Journal of Chromatography, 592 (1992) 239–247
Elsevier Science Publishers B.V., Amsterdam

CHROMSYMP. 2443

High-temperature open-tubular capillary column liquid chromatography

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ABSTRACT

In open-tubular liquid chromatography (OTLC), elevation of the column temperature has the same effect as reduction of the column diameter in obtaining high column efficiencies, with the additional benefits of easier column handling and fewer problems with detection. On an experimental set-up with commercially available 50 and 100 μm I.D. reversed-phase columns working in the 100–200°C range, all the advantages predicted by OTLC theory for elevated column temperatures were confirmed: smaller slope of the plate height *vs.* linear velocity (H *vs.* u) curve, higher optimum linear velocity (u_{opt}) value and less pronounced influence of the retention on efficiency. High-temperature operation makes high-efficiency and high-speed OTLC analysis feasible. On a 19.6 m × 50 μm I.D. SB-Methyl-100 column operated at 200°C with acetophenone as the test sample, 1 143 000 theoretical plates were obtained within 50 min.

INTRODUCTION

In open-tubular capillary column liquid chromatography (OTLC) there is a large discrepancy between the theoretical and fundamental research and the practical application of the technique. Almost 40 years ago, Taylor [1,2] first described the band broadening of a solute in a liquid flow passing through tubular columns. A few years later, Golay [3] published his classical paper on capillary column chromatography. These works, together with Sternberg's detailed analysis of the band dispersions caused by various extra-column factors [4], built up the theoretical framework of capillary chromatography. The correctness of Golay's theory was proved by a large number of experiments [5–8]. In the meantime, Golay [9] and Tijssen [10] carried out important complementary studies on the secondary flow effect. Many groups have been involved in recent years [11–14] in the development of high-efficiency OTLC systems by using small bore (I.D. < 10 μm) columns. In all instances the established theory proved to be reliable.

In contrast, reports on the practical application of OTLC are rarely found in the literature. One explanation for this situation is in the fact that OTLC, when operated at room temperature, is of relatively low efficiency as compared with other separation techniques, such as capillary GC, capillary zone electrophoresis and capillary supercritical fluid chromatography. When very small-bore columns are used to obtain reasonable efficiency, the existing detection problem of OTLC becomes even more serious [15].

The height equivalent to a theoretical plate (HETP or H) of an open-tubular column is given by the Golay equation:

$$H = \frac{2D_{\mathrm{m}}}{u} + \frac{1 + 6k' + 11k'^2}{96(1 + k')^2} \cdot \frac{d_{\mathrm{c}}^2}{D_{\mathrm{m}}} \cdot u + \frac{2k'}{3(1 + k')^2} \cdot \frac{d_{\mathrm{f}}^2}{D_{\mathrm{s}}} \cdot u \qquad (1)$$

where D_{m} is the diffusivity of the solute in the mobile phase (cm^2/s), D_{s} is the diffusivity of the solute in the stationary phase (cm^2/s), d_{c} is the column inner

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diameter (cm), d_f is the film thickness (cm), u is the velocity of the mobile phase (cm/s) and k' is the capacity factor of the solute. For larger bore (I.D. $\geqslant 50$ μm) and thin-film (thickness $\leqslant 0.25$ μm) columns, the Golay equation can be simplified to the following forms:

$$H = \frac{2D_m}{u} + \frac{1 + 6k' + 11k'^2}{96(1 + k')^2} \cdot \frac{d_c^2}{D_m} \cdot u \qquad (2)$$

or, for $k' = 0$,

$$H = \frac{2D_m}{u} + \frac{1}{96} \cdot \frac{d_c^2}{D_m} \cdot u \qquad (3)$$

The optimum velocity (u_{opt}), at which the plate height reaches the minimum, is given by

$$u_{opt} = (1 + k') \cdot \frac{D_m}{d_c} \left(\frac{192}{1 + 6k' + 11k'^2} \right)^{1/2}$$

or, for $k' = 0$,

$$u_{opt} = 13.86 \cdot \frac{D_m}{d_c} \qquad (4)$$

The analysis time for a practical separation problem is given by

$$t_R = N \cdot \frac{H}{u} \cdot (1 + k') = N \cdot \frac{1 + 6k' + 11k'^2}{96(1 + k')} \cdot \frac{d_c^2}{D_m} \qquad (5)$$

where N is the plate number needed for the separation. From eqns. 4 and 5, it is seen that both a decrease in column diameter and an increase in D_m improve the efficiency. For aqueous systems, an increase in column temperature from 20 to 200°C has the same effect in reducing the analysis time as a decrease in column diameter from 50 to 12 μm, because D_m increases with increasing column temperature [$D_{m200} = D_{m20}(\eta_{20}/\eta_{200})$ (473/293) = 17.47 D_{m20}, where D_{m200} and D_{m20} are the diffusivities of the solute and η_{200} and η_{20} are the viscosities of water at 200 and 20°C, respectively].

The effect of column temperature on efficiency has been studied by many workers on conventional RP-LC columns [16–19]. Antia and Horváth [20] performed an in-depth theoretical analysis of the effect of elevated column temperature on column performance for both porous and pellicular packings. Erni [21] recently pointed out not only that elevated column temperature is beneficial for packed-column chromatography, but also HPLC in open tubes is more feasible at higher temperatures even when the inner diameters are in the range 50–100 μm. He also showed that the strong dependence of the column efficiency on the k' values is much less noticeable at higher temperatures.

Even though the beneficial effect of higher column temperatures in OTLC is reasonable in theory, this has not so far been proved experimentally. The aim of this work was to verify the benefits predicted by OTLC theory for elevated column temperatures, study the influences of various instrumental and operating parameters, realize high-efficiency OTLC separations with commercially available columns and instruments and finally develop a prototype of high-temperature (HT) OTLC equipment that can be transferred to other users. This paper reports the experimental set-up and our preliminary results.

EXPERIMENTAL

The layout of the experimental unit is shown in Fig. 1. It consisted of two Kontron Model 420 HPLC pumps, a Rheodyne Model 7125 sample valve, a split tee-piece (Valco 1/16-in. tee, 0.01-in. bore), a capillary column, a Kratos Model 783 UV detector and a 0.5-l stainless-steel cylinder (Model DOT3E1800, Swagelok) which was used for generating a back-pressure. A 20-μl loop was used for the 20-m column and a 5-μl loop was used for other

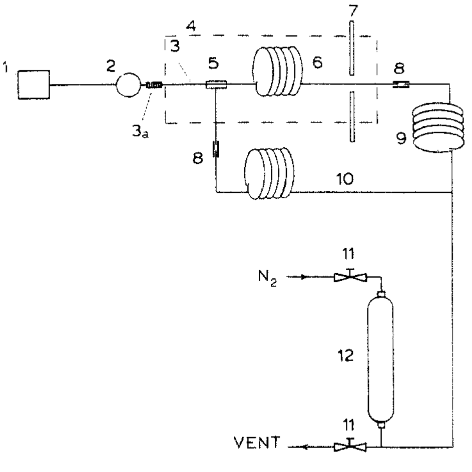

Fig. 1. Experimental set-up. 1 = HPLC pump; 2 = sample valve; 3 = preheating tube; 3a = heater; 4 = oven; 5 = split tee-piece; 6 = capillary column; 7 = optical fibre; 8 = zero dead-volume connector; 9 = restriction tube; 10 = split tube; 11 = needle valve; 12 = back-pressure vessel.

columns. A 3-mm segment of the polyimide coating of the capillary column (about 12 cm from the column end) was burned off to make a window for UV detection. The Kratos UV detector was modified in order to work with optical fibres (Superguide SFS 200/240 T; Luxmatic, Baar, Switzerland) to carry out on-column detection. A Valco 0.01-in. bore cross was used as detector cell where the capillary column passed through one channel, and the source and the collecting optical fibres were positioned on opposite sides of the window.

The split tee-piece, the capillary column and the detector cell were placed in an oven of our construction which was controlled by a temperature regulator (Digitemp mC 5100; Schuntermann & Benninghoven, Hilden, Switzerland). A 40 cm × 0.2 mm I.D. serpentine tube (Scientific Systems, State college, PA, USA) was used as the preheating tube between the injection valve and the split tee-piece. This was heated separately. The preheating temperature was always 4–6°C higher than the column temperature used. The coil diameter was 8.5 cm for all columns.

Fused-silica tubes of 2.0 m × 100 μm I.D. and 2.4 m × 18.5 μm I.D. were used as the split tube and the restriction tube (components 10 and 9 in Fig. 1), respectively, to give a splitting ratio of about 1000.

SB Series columns of 50 and 100 μm I.D. and of different lengths were obtained from Lee Scientific (Salt Lake City, UT, USA) and fused-silica capillaries of different sizes from Polymicro Technologies (Phoenix, AZ, USA). Acetonitrile and methanol were HPLC-grade solvents from Rathburn (Walkerburn, UK). All other chemicals were of analytical-reagent grade from Fluka (Buchs, Switzerland) and were used as received.

Data collection and processing were carried out on a Perkin-Elmer CLAS system. Sodium nitrite in water or in methanol was used to determine column dead volumes. Other water-soluble substances, such as thiourea, can also be used; they all gave the same dead volume value.

RESULTS AND DISCUSSION

HT-OTLC system

In order to check the working performance of the high-temperature split system, the reproducibility of column flow-rate was determined at different splitting ratios and at different temperatures up to 200°C. In most instances the relative standard deviation of the column flow-rate was between 0.3 and 0.6%. This precision indicates that the stability of the flow-rate and the temperature control system is acceptable for most practical applications.

When very long columns (16–20 m) were used, especially new columns, partial blockage of the restriction tube by column bleeding may bring larger variations in column flow-rate (sometimes up to 5–10%). Even under these unfavourable conditions the relative standard deviations of the capacity factors were still in the range 0.4–0.6%, except for the early eluting solutes (Table I). These results show that the working performance of the HT-OTLC system is satisfactory.

Extra-column band broadening in HT-OTLC system

In the development of a high efficiency OTLC system, the study of extra-column band broadening is of great importance. The total peak variance, σ^2, is the sum of the column variance and all other independent extra-column variances:

$$\sigma^2 = \sigma_c^2 + \sigma_d^2 + \sigma_t^2 + \sigma_{inj}^2 + \sigma_s^2 \qquad (6)$$

where σ_c^2 is the column variance, σ_d^2 is the variance caused by detector cell volume, σ_t^2 is the variance caused by the preheating tube, σ_{inj}^2 is the variance caused by injection volume and σ_s^2 is the contribution of all other factors, such as dead volumes between connections, electrical response delay, insufficient preheating and column diameter inhomogeneity.

In our system with on-column detection, the detector dead volume is in the range 0.16–0.4 nl, depending on the column diameter. Its contribution to the observed peak dispersion is negligible.

For a rectangular sample plug of volume V_{inj}, using the manual peak half-width method, $\sigma_{inj}^2 = 0.25V_{inj}^2$ [22]. This relationship is also valid in HT-OTLC (Fig. 2). If a 20% efficiency loss is the acceptable limit, it can be calculated that an 8-nl injection volume is allowed for a 1.2 m × 50 μm I.D. column in fast analysis and a 30-nl volume can be used for a 16-m column.

It has also been proved in HT-OTLC [23] that σ_t^2 can be calculated by the equation

$$\sigma_t^2 = \sigma_{tube}^2/(SR)^2 \qquad (7)$$

TABLE I

PRECISION OF THE CAPACITY FACTORS (k')

Column, SB-Methyl-100 (20 m × 50 μm I.D.); temperature, 180°C; mobile phase, acetonitrile–water (50:50); flow-rate, 0.66 μl/min.

Injection No.	k'					
	Benzene	Chlorobenzene	1,4-Dichlorobenzene	1,3,5-Trichlorobenzene	1,2,4,5-Tetrachlorobenzene	Petachlorobenzene
1	0.0278	0.0329	0.0426	0.0759	0.0835	0.1077
2	0.0282	0.0331	0.0428	0.0761	0.0834	0.1074
3	0.0279	0.0331	0.0427	0.0766	0.0842	0.1089
4	0.0271	0.0322	0.0424	0.0762	0.0840	0.1086
5	0.0277	0.0326	0.0425	0.0760	0.0835	0.1082
6	0.0279	0.0330	0.0429	0.0766	0.0843	0.1090
7	0.0276	0.0326	0.0426	0.0762	0.0836	0.1080
Mean	0.0277	0.0328	0.0426	0.0762	0.0838	0.1083
S.D.[a]	±0.00034	±0.00033	±0.00017	±0.00028	±0.00037	±0.00061
R.S.D.[a] (%)	1.2	1.0	0.4	0.4	0.4	0.6

[a] S.D. = Standard deviation; R.S.D. = relative standard deviation.

where σ^2_{tube} is the band broadening in the preheating tube calculated by the Taylor equation [1,2] and SR is the splitting ratio. For efficient preheating, a 40 cm × 0.2 mm I.D. tube was used in our work. This size is much larger than that for a normal connection tube. Calculation shows that even for the 1.2-m column, the efficiency loss caused by this tube is less than 4%, provided that a splitting ratio of about 1000 is used.

The term σ_s^2 is hard to predict and can only be evaluated experimentally. In Fig. 2, the difference between the theoretical column variance σ_c^2, and the intercept of the σ^2 *vs.* V_{inj}^2 plot with the ordinate is about 25.2 nl^2. Although this value contains the contribution of the preheating tube, it is a good estimation of σ_s^2, as σ_t^2 is insignificant. The small σ_s^2 value indicates that other extra-column factors and factors which effect the column efficiency itself do not contribute much to the overall band broadening. Therefore, the HT-OTLC system can be used in high-efficiency separations.

Influence of mobile phase preheating on column efficiency

Many workers [24,25] have noticed that the effect of temperature on column efficiency is strongly influenced by the mobile phase preheating. Only when the mobile phase entering the column is at the same temperature as the column can the beneficial effect of temperature be fully exploited. Any "cold point" in the column inlet must be removed. For a

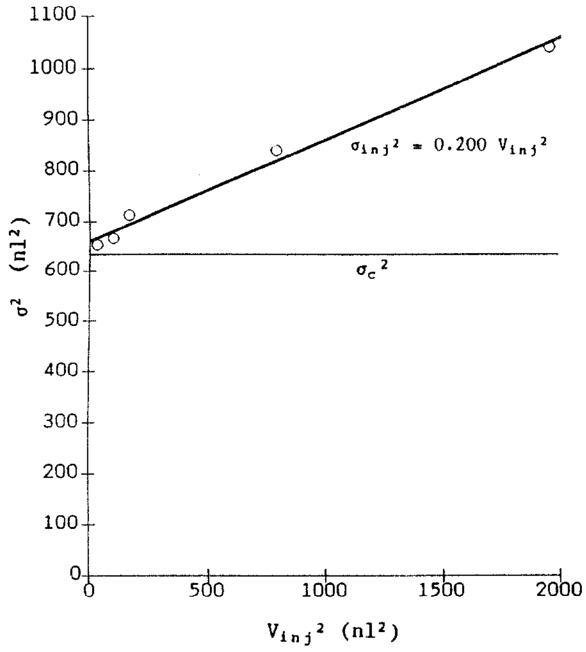

Fig. 2. σ^2 *vs.* V_{inj}^2 plot. Column, 8.4 m × 51 μm I.D. SB-Octyl-50; temperature, 100°C; mobile phase, methanol; u = 0.27 cm/s; sample, 0.2% acetophenone in methanol.

better understanding of this influence, a quantitative study of the effectiveness of different heating methods was carried out.

In Fig. 3, the H *vs.* u curves obtained for different heating methods are presented, together with the theoretical curve. The vertical distance between the experimental and theoretical curves is an indication of efficiency loss:

$$\text{loss}\,(\%) = [(H_{\text{obs}} - H_{\text{c}})/H_{\text{obs}}] \cdot 100 \qquad (8)$$

where H_c is the theoretical and H_{obs} the experimental plate height. The most effective heating method should give the least loss. If the efficiency loss is calculated at a relatively high linear velocity (*e.g.*, at u = 1.5 cm/s), the contribution of other extra-column factors becomes less important. The calculated values thus reflect the effectiveness of different heating methods. Table II gives the results; the effectiveness of different heating methods is tabulated without separating the contributions of the preheating tube and the additional heating tube before the injection valve.

According to Table II, the simultaneous mobile phase heating before and after the sample valve is the most effective way to diminish the "cold point" effect. However, this is not feasible for higher operating temperatures (150–200°C) owing to the need for a high-temperature sample valve. The serpentine tube performs very well in view of mobile phase heating, although its variance contribution is still significant (0.05 μl^2/cm). This, however, does not cause appreciable problems even for the 1.2-m column, provided that the splitting ratio is about 1000 or higher.

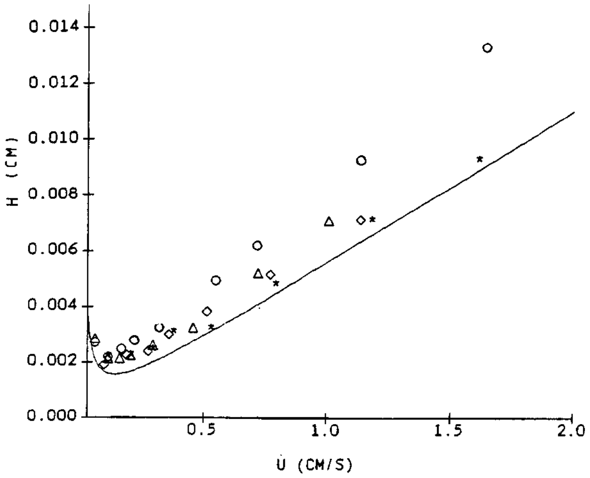

Fig. 3. Influence of heating methods on column efficiency. Column, 124 cm × 54 μm I.D. SB-Octyl-50; temperature, 100°C; mobile phase, methanol; sample, acetophenone in methanol. Solid line: theoretical H *vs.* u curve. Experimental points: ○ = method A; △ = method B; ◇ = method C; ✱ = method D in Table II.

Validity of OT theory in HT-OTLC

A fused-silica colmn (85 cm × 75 μm I.D.) was

TABLE II

EFFECTIVENESS OF DIFFERENT HEATING METHODS

Column, 124 cm × 54 μm I.D.; temperature, 100°C; mobile phase, methanol; u = 1.5 cm/s.

Method	Additional heating tube before sample valve	Preheating tube	Column oven heating medium	Loss (%)
A	None	12 cm × 0.52 mm I.D. stainless steel, in oven	Air	31.9
B	60 cm × 0.5 mm I.D. stainless steel, in oven	100 cm × 75 μm I.D. fused silica, in oven	Metal powder	18.9
C	None	40 cm × 0.2 mm I.D. serpentine tube, separate heating, 105°C^{a}	Metal powder	9.2
D	40 cm × 0.2 mm I.D. serpentine tube, separate heating, 105°C	20 cm × 0.12 mm I.D. stainless steel, separate heating, 105°C^{a}	Metal powder	5.1

[a] Measured at the tube wall surface.

used to show the influence of the column temperature on column efficiency. Methanol was used as the mobile phase to eliminate possible retentions caused by column wall adsorption. As $k' = 0$, the H *vs.* u curves obtained at different column temperatures (20, 100 and 150°C, respectively; Fig. 4) simply indicate the influence of temperature. As can be seen from Fig. 4, the H *vs.* u curve became much flatter in the high-velocity region when the column temperature increased from 20 to 150°C.

The diffusivity of the solute (acetophenone) at the three temperatures is $1.64 \cdot 10^{-5}$, $5.55 \cdot 10^{-5}$ and $10.16 \cdot 10^{-5}$ cm^2/s, respectively (calculated from the Wilke–Chang eqation). Eqn. 4 gives u_{opt} values of 0.030 (20°C), 0.103 (100°C) and 0.188 cm/s (150°C). From the curves in Fig. 4, the corresponding $_{opt}$ values were found to be *ca.* 0.03, 0.09 and 0.19 cm/s. The experimental results confirmed the theoretical predictions.

At room temperature, the observed plate height becomes noticeably smaller than the theoretical value at velocities above 0.3 cm/s. Knox nd Gilbert [7] reported similar results and attributed this effect to the additional mixing due to the secondary flow. At higher column temperatures this has not been observed. Perhaps the "cold point" effect caused by insufficient heating predominated over the secondary flow effect.

To verify the correctness of the retention correction expression, the H *vs.* u dependence of naphthalene and tropolone on an SB-Octyl-50 colmn was studied. They have identical D_m vales but different retentions. The H *vs.* u curves were measured at 22 and at 100°C, respectively (Fig. 5). As the D_m value is the same, the ratio of the slopes of their respective H *vs.* u curves equals the retention correction factor according to eqns. 2 and 3.

Tropolone had no retention under the chromatographic conditions used and the capacity factor of naphthalene was 0.354 (22°C) and 0.303 (100°C). The calculated slope ratio of retained and unretained solutes was 2.46 (22°C) and 2.25 (100°C), while the experimentally obtained slope ratio was 2.40 (22°C) and 2.18 (100°C). The agreement was fairly good and confirms the validity of the retention correction factor. If the peak shape of naphthalene and tropolone obtained at room temperature is compared with that at 100°C (Fig. 6), it is clear that the influence of retention on efficiency is much less at higher temperatures.

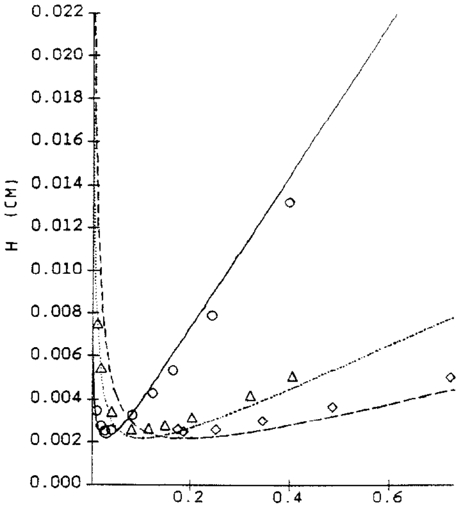

Fig. 4. Influence of temperature on column efficiency. Column, 0.85 m × 75 μm I.D. fused silica; mobile phase, methanol; sample, acetophenone in methanol. Solid line, theoretical curve at 22°C; ○ = experimental data at 22°C. Dotted line, theoretical curve at 100°C; △ = experimental data at 100°C. Dashed line, theoretical curve at 150°C; ◇ = experimental data at 150°C.

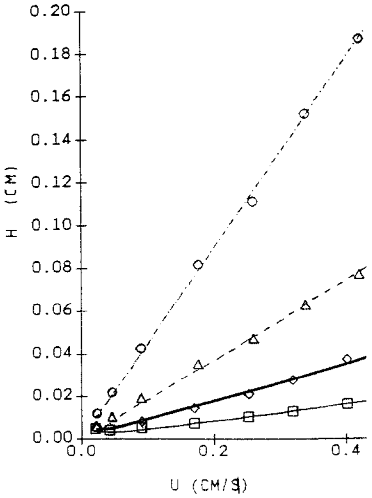

Fig. 5. Influence of retention on column efficiency. Column, 121 cm × 100 μm I.D. SB-Octyl-50. Mobile phase: broken lines, methanol–water (50:50), 22°C, ○ = naphthalene ($k' = 0.354$) and △ = tropolone ($k' = 0$); solid lines, methanol–water (30:70), 100°C, ◇ = naphthalene ($k' = 0.303$) and □ = tropolone ($k' = 0$).

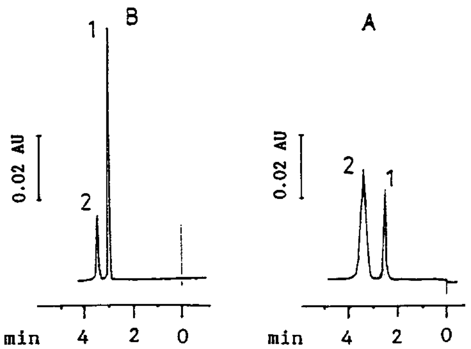

Fig. 6. Influence of temperature on peak shape. Column, 121 cm × 100 μm I.D. SB-Octyl-50. Mobile phase: (A) methanol–water (50:50), 22°C; (B) methanol–water (30:70), 100°C. Peaks: 1 = tropolone; 2 = naphthalene.

The performance $P = N/t$, as expressed by the plate numbers generated in unit time for an unretained peak with retention time t_0, is given by

$$\frac{N}{t_0} = \frac{48D_m}{d_c^2} \tag{9}$$

for $u = u_{opt}$, or

$$\frac{N}{t_0} = \frac{96D_m}{d_c^2} \tag{10}$$

for $u \geqslant 3u_{opt}$. To obtain the highest plate number for a certain column, one must work at $u = u_{opt}$. In order to obtain the highest analysis speed, one must work at a higher velocity. Eqn. 10 indicates that the analysis speed reaches a maximum and becomes independent of the velocity after $u \geqslant 3\ u_{opt}$. To prove this conclusion, the experimental N/t_0 values obtained on a 54 μm I.D. column at 100°C and on a 51 μm I.D. column at 150°C are listed in Table III. The theoretical N/t values are also given. According to the results, compared with the speed at $u = u_{opt}$, a doubled analysis speed was obtained experimentally at $u = 3u_{opt}$, as predicted by eqns. 9 and 10.

High-efficiency separations

The minimum plate height, H_{min}, can be calculated by the equation

$$H_{min} = \frac{1}{1+k'}\left(\frac{1 + 6k' + 11k'^2}{12}\right)^{1/2} d_c$$

or, for $k' = 0$,

$$H_{min} = 0.2887d_c \tag{11}$$

If 50 μm I.D. columns are used, 10^6 plates could be obtained on a 14.5-m column. If a 10–20% efficiency loss is taken into consideration, a 16–17-m column will be long enough to generate this order of plate number. On a 19.6 m × 51.2 μm I.D. SB-Methyl-100 colmn operated at 200°C with methanol as the mobile phase and with acetophenone as the test sample, the following results were obtained as averages of six injections: retention time t_R = 49.32 ± 0.34 min, N = 1 143 000 ± 39 000 plates and N/t = 386 ± 11 plates/s. The efficiency obtained shows a 14% loss relative to the theoretical value.

One million plates have also been realized in the separation of chlorobenzenes (Fig. 7). All peaks are highly symmetrical, indicating the excellent performance of the system.

TABLE III

PERFORMANCE N/t AS A FUNCTION OF LINEAR VELOCITY u

System[a]	Parameter	Values							
A	u (cm/s)	1.61	1.18	0.785	0.521	0.372	0.289	0.200	0.106
	t_0 (min)	1.28	1.75	2.63	3.96	5.54	7.15	10.30	19.40
	N	13 400	17 600	26 100	39 200	40 800	51 900	56 600	57 700
	N/t_0 (s^{-1})	175	167	165	165	123	121	92	50
B	u (cm/s)	1.53	1.05	0.710	0.505	0.391	0.273	0.256	
	t_0 (min)	9.15	13.3	19.8	27.8	35.9	51.4	54.6	
	N	160 000	243 000	334 000	406 000	442 000	494 000	470 000	
	N/t_0 (s^{-1})	291	305	282	244	205	160	143	

[a] (A) Column, 123.7 cm × 54.07 μm I.D. SB-Octyl-50; temperature, 100°C; D_m = $5.55 \cdot 10^{-5}$ cm²/s; u_{opt} = 0.142 cm/s; N/t = 91 ($u = u_{opt}$), 182 ($u = 3u_{opt}$). (B) Column, 842 cm × 51.43 μm I.D. SB-Octyl-50; temperature, 150°C; D_m = $10.16 \cdot 10^{-5}$ cm²/s; u_{opt} = 0.274 cm/s; N/t = 185 ($u = u_{opt}$), 369 ($u = 3u_{opt}$).

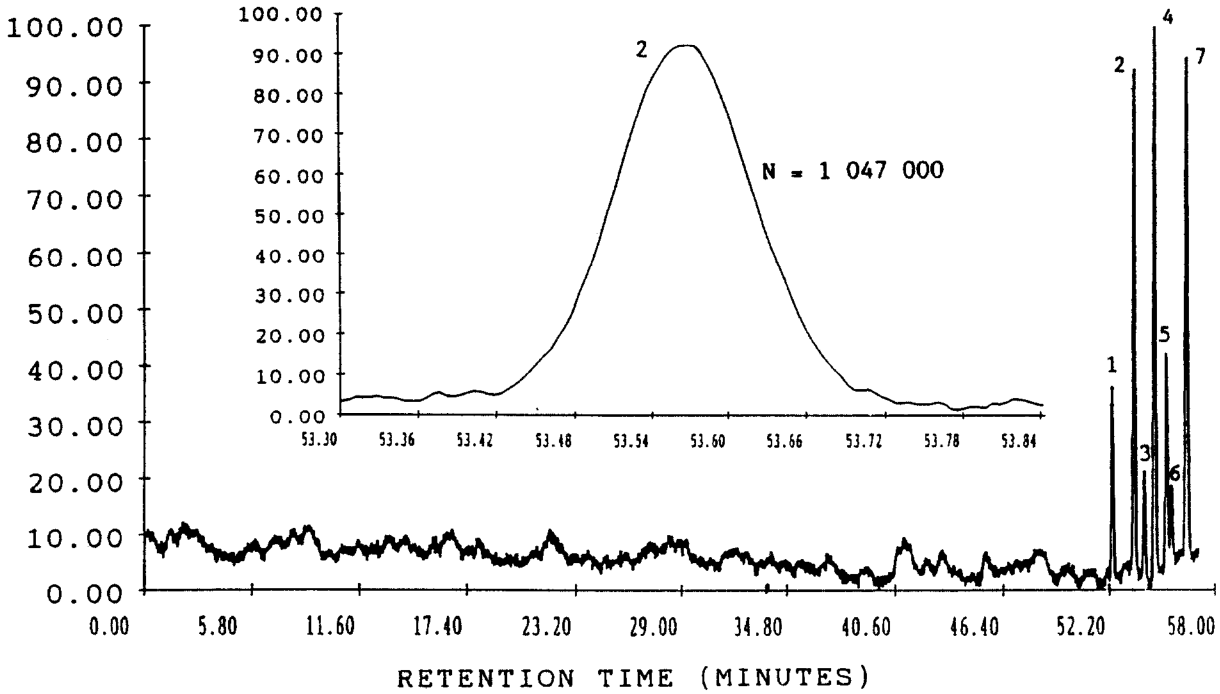

Fig. 7. Separation of chlorobenzenes. Column, 20 m × 51 μm I.D. SB-Methyl-100; temperature, 200°C; mobile phase, acetonitrile–water (50:50); flow-rate 0.65 μl/min. Peaks: 1 = tropolone; 2 = benzene (N = 1 047 000); 3 = 1,4-dichlorobenzene (N = 922 000); 4 = 1,2,4-trichlorobenzene; 5 = 1,3,5-trichloro-benzene (N = 800 000); 6 = 1,2,4,5-tetrachlorobenzene; 7 = pentachlorobenzene (N = 692 000).

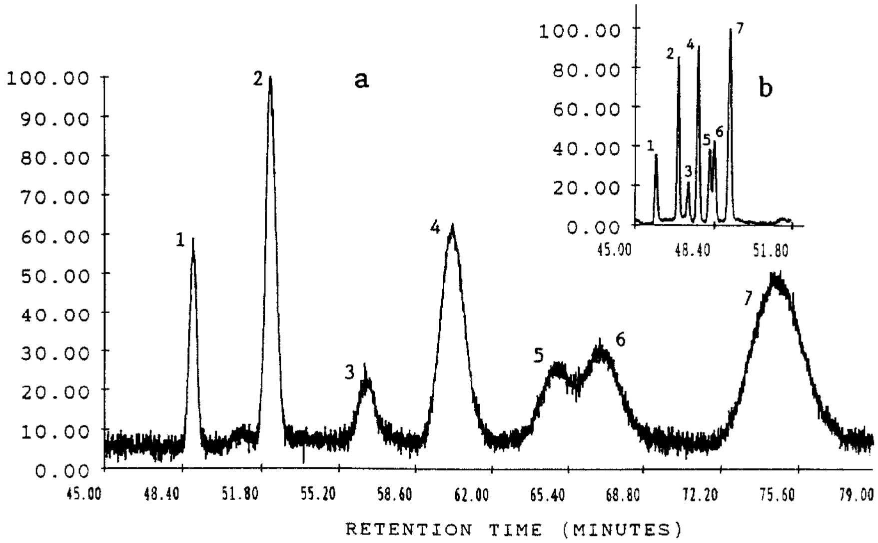

Fig. 8. Comparison of separations at different column temperatures: (a) 22°C; (b) 200°C. Chromatographic conditions (except temperature and flow-rate 0.75 μl/min) and peak identities as in Fig. 7.

Fig. 8 shows two chromatograms obtained under the same chromatographic conditions except for the column temperature: (a) room temperature and (b) 200°C. Although the k' value of the last peak at 200°C is about one seventh of the value at room temperature, the separation does not suffer, owing to the high efficiency obtained at elevated temperature.

CONCLUSIONS

Our preliminary results have shown that the existing OTLC theory is applicable in HT-OTLC. All practical benefits predicted by the theory for a higher column temperature are experimentally verified. At higher temperature the serious limitation on analysis speed due to a linear flow-rate is eased, and high-efficiency separations can now be performed in a realistic analysis time. At present, the highest analysis speed obtained on our system is 395 plates/s, or 10^6 plates in *ca.* 42 min (51 μm I.D. column operated at 200°C).

Another advantage of a higher column temperature is a higher total efficiency. The reduced mobile phase viscosity at higher temperature allows the use of very long columns. On a 19.6 m × 51 μm I.D. column, the highest efficiency obtained was 1 180 000 plates; this represents only an 11% loss relative to the theoretical value (1 326 000 plates).

In the separation of chlorobenzenes, 10^6 plates were obtained on a 20-m column in less than 60 min. It is therefore possible to obtain 40 000–50 000 plates in 3 min on a 1.2-m column. This efficiency could meet the demands of most routine work.

Studies on the stabilities and selectivities of different column types and studies of rapid separations on short columns are in progress.

ACKNOWLEDGEMENT

The authors thank Vladimir Sedmak for technical assistance in designing the column oven.

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