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Short communication

# New chiral crown ether stationary phase for the liquid chromatographic resolution of α-amino acid enantiomers

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**Abstract**

A new chiral stationary phase (CSP) for the liquid chromatographic separation of enantiomers was prepared by bonding a novel enantiopure (diphenyl-substituted 1,1′-binaphthyl) crown ether to 5 μm silica gel. The resulting CSP was applied to the separation of the enantiomers of various natural and unnatural α-amino acids. All α-amino acids tested were resolved very well on the new CSP, with the exception of proline, which does not contain a primary amino group. The resolution of α-amino acid enantiomers on this new CSP was found to be dependent on the type and amounts of organic and acidic modifiers, and on column temperature. 

*Keywords:* Chiral stationary phases, LC; Enantiomer separation; Crown ether; Amino acids

## 1. Introduction

The liquid chromatographic separation of enantiomers on chiral stationary phases (CSPs) has emerged as the most effective and convenient means for determining the enantiomeric composition of many chiral compounds, including a variety of chiral drugs [1,2]. CSPs based on chiral crown ethers have been shown to be very effective in resolving the enantiomers of those compounds that contain a primary amine functional group [3]. In pioneering work in the late 1970s, Cram and co-workers immobilized bis-(1,1′-binaphthyl)-22-crown-6 on polystyrene or silica gel to afford CSPs, which showed reasonable resolution of the enantiomers of α-amino acids and their derivatives [4,5]. In the subsequent work of Shinbo and co-workers, structurally related chiral crown ethers based on disubstituted 1,1′-binaphthyl-20-crown-6 were dynamically coated on octadecyl silica gel to afford useful chiral crown ether CSPs [6,7]. A related CSP, commercialized as CROWNPAK CR (Daicel Chemical Industries), has proven to be useful for the chromatographic resolution of chiral compounds containing a primary amino group [8,9]. However, the dynamically coated nature of this CSP has somewhat limited its utility. For example, the use of a mobile phase containing more than 15% methanol with this CSP results in leaching of the chiral crown selector and deterioration of CSP

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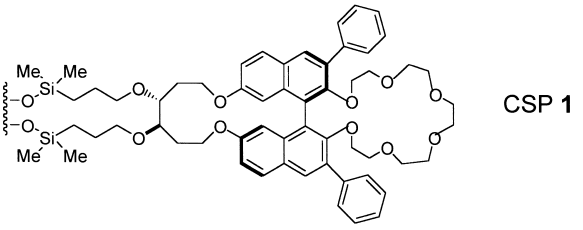


Fig. 1. Structure of CSP **1**.

performance [10]. Very recently, covalently-linked CSPs based on (+)-(18-crown-6)-2,3,11,12-tetracarboxylic acid have been developed by others and us and successfully utilized in resolving various racemic compounds containing a primary amino group [11–15].

In the present study, we report the preparation of a novel chiral crown ether stationary phase (CSP **1**) (Fig. 1) starting from diphenyl substituted *cyclo*-BINOL and show its application to the resolution of α-amino acid enantiomers. The chiral crown ether moiety of CSP **1** bears some similarity to the one utilized by Shinbo and co-workers [6,7], but differs in the presence of functionality that can enable covalent immobilization to silica gel. Consequently, covalently-linked CSP **1** is expected to be useful in a variety of mobile phases.

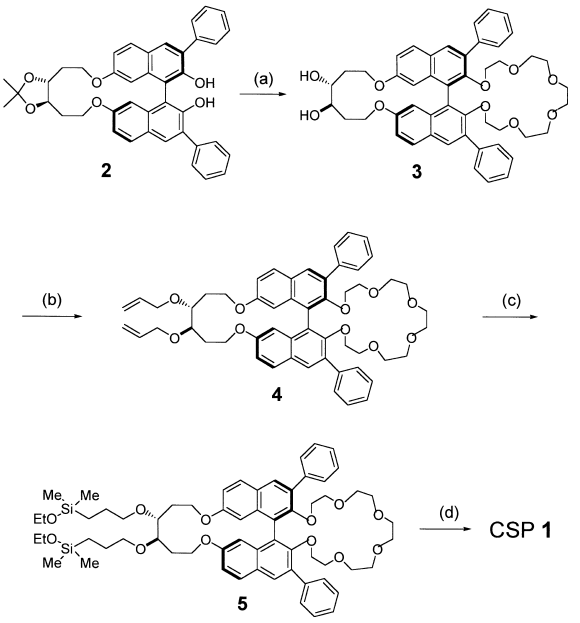


Fig. 2. Scheme for the preparation of CSP **1**. (a) (1) KOH, pentaethyleneglycol ditosylate, tetrahydrofuran (THF), reflux for 72 h. (2) 1 *M* HCl solution, methanol. (b) (1) NaH, THF, reflux for 30 min. (2) Allyl bromide, THF, reflux for 4 h. (c) (1) $(CH_3)_2ClSiH$, $H_2PtCl_6 \cdot 6H_2O$, methylene chloride, reflux for 5 h. (2) Ethanol–triethylamine (1:1, v/v), methylene chloride. (d) 5 μm silica gel, Dean–Stark trap, toluene, reflux for 72 h.

## 2. Experimental

CSP **1** was prepared as shown in Fig. 2. Diphenyl substituted (*R*)-*cyclo*-BINOL **2**, prepared via a previously reported procedure [16], was treated with KOH and pentaethyleneglycol ditosylate in tetrahydrofuran and then HCl solution to afford chiral crown ether **3**. Chiral crown ether **3** was treated with NaH and then allyl bromide in tetrahydrofuran to afford *O*-allylated chiral crown ether **4**. *O*-Allylated chiral crown ether **4** thus prepared was treated with dimethylchlorosilane in the presence of hydrated chloroplatinic acid ($H_2PtCl_6 \cdot 6H_2O$) as a catalyst in methylene chloride and then treated with the mixed solvent of ethanol–triethylamine (1:1, v/v) to afford hydrosilylated chiral crown ether **5**. Finally chiral crown ether **5** was treated with 5 μm Rexchrom silica gel (Regis Technologies, Morton Grove, IL, USA) to afford CSP **1**. The amount of chiral selector bonded to silica gel was calculated to be 0.10 mmol of chiral selector per gram of stationary phase based on elemental analysis of the bonded phase (C, 6.87%; H, 0.98%). Finally, CSP **1** was packed into 250 mm×4.6 mm I.D. stainless steel high-performance liquid chromatography (HPLC) column using a conventional slurry packing method with an Alltech slurry packer.

The chiral column packed with CSP **1** was evaluated using an HPLC system consisting of a Waters Model 515 HPLC pump, a Rheodyne Model 7125 injector with a 20-μl sample loop, a Younglin M720 absorbance detector (variable wavelength) and a Youngin D520B computing integrator. The temperature of the column was controlled by using a Julabo F30 Ultratemp 2000 cooling circulator. Injection samples prepared by dissolving α-amino acids in water at a concentration of 1.0 mg/ml were available from a previous study [14] and an injection size of 3 μl was typically used. The chiral column was

equally effective for the chiral separation during the period of this experiment (more than 3 months).

## 3. Results and discussion

CSP **1,** prepared by covalently bonding chiral crown ether **3** to 5 μm silica gel, was evaluated in the separation of various α-amino acid enantiomers. The resolution results are summarized in Table 1 and representative chromatograms are shown in Fig. 3. All data shown in Table 1 and the chromatograms shown in Fig. 3 were obtained under identical chromatographic conditions given in the footnote of Table 1. These conditions, while not optimal for any of the α-amino acids, were generally useful.

It was previously demonstrated that complexation of the ammonium ion ($R\text{-}NH_3^+$) inside the cavity of the crown ether ring is essential for chiral recognition of primary amino compounds [17]. In this instance, the acid added to the mobile phase is necessary for protonation of α-amino acids to enhance the formation of diastereomeric complexes of α-amino acids with the crown ether of CSP **1**. Ammonium acetate added to the mobile phase is used to reduce the retention of the two enantiomers on the chiral column, by competing with the ammonium ion ($R\text{-}NH_3^+$) of α-amino acids for complexation by the crown ether of the CSP.

As shown in Table 1, the enantiomers of all natural and unnatural α-amino acids were resolved very well on CSP **1** except for proline, which does not contain a primary amino group. The elution orders shown in Table 1 were determined by inject-

Table 1
Resolution of various α-amino acids on CSP **1**[a]

| Amino acid | $k_1'$[b] | $k_2'$[c] | $\alpha$[d] | $R_s$[e] | Configuration[f] |
|---|---|---|---|---|---|
| Alanine | 0.58 | 2.18 | 3.76 | 4.10 | D |
| Arginine | 0.58 | 0.90 | 1.55 | 1.20 | D |
| Asparagine | 0.28 | 0.46 | 1.64 | 1.40 | D |
| Aspartic acid | 0.42 | 1.02 | 2.43 | 2.70 | D |
| Cysteine | 0.38 | 0.97 | 2.55 | 1.80 | D |
| Glutamic acid | 0.59 | 3.48 | 5.90 | 8.40 | D |
| Glutamine | 0.36 | 1.76 | 4.89 | 3.80 | D |
| Histidine | 0.33 | 0.49 | 1.48 | 1.00 | D |
| Isoleucine | 0.31 | 0.58 | 1.87 | 1.30 | D |
| Leucine | 0.64 | 3.16 | 4.94 | 5.00 | D |
| Methionine | 0.77 | 3.46 | 4.49 | 5.70 | D |
| Phenylalanine | 0.79 | 2.38 | 3.01 | 4.80 | D |
| Proline | 0.23 | 0.23 | 1.00 | | |
| Serine | 0.27 | 0.50 | 1.85 | 1.50 | D |
| Threonine | 0.24 | 0.64 | 2.67 | 2.00 | D |
| Tryptophan | 1.40 | 4.03 | 2.88 | 7.30 | D |
| Tyrosine | 0.62 | 1.91 | 3.08 | 7.60 | D |
| Valine | 0.28 | 0.52 | 1.86 | 1.30 | D |
| Phenylglycine | 0.70 | 3.97 | 5.67 | 7.78 | D |
| 4-Hydroxyphenylglycine | 0.67 | 4.71 | 7.03 | 11.61 | D |
| 3,4-Dihydroxyphenylalanine (dopa) | 0.73 | 1.99 | 2.73 | 5.74 | |
| 2-Aminopentanoic acid (Norval) | 0.56 | 2.26 | 4.04 | 6.90 | |
| 2-Aminohexanoic acid (Norleu) | 0.71 | 2.81 | 3.96 | 4.20 | |
| 2-Aminooctanoic acid | 2.62 | 8.13 | 3.10 | 4.60 | |

[a] Mobile phase: 50% $CH_3OH$ in water+$HClO_4$ ($1.0\cdot10^{-2}$ *M*)+$CH_3COONH_4$ ($1.0\cdot10^{-3}$ *M*). Flow-rate: 0.5 ml/min. Detection: UV at 225 nm. Temperature: 20°C.

[b] Retention factor of the first eluted enantiomer.

[c] Retention factor of the second eluted enantiomer.

[d] Separation factor.

[e] Resolution factor.

[f] Absolute configuration of the second eluted enantiomer. For blanks, the elution orders were not determined.

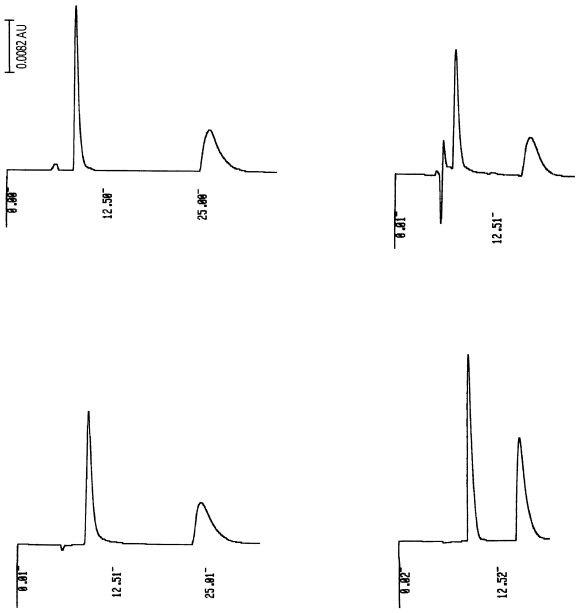


Fig. 3. Representative chromatograms for the resolution of (top left) phenylglycine, (top right) glutamic acid, (bottom left) methionine and (bottom right) 3,4-dihydroxyphenylalanine (dopa). For chromatographic conditions, see the footnote to Table 1. The scale of the abscissa is presented in min.

ing configurationally known samples. The elution orders were consistent, the D-enantiomers being retained longer than the L-enantiomers. The separation factors ($\alpha$) were reasonably good and consistently better than those on the related commercial CROWNPAK CR CSP.

In order to investigate the effect of mobile phase modifiers and temperature on the enantioselectivity of CSP **1**, the well resolved analytes, phenylglycine and methionine, were chosen for more detailed study. The effects of three organic modifiers (methanol, ethanol and acetonitrile) are summarized in Table 2. The enantioselectivity ($\alpha$) and the resolution ($R_s$) were slightly greater when acetonitrile was used as an organic modifier. The retention factor ($k_1'$), enantioselectivity ($\alpha$), and resolution ($R_s$) were all significantly influenced by the concentration of organic modifier in the mobile phase. Enantioselectivity and resolution both increased with increasing organic modifier concentration, whereas the retention factor ($k_1'$) decreased with increasing organic modifier concentration, consistent with a simple lipophilic retention model.

The effects of acidic modifiers in the mobile phase were also investigated and summarized in Table 3. As shown in Table 3, the enantioseparation of phenylglycine and methionine with mobile phase consisting of 50% methanol containing trifluoroacetic acid (10 m*M*) on CSP **1** is very similar to those with mobile phase consisting of 50% methanol containing perchloric acid (10 m*M*) or sulfuric acid (10 m*M*). The retention factor ($k_1'$) shows a maximum at a certain concentration of acidic modifier. As

Table 2
Resolution of phenylglycine and methionine on CSP **1** with the variation of the organic modifiers and their content in the mobile phase at the constant concentration of ammonium acetate ($1.0 \cdot 10^{-3}$ *M*) at 20°C[a]

| Mobile phase | Phenylglycine | | | Methionine | | |
|---|---|---|---|---|---|---|
| | $k_1'$[b] | $\alpha$[c] | $R_s$[d] | $k_1'$[b] | $\alpha$[c] | $R_s$[d] |
| 100% $H_2O$+$H_2SO_4$ (10 m*M*) | 1.15 | 3.94 | 4.33 | 0.95 | 2.22 | 2.12 |
| 20% MeOH+$H_2SO_4$ (10 m*M*) | 1.06 | 5.44 | 5.60 | 0.93 | 3.70 | 3.60 |
| 50% MeOH+$H_2SO_4$ (10 m*M*) | 0.65 | 5.86 | 7.55 | 0.68 | 4.76 | 5.50 |
| 80% MeOH+$H_2SO_4$ (10 m*M*) | 0.37 | 7.70 | 10.00 | 0.55 | 6.42 | 10.50 |
| 20% Ethanol+$H_2SO_4$ (10 m*M*) | 0.87 | 5.01 | 6.10 | 0.72 | 3.63 | 3.80 |
| 50% Ethanol+$H_2SO_4$ (10 m*M*) | 0.57 | 5.32 | 7.80 | 0.60 | 4.20 | 4.40 |
| 80% Ethanol+$H_2SO_4$ (10 m*M*) | 0.35 | 6.86 | 8.60 | 0.46 | 5.96 | 7.60 |
| 20% $CH_3CN$+$H_2SO_4$ (10 m*M*) | 1.06 | 7.00 | 12.80 | 0.89 | 4.24 | 6.00 |
| 50% $CH_3CN$+$H_2SO_4$ (10 m*M*) | 0.69 | 9.32 | 14.60 | 0.59 | 6.34 | 7.20 |
| 80% $CH_3CN$+$H_2SO_4$ (10 m*M*) | 0.51 | 10.49 | 15.00 | 0.52 | 7.54 | 11.60 |

[a] Flow-rate: 0.5 ml/min. Detection: UV at 225 nm UV.
[b] Retention factor.
[c] Separation factor.
[d] Resolution factor.

Table 3
Resolution of phenylglycine and methionine on CSP **1** with the variation of the acidic modifiers and their content in the mobile phase at the constant concentration of ammonium acetate ($1.0 \cdot 10^{-3}$ *M*) at 20°C[a]

| Mobile phase | Phenylglycine | | | Methionine | | |
|---|---|---|---|---|---|---|
| | $k_1'$[b] | $\alpha$[c] | $R_s$[d] | $k_1'$[b] | $\alpha$[c] | $R_s$[d] |
| 50% MeOH+$CF_3COOH$ (10 m*M*) | 0.95 | 5.44 | 7.00 | 1.02 | 4.24 | 5.10 |
| 50% MeOH[e] | 0.16 | 2.00 | 1.00 | 0.15 | 1.60 | 0.71 |
| 50% MeOH+$HClO_4$ (1 m*M*) | 0.27 | 3.07 | 2.40 | 0.32 | 2.25 | 1.23 |
| 50% MeOH+$HClO_4$ (5 m*M*) | 0.86 | 4.97 | 7.16 | 0.91 | 4.20 | 5.71 |
| 50% MeOH+$HClO_4$ (10 m*M*) | 0.70 | 5.67 | 7.78 | 0.77 | 4.49 | 5.70 |
| 50% MeOH+$HClO_4$ (15 m*M*) | 0.62 | 6.24 | 7.89 | 0.64 | 4.83 | 5.40 |
| 50% MeOH+$H_2SO_4$ (1 m*M*) | 0.42 | 4.64 | 5.14 | 0.42 | 5.45 | 4.40 |
| 50% MeOH+$H_2SO_4$ (5 m*M*) | 0.87 | 5.16 | 8.00 | 0.87 | 4.46 | 5.17 |
| 50% MeOH+$H_2SO_4$ (10 m*M*) | 0.65 | 5.86 | 7.55 | 0.68 | 4.76 | 5.50 |
| 50% MeOH+$H_2SO_4$ (15 m*M*) | 0.57 | 6.30 | 8.13 | 0.59 | 4.95 | 5.56 |

[a] Flow-rate: 0.5 ml/min. Detection: UV at 225 nm.
[b] Retention factor.
[c] Separation factor.
[d] Resolution factor.
[e] Without acidic modifier.

the concentration of acidic modifier in the mobile phase increases from 0 to 5 m*M*, the retention factor ($k_1'$) increases. However, further increases in the acid concentration diminish the retention of the enantiomers. The initial increase in $k_1'$ might be rationalized by increased complexation of the primary ammonium ion ($R\text{-}NH_3^+$) by the crown ether of the CSP, while the decrease in $k_1'$ at higher acid concentrations may be a result of the greater ionic strength of the mobile phase. Enantioselectivity ($\alpha$) generally increases as the concentration of acidic modifier in the mobile phase increases. Resolution ($R_s$) initially improves with increasing concentration of acid modifier up to a certain value (for example 5 m*M*), at which point further increases in acid concentration does not significantly change $R_s$.

The effect of ammonium ion modifier on the resolution of phenylglycine and methionine on CSP **1** is summarized in Table 4. Without ammonium ion modifiers in the mobile phase, the resolution of phenylglycine and methionine on CSP **1** takes quite a long time. For example, elution of the D-enantiomer of phenylglycine from the column takes 102 min and elution of the L-enantiomer takes 15 min when even 80% $CH_3CN$ containing perchloric acid ($1.0 \cdot 10^{-2}$ *M*) is used as a mobile phase. The retention time is

Table 4
Resolution of phenylglycine and methionine on CSP **1** with the variation of ammonium salt in the mobile phase at 20°C[a]

| Mobile phase | Phenylglycine | | | Methionine | | |
|---|---|---|---|---|---|---|
| | $k_1'$[b] | $\alpha$[c] | $R_s$[d] | $k_1'$[b] | $\alpha$[c] | $R_s$[d] |
| 80% $CH_3CN$+$H_2SO_4$ (10 m*M*)+$CH_3COONH_4$ (0 m*M*) | 1.96 | 9.57 | 8.10 | 1.91 | 6.36 | 4.30 |
| 80% $CH_3CN$+$H_2SO_4$ (10 m*M*)+$CH_3COONH_4$ (1 m*M*) | 0.51 | 10.49 | 15.00 | 0.52 | 7.54 | 11.60 |
| 80% $CH_3CN$+$H_2SO_4$ (10 m*M*)+$CH_3COONH_4$ (5 m*M*) | 0.16 | 12.75 | 9.90 | 0.20 | 7.30 | 7.10 |
| 50% Methanol+$H_2SO_4$ (10 m*M*)+$CH_3COONH_4$ (1 m*M*) | 0.65 | 5.86 | 7.55 | 0.68 | 4.76 | 5.50 |
| 50% Methanol+$H_2SO_4$ (10 m*M*)+$NH_4Cl$ (1 m*M*) | 0.60 | 5.93 | 7.85 | 0.72 | 4.56 | 5.60 |
| 50% Methanol+$HClO_4$ (10 m*M*)+$CH_3COONH_4$ (1 m*M*) | 0.70 | 5.67 | 7.78 | 0.77 | 4.49 | 5.70 |
| 50% Methanol+$HClO_4$ (10 m*M*)+$NH_4Cl$ (1 m*M*) | 0.81 | 5.60 | 8.40 | 0.77 | 4.13 | 5.93 |

[a] Flow-rate: 0.5 ml/min. Detection: UV at 225 nm.
[b] Retention factor.
[c] Separation factor.
[d] Resolution factor.

reduced dramatically when ammonium acetate (1.0 m*M*) is added to the mobile phase, owing to competition for crown ether complex formation. These results are very similar to those reported for the separation of racemic amino compounds on CROWNPAK CR with the addition of cations such as $NH_4^+$ to the mobile phase [18].

Addition of ammonium acetate (1.0 m*M*) to the mobile phase, however, increased enantioselectivity ($\alpha$) and especially resolution ($R_s$). Introduction of additional ammonium acetate (5.0 m*M*) to the mobile phase resulted in a further decrease in retention factor ($k'$). In this case, the enantioselectivity ($\alpha$) increases for phenylglycine but decreases for methionine, whereas, the resolution ($R_s$) of both compounds decreases sharply. From these results, the optimum concentration of ammonium acetate seems to be ca. 1.0 m*M*. In addition to ammonium acetate, ammonium chloride was tested as a mobile phase additive. As shown in Table 4, little difference between ammonium acetate and ammonium chloride was observed. The anions in the mobile phase have been reported to show significant effects on the chromatographic resolution behaviors on CROWNPAK CR because of the hydrophobic interaction affected by the chaotropicity of the counteranions [18]. However, the hydrophobic interaction between the stationary phase and the solute–counteranion complex on CSP **1** does not seem to be as significant as on CROWNPAK CR because CSP **1** does not contain $C_{18}$ groups.

The effect of column temperature on the enantioselectivity for phenylglycine and methionine on CSP **1** is shown in Table 5. As the temperature of the column decreases, the retention factors ($k_1'$) and the enantioselectivity ($\alpha$) both increase. At lower temperature, the diastereomeric complexes formed between the individual enantiomers and the CSP are expected to each become more energetically favorable. This is typically more significant for the more stable diastereomeric complex. Consequently, the retention and the separation factors ($k_1'$ and $\alpha$) both increase as the temperature of the column decreases. The effect of temperature on resolution ($R_s$) was not significant.

In conclusion, CSP **1** prepared by bonding chiral crown ether **3** to silica gel is an excellent agent for resolving the enantiomers of various natural and unnatural α-amino acids. The resolution results with this CSP are highly competitive with those derived from commercial crown ether-based CSPs (such as CROWNPAK CR from Daicel). Another important advantage of CSP **1** over the CROWNPAK CR column is that it can be used with various mobile phases without significant deterioration in its chiral recognition ability, since the chiral selector of CSP **1** is covalently bonded to silica gel. Mobile phase modifiers including organic solvents, acids and ammonium salts can be used to control to some extent chromatographic factors such as retention ($k'$), separation ($\alpha$) and resolution ($R_s$). The temperature of the column is also an important factor in controlling chromatographic behavior. The chiral recognition mechanism is not completely clear at present, however, diastereoselective complexation of the primary ammonium group ($R\text{-}NH_3^+$) of the analyte enantiomers within the chiral crown ether cavity of the CSP is expected to be important. In addition to α-amino acids, other chiral compounds containing a primary amino group, such as amines and amino alcohols are expected to be resolved on CSP **1**. Investigations into the enantioseparation of these and other compounds on CSP **1** are underway in our laboratory and will be reported in due course.

Table 5
Resolution of phenylglycine and methionine on CSP **1** with the variation of the temperature of the column[a]

| Temperature (°C) | Phenylglycine | | | Methionine | | |
|---|---|---|---|---|---|---|
| | $k_1'$[b] | $\alpha$[c] | $R_s$[d] | $k_1'$[b] | $\alpha$[c] | $R_s$[d] |
| 25 | 0.65 | 4.97 | 8.46 | 0.67 | 4.04 | 5.25 |
| 20 | 0.70 | 5.67 | 7.78 | 0.77 | 4.49 | 5.70 |
| 15 | 0.80 | 6.65 | 8.52 | 0.84 | 5.29 | 6.00 |
| 10 | 0.85 | 8.15 | 8.52 | 0.96 | 6.08 | 6.17 |

[a] Mobile phase: 50% MeOH in water+$HClO_4$ ($1.0\cdot10^{-2}$ *M*)+$CH_3COONH_4$ ($1.0\cdot10^{-3}$ *M*). Flow-rate: 0.5 ml/min. Detection: UV at 225 nm.
[b] Retention factor.
[c] Separation factor.
[d] Resolution factor.

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