

Notes

CHROM. 4398

Dependence of film thickness on column radius and coating rate in preparation of capillary columns for gas chromatography

Mainly because of its simplicity, the dynamic coating procedure¹ for the preparation of capillary columns for gas chromatography has been widely used, and preferred to the static version originally proposed by GOLAY². KAISER³ studied the dependence of the average thickness of the stationary phase layer, d_F , on the operating conditions during dynamic coating. He reported the following relation between d_F , the % v/v concentration of the phase in hexane solution, c , the velocity of the coating plug, u , and the inner radius of the capillary, r :

$$d_F = \frac{c}{100} \frac{1}{2r} (0.265 u + 0.25) \quad (1)$$

The wetting of capillaries was, however, studied by FAIRBROTHER AND STUBBS⁴ in connection with electro-osmotic measurements as early as 1935, and their conclusions have been more recently confirmed by other authors^{5,6}. In contrast to eqn. (1), the FAIRBROTHER-STUBBS equation, adjusted to our case, relates d_F directly to the column radius and to the square root of the coating rate:

$$d_F = \frac{c}{100} \frac{r}{2} \sqrt{u \frac{\eta}{\sigma}} \quad (2)$$

where η and σ are the viscosity and surface tension of the coating solution, respectively.

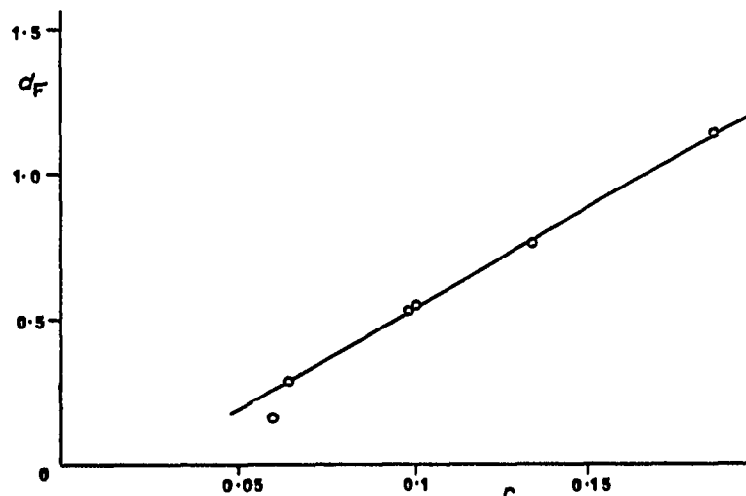


Fig. 1. Graph of film thickness, d_F (μ), against column radius, r (mm), for vapour-phase silanised Pyrex glass capillary columns coated with a 10% v/v solution of SF-96 in toluene at 5 mm/sec.

We have been recently concerned with the reproducible preparation of highly efficient glass capillary columns for the analysis of tobacco smoke⁷. In order to establish a reliable technique for the preparation of columns of controlled efficiency and capacity, some factors affecting their coating were investigated in work to be described elsewhere⁸. A study of the dependence of d_F on r and u (Figs. 1 and 2) showed that eqn. (2) is indeed applicable to the coating of capillary columns.

The FAIRBROTHER-STUBBS equation also has the advantage of being dimensionally correct. Moreover, the dependence of d_F on the viscosity and surface tension of the coating solution is presumably responsible for the so-called "solvent-polarity effect" suggested by KAISER³.

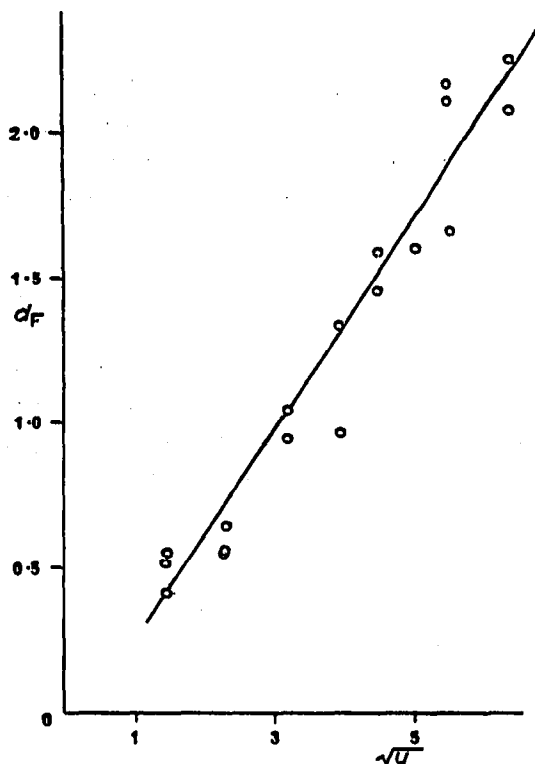


Fig. 2. Graph of film thickness, d_F (μ), against square root of coating velocity u (mm/sec) for 0.2 mm I.D. vapour-phase silanised Pyrex glass capillary columns coated with a 10% v/v solution of SF-96 in toluene.

TAYLOR has shown⁵ that the FAIRBROTHER-STUBBS equation is valid only if the product $u\eta/\sigma < 0.09$. In fact, this holds for most solutions of stationary liquids used for the coating of capillary columns, but for more viscous solutions (*e.g.*, of SE-30 or OV-1 silicone rubbers) some correction factor may be necessary.

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- 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.

Received September 25th, 1969

J. Chromatog., 45 (1969) 469-471

CHROM. 4403

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

J. Chromatog., 45 (1969) 471-473