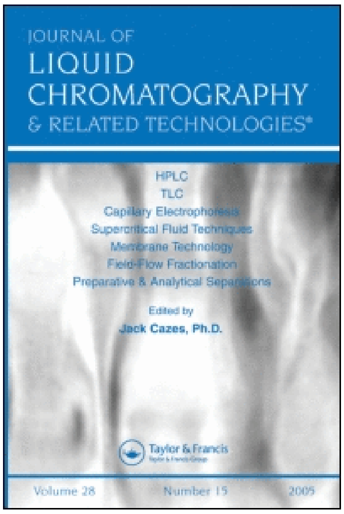

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Development of a Self Compressed Column System

Hailin Ge[a]; G. G. Wallace[a]
[a] Chemistry Department, The University of Wollongong, New South Wales, Australia

To cite this Article Ge, Hailin and Wallace, G. G.(1991) 'Development of a Self Compressed Column System', Journal of Liquid Chromatography & Related Technologies, 14: 8, 1615 — 1629
To link to this Article: DOI: 10.1080/01483919108049639
URL: http://dx.doi.org/10.1080/01483919108049639

JOURNAL OF LIQUID CHROMATOGRAPHY, 14(8), 1615-1629 (1991)

DEVELOPMENT OF A SELF COMPRESSED COLUMN SYSTEM

HAILIN GE AND G. G. WALLACE*
Chemistry Department
The University of Wollongong
P.O. Box 1144
Wollongong, New South Wales 2500, Australia

ABSTRACT

In the course of this work a novel HPLC column system has been developed. The system consists of a flexible-walled column made of Teflon tubing and a compression chamber through which the eluent is pumped. The column is placed in the chamber and compressed by a hydraulic pressure caused by the column back pressure. The performance of this system was characterized using commercially available C_{18} packing materials. The system is particularly useful for characterization or application of new stationary phases which are not commercially available.

INTRODUCTION

Both rigid-walled and flexible-walled columns have been used to perform High Performance Liquid Chromatographic

*Correspondence

(HPLC) separations (1, 2). A distinct characteristic of the flexible-walled column is that the column wall is deformed by external compression. As a result, wall effects are minimized (3) and the dead volume is lowered (4, 5). Furthermore, packing a flexible-walled column does not require the attention to detail required for packing rigid walled columns (6). Consequently, equipment such as a high pressure slurry packing pump is not required. At present the cost of both column systems remains high. There is undoubtedly a need for an efficient and economic column which is easily prepared in the laboratory. This is particularly so for teaching laboratories or those of us concerned with new application studies and development of new stationary phases where pre-packed columns are commercially unavailable and column efficiencies are not critical.

The column system developed in the course of this work consists of a flexible-walled column and a stainless steel chamber in which the column is fitted. The column wall is compressed when mobile phase passes through the chamber. Hence, the terminolgy - self-compressed column (SCC) system. The performance of the SCC has been investigated using commercially available C_{18} packing materials.

EXPERIMENTAL

Design and Preparation of the SCC

A schematic of the SCC system is shown in Figure 1. A flexible-walled column was placed in a chamber, and when the eluent flows through the system, a pressure generated in the chamber by the column back pressure results in compression of the column wall. To ensure a higher pressure in the chamber than that on the column inlet, a pressure regulator may be used. The compression force applied to the column wall is equal to the sum of the pressure drop (P) across the column (ΔP_2) and that across the regulator ΔP_1, i e

$$P = \Delta P_1 + \Delta P_2 \qquad (1)$$

A more detailed schematic of the chamber is shown in Figure 2.

Materials and Reagents

The materials used for construction of a self-compressed column are:

1. Teflon tubing (Waters);
2. Stainless steel chamber (made in the Science Faculty Workshop; the University of Wollongong);

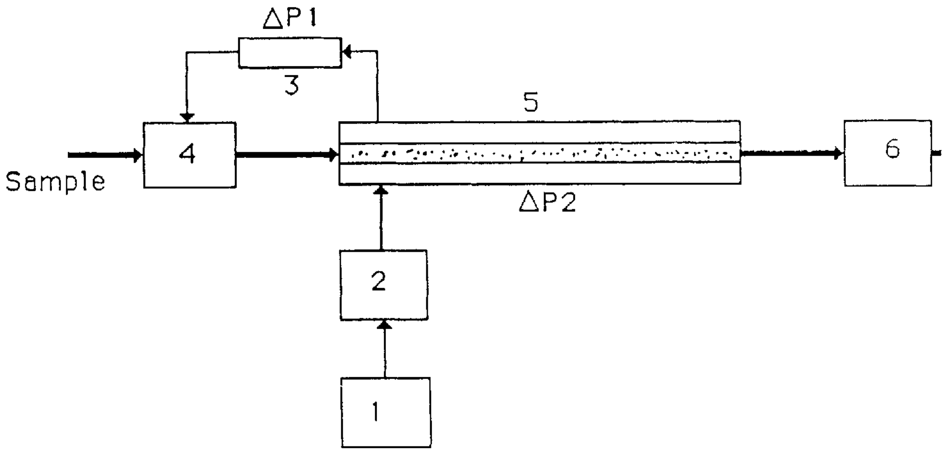

Figure 1 *Diagram of self-compressed column system*

1. solvent reservoir;
2. HPLC pump;
3. regulator;
4. injector;
5. self-compressed column system;
6. detector.

3. Frit (2μm, Activon); and

4. Fitting (1/4x28, Dionex).

ODS-Hypersil (10 μm Shandon) was chosen as the stationary phase for the testing of the column performance. A 65% methanol and 35% water mixture was used as an eluent. All tests were carried out using dimethylphthalate and benzene as test samples.

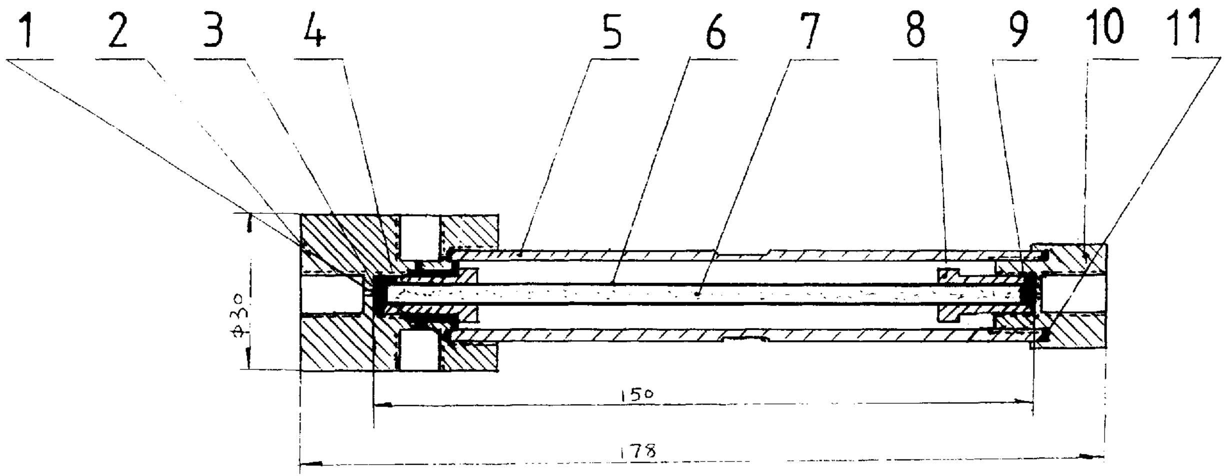

Figure 2 *The self-compressed column system*

1. and 11. sealer (Teflon ring);
2. chamber head (stainless steel);
3 and 9. frits (2 μm);
4 and 8 fitting (Dionex);
5. chamber (stainless steel).
6. column (Teflon tubing)
7. packing
10. chamber end cap (stainless steel).

The following procedure was used to prepare the self-compressed columns and to set up the system.

1. A length of Teflon tubing (70 mm) was cut off with a scalpel, two Dionex fittings were inserted and then both ends of the tubing were flared using a warm Dionex Teflon tubing modifier to form a seal.
2. Teflon tubing (I.D.= 1.6 mm and O.D.=3.2 mm or I.D.=3.2 and O.D.= 3.75 mm) was employed. For the latter a piece of Teflon tubing flared at one end (I.D.=1.6 mm, O.D.=3.2 mm and Length=4 mm) was inserted into both ends of the column to support the tube.
3. A Dionex fitting, in which a stainless steel frit (2 μm) with a Kel-F ring (6.2 mm diameter) was attached to the column.
4. The column was fitted to the packing chamber, and the system was connected as shown in Figure 3.
5. Immediately after steps [3] and [4], the slurry packing process was started at a preset flow rate to produce a pressure less than the column pressure tolerance (68 atm for the 1.6 mm I.D tubing and 28 atm for the 3.2 mm I.D tubing).
6. The packed column was placed into the column chamber as in Figure 2.
7. The column was connected to the injector after the air in the column chamber was driven out by flushing the chamber with the eluent.

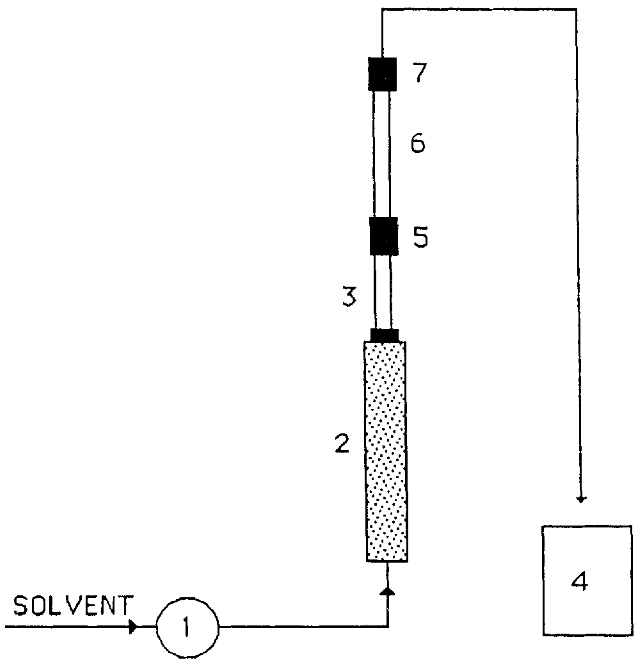

Figure 3 *Diagram of devices used for slurry packing of a self-compressed column*

1. HPLC pump;
2. packer (modified from a 30 cm Waters empty column);
3. buffer tubing (3.2 mm O.D x 1.6 mm I.D. Teflon tubing);
4. container;
5 and 7. Dionex female fitting;
6. SCC assembly.

Instrumentation

The HPLC system consisted of a Kortec K350 pump (ICI), a Rheodyne 7125 injector with 20 μl sample loop, a Kortec K95 variable wavelength detector (ICI) and a DP 600 chart recorder (ICI). Retention times were recorded using a stopwatch.

RESULTS AND DISCUSSION

Teflon tubing was found to be suitable for use as the column material. The mechanical flexibility, chemical inertness and the semitransparent nature which allows the packing material to be observed, were found to be useful properties. The latter enabling visual inspection of the column.

The column materials used for the SCC were subject to a pressure tolerance test by blocking one end of the tubing and pumping methanol through the other end until a pressure was reached at which the column materials broke down. The pressure tolerance was found to be 68 and 28 atm for the 3.2 mm O.D/1.6 mm I.D. and 3.75 mm O.D./3.2 mm I.D. columns respectively. The packing pressure employed was 40 and 17 atm respectively for the above columns. A compact bed was obtained using this packing pressure.

Effect of Self-Compression

The Effect of self-compression on the dead volume and efficiencies obtained with this system are shown in Table 1.

TABLE 1 *Effect of Self-Compression*

Flow rate (ml/min)	pressure (atm)		V_m (ml)		N (plate/meter)	
	A	B	A	B	A	B
0.1	4.5	4.5	0.311	0.295	4830	11470
0.2	8.5	8.5	0.317	0.296	5130	11780
0.3	12.5	12.5	0.311	0.297	6600	9500

Test column: 3.75 mm O.D x 3.2 mm I.D .x 64 mm 10 μm, ODS-Hypersil

Test sample: benzene

A: no self-compression

B: application of self-compression

The SCC system was evaluated using a 3.75 mm O.D. x 3.20 mm I.D column. This resulted in a ratio of wall thickness to inner diameter of 1:12. The dead volume was determined by injecting water without self-compression. The dead volume decreased by 5% after self-compression was induced. This was due to compression by the external hydraulic pressure. As a result, the column efficiency almost doubled. The column pressure drop was not affected significantly by self-compression. The column pressure drop increased with increased flow rate (Figure 4). The fact that at higher flow rates the total pressure was greater than the sum of the

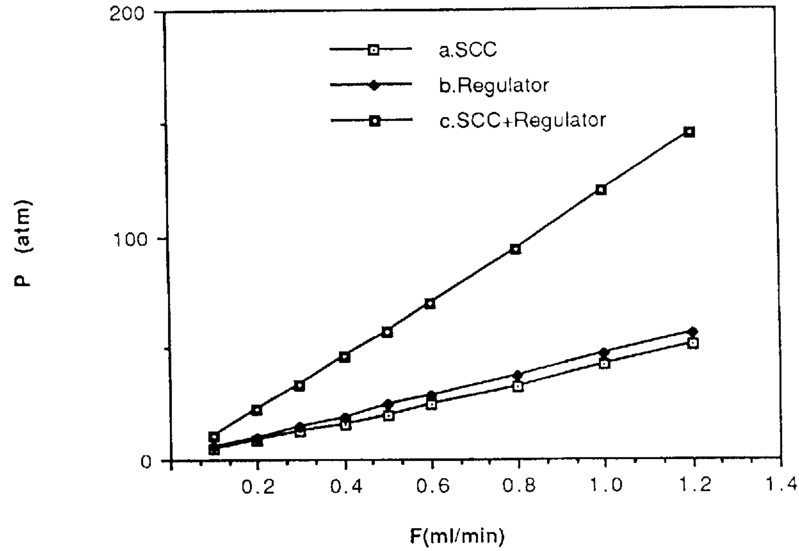

Figure 4 *Pressure drop vs. eluent flow rate on a self-compressed column*

Eluent: 65% MeOH and 35% H_2O;

Column: 3.2 mm I.D. x 3.75 mm O.D. x 64 mm Teflon column Packed with 10 µm ODS-Hypersil;

Curves: a. SCC; b. pressure regulator and c. column plus pressure regulator.

two pressures (SCC and regulator) indicated that self compression was in fact in operation.

Column Efficiency and Chromatography on the SCC

Chromatographic kinetics were investigated by plotting HETP vs. mobile phase velocity (7-9). This data was used to

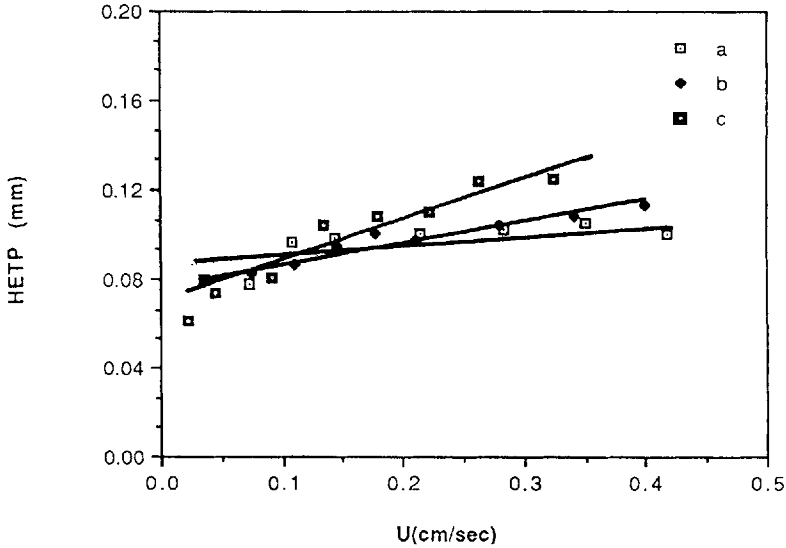

Figure 5 *HETP vs. eluent velocity*

Eluent: 65% MeOH and 35% H_2O

Column: a. 10 µm ODS-Hypersil in a 3.2 mm I.D. x 64 mm SCC; b. the same column as (a) but with a pressure regulator and c. 10 µm ODS-Hypersil in a 3.9 mm I.D. x 150 mm stainless steel column;

Sample: benzene.

compare the column efficiency for two different column systems using the same packing materials. It was found that using a C_{18} packing material, the column efficiency obtained with the self-compressed column was similar to that obtained with a stainless steel column which was

TABLE 2 *Extra Column Band Broadening*

Column system	Stainless steel column 0.39 mm I.D x 150 mm	Addition of 2 frits and 2 fittings (as used in the SCC system)
Eluent	65:35/MeOH:H_2O	65:35/MeOH:H_2O
F(ml/min)	0.6	0.6
Test sample	Benzene	Benzene
Capacity factor	1.7	1.7
Half peak width (μl)	156	178
Extra band broadening (μl)	-	22

packed using a slurry packing method at 620 atm (Figure 5). No significant decrease in the column efficiency was observed when a pressure regulator was not used. The pressure generated from the column itself was high enough to produce the self-compression effect and excessive pressure had no significant effect. At higher flow rates, the column efficiencies obtained using the SCC system were higher than these obtained using the stainless steel column system.

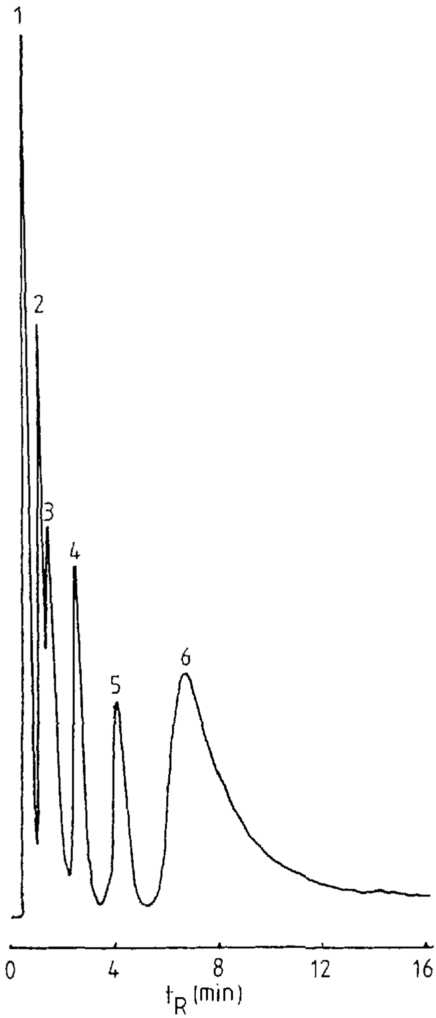

Figure 6 *Separation on a self-compressed column*

Column: 3.2 mm I.D. x 64 mm SCC packed with 10 µm ODS-Hypersil;

Eluent: 65% MeOH and 35% H_2O at 0.3 ml/min.;

Detector: UV at 254 nm;

Sample: 20 µl of 1.20 ppm benzoic acid, 2. 15 ppm phenol, 3. 5 ppm aniline, 4. 5 ppm DEP, 5. 5 ppm toluene and 6. 5 ppm DMA in the eluent.

Extra column effects due to connections and fittings reduced the column efficiency significantly when the present design of the self-compressed column was used. This was confirmed by introducing the same fittings as used with the stainless steel column system (Table 2). The extra column band broadening contributed was 14% (increase in half peak width) due to the additional fittings used in an SCC. This resulted in a 30% loss of the column efficiency.

An example of the chromatography achieved using the SCC is given in Figure 6.

CONCLUSION

The self-compressed column has been designed to obtain a reasonably efficient system which may be packed within the laboratory using simple procedures and allows to test or employ new stationary phases. The principle of self-compression is similar to that employed with other flexible-walled column systems (1, 2, 4-6). However, in the self-compression mode the eluent acts as the compression fluid. The preparation of the SCC with a normal HPLC pump was possible and relatively high performance was achieved.

REFERENCES

[1] Eon, C. H., *J. Chromatogr.* 149, 29, 1978.

[2] Landy, J. S., Ward, J. L. and Dorsey, J. G., *J. Chromatogr. Sci.*, 21, 49, 1983.

[3] Knox, J. H., Laird, G. R. and Raven, P. A., *J. Chromatogr.*, 122, 129, 1976.

[4] Assenza, S. P. and Brown, P. R., *J. Liq. Chromatogr.*, 3, 41, 1980.

[5] Little, J. N., Cotter, R. L., Predergast, J. A. and Patrick, D., *J. Chromatogr.*, 126, 439, 1976.

[6] Poole, C. F.and Schuette, S. A., Contemporary Practice of Chromatography, Elsevier, Amsterdam, 1984.

[7] Hamilton, R. J. and Sewell, P. A., Introduction to High Performance Liquid Chromatography (Second Edn.), Chapman and Hall, New York, 1982.

[8] Done, J. N., Knox, J. H. and Loheac, J., Application of High-Speed Liquid Chromatography, John Wiley and Sons, London, 1974.

[9] Jonssan, J. A., Chromatographic Theory and Basic Principles, Chromatography Sci. Series, V. 38, Marcel Dekker, Inc., New York, 1987.