_2SO_4 or $SbCl_5$ solution. In Fig. 1, the plate was sprayed with phosphomolybdic acid. The important part of this technique is that, even if one can separate the epoxidized oil by another technique and follow it by IR analysis, the IR spectra do not differentiate

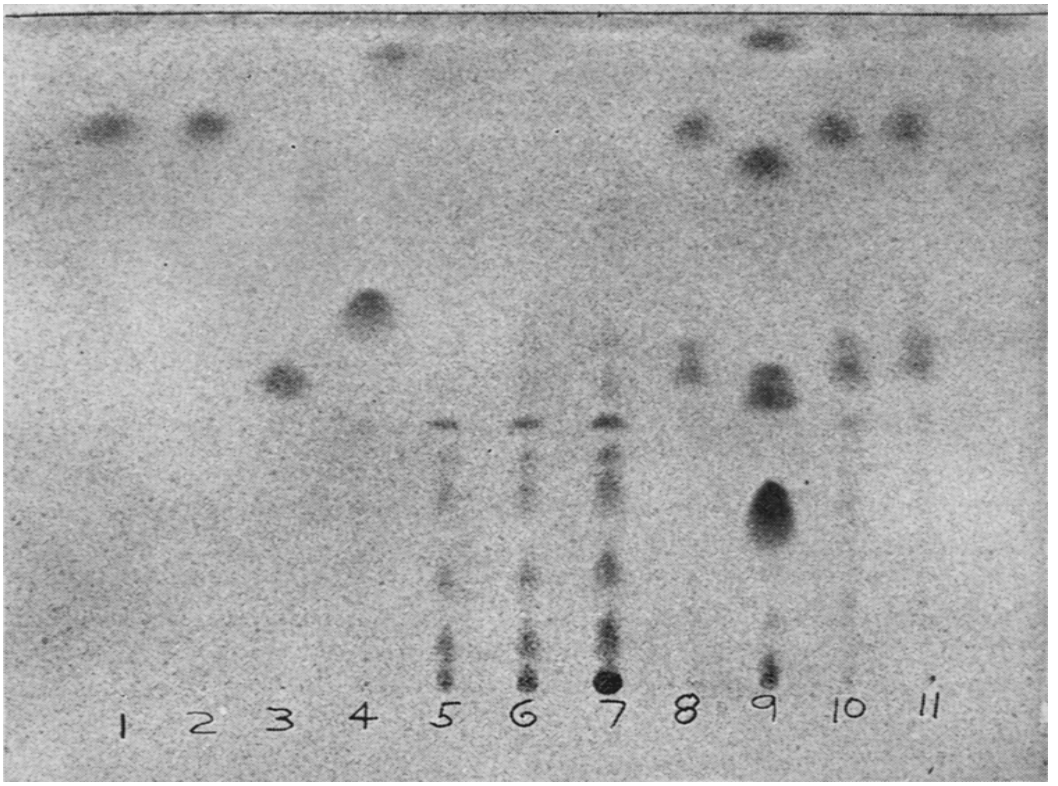

Fig. 1. Thin-layer chromatogram of epoxidized and non-epoxidized plasticizers. 5-μl spottings of 0.5% by weight concentration on 500 μ thick coating of Silica Gel G. 1 = dioctyl phthalate (DOP); 2 = dioctyl adipate (DOA); 3 = Santicizer 141 (octyl diphenyl phosphate); 4 = stearic acid; 5 = Paraplex G-62 (epoxidized soya oil); 6 = Admex 711 (epoxidized soya oil); 7 = Flexol EPO (epoxidized soya oil 1% by weight); 8 = Santicizer S-73 (epoxidized tallate oil); 9 = Epoxol 8-2B (butyl epoxidized linseed oil); 10 = Drapex 4.4 (octyl epoxy stearate); 11 = Admex 746 (epoxidized octyl tallate).

between a tallate epoxidized oil, a linseed epoxidized oil, or an epoxidized soya oil, whereas TLC does accomplish this in a very simple way.

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