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Letter to the Editor

Static coating methods for glass capillary columns at elevated temperatures

Sir,

The continuing discussion of column preparation techniques demonstrates that more laboratories prepare their own columns than one tends to think. Today's techniques of column preparation have been developed to such perfection that they are both rapid and reliable.

Recently, Huang and Sun[1] reported on their experience with static coating at high temperature, for which we prefer the expression "free release static coating", introduced by Xu and Vermeulen[2]. I would like to add some of our experience using the same technique.

I agree with the above authors that short-term temperature stability of the heating bath is important as any relatively rapid temperature changes cause expansion or contraction of the coating solution and, as a consequence, an irregular withdrawal of the meniscus and an uneven thickness of the stationary phase film. On the other hand, slow temperature changes may be fairly drastic (many degrees) without affecting the column performance significantly. The authors suggest using a very precise thermostat, maintaining the temperature to within ±0.05°C. We prefer a simpler system, in which the column is immersed in an only indirectly thermostated water-bath, *i.e.*, a tank of water situated in a thermostated bath. Heat transfer to the column is slow in this way, damping temperature changes in the thermostated bath. Even a thermostat of very moderate quality is good enough to give a smooth withdrawal of the meniscus.

Both Xu and Vermeulen[2] and Huang and Sun[1] reported the use of acetone in the coating solution. We hesitate to use acetone because of the observation that silicone columns suffer an increased bleeding rate. Therefore, we prefer the conventional dichloromethane–pentane mixture suggested by Kong and Lee[3] and Grob and Grob[4] or dichloromethane–isopentane mixtures.

Huang and Sun[1] mentioned a reduced coating speed if fairly concentrated coating solutions were used, but concentrations of coating solutions only up to 0.8% were tested. We observed a rather drastic reduction in the coating speed with a further increase in the concentration of the stationary phase. In fact, the speeds of solvent evaporation hardly exceeded those obtained by the classical static coating involving application of a vacuum. Further, we experienced a high risk of bumping (breakthrough) when using concentrated coating solutions, prompting us to return to the conventional method of static coating[5] at least for these applications.

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REFERENCES

1 X. B. A. Huang and Y. Sun, *J. Chromatogr.*, 362 (1986) 121.
2 B. Xu and N. P. E. Vermeulen, *Chromatographia*, 18 (1984) 520 and 642.
3 R. C. Kong and M. L. Lee, *J. High Resolut. Chromatogr. Chromatogr. Commun.*, 6 (1983) 319.
4 K. Grob and G. Grob, *J. High Resolut. Chromatogr. Chromatogr. Commun.*, 8 (1985) 856.
5 K. Grob, *Making and Manipulating Capillary Columns for Gas Chromatography*, Hüthig, Heidelberg, 1986.

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