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Electrocatalytic intramolecular dehydrogenative annulation for the synthesis of indoles

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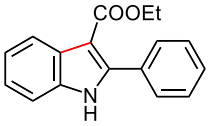
General information

All glassware was oven dried at 110 °C for hours and cooled down under vacuum. **1a-1o**,[1] **1p-1q**[2] were prepared according to reported procedures. The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). The anode electrode and cathode electrode all are platinum plate (15 mm×15 mm×0.3 mm). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 °C). Gas chromatographic analyses were performed on SHIMADZU GC-2014 gas chromatography instrument with a FID detector and naphthalene was added as internal standard. GC-MS spectra were recorded on Varian GC MS 3900-2100T or SHIMADZU GC MS-2010. ^{1}H and ^{13}C NMR data were recorded with Bruker Advance III (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.00 ppm, $CDCl_3$; 39.60 ppm, DMSO), respectively.

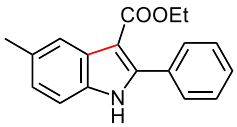
General procedure

In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, substrate (0.2 mmol), KI (249 mg, 1.5 mmol), DMF (10 mL) and H_2O (0.2 mL) were combined and added. The bottle was equipped with platinum plate (15 mm×15 mm×0.3 mm) as both the anode and cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 7 mA (J_{anode} = 12 mA/cm^2) under room temperature for 3 h (3.9 F). When the reaction was finished, the resulting reaction solution was quenched with 100 mL brine and extracted with 4 × 60 mL ethylacetate. The extract was dried with Na_2SO_4. The solvent was removed with a rotary evaporator. The pure product was obtained by flash column chromatography on silica gel (petroleum: ethyl ether = 10:1).

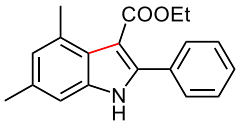
Detail descriptions for products



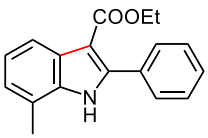
Ethyl 2-phenyl-1H-indole-3-carboxylate (2a):[3] white solid (mp. 145-148 °C) was obtained in 96% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (s, 1H), 8.30 – 8.11 (m, 1H), 7.68 – 7.48 (m, 2H), 7.39 – 7.32 (m, 3H), 7.32 – 7.19 (m, 3H), 4.23 (q, *J* = 7.1 Hz, 2H), 1.27 (t, *J* = 7.0 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 165.55, 144.63, 135.15, 131.90, 129.52, 129.02, 127.94, 127.48, 123.03, 121.93, 111.13, 104.34, 59.69, 14.22.



Ethyl 5-methyl-2-phenyl-1H-indole-3-carboxylate (2b):[3] white solid (mp. 144-145 °C) was obtained in 93% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (s, 1H), 8.03 (s, 1H), 7.67 – 7.59 (m, 2H), 7.48 – 7.39 (m, 3H), 7.26 (d, *J* = 9.3 Hz, 1H), 7.09 (dd, *J* = 8.2, 1.8 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 2.50 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.43, 144.38, 133.42, 132.19, 131.50, 129.55, 129.03, 128.03, 127.87, 124.71, 121.73, 110.60, 104.21, 59.61, 21.73, 14.28.

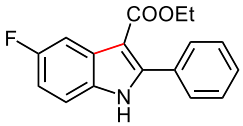


Ethyl 4,6-dimethyl-2-phenyl-1H-indole-3-carboxylate (2c): white solid (mp. 108-110 °C) was obtained in 94% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 7.47 – 7.41 (m, 2H), 7.37 – 7.29 (m, 3H), 6.87 (s, 1H), 6.81 (s, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 2.61 (s, 3H), 2.35 (s, 3H), 1.11 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.08, 140.42, 136.07, 132.98, 132.38, 130.79, 128.61, 128.41, 128.18, 125.20, 123.51, 108.72, 106.80, 60.40, 21.34, 21.04, 13.83. HRMS (ESI) calculated for $C_{19}H_{19}NO_2$ $[M+H]^+$: 294.1489; found: 294.1488.

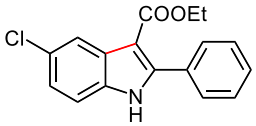


Ethyl 7-methyl-2-phenyl-1H-indole-3-carboxylate (2d):[3] white solid (mp. 145-146 °C) was obtained in 79% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (s, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.66 – 7.57 (m, 2H), 7.42 – 7.34 (m, 3H), 7.21 – 7.15 (m, 1H), 7.08 – 7.01 (m, 1H), 4.25 (q, *J* = 7.1

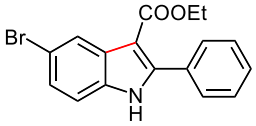
Hz, 2H), 2.47 (s, 3H), 1.28 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.43, 144.25, 134.66, 132.12, 129.58, 128.98, 127.94, 127.11, 123.69, 122.15, 120.22, 119.71, 105.03, 59.61, 16.49, 14.23.



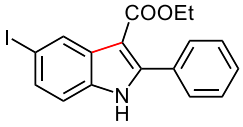
Ethyl 5-fluoro-2-phenyl-1H-indole-3-carboxylate (2e):[4] white solid (mp. 146-148 °C) was obtained in 86% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (s, 1H), 7.84 (dd, J = 10.1, 2.6 Hz, 1H), 7.60 – 7.51 (m, 2H), 7.40 – 7.31 (m, 3H), 7.20 (dd, J = 8.8, 4.4 Hz, 1H), 6.96 (td, J = 9.0, 2.6 Hz, 1H), 4.23 (q, J = 7.1 Hz, 2H), 1.28 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.06, 159.16 (d, $J_{C\text{-}F}$ = 237.2 Hz), 145.96, 131.58 (d, $J_{C\text{-}F}$ = 8.4 Hz), 129.47, 129.34, 128.31 (d, $J_{C\text{-}F}$ = 11.2 Hz), 128.11, 111.79 (d, $J_{C\text{-}F}$ = 9.9 Hz), 111.54 (d, $J_{C\text{-}F}$ = 26.6 Hz), 107.44 (d, $J_{C\text{-}F}$ = 25.5 Hz), 104.82 (d, $J_{C\text{-}F}$ = 4.5 Hz), 59.84, 14.27. ^{19}F NMR (377 MHz, $CDCl_3$) δ -121.22.



Ethyl 5-chloro-2-phenyl-1H-indole-3-carboxylate (2f):[4] white solid (mp. 149-151 °C) was obtained in 86% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (s, 1H), 8.17 (d, J = 2.0 Hz, 1H), 7.61 – 7.55 (m, 2H), 7.42 – 7.35 (m, 3H), 7.26 – 7.17 (m, 2H), 4.25 (q, J = 7.1 Hz, 2H), 1.29 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.02, 145.64, 133.47, 131.46, 129.48, 129.40, 128.64, 128.11, 127.79, 123.50, 121.66, 112.09, 104.31, 59.93, 14.28.

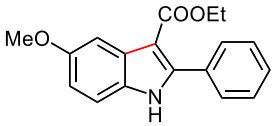


Ethyl 5-bromo-2-phenyl-1H-indole-3-carboxylate (2g):[3] white solid (mp. 148-150 °C) was obtained in 92% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (s, 1H), 8.35 (d, J = 1.9 Hz, 1H), 7.64 – 7.55 (m, 2H), 7.43 – 7.37 (m, 3H), 7.36 – 7.31 (m, 1H), 7.19 (d, J = 8.6 Hz, 1H), 4.26 (q, J = 7.1 Hz, 2H), 1.29 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.02, 145.46, 133.75, 131.38, 129.47, 129.37, 129.18, 128.07, 126.04, 124.66, 115.46, 112.51, 104.13, 59.93, 14.24.

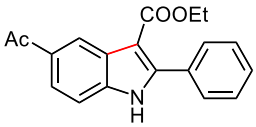


Ethyl 5-iodo-2-phenyl-1H-indole-3-carboxylate (2h):[3] white solid (mp. 140-142 °C) was obtained in 92% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.06 (s, 1H), 8.58 (d, J = 1.8 Hz, 1H), 7.60 –

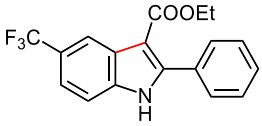
7.55 (m, 2H), 7.51 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.41 – 7.35 (m, 3H), 7.08 (d, *J* = 8.5 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.15, 145.12, 134.23, 131.45, 131.27, 130.75, 129.78, 129.43, 129.28, 127.98, 113.07, 103.60, 86.03, 59.92, 14.18.



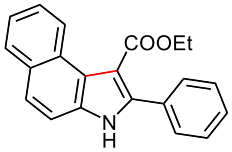
Ethyl 5-methoxy-2-phenyl-1H-indole-3-carboxylate (2i):[3] white solid (mp. 145-147 °C) was obtained in 86% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 7.74 (d, *J* = 2.5 Hz, 1H), 7.63 (dd, *J* = 6.7, 3.0 Hz, 2H), 7.43 (q, *J* = 3.2, 2.5 Hz, 3H), 7.26 (s, 1H), 6.91 (dd, *J* = 8.8, 2.5 Hz, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.90 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.42, 155.72, 144.69, 132.15, 130.08, 129.51, 129.05, 128.52, 128.02, 113.46, 111.77, 104.42, 103.56, 59.59, 55.67, 14.22.



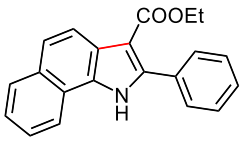
3-Ethyl 5-methyl 2-phenyl-1H-indole-3,5-dicarboxylate (2j): white solid (mp. 192-193 °C) was obtained in 82% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.33 (s, 1H), 8.89 (s, 1H), 7.93 (d, *J* = 8.6 Hz, 1H), 7.65 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.50 – 7.34 (m, 4H), 4.31 (q, *J* = 7.1 Hz, 2H), 2.69 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.70, 164.93, 145.86, 137.84, 131.62, 131.37, 129.56, 129.49, 128.14, 127.21, 124.54, 123.11, 111.25, 105.67, 59.98, 26.62, 14.20. HRMS (ESI) calculated for $C_{19}H_{17}NO_3$ $[M+H]^+$: 308.1281; found: 308.1286.



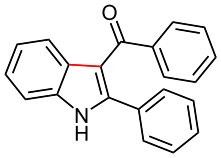
Ethyl 2-phenyl-5-(trifluoromethyl)-1H-indole-3-carboxylate (2k):[3] white solid (mp. 172-174 °C) was obtained in 56% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (s, 1H), 8.51 (s, 1H), 7.63 – 7.55 (m, 2H), 7.50 – 7.43 (m, 1H), 7.42 – 7.33 (m, 4H), 4.27 (q, *J* = 7.1 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.82, 145.99, 136.45, 131.25, 129.58, 129.51, 128.19, 127.06, 125.05 (q, J_{C-F} = 272.7Hz), 124.37 (q, J_{C-F} = 31.9Hz), 119.99 (q, J_{C-F} = 4.3Hz), 111.36, 105.37, 60.07, 14.19.



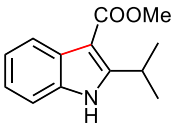
Ethyl 2-phenyl-3H-benzo[e]indole-1-carboxylate (2l): white solid (mp. 164-166 °C) was obtained in 95% isolated yield. ^{1}H NMR (400 MHz, CDCl3) δ 8.96 (d, *J* = 8.4 Hz, 1H), 8.77 (s, 1H), 7.95 – 7.85 (m, 1H), 7.64 (d, *J* = 8.8 Hz, 1H), 7.59 – 7.49 (m, 3H), 7.48 – 7.37 (m, 5H), 4.29 (q, *J* = 7.2 Hz, 2H), 1.13 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.60, 139.88, 132.53, 132.47, 130.38, 128.74, 128.44, 128.24, 128.00, 125.94, 125.07, 124.87, 123.94, 120.91, 112.21, 108.56, 60.67, 13.76. HRMS (ESI) calculated for $C_{21}H_{17}NO_2$ $[M+H]^+$: 316.1332; found: 316.1333.



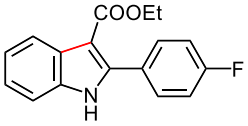
Ethyl 2-phenyl-1H-benzo[g]indole-3-carboxylate (2m):[3] white solid (mp. 197-198 °C) was obtained in 80% isolated yield. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.77 (s, 1H), 8.59 (dd, *J* = 8.2, 1.1 Hz, 1H), 8.21 (d, *J* = 8.8 Hz, 1H), 7.99 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.78 – 7.71 (m, 2H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.63 – 7.57 (m, 1H), 7.57 – 7.46 (m, 4H), 4.23 (q, *J* = 7.1 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.66, 142.75, 132.14, 130.51, 130.44, 130.20, 128.79, 128.51, 127.89, 126.01, 124.60, 123.63, 122.24, 121.71, 121.28, 120.87, 104.99, 59.28, 14.29.



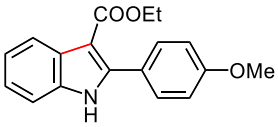
Phenyl(2-phenyl-1H-indol-3-yl)methanone (2n):[2] white solid was obtained in 62% isolated yield. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.23 (s, 1H), 7.78 – 7.72 (m, 1H), 7.55 – 7.50 (m, 3H), 7.42 – 7.34 (m, 3H), 7.30 – 7.14 (m, 7H). ^{13}C NMR (101 MHz, DMSO) δ 192.63, 144.60, 140.25, 136.28, 132.01, 131.82, 130.01, 129.53, 128.95, 128.63, 128.51, 128.25, 123.35, 121.90, 121.04, 112.56, 112.34.



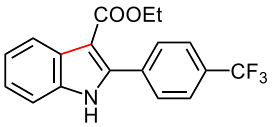
Methyl 2-isopropyl-1H-indole-3-carboxylate (2o):[3] pale yellow oil was obtained in 62% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 8.19 – 8.08 (m, 1H), 7.39 – 7.30 (m, 1H), 7.25 – 7.16 (m, 2H), 4.15 (p, *J* = 7.0 Hz, 1H), 3.94 (s, 3H), 1.36 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.44, 153.73, 134.41, 127.00, 122.29, 121.68, 121.43, 110.77, 102.64, 50.78, 26.28, 21.74.



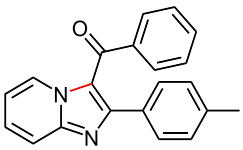
Ethyl 2-(4-fluorophenyl)-1H-indole-3-carboxylate (2p):[4] white solid (mp. 167-168 °C) was obtained in 89% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.82 (s, 1H), 8.23 – 8.15 (m, 1H), 7.61 – 7.53 (m, 2H), 7.35 – 7.30 (m, 1H), 7.29 – 7.21 (m, 2H), 7.04 (t, *J* = 8.7 Hz, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.49, 163.11 (q, J_{C-F} = 250.5 Hz), 143.50, 135.11, 131.45 (q, J_{C-F} = 8.4 Hz), 127.94 (q, J_{C-F} = 3.2 Hz), 127.39, 123.23, 122.06 (q, J_{C-F} = 6.3 Hz), 115.17, 114.95, 111.08, 104.56, 59.78, 14.26. ^{19}F NMR (377 MHz, $CDCl_3$) δ -111.59.



Ethyl 2-(4-methoxyphenyl)-1H-indole-3-carboxylate (2q):[4] white solid (mp. 164-165 °C) was obtained in 86% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.26 – 8.10 (m, 1H), 7.53 (d, *J* = 8.8 Hz, 2H), 7.35 – 7.28 (m, 1H), 7.27 – 7.18 (m, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.76 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.68, 160.18, 144.76, 135.03, 130.86, 127.59, 124.06, 122.84, 121.91, 121.84, 113.44, 111.00, 103.85, 59.67, 55.24, 14.34.

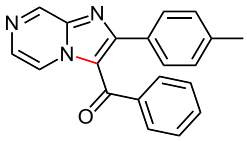


Ethyl 2-(4-(trifluoromethyl)phenyl)-1H-indole-3-carboxylate (2r): white solid (mp. 151-152 °C) was obtained in 80% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (s, 1H), 8.28 – 8.13 (m, 1H), 7.69 (d, *J* = 8.1 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.38 – 7.31 (m, 1H), 7.31 – 7.22 (m, 2H), 4.26 (q, *J* = 7.1 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.39, 142.52, 135.47, 135.35, 130.81 (q, J_{C-F} = 32.7 Hz), 129.95, 127.25, 124.88 (q, J_{C-F} = 3.7 Hz), 123.87 (q, J_{C-F} = 273.1 Hz),123.64, 122.33, 122.16, 111.25, 105.33, 59.97, 14.19. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.68. HRMS (ESI) calculated for $C_{18}H_{14}F_3NO_2$ $[M+H]^+$: 334.1049; found: 334.1056.



Phenyl(2-(p-tolyl)imidazo[1,2-a]pyridin-3-yl)methanone (3a): white solid was obtained in 97% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.52 (dt, *J* = 6.9, 1.2 Hz, 1H), 7.79 (dt, *J* = 8.9, 1.2 Hz,

1H), 7.56 – 7.46 (m, 3H), 7.29 – 7.24 (m, 1H), 7.24 – 7.19 (m, 2H), 7.13 – 7.04 (m, 3H), 6.88 (d, *J* = 7.9 Hz, 2H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 187.41, 155.03, 147.37, 138.68, 138.16, 131.64, 130.97, 130.09, 129.58, 129.12, 128.45, 128.20, 127.75, 119.85, 117.34, 114.48, 21.23. HRMS (ESI) calculated for $C_{21}H_{16}N_2O$ $[M+H]^+$: 313.1335; found: 313.1324.



Phenyl(2-(p-tolyl)imidazo[1,2-a]pyrazin-3-yl)methanone (3b): white solid was obtained in 94% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.29 (d, *J* = 1.5 Hz, 1H), 9.21 (dd, *J* = 4.6, 1.5 Hz, 1H), 8.16 (d, *J* = 4.7 Hz, 1H), 7.62 – 7.51 (m, 2H), 7.38 – 7.31 (m, 1H), 7.28 – 7.23 (m, 2H), 7.15 (t, *J* = 7.8 Hz, 2H), 6.93 (d, *J* = 7.9 Hz, 2H), 2.25 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 187.57, 154.36, 143.62, 141.33, 138.86, 137.40, 132.47, 131.85, 129.99, 129.89, 129.53, 128.66, 127.95, 120.06, 21.19. HRMS (ESI) calculated for $C_{20}H_{15}N_3O$ $[M+H]^+$: 313.1288; found: 314.1293.

References

(1) Bartoli, G.; Bosco, M.; Locatelli, M.; Marcantoni, E.; Melchiorre, P.; Sambri, L. *Synlett* **2004**, *2004*, 0239.

(2) Liu, J.; Wei, W.; Zhao, T.; Liu, X.; Wu, J.; Yu, W.; Chang, J. *J. Org. Chem.* **2016**, *81*, 9326.

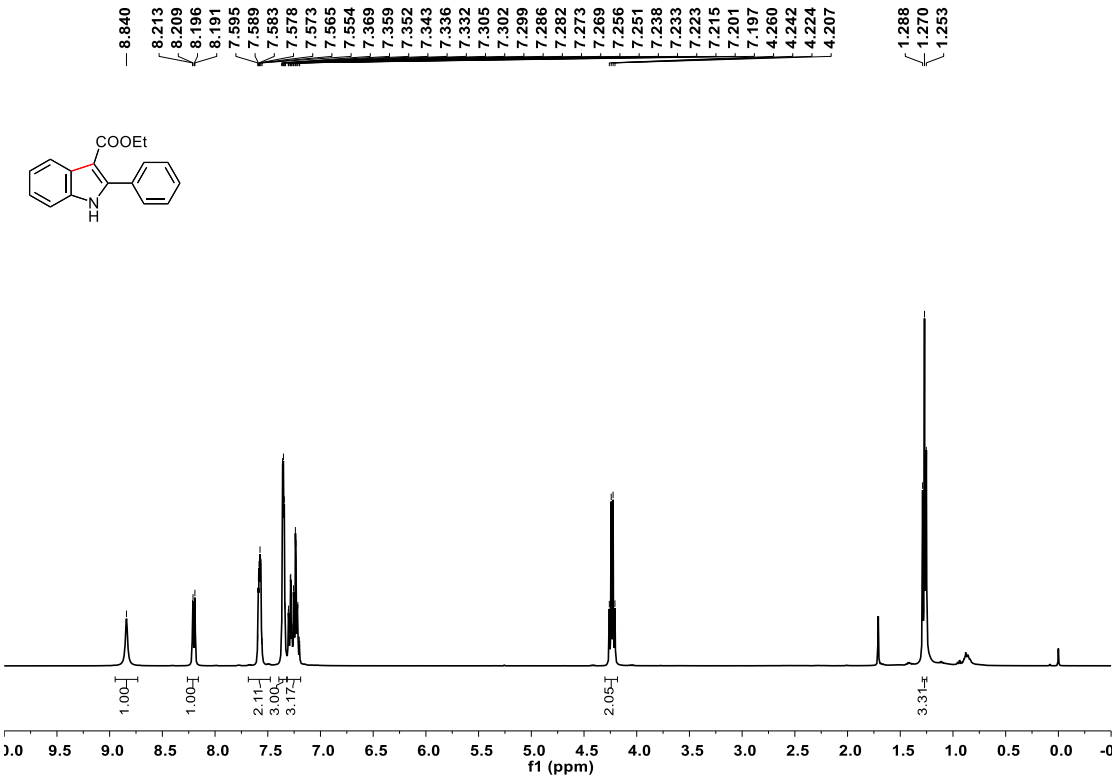
(3) He, Z.; Liu, W.; Li, Z. *Chem. –Asian. J* **2011**, *6*, 1340.

(4) Wu, C.-J.; Meng, Q.-Y.; Lei, T.; Zhong, J.-J.; Liu, W.-Q.; Zhao, L.-M.; Li, Z.-J.; Chen, B.; Tung, C.-H.; Wu, L.-Z. *ACS Catalysis* **2016**, *6*, 4635.

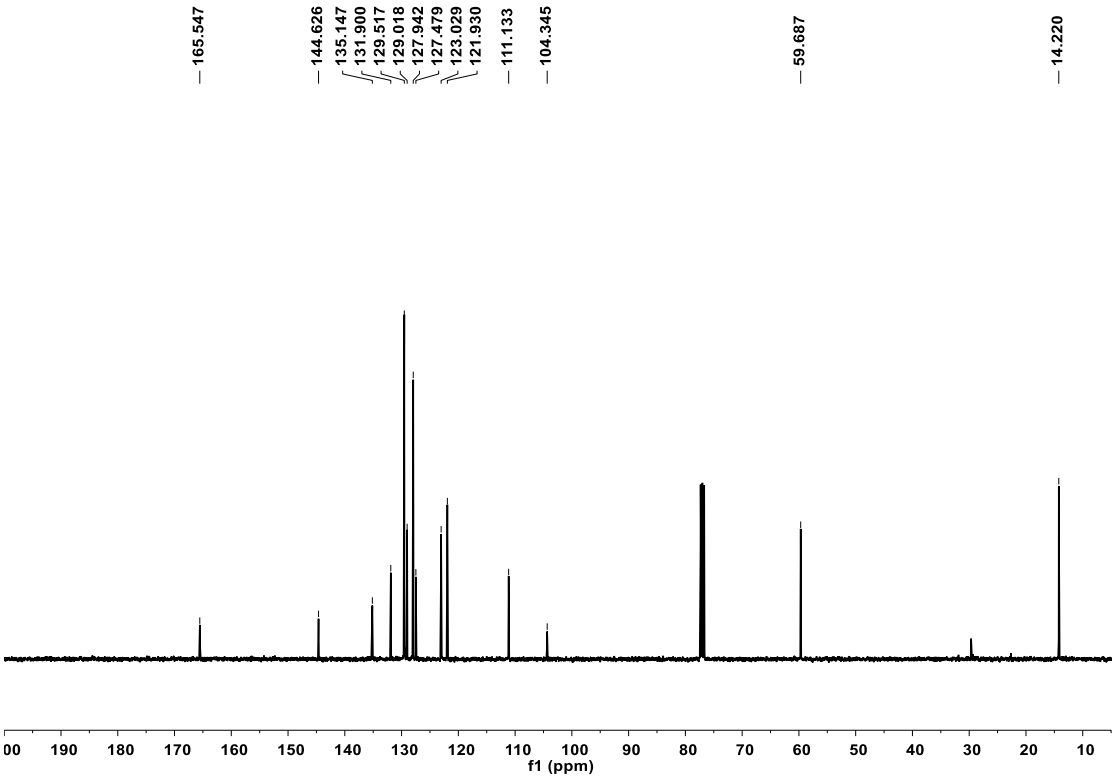
Copies of product NMR Spectra

2a

^{1}H NMR

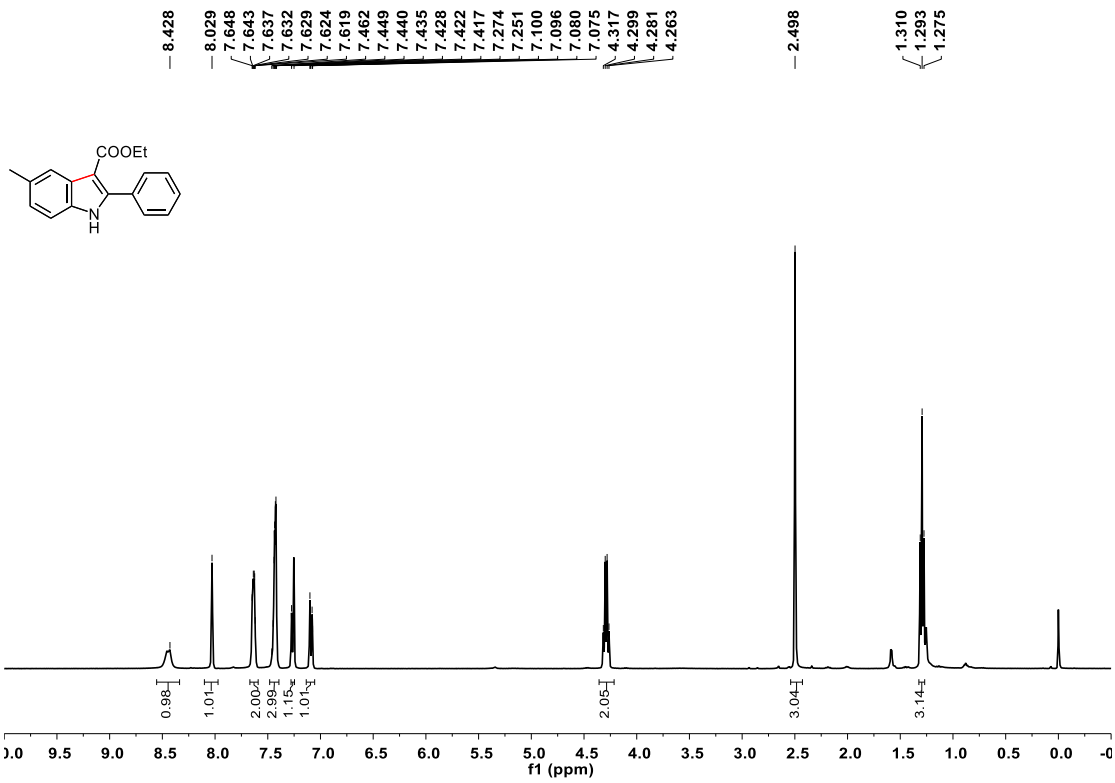


^{13}C NMR



2b

^{1}H NMR



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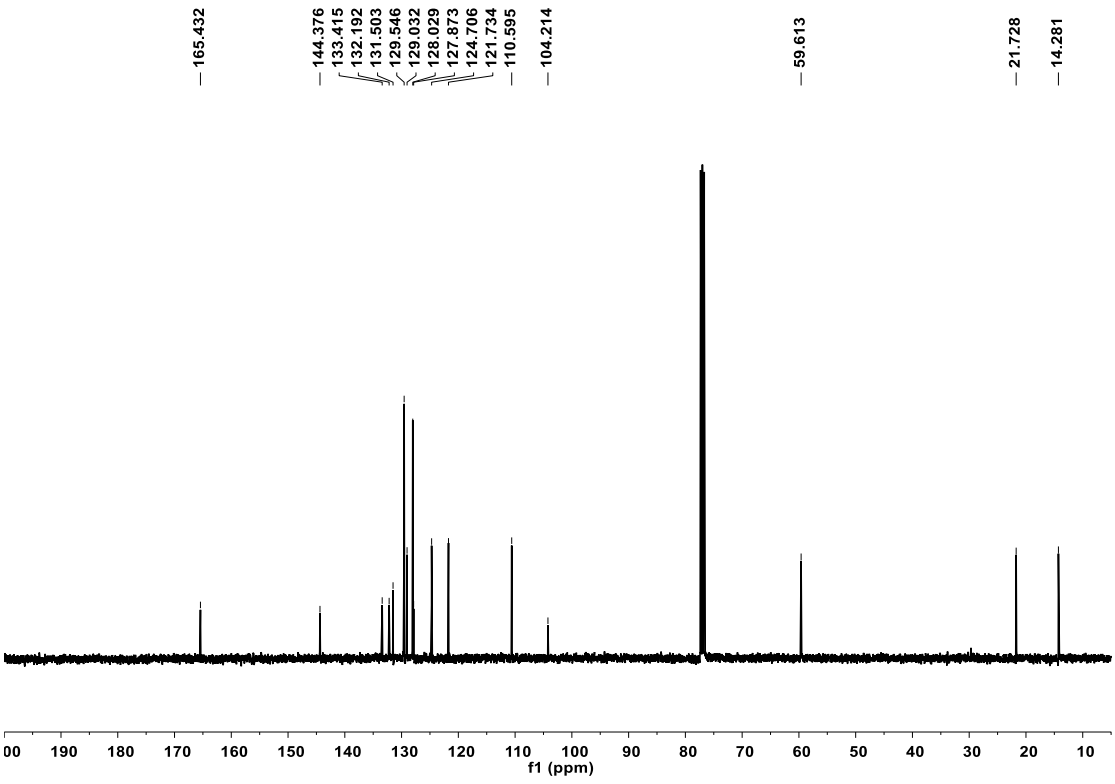

2c

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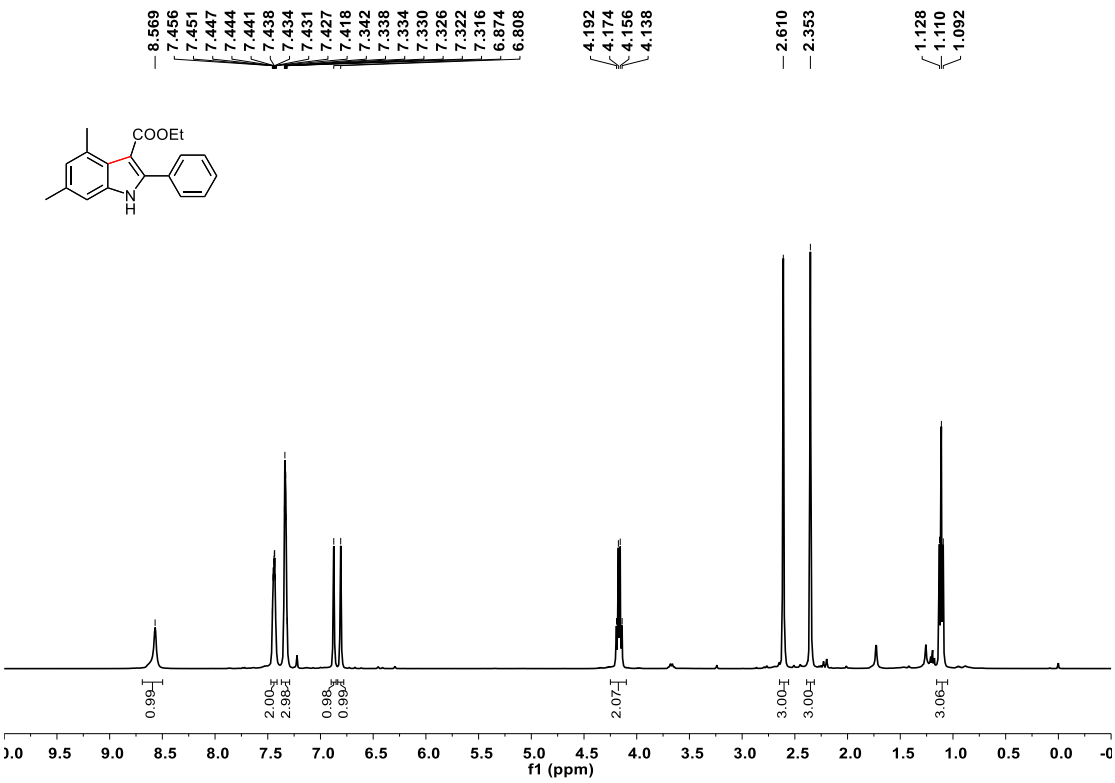

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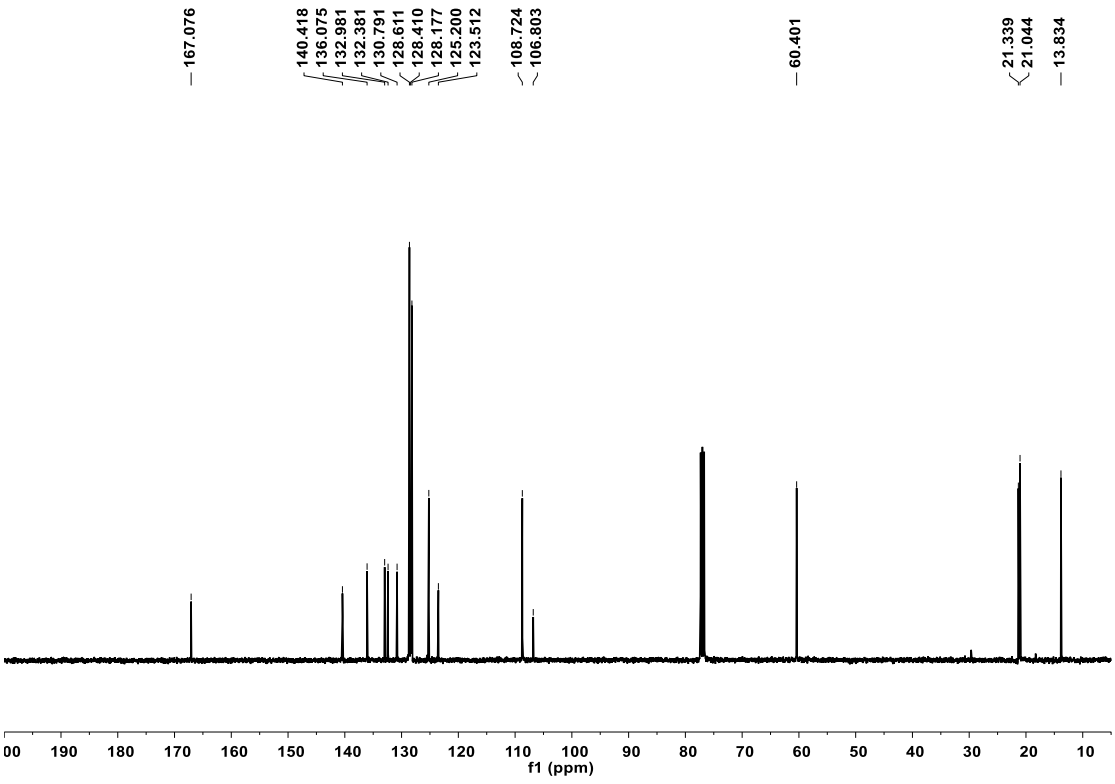

2d

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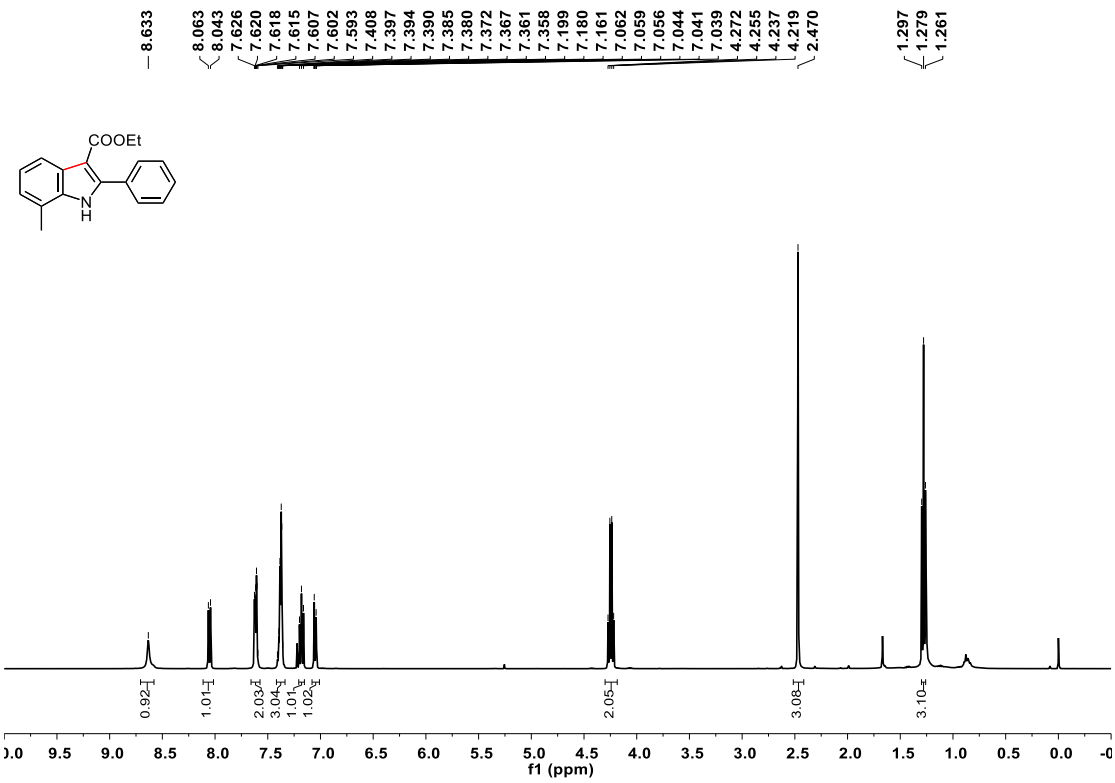

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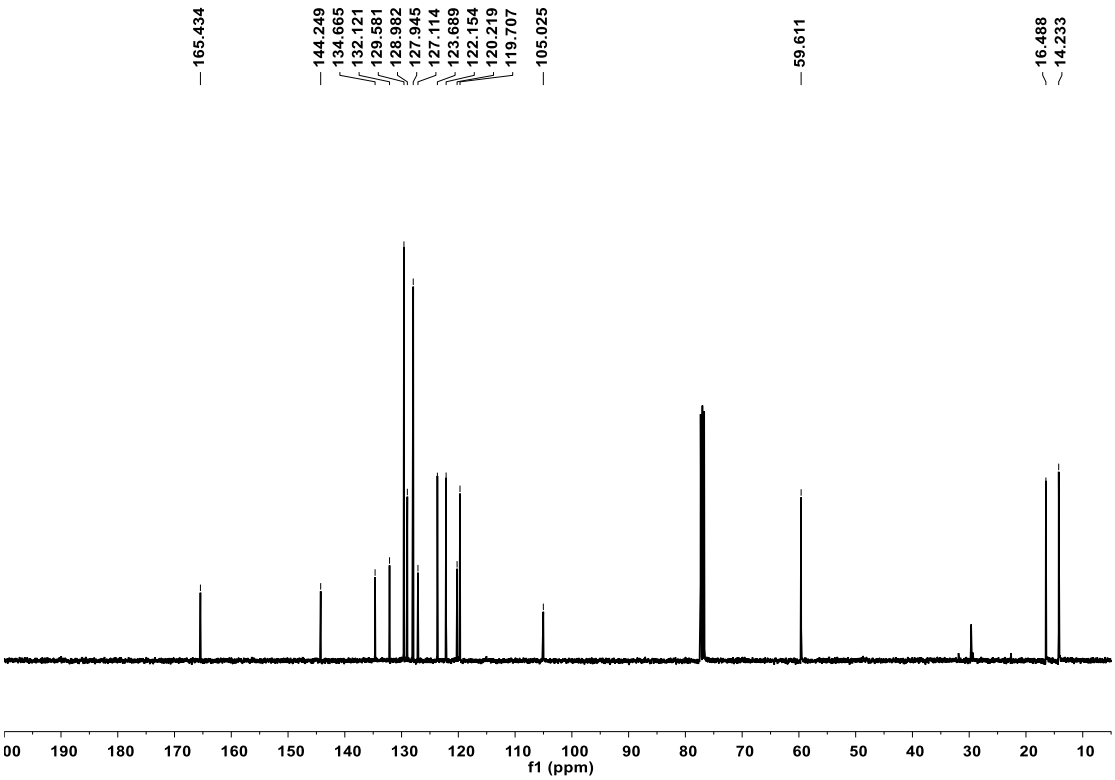

2e

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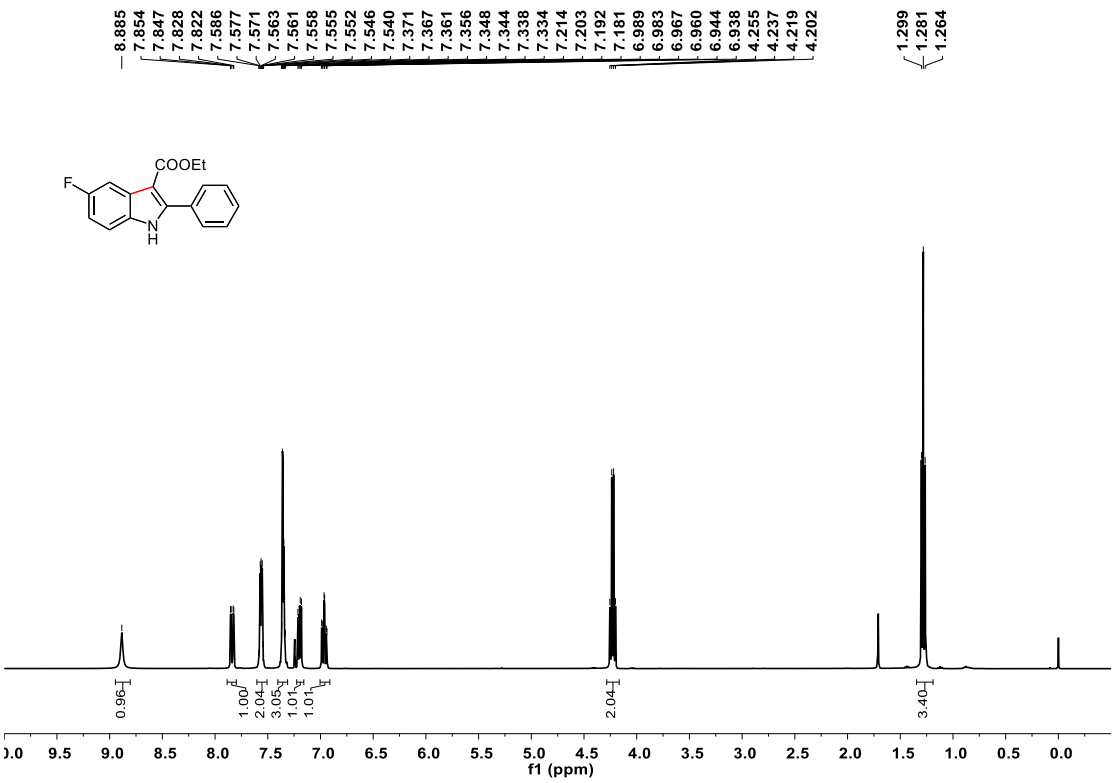

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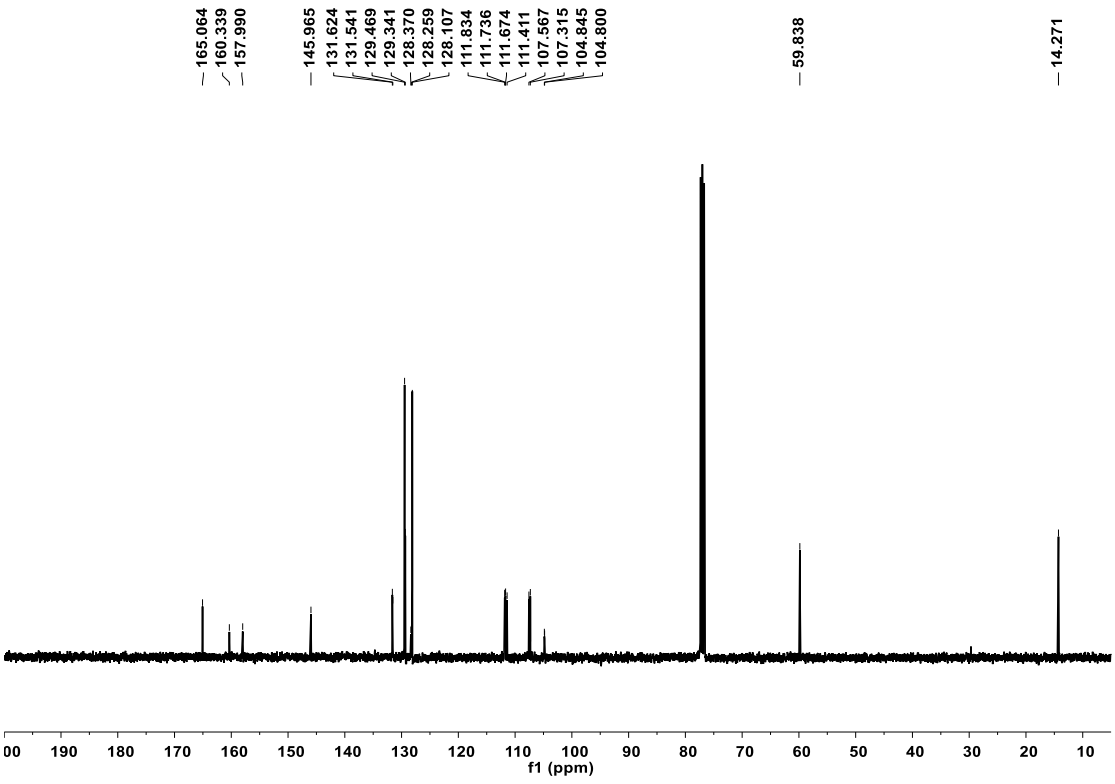

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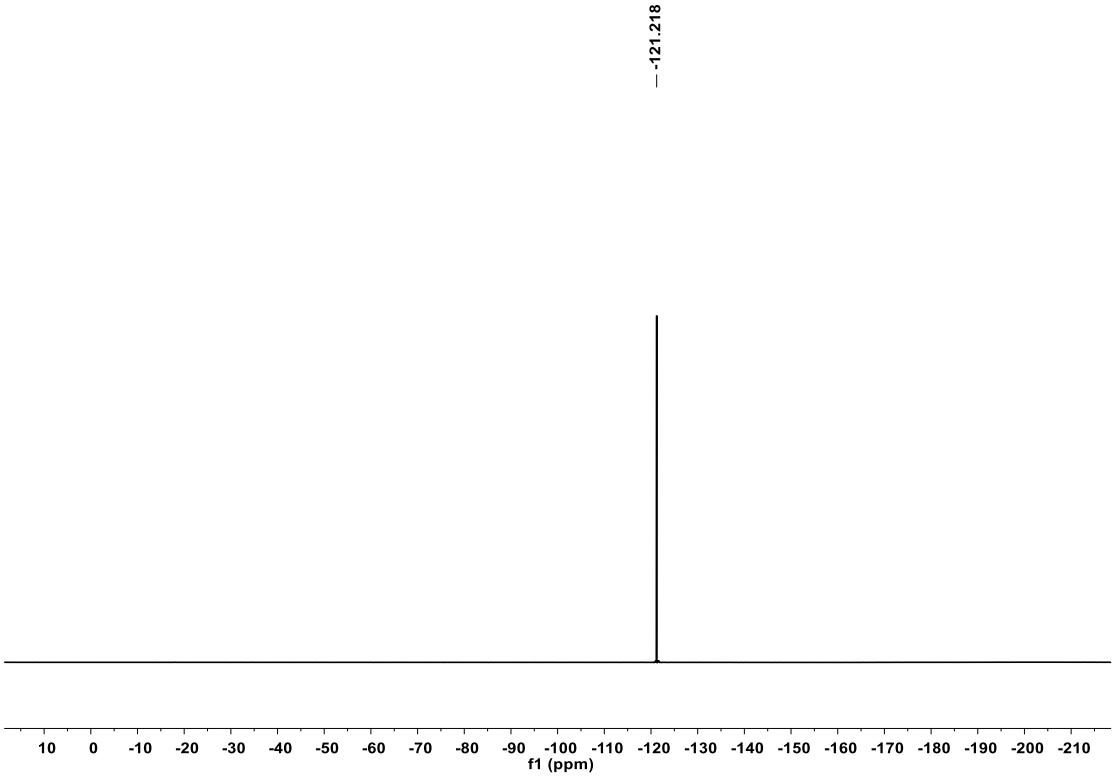
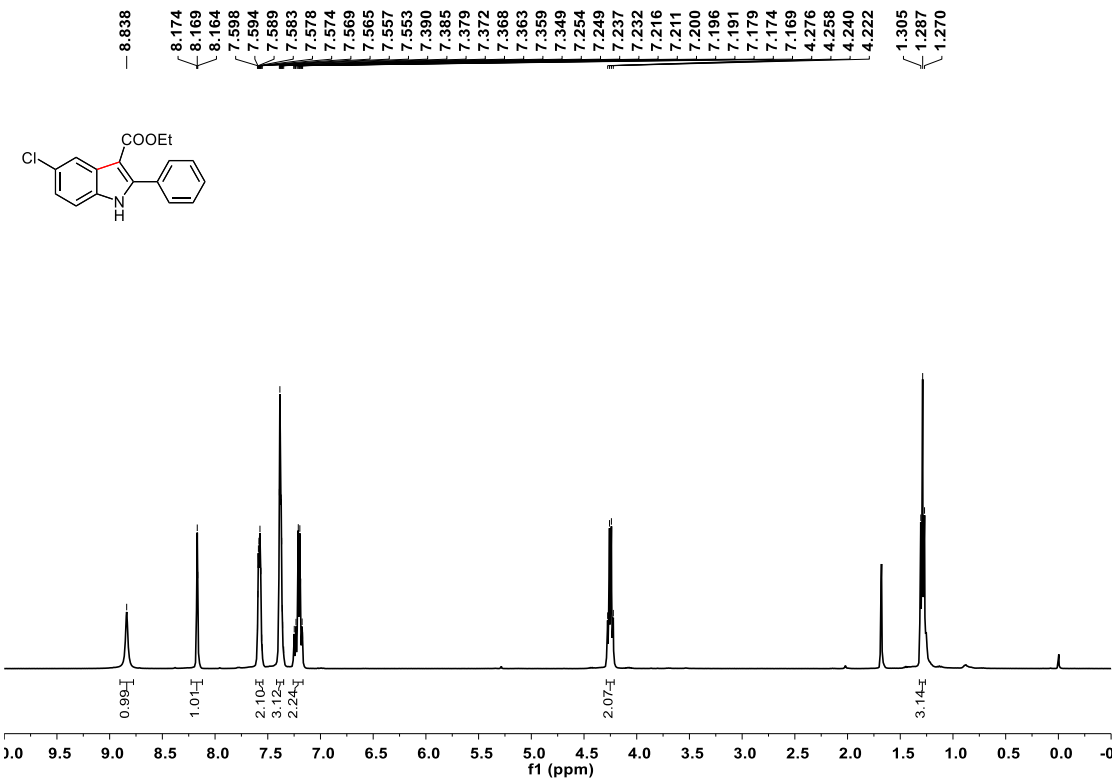
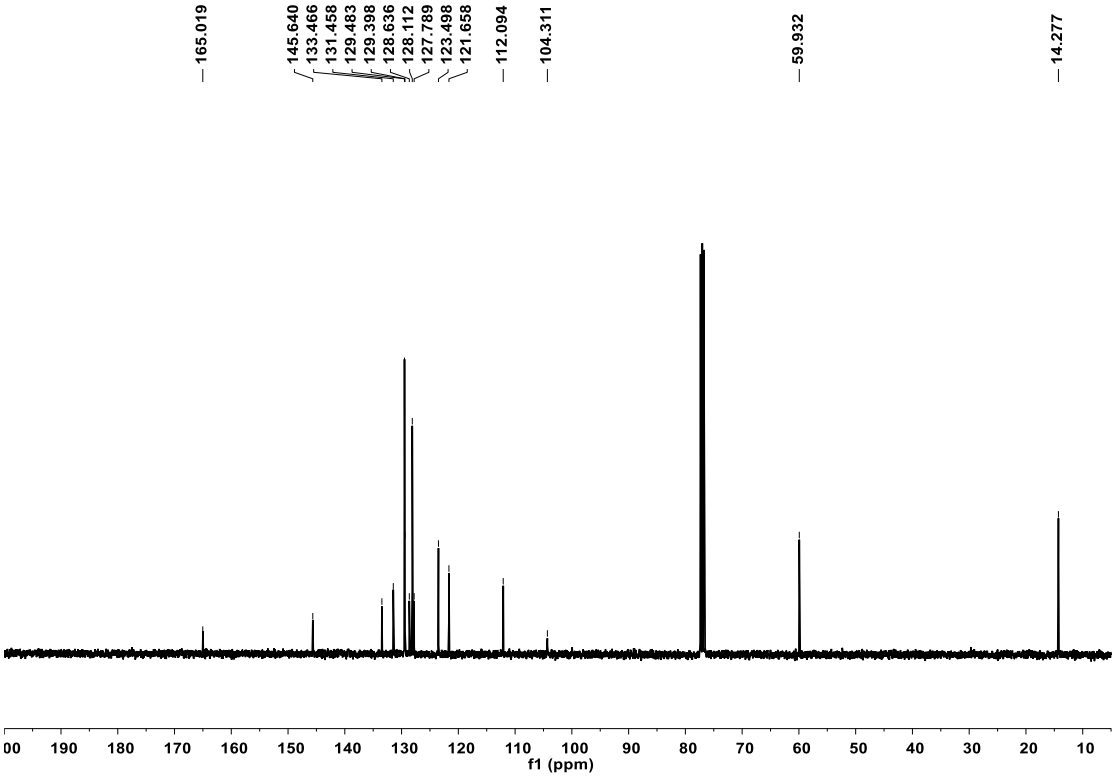

-121.218
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

2f

^{1}H NMR

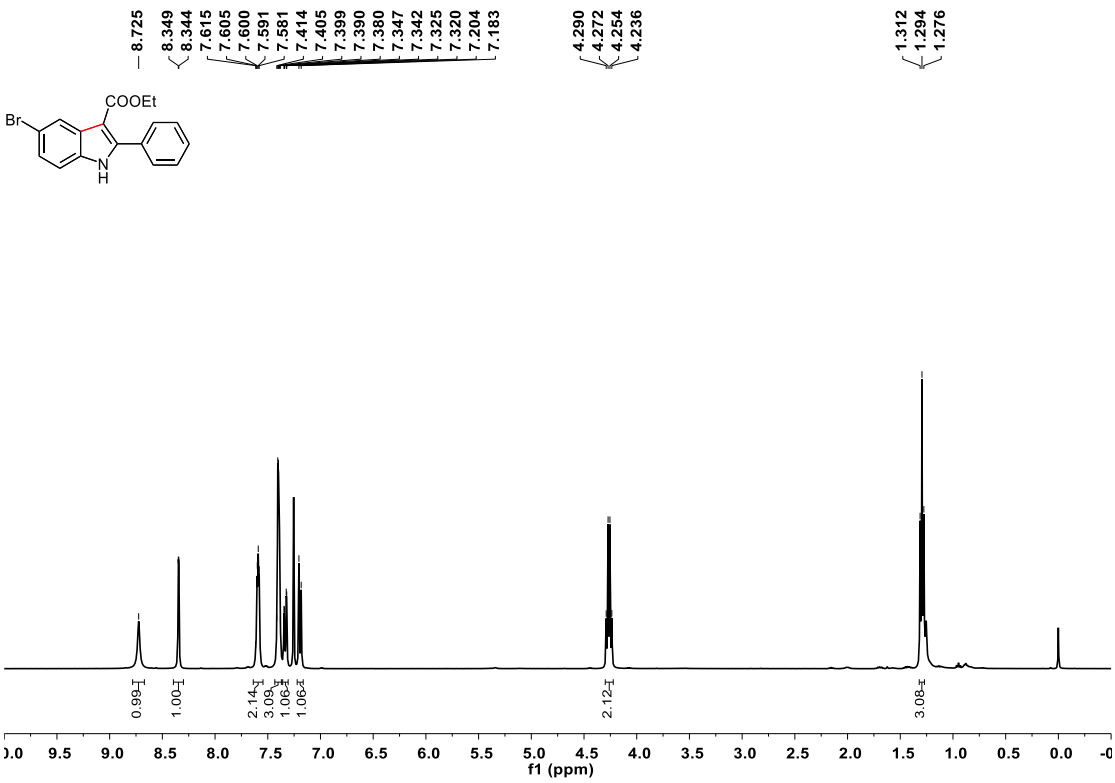


^{13}C NMR



2g

^{1}H NMR



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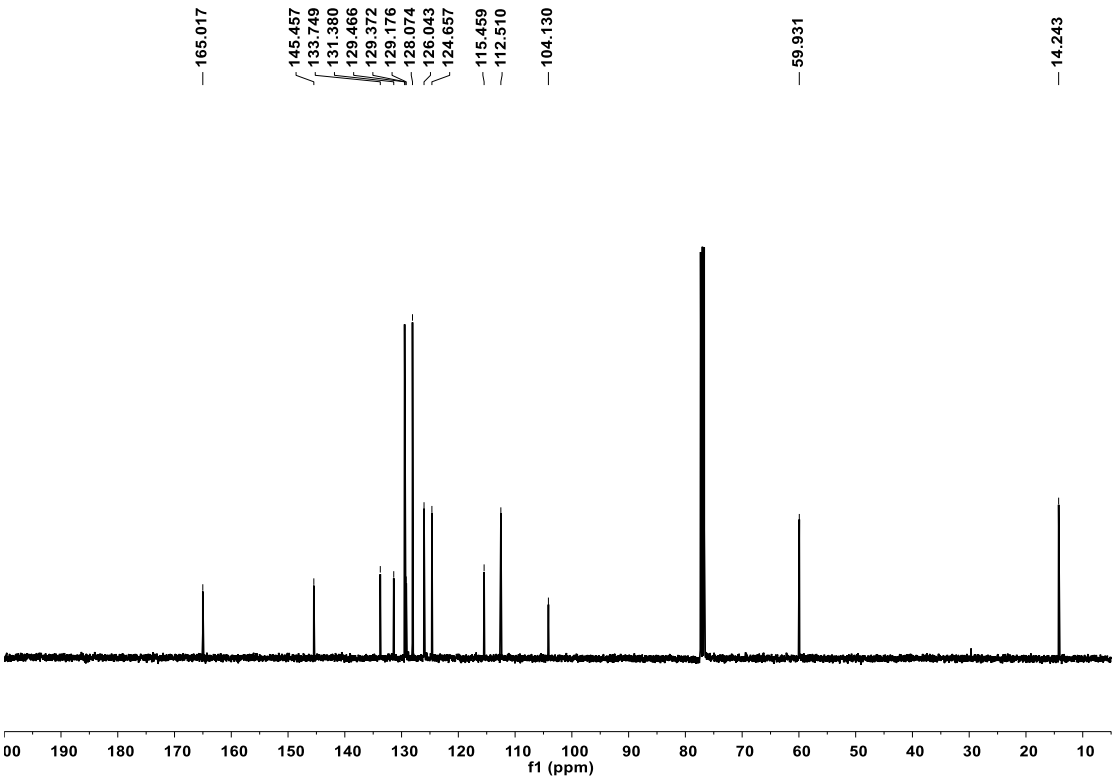

2h

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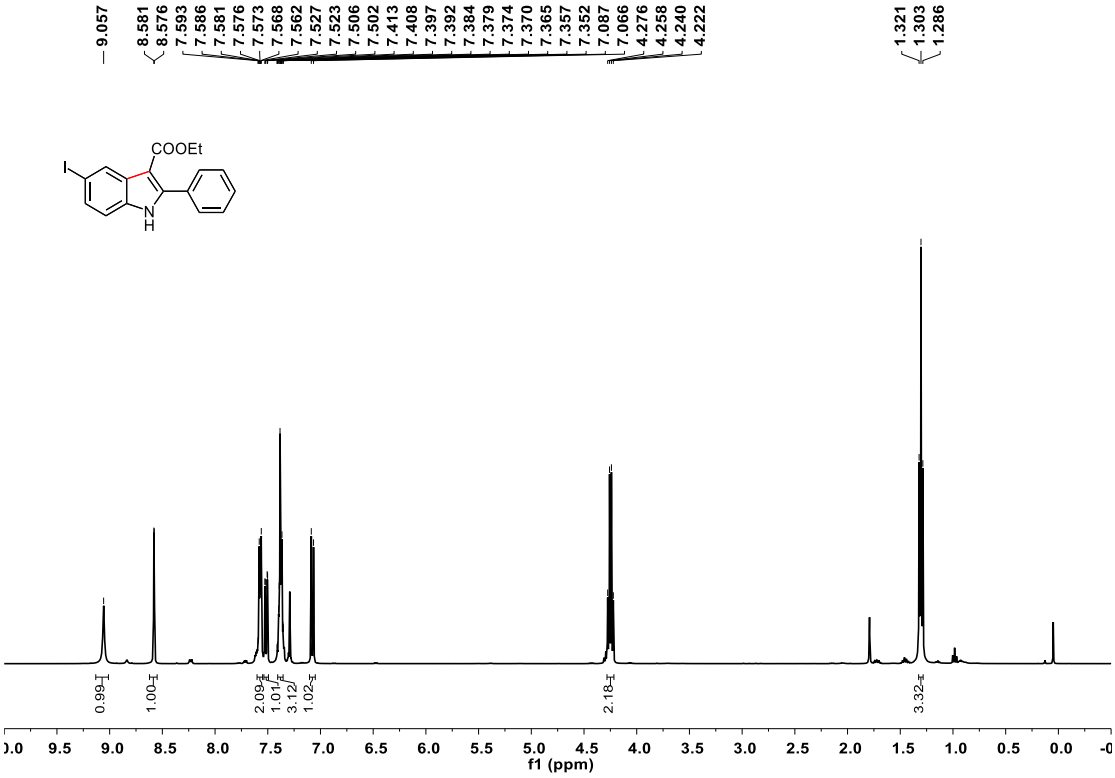

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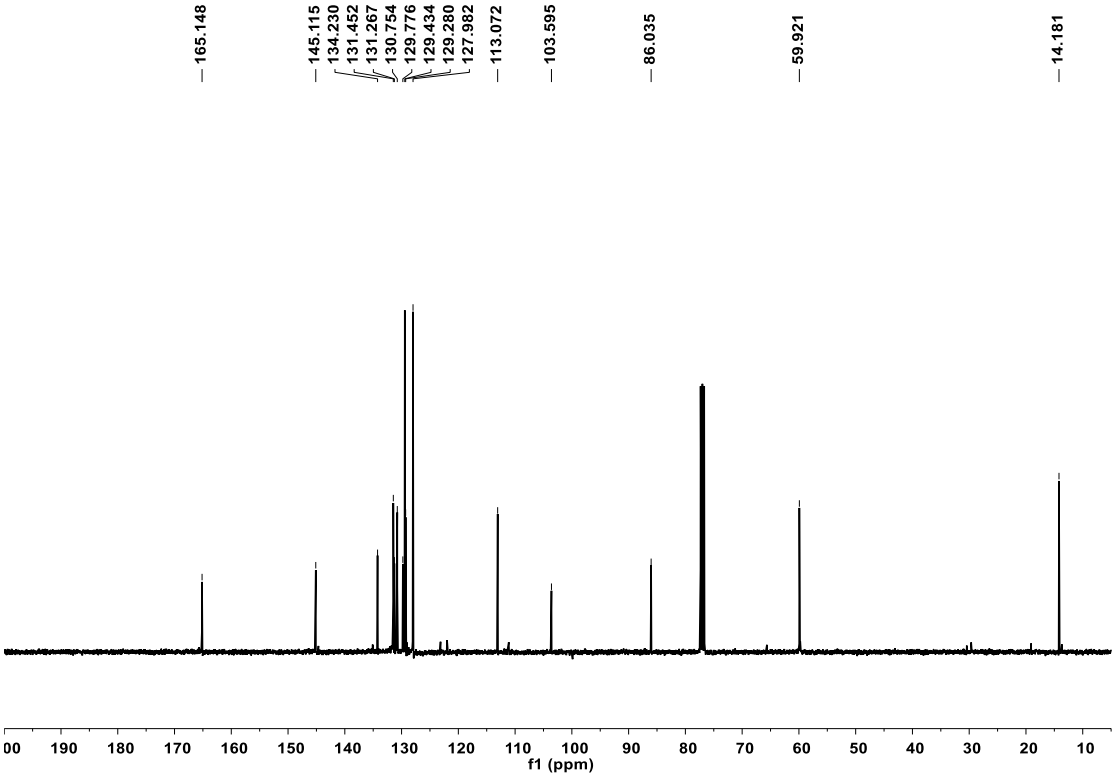

2i

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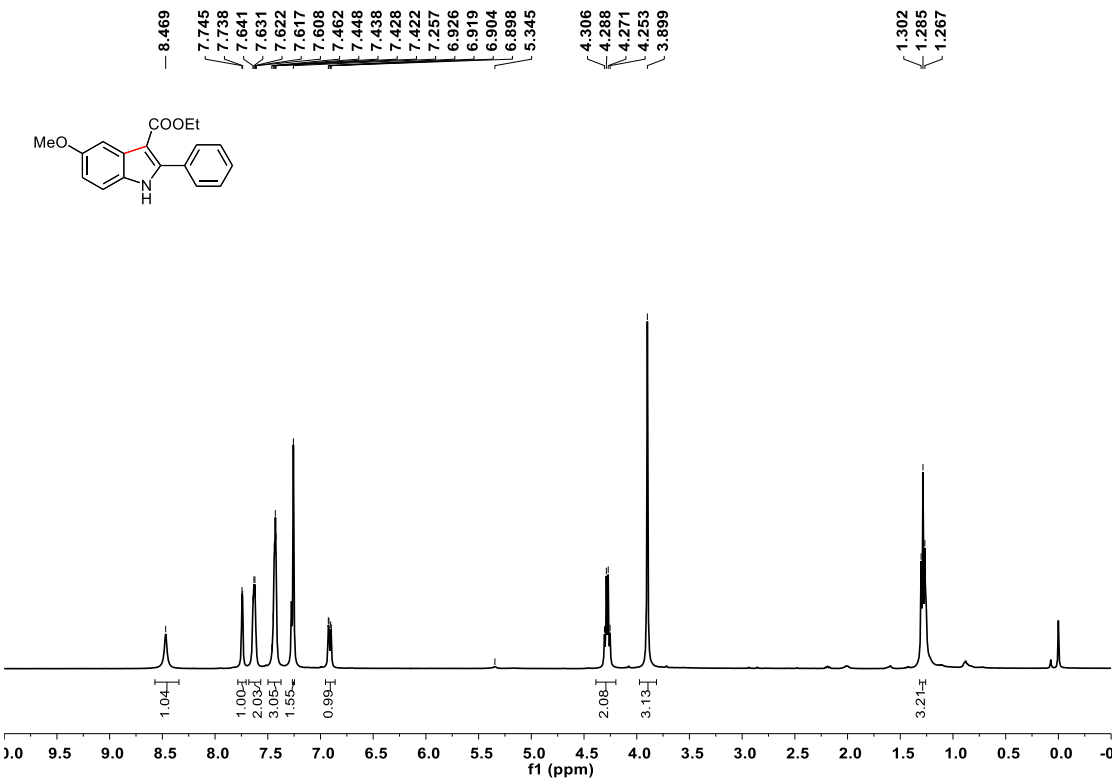

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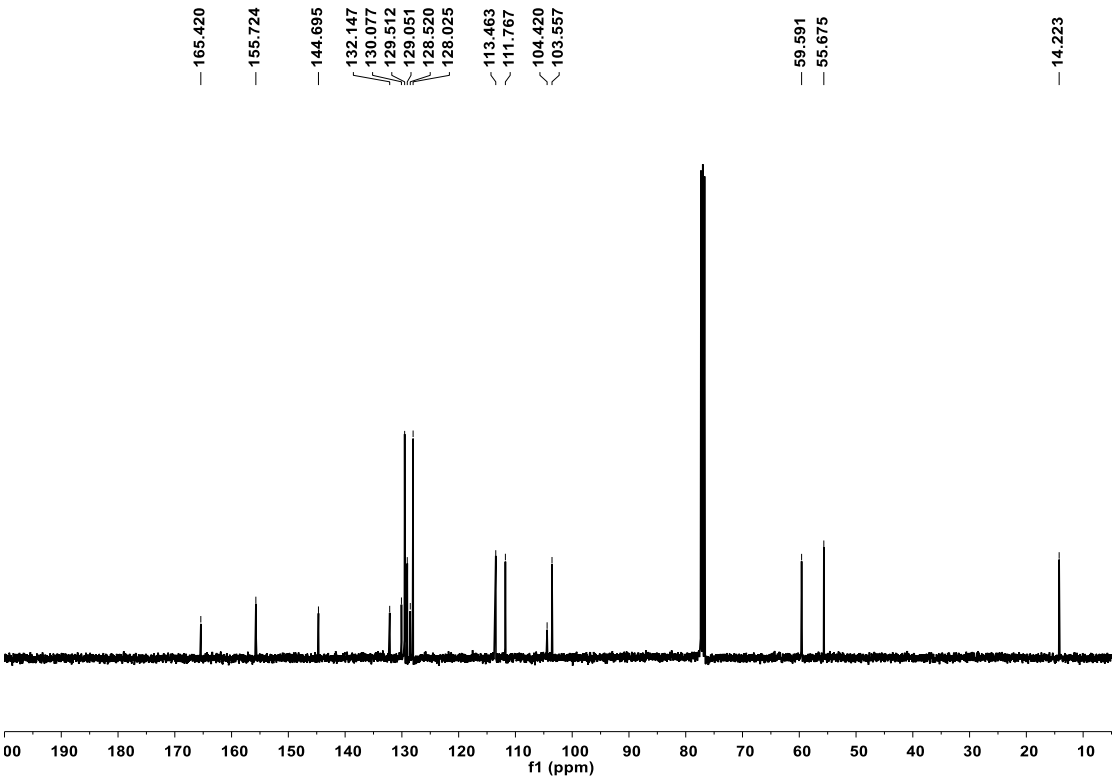

2j

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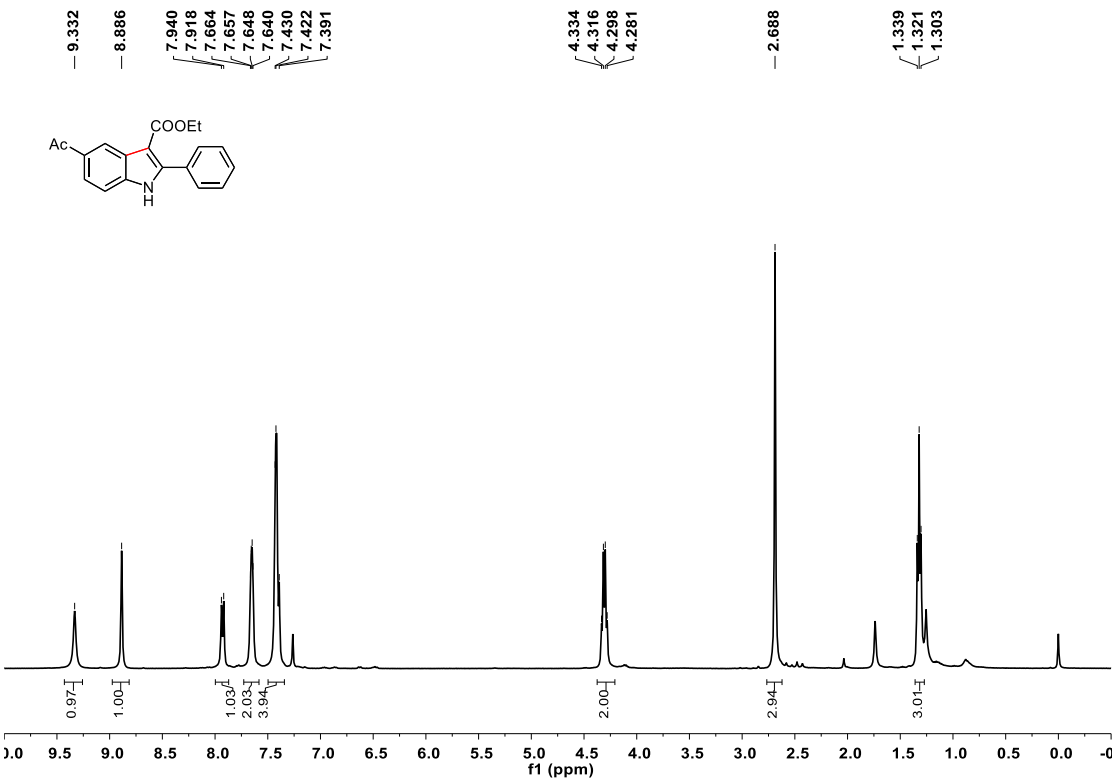

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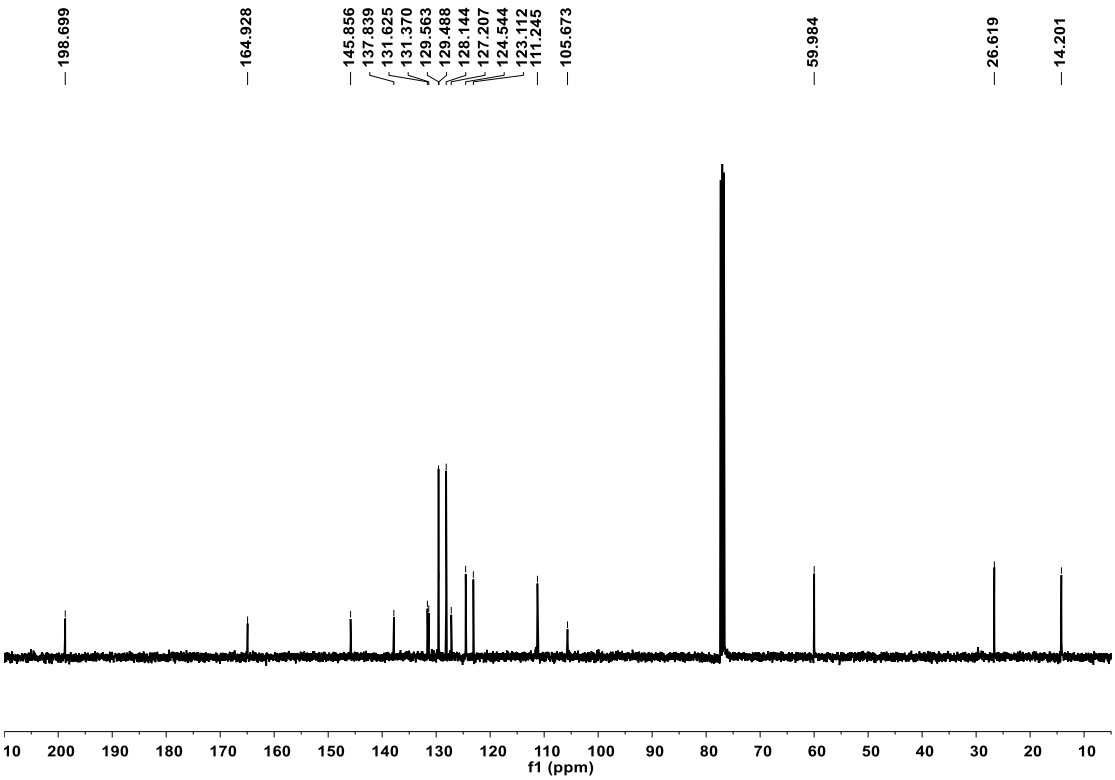

2k

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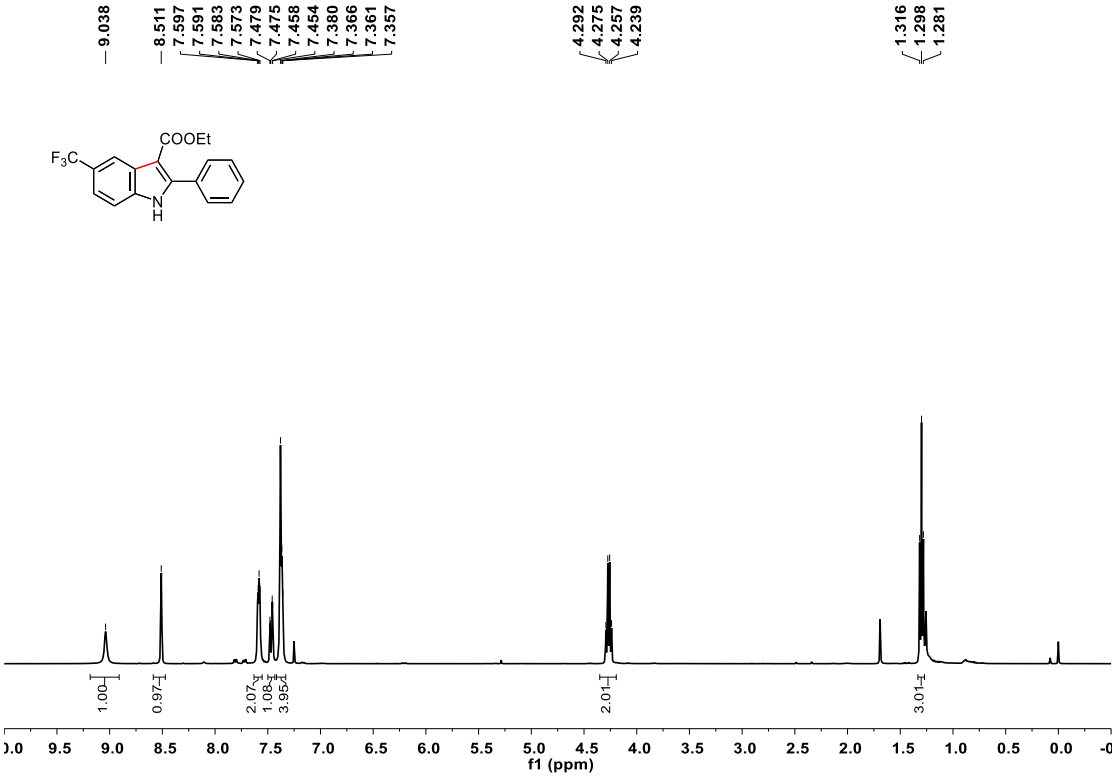

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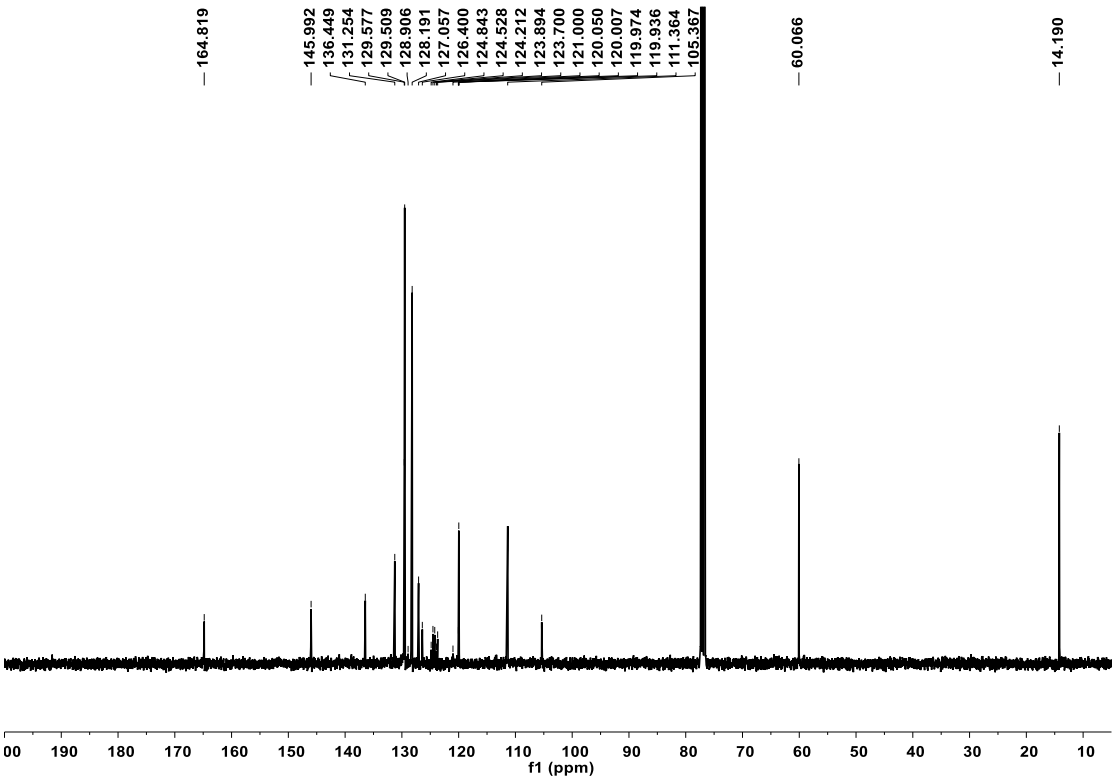
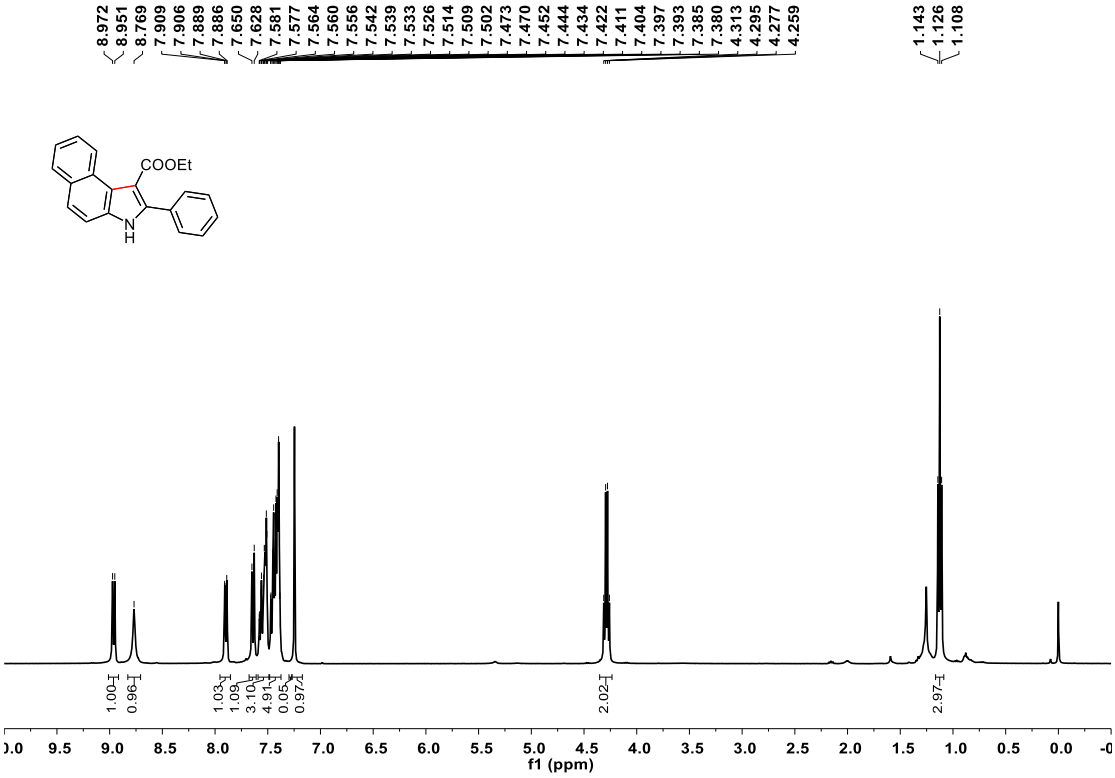
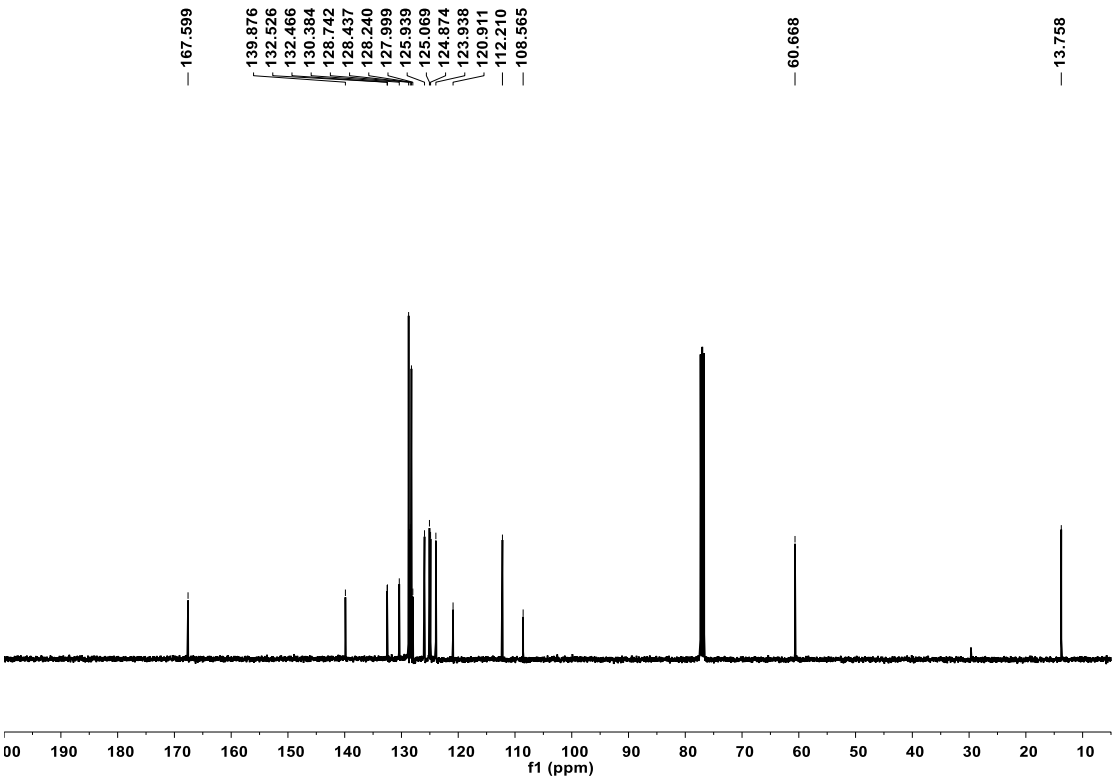


2l

^{1}H NMR



^{13}C NMR



2m

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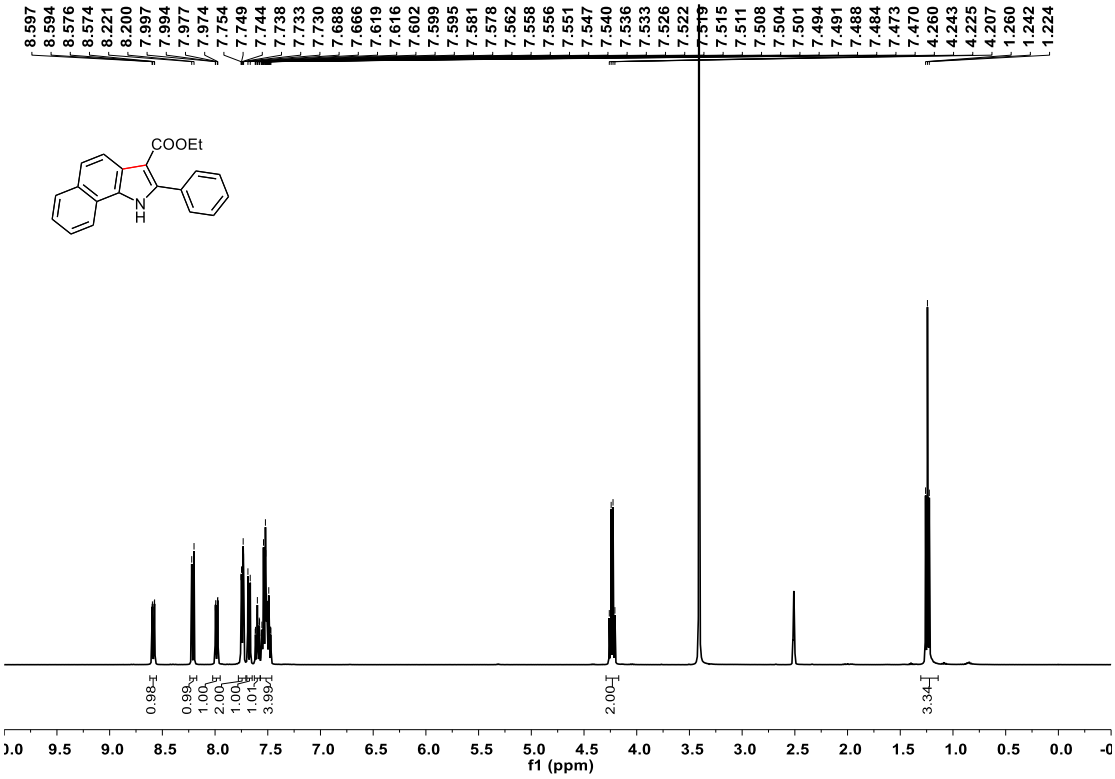
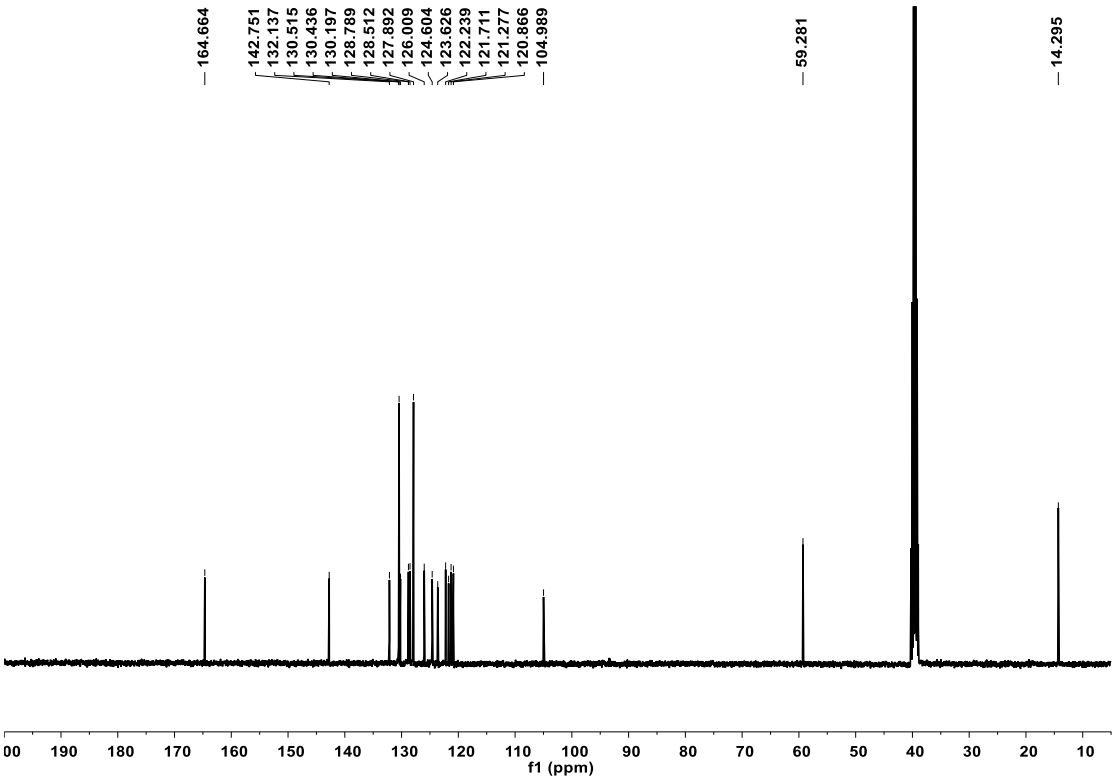
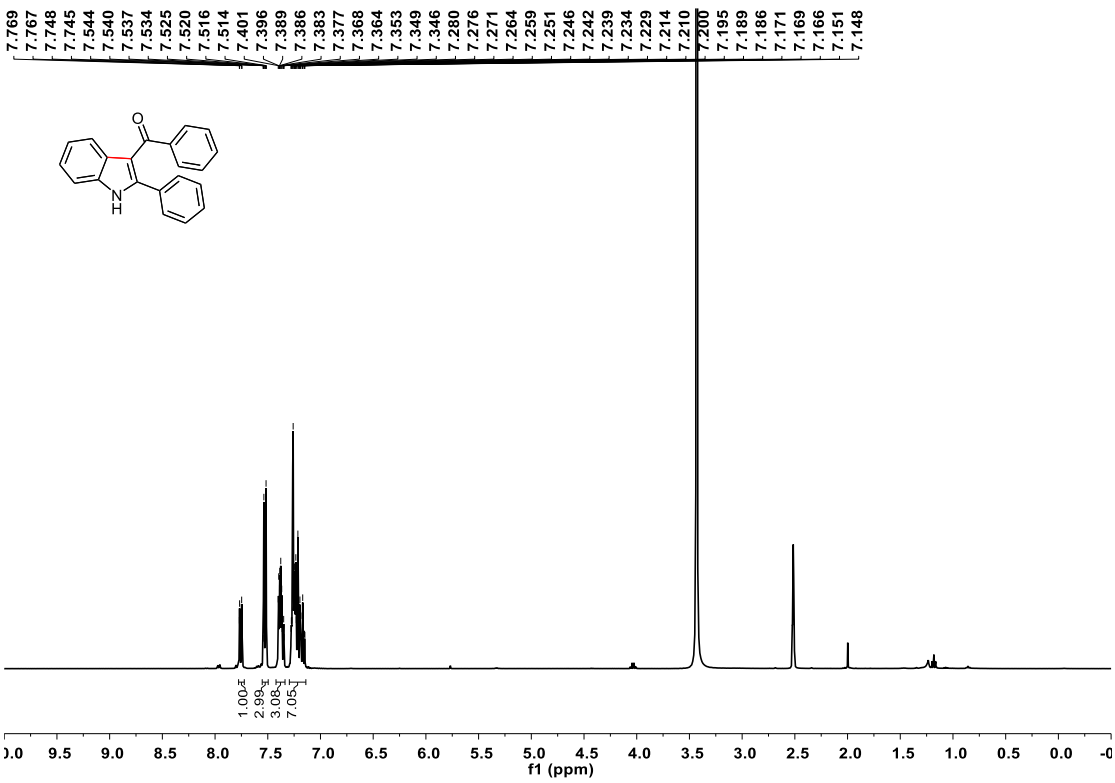


^{13}C NMR

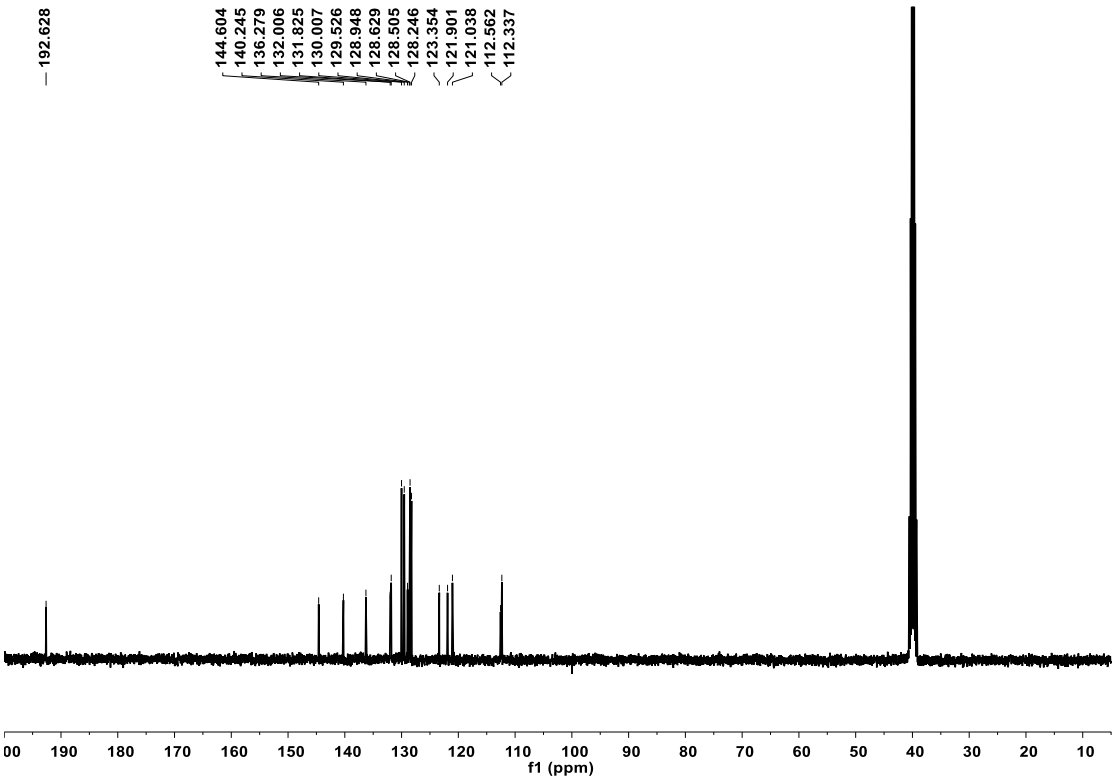


2n

^{1}H NMR

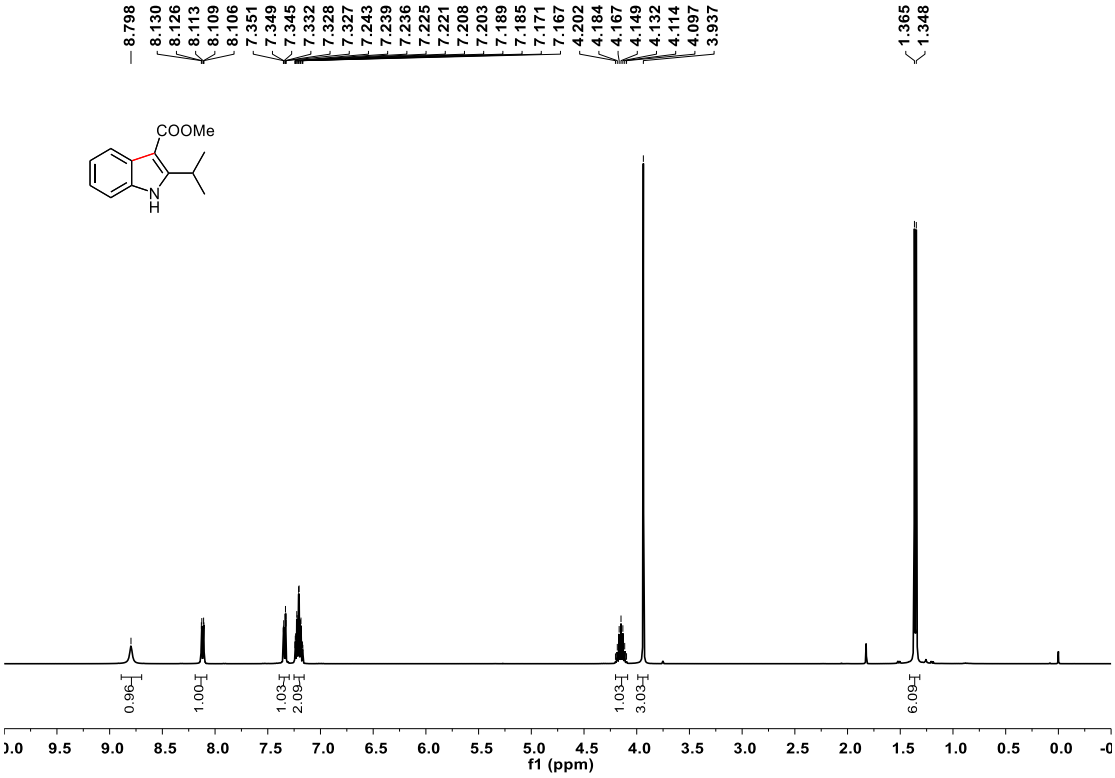


^{13}C NMR

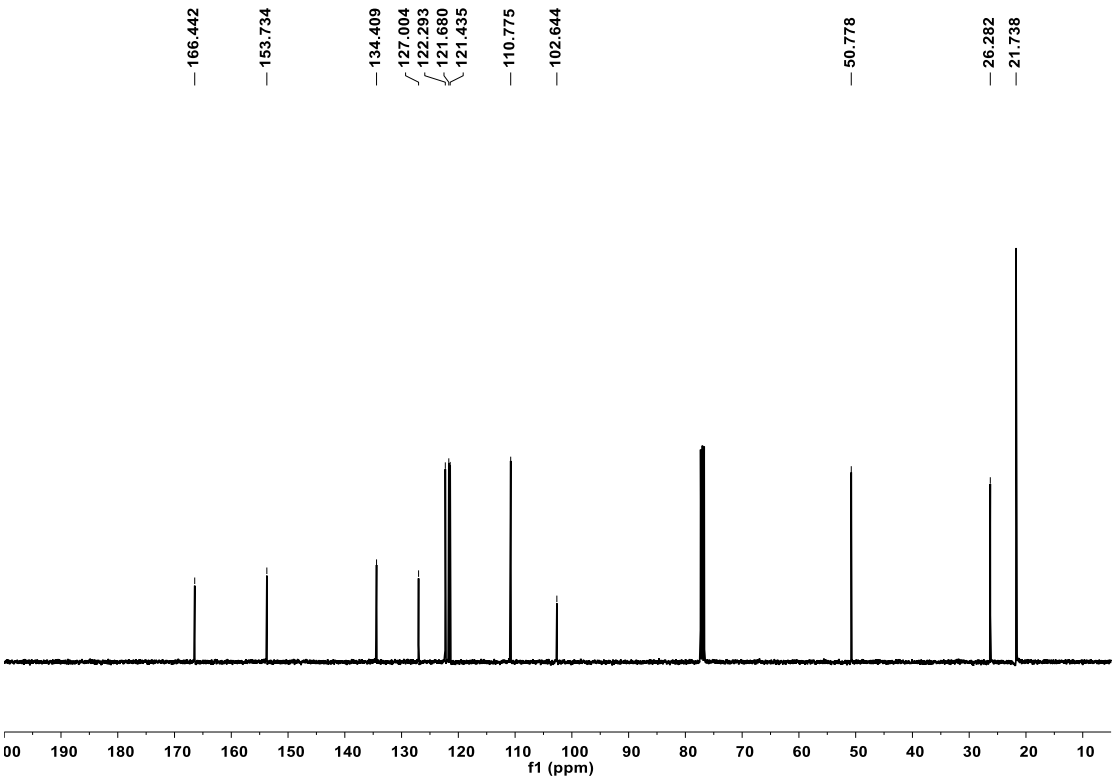


2o

^{1}H NMR

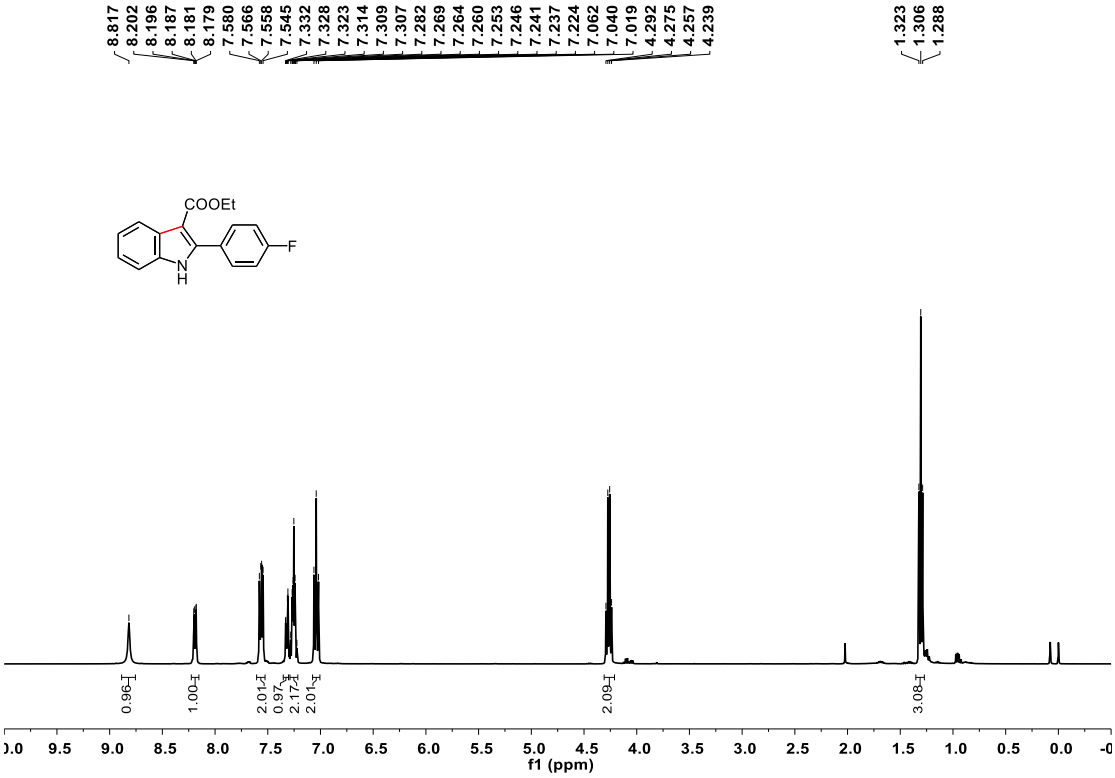


^{13}C NMR

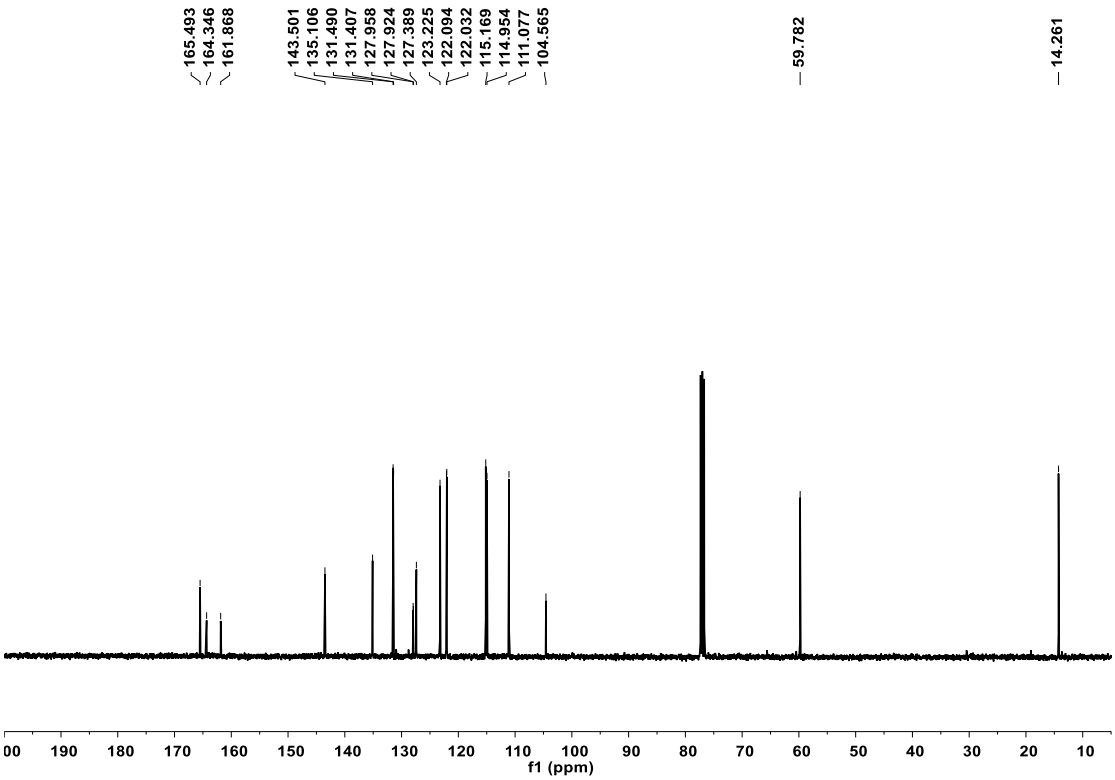


2p

^{1}H NMR



^{13}C NMR



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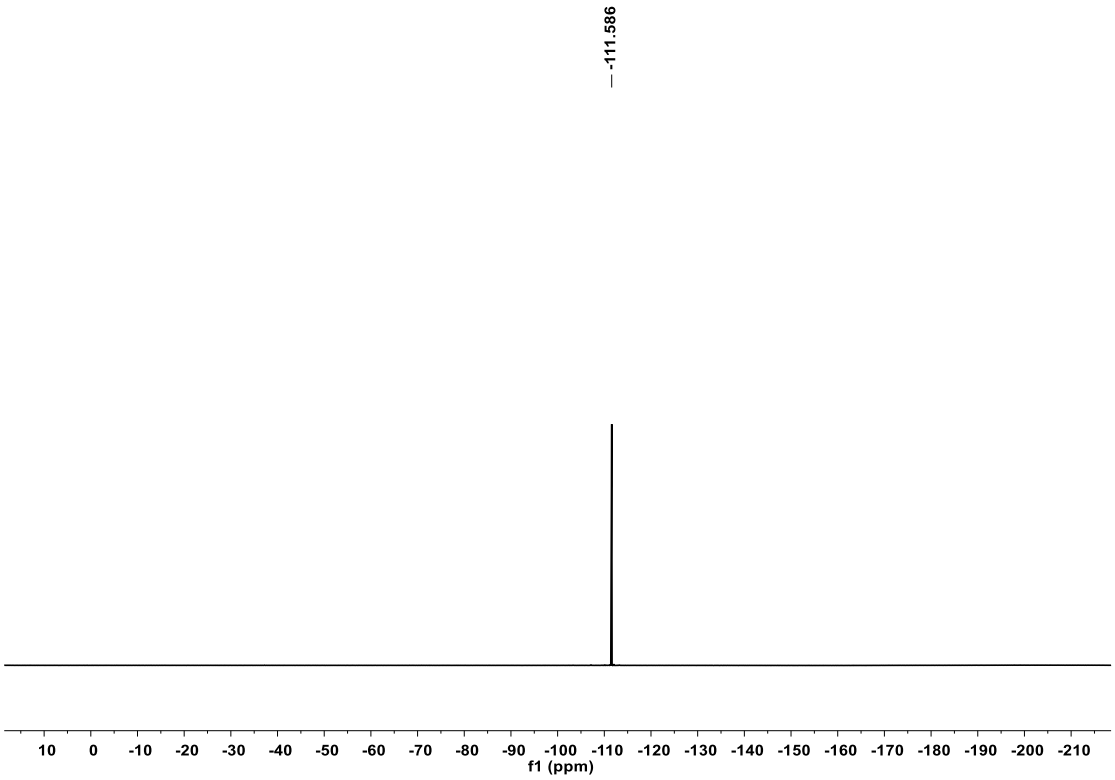
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10
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-70
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-90
-100
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-120
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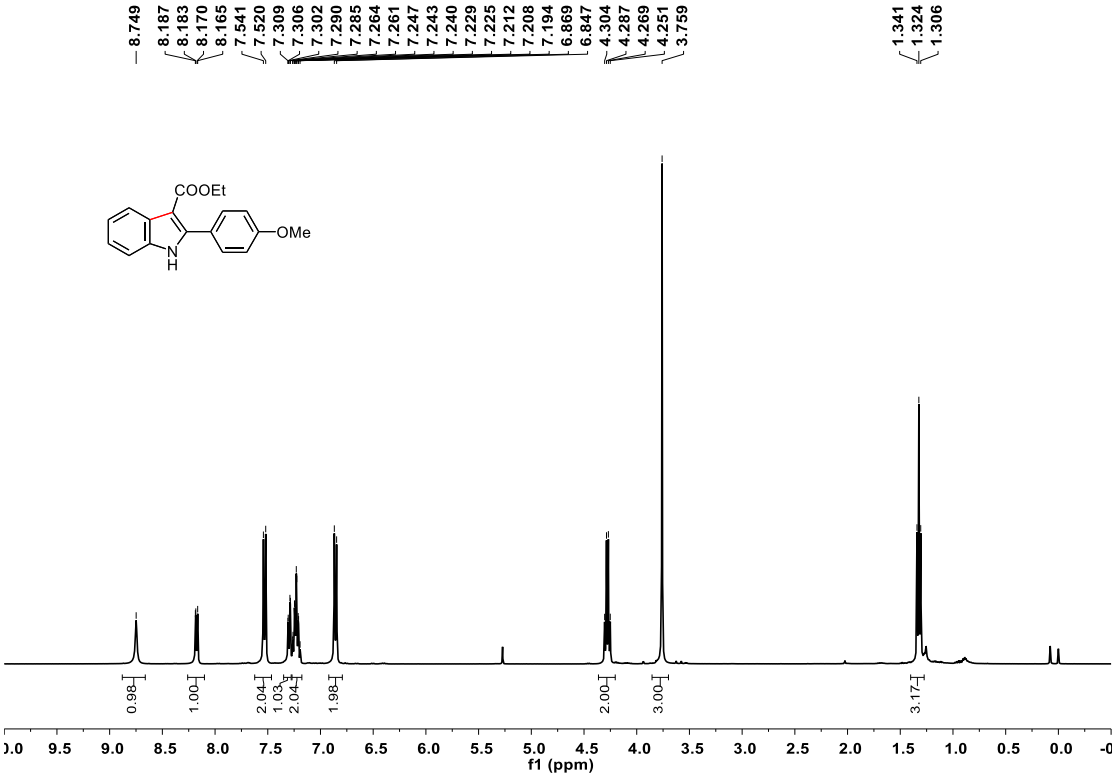
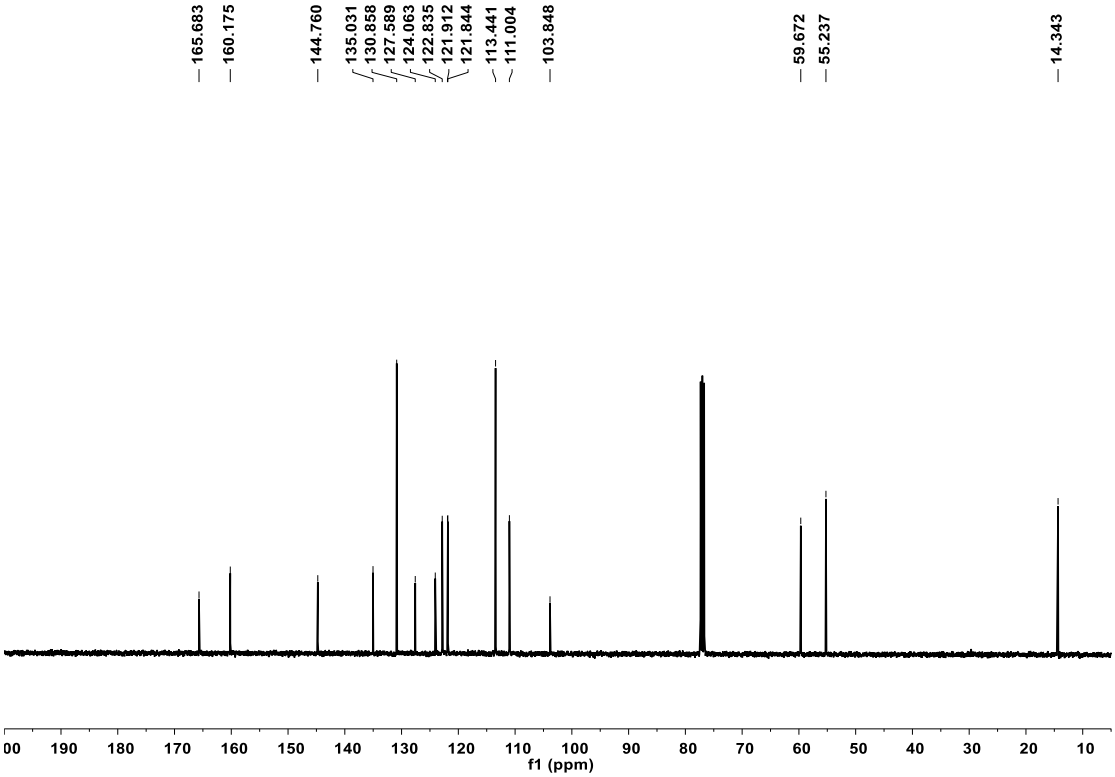
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

2q

^{1}H NMR



^{13}C NMR



2r

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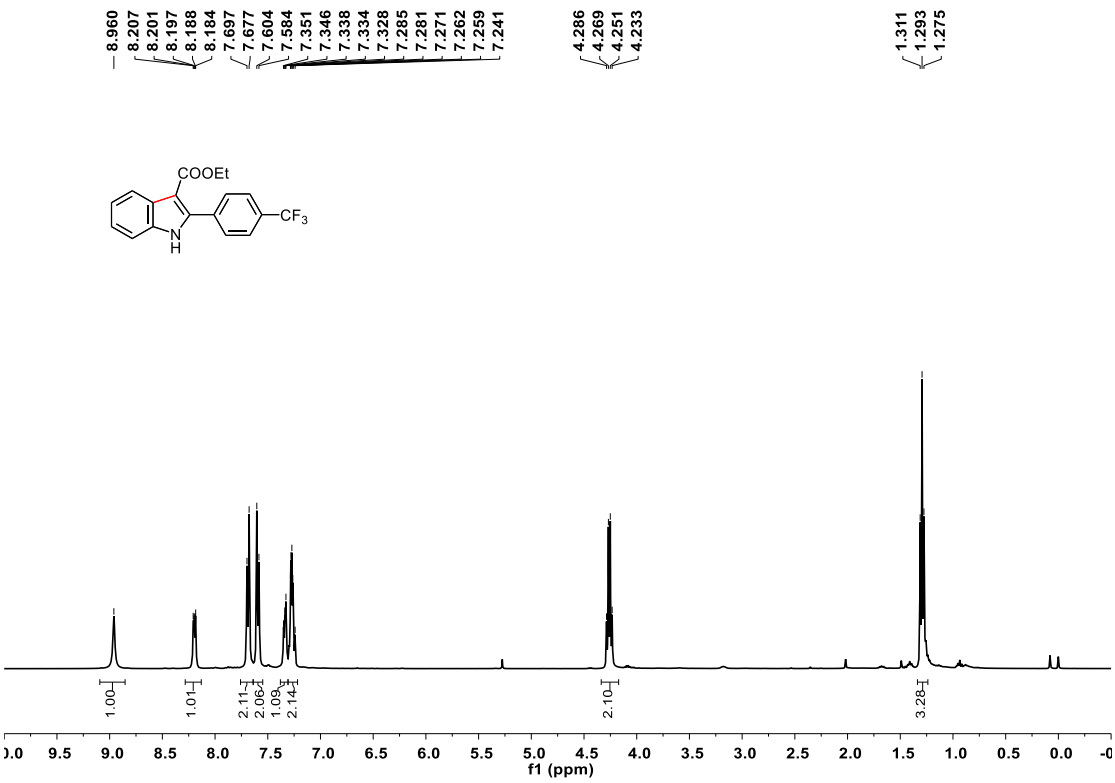
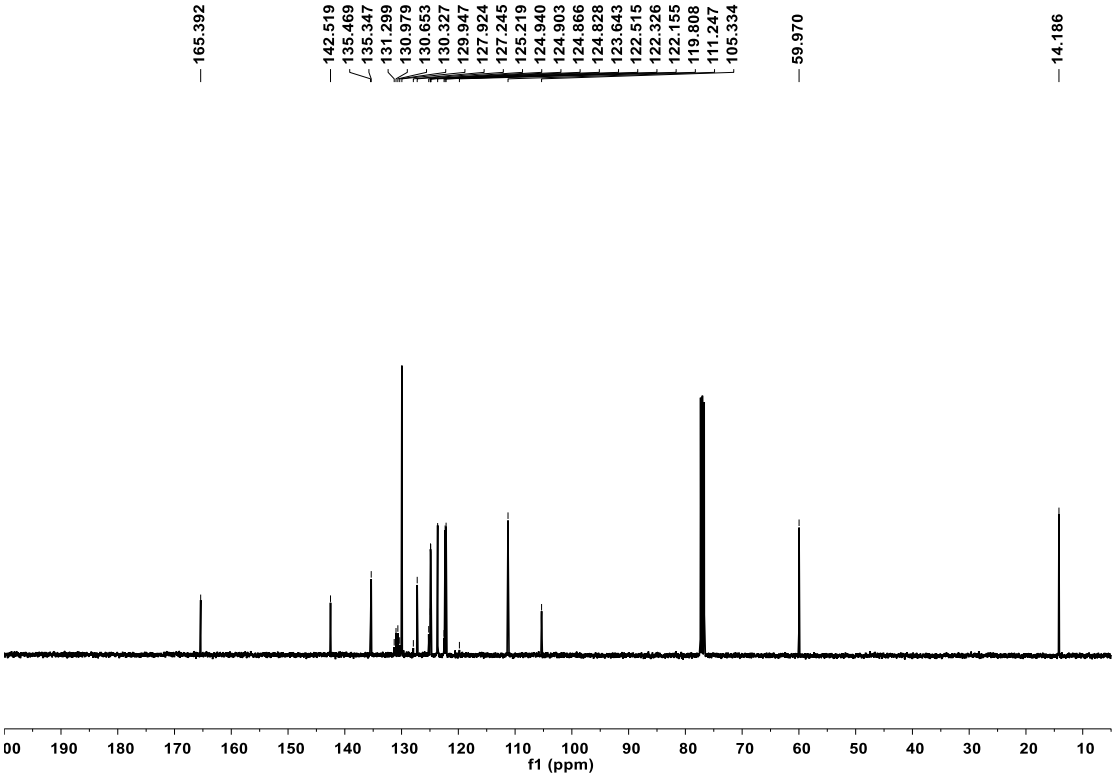
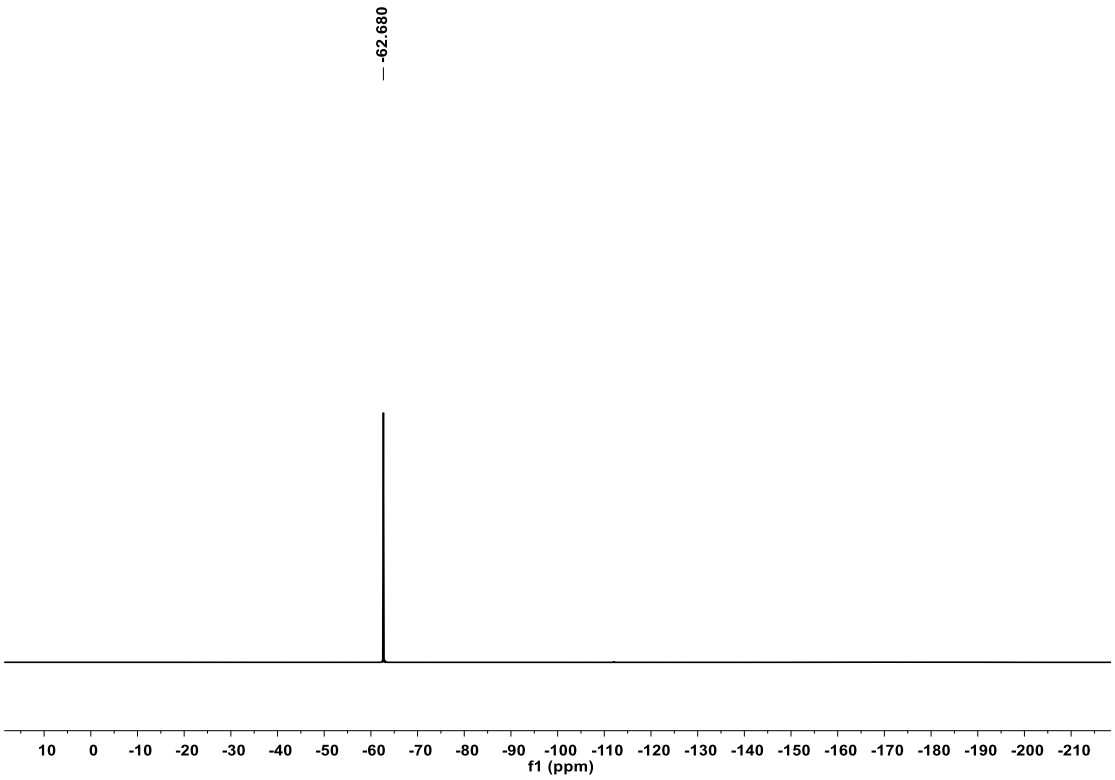


^{13}C NMR

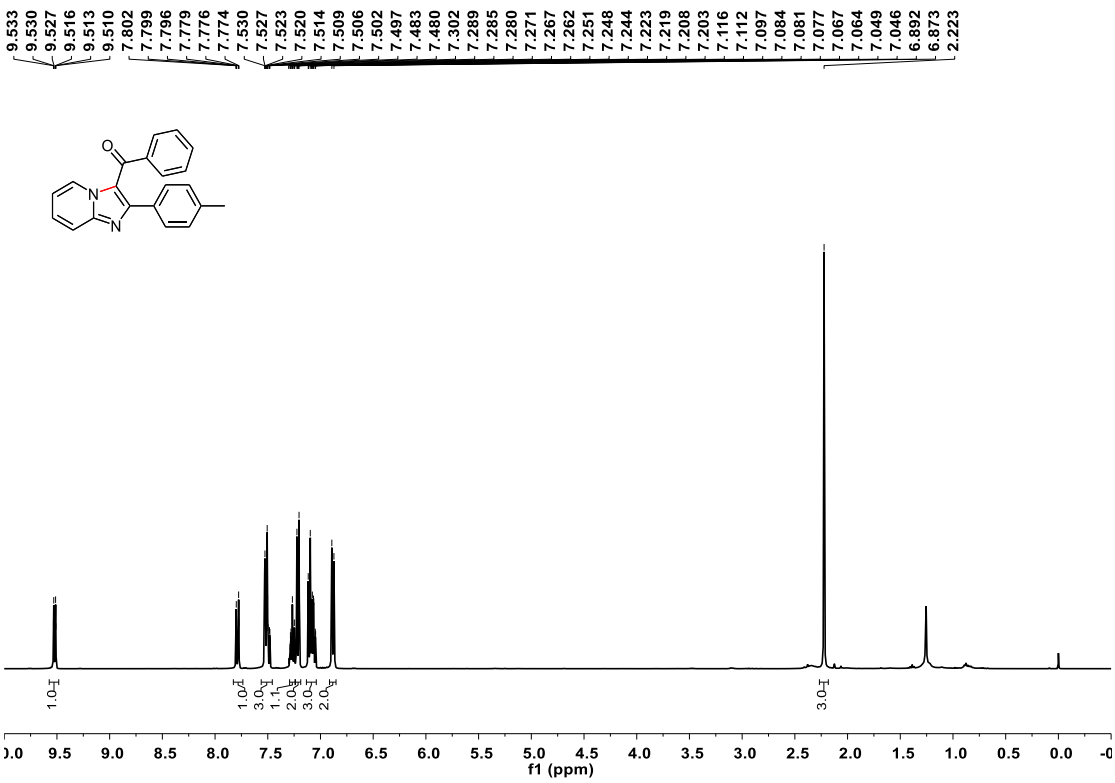


^{19}F NMR

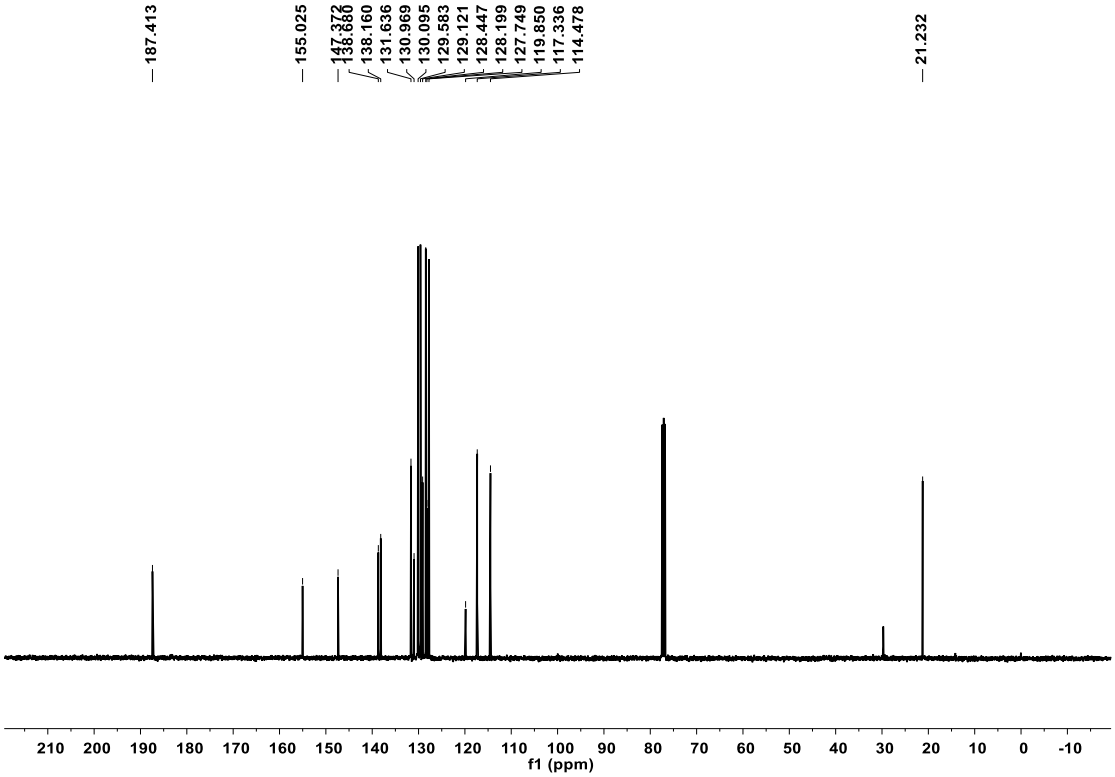

-62.680
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3a

^{1}H NMR

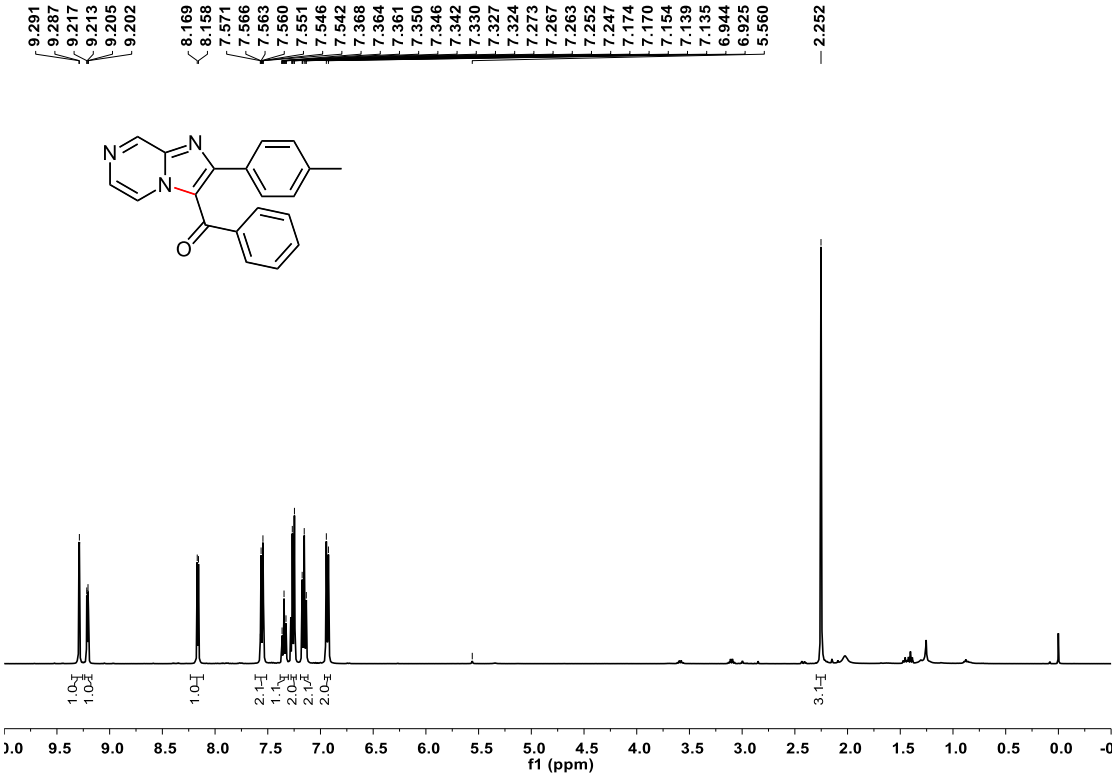


^{13}C NMR



3b

^{1}H NMR



^{13}C NMR