Journal of Chromatography, 516 (1990) 461–466
Elsevier Science Publishers B.V., Amsterdam

CHROM. 22 542

Note

Modification of a horizontal sandwich chamber for thin-layer chromatography

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(First received November 6th, 1988; revised manuscript received May 8th, 1990)

Thin-layer chromatography (TLC) is still commonly used, and new analytical applications and recent developments have been reported[1–5]. In recent years sandwich chambers have become more popular than conventional cylindrical or rectangular containers in which the plate is placed in an approximately vertical position. In sandwich chambers much less eluent is consumed; the eluent is poured into a narrow trough and is delivered to the adsorbent layer by various methods[6–8], *e.g.*, by a wick made of filter-paper, felt or a frit. In another construction[9], the role of the wick is played by a long porous strip placed in the eluent trough under the plate with the adsorbent layer underneath; the development starts when the reservoir (trough) is elevated so that the strip is pressed against the adsorbent layer. In Soczewiński's chamber[10–12], the eluent is delivered by a capillary siphon to a flat horizontal slit formed between the distributor beneath the cover-plate and the margin of the carrier plate cleaned of adsorbent; development is started by shifting the distributor (a thin strip of glass) over the edge of adsorbent layer. In the Camag linear developing chamber[3], the eluent is delivered from the trough to the edge of the plate through a narrow slit formed between the vertical wall of the trough and an adjacent glass plate.

Horizontal sandwich chambers have several advantages over conventional saturated chambers, *e.g.*, the horizontal position of the plate accelerates the migration of the mobile phase, the consumption of eluent is much lower and the sample can be applied as a band for micropreparative separations. Their disadvantage is a more complex construction and a correspondingly higher price.

In this paper a modified sandwich chamber is described.

CONSTRUCTION OF THE CHAMBER

The modified construction of the horizontal sandwich chamber developed in this laboratory[13,14] is relatively simple (Fig. 1). Fig. 1a shows its cross-section before development: the glass plate (1) covers a shallow (1 mm deep) rectangular reservoir (2) with the eluent (3). The precoated chromatographic plate (4) is placed (adsorbent layer downwards) on two parallel 1-mm ledges (6) (in Fig. 1 one is visible) on its longer edges, several millimetres from the eluent reservoir.

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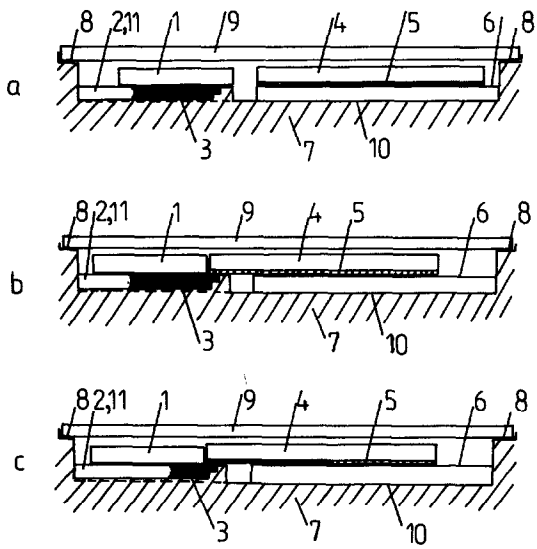

Fig. 1. Principle of action of modified sandwich chamber with a horizontal bottom of the eluent reservoir. 1 = Glass cover-plate; 2 = eluent reservoir; 3 = solvent; 4 = chromatographic plate; 5 = (dotted area) thin layer of adsorbent; 6 = ledge supporting the TLC plate; 7 = body of the chamber; 8 = rectangular depression; 9 = glass cover-plate of the chamber; 10 = bottom; 11 = edges of the eluent reservoir. (a) Original position; (b) beginning of development; (c) advanced development.

The chromatographic process is started by shifting the chromatographic plate 4 to the left (together with the cover-plate 1) so that its edge rests on the threshold of the reservoir and comes into contact with the eluent 2 along a narrow (*ca.* 1 mm) zone. Because the eluent reservoir is shallow (*ca.* 1 mm), a vertical meniscus is formed between its bottom and the cover-plate; the meniscus shifts to the right as the eluent is absorbed by the thin layer of adsorbent (Fig. 1c). To the right, the bottom is shallower, in the form of a step; the surface tension accumulates the last drops of eluent between this step and the adsorbent layer to the end, thus securing uniform delivery of eluent on the whole width of the adsorbent layer.

Another design which is advantageous owing to the sucking of the eluent by capillary forces in the direction of the edge of the chromatographic plate is to use a reservoir with a slanted bottom with the depth varying, *e.g.*, from 2 to 0.5 mm on the side of the plate, as illustrated in Fig. 2 for situations analogous to that in Fig. 1: (a) preliminary position; (b) beginning of development; and (c) partly developed plate with diminished volume of eluent in the reservoir.

Another version of development, suitable also for less stable self-made layers, consists in putting the plate 4 from the beginning in position (b) (Figs. 1 and 2) in a dry chamber. The reservoir is covered with the cover-plate 1, leaving a 4 mm gap between the latter and the chromatographic plate 4. A suitable volume of eluent is introduced into the reservoir and then the cover-plate is shifted to the right to close the gap and to start development.

A detailed view of the chamber for standard 50 × 100 mm glass plates is

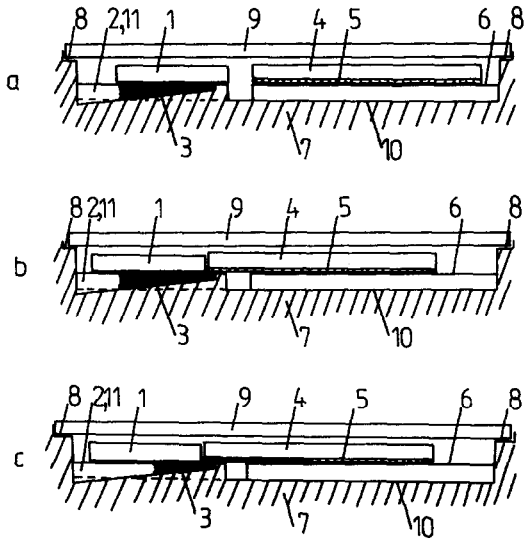

Fig. 2. Modified construction with slanted bottom of the reservoir. Notation as in Fig. 1.

presented in Fig. 3 in three projections: (a) overhead, (b) parallel to the shorter edge and (c) parallel to the longer edge. The chamber is made of polytetrafluoroethylene (PTFE) and consists of a PTFE plate (7) with a 1-mm deep depression (8) for the rectangular glass cover-plate (9). The bottom (10) of the chamber is 6 mm below the

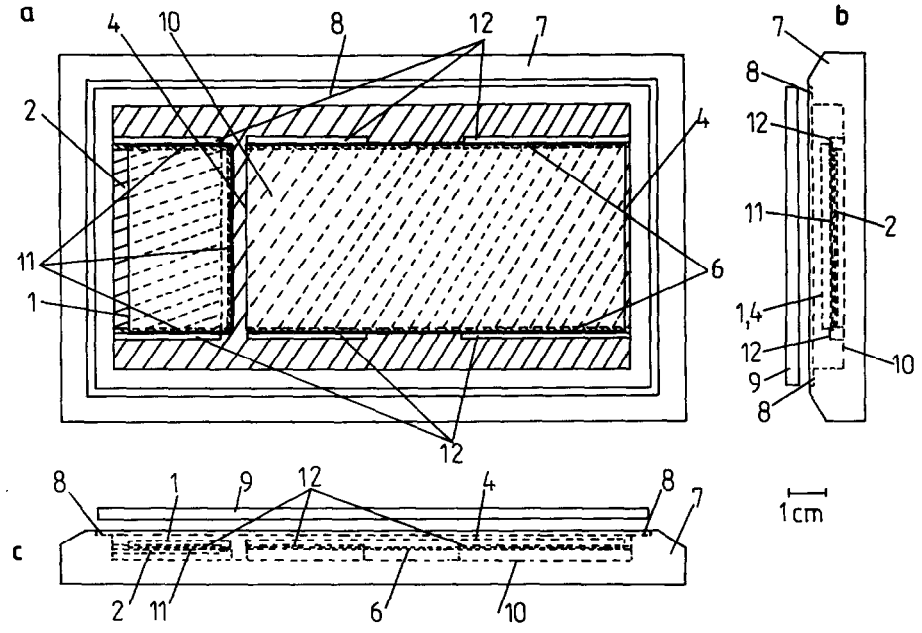

Fig. 3. Detailed design of the chamber for single precoated Merck plate (50 × 100 mm). Notation as in Fig. 1; 12 = vertical walls holding the plate in place.

upper surface of the plate; in parallel, another rectangular depression (2) (30 × 48 mm) forms the reservoir of the eluent, 1 mm deep. Along the right-hand edge, adjacent to the chromatographic plate, the reservoir is only 0.5 mm deep (the step is 1 mm wide) to facilitate uniform absorption of the last drops of the eluent. The edges of the reservoir (2) are on the same level as two parallel ledges (6), 1 mm wide, on which the chromatographic plate is placed; the ledges have low vertical walls (12) which prevent the chromatographic plate and the glass plate covering the reservoir from shifting across the chamber. The glass cover-plate of the reservoir is shorter by 5 mm from the shorter edge (11) of the reservoir. The above construction permits the development of a single plate.

Another construction with two reservoirs of eluent on the opposite sides of the chamber (Fig. 4) permits the simultaneous development of two chromatograms (on two separate plates).

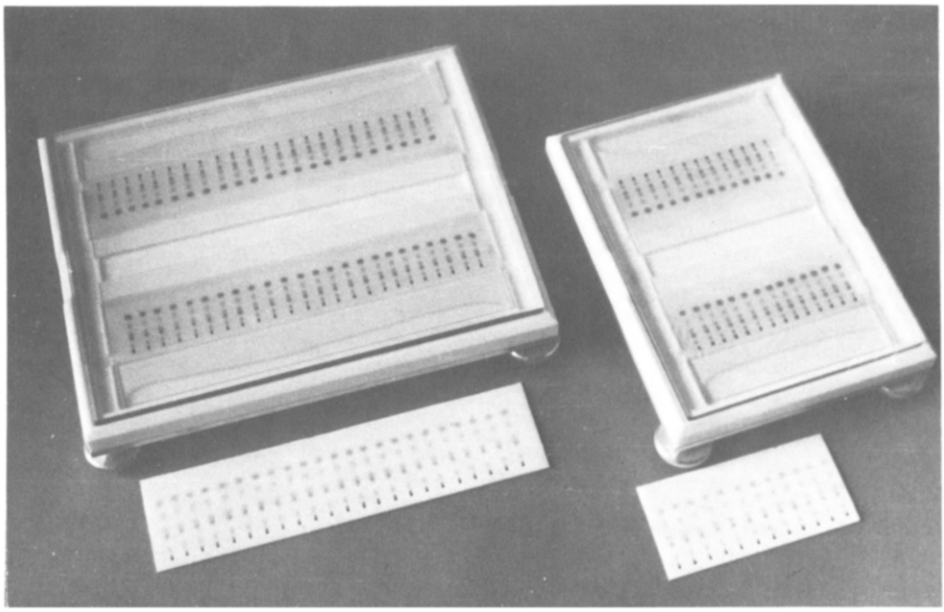

Fig. 4. Twin chambers for 10- and 20-cm wide plates during the development process. Manufacturer and distributor: Modin, Lublin, Poland.

The design of the chamber is suitable for the development of precoated or laboratory-made glass plates which can be placed (adsorbent layer downward) in the chamber with visual control of the process.

SPECIAL APPLICATIONS

Owing to complete absorption of the eluent from the reservoir, stepwise gradient elution is possible by stagewise introduction of eluent fractions of increasing eluent strength (as in ref. 15, Fig. 2) or by absorption of solvent vapour from the bottom of the

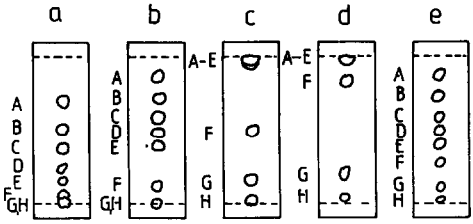

Fig. 5. Thin-layer chromatograms obtained with the chamber. (a–d) Isocratic elution; (e) gradient elution (see text). 10 × 10 cm TLC plates with silica (Merck). Samples: A = 4-chloro-4′-dimethylaminoazobenzene; B = fast yellow; C = 2-nitroaniline; D = 1-(2-methoxyphenylazo)-2-naphthol; E = 4-nitroaniline; F = 1-(4-hydroxyphenylazo)-2-naphthol; G = iodoeosin; H = phenol red.

chamber (ref. 1, Figs. 149–151) as in the well known chambers of Geiss and Schlitt[6], De Zeeuw[8] and Kaiser[16] and the linear Camag chamber.

Fig. 5 shows examples of chromatograms obtained using the horizontal chamber and isocratic elution with pure solvents, (a) toluene, (b) methylene chloride, (c) ethyl acetate, (d) acetone and (e) three-step gradient elution with (1) 1 cm^3 of methylene chloride, (2) 0.25 cm^3 of ethyl acetate and (3) 0.25 cm^3 of acetone, each portion of solvent being delivered into the solvent reservoir with a syringe, after complete absorption of the previous portion by the adsorbent layer. It can be seen that the horizontal chamber is suitable for the gradient chromatographic resolution of mixtures that cannot be completely separated by isocratic development.

The complete absorption of the eluent from the reservoir also permits the zonal application of large volumes of the sample for micropreparative separations, as in the equilibrium sandwich chamber with a glass distributor; the introduction of the sample from the edge of the layer (frontal + elution chromatography) greatly improves the separation capacity[2].

Fig. 6 shows an example of zonal application of a mixture on the TLC plate in a horizontal chamber with two reservoirs. The mixture was introduced into the first reservoir and the eluent (toluene) into the second. The chromatographic plate was brought into contact with the mixture to develop the TLC plate a distance of 2 cm (Fig. 6a). The development was then interrupted and the plate was left in the chamber to absorb the remainder of the mixture from the edge of the TLC plate. The

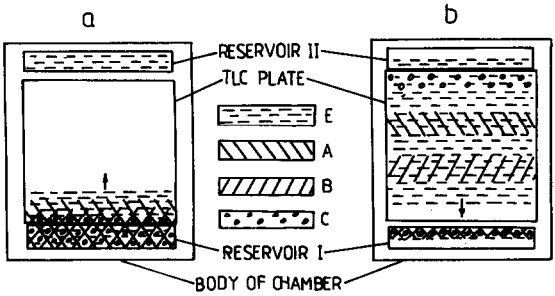

Fig. 6. Zonal application of the mixture using a twin chamber. A = 4-Chloro-4′-dimethylaminoazobenzene; B = 2-nitroaniline; C = 1-(4-hydroxyphenylazo)-2-naphthol; E = toluene (eluent).

chromatographic plate with the wide starting zone was then turned through 180° and developed with the eluent from the second reservoir. Fig. 6b shows the situation on the plate in the last stage of development of the micropreparative chromatogram.

The construction of the chamber is simpler than that of other chambers of this type. Its depth is only 6 mm so that the manufacture of the chamber is simplified[13,14].

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