

*Supporting Information*

**A formal intermolecular [4+1] cycloaddition reaction of  
3-chlorooxindole and o-quinone methides: facile synthesis of  
spirocyclic oxindoles**

Chao Lin<sup>\*a</sup>, Qi Xing<sup>a</sup>, Honglei Xie<sup>\*a</sup>

<sup>a</sup> Yantai Key Laboratory of Nanomedicine & Advanced Preparations,  
Yantai Institute of Materia Medica, Shandong 264000, China. e-mail:  
linchao46@163.com; qingteng51@163.com

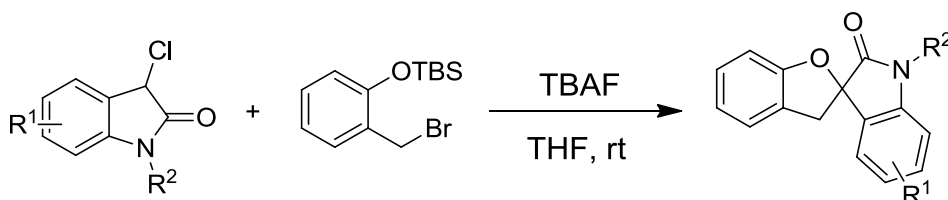
Table of contents

General Methods.....	S2
General procedure for intermolecular [4 + 1] cycloaddition reactions.....	S2
Copies of NMR spectra.....	S11

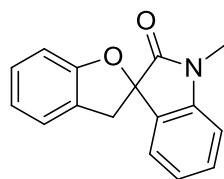
## General Methods.

Reagents and solvents were purchased from commercial sources and were used as received unless otherwise stated. Reactions were monitored by TLC visualized by UV (254 nm). Column chromatography was performed on silica gel (200-300 mesh).  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR (400 and 100 MHz, respectively) spectra were recorded in DMSO- $d_6$ .  $^1\text{H}$  NMR chemical shifts are reported in ppm relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal reference (DMSO- $d_6$  at 2.50 ppm).  $^{13}\text{C}$  NMR chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal reference (DMSO-  $d_6$  at 39.51 ppm).

### General procedure for intermolecular [4 + 1] cycloaddition reaction of 3-chlorooxindole and O-silylated phenols:



To a solution of 3-chlorooxindole **1** (0.3 mmol, 1.0 equiv.) and O-silylated phenol **2** (0.45 mmol, 1.5 equiv) in dry THF (3 mL) was added TBAF (1.0 M in THF, 1.2 mmol, 4.0 equiv) at room temperature. After the addition, the mixture was stirred at room temperature for 6 h. The reaction was concentrated under reduced pressure and purified by flash chromatography on silica gel (petroleum ether : ethyl acetate = 10 : 1).

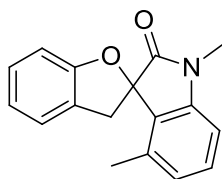


**3a**, yellow solid, 94% yield.

$^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  7.42 (td,  $J = 7.6, 1.2$  Hz, 1H), 7.37-7.34 (m, 1H), 7.32-7.29 (m, 1H), 7.21-7.15 (m, 1H), 7.12-7.05 (m, 2H), 6.94 (td,  $J = 7.6, 1.2$  Hz, 1H), 6.86-6.81 (m, 1H), 3.53 (dd,  $J = 26.8, 16.4$  Hz, 2H), 3.15 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz, DMSO- $D_6$ ):  $\delta$  174.12, 158.71, 143.66, 130.80, 128.69, 128.31, 126.21, 125.07, 123.94, 123.23, 121.13, 109.24, 84.94, 38.13, 26.16.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  252.1019, found 252.1022.

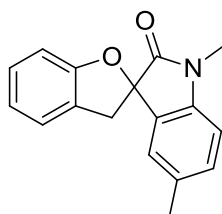


**3b**, yellow solid, 72% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.33-7.28 (m, 2H), 7.19 (t,  $J = 7.3$  Hz, 1H), 6.98-6.88 (m, 3H), 6.86 (d,  $J = 8.0$  Hz, 1H), 3.60-3.47 (m, 2H), 3.14 (s, 3H), 2.04 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.47, 158.98, 143.76, 135.45, 130.41, 128.46, 126.36, 125.92, 125.16, 124.92, 121.16, 108.94, 106.84, 85.19, 36.85, 26.22, 17.25.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  266.1176, found 226.1178.

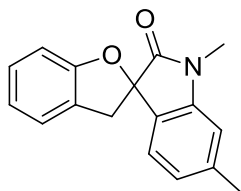


**3c**, yellow solid, 87% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.33-7.28 (m, 1H), 7.24-7.14 (m, 3H), 7.00-6.90 (m, 2H), 6.83 (d,  $J = 7.9$  Hz, 1H), 3.51 (dd,  $J = 31.2, 16.4$  Hz, 2H), 3.13 (s, 3H), 2.25 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.06, 158.70, 141.18, 132.42, 130.83, 128.79, 128.30, 126.12, 125.05, 124.47, 121.09, 109.19, 108.97, 85.02, 38.16, 26.17, 20.53.

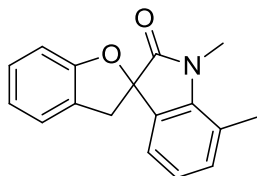
HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  266.1176, found 266.1179.



**3d**, yellow solid, 75% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.29 (d,  $J = 7.4$  Hz, 1H), 7.22 (d,  $J = 7.5$  Hz, 1H), 7.17 (t,  $J = 8.0$  Hz, 1H), 6.97-6.86 (m, 3H), 6.82 (d,  $J = 7.9$  Hz, 1H), 3.49 (dd,  $J = 31.2, J = 16.3$  Hz, 2H), 3.13 (s, 3H), 2.35 (s, 3H).

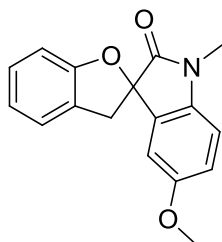
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ )  $\delta$  174.38, 158.72, 143.75, 140.78, 128.26, 126.23, 125.80, 125.01, 123.71, 123.52, 121.03, 109.94, 109.18, 84.91, 38.09, 26.10, 21.55.  
HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  266.1176, found 266.1186.



**3e**, yellow solid, 77% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.29 (d,  $J = 7.3$  Hz, 1H), 7.22-7.13 (m, 3H), 7.01-6.90 (m, 2H), 6.83 (d,  $J = 8.0$  Hz, 1H), 3.57-3.43 (m, 2H), 3.41 (s, 3H), 2.56 (s, 3H).

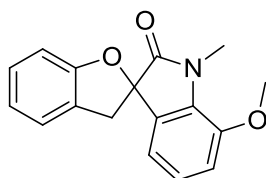
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ )  $\delta$  174.79, 158.73, 141.16, 134.22, 129.45, 128.29, 126.18, 125.04, 123.23, 121.77, 121.07, 120.57, 109.19, 84.42, 38.54, 29.17, 18.39.  
HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  266.1176, found 266.1180.



**3f**, yellow solid, 81% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.30 (d,  $J = 7.3$  Hz, 1H), 7.18 (t,  $J = 8.2$  Hz, 1H), 7.05-6.91 (m, 4H), 6.84 (d,  $J = 8.0$  Hz, 1H), 3.71 (s, 3H), 3.54 (s, 2H), 3.12 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ )  $\delta$  173.93, 158.77, 156.09, 136.91, 129.63, 128.26, 126.32, 125.03, 121.09, 115.50, 110.90, 109.84, 109.22, 85.28, 55.74, 38.12, 26.19.  
HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  282.1125, found 282.1125.



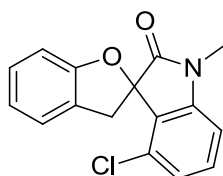
**3g**, yellow solid, 63% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.30 (dd,  $J = 7.2, 1.2$  Hz, 1H), 7.20-7.15 (m, 1H),

7.15-7.12 (m, 1H), 7.08-7.01 (m, 1H), 6.96-6.91 (m, 2H), 6.83 (d,  $J = 7.7$  Hz, 1H), 3.86 (s, 3H), 3.49 (dd,  $J = 40.0, 16.4$  Hz, 2H), 3.36 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.43, 158.69, 145.13, 130.89, 130.20, 128.32, 126.12, 125.06, 124.26, 121.13, 116.43, 115.36, 109.22, 84.87, 56.51, 38.49, 29.35.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  282.1125, found 282.1128.

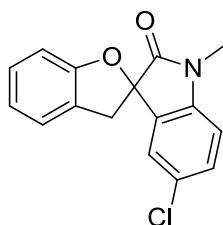


**3h**, yellow solid, 82% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.46 (t,  $J = 8.0$  Hz, 1H), 7.31 (dd,  $J = 7.6, 0.8$  Hz, 1H), 7.20-7.15 (m, 1H), 7.15-7.09 (m, 2H), 6.94 (td,  $J = 7.6, 1.2$  Hz, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.60 (dd,  $J = 56.5, 16.5$  Hz, 2H), 3.17 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.76, 158.95, 145.74, 132.40, 130.47, 128.27, 126.01, 125.17, 124.73, 123.58, 121.14, 108.92, 108.38, 84.73, 36.34, 26.51.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  286.0629, found 286.0631.

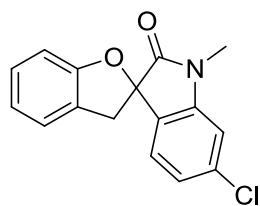


**3i**, yellow solid, 74% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.55-7.43 (m, 2H), 7.36-7.27 (m, 1H), 7.19 (t,  $J = 7.4$  Hz, 1H), 7.12 (d,  $J = 8.3$  Hz, 1H), 6.95 (t,  $J = 7.2$  Hz, 1H), 6.85 (d,  $J = 7.8$  Hz, 1H), 3.62-3.51, (m, 2H) 3.15, (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.79, 158.51, 142.64, 130.53, 130.41, 128.32, 127.17, 126.12, 125.09, 124.27, 121.28, 110.84, 109.27, 84.74, 37.96, 26.32.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  286.0629, found 286.0626.

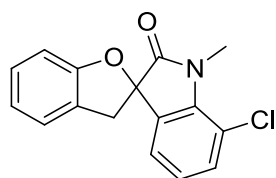


**3j**, yellow solid, 71% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.45-7.35 (m, 1H), 7.31 (d,  $J = 7.2$  Hz, 1H), 7.26 (d,  $J = 1.7$  Hz, 1H), 7.18 (t,  $J = 7.7$  Hz, 1H), 7.12 (dd,  $J = 7.9, 1.8$  Hz, 1H), 6.95 (t,  $J = 7.4$  Hz, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.59-3.47 (m, 2H), 3.15 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.14, 158.54, 145.30, 135.26, 128.34, 127.40, 126.06, 125.41, 125.08, 122.75, 121.24, 109.86, 109.27, 84.46, 38.02, 26.39.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  286.0629, found 286.0631.

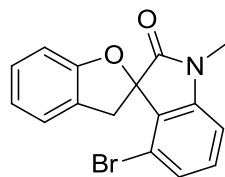


**3k**, yellow solid, 74% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.42 (d,  $J = 8.1$  Hz, 1H), 7.36-7.28 (m, 2H), 7.22-7.15 (m, 1H), 7.08 (t,  $J = 7.8$  Hz, 1H), 6.95 (t,  $J = 7.4$  Hz, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.62-3.47 (m, 2H), 3.46 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.54, 158.50, 139.37, 132.67, 131.81, 128.40, 126.00, 125.11, 124.66, 123.13, 121.33, 114.84, 109.29, 84.27, 38.57, 29.41.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  286.0629, found 286.0633.

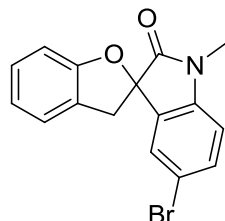


**3l**, yellow solid, 76% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.38 (t,  $J = 8.0$  Hz, 1H), 7.32-7.27 (m, 2H), 7.20-7.11 (m, 2H), 6.93 (td,  $J = 7.5, 0.8$  Hz, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.59 (dd,  $J = 77.6, 16.8$  Hz, 2H), 3.15 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.75, 159.18, 146.00, 132.51, 128.25, 126.76, 126.62, 126.15, 124.69, 121.10, 118.95, 108.92, 108.76, 85.27, 36.31, 26.40.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  330.0124, found 330.0126.

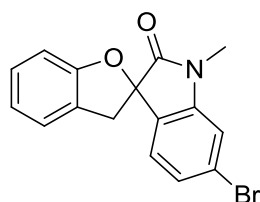


**3m**, yellow solid, 64% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.65-7.60 (m, 1H), 7.55 (d,  $J = 2.0$  Hz, 1H), 7.31 (d,  $J = 7.3$  Hz, 1H), 7.19 (t,  $J = 7.6$  Hz, 1H), 7.08 (d,  $J = 8.4$  Hz, 1H), 6.95 (t,  $J = 7.4$  Hz, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.62-3.50 (m, 2H), 3.15-3.13 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.69, 158.49, 143.07, 133.41, 130.78, 128.33, 126.91, 126.13, 125.11, 121.29, 114.78, 111.36, 109.28, 84.70, 37.96, 26.31.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  330.0124, found 330.0126.

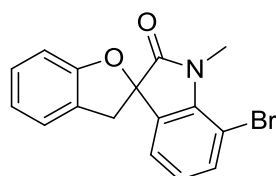


**3n**, yellow solid, 74% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.38 (d,  $J = 1.6$  Hz, 1H), 7.33-7.29 (m, 2H), 7.26 (dd,  $J = 7.9, 1.7$  Hz, 1H), 7.21-7.15 (m, 1H), 6.94 (td,  $J = 7.4, 1.0$  Hz, 1H), 6.84 (d,  $J = 7.6$  Hz, 1H), 3.59-3.46 (m, 2H), 3.15 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.03, 158.54, 145.34, 128.34, 127.86, 126.04, 125.69, 125.08, 123.64, 121.25, 112.55, 109.27, 84.51, 38.00, 26.38.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  330.0124, found 330.0125.

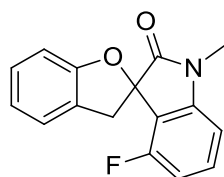


**3o**, yellow oil, 62% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.57 (d,  $J = 8.2$  Hz, 1H), 7.37 (d,  $J = 7.3$  Hz, 1H), 7.31 (d,  $J = 7.3$  Hz, 1H), 7.19 (t,  $J = 7.7$  Hz, 1H), 7.05-6.98 (m, 1H), 6.95 (t,  $J = 7.3$  Hz, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.62-3.49 (m, 2H), 3.47 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.78, 159.19, 146.02, 132.54, 128.28, 126.65, 126.17, 124.72, 121.13, 118.98, 108.95, 108.79, 85.29, 36.32, 26.43.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  330.0051, found 328.0121.



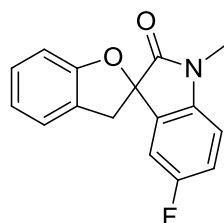
**3p**, yellow oil, 63% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.53-7.44 (m, 1H), 7.34-7.28 (m, 1H), 7.22-7.10 (m, 1H), 7.01-6.87 (m, 3H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.58 (s, 2H), 3.17 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.68, 159.95, 158.39, 157.47, 145.80, 133.16, 128.31, 125.82, 124.94, 121.27, 114.36, 110.69, 109.13, 106.00, 83.94, 37.03, 26.73.

$^{19}\text{F}$  NMR (377 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  -117.60 (dd,  $J = 9.4, 5.6$  Hz).

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  270.0925, found 270.0932.



**3q**, yellow solid, 71% yield.

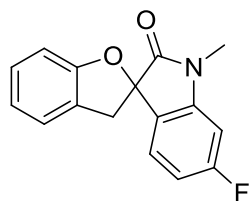
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.37-7.23 (m, 3H), 7.18 (t,  $J = 7.7$  Hz, 1H), 7.14-7.06 (m, 1H), 6.95 (t,  $J = 7.4$  Hz, 1H), 6.85 (d,  $J = 7.9$  Hz, 1H), 3.55 (s, 2H), 3.14 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.04, 158.62, 140.00, 128.34, 126.19, 125.10, 121.27, 116.88, 112.17, 110.37, 109.30, 84.96, 77.27, 38.04, 26.34.

$^{19}\text{F}$  NMR (377 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  -119.76 - -119.81 (m).

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  270.0925, found 270.0932.





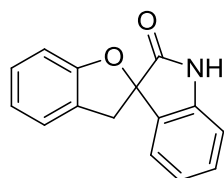
**3r**, yellow solid, 87% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.41 (dd,  $J = 8.0, 5.2$  Hz, 1H), 7.31 (dd,  $J = 7.6, 0.8$  Hz, 1H), 7.21-7.14 (m, 1H), 7.09 (dd,  $J = 9.5, 2.3$  Hz, 1H), 6.94 (td,  $J = 7.4, 0.9$  Hz, 1H), 6.90-6.79 (m, 2H), 3.58-3.49 (m, 2H), 3.15 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  174.47, 165.15, 162.71, 158.57, 145.92, 128.31, 126.17, 125.06, 121.18, 109.24, 108.91, 98.38, 98.10, 84.51, 38.00, 26.42.

$^{19}\text{F}$  NMR (377 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  -108.87 (td,  $J = 9.4, 5.6$  Hz).

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  270.0925, found 270.0926.

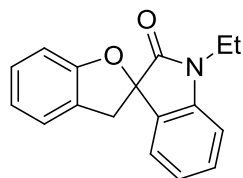


**3s**, yellow solid, 65% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  10.57 (s, 1H), 7.34-7.26 (m, 3H), 7.17 (t,  $J = 7.7$  Hz, 1H), 7.00 (t,  $J = 7.5$  Hz, 1H), 6.96-6.87 (m, 2H), 6.84 (d,  $J = 8.0$  Hz, 1H), 3.60-3.42 (m, 2H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  175.97, 158.79, 142.17, 130.70, 129.36, 128.27, 126.23, 125.03, 124.35, 122.60, 121.04, 110.27, 109.20, 85.26, 38.14.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  238.0863, found 238.0870.



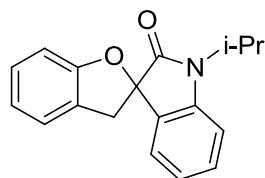
**3t**, yellow solid, 75% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.41 (td,  $J = 7.8, 1.2$  Hz, 1H), 7.36 (d,  $J = 7.4$  Hz, 1H), 7.30 (d,  $J = 7.9$  Hz, 1H), 7.21-7.12 (m, 2H), 7.08 (td,  $J = 8.2, 7.6, 0.8$  Hz, 1H),

6.97-6.91 (m, 1H), 6.85 (d,  $J = 7.9$  Hz, 1H), 3.78-3.65 (m, 2H), 3.60-3.45 (dd,  $J = 16.4, 12.0$  Hz, 2H), 1.18 (t,  $J = 7.2$  Hz, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ )  $\delta$  173.82, 158.73, 142.62, 130.86, 128.88, 128.34, 126.21, 125.09, 124.22, 123.17, 121.16, 109.33, 109.28, 84.94, 38.14, 34.33, 12.48.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  266.1176, found 266.1180.

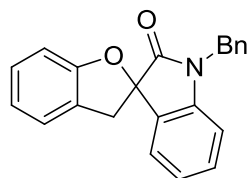


**3u**, yellow solid, 61% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.43-7.22 (m, 2H), 7.30 (d,  $J = 6.8$  Hz, 1H), 7.25 (d,  $J = 7.9$  Hz, 1H), 7.18 (t,  $J = 7.7$  Hz, 1H), 7.09-7.03 (m, 1H), 6.97-6.90 (m, 1H), 6.85 (d,  $J = 7.9$  Hz, 1H), 4.55-4.42 (m, 1H), 3.59-3.44 (m, 2H), 1.43 (d,  $J = 6.9$  Hz, 6H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ ):  $\delta$  173.91, 158.75, 142.25, 130.72, 129.09, 128.29, 126.22, 125.04, 124.33, 122.85, 121.10, 110.44, 109.23, 84.76, 43.67, 38.25, 19.02, 18.93.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  280.1332, found 280.1339.

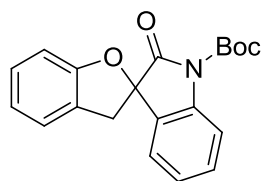


**3v**, yellow solid, 61% yield.

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.43-7.26 (m, 8H), 7.20 (t,  $J = 7.7$  Hz, 1H), 7.06 (t,  $J = 7.5$  Hz, 1H), 7.02-6.93 (m, 2H), 6.89 (d,  $J = 7.9$  Hz, 1H), 4.99-4.83 (m, 2H), 3.61 (dd,  $J = 34.4, 16.4$  Hz, 2H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ )  $\delta$  174.42, 158.70, 142.58, 136.00, 130.72, 128.84, 128.72, 128.37, 127.64, 127.30, 126.15, 125.11, 124.22, 123.43, 121.23, 109.85, 109.33, 84.99, 42.81, 38.33.

HRMS (ESI):  $[\text{M}+\text{H}]^+$   $m/z$  328.1332, found 328.1333.

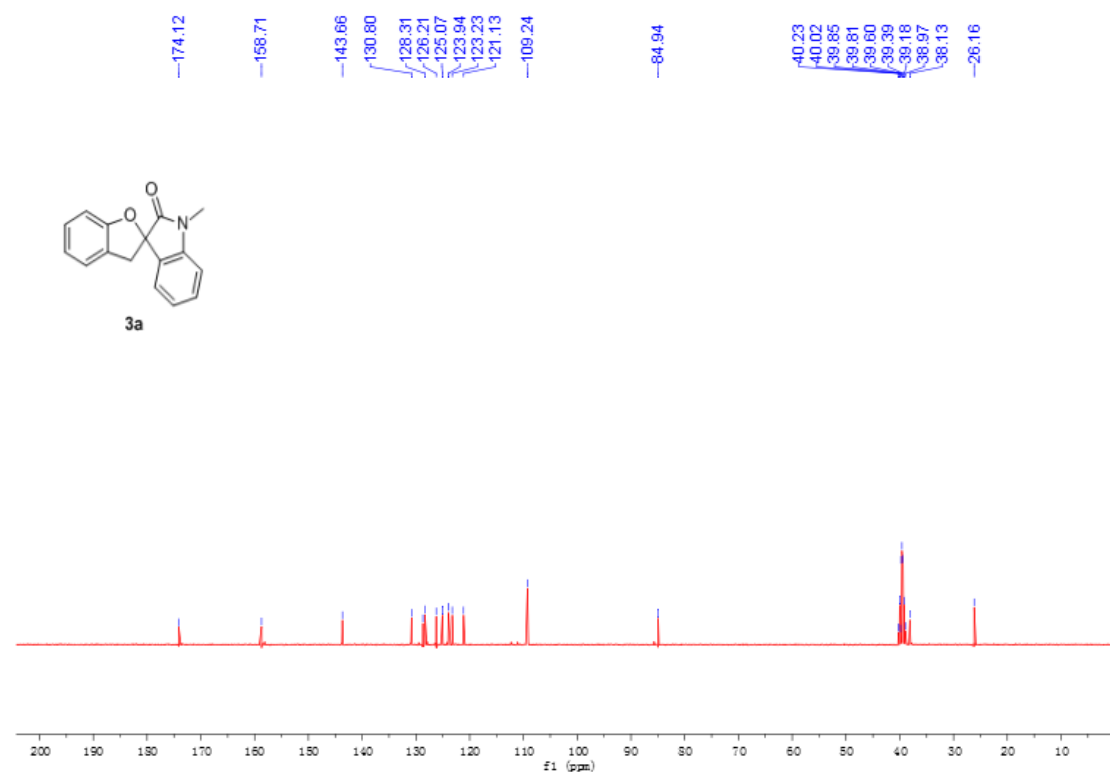
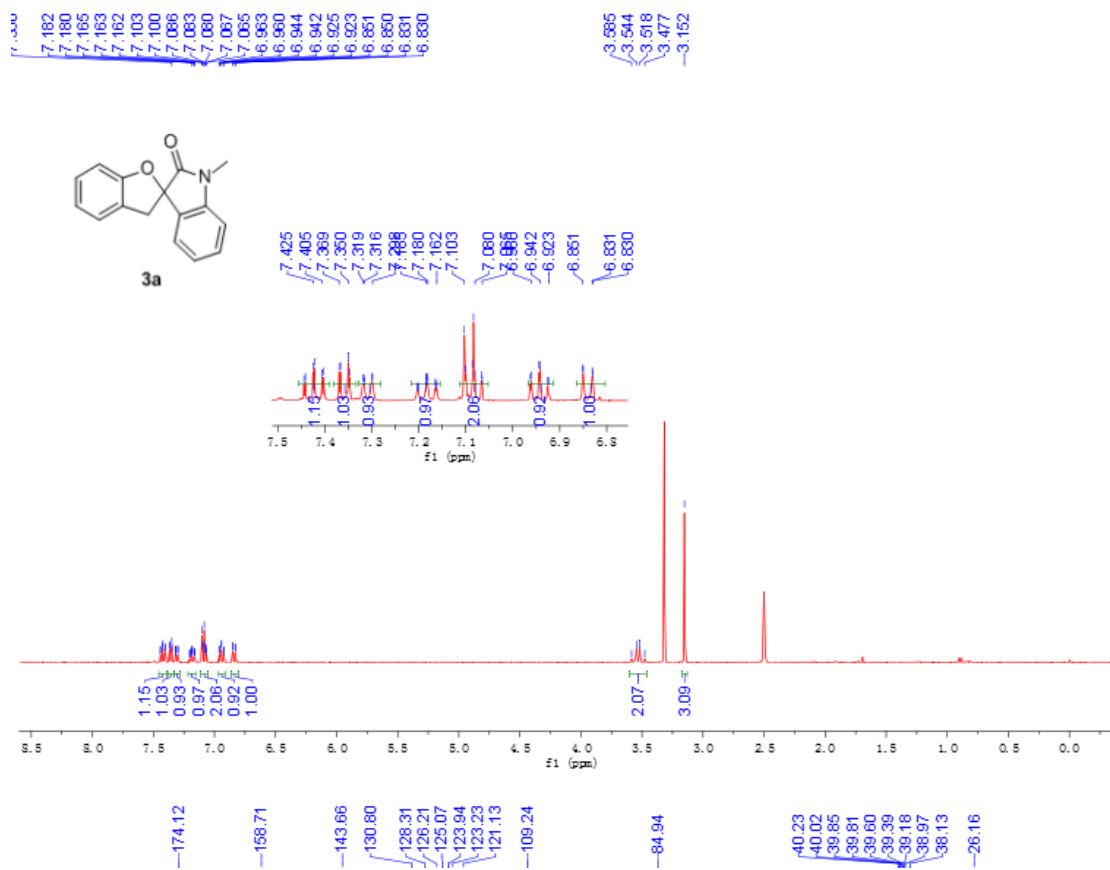


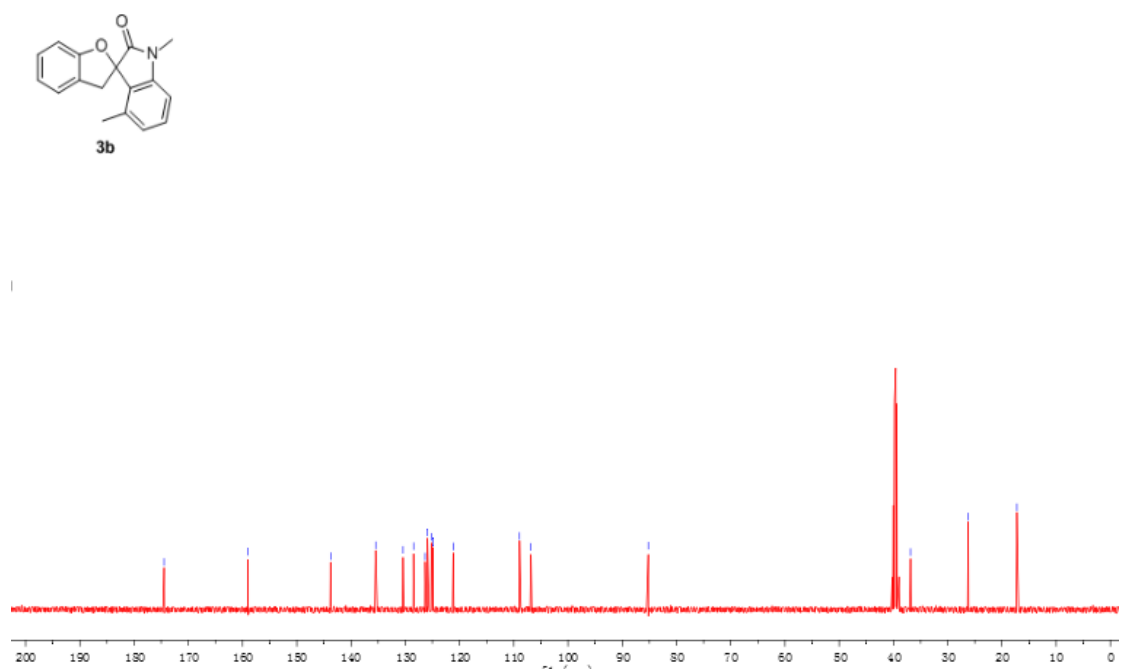
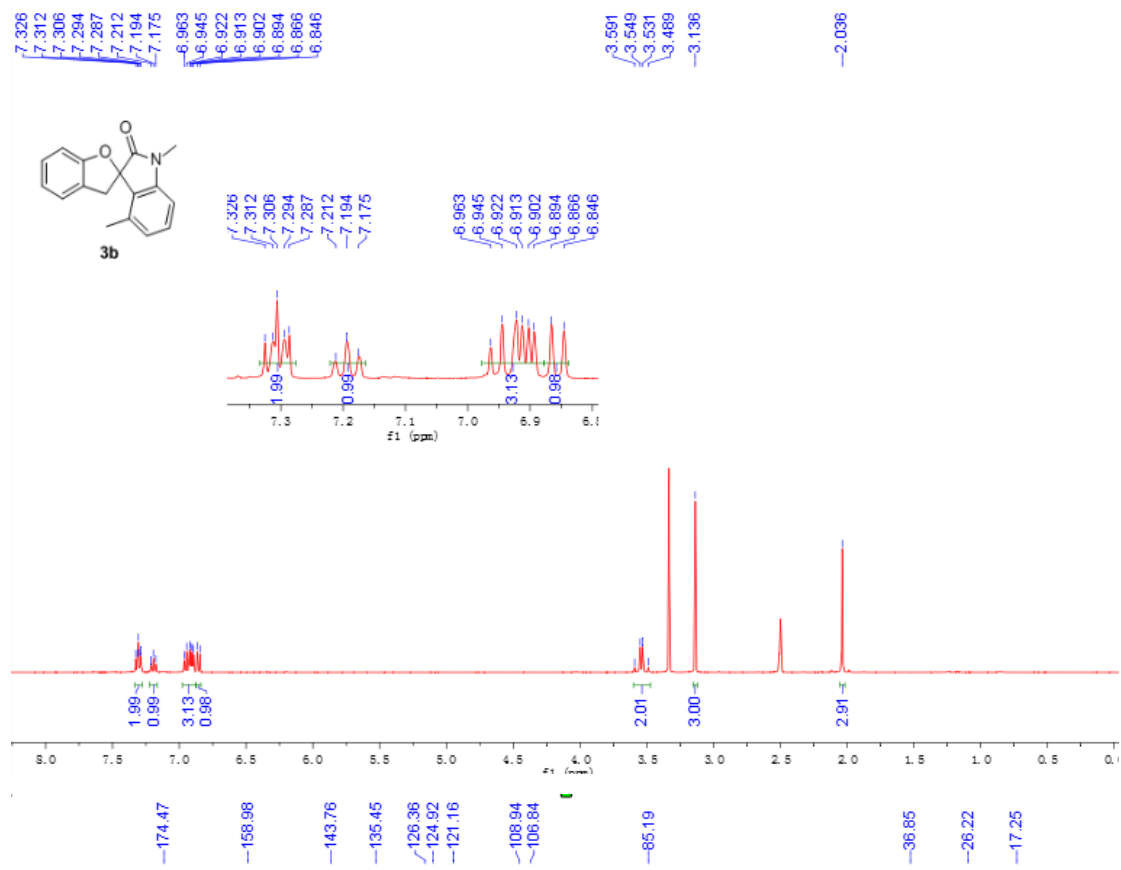
**3w**, yellow solid, 60% yield.

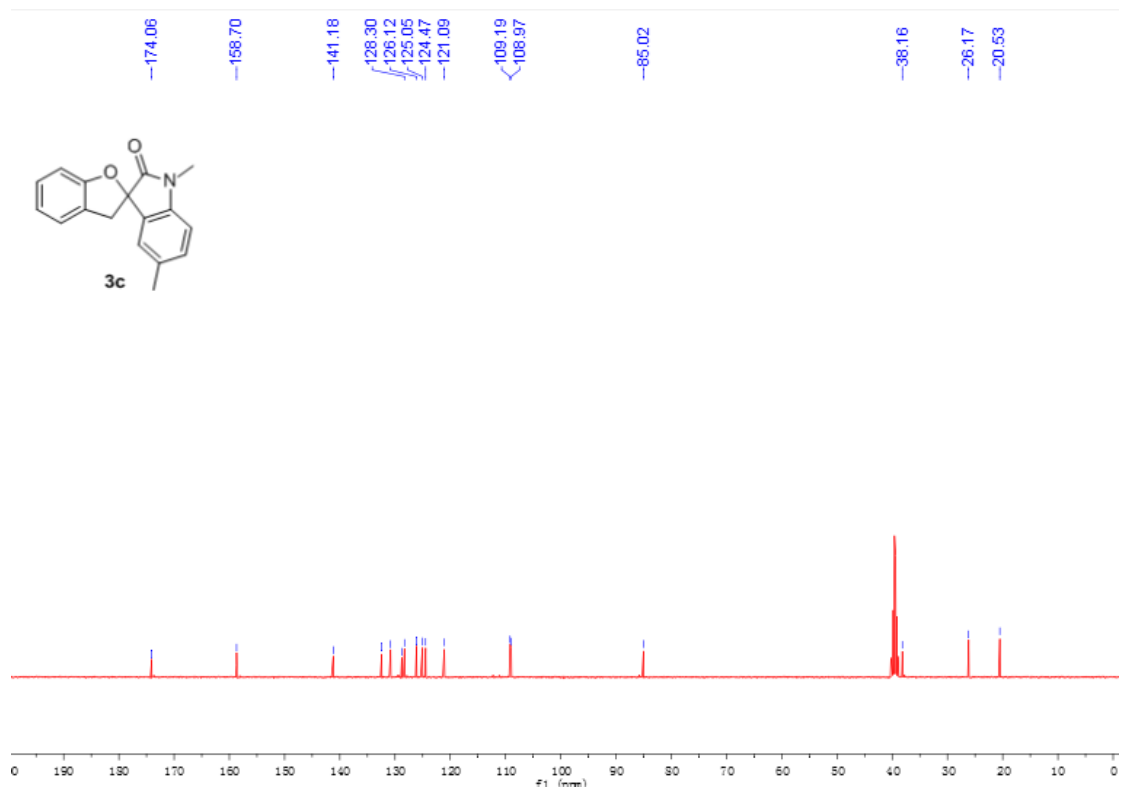
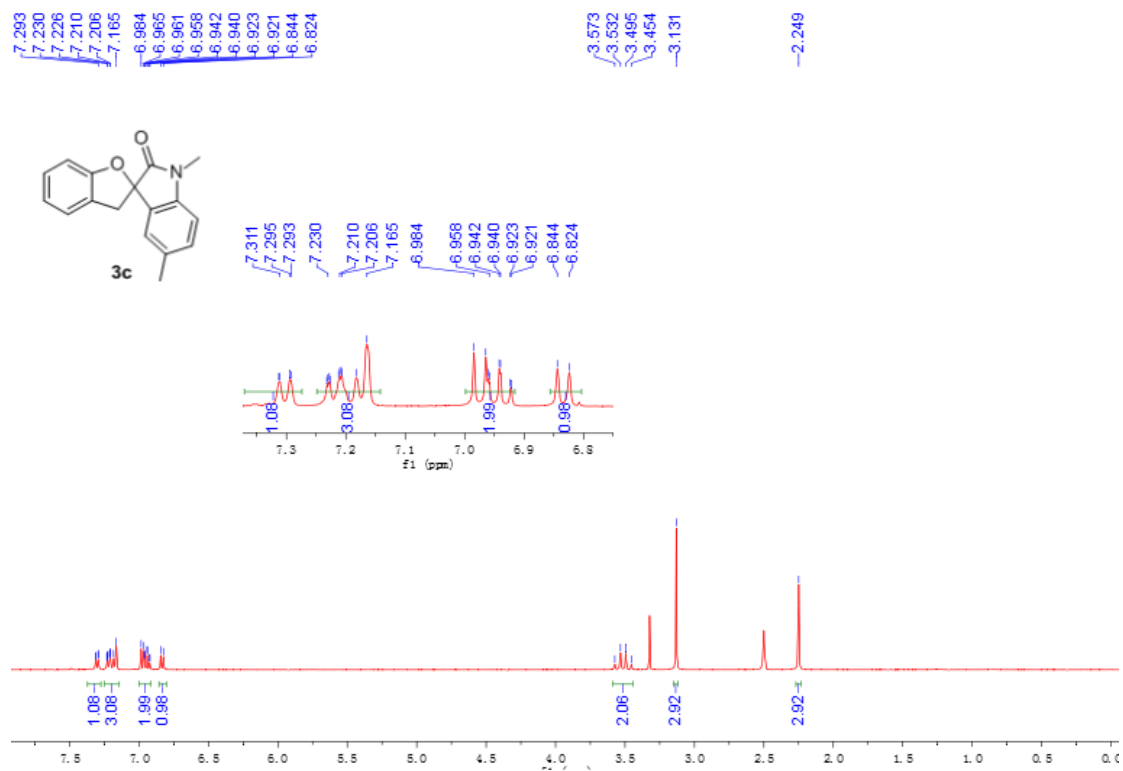
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  7.82 (d,  $J = 8.0$  Hz, 1H), 7.53-7.46 (m, 2H), 7.31 (d,  $J = 6.2$  Hz, 1H), 7.28-7.23 (m, 1H), 7.22-7.17 (m, 1H), 6.99-6.93 (m, 1H), 6.85 (d,  $J = 8.0$  Hz, 1H), 3.76-3.48 (m, 2H), 1.57 (s, 9H).

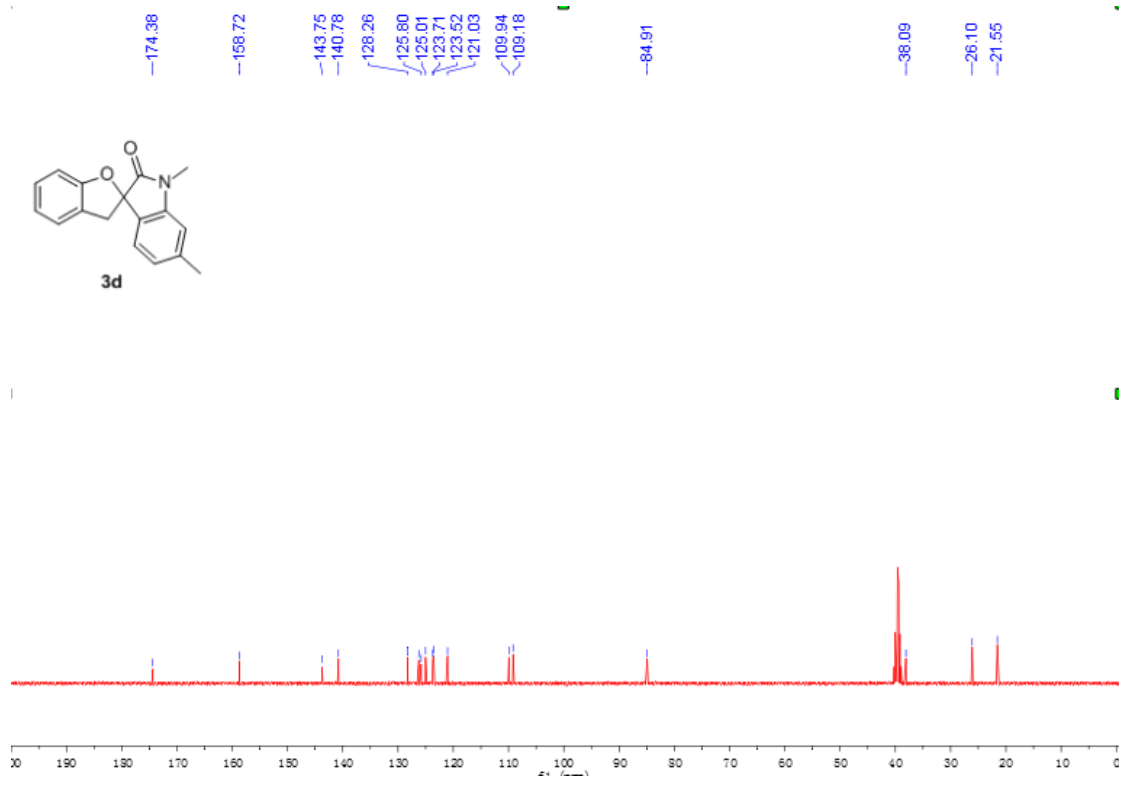
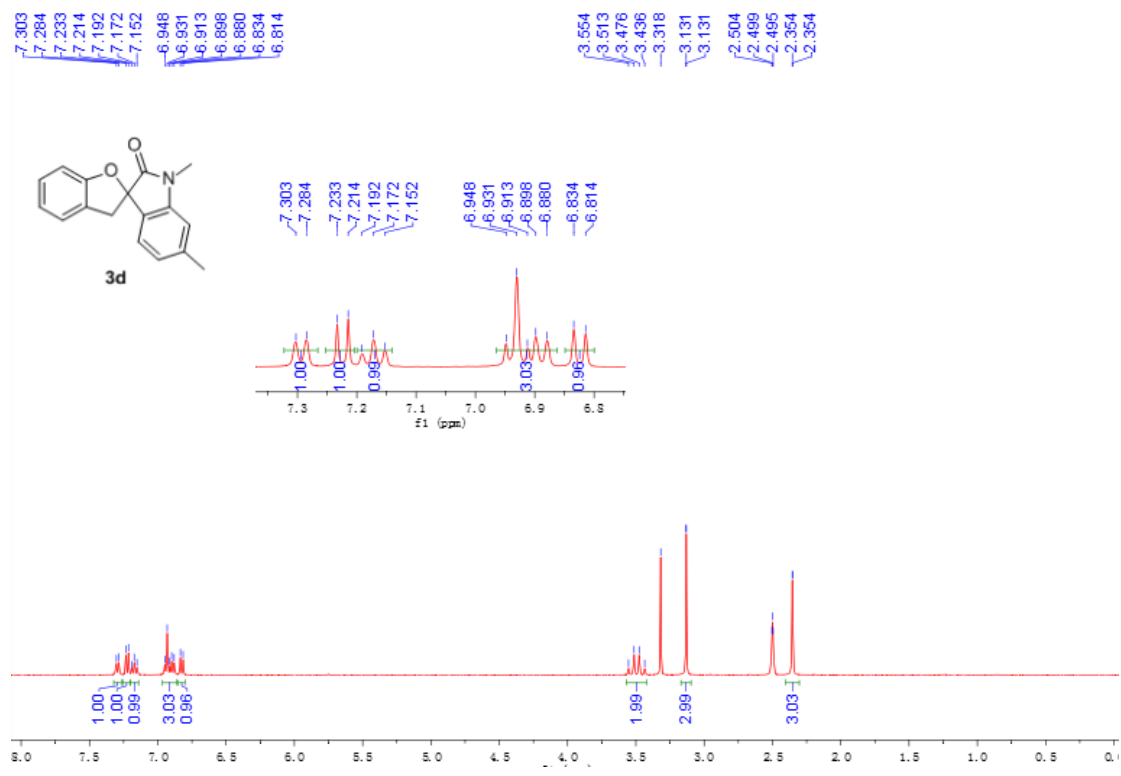
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}D_6$ )  $\delta$  173.76, 158.95, 145.74, 132.40, 130.47, 128.27, 126.01, 125.17, 124.73, 123.58, 121.14, 108.92, 108.38, 84.73, 36.34, 26.51.

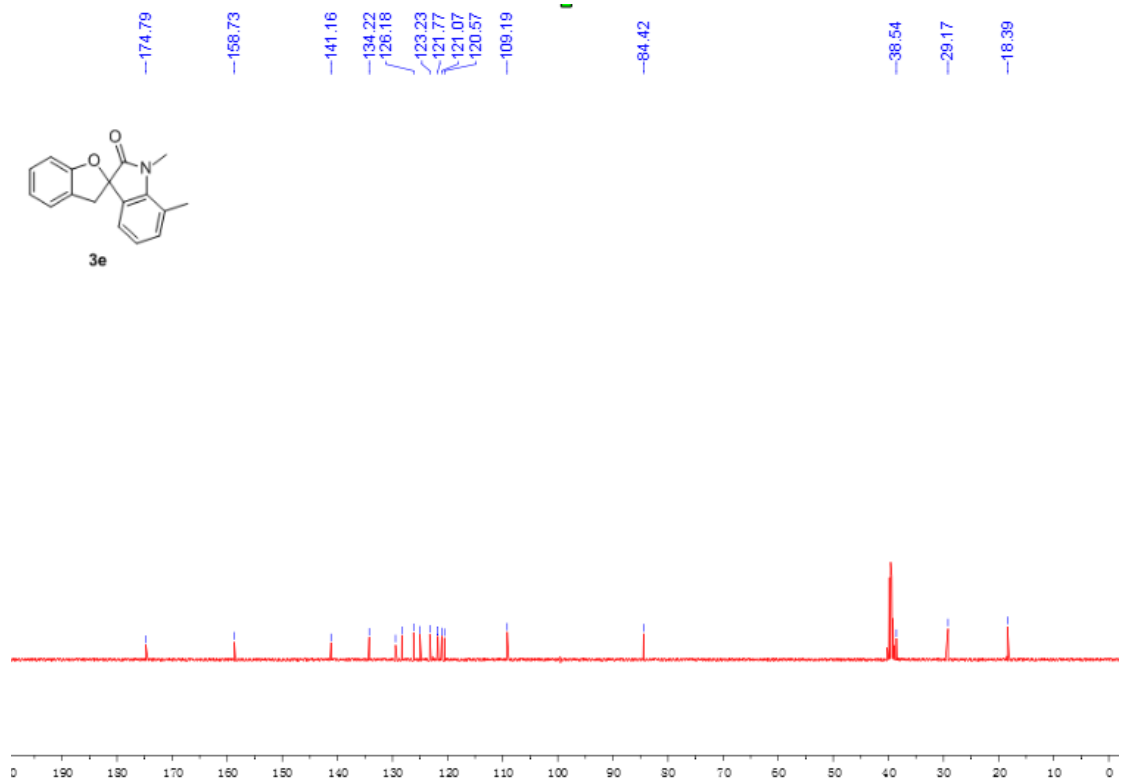
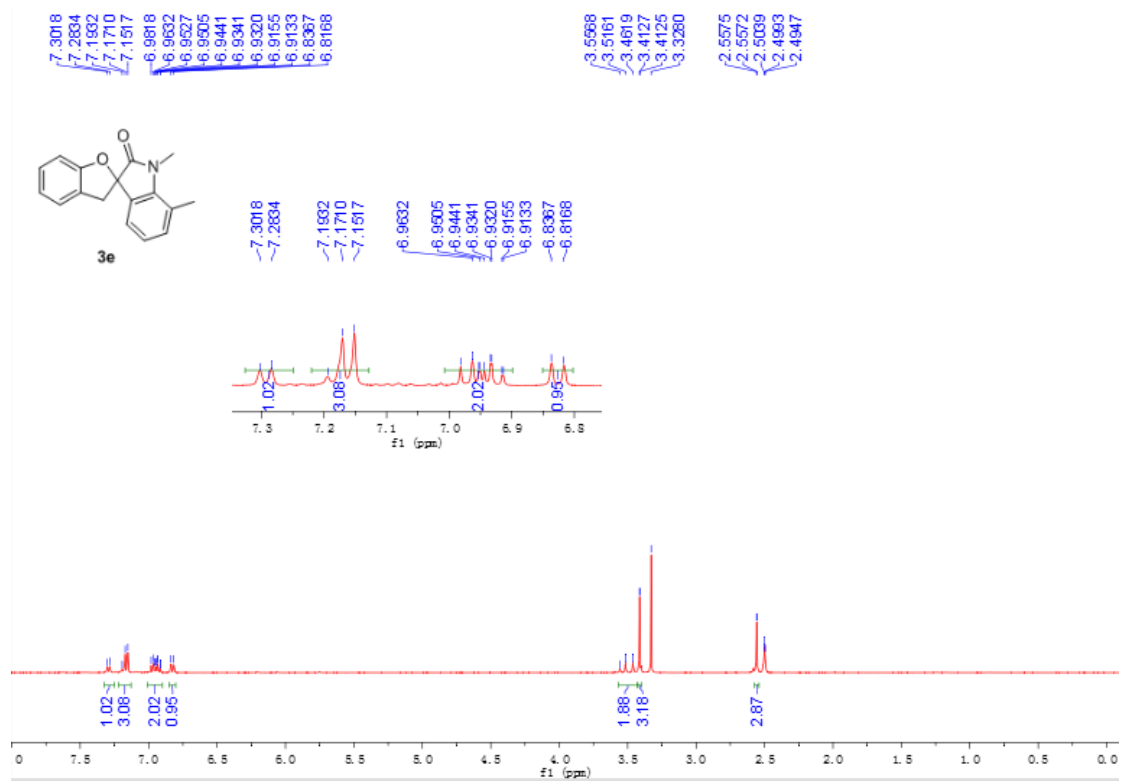
HRMS (ESI):  $[2\text{M}+\text{Na}]^+$   $m/z$  697.2526, found 697.2531.



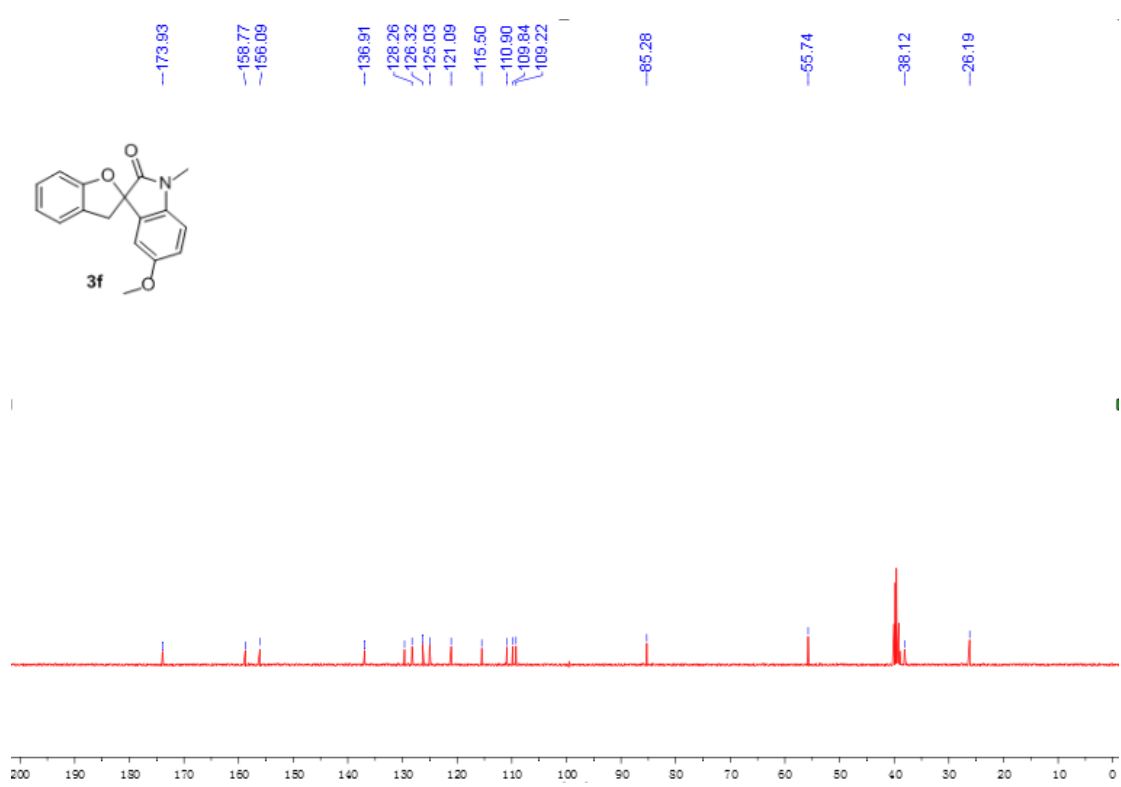
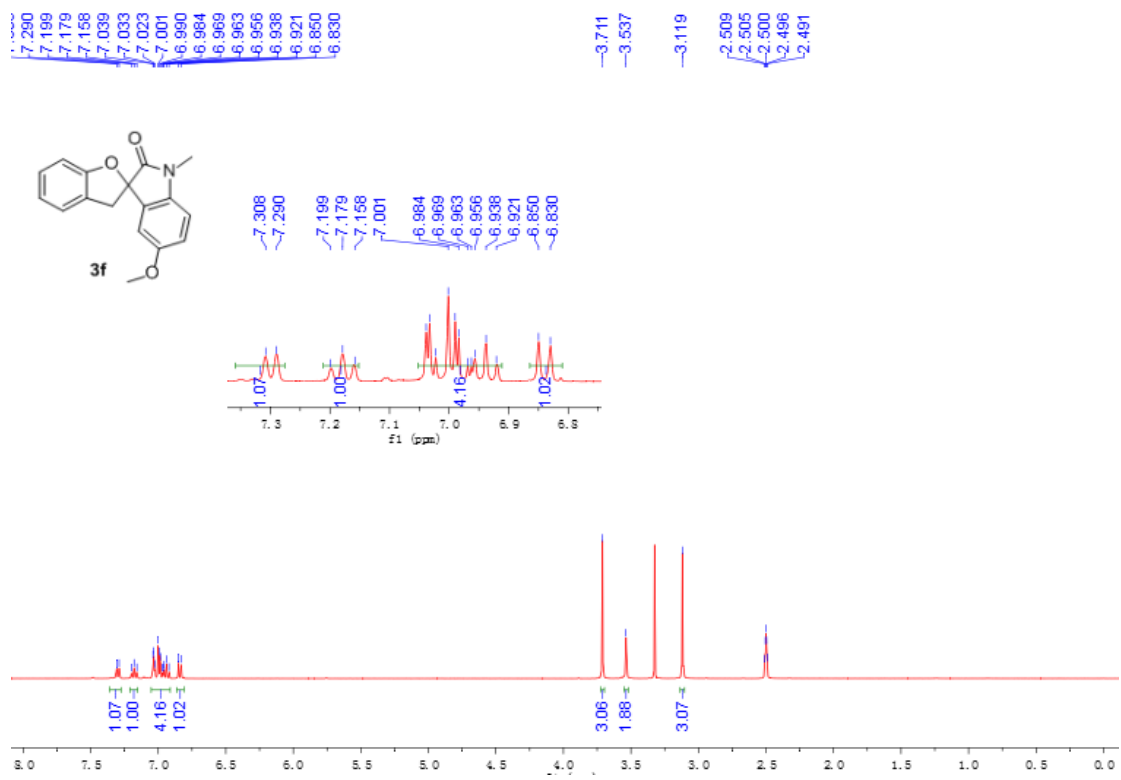


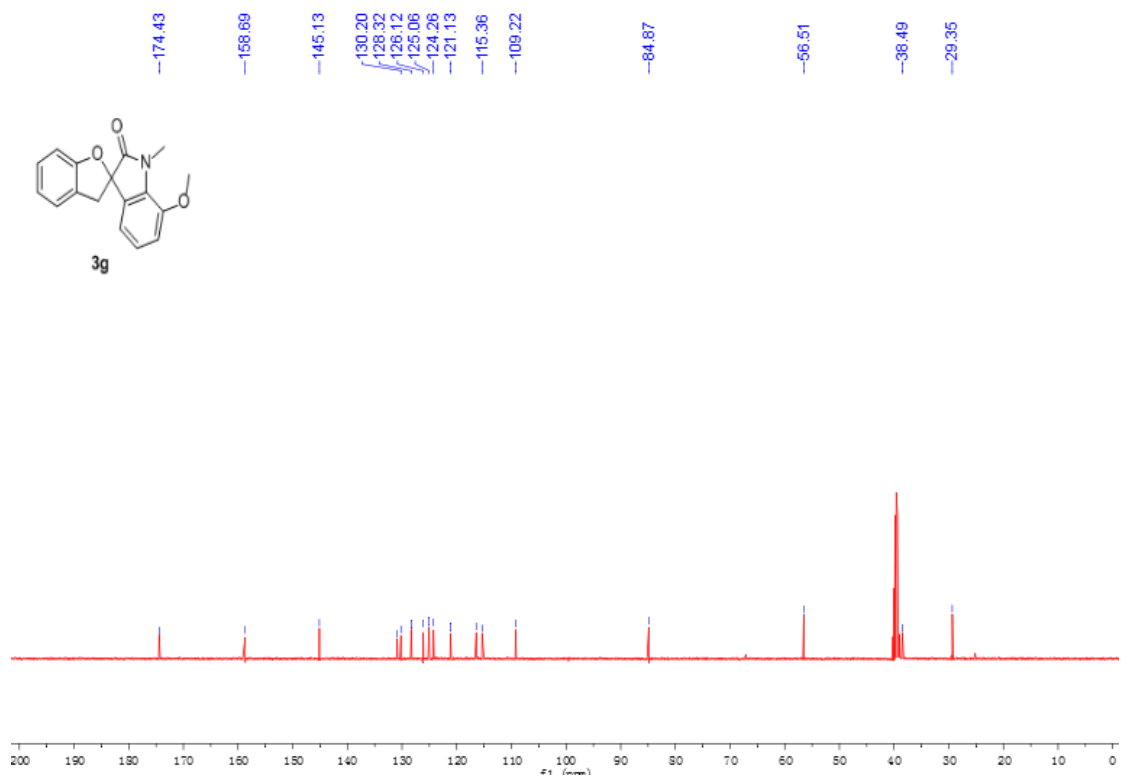
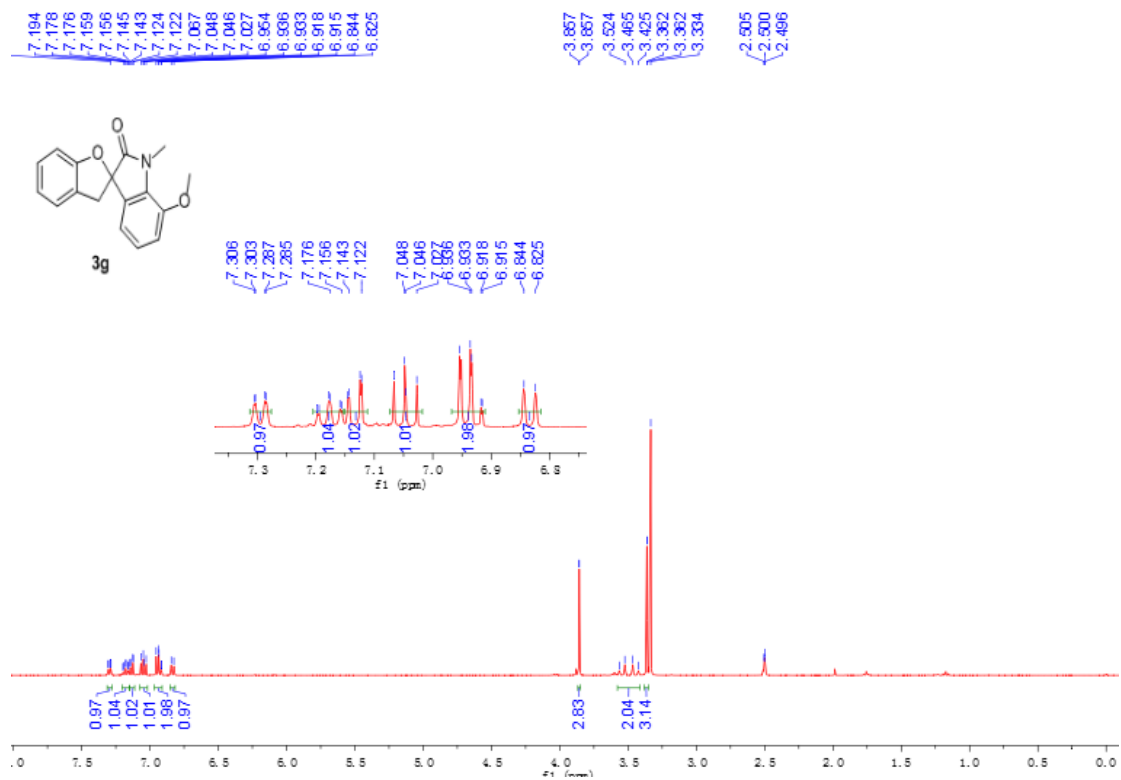


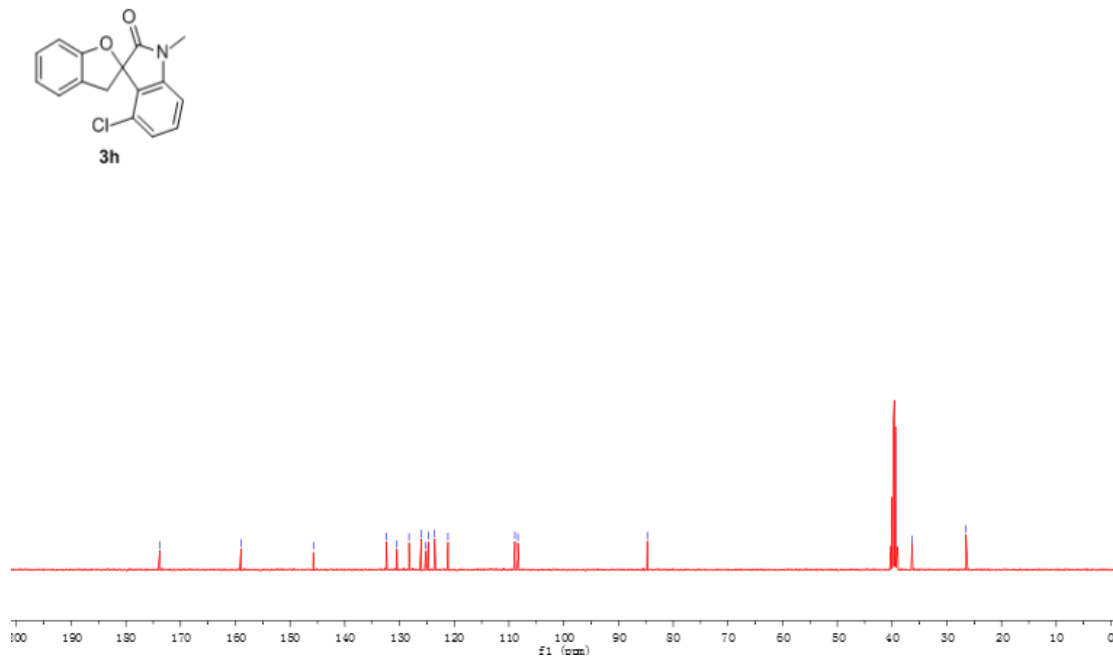
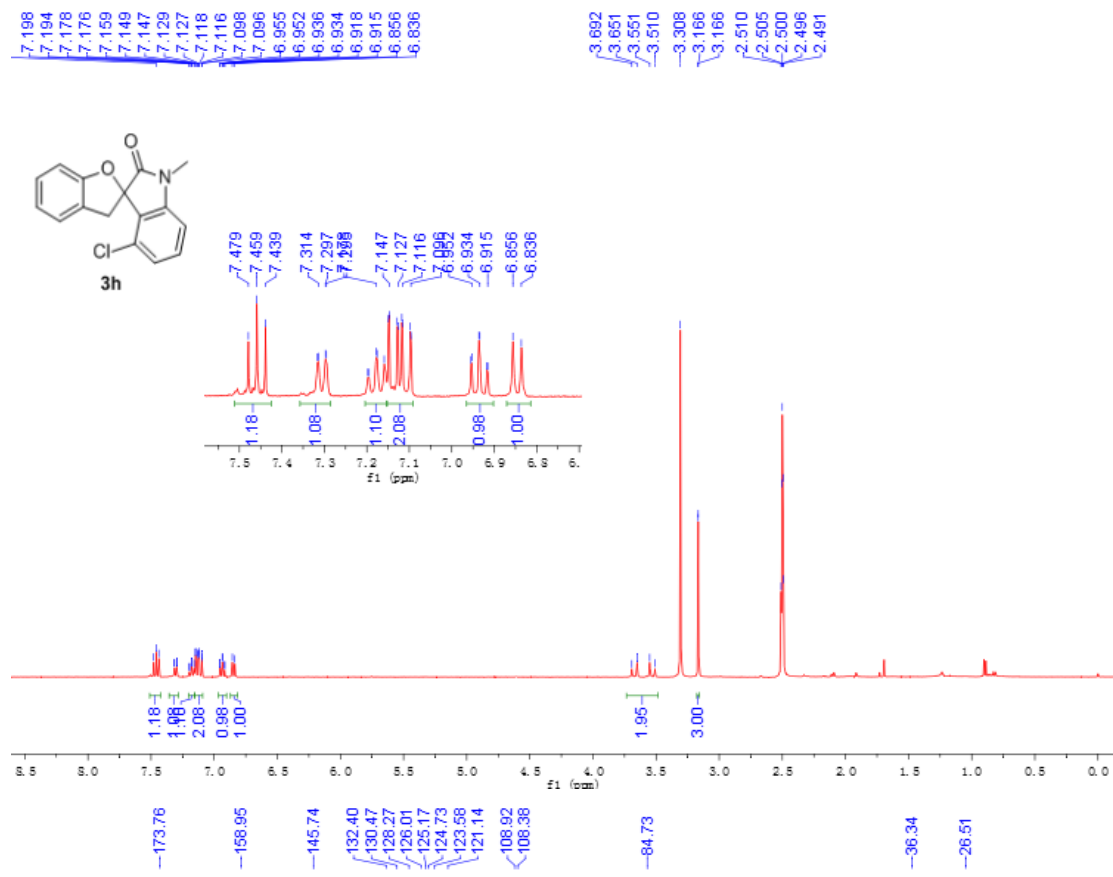


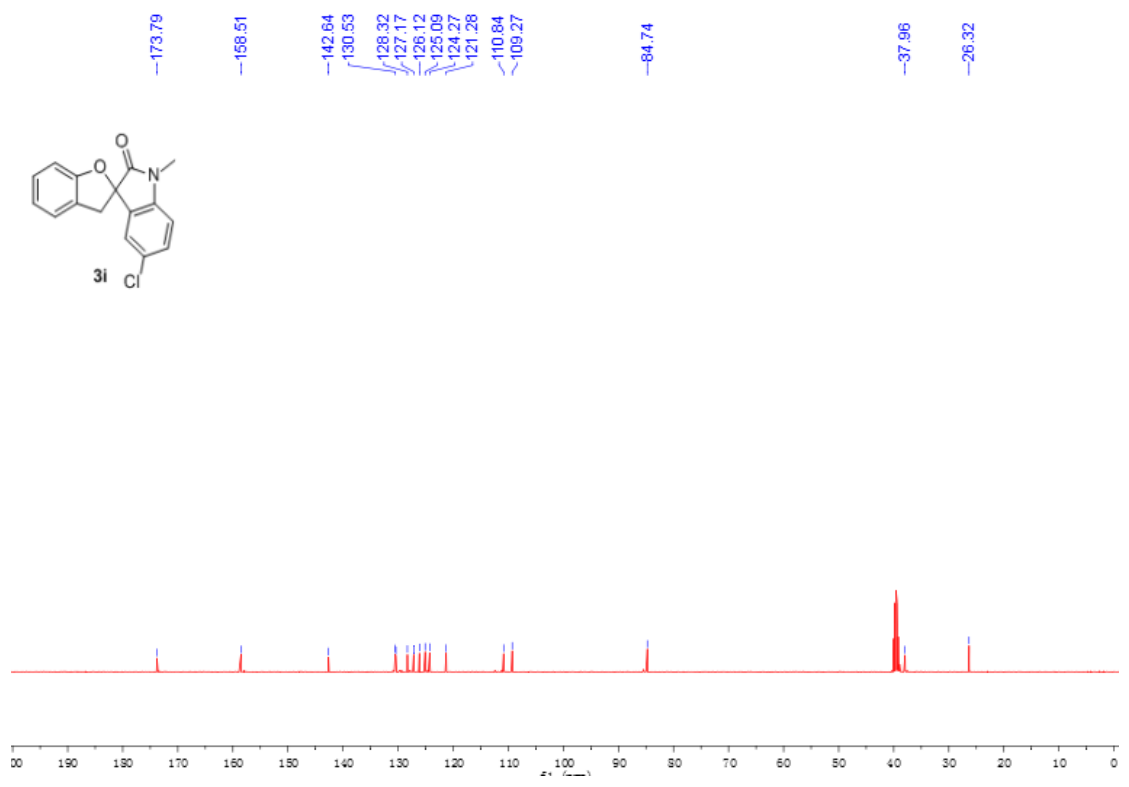
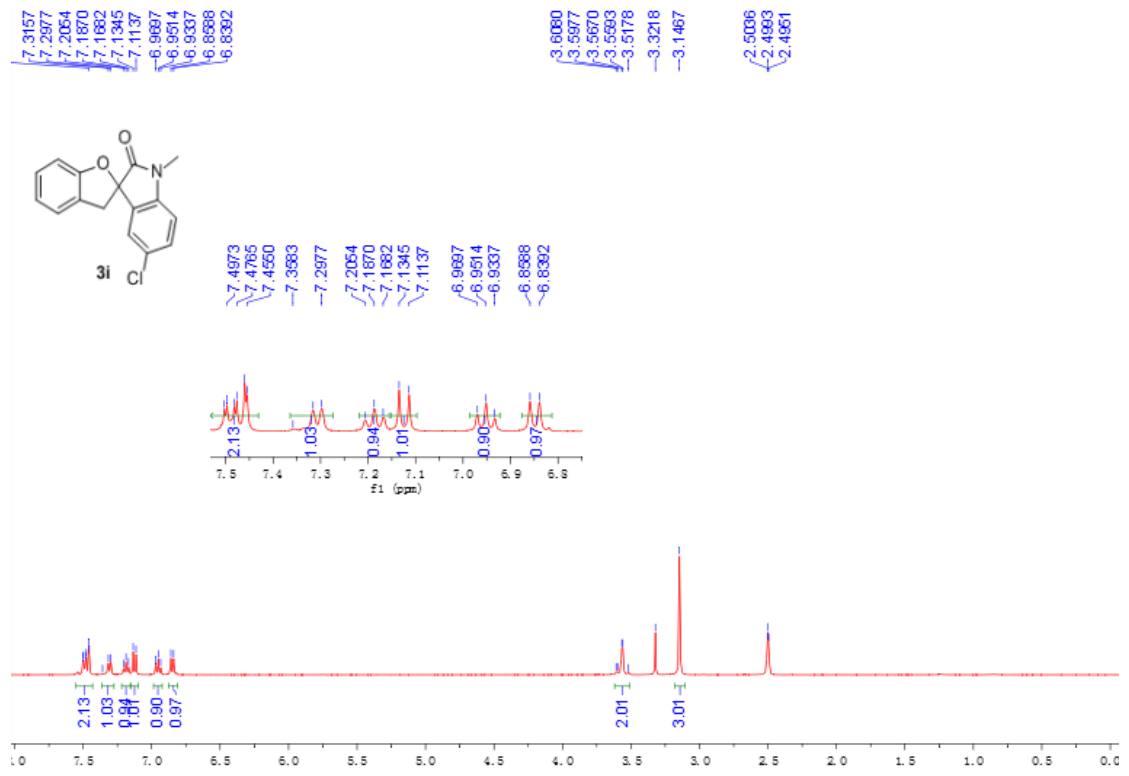


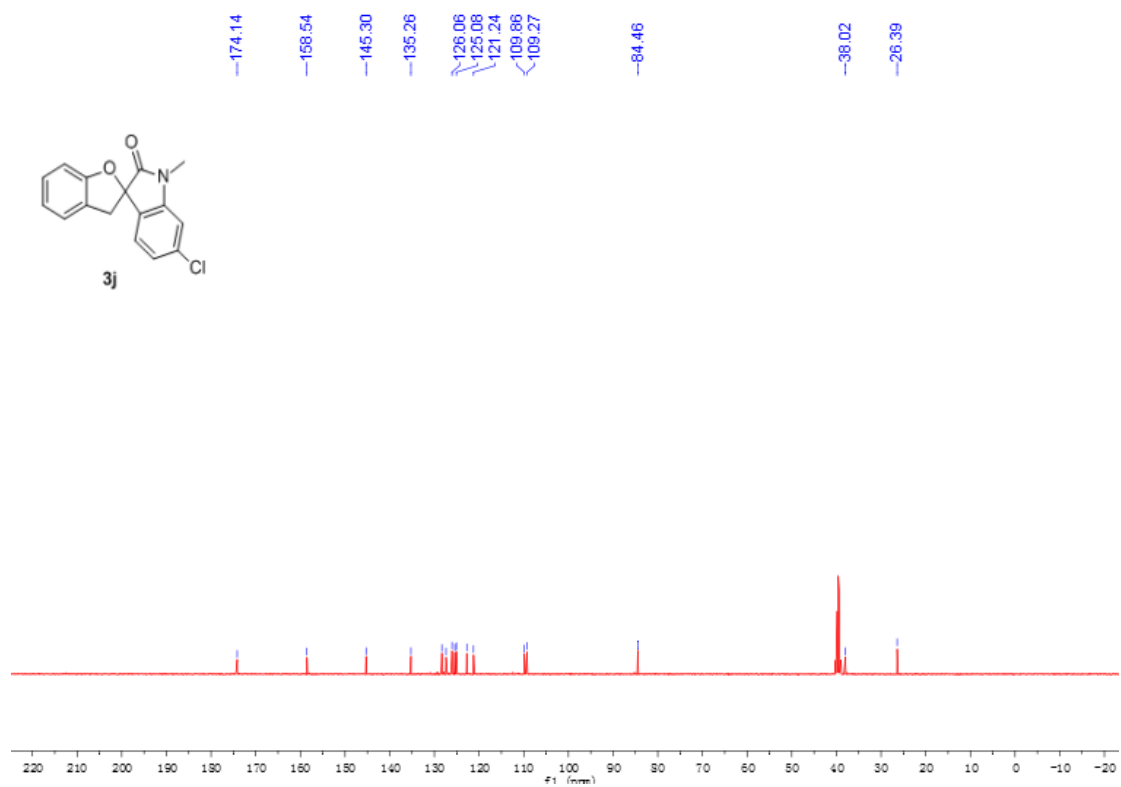
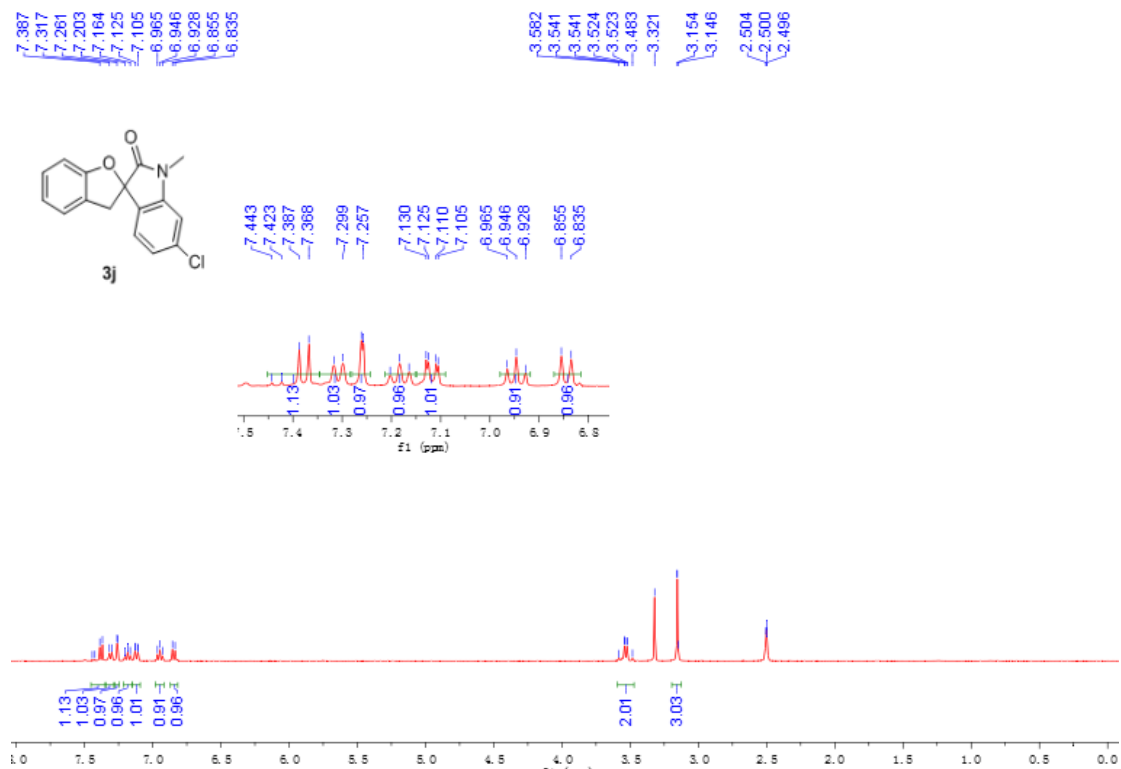


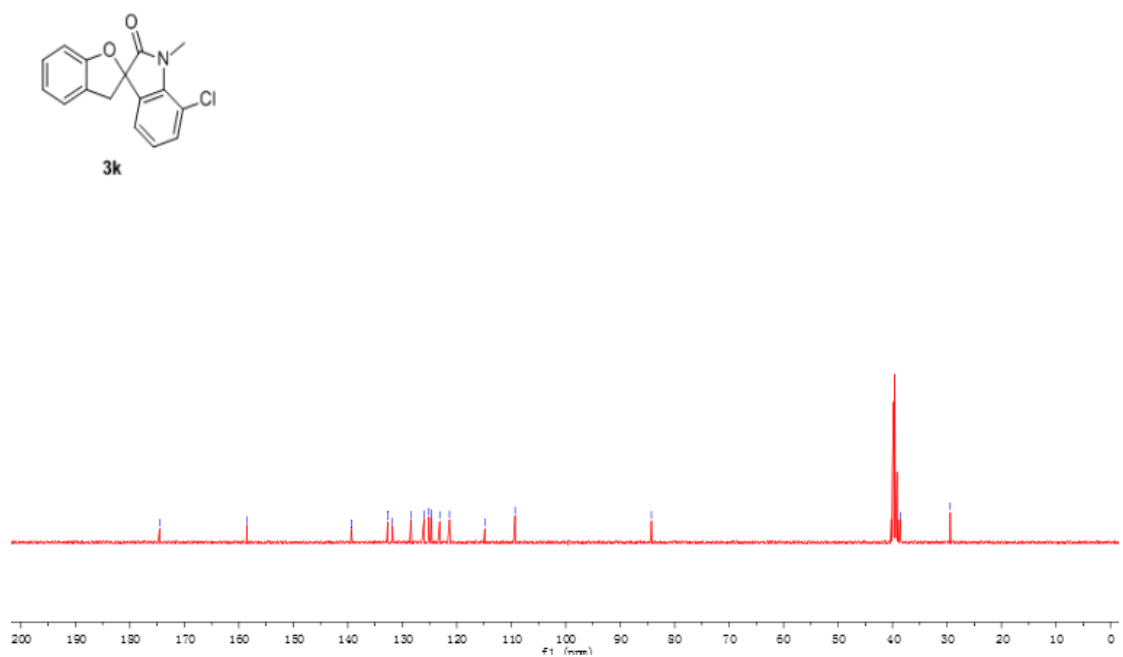
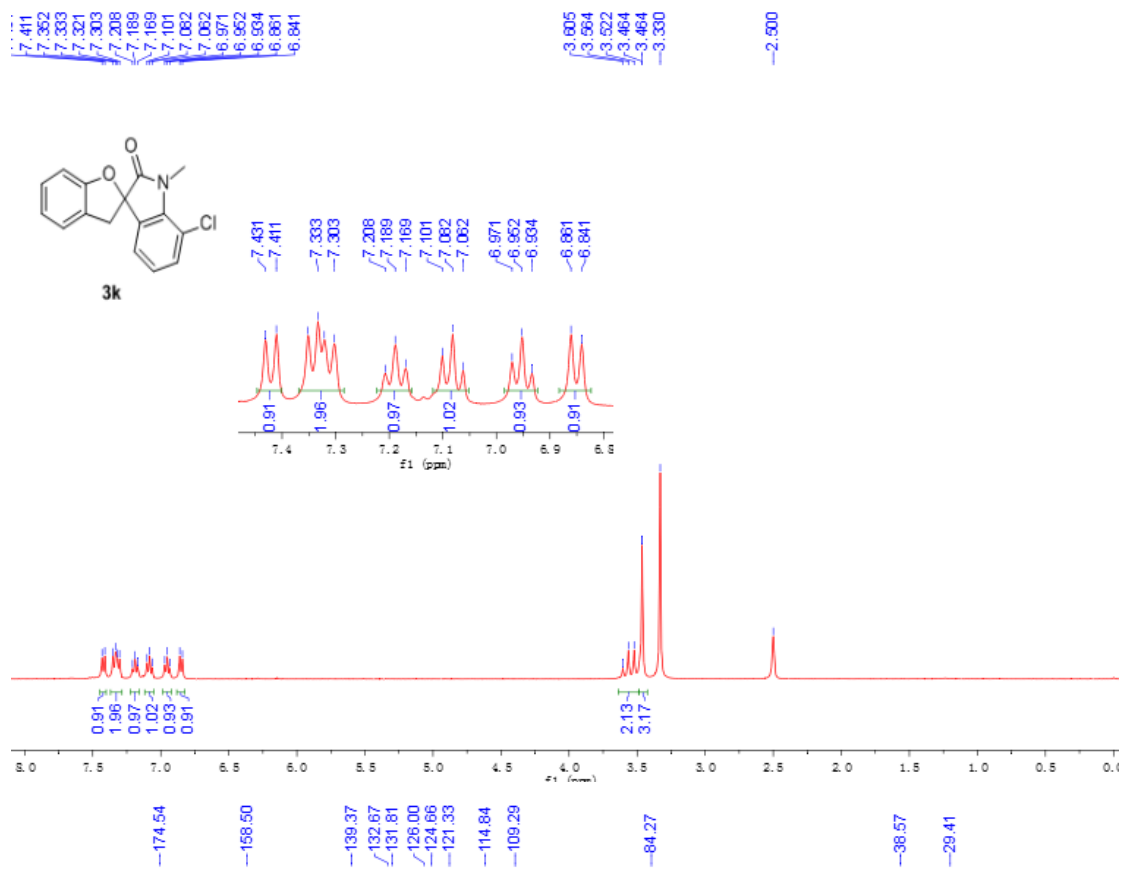


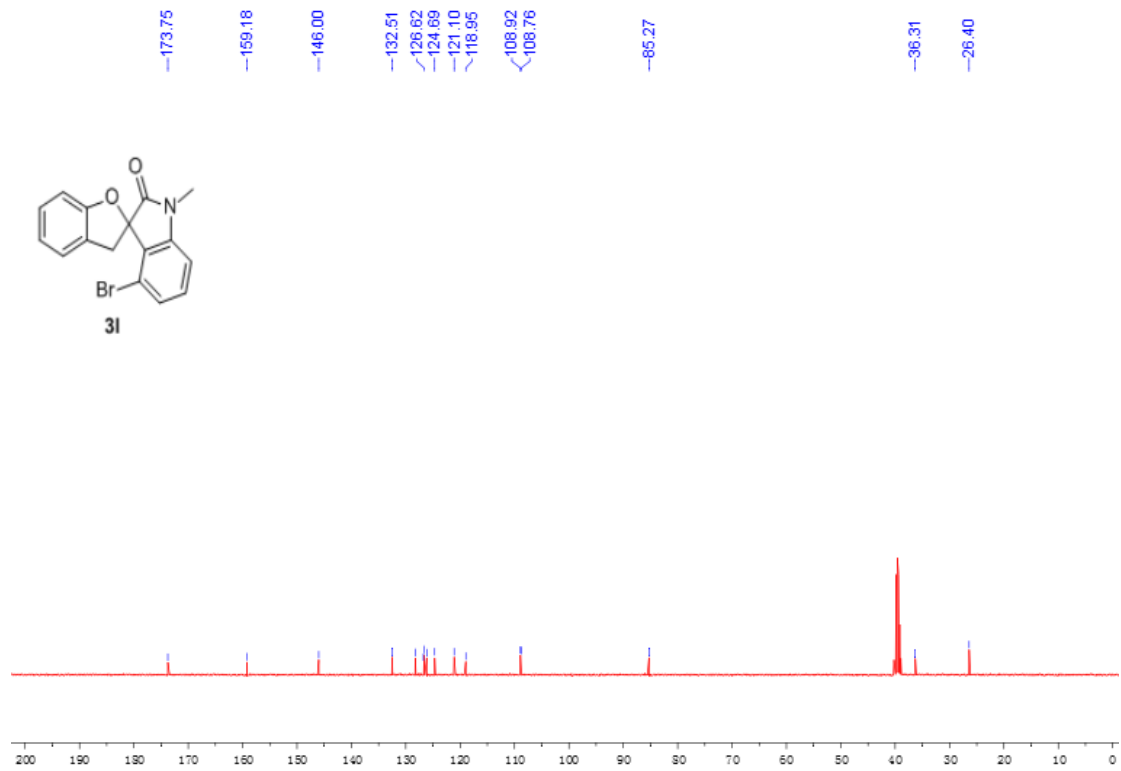
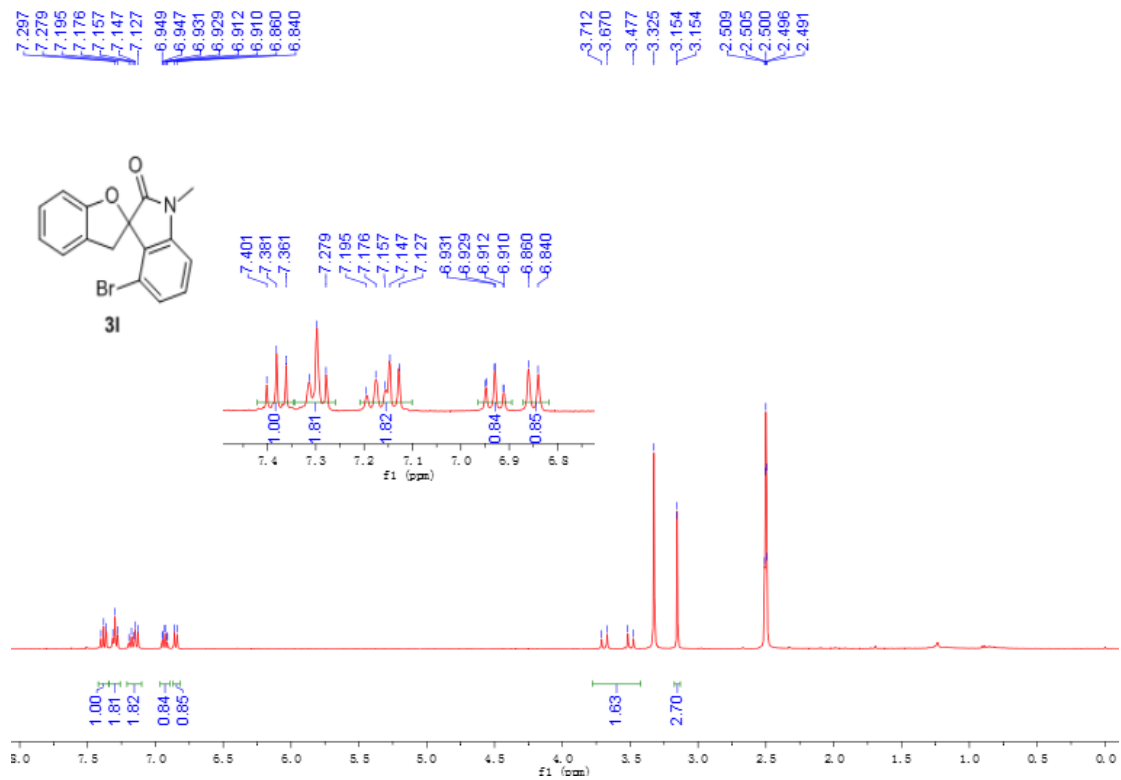


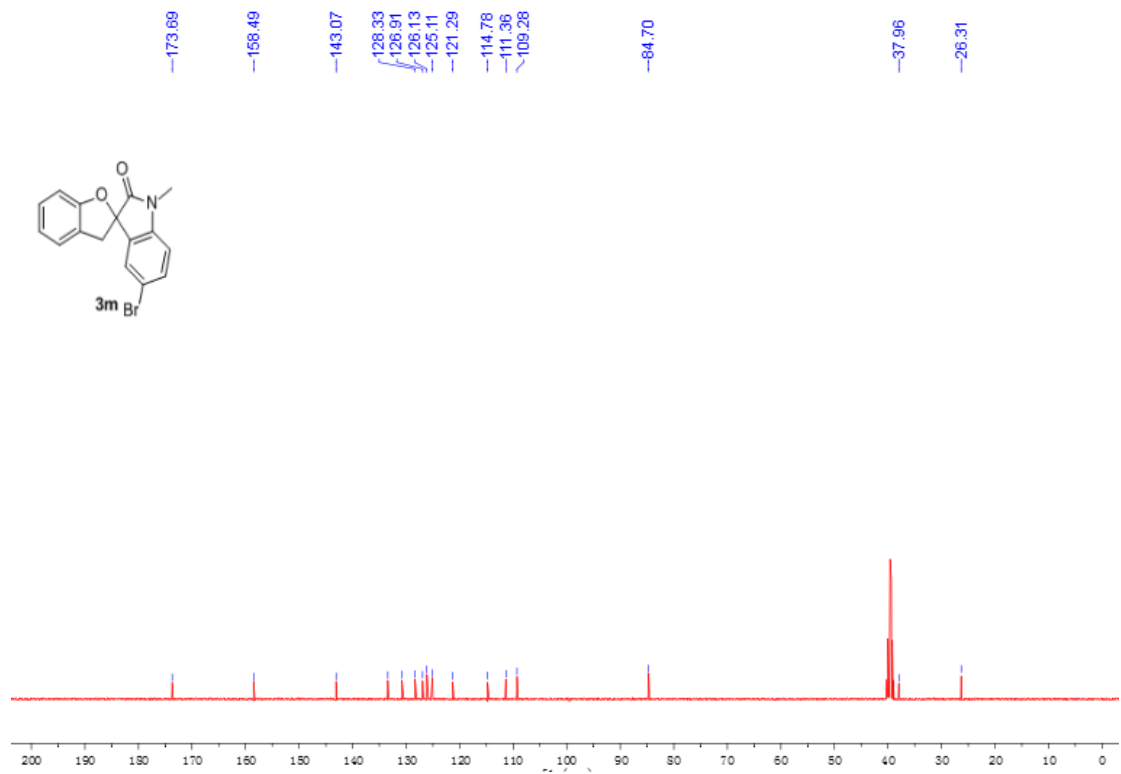
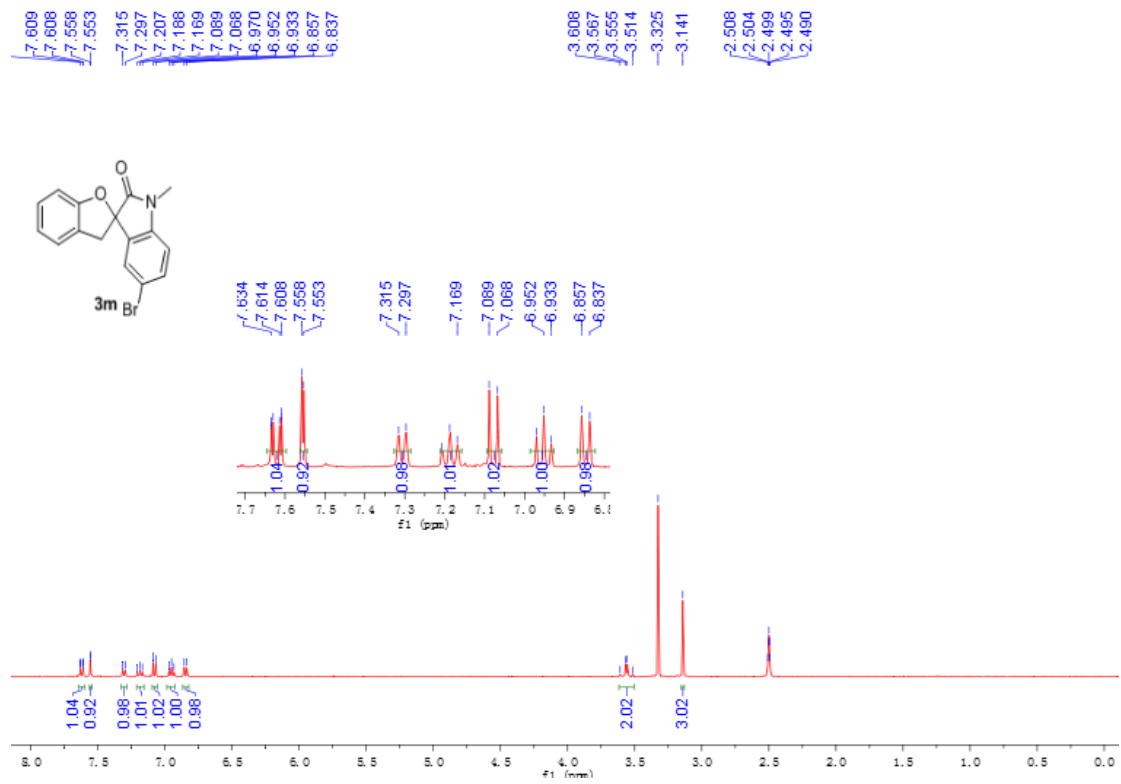




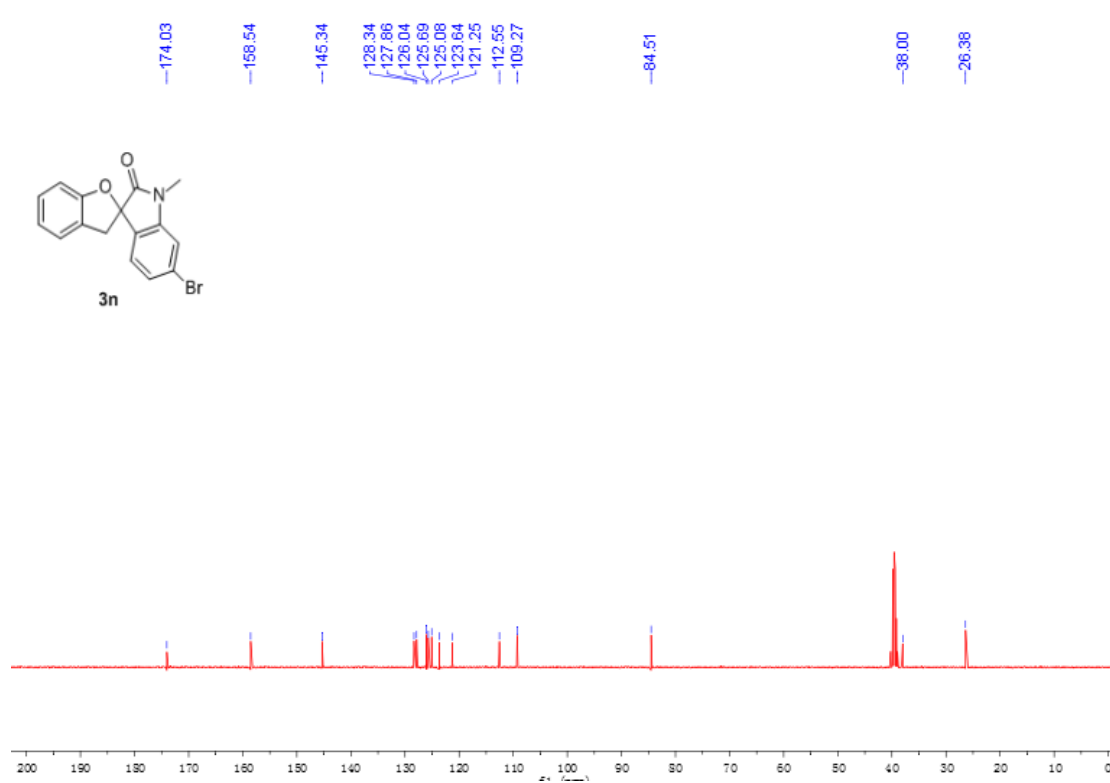
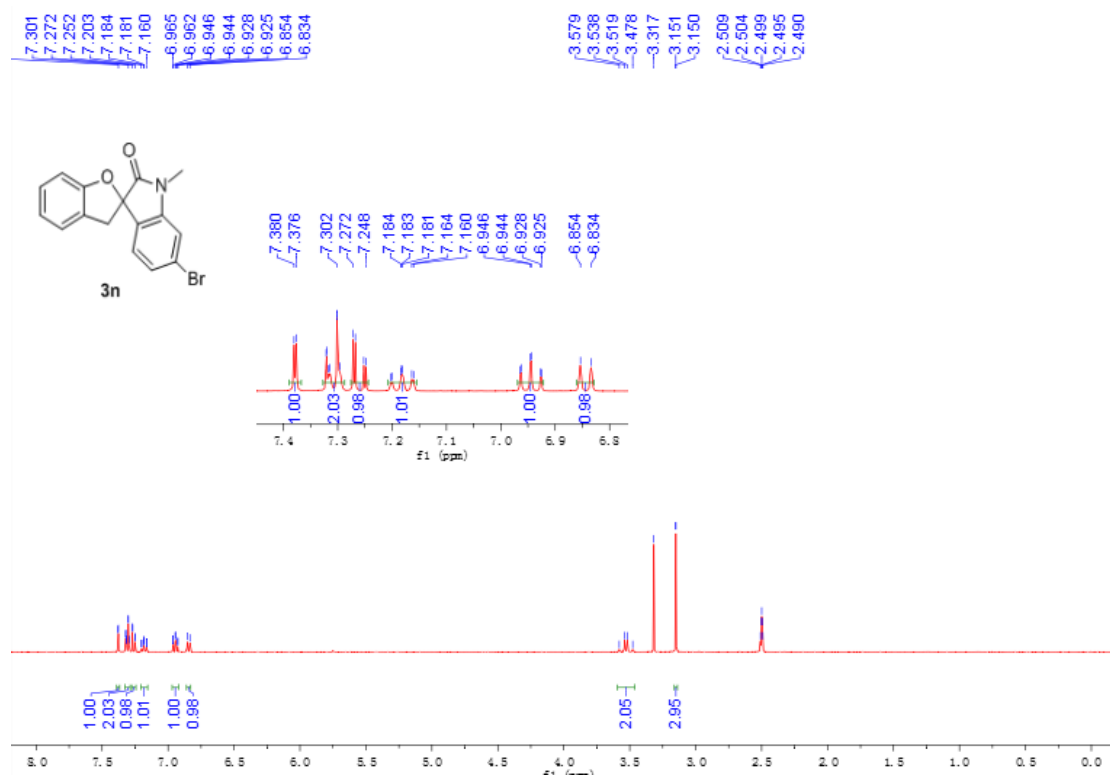


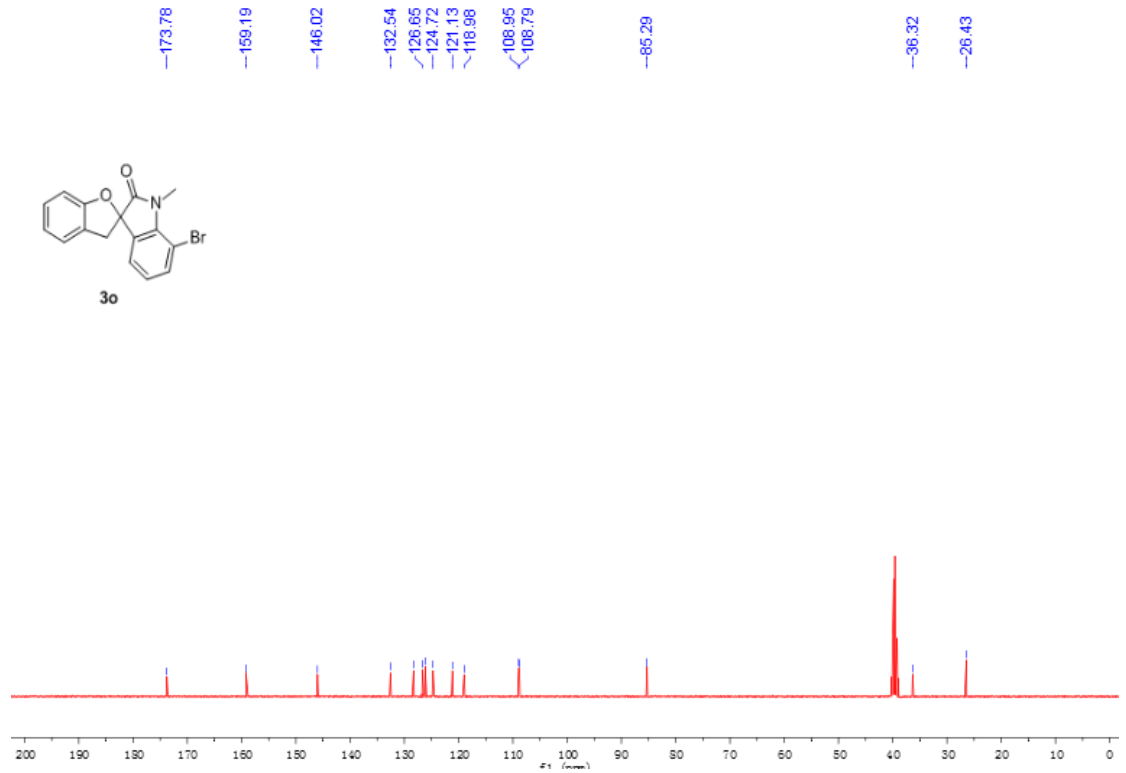
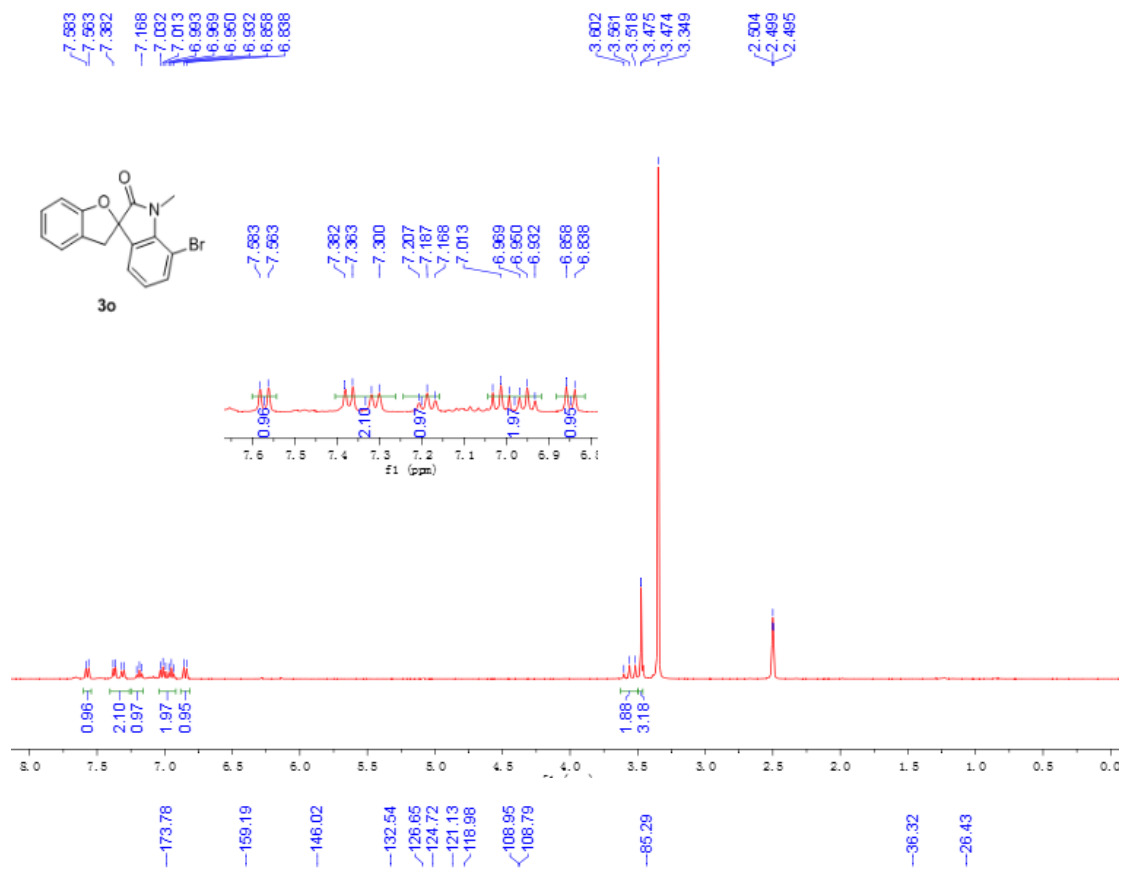


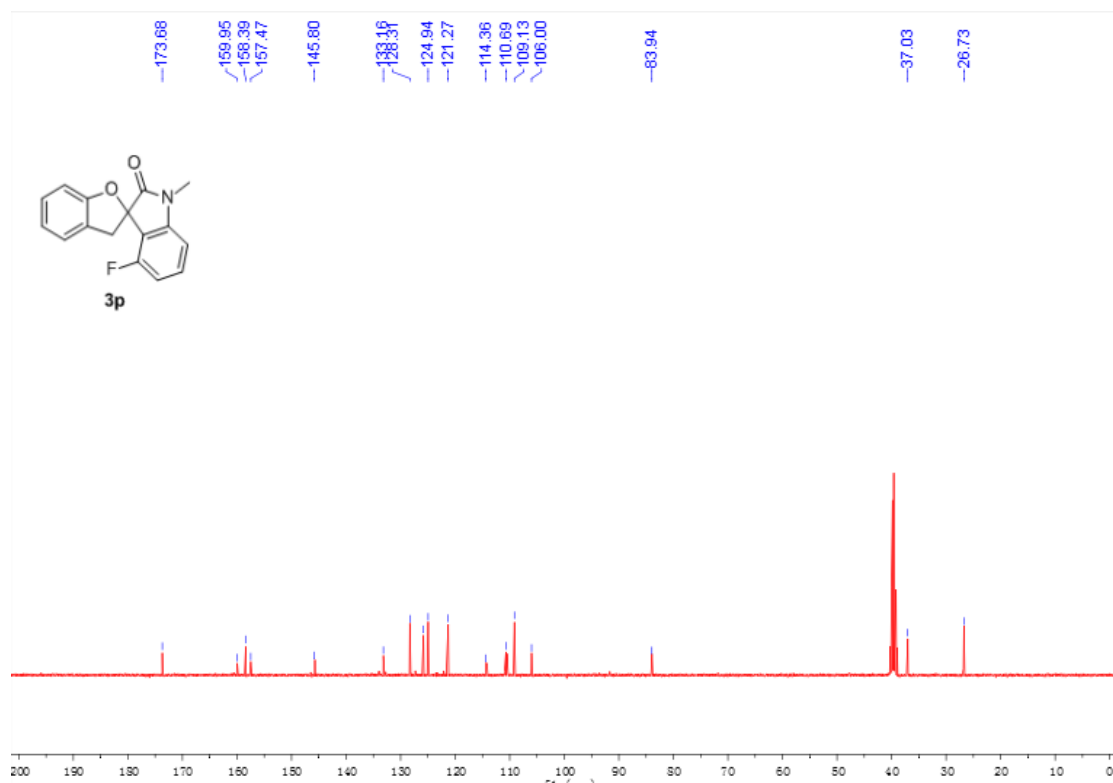
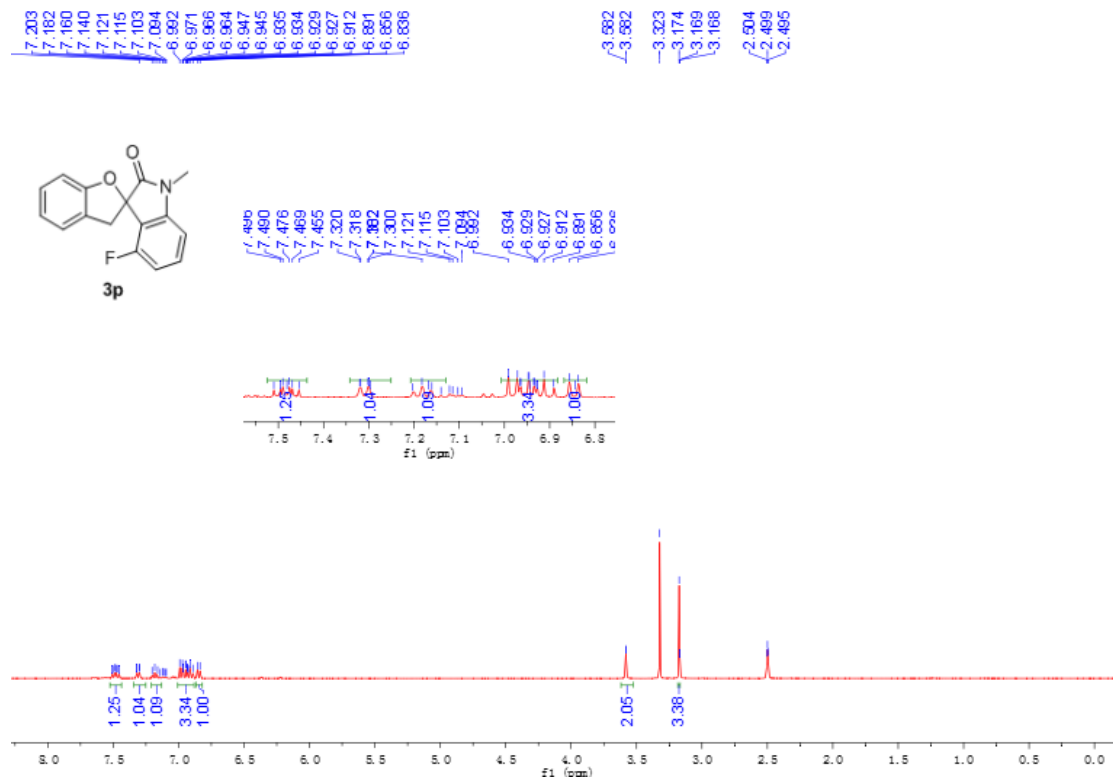


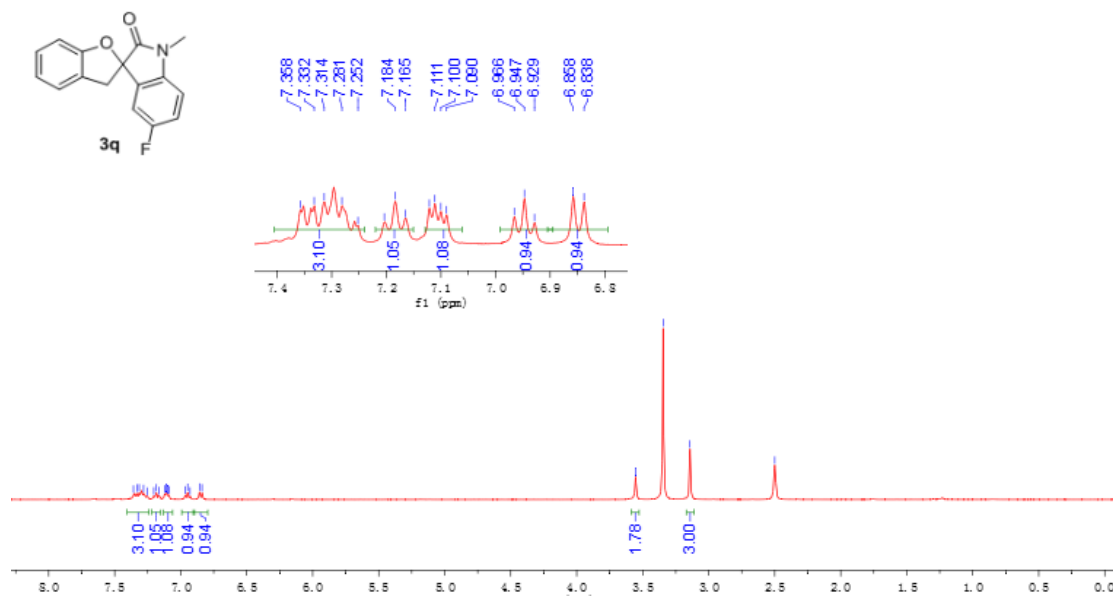
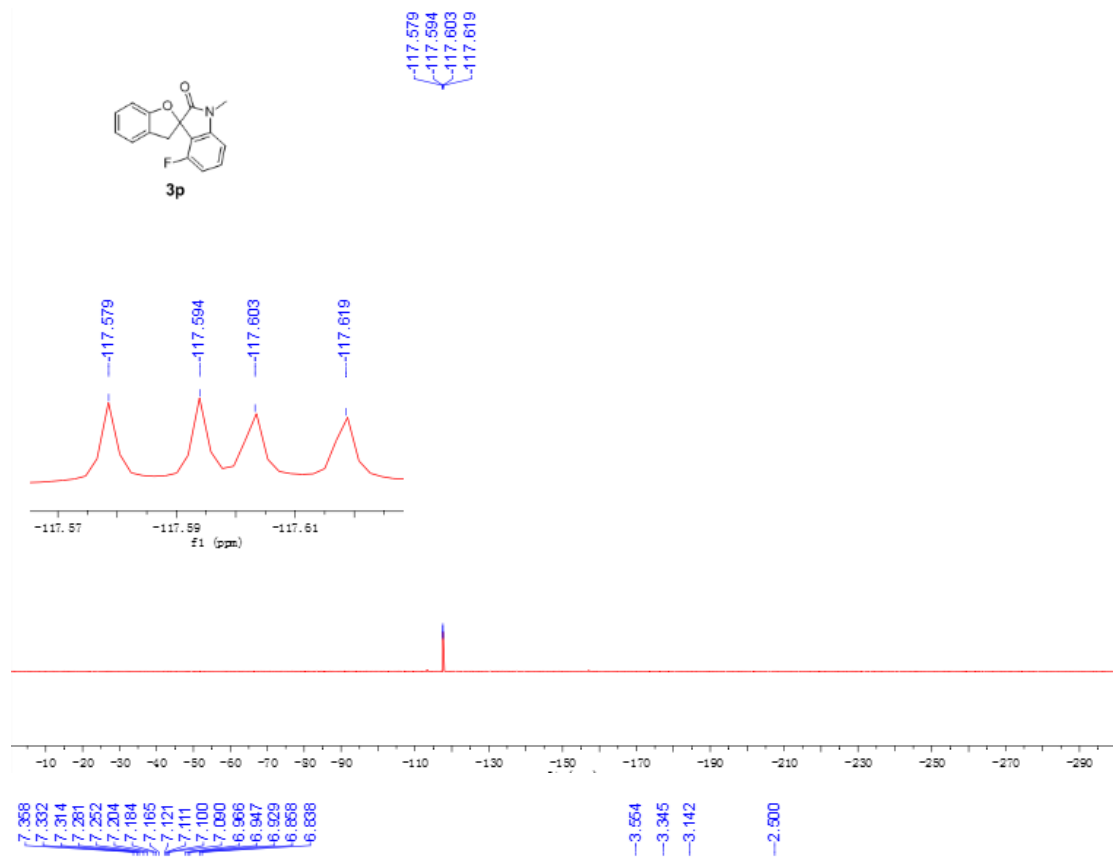


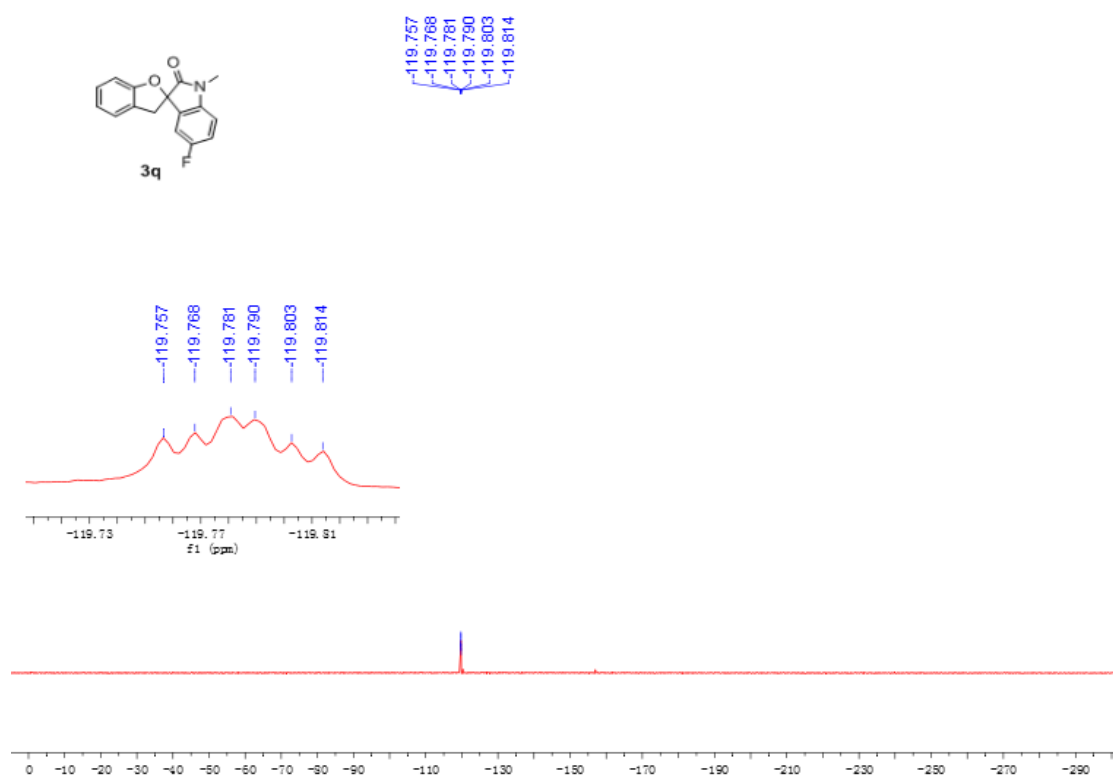
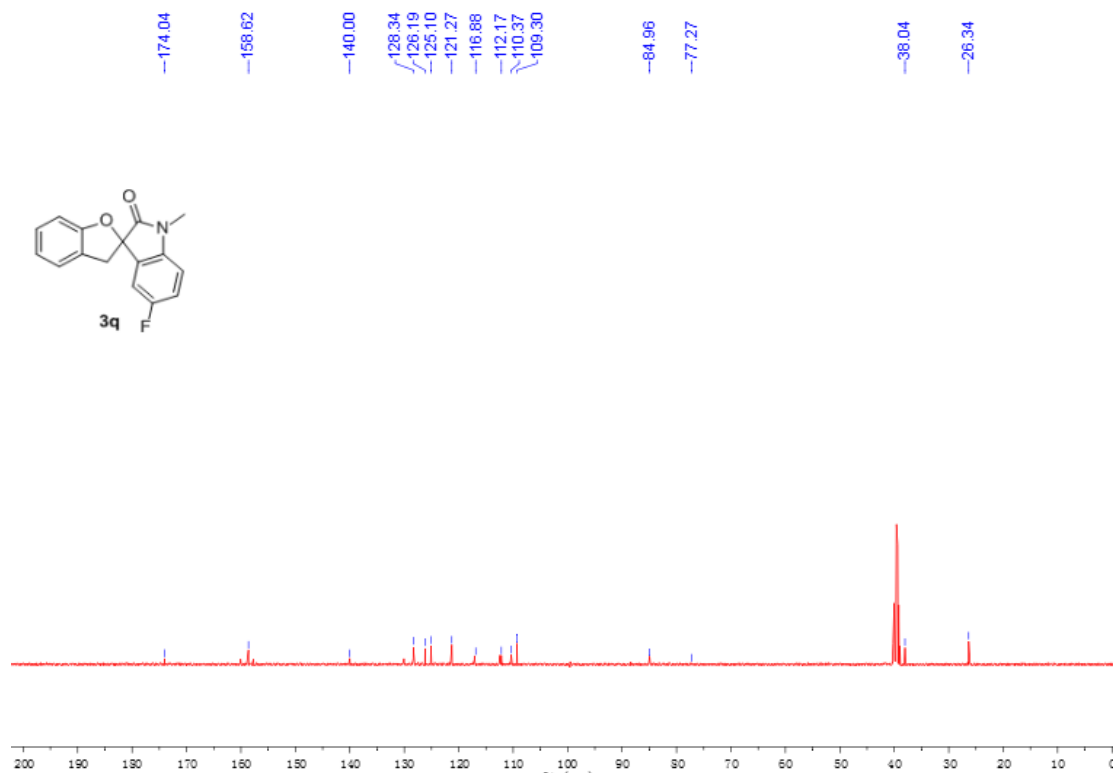


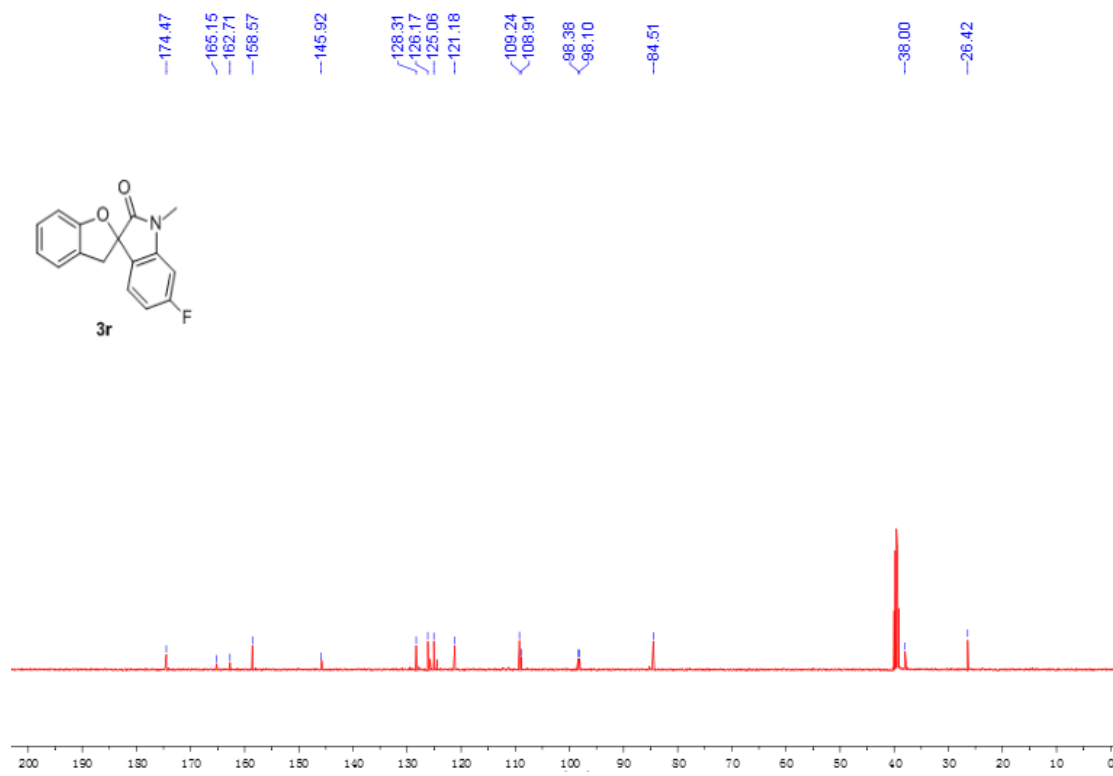
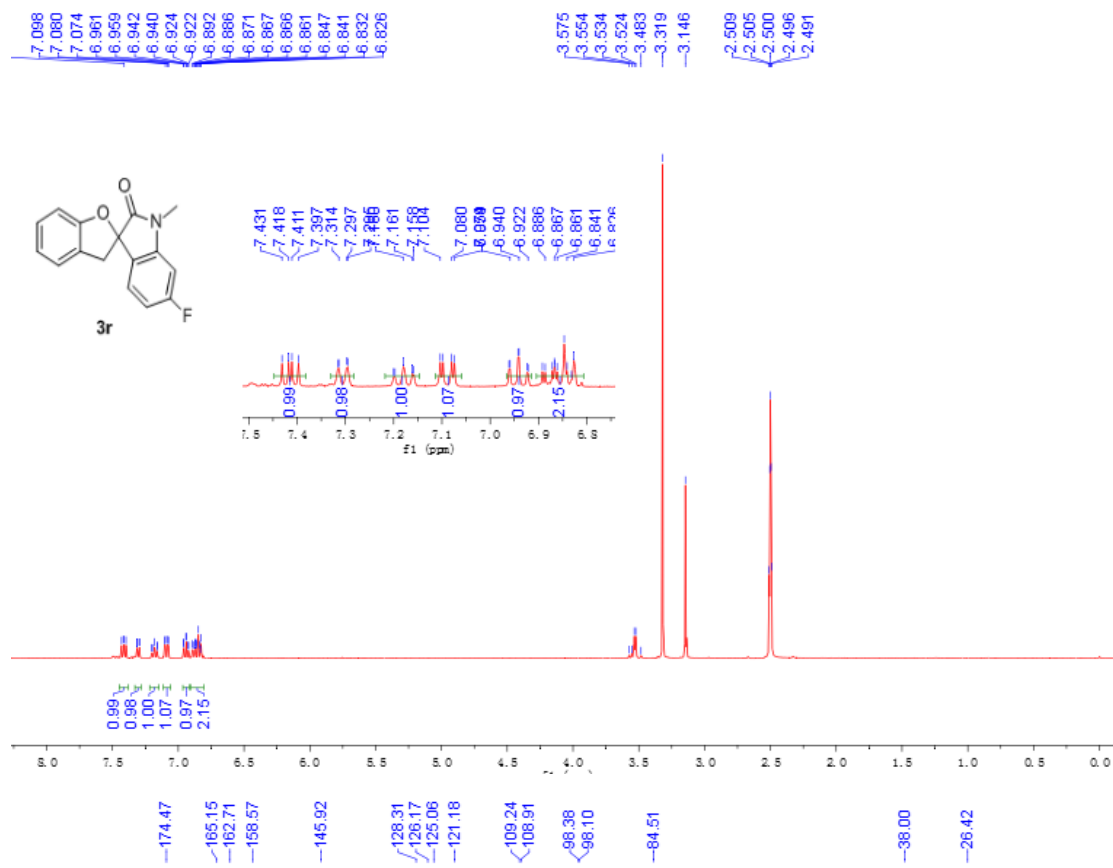


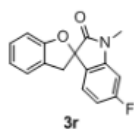




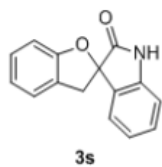
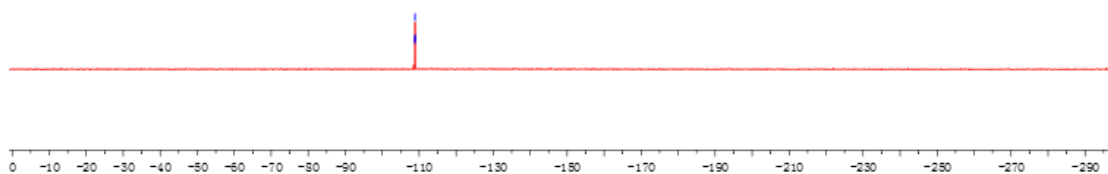
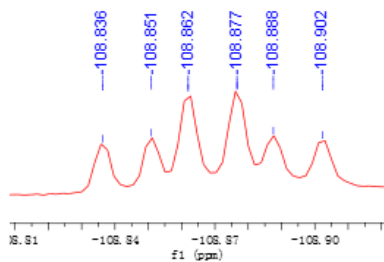








108.836  
108.851  
108.862  
108.877  
108.888  
108.902



7.333  
7.304  
7.286  
7.192  
7.173  
7.153  
7.019  
6.982  
6.929  
6.911  
6.882  
6.832

3.576  
3.535  
3.490  
3.449  
3.336

-2.469

