

Supporting Information

An Intramolecular Cascade Cyclization of 2-Aryl Indoles: Efficient Methods for the  
Construction of 2,3-Functionalized Indolines and 3-Indolinones

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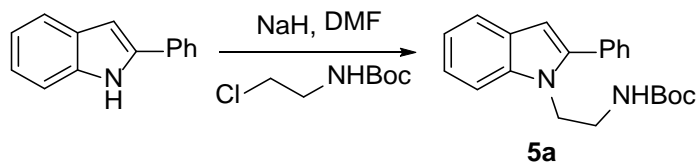
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**1. General Information:** Those reactions which required anhydrous conditions were carried out under an argon atmosphere using oven-dried glassware (120 °C). All chemicals and reagents were purchased from commercial suppliers and used without further purification. Anhydrous solvents were obtained as follows: anhydrous diethyl ether and toluene were distilled from sodium metal under argon, and anhydrous dichloromethane and tetrachloromethane were dried *via* distillation from CaH<sub>2</sub> immediately prior to use under argon. All other solvents were reagent grade. TLC analysis was conducted using glass-backed Thin-Layer Silica Gel Chromatography Plates (60 Å, 250 µm thickness, F-254 indicator). Flash chromatography was performed using 230-400 mesh, 60 Å pore diameter silica gel. <sup>1</sup>H NMR spectra were recorded at 400 or 500 MHz. <sup>13</sup>C NMR spectra were recorded at 100 or 150 MHz. Chemical shifts are reported in parts per million and are referenced to the deuterated residual solvent peak. NMR data is reported as: δ value (chemical shift, *J*-value (Hz), integration, where s = singlet, d = doublet, t = triplet, q = quartet, brs = broad singlet). IR spectra were recorded on a Varian 2000 Infrared spectrophotometer and are reported as cm<sup>-1</sup>. LRMS spectra were recorded at the Purdue University Department of Chemistry Mass Spectrometry Center. Melting point was measured on a melting point apparatus and was uncorrected.

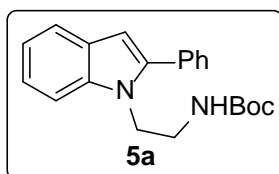
## 2. Experimental details for substrates and products:

### 2.1 General procedure for the synthesis of indoles **5**:

Compound **5a** was prepared as follows:

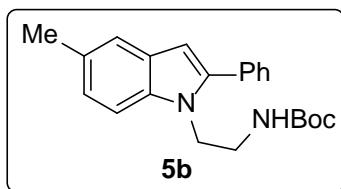


To a solution of 2-phenylindole (193 mg, 1.0 mmol) in anhydrous DMF (2 mL) at 0 °C under argon was added NaH (60%, 60 mg, 1.5 mmol). After stirring at 0 °C for 1 h, a solution of tert-butyl(2-chloroethyl)carbamate (269 mg, 1.5 mmol) in DMF (1 mL) was added dropwise. The resulting mixture was heated up at 45 °C for 12 h, cooled to 0 °C, and diluted with Et<sub>2</sub>O (5 mL) and water (3 mL). The aqueous phase was extracted with Et<sub>2</sub>O (3 × 5 mL). The combined organic extracts were washed by saturated salt water (2 × 5 mL). The organic was then dried over MgSO<sub>4</sub>, filtered, and concentrated. The residue was purified *via* silica gel chromatography (20:1 to 10:1 hexane/ethyl acetate) to afford **5a** (273 mg, 81%) as a light yellow oil. Compounds **5b-5j** were prepared in the same manner.



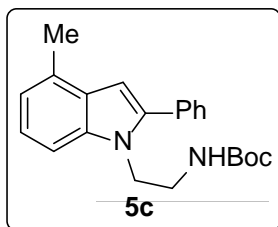
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.64 (d, *J* = 7.6 Hz, 1H), 7.55-7.35 (m, 6H), 7.34-7.21 (m, 1H), 7.16 (t, *J* = 7.2 Hz, 1H), 6.55 (s, 1H), 4.39 (brs, 1H), 4.33 (t, *J* = 5.6 Hz, 2H), 3.33 (dd, *J* = 12.0, 5.6 Hz, 2H), 1.38 (brs, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 155.6, 141.2, 137.7, 132.9, 129.5, 128.6, 128.1, 121.9, 120.5, 120.1, 110.1, 102.9, 79.4, 43.5, 40.3, 28.3; IR (neat) 3415, 1694, 1462, 1367, 1167 cm<sup>-1</sup>; LRMS (ESI), *m/z* 359.2 (M+Na)<sup>+</sup>.

Compounds **5b**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5b** (83% yield) as a light yellow oil.



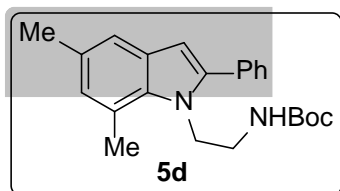
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.52-7.34 (m, 7H), 7.09 (d, *J* = 8.0 Hz, 1H), 6.49 (s, 1H), 4.41 (brs, 1H), 4.30 (d, *J* = 5.5 Hz, 2H), 3.34 (d, *J* = 6.0 Hz, 2H), 2.50 (s, 3H), 1.40 (brs, 9H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 155.6, 141.3, 136.1, 133.0, 129.4, 129.3, 128.6, 128.4, 128.0, 123.5, 120.2, 109.8, 102.3, 79.3, 43.6, 40.3, 28.3, 21.4; IR (neat) 3410, 1698, 1474, 1366, 1171 cm<sup>-1</sup>; LRMS (ESI), *m/z* 373.2 (M+Na)<sup>+</sup>.

Compounds **5c**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5c** (80% yield) as a light yellow oil.



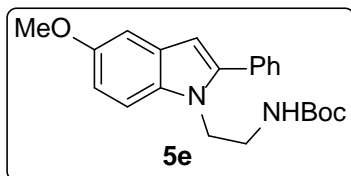
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60-7.39 (m, 5H), 7.33 (d,  $J = 8.0$  Hz, 1H), 7.19 (t,  $J = 8.0$  Hz, 1H), 6.98 (d,  $J = 7.2$  Hz, 1H), 6.59 (s, 1H), 4.43 (brs, 1H), 4.33 (t,  $J = 5.6$  Hz, 2H), 3.35 (d,  $J = 5.6$  Hz, 2H), 2.60 (s, 3H), 1.40 (brs, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.6, 140.6, 137.3, 133.0, 130.0, 129.4, 128.6, 128.0, 122.1, 120.3, 107.8, 101.4, 79.3, 43.6, 40.3, 28.3, 18.6; IR (neat) 3411, 1714, 1366, 1169, 761  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  373.2 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **5d**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5d** (86% yield) as a light yellow oil.



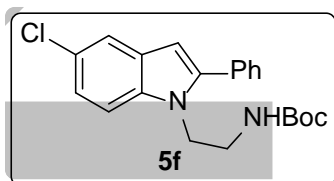
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57-7.32 (m, 5H), 7.28 (s, 1H), 6.84 (s, 1H), 6.49 (s, 1H), 4.44 (d,  $J = 5.2$  Hz, 2H), 4.03 (brs, 1H), 3.12 (d,  $J = 5.6$  Hz, 2H), 2.72 (s, 3H), 2.44 (s, 3H), 1.34 (brs, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.3, 143.2, 135.3, 133.2, 130.1, 129.8, 129.4, 128.6, 127.8, 127.1, 121.3, 118.4, 104.1, 79.2, 45.6, 41.4, 28.2, 21.0, 20.2; IR (neat) 3416, 1713, 1249, 1171, 701  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  387.2 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **5e**: prepared according to the general procedure with 15:1 to 8:1 hexane/ethyl acetate as eluent to afford **5e** (75% yield) as a light yellow oil.



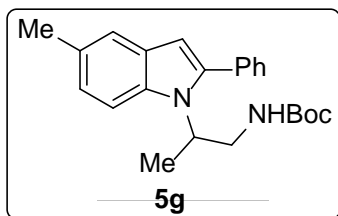
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52-7.31 (m, 6H), 7.11 (d,  $J = 1.6$  Hz, 1H), 6.91 (dd,  $J = 8.8, 2.4$  Hz, 1H), 6.48 (s, 1H), 4.44 (brs, 1H), 4.27 (d,  $J = 5.2$  Hz, 2H), 3.88 (s, 3H), 3.31 (d,  $J = 6.0$  Hz, 2H), 1.39 (brs, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.6, 154.4, 141.7, 132.9, 129.3, 128.6, 128.0, 112.1, 110.9, 102.5, 102.2, 79.4, 55.8, 43.6, 40.4, 28.3; IR (neat) 3373, 1694, 1471, 1215, 1034  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  389.2 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **5f**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5f** (79% yield) as a light yellow oil.



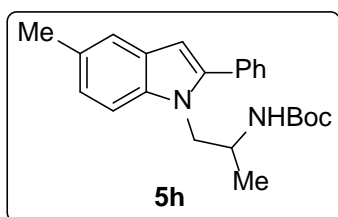
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (s, 1H), 7.55-7.31 (m, 6H), 7.18 (dd,  $J = 8.4, 2.0$  Hz, 1H), 6.48 (s, 1H), 4.39 (brs, 1H), 4.28 (d,  $J = 5.2$  Hz, 2H), 3.28 (dd,  $J = 12.0, 6.0$  Hz, 2H), 1.38 (brs, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.6, 142.4, 136.1, 132.4, 129.4, 129.1, 128.7, 128.4, 125.7, 122.1, 119.8, 111.1, 102.4, 79.5, 43.6, 40.3, 28.3; IR (neat) 3360, 1698, 1464, 1172, 914  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  393.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **5g**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5g** (70% yield) as a light yellow oil.



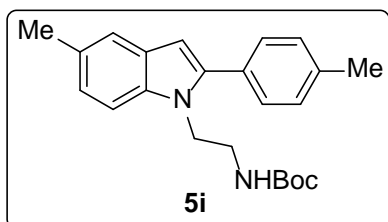
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.60-7.40 (m, 7H), 7.13 (d, *J* = 8.0 Hz, 1H), 6.49 (s, 1H), 4.45 (d, *J* = 10.5 Hz, 1H), 4.27 (brs, 1H), 4.17-4.00 (m, 2H), 2.52 (s, 3H), 1.45 (s, 9H), 0.73 (brs, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 154.8, 141.4, 136.1, 133.3, 129.6, 129.1, 128.5, 128.4, 127.8, 123.4, 120.1, 110.4, 102.3, 79.1, 48.3, 46.1, 28.2, 21.4, 17.9; IR (neat) 3344, 1706, 1473, 1366, 1168 cm<sup>-1</sup>; LRMS (ESI), *m/z* 387.2 (M+Na)<sup>+</sup>.

Compounds **5h**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5h** (72% yield) as a light yellow oil.



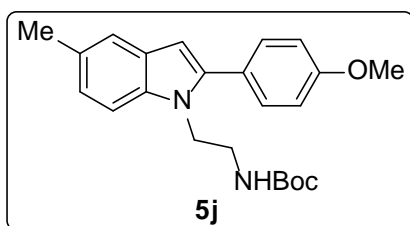
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62-7.34 (m, 7H), 7.11 (d, *J* = 8.4 Hz, 1H), 6.47 (s, 1H), 4.42 (brs, 1H), 4.23 (brs, 1H), 4.17-3.95 (m, 2H), 2.50 (s, 3H), 1.42 (s, 9H), 0.72 (brs, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 154.8, 141.4, 136.0, 133.3, 129.7, 129.2, 128.5, 127.8, 123.4, 120.1, 110.4, 102.4, 79.2, 48.3, 46.1, 28.3, 21.4, 18.0; IR (neat) 3349, 1705, 1473, 1168, 1058 cm<sup>-1</sup>; LRMS (ESI), *m/z* 387.2 (M+Na)<sup>+</sup>.

Compounds **5i**: prepared according to the general procedure with 20:1 to 10:1 hexane/ethyl acetate as eluent to afford **5i** (87% yield) as a light yellow oil.



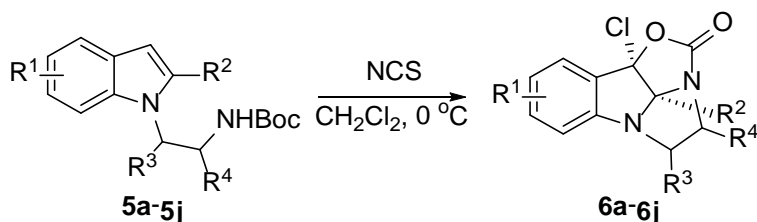
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.44 (s, 1H), 7.43-7.32 (m, 3H), 7.31-7.21 (m, 2H), 7.09 (d, *J* = 8.4 Hz, 1H), 6.46 (s, 1H), 4.42 (brs, 1H), 4.30 (brs, 2H), 3.34 (d, *J* = 5.6 Hz, 2H), 2.50 (s, 3H), 2.45 (s, 3H), 1.41 (s, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 155.6, 141.3, 137.8, 136.0, 130.0, 129.3, 128.4, 123.3, 120.1, 109.7, 102.0, 79.3, 43.5, 40.3, 28.2, 21.3, 21.2; IR (neat) 3412, 1714, 1505, 1170, 733 cm<sup>-1</sup>; LRMS (ESI), *m/z* 387.2 (M+Na)<sup>+</sup>.

Compounds **5j**: prepared according to the general procedure with 15:1 to 8:1 hexane/ethyl acetate as eluent to afford **5j** (79% yield) as a light yellow oil.



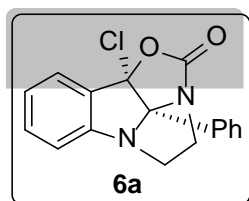
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.45-7.37 (m, 3H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 2H), 6.42 (s, 1H), 4.40 (brs, 1H), 4.27 (brs, 2H), 3.87 (s, 3H), 3.33 (d, *J* = 5.5 Hz, 2H), 2.48 (s, 3H), 1.39 (s, 9H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 159.4, 155.6, 141.1, 135.9, 130.6, 129.2, 128.4, 125.3, 123.2, 120.0, 114.0, 109.7, 101.8, 79.3, 55.3, 43.5, 40.4, 28.3, 21.4; IR (neat) 3405, 1710, 1503, 1249, 1175 cm<sup>-1</sup>; LRMS (ESI), *m/z* 403.2 (M+Na)<sup>+</sup>.

## 2.2 General Procedure for the intramolecular *N*-nucleophilic cyclization of 2-aryl indoles:



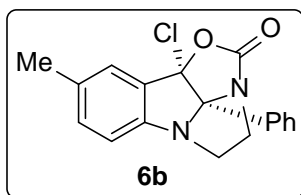
To a solution of indole **5** (0.2 mmol) in freshly distilled  $\text{CH}_2\text{Cl}_2$  (2 mL) at  $0^\circ\text{C}$  under argon was added NCS (0.6 mmol). The mixture was stirred at  $0^\circ\text{C}$  for 3.5 h. After the substrate disappeared completely (*via* TLC), the reaction mixture was directly subjected to silica gel chromatography affording the corresponding product **6a-6j**.

Compounds **6a**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6a** (91% yield) as a sticky yellow oil.



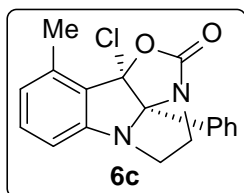
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69-7.58 (m, 2H), 7.55-7.39 (m, 5H), 7.10 (dt,  $J = 7.5, 0.5$  Hz, 1H), 6.90 (d,  $J = 8.0$  Hz, 1H), 4.10-4.02 (m, 1H), 3.85-3.59 (m, 2H), 3.40-3.31 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.4, 150.9, 135.0, 133.0, 129.6, 129.3, 128.4, 127.6, 125.7, 123.5, 114.1, 103.9, 97.9, 53.1, 47.2; IR (neat) 1784, 1606, 1477, 1342, 983  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  335.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **6b**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6b** (93% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70-7.57 (m, 2H), 7.55-7.38 (m, 3H), 7.35-7.17 (m, 2H), 6.81 (d,  $J = 8.4$  Hz, 1H), 4.10-3.97 (m, 1H), 3.75-3.52 (m, 2H), 3.40-3.36 (m, 1H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.4, 148.6, 135.2, 133.8, 133.4, 129.6, 129.3, 128.4, 127.6, 125.8, 113.9, 104.0, 98.3, 53.3, 47.0, 20.8; IR (neat) 1784, 1493, 1340, 983, 734  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  349.1 ( $\text{M}+\text{Na}$ ) $^+$ .

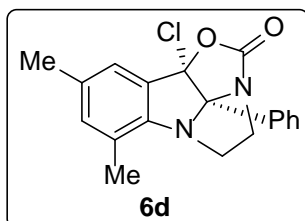
Compounds **6c**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6c** (87% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74-7.60 (m, 2H), 7.59-7.38 (m, 3H), 7.37-7.20 (m, 1H), 6.86 (d,  $J = 7.6$  Hz, 1H), 6.75 (d,  $J = 8.0$  Hz, 1H), 3.99-3.72 (m, 2H), 3.68-3.52 (m, 1H), 3.39-3.24 (m, 1H), 2.51 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.3, 151.1, 137.9, 135.1, 132.6, 129.6, 128.4, 127.7, 126.8,

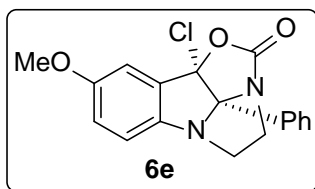
125.5, 111.5, 104.3, 98.1, 53.8, 46.0, 17.6; IR (neat) 1784, 1597, 1450, 1344, 978  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  349.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

Compounds **6d**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6d** (92% yield) as a sticky yellow oil.



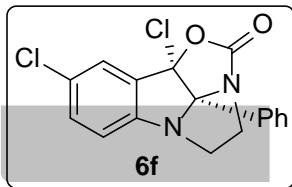
<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68-7.55 (m, 2H), 7.48-7.35 (m, 3H), 7.13 (s, 1H), 7.05 (s, 1H), 4.32-4.20 (m, 1H), 3.72-3.61 (m, 1H), 3.43-3.28 (m, 2H), 2.34 (s, 3H), 2.31 (s, 3H); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  158.0, 147.5, 136.2, 135.3, 134.1, 129.8, 129.4, 128.2, 127.4, 124.9, 123.3, 104.5, 98.1, 53.2, 48.9, 20.7, 17.7; IR (neat) 1790, 1486, 1335, 1040, 984  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  363.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

Compounds **6e**: prepared according to the general procedure with 8:1 to 6:1 hexane/ethyl acetate as eluent to afford **6e** (81% yield) as a light yellow solid. Melting point: 150-152 °C.



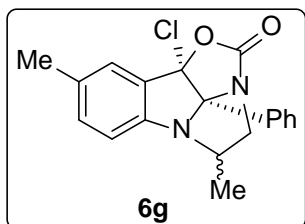
<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71-7.58 (m, 2H), 7.56-7.35 (m, 3H), 7.08-6.91 (m, 2H), 6.83 (d,  $J = 8.4$  Hz, 1H), 4.08-3.95 (m, 1H), 3.80 (s, 3H), 3.70-3.51 (m, 2H), 3.49-3.28 (m, 1H); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.3, 156.6, 144.3, 135.2, 129.8, 129.6, 128.4, 127.5, 120.7, 115.3, 109.0, 104.0, 98.7, 55.9, 53.6, 47.0; IR (neat) 1790, 1494, 1278, 985, 735  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  365.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

Compounds **6f**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6f** (95% yield) as a sticky yellow oil.



<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70-7.31 (m, 7H), 6.84 (d,  $J = 8.4$  Hz, 1H), 4.18-4.00 (m, 1H), 3.72-3.52 (m, 2H), 3.42-3.29 (m, 1H); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.1, 149.4, 134.6, 133.1, 130.9, 129.8, 128.5, 127.4, 125.7, 115.3, 102.9, 98.3, 53.1, 47.3; IR (neat) 1784, 1475, 1343, 987, 730  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  369.0 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

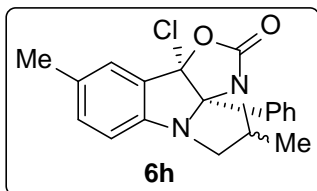
Compounds **6g**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6g** (86% yield) as a sticky yellow oil.



<sup>1</sup>H NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70-7.58 (m, 4H), 7.56-7.38 (m, 6H), 7.32-7.19 (m, 4H), 6.85-6.78 (m, 2H), 4.22-4.12 (m, 1H), 4.00 (dd,  $J = 12.5, 7.5$  Hz, 1H), 3.82-3.74 (m, 2H), 3.38-3.30 (m, 1H), 3.15 (dd,  $J = 12.5, 7.5$  Hz, 1H), 2.35 (s, 3H), 2.34 (s, 3H), 1.43 (d,  $J = 6.5$  Hz, 3H),

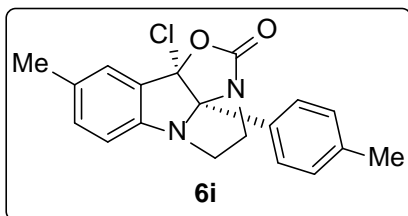
1.15 (d,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6, 154.1, 149.3, 147.9, 136.0, 135.4, 133.7, 133.4, 133.3, 129.5, 129.3, 129.1, 128.4, 128.3, 127.7, 125.9, 113.8, 113.6, 104.1, 103.1, 99.2, 98.2, 62.1, 61.0, 56.2, 56.0, 20.8, 20.6, 16.8; IR (neat) 1781, 1493, 1326, 980, 740  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  363.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **6h**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6h** (84% yield) as a sticky yellow oil.



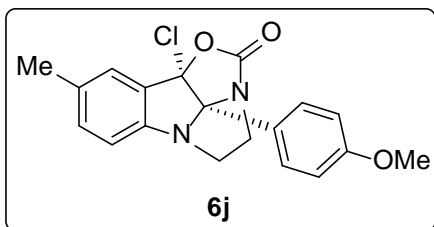
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72-7.56 (m, 6H), 7.53-7.35 (m, 8H), 7.33-7.20 (m, 6H), 6.83-6.77 (m, 3H), 4.23-4.11 (m, 1H), 3.99 (dd,  $J = 12.5, 7.5$  Hz, 1H), 3.82-3.72 (m, 4H), 3.39-3.29 (m, 2H), 3.14 (dd,  $J = 12.5, 7.5$  Hz, 1H), 2.35 (s, 6H), 2.33 (s, 3H), 1.43 (d,  $J = 6.5$  Hz, 6H), 1.15 (d,  $J = 6.5$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6, 154.2, 149.3, 147.9, 136.0, 135.4, 133.7, 133.5, 133.3, 129.5, 129.3, 129.2, 128.4, 128.3, 127.7, 125.9, 113.8, 113.6, 104.1, 103.1, 99.3, 98.2, 62.1, 61.0, 56.2, 56.0, 20.8, 20.6, 16.8; IR (neat) 1779, 1492, 1326, 980, 738  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  363.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **6i**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **6i** (95% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (s, 1H), 7.50 (s, 1H), 7.40-7.11 (m, 4H), 6.80 (d,  $J = 8.0$  Hz, 1H), 4.10-3.91 (m, 1H), 3.73-3.52 (m, 2H), 3.39-3.25 (m, 1H), 2.41 (s, 3H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.4, 148.6, 139.6, 133.7, 133.3, 132.2, 129.3, 129.1, 127.5, 125.8, 113.9, 104.0, 98.3, 53.3, 46.9, 21.2, 20.8; IR (neat) 1784, 1493, 1342, 984, 731  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  363.1 ( $\text{M}+\text{Na}$ ) $^+$ .

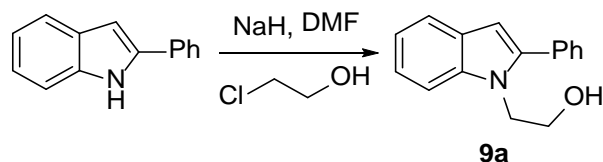
Compounds **6j**: prepared according to the general procedure with 8:1 to 6:1 hexane/ethyl acetate as eluent to afford **6j** (93% yield) as a sticky yellow oil.



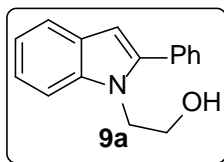
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54 (d,  $J = 8.0$  Hz, 2H), 7.27 (s, 1H), 7.21 (d,  $J = 8.0$  Hz, 1H), 6.96 (d,  $J = 8.0$  Hz, 2H), 6.79 (d,  $J = 8.0$  Hz, 1H), 4.08-3.92 (m, 1H), 3.85 (s, 3H), 3.72-3.52 (m, 2H), 3.37-3.23 (m, 1H), 2.33 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.6, 157.4, 148.5, 133.7, 133.4, 129.3, 128.9, 127.1, 125.8, 113.9, 113.7, 104.2, 98.2, 55.3, 53.4, 46.9, 20.8; IR (neat) 1783, 1493, 1253, 984, 731  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  379.1 ( $\text{M}+\text{Na}$ ) $^+$ .

### 2.3 General procedure for the synthesis of indoles **9**:

Compound **9a** was prepared as follows:

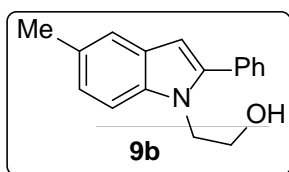


To a solution of 2-phenylindole (193 mg, 1.0 mmol) in anhydrous DMF (2 mL) at 0 °C under argon was added NaH (60%, 100 mg, 2.5 mmol). After stirring at 0 °C for 1 h, a solution of 2-chloroethanol (105 mg, 1.3 mmol) in DMF (1 mL) was added dropwise. The resulting mixture was heated up at 45 °C for 12 h, cooled to 0 °C, and diluted with Et<sub>2</sub>O (5 mL) and water (3 mL). The aqueous phase was extracted with Et<sub>2</sub>O (3 × 5 mL). The combined organic extracts were washed by saturated salt water (2 × 5 mL). The organic was then dried over MgSO<sub>4</sub>, filtered, and concentrated. The residue was purified *via* silica gel chromatography (8:1 to 5:1 hexane/ethyl acetate) to afford **9a** (202 mg, 85%) as a light yellow oil. Compounds **9b-9o** were prepared in the same manner.



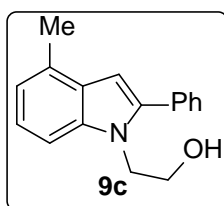
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.65 (d, *J* = 7.6 Hz, 1H), 7.64-7.30 (m, 6H), 7.26 (t, *J* = 7.6 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 6.57 (s, 1H), 4.33 (t, *J* = 6.0 Hz, 2H), 3.82 (t, *J* = 6.0 Hz, 2H), 1.47 (brs, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 141.6, 137.6, 132.8, 129.7, 128.5, 128.2, 128.1, 121.8, 120.6, 120.1, 110.1, 102.7, 61.7, 45.9; IR (neat) 3387, 1462, 1348, 1052, 750 cm<sup>-1</sup>; LRMS (ESI), *m/z* 260.1 (M+Na)<sup>+</sup>.

Compounds **9b**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as eluent to afford **9b** (87% yield) as a light yellow oil.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62-7.39 (m, 6H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 1H), 6.50 (s, 1H), 4.28 (t, *J* = 6.0 Hz, 2H), 3.76 (t, *J* = 6.0 Hz, 2H), 2.52 (s, 3H), 1.64 (brs, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 141.6, 136.1, 132.9, 129.6, 129.3, 128.5, 127.9, 123.4, 120.2, 109.8, 102.2, 61.6, 45.9, 21.3; IR (neat) 3382, 1472, 1332, 1051, 763 cm<sup>-1</sup>; LRMS (ESI), *m/z* 274.1 (M+Na)<sup>+</sup>.

Compounds **9c**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as eluent to afford **9c** (83% yield) as a light yellow oil.

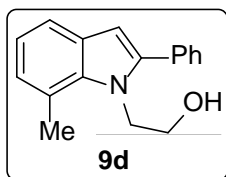


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.65-7.38 (m, 5H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 6.98 (d, *J* = 7.2 Hz, 1H), 6.60 (s, 1H), 4.33 (t, *J* = 6.0 Hz, 2H), 3.82 (t, *J* = 6.0 Hz, 2H), 2.60 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ



141.0, 137.3, 133.0, 130.2, 129.7, 128.5, 128.0, 122.0, 120.3, 107.7, 101.3, 61.7, 46.0, 18.6; IR (neat) 3374, 1481, 1348, 1054, 758  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  274.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

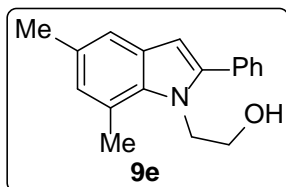
Compounds **9d**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as eluent to afford **9d** (79% yield) as a light yellow oil.



<sup>1</sup>H NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54-7.39 (m, 6H), 7.07 (t,  $J = 7.5$  Hz, 1H), 6.99 (d,  $J = 7.0$  Hz, 1H), 6.56 (s, 1H), 4.51 (t,  $J = 6.0$  Hz, 2H), 3.54 (t,  $J = 6.0$  Hz, 2H), 2.76 (s, 3H); <sup>13</sup>C NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  142.9, 136.8, 133.2, 129.7, 129.5, 128.5, 128.0, 125.4, 121.4, 120.5, 118.8, 104.2, 62.8, 47.3, 20.5;

IR (neat) 3364, 1451, 1314, 1045, 745  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  274.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

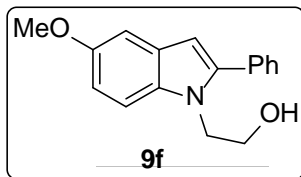
Compounds **9e**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as eluent to afford **9e** (84% yield) as a light yellow oil.



<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62-7.35 (m, 5H), 7.29 (s, 1H), 6.85 (s, 1H), 6.49 (s, 1H), 4.48 (t,  $J = 6.0$  Hz, 2H), 3.51 (t,  $J = 6.0$  Hz, 2H), 2.72 (s, 3H), 2.45 (s, 3H); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  143.0, 135.1, 133.3, 129.8, 129.6, 128.5, 127.9, 127.1, 121.0, 118.4, 103.8, 62.8, 47.3, 21.0, 20.3; IR (neat) 3369, 1471, 1329, 1050, 765  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$

288.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

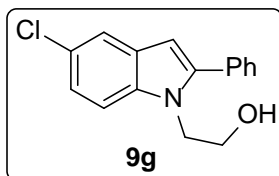
Compounds **9f**: prepared according to the general procedure with 6:1 to 4:1 hexane/ethyl acetate as eluent to afford **9f** (80% yield) as a light yellow oil.



<sup>1</sup>H NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58-7.38 (m, 5H), 7.34 (d,  $J = 8.5$  Hz, 1H), 7.11 (d,  $J = 2.0$  Hz, 1H), 6.90 (dd,  $J = 8.5, 2.5$  Hz, 1H), 6.49 (s, 1H), 4.29 (t,  $J = 6.0$  Hz, 2H), 3.87 (s, 3H), 3.81 (t,  $J = 6.0$  Hz, 2H), 1.51 (brs, 1H); <sup>13</sup>C NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.4, 142.2, 133.0, 132.9,

129.6, 128.5, 128.0, 112.0, 110.9, 102.4, 102.3, 61.8, 55.9, 46.1; IR (neat) 3419, 1472, 1214, 1033, 762  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  290.1 ( $\text{M}+\text{Na}$ )<sup>+</sup>.

Compounds **9g**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as eluent to afford **9g** (77% yield) as a light yellow oil.

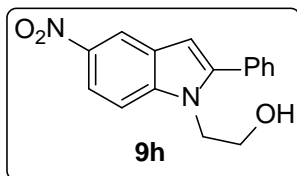


<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.59 (s, 1H), 7.56-7.38 (m, 5H), 7.34 (d,  $J = 8.8$  Hz, 1H), 7.18 (dd,  $J = 8.8, 1.6$  Hz, 1H), 6.48 (s, 1H), 4.28 (t,  $J = 5.6$  Hz, 2H), 3.77 (t,  $J = 5.6$  Hz, 2H), 1.56 (brs, 1H); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  142.9, 136.1, 132.3, 129.6, 129.1, 128.6, 128.4, 125.7, 122.0,

119.9, 111.1, 102.2, 61.6, 46.0; IR (neat) 3378, 1463, 1329, 1065, 763  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  294.1

(M+Na)<sup>+</sup>.

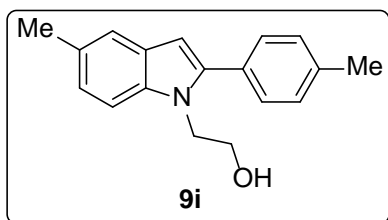
Compounds **9h**: prepared according to the general procedure with 6:1 to 3:1 hexane/ethyl acetate as eluent to afford **9h** (71% yield) as a yellow oil.



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.53 (d, *J* = 2.0 Hz, 1H), 8.10 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.58-7.41 (m, 6H), 6.70 (s, 1H), 4.37 (t, *J* = 5.5 Hz, 2H), 3.86 (t, *J* = 5.5 Hz, 2H), 1.63 (brs, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ

144.8, 141.8, 140.6, 131.6, 129.7, 128.9, 128.8, 127.3, 117.6, 117.3, 110.2, 104.7, 61.6, 46.3; IR (neat) 3445, 1511, 1333, 1069, 753 cm<sup>-1</sup>; LRMS (ESI), *m/z* 305.1 (M+Na)<sup>+</sup>.

Compounds **9i**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as

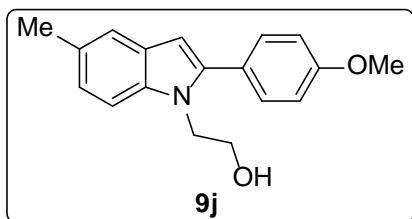


eluent to afford **9i** (87% yield) as a light yellow oil.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.51-7.20 (m, 6H), 7.08 (d, *J* = 8.4 Hz, 1H), 6.46 (s, 1H), 4.29 (t, *J* = 6.0 Hz, 2H), 3.79 (t, *J* = 6.0 Hz, 2H), 2.50 (s, 3H), 2.45 (s, 3H), 1.51 (brs, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ

141.7, 137.9, 136.0, 130.0, 129.5, 129.2, 128.5, 123.2, 120.2, 109.7, 101.9, 61.7, 45.9, 21.3, 21.2; IR (neat) 3368, 1475, 1331, 1052, 791 cm<sup>-1</sup>; LRMS (ESI), *m/z* 288.1 (M+Na)<sup>+</sup>.

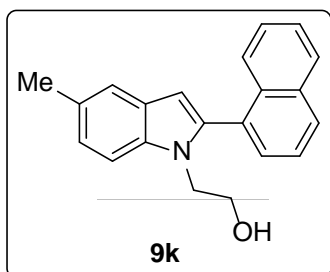
Compounds **9j**: prepared according to the general procedure with 6:1 to 4:1 hexane/ethyl acetate as eluent to afford **9j** (78% yield) as a light yellow oil.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.41 (s, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.42 (s, 1H), 4.28 (t, *J* = 5.6 Hz, 2H), 3.87 (s, 3H), 3.82 (t, *J* = 5.6 Hz, 2H), 2.47 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ

159.5, 141.5, 135.9, 130.9, 129.3, 128.5, 125.3, 123.1, 120.1, 114.0, 109.6, 101.8, 61.8, 55.3, 45.9, 21.4; IR (neat) 3403, 1476, 1250, 1038, 791 cm<sup>-1</sup>; LRMS (ESI), *m/z* 304.1 (M+Na)<sup>+</sup>.

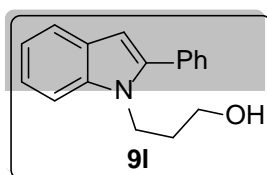
Compounds **9k**: prepared according to the general procedure with 8:1 to 5:1 hexane/ethyl acetate as eluent to afford **9k** (65% yield) as a light yellow oil.



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.04-7.88 (m, 2H), 7.66 (d, *J* = 8.5 Hz, 1H), 7.61-7.35 (m, 6H), 7.13 (d, *J* = 8.0 Hz, 1H), 6.57 (s, 1H), 4.25-4.10 (m, 1H), 4.00-3.88 (m, 1H), 3.68-3.52 (m, 2H), 2.52 (s, 3H); <sup>13</sup>C

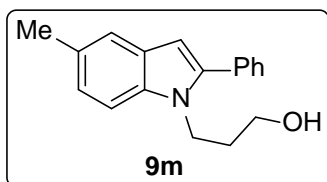
NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  138.9, 135.6, 133.5, 133.0, 130.5, 129.3, 129.1, 128.5, 128.3, 126.7, 126.1, 125.8, 125.2, 123.4, 120.3, 109.6, 103.6, 61.9, 46.1, 21.4; IR (neat) 3391, 1458, 1394, 1052, 779 cm<sup>-1</sup>; LRMS (ESI),  $m/z$  324.1 (M+Na)<sup>+</sup>.

Compounds **9l**: prepared according to the general procedure with 6:1 to 4:1 hexane/ethyl acetate as eluent to afford **9l** (63% yield) as a light yellow oil.



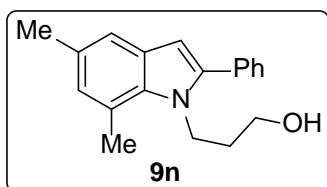
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.68 (d,  $J$  = 7.6 Hz, 1H), 7.52-7.35 (m, 6H), 7.27 (t,  $J$  = 7.6 Hz, 1H), 7.18 (t,  $J$  = 7.6 Hz, 1H), 6.58 (s, 1H), 4.35 (t,  $J$  = 6.8 Hz, 2H), 3.41 (t,  $J$  = 6.0 Hz, 2H), 1.95-1.80 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.2, 137.4, 133.0, 129.3, 128.6, 128.2, 128.0, 121.6, 120.6, 119.9, 110.0, 102.4, 59.5, 40.3, 32.5; IR (neat) 3352, 1462, 1347, 1039, 750 cm<sup>-1</sup>; LRMS (ESI),  $m/z$  274.1 (M+Na)<sup>+</sup>.

Compounds **9m**: prepared according to the general procedure with 6:1 to 4:1 hexane/ethyl acetate as eluent to afford **9m** (67% yield) as a light yellow oil.



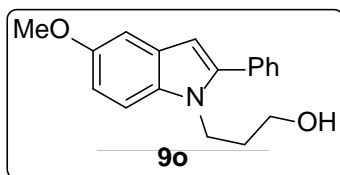
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.60-7.39 (m, 6H), 7.33 (d,  $J$  = 8.4 Hz, 1H), 7.08 (d,  $J$  = 8.4 Hz, 1H), 6.48 (s, 1H), 4.32 (t,  $J$  = 6.8 Hz, 2H), 3.39 (t,  $J$  = 6.0 Hz, 2H), 2.49 (s, 3H), 1.93-1.79 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.3, 135.9, 133.2, 129.3, 129.1, 128.6, 128.5, 128.0, 123.3, 120.3, 109.7, 101.9, 59.6, 40.3, 32.5, 21.4; IR (neat) 3376, 1473, 1333, 1038, 763 cm<sup>-1</sup>; LRMS (ESI),  $m/z$  288.1 (M+Na)<sup>+</sup>.

Compounds **9n**: prepared according to the general procedure with 6:1 to 4:1 hexane/ethyl acetate as eluent to afford **9n** (68% yield) as a light yellow oil.



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.54-7.43 (m, 4H), 7.43-7.38 (m, 1H), 7.27 (s, 1H), 6.82 (s, 1H), 6.47 (s, 1H), 4.47 (t,  $J$  = 7.0 Hz, 2H), 3.24 (t,  $J$  = 6.0 Hz, 2H), 2.73 (s, 3H), 2.41 (s, 3H), 1.70-1.63 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  142.9, 135.0, 133.3, 130.0, 129.6, 129.5, 128.7, 127.9, 126.9, 121.1, 118.4, 103.5, 59.5, 42.2, 34.1, 21.1, 20.0; IR (neat) 3364, 1471, 1322, 1068, 702 cm<sup>-1</sup>; LRMS (ESI),  $m/z$  302.2 (M+Na)<sup>+</sup>.

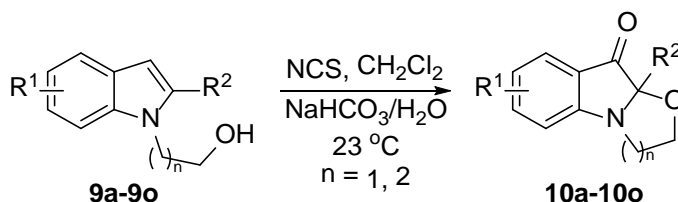
Compounds **9o**: prepared according to the general procedure with 6:1 to 3:1 hexane/ethyl acetate as eluent to afford **9o** (61% yield) as a light yellow oil.



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.51-7.39 (m, 5H), 7.33 (d,  $J$  = 9.0 Hz, 1H), 7.11 (d,  $J$  = 2.0 Hz, 1H), 6.91 (dd,  $J$  = 9.0, 2.0 Hz, 1H),

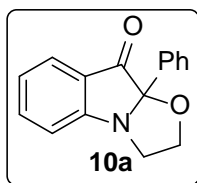
6.48 (s, 1H), 4.31 (t,  $J = 7.0$  Hz, 2H), 3.88 (s, 3H), 3.40 (t,  $J = 6.0$  Hz, 2H), 1.90-1.81 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.3, 141.8, 133.1, 132.8, 129.3, 128.6, 128.5, 128.0, 111.9, 110.8, 102.3, 102.1, 59.6, 55.9, 40.5, 32.6; IR (neat) 3411, 1471, 1216, 1033, 762  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  304.1 ( $\text{M}+\text{Na}$ ) $^+$ .

#### 2.4 General Procedure for the intramolecular *O*-nucleophilic cyclization of 2-aryl indoles:



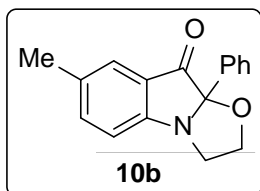
To a solution of indole **9** (0.2 mmol) in freshly distilled  $\text{CH}_2\text{Cl}_2$  (2 mL) at 23  $^\circ\text{C}$  was added  $\text{NaHCO}_3$  (0.6 mmol),  $\text{H}_2\text{O}$  (0.6 mmol), and NCS (0.4 mmol). The mixture was stirred at 23  $^\circ\text{C}$  for 5 h. After the substrate disappeared completely (*via* TLC), the reaction mixture was directly subjected to silica gel chromatography affording the corresponding product **10a-10o**.

Compounds **10a**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10a** (94% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72-7.50 (m, 4H), 7.48-7.29 (m, 3H), 7.18-6.98 (m, 2H), 4.15-3.95 (m, 2H), 3.75-3.61 (m, 1H), 3.59-3.45 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  197.1, 163.9, 137.7, 136.0, 128.9, 128.3, 126.5, 125.6, 122.7, 122.6, 114.7, 99.6, 67.9, 50.1; IR (neat) 1721, 1609, 1473, 1318, 1005  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  274.1 ( $\text{M}+\text{Na}$ ) $^+$ .

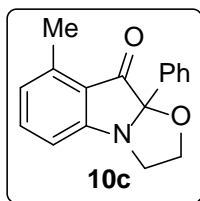
Compounds **10b**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10b** (92% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72-7.56 (m, 2H), 7.49-7.30 (m, 5H), 7.01 (d,  $J = 8.0$  Hz, 1H), 4.12-3.97 (m, 2H), 3.66-3.57 (m, 1H), 3.55-3.46 (m, 1H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  197.3, 162.1, 138.9, 136.3, 132.4, 128.8, 128.3, 126.5, 125.2, 123.0, 114.7, 100.0, 67.8, 50.3, 20.6; IR (neat) 1723, 1618, 1491, 1284, 1005  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  288.1

( $\text{M}+\text{Na}$ ) $^+$ .

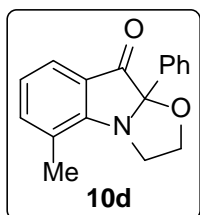
Compounds **10c**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10c** (93% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72-7.57 (m, 2H), 7.50-7.29 (m, 4H), 6.90 (d,  $J$  = 8.4 Hz, 1H), 6.81 (d,  $J$  = 7.2 Hz, 1H), 4.09-3.95 (m, 2H), 3.70-3.60 (m, 1H), 3.59-3.47 (m, 1H), 2.54 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  197.7, 164.4, 141.1, 137.0, 136.3, 128.8, 128.3, 126.5, 124.2, 120.5, 111.9, 99.3, 67.6, 50.2, 18.0; IR (neat) 1715, 1597, 1487, 1311, 1017  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  288.1

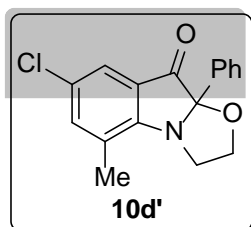
( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **10d**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10d** (91% yield) as a sticky yellow oil.



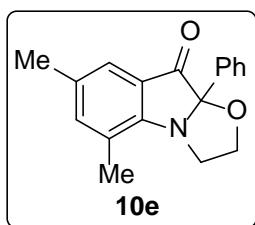
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (dd,  $J$  = 8.0, 2.0 Hz, 2H), 7.47 (d,  $J$  = 7.5 Hz, 1H), 7.44 (d,  $J$  = 7.5 Hz, 1H), 7.40-7.34 (m, 3H), 7.01 (t,  $J$  = 7.5 Hz, 1H), 4.18-4.12 (m, 1H), 4.10-4.04 (m, 1H), 3.70-3.62 (m, 1H), 3.57-3.50 (m, 1H), 2.48 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.4, 162.7, 139.3, 136.3, 128.9, 128.3, 126.5, 126.1, 123.8, 123.1, 123.0, 100.1, 68.3, 50.4, 17.5; IR (neat) 1724, 1594, 1489, 1282, 1011  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  288.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **10d'**: Treatment **9d** (0.2 mmol) with  $\text{NaHCO}_3$  (1.5 mmol),  $\text{H}_2\text{O}$  (1.5 mmol), and NSC (1.0 mmol) in freshly distilled  $\text{CH}_2\text{Cl}_2$  (3 mL) at 23  $^\circ\text{C}$  under air for 24 h. Then, the reaction mixture was directly subjected to silica gel chromatography (10:1 to 8:1 hexane/ethyl acetate) affording the corresponding product **10d'** (93% yield) as a sticky yellow oil.



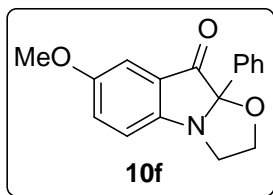
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67-7.64 (m, 2H), 7.44-7.35 (m, 5H), 4.19-4.13 (m, 1H), 4.10-4.04 (m, 1H), 3.68-3.62 (m, 1H), 3.53-3.46 (m, 1H), 2.46 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.2, 161.1, 138.8, 135.8, 129.1, 128.5, 128.4, 127.9, 126.4, 124.9, 122.3, 100.5, 68.4, 50.4, 17.4; IR (neat) 1729, 1599, 1470, 1250, 1005  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  322.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **10e**: prepared according to the general procedure with 8:1 to 6:1 hexane/ethyl acetate as eluent to afford **10e** (95% yield) as a light yellow solid. Melting point: 108-110  $^\circ\text{C}$ .



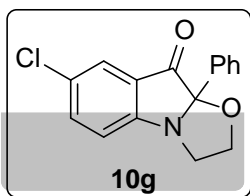
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (s, 1H), 7.67 (s, 1H), 7.40-7.20 (m, 5H), 4.20-4.00 (m, 2H), 3.69-3.55 (m, 1H), 3.54-3.42 (m, 1H), 2.44 (s, 3H), 2.31 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  198.6, 160.8, 140.6, 136.6, 132.9, 128.7, 128.3, 126.4, 125.9, 124.0, 122.5, 100.5, 68.1, 50.6, 20.5, 17.4; IR (neat) 1722, 1619, 1488, 1277, 1018  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  302.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **10f**: prepared according to the general procedure with 8:1 to 6:1 hexane/ethyl acetate as eluent to afford **10f** (91% yield) as a sticky yellow oil.



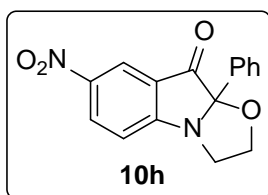
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.66 (d, *J* = 3.0 Hz, 1H), 7.64 (d, *J* = 1.2 Hz, 1H), 7.45-7.30 (m, 3H), 7.26 (dd, *J* = 6.8, 2.8 Hz, 1H), 7.07 (d, *J* = 2.8 Hz, 1H), 7.05 (d, *J* = 8.8 Hz, 1H), 4.15-3.95 (m, 2H), 3.80 (s, 3H), 3.62-3.41 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 197.6, 158.8, 155.9, 136.3, 128.9, 128.3, 127.4, 126.4, 123.4, 116.2, 106.0, 100.5, 67.8, 55.9, 50.6; IR (neat) 1722, 1490, 1278, 1233, 1026 cm<sup>-1</sup>; LRMS (ESI), *m/z* 304.1 (M+Na)<sup>+</sup>.

Compounds **10g**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10g** (83% yield) as a sticky yellow oil.



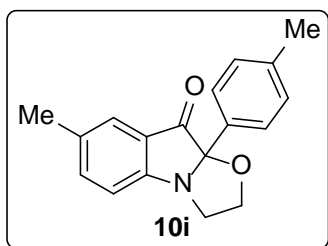
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.68-7.50 (m, 4H), 7.45-7.33 (m, 3H), 7.05 (d, *J* = 8.4 Hz, 1H), 4.12-4.00 (m, 2H), 3.67-3.48 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.9, 162.2, 137.5, 135.5, 129.1, 128.5, 128.1, 126.4, 125.0, 123.9, 116.0, 100.0, 68.0, 50.1; IR (neat) 1731, 1603, 1464, 1184, 1003 cm<sup>-1</sup>; LRMS (ESI), *m/z* 308.0 (M+Na)<sup>+</sup>.

Compounds **10h**: prepared according to the general procedure with 6:1 to 4:1 hexane/ethyl acetate as eluent to afford **10h** (80% yield) as a sticky yellow oil.



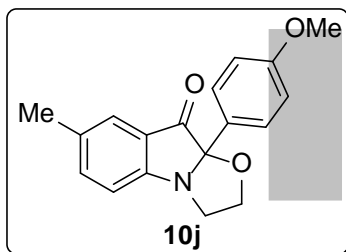
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.48 (d, *J* = 2.5 Hz, 1H), 8.45 (d, *J* = 2.0 Hz, 1H), 7.60-7.50 (m, 2H), 7.40-7.37 (m, 3H), 7.13 (d, *J* = 9.0 Hz, 1H), 4.22-4.15 (m, 1H), 4.14-4.08 (m, 1H), 3.76-3.69 (m, 1H), 3.68-3.62 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.5, 166.9, 143.0, 134.2, 132.8, 129.5, 128.7, 126.3, 122.4, 122.2, 114.1, 100.1, 68.4, 49.0; IR (neat) 1731, 1614, 1324, 1161, 819 cm<sup>-1</sup>; LRMS (ESI), *m/z* 319.1 (M+Na)<sup>+</sup>.

Compounds **10i**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10i** (93% yield) as a sticky yellow oil.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.52 (d, *J* = 8.0 Hz, 2H), 7.44 (s, 1H), 7.42 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 6.99 (d, *J* = 8.0 Hz, 1H), 4.10-3.92 (m, 2H), 3.64-3.56 (m, 1H), 3.54-3.45 (m, 1H), 2.35 (s, 3H), 2.34 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 197.4, 162.1, 138.8, 138.7, 133.3, 132.3, 129.1, 126.4, 125.2, 123.0, 114.6, 100.0, 67.7, 50.3, 21.2, 20.6; IR (neat) 1723, 1618, 1491, 1283, 820 cm<sup>-1</sup>; LRMS (ESI), *m/z* 302.1 (M+Na)<sup>+</sup>.

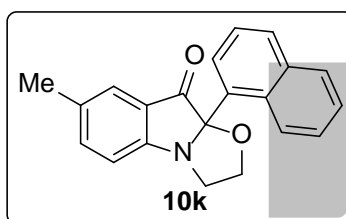
Compounds **10j**: prepared according to the general procedure with 8:1 to 6:1 hexane/ethyl acetate as eluent to afford **10j** (82% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.59-7.55 (m, 2H), 7.44 (s, 1H), 7.42 (s, 1H), 6.99 (d,  $J = 8.0$  Hz, 1H), 6.90 (d,  $J = 8.5$  Hz, 2H), 4.08-3.97 (m, 2H), 3.81 (s, 3H), 3.63-3.56 (m, 1H), 3.54-3.47 (m, 1H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  197.5, 162.0, 160.2, 138.8, 132.3, 128.2, 127.8, 125.2, 123.0, 114.6, 113.8, 99.9, 67.7, 55.3, 50.4, 20.6; IR (neat) 1722, 1616, 1490, 1248, 1030  $\text{cm}^{-1}$ ; LRMS

(ESI),  $m/z$  318.1 ( $\text{M}+\text{Na}$ ) $^+$ .

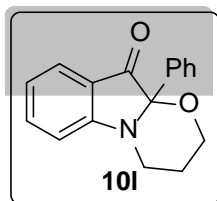
Compounds **10k**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10k** (88% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d,  $J = 7.2$  Hz, 1H), 7.95-7.75 (m, 3H), 7.60-7.30 (m, 5H), 7.07 (d,  $J = 8.4$  Hz, 1H), 4.21 (dd,  $J = 7.2, 2.0$  Hz, 1H), 4.03 (q,  $J = 8.0$  Hz, 1H), 3.78-3.68 (m, 1H), 3.41 (dd,  $J = 18.8, 8.8$  Hz, 1H), 2.40 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.5, 160.6, 138.8, 134.3, 132.7, 132.1, 130.9, 130.1,

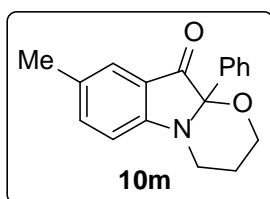
128.6, 126.1, 126.0, 125.7, 125.6, 125.5, 124.8, 123.0, 114.6, 101.0, 67.5, 49.5, 20.7; IR (neat) 1727, 1617, 1490, 1284, 996  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  338.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **10l**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10l** (80% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67-7.50 (m, 4H), 7.46-7.36 (m, 3H), 6.92 (d,  $J = 8.5$  Hz, 1H), 6.81 (t,  $J = 7.5$  Hz, 1H), 3.97-3.83 (m, 3H), 3.47-3.41 (m, 1H), 2.06-1.97 (m, 1H), 1.44-1.38 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.1, 160.8, 137.7, 133.7, 129.0, 127.6, 126.4, 118.8, 109.2, 90.4, 62.8, 39.8, 24.5; IR (neat) 1720, 1614, 1480, 1320, 1026  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  288.1 ( $\text{M}+\text{Na}$ ) $^+$ .

Compounds **10m**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10m** (81% yield) as a sticky yellow oil.

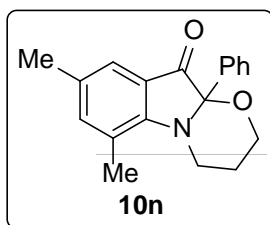


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (s, 1H), 7.57 (s, 1H), 7.45-7.34 (m, 5H), 6.84 (d,  $J = 8.5$  Hz, 1H), 3.96-3.80 (m, 3H), 3.41 (dt,  $J = 13.5, 3.5$  Hz, 1H), 2.29 (s, 3H), 2.06-1.93 (m, 1H), 1.38 (dd,  $J = 13.5, 1.0$  Hz, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.2, 159.2, 138.8, 134.0, 128.9, 128.8, 128.3, 127.6, 126.0, 118.9, 109.2, 90.7, 62.8, 40.0, 24.3, 20.4; IR (neat) 1722,

1622, 1495, 1288, 1026  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  302.1 ( $\text{M}+\text{Na}$ ) $^+$ .

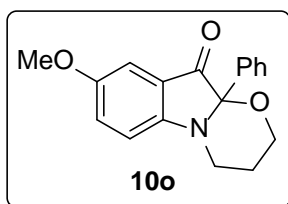
Compounds **10n**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as

eluent to afford **10n** (87% yield) as a sticky yellow oil.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61 (d,  $J = 1.0$  Hz, 1H), 7.60 (s, 1H), 7.44-7.33 (m, 3H), 7.28 (s, 1H), 7.13 (s, 1H), 4.31 (d,  $J = 14.0$  Hz, 1H), 3.94-3.82 (m, 2H), 3.57-3.50 (m, 1H), 2.50 (s, 3H), 2.24 (s, 3H), 2.03-1.92 (m, 1H), 1.49-1.42 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.1, 157.6, 142.2, 134.3, 128.9, 128.8, 127.5, 123.6, 120.8, 120.0, 91.0, 62.3, 41.3, 25.1, 20.4, 20.1; IR (neat) 1721, 1621, 1490, 1283, 1028  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  316.1 ( $\text{M}+\text{Na}$ ) $^+$ .

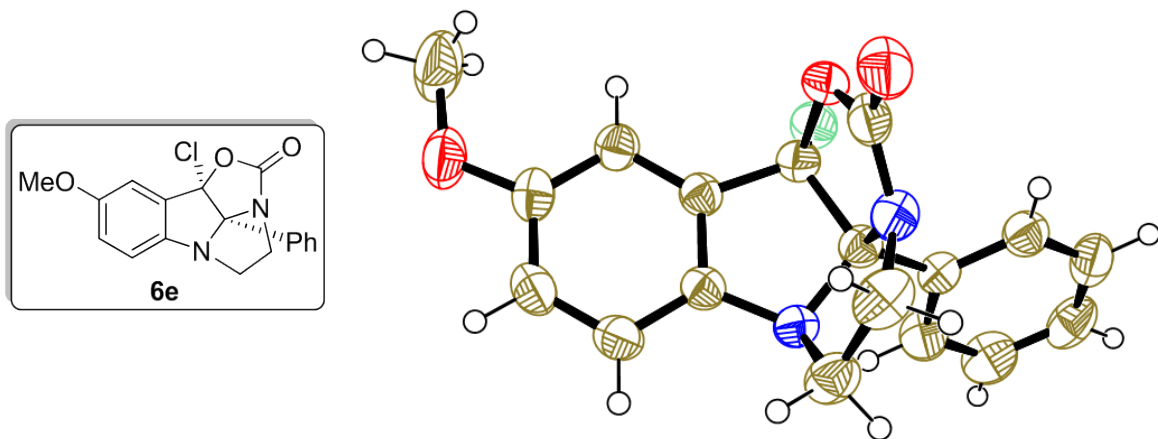
Compounds **10o**: prepared according to the general procedure with 10:1 to 8:1 hexane/ethyl acetate as eluent to afford **10o** (83% yield) as a sticky yellow oil.



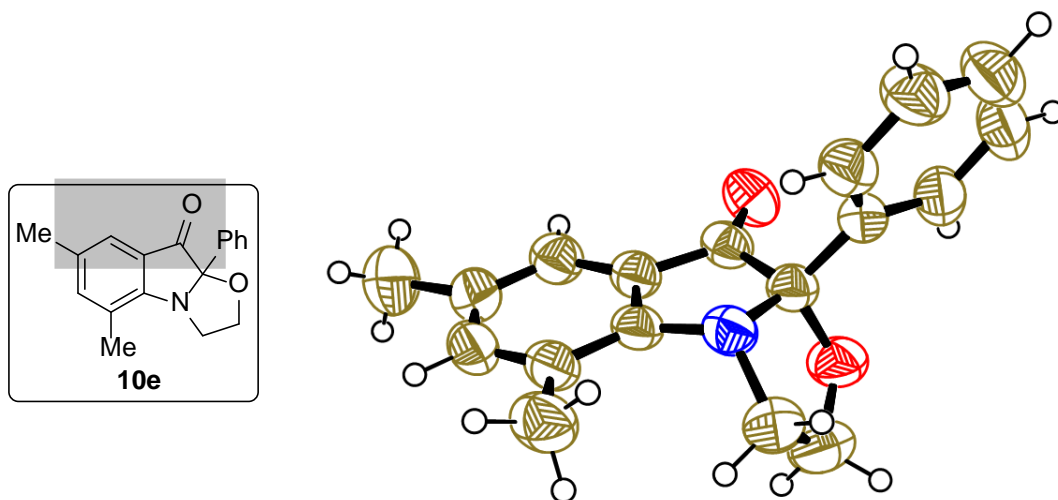
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (s, 1H), 7.58 (s, 1H), 7.46-7.34 (m, 3H), 7.20 (dd,  $J = 9.0, 2.5$  Hz, 1H), 7.07 (d,  $J = 2.5$  Hz, 1H), 6.89 (d,  $J = 8.5$  Hz, 1H), 3.95-3.90 (m, 1H), 3.89-3.81 (m, 2H), 3.77 (s, 3H), 3.41 (dt,  $J = 13.5, 3.0$  Hz, 1H), 2.05-1.93 (m, 1H), 1.38 (d,  $J = 13.0$  Hz, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.4, 156.6, 153.1, 134.0, 128.9, 127.7, 127.5, 118.9, 110.7, 107.1, 91.0, 62.8, 55.9, 40.1, 24.1; IR (neat) 1716, 1494, 1280, 1237, 1026  $\text{cm}^{-1}$ ; LRMS (ESI),  $m/z$  318.1 ( $\text{M}+\text{Na}$ ) $^+$ .



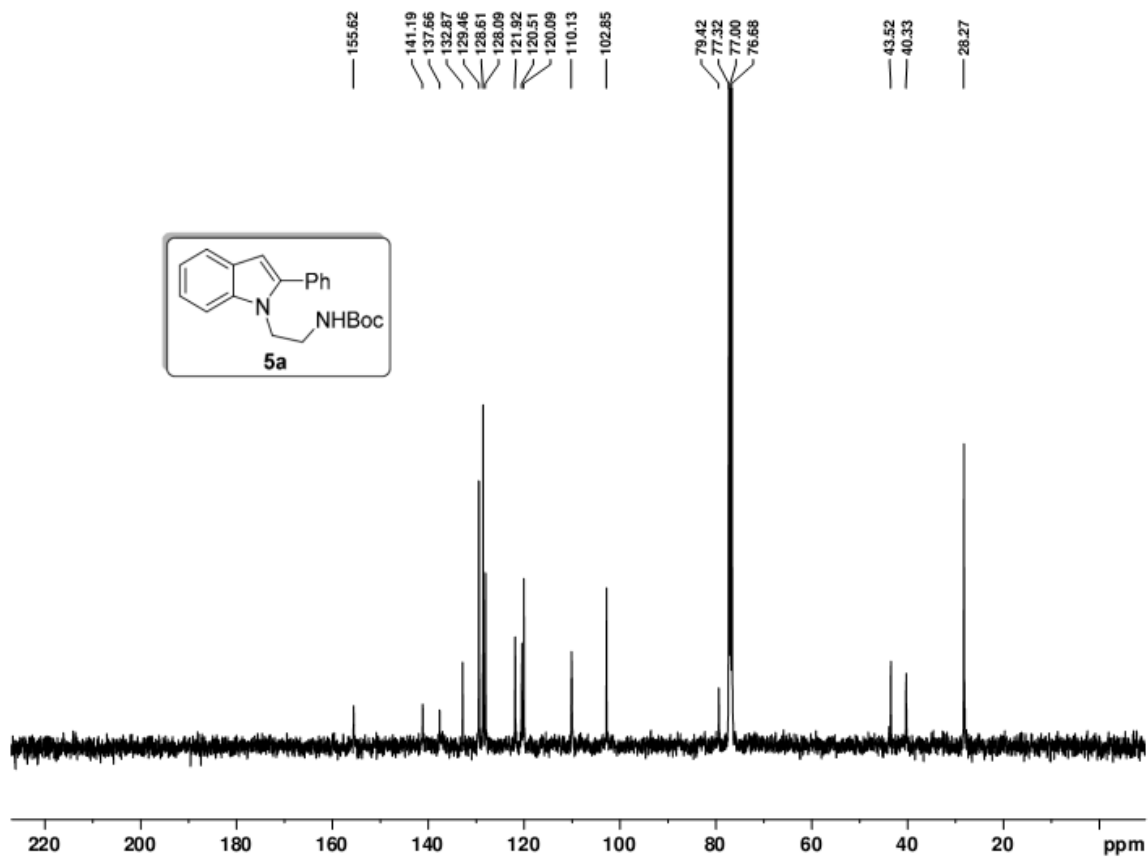
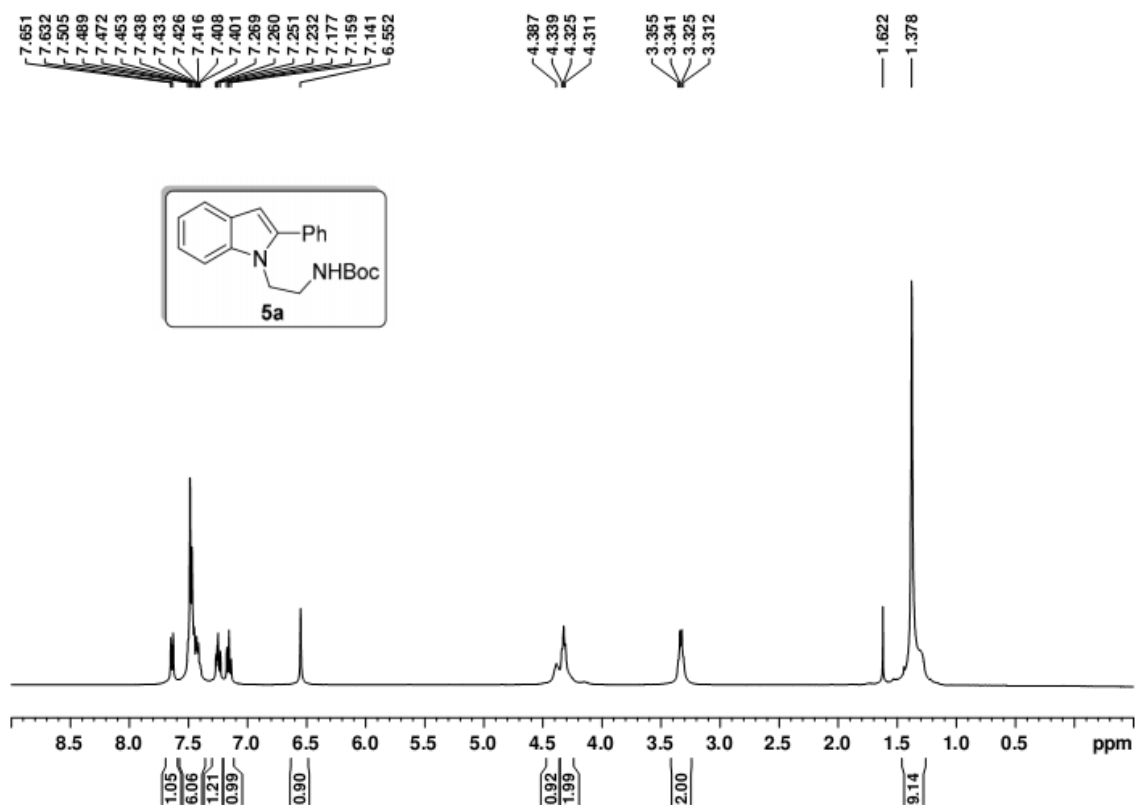
### 3. X-Ray Ellipsoid Plots of 6e and 10e:

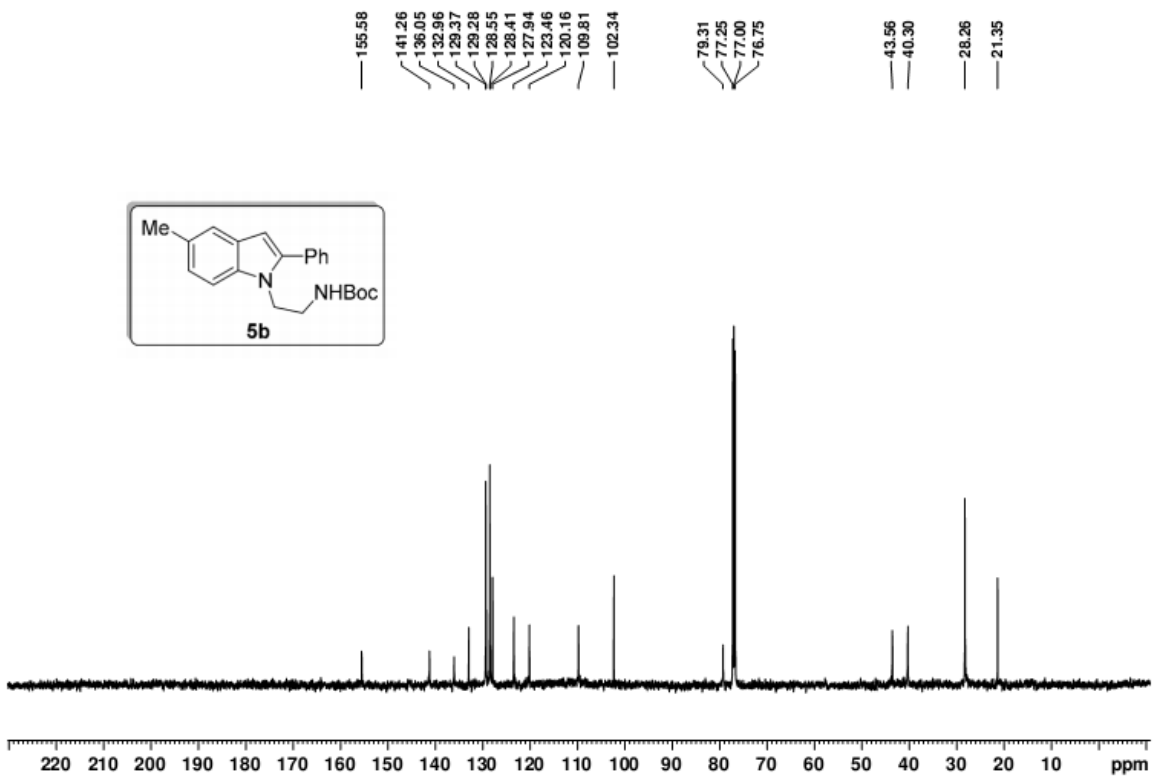
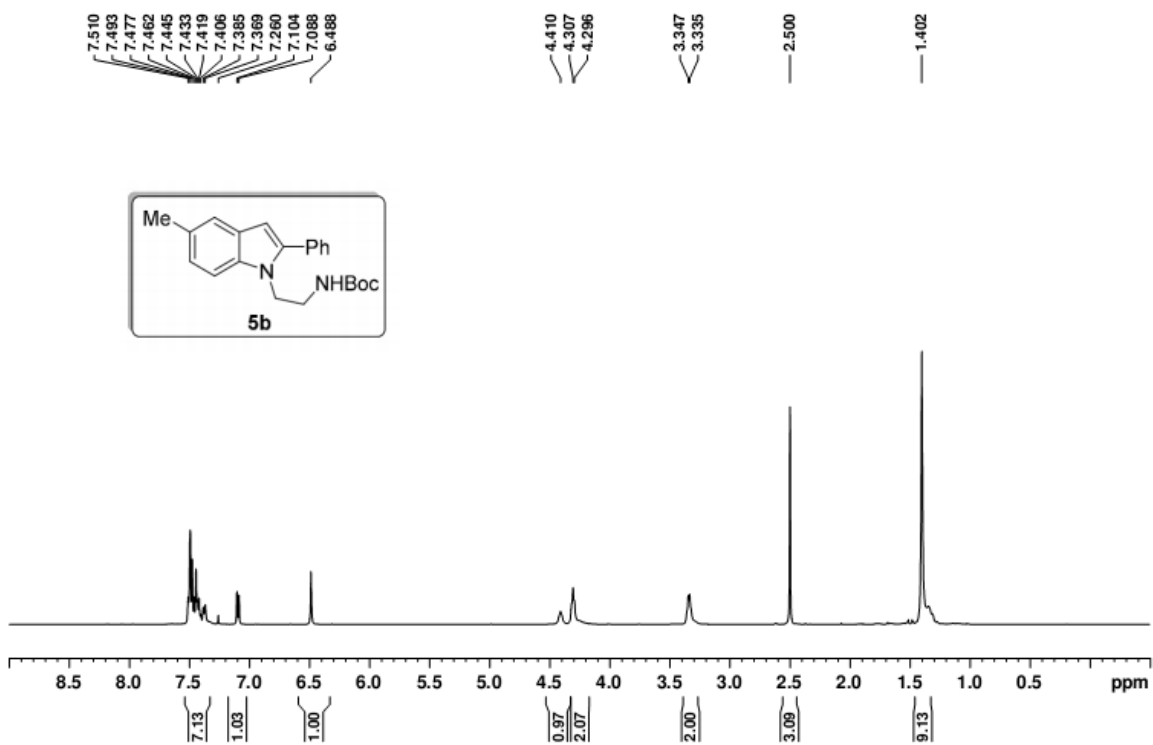


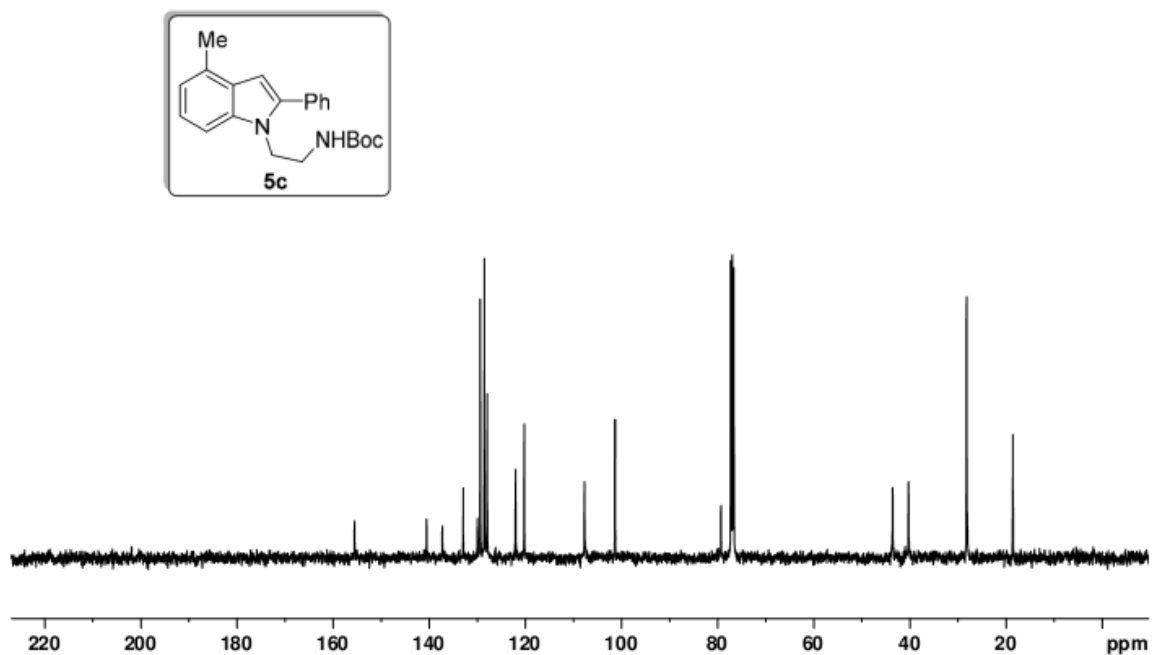
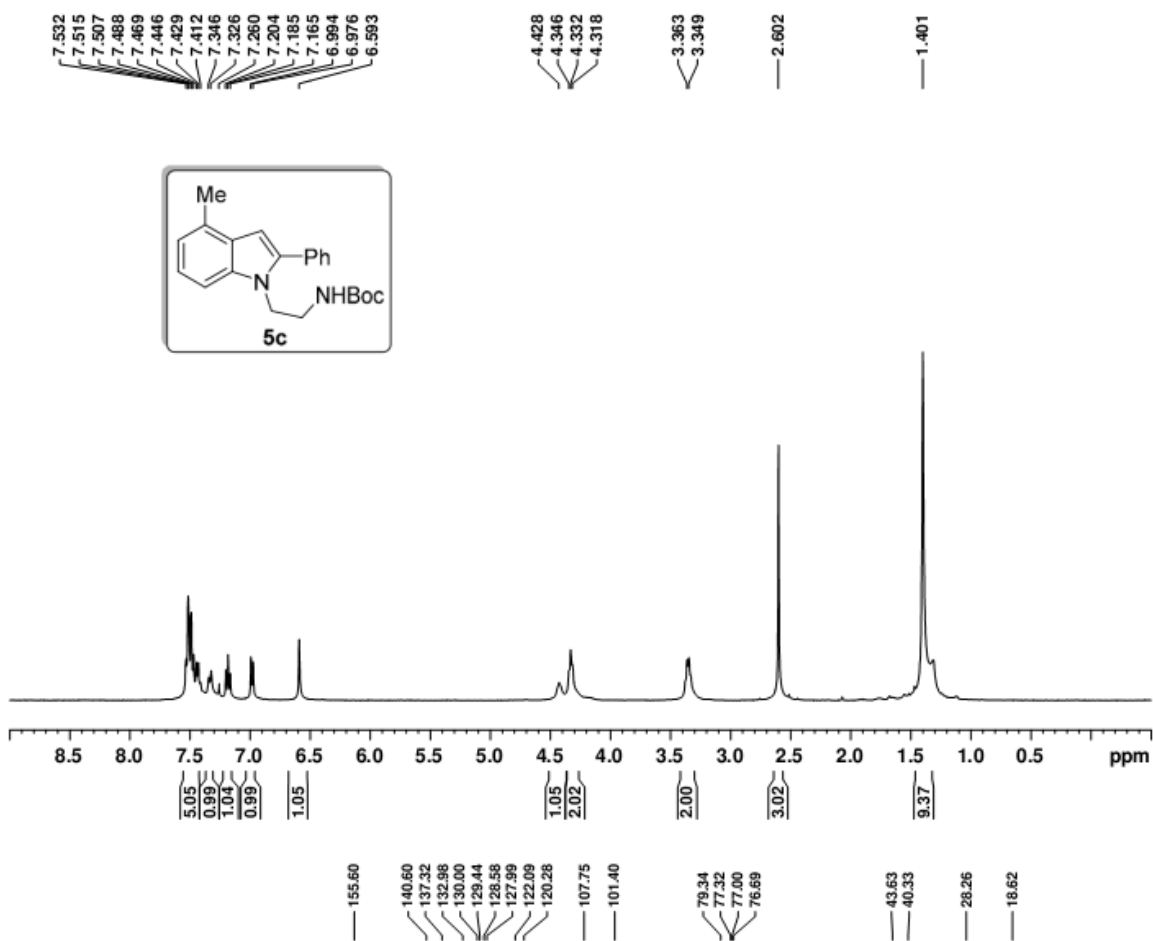
The structure of our synthetic polycyclic compound **6e** was corroborated by single-crystal. The crystal structure has been deposited at the Cambridge Crystallographic Data Centre and allocated the deposition number: 982161.

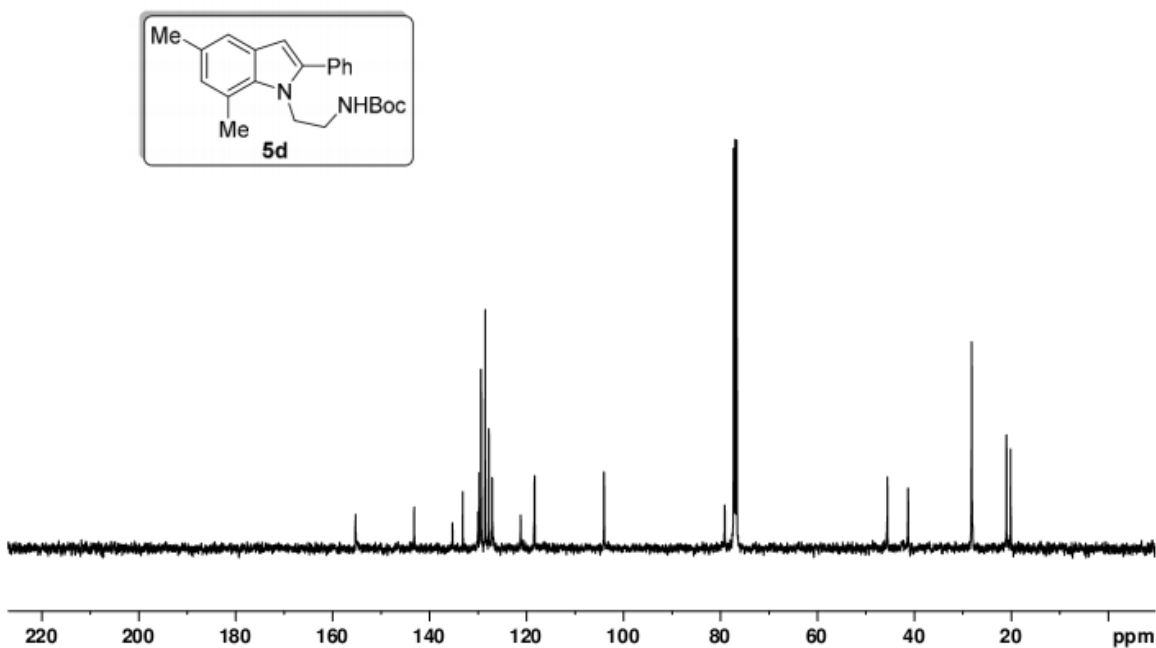
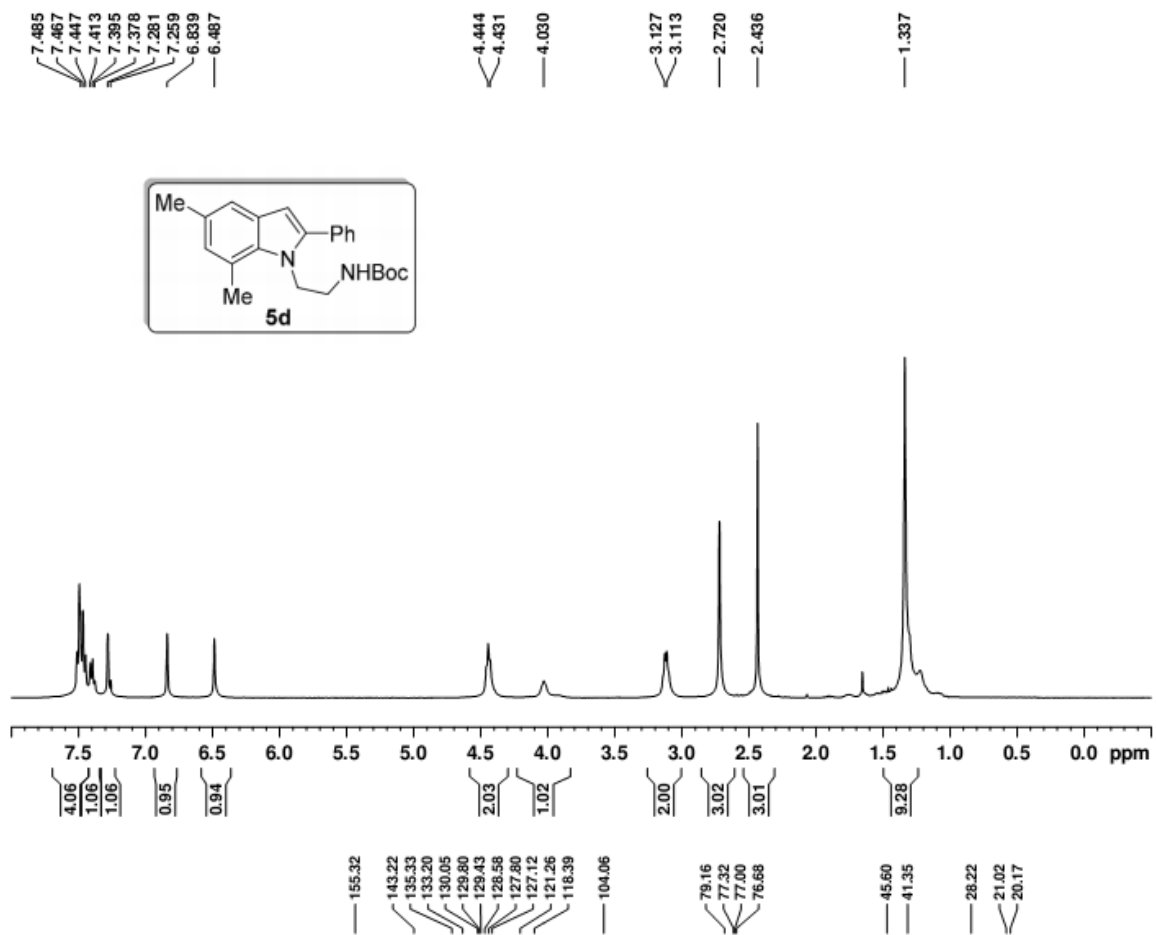


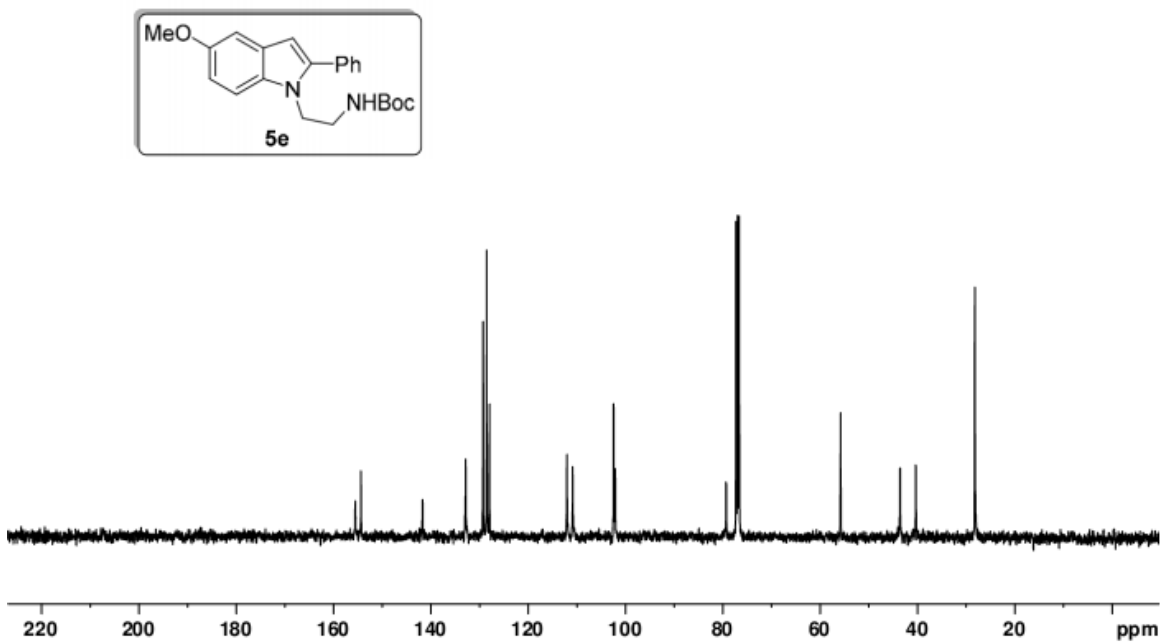
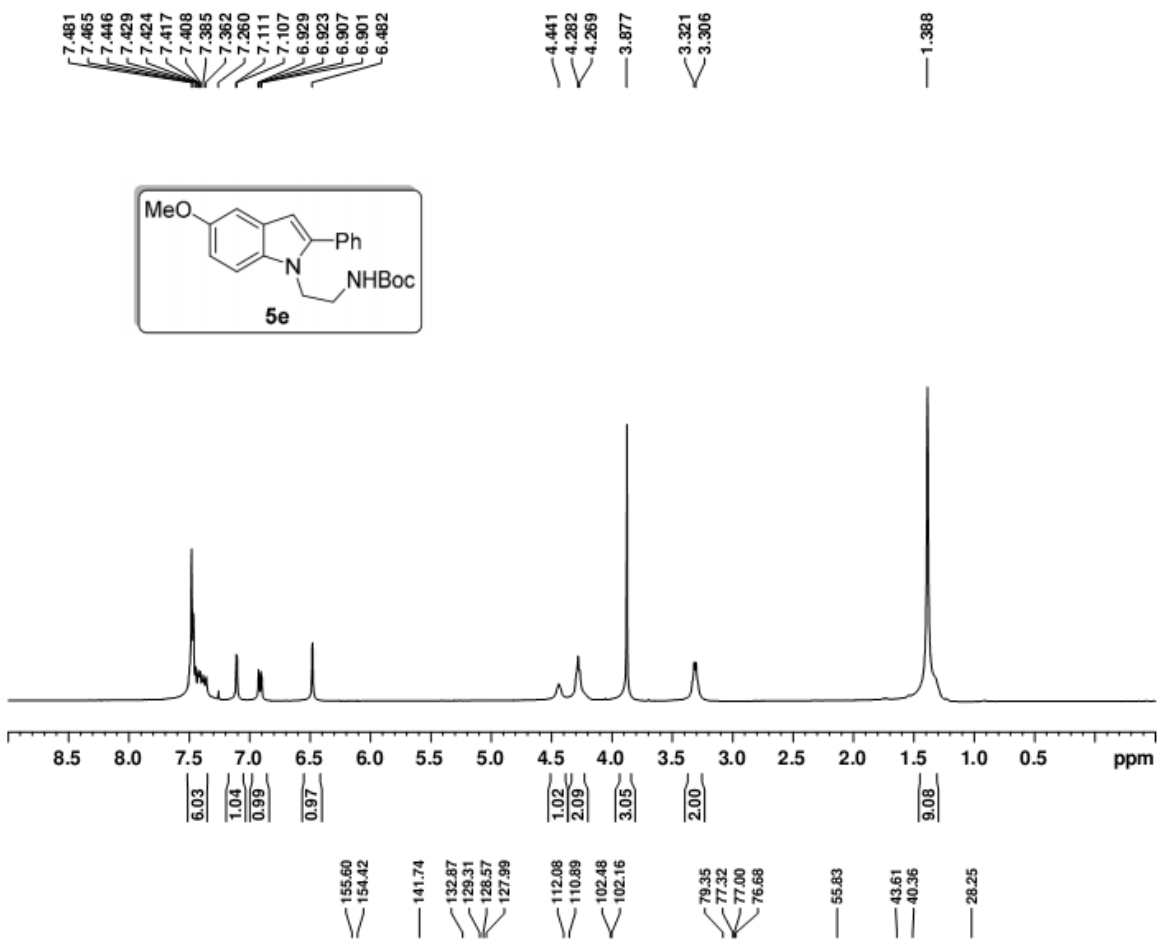
The structure of our synthetic polycyclic compound **10e** was corroborated by single-crystal. The crystal structure has been deposited at the Cambridge Crystallographic Data Centre and allocated the deposition number: 983003.

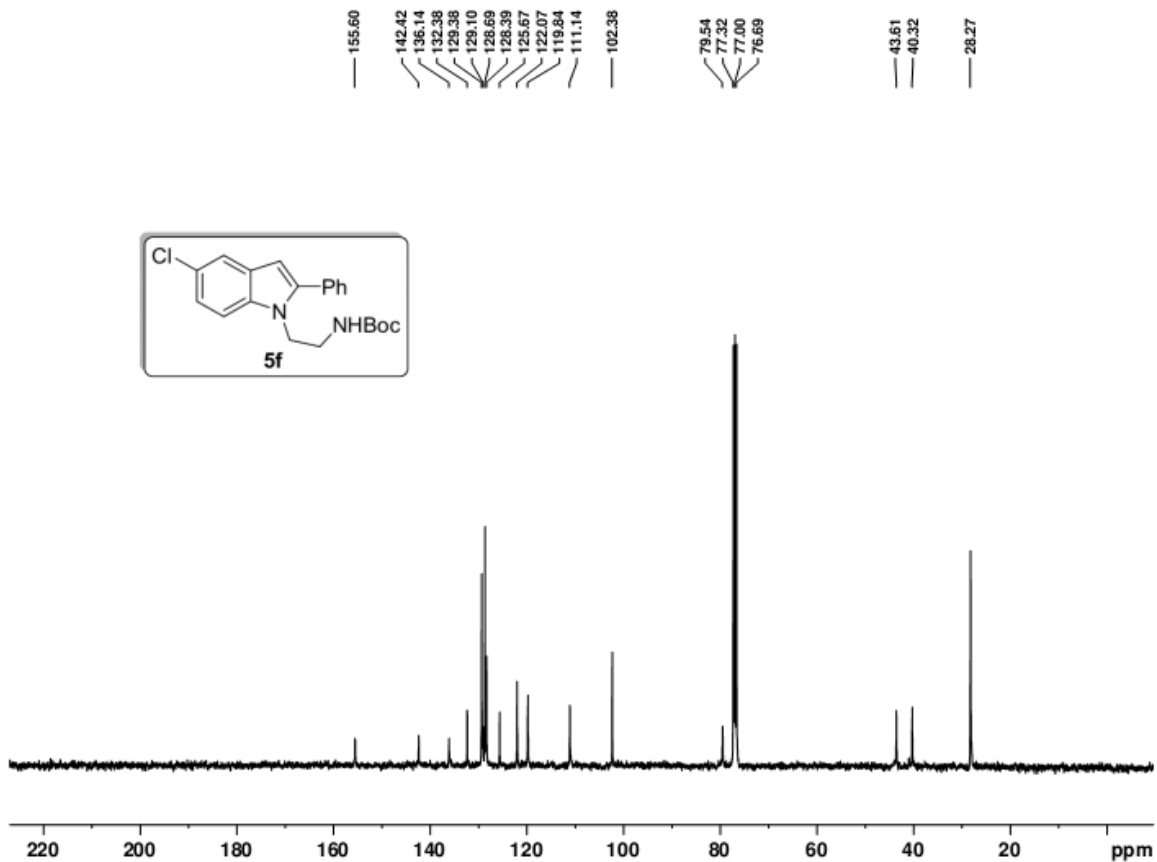
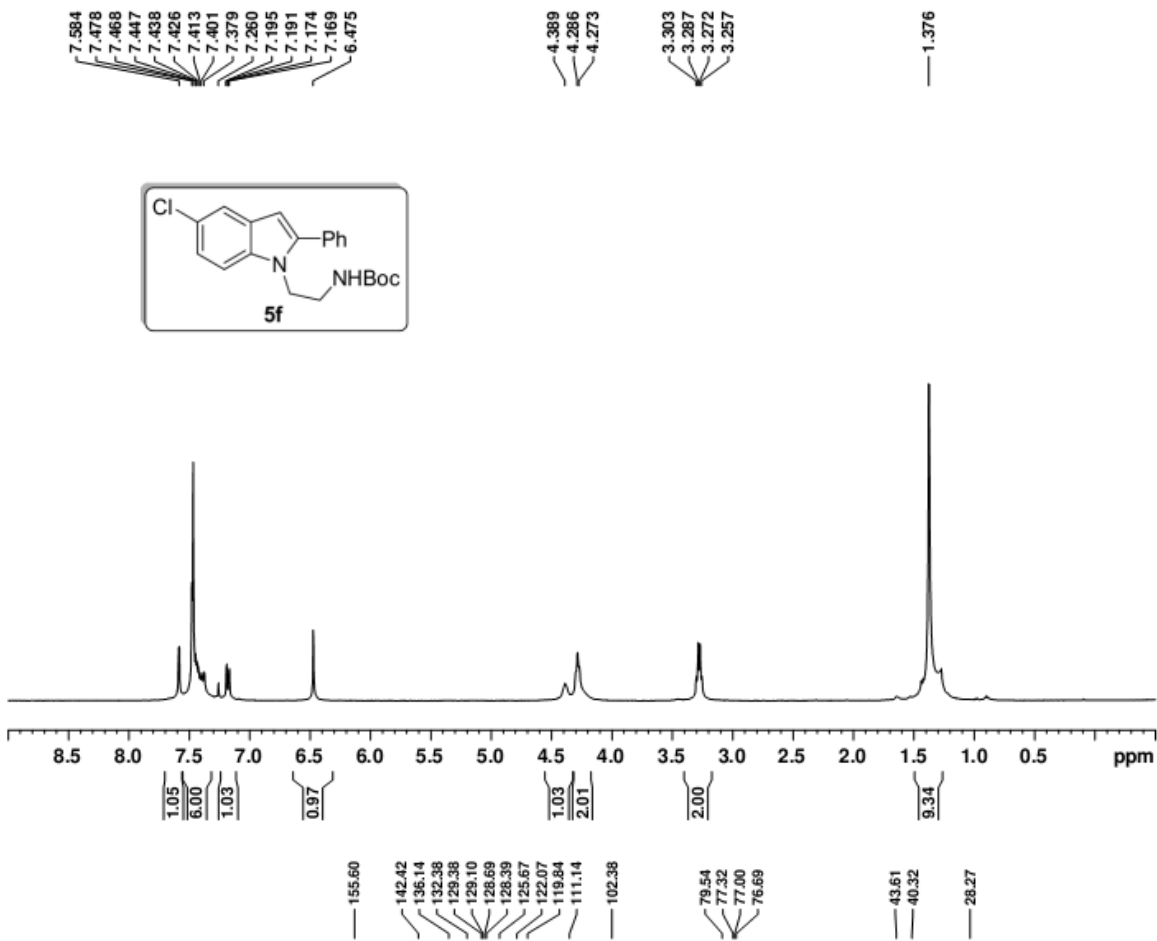


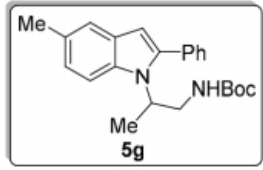
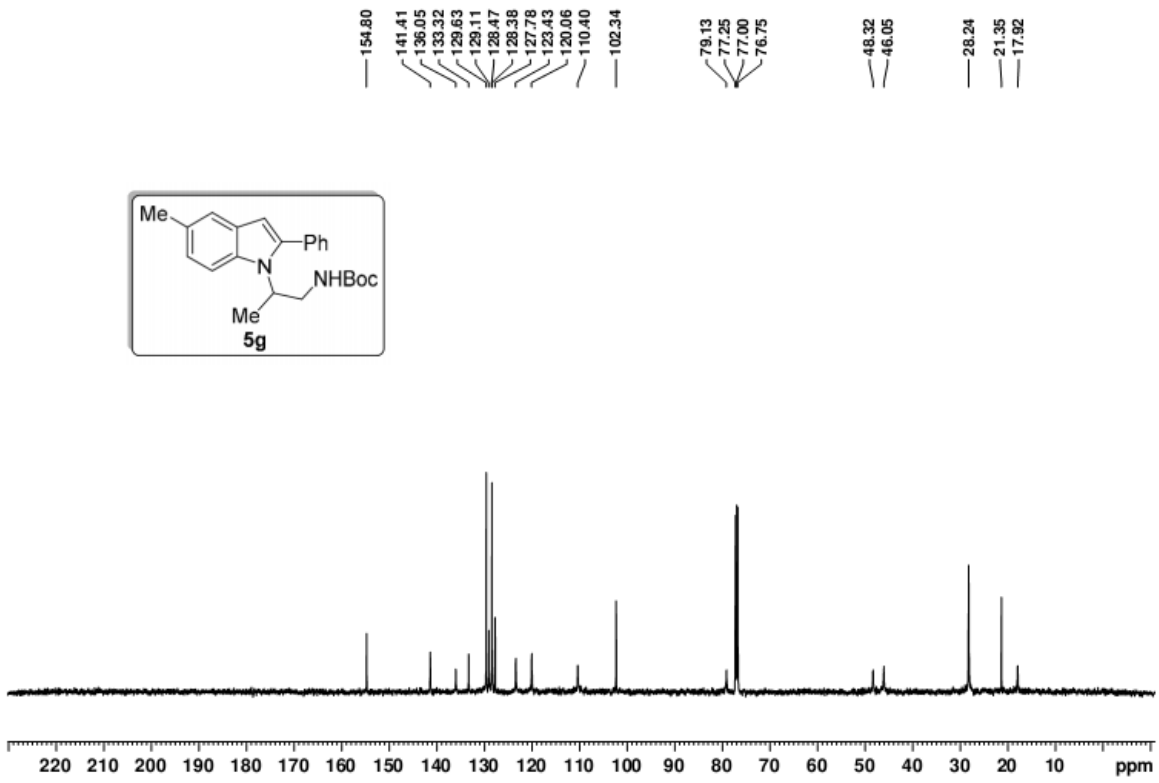
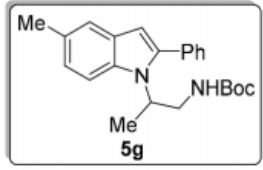
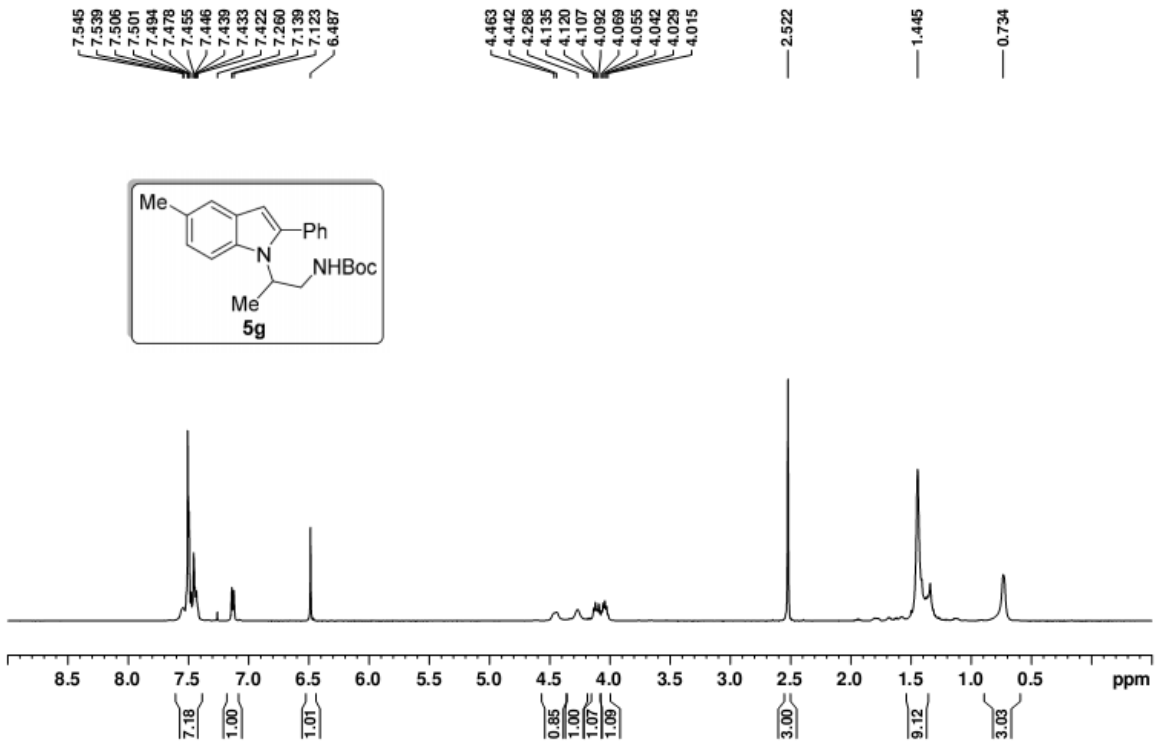




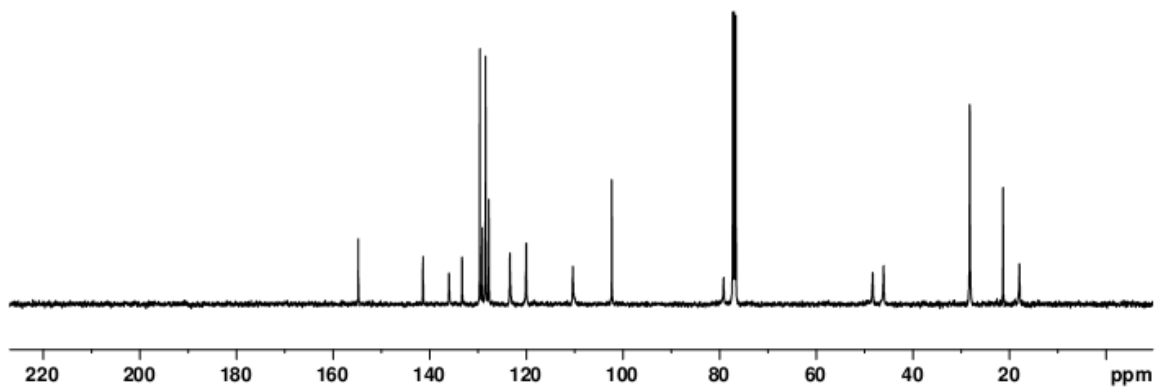
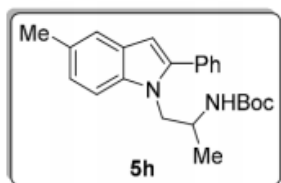
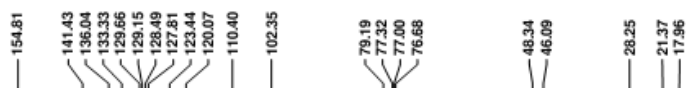
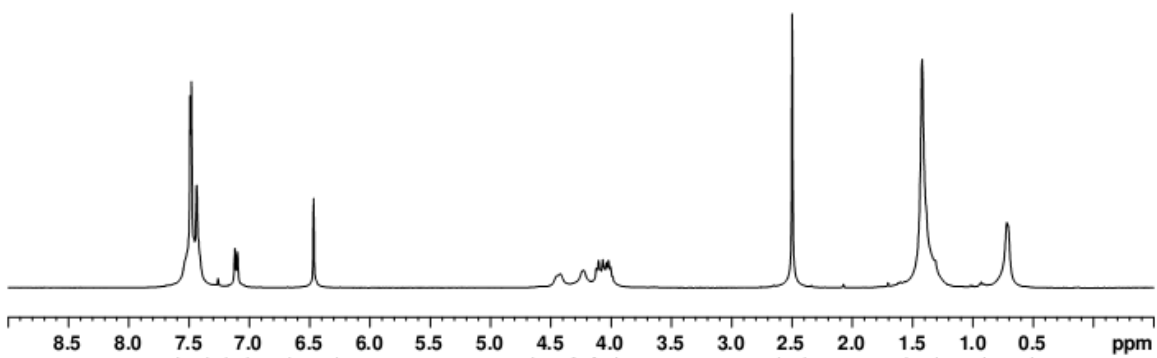
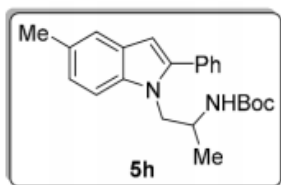
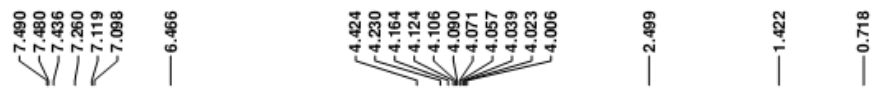


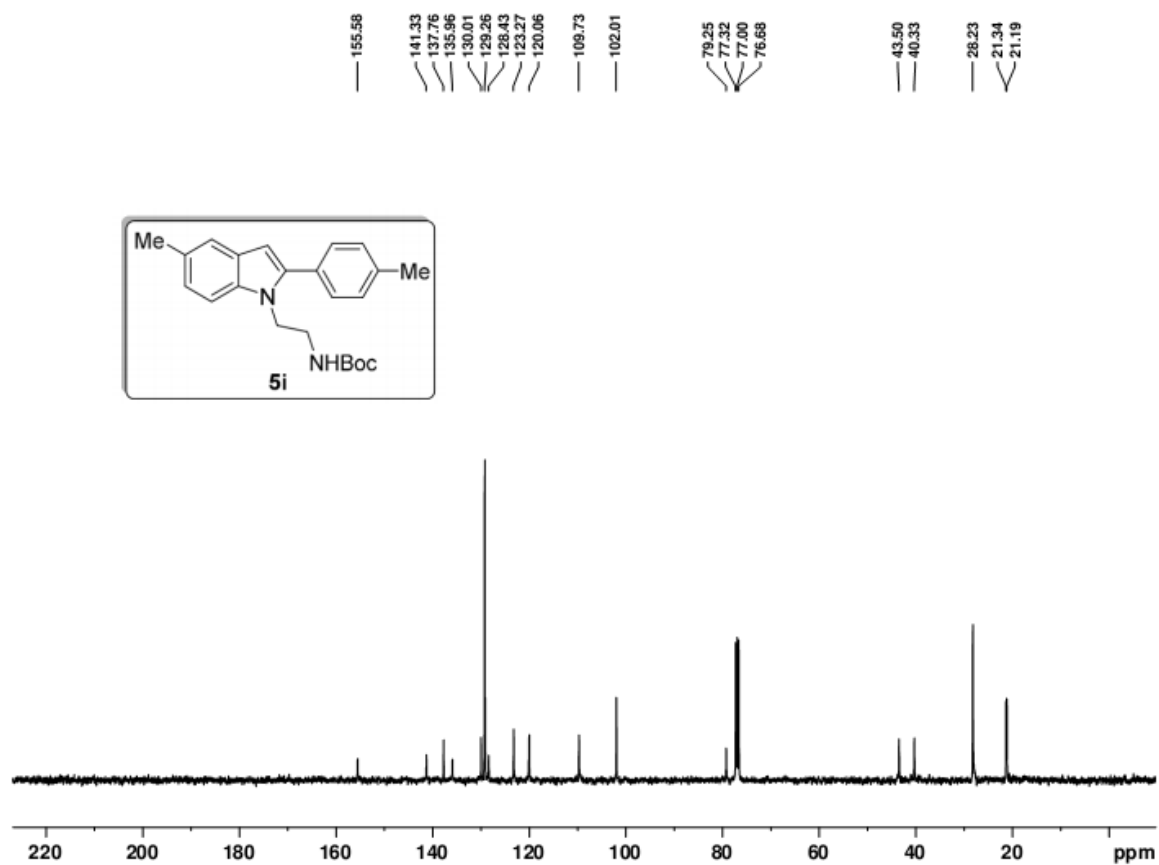
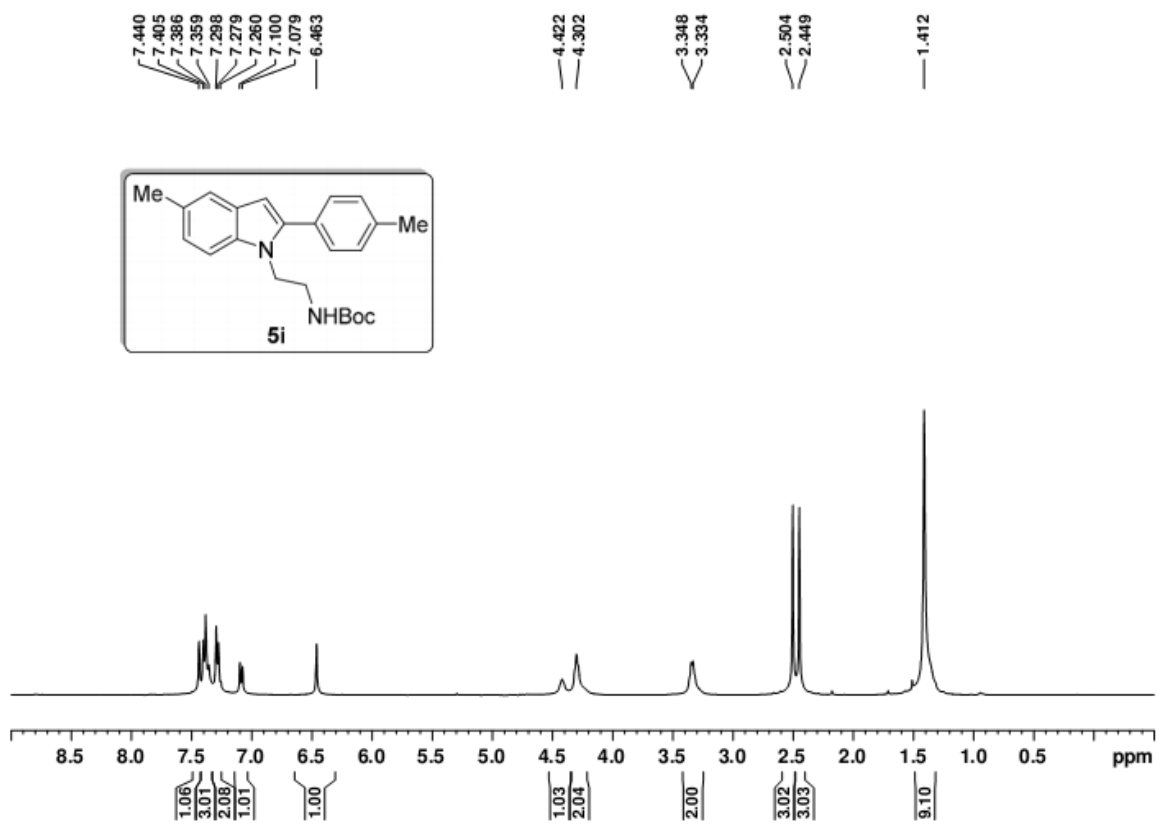


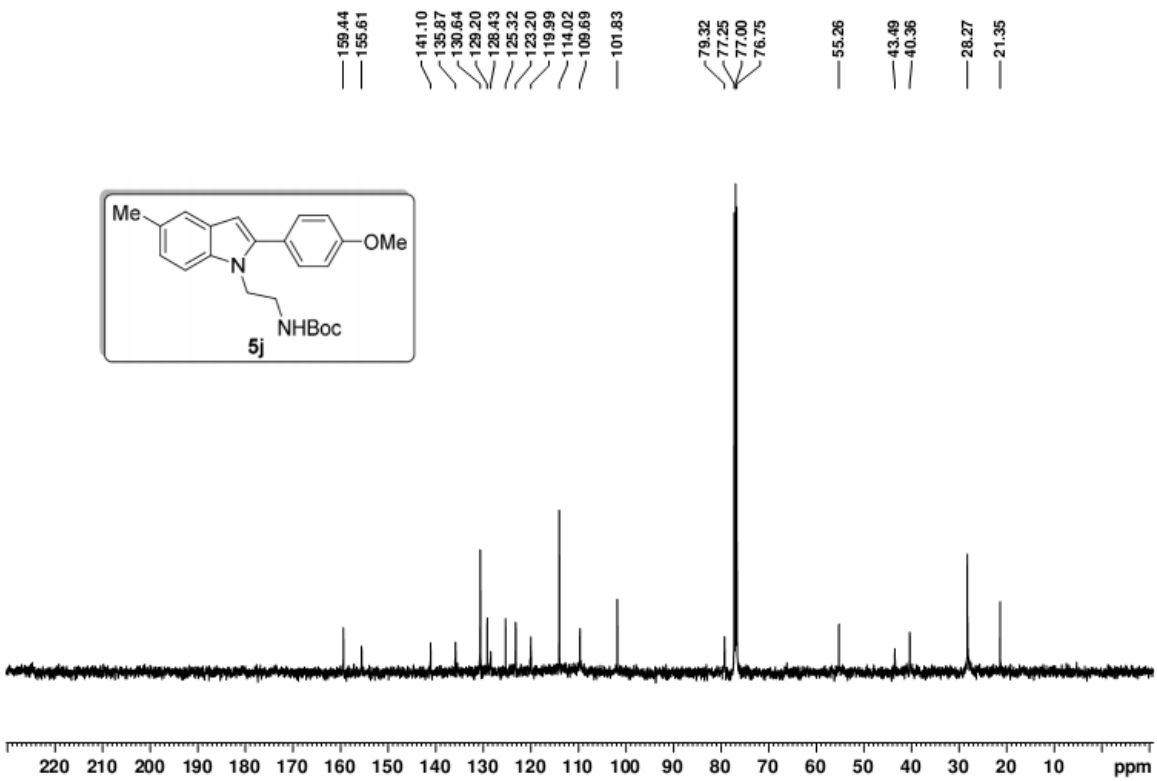
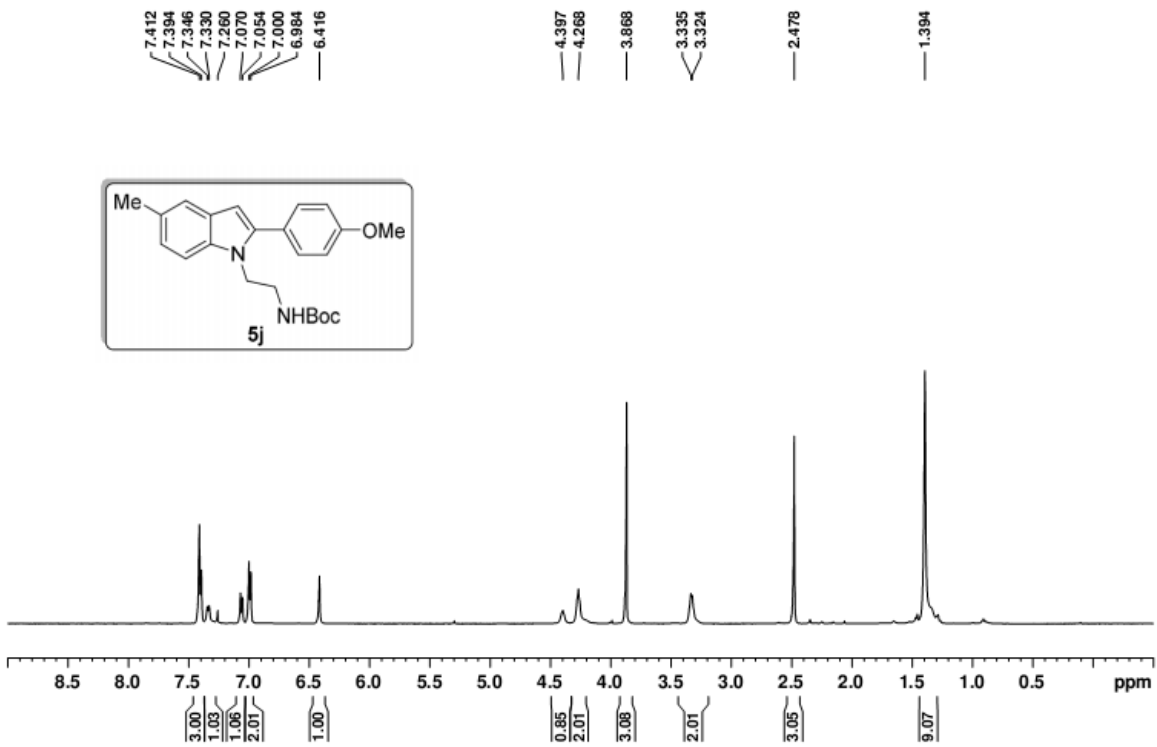


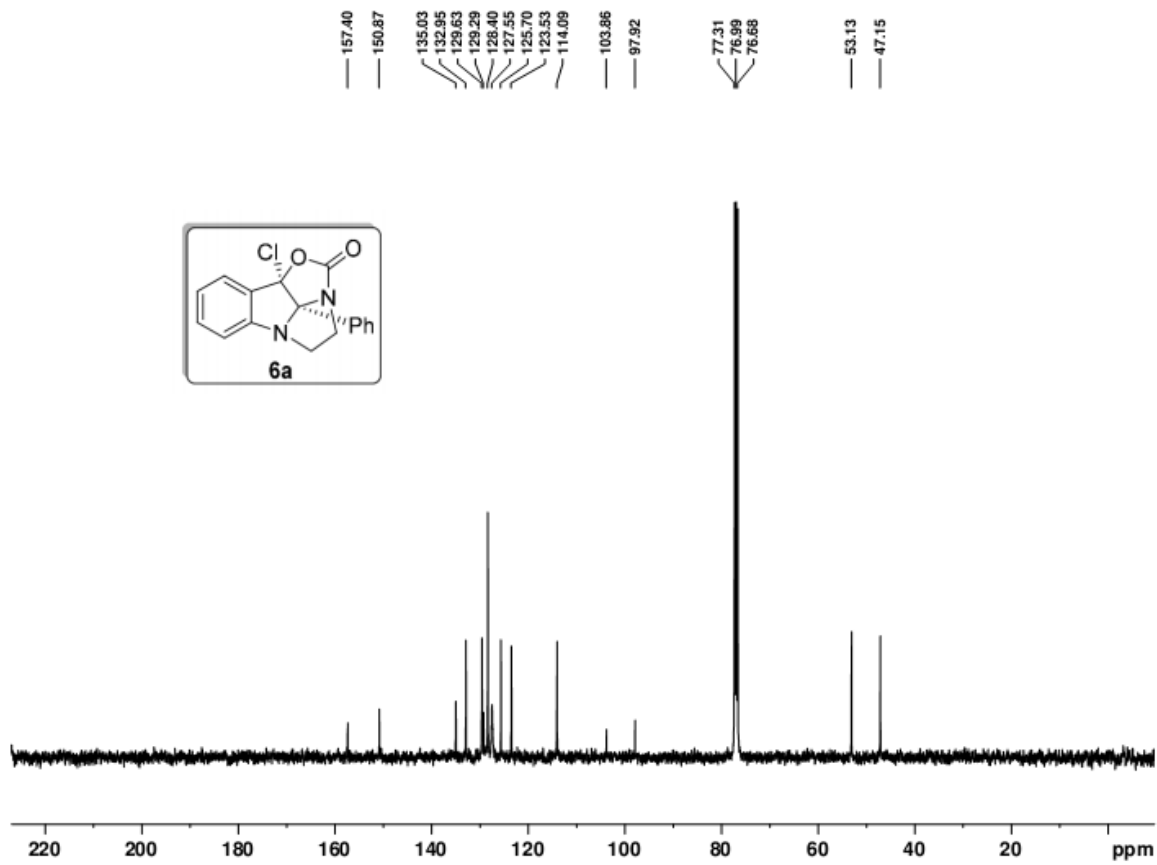
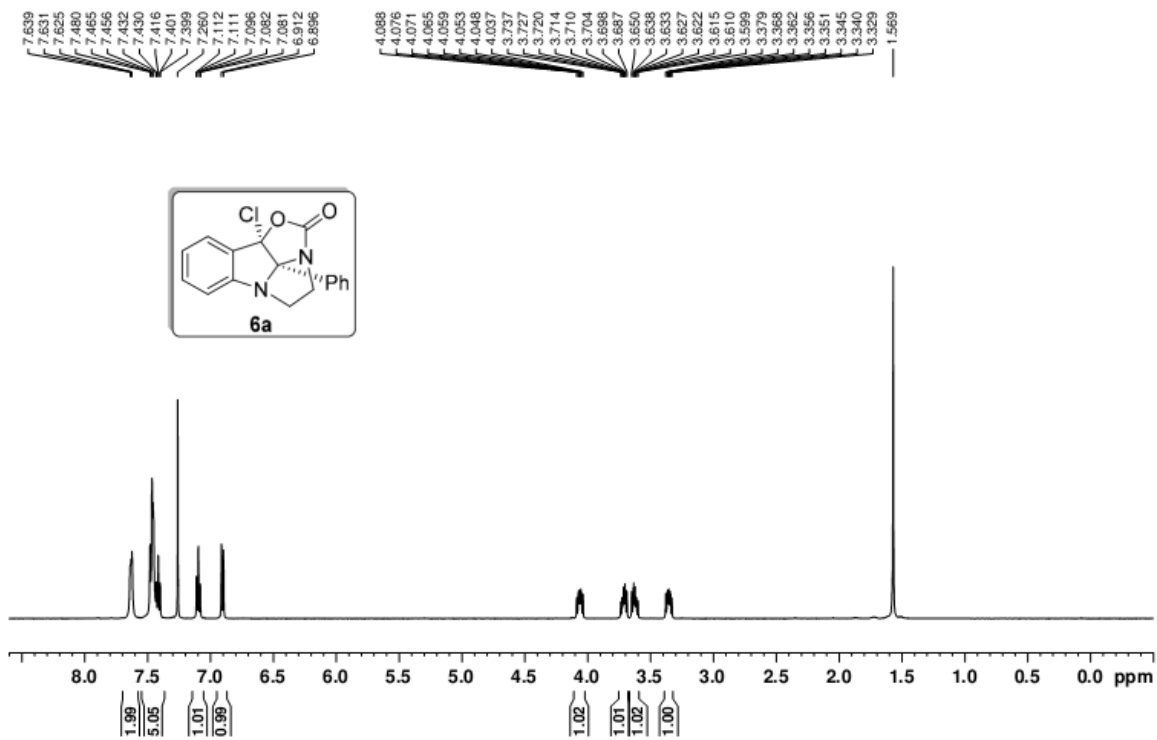




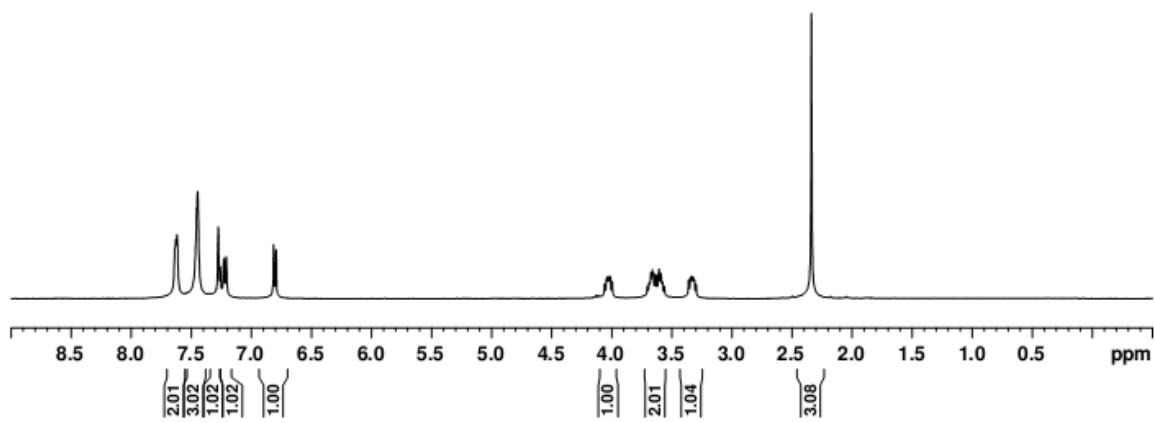
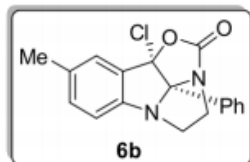




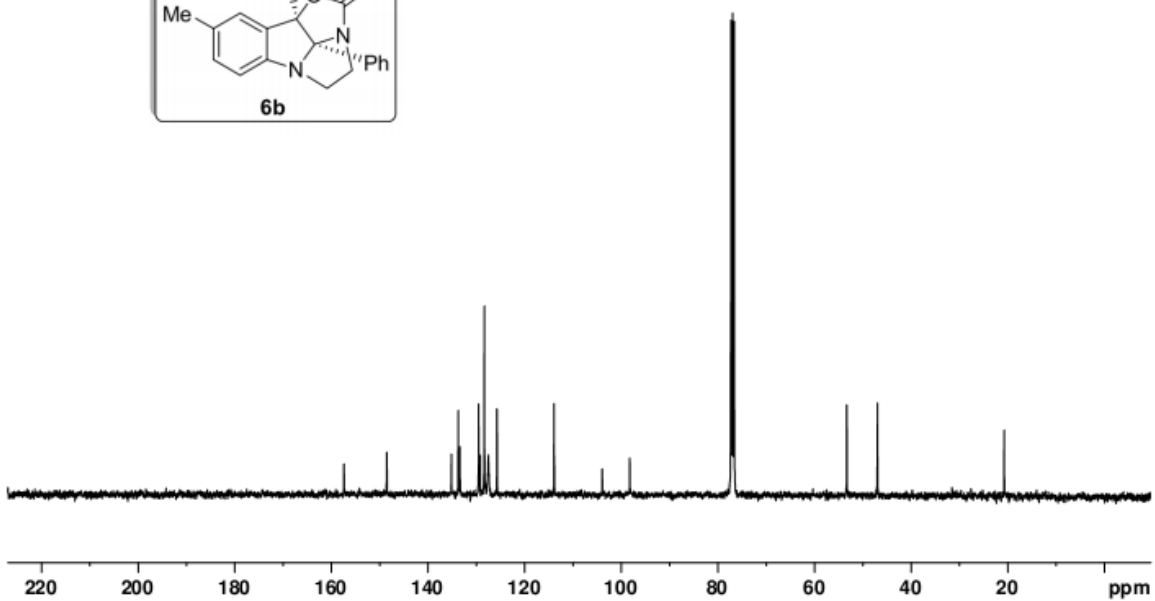
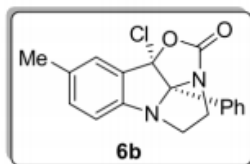


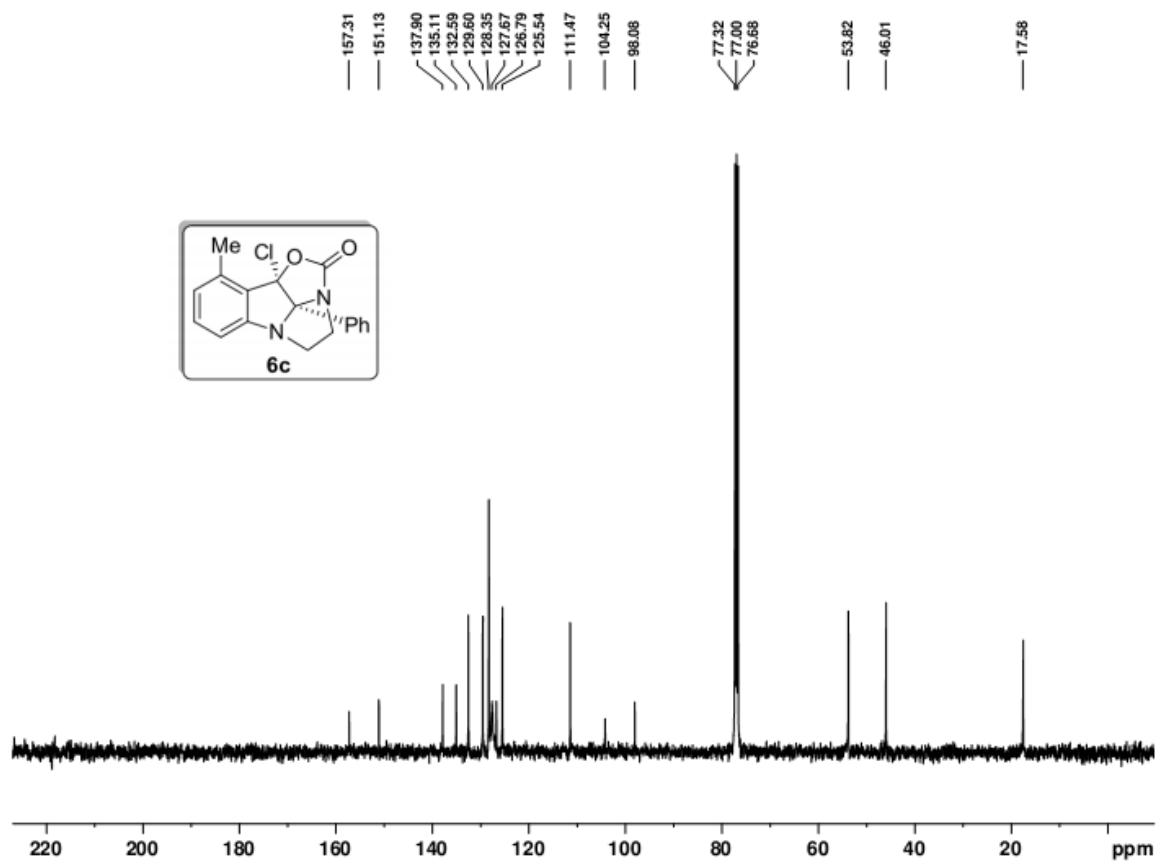
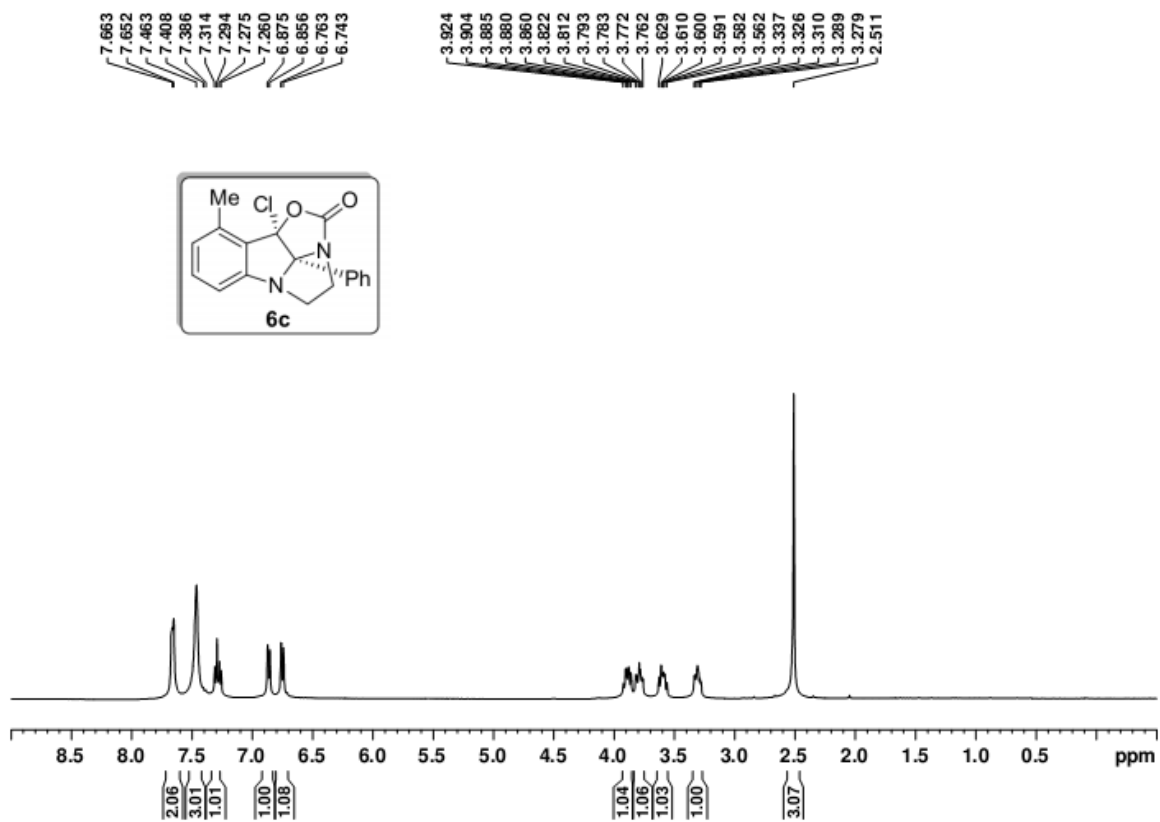


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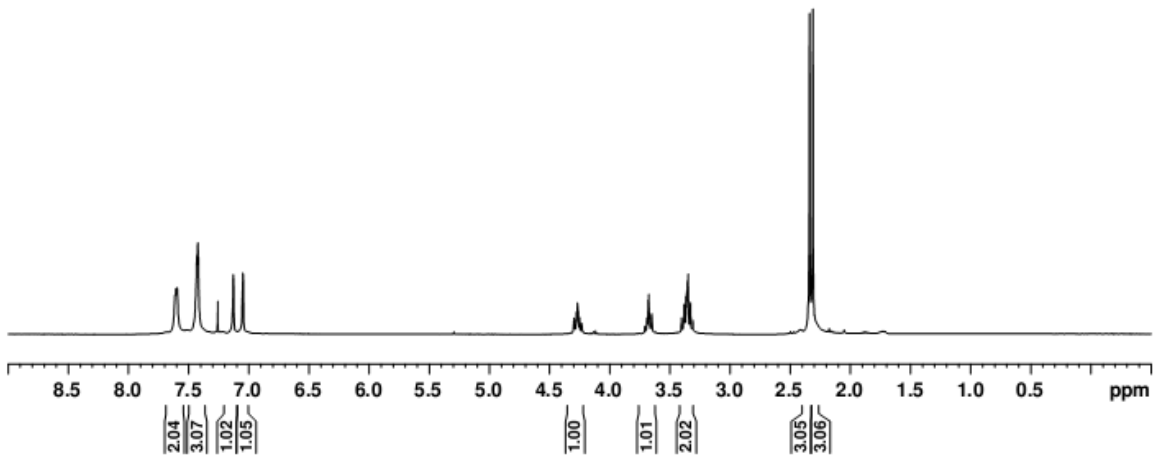
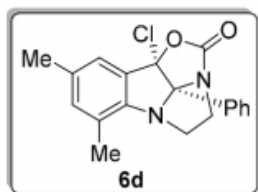
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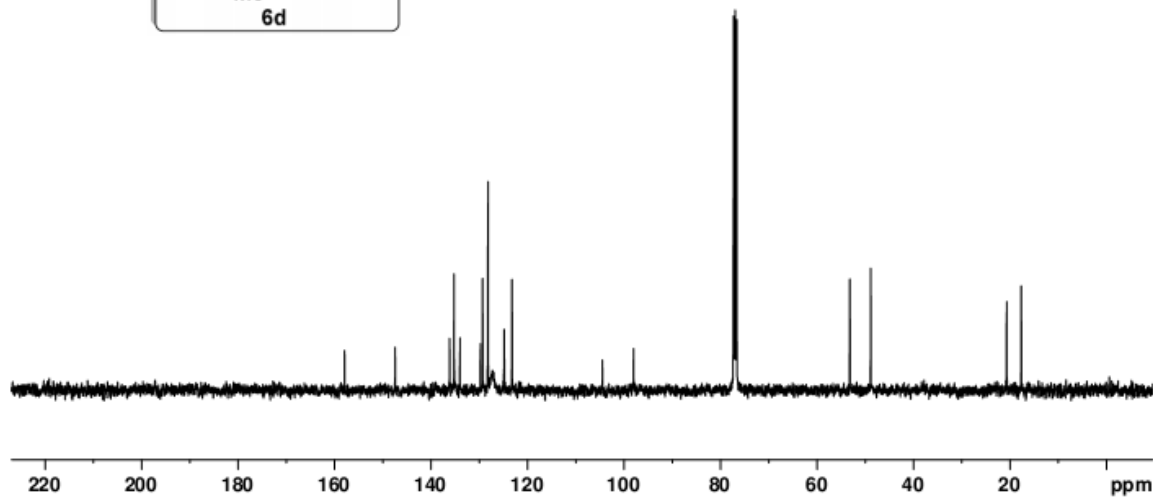
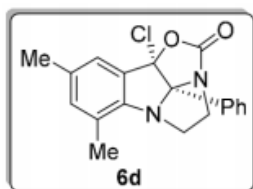


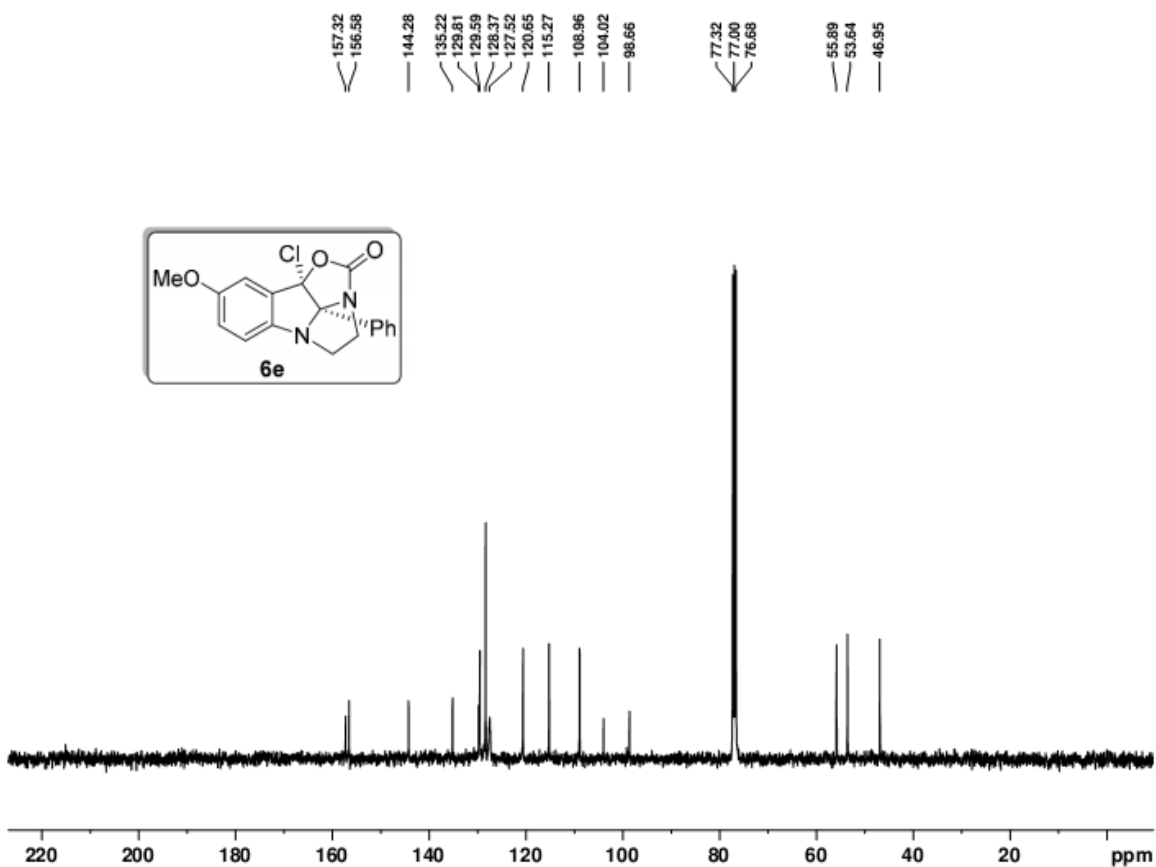
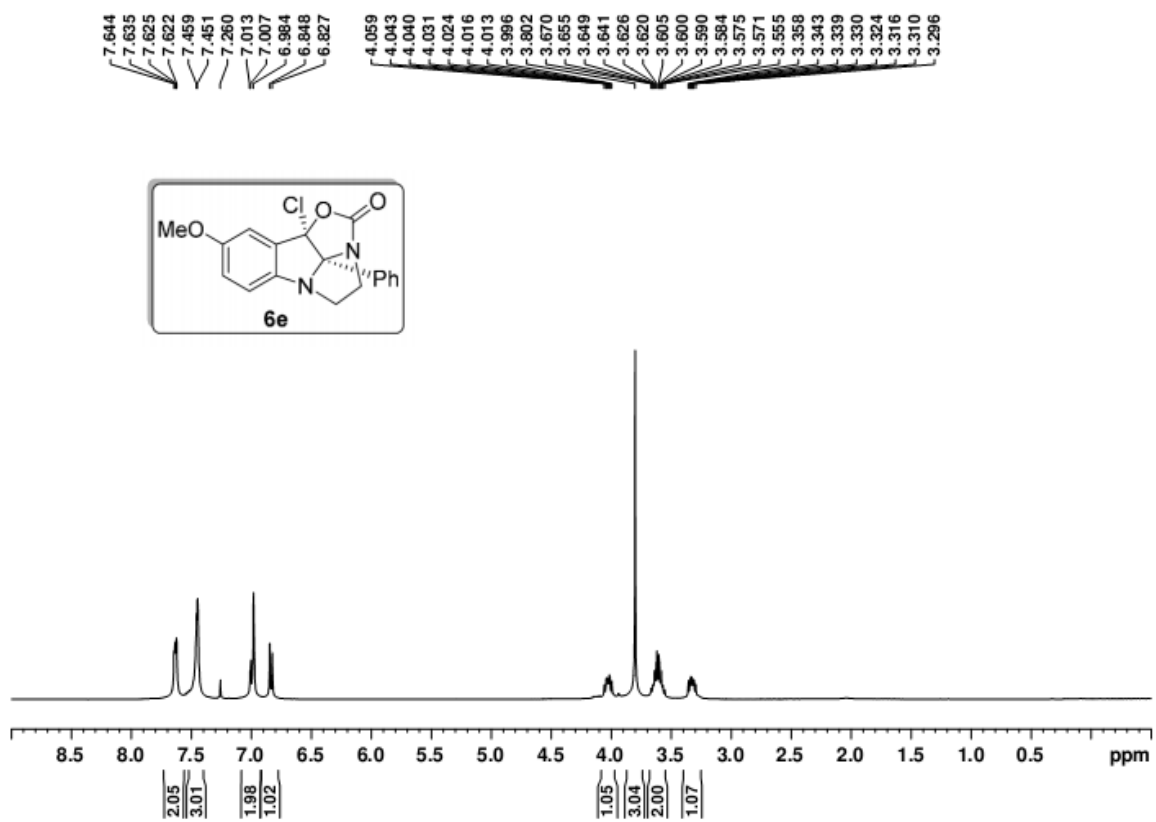
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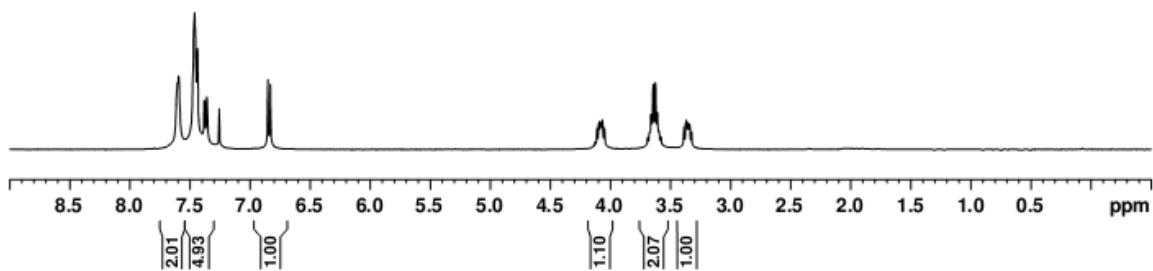
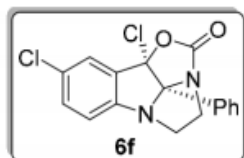




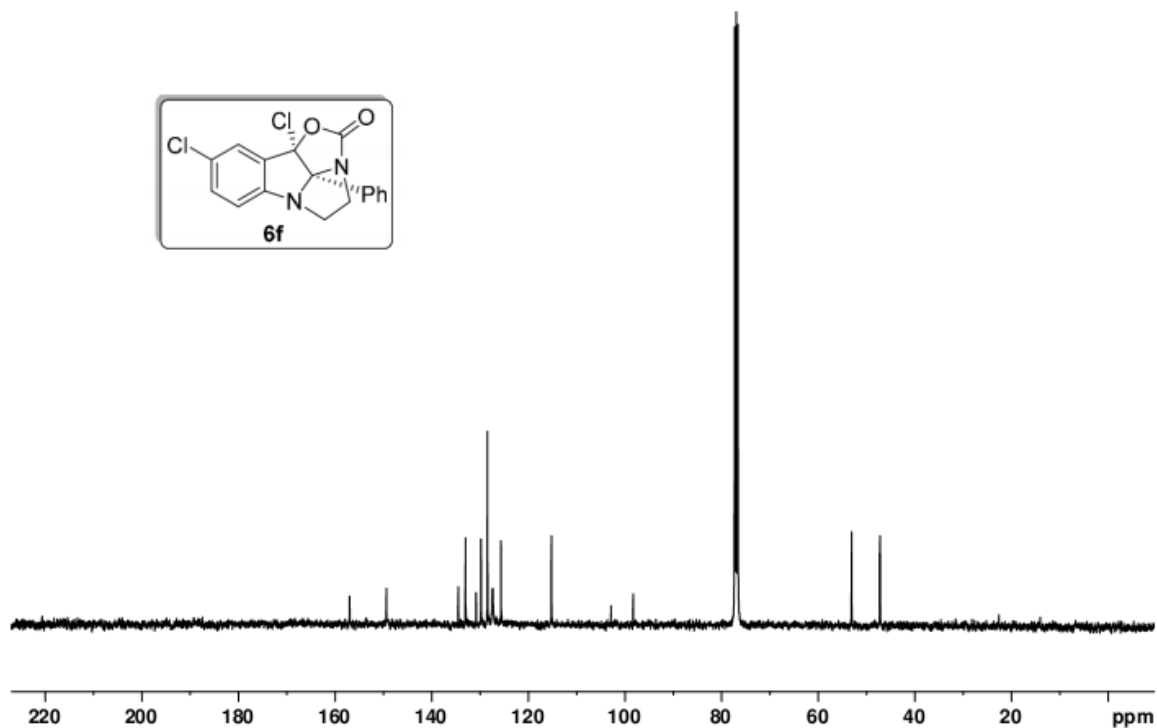
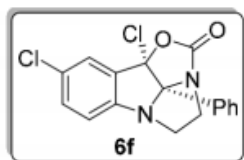


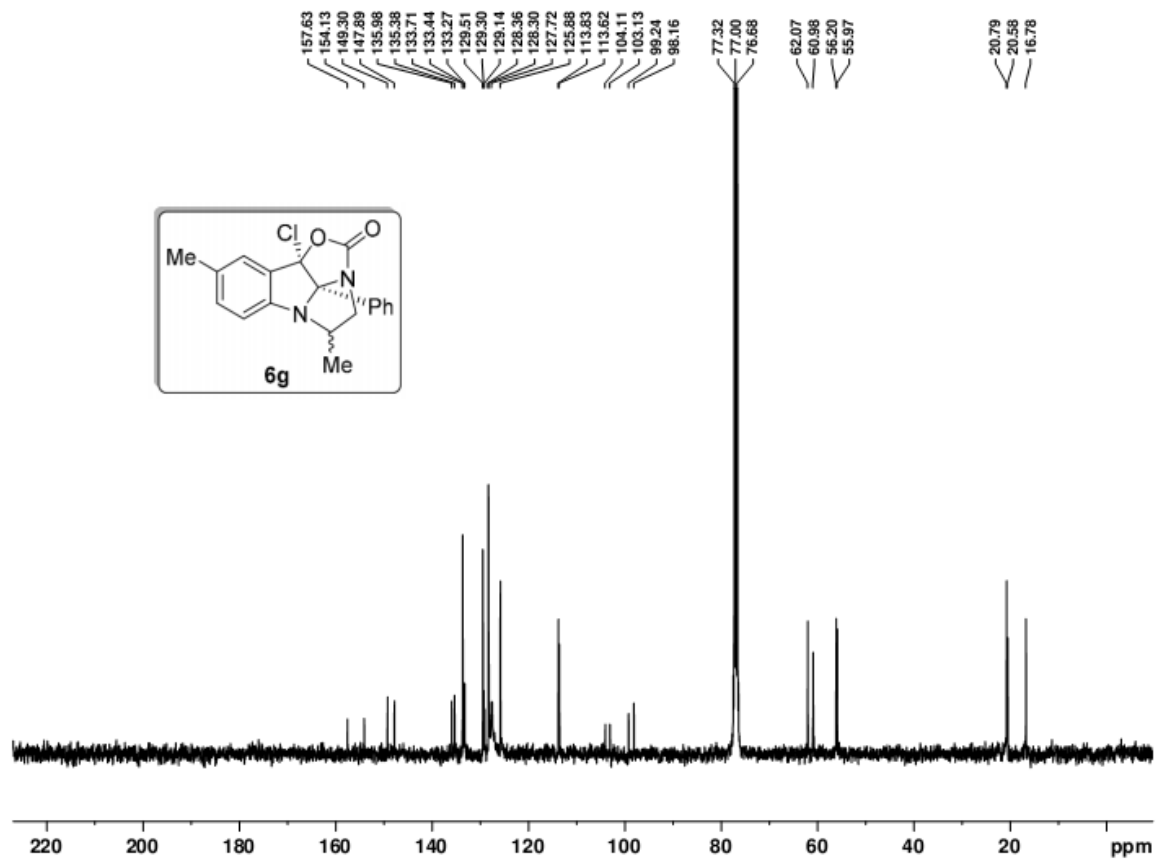
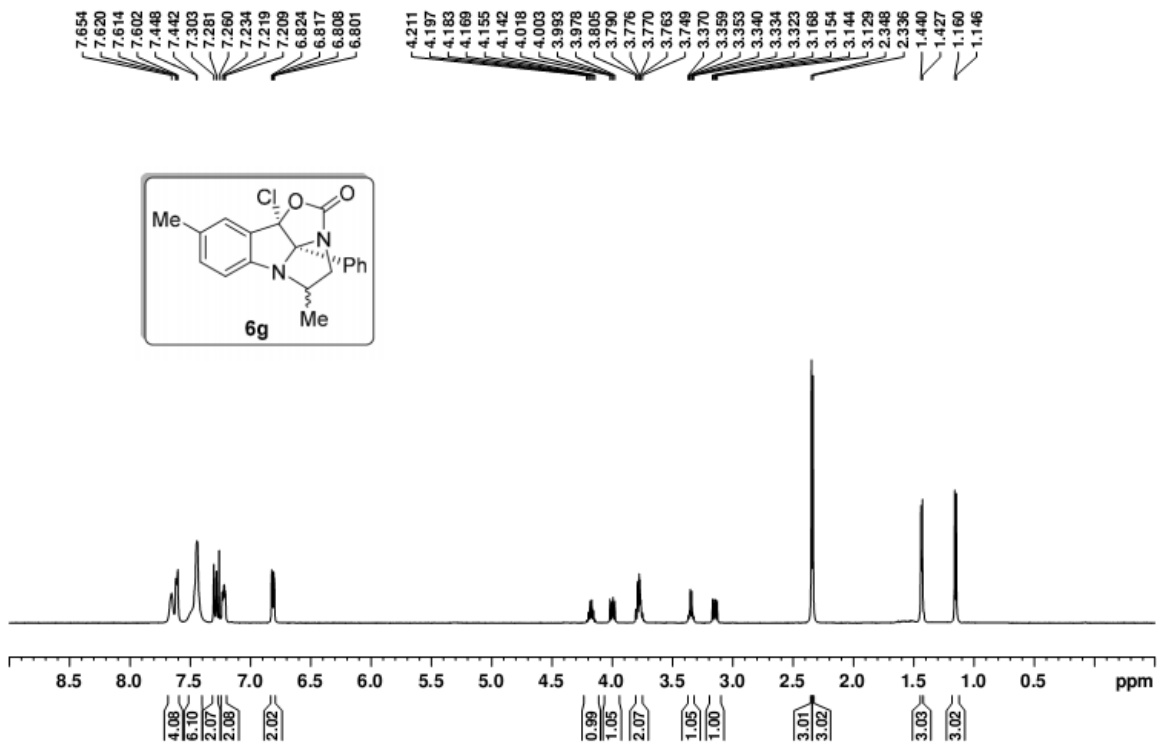
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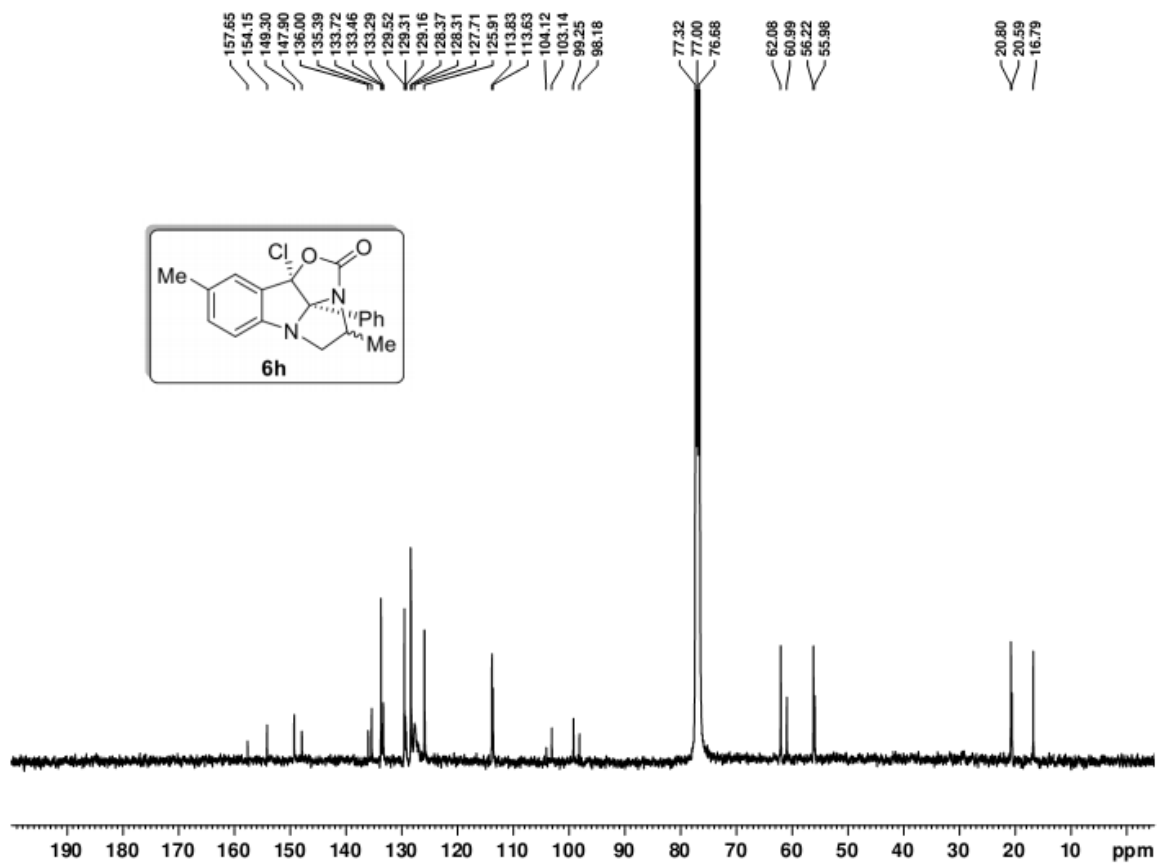
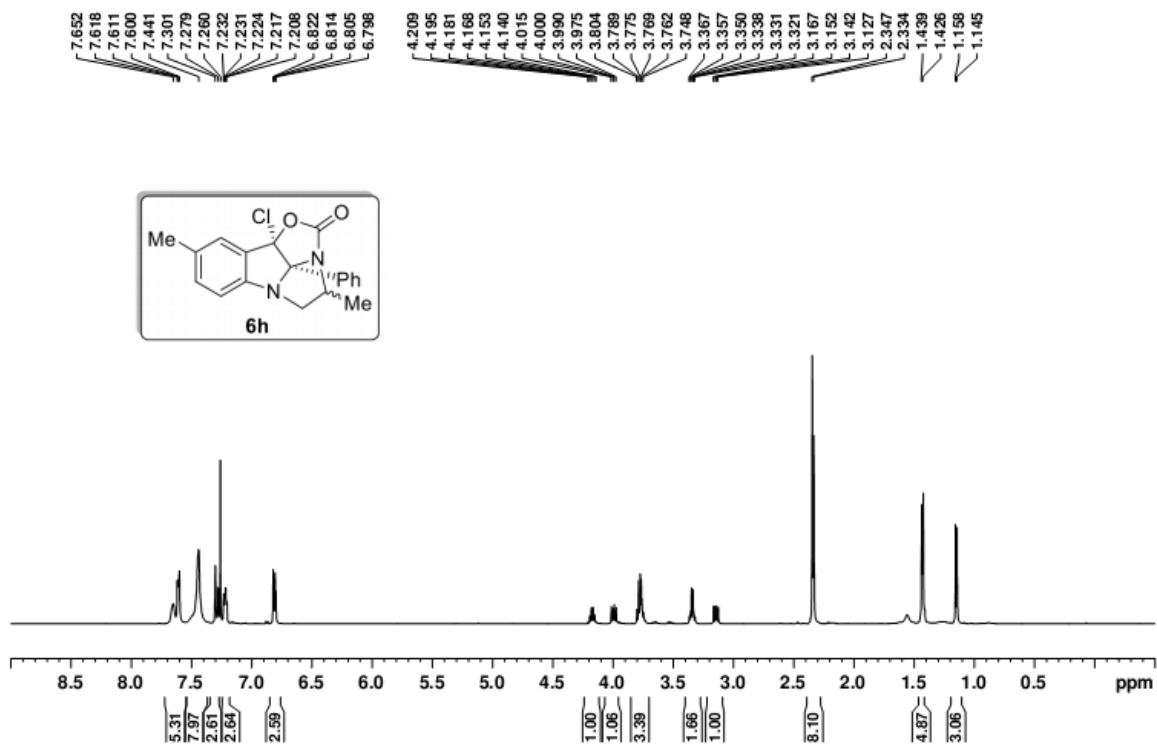
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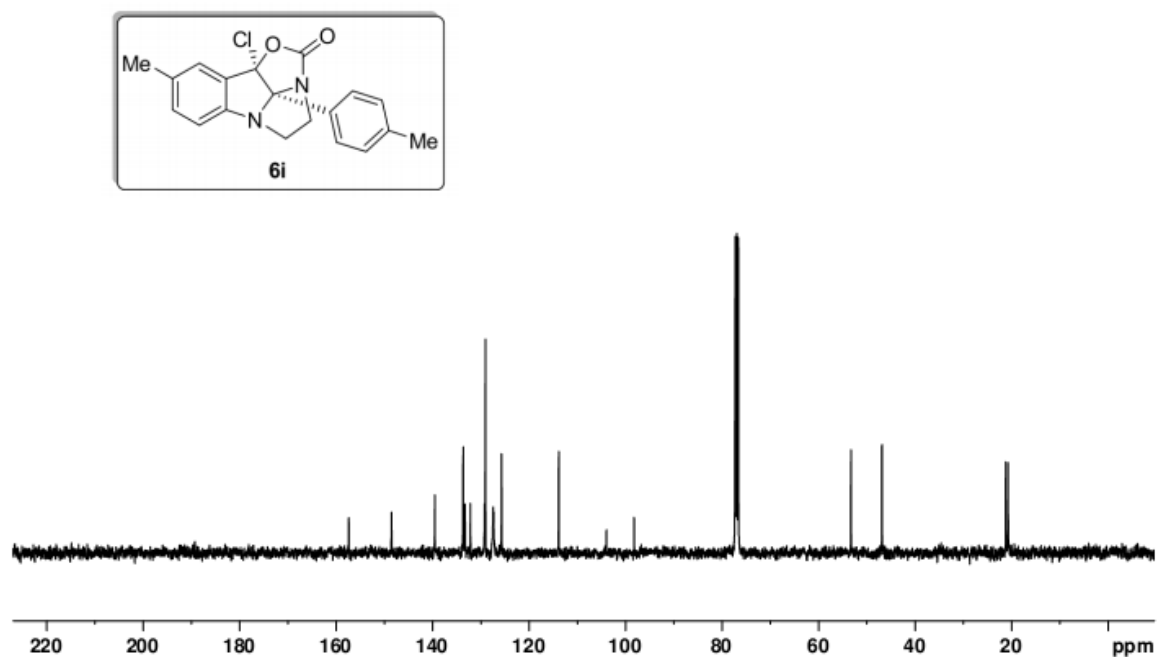
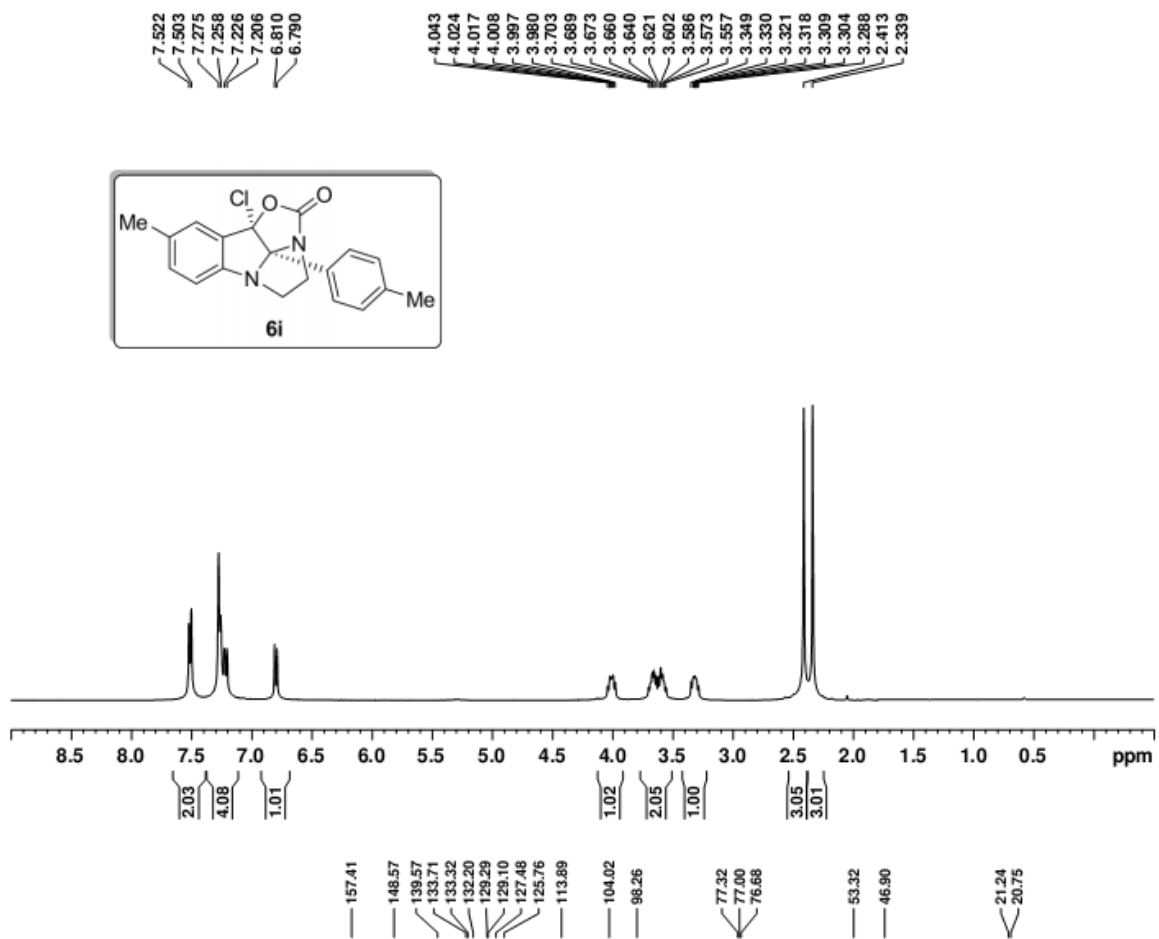


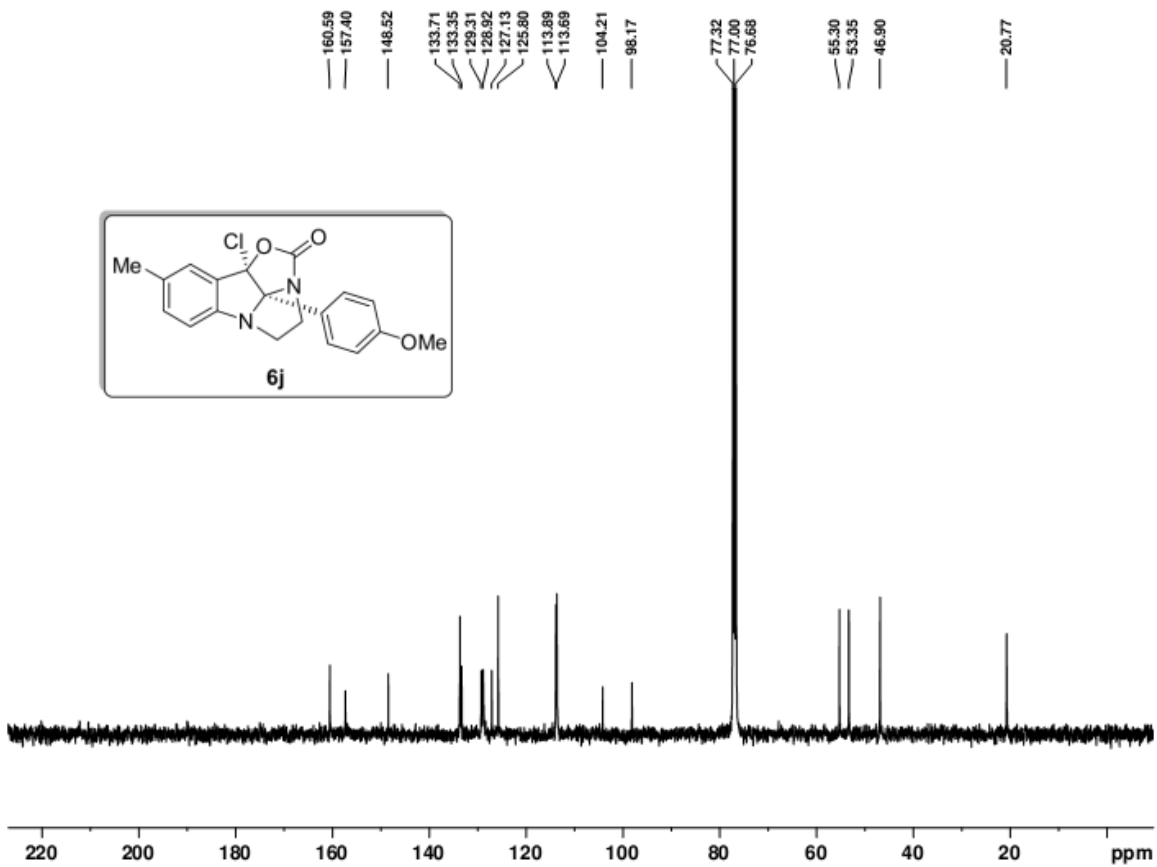
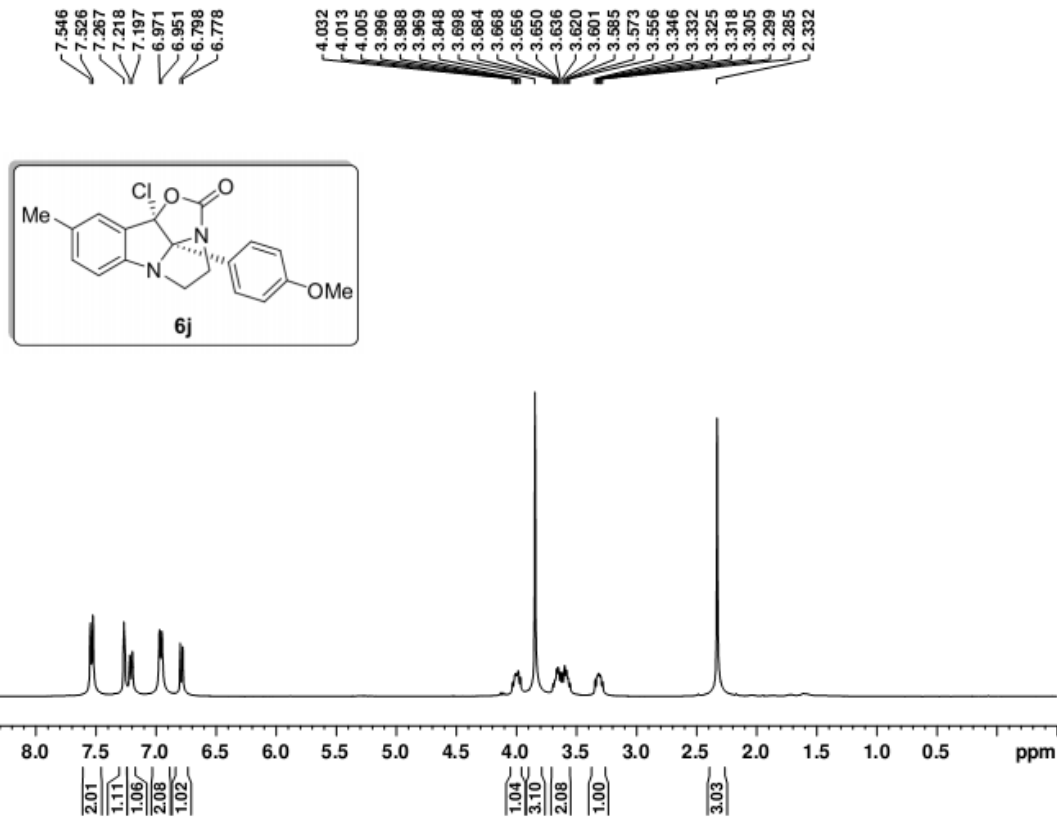
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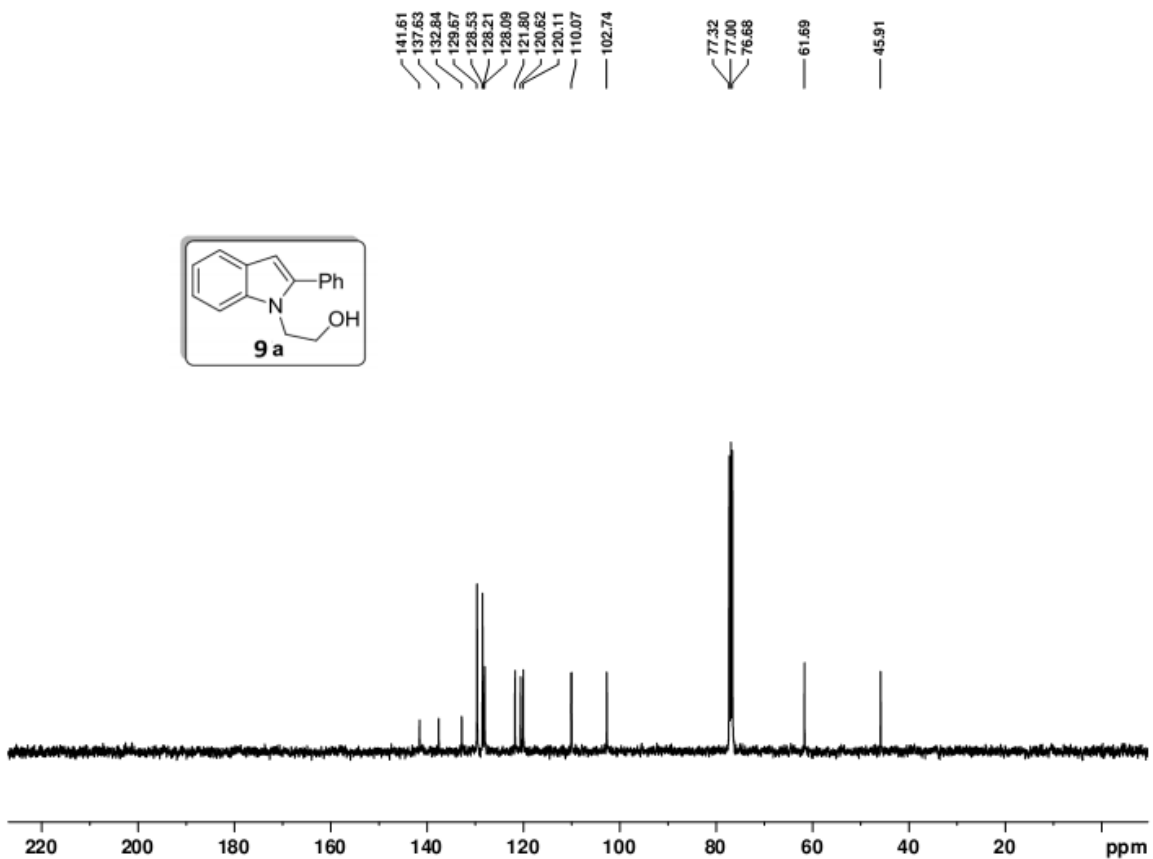
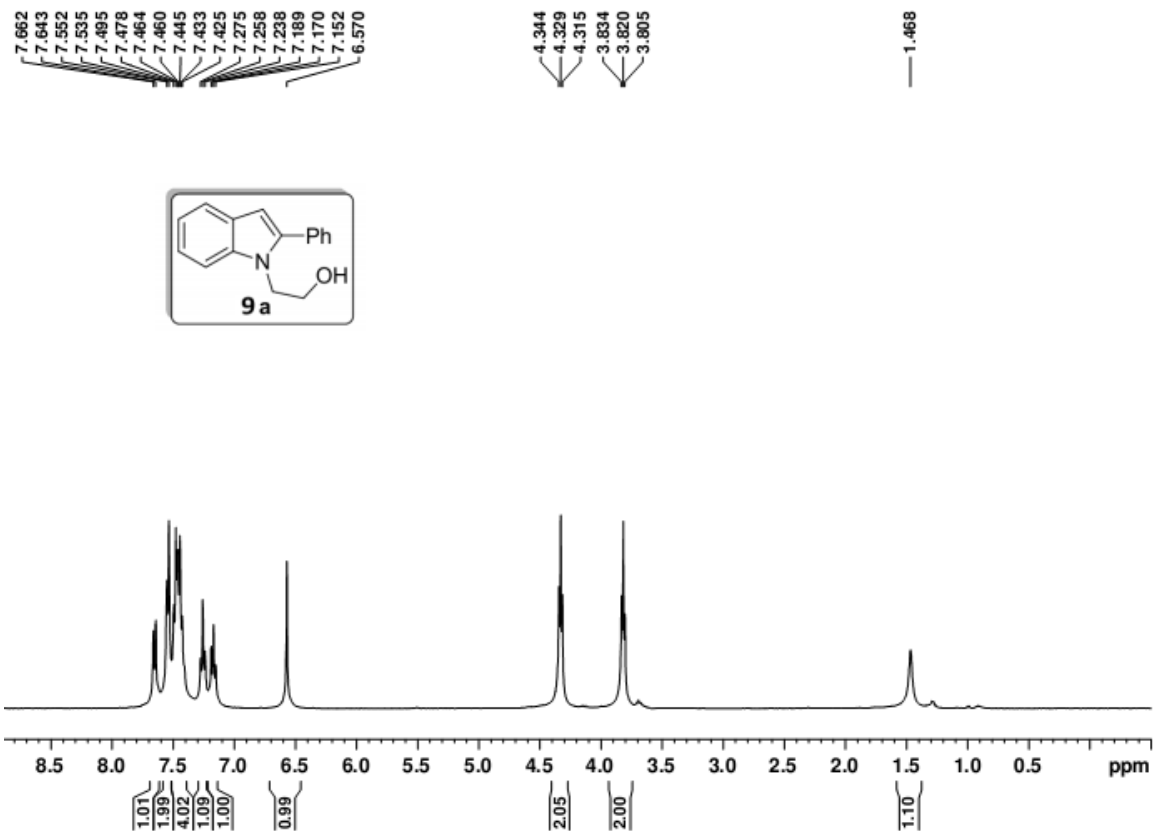


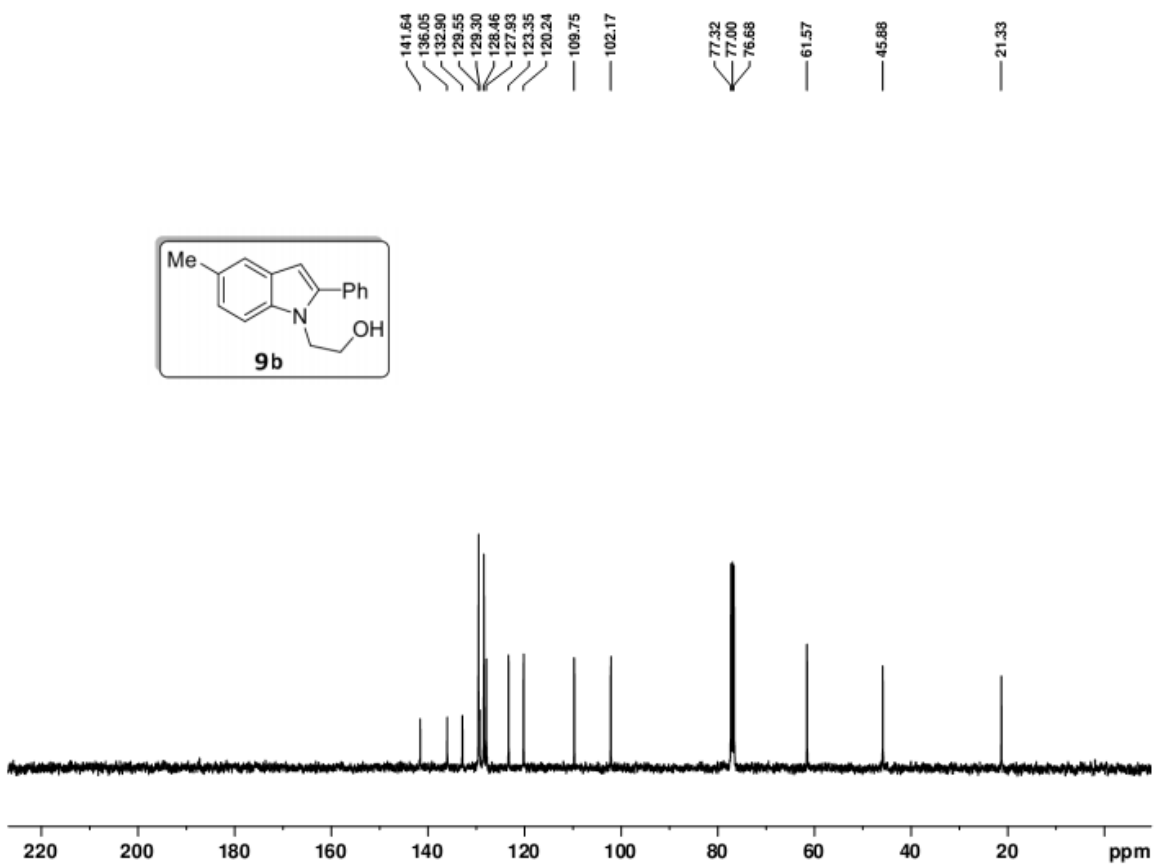
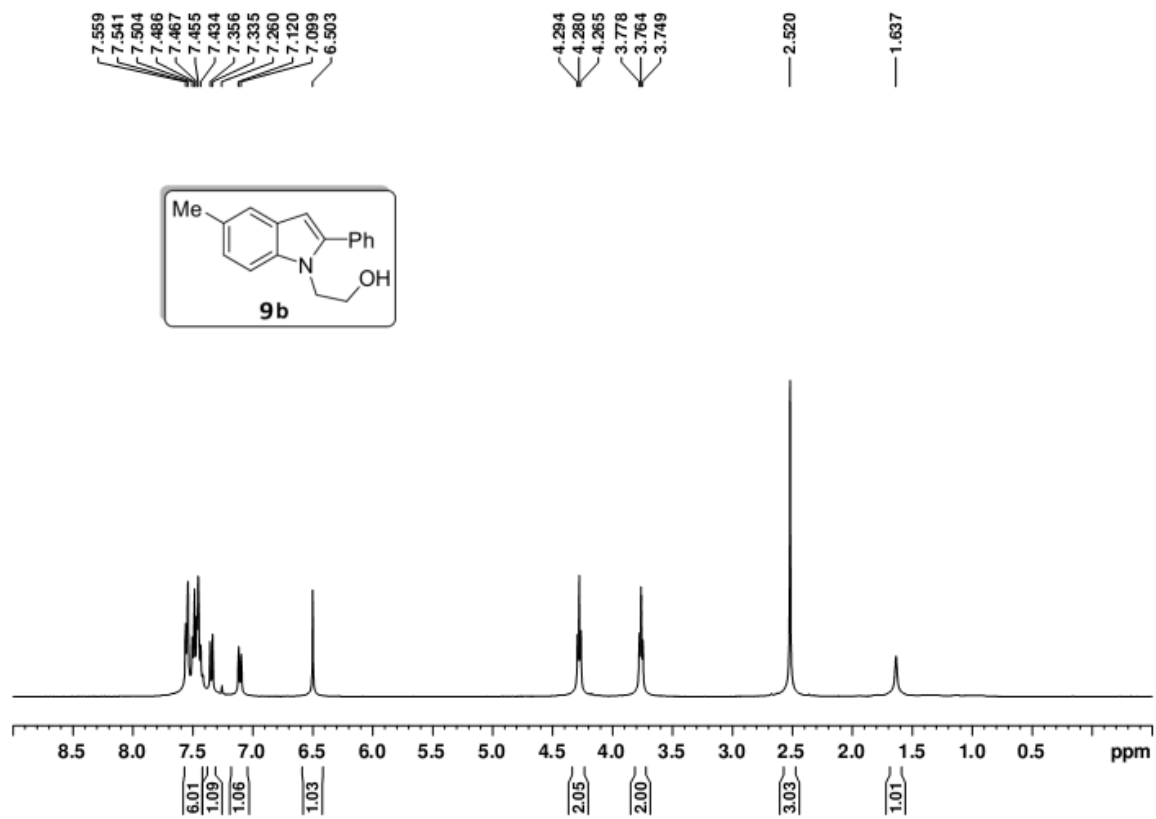


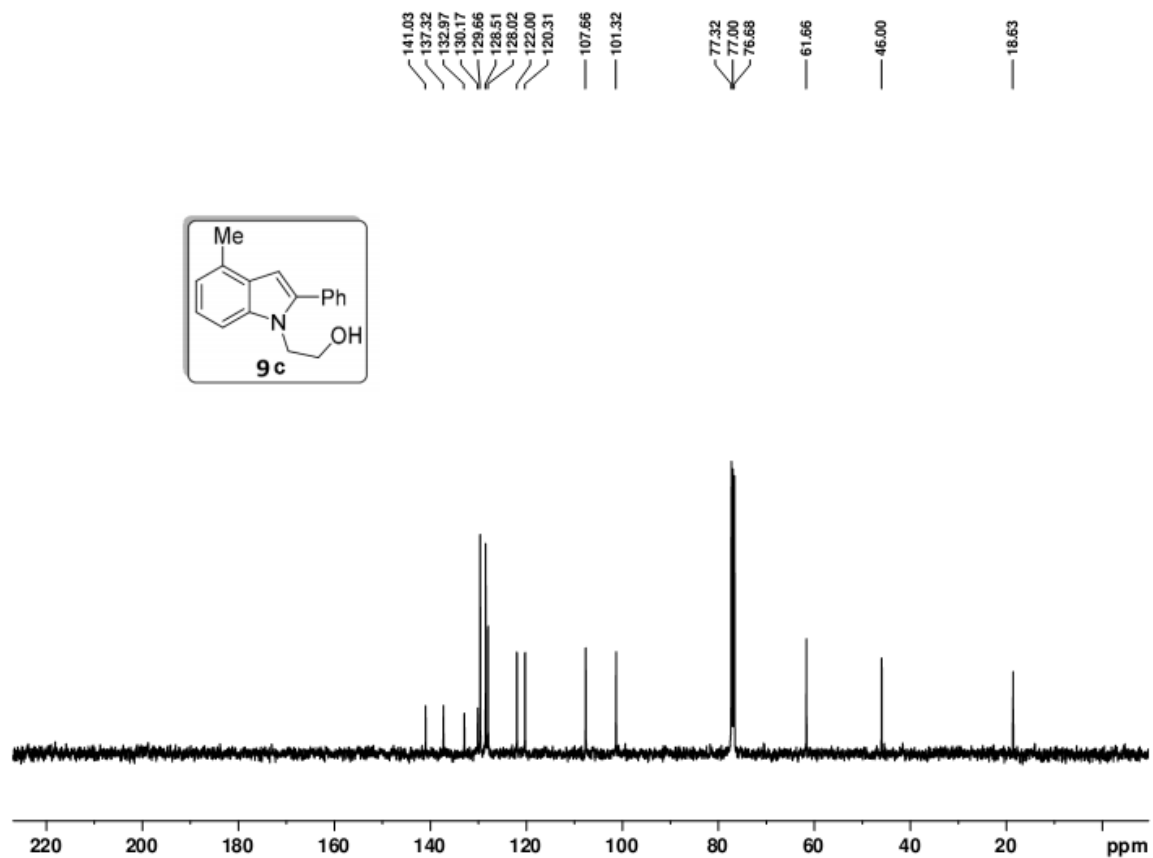
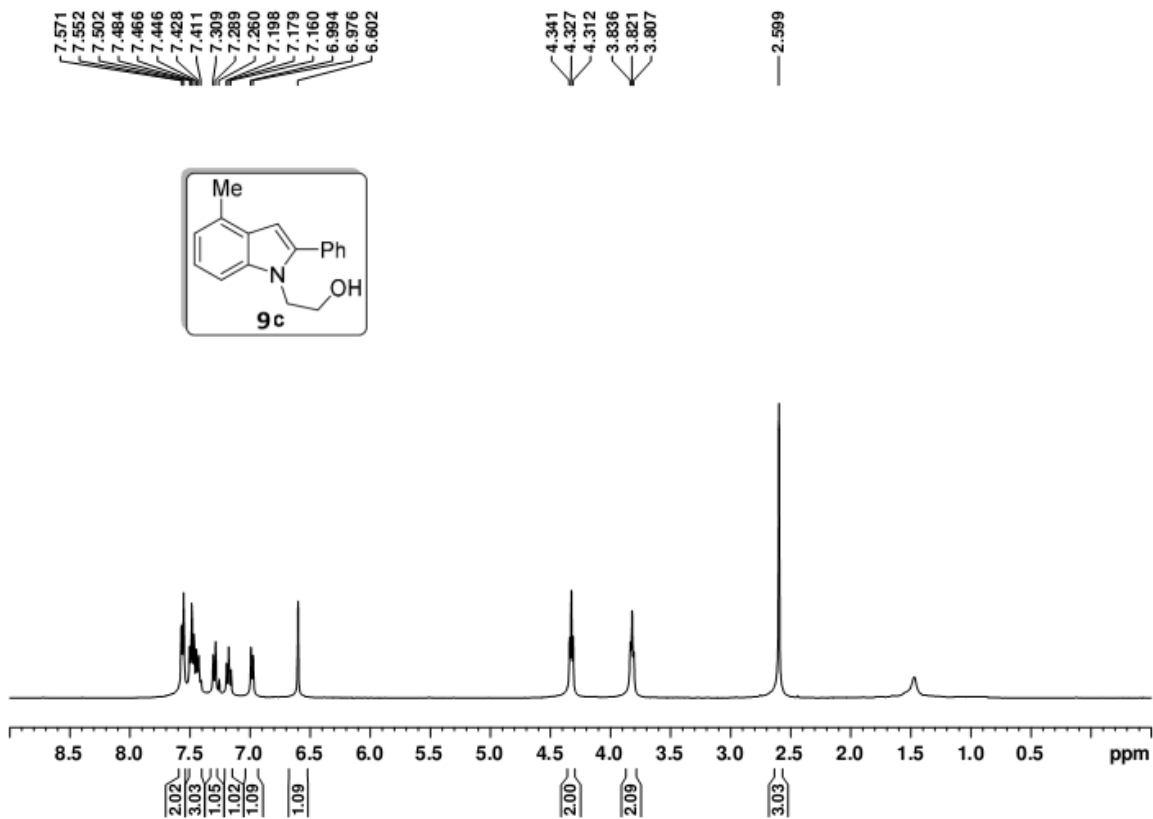




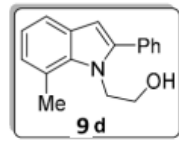
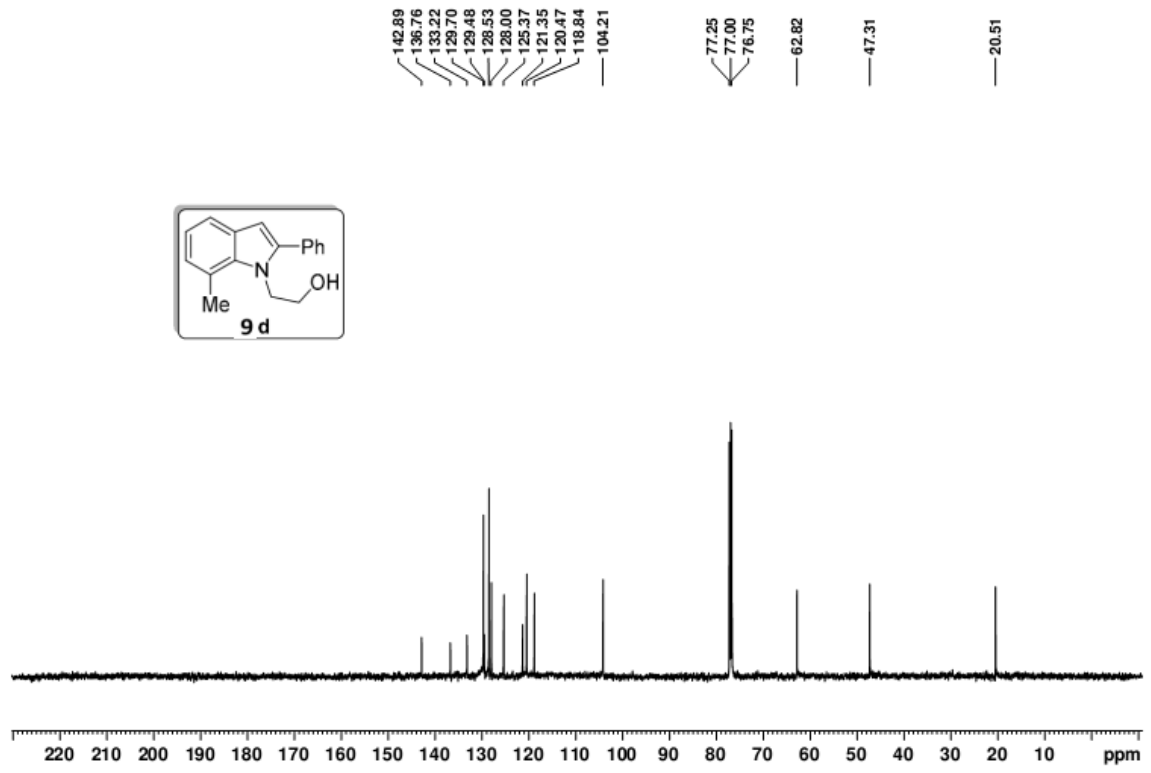
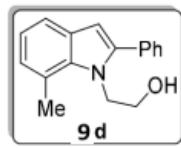
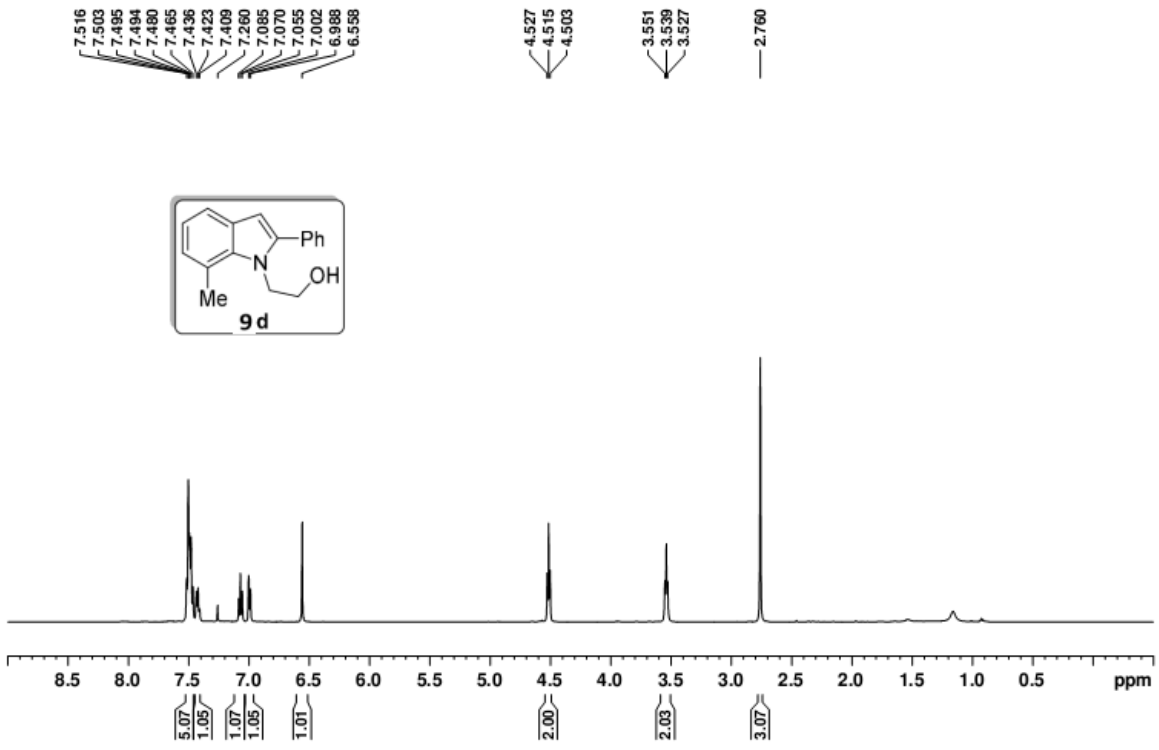










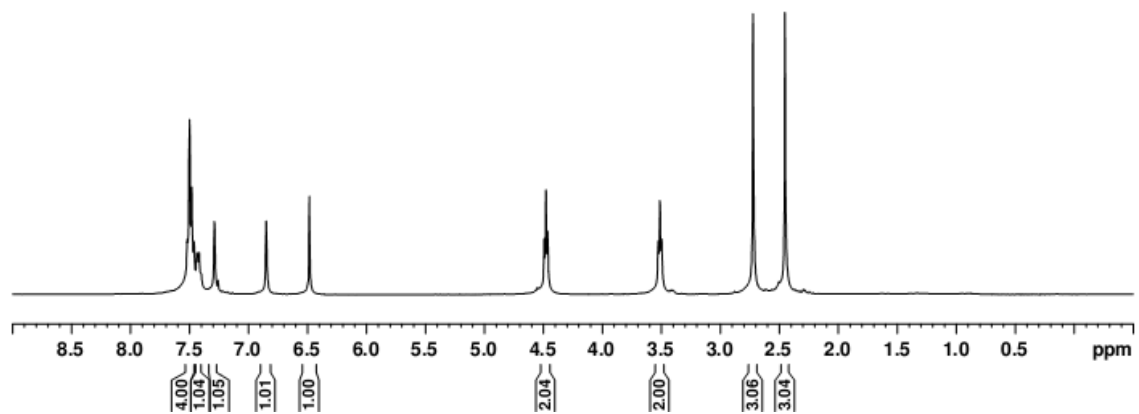
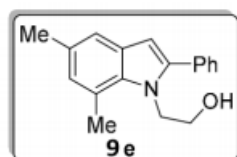


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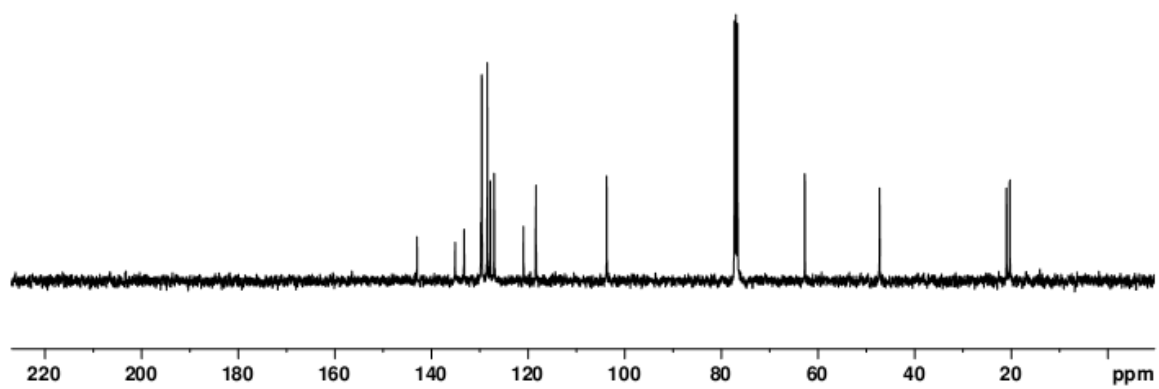
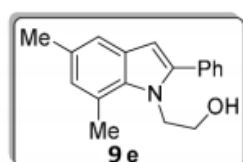
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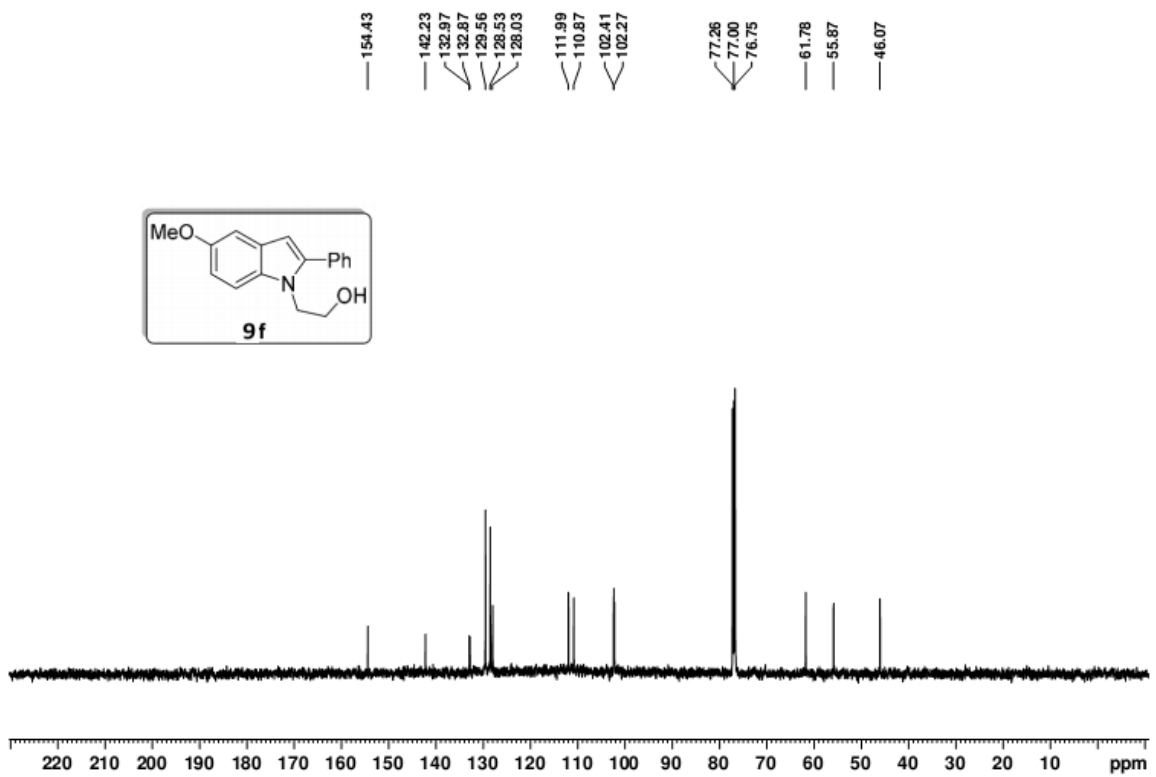
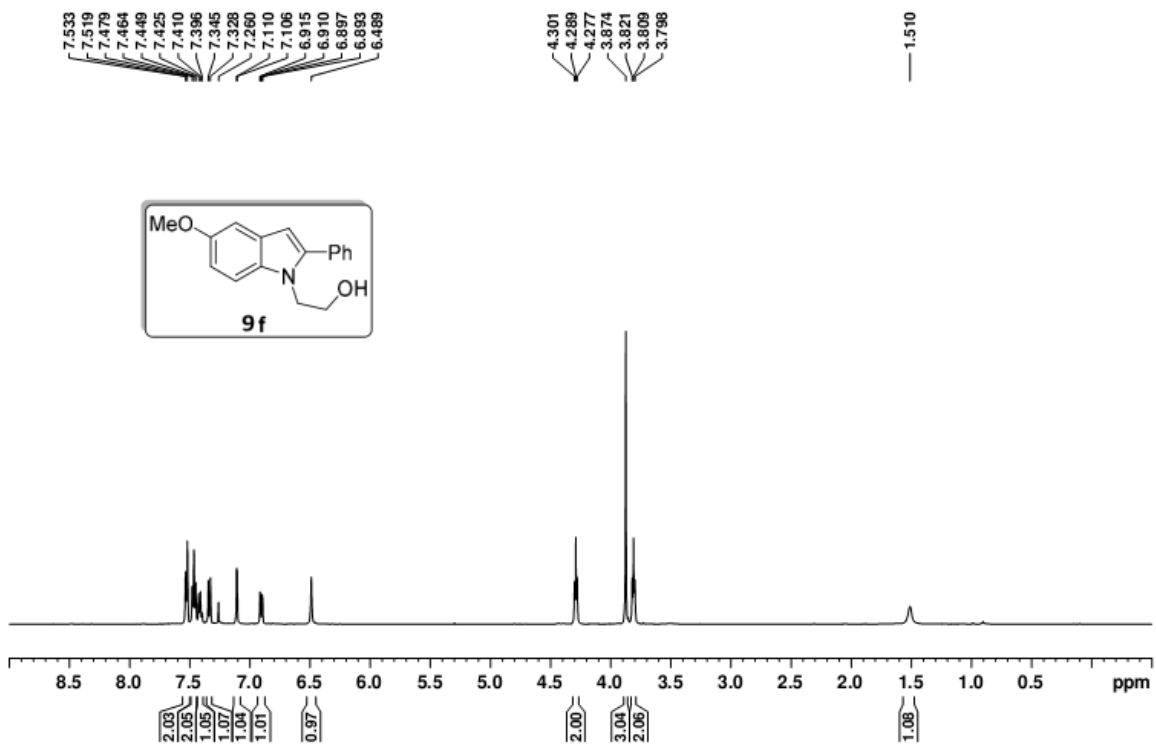
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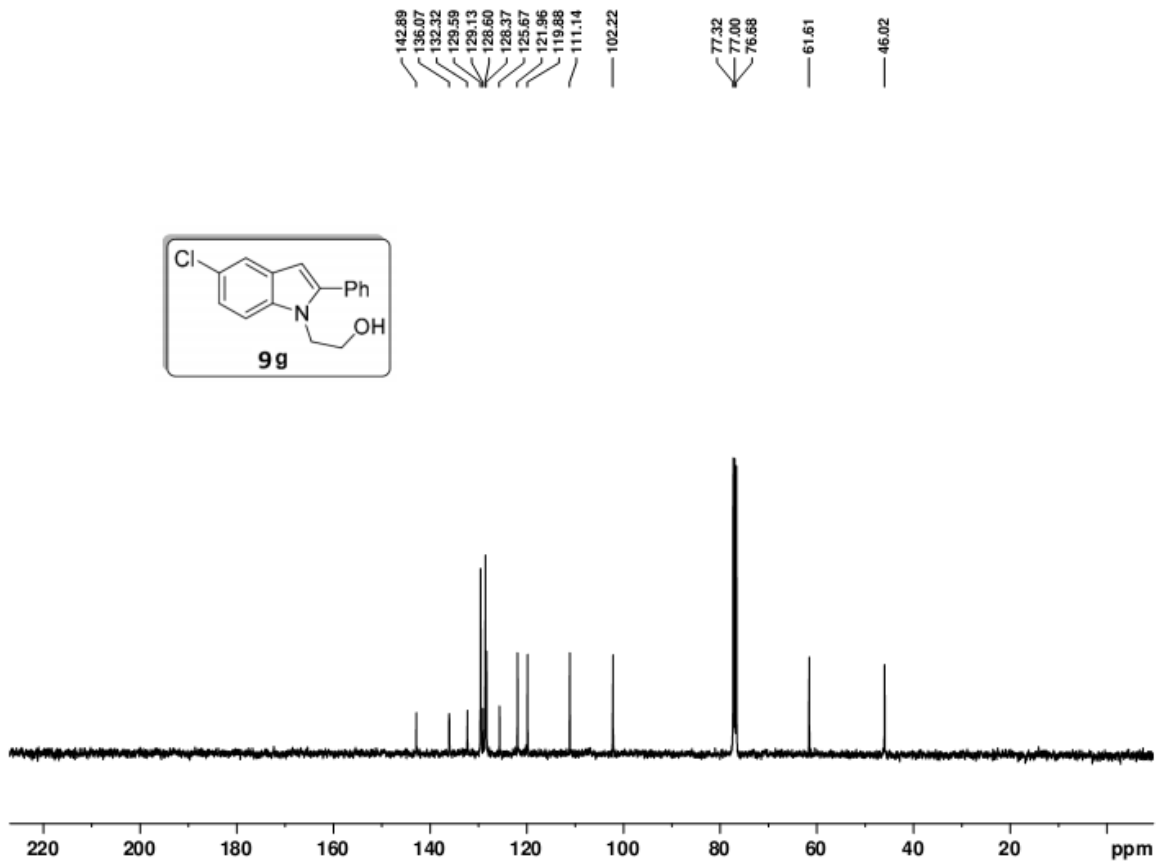
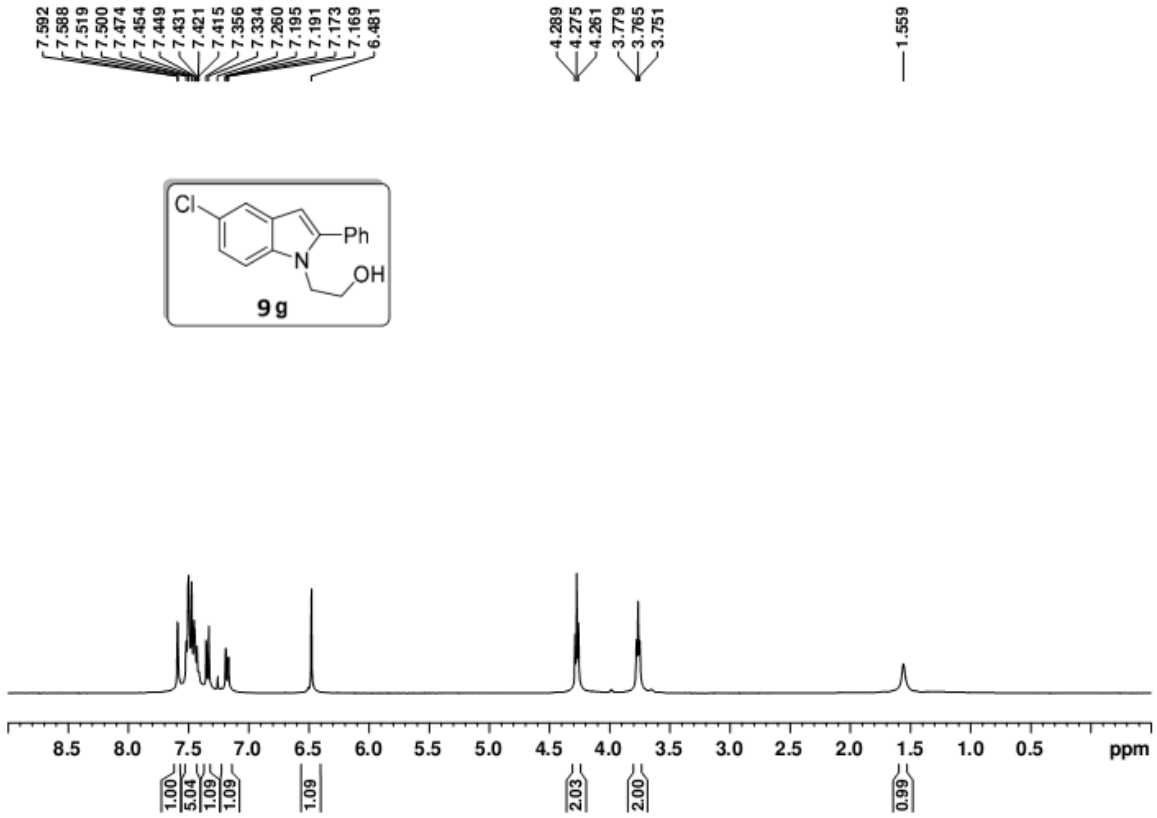
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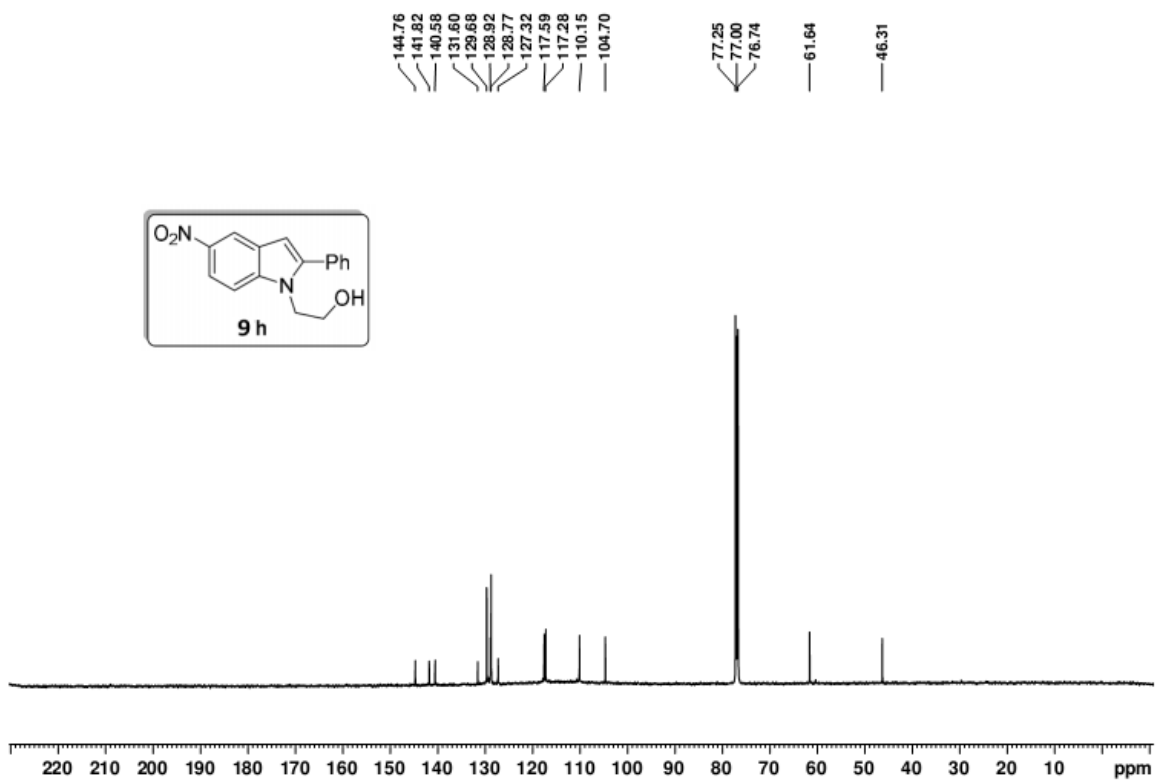
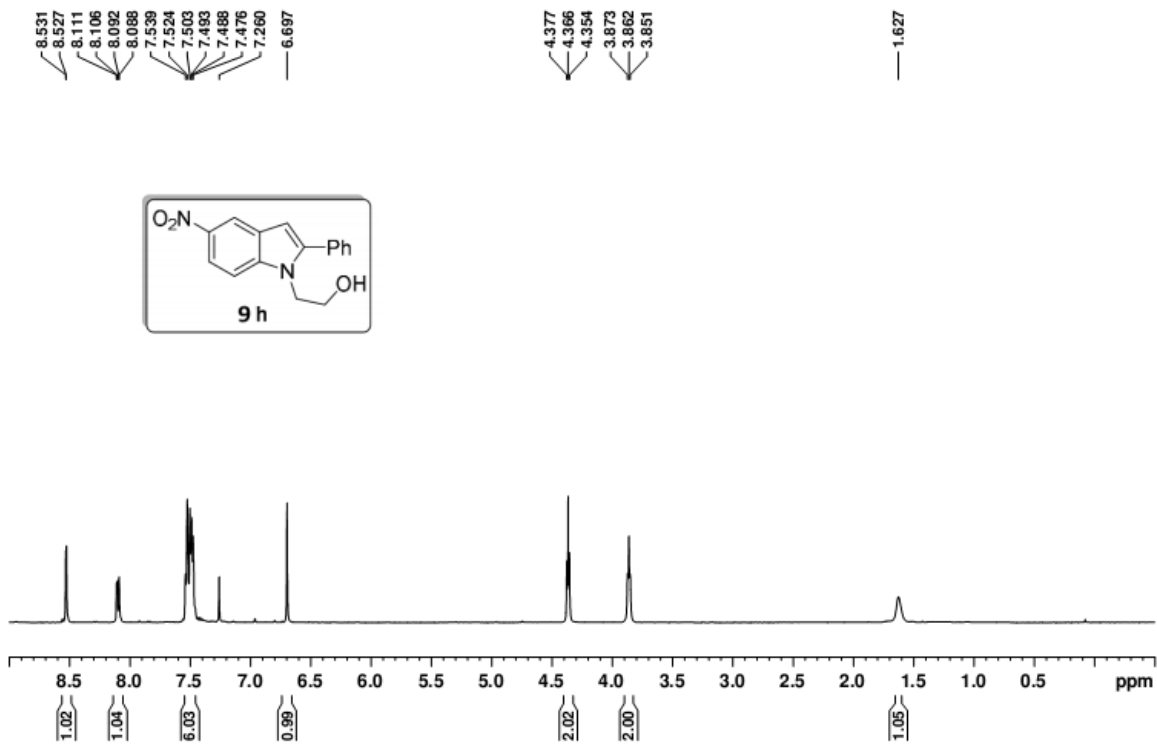
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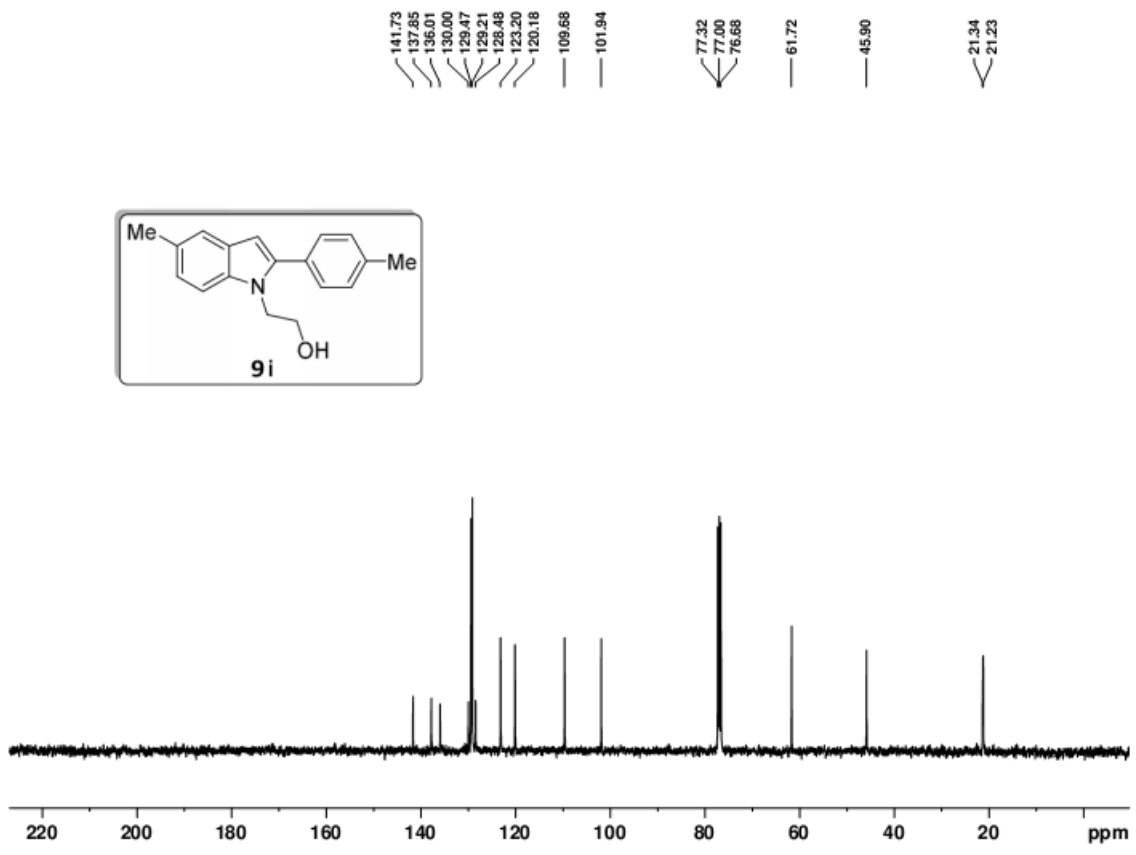
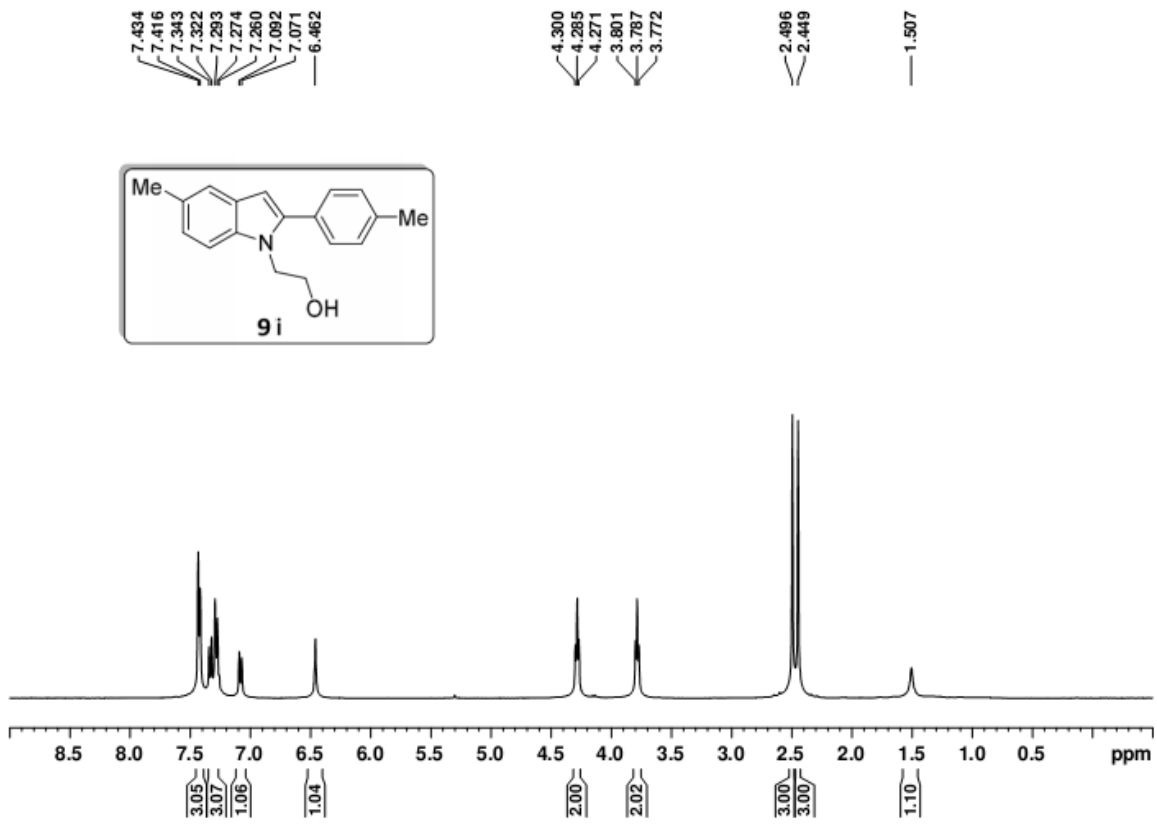
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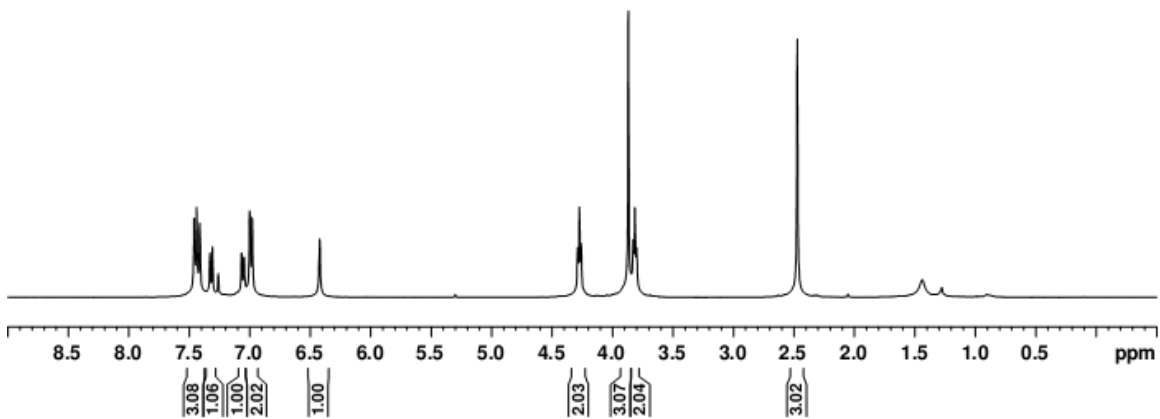
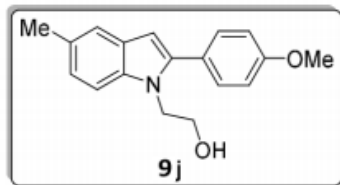




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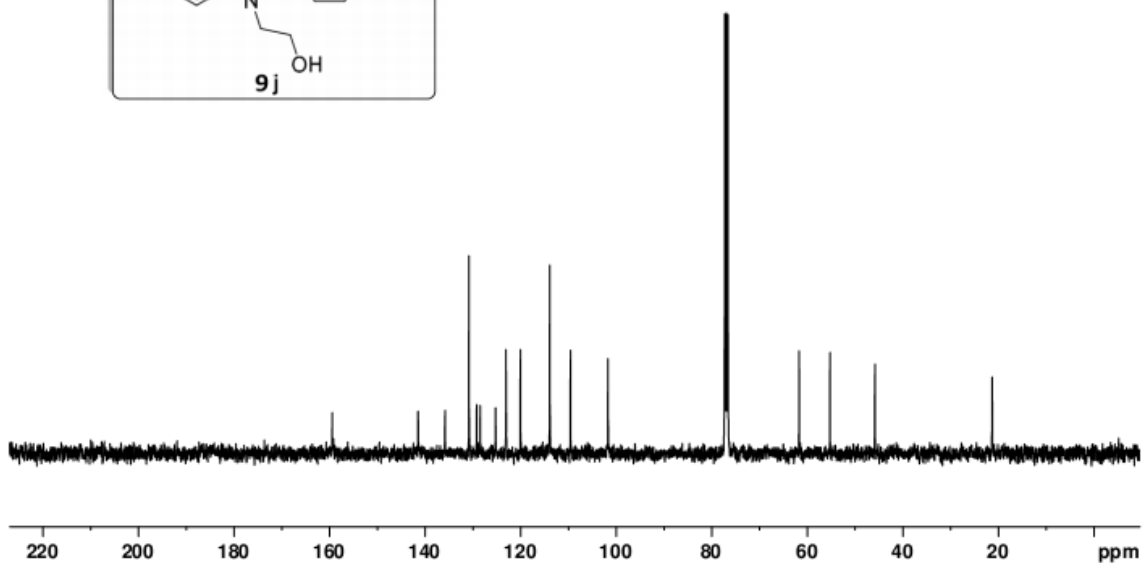
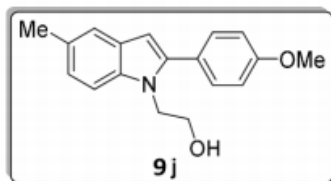
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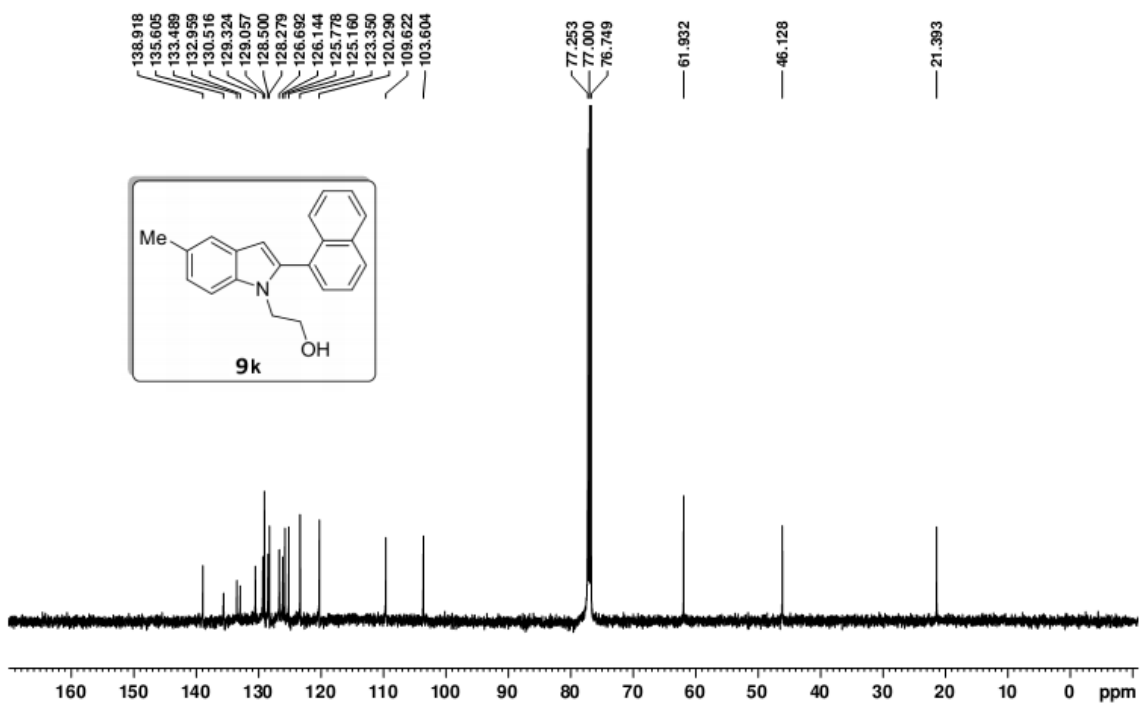
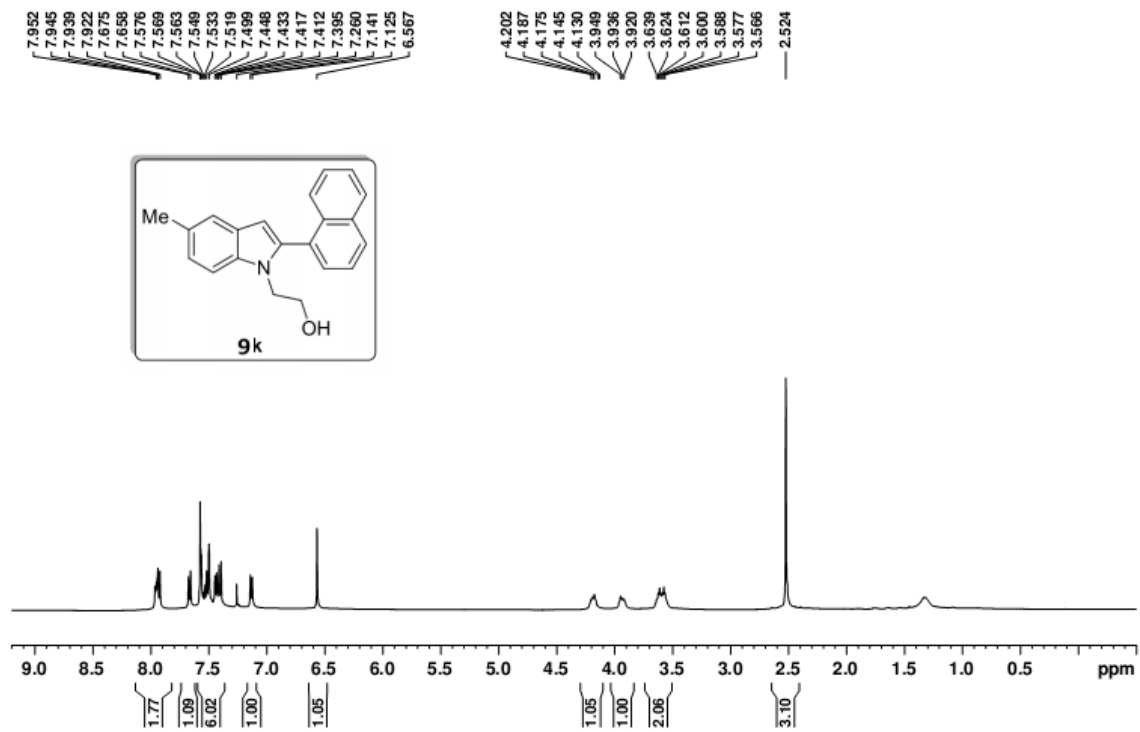
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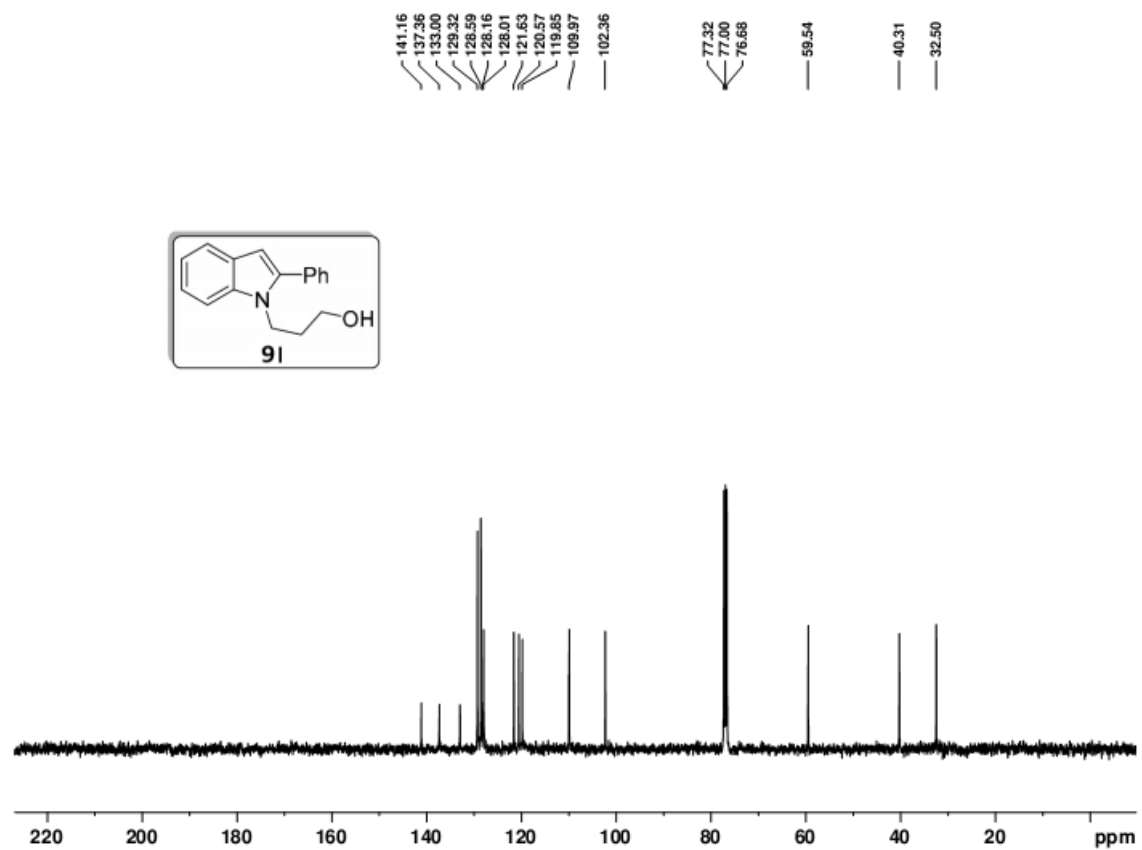
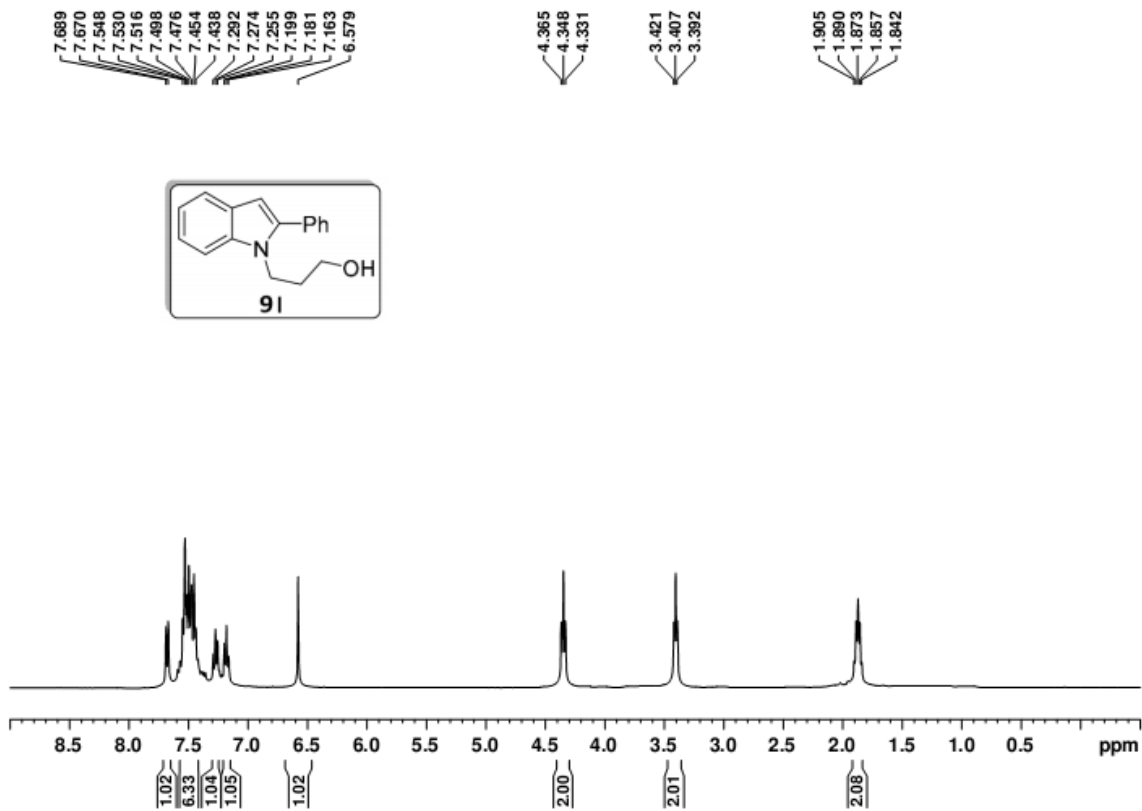
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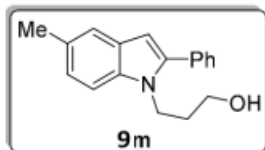
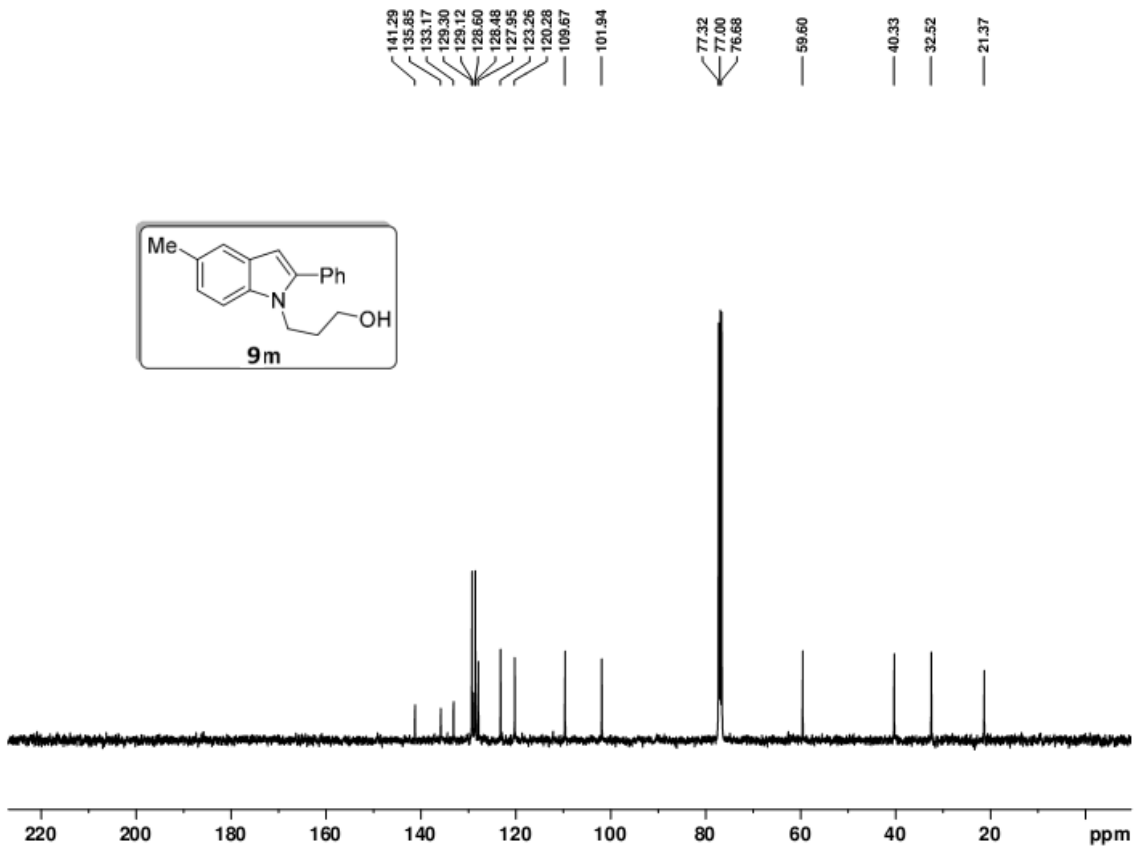
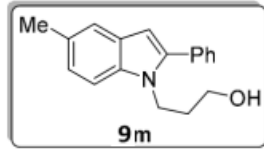
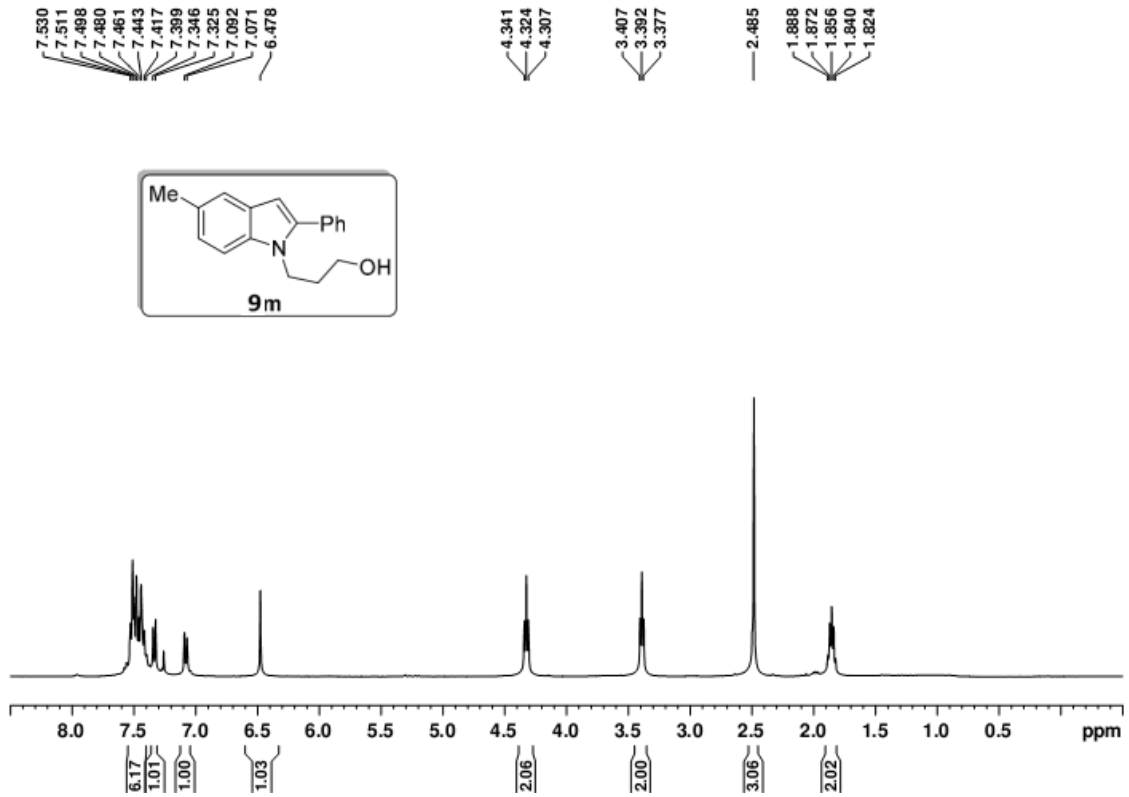
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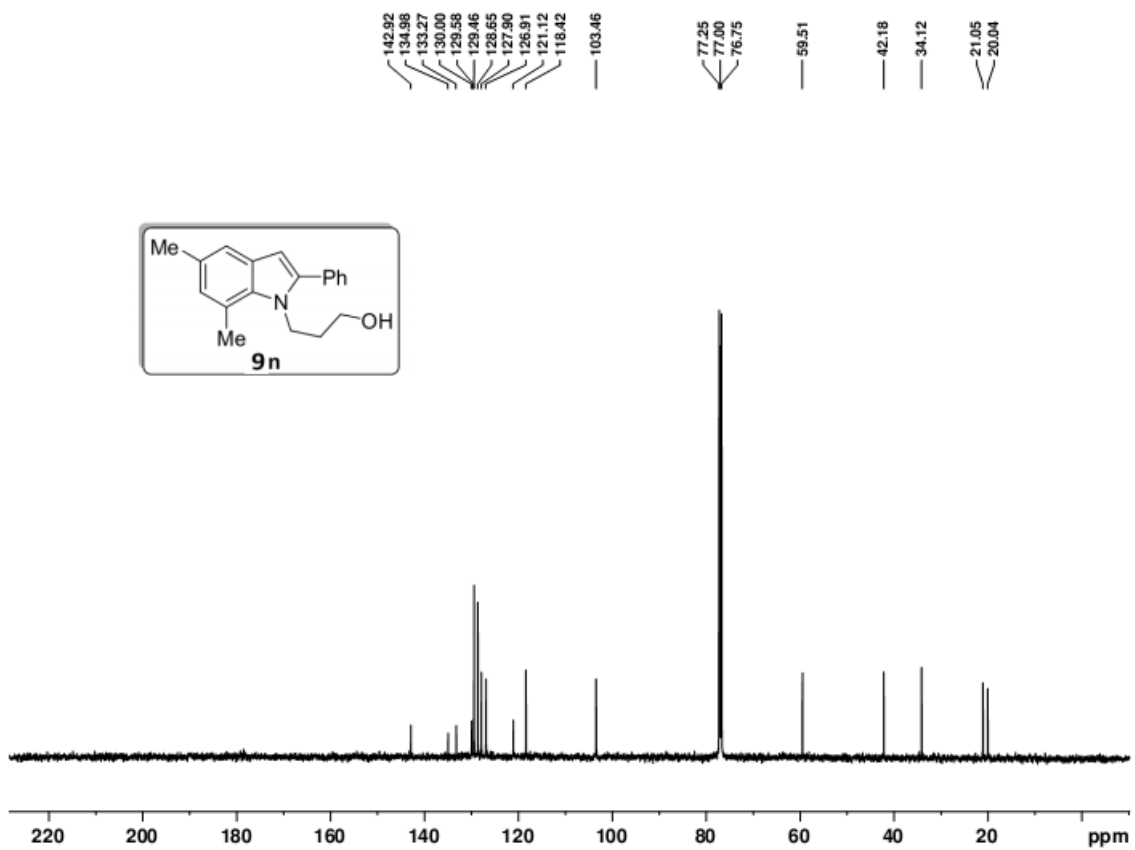
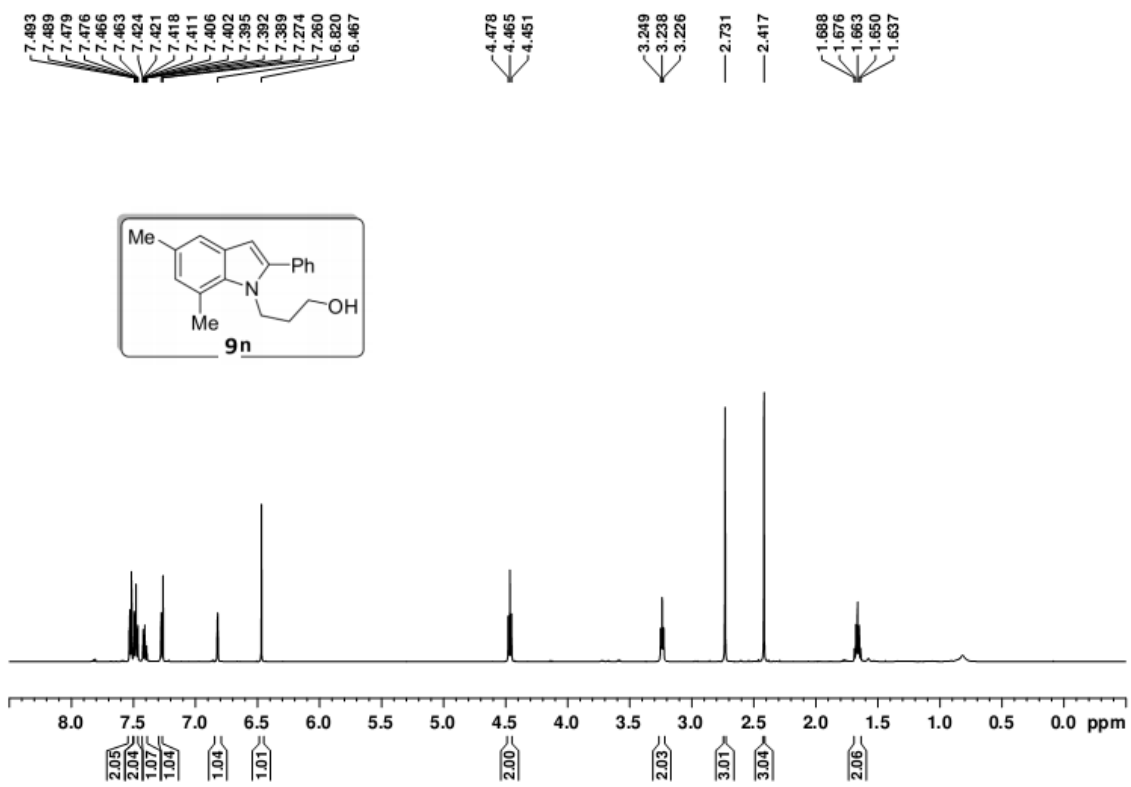


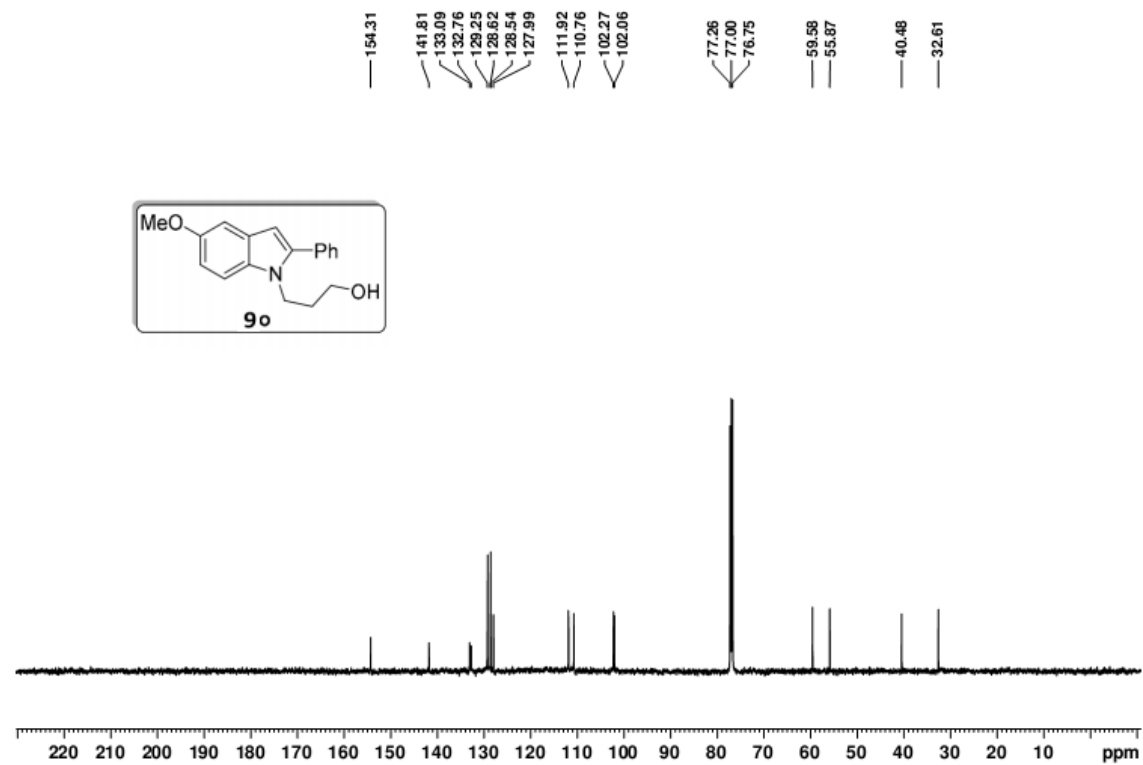
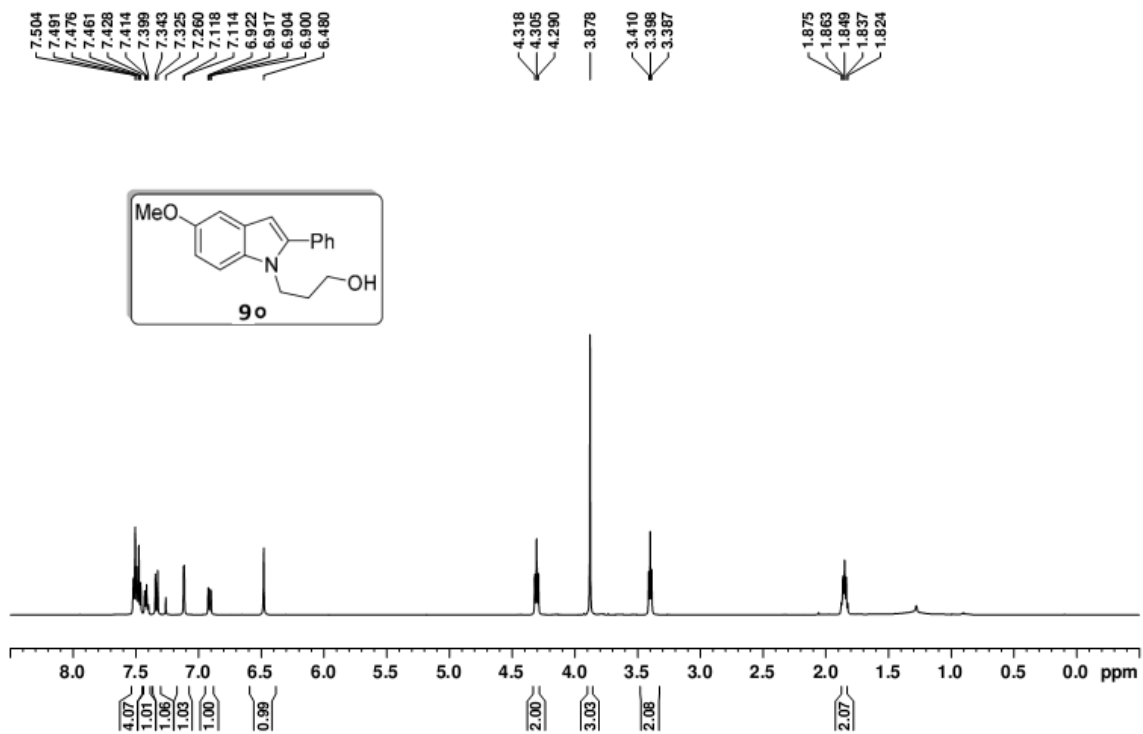


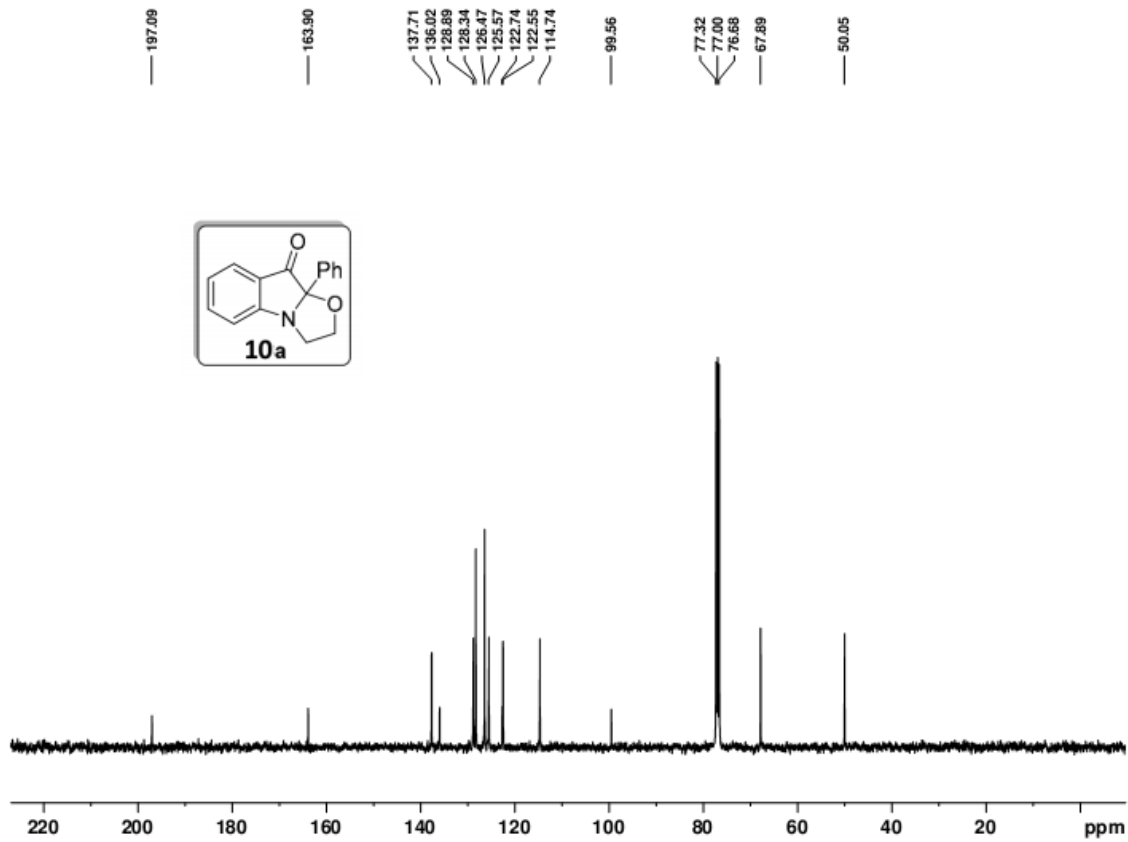
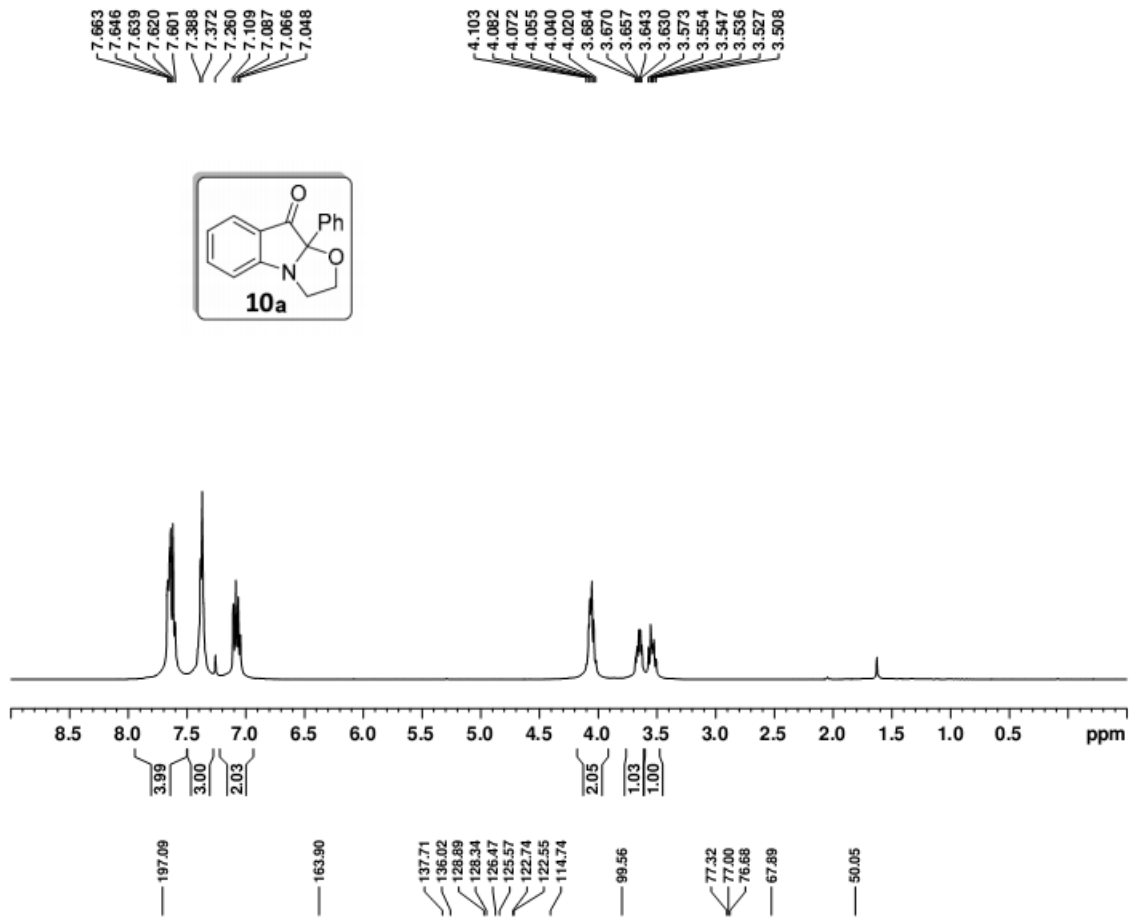


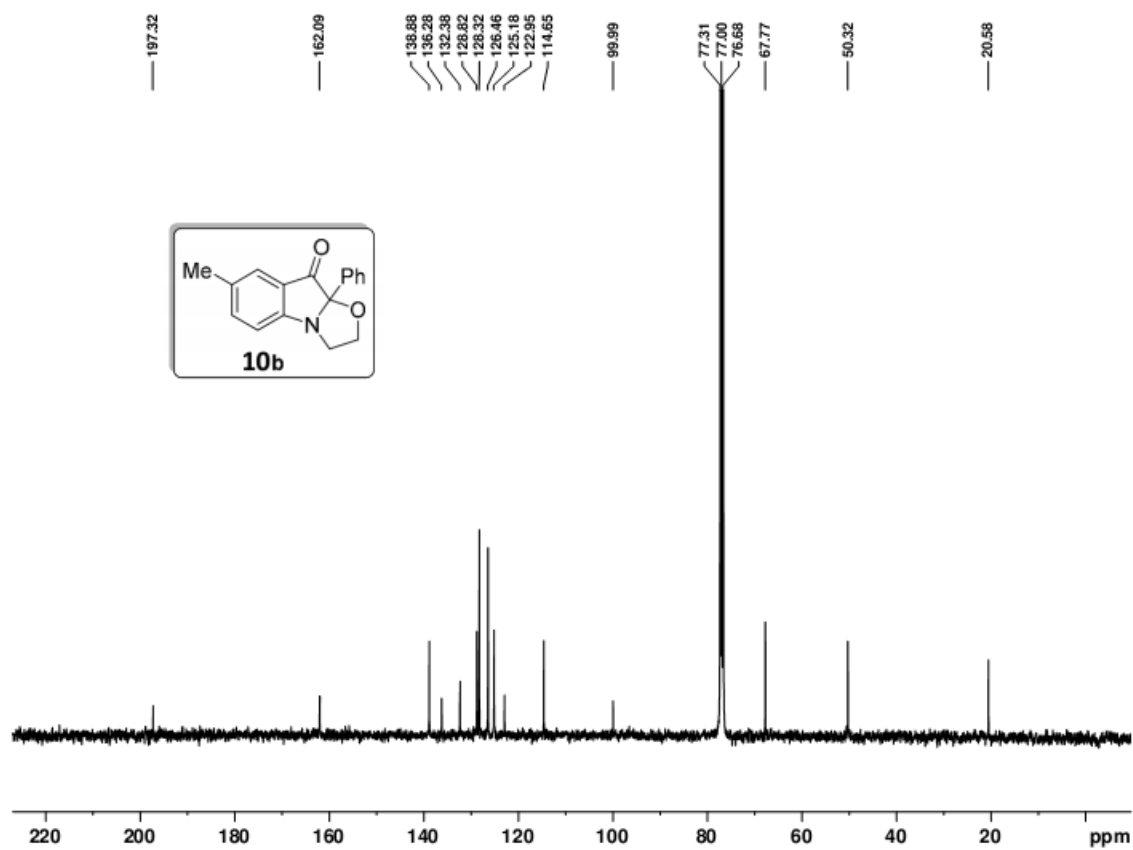
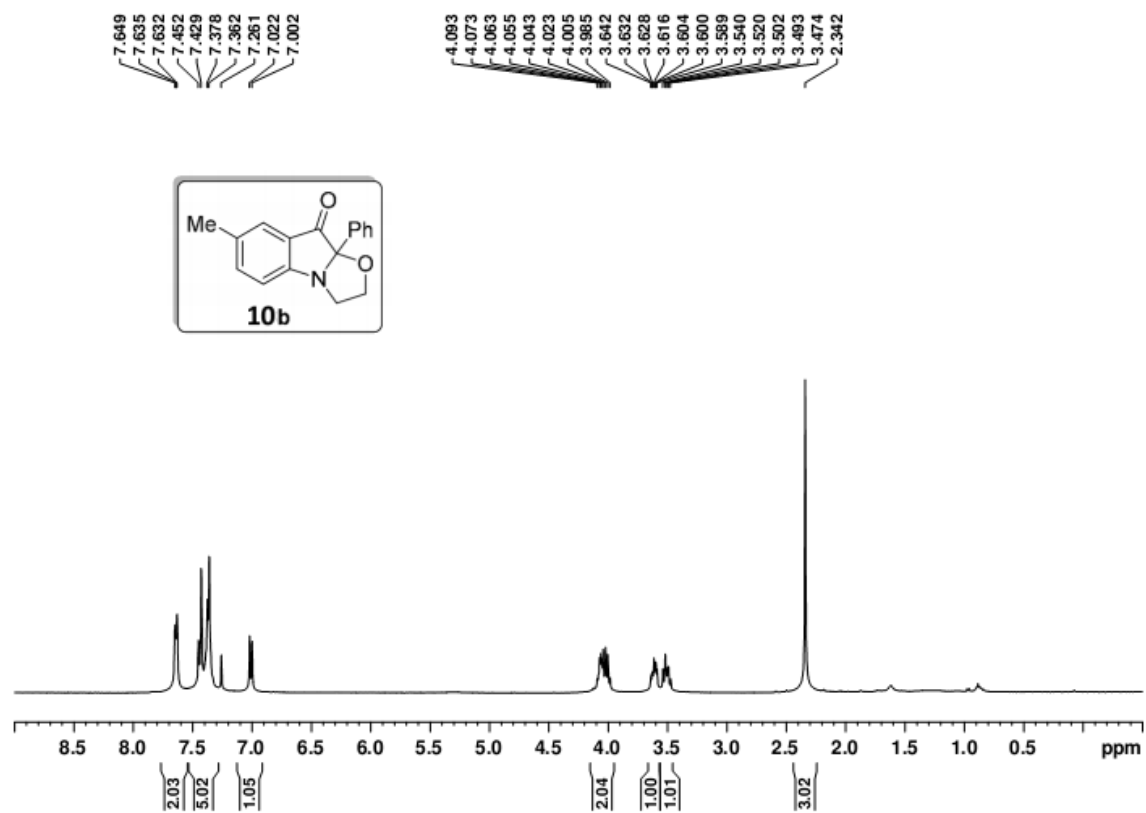


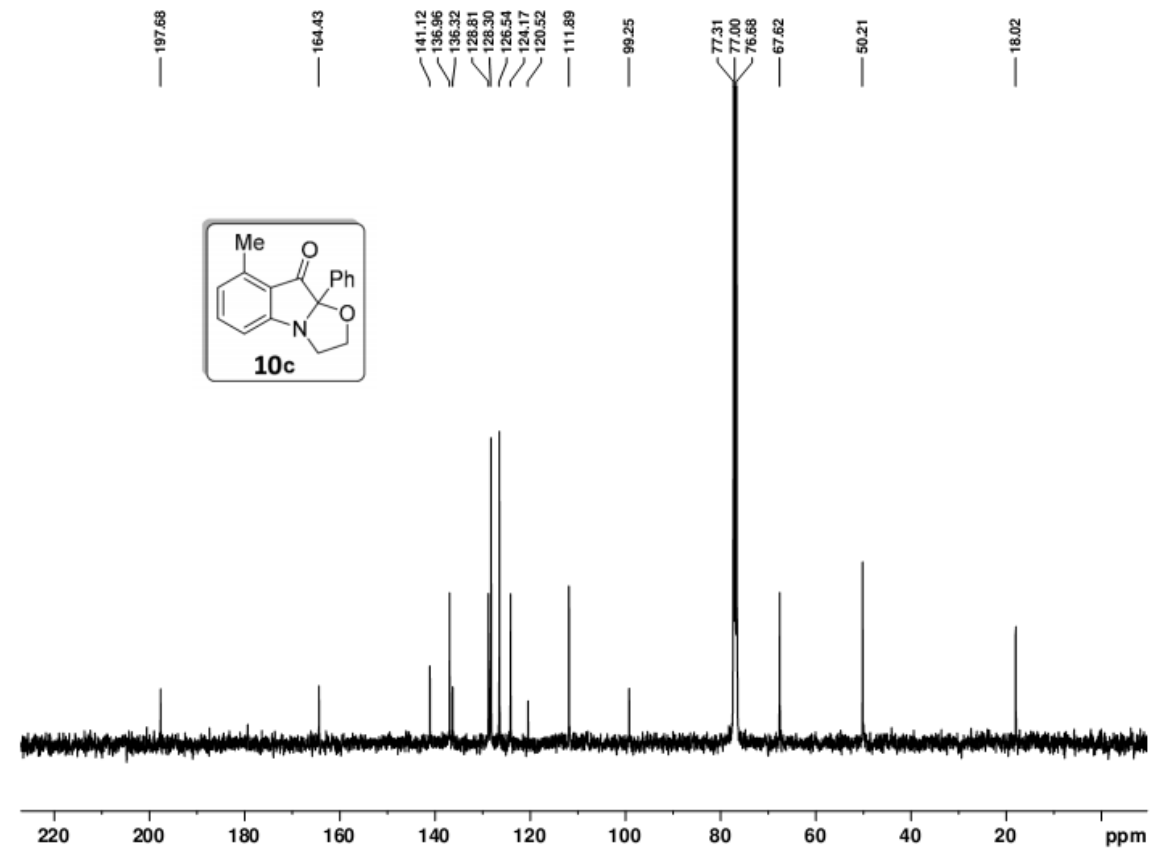
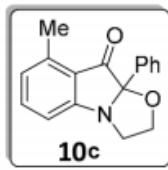
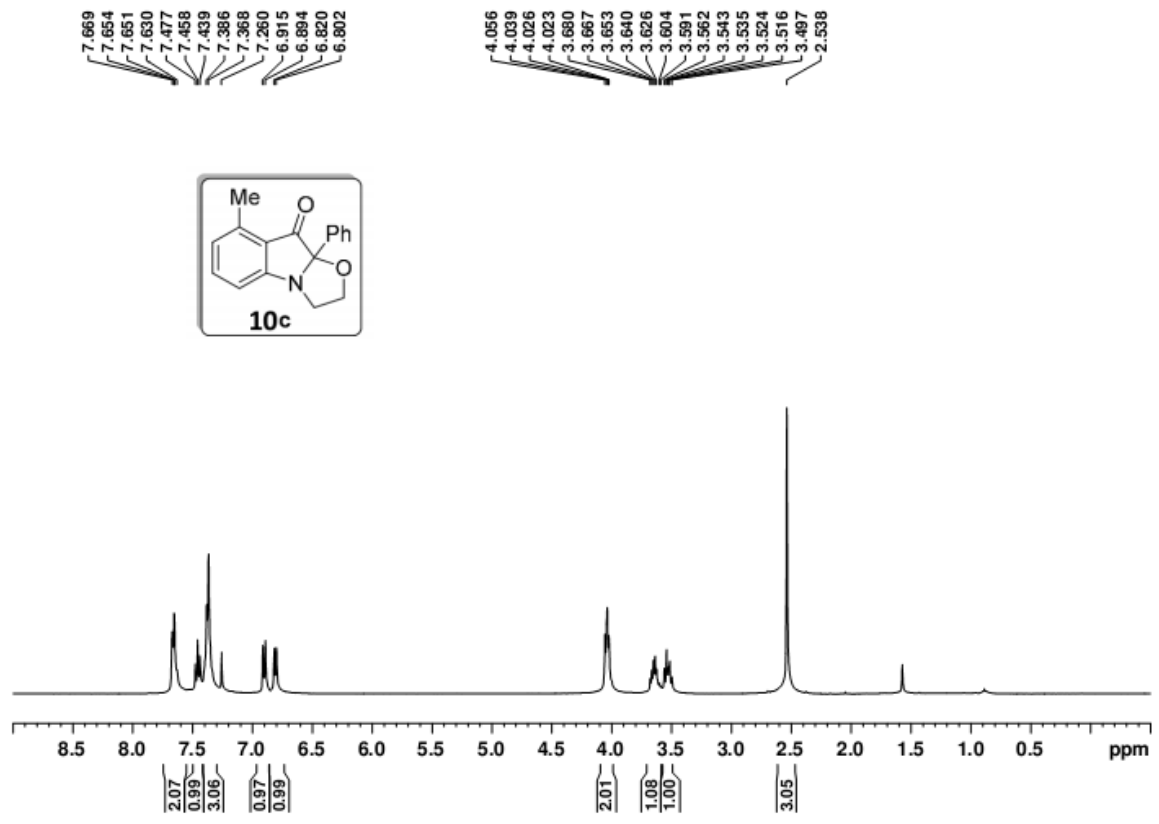


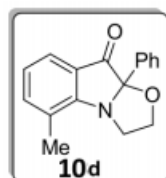
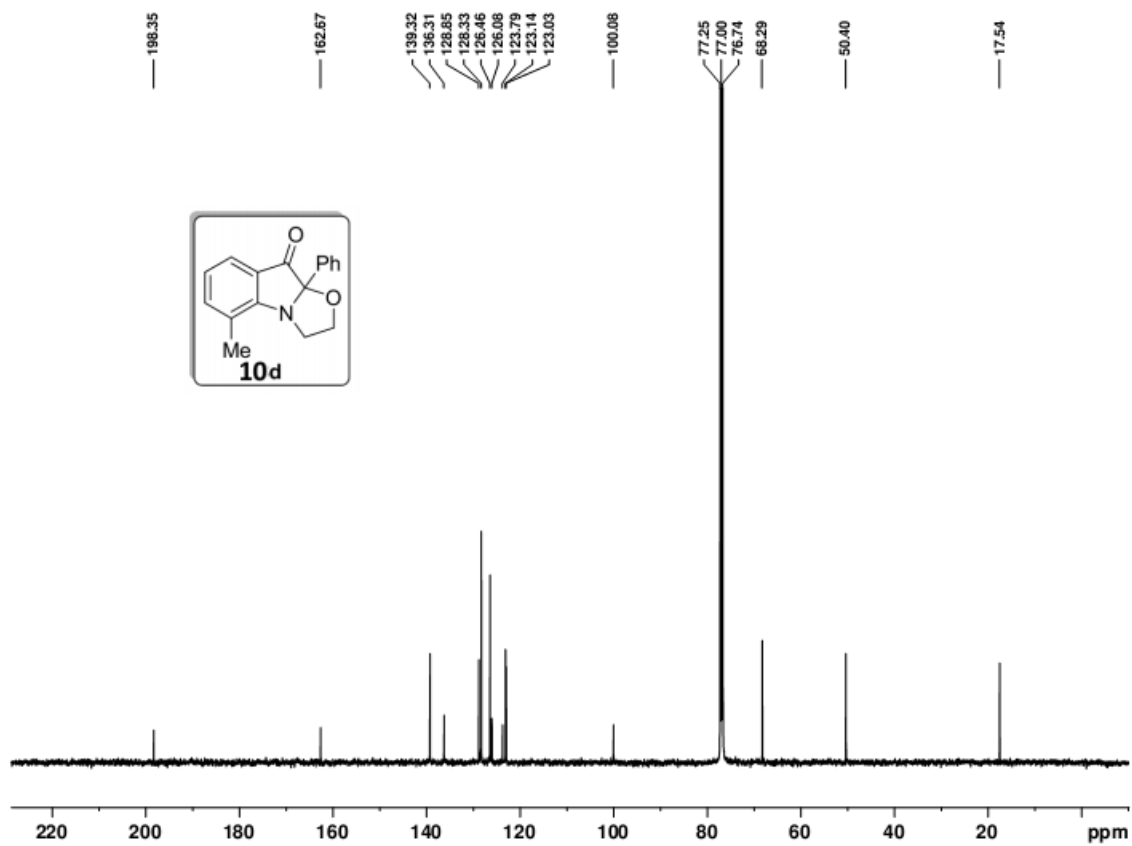
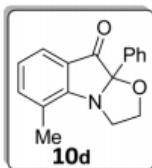
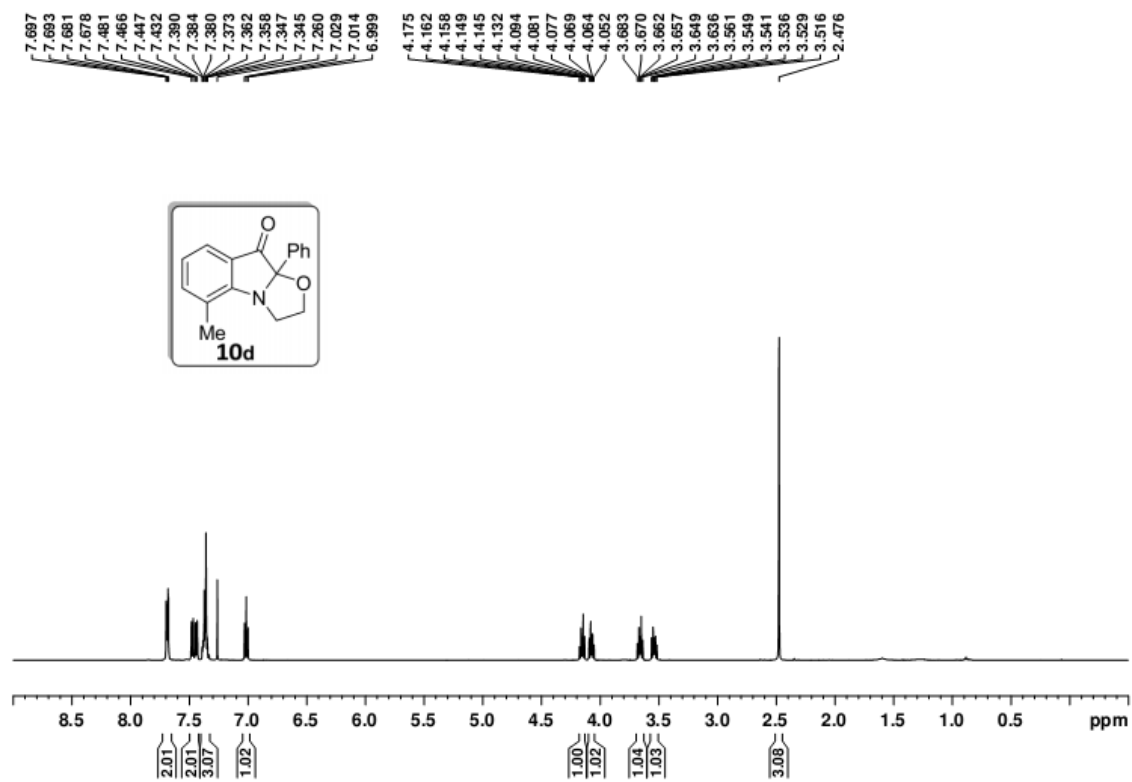




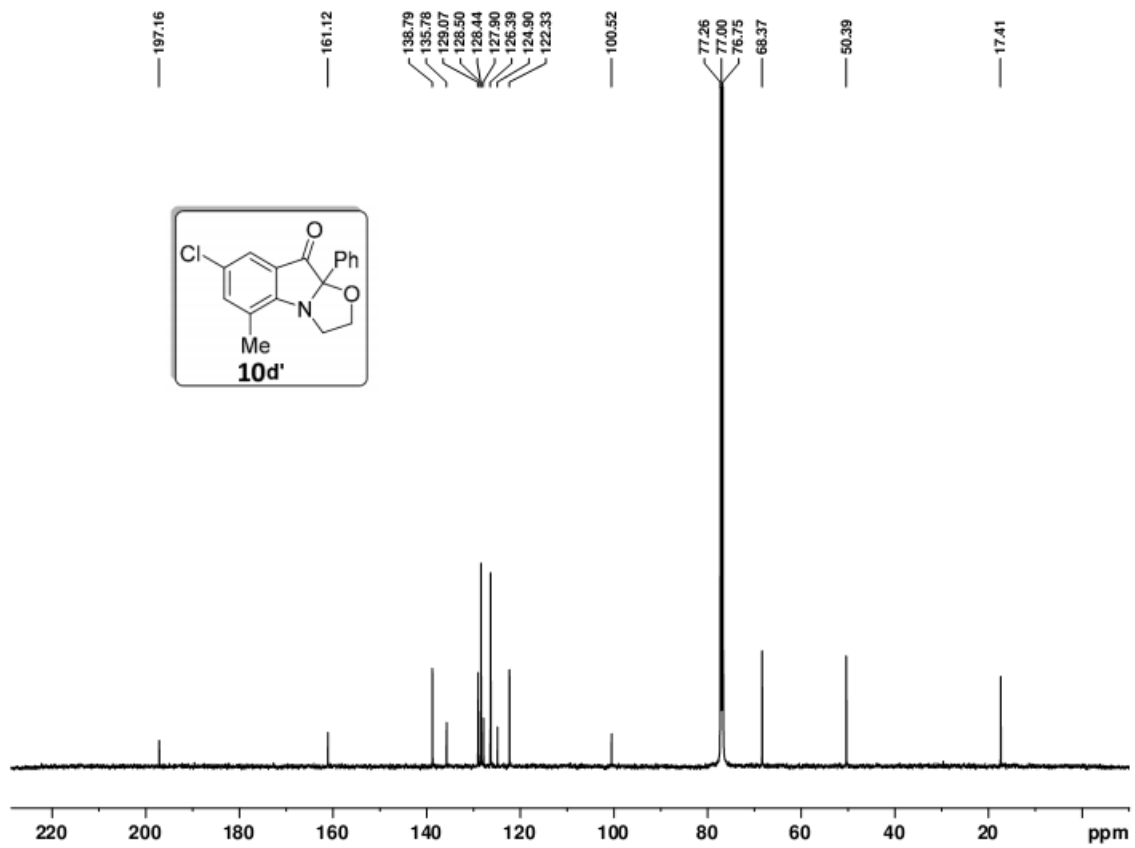
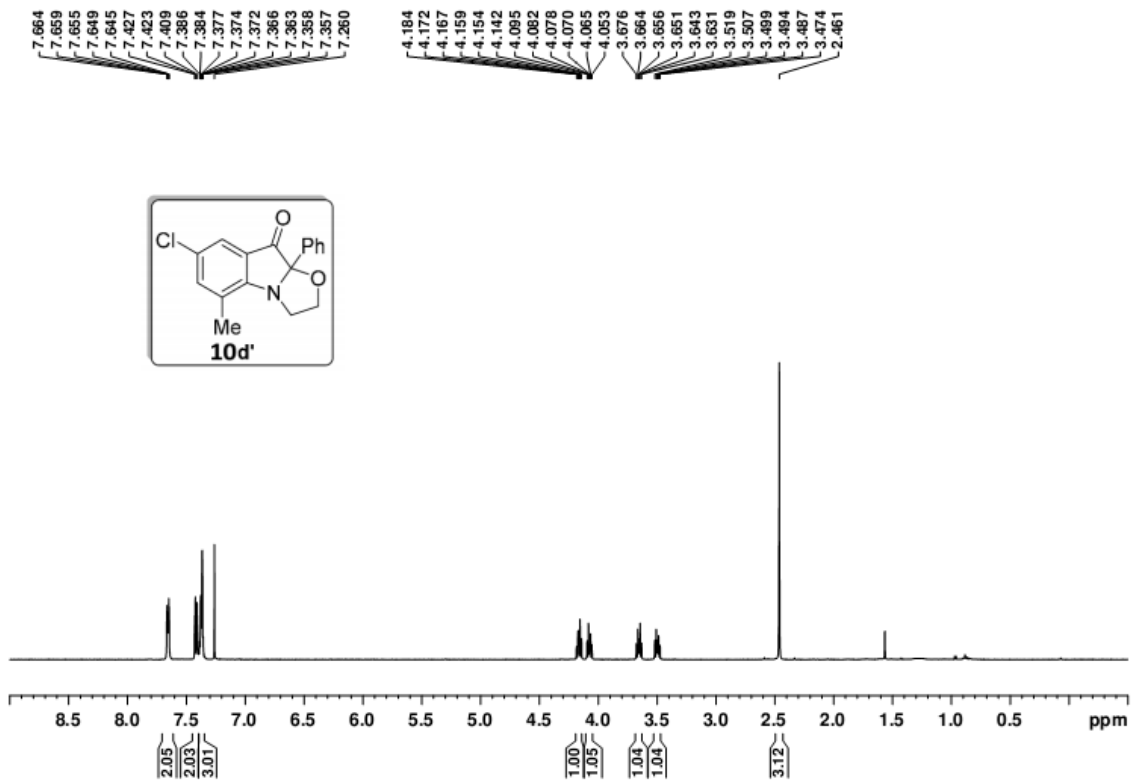






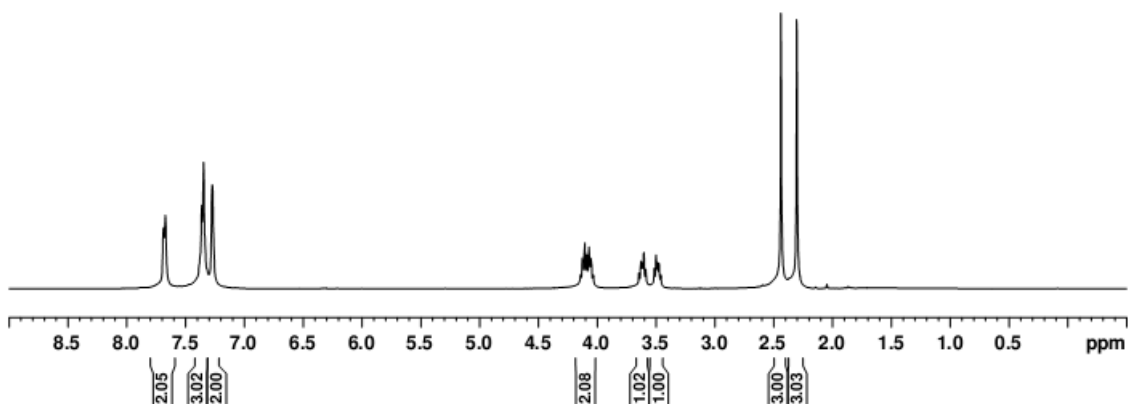
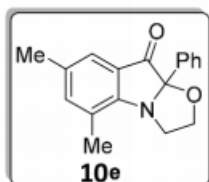




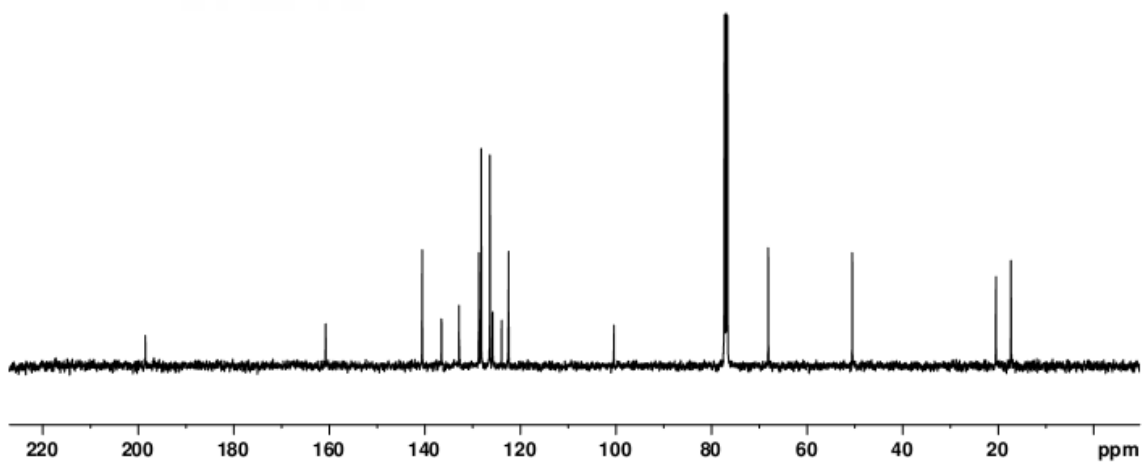
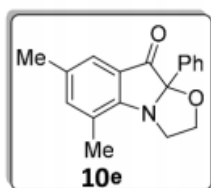


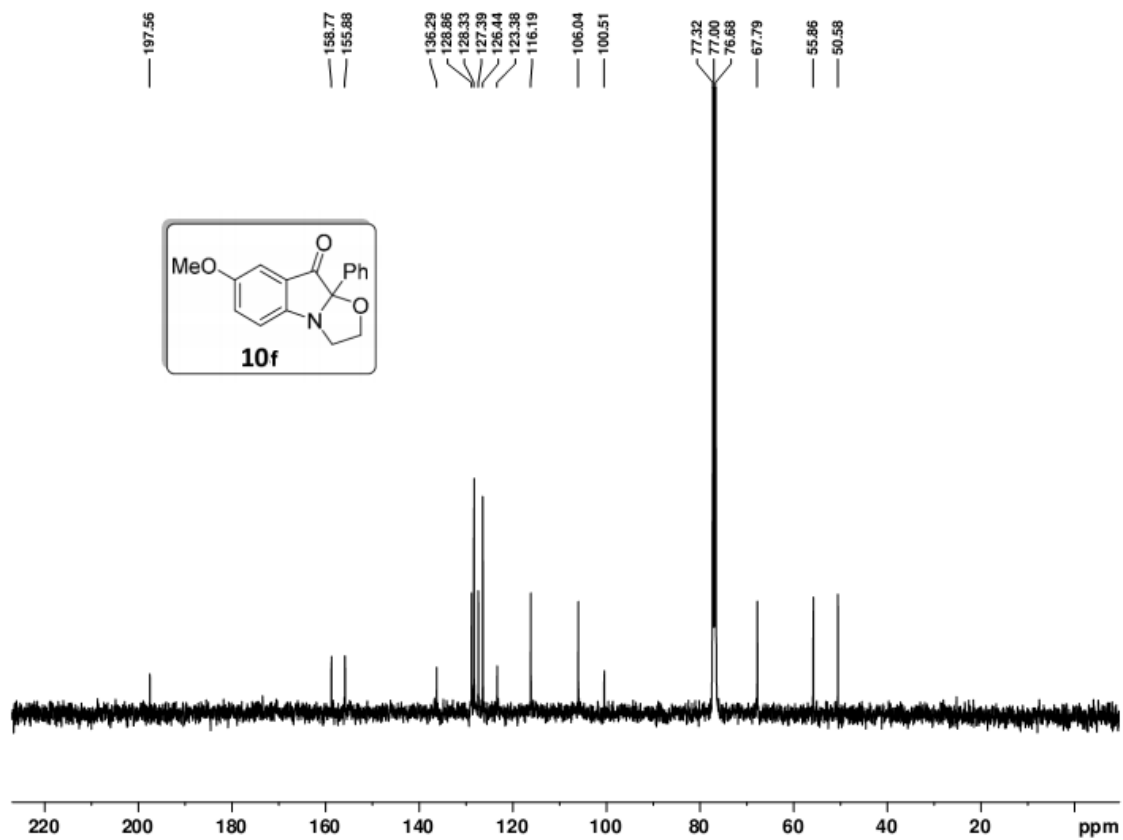
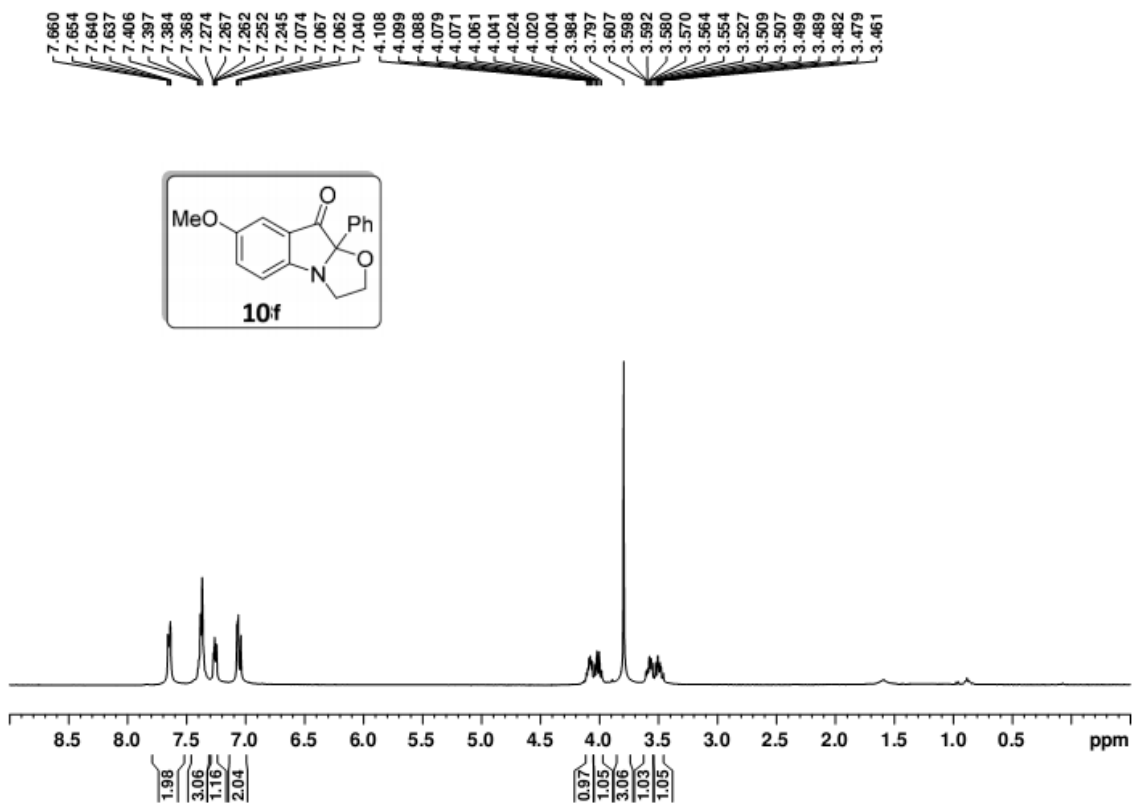
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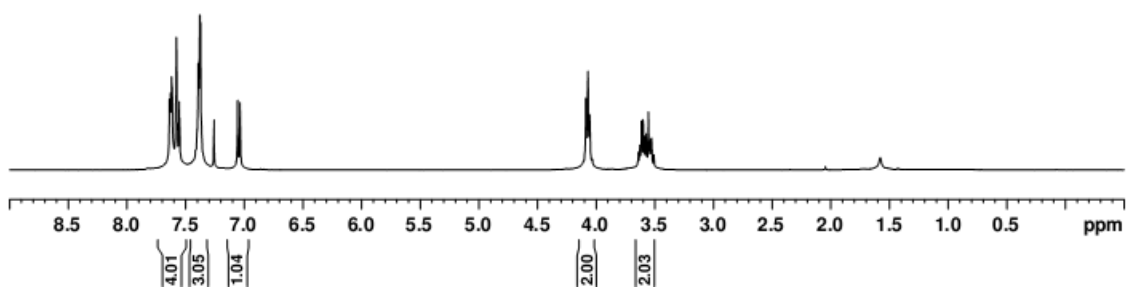
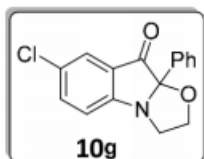
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17.35



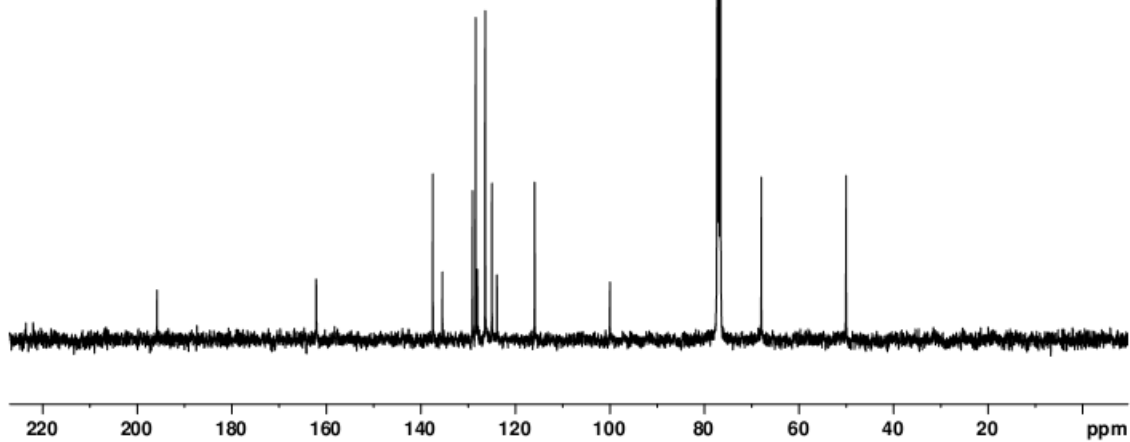
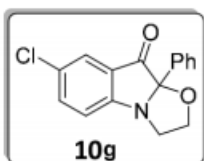


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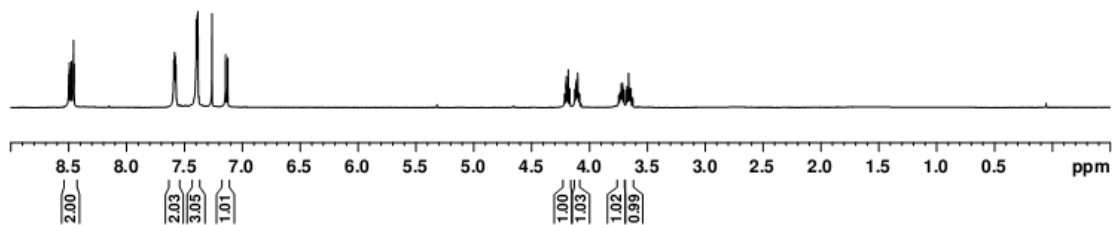
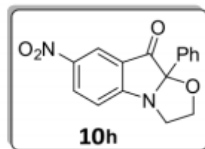
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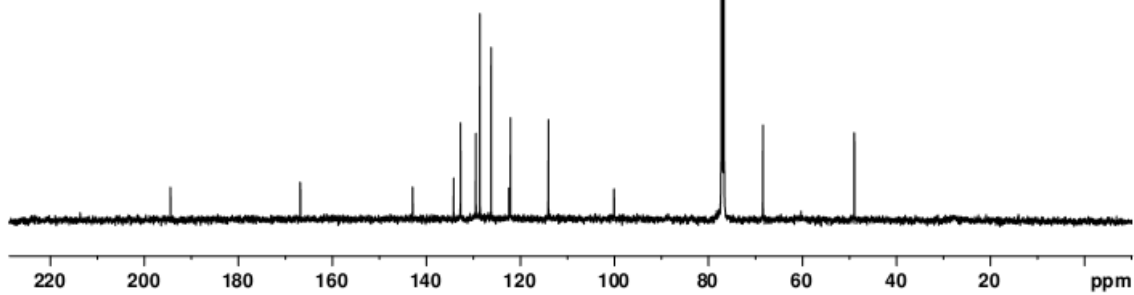
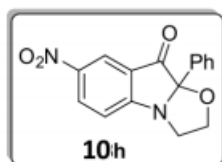
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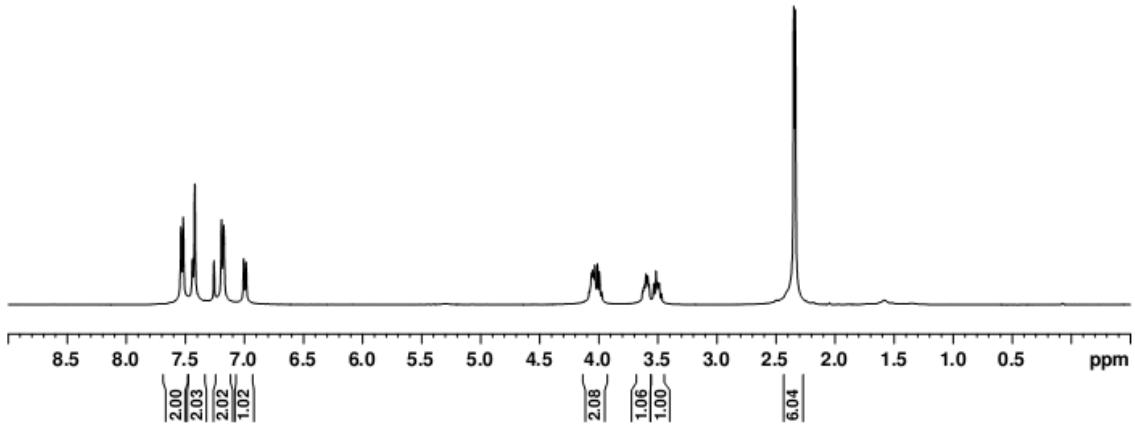
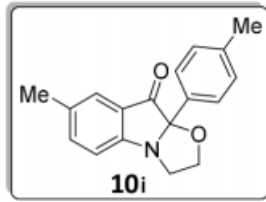


194.48  
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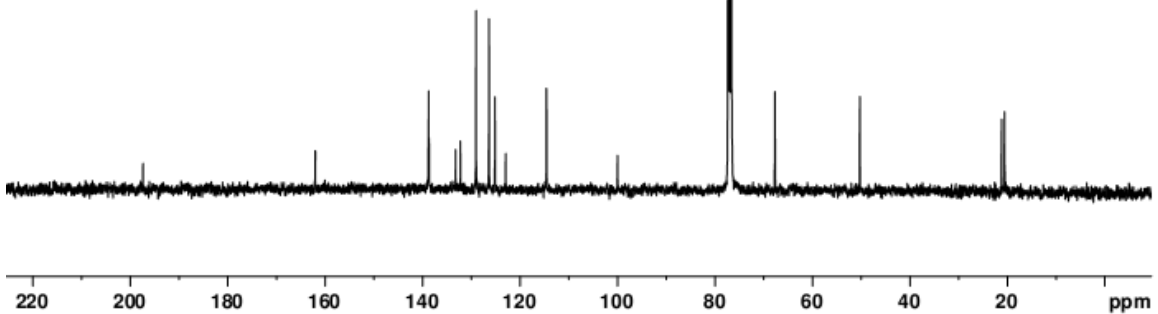
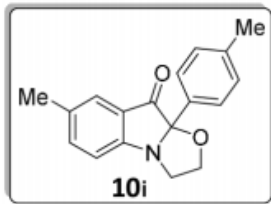


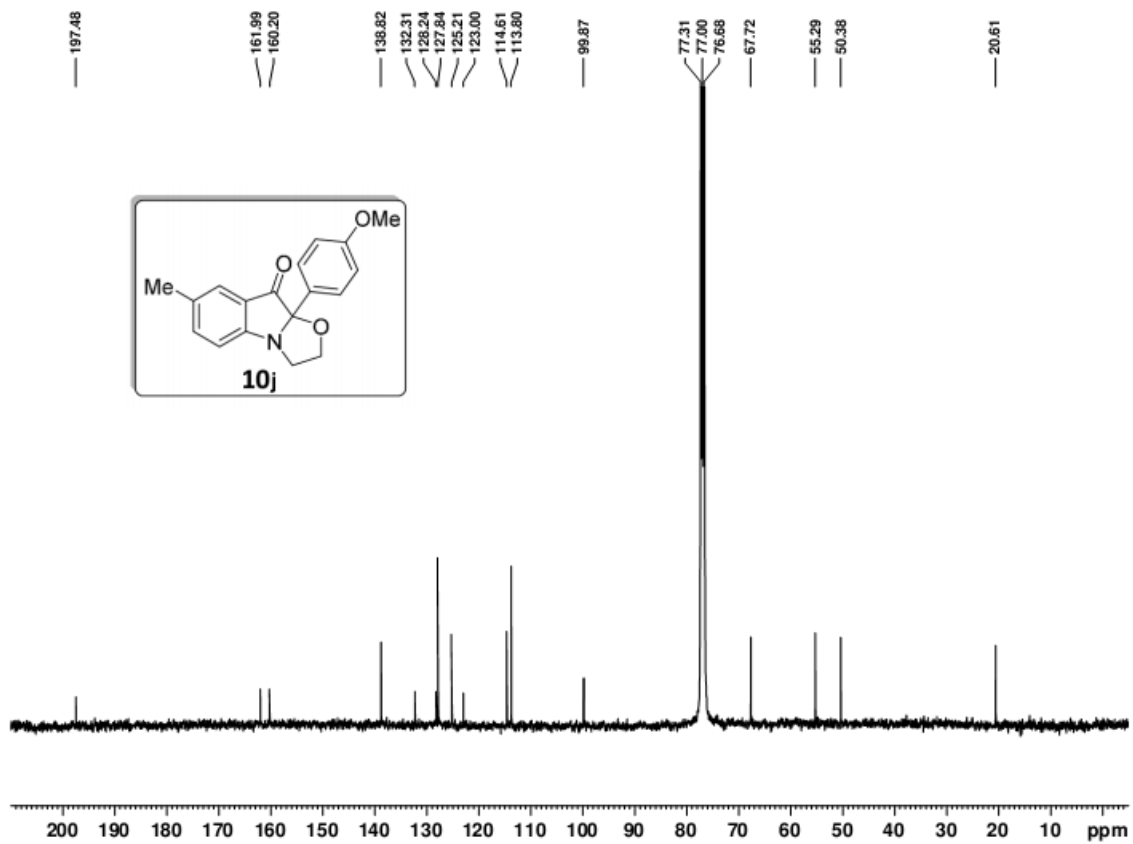
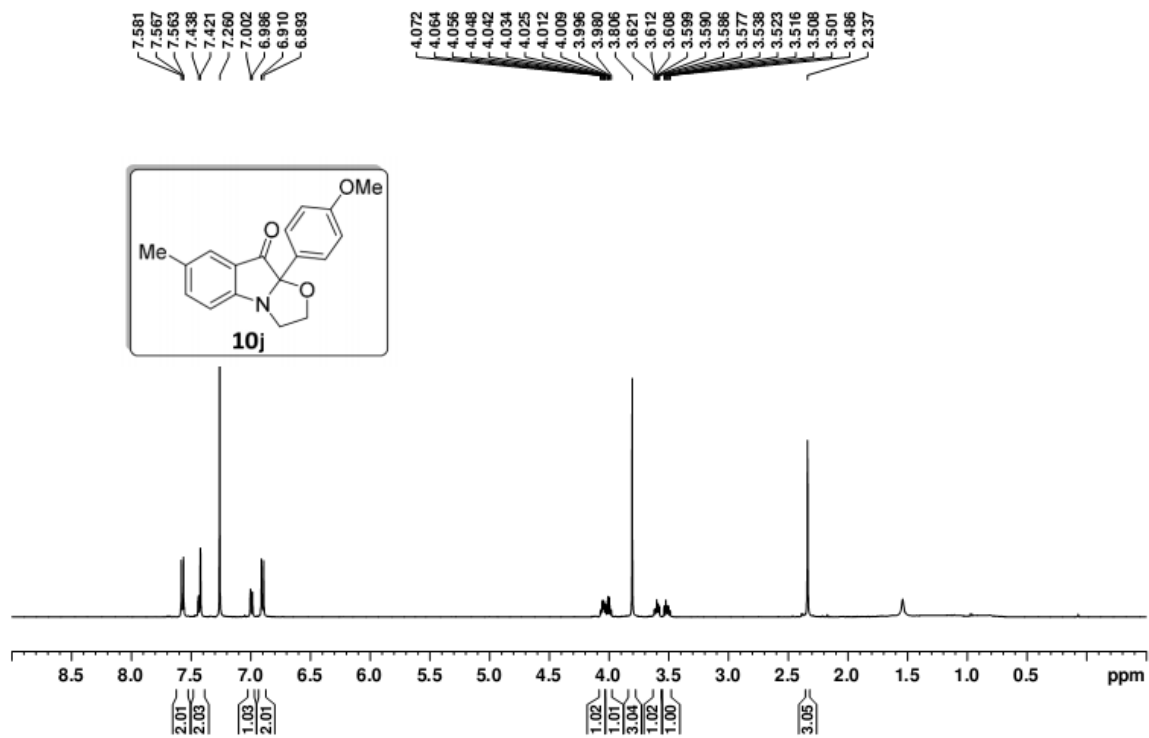
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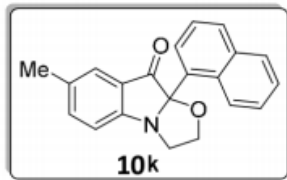
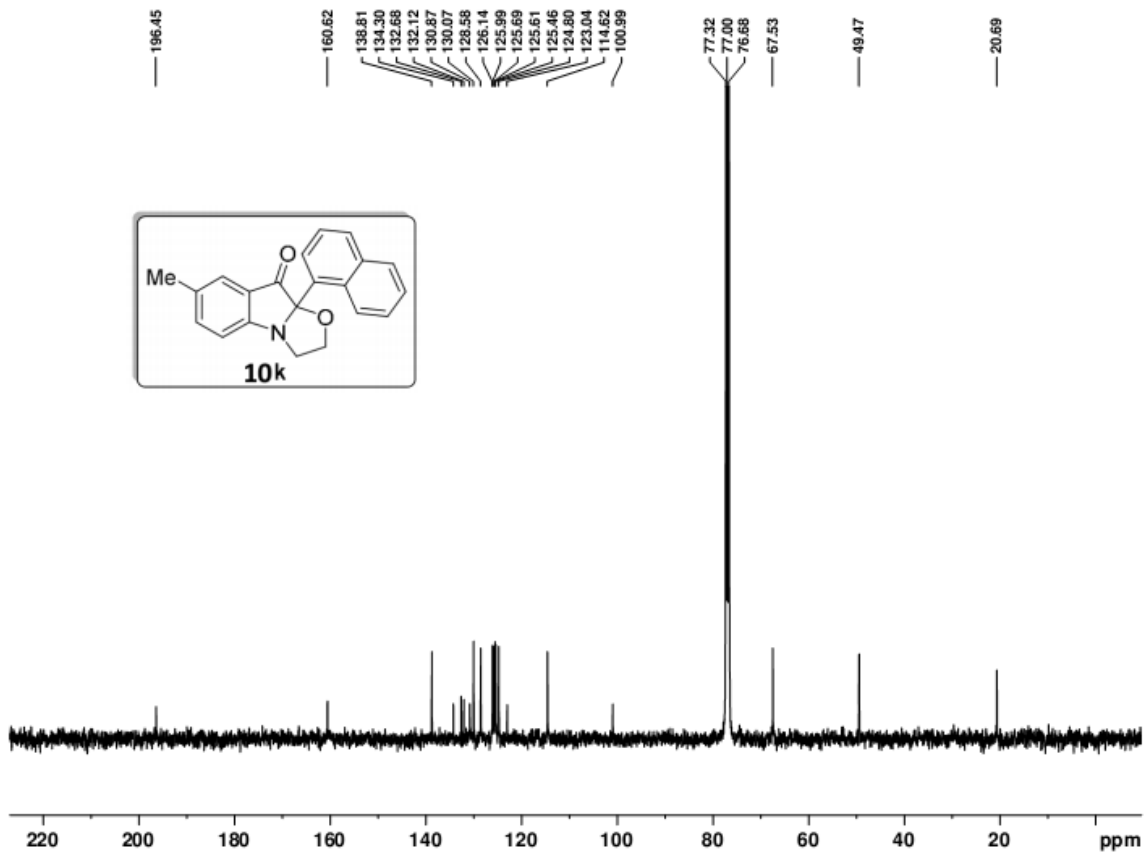
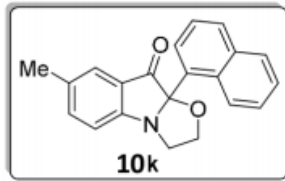
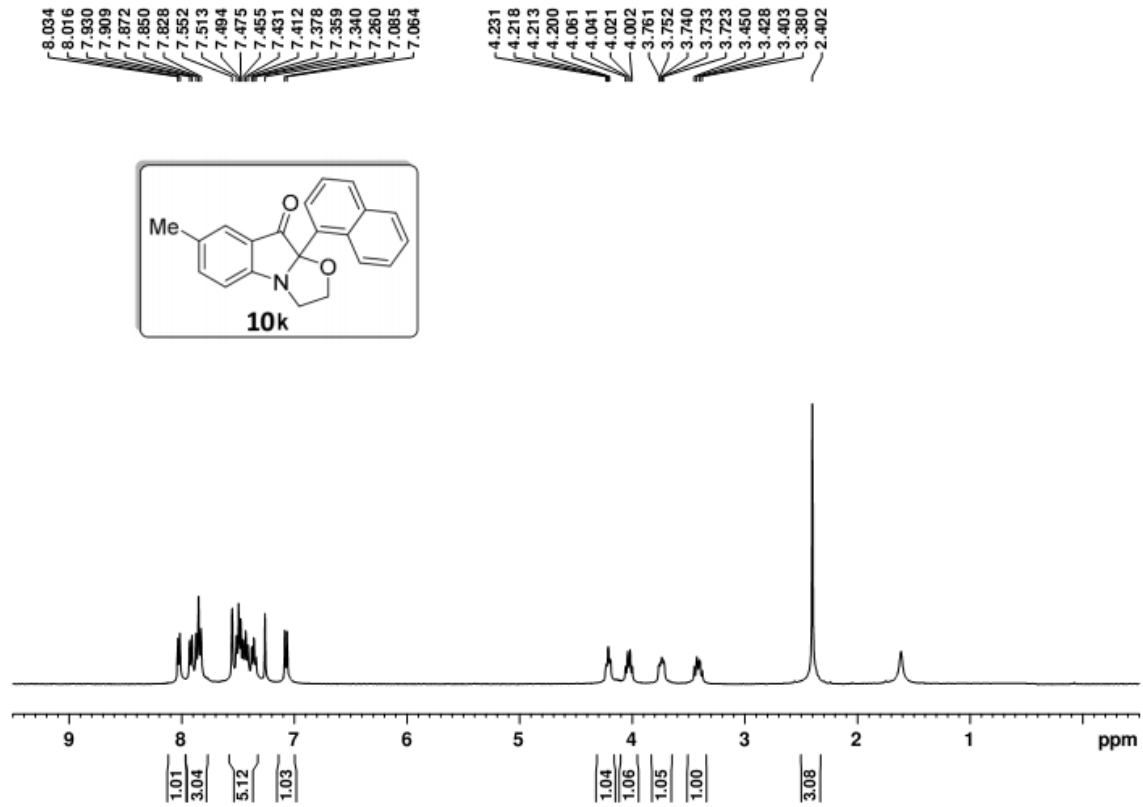
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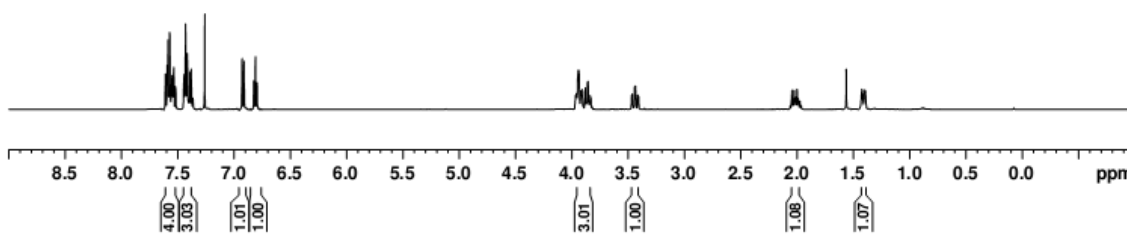
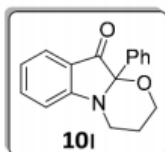








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