A New Chiral Stationary Phase Derived from Cyclohexylamide Derivative of (S)-Naproxen for the Liquid Chromatographic Resolution of Enantiomers

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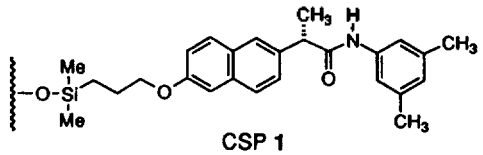
A new chiral stationary phase (CSP 2) derived from cyclohexylamide of (S)-naproxen has been prepared. CSP 2 has shown greater enantioselectivities for the two enantiomers of N-(3,5-dinitrobenzoyl)-a-amino esters and amides than the CSP derived from 3,5-dimethylanilide of (S)-naproxen (CSP 1) as expected from the reciprocity conception of chiral recognition. However, CSP 2 has been found to be worse than CSP 1 in resolving N-(3,5-dinitrobenzoyl)-a-arylalkylamines, supporting the previously proposed chiral recognition mechanism which utilizes the 3,5-dimethylphenyl group of CSP 1 as an alternative π-basic interaction site. In addition, CSP 2 has been found to be reasonably good in resolving the two enantiomers of a variety of other π-acidic racemates.

Introduction

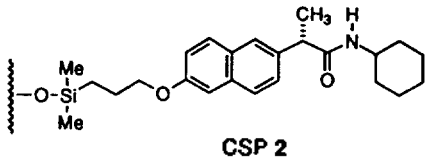
Pirkle type chiral stationary phases (CSPs) have been known to resolve racemic compounds through the stereoselective interactions including charge transfer π-π interactions between aromatic rings with the two enantiomers of racemates.[1] For the effective π-π interaction between aromatic rings, both of the CSP and the analyte should contain appropriate π-acidic or π-basic aromatic rings.[2] In this context, (S)-naproxen, which is a well known anti-inflammatory drug and readily available as an optically active form,[3] has been considered to be an attractive candidate as a chiral selector of Pirkle type CSPs because its 6-methoxy-2-naphthyl group can be utilized as an effective π-basic interaction site and indeed various CSPs derived from it have been reported.[4]

We also have been interested in the use of (S)-naproxen as a chiral selector of Pirkle type CSPs and consequently have developed several CSPs based on it.[5] For example, CSP 1 prepared from 3,5-dimethylanilide derivative of (S)-naproxen was found to show greater enantioselectivity than the CSP derived from a simple alkyl amide derivative of (S)-naproxen in resolving various π-acidic racemates.[6]

Even though the CSPs based on (S)-naproxen developed in our laboratory were successful in resolving various racemates, our efforts to develop further improved CSPs are still going on. In connection with this, we recently prepared various derivatives of racemic naproxen and resolved them on a π-acidic CSP derived from (S)-N-(3,5-dinitrobenzoyl)leucine.[7] From this study we found that cyclohexylamide derivative of racemic naproxen was resolved much better than the corresponding 3,5-dimethylanilide derivative on a π-acidic CSP derived from (S)-N-(3,5-dinitrobenzoyl)leucine.[7] This result suggests in connection with the reciprocity conception of chiral recognition[8] that cyclohexylamide derivative of (S)-naproxen could be a better candidate as a chiral selector than the corresponding 3,5-dimethylanilide derivative, which is the actual chiral selector of CSP 1[6] and prompts us to develop an improved CSP (CSP 2) derived from cyclohexylamide derivative of (S)-naproxen. In addition, CSP 2 is expected to elucidate the role of the second π-basic group such as 3,5-dimethylphenyl group of CSP 1 which was assumed to be used as an alternative π-basic interaction site in resolving N-(3,5-dinitrobenzoyl)-α-arylalkylamine.[6]



CSP 1



CSP 2

Experimental

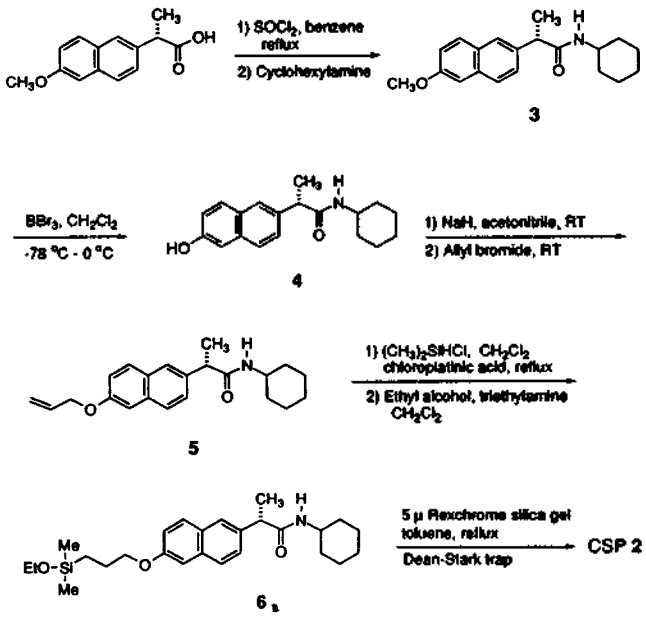
General

^{1}H and ^{13}C NMR spectra were recorded on a Varian Gemini 200 spectrometer. IR spectra were measured on a Mattson Polaris FT IR spectrometer. Melting points were taken on a Rigaku TAS 100 thermal analyzer. Elemental analysis were performed at the OCRC center, Sogang University, Seoul, Korea.

HPLC analyses were performed with an HPLC system consisting of a Waters Model 510 pump, a Rheodyne Model 7125 injector with a 20 μL sample loop, a Youngin Model 710 absorbance detector with a 254 nm UV filter and a Youngin D520B computing integrator. All chromatographic data were collected using 20% isopropyl alcohol in hexane as the mobile phase with a flow rate of 2.0 mL/min at 20 °C. The column void volume was determined by injecting 1,3,5-tri-*tert*-butylbenzene, a presumed unretained solute.[9] Racemic or optically enriched analytes used in this study were available from the previous studies.[6,10]

Preparation of CSP 2

CSP 2 was prepared starting from (S)-naproxen as shown in Scheme 1. All reactions were carried out under an argon



Scheme 1.

atmosphere.

(S)-α-(6-Methoxy-2-naphthyl)propion cyclohexylamide 3. Following the procedure given for the preparation of (S)-α-(6-methoxy-2-naphthyl)propion-3,5-dimethylanilide,[6] reaction of (S)-naproxen (5.00 g, 0.022 mole) with thionylchloride (9.5 mL, 0.132 mole) and subsequent treatment with cyclohexylamine (2.98 mL, 0.035 mole) afforded **3** (5.85 g, 86.5%) as a white solid. The enantiomeric purity of amide **3** was greater than 98% by the HPLC analysis on a commercial CSP derived from (S)-N-(3,5-dinitrobenzoyl)leucine ester.[7] mp 157-159 ℃. ^{1}H NMR ($CDCl_3$) δ 0.98-1.98 (m, 10H), 1.58 (d, 3H), 3.60-3.80 (m, 1H), 3.65 (q, 1H), 3.93 (s, 3H), 5.18 (d, 1H), 7.13-7.74 (m, 6H). ^{13}C NMR ($CDCl_3$) δ 18.45, 24.60, 24.67, 25.29, 32.66, 32.74, 46.75, 48.20, 55.13, 105.52, 118.86, 125.84, 126.14, 127.23, 128.82, 129.06, 133.52, 136.58, 157.52, 173.35. IR (KBr) cm^{-1} 3293, 3060, 2933, 1640, 1609, 1543.

(S)-α-(6-Hydroxy-2-naphthyl)propion cyclohexylamide 4. Following the procedure given for the preparation of (S)-α-(6-hydroxy-2-naphthyl)propion-3,5-dimethylanilide,[6] amide **3** (4.0 g, 0.013 mole) was treated with BBr_3 (25 g, 0.100 mole) to afford hydroxy compound **4** (3.68 g, 96%) as a white solid. The enantiomeric purity of amide **4** was greater than 98% by the HPLC analysis on a commercial CSP derived from (S)-N-(3,5-dinitrobenzoyl)leucine ester.[7] mp 154-155 ℃. ^{1}H NMR ($CDCl_3$) δ 0.95-1.95 (m, 10H), 1.60 (d, 3H), 3.60-3.85 (m, 1H), 3.67 (q, 1H), 5.30 (d, 1H), 6.14 (s, 1H), 7.06-7.70 (m, 6H). ^{13}C NMR ($CDCl_3$) δ 18.36, 24.64, 24.69, 25.37, 32.80, 32.85, 47.09, 48.52, 109.35, 118.70, 125.94, 126.08, 127.12, 128.56, 129.26, 133.91, 135.54, 154.60, 174.31. IR (KBr) cm^{-1} 3300, 2932, 2854, 2360, 1641, 1605, 1540.

(S)-α-(6-Allyloxy-2-naphthyl)propion cyclohexylamide 5. O-Allylation of hydroxy compound **4** (3.58 g, 0.012 mole) was performed by following the procedure given for the preparation of (S)-α-(6-allyloxy-2-naphthyl)propion-3,5-dimethylanilide[6] except the use of NaH (1.15 g, 0.03 mole, 60% dispersion in mineral oil) instead of K_2CO_3 to afford allyloxy compound **5** (3.50 g, 86.3%) as a white solid. The enantiomeric purity of amide **5** was greater than 98% by the HPLC analysis on a commercial CSP derived from (S)-N-(3,5-dinitrobenzoyl)leucine ester.[7] mp 114-117 ℃. ^{1}H NMR ($CDCl_3$) δ 0.85-1.90 (m, 10H), 1.58 (d, 3H), 3.62-3.80 (m, 1H), 3.67 (q, 1H), 4.60-4.70 (m, 2H), 5.28-5.51 (m, 2H), 5.60 (broad s, 1H), 6.02-6.20 (m, 1H), 7.12-7.72 (m, 6H). ^{13}C NMR ($CDCl_3$) δ 18.50, 24.60, 24.67, 25.28, 32.67, 32.75, 46.78, 48.12, 68.65, 106.77, 117.55, 119.09, 125.79, 126.12, 127.22, 128.87, 129.07, 132.96, 133.42, 136.75, 156.40, 173.21. IR (KBr) cm^{-1} 3294, 2933, 2854, 2365, 1641, 1605, 1540.

(S)-α-[6-(3-Ethoxydimethylsilylpropyl)-2-naphthyl] propion cyclohexylamide 6. Hydrosilylation of **6** (3.0 g, 0.003 mole) with dimethylchlorosilane in the presence of catalytic amount (*ca.* 10 mg) of $H_2PtCl_6 \cdot 6H_2O$ was carried out by following the procedure given for the preparation of (S)-α-[6-(3-ethoxydimethylsilylpropyl)-2-naphthyl]propion-3,5-dimethylanilide[6] gave hydrosilylated compound **6** (2.06 g, 52%) as a right brown sticky solid material. The enantiomeric purity of amide **5** was greater than 98% by the HPLC analysis on a commercial CSP derived from (S)-N-(3,5-dinitrobenzoyl)leucine ester.[7] ^{1}H NMR ($CDCl_3$) δ 0.16 (s, 6H), 0.73-0.76 (m, 2H), 0.90-2.00 (m, 12H), 1.20 (t, 3H), 1.57 (d, 3H), 3.59-3.80 (m, 2H), 3.70 (q, 1H), 4.05 (t, 2H), 5.60 (broad s, 1H), 5.27 (d, 1H), 7.11-7.72 (m, 6H). ^{13}C NMR ($CDCl_3$) δ −1.58, 12.99, 19.06, 19.15, 23.67, 25.24, 25.31, 25.97, 33.35, 33.44, 47.54, 48.71, 58.77, 70.90, 107.06, 119.80, 126.46, 126.70, 127.84, 129.42, 129.63, 134.23, 137.23, 157.63, 173.83. IR (KBr) cm^{-1} 3296, 3058, 2933, 2854, 2360, 1641, 1607, 1545.

Bonding of 6 to silica gel and HPLC column packing. Bonding of hydrosilylated compound **6** (1.89 g, 0.004 mole) to silica gel (Spherisorb 5 μm, 4.5 g) was performed according to the procedure described previously.[6] Elemental analysis of the bonded chiral phase (C 4.76%, H 0.64%, N 0.17%) showed a loading of 0.17 mmole of chiral selector per gram of chiral phase based on C or 0.12 mmole of chiral selector per gram of chiral phase based on N. Packing the bonded chiral phase slurried in methanol into a 250 mm×4.6 mm I.D. stainless-steel HPLC column was carried out by using a conventional slurry packing method with an Alltech slurry packer. After washing the HPLC chiral column thus packed with 100 mL of dichloromethane, a solution of 2 mL of hexamethyldisilazane in 50 mL of dichloromethane was eluted through the column to end-cap the residual silanol groups and then additional 50 mL of dichloromethane was eluted to wash out the unreacted hexamethyldisilazane.

Results and Discussion

CSP **2** prepared *via* the procedure shown in Scheme 1 was applied to resolving various π-acidic racemates shown in Figure 1 including those resolved on CSP **1**. Table 1 summarizes the resolution of N-(3,5-dinitrobenzoyl) derivatives of various racemic α-amino esters and amides (**7**). As shown in Table 1, the resolution results are quite excellent. The enantioselectivities exerted by CSP **2** for the two enantiomers of π-acidic derivatives of racemic α-amino esters and amides shown in Table 1 are found to be greater than those exerted by CSP **1** for the two enantiomers of the same π-acidic racemates.[6] These results are exactly consistent with what we expected from the reciprocity conception of chiral recognition. The separation factors for resolving the two enantiomers of N-(3,5-dinitrobenzoyl) derivatives of a homolo-

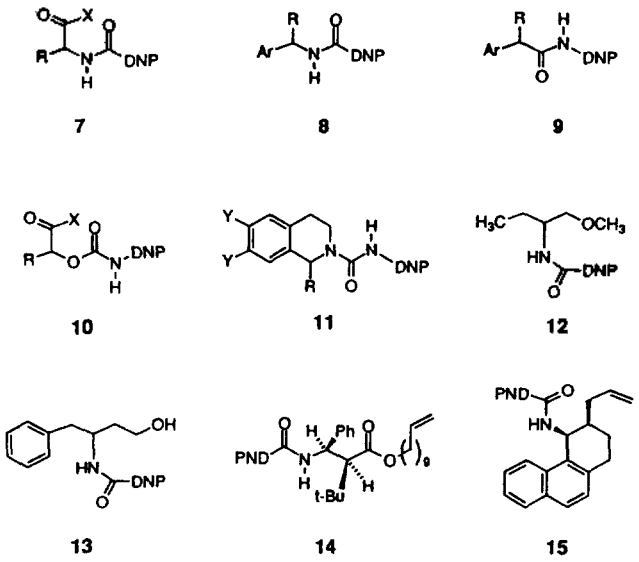


Figure 1. Structures of the racemic compounds resolved on CSP **2**. DNP means 3,5-dinitrophenyl group.

Table 1. Resolution of N-(3,5-dinitrobenzoyl)-α-amino esters and amides (**7**) on CSP **2**.[a]

Anal[b]	R	X	k_1'[c]	k_2'[c]	α[d]	Conf.[e]
7a	CH_3	OCH_2CH_3	4.99	9.62	1.93	R
b	$(CH_2)_2CH_3$	OCH_2CH_3	4.77	13.80	2.89	
c	$(CH_2)_4CH_3$	OCH_2CH_3	4.56	13.58	2.98	
d	$(CH_2)_6CH_3$	OCH_2CH_3	4.25	12.65	2.98	
e	$(CH_2)_7CH_3$	OCH_2CH_3	4.13	12.62	3.06	
f	$(CH_2)_9CH_3$	OCH_2CH_3	4.13	12.56	3.04	
g	$CH(CH_3)_2$	OCH_2CH_3	3.74	9.57	2.56	R
h	$CH_2CH(CH_3)_2$	OCH_3	5.88	20.97	3.57	R
i	$CH_2CH(CH_3)_2$	OCH_2CH_3	4.82	16.57	3.44	R
j	$CH_2CH(CH_3)_2$	$O(CH_2)_2CH_3$	4.44	14.51	3.27	R
k	$CH_2CH(CH_3)_2$	$O(CH_2)_3CH_3$	4.21	13.56	3.22	R
l	$CH_2CH(CH_3)_2$	$O(CH_2)_5CH_3$	3.87	12.52	3.24	R
m	$CH_2CH(CH_3)_2$	$O(CH_2)_7CH_3$	3.60	11.58	3.22	R
n	$CH_2CH(CH_3)_2$	$O(CH_2)_9CH_3$	3.46	10.48	3.03	R
o	$CH_2CH(CH_3)_2$	$O(CH_2)_{11}CH_3$	3.14	9.89	3.15	R
p	$CH_2CH(CH_3)_2$	$O(CH_2)_{13}CH_3$	3.01	9.05	3.01	R
q	$CH_2CH(CH_3)_2$	$O(CH_2)_{15}CH_3$	2.80	8.47	3.03	R
r	Phenyl	OCH_3	7.84	12.12	1.55	R
s	Benzyl	OCH_3	8.83	25.55	2.89	R
t	CH_3	$NH(CH_2)_2CH_3$	1.71	2.05	1.20	R
u	$CH(CH_3)_2$	$NH(CH_2)_2CH_3$	0.75	1.28	1.71	R
v	$CH_2CH(CH_3)_2$	$NH(CH_2)_2CH_3$	0.92	1.69	1.84	R

[a]See the Experimental part for the chromatographic conditions. [b]Racemic analytes. [c]Capacity factors. [d]Separation factors. [e]Absolute configuration of the second eluted enantiomers. For Blanks, the elution orders have not been determined.

gous series of α-alkylglycine ethyl esters (**7a-f**) and leucine alkyl esters (**7b-q**) remain nearly constant as the alkyl chain of analytes increases in length as shown in Table 1. These results are compatible with those on CSP **1** and in consequence the chiral recognition mechanism proposed to be working on CSP **1** might be applicable to CSP **2**.[6] However, the role of the cyclohexyl group of CSP **2** for the greater enantioselectivities on CSP **2** than those on CSP **1** is not clear yet.

Table 2. Resolution of N-(3,5-dinitrobenzoyl)-α-arylalkylamines (**8**) on CSP **2**.[a]

Anal[b]	Ar	R	k_1'[c]	k_2'[c]	α[d]	Conf.[e]
8a	Phenyl	CH_3	11.37	11.37	1.00	
b	Phenyl	CH_2CH_3	12.25	13.78	1.12	
c	Phenyl	$(CH_2)_4CH_3$	11.97	14.33	1.20	
d	Phenyl	$(CH_2)_7CH_3$	10.63	13.43	1.26	
e	Phenyl	$(CH_2)_9CH_3$	9.95	12.94	1.30	
f	Phenyl	$(CH_2)_{12}CH_3$	8.95	12.06	1.35	
g	Phenyl	$(CH_2)_{16}CH_3$	7.87	10.95	1.39	
h	4-CH_3-Phenyl	CH_3	10.18	10.18	1.00	
i	4-$CH_3(CH_2)_2$-Phenyl	CH_3	9.32	9.32	1.00	
j	4-$CH_3(CH_2)_7$-Phenyl	CH_3	7.44	8.03	1.08	
k	4-$CH_3(CH_2)_9$-Phenyl	CH_3	6.80	7.54	1.11	
l	4-$CH_3(CH_2)_{11}$-Phenyl	CH_3	6.26	7.15	1.14	
m	4-Methoxyphenyl	CH_3	13.84	14.82	1.07	R
n	4-Methoxyphenyl	CH_2CH_3	14.01	17.25	1.23	
o	4-Methoxyphenyl	$(CH_2)_2CH_3$	13.94	18.22	1.31	
p	4-Methoxyphenyl	$(CH_2)_7CH_3$	11.80	16.16	1.37	
q	4-Methoxyphenyl	$(CH_2)_{12}CH_3$	10.08	14.75	1.46	
r	1-Naphthyl	CH_3	13.39	15.72	1.17	R

[a]See the Experimental part for the chromatographic conditions. [b]Racemic analytes. [c]Capacity factors. [d]Separation factors. [e]Absolute configuration of the second eluted enantiomers. For Blanks, the elution orders have not been determined.

Resolution of N-(3,5-dinitrobenzoyl) derivatives of various α-arylalkylamines (**8**) on CSP **2** are presented in Table 2. The separation factors shown in Table 2 are worse than those on CSP **1**.[6] In the previous report, the 3,5-dimethylanilide group of CSP **1** was proposed to act as an alternative π-basic interaction site for the π-π interaction with the π-acidic 3,5-dinitrobenzoyl group of analytes **8**.[6] In this context, the worse enantioselectivities observed on CSP **2** for the resolution of the two enantiomers of analytes **8** might stem from the lack of the alternative π-basic interaction site in CSP **2**. Consequently, these results might be considered to be an evidence supporting the proposed chiral recognition mechanism utilizing the 3,5-dimethylanilide group as an alternative π-basic interaction site for the resolution of N-(3,5-dinitrobenzoyl)-α-arylalkylamines **8** on CSP **1**.

CSP **2** was also employed in resolving the two enantiomers of 3,5-dinitroanilide derivatives of α-arylpropionic and α-arylalkanoic acids (**9**), 3,5-dinitrophenylcarbamates of α-hydroxycarboxylic esters (**10**), 3,5-dinitrophenylureides of cyclic amines (**11**) and other π-acidic racemates (**12**, **13**, **14**, **15**). The resolution results for resolving racemates **9-15** on CSP **2** are summarized in Table 3. All resolutions shown in Table 3 are reasonably good. Among others, resolution of 3,5-dinitroanilide derivatives of α-arylpropionic acids (**9**), 3,5-dinitrophenylcarbamates of α-hydroxycarboxylic esters (**10**), 3,5-dinitrophenylureides of cyclic amines (**11**) on CSP **2** are espe-

Table 3. Resolution of other various π-acidic racemates on CSP **2**.[a]

Anal[b]	Ar (or R or Y)	R (or X)	k_1'[c]	k_2'[c]	α[d]	Conf.[e]
9a	Phenyl	CH_2CH_3	4.57	5.10	1.12	
b	Phenyl	$(CH_2)_4CH_3$	4.29	4.90	1.14	
c	Phenyl	$(CH_2)_6CH_3$	4.06	4.67	1.15	
d	Phenyl	$(CH_2)_9CH_3$	3.62	4.23	1.17	
e	4-CH_3Phenyl	CH_3	4.11	4.91	1.19	
f	4-$CH_3(CH_2)_2$Phenyl	CH_3	3.82	4.60	1.20	
g	4-$CH_3(CH_2)_5$Phenyl	CH_3	3.35	3.92	1.17	
h	4-$CH_3(CH_2)_9$Phenyl	CH_3	2.90	3.34	1.15	
i	4-Isobutylphenyl	CH_3	3.54	4.29	1.21	
j	3-Phenoxyphenyl	CH_3	6.73	7.82	1.16	
k	3-Benzoylphenyl	CH_3	5.40	6.50	1.20	
l	6-Methoxy-2-naphthyl	CH_3	6.80	8.66	1.27	S
m	5-Benzoyl-2-thienyl	CH_3	9.49	10.85	1.14	
10a	CH_3	OCH_2CH_3	1.79	2.49	1.39	R
b	CH_2CH_3	OCH_2CH_3	1.55	2.25	1.45	
c	$(CH_2)_3CH_3$	OCH_2CH_3	1.36	1.97	1.45	
d	$(CH_2)_5CH_3$	OCH_2CH_3	1.24	1.85	1.49	
e	$(CH_2)_7CH_3$	OCH_2CH_3	1.12	1.71	1.53	
f	CH_3	$O(CH_2)_2CH_3$	1.55	2.10	1.35	R
g	CH_3	$O(CH_2)_3CH_3$	1.41	1.91	1.35	R
h	CH_3	$O(CH_2)_7CH_3$	1.12	1.46	1.30	R
11a	H	CH_3	5.16	5.70	1.10	
b	H	Benzyl	4.44	6.96	1.57	
c	Methoxy	Phenyl	19.89	27.00	1.36	
d	Methoxy	Benzyl	13.46	20.60	1.53	
e	Methoxy	CH_3	17.75	21.97	1.24	
12			5.54	8.58	1.55	
13			6.55	11.97	1.83	
14			9.34	34.52	3.70	
15			23.54	48.78	2.07	

[a] See the Experimental part for the chromatographic conditions. [b] Racemic analytes. [c] Capacity factors. [d] Separation factors. [e] Absolute configuration of the second eluted enantiomers. For Blanks, the elution orders have not been determined.

cially interesting in that all of these analytes are derivatives of biologically active materials or analogous.

In conclusion, the enantioselectivities exerted by CSP **2** for resolving various π-acidic racemates have been shown to be generally greater than or compatible with those exerted by CSP **1** except for resolving N-(3,5-dinitrobenzoyl)-α-arylalkylamines. The greater enantioselectivities exerted by CSP **2** compared with those exerted by CSP **1** especially for resolving N-(3,5-dinitrobenzoyl)-α-amino esters (**7**) have been consistent with what we expected in connection with the reciprocity conception of chiral recognition. The worse enantioselectivities exerted by CSP **2** compared with those exerted by CSP **1** for resolving N-(3,5-dinitrobenzoyl)-α-arylalkylamines (**8**) have been assumed to be utilized as an evidence supporting the previously proposed chiral recognition mechanism utilizing the 3,5-dimethylanilide group of CSP **1** as an alternative π-basic interaction site. However, the detailed chiral recognition mechanism is not clear yet and needs further studies.

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