Half-Sandwich Chiral Rare-Earth Metal Complexes with Linked Tridentate Amido-Indenyl Ligand: Synthesis, Characterization, and Catalytic Properties for Intramolecular Hydroamination

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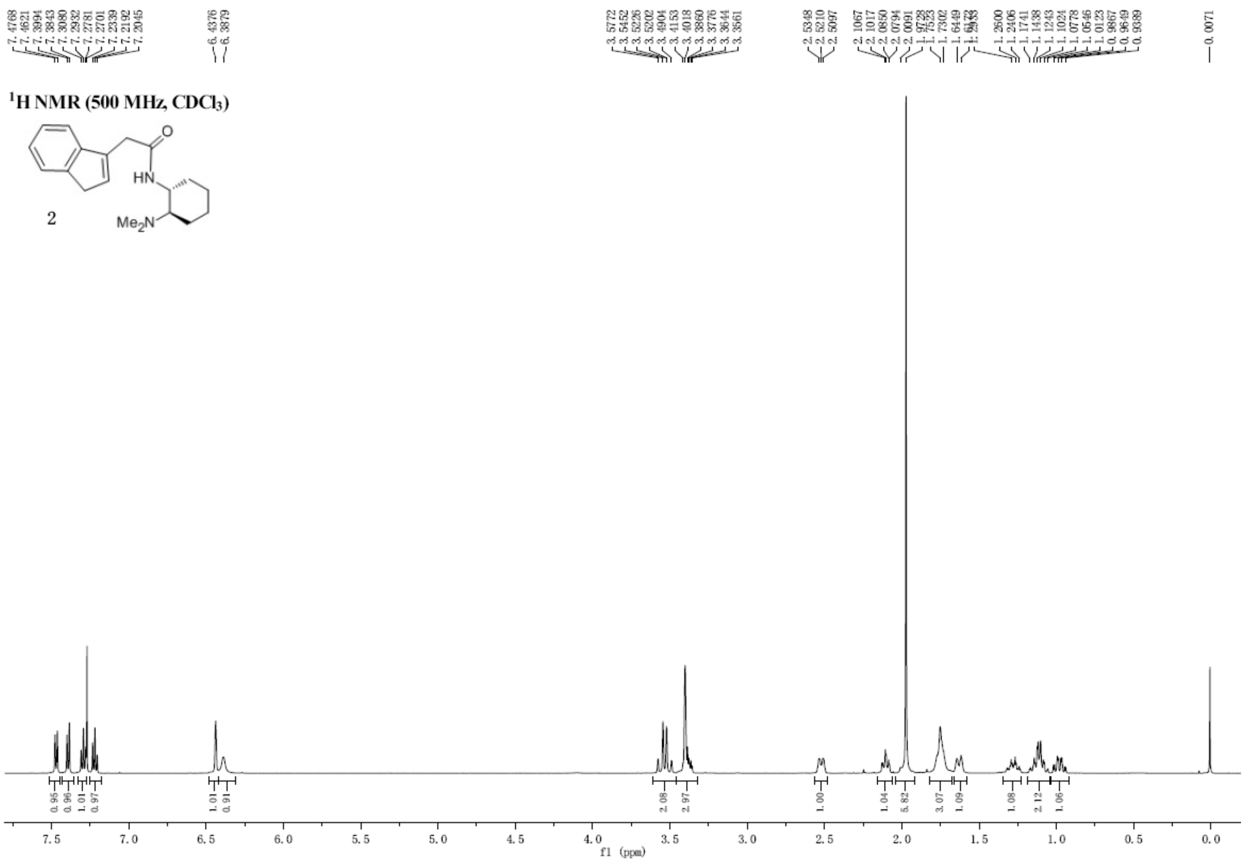
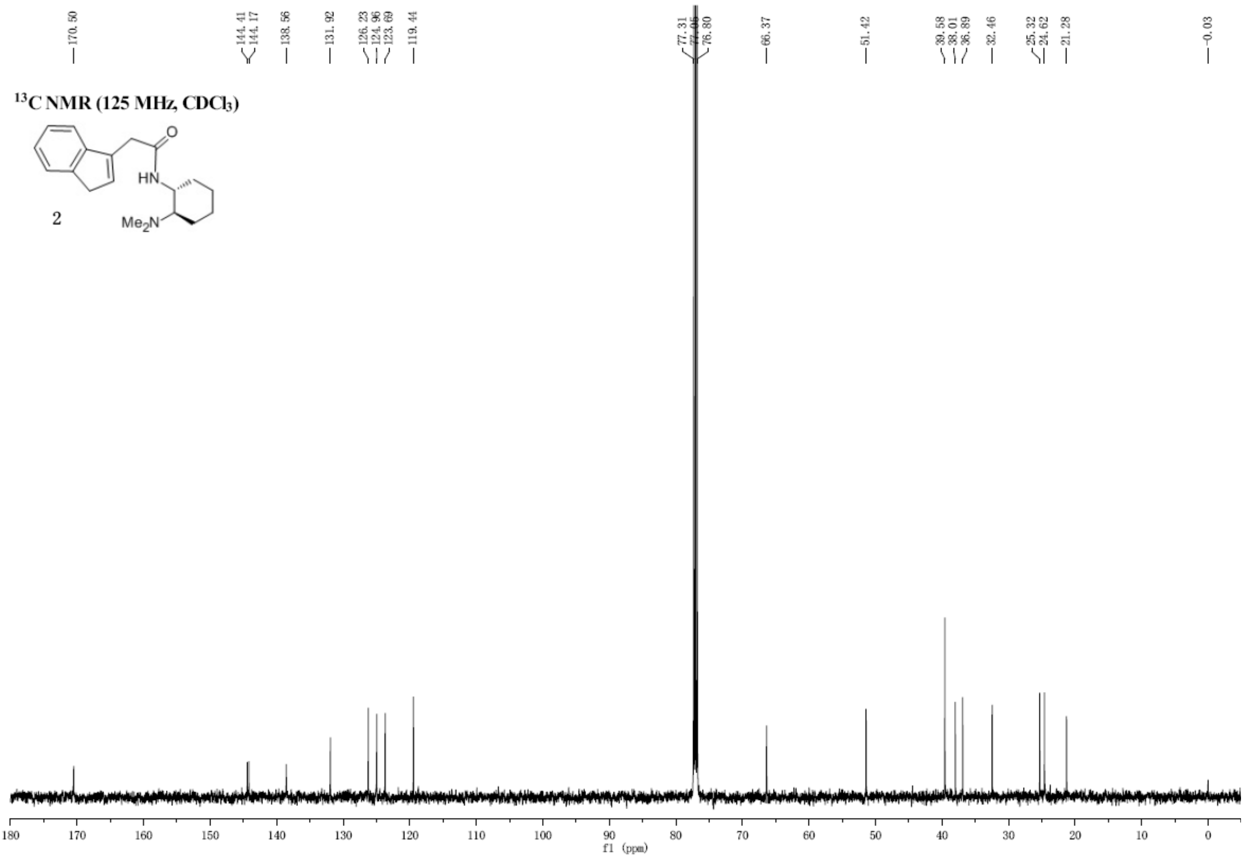
Electronic Supplementary Information

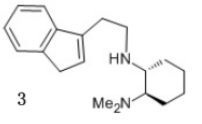
Content

Copies of ^{1}H NMR and ^{13}C NMR for ligand





^{1}H NMR (500 MHz, C_6D_6)



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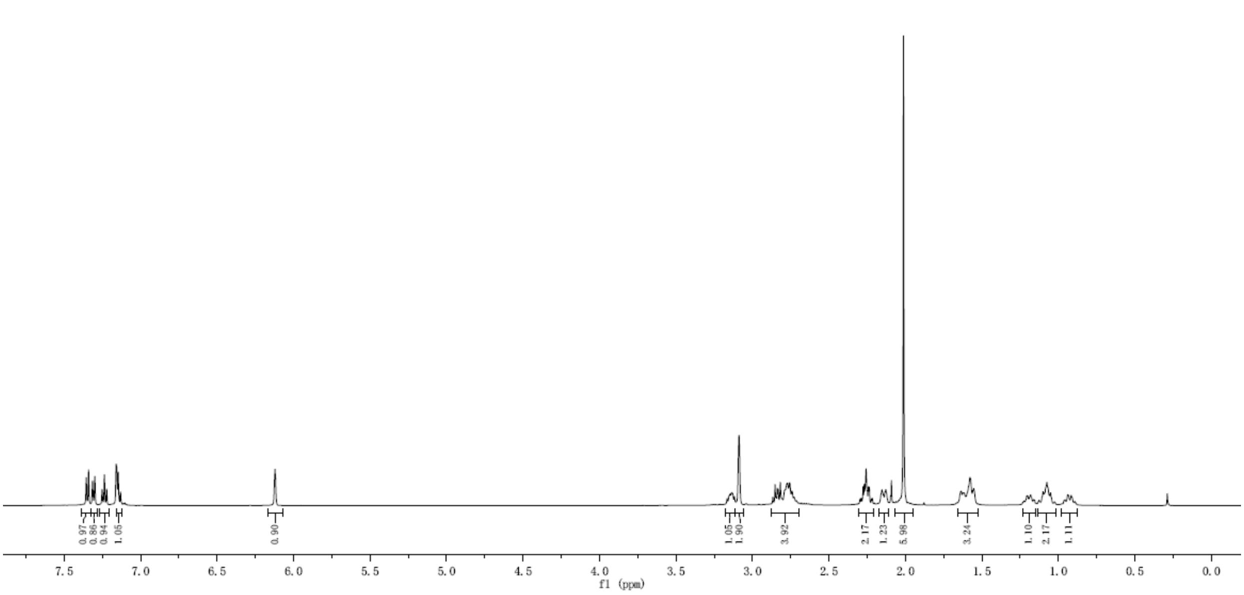
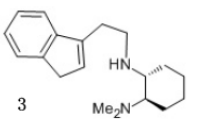


^{13}C NMR (75.5 MHz, $CDCl_3$)



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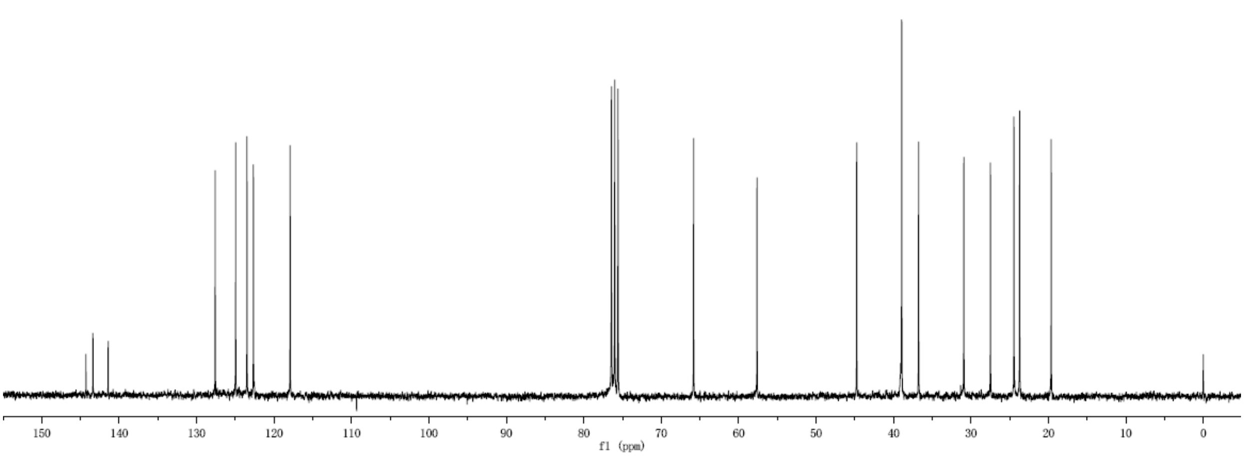
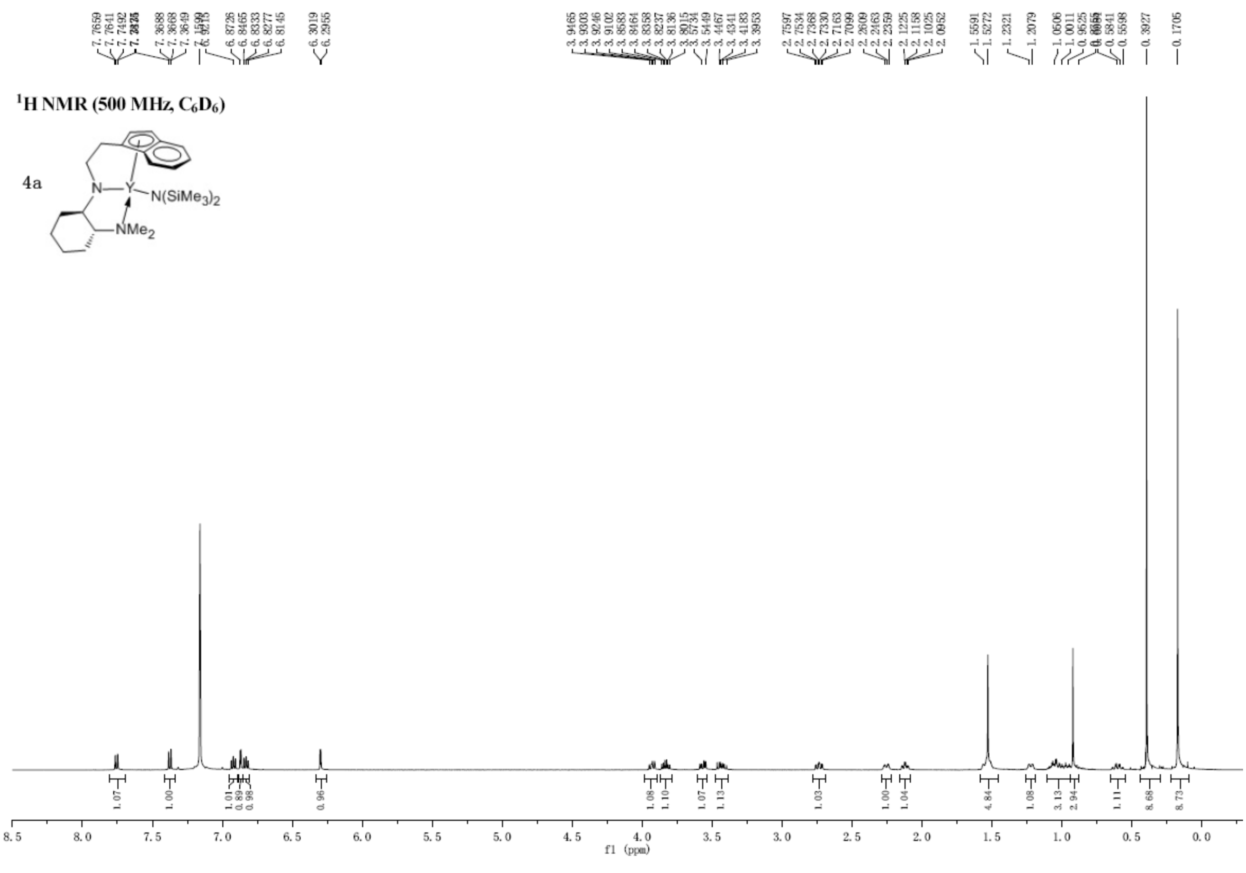
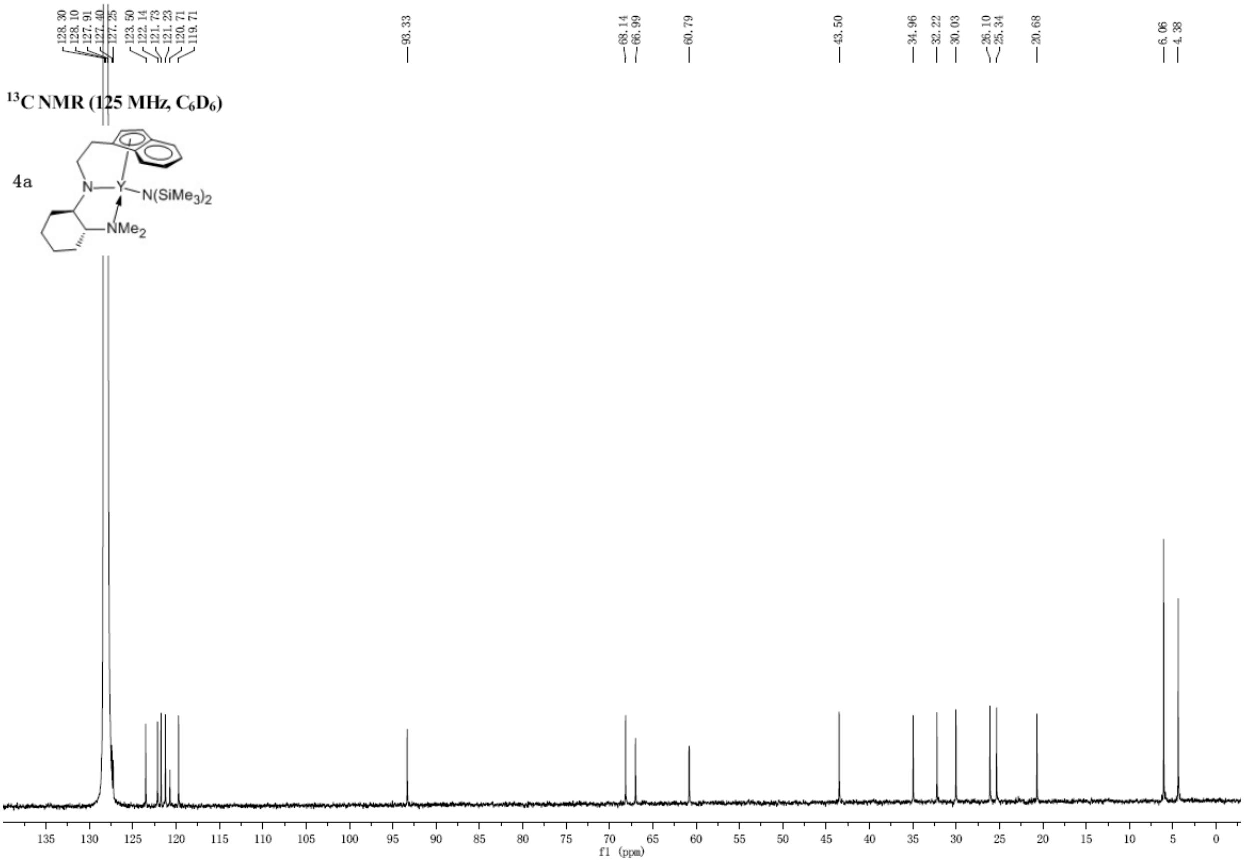


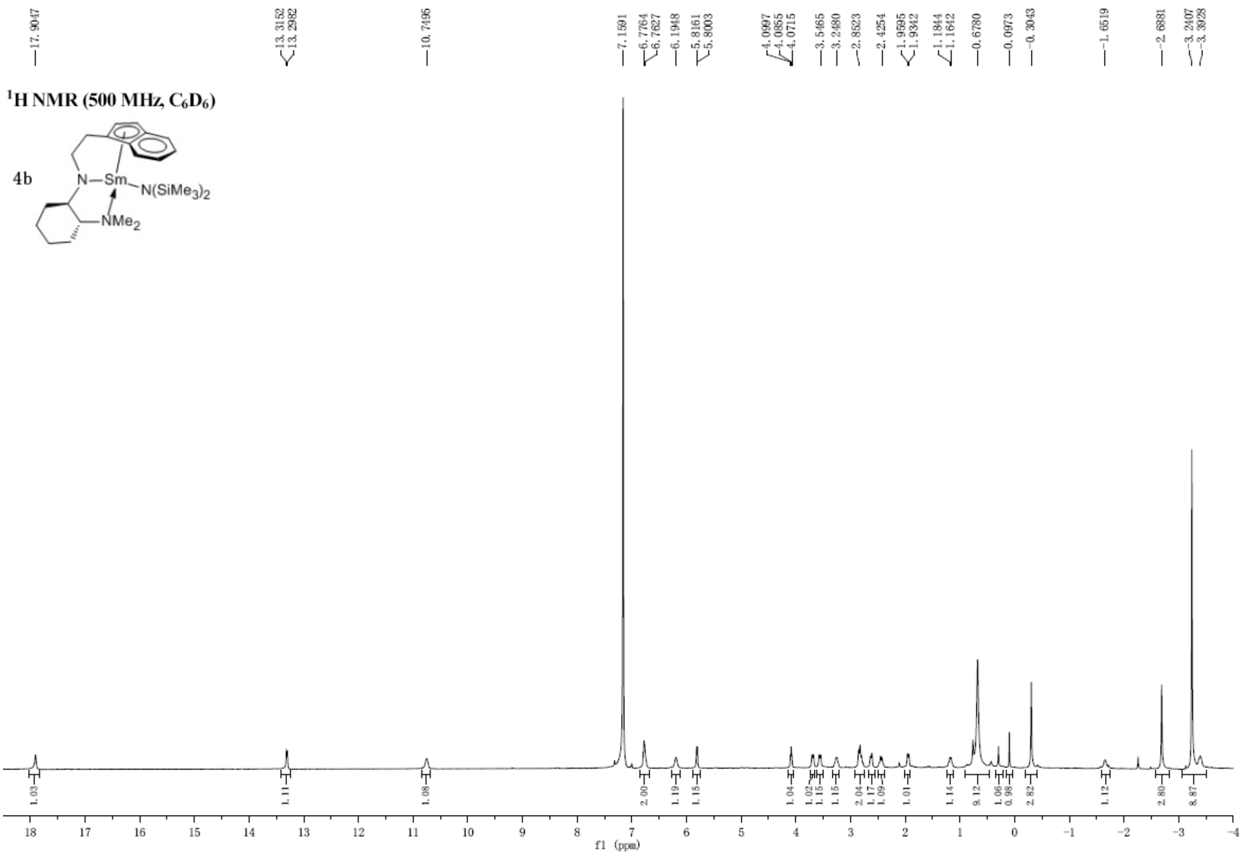
Copies of ^{1}H NMR and ^{13}C NMR for rare-earth metal complexes





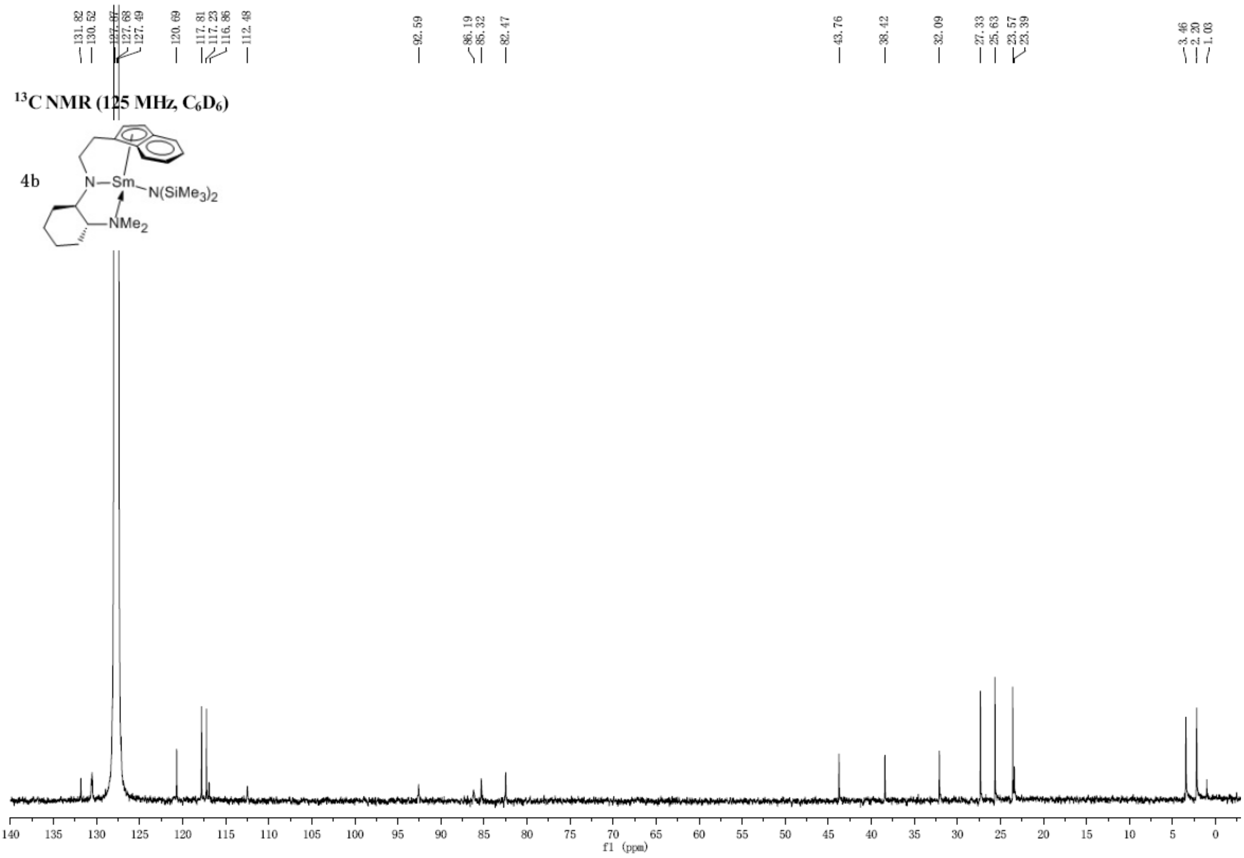
^{1}H NMR (500 MHz, C_6D_6)

4b



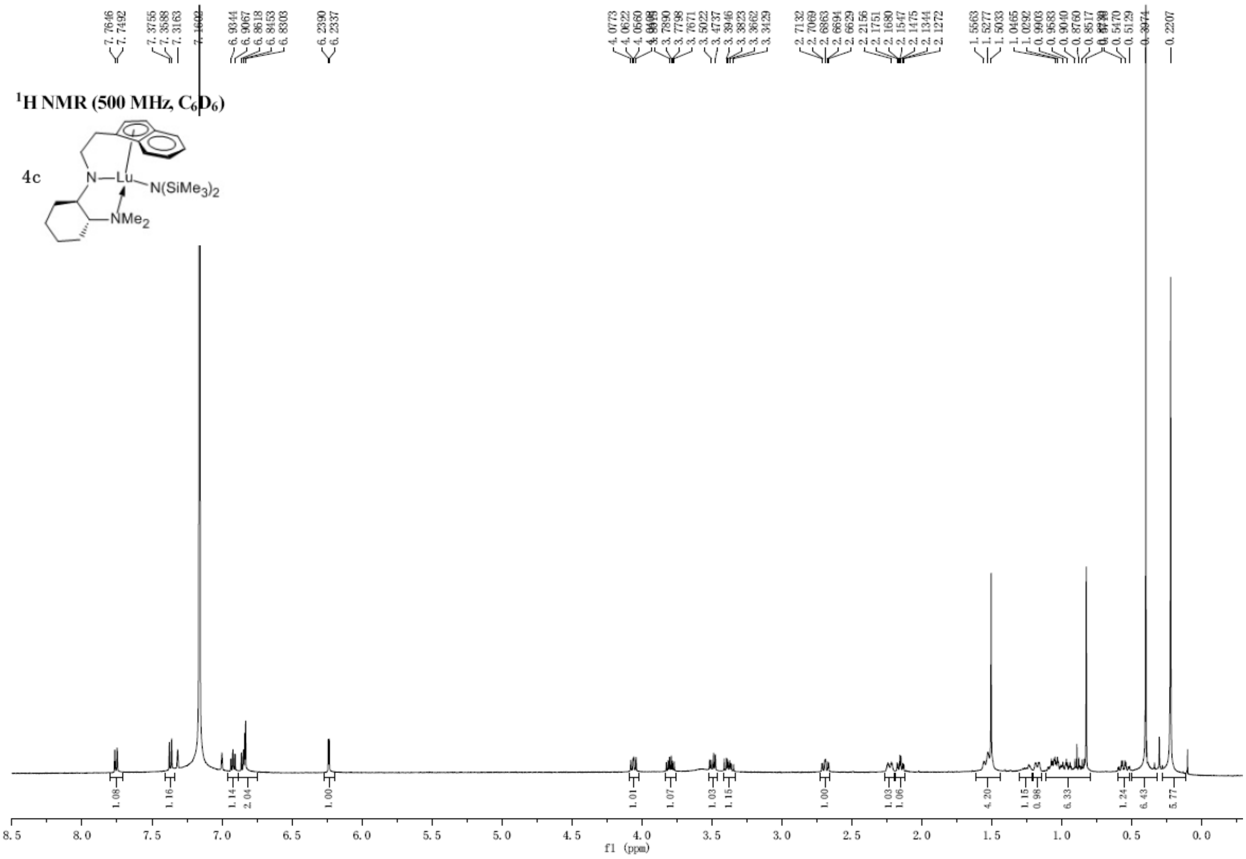
^{13}C NMR (125 MHz, C_6D_6)

4b



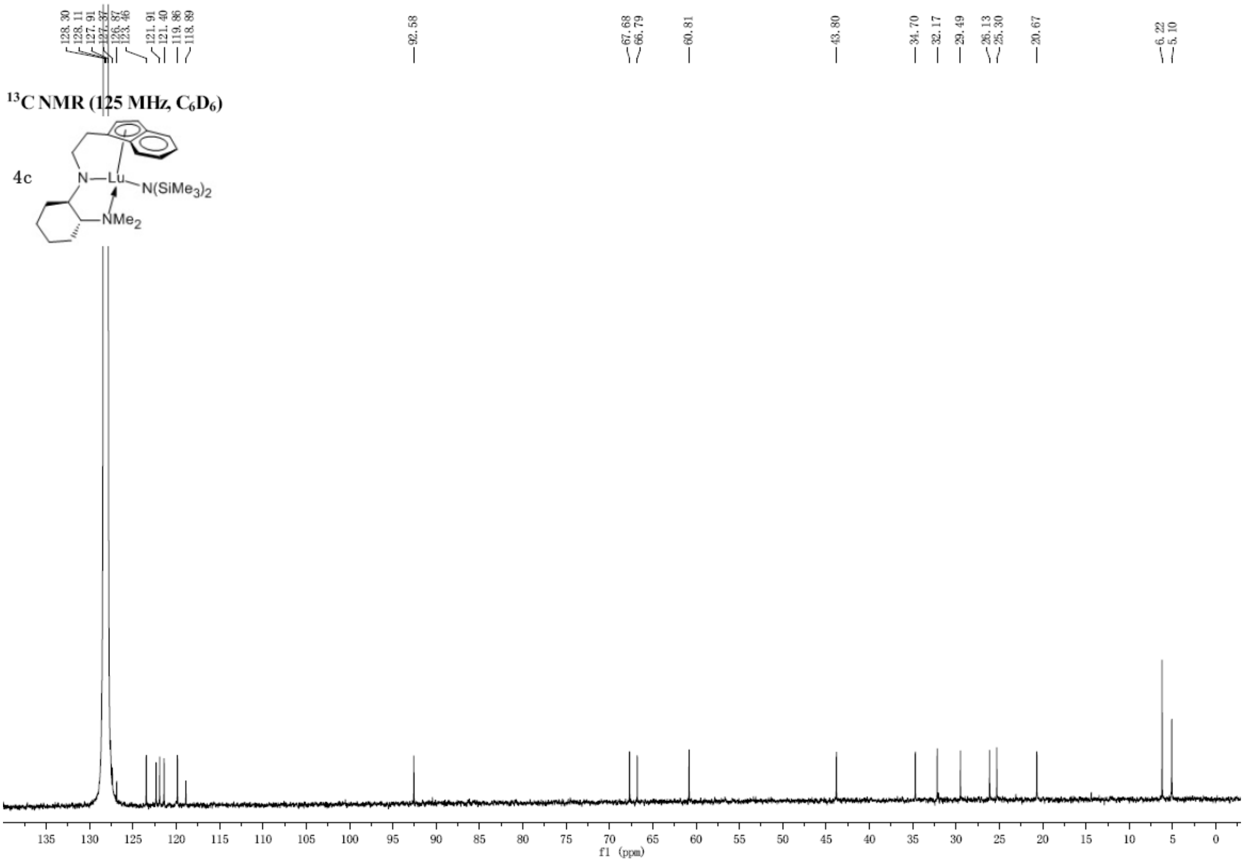
[1]H NMR (500 MHz, C_6D_6)

4c



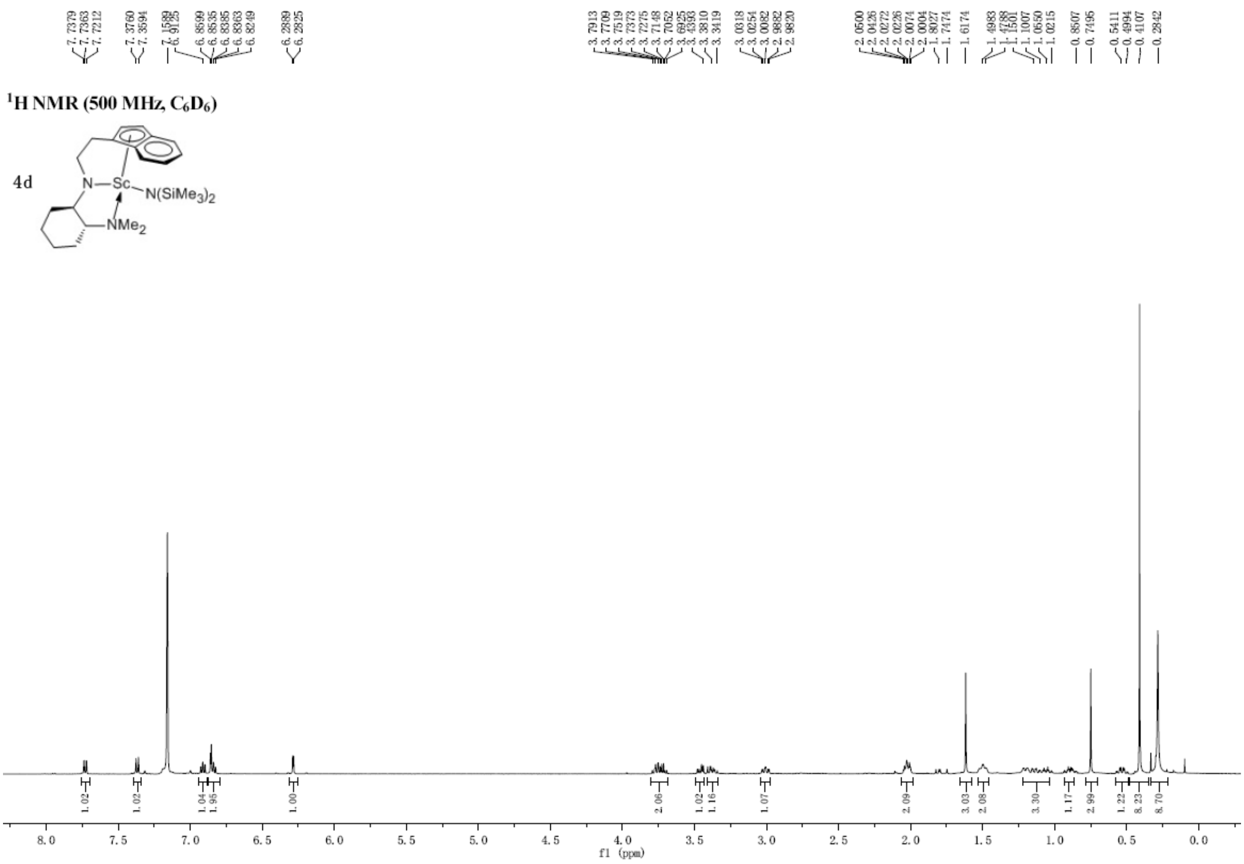
[13]C NMR (125 MHz, C_6D_6)

4c



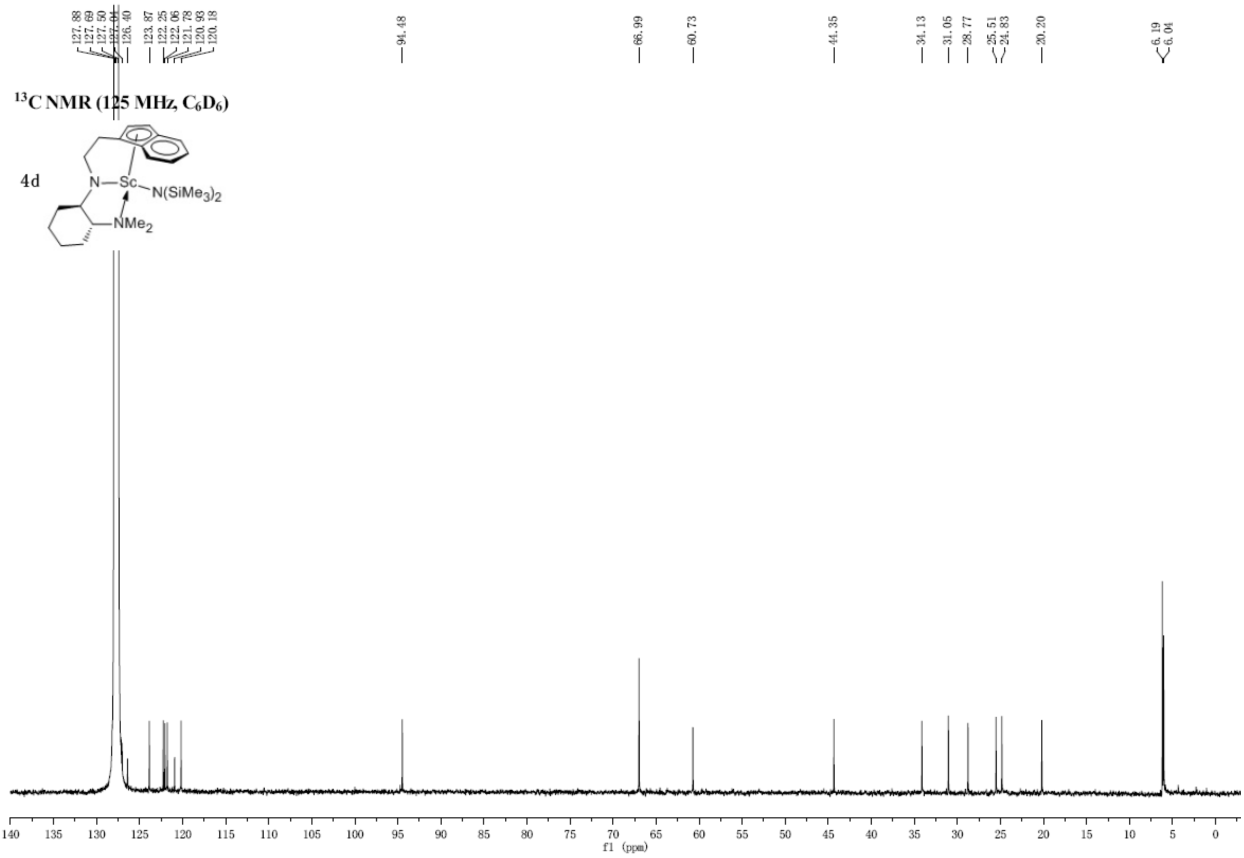
^{1}H NMR (500 MHz, C_6D_6)

4d

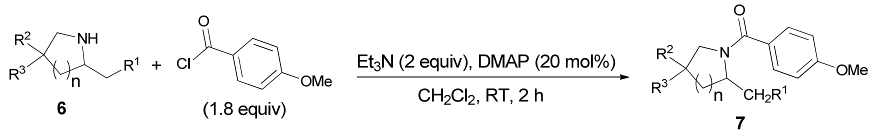


^{13}C NMR (125 MHz, C_6D_6)

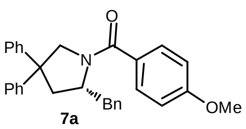
4d



Determination of the enantiomeric excess (*ee*) values

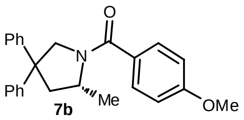


The enantiomeric excess values were determined by HPLC analysis of **7** using AS-H, AD-H, or OJ-H column. Typical procedure of derivatization: To a solution of the corresponding cyclized product **6** (0.16 mmol) in CH_2Cl_2 (5 mL) was added 4-dimethylaminopyridine (DMAP, 4.8 mg, 0.04 mmol), triethylamine (45 μL, 0.3 mmol), and 4-methoxybenzoyl chloride (40 μL, 0.3 mmol) at ambient temperature. After stirring for 2 h, a saturated aqueous solution of ammonium chloride (5 mL) was poured into the reaction mixture and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3×5 mL). The combined organic layers were washed with saturated aqueous solution of ammonium chloride (5 mL), dried over anhydrous magnesium sulfate, filtered, and concentrated in vacuo. The crude product was purified by preparative TLC (silica gel).



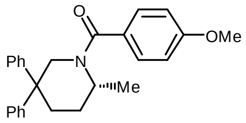
7a

(**7a**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 94% yield (87% ee). The ee was determined by HPLC analysis using a Chiralcel OD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 6.2 (minor), 8.1 (major) min). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.52 (d, J = 8.8 Hz, 2H), 7.36–7.08 (m, 11H), 7.06–6.91 (m, 6H), 4.39–4.25 (m, 2H), 3.87 (s, 3H), 3.57 (d, J = 11.1 Hz, 1H), 3.31 (dd, J = 13.2, 3.0 Hz, 1H), 3.04 (dd, J = 12.9, 8.1 Hz, 1H), 2.74–2.64 (m, 1H), 2.49 (dd, J = 12.6, 10.5 Hz, 1H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 169.9, 161.3, 145.2, 144.1, 138.1, 130.0, 129.5, 129.1, 128.6, 128.5, 128.4, 126.7, 126.6, 126.5, 126.4, 113.7, 60.2, 57.3, 55.4, 53.4, 42.0, 38.5. HRMS (ESI) calcd for $C_{31}H_{29}NO_2Na$ ($[M + Na]^+$) 470.2096, found 470.2095.

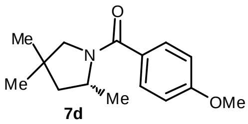


7b

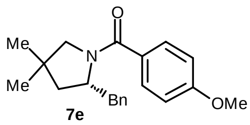
(**7b**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 92% yield (68% ee). The ee was determined by HPLC analysis using a Chiralcel AS-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 37.9 (major), 11.6 (minor) min). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.54 (d, J = 8.2 Hz, 2H), 7.33–7.10 (m, 8H), 7.03 (d, J = 7.5 Hz, 2H), 6.94 (d, J = 8.2 Hz, 2H), 4.38 (d, J = 11.1 Hz, 1H), 4.17–4.01 (m, 1H), 3.94–3.76 (m, 4H), 2.96–2.81 (m, 1H), 2.36 (t, J = 11.3 Hz, 1H), 1.45 (d, J = 5.8 Hz, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 168.8, 160.2, 144.4, 143.3, 128.5, 128.3, 127.6, 127.5, 125.7, 125.6, 125.5, 125.3, 112.6, 58.7, 54.3, 52.6, 51.5, 44.6, 18.8. HRMS (ESI) calcd for $C_{25}H_{26}NO_2$ ($[M + H]^+$) 372.1964, found 372.1964.


7c

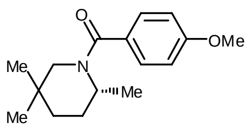
(**7c**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 94% yield (45% ee). Mp 187–188 °C. The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 8.2 (major), 20.6 (minor) min). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.44–7.12 (m, 12H), 6.93–6.83 (m, 2H), 5.41–5.10 (brs, 1H), 4.34–4.07 (brs, 1H), 3.81 (s, 3H), 3.17 (d, *J* = 13.5 Hz, 1H), 2.66–2.49 (m, 2H), 1.84–1.66 (m, 1H), 1.43 (d, *J* = 13.5 Hz, 1H), 1.20 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 171.0, 160.4, 147.4, 143.8, 129.3, 128.5, 128.4, 128.0, 127.7, 126.5, 126.4, 126.1, 113.8, 55.3, 48.4, 47.2, 45.9, 29.5, 26.2, 17.2. HRMS (ESI) calcd for $C_{26}H_{28}NO_2$ ($[M + H]^+$) 386.2120, found 386.2120.


7d

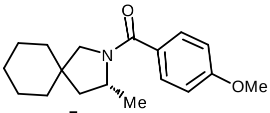
(**7d**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a viscous colorless oil in 91% yield (56% ee). The ee was determined by HPLC analysis using a Chiralcel AS-H column (4/1 hexane/*i*-PrOH; flow rate 0.8 mL/min; λ = 254 nm; t_R = 9.03 (minor), 11.2 (major) min). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.4 Hz, 2H), 6.96–6.84 (m, 2H), 4.44–4.23 (m, 1H), 3.84 (s, 3H), 3.39–3.25 (m, 1H), 3.24–3.07 (m, 1H), 1.99–1.85 (m, 1H), 1.49–1.25 (m, 4H), 1.06 (s, 3H), 0.90 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 169.8, 161.0, 129.6, 113.4, 62.9, 55.3, 53.0, 47.6, 38.3, 25.8, 25.5, 20.3. HRMS (ESI) calcd for $C_{15}H_{21}NO_2Na$ ($[M + Na]^+$) 270.1470, found 270.1470.


7e

(**7e**): Purified by preparative TLC (petroleum ether/ethyl acetate = 6/1) to afford the product as a viscous colorless oil in 90% yield (52% ee). The ee was determined by HPLC analysis using a Chiralcel AS-H column (2/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 9.47 (major), 6.83 (minor) min). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56–7.48 (m, 2H), 7.34–7.18 (m, 5H), 6.95–6.87 (m, 2H), 4.65–4.50 (m, 1H), 3.84 (s, 3H), 3.27 (dd, *J* = 12.9, 2.6 Hz, 1H), 3.13 (d, *J* = 13.2 Hz, 1H), 3.05–2.88 (m, 2H), 1.70 (ddd, *J* = 12.6, 7.4, 1.7 Hz, 1H), 1.56 (dd, *J* = 12.5, 10.0 Hz, 1H), 0.95 (s, 3H), 0.84 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 169.9, 161.1, 138.4, 129.9, 129.6, 129.2, 128.2, 126.2, 113.4, 63.5, 57.9, 55.3, 43.9, 38.9, 38.0, 25.6, 25.5. HRMS (ESI) calcd for $C_{21}H_{26}NO_2$ ($[M + H]^+$) 324.1964, found 324.1963.

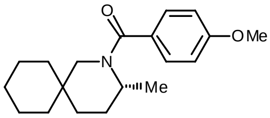

7f

(**7f**): Purified by preparative TLC (petroleum ether/ethyl acetate = 5/1) to afford the product as a viscous colorless oil in 93% yield (41% ee). The ee was determined by HPLC analysis using a Chiralcel AS-H column (3/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 10.12 (major), 12.32 (minor) min). ^{1}H NMR (300 MHz, $CDCl_3$): *δ* 7.31 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 1H), 4.79–4.27 (brs, 1H), 3.83 (s, 3H), 3.76–3.36 (brs, 1H), 2.79 (d, *J* = 12.9 Hz, 1H), 2.00–1.83 (m, 1H), 1.65–1.50 (m, 1H), 1.42–1.24 (m, 2H), 1.20 (d, *J* = 6.9 Hz, 3H), 0.93 (s, 3H), 0.90 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$): *δ* 170.9, 160.2, 129.4, 128.3, 113.7, 55.3, 32.4, 31.4, 29.1, 26.1, 23.0, 16.2. HRMS (ESI) calcd for $C_{16}H_{24}NO_2$ ($[M + H]^+$) 262.1807, found 262.1806.



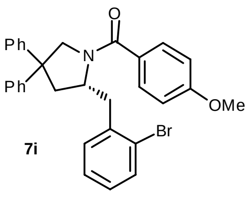
7g

(**7g**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a viscous colorless oil in 91% yield (65% ee). The ee was determined by HPLC analysis using a Chiralcel AS-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 12.52 (major), 7.61 (minor) min). ^{1}H NMR (300 MHz, $CDCl_3$): *δ* 7.51 (d, *J* = 8.1 Hz, 2H), 6.95–6.85 (m, 2H), 4.39–4.17 (brs, 1H), 3.84 (s, 3H), 3.36 (d, *J* = 10.2 Hz, 1H), 3.22 (d, *J* = 10.5 Hz, 1H), 2.12 (dd, *J* = 12.3, 7.5 Hz, 1H), 1.60–1.12 (m, 14H). ^{13}C NMR (75.5 MHz, $CDCl_3$): *δ* 169.9, 160.9, 129.5, 113.4, 60.7, 55.3, 52.1, 44.7, 42.3, 36.3, 33.4, 26.1, 23.8, 22.5, 20.3. HRMS (ESI) calcd for $C_{18}H_{26}NO_2$ ($[M + H]^+$) 288.1963, found 288.1961.



7h

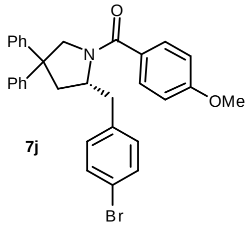
(**7h**): Purified by preparative TLC (petroleum ether/ethyl acetate = 5/1) to afford the product as a viscous colorless oil in 89% yield (43% ee). The ee was determined by HPLC analysis using a Chiralcel AS-H column (3/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 13.36 (major), 7.88 (minor) min). ^{1}H NMR (300 MHz, $CDCl_3$): *δ* 7.31 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 4.73–4.31 (brs, 1H), 3.84 (s, 3H), 2.70 (d, *J* = 12.8 Hz, 1H), 2.00–1.69 (m, 2H), 1.62–1.12 (m, 16H). ^{13}C NMR (75.5 MHz, $CDCl_3$): *δ* 170.9, 160.2, 129.6, 128.3, 113.7, 55.3, 38.3, 33.8, 30.9, 29.9, 26.5, 25.4, 21.6, 21.4, 16.4. HRMS (ESI) calcd for $C_{19}H_{28}NO_2$ ($[M + H]^+$) 302.2120, found 302.2119.



7i

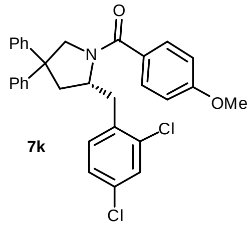
(**7i**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a viscous colorless oil in 90% yield (65% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 25.31 (major), 12.25 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): *δ* 7.58 (d, *J* = 7.9 Hz, 2H), 7.33 (d, *J* = 7.8 Hz, 2H), 7.25–7.02 (m, 10H), 7.01–6.92 (m, 4H), 4.43–4.32 (m, 2H), 3.89 (s, 3H), 3.67 (d, *J* = 12.1 Hz, 1H), 3.57 (d, *J* = 13.4 Hz, 1H), 3.28–3.21 (m, 1H), 2.75–2.68 (m, 1H), 2.63–2.55 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): *δ* 170.2, 161.4, 145.2, 144.0, 138.1, 132.9, 132.0, 129.7, 129.0, 128.65, 128.56, 128.1, 127.4, 126.7, 126.64, 126.55, 126.4, 125.4, 113.8, 60.1, 57.3, 55.4, 53.5, 42.0, 37.8. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{31}H_{29}BrNO_2$ 526.1382, found

526.1378.



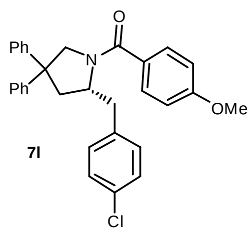
7j

(**7j**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 94% yield (80% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 32.58 (major), 12.82 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, J =8.6 Hz, 2H), 7.43 (d, J =8.2 Hz, 2H), 7.33–7.09 (m, 8H), 7.04 (d, J =7.9 Hz, 2H), 7.00–6.93 (m, 4H), 4.35 (dd, J = 11.2, 1.5 Hz, 1H), 4.29–4.21 (m, 1H), 3.88 (s,3H), 3.58 (d, J = 11.2 Hz, 1H), 3.25 (dd, J =13.2, 2.8 Hz, 1H), 3.02 (dd, J = 13.2, 8.1 Hz, 1H), 2.69 (ddd, J = 12.2, 6.3, 1.5 Hz), 2.43 (dd, J = 12.0, 11.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.1, 161.5, 145.1, 143.9, 137.1, 131.7, 131.5, 129.5, 128.9, 128.7, 128.6, 126.7, 126.65, 126.60, 126.3, 120.4, 113.8, 60.2, 57.1, 55.4, 53.4, 42.0, 37.9. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{31}H_{29}BrNO_2$ 526.1382, found 526.1380.



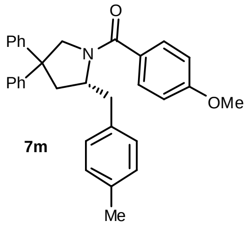
7k

(**7k**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 86% yield (51% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 12.01 (major), 8.1 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 (d, J = 8.6 Hz, 2H), 7.41 (d, J = 1.9 Hz, 1H), 7.28–7.10 (m, 8H), 7.06 (d, J = 7.4 Hz, 2H), 7.00–6.94 (m, 4H), 4.40 (dd, J = 11.0, 1.2 Hz, 1H), 4.34–4.26 (m, 1H), 3.88 (s, 3H), 3.67 (d, J = 11.2 Hz, 1H), 3.49 (dd, J = 13.5, 3.5 Hz, 1H), 3.19 (dd, J = 13.5, 7.7 Hz, 1H), 2.75–2.68 (m, 1H), 2.49 (dd, J = 12.0, 11.2 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.3, 161.5, 145.1, 143.9, 135.3, 135.0, 132.9, 132.8, 129.6, 129.3, 128.8, 128.7, 128.6, 127.1, 126.71, 126.67, 126.6, 126.3, 113.8, 60.1, 57.2, 55.4, 53.5, 42.0, 34.9. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{31}H_{28}Cl_2NO_2$ 516.1497, found 516.1492.



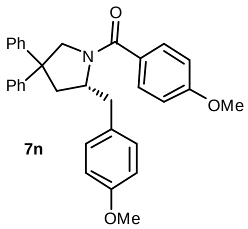
7l

(**7l**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 92% yield (80% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 30.1 (major), 10.6 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, J = 8.7 Hz, 2H), 7.28 (d, J = 8.3 Hz, 2H), 7.25–7.10 (m, 8H), 7.04 (d, J = 7.3 Hz, 2H),, 7.00–6.94 (m, 4H), 4.35 (dd, J = 11.2, 1.9 Hz, 1H), 4.30–4.22 (m, 1H), 3.88 (s, 3H), 3.57 (d, J = 11.1 Hz, 1H), 3.26 (dd, J = 13.2, 2.8 Hz, 1H), 3.04 (dd, J = 13.2, 8.1 Hz, 1H), 2.72–2.66 (m, 1H), 2.43 (dd, J = 12.2, 10.9 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.1, 161.4, 145.1, 144.0, 136.6, 132.3, 131.3, 129.5, 128.9, 128.7, 128.6, 128.5, 126.7, 126.65, 126.60, 126.3, 113.8, 60.2, 57.2, 55.4, 53.4, 42.0, 37.8. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{31}H_{29}ClNO_2$ 482.1887, found 482.1884.



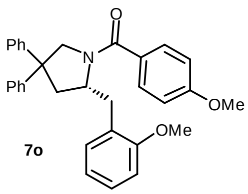
7m

(**7m**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 93% yield (67% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 31.03 (major), 14.44 (minor) min).^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56–7.50 (m, 2H), 7.24–7.08 (m, 10H), 7.07–7.01 (m, 2H), 7.00–6.92 (m, 4H), 4.36–4.25 (m, 2H), 3.87 (s, 3H), 3.58 (d, J = 11.2 Hz, 1H), 3.26 (dd, J = 13.1, 2.7 Hz, 1H), 3.01 (dd, J = 13.2, 8.1 Hz, 1H), 2.73–2.65 (m, 1H), 2.48 (dd, J = 12.3, 10.7 Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.9, 161.3, 145.3, 144.2, 135.8, 135.0, 129.9, 129.5, 129.2, 129.0, 128.6, 128.5, 126.7, 126.6, 126.5, 126.4, 113.7, 60.2, 57.4, 55.4, 53.4, 42.0, 38.1, 21.1. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{32}H_{32}NO_2$ 462.2433, found 462.2430.



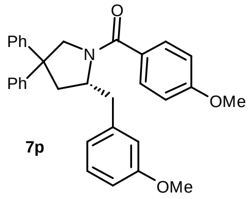
7n

(**7n**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 95% yield (28% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 26.67 (major), 10.33 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, J = 8.0 Hz, 2H), 7.33–7.09 (m, 9H), 7.07–6.92 (m, 5H), 6.84 (d, J = 7.7 Hz, 2H), 4.37–4.22 (m, 2H), 3.87 (s, 3H), 3.80 (s, 3H), 3.58–3.50 (m, 1H), 3.24–3.16 (m, 1H), 3.02 (dd, J = 13.2, 8.2Hz, 1H), 2.74–2.64 (m, 1H), 2.52–2.44 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.9, 161.3, 158.3, 145.3, 144.2, 130.9, 130.1, 129.5, 129.2, 128.6, 128.5, 126.7, 126.6, 126.5, 126.4, 113.77, 113.74, 60.2, 57.4, 55.4, 55.3, 53.4, 41.9, 37.5. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{32}H_{32}NO_3$ 478.2382, found 478.2381.

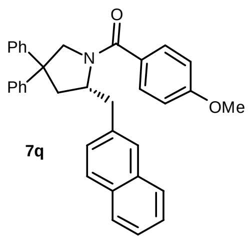


7o

(**7o**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 96% yield (61% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 38.05 (major), 9.4 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.53 (d, J = 8.0 Hz, 2H), 7.32–7.09 (m, 10H), 7.07–6.85 (m, 6H), 4.43–4.27 (m, 2H), 3.88 (s, 3H), 3.84 (s, 3H), 3.63–3.56 (m, 1H), 3.44–3.35 (m, 1H), 3.14–3.04 (m, 1H), 2.75–2.66 (m, 1H), 2.58–2.48 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.9, 161.2, 157.9, 145.4, 144.3, 131.8, 129.6, 129.4, 128.6, 128.4, 127.6, 126.8, 126.7, 126.48, 126.45, 126.42, 120.5, 113.7, 110.5, 60.1, 56.9, 55.43, 55.39, 53.5, 42.0, 32.0. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{32}H_{32}NO_3$ 478.2382, found 478.2377.

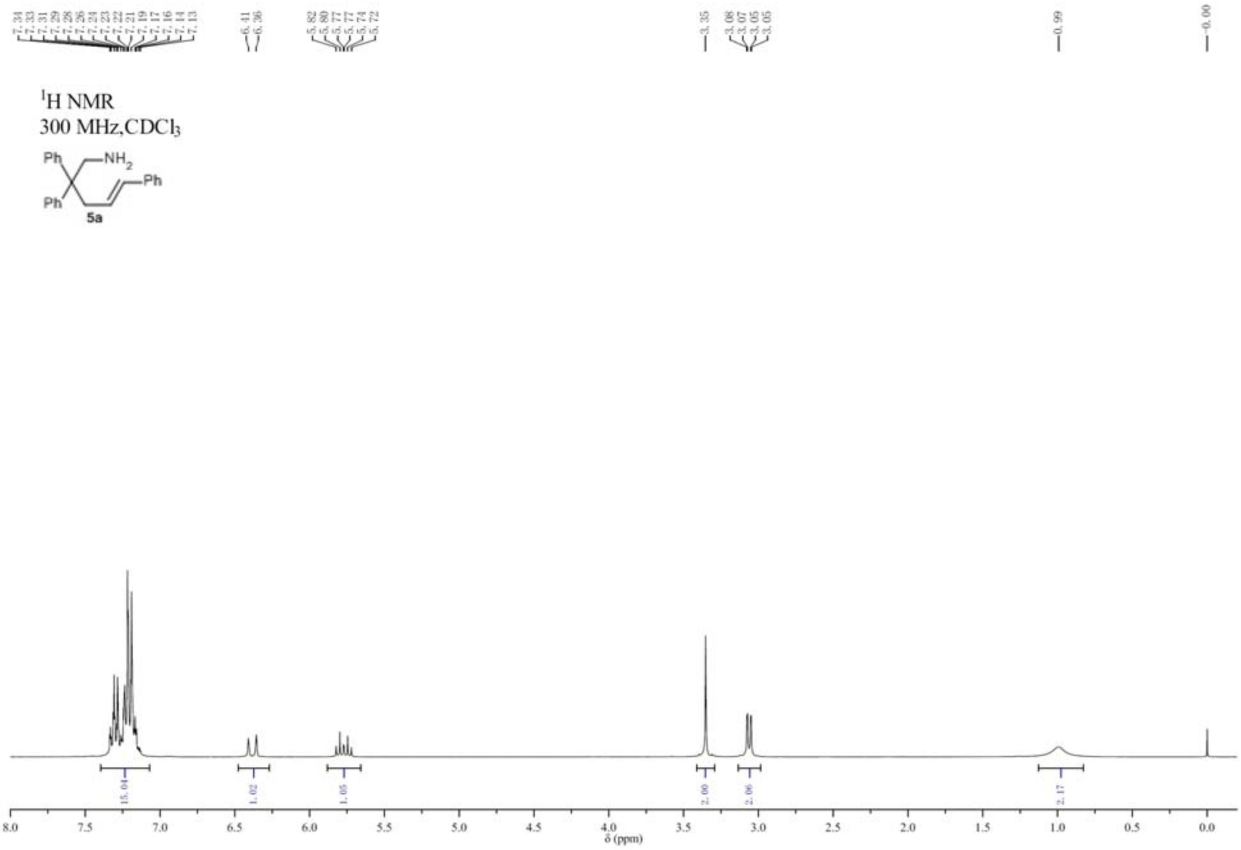


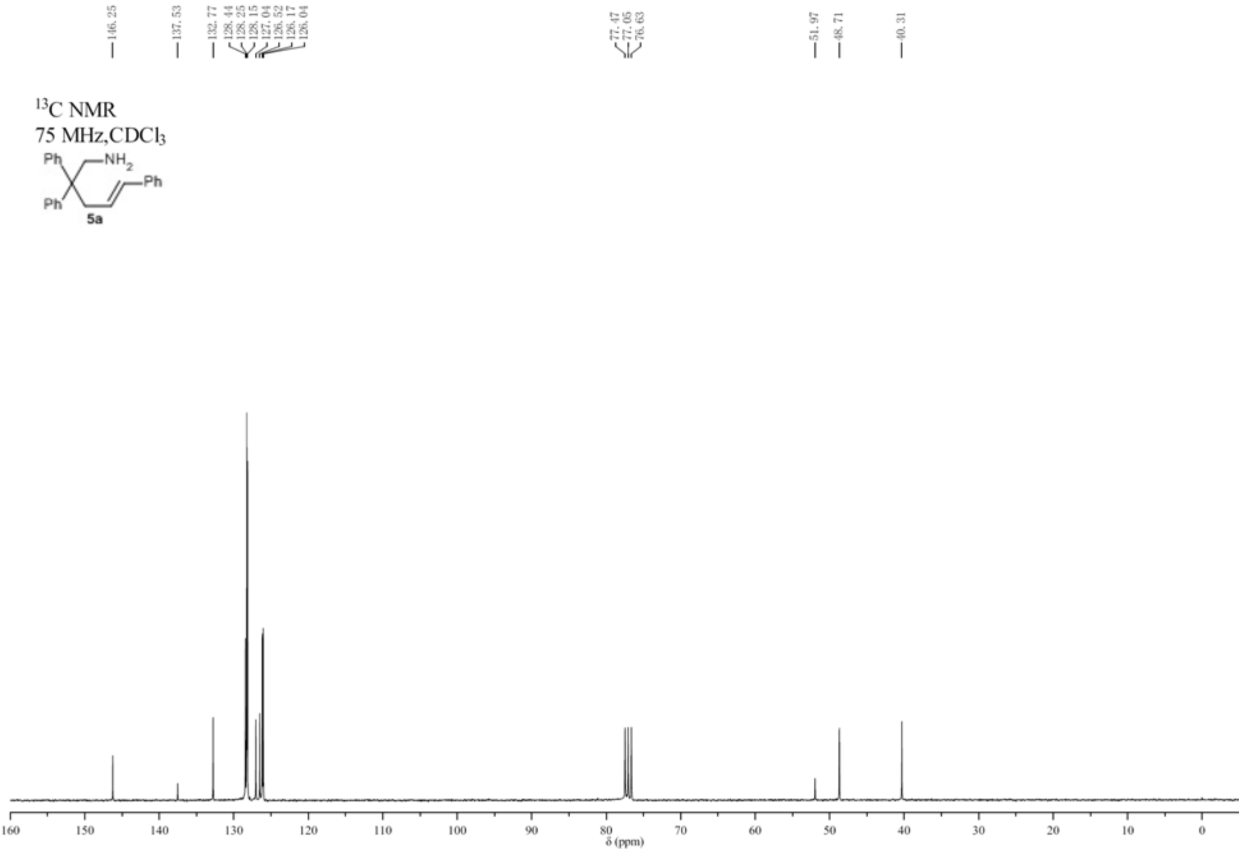
(**7p**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 88% yield (83% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 28.67 (major), 16.46 (minor) min)**.** ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.54 (d, J = 8.6 Hz, 2H), 7.25–7.10 (m, 7H), 7.05 (d, J = 7.4 Hz, 2H), 7.01–6.94 (m, 4H), 6.87 (d, J = 7.4 Hz, 1H), 6.83–6.76 (m, 2H), 4.38–4.27 (m, 2H), 3.88 (s, 3H), 3.77 (s, 3H), 3.63 (d, J = 11.2 Hz, 1H), 3.33 (dd, J = 13.1, 7.9 Hz, 1H), 2.98 (dd, J = 13.1, 8.4 Hz, 1H), 2.71 (ddd, J = 12.6, 6.6, 2.0 Hz, 1H), 2.50 (dd, J = 12.3, 10.7 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.9, 161.4, 159.6, 145.3, 144.1, 139.8, 129.6, 129.3, 129.1, 128.6, 128.5, 126.7, 126.6, 126.5, 126.4, 122.4, 115.3, 113.7, 112.1, 60.2, 57.3, 55.4, 55.2, 53.4, 42.1, 38.8. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{32}H_{32}NO_3$ 478.2382, found 478.2379.



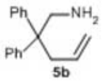
(**7q**): Purified by preparative TLC (petroleum ether/ethyl acetate = 4/1) to afford the product as a white solid in 90% yield (78% ee). The ee was determined by HPLC analysis using a Chiralcel AD-H column (1/1 hexane/*i*-PrOH; flow rate 1.0 mL/min; λ = 254 nm; t_R = 23.73 (major), 18.24 (minor) min). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.85–7.76 (m, 3H), 7.71 (s, 1H), 7.55 (d, J = 8.6 Hz, 2H), 7.50–7.41 (m, 3H), 7.22–7.10 (m, 6H), 7.06–6.92 (m, 6H), 4.43–4.31 (m, 2H), 3.89 (s, 3H), 3.56 (d, J = 11.2 Hz, 1H), 3.48 (dd, J = 13.1, 2.9 Hz, 1H), 3.21 (dd, J = 13.2, 8.2 Hz, 1H), 2.75–2.69 (m, 1H), 2.58–2.50 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.1, 161.4, 145.2, 144.0, 135.7, 133.6, 132.3, 129.6, 129.1, 128.6, 128.5, 128.4, 127.9, 127.7, 127.6, 126.7, 126.6, 126.5, 126.3, 126.0, 125.4, 113.8, 60.2, 57.5, 55.4, 53.4, 42.1, 38.7. HRMS (ESI–TOF) m/z $[M + H]^+$ calcd for $C_{35}H_{32}NO_2$ 498.2433, found 498.2432.

Copies of ^{1}H NMR and ^{13}C NMR for aminoalkene substrates

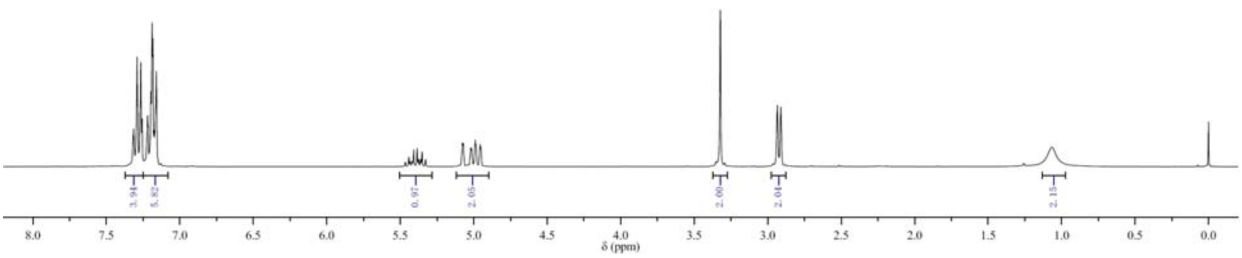




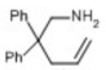
^1H NMR
300 MHz, $CDCl_3$



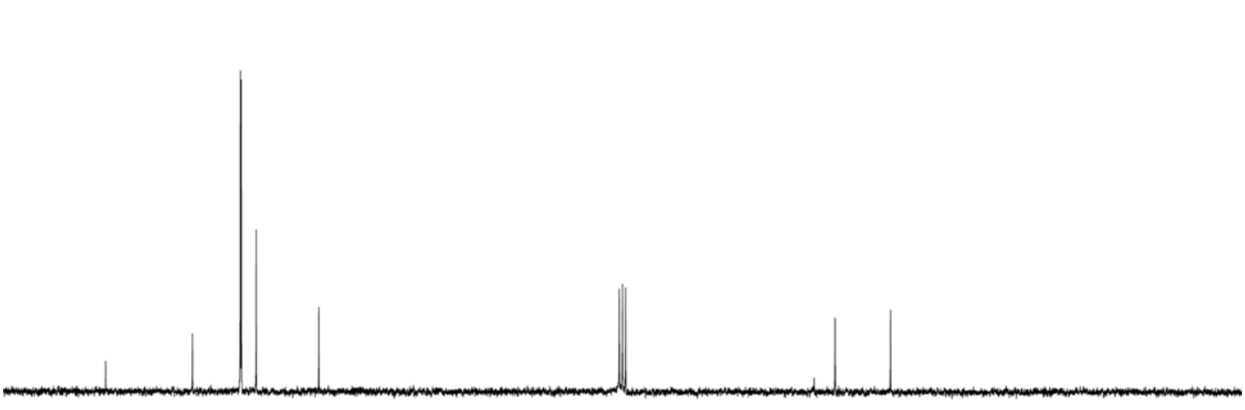
5b

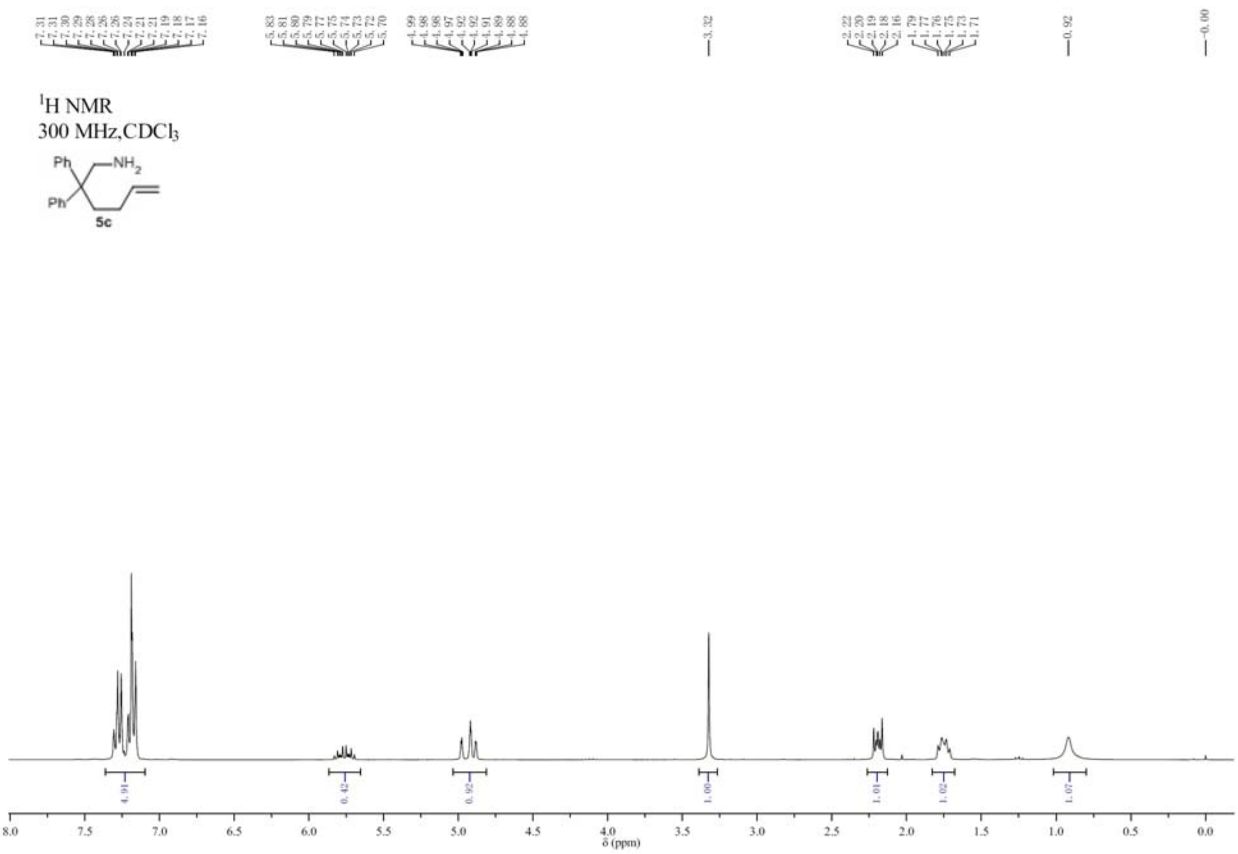


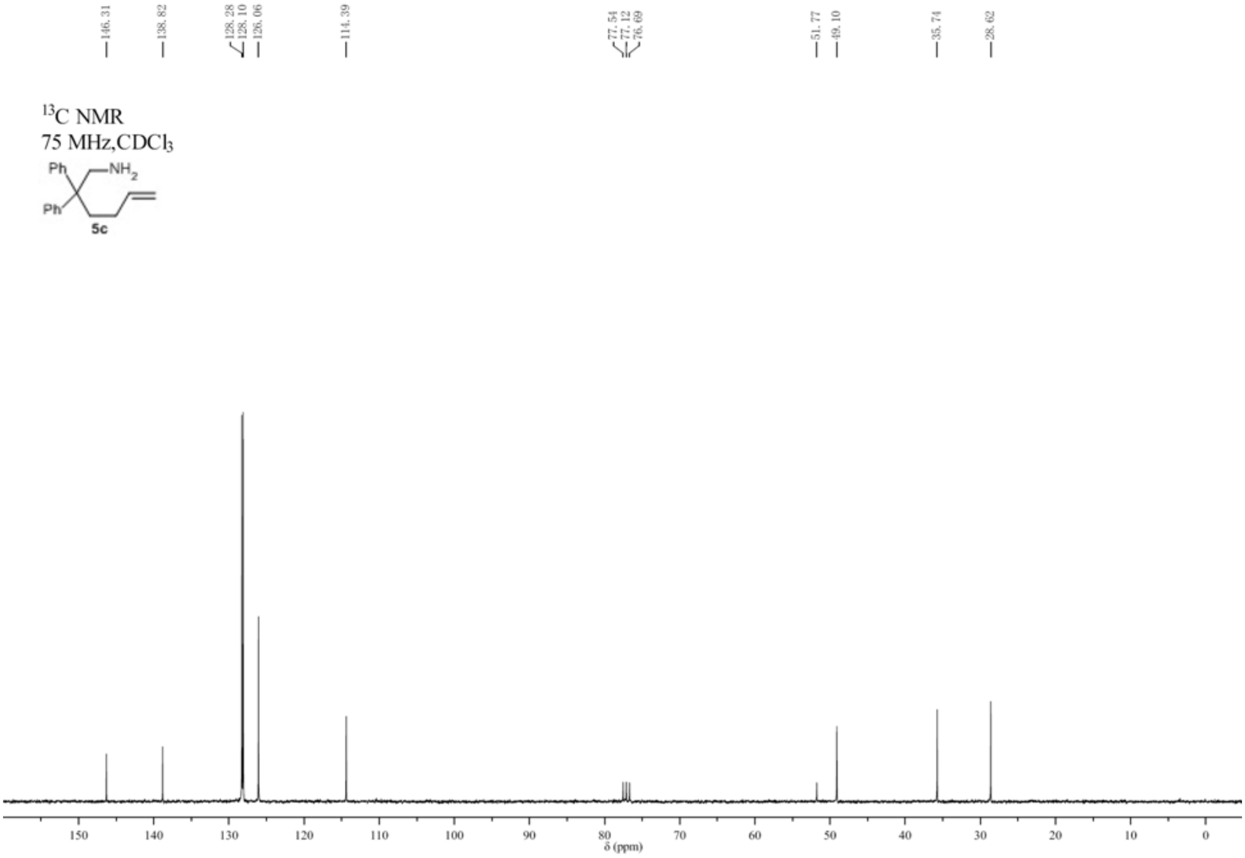
^{13}C NMR
75 MHz, $CDCl_3$



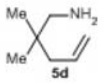
5b



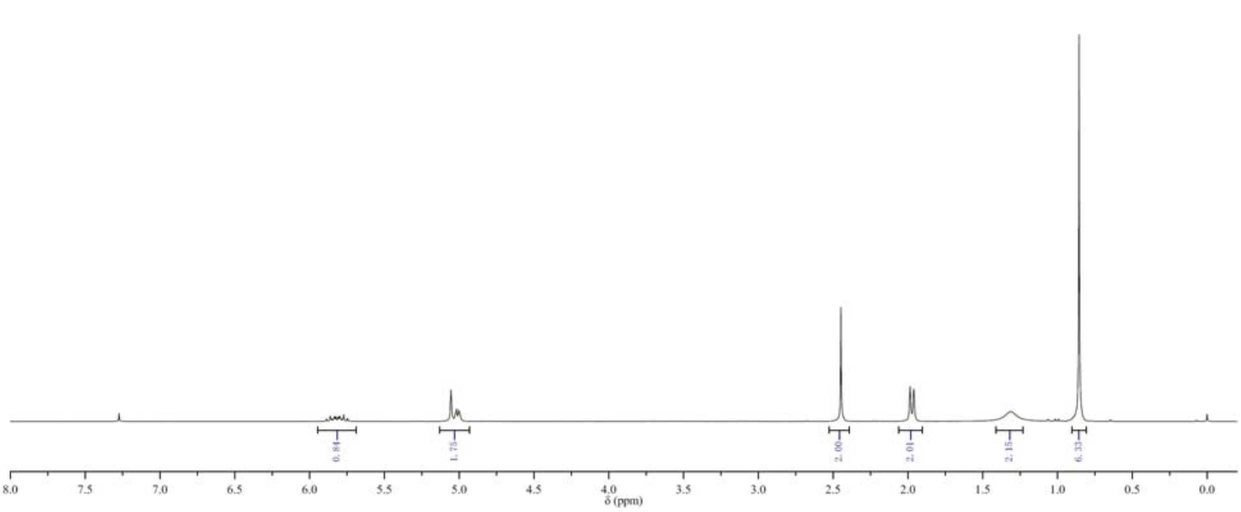

7.31 7.31 7.30 7.29 7.28 7.26 7.26 7.24 7.21 7.21 7.19 7.18 7.17 7.16
5.83 5.81 5.80 5.79 5.77 5.75 5.74 5.73 5.72 5.70
4.99 4.98 4.98 4.97 4.92 4.92 4.91 4.89 4.88 4.88
3.32
2.22 2.20 2.19 2.18 2.16
1.79 1.77 1.76 1.75 1.73 1.71
0.92
-0.00
^{1}H NMR
300 MHz,$CDCl_3$
Ph
NH$_2$
Ph
5c
4.91
0.42
0.92
1.00
1.01
1.02
1.07
δ (ppm)


146.31
138.82
128.28 128.10 126.06
114.39
77.54 77.12 76.69
51.77
49.10
35.74
28.62
^{13}C NMR
75 MHz,$CDCl_3$
Ph
NH$_2$
Ph
5c
δ (ppm)

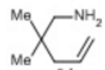
^{1}H NMR
300 MHz, $CDCl_3$



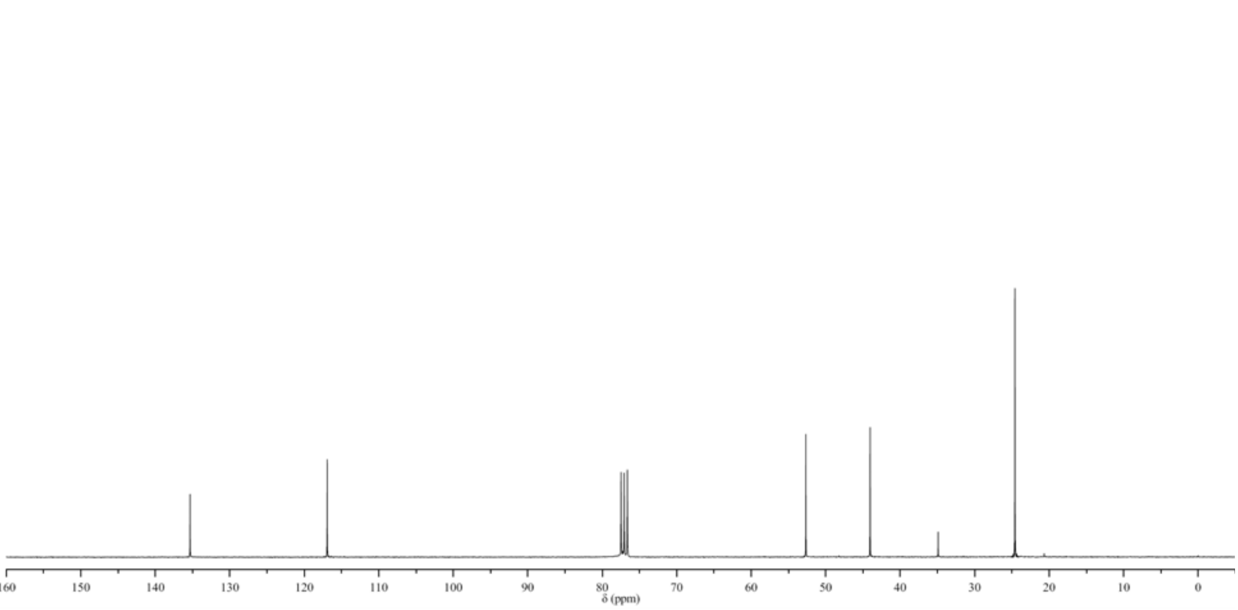
5d

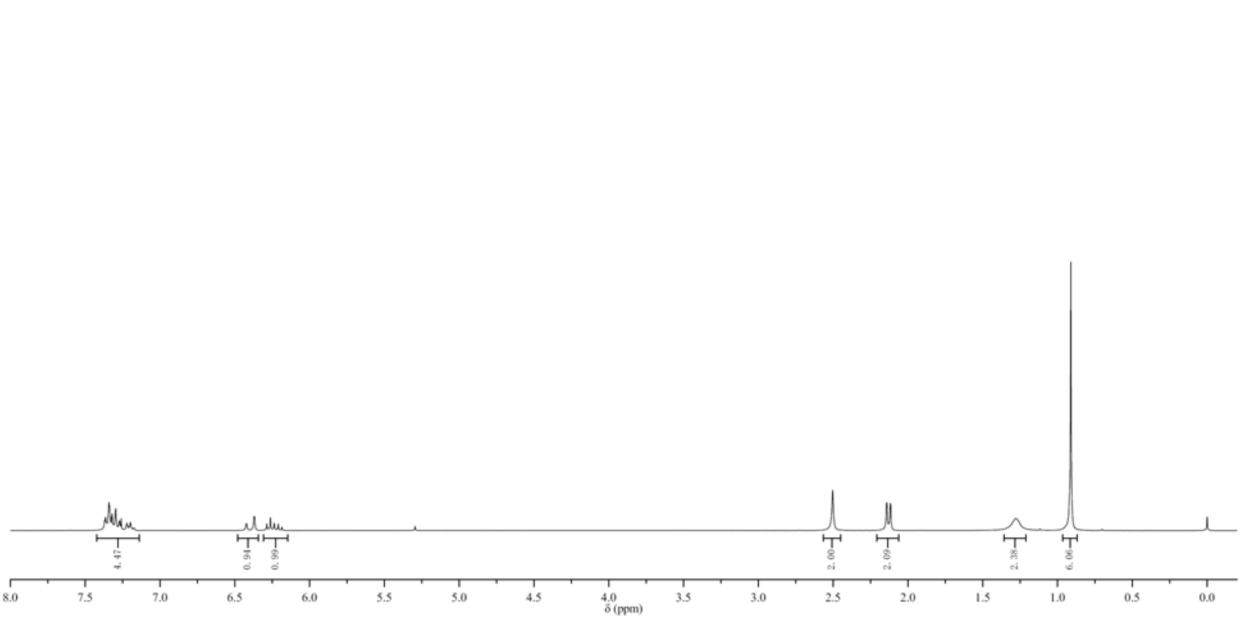


^{13}C NMR
75 MHz, $CDCl_3$

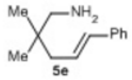


5d

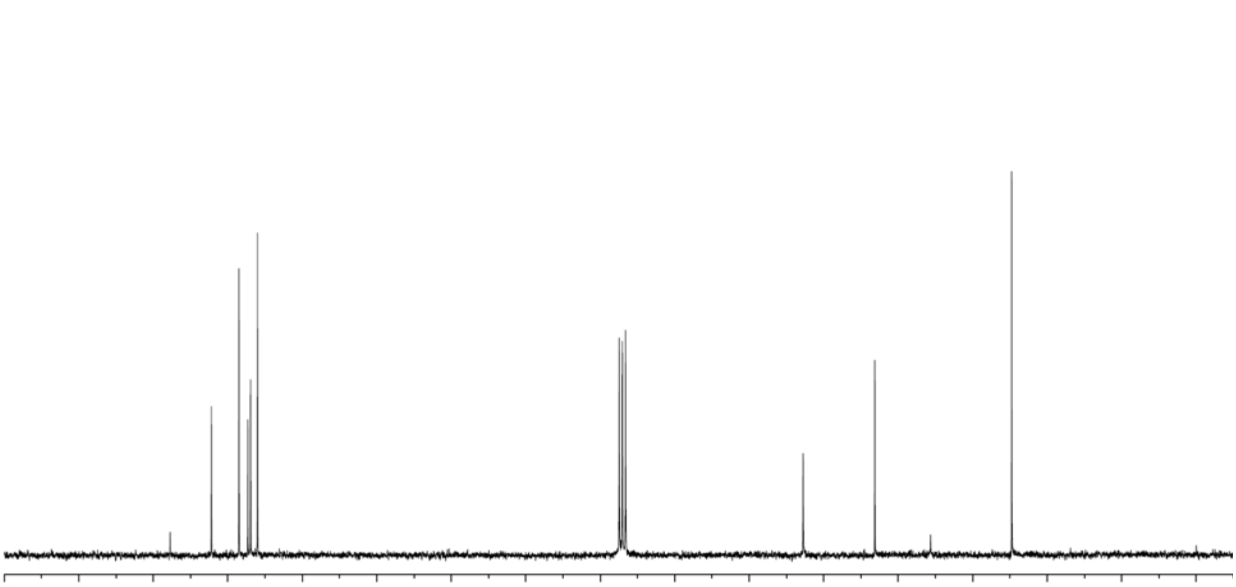




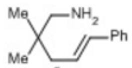
^{1}H NMR
300 MHz, $CDCl_3$



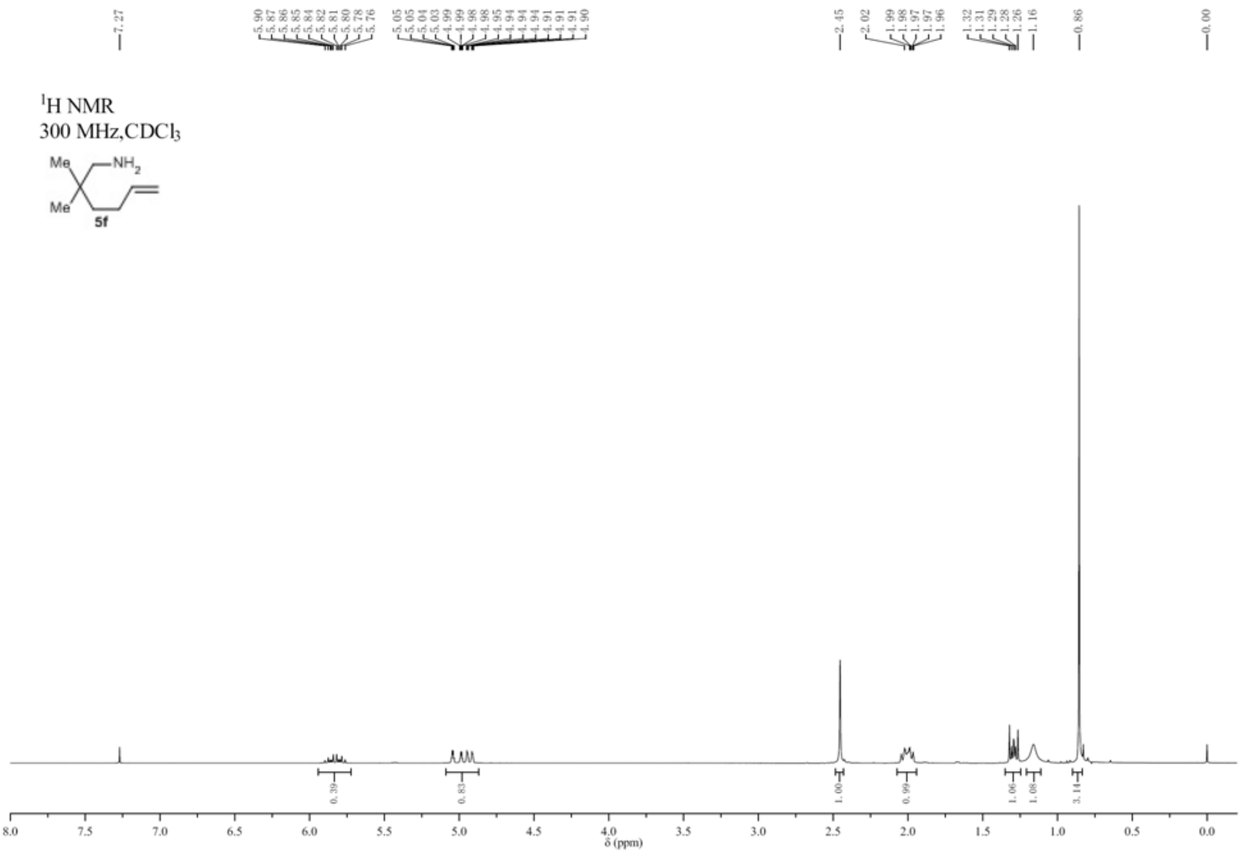
5e

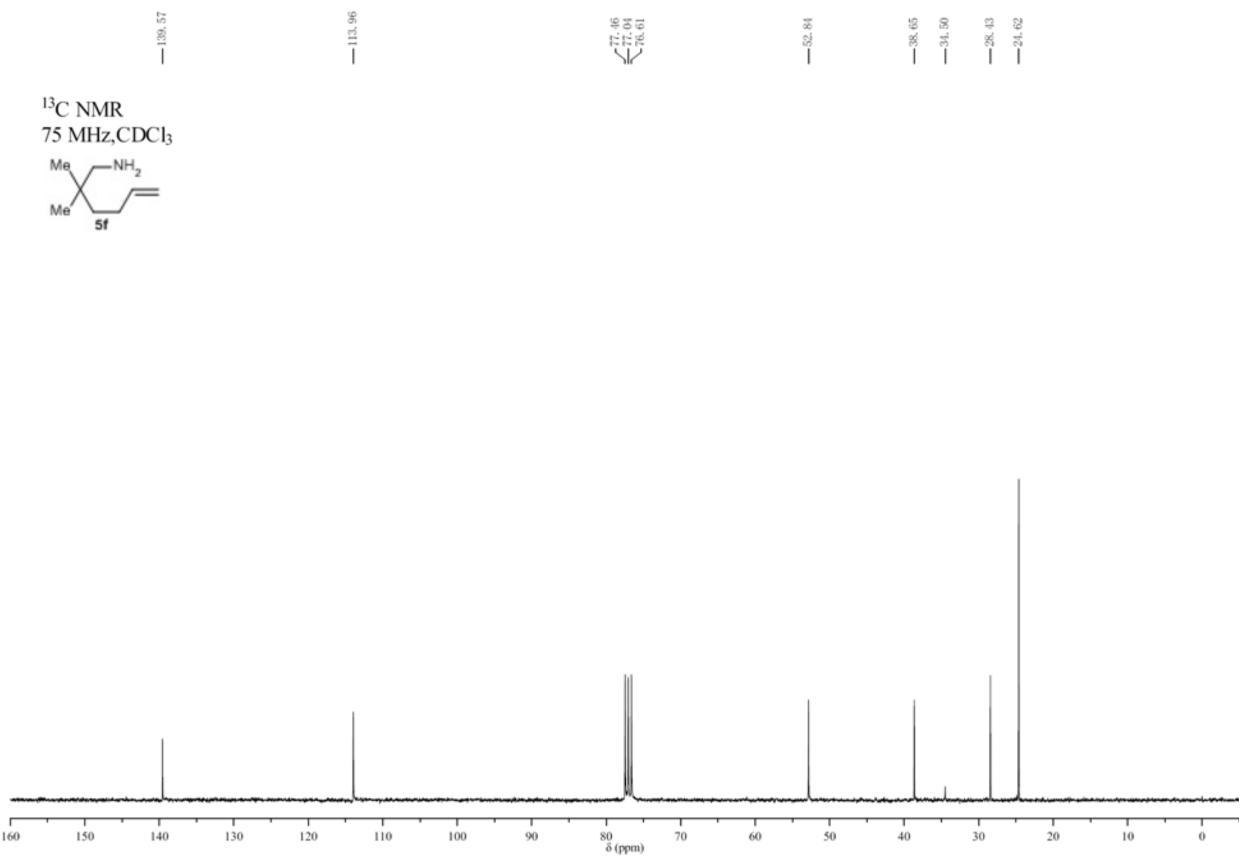


^{13}C NMR
75 MHz, $CDCl_3$



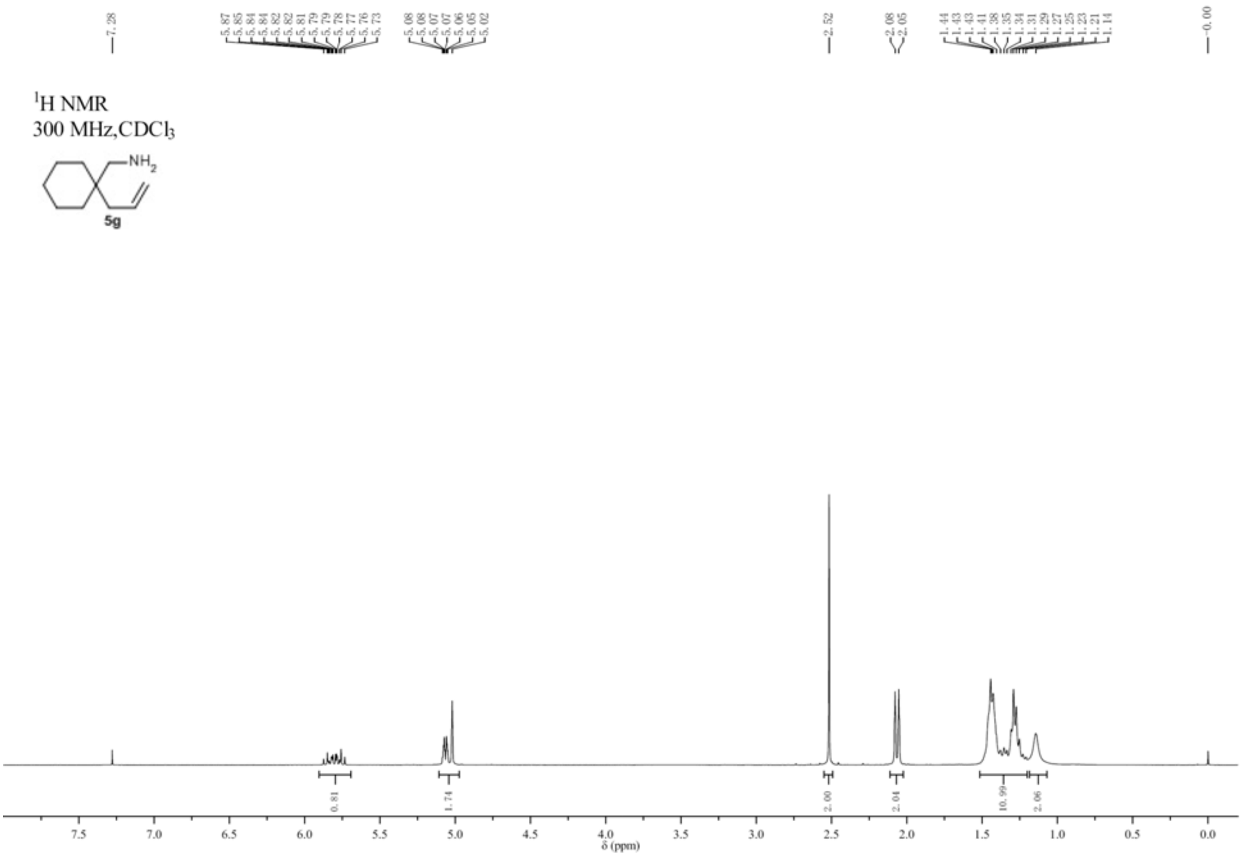
5e


7.27
5.90 5.87 5.86 5.85 5.84 5.82 5.81 5.80 5.78 5.76
5.05 5.05 5.04 5.03 4.99 4.99 4.98 4.98 4.95 4.94 4.94 4.94 4.91 4.91 4.91 4.90
2.45
2.02 1.99 1.98 1.97 1.97 1.96
1.32 1.31 1.29 1.28 1.26 1.16
0.86
0.00
^{1}H NMR
300 MHz,CDCl$_3$
Me
NH$_2$
Me
5f
0.39
0.83
1.00
0.99
1.06
1.08
3.14
δ (ppm)


139.57
113.96
77.46 77.04 76.61
52.84
38.65
34.50
28.43
24.62
^{13}C NMR
75 MHz,CDCl$_3$
Me
NH$_2$
Me
5f
δ (ppm)

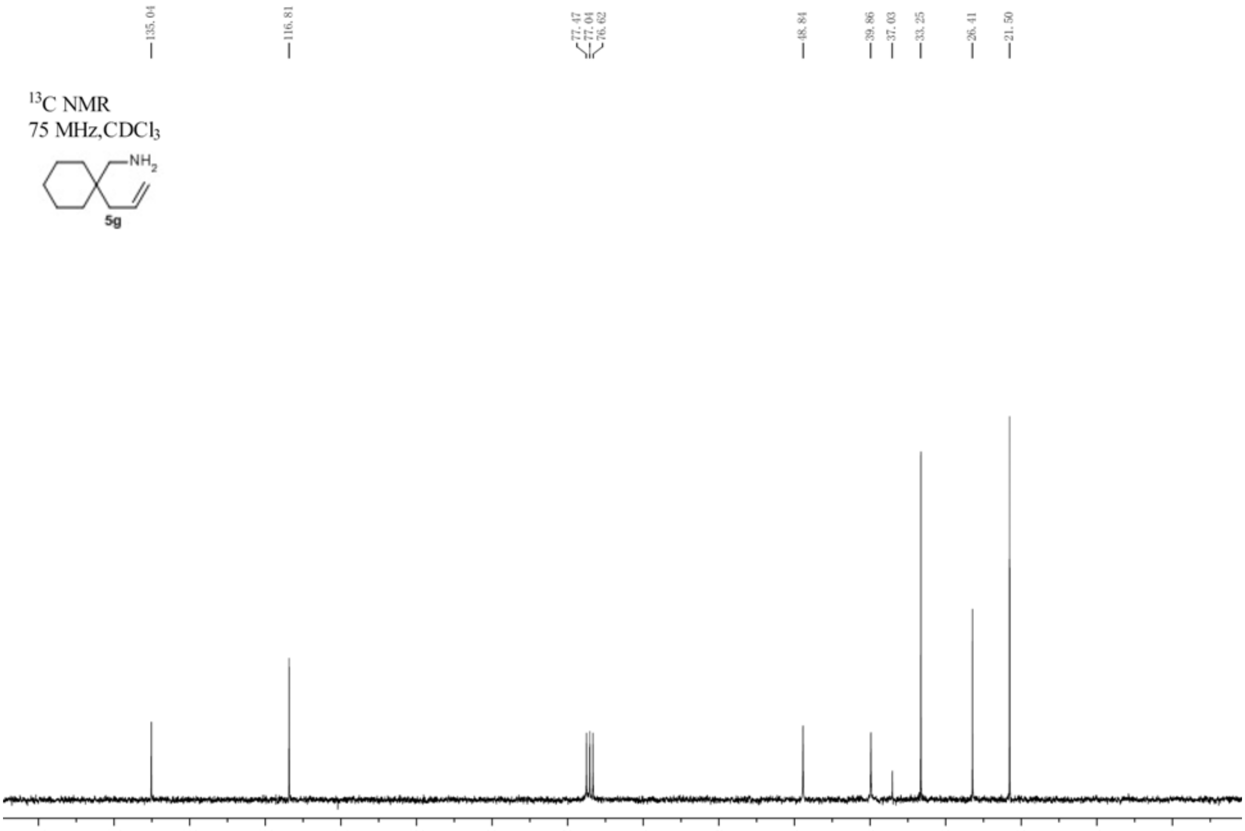
^{1}H NMR
300 MHz, $CDCl_3$

5g



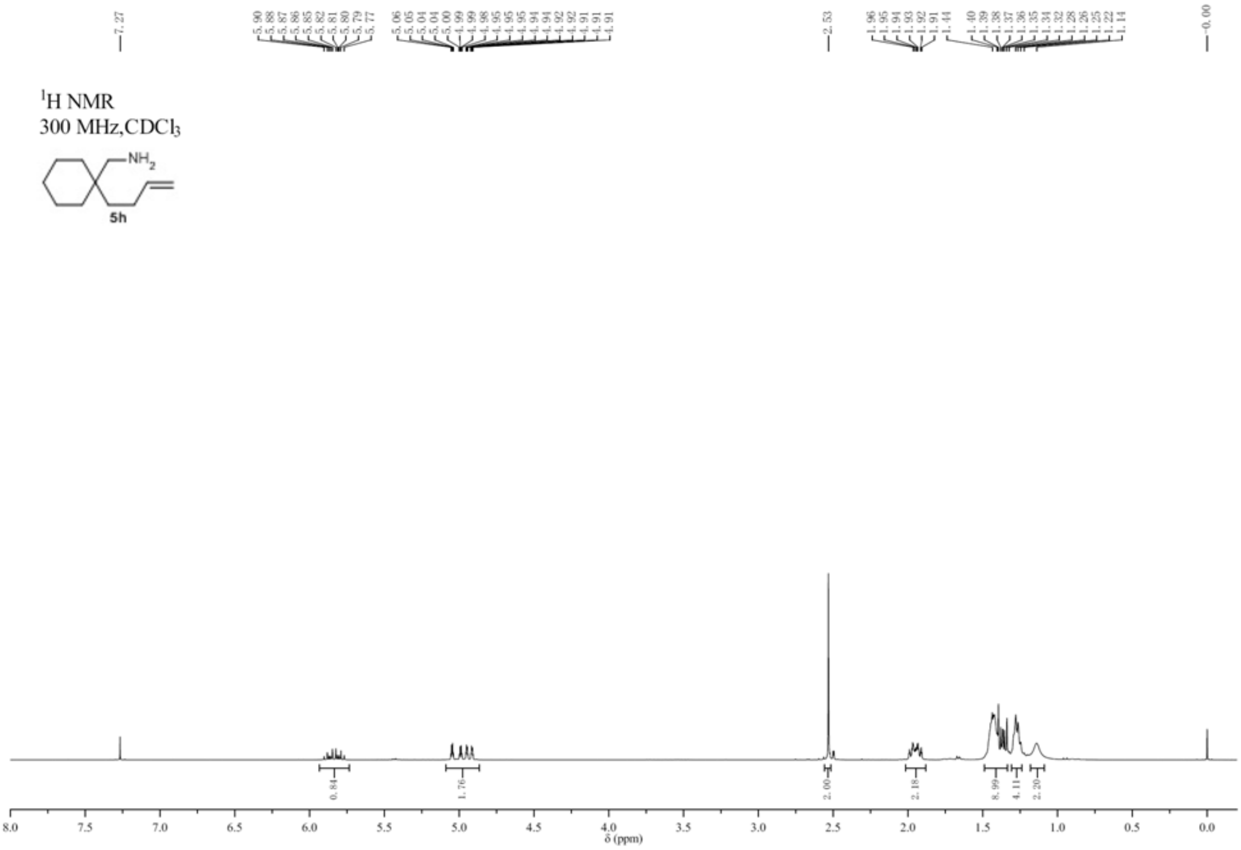
^{13}C NMR
75 MHz, $CDCl_3$

5g



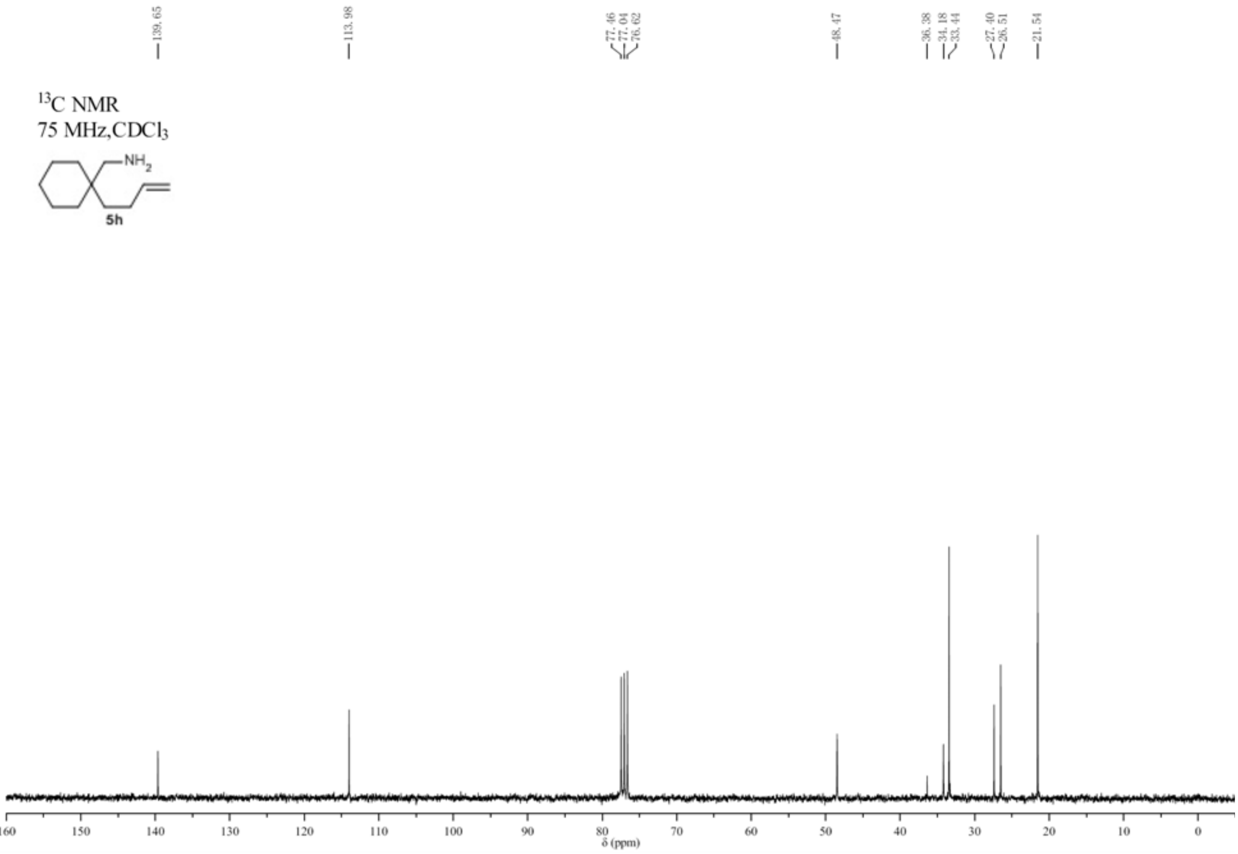
^{1}H NMR
300 MHz, $CDCl_3$

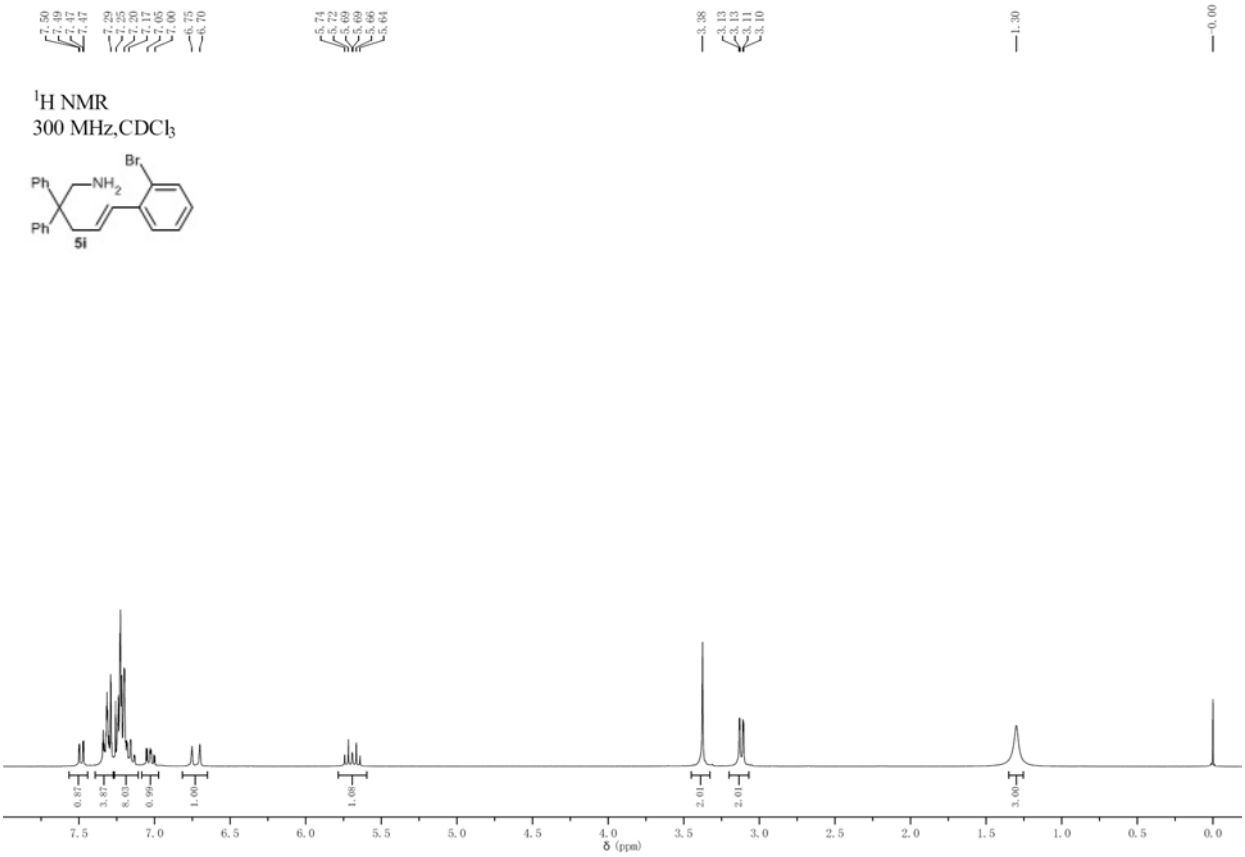
5h

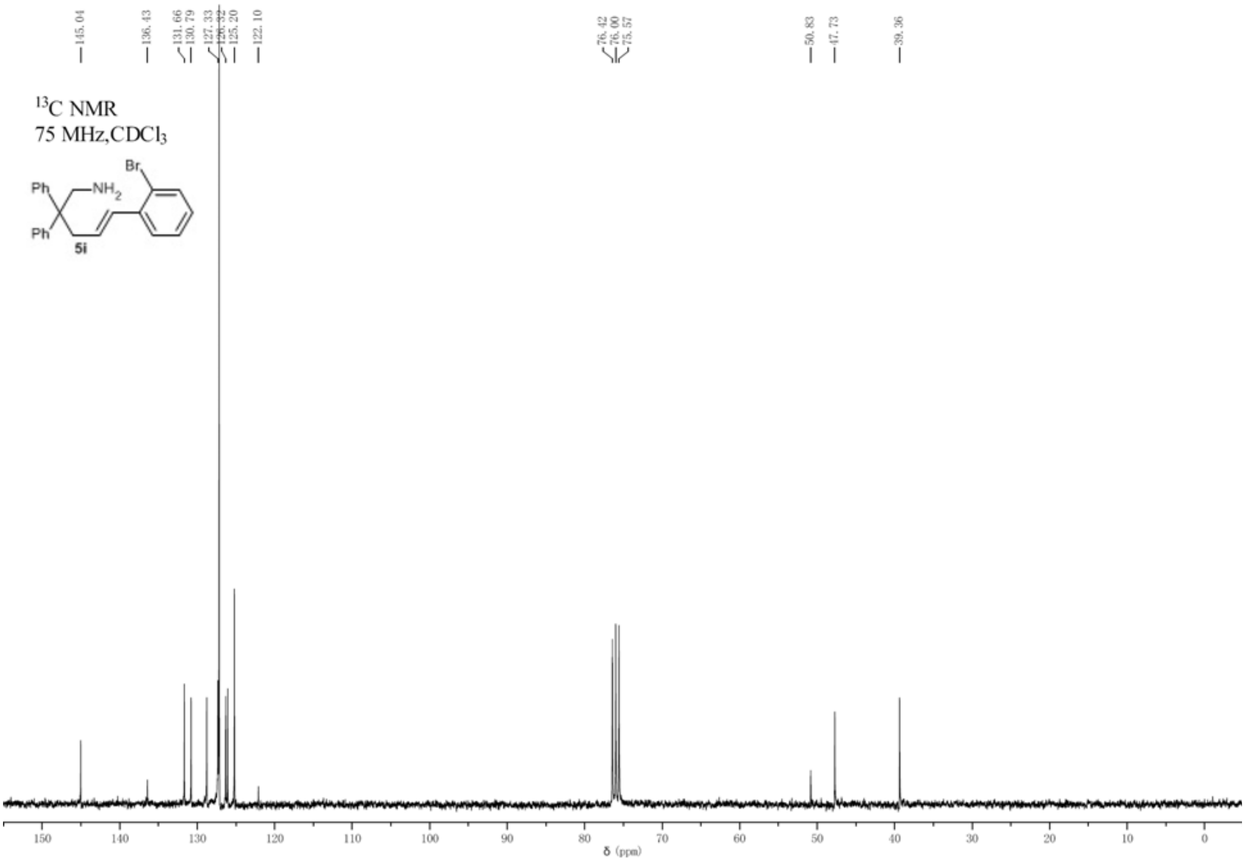


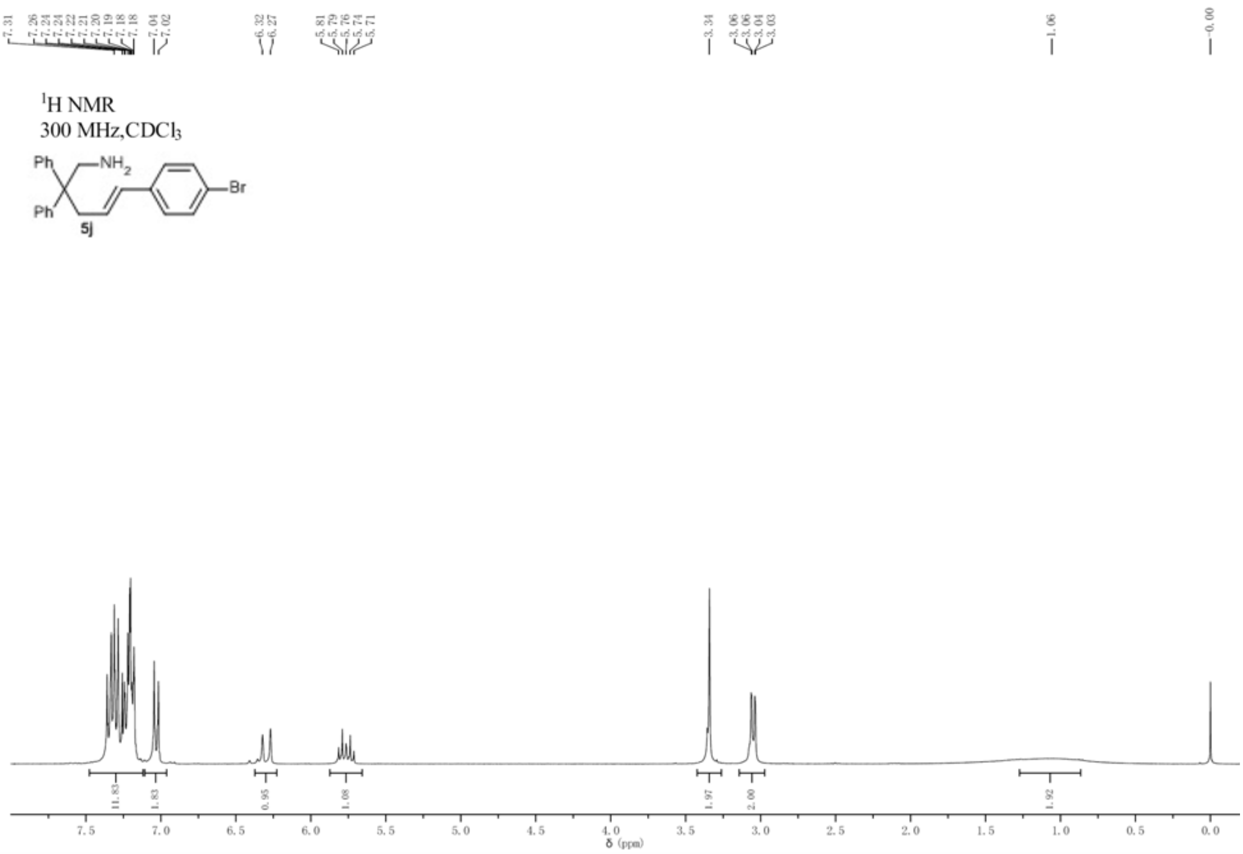
^{13}C NMR
75 MHz, $CDCl_3$

5h

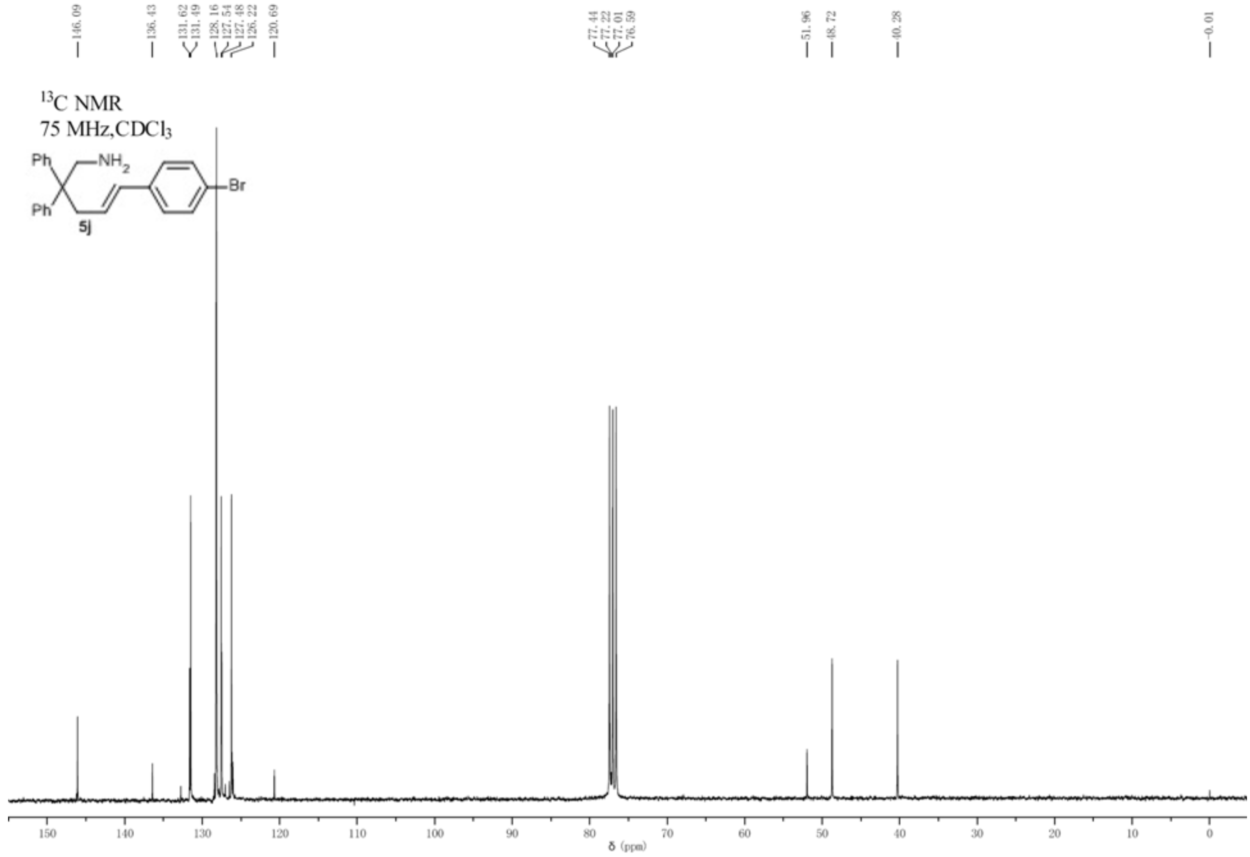



7.50
7.49
7.47
7.47
7.29
7.25
7.20
7.17
7.05
7.00
6.75
6.70
5.74
5.72
5.69
5.69
5.66
5.64
3.38
3.13
3.13
3.11
3.10
1.30
0.00
1H NMR
300 MHz,CDCl3
Br
Ph
NH2
Ph
5i
0.87
3.87
8.03
0.99
1.00
1.08
2.01
2.01
3.00
δ (ppm)

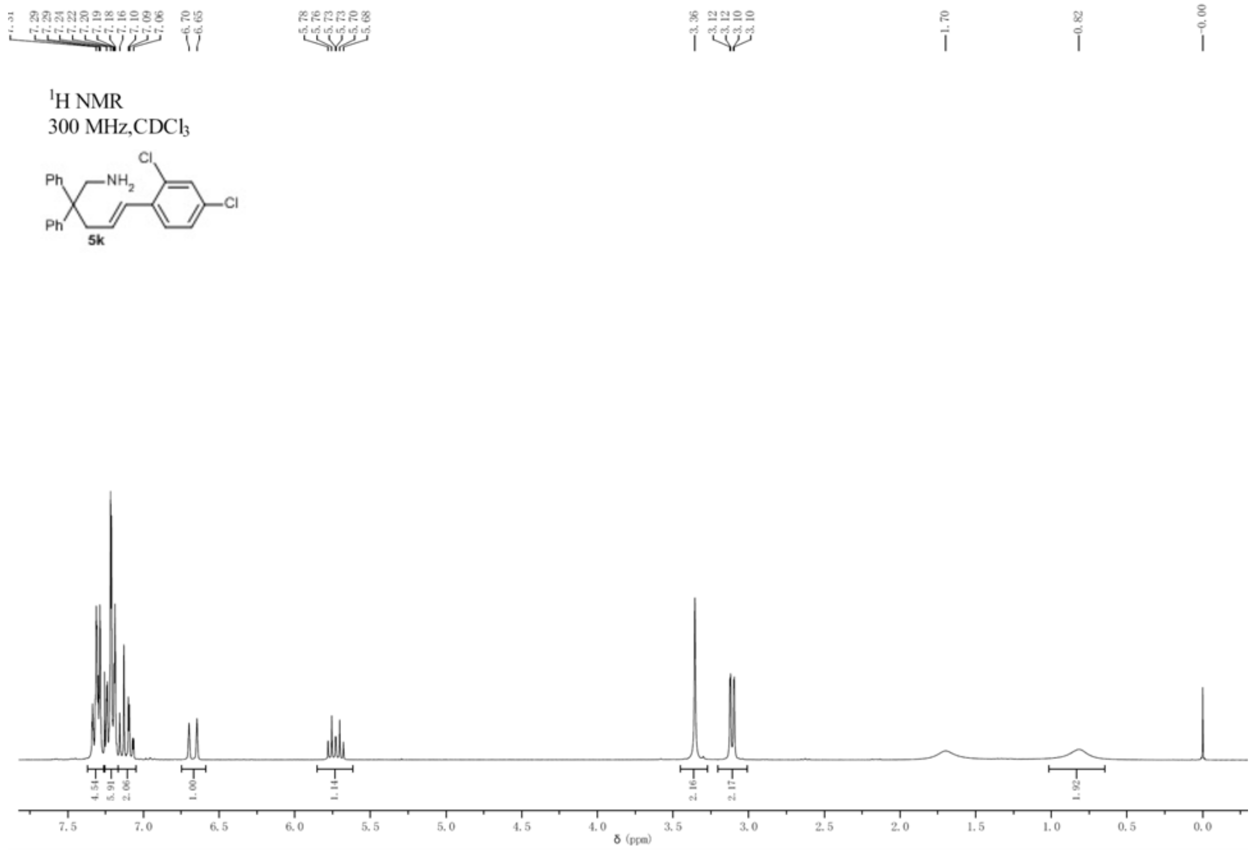

145.04
136.43
131.66
130.79
127.33
126.32
125.20
122.10
76.42
76.00
75.57
50.83
47.73
39.36
13C NMR
75 MHz,CDCl3
Br
Ph
NH2
Ph
5i
δ (ppm)



^{1}H NMR
300 MHz,CDCl$_3$
Ph
NH$_2$
Br
Ph
5j
δ (ppm)

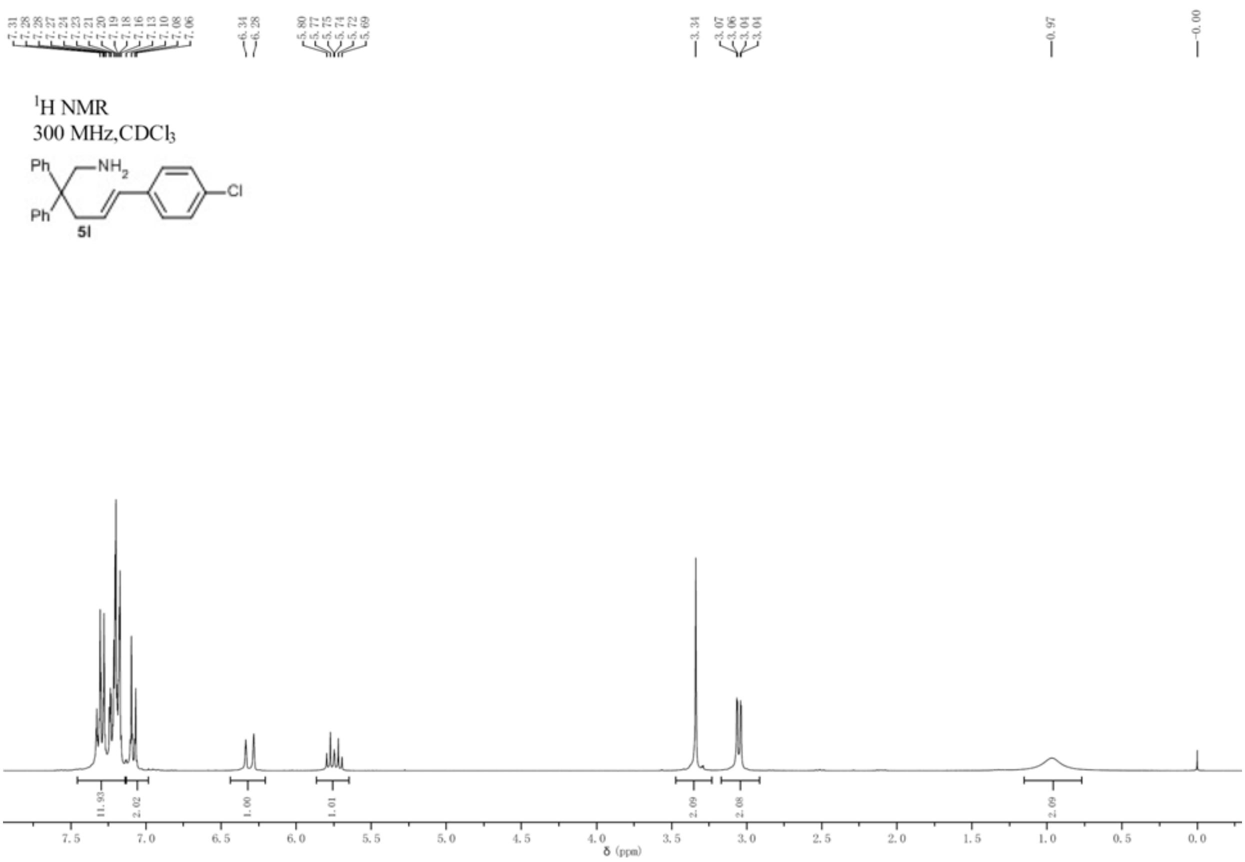


^{13}C NMR
75 MHz,CDCl$_3$
Ph
NH$_2$
Br
Ph
5j
δ (ppm)



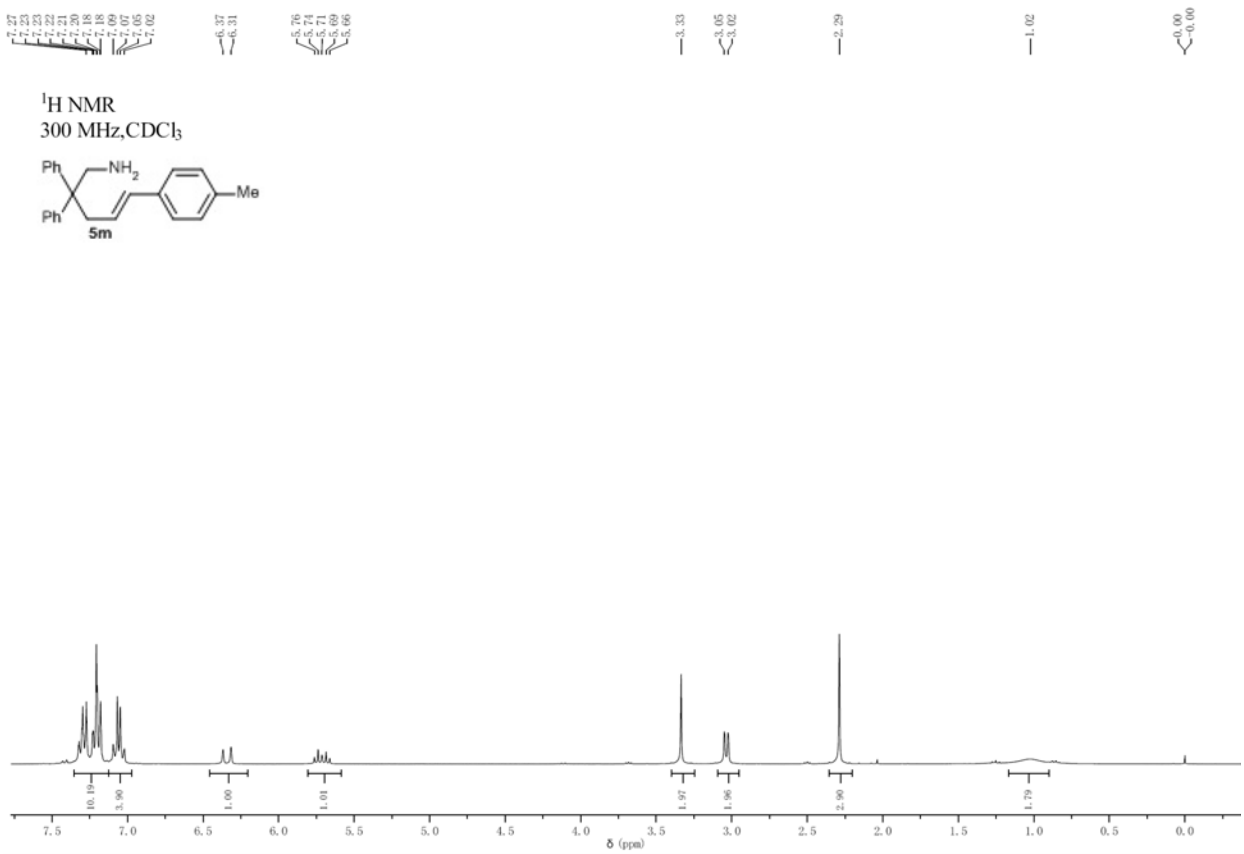
^{1}H NMR
300 MHz,CDCl$_3$
Ph
NH$_2$
Cl
Cl
Ph
5k
δ (ppm)

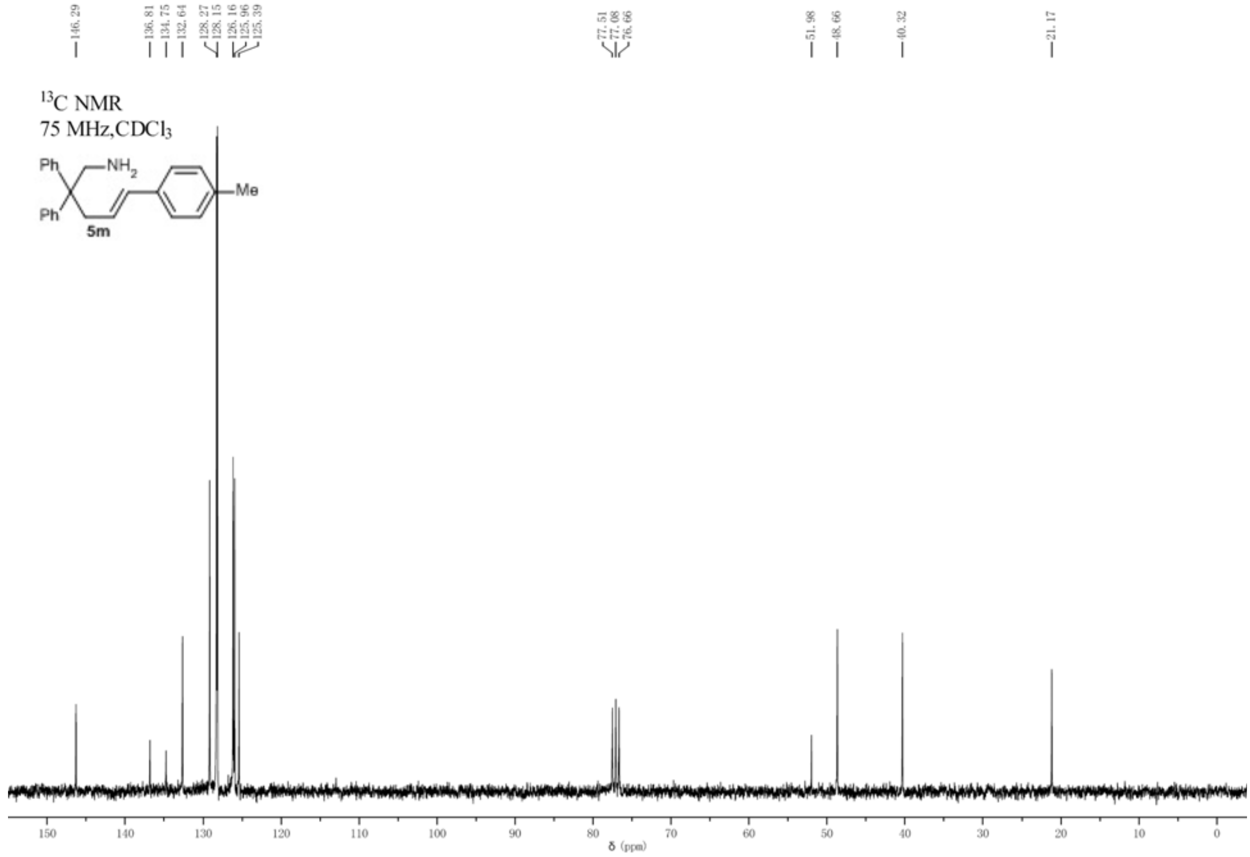
^{13}C NMR
75 MHz,CDCl$_3$
Ph
NH$_2$
Cl
Cl
Ph
5k
δ (ppm)



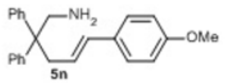
^{1}H NMR
300 MHz, $CDCl_3$
5l

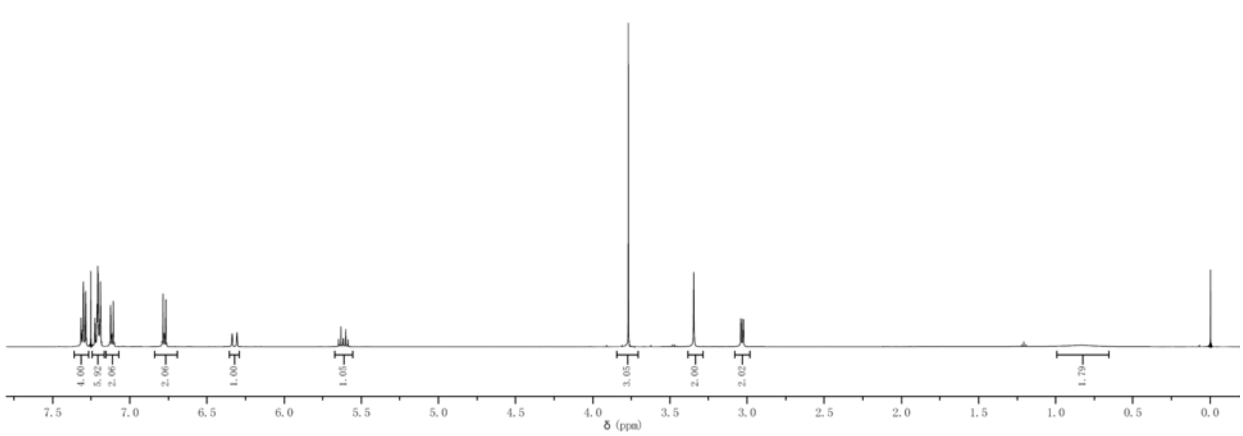
^{13}C NMR
75 MHz, $CDCl_3$
5l


7.27 7.23 7.23 7.22 7.21 7.20 7.18 7.09 7.07 7.05 7.02
6.37 6.31
5.76 5.74 5.71 5.69 5.66
3.33
3.05 3.02
2.29
1.02
0.00 -0.00
^{1}H NMR
300 MHz,CDCl$_3$
Ph
NH$_2$
Me
Ph
5m
10.19 3.90 1.00 1.01 1.97 1.96 2.90 1.79
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)

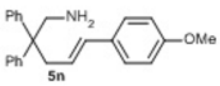

146.29
136.81 134.75 132.64 128.27 128.15 126.16 125.96 125.39
77.51 77.08 76.66
51.98
48.66
40.32
21.17
^{13}C NMR
75 MHz,CDCl$_3$
Ph
NH$_2$
Me
Ph
5m
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

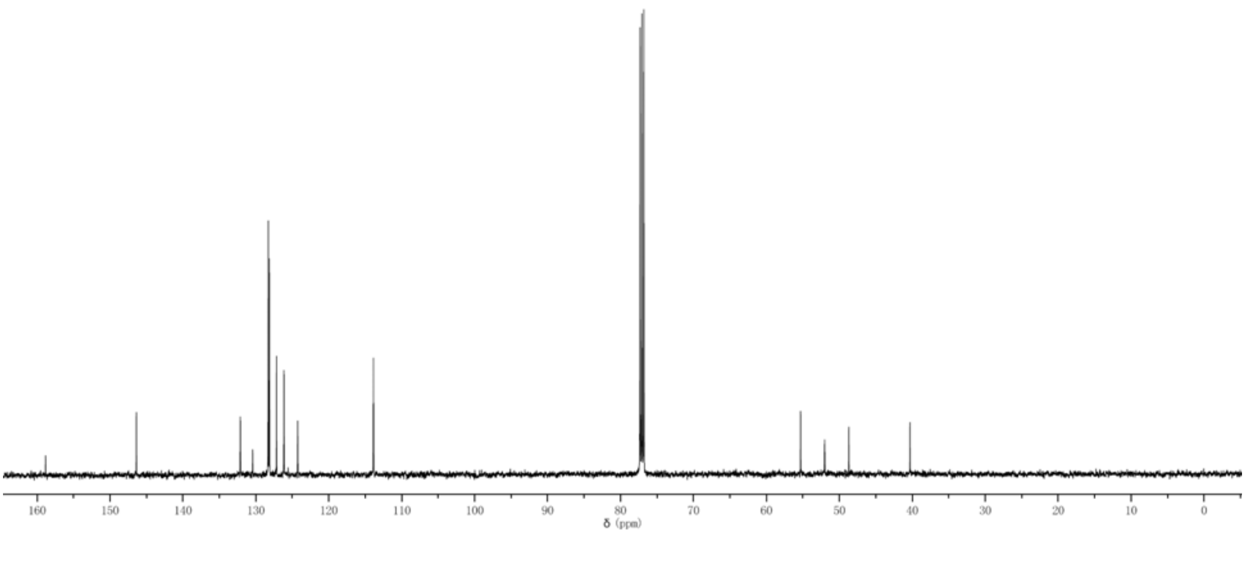
[1]H NMR
500 MHz,CDCl$_3$

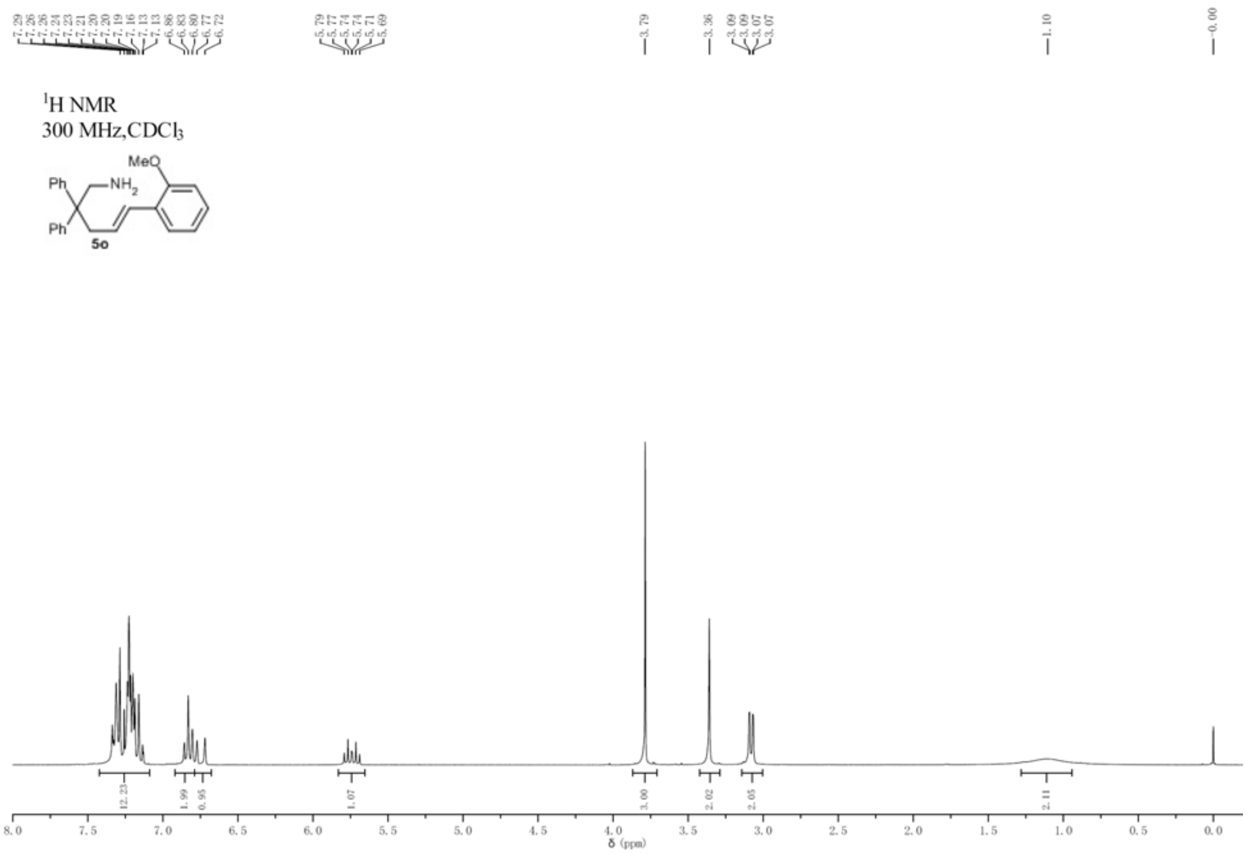




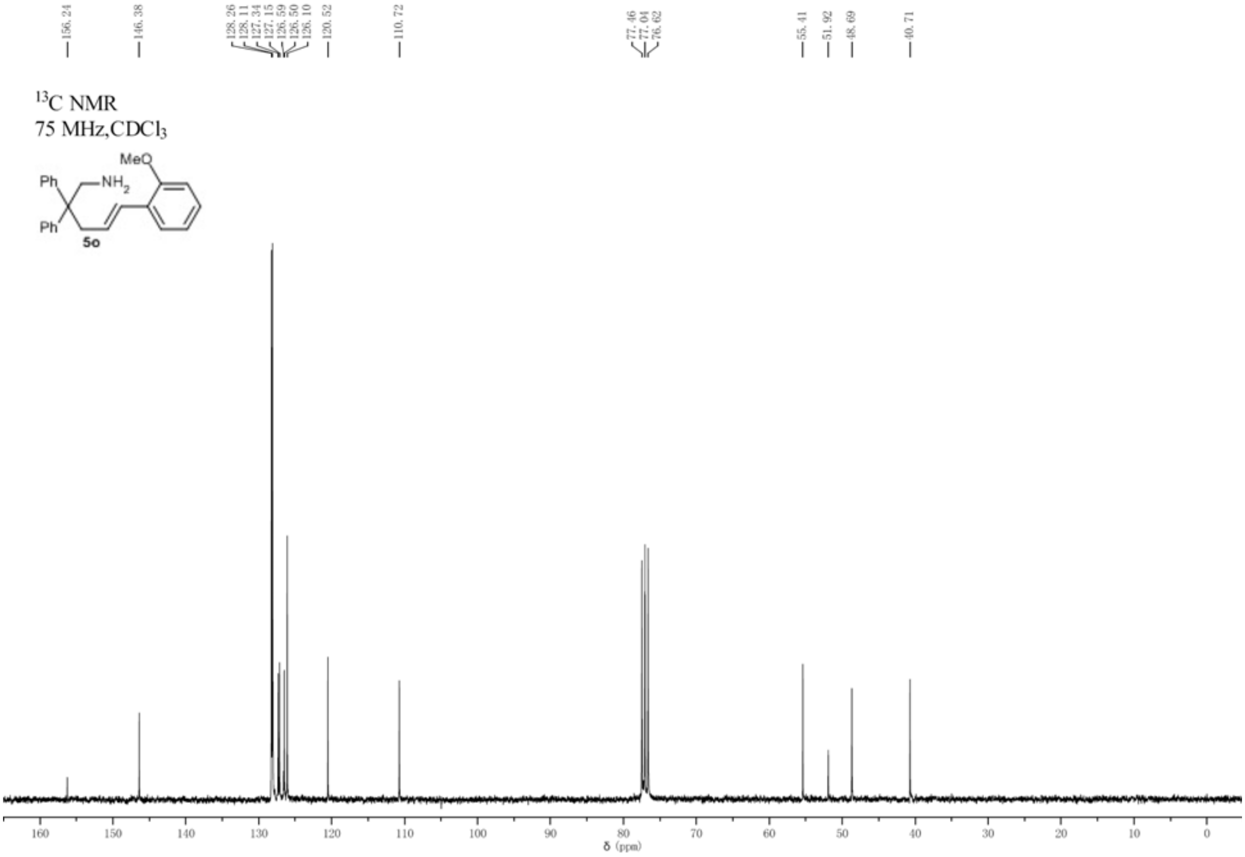
[13]C NMR
125 MHz,CDCl$_3$



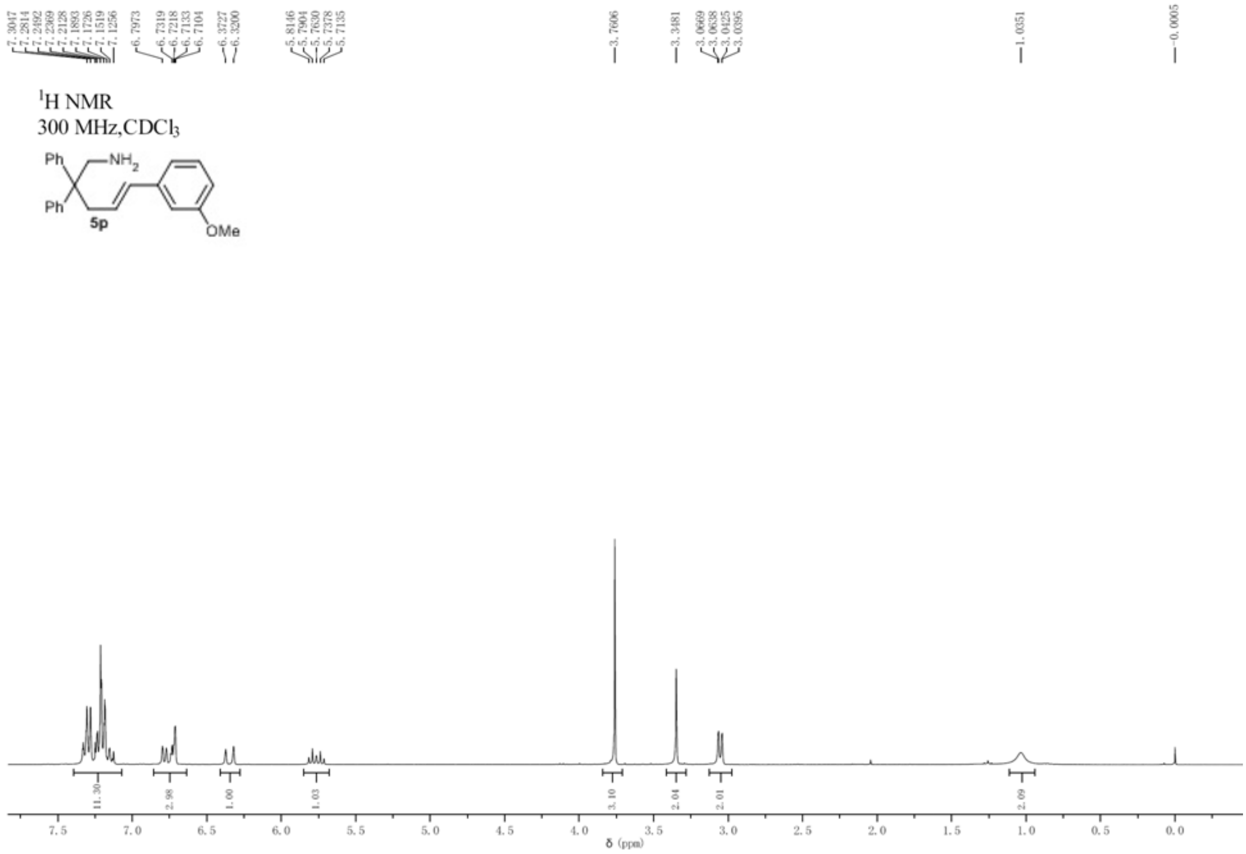


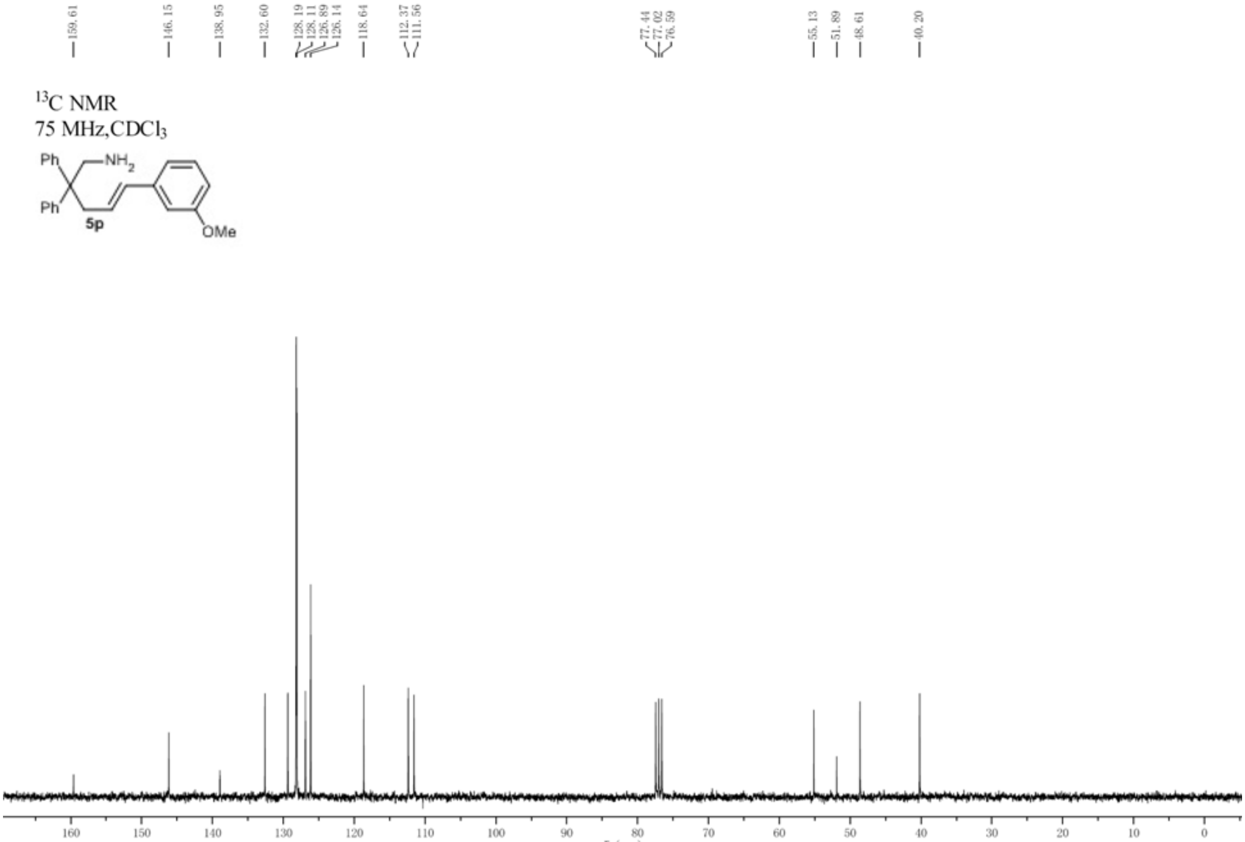


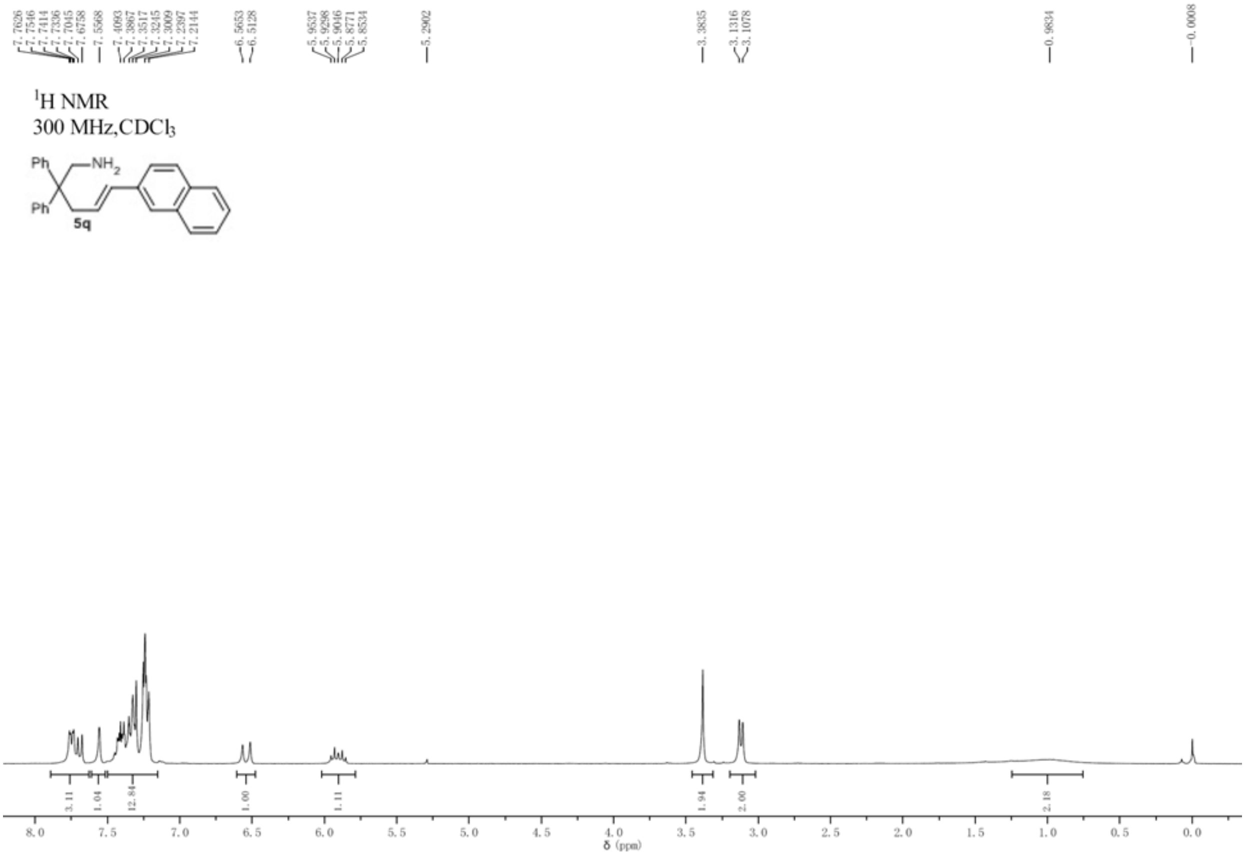
7.29 7.26 7.24 7.23 7.21 7.20 7.20 7.19 7.16 7.13 7.13 6.86 6.83 6.80 6.77 6.72
5.79 5.77 5.74 5.74 5.71 5.69
3.79
3.36 3.09 3.09 3.07 3.07
1.10
0.00
¹H NMR
300 MHz,CDCl₃
MeO
Ph
NH₂
Ph
5o
12.23
1.99
0.95
1.07
3.00
2.02
2.05
2.11
δ (ppm)

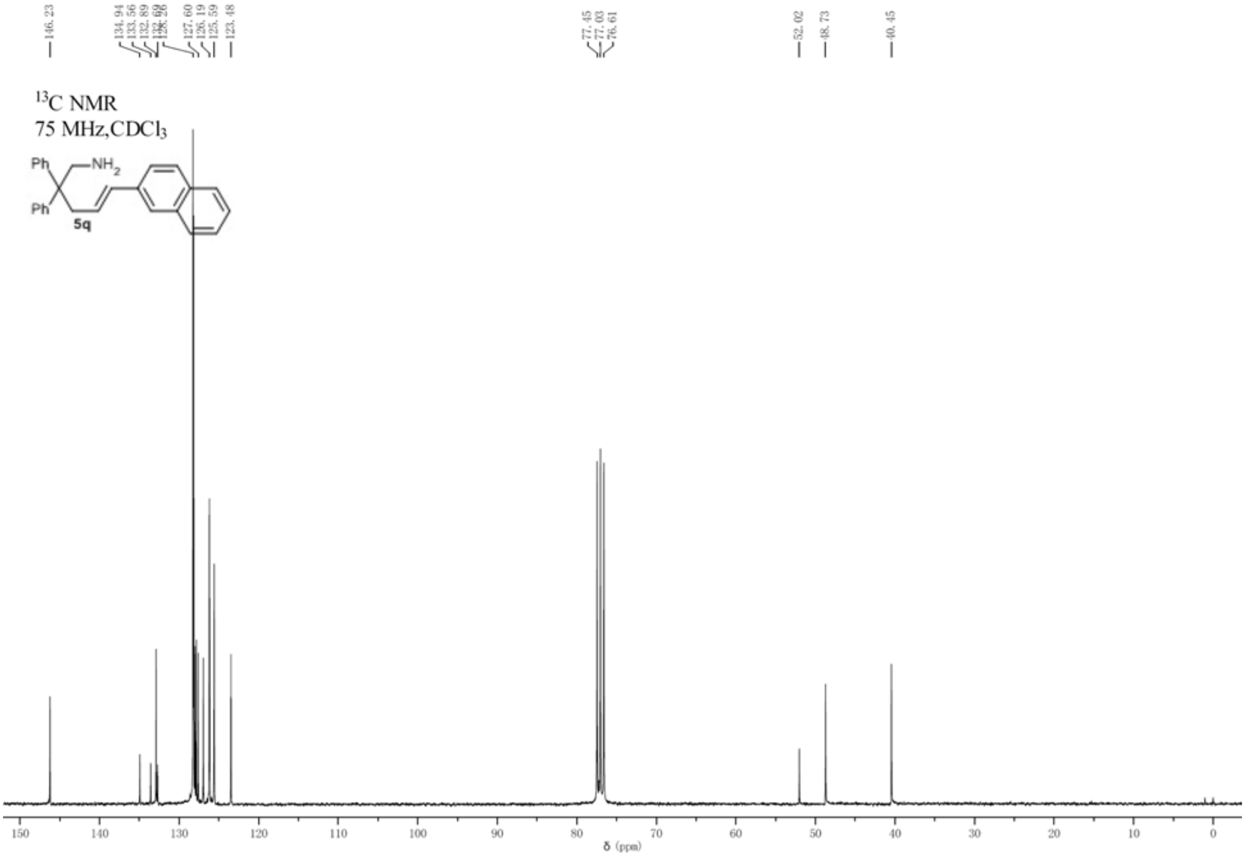


156.24
146.38
128.26 128.11 127.34 127.15 126.59 126.50 126.10
120.52
110.72
77.46 77.04 76.62
55.41
51.92
48.69
40.71
¹³C NMR
75 MHz,CDCl₃
MeO
Ph
NH₂
Ph
5o
δ (ppm)

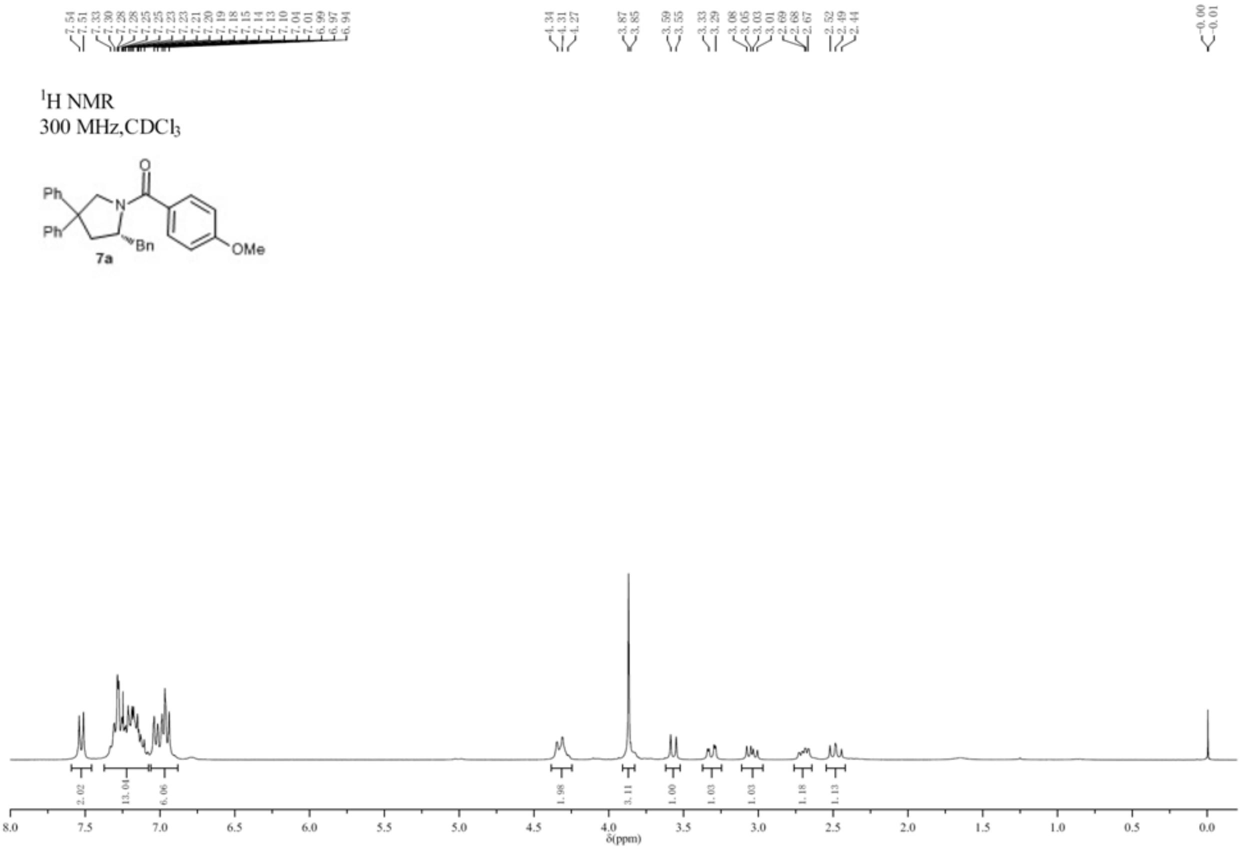

7.3047
7.2814
7.2492
7.2369
7.2128
7.1893
7.1726
7.1519
7.1256
6.7973
6.7319
6.7218
6.7133
6.7104
6.3727
6.3200
5.8146
5.7904
5.7630
5.7378
5.7135
3.7606
3.3481
3.0669
3.0638
3.0425
3.0395
1.0351
-0.0005
1H NMR
300 MHz,CDCl3
Ph
Ph
NH2
5p
OMe
11.30
2.98
1.00
1.03
3.10
2.04
2.01
2.09
δ (ppm)

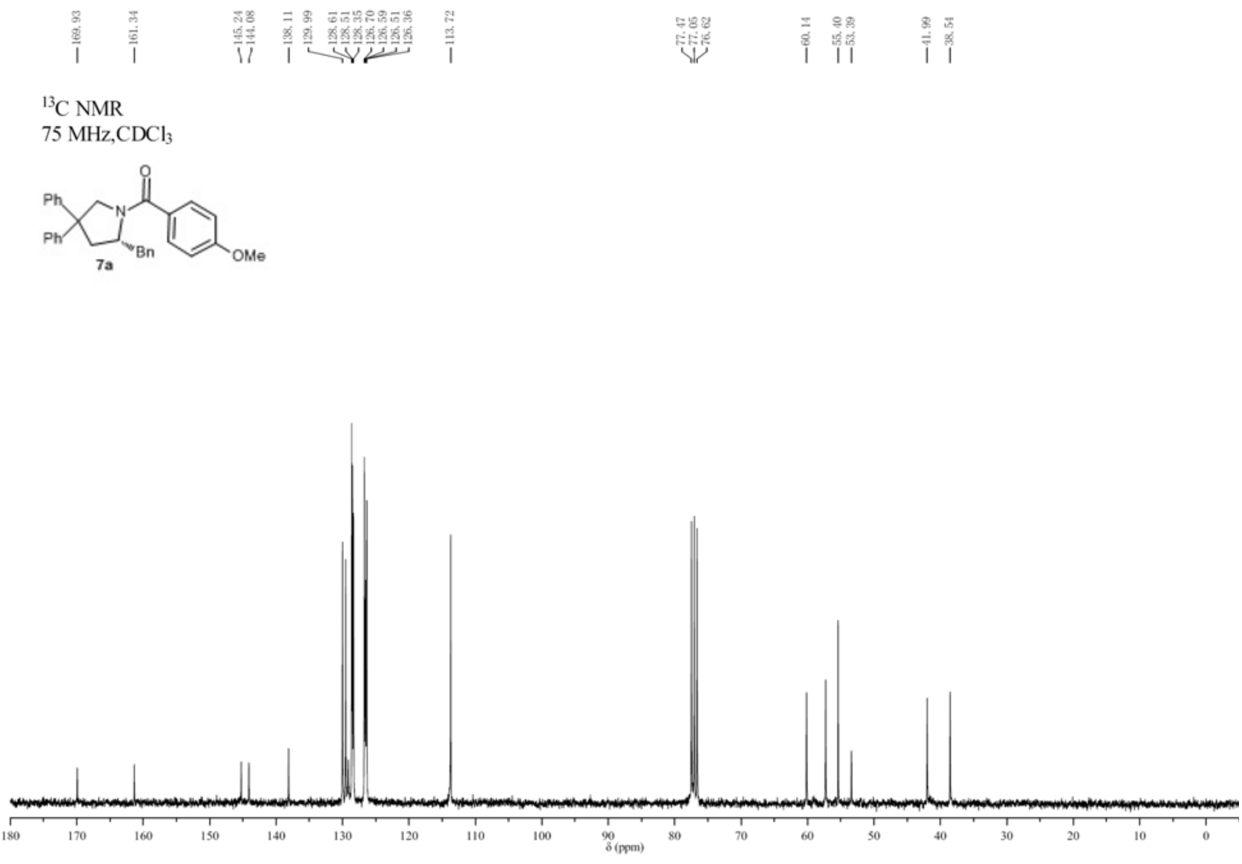

159.61
146.15
138.95
132.60
128.19
128.11
126.89
126.14
118.64
112.37
111.56
77.44
77.02
76.59
55.13
51.89
48.61
40.20
13C NMR
75 MHz,CDCl3
Ph
Ph
NH2
5p
OMe
δ (ppm)


7.7626
7.7546
7.7414
7.7336
7.7045
7.6758
7.5568
7.4093
7.3867
7.3517
7.3245
7.3009
7.2397
7.2144
6.5653
6.5128
5.9537
5.9298
5.9046
5.8771
5.8534
5.2902
3.3835
3.1316
3.1078
0.9834
-0.0008
^{1}H NMR
300 MHz,CDCl$_3$
Ph
NH$_2$
Ph
5q
3.11
1.04
12.84
1.00
1.11
1.94
2.00
2.18
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ (ppm)

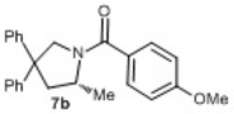

146.23
134.94
133.56
132.89
132.69
128.26
127.60
126.19
125.39
123.48
77.45
77.03
76.61
52.02
48.73
40.45
^{13}C NMR
75 MHz,CDCl$_3$
Ph
NH$_2$
Ph
5q
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

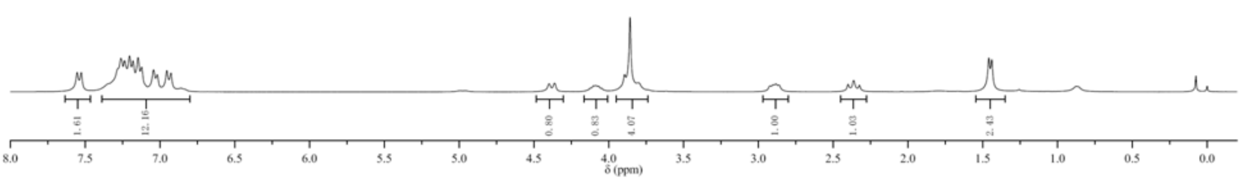
Copies of ^{1}H NMR and ^{13}C NMR for 4-methoxybenzoyl amides



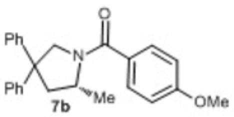


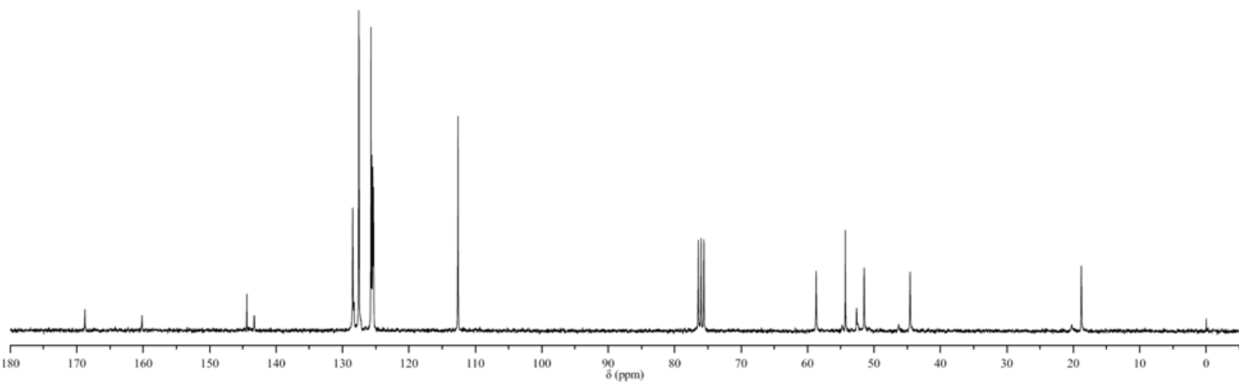
[1]H NMR
300 MHz,$CDCl_3$



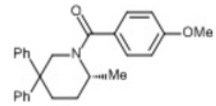


[13]C NMR
75 MHz,$CDCl_3$

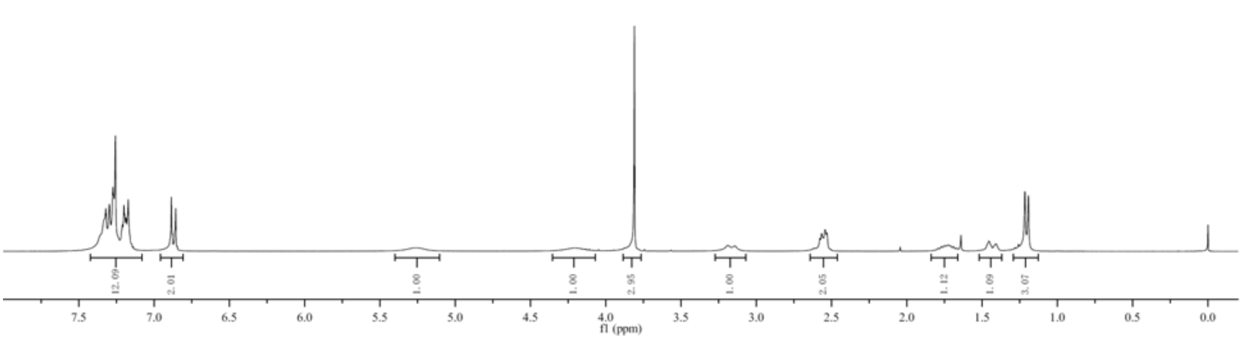




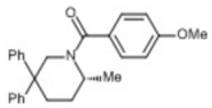
[1]H NMR
300 MHz, $CDCl_3$



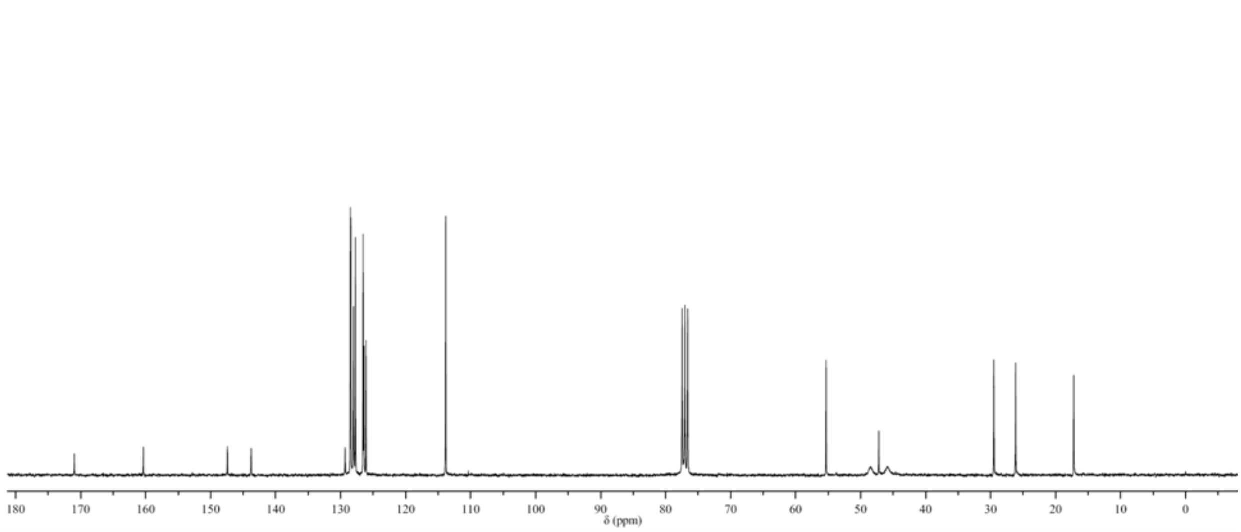
7c

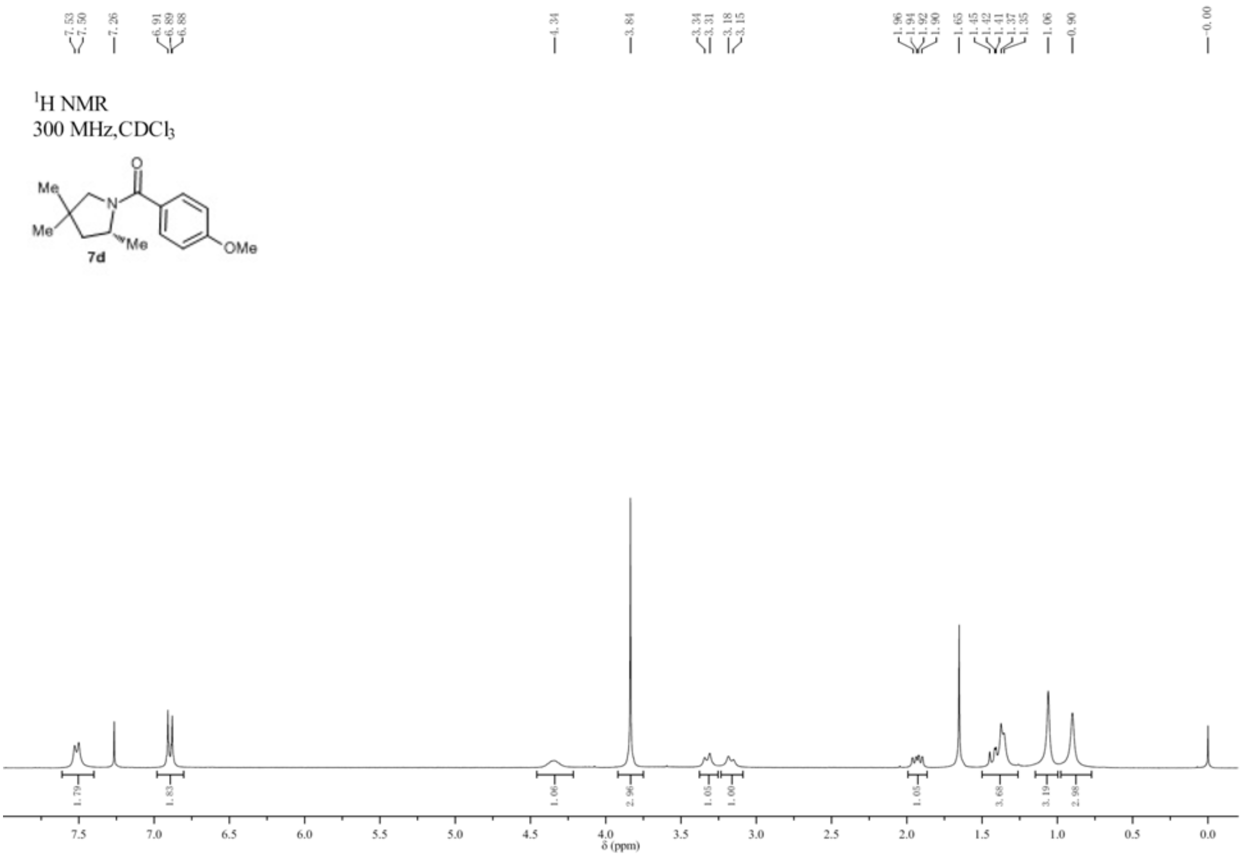


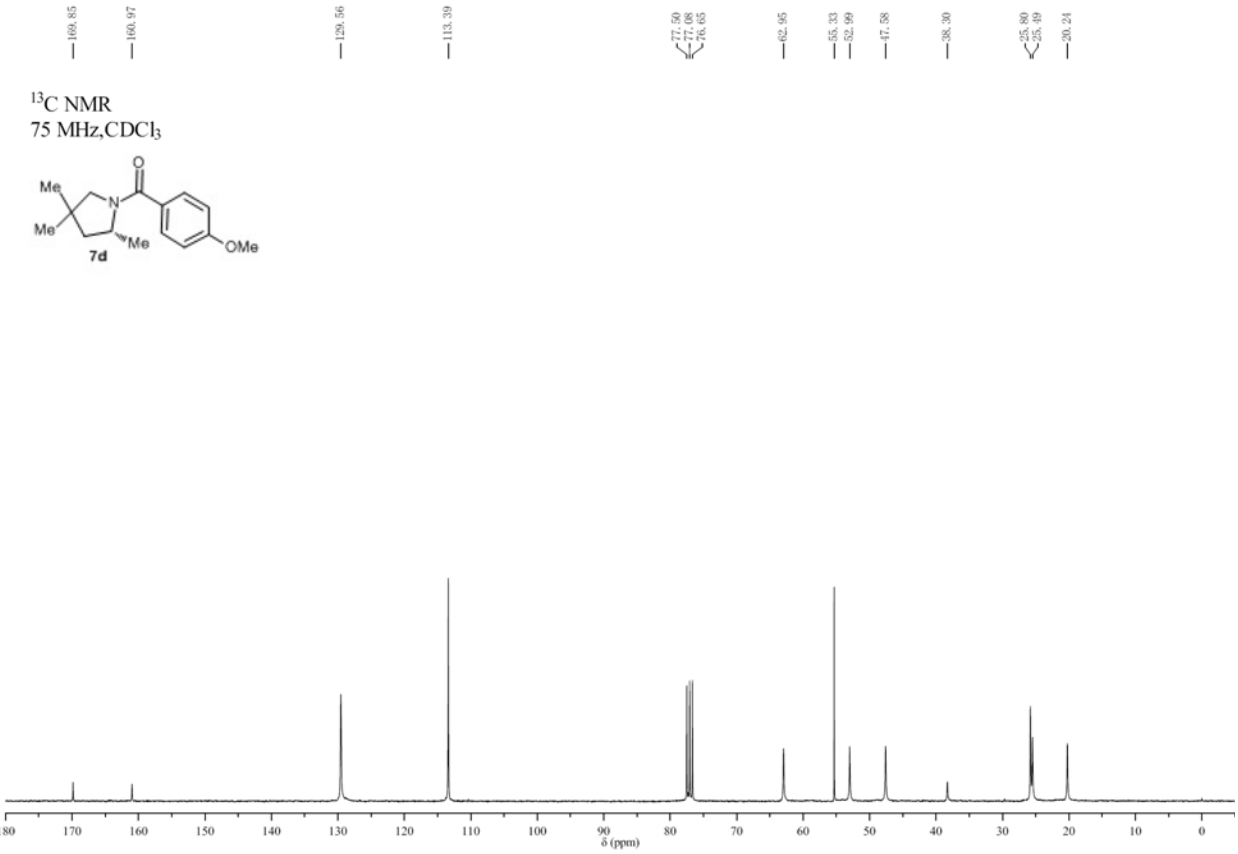
[13]C NMR
75 MHz, $CDCl_3$

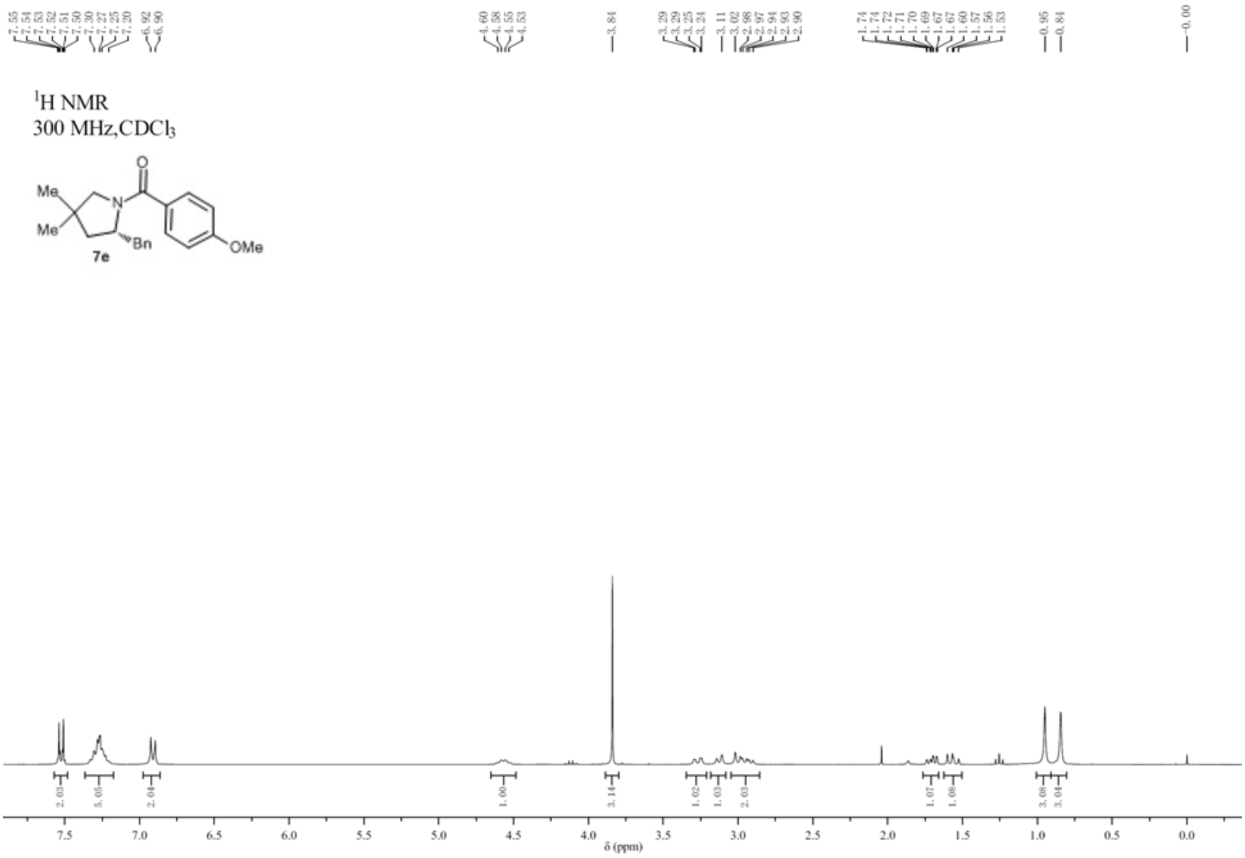


7c

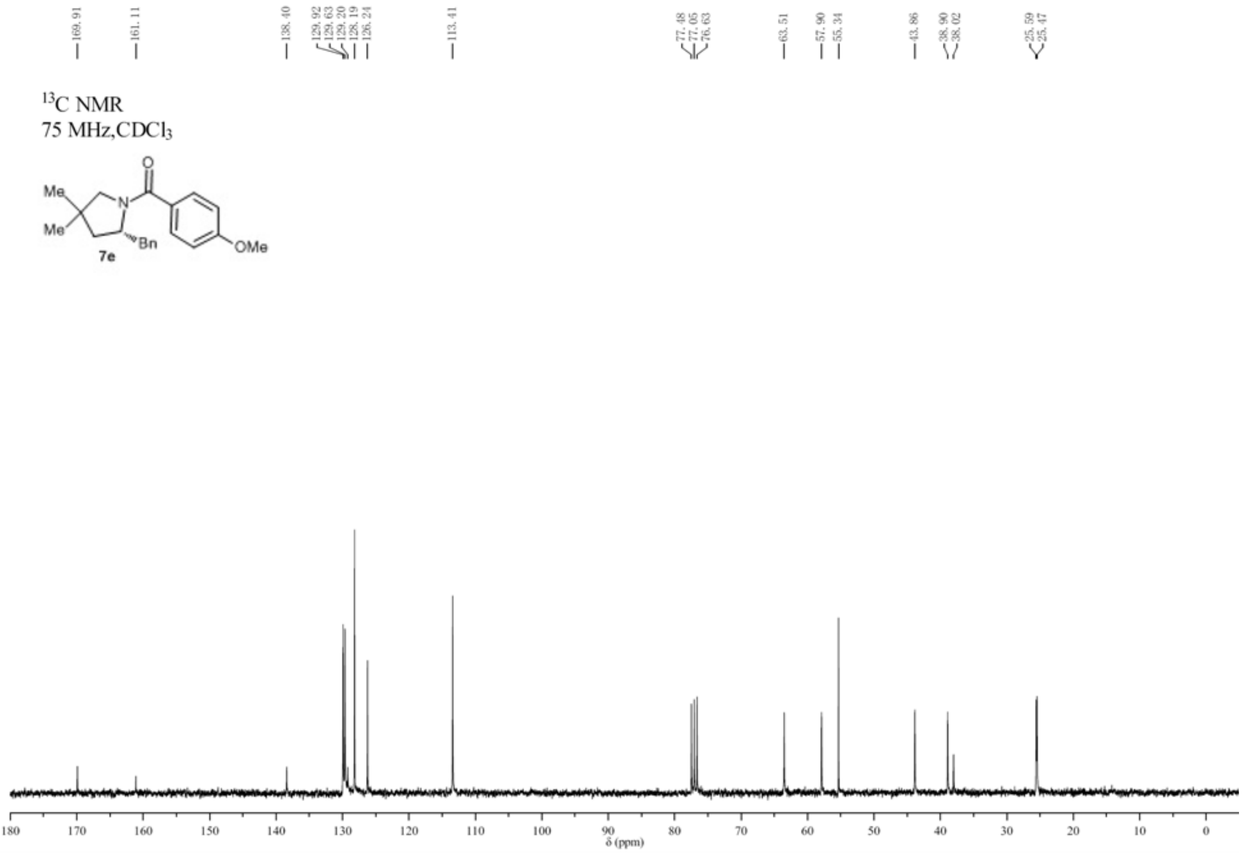



7.53
7.50
7.26
6.91
6.89
6.88
4.34
3.84
3.34
3.31
3.18
3.15
1.96
1.94
1.92
1.90
1.65
1.45
1.42
1.41
1.37
1.35
1.06
0.90
-0.00
1H NMR
300 MHz,CDCl3
O
Me
Me
N
Me
OMe
7d
1.79
1.83
1.06
2.96
1.05
1.00
1.05
3.68
3.19
2.98
δ (ppm)

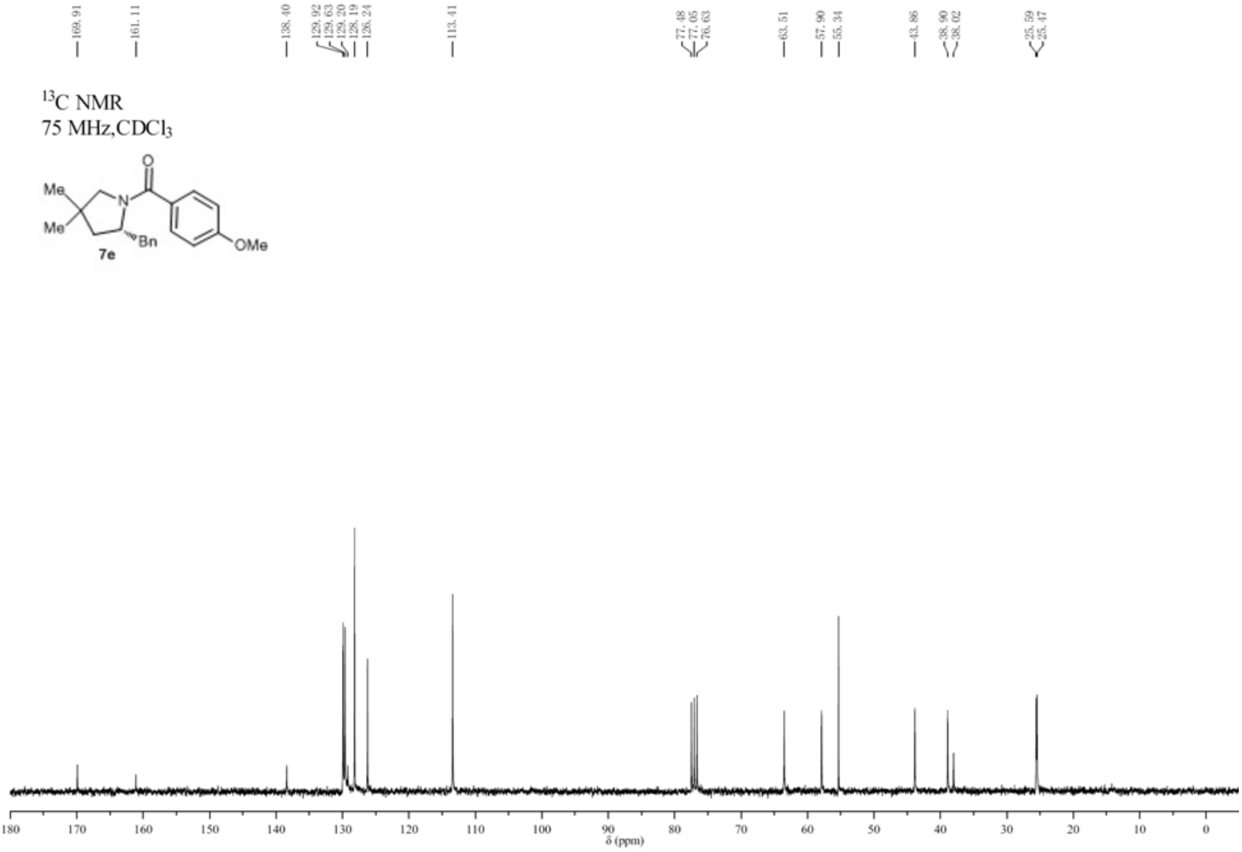

169.85
160.97
129.56
113.39
77.50
77.08
76.65
62.95
55.33
52.99
47.58
38.30
25.80
25.49
20.24
13C NMR
75 MHz,CDCl3
O
Me
Me
N
Me
OMe
7d
δ (ppm)



7.55 7.54 7.53 7.52 7.51 7.50 7.30 7.27 7.25 7.20 6.92 6.90
4.60 4.58 4.55 4.53
3.84
3.29 3.29 3.25 3.24 3.11 3.02 2.98 2.97 2.94 2.93 2.90
1.74 1.74 1.72 1.71 1.70 1.69 1.67 1.60 1.57 1.56 1.53
0.95 0.84
0.00
1H NMR
300 MHz,CDCl3
Me
Me
O
N
Bn
OMe
7e
2.03 5.05 2.04 1.00 3.14 1.02 1.03 2.03 1.07 1.08 3.08 3.04
δ (ppm)



169.91
161.11
138.40
129.92 129.63 129.20 128.19 126.24
113.41
77.48 77.05 76.63
63.51
57.90 55.34
43.86
38.90 38.02
25.59 25.47
13C NMR
75 MHz,CDCl3
Me
Me
O
N
Bn
OMe
7e
δ (ppm)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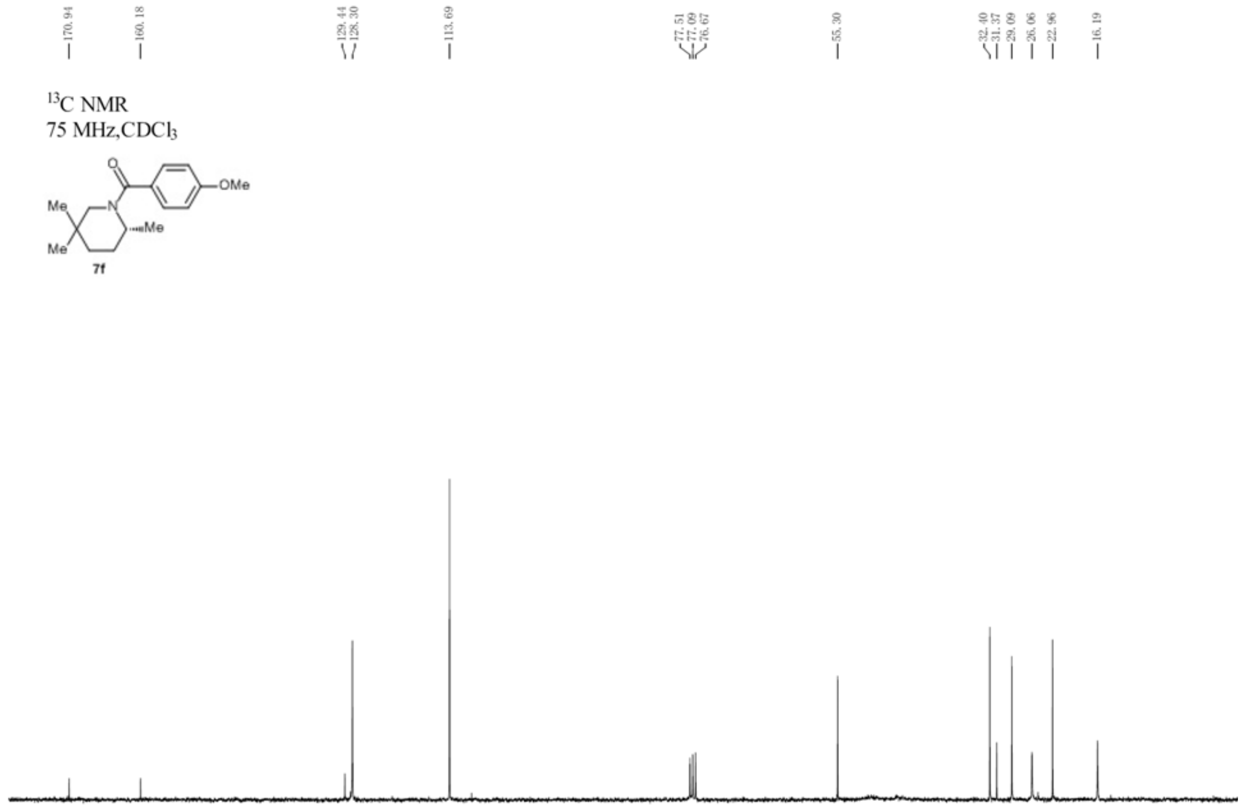
161.11
138.40
129.92
129.63
129.20
128.19
126.24
113.41
77.48
77.05
76.63
63.51
57.90
55.34
43.86
38.90
38.02
25.59
25.47
^{13}C NMR
75 MHz,$CDCl_3$
Me
Me
N
O
Bn
OMe
7e
δ (ppm)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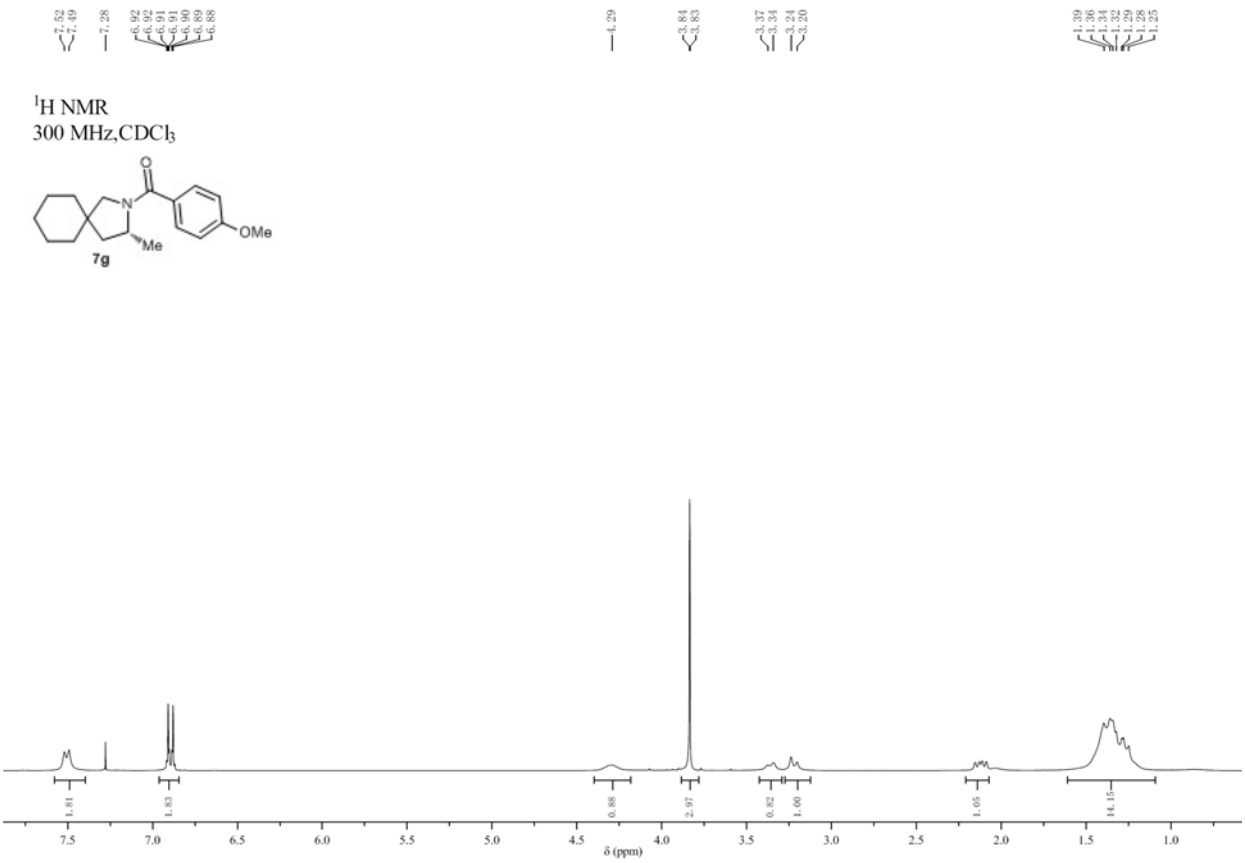
129.44
128.30
113.69
77.51
77.09
76.67
55.30
32.40
31.37
29.09
26.06
22.96
16.19
^{13}C NMR
75 MHz,$CDCl_3$
Me
Me
N
O
Me
OMe
7f
δ (ppm)


7.52
7.49
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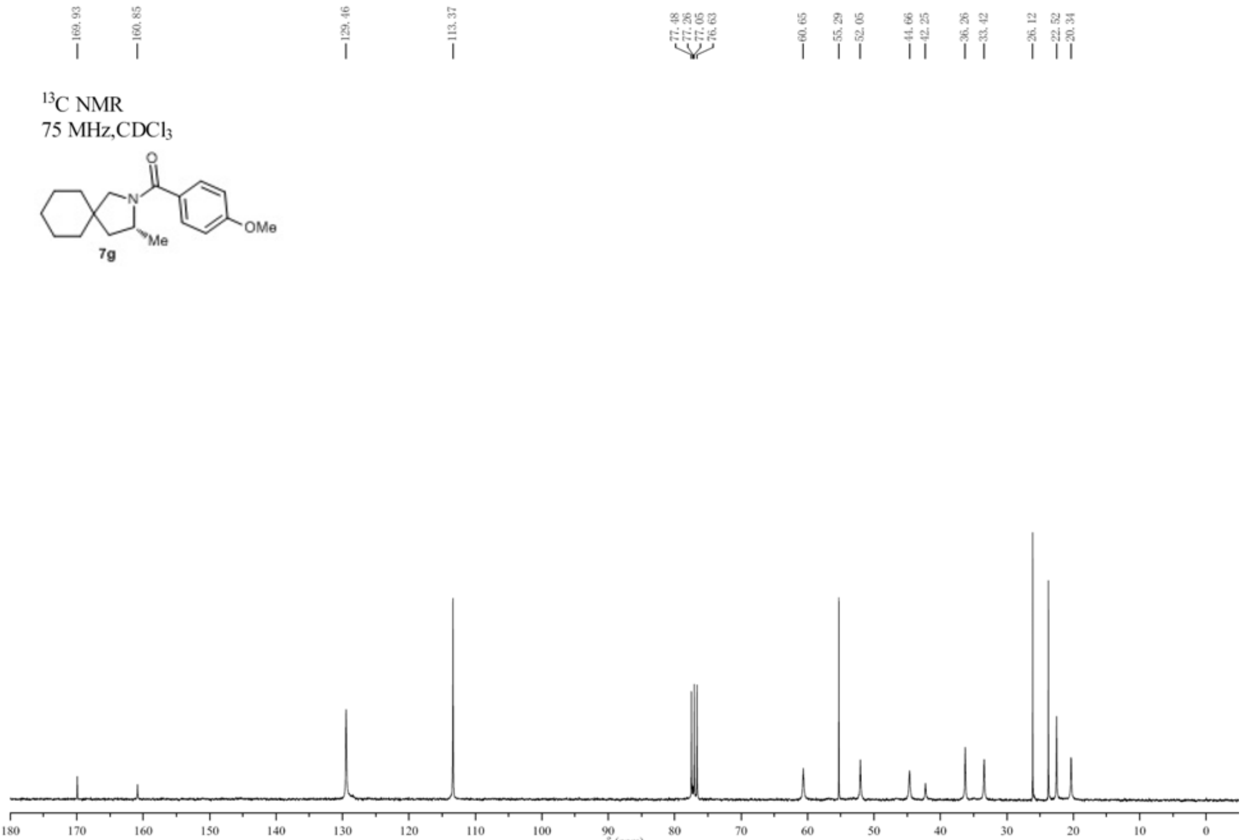
6.92
6.92
6.91
6.91
6.90
6.89
6.88
4.29
3.84
3.83
3.37
3.34
3.24
3.20
1.39
1.36
1.34
1.32
1.29
1.28
1.25
1H NMR
300 MHz,CDCl3
O
N
OMe
Me
7g
1.81
1.83
0.88
2.97
0.82
1.00
1.05
14.15
δ (ppm)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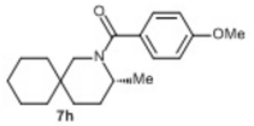
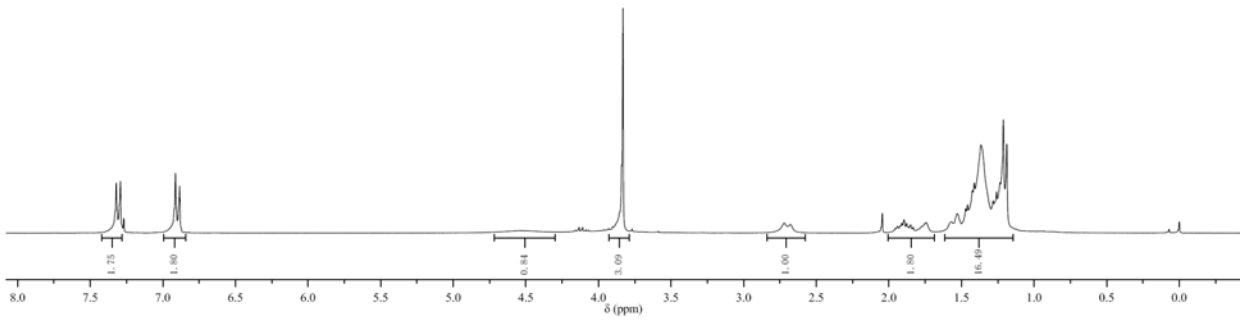
113.37
77.48
77.26
77.05
76.63
60.65
55.29
52.05
44.66
42.25
36.26
33.42
26.12
22.52
20.34
13C NMR
75 MHz,CDCl3
O
N
OMe
Me
7g
δ (ppm)

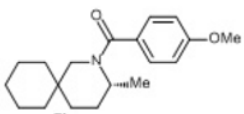
^{1}H NMR
300 MHz, $CDCl_3$



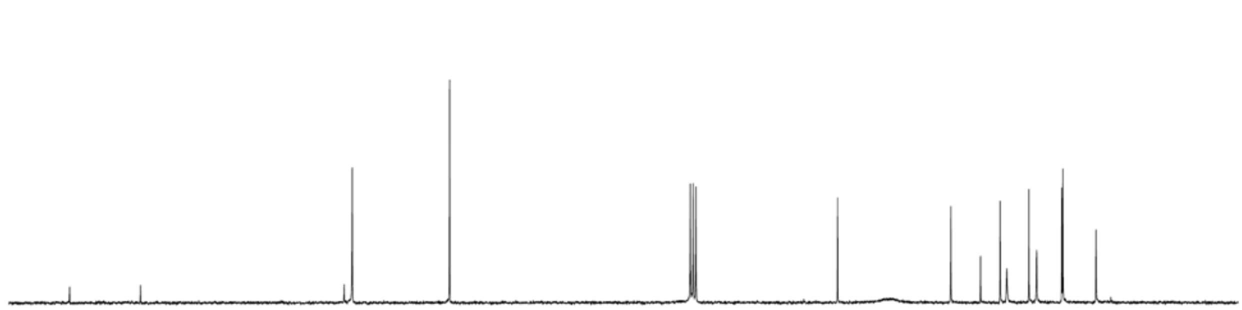
7h

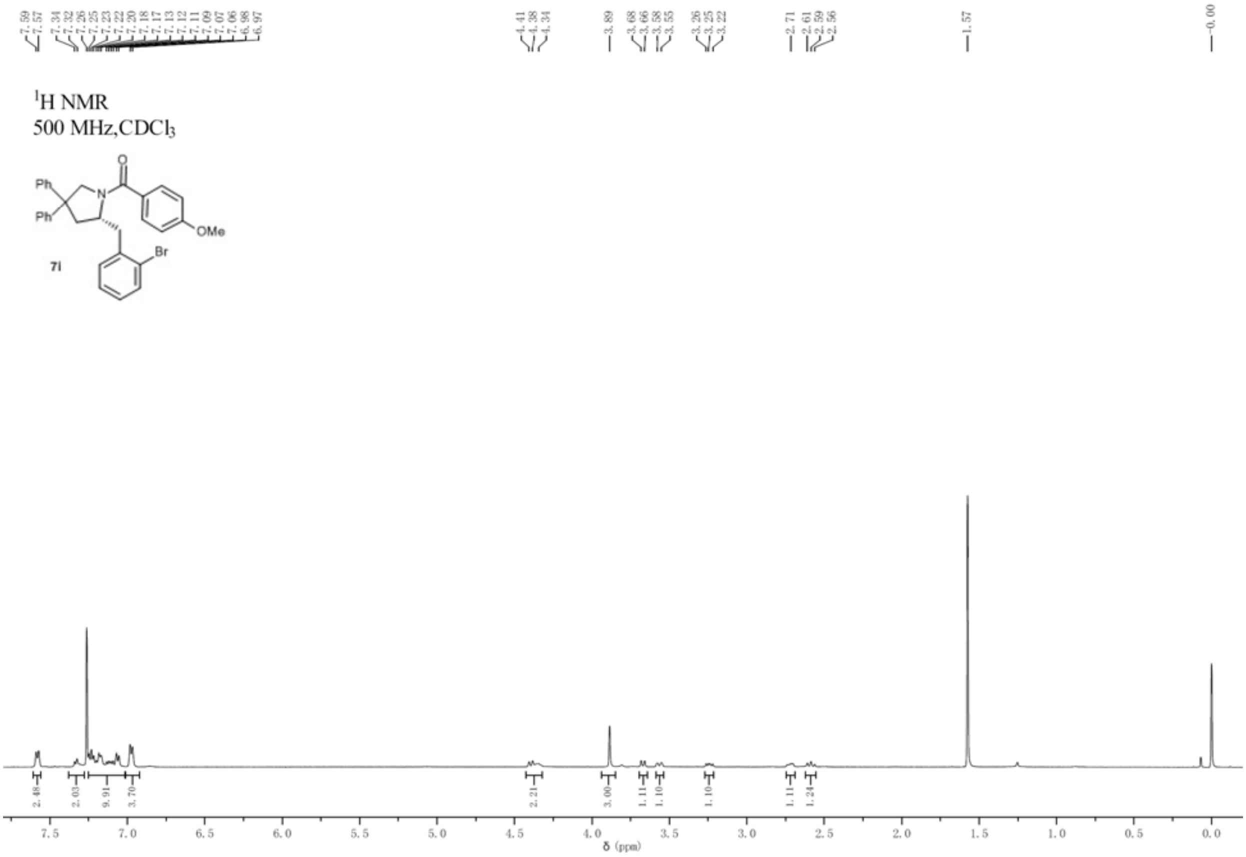


^{13}C NMR
75 MHz, $CDCl_3$



7h




7.59
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7.32
7.26
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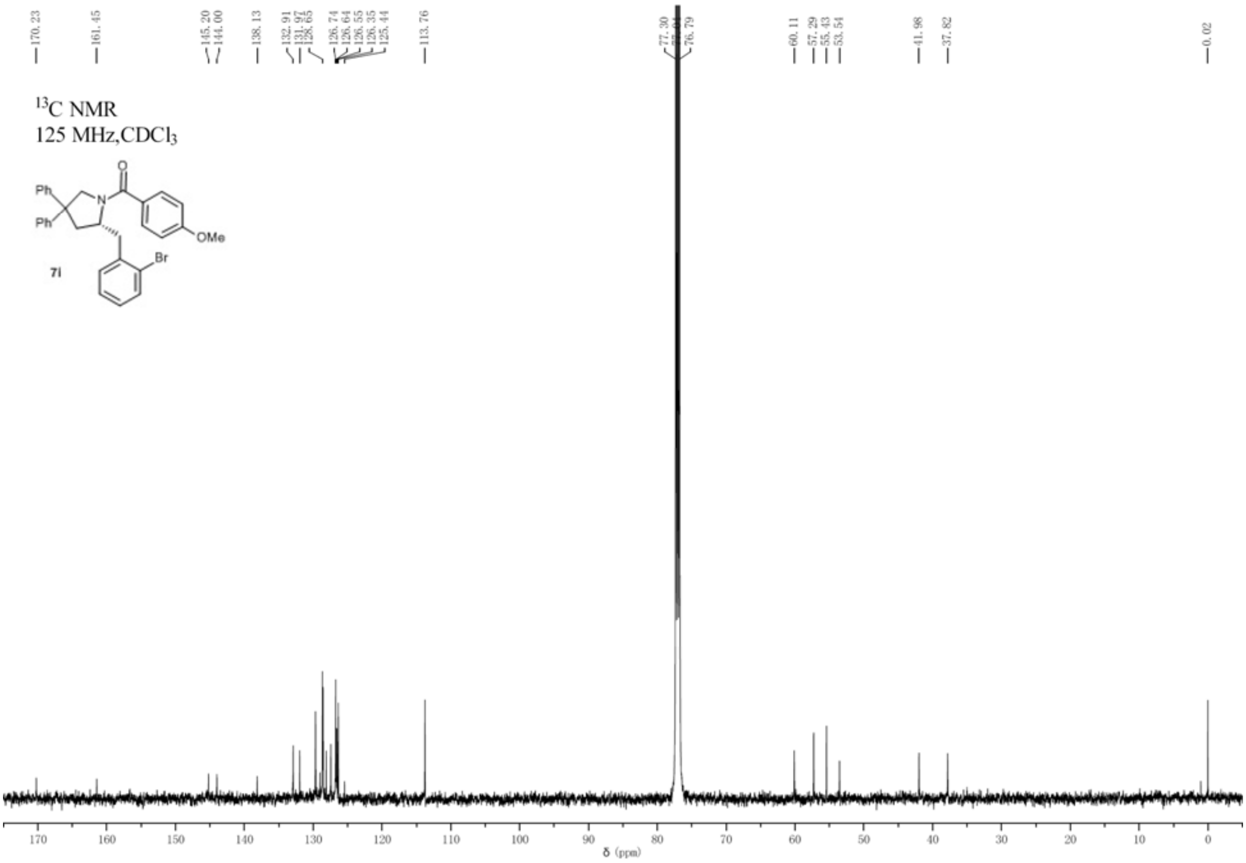
7.23
7.22
7.20
7.18
7.17
7.13
7.12
7.11
7.09
7.07
7.06
6.98
6.97
4.41
4.38
4.34
3.89
3.68
3.66
3.58
3.55
3.26
3.25
3.22
2.71
2.61
2.59
2.56
1.57
0.00
^{1}H NMR
500 MHz,CDCl$_3$
Ph
Ph
N
O
OMe
Br
7i
2.48
2.03
9.91
3.70
2.21
3.00
1.11
1.10
1.10
1.11
1.24
δ (ppm)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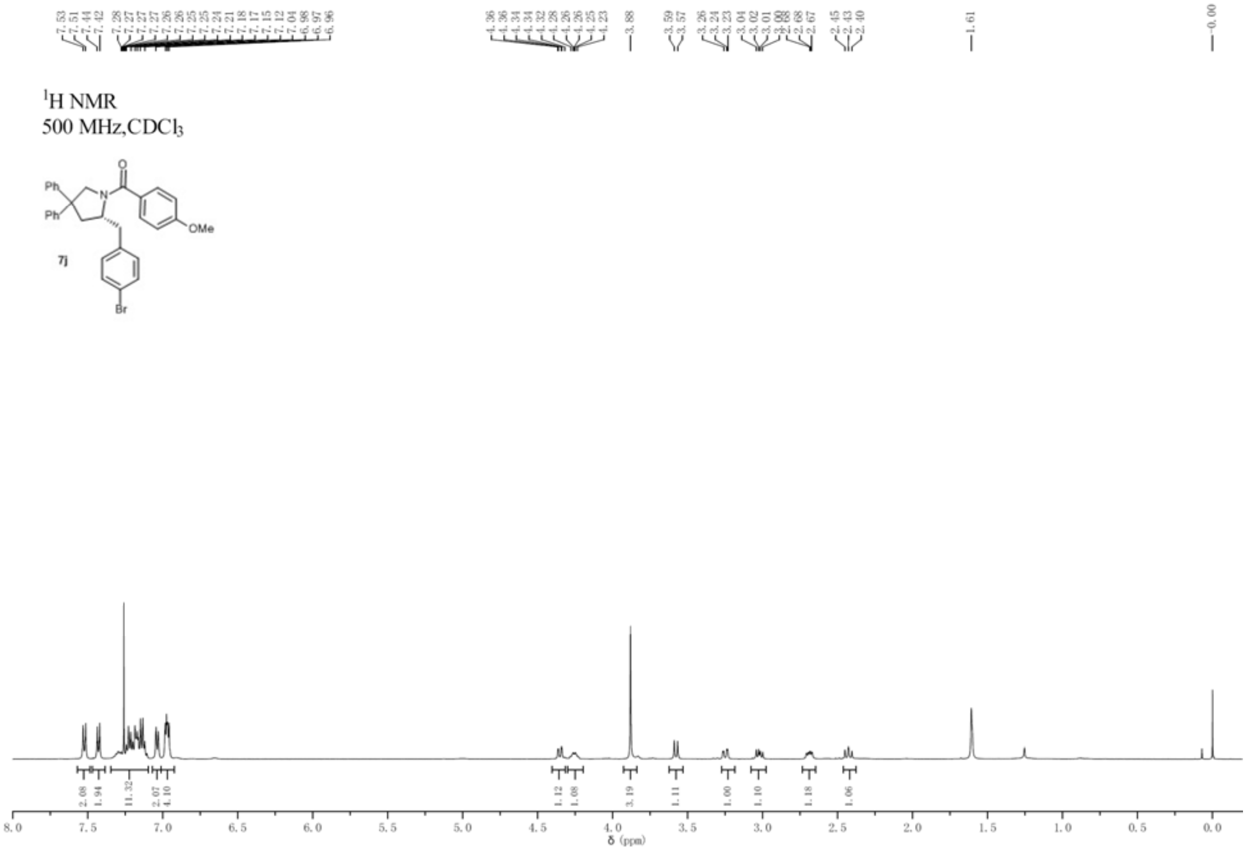
144.00
138.13
132.91
131.97
128.65
126.74
126.64
126.55
126.35
125.44
113.76
77.30
76.79
60.11
57.29
55.43
53.54
41.98
37.82
0.02
^{13}C NMR
125 MHz,CDCl$_3$
Ph
Ph
N
O
OMe
Br
7i
δ (ppm)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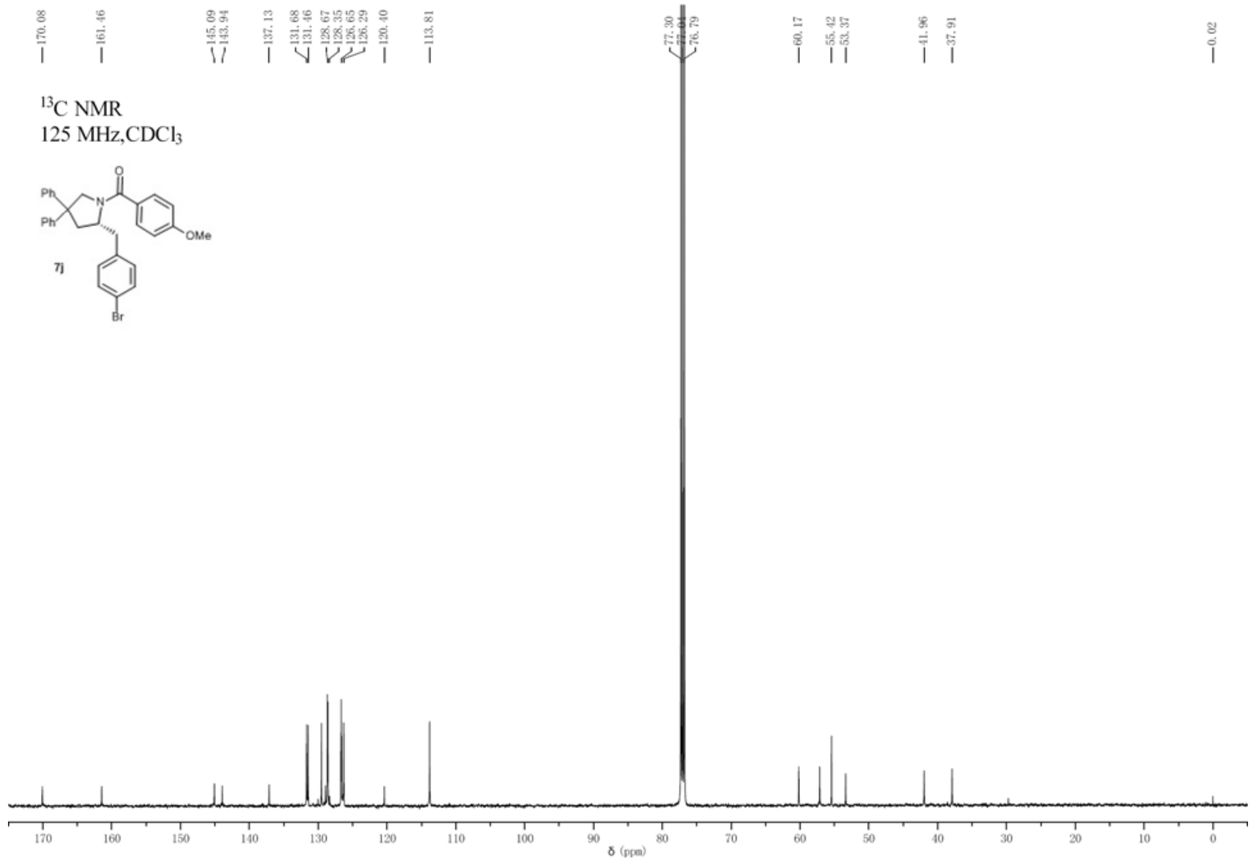
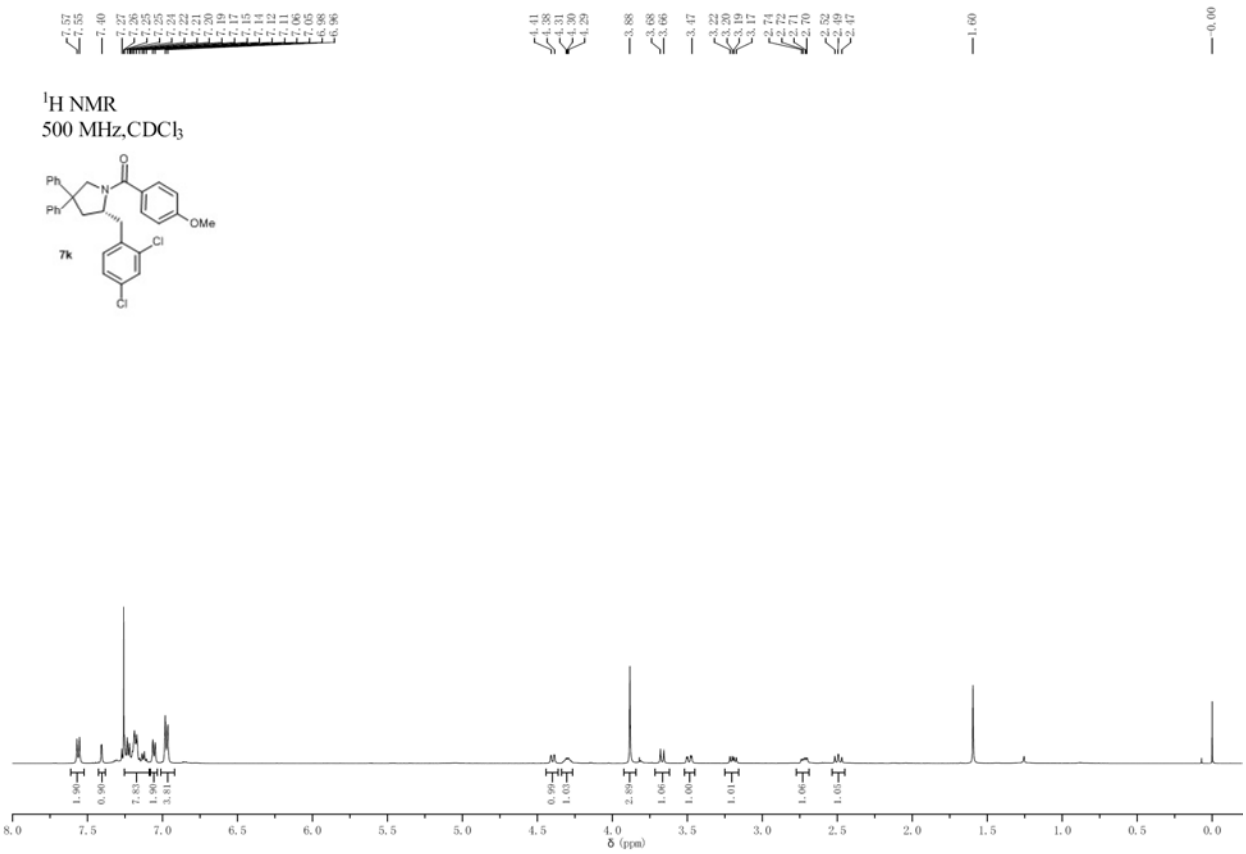
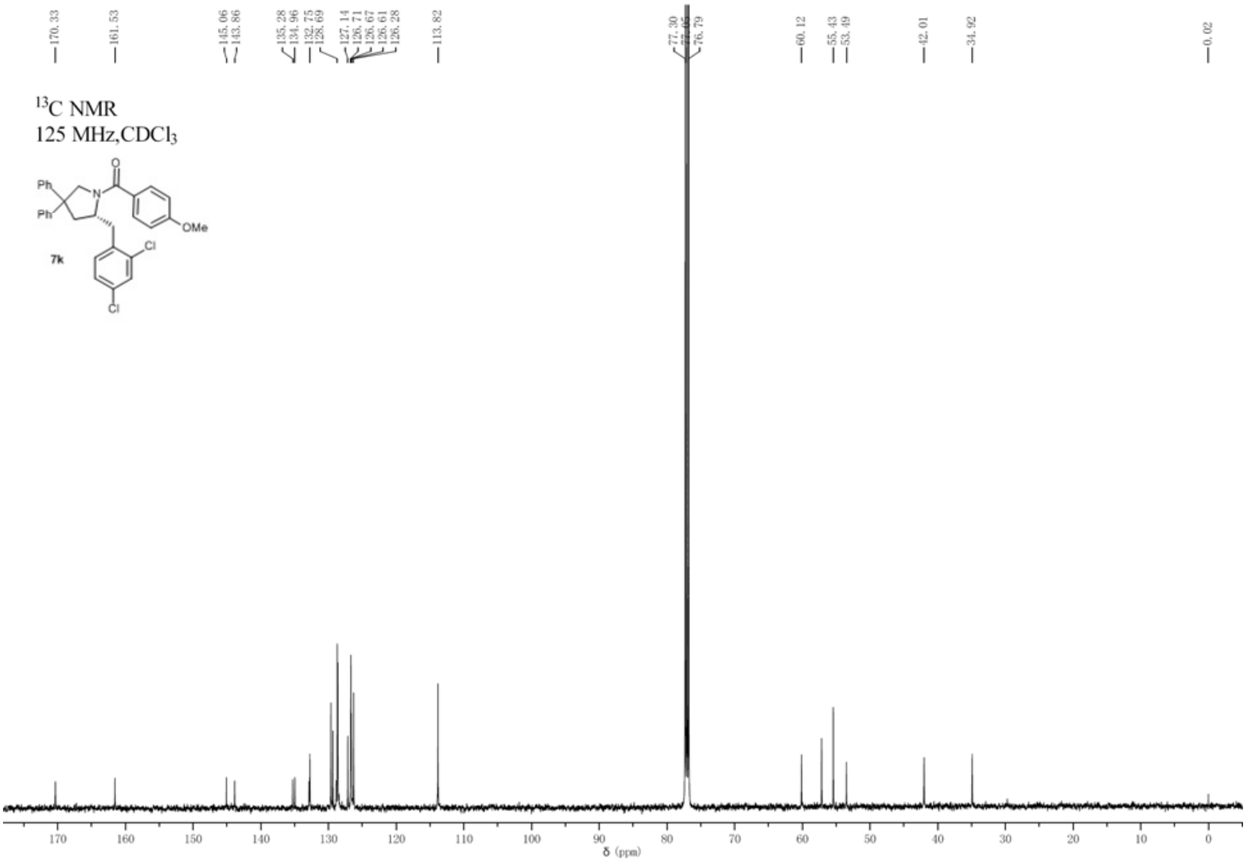
7.27
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.21
7.18
7.17
7.15
7.12
7.04
6.98
6.97
6.96
4.36
4.36
4.34
4.34
4.32
4.28
4.26
4.26
4.25
4.23
3.88
3.59
3.57
3.26
3.24
3.23
3.04
3.02
3.01
2.68
2.67
2.45
2.43
2.40
1.61
0.00
^{1}H NMR
500 MHz,CDCl$_3$
Ph
Ph
N
O
OMe
7j
Br
2.08
1.94
11.32
2.07
4.10
1.12
1.08
3.19
1.11
1.00
1.10
1.18
1.06
δ (ppm)

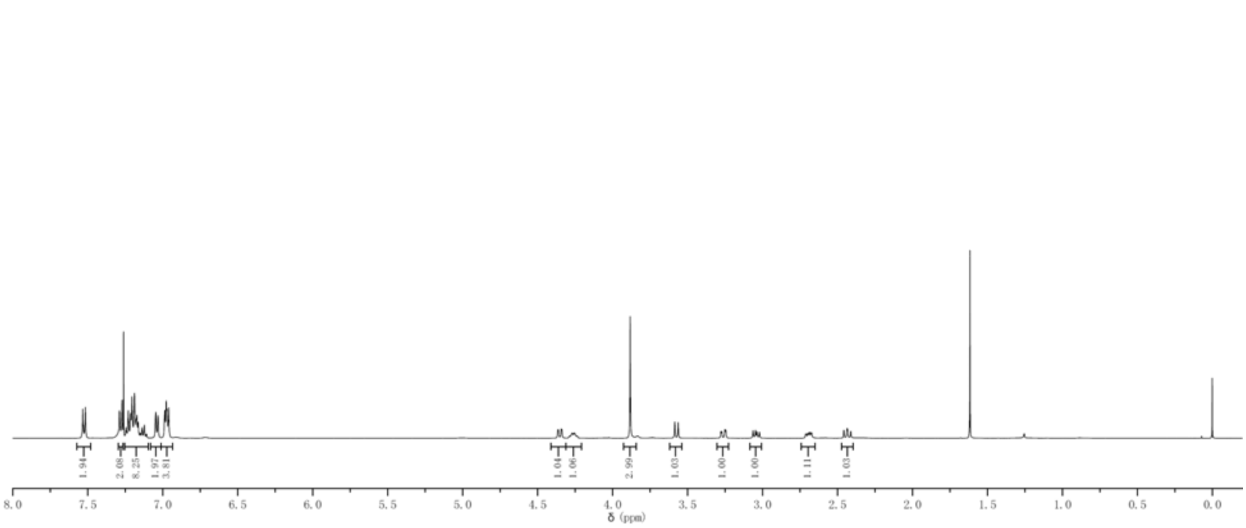

170.08
161.46
145.09
143.94
137.13
131.68
131.46
128.67
128.35
126.65
126.29
120.40
113.81
77.30
76.79
60.17
55.42
53.37
41.96
37.91
0.02
^{13}C NMR
125 MHz,CDCl$_3$
Ph
Ph
N
O
OMe
7j
Br
δ (ppm)

^{1}H NMR
500 MHz, $CDCl_3$

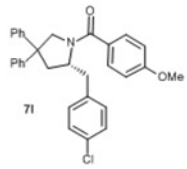


^{13}C NMR
125 MHz, $CDCl_3$

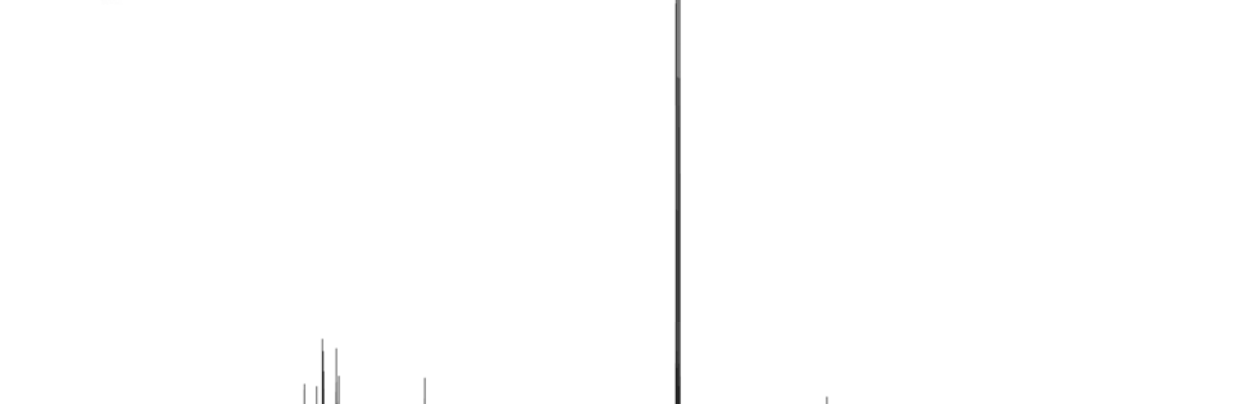




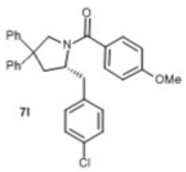
^{1}H NMR
500 MHz, $CDCl_3$



7l

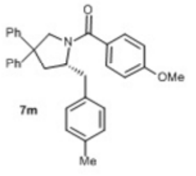


^{13}C NMR
125 MHz, $CDCl_3$

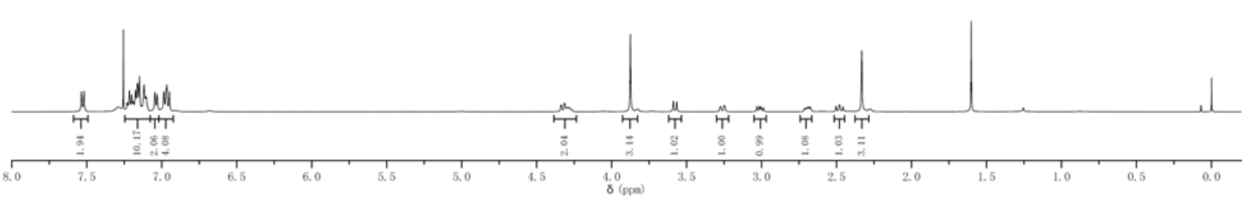


7l

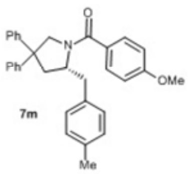
^{1}H NMR
500 MHz, $CDCl_3$



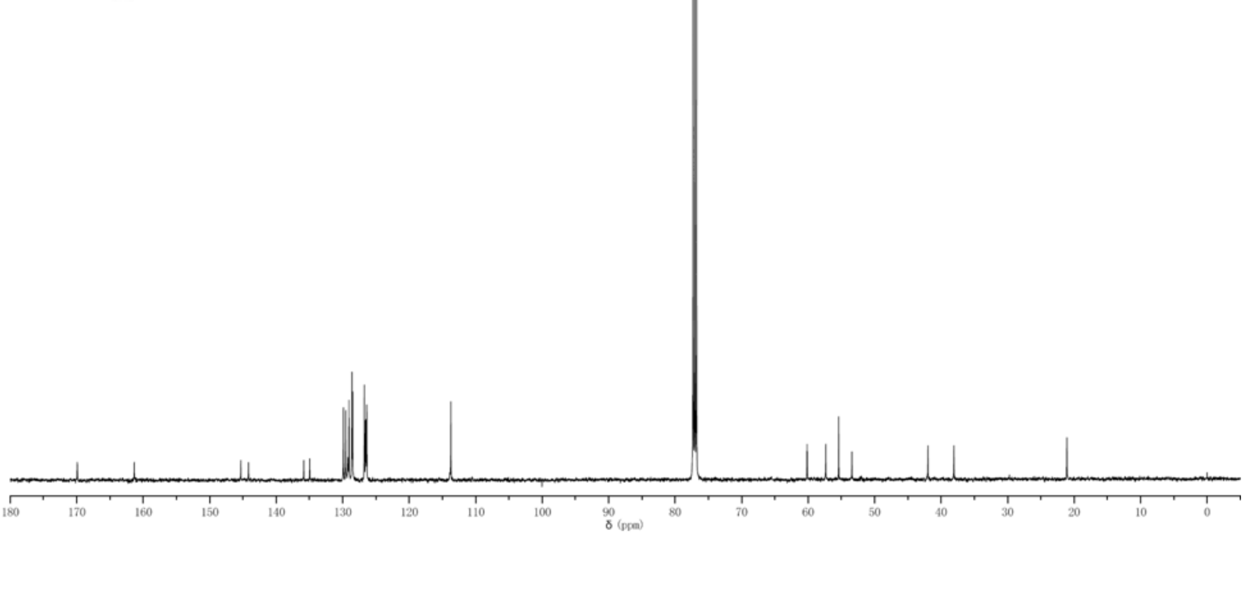
7m



^{13}C NMR
125 MHz, $CDCl_3$

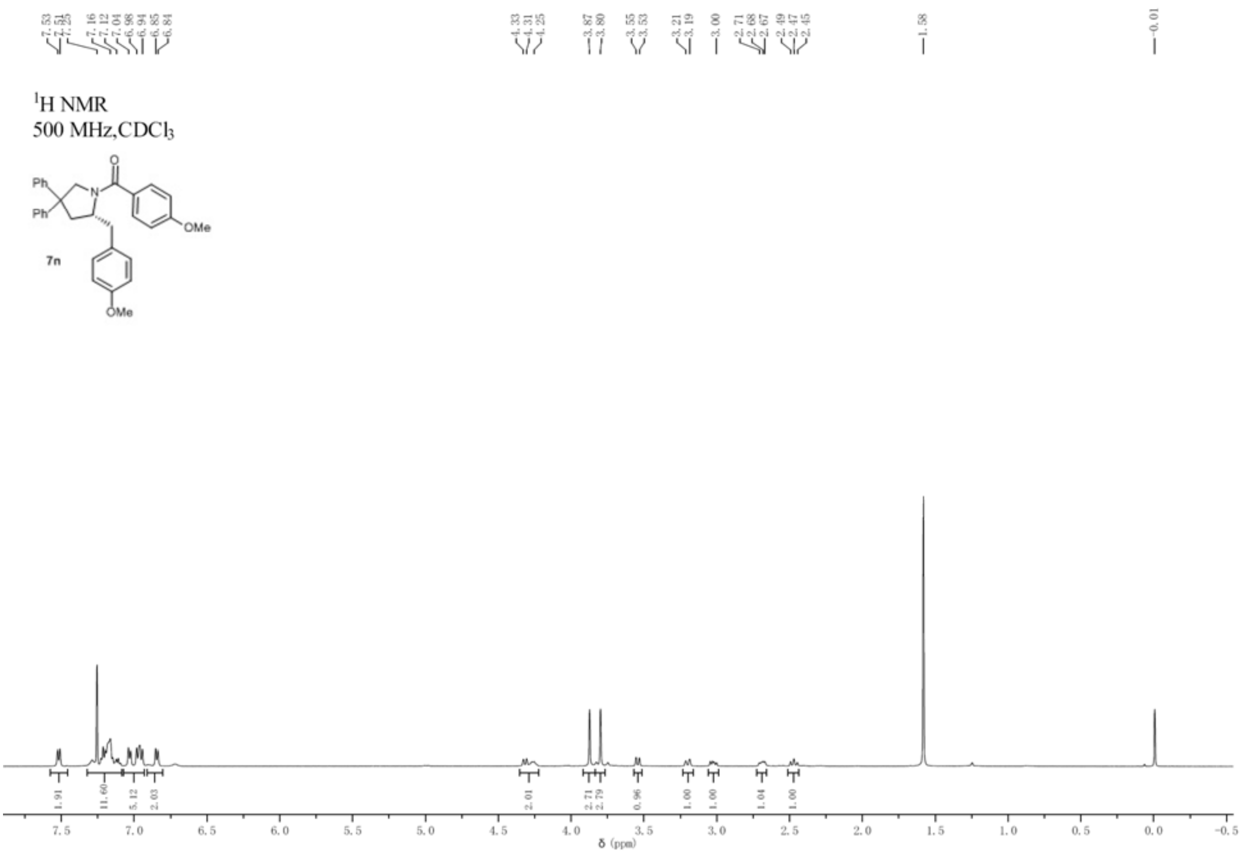


7m



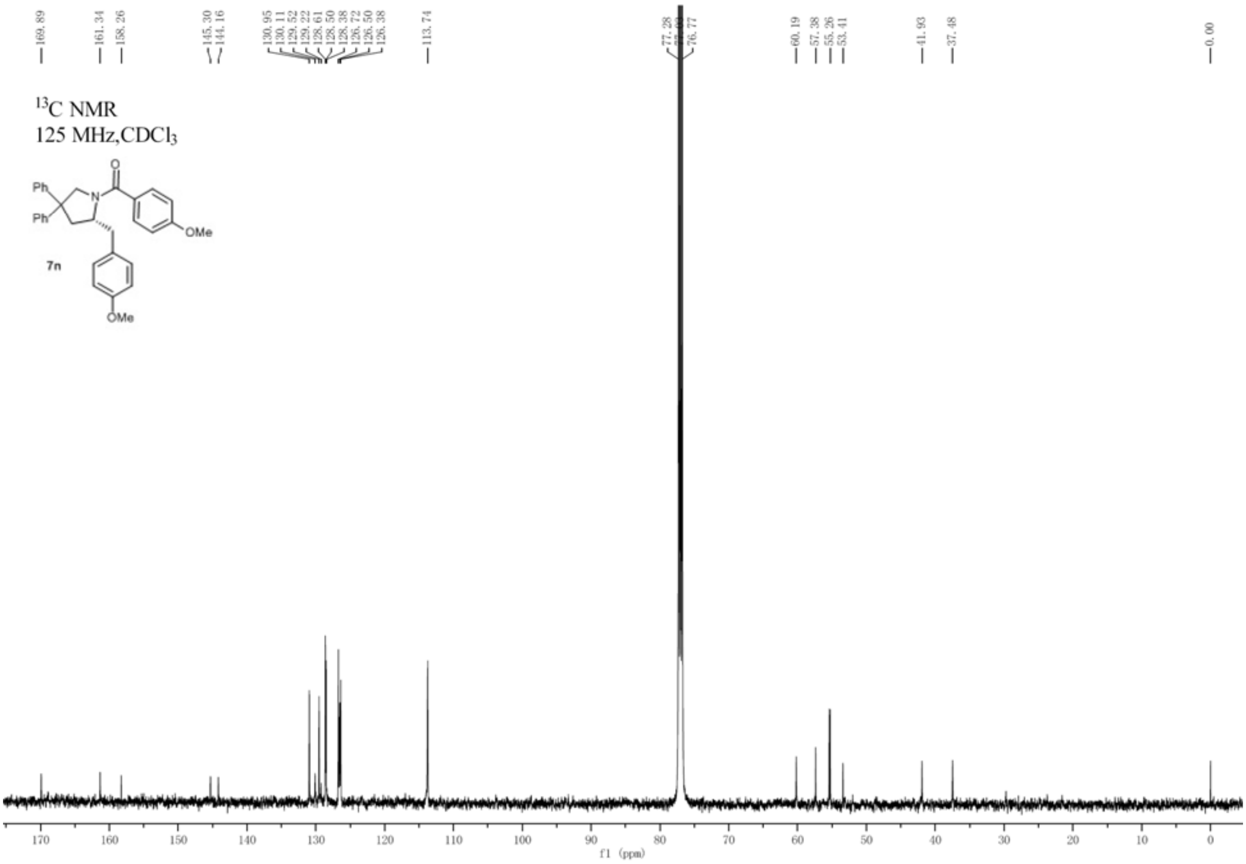
^{1}H NMR
500 MHz, $CDCl_3$

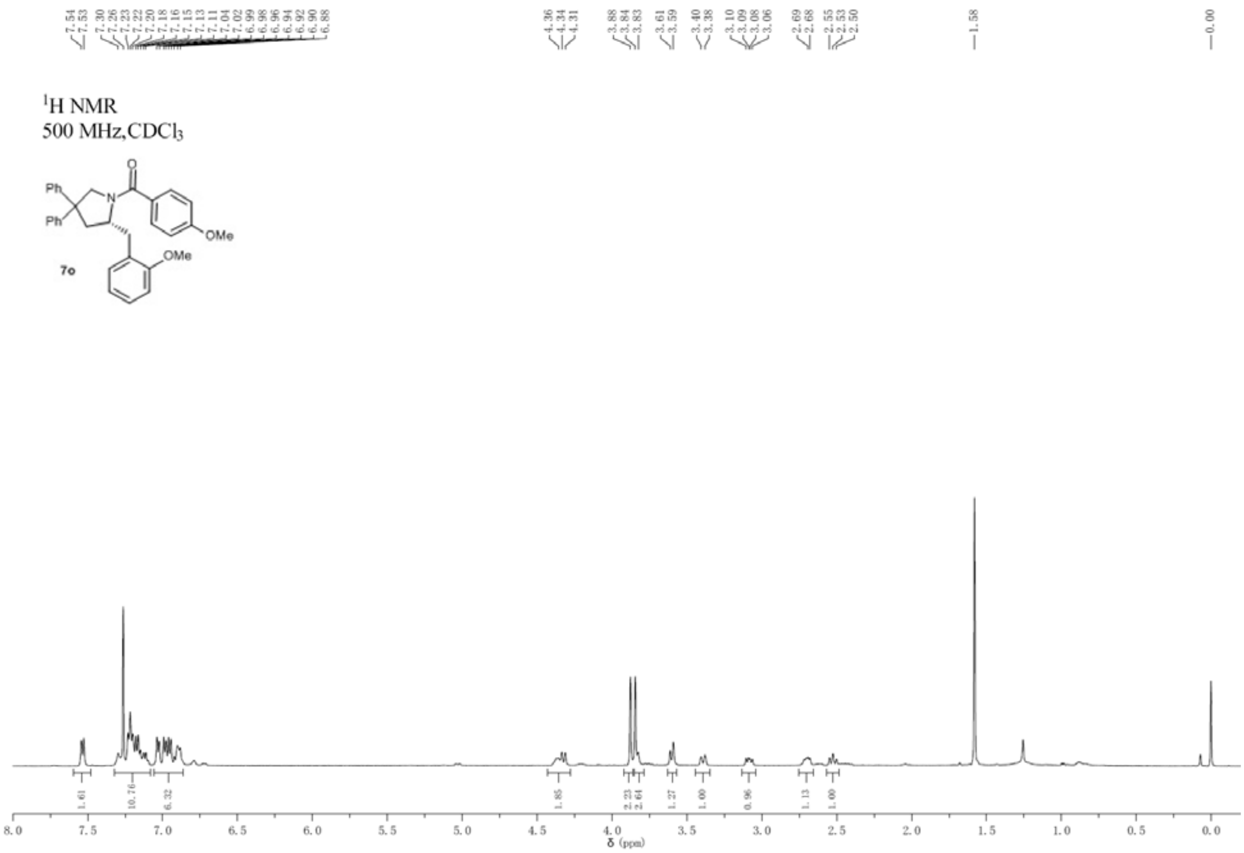
7n



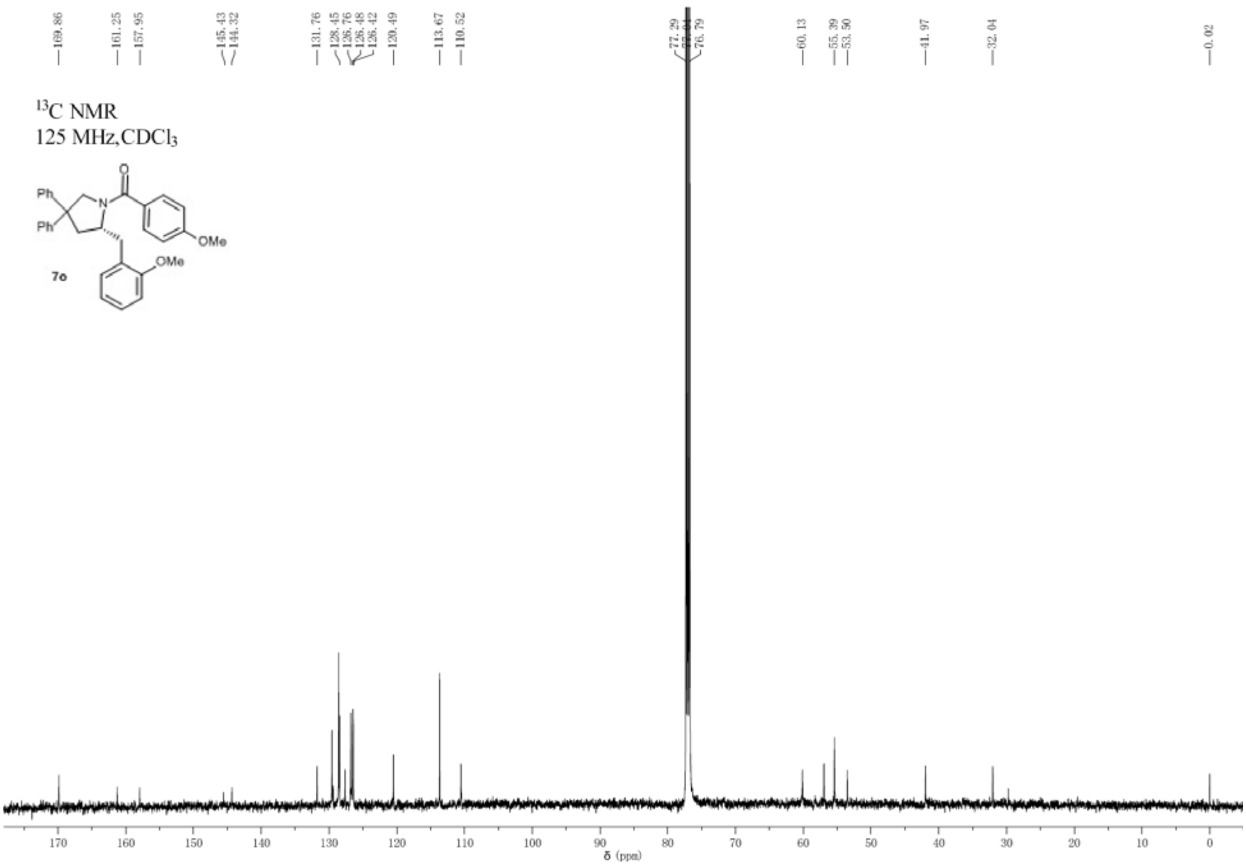
^{13}C NMR
125 MHz, $CDCl_3$

7n

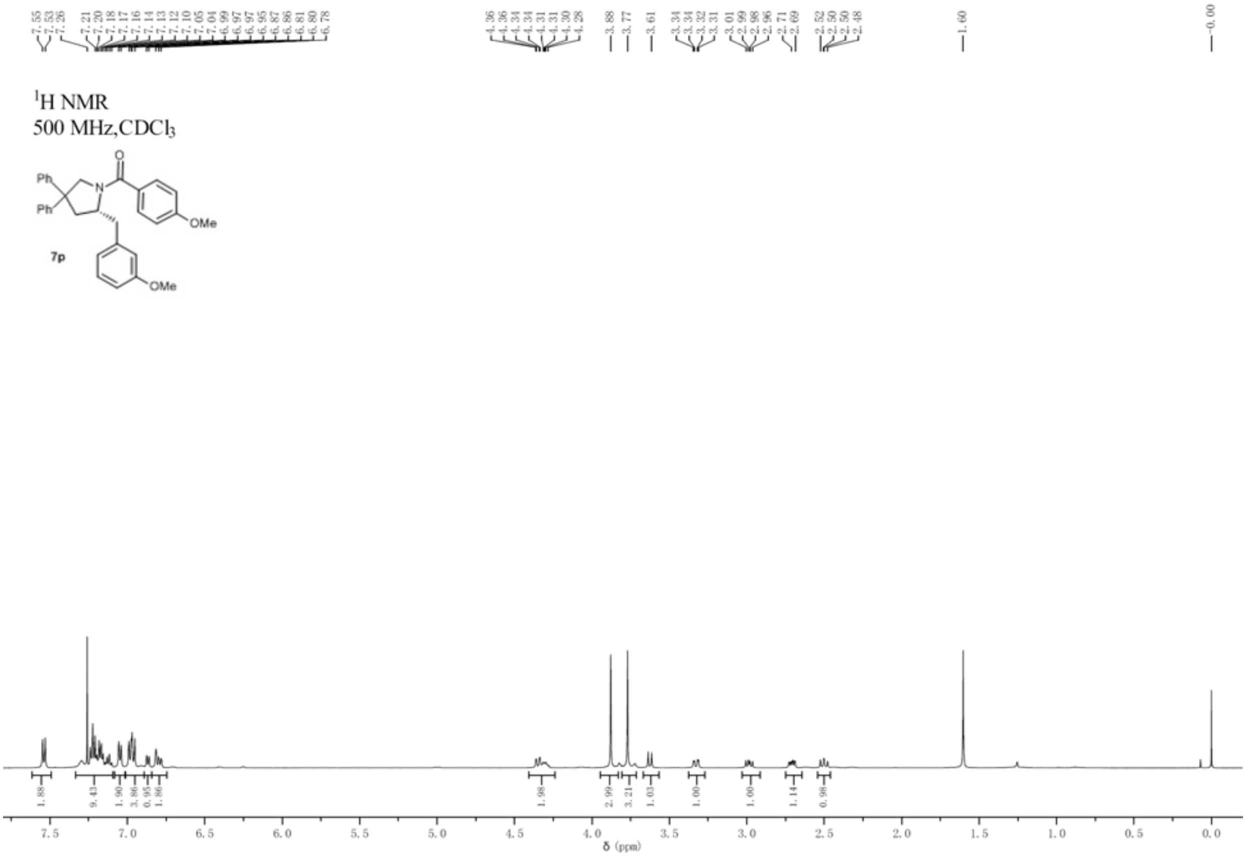




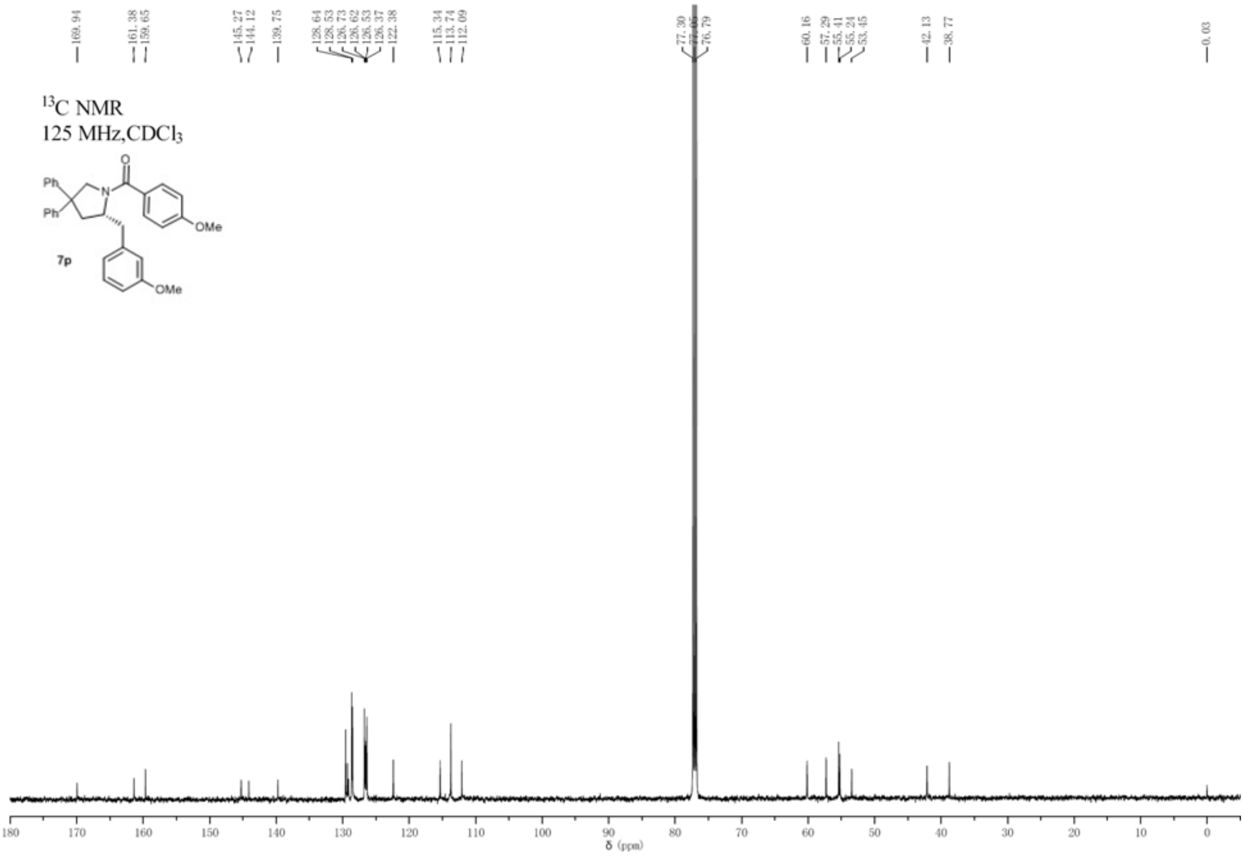
7.54
7.53
7.30
7.26
7.23
7.22
7.20
7.18
7.16
7.15
7.13
7.11
7.04
7.02
6.99
6.98
6.96
6.94
6.92
6.90
6.88
4.36
4.34
4.31
3.88
3.84
3.83
3.61
3.59
3.40
3.38
3.10
3.09
3.08
3.06
2.69
2.68
2.55
2.53
2.50
1.58
0.00
^{1}H NMR
500 MHz,CDCl$_3$
Ph
Ph
N
O
OMe
OMe
7o
1.61
10.76
6.32
1.85
2.23
2.64
1.27
1.00
0.96
1.13
1.00
δ (ppm)



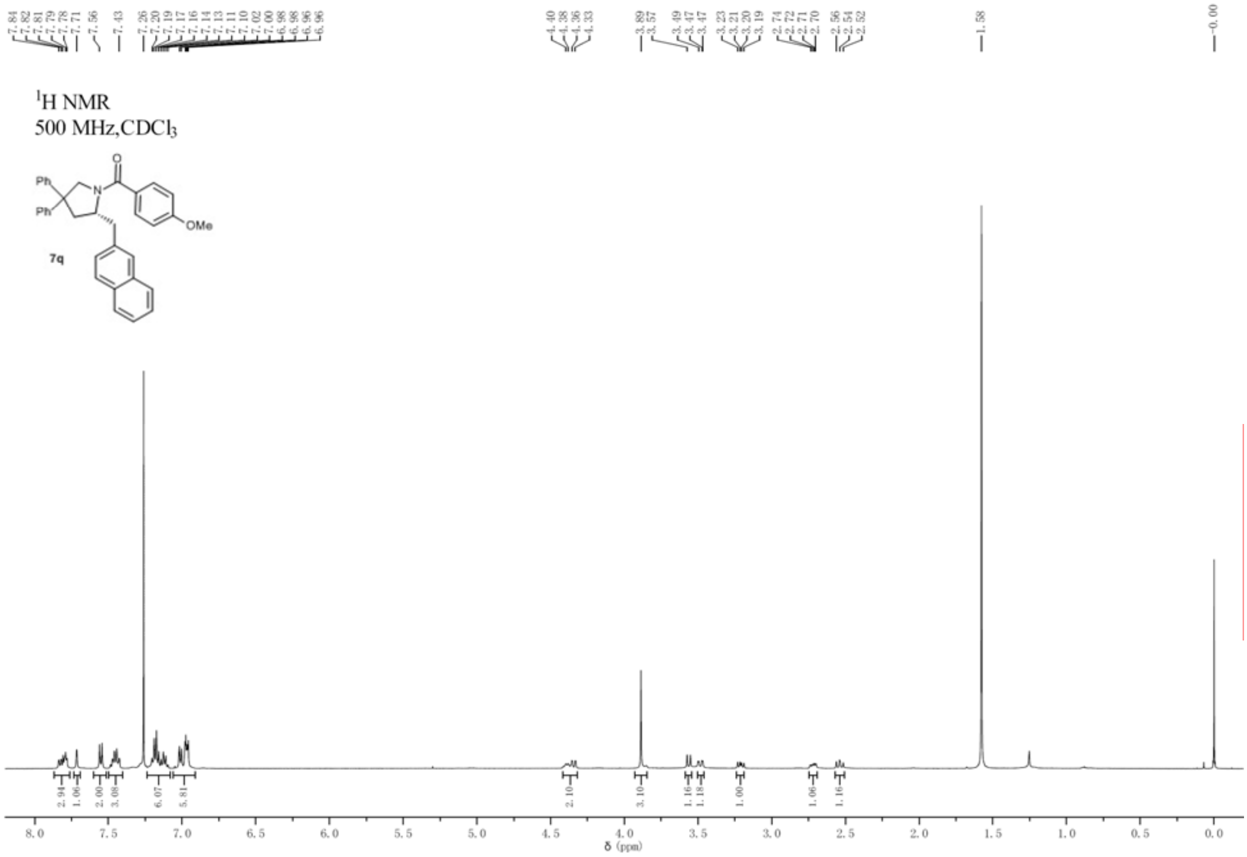
169.86
161.25
157.95
145.43
144.32
131.76
128.45
126.76
126.48
126.42
120.49
113.67
110.52
77.29
76.79
60.13
55.39
53.50
41.97
32.04
0.02
^{13}C NMR
125 MHz,CDCl$_3$
Ph
Ph
N
O
OMe
OMe
7o
δ (ppm)



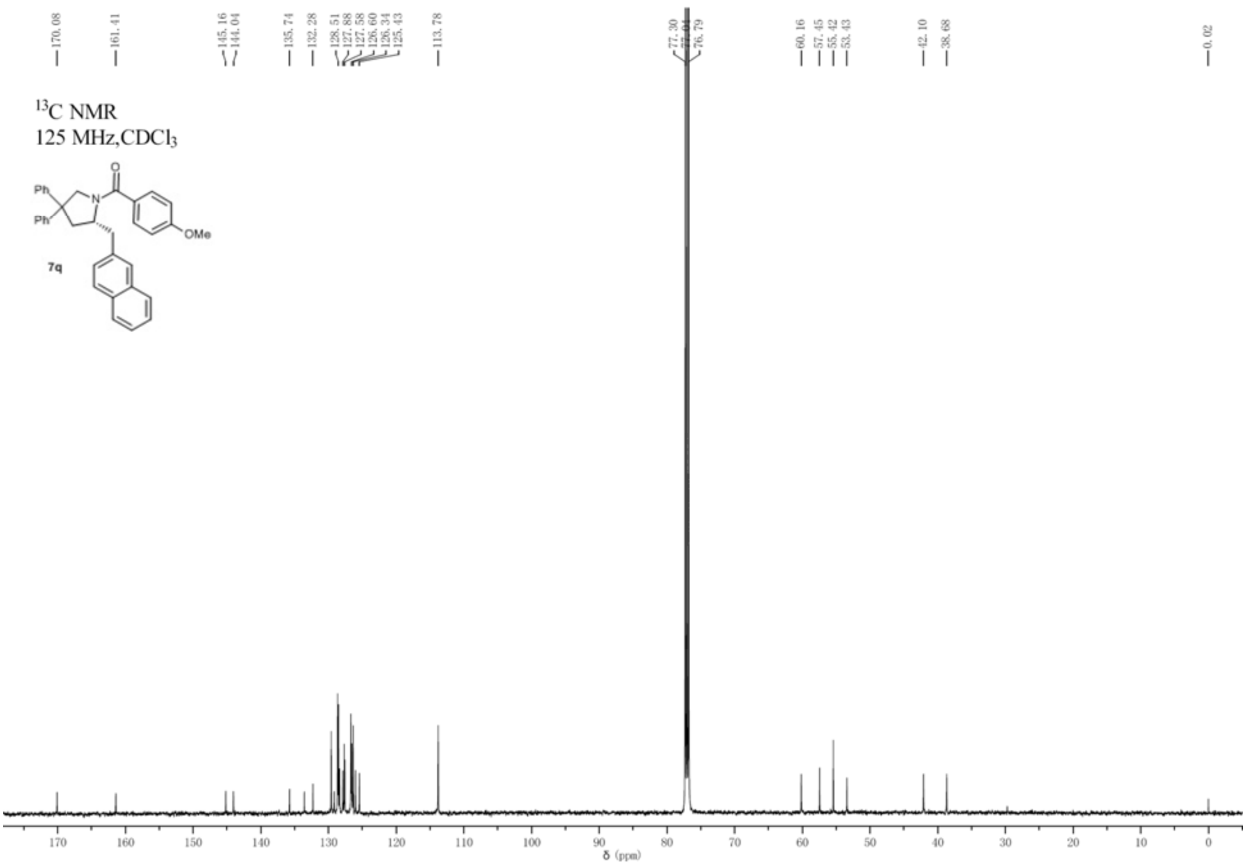
^{1}H NMR
500 MHz,CDCl$_3$
7p



^{13}C NMR
125 MHz,CDCl$_3$
7p

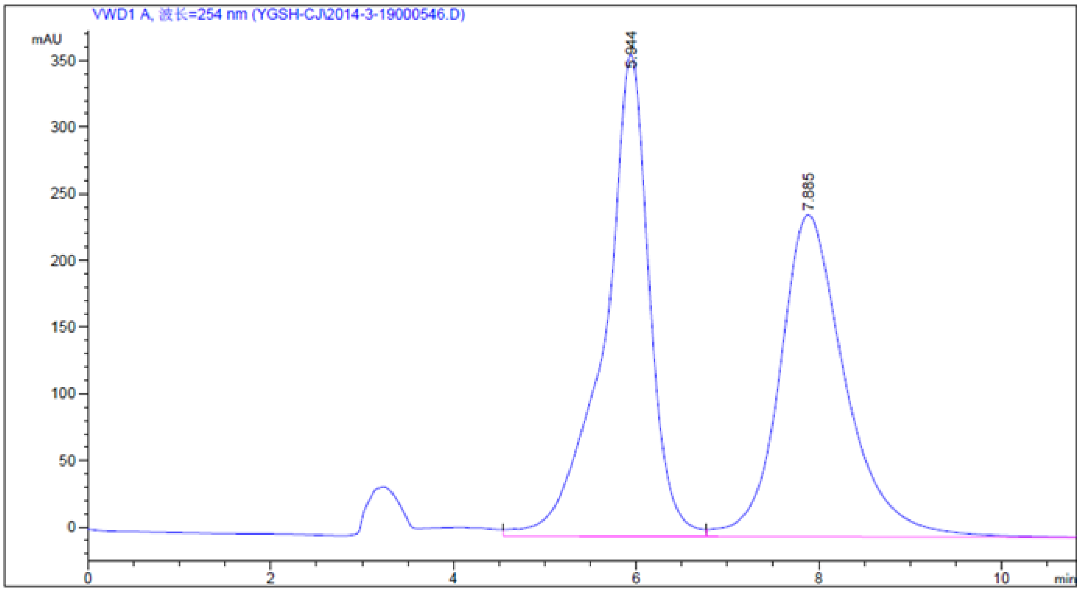


1H NMR
500 MHz,CDCl3
7q
OMe
Ph
δ (ppm)



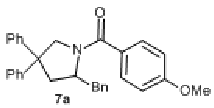
13C NMR
125 MHz,CDCl3
7q
OMe
Ph
δ (ppm)

HPLC profile of 4-methoxybenzoyl amides



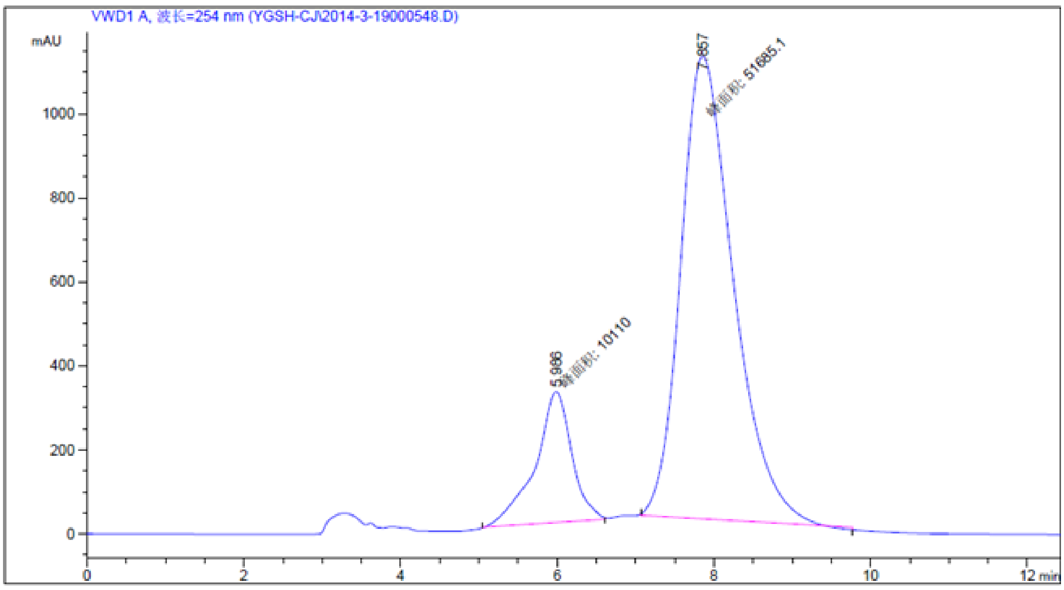
面积百分比报告

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.944	VV	0.4936	1.23850e4	360.86856	50.7599
2	7.885	VBA	0.7386	1.20141e4	241.11989	49.2401



Racemic

OD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.986	MM	0.5411	1.01100e4	311.42639	16.3606
2	7.857	MM	0.7819	5.16851e4	1101.75171	83.6394

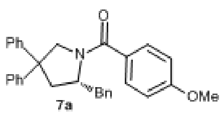
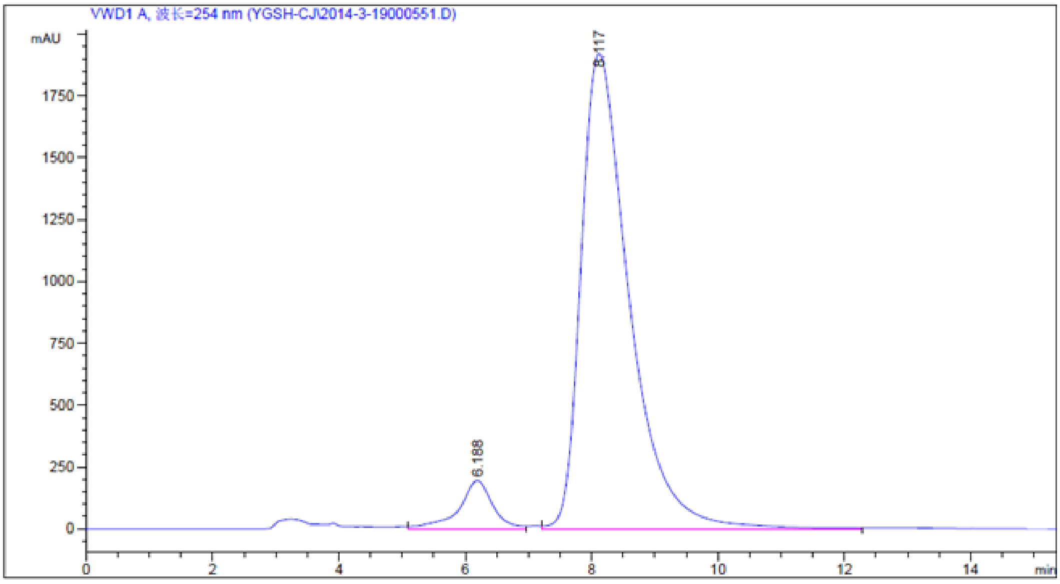


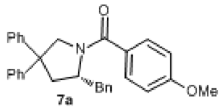
Table 2 entry 1 and Table 3 entry 1

OD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

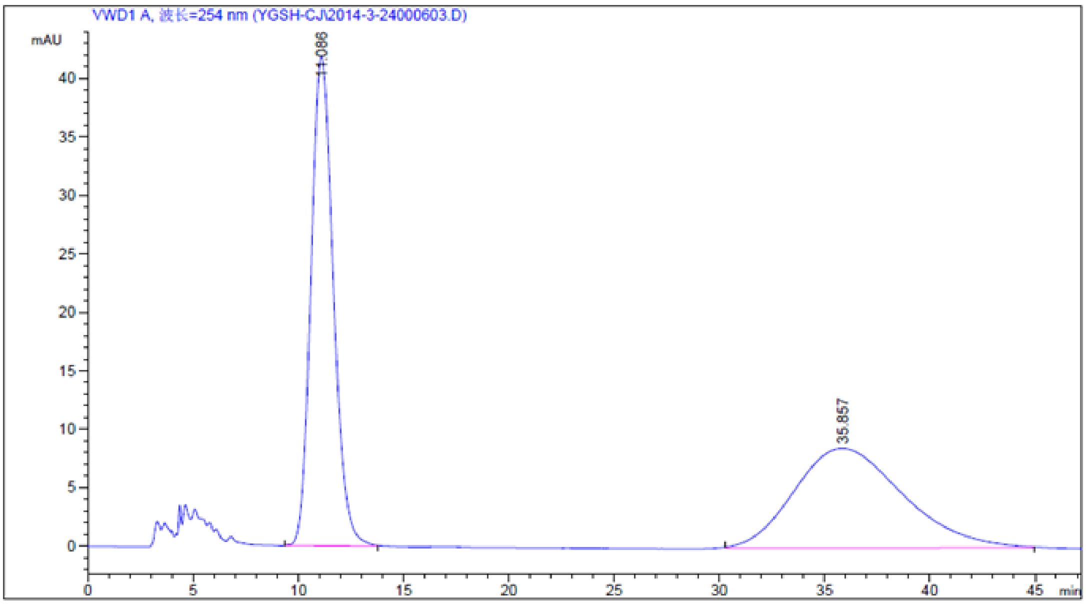
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	6.188	VV	0.5404	7288.65430	195.22932	6.6629
2	8.117	VV	0.8006	1.02104e5	1921.02576	93.3371



7a

Table 2 entry 3
and
Table 4 entry 1

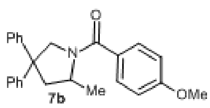
OD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

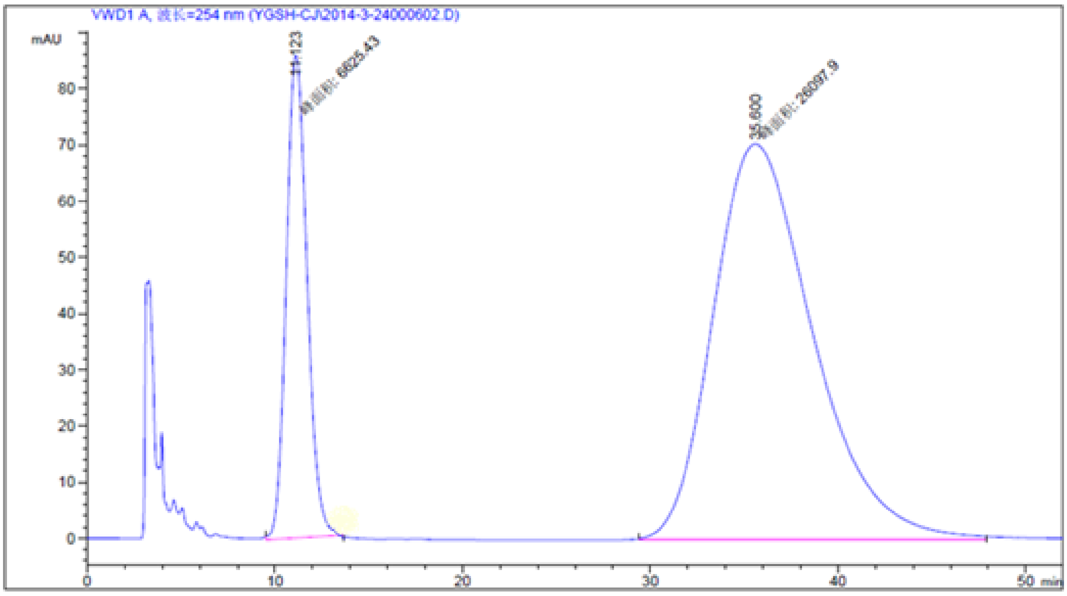
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	11.086	BB	1.1472	3093.23022	41.87378	50.3221
2	35.857	BB	4.2177	3053.63184	8.51730	49.6779

Racemic



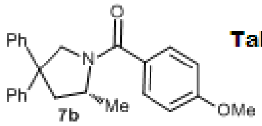
7b

AS-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

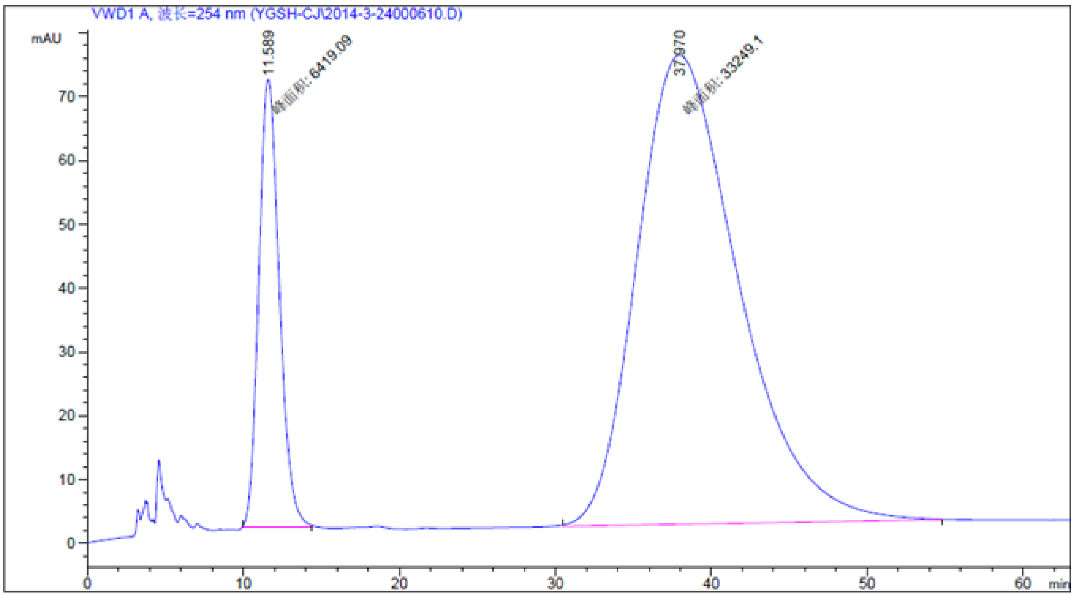
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	11.123	MM	1.2854	6625.43359	85.90742	20.2468
2	35.600	MM	6.1738	2.60979e4	70.45306	79.7532



7b

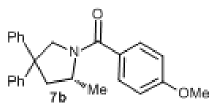
Table 3 entry 2

AS-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

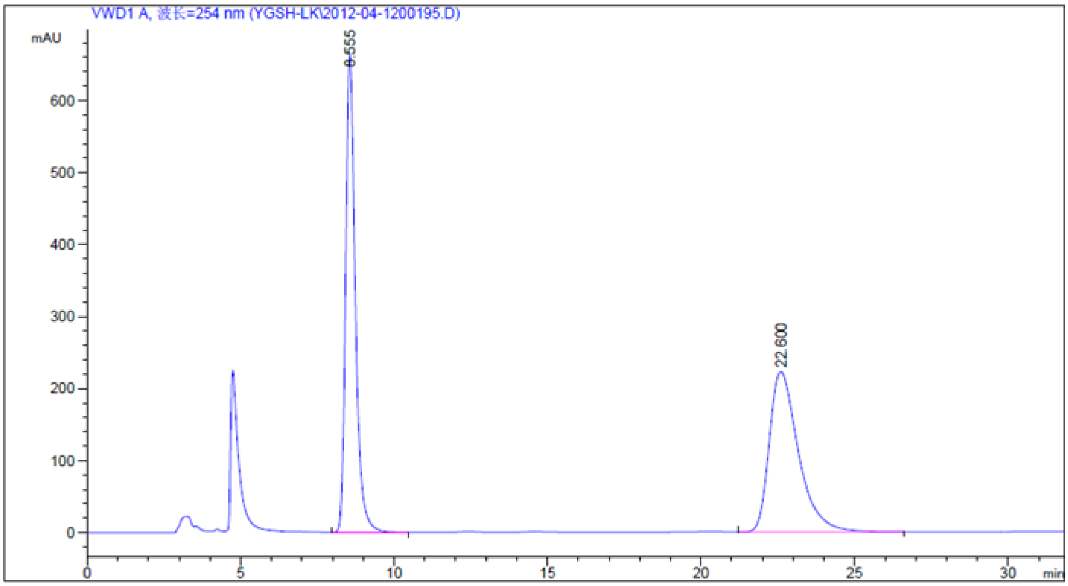
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	11.589	MM	1.5264	6419.09033	70.09112	16.1819
2	37.970	MM	7.5326	3.32491e4	73.56719	83.8181



7b

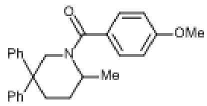
Table 4 entry 2

AS-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

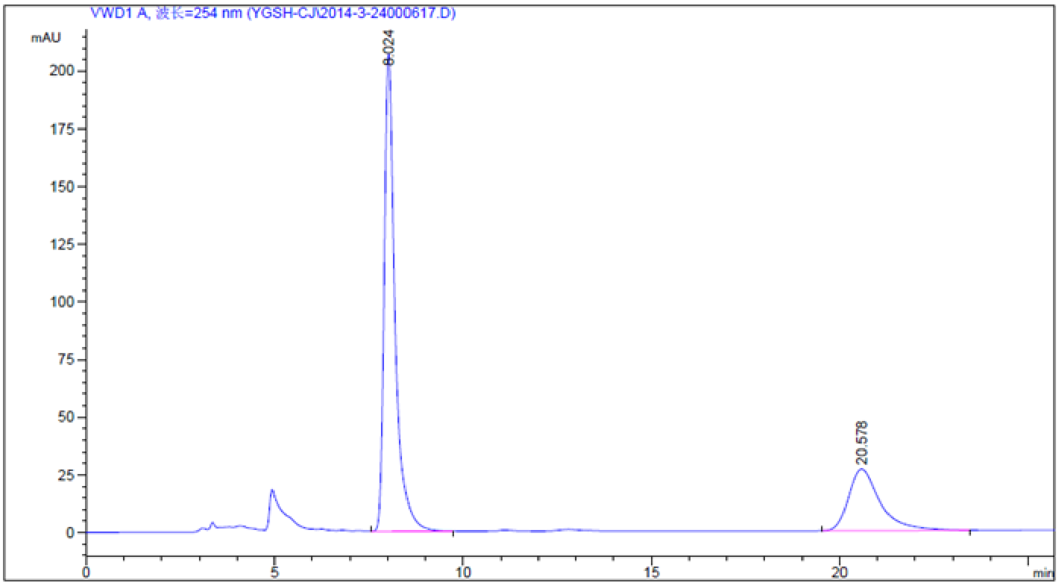
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.555	VB	0.3341	1.47049e4	663.85712	50.0509
2	22.600	VB	1.0038	1.46750e4	222.09303	49.9491



7c

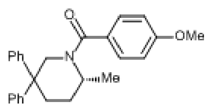
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

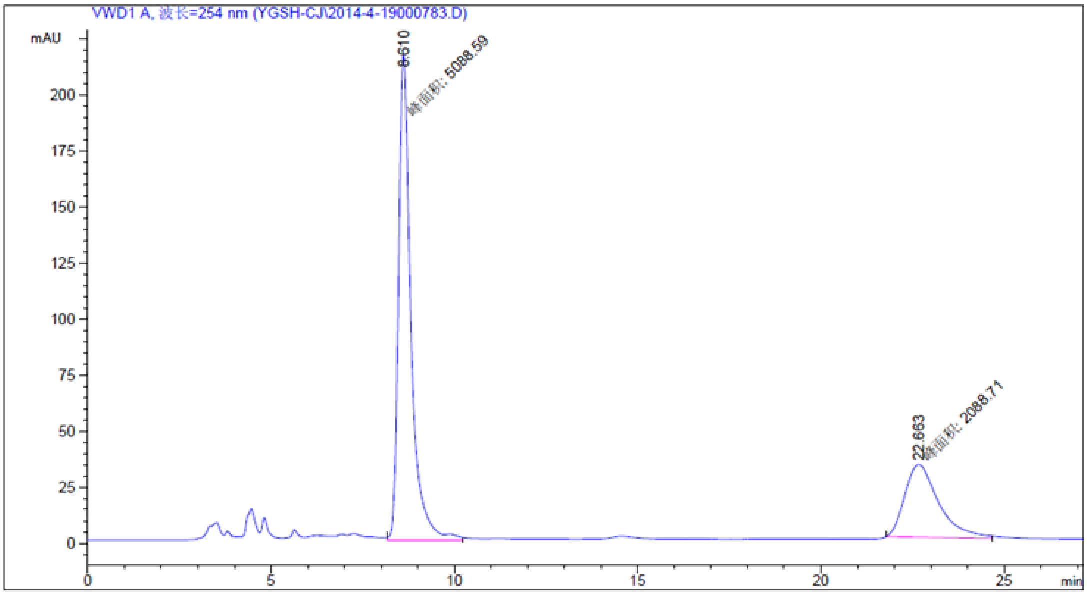
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.024	VB	0.2995	4175.95752	206.91273	72.6165
2	20.578	BB	0.8786	1574.74304	26.76647	27.3835



7c

Table 3 entry 3

AD-H, hexane/2-propanol=1:1,1mL/min



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面积百分比报告

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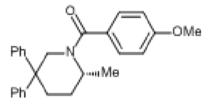
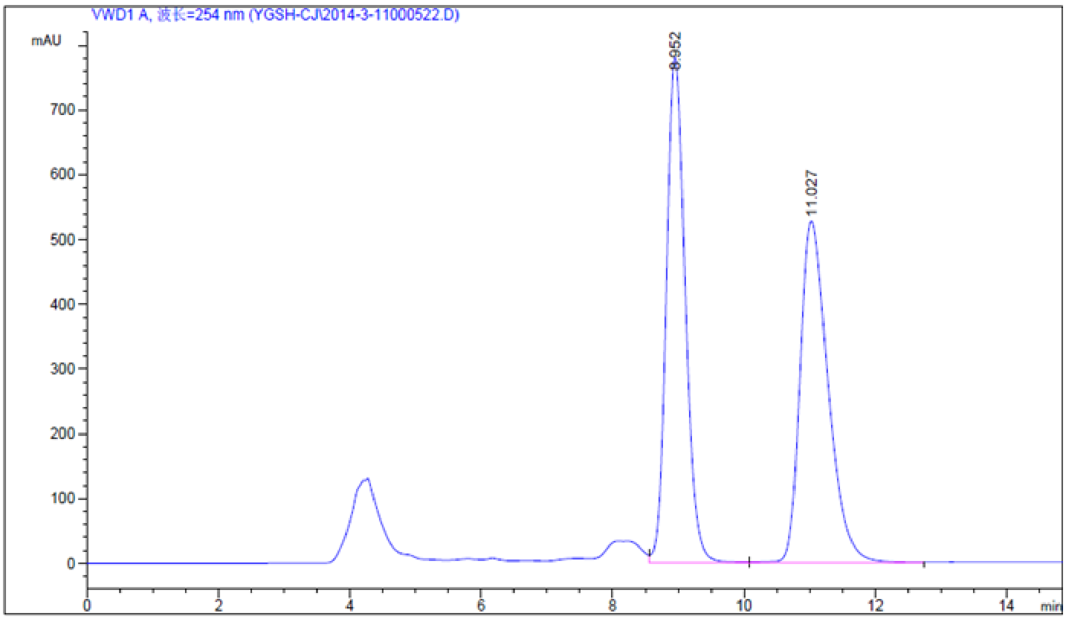
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.610	MM	0.3911	5088.59180	216.87120	70.8984
2	22.663	MM	1.0763	2088.70874	32.34435	29.1016



7c

Table 4 entry 3

AD-H, hexane/2-propanol=1:1,1mL/min



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面积百分比报告

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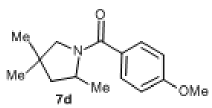
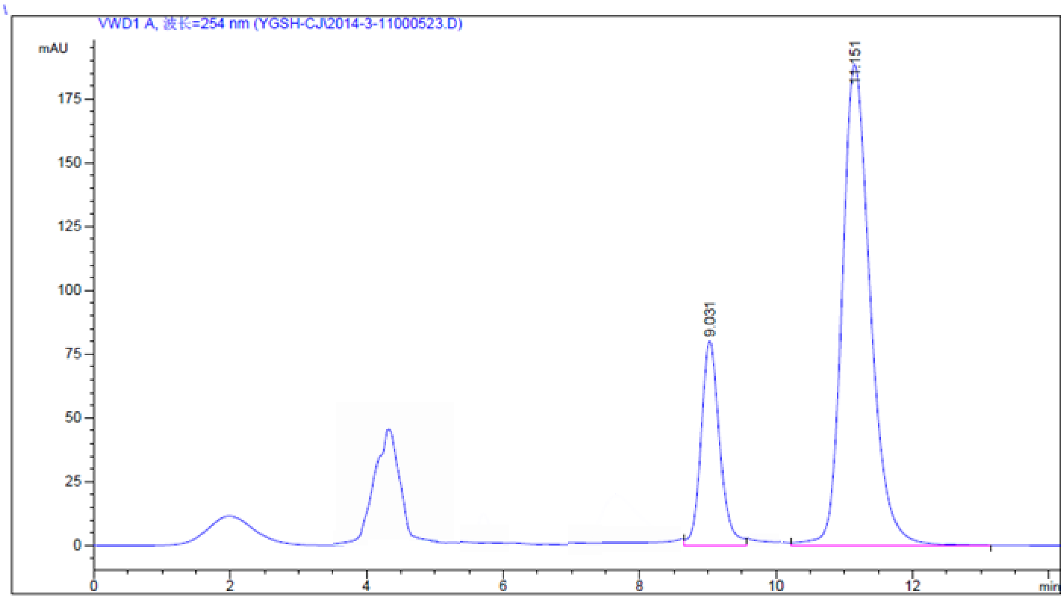
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.952	VV	0.3093	1.56110e4	779.74170	49.9260
2	11.027	VV	0.4571	1.56573e4	527.29132	50.0740



7d

Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	9.031	VV	0.2908	1515.78955	80.06336	21.8012
2	11.151	VB	0.4414	5437.00195	188.44278	78.1988

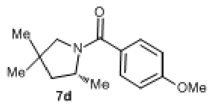
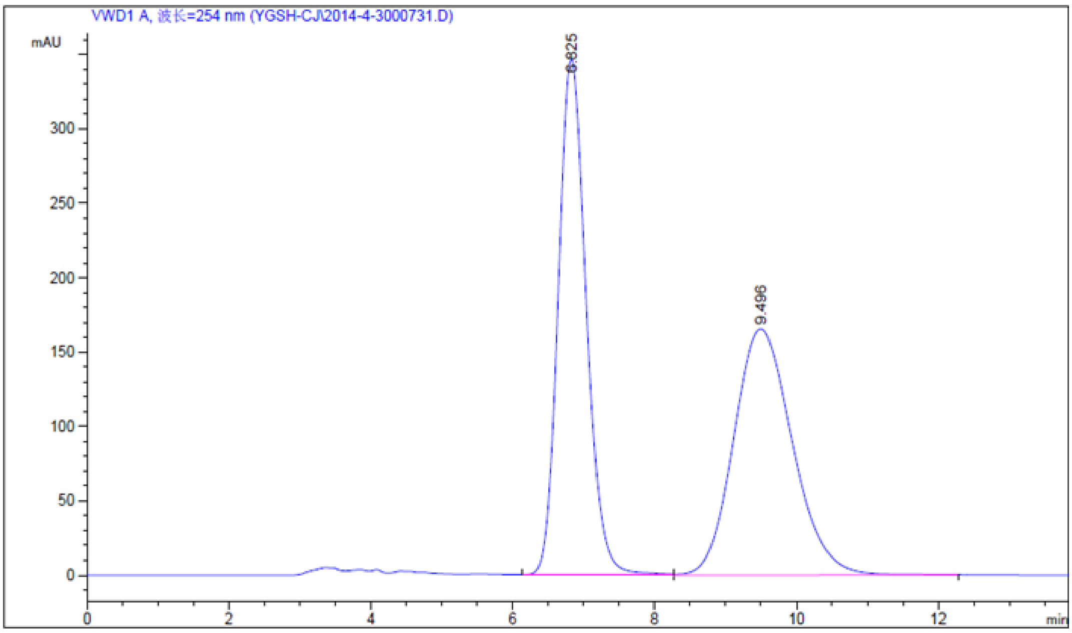


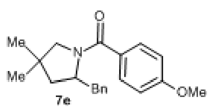
Table 3 entry 4

AD-H, hexane/2-propanol=1:1,1mL/min



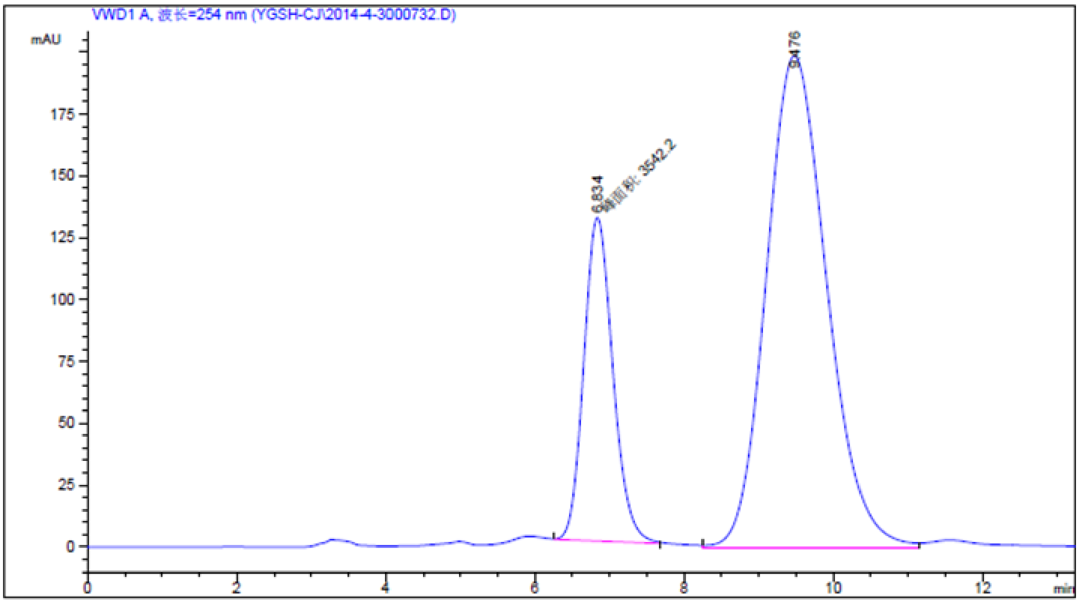
面积百分比报告

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	6.825	VV	0.4259	9541.03027	346.74686	50.0763
2	9.496	VB	0.8917	9511.93945	165.52412	49.9237



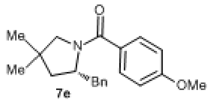
Racemic

AS-H, hexane/2-propanol=2:1,1mL/min



面积百分比报告

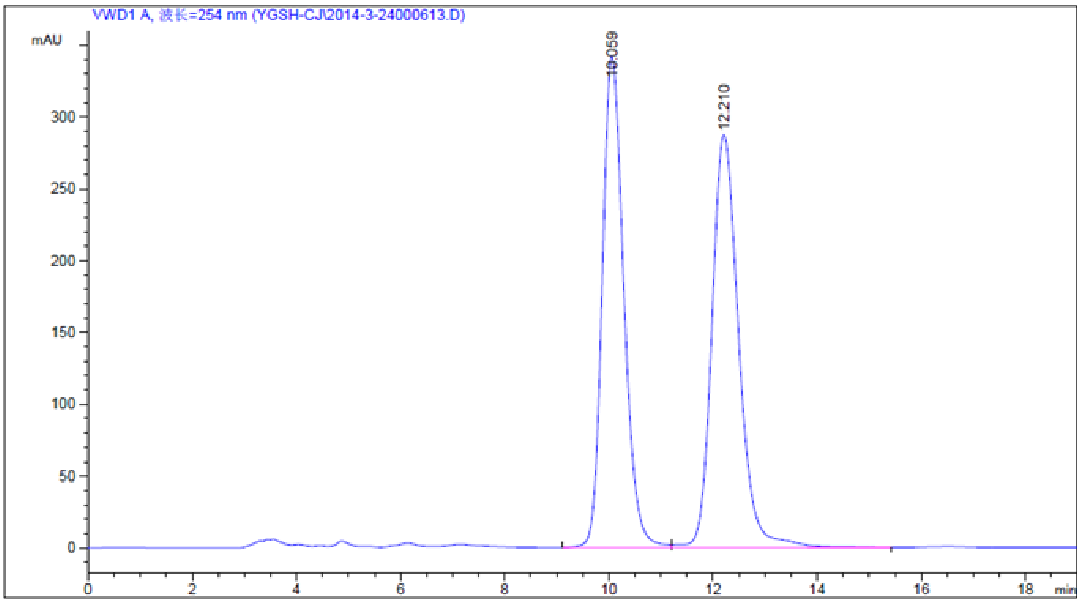
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	6.834	MM	0.4518	3542.19946	130.65689	24.0490
2	9.476	VV	0.8794	1.11869e4	198.32390	75.9510



7e

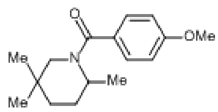
Table 3 entry 5

AS-H, hexane/2-propanol=2:1,1mL/min



面积百分比报告

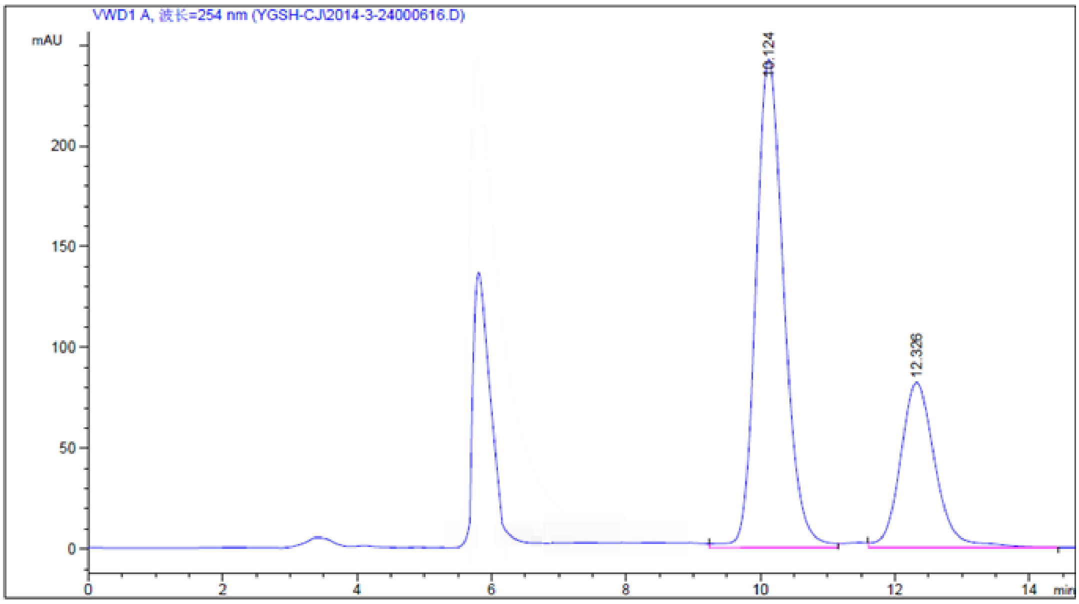
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	10.059	BV	0.4460	9827.25391	341.91306	48.9480
2	12.210	VB	0.5503	1.02497e4	287.22165	51.0520



7f

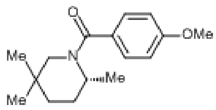
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

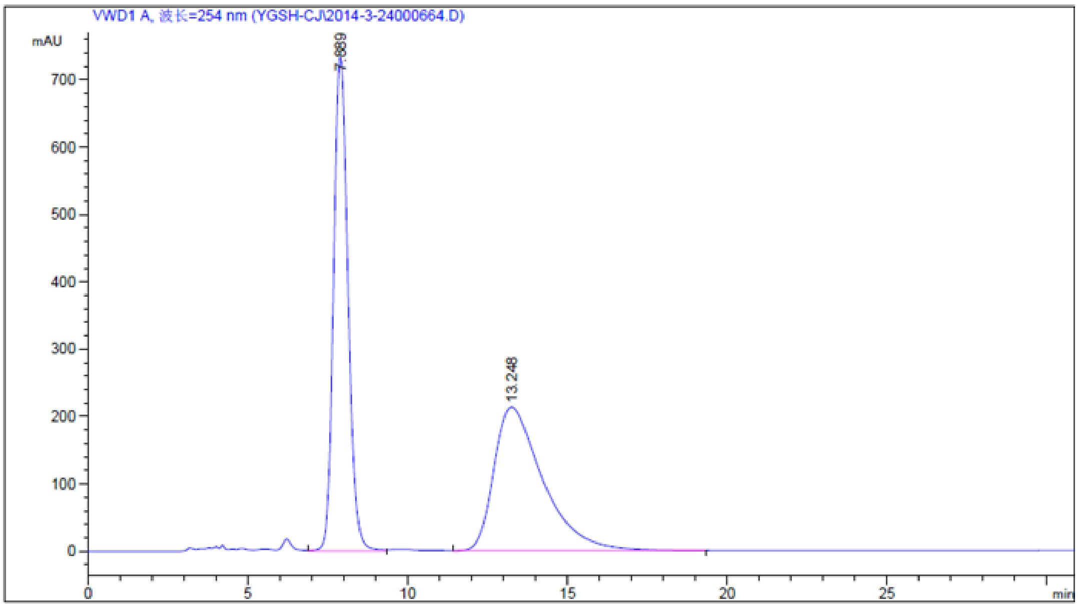
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	10.124	VV	0.4494	7030.96387	242.14676	70.5207
2	12.326	VB	0.5471	2939.11353	81.83918	29.4793



7f

Table 3 entry 6

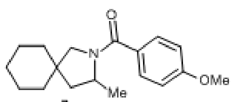
AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

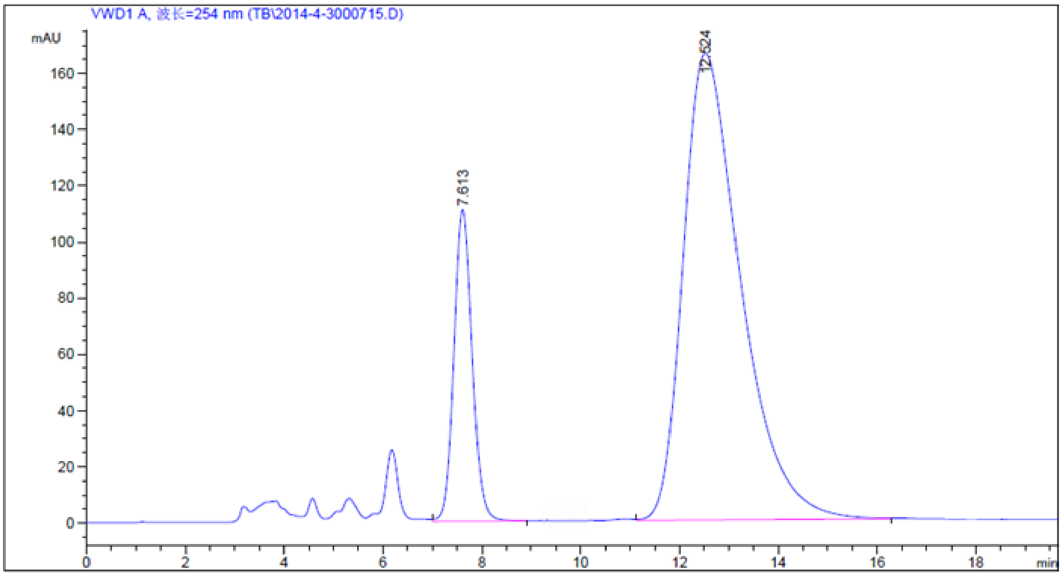
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	7.889	VV	0.4863	2.28757e4	733.12872	50.0533
2	13.248	VB	1.6042	2.28270e4	213.23659	49.9467

Racemic



7g

AS-H, hexane/2-propanol=1:1,1mL/min



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面积百分比报告
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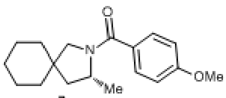
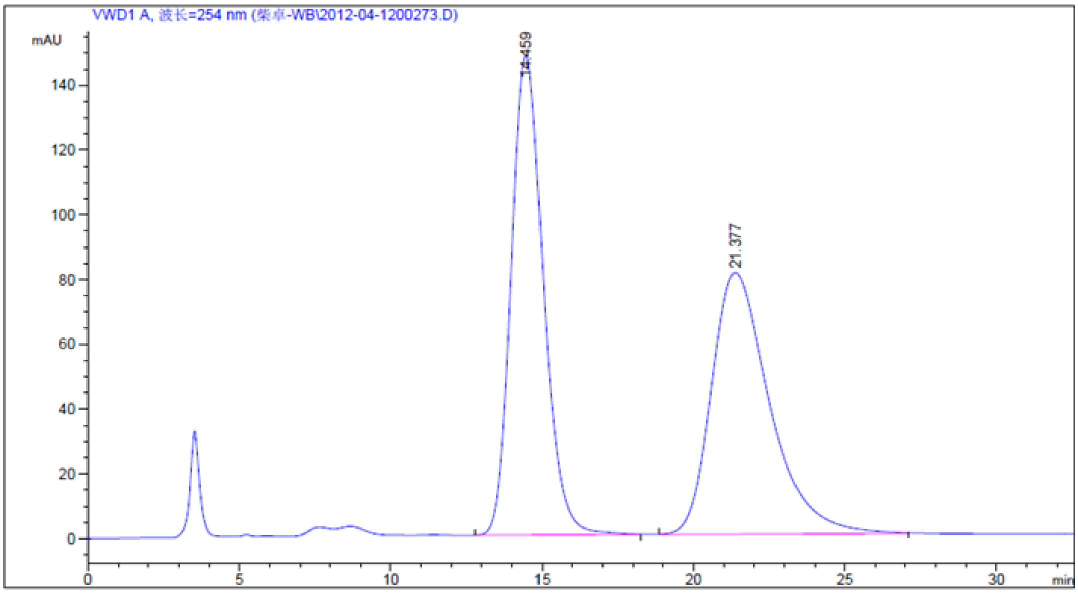
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	7.613	BB	0.3987	2844.62207	110.71951	17.4406
2	12.524	VB	1.2315	1.34657e4	166.05386	82.5594



7g

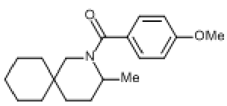
Table 3 entry 7

AS-H, hexane/2-propanol=1:1,1mL/min



==
面积百分比报告
==

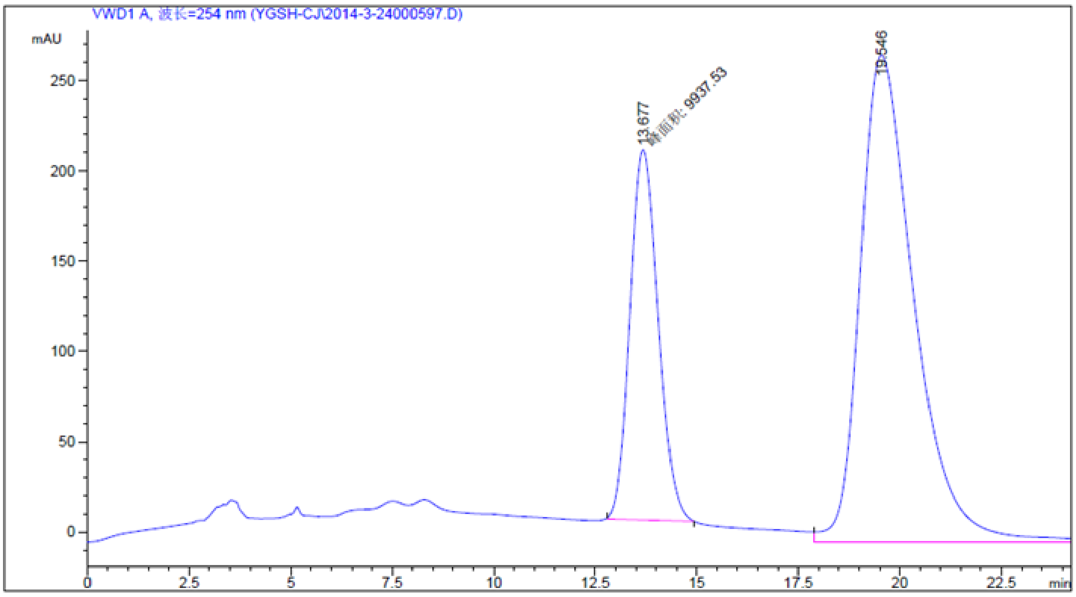
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	14.459	BB	1.1399	1.09270e4	148.16748	50.3524
2	21.377	BB	2.0271	1.07740e4	80.81483	49.6476



7h

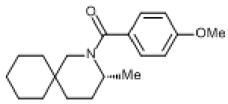
Racemic

AS-H, hexane/2-propanol=3:1,1mL/min



面积百分比报告

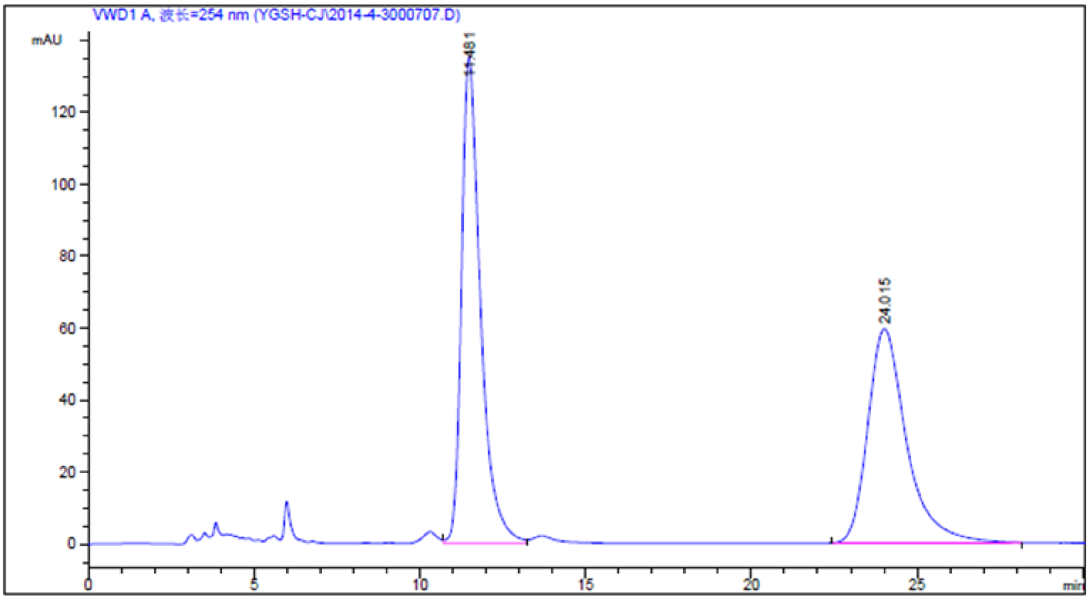
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	13.677	MM	0.8084	9937.53320	204.88022	28.5076
2	19.546	VBA	1.3988	2.49217e4	269.58353	71.4924



7h

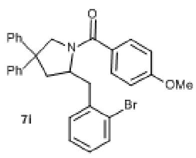
Table 3 entry 8

AS-H, hexane/2-propanol=3:1,1mL/min



面积百分比报告

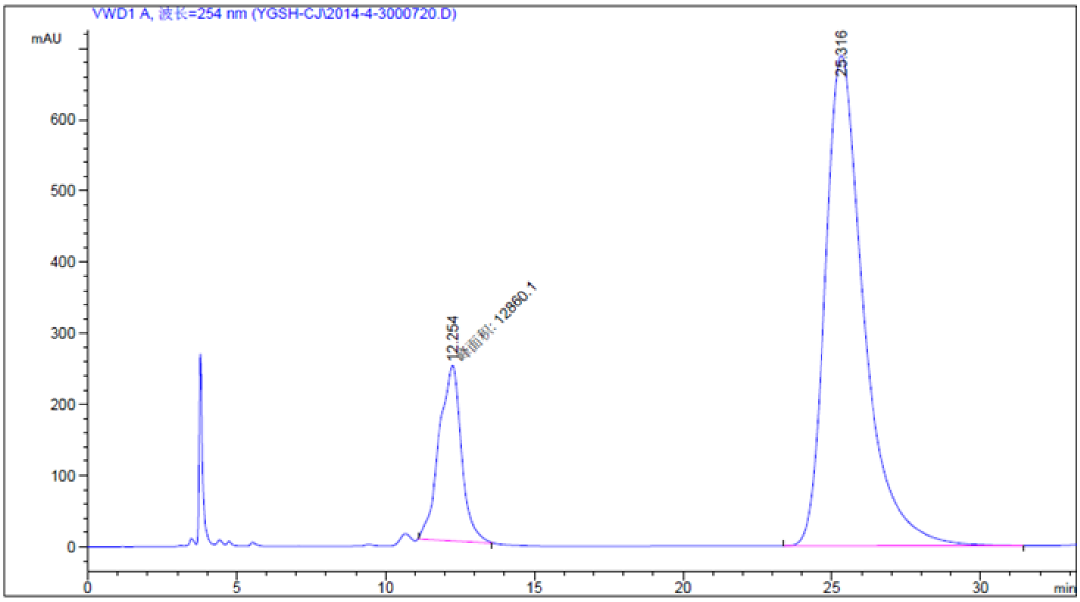
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	11.481	VV	0.5991	5395.84912	135.35028	52.7013
2	24.015	BB	1.2222	4842.69287	59.56833	47.2987



7l

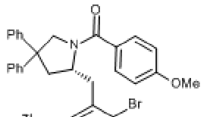
Racemic

AD-H, hexane/2-propanol=1:1, 1mL/min



面积百分比报告

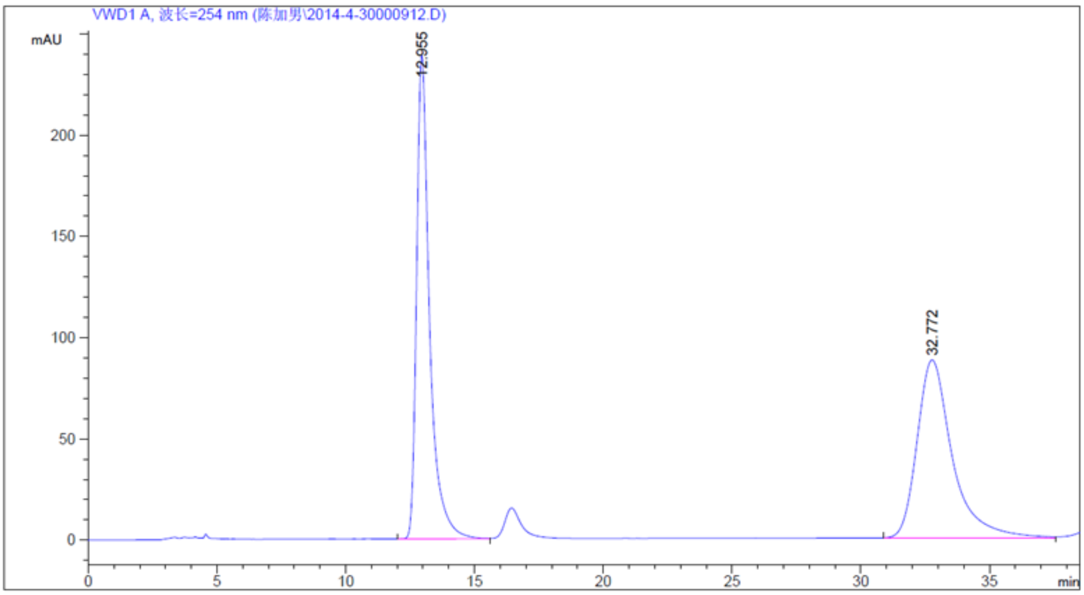
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	12.254	MM	0.8710	1.28601e4	246.09276	17.2156
2	25.316	BB	1.3499	6.18405e4	689.06354	82.7844



7l

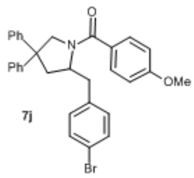
Table 4 entry 4

AD-H, hexane/2-propanol=1:1, 1mL/min



面积百分比报告

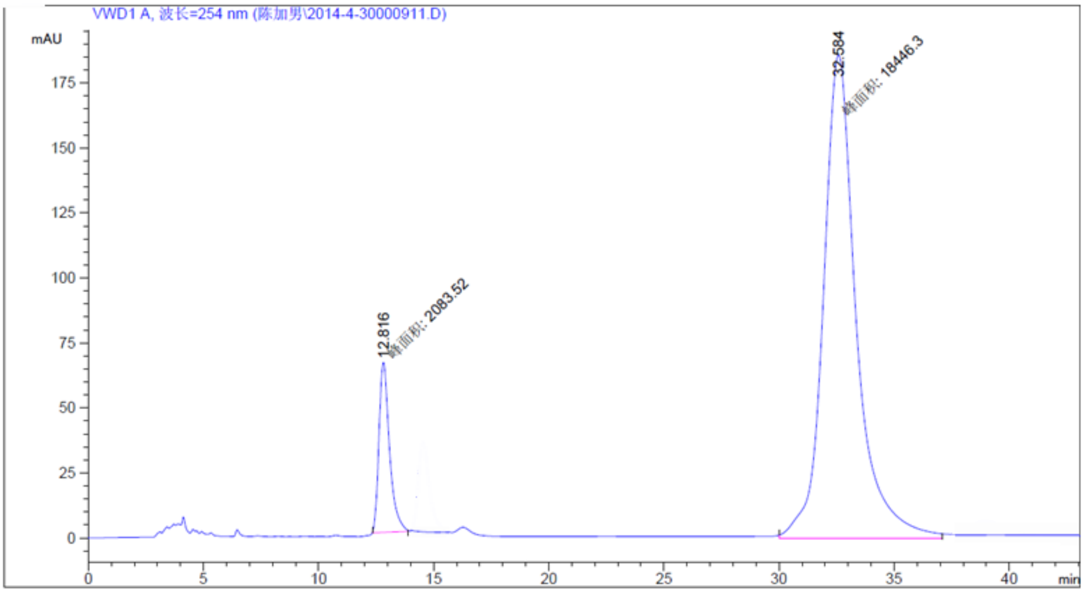
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	12.955	BV	0.5194	8358.78320	238.60399	49.9170
2	32.772	BV	1.4272	8386.58008	87.92950	50.0830



7j

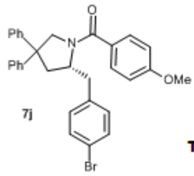
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

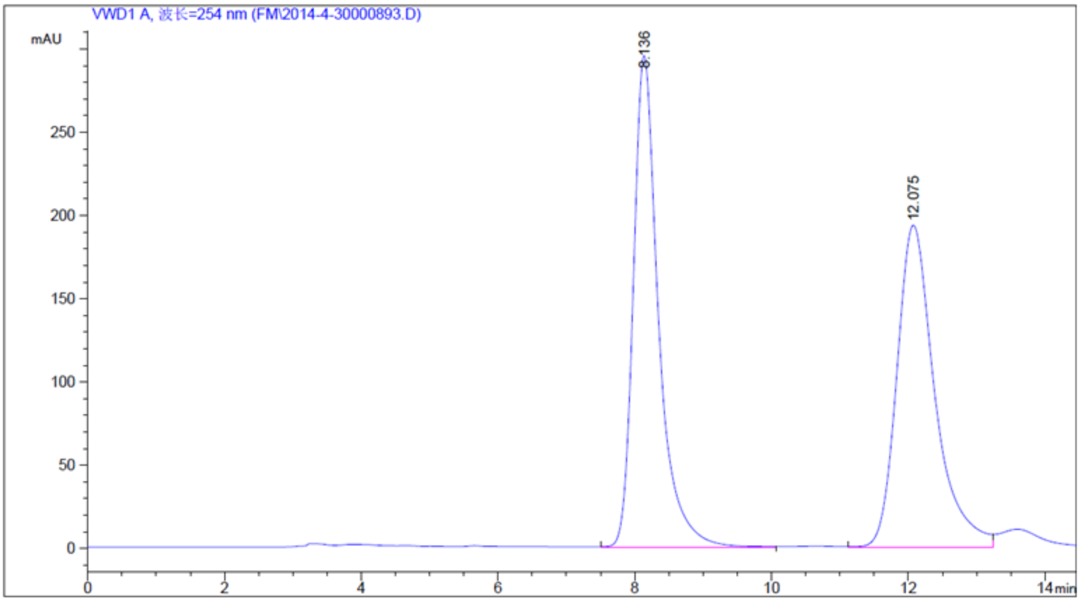
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	12.816	MM	0.5306	2083.51514	65.44341	10.1487
2	32.584	MM	1.6529	1.84463e4	185.99442	89.8513



7j

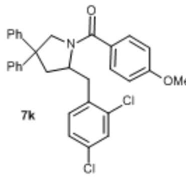
Table 4 entry 5

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

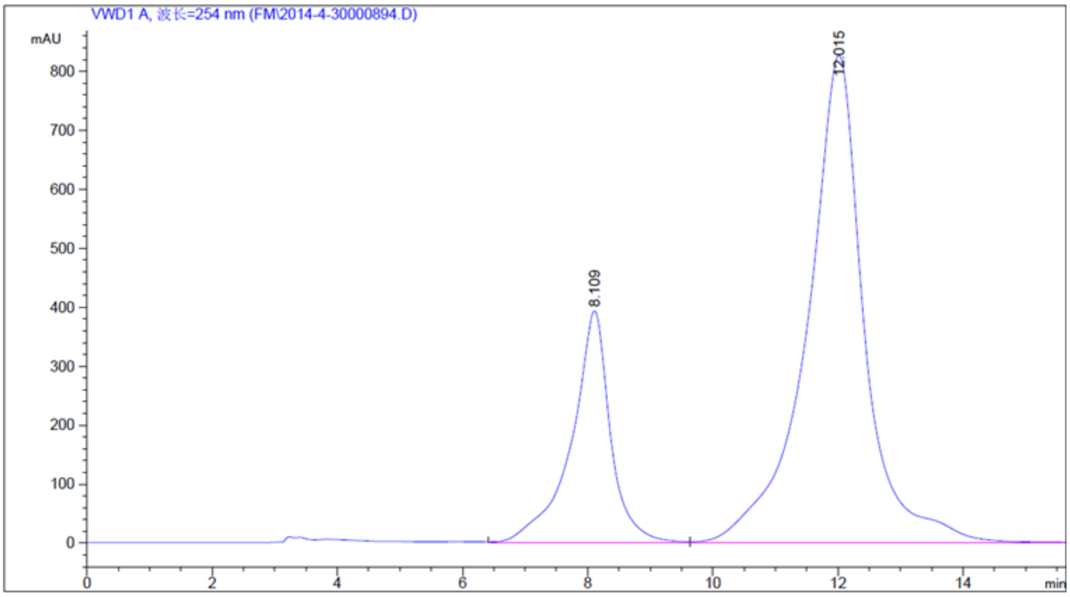
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.136	BB	0.3796	7534.80518	294.95108	50.3635
2	12.075	VV	0.5763	7426.02734	193.29860	49.6365



7k

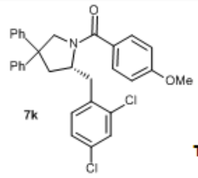
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

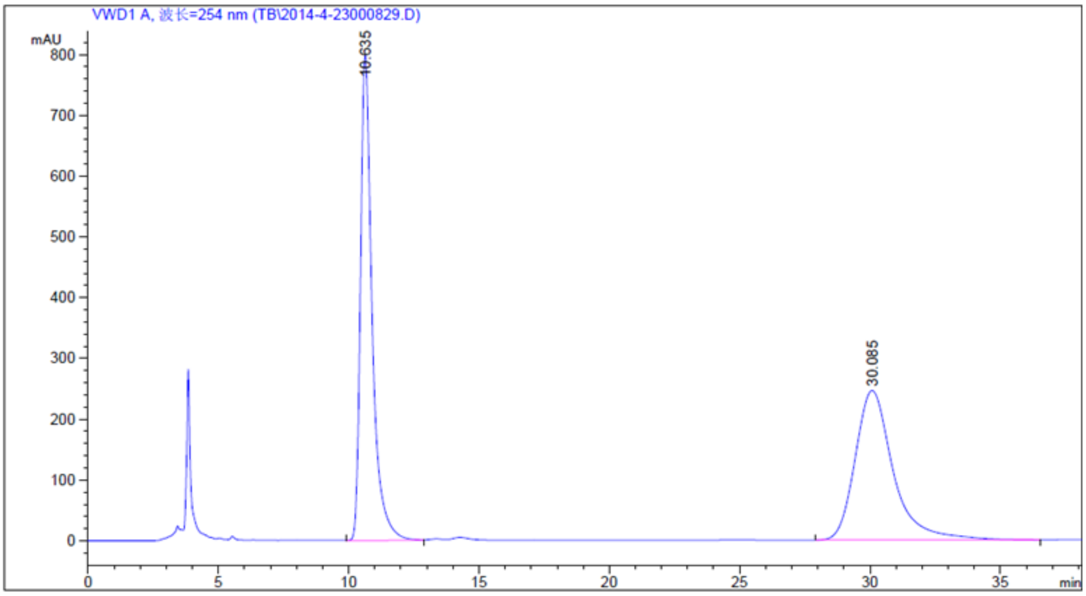
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.109	VV	0.6048	1.70274e4	392.74094	24.5038
2	12.015	VBA	0.8981	5.24617e4	826.16296	75.4962



7k

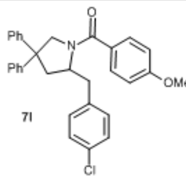
Table 4 entry 6

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

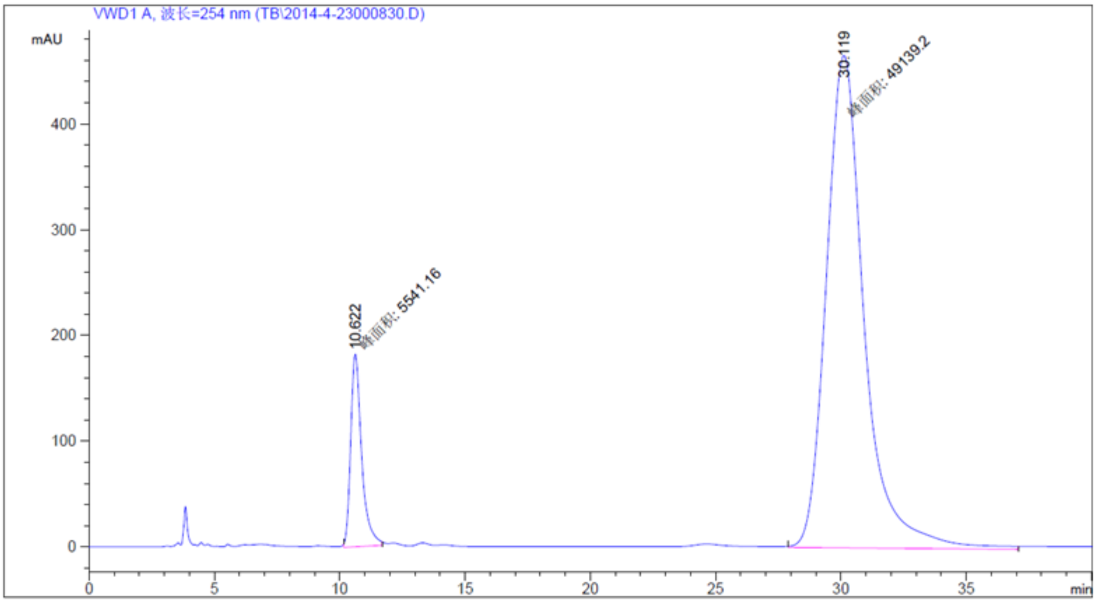
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	10.635	VV	0.4673	2.47952e4	798.13593	49.7865
2	30.085	BB	1.5410	2.50079e4	246.04849	50.2135



7l

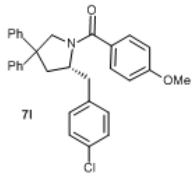
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

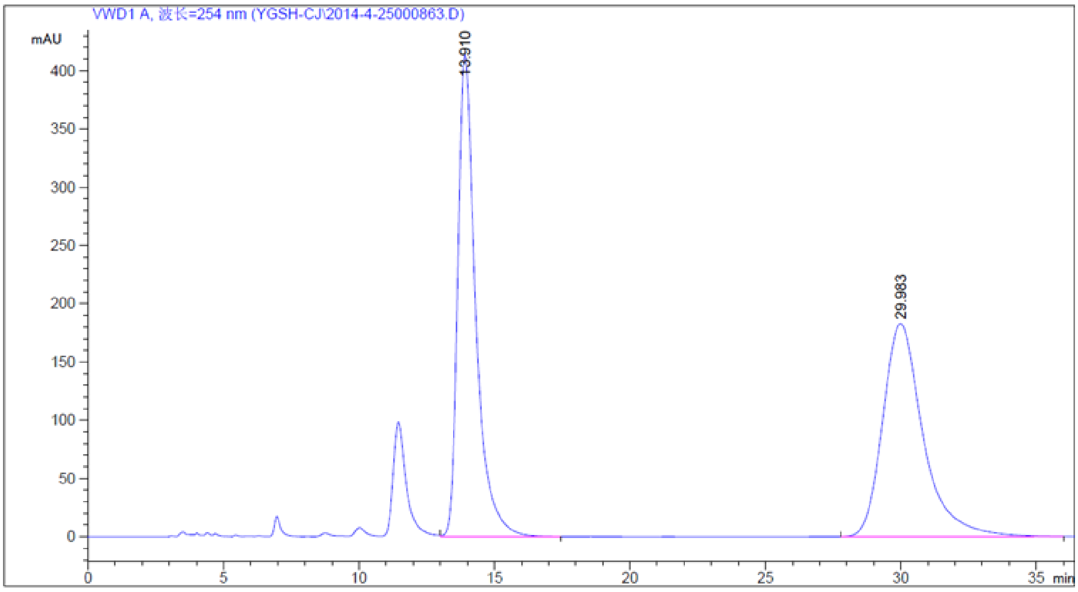
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	10.622	MM	0.5068	5541.16016	182.22890	10.1337
2	30.119	MM	1.7585	4.91392e4	465.74265	89.8663



7l

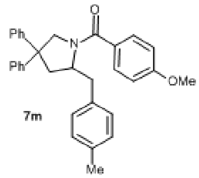
Table 4 entry 7

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

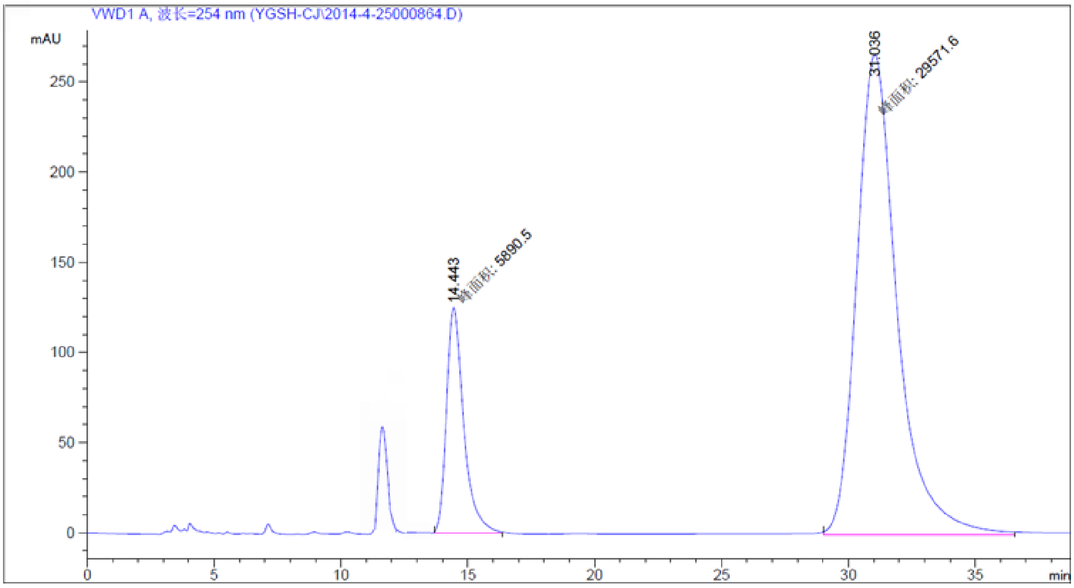
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	13.910	VB	0.6878	1.90110e4	413.49207	50.0967
2	29.983	BB	1.5644	1.89377e4	182.73145	49.9033



7m

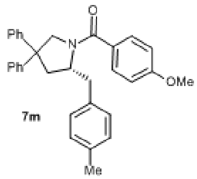
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

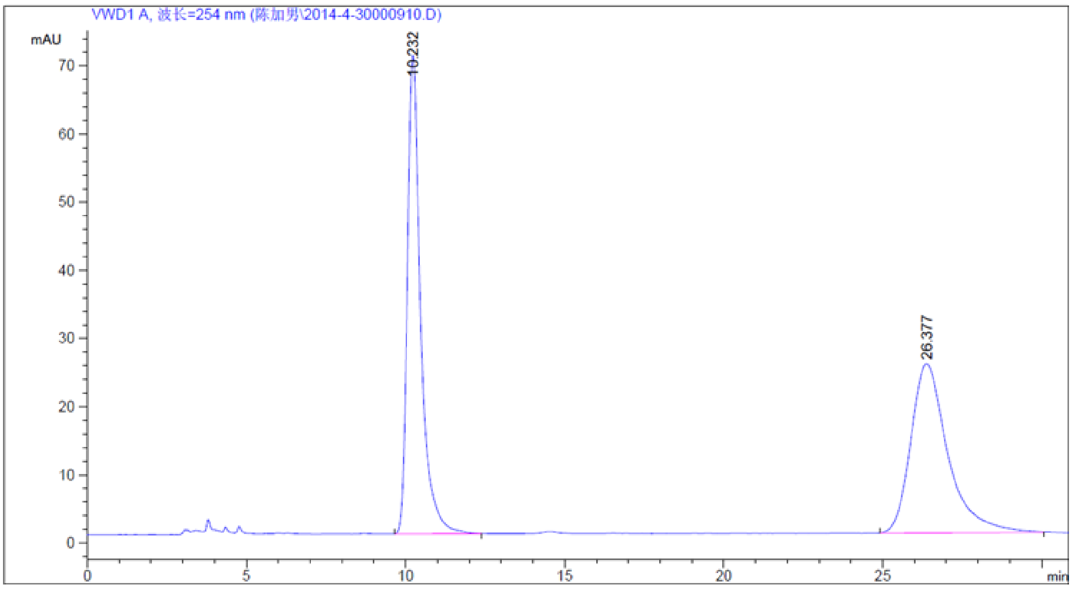
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	14.443	MM	0.7843	5890.50342	125.17408	16.6107
2	31.036	MM	1.8510	2.95716e4	266.26874	83.3893



7m

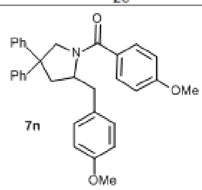
Table 4 entry 8

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

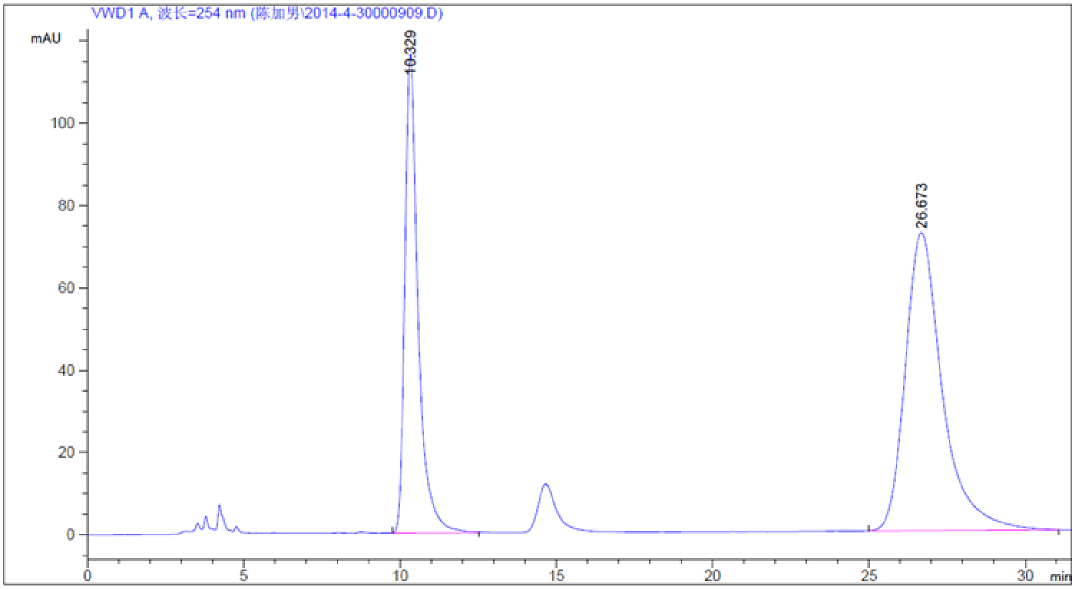
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	10.232	BB	0.4311	2034.22327	70.24006	50.2995
2	26.377	BB	1.2138	2009.99963	24.79114	49.7005



7n

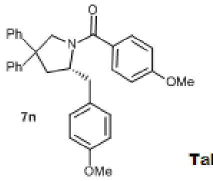
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



==
面积百分比报告
==

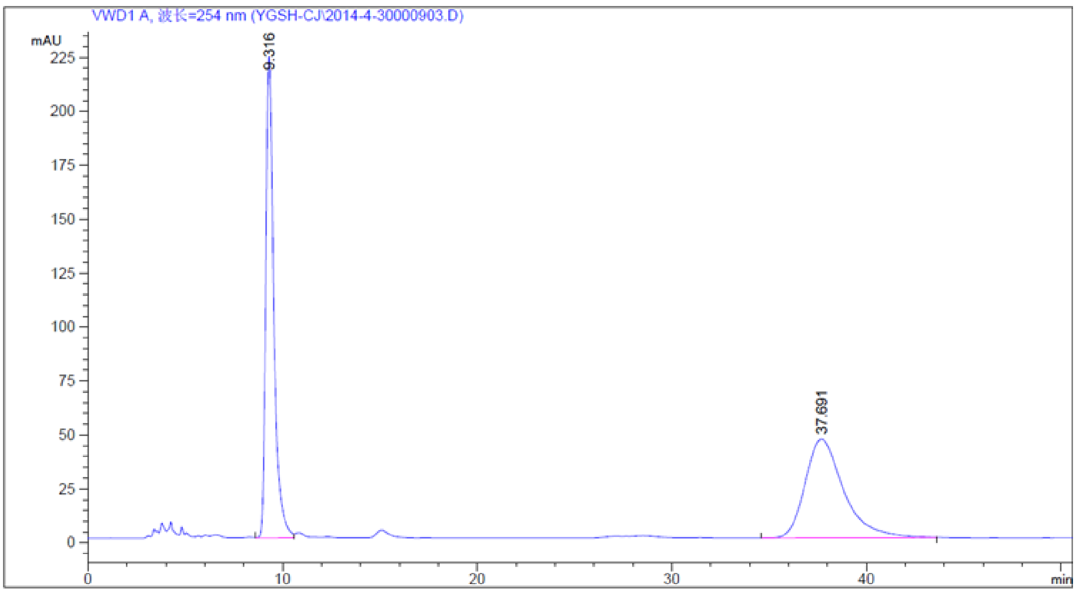
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	10.329	BB	0.4362	3418.42261	116.25488	36.4438
2	26.673	BB	1.2410	5961.56836	72.34367	63.5562



7n

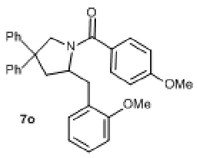
Table 4 entry 9

AD-H, hexane/2-propanol=1:1,1mL/min



==
面积百分比报告
==

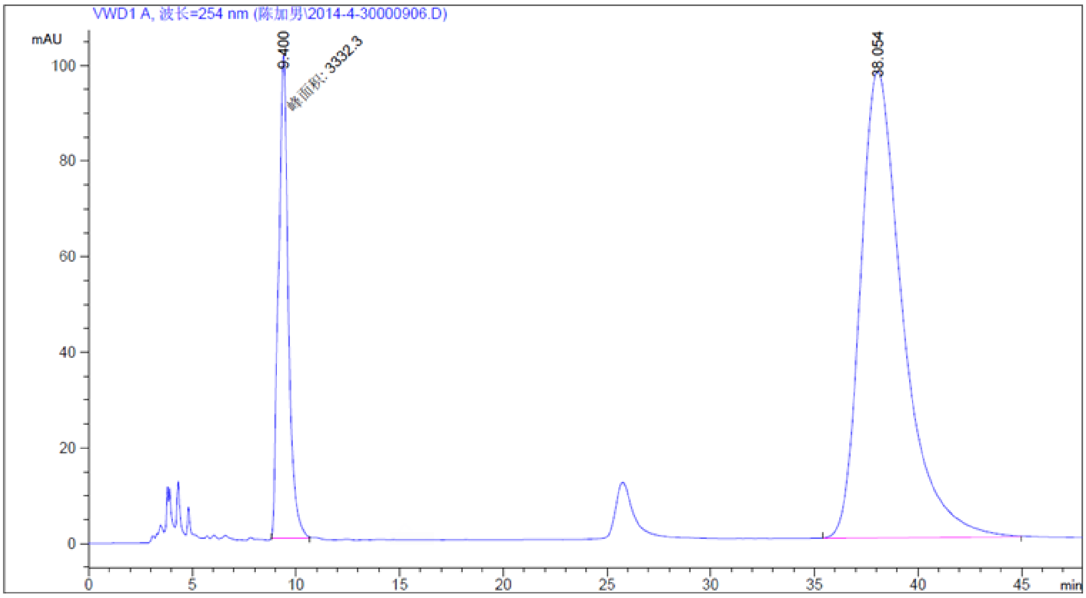
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	9.316	VV	0.4258	6360.93262	223.08519	50.1442
2	37.691	BB	2.0837	6324.35352	45.77358	49.8558



7o

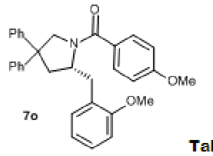
Racemic

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

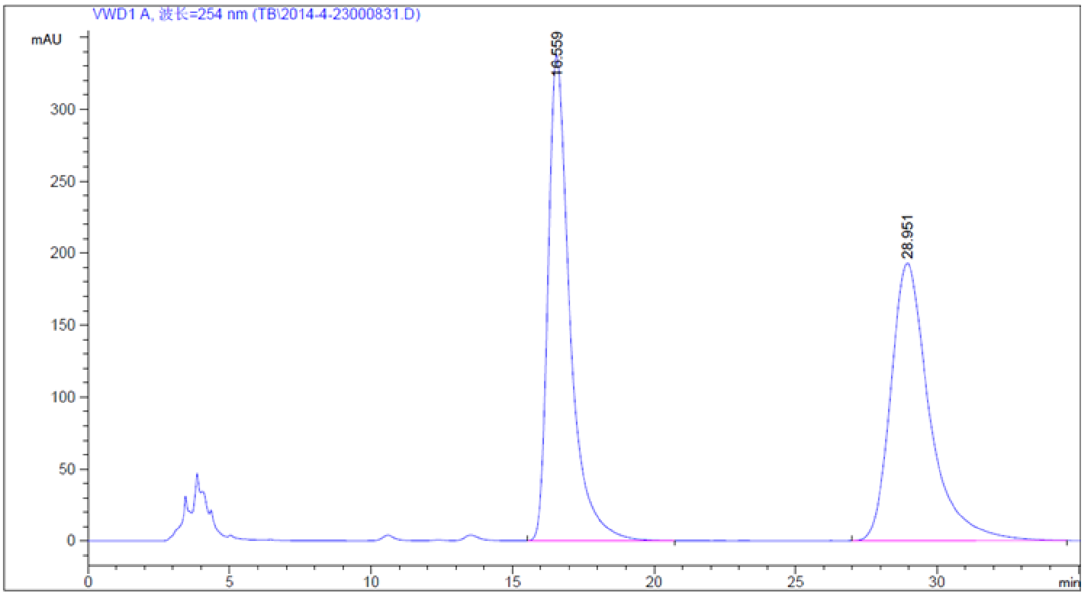
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	9.400	MM	0.5499	3332.30444	100.99450	19.4159
2	38.054	BB	2.1423	1.38304e4	97.60650	80.5841



7o

Table 4 entry 10

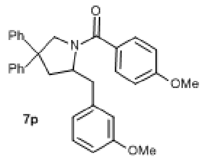
AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

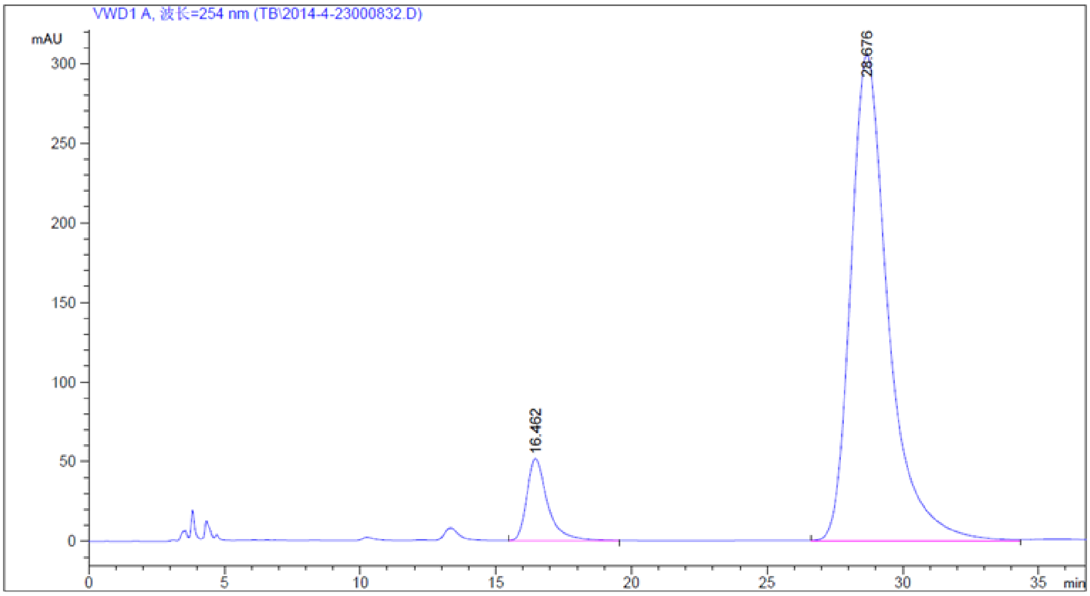
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	16.559	BB	0.7997	1.80694e4	337.33194	49.7383
2	28.951	BB	1.4258	1.82595e4	192.69733	50.2617

Racemic



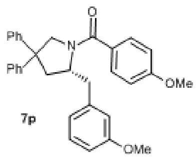
7p

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

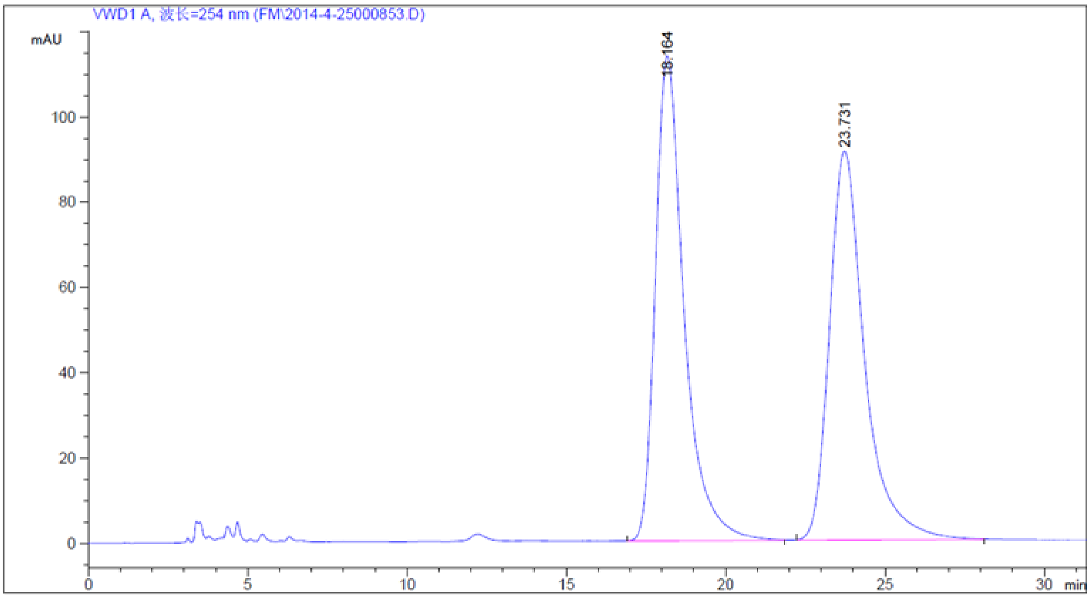
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	16.462	BB	0.7975	2761.26758	51.72498	8.6298
2	28.676	BB	1.4388	2.92357e4	304.94705	91.3702



7p

Table 4 entry 11

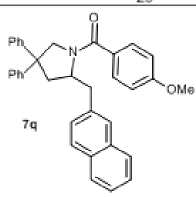
AD-H, hexane/2-propanol=1:1,1mL/min



Racemic

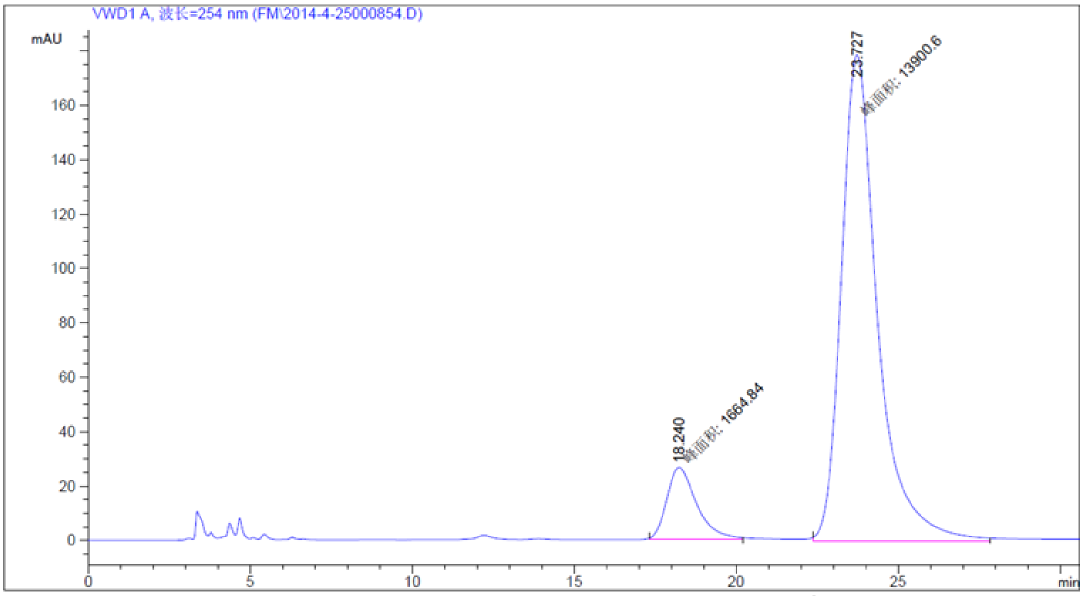
面积百分比报告

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	18.164	BB	0.9346	7123.69043	113.72739	50.4220
2	23.731	BB	1.1529	7004.43945	91.14078	49.5780



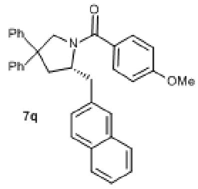
7q

AD-H, hexane/2-propanol=1:1,1mL/min



面积百分比报告

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	18.240	MM	1.0552	1664.83801	26.29528	10.6957
2	23.727	MM	1.2963	1.39006e4	178.72612	89.3043



7q

Table 4 entry 12

AD-H, hexane/2-propanol=1:1,1mL/min

^{1}H NMR monitoring of reactions in Table 4

Table 4, Entry 1

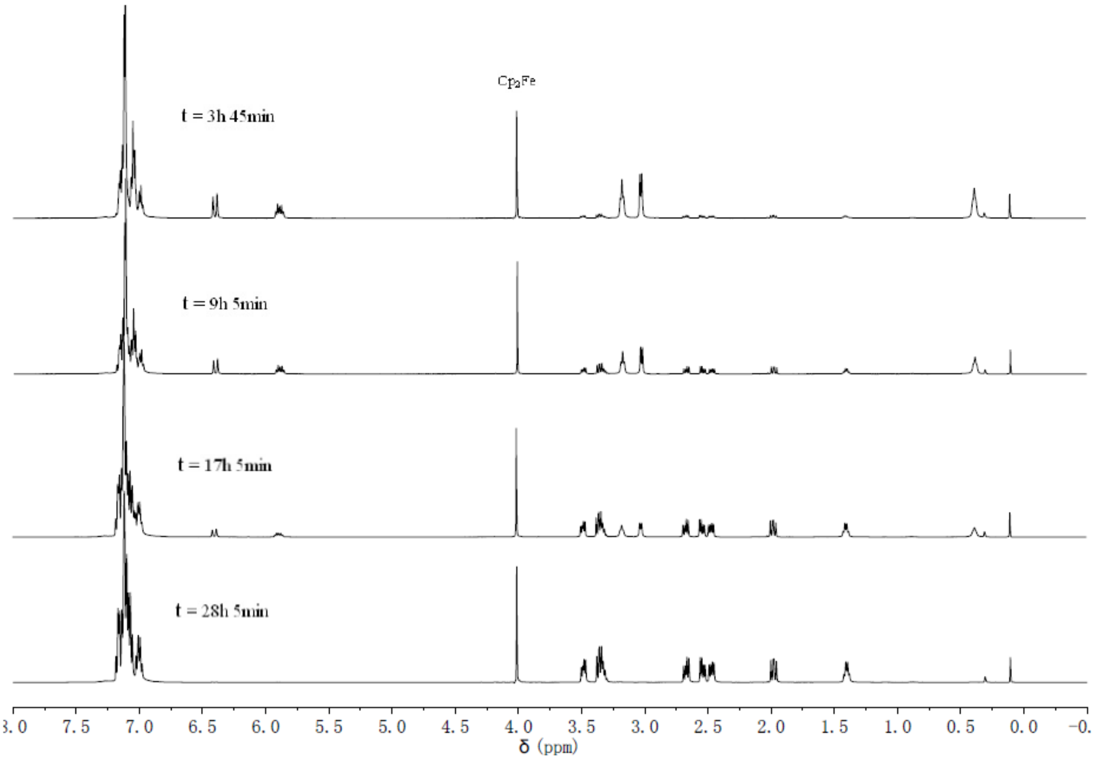


Table 4, Entry 2

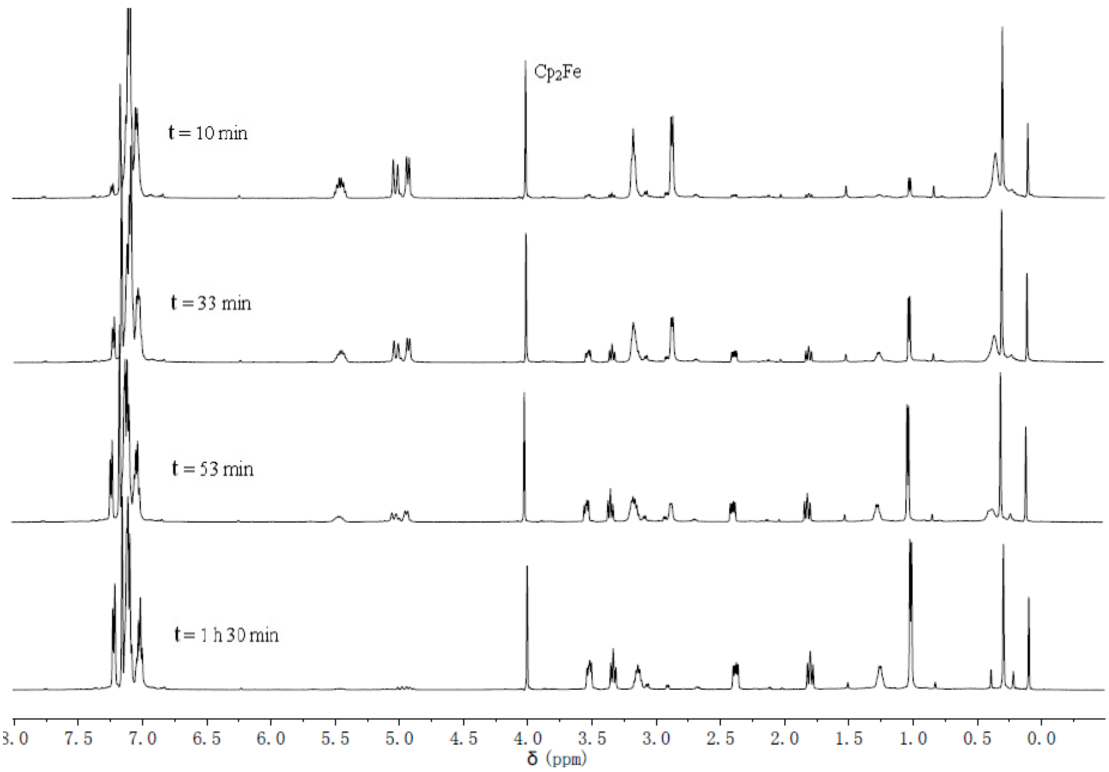


Table 4, Entry 3

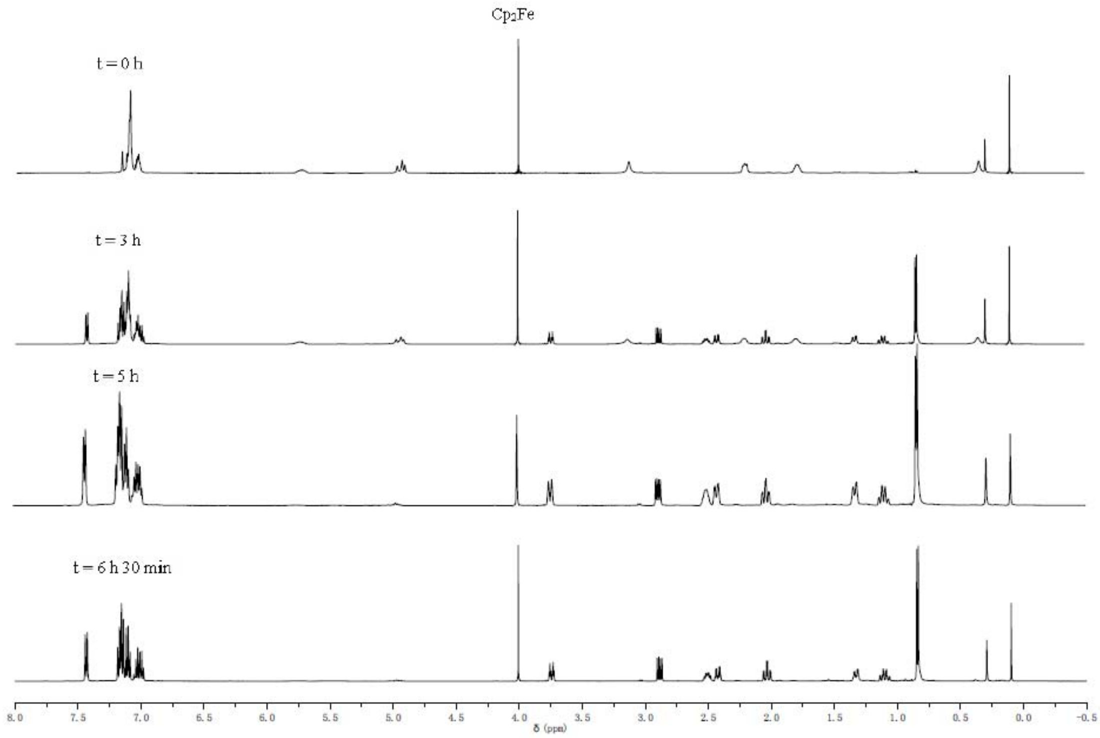


Table 4, Entry 4

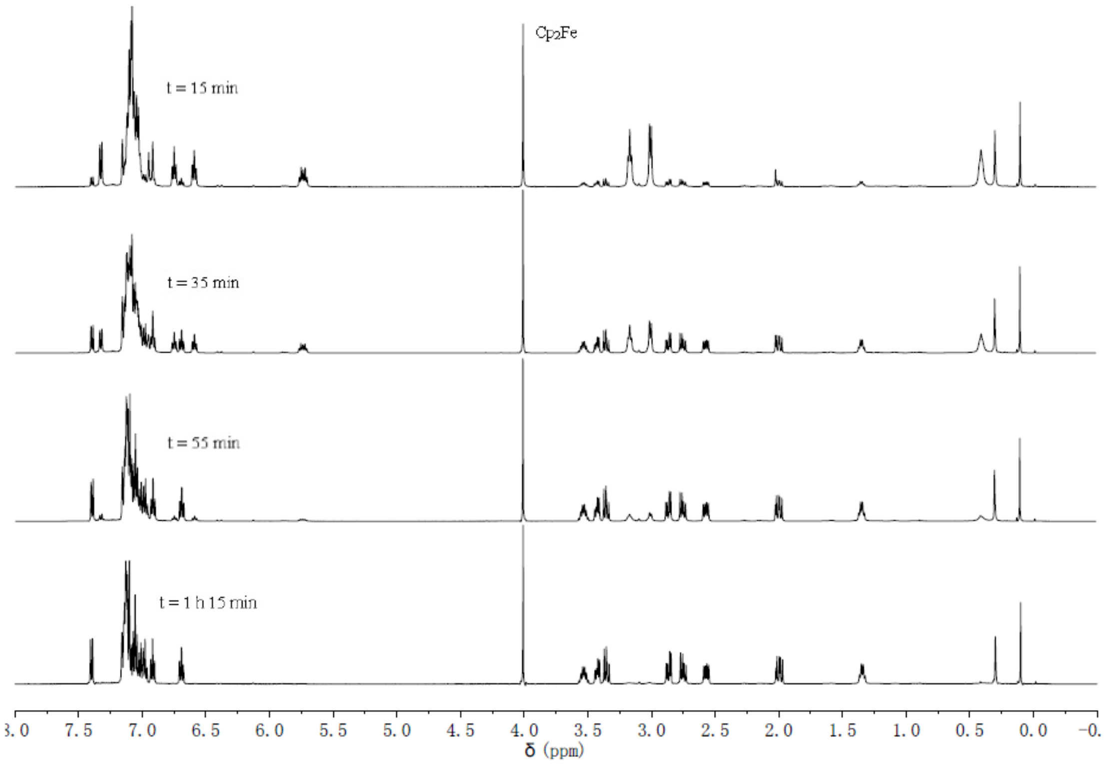


Table 4, Entry 5

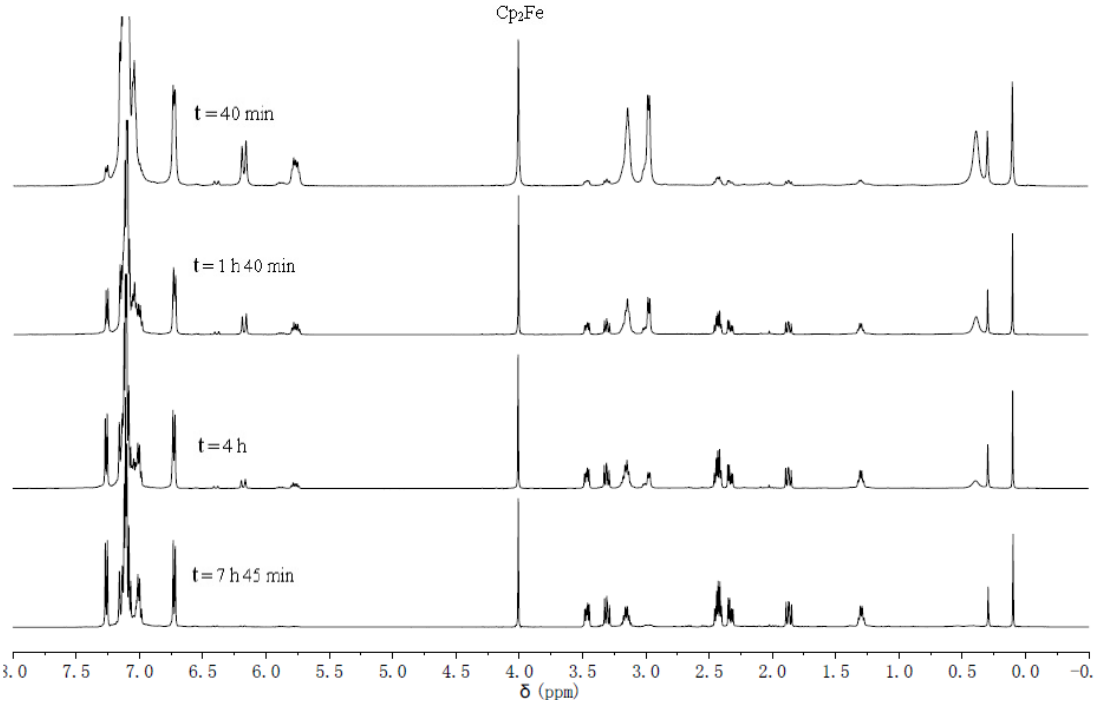


Table 4, Entry 6

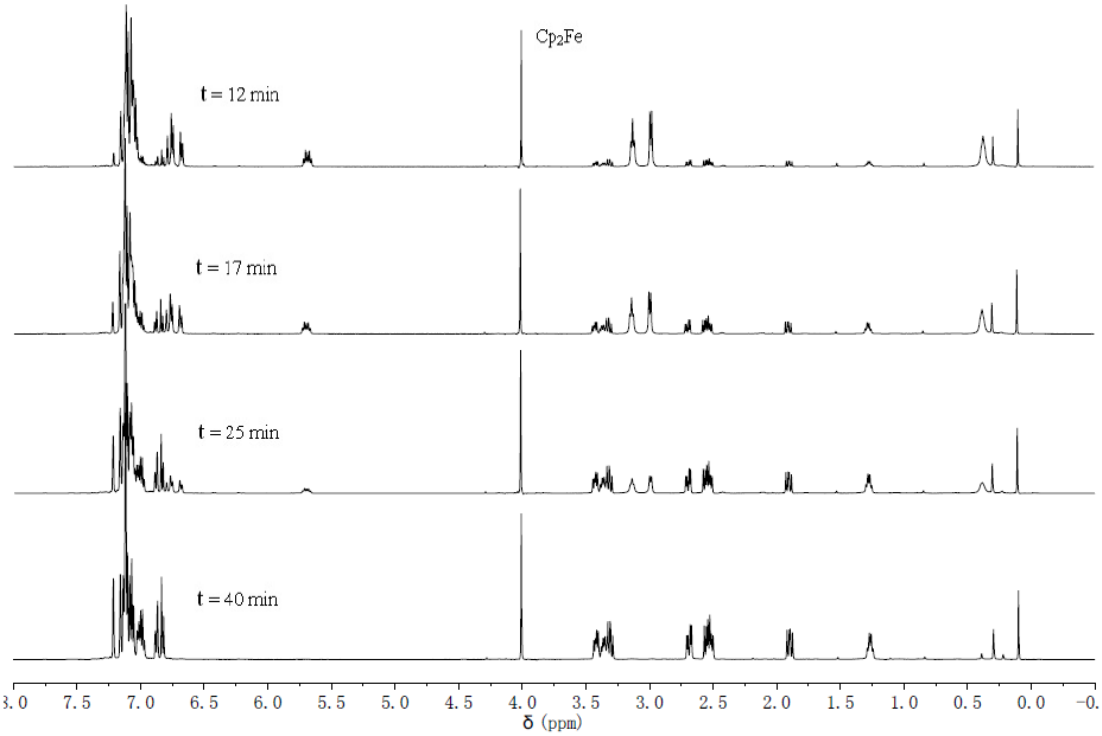
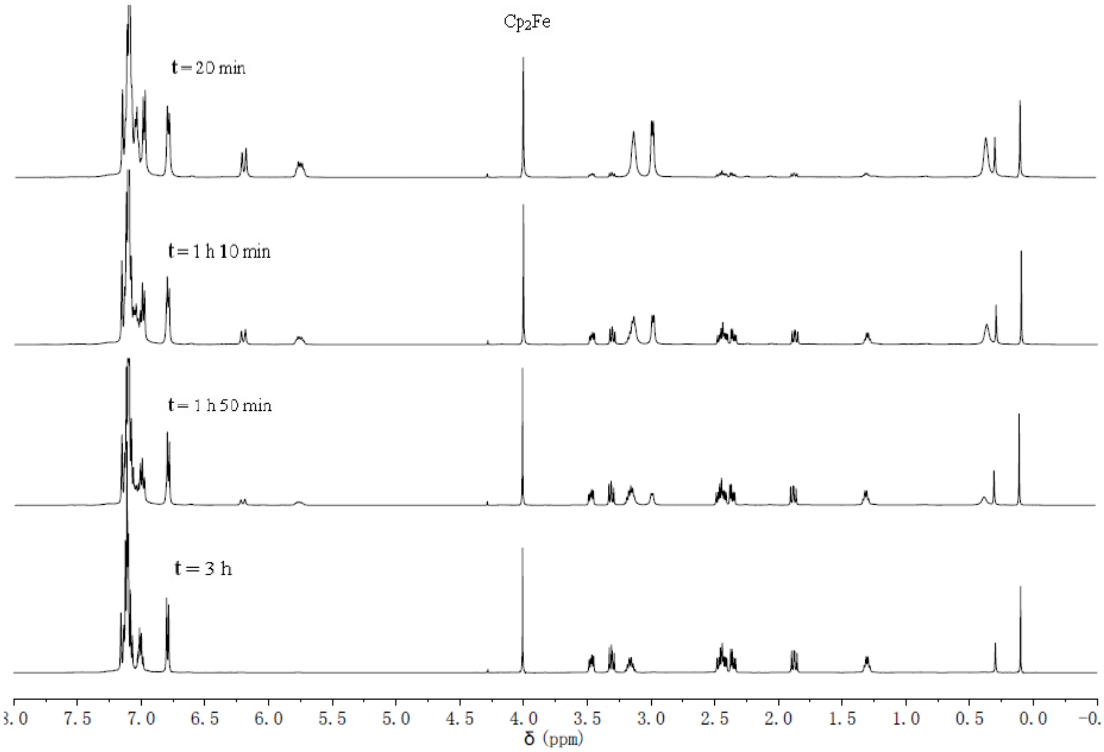


Table 4, Entry 7



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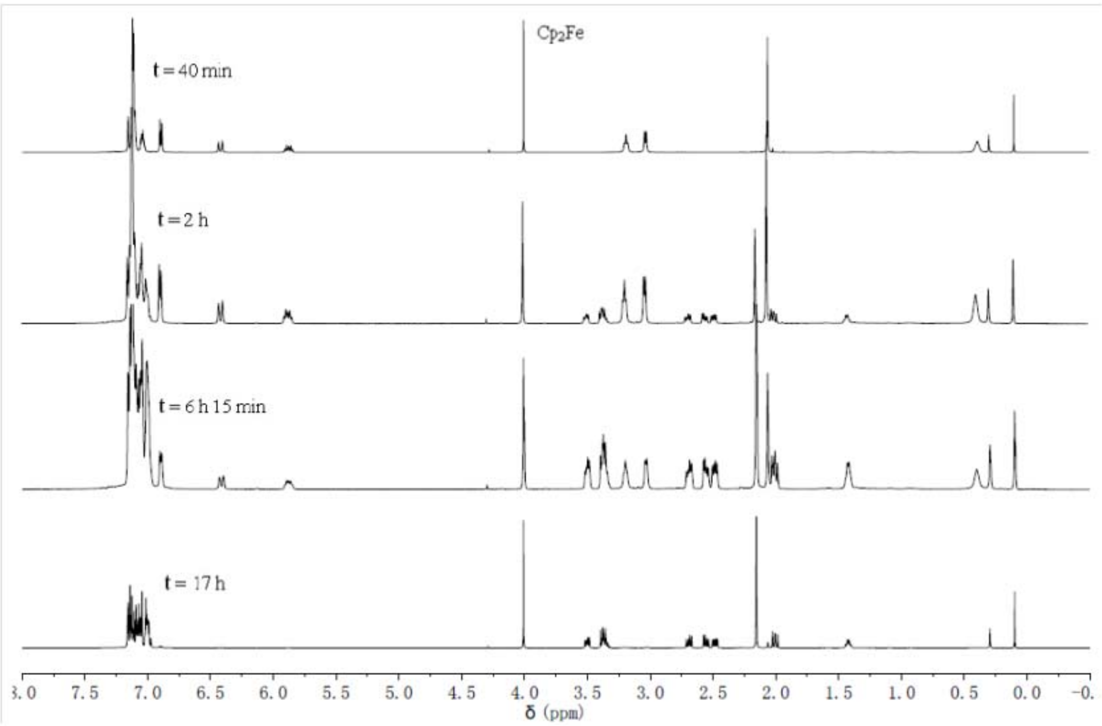

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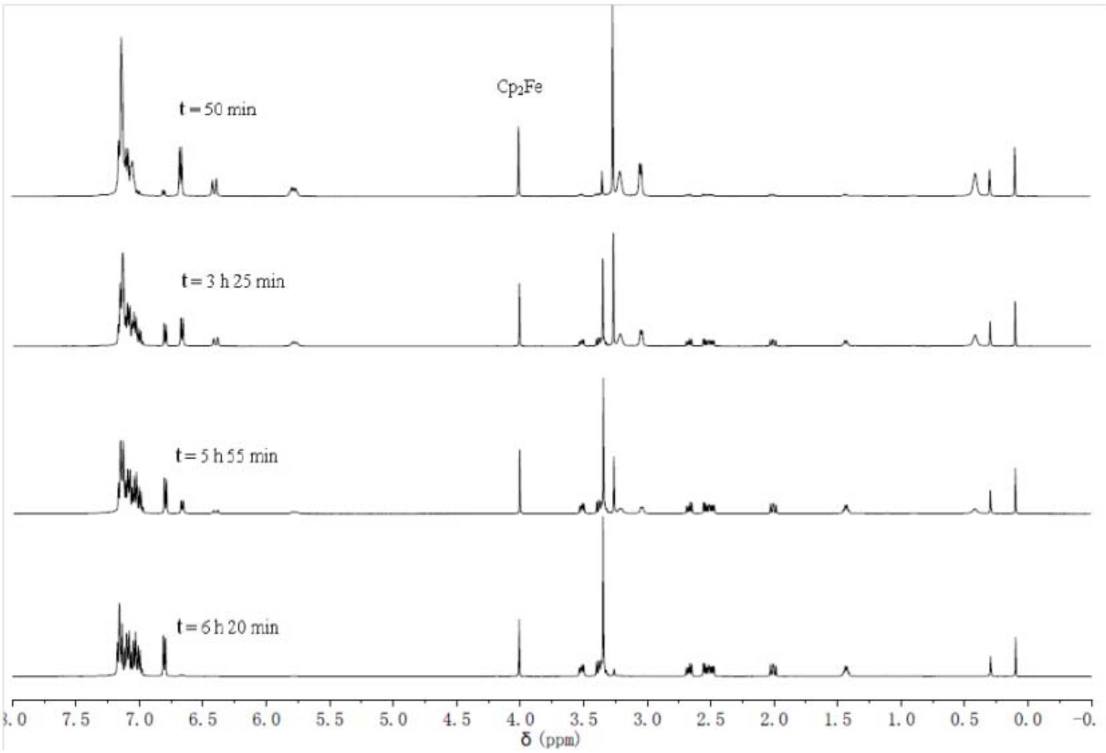

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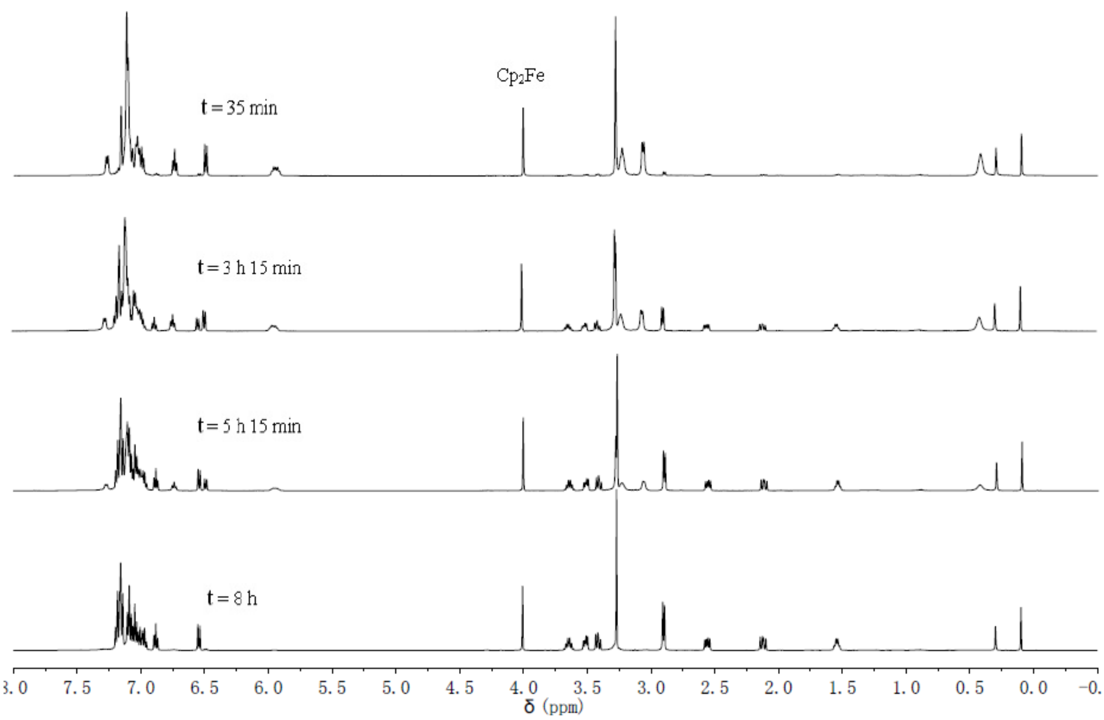

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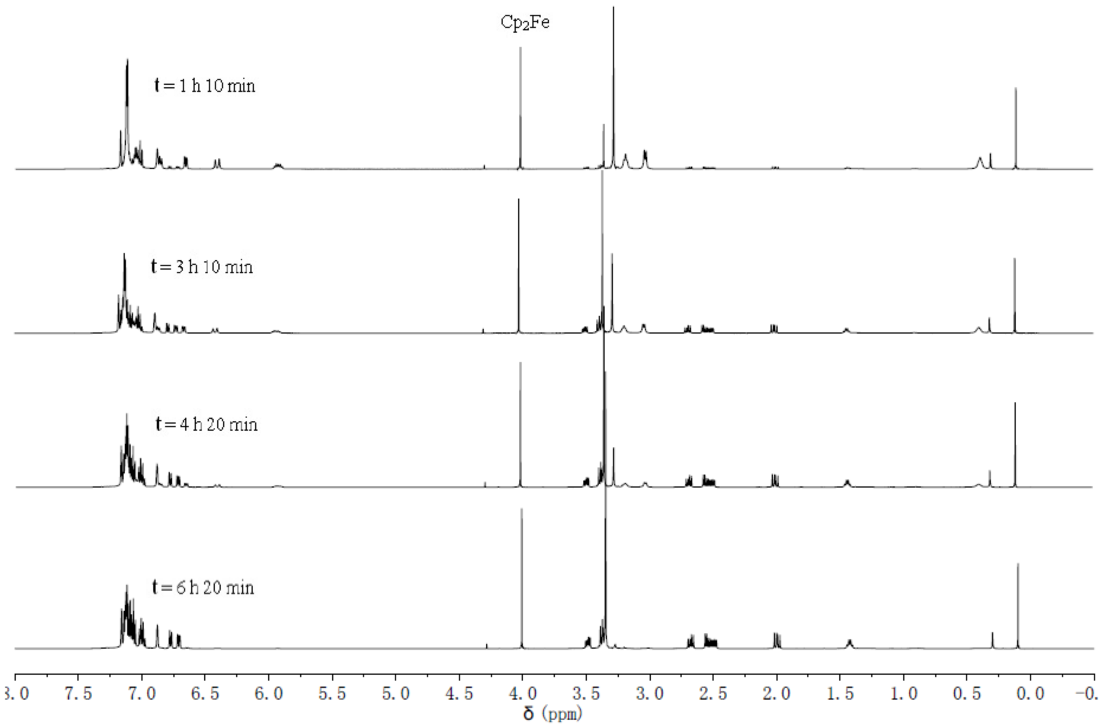

Table 4, Entry 12