Supporting Information

Visible-Light-Induced Dearomatization Oxamination of Indoles and Dearomatization Amidation of Phenols

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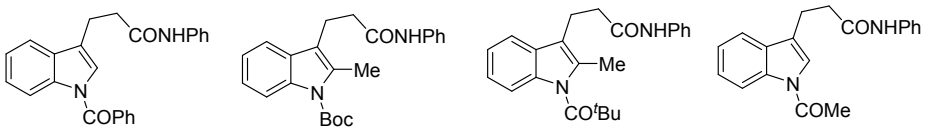
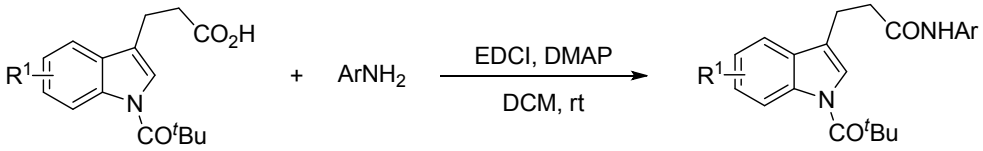
1. Unsuccessful substrates

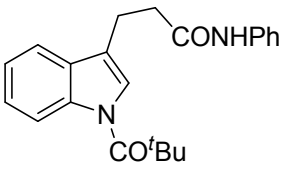


2. General procedure for the preparation of compounds 1 or 4.



A mixture of 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI, 1.04g, 6.5 mmol, 1.3 equiv), DMAP (0.92g, 7.5 mmol, 1.5 equiv), acid (5 mmol, 1.0 equiv), and amine (5 mmol, 1.0 equiv) in CH_2Cl_2 (20 mL) was stirred at room temperature for 6 h. After completing reaction, the mixture was added 30 mL H_2O. The organic layer was separated and the aqueous layer was extracted with dichloromethane (20mL × 3). The combined organic phase was washed with brine (20 mL), and then dried over Na_2SO_4. After filtration, the solvent was concentrated in vacuo. The products were purified by column chromatography with petroleum ether and ethyl acetate as an eluent.

N-phenyl-3-(1-pivaloyl-1H-indol-3-yl)propanamide (1a)

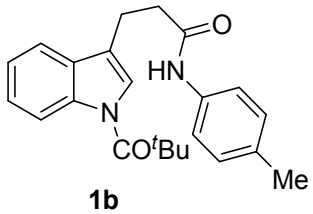


1a

White solid. M.p. = 122-123 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 8.3 Hz, 1H), 7.57 (s, 1H), 7.52 (d, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 7.9 Hz, 2H), 7.35 (t, *J* = 7.7 Hz, 1H), 7.31 – 7.22 (m, 3H), 7.20 (s, 1H), 7.07 (t, *J* = 7.4 Hz, 1H), 3.15 (t, *J* =

7.1 Hz, 2H), 2.71 (t, *J* = 7.2 Hz, 2H), 1.42 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.3, 137.6, 137.2, 128.9, 125.4, 124.4, 123.4, 123.1, 119.9, 119.8, 118.2, 117.6, 41.1, 37.4, 28.5, 20.8. HRMS (ESI): calcd. for $C_{22}H_{25}N_2O_2$ $[M + H]^+$ 349.1911; found 349.1913. (Petroleum ether/EtOAc, 5/1).

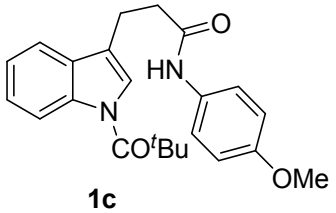
3-(1-pivaloyl-1*H*-indol-3-yl)-*N*-(p-tolyl)propanamide (1b)



1b

White solid. M.p. = 138-140 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, *J* = 8.3 Hz, 1H), 7.61 (s, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.42 – 7.24 (m, 4H), 7.18 (s, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 3.17 (t, *J* = 7.1 Hz, 2H), 2.73 (t, *J* = 7.2 Hz, 2H), 2.32 (s, 3H), 1.46 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.2, 137.2, 135.0, 134.0, 129.4, 129.0, 125.3, 123.4, 123.1, 120.0, 119.9, 118.2, 117.6, 41.1, 37.3, 28.5, 20.8, 20.8. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2063. (Petroleum ether/EtOAc, 4/1).

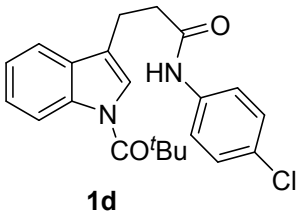
***N*-(4-methoxyphenyl)-3-(1-pivaloyl-1*H*-indol-3-yl)propanamide (1c)**



1c

White solid. M.p. = 129-131 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.0 Hz, 1H), 7.59 (s, 1H), 7.53 (d, *J* = 6.9 Hz, 1H), 7.44 – 7.24 (m, 5H), 6.81 (d, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 3.14 (t, *J* = 6.0 Hz, 2H), 2.70 (t, *J* = 6.1 Hz, 2H), 1.45 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.3, 156.4, 137.1, 130.8, 129.1, 125.3, 123.4, 123.0, 121.8, 121.8, 120.1, 118.2, 117.6, 114.0, 55.4, 41.1, 37.1, 28.5, 20.8. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_3$ $[M + H]^+$ 379.2016; found 379.2012. (Petroleum ether/EtOAc, 4/1).

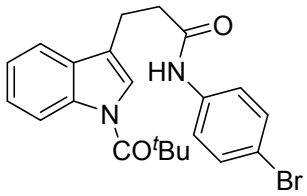
***N*-(4-chlorophenyl)-3-(1-pivaloyl-1*H*-indol-3-yl)propanamide (1d)**



1d

White solid. M.p. = 151-153 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, *J* = 8.3 Hz, 1H), 7.60 (s, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.43 – 7.35 (m, 3H), 7.35 – 7.22 (m, 3H), 7.19 – 7.07 (m, 1H), 3.18 (t, *J* = 7.0 Hz, 2H), 2.74 (t, *J* = 7.1 Hz, 2H), 1.46 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.2, 137.2, 136.1, 128.9, 128.9, 125.4, 123.4, 123.2, 120.9, 119.7, 118.1, 117.6 41.1, 37.4, 28.5, 20.7. HRMS (ESI): calcd. for $C_{22}H_{24}ClN_2O_2$ $[M + H]^+$ 383.1521; found 383.1520. (Petroleum ether/EtOAc, 5/1).

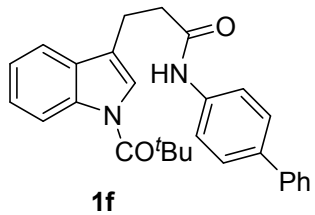
***N*-(4-bromophenyl)-3-(1-pivaloyl-1*H*-indol-3-yl)propanamide (1e)**



1e

White solid. M.p. = 171-173 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.3 Hz, 1H), 7.58 (s, 1H), 7.52 (d, *J* = 7.6 Hz, 1H), 7.43 – 7.25 (m, 7H), 3.15 (t, *J* = 7.0 Hz, 2H), 2.72 (t, *J* = 7.1 Hz, 2H), 1.45 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.9, 170.4, 137.1, 136.7, 131.9, 128.9, 125.4, 123.5, 123.1, 121.3, 119.8, 118.2, 117.6, 116.9, 41.1, 37.4, 28.5, 20.7. HRMS (ESI): calcd. for $C_{22}H_{24}BrN_2O_2$ $[M + H]^+$ 427.1016; found 427.1016. (Petroleum ether/EtOAc, 5/1).

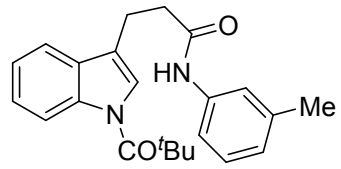
_N_-([1,1'-biphenyl]-4-yl)-3-(1-pivaloyl-1_H_-indol-3-yl)propanamide (1f)



1f

White solid. M.p. = 163-165 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 8.3 Hz, 1H), 7.57 (s, 1H), 7.54 – 7.44 (m, 6H), 7.44 – 7.20 (m, 7H), 3.14 (t, *J* = 7.1 Hz, 2H), 2.72 (t, *J* = 7.2 Hz, 2H), 1.41 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 170.5, 140.3, 137.2, 136.9, 129.0, 128.8, 127.5, 127.2, 126.8, 125.4, 123.5, 123.1, 120.2, 120.0, 118.2, 117.6, 41.1, 37.4, 28.5, 20.8. HRMS (ESI): calcd. for $C_{28}H_{29}N_2O_2$ $[M + H]^+$ 425.2224; found 425.2221. (Petroleum ether/EtOAc, 5/1).

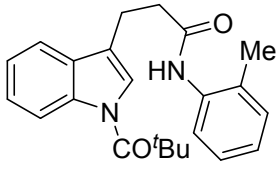
3-(1-pivaloyl-1_H_-indol-3-yl)-_N_-(m-tolyl)propanamide (1g)



1g

White solid. M.p. = 123-125 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 8.2 Hz, 1H), 7.61 (s, 1H), 7.54 (d, *J* = 4.8 Hz, 1H), 7.42 – 7.07 (m, 6H), 6.93 (d, *J* = 6.9 Hz, 1H), 3.17 (s, 2H), 2.73 (t, *J* = 6.8 Hz, 2H), 2.32 (s, 3H), 1.46 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.4, 138.8, 137.6, 137.2, 129.0, 128.7, 125.4, 125.2, 123.4, 123.1, 120.5, 120.0, 118.2, 117.6, 117.0, 41.1, 37.4, 28.5, 21.5 , 20.8. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2065. (Petroleum ether/EtOAc, 5/1).

3-(1-pivaloyl-1_H_-indol-3-yl)-_N_-(o-tolyl)propanamide (1h)

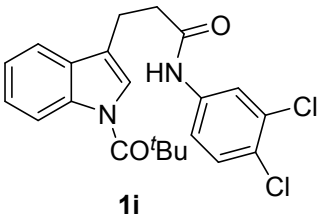


1h

White solid. M.p. = 121-123 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 8.2 Hz, 1H), 7.79 – 7.70 (m, 1H), 7.64 – 7.53 (m, 2H), 7.43 – 7.26 (m, 2H), 7.22 – 6.88 (m, 4H), 3.20 (t, *J* = 6.8 Hz, 2H), 2.80 (t, *J* = 7.0 Hz, 2H), 2.04 (s, 3H), 1.47 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.2, 137.2, 135.4, 130.4, 129.0, 126.6, 125.4, 125.2, 123.4, 123.2, 119.9, 118.2, 117.6, 41.1, 37.2, 28.6,

21.0, 17.4. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2067. (Petroleum ether/EtOAc, 5/1).

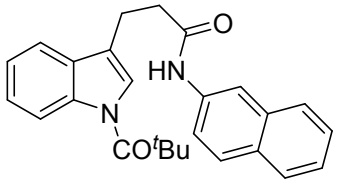
_N_-(3,4-dichlorophenyl)-3-(1-pivaloyl-1_H_-indol-3-yl)propanamide (1i)



1i

White solid. M.p. = 123-125 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, J = 8.3 Hz, 1H), 7.69 (s, 1H), 7.57 (s, 1H), 7.52 (d, J = 7.6 Hz, 1H), 7.41 – 7.23 (m, 3H), 7.17 (d, J = 8.7 Hz, 1H), 7.06 (s, 1H), 3.15 (t, J = 6.8 Hz, 2H), 2.72 (t, J = 6.4 Hz, 2H), 1.44 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.3, 137.2, 136.9, 132.7, 130.4, 128.8, 127.5, 125.4, 123.5, 123.3, 121.4, 119.5, 118.8, 118.1, 117.7, 41.1, 37.4, 28.5, 20.6. HRMS (ESI): calcd. for $C_{22}H_{23}Cl_2N_2O_2$ $[M + H]^+$ 417.1131; found 417.1129. (Petroleum ether/EtOAc, 5/1).

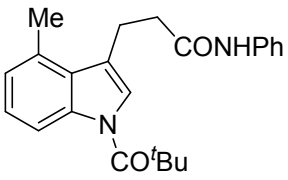
_N_-(naphthalen-2-yl)-3-(1-pivaloyl-1_H_-indol-3-yl)propanamide (1j)



1j

White solid. M.p. = 143-145 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, J = 8.3 Hz, 1H), 8.13 (s, 1H), 7.74 (t, J = 7.6 Hz, 3H), 7.61 (s, 1H), 7.57 – 7.51 (m, 1H), 7.48 – 7.34 (m, 3H), 7.34 – 7.19 (m, 3H), 3.19 (s, 2H), 2.78 (t, J = 5.6 Hz, 2H), 1.41 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 170.4, 137.2, 134.9, 133.7, 130.6, 128.9, 128.7, 127.6, 127.5, 126.5, 125.4, 125.0, 123.4, 123.2, 119.8, 119.6, 118.2, 117.6, 116.6, 41.1, 37.6, 28.5, 20.8. HRMS (ESI): calcd. for $C_{26}H_{27}N_2O_2$ $[M + H]^+$ 399.2067; found 399.2066. (Petroleum ether/EtOAc, 3/1).

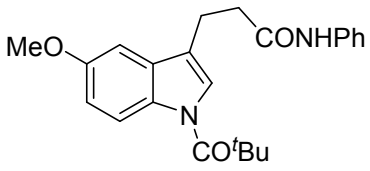
3-(4-methyl-1-pivaloyl-1_H_-indol-3-yl)-_N_-phenylpropanamide (1k)



1k

White solid. M.p. = 137-139 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, J = 8.4 Hz, 1H), 7.56 (s, 1H), 7.46 (d, J = 7.9 Hz, 2H), 7.34 – 7.28 (m, 2H), 7.25 (t, J = 7.9 Hz, 1H), 7.19 (s, 1H), 7.11 (t, J = 7.3 Hz, 1H), 7.05 (d, J = 7.3 Hz, 1H), 3.34 (t, J = 7.0 Hz, 2H), 2.82 – 2.59 (m, 5H), 1.43 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 170.0, 137.7, 137.6, 130.1, 128.9, 127.3, 125.5, 125.2, 124.3, 123.5, 120.6, 119.7, 115.3, 41.2, 38.5, 28.5, 22.9, 20.1. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2066. (Petroleum ether/EtOAc, 5/1).

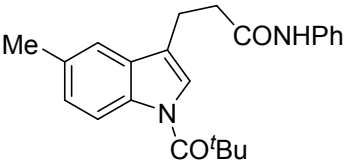
3-(5-methoxy-1-pivaloyl-1_H_-indol-3-yl)-_N_-phenylpropanamide (1l)



1l

White solid. M.p. = 155-157 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, *J* = 8.9 Hz, 1H), 7.52 (s, 1H), 7.38 (d, *J* = 7.9 Hz, 2H), 7.27 – 7.20 (m, 2H), 7.17 – 7.11 (m, 1H), 7.05 (t, *J* = 7.3 Hz, 1H), 6.95 – 6.83 (m, 2H), 3.81 (s, 3H), 3.08 (t, *J* = 6.9 Hz, 2H), 2.67 (t, *J* = 7.0 Hz, 2H), 1.39 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 170.3, 156.4, 137.6, 131.8, 130.0, 128.9, 124.3, 123.7, 119.7, 118.4, 113.2, 101.5, 55.7, 40.9, 37.3, 28.6, 20.8. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_3$ $[M + H]^+$ 379.2016; found 379.2013. (Petroleum ether/EtOAc, 5/1).

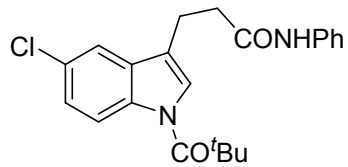
3-(5-methyl-1-pivaloyl-1*H*-indol-3-yl)-*N*-phenylpropanamide (1m)



1m

White solid. M.p. = 146-148 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 8.5 Hz, 1H), 7.57 (s, 1H), 7.43 (d, *J* = 7.8 Hz, 2H), 7.31 (dd, *J* = 15.7, 8.3 Hz, 3H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.15 – 7.03 (m, 2H), 3.16 (t, *J* = 6.8 Hz, 2H), 2.74 (t, *J* = 6.9 Hz, 2H), 2.47 (s, 3H), 1.44 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 170.2, 137.6, 135.4, 133.0, 129.1, 128.9, 126.6, 124.3, 123.2, 119.7, 118.1, 117.3, 41.0, 37.6, 28.5, 21.3, 20.9. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2067. (Petroleum ether/EtOAc, 5/1).

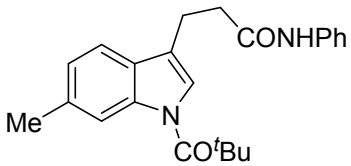
3-(5-chloro-1-pivaloyl-1H-indol-3-yl)-N-phenylpropanamide (1n)



1n

White solid. M.p. = 146-148 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.8 Hz, 1H), 7.61 (s, 1H), 7.52 – 7.39 (m, 3H), 7.32 – 7.16 (m, 4H), 7.08 (t, *J* = 7.1 Hz, 1H), 3.10 (s, 2H), 2.69 (t, *J* = 6.8 Hz, 2H), 1.42 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 170.0, 137.5, 135.5, 130.3, 129.0, 125.3, 124.5, 124.4, 119.8, 119.8, 119.2, 118.7, 117.9, 41.1, 37.2, 28.5, 20.5. HRMS (ESI): calcd. for $C_{22}H_{24}ClN_2O_2$ $[M + H]^+$ 383.1521; found 383.1520. (Petroleum ether/EtOAc, 5/1).

3-(6-methyl-1-pivaloyl-1*H*-indol-3-yl)-*N*-phenylpropanamide (1o)

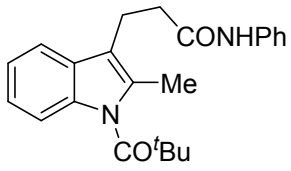


1o

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.52 (s, 1H), 7.43 (t, *J* = 6.3 Hz, 3H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.23 (s, 1H), 7.11 (dd, *J* = 17.8, 7.7 Hz, 2H), 3.15 (t, *J* = 7.0 Hz, 2H), 2.73 (t, *J* = 7.1 Hz, 2H),

2.50 (s, 3H), 1.44 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 170.3, 137.6, 135.5, 128.9, 126.7, 124.7, 124.3, 122.5, 119.9, 119.8, 117.9, 117.8, 41.1, 37.5, 28.5, 21.9, 20.9. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2066. (Petroleum ether/EtOAc, 5/1).

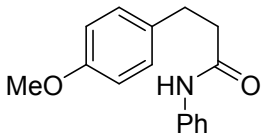
3-(2-methyl-1-pivaloyl-1*H*-indol-3-yl)-*N*-phenylpropanamide (1p)



1p

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 7.3 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.28 – 7.09 (m, 6H), 7.05 (t, *J* = 7.3 Hz, 1H), 3.10 (t, *J* = 7.2 Hz, 2H), 2.61 (t, *J* = 7.2 Hz, 2H), 2.28 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.6, 170.8, 137.8, 135.4, 132.7, 128.8, 128.2, 124.1, 122.1, 120.8, 119.8, 118.2, 113.9, 112.0, 44.2, 38.0, 28.2, 20.2, 11.5. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2067; found 363.2067. (Petroleum ether/EtOAc, 5/1).

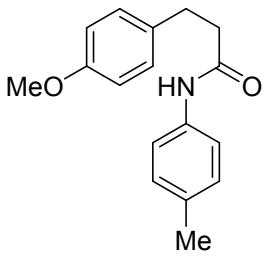
3-(4-methoxyphenyl)-*N*-phenylpropanamide (4a)



4a

White solid. M.p. = 127-129 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.18 (m, 5H), 7.13 – 7.05 (m, 3H), 6.89 -6.70 (m, 2H), 3.76 (s, 3H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.60 (t, *J* = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 158.1, 137.8, 132.7, 129.4, 128.9, 124.3, 120.0, 114.0, 55.3, 39.7, 30.7. HRMS (ESI): calcd. for $C_{16}H_{18}NO_2$ $[M + H]^+$ 256.1332; found 256.1335. (Petroleum ether/EtOAc, 5/1).

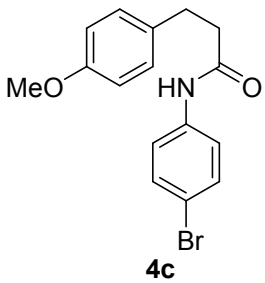
3-(4-methoxyphenyl)-*N*-(p-tolyl)propanamide (4b)



4b

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 6.8 Hz, 2H), 7.19 – 6.97 (m, 5H), 6.83 (d, *J* = 6.7 Hz, 2H), 3.77 (s, 3H), 2.97 (t, *J* = 6.8 Hz, 2H), 2.59 (t, *J* = 6.6 Hz, 2H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 158.1, 135.2, 133.9, 132.7, 129.4, 129.3, 120.1, 114.0, 55.2, 39.6, 30.7, 20.8. HRMS (ESI): calcd. for $C_{17}H_{20}NO_2$ $[M + H]^+$ 270.1489; found 270.1488. (Petroleum ether/EtOAc, 5/1).

***N*-(4-bromophenyl)-3-(4-methoxyphenyl)propanamide (4c)**

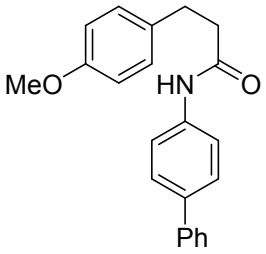


4c

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.29 (m, 5H), 7.13 (d, *J* = 8.1 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 3.80 (s,

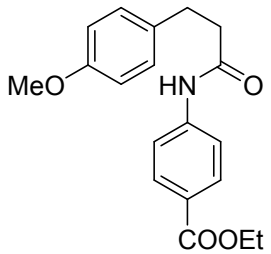
3H), 2.98 (t, J = 7.3 Hz, 2H), 2.62 (t, J = 7.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 158.2, 136.8, 132.4, 131.9, 129.3, 121.5, 116.8, 114.0, 55.2, 39.6, 30.6. HRMS (ESI): calcd. for $C_{16}H_{17}BrNO_2$ $[M + H]^+$ 334.0437; found 334.0438. (Petroleum ether/EtOAc, 5/1).

_N_-([1,1'-biphenyl]-4-yl)-3-(4-methoxyphenyl)propanamide (4d)



Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.30 (m, 9H), 7.23 – 7.02 (m, 3H), 6.88 (d, J = 7.4 Hz, 2H), 3.81 (s, 3H), 3.04 (s, 2H), 2.69 (d, J = 6.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 158.2, 140.4, 137.1, 137.0, 132.6, 129.4, 128.7, 127.6, 127.1, 126.8, 120.1, 114.0, 55.2, 39.8, 30.7. HRMS (ESI): calcd. for $C_{22}H_{22}NO_2$ $[M + H]^+$ 332.1645; found 332.1644. (Petroleum ether/EtOAc, 5/1).

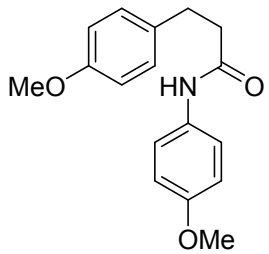
Ethyl 4-(3-(4-methoxyphenyl)propanamido)benzoate (4e)



4e

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.5 Hz, 2H), 7.72 – 7.49 (m, 3H), 7.25 (d, J = 8.2 Hz, 2H), 6.95 (d, J = 8.3 Hz, 2H), 4.47 (q, J = 7.0 Hz, 2H), 3.90 (s, 3H), 3.11 (t, J = 7.4 Hz, 2H), 2.77 (t, J = 7.4 Hz, 2H), 1.50 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 166.1, 158.2, 141.9, 132.4, 130.7, 129.3, 125.8, 118.8, 114.0, 60.9, 55.2, 39.8, 30.5, 14.3. HRMS (ESI): calcd. for $C_{19}H_{22}NO_4$ $[M + H]^+$ 328.1543; found 328.1543. (Petroleum ether/EtOAc, 5/1).

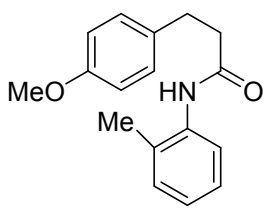
_N_,3-bis(4-methoxyphenyl)propanamide (4f)



4f

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, J = 8.1 Hz, 2H), 7.15 (d, J = 7.8 Hz, 2H), 7.05 (s, 1H), 6.87 – 6.75 (m, 4H), 3.78 (s, 6H), 2.98 (t, J = 7.5 Hz, 2H), 2.59 (t, J = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 158.1, 156.4, 132.7, 130.8, 129.3, 121.9, 114.1, 114.0, 55.4, 55.2, 39.6, 30.8. HRMS (ESI): calcd. for $C_{17}H_{20}NO_3$ $[M + H]^+$ 286.1438; found 286.1433. (Petroleum ether/EtOAc, 5/1).

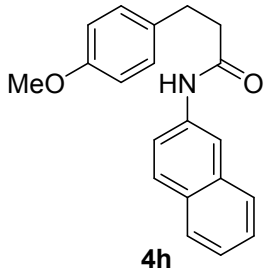
3-(4-methoxyphenyl)-N-(o-tolyl)propanamide (4g)



4g

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.7 Hz, 1H), 7.21 – 7.11 (m, 4H), 7.05 (t, *J* = 7.3 Hz, 1H), 6.84 (d, *J* = 8.3 Hz, 3H), 3.78 (s, 3H), 3.00 (t, *J* = 7.4 Hz, 2H), 2.66 (t, *J* = 7.4 Hz, 2H), 2.06 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 158.2, 135.5, 132.5, 130.4, 129.4, 126.7, 125.2, 123.3, 114.0, 55.3, 39.5, 30.8, 17.5. HRMS (ESI): calcd. for $C_{17}H_{20}NO_2$ $[M + H]^+$ 270.1489; found 270.1488. (Petroleum ether/EtOAc, 5/1).

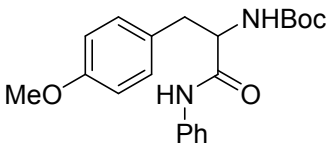
4-(4-methoxyphenyl)-*N*-(naphthalen-2-yl)propanamide (4h)



4h

Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.86 – 7.69 (m, 3H), 7.50 – 7.29 (m, 4H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 3.77 (s, 3H), 3.02 (t, *J* = 7.5 Hz, 2H), 2.67 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 158.2, 135.2, 133.8, 132.6, 130.6, 129.4, 128.7, 127.6, 127.5, 126.5, 125.0, 119.8, 116.6, 114.0, 55.3, 39.8, 30.7. HRMS (ESI): calcd. for $C_{20}H_{20}NO_2$ $[M + H]^+$ 306.1489; found 306.1488. (Petroleum ether/EtOAc, 4/1).

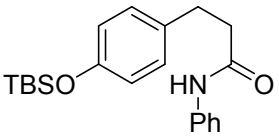
***tert*-butyl (3-(4-methoxyphenyl)-1-oxo-1-(phenylamino)propan-2-yl)carbamate (4i)**



4i

White solid. M.p. = 116-118 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.41 (d, *J* = 7.9 Hz, 2H), 7.32 – 7.23 (m, 2H), 7.19 (d, *J* = 8.2 Hz, 2H), 7.10 (t, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 8.3 Hz, 2H), 5.35 (s, 1H), 4.52 (s, 1H), 3.79 (s, 3H), 3.11 (s, 2H), 1.44 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 158.6, 155.8, 137.4, 130.3, 128.9, 128.5, 124.4, 120.0, 114.1, 80.5, 56.8, 55.2, 37.6, 28.3. HRMS (ESI): calcd. for $C_{21}H_{27}N_2O_4$ $[M + H]^+$ 371.1965; found 371.1964. (Petroleum ether/EtOAc, 4/1).

3-(4-((tert-butyldimethylsilyl)oxy)phenyl)-N-phenylpropanamide (4j)

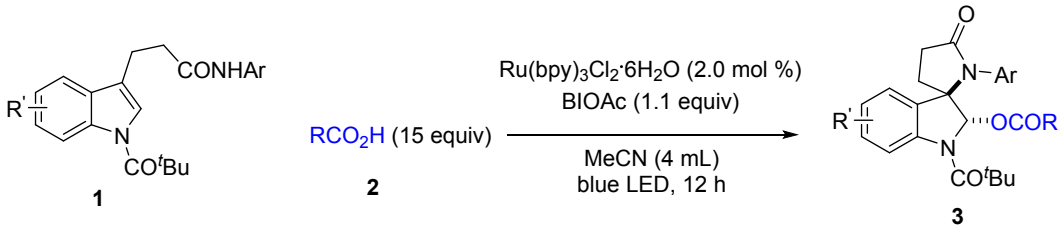


4j

White solid. M.p. = 117-119 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.8 Hz, 2H), 7.28 (dd, *J* = 13.1, 5.1 Hz, 2H), 7.08 (d, *J* = 7.4 Hz, 4H), 6.76 (d, *J* = 8.2 Hz, 2H), 2.97 (t, *J* = 7.5 Hz, 2H), 2.61 (t, *J* = 7.5 Hz, 2H), 0.97 (s, 9H), 0.18 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.9, 138.4, 133.9, 130.0, 129.7, 125.0, 120.9, 120.6,

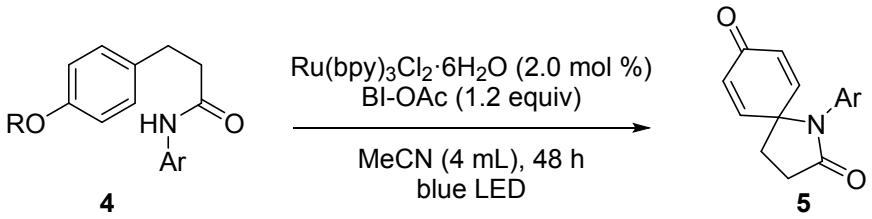
40.5, 31.6, 26.4, 18.9, 3.7. HRMS (ESI): calcd. for $C_{21}H_{30}NO_2Si$ $[M + H]^+$ 256.2040; found 256.2042. (Petroleum ether/EtOAc, 5/1).

General experimental procedure for 3.



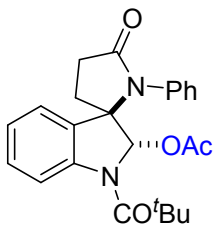
To a 10 mL glass vial was added **1** (0.25 mmol, 1.0 equiv), $Ru(bpy)_3Cl_2$•$6H_2O$ (3.7 mg, 0.005 mmol, 2.0 mol %), BI-OAc (84.3 mg, 0.275 mmol, 1.1 equiv), **2** (3.75 mmol, 15 equiv) and 4.0 mL of acetonitrile. The reaction mixture was degassed by bubbling with Ar for 15 s with an outlet needle and the vial was sealed with PTFE cap. And the mixture was stirred under the irradiation with blue LED for 12 h at room temperature . Upon completion, the solvent was removed directly under reduced pressure to afford the crude product, which was purified by flash column chromatography to afford the desired product **3**.

3. General procedure for oxamination of indoles and amidation of phenols General experimental procedure for 5.



To a 10 mL glass vial was added **4** (0.25 mmol, 1.0 equiv), $Ru(bpy)_3Cl_2$•$6H_2O$ (3.7 mg, 0.005 mmol, 2.0 mol %), BI-OAc (92 mg, 0.3 mmol, 1.2 equiv) and 4.0 mL of acetonitrile. The reaction mixture was degassed by bubbling with Ar for 15 s with an outlet needle and the vial was sealed with PTFE cap. And the mixture was stirred under the irradiation with blue LED for 48 h at room temperature . Upon completion, the solvent was removed directly under reduced pressure to afford the crude product, which was purified by flash column chromatography to afford the desired product **5**.

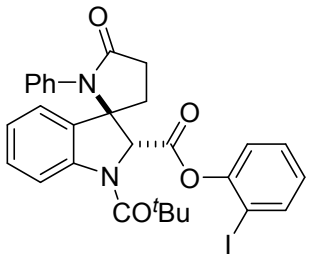
5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3a)



3a

White solid (82 mg, 81%). M.p. = 127-129 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.2 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.41 (t, *J* = 7.8 Hz, 1H), 7.31 – 7.23 (m, 1H), 7.19 – 7.08 (m, 3H), 6.89 (s, 1H), 6.69 – 6.55 (m, 2H), 2.93 – 2.73 (m, 2H), 2.67 – 2.48 (m, 1H), 2.41 – 2.25 (m, 1H), 2.13 (s, 3H), 1.03 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 174.1, 170.6, 145.0, 135.1, 131.3, 130.6, 129.0, 128.2, 127.7, 124.9, 123.5, 119.2, 89.2, 74.2, 40.9, 30.3, 28.0, 26.4, 21.1. HRMS (ESI): calcd. for $C_{24}H_{27}N_2O_4$ $[M + H]^+$ 407.1965; found 407.1964. (Petroleum ether/EtOAc, 2/1).

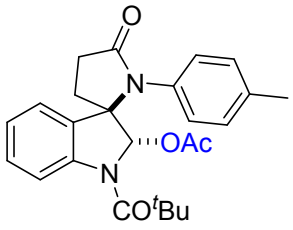
2-iodophenyl-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidine]-2-carboxylate (3a' or 3t)



3a'

White solid (107 mg, 72%). M.p. = 163-165 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.9 Hz, 1H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.55 (d, *J* = 7.4 Hz, 1H), 7.42 (dt, *J* = 22.9, 7.5 Hz, 2H), 7.34 – 7.27 (m, 1H), 7.24 – 7.12 (m, 4H), 6.70 (dd, *J* = 5.3, 1.7 Hz, 2H), 3.07 – 2.76 (m, 2H), 2.73 – 2.59 (m, 1H), 2.58 – 2.44 (m, 1H), 1.06 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 174.1, 165.4, 145.1, 142.2, 135.2, 133.9, 132.1, 131.7, 131.5, 130.7, 129.1, 128.2, 128.1, 127.7, 125.0, 123.6, 119.2, 95.1, 90.2, 74.5, 41.1, 30.4, 28.1, 26.7. HRMS (ESI): calcd. for $C_{29}H_{28}IN_2O_4$ $[M + H]^+$ 595.1088; found 595.1089. (Petroleum ether/EtOAc, 3/1).

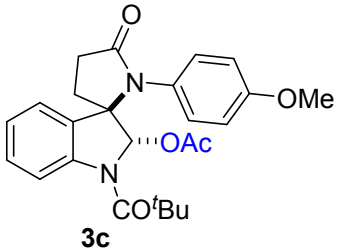
5'-oxo-1-pivaloyl-1'-(p-tolyl)spiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3b)



3b

White solid (89 mg, 85%). M.p. = 181-183 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.2 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 1H), 7.30 – 7.18 (m, 1H), 6.99 – 6.83 (m, 3H), 6.51 (d, *J* = 8.1 Hz, 2H), 2.90 – 2.73 (m, 2H), 2.64 – 2.49 (m, 1H), 2.40 – 2.29 (m, 1H), 2.21 (s, 3H), 2.12 (s, 3H), 1.04 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 174.1, 170.6, 144.9, 138.1, 132.4, 131.4, 130.5, 129.7, 127.5, 124.8, 123.5, 119.1, 89.3, 74.1, 40.9, 30.2, 28.0, 26.4, 21.1, 20.9.HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2123. (Petroleum ether/EtOAc, 2/1).

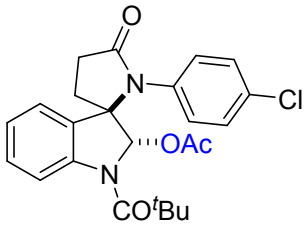
1'-(4-methoxyphenyl)-5'-oxo-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3c)



3c

White solid (83 mg, 76%). M.p. = 166-168 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.2 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 1H), 7.30 – 7.21 (m, 1H), 6.89 (s, 1H), 6.65 (d, *J* = 8.8 Hz, 2H), 6.55 (d, *J* = 8.7 Hz, 2H), 3.68 (s, 3H), 2.89 – 2.70 (m, 2H), 2.57 (dt, *J* = 14.2, 8.3 Hz, 1H), 2.41 – 2.26 (m, 1H), 2.13 (s, 3H), 1.07 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 174.2, 170.6, 159.1, 144.9, 131.3, 130.6, 128.9, 127.6, 124.8, 123.5, 119.1, 119.1, 114.3, 89.3, 74.0, 55.3, 41.0, 30.2, 28.0, 26.3, 21.1. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_5$ $[M + H]^+$ 437.2071; found 437.2070. (Petroleum ether/EtOAc, 3/1).

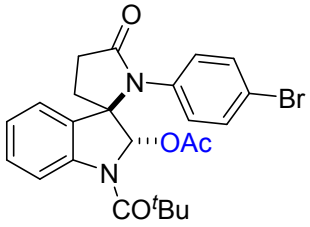
1'-(4-chlorophenyl)-5'-oxo-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3d)



3d

White solid (91 mg, 83%). M.p. = 184-186 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.2 Hz, 1H), 7.52 – 7.46 (m, 1H), 7.46 – 7.37 (m, 1H), 7.31 – 7.21 (m, 1H), 7.16 – 7.05 (m, 2H), 6.84 (s, 1H), 6.61 – 6.49 (m, 2H), 2.94 – 2.73 (m, 2H), 2.60 (dt, *J* = 13.9, 8.5 Hz, 1H), 2.41 – 2.28 (m, 1H), 2.13 (s, 3H), 1.06 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 174.1, 170.6, 145.0, 133.9, 133.7, 130.9, 129.2, 128.9, 125.0, 123.5, 119.3, 89.2, 74.1, 40.9, 30.2, 27.9, 26.3, 21.0. HRMS (ESI): calcd. for $C_{24}H_{26}ClN_2O_4$ $[M + H]^+$ 441.1576; found 441.1572. (Petroleum ether/EtOAc, 2/1).

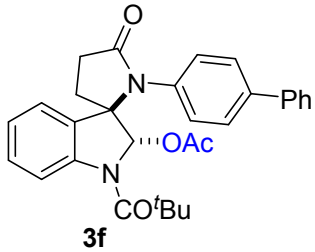
1'-(4-bromophenyl)-5'-oxo-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3e)



3e

White solid (93 mg, 77%). M.p. = 186-188 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.2 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.33 – 7.18 (m, 3H), 6.83 (s, 1H), 6.50 (d, *J* = 7.5 Hz, 2H), 2.92 – 2.72 (m, 2H), 2.67 – 2.50 (m, 1H), 2.41 – 2.25 (m, 1H), 2.13 (s, 3H), 1.06 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 174.0, 170.6, 145.0, 134.2, 132.1, 130.9, 129.2, 125.0, 123.5, 122.0, 119.3, 89.2, 74.1, 40.9, 30.2, 27.9, 26.3, 21.0. HRMS (ESI): calcd. for $C_{24}H_{26}BrN_2O_4$ $[M + H]^+$ 485.1070; found 485.1072. (Petroleum ether/EtOAc, 2/1).

1'-([1,1'-biphenyl]-4-yl)-5'-oxo-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3f)

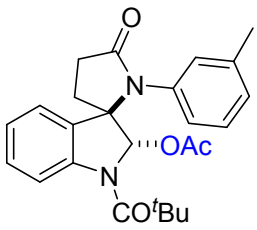


3f

Colorless oil (96 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.2 Hz, 1H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.49 –

7.22 (m, 9H), 6.94 (s, 1H), 6.74 (d, J = 7.9 Hz, 2H), 2.88 (t, J = 7.9 Hz, 2H), 2.68 – 2.55 (m, 1H), 2.49 – 2.31 (m, 1H), 2.15 (s, 3H), 1.04 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 174.1, 170.6, 144.9, 141.1, 140.1, 134.4, 131.6, 130.7, 128.7, 127.9, 127.8, 127.5, 127.0, 125.0, 123.6, 119.2, 89.3, 74.2, 40.9, 30.3, 27.9, 26.6, 21.0. HRMS (ESI): calcd. for $C_{30}H_{31}N_2O_4$ $[M + H]^+$ 483.2278; found 483.2277. (Petroleum ether/EtOAc, 2/1).

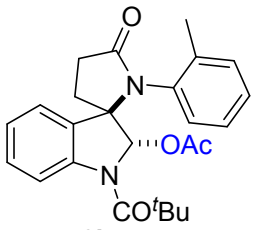
5'-oxo-1-pivaloyl-1'-(*m*-tolyl)spiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3g)



3g

Colorless oil (91 mg, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.2 Hz, 1H), 7.47 (d, J = 7.5 Hz, 1H), 7.36 (t, J = 7.8 Hz, 1H), 7.27 – 7.14 (m, 1H), 7.02 – 6.90 (m, 2H), 6.85 (s, 1H), 6.48 (s, 1H), 6.29 (d, J = 7.1 Hz, 1H), 2.88 – 2.69 (m, 2H), 2.63 – 2.49 (m, 1H), 2.36 – 2.24 (m, 1H), 2.11 (s, 3H), 2.10 (s, 3H), 1.02 (s, 9H).^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 174.0, 170.6, 145.0, 138.9, 134.9, 131.4, 130.5, 128.9, 128.8, 128.5, 124.8, 124.5, 123.5, 119.1, 89.2, 74.2, 40.9, 30.3, 27.9, 26.3, 21.0. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2120. (Petroleum ether/EtOAc, 2/1).

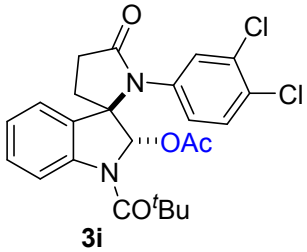
5'-oxo-1-pivaloyl-1'-(*o*-tolyl)spiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3h)



3h

White solid (67 mg, 64%). M.p. = 156-158 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, J = 8.3 Hz, 1H), 7.53 (d, J = 7.5 Hz, 1H), 7.46 – 7.38 (m, 1H), 7.28 – 7.20 (m, 2H), 7.15 – 7.07 (m, 1H), 6.90 – 6.71 (m, 2H), 5.86 (d, J = 7.9 Hz, 1H), 3.06 – 2.89 (m, 1H), 2.84 – 2.69 (m, 1H), 2.63 – 2.47 (m, 2H), 2.40 (s, 3H), 2.12 (s, 3H), 0.91 (s, 9H).^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 172.6, 171.1, 144.5, 136.8, 134.0, 133.0, 131.3, 130.5, 128.7, 126.8, 126.2, 125.2, 122.8, 119.1, 119.1, 87.9, 74.2, 40.8, 29.9, 28.2, 27.7, 27.6, 21.1, 18.4. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2118. (Petroleum ether/EtOAc, 2/1).

1'-(3,4-dichlorophenyl)-5'-oxo-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3i)

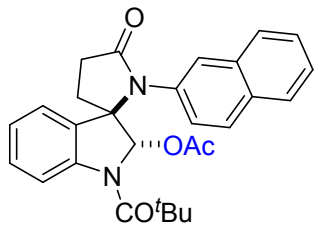


3i

White solid (62 mg, 52%). M.p. = 150-152 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.2 Hz, 1H), 7.54 – 7.41 (m, 2H), 7.33 – 7.27 (m, 1H), 7.17 (d, J = 8.6 Hz, 1H), 6.93 – 6.78 (m, 2H), 6.41 – 6.21 (m, 1H), 2.89 (dt, J = 17.6,

7.6 Hz, 1H), 2.82 – 2.72 (m, 1H), 2.64 (dt, *J* = 13.2, 8.9 Hz, 1H), 2.37 – 2.27 (m, 1H), 2.15 (s, 3H), 1.12 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 174.0, 170.6, 145.1, 134.4, 132.7, 132.3, 131.2, 130.4, 130.1, 130.0, 126.7, 125.1, 123.5, 119.4, 89.1, 74.3, 41.0, 30.1, 28.0, 26.0, 21.0. HRMS (ESI): calcd. for $C_{24}H_{25}Cl_2N_2O_4$ $[M + H]^+$ 475.1186; found 475.1184. (Petroleum ether/EtOAc, 2/1).

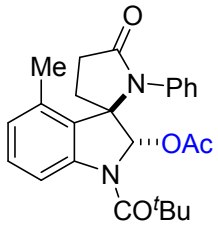
1'-(naphthalen-2-yl)-5'-oxo-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3j)



3j

Colorless oil (74 mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.2 Hz, 1H), 7.71 (d, *J* = 7.4 Hz, 1H), 7.66 – 7.50 (m, 3H), 7.48 – 7.36 (m, 3H), 7.34 – 7.25 (m, 1H), 7.16 (s, 1H), 6.98 (s, 1H), 6.72 (dd, *J* = 8.7, 1.8 Hz, 1H), 3.02 – 2.80 (m, 2H), 2.67 (dt, *J* = 14.0, 8.4 Hz, 1H), 2.50 – 2.32 (m, 1H), 2.15 (s, 3H), 0.88 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 174.2, 170.6, 145.0, 133.2, 132.6, 132.5, 131.3, 130.7, 128.8, 127.8, 127.4, 126.6, 126.5, 126.3, 125.2, 124.9, 123.7, 119.1, 89.4, 74.3, 40.8, 30.3, 27.7, 26.5, 21.1. HRMS (ESI): calcd. for $C_{28}H_{29}N_2O_4$ $[M + H]^+$ 457.2122; found 457.2121. (Petroleum ether/EtOAc, 2/1).

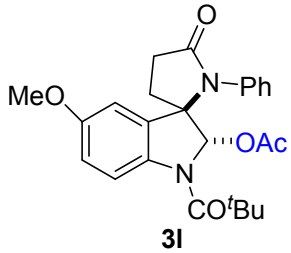
4-methyl-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3k)



3k

Colorless oil (77 mg, 73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.2 Hz, 1H), 7.27 (t, *J* = 7.9 Hz, 1H), 7.17 – 7.14 (m, 3H), 7.01 (d, *J* = 7.5 Hz, 1H), 6.84 (s, 1H), 6.73 – 6.67 (m, 2H), 2.85 – 2.75 (m, 2H), 2.74 – 2.63 (m, 1H), 2.50 (s, 3H), 2.35 (dq, *J* = 10.2, 6.2 Hz, 1H), 2.16 (s, 3H), 1.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 174.2, 170.7, 145.2, 135.4, 134.7, 130.2, 129.3, 129.1, 128.0, 127.5, 127.5, 116.5, 89.8, 75.1, 41.2, 29.6 28.0, 23.6, 21.1, 18.7. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2121. (Petroleum ether/EtOAc, 2/1).

5-methoxy-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3l)

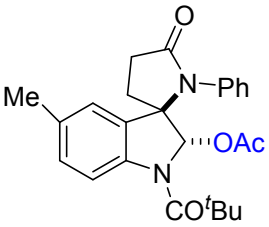


3l

White solid (92 mg, 84%). M.p. = 188-190 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 9.0 Hz, 1H), 7.18 – 7.11 (m, 3H), 7.01 (d, *J* = 2.5 Hz, 1H), 6.93 (dd, *J* = 9.0, 2.6 Hz, 1H), 6.88 (s, 1H), 6.70 – 6.62 (m, 2H), 3.86 (s, 3H), 2.93 – 2.71 (m, 2H), 2.55 (dt, *J* = 13.9, 8.2 Hz, 1H), 2.40 – 2.22 (m, 1H),

2.12 (s, 3H), 1.01 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 173.9, 170.6, 157.1, 138.6, 135.1, 132.8, 129.0, 128.1, 127.6, 120.1 115.6, 108.9, 89.4, 74.3, 55.8, 40.7, 30.2, 28.0, 26.4, 21.0. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_5$ $[M + H]^+$ 437.2071; found 437.2071. (Petroleum ether/EtOAc, 2/1).

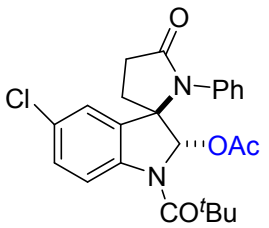
6-methyl-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3m)



3m

White solid (90 mg, 85%). M.p. = 185-187 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.4 Hz, 1H), 7.29 (s, 1H), 7.23 – 7.09 (m, 4H), 6.87 (s, 1H), 6.68 – 6.59 (m, 2H), 2.82 (t, J = 8.0 Hz, 2H), 2.62 – 2.50 (m, 1H), 2.47 – 2.28 (m, 4H), 2.11 (s, 3H), 1.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.3, 174.0, 170.6, 142.7, 135.2, 134.7, 131.6, 131.2, 129.0, 128.1, 127.6, 123.8, 118.9, 89.3, 74.2, 40.8, 30.3, 27.9, 26.4, 21.1, 21.0. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2120. (Petroleum ether/EtOAc, 2/1).

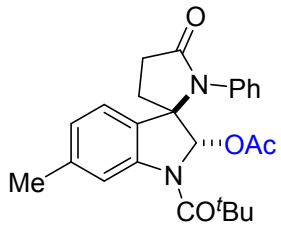
5-chloro-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3n)



3n

White solid (77 mg, 70%). M.p. = 200-202 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.8 Hz, 1H), 7.46 (d, J = 1.9 Hz, 1H), 7.36 (dd, J = 8.8, 2.0 Hz, 1H), 7.21 – 7.07 (m, 3H), 6.87 (s, 1H), 6.66 (dd, J = 7.2, 1.9 Hz, 2H), 2.89 – 2.71 (m, 2H), 2.62 – 2.45 (m, 1H), 2.35 (ddd, J = 23.3, 13.0, 8.1 Hz, 1H), 2.13 (s, 3H), 1.02 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.4, 173.8, 170.5, 143.6, 134.9, 133.3, 130.6, 129.9, 129.2, 128.4, 127.7, 123.6, 120.3, 89.2, 73.9, 41.0, 30.0, 27.9, 26.4, 21.0. HRMS (ESI): calcd. for $C_{24}H_{26}ClN_2O_4$ $[M + H]^+$ 441.1576; found 441.1575. (Petroleum ether/EtOAc, 3/1).

6-methyl-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl acetate (3o)

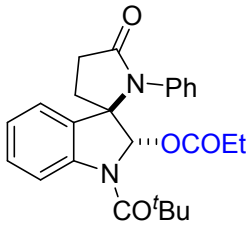


3o

White solid (87 mg, 83%). M.p. = 185-187 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.37 (d, J = 7.0 Hz, 1H), 7.18 –

7.04 (m, 4H), 6.87 (s, 1H), 6.69 – 6.61 (m, 2H), 2.87 – 2.74 (m, 2H), 2.61 – 2.49 (m, 1H), 2.43 – 2.26 (m, 4H), 2.12 (s, 3H), 1.02 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 174.0, 170.6, 145.2, 141.1, 135.2, 129.0, 128.5, 128.1, 127.7, 125.8, 123.1, 119.8, 89.5, 74.1, 41.0, 30.3, 28.0, 26.6, 21.9, 21.1. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2122. (Petroleum ether/EtOAc, 3/1).

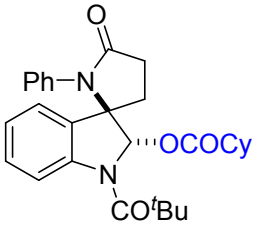
5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl propionate (3p)



3p

White solid (84 mg, 80%). M.p. = 120-122 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.2 Hz, 1H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.30 – 7.24 (m, 1H), 7.20 – 7.09 (m, 3H), 6.97 – 6.87 (m, 1H), 6.72 – 6.55 (m, 2H), 2.85 (dd, *J* = 11.1, 5.3 Hz, 2H), 2.59 (dt, *J* = 16.1, 8.2 Hz, 1H), 2.47 – 2.29 (m, 3H), 1.18 (td, *J* = 7.5, 1.8 Hz, 3H), 1.03 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 174.1, 174.0, 145.0, 135.2, 131.5, 130.6, 129.0, 128.1, 127.7, 124.9, 123.5, 119.2, 89.0, 74.2, 40.9, 30.3, 28.0, 27.7, 26.4, 8.9. HRMS (ESI): calcd. for $C_{25}H_{29}N_2O_4$ $[M + H]^+$ 421.2122; found 421.2120. (Petroleum ether/EtOAc, 3/1).

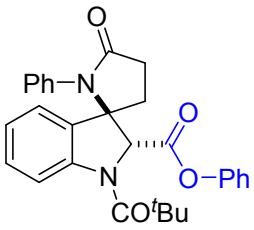
5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-2-yl cyclohexanecarboxylate (3q)



3q

White solid (89 mg, 75%). M.p. = 157-159 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.2 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.8 Hz, 1H), 7.26 (d, *J* = 6.3 Hz, 1H), 7.18 – 7.09 (m, 3H), 6.90 (s, 1H), 6.62 (dd, *J* = 7.5, 1.8 Hz, 2H), 2.81 (t, *J* = 7.9 Hz, 2H), 2.54 (dt, *J* = 13.8, 8.0 Hz, 1H), 2.43 – 2.18 (m, 2H), 1.88 (t, *J* = 13.1 Hz, 2H), 1.75 (d, *J* = 5.7 Hz, 2H), 1.66 (s, 1H), 1.51 – 1.33 (m, 2H), 1.29 – 1.14 (m, 3H), 1.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 175.7, 174.0, 145.1, 135.2, 131.7, 130.5, 129.0, 128.1, 127.6, 124.8, 123.4, 119.3, 88.6, 74.3, 43.3, 41.0, 30.3, 29.1, 28.5, 28.0, 26.5, 25.4, 25.3, 25.1. HRMS (ESI): calcd. for $C_{29}H_{35}N_2O_4$ $[M + H]^+$ 475.2591; found 475.2590. (Petroleum ether/EtOAc, 2/1).

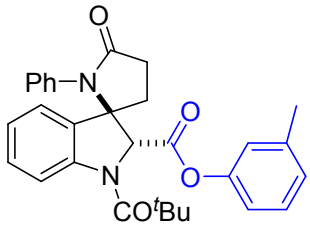
Phenyl-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidine]-2-carboxylate (3r)



3r

Colorless oil (82 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 7.90 (m, 3H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.54 – 7.50 (m, 1H), 7.44 (t, *J* = 7.8 Hz, 3H), 7.32 – 7.24 (m, 1H), 7.20 – 7.12 (m, 4H), 6.75 – 6.63 (m, 2H), 2.96 – 2.71 (m, 2H), 2.57 (ddd, *J* = 14.0, 8.9, 7.6 Hz, 1H), 2.43 (ddd, *J* = 14.0, 9.5, 5.8 Hz, 1H), 1.01 (s, 9H).^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 174.2, 166.1, 145.2, 135.2, 134.1, 131.6, 130.7, 130.0, 129.1, 128.7, 128.3, 128.2, 127.7, 124.9, 123.6, 119.2, 89.6, 74.7, 41.1, 30.4, 28.1, 26.7. HRMS (ESI): calcd. for $C_{29}H_{29}N_2O_4$ $[M + H]^+$ 469.2122; found 469.2125. (Petroleum ether/EtOAc, 3/1).

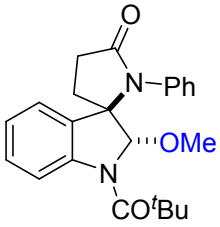
***m*-tolyl-5'-oxo-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidine]-2-carboxylate (3s)**



3s

White solid (94 mg, 78%). M.p. = 95-97 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.2 Hz, 1H), 7.85 – 7.74 (m, 2H), 7.53 (d, *J* = 7.3 Hz, 1H), 7.47 – 7.38 (m, 2H), 7.31 (dd, *J* = 14.4, 7.3 Hz, 2H), 7.21 – 7.11 (m, 4H), 6.74 – 6.60 (m, 2H), 2.95 – 2.72 (m, 2H), 2.62 – 2.50 (m, 1H), 2.49 – 2.21 (m, 4H), 1.02 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 174.2, 166.3, 145.2, 138.7, 135.3, 135.0, 131.6, 130.7, 130.4, 129.1, 128.6, 128.2, 128.1, 127.7, 127.2, 124.9, 123.6, 119.2, 89.6, 74.7, 41.1, 30.4, 28.1, 26.7, 21.2. HRMS (ESI): calcd. for $C_{30}H_{31}N_2O_4$ $[M + H]^+$ 483.2278; found 483.2277. (Petroleum ether/EtOAc, 3/1).

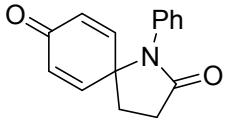
2-methoxy-1'-phenyl-1-pivaloylspiro[indoline-3,2'-pyrrolidin]-5'-one (3v)



3v

White solid (50 mg, 53%). M.p. = 120-122 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.2 Hz, 1H), 7.47 (d, *J* = 7.4 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 7.27 – 7.07 (m, 4H), 6.62 – 6.49 (m, 2H), 5.84 (s, 1H), 3.05 (s, 3H), 2.96 (ddd, *J* = 22.3, 11.2, 5.2 Hz, 2H), 2.88 – 2.74 (m, 1H), 2.61 – 2.46 (m, 1H), 1.07 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8, 173.9, 145.2, 135.3, 132.9, 130.5, 129.1, 128.2, 127.9, 124.7, 122.8, 118.4, 92.8 73.4, 52.0, 41.4, 30.3, 28.1, 24.9. HRMS (ESI): calcd. for $C_{23}H_{27}N_2O_3$ $[M + H]^+$ 379.2016; found 379.2015. (Petroleum ether/EtOAc, 3/1).

3-1-phenyl-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5a)

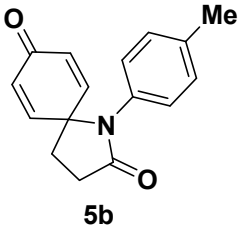


5a

White solid (48 mg, 80%). M.p. = 148-150 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.22 (m, 3H), 7.17 (d, *J* = 7.4 Hz, 2H),

6.99 (d, *J* = 10.1 Hz, 2H), 6.26 (d, *J* = 10.0 Hz, 2H), 2.78 (t, *J* = 8.0 Hz, 2H), 2.33 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.3, 174.0, 149.8, 136.3, 129.8, 129.2, 127.8, 126.1, 63.9, 31.7, 29.8. HRMS (ESI): calcd. for $C_{15}H_{14}NO_2$ $[M + H]^+$ 240.1019; found 240.1021. (Petroleum ether/EtOAc, 2/1).

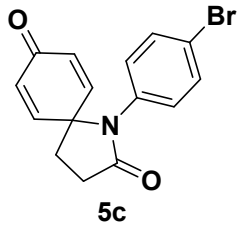
1-(4-chlorophenyl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5b)



5b

White solid (48 mg, 71%). M.p. = 123-125 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.16 (m, 3H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 7.8 Hz, 1H), 6.74 (dd, *J* = 10.1, 3.1 Hz, 1H), 6.32 (dd, *J* = 10.1, 1.9 Hz, 1H), 6.13 (dd, *J* = 10.1, 1.9 Hz, 1H), 2.91 – 2.70 (m, 2H), 2.45 (dt, *J* = 13.1, 9.6 Hz, 1H), 2.39 – 2.31 (m, 1H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.3, 173.3, 149.3, 148.1, 136.3, 134.5, 131.4, 130.6, 129.2, 128.8, 127.2, 126.6, 64.3, 31.8, 29.5, 18.7. HRMS (ESI): calcd. for $C_{15}H_{13}ClNO_2$ $[M + H]^+$ 274.0629; found 274.0627. (Petroleum ether/EtOAc, 2/1).

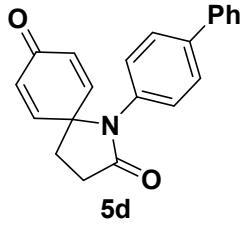
1-(4-bromophenyl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5c)



5c

White solid (60 mg, 76%). M.p. = 161-162 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.7 Hz, 2H), 7.09 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 10.0 Hz, 2H), 6.30 (d, *J* = 10.0 Hz, 2H), 2.78 (t, *J* = 8.0 Hz, 2H), 2.34 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.9, 173.8, 149.4, 135.4, 132.3, 130.0, 127.3, 121.2, 63.8, 31.7, 29.7. HRMS (ESI): calcd. for $C_{15}H_{13}BrNO_2$ $[M + H]^+$ 318.0124; found 318.0121. (Petroleum ether/EtOAc, 2/1).

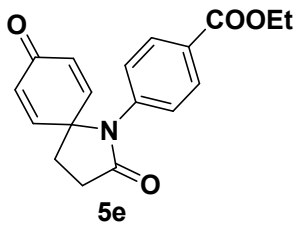
1-([1,1'-biphenyl]-4-yl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5d)



5d

White solid (49 mg, 62%). M.p. = 128-130 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 4H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.37 – 7.30 (m, 1H), 7.28 – 7.21 (m, 2H), 7.07 – 6.95 (m, 2H), 6.30 (d, *J* = 10.1 Hz, 2H), 2.80 (t, *J* = 8.0 Hz, 2H), 2.35 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.3, 174.0, 149.8, 140.5, 140.0, 135.4, 129.9, 128.8, 127.8, 127.6, 127.1, 126.2, 64.0, 31.7, 29.9. HRMS (ESI): calcd. for $C_{21}H_{18}NO_2$ $[M + H]^+$ 316.1332; found 316.1331. (Petroleum ether/EtOAc, 2/1).

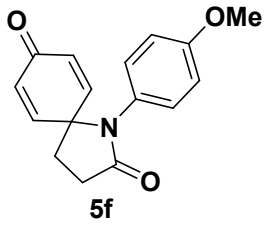
Ethyl 4-(2,8-dioxo-1-azaspiro[4.5]deca-6,9-dien-1-yl)benzoate (5e)



5e

White solid (62 mg, 80%). M.p. = 94-96 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (dd, *J* = 8.5, 1.7 Hz, 2H), 7.32 (dd, *J* = 8.6, 1.9 Hz, 2H), 7.00 (dd, *J* = 10.1, 1.9 Hz, 2H), 6.33 (dd, *J* = 9.9, 1.6 Hz, 2H), 4.35 (qd, *J* = 7.1, 1.9 Hz, 2H), 2.80 (dd, *J* = 11.2, 4.7 Hz, 2H), 2.44 – 2.22 (m, 2H), 1.37 (td, *J* = 7.1, 2.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.9, 173.7, 165.7, 149.7, 140.6, 130.4, 130.0, 128.9, 124.4, 64.0, 61.1, 32.1, 30.0, 14.3. HRMS (ESI): calcd. for $C_{18}H_{18}NO_4$ $[M + H]^+$ 312.1230; found 312.1232. (Petroleum ether/EtOAc, 2/1).

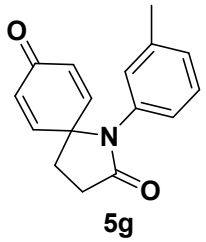
1-(4-methoxyphenyl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5f)



5f

White solid (49 mg, 73%). M.p. = 154-156 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, *J* = 8.9 Hz, 2H), 6.96 (d, *J* = 10.0 Hz, 2H), 6.83 (d, *J* = 8.9 Hz, 2H), 6.24 (d, *J* = 10.0 Hz, 2H), 3.77 (s, 3H), 2.76 (t, *J* = 8.1 Hz, 2H), 2.32 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.3, 174.2, 159.0, 149.6, 129.8, 128.6, 128.0, 114.4, 63.8, 55.4, 31.1, 29.6. HRMS (ESI): calcd. for $C_{16}H_{16}NO_3$ $[M + H]^+$ 270.1125; found 270.1123. (Petroleum ether/EtOAc, 2/1).

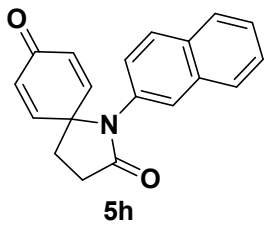
1-(*m*-tolyl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5g)



5g

White solid (53 mg, 84%). M.p. = 110-112 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, *J* = 7.8 Hz, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 7.02 – 6.84 (m, 4H), 6.31 – 6.16 (m, 2H), 2.77 (t, *J* = 8.0 Hz, 2H), 2.38 – 2.23 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.3, 174.0, 149.8, 139.1, 136.0, 129.7, 128.9, 128.8, 127.2, 123.2, 63.9, 31.5, 29.8, 21.3. HRMS (ESI): calcd. for $C_{16}H_{16}NO_2$ $[M + H]^+$ 254.1176; found 254.1175. (Petroleum ether/EtOAc, 2/1).

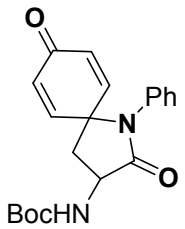
1-(naphthalen-2-yl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (5h)



5h

White solid (31 mg, 43%). M.p. = 139-141 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.70 (m, 3H), 7.63 (d, *J* = 1.6 Hz, 1H), 7.52 – 7.42 (m, 2H), 7.30 (dd, *J* = 8.8, 2.1 Hz, 1H), 7.13 – 6.96 (m, 2H), 6.33 – 6.17 (m, 2H), 2.82 (t, *J* = 8.0 Hz, 2H), 2.36 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 174.2, 149.8, 133.8, 133.2, 132.3, 129.9, 129.1, 127.9, 127.6, 126.6, 126.5, 124.5, 124.2, 64.1, 31.8, 29.9. HRMS (ESI): calcd. for $C_{19}H_{16}NO_2$ $[M + H]^+$ 290.1176; found 290.1174. (Petroleum ether/EtOAc, 2/1).

tert-butyl (2,8-dioxo-1-phenyl-1-azaspiro[4.5]deca-6,9-dien-3-yl)carbamate (5i)

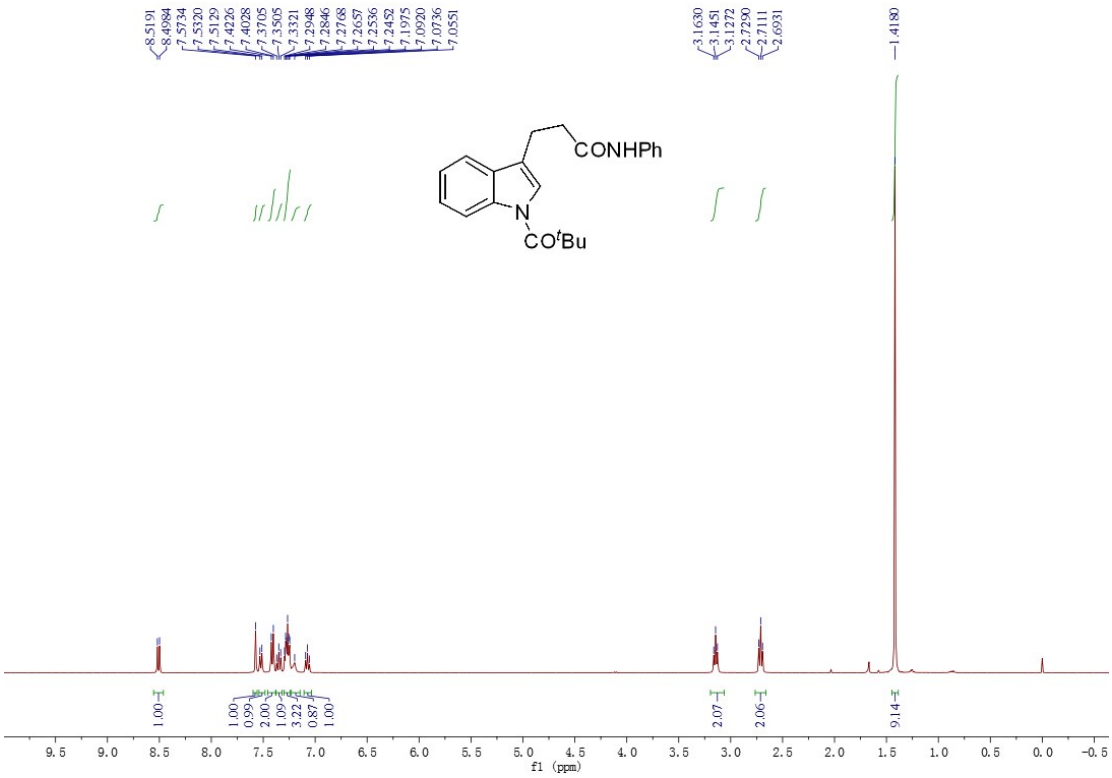


5i

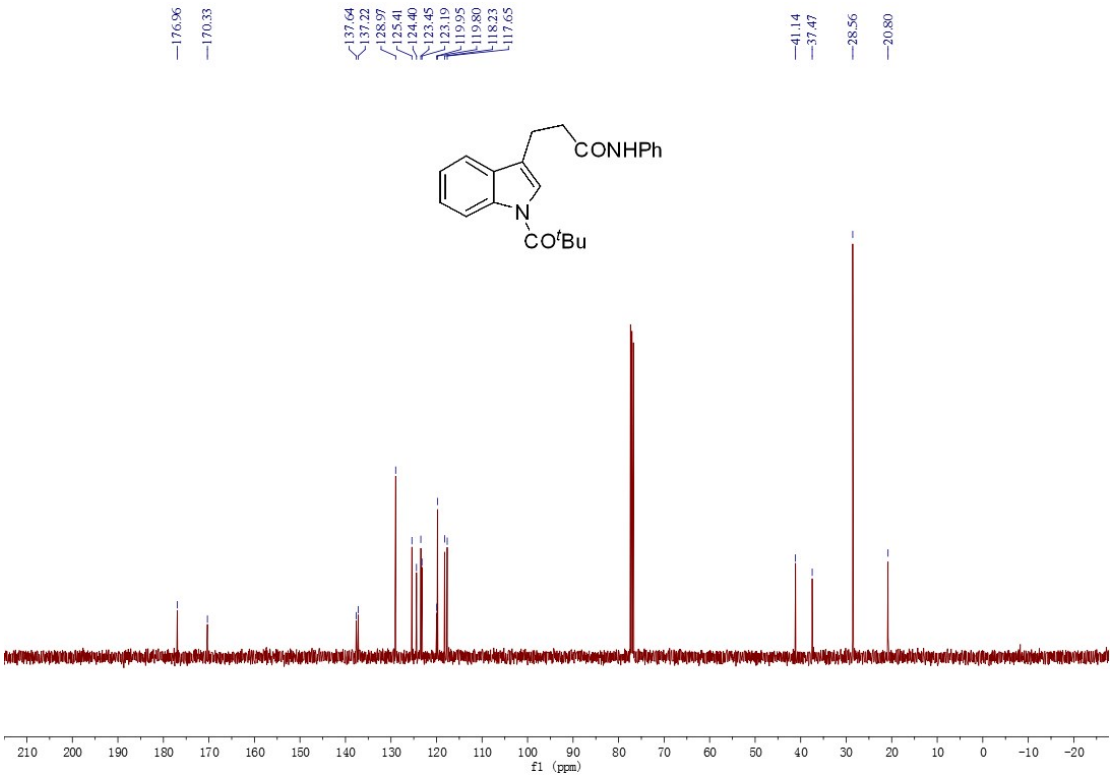
Colorless oil (50 mg, 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.28 (m, 3H), 7.25 – 7.13 (m, 3H), 6.80 (dd, *J* = 10.0, 2.7 Hz, 1H), 6.36 (dd, *J* = 10.1, 1.8 Hz, 1H), 6.24 (dd, *J* = 10.1, 1.7 Hz, 1H), 5.46 (d, *J* = 4.0 Hz, 1H), 4.58 (s, 1H), 2.94 – 2.66 (m, 1H), 2.50 – 2.33 (m, 1H), 1.51 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.1, 171.5, 155.6, 149.4, 149.0, 135.9, 130.6, 129.5, 129.2, 128.0, 125.8, 80.5, 61.4, 51.3, 40.0, 28.3. HRMS (ESI): calcd. for $C_{20}H_{23}N_2O_4$ $[M + H]^+$ 355.1652; found 355.1652. (Petroleum ether/EtOAc, 1/1).

4. NMR Spectra

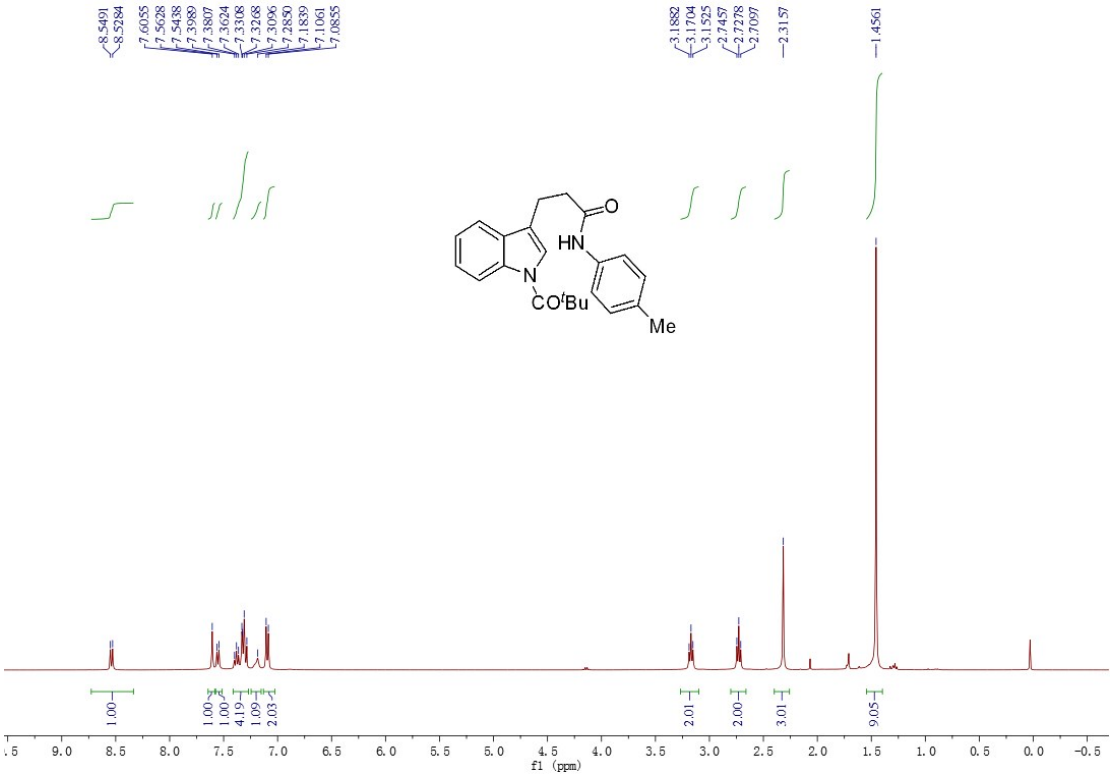
^{1}H NMR spectrum of compound **1a**



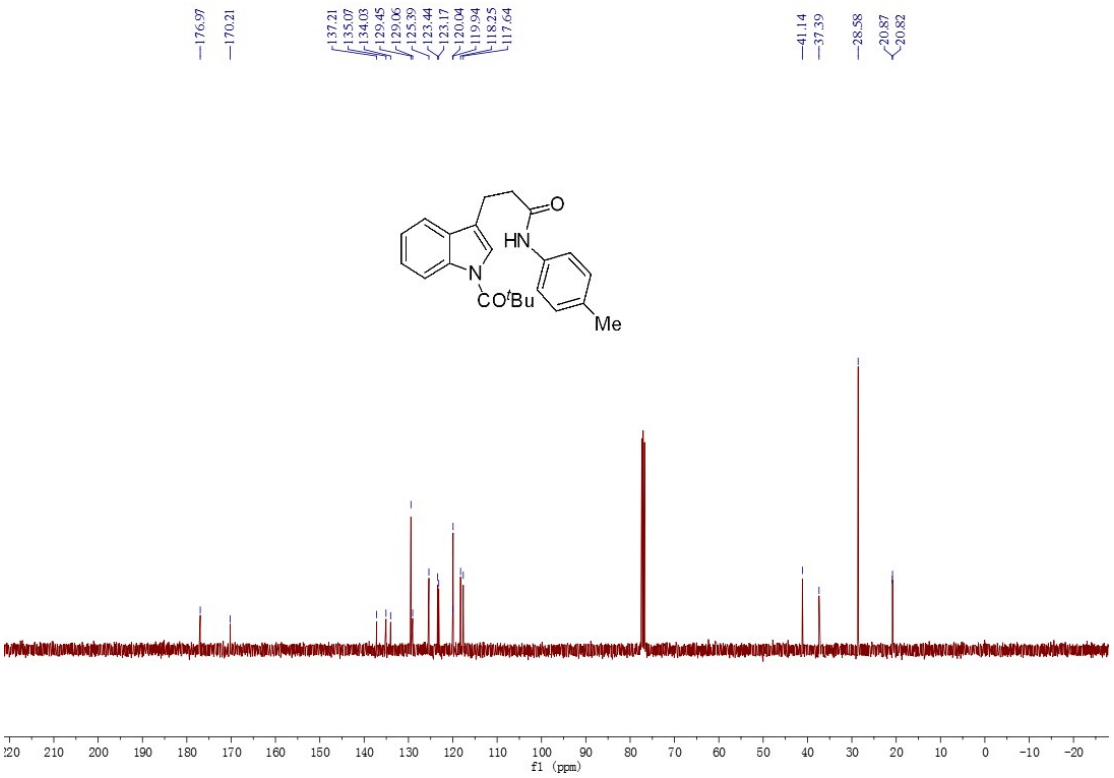
^{13}C NMR spectrum of compound **1a**



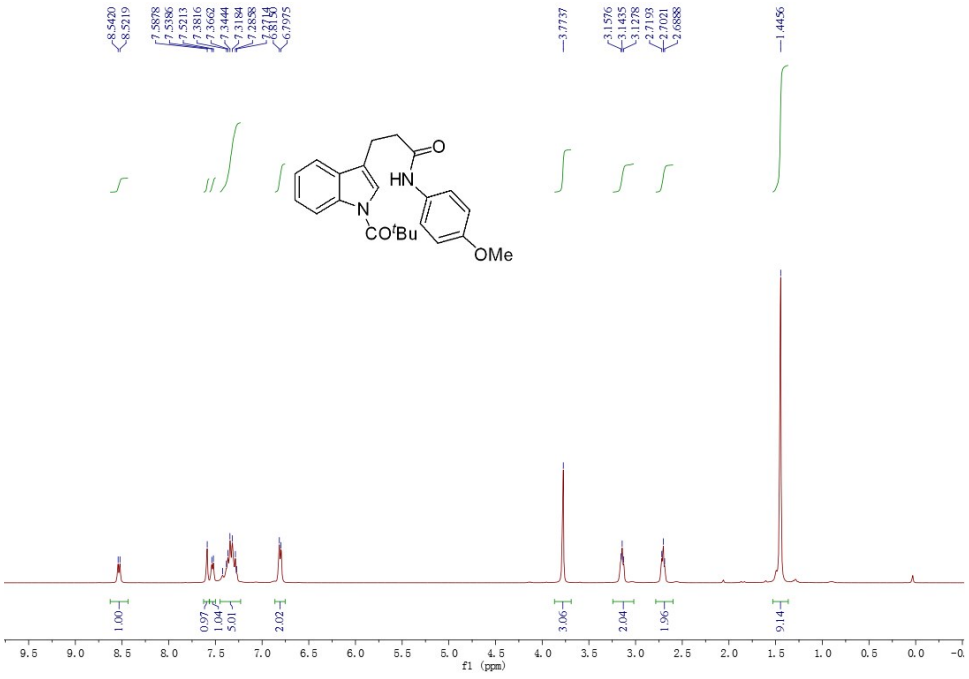
^{1}H NMR spectrum of compound **1b**



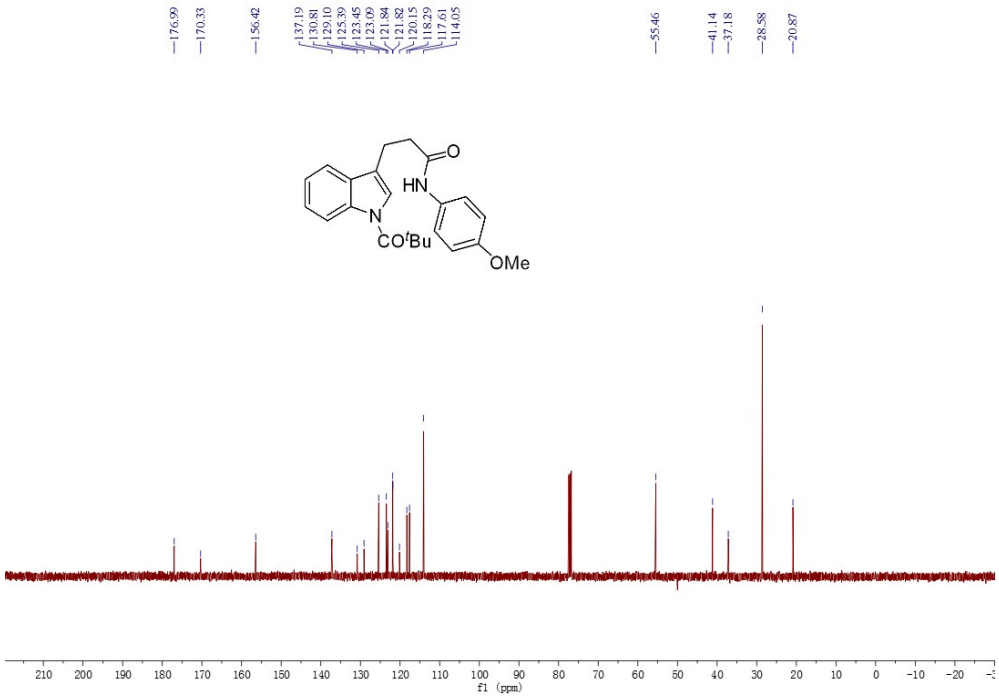
^{13}C NMR spectrum of compound **1b**



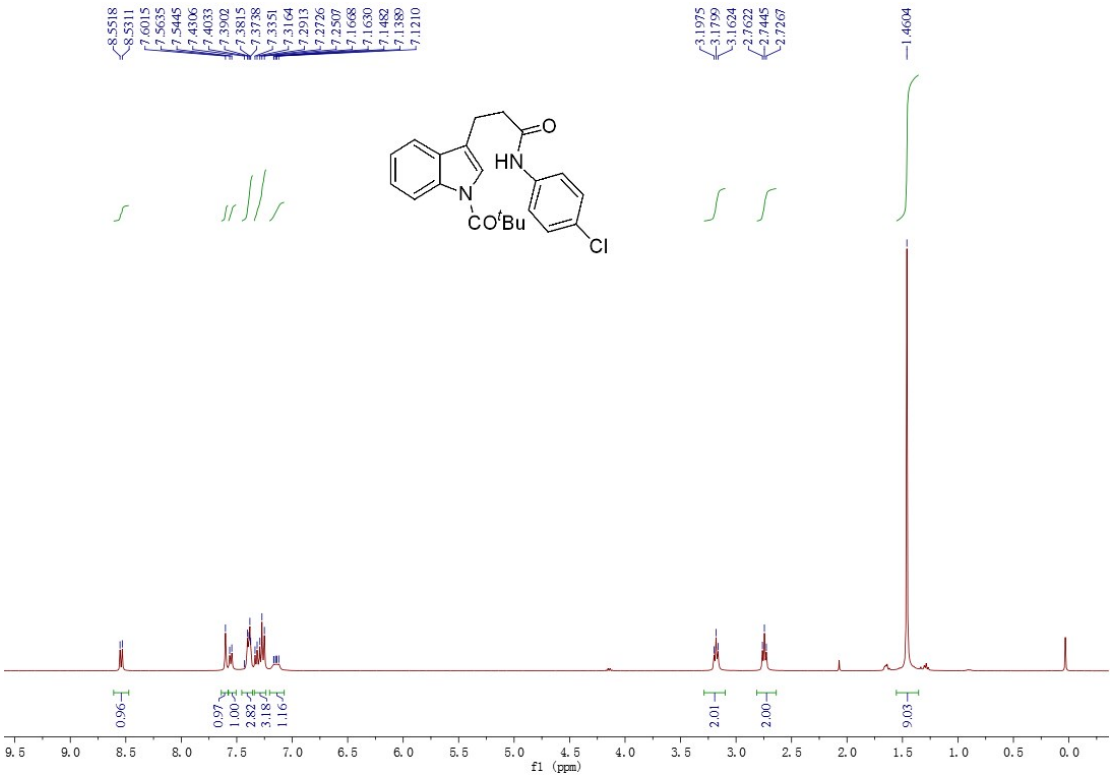
^{1}H NMR spectrum of compound **1c**



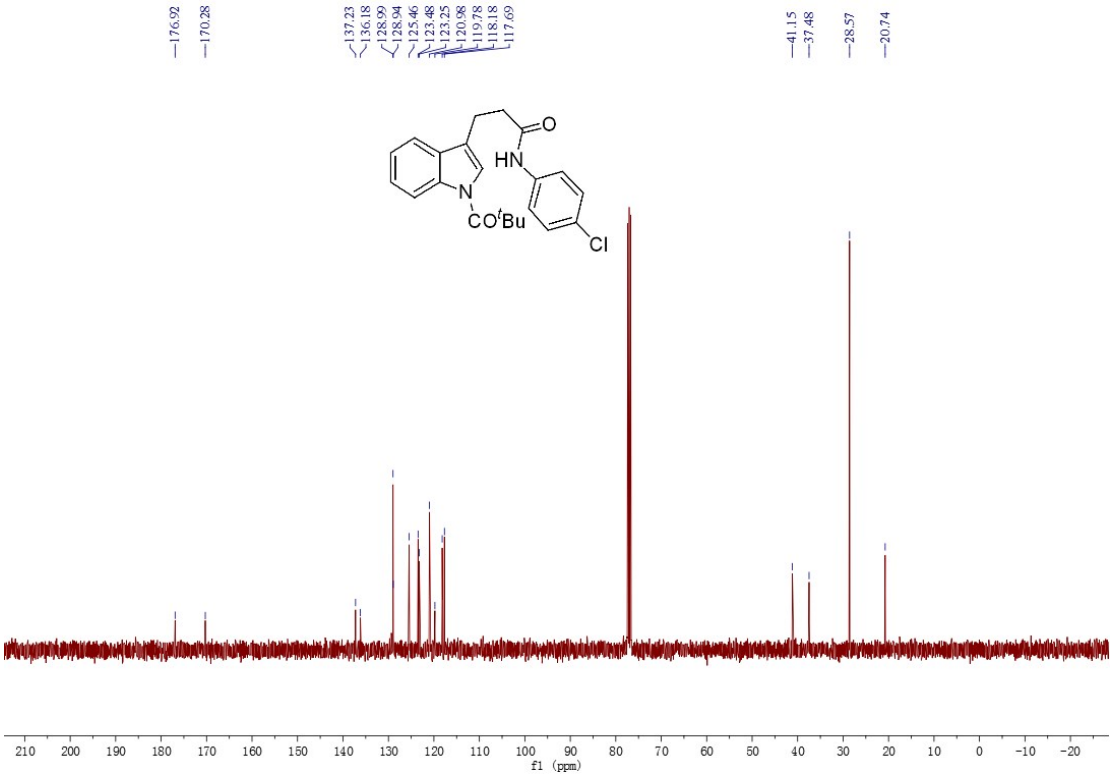
^{13}C NMR spectrum of compound **1c**



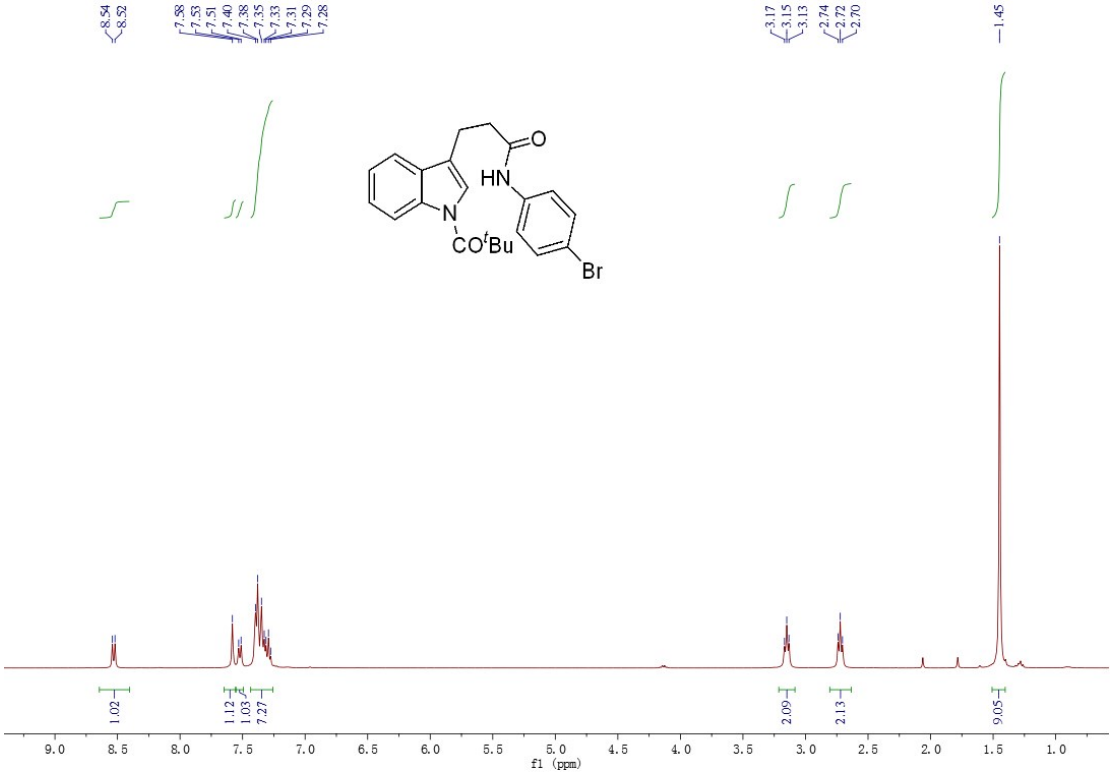
^{1}H NMR spectrum of compound **1d**



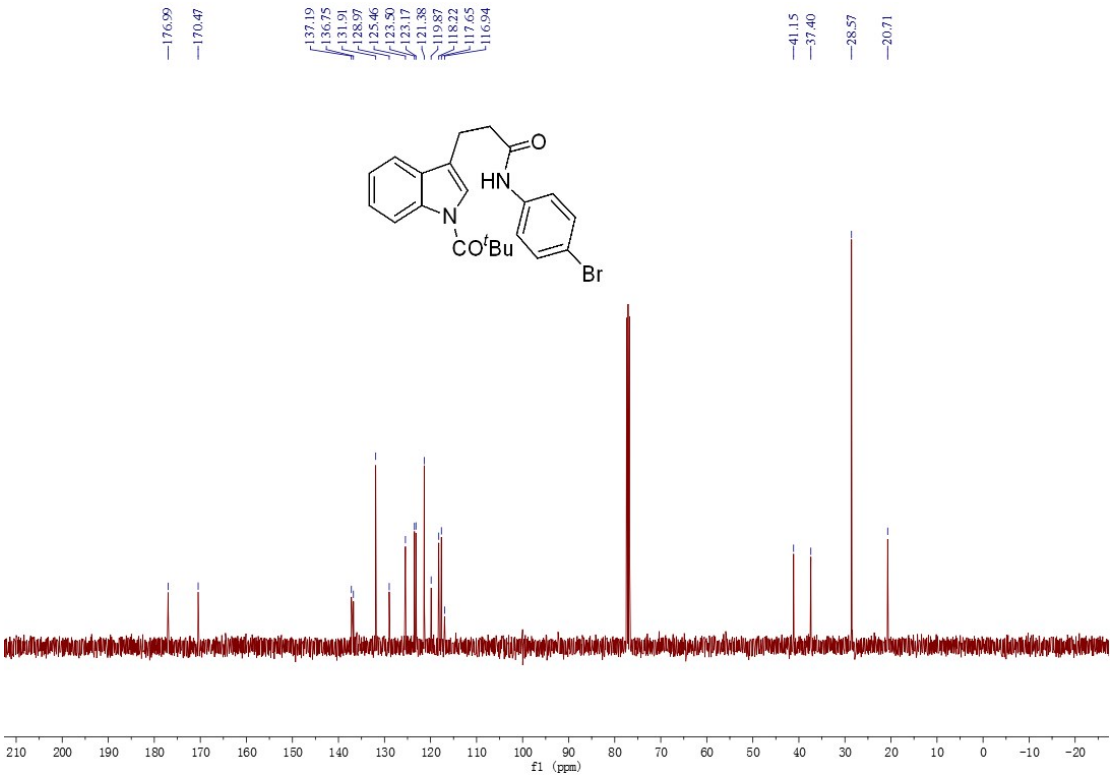
^{13}C NMR spectrum of compound **1d**



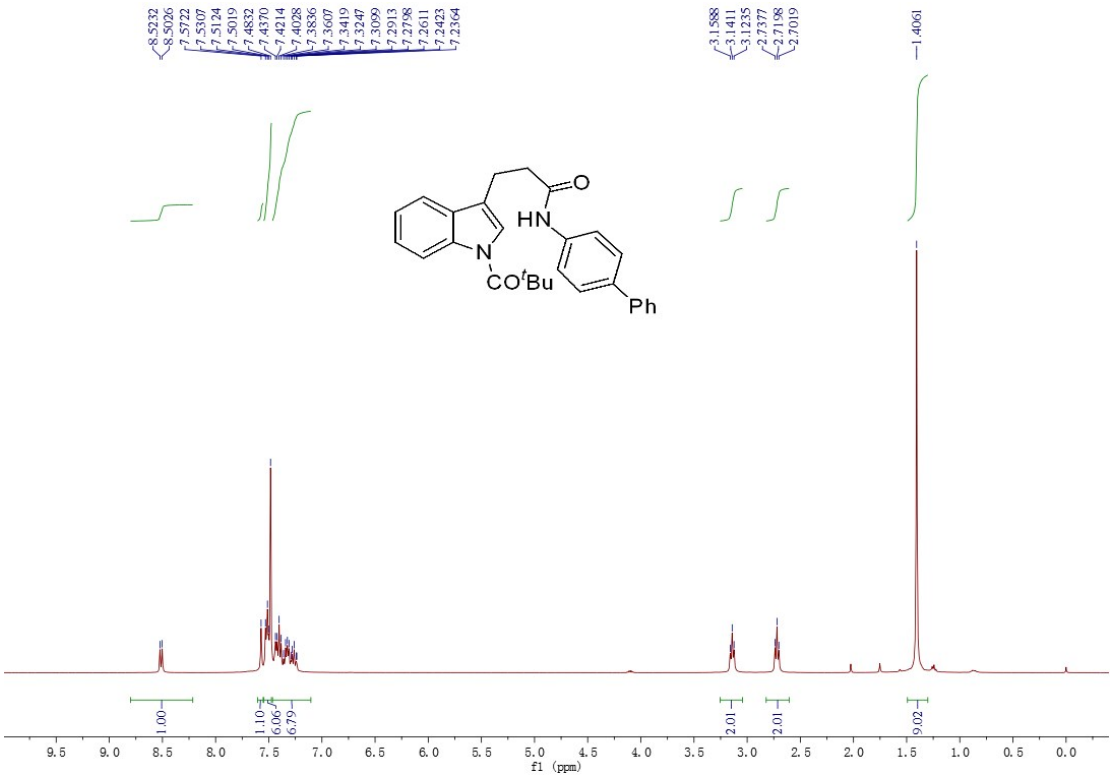
^{1}H NMR spectrum of compound **1e**



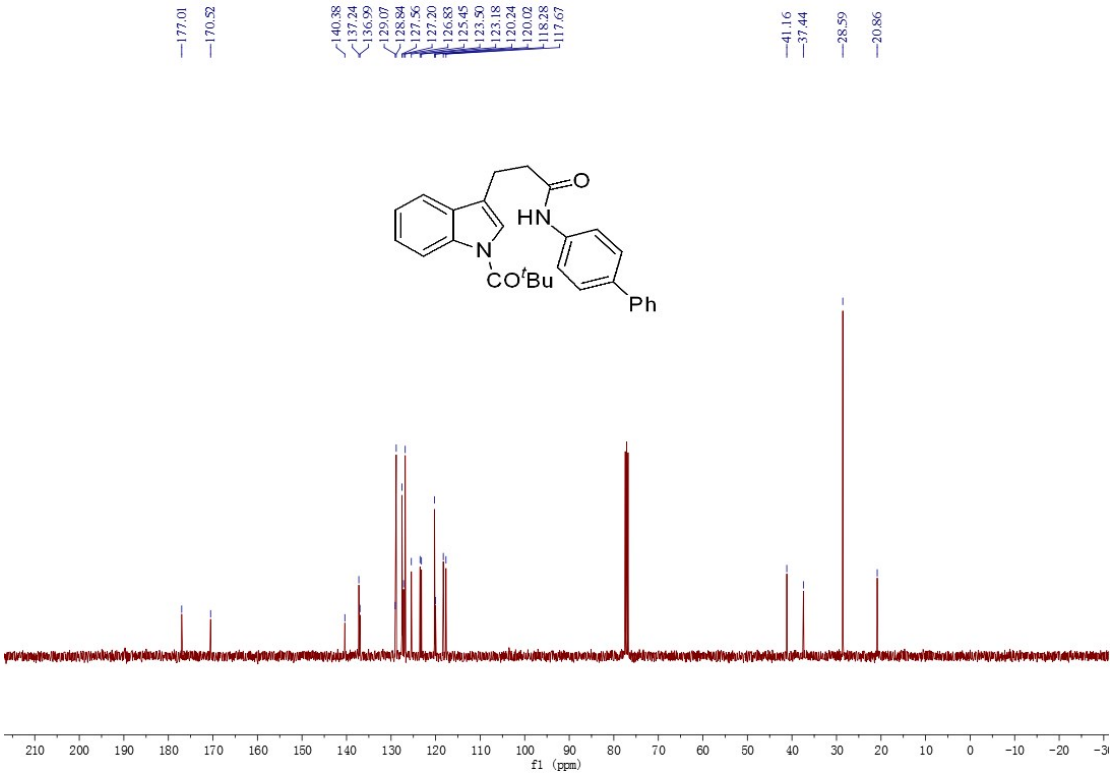
^{13}C NMR spectrum of compound **1e**



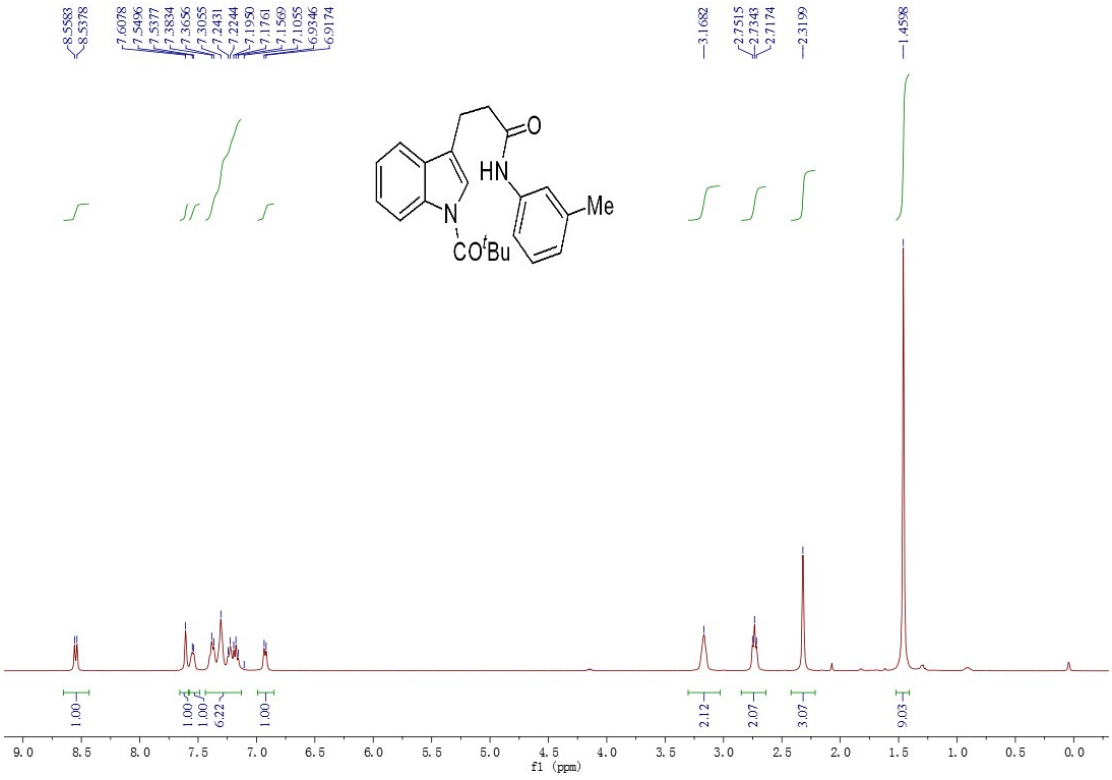
^{1}H NMR spectrum of compound **1f**



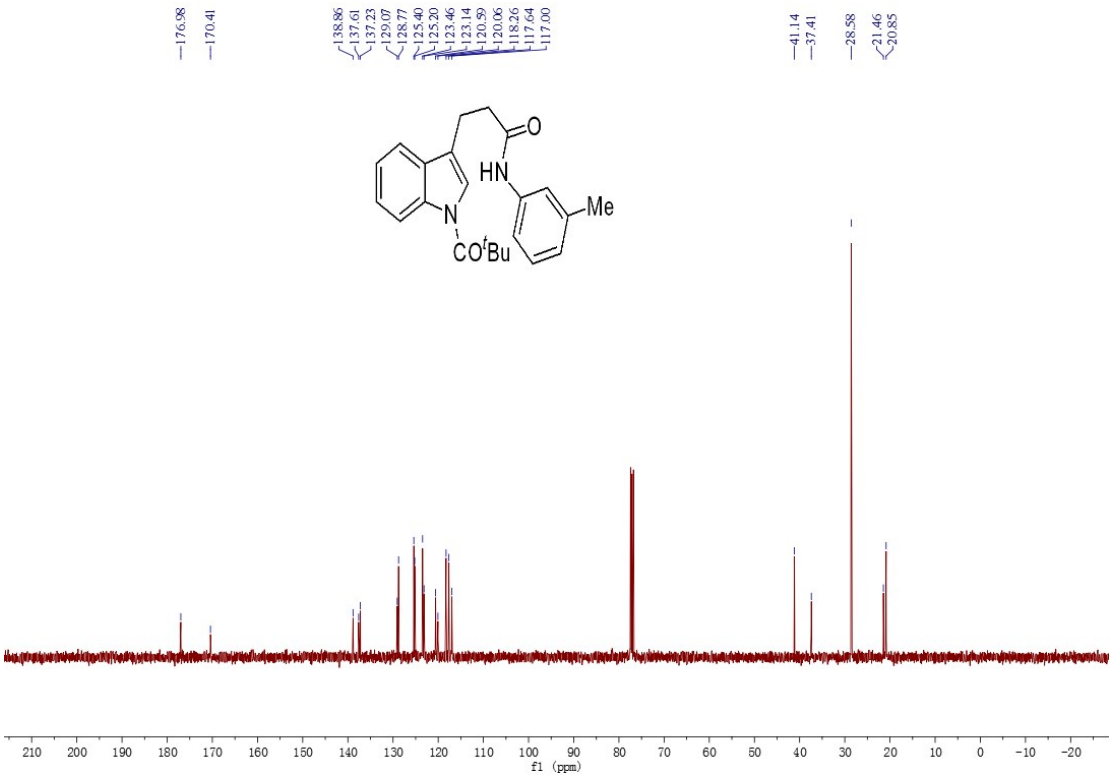
^{13}C NMR spectrum of compound **1f**



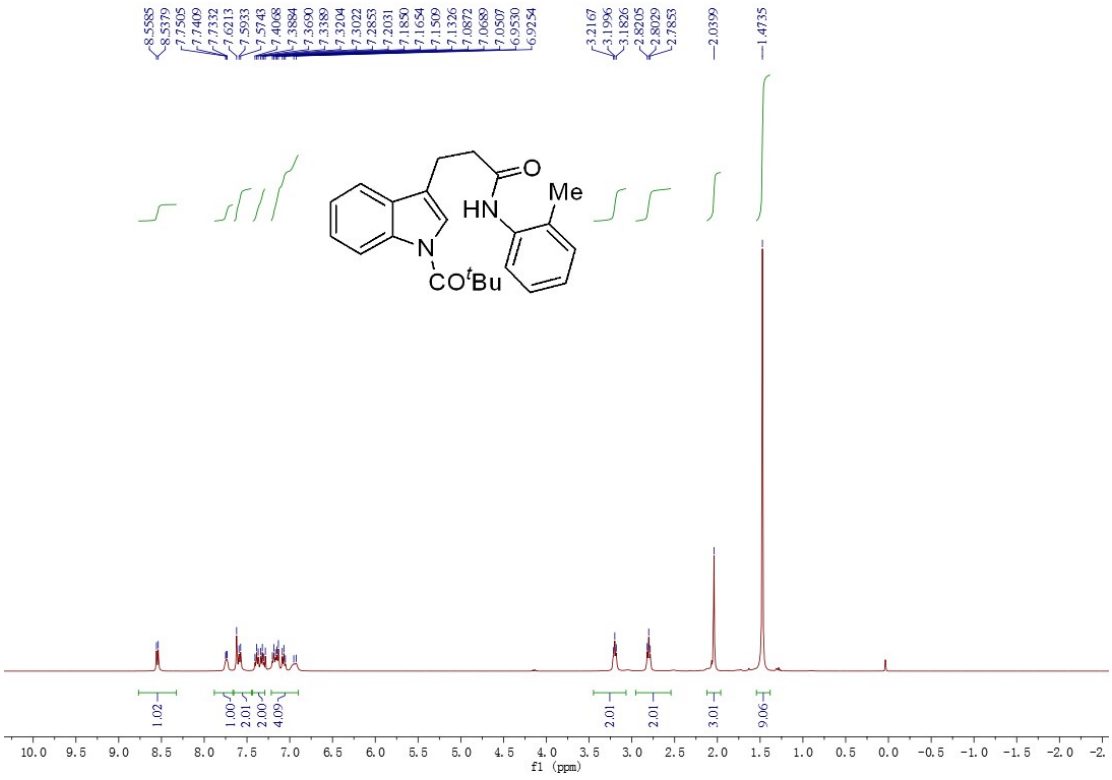
^{1}H NMR spectrum of compound **1g**



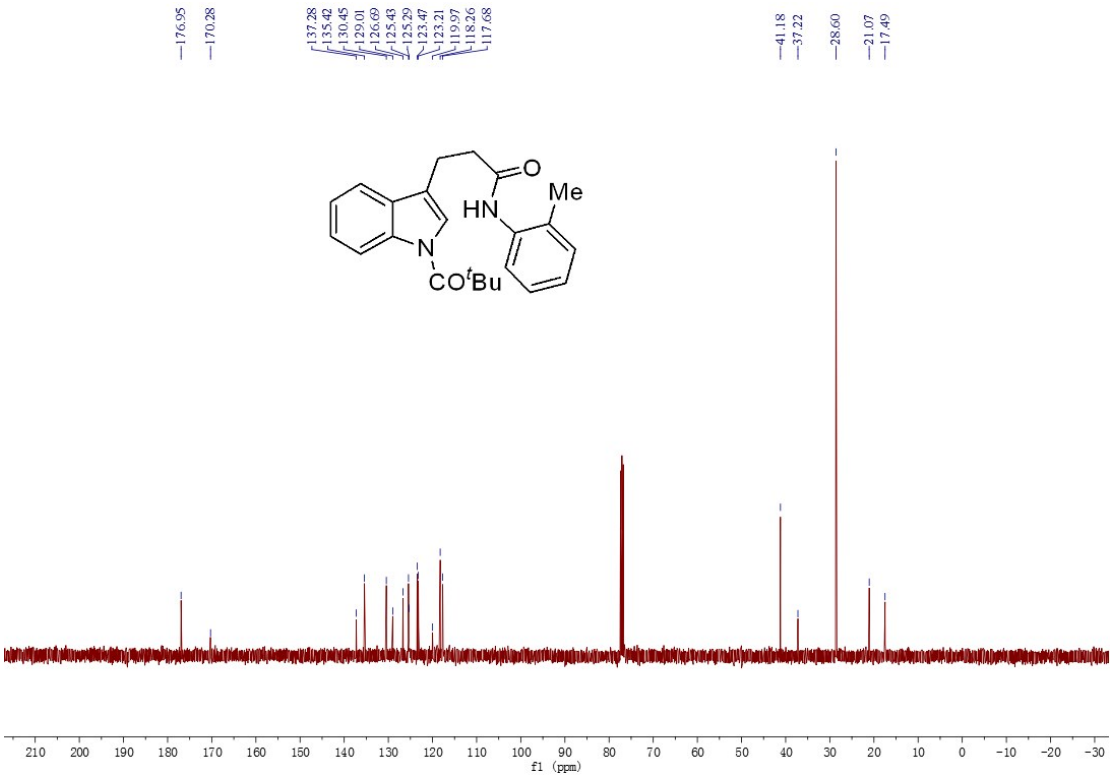
^{13}C NMR spectrum of compound **1g**



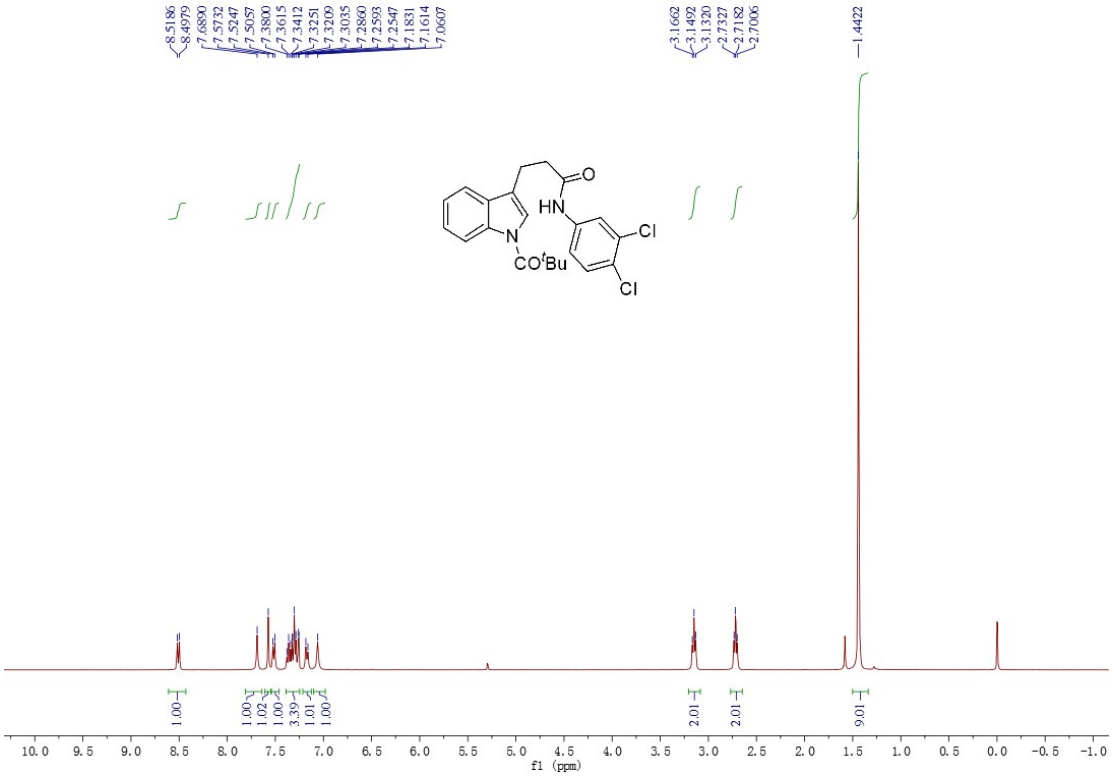
^{1}H NMR spectrum of compound **1h**



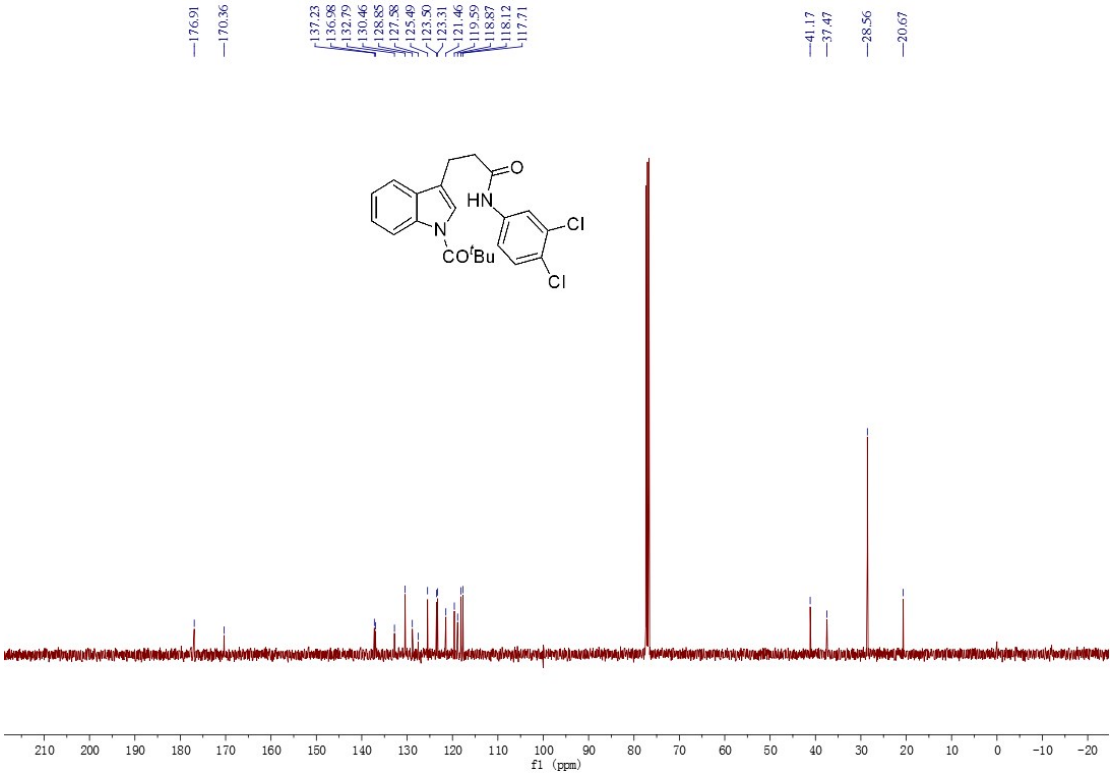
^{13}C NMR spectrum of compound **1h**



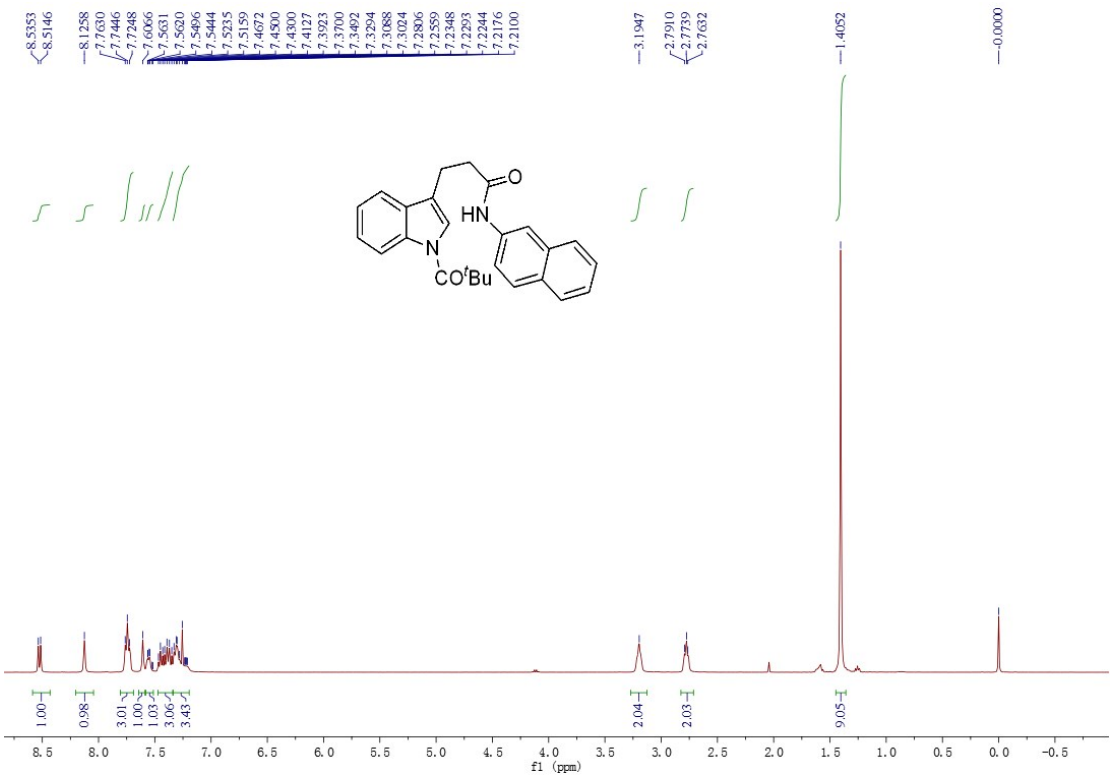
^{1}H NMR spectrum of compound **1i**



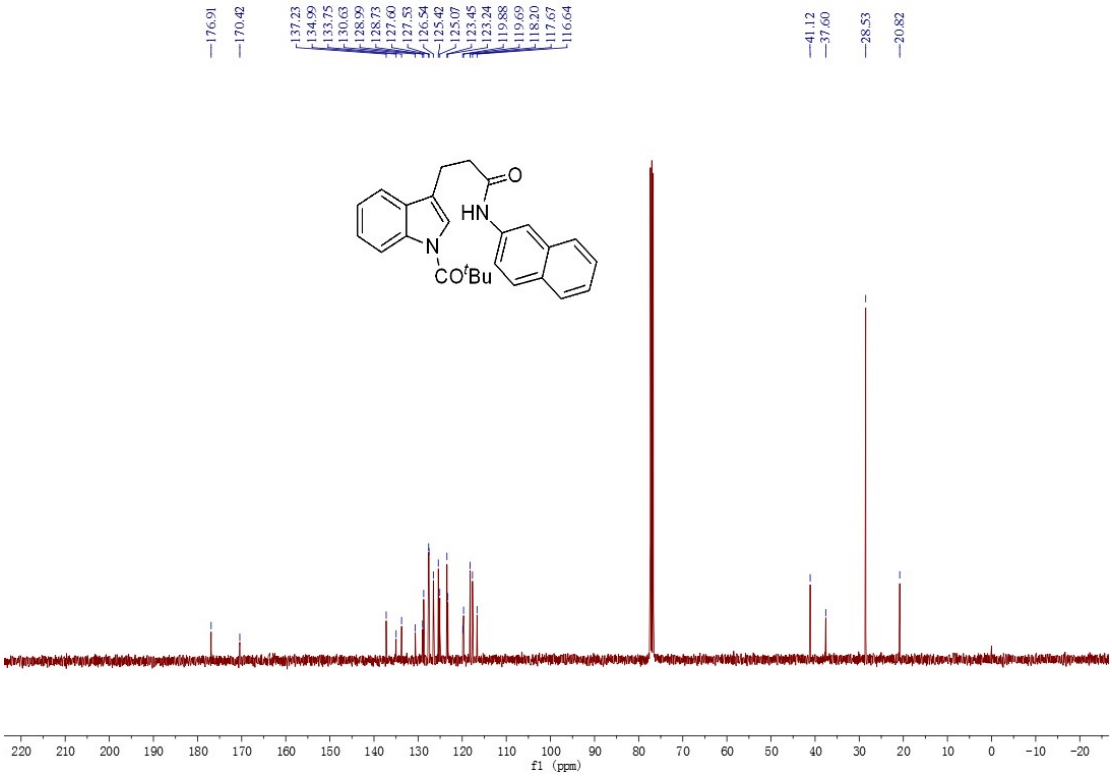
^{13}C NMR spectrum of compound **1i**



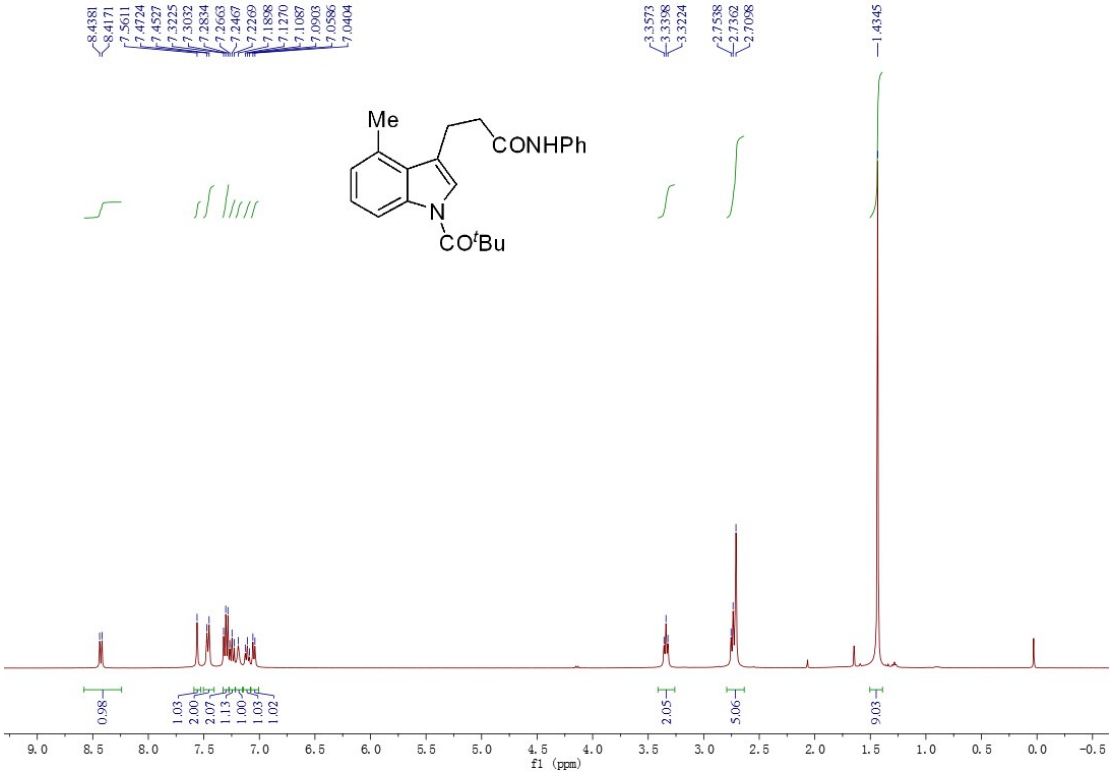
^{1}H NMR spectrum of compound **1j**



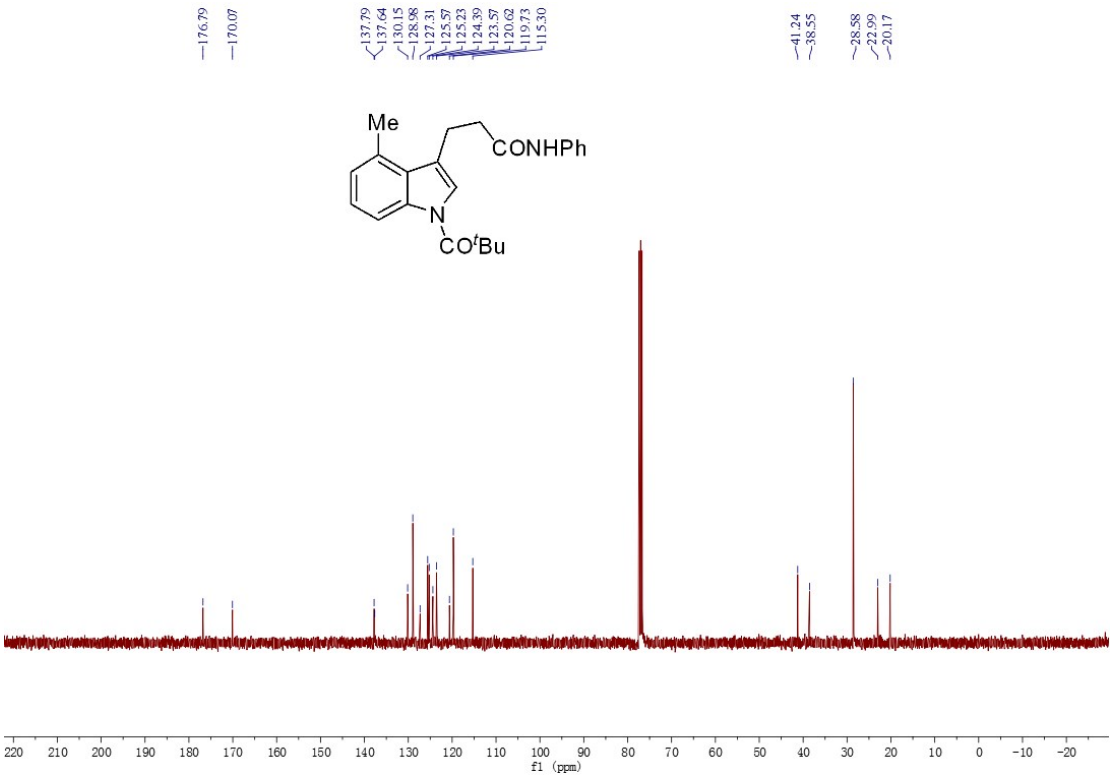
^{13}C NMR spectrum of compound **1j**



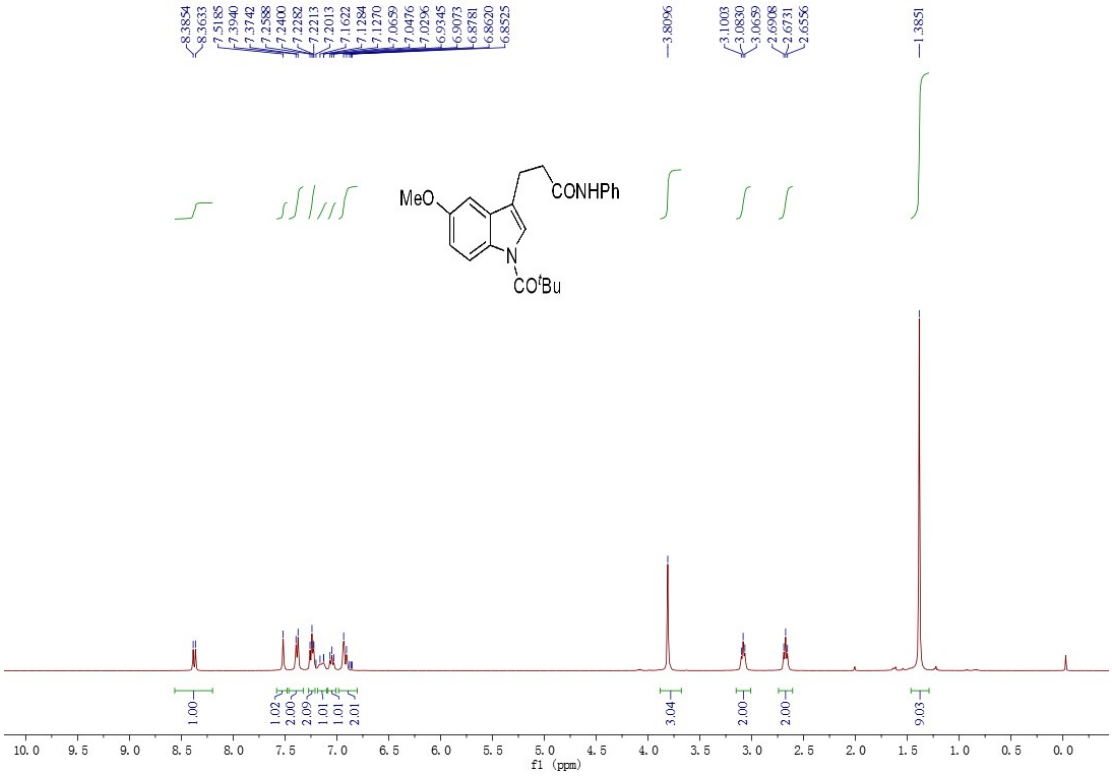
^{1}H NMR spectrum of compound **1k**



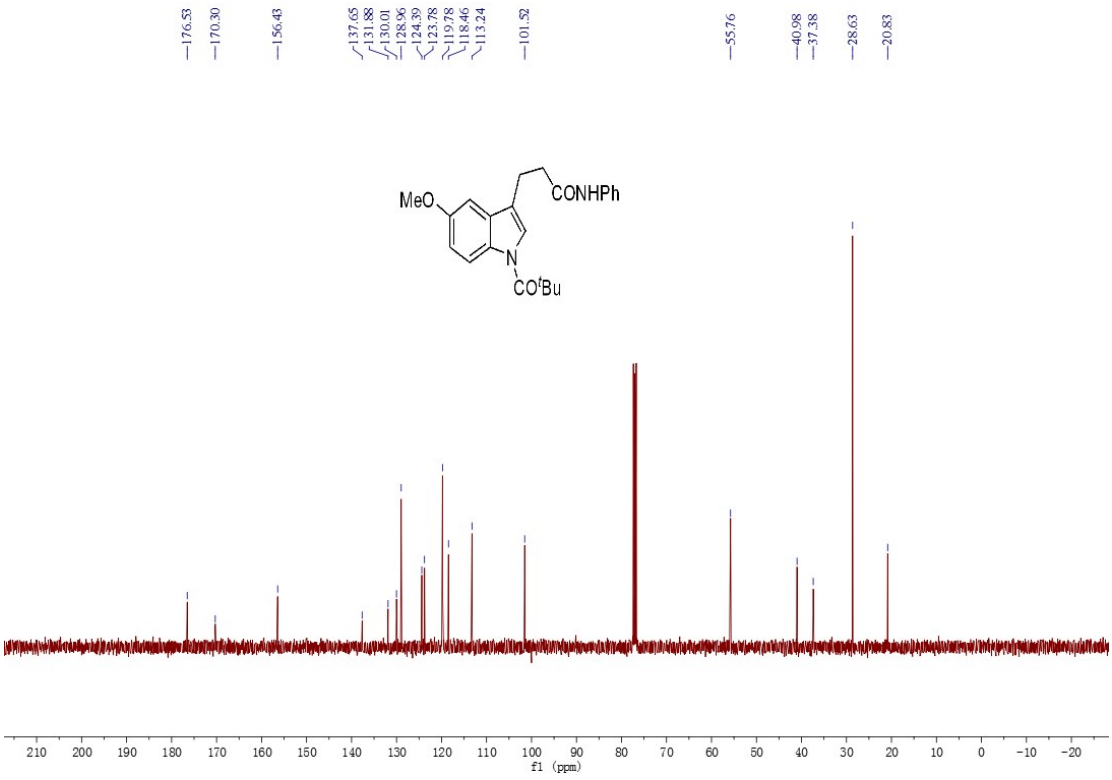
^{13}C NMR spectrum of compound **1k**



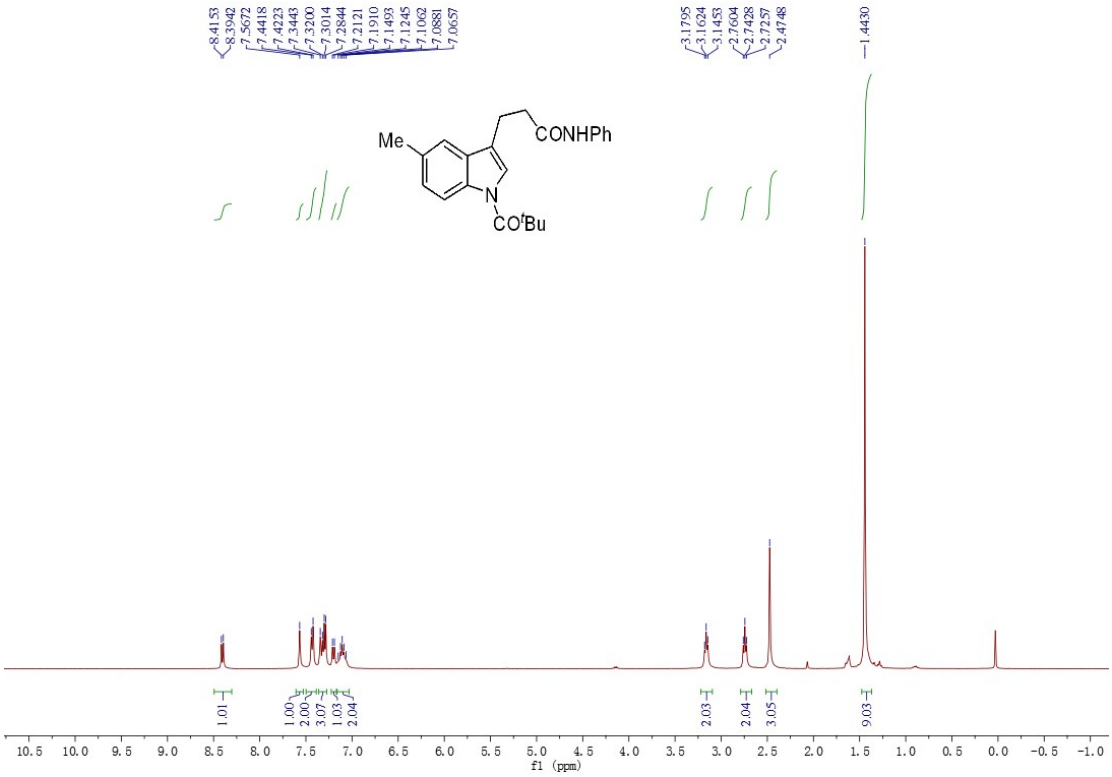
^{1}H NMR spectrum of compound **1l**



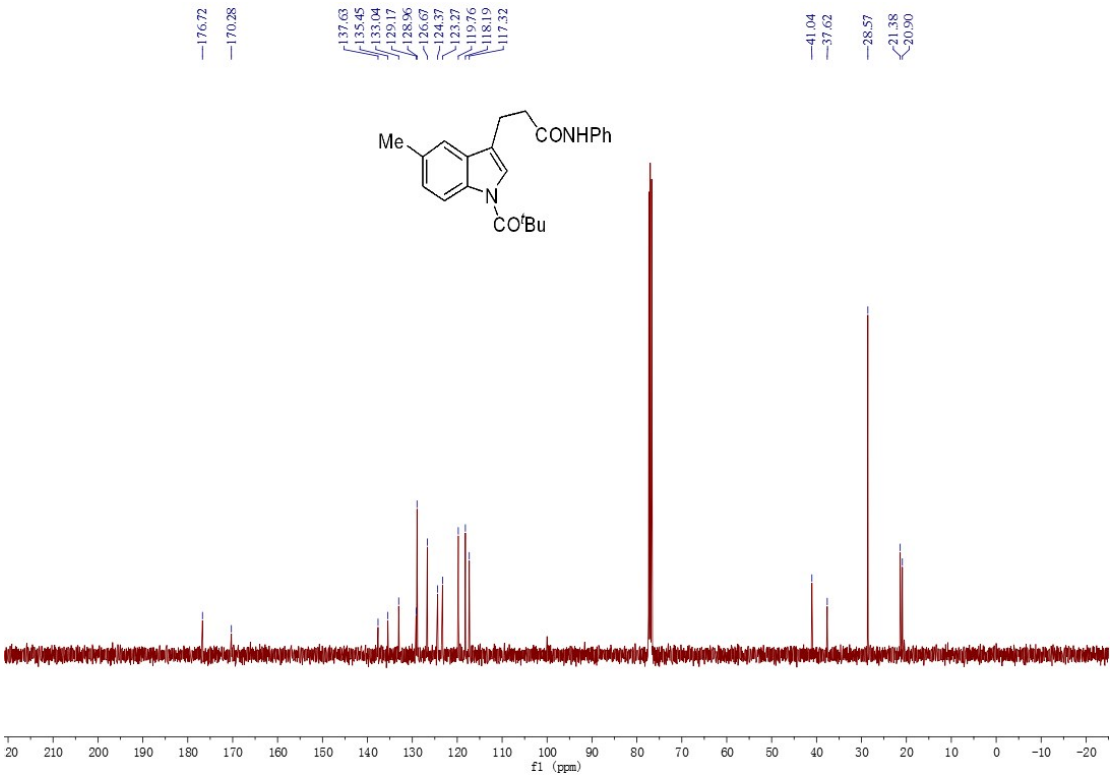
^{13}C NMR spectrum of compound **1l**



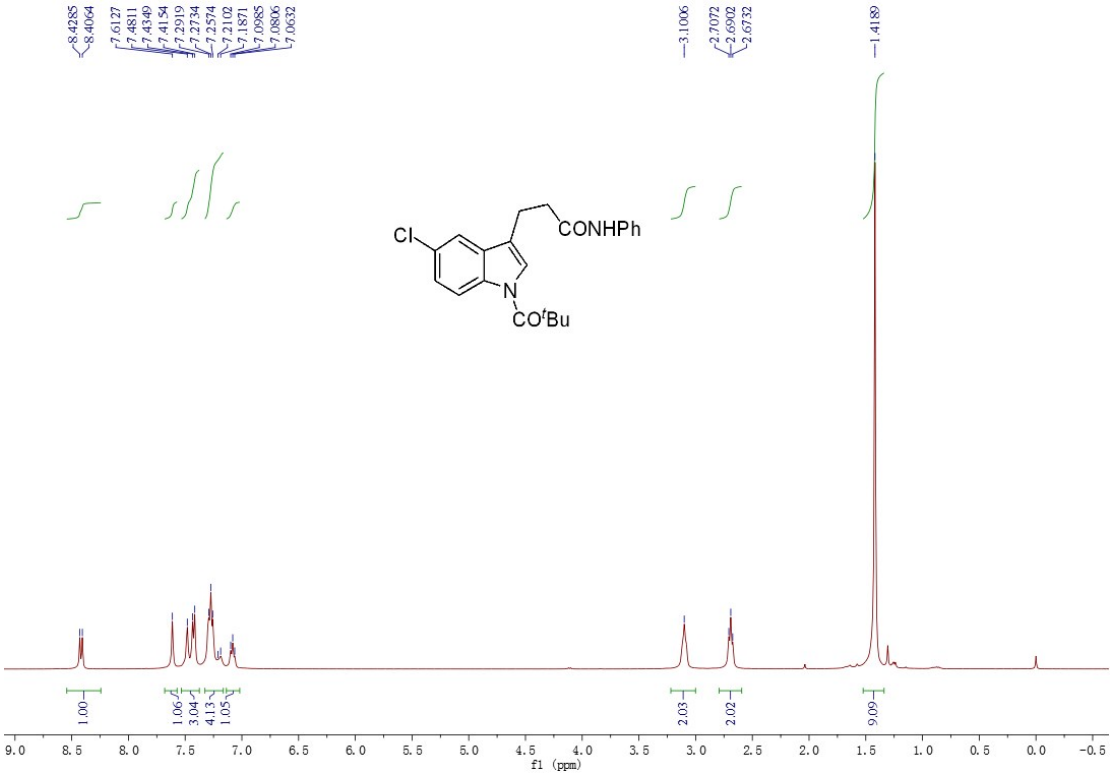
^{1}H NMR spectrum of compound **1m**



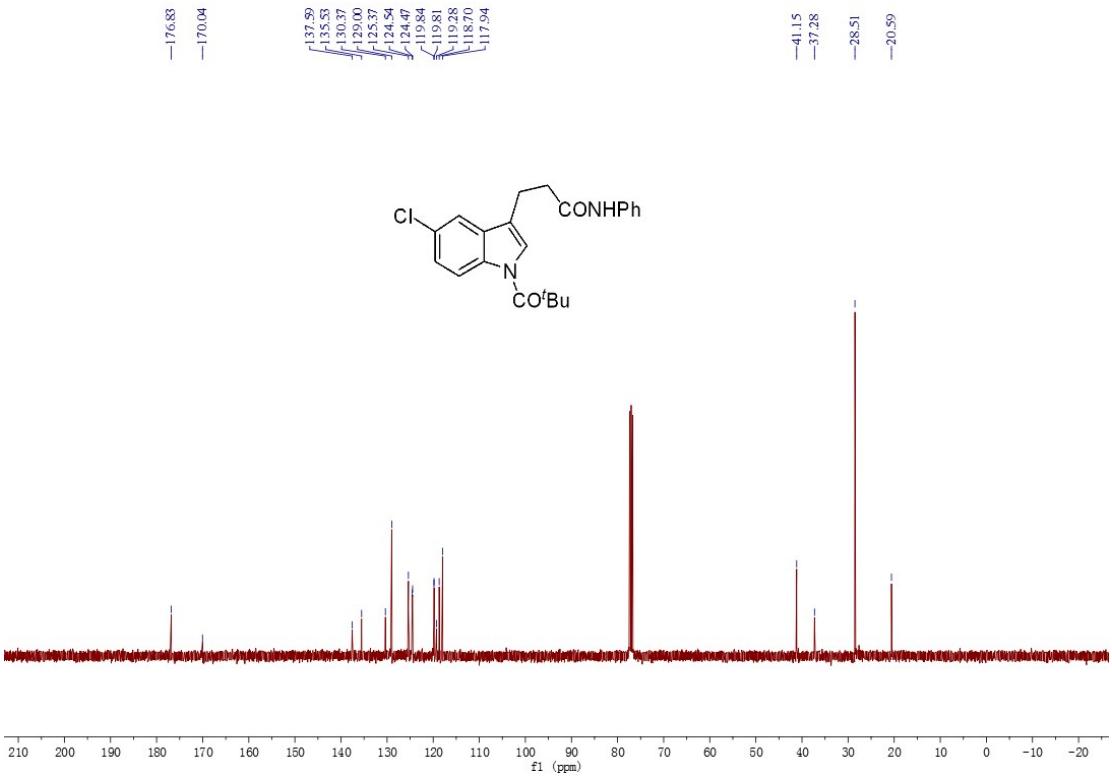
^{13}C NMR spectrum of compound **1m**



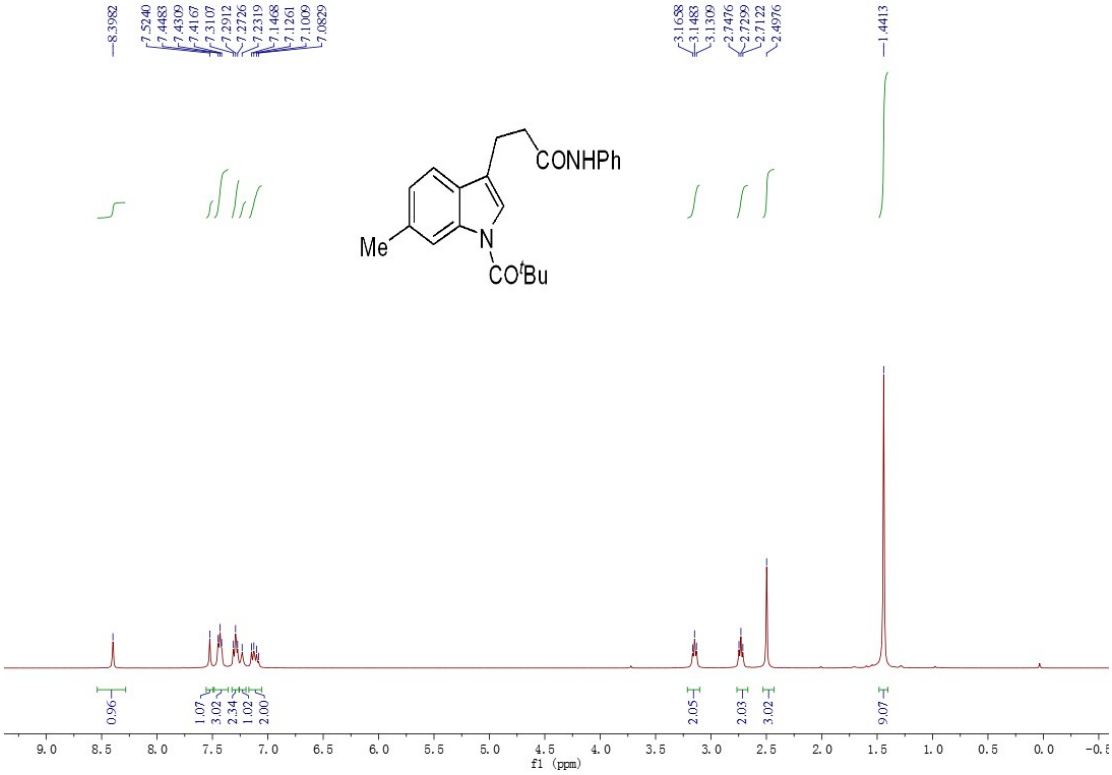
^{1}H NMR spectrum of compound **1n**



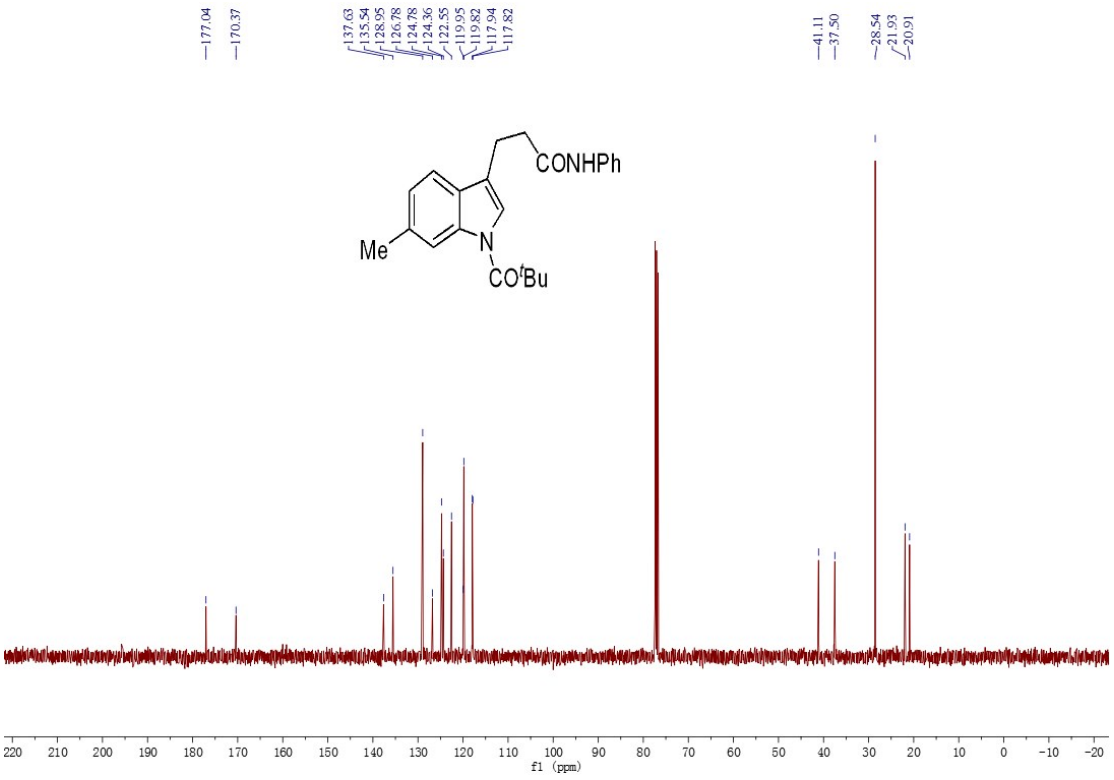
^{13}C NMR spectrum of compound **1n**



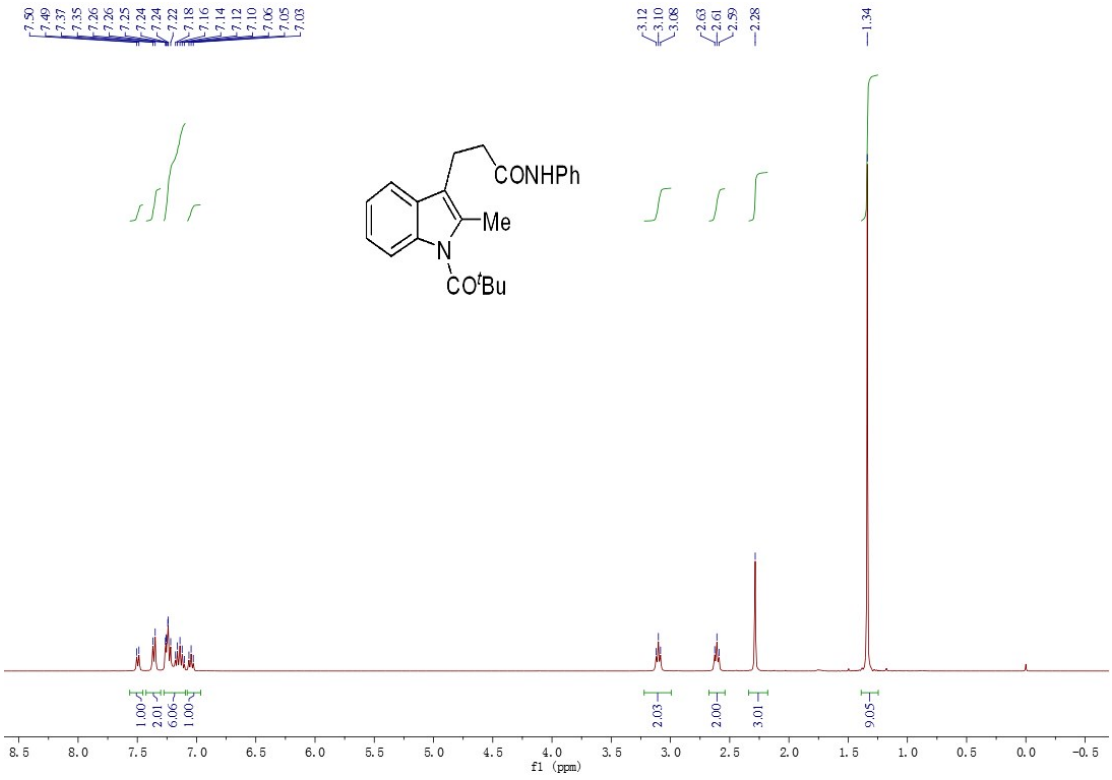
^{1}H NMR spectrum of compound **1o**



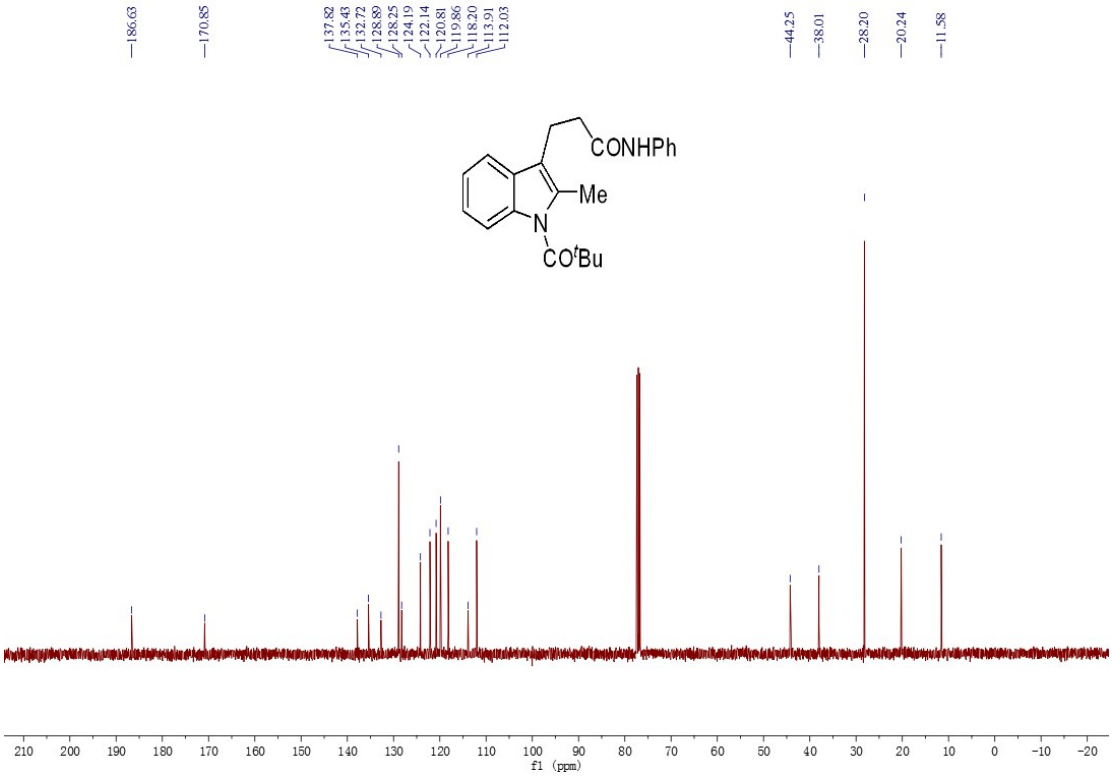
^{13}C NMR spectrum of compound **1o**



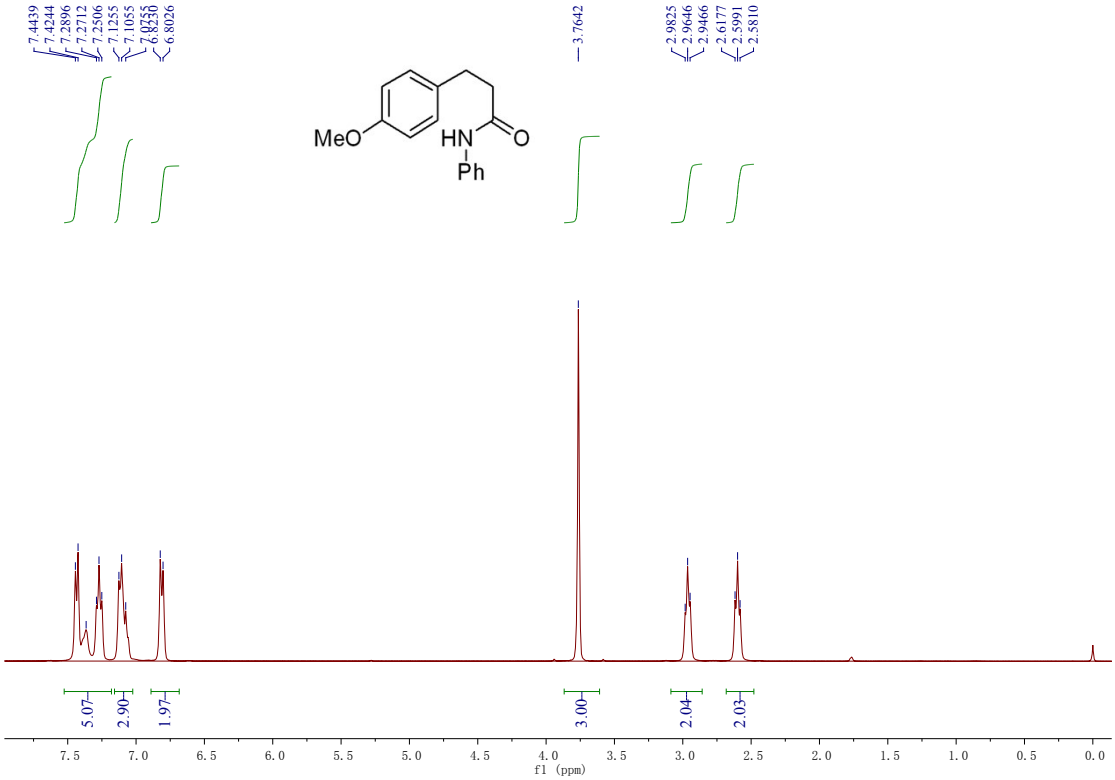
^{1}H NMR spectrum of compound **1t**



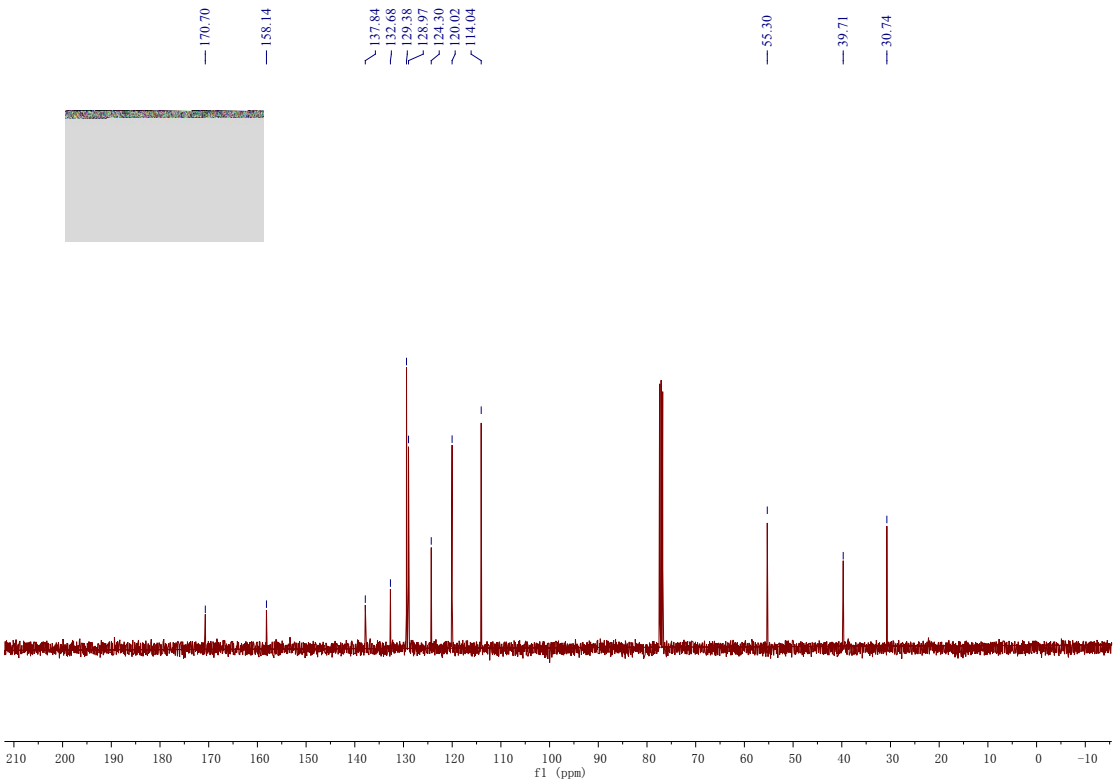
^{13}C NMR spectrum of compound **1t**



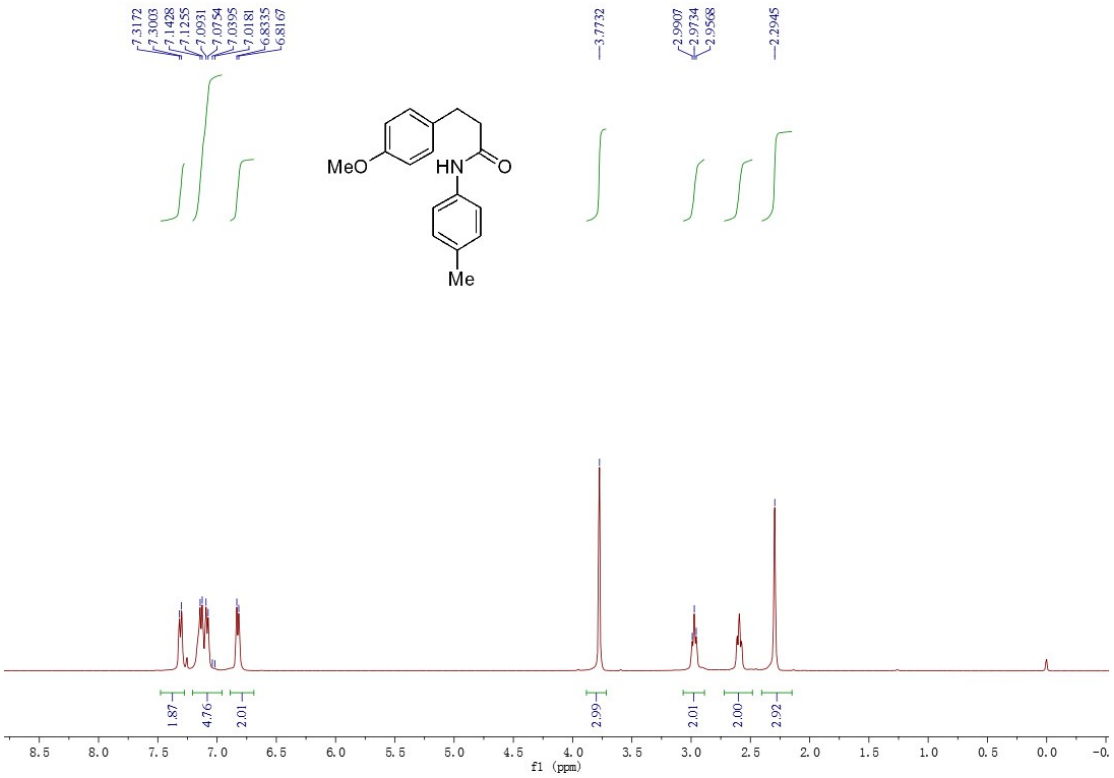
^{1}H NMR spectrum of compound **4a**



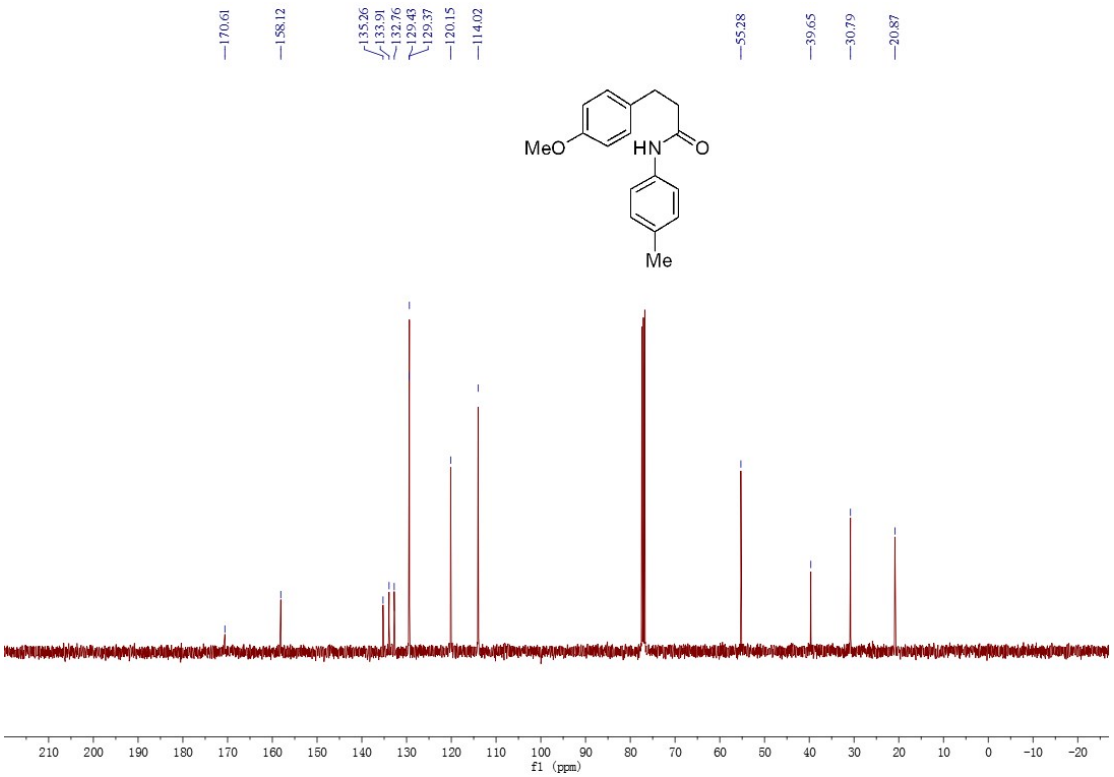
^{13}C NMR spectrum of compound **4a**



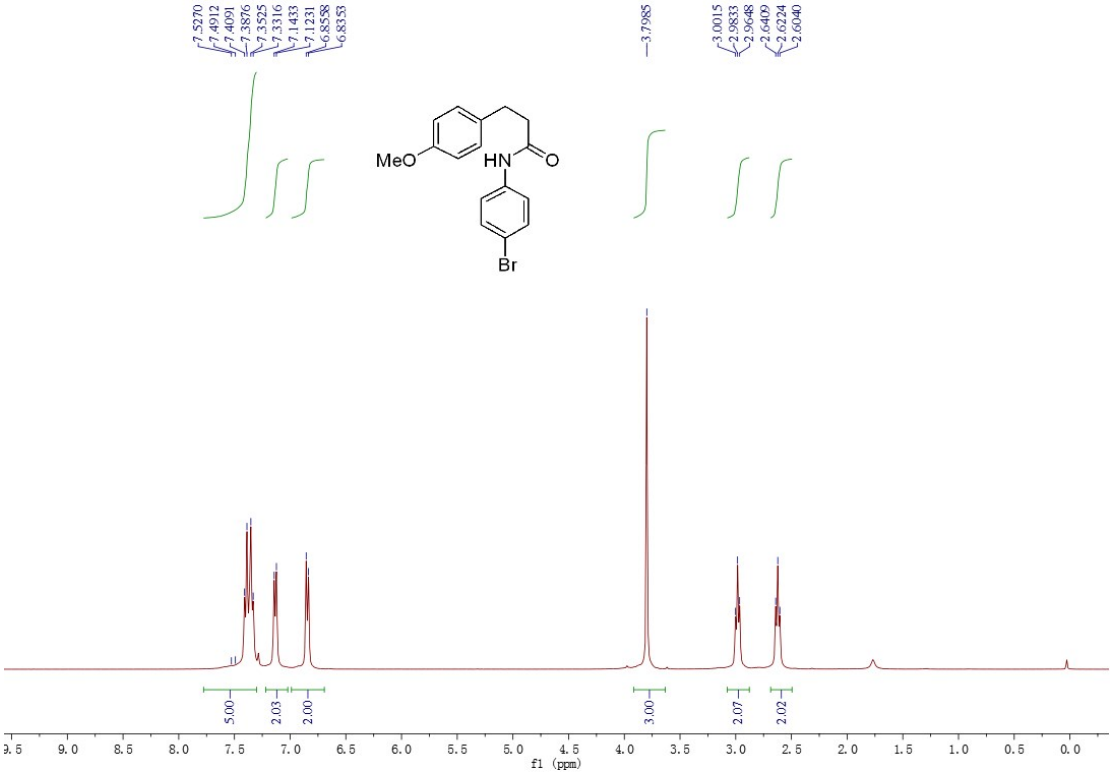
^{1}H NMR spectrum of compound **4b**



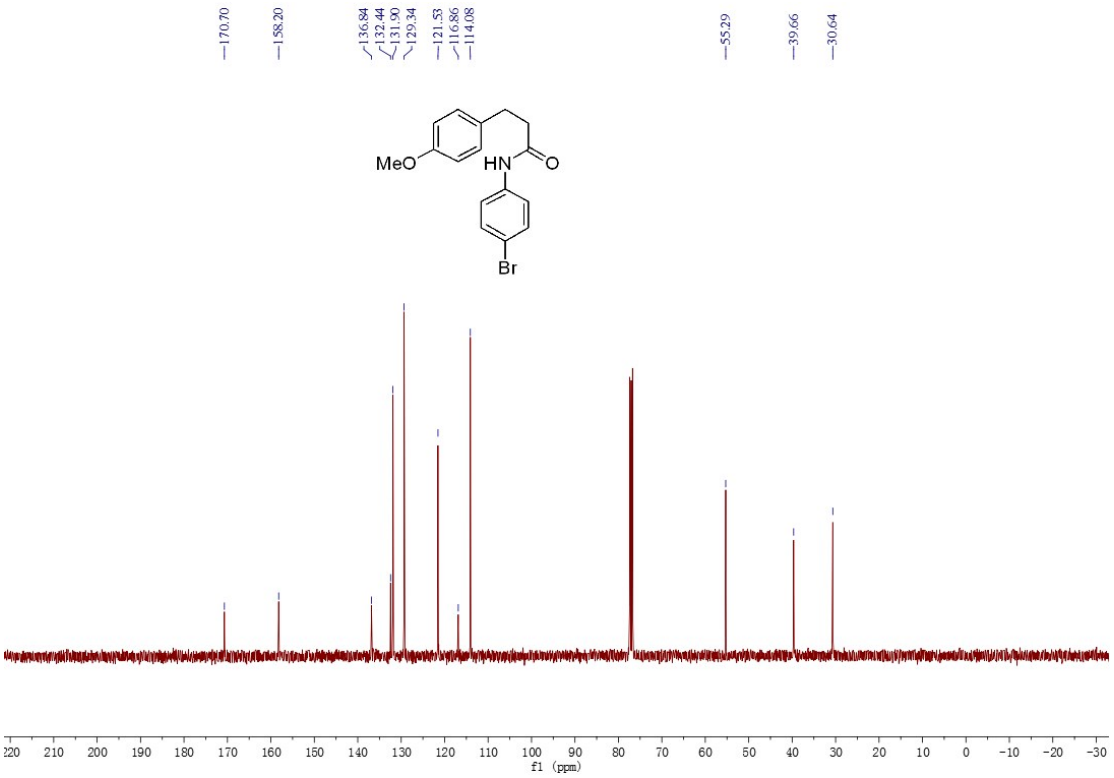
^{13}C NMR spectrum of compound **4b**



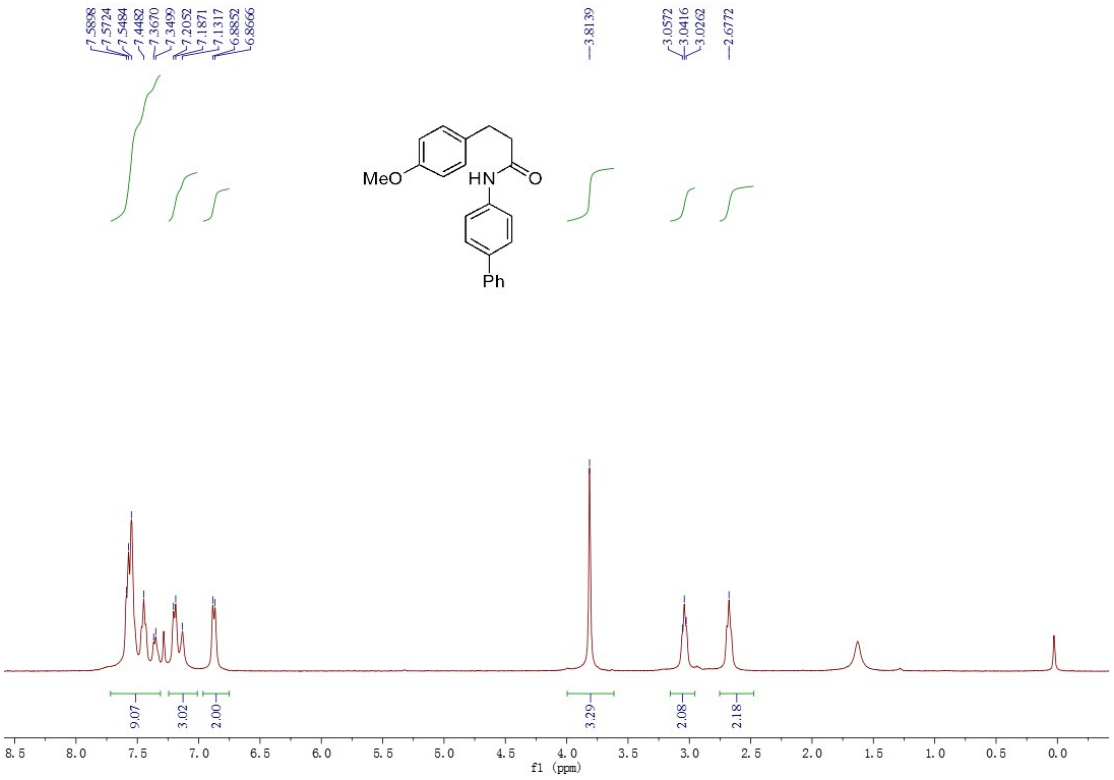
^{1}H NMR spectrum of compound **4c**



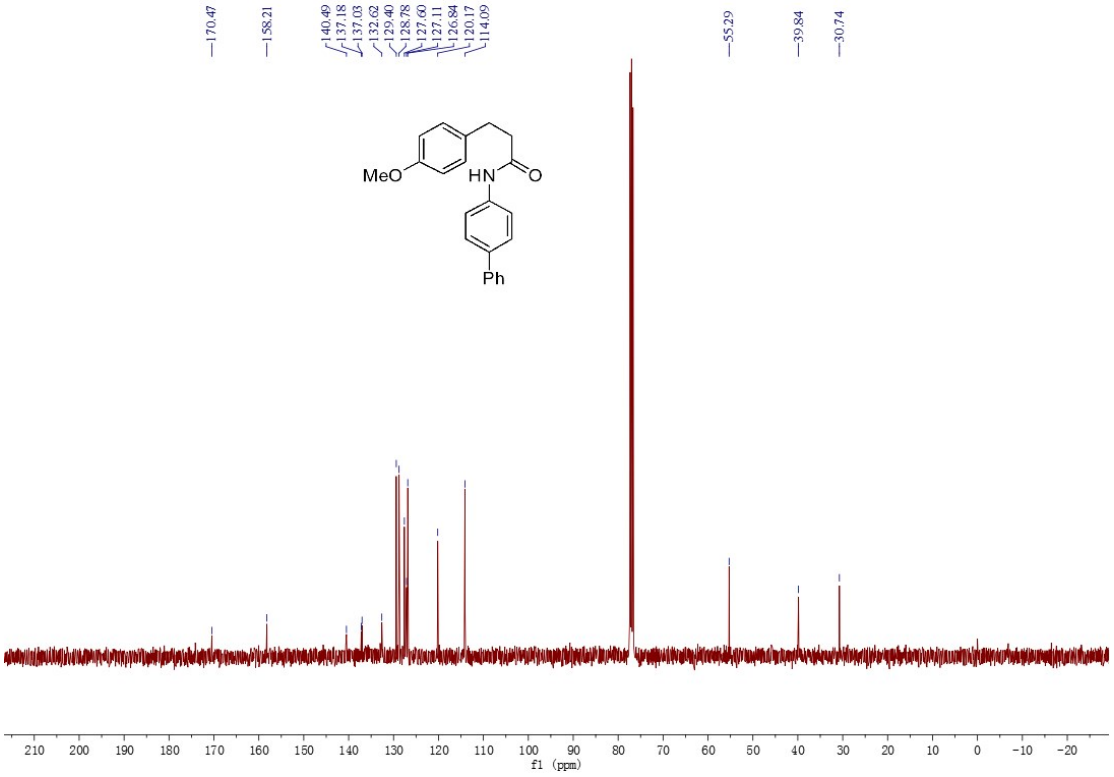
^{13}C NMR spectrum of compound **4c**



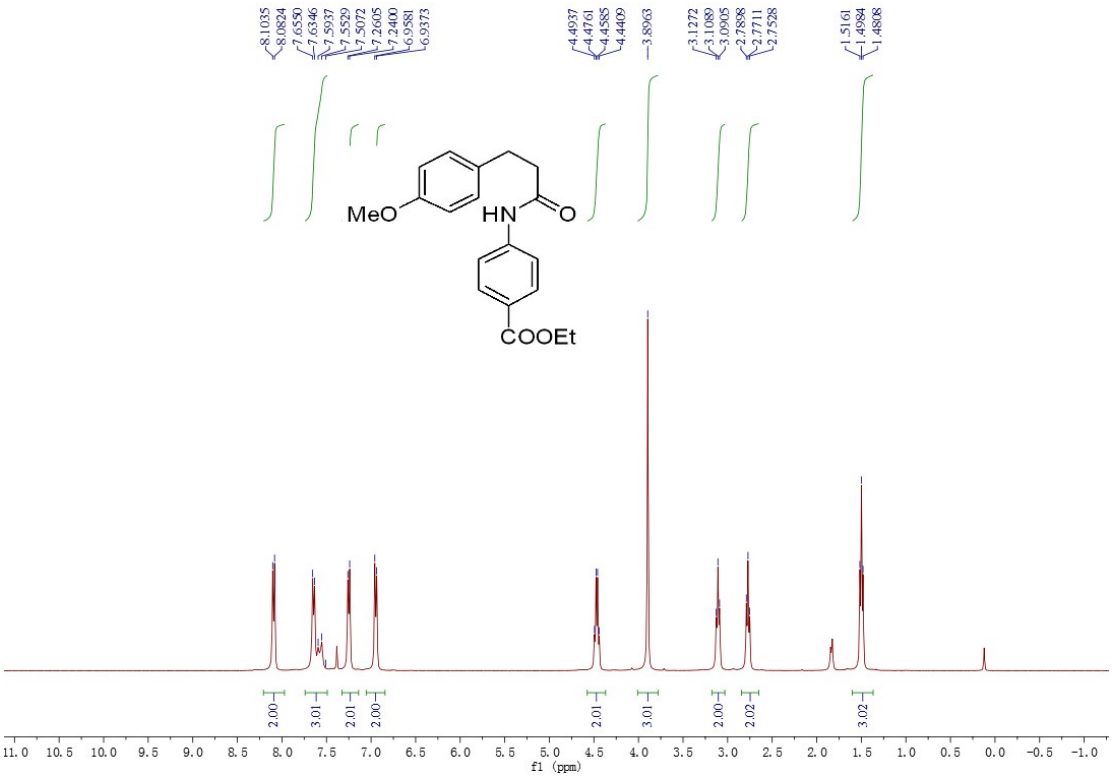
^{1}H NMR spectrum of compound **4d**



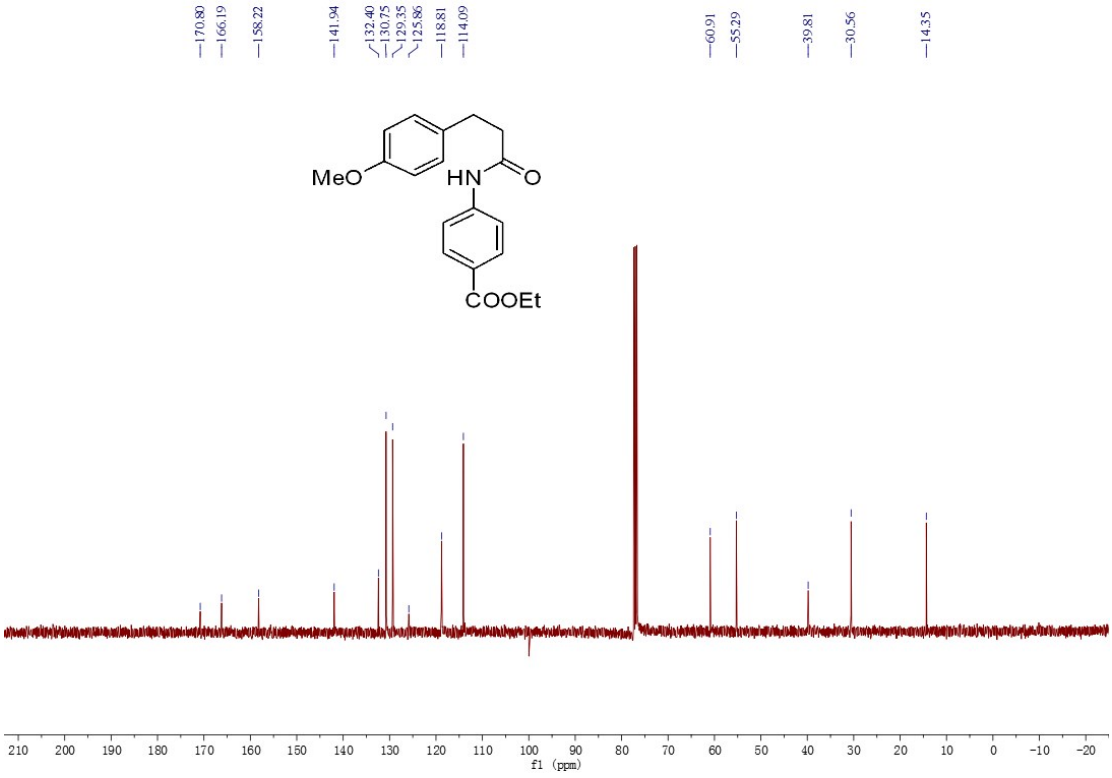
^{13}C NMR spectrum of compound **4d**



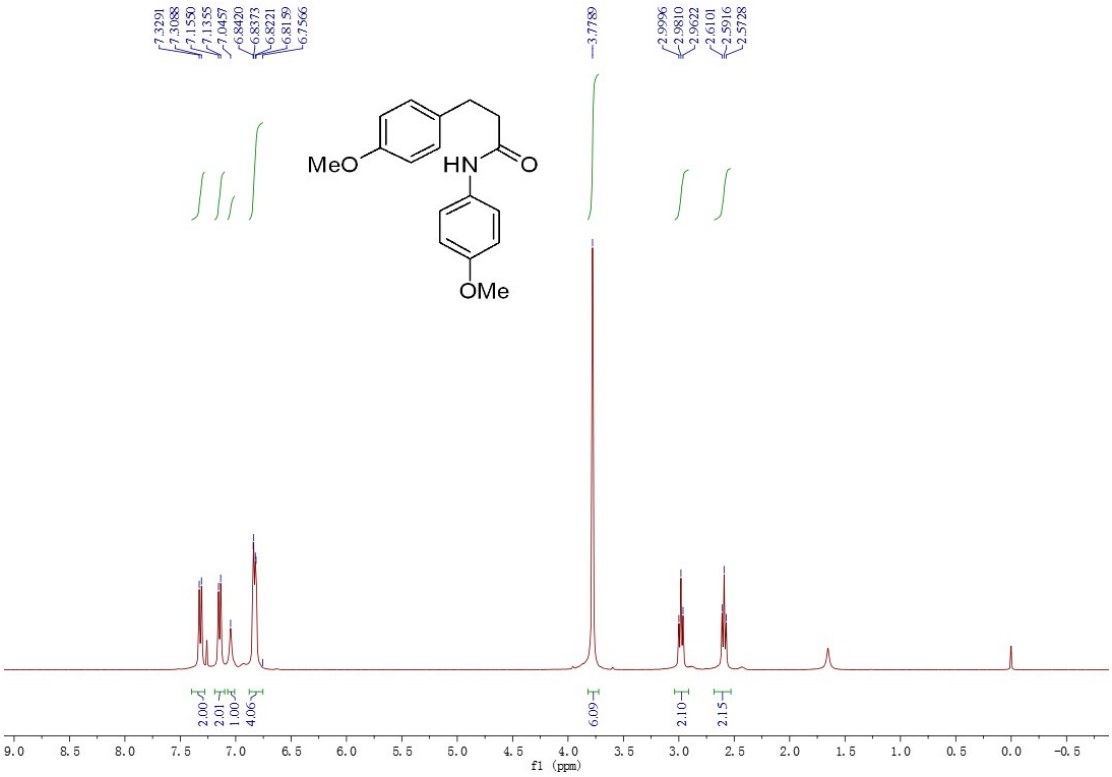
^{1}H NMR spectrum of compound **4e**



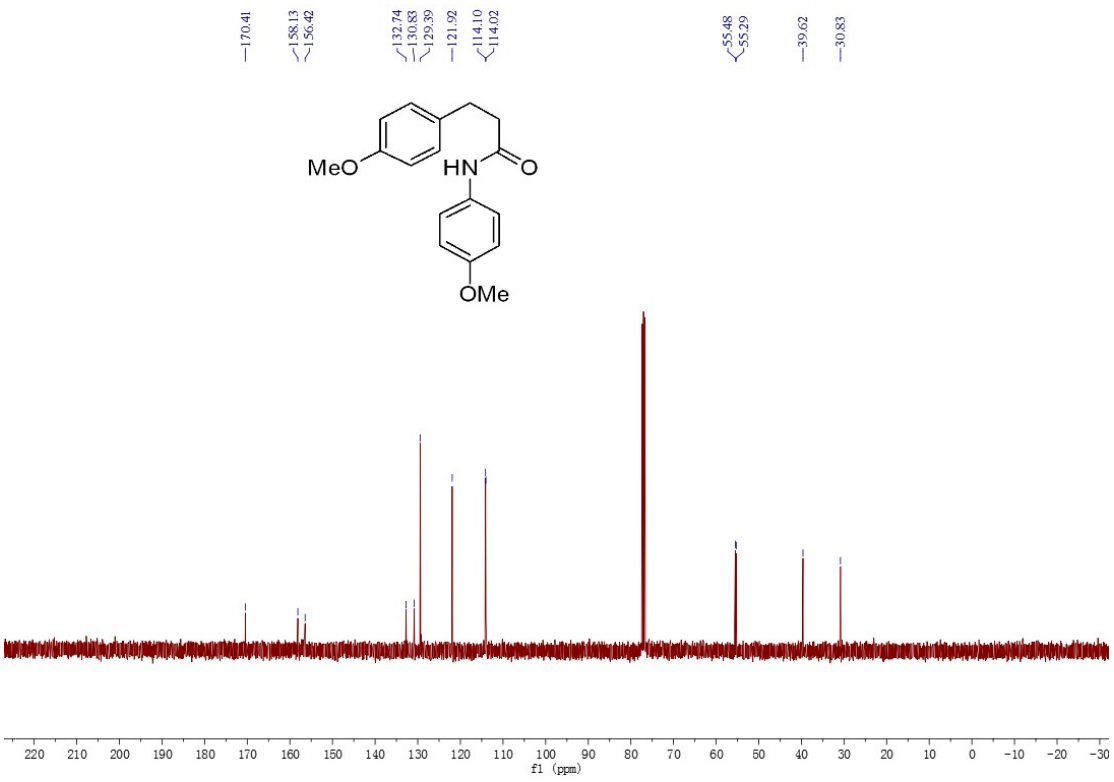
^{13}C NMR spectrum of compound **4e**



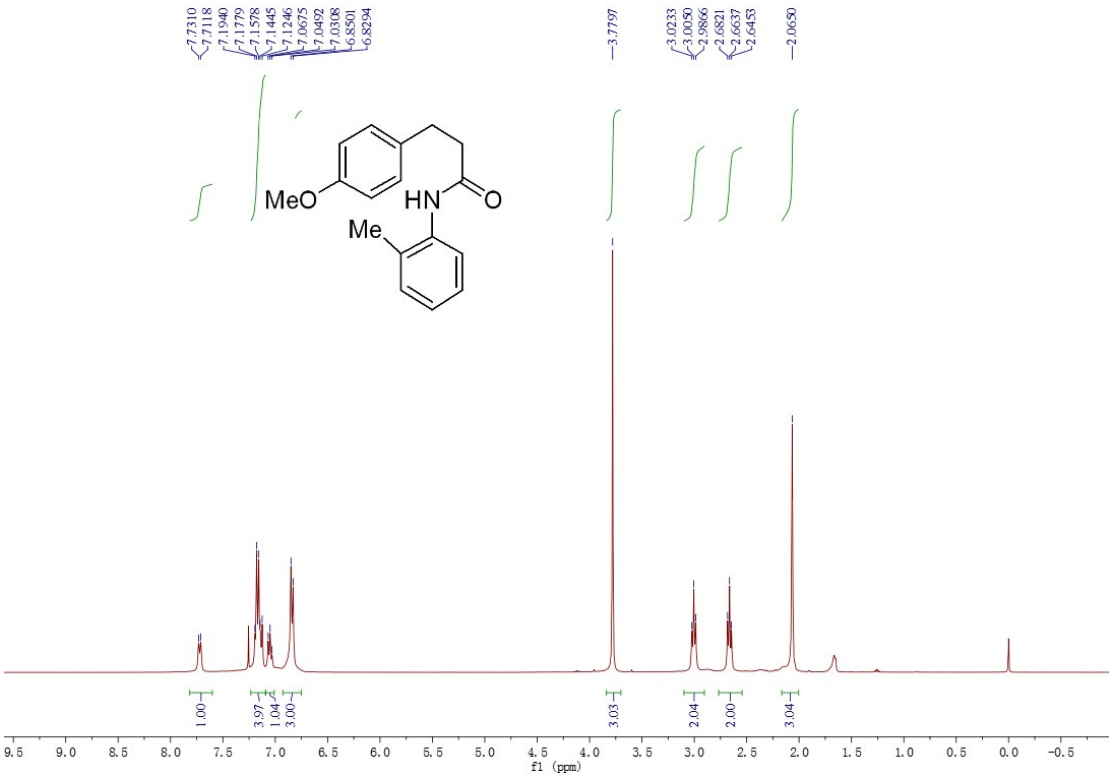
^{1}H NMR spectrum of compound **4f**



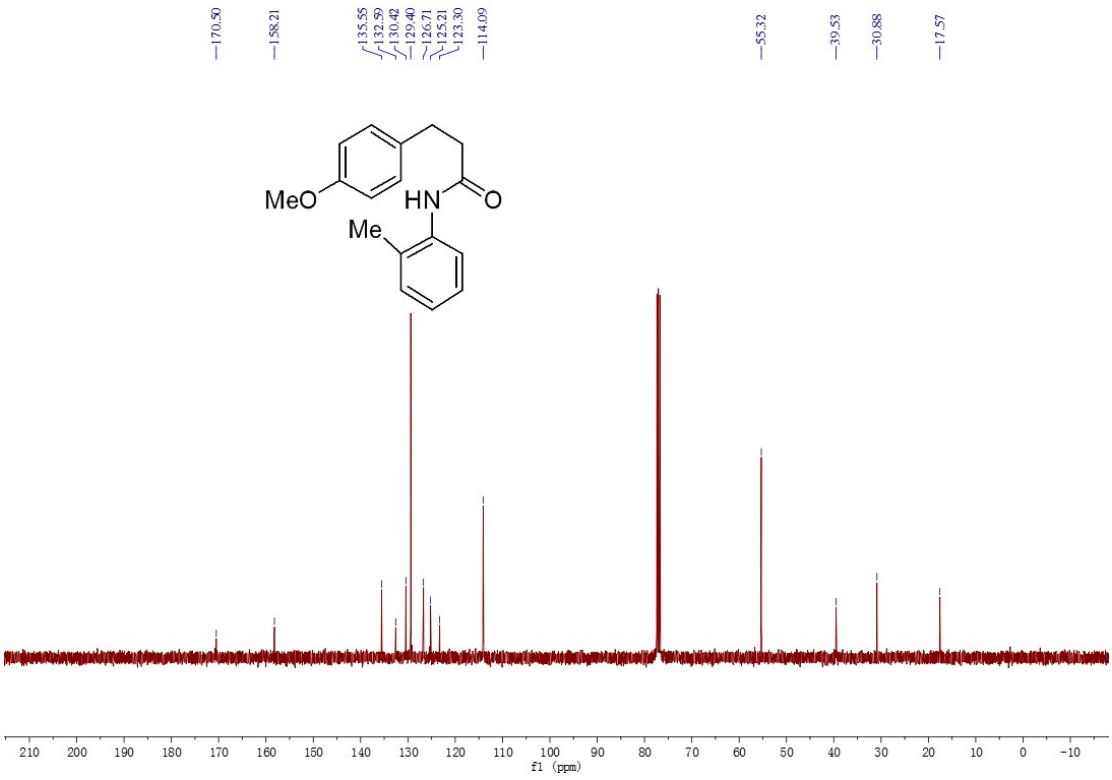
^{13}C NMR spectrum of compound **4f**



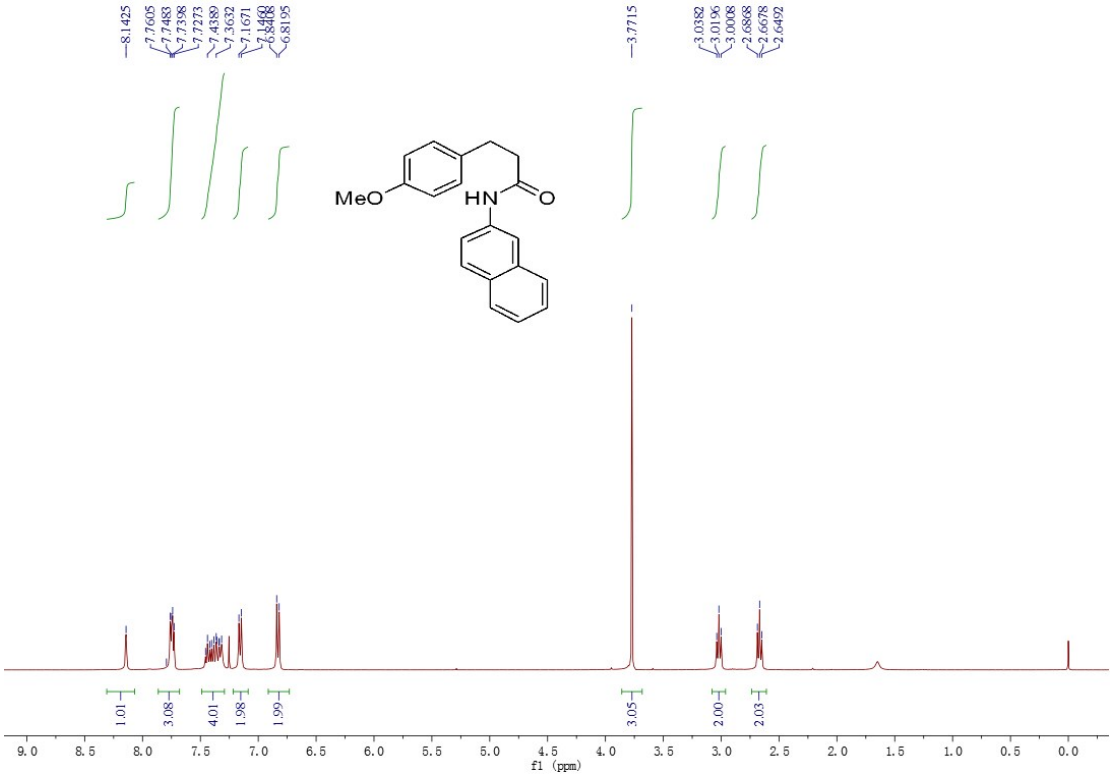
^{1}H NMR spectrum of compound **4g**



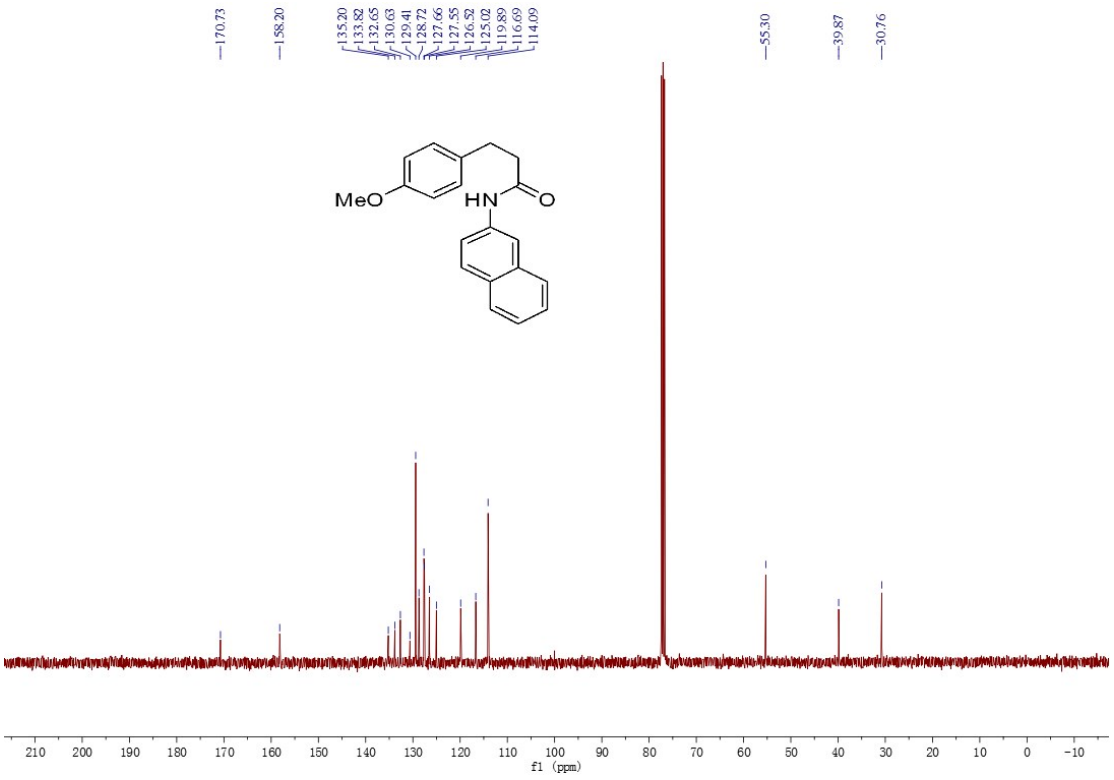
^{13}C NMR spectrum of compound **4g**



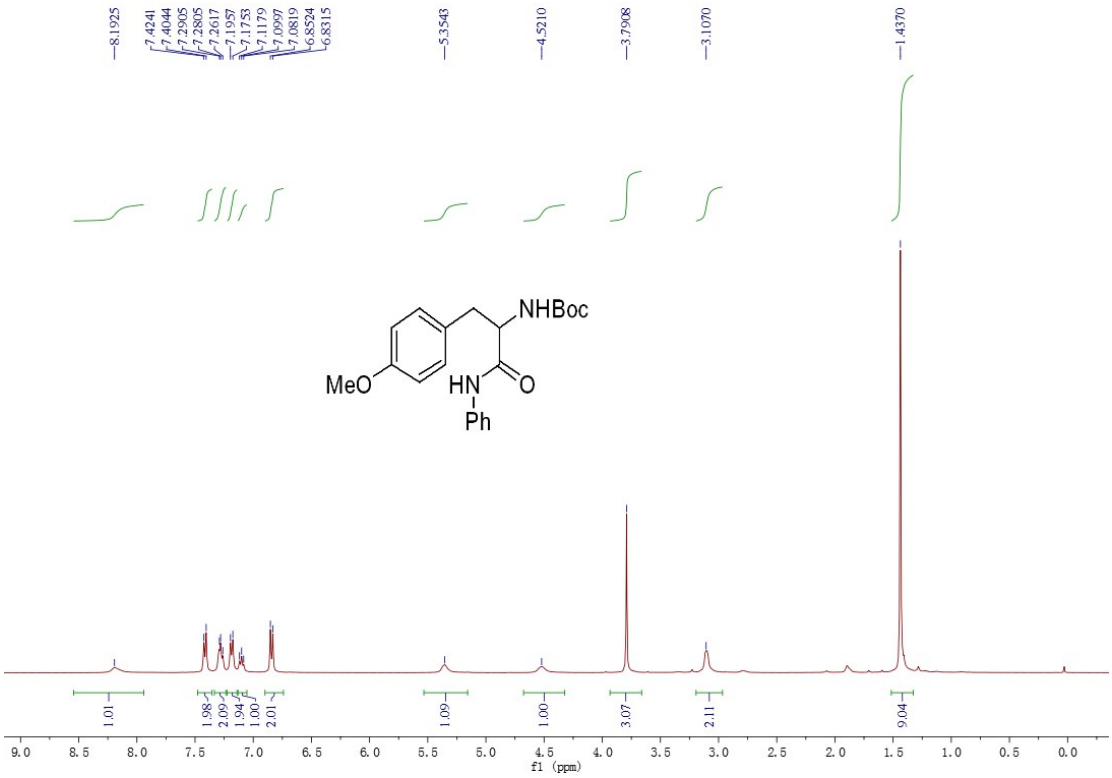
^{1}H NMR spectrum of compound **4h**



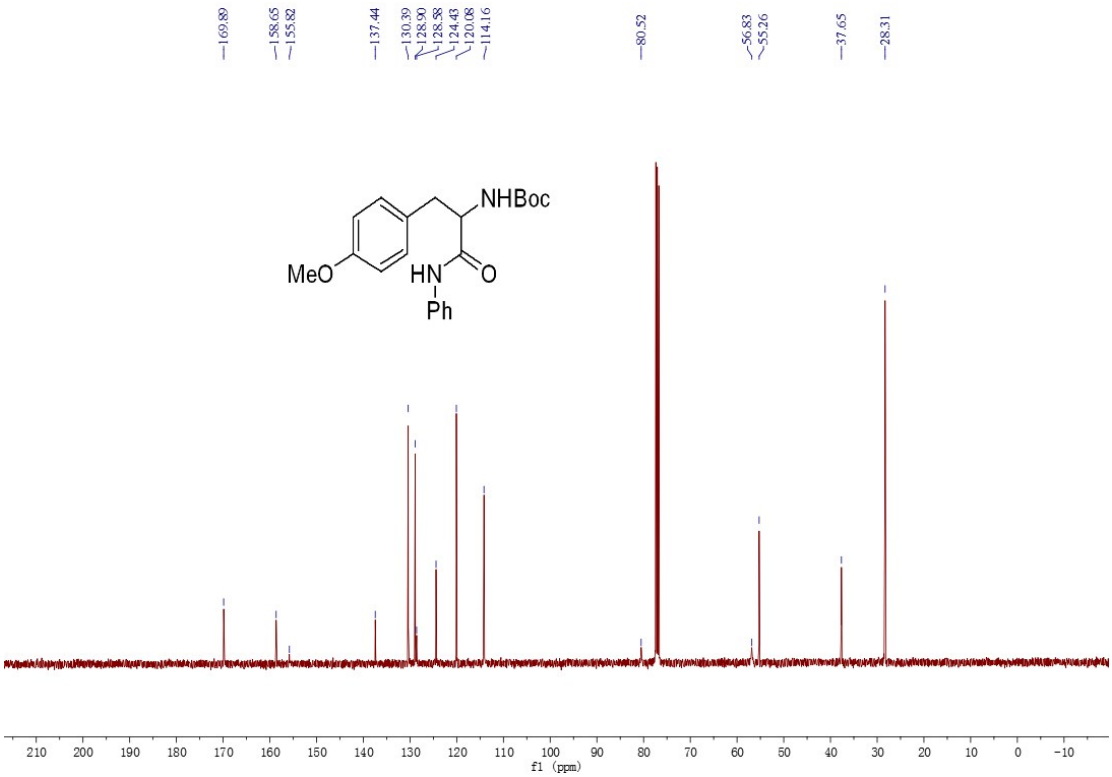
^{13}C NMR spectrum of compound **4h**



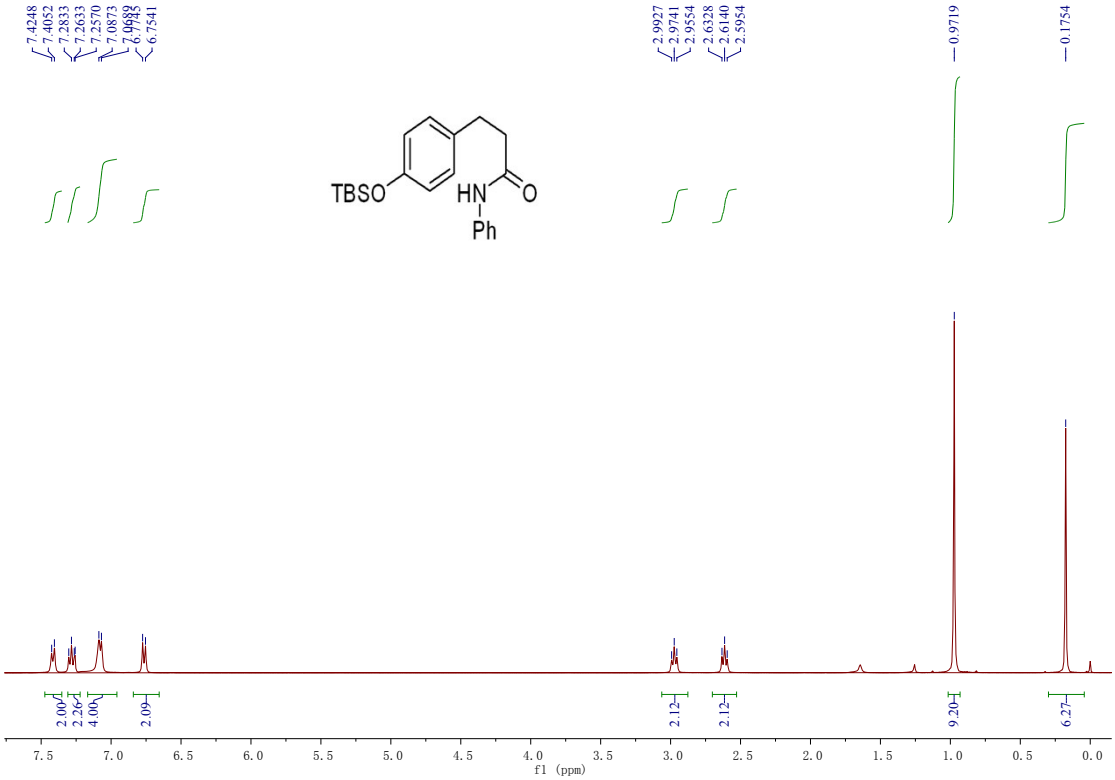
^{1}H NMR spectrum of compound **4i**



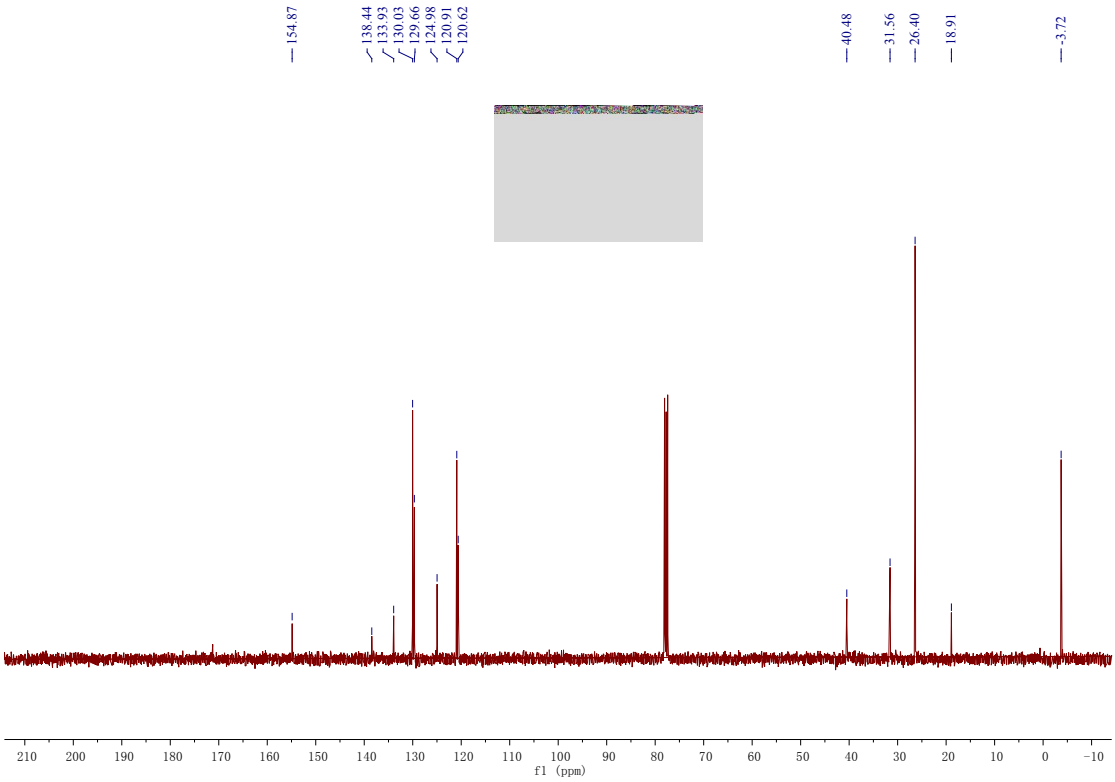
^{13}C NMR spectrum of compound **4i**



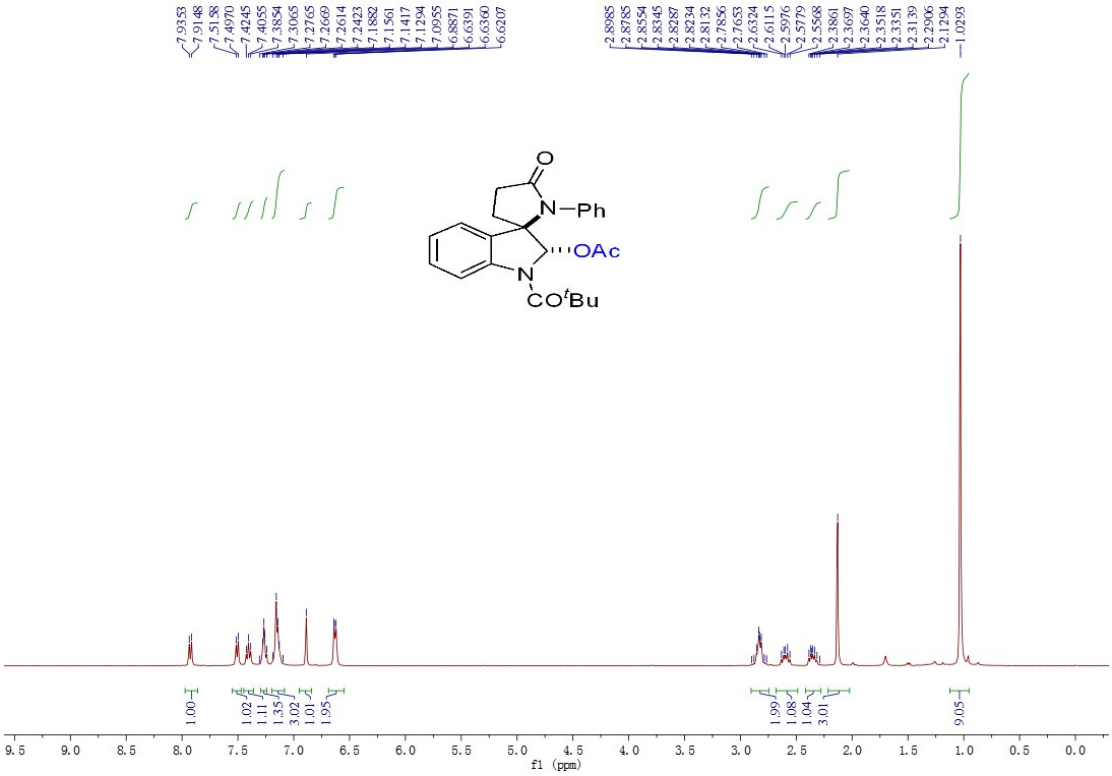
^{1}H NMR spectrum of compound **4j**



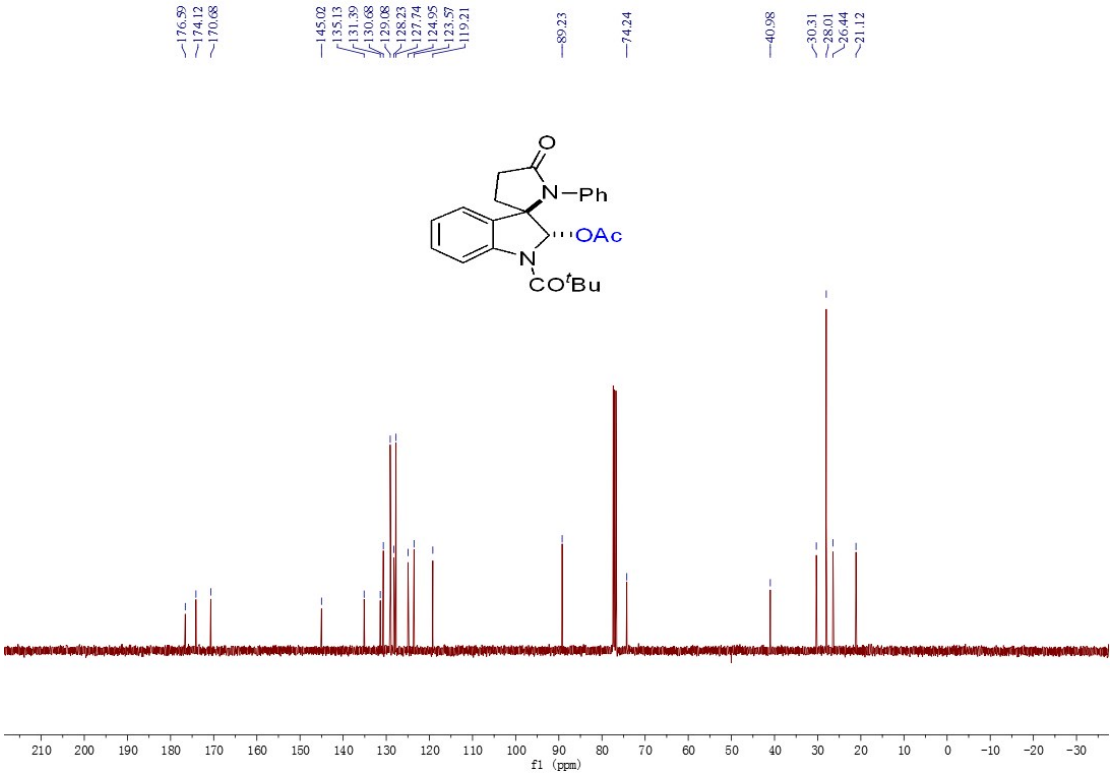
^{13}C NMR spectrum of compound **4j**



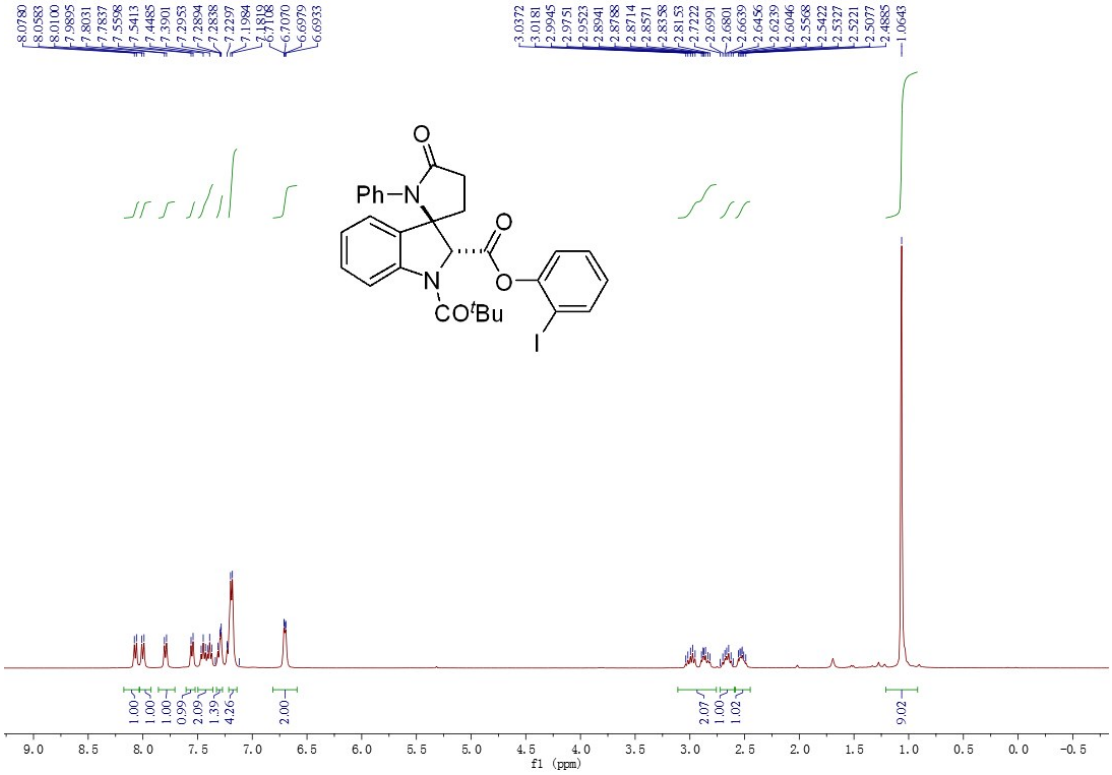
^{1}H NMR spectrum of compound **3a**



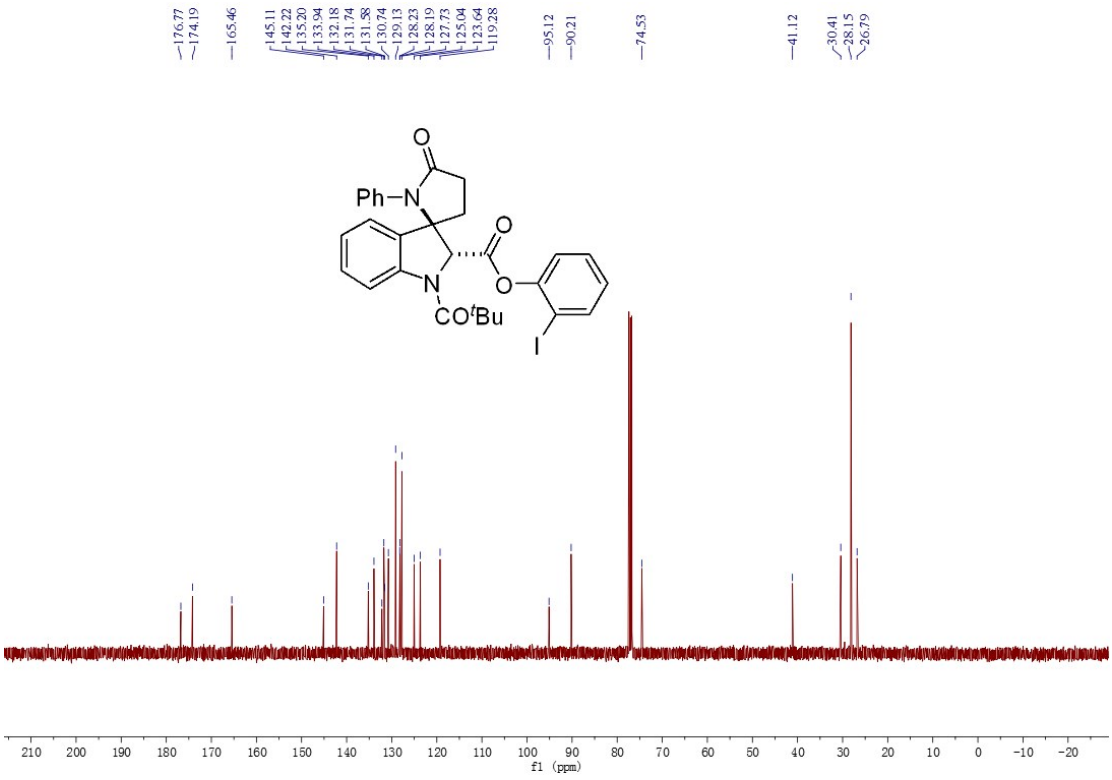
^{13}C NMR spectrum of compound **3a**



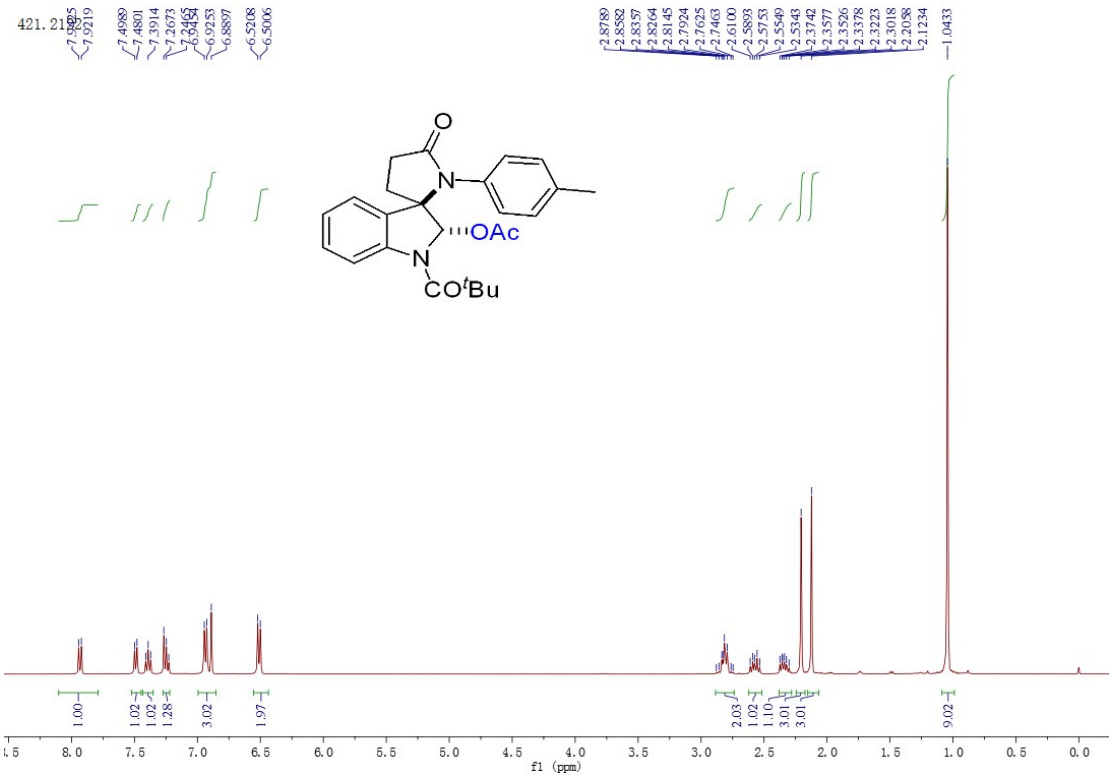
^{1}H NMR spectrum of compound **3a'**



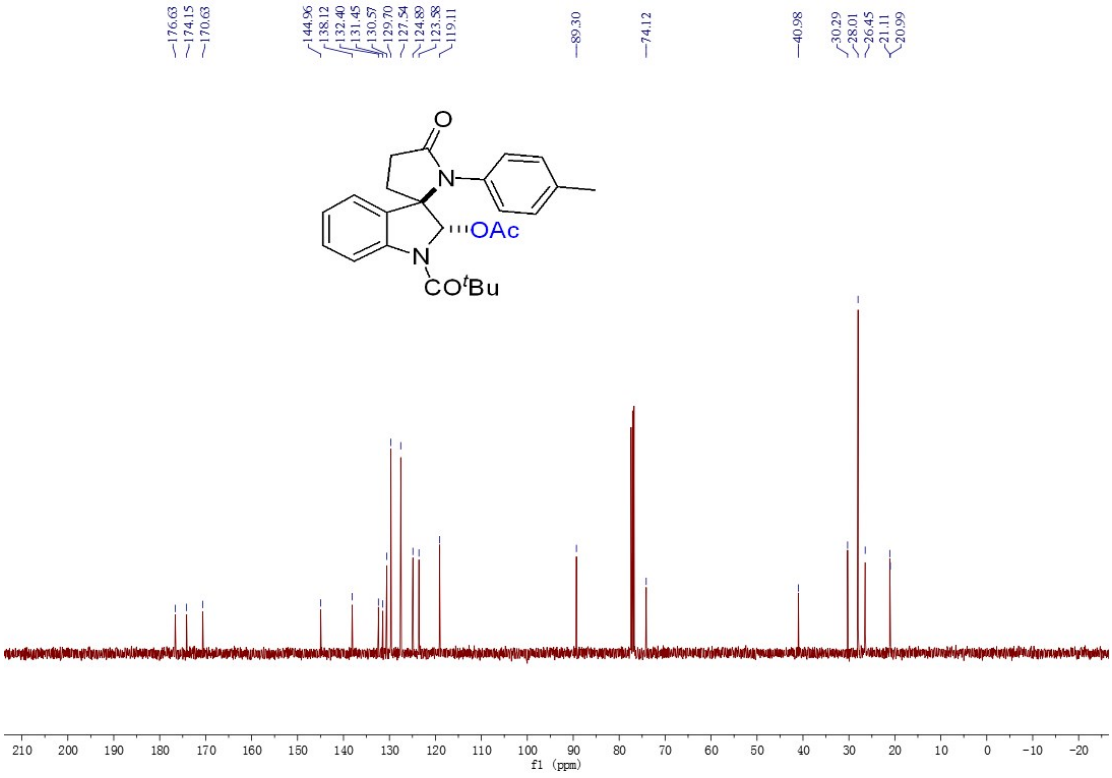
^{13}C NMR spectrum of compound **3a'**



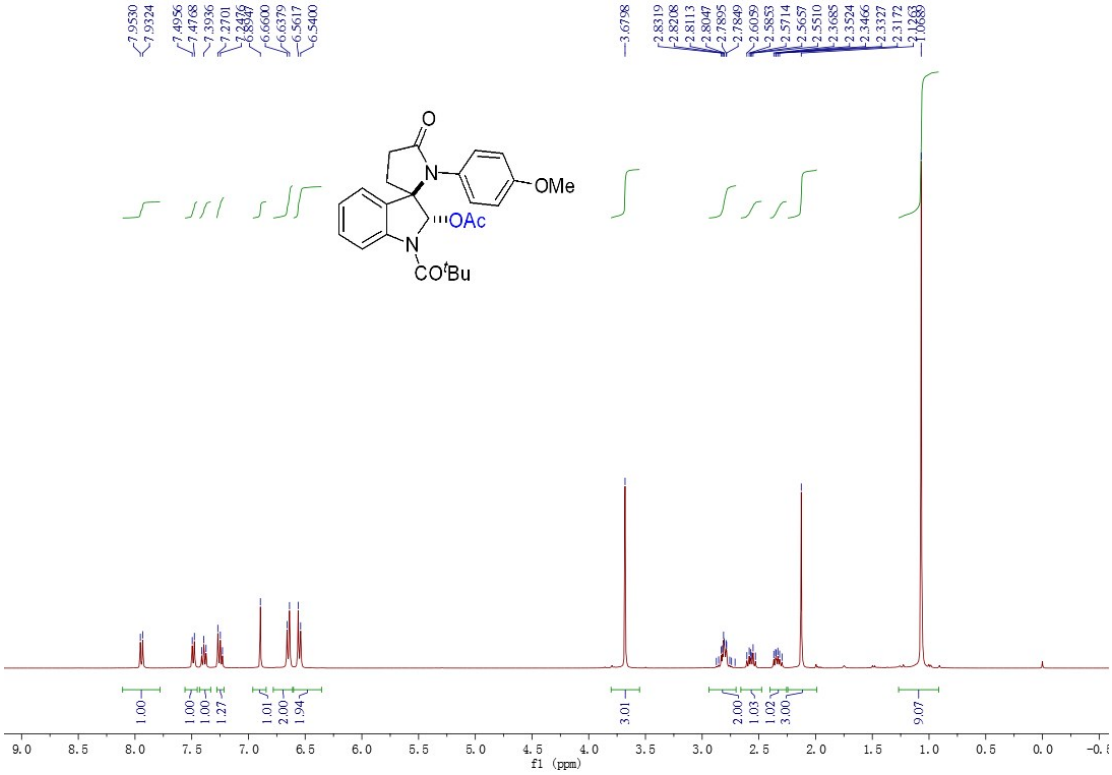
^{1}H NMR spectrum of compound **3b**



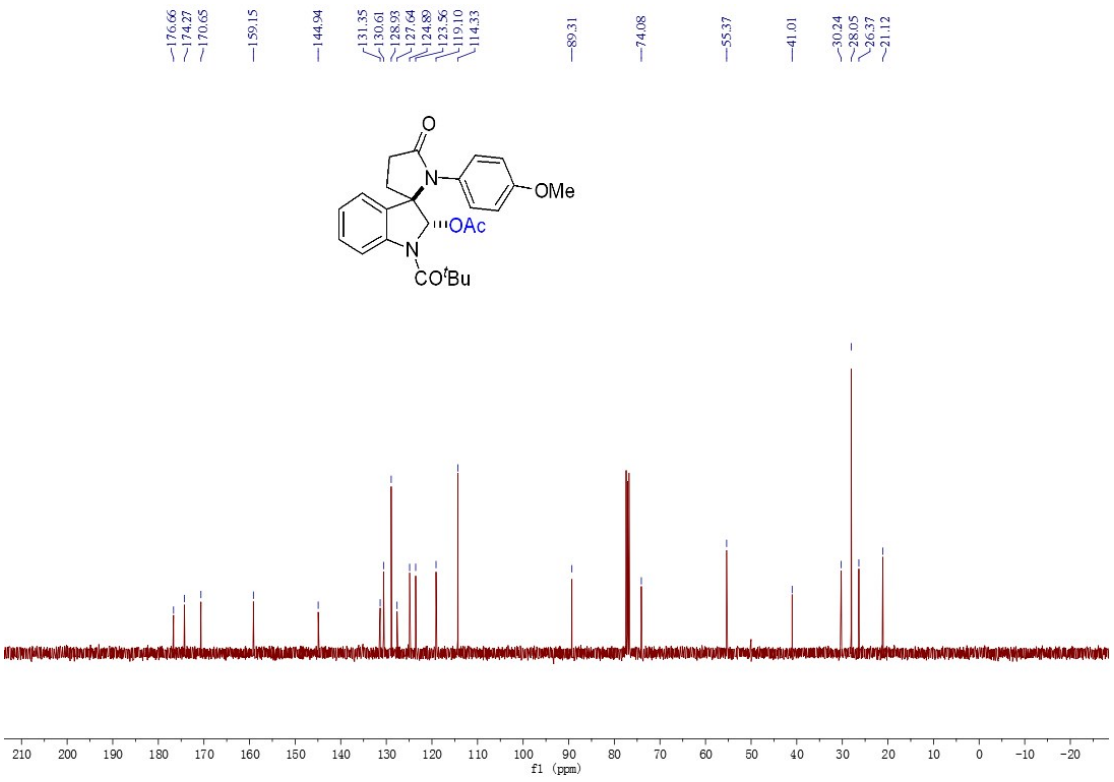
^{13}C NMR spectrum of compound **3b**



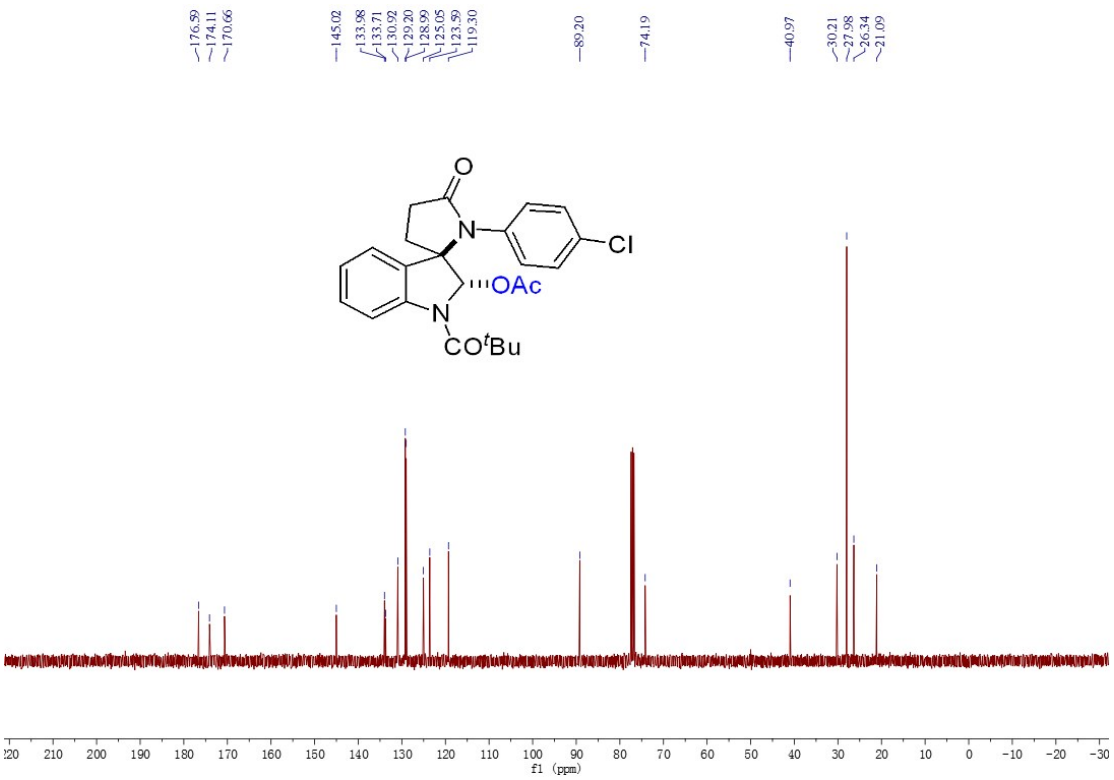
^{1}H NMR spectrum of compound **3c**



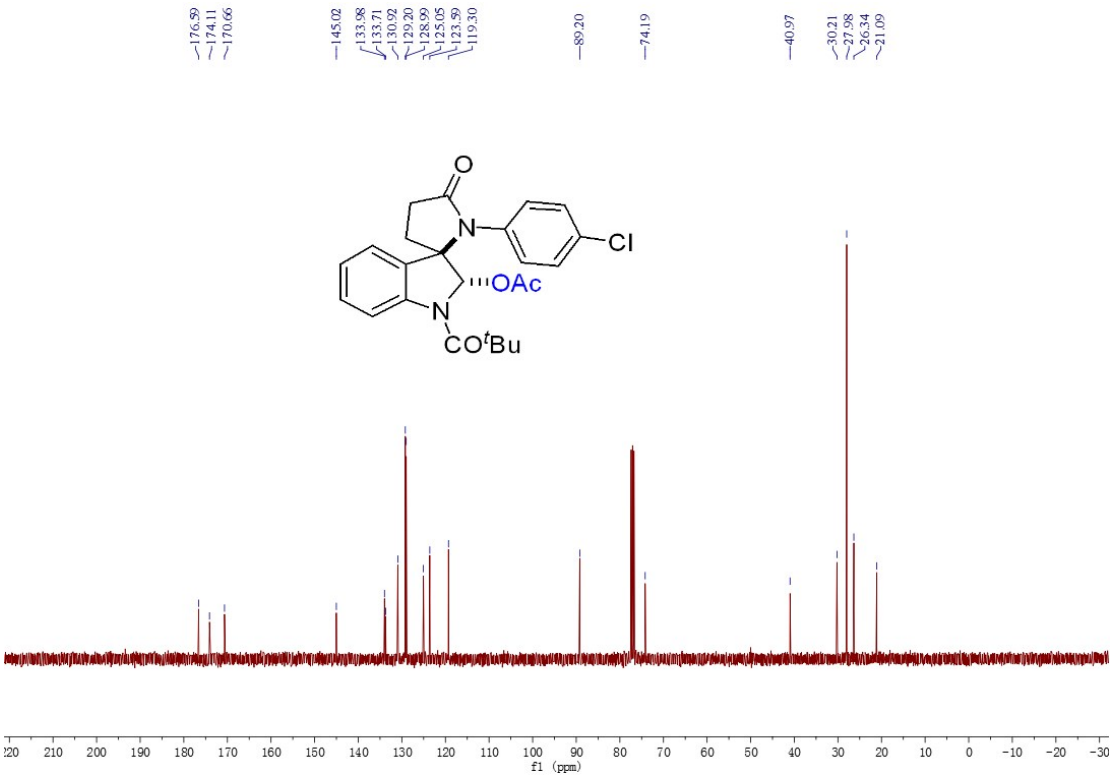
^{13}C NMR spectrum of compound **3c**



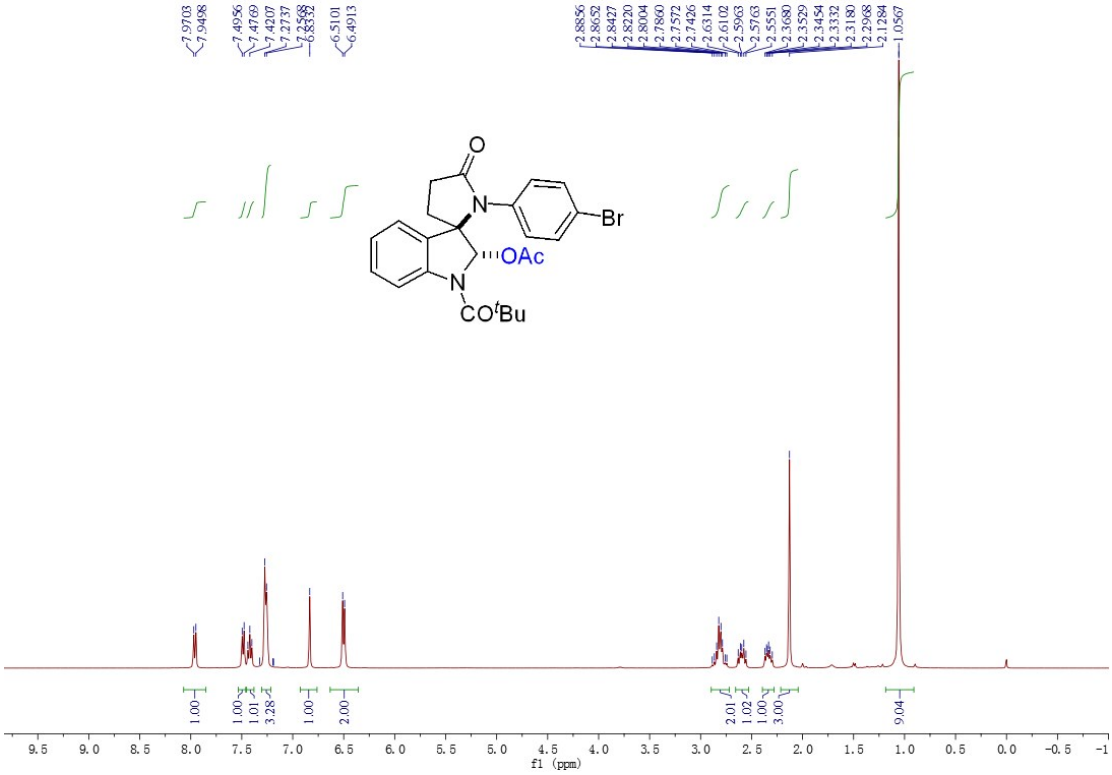
^{1}H NMR spectrum of compound **3d**



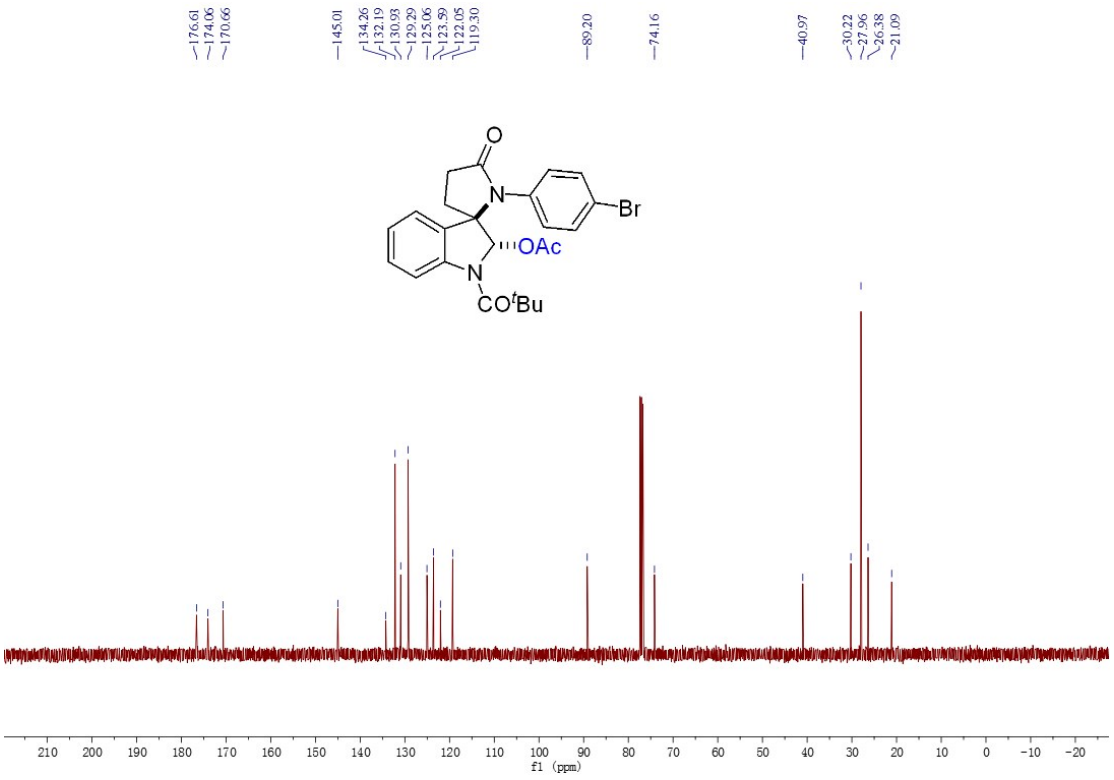
^{13}C NMR spectrum of compound **3d**



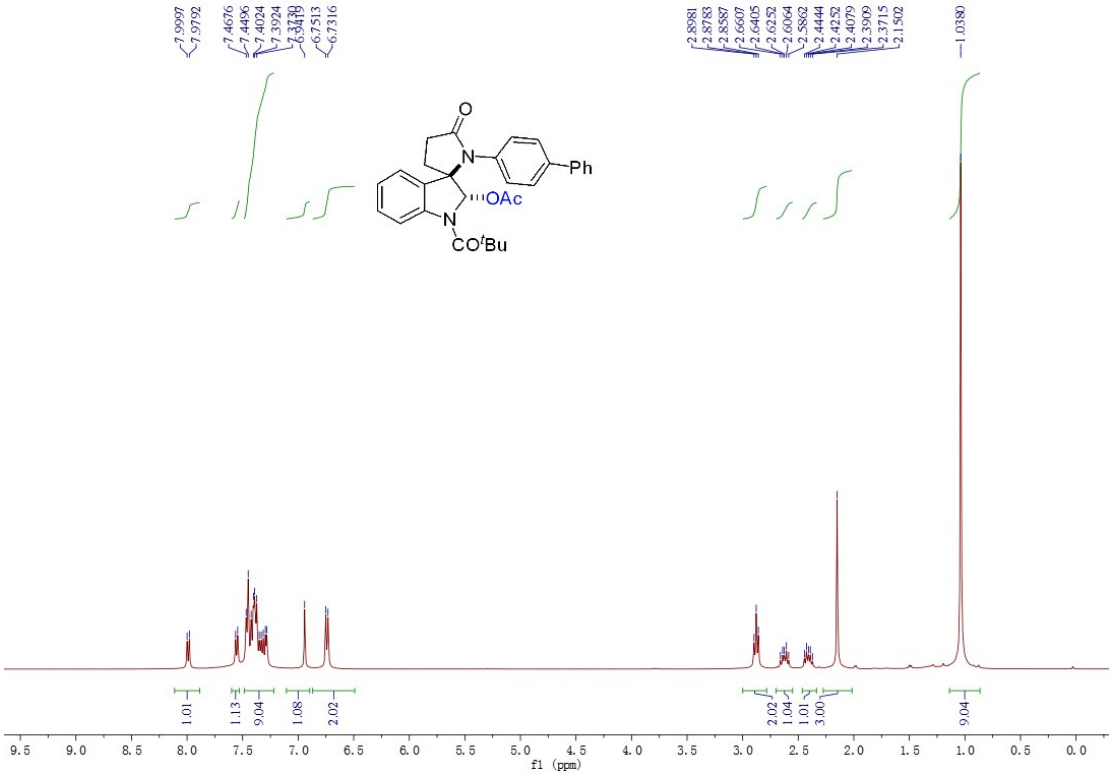
[1]H NMR spectrum of compound **3e**



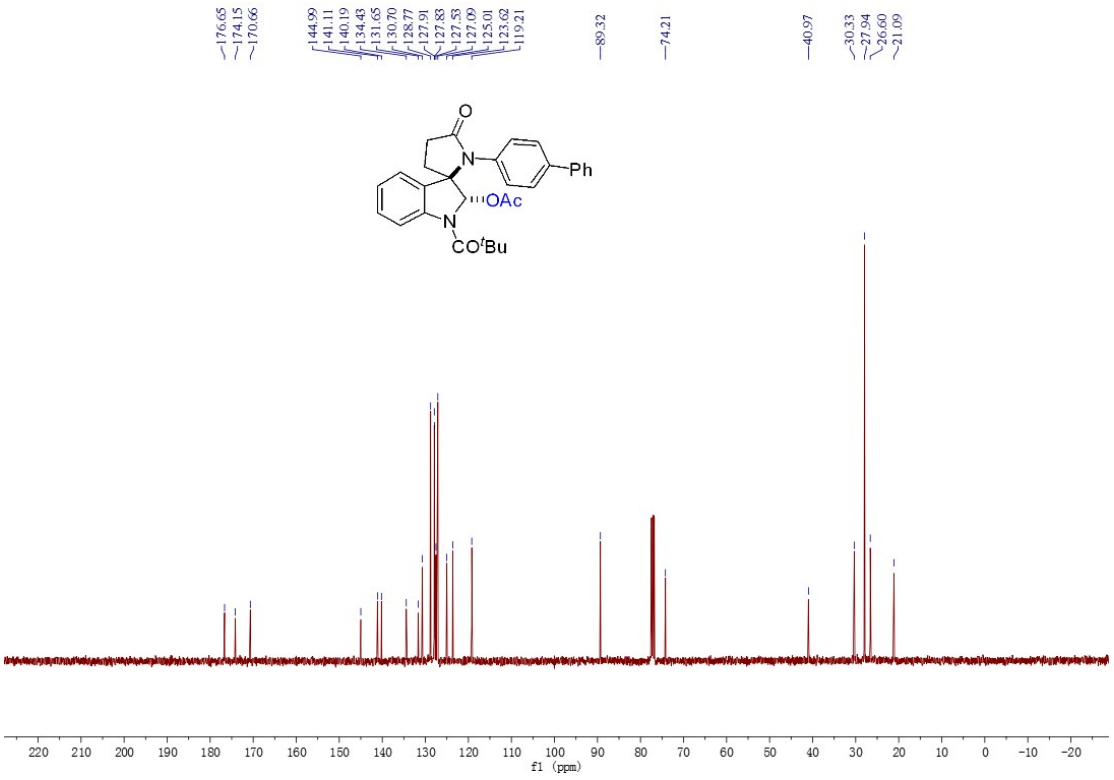
[13]C NMR spectrum of compound **3e**



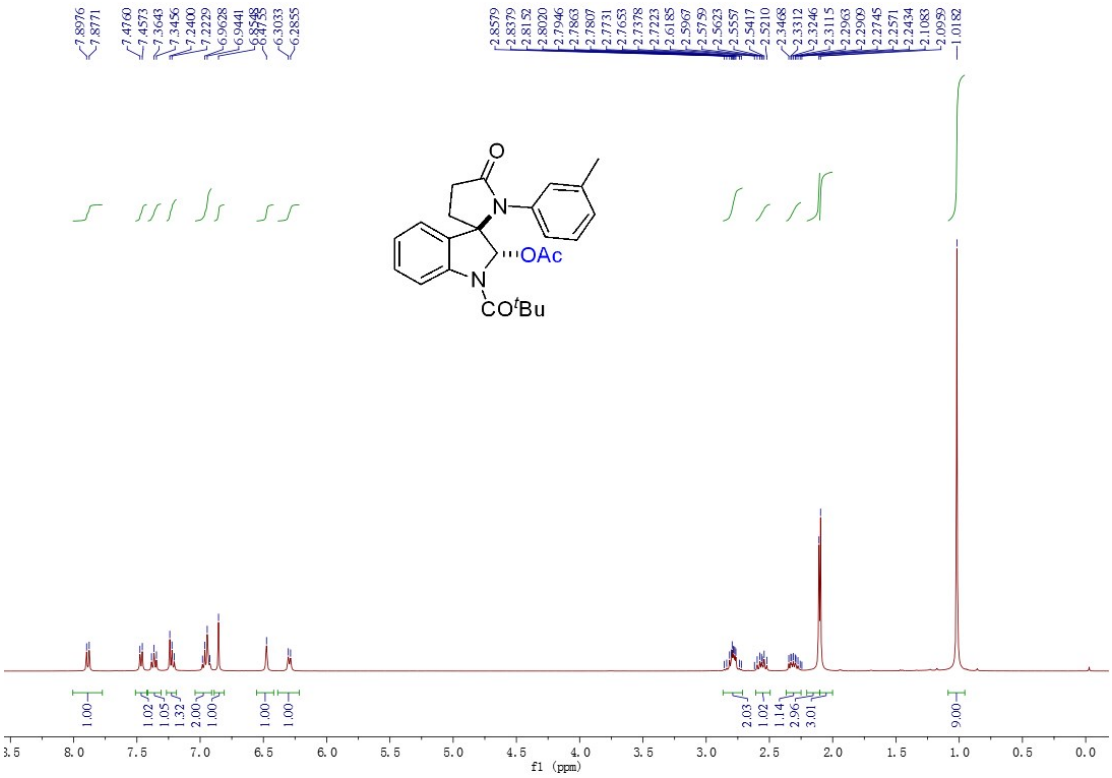
^{1}H NMR spectrum of compound **3f**



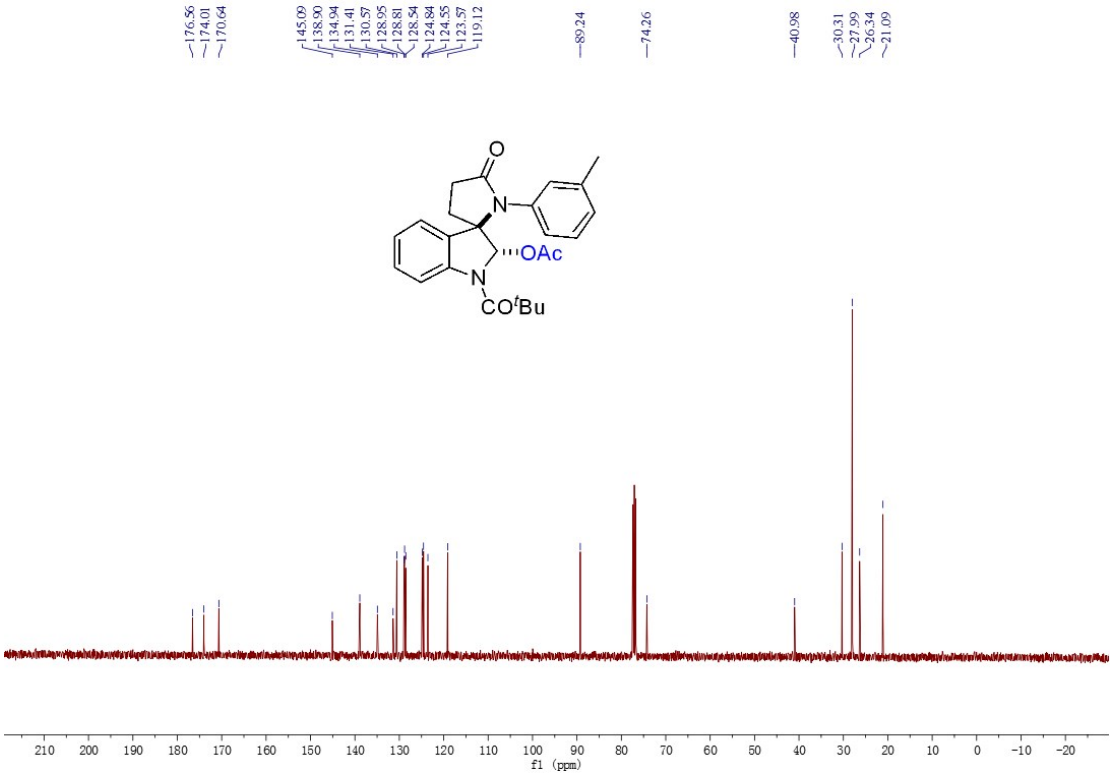
^{13}C NMR spectrum of compound **3f**



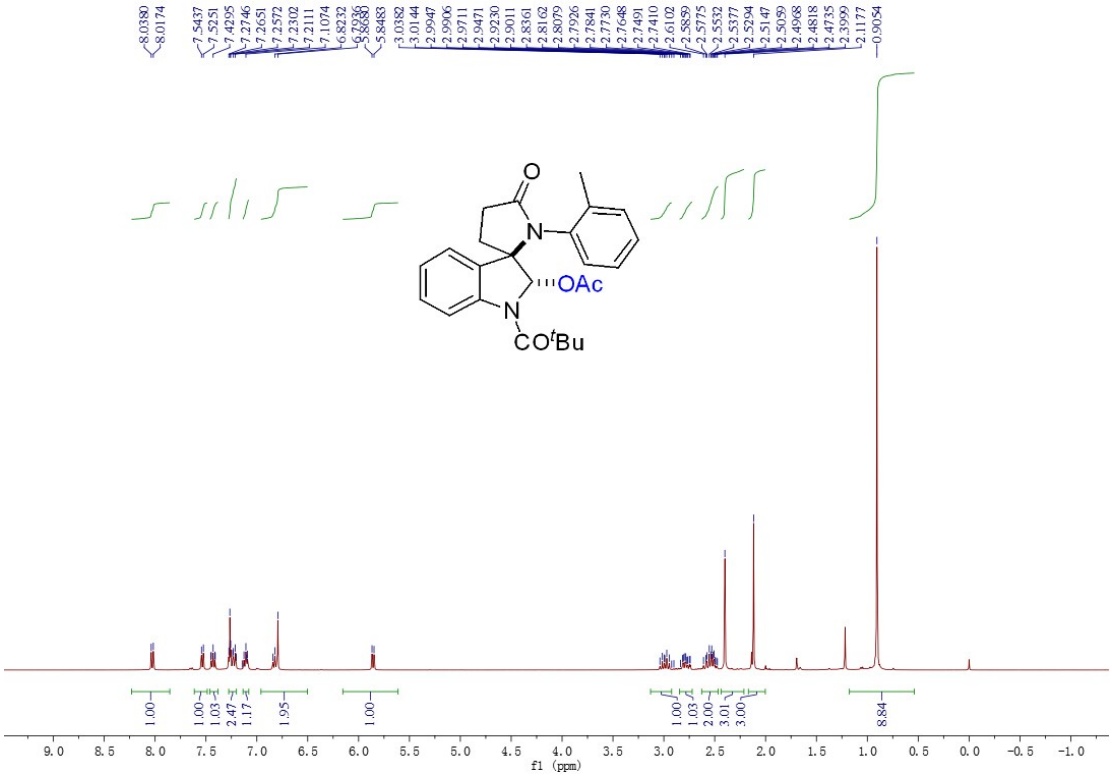
^{1}H NMR spectrum of compound **3g**



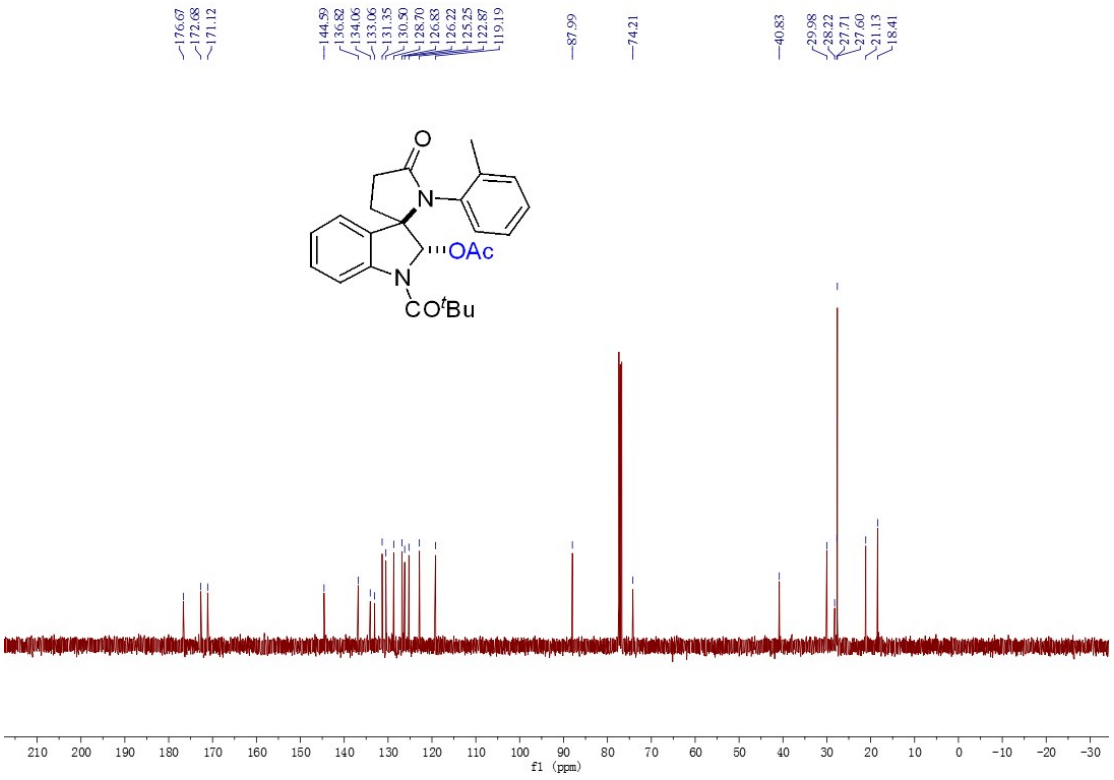
^{13}C NMR spectrum of compound **3g**



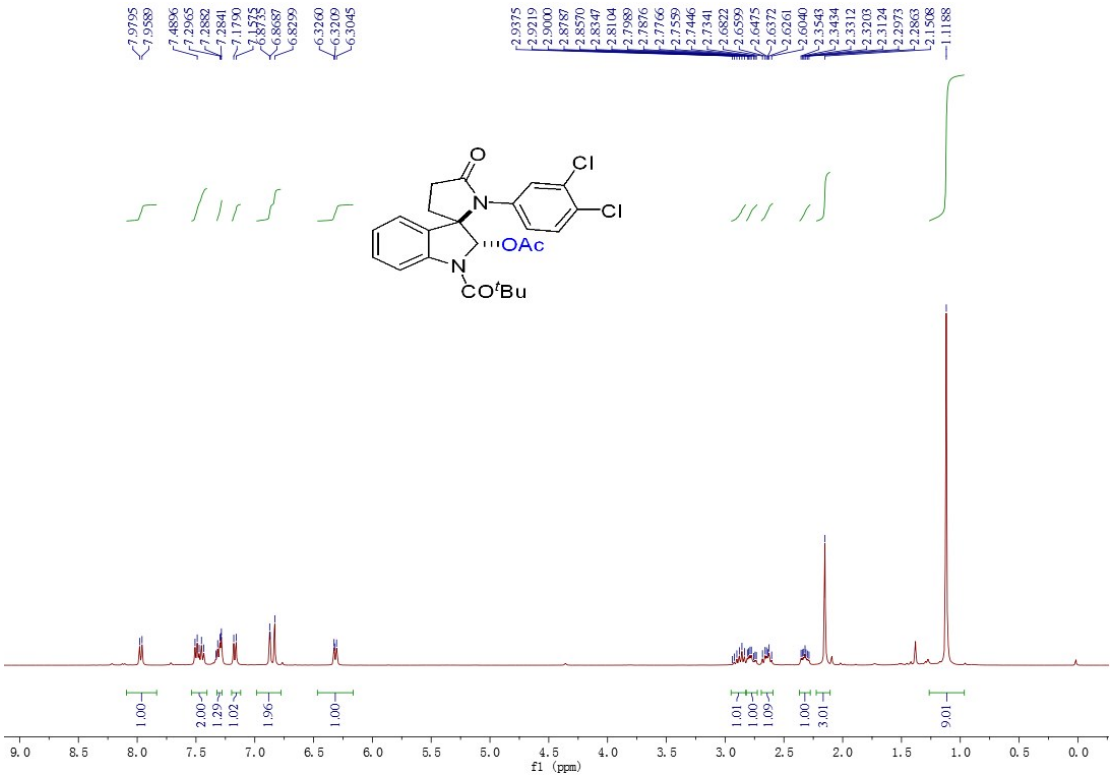
^{1}H NMR spectrum of compound **3h**



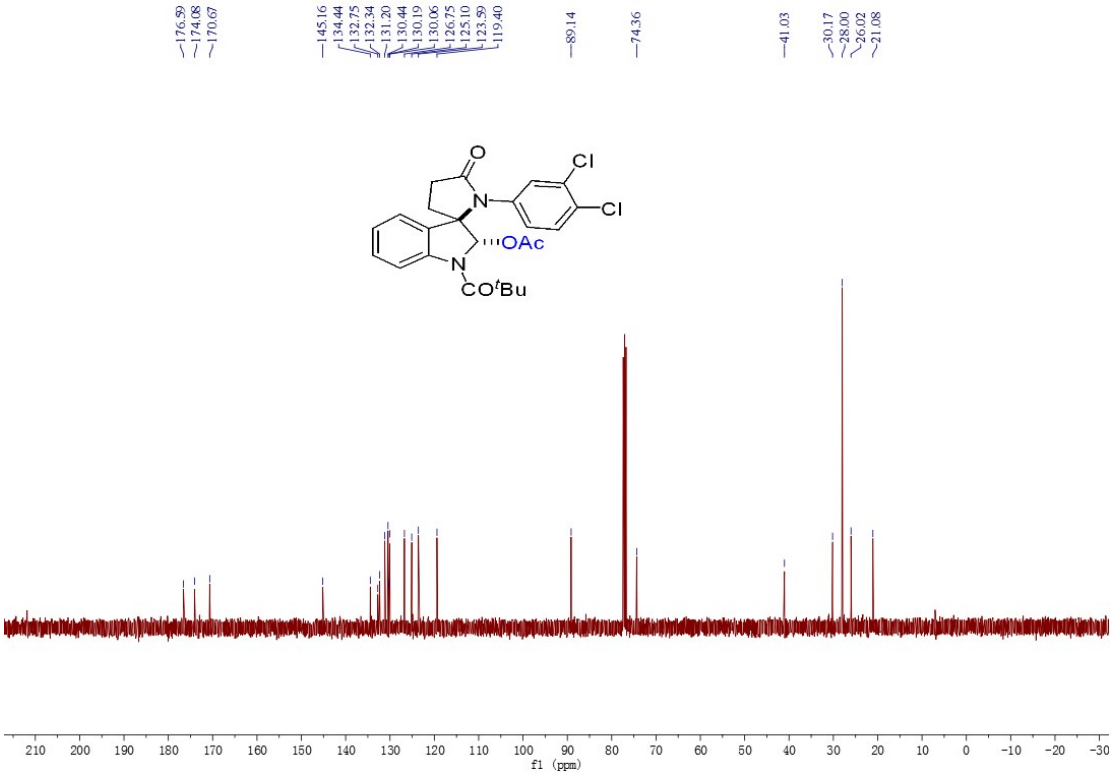
^{13}C NMR spectrum of compound **3h**



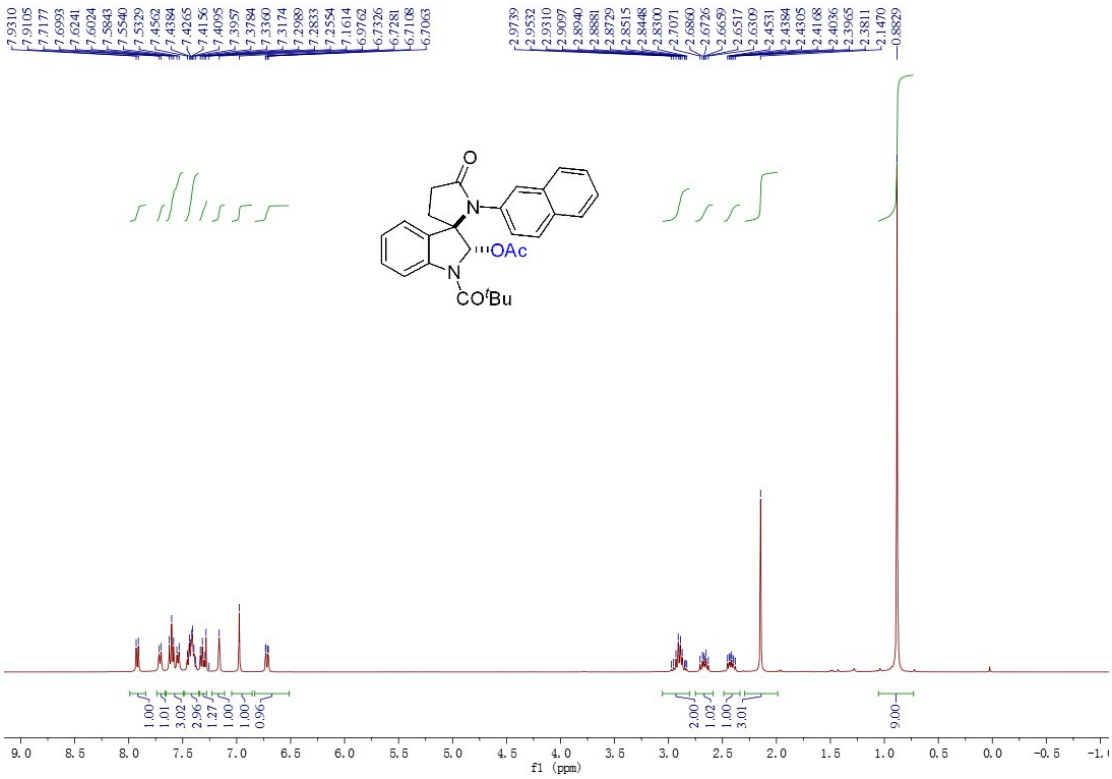
^{1}H NMR spectrum of compound **3i**



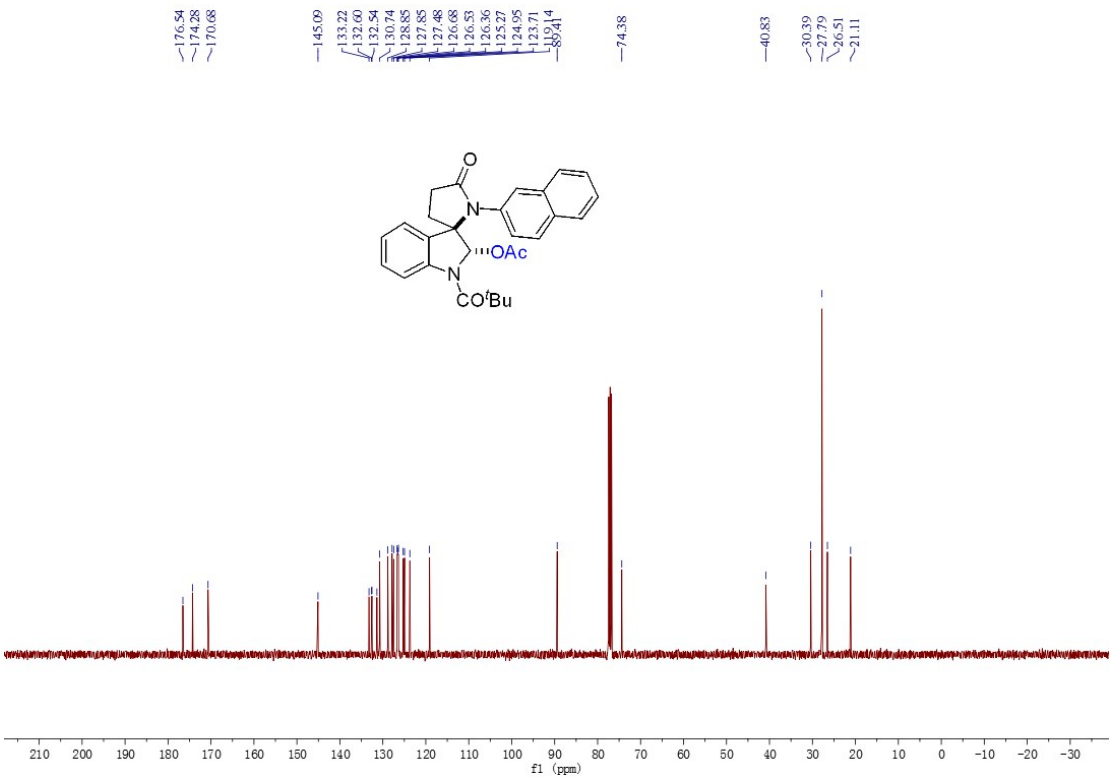
^{13}C NMR spectrum of compound **3i**



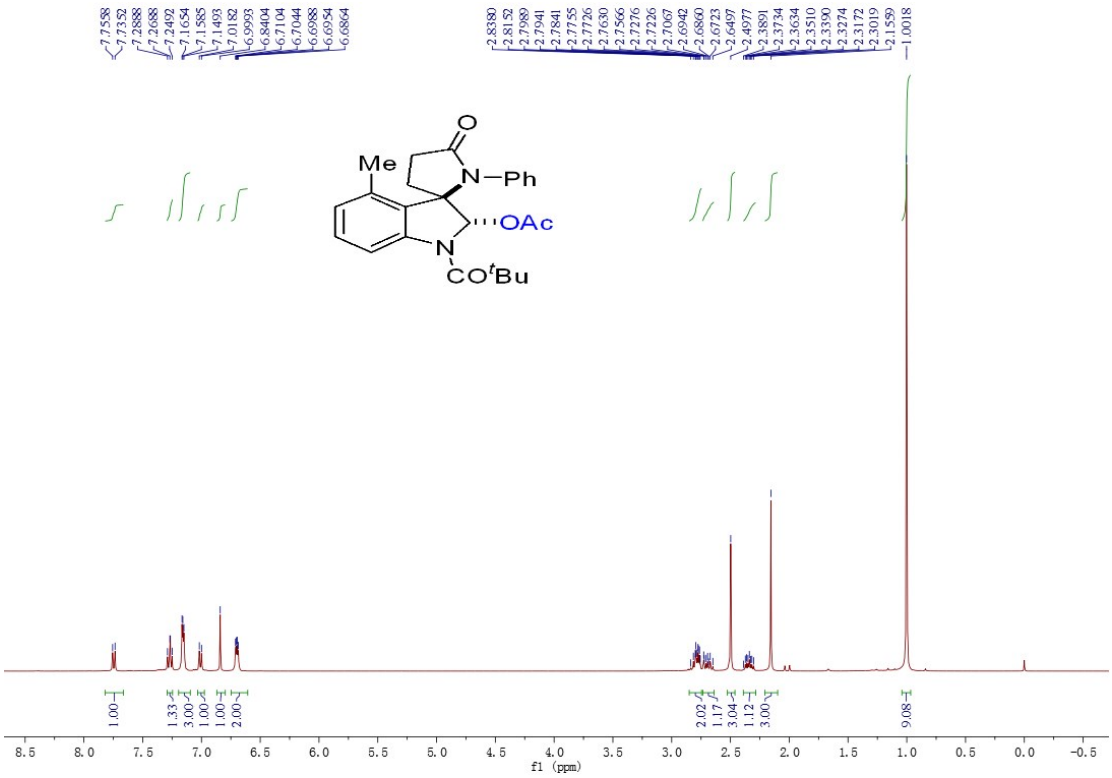
^{1}H NMR spectrum of compound **3j**



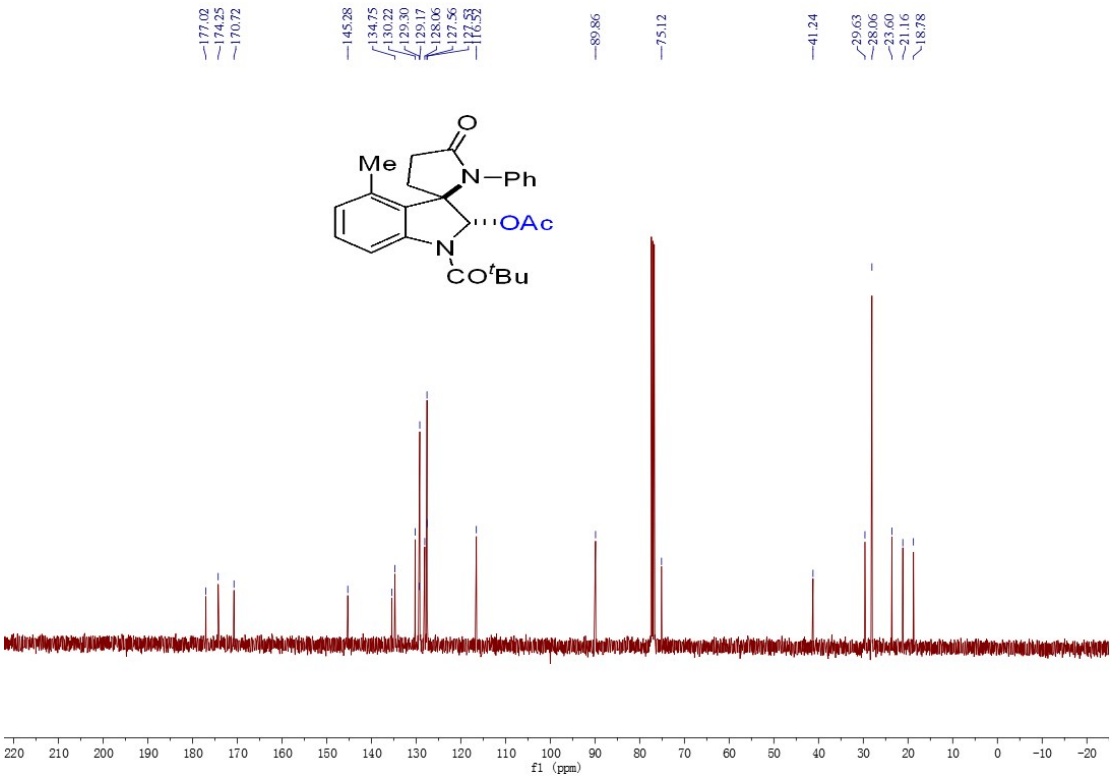
^{13}C NMR spectrum of compound **3j**



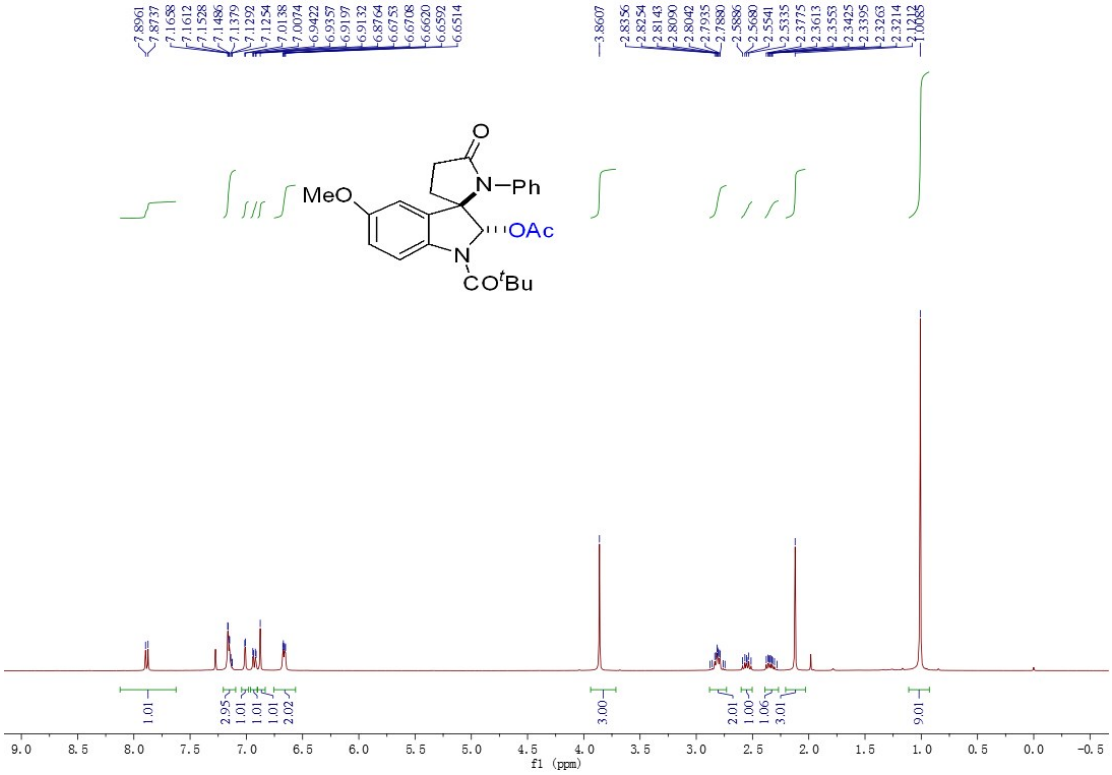
^{1}H NMR spectrum of compound **3k**



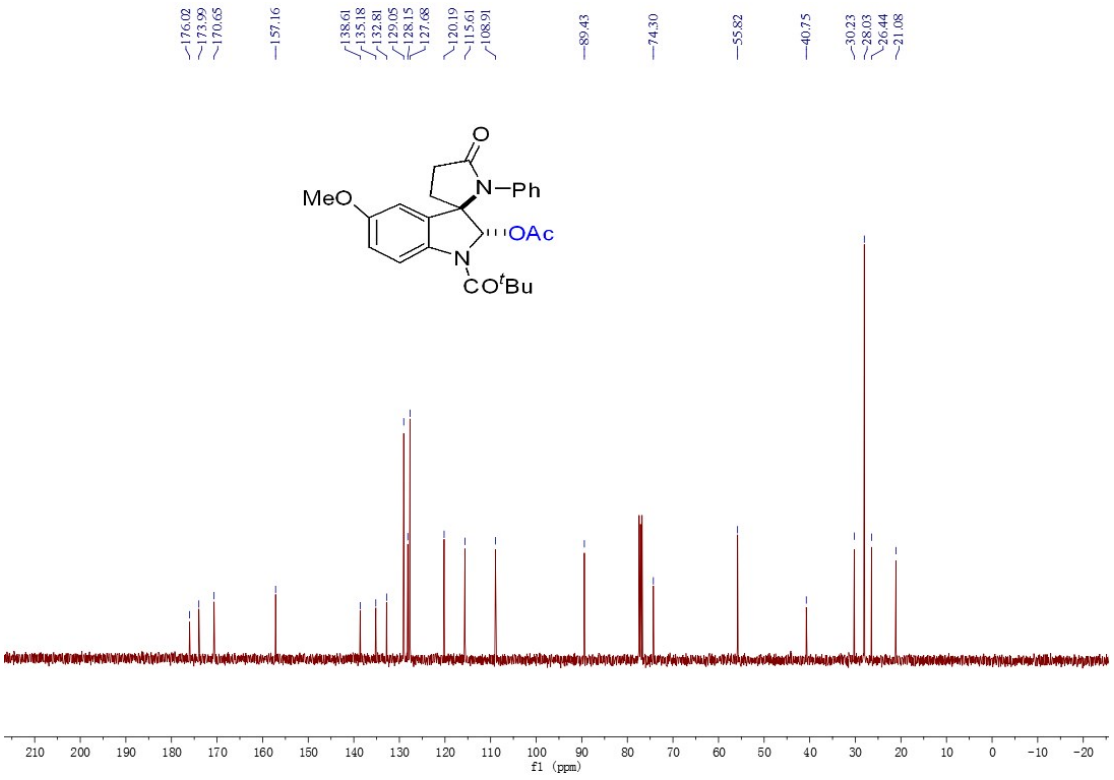
^{13}C NMR spectrum of compound **3k**



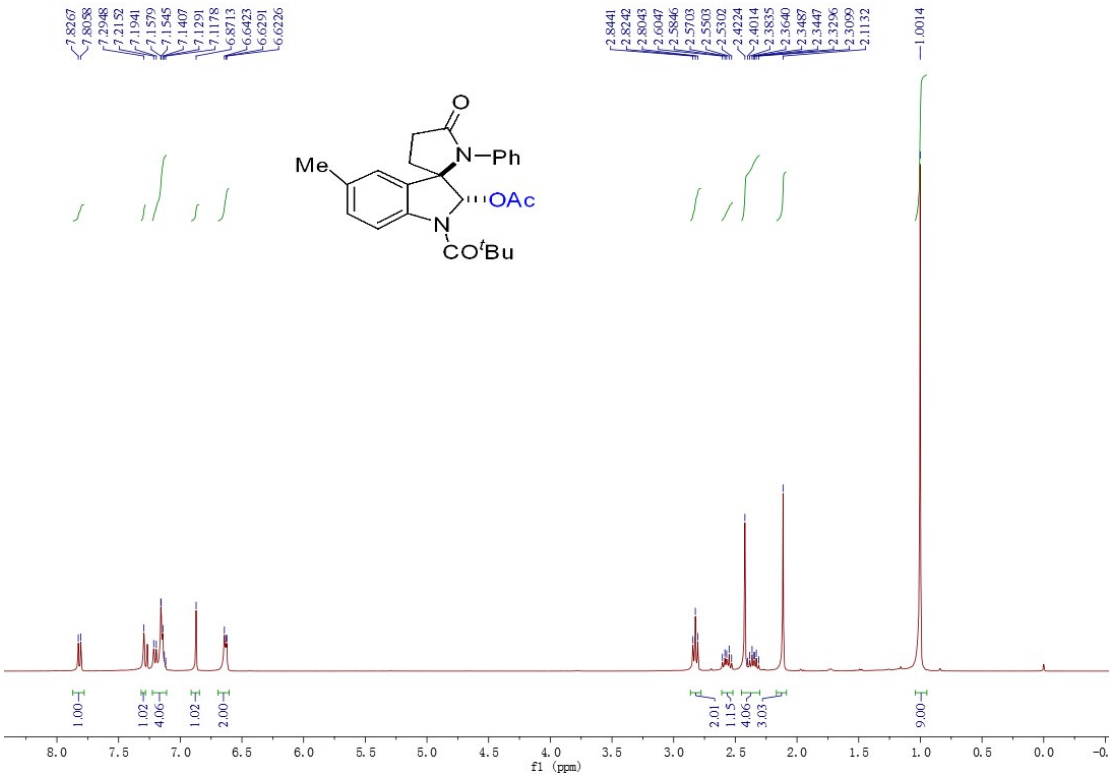
^{1}H NMR spectrum of compound **3l**



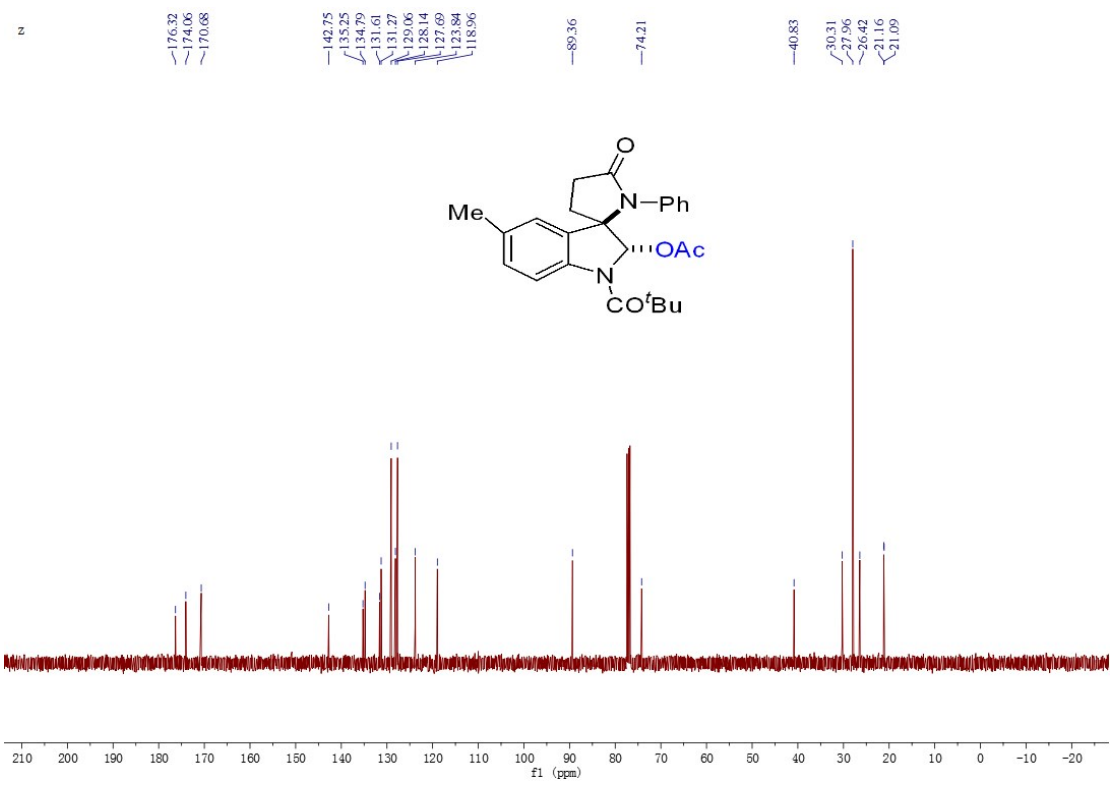
^{13}C NMR spectrum of compound **3l**



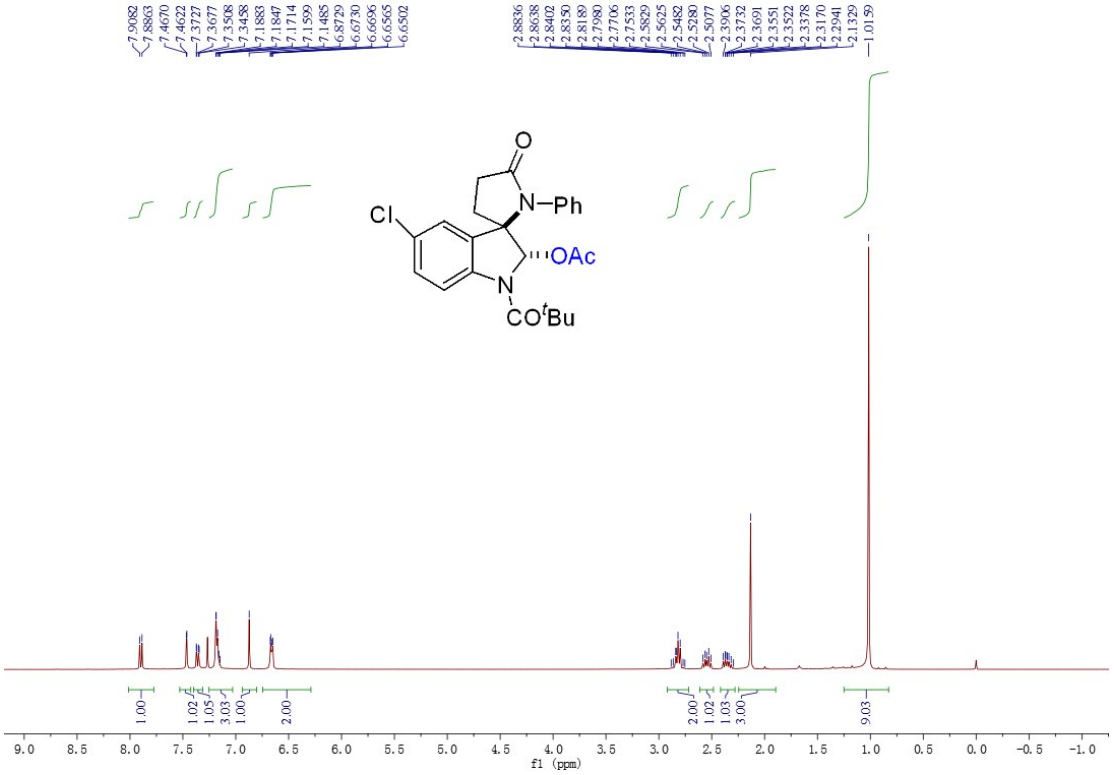
^{1}H NMR spectrum of compound **3m**



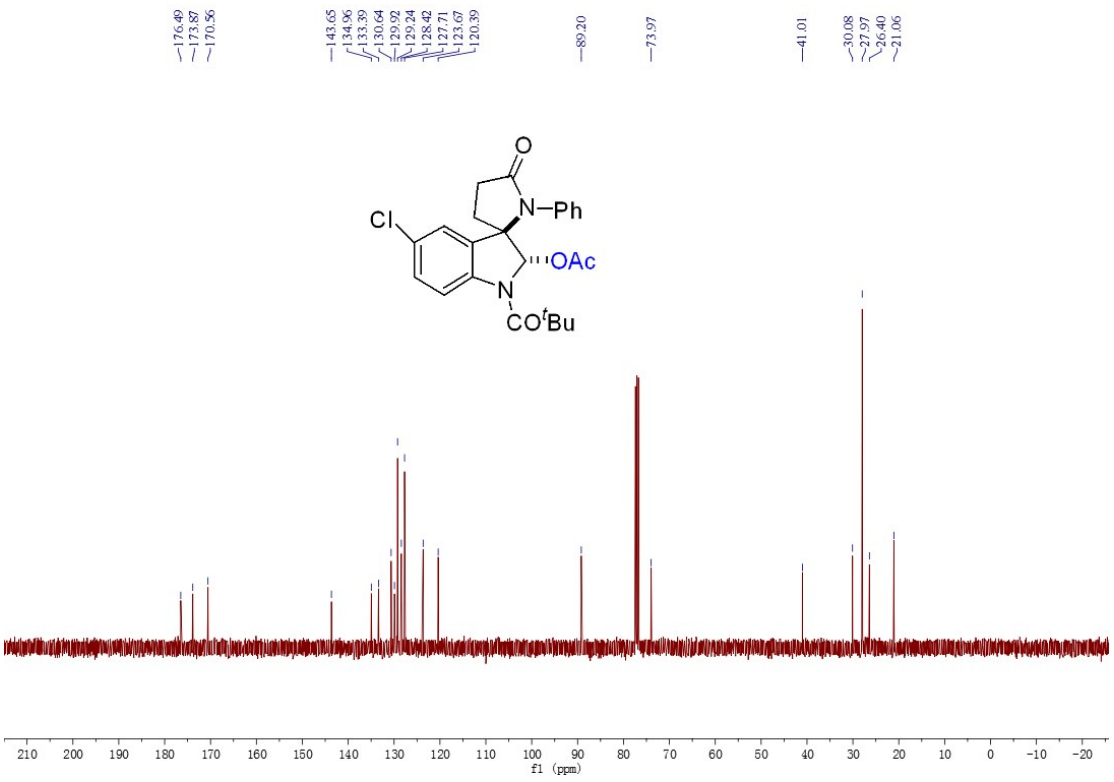
^{13}C NMR spectrum of compound **3m**



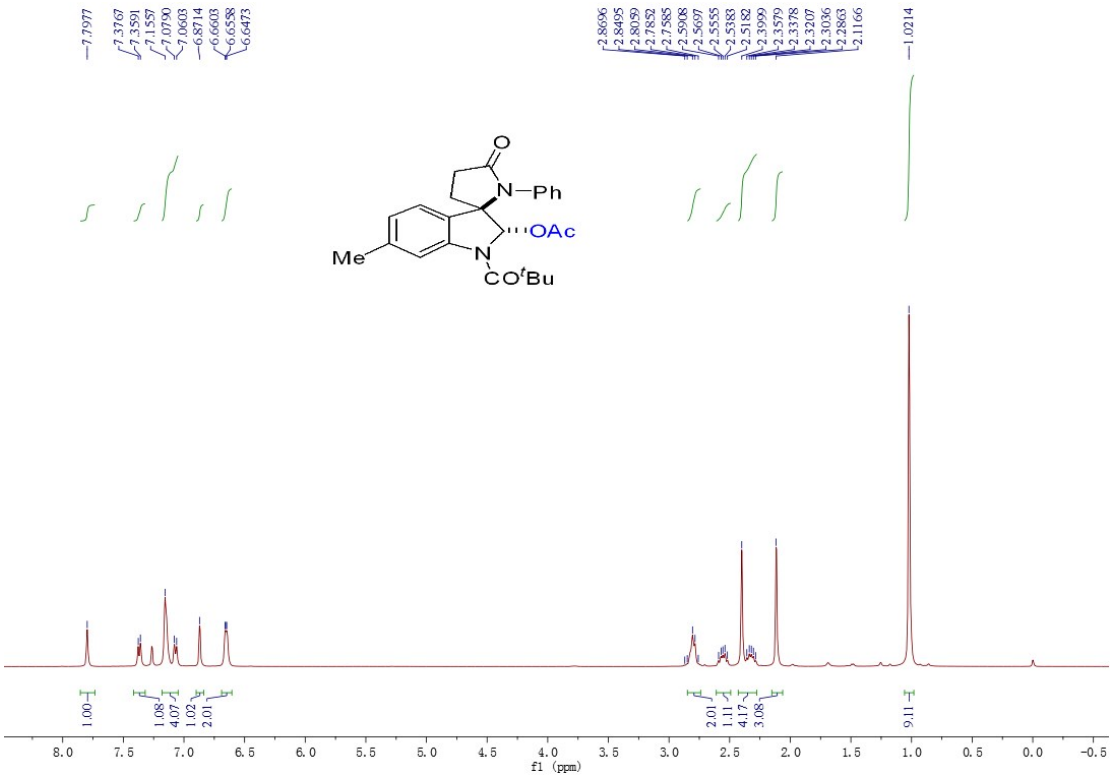
^{1}H NMR spectrum of compound **3n**



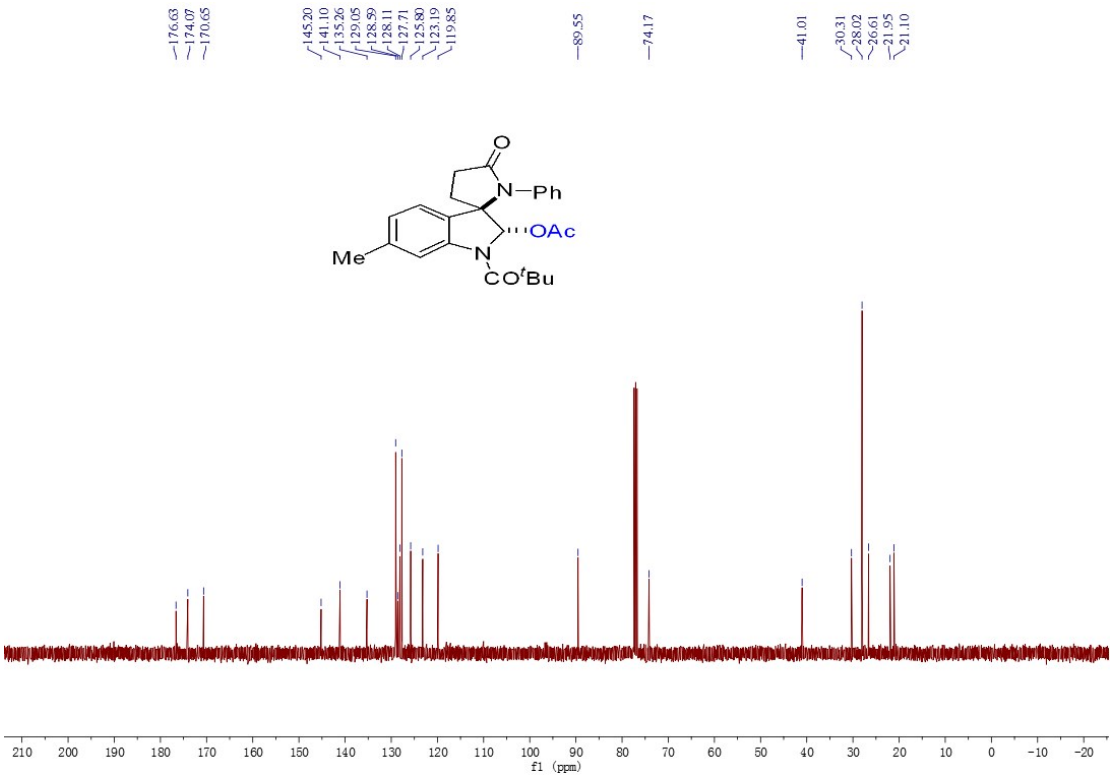
^{13}C NMR spectrum of compound **3n**



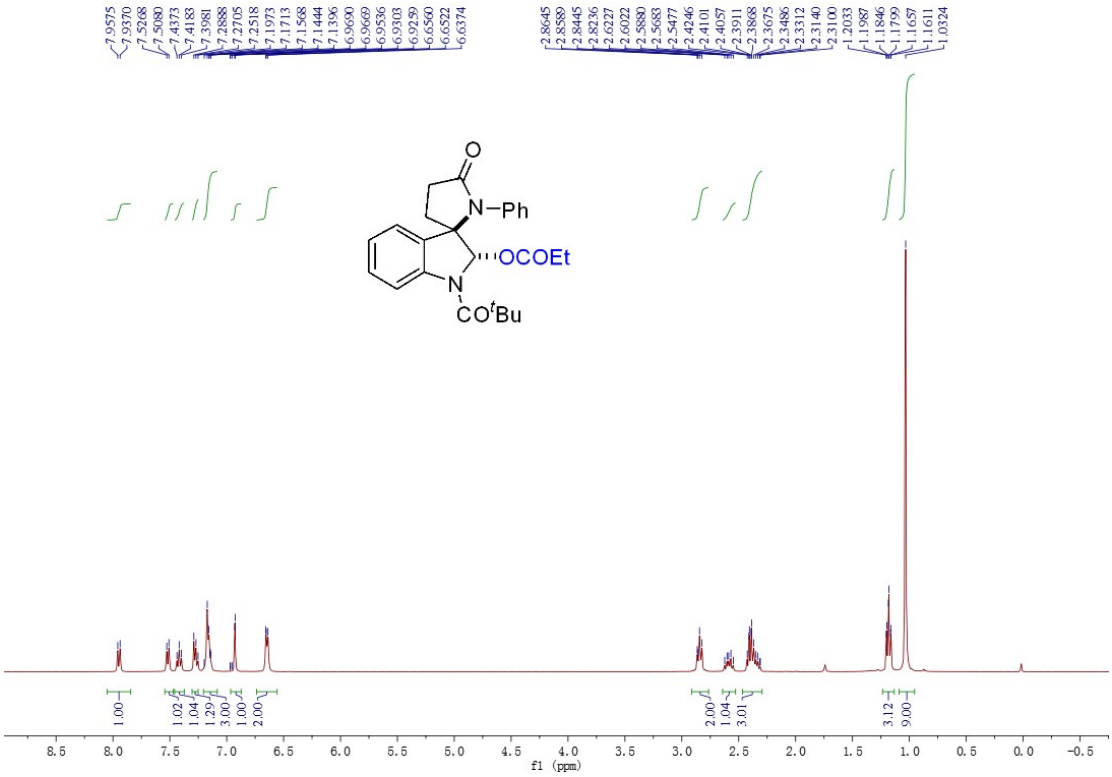
^{1}H NMR spectrum of compound **3o**



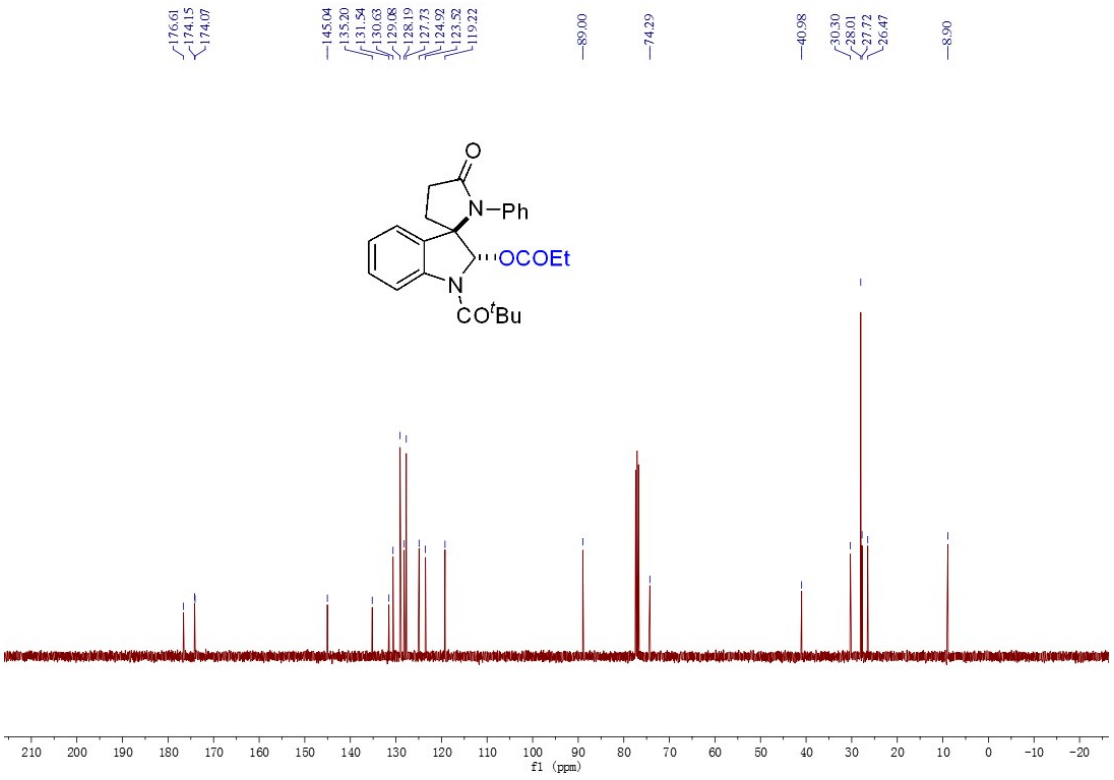
^{13}C NMR spectrum of compound **3o**



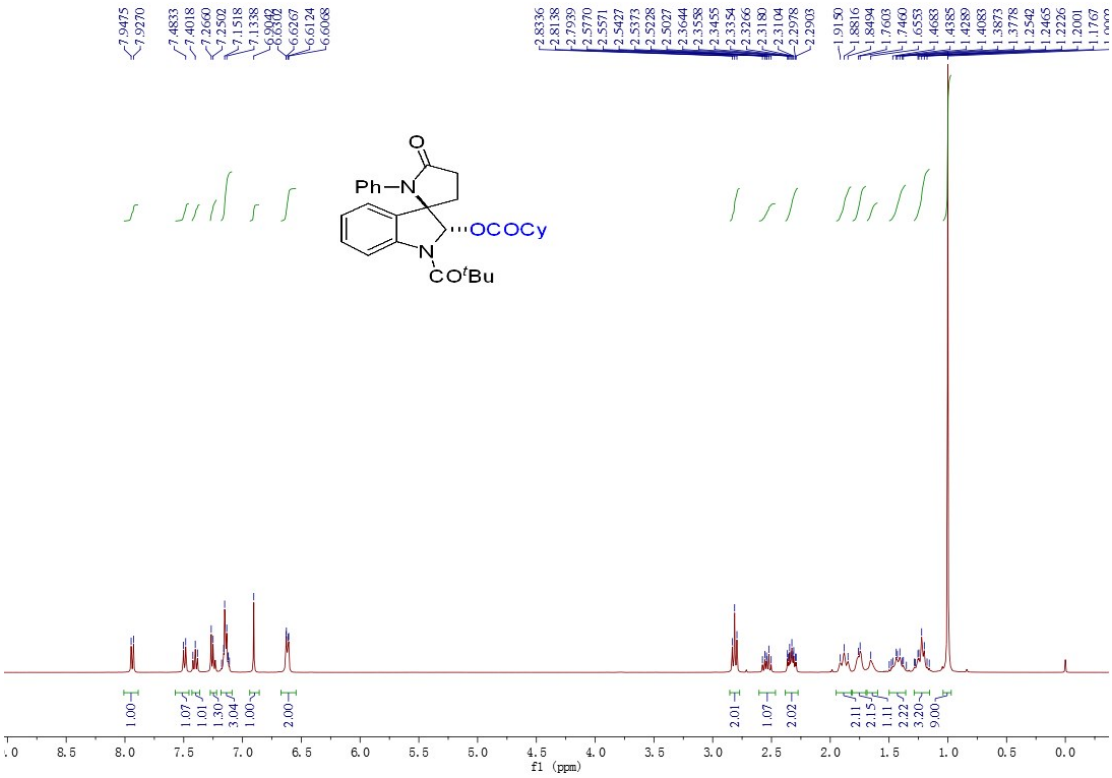
^{1}H NMR spectrum of compound **3p**



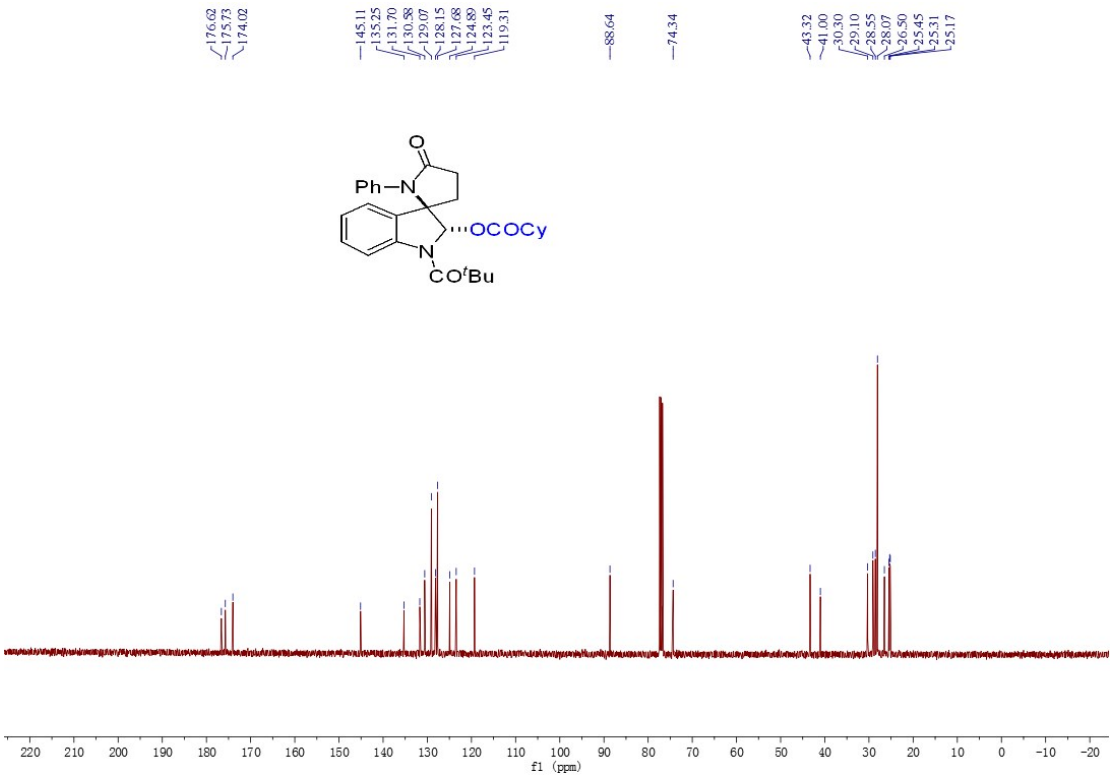
^{13}C NMR spectrum of compound **3p**



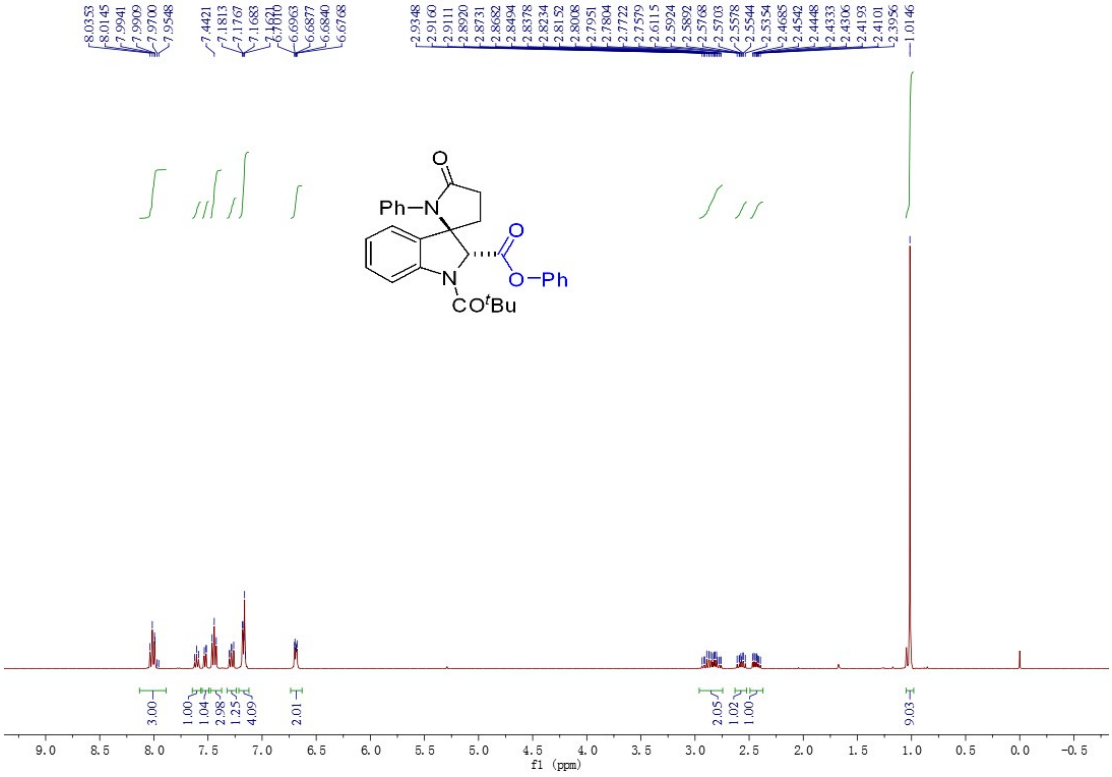
^{1}H NMR spectrum of compound **3q**



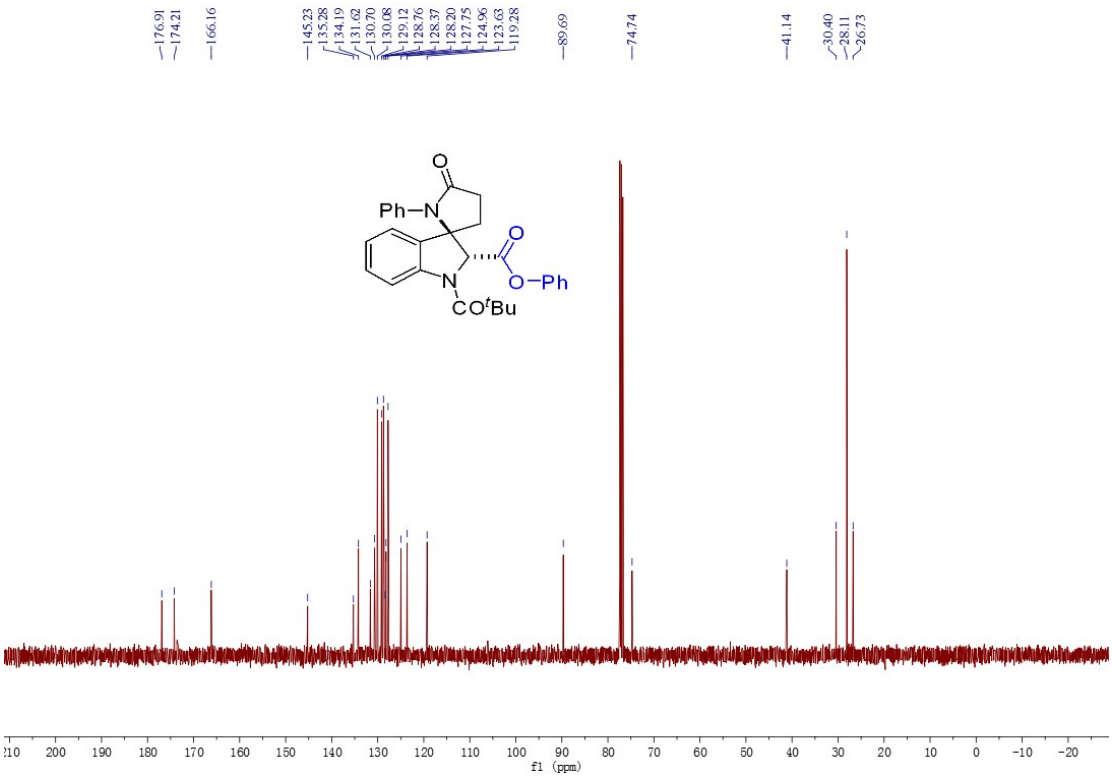
^{13}C NMR spectrum of compound **3q**



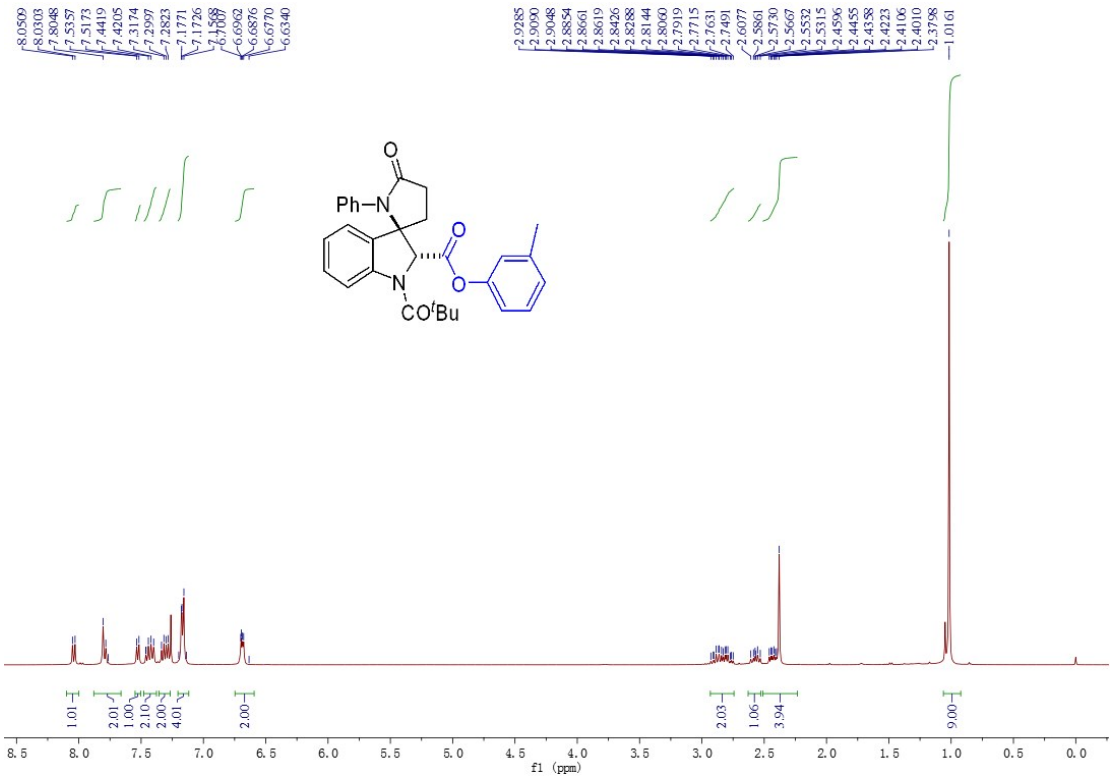
^{1}H NMR spectrum of compound **3r**



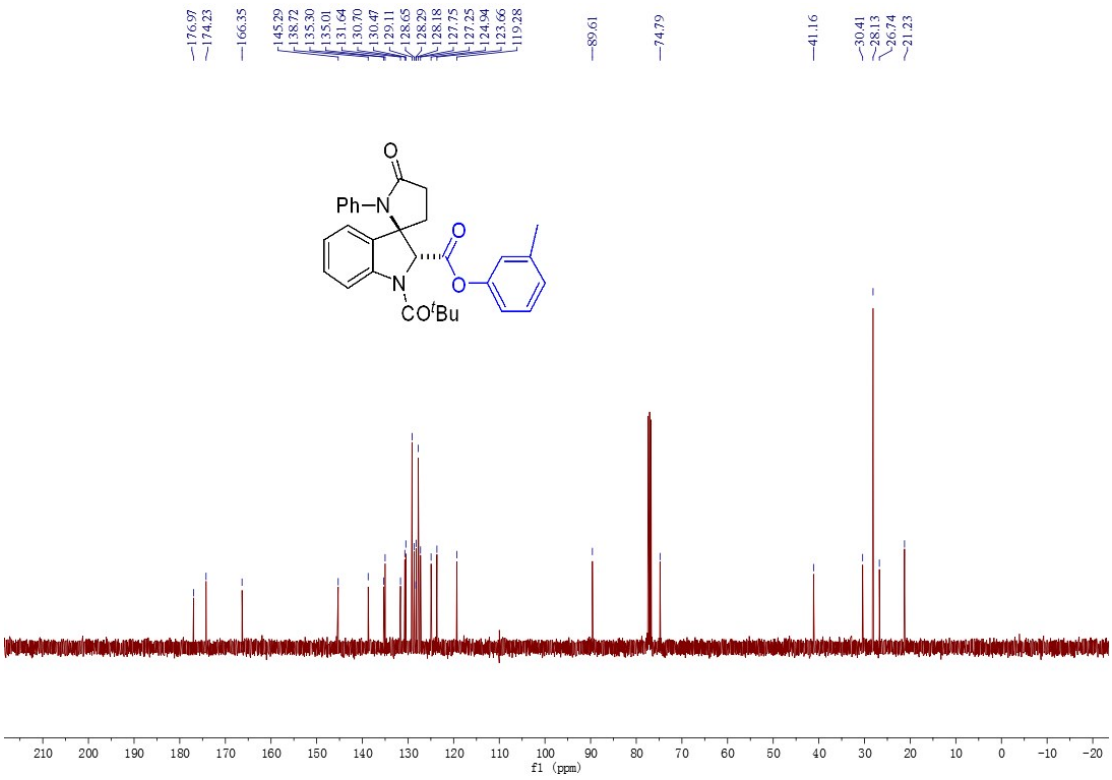
^{13}C NMR spectrum of compound **3r**



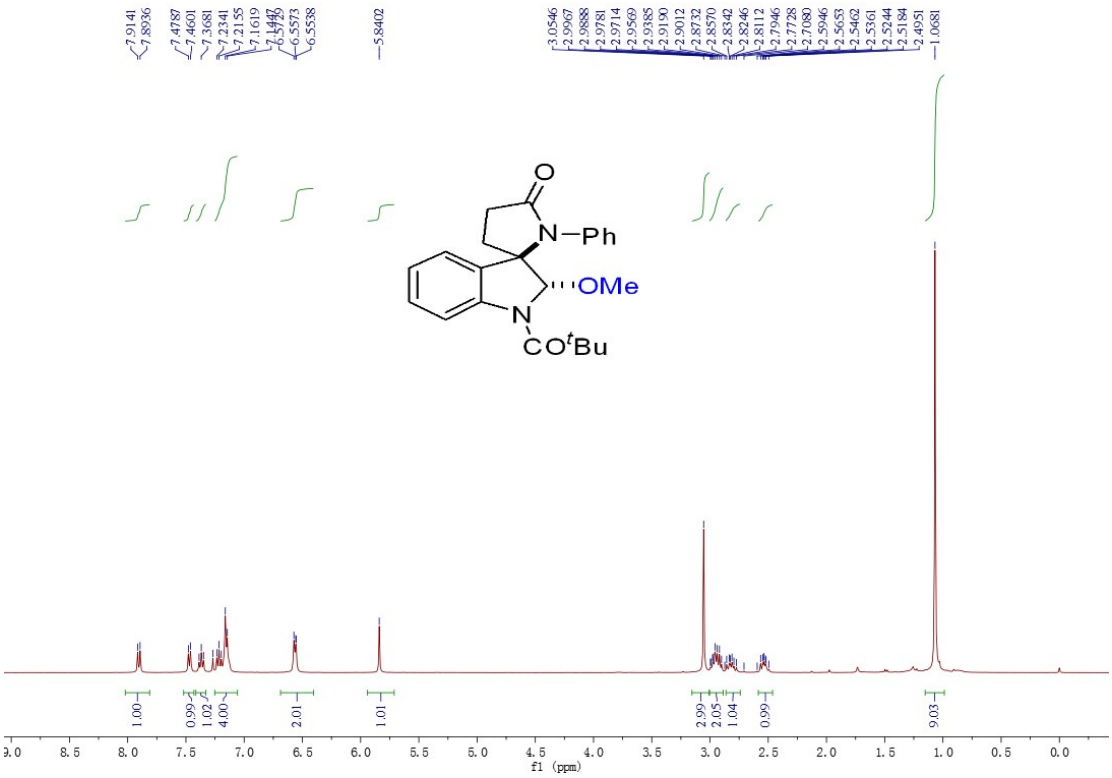
^{1}H NMR spectrum of compound **3s**



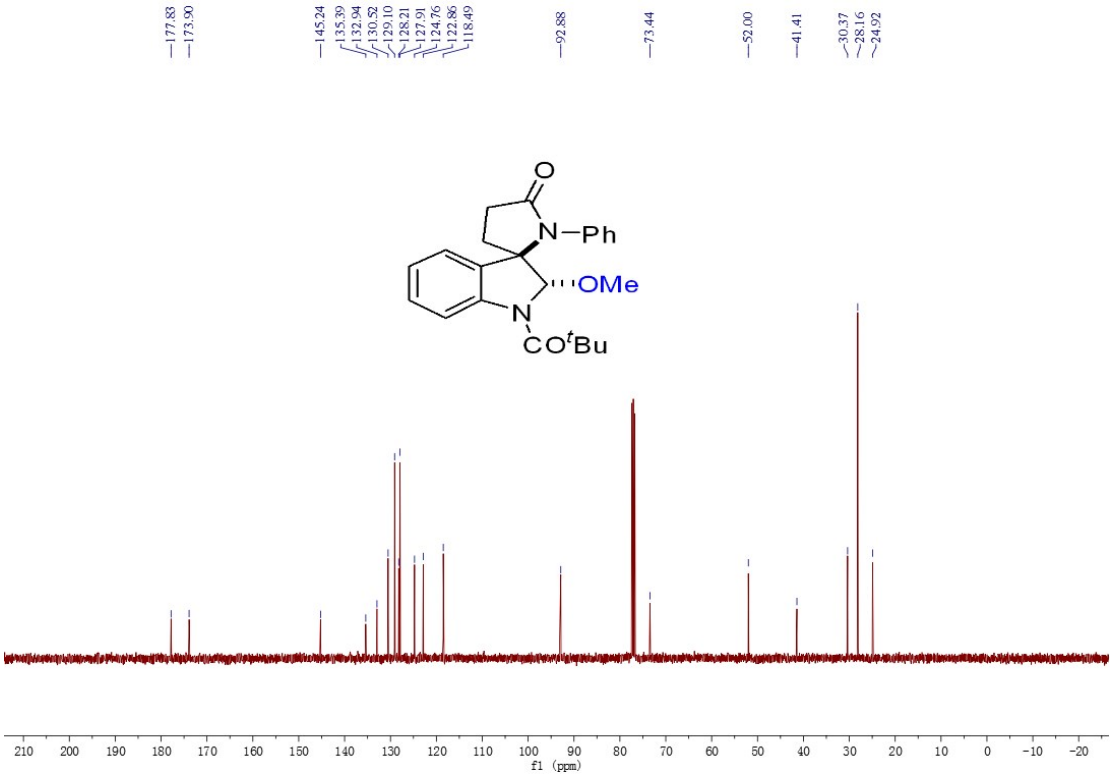
^{13}C NMR spectrum of compound **3s**



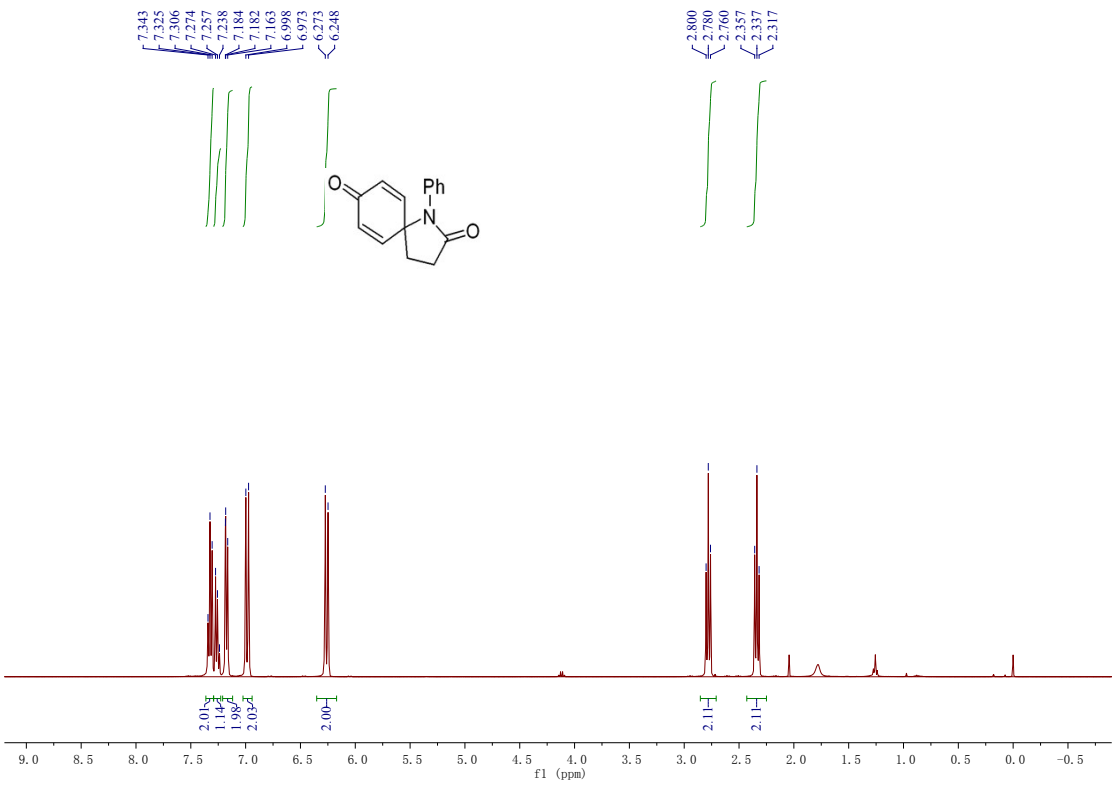
^{1}H NMR spectrum of compound **3v**



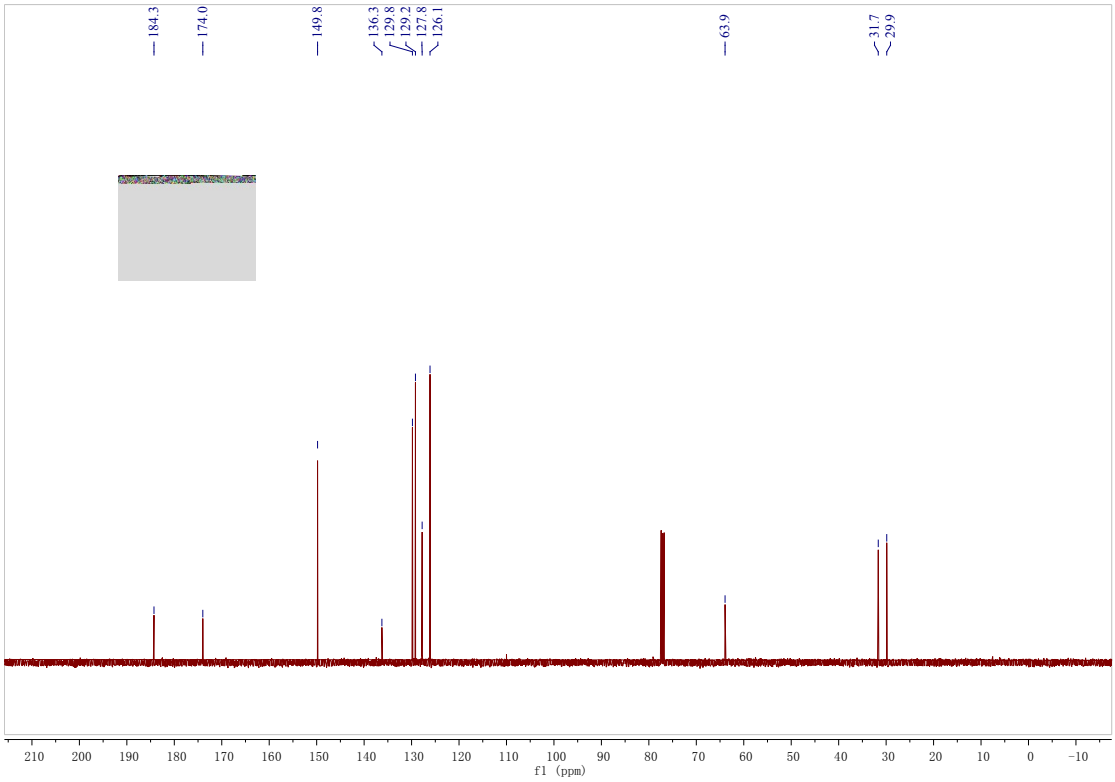
^{13}C NMR spectrum of compound **3v**



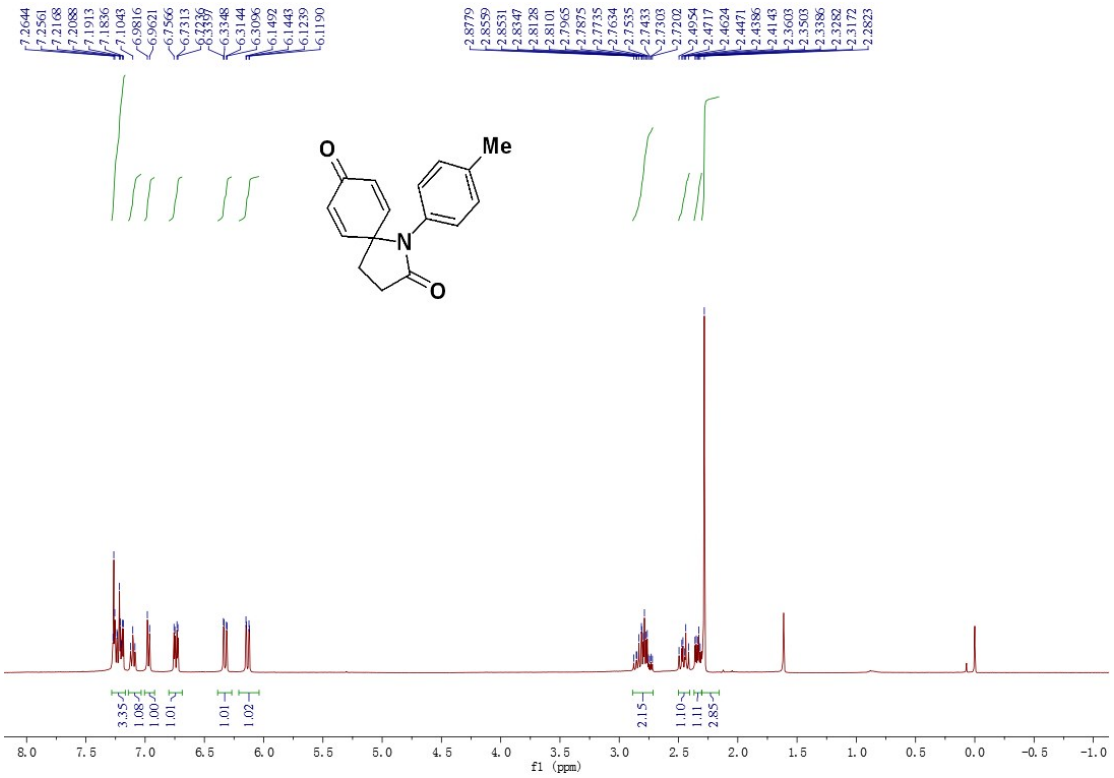
^{1}H NMR spectrum of compound **5a**



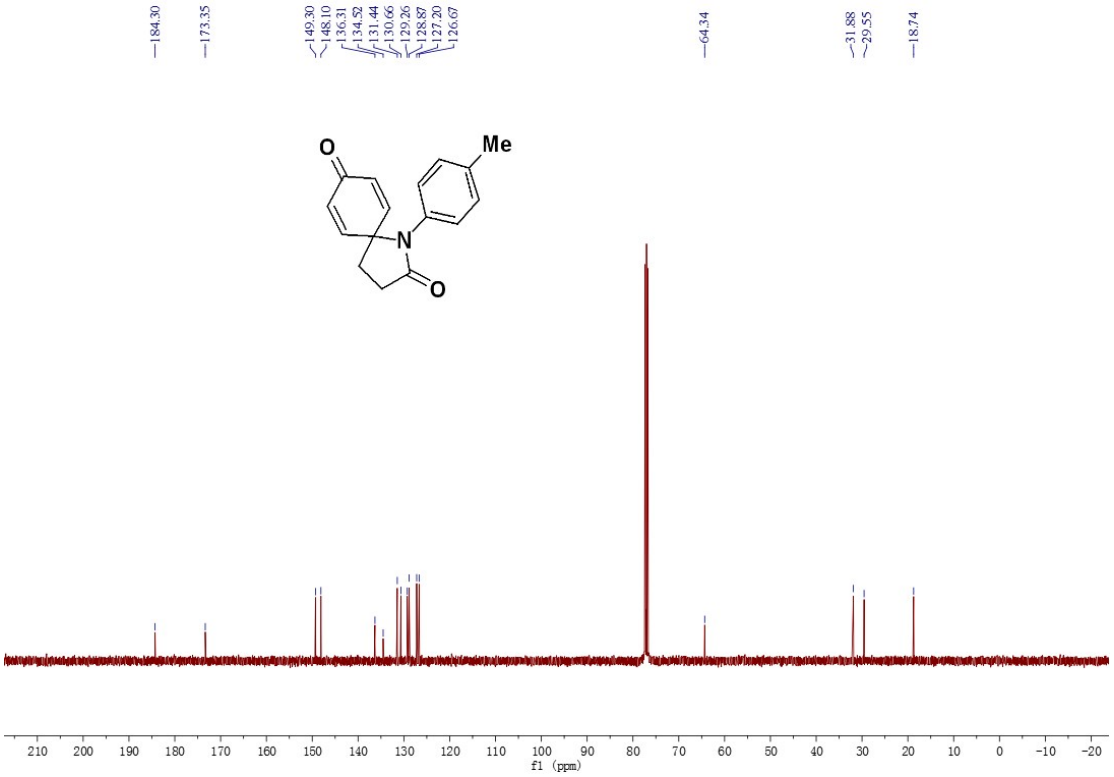
^{13}C NMR spectrum of compound **5a**



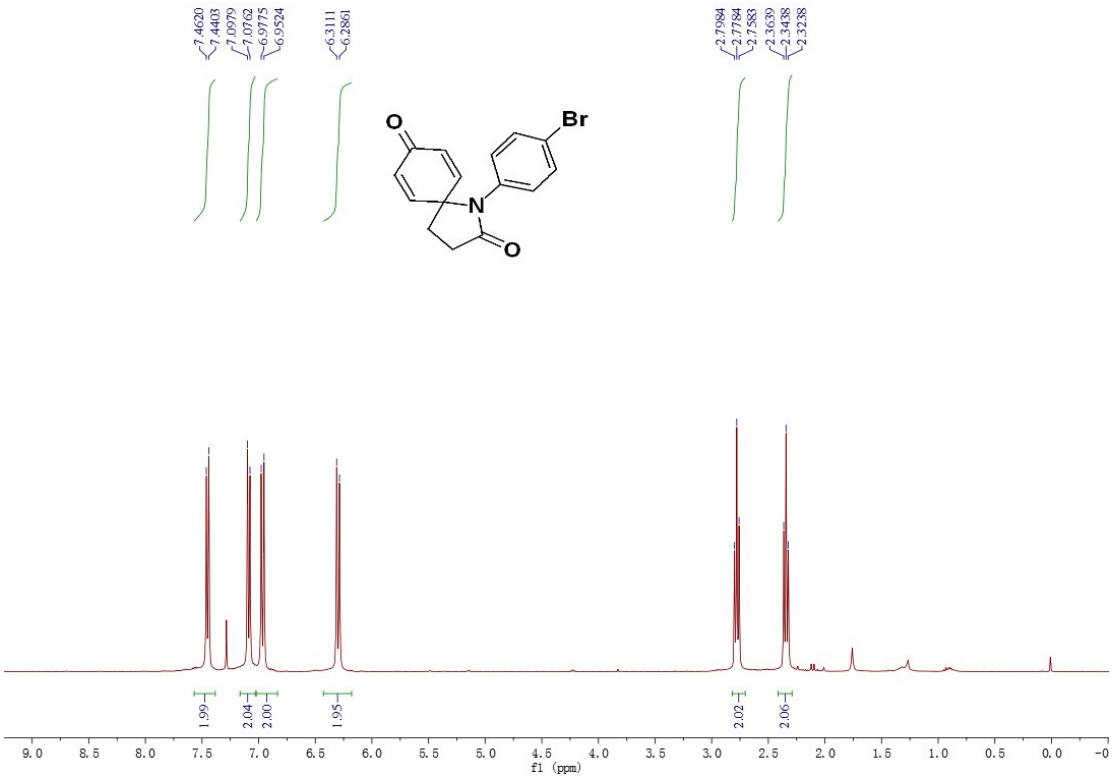
^{1}H NMR spectrum of compound **5b**



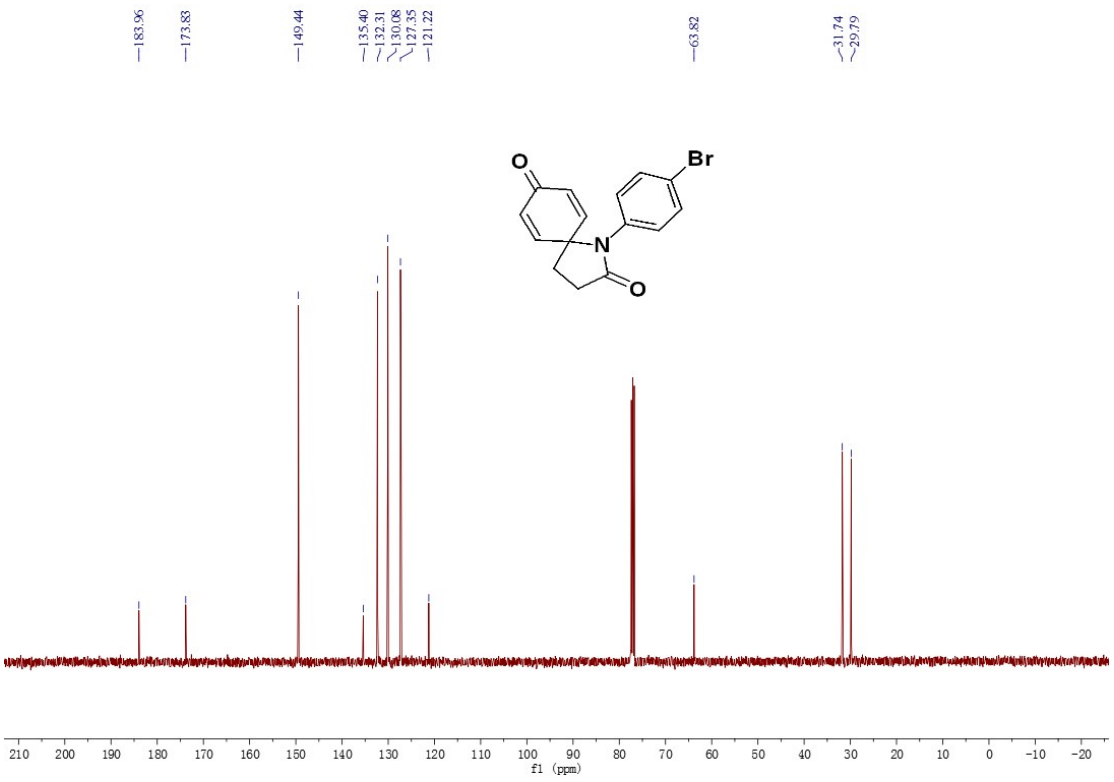
^{13}C NMR spectrum of compound **5b**



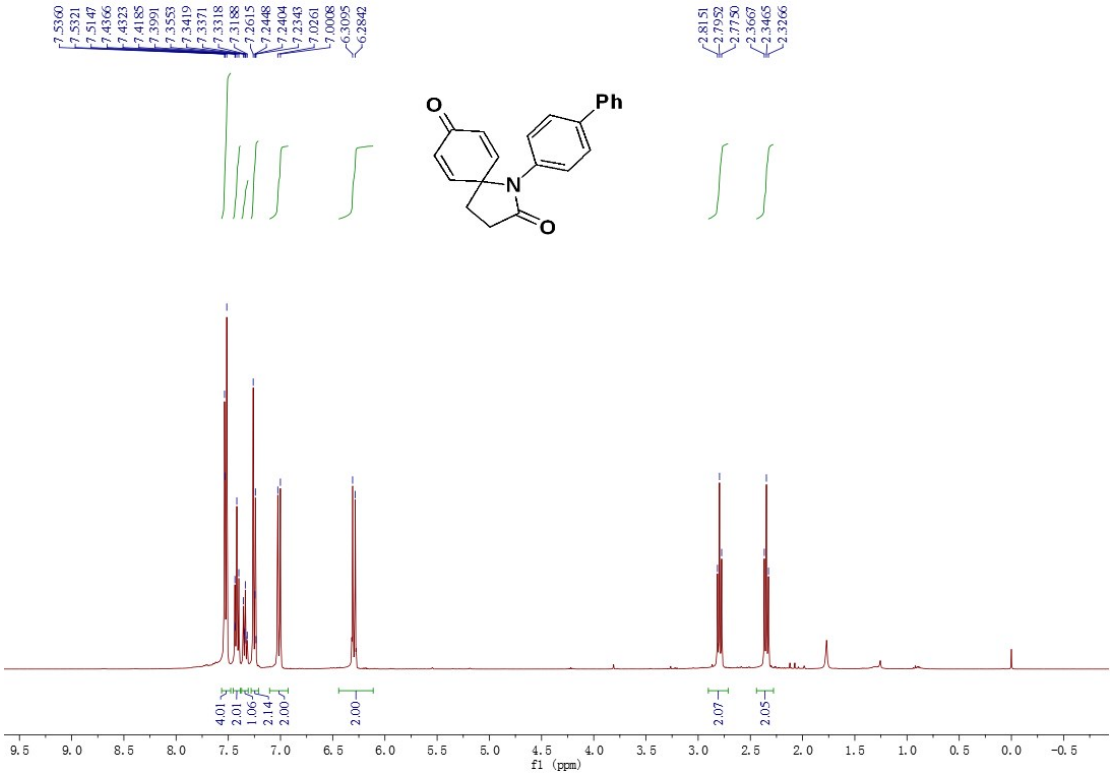
^{1}H NMR spectrum of compound **5c**



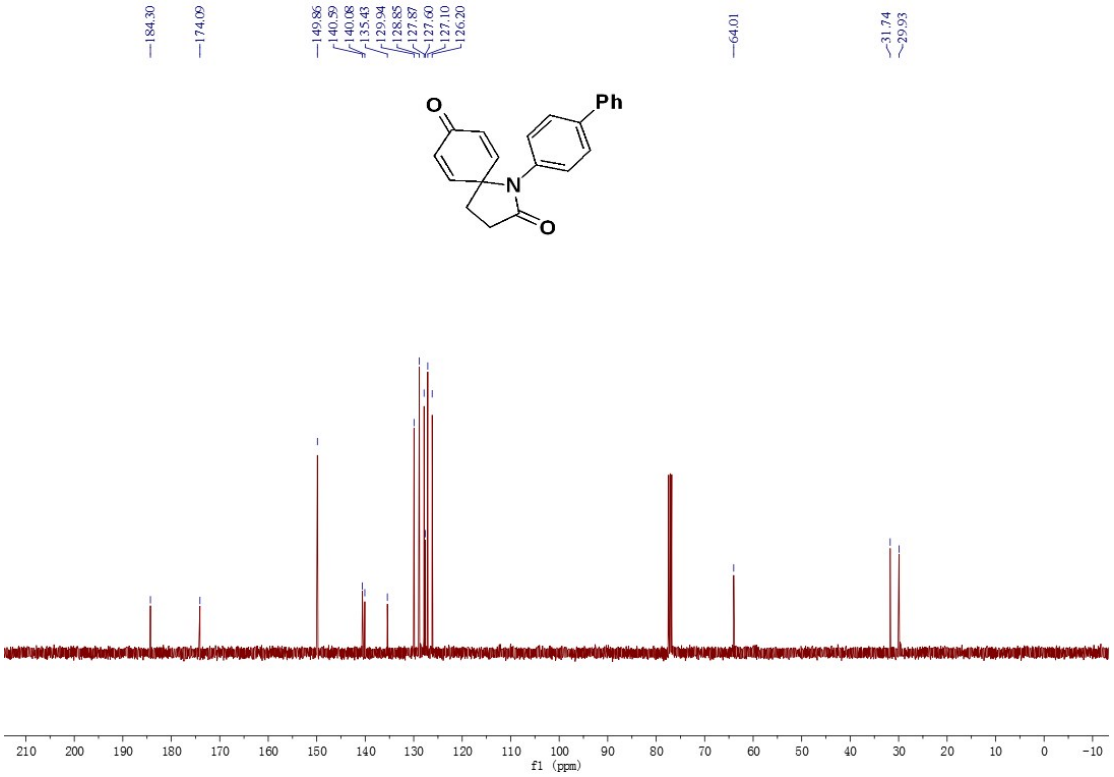
^{13}C NMR spectrum of compound **5c**



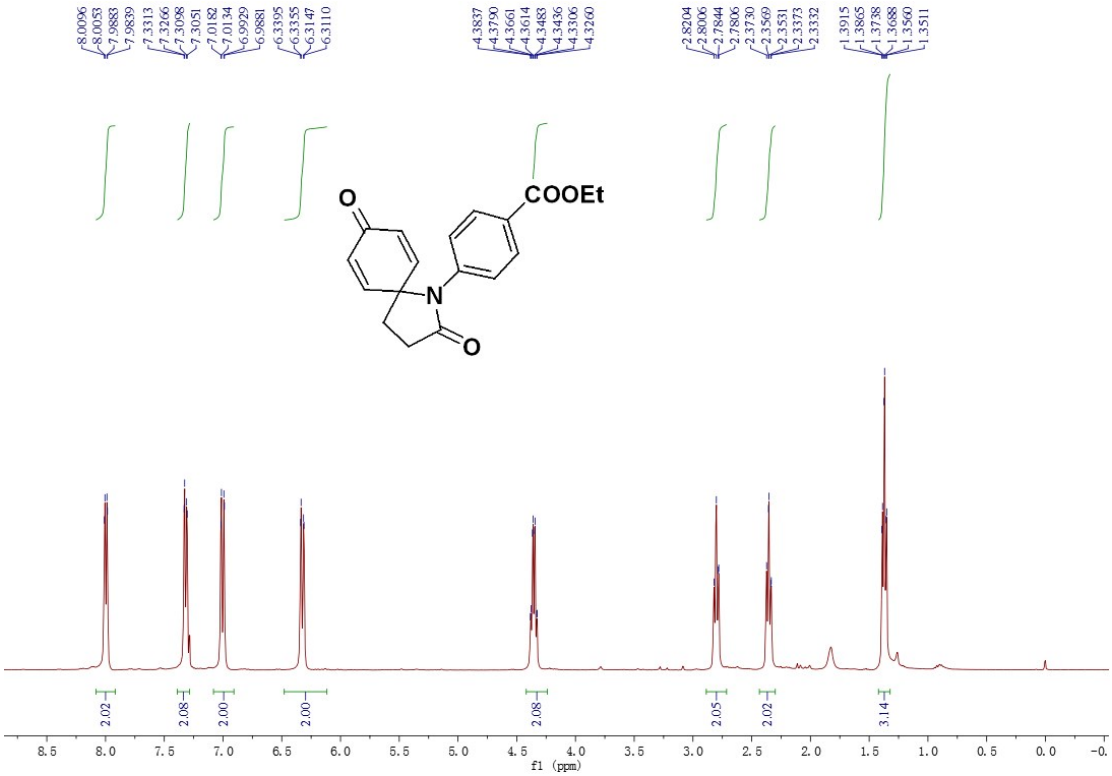
^{1}H NMR spectrum of compound **5d**



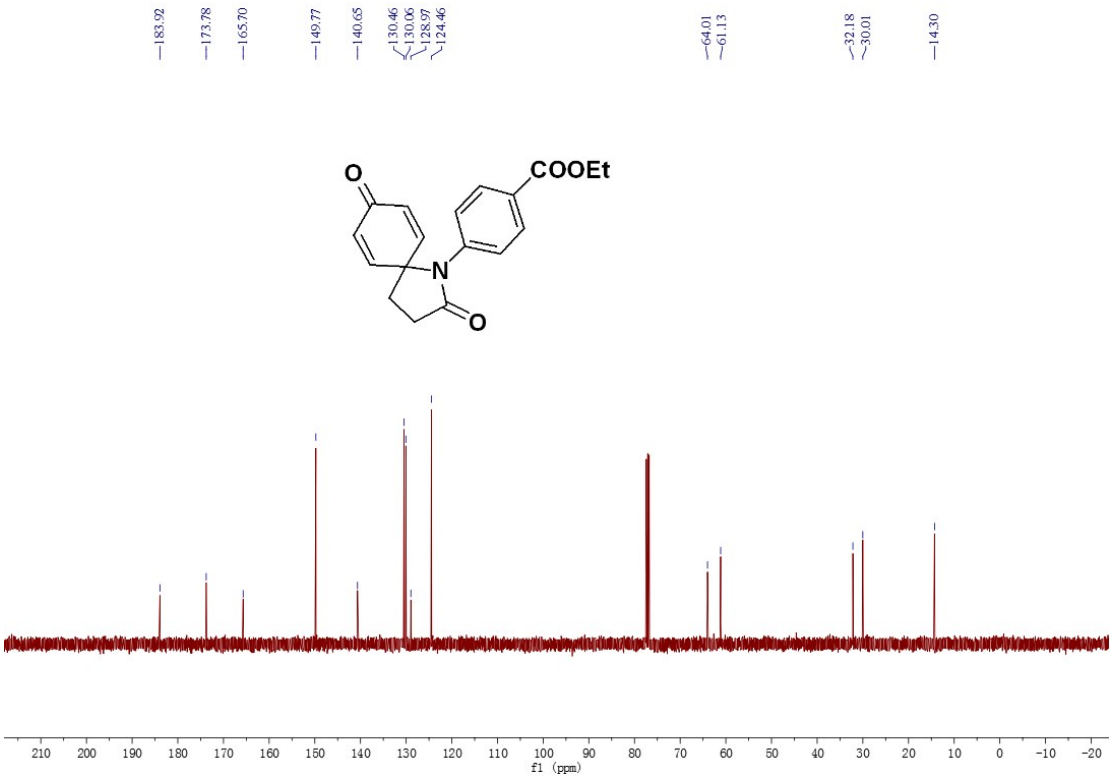
^{13}C NMR spectrum of compound **5d**



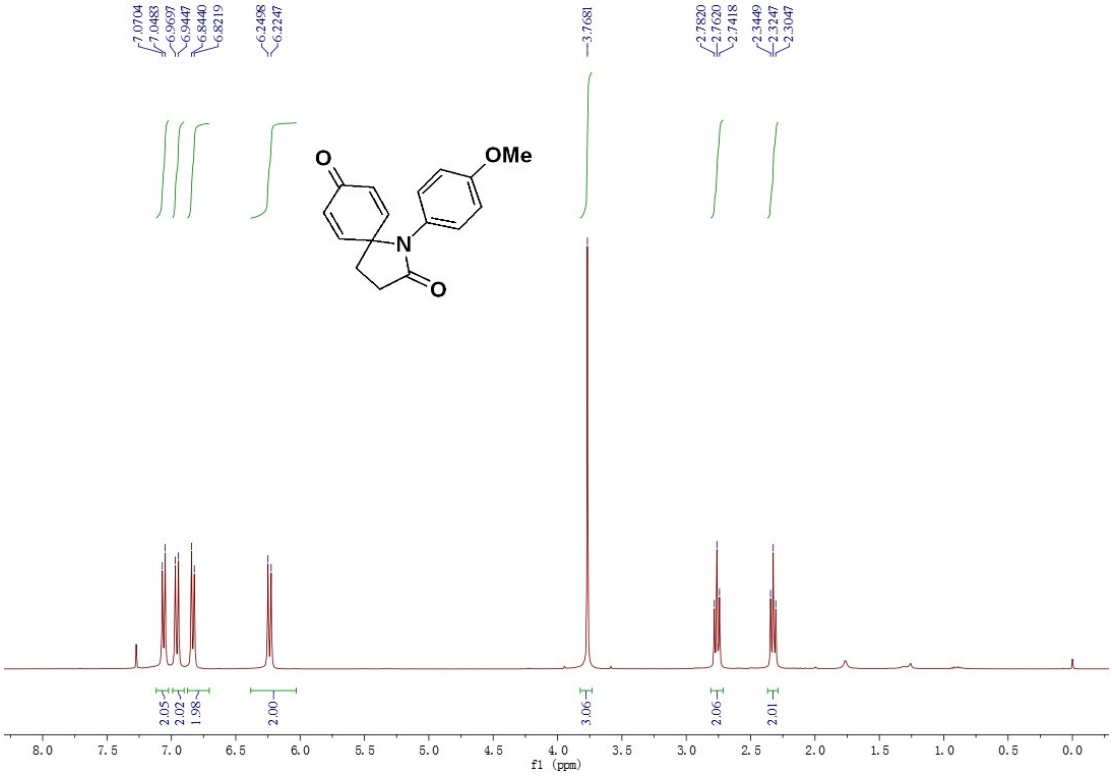
^{1}H NMR spectrum of compound **5e**



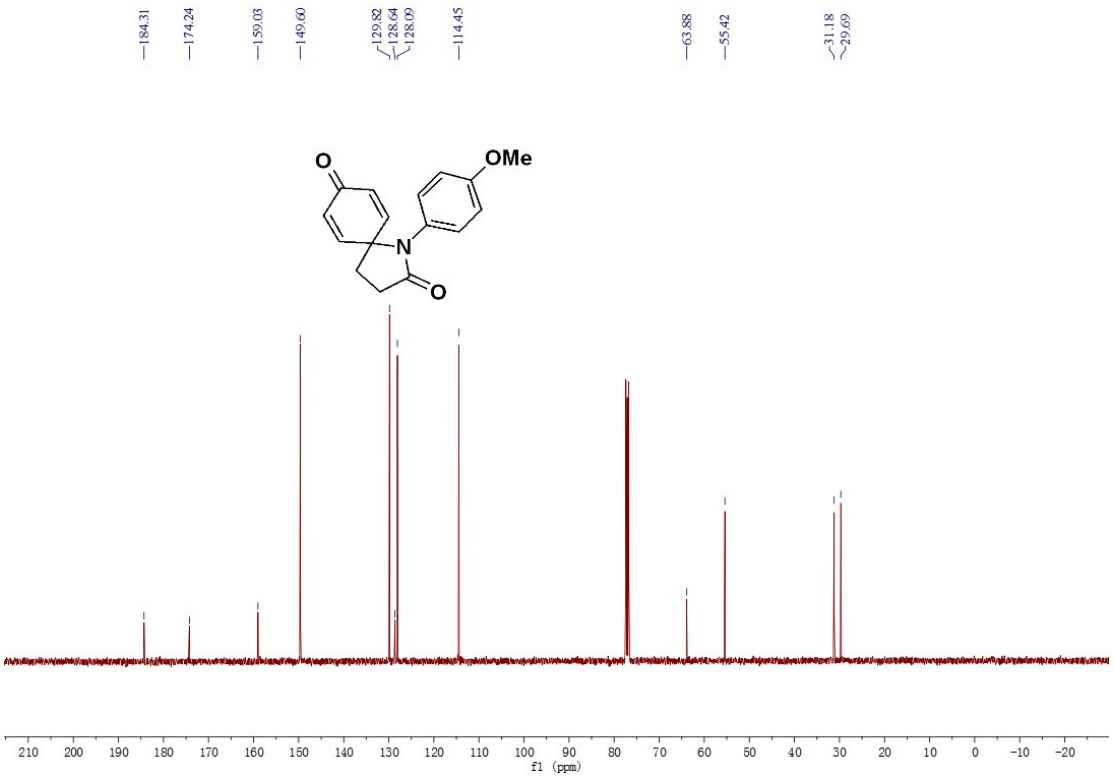
^{13}C NMR spectrum of compound **5e**



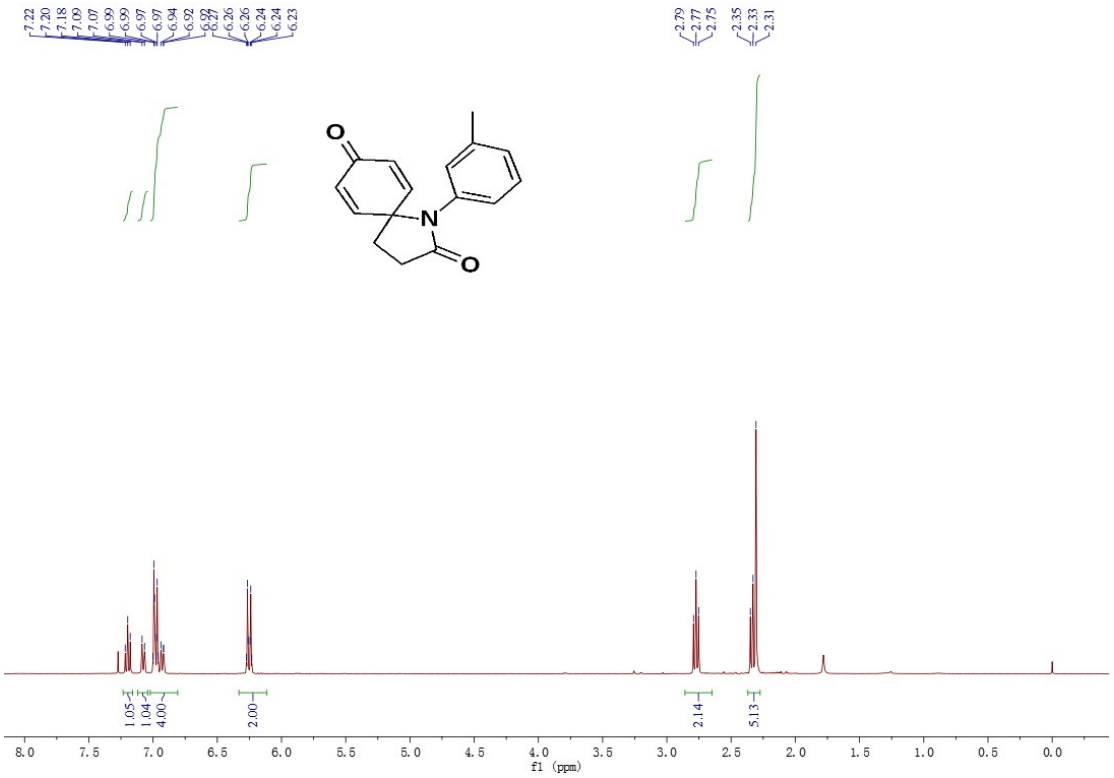
^{1}H NMR spectrum of compound **5f**



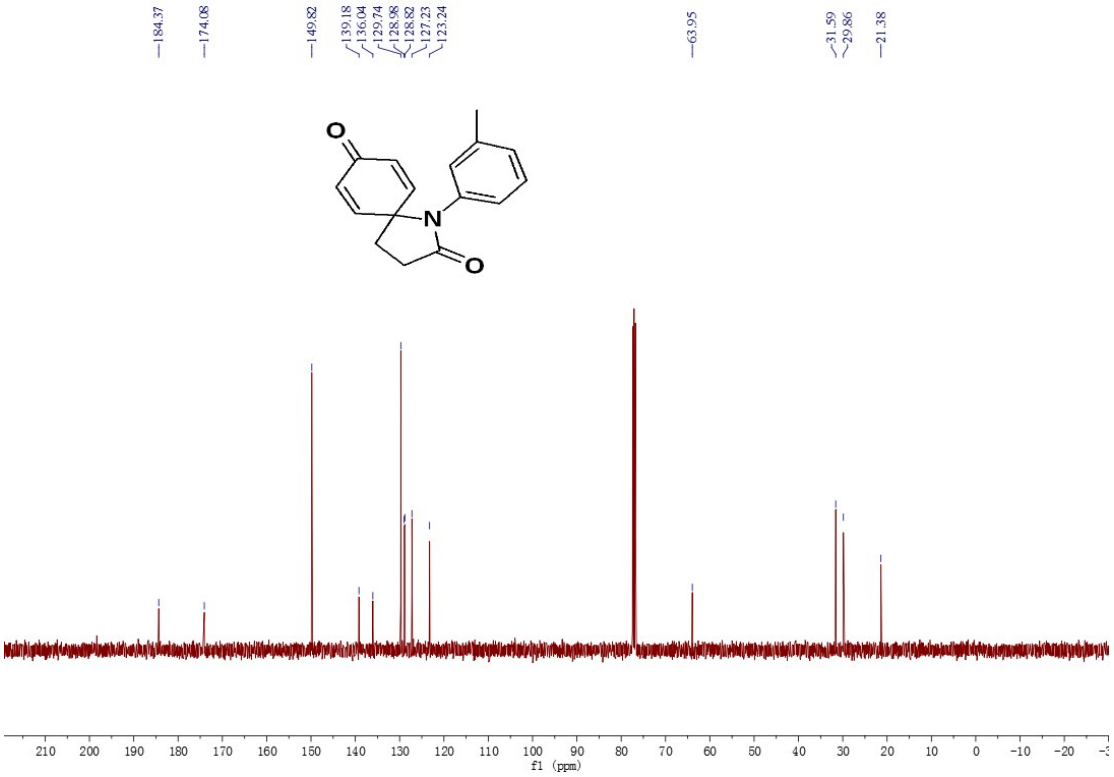
^{13}C NMR spectrum of compound **5f**



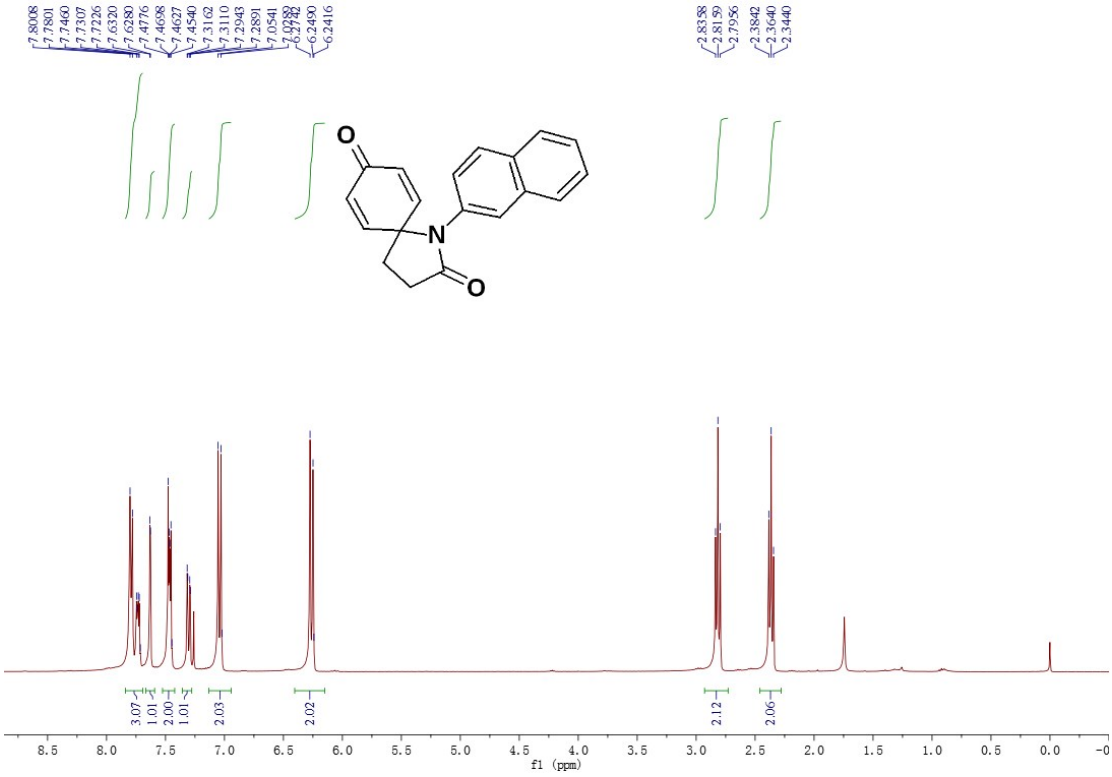
^{1}H NMR spectrum of compound **5g**



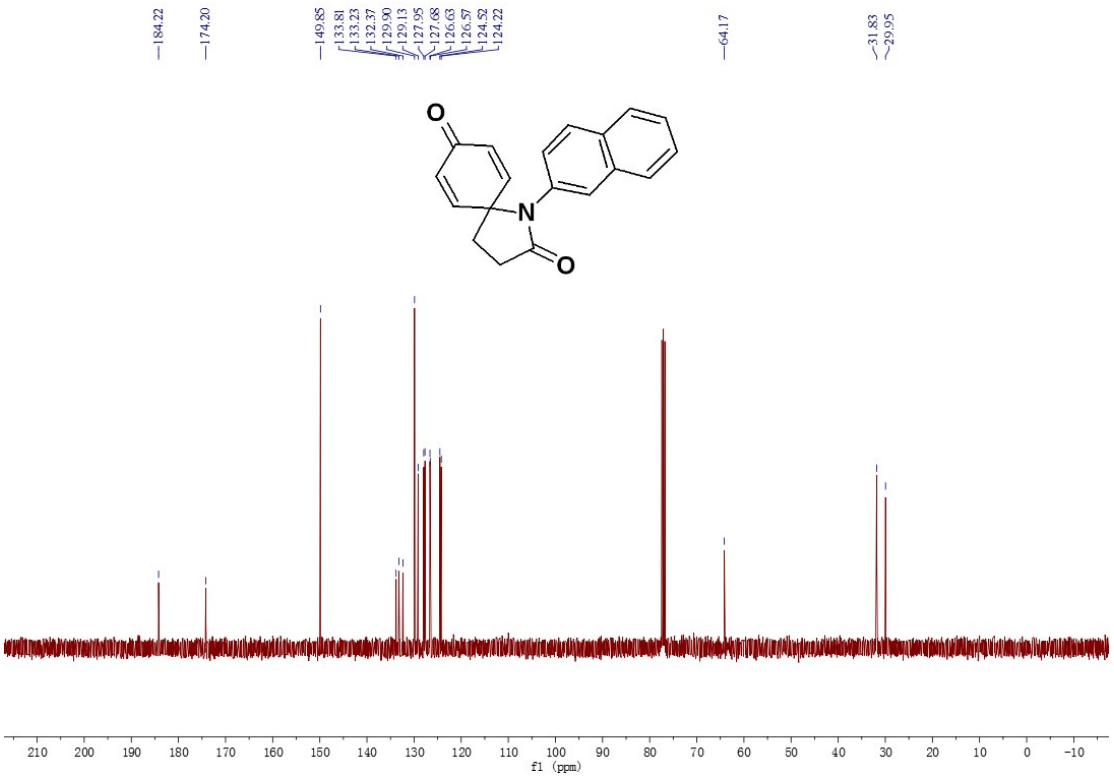
^{13}C NMR spectrum of compound **5g**



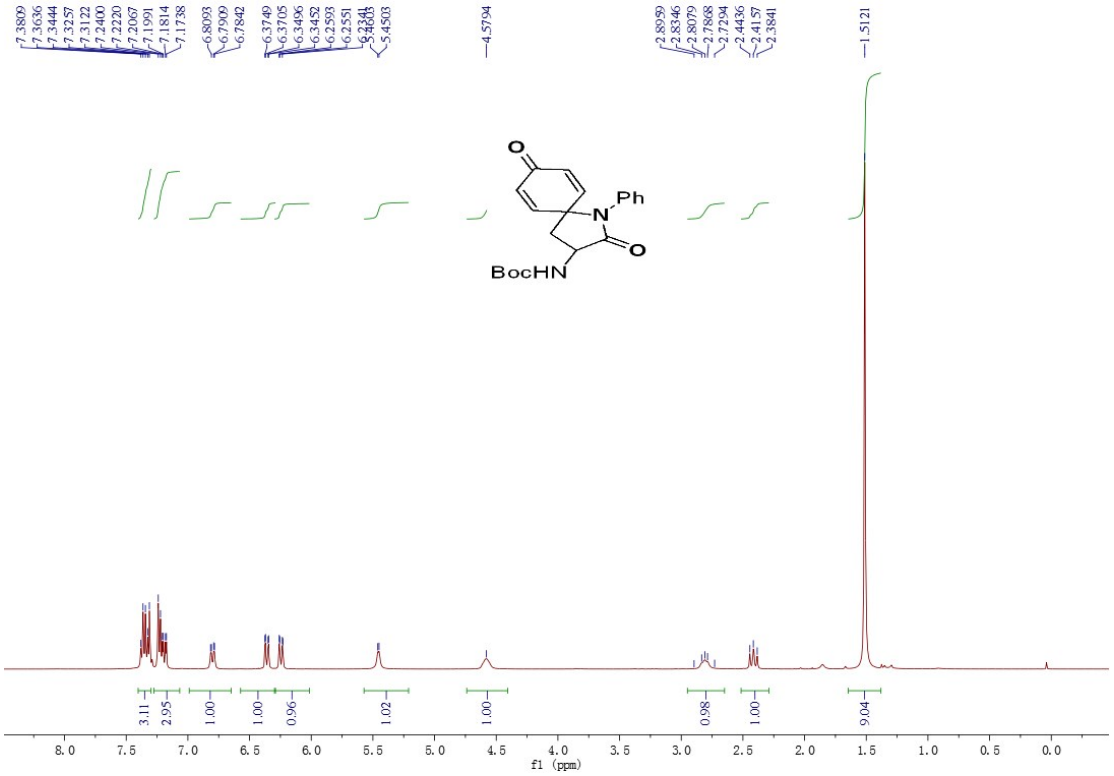
^{1}H NMR spectrum of compound **5h**



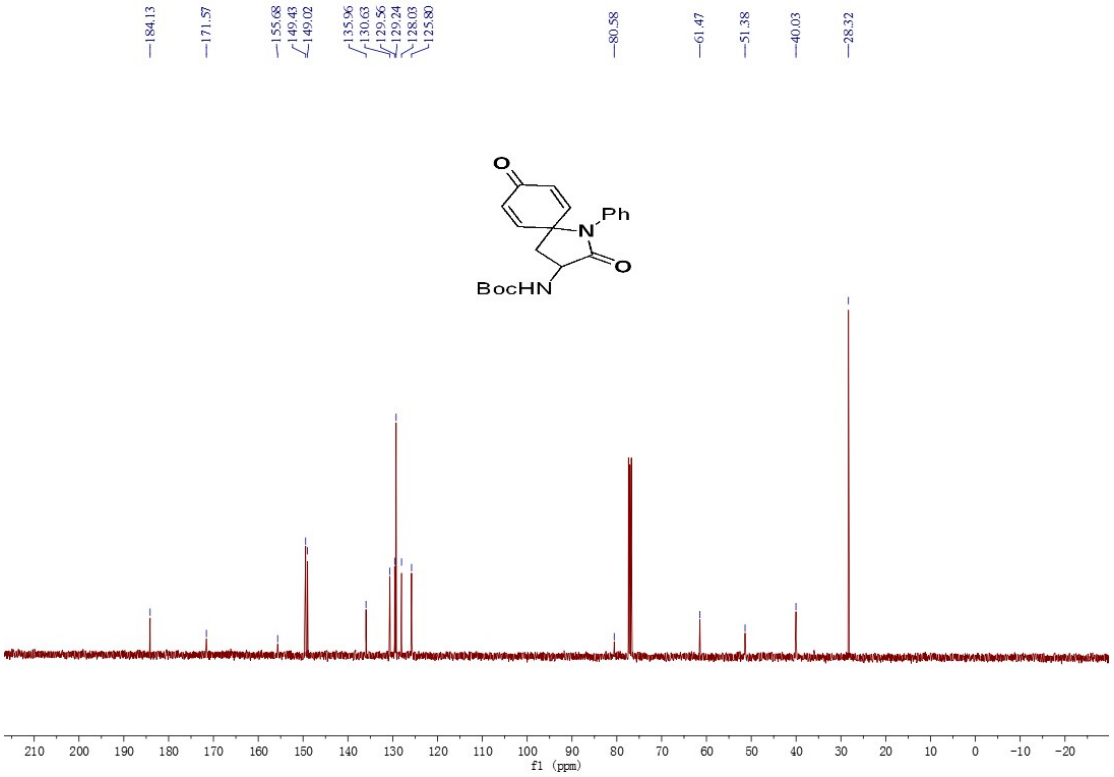
^{13}C NMR spectrum of compound **5h**



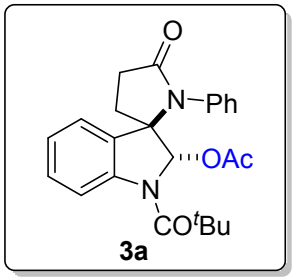
^{1}H NMR spectrum of compound **5i**



^{13}C NMR spectrum of compound **5i**



5. X-ray single crystal data for product



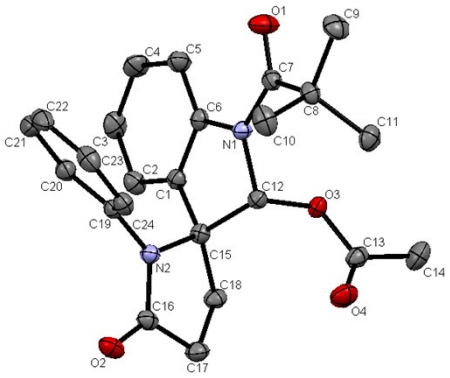


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Table 1. Crystal data and structure refinement for **r20191113b**.

Table 1 Crystal data and structure refinement for r20191113b.

Identification code	r20191113b
Empirical formula	$C_{24}H_{26}N_2O_4$
Formula weight	406.47
Temperature/K	123.15
Crystal system	triclinic

Space group	P-1
a/Å	9.6454(4)
b/Å	10.4111(7)
c/Å	11.3573(5)
α/°	75.236(5)
β/°	81.627(4)
γ/°	67.452(5)
Volume/Å^3	1017.07(10)
Z	2
ρ_{calc}g/cm^3	1.327
μ/mm^{-1}	0.091
F(000)	432.0
Crystal size/mm^3	0.2 × 0.18 × 0.16
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.34 to 52.738
Index ranges	-11 ≤ h ≤ 12, -13 ≤ k ≤ 13, -14 ≤ l ≤ 14
Reflections collected	10720
Independent reflections	4138 [R_{int} = 0.0267, R_{sigma} = 0.0284]
Data/restraints/parameters	4138/0/275
Goodness-of-fit on F^2	0.997
Final R indexes [I>=2σ (I)]	R_1 = 0.0414, wR_2 = 0.1080

Final R indexes [all data]	R_1 = 0.0461, wR_2 = 0.1135
Largest diff. peak/hole / e Å^{-3}	0.23/-0.26

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for r20191113b. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	5652.4(11)	2004.2(10)	137.1(9)	32.3(2)
O2	1464.0(11)	3590.6(10)	5905.8(8)	26.0(2)
O3	2152.9(10)	1067.2(9)	2053.7(8)	19.1(2)
O4	1943.3(11)	-287.2(10)	3920.1(9)	30.3(2)
N1	3766.8(11)	2320.7(10)	1578.6(9)	16.6(2)
N2	2051.1(11)	3603.0(11)	3870.2(9)	17.3(2)
C1	1578.4(13)	4261.6(12)	1661.2(11)	17.2(3)
C2	525.3(15)	5610.0(13)	1268.8(11)	22.5(3)
C3	816.3(16)	6444.2(14)	165.4(12)	26.2(3)
C4	2150.0(16)	5928.6(14)	-505.3(12)	27.3(3)
C5	3212.1(15)	4567.6(14)	-128.4(11)	23.8(3)
C6	2891.5(13)	3734.3(12)	961.1(11)	17.4(3)
C7	5170.7(14)	1514.9(13)	1129.4(11)	19.2(3)
C8	6159.6(14)	73.9(13)	1892.8(11)	20.1(3)

C9	7590.3(15)	-497.1(15)	1095.6(13)	29.5(3)
C10	6612.5(16)	313.2(15)	3034.8(13)	29.1(3)
C11	5449.2(15)	-1070.5(13)	2244.6(13)	25.8(3)
C12	2819.4(13)	1830.1(12)	2559.8(11)	16.6(2)
C13	1789.0(14)	5.0(13)	2841.7(12)	22.7(3)
C14	1204.2(18)	-721.9(16)	2162.9(14)	32.4(3)
C15	1585.6(13)	3217.4(12)	2857.9(10)	16.5(2)
C16	1247.5(14)	3382.8(13)	4949.9(11)	19.2(3)
C17	85.8(15)	2834.1(14)	4744.1(12)	23.4(3)
C18	42.7(13)	3138.8(13)	3364.0(11)	20.6(3)
C19	3328.3(13)	4019.5(13)	3764.6(10)	17.4(3)
C20	3299.8(14)	5321.9(13)	3031.0(11)	21.2(3)
C21	4555.2(15)	5693.8(15)	2919.9(12)	25.4(3)
C22	5835.1(15)	4783.3(15)	3532.7(13)	27.1(3)
C23	5855.6(15)	3492.1(15)	4272.5(13)	26.0(3)
C24	4599.7(14)	3115.1(13)	4396.2(11)	21.2(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for r20191113b. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	25.8(5)	29.5(5)	28.4(5)	0.3(4)	9.4(4)	-3.8(4)
O2	27.5(5)	30.5(5)	17.6(4)	-6.7(4)	1.4(4)	-8.1(4)

O3	21.5(4)	16.4(4)	20.6(4)	-2.5(3)	-1.5(3)	-9.0(4)
O4	34.5(6)	27.6(5)	25.9(5)	2.2(4)	0.2(4)	-13.7(4)
N1	15.5(5)	14.8(5)	16.9(5)	-1.9(4)	0.7(4)	-4.4(4)
N2	17.7(5)	18.9(5)	15.6(5)	-4.0(4)	0.5(4)	-7.3(4)
C1	19.0(6)	17.2(6)	16.2(6)	-3.3(5)	-2.4(4)	-7.1(5)
C2	21.8(6)	21.4(6)	21.1(6)	-4.8(5)	-2.9(5)	-3.6(5)
C3	28.6(7)	16.9(6)	25.7(7)	-0.1(5)	-6.9(5)	-1.3(5)
C4	33.7(7)	22.7(7)	19.6(6)	2.8(5)	-1.2(5)	-8.6(6)
C5	26.2(7)	22.2(6)	19.5(6)	-3.0(5)	2.4(5)	-7.2(5)
C6	18.5(6)	15.2(6)	18.1(6)	-2.9(5)	-3.6(5)	-5.3(5)
C7	17.4(6)	19.0(6)	22.1(6)	-7.0(5)	-0.1(5)	-6.4(5)
C8	17.9(6)	18.0(6)	22.4(6)	-5.8(5)	-1.9(5)	-3.2(5)
C9	20.4(7)	27.0(7)	34.8(8)	-9.7(6)	2.3(6)	-1.0(6)
C10	26.1(7)	27.0(7)	32.3(7)	-10.0(6)	-10.1(6)	-2.1(6)
C11	24.7(7)	18.3(6)	31.9(7)	-3.5(5)	-4.1(5)	-5.4(5)
C12	17.2(6)	16.9(6)	16.2(6)	-3.0(4)	-1.3(4)	-7.0(5)
C13	19.2(6)	16.7(6)	28.3(7)	-1.1(5)	1.2(5)	-5.8(5)
C14	35.8(8)	27.3(7)	39.4(8)	-3.6(6)	-3.4(6)	-19.2(6)
C15	15.7(6)	16.6(6)	17.1(6)	-3.3(4)	-2.0(4)	-5.5(5)
C16	18.4(6)	16.2(6)	18.2(6)	-2.4(5)	0.9(5)	-2.4(5)
C17	20.6(6)	24.6(6)	23.6(6)	-4.1(5)	4.3(5)	-9.2(5)
C18	15.3(6)	22.1(6)	23.2(6)	-4.0(5)	0.7(5)	-6.7(5)

C19	18.3(6)	19.2(6)	16.3(6)	-7.7(5)	1.4(4)	-6.9(5)
C20	23.2(6)	20.1(6)	20.4(6)	-5.9(5)	-0.7(5)	-7.2(5)
C21	30.1(7)	25.5(7)	25.2(7)	-9.0(5)	5.8(5)	-15.6(6)
C22	21.1(6)	35.7(8)	32.6(7)	-19.1(6)	7.6(5)	-14.7(6)
C23	20.0(6)	30.6(7)	28.9(7)	-14.2(6)	-3.2(5)	-5.0(5)
C24	23.4(6)	20.5(6)	19.4(6)	-6.5(5)	-1.6(5)	-6.0(5)

Table 4 Bond Lengths for r20191113b.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C7	1.2174(15)	C5	C6	1.3880(17)
O2	C16	1.2203(15)	C7	C8	1.5468(17)
O3	C12	1.4514(14)	C8	C9	1.5359(18)
O3	C13	1.3597(15)	C8	C10	1.5324(18)
O4	C13	1.2013(16)	C8	C11	1.5338(18)
N1	C6	1.4367(15)	C12	C15	1.5590(17)
N1	C7	1.3940(16)	C13	C14	1.4955(19)
N1	C12	1.4523(15)	C15	C18	1.5402(16)
N2	C15	1.4861(14)	C16	C17	1.5073(18)
N2	C16	1.3686(15)	C17	C18	1.5214(18)
N2	C19	1.4354(15)	C19	C20	1.3913(17)
C1	C2	1.3859(17)	C19	C24	1.3897(17)
C1	C6	1.3906(17)	C20	C21	1.3864(18)

C1	C15	1.5076(16)		C21	C22	1.387(2)	
C2	C3	1.3904(18)		C22	C23	1.386(2)	
C3	C4	1.385(2)		C23	C24	1.3886(19)	
C4	C5	1.3949(18)					

Table 5 Bond Angles for r20191113b.

Atom	Atom	Atom	Angle/	Atom	Atom	Atom	Angle/
C13	O3	C12	116.99(9)	O3	C12	N1	105.92(9)
C6	N1	C12	108.05(9)	O3	C12	C15	110.23(9)
C7	N1	C6	123.36(10)	N1	C12	C15	104.90(9)
C7	N1	C12	127.52(10)	O3	C13	C14	110.03(11)
C16	N2	C15	113.77(10)	O4	C13	O3	123.45(12)
C16	N2	C19	122.74(10)	O4	C13	C14	126.52(12)
C19	N2	C15	123.16(9)	N2	C15	C1	112.40(9)
C2	C1	C6	121.04(11)	N2	C15	C12	109.70(9)
C2	C1	C15	128.60(11)	N2	C15	C18	101.59(9)
C6	C1	C15	110.22(10)	C1	C15	C12	100.97(9)
C1	C2	C3	118.71(12)	C1	C15	C18	115.47(10)
C4	C3	C2	119.81(12)	C18	C15	C12	117.05(10)
C3	C4	C5	122.08(12)	O2	C16	N2	124.92(11)
C6	C5	C4	117.48(12)	O2	C16	C17	126.93(11)
C1	C6	N1	109.25(10)	N2	C16	C17	108.15(10)

C5	C6	N1	129.88(11)	C16	C17	C18	104.42(10)
C5	C6	C1	120.82(11)	C17	C18	C15	105.58(10)
O1	C7	N1	118.50(11)	C20	C19	N2	120.30(11)
O1	C7	C8	119.72(11)	C24	C19	N2	119.78(11)
N1	C7	C8	121.66(10)	C24	C19	C20	119.92(11)
C9	C8	C7	106.51(10)	C21	C20	C19	119.51(12)
C10	C8	C7	109.77(10)	C20	C21	C22	120.73(12)
C10	C8	C9	108.28(11)	C23	C22	C21	119.66(12)
C10	C8	C11	110.30(11)	C22	C23	C24	120.02(12)
C11	C8	C7	114.71(10)	C23	C24	C19	120.15(12)
C11	C8	C9	106.99(11)				

Table 6 Torsion Angles for r20191113b.

A	B	C	D	Angle/	A	B	C	D	Angle/
O1	C7	C8	C9	-4.80(16)	C7	N1	C6	C5	-0.63(19)
O1	C7	C8	C10	112.22(13)	C7	N1	C12	O3	-75.67(14)
O1	C7	C8	C11	-122.95(13)	C7	N1	C12	C15	167.72(11)
O2	C16	C17	C18	166.02(12)	C12	O3	C13	O4	2.68(18)
O3	C12	C15	N2	152.45(9)	C12	O3	C13	C14	-176.65(10)
O3	C12	C15	C1	-88.75(10)	C12	N1	C6	C1	13.00(12)
O3	C12	C15	C18	37.46(13)	C12	N1	C6	C5	-169.57(12)
N1	C7	C8	C9	179.20(11)	C12	N1	C7	O1	163.20(11)

N1	C7	C8	C10 -63.79(14)
N1	C7	C8	C11 61.05(15)
N1	C12	C15	N2 -93.93(10)
N1	C12	C15	C1 24.86(11)
N1	C12	C15	C18 151.07(10)
N2	C15	C18	C17 -24.32(12)
N2	C16	C17	C18 -14.59(13)
N2	C19	C20	C21 -178.67(11)
N2	C19	C24	C23 178.21(11)
C1	C2	C3	C4 -1.07(19)
C1	C15	C18	C17 -146.22(10)
C2	C1	C6	N1 -179.67(10)
C2	C1	C6	C5 2.62(18)
C2	C1	C15	N2 -76.92(15)
C2	C1	C15	C12 166.25(12)
C2	C1	C15	C18 38.99(17)
C2	C3	C4	C5 1.9(2)
C3	C4	C5	C6 -0.4(2)
C4	C5	C6	N1 -179.00(12)
C4	C5	C6	C1 -1.82(18)
C6	N1	C7	O1 -3.50(18)
C6	N1	C7	C8 172.55(10)
C12	N1	C7	C8 -20.75(17)
C12	C15	C18	C17 95.10(12)
C13	O3	C12	N1 151.47(10)
C13	O3	C12	C15 -95.57(12)
C15	N2	C16	O2 178.02(11)
C15	N2	C16	C17 -1.38(13)
C15	N2	C19	C20 69.87(15)
C15	N2	C19	C24 -109.97(13)
C15	C1	C2	C3 174.23(12)
C15	C1	C6	N1 4.20(13)
C15	C1	C6	C5 -173.51(11)
C16	N2	C15	C1 140.37(10)
C16	N2	C15	C12 -108.15(11)
C16	N2	C15	C18 16.36(12)
C16	N2	C19	C20 -117.13(13)
C16	N2	C19	C24 63.02(16)
C16	C17	C18	C15 24.26(13)
C19	N2	C15	C1 -46.06(14)
C19	N2	C15	C12 65.42(13)
C19	N2	C15	C18 -170.07(10)
C19	N2	C16	O2 4.43(19)
C19	N2	C16	C17 -174.98(10)

C6N1 C12O3	92.67(10)	C19C20C21C22	-0.09(19)
C6N1 C12C15	-23.94(11)	C20C19C24C23	-1.64(18)
C6C1 C2 C3	-1.13(18)	C20C21C22C23	-0.53(19)
C6C1 C15N2	98.84(11)	C21C22C23C24	0.1(2)
C6C1 C15C12	-17.98(12)	C22C23C24C19	1.02(19)
C6C1 C15C18	-145.24(11)	C24C19C20C21	1.17(18)
C7N1 C6 C1	-178.07(10)		

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for r20191113b.

Atom	*x*	*y*	*z*	U(eq)
H2	-377.94	5957.64	1744.4	27
H3	101.96	7365.42	-127.36	31
H4	2348.15	6520.4	-1245.64	33
H5	4120.38	4224.1	-599.56	29
H9A	7327.44	-695.07	376.25	44
H9B	8289.24	-1376.64	1565.86	44
H9C	8067.05	216	836	44
H10A	7032.72	1068.77	2800.18	44
H10B	7368.53	-570.42	3444.08	44
H10C	5725.78	594.18	3590.55	44
H11A	4655.1	-838.79	2885.7	39

H11B	6220.89	-2000.35	2544.66	39
H11C	5017.65	-1103.73	1528.22	39
H12	3403.74	1214.03	3287.32	20
H14A	378.22	-11.4	1671.62	49
H14B	837.75	-1410.33	2744.95	49
H14C	2013.48	-1220.28	1627.4	49
H17A	-907.93	3338.61	5113.93	28
H17B	382.15	1799.49	5097.94	28
H18A	-775.22	4052.19	3064.9	25
H18B	-121.69	2367.73	3111.33	25
H20	2426.57	5951.66	2609.35	25
H21	4538.47	6581.97	2418.46	30
H22	6693.34	5043.13	3446.01	33
H23	6729.12	2865.13	4694.93	31
H24	4609.56	2236.97	4913.7	25

Experimental

Single crystals of $C_{24}H_{26}N_2O_4$ **[r20191113b]** were **[]**. A suitable crystal was selected and **[]** on a **'Rigaku Saturn 70 CCD'** diffractometer. The crystal was kept at 123.15 K during data collection. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [r20191113b]

Crystal Data for $C_{24}H_{26}N_2O_4$ (M =406.47 g/mol): triclinic, space group P-1 (no. 2), a = 9.6454(4) Å, b = 10.4111(7) Å, c = 11.3573(5) Å, α = 75.236(5)°, β = 81.627(4)°, γ = 67.452(5)°, V = 1017.07(10) Å^3, Z = 2, T = 123.15 K, μ(MoKα) = 0.091 mm^{-1}, *Dcalc* = 1.327 g/cm^3, 10720 reflections measured (4.34° ≤ 2Θ ≤ 52.738°), 4138 unique (R_{int} = 0.0267, R_{sigma} = 0.0284) which were used in all calculations. The final R_1 was 0.0414 (I > 2σ(I)) and wR_2 was 0.1135 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups

2.a Ternary CH refined with riding coordinates:

C12(H12)

2.b Secondary CH2 refined with riding coordinates:

C17(H17A,H17B), C18(H18A,H18B)

2.c Aromatic/amide H refined with riding coordinates:

C2(H2), C3(H3), C4(H4), C5(H5), C20(H20), C21(H21), C22(H22), C23(H23),

C24(H24)

2.d Idealised Me refined as rotating group:

C9(H9A,H9B,H9C), C10(H10A,H10B,H10C), C11(H11A,H11B,H11C), C14(H14A,H14B,H14C)

6. Analytical data of HRMS

When 2.0 eq TEMPO or BHT was added to the above reaction mixture, the reaction

was inhibited. When 2.0 eq TEMPO or BHT was added to the reaction of **1a**, the reaction was monitored by mass spectrometry experiment. Byproducts **6** and **7** were observed by mass spectrometry, which corresponds to the products of the radical captured by TEMPO and BHT. So we think this is a radical mechanism.