Supporting Information

Cobalt-Catalyzed Ozone and Hydrogen Peroxide Synergistic Oxidation of Benzylic C–H Bonds †

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† Electronic supplementary information (ESI) available

Content

1. General Remarks

Unless otherwise specified, dry solvents were used in all reactions. The chemicals were purchased from commercial suppliers and used without further purification. ^{1}H NMR spectra were recorded on a Bruke Avance operating at 400 or 600 MHz, ^{13}C NMR at 101 or 151 MHz, using TMS as an internal standard. The residual solvent signals were used as references for ^{1}H and ^{13}C NMR spectra and the chemical shifts were converted to the TMS scale ($CDCl_3$, 7.26 ppm for ^{1}H NMR and 77.16 ppm for ^{13}C NMR; DMSO-d_6, 2.50 ppm for ^{1}H NMR and 39.5 ppm for ^{13}C NMR). The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, dq, doublet of quartets, ddd = doublet of doublet of doublets, tdd = triplet of doublet of doublets, ddt, doublet of doublet of triplets, m = multiplet. The coupling constants *J* were given in Hertz. Column chromatography was performed using EM silica gel 60 (100−200 or 200−300 mesh). Ozone is generated by an ozone generator (220 V, 25 L/ min).

2. Optimization of reaction conditions

Time-course profile of 2-chloro-4-fluoro-1-methylbenzene. The reaction progress was detected by GC.

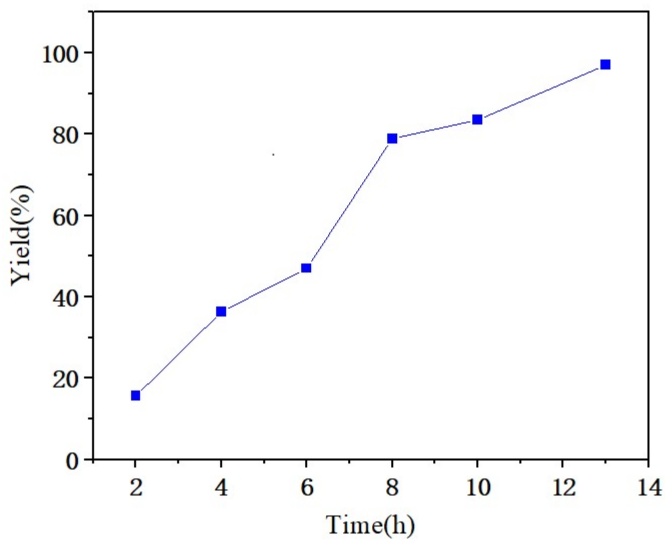
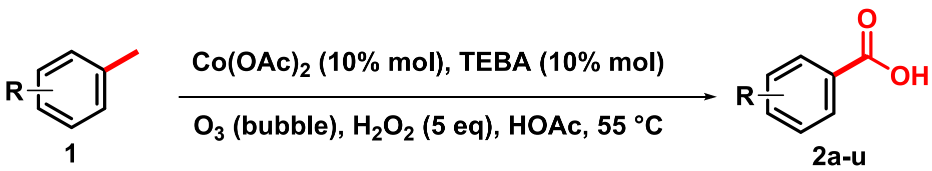


Figure S1 Time-course profile of **1a** under the optimal conditions

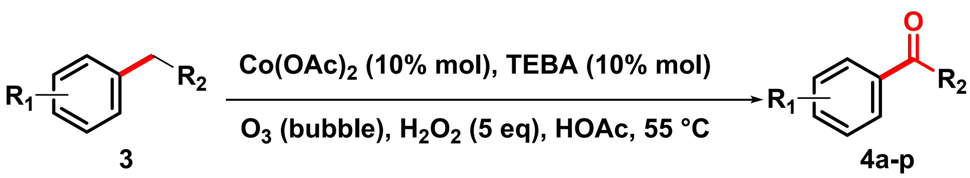
3. General procedure for the synthesis of carboxylic acids and ketones

3.1 General Procedure for the Synthesis of Aromatic Carboxylic Acids



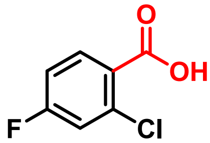
A 100 mL three-neck flask was charged with substrate (**1**, 2 mmol), $Co(OAc)_2$ (0.2 mmol, 0.1 equiv.), TEBA (0.2 mmol, 0.1 equiv.), and HOAc (15 mL). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 5 to 13 hours. Upon completion, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography, eluting with petroleum ether/EtOAc (5→20%, v/v) to afford the corresponding aromatic carboxylic acids (**2**).

3.2 General procedure for the synthesis of aromatic ketones



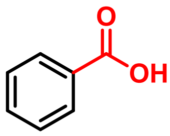
A 100 mL three-neck flask was charged with substrate (**3**, 2 mmol), $Co(OAc)_2$ (0.2 mmol, 0.1 equiv.), TEBA (0.2 mmol, 0.1 equiv.), and HOAc (15 mL). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 5 to 13 hours. Upon completion, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography, eluting with petroleum ether/EtOAc (5→20%, v/v) to afford the corresponding aromatic ketones (**4**).

2-chloro-4-fluorobenzoic acid (2a)[1]



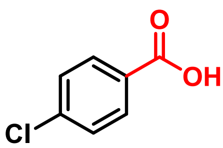
2a was synthesized according to the general procedure **A** with **1a**. Off-white solid. (**2a**: 340 mg, 97% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO-*d*$_6$)** δ 12.86 (brs, 1H), 7.88 (dd, *J* = 8.8, 6.3 Hz, 1H), 7.48 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.27 (td, *J* = 8.4, 2.6 Hz, 1H). **^{13}C NMR (101 MHz, DMSO-*d*$_6$)** δ 165.77, 163.16 (d, *J* = 252.7 Hz), 133.66 (d, *J* = 11.1 Hz), 133.29 (d, *J* = 9.9 Hz), 127.76, 118.14 (d, *J* = 25.2 Hz), 114.61 (d, *J* = 21.5 Hz).

benzoic acid (2b)[2]



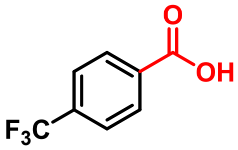
2b was synthesized according to the general procedure **A** with **1b**. White solid, (**2b**: 222 mg, 91% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, DMSO-*d*$_6$)** δ 12.96 (s, 1H), 8.08 – 7.85 (m, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.8 Hz, 2H). **^{13}C NMR (151 MHz, DMSO-*d*$_6$)** δ 167.49, 132.91, 130.92, 129.41(2C), 128.63(2C).

4-chlorobenzoic acid (2c)[3]



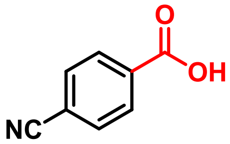
2c was synthesized according to the general procedure **A** with **1c**. White powder, (**2c**: 285 mg, 91% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, DMSO-*d*$_6$)** δ 13.18 (brs, 1H), 7.93 (d, *J* = 8.1 Hz, 2H), 7.53 (d, *J* = 8.1 Hz, 2H). **^{13}C NMR (151 MHz, DMSO-*d*$_6$)** δ 166.49, 137.83, 131.17(2C), 129.69, 128.75(2C).

4-(trifluoromethyl)benzoic acid (2d)[2]



2d was synthesized according to the general procedure **A** with **1d**. White powder, (**2d**: 365 mg, 96% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO)** δ 13.42 (brs, 1H), 8.11 (d, *J* = 8.1 Hz, 2H), 7.80 (d, *J* = 8.1 Hz, 2H). **^{13}C NMR (151 MHz, DMSO-*d$_6$*)** δ 166.32, 134.70, 132.65 (q, *J* = 32.0 Hz), 130.16(2C), 125.59 (d, *J* = 3.5 Hz), 125.54 (d, *J* = 3.8 Hz)., 123.88 (q, *J* = 272.6 Hz).

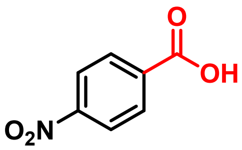
4-cyanobenzoic acid (2e)[3]



2e was synthesized according to the general procedure **A** with **1e**. White powder, (**2e**: 149 mg, 61% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO)** δ 13.35 (brs, 1H), 8.07 – 8.03 (m, 4H), 4.33 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).

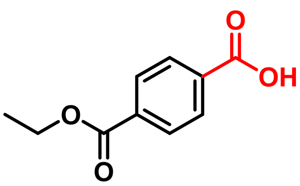
^{13}C NMR (151 MHz, DMSO-*d$_6$*) δ 166.10, 134.88, 132.69(2C), 129.96(2C), 118.22, 115.11.

4-nitrobenzoic acid (2f)[3]



2f was synthesized according to the general procedure **A** with **1f**. White powder, (**2f**: 134 mg, 40% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO-*d$_6$*)** δ 13.59 (brs, 1H), 8.29 – 8.22 (m, 2H), 8.15 – 8.07 (m, 2H). **^{13}C NMR (151 MHz, DMSO-*d$_6$*)** δ 165.87, 150.05, 136.45, 130.73(2C), 123.71(2C).

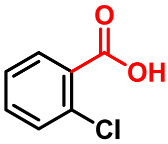
4-(ethoxycarbonyl)benzoic acid (2g)[4]



2g was synthesized according to the general procedure **A** with **1g**. White solid, (**2g**: 179 mg, 46% yield), flash chromatography conditions: petroleum ether/EtOAc (5 –

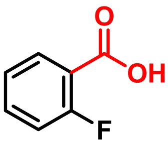
10%, v/v). ^{1}H NMR (600 MHz, DMSO-d_6) δ 13.32 (brs, 1H), 8.05 (d, J = 1.3 Hz, 4H), 4.35 (d, J = 7.1 Hz, 1H), 4.32 (d, J = 7.1 Hz, 1H), 1.33 (t, J = 7.1 Hz, 3H). **^{13}C NMR (151 MHz, DMSO-d_6)** δ 166.60, 165.12, 134.77, 133.46, 129.60(2C), 129.32(2C), 62.03, 13.41.

2-chlorobenzoic acid (2h)[1]



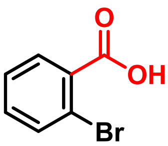
2h was synthesized according to the general procedure **A** with **1h**. White solid, (**2h**: 297 mg, 95% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). ^{1}H NMR (400 MHz, DMSO) δ 13.38 (s, 1H), 7.80 – 7.75 (m, 1H), 7.57 – 7.50 (m, 2H), 7.46 – 7.39 (m, 1H). **^{13}C NMR (101 MHz, DMSO)** δ 167.23, 133.09, 132.06, 131.98, 131.30, 131.12, 127.74.

2-fluorobenzoic acid (2i)[1]



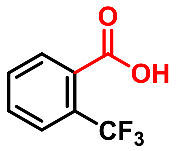
2i was synthesized according to the general procedure **A** with **1i**. White powder, (**2i**: 252 mg, 90% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (400 MHz, DMSO)** δ 13.24 (s, 1H), 7.90 – 7.83 (m, 1H), 7.67 – 7.60 (m, 1H), 7.34 – 7.26 (m, 2H). **^{13}C NMR (101 MHz, DMSO-d_6)** δ 165.07, 161.12 (d, J = 256.7 Hz), 134.72 (d, J = 9.0 Hz), 131.91, 124.46 (d, J = 3.8 Hz), 119.33 (d, J = 10.3 Hz), 116.93 (d, J = 22.2 Hz).

2-bromobenzoic acid (2j)[1]



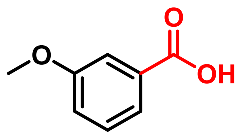
2j was synthesized according to the general procedure **A** with **1j**. White solid, (**2j**: 378 mg, 94% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (400 MHz, DMSO-d_6)** δ 13.40 (s, 1H), 7.77 – 7.72 (m, 1H), 7.71 (d, J = 6.8 Hz, 1H), 7.52 – 7.44 (m, 1H), 7.44 – 7.38 (m, 1H). **^{13}C NMR (101 MHz, DMSO-d_6)** δ 167.38, 133.76(2C), 132.53, 130.59, 127.72, 119.94.

2-(trifluoromethyl)benzoic acid (2k)[1]



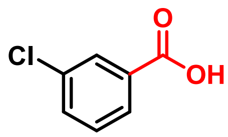
2k was synthesized according to the general procedure **A** with **1k**. White powder, (**2k**: 262 mg, 69% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 13.57 (s, 1H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.79 (d, *J* = 8.4 Hz, 1H), 7.75 (d, *J* = 8.9 Hz, 1H). **^{13}C NMR (151 MHz, DMSO-*d*$_6$)** δ 168.30, 133.06, 132.85 (q, *J* = 2.3 Hz), 131.60, 130.13, 126.99 (q, *J* = 31.6 Hz), 126.92 (q, *J* = 5.4 Hz), 124.02 (q, *J* = 273.3 Hz).

3-methoxybenzoic acid (2l)[1]



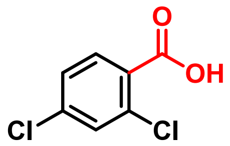
2l was synthesized according to the general procedure **A** with **1l**. White powder, (**2l**: 119 mg, 39% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, DMSO-*d*$_6$)** δ 12.97 (brs, 1H), 7.53 (d, *J* = 7.7 Hz, 1H), 7.44 (s, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.17 (dd, *J* = 8.7, 2.3 Hz, 1H), 3.79 (s, 3H). **^{13}C NMR (151 MHz, DMSO)** δ 167.26, 159.32, 132.30, 129.76, 121.65, 118.93, 114.01, 55.29.

3-chlorobenzoic acid (2m)[2]



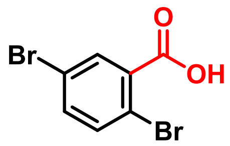
2m was synthesized according to the general procedure **A** with **1m**. White powder, (**2m**: 301 mg, 96% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, DMSO-*d*$_6$)** δ 13.23 (brs, 1H), 7.88 – 7.86 (m, 2H), 7.64 – 7.62 (m, 1H), 7.50 – 7.47 (m, 1H). **^{13}C NMR (151 MHz, DMSO)** δ 166.52, 133.80, 133.35, 133.02, 130.90, 129.31, 128.30.

2,4-dichlorobenzoic acid (2n)[1]



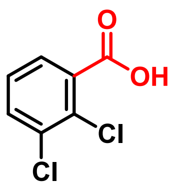
2n was synthesized according to the general procedure **A** with **1n**. White powder, (**2n**: 367 mg, 96% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO-d_6)** δ 13.45 (brs, 1H), 7.86 (d, J = 8.4 Hz, 1H), 7.71 (d, J = 2.1 Hz, 1H), 7.53 (dd, J = 8.4, 2.1 Hz, 1H). **^{13}C NMR (151 MHz, DMSO)** δ 165.88, 136.63, 133.18, 132.47, 130.26, 130.12, 127.52.

2,5-dibromobenzoic acid (2o)[5]



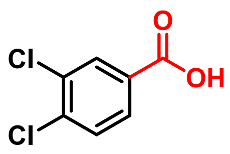
2o was synthesized according to the general procedure **A** with **1o**. White powder, (**2o**: 482 mg, 86% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO-d_6)** δ 13.72 (brs, 1H), 7.88 (d, J = 2.4 Hz, 1H), 7.65 (d, J = 8.5 Hz, 1H), 7.62 (dd, J = 8.5, 2.4 Hz, 1H). **^{13}C NMR (151 MHz, DMSO)** δ 166.06, 135.75, 135.70, 135.15, 132.88, 120.62, 119.04.

2,3-dichlorobenzoic acid(2p)[6]



2p was synthesized according to the general procedure **A** with **1p**. White powder, (**2p**: 344 mg, 90% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (400 MHz, DMSO-d_6)** δ 13.70 (s, 1H), 7.79 (dd, J = 8.0, 1.6 Hz, 1H), 7.70 (dd, J = 7.8, 1.6 Hz, 1H), 7.45 (t, J = 7.9 Hz, 1H). **^{13}C NMR (101 MHz, DMSO-d_6)** δ 166.39, 134.55, 132.85, 132.69, 129.15, 128.81, 128.47.

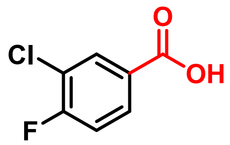
3,4-dichlorobenzoic acid(2q)[3]



2q was synthesized according to the general procedure **A** with **1q**. White powder, (**2q**: 351 mg, 92% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600

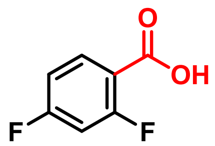
MHz, DMSO) δ 13.02 (brs, 1H), 8.03 – 7.98 (m, 1H), 7.87 – 7.82 (m, 1H), 7.73 – 7.67 (m, 1H). **^{13}C NMR (151 MHz, DMSO-d_6)** δ 165.50, 135.77, 131.57, 131.55, 130.98(2C), 129.29.

3-chloro-4-fluorobenzoic acid(2r)[7]



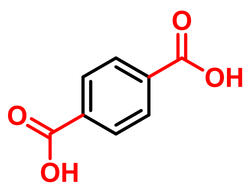
2r was synthesized according to the general procedure **A** with **1r**. White powder, (**2r**: 332 mg, 95% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (400 MHz, DMSO-d_6)** δ 13.38 (s, 1H), 8.05 (d, J = 7.3 Hz, 1H), 7.98 – 7.90 (m, 1H), 7.54 (t, J = 8.9 Hz, 1H). **^{13}C NMR (101 MHz, DMSO-d_6)** δ 165.86, 160.48 (d, J = 252.8 Hz), 132.15, 131.04 (d, J = 8.7 Hz), 129.18 (d, J = 3.5 Hz), 120.49 (d, J = 18.2 Hz), 117.81 (d, J = 21.3 Hz).

2,4-difluorobenzoic acid(2s)[8]



2s was synthesized according to the general procedure **A** with **1s**. White solid, (**2s**: 300 mg, 95% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, DMSO-d_6)** δ 12.65 (brs, 1H), 7.93 (q, J = 8.1 Hz, 1H), 7.27 (t, J = 10.3 Hz, 1H), 7.13 (t, J = 9.7 Hz, 1H). **^{13}C NMR (151 MHz, DMSO-d_6)** δ 165.01 (dd, J = 253.2, 12.2 Hz), 164.36 (d, J = 3.4 Hz), 162.28 (dd, J = 260.3, 13.0 Hz), 134.17 (dd, J = 11.0, 2.5 Hz), 116.19 (dd, J = 10.0, 3.6 Hz), 111.93 (dd, J = 21.4, 3.7 Hz), 105.38 (t, J = 26.1 Hz).

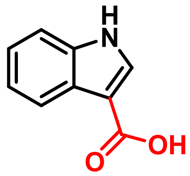
terephthalic acid(2t)[6]



2t was synthesized according to the general procedure **A** with **1t**. White solid, (**2t**: 186 mg, 56% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR**

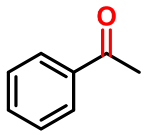
(600 MHz, DMSO-d_6) δ 13.21 (brs, 2H), 8.01 (s, 4H). **^{13}C NMR (151 MHz, DMSO-d_6)** δ 167.10(2C), 134.79(2C), 129.79(4C).

1H-indole-3-carboxylic acid(2u)[9]



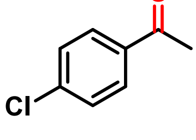
2u was synthesized according to the general procedure **A** with **1u**. Off-white solid. (**2u**: 58 mg, 18% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, DMSO-d_6)** δ 11.80 (s, 1H), 8.11 – 7.87 (m, 2H), 7.46 (d, J = 8.7 Hz, 1H), 7.26 – 7.00 (m, 2H). **^{13}C NMR (151 MHz, DMSO-d_6)** δ 166.44, 136.89, 132.71, 126.48, 122.58, 121.42, 121.05, 112.65, 107.88.

Acetophenone(4a)[10]



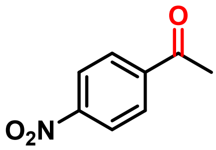
4a was synthesized according to the general procedure **B** with **3a**. Colorless liquid. (**4a**: 216 mg, 90% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-d)** δ 7.93 (d, J = 6.9 Hz, 2H), 7.53 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.8 Hz, 2H), 2.57 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-d)** δ 198.13, 137.12, 133.11, 128.57(2C), 128.30(2C), 26.59.

1-(4-chlorophenyl)ethan-1-one(4b)[10]



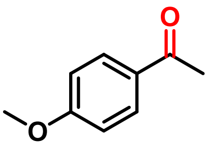
4b was synthesized according to the general procedure **B** with **3b**. Pale yellow liquid. (**4b**: 266 mg, 86% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-d)** δ 7.85 (d, J = 8.6 Hz, 2H), 7.39 (d, J = 8.6 Hz, 2H), 2.55 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-d)** δ 196.84, 139.57, 135.47, 129.77(2C), 128.91(2C), 26.42.

1-(4-nitrophenyl)ethan-1-one(4c)[8]



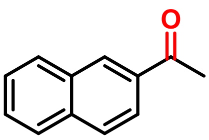
4c was synthesized according to the general procedure **B** with **3c**. Yellow crystal. (**4c**: 244 mg, 74% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 8.32 – 8.26 (m, 2H), 8.12 – 8.07 (m, 2H), 2.67 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 196.43, 150.45, 141.48, 129.41(2C), 123.95(2C), 27.07.

1-(4-methoxyphenyl)ethan-1-one(4d)[10]



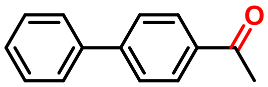
4d was synthesized according to the general procedure **B** with **3d**. Colorless crystal. (**4d**: 114 mg, 38% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 8.02 – 7.81 (m, 2H), 7.04 – 6.81 (m, 2H), 3.86 (s, 3H), 2.54 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 196.90, 163.59, 130.69(2C), 130.45, 113.79(2C), 55.57, 26.44.

1-(naphthalen-2-yl)ethan-1-one(4e)[8]



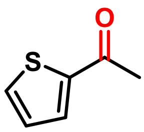
4e was synthesized according to the general procedure B with **3e**. White solid. (**4e**: 133 mg, 39% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-d)** δ 8.41 (s, 1H), 8.01 (d, J = 10.4 Hz, 1H), 7.92 (d, J = 8.2 Hz, 1H), 7.83 (dd, J = 8.5, 4.7 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 7.52 (t, J = 7.5 Hz, 1H), 2.68 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-d)** δ 198.04, 135.58, 134.48, 132.52, 130.20, 129.57, 128.48, 128.41, 127.79, 126.78, 123.88, 26.66.

1-([1,1'-biphenyl]-4-yl)ethan-1-one(4f)[8]



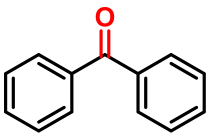
4f was synthesized according to the general procedure **B** with **3f**. White powder. (**4f**: 290 mg, 74% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-d)** δ 8.03 (s, 2H), 7.71 – 7.60 (m, 4H), 7.51 – 7.38 (m, 3H), 2.64 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-d)** δ 197.88, 145.89, 139.98, 135.97, 129.07(2C), 129.03(2C), 128.35, 127.38(2C), 127.34(2C), 26.77.

1-(thiophen-2-yl)ethan-1-one(4g)[11]



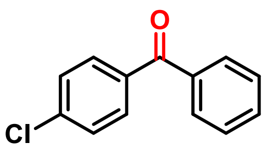
4g was synthesized according to the general procedure **B** with **3g**. Colorless liquid. (**4g**: 91 mg, 36% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.57 (dd, *J* = 3.8, 1.2 Hz, 1H), 7.51 (dd, *J* = 5.0, 1.2 Hz, 1H), 6.99 (dd, *J* = 5.0, 3.8 Hz, 1H), 2.42 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 190.67, 144.44, 133.82, 132.63, 128.19, 26.79.

Benzophenone(4h)[8]



4h was synthesized according to the general procedure **B** with **3h**. White crystals. (**4h**: 244 mg, 80% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.78 (dd, *J* = 8.4, 1.5 Hz, 4H), 7.56 – 7.51 (m, 2H), 7.47 – 7.39 (m, 4H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 196.43, 137.45(2C), 132.29(2C), 129.89(4C), 128.16(4C).

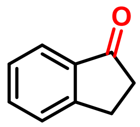
(4-chlorophenyl)(phenyl)methanone(4i)[12]



4i was synthesized according to the general procedure **B** with **3i.** White powder. (**4i**: 351 mg, 81% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR**

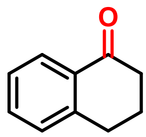
(600 MHz, Chloroform-*d*) δ 7.80 – 7.69 (m, 4H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.50 – 7.39 (m, 4H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 195.41, 138.88, 137.25, 135.89, 132.67, 131.48(2C), 129.94(2C), 128.65(2C), 128.43(2C).

2,3-dihydro-1H-inden-1-one(4j)[13]



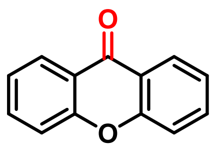
4j was synthesized according to the general procedure **B** with **3j**. Yellow-brown crystals. (**4j**: 219 mg, 83% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.78 (d, *J* = 7.7 Hz, 1H), 7.62 (td, *J* = 7.4, 1.3 Hz, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.42 – 7.37 (m, 1H), 3.20 – 3.12 (m, 2H), 2.74 – 2.65 (m, 2H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 206.79, 155.02, 136.89, 134.43, 127.09, 126.59, 123.43, 36.04, 25.64.

1-tetralone(4k)[8]



4k was synthesized according to the general procedure **B** with **3k**. Pale yellow liquid. (**4k**: 170 mg, 58% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 8.01 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.44 (td, *J* = 7.5, 1.5 Hz, 1H), 7.33 – 7.15 (m, 2H), 2.94 (t, *J* = 6.2 Hz, 2H), 2.74 – 2.48 (m, 2H), 2.12 (p, *J* = 6.6 Hz, 2H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 198.41, 144.54, 133.43, 132.65, 128.83, 127.18, 126.66, 39.21, 29.74, 23.33.

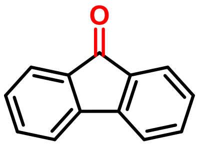
Xanthen-9-one(4l)[13]



4l was synthesized according to the general procedure **B** with **3l**. White powder. (**4l**: 365 mg, 93% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 8.30 (s, 1H), 8.29 (s, 1H), 7.72 – 7.62 (m, 2H), 7.43 (d, *J* = 8.5

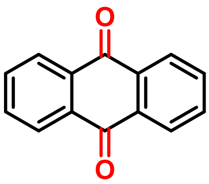
Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 177.14, 156.11(2C), 134.78(2C), 126.67(2C), 123.87(2C), 121.80(2C), 117.95(2C).

Fluoren-9-one(4m)[13]



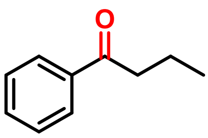
4m was synthesized according to the general procedure **B** with **3m**. Yellow crystals. (**4m**: 296 mg, 82% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.57 – 7.53 (m, 1H), 7.38 – 7.33 (m, 1H), 7.21 – 7.16 (m, 1H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 193.69, 144.25(2C), 134.55(2C), 133.96(2C), 128.92(2C), 124.06(2C), 120.21(2C).

Anthraquinone(4n)[13]



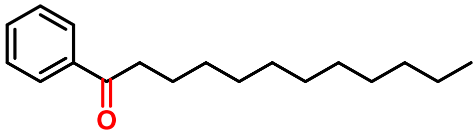
4n was synthesized according to the general procedure **B** with **3n**. Orange-yellow powder. (**4n**: 408 mg, 98% yield), flash chromatography conditions: petroleum ether/EtOAc (10 – 20%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 8.32 (dd, *J* = 5.8, 3.3 Hz, 4H), 7.81 (dd, *J* = 5.8, 3.3 Hz, 4H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 183.32(2C), 134.28(4C), 133.67(4C), 127.39(4C).

1-phenylbutan-1-one(4o)[14]



4o was synthesized according to the general procedure **B** with **3o**. Pale yellow liquid. (**4o**: 234 mg, 79% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.95 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.56 – 7.50 (m, 1H), 7.47 – 7.41 (m, 2H), 2.93 (t, *J* = 7.3 Hz, 2H), 1.76 (h, *J* = 7.3 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 200.46, 136.97, 132.92, 128.60(2C), 128.09(2C), 39.71, 17.71, 13.41.

1-phenyldodecan-1-one(4p)[15]



4p was synthesized according to the general procedure **B** with **3p**. White solid. (**4p**: 203 mg, 39% yield), flash chromatography conditions: petroleum ether/EtOAc (5 – 10%, v/v). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.95 (d, *J* = 6.5 Hz, 2H), 7.61 – 7.32 (m, 3H), 2.95 (t, *J* = 7.4 Hz, 2H), 1.73 (p, *J* = 7.5 Hz, 2H), 1.44 – 1.13 (m, 16H), 0.88 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 200.71, 137.23, 132.95, 128.65(2C), 128.17(2C), 38.75, 32.03, 29.75, 29.74, 29.64, 29.61, 29.51, 29.46, 24.51, 22.81, 14.23.

4. Mechanism Exploration Experiment

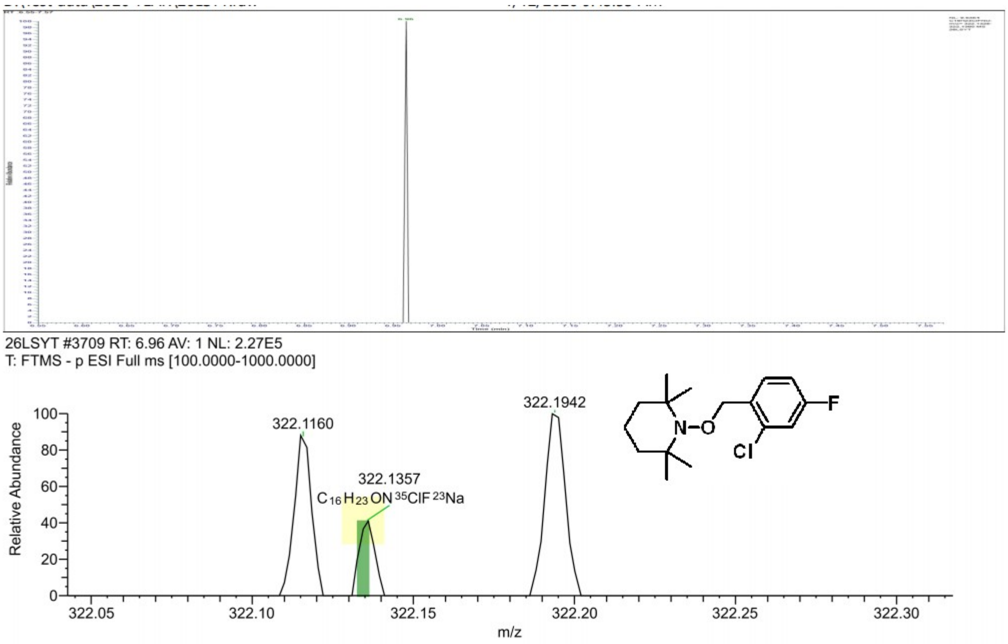
4.1 Radical capture experiment

1a 2a

Entry	Inhibitors	Species	Yield **2a** (%)
1	TEMPO	Radical	0
2	BHT	Radical	0
3	DABCO	$^1O_2$	63
4	Benzoquinone	$O_2^{\bullet}$	0
5	isopropanol	•OH	0

A 100 mL three-neck flask was charged with substrate (**1a**, 2 mmol), $Co(OAc)_2$ (0.2 mmol, 0.1 equiv.), TEBA (0.2 mmol, 0.1 equiv.), Inhibitors (3 equiv.) and HOAc (15 mL). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 13 hours. The reaction was analyzed by GC and HRMS.

When the radical trapping agent 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) or 2,6-di-tert-butyl-4-methylphenol (BHT) was added to the reaction system, the conversion of the reaction was completely inhibited; meanwhile, the benzylic radicals were captured by TEMPO and BHT in HRMS analysis (intermediates **5** and **6** were detected by HRMS), suggesting that the reaction proceeded via a radical mechanism. Moreover, the addition of the singlet oxygen inhibitor DABCO, the superoxide radical inhibitor benzoquinone and the hydroxyl radical inhibitor isopropanol had a significant negative impact on the reaction, indicating that reactive oxygen species, including singlet oxygen, superoxide radical and hydroxyl radical might be involved in the oxidation transformation.



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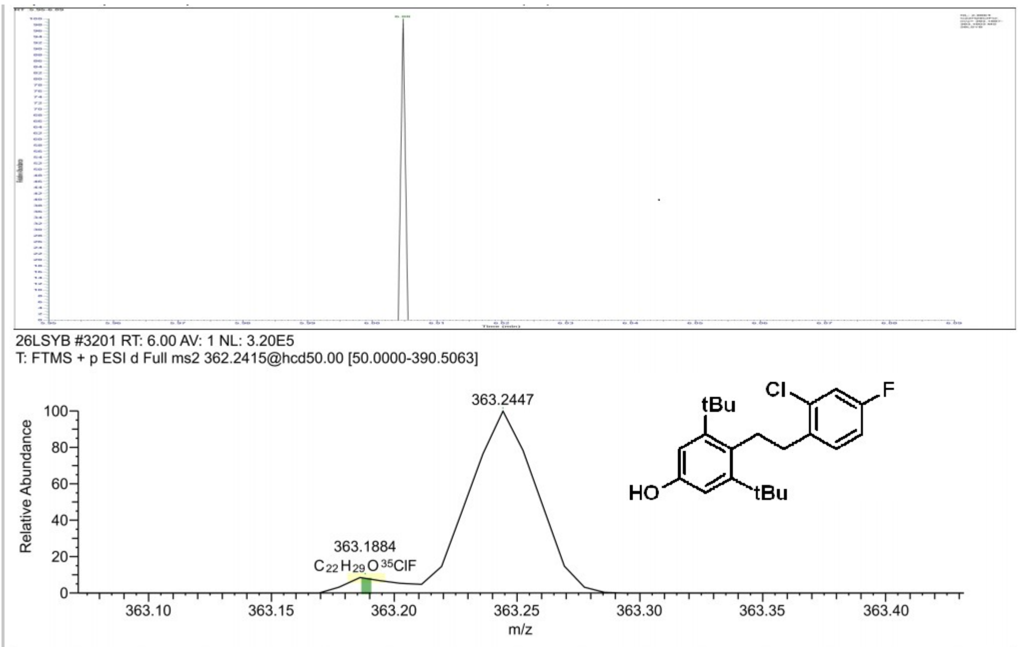
True	D:\Test data \2026 YEAR \26LSYT.raw	MS		Mass Range	MDF Ranges	322.1344	None	C16H23ClFNO	5	0	False				
False					MDF Ranges				NaN	0	False				

Peak Mass	Display F...	S Fit	RDB	Delta [pp...	Theo. ma...	Rank	Combine...	# Match...	# Missed...	MS Cov....	Pattern C...	MSMS M...
322.1357	$C_{16}H_{23}ON^{35}ClF^{23}Na$	44.237391571648	4.50	3.94	322.13444	1	97.07	1	0	100	100	(Collection)

Figure S2 HRMS spectrum of the intermediate **5**

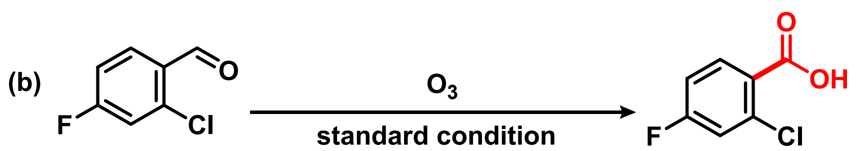


Display	File Na...	Detect...	Filter	Trace...	Mass...	Ranges	Smoot...	Chemi...	Mass...	Delay...	Refere...	Plot O...	Trace...	Range2	Comm...
True	D:\Test data \2026 YEAR \26LSYB.raw	MS		Mass Range	MDF Ranges	363.1885	None	C22H28ClFO	5	0	False				
False					MDF Ranges				NaN	0	False				

Peak Mass	Display F...	S Fit	RDB	Delta [pp...	Theo. ma...	Rank	Combine...	# Match...	# Missed...	MS Cov....	Pattern C...	MSMS M...
363.1884	$C_{22}H_{29}O^{35}ClF$	29.233653076796 3	7.50	-0.28	363.18855	1	9.3	1	1	8.19	74.66	(Collection)

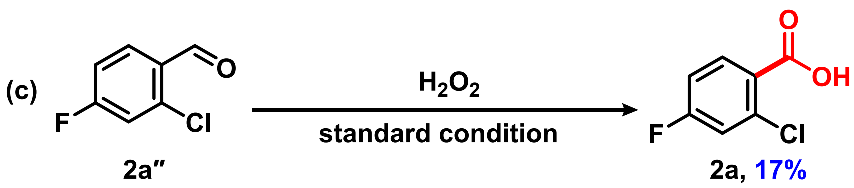
Figure S3 HRMS spectrum of the intermediate **6**

4.2 Control experiment

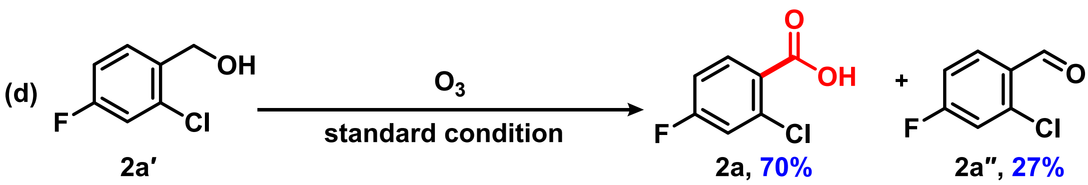


2a″ 2a, 97%

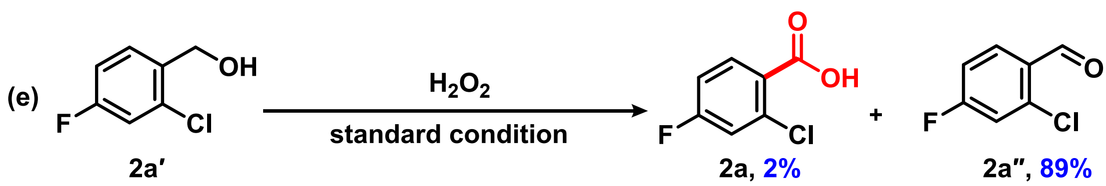
A 100 mL three-neck flask was charged with substrate **2a''**(317mg, 2 mmol), $Co(OAc)_2$ (0.2 mmol, 0.1 equiv.), TEBA (0.2 mmol, 0.1 equiv.) and HOAc (15 mL). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. The reaction mixture was stirred at 55 °C for 13 hours. The reaction was analyzed by GC and the yield of **2a** was 97%.



To a solution of **2a''** (317mg, 2 mmol) in HOAc (15 mL) was added $Co(OAc)_2$ (0.04g, 0.1 equiv.) and TEBA (0.05g, 0.1 equiv.). The mixture was heated to 55 °C using an oil bath. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction lasts for 13 hours. The reaction was analyzed by GC and the yield of **2a** was 17%.

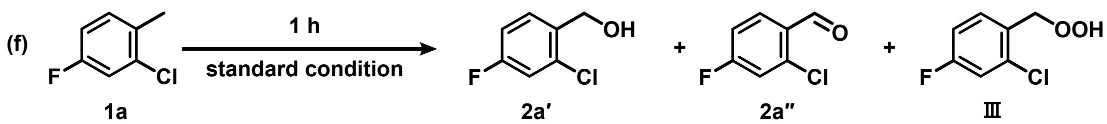


To a solution of **2a'** (321mg, 2 mmol) in HOAc (15 mL) was added $Co(OAc)_2$ (0.04g, 0.1 equiv.) and TEBA (0.05g, 0.1 equiv.). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. The reaction mixture was stirred at 55 °C for 13 hours. The reaction was analyzed by GC and the yield of **2a** was 70%, the yield of 2-chloro-4-fluorobenzaldehyde (**2a''**) was 27%.

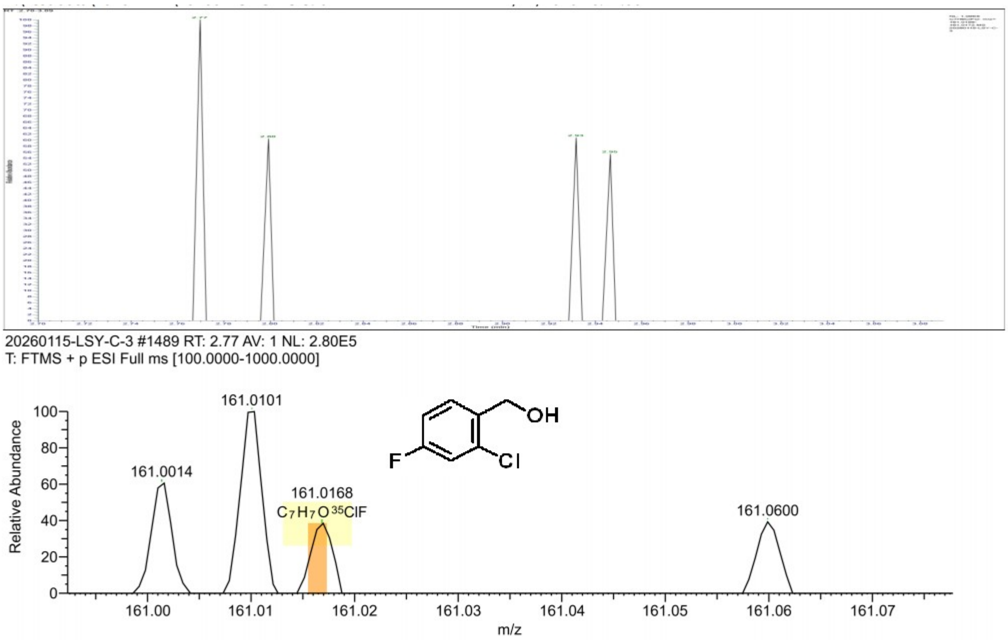


To a solution of **2a'** (321mg, 2 mmol) in HOAc (15 mL) was added $Co(OAc)_2$ (0.04g, 0.1 equiv.) and TEBA (0.05g, 0.1 equiv). The mixture was heated to 55 °C using an oil bath. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction lasts for 13 hours. The reaction was analyzed by GC and the yield of **2a** was 2%, the yield of 2-chloro-4-fluorobenzaldehyde (**2a''**) was 89%.

4.3 Intermediates trapping experiment



To a solution of **1a** (290mg, 2 mmol) in HOAc (15 mL) was added $Co(OAc)_2$ (0.04g, 0.1 equiv.) and TEBA (0.05g, 0.1 equiv.). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 1h and then analyzed by HRMS. Interestingly, alcohol (**2a′**), aldehyde (**2a″**) and peroxide intermediate (**III**) were captured by HRMS, indicated that these species were intermediate of the reaction.



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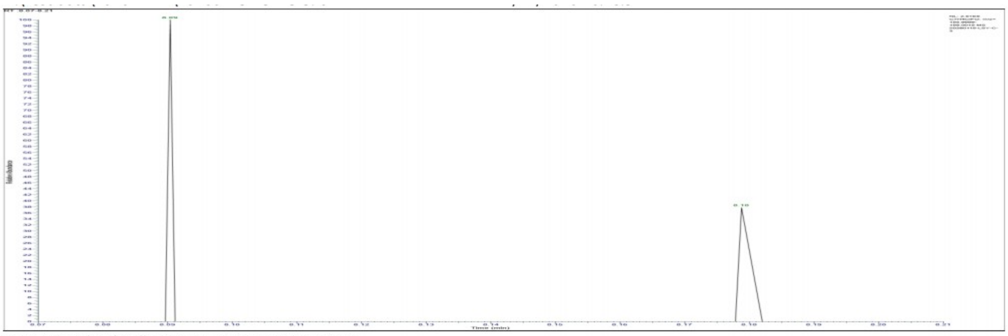
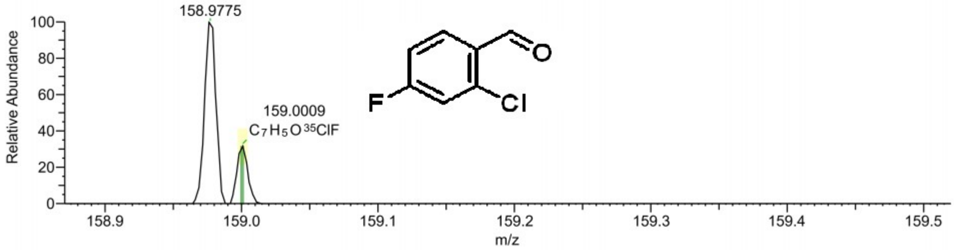
True	D:\Test data \2026 YEAR \20260115-LSY-C-3.raw	MS		Mass Range	MDF Ranges	161.0164	None	C7H6ClFO	5	0	False				
False					MDF Ranges				NaN	0	False				

Peak Mass	Display F...	S Fit	RDB	Delta [pp...	Theo. ma...	Rank	Combine...	# Match...	# Missed...	MS Cov....	Pattern C...	MSMS M...
161.0168	$C_7H_7O^{35}ClF$	0	3.50	2.78	161.01640	1	0	1	1	0	0	(Collection)

Figure S4 HRMS spectrum of the intermediate **2a'**



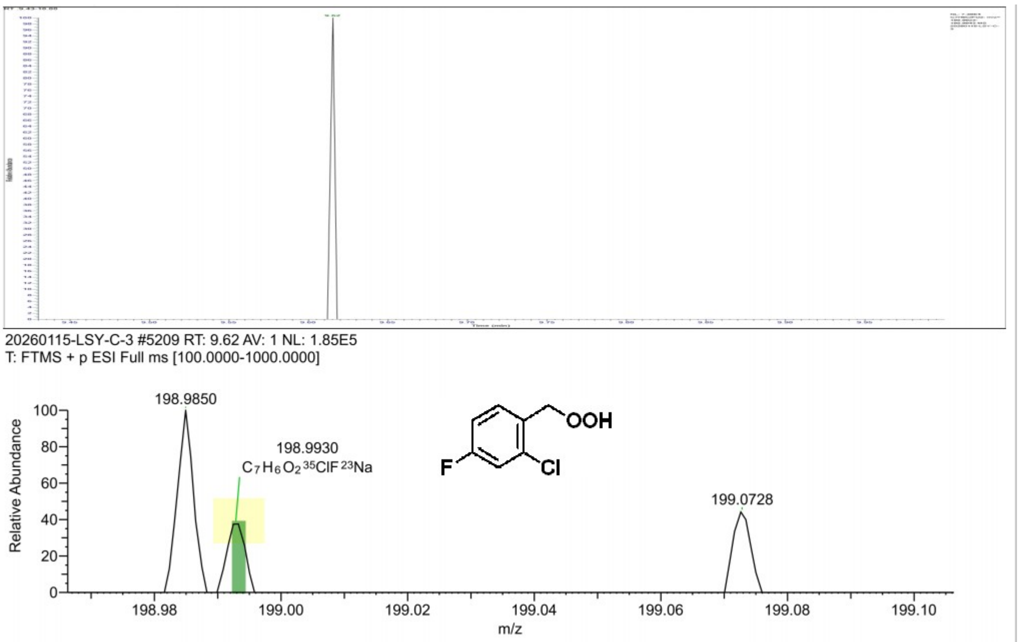
20260115-LSY-C-3 #4379 RT: 8.09 AV: 1 NL: 7.82E5
T: FTMS + p ESI d Full ms2 331.2481@hcd50.00 [50.0000-358.8931]



Display	File Na...	Detect...	Filter	Trace...	Mass...	Ranges	Smoot...	Chemi...	Mass...	Delay...	Refere...	Plot O...	Trace...	Range2	Comm...
True	D:\Test data \2026 YEAR \20260115-LSY-C-3.raw	MS		Mass Range	MDF Ranges	159.0007	None	C7H4ClFO	5	0	False				
False					MDF Ranges				NaN	0	False				

Peak Mass	Display F...	S Fit	RDB	Delta [pp...	Theo. ma...	Rank	Combine...	# Match...	# Missed...	MS Cov....	Pattern C...	MSMS M...
159.0009	$C_7H_5O^{35}ClF$	28.7604986639862	4.50	0.87	159.00075	1	77.86	1	1	80.59	75.78	(Collection)

Figure S5 HRMS spectrum of the intermediate **2a''**



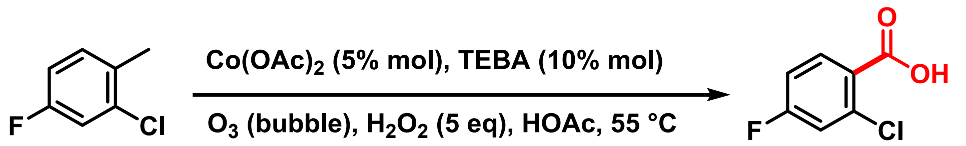
Display	File Na...	Detect...	Filter	Trace...	Mass...	Ranges	Smoot...	Chemi...	Mass...	Delay...	Refere...	Plot O...	Trace...	Range2	Comm...
True	D:\Test data\2026 YEAR\20260115-LSY-C-3.raw	MS		Mass Range	MDF Ranges	198.9933	None	C7H6ClFO2	5	0	False				
False					MDF Ranges				NaN	0	False				

Peak Mass	Display F...	S Fit	RDB	Delta [pp...	Theo. ma...	Rank	Combine...	# Match...	# Missed...	MS Cov....	Pattern C...	MSMS M...
198.9930	$C_7H_6O_2{}^{35}ClF{}^{23}Na$	82.5807480313189	3.50	-1.23	198.99326	1	99.08	1	0	100	100	(Collection)

Figure S6 HRMS spectrum of the intermediate Ⅲ

5. Gram scale experiments and applications

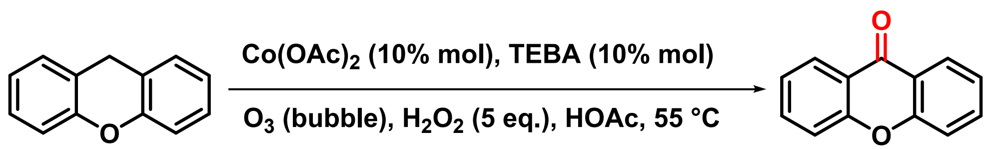
5.1 Gram scale syntheses 2-chloro-4-fluorobenzoic acid



1a, 13.9 mmol 2a, 1.86 g, 77%

A 100 mL three-neck flask was charged with substrate **1a** (2 g, 13.8 mmol), acetic acid (50 mL), cobalt acetate (0.12 g, 0.05 eq.), and TEBA (0.38 g, 0.1 eq.). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 5 to 13 hours. Upon completion, the reaction solution was concentrated and dissolved in ethyl acetate (15 mL). It was then washed with 10% sodium hydroxide solution to remove raw materials and neutral impurities. The aqueous phase was further adjusted to a pH of 5-6 with 10% dilute hydrochloric acid. The organic phase was then extracted with ethyl acetate (15 mL × 3), and the resulting organic phase was concentrated to obtain the target product **2a** (1.86 g, 77 % yield) as yellow oily.

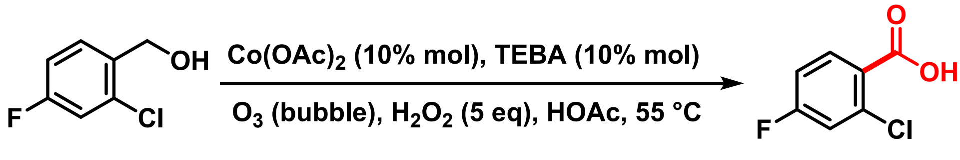
5.2 Gram scale syntheses xanthen-9-one



3l, 11.0mmol 4l, 2.11g, 98%

A 100 mL three-neck flask was charged with substrate **3l** (2 g, 11.0 mmol), acetic acid (50 mL), cobalt acetate (0.12 g, 0.05 eq.), and TEBA (0.38 g, 0.1 eq.). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 5 to 13 hours. Upon completion, the reaction solution was concentrated and dissolved in ethyl acetate (15 mL). It was then washed with 10% sodium hydroxide solution to remove raw materials and neutral impurities. The aqueous phase was further adjusted to a pH of 5-6 with 10% dilute hydrochloric acid. The organic phase was then extracted with ethyl acetate (15 mL × 3), and the resulting organic phase was concentrated to obtain the target product **4l** (2.11 g, 98 % yield) as yellow oily.

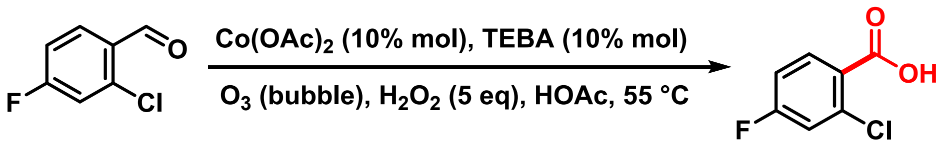
5.3 Oxidation of (2-chloro-4-fluorophenyl)methanol



2a, 91%

In a round-bottomed flask equipped with a magnetic stirrer, add (2-chloro-4-fluorophenyl)methanol (2 mmol, 1 equiv.), HOAc (15 mL), $Co(OAc)_2$ (0.2mmol, 0.1 equiv.), and TEBA (0.2mmol, 0.1 equiv.). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 5 to 13 hours. Upon completion, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography, eluting with petroleum ether/EtOAc (5→20%, v/v) to afford the corresponding **2a** (91 % yield).

5.4 Oxidation of 2-chloro-4-fluorobenzaldehyde



2a, 94%

In a round-bottomed flask equipped with a magnetic stirrer, add 2-chloro-4-fluorobenzaldehyde (2 mmol, 1 equiv.), HOAc (15 mL), $Co(OAc)_2$ (0.2mmol, 0.1 equiv.), and TEBA (0.2mmol, 0.1 equiv.). The mixture was heated to 55 °C using an oil bath. Ozone generated by the ozone generator was introduced into the reaction solution via bubbling. Subsequently, hydrogen peroxide (5 equiv.) was added dropwise at a controlled rate. The reaction mixture was stirred at 55 °C for 5 to 13 hours. Upon completion, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography, eluting with petroleum ether/EtOAc (5→20%, v/v) to afford the corresponding **2a** (94 % yield).

6. Reference

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7. NMR Spectra of Compounds

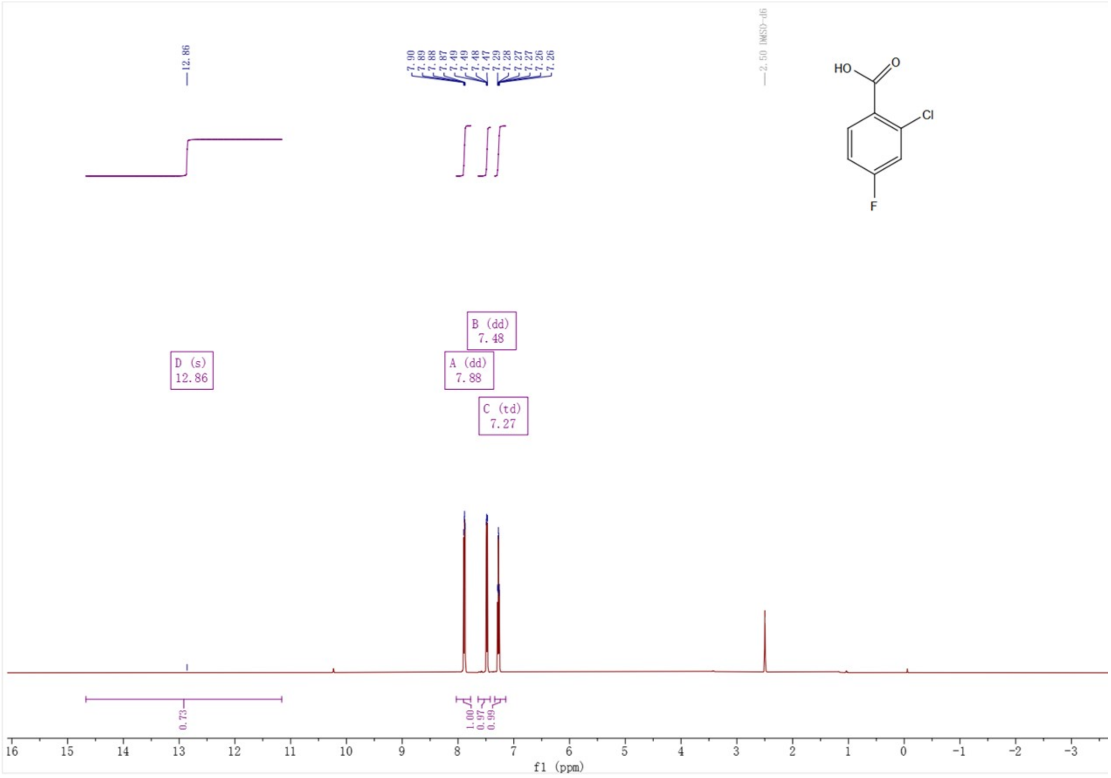

Figure S7 ^{1}H NMR spectrum of compound **2a** (600 MHz, DMSO-d_6)

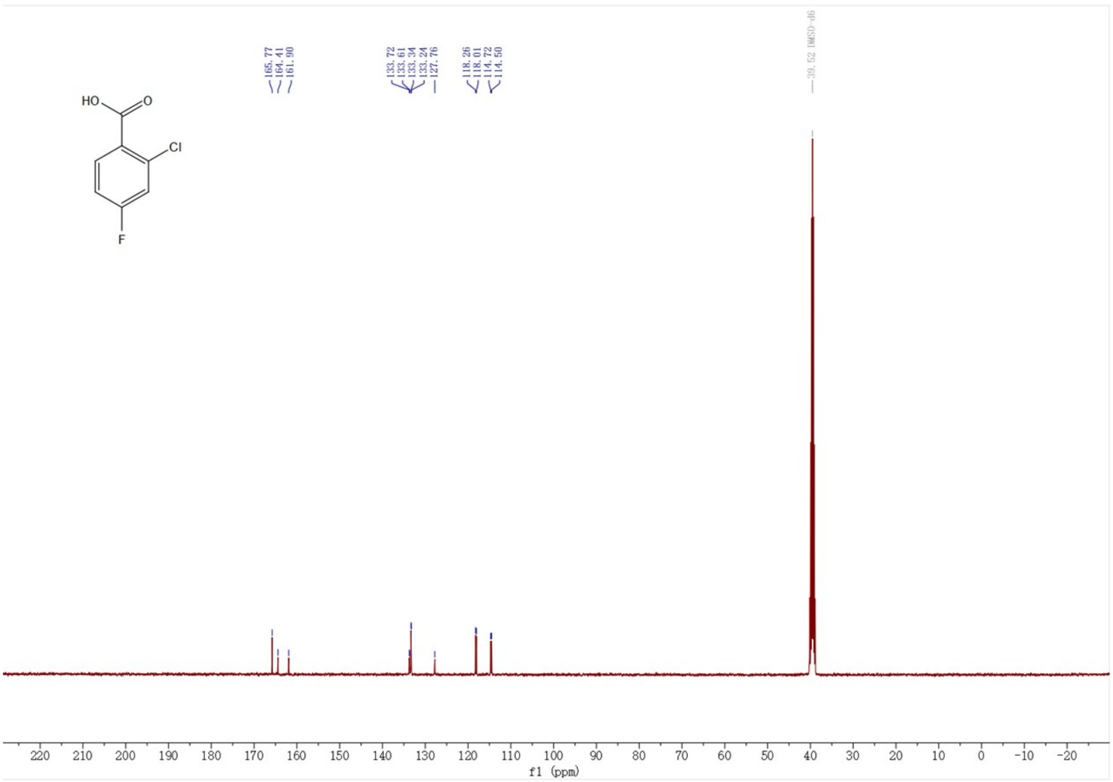

Figure S8 ^{13}C NMR spectrum of compound **2a** (101 MHz, DMSO-d_6)

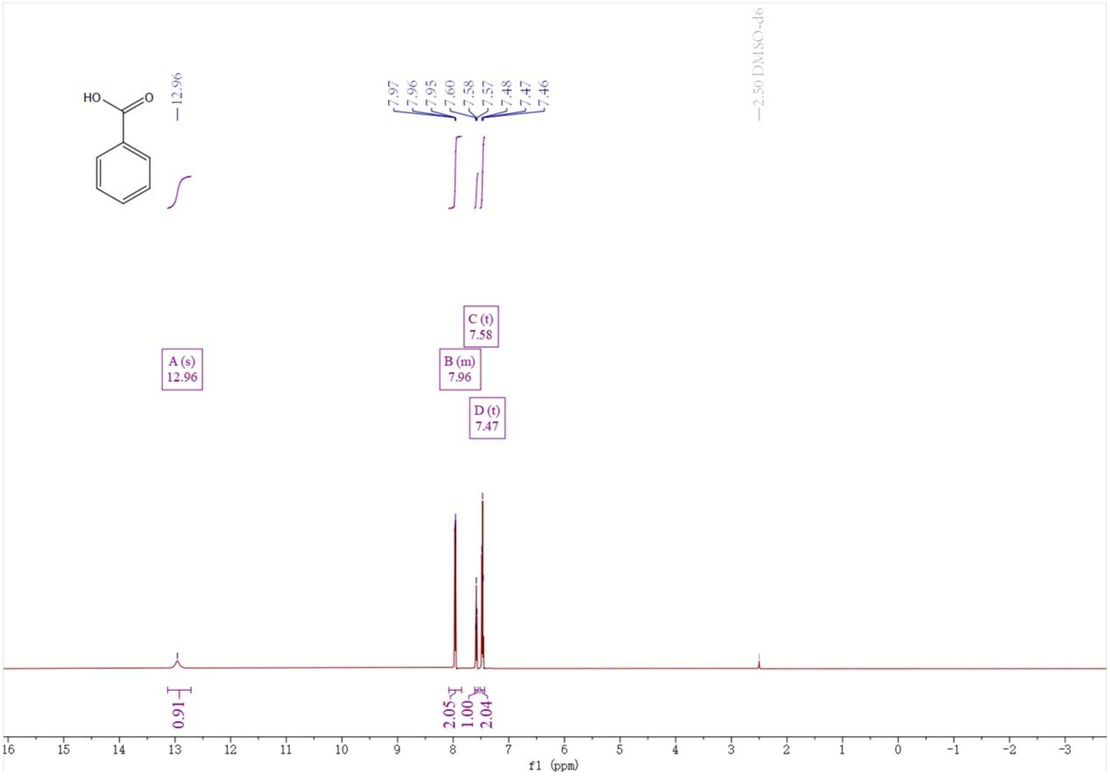

Figure S9 ^{1}H NMR spectrum of compound **2b** (600 MHz, DMSO-d_6)

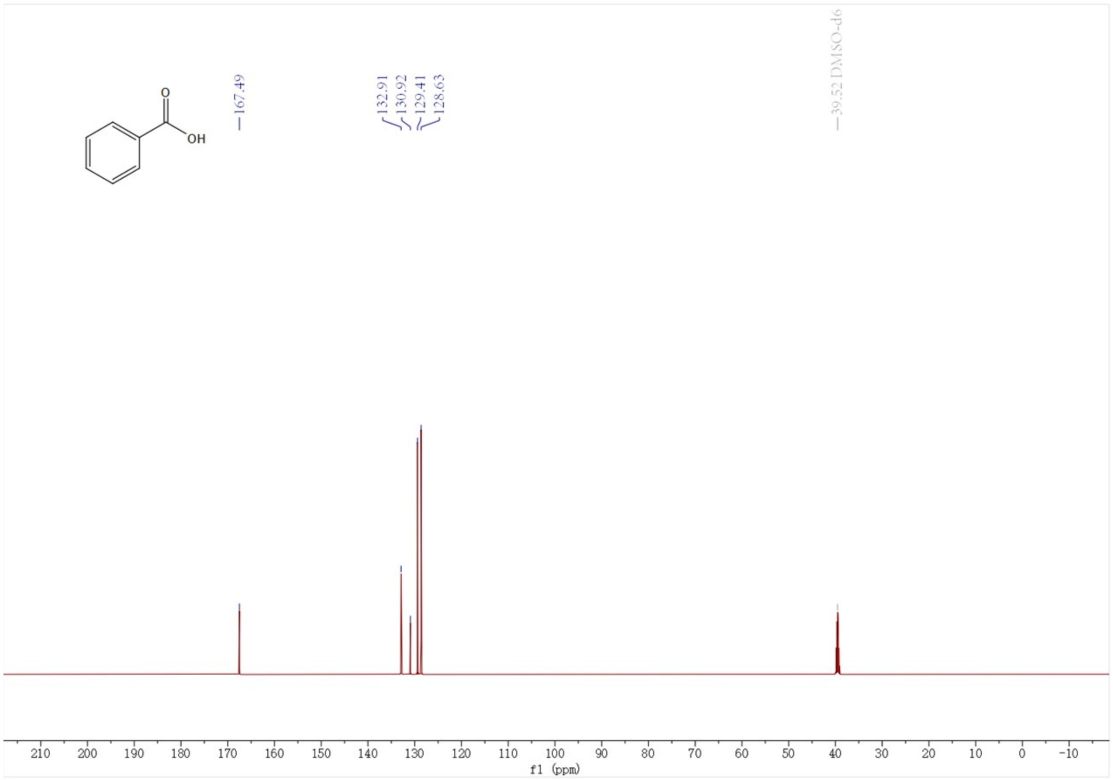

Figure S10 ^{13}C NMR spectrum of compound **2a** (151 MHz, DMSO-d_6)

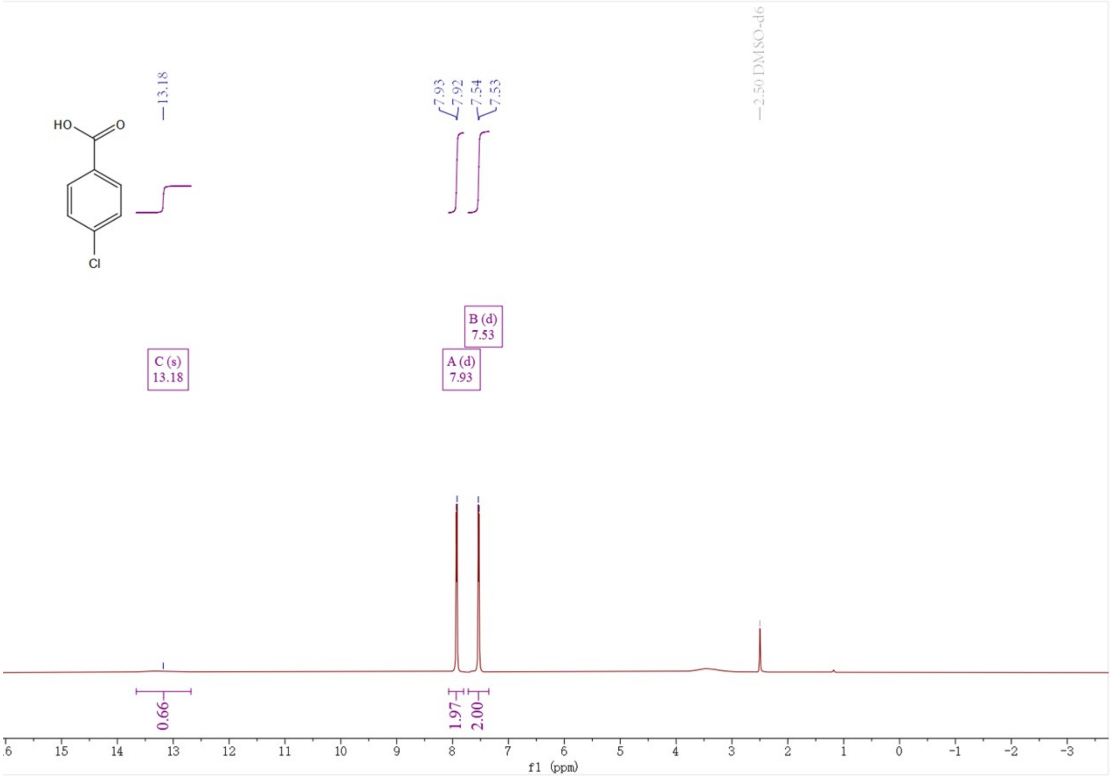

Figure S11 ^{1}H NMR spectrum of compound **2c** (600 MHz, DMSO-d_6)

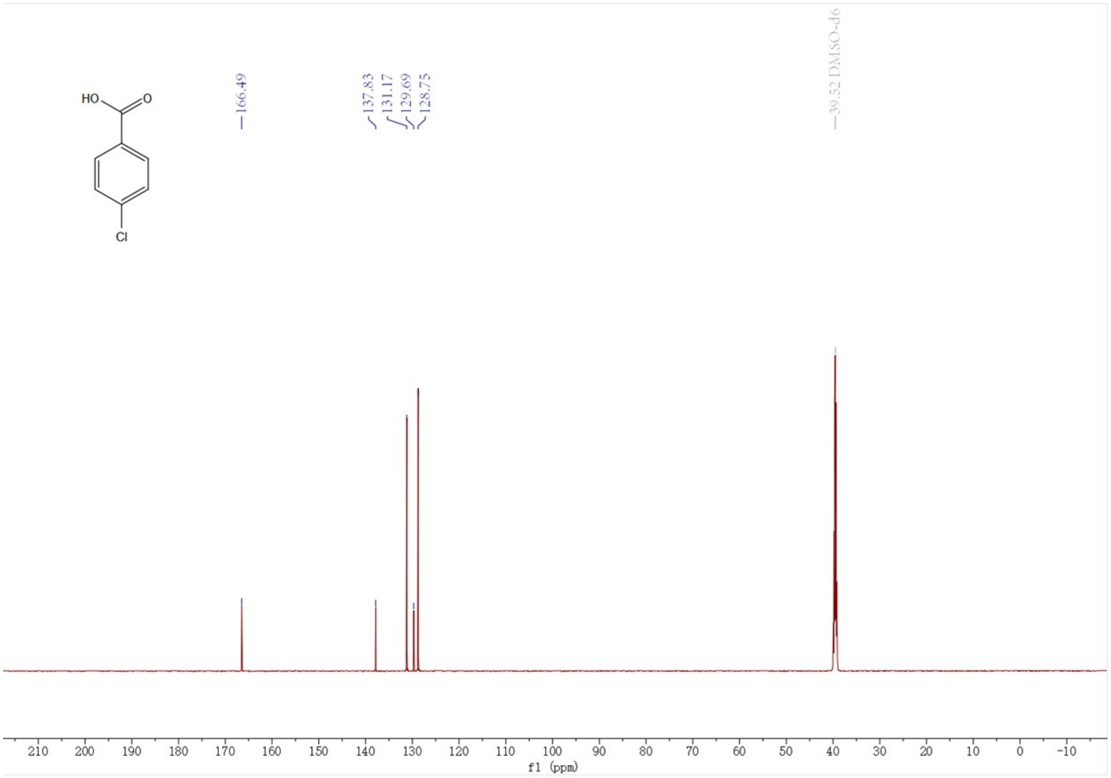

Figure S12 ^{13}C NMR spectrum of compound **2c** (151 MHz, DMSO-d_6)

Figure S13 ^{1}H NMR spectrum of compound **2d** (600 MHz, DMSO-d_6)

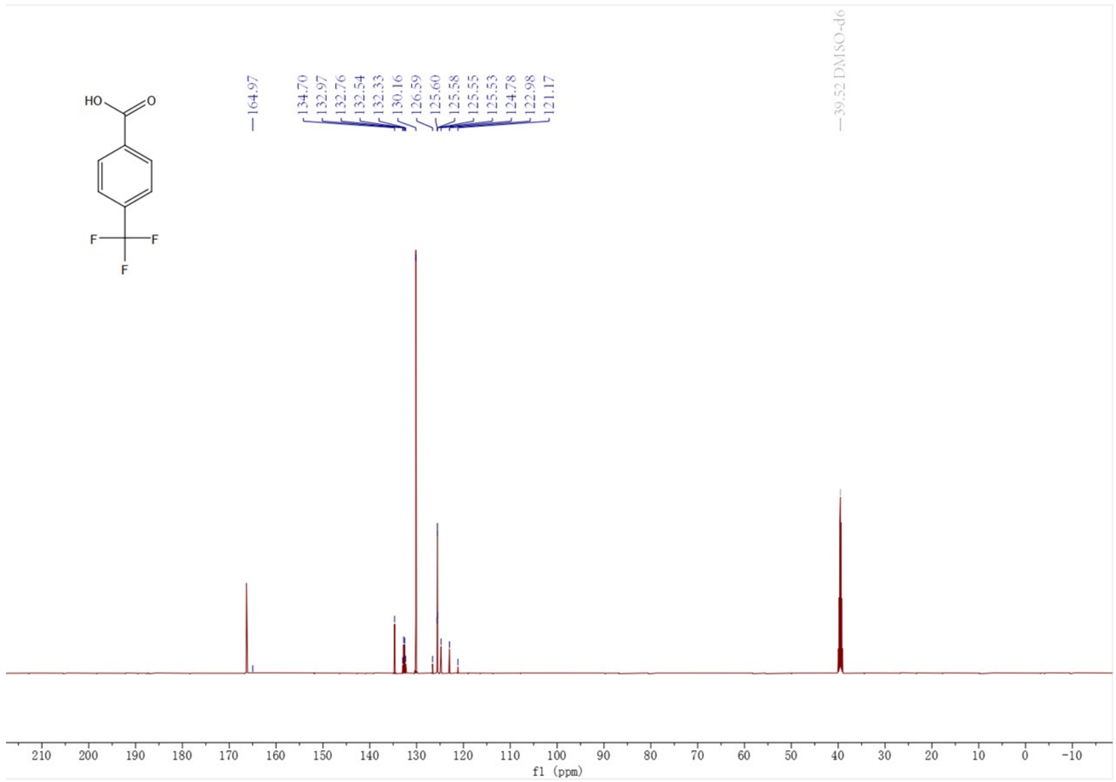

Figure S14 ^{13}C NMR spectrum of compound **2d** (151 MHz, DMSO-d_6)

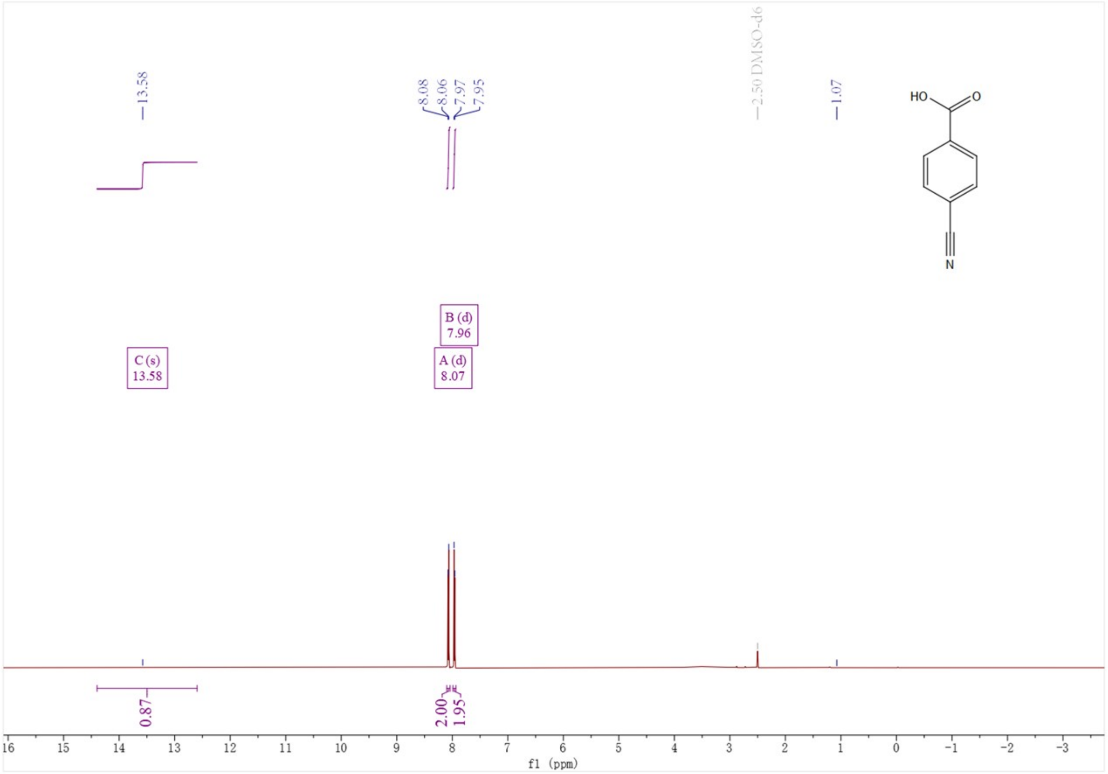

Figure S15 ^{1}H NMR spectrum of compound **2e** (600 MHz, DMSO-d_6)

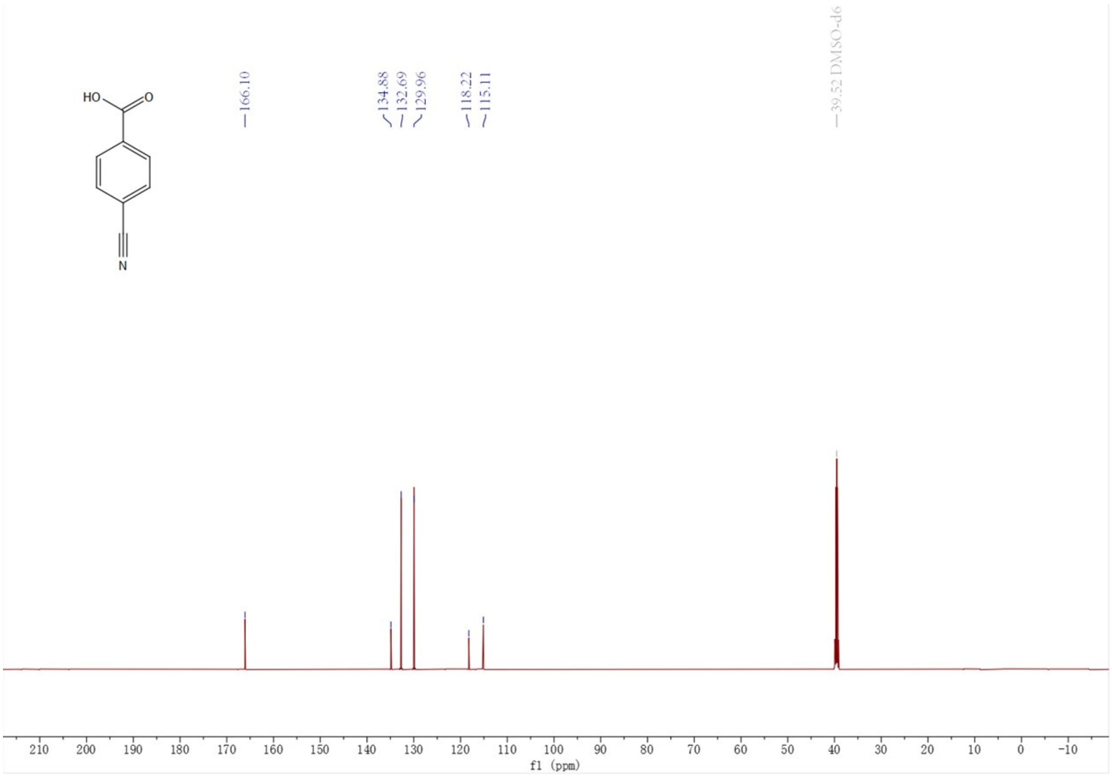

Figure S16 ^{13}C NMR spectrum of compound **2e** (151 MHz, DMSO-d_6)

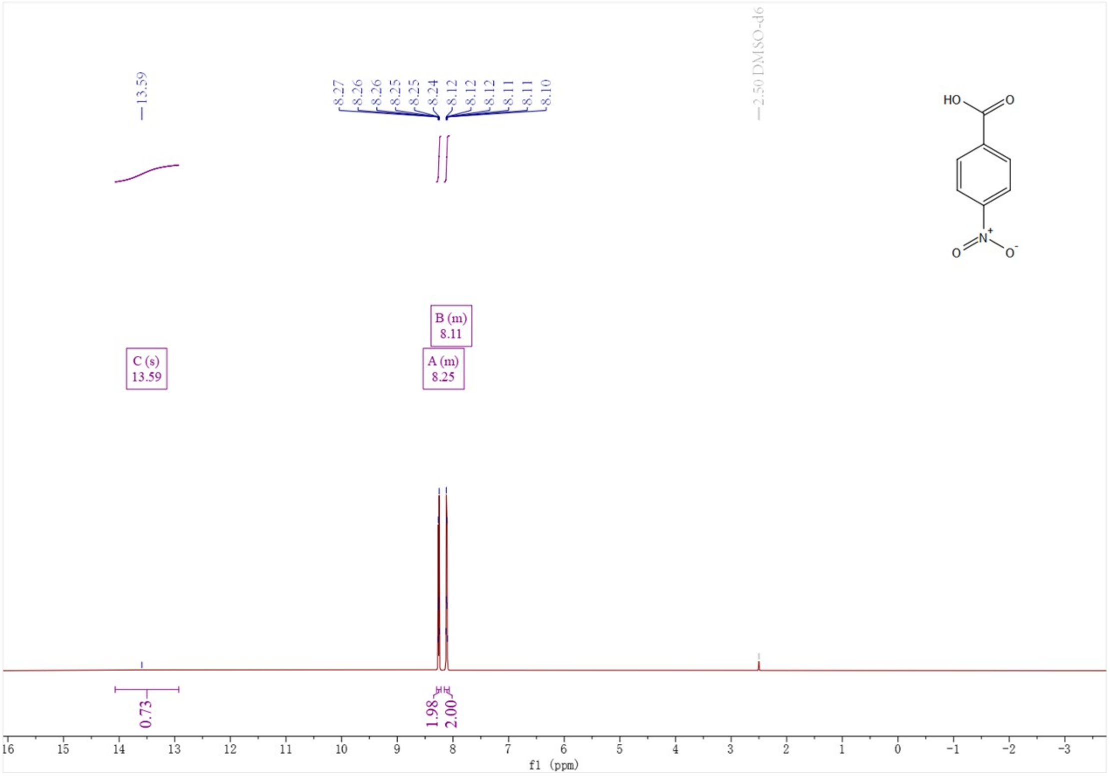

Figure S17 ^{1}H NMR spectrum of compound **2f** (600 MHz, DMSO-d_6)

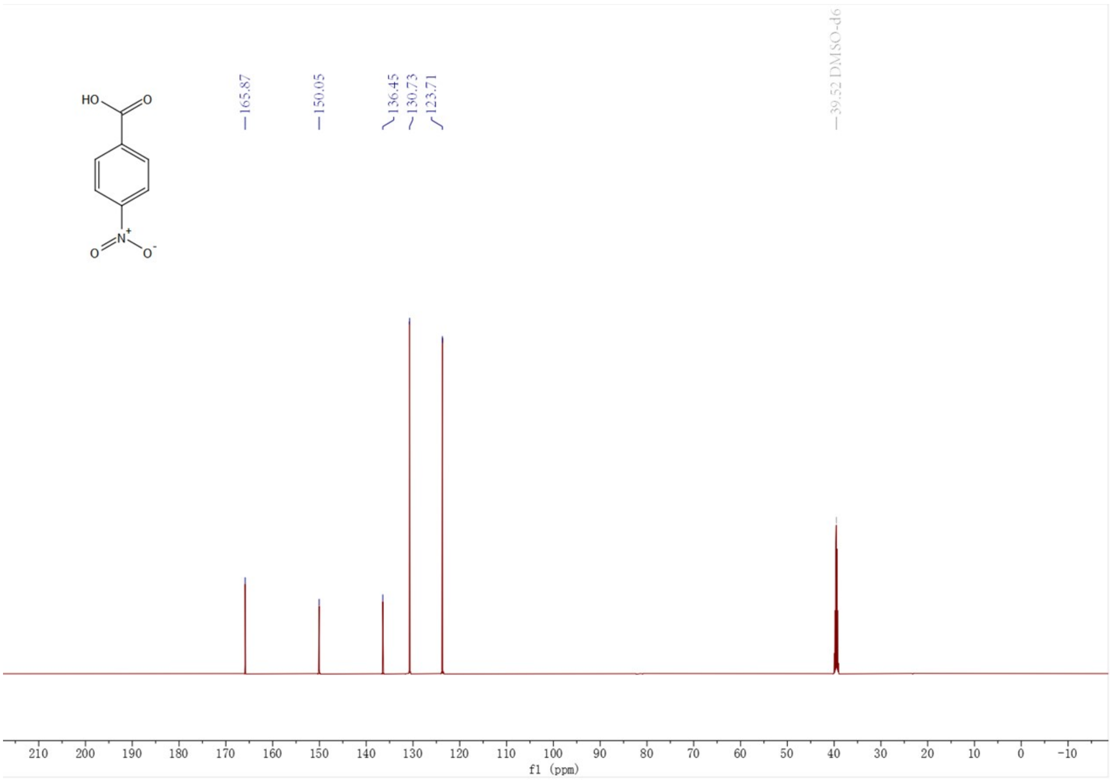

Figure S18 ^{13}C NMR spectrum of compound **2f** (151 MHz, DMSO-d_6)

Figure S19 ^{1}H NMR spectrum of compound **2g** (600 MHz, DMSO-d_6)

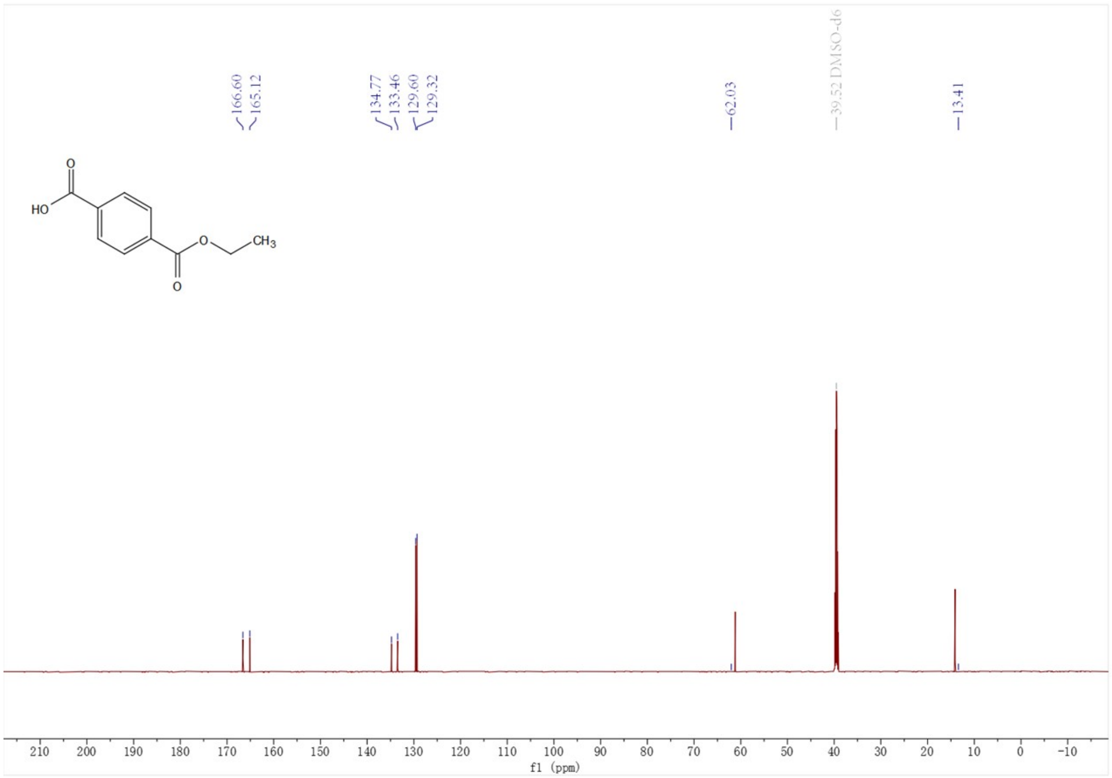

Figure S20 ^{13}C NMR spectrum of compound **2g** (151 MHz, DMSO-d_6)

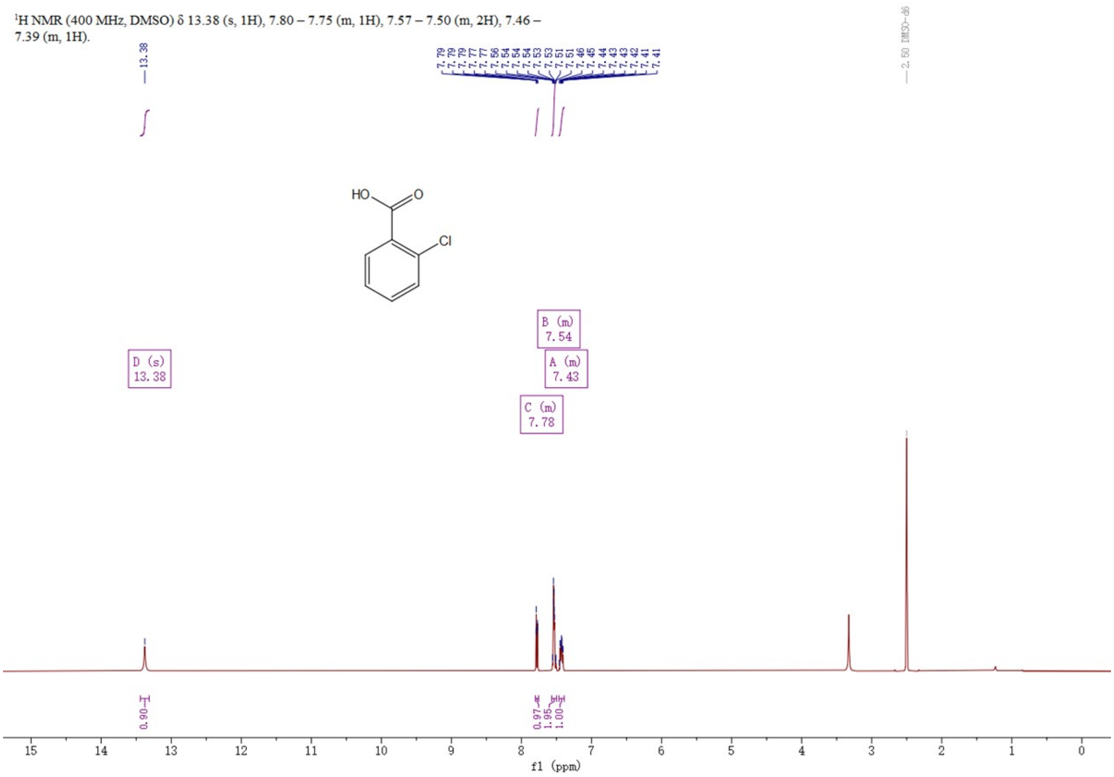

Figure S21 ^{1}H NMR spectrum of compound **2h** (400 MHz, DMSO-d_6)

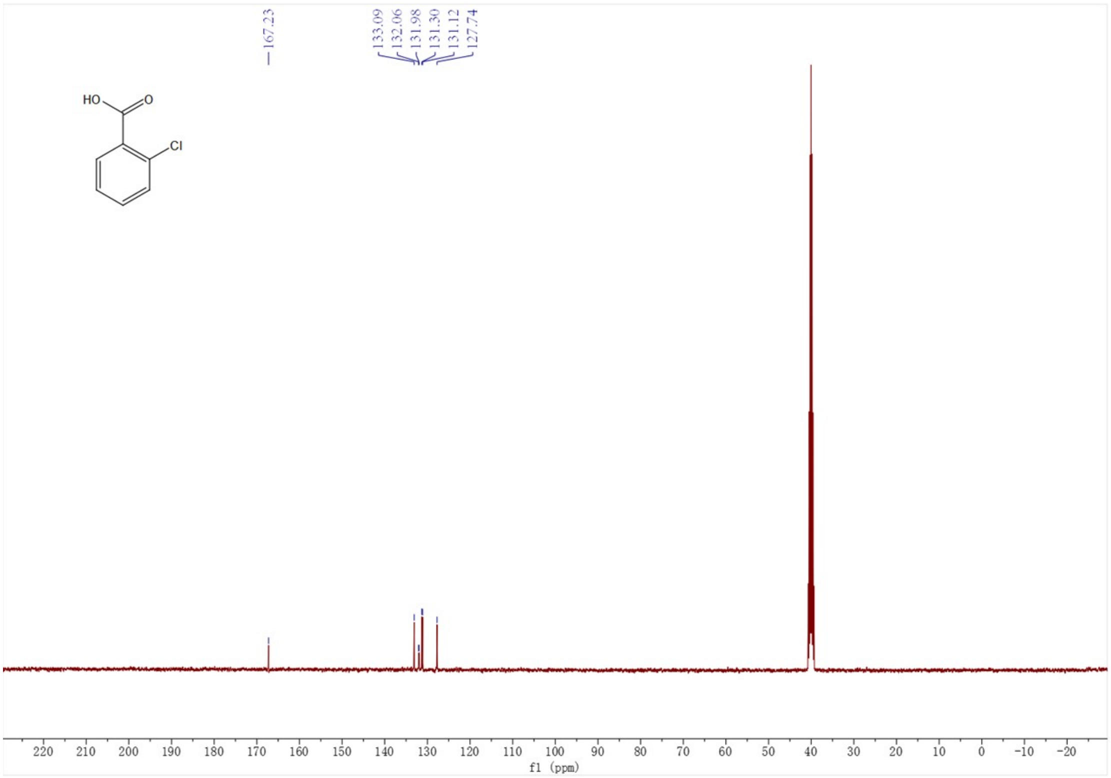

Figure S22 ^{13}C NMR spectrum of compound **2h** (101 MHz, DMSO-d_6)

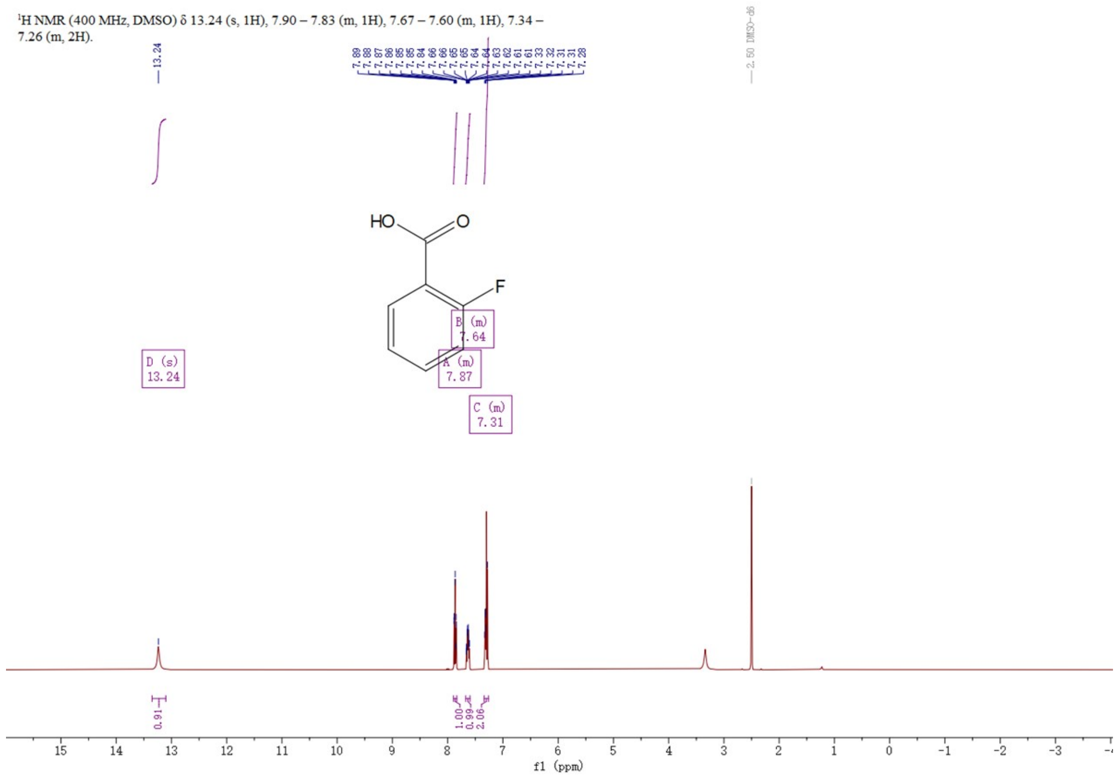

Figure S23 ^{1}H NMR spectrum of compound **2i** (400 MHz, DMSO-d_6)

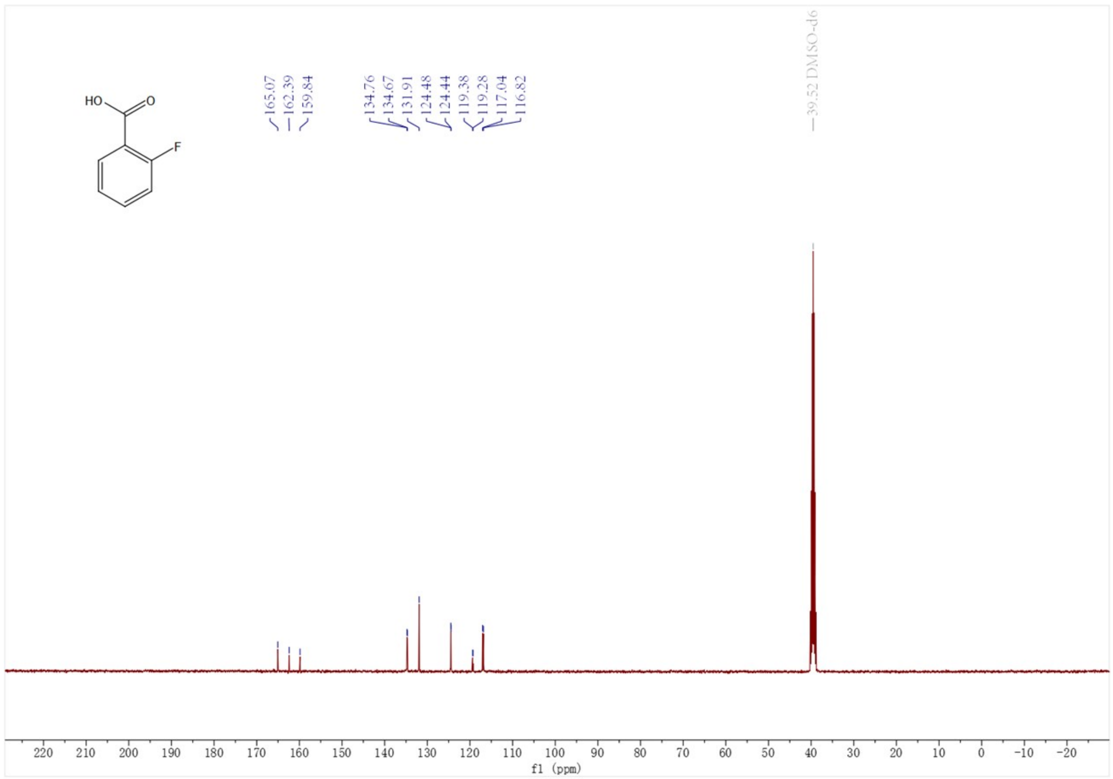

Figure S24 ^{13}C NMR spectrum of compound **2i** (101 MHz, DMSO-d_6)

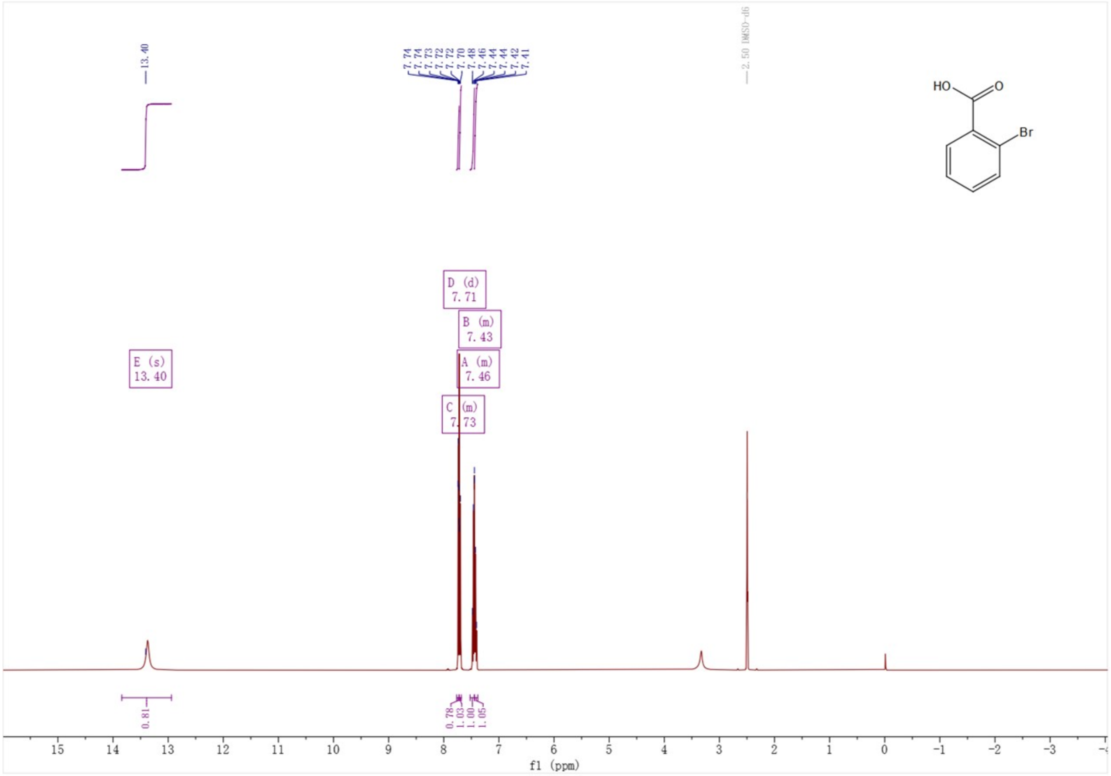

Figure S25 ^{1}H NMR spectrum of compound **2j** (400 MHz, DMSO-d_6)

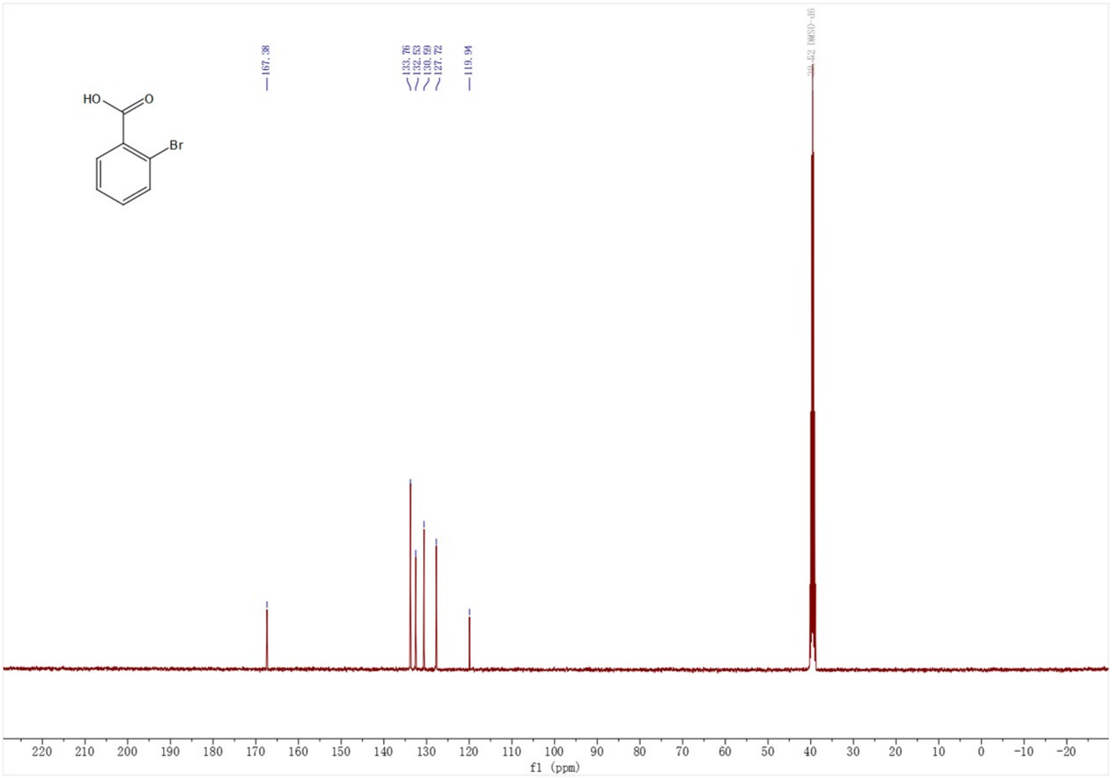

Figure S26 ^{13}C NMR spectrum of compound **2j** (101 MHz, DMSO-d_6)

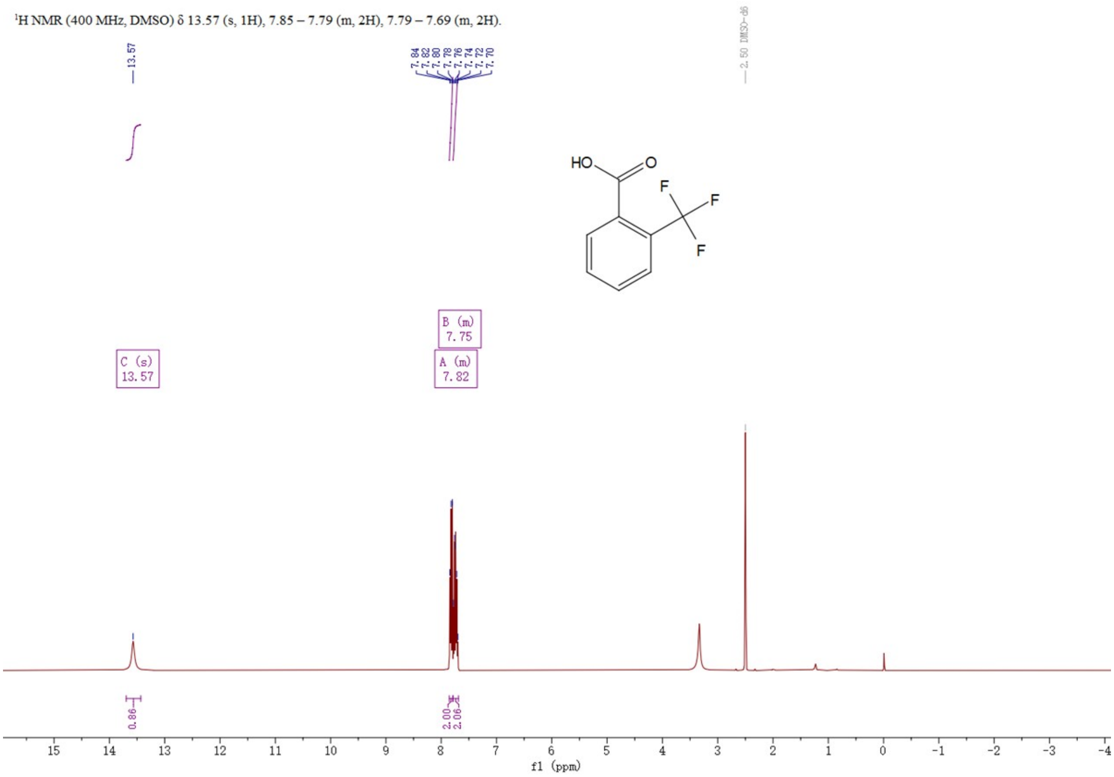

Figure S27 ^{1}H NMR spectrum of compound **2k** (400 MHz, DMSO-d_6)

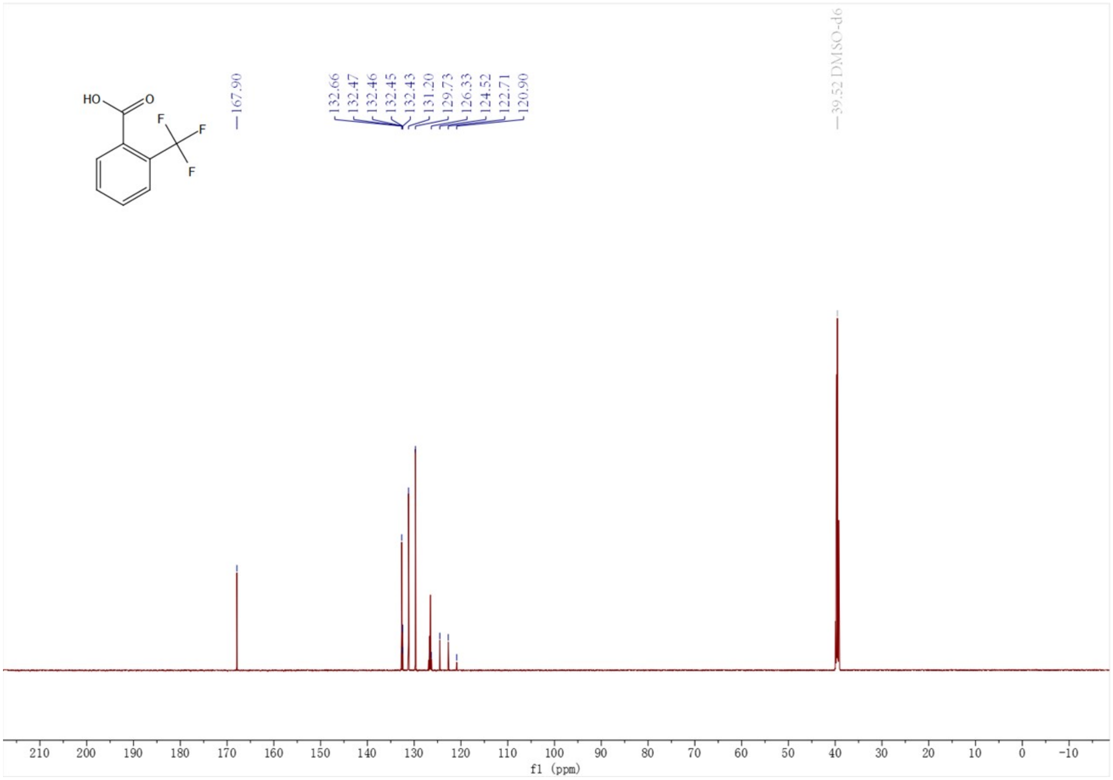

Figure S28 ^{13}C NMR spectrum of compound **2k** (151 MHz, DMSO-d_6)

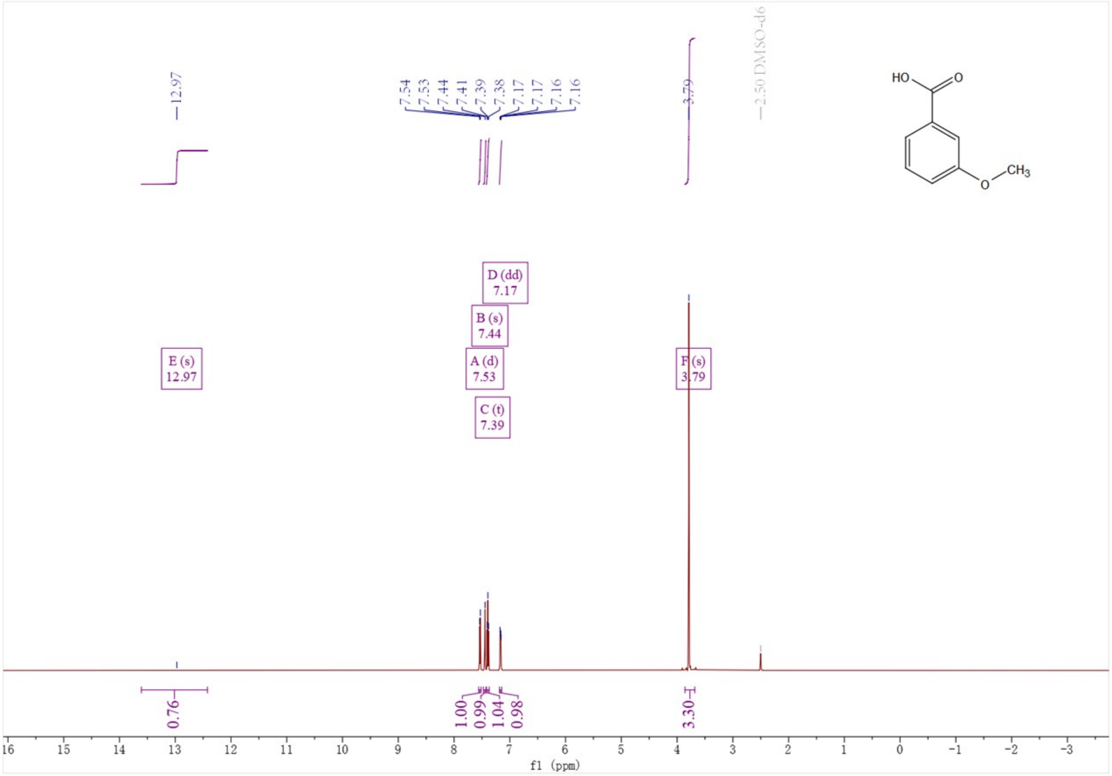

Figure S29 ^{1}H NMR spectrum of compound **2l** (600 MHz, DMSO-d_6)

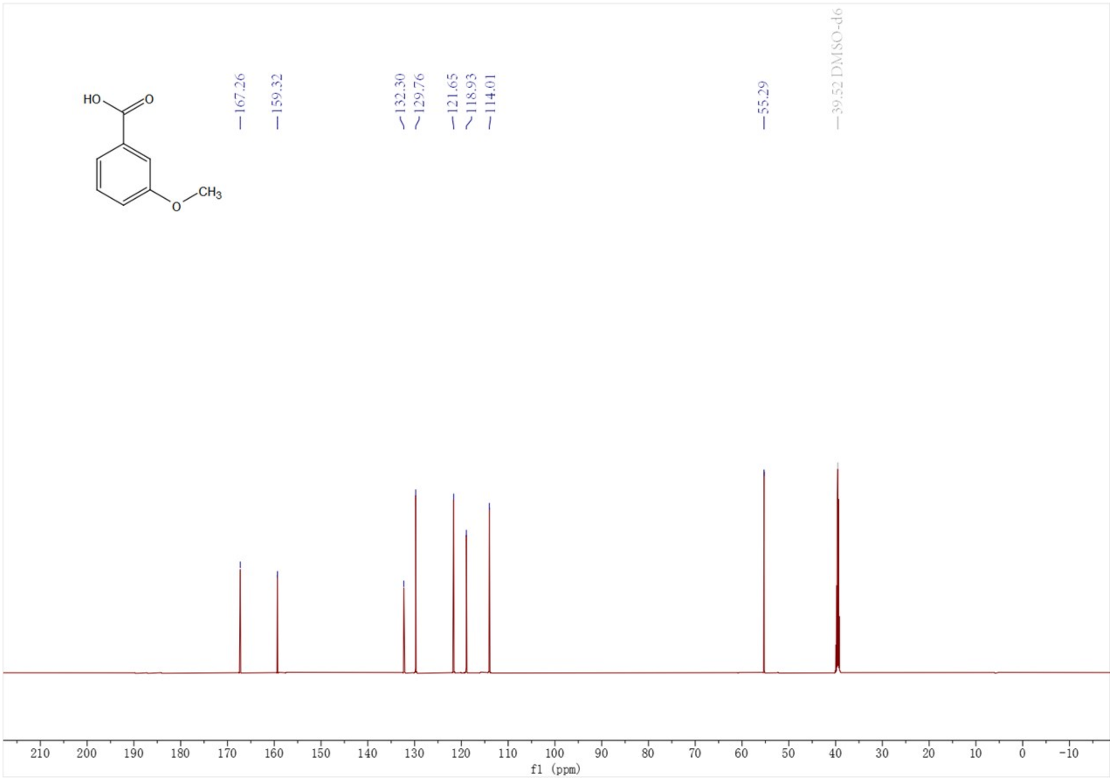

Figure S30 ^{13}C NMR spectrum of compound **2l** (151 MHz, DMSO-d_6)

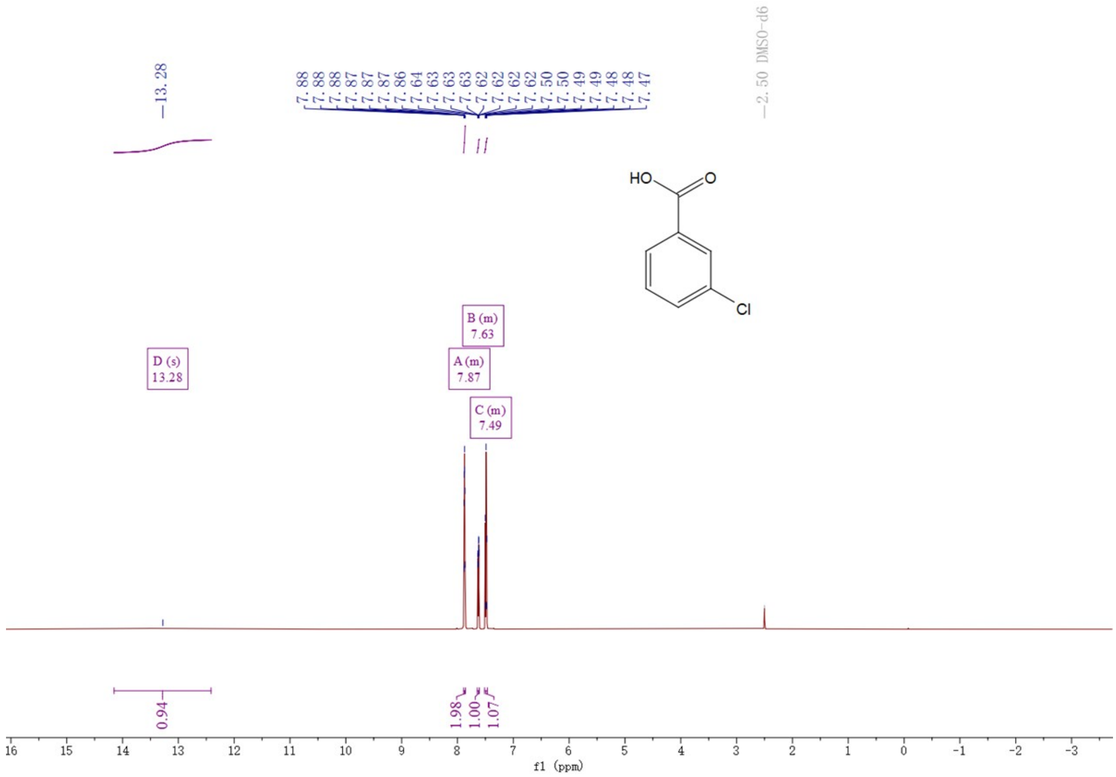

Figure S31 ^{1}H NMR spectrum of compound **2m** (600 MHz, DMSO-d_6)

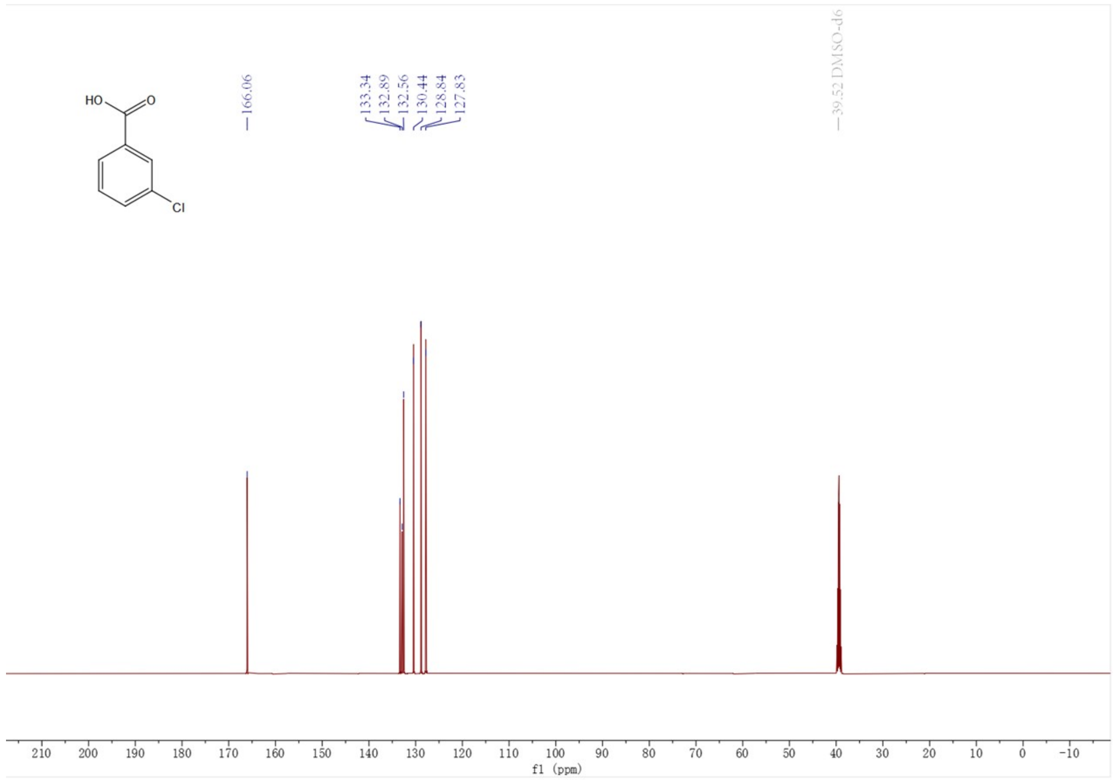

Figure S32 ^{13}C NMR spectrum of compound **2m** (151 MHz, DMSO-d_6)

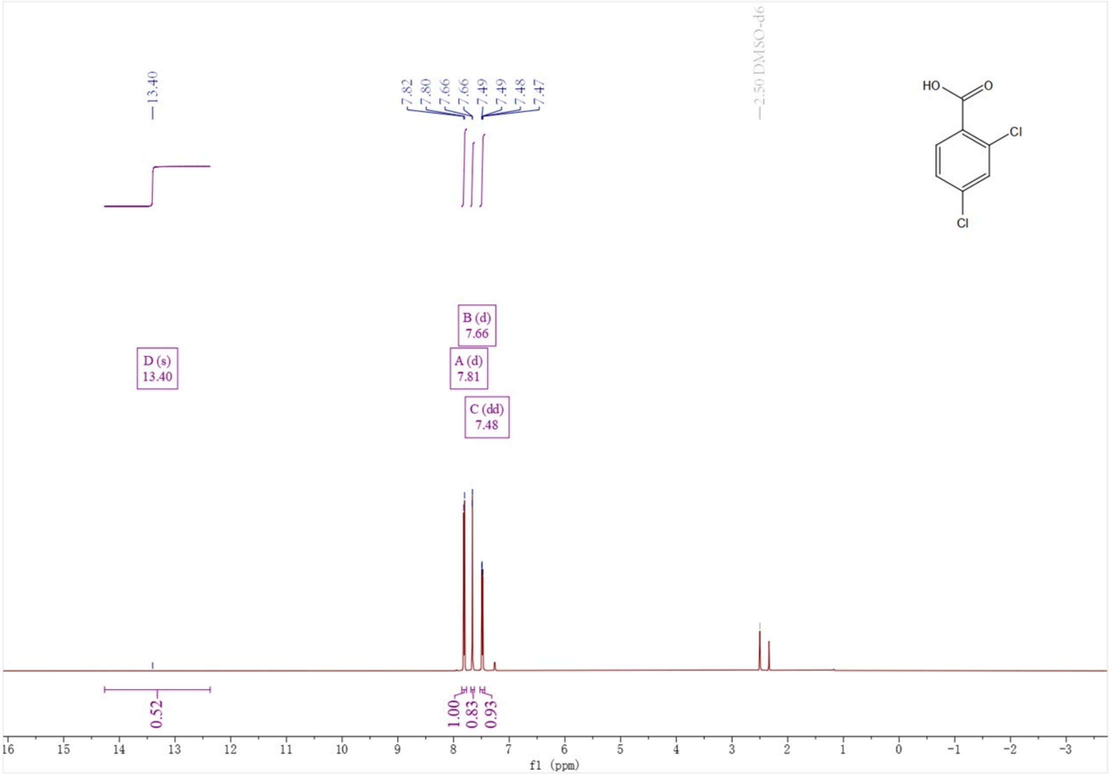

Figure S33 ^{1}H NMR spectrum of compound **2n** (600 MHz, DMSO-d_6)

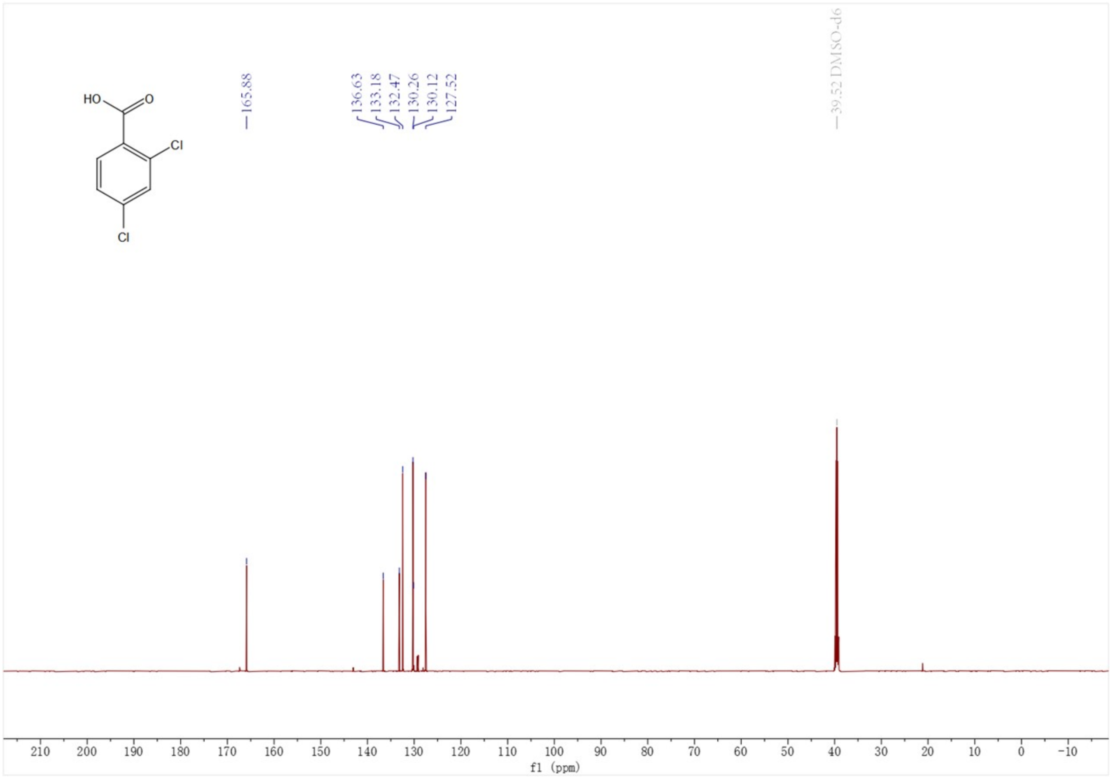

Figure S34 ^{13}C NMR spectrum of compound **2n** (151 MHz, DMSO-d_6)

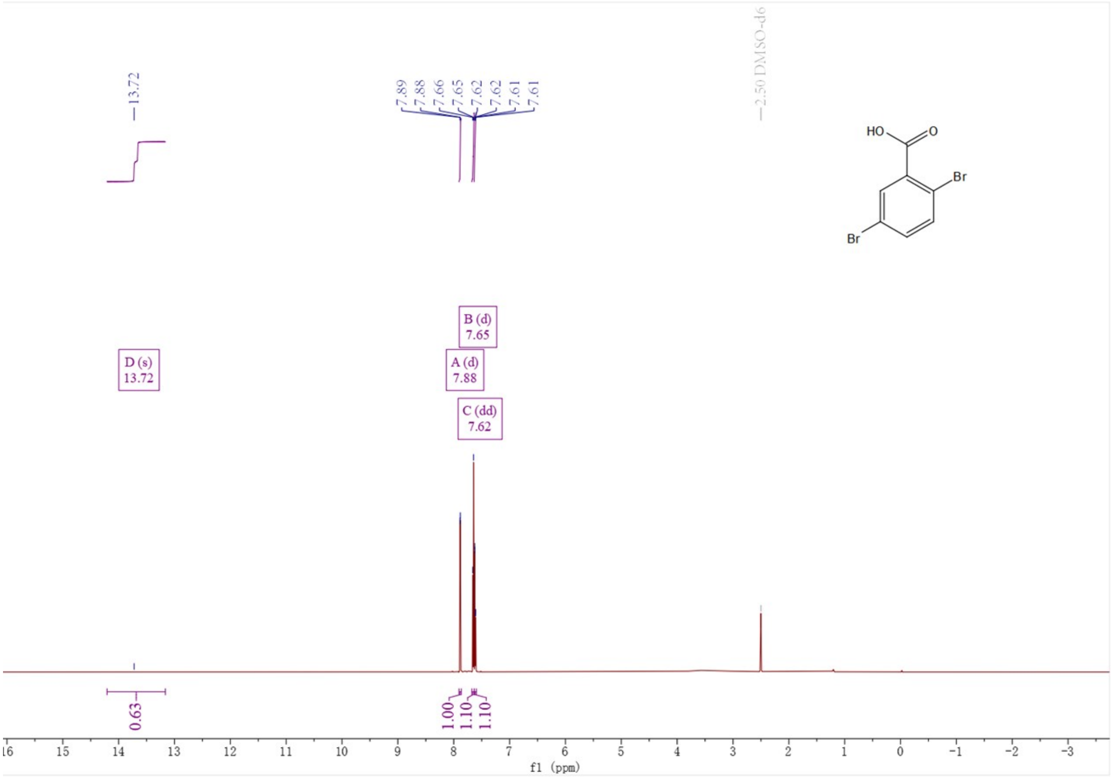

Figure S35 ^{1}H NMR spectrum of compound **2o** (600 MHz, DMSO-d_6)

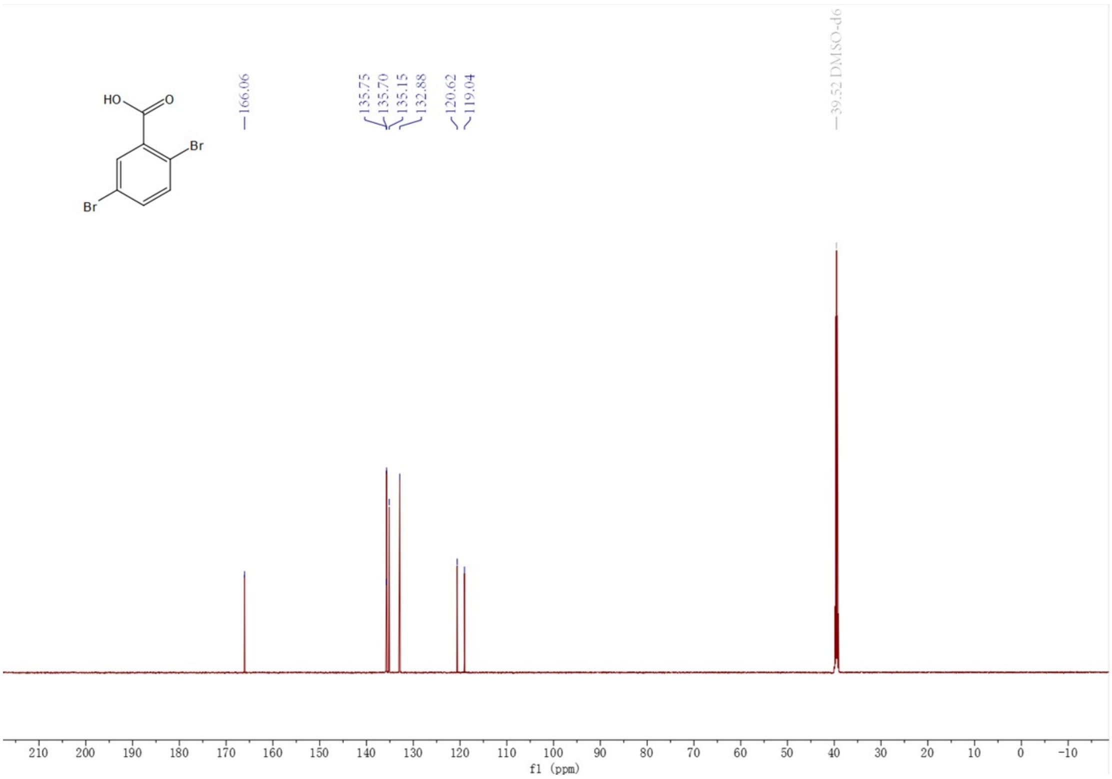

Figure S36 ^{13}C NMR spectrum of compound **2o** (151 MHz, DMSO-d_6)

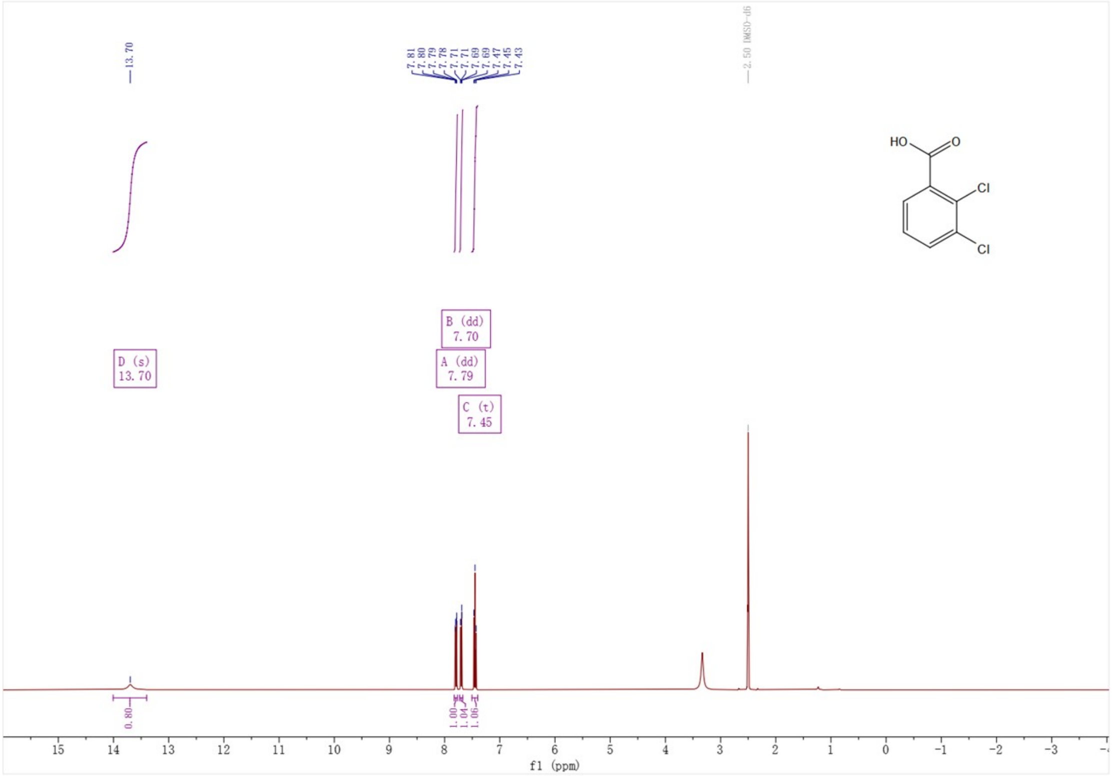

Figure S37 ^{1}H NMR spectrum of compound **2p** (400 MHz, DMSO-d_6)

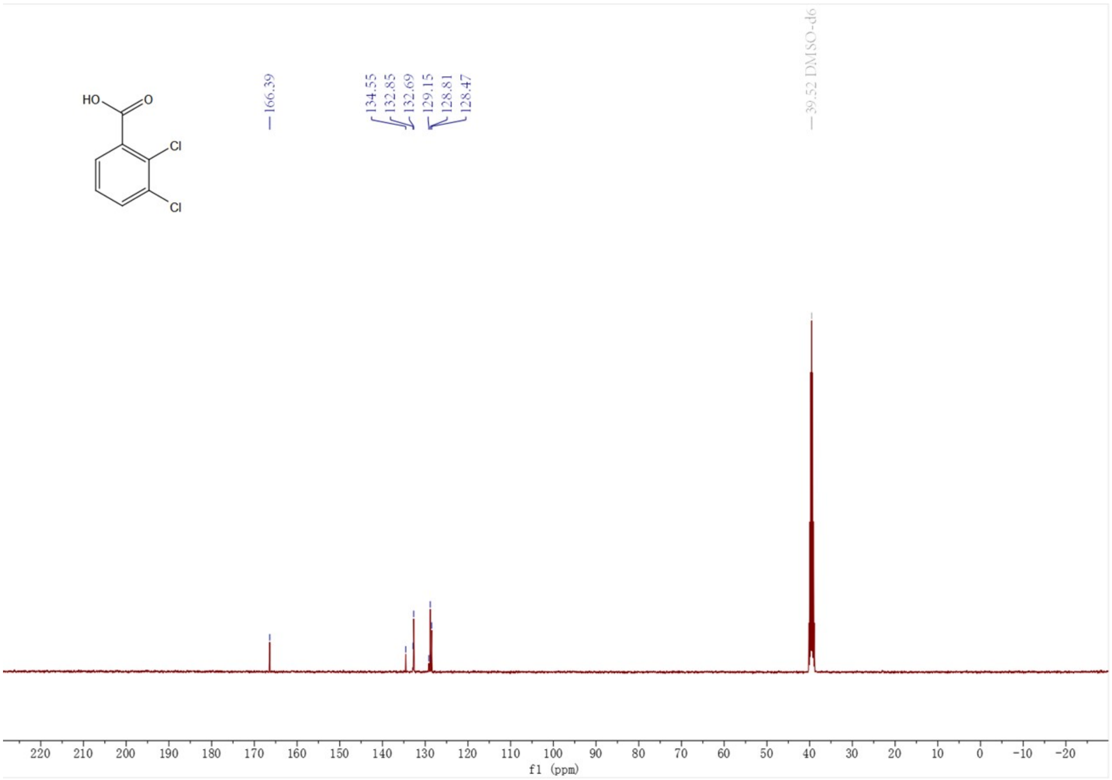

Figure S38 ^{13}C NMR spectrum of compound **2p** (101 MHz, DMSO-d_6)

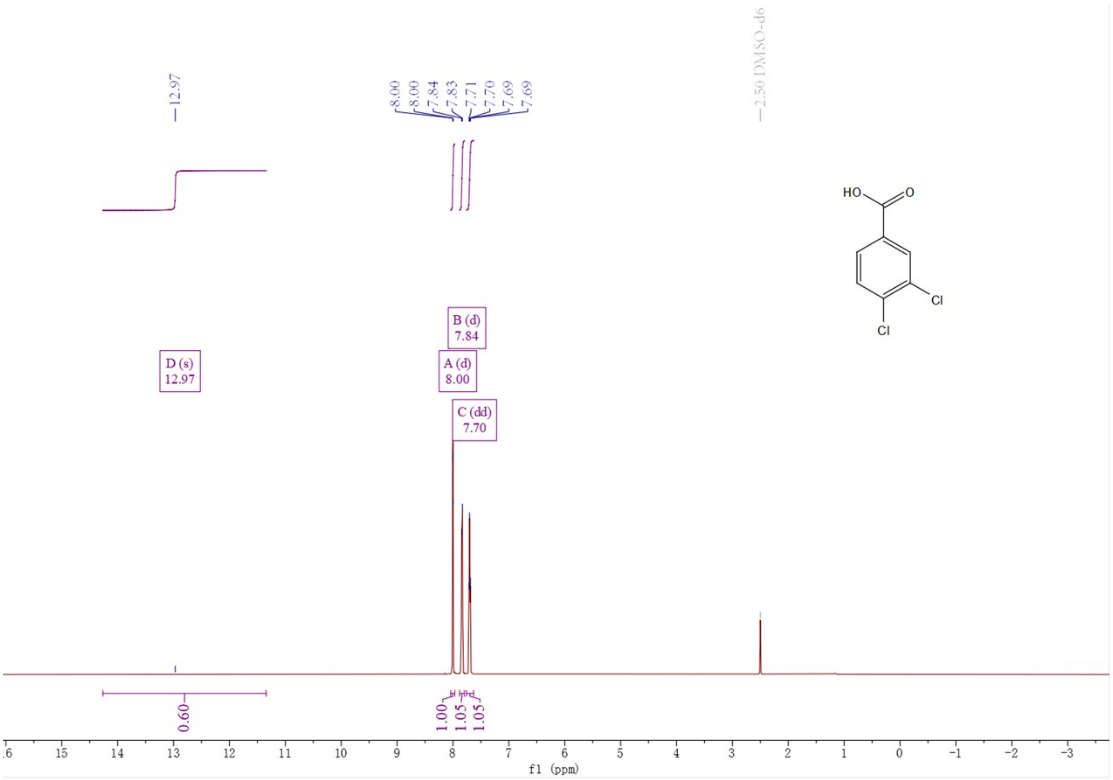

Figure S39 ^{1}H NMR spectrum of compound **2q** (600 MHz, DMSO-d_6)

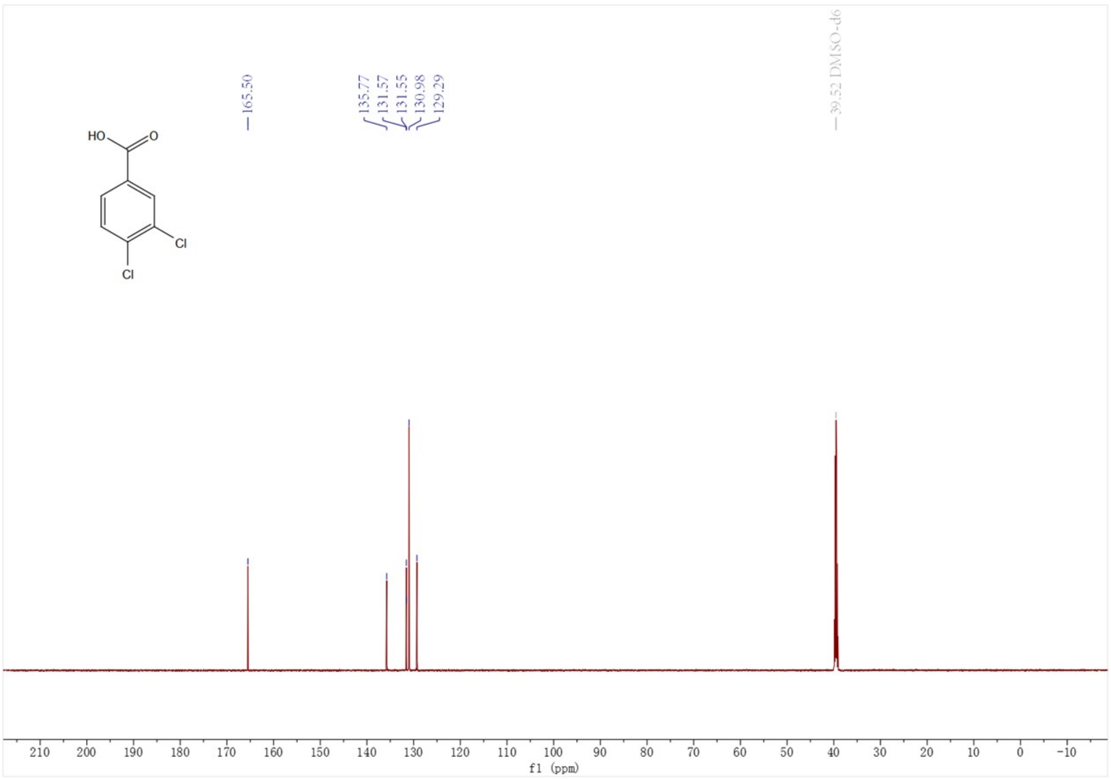

Figure S40 ^{13}C NMR spectrum of compound **2q** (151 MHz, DMSO-d_6)

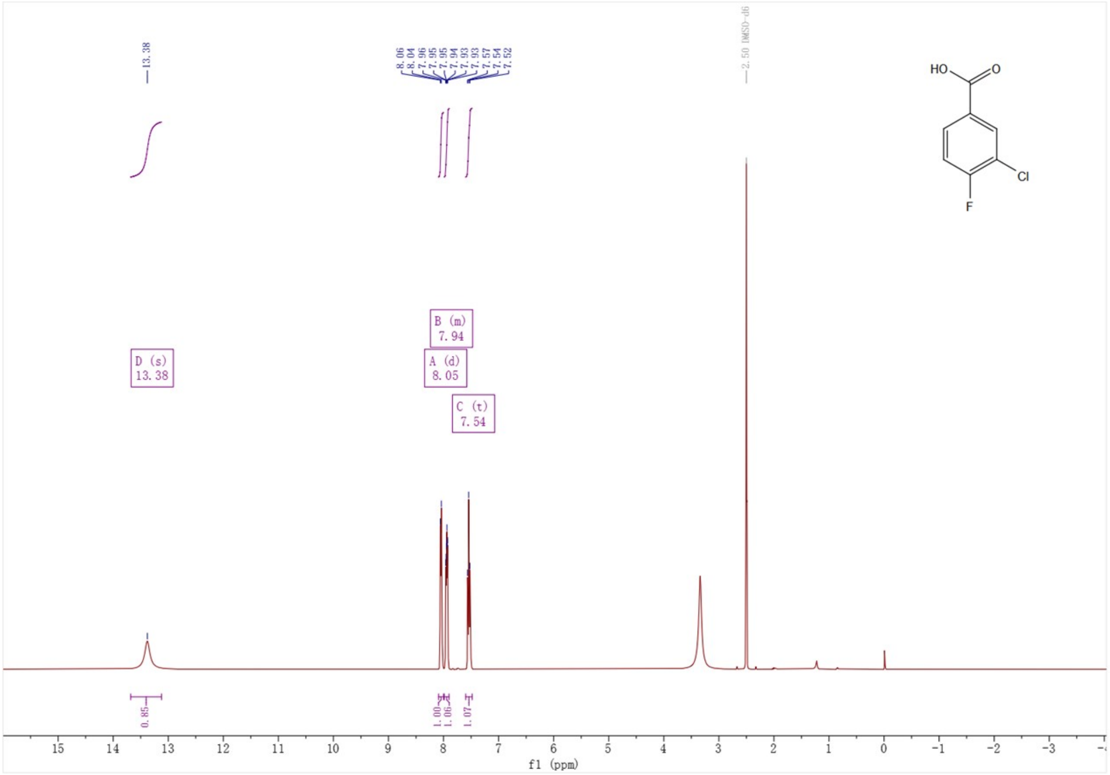

Figure S41 ^{1}H NMR spectrum of compound **2r** (400 MHz, DMSO-d_6)

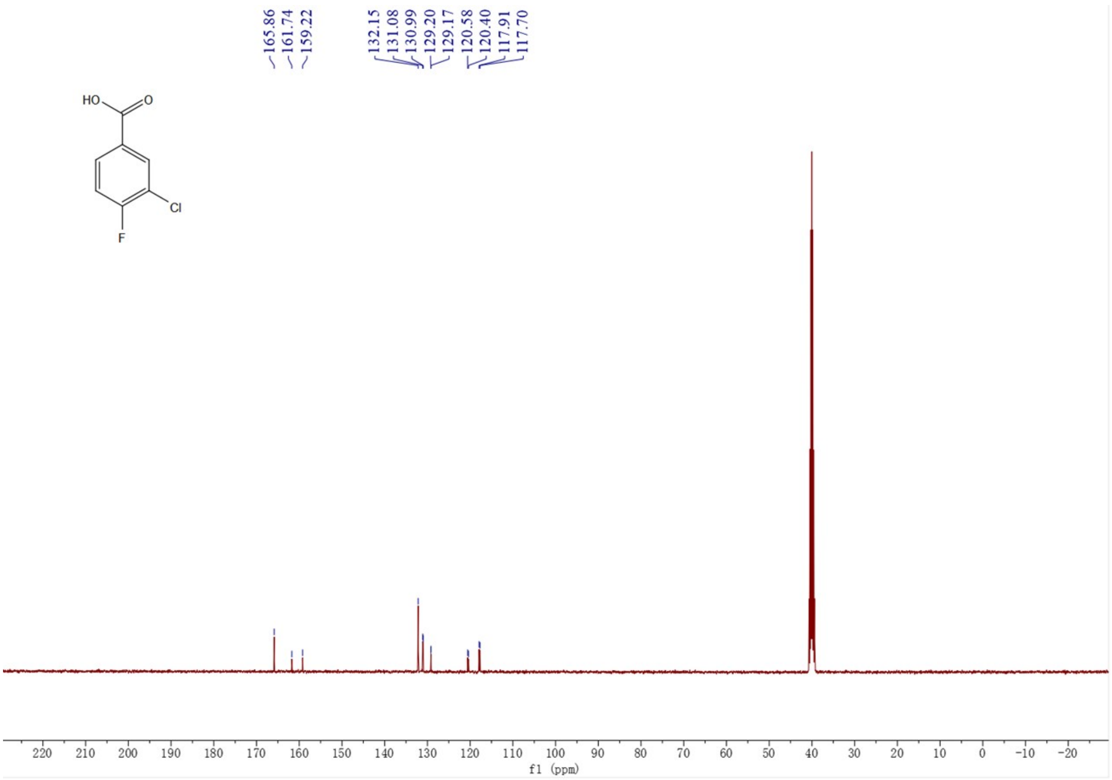

Figure S42 ^{13}C NMR spectrum of compound **2r** (101 MHz, DMSO-d_6)

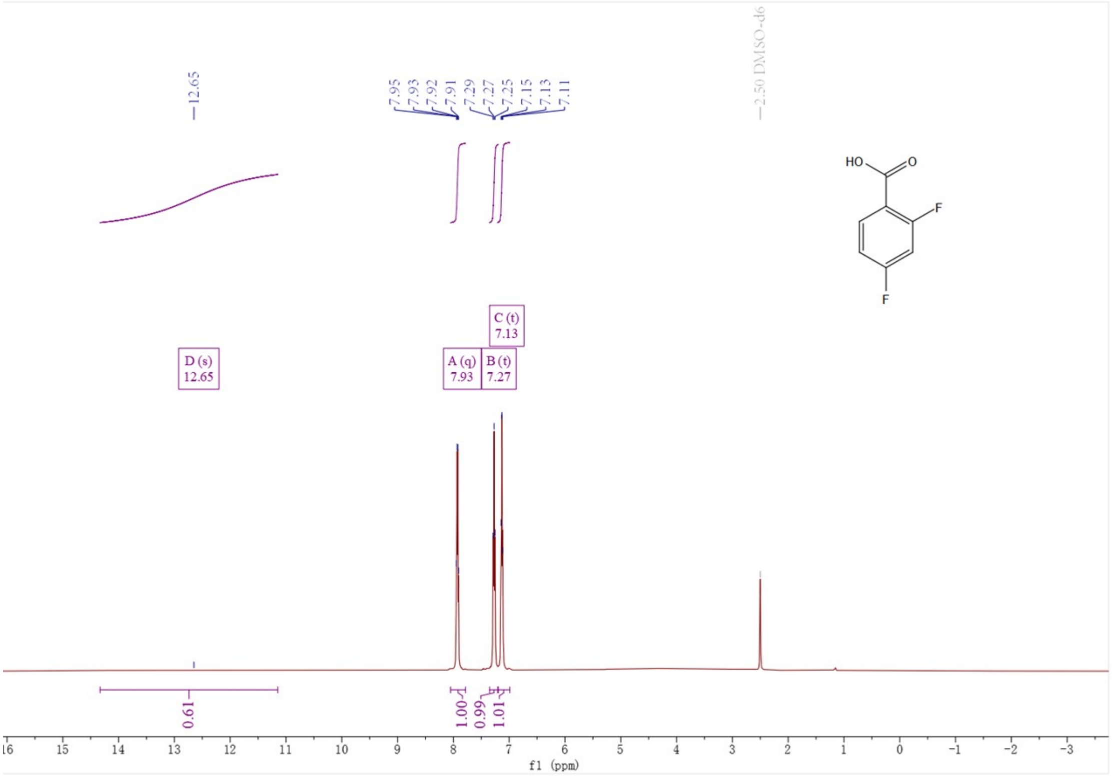

Figure S43 ^{1}H NMR spectrum of compound **2s** (600 MHz, DMSO-d_6)

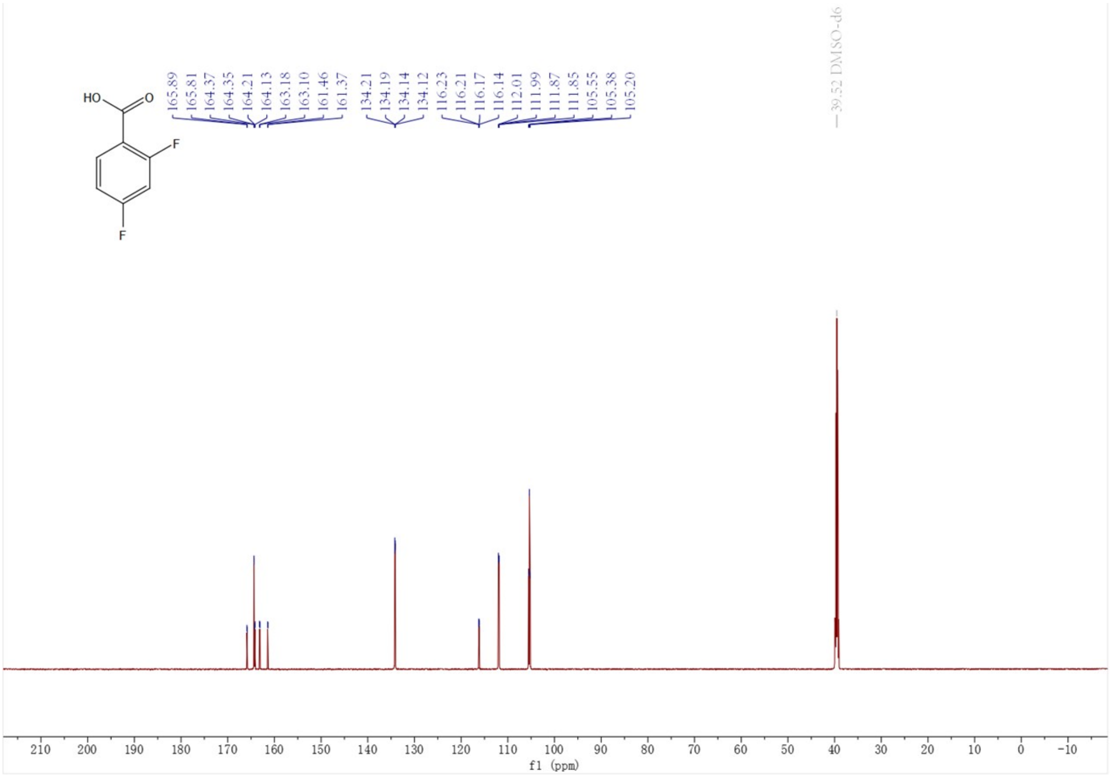

Figure S44 ^{13}C NMR spectrum of compound **2s** (151 MHz, DMSO-d_6)

Figure S45 ^{1}H NMR spectrum of compound **2t** (600 MHz, DMSO-d_6)

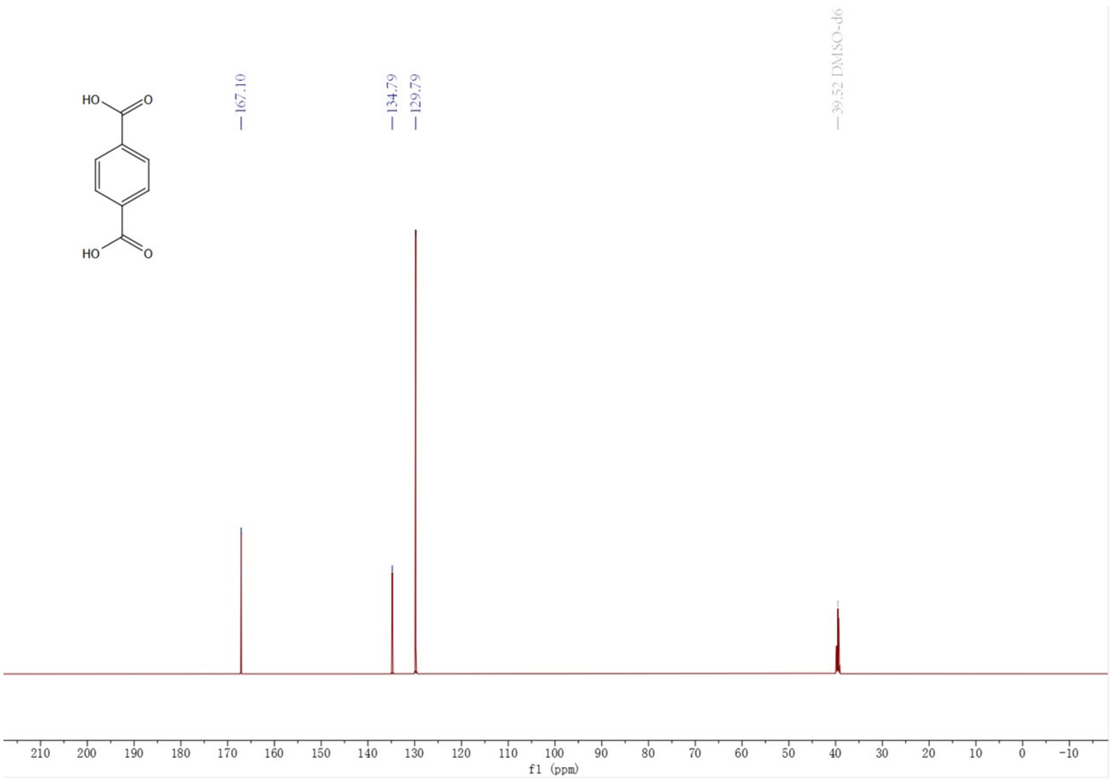

Figure S46 ^{13}C NMR spectrum of compound **2t** (151 MHz, DMSO-d_6)

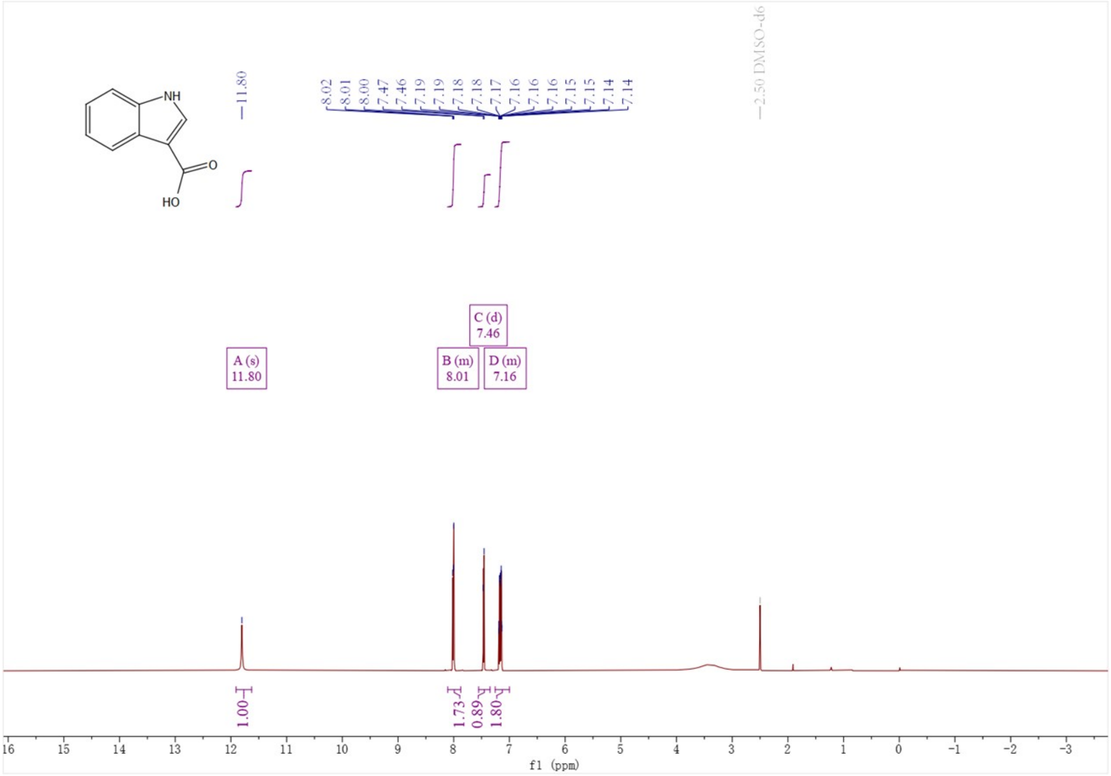

Figure S47 ^{1}H NMR spectrum of compound **2u** (600 MHz, DMSO-d_6)

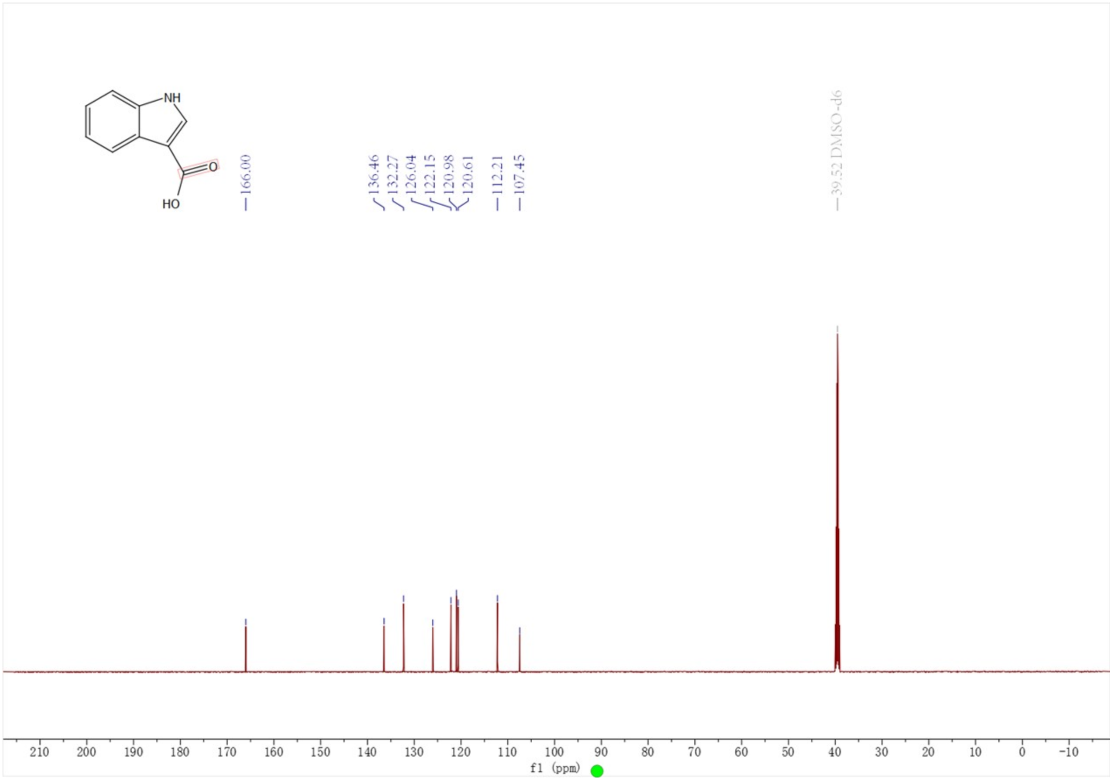

Figure S48 ^{13}C NMR spectrum of compound **2u** (151 MHz, DMSO-d_6)

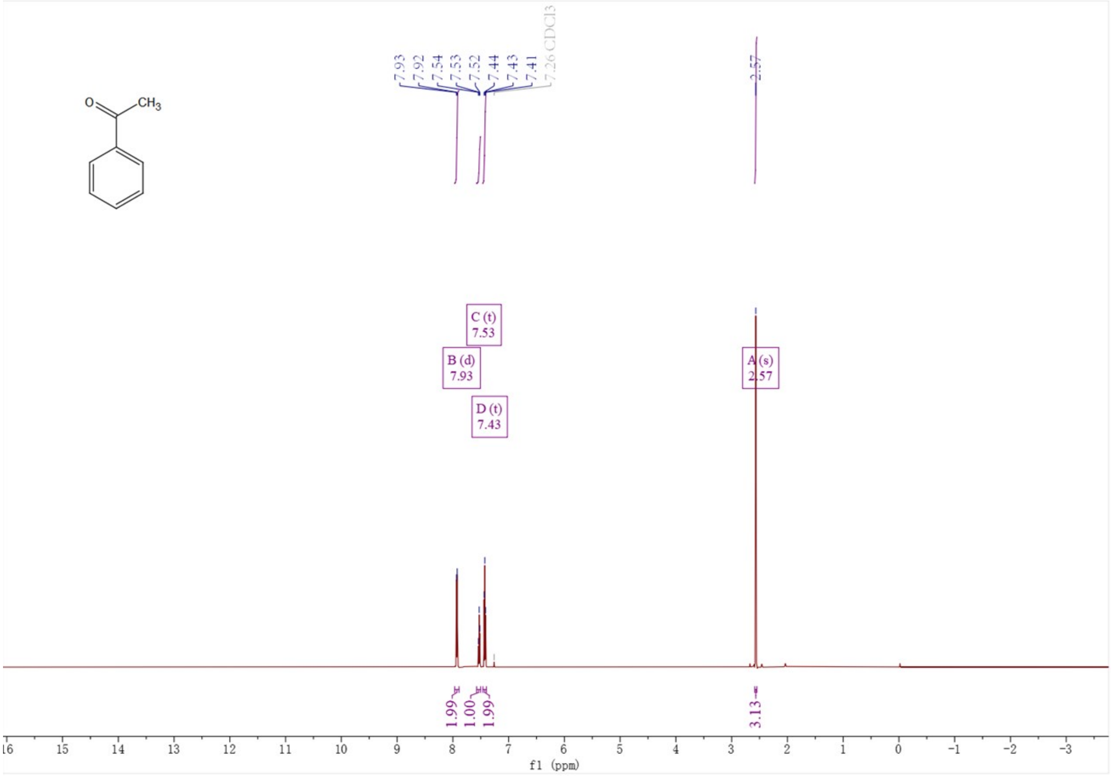

Figure S49 ^{1}H NMR spectrum of compound **4a** ($CDCl_3$, 600 MHz)

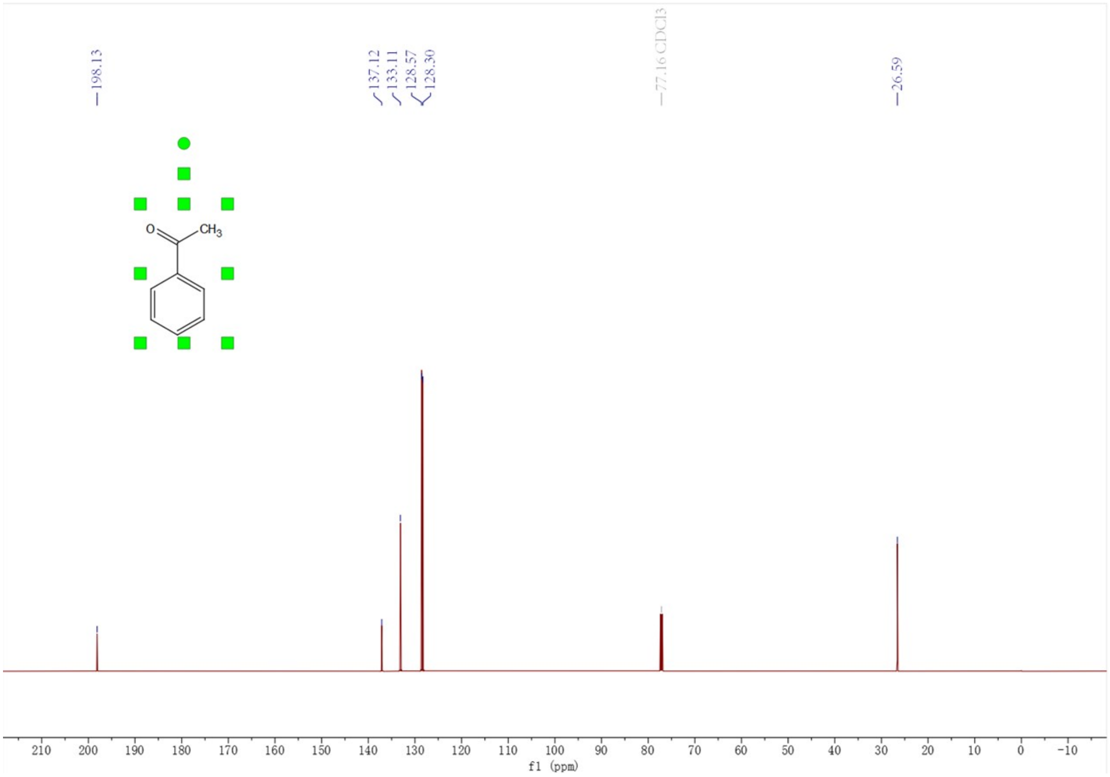

Figure S50 ^{13}C NMR spectrum of compound **4a** ($CDCl_3$, 151 MHz)

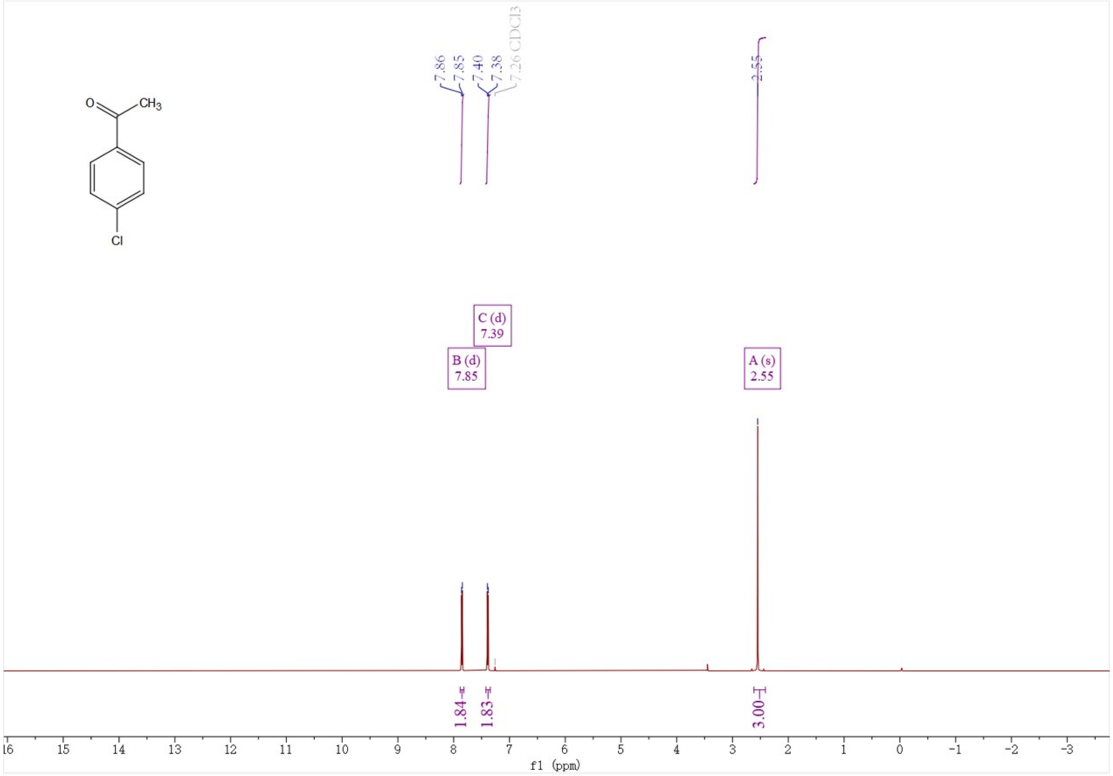

Figure S51 ^{1}H NMR spectrum of compound **4b** ($CDCl_3$, 600 MHz)

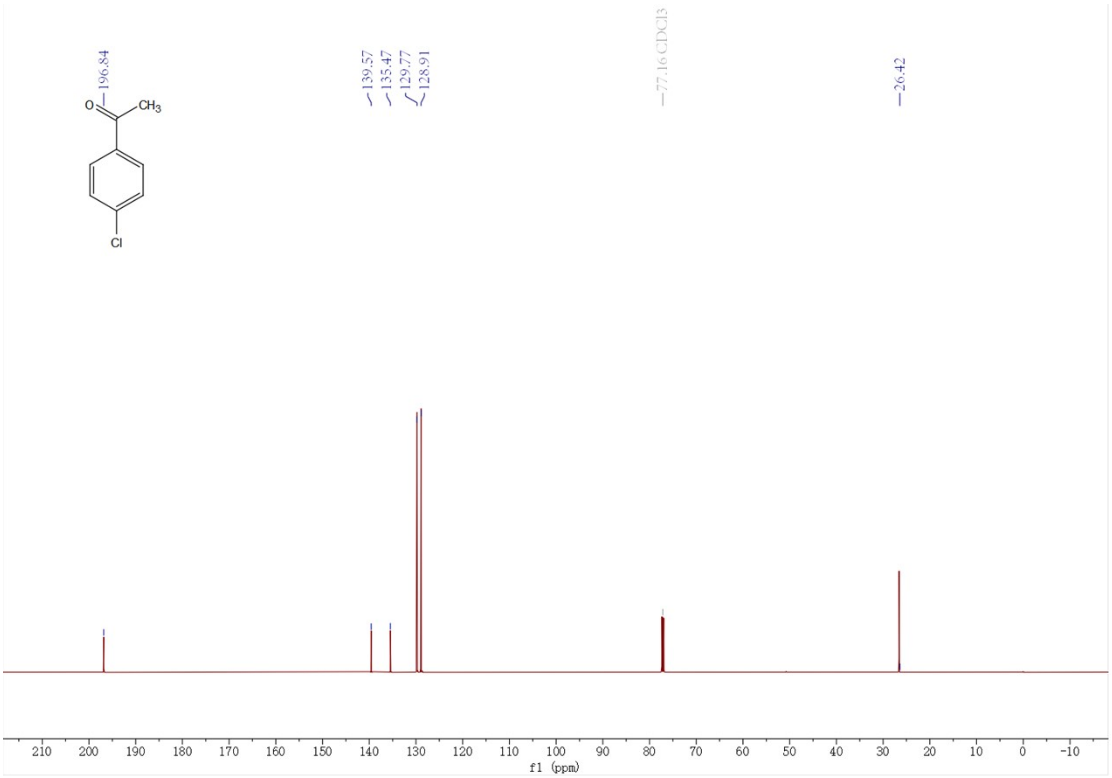

Figure S52 ^{13}C NMR spectrum of compound **4b** ($CDCl_3$, 151 MHz)

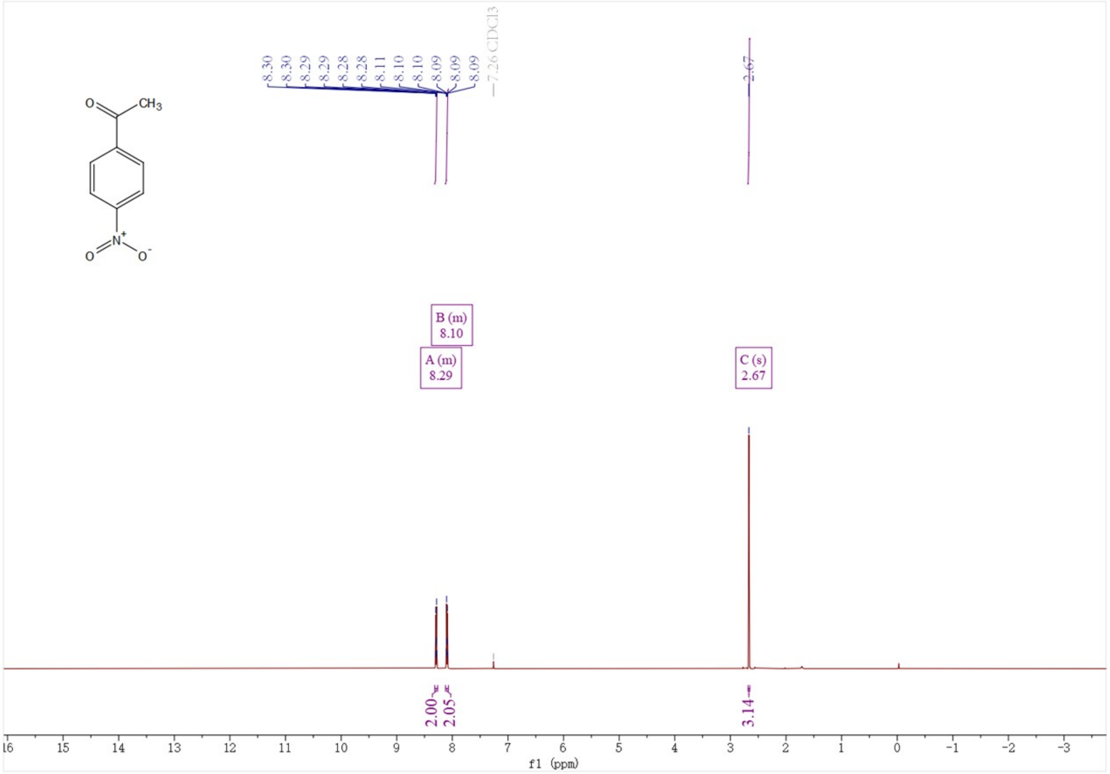

Figure S53 ^{1}H NMR spectrum of compound **4c** ($CDCl_3$, 600 MHz)

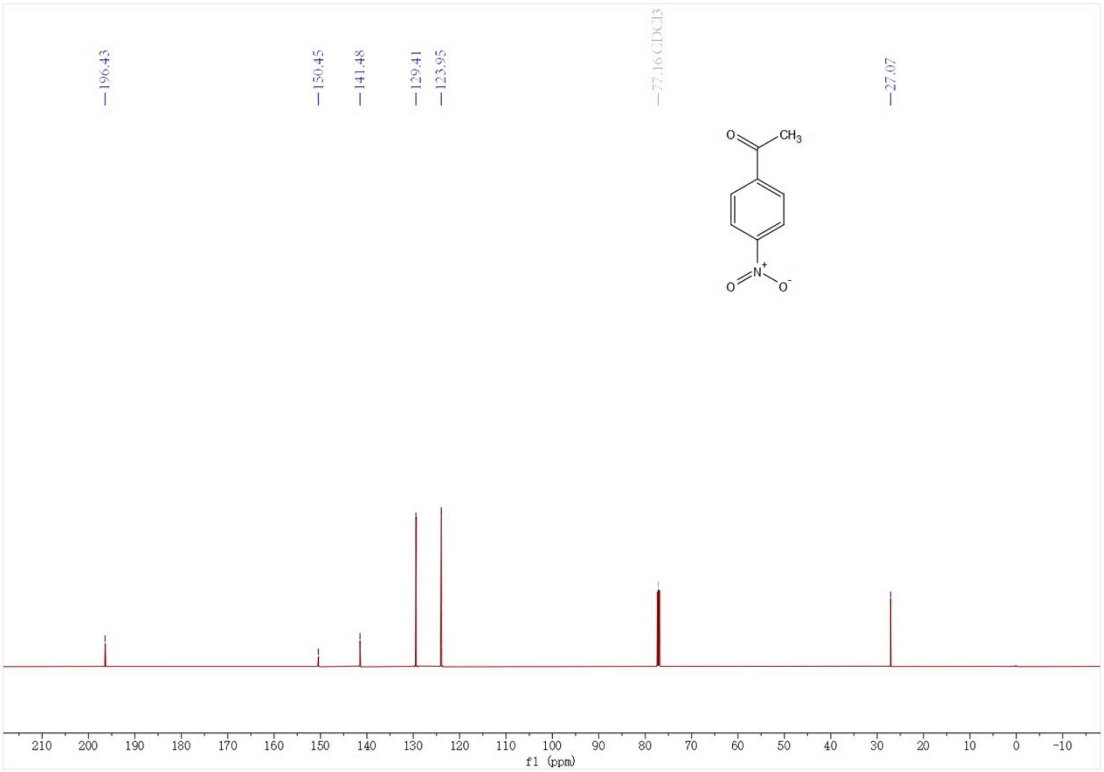

Figure S54 ^{13}C NMR spectrum of compound **4c** ($CDCl_3$, 151 MHz)

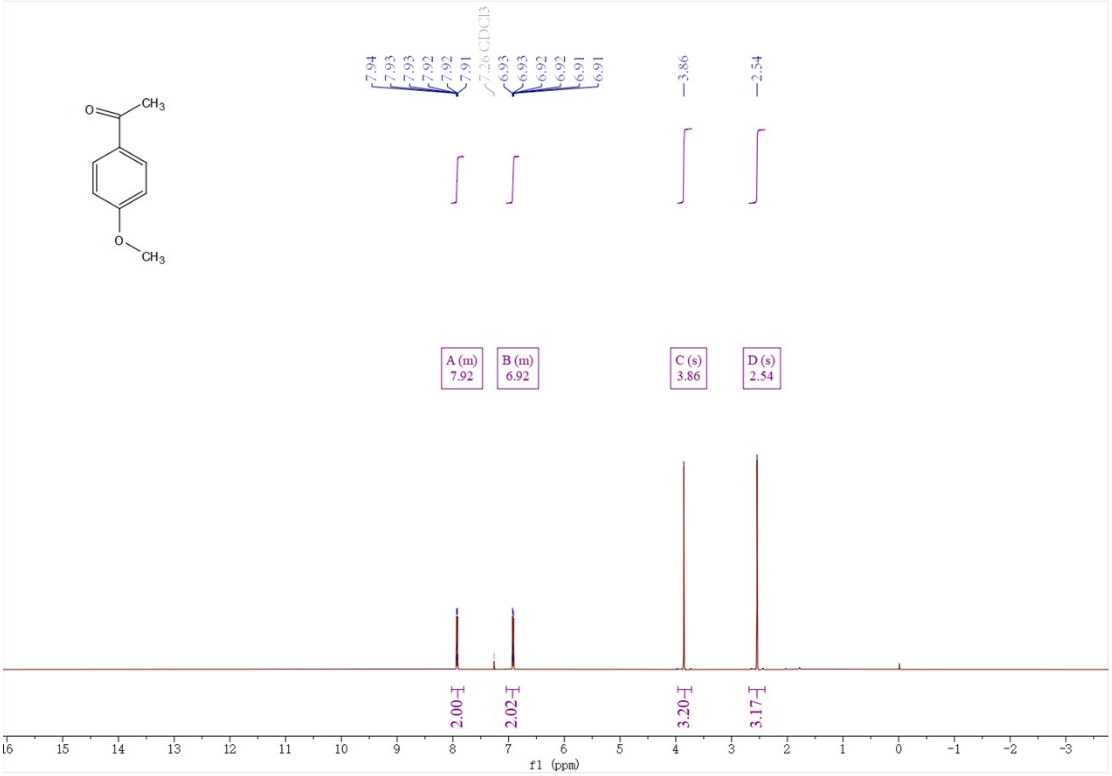

Figure S55 ^{1}H NMR spectrum of compound **4d** ($CDCl_3$, 600 MHz)

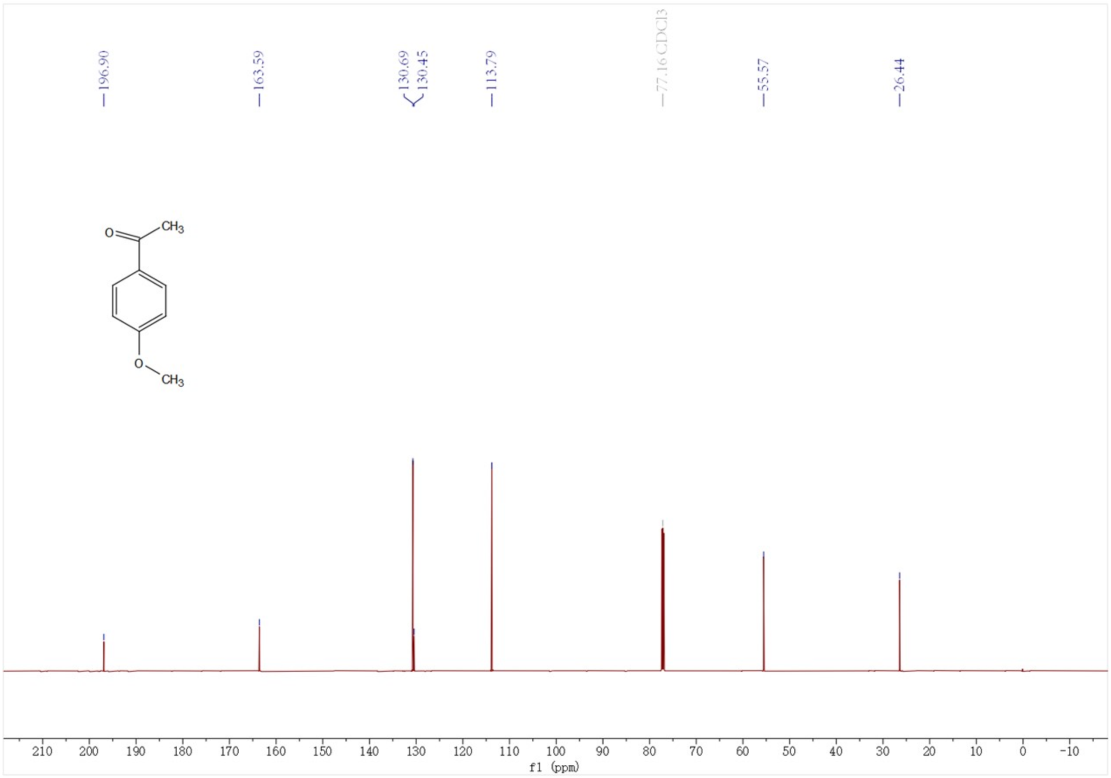

Figure S56 ^{13}C NMR spectrum of compound **4d** ($CDCl_3$, 151 MHz)

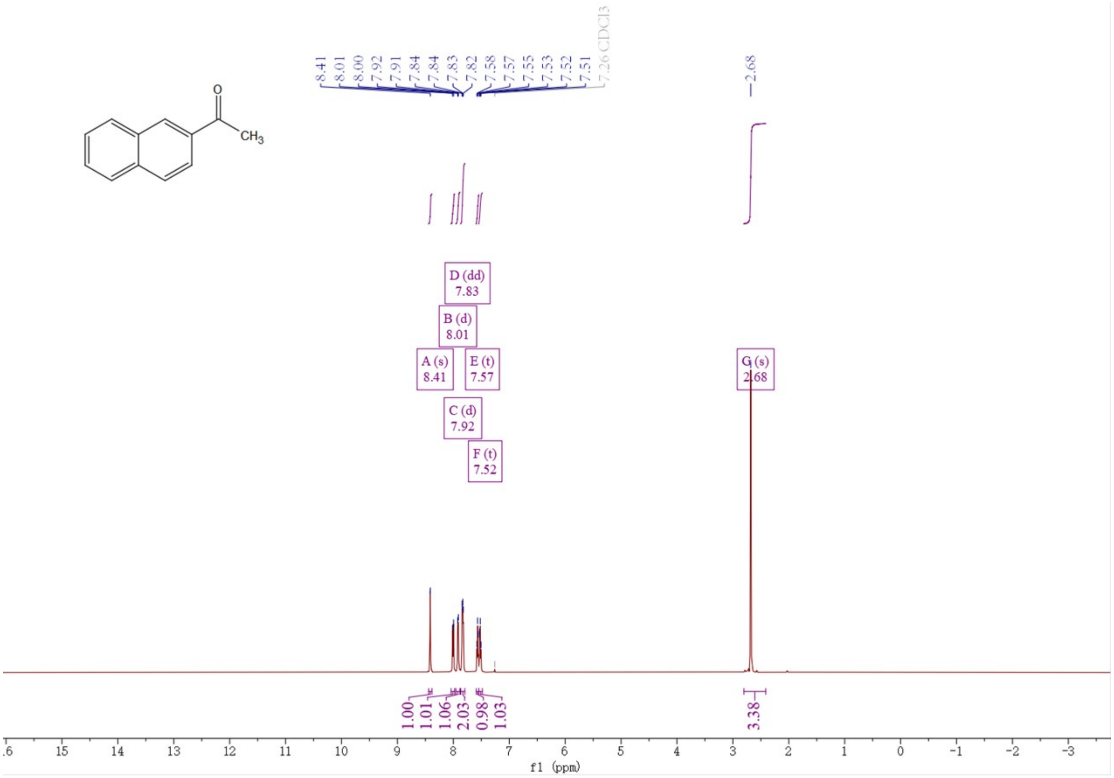

Figure S57 ^{1}H NMR spectrum of compound **4e** ($CDCl_3$, 600 MHz)

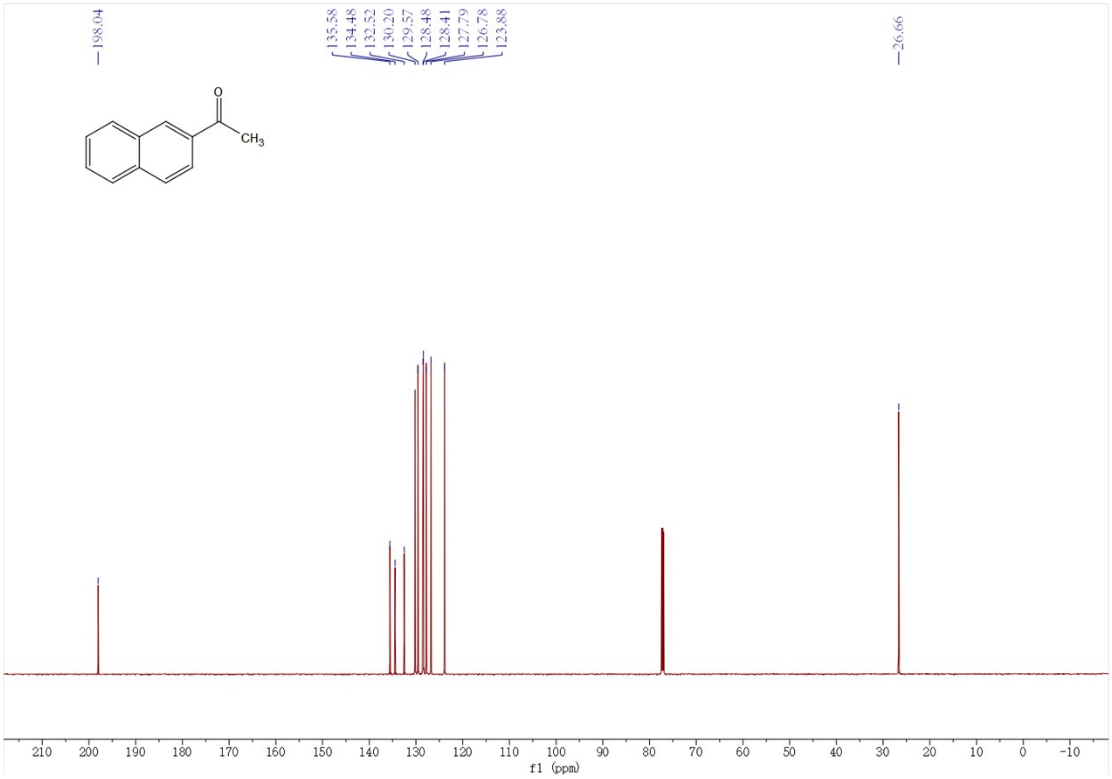

Figure S58 ^{13}C NMR spectrum of compound **4e** ($CDCl_3$, 151 MHz)

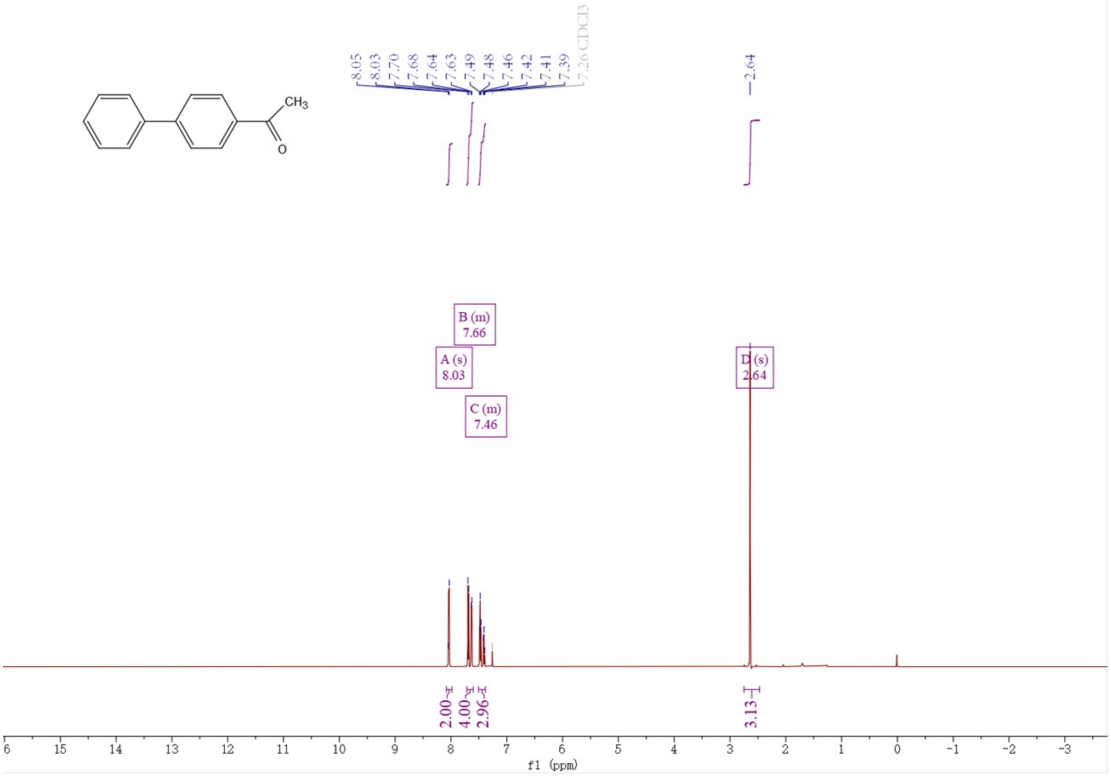

Figure S59 ^{1}H NMR spectrum of compound **4f** ($CDCl_3$, 600 MHz)

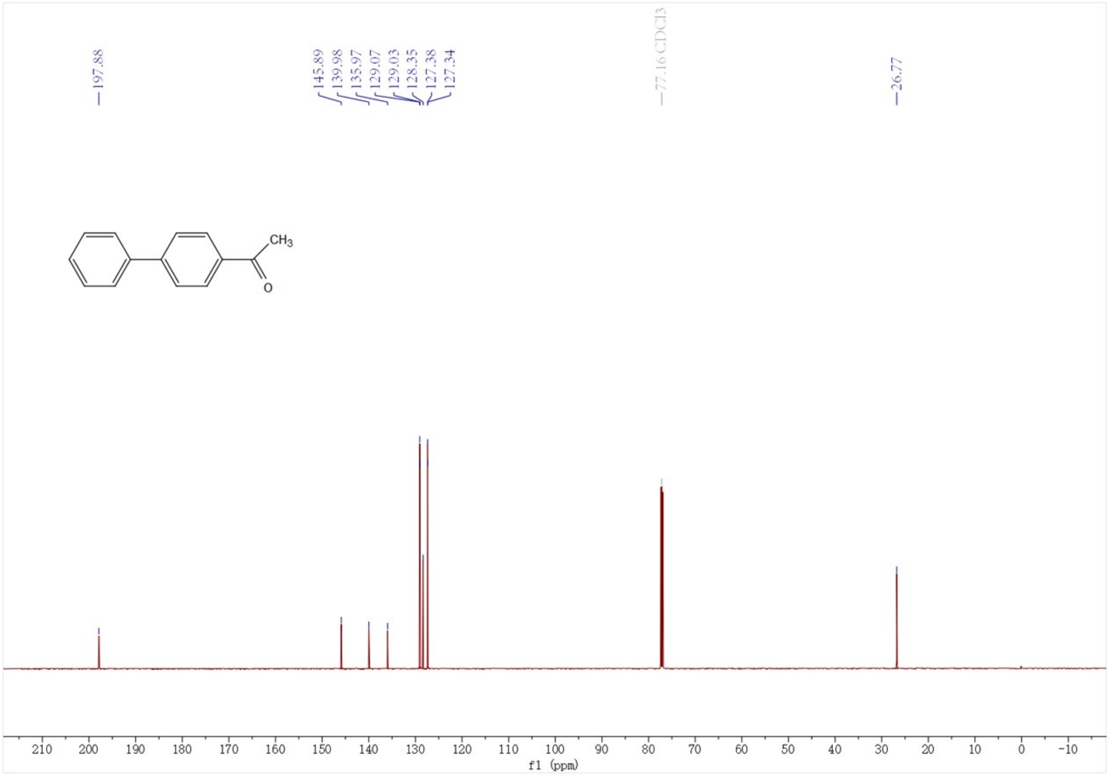

Figure S60 ^{13}C NMR spectrum of compound **4f** ($CDCl_3$, 151 MHz)

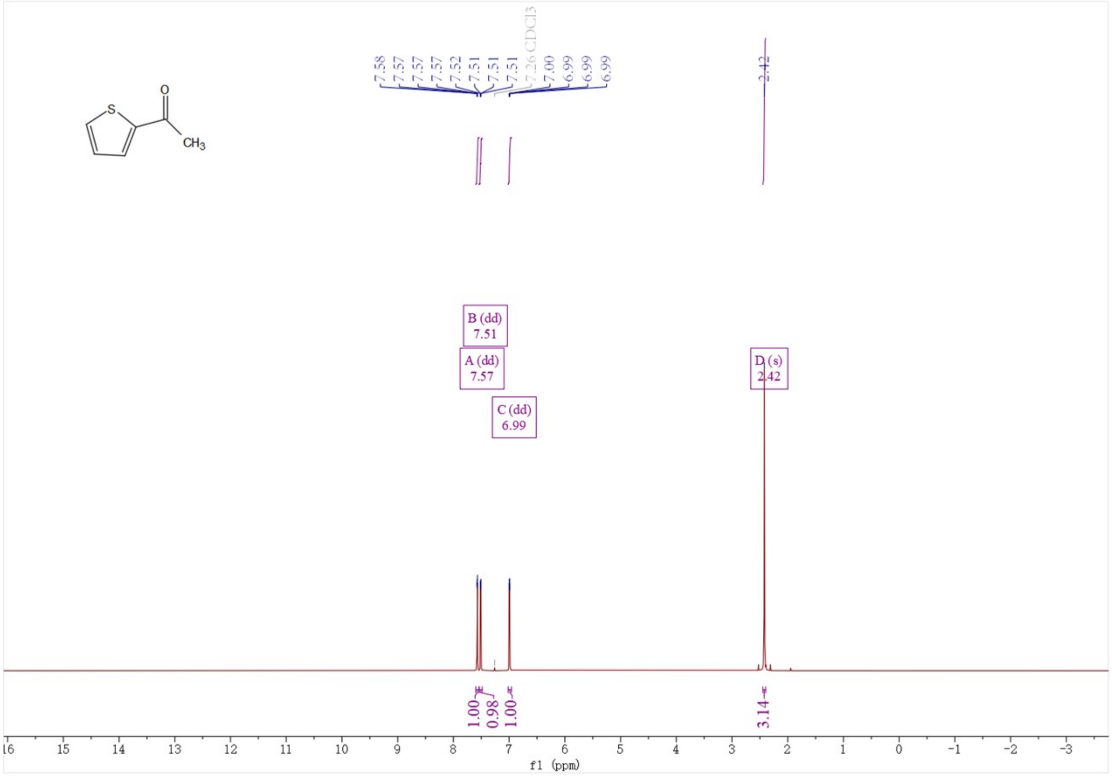

Figure S61 ^{1}H NMR spectrum of compound **4g** ($CDCl_3$, 600 MHz)

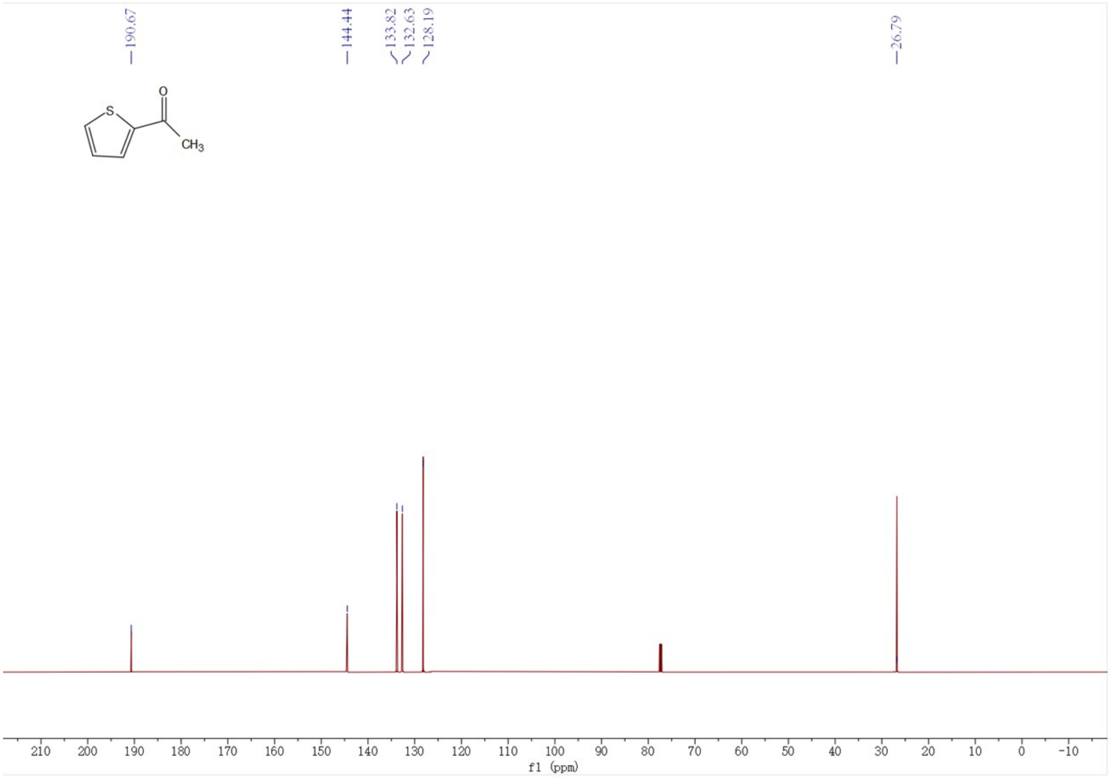

Figure S62 ^{13}C NMR spectrum of compound **4g** ($CDCl_3$, 151 MHz)

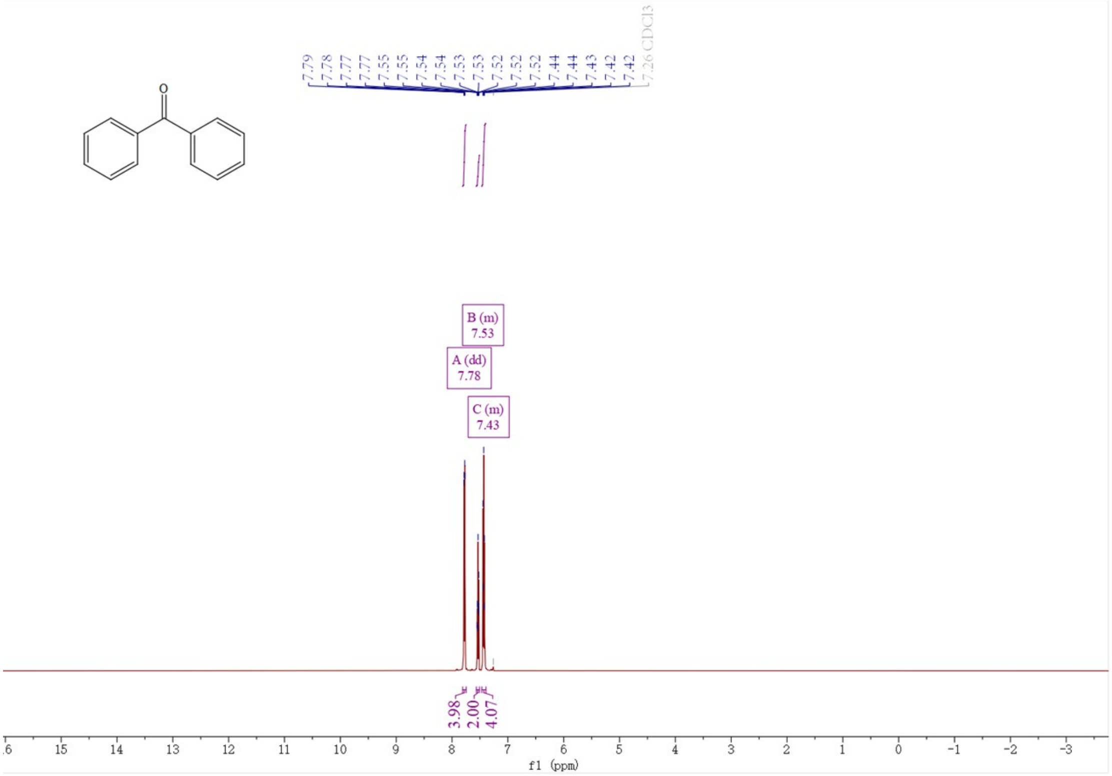

Figure S63 ^{1}H NMR spectrum of compound **4h** ($CDCl_3$, 600 MHz)

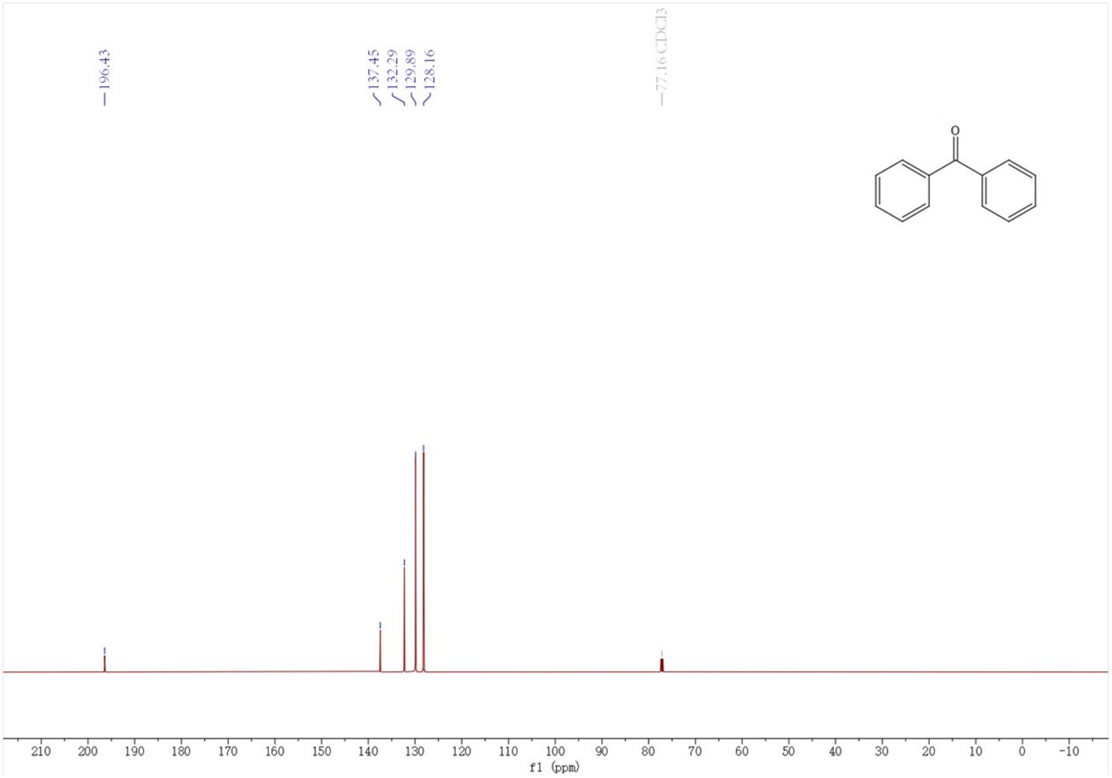

Figure S64 ^{13}C NMR spectrum of compound **4h** ($CDCl_3$, 151 MHz)

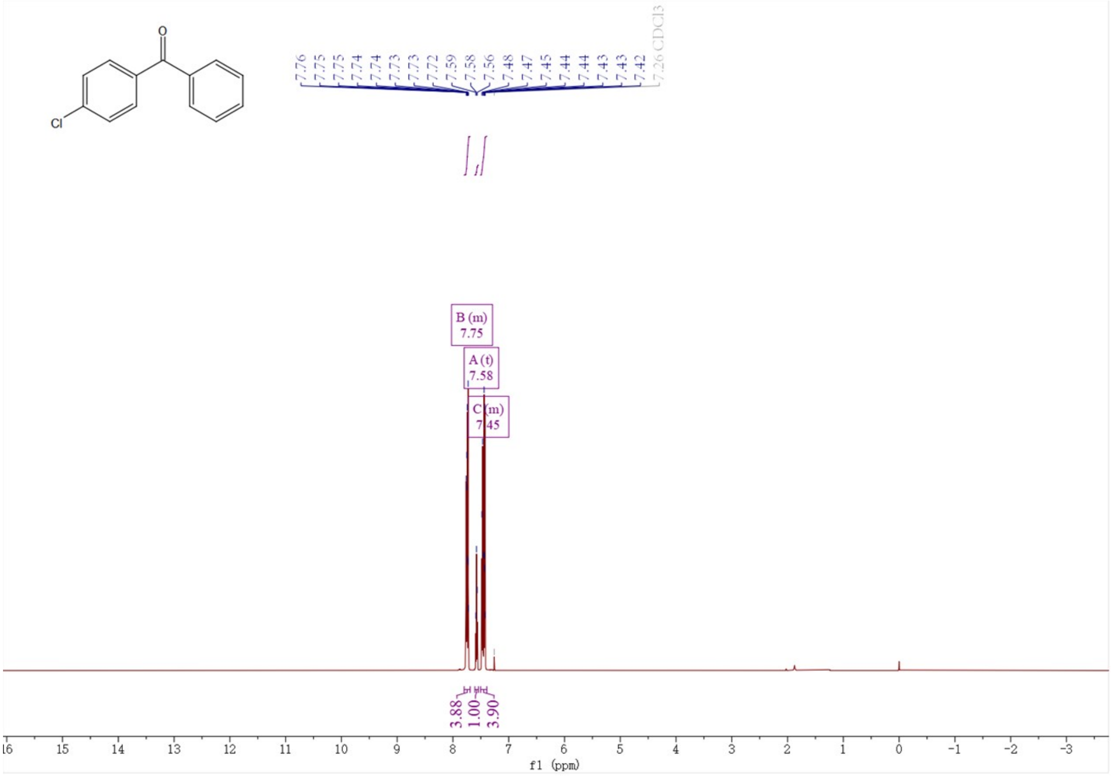

Figure S65 ^{1}H NMR spectrum of compound **4i** ($CDCl_3$, 600 MHz)

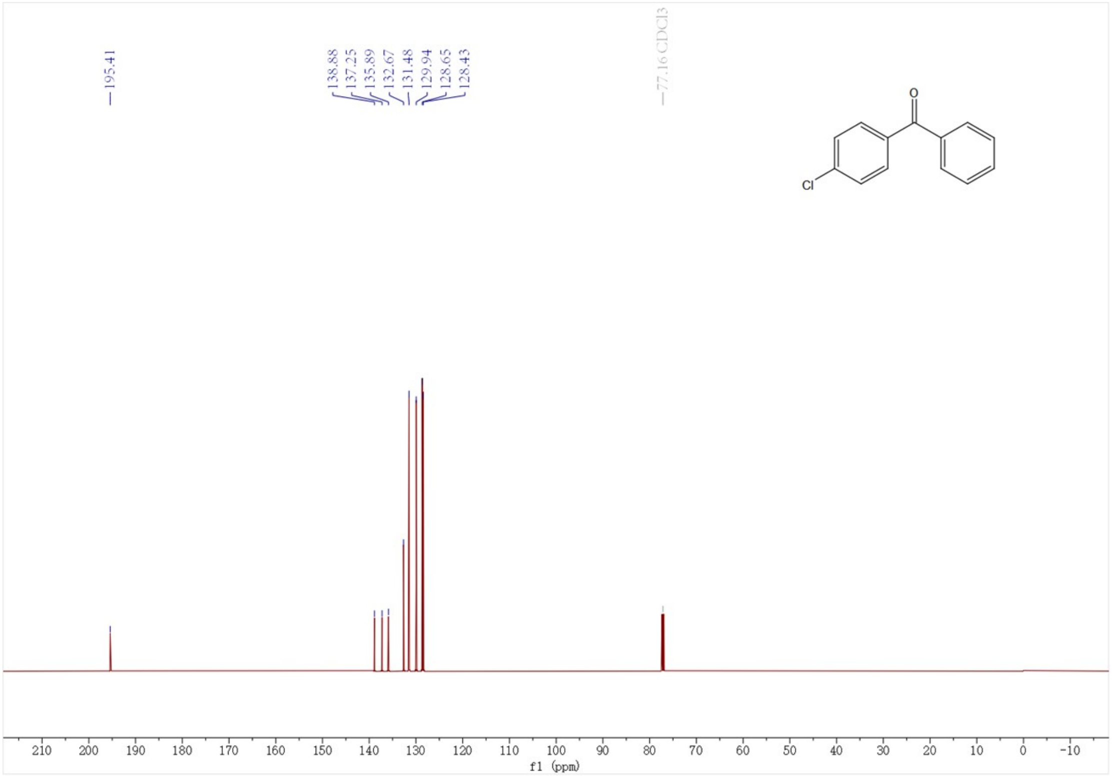

Figure S66 ^{13}C NMR spectrum of compound **4i** ($CDCl_3$, 151 MHz)

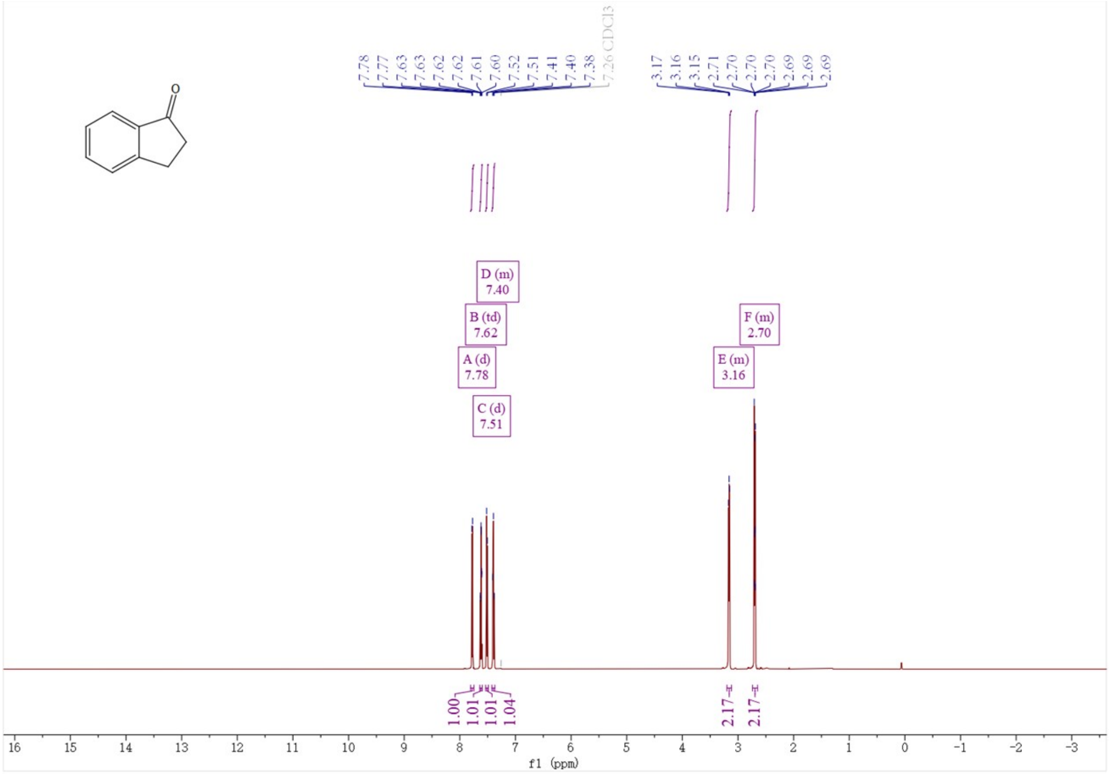

Figure S67 ^{1}H NMR spectrum of compound **4j** ($CDCl_3$, 600 MHz)

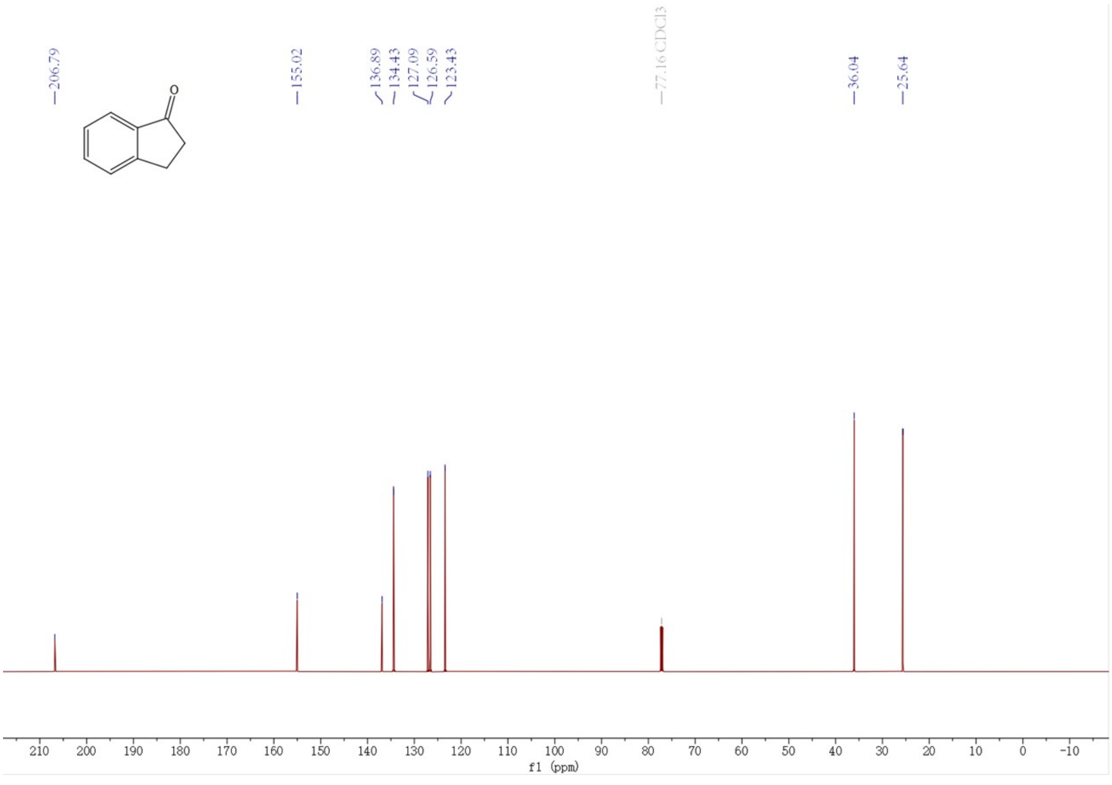

Figure S68 ^{13}C NMR spectrum of compound **4j** ($CDCl_3$, 151 MHz)

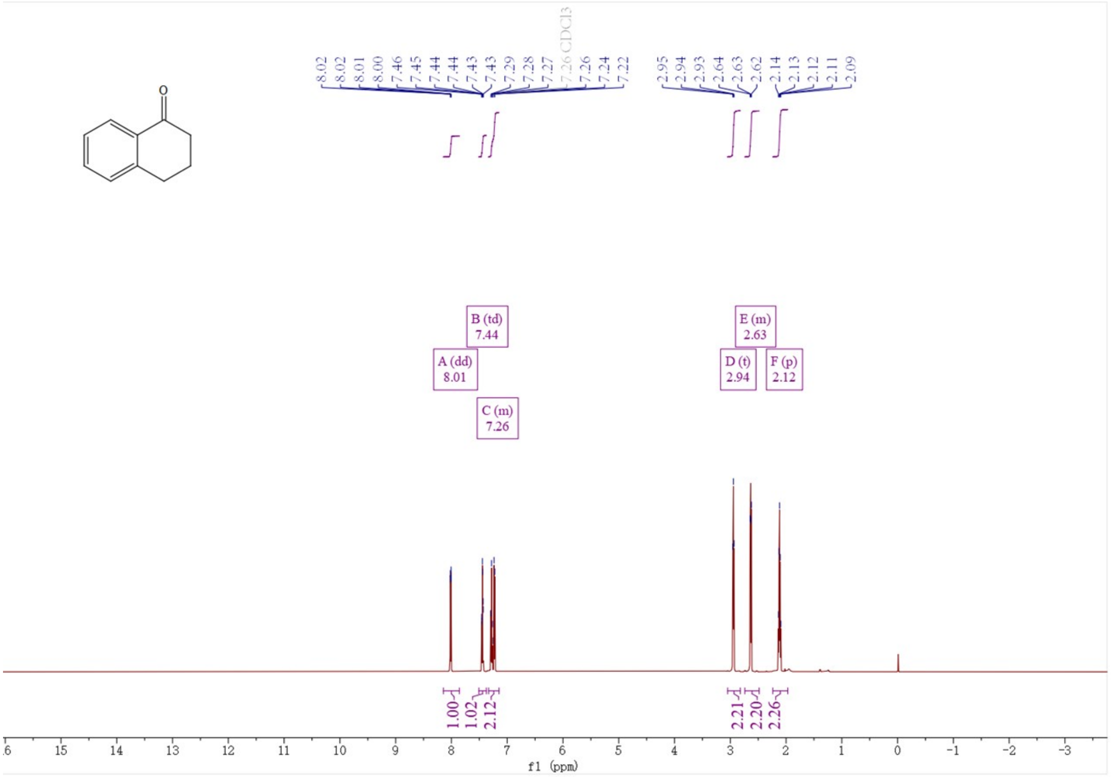

Figure S69 ^{1}H NMR spectrum of compound **4k** ($CDCl_3$, 600 MHz)

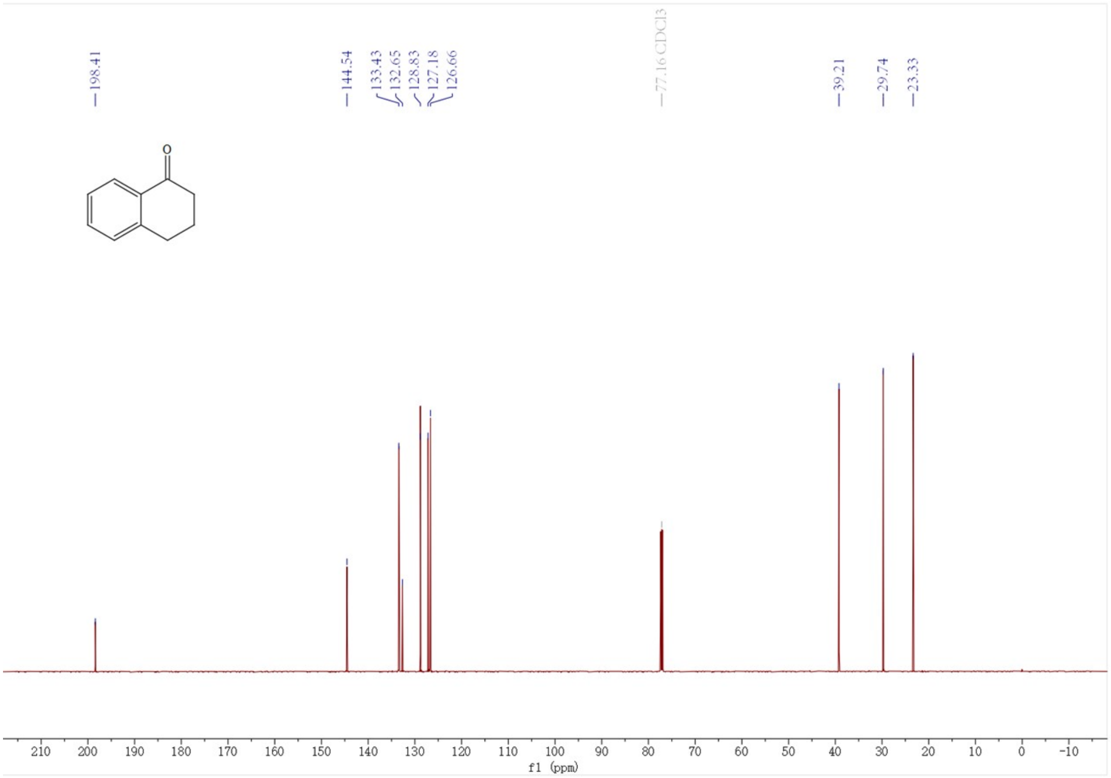

Figure S70 ^{13}C NMR spectrum of compound **4k** ($CDCl_3$, 151 MHz)

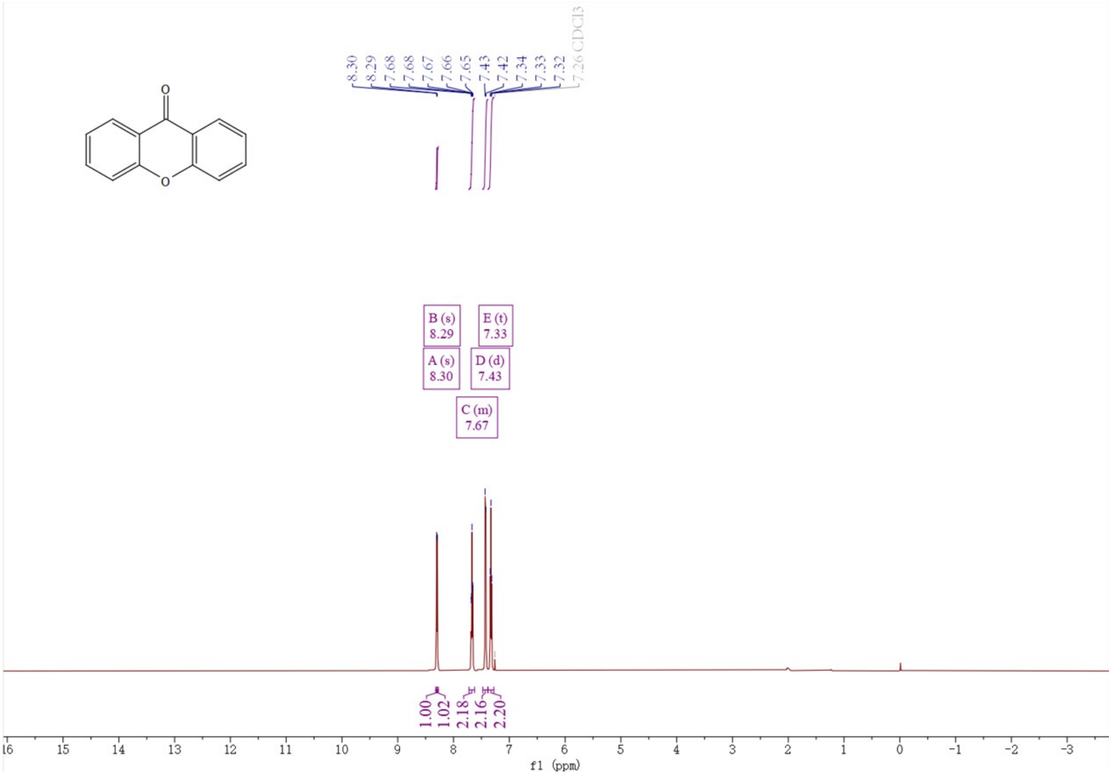

Figure S71 ^{1}H NMR spectrum of compound **4l** ($CDCl_3$, 600 MHz)

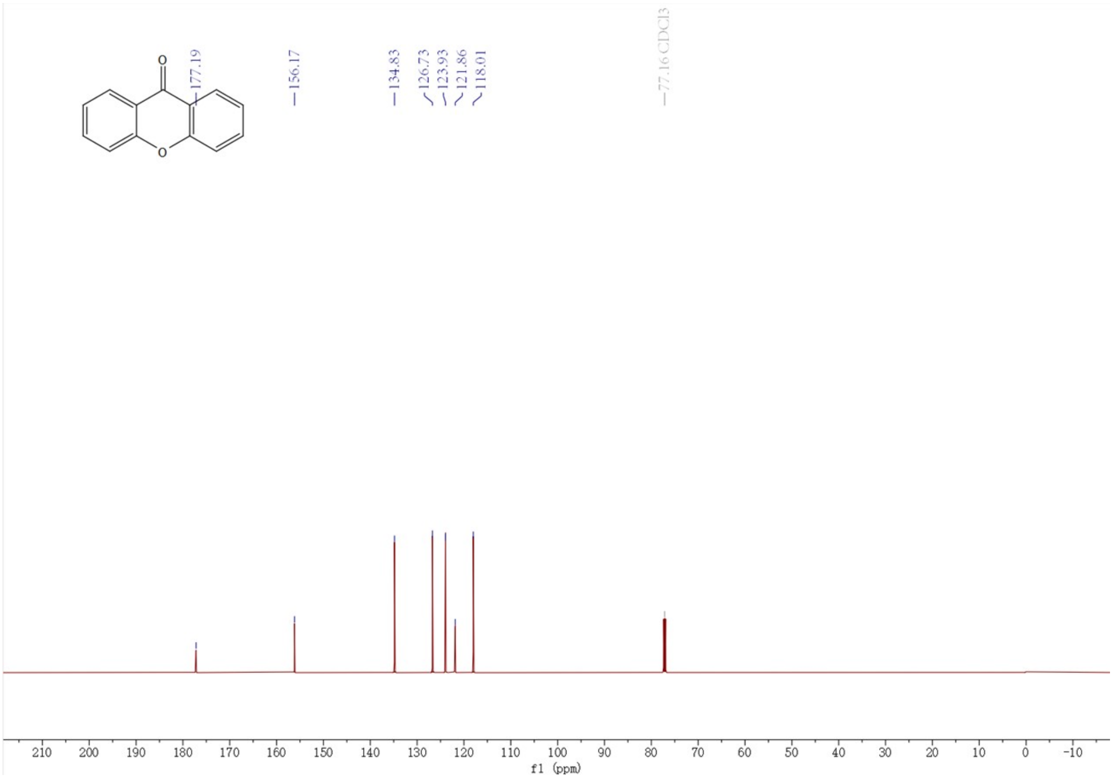

Figure S72 ^{13}C NMR spectrum of compound **4l** ($CDCl_3$, 151 MHz)

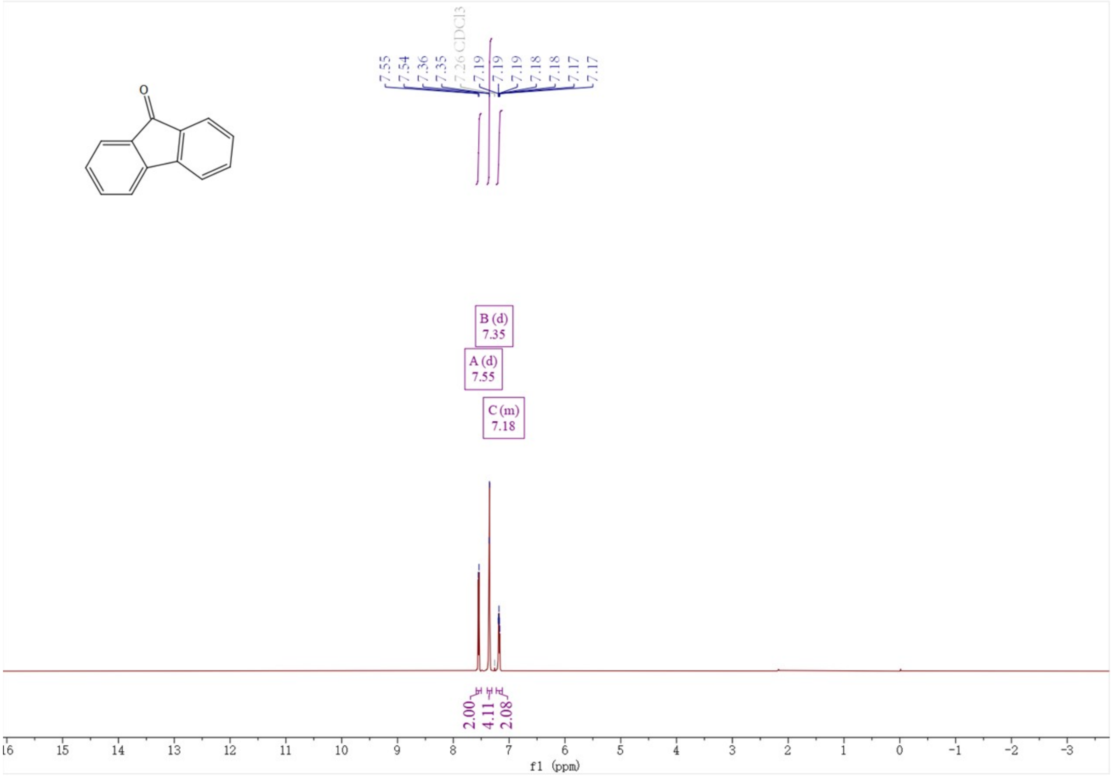

Figure S73 ^{1}H NMR spectrum of compound **4m** ($CDCl_3$, 600 MHz)

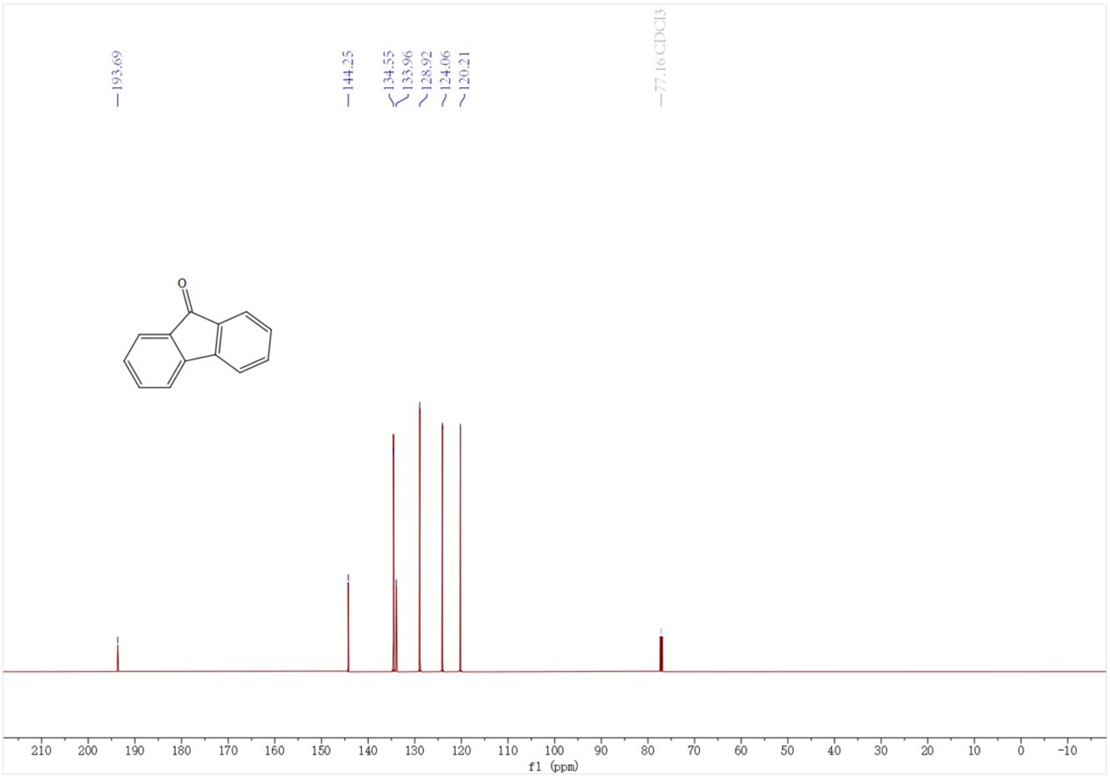

Figure S74 ^{13}C NMR spectrum of compound **4m** ($CDCl_3$, 151 MHz)

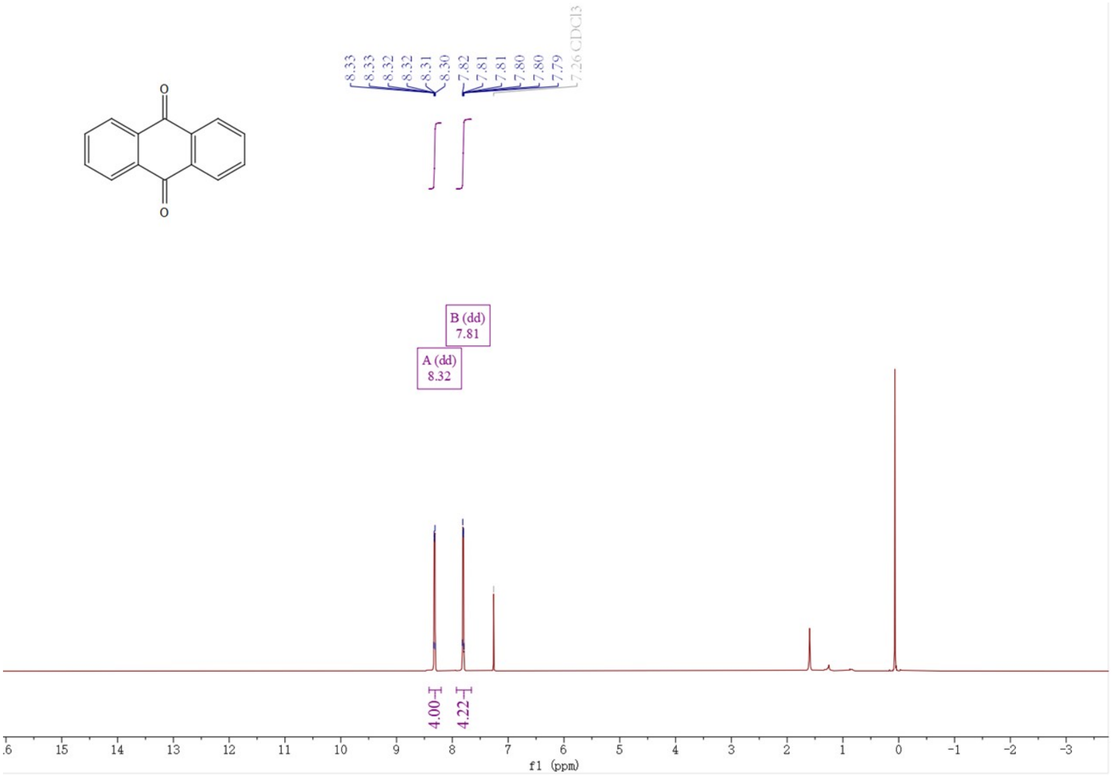

Figure S75 ^{1}H NMR spectrum of compound **4n** ($CDCl_3$, 600 MHz)

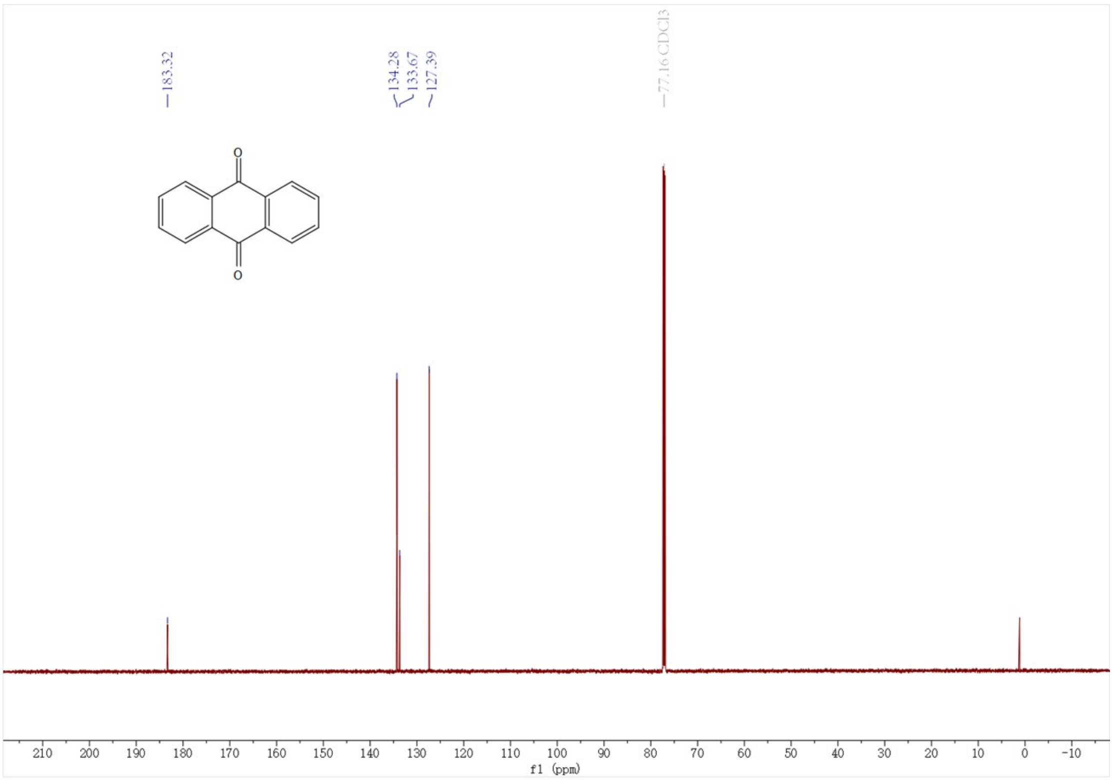

Figure S76 ^{13}C NMR spectrum of compound **4n** ($CDCl_3$, 151 MHz)

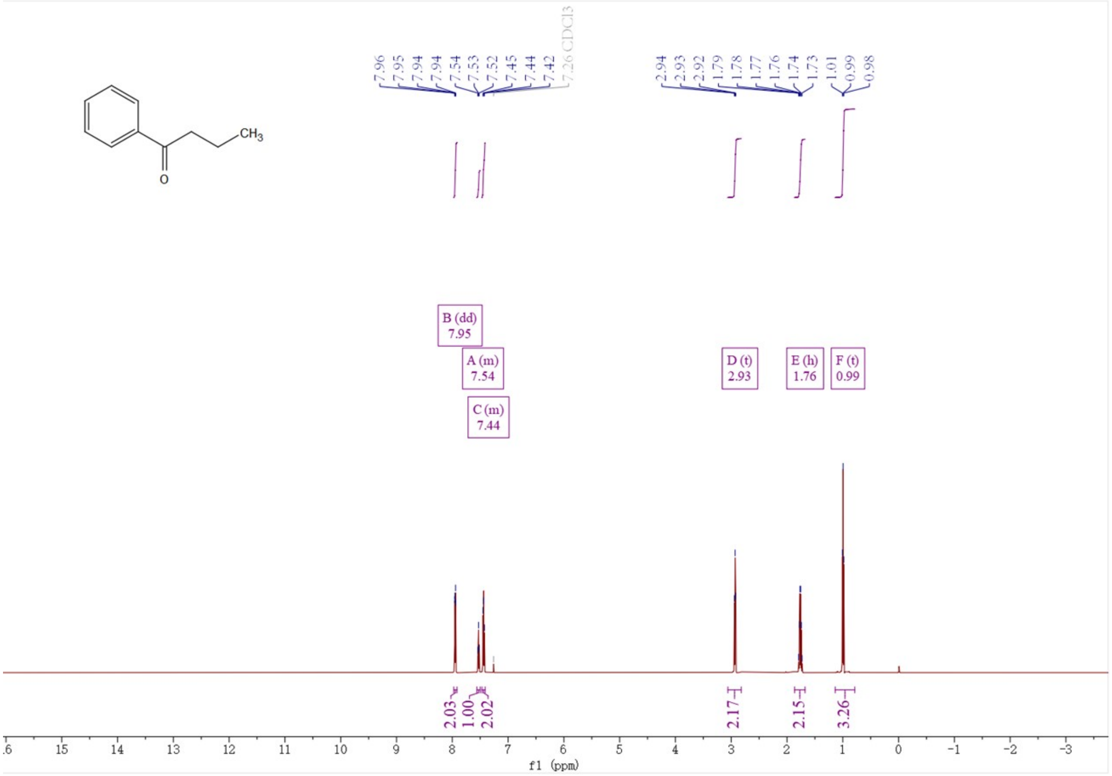

Figure S77 ^{1}H NMR spectrum of compound **4o** ($CDCl_3$, 600 MHz)

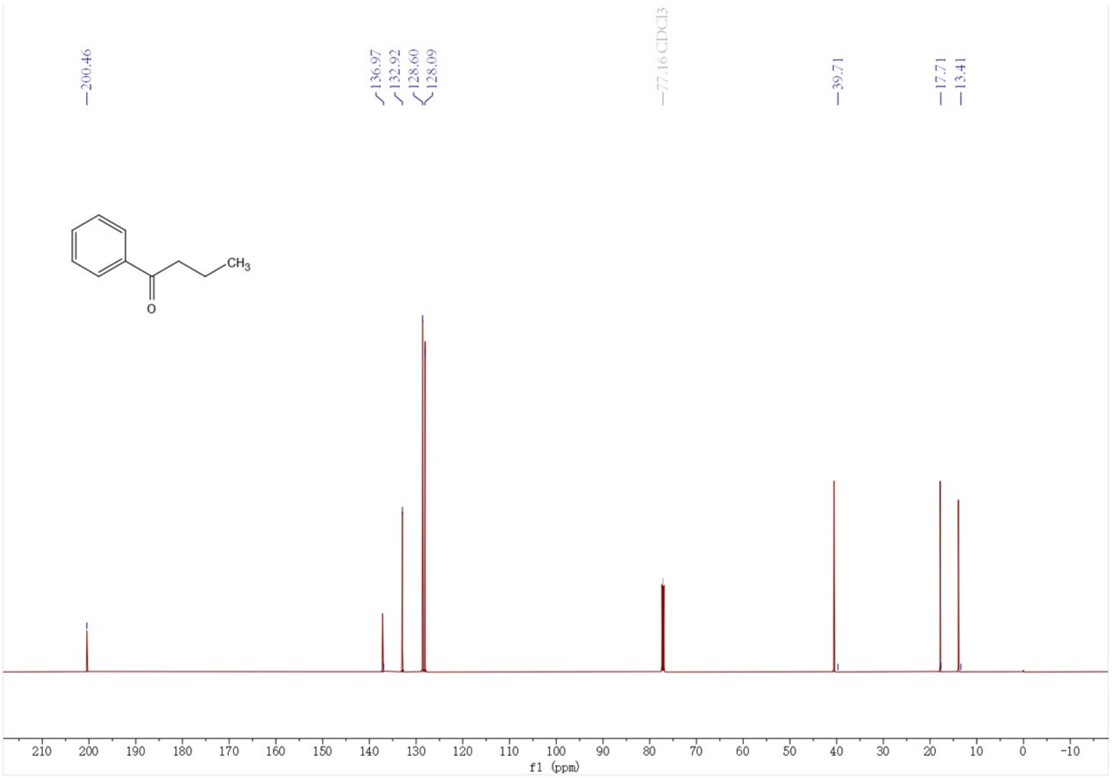

Figure S78 ^{13}C NMR spectrum of compound **4o** ($CDCl_3$, 151 MHz)

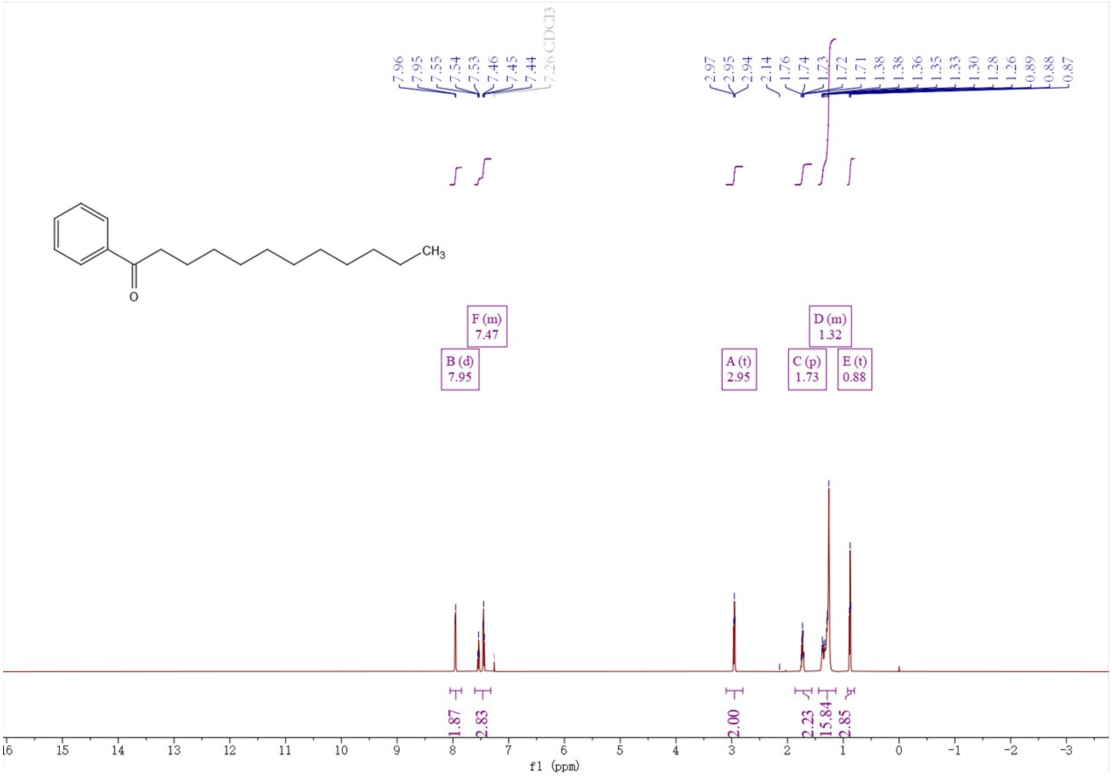

Figure S79 ^{1}H NMR spectrum of compound **4p** ($CDCl_3$, 600 MHz)

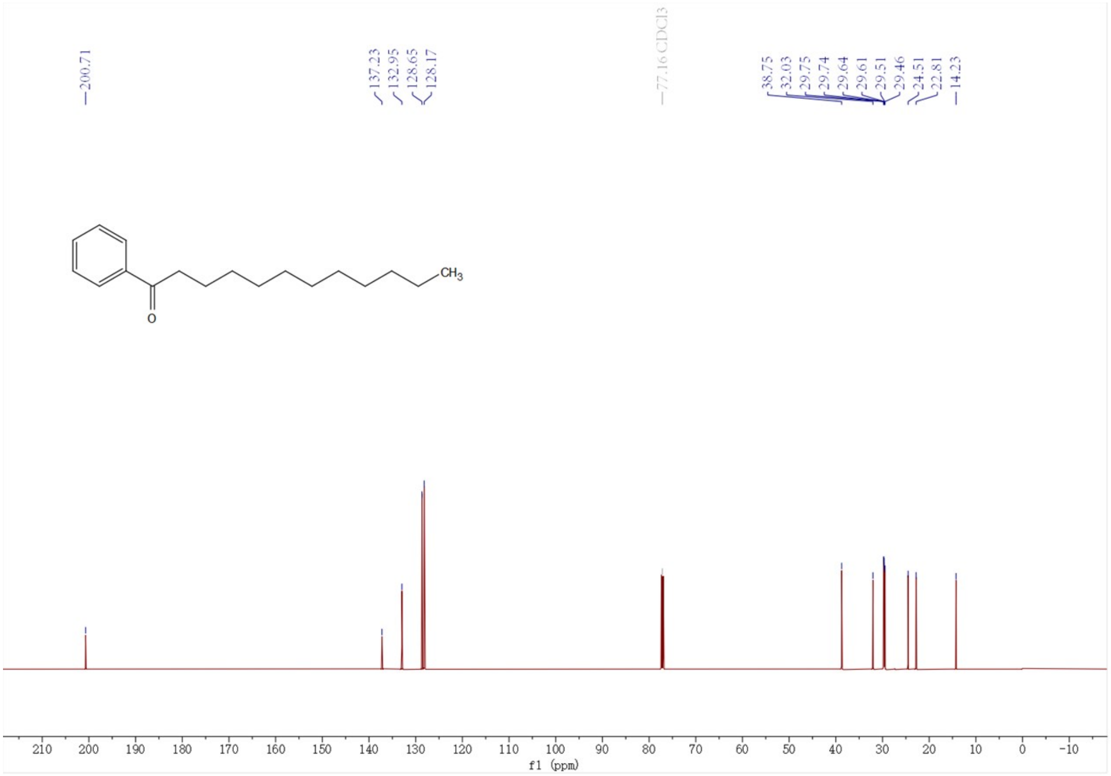

Figure S80 ^{13}C NMR spectrum of compound **4p** ($CDCl_3$, 151 MHz)