

**Asymmetric Reduction of Imines with Trichlorosilane  
Catalyzed by Valine-derived Formamide Immobilized onto  
Magnetic Nano-Fe<sub>3</sub>O<sub>4</sub>**

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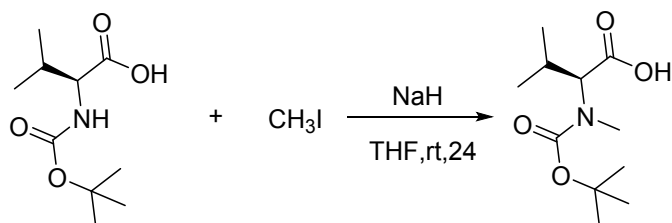
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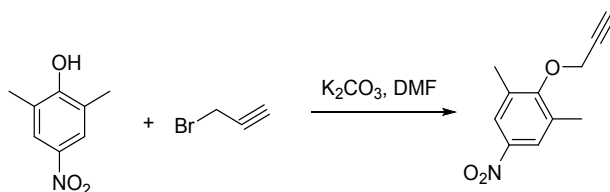
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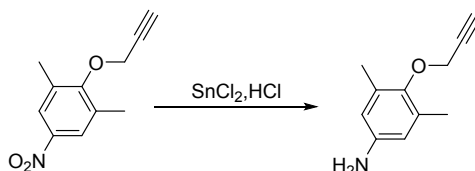
## General experimental procedure for the preparation of organocatalysts 4



Neat sodium hydride (240mg, 10mmol) was added to a cooled (0 °C) solution of N-BOC-valine (217mg, 1 mmol) and iodomethane (1.42g, 10mmol) in anhydrous THF (20 mL). The reaction mixture was stirred at room temperature for 24 h. Then quenched with water (30 mL). After the reaction mixture was extracted with EtOAc (2\*15 mL), the aqueous solution was acidified to pH 3. It extracted with EtOAc (3\*20 mL). The combined organic phase was dried over MgSO<sub>4</sub> and evaporated to afford the corresponding N-methylated product as a thick colorless oil (230mg, yield 99%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 10.04 (s, 1H), 4.49 – 3.93 (m, 1H), 2.87 (s, 3H), 2.23 (dd, *J* = 16.3, 9.9 Hz, 1H), 1.47 (s, 9H), 1.03 (d, *J* = 6.5 Hz, 3H), 0.92 (d, *J* = 6.7 Hz, 3H).

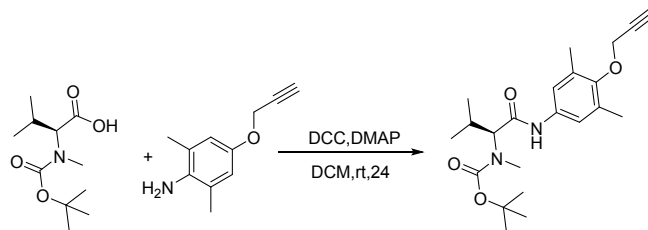


A mixture of phenol (167mg, 1 mmol) and anhydrous potassium carbonate (414 mg, 3 mmol) in dry DMF (15 mL) was heated to 60 °C for half an hour. The mixture was then cooled to room temperature and propargyl bromide (80 wt% toluene solution, 175 g, 1.2 mmol) was added. The mixture was stirred for 4 hours at 60 °C and poured in the ice water with stirring. Stirring continued for 10 minutes, solid separated was filtered and dried under vacuum to afford the desired compound (yield 93%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.85 (s, 2H), 4.54 (d, *J* = 2.4 Hz, 2H), 2.46 (t, *J* = 2.3 Hz, 1H), 2.33 (s, 6H).

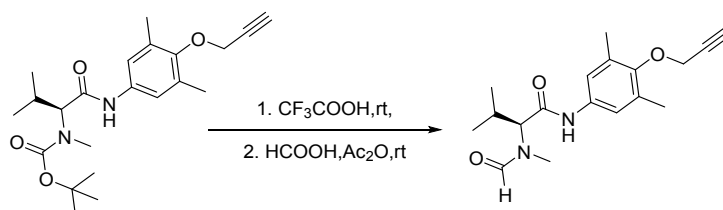


The above solid was dissolved in dioxane (5 mL) and the solution was cooled to 10 °C. A pre-cooled solution of stannous chloride dehydrate (677 mg, 3 mmol) in concentrated HCl (2 mL) was added dropwise and then the reaction mixture was stirred at room temperature for 30 hours. The solution was extracted with EtOAc (30\*3 mL). The organic layer was washed with brine (30 mL), and the combined organic phase was dried over MgSO<sub>4</sub>. The solvent was removed in vacuum, and the product was obtained (yield 87%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.33 (s, 2H), 4.41 (d, *J* = 2.3 Hz, 2H), 3.20 (s, 2H), 2.49 (t, *J* = 2.1 Hz, 1H), 2.23 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 147.97,

142.63, 131.81, 115.25, 79.71, 74.72, 60.05, 16.53.

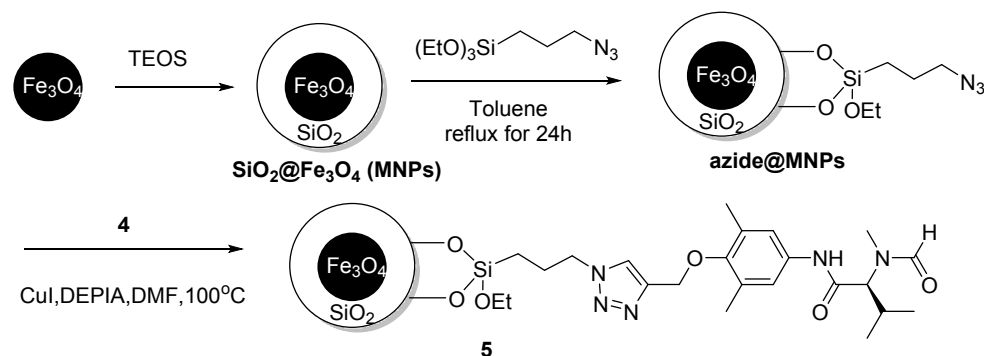


To a stirred solution of amine (175 mg, 1mmol) in  $\text{CH}_2\text{Cl}_2$  (100 mL) was added the (S)-2-(tert-Butoxycarbonyl-methyl-amino)-3-methyl-butanoic acid (231mg, 1 mmol), dicyclohexylcarbodiimide (DCC, 226 mg, 1.1 mmol) and 4-dimethylamino-pyridine (DMAP, 12 mg, 0.1 mmol). The reaction mixture was stirred at room temperature for 24 h. Afterwards the organic phase was filtered and evaporated under reduced pressure to give the crude product, which was purified by column chromatography through silica gel, eluting with 5:1 ethyl acetate/petroleum ether solvent mixture, to give the pure product. Yield 44%, yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.19 (br, 1H), 7.18 (s, 2H), 4.46 (d,  $J = 2.2$  Hz, 2H), 4.12 (d,  $J = 7.1$  Hz, 1H), 2.83 (s, 3H), 2.50 (t,  $J = 2.4$  Hz, 1H), 2.44 – 2.33 (m, 1H), 2.29 (s, 6H), 1.47 (s, 9H), 1.01 (d,  $J = 6.4$  Hz, 3H), 0.91 (d,  $J = 6.6$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.69, 157.41, 151.63, 134.12, 131.78, 120.11, 80.64, 79.26, 75.03, 65.99, 59.90, 30.44, 28.38, 25.98, 19.85, 18.58, 16.63.



The tert-butyl (S)-methyl(3-methyl-1-oxo-1-((4-(prop-2-yn-1-yloxy)phenyl)amino)butan-2-yl)carbamate (388 mg, 1 mmol) was dissolved in trifluoroacetic acid (2.0 mL) at room temperature. After 1 hour the reaction mixture was evaporated in vacuo, the residue was dissolved in formic acid (0.75 mL) and the resulting solution was cooled to 0 °C. Acetic anhydride (2 mL) was added dropwise and the mixture was allowed to stir at room temperature overnight. The solvent was then removed by reduced pressure. Purification using column chromatography on silica gel with a petroleum ether/ethyl acetate mixture (2:1) afforded the product. Yield 75%, Yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.40 (s, 1H), 8.14 (s, 1H), 7.21 (s, 2H), 4.47-4.41 (m, 3H), 3.02 (s, 3H), 2.50 (t,  $J = 2.2$  Hz, 1H), 2.47 – 2.38 (m, 1H), 2.28 (s, 6H), 1.04 (d,  $J = 6.4$  Hz, 3H), 0.91 (d,  $J = 6.6$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.18, 163.97, 151.84, 133.77, 131.79, 120.38, 79.23, 75.08, 62.97, 59.89, 31.61, 25.44, 19.50, 18.59, 16.63.

## General experimental procedure for the preparation of MNPs-supported organocatalyst **5**



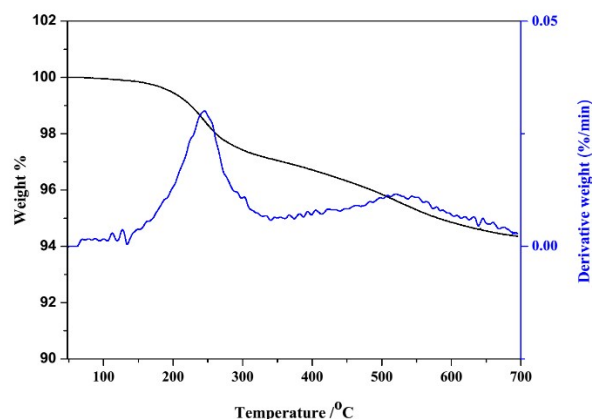
The magnetic nanoparticles ( $\text{Fe}_3\text{O}_4$ ) were prepared by the coprecipitation. To distilled water (125 ml) were added the  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (5.5 g, 20 mmol) and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (2.0 g, 10 mmol). The reaction was maintained at  $85^\circ\text{C}$  under a  $\text{N}_2$  atmosphere. Then, the aqueous ammonia (25wt%, 10 ml) was added rapidly. After 4 h stirring, the reaction was cooled to the room temperature. By the separation of an external magnet, washed with distilled water and dried in the vacuum, the magnetic nanoparticles ( $\text{Fe}_3\text{O}_4$ ) was obtained.

The naked  $\text{Fe}_3\text{O}_4$  (2.0 g) was dispersed in ethanol (120 mL) and water (40 ml) by ultrasonic irradiation. The concentrated  $\text{NH}_3 \cdot \text{H}_2\text{O}$  (6 mL) and TEOS (2 mL) were successively added into the solution, with continuous string for 24 h at room temperature. The MNPs were collected by an external magnet and washed three times with ethanol, followed by drying in vacuum.

The MNPs (0.8 g) was dispersed in dry toluene by ultrasonic irradiation. (3-azidopropyl)triethoxysilane (2.0 g) was added into the solution. The mixture was refluxed for 24 h under a nitrogen atmosphere. The azide@MNPs were collected by an external magnet and washed three times with ethanol, followed by drying in vacuum.

A solution of azide@MNPs (200 mg) and  $\text{CuI}$  (5.0 mg, 0.0255 mmol, 0.1 equiv.) in degassed DMF (5 mL) was stirred under nitrogen. The appropriate alkyne **4** (0.30 mmol, 1.2 equiv.) was then added, following by the addition of *N,N*-Diisopropylethylamine (DIPEA, 0.25 mmol, 32.0 mg, 1.0 equiv.). The solution was stirred at  $100^\circ\text{C}$  under nitrogen, and the reaction was reacted for 12 h. The final product **5** was obtained by by an external magnet and washed three times with ethanol, followed by drying in vacuum. The loading of the catalyst was determined to be 0.24 mmol/g by elemental analysis.

## TG–DTG analysis for azide@MNPs

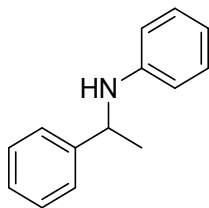


## General experimental procedure for the enantioselective reduction of imines with trichlorosilane catalyzed by catalyst 5

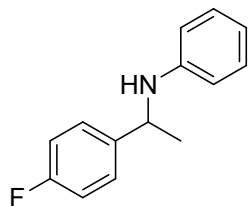
To a stirred solution of imine **6** (0.5 mmol) and catalyst **5** (10 mol %) in toluene (2 mL) was added the trichlorosilane (0.15 ml, 1.5 mmol) at room temperature and the reaction mixture was rocked at room temperature for 24 h. The catalyst was separated from the reaction mixture using an external magnet, washed with ethanol and ethyl acetate, and dried under vacuum, prior to being reused for next runs. Then, saturated NaHCO<sub>3</sub> (2 ml) was added and extracted with ethyl acetate (3\*10 ml). The combined organic phases were washed with brine, dried over MgSO<sub>4</sub>. Afterwards the organic phase was evaporated under reduced pressure to give the crude product, which was purified by column chromatography through silica gel, eluting with 1:99 ethyl acetate/petroleum ether solvent mixture, to give the pure product **7**. The ee value of the reduction product was determined by HPLC on chiral column (Daicel, Chiralpak, OD).

**Table 1.** Recovery of catalyst **5**

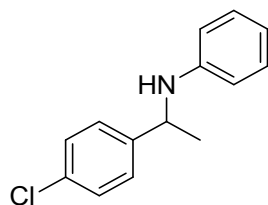
Run	Recovery/%
1	98
2	99
3	97
4	98
5	97



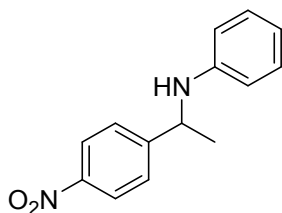
$[\alpha]_D^{20} = +15.4^\circ$  (c=1.05, MeOH), Yellow oil, 94% yield.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32 – 7.18 (m, 4H), 7.14 (t,  $J = 7.1$  Hz, 1H), 7.01 (t,  $J = 7.6$  Hz, 2H), 6.56 (t,  $J = 7.2$  Hz, 1H), 6.43 (d,  $J = 7.6$  Hz, 2H), 4.41 (q,  $J = 6.6$  Hz, 1H), 3.92 (br, 1H), 1.43 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  146.26, 144.20, 128.07, 127.60, 125.83, 124.82, 116.21, 112.29, 52.43, 23.98.



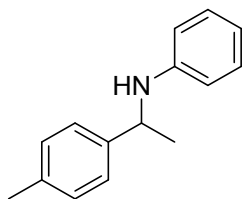
Yellow oil, 95% yield.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 – 7.24 (m, 2H), 7.08 (t,  $J = 7.9$  Hz, 2H), 7.01 – 6.90 (m, 2H), 6.64 (t,  $J = 7.3$  Hz, 1H), 6.47 (d,  $J = 7.9$  Hz, 2H), 4.43 (d,  $J = 6.7$  Hz, 1H), 4.01 (br, 1H), 1.46 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.82 (d,  $J = 244.3$  Hz), 147.13, 140.97, 140.94, 129.22, 127.41 (d,  $J = 8.0$  Hz), 117.54, 115.50 (d,  $J = 21.3$  Hz), 113.44, 52.98, 25.22.



Yellow oil, 97% yield.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33 – 7.20 (m, 4H), 7.12 – 7.00 (m, 2H), 6.64 (t,  $J = 7.3$  Hz, 1H), 6.52 – 6.36 (m, 2H), 4.42 (q,  $J = 6.7$  Hz, 1H), 4.01 (br, 1H), 1.45 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  147.03, 143.90, 132.45, 129.23, 128.87, 127.35, 117.61, 113.43, 53.06, 25.14.

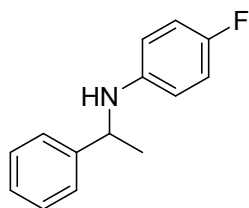


Yellow oil, 94% yield.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08 (d,  $J = 8.5$  Hz, 2H), 7.45 (d,  $J = 8.6$  Hz, 2H), 7.01 (t,  $J = 7.7$  Hz, 2H), 6.65 – 6.55 (m, 1H), 6.36 (d,  $J = 8.4$  Hz, 2H), 4.48 (q,  $J = 6.7$  Hz, 1H), 4.02 (s, 1H), 1.45 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  152.17, 146.02, 145.50, 128.20, 125.68, 123.04, 116.92, 112.27, 52.27, 23.89.

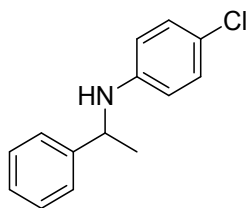


Yellow oil, 72% yield.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.22 (d,  $J = 8.1$  Hz, 2H), 7.12 – 7.00 (m, 4H), 6.61 (tt,  $J = 7.4, 1.0$  Hz, 1H), 6.51 – 6.44 (m, 2H), 4.42 (q,  $J = 6.7$  Hz, 1H), 3.95

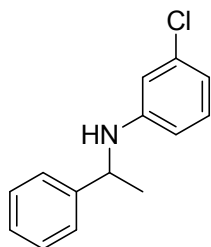
(br, 1H), 2.28 (s, 3H), 1.45 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  146.26, 141.14, 135.27, 128.13 (d,  $J = 23.1$  Hz), 124.69, 116.09, 112.24, 52.05, 23.92, 19.99.



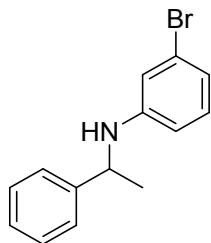
Yellow oil, 94% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.19 (m, 4H), 7.17 – 7.10 (m, 1H), 6.76 – 6.65 (m, 2H), 6.39 – 6.27 (m, 2H), 4.36 – 4.27 (m, 1H), 3.83 (br, 1H), 1.41 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  154.63 (d,  $J = 234.7$  Hz), 144.01, 142.59, 127.64, 125.92, 124.78, 114.46 (d,  $J = 22.2$  Hz), 113.06 (d,  $J = 7.3$  Hz), 113.02, 53.03, 24.03.



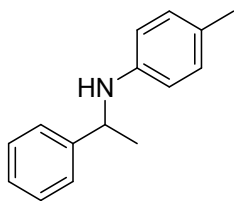
Yellow oil, 93% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.19 (m, 4H), 7.19 – 7.11 (m, 1H), 6.99 – 6.88 (m, 2H), 6.39 – 6.29 (m, 2H), 4.35 (q,  $J = 6.7$  Hz, 1H), 4.01 (br, 1H), 1.42 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  144.57, 143.53, 127.79 (d,  $J = 19.7$  Hz), 126.03, 124.77, 120.96, 113.50, 52.70, 23.86.



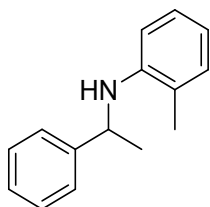
Yellow oil, 84% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.19 (m, 4H), 7.18 – 7.10 (m, 1H), 6.88 (t,  $J = 8.0$  Hz, 1H), 6.51 (ddd,  $J = 7.9, 1.9, 0.8$  Hz, 1H), 6.40 (t,  $J = 2.1$  Hz, 1H), 6.27 (ddd,  $J = 8.2, 2.3, 0.8$  Hz, 1H), 4.36 (q,  $J = 6.7$  Hz, 1H), 4.04 (br, 1H), 1.41 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  148.41, 144.57, 134.83, 130.13, 128.80, 127.14, 125.81, 117.17, 113.10, 111.52, 53.38, 24.89.



Yellow oil, 92% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.29 – 7.19 (m, 4H), 7.19 – 7.10 (m, 1H), 6.83 (t,  $J = 8.0$  Hz, 1H), 6.65 (dddd,  $J = 8.3, 6.4, 2.5, 1.3$  Hz, 1H), 6.58 (t,  $J = 2.1$  Hz, 1H), 6.34 – 6.27 (m, 1H), 4.43 – 4.28 (m, 1H), 4.04 (br, 1H), 1.42 (t,  $J = 5.9$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  147.43, 143.40, 129.34, 127.71, 126.06, 124.72, 122.01, 119.01, 114.97, 110.79, 52.28, 23.76.



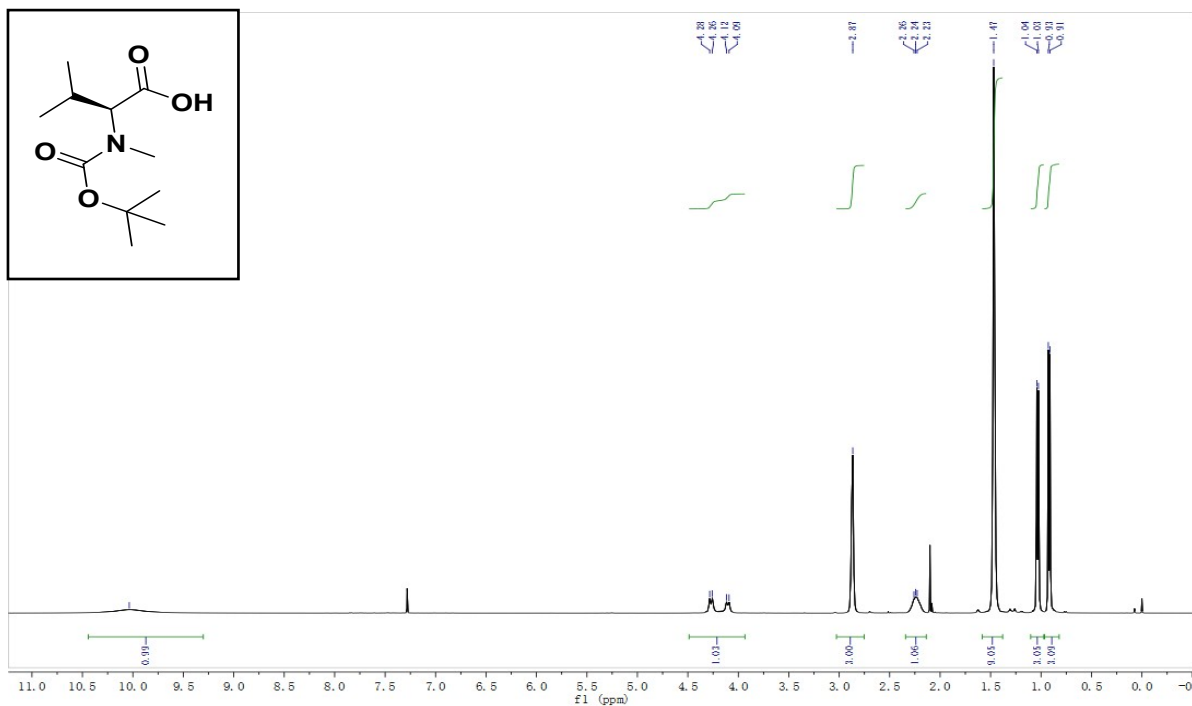
Yellow oil, 81% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30 – 7.16 (m, 4H), 7.15 – 7.08 (m, 1H), 6.80 (d,  $J = 8.1$  Hz, 2H), 6.37 – 6.28 (m, 2H), 4.35 (q,  $J = 6.7$  Hz, 1H), 3.77 (br, 1H), 2.09 (s, 3H), 1.39 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  144.37, 143.97, 128.54, 127.55, 125.73, 125.27, 124.80, 112.38, 52.61, 23.98, 19.30.

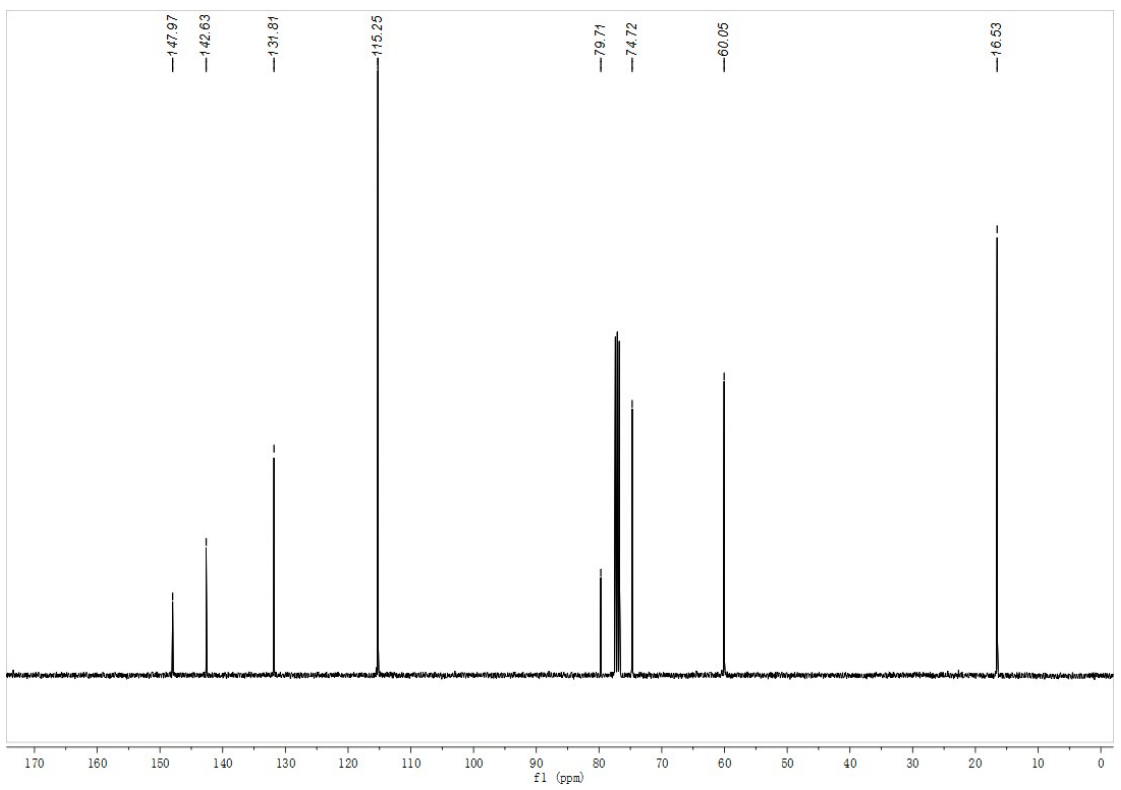
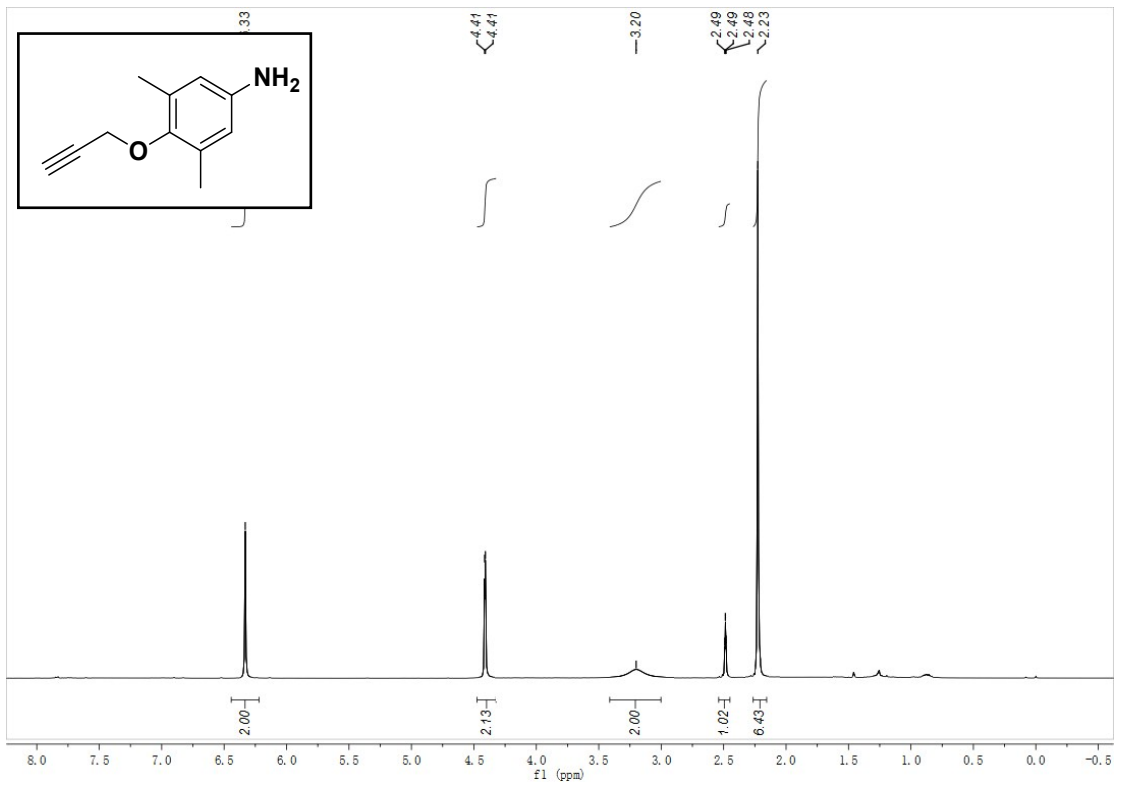


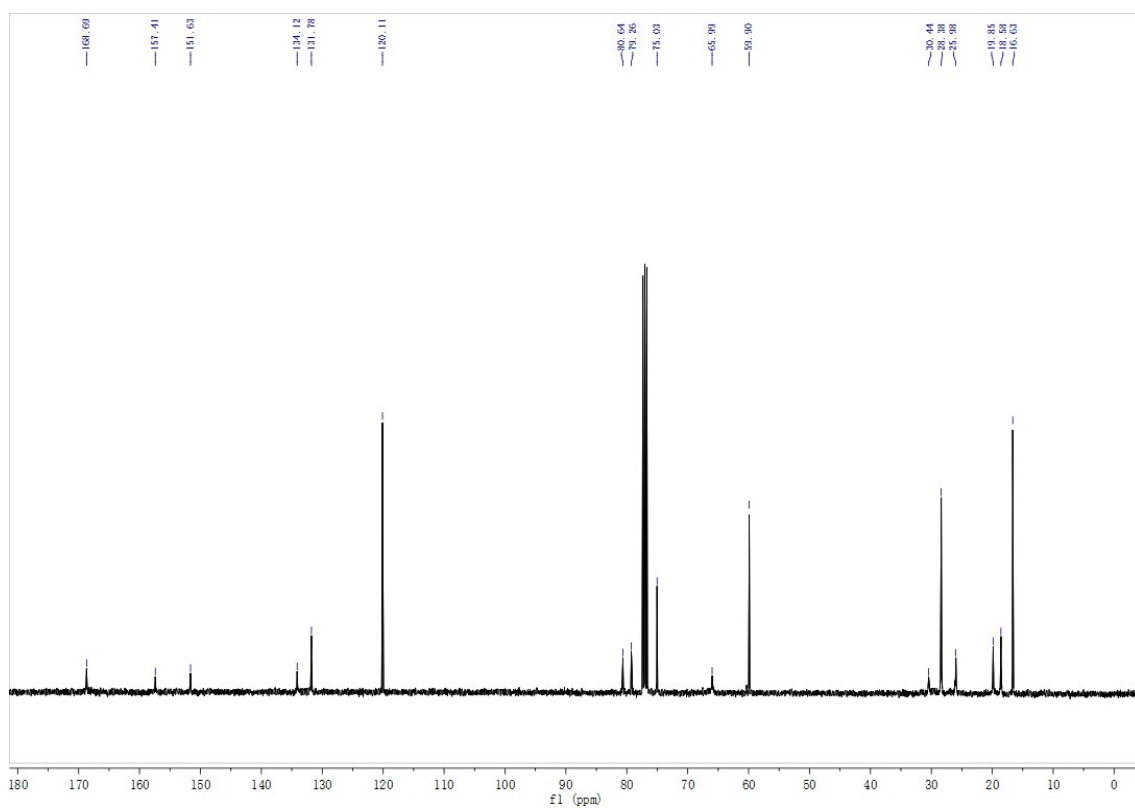
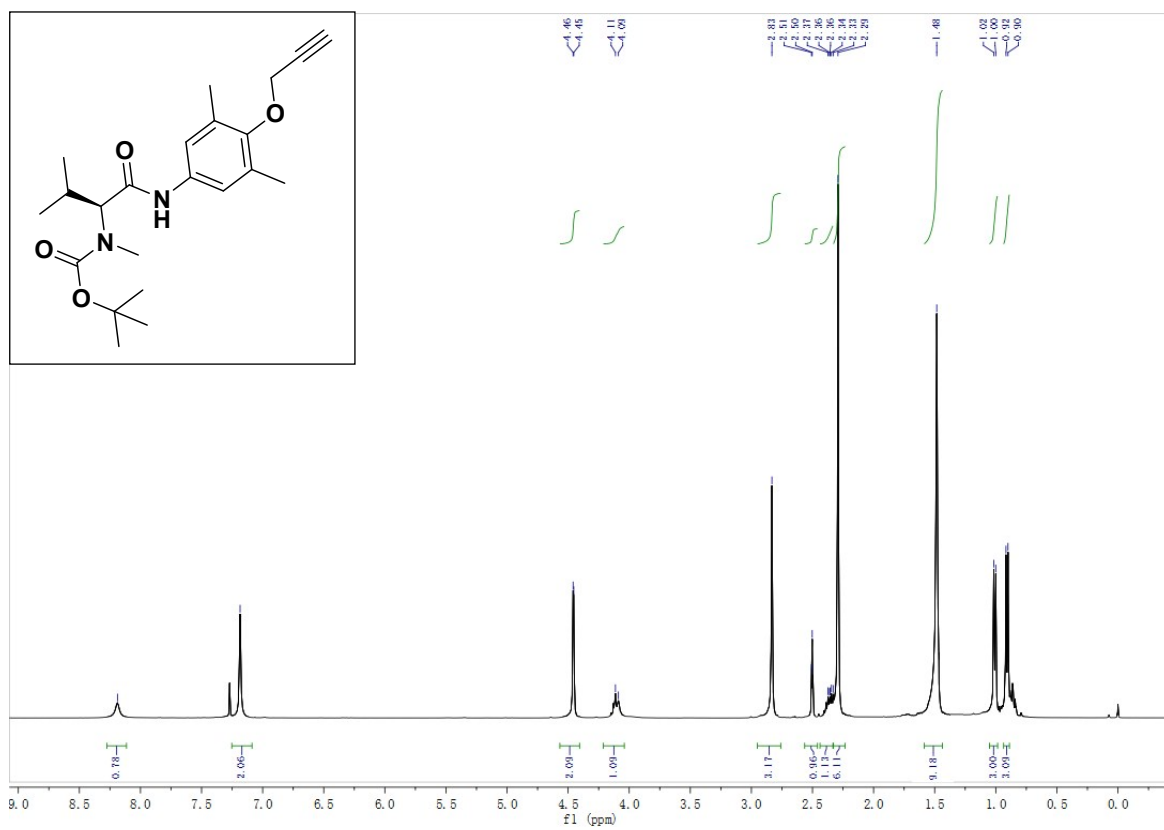
Yellow oil, 67% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.27 (dd,  $J = 8.2, 1.2$  Hz, 2H), 7.24 – 7.18 (m, 2H), 7.12 (ddd,  $J = 12.1, 6.4, 3.2$  Hz, 1H), 6.95 (d,  $J = 7.3$  Hz, 1H), 6.90 – 6.81 (m, 1H), 6.51 (td,  $J = 7.4, 0.9$  Hz, 1H), 6.28 (d,  $J = 7.9$  Hz, 1H), 4.44 (q,  $J = 6.7$  Hz, 1H), 3.75 (br, 1H), 2.13 (s, 3H), 1.46 (d,  $J = 6.7$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  144.17 (d,  $J = 12.5$  Hz), 128.92, 127.59, 125.87 (d,  $J = 15.1$  Hz), 124.73, 120.49, 115.79, 109.99, 52.25, 24.20, 16.57.

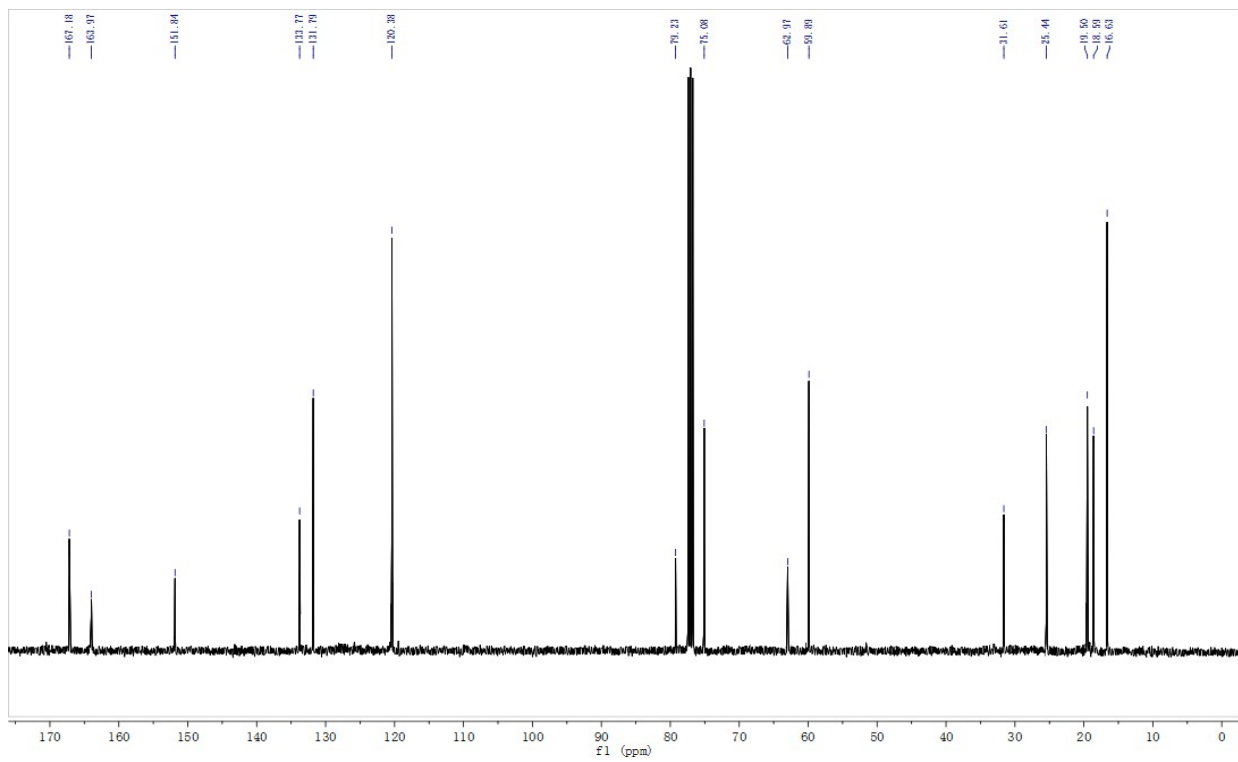
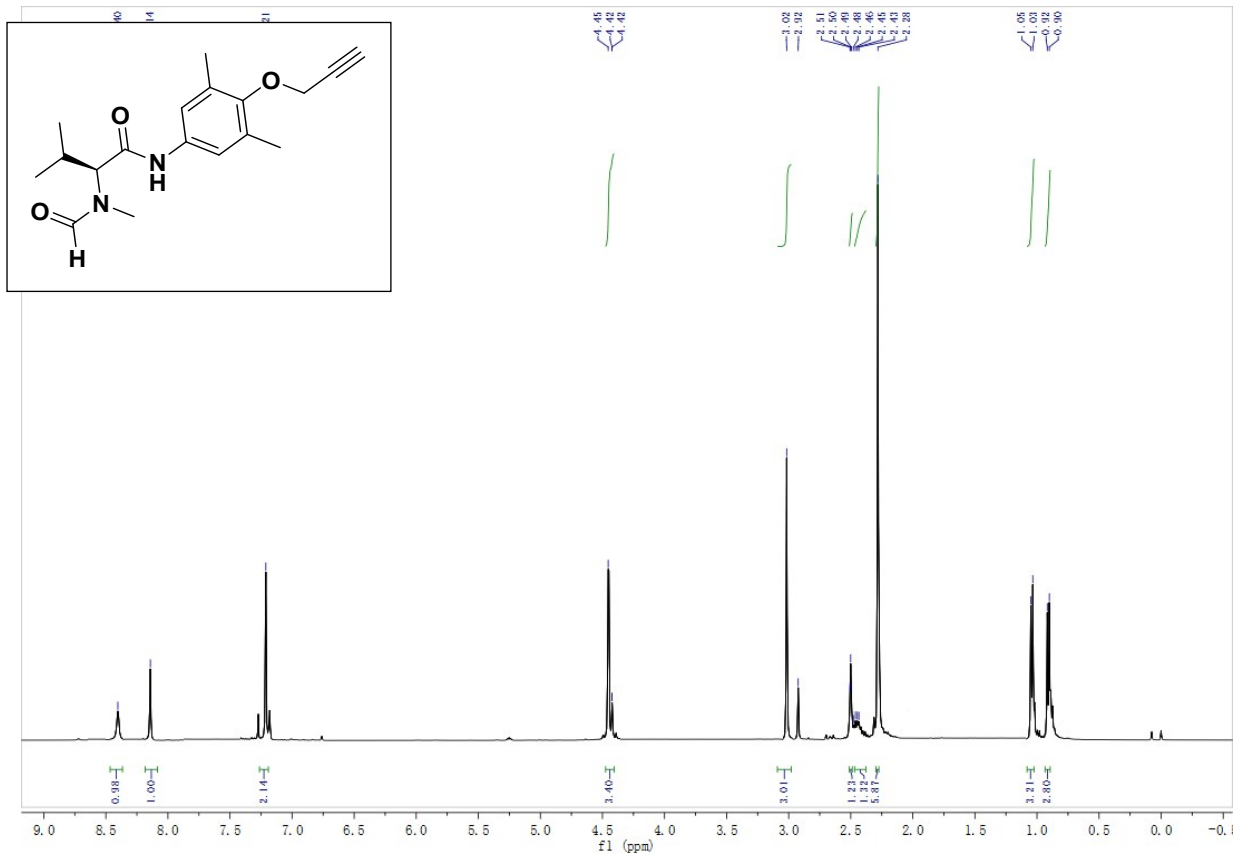


# NMR spectra of organocatalysts

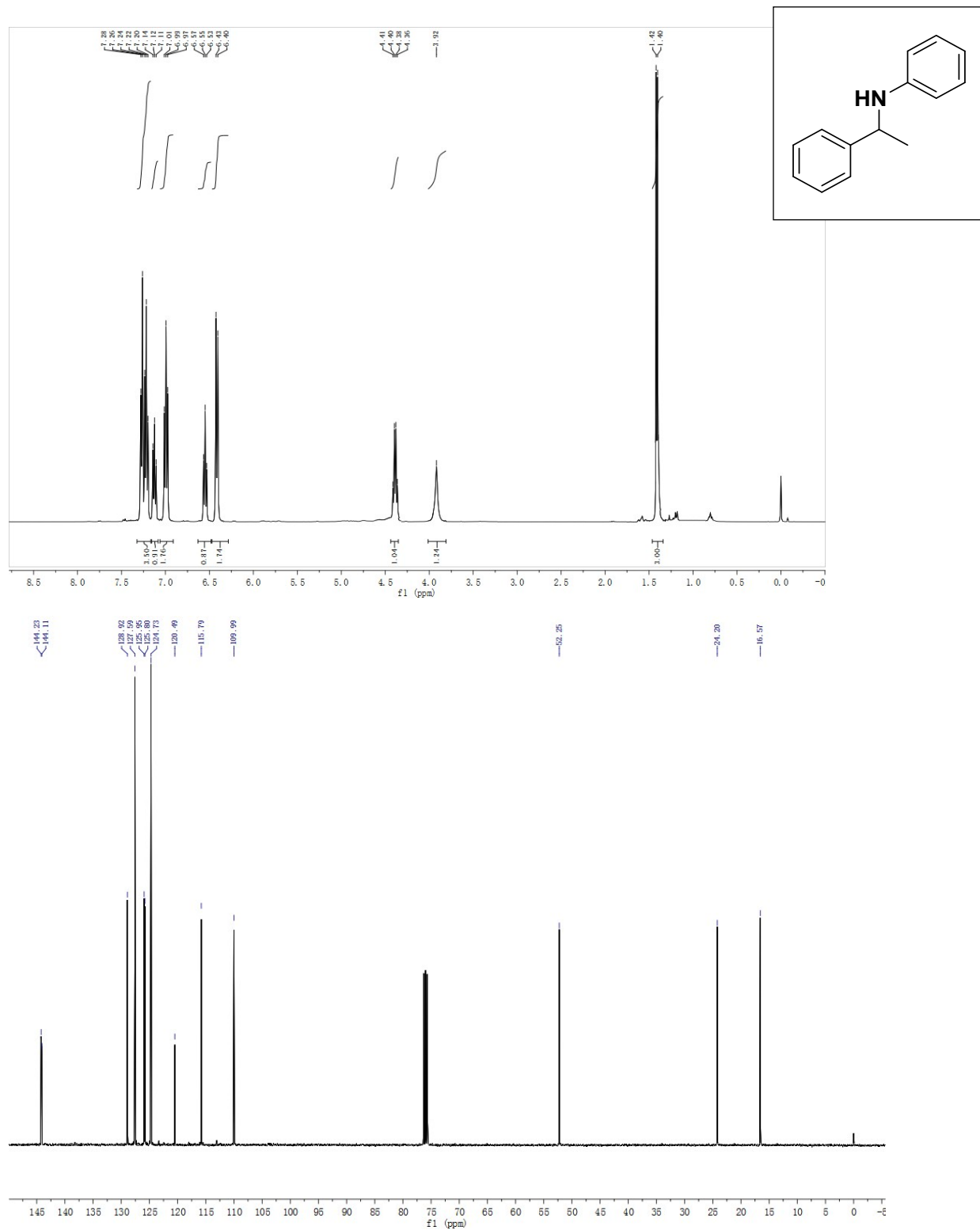




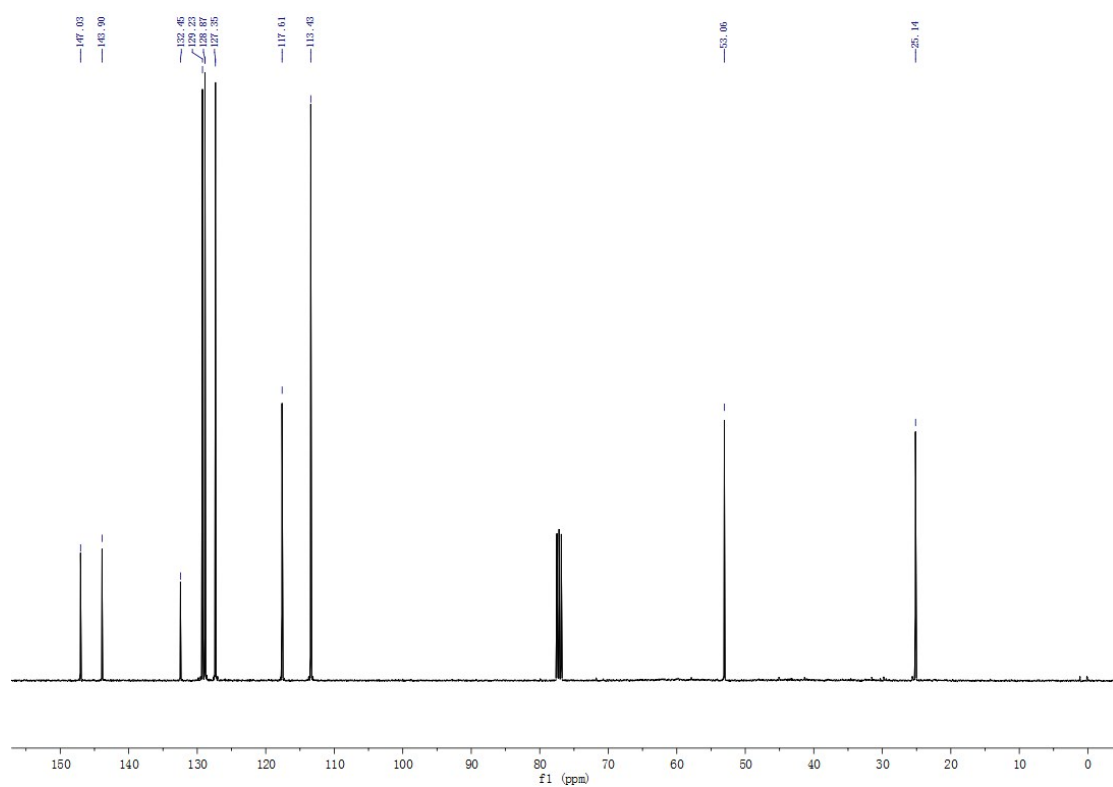
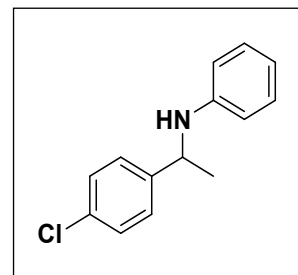
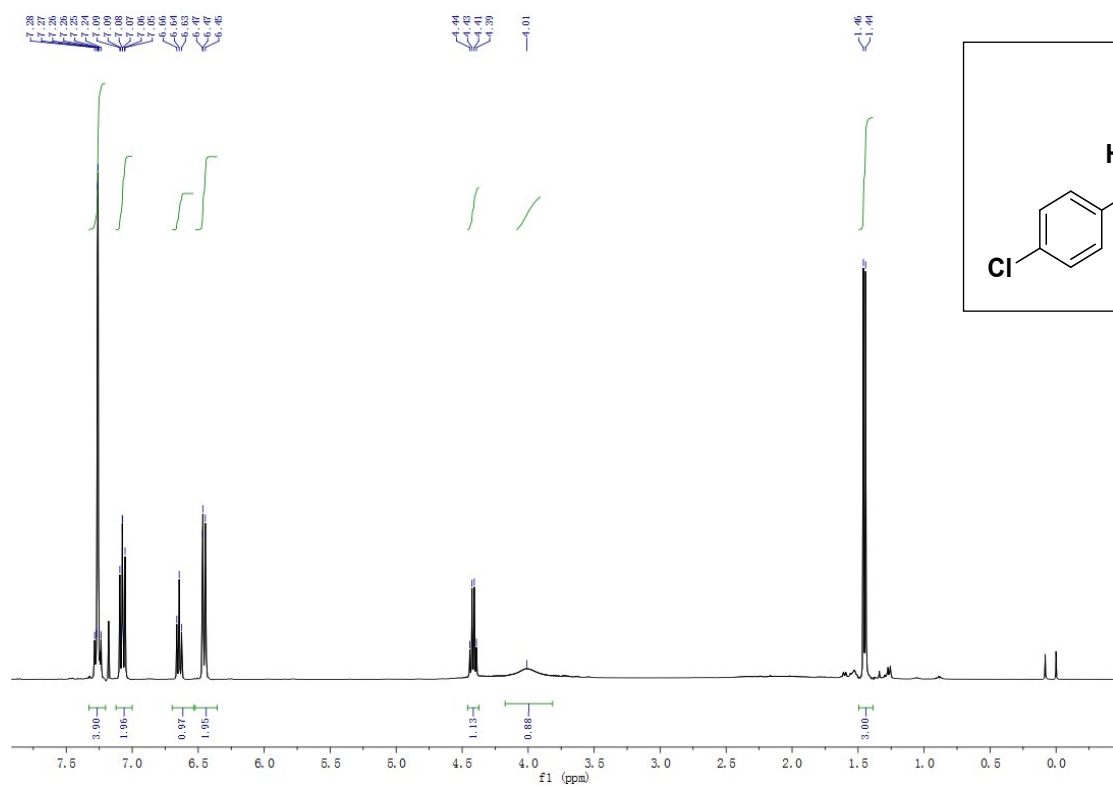


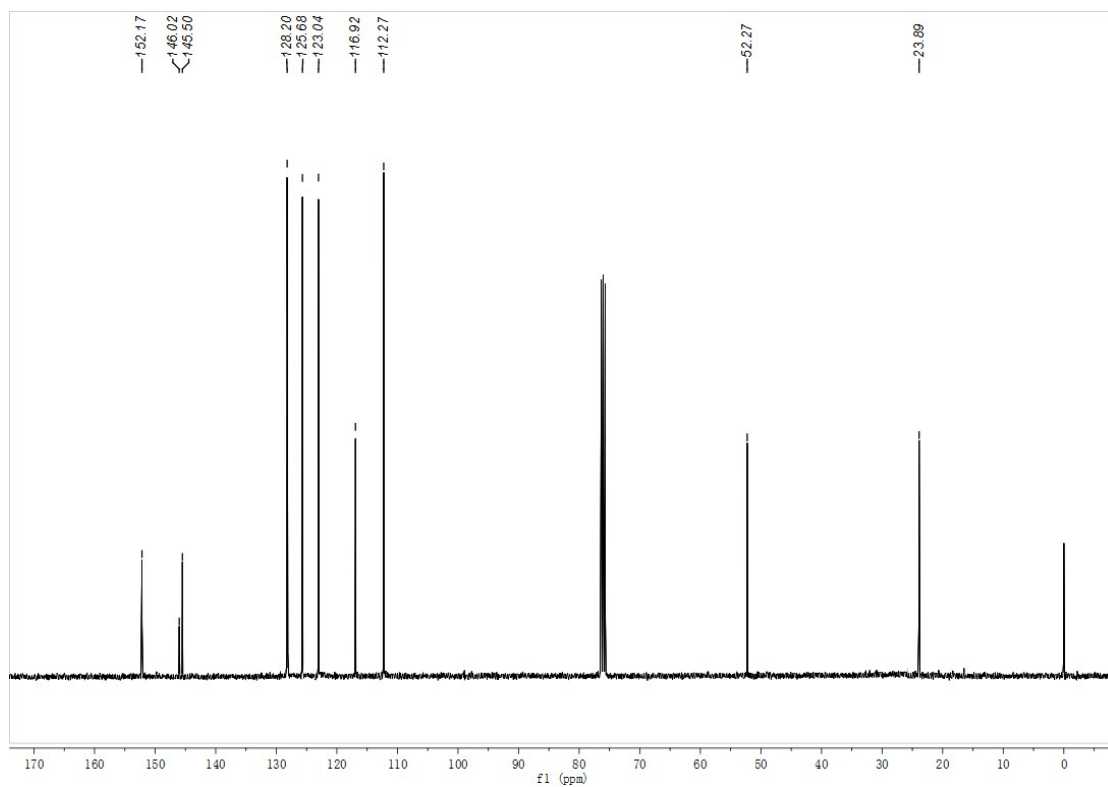
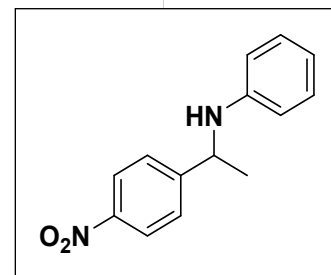
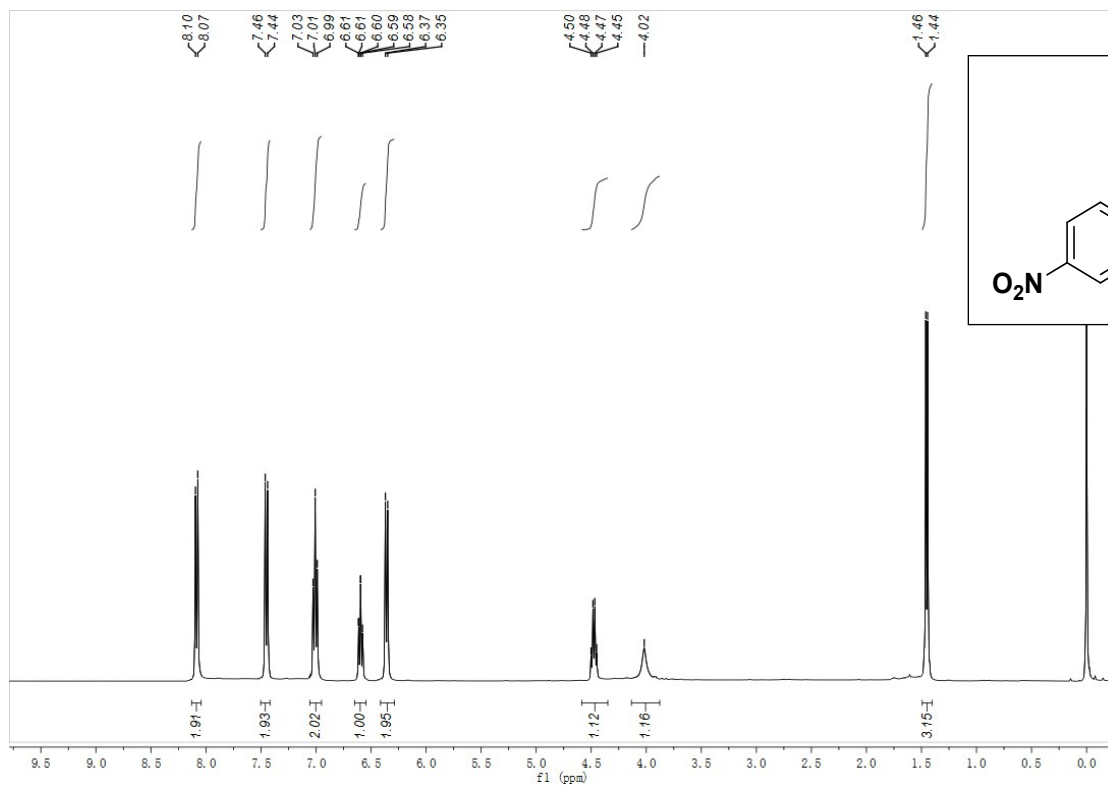


# NMR spectra of products

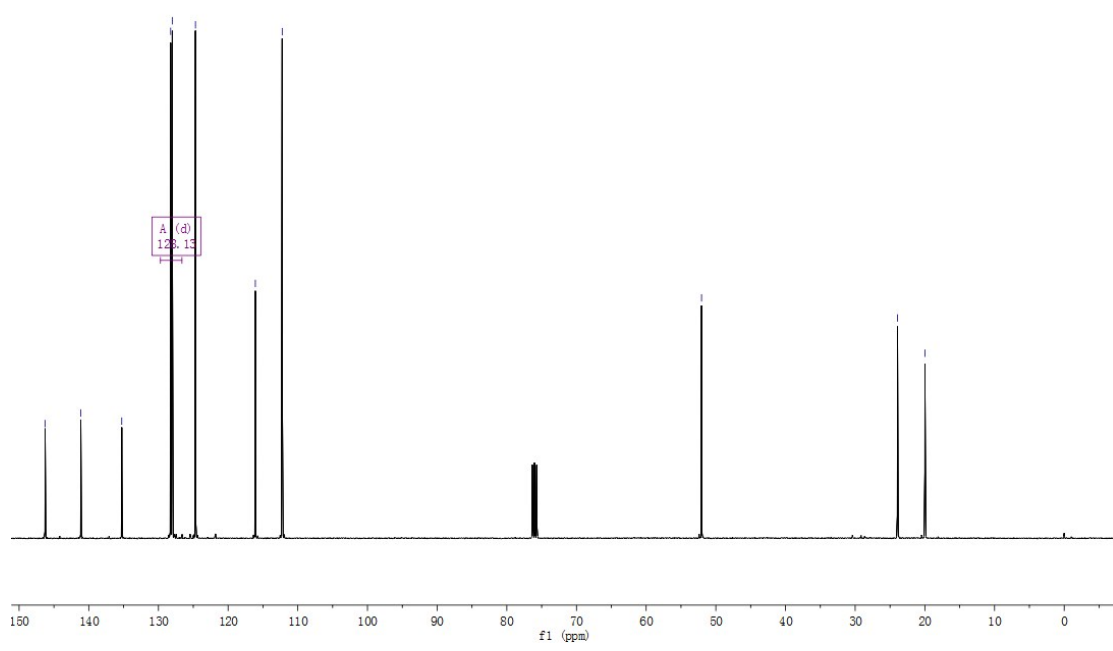
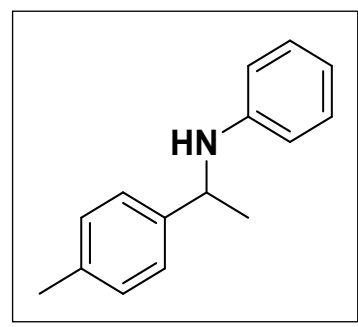
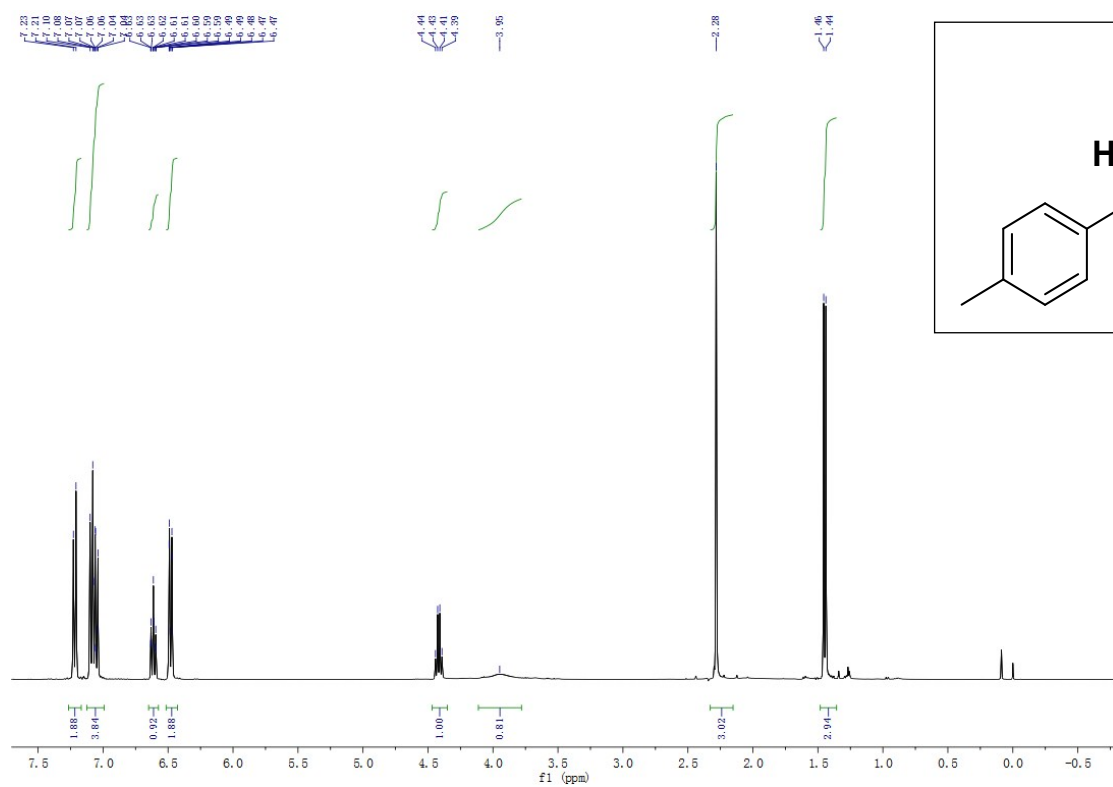


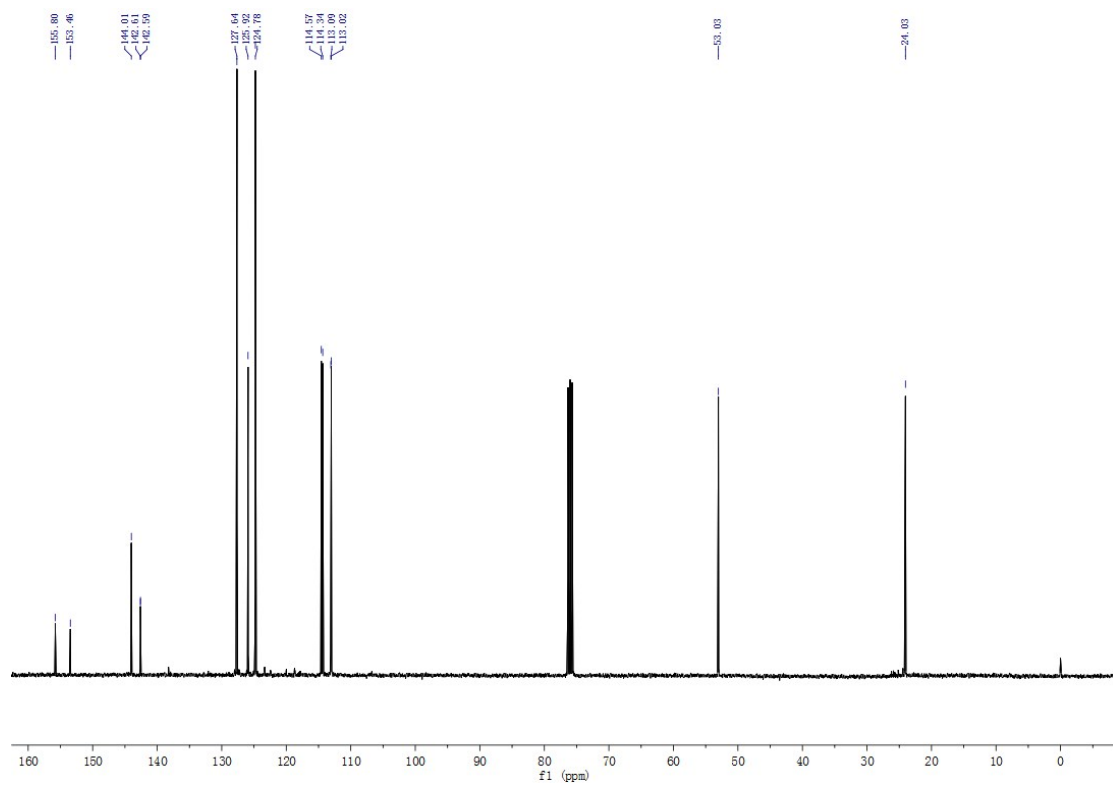
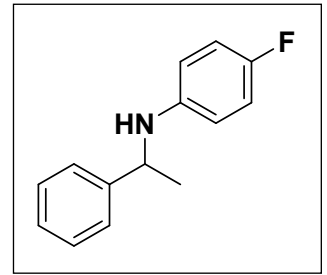
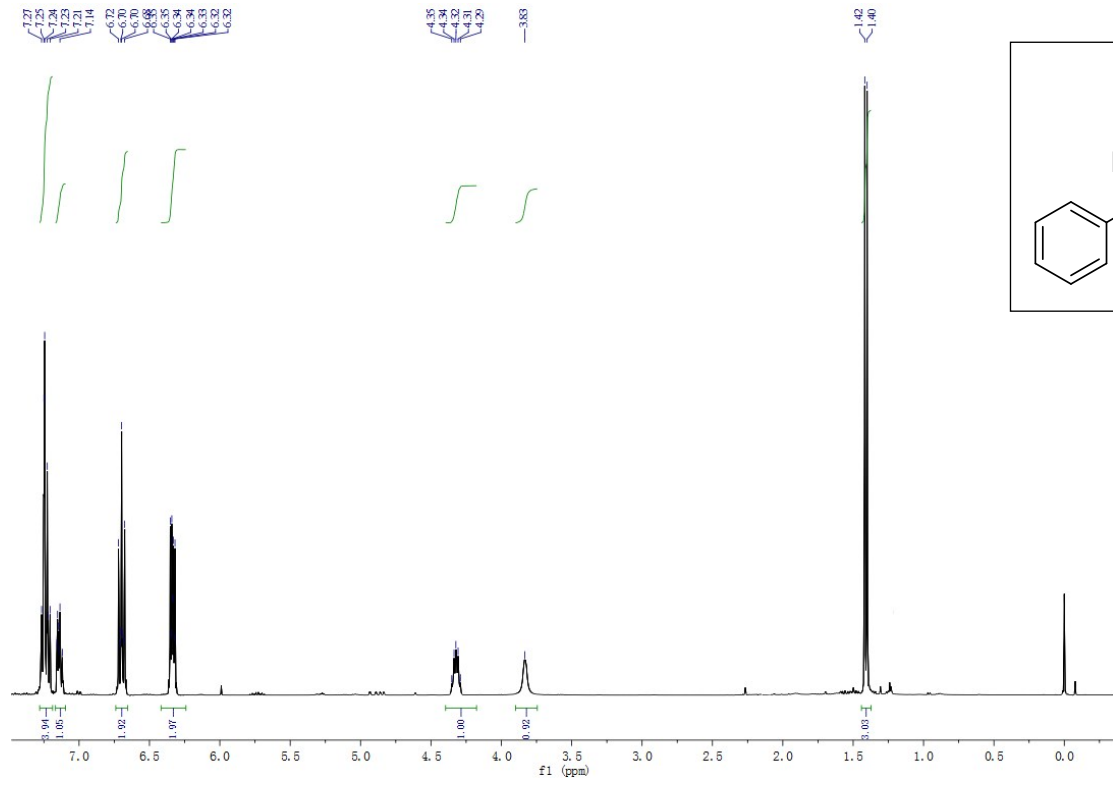


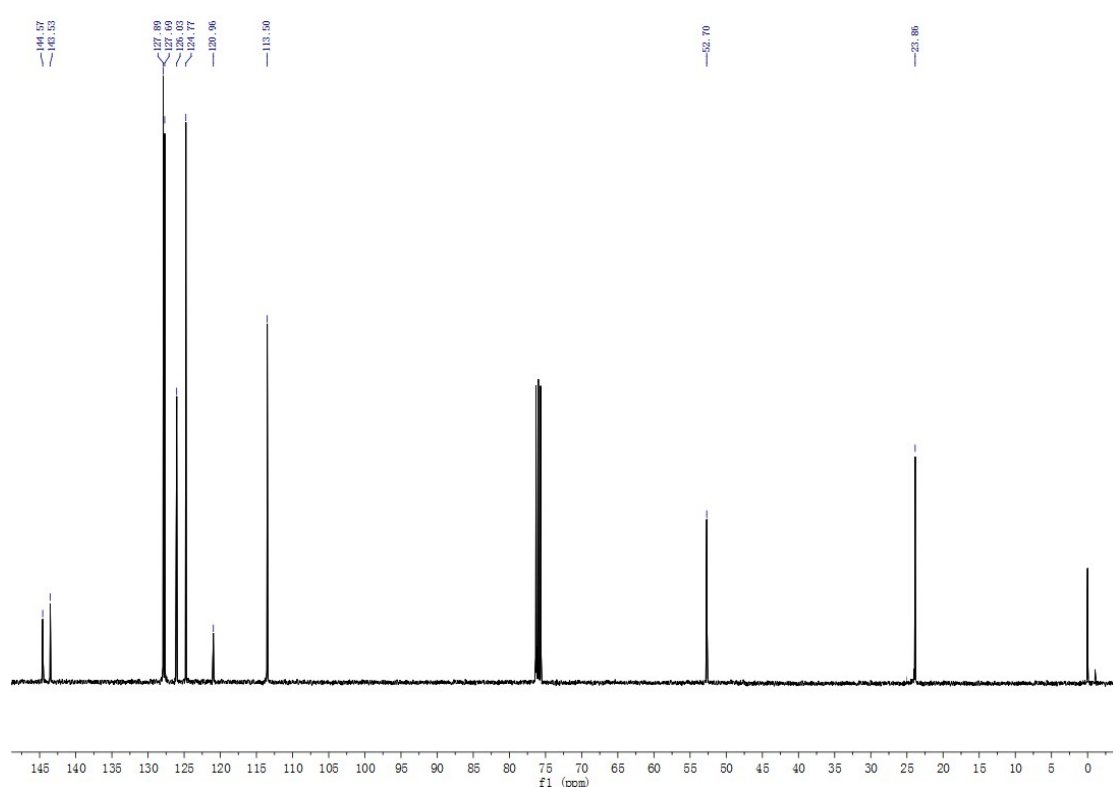
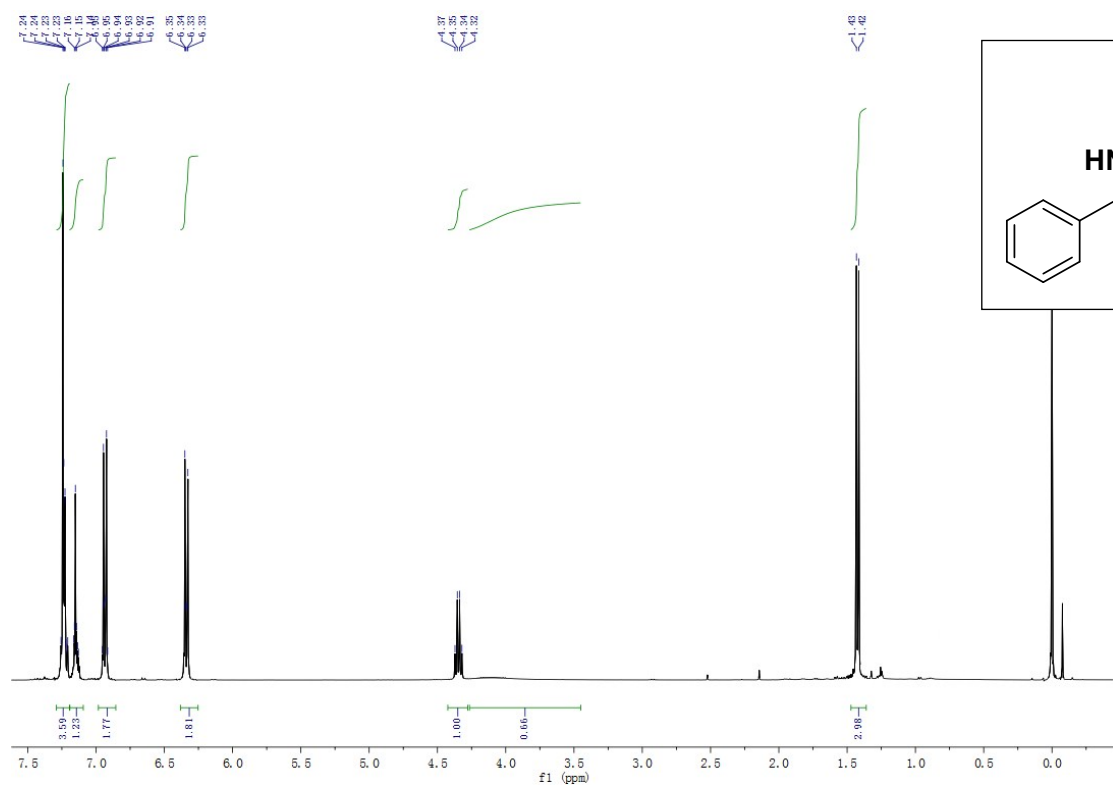


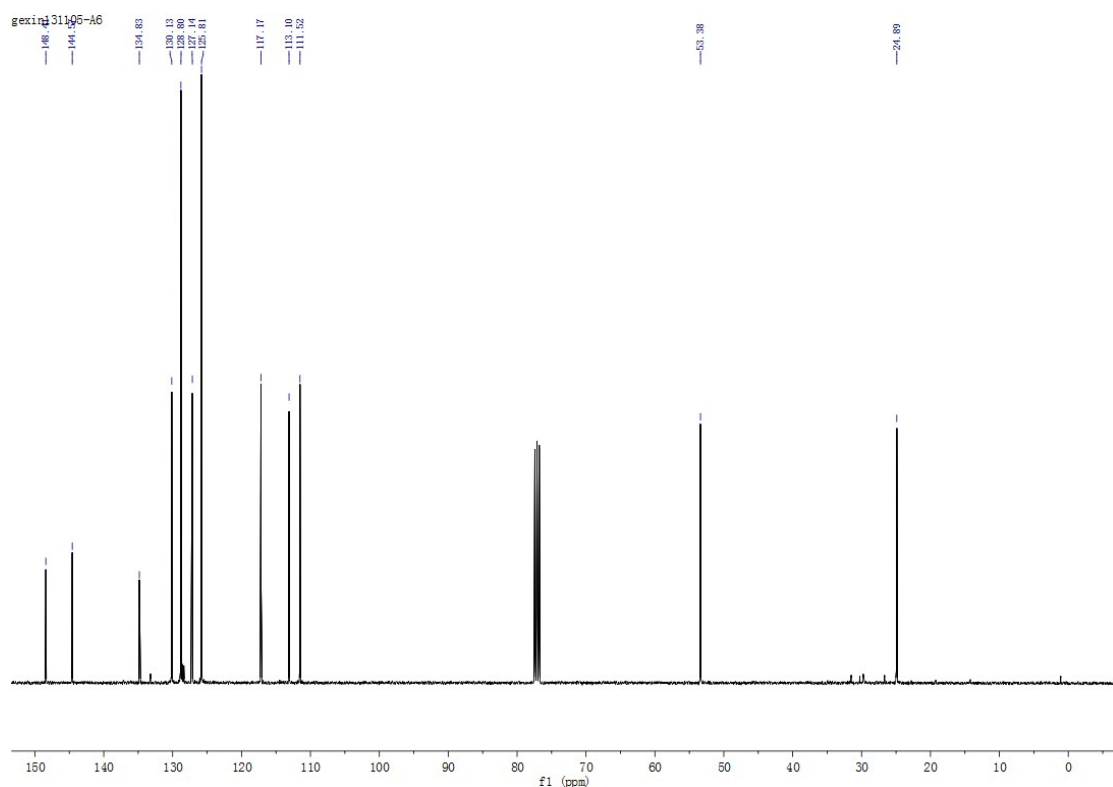
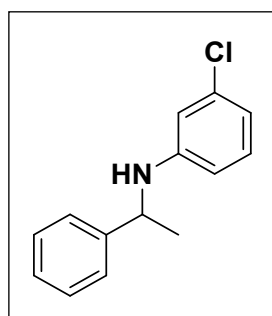
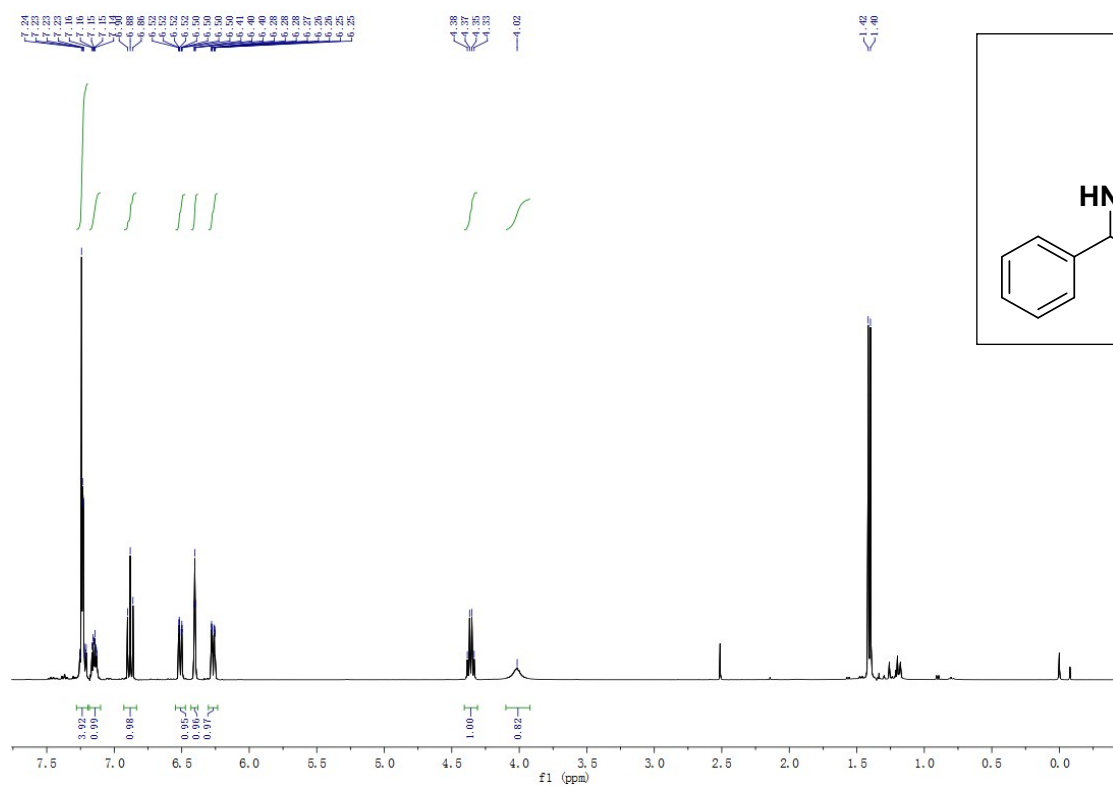


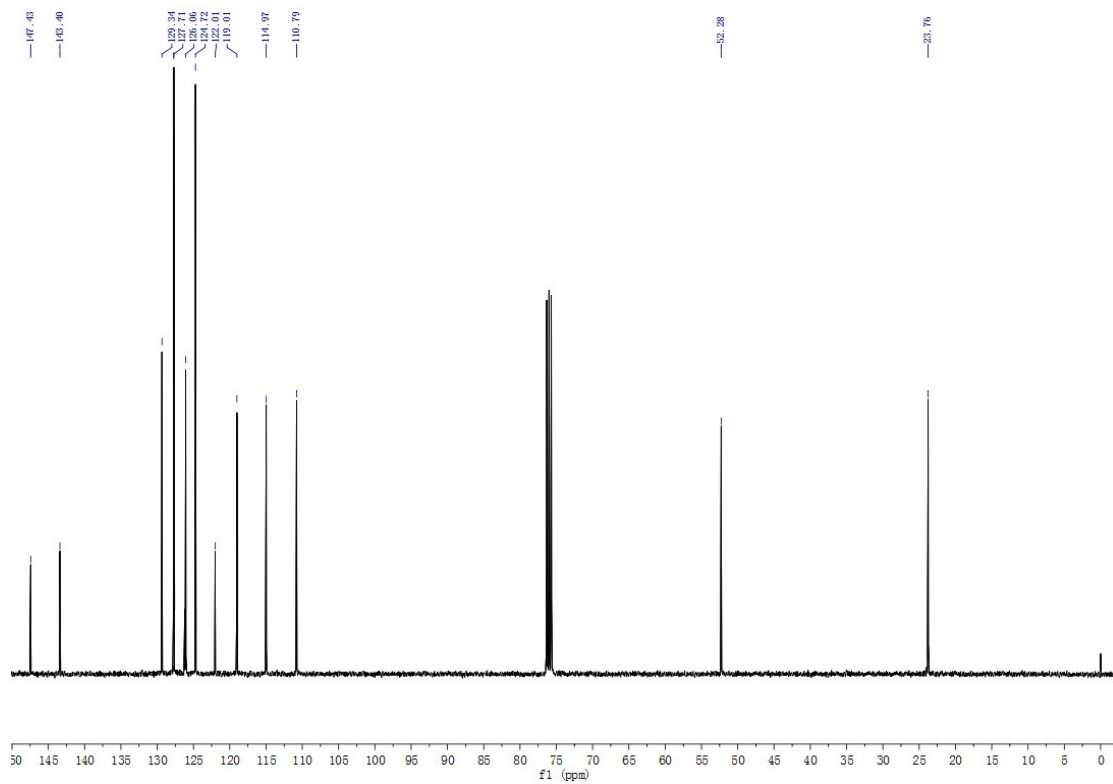
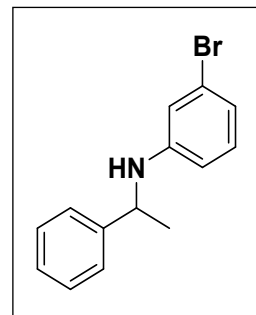
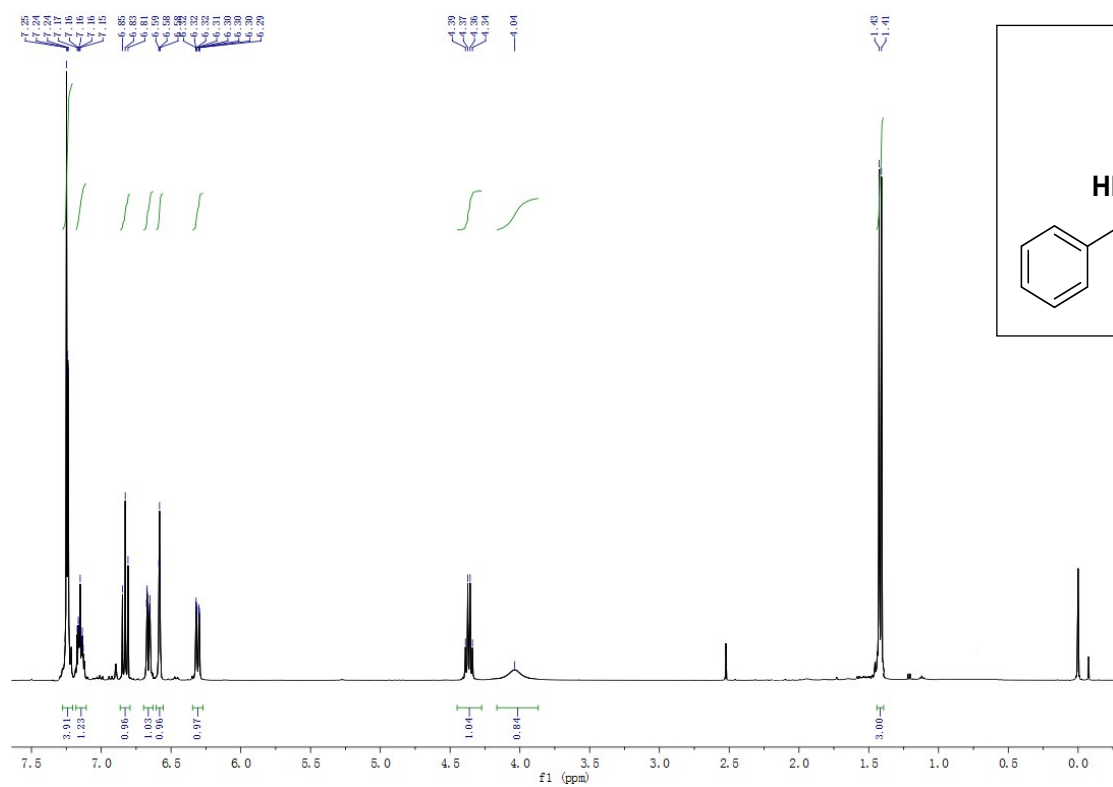


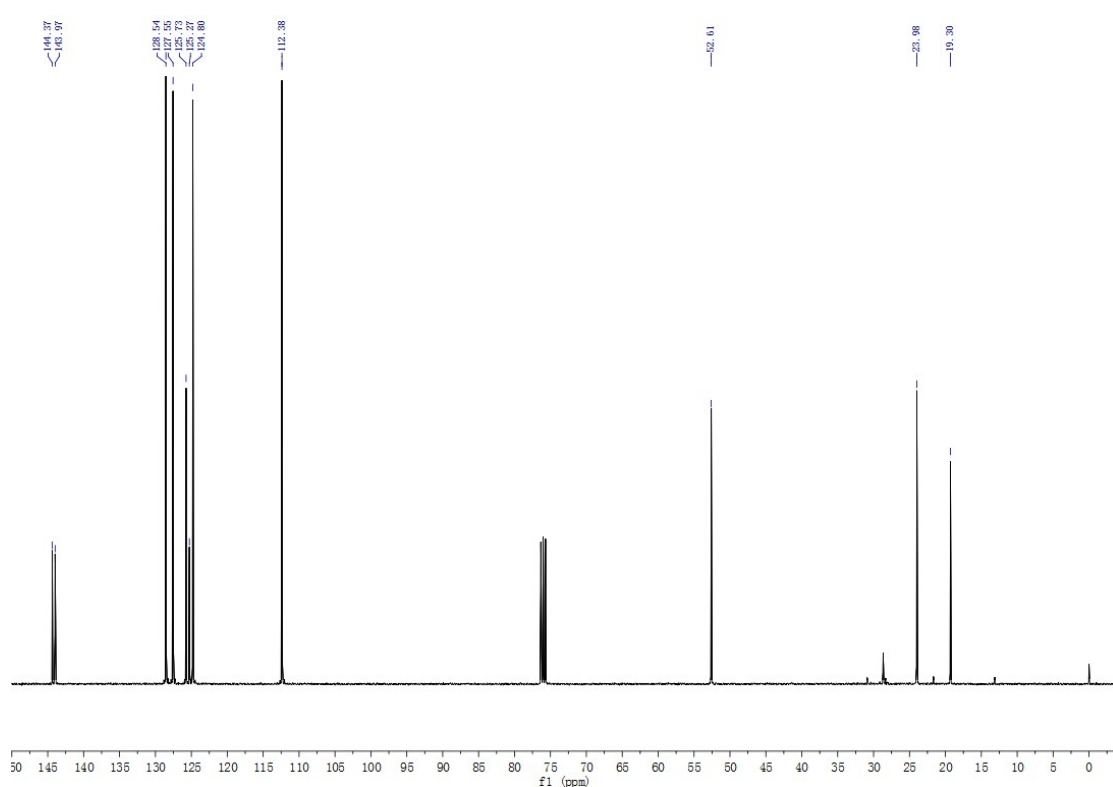
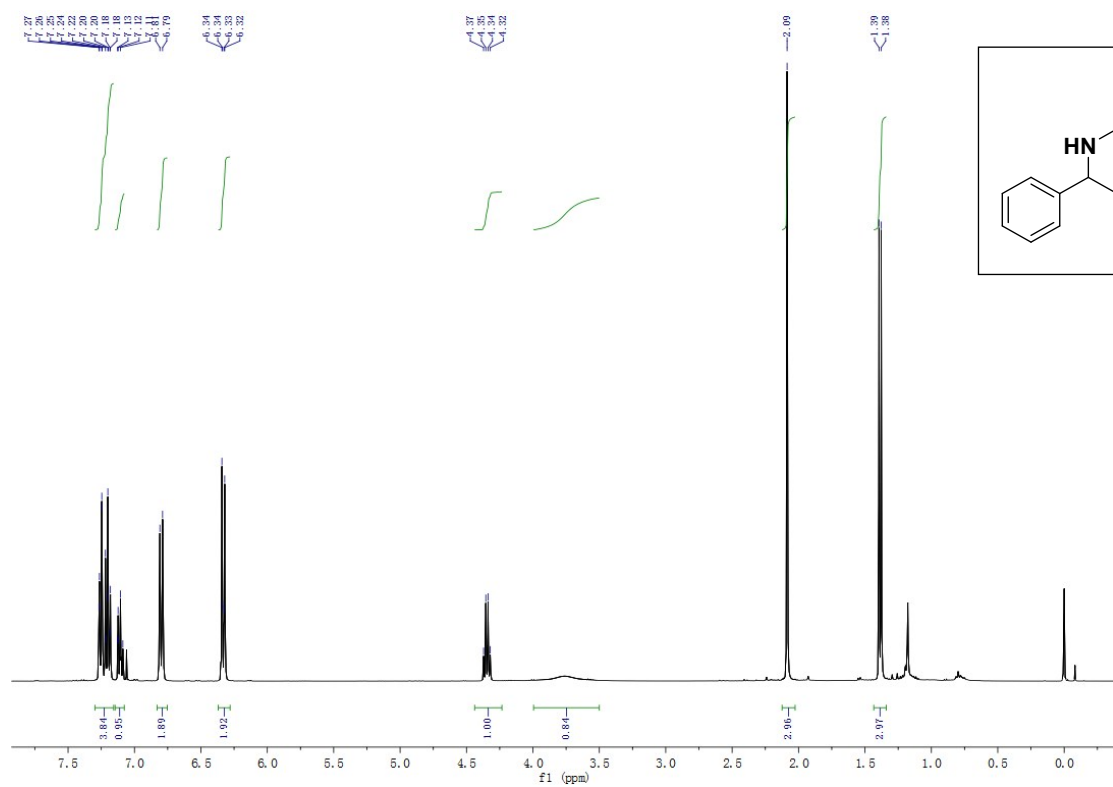


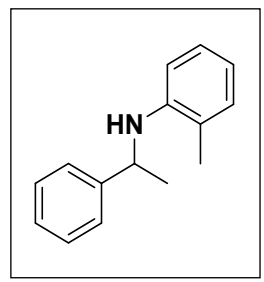
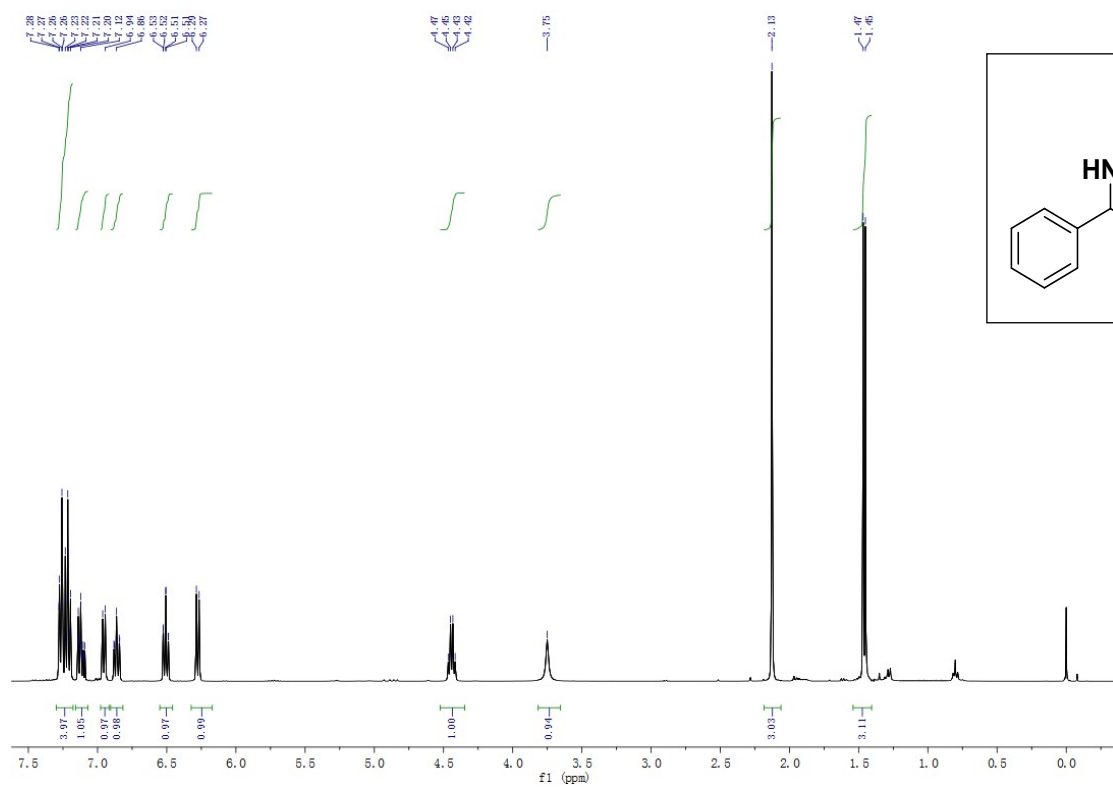




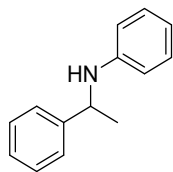






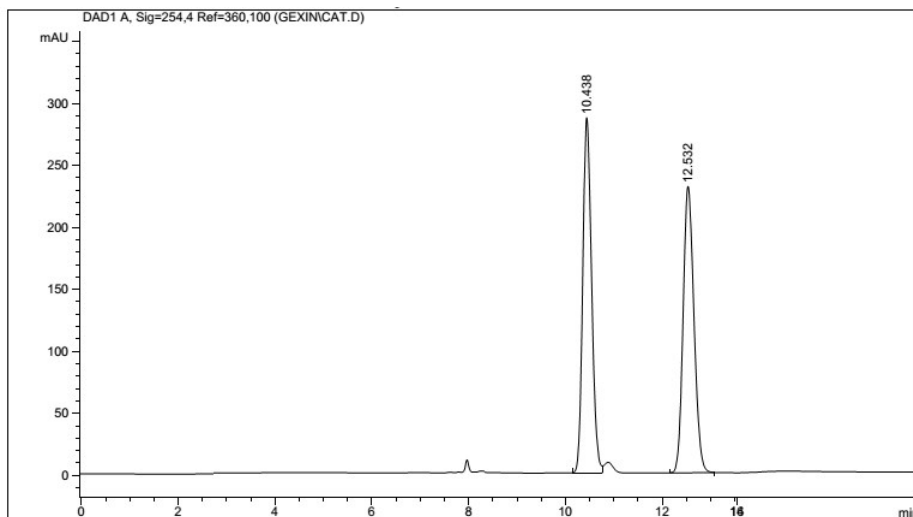


## HPLC spectra for compounds 7a-7k



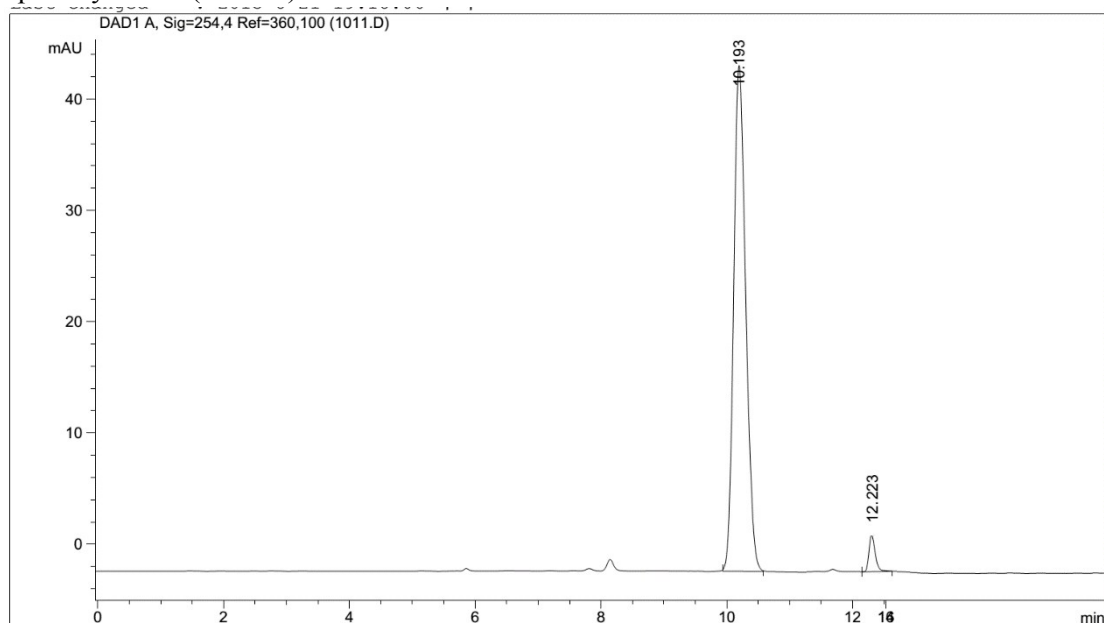
CHIRALCEL OD-H (Hexane/iPrOH = 99/1 1.0 mL/min, 254 nm)

Racemic



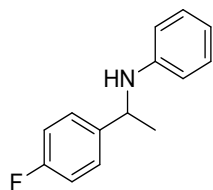
Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
10.438	286.64899	3639.61963	50.30
12.532	231.03723	3596.70337	49.70

Optically active (85% ee)



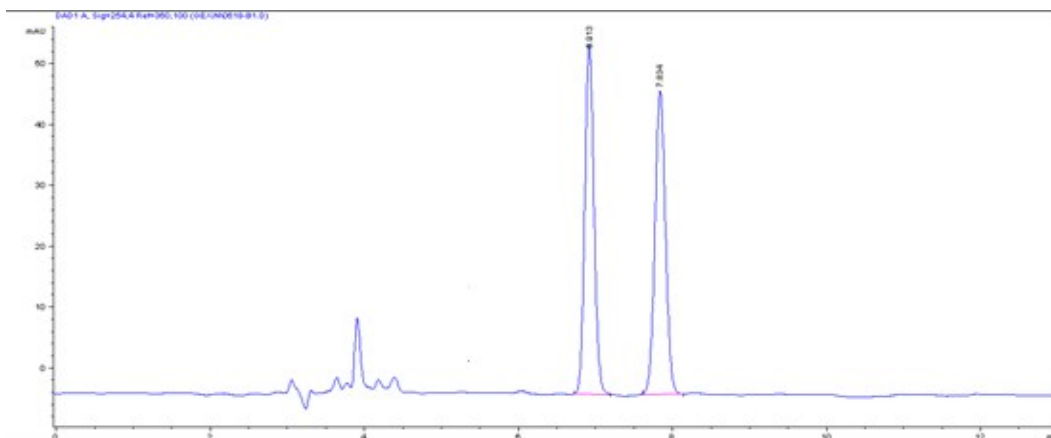


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
10.193	45.5	602.1	92.4
12.223	2.7	49.5	7.6



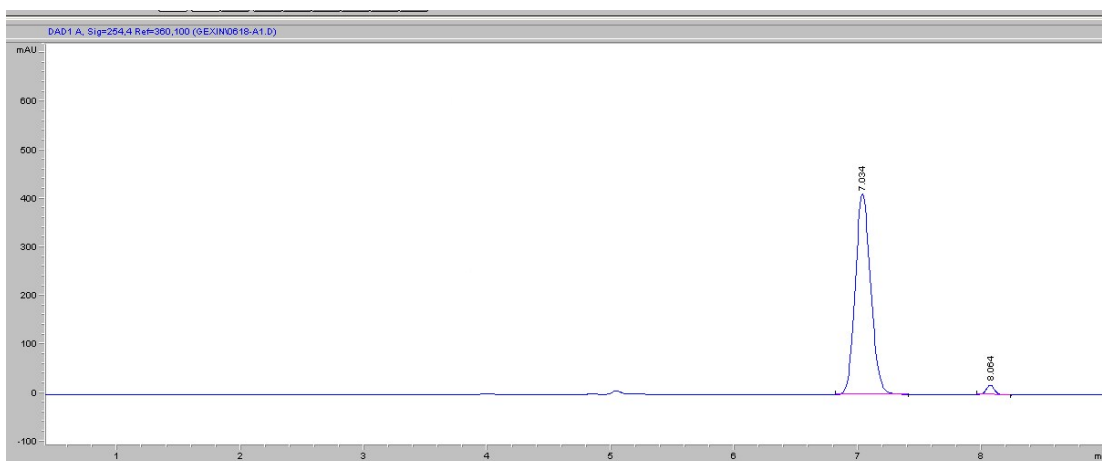
CHIRALCEL OD-H (Hexane/iPrOH = 95/5 1.0 mL/min, 254 nm)

Racemic

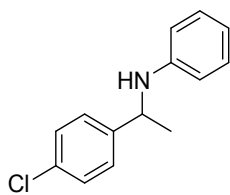


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
6.913	57.6	474.4	49.8
7.834	50	478.9	50.2

Optically active (88% ee)

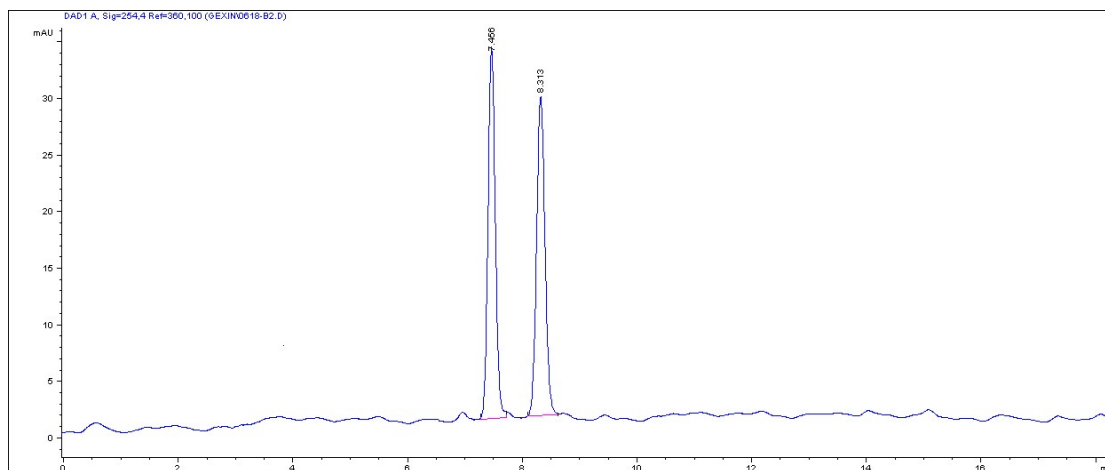


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
7.034	412.7	3589.4	94.1
8.064	25.4	225.1	5.9



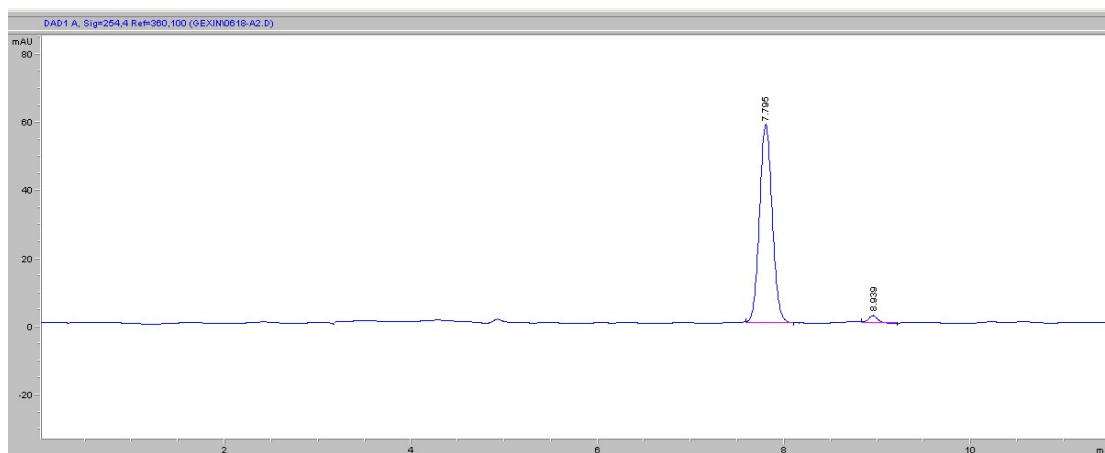
CHIRALCEL OD-H (Hexane/iPrOH = 95/5 1.0 mL/min, 254 nm)

Racemic

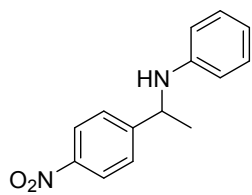


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
7.456	32.9	293.6	50.4
8.313	28.3	288.6	49.6

Optically active (91% ee)

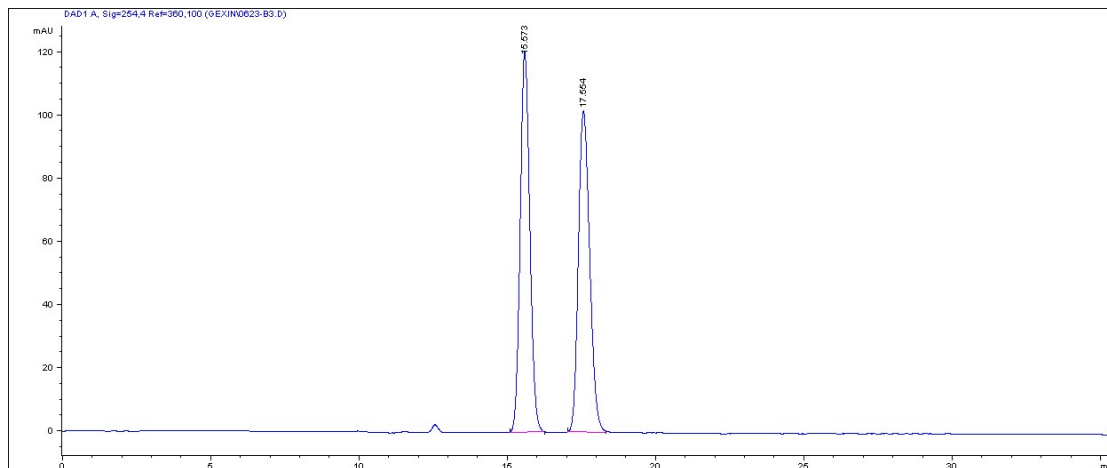


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
7.795	58.2	549.8	95.6
8.939	2.3	25.3	4.4



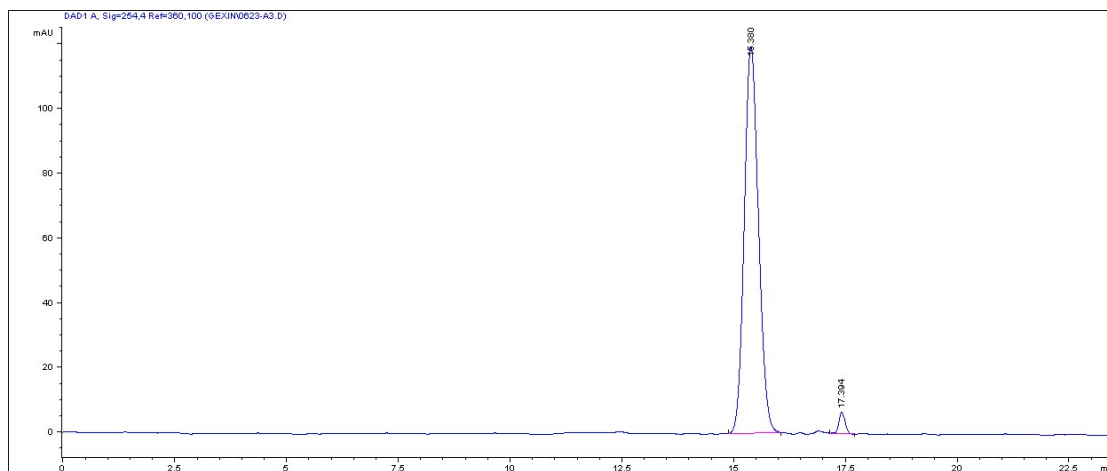
CHIRALCEL OD-H (Hexane/iPrOH = 85/15 1.0 mL/min, 254 nm)

Racemic

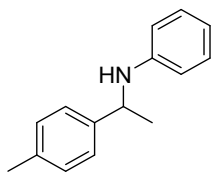


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
15.573	120.4	2717.9	50.1
17.554	101.7	2706.7	49.9

Optically active (92% ee)

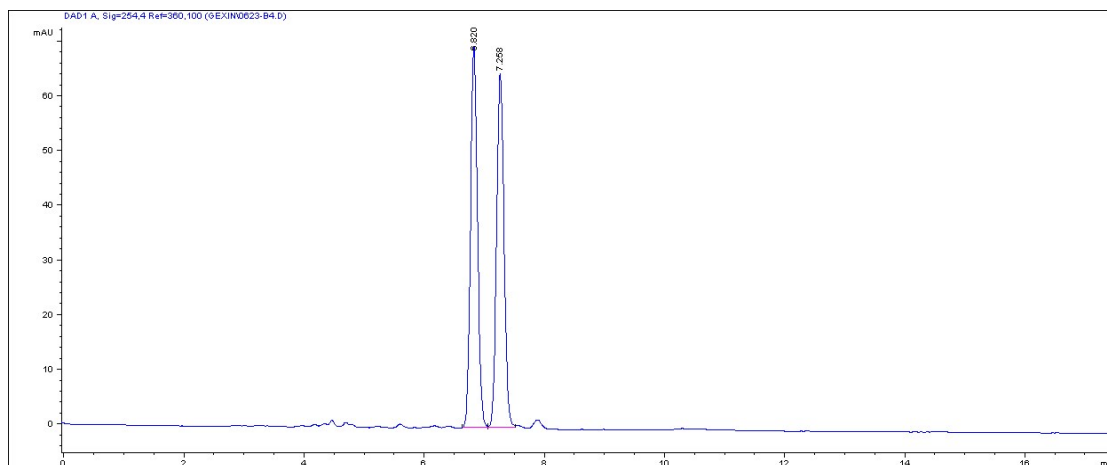


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
15.38	119.5	2637.1	96.2
17.394	8.1	104.1	3.8



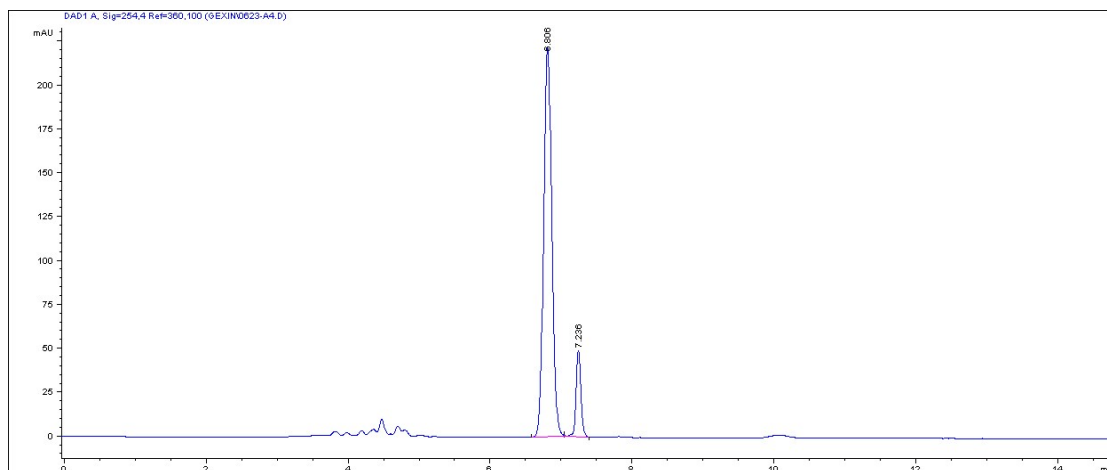
CHIRALCEL OD-H (Hexane/iPrOH = 99/1 1.0 mL/min, 254 nm)

Racemic

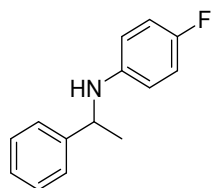


Ret. Time [min]	Height [mAU]	Area [mAU*s]	Rel. Area [%]
6.820	69.8	564.5	50.4
7.258	64.7	555.8	49.6

Optically active (67% ee)

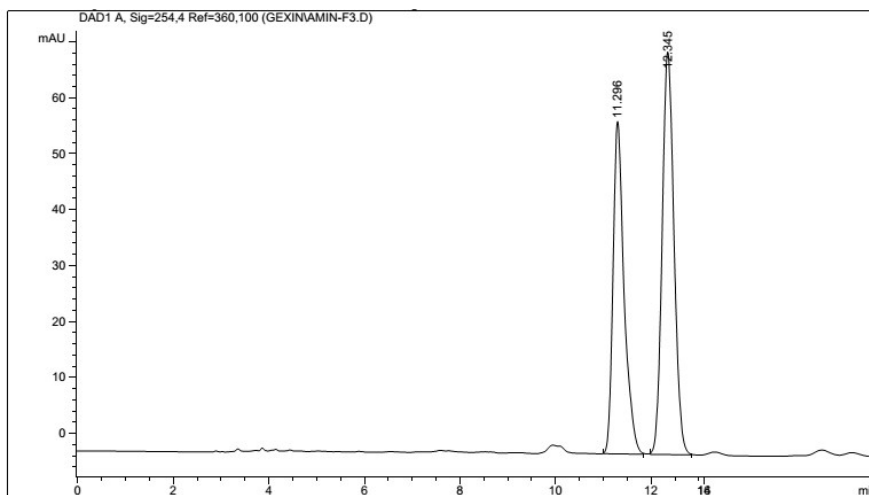


Ret. Time [min]	Height [mAU]	Area [mAU*s]	Rel. Area [%]
6.806	222.1	1785.5	83.3
7.236	50.4	358.0	16.7



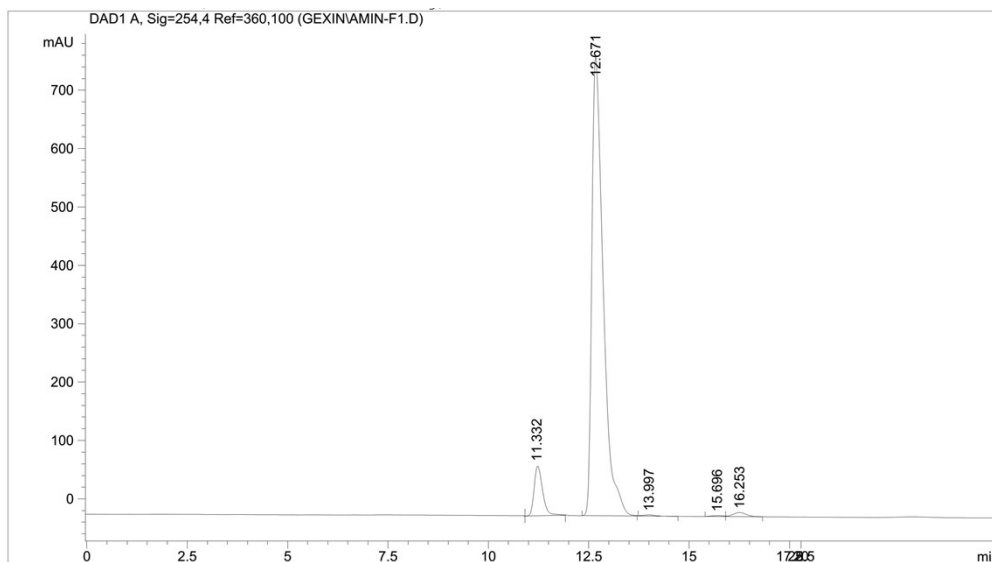
CHIRALCEL OD-H (Hexane/iPrOH = 99/1 1.0 mL/min, 254 nm)

Racemic

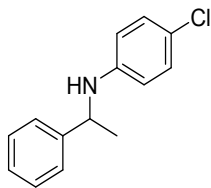


Ret. Time [min]	Height [mAU]	Area [mAU*s]	Rel. Area [%]
11.296	59.45957	1229.34862	49.92
12.345	72.01638	1233.52612	50.08

Optically active (81% ee)

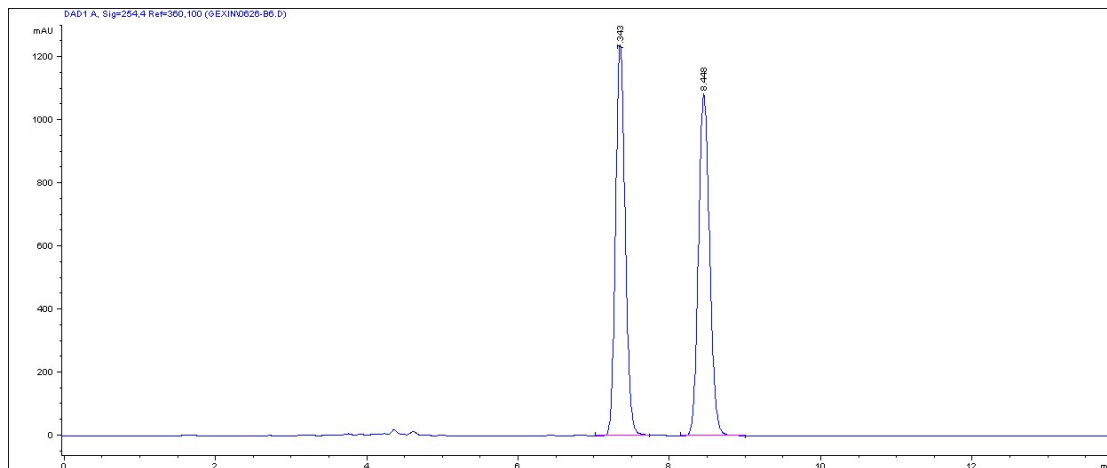


Ret. Time [min]	Height [mAU]	Area [mAU*s]	Rel. Area [%]
11.332	82.75500	1555.7	9.2
12.671	785.48450	1.54401e4	90.8



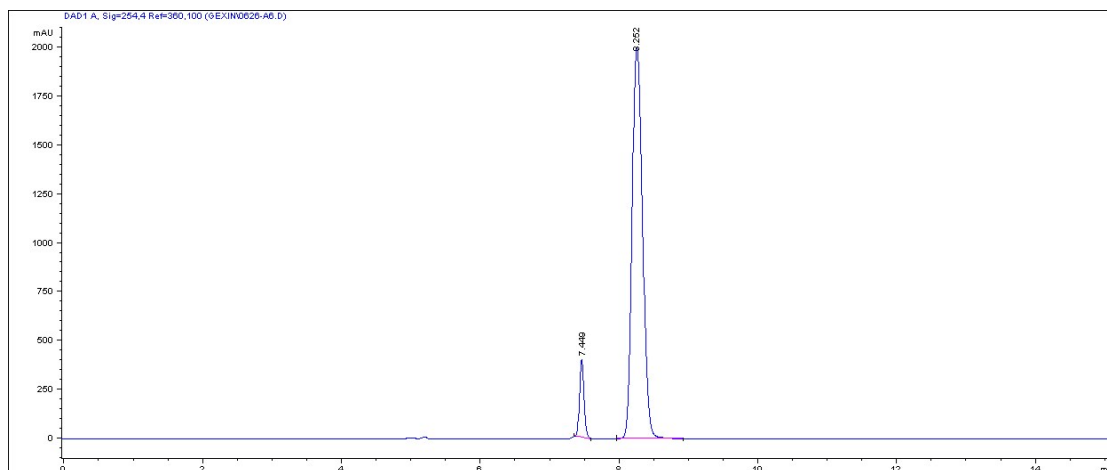
CHIRALCEL OD-H (Hexane/iPrOH = 95/5 1.0 mL/min, 254 nm)

Racemic

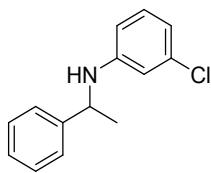


Ret.Time [min]	Height [mAu]	Area [mAU*s]	Rel.Area [%]
7.343	1239.8	10994.6	49.7
8.448	1084.1	11125.1	50.3

Optically active (83% ee)

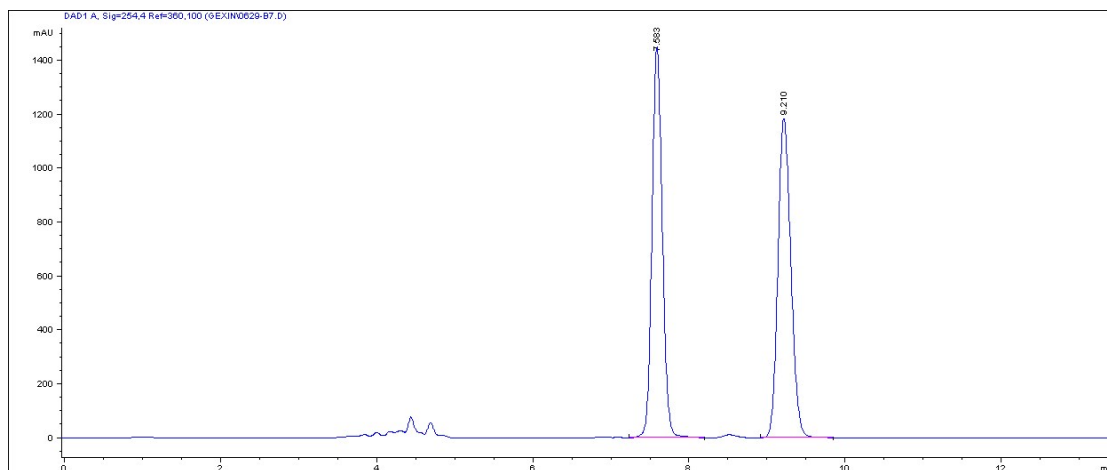


Ret.Time [min]	Height [mAu]	Area [mAU*s]	Rel.Area [%]
7.440	432.4	1947.6	8.5
8.252	2003.2	20946.3	91.5



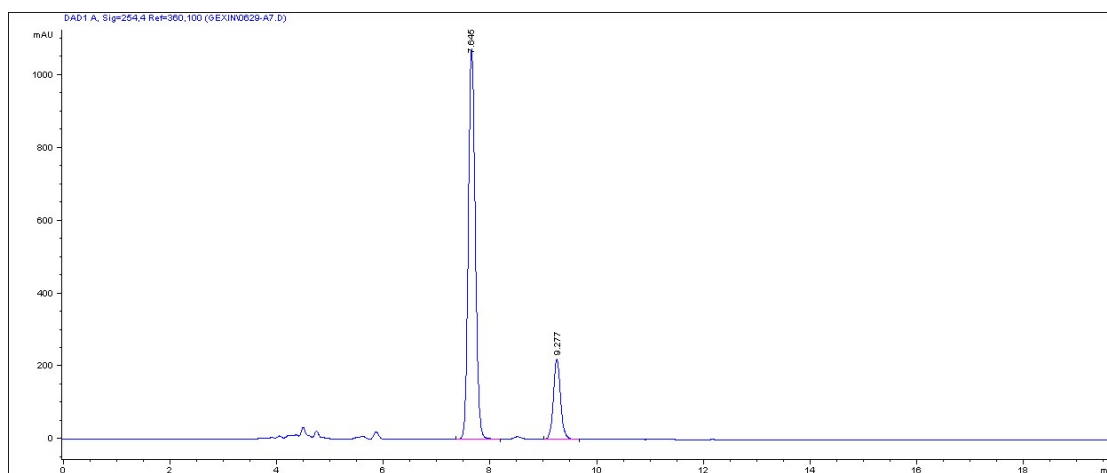
CHIRALCEL OD-H (Hexane/iPrOH = 95/5 1.0 mL/min, 254 nm)

Racemic

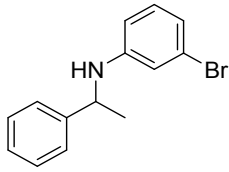


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
7.583	1450.1	13323.2	49.4
9.210	1184.4	13624	50.6

Optically active (74% ee)

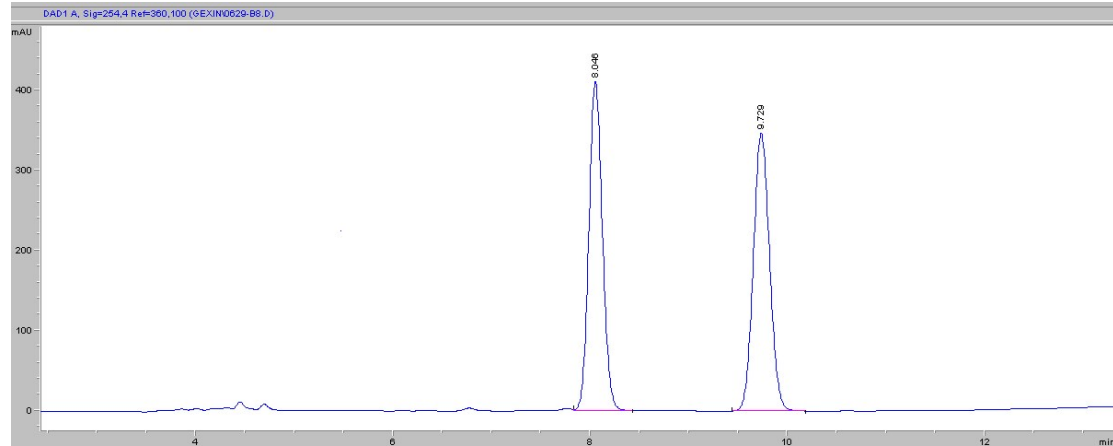


Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
7.645	1072.8	9687.8	87.1
9.277	205.1	1434.6	12.9



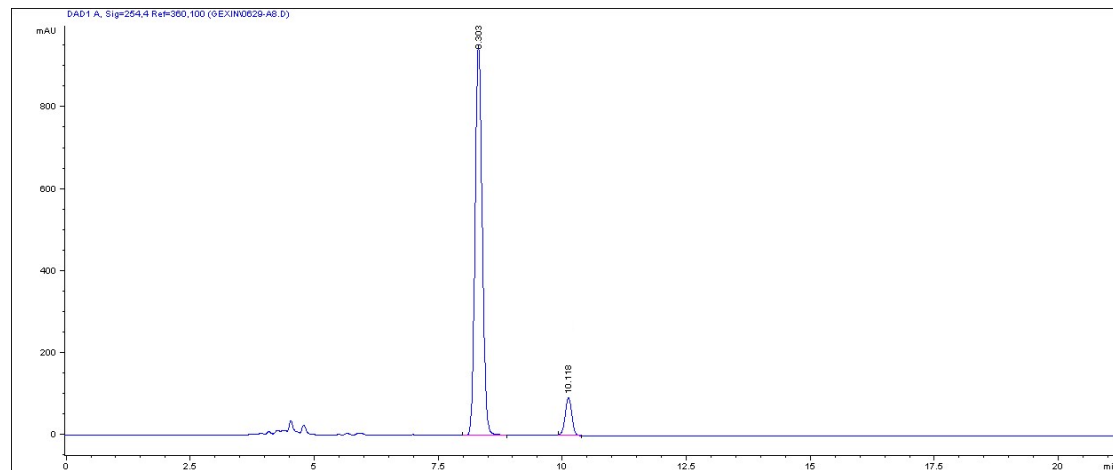
CHIRALCEL OD-H (Hexane/iPrOH = 95/5 1.0 mL/min, 254 nm)

Racemic



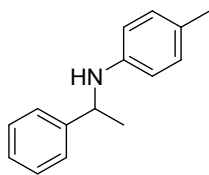
Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
8.046	411.8	3920.4	49.7
9.729	347.6	3972.2	50.3

Optically active (86% ee)



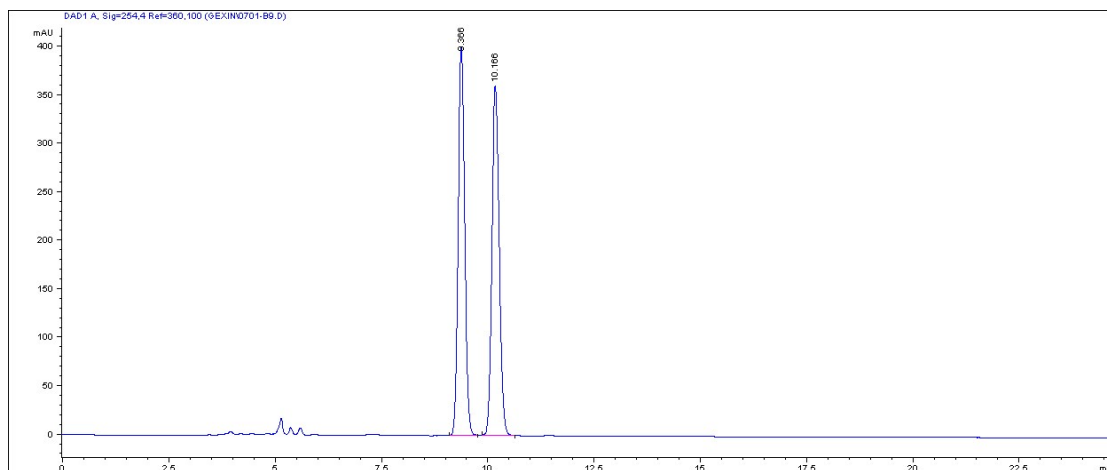
Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
8.303	953.0	9503	92.8
10.118	98.7	737.3	7.2





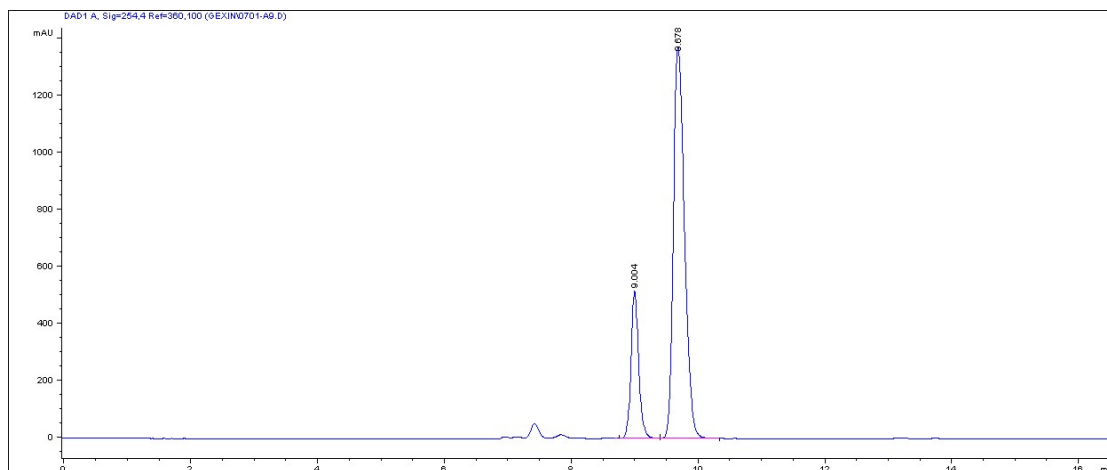
CHIRALCEL OD-H (Hexane/iPrOH = 99/1 1.0 mL/min, 254 nm)

Racemic

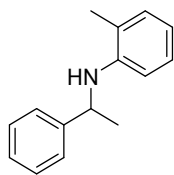


Ret. Time [min]	Height [mAU]	Area [mAU*s]	Rel. Area [%]
9.366	401	4275	49.9
10.166	361.1	4291.1	50.1

Optically active (52% ee)

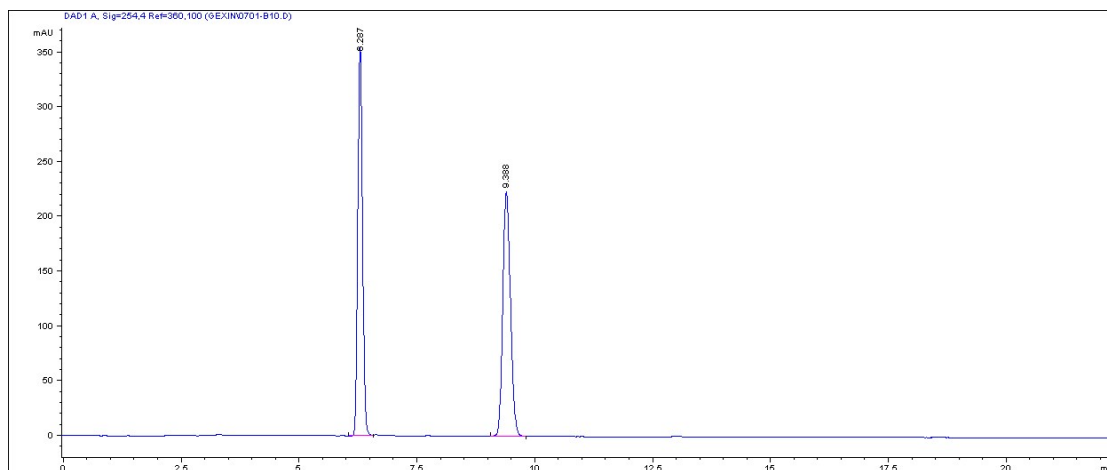


Ret. Time [min]	Height [mAU]	Area [mAU*s]	Rel. Area [%]
9.004	507.4	5244.7	23.9
9.678	1372.2	16699.8	76.1



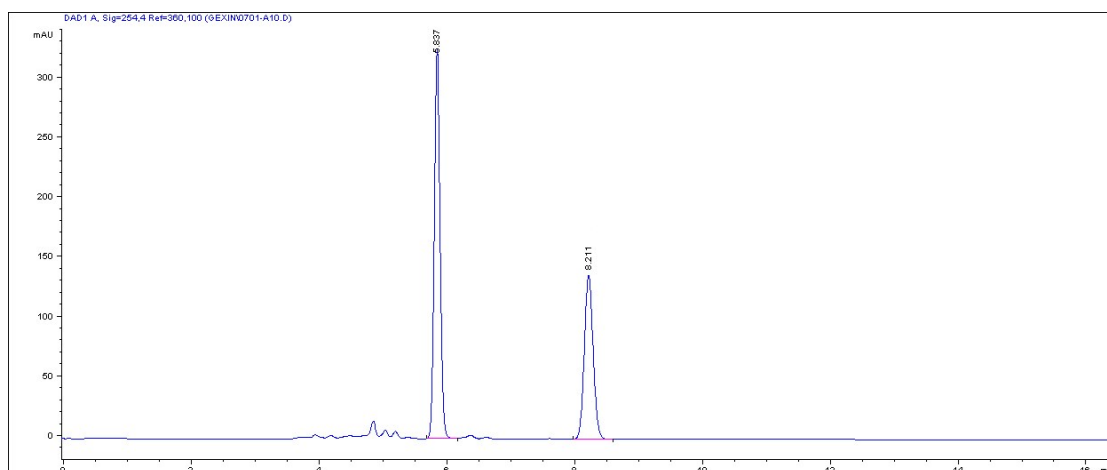
CHIRALCEL OD-H (Hexane/iPrOH = 99/1 1.0 mL/min, 254 nm)

Racemic



Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
6.287	356.2	2505.1	49.7
9.388	223.6	2533.7	50.3

Optically active (25% ee)



Ret. Time [min]	Height [mAu]	Area [mAU*s]	Rel. Area [%]
5.837	327	2086.4	62.6
8.211	136.4	1264.5	37.4