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Journal of Chromatography A, 883 (2000) 55–66

JOURNAL OF
CHROMATOGRAPHY A

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Fullerenes separation with monomeric type C₃₀ stationary phase in high-performance liquid chromatography

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Received 28 January 2000; received in revised form 16 March 2000; accepted 17 March 2000

Abstract

The temperature effect on the separation of fullerenes in LC was examined using monomeric type C₃₀, C₁₈ and C₈ alkyl bonded stationary phases. It appears that the C₃₀ phase exhibits superior separation ability for fullerenes. It is observed that the maximum retention temperature of fullerenes on the C₃₀ phase is around 20°C. A strong correlation between the changes in NMR spectra and the retention behavior of the solutes was found. The interpretation of the retention behavior of fullerenes on the alkyl bonded stationary phases, including the behavior in subambient temperature, is discussed using the information obtained by CP-MAS solid-state NMR spectroscopy and LC. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Stationary phases, LC; Temperature effects; Solid-state NMR spectroscopy; Nuclear magnetic resonance spectroscopy; Fullerenes

1. Introduction

Since the existence of fullerenes was confirmed [1–3], various chromatographic separation techniques have been required to separate and purify these molecules [4–33]. Liquid chromatography (LC) is probably the best method one can choose for this application. The number of LC stationary phases for this purpose has been increased tremendously over the past few years. Although many novel stationary phases have been used [20–33], the popular C₁₈ alkyl chain bonded stationary phase ‘ODS’ can be generally useful for this purpose [11–19].

Two main reasons why ODS is popular as the primary stationary phase are that it is easily commercially available and optimization of the separation is often easier than on novel stationary phases. Nevertheless the retention mechanism in the chromatographic process for compounds of which structures are composed by six-membered phenyl rings has not been clearly interpreted yet. A lot of scientists have been trying to elucidate the mechanism. Wise and Sander have examined this topic intensively [34–44]. They suggested that molecular shape recognition for polycyclic aromatic hydrocarbons (PAHs) is one important source of information on the retention mechanism. As the ODS phases can be divided into two different types, monomeric and polymeric, on the basis of the bonding chemistry used for their

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preparation, the monomeric phase has isolated single C_{18} chains but for the polymeric phase the possibility of networked chains exists. Wise and Sander have found that the polymeric ODS has a stronger tendency to recognize the molecular planarity of PAHs than with the monomeric phase, and then concluded that the structure of the polymeric ODS is more rigid and slit-like (they called 'slot-model' [34–36]) than that of the monomeric ODS. They also found that a number of factors (stationary phase density, chain lengths, column temperatures, etc.) influence column selectivity for PAH separations [34]. In addition to such basic studies on alkyl bonded phases, the relationship between the bonded phase structure and the retention behavior of the solutes using polymeric C_{30} phases have also been investigated by Albert's group [45–50] and by the authors [32]. In the previous studies of fullerene separation with the C_{18} phase [17,18,32,51,52], we have found that bonded phase association and the configuration change with temperature are important factors controlling the retention of fullerenes in LC. The stationary phase needs to have enough depth and space to catch and hold the bulky molecules within the ligand space. Thus it is reasonable to assume that isolated single and long chains stationary phase may interact more effectively with bulky fullerenes. In this study, therefore, we investigated fullerene separation, using a monomeric type C_{30} alkyl chain bonded stationary phase as the first time. Since the demands of the preparative and large scale separation of fullerenes, we need to use a high toluene concentration in the mobile phase. The C_{30} phase can realize such separations. The effects of the alkyl chain length of the bonded stationary phases and column temperature (especially in subambient temperature) on the retention of fullerenes were examined by liquid chromatographic and NMR spectroscopic studies.

2. Experimental

Develosil C30-UG-5 (5 μm , 18.2 C%, Nomura Chemical, Seto, Japan), Develosil ODS-UG-5 (17.6 C%) and Develosil C8-UG-5 (11.0 C%) were used as the monomeric type packing materials in 4.6 mm I.D. \times 250 mm length column. The mobile phases

were a toluene/acetonitrile mixture and *n*-hexane (obtained from Tokyo Kasei, Tokyo, Japan) with a typical flow-rate of 1 ml min^{-1} . C_{60} and C_{70} fullerenes were isolated from carbon soot produced by the process described in our previous publications [13–16]. Polycyclic aromatic hydrocarbons (PAHs), chrysene, triphenylene and pyrene (obtained from Wako Chemicals, Osaka, Japan) were used as the reference. The LC system consisted of a Jasco PU-980 pump (Tokyo, Japan), a Hewlett-Packard 1040 A photodiode array detector (Yokogawa Analytical Systems, Musashino, Japan) controlled by an HP 9000 computer, and a Rheodyne 7125 injector (Cotati, CA, USA). Column temperatures between 30 and 80°C were controlled by means of a Tosoh RE-8000 oven (Tokyo, Japan) and between 0 and 30°C by a Lab-Thermo Model LH-1000E system (Toyo Seisakusho, Tokyo, Japan); temperatures below 0°C were maintained with a mixture of methanol and dry ice using a home-made controller.

Solid state NMR spectra with Variable-Amplitude Cross-Polarization Magic Angle Spinning (VAC-PMAS) [53] and Two Pulse Phase-Modulation (TPPM) decoupling [54] were obtained on a Varian Unity INOVA 400 spectrometer (Palo Alto, CA, USA) with a variable temperature unit. The spinning rate was between 4000 and 5000 Hz depending on the temperature. A contact time of 5 ms and a repetition rate of 6 s were used for all spectral measurements.

3. Results and discussion

For the investigation of monomeric type C_{30} alkyl chain bonded stationary phase, the first task is to find the optimum mobile phase composition. As described in our previous publications [13–19], reasonable solubility of fullerenes in toluene suggests its use in the mobile phase for fullerenes separation, therefore, toluene–acetonitrile mixtures were evaluated as a possible counter. In Fig. 1, the separation of higher fullerenes using the monomeric type C_{30} stationary phase with various toluene/acetonitrile mobile-phases at 10% intervals are illustrated. As shown in this Figure the separation of C_{60} and C_{70} was achieved at 90% toluene concentration. The separation of higher fullerenes was possible at 80%

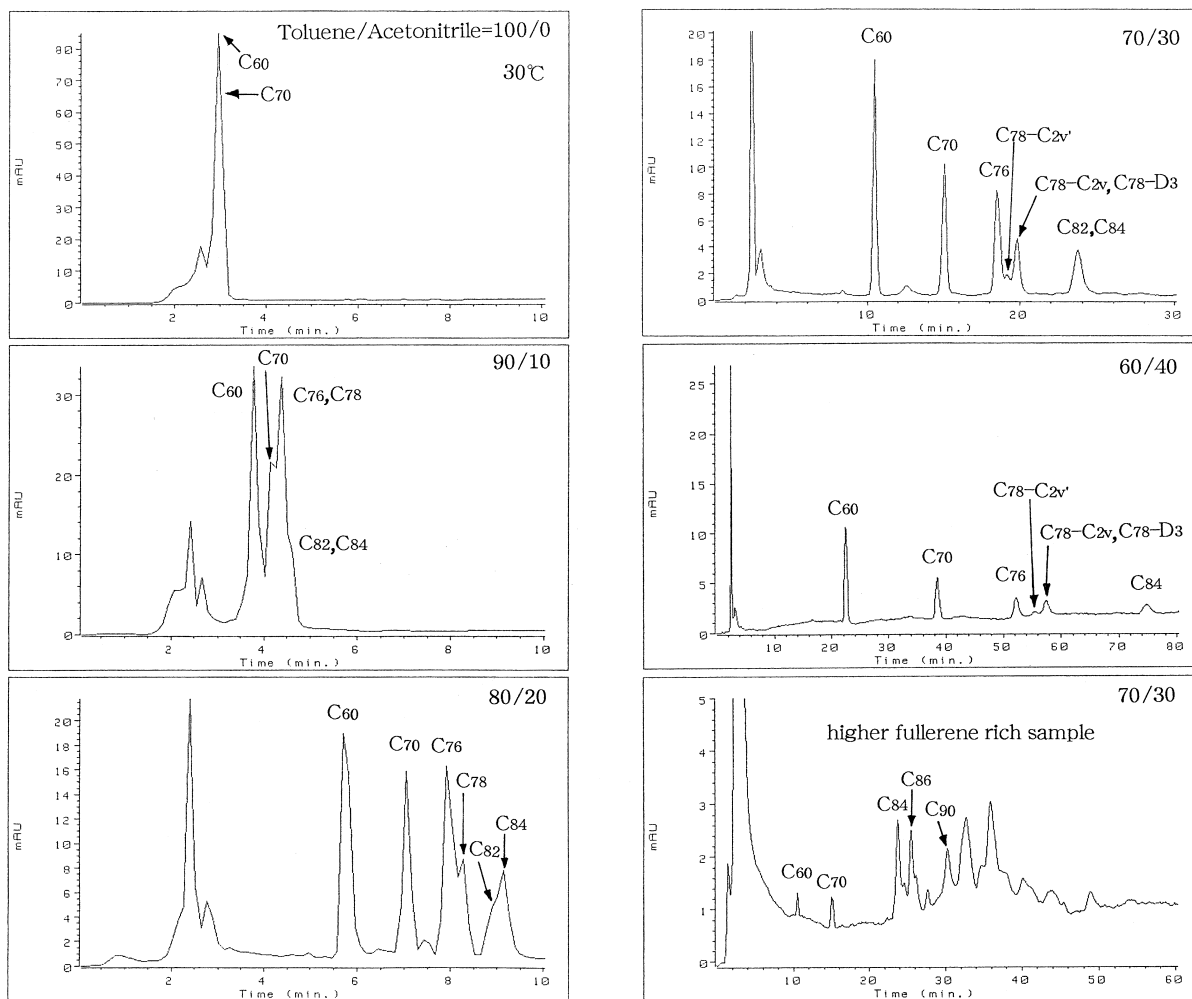


Fig. 1. Separations of higher fullerenes on the monomeric C_{30} phase (Develosil C30-UG-5) with various toluene concentration mobile phases. Column temperature: 30°C; flow rate: 1 ml/min; mobile phase: toluene/acetonitrile mixture; detection: UV at 312 nm.

toluene concentration. Focusing on the separation of the three C_{78} isomers, one of these isomers was separated at 70% toluene concentration. It was also observed that fullerenes larger than C_{86} began to be separated with the 70% toluene concentration. These results show that better separation of fullerenes can be achieved by the C_{30} phase even though a higher toluene concentration in the mobile phase was used. For the comparative study of the monomeric type C_{30} phases to other alkyl bonded phases such as monomeric C_{18} and C_8 , several chromatograms with higher toluene concentration mobile phase (toluene/acetoneitrile=80/20) were measured and the results

are as shown in Fig. 2. Focusing on the elution profiles of the fullerenes, the C_8 phase was not able to separate fullerenes at each temperature. For the C_{18} phase, although better separation of fullerene isomers was achieved at 0°C, increasing the temperature decreased the resolution and then the phase could not separate fullerenes at 60°C. In comparison to these phases, it appears that the C_{30} bonded phase exhibited superior separation ability for fullerenes, especially at a higher temperature around 60°C. The differences for three phases are clearly seen in Fig. 3. In this figure obtained at 60% toluene concentration, the superior separation of fullerenes by the C_{30} phase

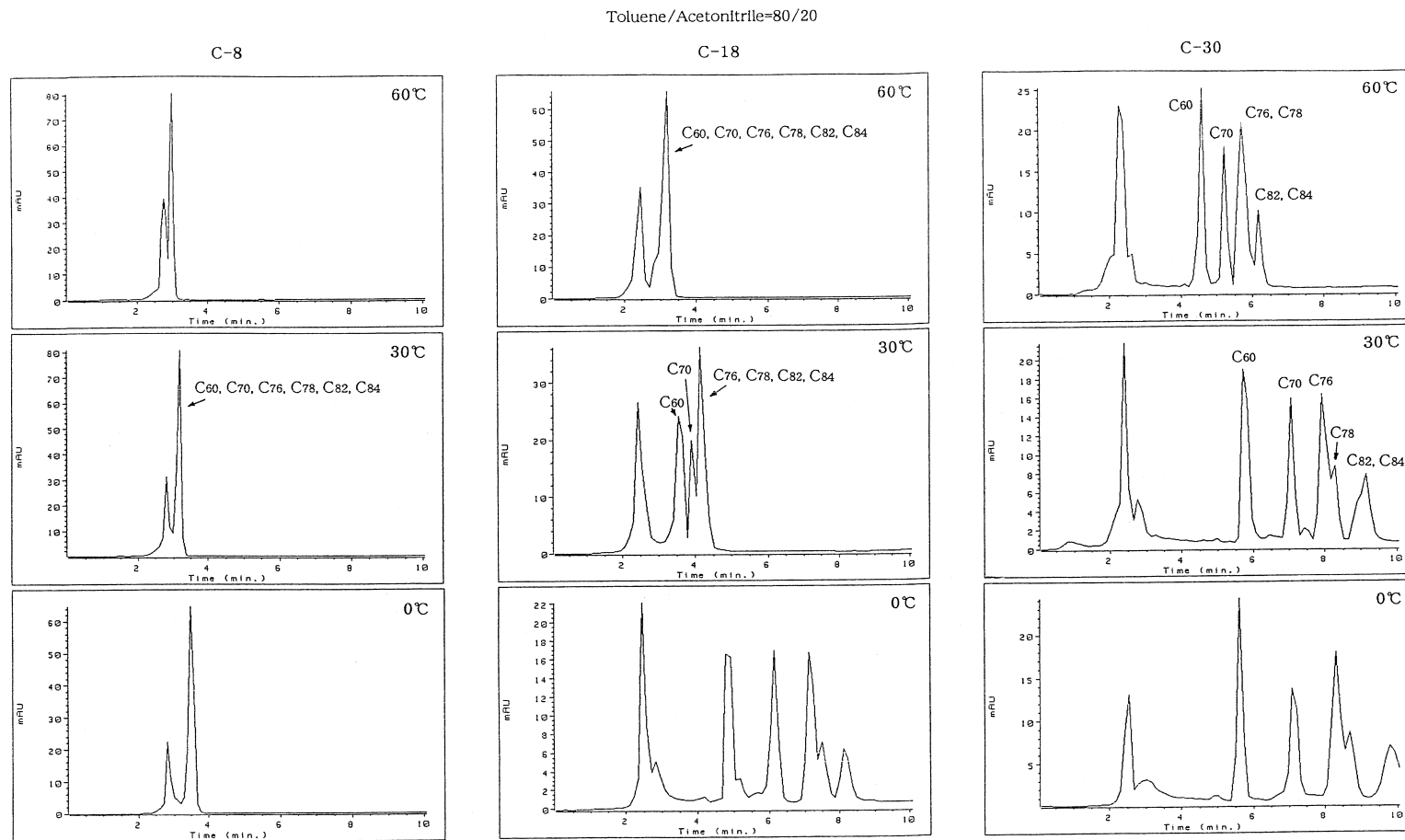


Fig. 2. Chromatograms for the separation of higher fullerenes at various temperatures with the monomeric type C₃₀ (Develosil C30-UG-5), C₁₈ (Develosil ODS-UG-5) and C₈ (Develosil C8-UG-5) stationary phases. Mobile phase: toluene/acetonitrile=80/20. Column temperature: 60°C, 30°C and 0°C. Flow rate: 1 ml/min. Detection: UV at 312 nm.

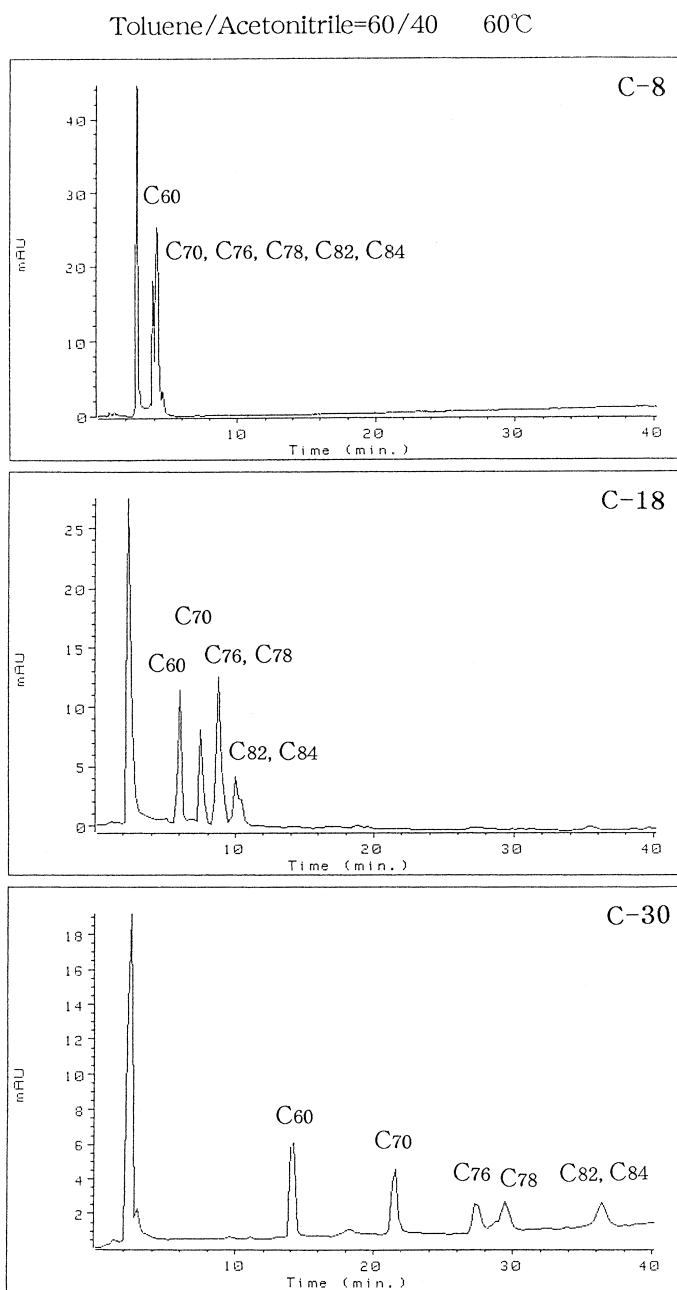


Fig. 3. Chromatograms for the separation of higher fullerenes with the monomeric type C_{30} , C_{18} and C_8 stationary phases. Mobile phase: toluene/acetonitrile=60/40. Column temperature: 60°C. Flow rate: 1 ml/min. Detection: UV at 312 nm.

is demonstrated. In these figures, the difference between the C_{30} and other two phases for fullerenes retention is remarkable at higher temperature. These observations indicate that the length of alkyl chains

in the bonded phase largely contributes to fullerene retention, with longer being better. Considering the solubility of fullerenes in toluene based mobile phase, it is reasonable to conclude that the mono-

meric C_{30} phase is the best choice for the fullerene separation with the available alkyl chain bonded silica stationary phases.

The detailed temperature effect on the separation of higher fullerenes for the monomeric C_{30} phase has been investigated and the results are summarized in Fig. 4. In this figure, it is apparent that there is a curvature in the plots of k vs. temperature at ca. 20°C . Namely, it was observed that this phase gave better separation of fullerenes with decreasing column temperature until a maximum in retention was achieved at temperature of about 20°C , then the retention of these solutes decreased with a further reduction of the column temperature. To explain the specific phenomenon, the retention of globular fullerenes and two-dimensional PAHs was investigated. Chromatograms for the separation of the mixture of fullerenes and PAHs are illustrated in Fig. 5. A different behavior between the retention of PAHs and

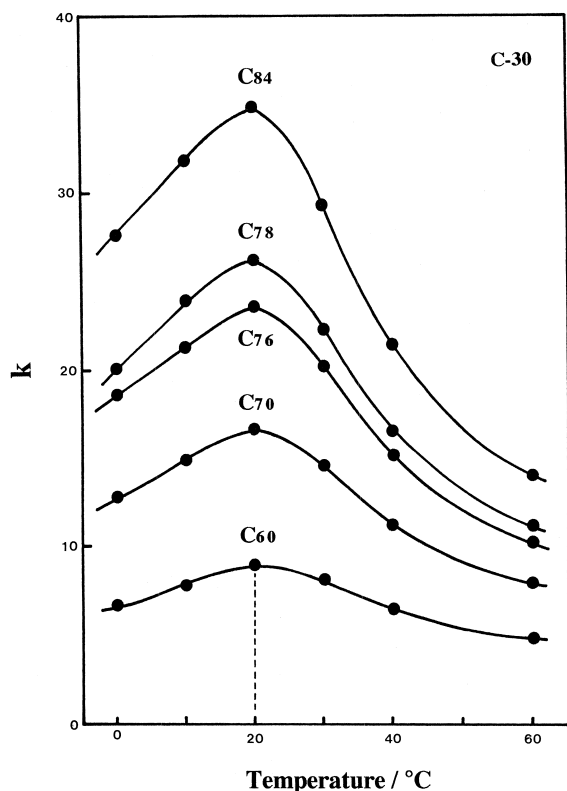


Fig. 4. Relationships between retention factors of higher fullerenes with the C_{30} alkyl bonded phase (Develosil C30-UG-5) versus column temperature.

fullerenes is apparently found in this figure. The behavior is much more clearly seen in the relationships between the logarithm of retention factor and the reciprocal of absolute temperature ($\ln k$ vs. $1/T$, van't Hoff plot) shown in Figs. 6 and 7. In Fig. 6 for the C_{30} phase, there was a monotonic decrease in retention for both of the fullerenes and the PAHs with increasing temperature above 20°C . The temperature dependency for the retention of fullerenes using the C_{30} phase is very minimal above 20°C . But below 20°C , a difference between the retention behavior of PAHs and fullerenes is dramatically seen. The retention of PAHs increased with decreasing temperature, on the other hand, the retention of C_{60} and C_{70} decreases drastically just below 20°C .

For a comparison between the C_{30} and the C_{18} phases, the plots with the C_{18} phase are given in Fig. 7. It is also found that there is a maximum in the plots for fullerenes around -20°C . It is also shown that increasing the temperature magnifies the difference of the retention of fullerenes between the C_{30} and the C_{18} phases, though so significant a difference could not be found for the retention of PAHs. The major difference between the C_{30} and the C_{18} phases was the maximum retention temperature for fullerenes, the former at 20°C and the latter at -20°C .

In LC with ODS phases, in general, solute retention is inversely related to temperature, i.e., retention decreases with increasing temperature. The existence of phase transitions in the bonded phase and the discontinuity in the van't Hoff plot was observed by Morel et al., [55,56]. In Fig. 6, for C_{30} phase, a remarkable inflection point is clearly seen, and the retention of PAHs increased linearly with decreasing temperature but there is a change of slope in the plot at approximately 30°C . An apparent inflection point in the van't Hoff plot for C_{60} and C_{70} was also found around 20°C . The $\ln k$ of C_{60} and C_{70} decreases linearly just below 20°C . For C_{18} phase, in Fig. 7, the critical change of the slope is not seen in the plot for PAHs, but there is a broad curvature in the plots for the C_{60} and C_{70} at ca. -20°C . From these results, it is assumed that the critical phase transition in the bonded phase occurs at around between 20°C and 30°C especially for the C_{30} phase. It was also assumed that a more graduated transition may occur at much lower temperature for the C_{18} phase.

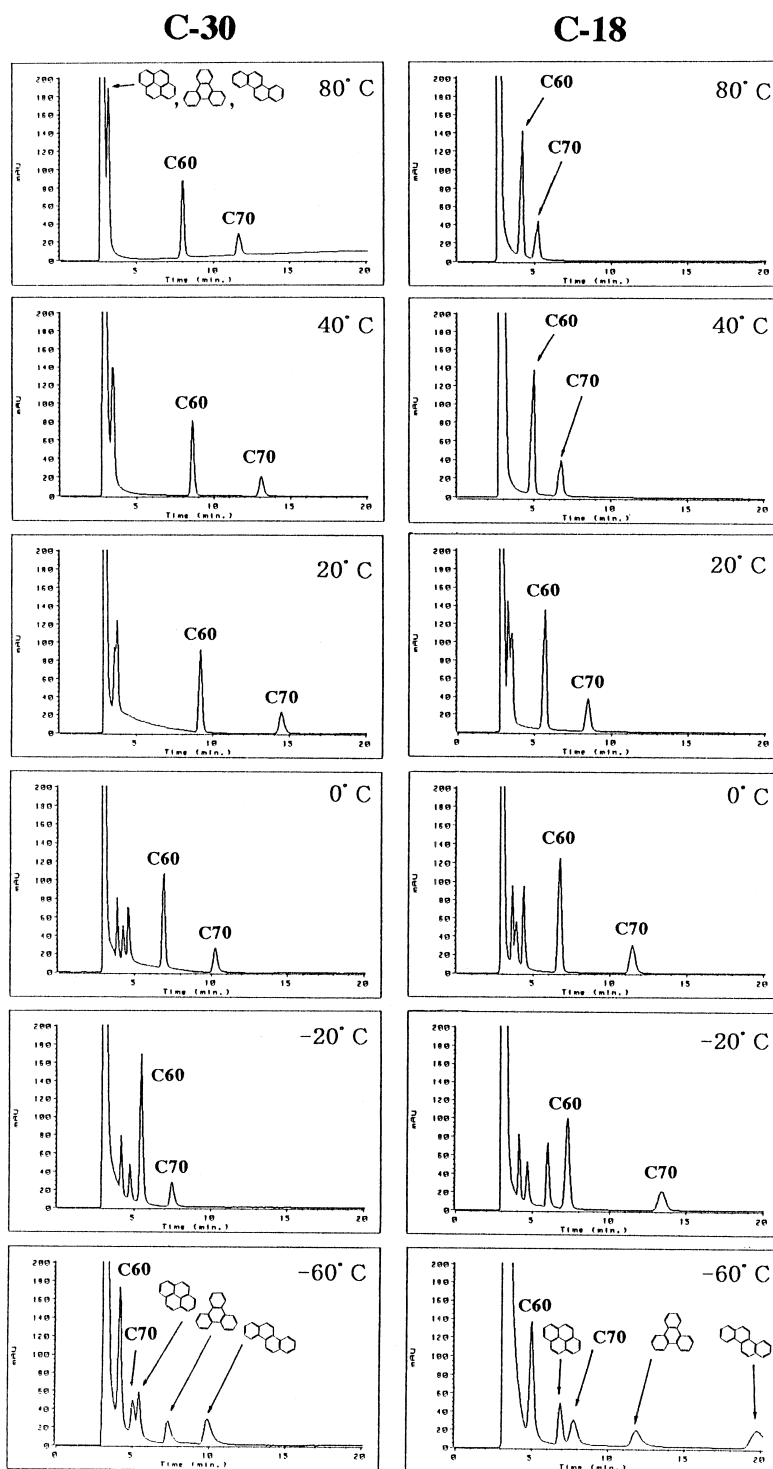


Fig. 5. Chromatograms for the separation of fullerenes (C_{60} and C_{70}) and PAHs (pyrene, triphenylene, chrysene) at various temperatures with the monomeric type C_{30} and the C_{18} stationary phases. Mobile phase: *n*-hexane. Flow rate: 1 ml/min. Detection: UV at 254 nm.

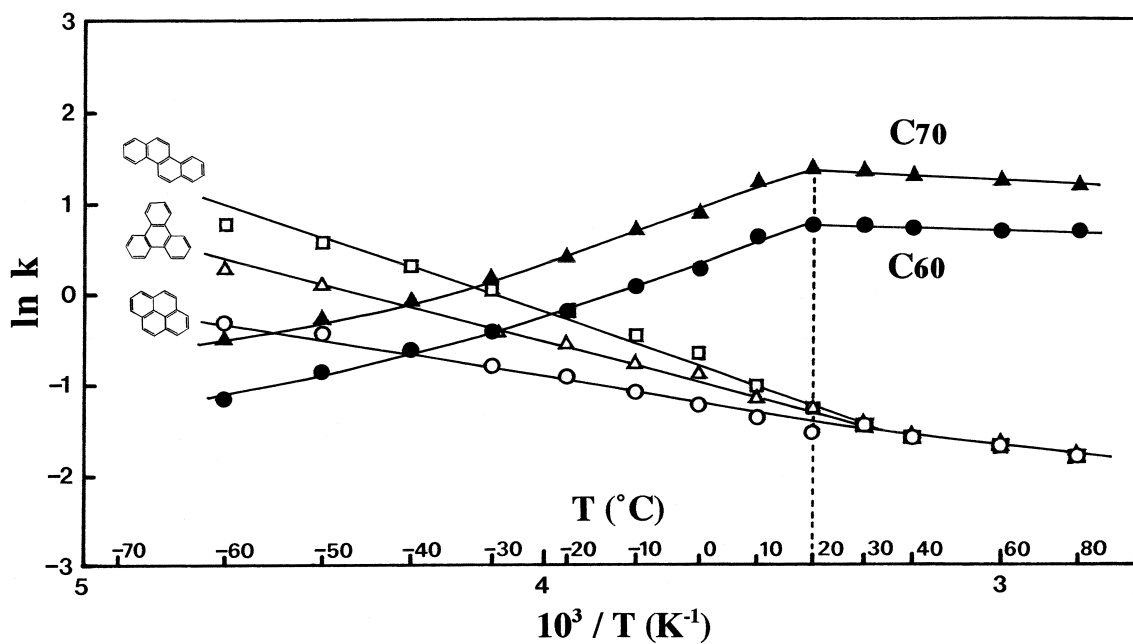


Fig. 6. Plots of $\ln k$ vs. temperature for C_{60} and C_{70} , and three PAHs (pyrene, triphenylene, chrysene) with the C_{30} alkyl bonded phases.

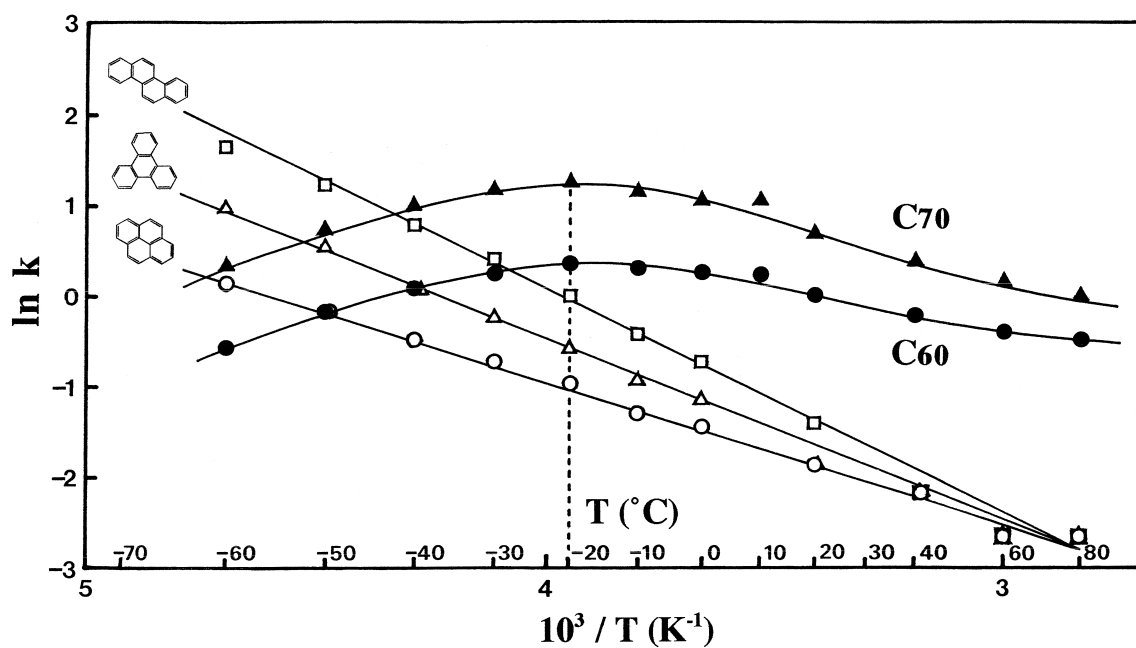


Fig. 7. Plots of $\ln k$ vs. temperature for C_{60} and C_{70} , and three PAHs (pyrene, triphenylene, chrysene) with the C_{18} bonded phases.

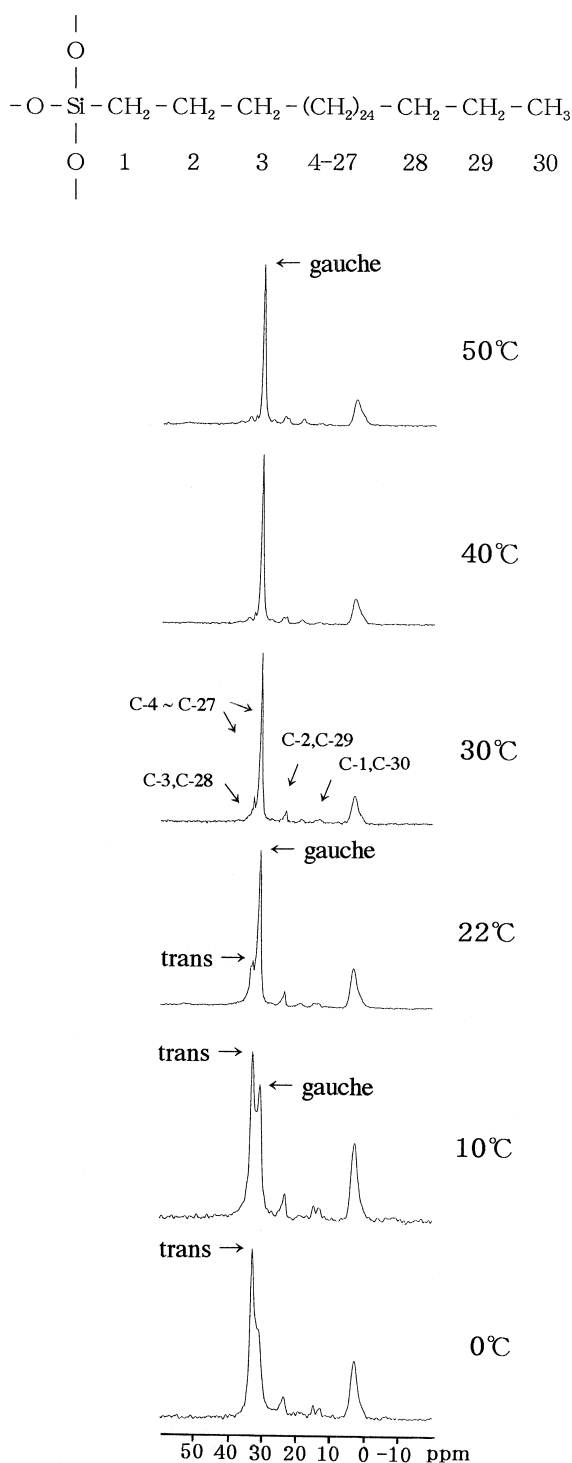


Fig. 8. ^{13}C NMR CP-MAS spectra of the C_{30} bonded phase at various temperatures.

For better understanding of these specific phenomena, solid-state NMR spectroscopy should be a very useful technique because the signal change has been shown to correlate with the chromatographic behavior of fullerenes in LC [18,32]. In the previous studies of fullerenes separation with the C_{18} phase [17,18,32], we have found that bonded phase association and the configuration change with temperature are important factors controlling the retention of fullerenes in LC. Therefore the effect of column temperature on the retention of fullerenes using the monomeric type C_{30} phase was also discussed by using LC and NMR spectroscopic information. Fig. 8 shows ^{13}C NMR CP-MAS spectral changes as a function of temperature for the monomeric type C_{30} phase. A stronger signal splitting of the main CH_2 groups for a polymeric C_{18} column were discovered by Jinno et al. [51,57], and Albert et al. [45–50] and the authors [32] also found the signal splitting for a polymeric C_{30} column. The 30 ppm peak represents a more mobile ‘gauche’ configuration and the 32.8 ppm peak represents a more rigid ‘trans’ structure. It is quite interesting that the stronger signal splitting around 30 ppm was clearly found even for the monomeric type phase. In this Figure it appears that the 30.0 ppm peak is dominant above 30°C , and then below 20°C the 32.8 ppm peak becomes larger. Namely, proportion of the ‘gauche’ configuration increases above 20°C and proportion of the ‘trans’ configuration increases below ca. 20°C – 30°C . It is also very interesting to note that these two NMR signal intensity changes correlate quite well with the inflection point in the van’t Hoff plots for the solutes as seen in Fig. 6. The $\ln k$ of fullerene compounds begins to decrease at about the same temperature that the proportion of the 32.8 ppm peak for ‘trans’ configuration just begins to increase. This correlation could be explained by an increased contact (interaction) occurring between the solute and the bonded moiety as the temperature is decreased, with the ligand having a rigid and ordered ‘trans’ structure that results in the exclusion of fullerene molecules.

Fig. 9 is a schematic for the proposed interactions between the monomeric type C_{30} bonded phase and fullerenes. For plane solutes such as PAHs, it was assumed that the rigidity and orderliness of the ligand below 20°C greatly facilitated the retention of the PAHs. Namely, because of a more polymeric-like

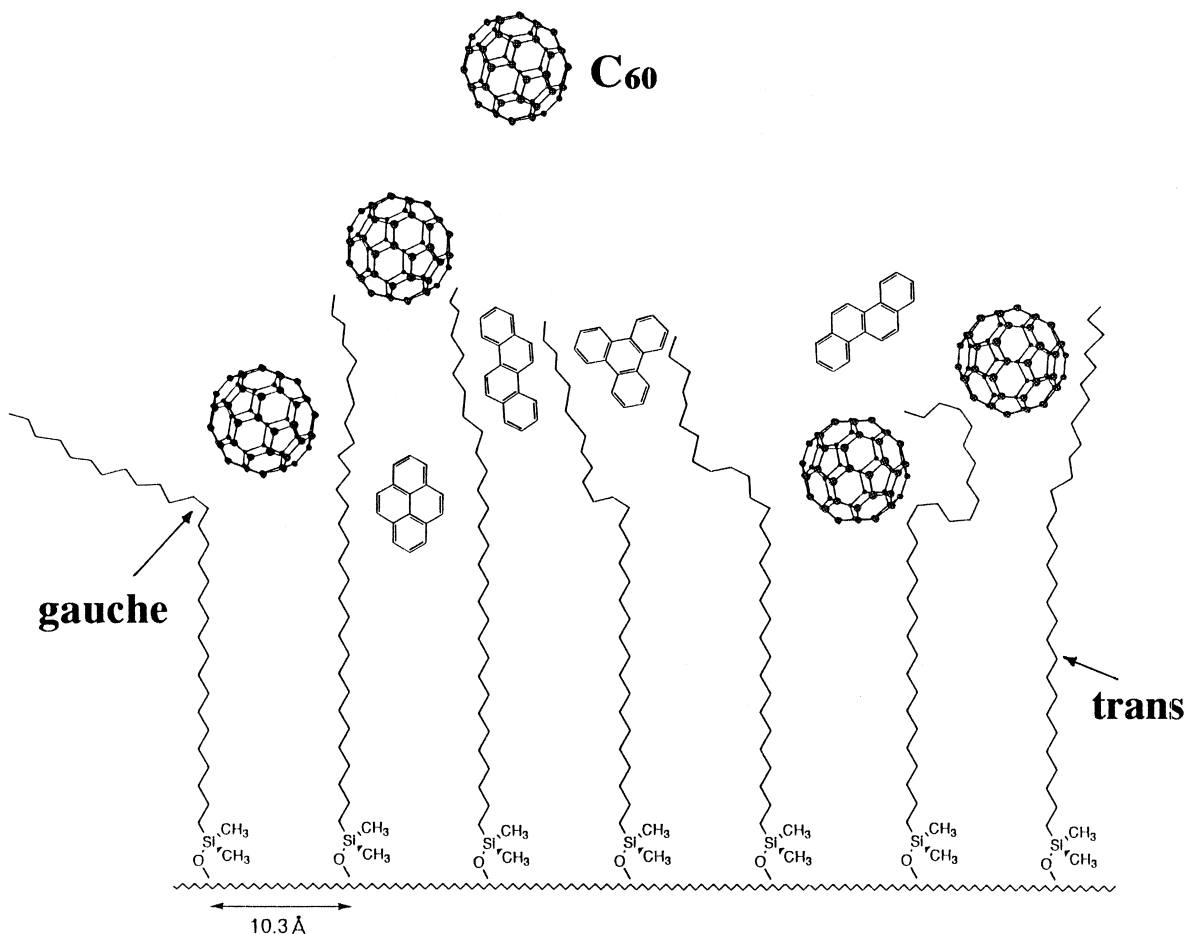


Fig. 9. Schematic for the interaction between the C_{30} bonded phase and fullerenes.

slot structure [34–36] of the bonded moiety at low temperature, the plane PAHs can easily access to the space of the bonded moiety resulting in strong retention of planar PAHs. Therefore, it can be assumed that the accessibility of these solutes to the bonded moiety is an important factor for the retention. From this concept, it is reasonable to assume that a critical chain length exists to interact effectively with fullerenes, with longer being better. The C_8 chains are too short to have an effective interaction with fullerene molecules. The shorter alkyl chains do not have enough depth to catch and hold bulky molecules within the ligand space. In comparison to this phase, C_{30} chains are very effective for interaction with fullerene molecules because of its very long chain so that strong retention of fullerenes is achieved.

Although further consideration about the retention mechanism of fullerenes with monomeric type C_{30} bonded stationary phases is needed, it can be concluded from the above systematic evaluation that the strong correlation between the changes in NMR spectra and retention indicate that the configurational change of the alkyl chains with temperature is one of the most important factors in controlling the retention of solutes in LC.

Acknowledgements

We greatly appreciate the contribution of Mr. J. Ashida of Varian Japan Ltd. for the help to measure the solid state NMR spectra of the bonded phases. A part of this work is financially supported by the

Grant-in-Aid for Scientific Research (Grant No.10915022) from the Ministry of Education and Culture, Japan.

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