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Note

A simplified device for centripetal thin-layer chromatography

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Horizontal circular centripetal thin-layer chromatography was first described by Van Dyk[1] in 1969. In this method, which can be considered as a combination of thin-layer and column chromatography, the direction of elution is opposite to the one used in the more conventional centrifugal technique. The sample is applied on an outer circular start-line, and elution proceeds from this circle towards the centre, where the separated components are continuously collected. The centripetal technique has the advantage of a long start-line, which can easily be adapted to the requirements of a particular separation. Moreover, the separated products can be withdrawn from the centre in very small amounts of solvent (the concentration per unit surface area increases with decreasing diameter); this simplifies detection and is particularly useful in preparative chromatography.

EXPERIMENTAL

We have used the centripetal chromatographic technique for almost three years with good results. However, the apparatus[1] described by Van Dyk and used in our first experiments had certain drawbacks, which are probably the reason why this elegant method has hitherto not been put into general practice.

The main inconveniences of the original apparatus of Van Dyk are:

(1) the need for a special coating apparatus,

(2) the need for a special glass support disc,

(3) the method of sucking up the developing solvent by the wire gauze does not always work correctly,

(4) the upward withdrawal of developing solvent from the centre sometimes gives rise to an irregular flow of effluent containing air bubbles, which constitutes a handicap for automatic detection, and

(5) the glass filter may become clogged by fine particles of adsorbent.

Most of these drawbacks have been eliminated by the system developed in our laboratory, a schematic representation of which is shown in Fig. 1.

We use the common size glass plates (200 × 200 mm), coated with an adsorbent layer by any conventional commercial coating apparatus.

Before applying the sample solution with the aid of a turn-table (an old 78-rpm grammophone and an Agla micrometer syringe can be used) in the same way as described by Van Dyk[1], the plate is put upside down on the circular developing chamber

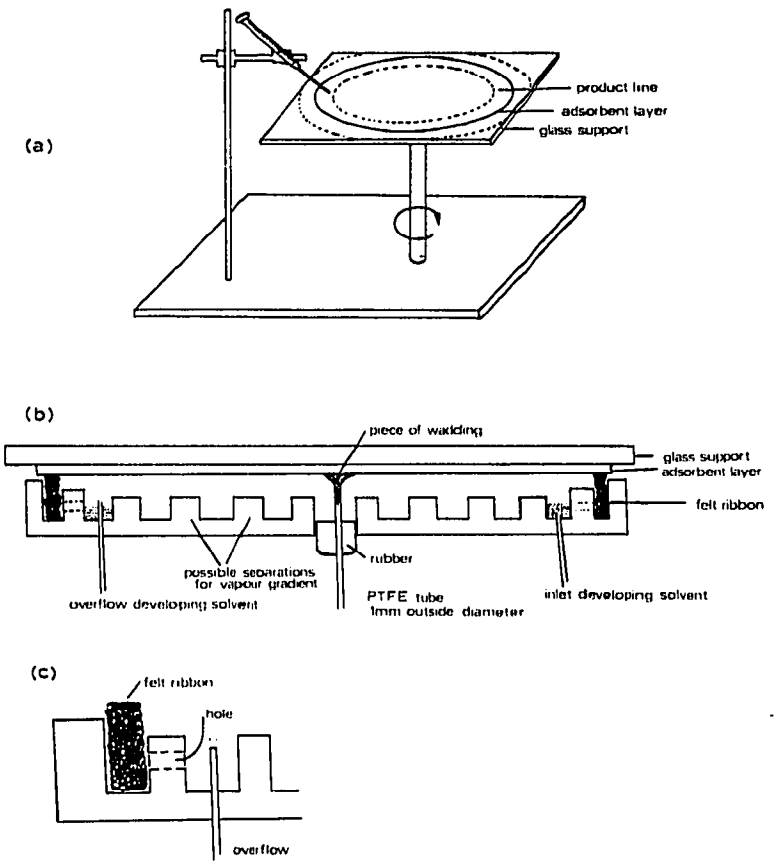

Fig. 1. Set-up of a new apparatus for centripetal thin-layer chromatography. (a) Turn-table; (b) cross-section of the developing chamber; (c) detail of the developing chamber.

and all adsorbent outside the circle is scraped off in order to prevent any condensation caused by evaporation of low-boiling developing solvents.

The developing chamber has a diameter of 190 mm, is made of anodised aluminium, and stands on three levelling screws. Instead of the wire-gauze system, we use a felt ribbon (*e.g.*, felt of S. S. Porrits-Spencer*) of thickness about 3 mm, pressed between the outside wall and a second separation wall (see Fig. 1); the felt ribbon is about 1 mm higher than the outside wall. In the second separation wall, holes are drilled at regular distances to permit the mobile phase to penetrate into the felt ribbon. The felt is cleaned before use by extraction with dichloromethane, methanol and water.

The solvent-withdrawal system consists of a PTFE tube (I.D. 0.5 mm) fitted in a hole in the centre of the developing chamber by means of a rubber stopper (*e.g.*, the stopper of a Drummond Microcap). At the end of the tube, a piece of cotton wool is attached, part of which (about 5 mm) is pressed into the tube and part of which

* Manufactured by Richert, Goede Koningstr. 7, 1150 Brussels, Belgium.

emerges as a small (about 1 mm) plug. The tube is previously filled with developing solvent so as to obtain an immediate flow at the start of withdrawal of the mobile phase.

When the mobile phase arrives at the centre, the filled tube is placed in position, while the piece of cotton wool is gently pressed against the adsorbent layer. From this moment, the developing solvent starts to flow (in a constant stream, without air bubbles) as a result of capillary action and gravity forces. The flow-velocity varies from 0.1 ml to 5 ml per h depending on the thickness of the layer, the composition of adsorbent and the nature of the mobile phase.

In order to keep the layer from drying out, enough solvent is supplied to the developing chamber to maintain a small overflow. Elution can be made in one run or may be interrupted. In the latter instance, the layer can be observed under UV radiation and dried at room temperature, and the elution can be re-started. The entire system must be protected from draughts (*e.g.*, by a plastic housing), otherwise the migrating circles formed by the different components will not remain concentric.

CONCLUSION

With this improved apparatus, we have obtained excellent results. We have found that the method is particularly suited to the separation of very small amounts of impurities in a product and their subsequent identification by mass spectrometry (fractions of about 1 mg suffice) and other identification techniques.

ACKNOWLEDGEMENT

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REFERENCE

1 J. H. Van Dyk, *Proceedings of the 5th International Symposium on Separation Techniques, Lausanne*, 1969. *1969; Chimia*, 24 (1970) 234.