

Supporting Information

Electrochemical regioselective reduction of α -keto amide with methanol as hydrogen source

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1. General information

Chemicals were purchased from commercial sources and were used as received unless mentioned otherwise. α -keto amides **1a-44a** were prepared from literature reports.¹⁻² All reactions were monitored by TLC, which used a UV lamp (254 nm) as a visualizing agent. All electrochemical reactions were carried out in an undivided cell equipped with Pt plate (10 mm $\times$ 15 mm) and graphite rod (ϕ 8 mm). ^1H and ^{13}C NMR spectra were measured on a Bruker spectrometer (AVIII 400 MHz). CDCl_3 (7.26 ppm in ^1H NMR; 77.16 ppm in ^{13}C NMR) or $\text{DMSO}-d_6$ (2.50 ppm in ^1H NMR; 39.52 ppm in ^{13}C NMR) served as the internal standard for ^1H NMR and ^{13}C NMR. HRMS (ESI) were recorded on a liquid chromatography-mass spectrometry-ion trap-time of flight (LCMS-IT-TOF) (Shimadzu, Japan); MS (ESI) were recorded on a Bruker ESQUIRELCTM ESI ion trap spectrometer. Cyclic voltammograms were obtained on a CHI 605D electrochemical workstation (Shanghai CH Instruments, Inc.) in an undivided cell equipped with a glassy carbon CHI 104 as the working electrode (ϕ 3 mm), and Pt plate (10 mm $\times$ 30 mm) and Ag/AgCl (KCl (sat)) as the counter and reference electrodes at a scan rate of 100 mV/s.

2. Experimental procedure

General procedure for the synthesis of α -keto acids¹

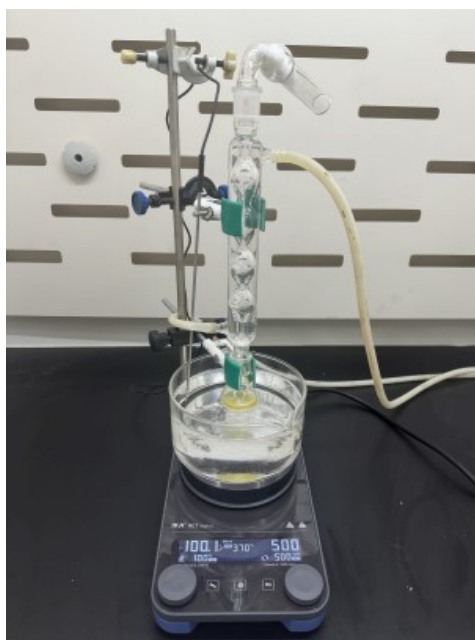
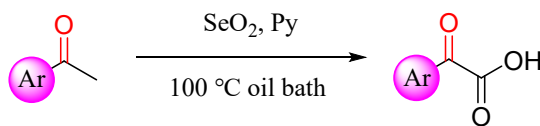


Figure S1. Reaction device.

An oven-dried 25 mL reaction flask equipped with a magnetic stirring bar was charged with appropriate acetophenone (10 mmol, 1 equiv.), selenium dioxide (20 mmol, 2 equiv.) and pyridine (70 mmol, 7 equiv.). The reaction mixture was stirred at 100 °C using oil bath and the progress of the reaction was monitored by TLC using petroleum ether and ethyl acetate as an eluent. After disappearance of the acetophenone on TLC, reaction was stopped and filtered with EtOAc. To this 11N HCl was added and organic compound was extracted with EtOAc (3 x 25 mL). Organic layers were washed with brine and with fresh water. The organic layers were dried on Na₂SO₄ and evaporated to give α -keto acids, which were used directly in the next step without further purification.

General procedure for the synthesis of α -keto amides (1a-44a)²

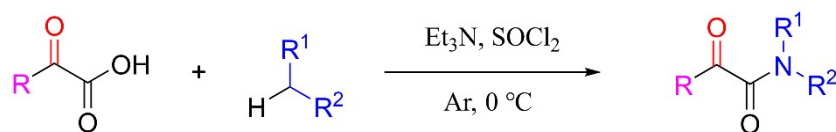


Figure S2. Reaction device.

Under the protection of an argon atmosphere, an oven-dried 100 mL reaction flask equipped with a magnetic stirring bar was charged with appropriate α -keto acid (10 mmol) and DCM (40 mL) and kept at 0-5 $^{\circ}\text{C}$, to this Et_3N (20 mmol, 2 equiv.) was added and with a gap of 5 min thionyl chloride (20 mmol, 2 equiv.) was added in dropwise. Later, appropriate aniline/amine (10 mmol, 1 equiv.) was added in portions (if aniline is liquid, dissolved in DCM and added in dropwise). Now, allow the reaction mixture to room temperature and continue to stir. The progress of the reaction was monitored by TLC using petroleum ether and ethyl acetate as an eluent. After completion of reaction, the excess SOCl_2 was removed under vacuum and the organic compound is extracted with ethyl acetate (3 x 100 mL). The organic layers were dried on Na_2SO_4 and evaporated to give α -keto amides, which were purified by column chromatography.

References

1. K. Wadhwa, C. Yang, P. R. West, K. C. Deming, S. R. Chemburkar and R. E. Reddy, *Synth. Commun.*, 2008, **38**, 4434-4444.
2. N. C. Mamillapalli and G. Sekar, *Chem. Commun.*, 2014, **50**, 7881-7884.

General procedure for the synthesis of α -hydroxy amides (1b-44b)

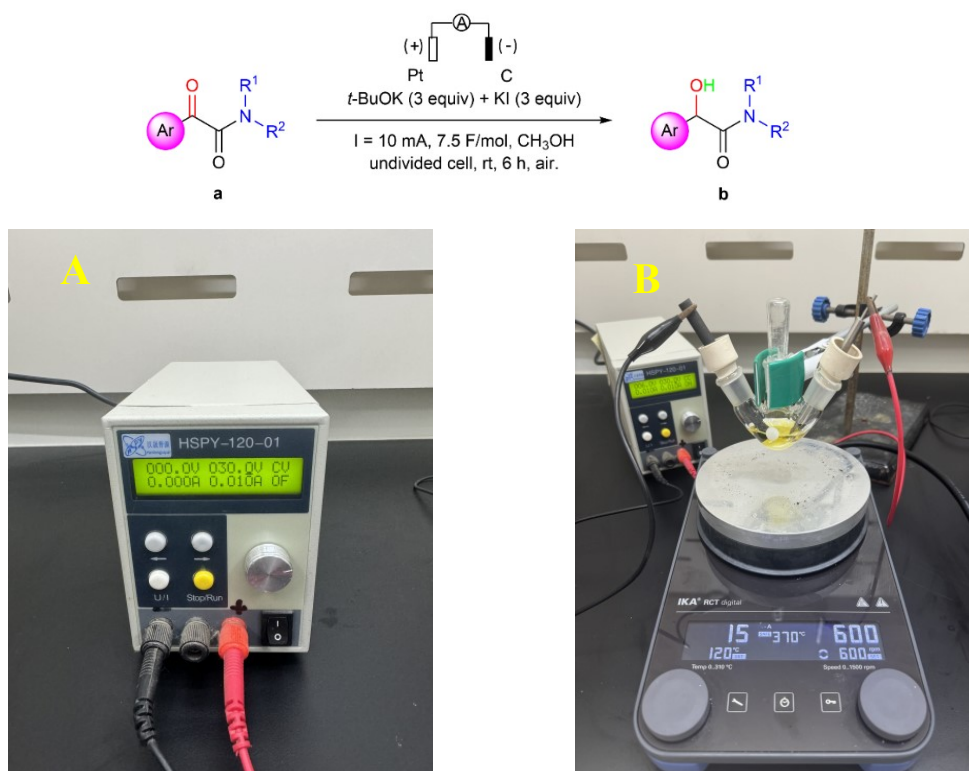
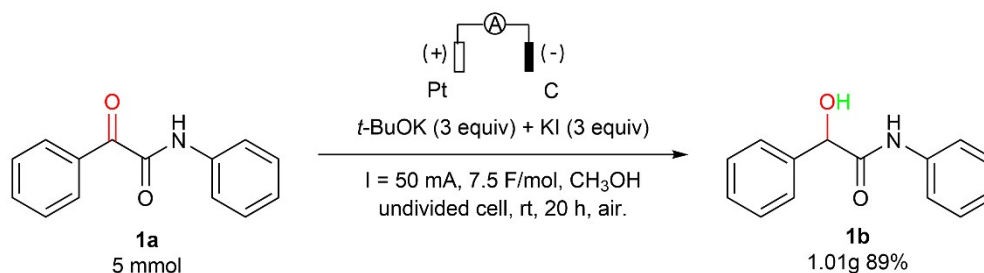


Figure S3. A) Programmable DC power supply. B) Reaction device.

An oven-dried 10 mL undivided three-necked flask was equipped with a magnetic stirrer, Pt plate (10 mm $\times$ 15 mm) as anode and graphite rod (ϕ 8 mm) as cathode (Figure S3 B). Substrates **a** (0.3 mmol, 1 equiv.), *t*-BuOK (0.9 mmol, 3 equiv.), KI (0.9 mmol, 3 equiv.), and MeOH (4 mL) were stirred and electrolyzed at a constant current (10 mA) at room temperature for 6 h (7.5 F/mol). The reaction was monitored by thin-layer chromatography. At the end of the reaction, the reaction solution was evaporated to remove methanol, and then purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain the product **b**.

3. Gram-Scale synthesis



An oven-dried 100 mL undivided three-necked flask was equipped with a magnetic stirrer and Pt plate (10 mm × 30 mm) as anode and graphite rod (ϕ 8 mm) as cathode. 2-oxo-*N*,2-diphenylacetamide **1a** (5 mmol, 1 equiv.), *t*-BuOK (15 mmol, 3 equiv.), KI (15 mmol, 3 equiv.), and MeOH (40 mL) were stirred and electrolyzed at a constant current (50 mA) at room temperature for 20 h. The reaction was monitored by thin-layer chromatography. At the end of the reaction, the reaction solution was evaporated to remove methanol, and then purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain the desired product **1b** (1.01 g, 89% yield).

4. Cyclic Voltammetry (CV) studies

Cyclic voltammograms were recorded with a CHI 605D potentiostat at room temperature in MeOH. Tetrabutylammonium tetrafluoroborate (0.15 M) was used as the supporting electrolyte. Working electrode: a glassy carbon (3 mm in diameter), counter electrode: Pt plate (10 mm × 30 mm), reference electrode: Ag-AgCl (KCl (sat)). The scan rate was 0.1 V/s, ranging from -2 V to 0 V; 0 V to 2 V. 20 mL of MeOH containing 0.15 M tetrabutylammonium tetrafluoroborate was poured into the beaker (glassware) in all experiments. The peak potentials vs. Ag/AgCl for used.

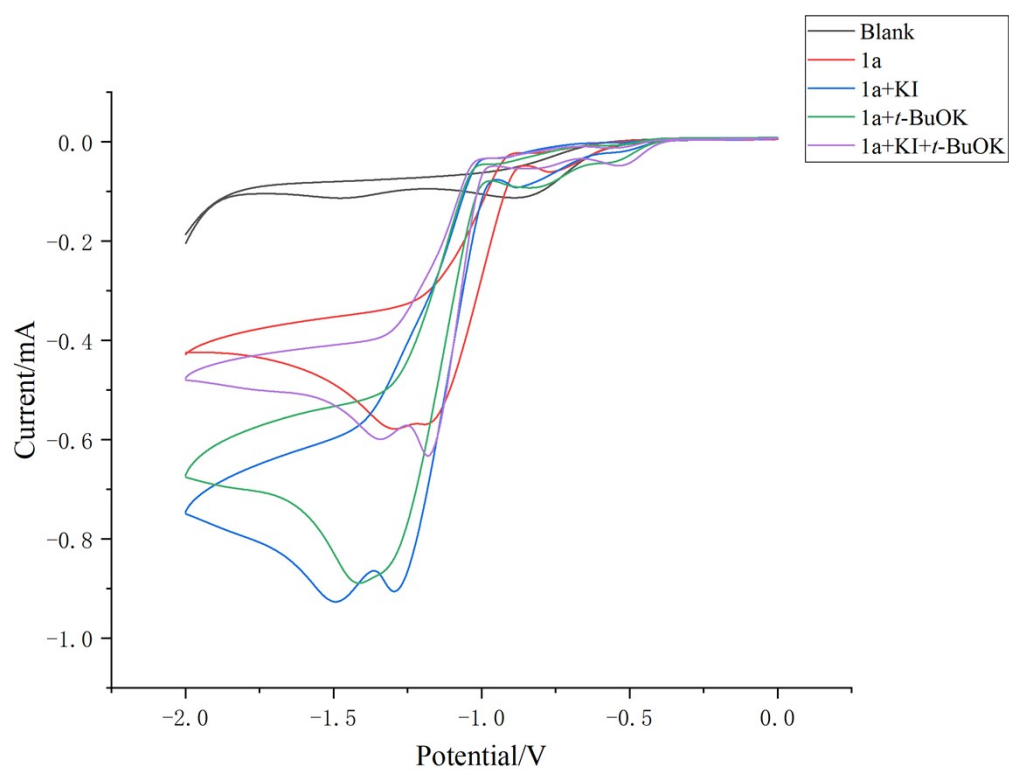


Figure S4. Cyclic voltammograms in MeOH as blank solvent (0.15 M of tetrabutylammonium tetrafluoroborate as the supporting electrolyte); red line, 1.5 mM **1a** in blank solution; blue line, 1.5 mM **1a** and 4.5 mM KI in blank solution; green line, 1.5 mM **1a** and 4.5 mM *t*-BuOK in blank solution; purple line, 1.5 mM **1a**, 4.5 mM KI and 4.5 mM *t*-BuOK in blank solution.

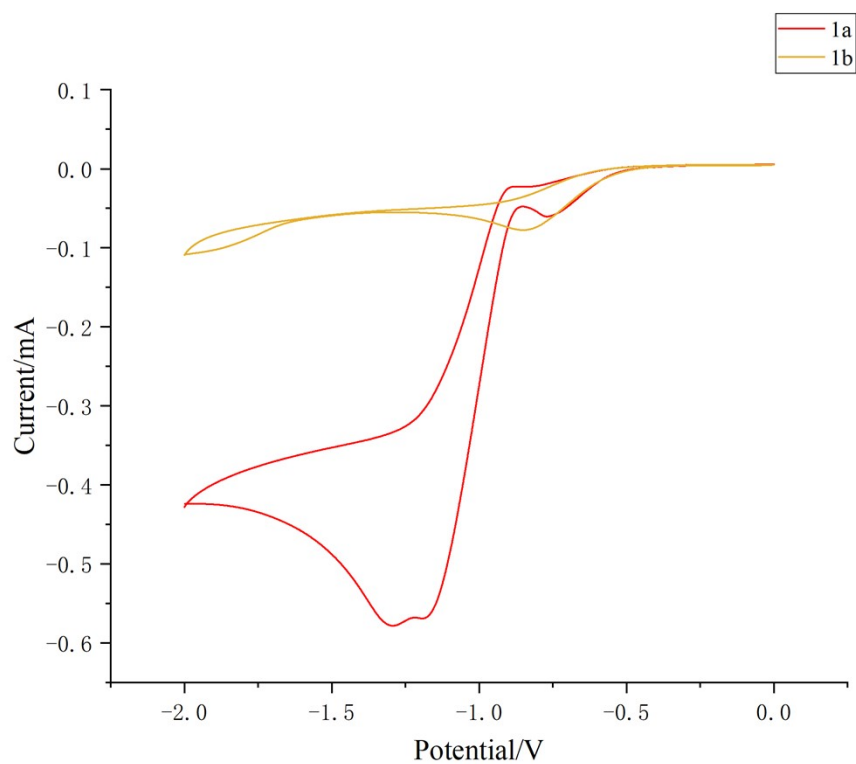


Figure S5. Cyclic voltammograms in MeOH as blank solvent (0.15 M of tetrabutylammonium tetrafluoroborate as the supporting electrolyte); red line, 1.5 mM **1a** in blank solution; yellow line, 1.5 mM **1b** in blank solution.

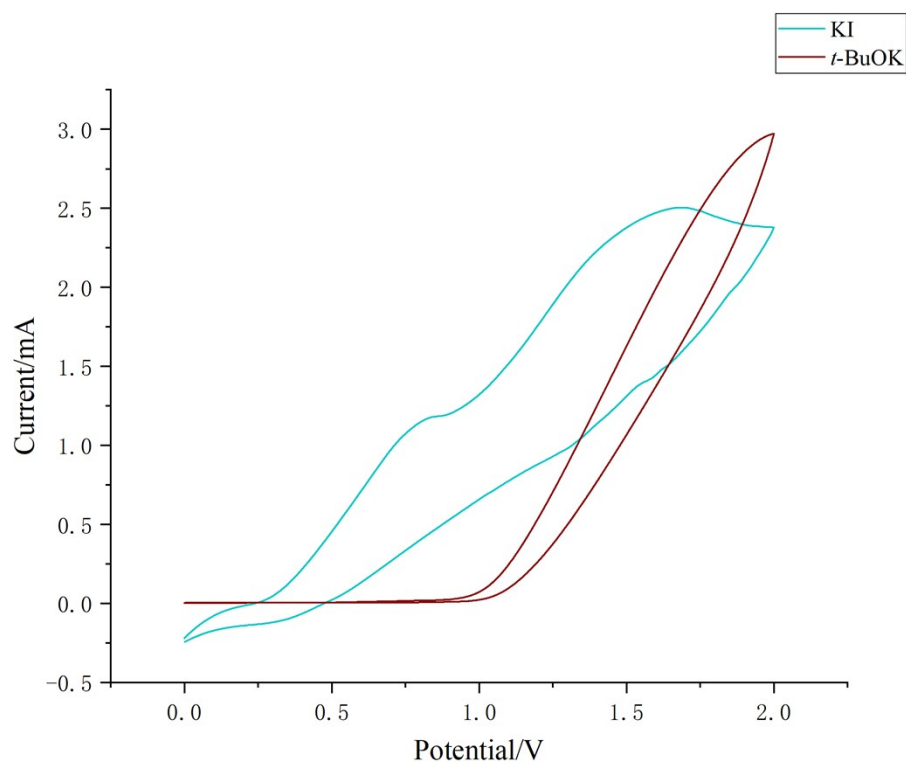


Figure S6. Cyclic voltammograms in MeOH as blank solvent (0.15 M of tetrabutylammonium tetrafluoroborate as the supporting electrolyte); light blue line, 4.5 mM KI in blank solution; brown line, 4.5 mM *t*-BuOK in blank solution.

Cyclic Voltammetry (CV) studies with reaction conditions in Table 1, entry 14

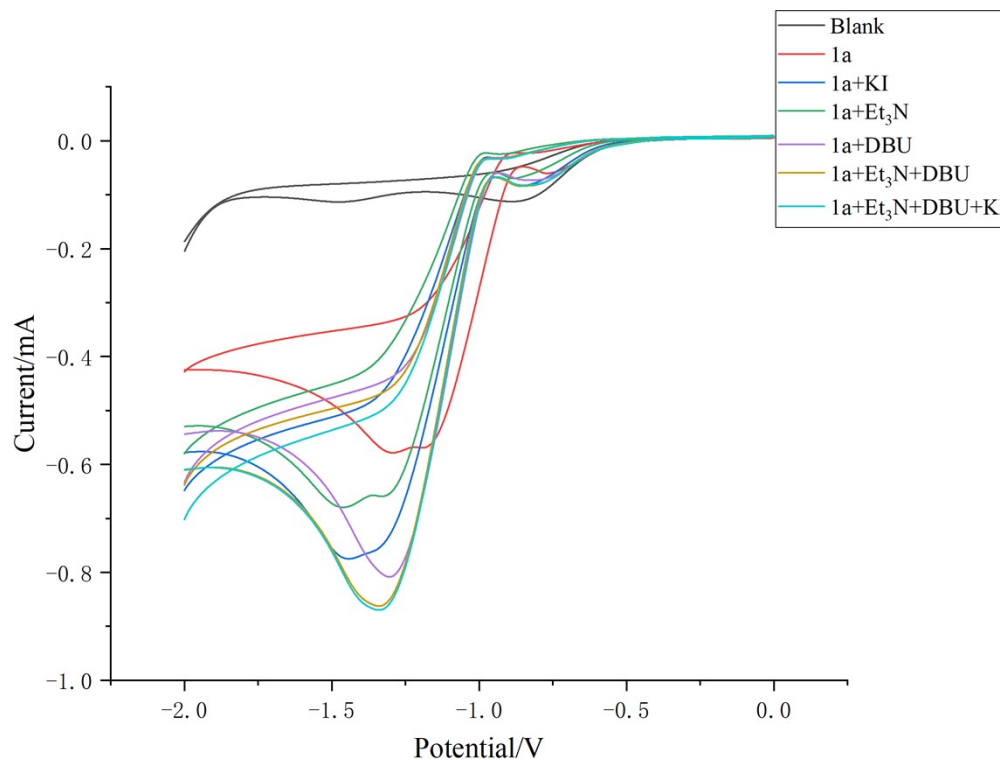


Figure S7. Cyclic voltammograms in MeOH as blank solvent (0.15 M of tetrabutylammonium tetrafluoroborate as the supporting electrolyte); red line, 1.5 mM **1a** in blank solution; blue line, 1.5 mM **1a** and 1.5 mM KI in blank solution; green line, 1.5 mM **1a** and 1.5 mM Et₃N in blank solution; purple line, 1.5 mM **1a** and 1.5 mM DBU in blank solution; yellow line, 1.5 mM **1a**, 1.5 mM Et₃N and 1.5 mM DBU in blank solution; light blue line, 1.5 mM **1a**, 1.5 mM Et₃N, 1.5 mM DBU and 1.5 mM KI in blank solution.

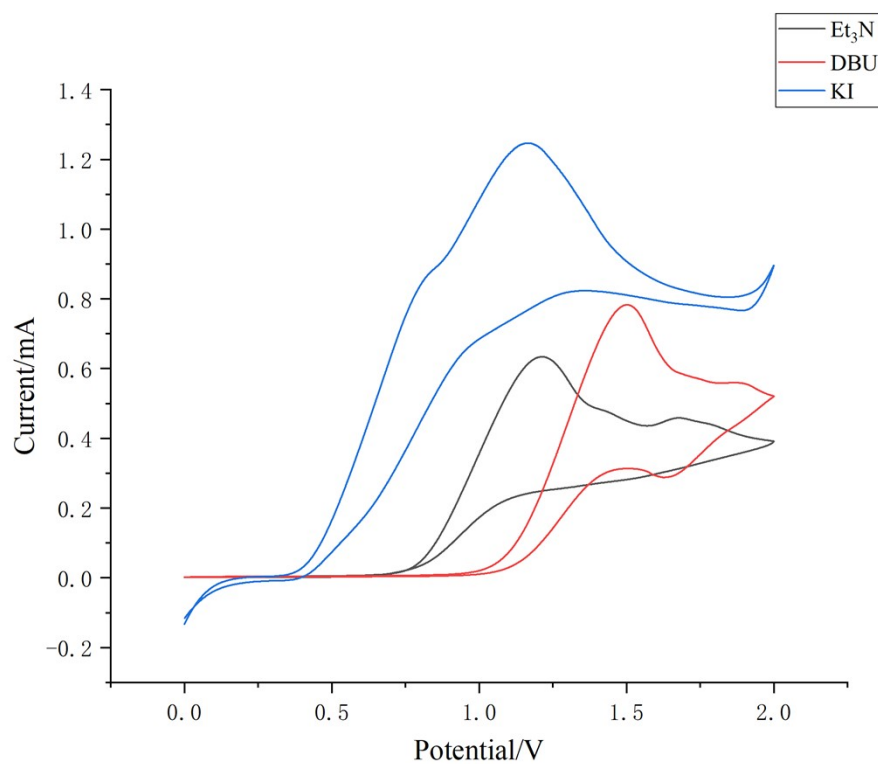


Figure S8. Cyclic voltammograms in MeOH as blank solvent (0.15 M of tetrabutylammonium tetrafluoroborate as the supporting electrolyte); black line, 1.5 mM Et₃N in blank solution; red line, 1.5 mM DBU in blank solution; blue line, 1.5 mM KI in blank solution.

5. Kinetic Isotope Effect (KIE) experiments

Parallel experiment: an oven-dried 10 mL undivided three-necked flask was equipped with a magnetic stirrer and Pt plate (10 mm × 15 mm) as anode and graphite rod (ϕ 8 mm) as cathode. 2-oxo-*N*,2-diphenylacetamide **1a** (0.3 mmol, 1 equiv.), *t*-BuOK (0.9 mmol, 3 equiv.), KI (0.9 mmol, 3 equiv.), CH₃OH (4 mL) or CH₃OD (4 mL) were stirred and electrolyzed at a constant current (10 mA) at room temperature for 6 h. When the reaction was completed, the compound was purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain the product **1b**. The ratio of $K_H/K_D = 1.22$, determined by ¹H NMR spectrum (Figures S9 and S10).

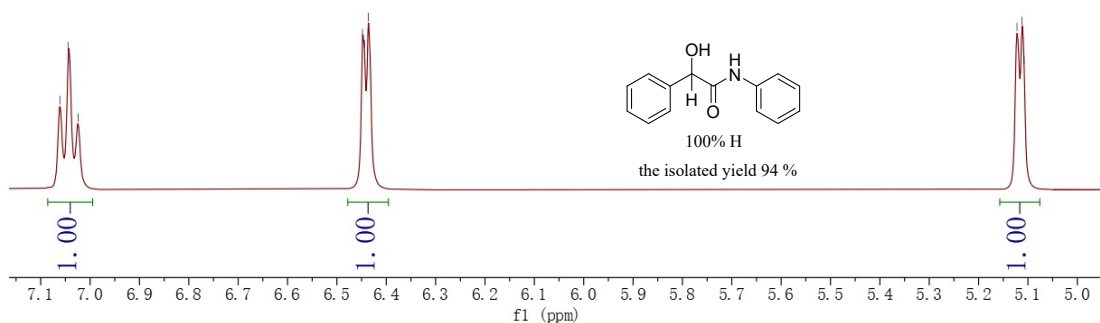


Figure S9. Parallel experiment 1: ¹H NMR of **1b** when 4 mL CH₃OH as solvent.

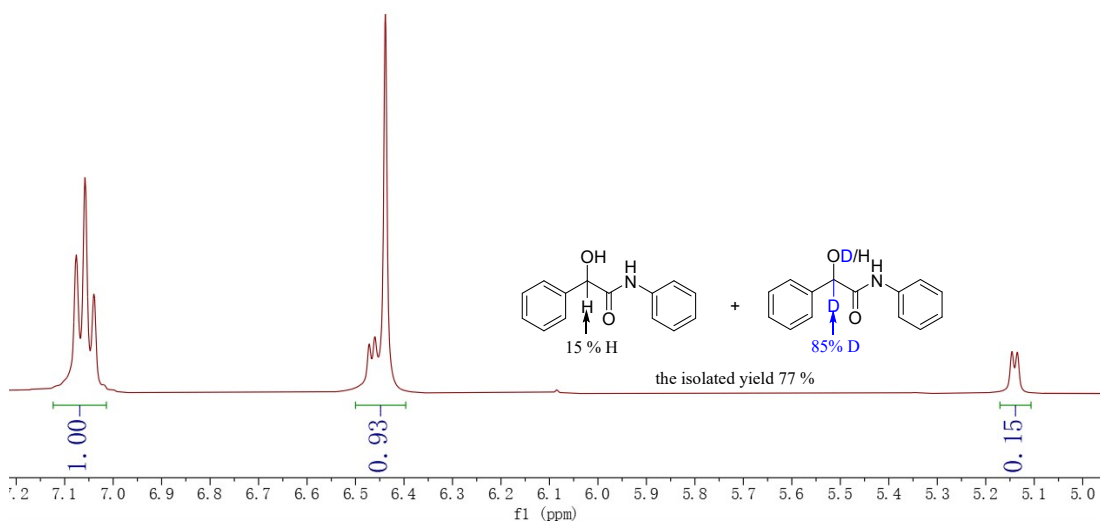


Figure S10. Parallel experiment 2: ¹H NMR of **1b** when 4 mL CH₃OD as solvent.

7. Hydrogen Source experiments

An oven-dried 10 mL undivided three-necked flask was equipped with a magnetic stirrer and Pt plate (10 mm × 15 mm) as anode and graphite rod (ϕ 8 mm) as cathode. 2-oxo-*N*,2-diphenylacetamide **1a** (0.3 mmol, 1 equiv.), *t*-BuOK (0.9 mmol, 3 equiv.), KI (0.9 mmol, 3 equiv.), CH₃OH (4 mL) or CH₃OD (4 mL) or CD₃OH (4 mL) were stirred and electrolyzed at a constant current (10 mA) at room temperature for 6 h. When the reaction was completed, the compound was purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain the product (Figures S9-11).

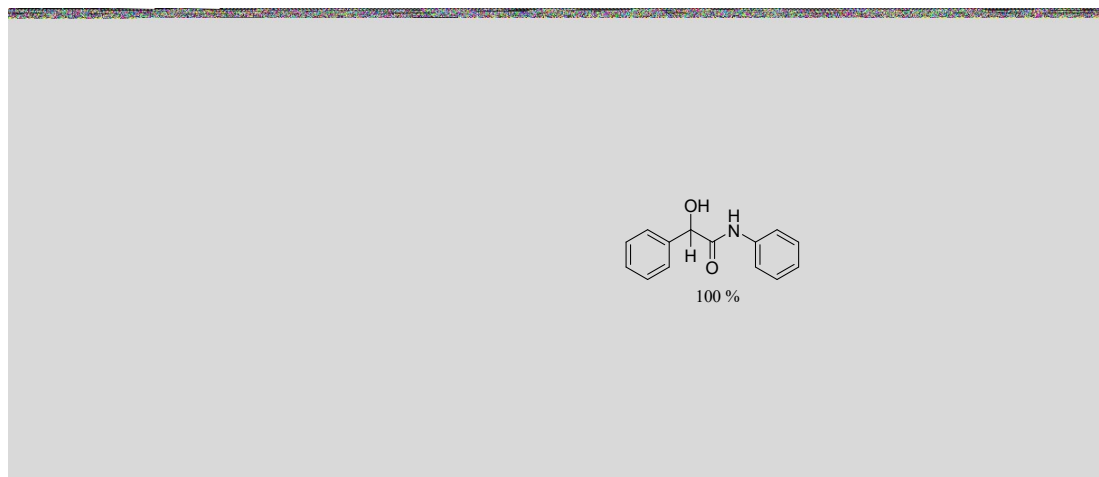


Figure S9. ¹H NMR of **1b** when 4 mL CH₃OH as solvent.

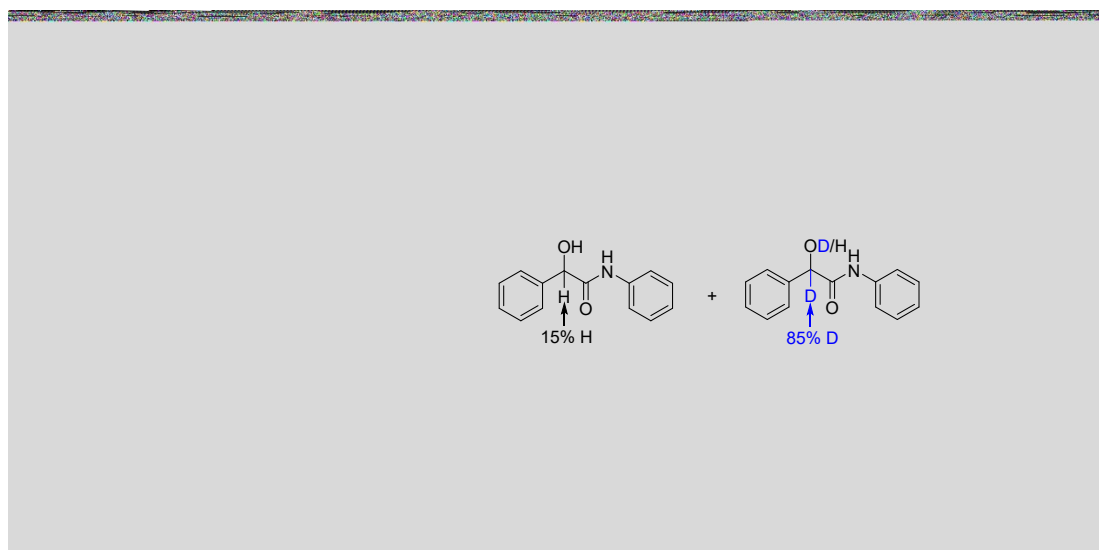


Figure S10. ¹H NMR of **1b** when 4 mL CH₃OD as solvent.

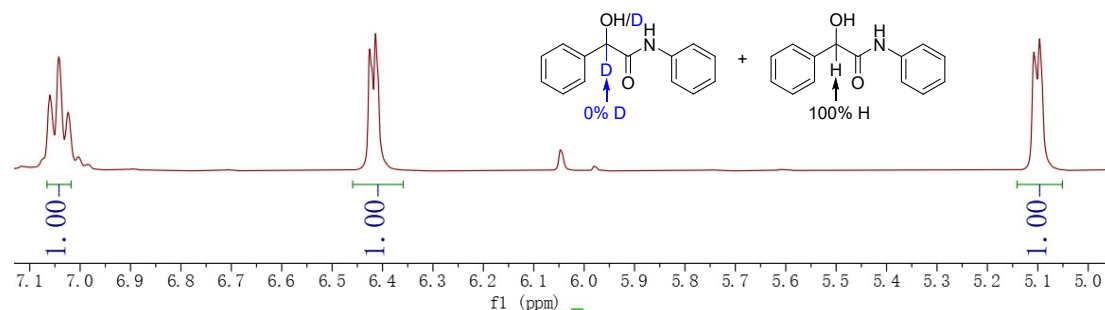


Figure S11. ^1H NMR of **1b** when 4 mL CD_3OH as solvent.

8. Working Electrode experiments

An oven-dried divided cell was equipped with a magnetic stirrer and Pt plate (10 mm $\times$ 15 mm) as anode and graphite rod (ϕ 8 mm) as cathode. 2-oxo-*N*,2-diphenylacetamide **1a** (0.3 mmol, 1 equiv.), *t*-BuOK (0.9 mmol, 3 equiv.), KI (0.9 mmol, 3 equiv.), CH_3OH (30 mL) were stirred and electrolyzed at a constant current (10 mA) at room temperature for 6 h.

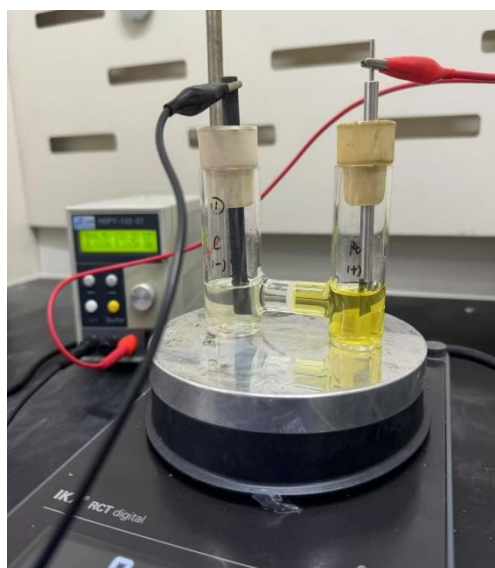


Figure S12. Electrolyzed by divided cell: 0.5 h from the start of the reaction.

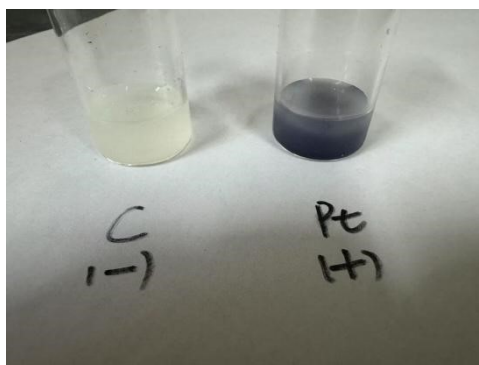


Figure S13. Electrolyzed by divided cell: the iodine was detected by starch solution.

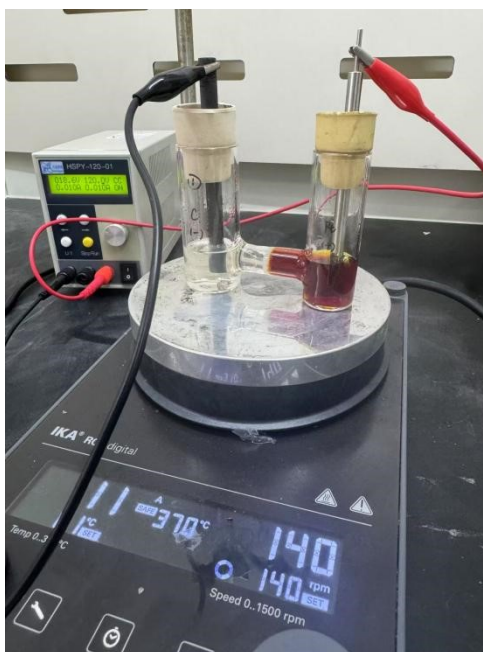


Figure S14. Electrolyzed by divided cell: 6 h from the start of the reaction.

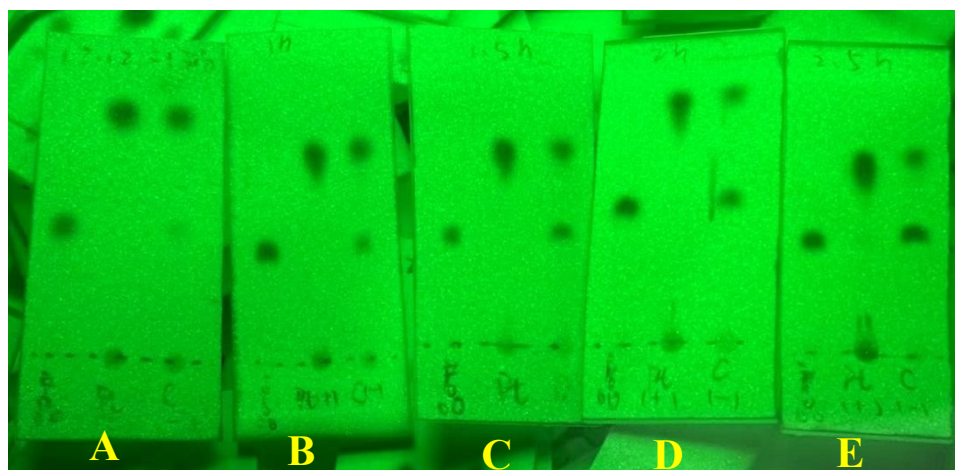


Figure S15. Perform thin-layer chromatography detection with a reaction interval of 0.5 h. From left to right are 0.5h (A), 1h (B), 1.5h (C), 2h (D), 2.5h (E). The corresponding positions on each silicone plate are product **1b**, anodic cell equipped with Pt (+), cathode cell equipped with C (-), respectively.

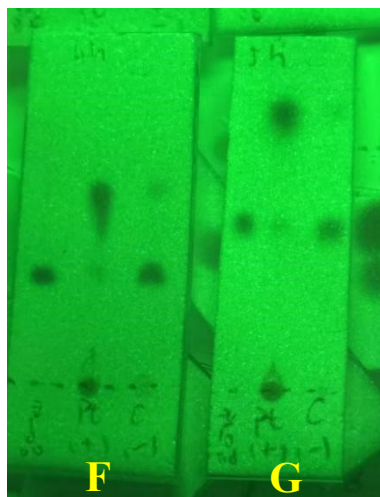


Figure S16. Perform thin-layer chromatography detection with 4 h (F) and 5 h (G). The corresponding positions on each silicone plate are product **1b**, anodic cell equipped with Pt (+), cathode cell equipped with C (-), respectively.

8. Single-Crystal X-Ray Diffraction

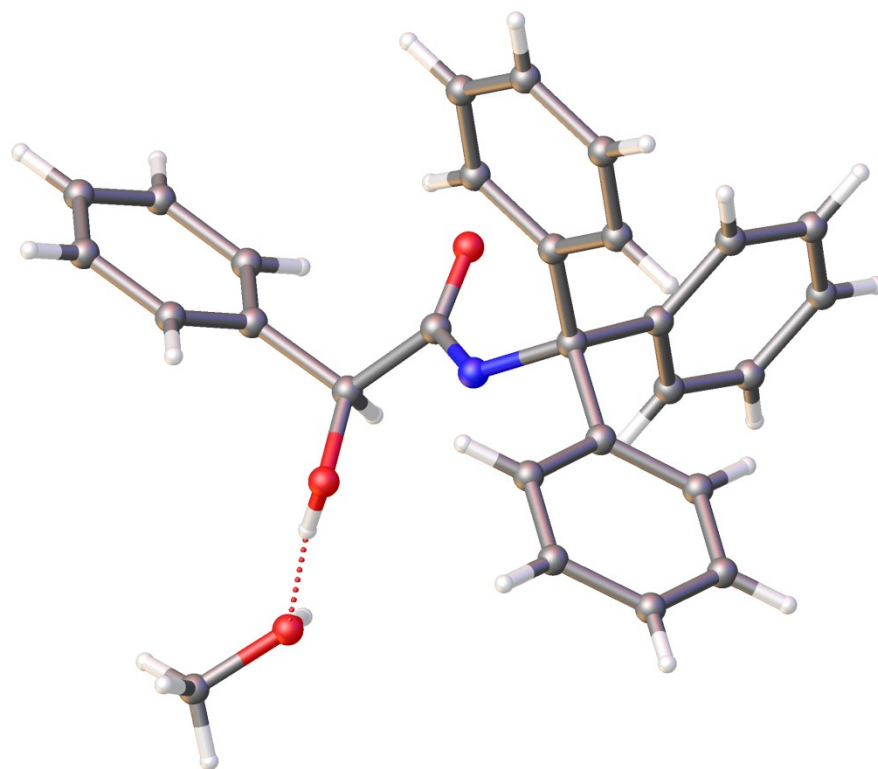
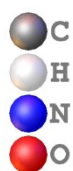
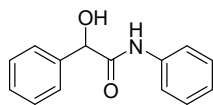
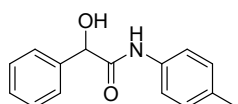


Figure S17. X-ray molecular structure of product **21b**. CCDC: 2407469

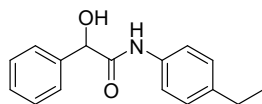
9. Detail descriptions for products



2-hydroxy-N,2-diphenylacetamide (1b).¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (64 mg, 94% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.15 (s, 1H), 7.57 – 7.27 (m, 9H), 7.12 (t, *J* = 7.4 Hz, 1H), 5.16 (s, 1H), 3.47 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 170.03, 139.11, 137.19, 129.21, 129.16, 129.08, 127.04, 124.87, 119.93, 74.85. MS *m/z* (ESI) calcd for C₁₄H₁₄NO₂ ([M+H]⁺): 228.1, found 228.1.

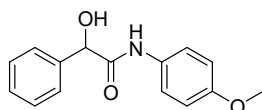


2-hydroxy-2-phenyl-N-(p-tolyl)acetamide (2b).¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (68 mg, 94% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.96 (s, 1H), 7.52 – 7.47 (m, 2H), 7.43 – 7.34 (m, 5H), 7.12 (d, *J* = 8.2 Hz, 2H), 5.20 (d, *J* = 3.3 Hz, 1H), 3.36 (d, *J* = 3.4 Hz, 1H), 2.31 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 169.88, 139.24, 134.67, 134.51, 129.68, 129.14, 129.03, 127.06, 119.94, 74.82, 21.02. MS *m/z* (ESI) calcd for C₁₅H₁₆NO₂ ([M+H]⁺): 242.1, found 242.1.

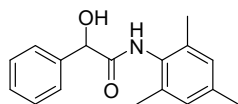


N-(4-ethylphenyl)-2-hydroxy-2-phenylacetamide (3b).¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (64 mg, 83% yield). ¹H NMR (400 MHz,

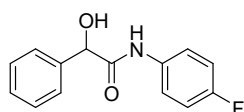
CDCl₃) δ 7.97 (s, 1H), 7.53 – 7.45 (m, 2H), 7.45 – 7.33 (m, 5H), 7.14 (d, J = 8.1 Hz, 2H), 5.20 (d, J = 3.5 Hz, 1H), 3.36 (d, J = 3.4 Hz, 1H), 2.65 – 2.56 (m, 2H), 1.20 (t, J = 7.6 Hz, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 170.93, 140.96, 138.94, 136.23, 128.08, 127.82, 127.58, 126.58, 119.76, 73.96, 27.61, 15.75. MS *m/z* (ESI) calcd for C₁₆H₁₈NO₂ ([M+H]⁺): 256.1, found 256.1.



2-hydroxy-*N*-(4-methoxyphenyl)-2-phenylacetamide (4b).¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a brown solid product (35 mg, 46% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.14 (s, 1H), 7.48 – 7.32 (m, 7H), 6.86 – 6.79 (m, 2H), 5.11 (s, 1H), 3.77 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.02, 156.78, 139.27, 130.29, 129.06, 128.93, 127.02, 121.73, 114.30, 74.69, 55.61. MS *m/z* (ESI) calcd for C₁₅H₁₆NO₃ ([M+H]⁺): 258.1, found 258.1.

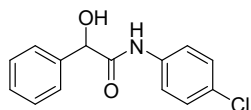


2-hydroxy-*N*-mesityl-2-phenylacetamide (5b).² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (31 mg, 38% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.52 – 7.47 (m, 2H), 7.43 – 7.33 (m, 3H), 7.30 (s, 1H), 6.83 (s, 2H), 5.18 (s, 1H), 2.23 (s, 3H), 2.00 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 171.02, 139.65, 137.32, 135.08, 130.25, 129.06, 129.06, 128.91, 126.93, 74.45, 21.02, 18.16, 18.16. MS *m/z* (ESI) calcd for C₁₇H₂₀NO₂ ([M+H]⁺): 270.1, found 270.1.

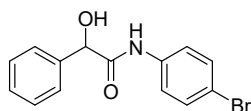


***N*-(4-fluorophenyl)-2-hydroxy-2-phenylacetamide (6b).**¹ The title compound was prepared

according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (55 mg, 75% yield). ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.03 (s, 1H), 7.79 – 7.69 (m, 2H), 7.56 – 7.48 (m, 2H), 7.40 – 7.25 (m, 3H), 7.13 (t, J = 8.9 Hz, 2H), 6.47 (d, J = 4.6 Hz, 1H), 5.11 (d, J = 4.6 Hz, 1H). ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$) δ 171.17, 158.19 (d, J = 238 Hz), 140.85, 134.99 (d, J = 2 Hz), 128.13, 127.66, 126.62, 121.55 (d, J = 7 Hz), 115.16 (d, J = 22 Hz), 74.05. MS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{13}\text{FNO}_2$ ($[\text{M}+\text{H}]^+$): 246.1, found 246.1.

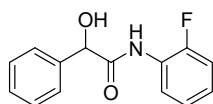


***N*-(4-chlorophenyl)-2-hydroxy-2-phenylacetamide (7b).**¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (45 mg, 60% yield). ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.09 (s, 1H), 7.79 – 7.72 (m, 2H), 7.51 (d, J = 7.3 Hz, 2H), 7.39 – 7.25 (m, 5H), 6.48 (d, J = 4.2 Hz, 1H), 5.11 (d, J = 4.1 Hz, 1H). ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$) δ 171.41, 140.69, 137.55, 128.50, 128.13, 127.67, 127.14, 126.59, 121.31, 74.06. MS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{13}\text{ClNO}_2$ ($[\text{M}+\text{H}]^+$): 262.1, found 262.1.

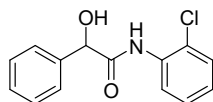


***N*-(4-bromophenyl)-2-hydroxy-2-phenylacetamide (8b).**² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (65 mg, 71% yield). ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.08 (s, 1H), 7.70 (d, J = 8.5 Hz, 2H), 7.57 – 7.42 (m, 4H), 7.40 – 7.23 (m, 3H),

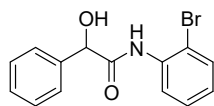
6.47 (d, $J = 4.5$ Hz, 1H), 5.10 (d, $J = 4.4$ Hz, 1H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 171.43, 140.66, 137.96, 131.40, 128.13, 127.67, 126.57, 121.70, 115.20, 74.06. MS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{13}\text{BrNO}_2$ ($[\text{M}+\text{H}]^+$): 306.0, found 306.0.



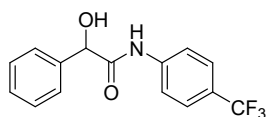
***N*-(2-fluorophenyl)-2-hydroxy-2-phenylacetamide (9b).** The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (56 mg, 76% yield). ^1H NMR (400 MHz, DMSO- d_6) δ 9.61 (s, 1H), 7.90 – 7.82 (m, 1H), 7.53 – 7.47 (m, 2H), 7.40 – 7.23 (m, 4H), 7.21 – 7.13 (m, 2H), 6.65 (d, $J = 4.7$ Hz, 1H), 5.18 (d, $J = 4.6$ Hz, 1H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 171.10, 153.72 (d, $J = 243$ Hz), 140.62, 128.16, 127.72, 126.69, 125.61, 125.51 (d, $J = 2$ Hz), 124.47 (d, $J = 4$ Hz), 123.61, 115.47 (d, $J = 19$ Hz), 73.55. HRMS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{12}\text{FNO}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 268.0744, found 268.0740.



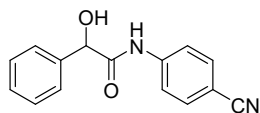
***N*-(2-chlorophenyl)-2-hydroxy-2-phenylacetamide (10b).**² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (71 mg, 91% yield). ^1H NMR (400 MHz, DMSO- d_6) δ 9.65 (s, 1H), 8.15 – 8.05 (m, 1H), 7.60 – 7.44 (m, 3H), 7.43 – 7.26 (m, 4H), 7.22 – 7.10 (m, 1H), 6.95 (d, $J = 3.9$ Hz, 1H), 5.18 (d, $J = 3.5$ Hz, 1H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 170.86, 140.40, 134.16, 129.37, 128.21, 127.86, 127.83, 126.71, 125.50, 123.80, 122.20, 73.63. MS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{13}\text{ClNO}_2$ ($[\text{M}+\text{H}]^+$): 262.1, found 262.1.



***N*-(2-bromophenyl)-2-hydroxy-2-phenylacetamide (11b).**³ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (75 mg, 82% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.77 (s, 1H), 8.37 – 8.30 (m, 1H), 7.55 – 7.48 (m, 3H), 7.45 – 7.34 (m, 3H), 7.32 – 7.26 (m, 1H), 7.02 – 6.94 (m, 1H), 5.22 (s, 1H), 3.55 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 170.30, 138.81, 135.18, 132.45, 129.20, 129.15, 128.54, 126.99, 125.65, 121.59, 113.75, 75.13. MS *m/z* (ESI) calcd for C₁₄H₁₃BrNO₂ ([M+H]⁺): 306.0, found 306.0.

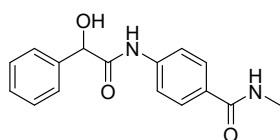


2-hydroxy-2-phenyl-*N*-(4-(trifluoromethyl)phenyl)acetamide (12b).¹ The title compound was prepared according to the general procedure and purified by recrystallization to obtain a white solid product (89 mg, 100% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.57 – 10.46 (m, 1H), 7.96 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.5 Hz, 2H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.38 – 7.25 (m, 3H), 6.57 – 6.52 (m, 1H), 5.20 (t, *J* = 5.1 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 172.01, 142.25, 140.62, 128.19, 127.76, 126.67, 125.91 (q, *J* = 3 Hz), 124.40 (q, *J* = 270 Hz), 123.68 (q, *J* = 31 Hz), 119.75, 74.16. MS *m/z* (ESI) calcd for C₁₅H₁₃F₃NO₂ ([M+H]⁺): 296.1, found 296.1.

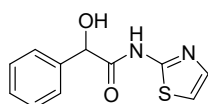


***N*-(4-cyanophenyl)-2-hydroxy-2-phenylacetamide (13b).**² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (46 mg, 61% yield). ¹H NMR (400 MHz,

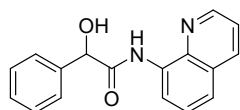
DMSO-*d*₆) δ 10.39 (s, 1H), 7.94 (d, *J* = 8.4 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.44 – 7.25 (m, 3H), 6.57 (d, *J* = 3.8 Hz, 1H), 5.15 (d, *J* = 3.2 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 172.14, 142.84, 140.40, 133.14, 128.21, 127.81, 126.61, 119.82, 119.05, 105.36, 74.16. MS *m/z* (ESI) calcd for C₁₅H₁₃N₂O₂ ([M+H]⁺): 253.1, found 253.1.



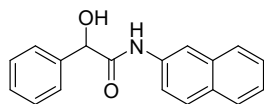
4-(2-hydroxy-2-phenylacetamido)-*N*-methylbenzamide (14b).³ The title compound was prepared according to the general procedure and purified by recrystallization to obtain a light yellow solid product (85 mg, 100% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.12 (s, 1H), 8.35 – 8.25 (m, 1H), 7.77 (s, 4H), 7.52 (d, *J* = 7.4 Hz, 2H), 7.41 – 7.25 (m, 3H), 6.49 (d, *J* = 4.6 Hz, 1H), 5.12 (d, *J* = 4.6 Hz, 1H), 2.75 (d, *J* = 4.5 Hz, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 171.54, 166.04, 140.96, 140.65, 129.35, 128.14, 127.72, 127.68, 126.58, 118.90, 74.05, 26.19. MS *m/z* (ESI) calcd for C₁₆H₁₇N₂O₃ ([M+H]⁺): 285.1, found 285.1.



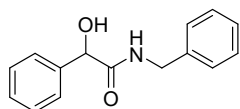
2-hydroxy-2-phenyl-*N*-(thiazol-2-yl)acetamide (15b).² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (31 mg, 44% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.54 – 7.48 (m, 2H), 7.42 – 7.35 (m, 4H), 6.97 (d, *J* = 3.6 Hz, 1H), 5.41 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 170.25, 159.13, 138.08, 136.85, 129.25, 129.15, 126.87, 114.21, 74.46. MS *m/z* (ESI) calcd for C₁₁H₁₁N₂O₂S ([M+H]⁺): 235.1, found 235.1.



2-hydroxy-2-phenyl-*N*-(quinolin-8-yl)acetamide (16b).² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a brown solid product (28 mg, 34% yield). ¹H NMR (400 MHz, CDCl₃) δ 10.66 (s, 1H), 8.79 – 8.75 (m, 1H), 8.74 – 8.68 (m, 1H), 8.14 – 8.09 (m, 1H), 7.61 – 7.55 (m, 2H), 7.51 – 7.47 (m, 2H), 7.43 – 7.30 (m, 4H), 5.35 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 170.80, 148.67, 139.47, 138.78, 136.40, 133.80, 128.99, 128.76, 128.05, 127.30, 126.98, 122.29, 121.77, 116.87, 75.16. MS m/z (ESI) calcd for C₁₇H₁₅N₂O₂ ([M+H]⁺): 279.1, found 279.1.

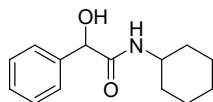


2-hydroxy-*N*-(naphthalen-2-yl)-2-phenylacetamide (17b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a brown solid product (31 mg, 38% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.30 (s, 1H), 8.23 (d, *J* = 2.2 Hz, 1H), 7.82 – 7.74 (m, 3H), 7.56 – 7.50 (m, 2H), 7.50 – 7.36 (m, 6H), 5.27 (d, *J* = 3.3 Hz, 1H), 3.36 (d, *J* = 3.4 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 171.57, 140.93, 136.25, 133.29, 129.87, 128.20, 128.08, 127.58, 127.44, 127.32, 126.62, 126.38, 124.68, 120.45, 115.73, 73.92. HRMS m/z (ESI) calcd for C₁₈H₁₅NO₂Na ([M+Na]⁺): 300.0995, found 300.0990.

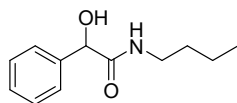


***N*-benzyl-2-hydroxy-2-phenylacetamide (18b).**¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (26 mg, 36% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.25 (m, 8H), 7.17 – 7.12 (m, 2H), 6.73 (d, *J* = 6.1 Hz, 1H), 4.99 (s, 1H), 4.43 – 4.29 (m, 2H). ¹³C NMR

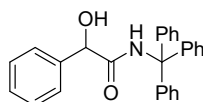
(101 MHz, CDCl₃) δ 172.25, 139.50, 137.79, 129.01, 128.85, 128.83, 127.73, 127.71, 126.96, 74.35, 43.62. MS m/z (ESI) calcd for C₁₅H₁₆NO₂ ([M+H]⁺): 242.1, found 242.1.



***N*-cyclohexyl-2-hydroxy-2-phenylacetamide (19b).**¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (58 mg, 82% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.40 – 7.27 (m, 5H), 6.24 (d, *J* = 8.3 Hz, 1H), 4.91 (s, 1H), 4.08 (s, 1H), 3.77 – 3.62 (m, 1H), 1.88 – 1.76 (m, 2H), 1.71 – 1.51 (m, 3H), 1.38 – 1.22 (m, 2H), 1.20 – 1.00 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.42, 139.85, 128.83, 128.55, 126.91, 74.11, 48.37, 32.96, 32.87, 25.50, 24.80, 24.77. MS m/z (ESI) calcd for C₁₄H₂₀NO₂ ([M+H]⁺): 234.1, found 234.1.

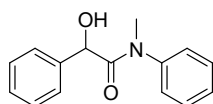


***N*-butyl-2-hydroxy-2-phenylacetamide (20b).**³ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (27 mg, 43% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.43 – 7.30 (m, 5H), 6.04 (s, 1H), 5.01 (s, 1H), 3.66 (s, 1H), 3.33 – 3.18 (m, 2H), 1.48 – 1.41 (m, 2H), 1.31 – 1.24 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 172.13, 139.68, 129.05, 128.82, 127.02, 74.27, 39.54, 31.63, 20.05, 13.81. MS m/z (ESI) calcd for C₁₂H₁₈NO₂ ([M+H]⁺): 208.1, found 208.1.

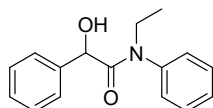


2-hydroxy-2-phenyl-*N*-tritylacetamide (21b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to

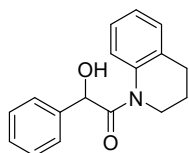
obtain a white solid product (54 mg, 46% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.38 – 7.30 (m, 6H), 7.23 – 7.18 (m, 8H), 7.09 – 7.03 (m, 6H), 4.95 (s, 1H), 3.38 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.19, 144.32, 139.48, 128.98, 128.74, 128.53, 128.11, 127.25, 126.73, 74.58, 70.41. HRMS m/z (ESI) calcd for $\text{C}_{27}\text{H}_{23}\text{NO}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 416.1621, found 416.1624.



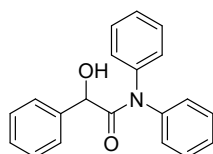
2-hydroxy-N-methyl-N,2-diphenylacetamide (22b).² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (17 mg, 24% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.33 – 7.10 (m, 6H), 6.87 – 6.74 (m, 4H), 5.04 – 4.97 (m, 1H), 4.55 (d, J = 6.3 Hz, 1H), 3.29 (d, J = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.85, 141.52, 139.33, 129.60, 128.37, 128.37, 128.08, 127.98, 127.34, 71.69, 38.35. MS m/z (ESI) calcd for $\text{C}_{15}\text{H}_{16}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 242.1 found 242.1.



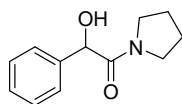
N-ethyl-2-hydroxy-N,2-diphenylacetamide (23b).⁴ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a colorless oily liquid product (41 mg, 53% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.50 – 7.08 (m, 6H), 6.98 – 6.50 (m, 4H), 4.93 (s, 1H), 4.00 – 3.88 (m, 1H), 3.64 – 3.53 (m, 1H), 1.12 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.34, 139.85, 139.50, 129.52, 129.12, 128.50, 128.45, 128.13, 127.45, 71.87, 45.50, 12.87. MS m/z (ESI) calcd for $\text{C}_{16}\text{H}_{18}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 256.1, found 256.1.



1-(3,4-dihydroquinolin-1(2H)-yl)-2-hydroxy-2-phenylethan-1-one (24b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (75 mg, 93% yield). ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.73 – 7.45 (m, 1H), 7.41 – 7.02 (m, 8H), 5.74 (d, J = 6.6 Hz, 1H), 5.61 (d, J = 6.7 Hz, 1H), 3.88 (s, 1H), 3.33 (s, 1H), 2.66 – 2.53 (m, 1H), 2.36 (s, 1H), 1.80 – 1.63 (m, 1H), 1.63 – 1.44 (m, 1H). ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$) δ 171.76, 140.09, 138.38, 128.60, 128.19, 127.59, 126.59, 126.59, 125.73, 125.08, 124.49, 71.24, 43.24, 25.60, 23.02. HRMS m/z (ESI) calcd for $\text{C}_{17}\text{H}_{18}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 268.1332, found 268.1330.

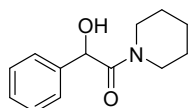


2-hydroxy-*N,N*,2-triphenylacetamide (25b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a brown solid product (21 mg, 23% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.12 (m, 11H), 6.90 (d, J = 6.9 Hz, 4H), 5.23 (s, 1H), 4.55 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 173.29, 138.98, 129.63, 129.33, 129.18, 128.60, 128.36, 127.44, 125.95, 72.30. HRMS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{17}\text{NO}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 326.1151, found 326.1150.

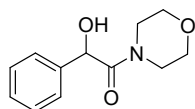


2-hydroxy-2-phenyl-1-(pyrrolidin-1-yl)ethan-1-one (26b).⁵ The title compound was prepared according to the general procedure and purified by column chromatography (dichloromethane/methanol = 50:1) to obtain a white solid product (40 mg, 65% yield). ^1H NMR

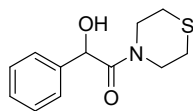
(400 MHz, CDCl₃) δ 7.39 – 7.28 (m, 5H), 5.04 (s, 1H), 4.79 (s, 1H), 3.66 – 3.31 (m, 3H), 2.89 – 2.77 (m, 1H), 1.90 – 1.70 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 170.77, 139.01, 129.00, 128.57, 127.86, 72.72, 46.68, 45.93, 25.96, 23.88. MS m/z (ESI) calcd for C₁₂H₁₆NO₂ ([M+H]⁺): 206.1, found 206.1.



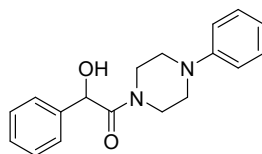
2-hydroxy-2-phenyl-1-(piperidin-1-yl)ethan-1-one (27b).⁶ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (35 mg, 53% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.27 (m, 5H), 5.21 (s, 1H), 4.94 (s, 1H), 3.84 – 3.70 (m, 1H), 3.51 – 3.37 (m, 1H), 3.16 (t, *J* = 5.6 Hz, 2H), 1.61 – 1.48 (m, 3H), 1.48 – 1.38 (m, 1H), 1.35 – 1.23 (m, 1H), 0.96 – 0.80 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 170.54, 139.76, 129.10, 128.50, 127.56, 71.53, 45.96, 44.12, 25.47, 25.26, 24.27. MS m/z (ESI) calcd for C₁₃H₁₈NO₂ ([M+H]⁺): 220.1, found 220.1.



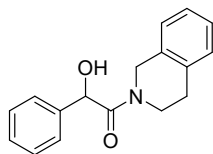
2-hydroxy-1-morpholino-2-phenylethan-1-one (28b).⁷ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (18 mg, 28% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.28 (m, 5H), 5.19 (d, *J* = 6.1 Hz, 1H), 4.71 (d, *J* = 6.3 Hz, 1H), 3.82 – 3.42 (m, 5H), 3.34 – 3.02 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.13, 139.21, 129.32, 128.86, 127.50, 71.64, 66.67, 65.92, 45.42, 43.27. MS m/z (ESI) calcd for C₁₂H₁₆NO₃ ([M+H]⁺): 222.1, found 222.1.



2-hydroxy-2-phenyl-1-thiomorpholinoethan-1-one (29b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (38 mg, 53% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.42 – 7.27 (m, 5H), 5.17 (d, J = 4.9 Hz, 1H), 4.81 (d, J = 5.8 Hz, 1H), 4.32 – 4.20 (m, 1H), 3.68 – 3.52 (m, 2H), 3.49 – 3.38 (m, 1H), 2.56 (t, J = 5.2 Hz, 2H), 2.25 – 2.13 (m, 1H), 1.85 – 1.70 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.08, 139.30, 129.37, 128.86, 127.56, 71.77, 47.56, 45.64, 27.26, 26.60. HRMS m/z (ESI) calcd for $\text{C}_{12}\text{H}_{15}\text{NO}_2\text{SNa}$ ($[\text{M}+\text{Na}]^+$): 260.0716, found 260.0719.

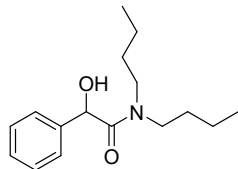


2-hydroxy-2-phenyl-1-(4-phenylpiperazin-1-yl)ethan-1-one (30b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (38 mg, 53% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.41 – 7.30 (m, 5H), 7.27 – 7.20 (m, 2H), 6.92 – 6.78 (m, 3H), 5.25 (s, 1H), 4.76 (s, 1H), 4.04 – 3.93 (m, 1H), 3.76 – 3.65 (m, 1H), 3.47 – 3.19 (m, 3H), 3.05 – 2.94 (m, 2H), 2.53 – 2.42 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.98, 150.74, 139.39, 129.34, 129.30, 128.82, 127.55, 120.81, 116.75, 71.70, 49.25, 48.96, 44.86, 42.87. HRMS m/z (ESI) calcd for $\text{C}_{18}\text{H}_{21}\text{N}_2\text{O}_2$ ($[\text{M}+\text{H}]^+$): 297.1598, found 297.1595.

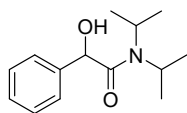


1-(3,4-dihydroisoquinolin-2(1H)-yl)-2-hydroxy-2-phenylethan-1-one (31b). The title

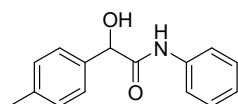
compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (68 mg, 85% yield). ^1H NMR (400 MHz, CDCl_3) (mixture of isomers) δ 7.40 – 7.27 (m, 10H), 7.22 – 7.08 (m, 6H), 7.03 – 6.99 (m, 1H), 6.87 – 6.82 (m, 1H), 5.33 – 5.28 (m, 2H), 4.95 (d, J = 17.2 Hz, 1H), 4.90 – 4.85 (m, 1H), 4.84 – 4.79 (m, 1H), 4.70 (d, J = 17.2 Hz, 1H), 4.50 (d, J = 16.0 Hz, 1H), 4.15 (d, J = 16.0 Hz, 1H), 4.11 – 4.04 (m, 1H), 3.75 – 3.67 (m, 1H), 3.50 – 3.42 (m, 3H), 2.97 – 2.78 (m, 1H), 2.65 – 2.55 (m, 1H), 2.28 – 2.17 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) (mixture of isomers) δ 171.52, 171.43, 139.38, 139.12, 134.60, 133.65, 132.37, 131.64, 129.22, 129.19, 128.80, 128.69, 128.65, 128.54, 127.63, 127.53, 127.19, 126.79, 126.74, 126.64, 126.51, 126.00, 71.98, 71.96, 46.61, 45.26, 42.47, 41.27, 28.40, 28.28. HRMS m/z (ESI) calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 290.1151, found 290.1151.



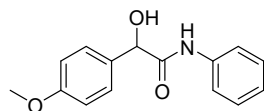
***N,N*-dibutyl-2-hydroxy-2-phenylacetamide (32b).**⁸ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow oily liquid product (55 mg, 70% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.27 (m, 5H), 5.14 (d, J = 4.9 Hz, 1H), 4.87 (d, J = 6.1 Hz, 1H), 3.55 – 3.45 (m, 1H), 3.26 – 3.16 (m, 1H), 3.13 – 3.02 (m, 1H), 2.95 – 2.85 (m, 1H), 1.57 – 1.43 (m, 2H), 1.41 – 1.33 (m, 1H), 1.33 – 1.22 (m, 2H), 1.17 – 1.05 (m, 2H), 1.03 – 0.94 (m, 1H), 0.91 (t, J = 7.3 Hz, 3H), 0.79 (t, J = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.88, 139.96, 129.12, 128.57, 127.60, 71.75, 46.65, 46.23, 30.13, 29.46, 20.25, 20.04, 13.92, 13.80. MS m/z (ESI) calcd for $\text{C}_{16}\text{H}_{26}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 264.2, found 264.2.



2-hydroxy-*N,N*-diisopropyl-2-phenylacetamide (33b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (40 mg, 60% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.24 (m, 5H), 5.22 – 5.05 (m, 2H), 3.86 – 3.75 (m, 1H), 3.41 – 3.29 (m, 1H), 1.48 (d, *J* = 6.8 Hz, 3H), 1.40 (d, *J* = 6.8 Hz, 3H), 1.15 (d, *J* = 6.7 Hz, 3H), 0.46 (d, *J* = 6.6 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.78, 140.26, 129.07, 128.37, 127.57, 71.98, 48.09, 46.49, 20.62, 20.62, 19.70, 18.72. HRMS *m/z* (ESI) calcd for C₁₄H₂₁NO₂Na ([M+Na]⁺): 258.1464, found 258.1469.

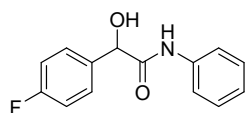


2-hydroxy-*N*-phenyl-2-(*p*-tolyl)acetamide (34b).⁹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (46 mg, 64% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.88 (s, 1H), 7.73 – 7.67 (m, 2H), 7.40 (d, *J* = 7.9 Hz, 2H), 7.32 – 7.24 (m, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 7.07 – 7.01 (m, 1H), 6.36 (d, *J* = 4.6 Hz, 1H), 5.07 (d, *J* = 4.6 Hz, 1H), 2.28 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 171.35, 138.60, 137.95, 136.79, 128.67, 128.63, 126.55, 123.51, 119.68, 73.88, 20.76. MS *m/z* (ESI) calcd for C₁₅H₁₆NO₂ ([M+H]⁺): 242.1, found 242.1.

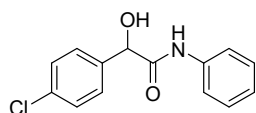


2-hydroxy-2-(4-methoxyphenyl)-*N*-phenylacetamide (35b).¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (31 mg, 40% yield). ¹H NMR (400 MHz,

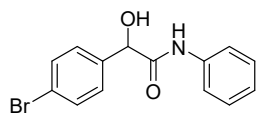
CDCl₃) δ 8.38 (s, 1H), 7.53 – 7.45 (m, 2H), 7.36 – 7.26 (m, 4H), 7.11 (t, J = 7.4 Hz, 1H), 6.90 – 6.82 (m, 2H), 5.03 (s, 1H), 3.77 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.75, 159.99, 137.19, 131.36, 129.13, 128.36, 124.78, 119.97, 114.37, 74.27, 55.42. MS m/z (ESI) calcd for C₁₅H₁₆NO₃ ([M+H]⁺): 258.1, found 258.1.



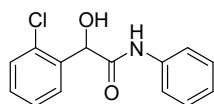
2-(4-fluorophenyl)-2-hydroxy-N-phenylacetamide (36b).² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (62 mg, 84% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.92 (s, 1H), 7.69 (d, J = 8.0 Hz, 2H), 7.59 – 7.50 (m, 2H), 7.28 (t, J = 7.7 Hz, 2H), 7.18 (t, J = 8.7 Hz, 2H), 7.05 (t, J = 7.3 Hz, 1H), 6.49 (d, J = 4.7 Hz, 1H), 5.12 (d, J = 4.7 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 171.02, 161.68 (d, J = 242 Hz), 138.48, 137.10 (d, J = 3 Hz), 128.61 (d, J = 5 Hz), 128.50, 123.59, 119.71, 114.88 (d, J = 21 Hz), 73.26. MS m/z (ESI) calcd for C₁₄H₁₃FNO₂ ([M+H]⁺): 246.1, found 246.1.



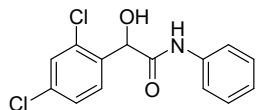
2-(4-chlorophenyl)-2-hydroxy-N-phenylacetamide (37b).⁹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (61 mg, 77% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.49 (s, 1H), 7.57 (d, J = 7.8 Hz, 2H), 7.47 (d, J = 8.3 Hz, 2H), 7.43 – 7.37 (m, 4H), 7.22 (t, J = 7.4 Hz, 1H), 5.19 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 169.99, 137.56, 136.92, 134.75, 129.22, 129.09, 128.23, 125.07, 120.05, 73.96. MS m/z (ESI) calcd for C₁₄H₁₃ClNO₂ ([M+H]⁺): 262.1, found 262.1.



2-(4-bromophenyl)-2-hydroxy-N-phenylacetamide (38b).⁹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (48 mg, 53% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.94 (s, 1H), 7.71 – 7.64 (m, 2H), 7.60 – 7.53 (m, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.29 (t, *J* = 7.9 Hz, 2H), 7.09 – 7.01 (m, 1H), 6.54 (d, *J* = 4.5 Hz, 1H), 5.11 (d, *J* = 4.2 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 170.72, 140.26, 138.43, 131.02, 128.73, 128.63, 123.64, 120.79, 119.74, 73.28. MS *m/z* (ESI) calcd for C₁₄H₁₃BrNO₂ ([M+H]⁺): 306.0, found 306.0.

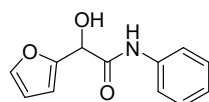


2-(2-chlorophenyl)-2-hydroxy-N-phenylacetamide (39b).² The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (68 mg, 87% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.03 (s, 1H), 7.76 – 7.68 (m, 2H), 7.60 – 7.54 (m, 1H), 7.48 – 7.42 (m, 1H), 7.40 – 7.26 (m, 4H), 7.11 – 7.03 (m, 1H), 6.65 (d, *J* = 3.9 Hz, 1H), 5.50 – 5.45 (m, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 170.02, 138.71, 138.51, 132.59, 129.42, 129.22, 129.15, 128.63, 127.21, 123.64, 119.79, 71.14. MS *m/z* (ESI) calcd for C₁₄H₁₃ClNO₂ ([M+H]⁺): 262.1, found 262.1.

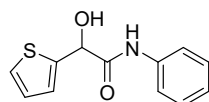


2-(2,4-dichlorophenyl)-2-hydroxy-N-phenylacetamide (40b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (77 mg, 87% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.06 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 2H), 7.63 – 7.56 (m, 2H), 7.50 – 7.43 (m,

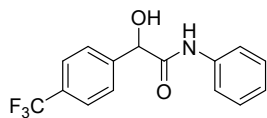
1H), 7.30 (t, $J = 7.7$ Hz, 2H), 7.07 (t, $J = 7.4$ Hz, 1H), 6.73 (d, $J = 5.3$ Hz, 1H), 5.45 (d, $J = 5.3$ Hz, 1H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 169.57, 138.44, 137.91, 133.43, 133.03, 130.49, 128.63, 127.42, 123.71, 119.81, 70.77. HRMS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{12}\text{Cl}_2\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 296.0240, found 296.0246.



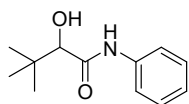
2-(furan-2-yl)-2-hydroxy-*N*-phenylacetamide (41b).¹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (29 mg, 45% yield). ^1H NMR (400 MHz, DMSO- d_6) δ 10.05 (s, 1H), 7.72 (d, $J = 8.0$ Hz, 2H), 7.61 (s, 1H), 7.30 (t, $J = 7.8$ Hz, 2H), 7.06 (t, $J = 7.4$ Hz, 1H), 6.55 (d, $J = 5.8$ Hz, 1H), 6.45 – 6.36 (m, 2H), 5.17 (d, $J = 4.6$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.72, 151.30, 143.37, 137.05, 129.24, 125.03, 120.03, 110.89, 109.23, 68.42. MS m/z (ESI) calcd for $\text{C}_{12}\text{H}_{12}\text{NO}_3$ ($[\text{M}+\text{H}]^+$): 218.1, found 218.1.



2-hydroxy-*N*-phenyl-2-(thiophen-2-yl)acetamide (42b).⁹ The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a light yellow solid product (20 mg, 28% yield). ^1H NMR (400 MHz, CDCl_3) δ 8.37 (s, 1H), 7.70 – 7.62 (m, 2H), 7.50 – 7.41 (m, 3H), 7.34 – 7.23 (m, 2H), 7.17 – 7.09 (m, 1H), 5.57 (s, 1H), 3.93 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 169.06, 141.97, 137.02, 129.24, 127.21, 126.56, 126.40, 125.04, 120.08, 70.76. MS m/z (ESI) calcd for $\text{C}_{12}\text{H}_{12}\text{NO}_2\text{S}$ ($[\text{M}+\text{H}]^+$): 234.1, found 234.1.



2-hydroxy-N-phenyl-2-(4-(trifluoromethyl)phenyl)acetamide (43b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (64 mg, 72% yield). ^1H NMR (400 MHz, DMSO- d_6) δ 10.00 (s, 1H), 7.80 – 7.65 (m, 6H), 7.29 (t, 2H), 7.06 (t, 1H), 6.68 (d, J = 4.8 Hz, 1H), 5.25 (d, J = 4.7 Hz, 1H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 170.45, 145.42, 138.37, 128.62, 128.23 (q, J = 31 Hz), 127.25, 125.01 (q, J = 4 Hz), 124.30 (q, J = 271 Hz), 123.70, 119.79, 73.38. HRMS m/z (ESI) calcd for $\text{C}_{15}\text{H}_{13}\text{F}_3\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 296.0893, found 296.0894.



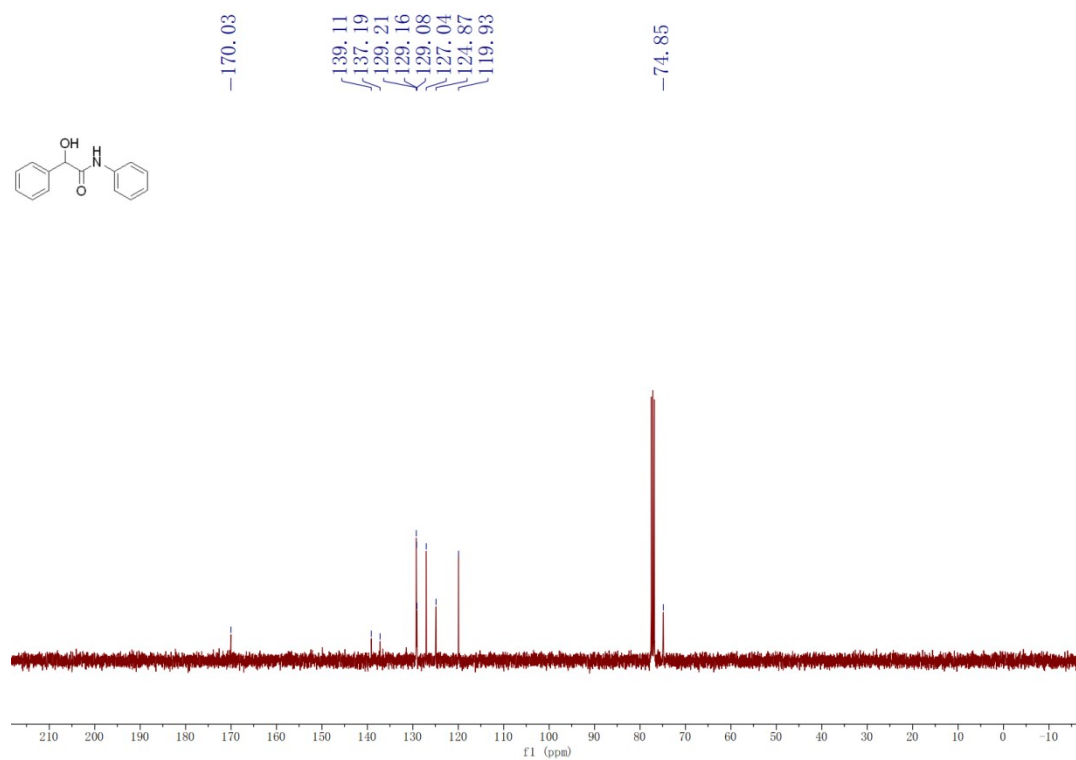
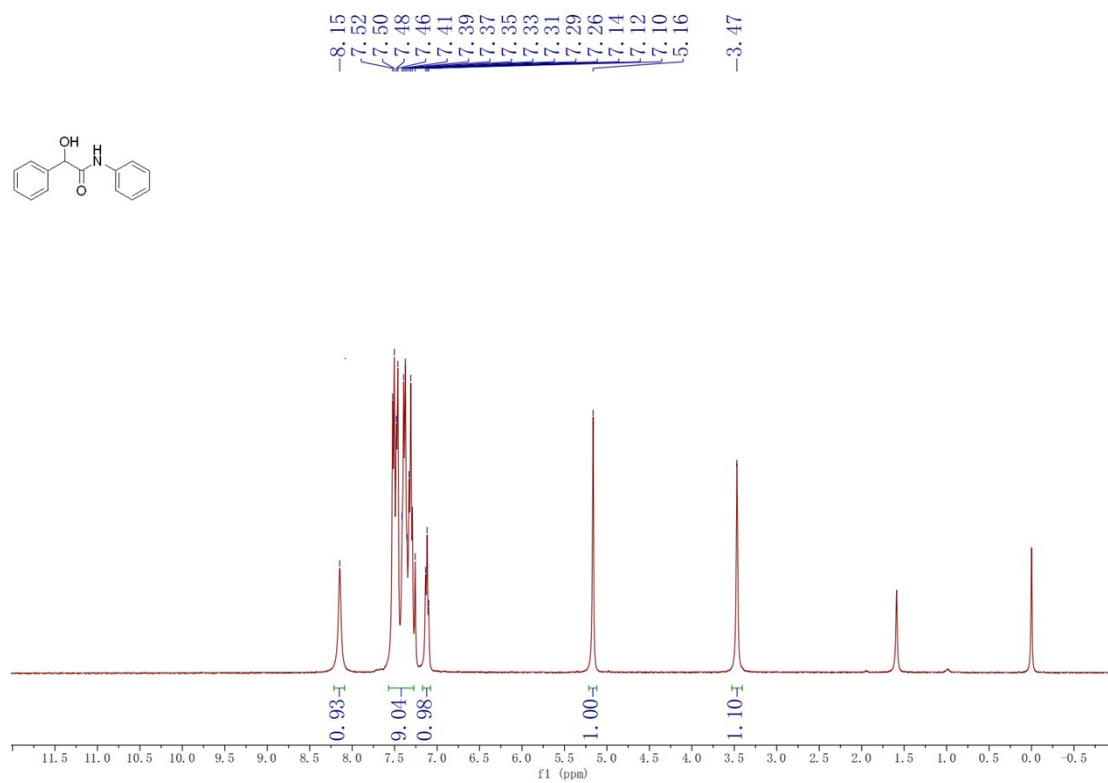
2-hydroxy-3,3-dimethyl-N-phenylbutanamide (44b). The title compound was prepared according to the general procedure and purified by column chromatography (petroleum ether/ethylacetate = 3:1) to obtain a white solid product (46 mg, 74% yield). ^1H NMR (400 MHz, DMSO- d_6) δ 9.52 (s, 1H), 7.68 (d, 2H), 7.28 (t, 2H), 7.04 (t, 1H), 5.71 (d, J = 5.6 Hz, 1H), 3.67 (d, J = 5.6 Hz, 1H), 0.96 (s, 9H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 171.65, 138.50, 128.54, 123.28, 119.71, 78.78, 34.75, 26.20. HRMS m/z (ESI) calcd for $\text{C}_{12}\text{H}_{18}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 208.1332, found 208.1334.

References

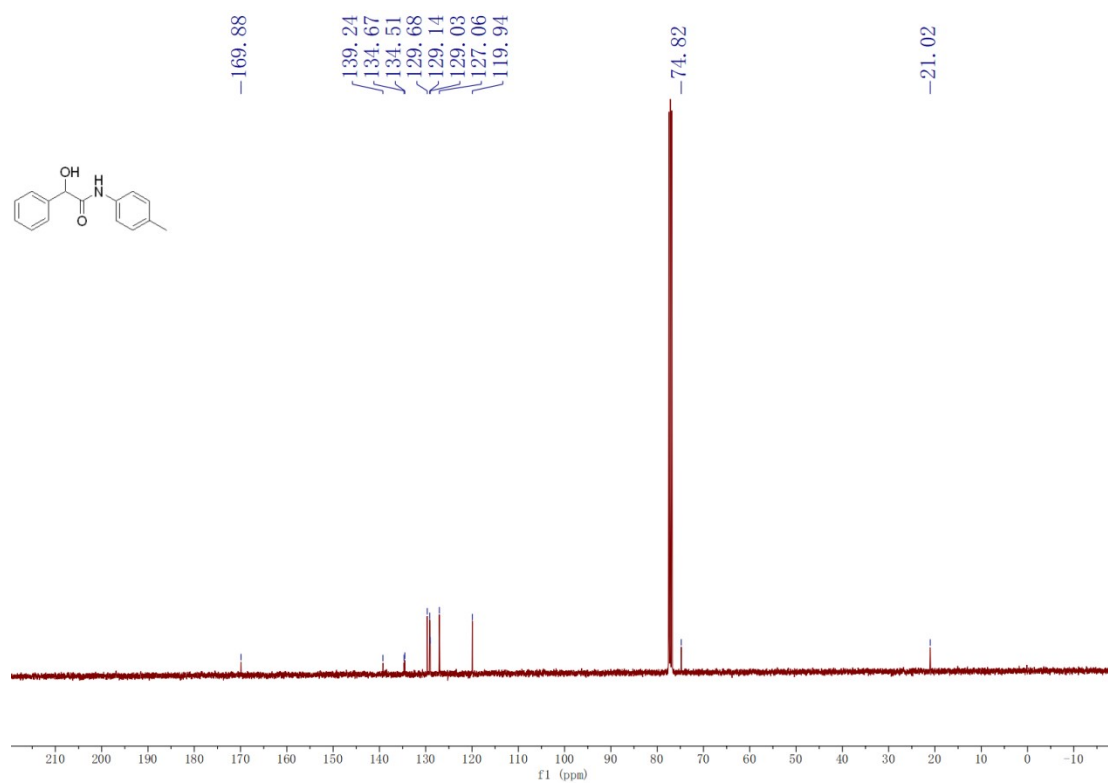
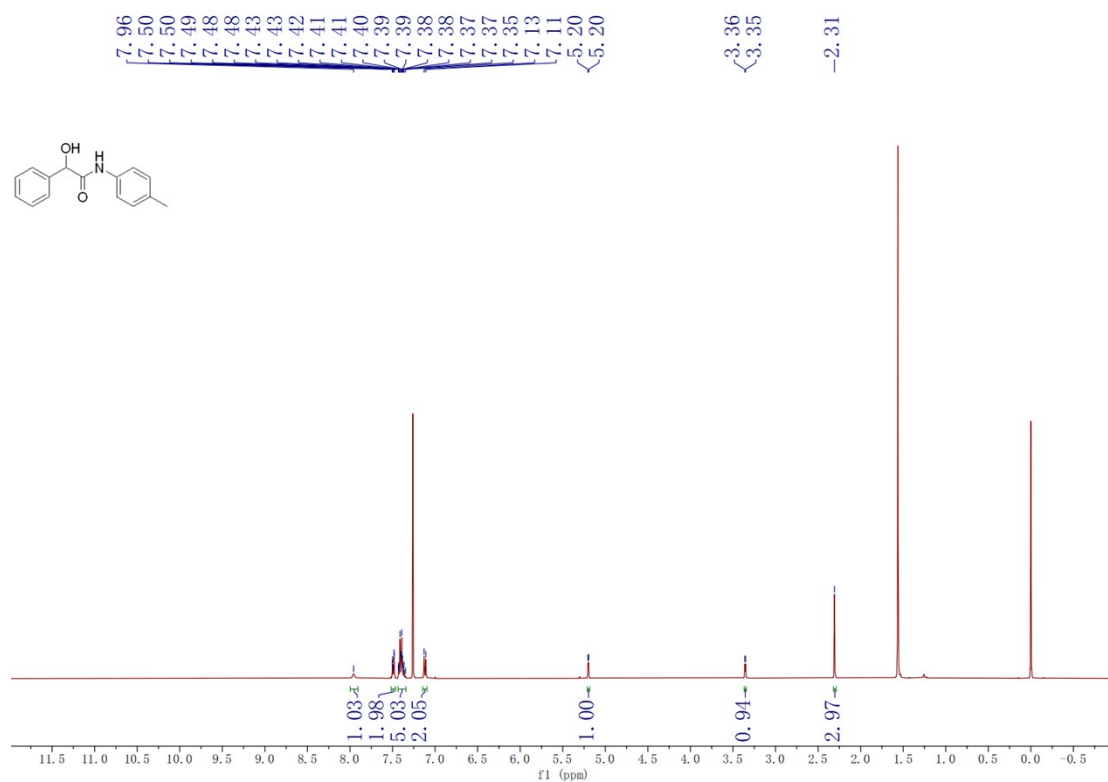
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10. Copies of ^1H NMR, ^{13}C NMR spectra

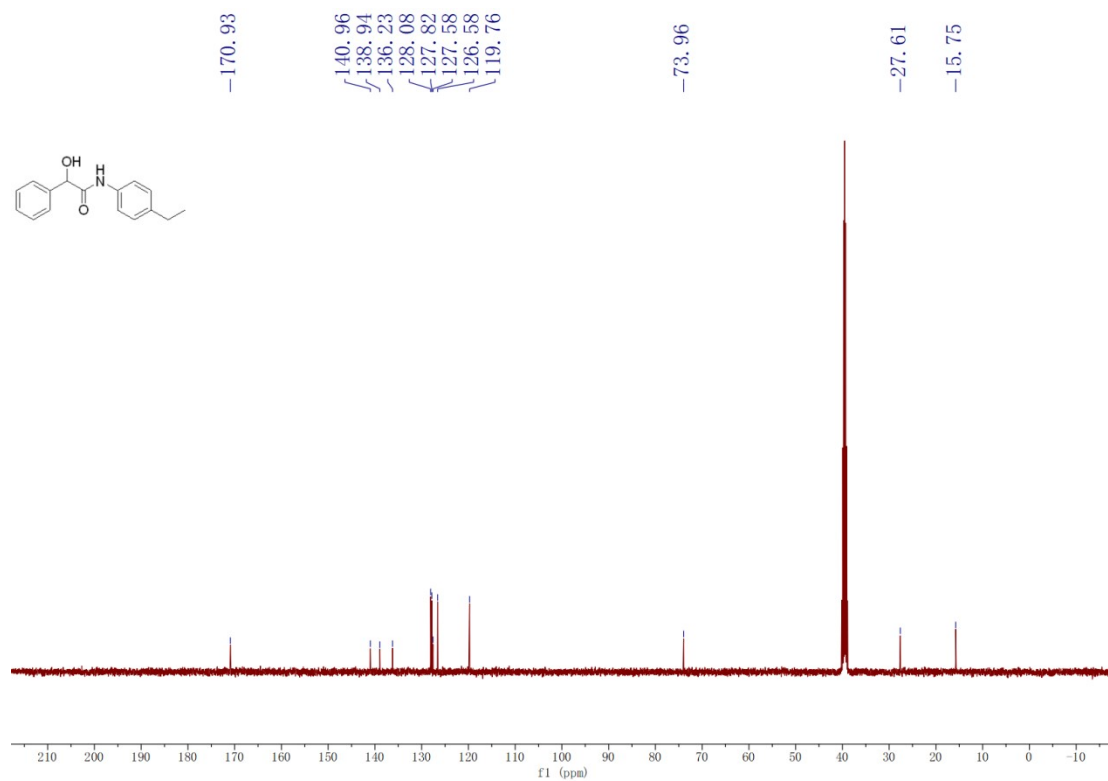
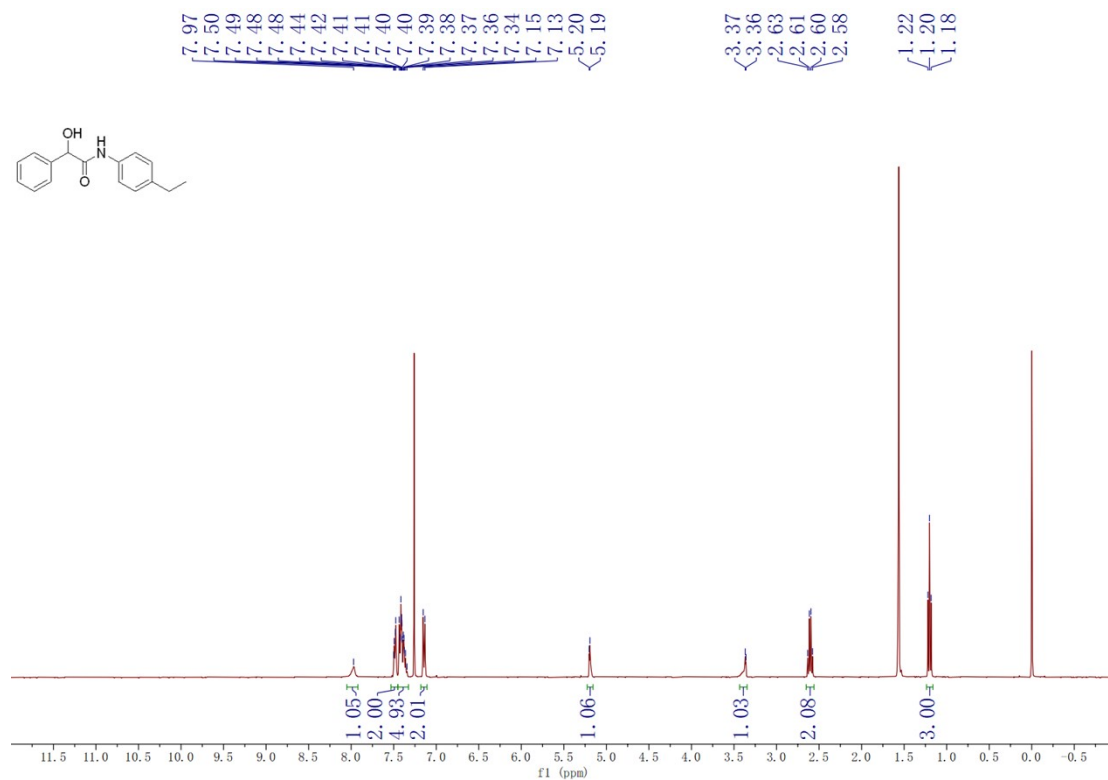
(1) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **1b**



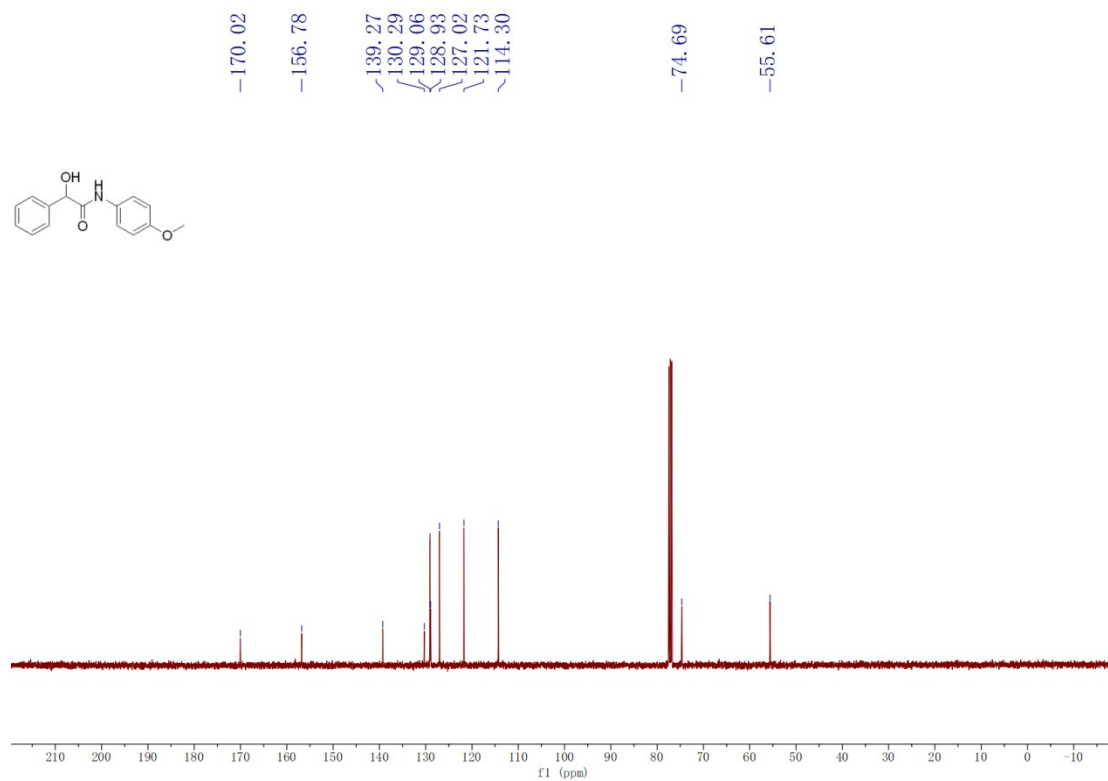
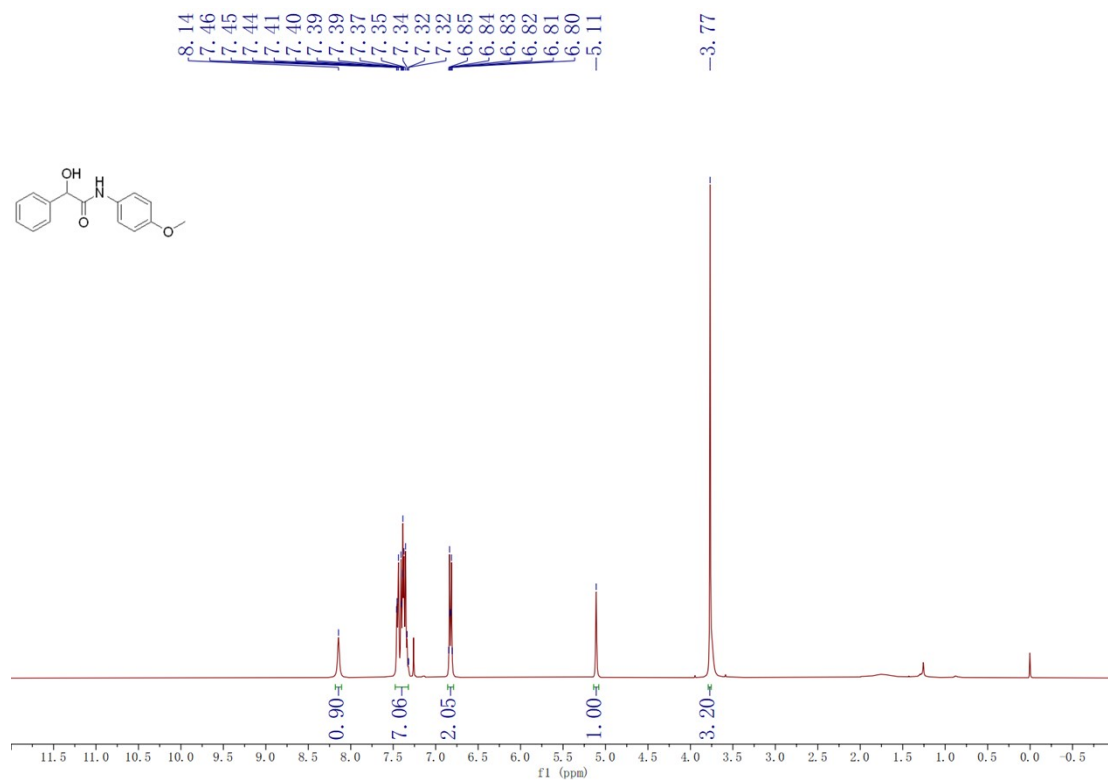
(2) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **2b**



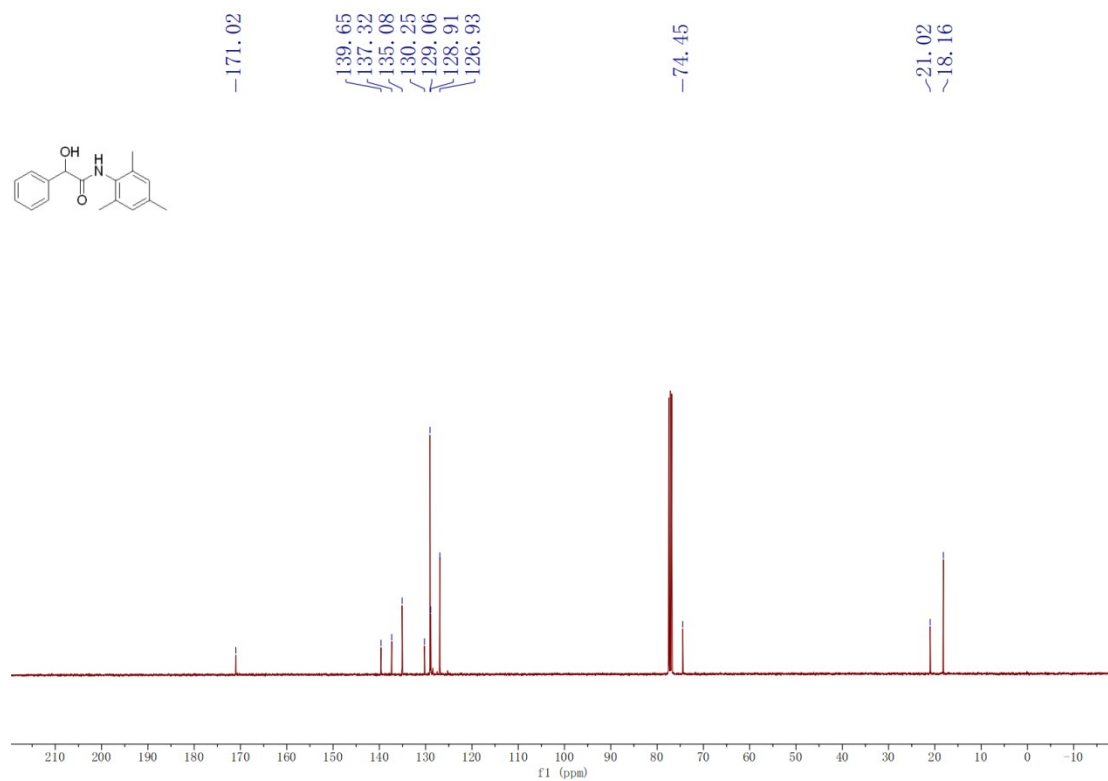
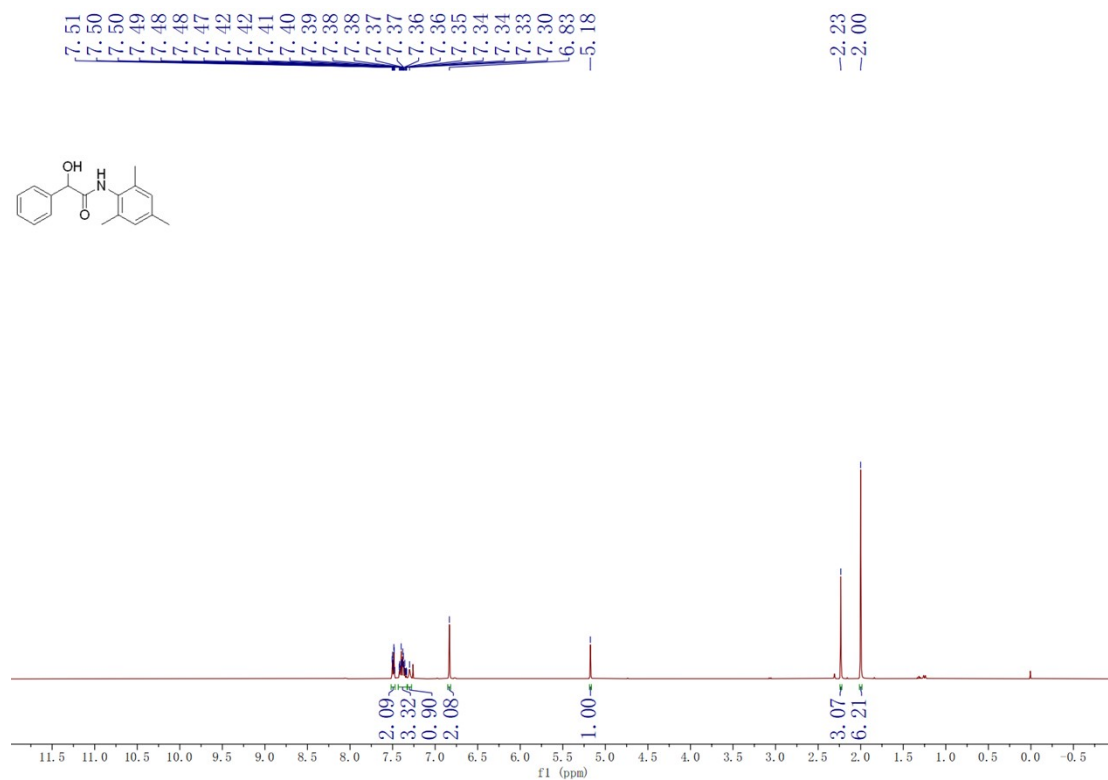
(3) ^1H NMR (CDCl_3) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **3b**



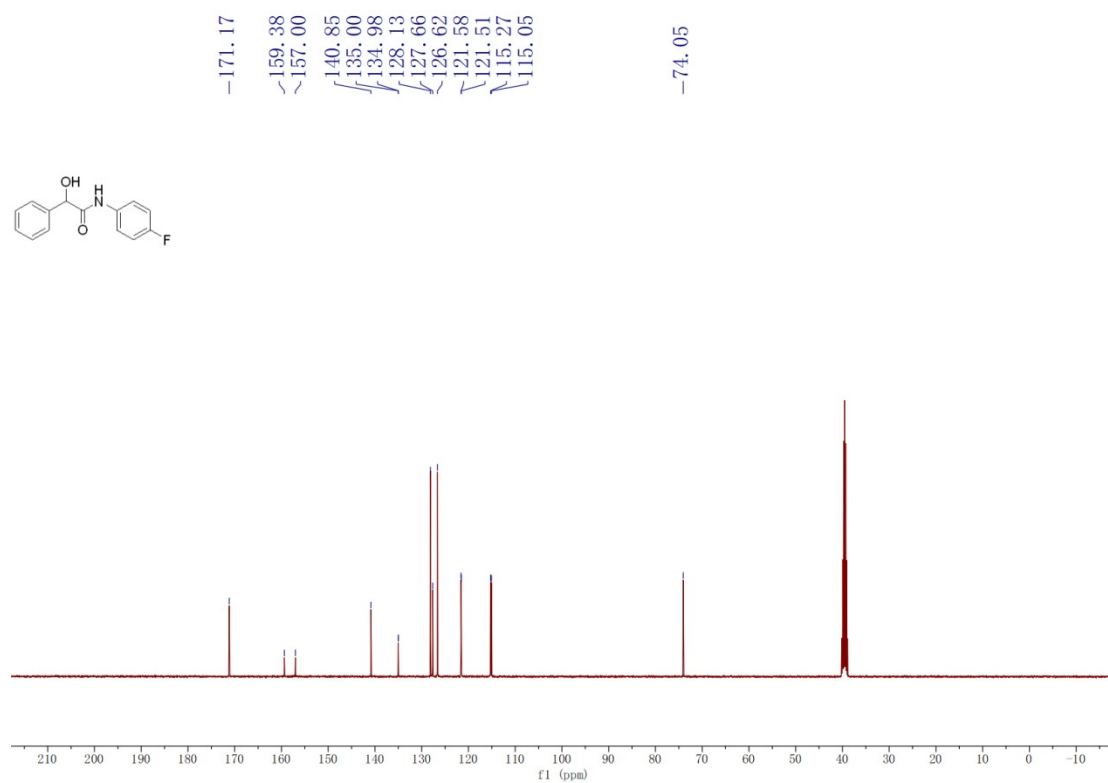
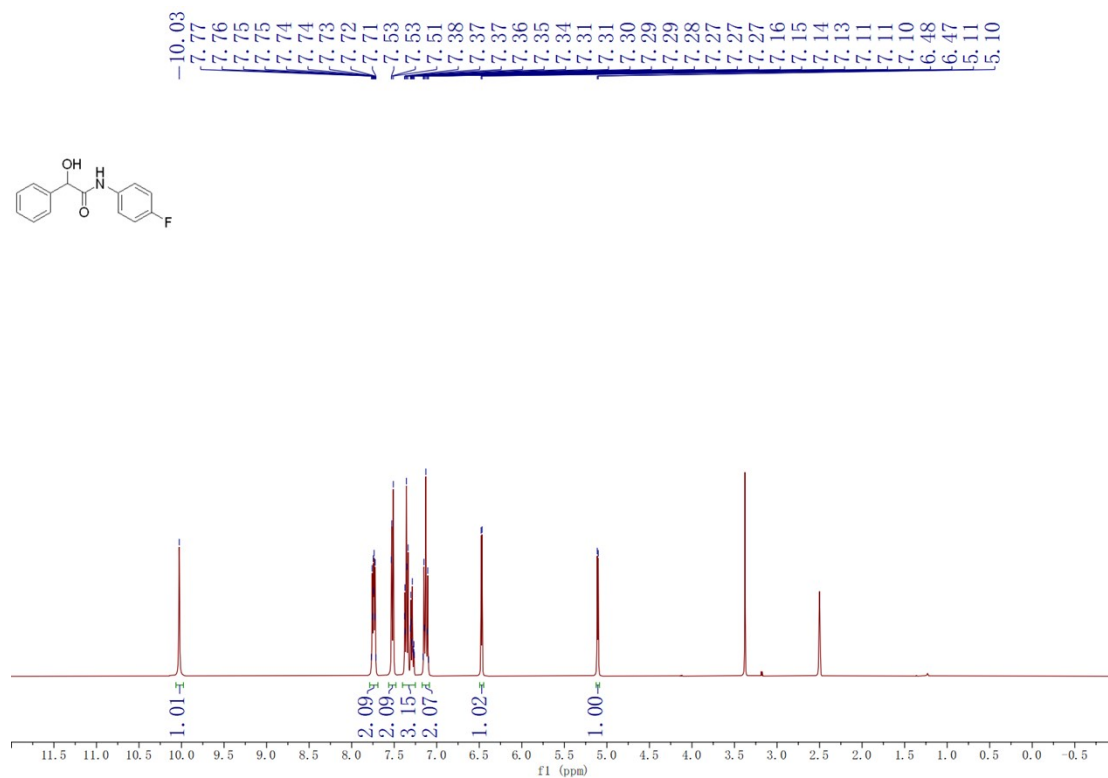
(4) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **4b**



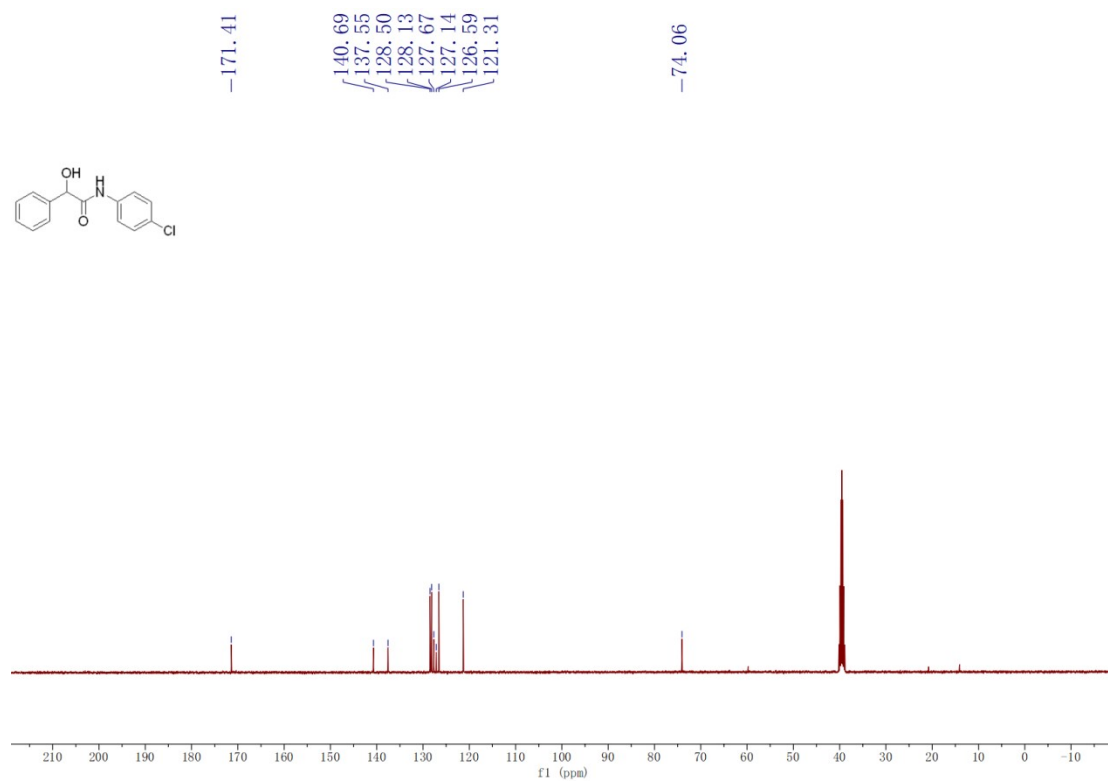
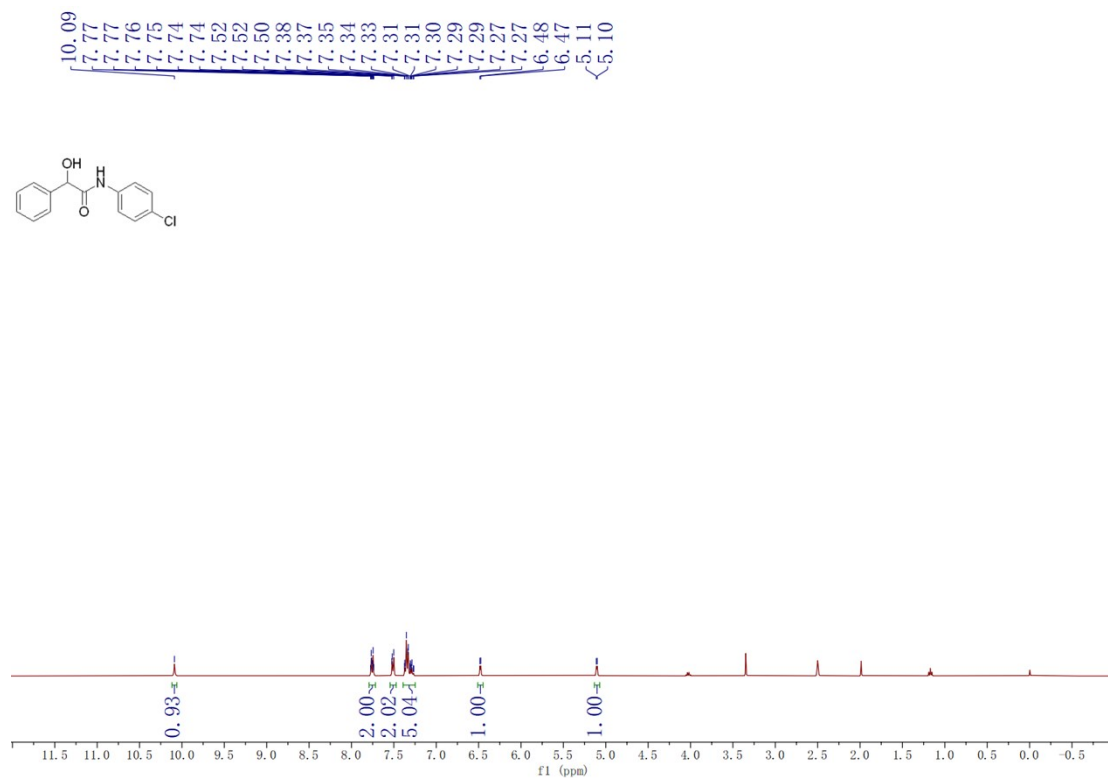
(5) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **5b**



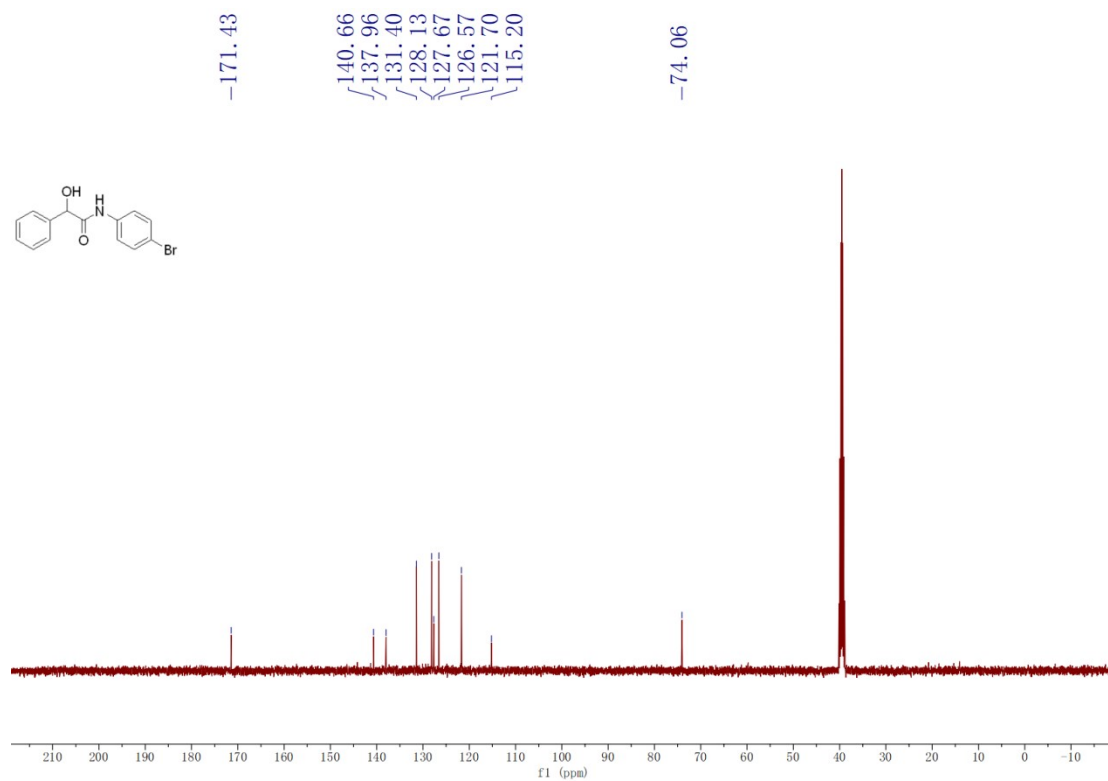
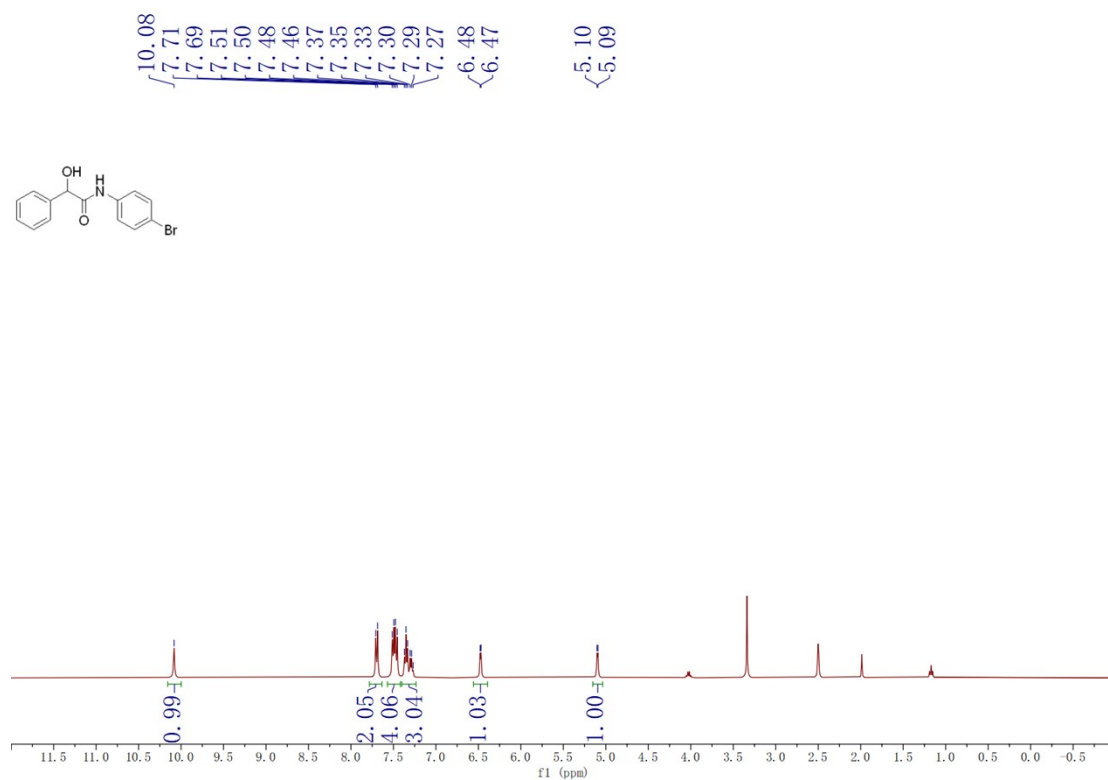
(6) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **6b**



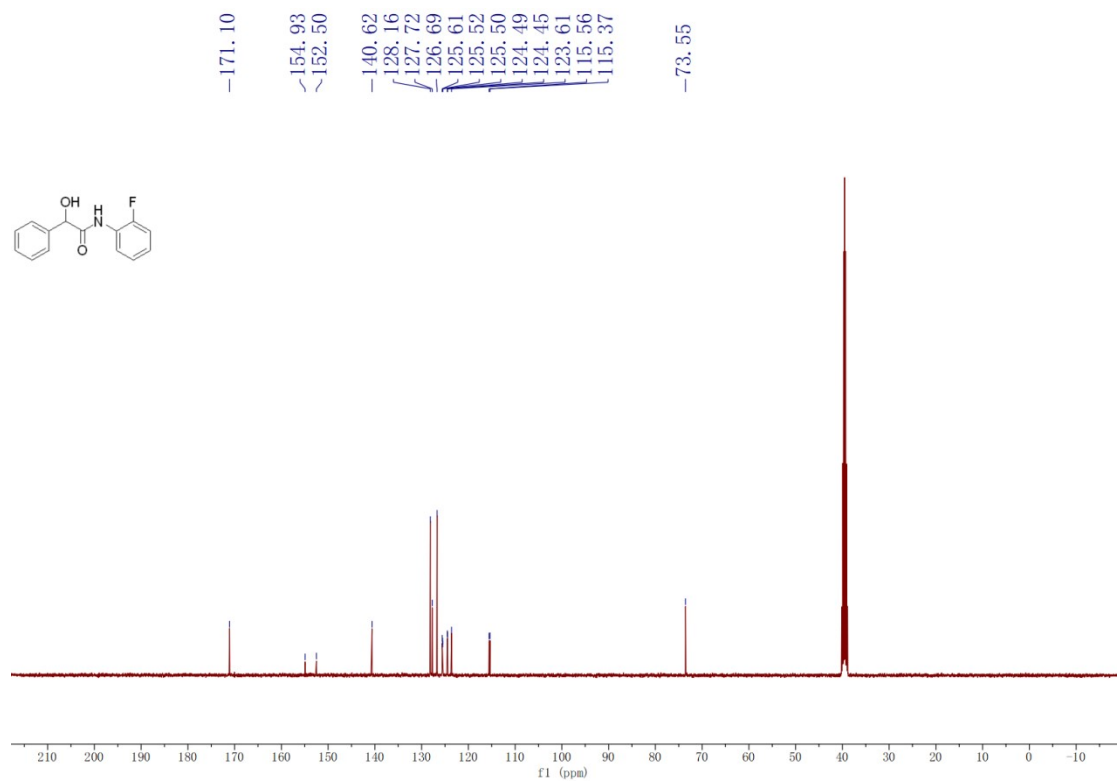
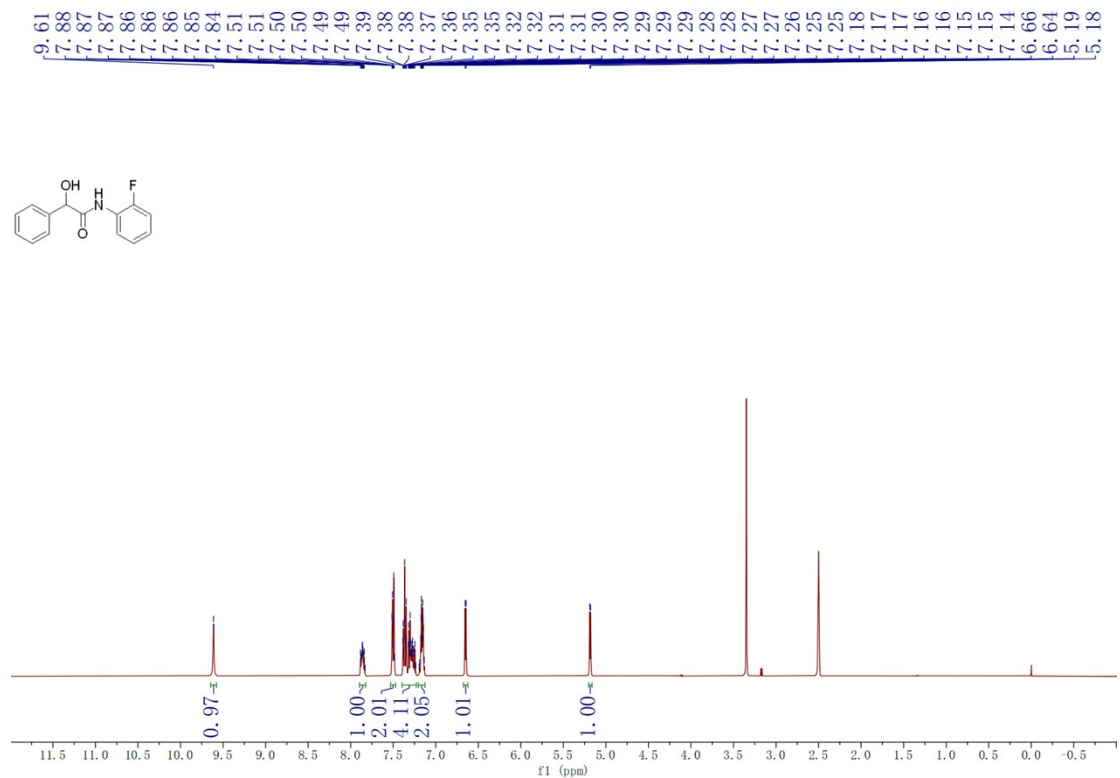
(7) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **7b**



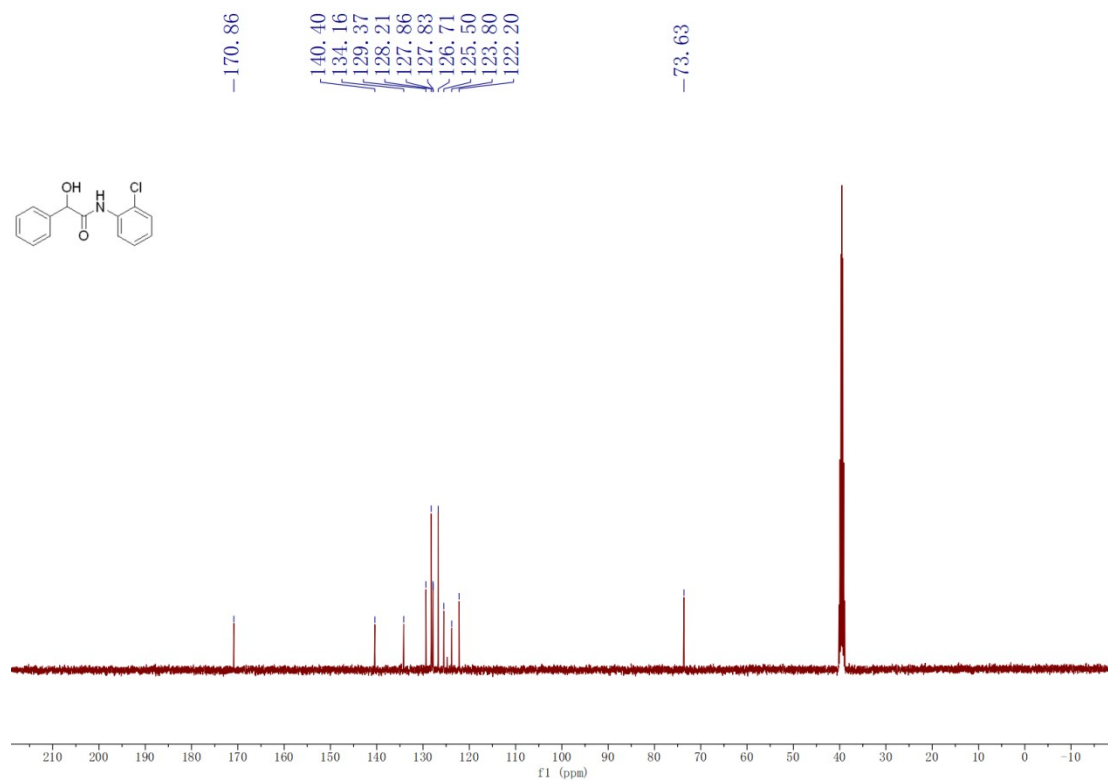
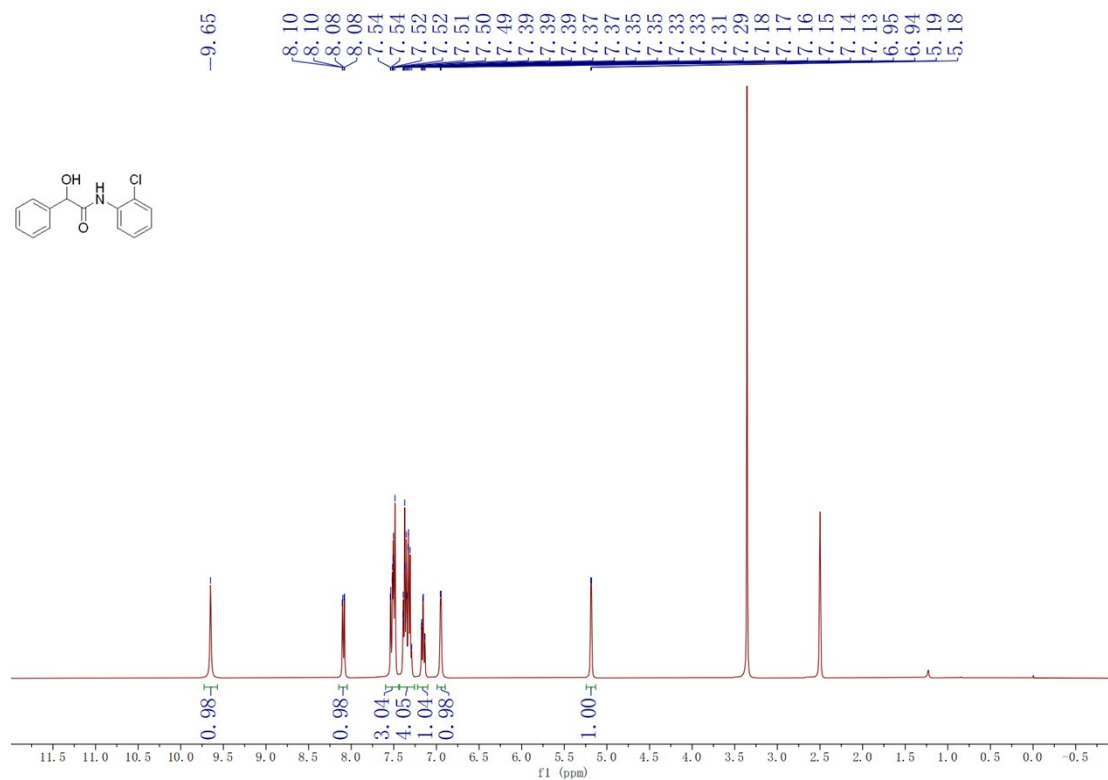
(8) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **8b**



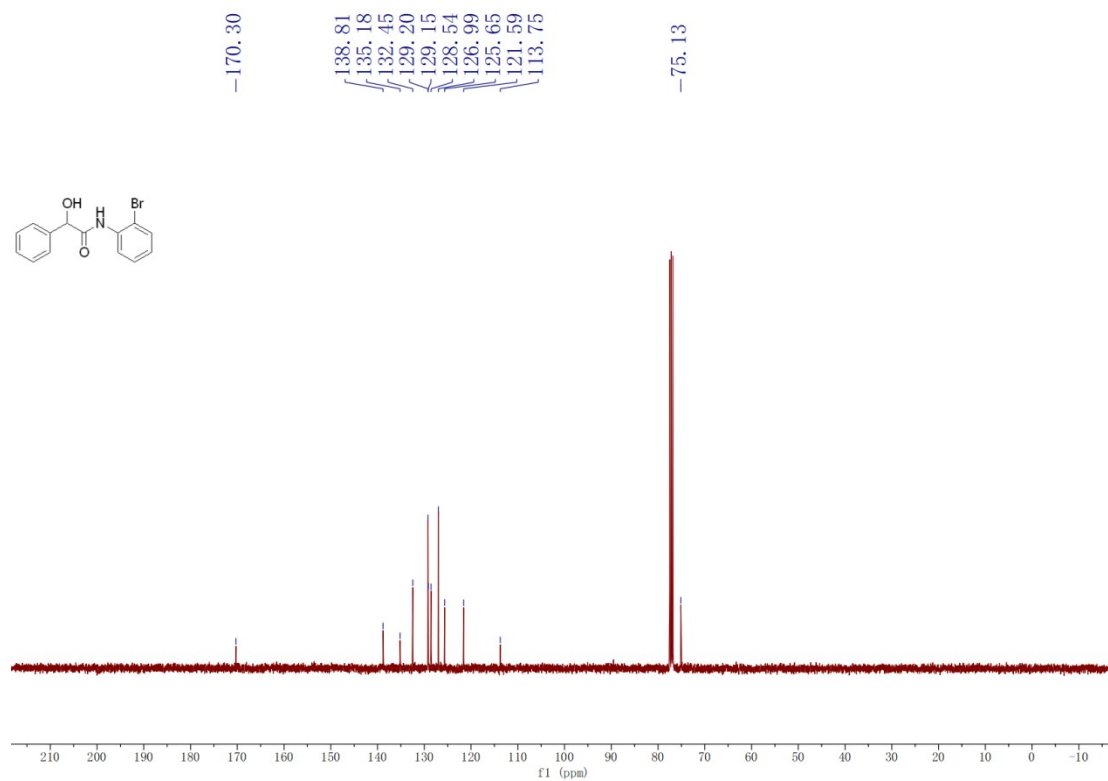
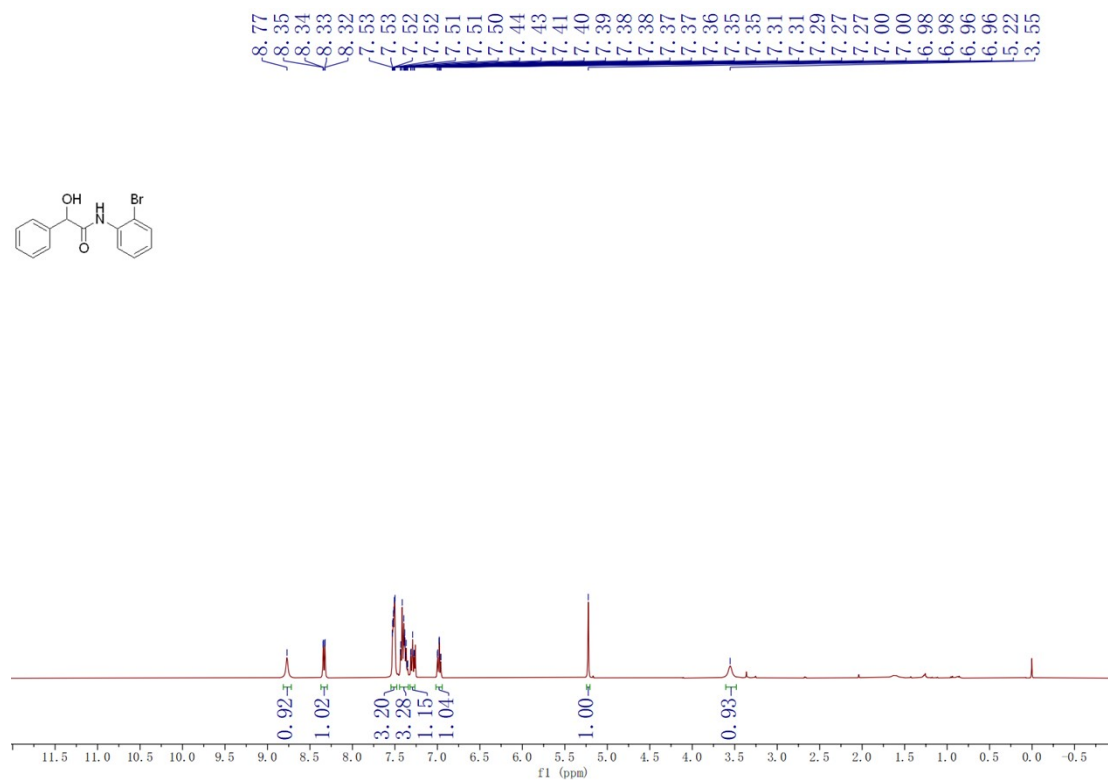
(9) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **9b**



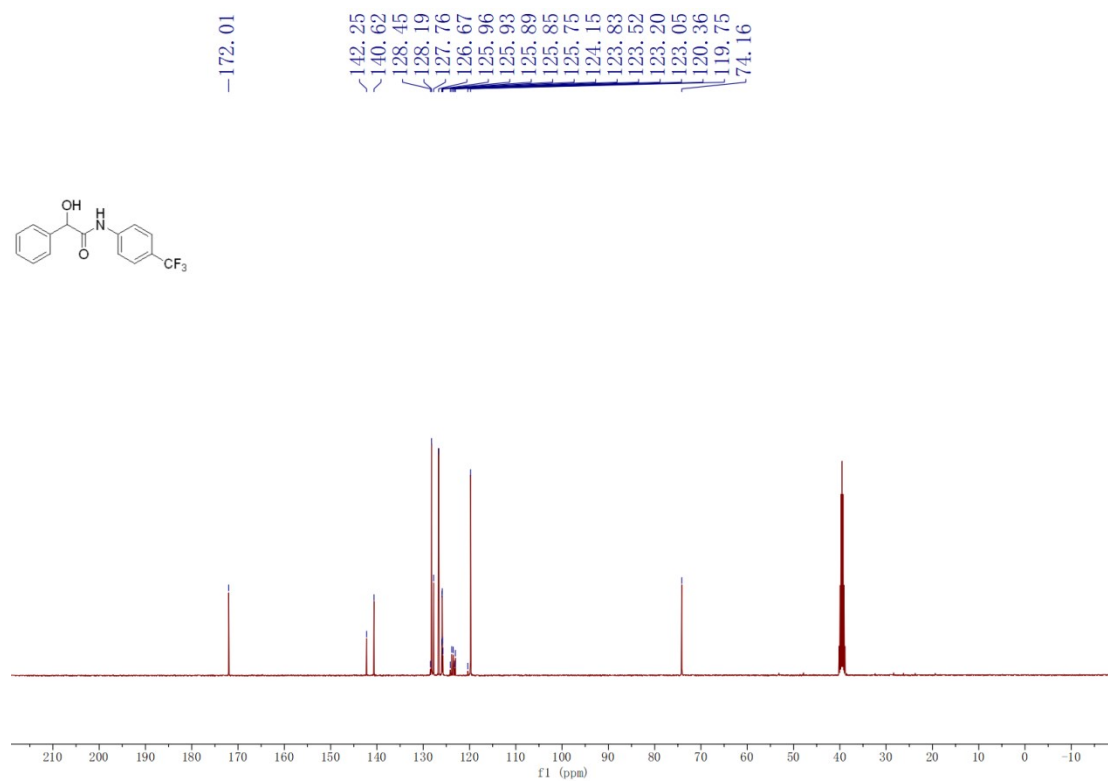
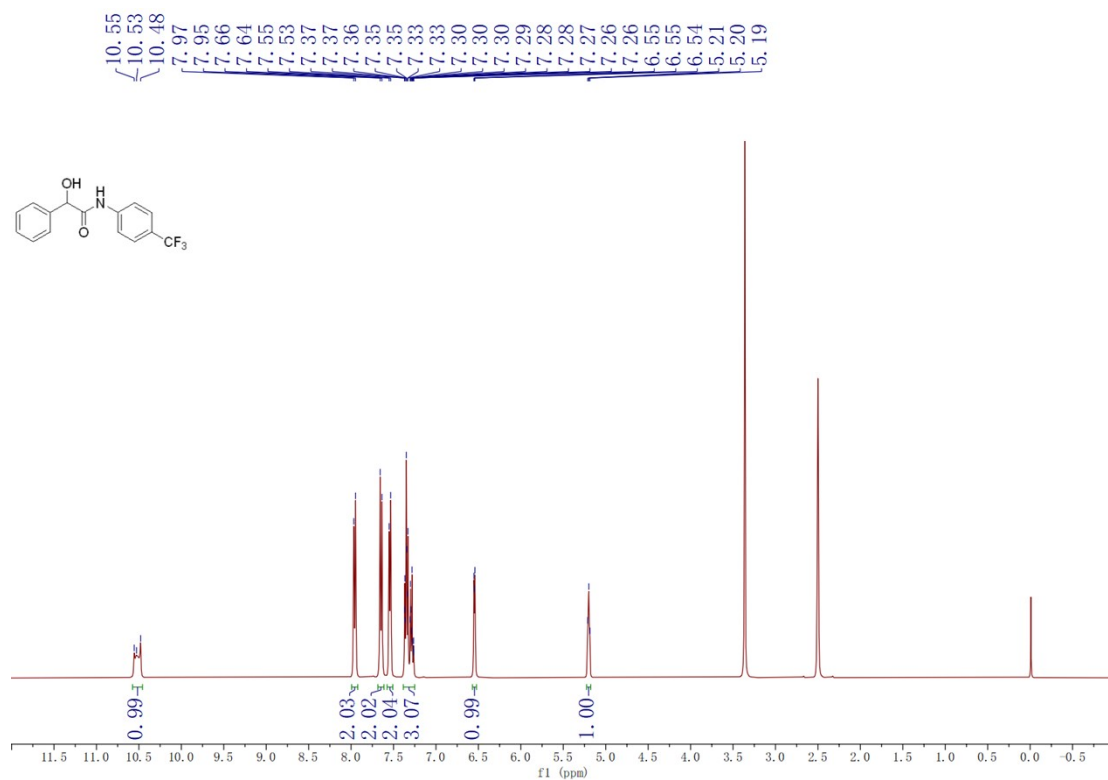
(10) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **10b**



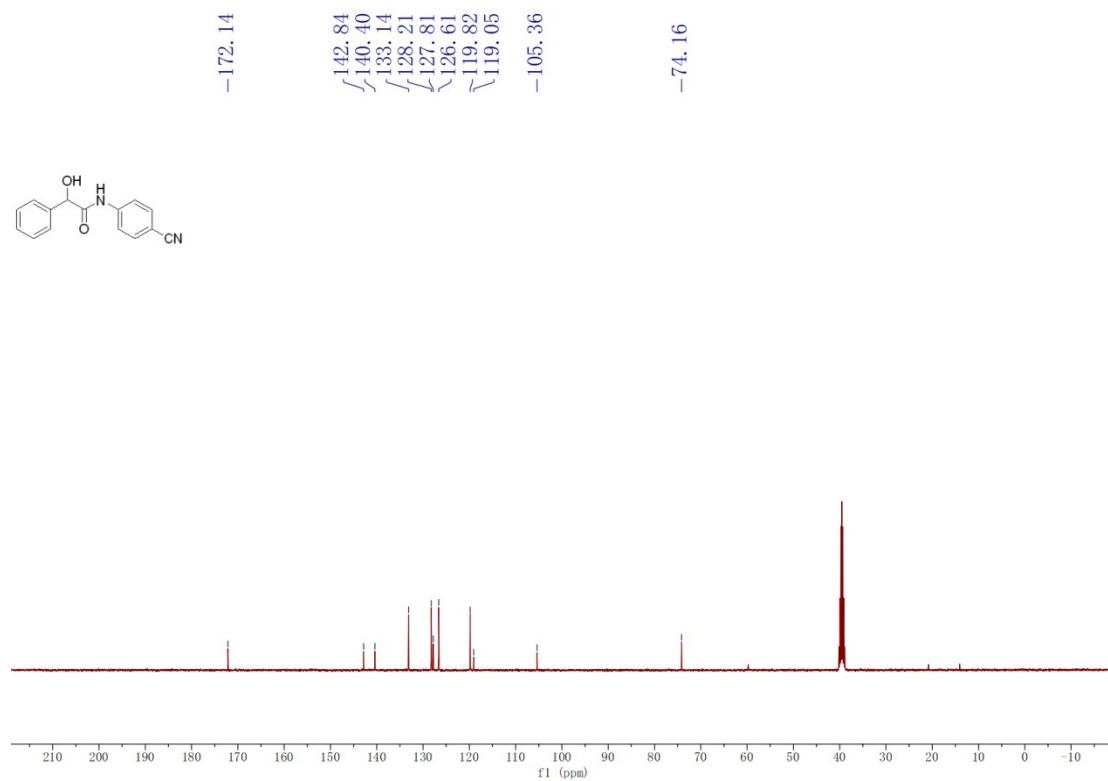
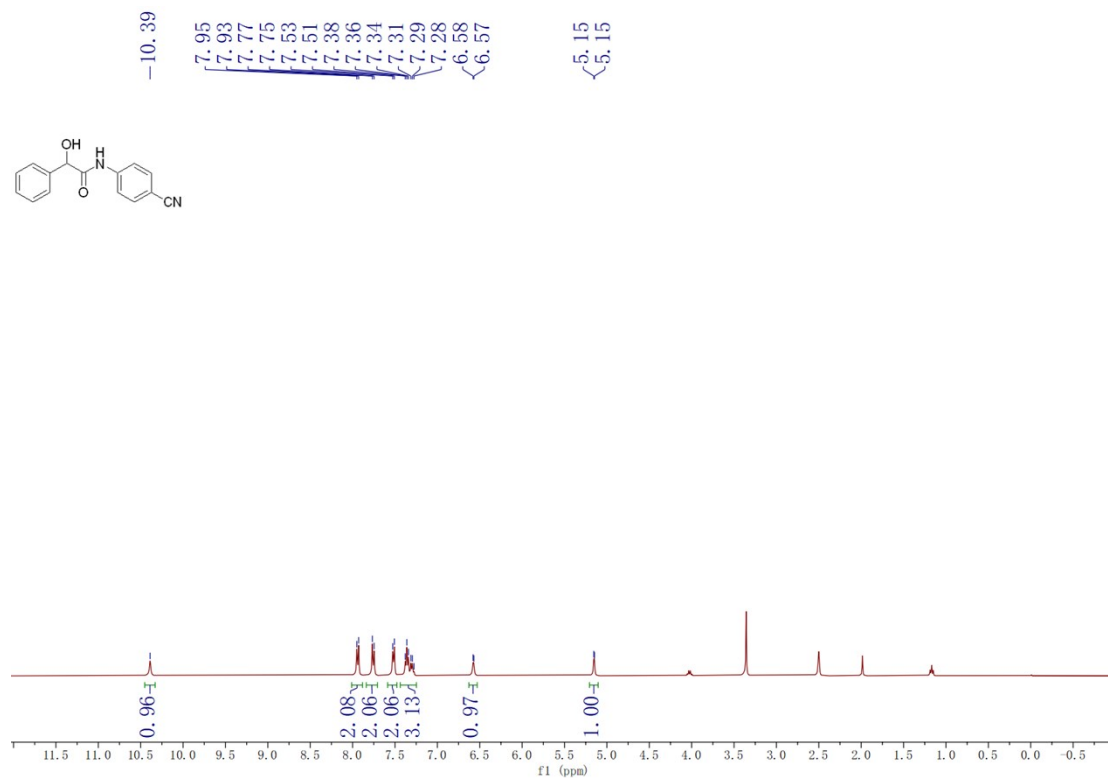
(¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **11b**



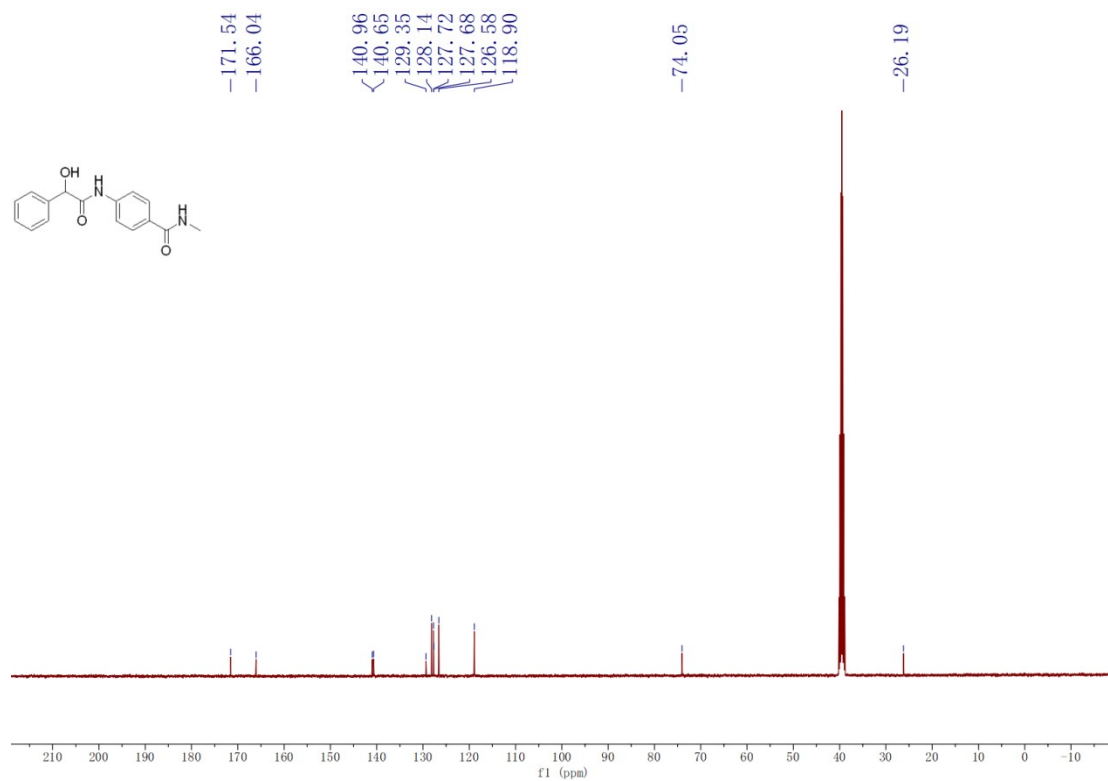
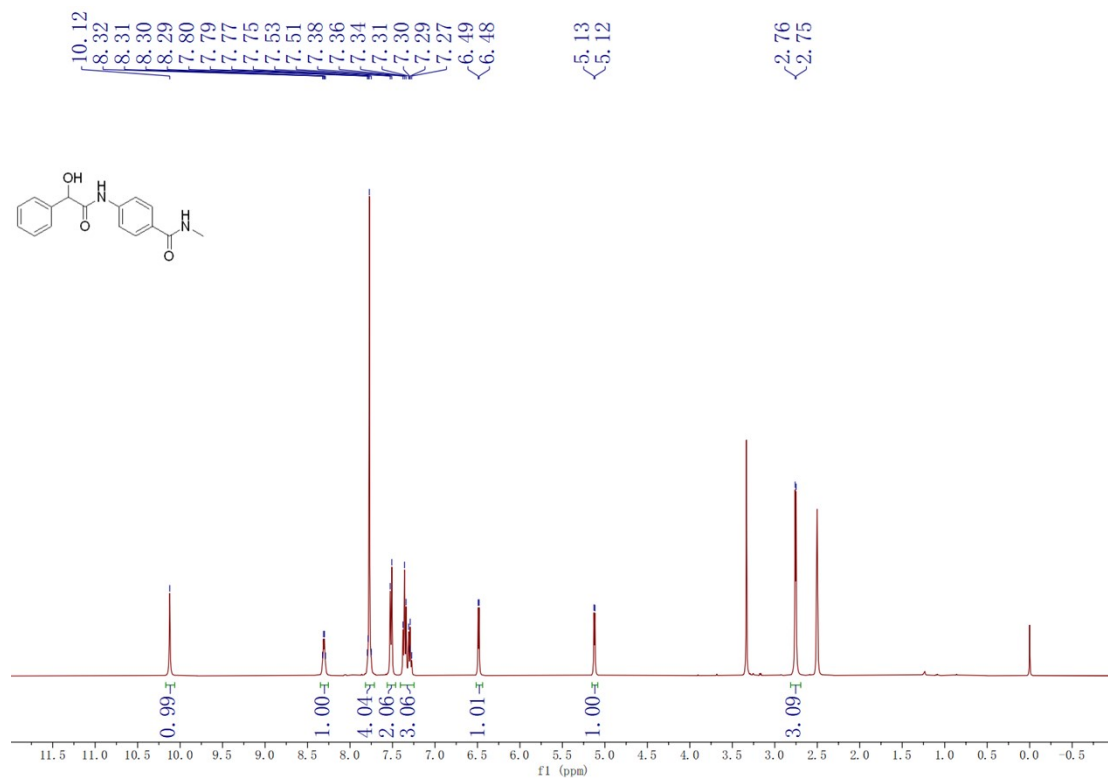
(12) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **12b**



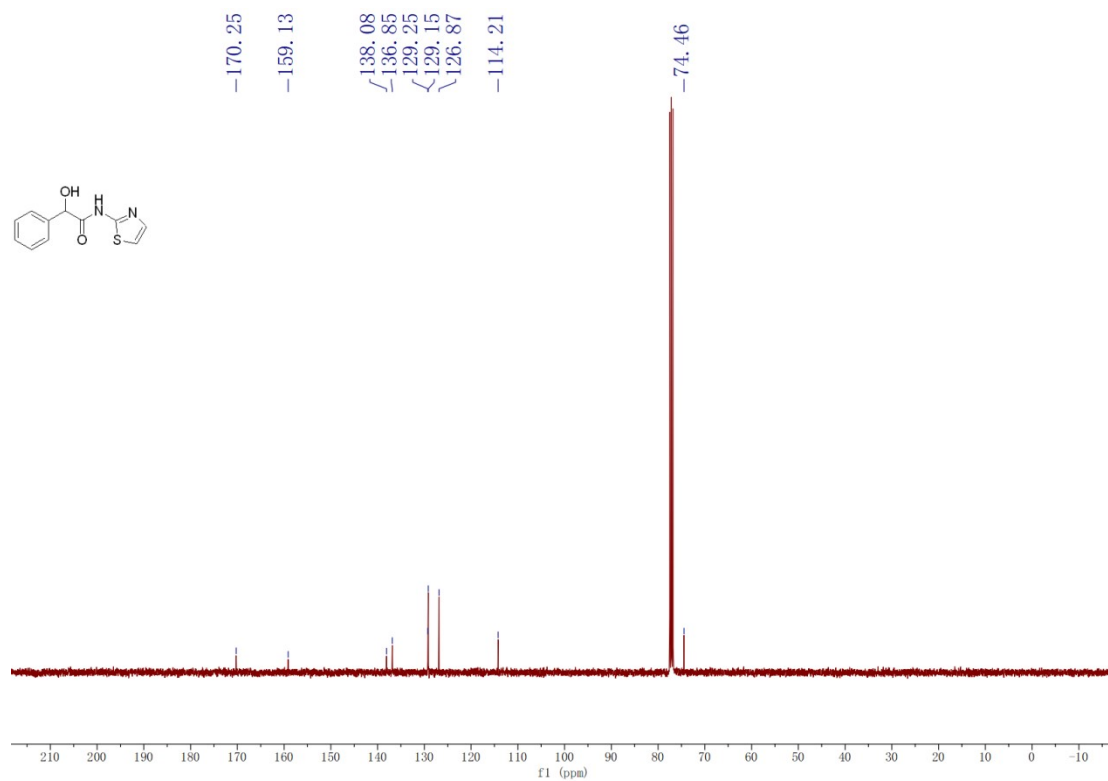
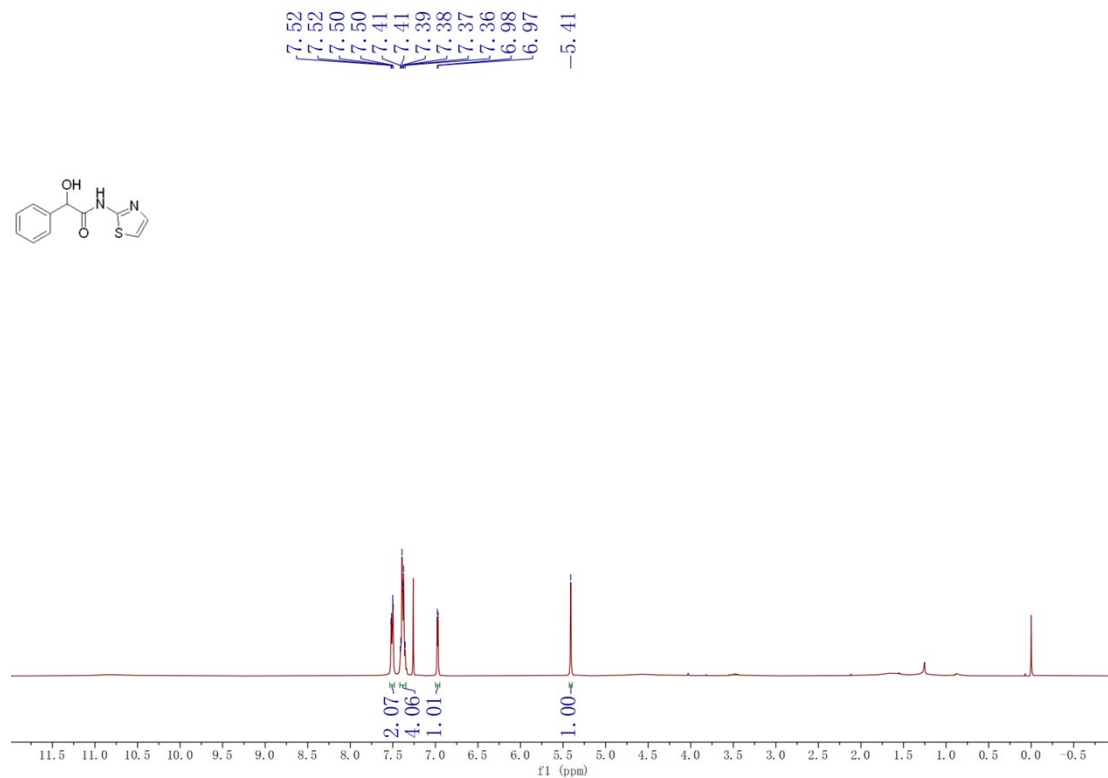
(13)¹H NMR (DMSO-*d*₆) and ¹³C NMR (DMSO-*d*₆) spectrum of **13b**



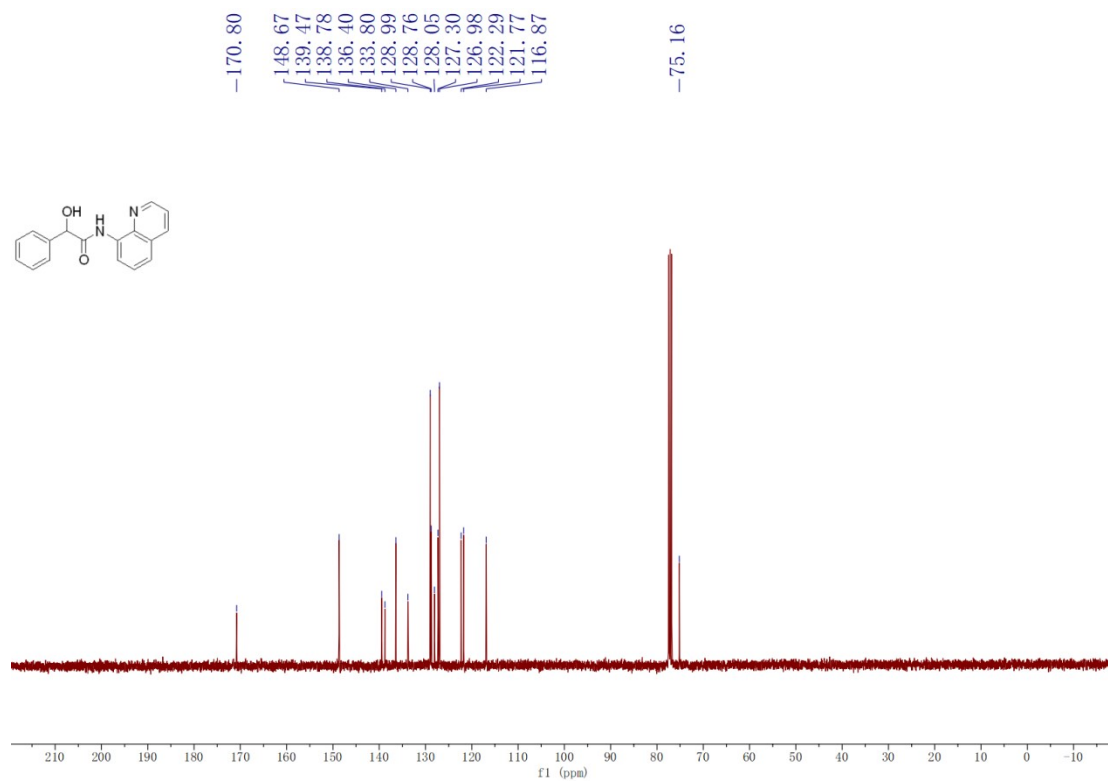
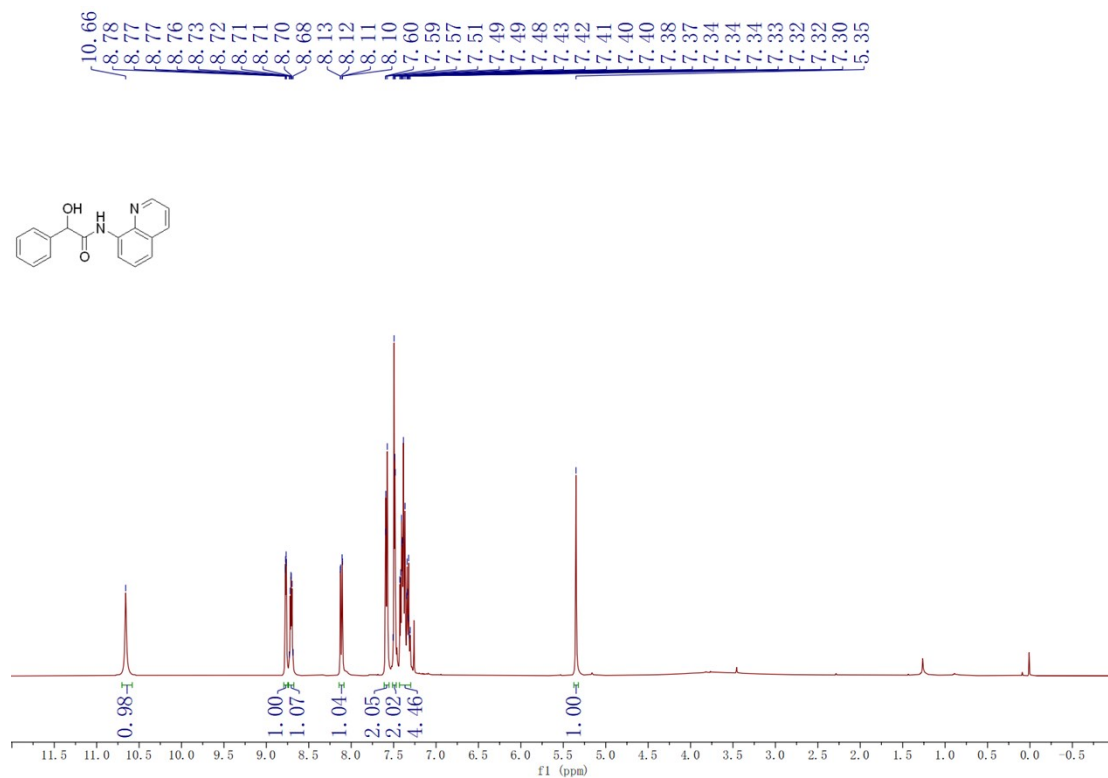
(14)¹H NMR (DMSO-*d*₆) and ¹³C NMR (DMSO-*d*₆) spectrum of **14b**



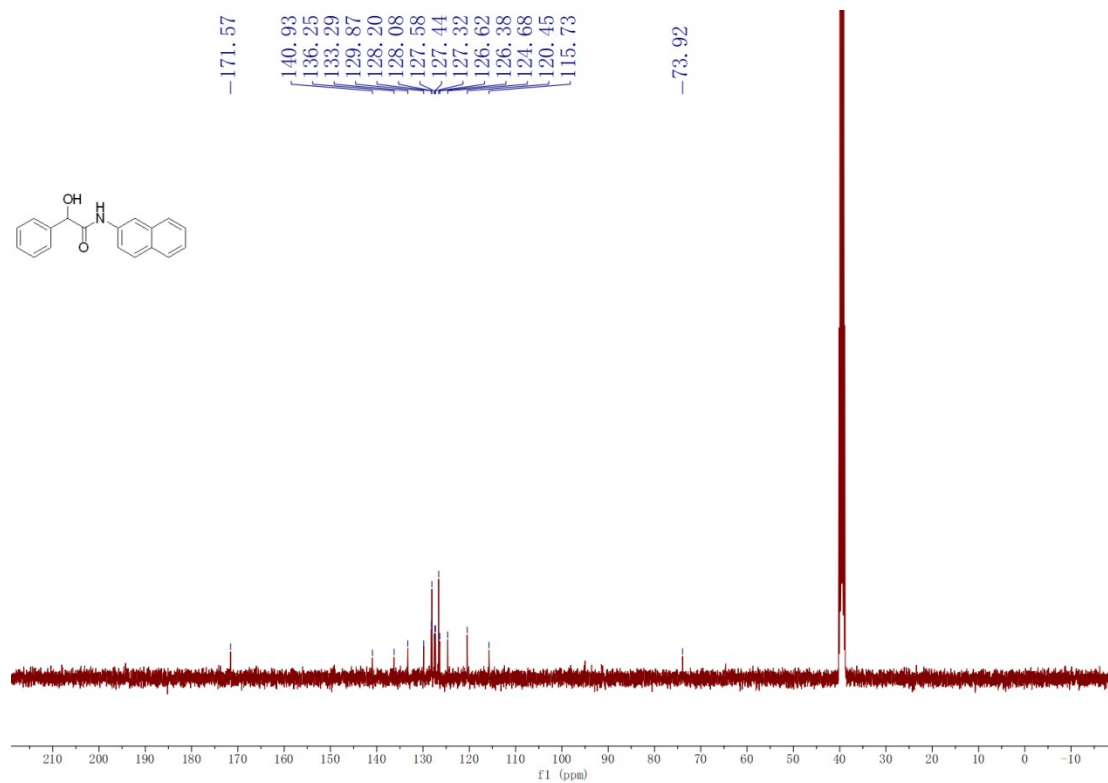
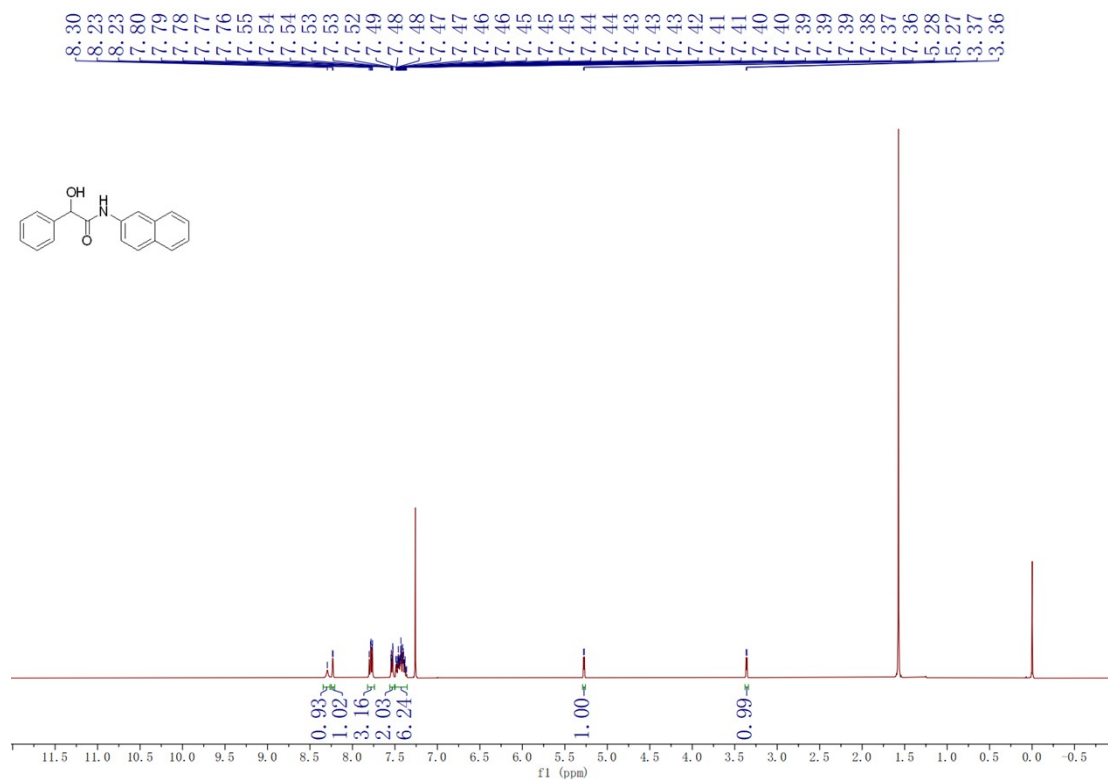
(15)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **15b**



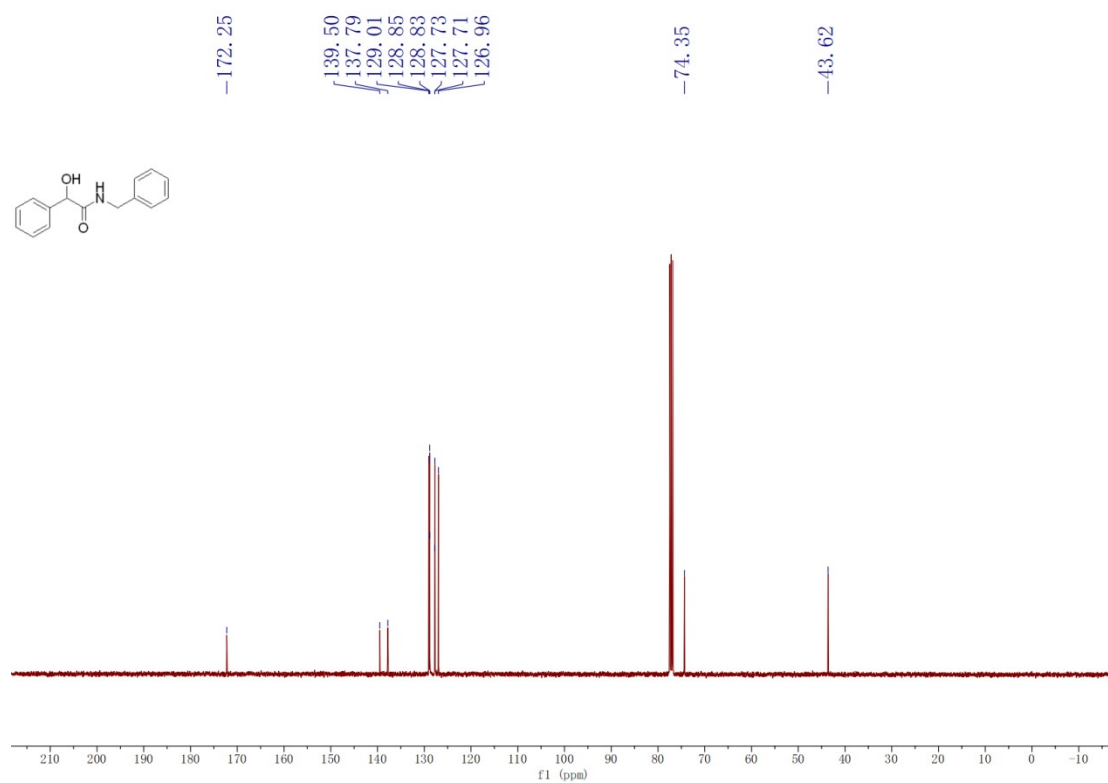
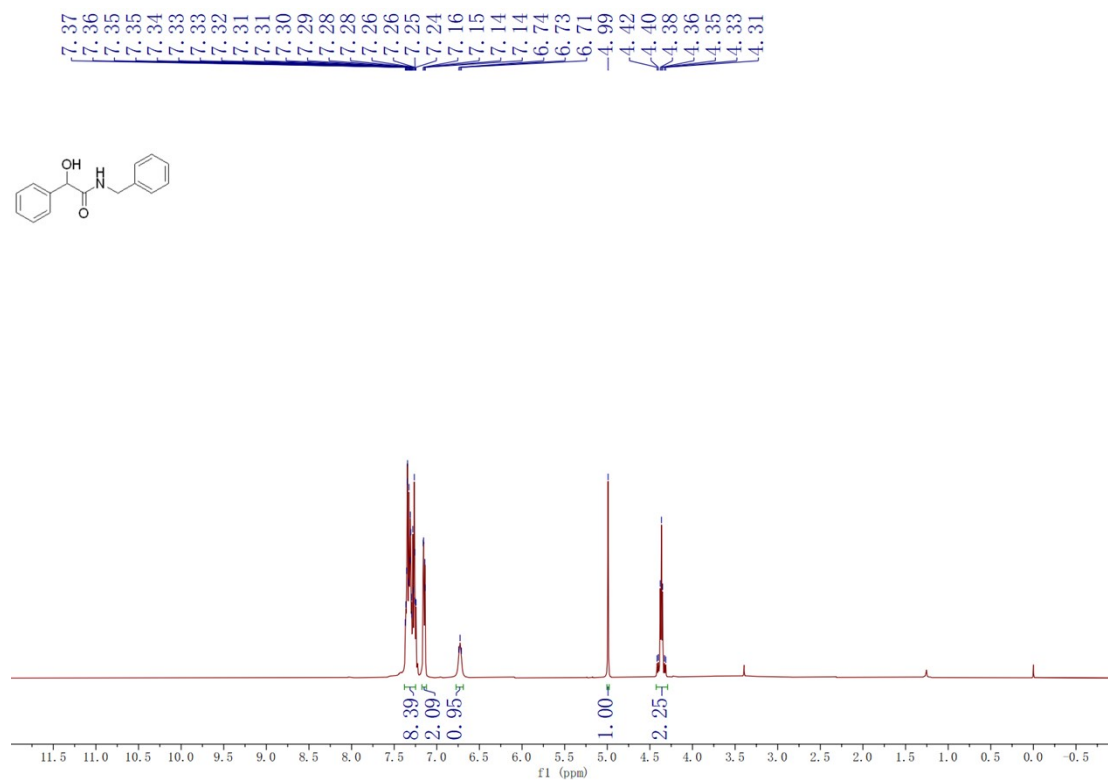
(16)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **16b**



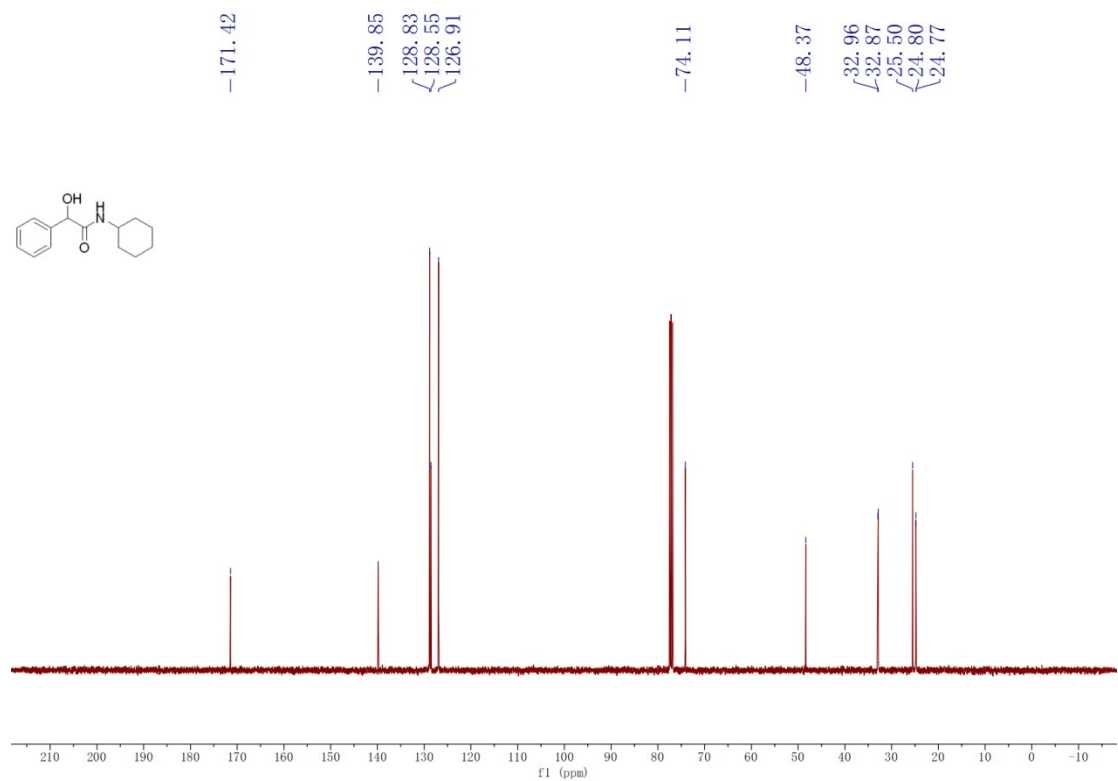
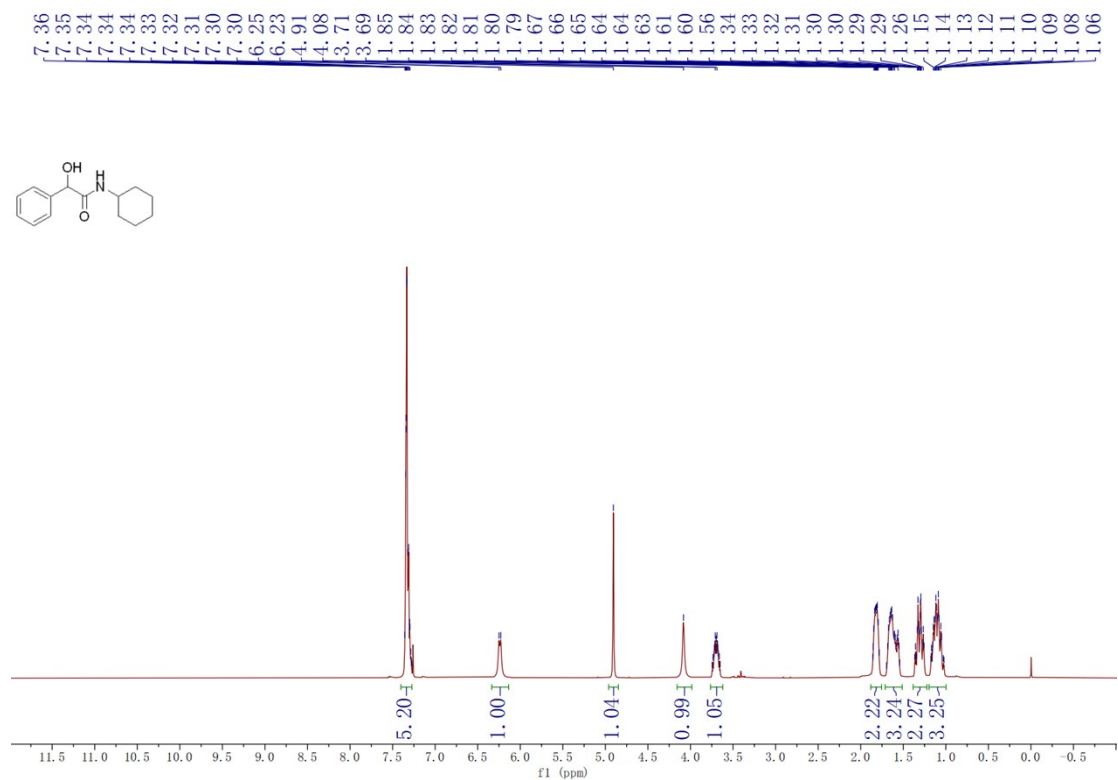
(17)¹H NMR (CDCl₃) and ¹³C NMR (DMSO-*d*₆) spectrum of **17b**



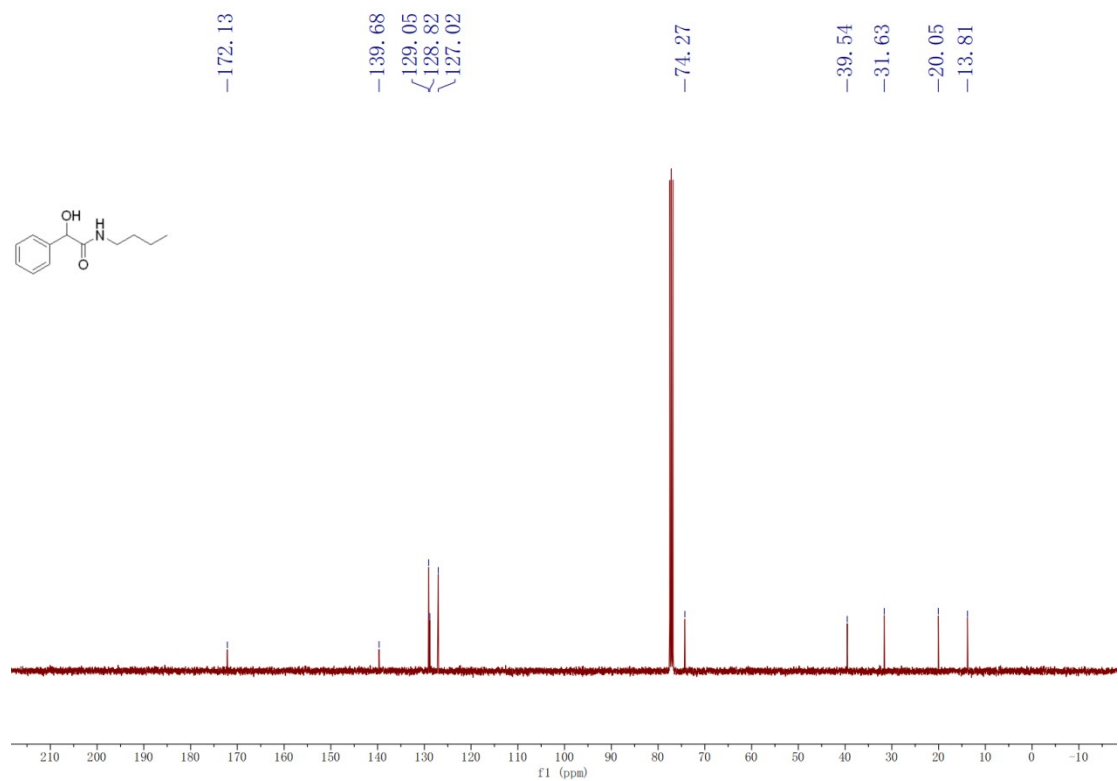
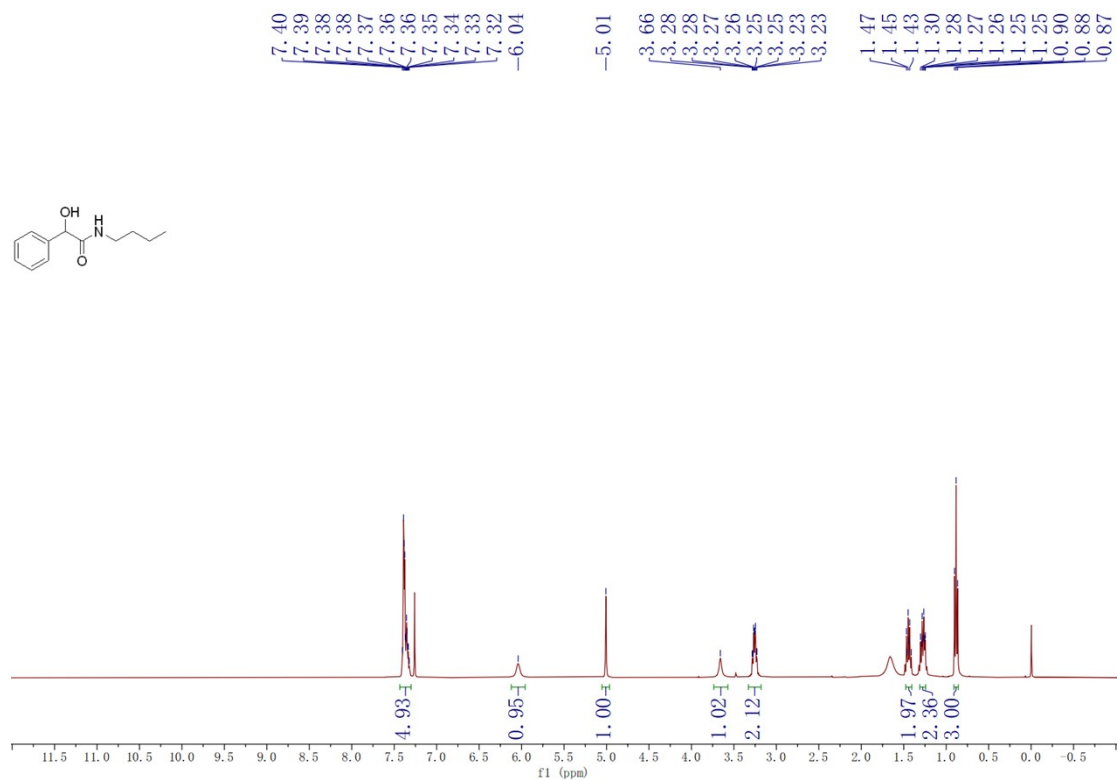
(¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **18b**



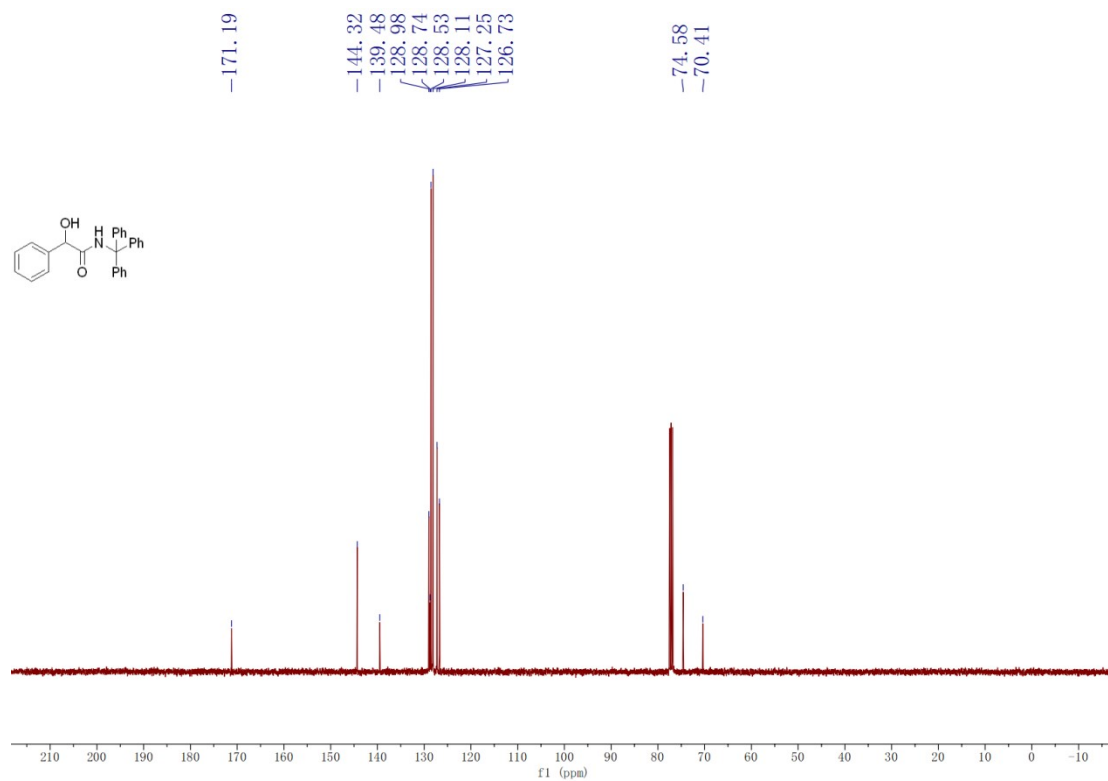
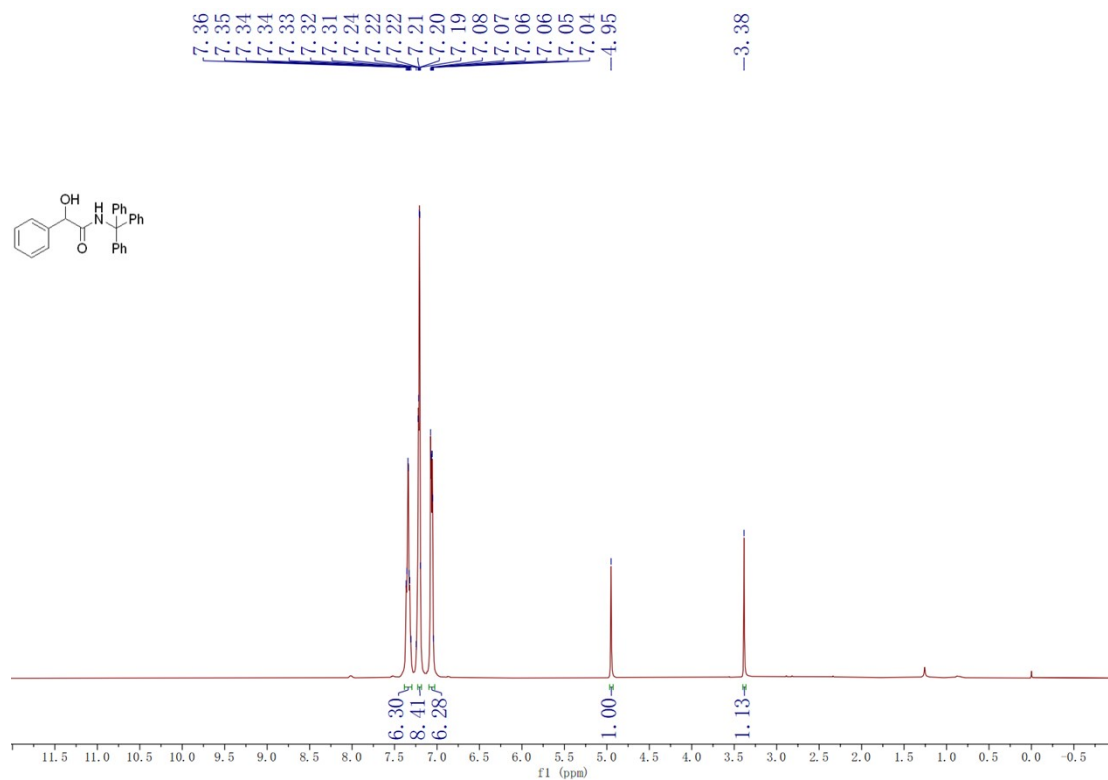
(19)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **19b**



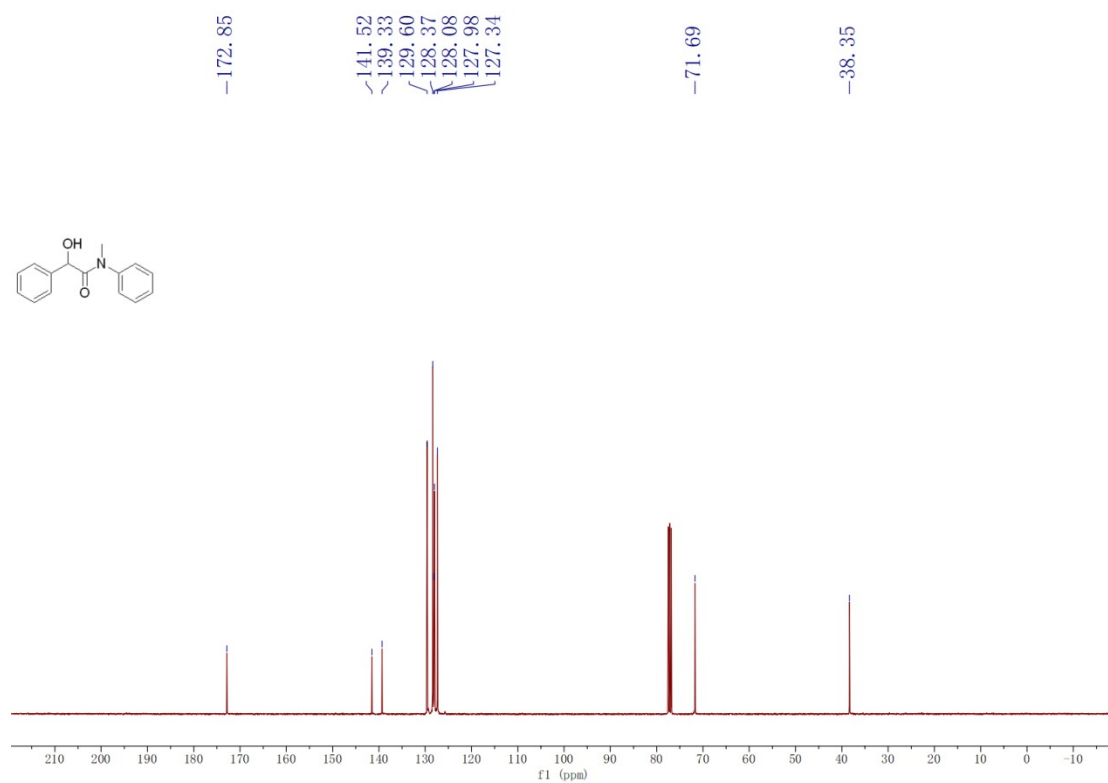
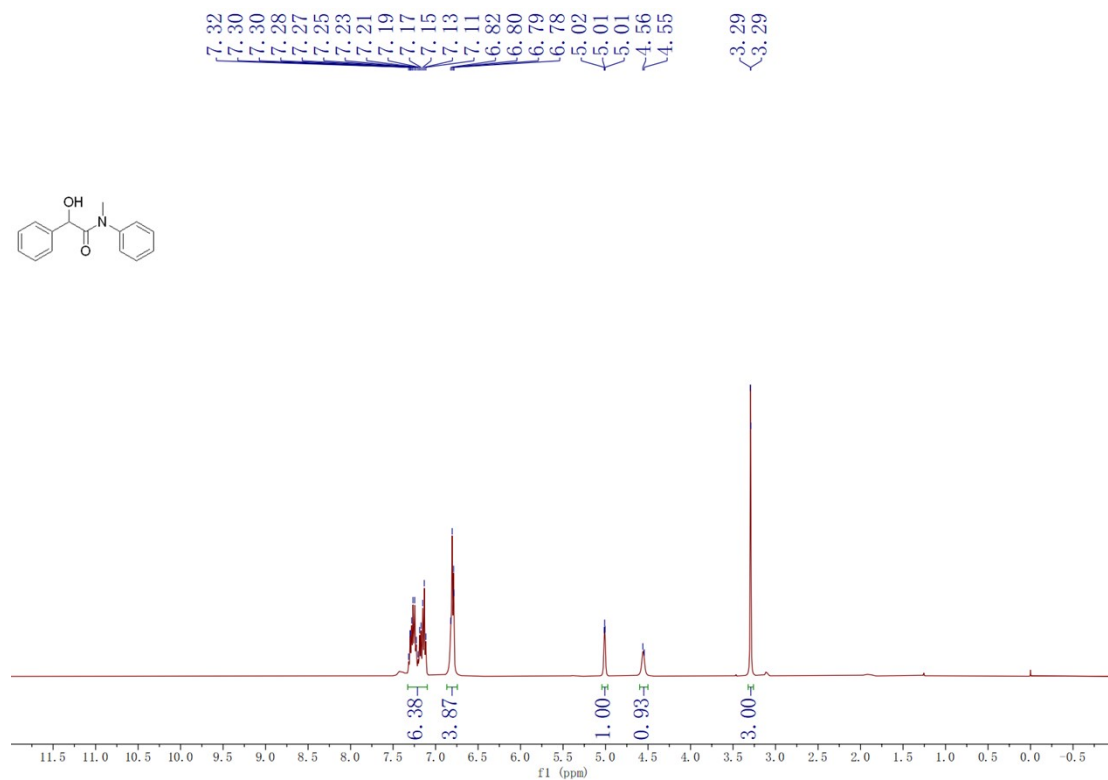
(20) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **20b**



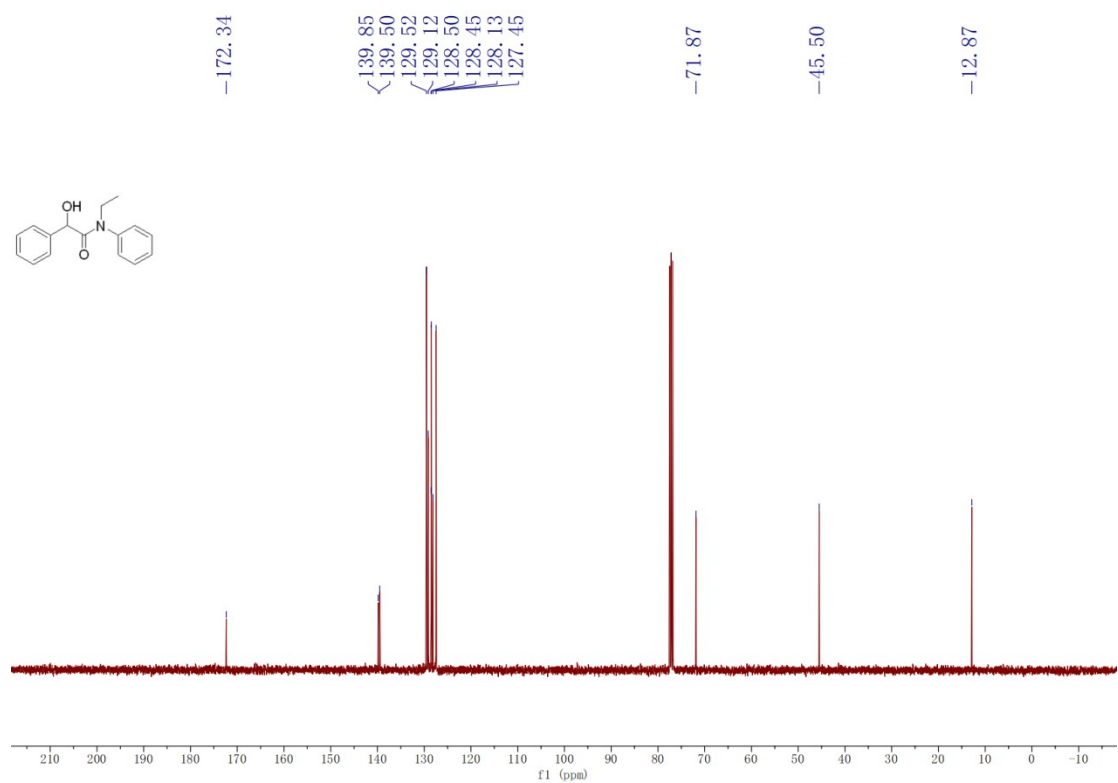
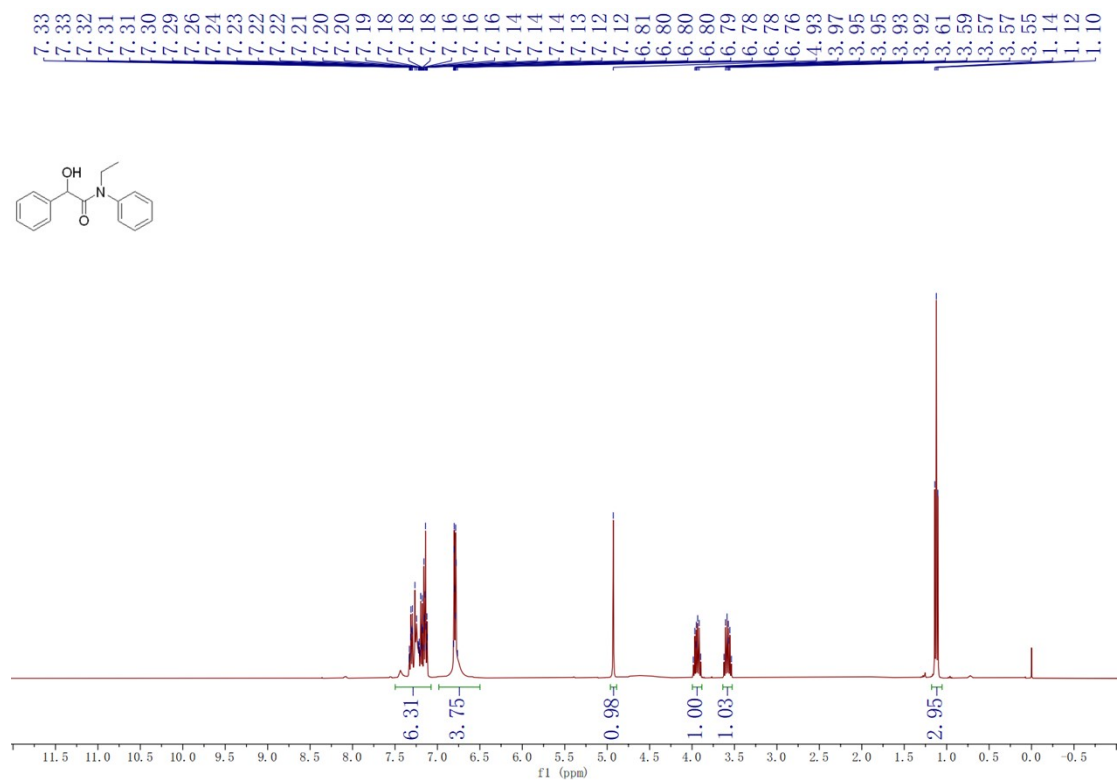
(21) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **21b**



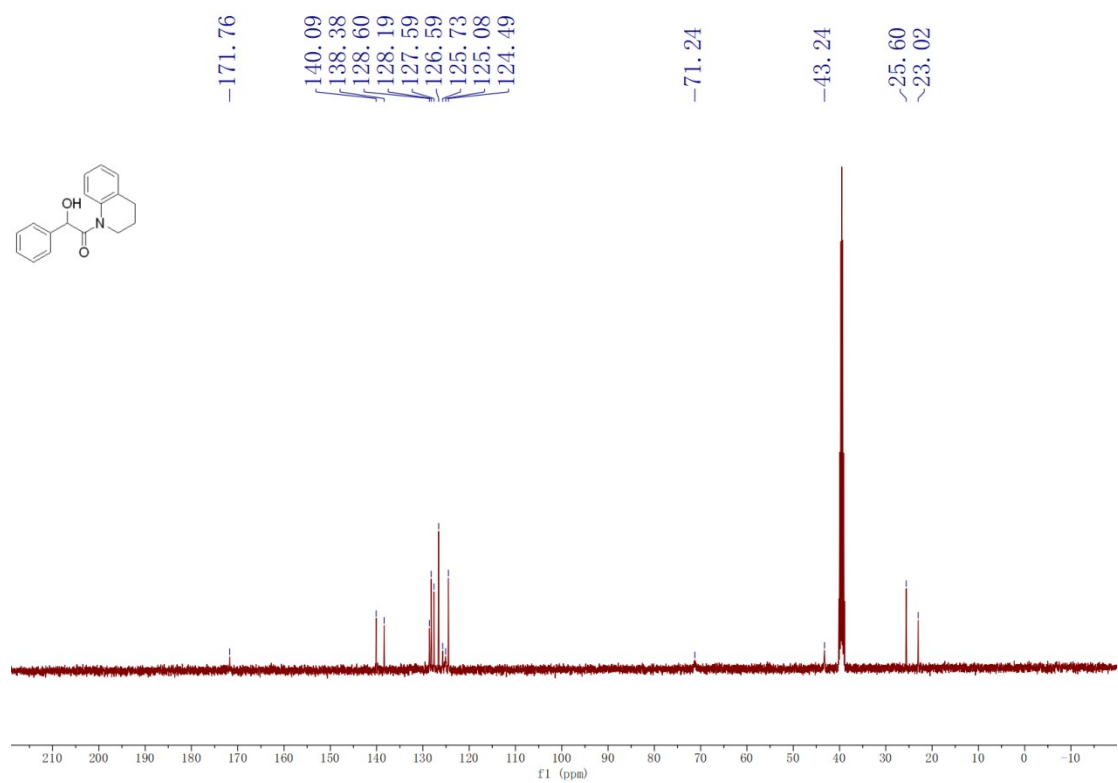
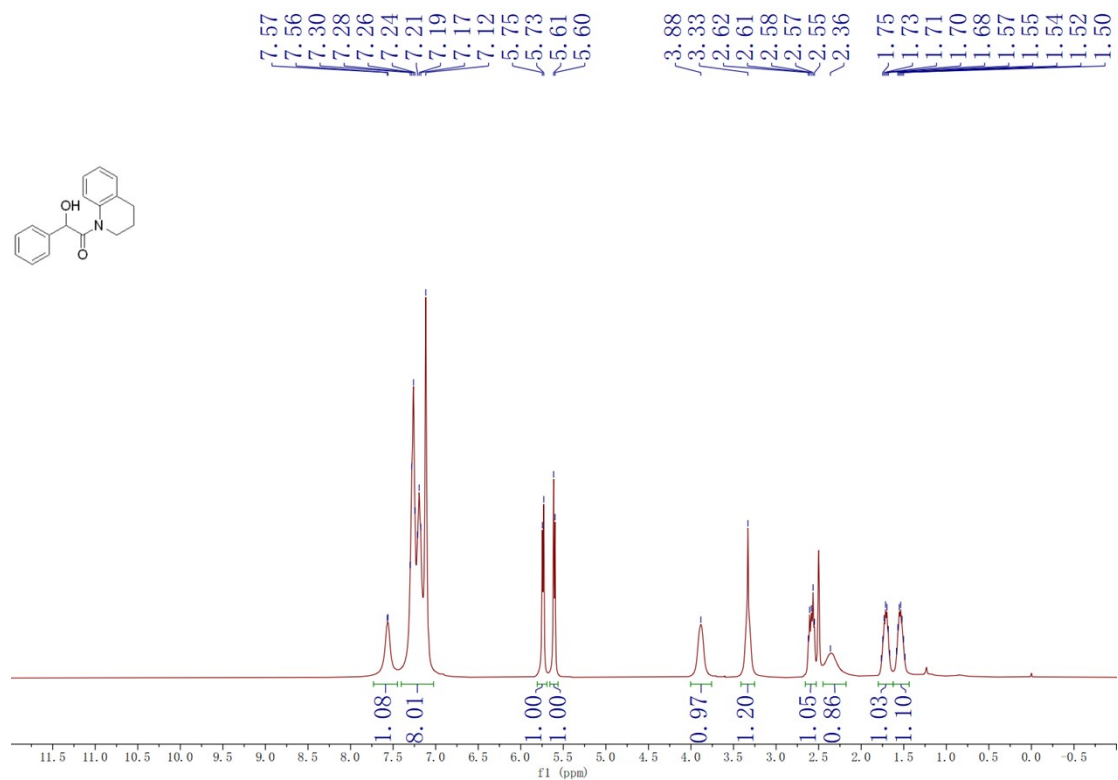
(22) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **22b**



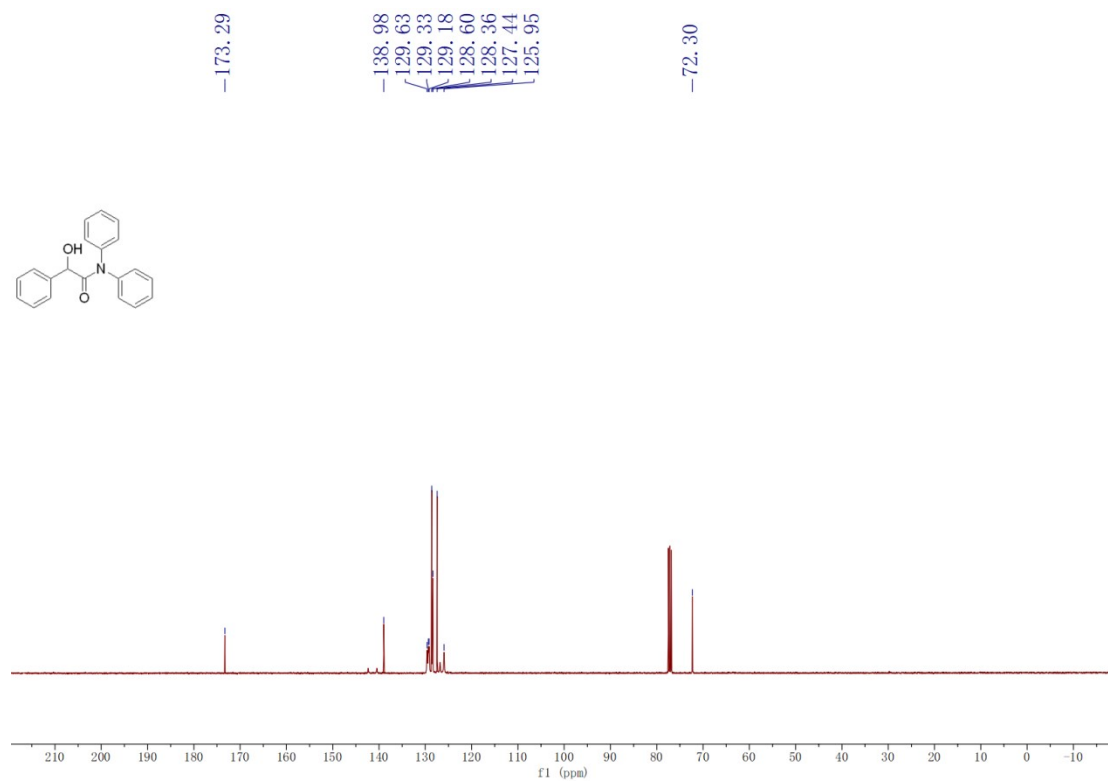
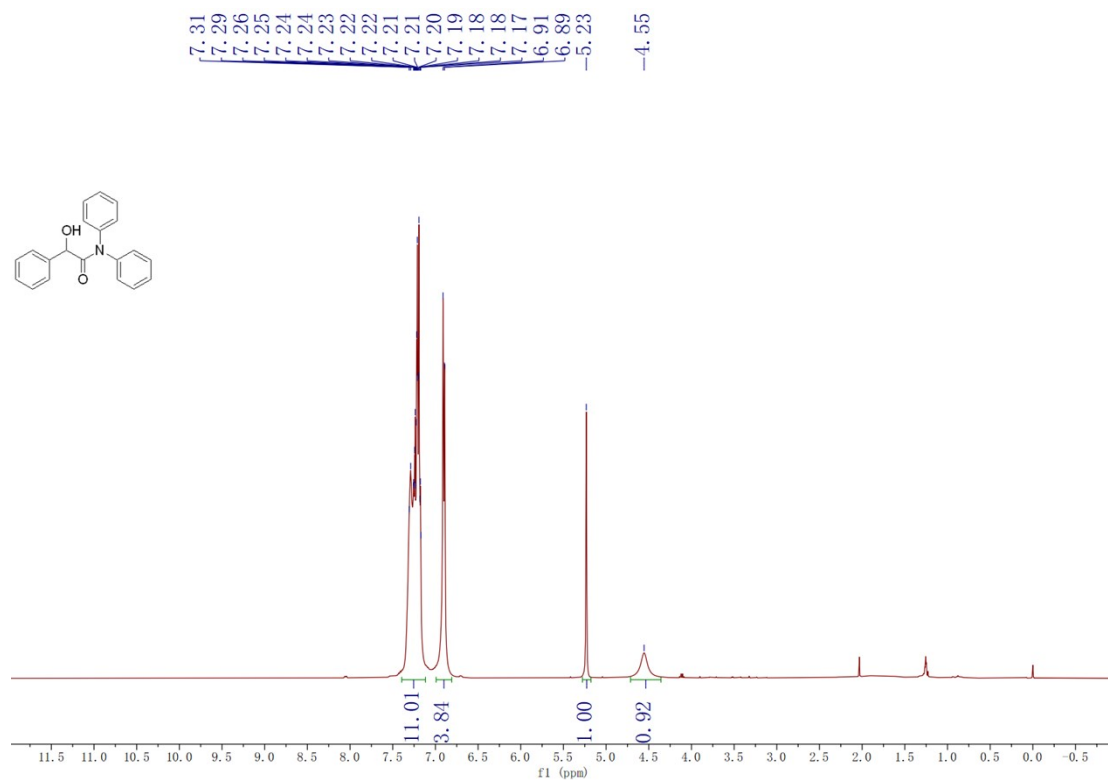
(23)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **23b**



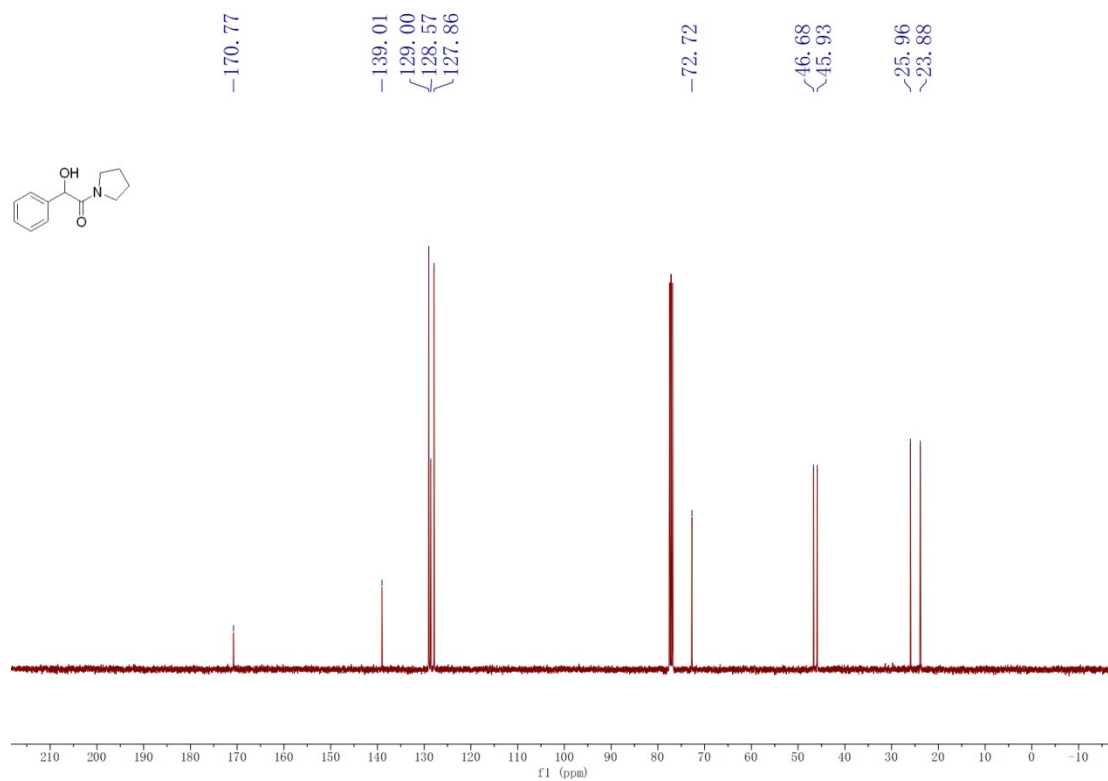
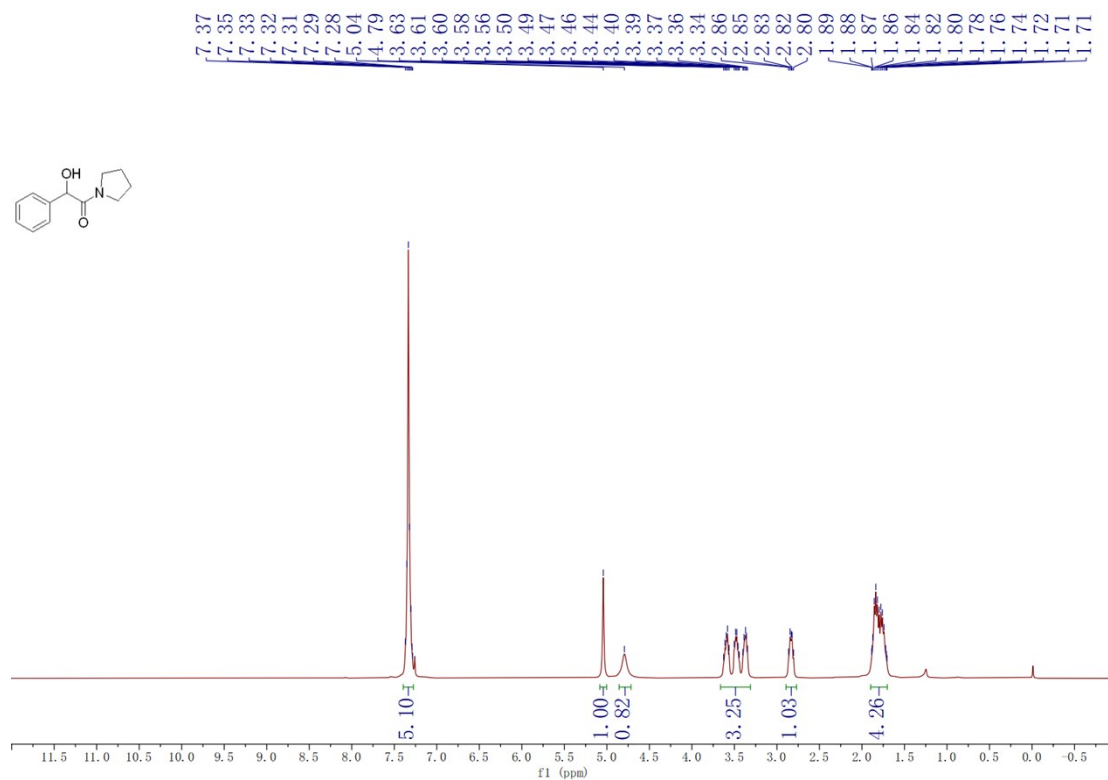
(24)¹H NMR (DMSO-*d*₆) and ¹³C NMR (DMSO-*d*₆) spectrum of **24b**



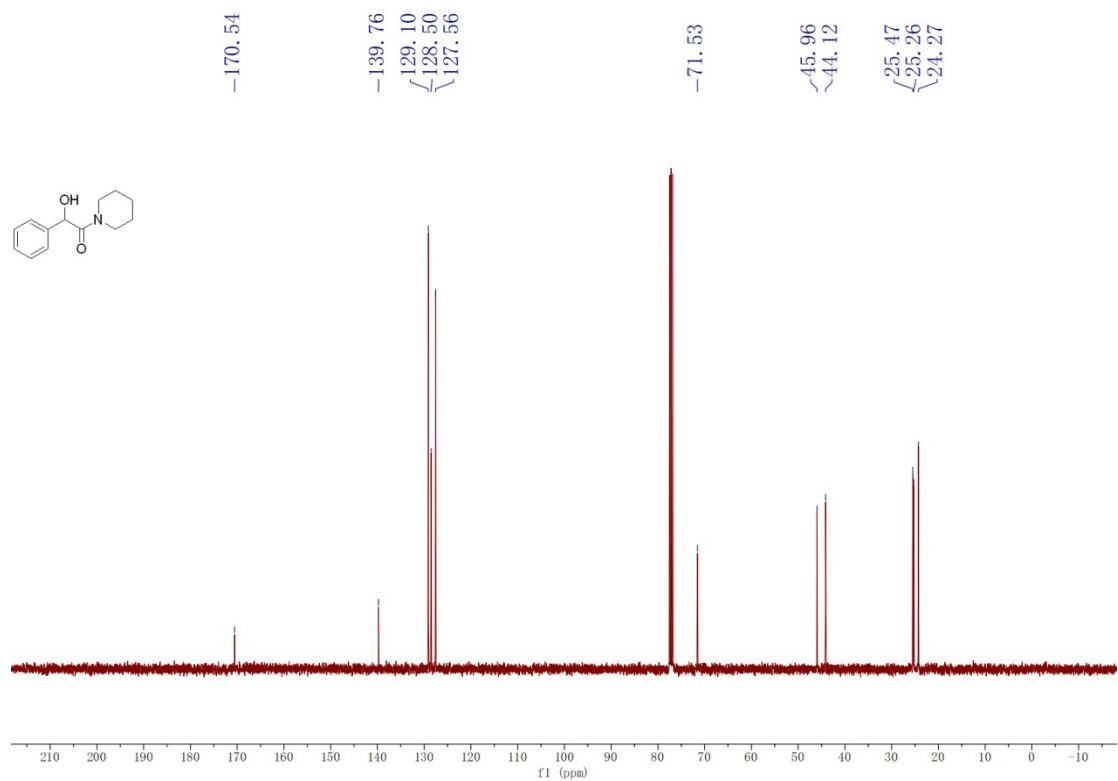
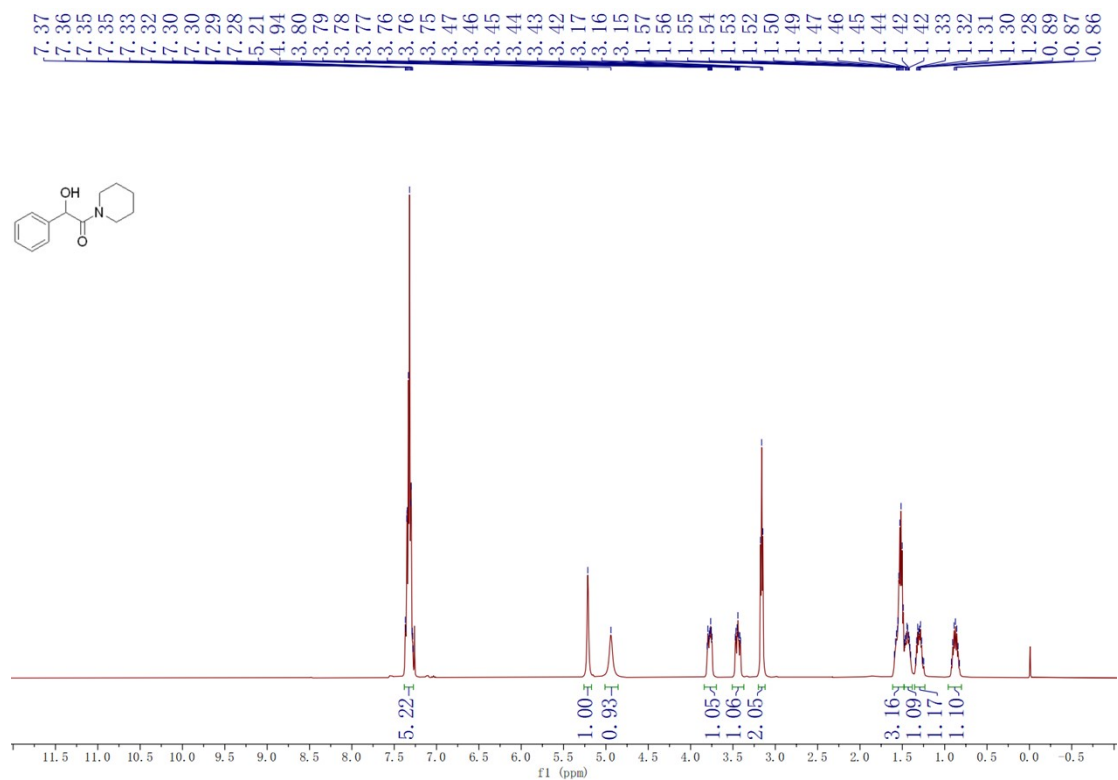
(25) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **25b**



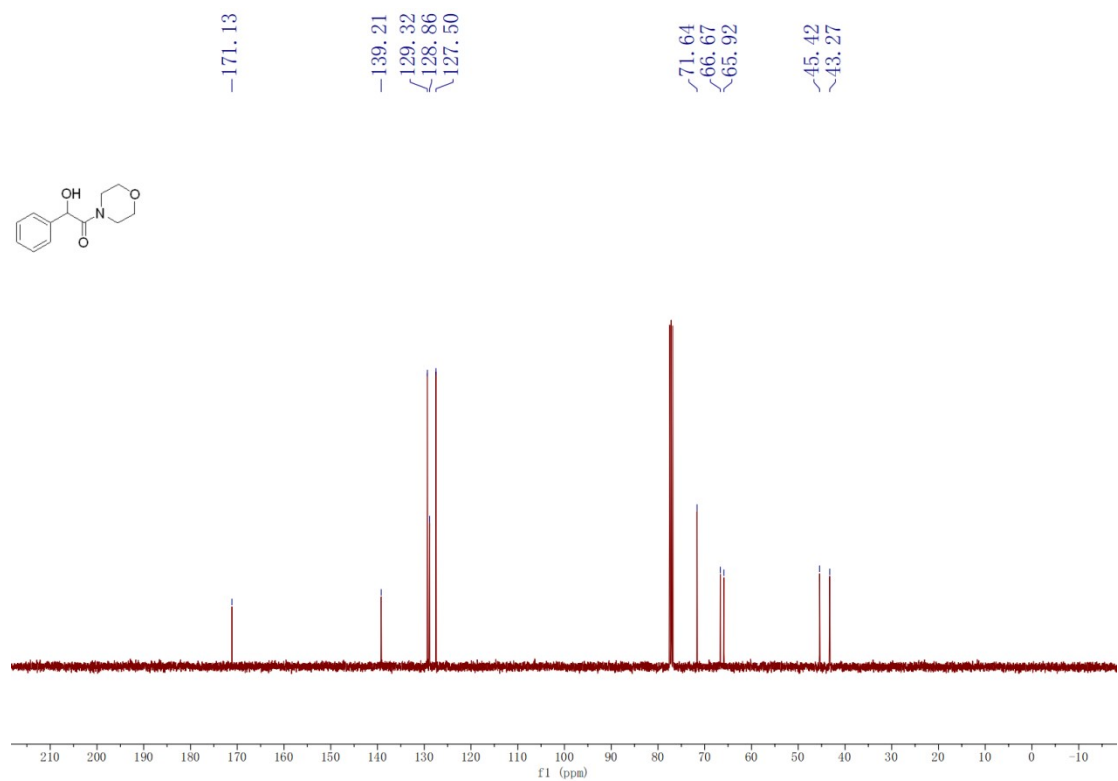
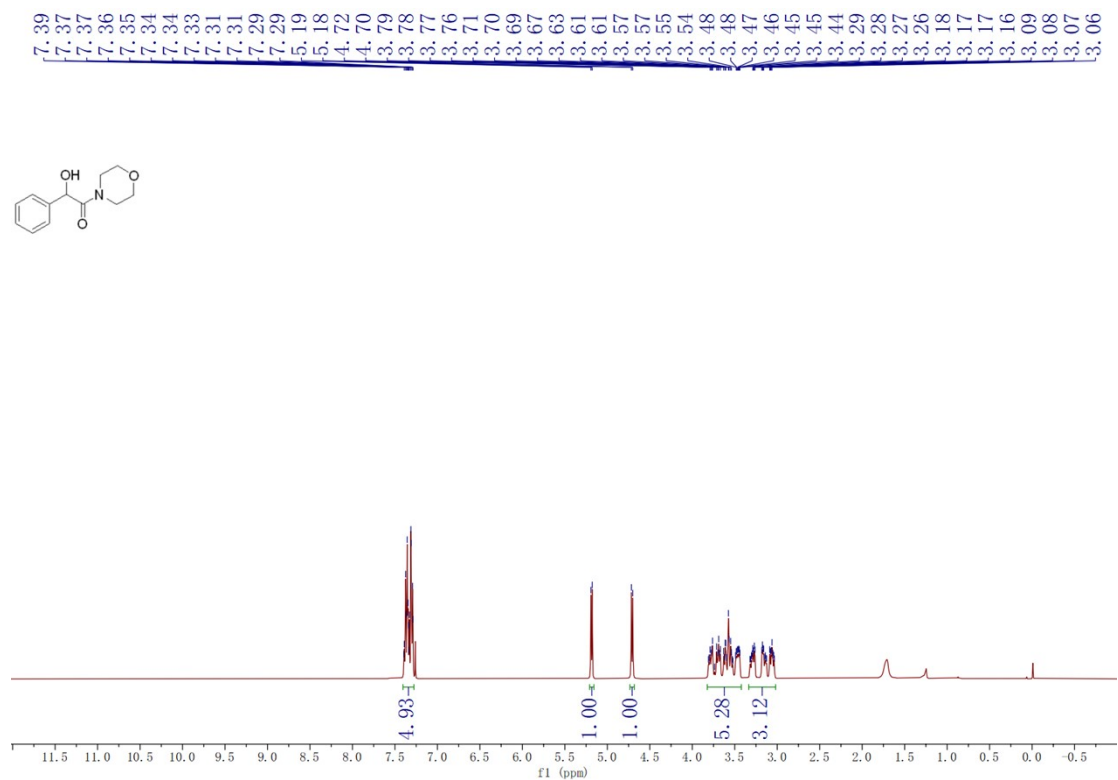
(26)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **26b**



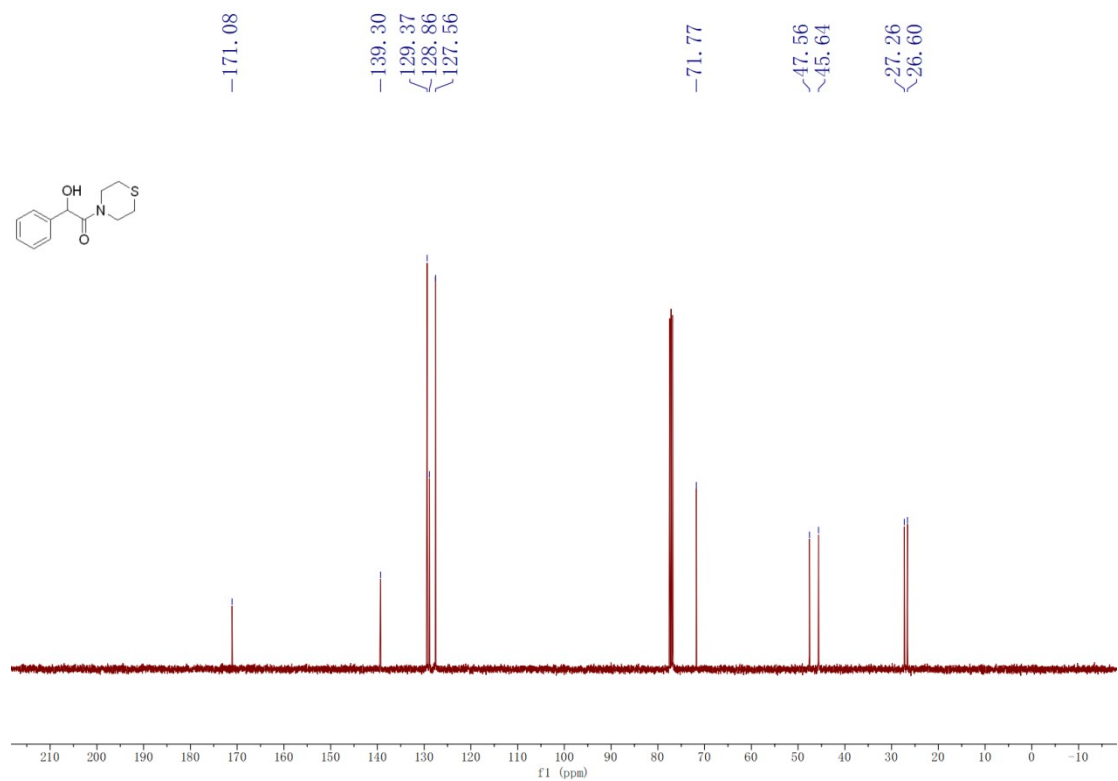
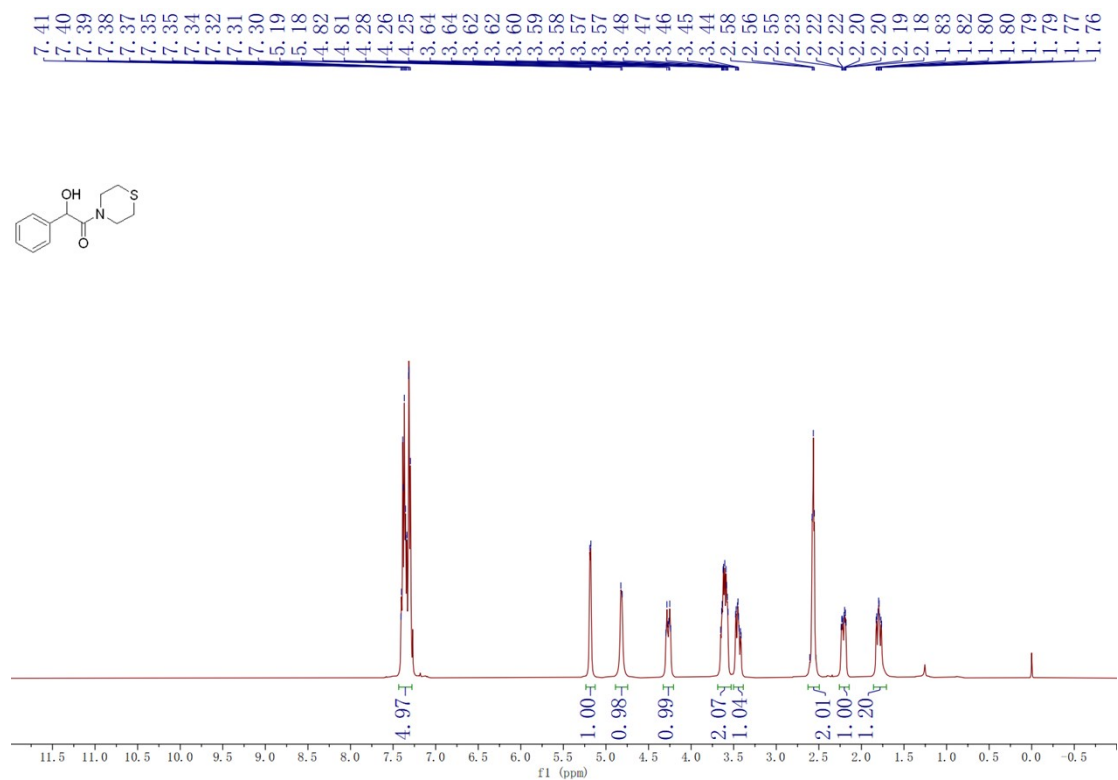
(27)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **27b**



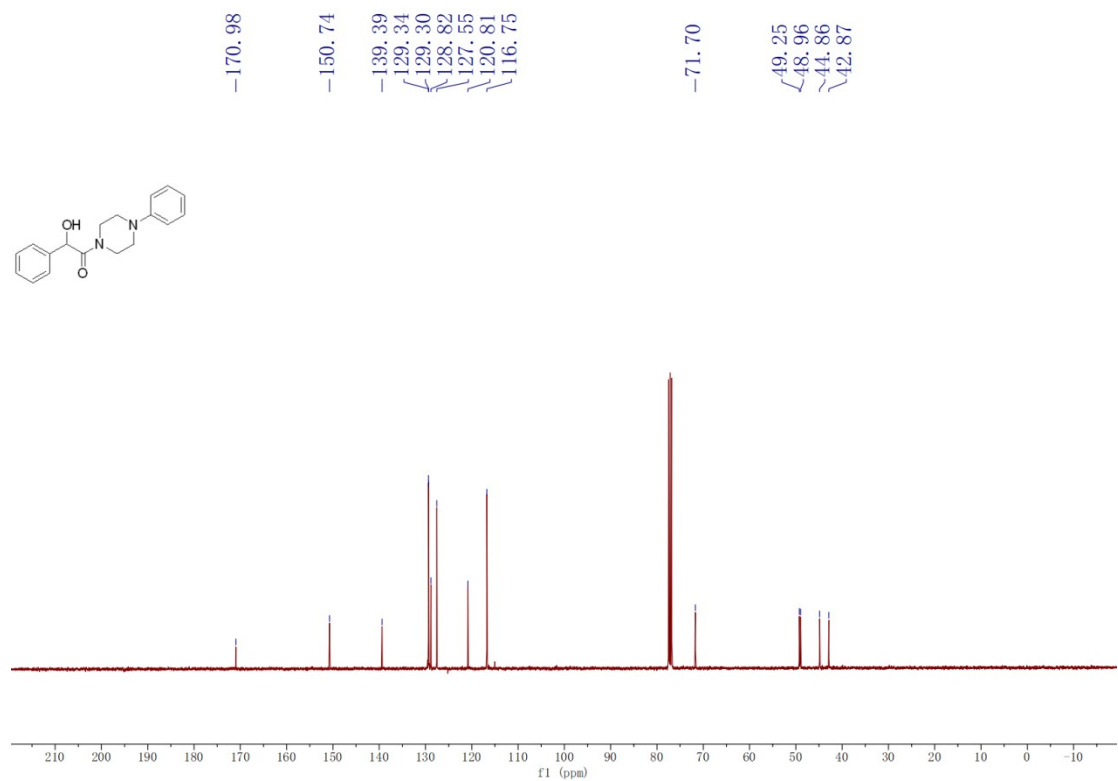
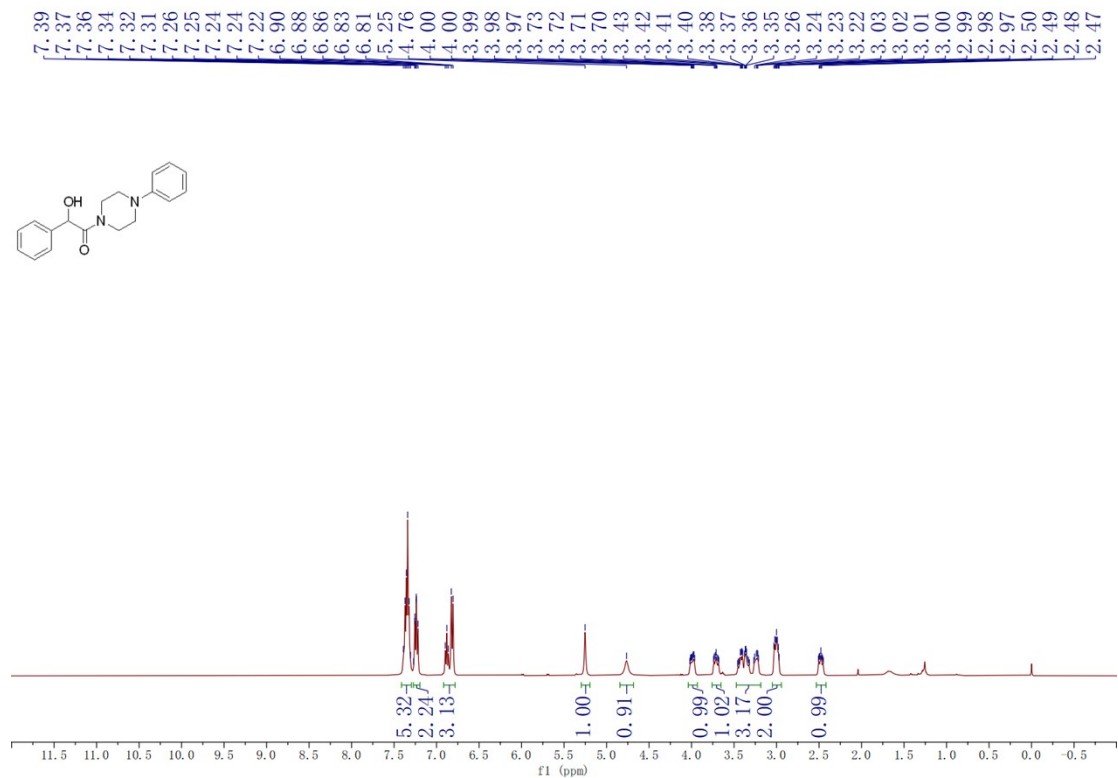
(28)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **28b**



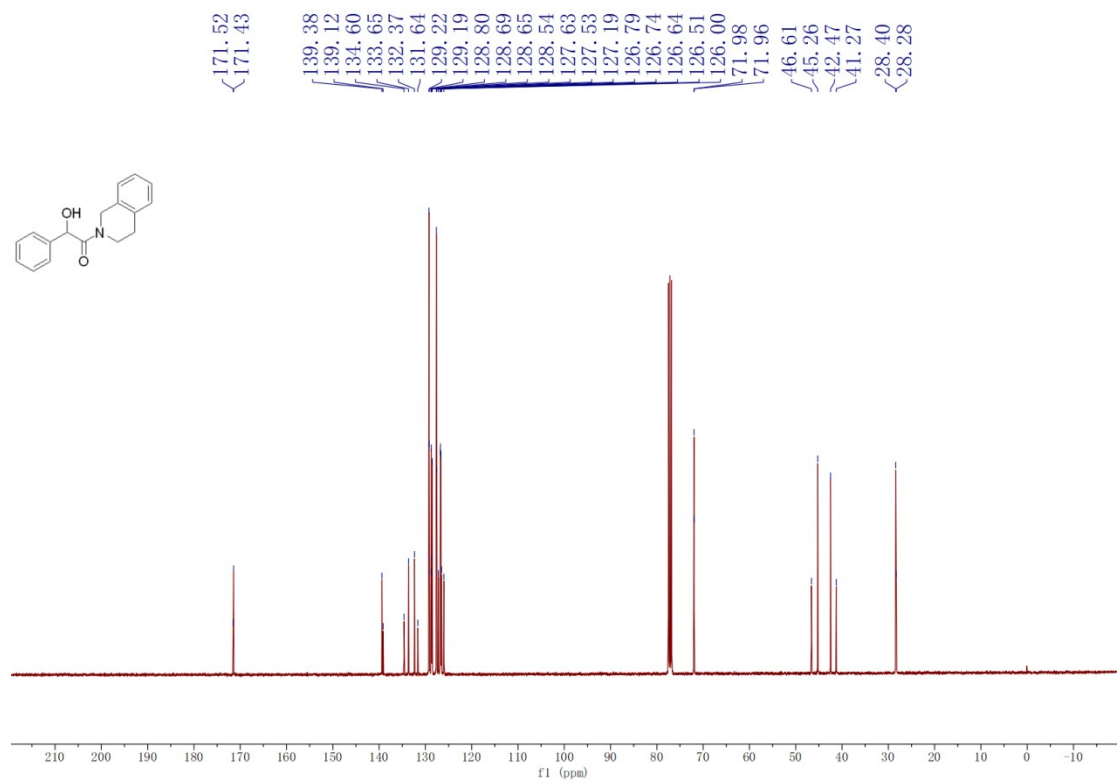
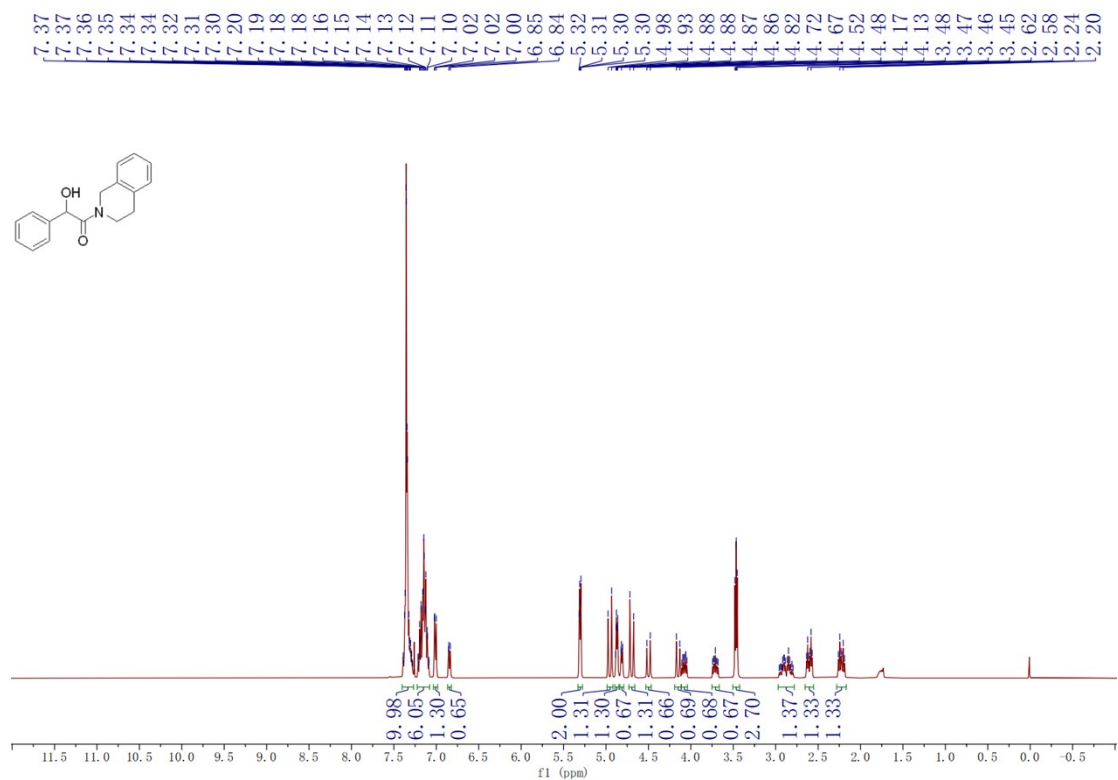
(29)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **29b**



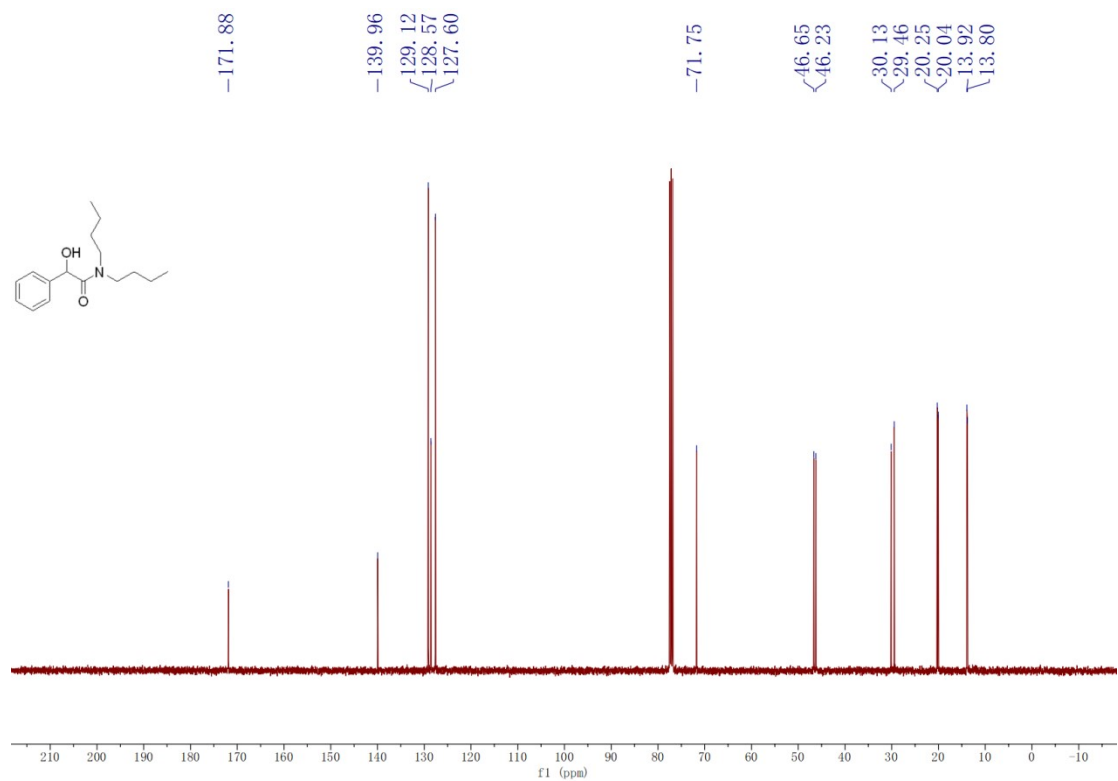
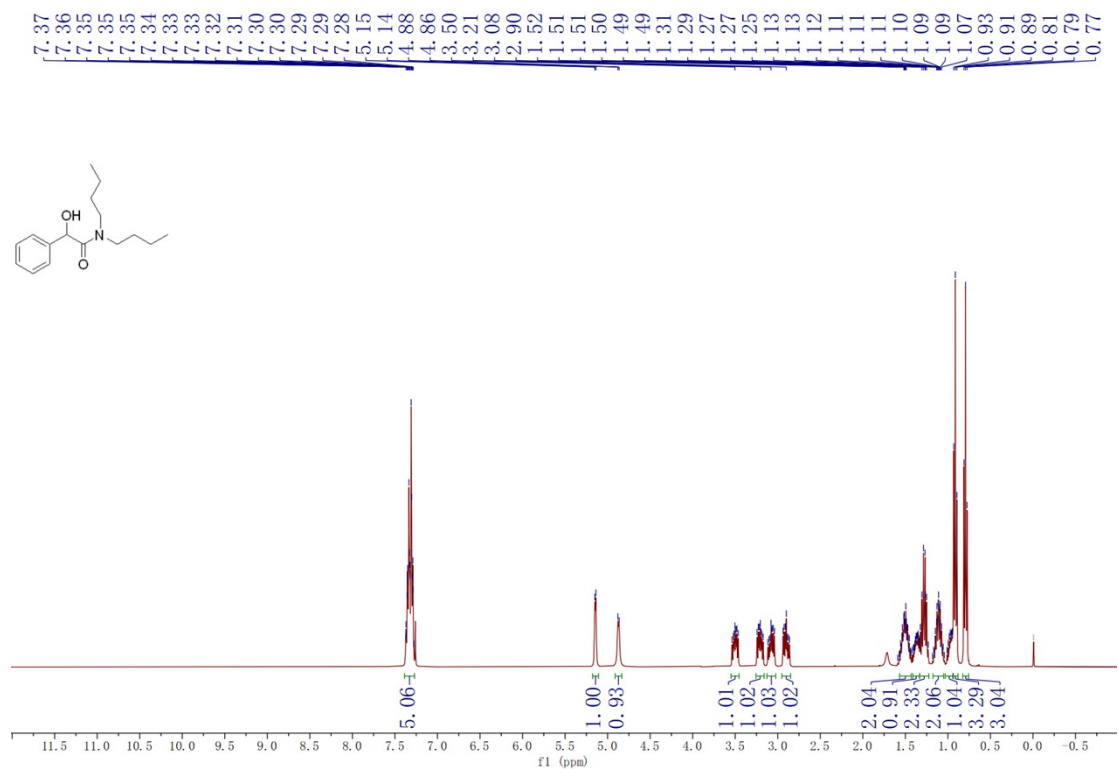
(³⁰)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **30b**



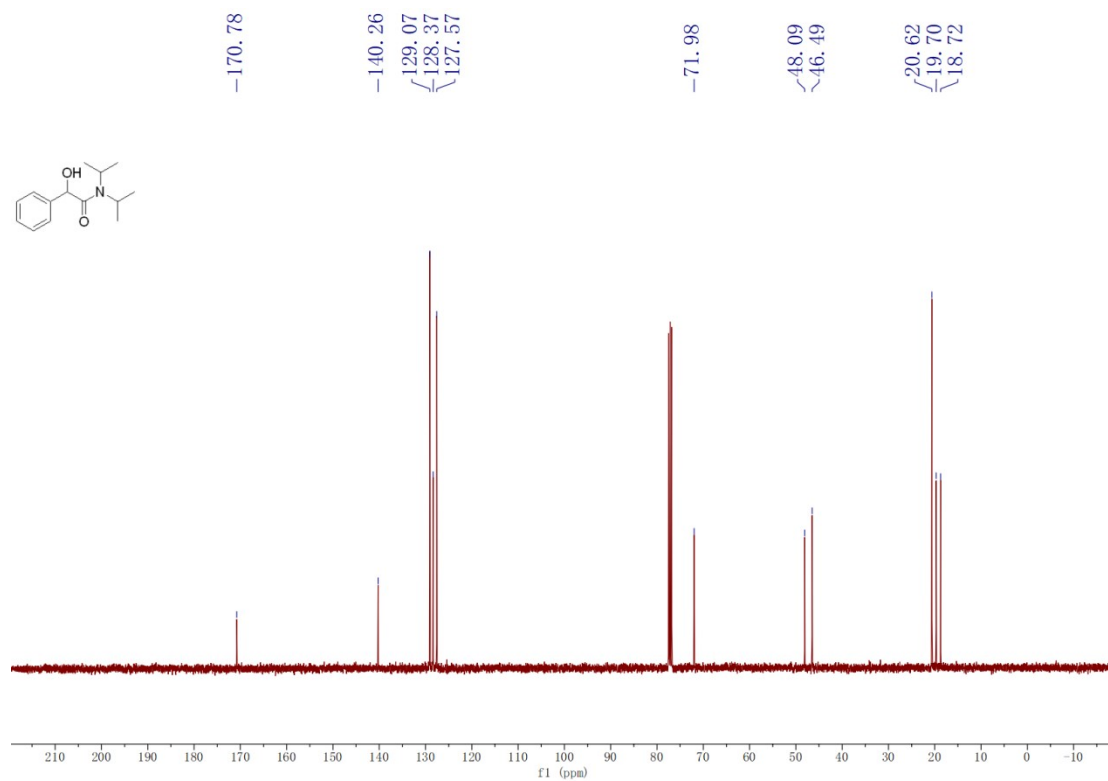
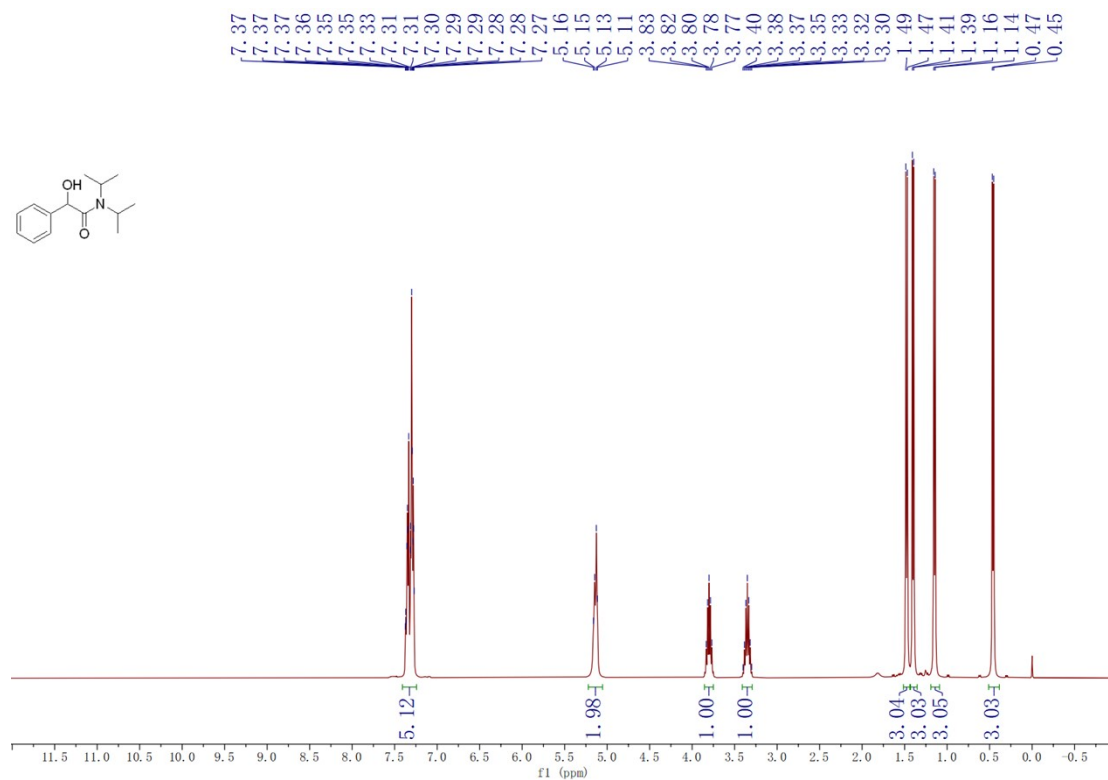
(31)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **31b**



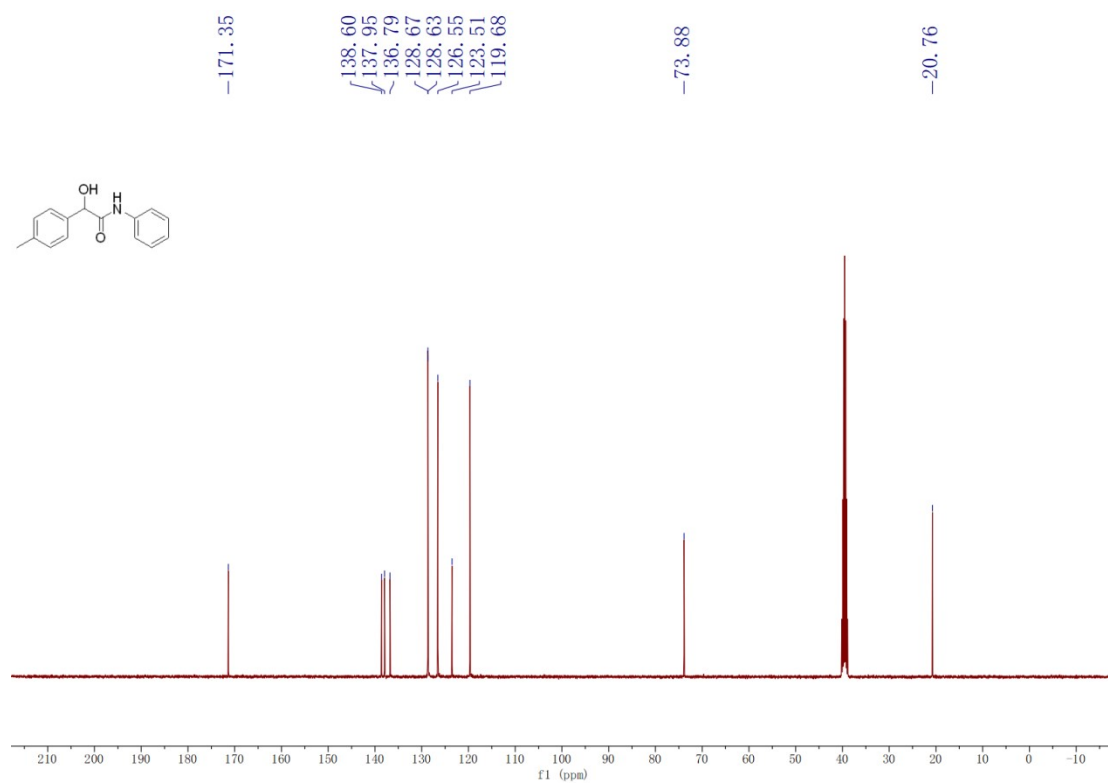
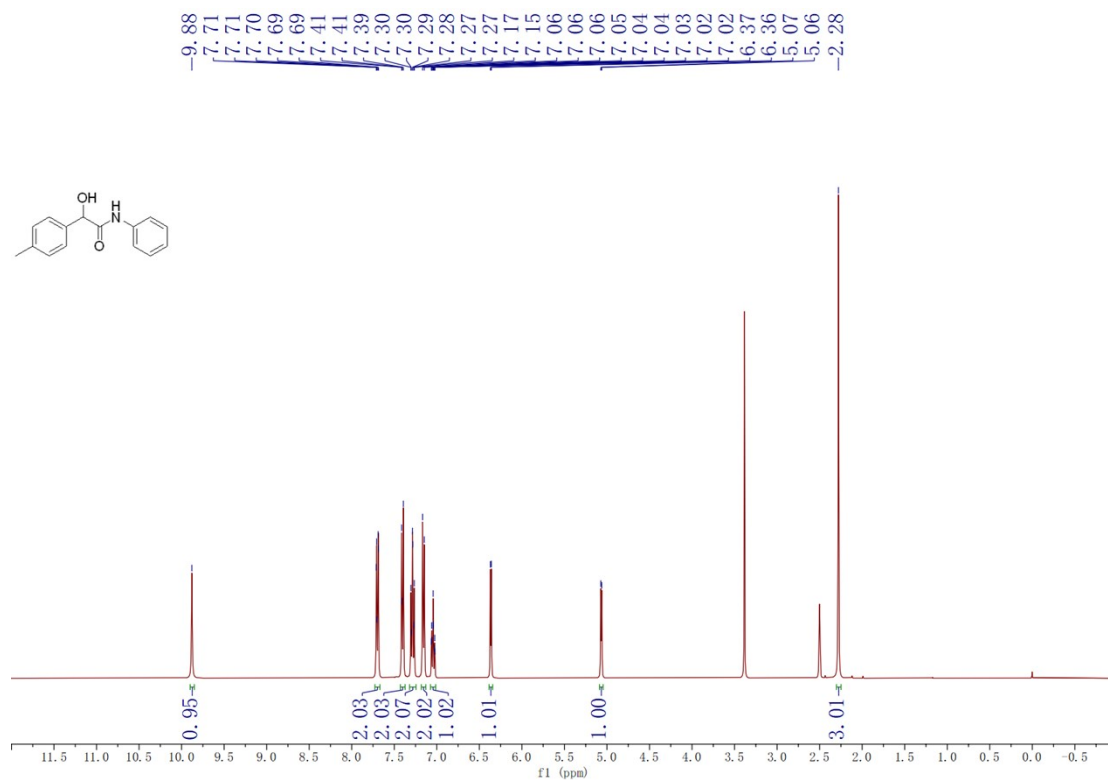
(32)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **32b**



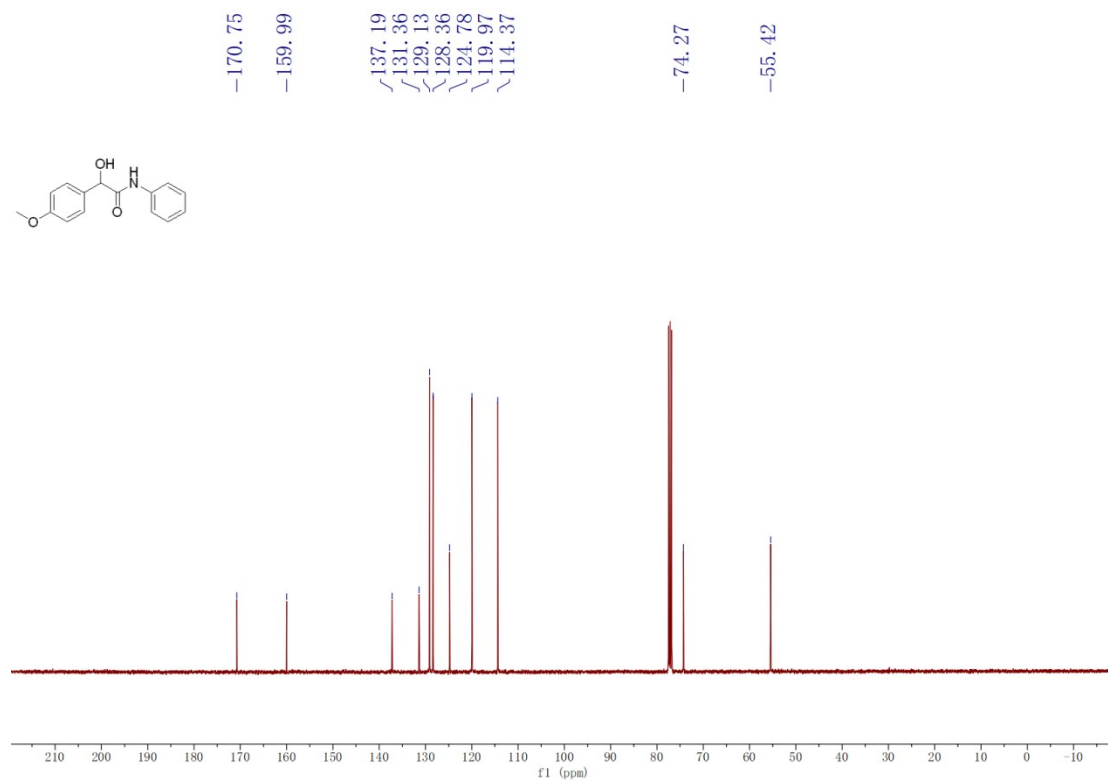
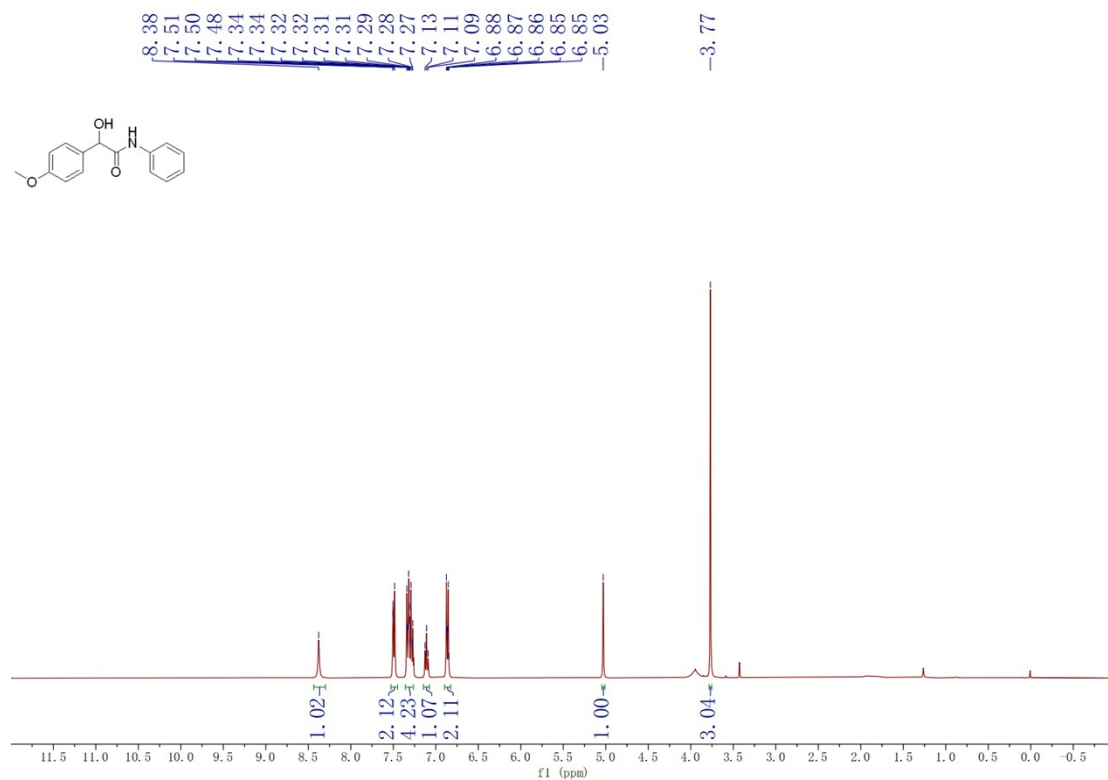
(³³)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **33b**



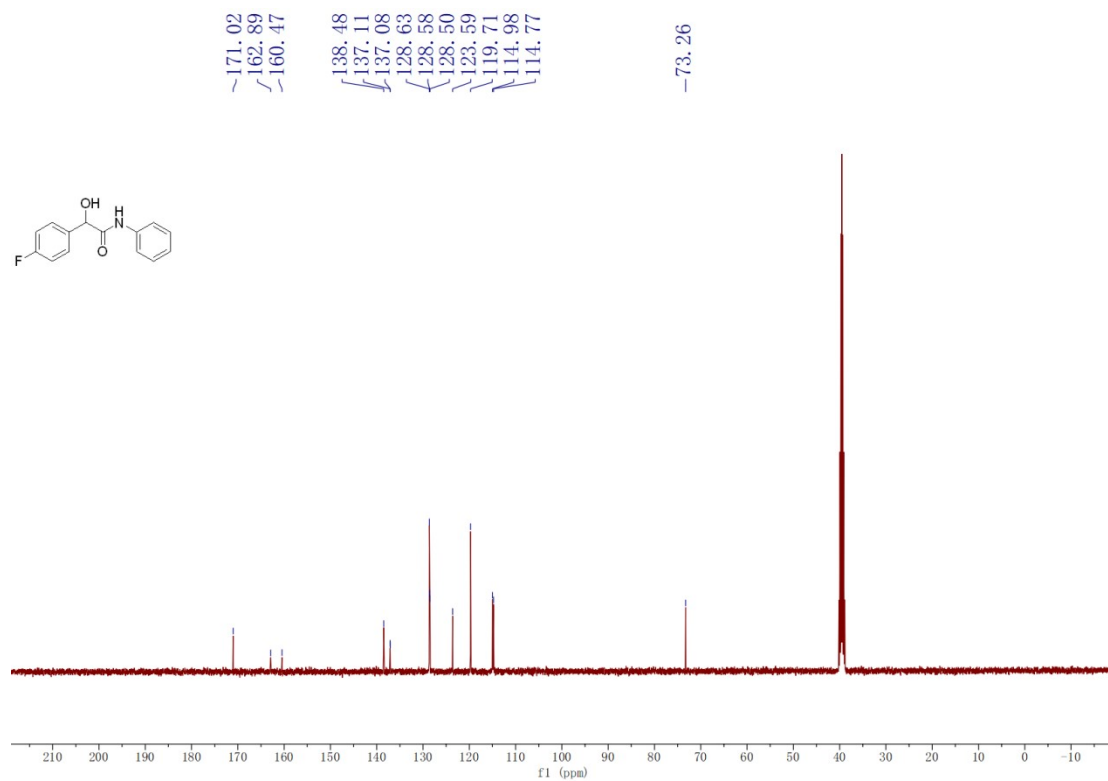
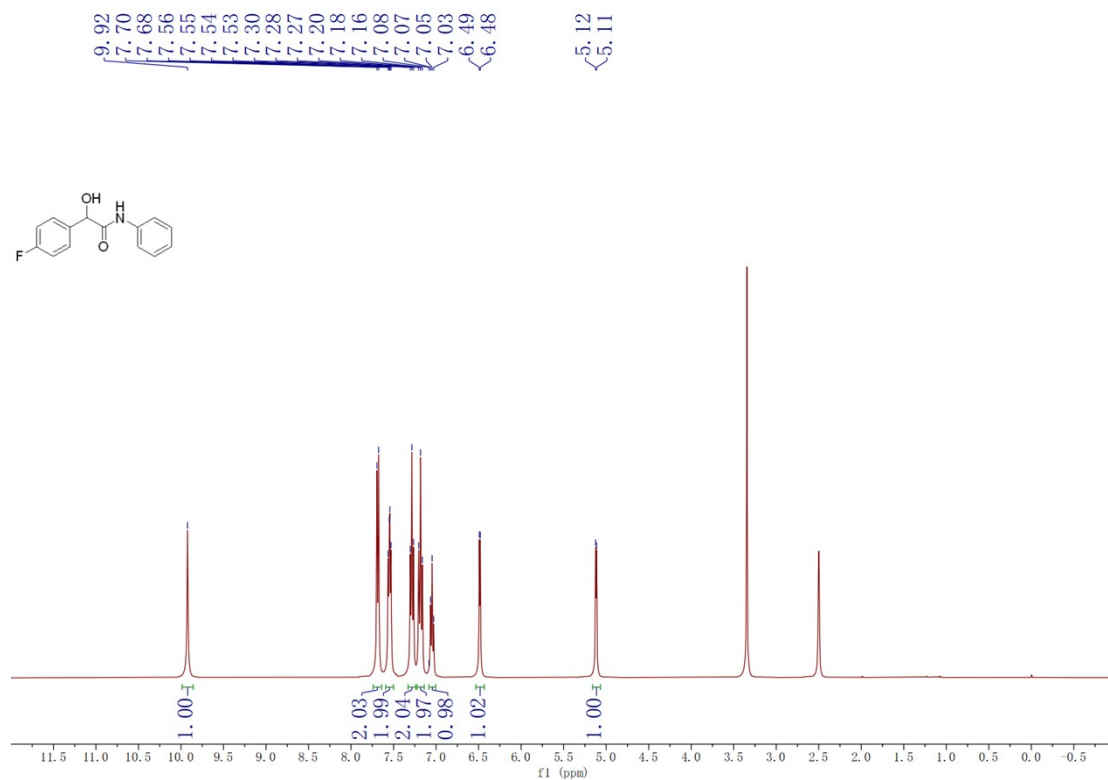
(34) ^1H NMR ($\text{DMSO-}d_6$) and ^{13}C NMR ($\text{DMSO-}d_6$) spectrum of **34b**



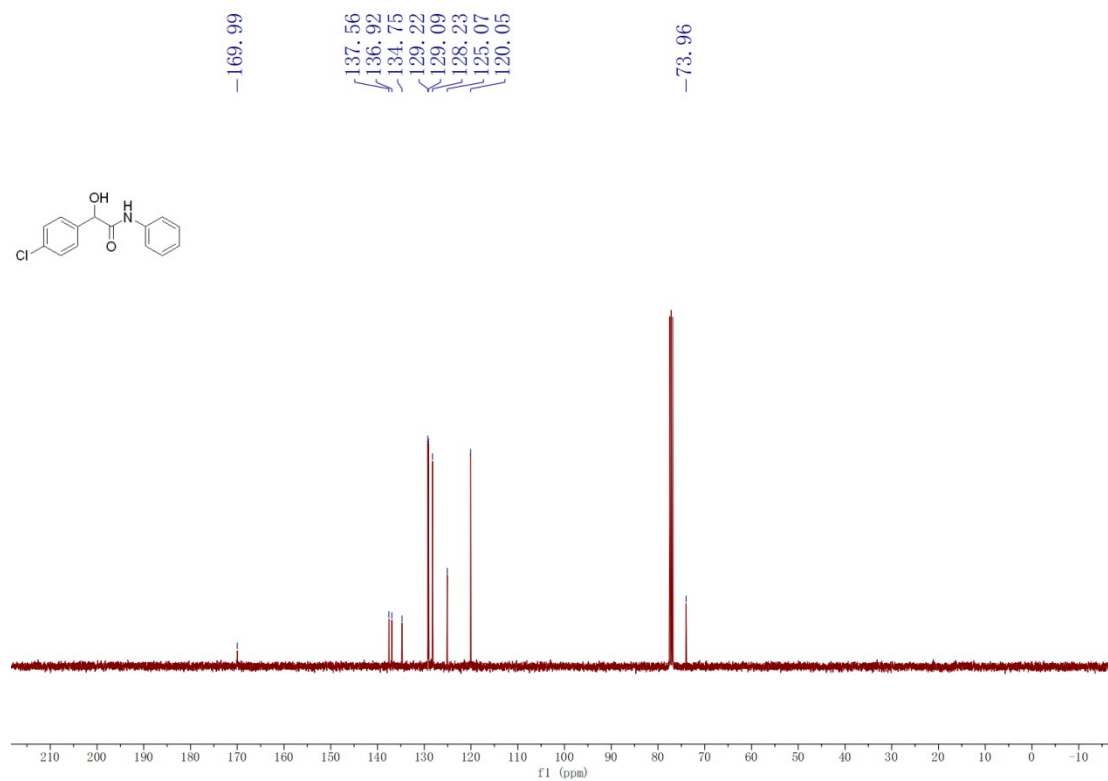
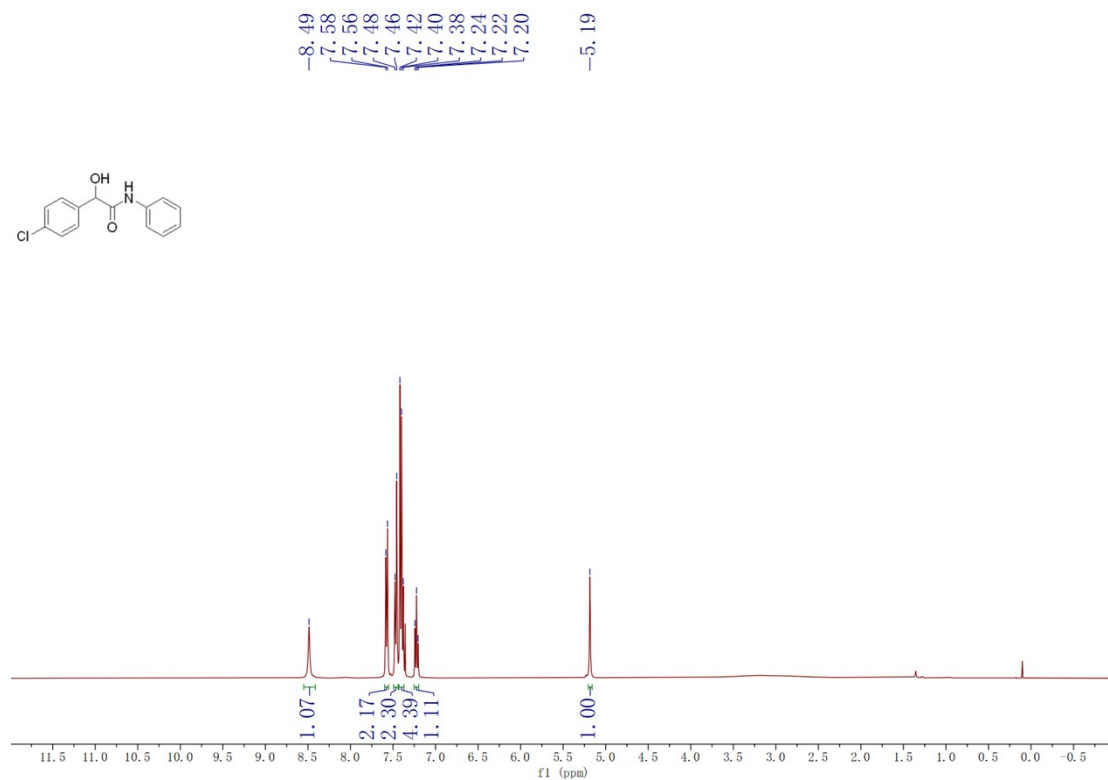
(35) ^1H NMR (CDCl_3) and ^{13}C NMR (CDCl_3) spectrum of **35b**



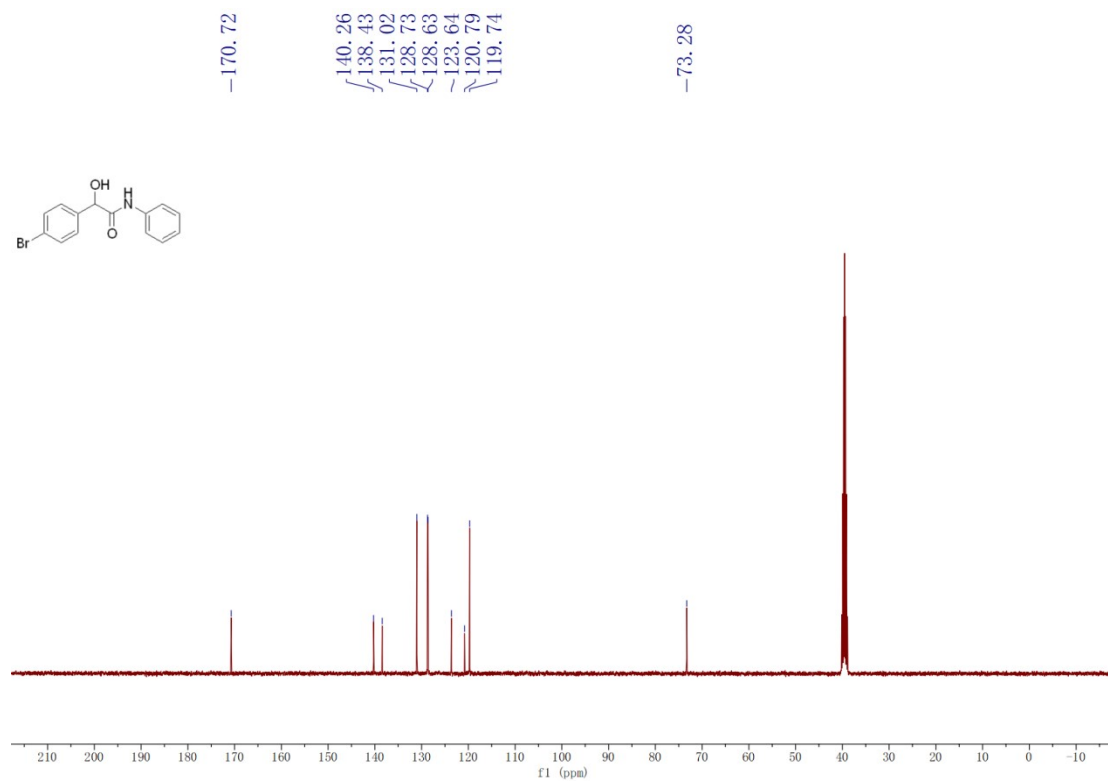
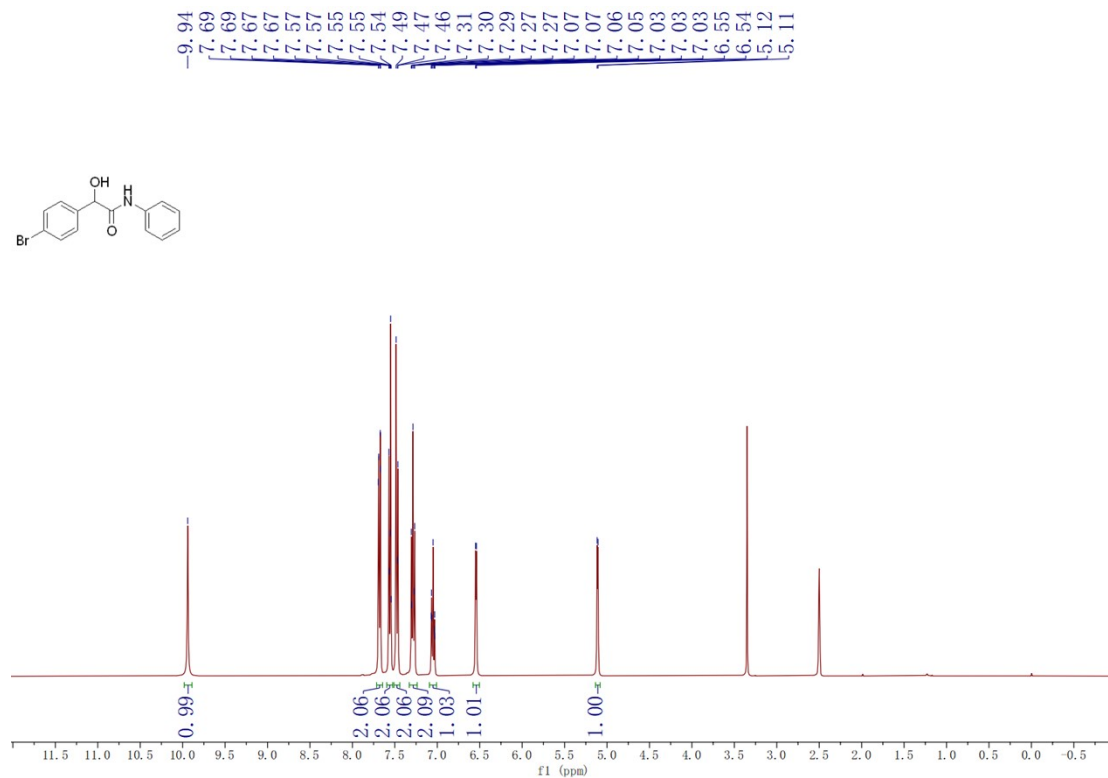
(36) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **36b**



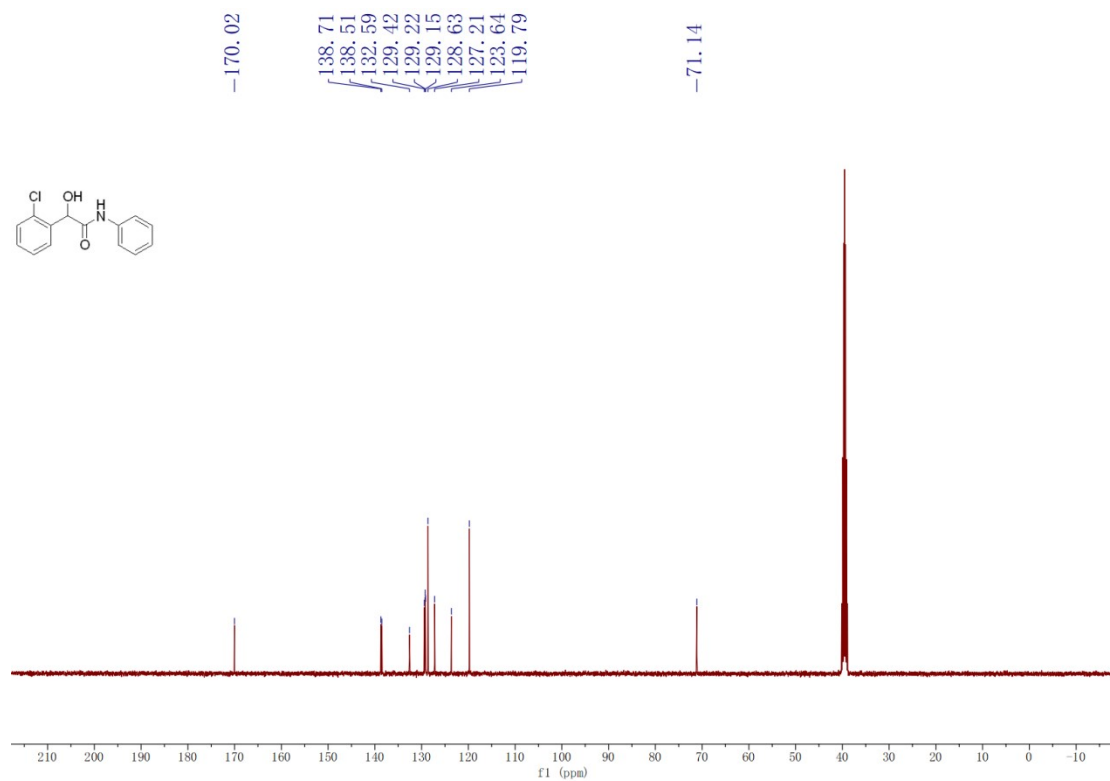
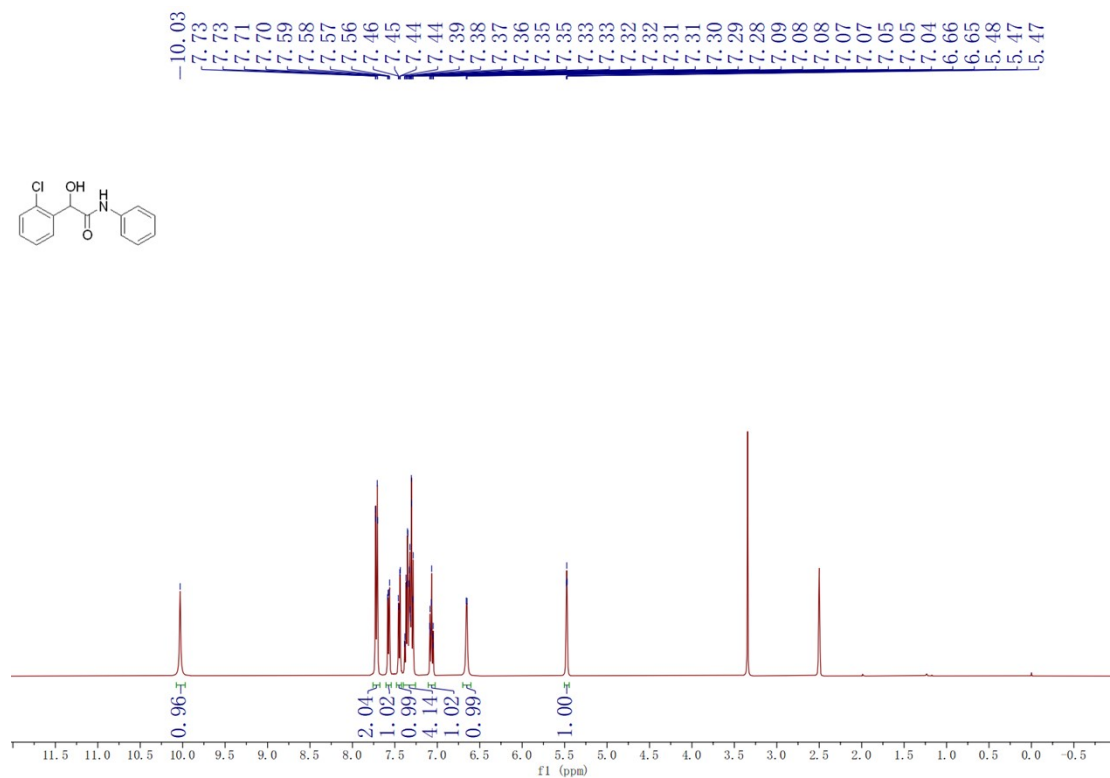
(37)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **37b**



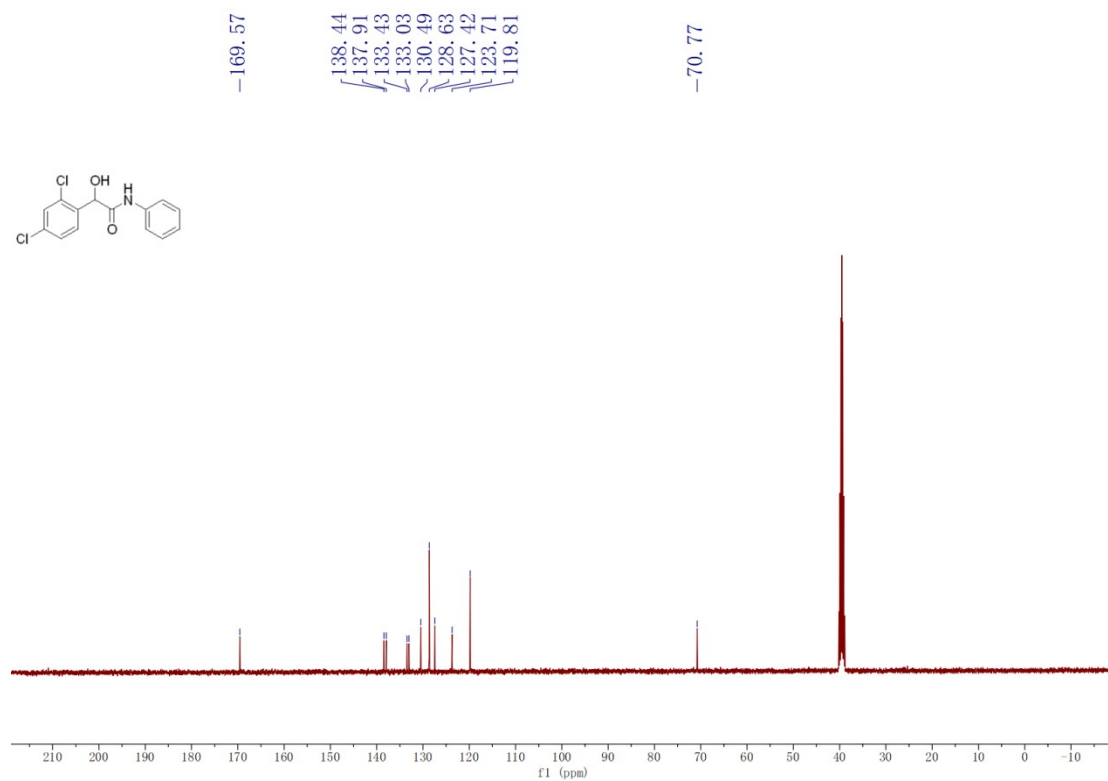
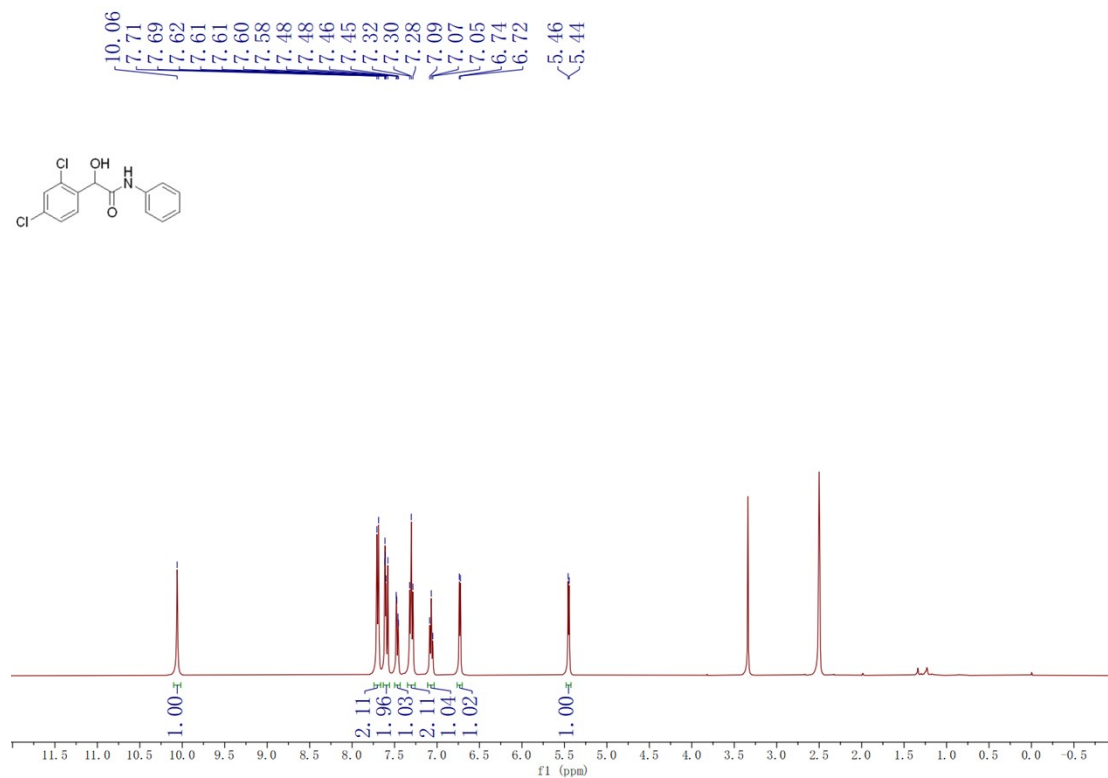
(38)¹H NMR (DMSO-*d*₆) and ¹³C NMR (DMSO-*d*₆) spectrum of **38b**



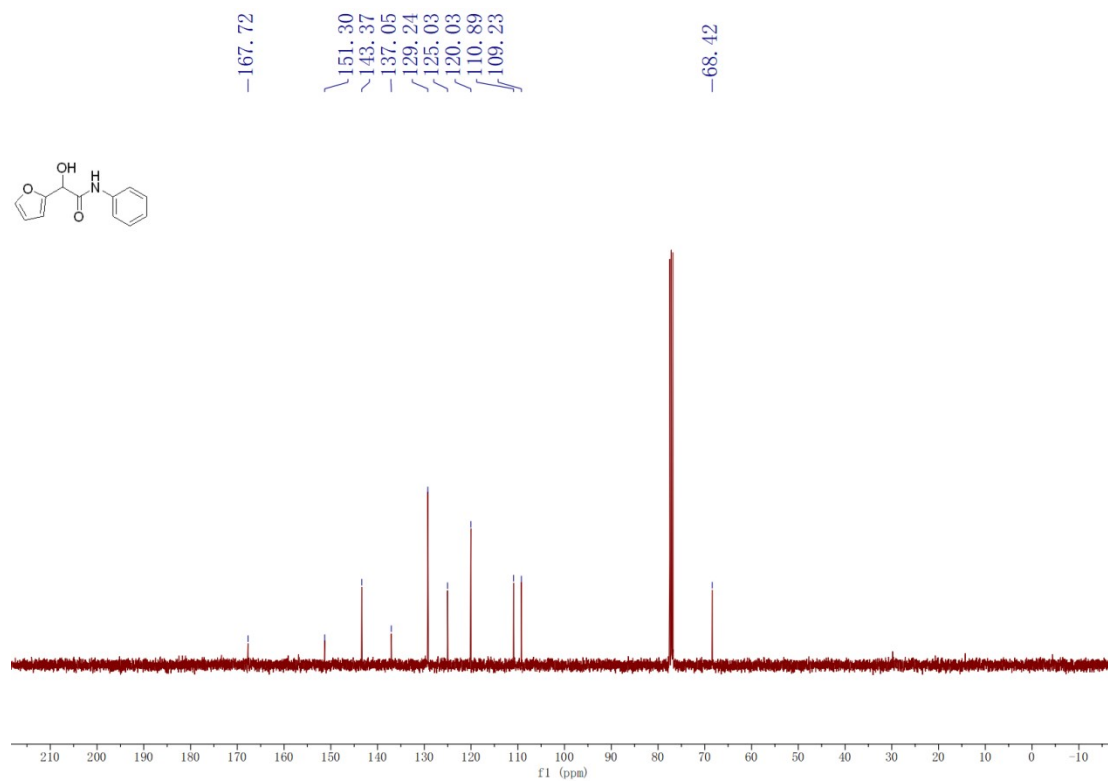
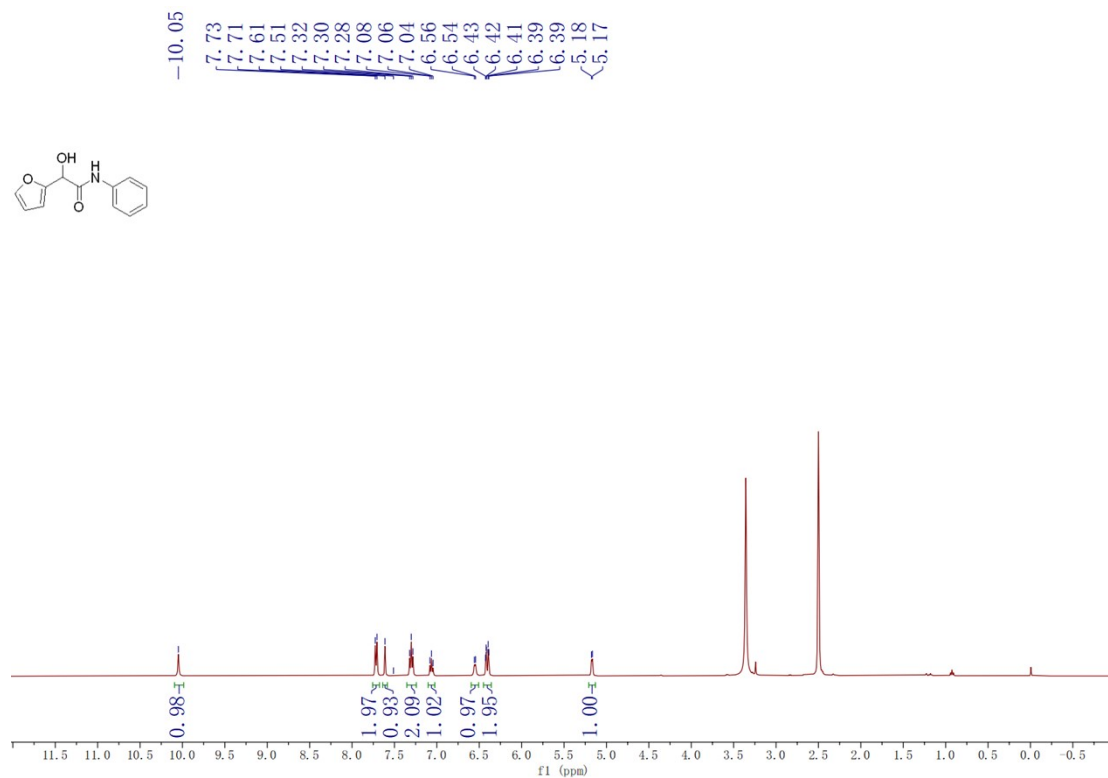
(39) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **39b**



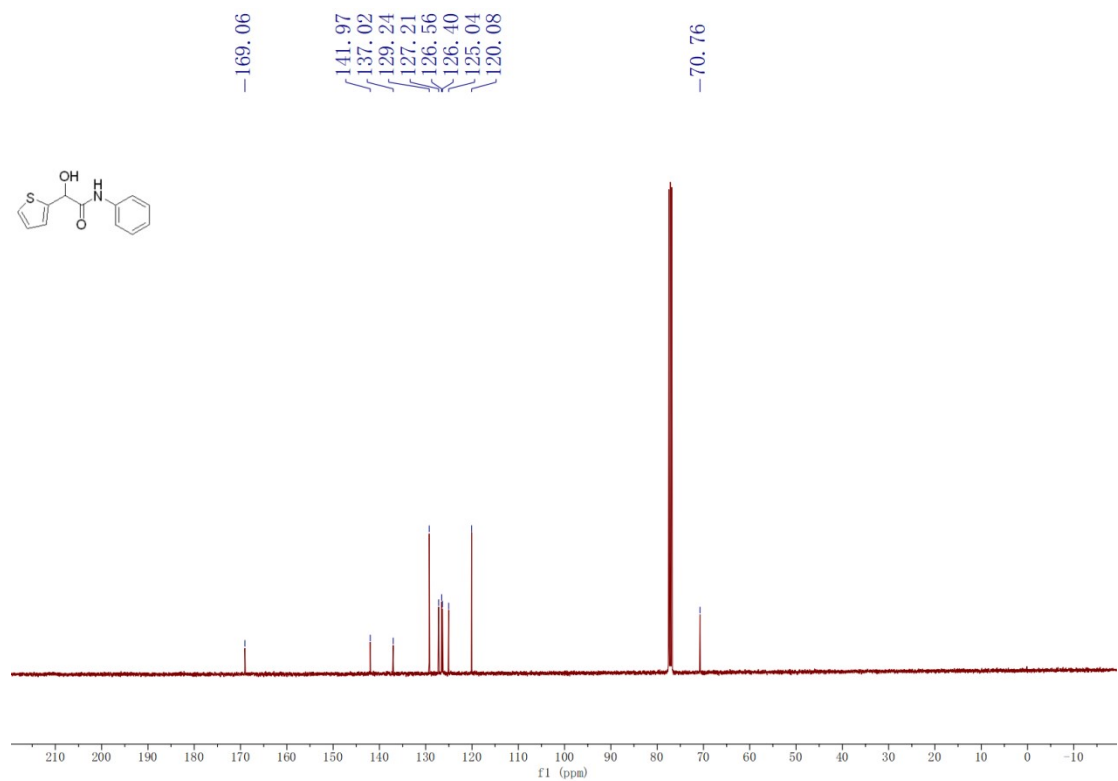
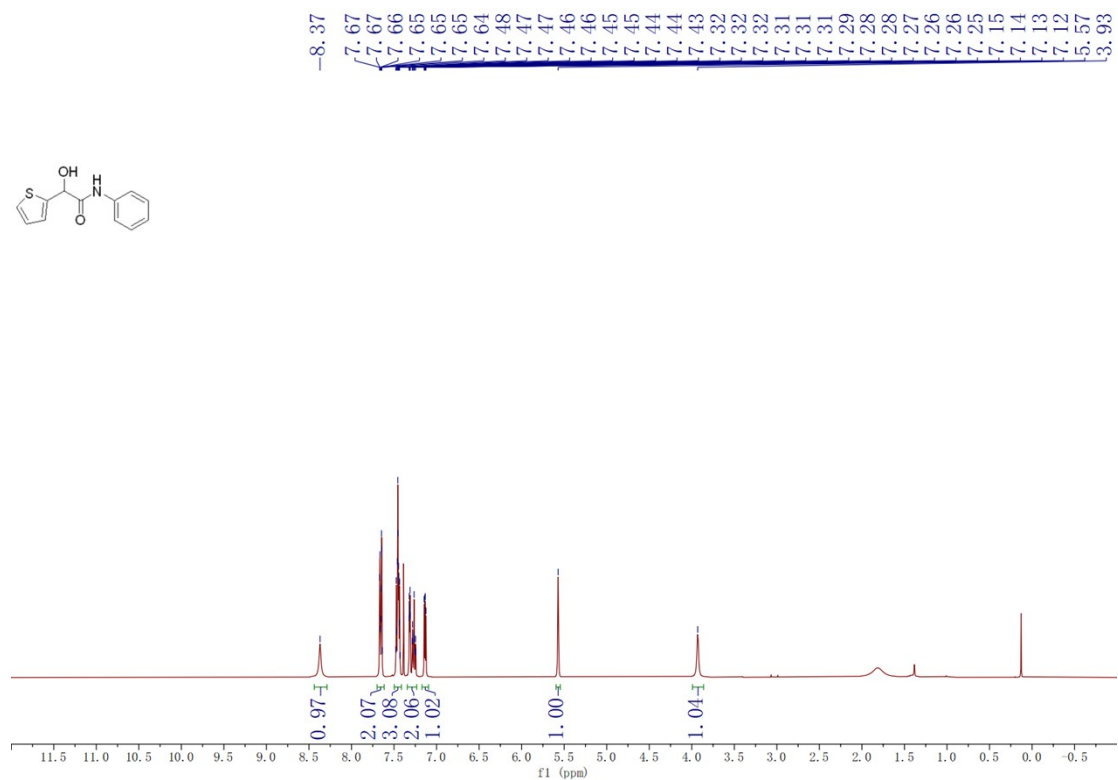
(40) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **40b**



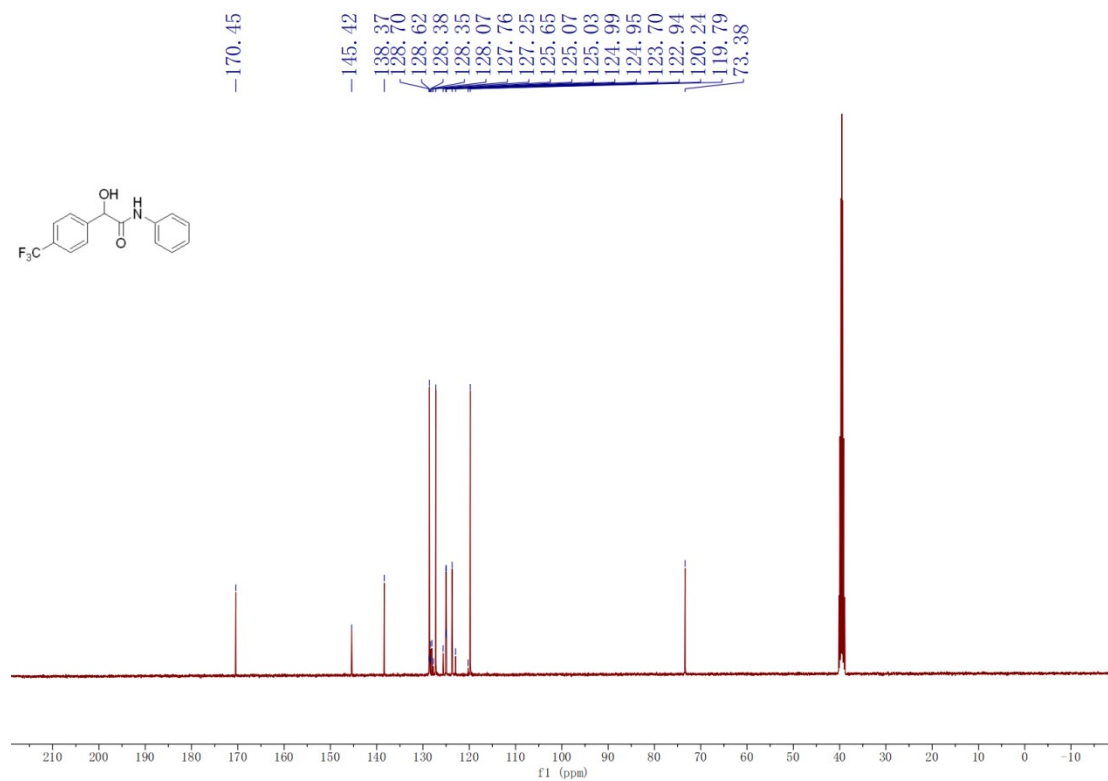
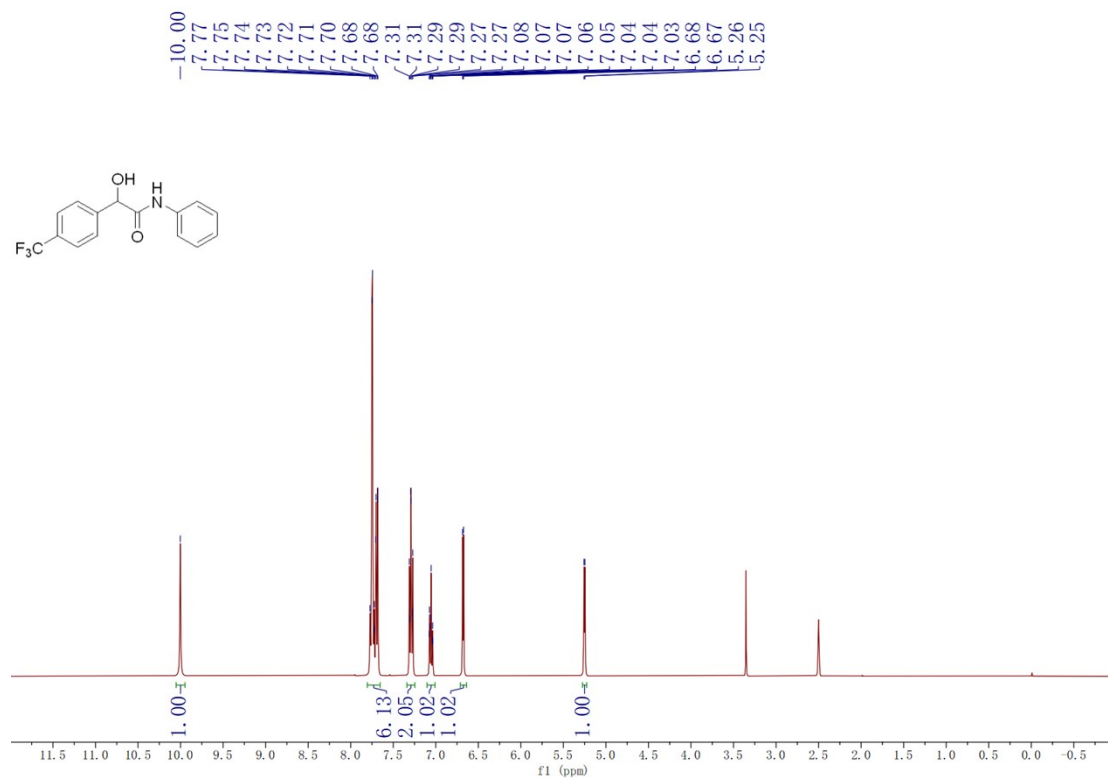
(41)¹H NMR (DMSO-*d*₆) and ¹³C NMR (CDCl₃) spectrum of **41b**



(42)¹H NMR (CDCl₃) and ¹³C NMR (CDCl₃) spectrum of **42b**



(43) ^1H NMR ($\text{DMSO-}d_6$) and ^{13}C NMR ($\text{DMSO-}d_6$) spectrum of **43b**



(44) ^1H NMR ($\text{DMSO}-d_6$) and ^{13}C NMR ($\text{DMSO}-d_6$) spectrum of **44b**

