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Note

Use of glass beads in gas chromatographic analysis of drugs

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In previous papers we have reported that gas chromatographic columns packed with glass beads coated with a thin layer of carbon[1] or sodium dodecylbenzenesulphonate (SDBS)[2] have characteristics superior to those of untreated glass beads. The layer of carbon or of SDBS, by increasing the surface wettability, reduces the "pudding effect" responsible for limited efficiency. These modifications do not reduce the surface activity sufficiently to analyse drugs with non-polar stationary phases which contain not only an amino group but also an acid or alcoholic hydroxyl group or both.

This paper describes the preparation and use of gas chromatographic columns packed with soda-lime glass beads coated with a thin layer of SDBS and successively with layers of two stationary phases, the former a polar one such as Carbowax 20M or FFAP, the latter a non-polar one such as Apiezon L or SP 525.

EXPERIMENTAL

Soda-lime glass beads, commercially available (Analabs, North Haven, Conn., U.S.A.) were sieved and the 70–90 mesh fraction was used. The beads were washed successively with water plus detergent, water alone, acetone, and diethyl ether, and dried in an oven at 120°. The SDBS-coated glass beads were prepared as described previously[2] and were coated successively with two stationary phases. The first, Carbowax 20M or FFAP, was dissolved with methanol or methylene chloride. The second, Apiezon L or SP 525, was dissolved with chloroform. The SP 525 phase (Supelco, Bellefonte, Pa., U.S.A.) is more aromatic than Apiezon L.

RESULTS AND DISCUSSION

The glass microbeads prepared as described yield gas chromatographic columns sufficiently inert to polar and to weak acid or basic compounds. Fig. 1 shows the gas chromatograms of amphetamine and phenol obtained at 100 and 120°, respectively, with a 1.8 m glass column packed with 70–90 mesh SDBS-coated glass beads and FFAP ($2 \cdot 10^{-2}$%, w/w) and SP 525 ($8 \cdot 10^{-2}$%, w/w) as the stationary phases. The amount of surfactant was $4 \cdot 10^{-2}$% (w/w). In this column the polar stationary phase reduces the peak tailing of the polar compounds. The optimum ratio between the support and polar stationary phase is not critical and depends on the mesh sizes of glass

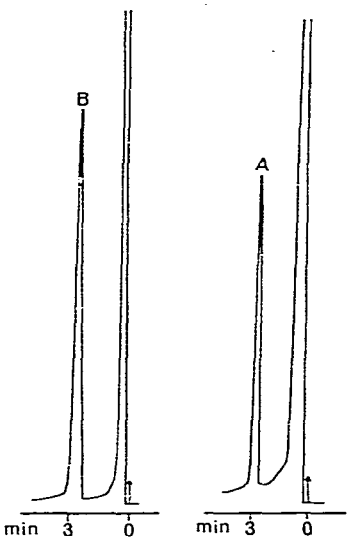

Fig. 1. Gas chromatograms of amphetamine (A) and phenol (B). Glass column 1.8 m × 0.3 cm I.D.; packing, 70–90-mesh glass beads coated with $4 \cdot 10^{-2}$% (w/w) of SDBS, $2 \cdot 10^{-2}$% (w/w) of FFAP and $8 \cdot 10^{-2}$% (w/w) of SP 525; column inlet pressure, 0.6 kg cm^{-2}; carrier gas, nitrogen; flame-ionization detector; column temperature, amphetamine 100°, phenol 120°; injector temperature, 150°.

beads. It was observed that for 70–90-mesh beads, ratios of 10^{-4} and $2 \cdot 10^{-4}$% (w/w) did not increase the specific retention volumes of the drugs measured using the non-polar stationary phase alone. The columns packed with FFAP as tailing reducer are thermally more stable than those packed with Carbowax 20M; it was observed that FFAP columns are stable at 250° whereas the Carbowax 20M columns are stable up to 230°. Apiezon L and SP 525 were selected as non-polar stationary phases

TABLE I

RELATIVE RETENTION TIMES (t_R) OF DRUGS

Temperature (°C)	*Drug*	t_R	*Temperature (°C)*	*Drug*	t_R
140	Phenmetrazine	0.082	240	*n*-Octacosane*	1.000
	Nicotine	0.192		Phenacetamide	1.084
	Prolintane	0.226		Diphenhydramine	2.184
	Ephedrine	0.342		Phenacetin	3.276
	Phencanphamine	0.411		Pyramidon	5.096
	Norephedrine	0.425		Caffeine	6.916
	Coramine	0.520		Procaine	7.826
	Methylphenidate	0.897		Pipradol	8.554
	Benzphetamine	0.972		Cocaine	10.92
	n-Docosane*	1.000		Metaqualone	13.83
				Heroin	15.47
195	Phendimetrazine	0.159		Morphine	26.66
	Bemegride	0.301		Papaverine	72.80
	Cardiazol	0.904			
	n-Tetracosane*	1.000			

* The retention times of normal aliphatic hydrocarbons were: *n*-docosane, 11.5 min; *n*-tetracosane, 5.0 min; *n*-octacosane, 0.9 min.

because of their high thermal stability and because they are the only low-polarity phases that yield columns of high efficiency with glass beads pre-treated with SDBS and Carbowax 20M or FFAP. The silicone gums, coasted on to SDBS-coated glass beads, are thermally less stable and, moreover, on SDBS-coated glass beads pre-treated with a tailing reducer they give a non-uniform layer.

Table I lists the retention times relative to normal aliphatic hydrocarbons, of the drugs analysed with the column shown in Fig. 1. The aromatic characteristics of SP 525 are responsible for the high retention times of those drugs with benzene rings, such as papaverine.

Fig. 2 shows the gas chromatogram of a pharmaceutical product containing phenacetamide, phenacetin, pyramidone and caffeine. Fig. 3 shows the chromatogram of heroin and morphine. Both of these chromatograms were obtained at 240° on a 1.6-m glass column packed with 70–90 mesh SDBS-coated glass beads with FFAP (0.02%, w/w) and SP 525 (0.06%, w/w) as stationary phases.

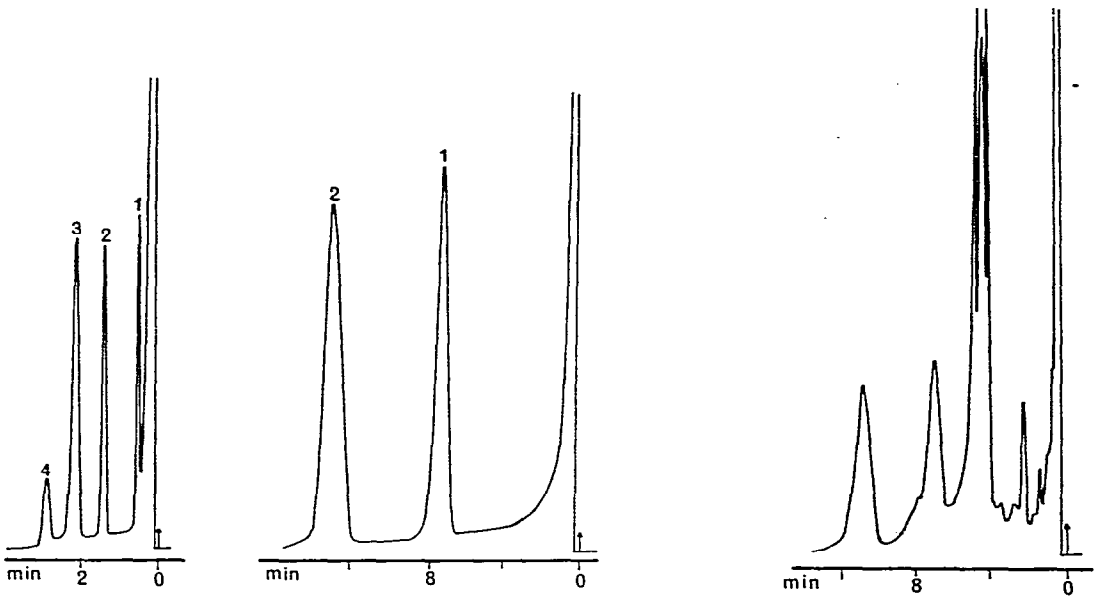

Fig. 2. Gas chromatogram of a pharmaceutical product containing phenacetamide (1), phenacetin (2), pyramidone (3), and caffeine (4). Glass column 1.6 m × 0.3 cm I.D.; packing, 70–90-mesh glass beads coated with $4 \cdot 10^{-2}$ (w/w) of SDBS, $2 \cdot 10^{-2}$% (w/w) of FFAP and $6 \cdot 10^{-2}$% (w/w) of SP 525; column inlet pressure, 0.5 kg cm^{-2}; carrier gas, nitrogen; flame-ionization detector; column temperature 240°; injector temperature, 250°.

Fig. 3. Gas chromatogram of heroin (1) and morphine (2). Conditions as for Fig. 2.

Fig. 4. Gas chromatogram of a variety of hashish. Conditions as for Fig. 2.

Fig. 4 shows the chromatogram of a variety of hashish analysed at 240° using the column containing FFAP ($2 \cdot 10^{-2}$%, w/w) and SP 525 ($8 \cdot 10^{-2}$%, w/w) as stationary phases.

REFERENCES

1 L. Zoccolillo and A. Liberti, *J. Chromatogr.*, 77 (1973) 69.
2 L. Zoccolillo and F. Salomoni, *J. Chromatogr.*, 106 (1975) 103.