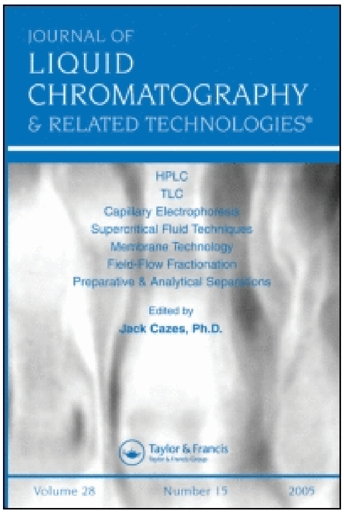

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EFFICIENCY OF GLASS CGC COLUMNS IN REVERSED-PHASE HPLC

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ABSTRACT

The design of metal-jacketed glass columns of CGC type is described, and the dependence of their efficiency on the flow rate is given, both for a single column and for two columns combined in series. The results have been treated in terms of reduced quantities; the optimal values of the reduced plate height $h \sim 2.5$ suggest a good quality of the sorbent packing. It is shown that the described combination of columns in series does not reduce the total efficiency of the system.

INTRODUCTION

Column is the heart of each chromatographic apparatus: its parameters govern the efficiency, resolving power and working throughput of each chromatograph. For all these reasons, manufacturing of columns for high-performance

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liquid chromatography is an exacting process, which of course is also reflected in the price of the column. If the latter loses its utility properties, the purchase of a new one means a comparatively high additional expense.

Many manufacturers try to solve this problem by introducing jacketed columns in which the jacket assumes several functions of the column. The latter may then be composed of a comparatively simple tube with closures, placed in the jacket (the so-called "cartridge" type). Advantages of such system cannot be denied; they not only reduce the price of the column itself, but also make possible a simple joining of several columns, or a combination precolumn - - column, without capillary connections and with a small loss of efficiency.

At present, there are several systems offered on the market which make use of the principle just mentioned. The columns are made of stainless steel; their jackets are always made of a combination of stainless steel and light metals. With respect to some disadvantages of metal tubes and to our own long-time experience with high-pressure glass columns for HPCL (1), a system of metal-jacketed glass columns has been developed by the Laboratory Instruments Works in cooperation with the Institute of Macromolecular Chemistry of the Czechoslovak Academy of Sciences(2). This system, while maintaining advantages of the cartridge columns, has stainless steel parts replaced with glass which in all respects is more advantageous. At present, the columns are manufactured by the Laboratory Instruments Works, Prague. Their design and some of their properties are dealt with in this paper.

Design of the column

Fig.1 shows the design diagram of the glass column. The CGC (compact glass column) consists of a glass tube (1), inner diameter 3.3 mm, 150 mm long. The ends of the tube are ground, smoothed by melting, and the whole tube is chemically reinforced so as to withstand average pressures of 80 MPa. At the ends, the glass tube is provided with cemented guide rings (2) which serve to fix the closing and sealing elements and to guide the column in the metal jacket. The guide rings contain centering collars (3) with a fine gauze which retains the column packing. A sealing collar (5) containing a pressed-in sealing ring (6) is placed on the centering collar. The centering and sealing collars are made from PTFE, the sealing ring is made from high-quality stainless steel. Under pressure, the sealing collars are pressed into the thread of the guide rings, thus preventing their loosening. The system makes possible an easy fixation of the column packing and an additional sealing of the column in the jacket by applying a relatively low axial pressure.

The column is placed in a jacket which consists of a metal tube (8) provided with threads for cap nuts (9) at its ends, and with end caps (10) screwed into the threads which serve for additional sealing of the column in the jacket and for joining the column with the chromatographic

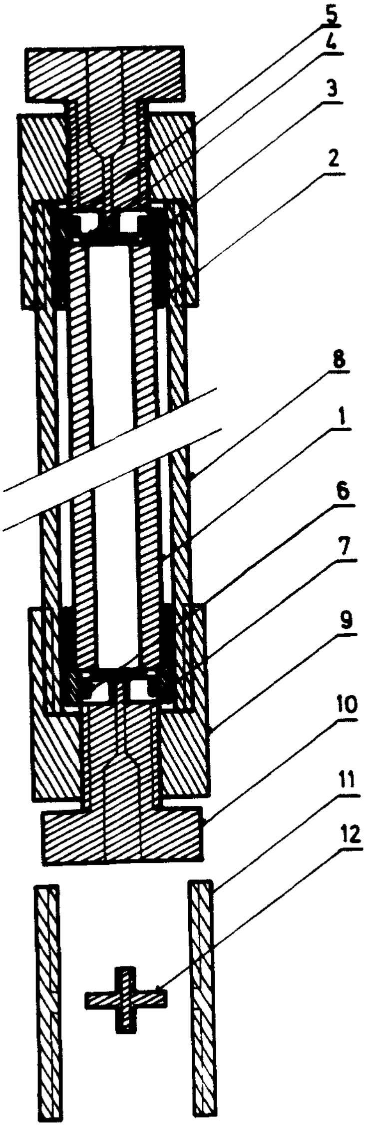

Fig.1 Schematic view of glass column of the CGC system (description cf. text)

apparatus. The jacket and nut caps are made from light metals, the end caps are made from stainless steel. The faces of the end caps are provided with grooves; after the column has been inserted into the jacket and the end caps have been screwed into the nut caps, the whole system is fixed by additional tightening of the nuts. This produces axial pressure which is needed to ensure stability of the system centering collar - sealing collar with the ring-grooved face of the end cap up to high pressures (50 MPa).

The columns are packed at 40-50 MPa by the slurry technique used for stainless steel columns. Only sorbents with particle size 5 µm are used as column packing; along with the design which minimizes dead volumes and the ideally smooth glass surface, they allow high chromatographic efficiencies to be achieved. The column dimensions have been chosen bearing in mind the use of 5 µm particles, and are optimized for limiting the wall effects according to(3).

The suggested system makes possible a simple joining of the columns in series and the use of protective precolumns. By using the cap nut (11) and the insert (12) (Fig.1), a column of double length can be obtained from the standard system using an additional jacket tube. The use of a short jacket allows a precolumn to be attached. Precolumns for the CGC system are 30 mm long.

EXPERIMENTAL PART

The liquid chromatograph used consisted of a syringe-type, positive displacement pump VLD 30(4,5) (Development

Workshop of the Czechoslovak Academy of Sciences, Prague), a home-made "stop-flow" sample injector (5), differential refractometer R 401 (Waters Ass., Milford, USA), and a potentiometric recorder (Servogor 220, Goerz, Austria).

The columns were packed with spherical silica (6), surface-modified with covalently bonded octadecyl groups (7) (SEPARON SIX C 18, manufactured by Laboratory Instruments Works, Prague). The average size of sorbent particles was $d_p = 5\mu m$. An acetonitrile-water mixture was the mobile phase (8:2 vol.), n-octyl alcohol (OcOH) and n-octyl acetate (OcAc) injected in a volume of 1 µl were used as the testing compounds.

Two columns (denoted as A and B) were examined with respect to their efficiency in the range of the mobile phase flowrate 0.1 - 1.2 ml/min, first each column separately and than both columns combined in series. The flowrate was first gradually increased from the minimal to the maximal and then decreased again. In this manner, each column was tested five times (to check the stability of the packing and to reduce the experimental error); the average from all measurements was taken for further treatment. A similar procedure was employed when both columns were connected in series. The results are summarized in Table I, together with the measured values of the capacity factor k' and with the corresponding values of pressure drop ΔP across the column or system of columns. The linear flow velocity u was calculated directly from the chromatogram using the retention time of an unretained peak

TABLE I

Plate number N, capacity factor k', and pressure drop, ΔP, at different volume flowrates, V, and linear velocities, u

	V	ΔP	u	OcOH		OcAc	
	ml/min	MPa	cm/min	k'	N	k'	N
Column A	0.1	0.75	2.01	1.36	8050	2.82	8400
	0.2	1.50	3.98	1.32	10000	2.75	10000
	0.4	3.25	7.76	1.29	11150	2.68	11250
	0.6	4.50	11.32	1.27	11300	2.66	11100
	0.8	6.25	15.00	1.28	10100	2.64	10250
	1.0	7.50	18.26	1.26	9200	2.63	9650
	1.2	9.25	23.50	1.27	8350	2.63	8900
Column B	0.1	0.50	1.74	1.35	8700	2.81	8800
	0.2	1.25	3.42	1.32	10500	2.74	10500
	0.4	2.75	8.11	1.28	11250	2.68	11000
	0.6	4.25	12.62	1.27	10600	2.66	10350
	0.8	5.50	16.36	1.26	9650	2.64	9750
	1.0	6.50	19.35	1.27	8650	2.64	9100
	1.2	7.75	22.98	1.26	8000	2.63	8500
Column A+B in series	0.1	1.25	2.00	1.37	21150	2.83	19350
	0.2	2.75	4.22	1.33	23200	2.75	24700
	0.4	5.50	7.96	1.30	25150	2.69	26500
	0.6	8.25	12.00	1.28	24650	2.65	25000
	0.8	11.25	16.36	1.26	21800	2.63	21500
	1.0	13.75	20.22	1.27	19850	2.63	19500
	1.2	16.00	23.29	1.27	18650	2.63	18450

and column length. The number of theoretical plates, N, was calculated from the peak width at half height.

RESULTS AND DISCUSSION

Table I demonstrates the good reproducibility of the capacity factor k', although its values seem to increase

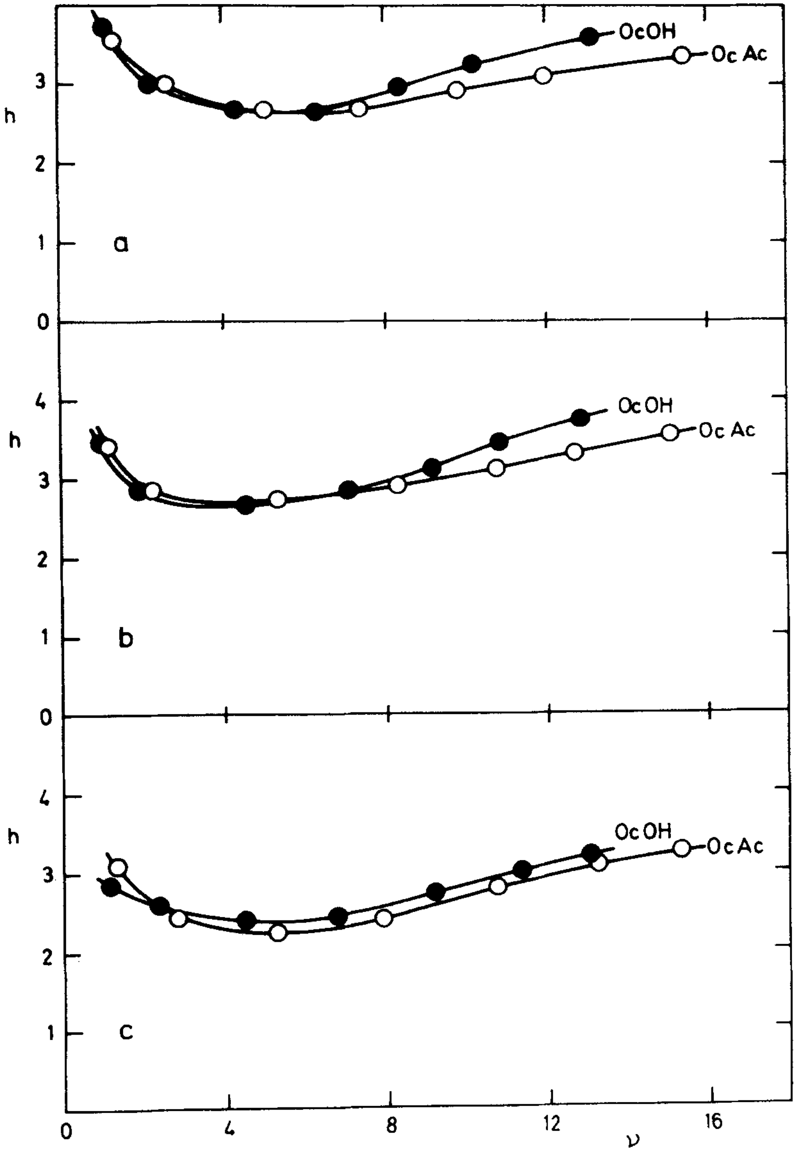

Fig.2 Dependence of reduced plate height, h, on reduced velocity, ν, for a) column A, b) column B, and c) columns A+B connected in series

somewhat at the minimal linear velocities of the mobile phase. The observed plate numbers are relatively high, considering that a column only 150 mm long packed with an octadecyl phase is involved; their dependence on the flow rate has a flat maximum near u = 10 cm/min.

It is worth mentioning that the sum of plate numbers of columns A and B is lower by 2-3 thousands on the average than that directly measured with both columns in series. This suggests that the mode of directly connecting the columns obviously does not impair essentially the efficiency of the whole system. It should be borne in mind, however, that with two columns connected in series, the extracolumn spreading in the refractometer cell is operative only once, while in the case of the individual columns A and B this negative effect is included twice in the sum of the plate numbers.

The experimental values were further evaluated in terms of reduced quantities; the required diffusion coefficients of the separated compounds in the mobile phase, D_m, were estimated from the approximative Wilke-Chang formula(8). The reduced plate height $h = H/d_p$ (where H is the actual plate height and d_p is the particle diameter) is plotted against the reduced velocity $\nu = u \cdot d_p/D_m$ for the individual columns (A,B) and for their combination in series (A+B) in Fig.2. The curves show a minimum of about $h \approx 2.5$ at $\nu \approx 5$; this comparatively low value of the reduced plate height indicates a good quality of the sorbent packing and its narrow particle size distribution.

CONCLUSION

The results of this study show that columns of the CGC system can be used in high-performance liquid chromatography within a broad range of flow rates. The column efficiency (with Separon SIX C 18 used as sorbent) does not depend to any considerable degree on the retention of the compound in the column. By connecting two CGC columns directly in series the obtained plate number is virtually doubled.

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