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# Separation of aromatic isomers on cyclophane-bonded stationary phases

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**Abstract**

A cyclophane (CP66)-bonded silica gel stationary phase (CP66-SP) was prepared and the retention of water-insoluble hydrophobic compounds on it was investigated in comparison with that on the CP44-bonded stationary phase (CP44-SP) reported previously. Like CP44-SP, it retained aromatic compounds more strongly than the corresponding alicyclic compounds, as was expected by the cavity size of the cyclophane. The CP66-SP also showed isomer-selectivity for monosubstituted and disubstituted naphthalenes, but its selectivity was perfectly reversed to that of the CP44-SP. On the CP66-SP, isomers having methyl and ethyl groups at β-position were eluted prior to those having groups at α-position, whereas on the CP44-SP β-substituted naphthalenes were retained more strongly than α-substituted ones. Isomers of three- and four-ring aromatic compounds were also separated on these cyclophane-bonded stationary phases. The retention order on the CP66-SP was almost opposite to that on the CP44-SP; on the CP66-SP, the retention order was phenanthrene> anthracene, and chrysene>1,2-benzanthracene >2,3-benzanthracene, whereas on the CP44-SP, anthracene>phenanthrene, and 2,3-benzanthracene>chrysene>1,2-benzanthracene. The retention mechanism of aromatic compounds is discussed on the basis of the structure of the cyclophane-involved complex. 

*Keywords:* Stationary phases, LC; Aromatic isomers; Cyclophane

## 1. Introduction

Macrocyclic organic host compounds have been studied extensively as a selector of HPLC stationary phases owing to their highly selective molecular recognition for guest compounds [1–8]. In the preceding paper [9], we reported the preparation of a cyclophane-bonded silica gel stationary phase (CP44-SP) and its application to the separation of isomers of naphthalene derivatives. The prepared CP44-SP showed preferential retention for aromatic compounds and interesting selectivity for isomers of naphthalene derivatives. Especially, some dimethylnaphthalene isomers that cannot be separated on ordinary reversed-phase stationary phases were finely separated on that stationary phase.

The used cyclophane, CP44 (Fig. 1), had a cavity whose rectangular opening was 0.35 nm×0.79 nm [10]. The length of the short side was about the same

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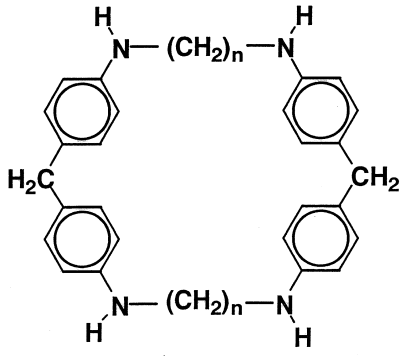



CP44 n = 4
66 = 6

Fig. 1. Structures of cyclophanes used in this study.

as the thickness of an aromatic ring, but the length of the long side was too short for the naphthalene ring to be accommodated with its long axis parallel to the cavity plane. The complex between CP44 and naphthalene had a ''pseudoaxial'' structure [11]. On the other hand, the cyclophane called CP66 (Fig. 1) has a cavity whose long side length is two methylene units larger than that of CP44, and can accommodate naphthalene with its long axis parallel to the cavity plane [12]. The stationary phase prepared using CP66 would give a different elution behavior from that having CP44 owing to a different complex structure.

This paper deals with the preparation of CP66-bonded silica gel stationary phase (CP66-SP) for HPLC and its retention and separation characteristics toward various aromatic compounds. CP66-SP was prepared by binding CP66 covalently to aminopropylsilylated silica gel through a peptide linkage, and unreacted aminopropyl residues were endcapped by acetylation. The retention behavior of various aromatic compounds on the CP66-SP was discussed in comparison with that of the CP44-SP.

## 2. Experimental

### *2.1. Reagents and materials*

Naphthalene, 1- and 2-methylnaphthalene, 1,2-, 1,3-, 1,4-, 1,5-, 1,6-, 2,6- and 2,7-dimethylnaphthalenes, *cis*- and *trans*-decahydronaphthalenes, biphenyl-, dicyclohexyl-, phenylcyclohexane, anthracene, phenanthrene, 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride (WSC) and 1-hydroxybenzotriazole (HOBT) were purchased from Wako Pure Chemical Industries Ltd. (Osaka, Japan). 2,3-Dimethylnaphthalene and 2,3-benzanthracene were purchased from Tokyo Kasei Kogyo Co. (Tokyo, Japan). 1- and 2-Ethylnaphthalene, 1,7- and 1,8-dimethylnaphthalene, 1,2-benzanthracene, chrysene, pyrene and methyl 4-(chloroformyl) butyrate were purchased from Sigma–Aldrich Japan K. K. (Tokyo, Japan). 1,8,22,29-Tetraaza [8.1.8.1] paracyclophane (**1**, CP66) was obtained from Daiichi Pure Chemicals Co. (Tokyo, Japan) and used after purification by column chromatography (silica gel/$CHCl_3$–ethyl acetate) and recrystallization from $CHCl_3$. All other reagents were of analytical reagent grade.

Aminopropylsilylated silica gel (Wakosil $5NH_2$; particle size 5 μm, pore size 120 Å, 1.2 mmol$NH_2$/g) was obtained from Wako. Octadecylsilylated silica gel-prepacked column (L-column ODS (polymeric); 150 mm×4.6 mm I.D.), abbreviated as ODS-SP in the followings, was obtained from Chemicals Inspection and Testing Institute (Tokyo, Japan).

### *2.2. Preparation of CP66-bonded stationary phase*

CP66-bonded silica gel was prepared according to the scheme in Fig. 2.

### *2.3. 1-(4-Carboxylbutanoyl)-8,22,29-triacetyl-1,8,22,29-tetraaza [8.1.8.1] paracyclophane (4)*

To a mixture of **1** (11.2g, 20 mmol) and anhydrous $K_2CO_3$ (6.0 g, 43 mmol) in dry $CH_2Cl_2$ (650 ml) was added dropwise a solution of methyl 4-(chloroformyl) butyrate (3.63 g, 22 mmol) in dry $CH_2Cl_2$ (15 ml) over a period of 1 h at 0°C. The reaction mixture was gradually warmed to room temperature and stirred for 24 h. The solution was washed with saturated $NaHCO_3$, water and brine, and dried. After removal of the solvent the residue was purified by chromatography over silica gel eluted with benzene–ethyl acetate to give 2.80 g of **2** (yield 20%).

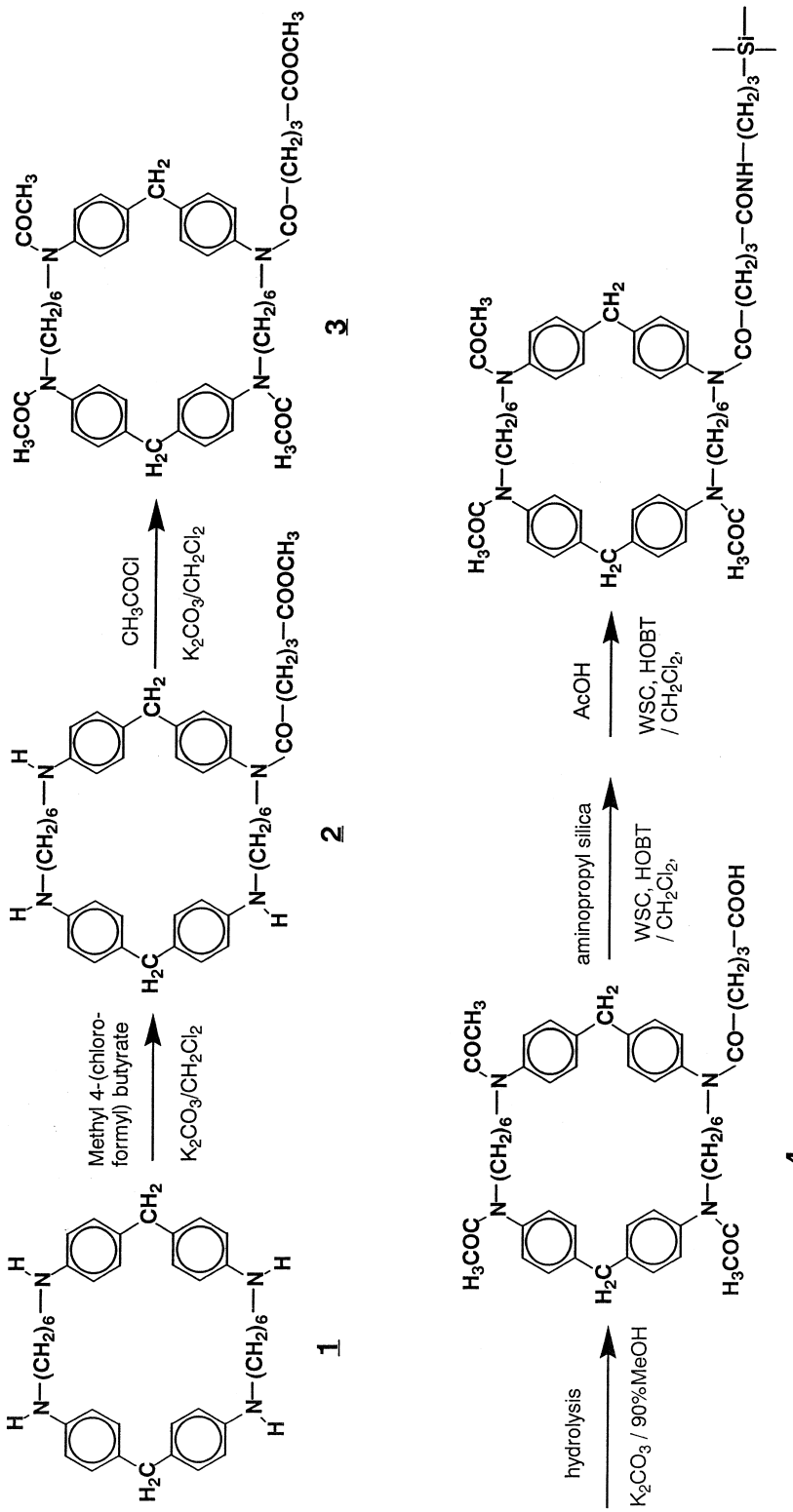



Fig. 2. Synthetic routes to CP66-bonded silica gel.

Acetylation of three residual amino groups of **2** (2.41 g, 3.5 mmol) was carried out similarly by using acetyl chloride (1.65 g, 21 mmol) instead of methyl 4-(chloroformyl) butyrate. Purification by chromatography over silica gel gave 2.48 g of **3** (yield 87%).

To a solution of $K_2CO_3$ (1.86 g, 13.5 mmol) in 90% aqueous solution of methanol (150 ml) was added **3** (3.67 g, 4.5 mmol), and the solution was refluxed for 2.5 h. After cooling, the solution was acidified with conc. HCl. The precipitated solid, **4**, was collected, and recrystallized from methanol–water (3.35 g, yield 93%).

### *2.4. CP66-bonded stationary phase*

To a suspension of **4** (1.60 g, 2.0 mmol), HOBT (0.30 g, 2.2 mmol) and aminopropyl-silylated silica gel (3.3 g) in dichloromethane (15 ml) was added a solution of WSC (0.42 g, 2.2 mmol) and tributylamine (0.52 ml, 2.2 mmol) in dichloromethane (10 ml), and the mixture was stirred for 24 h. The reaction mixture was then treated with a mixture of acetic acid (0.23 ml, 4.0 mmol), WSC (0.75 g, 3.9 mmol), tributylamine (0.93 ml, 3.9 mmol) and HOBT (0.53 g, 3.9 mmol) for 24 h to acetylate residual amino groups. The particles were filtered,

Table 1
Comparison of the retention between aromatics and the corresponding alicyclics

| Compound | CP66-SP[a] | | CP44-SP[b] | | ODS-SP[c] | |
|---|---|---|---|---|---|---|
| | $k'$ | $\alpha$ | $k'$ | $\alpha$ | $k'$ | $\alpha$ |
| Dicyclohexyl | 1.53 | 0.91 | 2.25 | 0.97 | 14.71 | 12.68 |
| Phenylcyclohexane | 1.14 | 0.68 | 1.58 | 0.68 | 2.86 | 2.47 |
| Biphenyl | 1.68 | 1.00 | 2.31 | 1.00 | 1.16 | 1.00 |
| *trans*-Decahydronaphthalene | 1.20 | 0.62 | 1.21 | 0.88 | 7.39 | 7.95 |
| *cis*-Decahydronaphthalene | 1.19 | 0.61 | 1.33 | 0.96 | 6.92 | 7.44 |
| Naphthalene | 1.95 | 1.00 | 1.38 | 1.00 | 0.93 | 1.00 |
| Indan | 1.02 | 0.90 | 1.00 | 0.96 | 1.22 | 1.49 |
| Indene | 1.13 | 1.00 | 1.04 | 1.00 | 0.82 | 1.00 |
| Cyclohexane | 1.13 | 1.33 | 0.73 | 1.26 | 2.18 | 3.96 |
| Benzene | 0.85 | 1.00 | 0.58 | 1.00 | 0.55 | 1.00 |

[a] Acetonitrile–water (70:30, v/v). Temperature: 25°C.
[b] Acetonitrile–water (60:40, v/v). Temperature: 25°C.
[c] Acetonitrile–water (85:15, v/v). Temperature: 25°C.

washed sequentially with dichloromethane, ethanol and water, and dried at 90°C under vacuum for 24 h. The quantity of bound CP66 estimated from the element analysis was 0.26 mmol/g of stationary phase.

### 2.5. Chromatographic measurements

The packing of the CP66-bonded stationary phase (CP66-SP) into a stainless-steel column (150 mm× 4.6 mm I.D.) was carried out by GL Sciences Co. (Tokyo, Japan) using the conventional high-pressure slurry-packing procedure.

Chromatography was performed using a standard apparatus: a pump (DIP-1, Jasco, Tokyo, Japan), a UV detector (SPD-10A, Shimadzu, Kyoto, Japan), a refractive index detector (SE-51, Syowa Denko, Tokyo, Japan), an integrator (C-R7A plus, Shimadzu, Kyoto, Japan) and an injector (model 7125, Rheodyne, CA, USA). The column temperature was controlled by dipping the column in a thermostated water bath. The eluent was a mixture of acetonitrile and water, and the flow-rate was 1 ml/min.

## 3. Results and discussion

Table 1 shows the retention of various aromatic compounds and their corresponding alicyclic compounds on the CP66-SP, in comparison with those on the CP44-SP and ODS-SP (octadecylsilylated silica gel stationary phase). $k'$ is the capacity factor, and $\alpha$ is the ratio of the capacity factor of each alicyclic compound to that of the corresponding aromatic compound. As seen from the table, the CP66-SP as well as the CP44-SP showed preferential retention toward aromatic compounds: The $\alpha$-values of dicyclohexyl and decahydronaphthalene on the CP66- and CP44-SP were $<1$, indicating that aromatic compounds were retained more strongly than alicyclic compounds. This is a marked contrast to the results obtained with the ordinary reversed-phase stationary phase (ODS-SP), where more hydrophobic

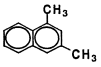
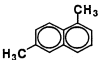
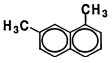
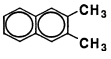
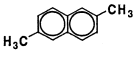
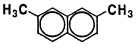
Table 2
The retention ($k'$) and the retention ratio ($\alpha$) of some naphthalene derivatives on CP66-SP, CP44-SP and ODS-SP

| Naphthalene derivative | | CP66-SP[a] | | CP44-SP[b] | | ODS-SP[c] | |
|---|---|---|---|---|---|---|---|
| | | $k'$ | $\alpha$ | $k'$ | $\alpha$ | $k'$ | $\alpha$ |
| Naphthalene |  | 1.70 | 1.00 | 1.46 | 1.00 | 1.15 | 1.00 |
| 1-Methylnaphthalene |  | 2.38 | 1.40 | 1.37 | 0.94 | 1.57 | 1.37 |
| 2-Methylnaphthalene |  | 1.71 | 1.01 | 1.74 | 1.19 | 1.62 | 1.41 |
| 1-Ethylnaphthalene |  | 2.34 | 1.38 | 1.54 | 1.05 | 2.02 | 1.76 |
| 2-Ethylnaphthalene |  | 1.77 | 1.04 | 1.99 | 1.36 | 2.10 | 1.83 |
| 1,5-Dimethylnaphthalene |  | 2.93 | 1.72 | 1.51 | 1.03 | 2.18 | 1.90 |
| 2,6-Dimethylnaphthalene |  | 1.96 | 1.15 | 2.10 | 1.44 | 2.34 | 2.03 |

[a] Acetonitrile–water (75:25, v/v). Temperature: 20°C.
[b] Acetonitrile–water (60:40, v/v). Temperature: 20°C.
[c] Acetonitrile–water (85:15, v/v). Temperature: 20°C.

alicyclic compounds were eluted after the corresponding aromatic compounds. As discussed in the preceding paper [9], the preferential retention of aromatic compounds on the cyclophane-bonded stationary phases reflects the effectiveness of bonded cyclophanes. The cyclophanes used are effective only on aromatic compounds because they do not form complexes with aliphatic compounds due to their narrow cavities. The preferential retention of aromatic compounds also means that the cyclophane-involved interaction worked for aromatic compounds was stronger than the hydrophobic interaction for alicyclic compounds. The low complexation ability of cyclophane with benzene derivatives [6,12] will explain why, in the case of one-ring compounds (benzene and cyclohexane), no reversal of elution

Table 3
The retention ($k'$) and the retention ratio ($\alpha$) of dimethylnaphthalene isomers on CP66-SP, CP44-SP and ODS-SP

| Dimethyl-naphthalene | | CP66-SP[a] | | CP44-SP[b] | | ODS-SP[b] | |
|---|---|---|---|---|---|---|---|
| | | $k'$ | $\alpha$ | $k'$ | $\alpha$ | $k'$ | $\alpha$ |
| 1,4-Dimethylnaphthalene |  | 4.41 | 1.71 | 1.45 | 0.73 | 9.49 | 0.90 |
| 1,5-Dimethylnaphthalene |  | 4.35 | 1.69 | 1.51 | 0.76 | 9.51 | 0.90 |
| 1,8-Dimethylnaphthalene |  | 4.48 | 1.74 | 1.39 | 0.70 | 8.65 | 0.82 |
| 1,2-Dimethylnaphthalene |  | 3.53 | 1.37 | 1.56 | 0.78 | 9.06 | 0.86 |
| 1,3-Dimethylnaphthalene |  | 3.92 | 1.52 | 1.58 | 0.79 | 9.93 | 0.94 |
| 1,6-Dimethylnaphthalene |  | 3.19 | 1.24 | 1.60 | 0.80 | 9.88 | 0.93 |
| 1,7-Dimethylnaphthalene |  | 3.56 | 1.38 | 1.46 | 0.73 | 9.59 | 0.91 |
| 2,3-Dimethylnaphthalene |  | 2.68 | 1.04 | 1.98 | 0.99 | 9.59 | 0.91 |
| 2,6-Dimethylnaphthalene |  | 2.93 | 1.14 | 2.10 | 1.06 | 10.71 | 1.01 |
| 2,7-Dimethylnaphthalene |  | 2.58 | 1.00 | 1.99 | 1.00 | 10.59 | 1.00 |

[a] Acetonitrile–water (70:30, v/v). Temperature: 20°C.
[b] Acetonitrile–water (60:40, v/v). Temperature: 20°C.

order between the cyclophane-bonded and the reversed-phase stationary phases was observed.

Table 2 shows the retention ($k'$) and the retention ratio ($\alpha$) of naphthalene derivatives, where $\alpha$ stands for the ratio of the capacity factor of each aromatic compound to that of naphthalene. It is noteworthy that the retention orders of the CP66-SP and CP44-SP were not only quite different from that of the ODS-SP, but also completely reversed to each other. The retention order of α- and β-monosubstituted naphthalenes (methyl- and ethylnaphthalenes) on the CP66-SP was α>β, whereas that on the CP44-SP was α<β. This reversal of elution order was also observed for ten geometrical isomers of dimethylnaphthalenes as shown in Table 3, where $\alpha$ is defined as the ratio of the capacity factor of each dimethylnaphthalene to that of 2,7-dimethylnaphthalene. The retention order on the CP66-SP was: 2,7-<2,3-<2,6-<1,6-<1,2-<1,7-<1,3-<1,5-<1,4-<1,8-, whereas that on the CP44-SP was: 1,8-<1,4-<1,7-<1,5-<1,2-<1,3-<1,6-<2,3-<2,7-<2,6-. That is to say, the retention on the CP66-SP is β,β-dimethylnaphthalene<α,β-dimethylnaphthalene<α,α-dimethylnaphthalene, whereas that on the CP44-SP, α,α-dimethylnaphthalene<α,β-dimethylnaphthalene<β,β-dimethylnaphthalene. Similarly, there was a big difference in retention of multi-ring aromatic compounds between the CP66- and CP44-SP (Table 4). The CP66-SP showed preferential retention for ''bent'' structure aromatic compounds such as phenanthrene, 1,2-benzanthracene and chrysene, whereas the CP44-SP, for ''linear'' structured ones, such as anthracene and 2,3-benzanthracene.

Odashima et al. proposed the complex structures of aromatic compounds with CP44 and CP66 on the basis of NMR and structure-modification studies [11–13]. The complex between CP44 and naphthalene has the structure where the naphthalene ring is included in the cyclophane cavity with its long axis tilted about 30° (''pseudoaxial'' form), since the cavity of CP44 is too short to accommodate the naphthalene ring with its long axis parallel to the cavity plane. According to this ''pseudoaxial'' complex form, the distance between a methyl moiety of methylnaphthalene and the cyclophane ring is greater for the β-position than for the α-position. On the other hand, in the case of CP66, the naphthalene ring

Table 4
The retention ($k'$) and the retention ratio ($\alpha$) of three- and four-ring aromatic compounds on CP66-SP, CP44-SP and ODS-SP

| Aromatics | CP66-SP[a] | | CP44-SP[b] | | ODS-SP[c] | |
|---|---|---|---|---|---|---|
| | $k'$ | $\alpha$ | $k'$ | $\alpha$ | $k'$ | $\alpha$ |
| Naphthalene | 1.70 | 1.00 | 1.46 | 1.00 | 1.15 | 1.00 |
| Anthracene | 3.97 | 2.34 | 4.40 | 3.01 | 2.23 | 1.94 |
| Phenanthrene | 5.17 | 3.04 | 3.18 | 2.18 | 2.04 | 1.77 |
| 1,2-Benzanthracene | 12.65 | 7.44 | 7.03 | 4.82 | 4.15 | 3.61 |
| 2,3-Benzanthracene | 12.40 | 7.29 | 12.15 | 8.32 | 5.05 | 4.39 |
| Chrysene | 16.57 | 9.75 | 7.84 | 5.37 | 4.08 | 3.55 |
| Pyrene | 17.49 | 10.29 | 3.52 | 2.41 | 3.30 | 2.87 |

[a] Acetonitrile–water (75:25, v/v). Temperature: 20°C.
[b] Acetonitrile–water (60:40, v/v). Temperature: 20°C.
[c] Acetonitrile–water (85:15, v/v). Temperature: 20°C.

can be accommodated in the cyclophane cavity with its long axis parallel to the cavity plane, because the cavity of CP66 is two methylene units longer than that of CP44. In this complex structure, the distance between the methyl group and the cyclophane ring becomes greater for the α-position than for the β-position. These steric hindrances make the complex of α- and β-derivatives more stable for CP66 and CP44, respectively. The more direct proof of complex stability has been given for CP44 and CP56 by using sulfonaphthalenes as guest [12]. The stability constants of CP44 with 1- and 2-sulfonaphthalenes and 1,5- and 2,6-disulfonaphthalenes in an aqueous solution were $1.5\times10^3$ and $1.9\times10^4$, $4.4\times10^3$ and $1.8\times10^5$, respectively, whereas those of CP56 were, $3.8\times10^3$ and $2.9\times10^3$, $1.1\times10^5$ and $3.3\times10^4$, re-

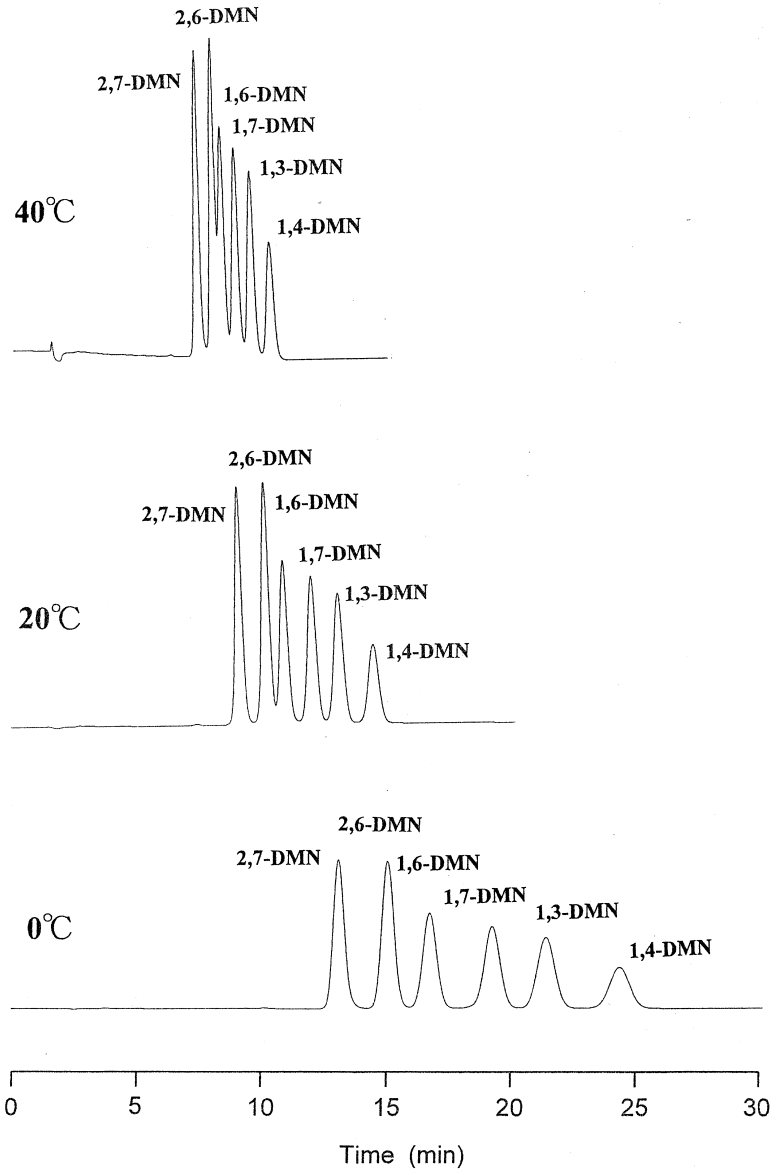



Fig. 3. Temperature dependence of the separation of a mixture of six dimethylnaphthalenes on the CP66-SP. DMN stands for dimethylnaphthalene. Eluent: $CH_3CN–H_2O$ (60:40, v/v), flow-rate: 1 ml/min, detection: UV 254 nm.

spectively. The order of stability constants of CP44 toward α- and β-substituted naphthalenes was completely reversed to that of CP56. Although the cavity size of CP66 is not the same as that of CP56, we can assume without serious error that CP66 would have selectivity similar to CP56. This difference in complexing ability explains the difference in retention order of aromatic isomers between CP44 and CP66. The difference in retention of multi-ring aromatic compounds can be explained similarly because anthracene and 2,3-benzanthracene are regarded as β,β-disubstituted naphthalenes, and phenanthrene, 1,2-benzanthracene and chrysene are as α,β-disubstituted naphthalenes. It is noteworthy that pyrene showed extraordinarily strong retention on the CP66-SP. This finding gives another proof that only CP66 has a cavity enough to accommodate naphthalene with its long axis parallel to the cavity plane.

The retention of all the dimethylnaphthalenes was increased with the decrease of temperature, and like the CP44-SP, the difference in retention of isomers became larger with the decrease of temperature. This phenomenon is observed in many host-involved separation systems and can be explained thermodynamically [2,3,14,15]. Fig. 3 shows the temperature dependence of chromatograms of six dimethylnaphthalenes on the CP66-SP. It is noteworthy that complete separation of 2,6-dimethylnaphthalene from 2,7-dimethylnaphthalene was observed on this CP66-SP as well as previously reported CP44-SP. Since their mutual separation is very difficult in usual methods, this finding gives promising possibility of the extensive usage of host compounds as selectors of stationary phases.

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