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Note

Glass columns with high resistance to internal pressure for high-performance liquid chromatography

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Most columns used in high-performance liquid chromatography (HPLC) are made of precision-bore stainless-steel tubing. However, these columns have some distinct drawbacks: (i) the inner surface of stainless-steel tubes is not sufficiently smooth, although it is often additionally polished, and this can impair the separation efficiency when microparticulate sorbents (mean particle diameter, $d_p \leqslant 10\,\mu m$) are used[1,2]; (ii) in some applications stainless steel is not sufficiently inert either to some sensitive solutes or to aggressive mobile phases employed[3]; (iii) the quality of the packing, its uniform coverage by a stationary phase (and in some cases also the passage of chromatographic zones) cannot be followed visually. Obviously, glass columns would be preferable in all these respects, but their use in HPLC is limited by their rather low resistance to internal pressure. Commercially available columns made of heavy-wall glass tubing can withstand pressures up to *ca.* 5 MPa and cannot be therefore slurry-packed by microparticulates. Glass-lined metal columns can be used[4] at higher pressures, but are rather expensive.

This note describes glass columns for HPLC with enhanced pressure resistance which can be slurry-packed and operated at pressures up to 30 MPa.

EXPERIMENTAL

The columns[5] were made of heavy-wall glass tubing (borosilicate glass SIAL, Kavalier, Sázava), nominal I.D. 4 mm, nominal O.D. 9 mm, in lengths of 150 or 300 mm. The design of the column and end-fittings is shown schematically in Fig. 1. The retaining nut (2) is fitted to a flange on the end of glass column (1) by means of an elastomer ring (3). The packing is retained in the column by stainless-steel gauze (7), sealed by means of PTFE disc (5) provided by a central hole for the stainless-steel capillary (6). By tightening the fixing nut (4), the seal (5) is pressed against the rim of the column, and the ring (3) that prevents the glass coming into direct contact with the metallic nut is elastically deformed and ensures permanent tightness.

The pressure resistance was enhanced by chemical reinforcement of the glass by diffusion of potassium ions into the surface layer[5]; this treatment does not affect the chemical inertness of the glass and is stable for a long time.

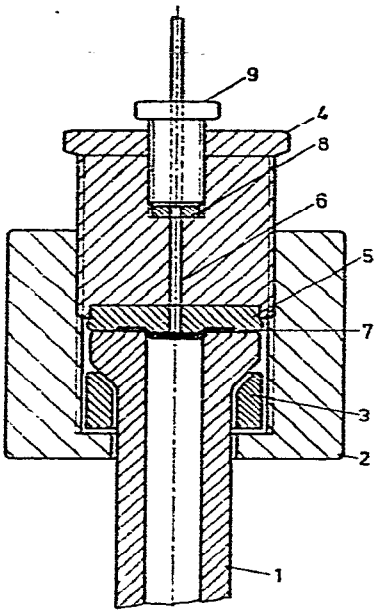

Fig. 1. Design of column end-fittings. 1 = Glass column; 2 = retaining nut; 3 = elastomer ring; 4 = fixing nut; 5 = PTFE disc; 6 = outlet (inlet) stainless-steel capillary; 7 = gauze; 8 = seal; 9 = fixing screw.

In testing the pressure resistance of empty columns, a syringe-type positive displacement pump of our own design[6] was used for non-treated columns, and a home-made reciprocating membrane pump (200 strokes per minute) was employed with the chemically reinforced columns. (The latter arrangement corresponds more closely to the conditions prevailing in the slurry-packing.) One column was also tested with a manual hydraulic pump (*ca.* 5 strokes/min). In all cases the tested column provided with the end-fittings shown in Fig. 1 was connected to the pump by means of a stainless-steel capillary (0.5 mm I.D.), and then filled with the solvent before its outlet was blanked off. Prior to testing or to chemical reinforcement all columns were kept at 450° for 24 h in order to remove internal stress.

Non-treated columns were pressurized to destruction. Chemically treated columns were first pressurized to 50 MPa; after 2 min the pressure was increased until either the column was destroyed or the upper pressure limit of the pump was reached. The maximum pressure of the membrane pump was 75 MPa; if the column was not destroyed, the pressure was reduced and the test repeated.

RESULTS AND DISCUSSION

The results of all tests are summarized in Table I. The non-treated columns were destroyed at pressures between 12 and 16 MPa, in accord with results obtained previously[7] on the same type of glass. Accordingly, they cannot be used in HPLC where the pressures required to pack a microparticulate sorbent to a stable bed are much higher.

TABLE I

EFFECT OF CHEMICAL REINFORCEMENT ON RESISTANCE OF GLASS COLUMNS

Column number	*Surface treated*	*I.D. (mm)*	*O.D. (mm)*	*Length (mm)*	*Pressure at destruction (MPa)*	*Note*
1	No	4.0	9.0	300	13.0	Columns 1–5 tested with syringe-type pump
2	No	4.1	9.2	300	12.0	
3	No	3.9	9.0	300	16.0	
4	No	4.1	9.4	300	15.0	
5	No	3.8	9.0	150	12.0	
6	Yes	4.0	9.1	300	84.0	No destruction at 840 bar; tested with manual pump
7	Yes	3.8	8.8	300	75.0	No destruction at 750 bar; columns 7–12 tested with membrane pump
8	Yes	3.9	9.0	300	61.0	
9	Yes	4.2	8.8	150	75.0	Not destroyed at 750 bar; in repeated pressurization destruction after 30 sec at 75 MPa
10	Yes	4.3	9.2	300	67.0	
11	Yes	4.2	8.8	150	60.0	
12	Yes	4.0	9.0	300	75.0	Not destroyed at 750 bar; in repeated pressurization destruction after 30 sec at 75 MPa

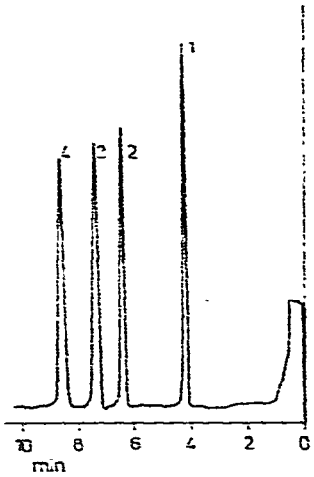

Fig. 2. Normal-phase chromatogram of a test mixture on a glass column (300 × 4 mm I.D.) packed with spherical silica ($d_p \approx 8\,\mu m$). Eluent, heptane with 0.1% propan-2-ol; flow-rate 1 ml/min; ΔP = 5.2 MPa; UV detection, 254 nm. 1 = Biphenyl (plate number N = 9400); 2 = nitrobenzene (N = 11,600); 3 = ethyl benzoate (N = 9100); 4 = benzil (N = 11,200).

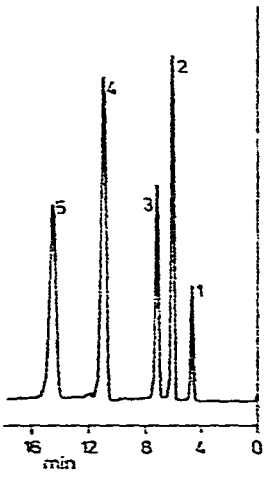

Fig. 3. Reversed-phase chromatography of a test mixture on a glass column (300 × 4 mm I.D.) packed with ODS silica ($d_p \approx 10\,\mu m$, 20.1% carbon). Eluent, methanol–water (80:20); flow-rate 0.83 ml/min; ΔP = 6.5 MPa; UV detection, 254 nm. 1 = Phenol (N = 5600); 2 = nitrobenzene (N = 5200); 3 = benzene (N = 5400); 4 = naphthalene (N = 5100); 5 = biphenyl (N = 5300).

All chemically reinforced columns withstood the pressure of 50 MPa, and the pressure at destruction was never lower than 60 MPa. The somewhat higher resistance of column 6 (see Table I) could have been due to the different mode of pressurization: in all cases the pressure was measured by a Bourdon gauge, and with the high-speed membrane pump the peak values of pressure can be considerably higher owing to the delay action of the manometer.

It is evident from the data in Table I that the pressure resistance of chemically reinforced columns satisfies the requirements of modern HPLC. These columns packed with microparticulate spherical silica[8] (Laboratory Instruments Works, Prague, Czechoslovakia; trade mark Separon SI VSK) have been in constant use in our laboratory for two years; they are slurry-packed by standard techniques[4] at a pressure of 30 MPa. As an example of their possibilities and of the separation efficiency routinely achieved, a chromatogram of a test mixture is given in Fig. 2 as obtained on a glass column packed with spherical silica (mean particle diameter $d_p \approx 8\ \mu m$); Fig. 3 gives another example of reversed-phase separation on a similar column packed with spherical silica ($d_p \approx 10\ \mu m$) modified by octadecyl bonded phase.

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