

- 1 G. DIJKSTRA AND J. DE GOEY, in D. H. DESTY (Editor), *Gas Chromatography 1958*, Academic Press, New York, London, 1958, p. 56.
- 2 M. J. E., GOLAY, in D. H. DESTY (Editor), *Gas Chromatography 1958*, Academic Press, New York, London, 1958, p. 67.
- 3 R. KAISER, *Gas Phase Chromatography. Vol. II. Capillary Chromatography*, Butterworths, London, 1963.
- 4 F. FAIRBROTHER AND A. E. STUBBS, *J. Chem. Soc.*, (1935) 527.
- 5 G. I. TAYLOR, *J. Fluid Mech.*, 10 (1961) 161.
- 6 H. L. GOLDSMITH AND S. G. MASON, *J. Colloid Sci.*, 18 (1963) 237.
- 7 K. D. BARTLE, L. BERGSTEDT, M. NOVOTNÝ AND G. WIDMARK, submitted for publication.
- 8 M. NOVOTNÝ, L. BLOMBERG AND K. D. BARTLE, in preparation.

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A simple device for descending preparative thin-layer chromatography

Several devices for descending and ascending preparative thin-layer chromatography have been described recently¹⁻¹². Those which are designed for preparative chromatography using "normal" (200 × 200 mm) glass plates collect fractions by methods which either expose the substances to the atmosphere to some extent (involving scraping off the adsorbent layer), or do not completely separate them (involving washing out the fractions from the bottom of the chamber). Where the adsorbent containing the desired compound is removed and extracted with a suitable solvent, the extract usually contains fine particles of the adsorbent which interfere with spectroscopy. The complete removal of these particles by filtering or centrifuging is difficult and, further, the solvent will also be contaminated with small amounts of organic compounds present in most commercial adsorbents^{13,14}.

The apparatus shown in Fig. 1 has been constructed to avoid these disadvantages. It uses materials commonly available in most chemical laboratories and requires only simple skills for its construction. The apparatus has been designed for "normal" thin-layer glass plates (200 × 200 mm) but slight changes in design will permit the use of other sizes and types of plate.

The eluent transfer roll A, which rests on an open stainless steel frame E, and the adsorbent layer B attached to the stainless steel plate C, are composed of gypsum and glass powder (mixed 1:2 w/w), but other materials such as gypsum and finely ground sand or gypsum and Celite have also been shown to be suitable. The eluent roll was cast using a split teflon tube as a mould and has a glass rod for its axis. The stainless steel plate is bent in two and hangs over a glass rod J. Its front surface holds the adsorbent layer B and the plate was sandblasted to improve the adhesion of this layer. The front surface is cut in triangular shape to reduce the surface area of adsorbent and to provide a lowest point for fraction collection. The adsorbent layer can be trimmed back to reduce its surface still further.

Fractions are collected by tiny glass capillaries brought into contact with the lowest point of the adsorbent layer B. The length and diameter of the glass capillaries are rather critical. For organic solvents a minimum length of 70 mm and a maximum

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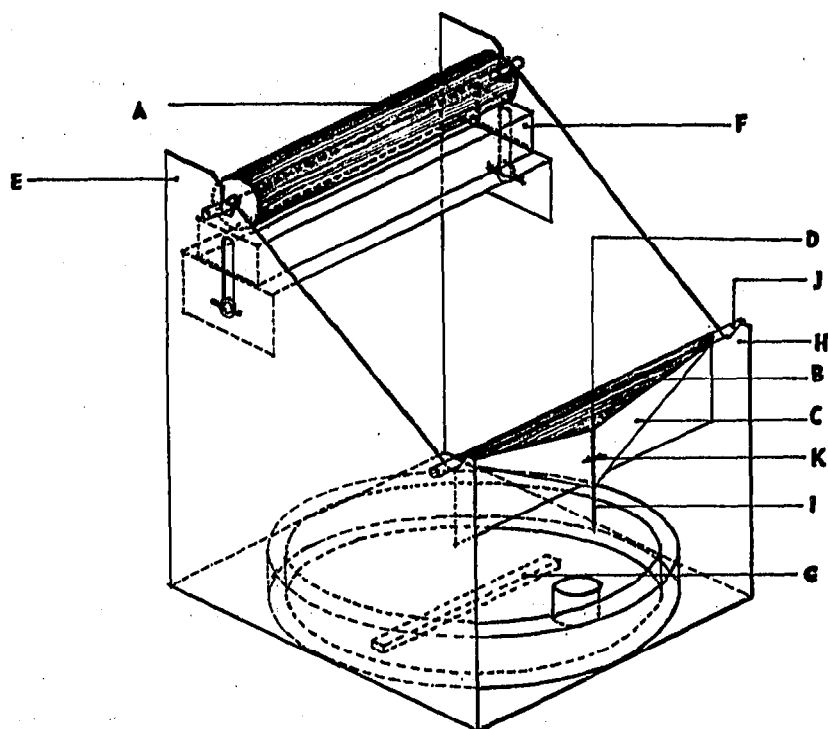


Fig. 1. A sketch of the device without thin-layer plate. For explanation of letters, see text.

inner diameter of 0.15 mm are needed. To protect these tiny capillaries, three to five of them are inserted in a melting point tube I, held in position by the weak stainless steel spring K. Trough F is made of glass but any other resistant material could be used. If the sorbent layer on the glass plate is thick and as a consequence a richer eluent supply is desired, a cotton flannel loop around the roll A dipping into the eluent trough can be used. The resistance of the material to the solvents used should, of course, be checked.

Fractions are collected in a series of small beakers placed in a glass dish floating on mercury inside a slightly larger dish. We found a large culture dish or moist chamber, 140 mm diameter and 40 mm deep, suitable for this purpose. The fraction collector is turned by an external magnet acting on the plastic covered bar magnet G placed in the inside dish. If the fraction collector is very light, it should be possible to use eluent instead of mercury to support the inner dish, but as far as we could ascertain, the mercury vapour did not interfere with the developing procedure at room temperature.

The coated glass plate is placed face down, resting on roll A and on the coated stainless steel plate C and held in position by the lugs H. The angle of rest of the glass plate does not appear to be critical. We were unable to confirm the statement of REISERT AND SCHUMACHER¹⁰ that a steep slope in itself causes irregular development.

To view the spots we irradiated the adsorbent layer with a mercury lamp covered with a Wood's glass screen. Most commercial fluorescing adsorbents are prepared for irradiation with short wavelength UV light (254 m μ), but we found such light less suitable than longer wavelengths for most cases since it cannot penetrate the glass walls of the developing chamber, it destroys some sensitive substances and it is ab-

sorbed by many solvents, etc. We therefore prepared plates containing a material (about 1% w/w) which fluoresces at 365 m μ , such as Riedel de Haen Leuchtstoffe Grun N, Vanino's pigment and salts of 3,5-dihydroxypyrene-8,10-disulphonic acid¹⁵. In the few cases where the spots to be visualized did not absorb 365 m μ UV-light, we used 254 m μ sensitized plates, but in these cases the choice of eluents is restricted (must be non-absorbers for the actual wavelength) and special arrangements must be made to allow the light to enter the chamber.

No difficulty should be experienced in collecting fractions provided that the outlet capillaries are of suitable length and diameter. To avoid the eluent creeping between the capillaries and the stainless steel plate, the capillaries were inserted in a melting point tube about 10 mm shorter than the tiny capillaries in such a way that the melting point tube did not touch the eluent-carrying coated layer.

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- 1 A. MEHLITZ AND T. MINAS, *Chemiker Ztg.*, 90 (1966) 67.
- 2 J. H. VAN DIJK, *Z. Anal. Chem.*, 236 (1968) 326.
- 3 E. A. MISTRYUKOV, *J. Chromatog.*, 9 (1962) 311.
- 4 B. G. JOHANSSON AND L. RYMO, *Acta Chem. Scand.*, 16 (1962) 2067.
- 5 J. G. KIRCHNER, J. M. MILLER AND G. J. KELLER, *Anal. Chem.*, 23 (1951) 420.
- 6 W. L. STANLEY AND S. H. VANNIER, *J. Assoc. Offic. Agr. Chemists*, 40 (1957) 582.
- 7 M. K. SEIKEL, M. A. MILLETT AND J. F. SAEMAN, *J. Chromatog.*, 15 (1964) 115.
- 8 L. BIRKOFER, C. KAISER, H. A. MAYER-STOOL AND F. SUPPAN, *Z. Naturforsch.*, 17 B (1962) 352.
- 9 L. GÖLDEL, W. ZIMMERMANN AND D. LOMMER, *Z. Physiol. Chem.*, 333 (1963) 35.
- 10 P. M. REISERT AND D. SCHUMACHER, *Experientia*, 19 (1963) 84.
- 11 N. ZÖLLNER AND G. WOLFRAM, *Klin. Wochschr.*, 40 (1962) 1098.
- 12 H. HALPAAP, *Chem. Ingr. Tech.*, 35 (1963) 488.
- 13 F. GEISS, A. KLOSE AND A. COPET, *Z. Anal. Chem.*, 211 (1965) 37.
- 14 J. L. MULDER AND G. J. VEENSTRA, *J. Chromatog.*, 24 (1966) 250.
- 15 R. TSCHESCHE, G. BIERNOTH AND G. WULFF, *J. Chromatog.*, 12 (1963) 342.

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The separation and detection of several mycotoxins by thin-layer chromatography

The study of mycotoxicoses¹ especially aflatoxicosis^{2,3} emphasized the existence of fungal metabolites harmful to higher organisms. This evidence and the ability of various ubiquitous fungi, e.g. *Aspergillus flavus*³ and *Penicillium islandicum*⁴ to elaborate potent carcinogens prompted theories on a possible relationship between the consumption of mycotoxins and diseases of unknown etiology, e.g. the high incidence of hepatocarcinogenicity in Africa⁵. It is therefore essential that rapid and sensitive analytical methods be developed for the detection of these hazardous compounds in

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