

Quantitative thin-layer chromatography on aluminium foil

Since the introduction of thin-layer chromatography, efforts have been made to find methods for the quantitative evaluation of the separated substances. Among these methods the most reliable seem to be those in which extraction of the substance from the adsorbent is followed by quantitative analysis of the extract. Quantitative removal of the relevant part of the adsorbent (the "spot") from the glass plate is necessary. This is obviously a drawback of the thin-layer technique in comparison with paper chromatography, where the spot is simply cut out. After removal of the adsorbent some substance may adhere to the glass plate and it is not surprising that low recoveries often occur. Simultaneous analysis of known amounts of the substance as a reference standard is of course advisable¹.

The present method obviates the necessity of removing the adsorbent from the glass plate and reverts to cutting the spot out of the layer. On the other hand, it profits fully by the perfect flatness of a glass plate, which makes it possible to obtain a uniform layer of adsorbent.

For this purpose aluminium foil is fixed to a glass plate and the thin layer of adsorbent is spread on the aluminium. After chromatography the spots are cut out.

Experimental

Materials

Glass plate: *e.g.* 1500 × 500 × 5 mm, smooth surface

Aluminium foil: *e.g.* Indufol, 1000 × 300 mm

Strips of filter-paper: 1200 × 70 mm

Scotch tape: 50 mm

Stainless-steel frame: 200 × 200 × 30 mm

Hydrofluoric acid: 1% aqueous solution in water

Method

Paste with water 100 cm aluminium foil on the plate and make the surface flat, using a piece of cloth. Degrease the aluminium foil with hydrofluoric acid. Rinse with water and dry. Fix two strips of filter-paper on the foil by means of Scotch tape, leaving a strip of approximately 18.5 cm of the foil visible (see Fig. 1). Pour the slurry of the adsorbent on the foil and spread it with a glass rod². Allow to stand for half an hour in the open air, then remove the filter-paper. Cut the foil into squares of 18 × 18

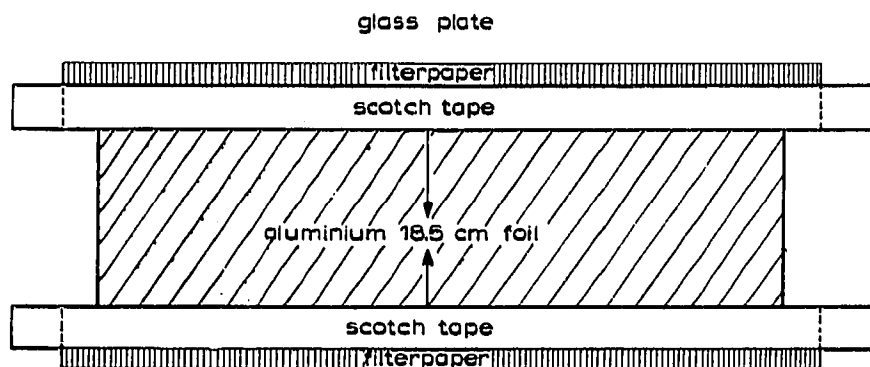


Fig. 1. Aluminium foil on a glass plate, ready for covering.

cm with a lancet and mount these on frames. Dry at 110° for one hour and the framed thin layer is ready for use.

The thickness of the layer (0.2 mm) may be varied by using more strips of filter paper. The separation on aluminium foil is the same as on a glass plate.

Partition chromatography of 2,4,5,4'-tetrachlorodiphenyl sulphide

For this purpose thin layers were prepared consisting of silica gel with polyethylene glycol as a stationary phase³. The slurry for coating was obtained by stirring 30 g of silica gel GF 254 (purified by extraction with methanol and methylene chloride) with a solution of 3 ml of polyethylene glycol in 60 ml of distilled water.

A micro pipette was used to deliver 200 μ l of petroleum ether, containing 100 to 800 μ g of the sulphide, on the silica gel layer. The chromatogram was developed with *n*-hexane using an ascending technique until the solvent front had ascended a distance of 17 cm. The foil was dried in the open air for 3 minutes and rechromatographed twice in the same way.

TABLE I

RECOVERY OF 2,4,5,4'-TETRACHLORODIPHENYL SULPHIDE

	<i>Without running</i>	<i>After 3 runs</i>
Average recovery	99.5 %	100.1 %
No. of determinations	5	8
Range	98.3–100.1 %	99.9–100.5 %
Standard deviation	0.73	0.25
R_F after 1 run	—	0.3

After the third run the foil was dried for 3 minutes. The location of the spot was clearly visible in ultraviolet light (254 nm); in this light the dark spot was cut out and the snip extracted with methanol. The absorption was measured spectrophotometrically at 258 nm ($a_{1\text{cm}}^{1\%} = 500$).

The results are given in Table I.

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