

**Visible-light-induced Ritter-type amidation of α -hydroxy ketones in
the selectively synthesis of α,α -diamido and monoamido ketones**

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1. General Information

All α -hydroxyketones **1** except **1a** were synthesized following literature process.¹ 2-Hydroxy-1-phenylethan-1-one **1a** and all other chemicals and solvents used in the experiments were obtained from commercial sources and used directly without further treatment. The organic solvents were treated following standard procedures before use. The NMR spectra were recorded in 400 MHz apparatus, and the frequencies for ¹H NMR and ¹³C NMR test are 400 MHz and 100 MHz, respectively. The coupling of peaks were marked as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet). The chemical shifts were reported in ppm with TMS as internal standard. HMRS data for new compounds were acquired in the mass spectrometer equipped with TOF analyzer under ESI mode. Melting points were rested in an X-4A apparatus without correcting temperature.

Characterization of α -hydroxyketones **1**

Compounds **1b**, **1c**, **1d**, **1e**, **1g**, **1m**, **1n**, **1o**, **1k**; ^{1a} **1f**, **1h**, **1af**, **1ag**; ^{1b} **1i**, **1j** ^{1c}, **1l**; ^{1d} **1o**; ^{1e} have been synthesized using literature method.

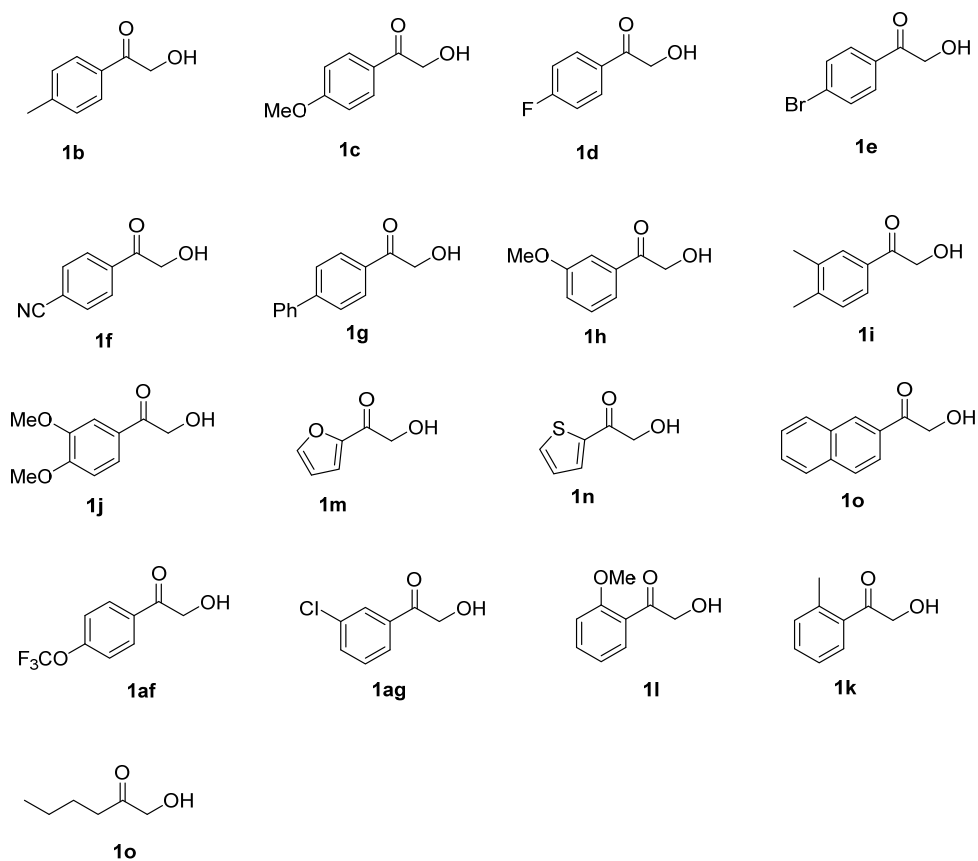


Figure S1 Structures of the α -hydroxy ketone substrates

2. Experimental Procedures

2.1 General procedure for the synthesis of **3**

To a 10 mL Schlenk tube were added α -hydroxy ketone **1** (0.5 mmol, 1.0 equiv), I_2 (1 mmol, 2.0 equiv), CF_3SO_2Na , (0.015 mmol, 0.03 equiv), H_2O (aq) (2.5 mmol, 5 equiv), and MeCN (3 mL). The mixture was then stirred at room temperature for 14 h with the irradiation of blue LEDs (10 W, 400–480 nm, $\lambda_{max} = 456.0$ nm) under an air atmosphere with a GHX-V apparatus (Hangzhou Huichuang Co., Ltd.). After completion of the reaction, the mixture was quenched with saturation $Na_2S_2O_3$ solution (10 mL), extracted with EtOAc (3×30 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The mixture was transferred into the round bottom flask, after removing the solvent under reduced pressure, the resulting residue was recrystallized with EA/PET to afford products **3**.

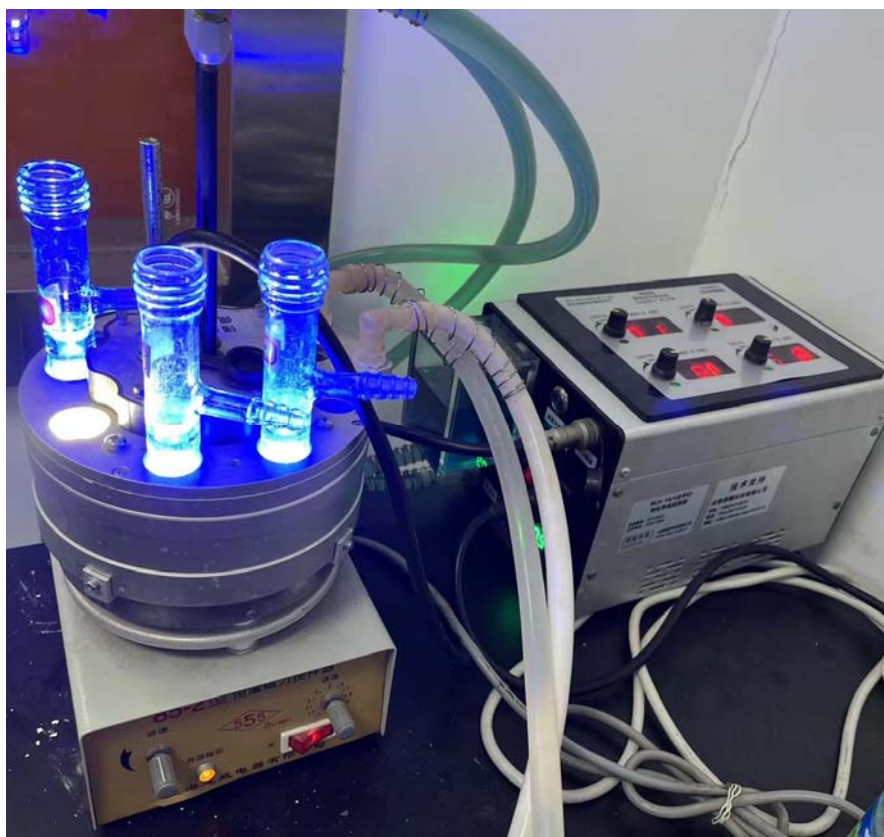


Figure S2 The set-up for the photocatalytic reactions

2.2 General procedure for the synthesis of **4**

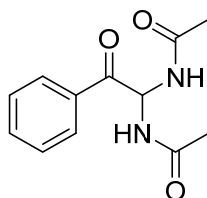
To a 10 mL Schlenk tube were added α -hydroxy ketone **1** (0.5 mmol, 2.5 equiv), I_2 (1 mmol, 5.0 equiv), CF_3SO_2Na , (0.015 mmol, 0.075 equiv), H_2O (aq) (2.5 mmol, 12.5 equiv), MeCN (0.2 mmol, 1 equiv) and DCM (2 mL). The mixture was then stirred at room temperature for 20 h with the irradiation of blue LEDs (10 W, 400–480 nm, $\lambda_{max} = 456.0$ nm) under an air atmosphere with a GHX-V apparatus (Hangzhou Huichuang Co., Ltd.). After completion of the reaction, the mixture was quenched with saturation $Na_2S_2O_3$ solution (10 mL), extracted with EtOAc (3×30 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. After the mixture was concentrated, subjected to flash chromatography on silica gel with mixed petroleum ether, ethyl acetate and methanol ($V_{PET}/V_{EA}/V_{DCM} = 32:64:1 \sim 64:32:1$) as eluent to afford pure products **4**.

2.3 Procedure for the 10 mmol scale reaction for the synthesis of 3a

A 250 mL round-bottom flask was charged with α -hydroxy ketone **1a** (10 mmol, 1 equiv), I₂ (20 mmol, 2 equiv), CF₃SO₂Na (0.3 mmol, 0.03 equiv), H₂O (aq) (50 mmol, 5 equiv), and MeCN (60 mL). The mixture was then stirred at room temperature for 24 h with the irradiation of blue LEDs following the operation in the general procedure. After completion of the reaction, the mixture was quenched with saturation Na₂S₂O₃ solution (40 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The mixture was transferred into the round bottom flask, after removing the solvent under reduced pressure, the resulting residue was recrystallized with EA/PET to afford **3a**. (1134.2 mg, 48% yield).

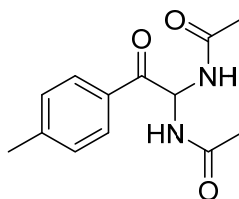
3. Characterization Data for Synthesized Compounds

3.1 Characterization data of products 3



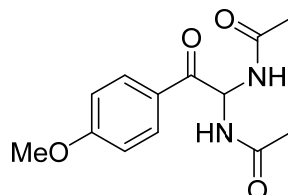
N,N'-(2-oxo-2-phenylethane-1,1-diyl)diacetamide (**3a**).³

White solid (98.1 mg, 84% yield). mp 211-214 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 8.75 (d, *J* = 7.7 Hz, 2H), 7.90 (d, *J* = 7.7 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 6.56 (t, *J* = 7.7 Hz, 1H), 1.84 (s, 6H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.8 (C), 169.8(C), 134.8(C), 133.9(CH), 129.1(CH), 128.5(CH), 58.4 (CH), 22.7(CH₃).

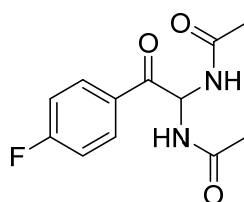


N,N'-(2-oxo-2-(p-tolyl)ethane-1,1-diyl)diacetamide (**3b**).³

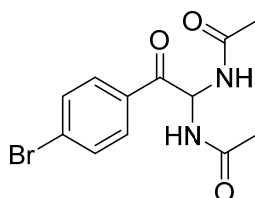
White solid (90.8 mg, 73% yield); mp 243-246 °C. $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ (ppm) = 8.69 (d, $J = 7.8$ Hz, 2H), 7.82 (d, $J = 7.8$ Hz, 2H), 7.33 (d, $J = 7.9$ Hz, 2H), 6.57 (t, $J = 7.7$ Hz, 1H), 2.37 (s, 3H), 1.84 (s, 6H). $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ (ppm) = 193.3(C), 169.6(C), 144.4(C), 132.3(C), 129.7(CH), 128.7(CH), 58.2(CH), 22.7(CH_3), 21.6(CH_3).



N,N'-(2-(4-methoxyphenyl)-2-oxoethane-1,1-diyl)diacetamide (**3c**).³ White solid (98.4 mg, 75% yield); mp 251-254 °C. $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ (ppm) = 8.66 (d, $J = 7.8$ Hz, 2H), 7.92 (d, $J = 8.5$ Hz, 2H), 7.06 (d, $J = 8.8$ Hz, 2H), 6.61 (t, $J = 7.8$ Hz, 1H), 3.85 (s, 3H), 1.85 (s, 6H). $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ (ppm) = 192.1(C), 169.5(C), 163.9(C), 131.0(C), 127.4(CH), 114.5(CH), 57.9(CH), 56.0(CH_3), 22.7(CH_3).

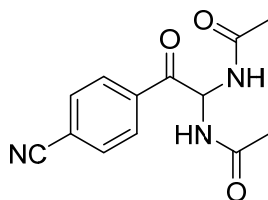


N,N'-(2-(4-fluorophenyl)-2-oxoethane-1,1-diyl)diacetamide (**3d**).³ White solid (70.3 mg, 56% yield); mp 249-252 °C. $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ (ppm) = 8.78 (d, $J = 7.6$ Hz, 2H), 8.02 – 7.91 (m, 2H), 7.36 (t, $J = 8.8$ Hz, 2H), 6.50 (t, $J = 7.6$ Hz, 1H), 1.84 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ (ppm) = 192.4(C), 169.7(C), 165.5 (C, d, $J = 252.0$ Hz), 131.6 (C), 131.5(CH), 116.2 (CH, d, $J = 22.0$ Hz), 58.5(CH), 22.7(CH_3). $^{19}\text{F NMR}$ (376 MHz, $\text{DMSO-}d_6$) δ (ppm) = -105.43.

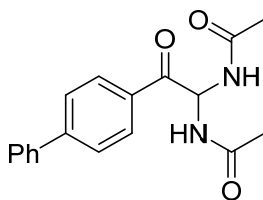


N,N'-(2-(4-bromophenyl)-2-oxoethane-1,1-diyl)diacetamide (**3e**).³ White solid (117.4 mg, 75% yield); mp 253-256 °C. $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ (ppm) = 8.79 (d, $J = 7.6$ Hz, 2H), 7.81 (d, $J = 8.6$ Hz, 2H), 7.74 (d, $J = 8.4$ Hz, 2H), 6.45 (t, $J = 7.5$

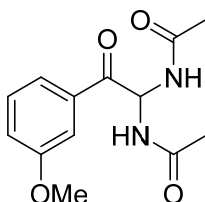
Hz, 1H), 1.83 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 193.1(C), 169.8(C), 134.0(C), 132.2(CH), 130.5(CH), 127.8(C), 58.7(CH), 22.6(CH $_3$).



***N,N'*-(2-(4-cyanophenyl)-2-oxoethane-1,1-diyl)diacetamide (3f)**. White solid (84.2 mg, 65% yield); mp 245-247 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.89 (d, J = 7.4 Hz, 2H), 8.02 – 7.95 (m, 4H), 6.40 (t, J = 7.3 Hz, 1H), 1.82 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 193.5(C), 169.9(C), 138.8(C), 133.0(CH), 129.0(CH), 118.6(C), 115.4(C), 59.2(CH), 22.6(CH $_3$). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C $_{13}$ H $_{14}$ N $_3$ O $_3$ 260.1030; Found 260.1029.

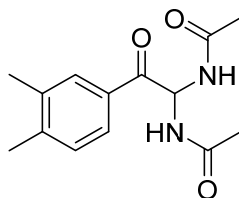


***N,N'*-(2-([1,1'-biphenyl]-4-yl)-2-oxoethane-1,1-diyl)diacetamide (3g)**. White solid (109.2 mg, 70% yield); mp 275-277 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.80 (d, J = 7.7 Hz, 2H), 7.99 (d, J = 8.3 Hz, 2H), 7.84 (d, J = 8.4 Hz, 2H), 7.76 (d, J = 7.3 Hz, 2H), 7.51 (t, J = 7.4 Hz, 2H), 7.44 (t, J = 7.3 Hz, 1H), 6.59 (t, J = 7.7 Hz, 1H), 1.86 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 193.3(C), 169.7(C), 145.2(C), 139.2(C), 133.6(C), 129.6(CH), 129.3(CH), 128.9(CH), 127.5(CH), 127.3(CH), 58.4(CH), 22.7(CH $_3$). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C $_{18}$ H $_{19}$ N $_2$ O $_3$ 311.1390; Found 311.1392.

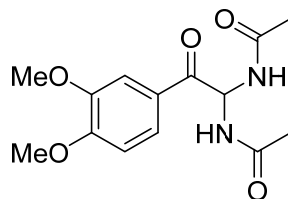


***N,N'*-(2-(3-methoxyphenyl)-2-oxoethane-1,1-diyl)diacetamide (3h)**.³ White solid (82.4 mg, 62% yield); mp 204-206 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.76 (d, J = 7.7 Hz, 2H), 7.51 – 7.40 (m, 3H), 7.22 (d, J = 6.6 Hz, 1H), 6.55 (t, J = 7.7 Hz,

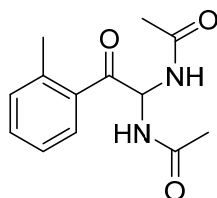
1H), 3.81 (s, 3H), 1.84 (s, 6H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.5(C), 169.7(C), 159.7(C), 136.1(C), 130.3(CH), 120.9(CH), 119.9(CH), 113.3(CH), 58.4(CH), 55.8(CH₃), 22.7(CH₃).



***N,N'*-(2-(3,4-dimethylphenyl)-2-oxoethane-1,1-diyl)diacetamide (3i)**. White solid (99.2 mg, 76% yield); mp 238-240 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 8.69 (d, *J* = 7.8 Hz, 2H), 7.69 (s, 1H), 7.64 (d, *J* = 7.9 Hz, 1H), 7.28 (d, *J* = 7.8 Hz, 1H), 6.56 (t, *J* = 7.8 Hz, 1H), 2.28 (d, *J* = 5.7 Hz, 6H), 1.84 (s, 6H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.4(C), 169.6(C), 143.3(C), 137.2(C), 132.5(C), 130.1(CH), 129.5(CH), 126.3(CH), 58.1(CH), 22.7(CH₃), 20.1(CH₃), 19.9(CH₃). **HRMS** (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₄H₁₉N₂O₃ 263.1390; Found 263.1390.

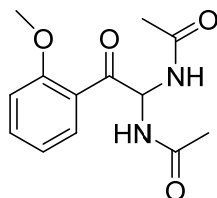


***N,N'*-(2-(3,4-dimethoxyphenyl)-2-oxoethane-1,1-diyl)diacetamide (3j)**. White solid (119.4 mg, 81% yield); mp 259-261 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 8.70 (s, 2H), 7.76 – 7.43 (m, 2H), 7.10 (s, 1H), 6.81 – 6.56 (m, 1H), 3.82 (d, *J* = 19.4 Hz, 6H), 1.85 (s, 6H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 192.1(C), 169.5(C), 153.9(C), 149.0(C), 127.2(C), 123.2(CH), 111.6(CH), 111.3(CH), 57.7(CH), 56.3(CH₃), 56.0(CH₃), 22.8(CH₃). **HRMS** (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₄H₁₉N₂O₅ 295.1289; Found 295.1289.

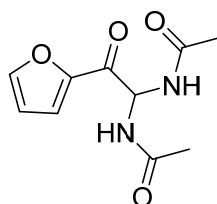


***N,N'*-(2-oxo-2-(o-tolyl)ethane-1,1-diyl)diacetamide (3k)**. White solid (85.4 mg, 69% yield); mp 229-231 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 8.77 (d, *J* = 7.5 Hz, 2H), 7.67 – 7.59 (m, 1H), 7.44 – 7.35 (m, 1H), 7.31 – 7.21 (m, 2H), 6.30 (t, *J* = 7.5 Hz,

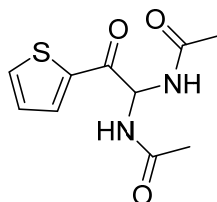
1H), 2.42 (s, 3H), 1.80 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 197.5(C), 170.0(C), 138.7(C), 135.8(C), 131.9(CH), 131.6(CH), 128.4(CH), 125.7(CH), 60.0(CH), 22.5(CH₃), 20.7(CH₃). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C₁₃H₁₇N₂O₃ 249.1234; Found 249.1239.



***N,N'*-(2-(2-methoxyphenyl)-2-oxoethane-1,1-diyl)diacetamide (3l)**. White solid (75.1 mg, 57% yield); mp 255-257 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.69 (d, J = 7.7 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.44 – 7.37 (m, 1H), 7.10 (d, J = 8.4 Hz, 1H), 6.99 (t, J = 7.5 Hz, 1H), 6.21 (t, J = 7.6 Hz, 1H), 3.84 (s, 3H), 1.77 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 197.0(C), 169.9(C), 157.7(C), 133.6(C), 130.7(CH), 126.8(CH), 120.7(CH), 112.0(CH), 62.2(CH), 56.4(CH₃), 22.5(CH₃). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C₁₃H₁₇N₂O₄ 265.1183; Found 265.1195.

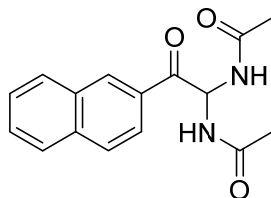


***N,N'*-(2-(furan-2-yl)-2-oxoethane-1,1-diyl)diacetamide (3m)**. White solid (79.7 mg, 71% yield); mp 274-276 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.75 (d, J = 7.2 Hz, 2H), 8.03 (s, 1H), 7.41 (s, 1H), 6.73 (s, 1H), 6.39 – 6.26 (m, 1H), 1.86 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 182.1(C), 169.8(C), 150.1(C), 148.8(CH), 119.6(CH), 113.0(CH), 58.5(CH), 22.7(CH₃). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C₁₀H₁₃N₂O₄ 225.0870; Found 225.0869.

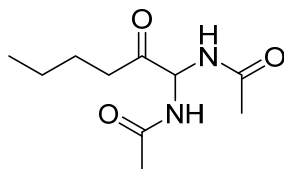


***N,N'*-(2-oxo-2-(thiophen-2-yl)ethane-1,1-diyl)diacetamide (3n)**.³ White solid (87.4 mg, 73% yield); mp 261-263 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.80 (d, J = 7.6 Hz, 2H), 8.04 (d, J = 4.3 Hz, 1H), 7.93 – 7.81 (m, 1H), 7.32 – 7.21 (m, 1H),

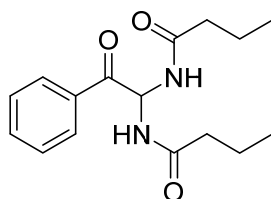
6.47 – 6.38 (m, 1H), 1.88 (d, $J = 9.3$ Hz, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 186.8(C), 169.8(C), 140.9(C), 135.8(CH), 133.5(CH), 129.2(CH), 59.1(CH), 22.7(CH₃).



***N,N'*-(2-(naphthalen-2-yl)-2-oxoethane-1,1-diyl)diacetamide (3o)**. White solid (98.4 mg, 69% yield); mp 221-223 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.86 (d, $J = 7.6$ Hz, 2H), 8.56 (s, 1H), 8.09 – 7.99 (m, 3H), 7.94 – 7.90 (m, 1H), 7.69 (t, $J = 7.0$ Hz, 1H), 7.63 (t, $J = 7.1$ Hz, 1H), 6.68 (t, $J = 7.6$ Hz, 1H), 1.84 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 198.6(C), 174.5(C), 140.3(C), 137.1(C), 137.0(C), 134.9(CH), 134.8(CH), 134.0(CH), 133.5(CH), 132.9(CH), 132.3(CH), 129.1(CH), 63.4(CH), 27.4(CH₃). HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C₁₆H₁₇N₂O₃ 285.1234; Found 285.1233.

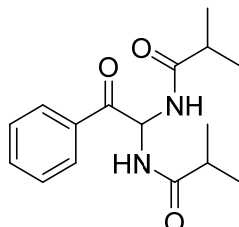


***N,N'*-(2-oxohexane-1,1-diyl)diacetamide (3p)**. White solid (44.8 mg, 42% yield); mp 166-168 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.62 (d, $J = 7.6$ Hz, 2H), 5.45 (t, $J = 7.6$ Hz, 1H), 2.44 (t, $J = 7.3$ Hz, 2H), 1.87 (s, 6H), 1.47 – 1.39 (m, 2H), 1.29 – 1.20 (m, 2H), 0.85 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 204.5(C), 170.1(C), 61.7(CH), 37.5(CH), 25.7(CH), 22.6(CH), 22.1(CH₃), 14.2(CH₃). HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for C₁₀H₁₉N₂O₃ 215.1390; Found 215.1400.

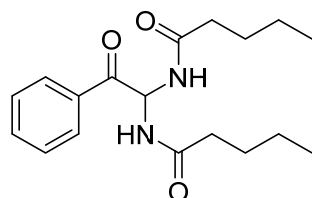


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)dibutyramide (3q)**. White solid (127.5mg, 88% yield); mp 220-222 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.69 (d, $J =$

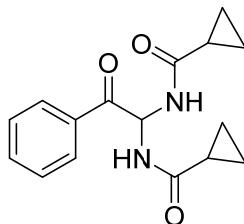
7.7 Hz, 2H), 7.88 (d, $J = 7.5$ Hz, 2H), 7.63 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 2H), 6.54 (t, $J = 7.6$ Hz, 1H), 2.09 (t, $J = 7.2$ Hz, 4H), 1.51 – 1.39 (m, 4H), 0.76 (t, $J = 7.4$ Hz, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 194.0(C), 172.5(C), 135.0(C), 133.7(CH), 128.9(CH), 128.5(CH), 58.6(CH), 37.2(CH $_2$), 18.9(CH $_2$), 13.8(CH $_3$). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{23}\text{N}_2\text{O}_3$ 291.1703; Found 291.1703.



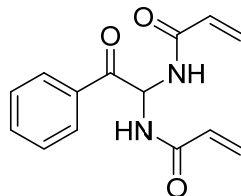
***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(2-methylpropanamide) (3r)**. White solid (100.4mg, 69% yield); mp 214-216 °C. ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 8.00 (d, $J = 7.3$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 3H), 7.43 (t, $J = 7.7$ Hz, 2H), 6.16 (s, 1H), 2.45 – 2.34 (m, 2H), 1.09 (d, $J = 6.9$ Hz, 6H), 1.03 (d, $J = 6.8$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 193.1(C), 177.2(C), 134.3(C), 133.3(CH), 128.6(CH), 128.3(CH), 59.8(CH), 35.0(CH), 19.1(CH $_3$), 19.0(CH $_3$). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{23}\text{N}_2\text{O}_3$ 291.1703; Found 291.1703.



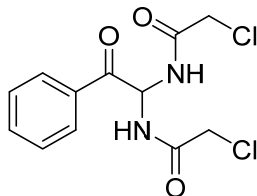
***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)dipentanamide (3s)**. White solid (87.3 mg, 55% yield); mp 186-188 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 8.69 (d, $J = 7.6$ Hz, 2H), 7.87 (d, $J = 7.4$ Hz, 2H), 7.62 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.6$ Hz, 2H), 6.51 (t, $J = 7.6$ Hz, 1H), 2.10 (t, $J = 7.3$ Hz, 4H), 1.45 – 1.36 (m, 4H), 1.20 – 1.11 (m, 4H), 0.79 (t, $J = 7.3$ Hz, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 194.0(C), 172.6(C), 135.0(C), 133.6(CH), 128.9(CH), 128.5(CH), 58.6(CH), 34.9(CH $_2$), 27.6(CH $_2$), 22.0(CH $_2$), 14.1(CH $_3$). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{27}\text{N}_2\text{O}_3$ 319.2016; Found 319.2016.



***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)dicyclopropanecarboxamide (3t)**. White solid (101.9 mg, 71% yield); mp 269-271 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 9.00 (d, *J* = 7.7 Hz, 2H), 7.87 (d, *J* = 7.2 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 2H), 6.62 (t, *J* = 7.7 Hz, 1H), 1.74 – 1.65 (m, 2H), 0.70 – 0.61 (m, 8H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.9(C), 173.1(C), 134.9(C), 133.8(CH), 129.1(CH), 128.5(CH), 58.5(CH), 13.7(CH), 7.22(CH₂), 7.16(CH₂). HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₆H₁₉N₂O₃ 287.1390; Found 287.1390.

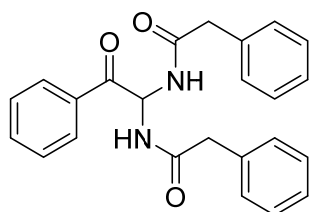


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)diacrylamide (3u)**. White solid (63.0mg, 49% yield); mp 294-296 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 9.11 (d, *J* = 7.5 Hz, 2H), 7.92 (d, *J* = 7.7 Hz, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 2H), 6.72 (t, *J* = 7.5 Hz, 1H), 6.34 (dd, *J* = 17.1, 10.2 Hz, 2H), 6.14 (d, *J* = 17.0 Hz, 2H), 5.66 (d, *J* = 11.1 Hz, 2H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.4(C), 164.8(C), 134.7(C), 134.0(CH), 131.2(CH), 129.2(CH), 128.6(CH₂), 127.4(CH), 58.7(CH). HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₄H₁₅N₂O₃ 259.1077; Found 259.1077.

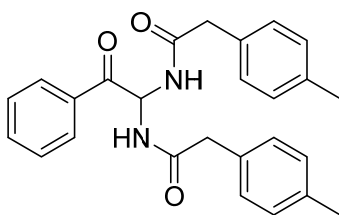


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(2-chloroacetamide) (3v)**. White solid (61.4 mg, 41% yield); mp 201-203 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 9.26 (d, *J* = 7.5 Hz, 2H), 7.91 (d, *J* = 7.4 Hz, 2H), 7.69 – 7.50 (m, 3H), 6.57 (t, *J* = 7.6 Hz, 1H), 4.14 (s, 4H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 192.9(C), 166.4(C),

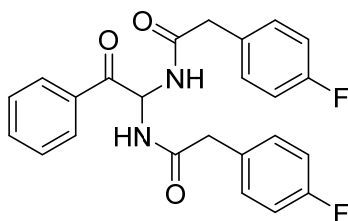
134.3(C), 134.2(CH), 129.1(CH), 128.8(CH), 59.1(CH), 42.6(CH₂); **HRMS** (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₁₂H₁₃Cl₂N₂O₃ 303.0298; Found 303.0298.



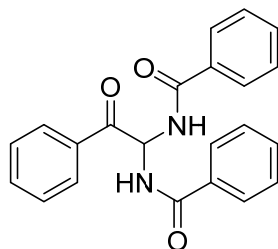
***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(2-phenylacetamide) (3w)**. White solid (104.7 mg, 54% yield); mp 197-199 °C. **¹H NMR** (400 MHz, DMSO-*d*₆) δ (ppm) = 9.06 (d, J = 7.6 Hz, 2H), 7.85 (d, J = 7.5 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 7.27 – 7.12 (m, 10H), 6.53 (t, J = 7.6 Hz, 1H), 3.49 (s, 4H). **¹³C NMR** (100 MHz, DMSO-*d*₆) δ (ppm) = 193.8(C), 170.6(C), 136.2(C), 134.8(C), 133.7(CH), 129.4(CH), 129.0(CH), 128.6(CH), 128.6(CH), 126.8(CH), 59.0(CH), 42.1(CH₂). **HRMS** (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₂₄H₂₃N₂O₃ 387.1703; Found 387.1704.



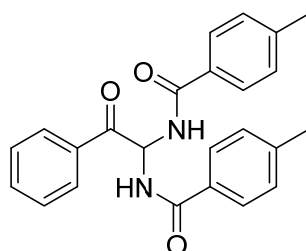
***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(2-(p-tolyl)acetamide) (3x)**. White solid (142.4 mg, 69% yield); mp 224-226 °C. **¹H NMR** (400 MHz, DMSO-*d*₆) δ (ppm) = 8.98 (d, J = 7.6 Hz, 2H), 7.86 – 7.82 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.8 Hz, 2H), 7.03 (s, 8H), 6.51 (t, J = 7.6 Hz, 1H), 3.43 (s, 4H), 2.26 (s, 6H). **¹³C NMR** (100 MHz, DMSO-*d*₆) δ (ppm) = 193.8(C), 170.7(C), 135.8(CH), 134.8(C), 133.7(CH), 133.1(C), 129.3(CH), 129.2(CH), 128.9(CH), 128.6(CH), 58.9(CH), 41.7(CH₂), 21.1(CH₃). **HRMS** (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₂₆H₂₇N₂O₃ 415.2016; Found 415.2018.



***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(2-(4-fluorophenyl)acetamide) (3y)**. White solid (135.3 mg, 64% yield); mp 227-229 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 9.07 (d, *J* = 7.6 Hz, 2H), 7.82 (d, *J* = 7.7 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.22 – 7.12 (m, 4H), 7.05 (t, *J* = 8.8 Hz, 4H), 6.49 (t, *J* = 7.5 Hz, 1H), 3.47 (s, 4H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.8(C), 170.5(C), 161.5 (d, *J* = 242.0 Hz, C), 134.8(C), 133.7(C), 132.3 (d, *J* = 3.1 Hz, CH), 131.3(CH), 131.2(CH), 128.7 (d, *J* = 37.2 Hz, CH), 115.3 (d, *J* = 21.0 Hz, CH), 59.0(CH), 41.1(CH₂). ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ (ppm) = -116.75. HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₄H₂₁F₂N₂O₃ 423.1515; Found 423.1517.

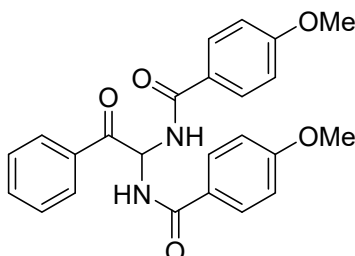


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)dibenzamide (3z)**. White solid (97.3 mg, 54% yield); mp 249-251 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 9.29 (d, *J* = 7.4 Hz, 2H), 8.03 (d, *J* = 7.4 Hz, 2H), 7.92 (d, *J* = 7.3 Hz, 4H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.59 – 7.44 (m, 8H), 7.06 (t, *J* = 7.4 Hz, 1H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm) = 193.1(C), 166.6(C), 134.8(C), 134.0(C), 133.7(CH), 132.3(CH), 129.2(CH), 128.9(CH), 128.6(CH), 128.0(CH), 60.2(CH). HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₂H₁₉N₂O₃ 359.1390; Found 359.1391.

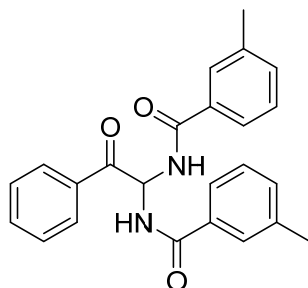


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(4-methylbenzamide) (3aa)**. White solid (117.3 mg, 61% yield); mp 250-252 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 9.16 (d, *J* = 7.4 Hz, 2H), 8.02 (d, *J* = 7.7 Hz, 2H), 7.82 (d, *J* = 7.9 Hz, 4H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 4H), 7.03 (t, *J* = 7.4 Hz, 1H),

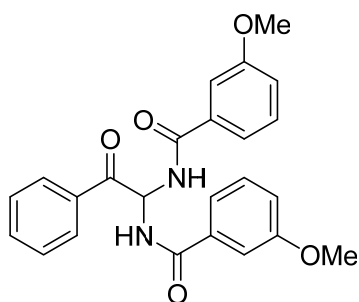
2.35 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 193.2(C), 166.4(C), 142.3(C), 134.9(C), 133.9(CH), 131.0(C), 129.4(CH), 129.2(CH), 128.6(CH), 128.0(CH), 60.0(CH), 21.5(CH₃). HRMS (ESI-TOF) m/z : [M + H]⁺ Calcd for C₂₄H₂₃N₂O₃ 387.1703; Found 387.1704.



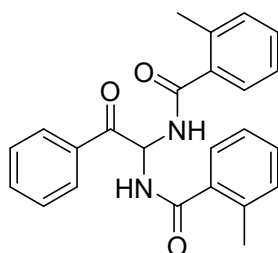
N,N'-(2-oxo-2-phenylethane-1,1-diyl)bis(4-methoxybenzamide) (**3ab**). White solid (146.8 mg, 70% yield); mp 256-258 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 9.03 (d, J = 7.4 Hz, 2H), 8.01 (d, J = 7.3 Hz, 2H), 7.88 (d, J = 8.8 Hz, 4H), 7.63 (t, J = 7.4 Hz, 1H), 7.53 (t, J = 7.6 Hz, 2H), 7.05 – 6.97 (m, 5H), 3.81 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 193.4(C), 165.9(C), 162.5(C), 134.9(CH), 133.9(C), 129.9(C), 129.2(CH), 128.6(CH), 125.9(CH), 114.1(CH), 60.0(CH), 55.9(CH₃). HRMS (ESI-TOF) m/z : [M + H]⁺ Calcd for C₂₄H₂₃N₂O₅ 419.1602; Found 419.1603.



N,N'-(2-oxo-2-phenylethane-1,1-diyl)bis(3-methylbenzamide) (**3ac**). White solid (125.1 mg, 65% yield); mp 205-207 °C. ^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 9.17 (d, J = 7.5 Hz, 2H), 8.02 (d, J = 7.4 Hz, 2H), 7.76 – 7.67 (m, 4H), 7.64 (t, J = 7.4 Hz, 1H), 7.54 (t, J = 7.6 Hz, 2H), 7.40 – 7.34 (m, 4H), 7.04 (t, J = 7.5 Hz, 1H), 2.36 (s, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ (ppm) = 193.1(C), 166.6(C), 138.2(C), 134.8(C), 134.0(CH), 133.7(CH), 132.9(CH), 129.2(CH), 128.8(CH), 128.6(CH), 128.5(CH), 125.2(CH), 60.0(CH), 21.3(CH₃). HRMS (ESI-TOF) m/z : [M + H]⁺ Calcd for C₂₄H₂₃N₂O₃ 387.1703; Found 387.1704.

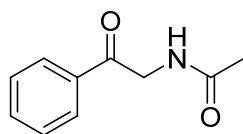


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(3-methoxybenzamide) (3ad).** White solid (141.7 mg, 68% yield); mp 154-156 °C. **¹H NMR** (400 MHz, DMSO-*d*₆) δ (ppm) = 9.27 (d, *J* = 7.4 Hz, 2H), 8.03 (d, *J* = 7.3 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.57 – 7.46 (m, 6H), 7.41 (t, *J* = 7.9 Hz, 2H), 7.18 – 7.11 (m, 2H), 7.06 (t, *J* = 7.4 Hz, 1H), 3.81 (s, 6H). **¹³C NMR** (100 MHz, DMSO-*d*₆) δ (ppm) = 193.0(C), 166.3(C), 159.7(C), 135.1(CH), 134.8(C), 134.0(CH), 130.0(CH), 129.2(CH), 128.6(CH), 120.3(CH), 118.2(CH), 113.2(CH), 60.2(CH), 55.8(CH₃). **HRMS** (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₄H₂₃N₂O₅ 419.1602; Found 419.1604.

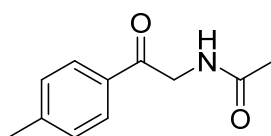


***N,N'*-(2-oxo-2-phenylethane-1,1-diyl)bis(2-methylbenzamide) (3ae).** White solid (88.3 mg, 46% yield); mp 190-192 °C. **¹H NMR** (400 MHz, DMSO-*d*₆) δ (ppm) = 9.15 (d, *J* = 7.5 Hz, 2H), 8.01 (d, *J* = 7.2 Hz, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.32 (m, 4H), 7.26 – 7.21 (m, 4H), 6.90 (t, *J* = 7.5 Hz, 1H), 2.29 (s, 6H). **¹³C NMR** (100 MHz, DMSO-*d*₆) δ (ppm) = 193.4(C), 169.2(C), 136.3(C), 136.1(CH), 134.9(C), 133.9(CH), 131.0(CH), 130.2(CH), 129.1(CH), 128.7(CH), 127.7(CH), 125.9(CH), 59.8(CH), 19.7(CH₃). **HRMS** (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₄H₂₃N₂O₃ 387.1703; Found 387.1704.

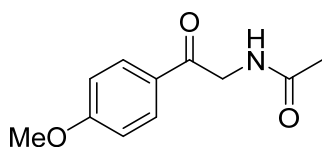
4.2 Characterization data of products 4



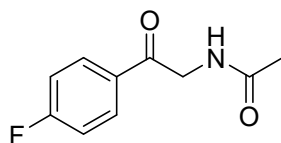
***N*-(2-oxo-2-phenylethyl)acetamide (4a).**⁴ Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1; White solid (19.4 mg, 55% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.99 (d, *J* = 7.4 Hz, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 2H), 6.62 (s, 1H), 4.78 (d, *J* = 4.0 Hz, 2H), 2.12 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 194.2(C), 170.3(C), 134.3(C), 134.2(CH), 129.0(CH), 128.0(CH), 46.6(CH₂), 23.1(CH₃).



***N*-(2-oxo-2-(p-tolyl)ethyl)acetamide (4b).** Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1; White solid (12.9 mg, 34% yield); mp 98-100 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.80 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 6.61 (s, 1H), 4.67 (d, *J* = 4.2 Hz, 2H), 2.35 (s, 3H), 2.03 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 193.8(C), 170.4(C), 145.3(C), 131.9(C), 129.6(CH), 128.0(CH), 46.4(CH₂), 23.1(CH₃), 21.8(CH₃). HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₁H₁₄NO₂ 192.1019; Found 192.1036.

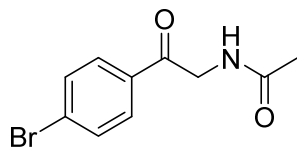


***N*-(2-(4-methoxyphenyl)-2-oxoethyl)acetamide (4c).**⁴ Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1; White solid (18.3 mg, 44% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.89 (d, *J* = 8.9 Hz, 2H), 6.90 (d, *J* = 8.9 Hz, 2H), 6.57 (s, 1H), 4.64 (d, *J* = 4.2 Hz, 2H), 3.82 (s, 3H), 2.04 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 192.6(C), 170.4(C), 164.4(C), 130.3(C), 127.3(CH), 114.2(CH), 55.6(CH₂), 46.2(CH₃), 23.1(CH₃).

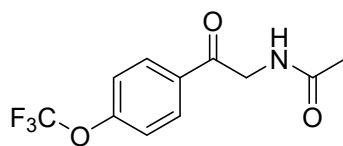


***N*-(2-(4-fluorophenyl)-2-oxoethyl)acetamide (4d).** Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1 ; White solid (11.4 mg, 29% yield); mp 138-140 °C. ¹H NMR (400 MHz,

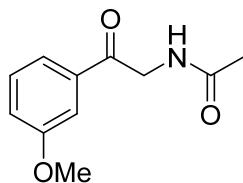
CDCl₃) δ (ppm) = 7.97 – 7.92 (m, 2H), 7.11 (t, J = 8.6 Hz, 2H), 6.55 (s, 1H), 4.67 (d, J = 4.3 Hz, 2H), 2.04 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 192.7(C), 170.4(C), 166.3 (d, J = 256.7 Hz, C), 162.6(C), 130.7 (d, J = 9.5 Hz, CH), 116.2 (d, J = 22.1 Hz, CH), 46.4(CH₂), 23.0(CH₃). ¹⁹F NMR (376 MHz, CDCl₃) δ (ppm) = -102.82. HRMS (ESI-TOF) m/z : [M + H]⁺ Calcd for C₁₀H₁₁FNO₂ 196.0769; Found 196.0770.



***N*-(2-(4-bromophenyl)-2-oxoethyl)acetamide (4e).**⁴ Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1; White solid (16.0 mg, 31% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.77 (d, J = 8.5 Hz, 2H), 7.58 (d, J = 8.4 Hz, 2H), 6.53 (s, 1H), 4.66 (d, J = 4.3 Hz, 2H), 2.04 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 193.4(C), 170.4(C), 133.0(CH), 132.3(C), 129.6(CH), 129.4(C), 46.5(CH₂), 23.1(CH₃).

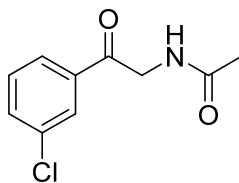


***N*-(2-oxo-2-(4-(trifluoromethoxy)phenyl)ethyl)acetamide (4f).** Eluent: V_{PET}/V_{EA}/V_{DCM} = 64:32:1; White solid (19.2 mg, 37% yield). mp 167-169 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 8.09 – 8.01 (m, 2H), 7.37 – 7.30 (m, 2H), 6.58 (s, 1H), 4.76 (d, J = 4.3 Hz, 2H), 2.12 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 192.8(C), 170.3(C), 153.4(C), 132.5(CH), 130.1(C), 120.6(CH), 120.2 (d, J = 259.3 Hz, CH), 46.5(CH₂), 23.0(CH₃). ¹⁹F NMR (376 MHz, CDCl₃) δ (ppm) = -57.61. HRMS (ESI-TOF) m/z : [M + H]⁺ Calcd for C₁₁H₁₁F₃NO₃ 262.0686; Found 262.0691.

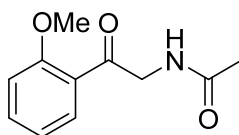


***N*-(2-(3-methoxyphenyl)-2-oxoethyl)acetamide (4g).**⁴ Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1; White solid (16.3 mg, 39% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.49 (d, J = 7.7 Hz, 1H), 7.44 – 7.40 (m, 1H), 7.33 (t, J = 7.9 Hz, 1H), 7.12 – 7.07 (m, 1H), 6.54 (s, 1H), 4.69 (d, J = 4.3 Hz, 2H), 3.79 (s, 3H), 2.04 (s, 3H). ¹³C NMR (100

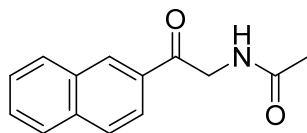
MHz, CDCl₃) δ (ppm) = 194.1(C), 170.3(C), 160.0(C), 135.6(C), 130.0(CH), 120.8(CH), 120.5(CH), 112.1(CH), 55.5(CH₃), 46.7(CH₂), 23.1(CH₃).



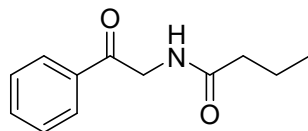
***N*-(2-(3-chlorophenyl)-2-oxoethyl)acetamide (4h).**⁴ Eluent: V_{PET}/V_{EA}/V_{DCM} = 64:32:1; White solid (14.1 mg, 33% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.97 (s, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.64 – 7.57 (m, 1H), 7.46 (t, *J* = 7.9 Hz, 1H), 6.57 (s, 1H), 4.75 (d, *J* = 4.3 Hz, 2H), 2.12 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 193.2(C), 170.3(C), 135.9(C), 135.4(C), 134.1(CH), 130.3(CH), 128.1(CH), 126.0(CH), 46.6(CH₂), 23.0(CH₃).



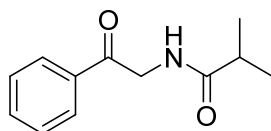
***N*-(2-(2-methoxyphenyl)-2-oxoethyl)acetamide (4i).** Eluent: V_{PET}/V_{EA}/V_{DCM} = 64:32:1; White solid (17.1 mg, 41% yield). mp 120-122 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.91 – 7.81 (m, 1H), 7.50 – 7.41 (m, 1H), 7.00 – 6.89 (m, 2H), 6.64 (s, 1H), 4.64 (d, *J* = 4.5 Hz, 2H), 3.88 (s, 3H), 2.01 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 195.1(C), 170.2(C), 159.8(C), 135.1(CH), 131.0(CH), 124.4(C), 120.8(CH), 111.7(CH), 55.7(CH₃), 51.2(CH₂), 23.2(CH₃). HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₁H₁₄NO₃ 208.0987; Found 208.0987.



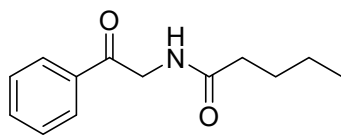
***N*-(2-(naphthalen-2-yl)-2-oxoethyl)acetamide (4j).**⁴ Eluent: V_{PET}/V_{EA}/V_{DCM} = 32:64:1; White solid (19.2 mg, 42% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 8.43 (s, 1H), 7.96 – 7.90 (m, 1H), 7.89 – 7.76 (m, 3H), 7.58 – 7.45 (m, 2H), 6.66 (s, 1H), 4.83 (d, *J* = 4.2 Hz, 2H), 2.06 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 194.1(C), 170.5(C), 136.1(C), 132.4(C), 131.6(C), 130.0(CH), 129.7(CH), 129.1(CH), 128.9(CH), 127.9(CH), 127.2(CH), 123.2(CH), 46.7(CH₂), 23.1(CH₃).



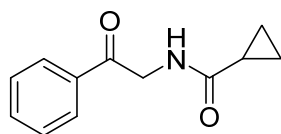
***N*-(2-oxo-2-phenylethyl)butyramide (4k).** Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$;
Colorless liquid (17.3 mg, 42% yield). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.91 (d, $J = 7.3$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 6.54 (s, 1H), 4.71 (d, $J = 4.2$ Hz, 2H), 2.23 (t, $J = 7.5$ Hz, 2H), 1.70 – 1.60 (m, 2H), 0.91 (t, $J = 7.3$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 194.4(C), 173.3(C), 134.4(C), 134.2(CH), 128.9(CH), 127.9(CH), 46.4(CH_2), 38.5(CH_2), 19.1(CH_2), 13.8(CH_3). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{12}\text{H}_{16}\text{NO}_2$ 206.1176; Found 206.1178.



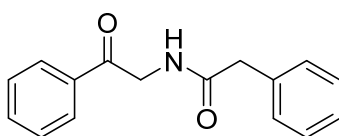
***N*-(2-oxo-2-phenylethyl)isobutyramide (4l).** Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$;
Colorless liquid (16.6 mg, 40% yield). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.98 (d, $J = 8.3$ Hz, 2H), 7.62 (t, $J = 7.3$ Hz, 1H), 7.50 (t, $J = 7.7$ Hz, 2H), 6.67 (s, 1H), 4.77 (d, $J = 4.3$ Hz, 2H), 2.59 – 2.47 (m, 1H), 1.22 (d, $J = 6.9$ Hz, 6H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 194.5(C), 177.2(C), 134.4(C), 134.1(CH), 128.9(CH), 127.9(CH), 46.4(CH_2), 35.5(CH), 19.6(CH_3). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{12}\text{H}_{16}\text{NO}_2$ 206.1176; Found 206.1178.



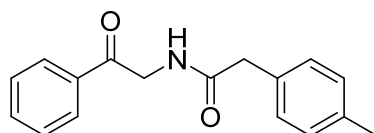
***N*-(2-oxo-2-phenylethyl)pentanamide (4m).** Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$;
Colorless liquid (16.7 mg, 38% yield). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.99 (d, $J = 8.4$ Hz, 2H), 7.64 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.7$ Hz, 2H), 6.62 (s, 1H), 4.79 (d, $J = 4.2$ Hz, 2H), 2.33 (t, $J = 7.7$ Hz, 2H), 1.74 – 1.63 (m, 2H), 1.45 – 1.34 (m, 2H), 0.95 (t, $J = 7.3$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 194.4(C), 173.5(C), 134.4, 134.2(C), 128.9(CH), 127.9(CH), 46.4(CH_2), 36.3(CH_2), 27.8(CH_2), 22.4(CH_2), 13.8(CH_3). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{18}\text{NO}_2$ 220.1332; Found 220.1330.



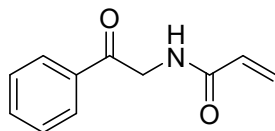
***N*-(2-oxo-2-phenylethyl)cyclopropanecarboxamide (4n).**⁴ Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$; White solid (16.2 mg, 40% yield). **¹H NMR** (400 MHz, CDCl_3) δ (ppm) = 7.92 (d, $J = 7.4$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 6.71 (s, 1H), 4.73 (d, $J = 4.2$ Hz, 2H), 1.54 – 1.44 (m, 1H), 1.00 – 0.89 (m, 2H), 0.78 – 0.68 (m, 2H). **¹³C NMR** (100 MHz, CDCl_3) δ (ppm) = 194.5(C), 173.8(C), 134.4(C), 134.1(CH), 128.9(CH), 127.9(CH), 46.7(CH₂), 14.7(CH), 7.4(CH₂).



***N*-(2-oxo-2-phenylethyl)-2-phenylacetamide (4o).** Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$; Colorless liquid (22.9 mg, 45% yield). **¹H NMR** (400 MHz, CDCl_3) δ (ppm) = 7.85 (d, $J = 7.3$ Hz, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.43 – 7.20 (m, 7H), 6.52 (s, 1H), 4.65 (d, $J = 4.4$ Hz, 2H), 3.60 (s, 2H). **¹³C NMR** (100 MHz, CDCl_3) δ (ppm) = 194.0(C), 171.2(C), 134.6(C), 134.3(C), 134.2(CH), 129.5(CH), 129.0(CH), 128.9(CH), 127.9(CH), 127.5(CH), 46.5(CH₂), 43.6(CH₂). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{16}\text{NO}_2$ 254.1176; Found 254.1175.



***N*-(2-oxo-2-phenylethyl)-2-(p-tolyl)acetamide (4p).** Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$; Colorless liquid (25.4 mg, 48% yield). **¹H NMR** (400 MHz, CDCl_3) δ (ppm) = 7.88 – 7.82 (m, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.7$ Hz, 2H), 7.16 – 7.08 (m, 4H), 6.51 (s, 1H), 4.65 (d, $J = 4.4$ Hz, 2H), 3.56 (s, 2H), 2.28 (s, 3H). **¹³C NMR** (100 MHz, CDCl_3) δ (ppm) = 194.1(C), 171.6(C), 137.1(C), 134.3(C), 134.2(CH), 131.4(C), 129.8(CH), 129.4(CH), 128.9(CH), 127.9(CH), 46.5(CH₂), 43.2(CH₂), 21.1(CH₃). **HRMS** (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{18}\text{NO}_2$ 268.1332; Found 268.1330.

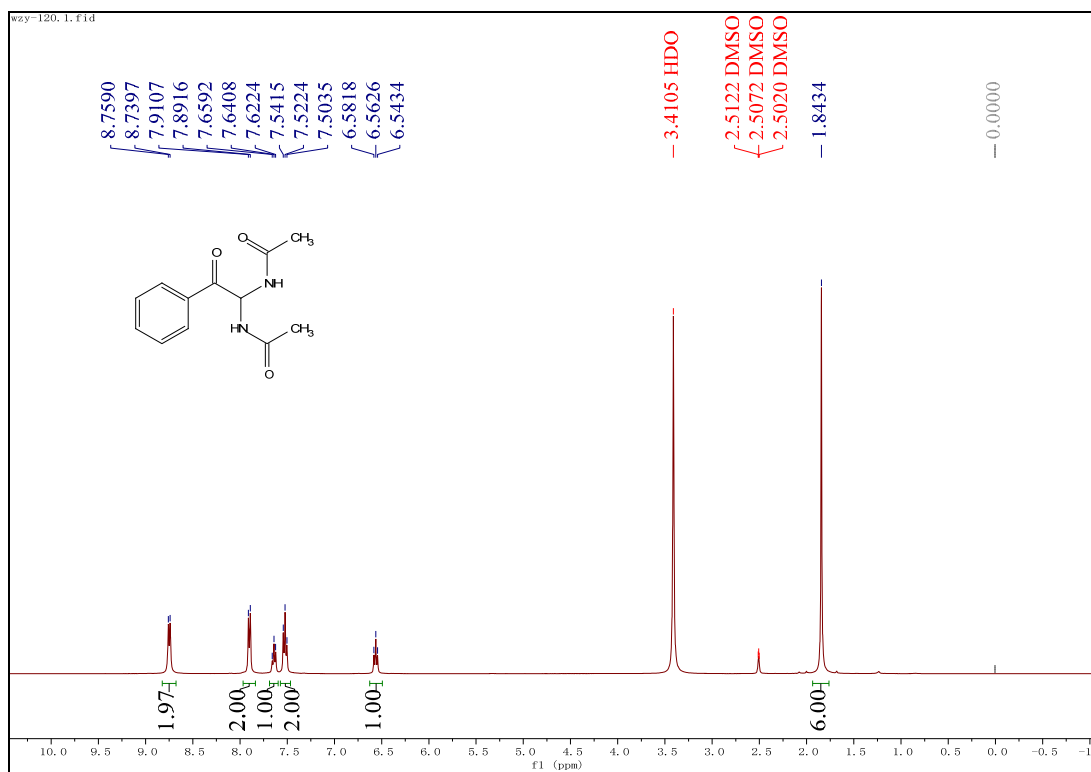


***N*-(2-oxo-2-phenylethyl)acrylamide (4q).**⁴ Eluent: $V_{\text{PET}}/V_{\text{EA}}/V_{\text{DCM}} = 64:32:1$; White solid (13.7 mg, 36% yield). ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.99 – 7.89 (m, 2H), 7.62 – 7.51 (m, 1H), 7.50 – 7.40 (m, 2H), 6.72 (s, 1H), 6.29 (dd, $J = 17.1, 1.7$ Hz, 1H), 6.19 (dd, 1H), 5.65 (dd, $J = 9.9, 1.8$ Hz, 1H), 4.79 (d, $J = 4.3$ Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 194.1(C), 165.6(C), 134.34(C), 134.26(CH), 130.4(CH), 129.0(CH), 128.0(CH), 127.1(CH₂), 46.5(CH₂).

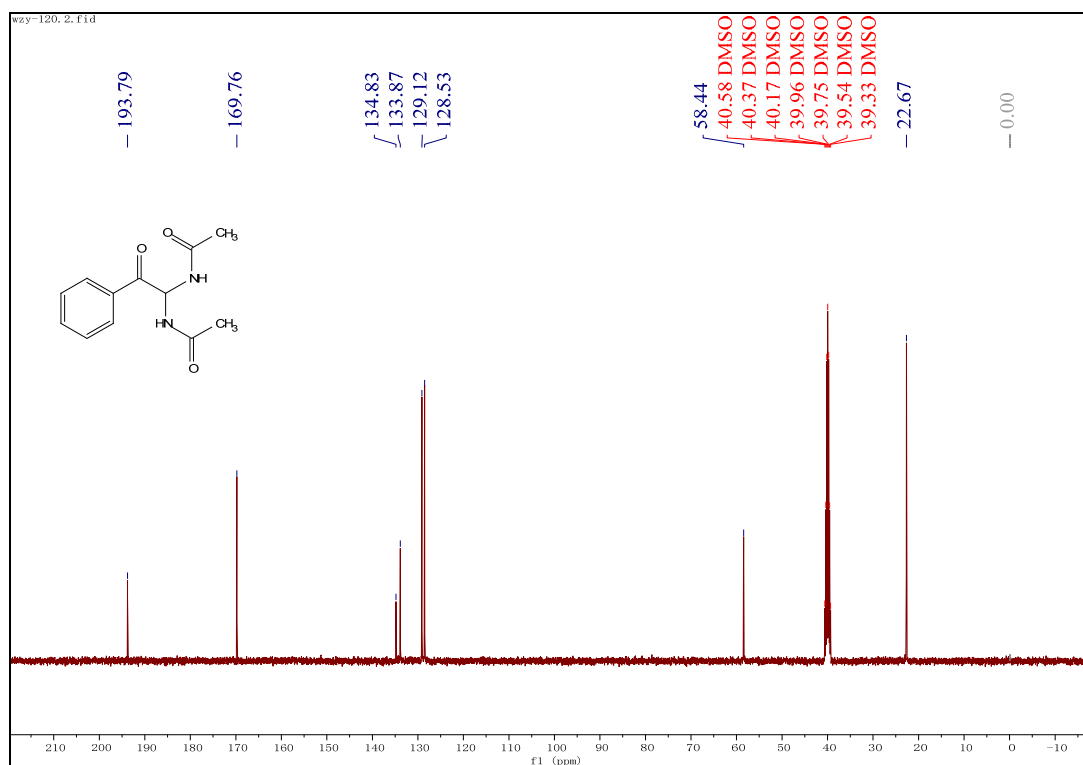
4. References

1. (a) Zhang Z.; Jiang, X. *Org. Lett.* **2014**, *16*, 17, 4400–4403. (b) Zhang, Y.; He, L.; Shi, L. *Adv. Synth. Catal.* **2018**, *360*, 1926-1931. (c) Gan, L.; Liu, Y.; Wang, C.; Wan, J.-P. *ChemistrySelect* **2023**, *8*, e202301791. (d) Pan, T.; Gao, X.; Yang, S.; Wang, L.; Hu, Y.; Liu, M.; Wang, W.; Wu, Y.; Zheng, B.; Guo, H. *Org. Lett.* **2021**, *23*, 5750-5754. (e) Liu, Y.; Yi, Z.; Yang, X.; Wang, H.; Yin, C.; Wang, M.; Dong, X.-Q.; Zhang, X. *ACS Catal.* **2020**, *10*, 11153-11161.
2. Yin, G. D.; Gao, M.; She, N. F.; Hu, S. L.; Wu, A. X.; Pan, Y. *J. Synthesis* **2007**, *20*, 3113.
3. Khan, S.; Kumar, A.; Gupta, R.; Ahmed, Q. N. *ChemistrySelect* **2017**, *2*, 11336.
4. Wang, Y.; Yang, M.; Lao, C.; Jiang, Z. *Org. Lett.* **2022**, *24*, 2625-2629.

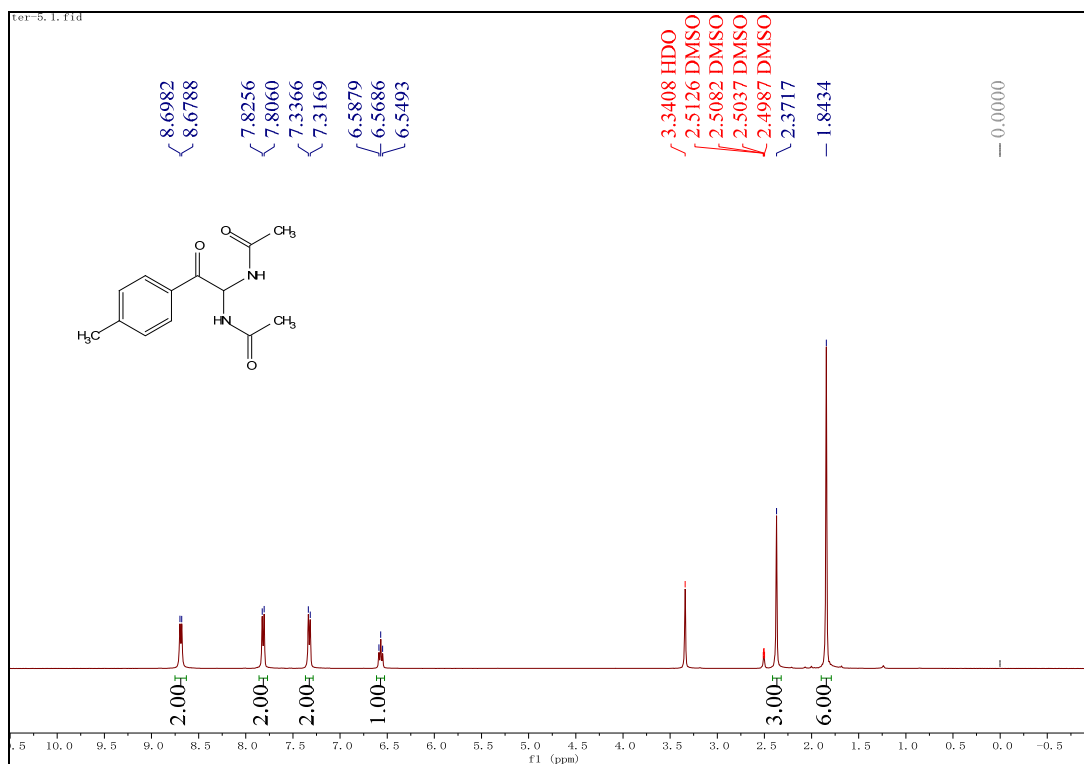
5. ^1H and ^{13}C NMR Spectra for Synthesized Compounds



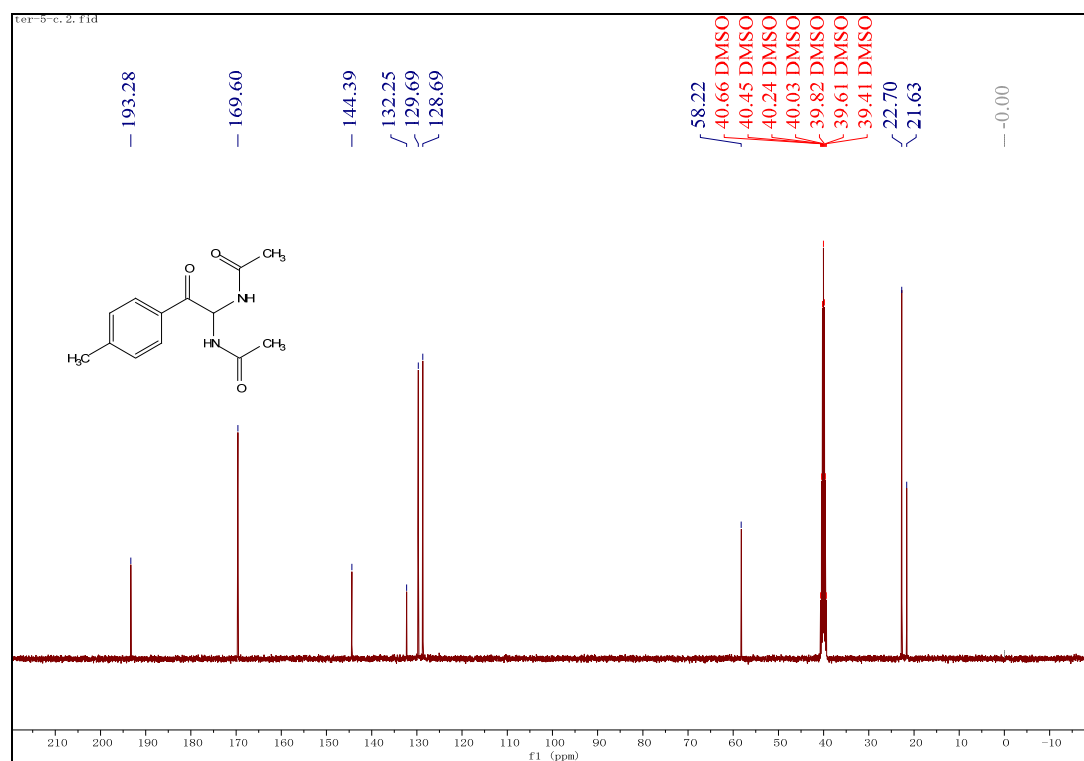
^1H NMR spectrum of **3a** (400 MHz, $\text{DMSO}-d_6$)



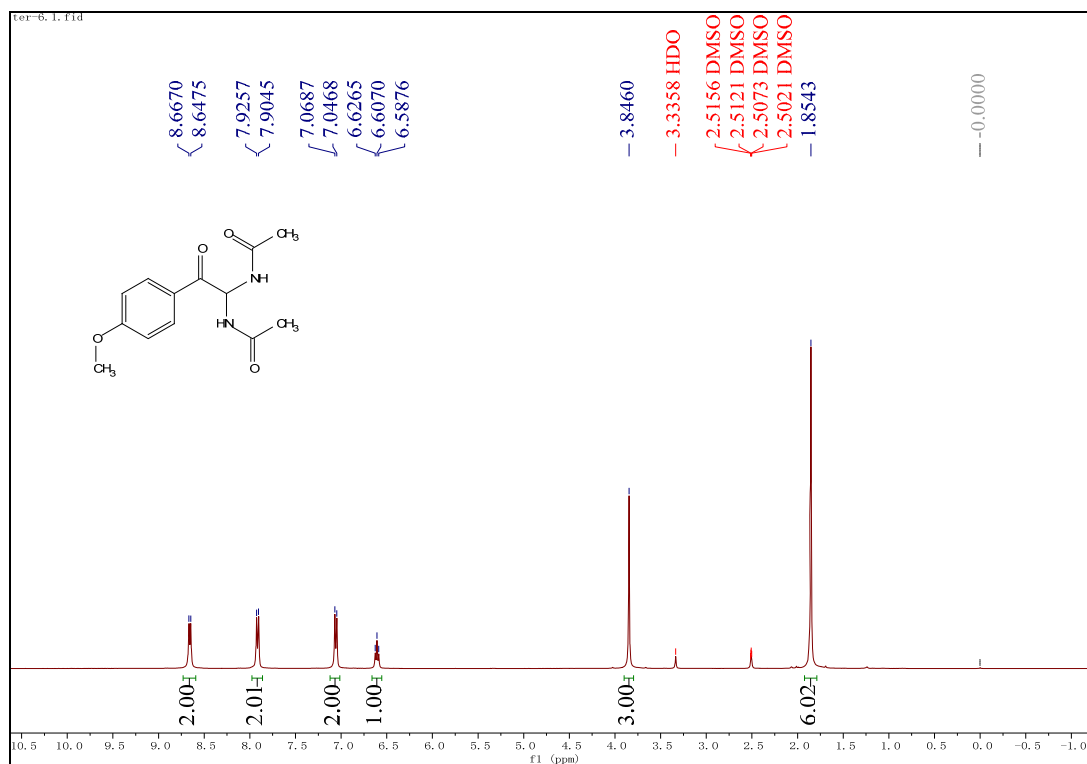
^{13}C NMR spectrum of **3a** (100 MHz, $\text{DMSO}-d_6$)



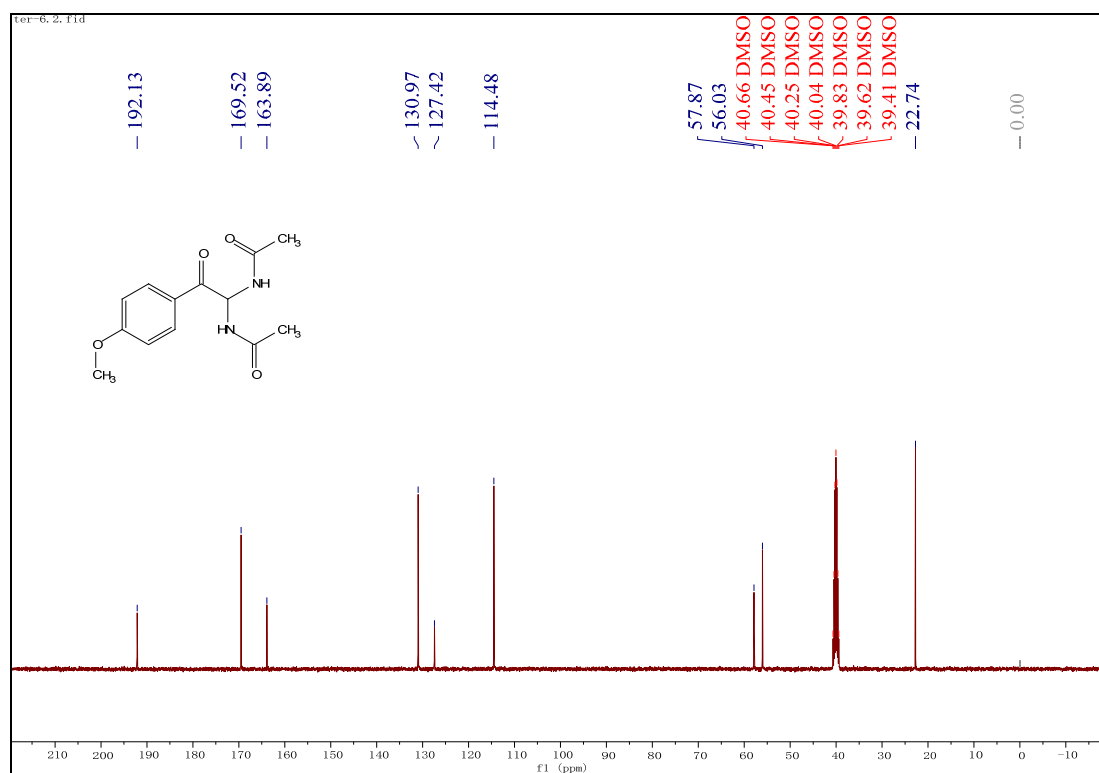
¹H NMR spectrum of **3b** (400 MHz, DMSO-*d*₆)



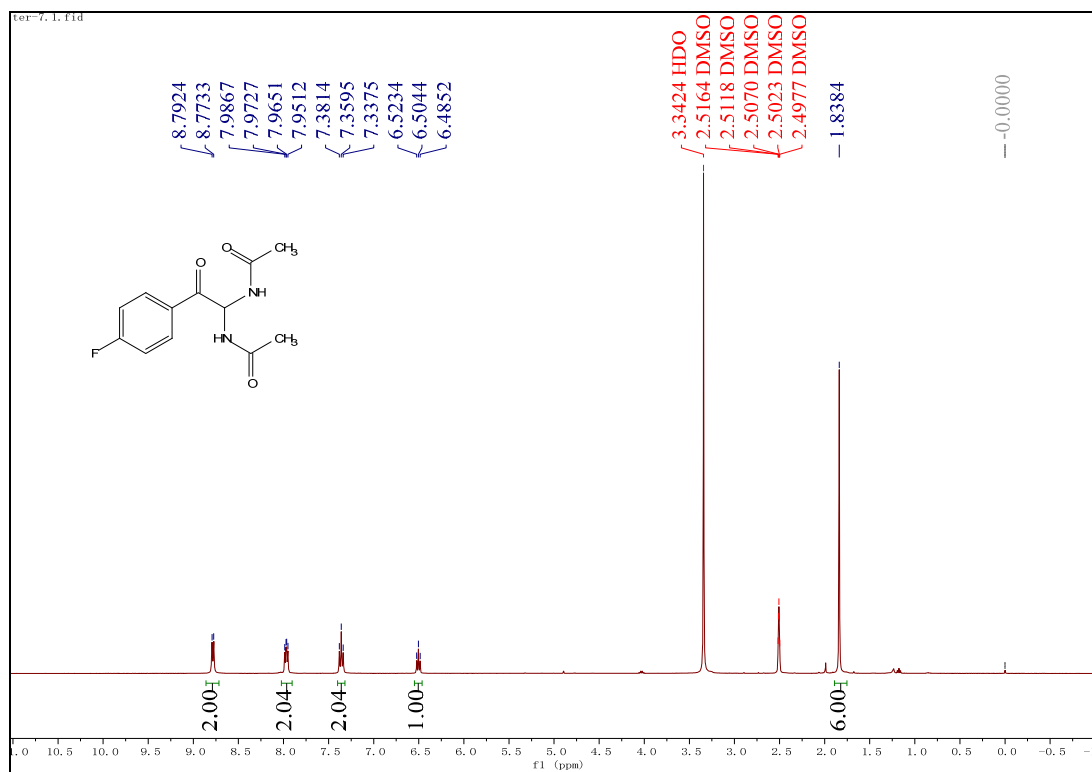
¹³C NMR spectrum of **3b** (100 MHz, DMSO-*d*₆)



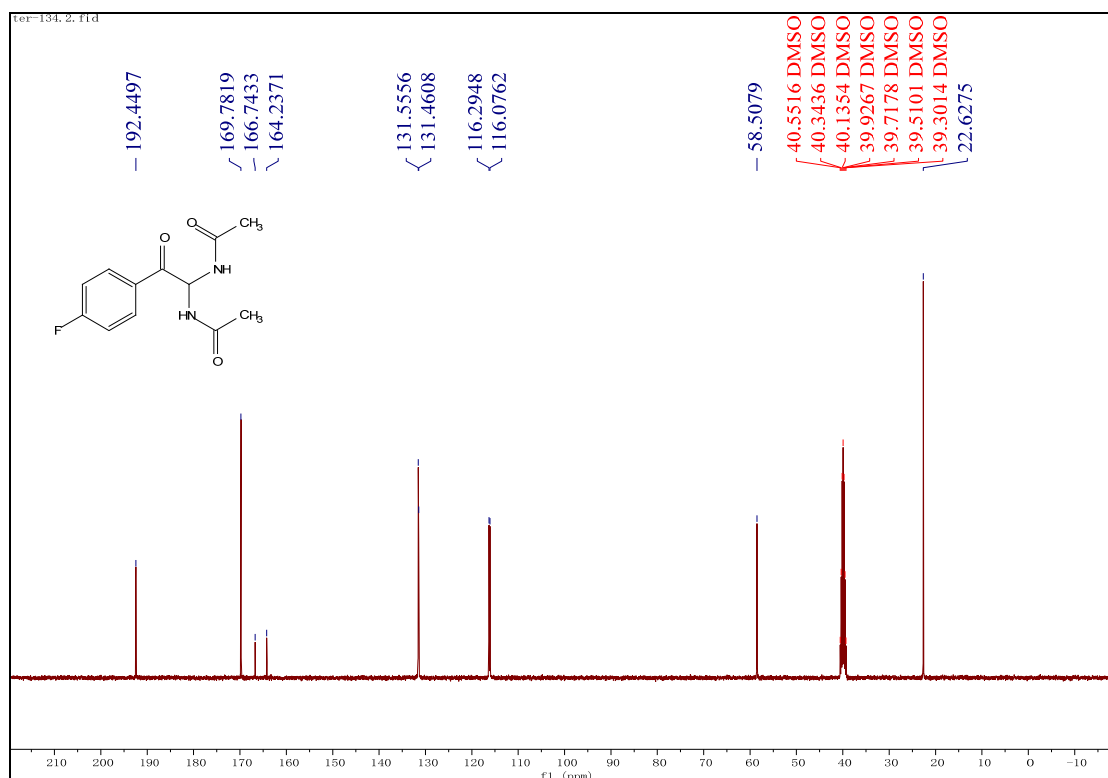
¹H NMR spectrum of **3c** (400 MHz, DMSO-*d*₆)



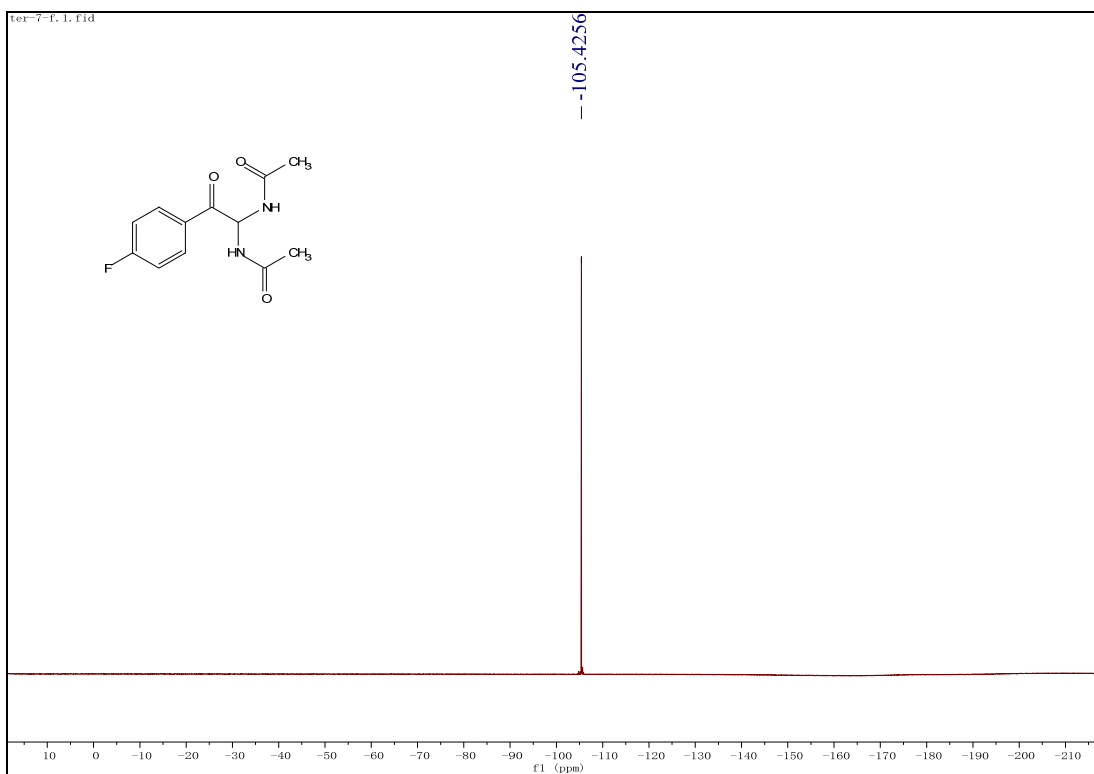
¹³C NMR spectrum of **3c** (100 MHz, DMSO-*d*₆)



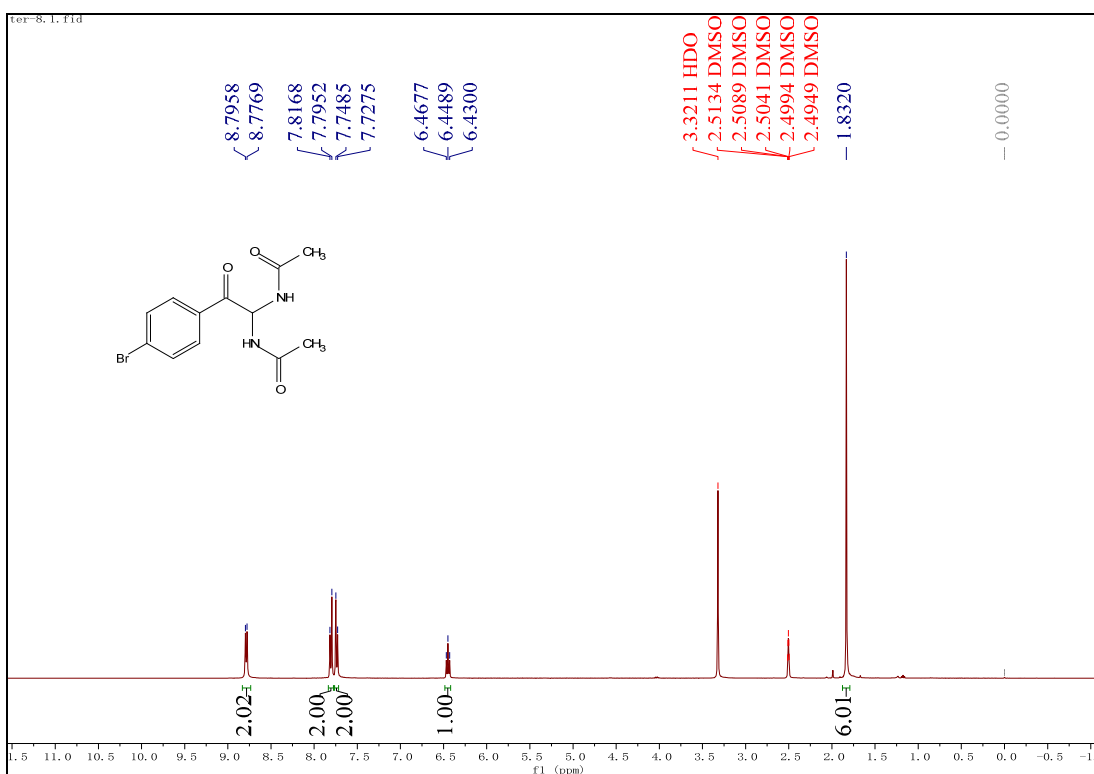
¹H NMR spectrum of **3d** (400 MHz, DMSO-*d*₆)



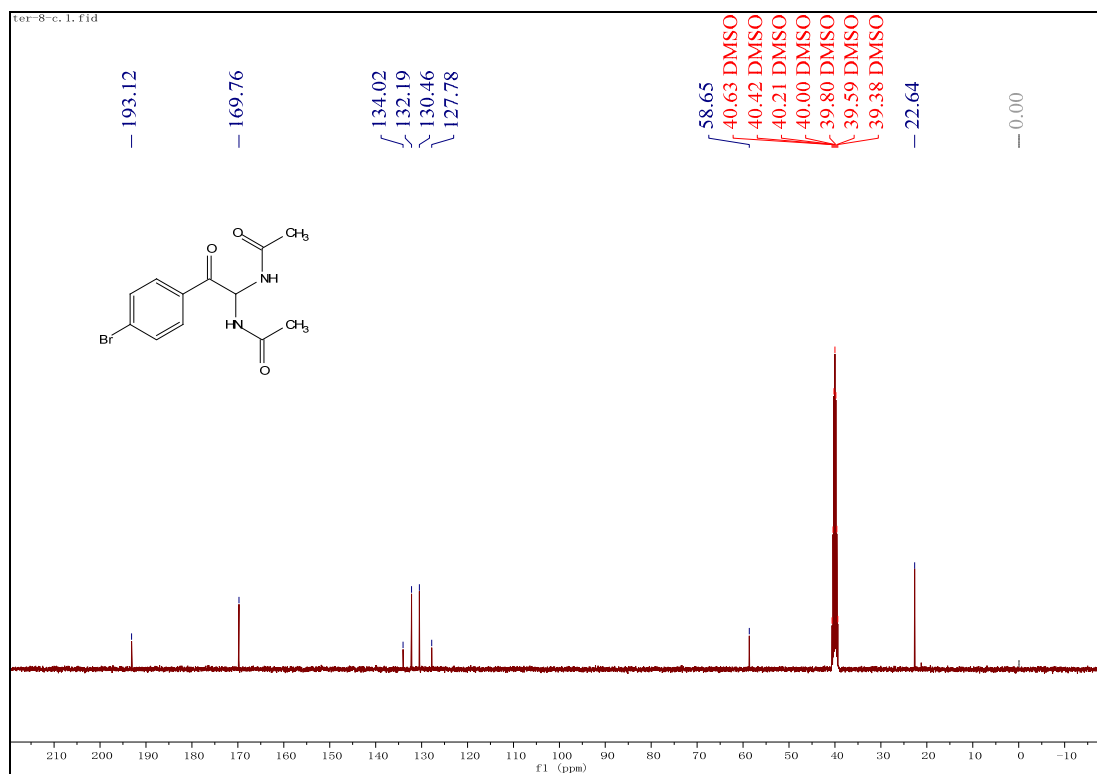
¹³C NMR spectrum of **3d** (100 MHz, DMSO-*d*₆)



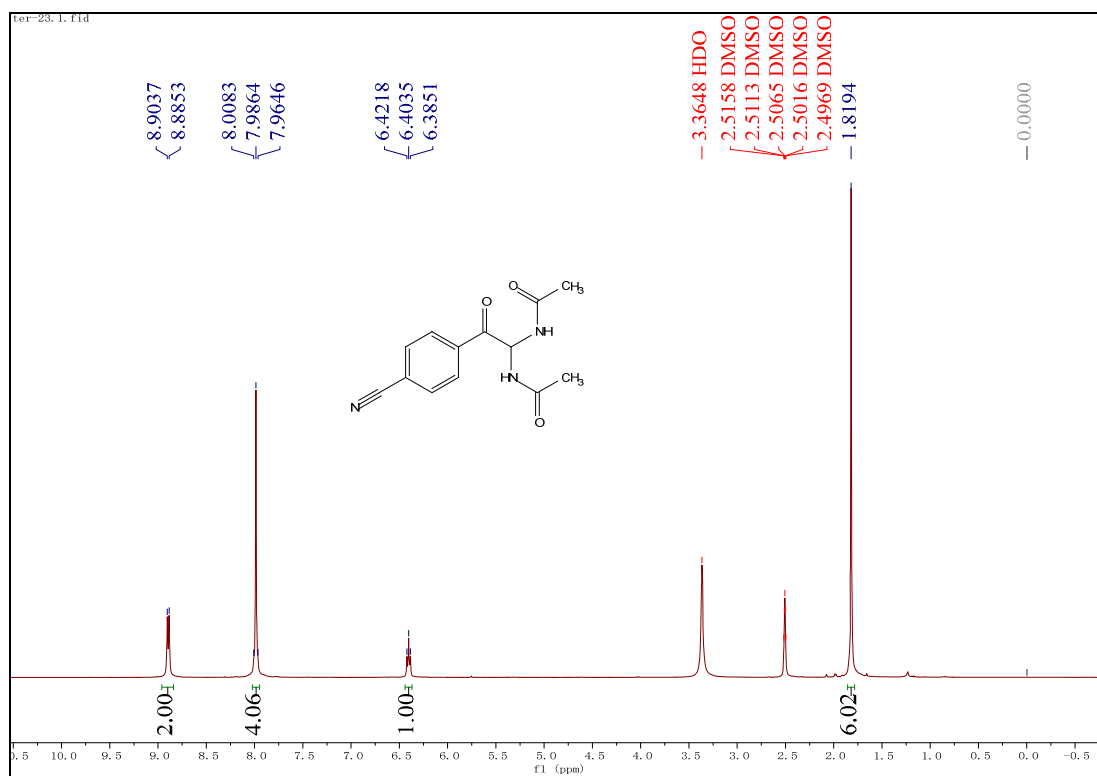
^{19}F NMR spectrum of **3d** (376 MHz, $\text{DMSO-}d_6$)



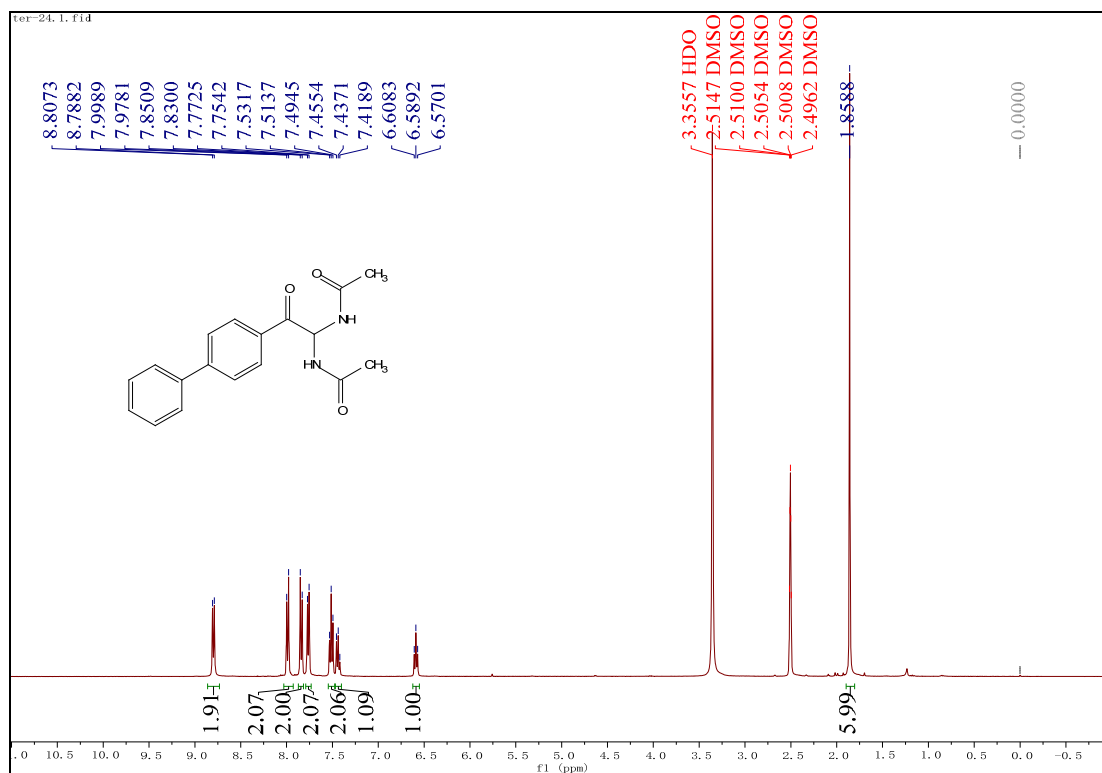
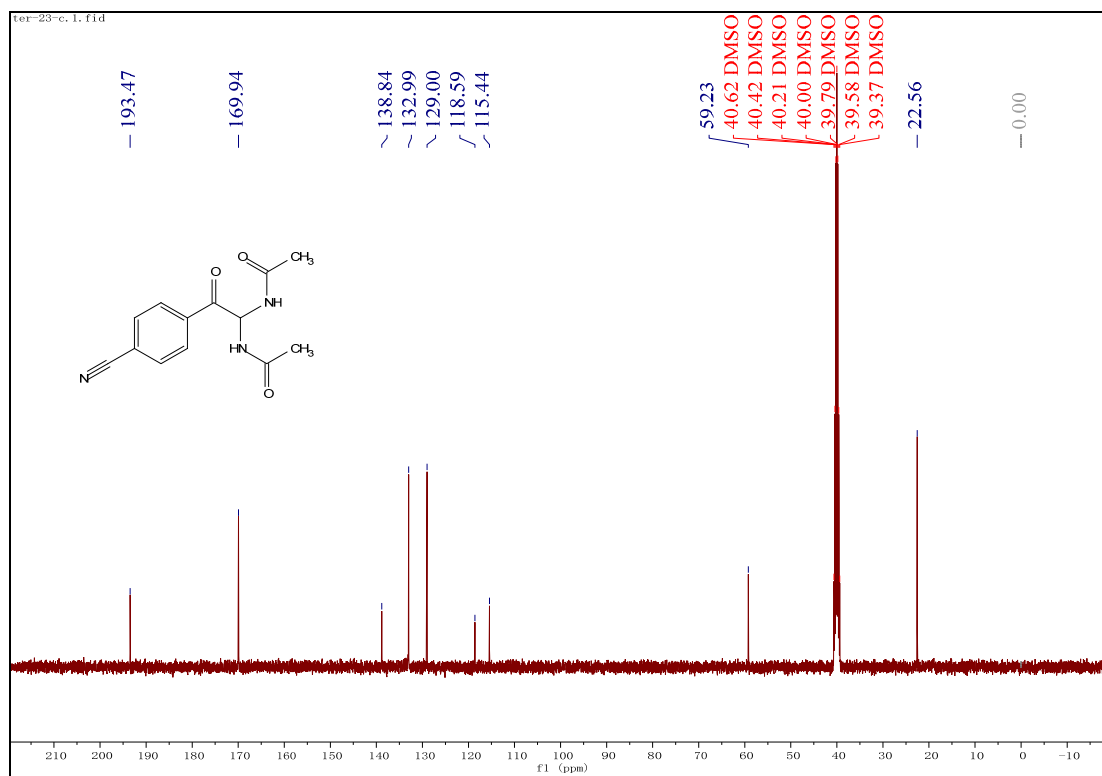
^1H NMR spectrum of **3e** (400 MHz, $\text{DMSO-}d_6$)

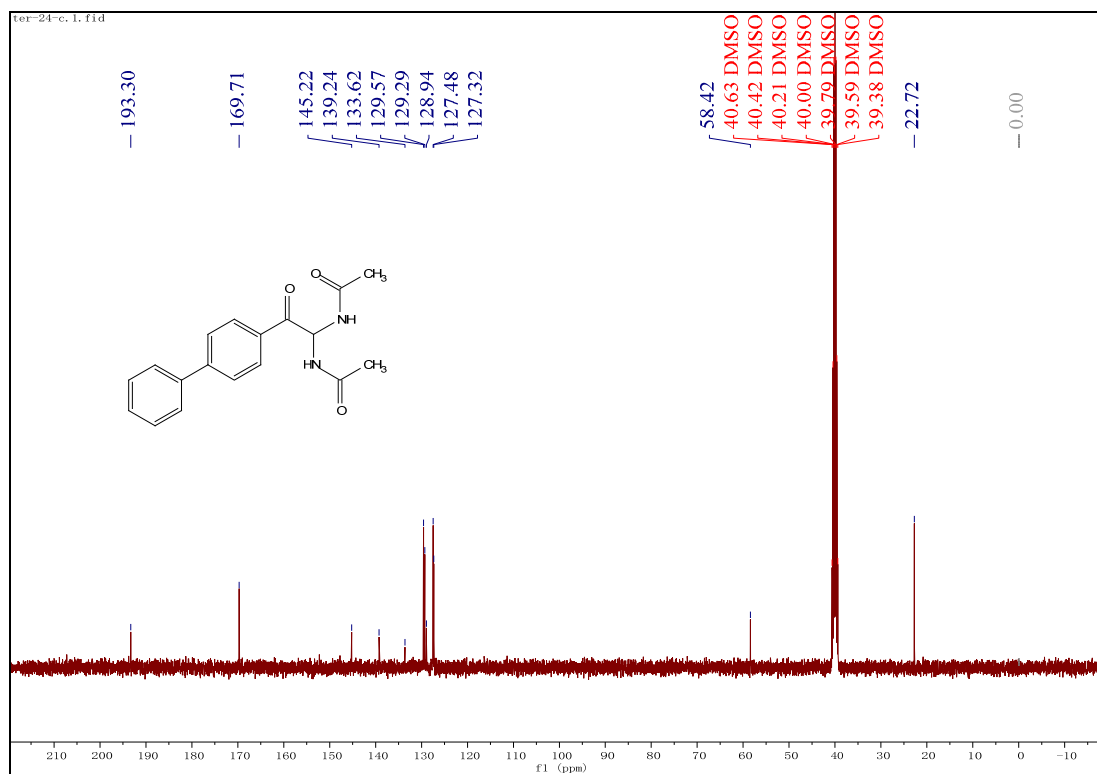


¹³C NMR spectrum of **3e** (100 MHz, DMSO-*d*₆)

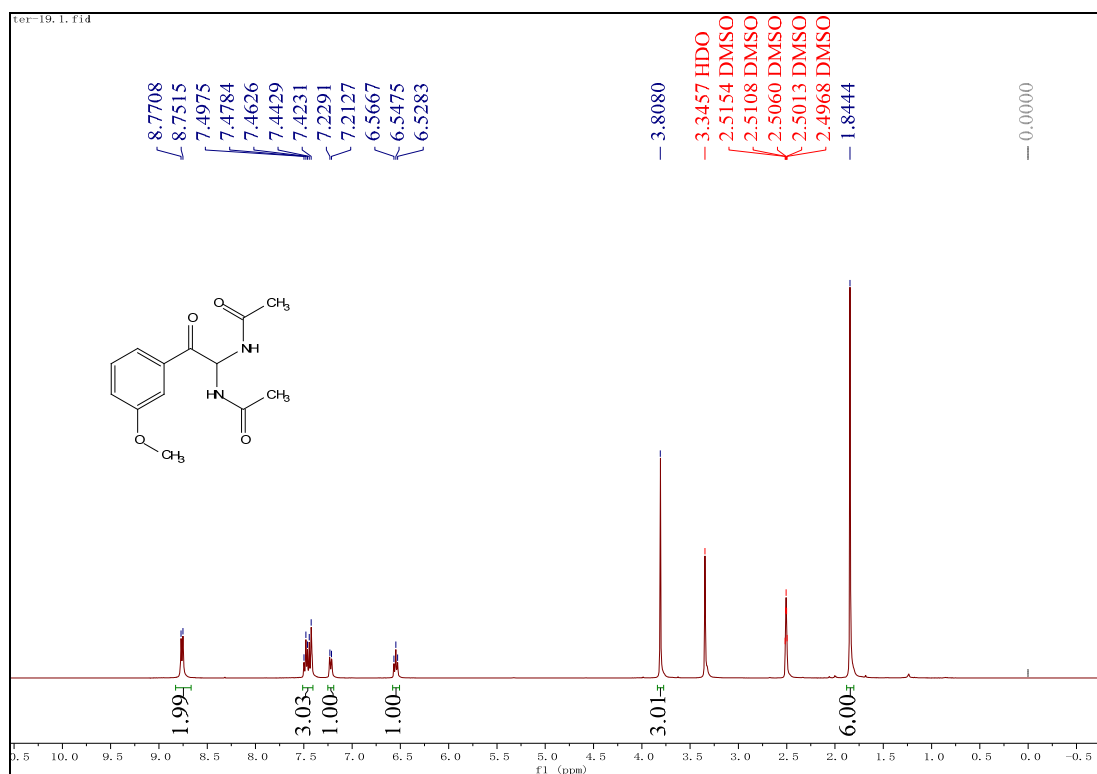


¹H NMR spectrum of **3f** (400 MHz, DMSO-*d*₆)

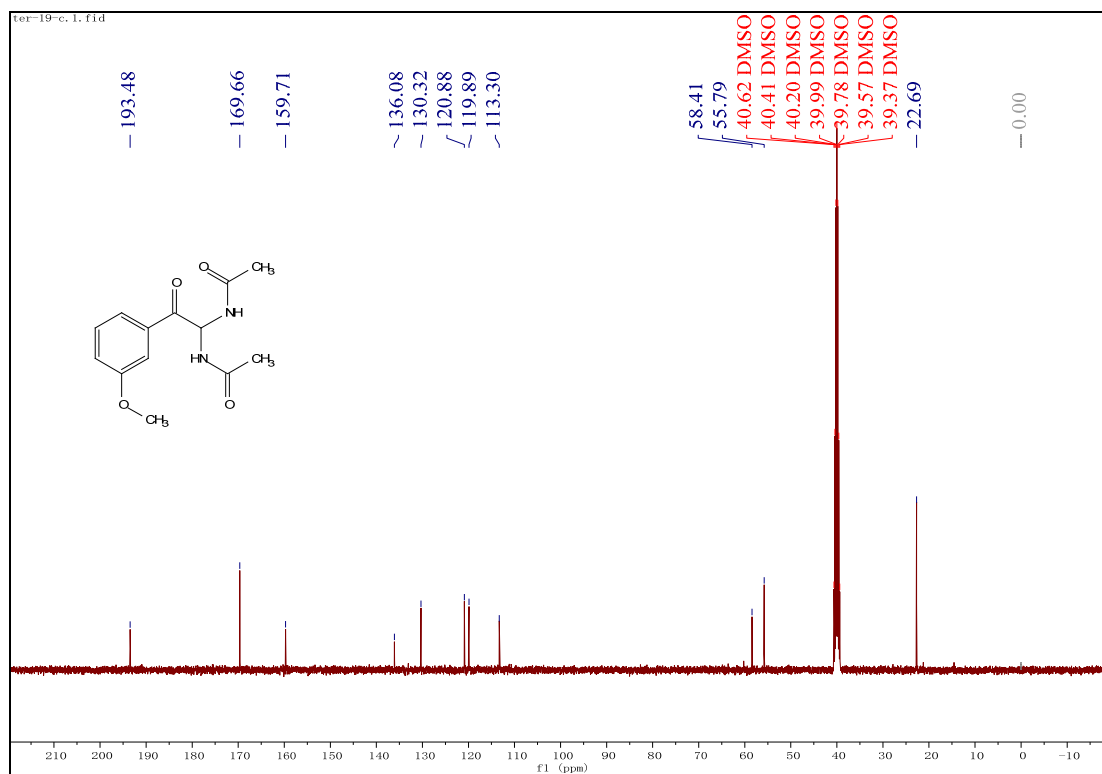




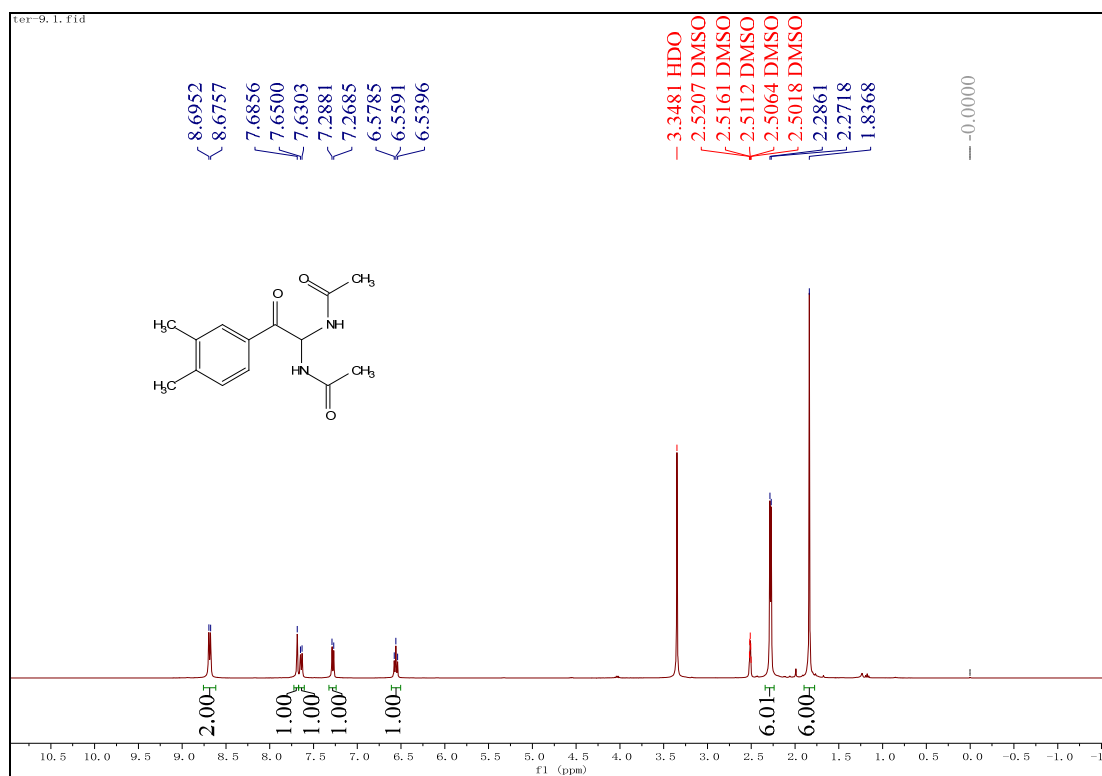
¹³C NMR spectrum of **3g** (100 MHz, DMSO-*d*₆)



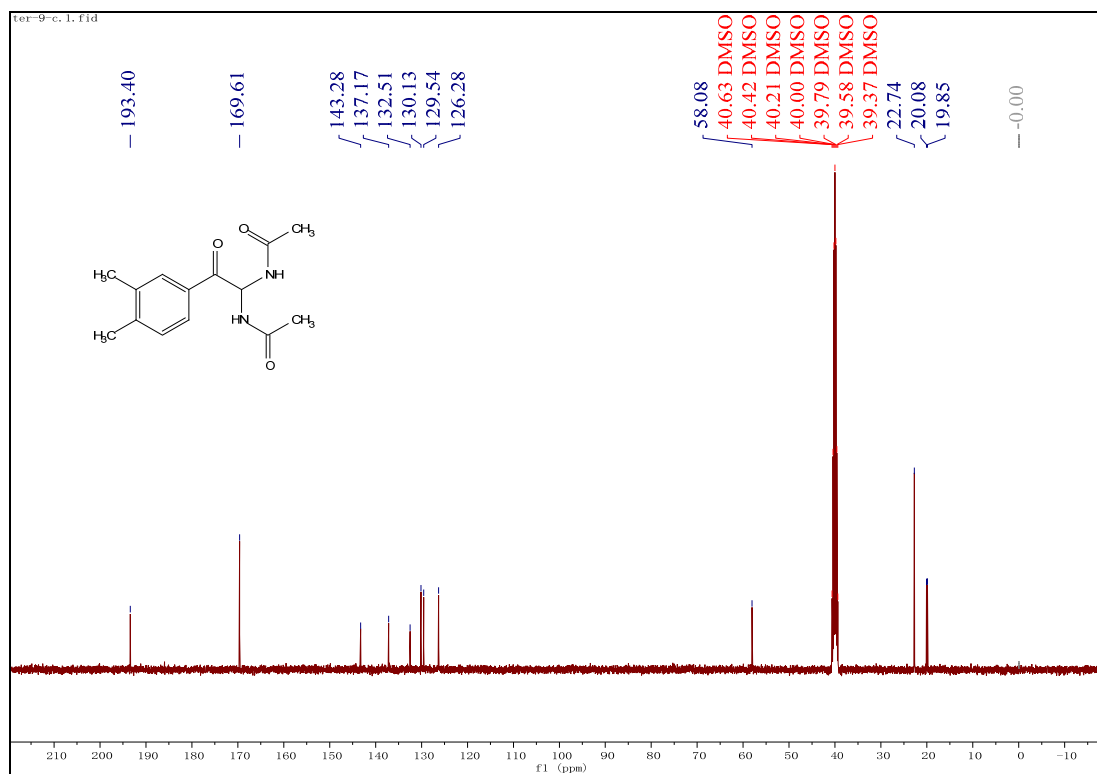
¹H NMR spectrum of **3h** (400 MHz, DMSO-*d*₆)



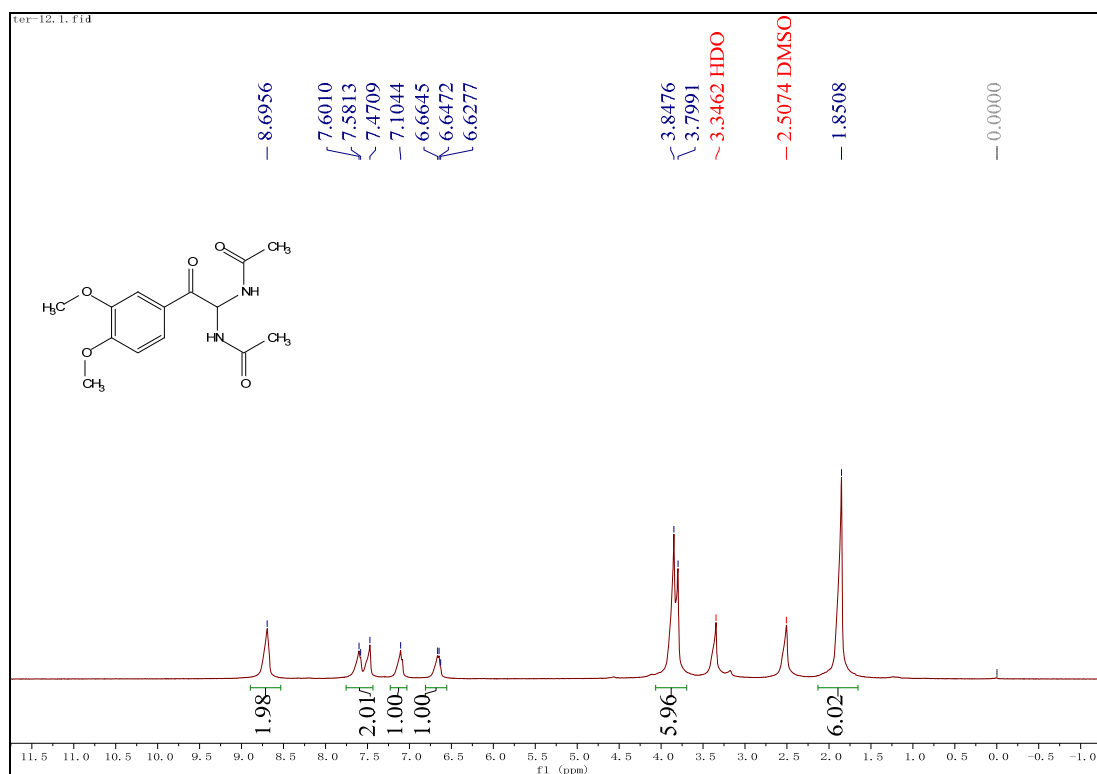
^{13}C NMR spectrum of **3h** (100 MHz, $\text{DMSO-}d_6$)



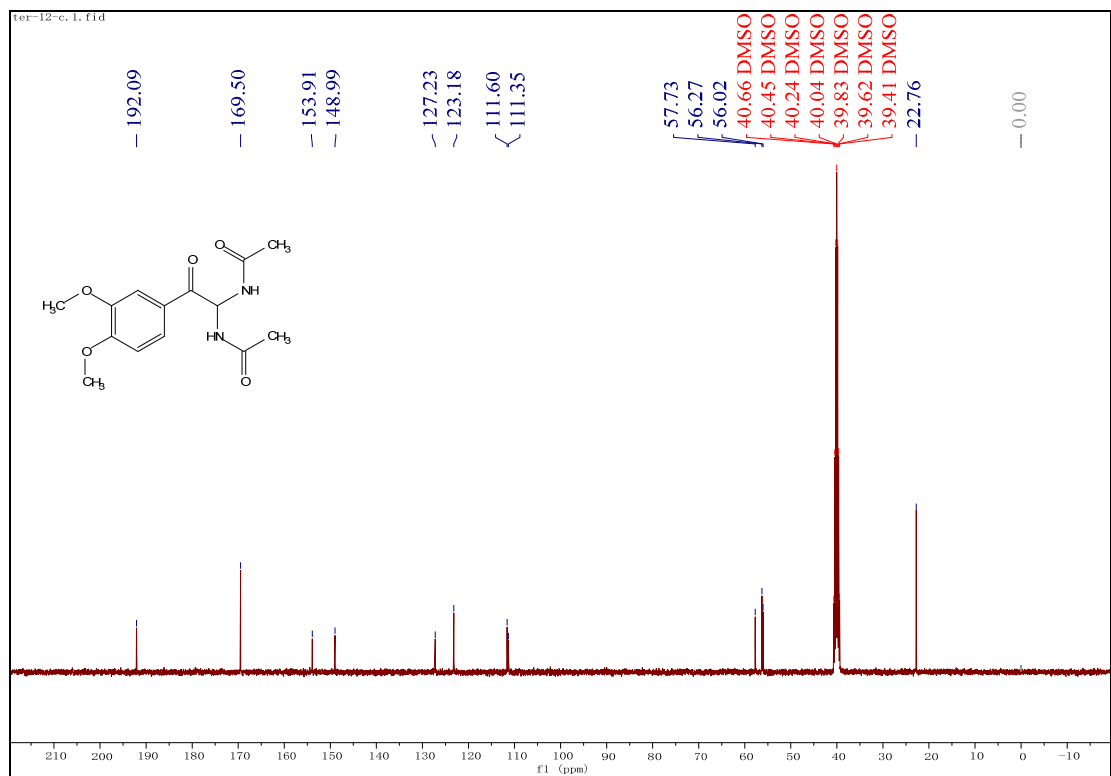
^1H NMR spectrum of **3i** (400 MHz, $\text{DMSO-}d_6$)



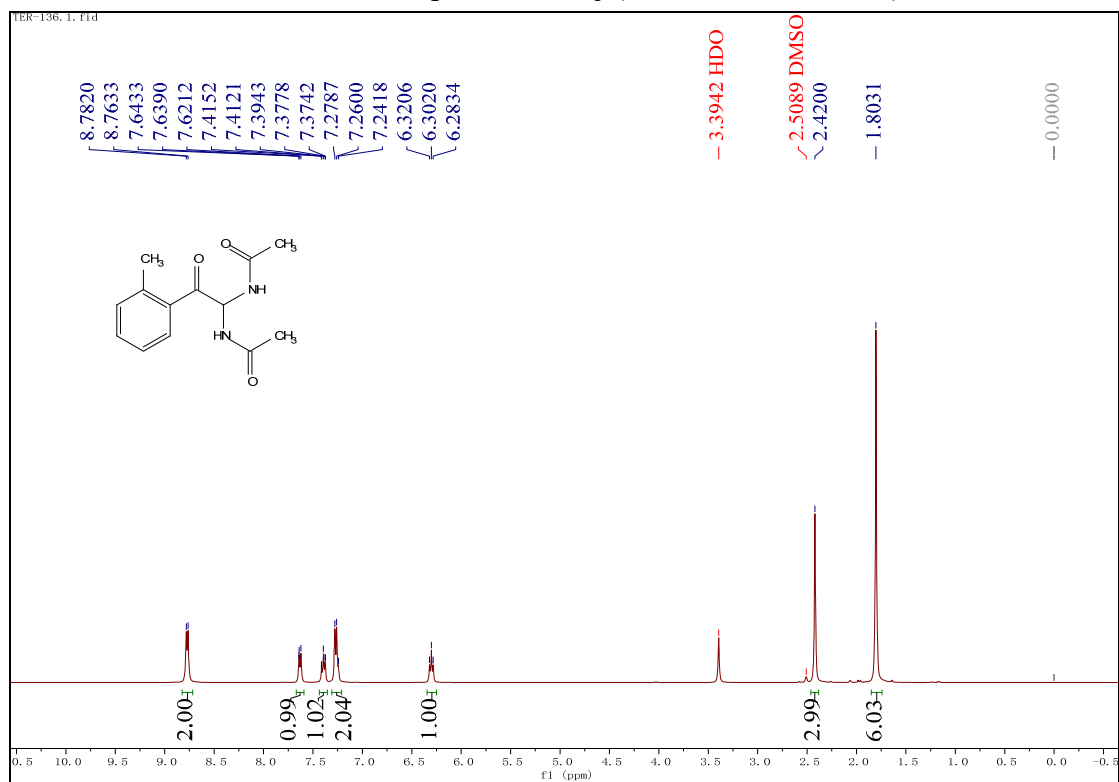
^{13}C NMR spectrum of **3i** (100 MHz, DMSO- d_6)



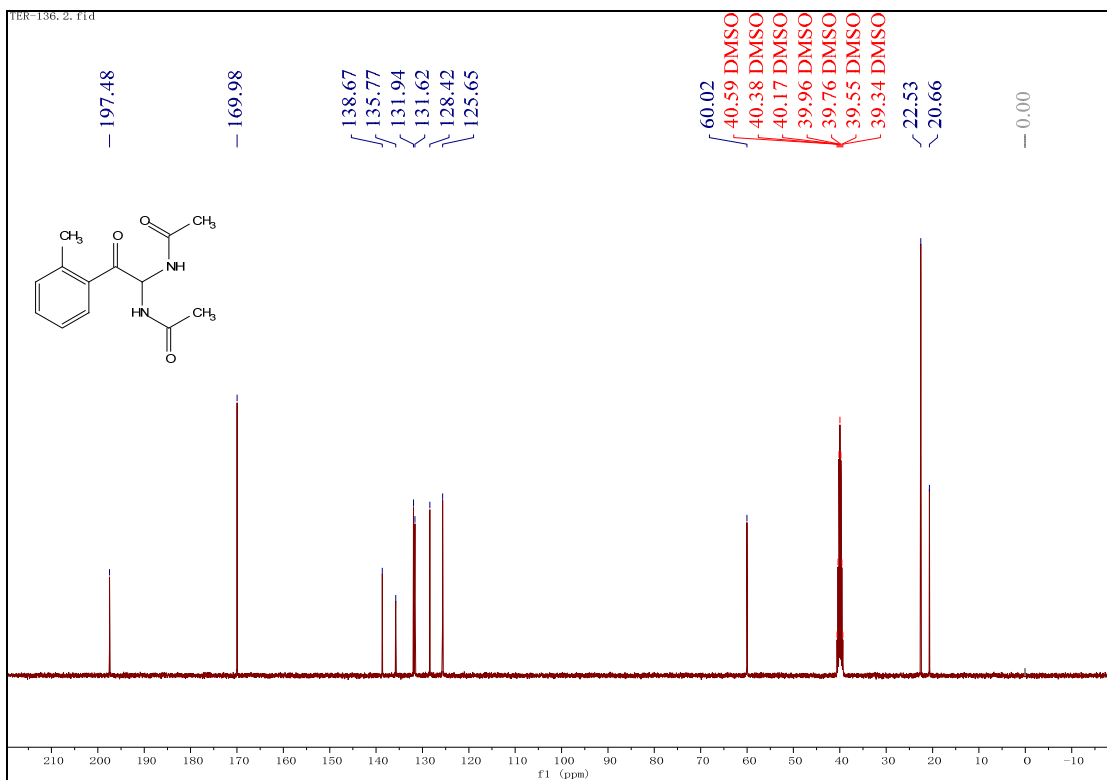
^1H NMR spectrum of **3j** (400 MHz, DMSO- d_6)



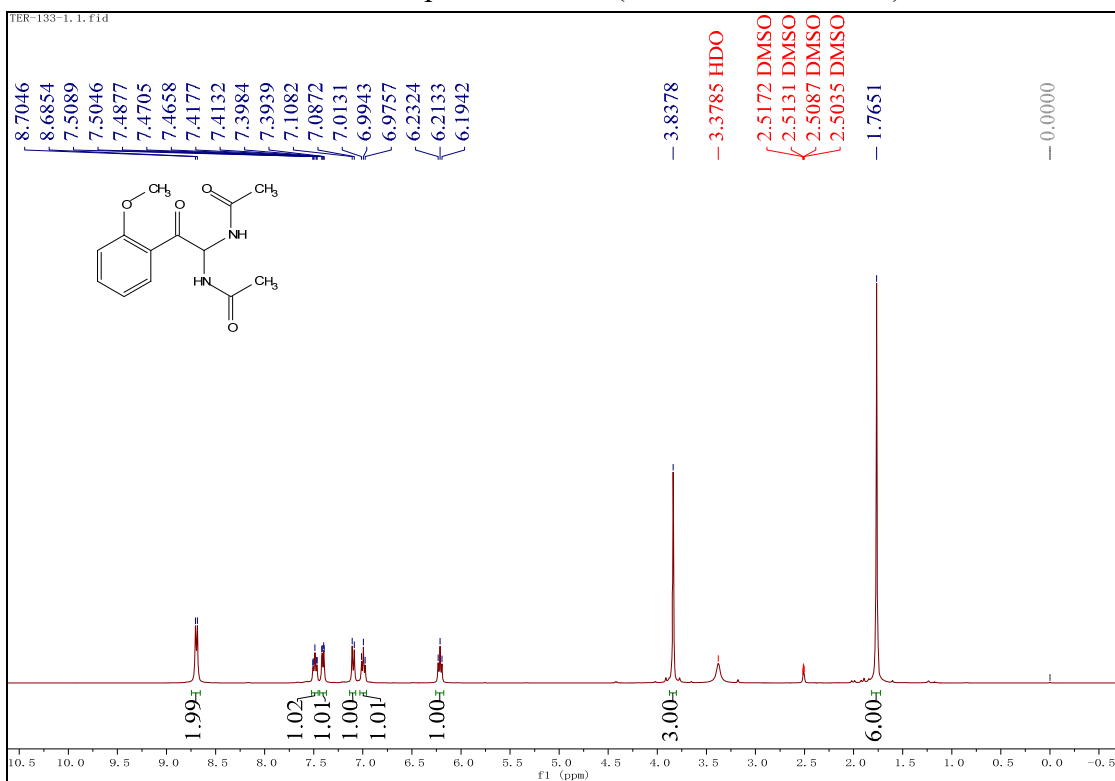
¹³C NMR spectrum of **3j** (100 MHz, DMSO-*d*₆)



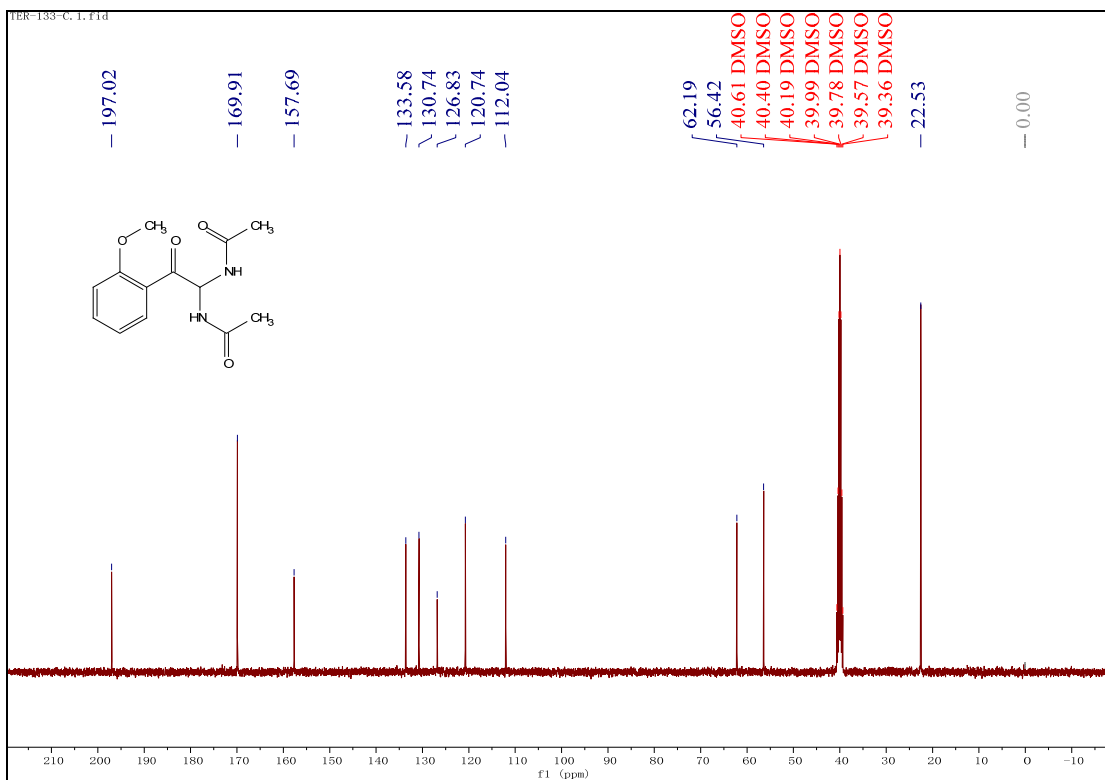
¹H NMR spectrum of **3k** (400 MHz, DMSO-*d*₆)



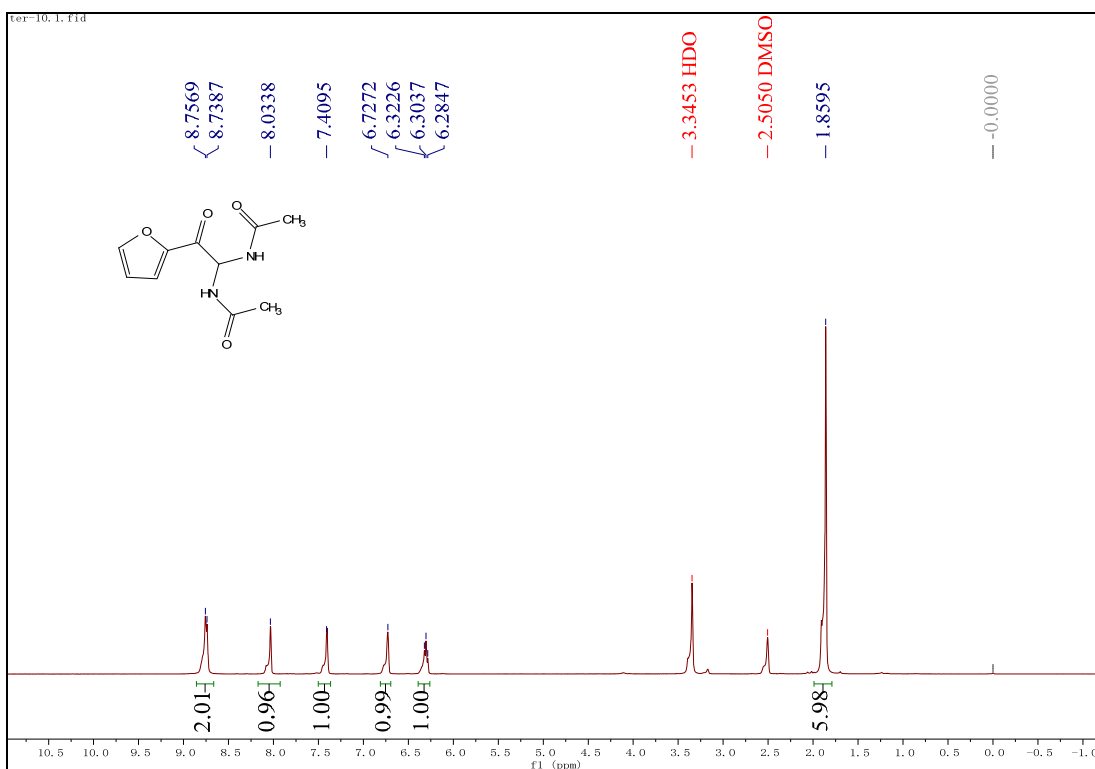
^{13}C NMR spectrum of **3k** (100 MHz, DMSO- d_6)



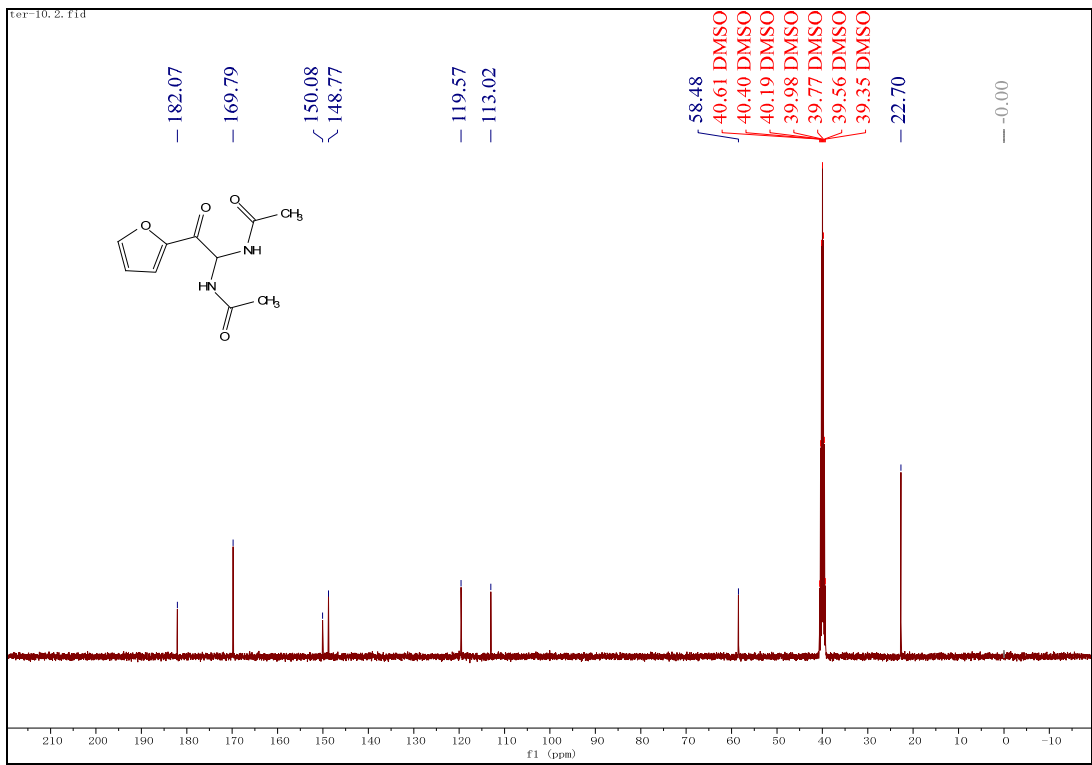
^1H NMR spectrum of **3l** (400 MHz, DMSO- d_6)



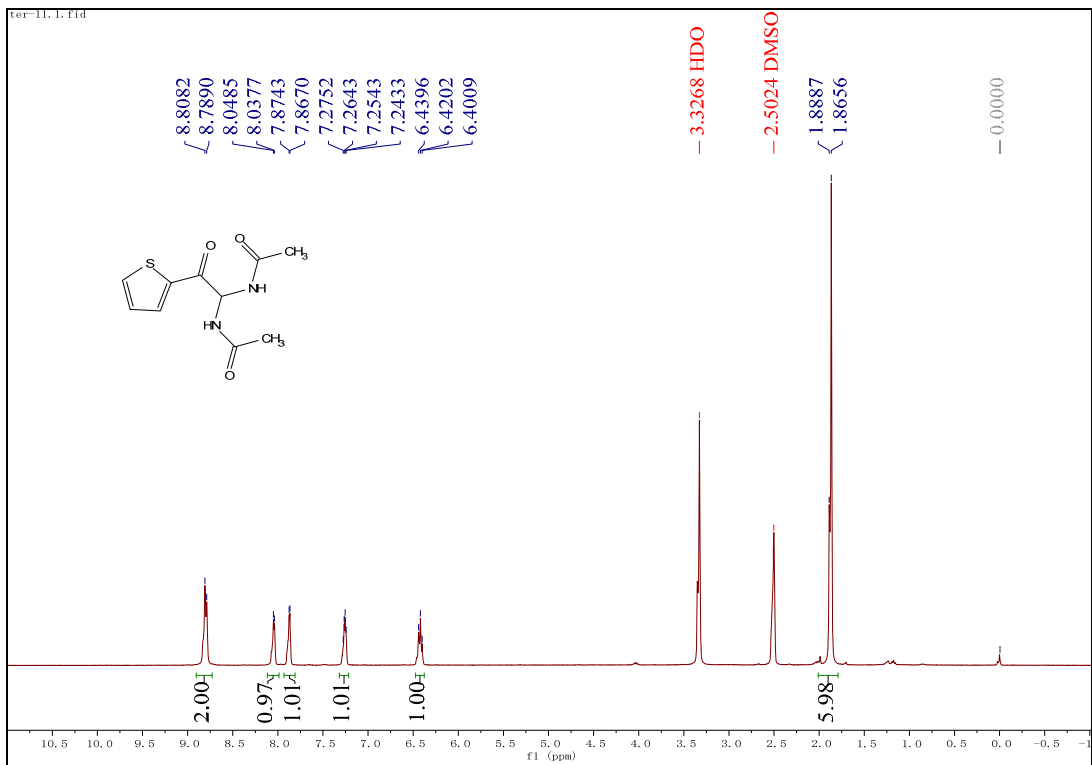
¹³C NMR spectrum of **31** (100 MHz, DMSO-*d*₆)



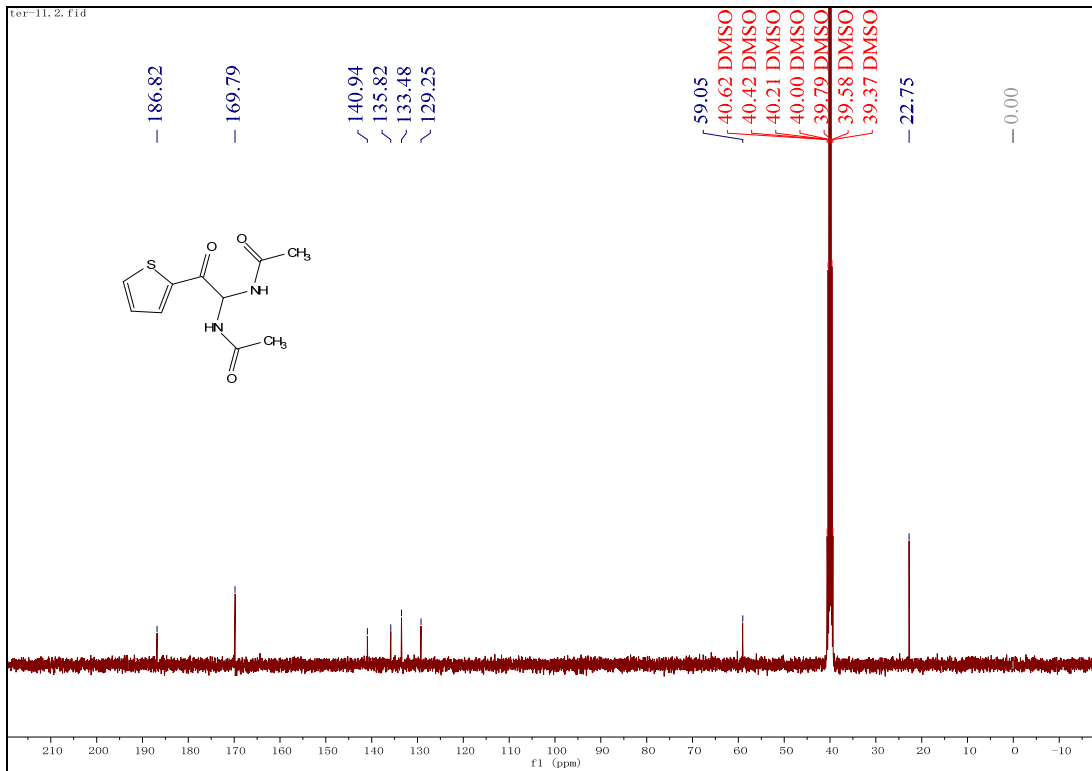
¹H NMR spectrum of **3m** (400 MHz, DMSO-*d*₆)



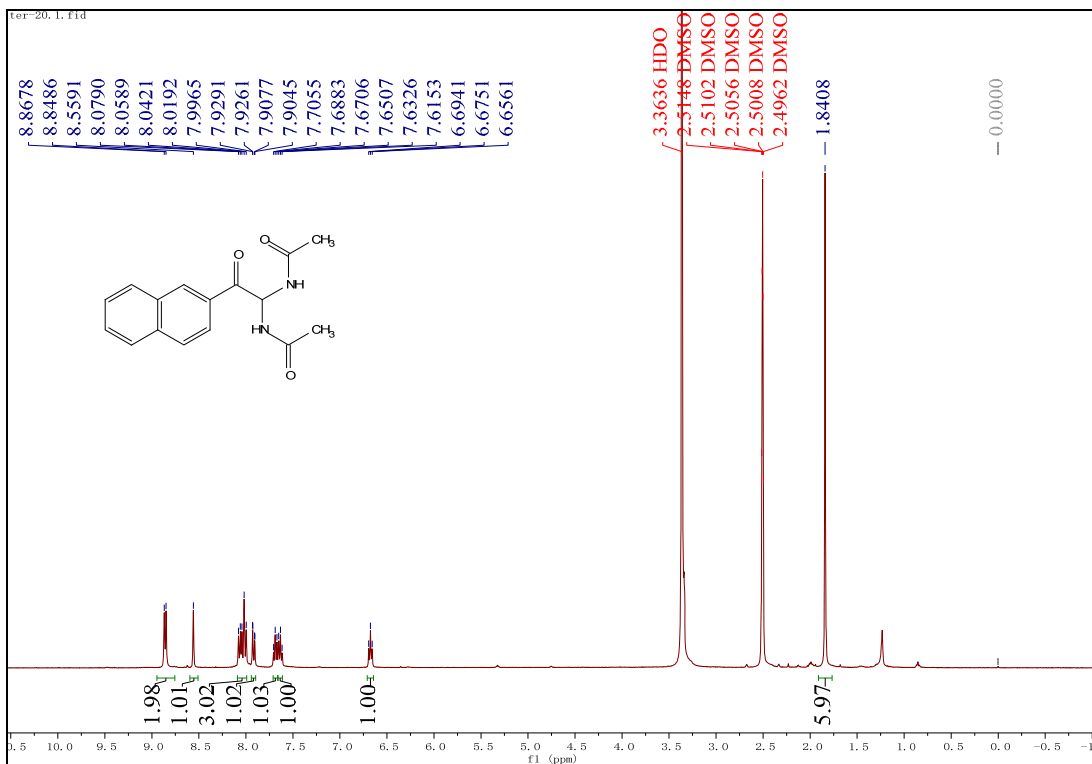
¹³C NMR spectrum of **3m** (100 MHz, DMSO-*d*₆)



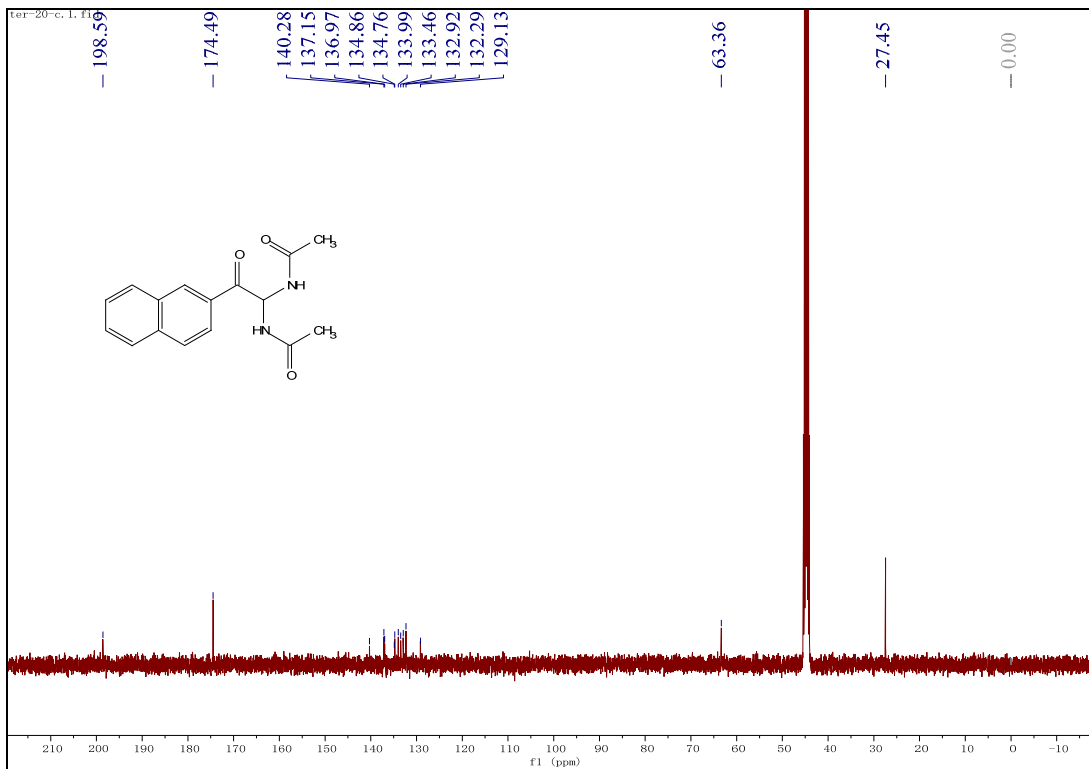
¹H NMR spectrum of **3m** (400 MHz, DMSO-*d*₆)



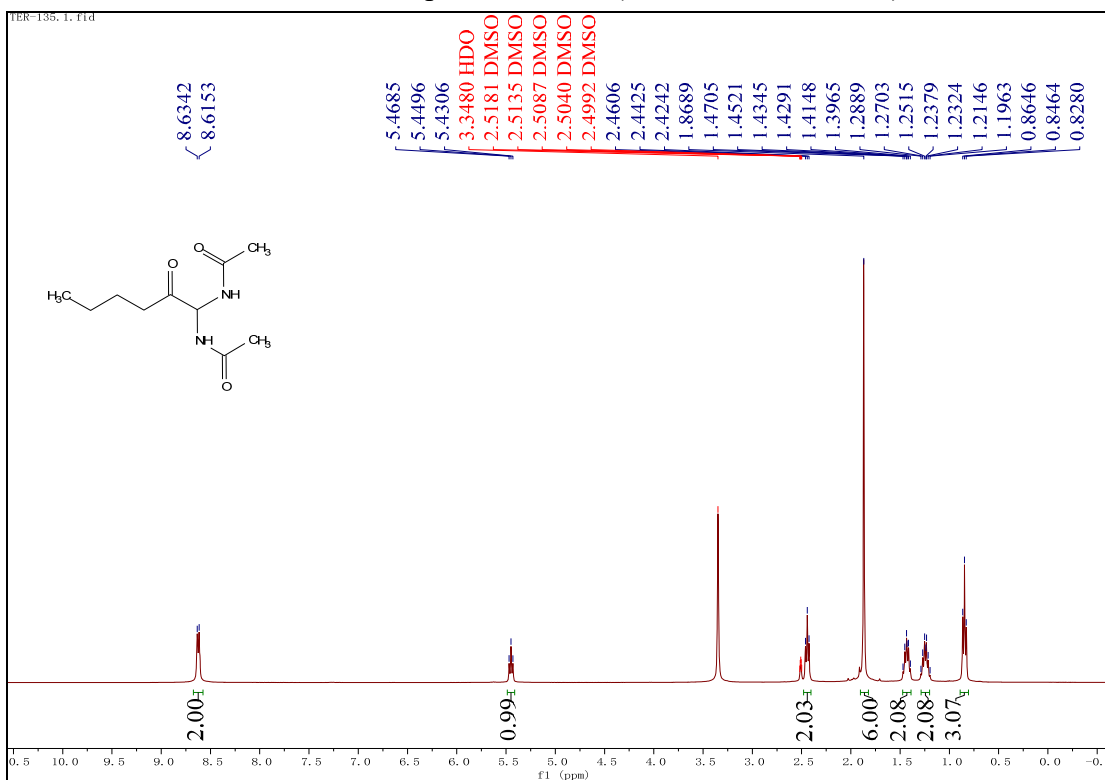
^{13}C NMR spectrum of **3n** (100 MHz, $\text{DMSO-}d_6$)



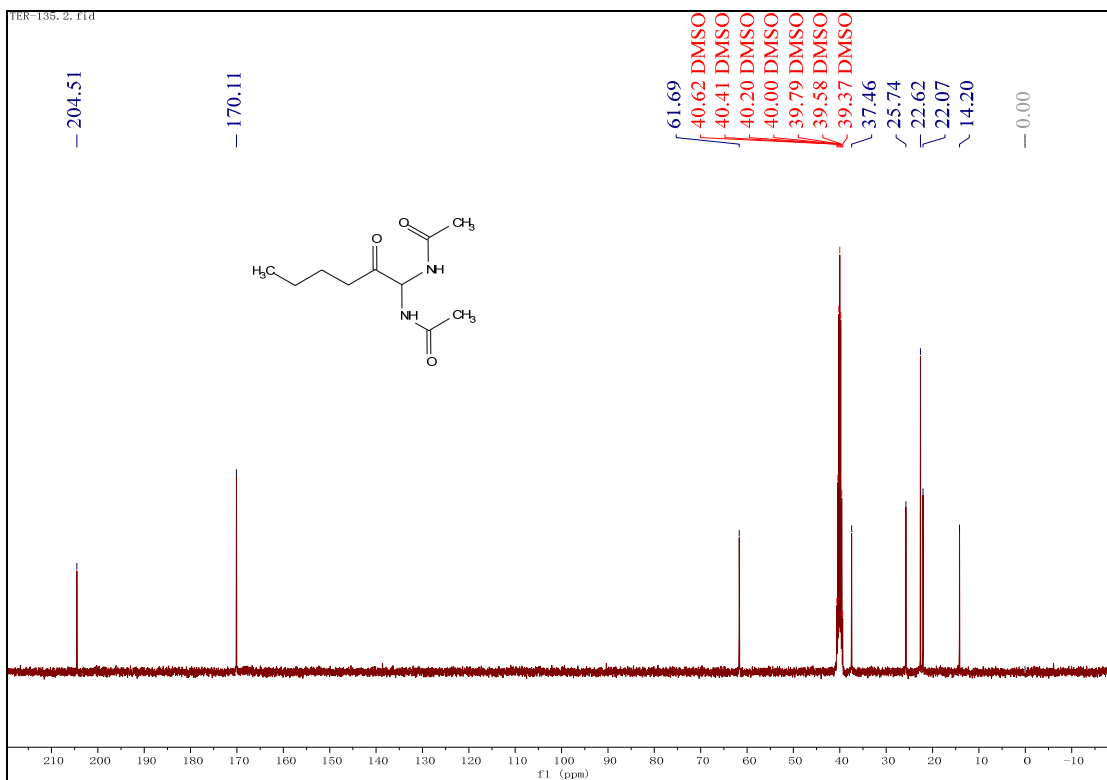
^1H NMR spectrum of **3o** (400 MHz, $\text{DMSO-}d_6$)



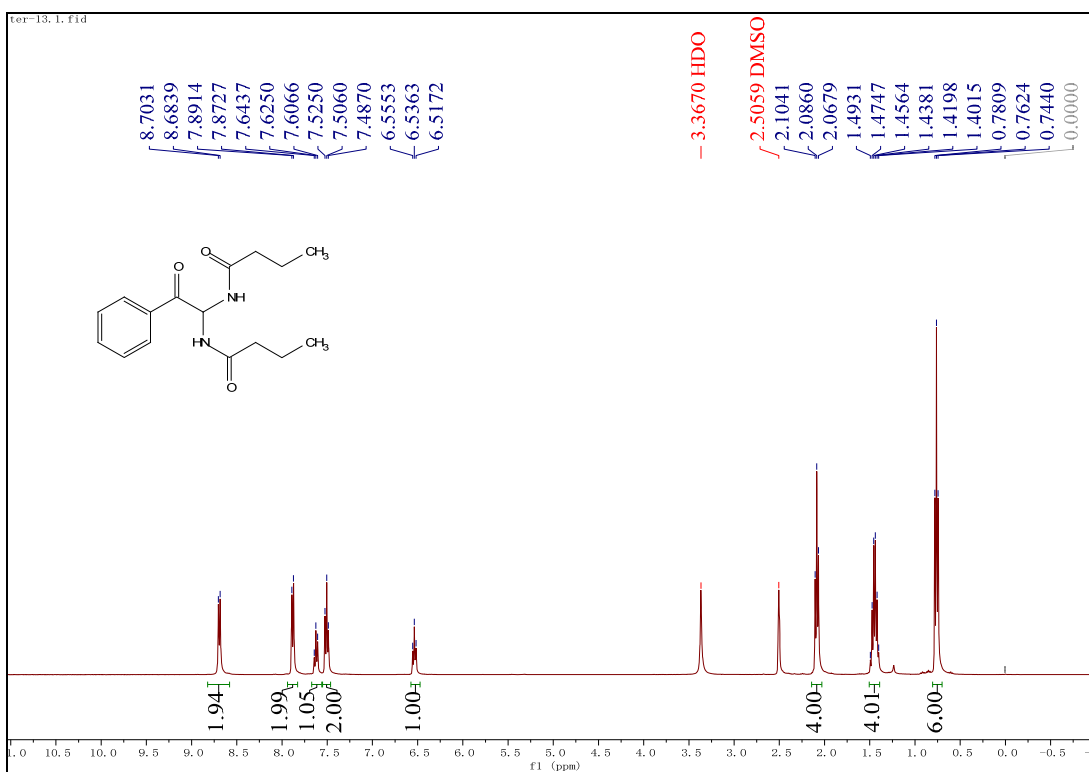
¹³C NMR spectrum of **3o** (100 MHz, DMSO-*d*₆)



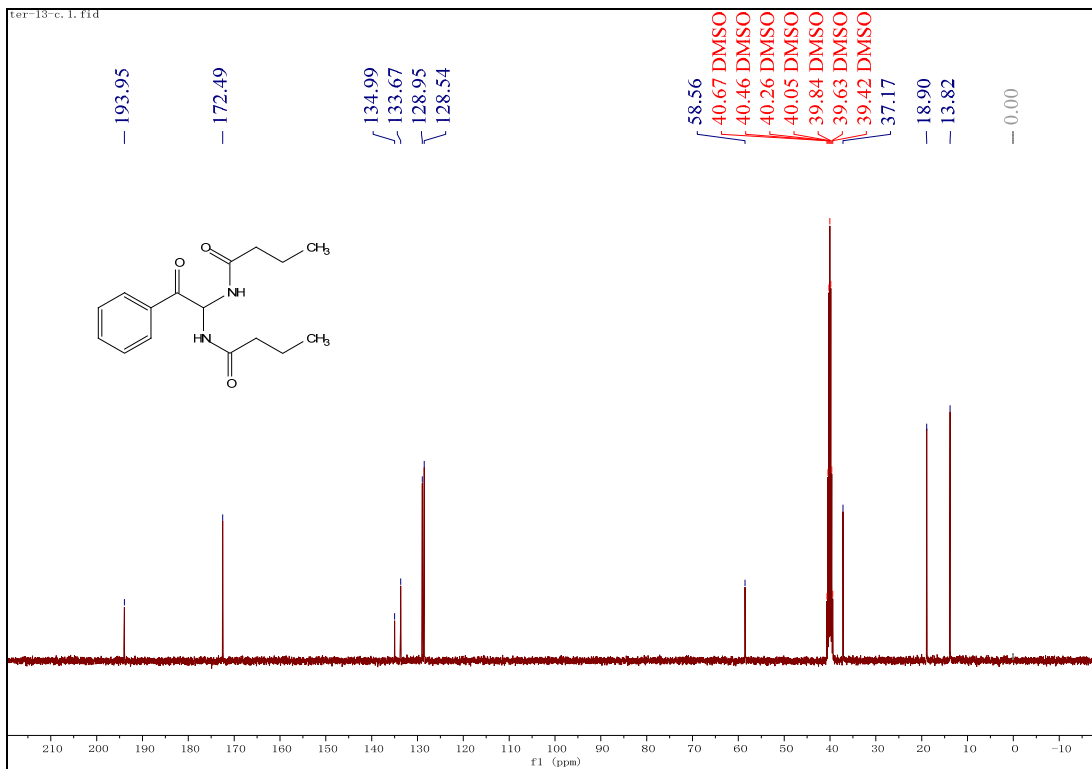
¹H NMR spectrum of **3p** (400 MHz, DMSO-*d*₆)



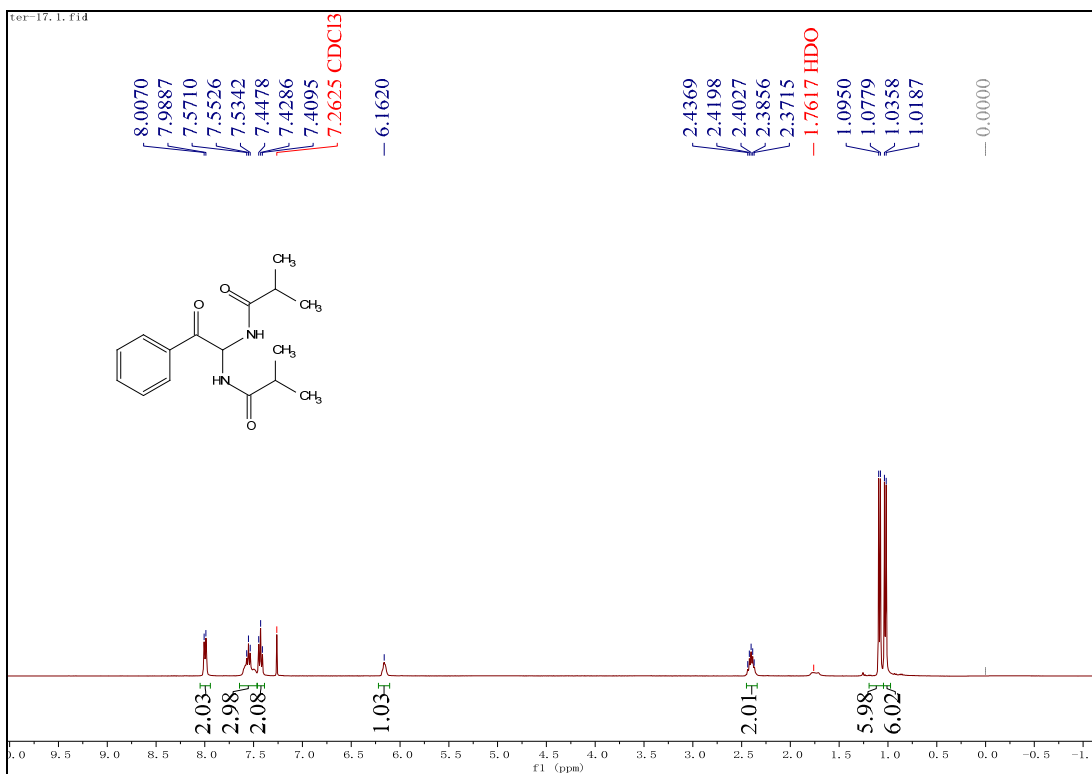
^{13}C NMR spectrum of **3p** (100 MHz, DMSO- d_6)



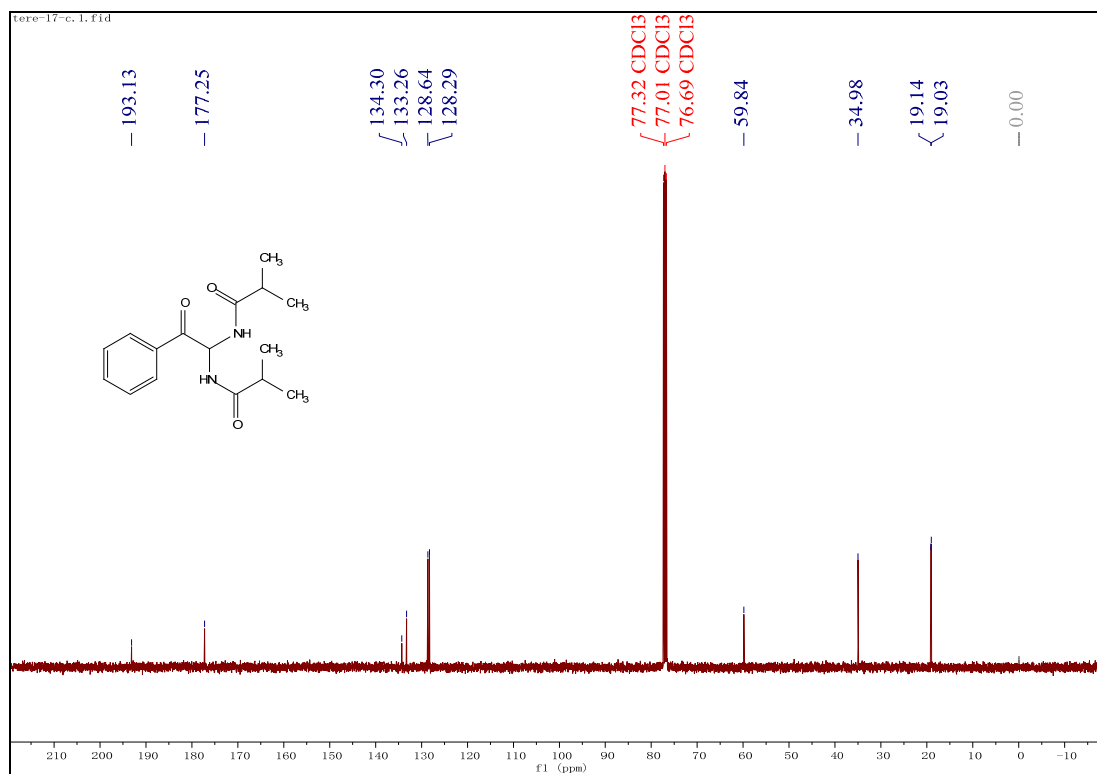
^1H NMR spectrum of **3q** (400 MHz, DMSO- d_6)



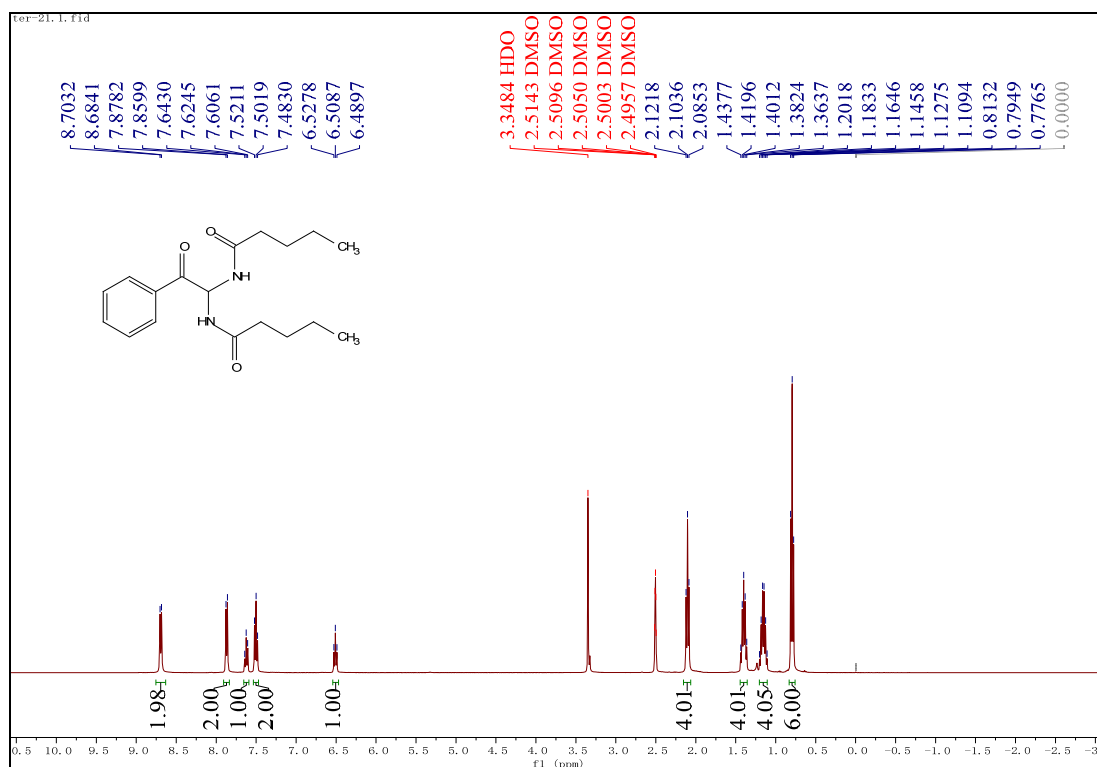
^{13}C NMR spectrum of **3q** (100 MHz, DMSO- d_6)



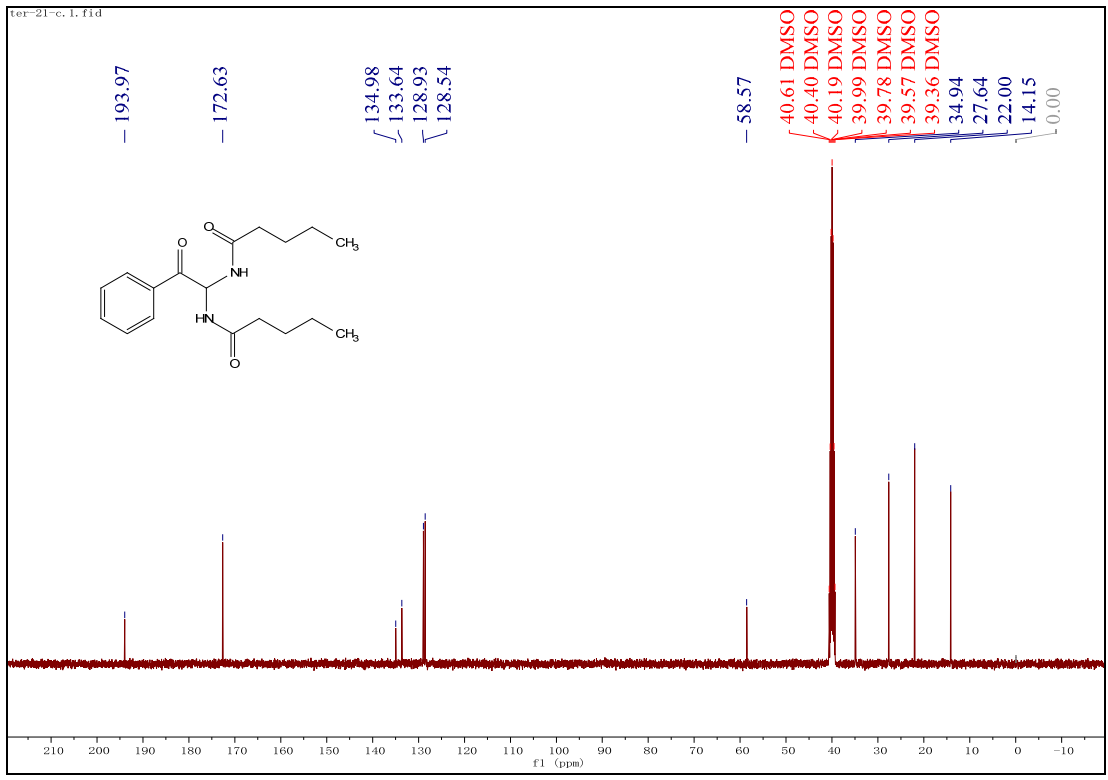
^1H NMR spectrum of **3r** (400 MHz, CDCl_3)



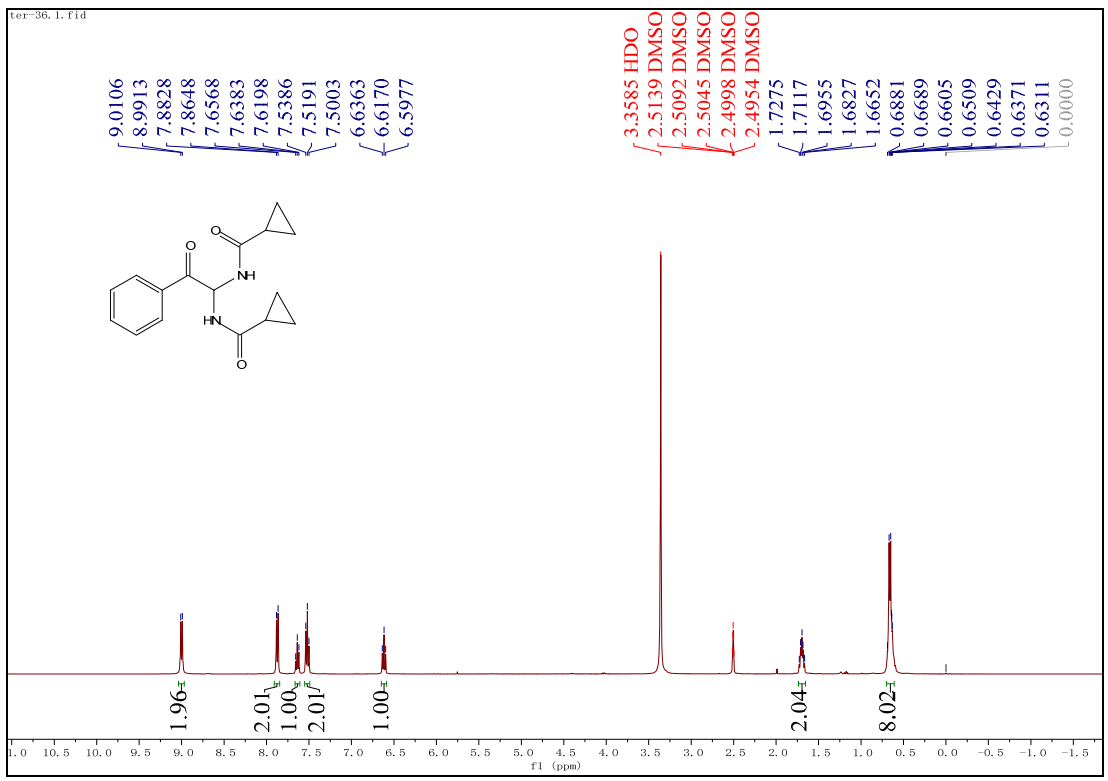
^{13}C NMR spectrum of **3r** (100 MHz, CDCl_3)



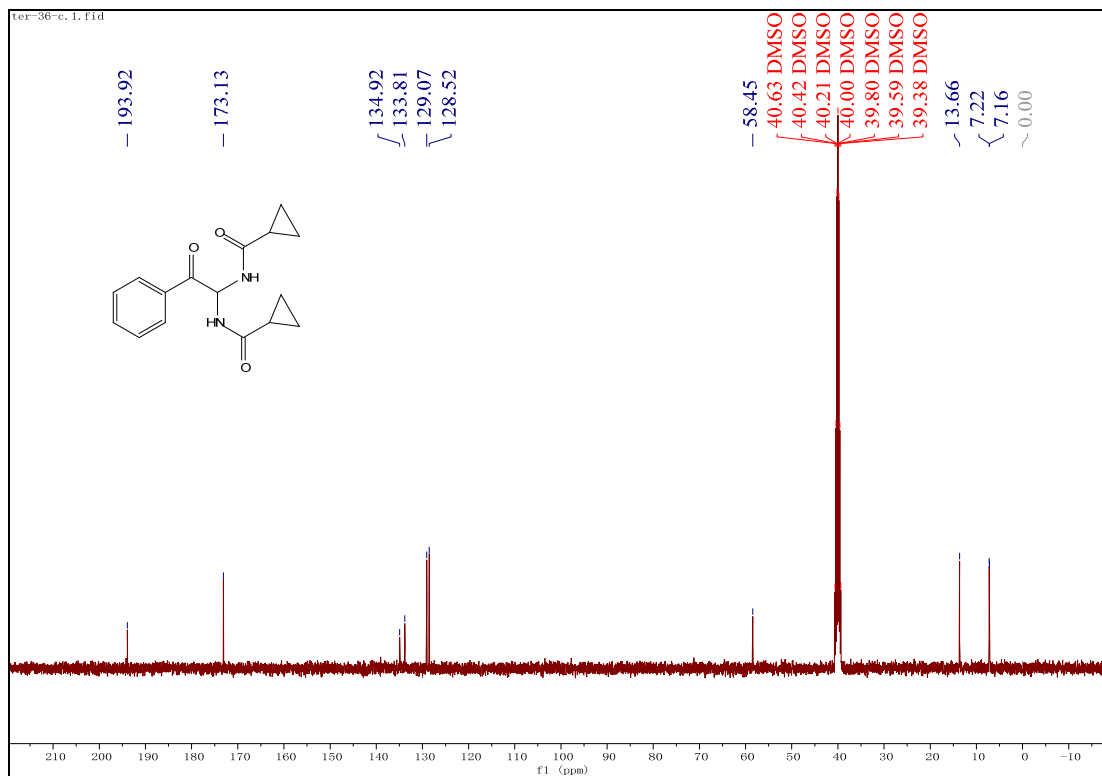
^1H NMR spectrum of **3s** (400 MHz, $\text{DMSO}-d_6$)



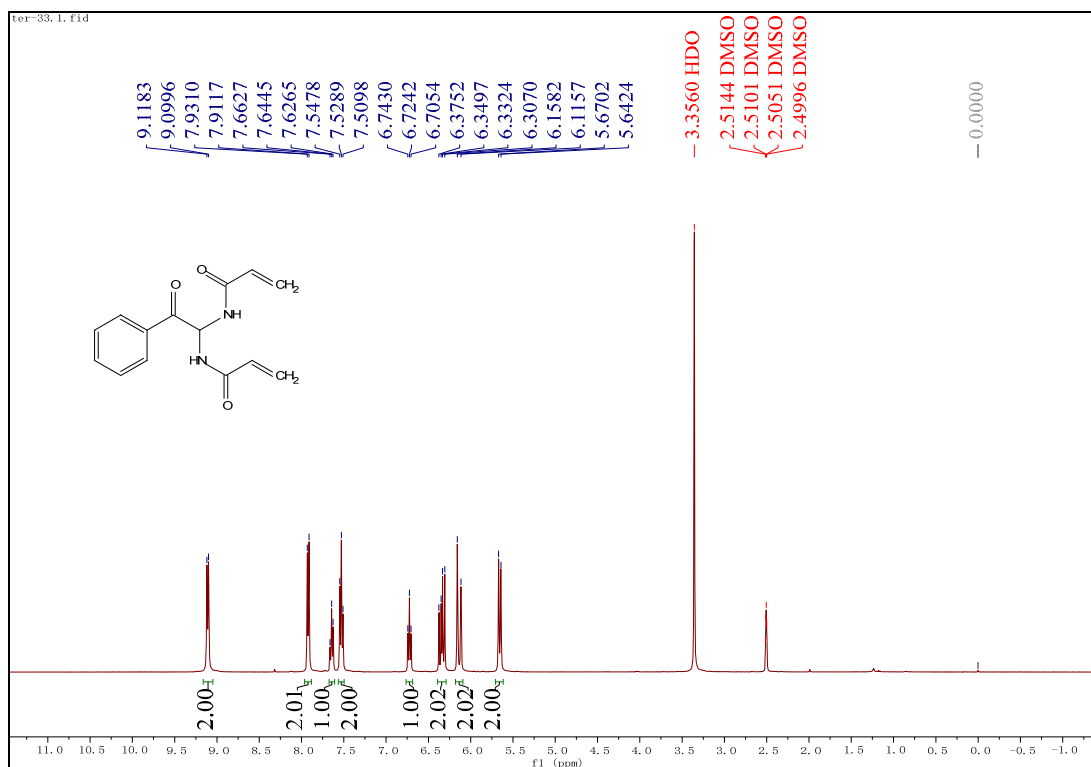
¹³C NMR spectrum of 3s (100 MHz, DMSO-*d*₆)



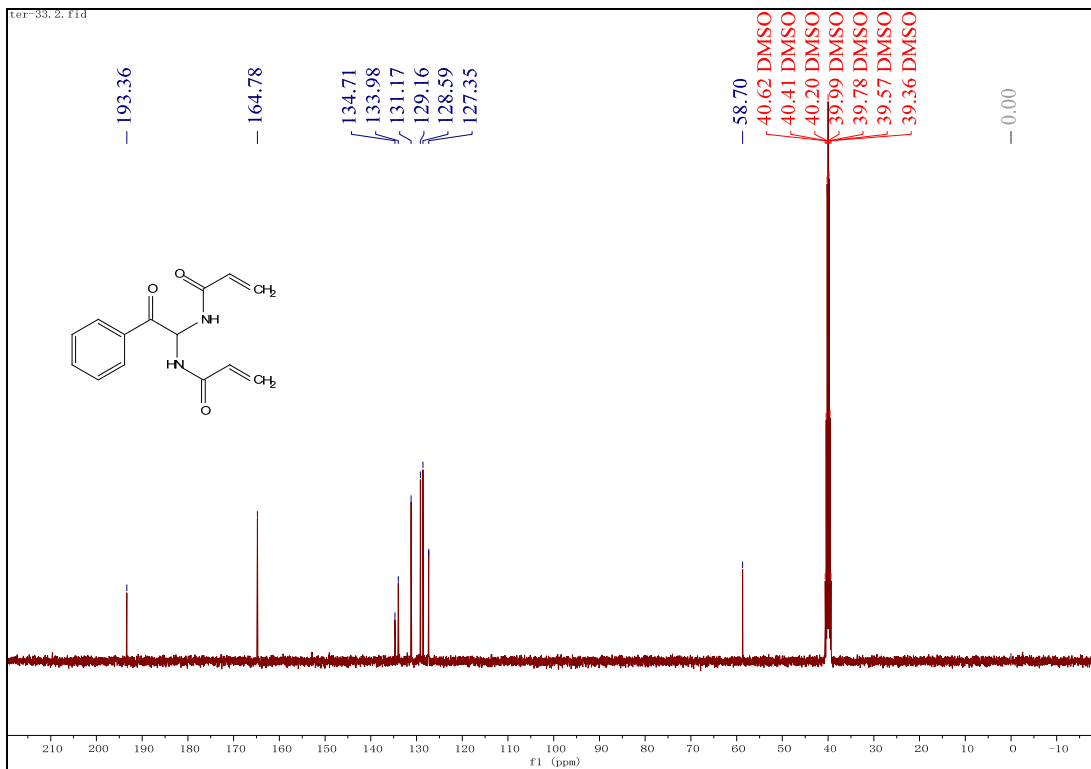
¹H NMR spectrum of 3t (400 MHz, DMSO-*d*₆)



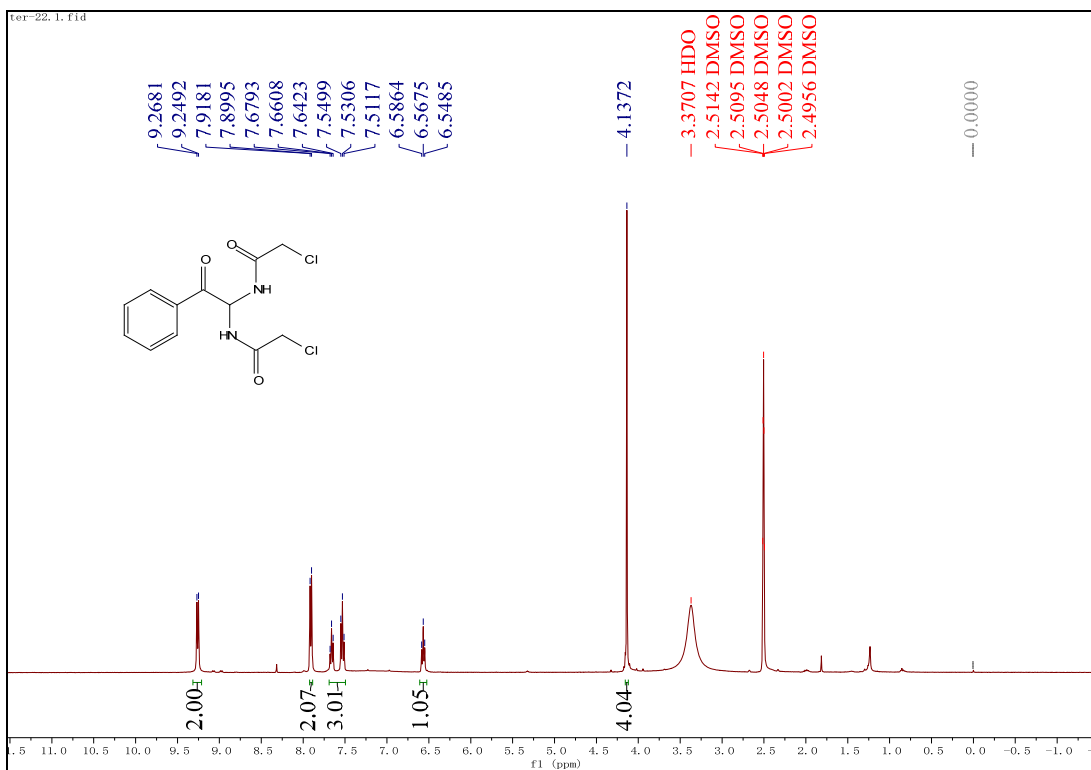
¹³C NMR spectrum of **3t** (100 MHz, DMSO-*d*₆)



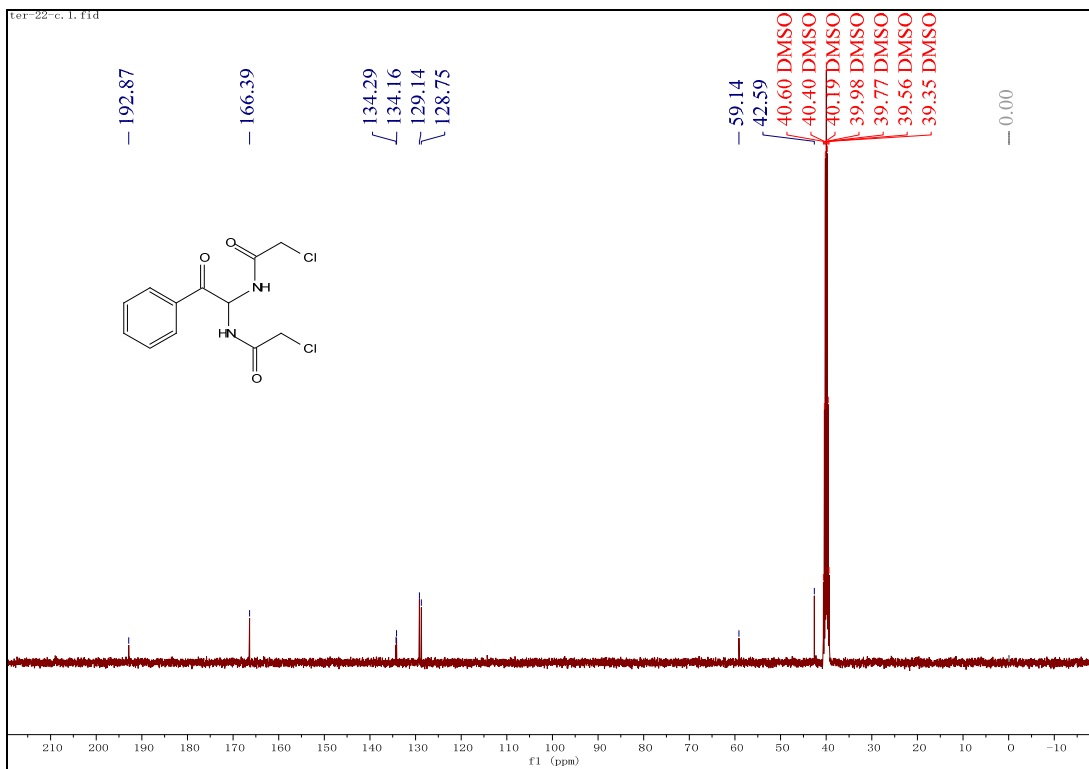
¹H NMR spectrum of **3u** (400 MHz, DMSO-*d*₆)



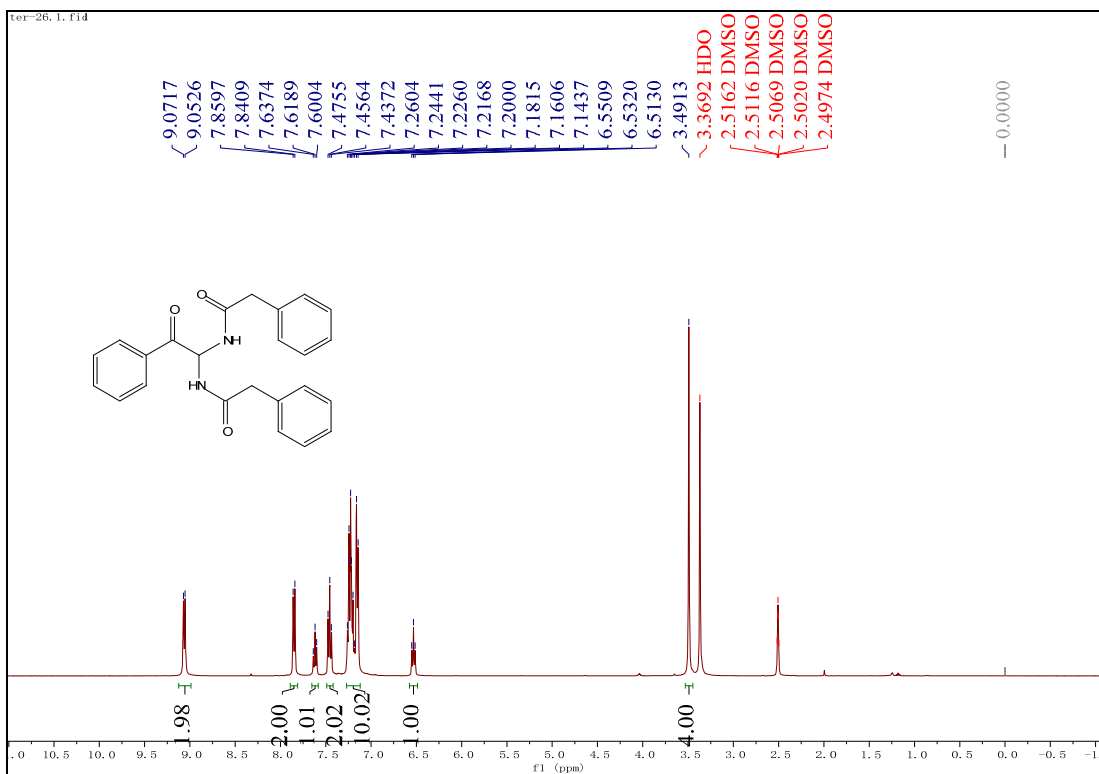
^{13}C NMR spectrum of **3u** (100 MHz, DMSO- d_6)



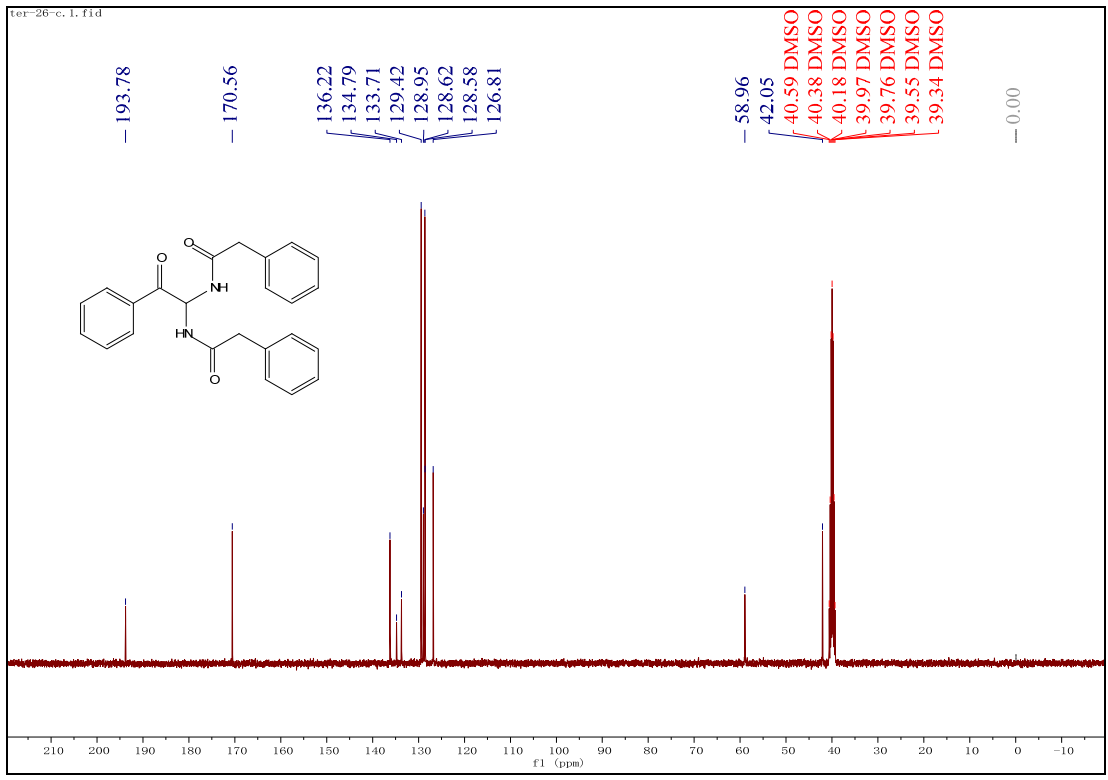
^1H NMR spectrum of **3v** (400 MHz, DMSO- d_6)



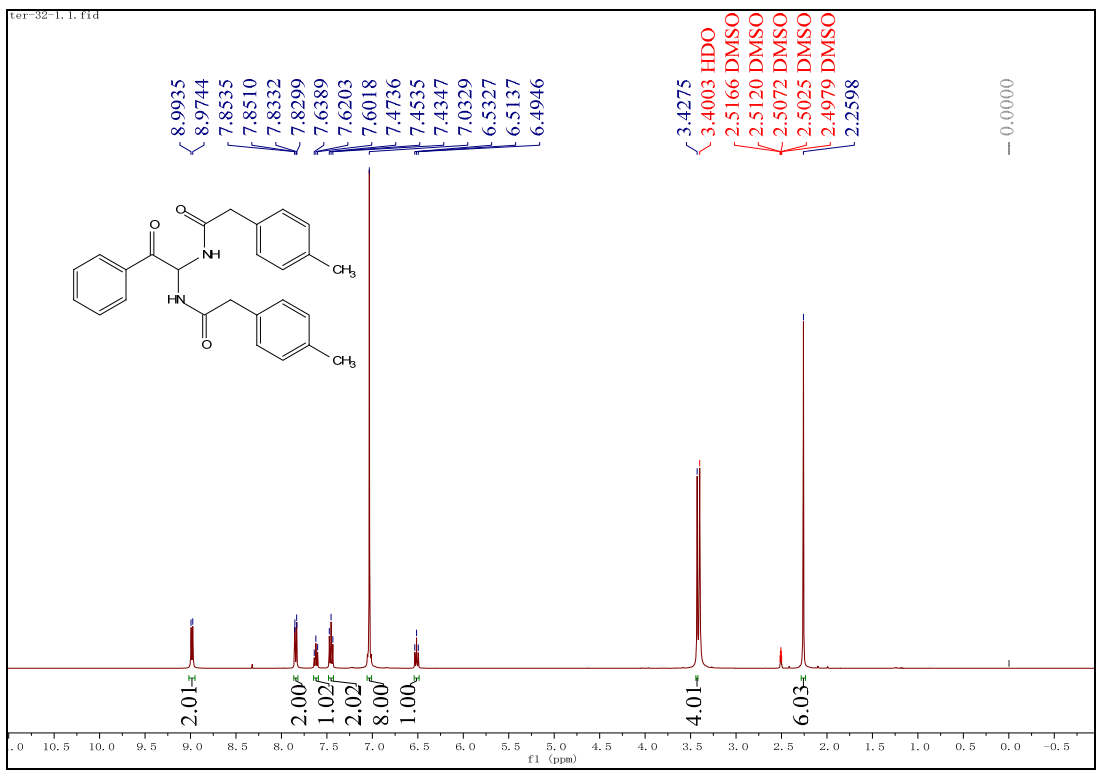
^{13}C NMR spectrum of **3v** (100 MHz, DMSO- d_6)



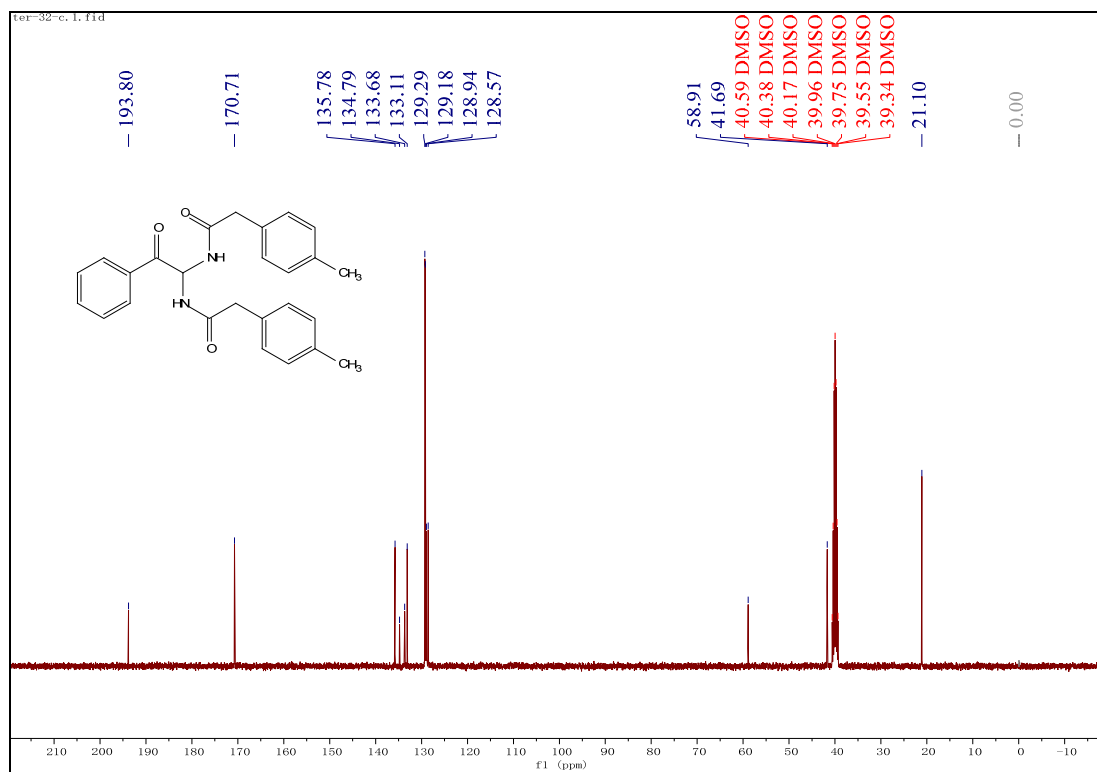
^1H NMR spectrum of **3w** (400 MHz, DMSO- d_6)



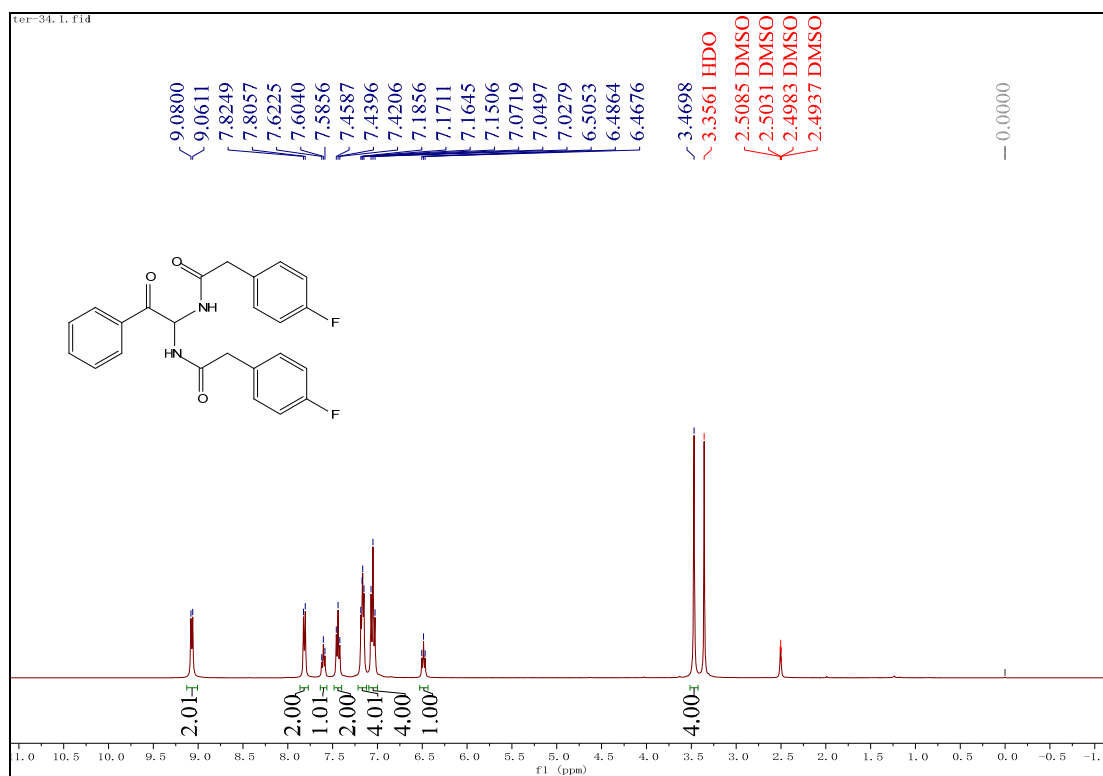
^{13}C NMR spectrum of **3w** (100 MHz, $\text{DMSO-}d_6$)



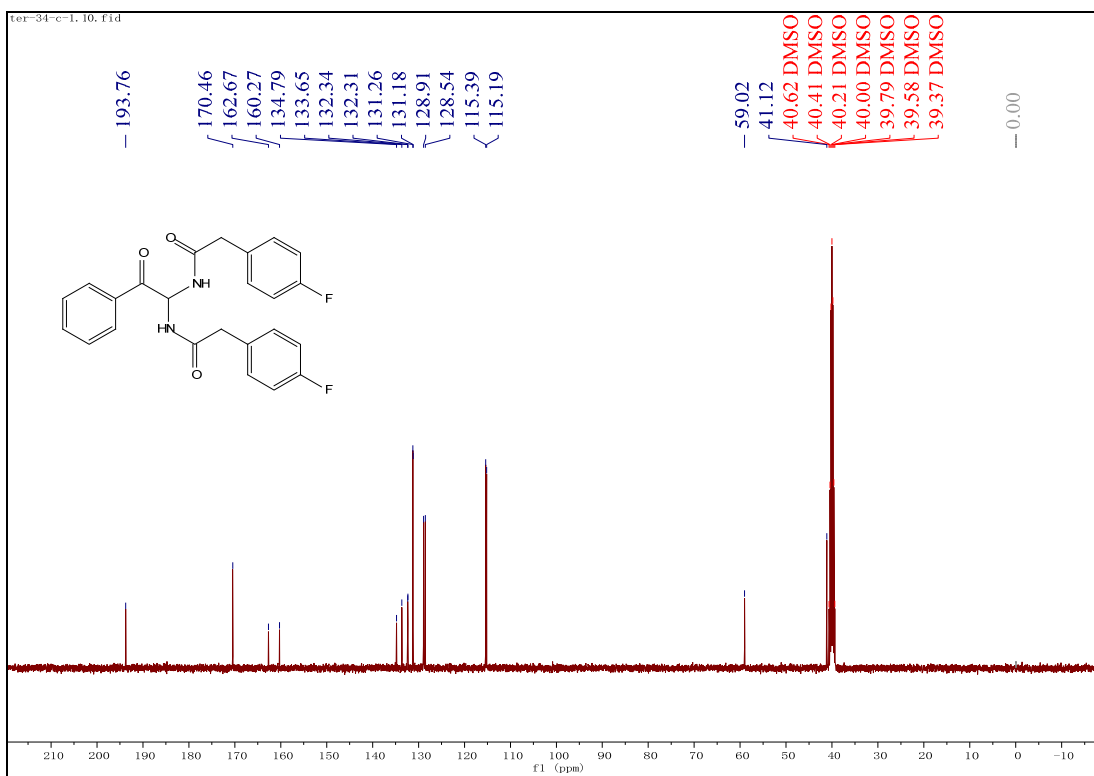
^1H NMR spectrum of **3x** (400 MHz, $\text{DMSO-}d_6$)



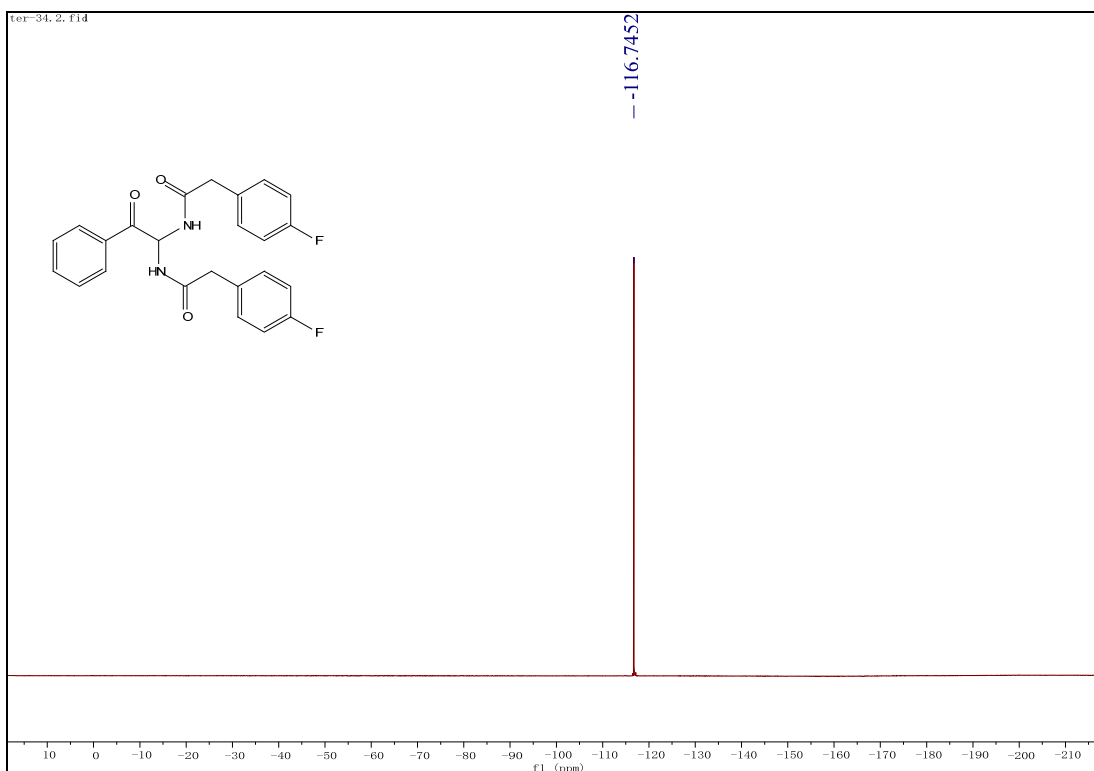
^{13}C NMR spectrum of **3x** (100 MHz, DMSO- d_6)



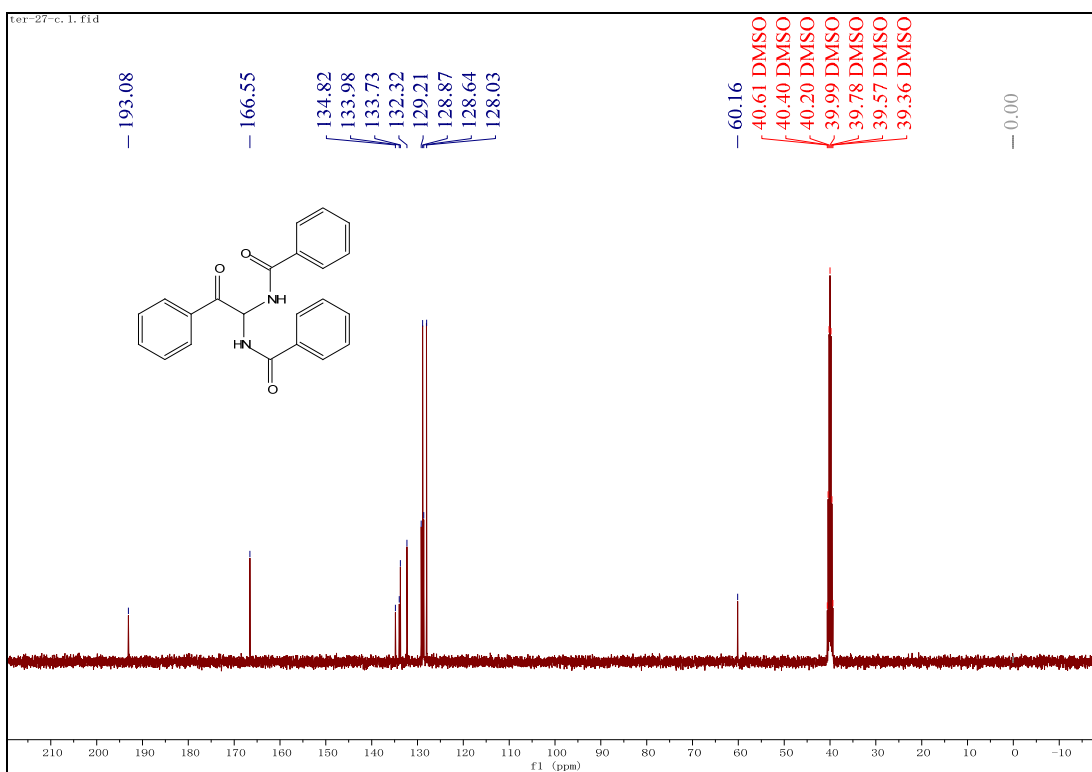
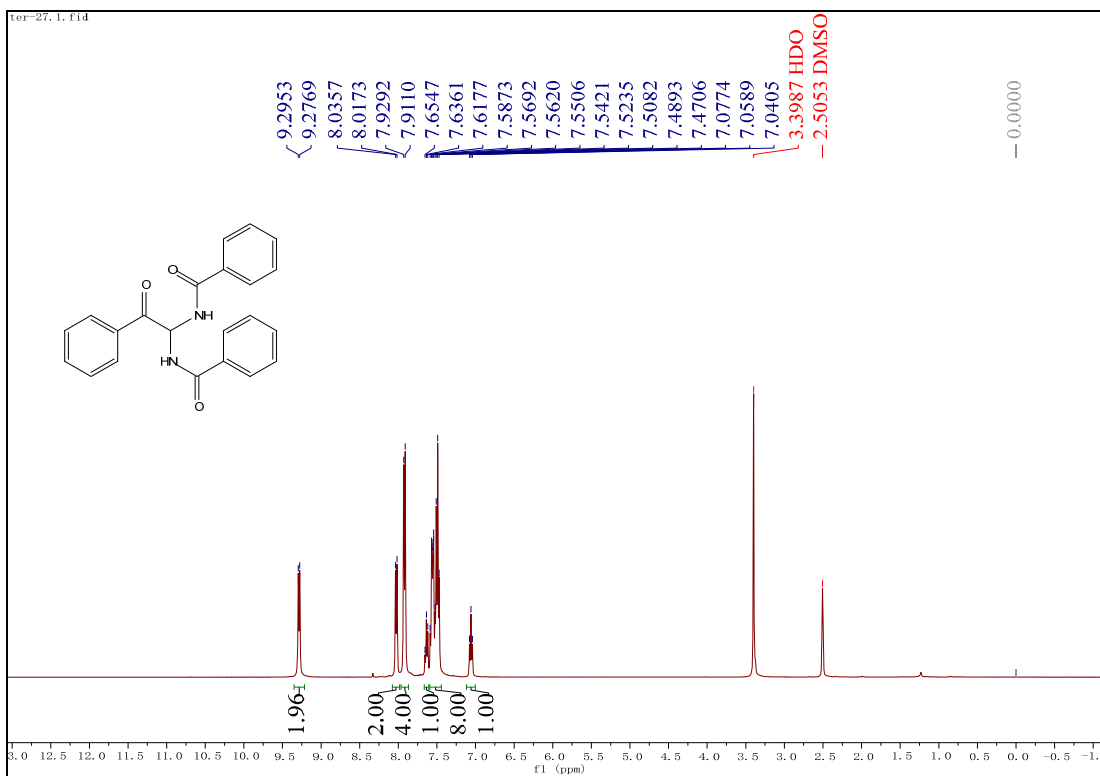
^1H NMR spectrum of **3y** (400 MHz, DMSO- d_6)

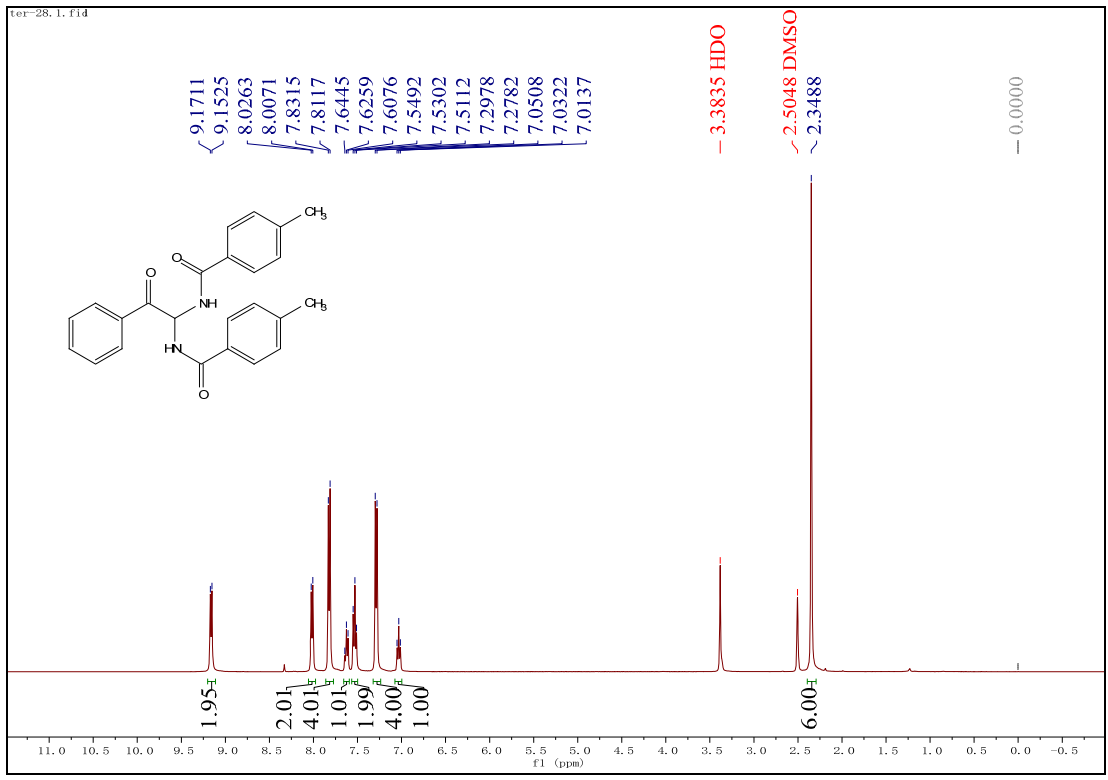


^{13}C NMR spectrum of **3y** (100 MHz, $\text{DMSO-}d_6$)

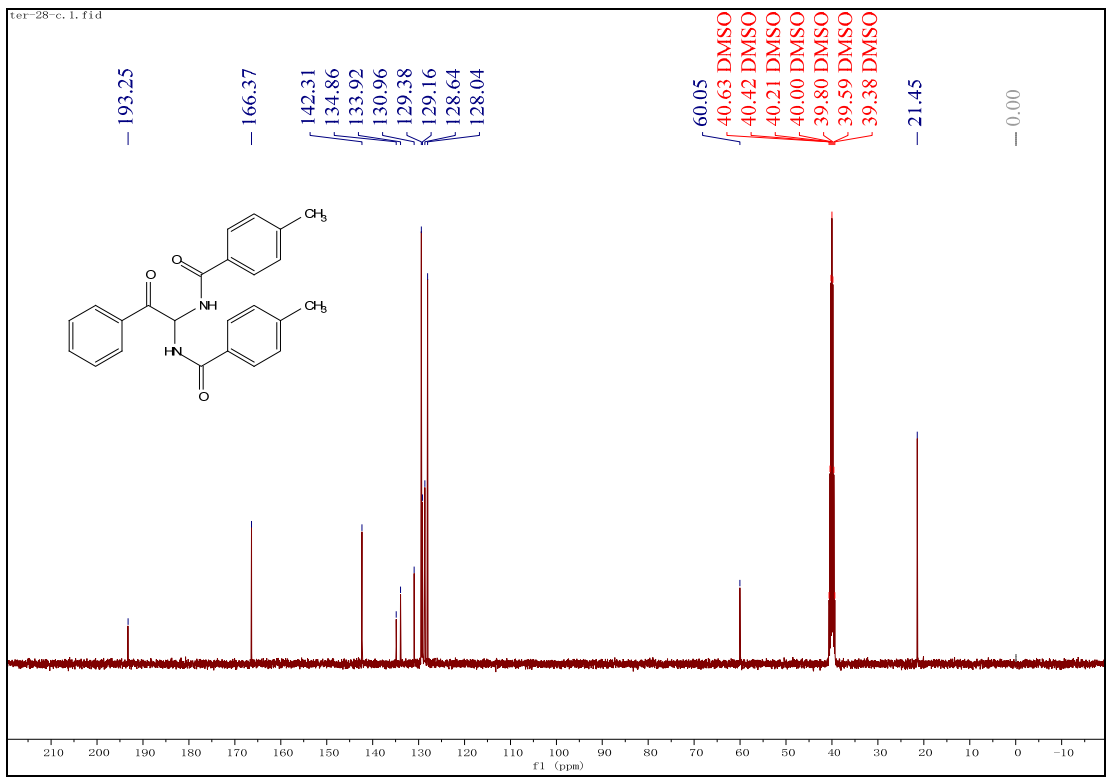


^{19}F NMR spectrum of **3v** (376 MHz, $\text{DMSO-}d_6$)

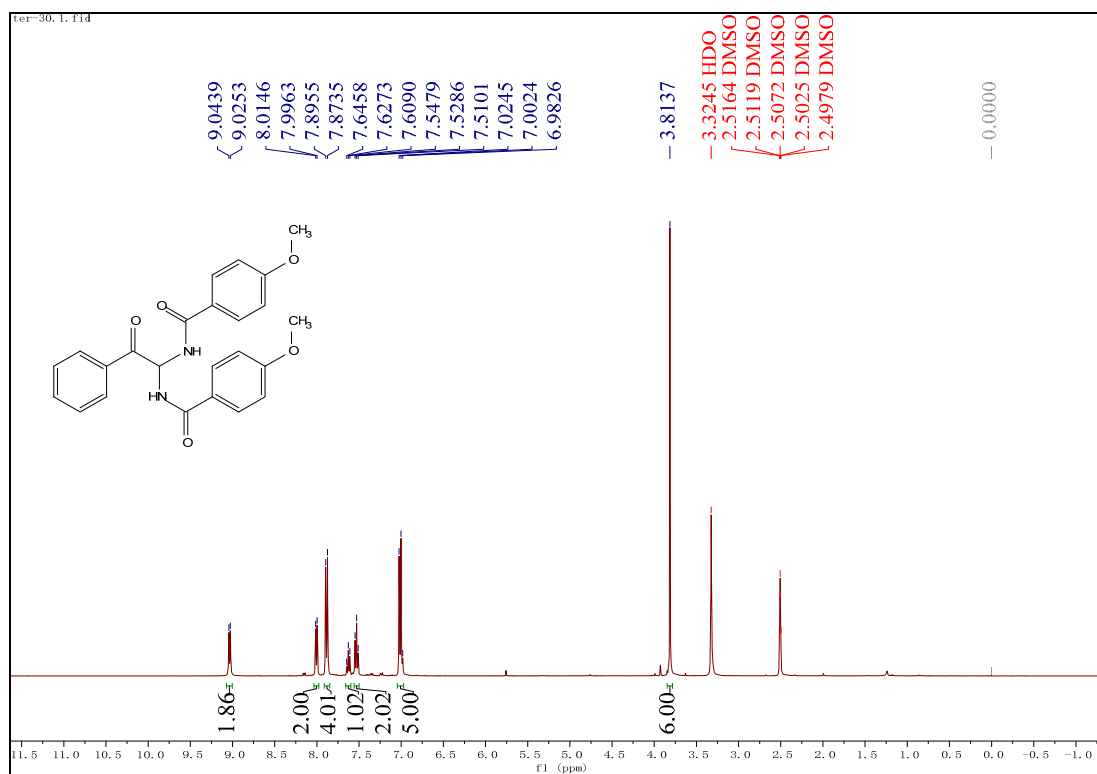




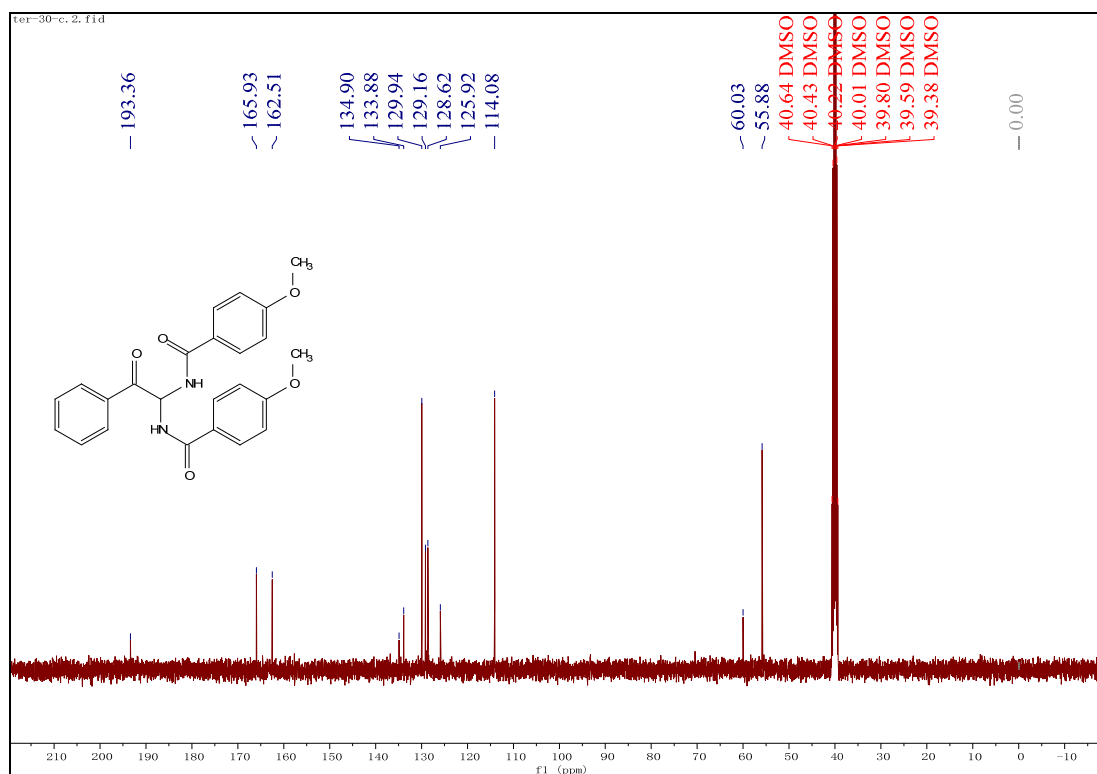
¹H NMR spectrum of **3aa** (400 MHz, DMSO-*d*₆)



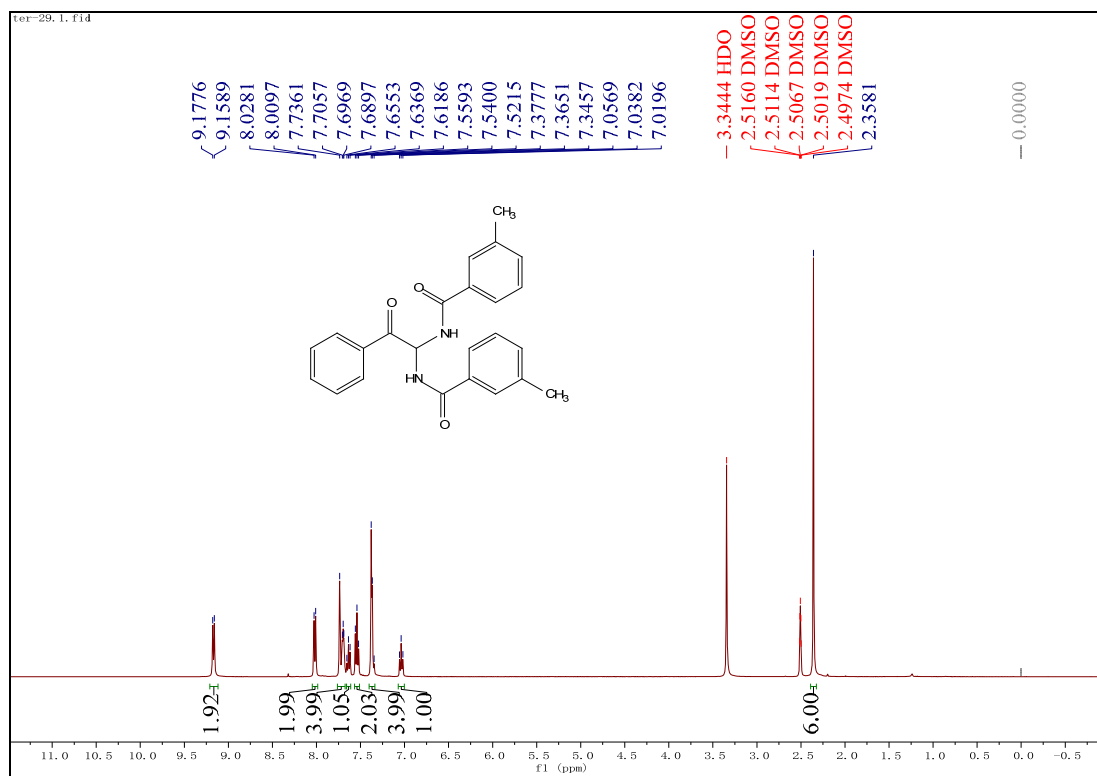
¹³C NMR spectrum of **3aa** (100 MHz, DMSO-*d*₆)



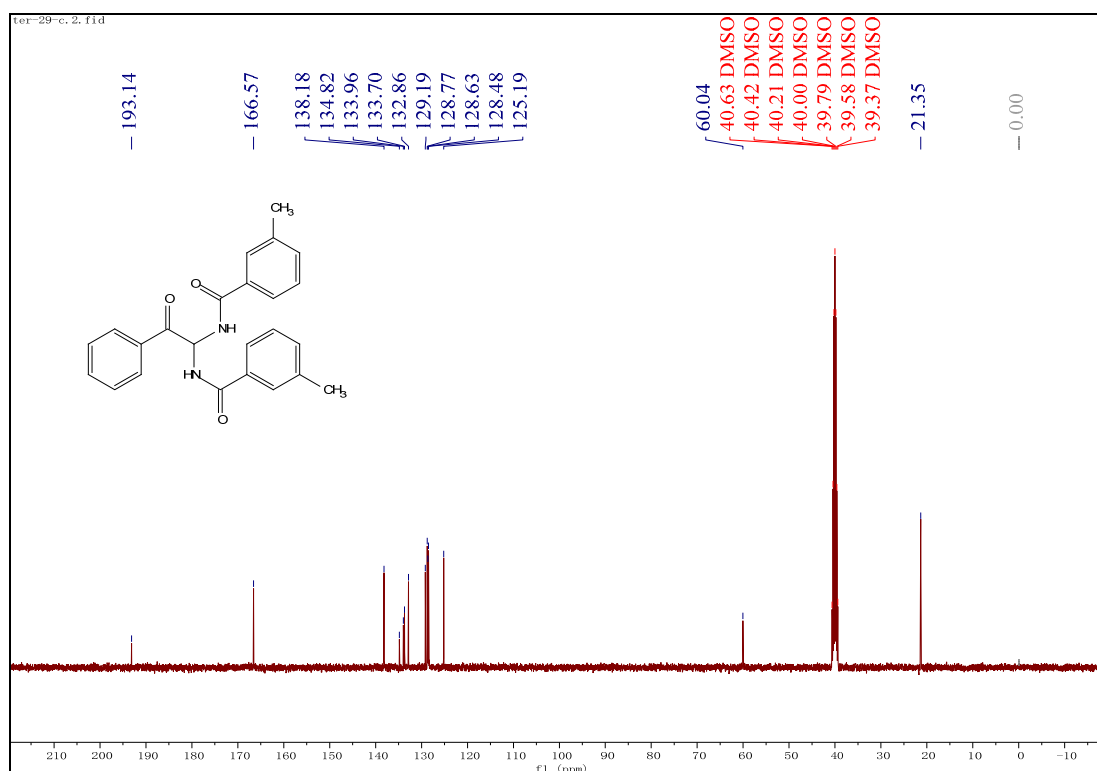
¹H NMR spectrum of **3ab** (400 MHz, DMSO-*d*₆)



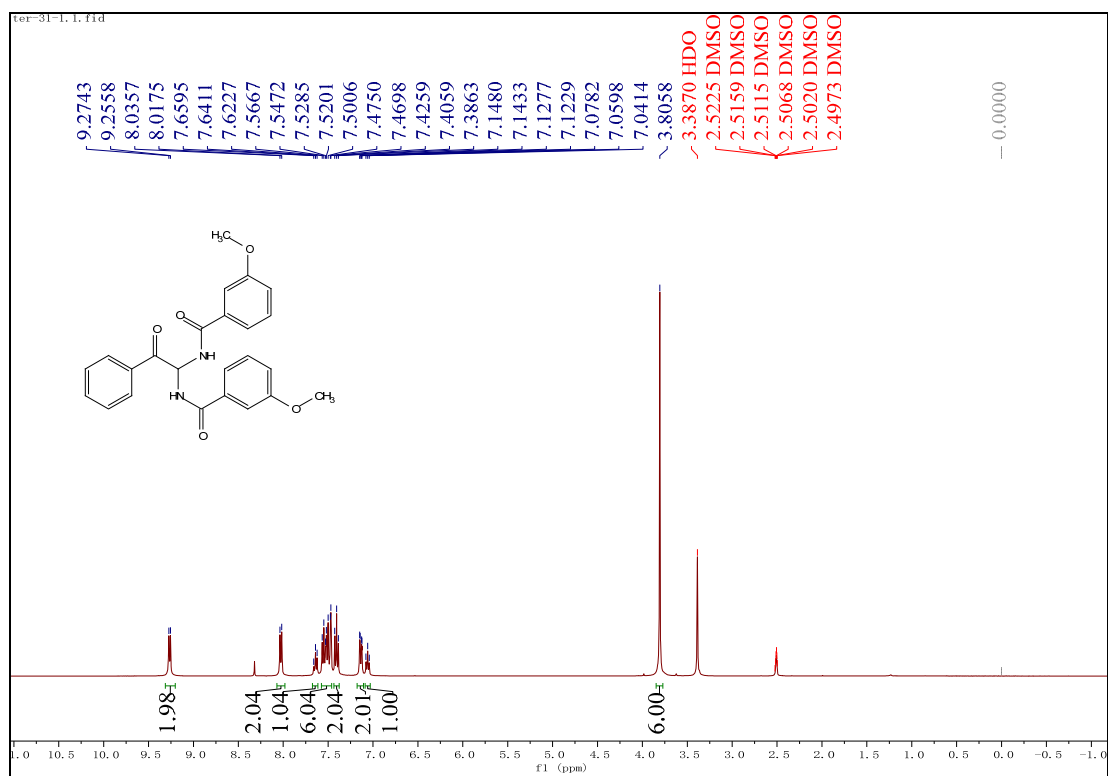
¹³C NMR spectrum of **3ab** (100 MHz, DMSO-*d*₆)



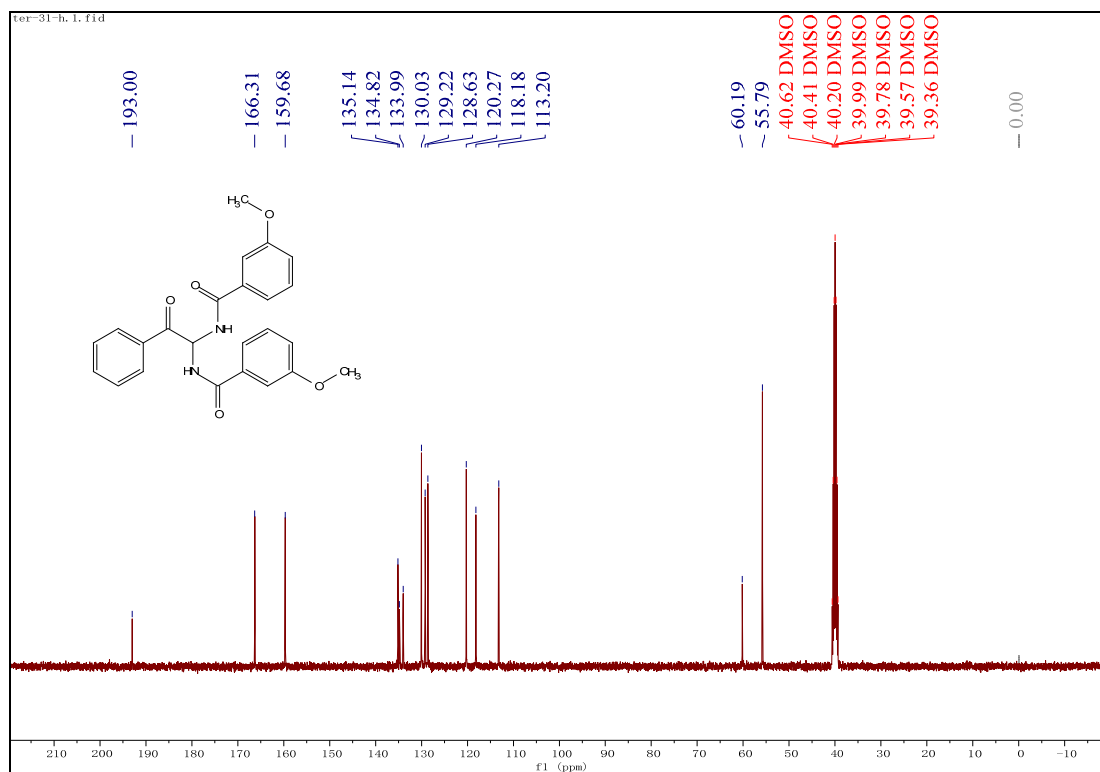
¹H NMR spectrum of **3ac** (400 MHz, DMSO-*d*₆)



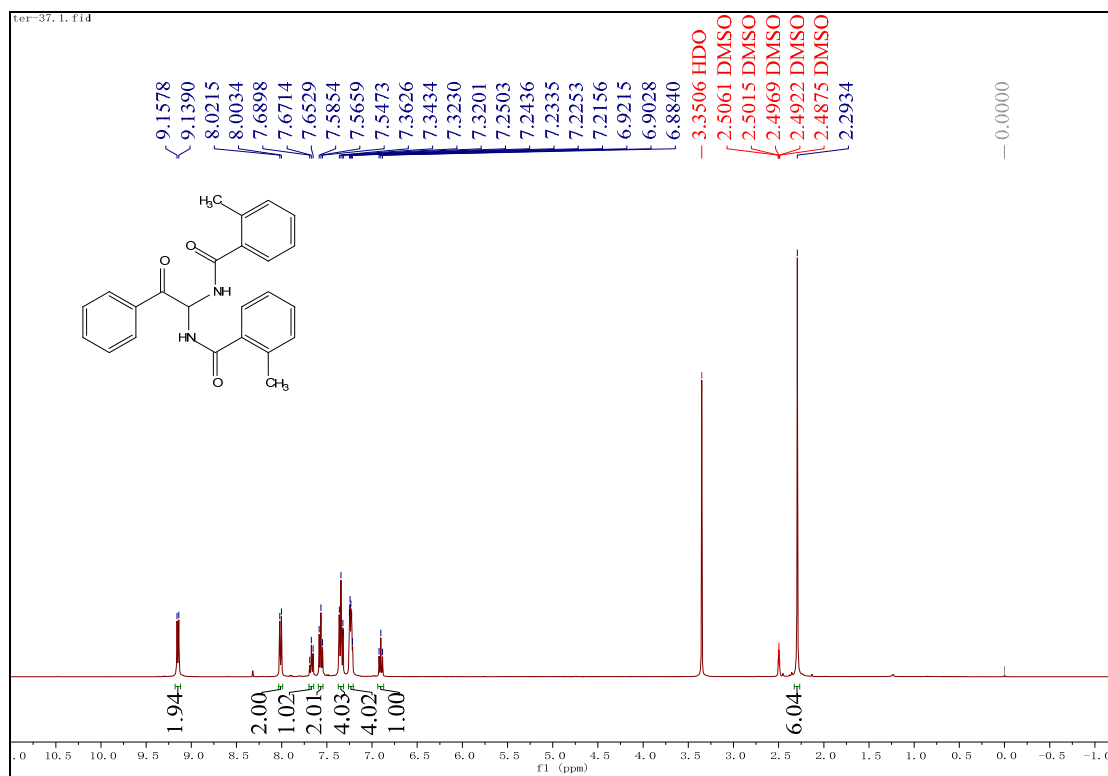
¹³C NMR spectrum of **3ac** (100 MHz, DMSO-*d*₆)



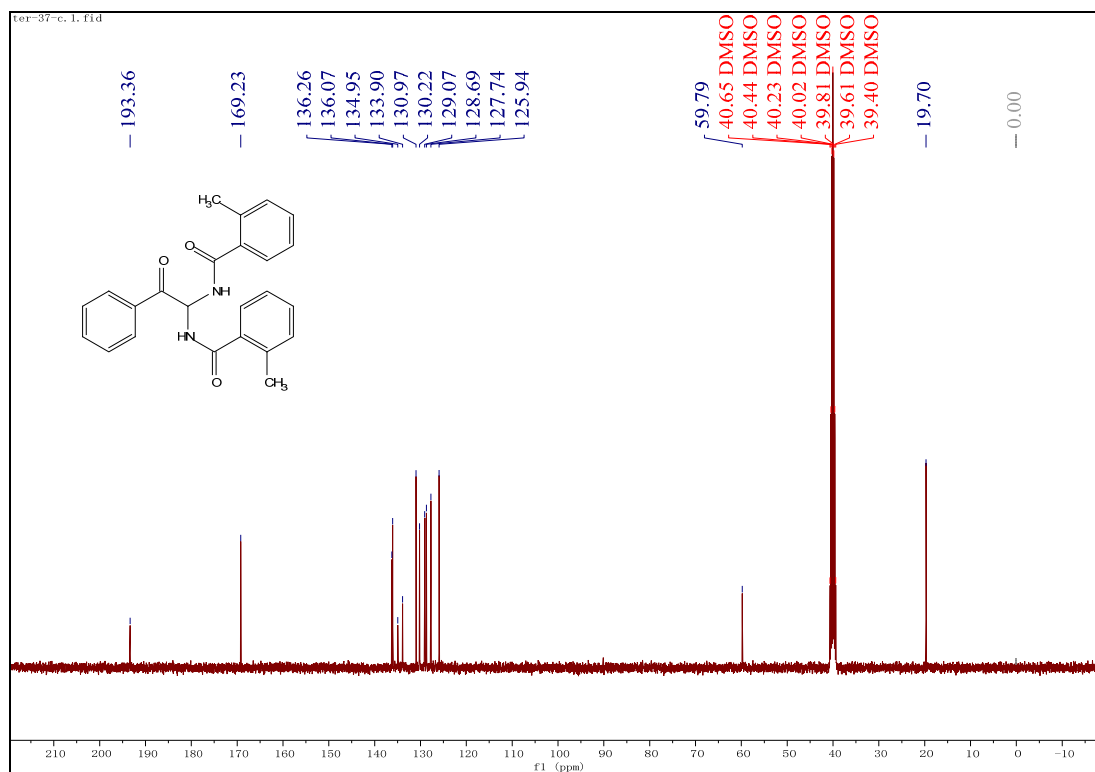
¹H NMR spectrum of **3ad** (400 MHz, DMSO-*d*₆)



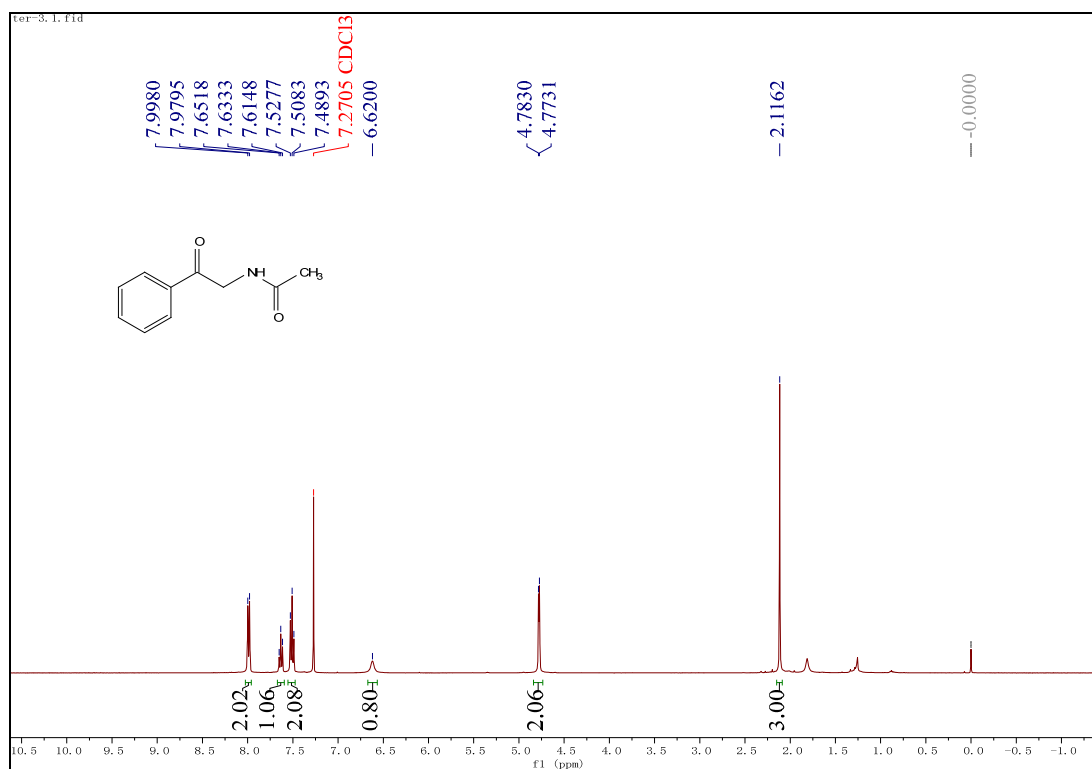
¹³C NMR spectrum of **3ad** (100 MHz, DMSO-*d*₆)



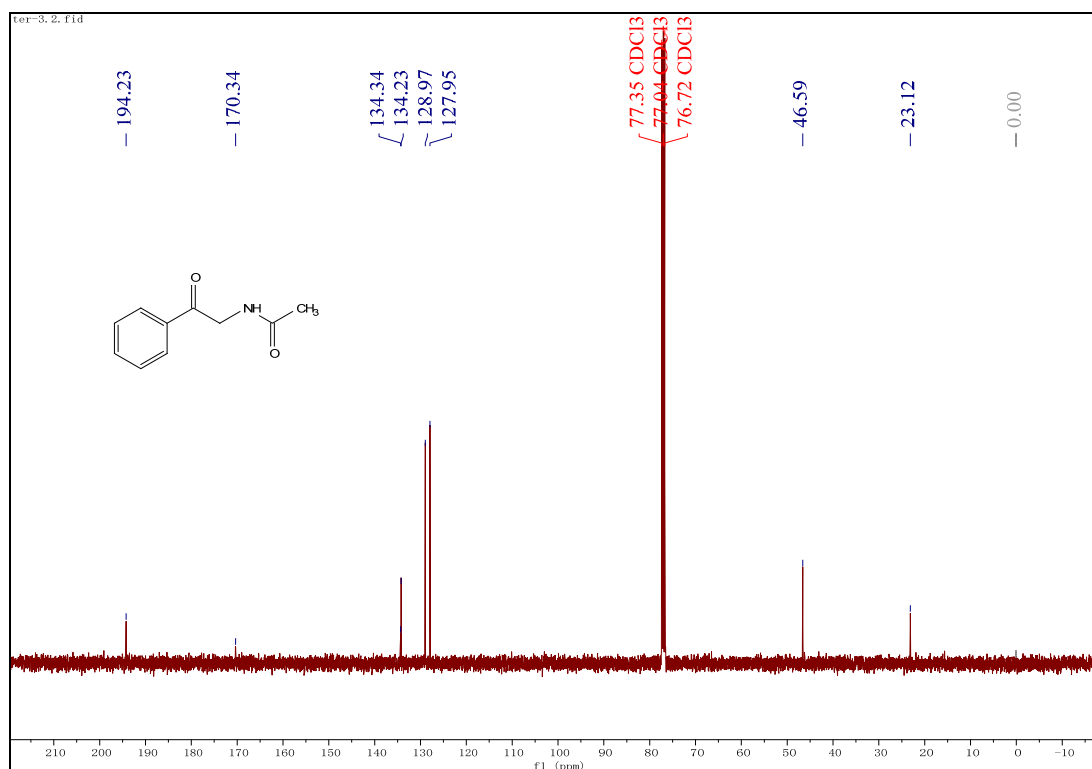
¹H NMR spectrum of **3ae** (400 MHz, DMSO-*d*₆)



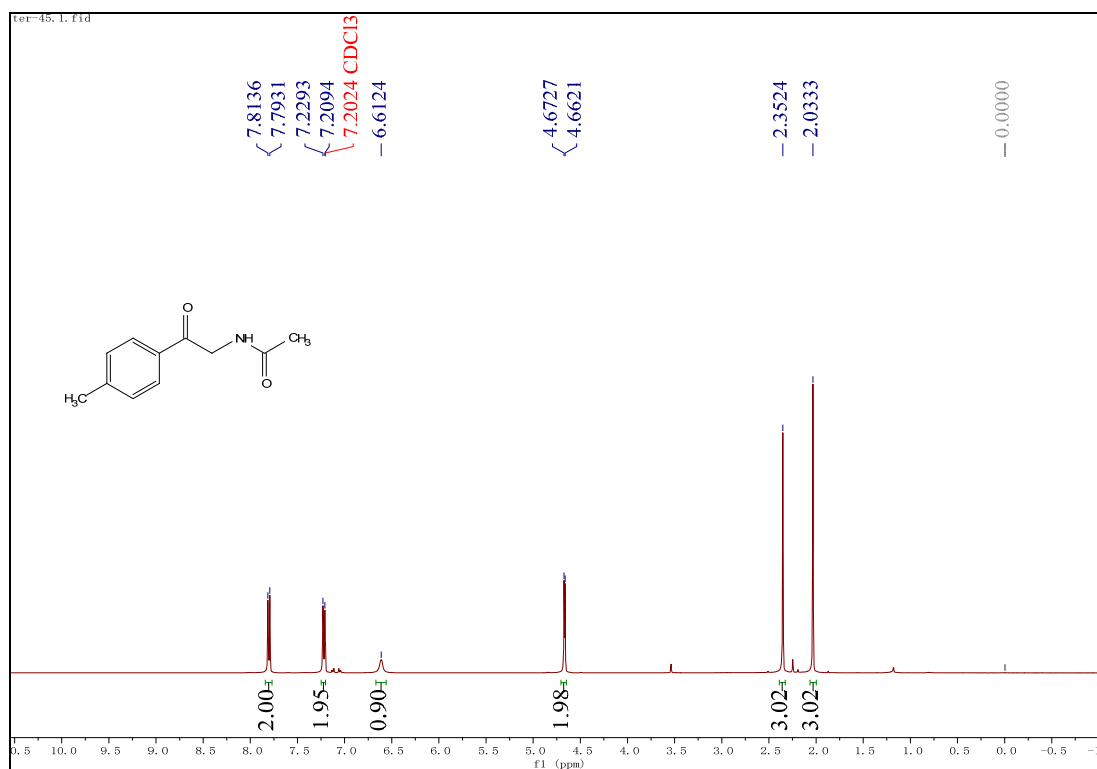
¹³C NMR spectrum of **3ae** (100 MHz, DMSO-*d*₆)



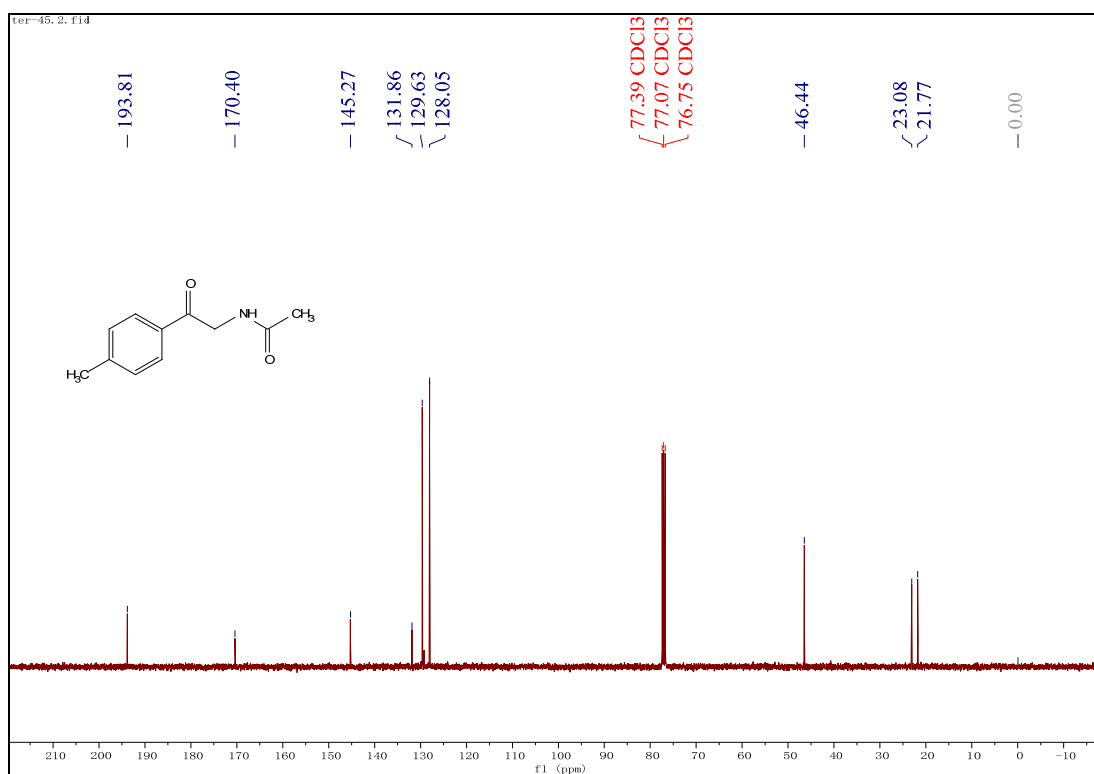
¹H NMR spectrum of **4a** (400 MHz, CDCl₃)



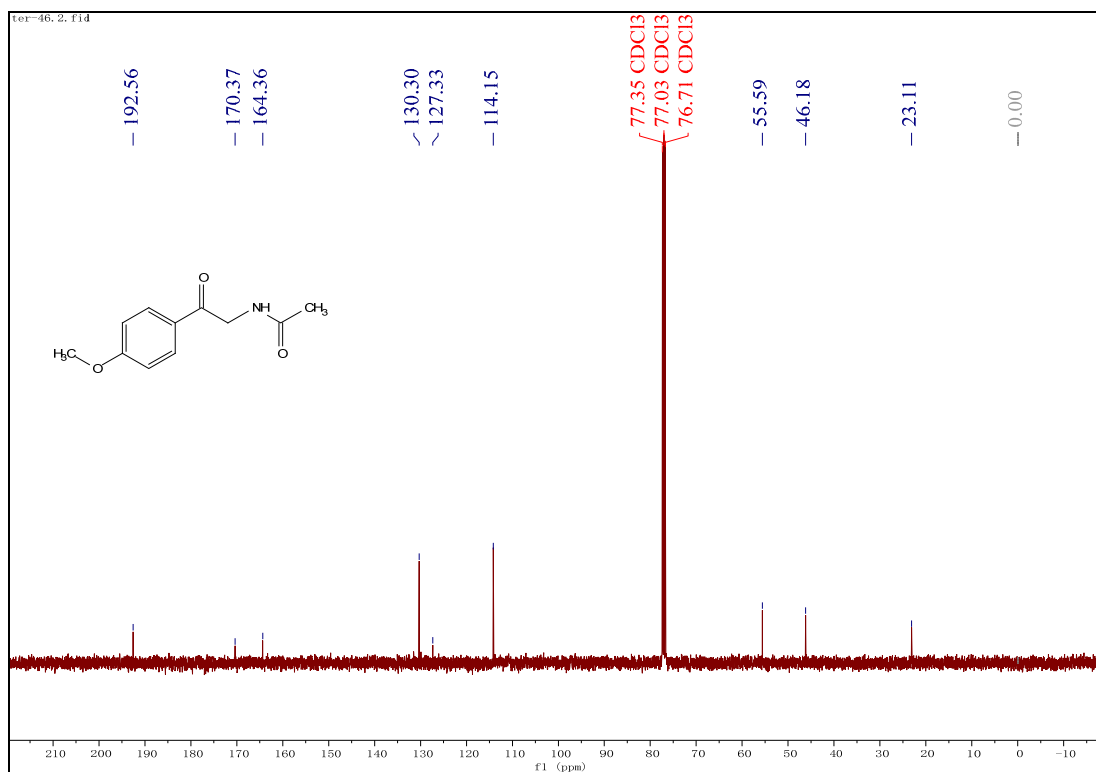
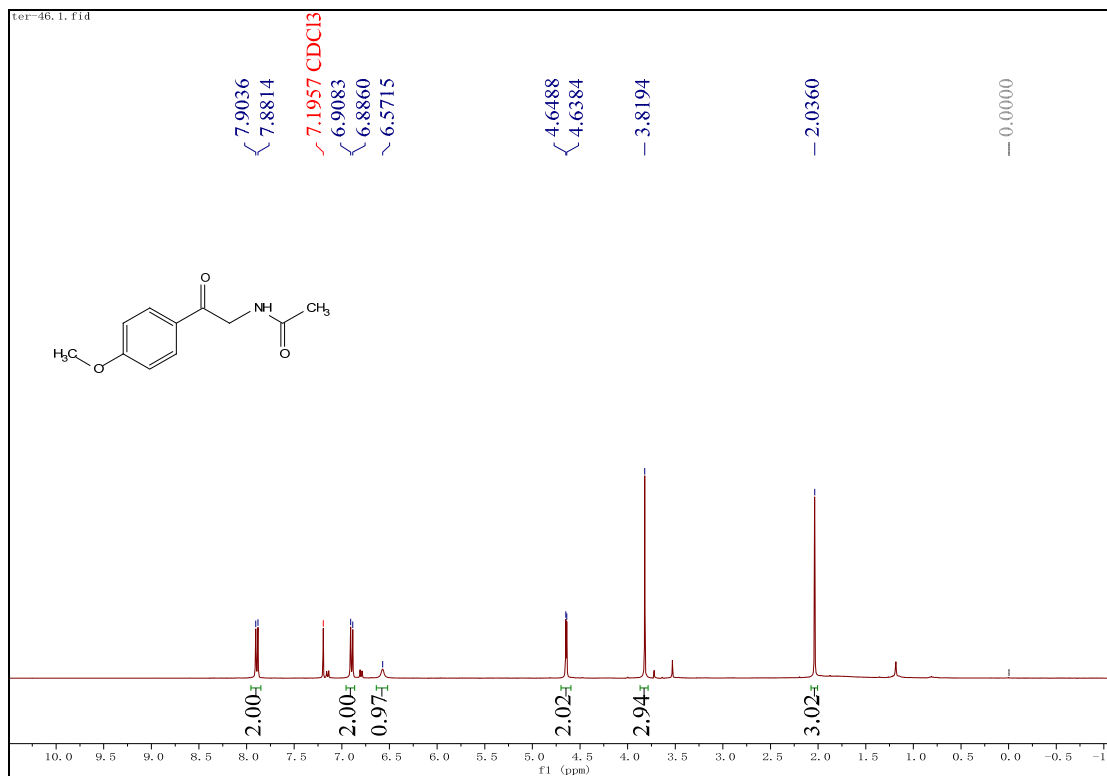
¹³C NMR spectrum of **4a** (100 MHz, CDCl₃)

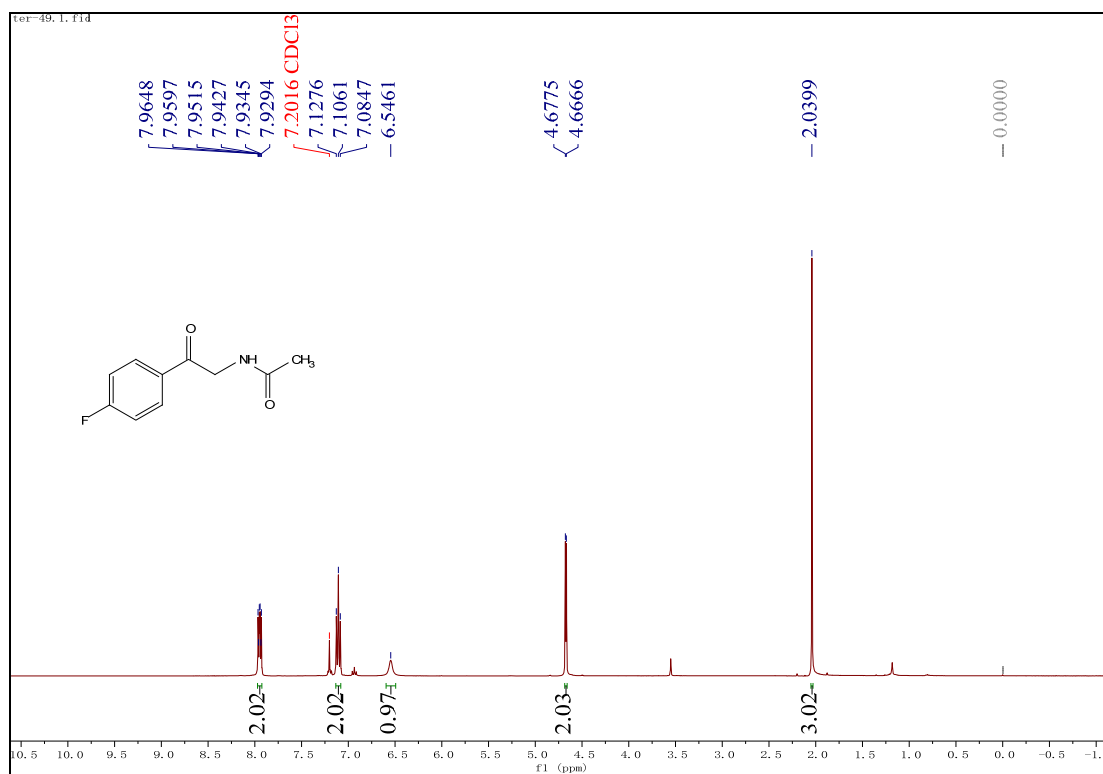


¹H NMR spectrum of **4b** (400 MHz, CDCl₃)

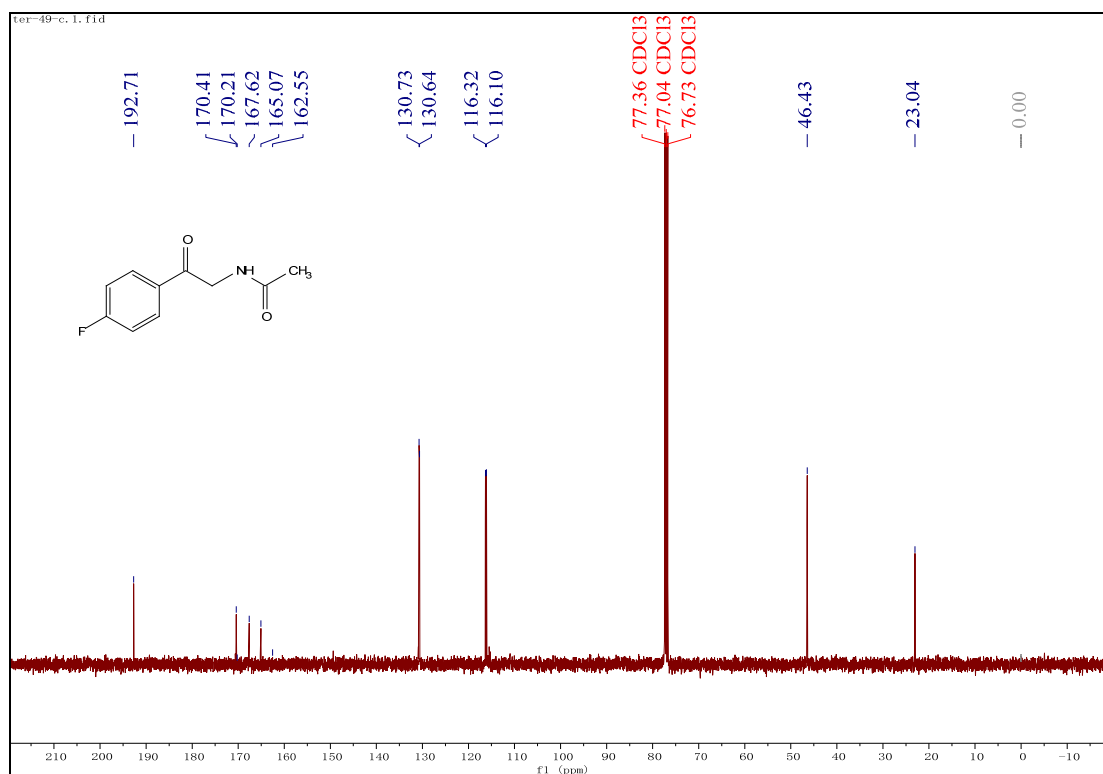


¹³C NMR spectrum of **4b** (100 MHz, CDCl₃)

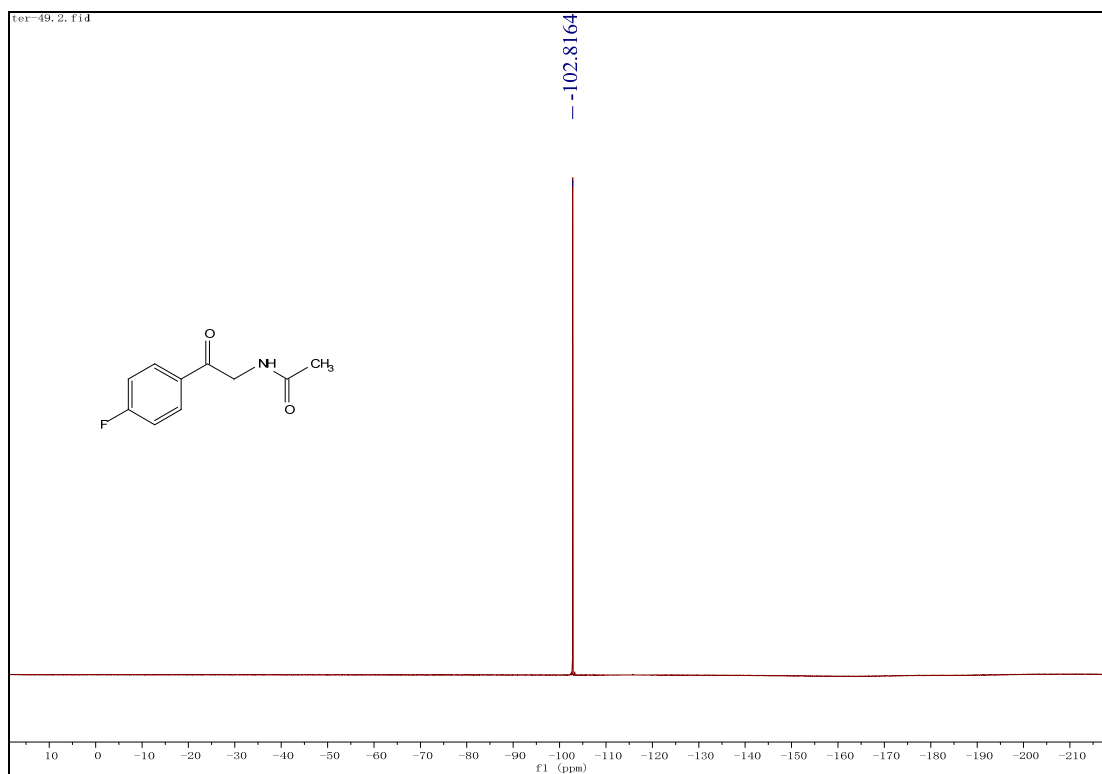




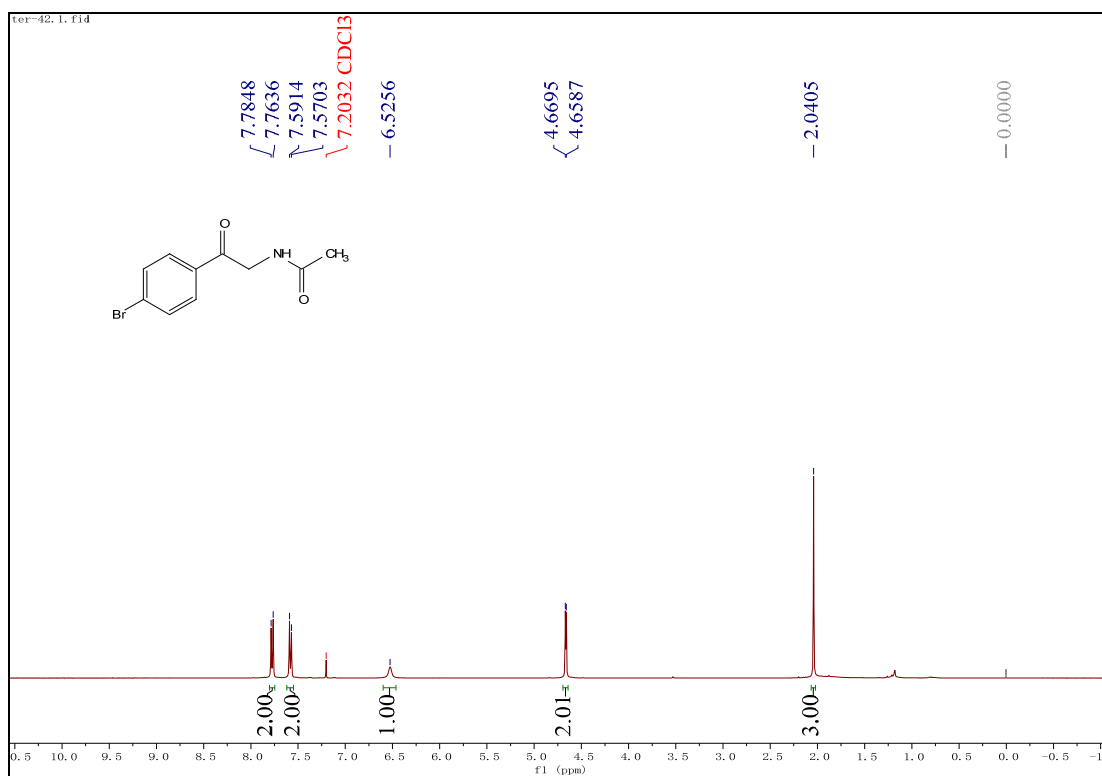
¹H NMR spectrum of **4d** (400 MHz, CDCl₃)



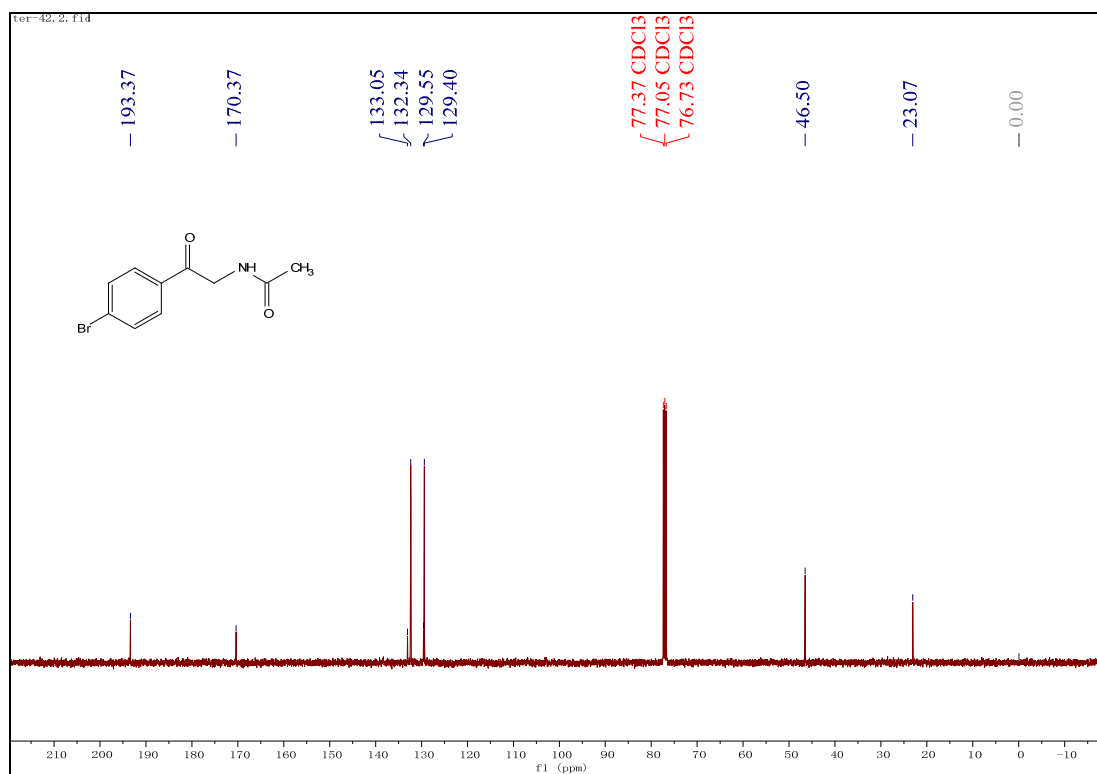
¹³C NMR spectrum of **4d** (100 MHz, CDCl₃)



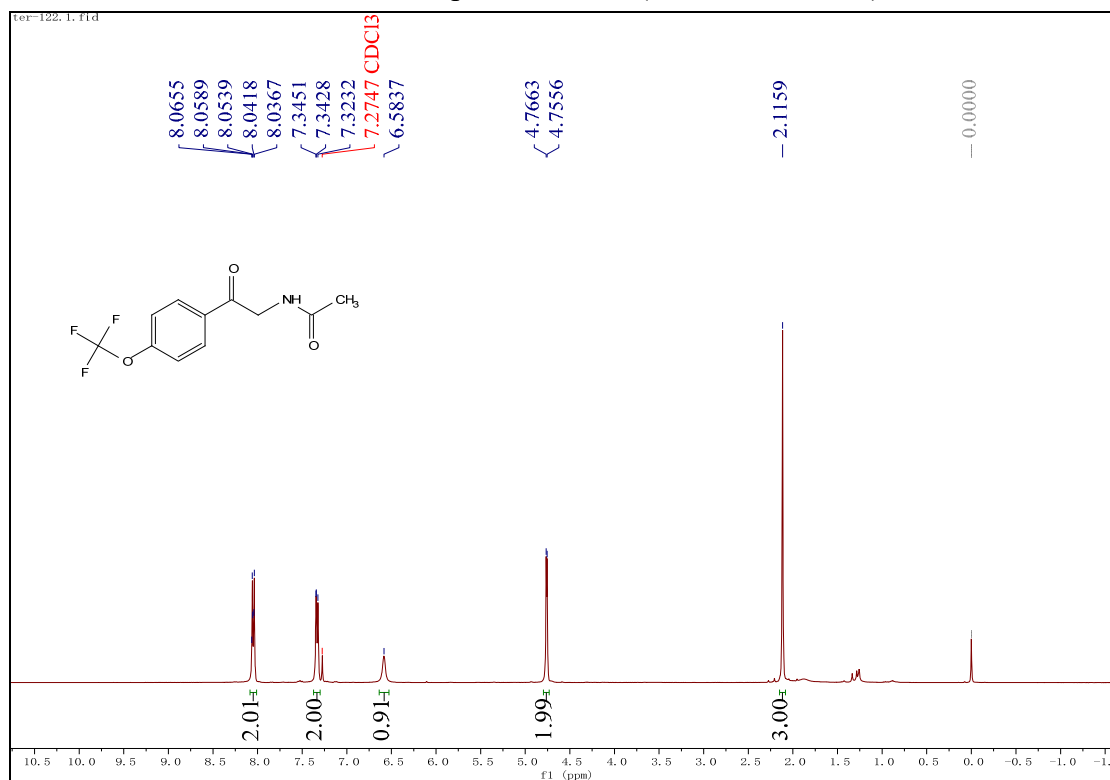
^{19}F NMR spectrum of **4d** (376 MHz, CDCl_3)



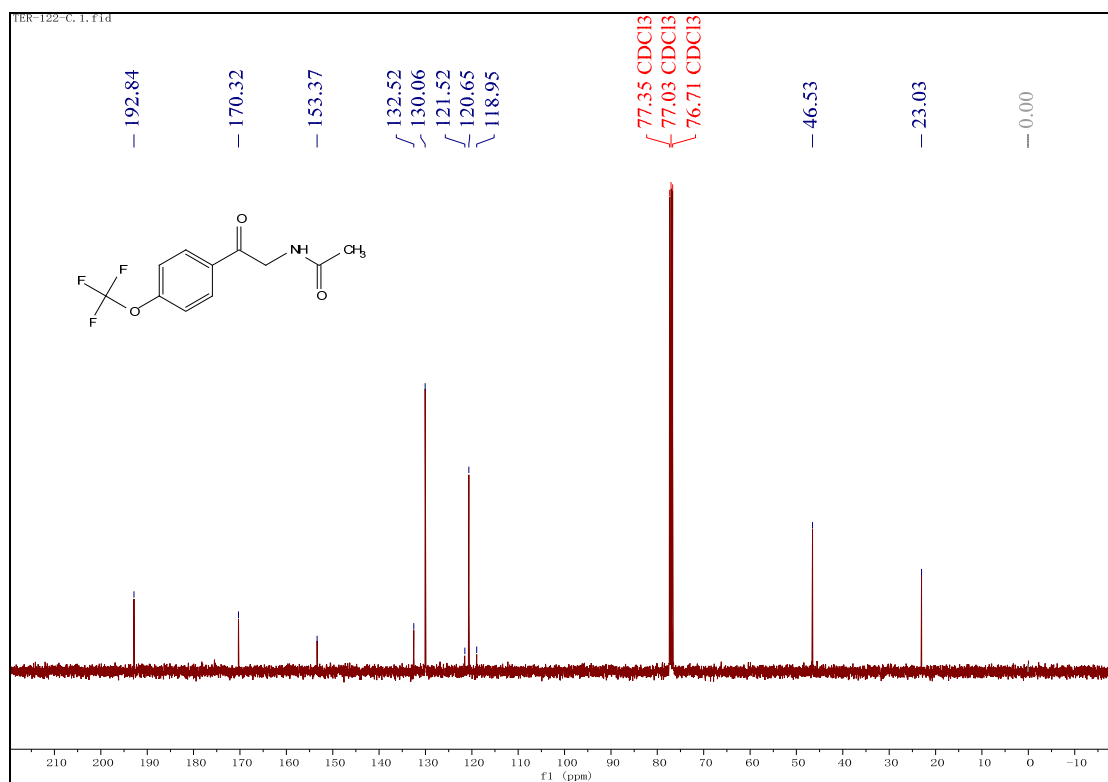
^1H NMR spectrum of **4e** (400 MHz, CDCl_3)



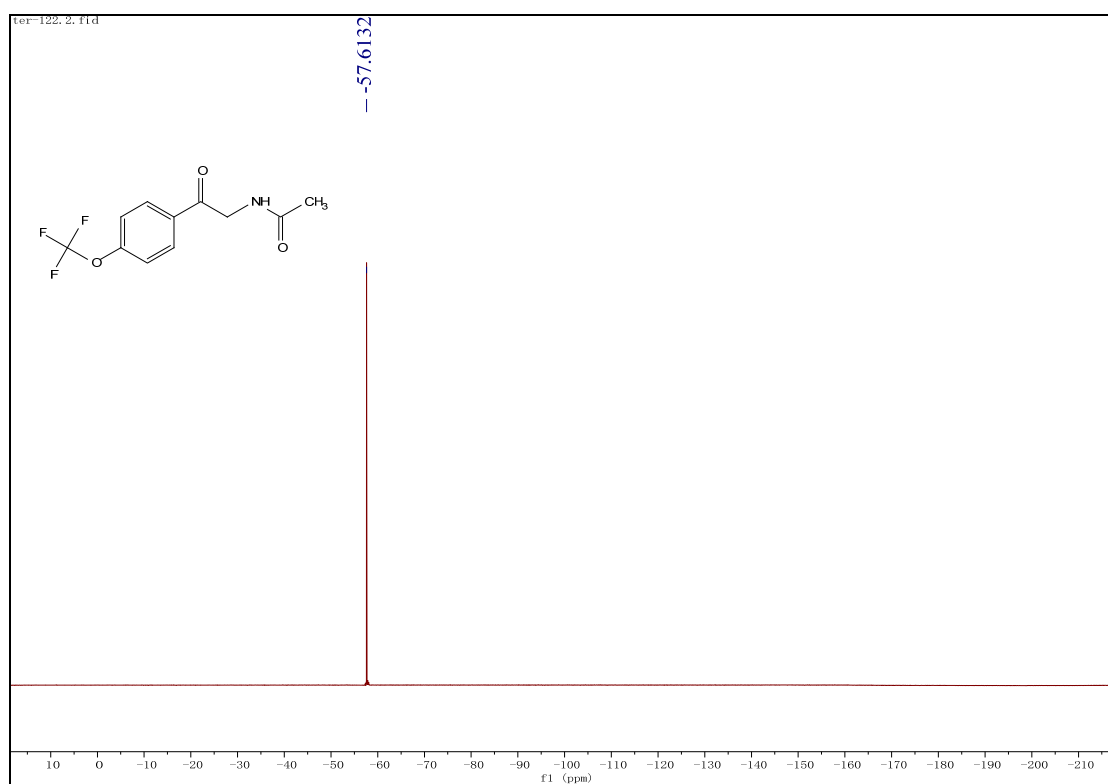
^{13}C NMR spectrum of **4e** (100 MHz, CDCl_3)



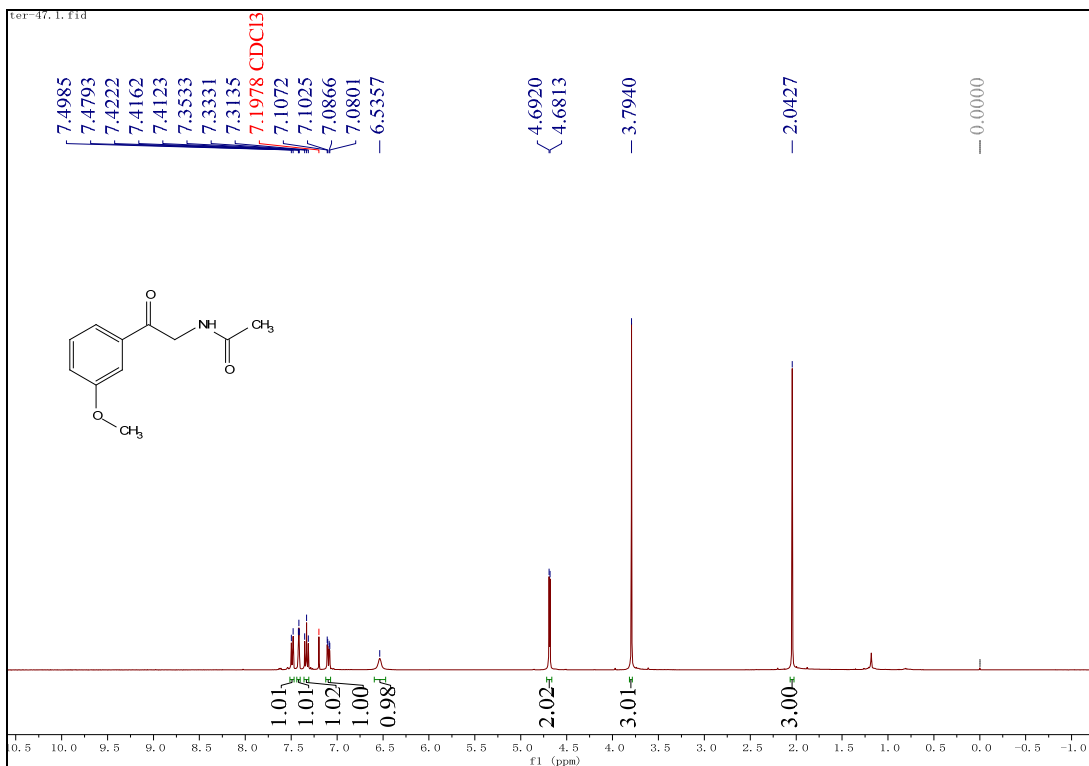
^1H NMR spectrum of **4f** (400 MHz, CDCl_3)



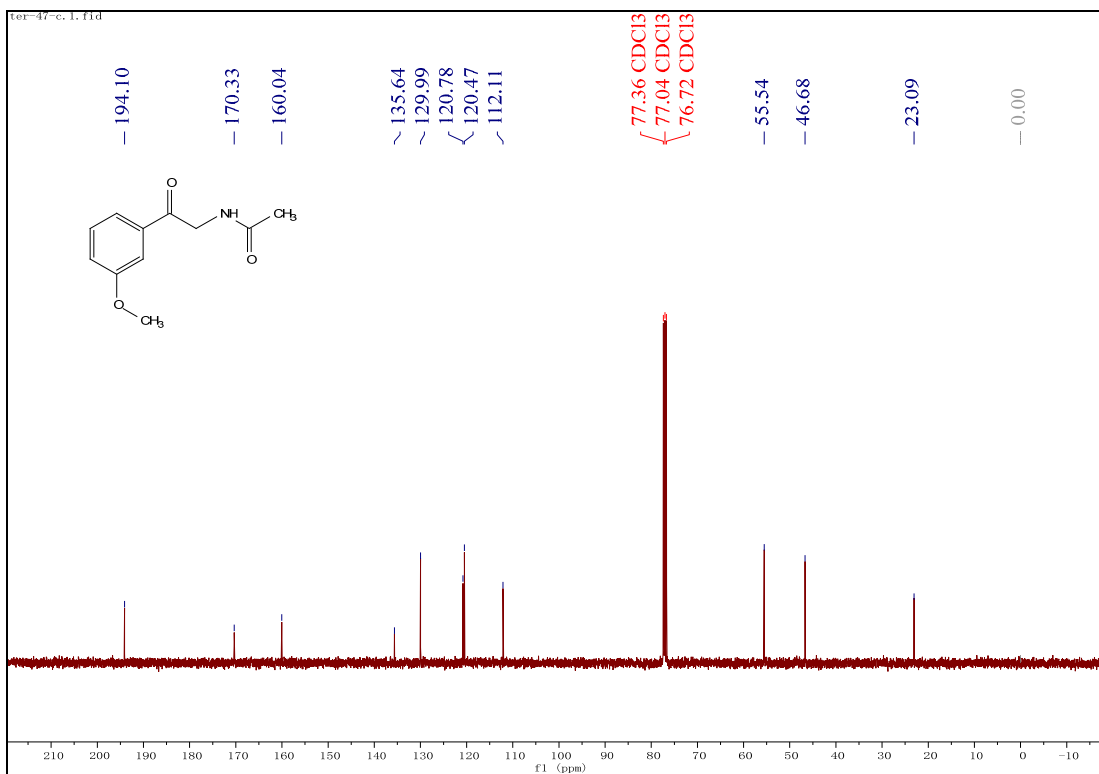
¹³C NMR spectrum of **4f** (100 MHz, CDCl₃)



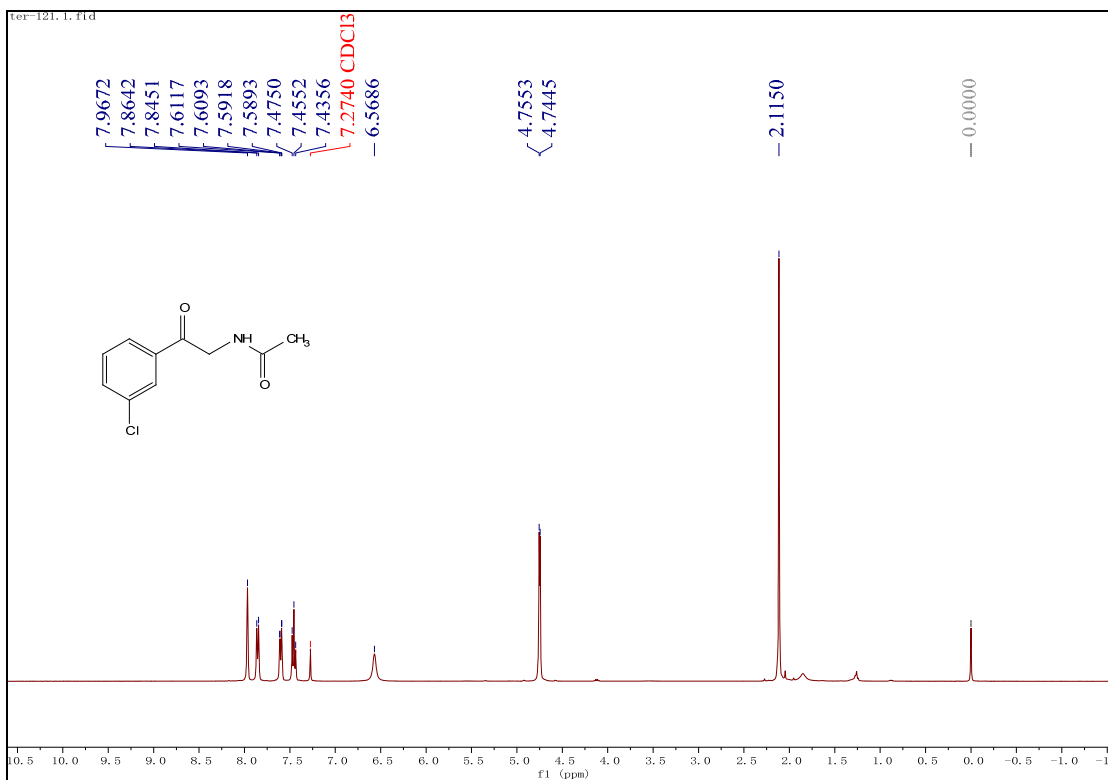
¹⁹F NMR spectrum of **4f** (376 MHz, CDCl₃)



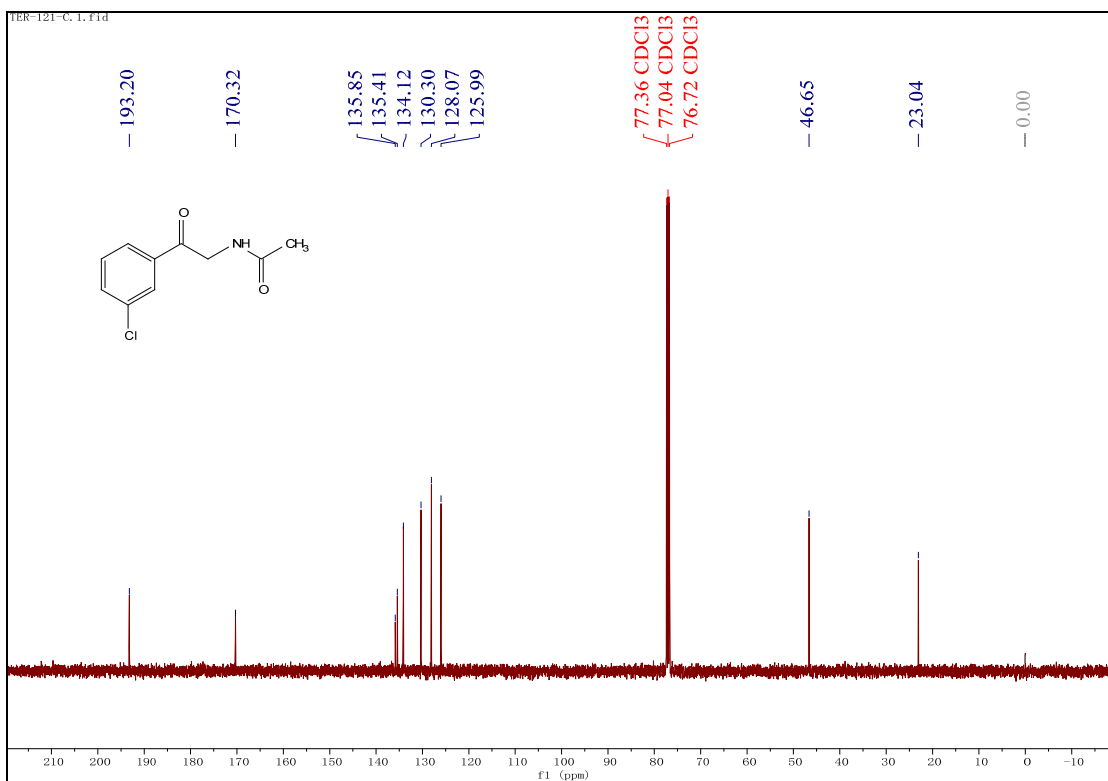
¹H NMR spectrum of **4g** (400 MHz, CDCl₃)



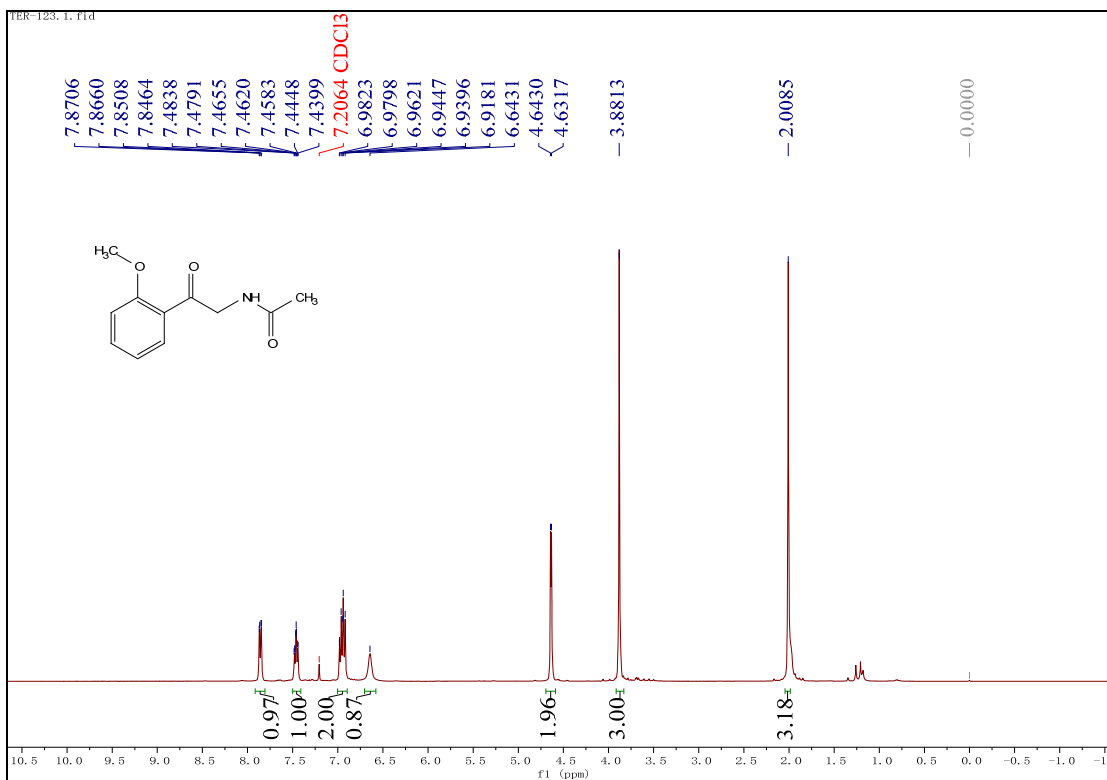
¹³C NMR spectrum of **4g** (100 MHz, CDCl₃)



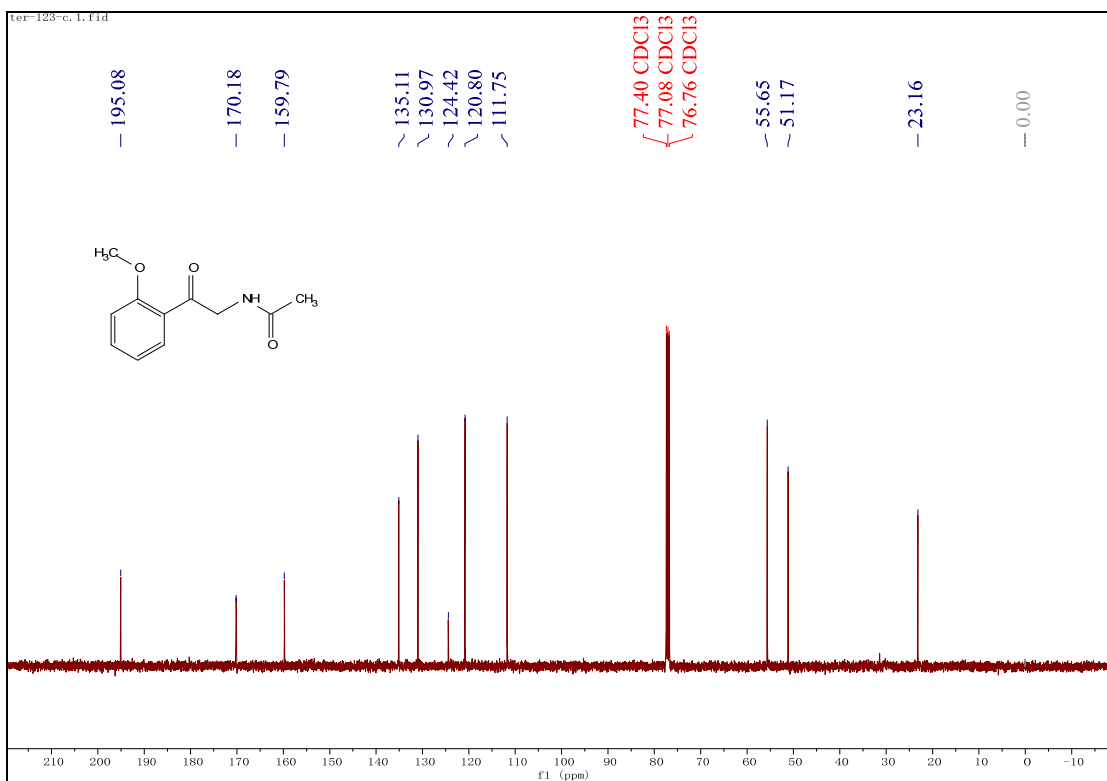
¹H NMR spectrum of **4h** (400 MHz, CDCl₃)



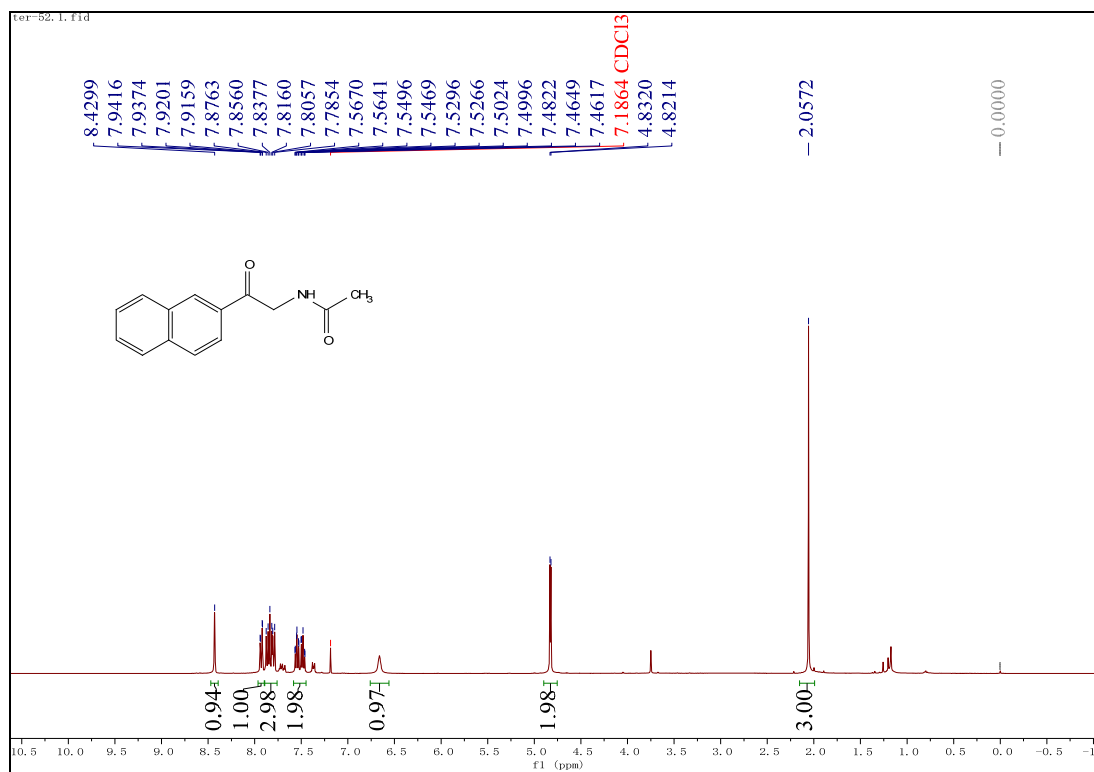
¹³C NMR spectrum of **4h** (100 MHz, CDCl₃)



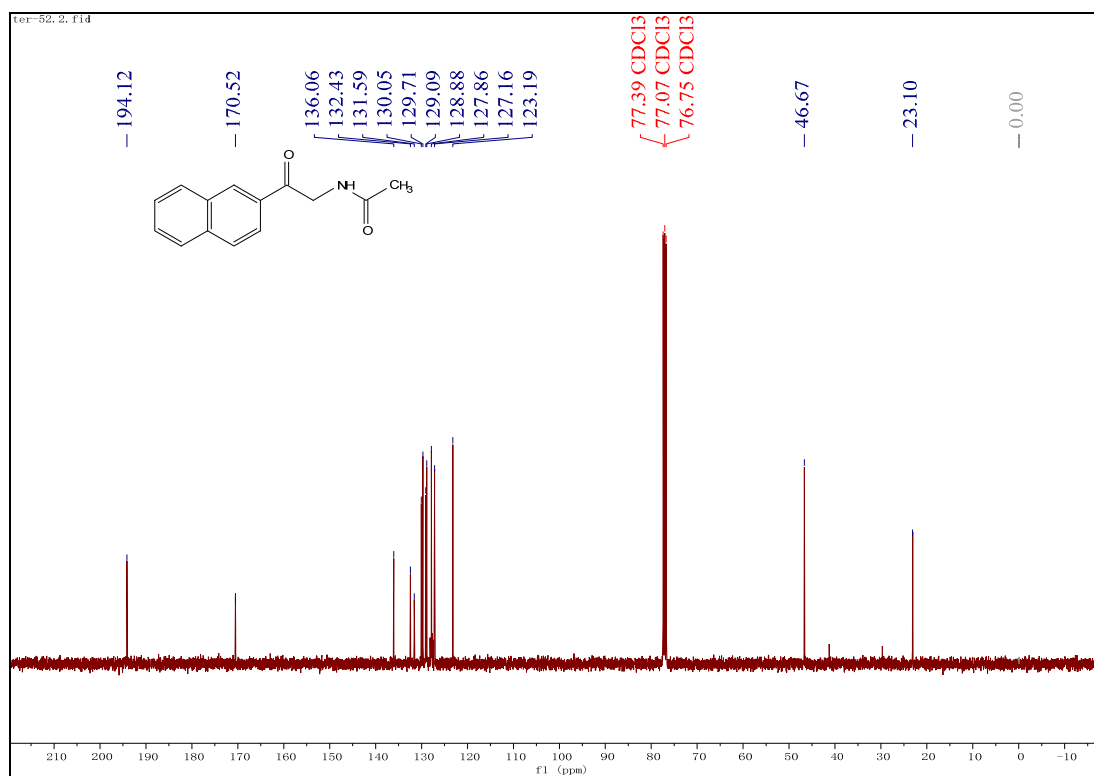
¹H NMR spectrum of **4i** (400 MHz, CDCl₃)



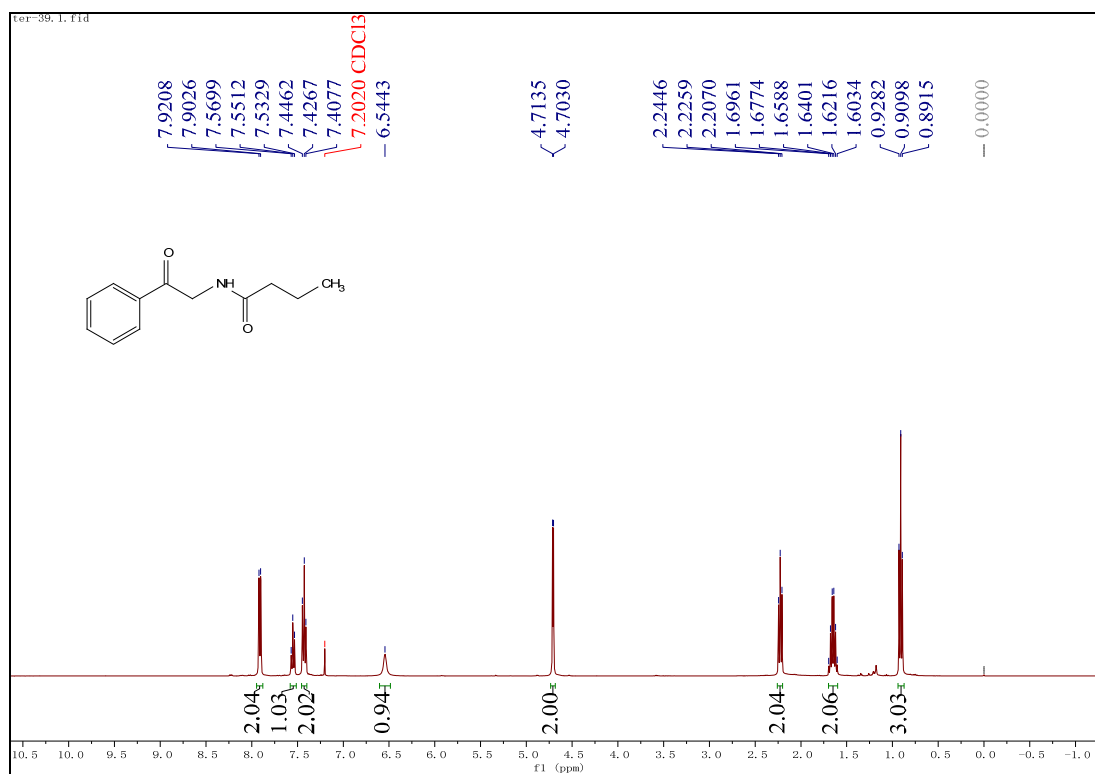
¹³C NMR spectrum of **4i** (100 MHz, CDCl₃)



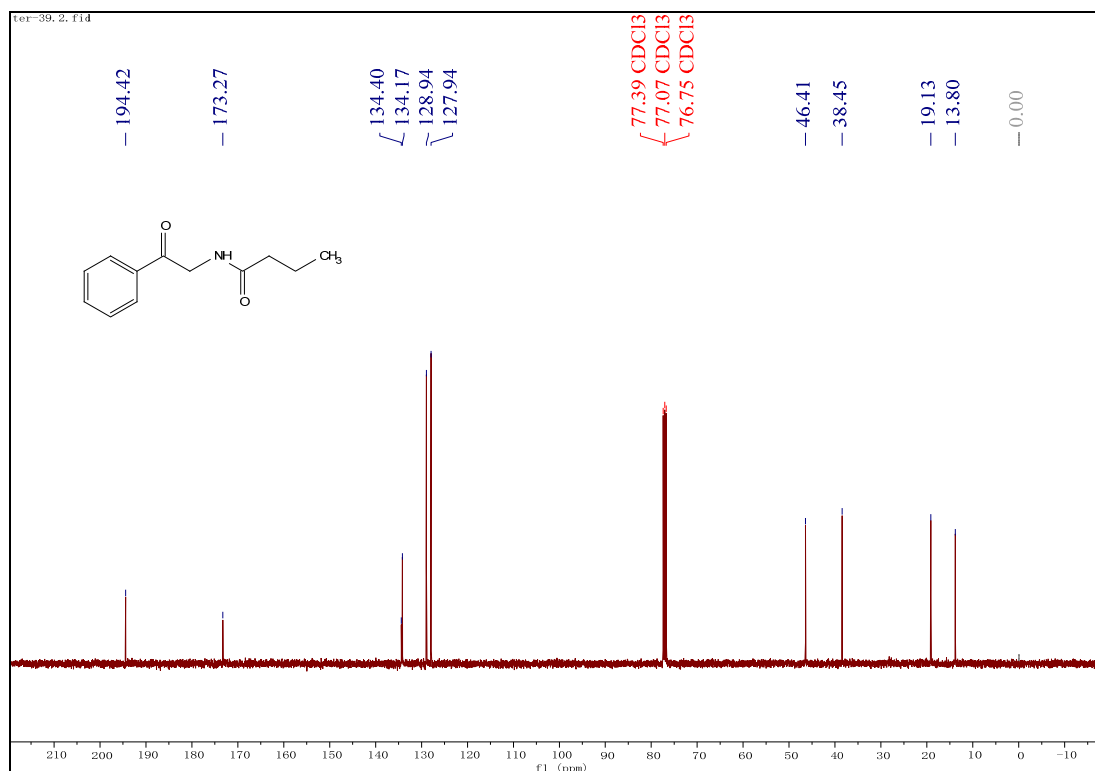
¹H NMR spectrum of **4j** (400 MHz, CDCl₃)



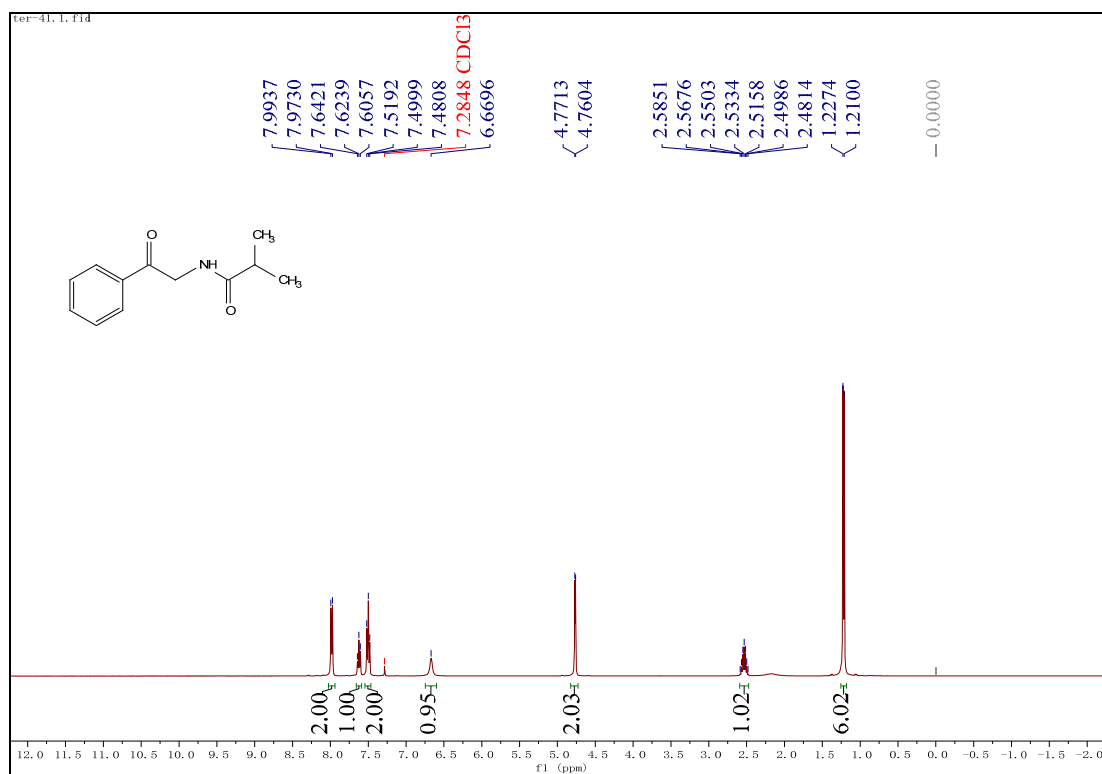
¹³C NMR spectrum of **4j** (100 MHz, CDCl₃)



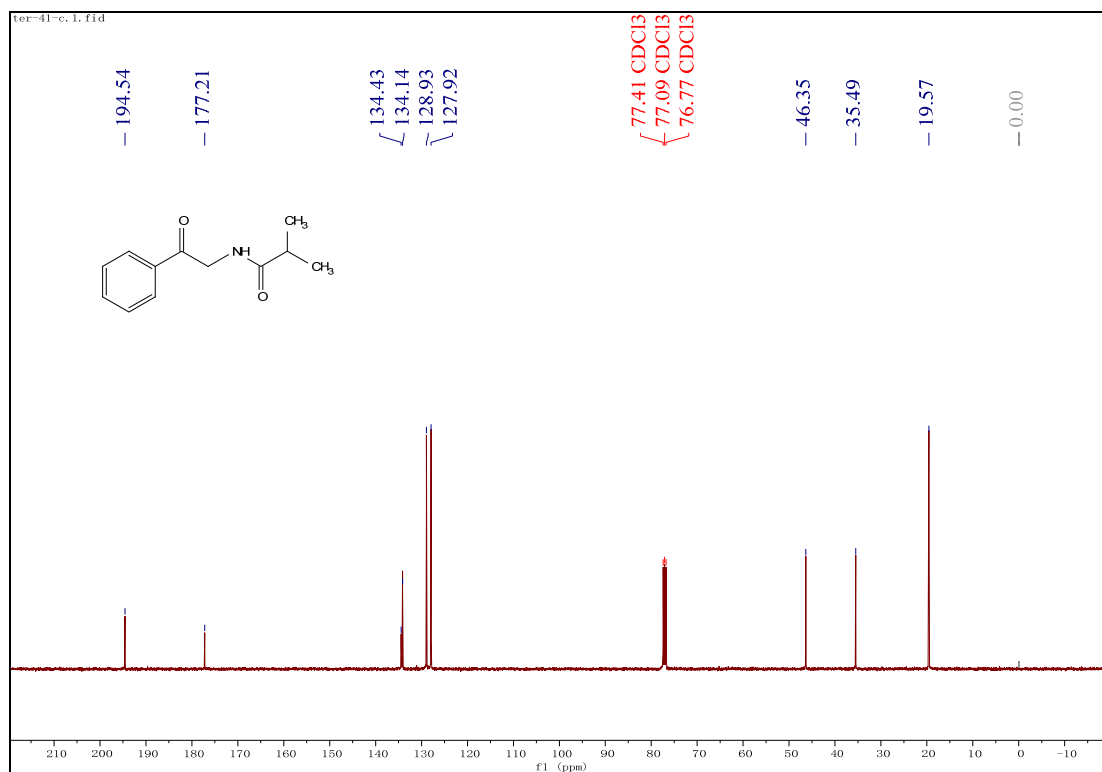
¹H NMR spectrum of **4k** (400 MHz, CDCl₃)



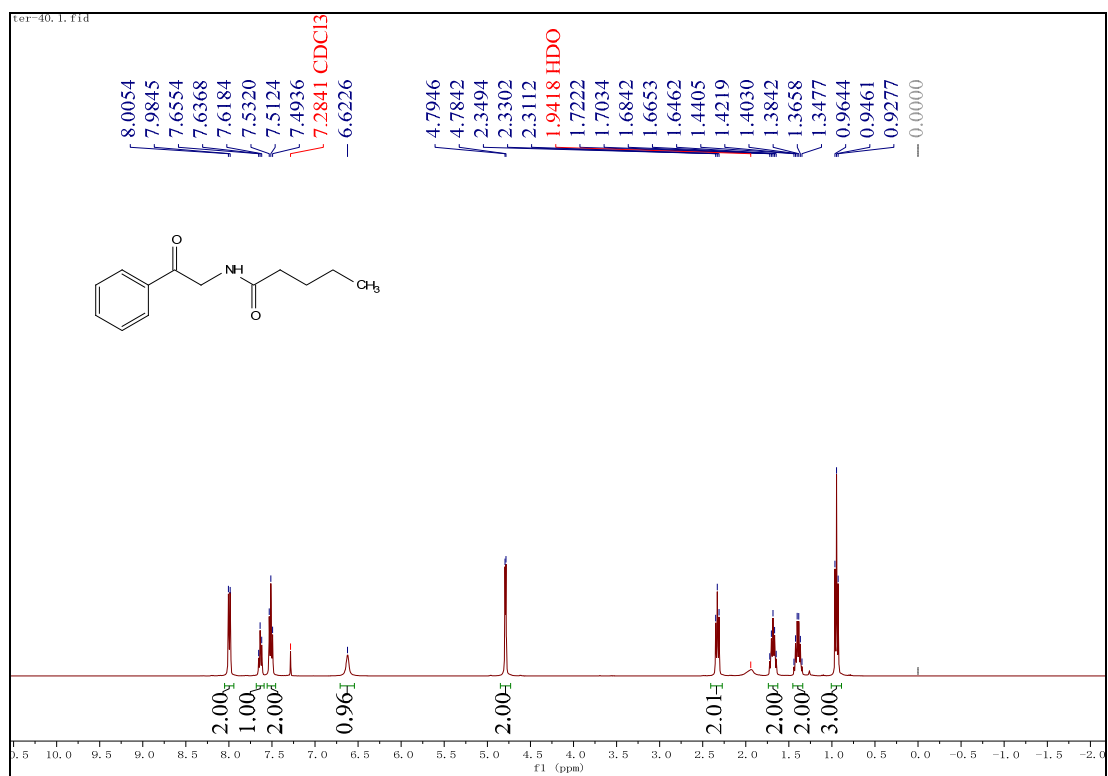
¹³C NMR spectrum of **4k** (100 MHz, CDCl₃)



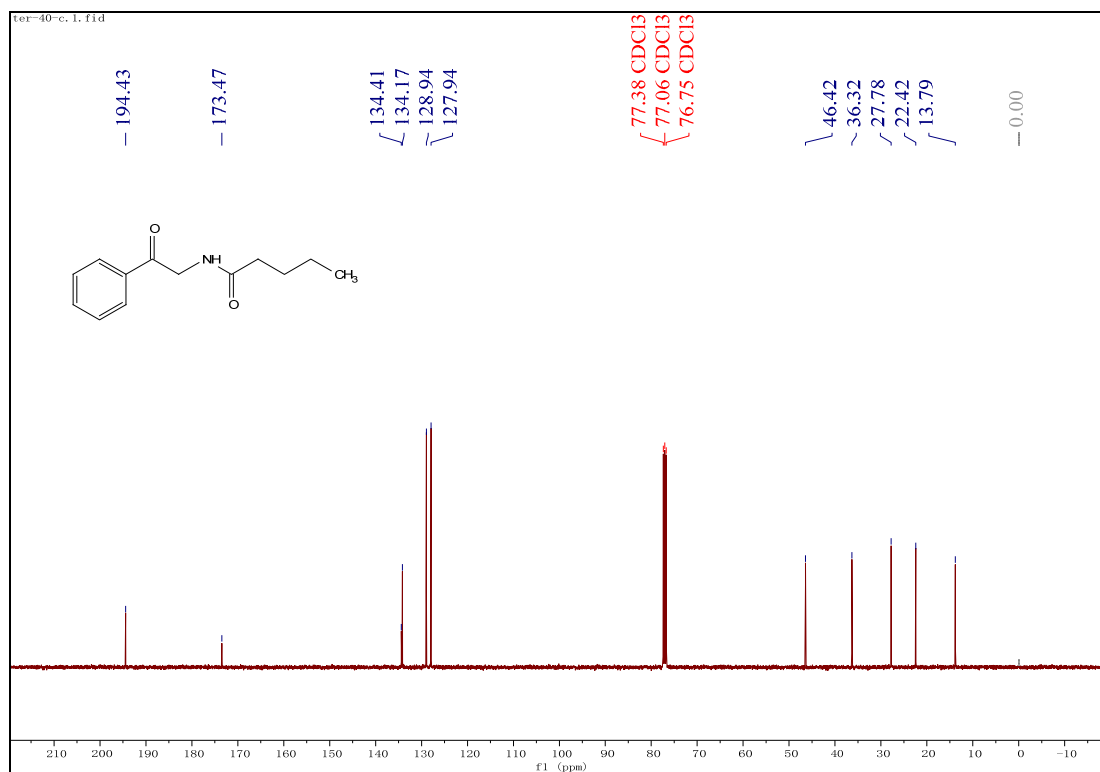
¹H NMR spectrum of **4I** (400 MHz, CDCl₃)



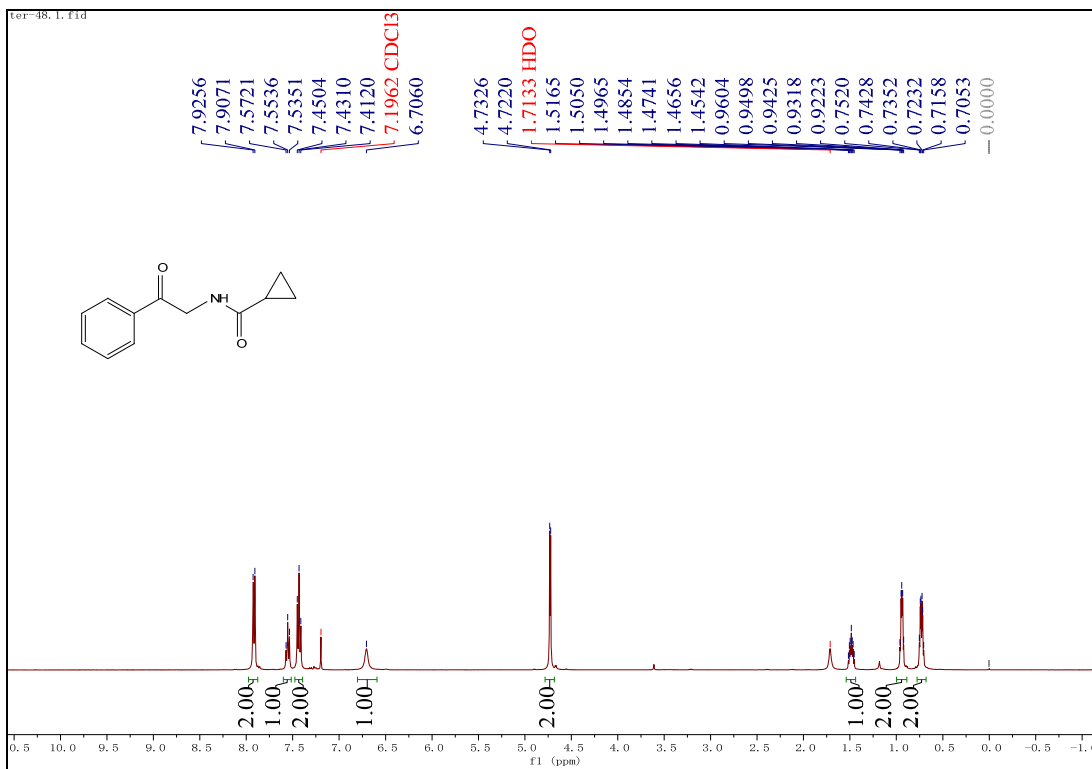
¹³C NMR spectrum of **4I** (100 MHz, CDCl₃)



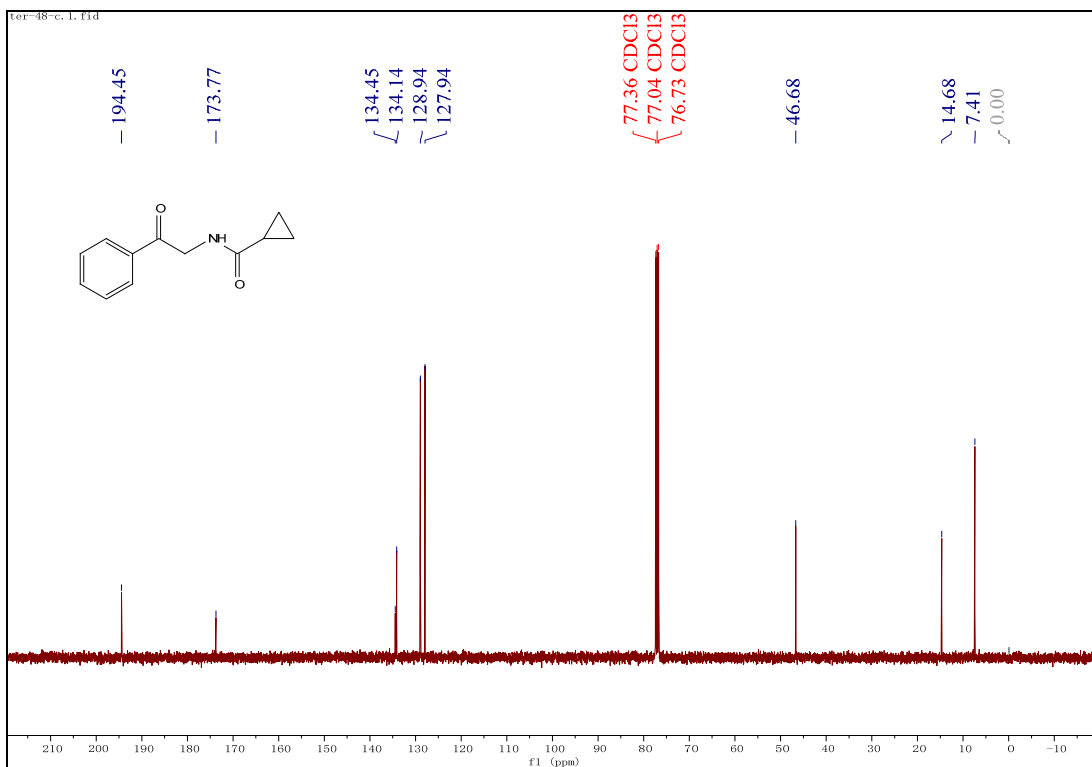
^1H NMR spectrum of **4m** (400 MHz, CDCl_3)



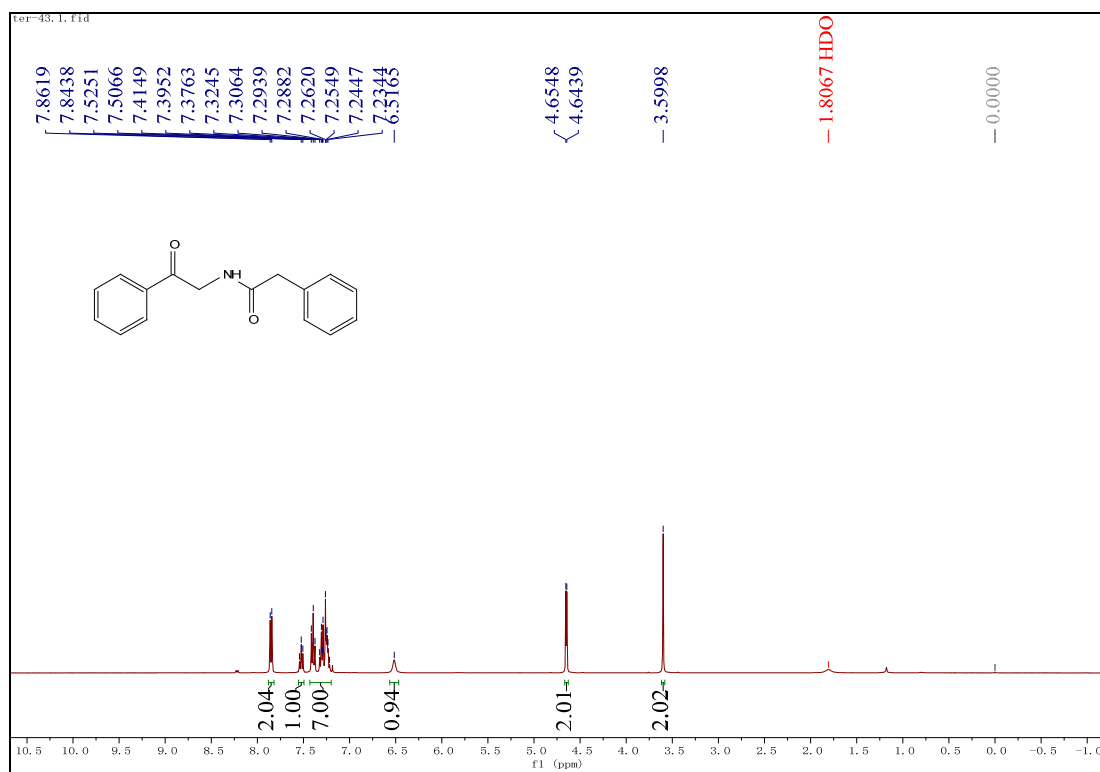
^{13}C NMR spectrum of **4m** (100 MHz, CDCl_3)



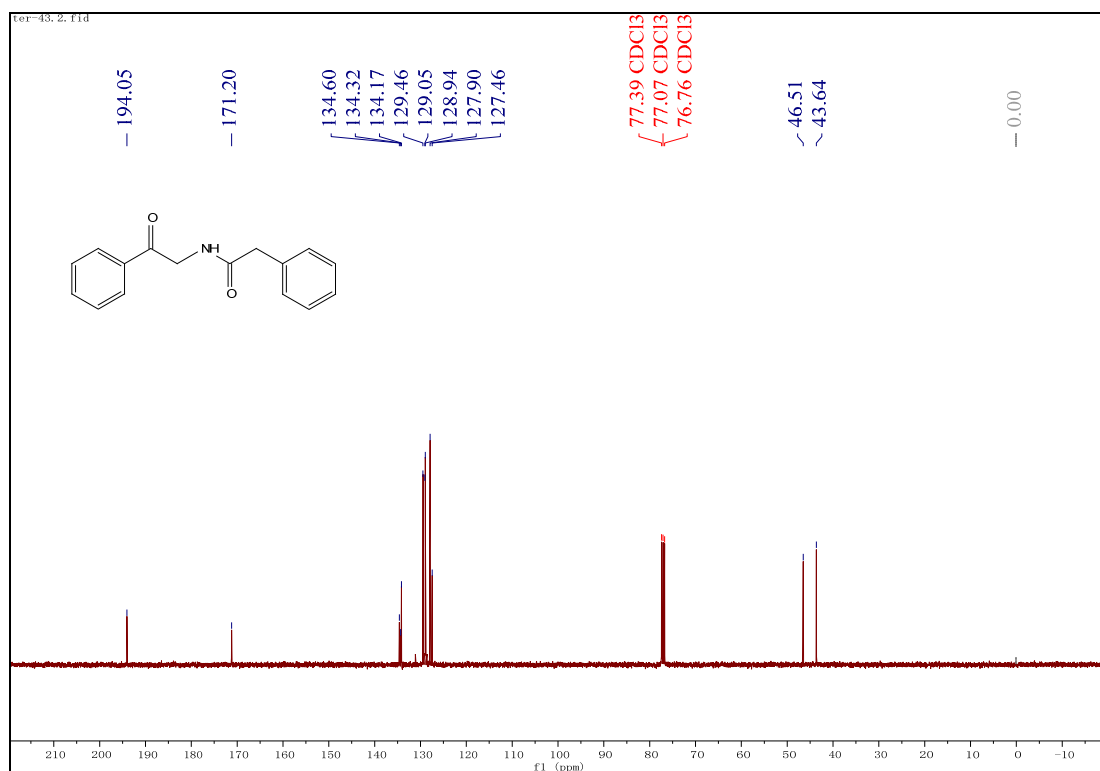
¹H NMR spectrum of **4n** (400 MHz, CDCl₃)



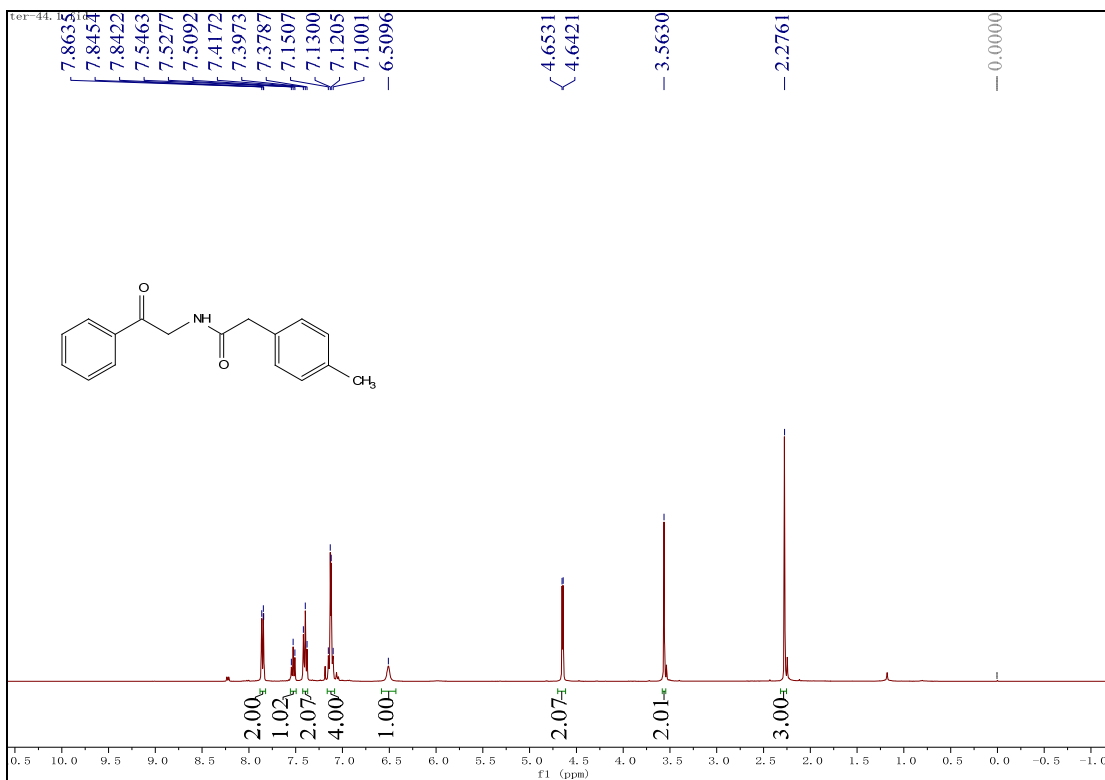
¹³C NMR spectrum of **4n** (100 MHz, CDCl₃)



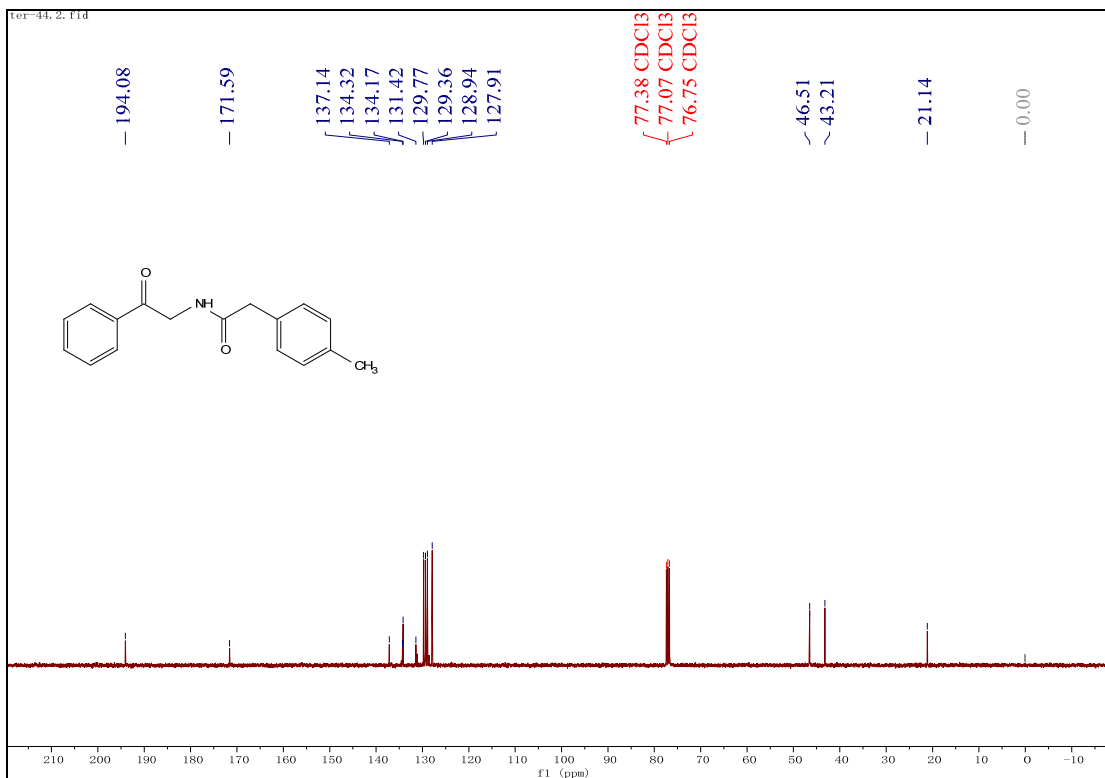
¹H NMR spectrum of **4o** (400 MHz, CDCl₃)



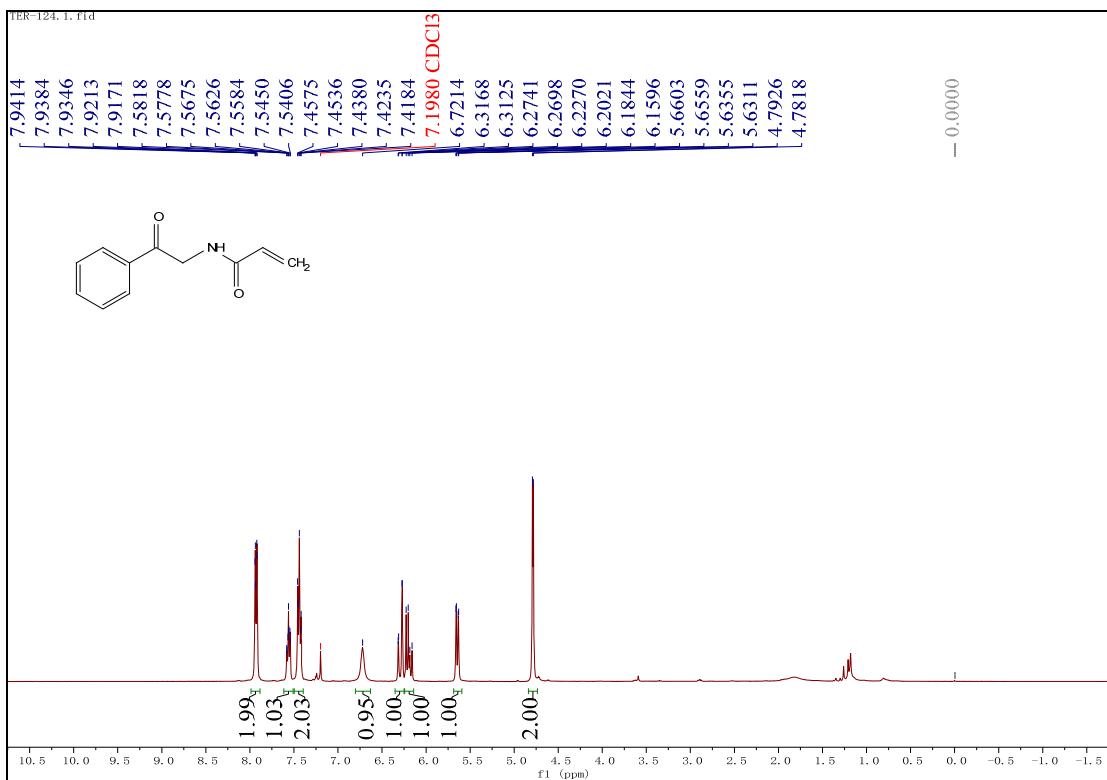
¹³C NMR spectrum of **4o** (100 MHz, CDCl₃)



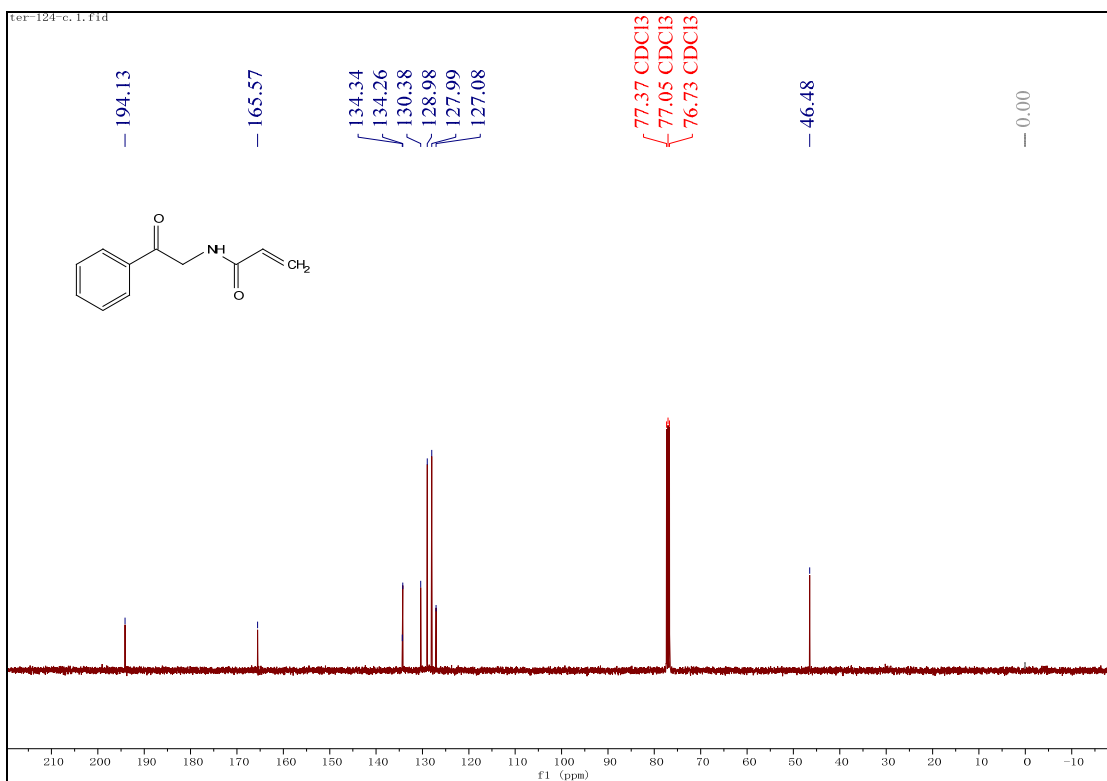
¹H NMR spectrum of **4p** (400 MHz, CDCl₃)



¹³C NMR spectrum of **4p** (100 MHz, CDCl₃)



¹H NMR spectrum of 4q (400 MHz, CDCl₃)



¹³C NMR spectrum of 4q (100 MHz, CDCl₃)