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Note

Use of Aerosil (200 and R-972) in the preparation of stable support-coated open tubular columns

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The higher sample capacity of support-coated open tubular (SCOT) columns and the stability of the liquid film deposited on the porous layer compared with standard wall-coated open tubular (WCOT) columns have increased notably their use in capillary gas chromatography (GC). These advantages permit their use for trace analysis, as the minimal detectable amount is generally 1–2 orders of magnitude greater and their operation at high temperatures, without a significant increase in phase bleeding, recommends their use for coupled systems, *e.g.*, GC–mass spectrometry or GC–Fourier transform IR spectroscopy. Although these advantages are obtained with non-polar or moderately polar stationary phases, some problems have been encountered in the preparation of polar SCOT columns by either the static or the dynamic method[1,2]. Many solid supports and procedures have been reported for overcoming this problem, including the use of modified polymers[3], carbonaceous materials[4–7] and siliceous materials such as Silanox 101[8–11], Aerosil 380[12], Cab-O-Sil[13,14] and Chromosorb R-6740[15].

This paper reports the results of the use of two siliceous materials, Aerosil 200 and Aerosil R-972, for the preparation of SCOT columns by static and dynamic methods. A group of stationary phases covering a wide range of polarity, SE-30 silicone gum (low polarity), OV-17 silicone fluid (medium polarity) and polyethylene glycol 20M (high polarity), were used in the procedures. The results demonstrate the possibility of using these siliceous materials as solid supports for the preparation of efficient and thermostable SCOT columns.

EXPERIMENTAL

Coating suspensions

The coating suspensions were prepared in the proportion of 1.8 vol.-% of the solid support and 3.5 vol.-% of the stationary phase in chloroform (Lachema, Brno, Czechoslovakia) dried over a molecular sieve. For the OV-17 phase the amount of

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support was increased to 3.5 vol.-%. The suspensions were sonicated in a water-bath at 25°C until they appeared stable (approximately 10–15 min). Aeros il 200 and Aerosil R-972 were purchased from Degussa (Frankfurt am Main, F.R.G.), SE-30 from Carlo Erba (Milan, Italy) and OV-17 and Carbowax 20M from Applied Science Labs. (Palo Alto, CA, U.S.A.).

Coating procedures

Static[16] and dynamic coating[17] were performed in a single-step procedure. In the former method the capillary end was sealed with epoxy resin at high pressure (15–20 atm)[18] and was used for the preparation of OV-17 and Carbowax 20M columns. For dynamic coating the capillary was completely filled with the coating suspension, which was forced out by a mercury plug at a linear velocity of 0.5–1 cm/sec. A flow of dry nitrogen (10–15 ml/min) was passed through the capillary for 1 h at room temperature.

Column conditioning

Each column was fitted to a gas chromatograph (Fractovap, Model C; Carlo Erba) with a splitting ratio of 1:10–1:25 and an average linear velocity for the carrier gas of 50–120 cm/sec. A temperature programme from 50 to 170°C at 1.25°C/min was applied, then the oven was cooled and the programme repeated but up to the practical maximum temperature limit for each phase.

Column testing

The columns were tested with standard test mixtures, including that of Grob, xylene and butanol mixtures, gasoline and a synthetic perfume.

RESULTS AND DISCUSSION

Comparative data for the columns prepared by the static and dynamic methods using Aerosil 200 and Aerosil R-972 as solid supports are shown in Table I. Both methods gave similar results with regard to efficiency, although the static method

TABLE I

COMPARATIVE DATA FOR SCOT COLUMNS PREPARED WITH AEROSIL SOLID SUPPORTS

Parameter	*Column*					
	1	*2*	*3*	*4*	*5*	*6*
Length (m)	50	15	20	17	25	25
Inner diameter (mm)	0.40	0.35	0.35	0.35	0.40	0.30
Stationary phase	SE-30	SE-30	OV-17	20M	20M	20M
Solid support (Aerosil)	R-972	R-972	R-972	200	200	200
Coating method	D	D	S	S	S	S
Theoretical plates per metre (n/L)	2021	1895	1052	1915	2160	3088
Partition ratio (k) at 100°C	5.8[a]	4.1[a]	4.4[b]	2.3[b]	1.9[b]	2.0[b]
Utilization of the theoretical efficiency (%)	60	60	30	60	60	62.5

gave a greater (1–12%) number of theoretical plates per metre. This comparison is discussed elsewhere[18].

The test chromatograms showed good resolution and no peak tailing. No significant phase bleeding occurred with any column, with either temperature programming or high temperatures, showing the good properties of Aerosil 200 and Aerosil R-972 as solid supports in SCOT columns, although these columns should be tested in long-term routine work.

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