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Note

Simple device for continuous thin-layer chromatography

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Thin-layer chromatography (TLC) in saturated tanks, although satisfactory for analytical work, has some drawbacks in certain types of applications because of the phenomena that occur in the chromatographic system, *viz.*, solvent demixing, gradient effects at the solvent front and adsorption of solvent vapour from the atmosphere[1]. Although some of the effects can be compensated for by the use of the correction factor, ξ, the application of TLC in physicochemical investigations and determinations of suitable solvent systems for column chromatography[2–4] is limited owing to poor equilibration of the mobile and stationary phases.

The analogy between TLC and column chromatography could be made much closer if continuous development of the chromatograms could be achieved (*e.g.*, ref. 5), which would permit spotting of the sample after the equilibration of the system. An apparatus of this type for radial development (which also permits conditioning of the layer) has recently become available from Camag (Muttenz, Switzerland)[6]; a simple device for continuous development has also been constructed in this laboratory and found to be suitable for special chromatographic investigations and for the chromatographic analysis of strongly retained compounds which require prolonged development.

The chromatographic tank is of the "sandwich" type with the volume of the tank atmosphere reduced to the minimum. The plate or foil (A) with the thin layer of the sorbent is placed in a horizontal position on a larger glass plate and three rectangular pieces of glass (B) of thickness exceeding that of the TLC plate by *ca.* 0.5 mm are placed alongside to form a U-form spacing (Fig. 1). The plate in then covered with two further rectangular glass plates which join over the starting line on the TLC plate; a small hole in one of the cover plates allows the introduction of the developing solvent on to the layer. A 2-cm section of the TLC plate protrudes from the cover plates so as to permit the solvent at the end of the chromatogram to evaporate, thus causing a continuous flow of solvent (see also ref. 7).

The support plate and the spacing plates (B) can also be replaced with a metal (*e.g.*, aluminium) block with a flat rectangular hole, the depth which exceeds the thickness of the plate by 0.5–1 mm. The chromatographic system can then be thermostated.

The most suitable rate of solvent delivery is established in the following way. The solvent reservoir is placed close to the plate in such a position that the solvent level is slightly lower than that of the TLC plate. The solvent is delivered by a U-shaped

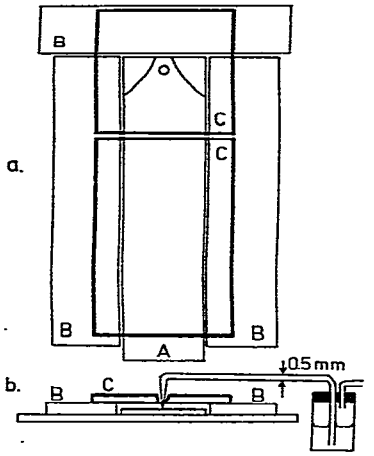

Fig. 1. (a). Schematic representation of a sandwich-type TLC apparatus for continuous development. A, TLC plate; B, spacing plates; c, cover plates. The bottom glass plate is not shown. The middle plates (A, B) are represented by thin lines and the cover plates with thick lines. (b) Solvent delivery.

glass (PTFE, stainless-steel) siphon (*ca.* 0.5 mm I.D.), the tip of which touches the thin layer. When the difference of levels between the capillary tip and the solvent is small then the solvent is held in the siphon by surface forces and the rate of its delivery is determined automatically by the thin layer itself. The method is simpler than that proposed by Blome (ref. 6, p. 62; solvent reservoir placed above the plate, rate of delivery controlled for individual solvents by the height of the reservoir and the length and diameter of the PTFE tubing).

The distance between the tip of the capillary and the starting line is chosen so that the solvent front is formed even before reaching the line of application of the samples. The distance is longer than the width of the plate so that narrow plates are more suitable for this technique, unless a special device is used to spread the solvent more rapidly at right-angles to the direction of development. The front is formed even more rapidly when the layer in the proximity of the siphon tip is shaped in the manner shown in Fig. 1a.

The samples are spotted behind the solvent front (or the last line of solvent demixing) after moving the second cover plate to form a 2-mm slit over the starting line (as shown in Fig. 1a). For simultaneous spotting of several samples it may be necessary to use a series of capillaries immobilized in a holder. The attainment of steady-state conditions can be observed by using a mixture of test dyes.

The determination of the solvent flow-rate for continuous development requires the use of a coloured test compound (marker) with $R_F = 1.00$. For prolonged development, test dyes with lower R_F values can be used (*e.g.*, R_F 0.20 for samples containing components whose R_F is less than 0.20 for a single development). The solutes reaching the end of the cover plate form a sharply defined zone.

The device can easily be adapted to gradient elution[6]. Another advantage of the apparatus for "quasi-column TLC" is the use of small amounts of the developing solvent. The device has also been found to be suitable for paper chromatography; the paper strip should be supported by several pieces of glass capillary in the proximity of the point of solvent delivery in order to avoid an irregular flow.

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