CHROM. 4552

Conical thin-layer chromatography using silica gel without binder

Preparative conical thin-layer chromatography was developed by BRENDEL *et al.*[1], who reported that it gave better separation of compounds with close R_F values than could be achieved with preparative thin-layer plates[1]. It was found especially helpful for compounds that tended to trail. This conical chromatographic method has been useful in our laboratory for separating a reaction mixture containing two components with close R_F values (see Figs. 1 and 2). However, the adsorbent binder interfered with our determination, and the procedure described previously for coating a frustum was not satisfactory when silica gel without binder was used[1]. We have developed a method for successfully coating frustums with Silica Gel HF_{254}, without $CaSO_4$ binder (Brinkmann Instruments Inc., Westbury, N.Y.) as described below.

A glass frustum of 15 cm height with ends 20 and 3 cm in diameter and the smaller end closed by taping a teflon plug cut to size[1], was placed in a 100° oven for 10–15 min. A slurry was made by mixing 25 g of Silica Gel HF_{254} with 45 ml of distilled water for 60 sec in an Osterizer Model 10 blender. Wearing heavy gloves, the operator poured the slurry into the hot frustum immediately after it was removed from the oven, and the slurry pool was manually spiraled onto the sides with a rapid turning motion. The gel hardened to an even coating almost as the spiraling operation was complete. Then, the frustum was allowed to dry for 3 h at room temperature before it was activated by heating for 1 h at 100°.

In an alternate procedure the handling of the hot frustum was avoided by using a frustum at room temperature and heating the slurry with a heat gun as it was spiraled. This procedure gave a less even coating than when a preheated frustum was used, but might be adaptable for some uses.

Fig. 1 shows preparative conical thin-layer chromatographic separation of 20 mg of 3,4-diacetoxy-2-acetyl-4,5-dihydrofuran[2] from an impurity with a slightly

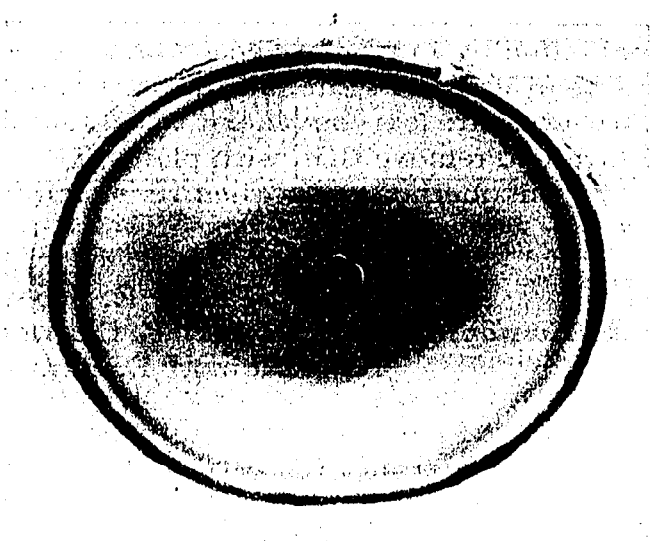

Fig. 1. Conical thin-layer chromatogram of a 20-mg mixture containing 3,4-diacetoxy-2-acetyl-4,5-dihydrofuran.

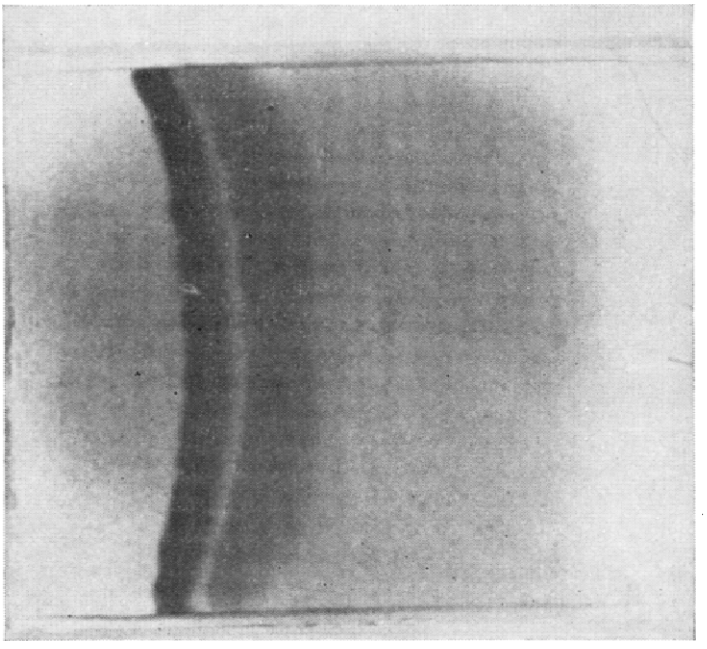

Fig. 2. Thin-layer chromatographic plate of 20 mg of the same mixture used in Fig. 1.

larger R_F value. The developing solvent mixture was the upper phase prepared by shaking in a separatory funnel 200 ml benzene, 47 ml ethanol, 15 ml water, and 1 ml conc. NH_4OH. Development time was 3–4 h.

Fig. 2 illustrates an 8 × 8 in. preparative thin-layer chromatographic plate spotted with 20 mg of the same impure furan mixture, and developed in the same solvent mixture. The adsorbent was a Silica Gel HF_{254} coating 1 mm thick. Development time was 20–30 min.

As BRENDEL *et al.* observed[1], preparative conical TLC can be advantageous in separating compounds with close R_F values, and particularly if they run near the solvent front. As Figs. 1 and 2 indicate, the two bands are visibly tighter and better separated by the conical method. In addition, after they were scraped from the frustum and eluted from the adsorbent, examination by TLC showed each band to be more cleanly separated from adjacent bands than was the same mixture separated by preparative TLC on the plate. However, frustums are less convenient to coat, and more difficult to spot and bands are more difficult to remove than with plates. These observations indicate the recommended use of this method only when its superior separation can be of advantage.

U.S. Fruit and Vegetable Products Laboratory,
Southern Utilization Research and Development Division,
Agricultural Research Service,
United States Department of Agriculture,
Winter Haven, Fla. 33880 (U.S.A.)

PHILIP E. SHAW
ROBERT E. CARTER
ROBERT E. BERRY

1 K. BRENDEL, R. S. STEELE AND E. A. DAVIDSON, *J. Chromatog.*, 30 (1967) 232.
2 TH. SEVERIN AND W. SEILMEIER, *Z. Lebensm.-Untersuch. Forsch.*, 137 (1968) 4.

Received December 1st, 1969