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# High speed solvating gas chromatography using packed capillaries containing sub-5 μm particles

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**Abstract**

In this study, fused-silica capillaries of 250 μm I.D. were packed with spherical porous and nonporous octadecyl bonded silica particles having diameters of 1.5 and 3 μm. These capillaries were used with $CO_2$ as mobile phase at elevated temperatures. At the column inlet, the mobile phase was a supercritical fluid, while at the column outlet, it was a gas. The mobile phase gradually changed from a supercritical fluid to a gas within the column. Because the mobile phase exhibited solvating ability for the analytes being separated, in contrast to conventional gas chromatography (GC) in which the mobile phase is only a carrier, this variation has been named "solvating gas chromatography" (SGC). Using this technique, capillaries packed with small particles produced column efficiencies as high as 1200 plates $s^{-1}$ for retained solutes. Therefore, packed capillary SGC is the method of choice to carry out high speed GC separations. The effects of column inlet pressure, column length and particle characteristics on mobile phase linear velocity, retention factor, column efficiency, speed of analysis and resolution were investigated. © 1997 Elsevier Science B.V.

*Keywords:* Solvating gas chromatography; Capillary columns; Linear velocity; Alkanes

## 1. Introduction

Although in the years between 1960–1980 several research groups investigated, both theoretically and practically, the potential of packed column gas chromatography (GC) using microparticles as packing materials [1–8], the rapid development of open tubular column technology delayed the study and the use of these advanced packing materials in GC. The excellent column permeability of open tubular columns makes it possible to use long columns to obtain extremely high total column efficiencies and to carry out separations of complex samples [9]. Currently, there is no doubt that packed columns cannot reach total column efficiencies as high as open tubular columns in GC.

However, packed columns containing microparticles can provide higher column efficiency per unit column length and greater retention of solutes than open tubular columns [10]. These two factors make packed columns ideal for fast separations.

In liquid chromatography (LC), extremely small particles with diameters less than 5 μm have been used for fast separations [11–14]. These small particles provide high column efficiency, large optimum mobile phase linear velocity and only gradual loss in column efficiency with increasing mobile phase linear velocity. The main practical problem resulting from the use of small particles in LC is the large pressure drop along the column, which imposes special requirements on the LC instrumentation to handle high pressures. In supercritical fluid chromatography (SFC), the effect of the pressure drop on chromatographic performance is relatively complex

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[15–18] and affects both column efficiency and retention of solutes. A compromise in the selection of particle size must be made to carry out fast separation when considering the effects of pressure drop on column efficiency, retention and resolution [19]. However, in GC, the carrier gas has little effect on the retention of solutes except as a result of its role as carrier.

The use of microparticles and the resultant high pressures in packed column GC introduces a practical difficulty in sample introduction. However, the well developed sample injection valves with small sample loops used in LC and SFC can minimize this problem [20]. The influence of the mobile phase linear velocity gradient on column efficiency was analyzed by Giddings et al. and they introduced a compressibility constant to correct for the loss in efficiency [1,2]. Even with the maximum compressibility correction using Giddings approach (i.e., 1.125), little loss in column efficiency would be expected. However, until now, few reports of high column efficiencies with reduced plate heights of 2–3, as are commonly obtained in LC, have been reported when using microparticles as packing materials in packed column GC. It seems that the effect of mobile phase linear velocity on column efficiency is more significant than theoretically estimated.

It should also be noted that the properties of the mobile phase can affect the chromatographic performance in packed column GC. It was experimentally shown that $CO_2$ as carrier gas could provide higher column efficiencies per unit time than lighter carrier gases in packed column GC [8] and, therefore, was investigated for use in high speed separations [21]. The use of gaseous $CO_2$ is limited by its relatively low critical pressure (72.9 atm; 1 atm= 101 325 Pa). When the pressure at the column inlet is greater than this value, the mobile phase in the column changes from a supercritical fluid at the column inlet to a gas at the column outlet. In this situation, the mobile phase has an obvious influence on the retention factors of solutes because of its solvating power. Therefore, we have named this variation of GC, "solvating gas chromatography" (SGC). SGC is different from SFC in which the elution of solutes depends mostly on their solubilities in the mobile phase. It is also different from conventional packed column GC in which the high pressure mobile phase only acts as a carrier.

In this study, porous (3 μm diameter) and nonporous (1.5 and 3 μm diameters) octadecyl-bonded silica particles were packed into fused-silica capillaries with an inner diameter of 250 μm. The relative performance of these packed capillaries in high speed SGC was investigated.

## 2. Experimental

### 2.1. Materials and instrumentation

Spherical porous octadecyl bonded silica (P-ODS) particles having a diameter of 3 μm and 80 Å pore size were purchased from Phase Separations (Norwalk, CT, USA). Spherical nonporous octadecyl bonded silica (NP-ODS) particles with diameters of 1.5 and 3 μm were obtained from Micra Scientific (Northbrook, IL, USA). Fused-silica capillary tubing was purchased from Polymicro Technologies (Phoenix, AZ, USA). Column connections were made using polyether ether ketone (PEEK) tubing and zero dead volume unions (Valco Instruments, Houston, TX, USA). Packing of the columns and GC experiments were carried out using a Lee Scientific Model 600 SFC instrument (Dionex, Salt Lake Division, Salt Lake City, UT, USA). SFC grade $CO_2$ (Scott Specialty Gases, Plumsteadville, PA, USA) was used for the preparation of packed capillary columns and as the mobile phase. Helium was used as the carrier gas in high pressure GC experiments conducted for comparison. Other chemicals used were purchased from Sigma (St. Louis, MO, USA) and Aldrich (Milwaukee, WI, USA).

### 2.2. Preparation of packed capillary columns

A similar packing procedure to that previously described [22] was used for preparation of the packed capillary columns. One end of the fused-silica capillary column was connected to a zero dead-volume union using PEEK tubing to position a stainless-steel screen (2 μm pores, Valco) to support the particles. The other end of the column was connected to a stainless-steel vessel, in which microparticles were introduced. For the preparation of capillaries packed with 1.5 μm nonporous octadecylsilane (NP-ODS) microparticles, the stainless-steel screen was placed at the other end of the union

before a 10 cm×25 μm I.D. fused-silica capillary tube. The column was placed in an ultrasonic bath and the packing process was carried out using $CO_2$ at room temperature and by slowly increasing the pressure from 50 to 200 atm in 30 min. After packing, the column was conditioned at 200 atm and room temperature with a $CO_2$ purge for 10 min while it was still in the ultrasonic bath (1 atm = 101 325 Pa). Finally, the column was slowly depressurized overnight.

### 2.3. SGC experiments

SGC experiments were carried out using a Lee Scientific Model 600 SFC instrument. A manual liquid injector (Valco Instruments) with a rotor volume of 0.2 μl was used for introduction of the samples. A tee which served as an injection splitter was connected to the injector valve using a 10 cm×125 μm I.D. stainless-steel tube. A 5 cm×20 μm I.D. fused-silica capillary column was used as the split line. The separation column was connected to the tee using PEEK tubing. A 10 cm×50 μm I.D. fused-silica capillary was used to connect the separation column to the flame ionization detector (FID). However, for capillaries packed with 1.5 μm particles, a 10 cm×25 μm I.D. fused-silica capillary was used to connect the column to the FID. $CO_2$ was used as the mobile phase in SGC, and its pressure was controlled using the SFC pump. The outlet of the pump was connected to the valve injector using a 50 cm×1 mm I.D. coiled stainless-steel preheater tube in the oven. Helium was used as the carrier gas for high pressure GC, and a cylinder containing helium at a pressure of 100 atm was directly connected to the valve injector through the preheater tube. All SGC experiments were carried out at constant temperature and constant average mobile phase linear velocity.

## 3. Results and discussion

### 3.1. SGC versus high pressure GC

In high pressure GC, the carrier gas has little effect on the retention of solutes other than as a carrier. The choice of carrier gas in GC is mainly dependent on its viscosity and the diffusion of solutes within it. Generally, light, permanent gases such as helium and hydrogen have low viscosities and provide the greatest diffusion of solutes in them. In SGC, the solvating power of the mobile phase produces relatively strong interactions with solutes and often with the stationary phase, and these interactions significantly affect solute retention. Fig. 1 shows chromatographic results of SGC and high pressure GC using a 3 μm NP-ODS particle packed capillary under the conditions of 100 atm column inlet pressure and 130°C. As can be seen, significantly different results were obtained. In high pressure GC, only a poor $CS_2$ peak was obtained in comparison to an excellent separation from SGC. Even by increasing the oven temperature up to 180°C, only the $CS_2$ peak eluted in high pressure GC. This probably resulted from the extremely large retention factors of solutes in high pressure GC, while the solvating power of the mobile phase in SGC greatly reduced the retention.

### 3.2. Generation of mobile phase flow for various columns

In SGC, the mobile phase linear velocity can be increased by increasing the column inlet pressure. When average values of linear velocity and viscosity are used, the relationship between the pressure drop of the mobile phase along the column and the mobile phase linear velocity can be expressed as follows [23]:

$$\Delta P = \frac{\phi \eta u L}{d_p^2} \tag{1}$$

In SGC, the column outlet pressure remains at 1 atm, and the column inlet pressure ($P_i$) is much higher than 1 atm. Therefore, Eq. (1) can be expressed as follows:

$$P_i = \frac{\phi \eta u L}{d_p^2} \tag{2}$$

From this equation, it is easily seen that the required column inlet pressure to obtain a certain mobile phase linear velocity ($u$) depends on the column length ($L$), the viscosity of the mobile phase ($\eta$), the column permeability ($\phi$) and the particle size ($d_p$). Since highly compressible mobile phases are used in SGC, the $u$ and $\eta$ parameters in Eqs. (1) and

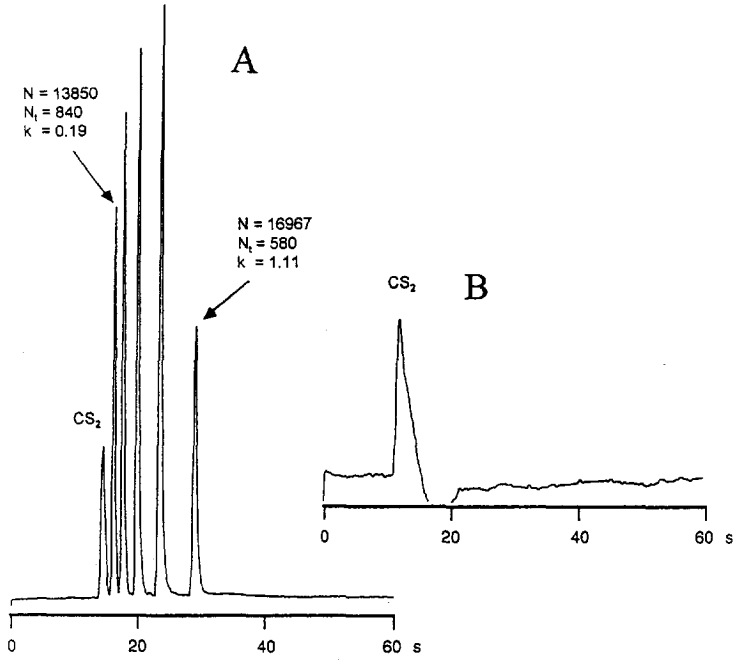


Fig. 1. Comparative chromatograms obtained using (A) SGC and (B) high pressure GC. Conditions: 30 cm×250 μm I.D. fused-silica capillary packed with 3 μm NP-ODS; 130°C; 100 atm column inlet pressure; FID; *n*-hydrocarbons from *n*-octane to *n*-dodecane; (A) $CO_2$ and (B) He mobile phases.

(2) should represent average values along the column.

Since $P_i$ is proportional to the inverse of the square of $d_p$, columns containing sub-5 μm particles require very large column inlet pressures. A previous study showed that columns containing nonporous particles provided better column permeability than columns packed with porous particles under GC, SFC and LC conditions, which was explained by the reasoning that columns containing nonporous particles were free from stagnant mobile phase which occurs in the pores of the porous particles [24].

At a specific column inlet pressure, the resultant mobile phase linear velocity is also dependent on the mobile phase viscosity. In contrast to high pressure GC, the mobile phase at the column inlet in SGC has solvating power. The relatively high viscosity of the supercritical mobile phase at the inlet imposes an increased column inlet pressure in SGC in order to obtain the desired mobile phase linear velocity. Of course, the required column inlet pressure is also dependent on the column length.

In this work, no separation between methane and ethane could be obtained using columns packed with either P-ODS or NP-ODS particles. Therefore, it was assumed that methane had no retention on these columns, and could be used as a marker to measure the dead time. Fig. 2 shows the relationship between column inlet pressure and mobile phase linear velocity for various columns. From this figure, it can be seen that an approximate linear relationship existed between the column inlet pressure and the mobile phase linear velocity. When 3 μm particles and the same inlet pressure were used, the column containing NP-ODS particles produced a higher mobile phase linear velocity than a column of the same length packed with P-ODS particles. For example, at 130 atm column inlet pressure, mobile phase linear velocities of 1.5 and 4.2 cm $s^{-1}$ were obtained for 20 cm columns packed with 3 μm P-ODS and NP-ODS particles, respectively. A 57 cm column packed with NP-ODS particles gave similar results of $u$ versus $P_i$ as a 20 cm column containing P-ODS particles of the same size.

It was also found that a greater dependence of mobile phase linear velocity on column inlet pressure

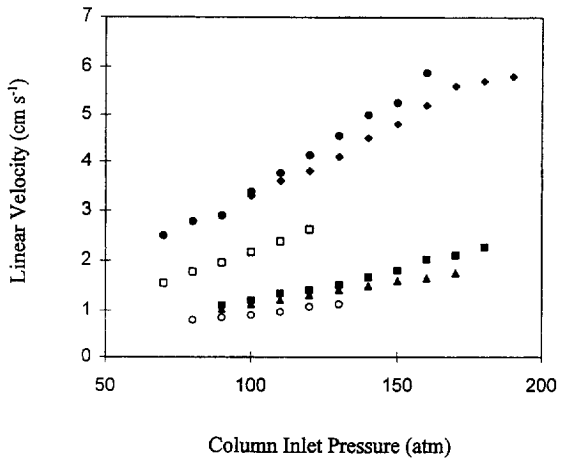


Fig. 2. Relationship between mobile phase linear velocity and column inlet pressure in SGC. Conditions: $CO_2$; 130°C; FID; methane used as unretained marker; (●) 20 cm×250 μm I.D. capillary packed with 3 μm NP-ODS particles; (♦) 10 cm×250 μm I.D. capillary packed with 3 μm P-ODS particles; (□) 30 cm×250 μm I.D. capillary packed with 3 μm NP-ODS particles; (■) 20 cm×250 μm I.D. capillary packed with 3 μm P-ODS particles; (▲) 57 cm×250 μm I.D. capillary packed with 3 μm NP-ODS particles; and (○) 20 cm×250 μm I.D. capillary packed with 1.5 μm NP-ODS particles; linear velocity as calculated from $\mu = L/t_0$.

existed for columns containing P-ODS particles, and shortening the column length had a more significant effect on increasing the mobile phase linear velocity than increasing the pressure. At 130°C and 130 atm, linear velocities of 1.5 and 4.2 cm $s^{-1}$ were obtained for 3 μm P-ODS packed columns with column lengths of 20 and 10 cm, respectively. The column was shortened by one-half, and the mobile phase linear velocity increased by 2.7 times. This increase remained constant over the range of column inlet pressures evaluated. For columns containing NP-ODS particles, shortening the column length increased the mobile phase linear velocity by approximately the same factor at a specific column inlet pressure. For example, at 130°C and 120 atm, when the column length was shortened from 57 cm to 30 cm, ca. 0.5 times, the mobile phase linear velocity was increased from 1.3 to 2.6 cm $s^{-1}$, or ca. 2.0 times. This means that shortening the column length has a more significant effect on increasing the mobile phase linear velocity for columns containing porous particles.

### 3.3. Retention factor

As discussed in Section 3.2, changes in the mobile phase linear velocity can be obtained by changing the column inlet pressure. Different length columns require different column inlet pressures to obtain specific mobile phase linear velocities. However, the solvating power of the mobile phase in SGC is affected by the pressure or density of the mobile phase, which affects the retention factor. This means that at the same mobile phase linear velocity, different length columns provide different retention factors for the same solutes.

Fig. 3 shows the relationship between retention factor and mobile phase linear velocity for columns containing NP-ODS particles. For specific conditions, two or three measurements were made, and the relative deviations were always less than 5%. Increasing the mobile phase linear velocity by increasing the column inlet pressure decreases the retention factors for all columns. Mobile phase linear velocity programming in SGC has a similar effect on retention as temperature programming in GC. At a specific mobile phase linear velocity, longer columns produced smaller retention factors, because a higher column inlet pressure is needed to obtain the specific mobile phase linear velocity for the longer column. When using the same length column, the column

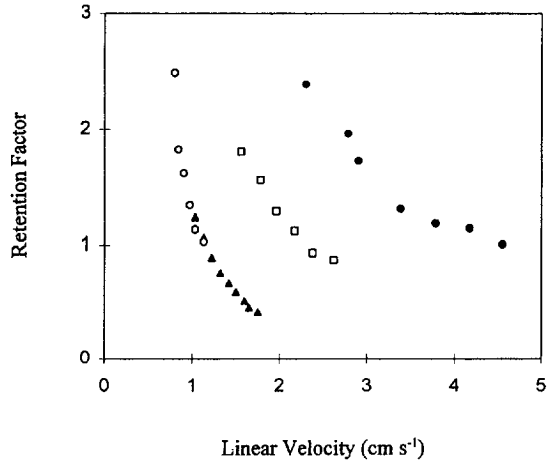


Fig. 3. Relationship between retention factor and mobile phase linear velocity for columns packed with nonporous particles in SGC. Conditions: as in Fig. 2; retention factor was calculated from $(t_r - t_0)/t_0$, dodecane test solute.

containing 1.5 μm NP-ODS particles produced a smaller retention factor than one containing 3 μm NP-ODS particles. Although 1.5 μm NP-ODS particles have a larger specific surface area and should provide a larger retention factor, the high column inlet pressure required for the 1.5 μm NP-ODS packed column offset this and became the dominant factor affecting retention.

Fig. 4 illustrates the relationship between the retention factor and mobile phase linear velocity for columns packed with 3 μm P-ODS particles. It was found that shortening the column or decreasing the mobile phase linear velocity increased the retention factor. Columns containing P-ODS particles provided much larger retention factors than columns packed with NP-ODS particles. For example, for a mobile phase linear velocity of 2.3 cm $s^{-1}$, the 20 cm long column packed with 3 μm NP-ODS particles produced a retention factor of 2.1, corresponding to a column inlet pressure of 70 atm; for the same mobile phase linear velocity and the same column length containing the same size P-ODS particles, a retention factor of 28.7 was obtained, corresponding to a column inlet pressure of 190 atm. Although higher column inlet pressure was used for the column containing P-ODS particles, it also provided a larger retention factor. This was because the porous particles had a much larger specific surface area than the nonporous particles.

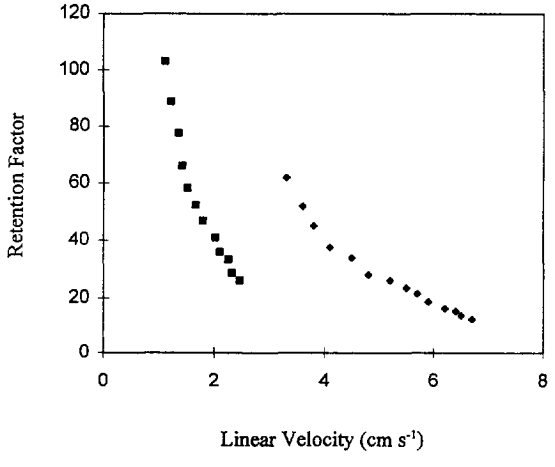


Fig. 4. Relationship between retention factor and mobile phase linear velocity for columns packed with porous particles in SGC. Conditions: as in Fig. 2, dodecane test solute.

### 3.4. Column efficiency

As a result of the use of a highly compressible and solvating mobile phase in SGC, the large difference in column inlet and outlet pressures results in a significant mobile phase linear velocity gradient and retention factor gradient. A theoretical analysis of the effect of the mobile phase linear velocity gradient on column efficiency showed that the maximum loss in efficiency produced by this gradient was 12.5% [1,2]. The effect of the retention factor gradient on column efficiency was studied in SFC [15–17], and it was found that the solubilities of the solutes in the mobile phase are very important. In SGC, the solubilities of the solutes in the mobile phase can be enhanced by elevating the temperature. The solutes remain vaporized in the column, and their elution is not dependent on their solubilities in the mobile phase. Of course, the solvating power of the mobile phase also has an effect on the retention factor.

In our experiments, the theoretical column efficiency was not obtained. A minimum reduced plate height of 4.1 was obtained for columns packed with 3 μm particles. These values are higher than the theoretical values of 2–3. This results from limitations imposed by the experimental conditions, including dead volume and a larger than appropriate recorder time constant. Fig. 5 shows the experimen-

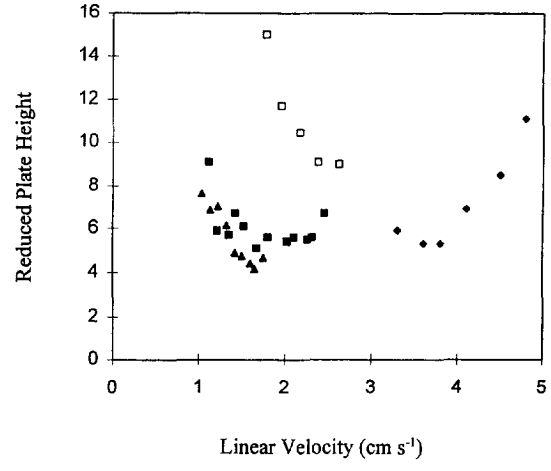


Fig. 5. Relationship between reduced plate height and mobile phase linear velocity in SGC. Conditions: octane test solute; other conditions and symbols as in Fig. 2; reduced plate height was calculated from $H/d_p$ ($H$ is the plate height).

tal relationship between the reduced plate height and mobile phase linear velocity for columns packed with 3 μm P- or NP-ODS particles. Except for the 10 cm long column packed with 3 μm P-ODS, all columns produced lower reduced plate heights when increasing the mobile phase linear velocity. When the column length was shortened, the optimum mobile phase linear velocity shifted from low to high values. Cutting off the column from 57 to 20 cm significantly increased the reduced plate height for the column containing 3 μm NP-ODS particles. It is noteworthy that the curves in Fig. 5 are different from common van Deemter plots in GC because different points on the curve represent different retention factors. Column efficiency in SGC is difficult to determine theoretically because many variables affect the efficiency.

For high speed chromatographic separations, the column efficiency per unit time is of greater interest. Fig. 6 shows the relationship between plate number per second and mobile phase linear velocity. Increasing the mobile phase linear velocity increased the plate number per second for all columns. It was found that by shortening the column length from 57 to 20 cm decreased the maximum (i.e., at the optimum mobile phase velocity) plate number per second from 1200 to 840 to 680 plates $s^{-1}$ when using 3 μm NP-ODS particles as packing material. The achievement of the high plate numbers per second for short columns was limited by the experimental conditions. The 20 cm long column packed with 3 μm NP-ODS particles produced a peak width at half height of 0.25 s when the maximum plate number per second (680 plates $s^{-1}$) was obtained at 100 atm column inlet pressure. The response time constant of the system was 0.25 s. When the column inlet pressure was increased, the peak width at half height remained at 0.25 s, and the measured column efficiency decreased.

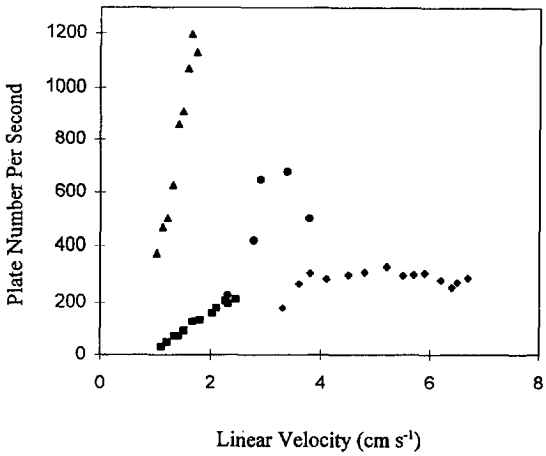


Fig. 6. Relationship between plate number per second and mobile phase linear velocity in SGC. Conditions: octane test solute; other conditions as in Fig. 2; $N_t$ was calculated from $N/t_R$.

Columns packed with P-ODS particles produced a lower plate number per second than columns packed with the same size NP-ODS particles. The highest values of 219 and 339 plates $s^{-1}$ were obtained for 20 and 10 cm long columns, respectively. This low plate number per second probably resulted from the slow diffusion of the solute from inside the pores of the particles.

### 3.5. Resolution

As discussed in Section 3.4, columns containing P-ODS particles provided a larger retention factor which helps to obtain high resolution. However, the better column permeability of NP-ODS particle packed columns allows the use of relatively long columns to obtain high total column efficiencies, which also favors high resolution. Increasing the mobile phase linear velocity can increase the column efficiency; however, it also decreases the retention factor. The effect of mobile phase linear velocity on resolution is dependent on which factor is dominant in its contribution to resolution. Fig. 7 shows the relationship between resolution and mobile phase linear velocity for various columns. The 20 cm long column packed with 3 μm P-ODS particles produced the highest resolution, and the resolution increased with increasing mobile phase linear velocity. Other columns showed a decrease in resolution when the mobile phase linear velocity was increased. Porous particle packed columns provided greater resolution than columns packed with nonporous particles. For example, at a linear velocity of 2.3 cm $s^{-1}$, resolution values of 4.0 and 9.6 were obtained on 20 cm long columns packed with 3 μm NP-ODS and P-ODS particles, respectively. Shortening the column length decreased the resolution.

For high speed chromatographic separations, a

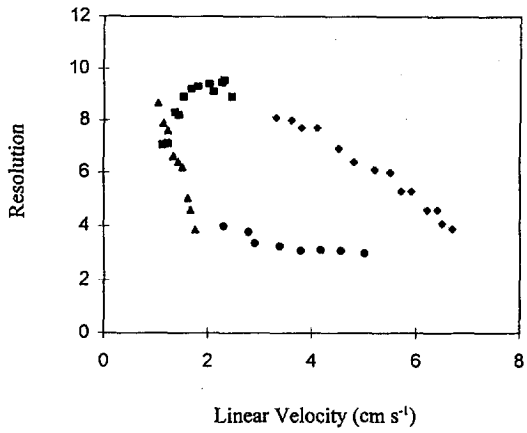


Fig. 7. Relationship between resolution and mobile phase linear velocity in SGC. Conditions: undecane and dodecane test solutes; other conditions as in Fig. 2; resolution ($R$) was calculated from $(t_{R2}-t_{R1})/[(W_{b1})_{1/2}+(W_{b2})_{1/2}]$, where $(W_{b1})_{1/2}$ is the peak width at half height.

minimum resolution is required. Fig. 8 shows the effects of column length and particle type on the resolution per minute at various mobile phase linear velocities. From this figure, it can be seen that the resolution per minute increased with an increase in mobile phase linear velocity for all columns; the resolution per minute increased by shortening the column length; and the columns containing NP-ODS particles produced a much higher resolution per minute than the column packed with P-ODS particles. Therefore, short columns (i.e., 10 cm) packed with porous particles, and relatively long columns (i.e., 20 cm) packed with nonporous particles are recommended for carrying out high speed SGC separations.

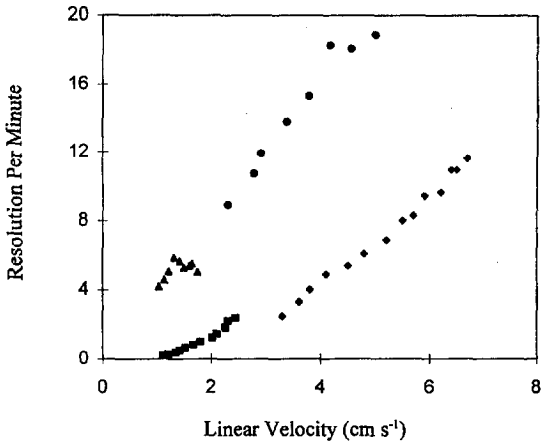


Fig. 8. Relationship between resolution per minute and mobile phase linear velocity in SGC. Conditions: undecane and dodecane test solutes; other conditions and symbols as in Fig. 2; resolution per minute was calculated from $R/t_{R2}$.

Several chromatograms illustrate the relationship between column efficiency, resolution and analysis time. Fig. 9 shows three chromatograms obtained by using 20 and 57 cm long columns packed with 3 μm NP-ODS particles and an 8.5 cm long column packed with 1.5 μm NP-ODS particles. Although the long column produced a high total column efficiency, a decrease in column length could provide approximately the same resolution in shorter time and provide a savings in analysis time. This probably resulted from the observation that the short column produced a slightly larger retention factor. Even a short 10 cm column packed with 3 μm NP-ODS particles was investigated, and the first two peaks were not baseline separated. When using 1.5 μm NP-ODS particles as packing material, less than 10 cm could be used for high speed separations, as illustrated in Fig. 9C.

Fig. 10 shows four chromatograms obtained by using various length columns packed with 3 μm P-ODS particles. For a 20 cm long column, high column efficiency and excellent resolution were obtained by using a 200 atm column inlet pressure. However, it is difficult to carry out high speed separations using this column and increasing the column inlet pressure. When this column length was cut to 10 cm, similar resolution was obtained in the same analysis time; however, only 94 atm column inlet pressure was needed, as illustrated in Fig. 10B. The low column inlet pressure allows this column to be used for high speed analysis. Using 200 atm column inlet pressure, the separation was finished within 30 s, as illustrated in Fig. 10C. Of course, the resolution decreased. In order to obtain a similar resolution and analysis time to those in Fig. 9B and C, a column inlet pressure of 250 atm was needed, as illustrated in Fig. 10D.

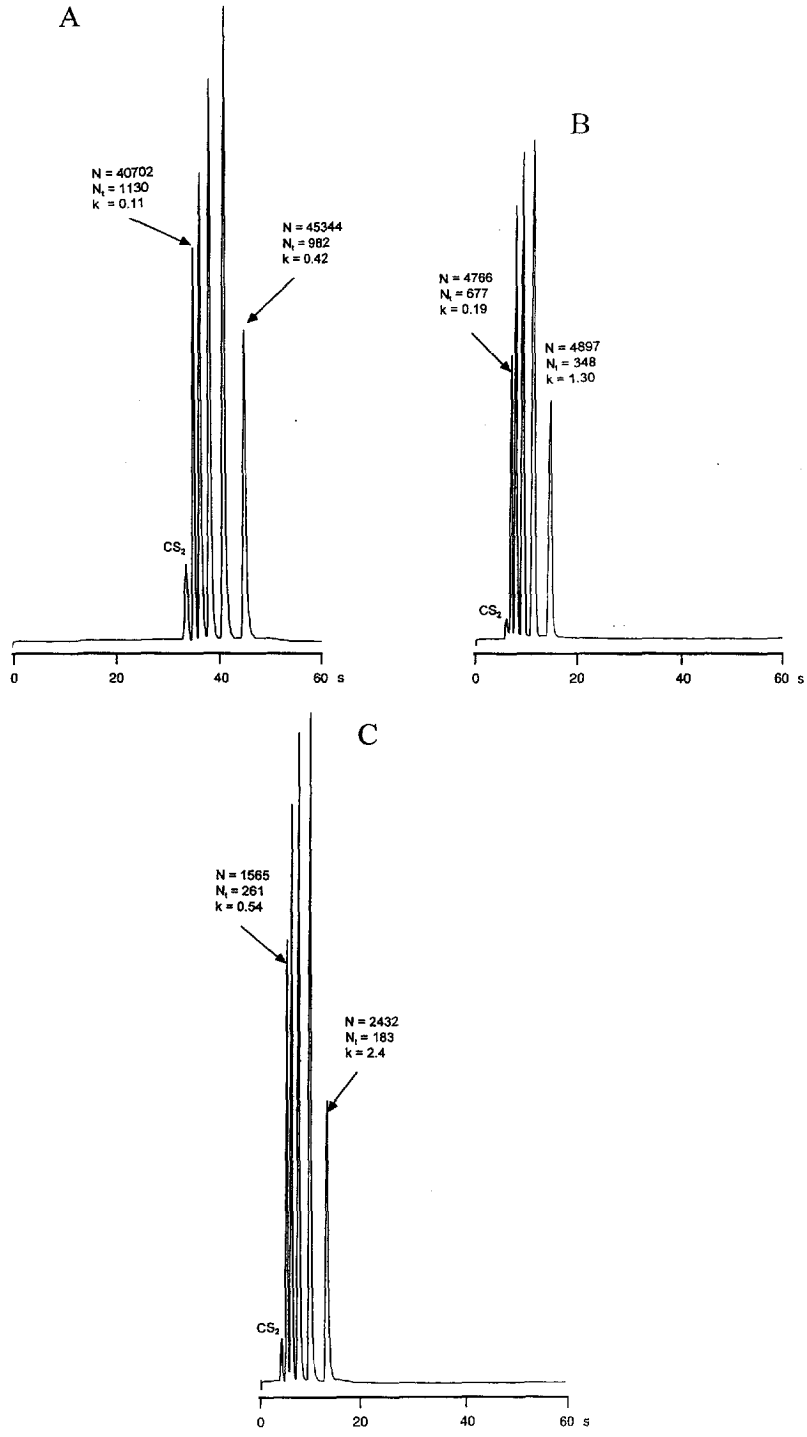


Fig. 9. Effect of column length on high speed SGC using nonporous particles. Conditions: $CO_2$; 130°C; FID; *n*-hydrocarbons from octane to dodecane used as test solutes; (A) 57 cm×250 μm I.D. capillary packed with 3 μm NP-ODS particles using 170 atm column inlet pressure, (B) 20 cm×250 μm I.D. capillary packed with 3 μm NP-ODS particles using 95 atm column inlet pressure, and (C) 8.5 cm×250 μm I.D. capillary packed with 1.5 μm NP-ODS particles using 100 atm column inlet pressure.

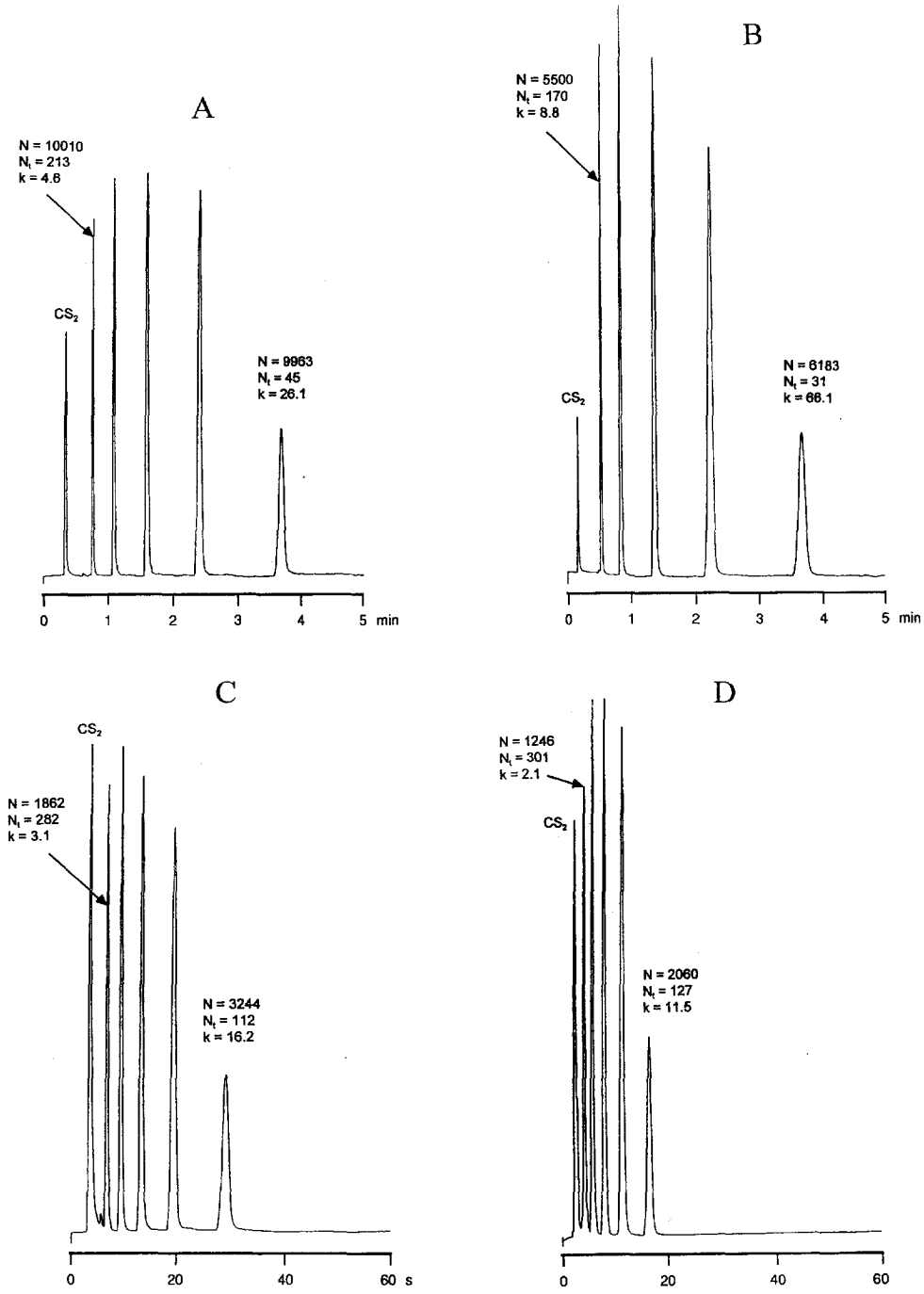


Fig. 10. Effect of column length on high speed SGC using porous particles. Conditions: (A) 20 cm×250 μm I.D. capillary packed with 3 μm P-ODS particles using 200 atm column inlet pressure; (B, C and D) 10 cm×250 μm I.D. capillary packed with 3 μm P-ODS particles using column inlet pressures of 94, 200 and 250 atm, respectively. Other conditions as in Fig. 9.

### 3.6. Effect of the activity of the ODS bonded particle surface on separations of polar compounds under SGC conditions

Because of the steric hindrance of octadecyl groups, it is impossible to eliminate all silanol groups on the particle surface by the bonding reactions. The residual silanol groups lead to adsorption of polar solutes under SGC conditions. Various polar compounds were analyzed using SGC conditions in this study.

For polarizable aromatic compounds, symmetrical peaks were obtained, and P-ODS particles were found to be more suitable for the separation of these compounds. Fig. 11 shows a high speed SGC separation of aromatic compounds at constant temperature and mobile phase linear velocity.

Ketones, esters and aldehydes were eluted from the column with symmetrical peaks. However, polar compounds, such as anilines and hydroxyl containing compounds, produced serious peak tailing, although elevated temperature (200°C) was used. The particles were end-capped using HMDS as previously described [25], and improved separations were obtained. However, there was still some peak tailing as illustrated in Figs. 12 and 13. Strongly polar compounds such as free carboxylic acids and amines were not eluted from the column. Therefore, inert packing materials are required for successful high speed SGC separations of polar compounds.

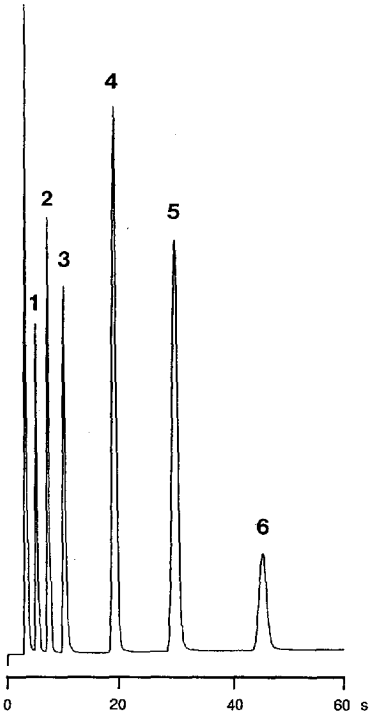


Fig. 11. High speed SGC of aromatic compounds. Conditions: 10 cm×250 μm I.D. capillary packed with 3 μm P-ODS particles; 120 atm column inlet pressure; 180°C. Other conditions as in Fig. 9. Peak identifications: (1) benzene, (2) toluene, (3) *p*-xylene, (4) butylbenzene, (5) naphthalene and (6) 1-methylnaphthalene.

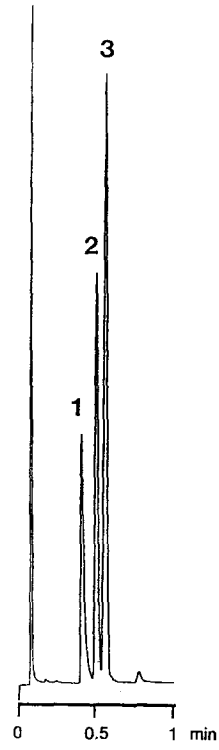


Fig. 12. High speed SGC of anilines. Conditions: 10 cm×250 μm I.D. capillary packed with 3 μm HMDS end-capped P-ODS particles; 170°C; 100 atm column inlet pressure. Other conditions as in Fig. 9. Peak identifications: (1) aniline, (2) N-methylaniline and (3) N,N-dimethylaniline.

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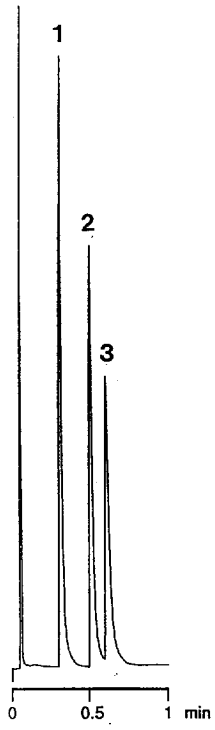


Fig. 13. High speed SGC of hydroxyl containing compounds. Conditions: 140°C; 110 atm column inlet pressure. Other conditions as in Fig. 9. Peak identifications: (1) phenol, (2) α-phenylalcohol and (3) 1-octanol.

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