Supporting Information

Organocatalytic direct Mannich/cyclization cascade as [3 + 2] annulations: Asymmetric synthesis of 2, 3-substituted pyrrolidines

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Table of Contents

General Experimental Methods:

All reactions under standard conditions were monitored by thin-layer chromatography (TLC) on SiO_2 gel F254 plates. The column chromatography was performed on silica gel (100-200 meshes) using EtOAc and petroleum ether with the distillation range of 60-80 ^oC. All other reagents were of analytical grade and used without further purification. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ solution and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard. High resolution mass spectra were recorded using quadrupole electrospray ionization (ESI) technique. HPLC was performed on Thermo Finnigan instrument using chiral Pack OD-H column and *i*-PrOH/Hexane solvent system.

General procedure for the organocatalytic Mannich/intramolecular cyclization /aromatization reaction cascade:

Succinaldehyde **3** (0.3 mL, 0.9 mmol, 3M solution) was added to a mixture of preformed *N*-PMP aldimine **2** (0.3 mmol) and L-proline (7.0 mg, 0.06 mmol) in DMSO (3.0 mL) at room temperature. The reaction mixture was stirred at 5 ^oC until the aldimine was consumed as monitored by TLC. The reaction was worked up by addition of saturated $NaHCO_3$ solution (3 mL) and extracted with ethyl acetate with three times. The combined organic extracts were washed with brine one time, dried over anhydrous Na_2SO_4 and concentrated in vacuum after filtration.

The crude adduct was taken in MeOH (3 mL) and CH_3CO_2H (50 mol%, 9 μL) and then $NaBH_4$ was cautiously at 0 ^oC and further stirred for 3 h and allows it come to room temperature. The reaction was subsequently quenched with saturated $NaHCO_3$ solution (3 mL). The aqueous solution was extracted with ethyl acetate twice and combined organic extracts were washed with brine once and dried over anhydrous Na_2SO_4 and concentrated in vacuum after filtration. Purification by silica gel column chromatography (hexane: EtOAc) gave *trans*-2,3-disubstituted pyrrolidines **6** with 56-78% yields.

The enantiomeric excess (ee) of products was determined by HPLC analysis using chiral OD-H column and absolute configuration could not be established by the single crystal X-ray of **6b**. The model chosen for refinement has C2-S, and C3-S stereochemistry, as expected through the well documented *syn*-selective direct Mannich reaction catalyzed by L-proline.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(2-nitrophenyl)pyrrolidin-3-yl)methanol (6a)

6a: ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.03-2.14 (m, 2H), 2.31-2.36 (m, 1H), 3.49-3.53 (m, 1H), 3.56-3.61 (m, 1H), 3.66-3.86 (m, 1H), 3.70 (s, 3H), 3.88-3.92 (m, 1H), 5.08 (d, *J* = 3.0 Hz, 1H), 6.37 (d, *J* = 8.8 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.50 (t, *J* = 7.7 Hz, 2H), 8.02 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.59, 47.39, 50.14, 55.74, 62.21, 63.80, 112.71 (2C), 114.91 (2C), 124.84, 128.25, 129.58, 134.00, 139.60, 140.68, 147.85, 151.18;

HRMS (ESI): Calcd for $C_{18}H_{20}N_2O_4$ (MH^+) 329.1501, Found 329.1497.

$[\alpha]_D^{22}$ = + 18.6 (*c* 1.0, $CHCl_3$, 98% ee)

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 85:15), 1.0 mL/min; minor enantiomer t_R = 11.347 min, major enantiomer t_R = 14.273 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(3-nitrophenyl)pyrrolidin-3-yl)methanol (6b)

6b: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.88-1.95 (m, 1H), 2.13-2.23 (m, 1H), 2.30-2.37 (m, 1H), 3.45 (dd, *J* = 16.3 Hz, 8.8 Hz, 1H), 3.69 (m, 2H), 3.71 (s, 3H), 3.79 (dt, *J* = 8.5 Hz, 3.2 Hz, , 1H), 4.63 (d, *J* = 2.7 Hz, 1H), 6.41 (d, *J* = 9.0 Hz, 2H), 6.75 (d, *J* = 9.1 Hz, 2H), 7.47 (t, *J* = 8.1 Hz, 2H), 7.60 (d, *J* = 7.8 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 8.14 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.49, 48.56, 51.49, 55.77, 63.84, 65.21, 113.43 (2C), 114.86(2C), 121.02, 121.95, 129.54, 132.27, 141.39, 147.11, 148.68, 151.39;

HRMS (ESI): Calcd for $C_{18}H_{20}N_2O_4$ (MH^+) 329.1501, Found 329.1512.

$[\alpha]_D^{22}$ = - 50.9 (*c* 1.0, $CHCl_3$, 92% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 70:30), 1.0 mL/min; minor enantiomer t_R = 7.223 min, major enantiomer t_R = 10.713 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(4-nitrophenyl)pyrrolidin-3-yl)methanol (6c)

6c: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.88-1.94 (m, 1H), 2.11-2.21 (m, 1H), 2.31-2.37 (m, 1H), 3.46 (dd, *J* = 16.3 Hz, 8.8 Hz, 1H), 3.68 (m, 2H), 3.70 (s, 3H), 3.75 (dt, *J* = 8.8 Hz, 3.3 Hz, 1H), 4.63 (d, *J* = 2.8 Hz, 1H), 6.38 (d, *J* = 9.1 Hz, 2H), 6.74 (d, *J* = 9.0 Hz, 2H), 7.42 (d, *J* = 8.8 Hz, 2H), 8.14 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.51, 48.43, 51.37, 55.75, 63.79, 65.22, 113.29 (2C), 114.84 (2C), 123.88 (2C), 126.86 (2C), 141.27, 146.84, 151.28, 152.52;

HRMS (ESI): Calcd for $C_{18}H_{20}N_2O_4$ (MH^+) 329.1501, Found 329.1505.

$[\alpha]_D^{22}$ = - 79.5 (*c* 1.0, $CHCl_3$, 96% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 70:30), 1.0 mL/min; minor enantiomer t_R = 8.060 min, major enantiomer t_R = 11.660 min.

((2*S*, 3*S*)- 2-(2-chlorophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6d)

6d: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.01-2.12 (m, 1H), 2.37-2.39 (m, 1H), 3.51 (dd, *J* = 16.5 Hz, 9.2 Hz, 1H), 3.61-3.67 (m, 1H), 3.70 (s, 3H), 3.72-3.78 (m, 1H), 3.84-3.89 (m, 1H), 4.74 (d, *J* = 2.4 Hz, 1H), 6.35 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 7.16-7.21 (m, 3H), 7.38 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.88, 47.72, 49.57, 55.82, 63.08, 64.20, 112.87 (2C), 114.91 (2C), 127.05, 127.55, 128.14, 129.75, 132.20, 140.75, 141.16, 151.07;

HRMS (ESI): Calcd for $C_{18}H_{20}ClNO_2$ (MH^+) 318.1261, Found: 318.1259.

$[\alpha]_D^{22}$ = +11.6 (*c* 1.0, $CHCl_3$, >99% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.0 mL/min; minor enantiomer t_R = 9.726 min, major enantiomer t_R = 14.433 min.

((2*S*, 3*S*)- 2-(3-chlorophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6e)

6e: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.84-1.89 (m, 1H), 2.14-2.21 (m, 1H), 2.28-2.37 (m, 1H), 3.40 (dd, 16.5 Hz, *J* = 8.6 Hz, 1H), 3.60-3.65 (m, 2H), 3.70 (s, 4H), 4.47 (d, *J* = 2.1 Hz, 1H), 6.40 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.8 Hz, 2H), 7.12-7.25 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.43, 48.32, 51.45, 55.79, 63.96, 65.29, 113.23 (2C), 114.79 (2C), 124.14, 126.03, 126.90, 129.85, 134.47, 141.65, 146.80, 151.07;

HRMS (ESI): Calcd for $C_{18}H_{20}ClNO_2$ (MH^+) 318.1261, Found: 318.1265.

$[\alpha]_D^{22}$ = - 33.5 (*c* 1.0, $CHCl_3$, 92% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 92:08), 1.3 mL/min; minor enantiomer t_R = 13.105 min, major enantiomer t_R = 33.857 min.

((2*S*, 3*S*)- 2-(4-chlorophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6f)

6f: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.84-1.91 (m, 1H), 2.11-2.20 (m, 1H), 2.26-2.33 (m, 1H), 3.42 (dd, *J* = 16.3 Hz, 8.8 Hz, 1H), 3.61-3.67 (m, 2H), 3.68-3.72 (m, 1H) 3.70 (s, 3H), 4.47 (d, *J* = 2.7 Hz, 1H), 6.38 (d, *J* = 9.1 Hz, 2H), 6.73 (d, *J* = 9.1 Hz, 2H), 7.17 (d, *J* = 8.6 Hz, 2H), 7.24 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.53, 48.38, 51.58, 55.82, 64.12, 65.10, 113.28 (2C), 114.82 (2C), 127.39 (2C), 128.73 (2C), 132.30, 141.65, 142.90, 151.12;

HRMS (ESI): Calcd for $C_{18}H_{20}ClNO_2$ (MH^+) 318.1261, Found: 318.1273.

$[\alpha]_D^{22}$ = - 49.0 (*c* 1.0, $CHCl_3$, 90% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 92:08), 1.3 mL/min; minor enantiomer t_R = 12.338 min, major enantiomer t_R = 29.488 min.

((2*S*, 3*S*)- 2-(2-fluorophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6g)

6g: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.96-2.00 (m, 1H), 2.10-2.18 (m, 1H), 2.36-2.39 (m, 1H), 3.44-3.47 (m, 1H), 3.62-3.67 (m, 2H), 3.70 (s, 3H), 3.73-3.78 (m, 1H), 4.76 (d, *J* = 2.4 Hz, 1H), 6.41 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 7.01 (t, *J* = 8.0 Hz, 1H), 7.06 (t, *J* = 8.0 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.43, 47.89, 50.31, 55.82, 59.55, 64.21, 113.02 (2C), 114.86 (2C), 124.21, 126.26, 127.64, 128.27, 130.80, 133.00, 141.46, 151.10;

HRMS (ESI): Calcd for $C_{18}H_{20}FNO_2$ (MH^+) 302.1556, Found 302.1548.

$[\alpha]_D^{22}$ = - 11.5 (*c* 0.5, $CHCl_3$, 93% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.0 mL/min; minor enantiomer t_R = 9.290 min, major enantiomer t_R = 18.738 min.

((2*S*, 3*S*)- 2-(4-fluorophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6h)

6h: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.84-1.89 (m, 1H), 2.12-2.19 (m, 1H), 2.26-2.32 (m, 1H), 3.42 (dd, *J* = 16.5 Hz, 8.8 Hz, 1H), 3.60-3.68 (m, 2H), 3.70 (s, 4H), 4.48 (d, *J* = 2.8 Hz, 1H), 6.41 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.98 (t, *J* = 8.5 Hz, 2H), 7.21 (dd, *J* = 5.5 Hz, 8.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.45, 48.30, 51.58, 55.78, 64.02, 64.96, 113.21 (2C), 114.77 (2C), 115.20, 115.41, 127.42, 139.87, 141.71, 150.95, 160.42, 160.85;

HRMS (ESI): Calcd for $C_{18}H_{20}FNO_2$ (MH^+) 302.1556, Found 302.1562.

$[\alpha]_D^{22}$ = - 37.9 (*c* 1.0, $CHCl_3$, 91% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.0 mL/min; minor enantiomer t_R = 11.775 min, major enantiomer t_R = 28.242 min.

((2*S*, 3*S*)- 2-(4-bromophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6i)

6i: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.83-1.90 (m, 1H), 2.10-2.19 (m, 1H), 2.26-2.31 (m, 1H), 3.43 (dd, *J* = 16.3 Hz, 8.6 Hz, 1H), 3.60-3.67 (m, 2H), 3.70 (s, 4H), 4.46 (d, *J* = 2.2 Hz, 1H), 6.40 (d, *J* = 9.1 Hz, 2H), 6.75 (d, *J* = 9.1 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 7.40 (dd, *J* = 5.5 Hz, 8.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.53, 48.39, 51.54, 55.83, 64.10, 65.15, 113.29 (2C), 114.84 (2C), 127.80 (2C), 128.59, 131.66 (2C), 141.63, 143.46, 151.13;

HRMS (ESI): Calcd for $C_{18}H_{20}BrNO_2$ (MH^+) 362.0755, Found 362.0759.

$[\alpha]_D^{22}$ = - 43.2 (*c* 1.0, $CHCl_3$, 90% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 92:08), 1.3 mL/min; minor enantiomer t_R = 12.542 min, major enantiomer t_R = 33.592 min.

((2*S*, 3*S*)- 2-(3-bromo-4-fluorophenyl)-1-(4-methoxyphenyl)pyrrolidin-3-yl)methanol (6j)

6j: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.83-1.88 (m, 1H), 2.10-2.17 (m, 1H), 2.25-2.28 (m, 1H), 3.40 (dd, *J* = 18.1 Hz, 9.5 Hz, 1H), 3.61 (d, *J* = 7.3 Hz, 2H), 3.70 (s, 4H), 4.46 (bs, 1H), 6.40 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 7.03 (t, *J* = 8.5 Hz, 1H), 7.14-7.17 (m, 1H), 7.45 (dd, *J* = 6.5 Hz, 2.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.27, 48.23, 51.37, 55.73, 63.40, 64.58, 113.21 (2C), 114.76 (2C), 126.29, 127.25, 130.68, 131.72, 141.49, 150.99, 156.45, 158.89;

HRMS (ESI): Calcd for $C_{18}H_{19}BrFNO_2$ (MH^+) 380.0661, Found 380.0667.

$[\alpha]_D^{22}$ = - 19.2 (*c* 1.0, $CHCl_3$, 92% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 92:08), 1.3 mL/min; minor enantiomer t_R = 12.560 min, major enantiomer t_R = 33.893 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-phenylpyrrolidin-3-yl)methanol (6k)

6k: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.85-1.90 (m, 1H), 2.18-2.24 (m, 1H), 2.31-2.37 (m, 1H), 3.41-3.48 (dd, *J* = 17.0 Hz, 8.8 Hz, 1H), 3.62-3.73 (m, 6H), 4.49 (d, *J* = 2.8 Hz, 1H), 6.41 (d, *J* = 8.8 Hz, 2H), 6.72 (d, *J* = 8.8 Hz, 2H), 7.18-7.31 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.56, 48.34, 51.62, 55.83, 64.31, 65.65, 113.18 (2C), 114.79 (2C), 125.94 (2C), 126.70, 128.57 (2C), 141.87, 144.28, 150.89;

HRMS (ESI): Calcd for $C_{18}H_{21}NO_2$ (MH^+) 284.1650, Found: 284.1660.

$[\alpha]_D^{22}$ = - 41.2 (*c* 1.0, $CHCl_3$, 90% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.0 mL/min; minor enantiomer t_R = 13.180 min, major enantiomer t_R = 29.432 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(naphthalen-1-yl)pyrrolidin-3-yl)methanol (6l)

6l: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.88-1.93 (m, 1H), 2.13-2.23 (m, 1H), 2.48-2.53 (m, 1H), 3.523.59 (dd, *J* = 16.1 Hz, 9.1 Hz, 1H), 3.68 (s, 3H), 3.71-3.86 (m, 3H), 5.39 (bs, 1H), 6.38 (d, *J* = 8.8 Hz, 2H), 6.71 (d, *J* = 8.8 Hz, 2H), 7.24 (d, *J* = 6.8 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.50-7.60 (m, 2H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.89 (d, *J* = 8.6 Hz, 1H), 8.40 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.19, 47.23, 49.10, 55.85, 62.39, 64.49, 112.64 (2C), 114.93 (2C), 123.24, 123.61, 125.53, 125.95, 127.40, 128.82, 128.92, 130.65, 134.16, 138.18, 141.40, 150.75;

HRMS (ESI): Calcd for $C_{22}H_{23}NO_2$ (MH^+): 334.1807, Found 334.1826.

$[\alpha]_D^{22}$ = +7.3 (*c* 1.0, $CHCl_3$, 97% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.0 mL/min; minor enantiomer t_R = 15.240 min, major enantiomer t_R = 27.638 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(naphthalen-2-yl)pyrrolidin-3-yl)methanol (6m)

6m: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.89-1.94 (m, 1H), 2.21-2.26 (m, 1H), 2.40-2.44 (m, 1H), 3.46 (dd, *J* = 16.5 Hz, 8.5 Hz, 1H), 3.68 (bs, 4H), 3.71-3.77 (m, 2H), 4.64 (d, *J* = 2.2 Hz, 1H), 6.47 (d, *J* = 8.8 Hz, 2H), 6.71 (d, *J* = 8.8 Hz, 2H), 7.40-7.46 (m, 3H), 7.75-3.81 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ

25.63, 48.51, 51.49, 55.82, 64.28, 65.96, 113.31 (2C), 114.81 (2C), 124.38, 124.49, 125.44, 126.00, 127.64, 127.78, 128.31, 128.49, 132.67, 133.48, 141.99, 150.98;

HRMS (ESI): Calcd for $C_{22}H_{23}NO_2$ (MH^+): 334.1807, Found 334.1815.

$[\alpha]_D^{22}$ = - 19.6 (*c* 0.5, $CHCl_3$, 90% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.0 mL/min; minor enantiomer t_R = 18.290 min, major enantiomer t_R = 37.092 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(pyridin-2-yl)pyrrolidin-3-yl)methanol (6n)

6n: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.77-1.85 (m, 1H), 2.06-2.14 (m, 1H), 2.47-2.54 (m, 1H), 3.56 (dd, *J* = 16.3 Hz, 8.4 Hz, 1H), 3.71 (bs, 4H), 3.78-3.80 (m, 2H), 4.64 (d, *J* = 5.5 Hz, 1H), 6.40 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.8 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 2H), 7.57-3.81 (dt, *J* = 7.8 Hz, 1.4 Hz, 1H), 8.55 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.49, 48.95, 49.80, 55.73, 64.91, 68.01, 113.46 (2C), 114.75 (2C), 120.24, 121.89, 137.31, 141.66, 148.83, 151.14, 163.33;

HRMS (ESI): Calcd for $C_{17}H_{20}N_2O_2$ (MH^+) 285.1603, Found: 285.1635.

$[\alpha]_D^{22}$ = - 37.7 (*c* 1.0, $CHCl_3$, 95% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 80:20), 1.0 mL/min; major enantiomer t_R = 10.297 min, minor enantiomer t_R = 14.238 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(pyridin-4-yl)pyrrolidin-3-yl)methanol (6o)

6o: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.26-2.34 (m, 1H), 2.37-2.43 (m, 1H), 2.96-2.98 (m, 1H), 3.33 (dd, *J* = 16.1 Hz, 9.6 Hz 1H), 3.71 (s, 4H), 3.74-3.82 (m, 2H), 5.02 (d, *J* = 2.2 Hz, 1H), 6.40 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 7.21 (d, *J* = 5.9 Hz, 2H), 8.53 (d, *J* = 4.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 23.88, 29.64, 48.15, 55.71, 59.88, 61.60, 113.58 (2C), 114.87 (2C), 121.28 (2C), 140.65, 149.99 (2C), 151.83, 152.70;

HRMS (ESI): Calcd for $C_{17}H_{20}N_2O_2$ (MH^+) 285.1603, Found: 285.1608.

$[\alpha]_D^{22}$ = +7.2 (*c* 1.0, $CHCl_3$, 98% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 80:20), 1.0 mL/min; minor enantiomer t_R = 12.457 min, major enantiomer t_R = 16.562 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(thiophen-2-yl)pyrrolidin-3-yl)methanol (6p)

6p: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.87-1.94 (m, 1H), 2.25-2.34 (m, 1H), 2.42-2.48 (m, 1H), 3.36 (dd, *J* = 16.5 Hz, 8.3 Hz 1H), 3.62-3.68 (m, 3H), 3.71 (s, 3H), 4.76 (d, *J* = 2.2 Hz, 1H), 6.53 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.91-6.93 (m, 2H), 7.12 (dd, *J* = 1.7 Hz, 4.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 2 432, 33.76, 45.13, 56.78, 59.46, 62.76, 113.58, 114.87, 124.54, 124.88, 130.24, 135.04, 142.08, 151.76;

HRMS (ESI): Calcd for $C_{16}H_{19}NO_2S$ (MH^+) 290.1212, Found 390.1225.

$[\alpha]_D^{22}$ = - 15.8 (*c* 1.0, $CHCl_3$, 95% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 92:08), 1.3 mL/min; minor enantiomer t_R = 14.488 min, major enantiomer t_R = 32.368 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(5-nitrofuran-2-yl)pyrrolidin-3-yl)methanol (6q)

6q: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.94-2.00 (m, 1H), 2.16-2.23 (m, 1H), 2.60-2.66 (m, 1H), 3.38 (dd, *J* = 16.1 Hz, 8.5 Hz, 1H), 3.56-3.68 (m, 3H), 3.72 (s, 3H), 4.70 (bs, 1H), 6.29 (d, *J* = 3.8 Hz, 1H), 6.48 (d, *J* = 8.8 Hz, 2H), 6.78 (d, *J* = 8.8 Hz, 2H), 7.19 (d, *J* = 3.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.74, 47.52, 48.04, 55.77, 59.49, 63.62, 109.90, 112.79, 113.36 (2C), 114.92 (2C), 140.91, 151.61, 151.81, 160.91;

HRMS (ESI): Calcd for $C_{16}H_{18}N_2O_4$ (MH^+) 319.1294, Found 334.1296.

$[\alpha]_D^{22}$ = - 58.8 (*c* 0.5, $CHCl_3$, 96% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 85:15), 1.3 mL/min; minor enantiomer t_R = 19.330 min, major enantiomer t_R = 26.727 min.

((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-((*E*)-styryl)pyrrolidin-3-yl)methanol (6r)

6r: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.84-1.90 (m, 1H), 2.18-2.24 (m, 1H), 2.32-2.36 (m, 1H), 3.34 (dd, *J* = 16.5 Hz, 8.3 Hz, 1H), 3.52-3.68 (m, 3H), 3.73 (s, 3H), 4.10 (bs, 1H), 6.20 (dd, *J* = 15.9 Hz, 5.8 Hz, 1H), 6.48 (d, *J* = 15.9 Hz, 1H), 6.59 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 7.26-7.37 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 25.65, 48.10, 55.75, 60.32, 63.42, 63.77, 113.17 (2C), 114.67 (2C), 126.22 (2C), 127.16, 128.37 (2C), 129.69, 131.61, 136.77, 142.20, 150.81;

HRMS (ESI): Calcd for $C_{20}H_{23}NO_2$ (MH^+) 310.1807, Found 310.1819.

$[\alpha]_D^{22}$ = - 54.3 (*c* 1.0, $CHCl_3$, 95% ee);

Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (*n*-Hexane: *i*-PrOH = 90:10), 1.3 mL/min; minor enantiomer t_R = 13.307 min, major enantiomer t_R = 27.058 min.

Single Crystal X-ray data for ((2*S*, 3*S*)-1-(4-methoxyphenyl)-2-(3-nitrophenyl)pyrrolidin-3-yl)methanol (**6b**): [(CCDC no: **864411**)]

The title compound, ((2S, 3S)-1-(4-methoxyphenyl)-2-(3-nitrophenyl)pyrrolidin-3-yl)methanol ($C_{18}H_{20}N_2O_4$), crystallizes in the monoclinic space group $P2_1$ with the following unit-cell parameters: a= 7.2761(4), b=7.6312(5), c = 14.6614(10)Å, $\beta = 95.224(5)^{o}$ and Z = 2. The crystal structure was solved by direct methods and refined by full-matrix least-squares procedures to a final R-value of 0.0467 for 2266 observed reflections. The pyrrolidine ring is in envelope conformation and makes a dihedral angle of $11.42(8)^{o}$ with the phenyl ring (C6-C11). Phenyl ring (C15-C20) is almost perpendicular to the pyrrolidine ring (dihedral angle $88.78(8)^{o}$). The molecules in the unit cell are arranged in layers and are stabilized by O-H…O, C-H…O hydrogen bonds and C-H…π interactions.

The X-ray intensity data of a well defined crystal (0.30 x 0.20 x 0.10 mm) were collected at room temperature (293K) by using a CCD area-detector diffractometer (*X'calibur system – Oxford diffraction make, U.K.*) which is equipped with graphite monochromated MoKα radiation (λ=0.71073 Å). The cell dimensions were determined by least-squares fit of angular settings of 4103 reflections in the θ range 3.7698 to 29.0804^{o}**.** A total number of 15385 reflections were collected of which 2266 reflections were treated as observed ($I > 2\sigma(I)$). Data were corrected for Lorentz and polarization and absorption factors.

The structure was solved by direct methods using SHELXS97 (Sheldrick, 2008). All non-hydrogen atoms of the molecule were located from the E-map. Full-matrix least-squares refinement was carried out by using SHELXL97 software (Sheldrick, 2008). All the hydrogen atoms were located from a difference electron density map and their positional and isotropic thermal parameters were included in the refinement. The final refinement cycle yielded an R-factor of 0.0467 (wR (F^2) = 0.0920) for the observed data. The residual electron density ranges from -0.145 to 0.189 $eÅ^{-3}$. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in Table 1. The CIF for this structure has been deposited at Cambridge Crystal Data Centre (**CCDC no: 864411**).

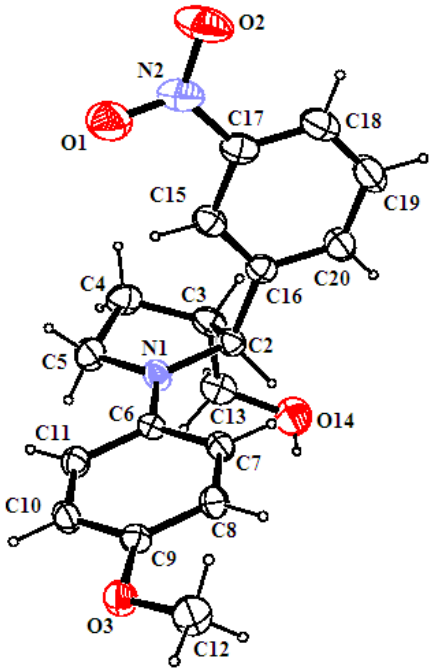


Fig. 1: *ORTEP* view of the molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

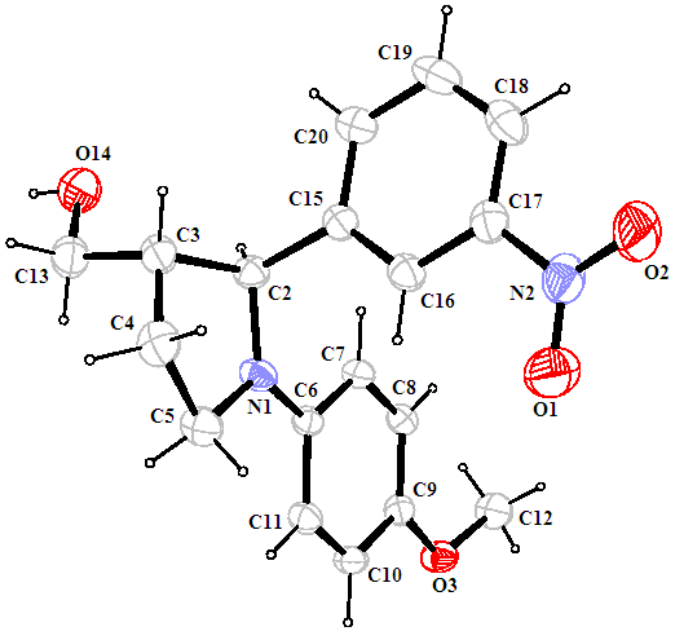


Fig. 2: *ORTEP* view of the molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

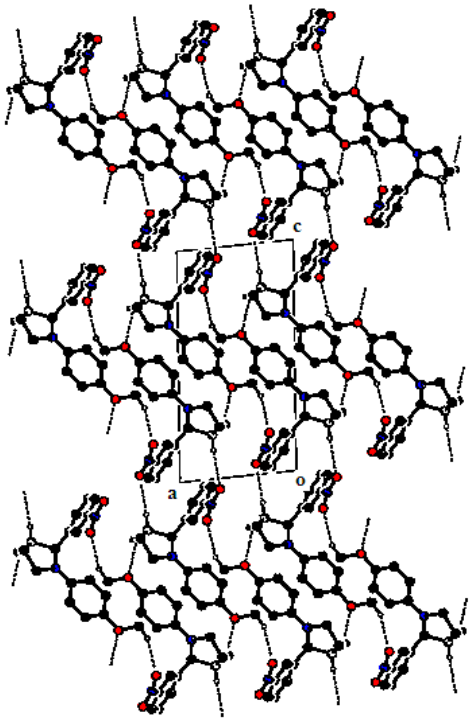


Fig. 3 The packing arrangement of molecules viewed down the b-axis.

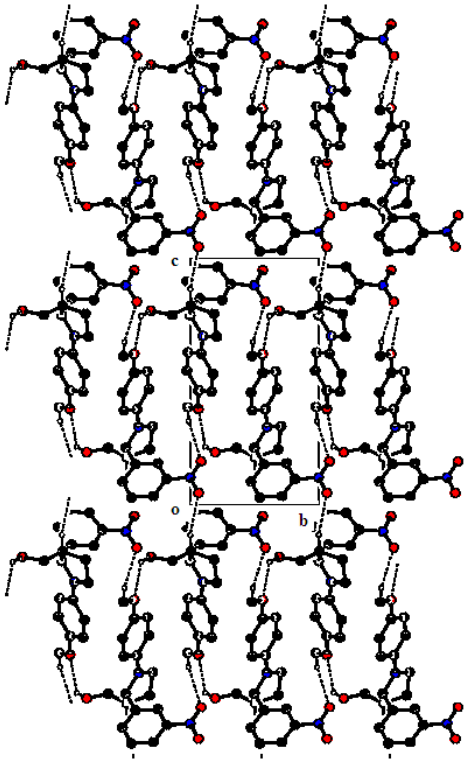


Fig. 4 The packing arrangement of molecules viewed down the a-axis.

Crystal data and other experimental details are given in Table 1. The final atomic coordinates with equivalent isotropic displacement parameters are presented in Table 2. An ORTEP view of the title compound with atomic labeling at different rotations is shown in Fig.1, and Fig.2 (Farrugia, 1997). Bond distances and angles in ((2S, 3S)-1-(4-methoxyphenyl)-2-(3-nitrophenyl)pyrrolidin-3-yl)methanol are in agreement with the theoretical values (Allen, et al., 1987). The pyrrolidine ring is in envelope conformation and makes a dihedral angle of 11.42(8)^{o} with the phenyl ring (C6-C11). Phenyl ring (C15-C20) is almost perpendicular to the pyrrolidine ring (dihedral angle 88.78(8)^{o}). Pyrrolidine has ***envelope*** conformation with the best mirror plane passing through C3 bisecting the opposing bond (N1-C5). The asymmetry parameter is**:** $\mathbf{\Delta C_s}$(C3) = 5.6.

Table 1. Crystal data and other experimental details

CCDC	864411
Crystal description	Red coloured block shaped
Crystal size	0.30 x 0.20 x 0.10 mm
Empirical formula	C_{18} H_{20} N_2 O_4
Formula weight	328.36
Measurement	*X'calibur system–Oxford diffraction make ,U.K.*
Radiation, Wavelength	MoKα, 0.71073 Å
Cell measurement Temperature	293(2) K
Unit cell dimensions	a= 7.2761(4), b= 7.6312(5)
	c = 14.6614(10)Å; β= 95.224(5)º
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell volume	810.70(9)Å^3
Density (calculated)	1.345Mgm^{-3}
No. of molecules per unit cell, Z	2

Absorption coefficient	0.096 mm^{-1}
F(000)	348
Refinement of unit cell	4103 reflections for 3.7698< θ< 29.0804 ^{o}
Scan mode	w scans
θ range for entire data collection	3.78 < θ < 26.00 ^{o}
Range of indices	*h* = -8 to 8, *k* =-9 to 9, *l* = -18 to 18
Reflections collected / unique	15385 / 3157
Reflections observed (I > 2σ(I))	2266
R_{int}	0.0568
R_{sigma}	0.0510
Structure determination	Direct methods
Refinement	Full-matrix least-squares on F^2
No. of parameters refined	218
Final R- factor	0.0467
wR(F^2)	0.0920
Goodness-of-fit	1.041
Final residual electron density	-0.145 < Δρ < 0.189 e$Å^{-3}$
Software for structure solution:	SHELXS97 (Sheldrick, 2008)
Software for refinement:	SHELXL97 (Sheldrick, 2008)
Software for molecular plotting:	ORTEP-3 (Farrugia,1997)
	PLATON (Spek, 2003)
Software for geometrical calculations	PLATON (Spek, 2003)
	PARST (Nardelli, 1995)

Table 2. Atomic coordinates and equivalent isotropic thermal parameters ($Å^2$) for non-hydrogen atoms (e.s.d.'s are given in parenthesis)

Atom	x	y	z	U_{eq}*
N1	1.0787(2)	0.0975(3)	0.68520(13)	0.0424(5)
N2	0.7352(3)	0.4990(4)	0.89068(19)	0.0657(7)
O1	0.7742(4)	0.5841(3)	0.82457(18)	0.0891(8)
O2	0.6562(4)	0.5637(4)	0.95255(17)	0.1014(9)
O3	0.5440(2)	0.0632(2)	0.38814(12)	0.0471(5)
C2	1.0778(3)	-0.0012(4)	0.76919(15)	0.0403(6).
C3	1.2822(3)	0.0047(4)	0.80865(17)	0.0480(7)
C4	1.3448(4)	0.1843(4)	0.77611(19)	0.0547(7)
C5	1.2409(3)	0.2070(4)	0.68191(18)	0.0515(7)
C6	0.9425(3)	0.0850(3)	0.61276(16)	0.0337(5)
C7	0.7851(3)	-0.0171(3)	0.61785(16)	0.0379(6)
C8	0.6508(3)	-0.0272(3)	0.54415(16)	0.0395(6)
C9	0.6699(3)	0.0638(3)	0.46454(17)	0.0370(6)
C10	0.8273(3)	0.1650(3)	0.45874(17)	0.0410(6)
C11	0.9602(3)	0.1765(3)	0.53124(16)	0.0397(6)
C12	0.3709(3)	-0.0208(4)	0.39648(19)	0.0562(8)
C13	1.3947(4)	-0.1399(4)	0.7705(2)	0.0570(8)
O14	1.3184(3)	-0.3062(3)	0.78774(14)	0.0715(6)
C15	0.9513(3)	0.0747(4)	0.83694(17)	0.0407(6)
C16	0.8968(3)	0.2480(4)	0.83184(18)	0.0455(7)
C17	0.7884(3)	0.3143(4)	0.89726(19)	0.0490(7)
C18	0.7304(4)	0.2131(5)	0.9660(2)	0.0598(8)
C19	0.7860(4)	0.0413(5)	0.9714(2)	0.0660(9)
C20	0.8957(3)	-0.0275(4)	0.90732(18)	0.0523(7)

Table 3. Bond lengths (Å), Bond angles and torsion angles for non hydrogen atoms (e.s.d.'s are given in parentheses)

N1- C6	1.388(3)	N1 -C2	1.444(3)
N1 -C5	1.450(3)	N2 -O1	1.221(3)
N2 -O2	1.222(3)	N2 -C17	1.463(4)
O3 -C9	1.381(3)	O3 -C12	1.429(3)
C2 -C15	1.528(3)	C2 -C3	1.546(3)
C3 -C13	1.511(4)	C3 -C4	1.535(4)
C4 -C5	1.523(4)	C6 -C7	1.393(3)
C6 -C11	1.400(3)	C7 -C8	1.391(3)
C8 -C9	1.376(3)	C9 -C10	1.390(3)
C10- C11	1.373(3)	C13 -O14	1.418(4)
C15 -C16	1.381(4)	C15 -C20	1.383(4)
C16 -C17	1.391(3)	C17 -C18	1.367(4)
C18 -C19	1.373(4)	C19 -C20	1.389(4)

Bond angles

C6- N1- C2	123.94(19)	C6 -N1 -C5	122.7(2)
C2 -N1 -C5	113.31(19)	O1 -N2 -O2	122.2(3)
O1 -N2 -C17	119.1(3)	O2 -N2 -C17	118.7(3)
C9 -O3 -C12	117.20(18)	N1 -C2 -C15	114.0(2)
N1 -C2 -C3	103.00(18)	C15- C2 -C3	111.17(19)
C13 -C3 -C4	110.5(2)	C13 -C3 -C2	112.0(2)
C4 -C3 -C2	102.3(2)	C5 -C4 -C3	104.2(2)
N1 -C5 -C4	104.1(2)	N1 -C6 -C7	122.0(2)
N1 -C6 -C11	120.3(2)	C7 -C6 -C11	117.7(2)
C8 -C7 -C6	120.8(2)	C9 -C8 -C7	120.9(2)
C8 -C9 -O3	125.1(2)	C8 -C9 -C10	118.7(2)

O3 -C9 -C10	116.2(2)	C11- C10- C9	120.9(2)
C10 -C11 -C6	121.0(2)	O14 -C13 -C3	110.7(2)
C16 -C15 -C20	118.6(2)	C16 -C15 -C2	121.0(2)
C20 -C15 -C2	120.4(2)	C15 -C16 -C17	119.3(3)
C18 -C17 -C16	122.4(3)	C18 -C17 -N2	119.7(3)
C16 -C17 -N2	117.9(3)	C17 -C18 -C19	118.2(3)
C18 -C19 -C20	120.5(3)	C15 -C20 -C19	121.1(3)

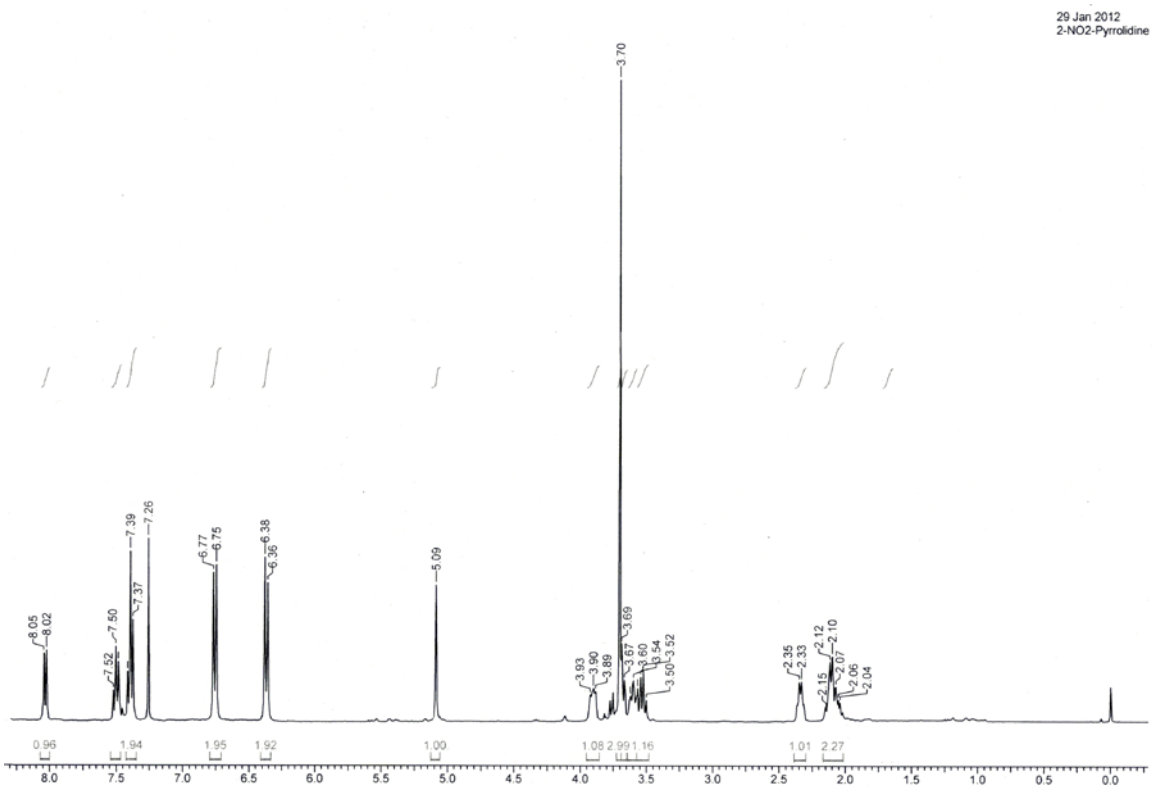
Torsion angles

C5- N1 -C2 -C3	-18.0(3)	N1- C2- C3- C4	32.4(2)
C2 -C3 -C4 -C5	-35.7(2)	C2- N1- C5- C4	-4.4(3)
C3 -C4- C5 -N1	25.2(3)		

Table 3 Geometry of Intra and inter molecular hydrogen bonds

D–H…A	D–H(Å)	H…A(Å)	D…A(Å)	θ [D–H…A(°)]
C3-H3...O2^{i}	0.9800	2.559(2)	3.518(4)	165.9(2)
C12-H12A...O1^{ii}	0.9600	2.522(3)	3.411(4)	154.1(2)
C12 -H12C --Cg(2)^{ii}	0.9600	2.7331	3.5128(3)	138.80
O14-H14...O3^{iii}	0.8200	2.414(2)	3.019(3)	131.3(1)
C4 -H4B --Cg(3)^{iv}	0.9700	3.1348	3.916(3)	138.65
C11 -H11 --Cg(2)^{v}	0.9300	2.8678	3.669(3)	145.10
C2-H2...O14	0.9800	2.461(2)	2.910(3)	107.5(1)
C16-H16...N1	0.9300	2.539(2)	2.866(3)	101.0(2)

Symmetry code (i) -x+2,+y-1/2,-z+2 (ii) -x+1,+y-1/2,-z+1 (iii) -x+2,+y-1/2,-z+1
(iv) -1+x,y,z (v) -x,1/2+y,1-z


29 Jan 2012
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7.52
7.50
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7.37
7.26
6.77
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6.36
5.09
3.93
3.90
3.89
3.70
3.69
3.67
3.60
3.54
3.52
2.35
2.33
2.15
2.12
2.10
2.07
2.06
2.04
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1.94
1.95
1.92
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1.08
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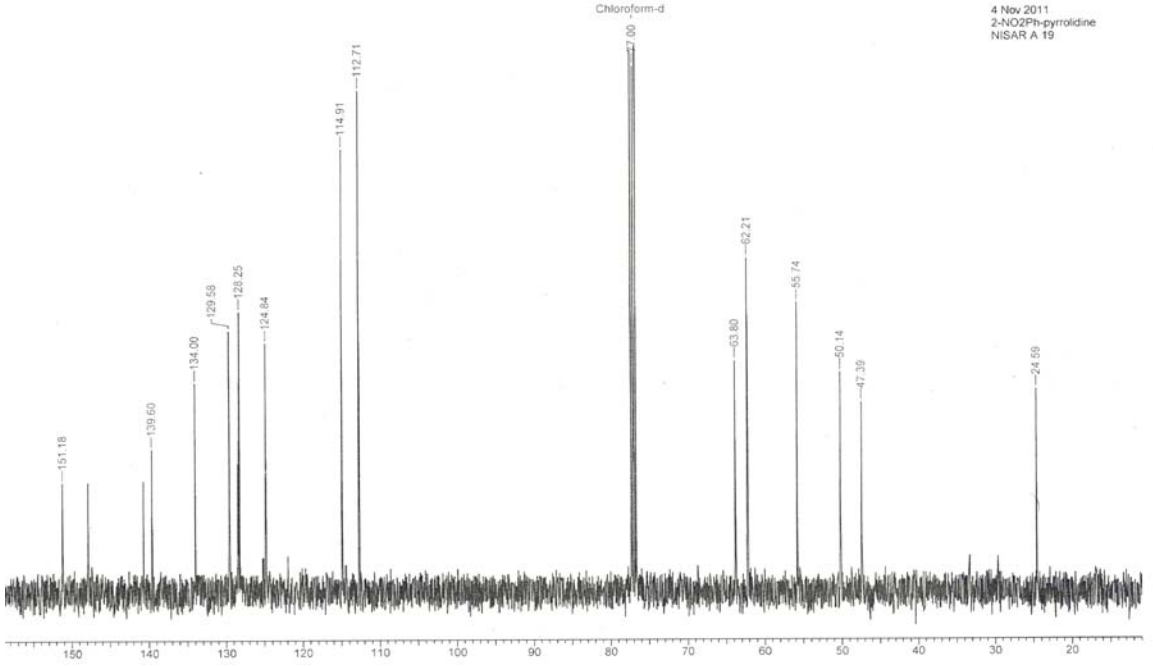
2.27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4 Nov 2011
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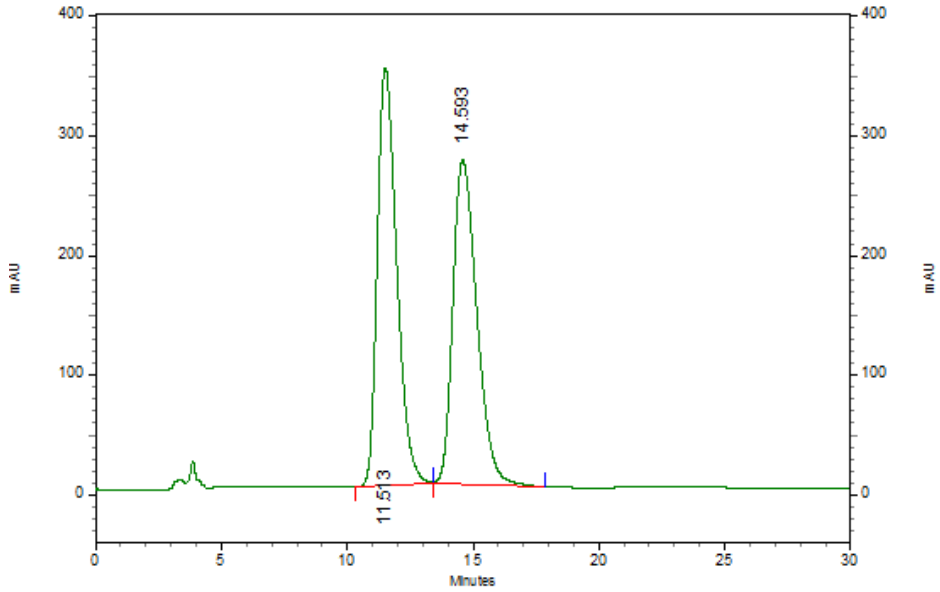
24.59



Racemic **6a**

Retention Time	Area	Area %
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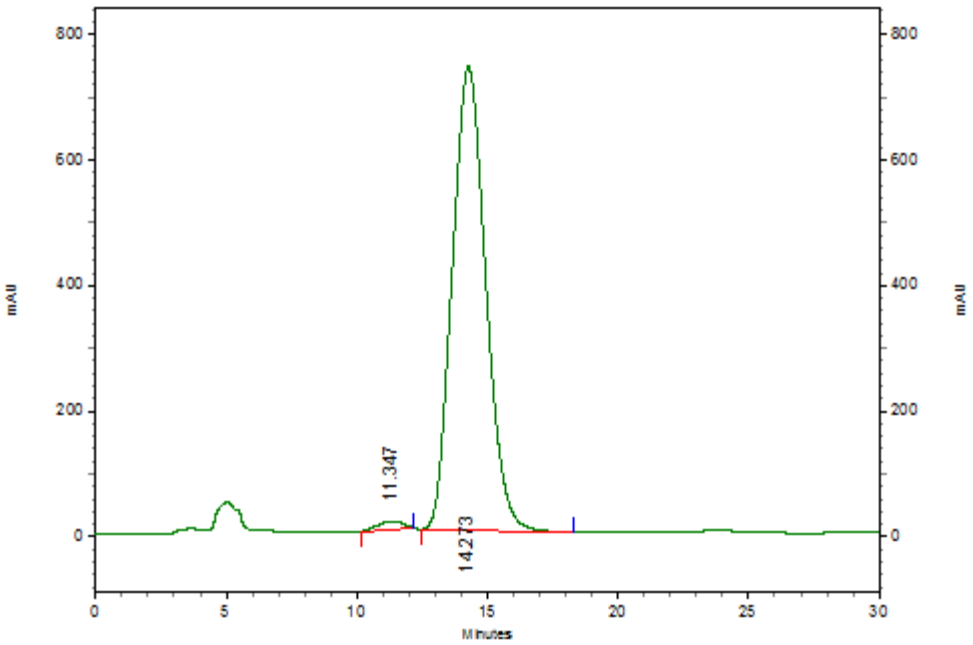
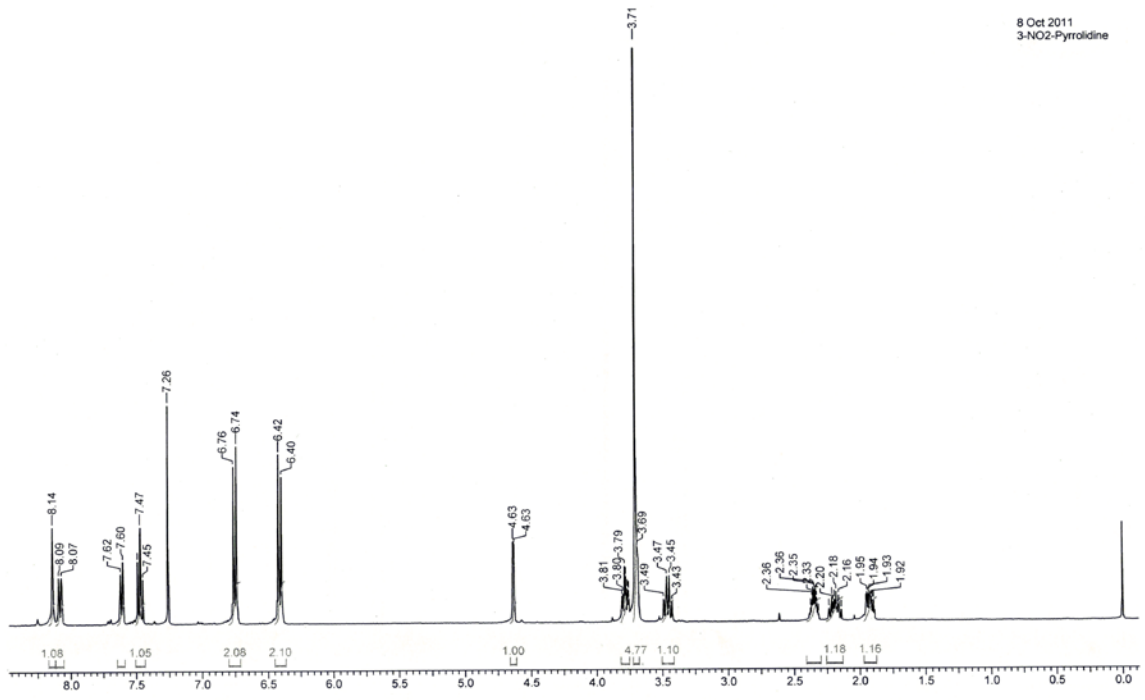
14.593	37424555	48.85
Totals	76611243	100.00



Chiral **6a** (~98% ee)

Retention Time	Area	Area %
11.347	1757442	1.24
14.273	137216302	98.76
Totals	138973744	100.00


8 Oct 2011
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7.26
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4.63
3.81
3.80
3.79
3.71
3.69
3.49
3.47
3.45
3.43
2.36
2.35
2.33
2.20
2.18
2.16
1.95
1.94
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1.92
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1.05
2.08
2.10
1.00
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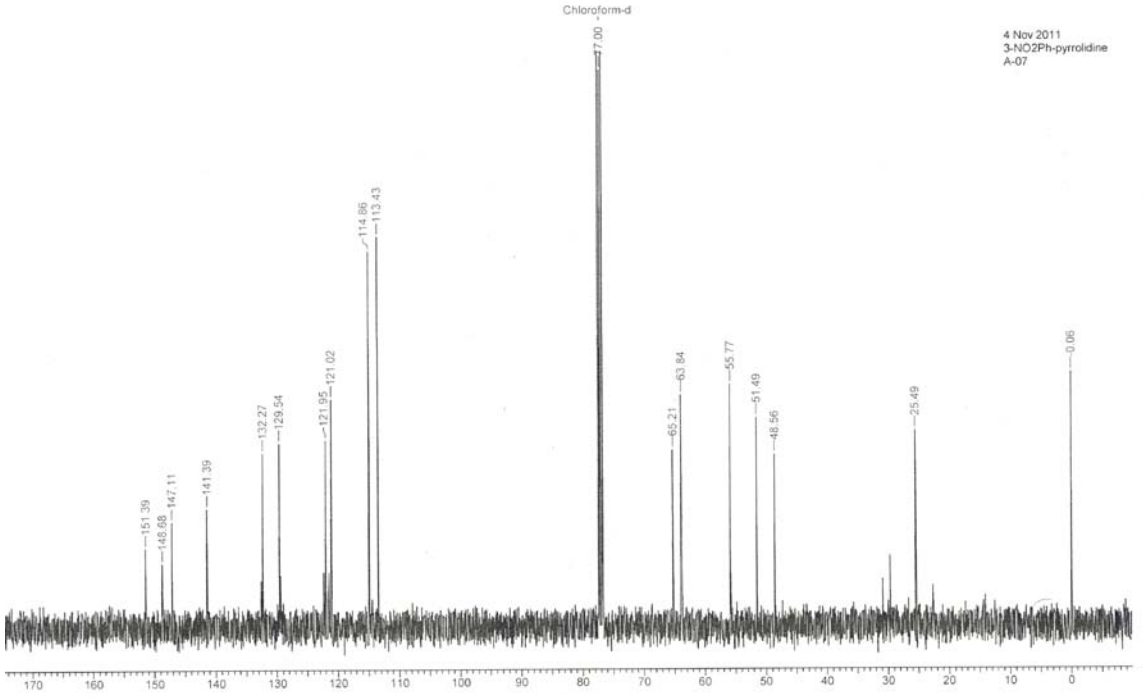
1.18
1.16


Chloroform-d
4 Nov 2011
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148.68
147.11
141.39
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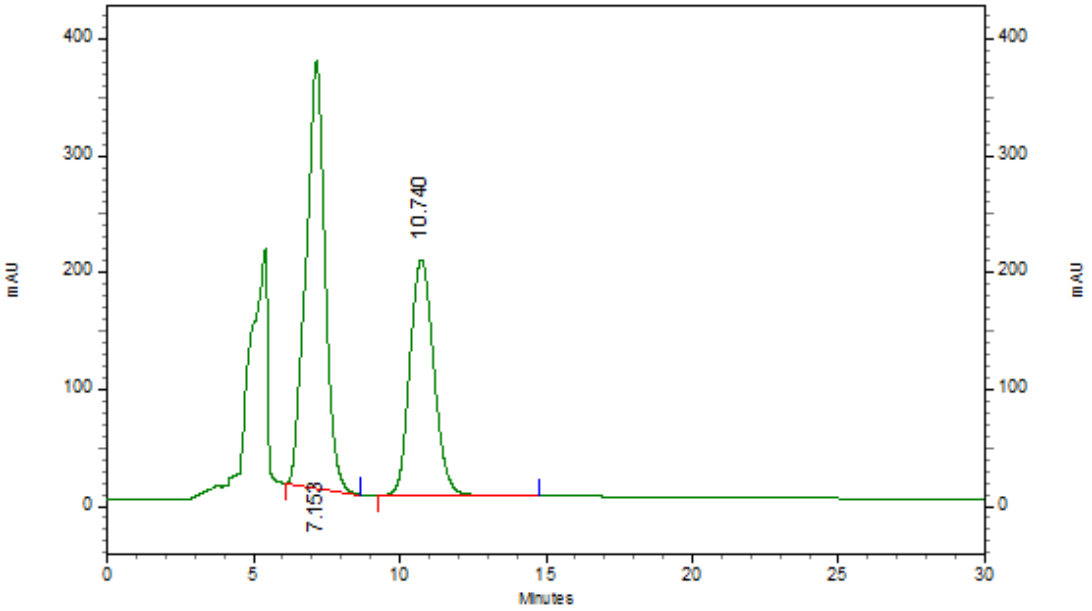
55.77
51.49
48.56
25.49
0.06



Racemic **6b**

Retention Time	Area	Area %
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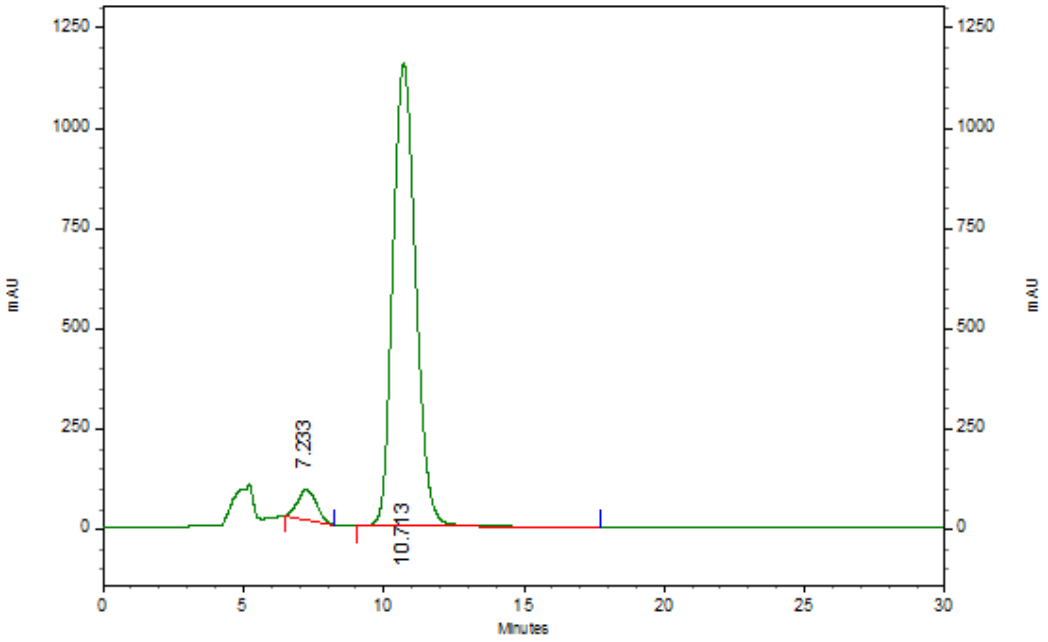
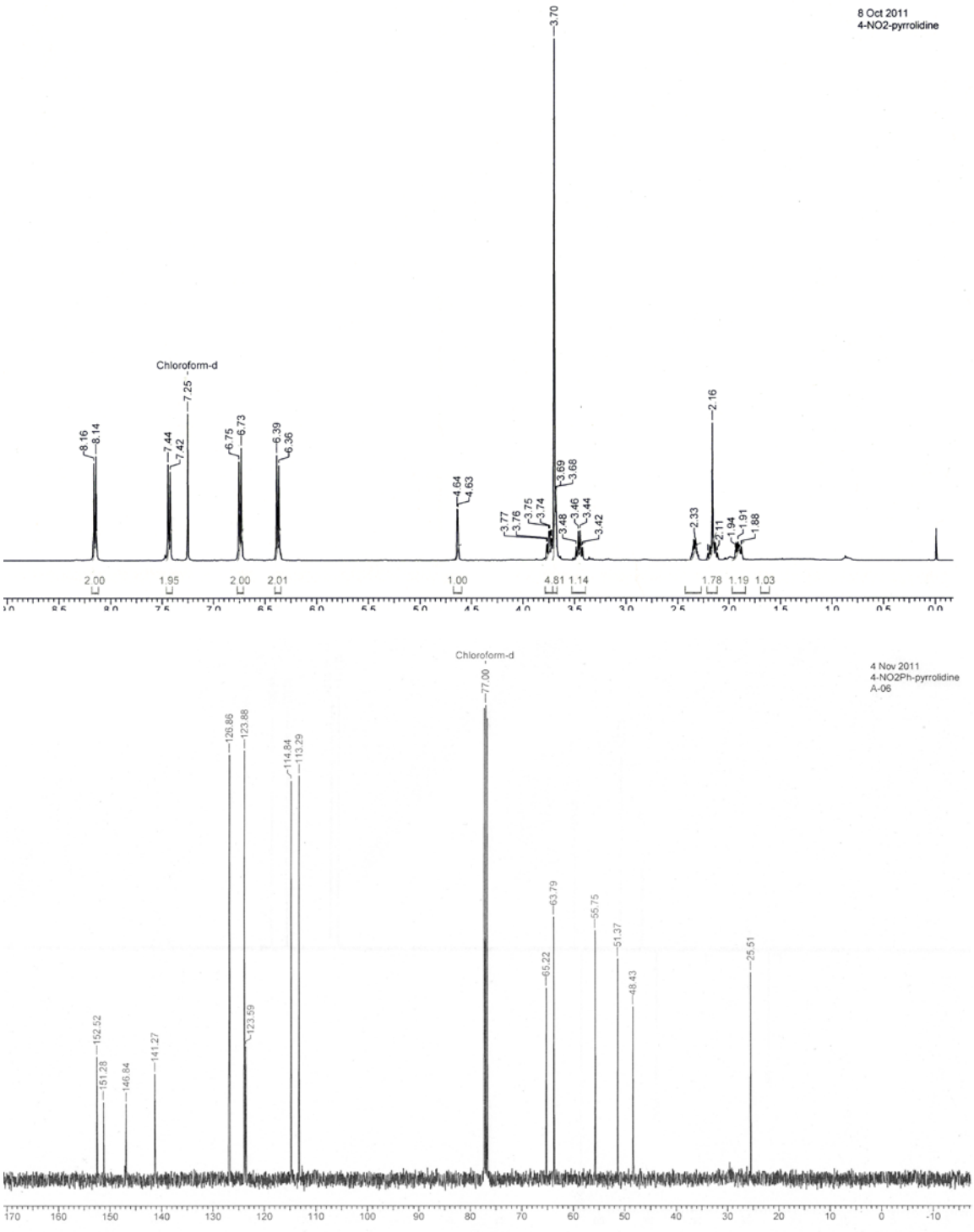
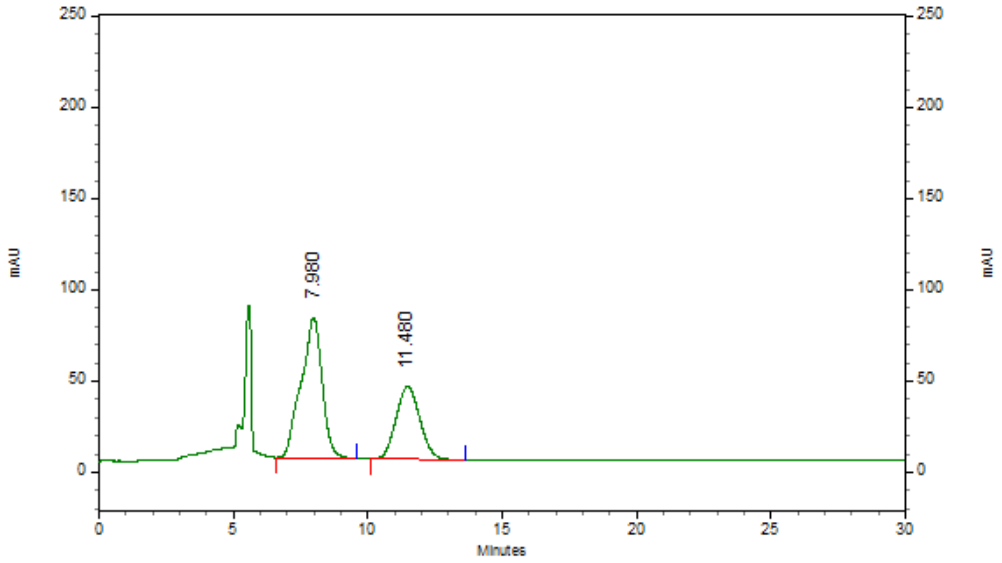
10.740	24174312	41.16
Totals	58735137	100.00



Chiral **6b** (92% ee)

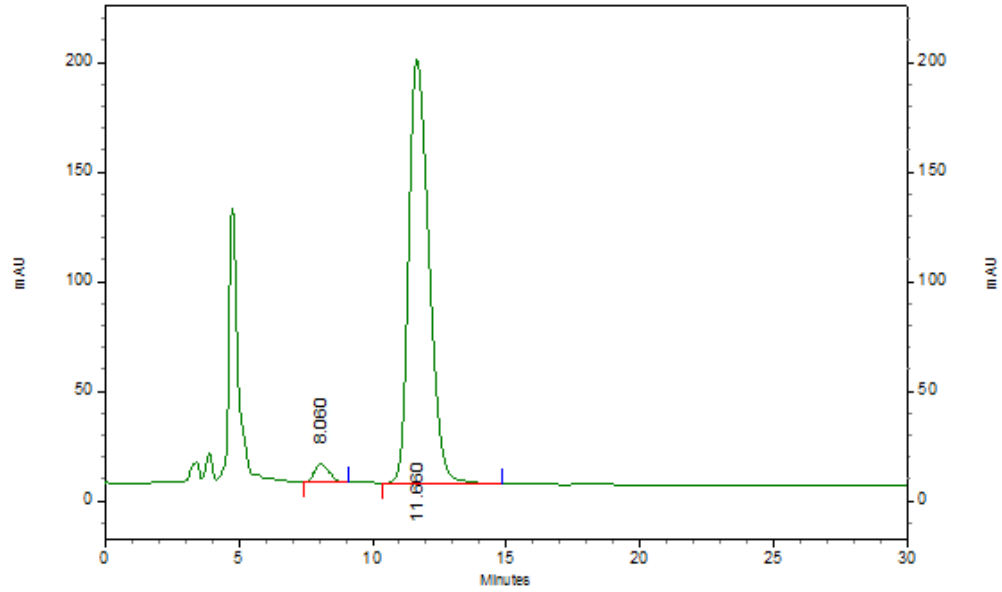
Retention Time	Area	Area %
7.233	5778051	4.05
10.713	136920657	95.95
Totals	142698708	100.00


8 Oct 2011
4-NO2-pyrrolidine
Chloroform-d
8.16
8.14
7.44
7.42
7.25
6.75
6.73
6.39
6.36
4.64
4.63
3.77
3.76
3.75
3.74
3.70
3.69
3.68
3.48
3.46
3.44
3.42
2.33
2.16
2.11
1.94
1.91
1.88
2.00
1.95
2.00
2.01
1.00
4.81
1.14
1.78
1.19
1.03
4 Nov 2011
4-NO2Ph-pyrrolidine
A-06
Chloroform-d
152.52
151.28
146.84
141.27
126.86
123.88
123.59
114.84
113.29
77.00
65.22
63.79
55.75
51.37
48.43
25.51



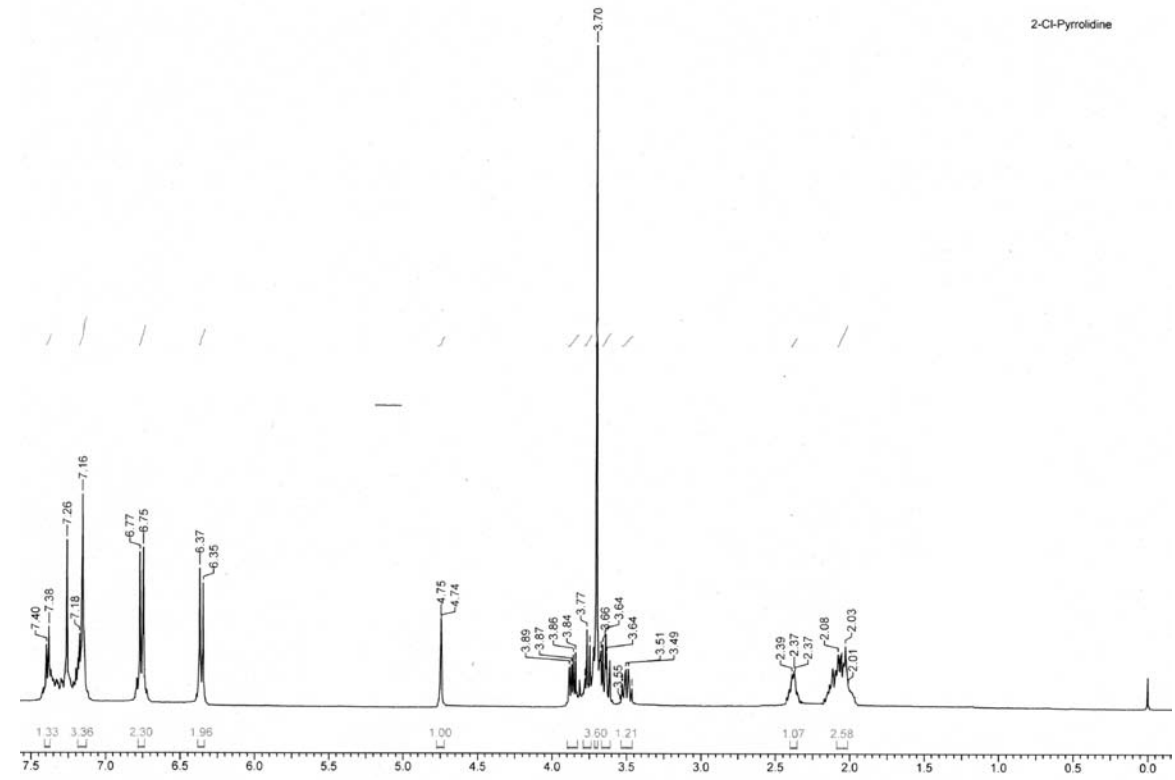
Racemic **6c**

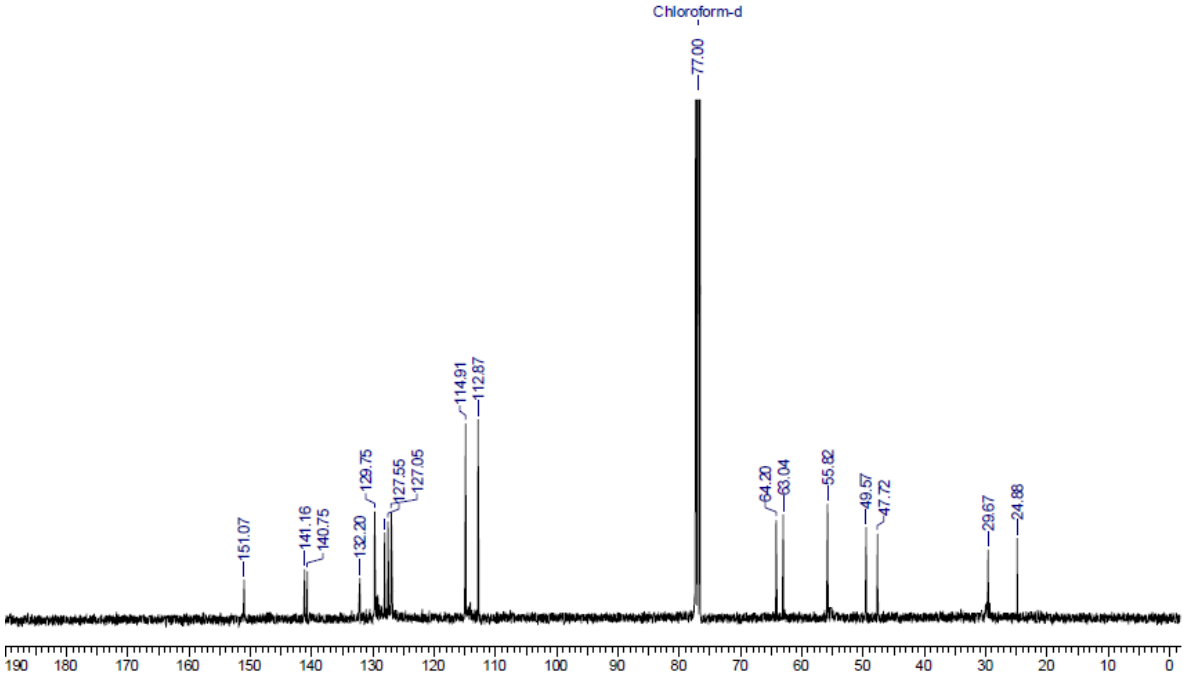
Retention Time	Area	Area %
7.980	8765655	62.80
11.480	5193095	37.20
Totals	13958750	100.00

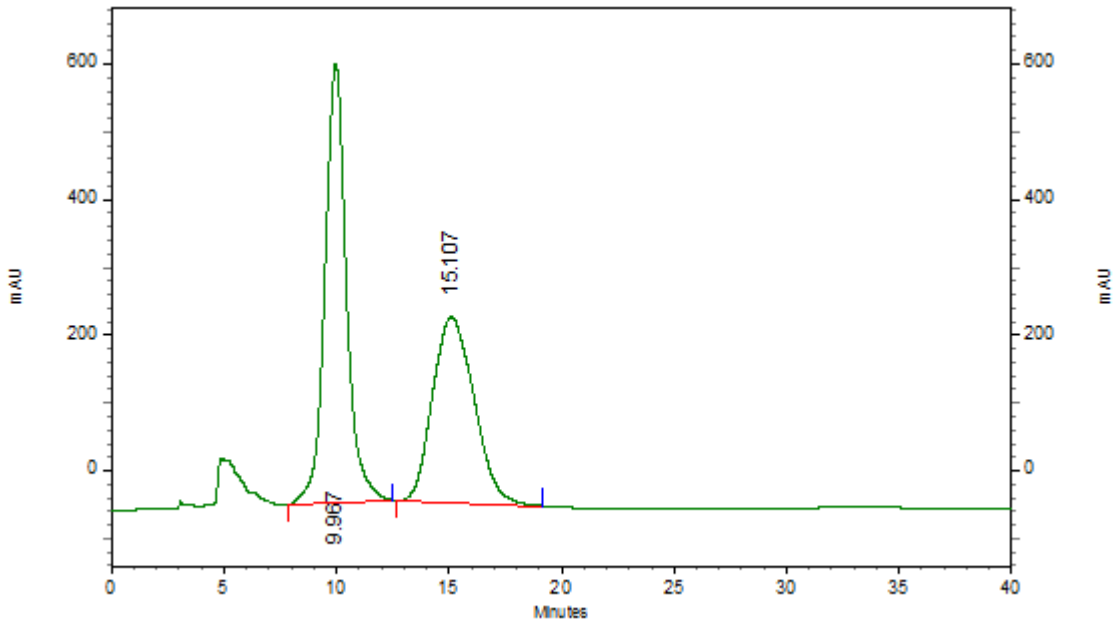


Chiral **6c** (96% ee)

Retention Time	Area	Area %
8.060	448002	2.05
11.660	21333355	97.95
Totals	21781357	100.00

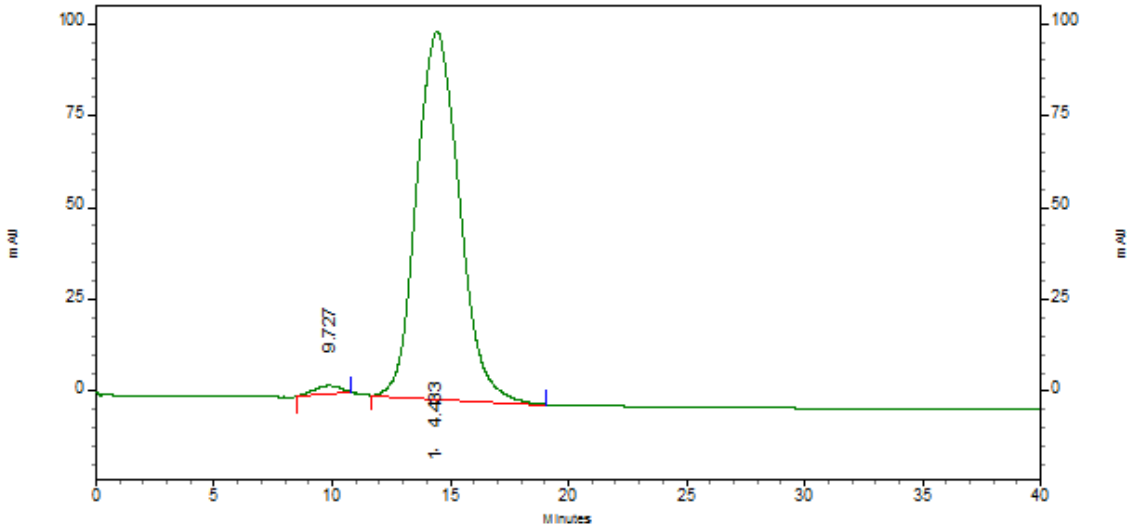

2-Cl-Pyrrolidine
3.70
7.40
7.38
7.26
7.18
7.16
6.77
6.75
6.37
6.35
4.75
4.74
3.89
3.87
3.86
3.84
3.77
3.66
3.64
3.64
3.55
3.51
3.49
2.39
2.37
2.37
2.08
2.03
2.01
1.33
3.36
2.30
1.96
1.00
3.60
1.21
1.07
2.58


Chloroform-d
77.00
151.07
141.16
140.75
132.20
129.75
127.55
127.05
114.91
112.87
64.20
63.04
55.82
49.57
47.72
29.67
24.88



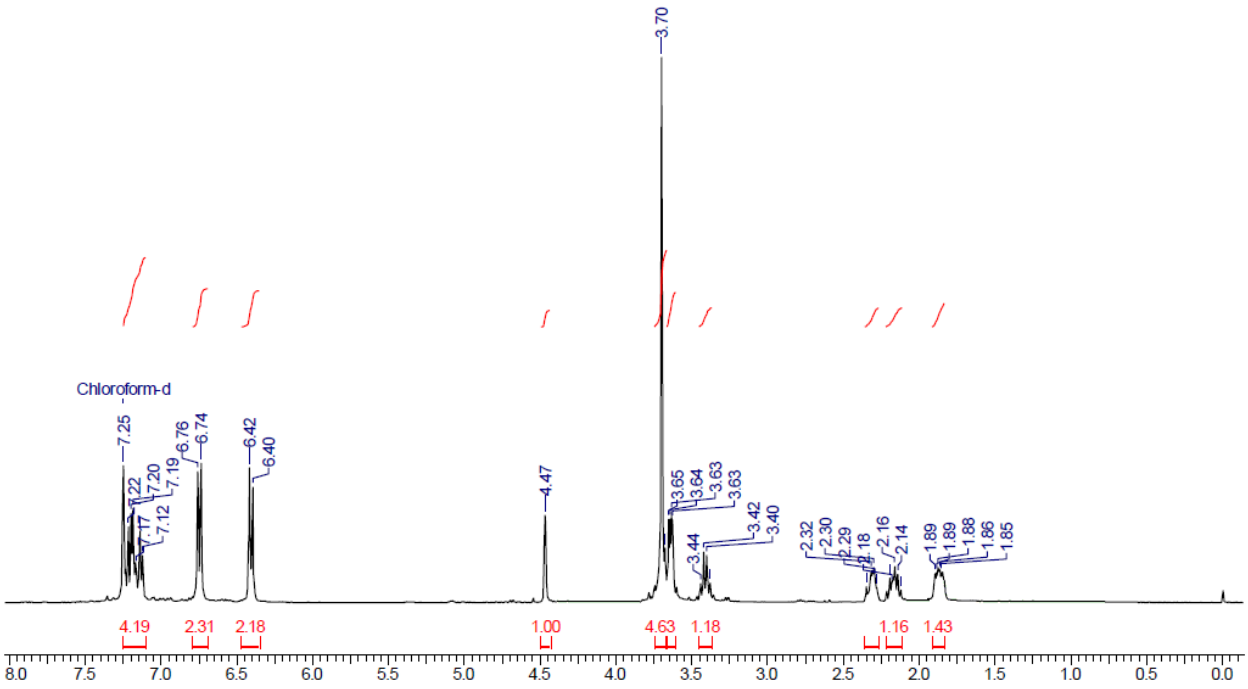
Racemic **6d**

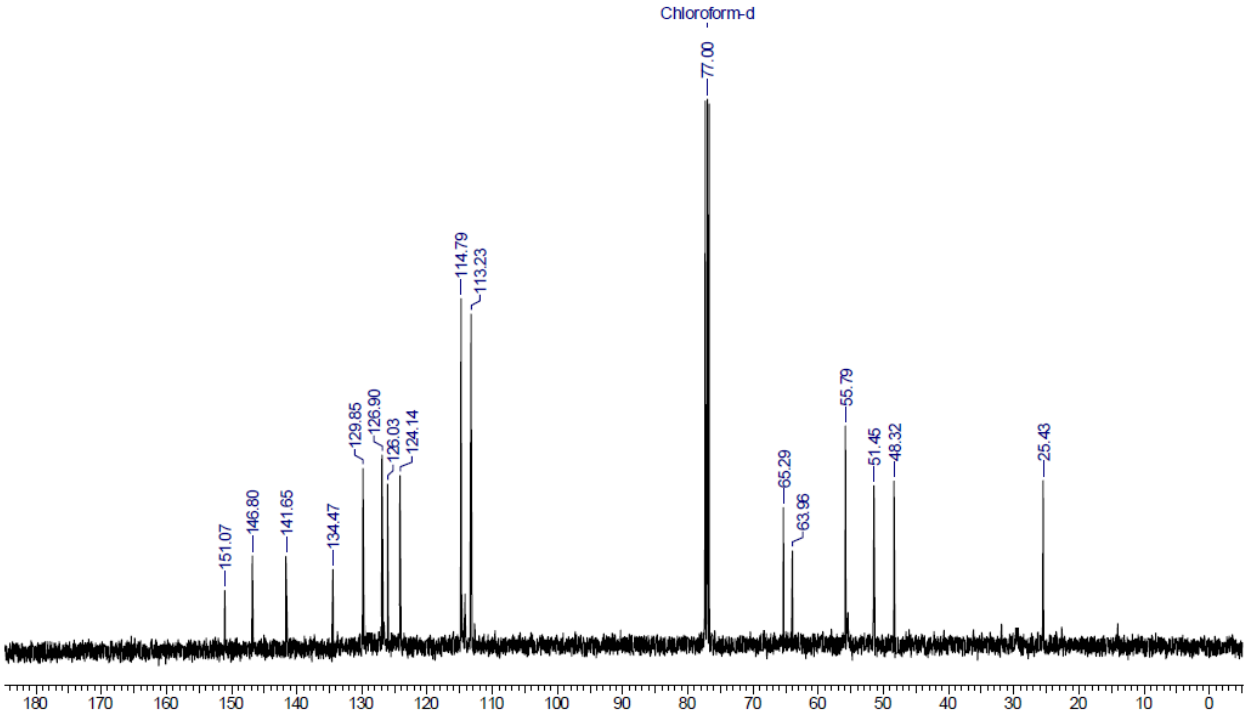
Retention Time	Area	Area %
9.967	90399843	55.13
15.107	73571507	44.87
Totals	163971350	100.00

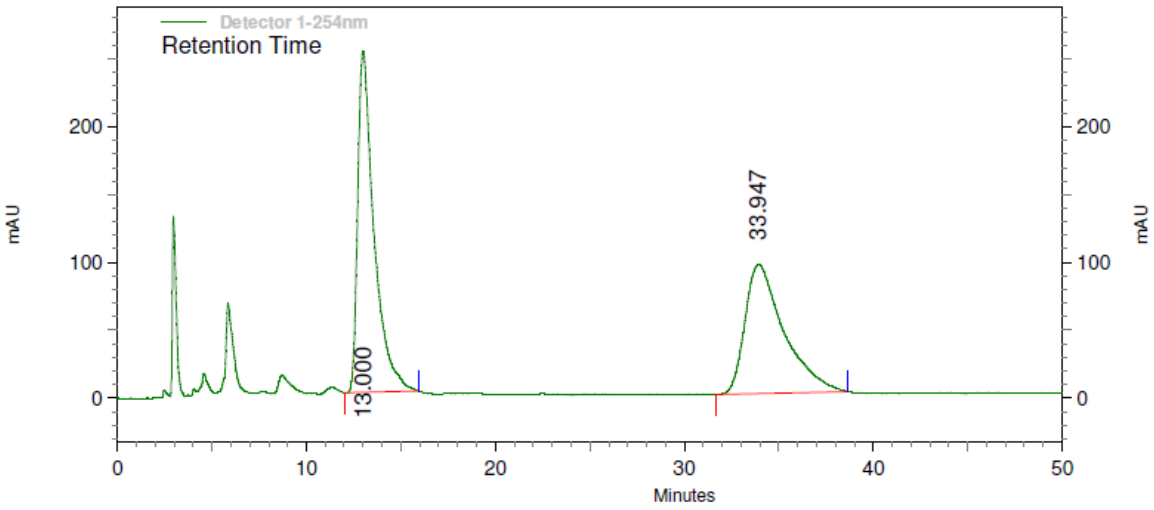


Chiral **6d** (>99% ee)

Retention Time	Area	Area %
9.727	107961	0.41
14.433	26332058	99.59
Totals	26439019	100.00

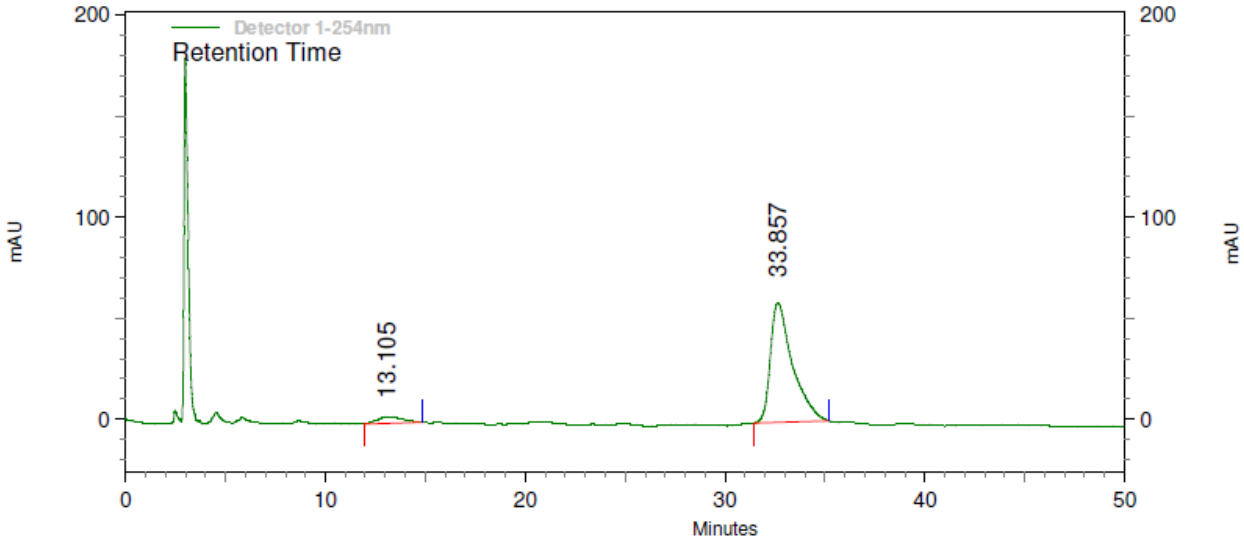

Chloroform-d
7.25
7.22
7.20
7.19
7.17
7.12
6.76
6.74
6.42
6.40
4.47
3.70
3.65
3.64
3.63
3.63
3.42
3.40
3.44
2.32
2.30
2.29
2.18
2.16
2.14
1.89
1.89
1.88
1.86
1.85
4.19
2.31
2.18
1.00
4.63
1.18
1.16
1.43


Chloroform-d
77.00
151.07
146.80
141.65
134.47
129.85
126.90
126.03
124.14
114.79
113.23
65.29
63.96
55.79
51.45
48.32
25.43



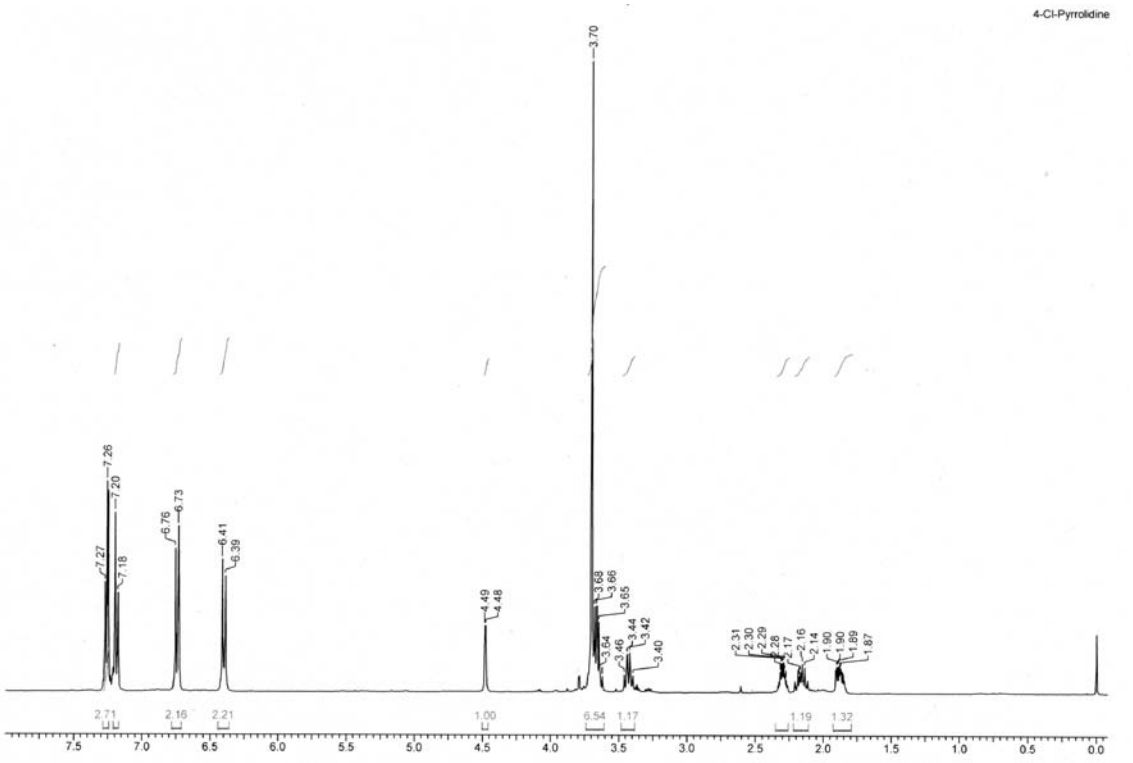
Racemic **6e**

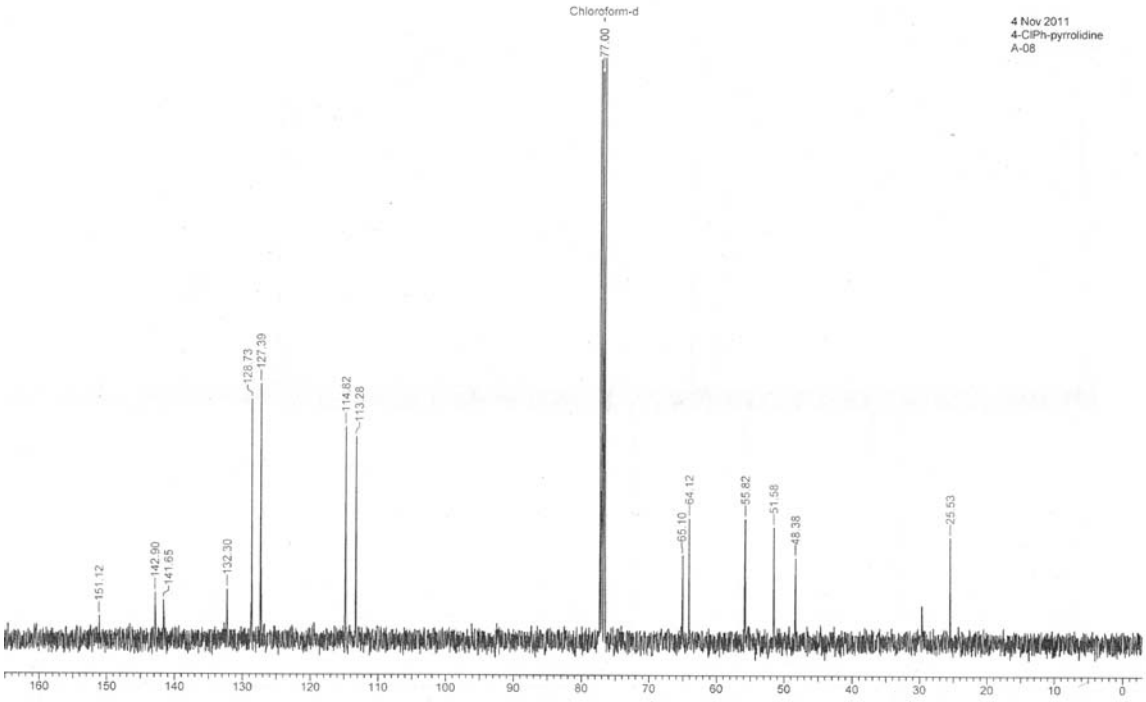
Retention Time	Area	Area %
13.000	15043028	55.59
39.947	13562341	47.41
Totals	28605369	100.00

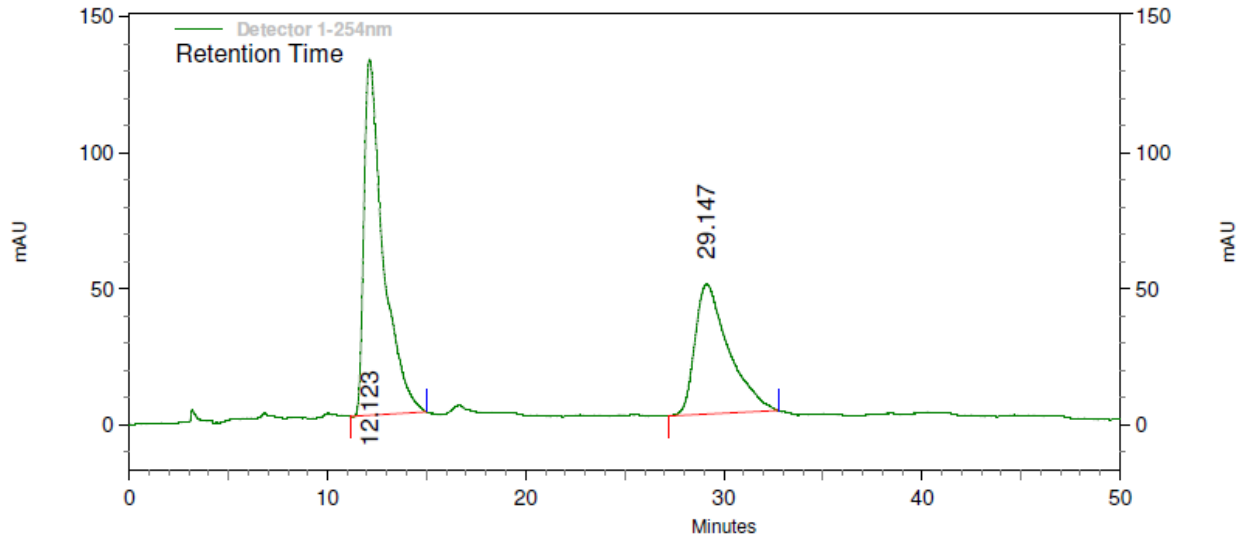


Chiral **6e** (92% ee)

Retention Time	Area	Area %
13.105	644753	4.11
33.857	15042679	95.89
Totals	15687432	100.00

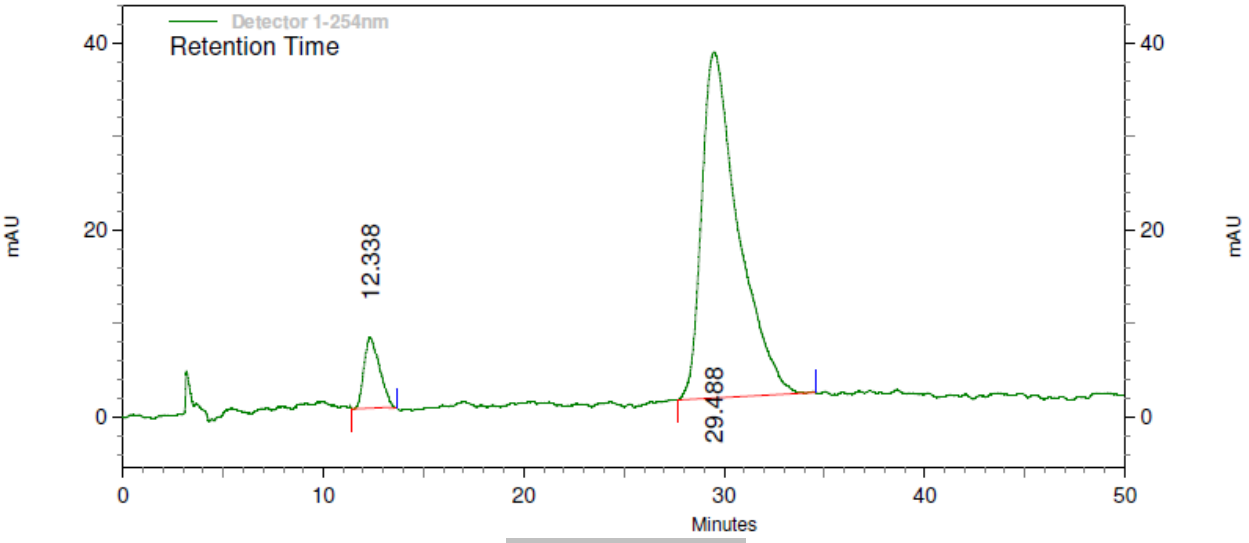

4-Cl-Pyrrolidine
7.26
7.20
7.27
7.18
6.76
6.73
6.41
6.39
4.49
4.48
3.70
3.68
3.66
3.65
3.64
3.46
3.44
3.42
3.40
2.31
2.30
2.29
2.28
2.17
2.16
2.14
1.90
1.89
1.87
2.71
2.16
2.21
1.00
6.54
1.17
1.19
1.32


Chloroform-d
77.00
4 Nov 2011
4-ClPh-pyrrolidine
A-08
151.12
142.90
141.65
132.30
128.73
127.39
114.82
113.28
65.10
64.12
55.82
51.58
48.38
25.53



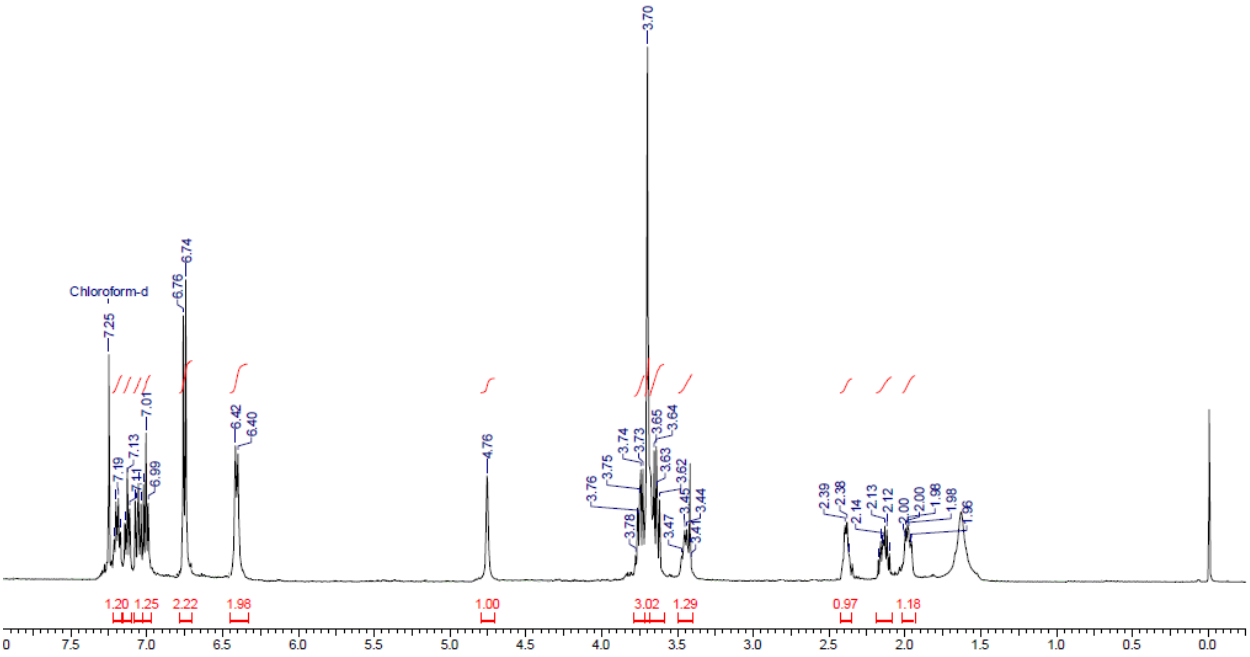
Racemic **6f**

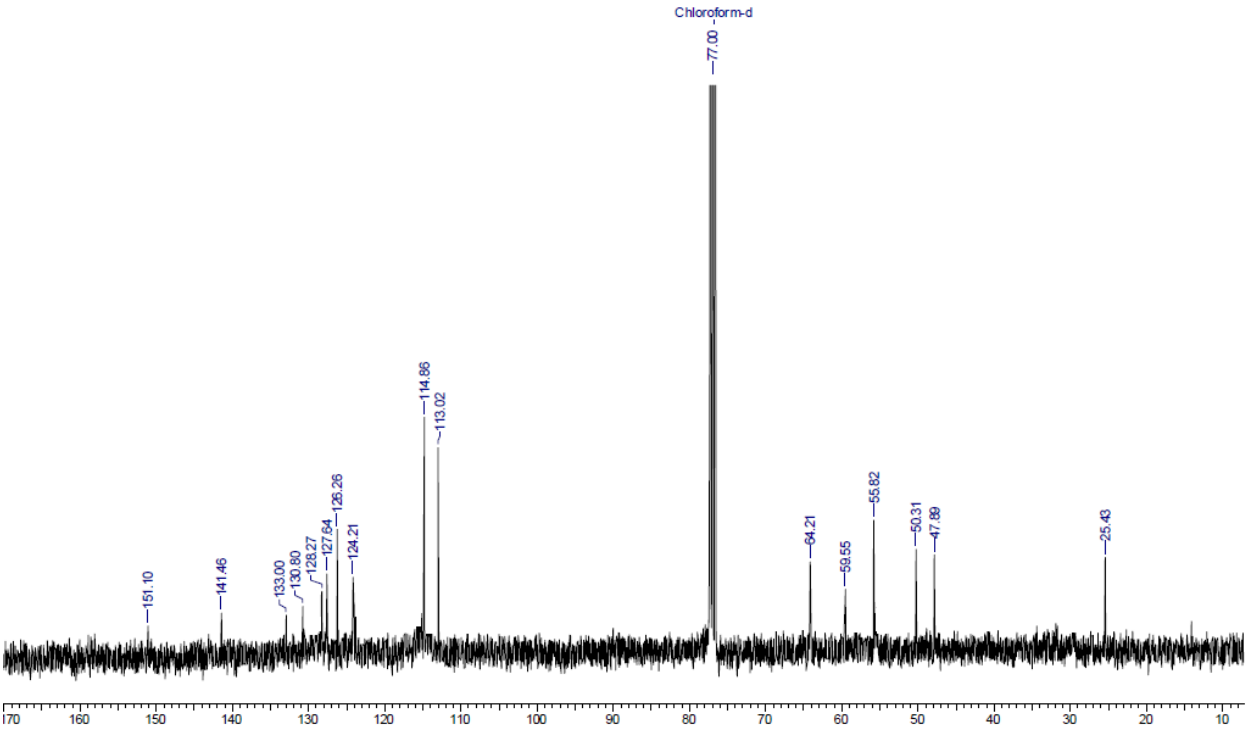
Retention Time	Area	Area %
12.123	8786594	60.90
29.147	5641683	39.10
Totals	14428277	100.00

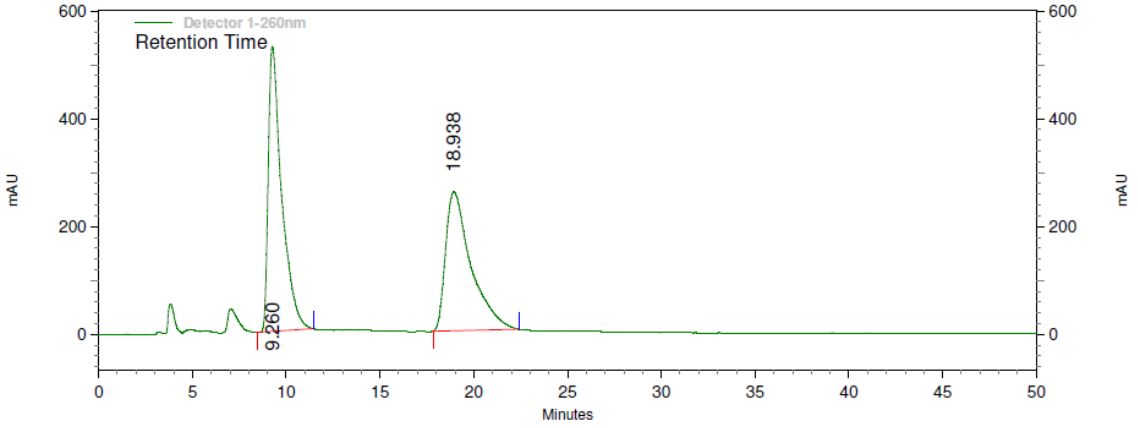


Chiral **6f** (90% ee)

Retention Time	Area	Area %
12.338	271627	5.11
29.488	5043967	94.89
Totals	5315594	100.00

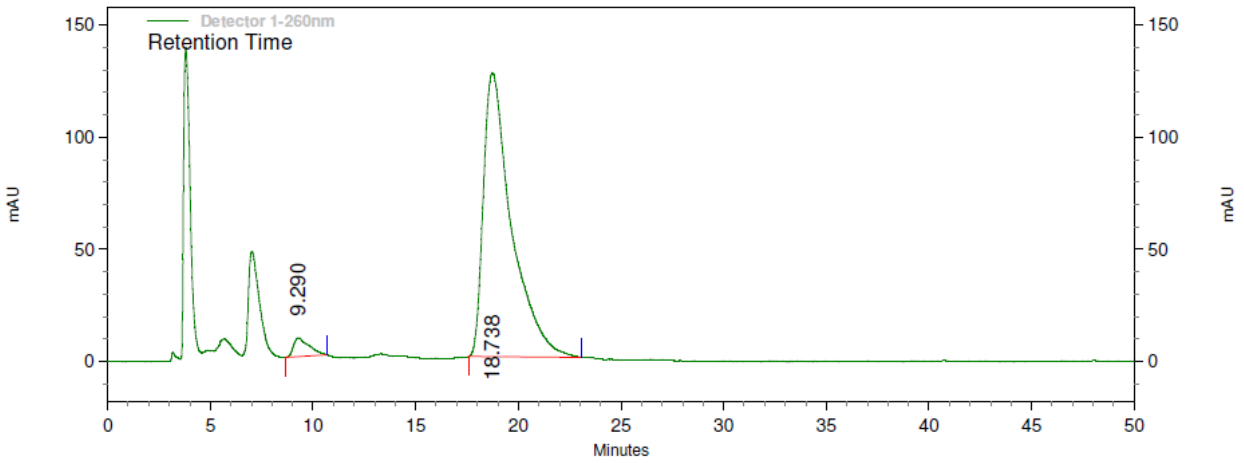

Chloroform-d
7.25
7.19
7.14
7.13
7.01
6.99
6.76
6.74
6.42
6.40
4.76
3.76
3.75
3.74
3.73
3.70
3.65
3.64
3.63
3.62
3.47
3.45
3.44
3.41
3.78
2.39
2.38
2.14
2.13
2.12
2.00
1.98
1.96
1.20
1.25
2.22
1.98
1.00
3.02
1.29
0.97
1.18


Chloroform-d
77.00
151.10
141.46
133.00
130.80
128.27
127.64
126.26
124.21
114.86
113.02
64.21
59.55
55.82
50.31
47.89
25.43



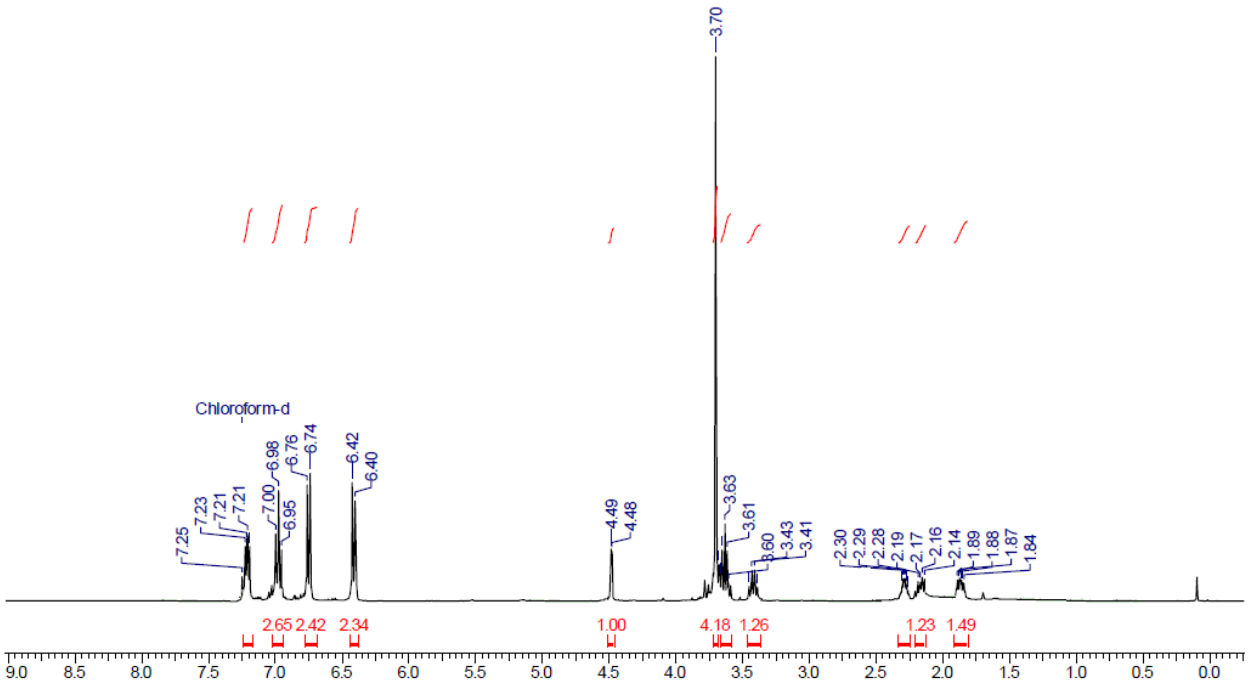
Racemic **6g**

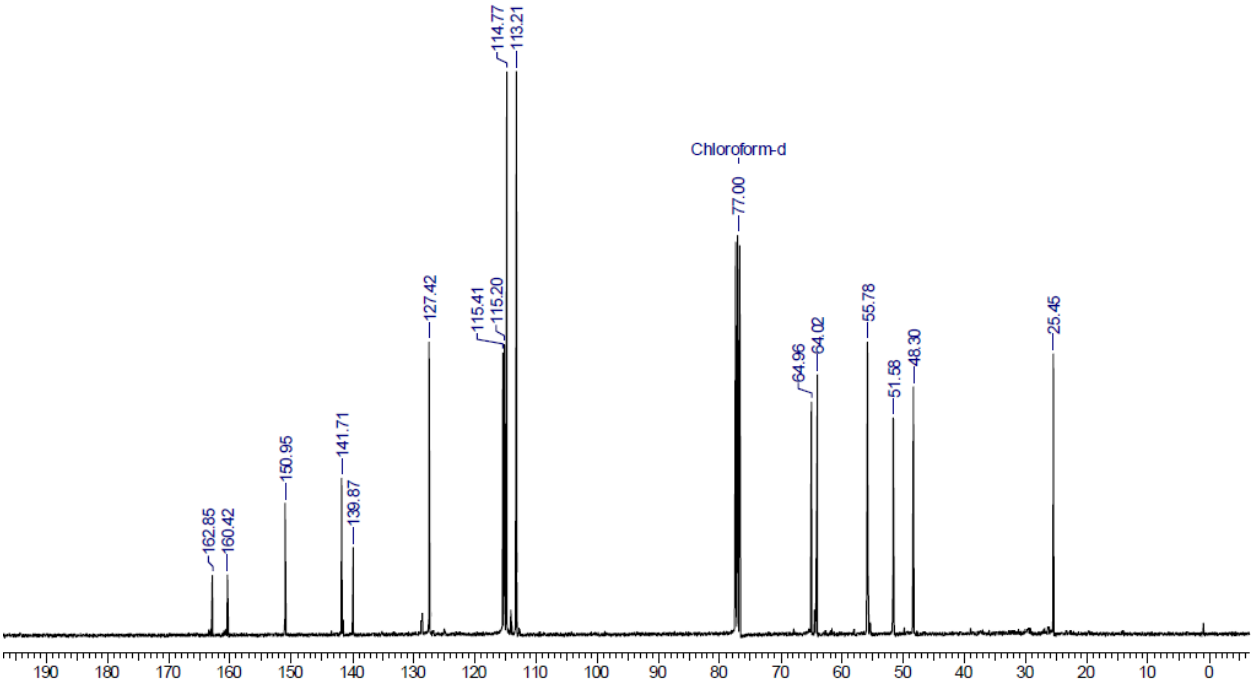
Retention Time	Area	Area %
9.260	26311563	52.02
18.938	24267934	47.98
Totals	50579497	100.00

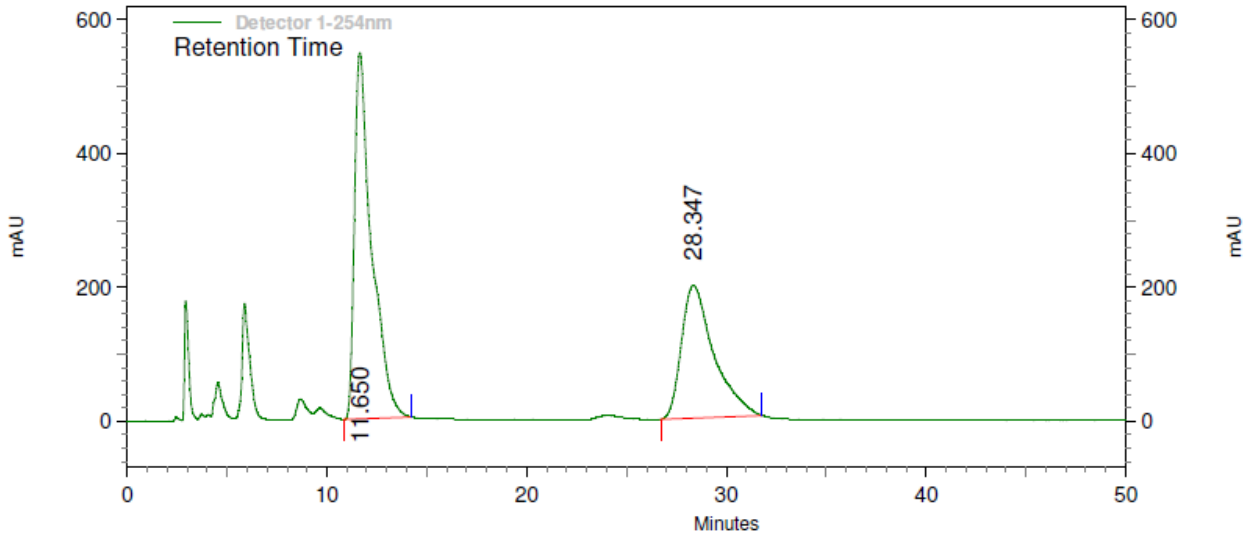


Chiral **6g** (93% ee)

Retention Time	Area	Area %
9.290	437773	3.51
18.738	12029633	96.49
Totals	12467406	100.00

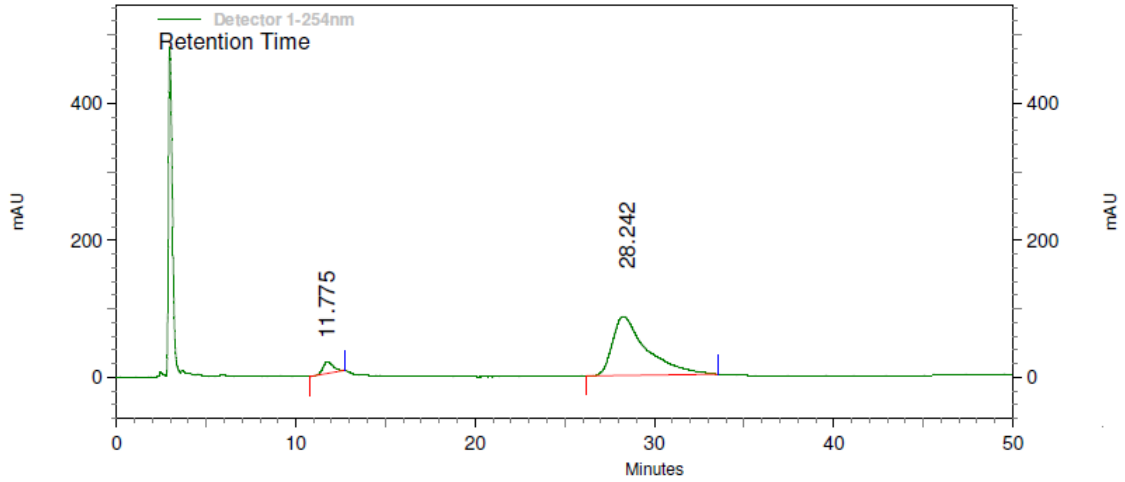

3.70
Chloroform-d
7.25
7.23
7.21
7.21
7.00
6.98
6.95
6.76
6.74
6.42
6.40
4.49
4.48
3.63
3.61
3.60
3.43
3.41
2.30
2.29
2.28
2.19
2.17
2.16
2.14
1.89
1.88
1.87
1.84
2.65
2.42
2.34
1.00
4.18
1.26
1.23
1.49


114.77
113.21
Chloroform-d
77.00
127.42
115.41
115.20
64.96
64.02
55.78
51.58
48.30
25.45
162.85
160.42
150.95
141.71
139.87



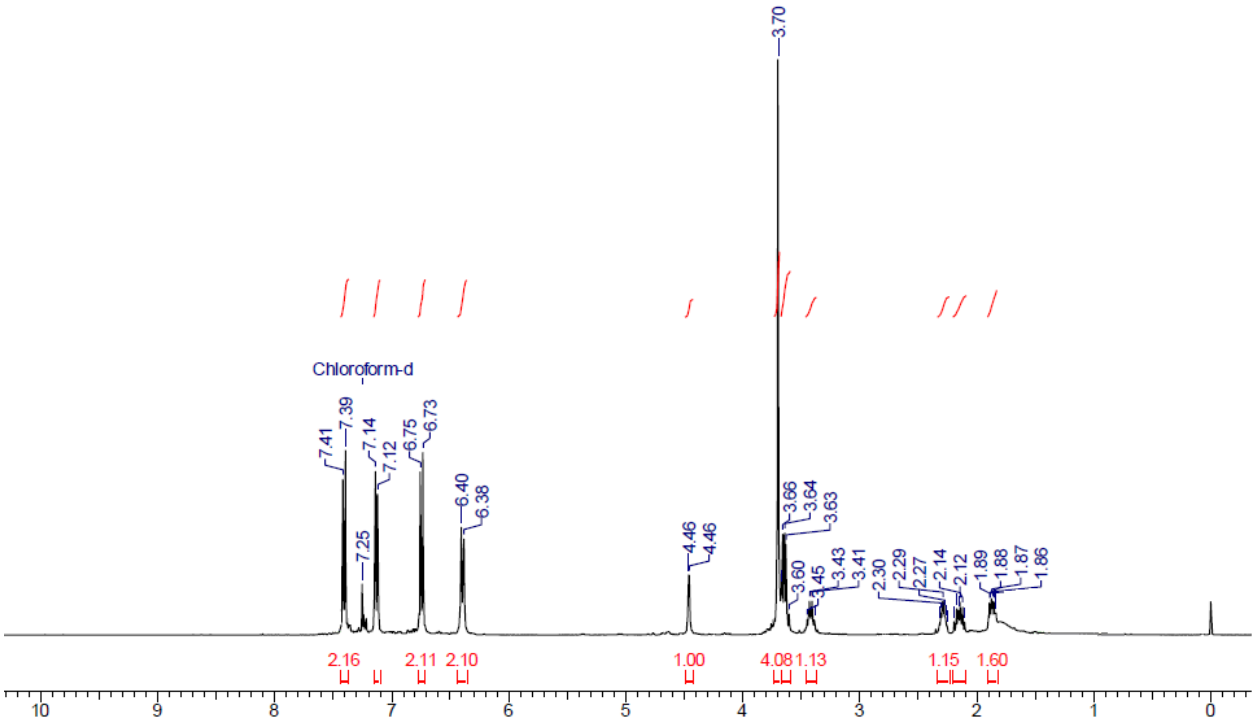
Racemic **6h**

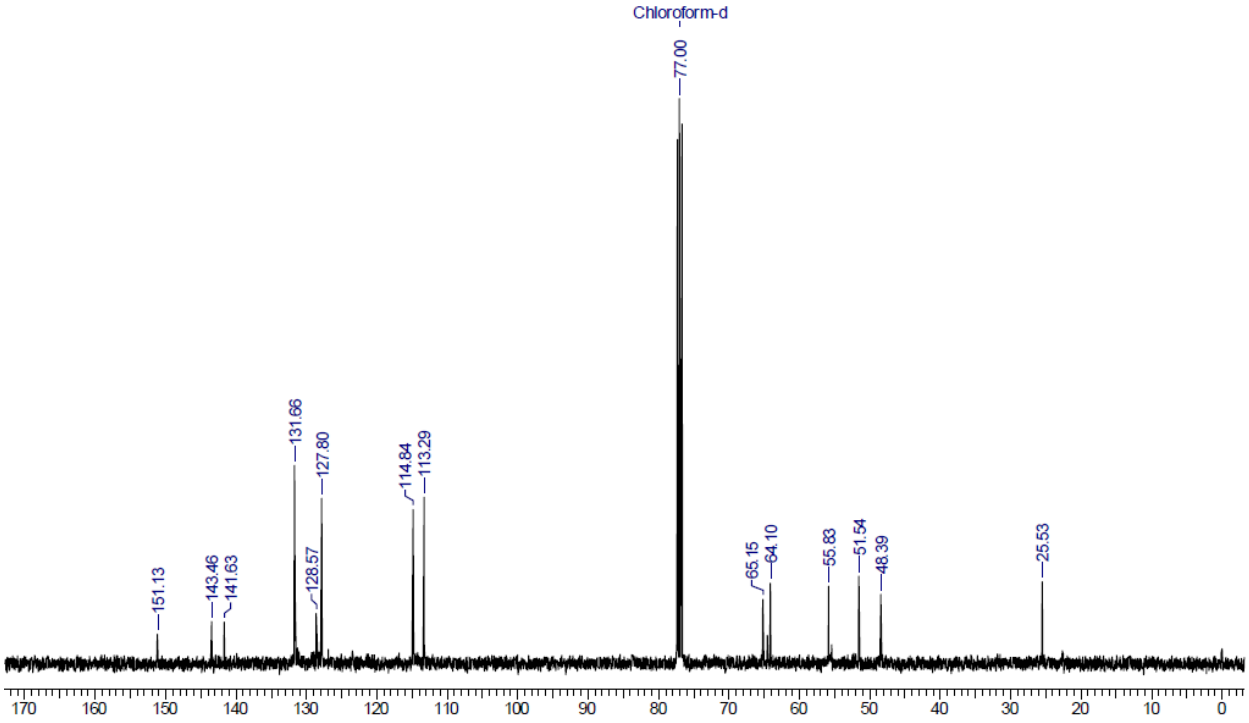
Retention Time	Area	Area %
11.650	32656216	59.36
28.347	22360831	40.64
Totals	50579497	100.00

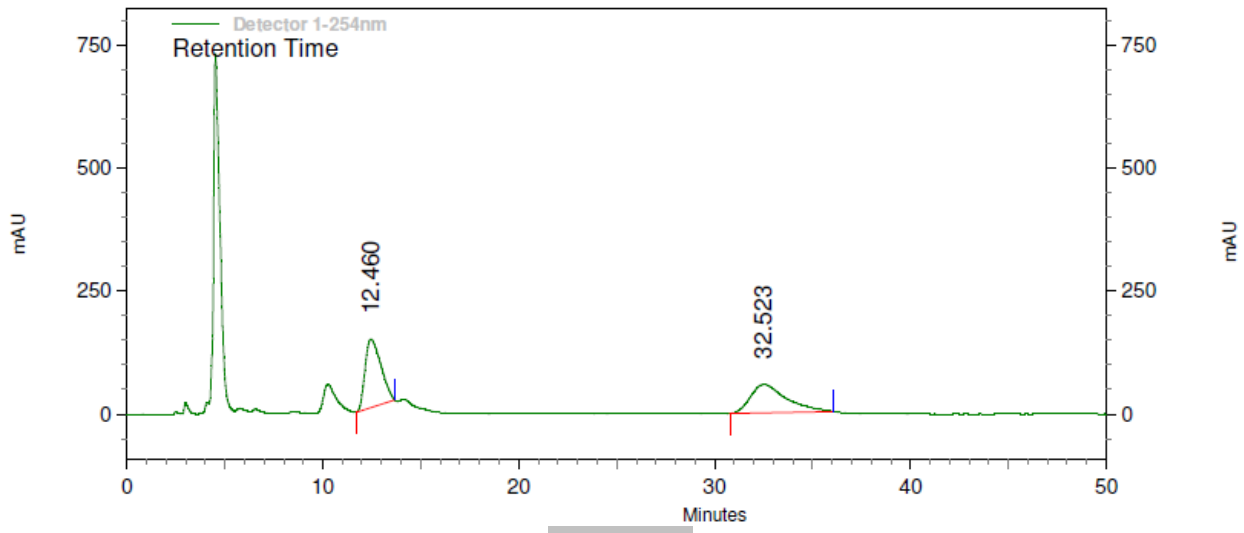


Chiral **6h** (91% ee)

Retention Time	Area	Area %
11.775	234623	4.46
28.242	5025969	95.54
Totals	5260592	100.00

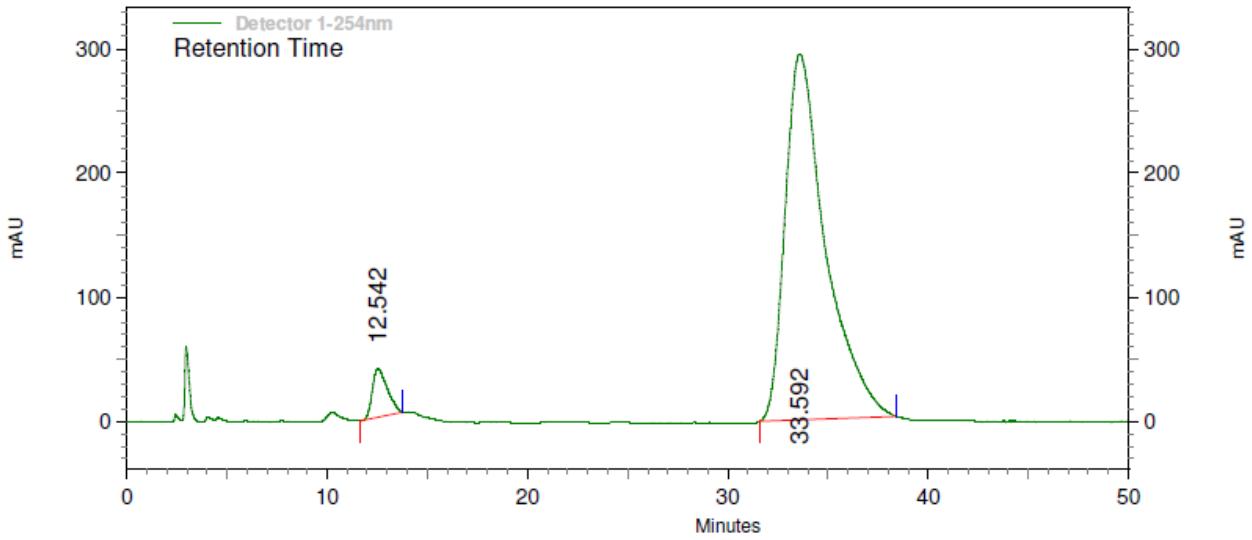

Chloroform-d
7.41
7.39
7.14
7.12
6.75
6.73
6.40
6.38
7.25
4.46
4.46
3.70
3.66
3.64
3.63
3.60
3.45
3.43
3.41
2.30
2.29
2.27
2.14
2.12
1.89
1.88
1.87
1.86
2.16
2.11
2.10
1.00
4.08
1.13
1.15
1.60


Chloroform-d
77.00
131.66
127.80
114.84
113.29
128.57
151.13
143.46
141.63
65.15
64.10
55.83
51.54
48.39
25.53



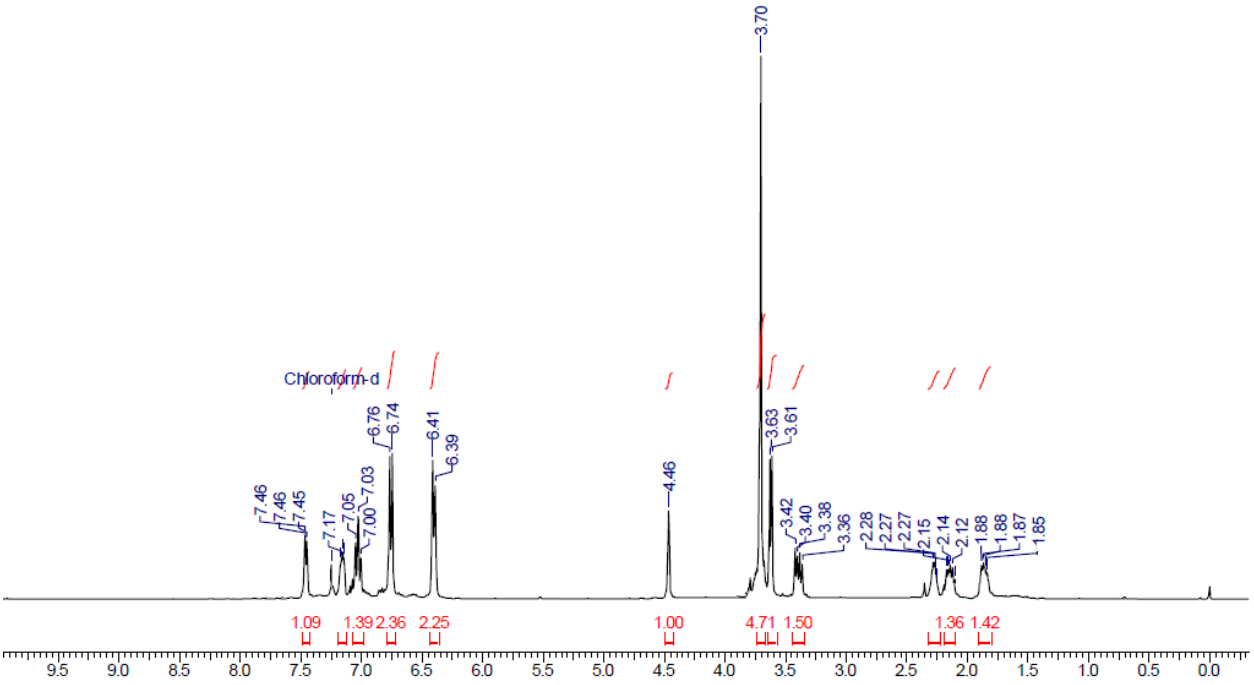
Racemic **6i**

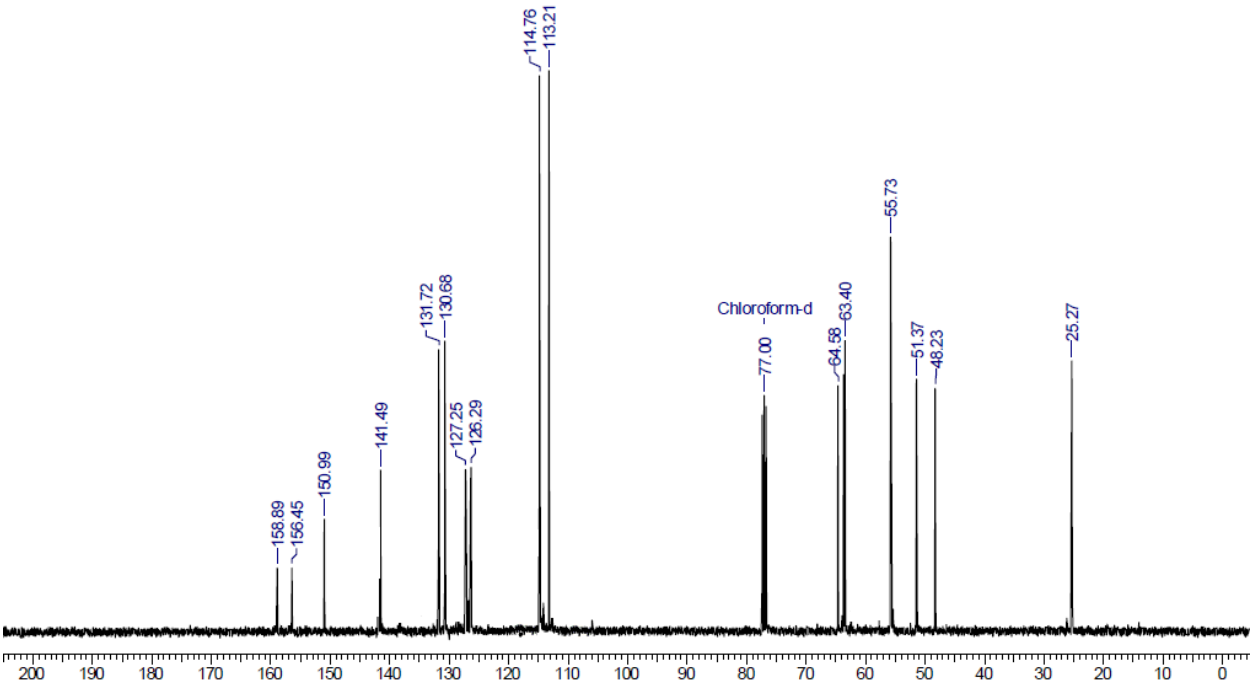
Retention Time	Area	Area %
12.460	7766921	51.30
32.523	7374558	48.70
Totals	15141479	100.00

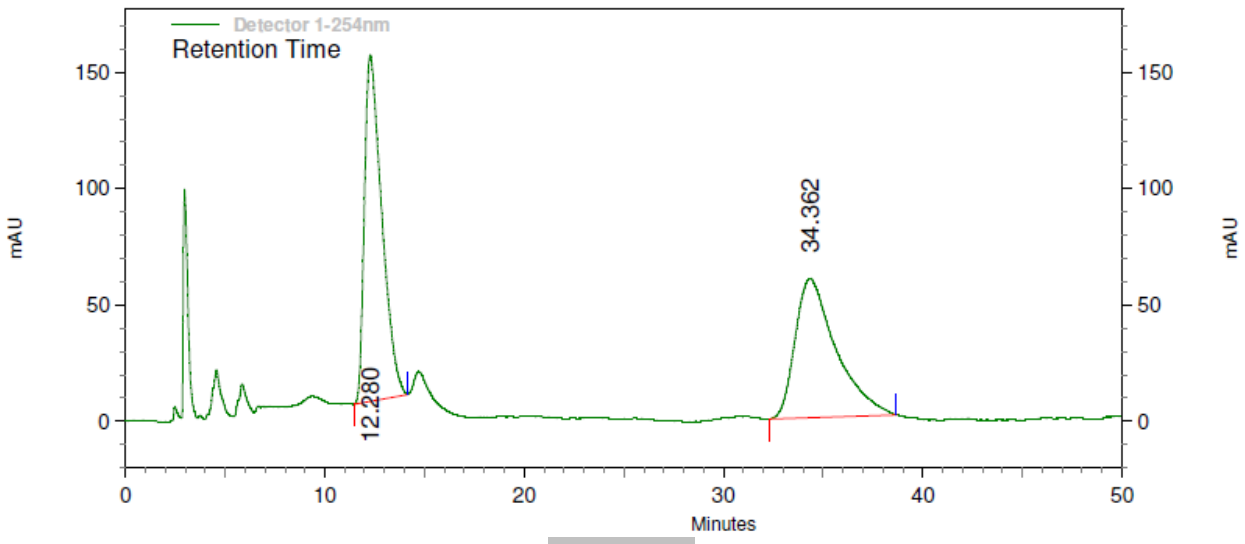


Chiral **6i** (90% ee)

Retention Time	Area	Area %
12.542	2012303	4.65
33.592	41278461	95.35
Totals	43290764	100.00

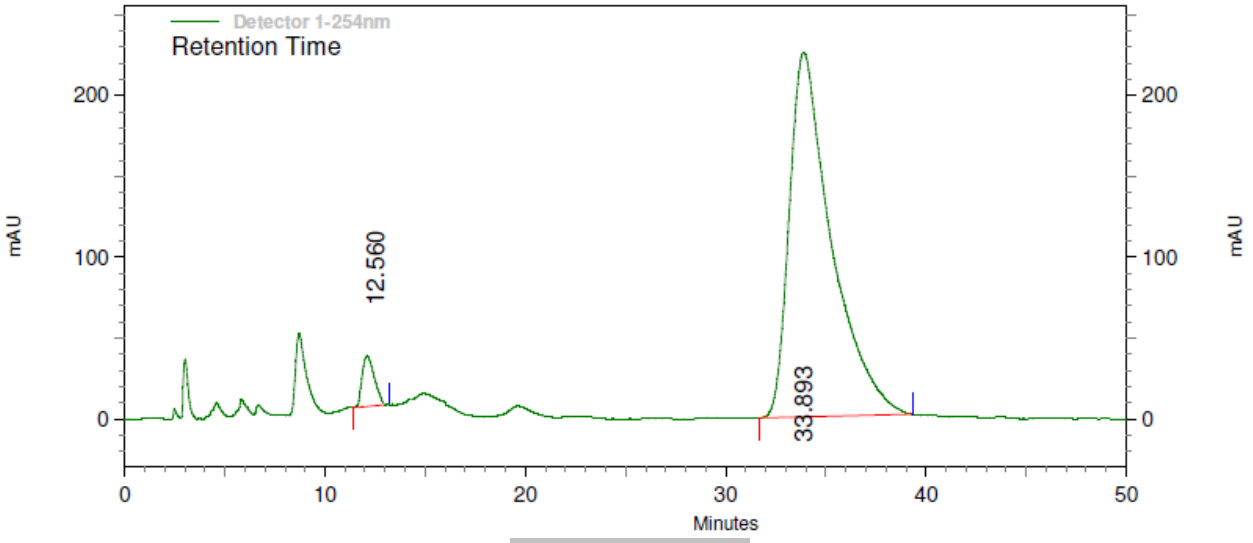

3.70
Chloroform-d
6.76
6.74
6.41
6.39
7.46
7.46
7.45
7.17
7.05
7.03
7.00
4.46
3.63
3.61
3.42
3.40
3.38
3.36
2.28
2.27
2.27
2.15
2.14
2.12
1.88
1.88
1.87
1.85
1.09
1.39
2.36
2.25
1.00
4.71
1.50
1.36
1.42


114.76
113.21
55.73
131.72
130.68
Chloroform-d
63.40
77.00
64.58
51.37
48.23
25.27
141.49
127.25
126.29
158.89
156.45
150.99



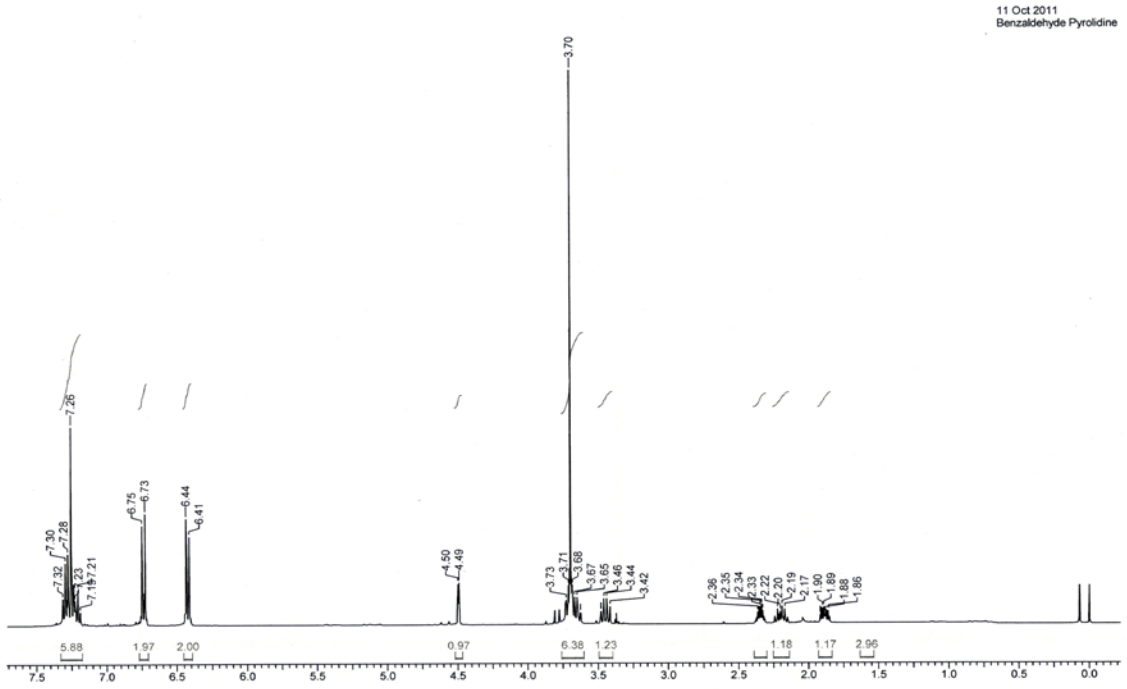
Racemic **6j**

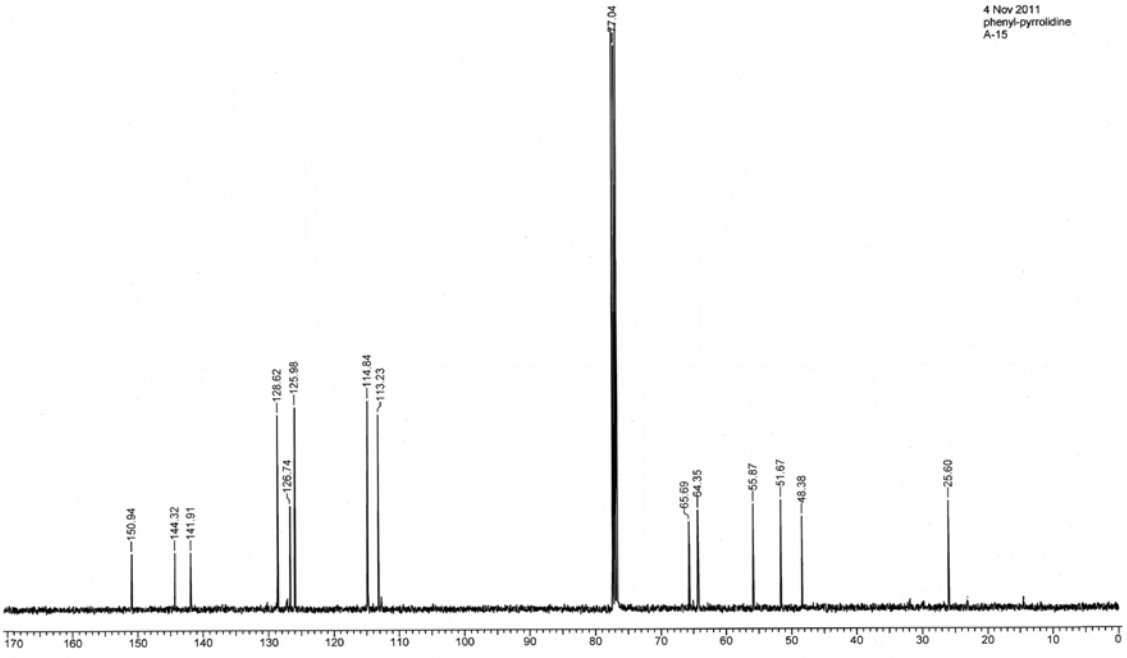
Retention Time	Area	Area %
12.280	9010109	51.50
34.362	8484488	48.50
Totals	17494597	100.00

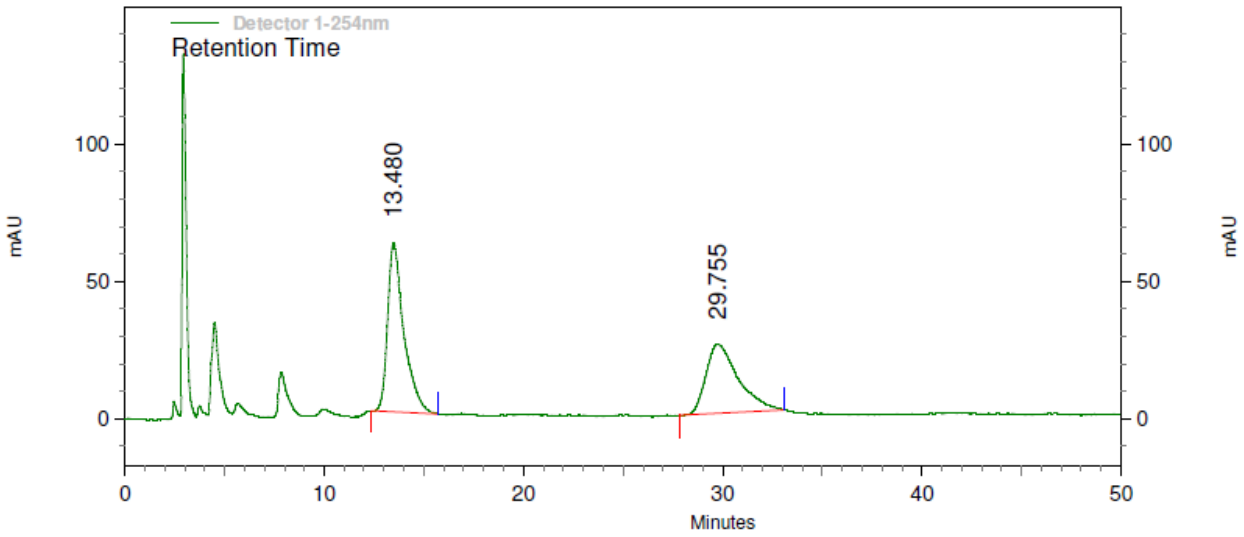


Chiral **6j** (92% ee)

Retention Time	Area	Area %
12.560	1478993	4.12
33.893	34418894	95.88
Totals	35897887	100.00

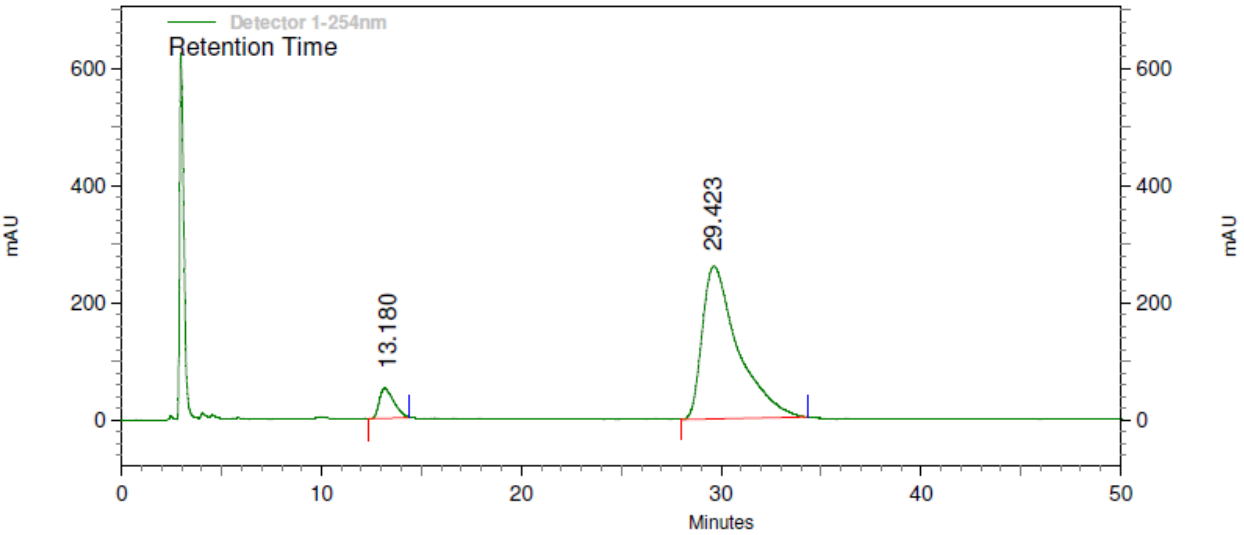

11 Oct 2011
Benzaldehyde Pyrolidine
3.70
7.26
7.30
7.28
7.32
7.23
7.19
7.21
6.75
6.73
6.44
6.41
4.50
4.49
3.73
3.71
3.68
3.67
3.65
3.46
3.44
3.42
2.36
2.35
2.34
2.33
2.22
2.20
2.19
2.17
1.90
1.89
1.88
1.86
5.88
1.97
2.00
0.97
6.38
1.23
1.18
1.17
2.96


4 Nov 2011
phenyl-pyrrolidine
A-15
47.04
128.62
125.98
126.74
114.84
113.23
150.94
144.32
141.91
65.69
64.35
55.87
51.67
48.38
25.60



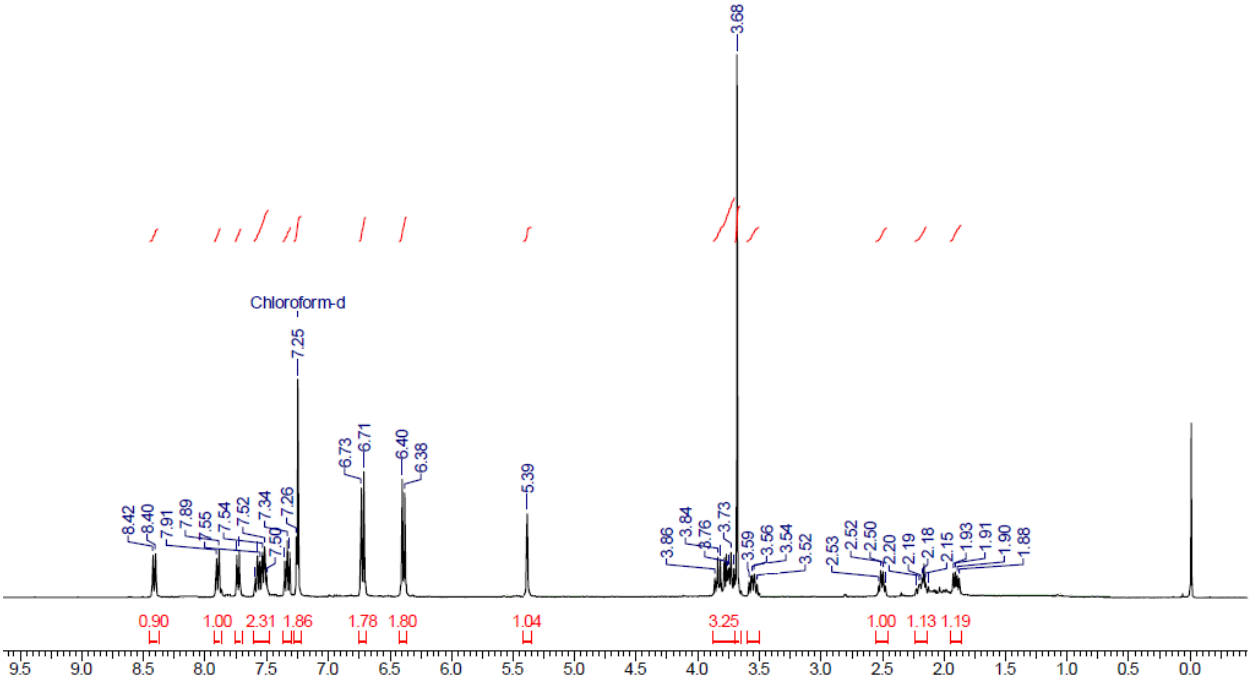
Racemic **6k**

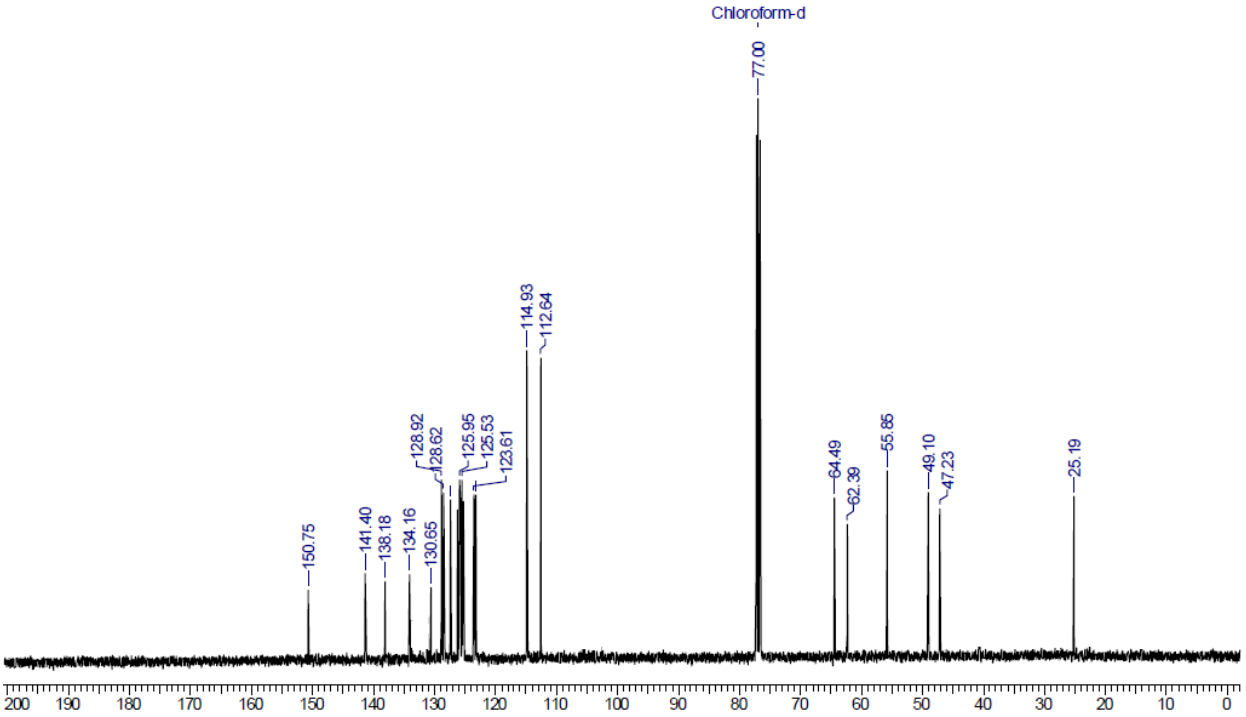
Retention Time	Area	Area %
13.480	3472072	55.06
29.755	2833472	44.94
Totals	6305544	100.00

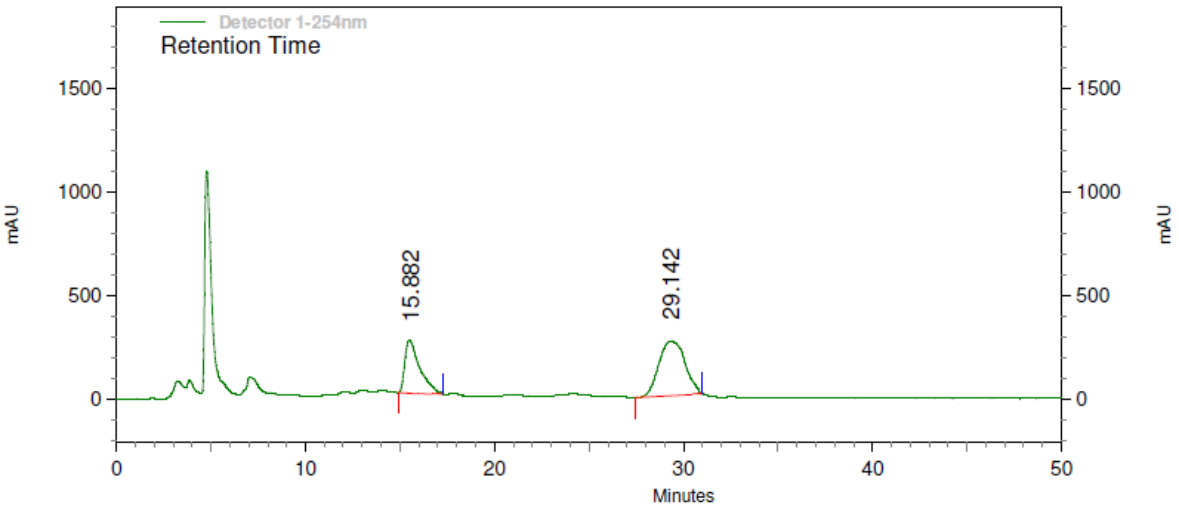


Chiral **6k** (90% ee)

Retention Time	Area	Area %
13.180	394659	4.96
29.423	7562175	95.04
Totals	7956834	100.00

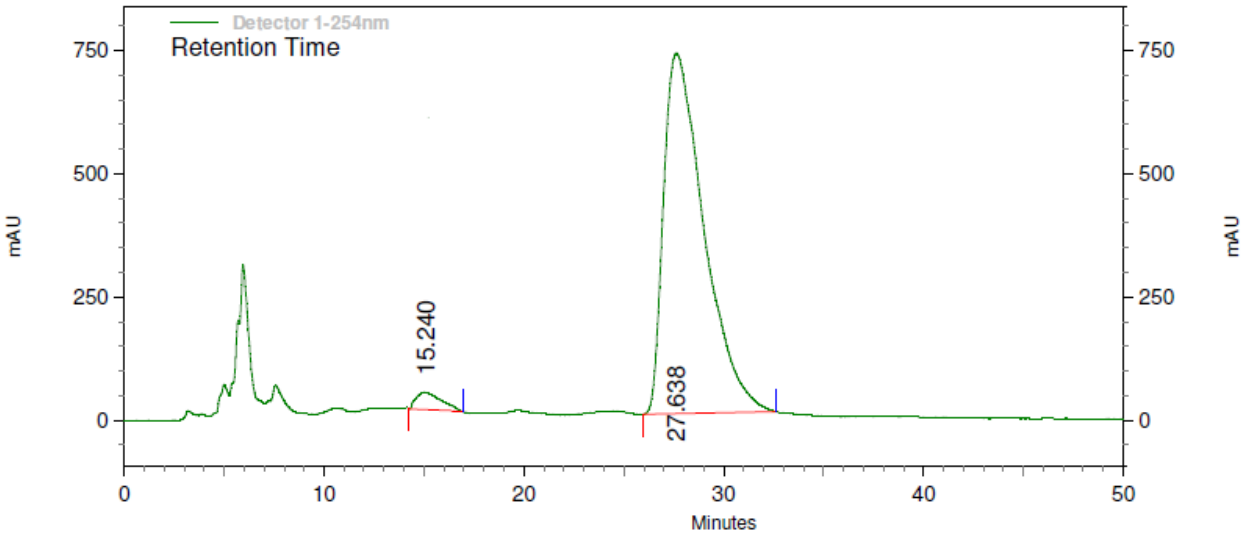

Chloroform-d
8.42
8.40
7.91
7.89
7.55
7.54
7.52
7.34
7.26
7.25
6.73
6.71
6.40
6.38
5.39
3.86
3.84
3.76
3.73
3.59
3.56
3.54
3.52
2.53
2.52
2.50
2.20
2.19
2.18
2.15
1.93
1.91
1.90
1.88
0.90
1.00
2.31
1.86
1.78
1.80
1.04
3.25
1.00
1.13
1.19


Chloroform-d
77.00
150.75
141.40
138.18
134.16
130.65
128.92
128.62
125.95
125.53
123.61
114.93
112.64
64.49
62.39
55.85
49.10
47.23
25.19



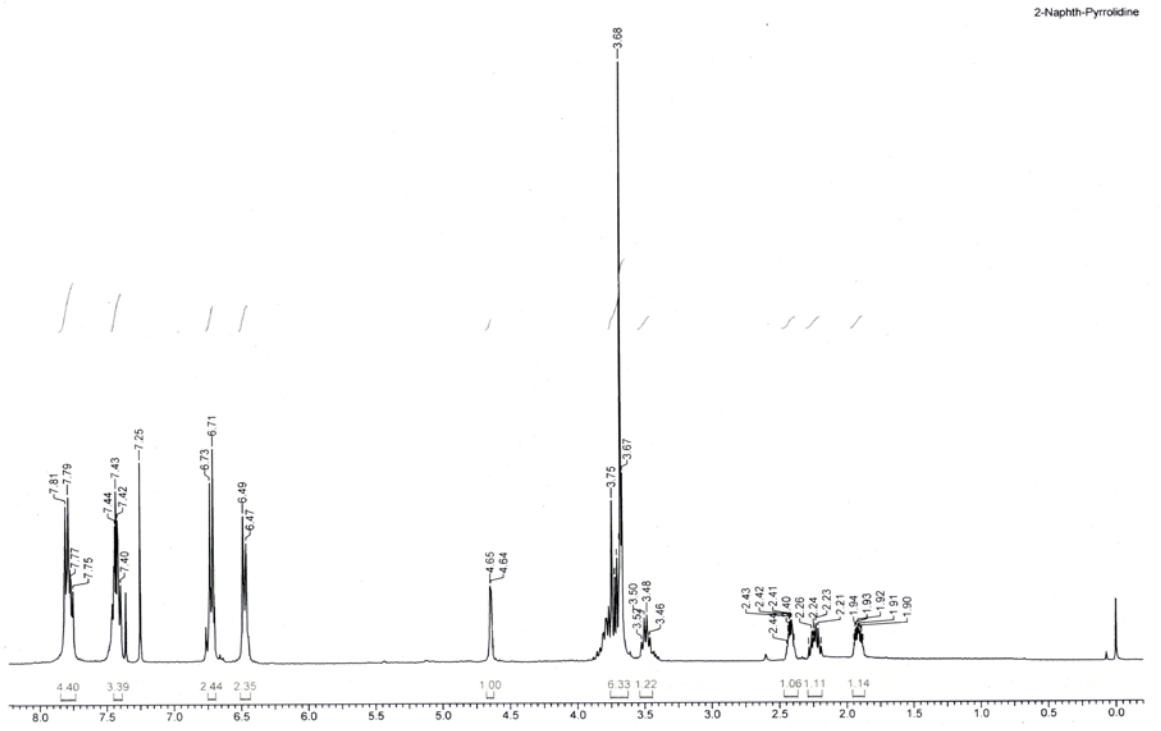
Racemic **6l**

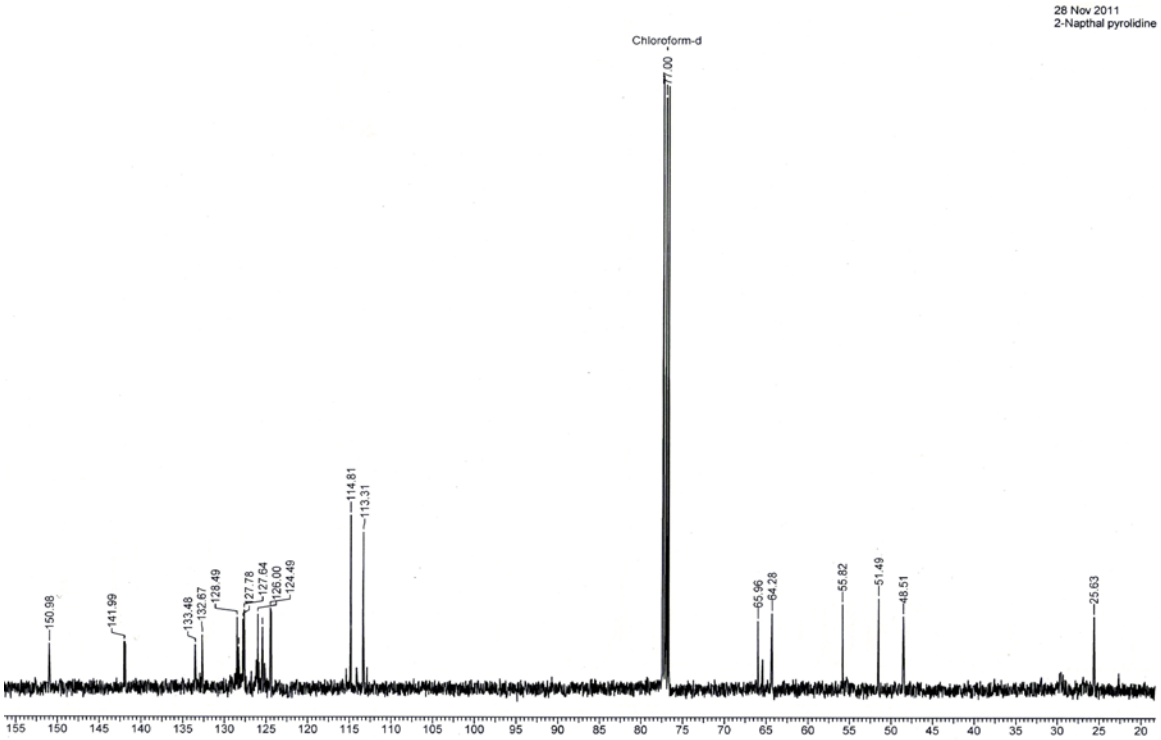
Retention Time	Area	Area %
15.882	1871693	39.85
29.142	2825152	60.15
Totals	4696845	100.00

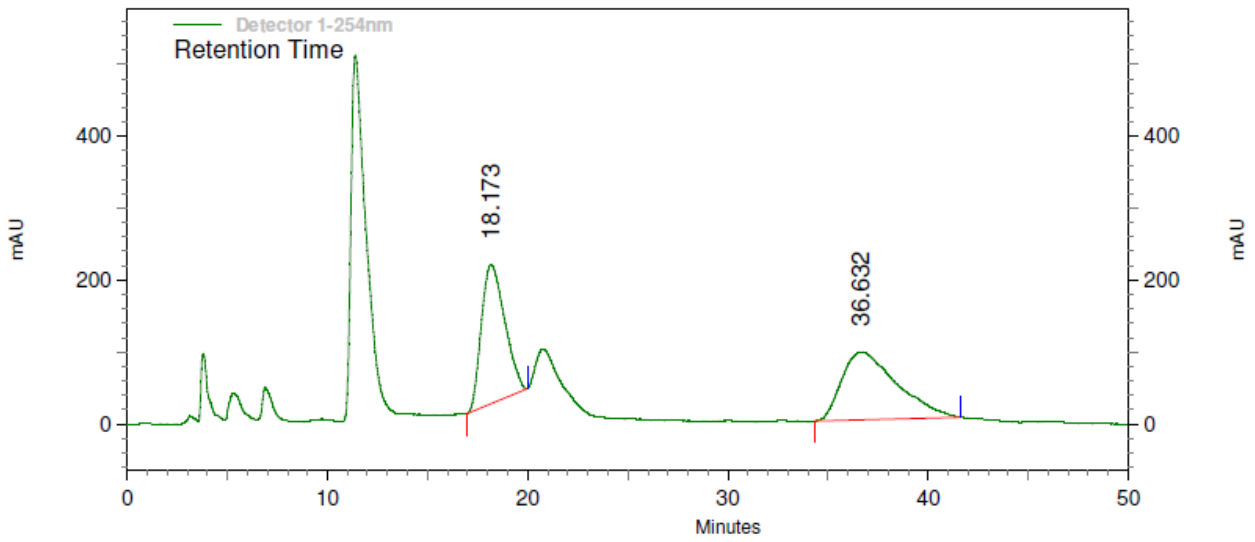


Chiral **6l** (97% ee)

Retention Time	Area	Area %
15.240	1601933	1.56
27.638	101086098	98.44
Totals	102688031	100.00

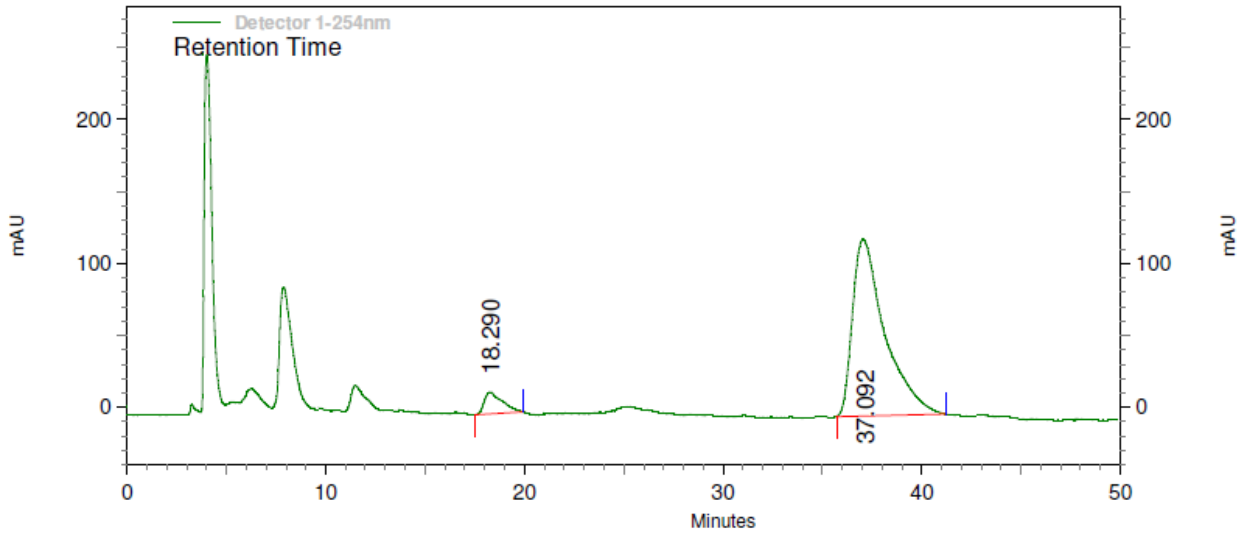

2-Naphth-Pyrrolidine
7.81
7.79
7.77
7.75
7.44
7.43
7.42
7.40
7.25
6.73
6.71
6.49
6.47
4.65
4.64
3.68
3.67
3.75
3.50
3.48
3.46
2.43
2.42
2.41
2.40
2.26
2.24
2.23
2.21
1.94
1.93
1.92
1.91
1.90
4.40
3.39
2.44
2.35
1.00
6.33
1.22
1.06
1.11
1.14


28 Nov 2011
2-Napthal pyrolidine
Chloroform-d
77.00
114.81
113.31
150.98
141.99
133.48
132.67
128.49
127.78
127.64
126.00
124.49
65.96
64.28
55.82
51.49
48.51
25.63



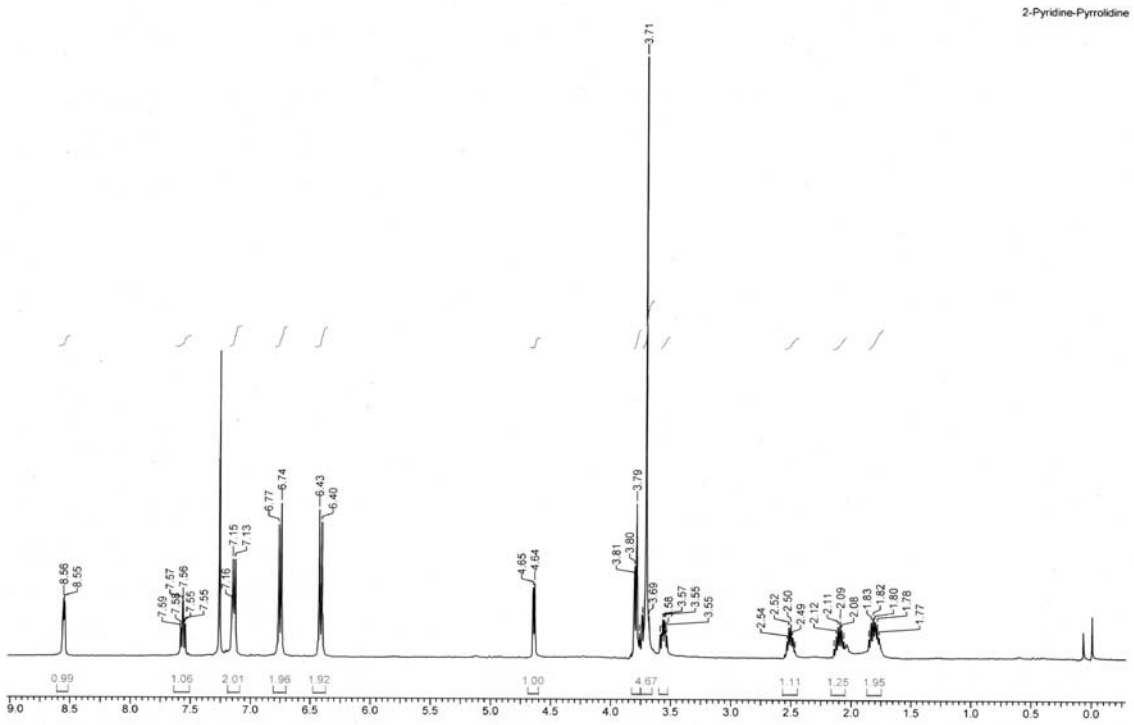
Racemic **6m**

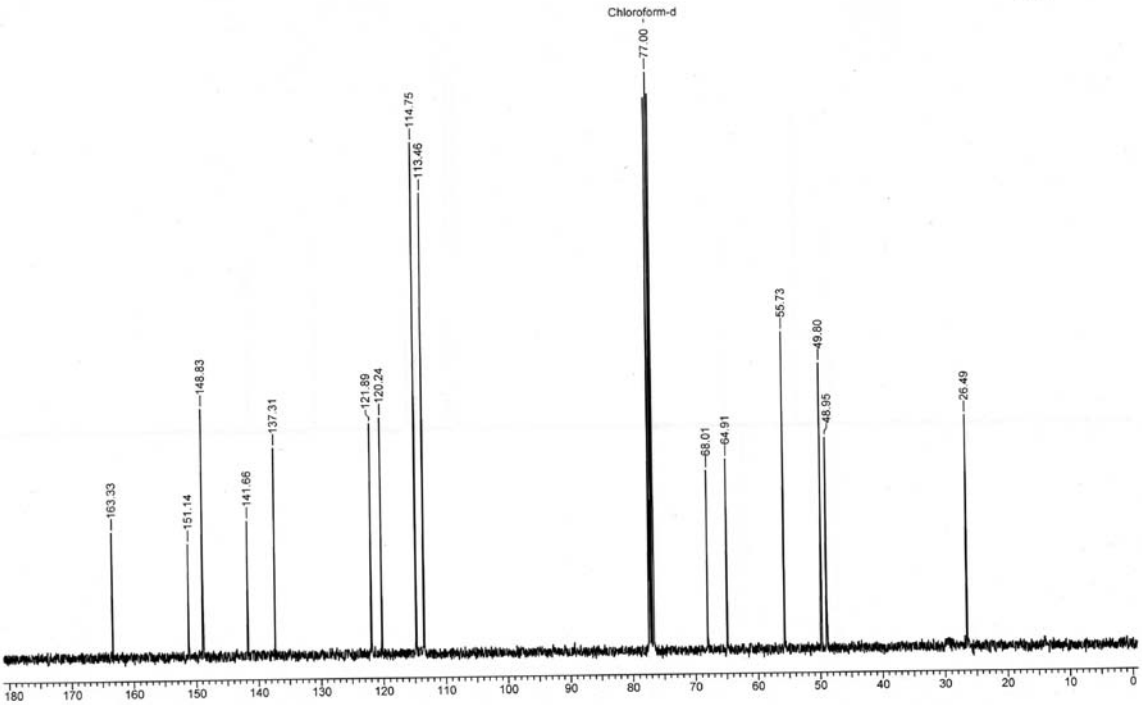
Retention Time	Area	Area %
18.173	15698049	46.68
36.632	17931841	53.32
Totals	33629890	100.00

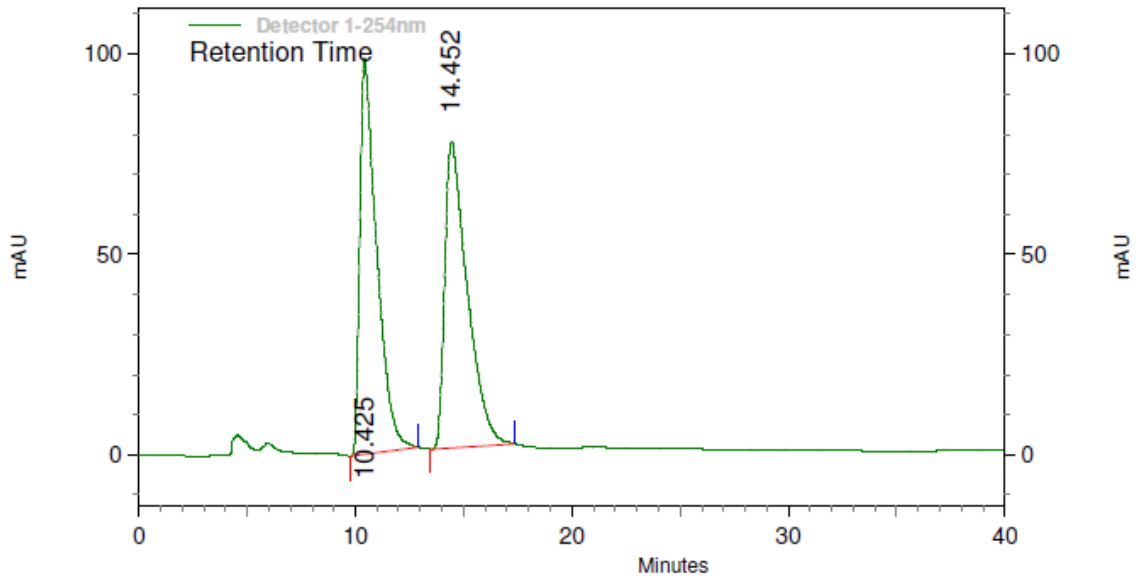


Chiral **6m** (90% ee)

Retention Time	Area	Area %
18.290	618572	5.15
37.092	11392527	94.85
Totals	12011099	100.00

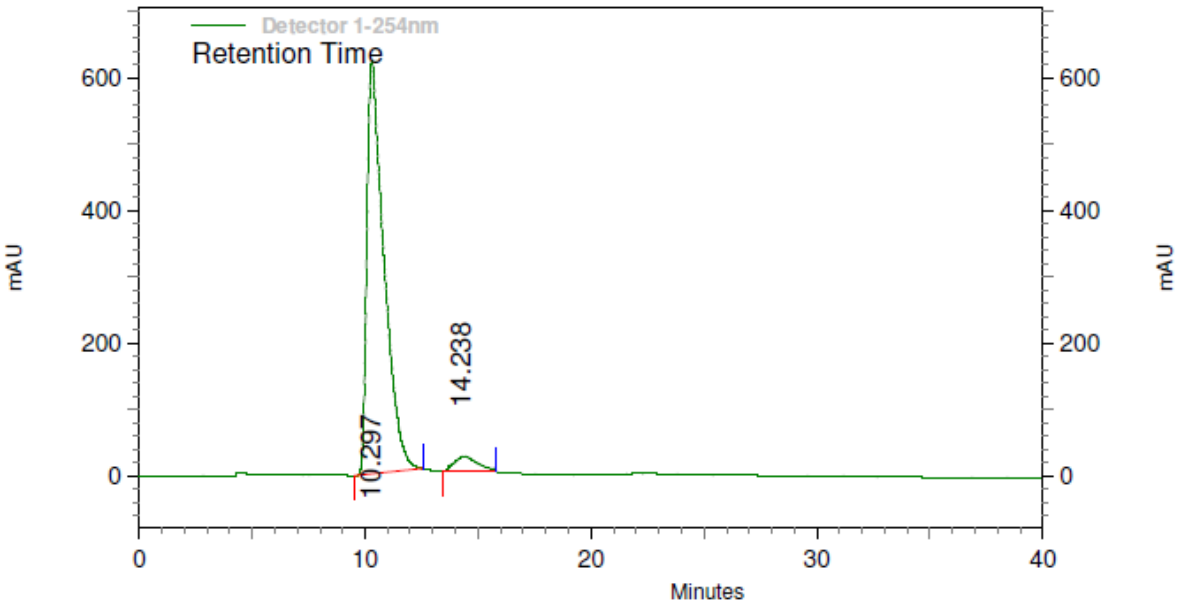

2-Pyridine-Pyrrolidine
8.56
8.55
7.59
7.58
7.57
7.56
7.55
7.16
7.15
7.13
6.77
6.74
6.43
6.40
4.65
4.64
3.81
3.80
3.79
3.71
3.69
3.58
3.57
3.55
3.55
2.54
2.52
2.50
2.49
2.12
2.11
2.09
2.08
1.83
1.82
1.80
1.78
1.77
0.99
1.06
2.01
1.96
1.92
1.00
4.67
1.11
1.25
1.95


4 Nov 2011
2-pyridine-pyrrolidine
A-16
Chloroform-d
77.00
163.33
151.14
148.83
141.66
137.31
121.89
120.24
114.75
113.46
68.01
64.91
55.73
49.80
48.95
26.49



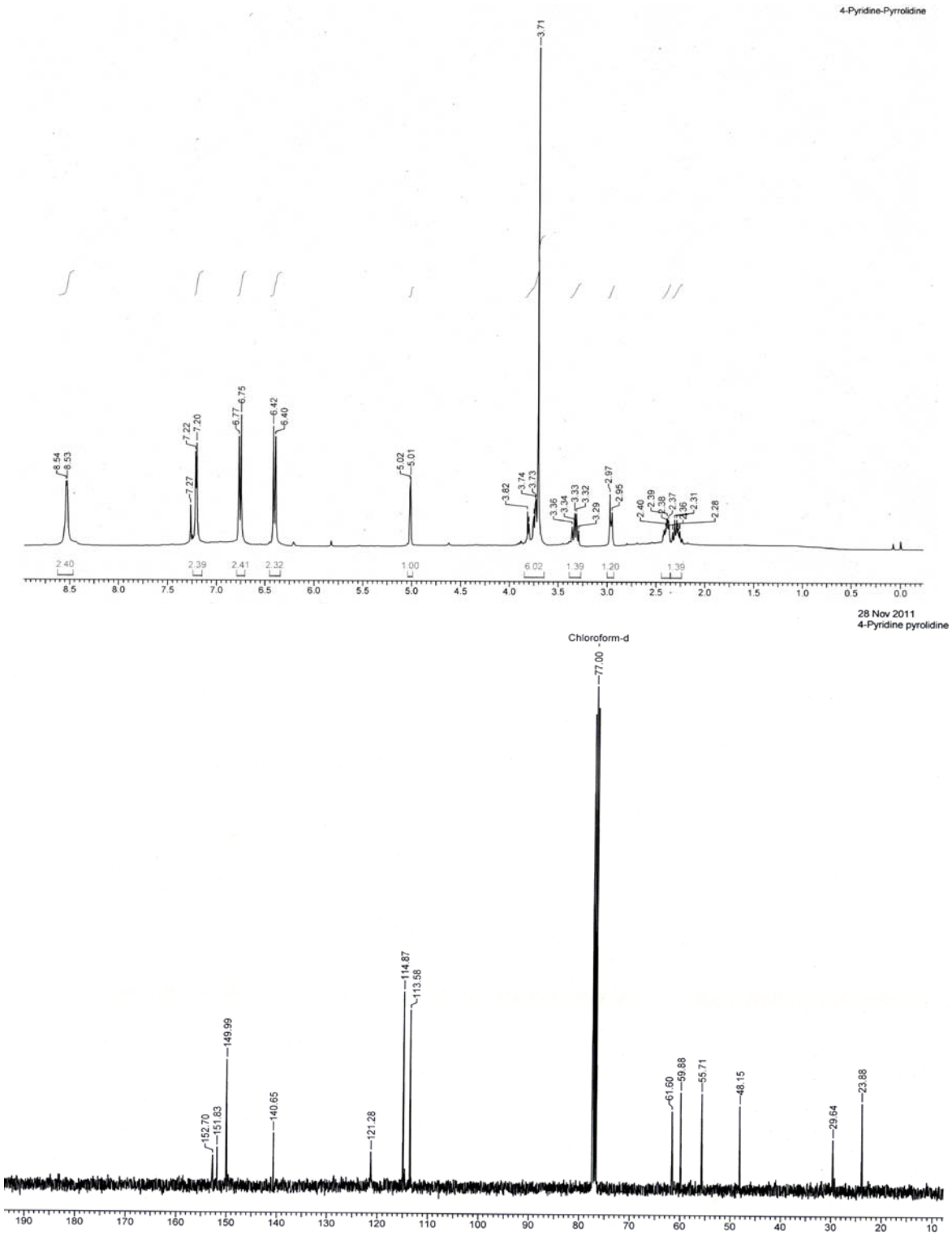
Racemic **6n**

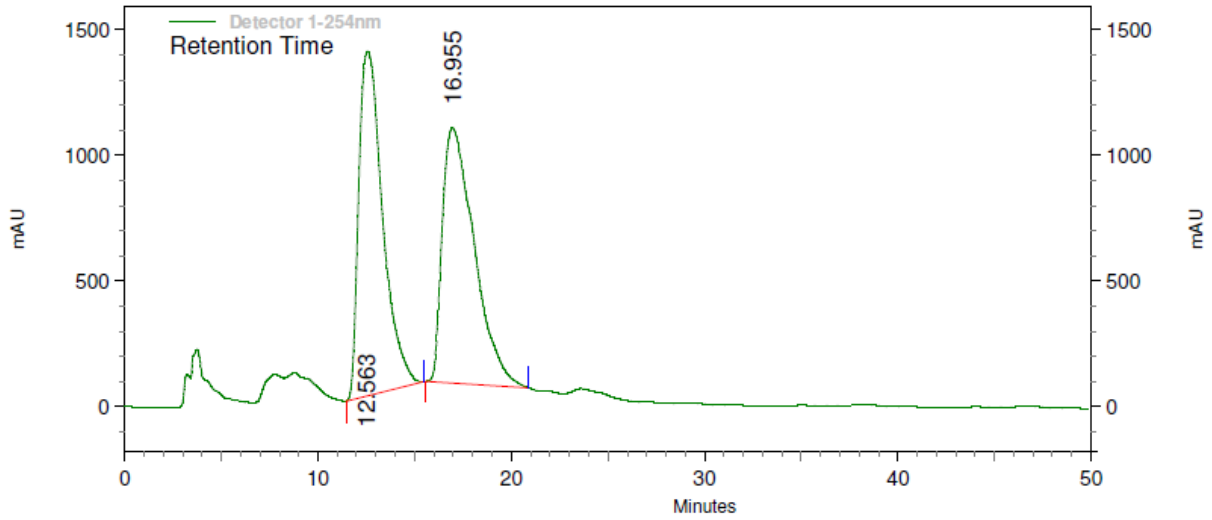
Retention Time	Area	Area %
10.425	5363544	49.25
14.452	5528112	50.25
Totals	10891656	100.00



Chiral **6n** (95% ee)

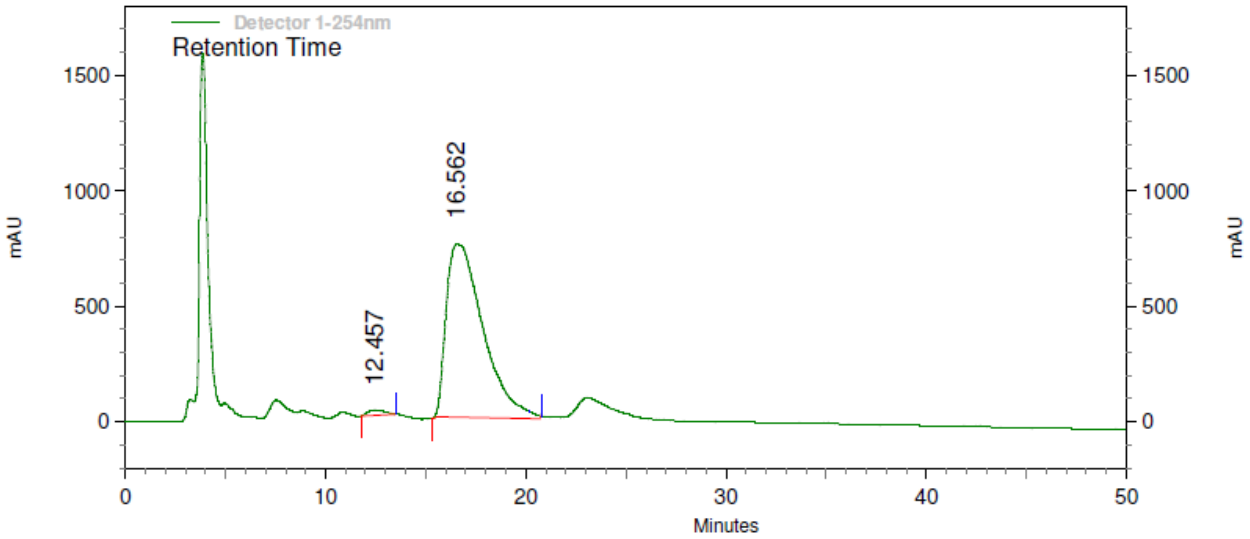
Retention Time	Area	Area %
10.297	35398921	97.49
14.238	911389	2.51
Totals	36310310	100.00


4-Pyridine-Pyrrolidine
8.54
8.53
7.27
7.22
7.20
6.77
6.75
6.42
6.40
5.02
5.01
3.82
3.74
3.73
3.71
3.36
3.34
3.33
3.32
3.29
2.97
2.95
2.40
2.39
2.38
2.37
2.36
2.31
2.28
2.40
2.39
2.41
2.32
1.00
6.02
1.39
1.20
1.39
28 Nov 2011
4-Pyridine pyrolidine
Chloroform-d
77.00
152.70
151.83
149.99
140.65
121.28
114.87
113.58
61.60
59.88
55.71
48.15
29.64
23.88



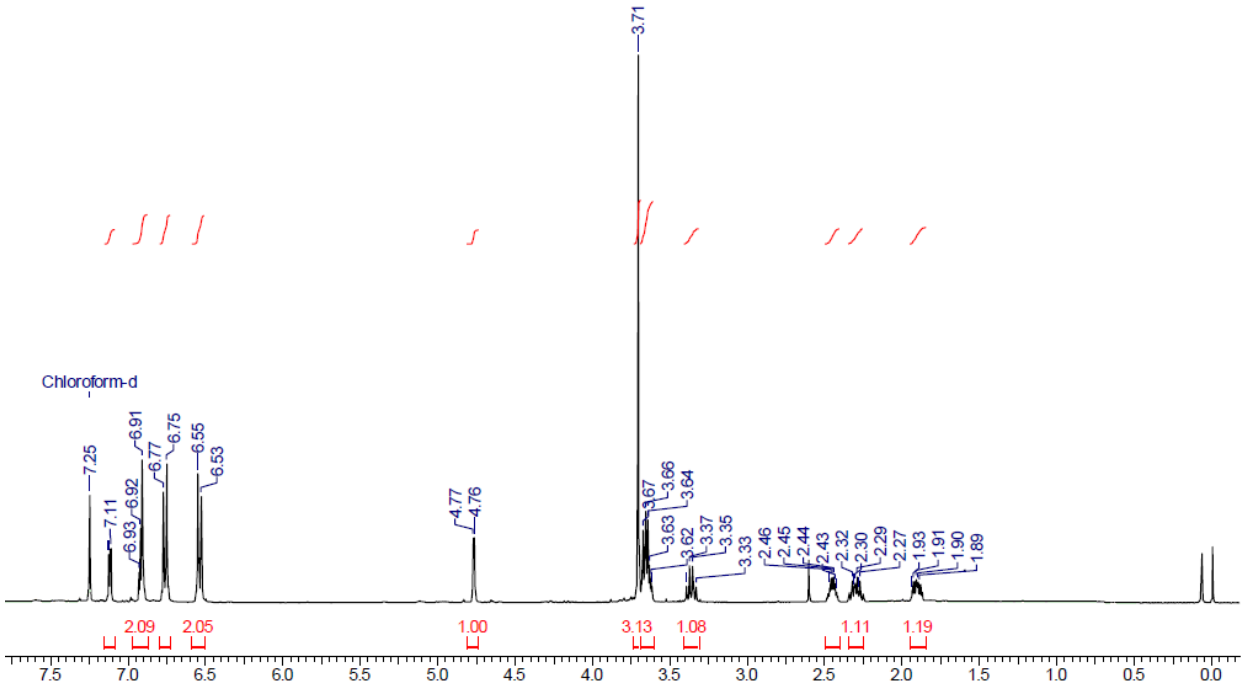
Racemic **6o**

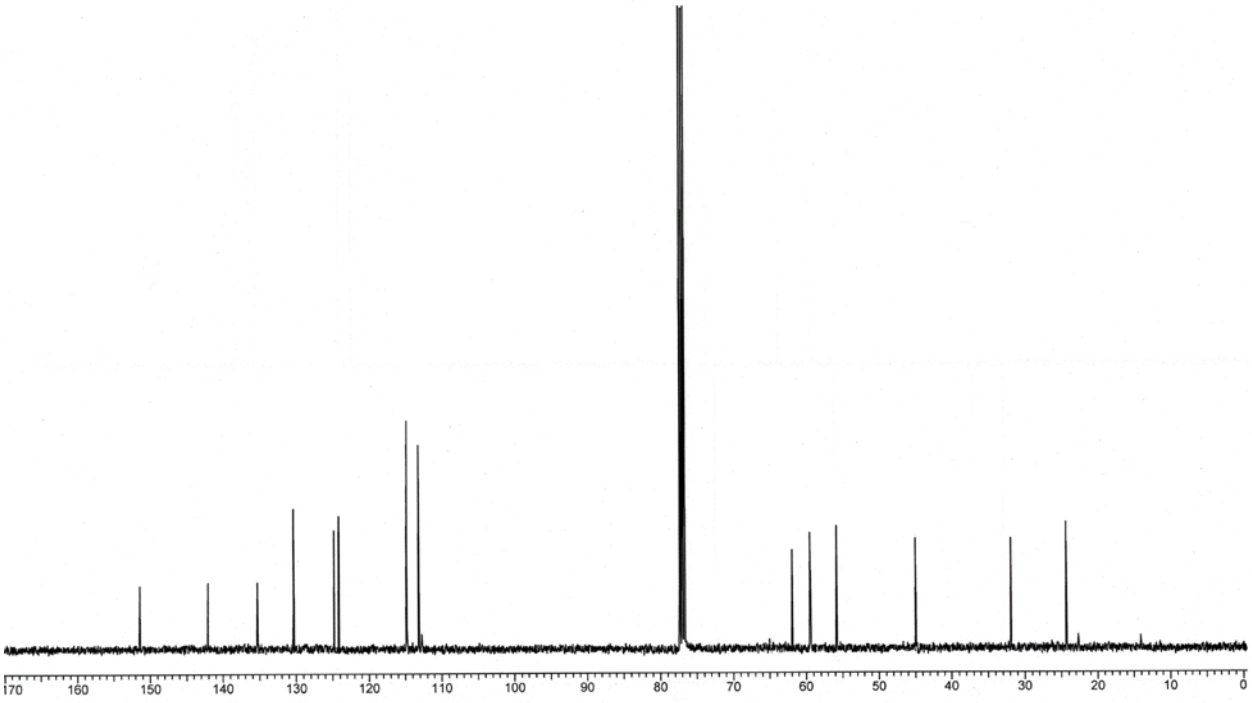
Retention Time	Area	Area %
12.563	118139288	50.28
16.955	116802635	49.72
Totals	234941923	100.00

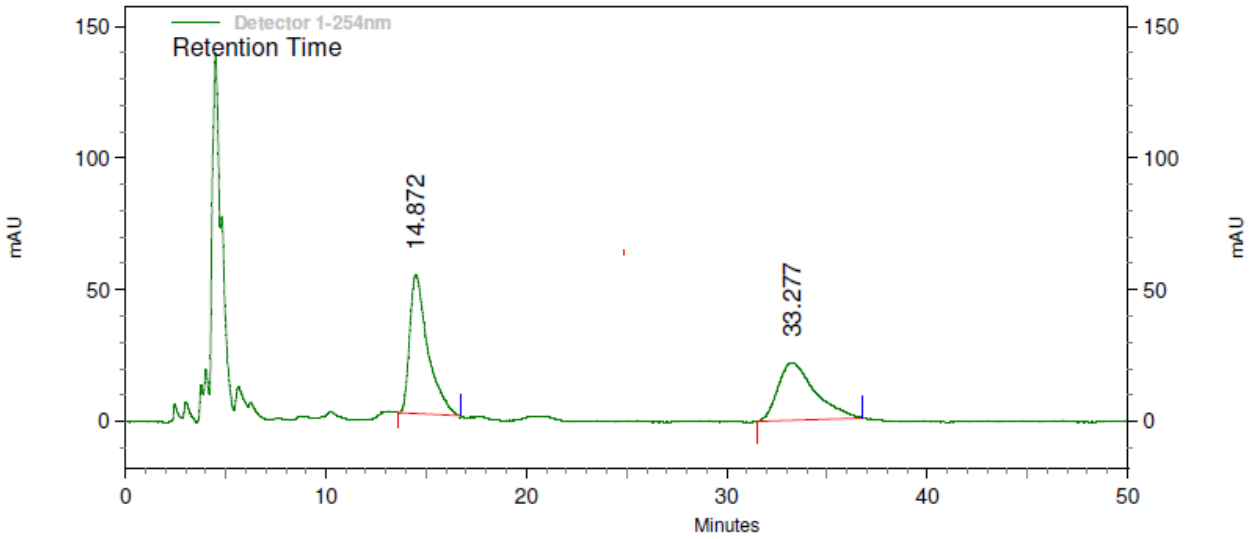


Chiral **6o** (98% ee)

Retention Time	Area	Area %
12.457	92463509	98.92
16.562	1009509	1.08
Totals	93473018	100.00

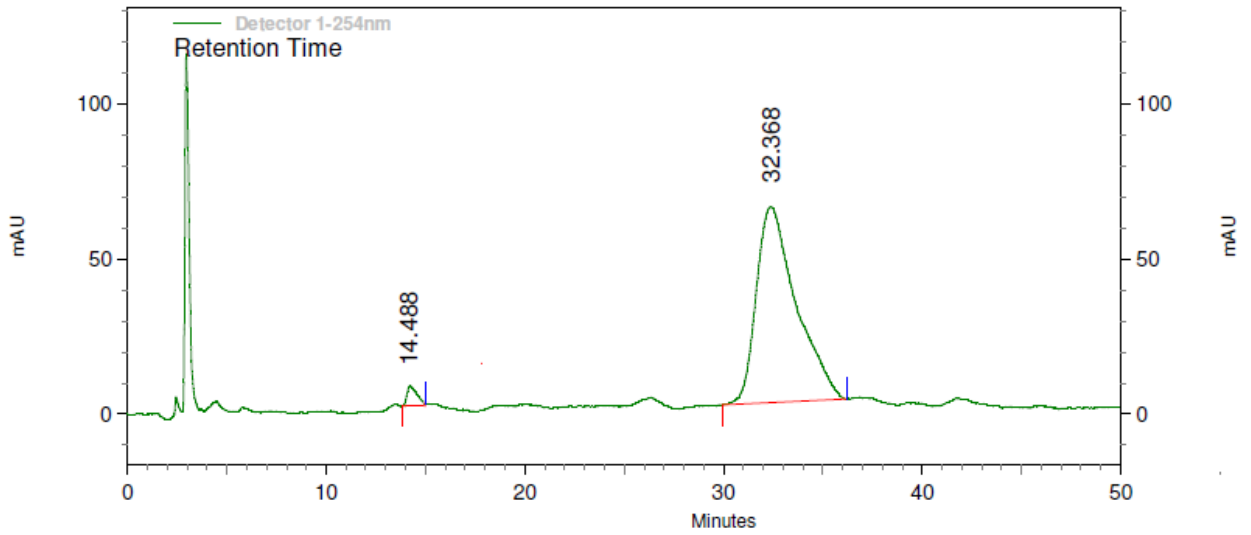






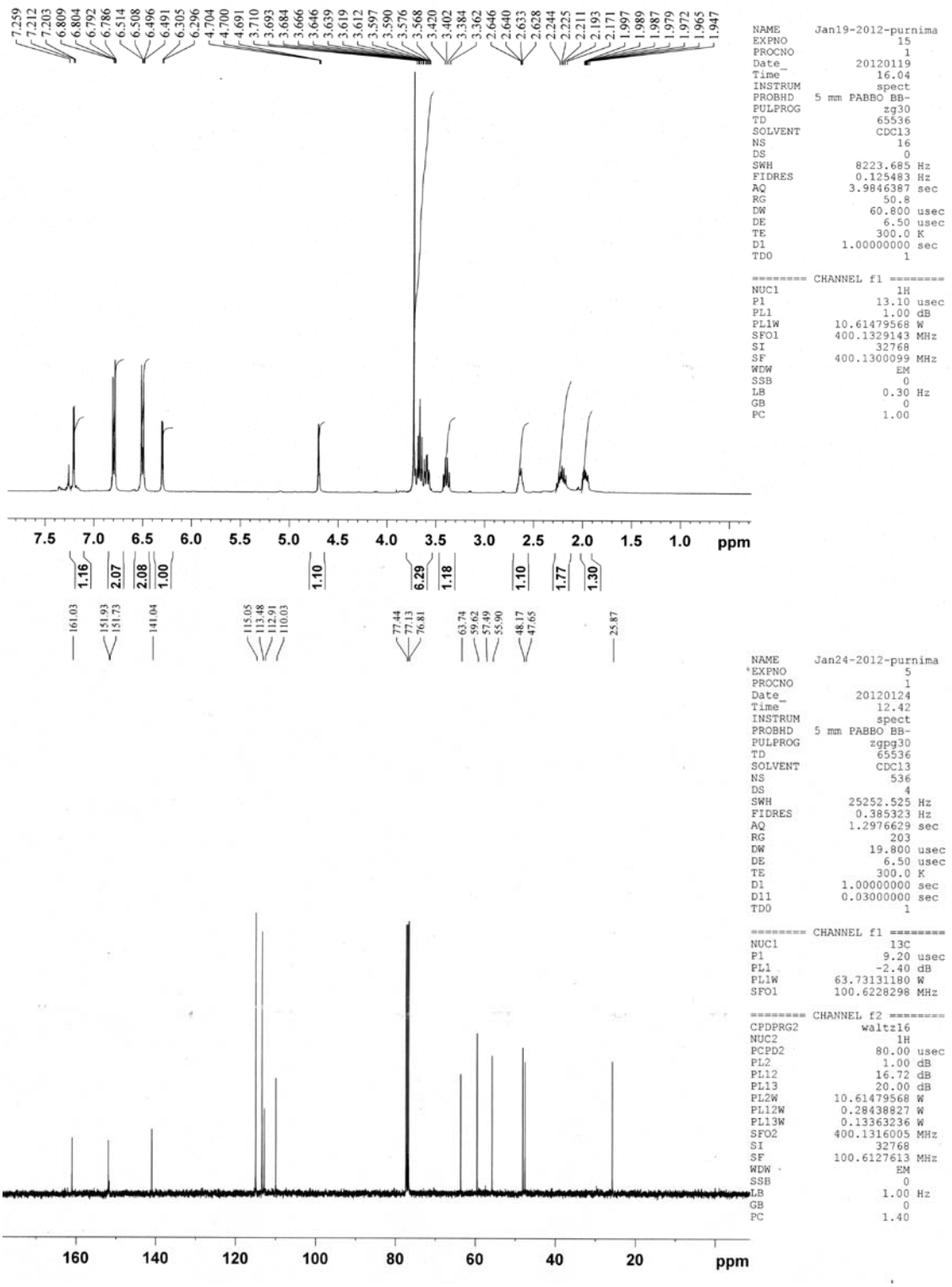
Racemic **6p**

Retention Time	Area	Area %
14.872	3009563	47.45
33.277	3333035	52.55
Totals	6342598	100.00



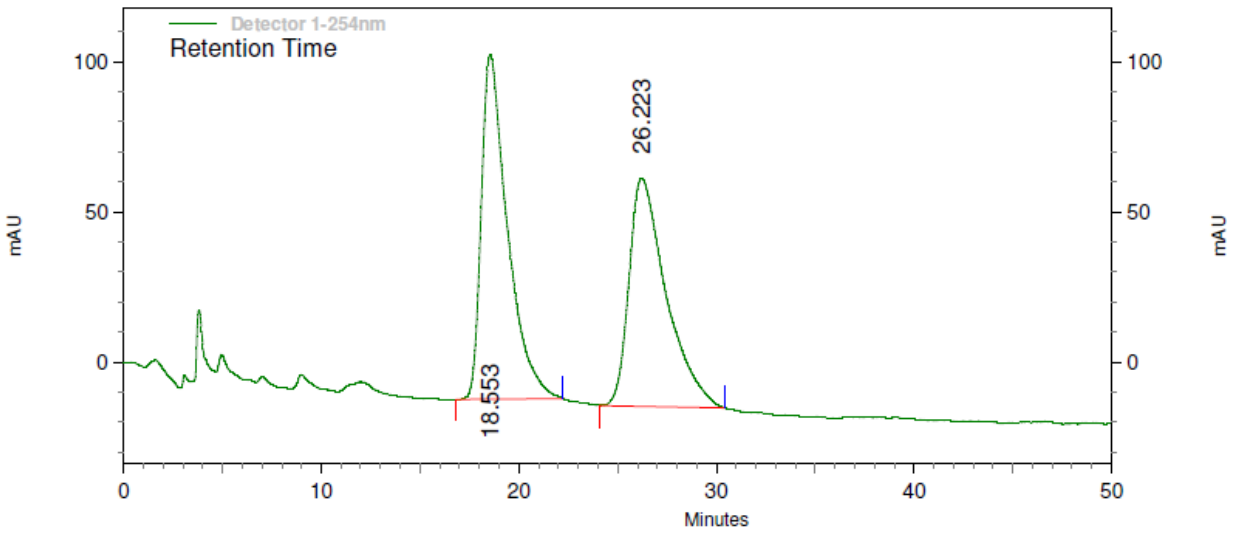
Chiral **6p** (95% ee)

Retention Time	Area	Area %
14.488	7896058	97.56
32.368	197482	2.44
Totals	8093540	100.00



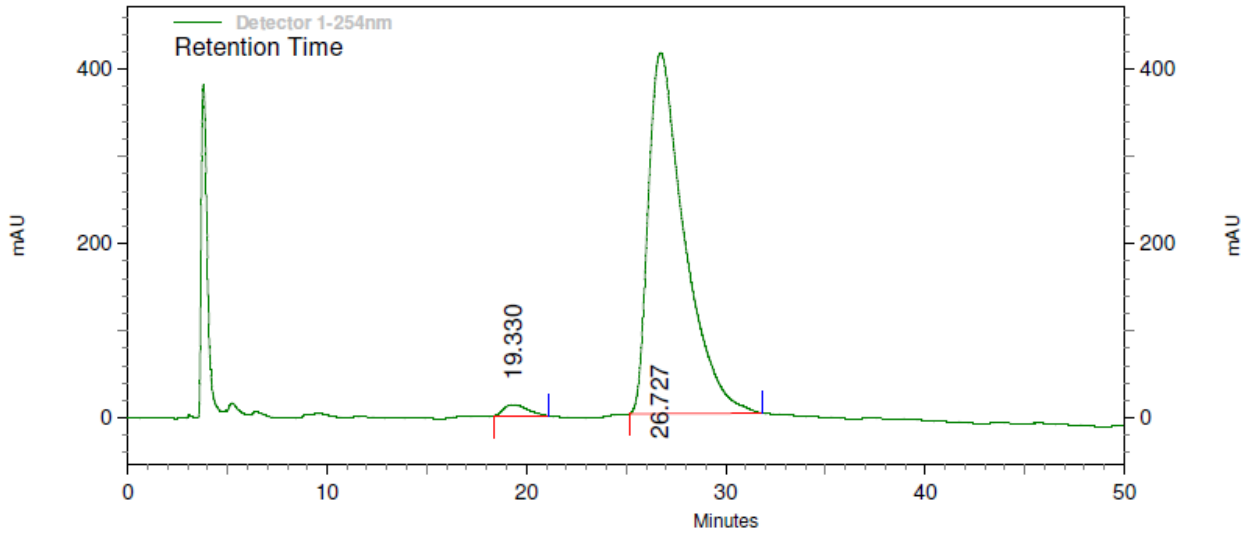
NAME	Jan19-2012-purnima
EXPNO	15
PROCNO	1
Date_	20120119
Time	16.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	50.8
DW	60.800 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	13.10 usec
PL1	1.00 dB
PL1W	10.61479568 W
SFO1	400.1329143 MHz
SI	32768
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

NAME	Jan24-2012-purnima
EXPNO	5
PROCNO	1
Date_	20120124
Time	12.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	536
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	203
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	13C
P1	9.20 usec
PL1	-2.40 dB
PL1W	63.73131180 W
SFO1	100.6228298 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.72 dB
PL13	20.00 dB
PL2W	10.61479568 W
PL12W	0.28438827 W
PL13W	0.13363236 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127613 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



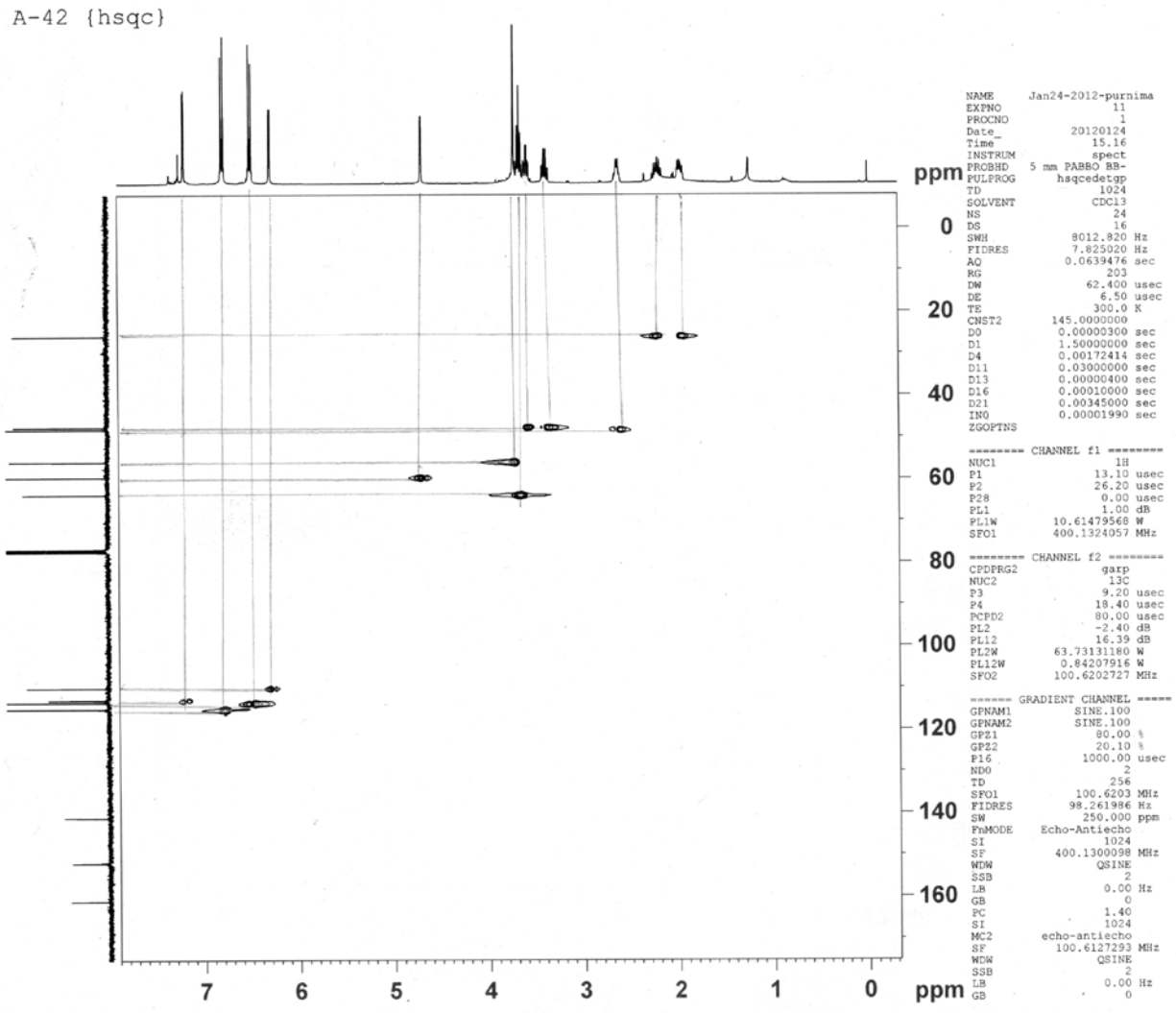
Racemic **6q**

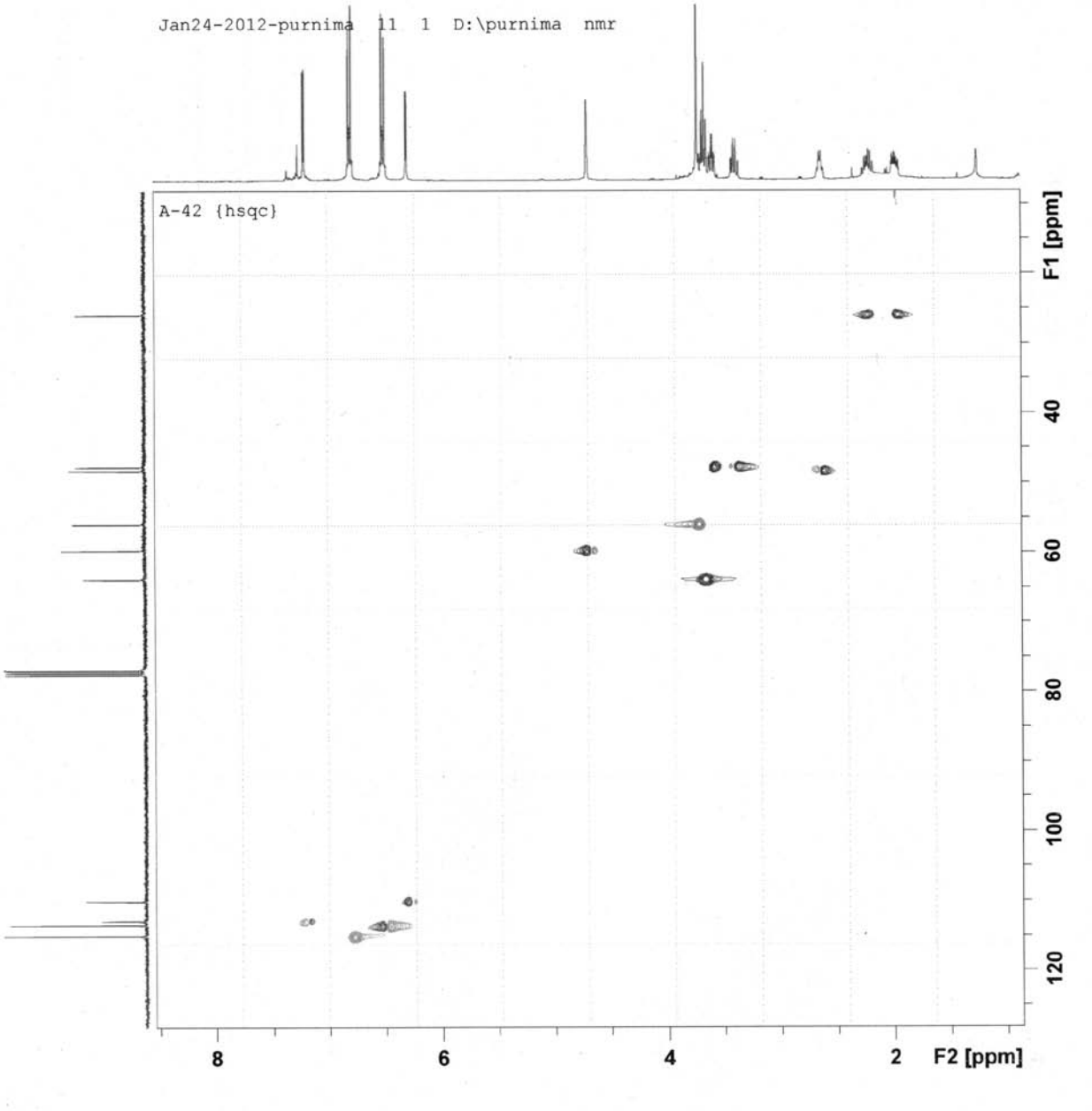
Retention Time	Area	Area %
18.553	10595640	51.65
26.223	9919950	48.35
Totals	20515590	100.00

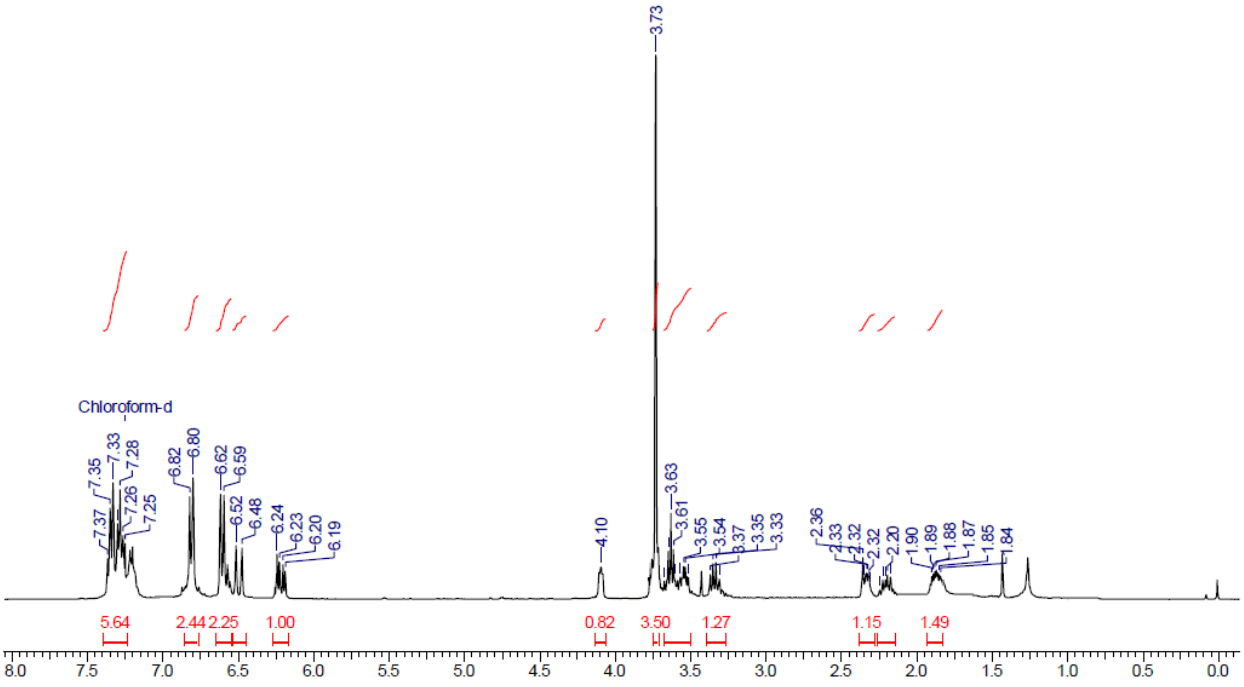


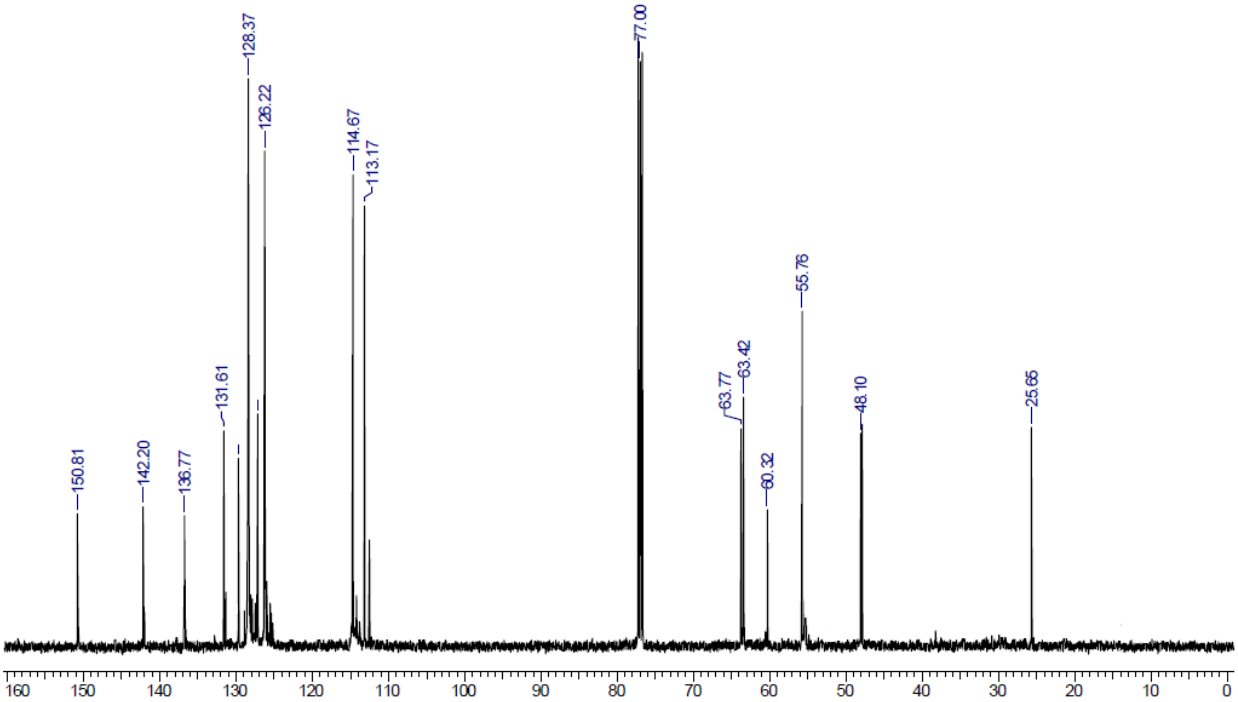
Chiral **6q** (96% ee)

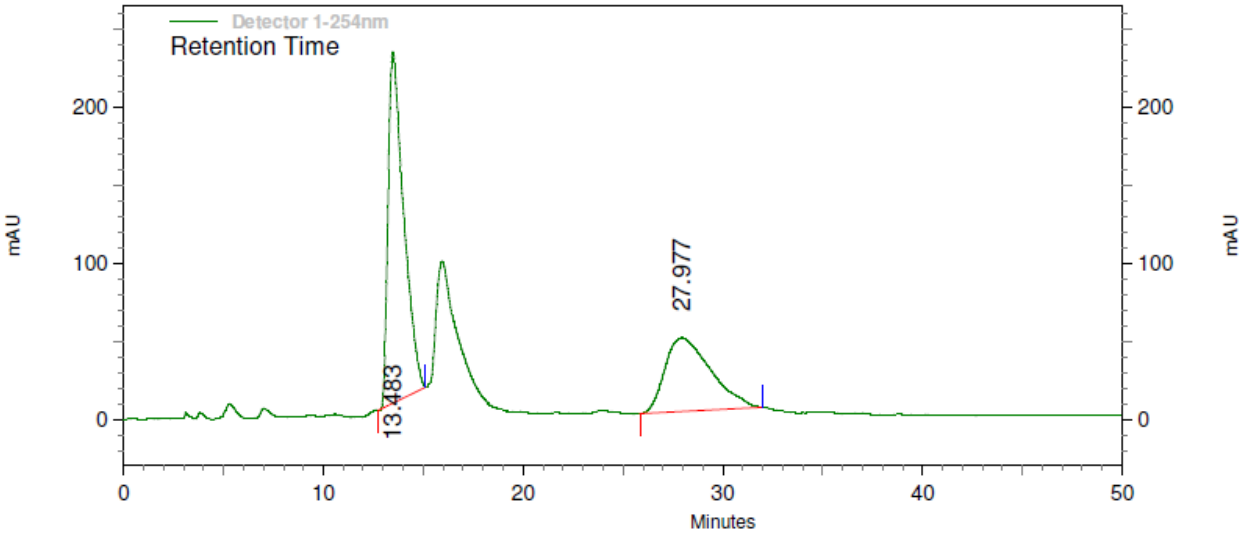
Retention Time	Area	Area %
19.330	1095784	2.06
26.727	52024498	97.94
Totals	53120282	100.00


A-42 {hsqc}
ppm
0
20
40
60
80
100
120
140
160
7
6
5
4
3
2
1
0
ppm
NAME Jan24-2012-purnima
EXPNO 11
PROCNO 1
Date_ 20120124
Time 15.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hsqcedetgp
TD 1024
SOLVENT CDCl3
NS 24
DS 16
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639476 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00010000 sec
D21 0.00345000 sec
IN0 0.00001990 sec
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 13.10 usec
P2 26.20 usec
P28 0.00 usec
PL1 1.00 dB
PL1W 10.61479568 W
SFO1 400.1324057 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 9.20 usec
P4 18.40 usec
PCPD2 80.00 usec
PL2 -2.40 dB
PL12 16.39 dB
PL2W 63.73131180 W
PL12W 0.84207916 W
SFO2 100.6202727 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec
ND0 2
TD 256
SFO1 100.6203 MHz
FIDRES 98.261986 Hz
SW 250.000 ppm
FnMODE Echo-Antiecho
SI 1024
SF 400.1300098 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 echo-antiecho
SF 100.6127293 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0


Jan24-2012-purnima 11 1 D:\purnima nmr
A-42 {hsqc}
F1 [ppm]
40
60
80
100
120
8
6
4
2
F2 [ppm]

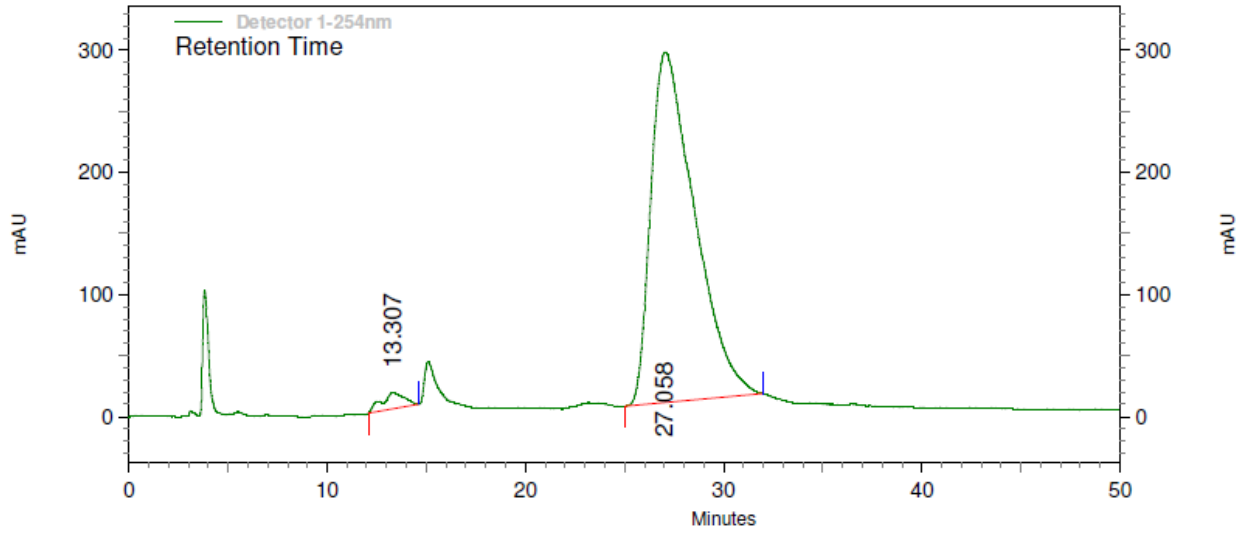

Chloroform-d
7.37
7.35
7.33
7.28
7.26
7.25
6.82
6.80
6.62
6.59
6.52
6.48
6.24
6.23
6.20
6.19
4.10
3.73
3.63
3.61
3.55
3.54
3.37
3.35
3.33
2.36
2.33
2.32
2.32
2.20
1.90
1.89
1.88
1.87
1.85
1.84
5.64
2.44
2.25
1.00
0.82
3.50
1.27
1.15
1.49


150.81
142.20
136.77
131.61
128.37
126.22
114.67
113.17
77.00
63.77
63.42
60.32
55.76
48.10
25.66



Racemic **6r**

Retention Time	Area	Area %
13.483	11589475	58.75
27.977	8137291	41.25
Totals	19726766	100.00



Chiral **6r** (95% ee)

Retention Time	Area	Area %
13.307	1049571	2.33
27.058	44008627	97.67
Totals	45058198	100.00