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GLASS COLUMNS WITH A SEPTUMLESS INJECTOR FOR HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

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SUMMARY

A construction of columns made from thick-walled glass tubes is described. The joints between the glass column and metal head are deformable. An injector is built into the column head. These columns were used at pressures up to 10 MPa and temperatures up to 80°.

INTRODUCTION

The use of hard glass as a column material in high-performance liquid chromatography (HPLC) has many advantages. Its surface is very smooth and chemically resistant and the columns are transparent, so that it is easy to follow mechanical compression of column bed and any changes in its colour. However, these advantages are offset by the fragility of glass. Because glass has a relatively low expansion coefficient, it is difficult to make a pressure-tight seal between stainless steel and glass.

A successful construction of glass columns was described by Stahl and Schuppe[1,2]. They used glass tubes of I.D. 2.3 mm and O.D. 9 mm, sealed to the stainless steel outlet and inlet parts by Viton O-rings and cylindrical inserts made from Kel-F. Both ends of the column were held together by a stainless-steel tube, surrounding the whole column and acting as a pressure shield. The pressurized eluent not only flows through the column but also surrounds the glass column, permitting pressures up to 30 MPa to be attained.

Tesařík and Kaláb[3] tested glass tubes for use as columns in liquid chromatography, and found that pressures up to about 60 MPa are necessary for their rupture. They characterized the tension in pressurized tubes by a dimensionless constant, A:

$$A = \frac{(D/d)^2 + 1}{(D/d)^2 - 1}$$

where D is the outer and d the inner diameter of the tube. If A is less than *ca.* 1.55, the tube should be suitable for application in liquid chromatography.

EXPERIMENTAL

We used glass tubes with I.D. 3.5 mm and O.D. 10 mm, made from SIAL glass, with $A = 1.28$.

In the first construction, the ends of tubes were cemented to brass or stainless-steel heads with epoxy resin. After several weeks of daily use the glass broke, usually at some distance from the seal. This effect was not overcome by temperature annealing the tubes and fire-polishing their ends, presumably because of tension in the glass due the difference in the thermal expansion coefficients of metal and glass.

Our aim was to construct a column that could be operated from room temperature to about 80°. Therefore, we had to avoid any firm joint between metal and glass. Further, in order to decrease the void volumes, we incorporated the injector into the column head.

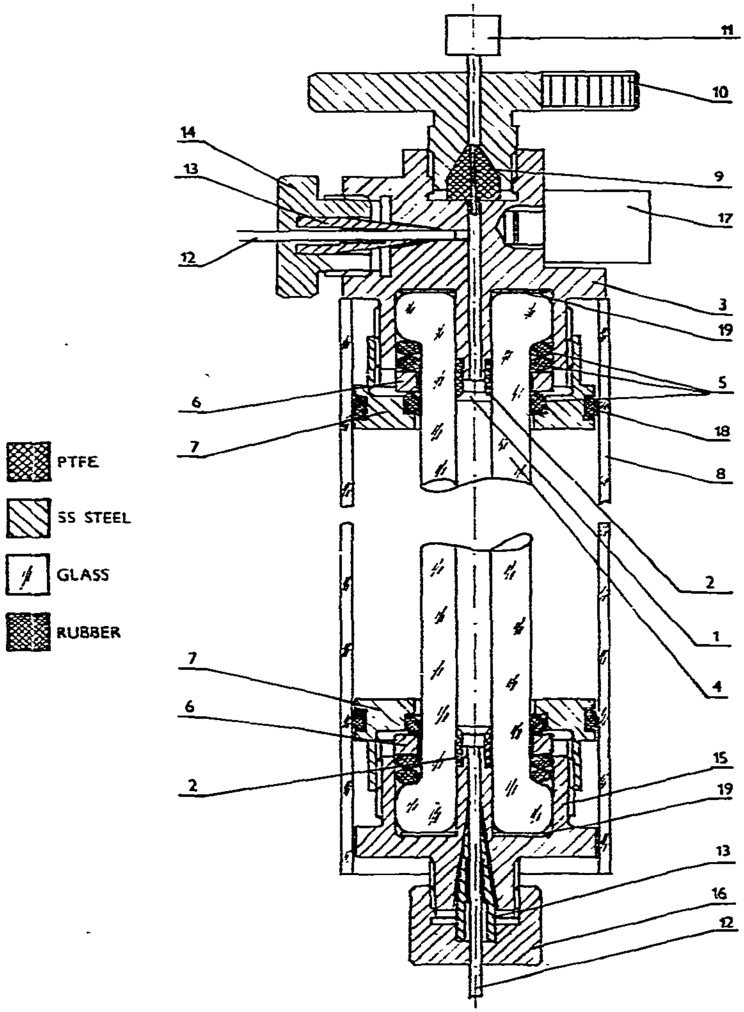

Fig. 1. Construction of column.

The final version of the construction is illustrated in Fig. 1*. A conical surface of glass or metal frit (1) is pressed against a polytetrafluoroethylene seal (2), forming a deformable joint between the inlet channel of the column head (3) and the glass column (4). The tube is fabricated with a very precise inner diameter (3.45 mm) and has two rounded rims on both ends. Between the glass tube rim, column head (3) and and washer (6) and nut (7) are inserted three rubber O-rings (5) and a plastic washer (19). A shaft (17) is used for mounting the column head on a stand.

The septumless injector is closed by a shaft (11) and a seal (9). When a sample is to be injected, the screw (10) is lifted, the shaft (11) is replaced with a syringe needle and the seal is re-tightened by the screw (10). The eluent is fed into the column by a capillary (12); the joint is made tight by a conical ferrule (13). A similar connection is made on the lower end of the column, but the shapes of the nuts (15) and (16) are different. The glass thermostating jacket (8) is simply held by an O-ring (18) in the groove of the nut (7).

The eluent comes into contact only with stainless steel, polytetrafluoroethylene and glass and it can therefore be used for most commonly used systems.

RESULTS AND DISCUSSION

These columns have been used routinely in our laboratory for more than 2 years with pressures up to 2.5 MPa and in some instances up to 10 MPa. No breakages of the glass column were observed. Because the column bed can be observed constantly, changes in the bed volume are easily detected. Operation at temperatures up to 80° caused no problems. It is difficult to make a quantitative estimation of influence of glass on the temperature of eluent in the column in comparison with columns made of stainless steel. Obviously, the heat transfer through a thick glass wall is less efficient than that through a relatively thin steel wall, but this may be not be very important. Firstly, the eluent flows through narrow bores in the metallic column head, which is kept at the same temperature as the column itself. Secondly, in both metallic and glass columns the most difficult part of the heat transfer is radially into the column bed, and in this respect both designs are equivalent.

Because the trend in HPLC in recent years has been towards smaller particles and pressures below 10 MPa, and because the interior surface of a stainless steel tube even if initially very smooth, may become corroded or eroded very easily, we believe, that there is a future for glass columns in this field.

ACKNOWLEDGEMENTS

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REFERENCES

1 K. G. Stahl and E. Schuppe, *GIT Fachz. Lab.*, 15 (1971) 28.
2 K. G. Stahl and E. Schuppe, *GIT Fachz. Lab.*, 17 (1973) 563.
3 K. Tesařík and P. Kaláb, *Chem. Listy*, 69 (1975) 1078.

* Patent applied for (PV-2693-77).