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Note

An ultrasonic method for producing graphite-coated glass capillary columns

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A simple procedure for coating the inner wall of glass capillary columns with a graphite layer is reported. The procedure consists in coating columns essentially by the dynamic method[1] using a colloidal graphite solution. This solution, which is stable for several days, is obtained by submitting a suspension of graphite (Sterling MT) in dichloromethane to ultrasonic waves, produced from a magnetostrictor as suggested by Crawford[2]. After the removal of the solvent, the graphite layer remaining on the wall becomes visible.

The graphite-coated capillary columns readily permit the use of a wide range of stationary phases. These columns therefore do not suffer the main disadvantage observed when the carbon layer is obtained by the method according to Grob[3], such columns being suitable only for a restricted range of moderately polar phases[4]. Apart from this aspect, Grob's columns require some critical steps in their preparation (speed of pyrolysis, uniform heat transfer, etc.) and are deactivated by moisture[5]. More sophisticated methods for the treatment of inner glass walls have been described (for a review see ref. 5), but according to Novotný[6] a universal method has not yet been found.

Although research is yet at an early stage, our graphite-coated capillary columns have the following properties.

(1) The layer is not subject to mechanical or chemical disturbances. We did not observe any blockage when the columns were subjected to repeated washing with polar and non-polar solvents. Hence a single column can be used for different stationary phases.

(2) The columns were coated with phases of different polarity (SE-30, SE-52, Dexsil, Carbowax 20M, PMPE and Apiezon L) and, although no special care was taken with the coating procedure, very high efficiencies were observed. Typical results are given in Table I.

TABLE I

CHARACTERISTICS OF SOME GRAPHITE COATED GLASS CAPILLARY COLUMNS

I.D. of columns = 0.5 mm.

Column number	1	2	3	4
Liquid phase	Apiezon L	SE-52	SE-30	Carbowax 20M
Column length (m)	14	4	16	13
Number of effective theoretical plates	20,000*	4000**	16,500**	13,800*
HETP (mm)	0.70	1.00	0.97	0.94
$\bar{u}$ (cm/sec)	6.8	10.0	9.0	7.5

* For methyl elaidinate (oven temperature 150°).

** For *n*-hexadecane (oven temperature 120°).

It should be added that coating can be carried out using any solid material that is capable of giving a colloidal solution by ultrasonics; in the first instance graphite was chosen because, according to Brodasky[7], it seems to be an excellent support material for gas–liquid chromatography. The method, in general, can be used to obtain coated open tubular capillary columns suitable for both gas–liquid and gas–solid chromatography. Capillary columns coated with the required material by ultrasonics could find applications in liquid chromatography also, as observed with alkali-treated glass capillary columns[8].

EXPERIMENTAL

The capillary columns were drawn out by the method of Desty *et al.*[9]. The colloidal graphite solution was obtained by irradiating a 1% solution of Sterling MT carbon black (Cabot Corp., Boston, Mass., U.S.A.) in methylene chloride for 15 min in the probe (Model L667) of an ultrasonic generator (Model MSE-386, Measuring & Scientific Equipment Ltd., London, Great Britain), using waves of 24 kHz frequency and 2 μ amplitude. The colloidal graphite was introduced into the capillary by applying a reduced pressure to one end by a water pump. When the column had been filled, the reservoir was removed and suction was continued so as to empty completely the capillary. The solvent was finally removed by a stream of nitrogen. The appropriate liquid film was then deposited by the dynamic procedure[1]. Experiments were carried out using a Carlo Erba (Milan, Italy) Model 2400 gas chromatograph equipped with a flame ionization detector.

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