

Supporting Information

Enantioselective benzylation of α -amino esters using (*S*)-1-Benzoyl-2-(α -acetoxyethyl)benzimidazole, a chiral benzimidazolide

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1. General

Optical rotations were measured on digital polarimeter. Enantiomeric excesses were determined on HPLC using Chiralcel OD column with UV detector. ^1H NMR spectra were scanned in CDCl_3 on 300, 400 and 500 MHz spectrometers with TMS as an internal standard. Mass spectrum was obtained by using MS in ESI mode. All melting points are uncorrected. Temperatures are recorded in $^\circ\text{C}$. Boiling point of petroleum-ether used was in the range of 60-80 $^\circ\text{C}$.

2. Preparation of (S)-2-(α -Acetoxyethyl)benzimidazole **2**

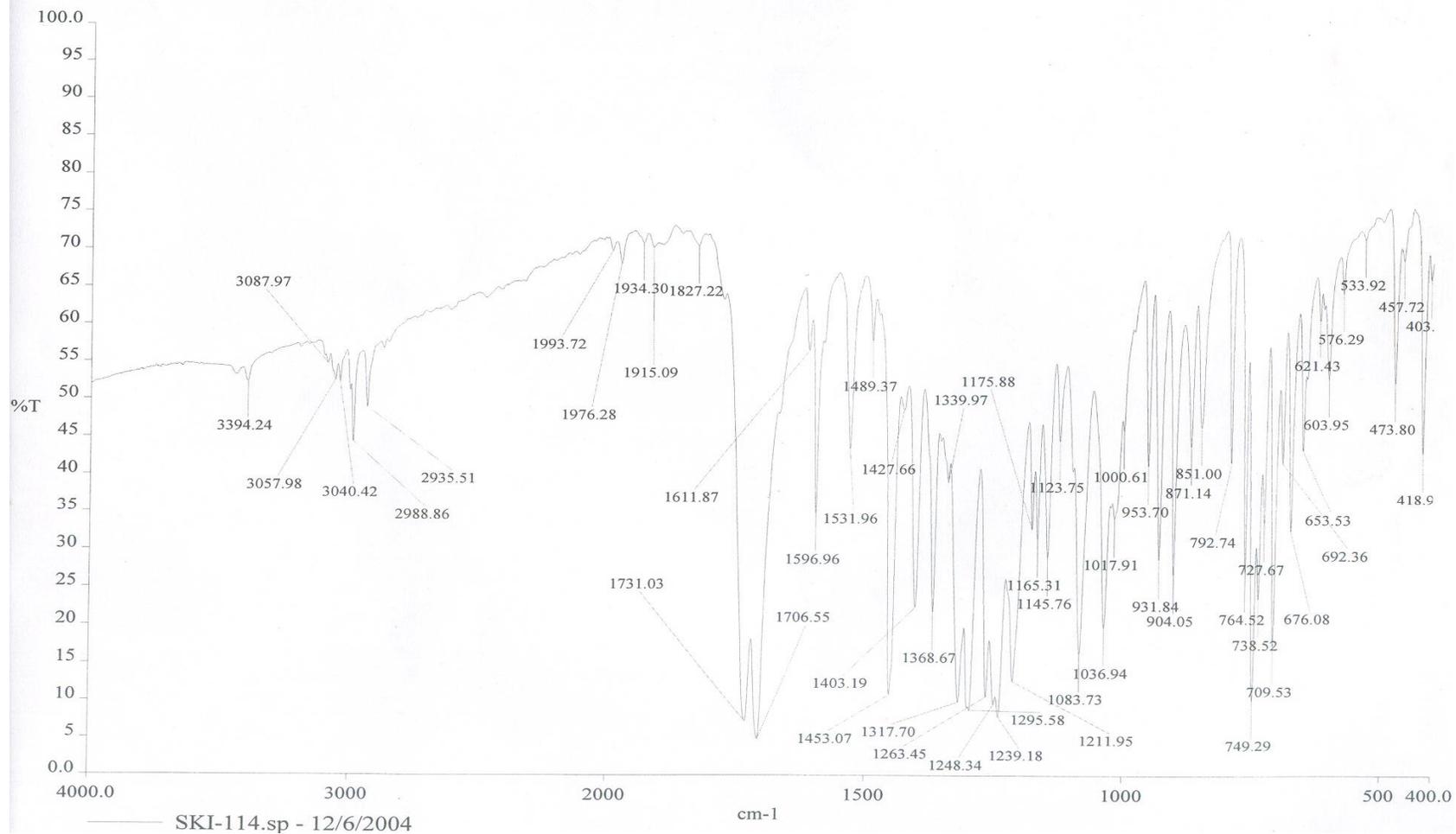
A solution of **1** (4.86 g, 30 mmol) and Ac_2O (3.8 mL, 40 mmol) in 50 mL of dry pyridine was stirred at 0 $^\circ\text{C}$ for 5 h. The reaction mixture was poured into cold water to give white crystalline solid which was filtered and washed with water to give 4.8 g of pure compound **2**. Yield 78%; mp 159-161 $^\circ\text{C}$, lit.¹ mp 153-155 $^\circ\text{C}$ for racemic **2**; $[\alpha]_{\text{D}}^{19} = -156.9$ (c 1, CHCl_3); IR (KBr): 3454, 3053, 2987, 2935, 2831, 2732, 2671, 2631, 1746, 1592, 1541, 1484, 1444 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): $\delta = 9.6$ (s, 1H), 7.80-7.78 (m, 1H), 7.44-7.43 (m, 1H), 7.28-7.26 (m, 2H), 6.08 (q, $J=6.7\text{Hz}$, 1H), 2.14 (s, 3H), 1.85 (d, $J=6.7\text{Hz}$, 3H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 171.25, 152.90, 123.08, 66.91, 31.04, 21.11, 18.88$; Anal. calcd for $\text{C}_{11}\text{H}_{12}\text{N}_2\text{O}_2$: C, 64.71; H, 5.88; N 13.73; found: C, 64.66; H, 5.91; N, 13.76; m/z : 205.89 $[\text{M}+\text{H}]^+$.

1. Gunes, H. S.; Cosar, G. *J. Fac. Pharm. Gazi* **1996**, *13*, 57.

3. Preparation of (S)-1-Benzoyl-2-(α -acetoxyethyl)benzimidazole (BAEB) **3**

Benzoyl chloride (3.5 mL, 30 mmol) was added dropwise to a stirred solution of **2** (4.08 g, 20 mmol) in 40 mL of dry pyridine at 0 °C. The solution was stirred at the same temperature for further 6 h. The reaction mixture was poured onto ice pieces. The solid obtained was filtered and washed with ice-cold water. The crude compound was crystallized using CHCl₃-petroleum ether to give 4.9 g of compound **3** as white crystals. Yield 80%; mp 87-89 °C; $[\alpha]_D^{19} = +167.8$ (c 1, CHCl₃); IR (KBr): 3057, 3040, 2988, 2935, 1731, 1706, 1596, 1531, 1489, 1453 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): $\delta = 7.81$ -7.79 (m, 2H), 7.74-7.68 (m, 1H), 7.56-7.51 (m, 2H), 7.31-7.24 (m, 2H), 7.14-7.08 (m, 1H), 6.67 (d, $J = 8.0$ Hz, 1H), 6.19 (q, $J = 6.6$ Hz, 1H), 2.01 (s, 3H), 1.86 (d, $J = 6.6$ Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): $\delta = 170.47, 168.61, 155.48, 142.17, 134.44, 133.59, 132.99, 130.36, 129.19, 124.45, 124.13, 120.54, 113.41, 66.24, 20.86, 19.02$; Anal. calcd for C₁₈H₁₆N₂O₃: C, 70.13; H, 5.20; N, 9.09; found: C, 70.19; H, 5.16; N, 9.05; m/z: 309.78 [M+H]⁺.

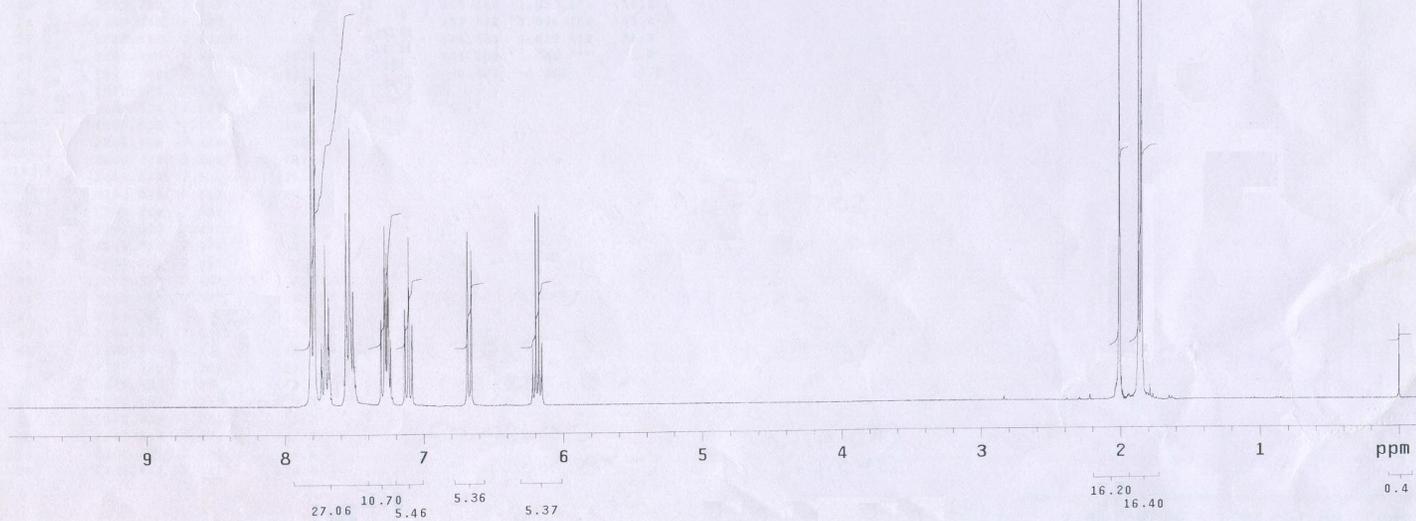
DEPARTMENT OF CHEMISTRY.
MUMBAI UNIVERSITY.



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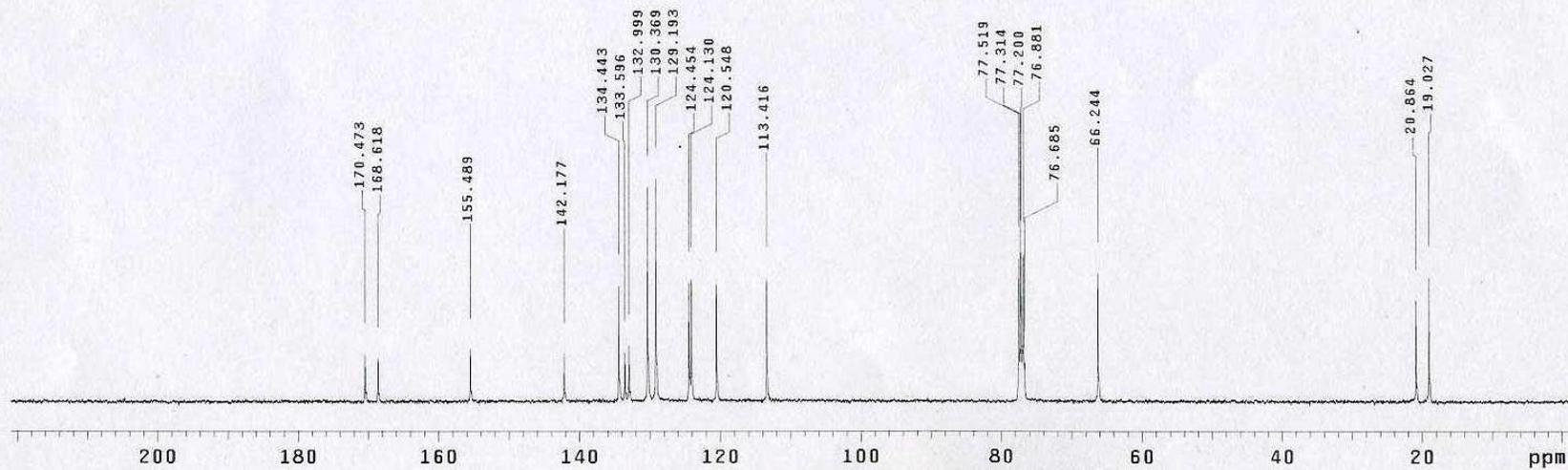
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ct	2016	PROCESSING	
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		th	5
		ai	ph



S7

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SK-1

2/13/2007 2:02:08 PM



5. Representative procedure for enantioselective benzylation of α -amino esters

A solution of **3** (2 mmol) and racemic α -amino ester (4 mmol) in 30 mL of THF was stirred at -10 °C for 12 h. The reaction was quenched with aq HCl and extracted with CHCl₃. The organic extract was dried over anhyd Na₂SO₄ which on evaporation afforded (*S*)-*N*-benzoyl amino ester (**4**). The unreacted α -amino ester was recovered from aqueous layer and on derivatisation with benzoyl chloride in presence of pyridine yielded (*R*)-*N*-benzoyl amino ester (**5**).

6. Spectral characterization of (*S*)-*N*-Benzoyl amino esters

N-Benzoyl-L-alanine ethyl ester (**4a**)

mp 74-75 °C, lit.² mp 77-78 °C; $[\alpha]_{\text{D}}^{19} = +33.4$ (c 1, CHCl₃), lit.² $[\alpha]_{\text{D}}^{20} = +19.9$ (53% ee, c 1, CHCl₃); IR (KBr): 3345, 1750, 1641 cm⁻¹; ¹H NMR (300MHz, CDCl₃): δ 7.81 (d, *J* = 7 Hz, 2H), 7.55-7.41 (m, 3H), 6.78 (d, *J* = 6.2 Hz, 1H), 4.84-4.74 (m, 1H), 4.25 (q, 2H), 1.53 (d, *J* = 7.3 Hz, 3H), 1.32 (t, 3H); Anal. calcd for C₁₂H₁₅NO₃: C, 65.16; H, 6.79; N, 6.33; found: C, 65.23; H, 6.75; N, 6.29.

N-Benzoyl-L-leucine methyl ester (**4b**)

mp 98-99 °C, lit.² mp 94-95 °C; $[\alpha]_{\text{D}}^{19} = +39.9$ (c 1, CHCl₃), lit.² $[\alpha]_{\text{D}}^{20} = +24.8$ (56% ee, c 1, CHCl₃); IR (KBr): 3288, 1747, 1637 cm⁻¹; ¹H NMR (400MHz, CDCl₃): δ 7.81 (d, *J* = 7 Hz, 2H), 7.54-7.50 (m, 1H), 7.47-7.43 (m, 2H), 6.52 (d, *J* = 7.9 Hz, 1H), 4.90-4.85 (m, 1H), 3.78 (s, 3H), 1.80-1.63 (m, 3H), 1.00 (d, *J* = 6.1 Hz, 3H), 0.98 (d, *J* = 6.1 Hz,

2. Liang, J.; Ruble, J. C.; Fu, G. C. *J. Org. Chem.* **1998**, *63*, 3154.

3H); Anal. calcd for $C_{14}H_{19}NO_3$: C, 67.47; H, 7.63; N 5.62; found: C, 67.39; H, 7.67; N, 5.65.

***N*-Benzoyl-L-phenylalanine ethyl ester (4c)**

mp 104-105 °C, lit.³ mp 100-102 °C; $[\alpha]_D^{19} = -39.1$ (c 1, CH_3OH), lit.⁴ $[\alpha]_D^{26} = -42.7$ (c 1, CH_3OH); IR (KBr): 3320, 1743, 1642 cm^{-1} ; 1H NMR (400MHz, $CDCl_3$): δ 7.73 (d, $J = 7$ Hz, 2H), 7.53-7.49 (m, 1H), 7.45-7.41 (m, 2H), 7.32-7.23 (m, 3H), 7.15 (d, $J = 6.1$ Hz, 2H), 6.59 (d, $J = 7.3$ Hz, 1H), 5.10-5.05 (m, 1H), 4.22 (q, 2H), 3.32-3.22 (m, 2H), 1.28 (t, 3H); Anal. calcd for $C_{18}H_{19}NO_3$: C, 72.73; H, 6.40; N 4.71; found: C, 72.77; H, 6.43; N, 4.65.

***N*-Benzoyl-L-valine methyl ester (4d)**

mp 112-113 °C, lit.⁵ mp 110-112 °C; $[\alpha]_D^{19} = +41.3$ (c 0.5, $CHCl_3$), lit.⁵ $[\alpha]_D^{19} = +46.2$ (c 0.65, $CHCl_3$); IR (KBr): 3347, 1739, 1641 cm^{-1} ; 1H NMR (300MHz, $CDCl_3$): δ 7.81 (d, $J = 7$ Hz, 2H), 7.56-7.43 (m, 3H), 6.62 (d, $J = 7.7$ Hz, 1H), 4.80 (dd, $J = 7.7, 4.8$ Hz, 1H), 3.78 (s, 3H), 2.33-2.23 (m, 1H), 1.02 (d, $J = 7$ Hz, 3H), 0.99 (d, $J = 7$ Hz, 3H); Anal. calcd for $C_{13}H_{17}NO_3$: C, 66.38; H, 7.23; N 5.96; found: C, 66.33; H, 7.29; N, 5.91.

3. Bender, M. L.; Turnquest, B. W. *J. Am. Chem. Soc.* **1955**, *77*, 4271.

4. Fryzuk, M. D.; Bosnich, B. *J. Am. Chem. Soc.* **1977**, *99*, 6262.

5. Al-Sehemi, A. G.; Atkinson, R. S.; Fawcett, J. *J. Chem. Soc., Perkin Trans. 1* **2002**, 257.

***N*-Benzoyl-L-phenylglycine methyl ester (4e)**

mp 104-105 °C, lit.² mp 102-103 °C; $[\alpha]_D^{19} = +107.9$ (c 0.5, CHCl₃), lit.⁶ $[\alpha]_D^{19} = +117.6$ (c 0.5, CHCl₃); IR (KBr): 3283, 1752, 1629 cm⁻¹; ¹H NMR (400MHz, CDCl₃): δ 7.83 (d, *J* = 7.3 Hz, 2H), 7.54-7.50 (m, 1H), 7.46-7.33 (m, 7H), 7.15 (d, *J* = 6.1 Hz, 1H), 5.78 (d, *J* = 6.1 Hz, 1H), 3.78 (s, 3H); Anal. calcd for C₁₆H₁₅NO₃: C, 71.38; H, 5.58; N 5.20; found: C, 71.45; H, 5.55; N, 5.15.

7. Chiral HPLC analysis of *N*-Benzoyl amino esters

Racemic *N*-Benzoyl amino esters were prepared as reference for chiral HPLC analysis of the products obtained in enantioselective benzoylation of α -amino esters following literature procedure.³⁻⁶

6. Dondoni, A.; Perrone, D.; Semola, T. *Synthesis* **1995**, 181.

N-Benzoyl-DL-alanine ethyl ester**Area % Report**

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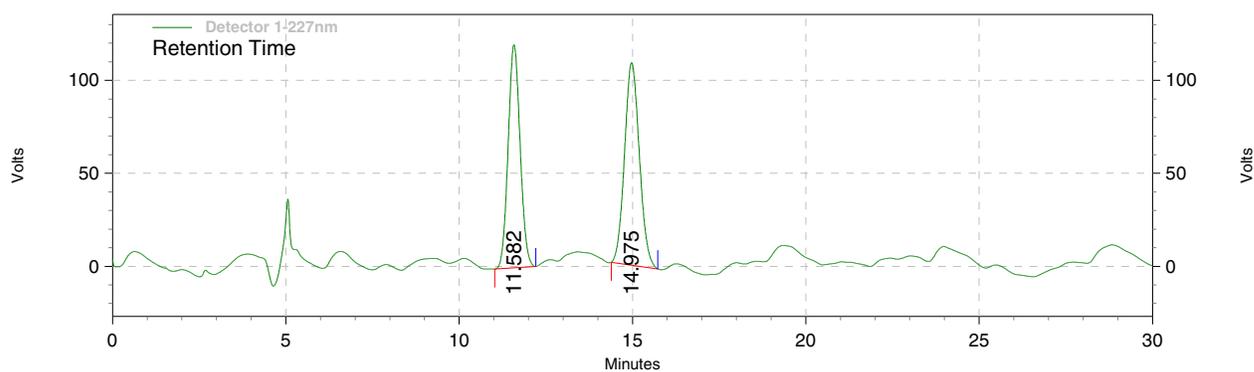
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14.975	3241021	52.83	108912	47.56

Totals	6134806	100.00	229006	100.00
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N-Benzoyl-L-alanine ethyl ester (Table 1, compound 4a)**Area % Report**

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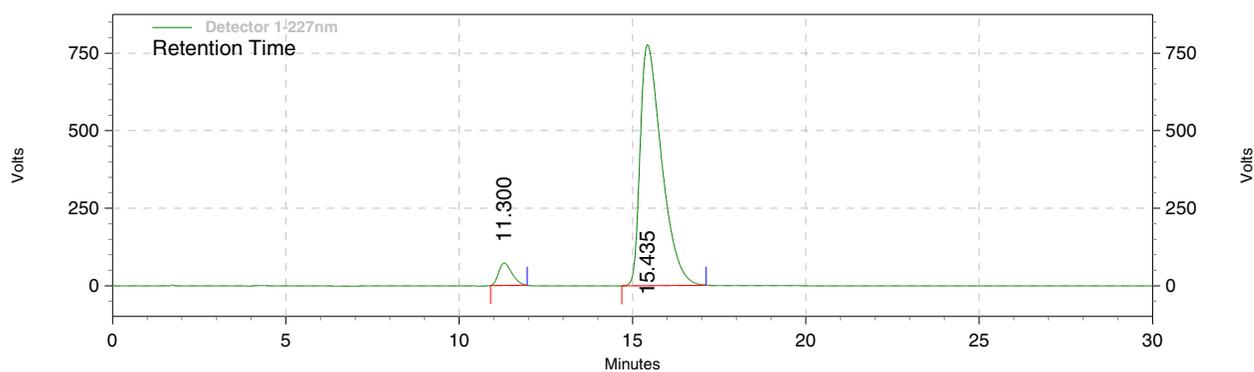
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Flow rate: 2.0 ml/ min

Acquired: 12/21/2006 2:47:36 PM

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**Detector 227nm****Results**

Retention Time	Area	Area %	Height	Height %
11.300	1924675	5.44	72060	8.48
15.435	33454537	94.56	777250	91.52

Totals	35379212	100.00	849310	100.00
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N-Benzoyl-D-alanine ethyl ester (Table 1, compound 5a)**Area % Report**

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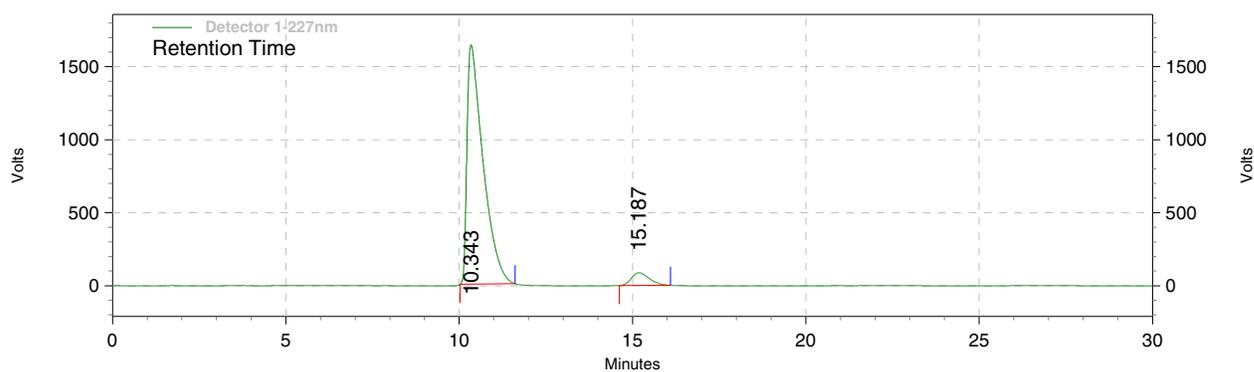
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**Detector 227nm****Results**

Retention Time	Area	Area %	Height	Height %
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15.187	3116753	5.56	86481	5.00

Totals	56046421	100.00	1729011	100.00
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N-Benzoyl-DL-leucine methyl ester**Area % Report**

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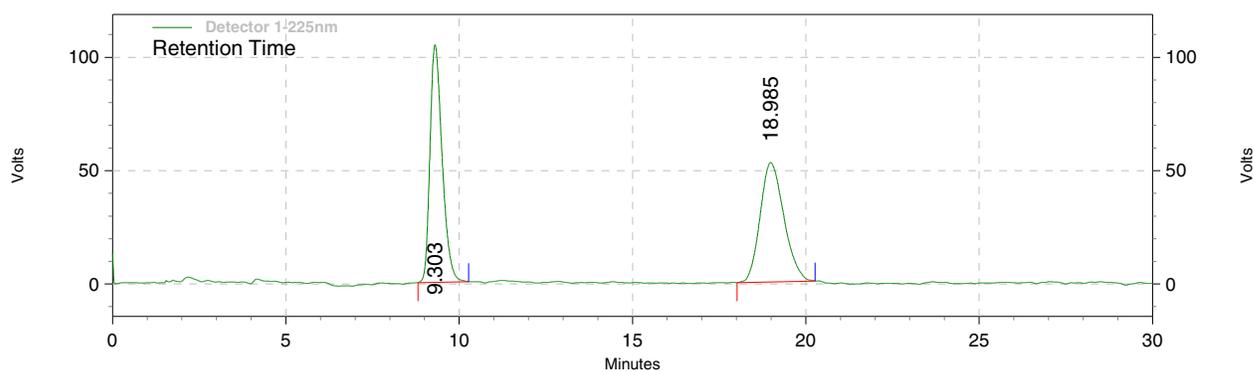
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
9.303	2548983	50.37	104815	66.55
18.985	2511455	49.63	52676	33.45

Totals	5060438	100.00	157491	100.00
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N-Benzoyl-L-leucine methyl ester (Table 1, compound 4b)**Area % Report**

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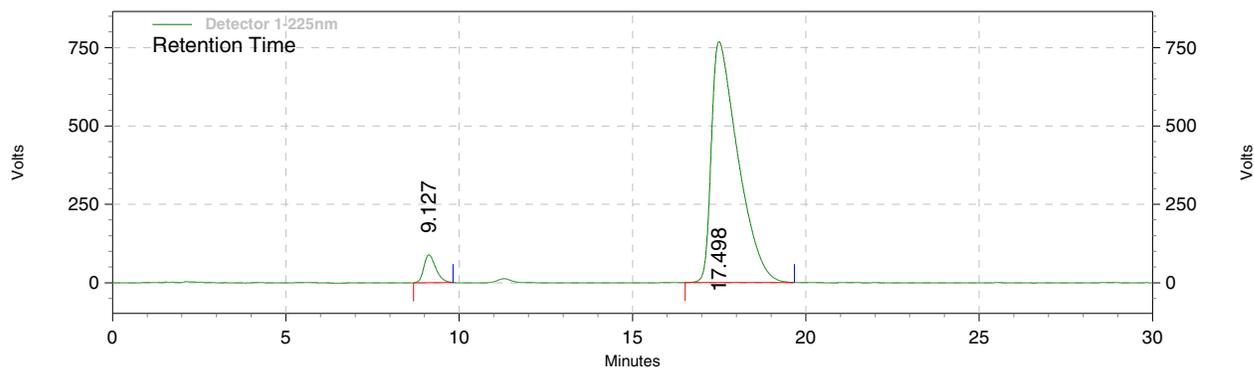
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

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Printed: 12/22/2006 4:19:16 PM

**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
9.127	2106906	4.90	88670	10.34
17.498	40919369	95.10	768759	89.66

Totals	43026275	100.00	857429	100.00
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N-Benzoyl-D-leucine methyl ester (Table 1, compound 5b)**Area % Report**

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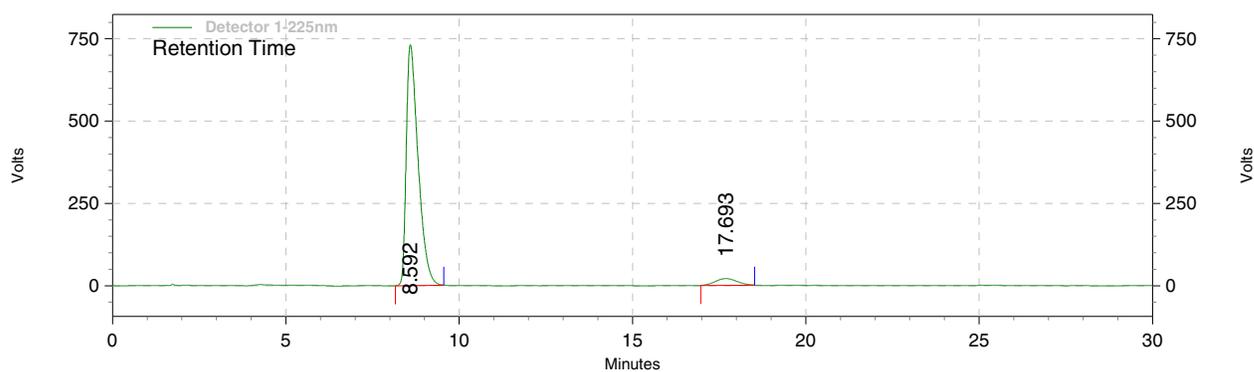
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Flow rate: 2.0 ml/ min

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
8.592	17340654	95.21	731832	97.30
17.693	872737	4.79	20302	2.70

Totals	18213391	100.00	752134	100.00
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N-Benzoyl-DL-phenylalanine ethyl ester**Area % Report**

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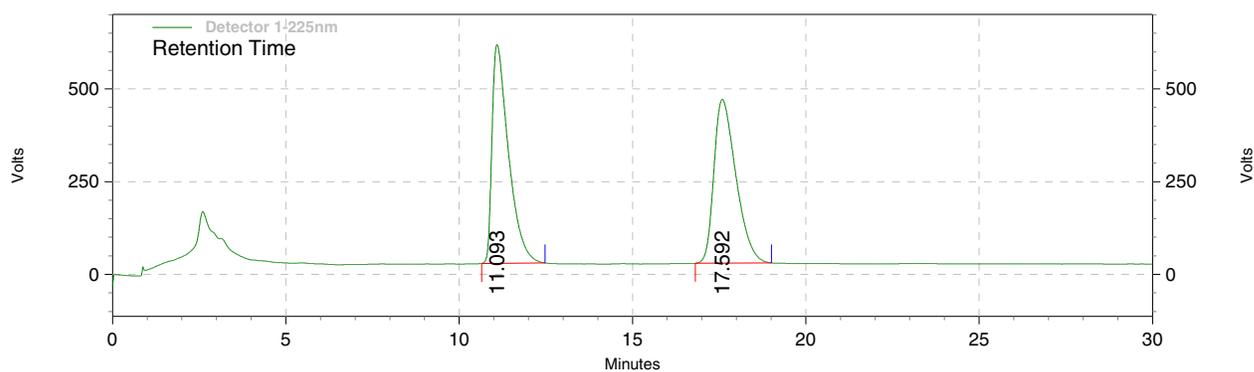
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

Acquired: 12/26/2006 11:47:21 AM

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
11.093	19453508	49.94	589626	57.18
17.592	19500438	50.06	441613	42.82

Totals	Area	Area %	Height	Height %
	38953946	100.00	1031239	100.00

N-Benzoyl-L-phenylalanine ethyl ester (Table 1, compound 4c)**Area % Report**

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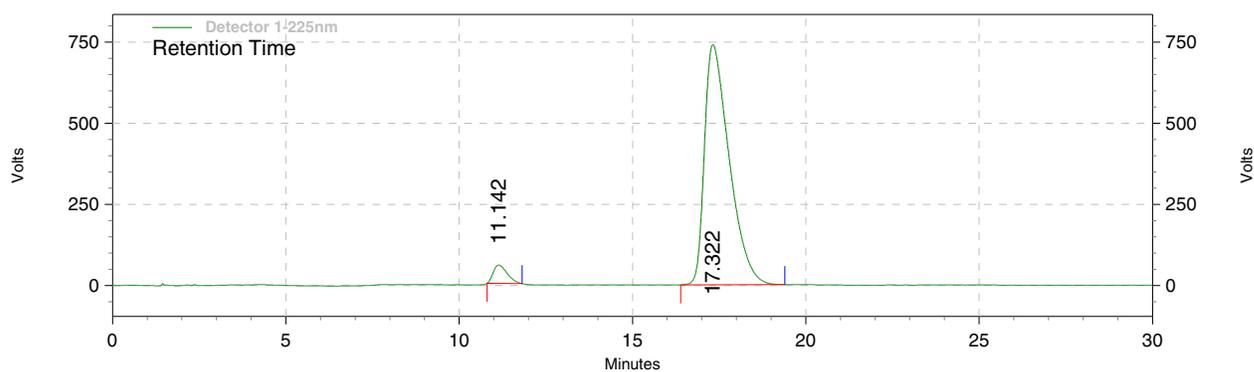
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
11.142	1574794	4.11	56459	7.09
17.322	36721410	95.89	740304	92.91

Totals	38296204	100.00	796763	100.00
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N-Benzoyl-D-phenylalanine ethyl ester (Table 1, compound 5c)**Area % Report**

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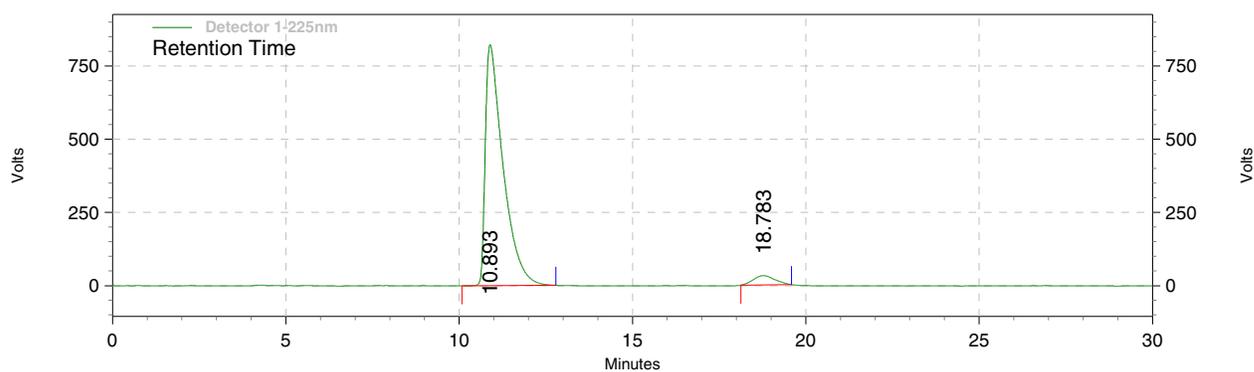
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
10.893	29280345	95.54	823298	96.29
18.783	1368305	4.46	31715	3.71

Totals	30648650	100.00	855013	100.00
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N-Benzoyl-DL-valine methyl ester**Area % Report**

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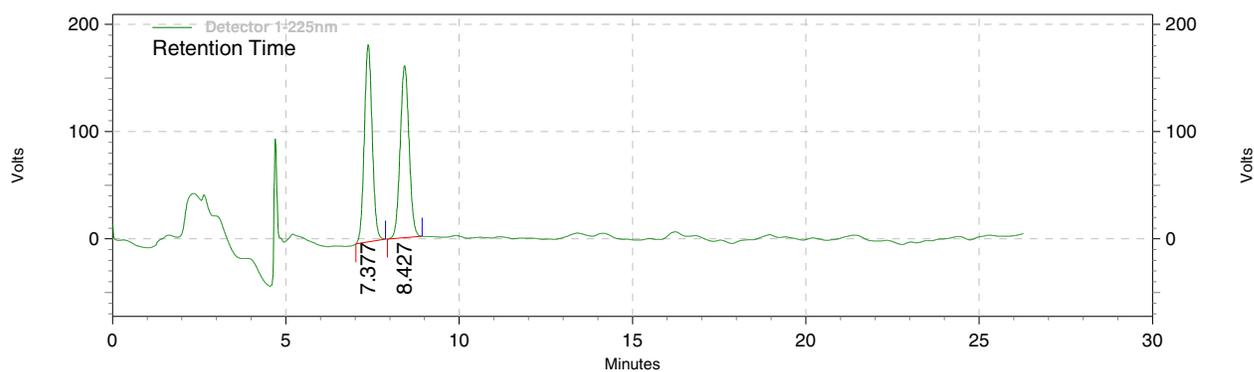
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Flow rate: 2.0 ml/ min

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
7.377	2905064	50.40	183824	53.40
8.427	2858624	49.60	160399	46.60

Totals	5763688	100.00	344223	100.00
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N-Benzoyl-L-valine methyl ester (Table 1, compound 4d)**Area % Report**

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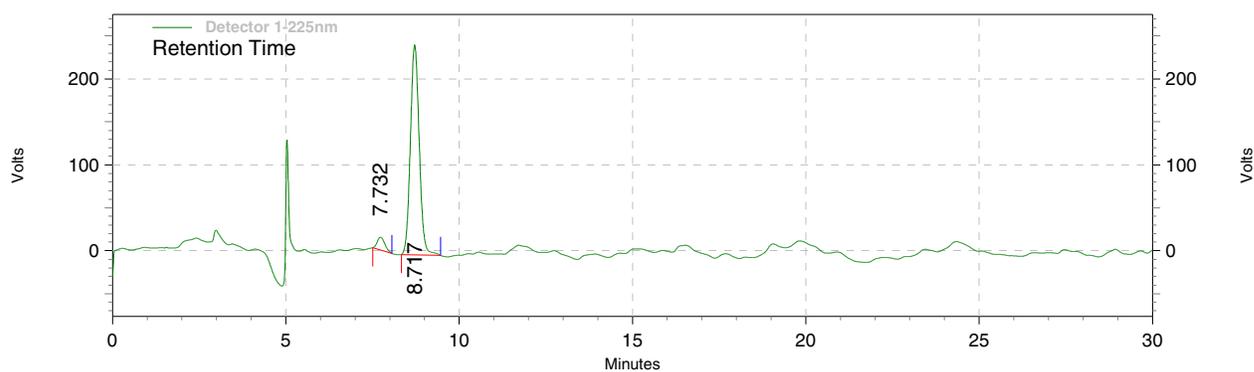
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

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**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
7.732	230133	5.03	15156	5.83
8.717	4341233	94.97	244748	94.17

Totals	Area	Area %	Height	Height %
	4571366	100.00	259904	100.00

N-Benzoyl-D-valine methyl ester (Table 1, compound 5d)**Area % Report**

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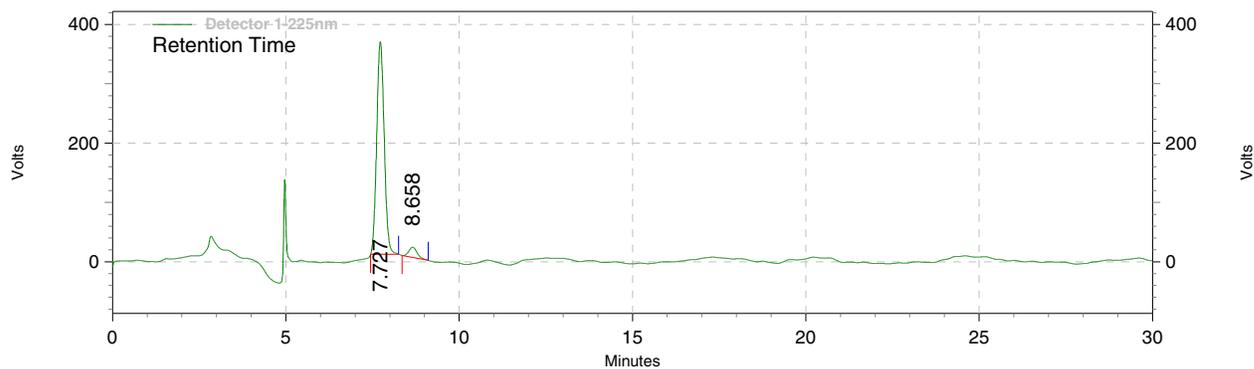
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Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

Acquired: 12/20/2006 2:06:44 PM

Printed: 12/28/2006 12:38:47 PM

**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
7.727	5388076	94.99	358646	95.42
8.658	283970	5.01	17216	4.58

Totals	5672046	100.00	375862	100.00
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N-Benzoyl-DL-phenylglycine methyl ester

Area % Report

Data File: C:\ChromQuest\MUMBAI ORG\ssk-13.dat

Method: C:\ChromQuest\Enterprise\Projects\Default\Method\untitled.met

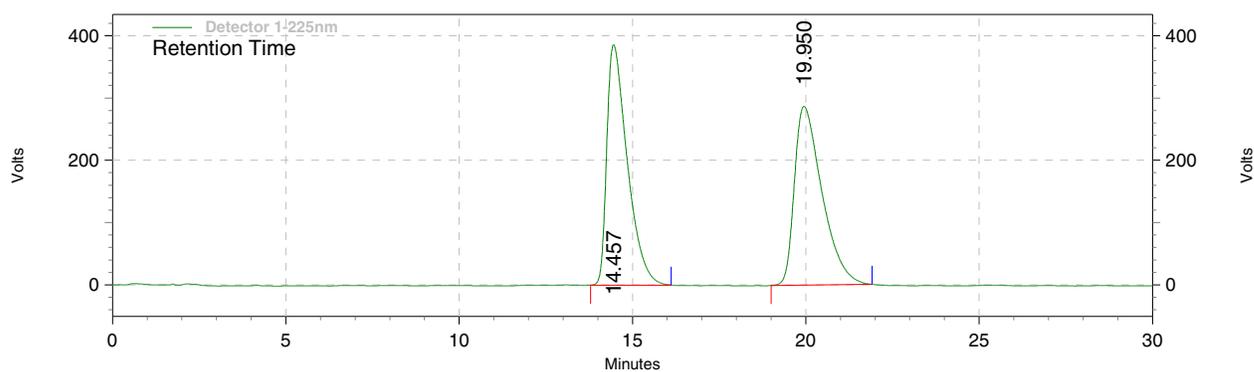
Column: Chiralcel OD

Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

Acquired: 12/27/2006 12:43:42 PM

Printed: 12/28/2006 12:53:16 PM



Detector 225nm

Results

Retention Time	Area	Area %	Height	Height %
14.457	15853266	49.95	386268	57.36
19.950	15882290	50.05	287104	42.64

Totals	31735556	100.00	673372	100.00
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N-Benzoyl-L-phenylglycine methyl ester (Table 1, compound 4e)**Area % Report**

Data File: C:\ChromQuest\MUMBAI ORG\ssk-14.dat

Method: C:\ChromQuest\Enterprise\Projects\Default\Method\untitled.met

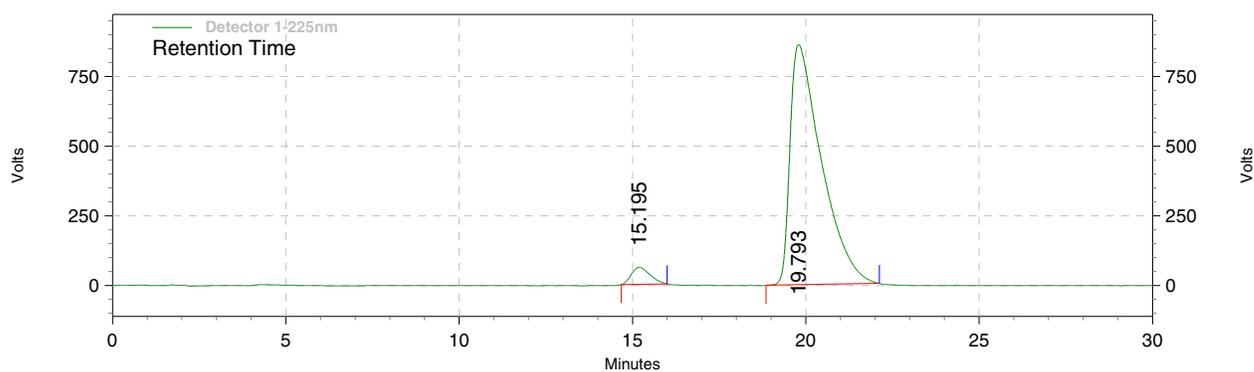
Column: Chiralcel OD

Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

Acquired: 12/28/2006 12:09:31 PM

Printed: 12/28/2006 2:29:34 PM

**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
15.195	2297482	4.06	61482	6.65
19.793	54289176	95.94	862593	93.35

Totals	56586658	100.00	924075	100.00
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N-Benzoyl-D-phenylglycine methyl ester (Table 1, compound 5e)**Area % Report**

Data File: C:\ChromQuest\MUMBAI ORG\ssk-15.dat

Method: C:\ChromQuest\Enterprise\Projects\Default\Method\untitled.met

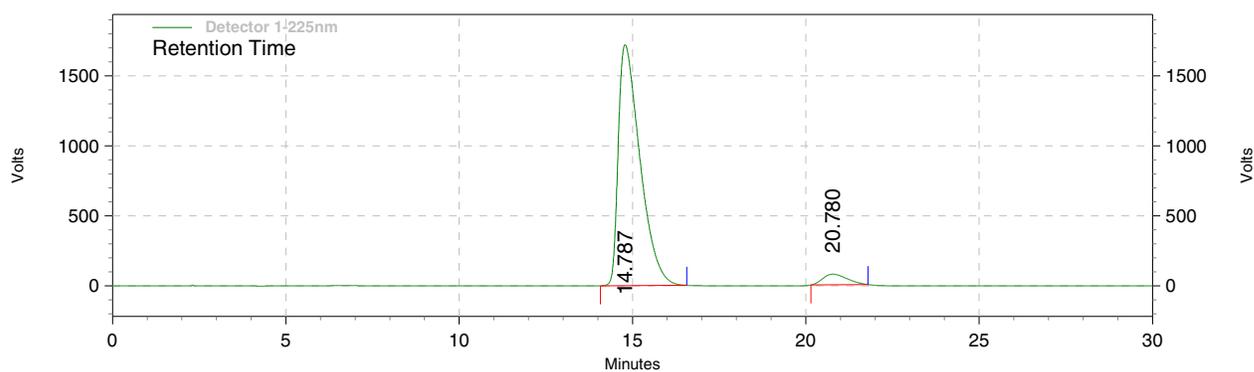
Column: Chiralcel OD

Mobile phase: n-Hexane/ 2-Propanol 98/ 2 (v/ v)

Flow rate: 2.0 ml/ min

Acquired: 12/28/2006 12:47:45 PM

Printed: 12/28/2006 2:33:02 PM

**Detector 225nm****Results**

Retention Time	Area	Area %	Height	Height %
14.787	74700568	95.30	1722169	95.76
20.780	3685417	4.70	76306	4.24

Totals	78385985	100.00	1798475	100.00
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