

Note

The use of plastic foil in small-scale thin-layer chromatography

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Plastic foil is used only seldom as an aid in thin-layer chromatography (TLC)^{1,2}. However, the impermeability of materials such as polyethylene and polyfluoroethylene to organic liquids and vapours makes them suitable as barriers for solvent mixtures used as eluents, as envelopes for sponge-like containers of solvents and for other similar applications. In this paper, several practical examples are described (Fig. 1).

In all experiments, the use of pre-coated TLC plates was preferred to that of home-made plates. The pre-coated plates are easily cut into pieces of appropriate size³ without damaging the thin layer; moreover, the separations obtained are of a higher quality. Small spots were applied with the help of a pointed paper-wick; for larger spots, an injection syringe can be used.

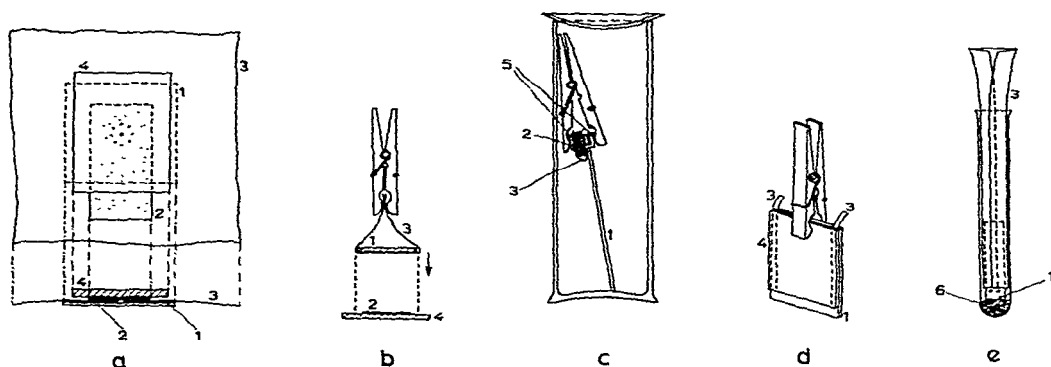


Fig. 1. Several types of apparatus for thin-layer chromatography: (a) and (b) radial development; (c) descending development; (d) and (e) ascending development. 1, Chromatoplate; 2, filter-paper; 3, plastic foil; 4, glass plate; 5, flat glass rods; 6, glass- or quartz-wool.

(a) For radial chromatography, a pre-coated plate is cut into pieces of *ca.* 2 × 2 cm, and a number of spots are applied around a mark made lightly with a pencil. The plate is covered with a relatively large piece of pore-free foil (thickness *ca.* 0.05 mm) that has been pierced at its centre with a pin. The hole (diameter 0.1–0.3 mm) in the foil should coincide with the pencil mark. Next, a ribbon of filter-paper is placed across the foil, covering the hole, and a piece of glass is placed on the sandwich

in such a way as to leave free the lower end of the paper. The solvent (mixture) selected as the mobile phase is applied on this end of the paper by means of a dropper. As soon as the paper has been sufficiently wetted, the solvent flows radially out of the hole on to the thin layer, and development starts. Owing to the low volume capacity of the piece of filter-paper, changing the speed of application of the solvent to the paper allows some control of the speed of elution.

(b) Radial chromatography can also be carried out so as to produce a fully circular chromatogram. A spot is applied at the centre of a pre-coated plate (size *ca.* 2×2 cm), and lightly touched with a pencil. The plate is enveloped with an oblong piece of plastic foil, held by a peg. After piercing the foil on the spot of the pencil mark, the whole is lowered on to a pad of filter-paper previously wetted with a suitable solvent mixture. Development produces a series of concentric circles.

(c) An array for descending chromatography is shown in Fig. 1c. A strip of filter-paper (size 20×4 cm) is folded 10 times and wrapped with a piece of plastic foil (size 8×4.5 cm) in such a way as to leave one end of the paper strip free. This end is bent over a thin-layer plate having the same width and is clamped by means of two flat glass rods and a peg, as shown in the figure. A margin of *ca.* 0.5 cm between the lower end of the bent filter-paper and the row of (previously applied) spots is sufficient. Development, which should be carried out in a saturated chamber, starts upon careful addition of small amounts of the eluent to the folded filter-paper.

(d) Small sandwich chambers are conveniently assembled from a pre-coated plate of size *e.g.* 5×4 cm, and an ordinary glass plate that is 0.5 cm shorter. Narrow ribbons of plastic foil along the sides serve as spacers; for easy handling, these ribbons should protrude slightly from the sandwich. The lower edge of the cover plate serves as a striker for application of the spots. The depth of solvent mixture in the chamber should not exceed 2 mm. If larger sized plates are to be used, the spacer foils must have a thickness of 0.2 mm or more. Moreover, the sides of the sandwich must be clamped in order to prevent bending of the glass plates, which should result in the cover plate touching the thin layer.

The method allows good preservation of activated plates, provided that assembly of the sandwich takes place when the plate is still hot, *i.e.*, in the oven, and cooling is carried out in a desiccator. During spotting, the sandwich will largely prevent the adsorption of water vapour by the thin layer.

A spaceless sandwich is obtained when the ribbons are replaced with a rectangular piece of plastic foil of the same size as the cover plate. However, as plastic materials are wetted by several organic solvents, the choice of eluent may then be limited.

(e) Ascending chromatography can also be carried out in a test-tube (size *e.g.* 15×1.5 cm). A wad of cotton- or quartz-wool is pressed against the bottom of the tube, several millilitres of solvent are added and the wad is completely immersed and levelled with the help of a flattened glass rod. Superfluous liquid is poured out and the tube is left standing upside down for 0.5 min. After cleaning the rim of the tube, a piece of thick (*ca.* 0.2 mm) plastic foil (18×4.5 cm) is inserted, which forms an overlapping inner lining. When this lining touches the wad, the remaining free liquid is sucked away by capillary action. Next, the foil is pulled up a few millimetres; if necessary, its inside is dried with a strip of filter-paper.

A 1-cm wide thin-layer plate, to which spots have been applied 0.5–1 cm from the lower end, is dropped on to the wad. Development starts immediately and a

straight solvent front is observed. After development, the plate is easily recovered by reversing the tube.

The above techniques have been used successfully for the separation, on cellulose, of a series of transition metal ions with acetone–hydrochloric acid mixtures, of alkaline earth ions with methanol–6 *N* hydrochloric acid (4:1, v/v), and of colour pen inks with *n*-butanol–acetic acid–water (4:1:1, v/v). Various mixtures of polychlorinated biphenyls and naphthalenes have been separated on activated silica gel plates, using dry *n*-hexane as eluent. Details of the latter separations have been published elsewhere^{4,5}.

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