

This article was downloaded by:

On: 24 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Liquid Chromatography & Related Technologies

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597273>

Comparison of Commercial Column Types in Liquid Chromatography

R. W. McCoy^a; R. E. Pauls^a

^a Standard Oil Company (Indiana), Naperville, IL

To cite this Article McCoy, R. W. and Pauls, R. E.(1982) 'Comparison of Commercial Column Types in Liquid Chromatography', *Journal of Liquid Chromatography & Related Technologies*, 5: 10, 1869 – 1897

To link to this Article: DOI: 10.1080/01483918208062860

URL: <http://dx.doi.org/10.1080/01483918208062860>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

COMPARISON OF COMMERCIAL COLUMN TYPES
IN LIQUID CHROMATOGRAPHY

R. W. McCoy and R. E. Pauls

Standard Oil Company (Indiana)
P. O. Box 400
Naperville, IL 60566

ABSTRACT

Several commercially available liquid chromatographic column types have been experimentally evaluated. A conventionally-sized column containing 5 μm packing, a microbore column containing 10 μm packing, a column containing 3 μm packing, and two short, wide-bore columns containing 5 μm packing were compared at optimum velocity (van Deemter minimum) and at twice the optimum velocity where possible. All columns contained reversed-phase media of the C-18 type. Attention was focused on establishing advantages and limitations of each column with regard to maximum available plate count, minimum separation time, and required pressure drop. A van Deemter plot was constructed for each column type and the number of plates generated per unit length, time, and pressure was determined. In addition reduced parameters, separation impedance, peak capacities, and analysis times at a given k' were calculated. Calculations indicated the highest possible plate counts should be obtained with coupled microbore columns. Small particle (3 μm) columns provided the best performance for high speed, moderate plate count separations. Conventional-sized columns containing 5 μm packing material appeared to be a good compromise between high speed and high total plate count.

INTRODUCTION

For the past several years most commercial manufacturers have employed a common design for analytical-scale liquid

chromatographic columns. These columns typically are stainless steel tubing of 4-5 mm internal diameter and 150-250 mm length packed with porous particles 5-10 μm in diameter. Recently several columns which deviate from this basic design have become available. These changes reflect a growing interest in high speed and high resolution separations. In striving toward these goals, the compromises in terms of column efficiency, analysis time, and available pressure dictated by each column design must be understood. With this knowledge, the practicing analytical chemist can wisely select the column design best suited for a particular separation problem.

Design, construction, and operation of packed, microbore columns have been the subject of several publications by Scott and co-workers (1-3). Their work demonstrates the extremely high efficiencies, on the order of 500,000 theoretical plates (1), obtained with 1 mm internal diameter columns packed with 10-20 μm particles. These columns provide maximum efficiency at volumetric flow rates in the range of 2-100 $\mu\text{l}/\text{min}$. These low flow rates provide improved solvent economy and mass sensitivity when utilizing concentration sensitive detectors such as the uv absorbance detector. However, these advantages are realized only with an extreme sacrifice in separation speed. At optimum conditions, separation times with microbore columns can be several hours (1).

Developments in the field of small-bore columns have not been limited to packed columns. The use of open tubular capillary columns of borosilicate glass (4,5) and fused silica (6) have been reported. Chemically-bonded, octadecyl groups provide separation surfaces similar to those of conventional reversed-phase packings. Packed capillary columns (7-9) containing either bare or surface modified alumina or silica gel have also been applied to the liquid chromatographic separation of complex mixtures. Recent theoretical comparisons (10,11) of microbore packed columns (PC), open tubular columns (OTC) and packed capillary columns (PCC) indicate no advantages for OTC and PCC type columns given the current operational limitations imposed by available instrumentation, principally detector cell volumes. However, if open tubular columns can be prepared with 5-10 μm internal diameter and detector cells of 1 nl or less volume are available, the inherent permeability advantages of these columns should provide performance beyond that realizable from packed columns (10).

The minimum height equivalent to a theoretical plate (H) for a well packed column is approximately equal to 2 particle diameters (3). Therefore, two equally well packed columns of the same length containing different size packing materials will provide total plates inversely proportional to the packing diameter. Conversely, if total plates are held constant, shorter columns containing smaller particles will provide faster analysis

times. Recently, several column manufacturers have begun offering columns packed with 3 μm particles to take advantage of potential savings in analysis times. Ettre, et. al. (12) have discussed high speed separations utilizing conventional sized columns containing these 3 μm packing materials.

According to Darcy's law (13), the pressure drop across a column is determined by both the packing particle diameter and the column length. Several commercial columns have recently become available utilizing conventional 5-10 μm packing materials in 100 mm lengths and 8-10 mm internal diameters. The stated advantages (14,15) of these columns is reduced back pressures allowing operation at higher volumetric flow rates resulting in reduced analysis times.

In this paper, various column designs will be compared at the optimum flow (minimum of the van Deemter plot) for each and at twice the optimum flow where possible. Based on this optimized evaluation, relative advantages and disadvantages of each design will be presented.

EXPERIMENTAL

Apparatus

The pumping system utilized in this work consisted of a Waters Associates (Milford, MA.) 6000A pump controlled by a Waters

660 controller. The single pump was connected to the "B-Pump" output of the controller. With this arrangement, the volumetric flow rate delivered by the pump was determined by the product of "% B" and "total flow" settings of the controller. Although it was conceptually possible to deliver flows from 1 $\mu\text{l}/\text{min}$ to 10 ml/min with this arrangement, delivered flow rates below 50 $\mu\text{l}/\text{min}$ differed significantly from the set values. Actual volumetric flow rates in these cases were determined by collecting mobile phase in a tared vessel for a fixed period of time (typically 30 minutes).

A Valco (Houston, TX) Model CFSV-6-HPAX sample injection valve with 0.2 μl internal loop volume was employed for all studies on microbore columns. For all other columns, a Rheodyne (Cotati, CA) Model 7125 sample injection valve with 0.5 μl internal loop was utilized. The maximum pressure ratings of the Valco and Rheodyne valves are 3000 and 6000 psi, respectively. Both valves were manually controlled.

The five reversed-phase columns employed in this study are listed in Table 1. Column dimensions, packings, particle sizes, and suppliers are provided. All columns were of fixed wall stainless steel construction except the RCSS column, which utilized flexible wall construction. During use, the RCSS column cartridge was placed in a Waters Model RCM 100 compression module.

TABLE 1

Columns Evaluated

<u>Column Type</u>	<u>Column Dimensions</u>	<u>Packing Type and Size</u>	<u>Manufacturer</u>
Conventional	250x4.6mm	Ultrasphere, ODS-5 μm	Altex/Beckman (Berkeley, CA)
Microbore	500x1.0mm	C-18 - 10 μm	Peterson Assoc. (Nutley, NJ)
Small Particle (3 μm)	100x4.6mm	C-18 - 3 μm	Perkin Elmer (Norwalk, CN)
RCSS ^a	100x8.0mm	C-18 - 5 μm	Waters Assoc. (Milford, MA)
RAC ^b	100x9.4mm	Partisil, ODS-3 - 5 μm	Whatman (Clifton, NJ)

(a) Radial Compression Separation System.

(b) Rapid Analysis Column.

The microbore column was connected directly to the injection valve and the detector flow cell. For all other columns, a minimum length (~ 6 cm) of 0.010 in internal diameter tubing was used for these connections. These connections accounted for approximately 3 μl of added dead volume.

A Schoeffel (Westwood, NJ) Model 770 variable wavelength detector equipped with Model SFA 234 0.5 μl flow cells was employed. Detector wavelength was adjusted to 254 nm for all studies and the detector time constant was set to the minimum value (fastest response). The detector analog output was

monitored with a Linear Instruments (Irvine, CA) Model 232 10 mv strip chart recorder. The analog output was also connected to a Modcomp II computer (Modular Computer Systems, Ft. Lauderdale, FL). Graphical display of chromatograms was possible on either a Tektronix (Beaverton, OR) Model 4010 video terminal or Model 4611 printer. All measurements of retention time and peak width were made from the output of the computer graphics system.

Digitization rates of the chromatographic data were varied from 7.5 to 1 points per second to suit individual experiments.

Reagents

A mobile phase of 60% (v/v) acetonitrile (Burdick and Jackson, Muskegon, MI) in water was employed in all studies. The water was distilled and purified through a Millipore (Bedford, MA) Milli-Q treatment system. Mobile phase was prepared by mixing exactly 400 ml water with exactly 600 ml acetonitrile and degassing the mixture under vacuum while stirring. Preparation in this manner negated volume changes due to mixing phenomena encountered with these two solvents.

The test solution for all column studies contained acetone (50% v/v), acetophenone (0.50% v/v), anisole (16.5% v/v), benzene (16.5% v/v), and toluene (16.5% v/v) of analytical reagent grade or better. This mixture was injected directly without further dilution.

Calculations

The number of theoretical plates, N , was calculated by

$$N = 5.54 (t_r/w_{1/2})^2 \quad (1)$$

where t_r is the solute retention time and $w_{1/2}$ is the width of the solute peak at half height. The plate height, H , is given by

$$H = L/N \quad (2)$$

where L is the column length. The reduced plate height, h , and reduced velocity, v , both dimensionless quantities, are defined by

$$h = H/d_p \quad (3)$$

$$v = ud_p/D_m \quad (4)$$

where d_p is the mean particle diameter, u is the linear mobile phase velocity, and D_m is the solute diffusion coefficient in the mobile phase.

The capacity factor, k' , is defined as

$$k' = (t_r - t_m)/t_m \quad (5)$$

where t_r and t_m are solute and nonretained peak retention times,

respectively. The separation impedance as defined by Bristow and Knox (16) was calculated by

$$E = (t_m/N)(\Delta P/N)(1/\eta) \quad (6)$$

where ΔP is the pressure drop across the column, η is mobile phase viscosity, and other terms are as previously defined. A value of 0.76 centipoise was assumed for η , the mobile phase viscosity, and values of 1.3×10^{-5} cm²/sec and 1.6×10^{-5} cm²/sec for toluene and acetone, respectively, were assumed for D_m , the solute diffusion coefficient (17).

RESULTS AND DISCUSSION

Plate height (H) values for all solutes were determined over linear mobile phase velocities of 0.1 to 5.0 mm/sec. These data for acetone ($k' \sim 0$), anisole ($k' \sim 1.2$), and toluene ($k' \sim 2$) are presented in Figures 1-5 in the form of van Deemter plots for each of the five columns under study. Since all columns were compared with the same mobile phase composition, k' varied for each component from column to column.

The van Deemter plot for a Altex/Beckman Ultrasphere-ODS column, representative of conventional liquid chromatographic columns, is provided in Figure 1. The optimum linear velocity was 1.5 mm/sec corresponding to a volumetric flow rate of 1.0 ml/min.

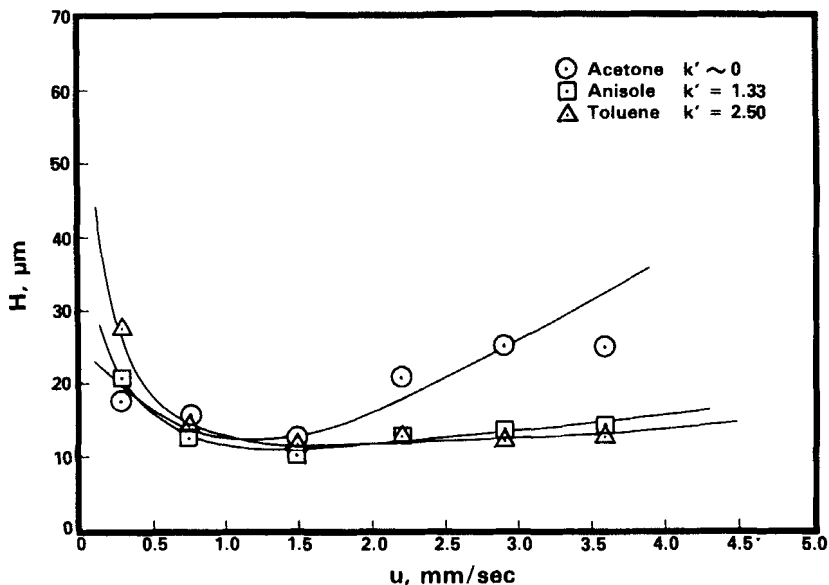


Figure 1. The van Deemter plot for test solutes on a 250 x 4.6 mm column containing 5 μm Ultrasphere ODS packing.

The plate height at optimum flow was roughly 11 μm for anisole and toluene and was slightly higher for acetone. The minimum of the van Deemter plot for this column was broad for anisole and toluene while the nonretained peak, acetone, displayed a rapid loss in efficiency at increased velocities.

Figure 2 contains the van Deemter plot for a 50 cm microbore column. The minimum plate height occurs at a velocity of approximately 0.5 mm/sec corresponding to a volumetric flow rate of 28 $\mu\text{l/min}$. At this flow H values ranged from 26 μm for toluene to 33 μm for acetone. For all solutes, the minimum of the van

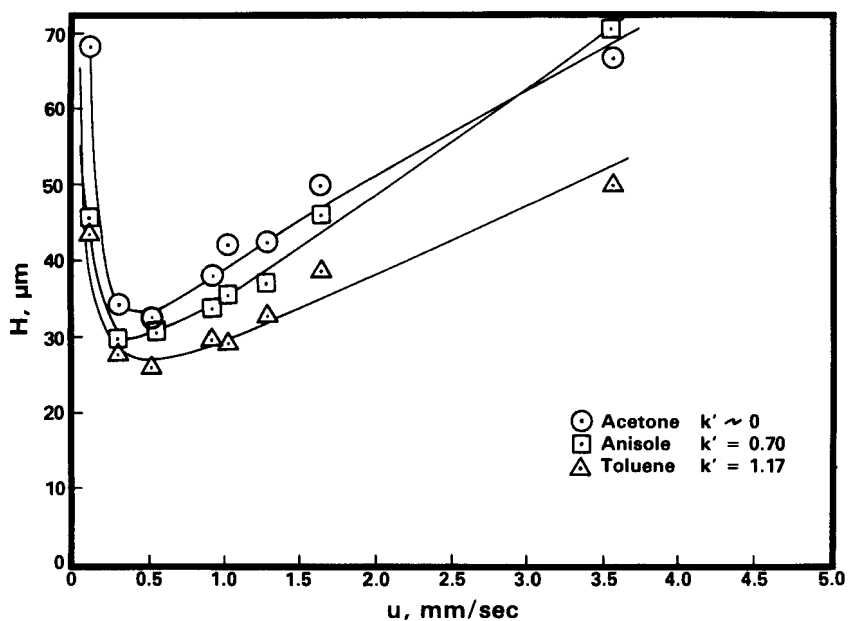


Figure 2. The van Deemter plot for test solutes on a 500 x 1.0 mm microbore column containing 10 μm packing.

Deemter plot was sharp and column efficiency degraded quickly with increased flow. Plate height values for the microbore were significantly larger than those found for the Ultrasphere column reflecting the increased particle diameter (10 μm vs. 5 μm).

Figure 3 shows the van Deemter plot for a Perkin-Elmer column packed with small particles (3 μm). This plot is flat throughout the entire linear velocity range examined. The minimum plate height for toluene of roughly 8 μm occurred at a velocity of 2.5-3.0 mm/sec. This velocity corresponded to a volumetric flow rate of 2.0 ml/min.

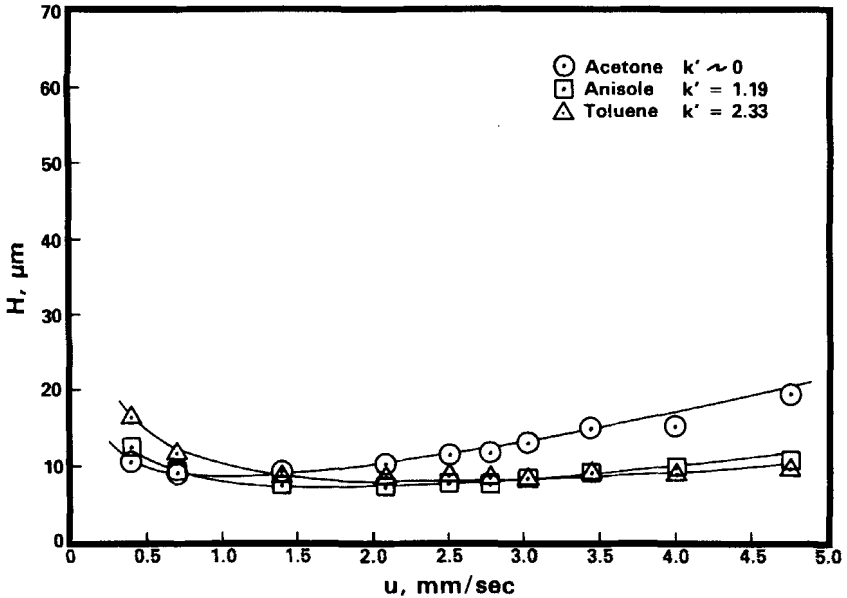


Figure 3. The van Deemter plot for test solutes on a 100×4.6 mm column containing $3 \mu\text{m}$ packing.

The van Deemter plot for a radial compression column (RCSS) packed with $5 \mu\text{m}$ particles is given in Figure 4. Acetone exhibits a minimum at a lower velocity than found for anisole or toluene. The minimum H for acetone occurred at a velocity of approximately 0.7 mm/sec while for toluene the minimum occurred at 1.0 mm/sec corresponding to a volumetric flow of 2.1 ml/min . The minimum H for toluene was approximately $10 \mu\text{m}$.

Plate height data for the Whatman RAC column is given in Figure 5. This plot is similar to that obtained with the RCSS column since acetone exhibited a minimum at a lower flow rate than

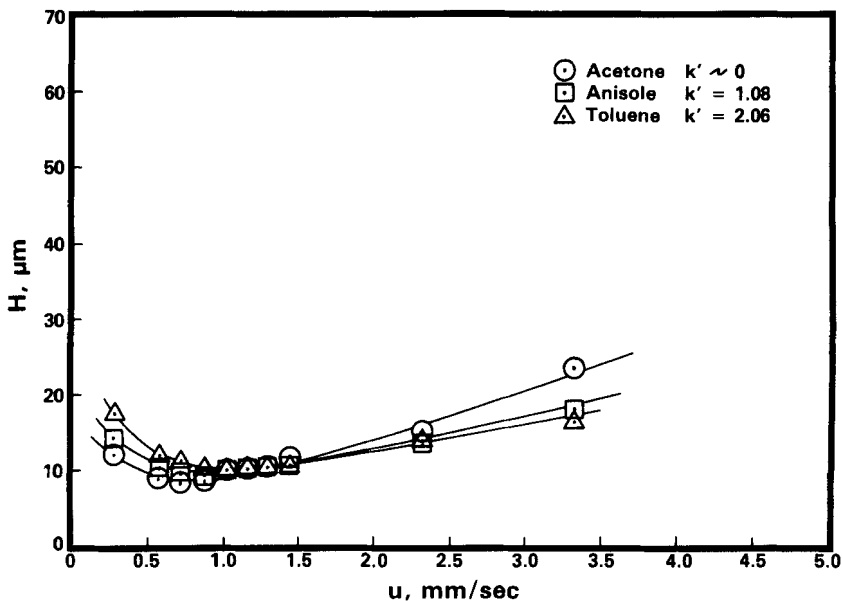


Figure 4. The van Deemter plot for test solutes on a 100 x 8.0 mm RCSS column containing 5 μm packing.

the other solutes. The minimum H for toluene was 14 μm . The linear velocity at the minimum was 2.1 mm/sec corresponding to a volumetric flow rate of 7.5 ml/min.

Figure 6 summarizes the plate height data obtained with toluene as solute for all five columns. As expected columns containing smaller diameter packing materials gave lower minimum H values. The smallest plate height was obtained with the Perkin-Elmer 3 μm column followed by the RCSS column and the Ultrasphere column. The minimum H value for the microbore column packed with 10 μm particles was significantly larger than those of the other columns.

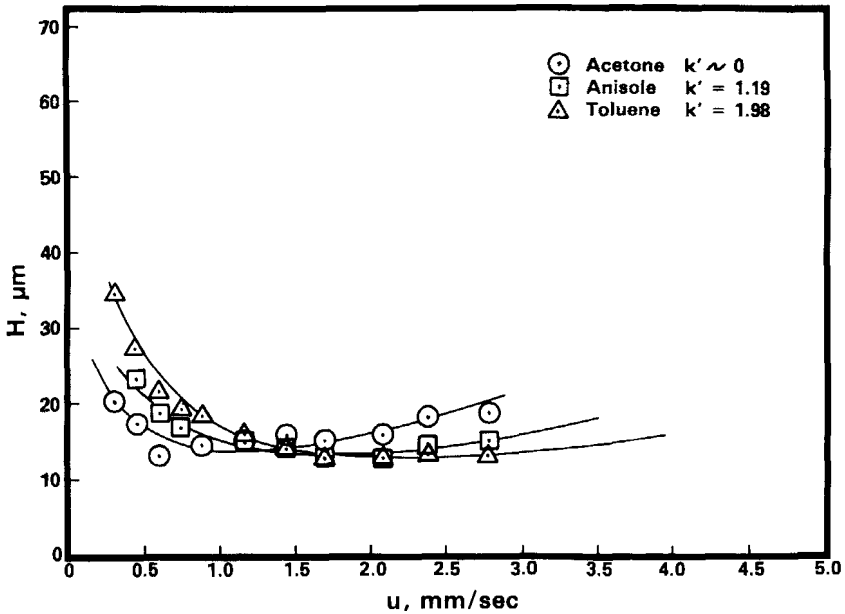


Figure 5. The van Deemter plot for test solutes on a 100 x 9.4 mm RAC column containing Partisil ODS-3 5 μm packing.

The optimum linear velocity for the microbore column was 0.5 mm/sec, smaller by a factor of 2-5 compared to the other columns. This, combined with the small internal diameter of the column, requires operation at low volumetric flow rates to achieve maximum column efficiency. The optimum linear velocities for the other four columns ranged from 1.0 to 2.5 mm/sec.

All columns except the microbore exhibited broad, flat van Deemter plots indicating only small losses in column efficiency need be sacrificed with operation at higher flow rates. However, with the microbore column severe losses in column efficiencies

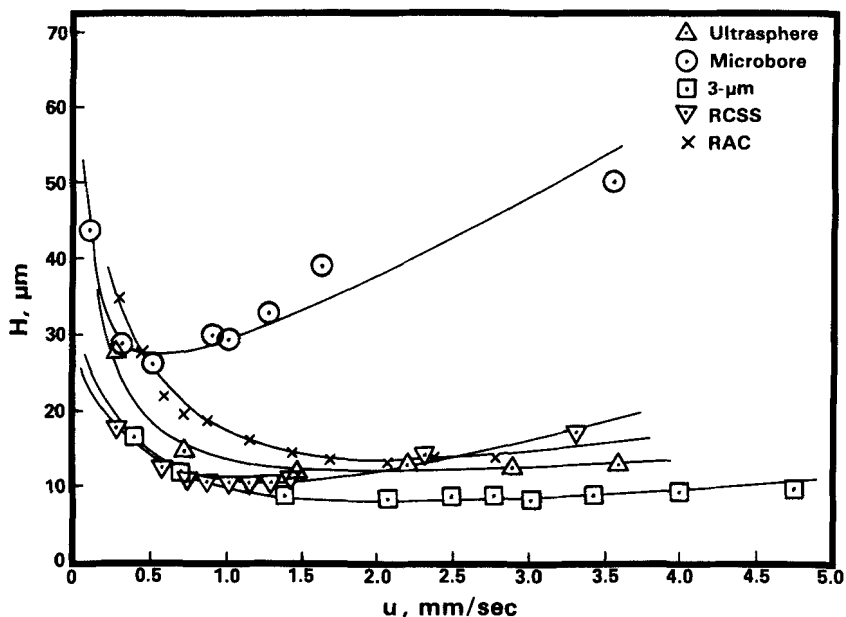


Figure 6. The van Deemter plot for toluene on all five columns evaluated.

would result with operation at velocities in excess of twice the optimum.

Well packed liquid chromatographic columns will normally have minimum reduced plate heights (h) of approximately 2 at reduced velocities (v) of approximately 3-20 (16). Values for h and v are presented in Table 2 for acetone and toluene. These values were calculated at the minimum of the van Deemter plot for each column. Reduced plate height values for toluene range from 2.04 to 2.80 with the RCSS column having the lowest value and the 3 μm column having the highest. This larger h value obtained with the 3 μm

TABLE 2

Reduced Plate Height and Velocity at Optimum Velocity

Column Type	Acetone, $k' \sim 0$		Toluene, $k' \sim 2$	
	h	v	h	v
Conventional	2.52	4.6	2.30	5.7
Microbore	3.24	3.2	2.62	3.9
Small Particle	2.93	1.3	2.80	6.4
RCSS	1.68	2.2	2.04	3.9
RAC	2.80	1.9	2.62	8.0

column might indicate some difficulties in packing columns with these small particles. The H value for this column, however, is still better than that obtained with any other column evaluated. All columns exhibit h values close to the expected 2 in a reduced velocity range of 1.3 to 8, consistent with the description of a well packed column. Therefore, subsequent comparisons are based on columns representative of state of the art commercially available.

A typical chromatogram is presented in Figure 7. The elution order was identical for all columns examined. Linear velocities, volumetric flow rates, total plate counts, column pressure drop, and retention characteristics for acetone and toluene at optimum conditions are summarized in Table 3. For the acetone peak ($k' \sim 0$), the greatest number of theoretical plates, 19500, was

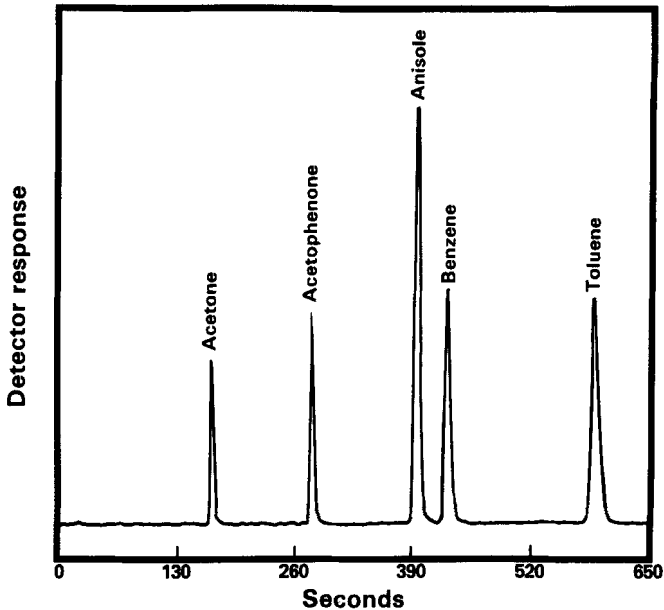


Figure 7. Chromatogram produced by test mixture on a 250 x 4.6 mm column containing 5 μm Ultrasphere ODS packing operated at optimum linear velocity, 1.48 mm/sec.

generated by the conventional Ultrasphere column followed by the microbore column with 15400 plates. The retention time for toluene was much greater at the low optimum flow rate for the microbore column than with any other column evaluated. The shorter columns, as expected, produced the fastest analyses. The column pressure drop varied significantly from 300 psi for the microbore to approximately 4000 psi for the RAC column.

The data in Table 3 indicate the raw number of plates available per column and the relative sacrifices in time and

TABLE 3
Column Characteristics at Optimum Linear Velocity

Column Type	u, mm/sec	Flow, ml/min	Pressure Drop, psi	Acetone		Toluene			
				Retention Time, sec	N	Retention Time, sec	N	k'	
Conventional	1.48	1.0	1750	169	19500	~0	592	21800	2.50
Microbore	0.513	0.028	300	975	15400	~0	2111	19100	1.17
Small Particle	2.78	2.0	3120	36	8330	~0	120	11900	2.33
RCSS	1.02	2.1	590	98	10000	~0	300	9820	2.06
RAC	2.08	7.5	3760	48	6250	~0	143	7650	1.98

pressure required to achieve these plate counts. To better assess the relative efficiencies of these columns, plates per column length, plates generated per second, and plates per unit pressure are given in Table 4. This table also contains values for the separation impedance, E. Separation impedance is a dimensionless quantity which measures the expense in time and pressure required to achieve a given plate count. Reports in the literature (11) demonstrate values of less than 1000 indicate excellent performance while values greater than 20000 indicate poor performance.

The data in Table 4 reveal the largest number of plates per meter were obtained on the 3 μm column (119000 plates per meter). This column also generated the largest number of plates per unit time (99 plates per second). At the same time, this column was next to last in plates generated per unit pressure drop

TABLE 4
Quantitative Column Comparison at Optimum Velocity

<u>Column Type</u>	<u>N/column</u>	<u>N/meter</u>	<u>N/sec</u>	<u>N/psi</u>	<u>E</u>
Conventional	21800	87200	36.8	12.5	5650
Microbore	19100	38200	9.05	63.7	7220
Small Particle	11900	119000	99.2	3.81	7200
RCSS	9820	98200	32.7	16.6	5440
RAC	7650	76500	53.5	2.03	27970

demonstrating the continual sacrifices that must be made between resolution, time, and pressure. The microbore column demonstrated the lowest number of plates per meter and plates per unit time. These plates however were achieved with a minimal sacrifice in pressure drop as indicated by the high value of 64 plates per psi. The remaining three columns were intermediate in plates per unit length and plates per unit time. Separation impedance values indicate that four of these columns had roughly equivalent performance. The RAC column demonstrated inferior performance as measured by this parameter.

For high speed analyses, the mobile phase velocity of choice is not necessarily the optimum linear velocity. By operating the column at twice the optimum velocity (optimum practical velocity) a reduction in analysis times of approximately 2 can be realized with a minimal sacrifice of 10-20% in plate count (3). The exact value of this reduction will be determined by the steepness of the van Deemter plot or the relative importance of the van Deemter C term for each column. The Ultrasphere, microbore, and RCSS columns were compared at the optimum practical velocity. Data for the three columns are presented in Table 5. The 3 μm and RAC columns could not be included in this comparison due to pressure (6000 psi) and flow (10 ml/min) restraints, respectively, imposed by the equipment.

Comparison of Tables 4 and 5 indicates analysis times were reduced as expected by a factor of 2 by operation at optimum

TABLE 5

Quantitative Column Comparison at Optimum Practical Velocity^a

<u>Column Type</u>	<u>N/column</u>	<u>N/meter</u>	<u>N/sec</u>	<u>N/psi</u>	<u>E</u>
Conventional	19960	79800	66.5	5.75	6770
Microbore	17060	34120	16.3	28.9	8880
RCSS	8330	83300	53.7	6.77	8150

(a) Optimum practical velocity equals twice optimum linear velocity.

practical velocity with a loss in total plates of 8%, 11%, and 15% for Ultrasphere, microbore, and RCSS columns, respectively. At the same time, however, the number of plates generated per unit pressure decreased by approximately a factor of 2. Table 5 shows the Ultrasphere column provided the largest number of plates per unit time and highest column performance as measured by the impedance parameter.

Guiochon (18) has shown that optimum analytical performance of a liquid chromatographic column is obtained when the last component of a mixture is eluted at $k' = 6.4$ (analysis time of 7.4 times the dead time) and that the peak capacity under these conditions is given by $N^{1/2}/2$. Peak capacity is defined as the number of components which can be reasonably well resolved in a given analysis time provided a column/mobile phase combination can be found which spread these components uniformly throughout the

chromatogram. Analysis times and peak capacities have been calculated at optimum linear velocities for the five columns and are presented in Table 6. The plate count of toluene for each column was employed in this calculation. The 3 μm column gave the shortest analysis time followed by the RAC column. This data again reflects the excellent potential for high speed separations with the 3 μm column. The other columns had considerably longer analysis times with the microbore column giving the longest. The conventional Ultrasphere column had the greatest peak capacity in this evaluation followed closely by the microbore column.

It has been shown (1) that several lengths of individually packed microbore columns can be connected to produce a column with plate counts equivalent to the sum of the parts. Since these columns

TABLE 6

Calculated Analysis Time ($k'=6.4$) and Peak Capacity^a

<u>Column Type</u>	<u>Analysis Time^b, sec</u>	<u>Peak Capacity</u>
Conventional	1251	74
Microbore	7215	69
Small Particle	266	54
RCSS	725	50
RAC	355	44

(a) Values calculated at optimum linear velocity.

(b) Analysis time equals retention time of component at $k'=6.4$.

operate at low volumetric flows and low pressure drops, several meters of microbore column can produce several hundred thousand plates at moderate pressures thus allowing separation of extremely complex mixtures. Assuming linear additivity of both total plates and pressure drops, the allowable column length consistent with a given maximum pressure drop was calculated for each of the five columns under study. For these calculations, operation at optimum linear velocity was selected and a maximum operating pressure of 5000 psi was imposed for all except the RCSS column.

The pressure limit of the compression module is 2000 psi and this was the value utilized for this column. The results from these calculations as well as other column characteristics are given in Table 7. The data show the microbore is clearly the column of

TABLE 7

Calculated Column Characteristics at Maximum Pressure, 5000 PSI^a

<u>Column Type</u>	<u>Maximum Column Length,m</u>	<u>N at Maximum Length</u>	<u>Analysis Time (k'=6.4) at Maximum Length,sec</u>	<u>Peak Capacity at Maximum Length</u>	<u>Elution Volume (k'=6.4) at Maximum Length,ml</u>
Conventional	0.714	62300	3570	125	59.5
Microbore	8.33	318000	120000	282	56.0
Small Particle	0.160	19000	425	69	14.2
RCSS ^b	0.339	33300	2460	91	86.1
RAC	0.133	10180	472	50	59.0

(a) Values calculated at optimum linear velocity.

(b) This column is limited to 2000 psi maximum pressure due to the design of the radial compression system.

choice for high efficiency separations with a peak capacity more than twice its closest rival. This column could separate 282 components evenly spaced over a 33 hour analysis at an expense of 56 ml of mobile phase. A total of 318000 theoretical plates could be generated. For separations requiring moderate efficiency (30-60000 plates) either the Ultrasphere or RCSS columns produce reasonable analysis times (40-60 min). The maximum lengths for the RAC and 3 μ m columns are limited due to high operating pressures per unit length. This in turn limits these columns to 10-20000 total plates. However, this limited efficiency is obtained in a short time period.

The length of column necessary to generate a fixed number of theoretical plates is another useful comparison of various chromatographic columns. Table 8 summarizes the length of each

TABLE 8

Calculated Column Characteristics at 10,000 Theoretical Plates^a

<u>Column Type</u>	<u>Column Length,mm</u>	<u>Pressure Drop,psi</u>	<u>Analysis Time (k'₁=6.4),sec</u>	<u>Elution Volume (k'₁=6.4),ml</u>
Conventional	115	805	575	9.59
Microbore	262	157	3780	1.76
Small Particle	84	2621	224	7.46
RCSS	102	602	738	25.8
RAC	131	4926	466	58.2

(a) Values calculated at optimum linear velocity.

column necessary to deliver 10000 theoretical plates and the resulting pressure drop, analysis time, and elution volume when operating at optimum velocity with $k' = 6.4$. As expected, the column length and analysis time are shortest for the highly efficient 3 μm column. An analysis time of 224 seconds would be required for a component with $k' = 6.4$. This is faster by a factor of at least 2 than any other column. The microbore column required the greatest length and the longest analysis time but achieved the separation at the expense of only 1.76 ml of mobile phase.

SUMMARY

This experimental study has compared the relative merits of various designs of commercial, analytical-scale liquid chromatographic columns. These comparisons were based on one column of each type and no effort was made to account for column-to-column variability. In addition all columns were evaluated with a single mobile phase composition resulting in solute k' values of 2 or less. Hence, no effort was made at evaluations at higher k' values or under conditions where all columns exhibited constant k' values.

The Ultrasphere ODS column, typical of most commercially available columns, exhibited intermediate performance in most aspects of the evaluation. This column produced more theoretical plates, 21800 for toluene, on a per column basis than any of the

other columns examined. Under the experimental conditions, assuming linear additivity of plates, approximately three 250 mm lengths placed in series would produce 62000 plates in 1 hour with a k' of 6.4.

The microbore column when operated at optimum linear velocity exhibited minimal pressure drop, however, analysis times were greatest for this column compared to all others in this evaluation. This column demonstrated the poorest performance in terms of plates per meter and plates per unit time while exhibiting best performance in terms of plates per unit pressure drop. Due to the low pressure drop, it is conceptually possible to link 8.3 meters of column to produce 318000 plates within a 5000 psi pressure limitation. However, the price for this high plate count is long retention time, 33 hours for a component eluting at a $k'=6.4$.

The small particle (3 μm) column exhibited the greatest number of plates per unit length (lowest H value) and greatest plates per unit time of the five columns examined. Due to the small packing diameter, the pressure drop was high resulting in low plates per unit pressure. The combination of short column length (100 mm) and high plates per unit time results in the fastest analysis time of the five columns evaluated. Due to the large pressure drop generated by 3 μm particles, only 160 mm of column length could be utilized at a 5000 psi pressure limit with

the above experimental conditions. This column length would generate 19000 plates with a component of $k' = 6.4$ eluting in approximately 6 minutes.

The radial compression column offered moderate plate count at low operating pressures, however, the potential advantage of this lower pressure drop is counteracted by the 2000 psi pressure limitation imposed by the compression module. This column gave the smallest reduced plate height of any column evaluated.

For the RAC column, the optimum linear velocity occurred at the high volumetric flow rate of 7.5 ml/min resulting in short retention times with a corresponding high back pressure. The separation impedance for this column was significantly higher than for any other column evaluated which might indicate an atypical column.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the contributions of R. D. Snyder in performing some of the experiments reported here.

REFERENCES

1. Scott, R.P.W. and Kucera, P., Mode of Operation and Performance Characteristics of Microbore Columns for use in Liquid Chromatography. *J. Chromatogr.*, 169, 51 (1979).
2. Scott, R.P.W., Microbore Columns in Liquid Chromatography. *J. Chromatogr. Sci.*, 18, 49 (1980).

3. Reese, C.E. and Scott, R.P.W., Microbore Columns - Design, Construction, and Operation. *J. Chromatogr. Sci.*, 18, 479 (1980).
4. Tsuda, T., Hibi, K., Nakanishi, T., Takenchi, T., and Ishii, D., Studies of Open-Tubular Micro-Capillary Liquid Chromatography. II. Chemically Bonded Octadecylsilane Stationary Phase. *J. Chromatogr.*, 158, 227 (1978).
5. Ishii, D. and Takenchi, T., Open Tubular Capillary LC. *J. Chromatogr. Sci.*, 18, 462 (1980).
6. Yang, F. J., Fused Silica Open Tubular Column for Liquid Chromatography. *J. of HRC and CC*, 3, 589 (1980).
7. Hirata, Y. and Novotný, M., Techniques of Capillary Liquid Chromatography. *J. Chromatogr.*, 186, 521 (1979).
8. Novotný, M., Capillary HPLC: Columns and Related Instrumentation. *J. Chromatogr. Sci.*, 18, 473 (1980).
9. Hirata, Y., Novotný, M., Peaden, P., and Lee, M., A Comparison of Capillary Chromatographic Techniques for the Separation of Very Large Polycyclic Aromatic Molecules. *Anal. Chimica Acta*, 127, 55 (1981).
10. Guiochon, G., Conventional Packed Columns vs. Packed or Open Tubular Microcolumns in Liquid Chromatography. *Anal. Chem.*, 53, 1318 (1981).
11. Knox, J.H., Theoretical Aspects of LC with Packed and Open Small-Bore Columns. *J. Chromatogr. Sci.*, 18, 453 (1980).
12. DiCesare, J.L., Deng, M.W., and Ettore, L.S., Very-High-Speed Liquid Chromatography: The System and Selected Applications. *Chromatographia*, 14, 257 (1981).
13. Giddings, J.C., Dynamics of Chromatography, Part I, Principles and Theory. Dekker, New York, 1965, Chapter 5.
14. Waters Associates Technical Bulletin, Radial Compression Separation System (RCSS)., 1979.
15. Whatman, Chem-Sep News, 1, (1981).
16. Bristow, P.A. and Knox, J.H., Standardization of Test Conditions for High Performance Liquid Chromatography Columns. *Chromatographia*, 10, 279 (1977).

17. Snyder, L.N. and Kirkland, J.J., Introduction to Modern Liquid Chromatography, Second Edition. Wiley, New York, 1979, p.838.
18. Guiochon, G., Preparation and Operation of Liquid Chromatographic Columns of Very High Efficiency. J. Chromatogr., 185, 3 (1979).