

# DABCO-Catalyzed Mono-/Diallylation of N-Unsubstituted Isatin N,N'-Cyclic Azomethine Imine 1,3-Dipoles with Morita-Baylis-Hillman Carbonates

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## 1. The phenomenon of the reaction and TLC.

The phenomenon of the reaction for **1a** and **2a** under DABCO in DCM at rt. (a) no DABCO (0 min, a yellow cloudy solution); (b) reaction finished (5 min, a yellow clear solution) (Figure 1). TLC of starting materials (**1a** and **2a**) and products (**3a** and **4a**) showed the result (Figure 2).

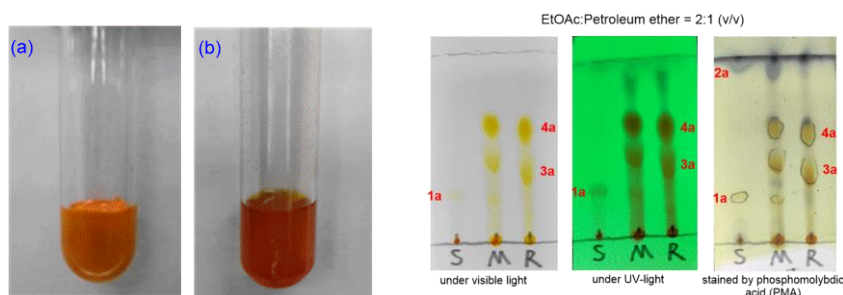
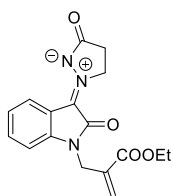
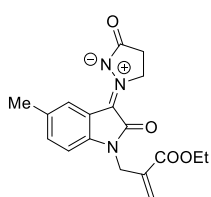


Fig. 1 (left) and Fig. 2 (right)

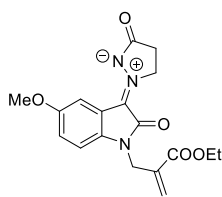
## 2. Data for all new compounds.



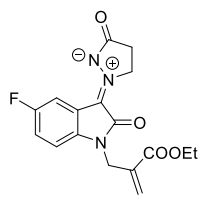
[Reaction time: 5 min]; **3a**: 297 mg, 91%, a yellow solid, m.p. 192.7-193.2 °C; IR (thin film):  $\nu_{\max}$  3684, 3987, 1710, 1694, 1573, 1383, 1267, 1128, 761  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.42 (d,  $J$  = 7.6 Hz, 1 H), 7.38 (td,  $J$  = 7.8, 0.8 Hz, 1 H), 7.15 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.85 (d,  $J$  = 8.0 Hz, 1 H), 6.34 (s, 1 H), 5.59 (s, 1 H), 5.00 (t,  $J$  = 7.6 Hz, 2 H), 4.64 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.4, 160.9, 141.6, 133.8, 132.6, 127.8, 126.5, 124.5, 123.5, 116.9, 109.2, 61.4, 57.4, 40.6, 28.4, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  350.1117, found 350.1116.



[Reaction time: 5 min]; **3b**: 266 mg, 78%, a red solid, m.p. 184.2-186.0 °C; IR (thin film):  $\nu_{\max}$  3671, 2996, 1720, 1699, 1578, 1368, 1149, 1132, 734  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.28 (s, 1 H), 7.19 (d,  $J$  = 8.0 Hz, 1 H), 7.74 (d,  $J$  = 8.0 Hz, 1 H), 6.33 (s, 1 H), 5.57 (s, 1 H), 5.00 ( $\psi$ t,  $J$  = 7.4 Hz, 2 H), 4.62 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 2.89 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H), 2.36 (s, 3 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.8, 165.4, 160.8, 139.5, 133.8, 133.4, 133.2, 128.2, 126.4, 124.4, 116.8, 108.9, 61.3, 57.4, 40.5, 28.5, 21.0, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{18}\text{H}_{20}\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  342.1454, found 342.1428.

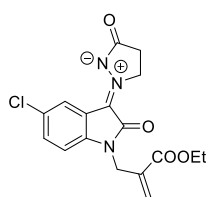


[Reaction time: 3 min]; **3c**: 303 mg, 86%, a red solid, m.p. 172.2-174.7 °C; IR (thin film):  $\nu_{\max}$  3684, 2986, 1725, 1701, 1570, 1367, 1264, 1014, 762  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.05 (d,  $J$  = 2.8 Hz, 1 H), 6.95 (dd,  $J$  = 8.8, 2.8 Hz, 1 H), 6.77 (d,  $J$  = 7.2 Hz, 1 H), 6.34 (s, 1 H), 5.59 (s, 1 H), 5.02 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H), 4.62 (s, 2 H), 4.28 (q,  $J$  = 7.2 Hz, 2 H), 3.84 (s, 3 H), 2.91-2.87 (m, 2 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.4, 160.9, 156.3, 135.5, 133.9, 126.5, 125.0, 119.4, 117.4, 112.3, 110.0, 61.3, 57.5, 56.2, 40.6, 28.4, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{18}\text{H}_{20}\text{N}_3\text{O}_5$   $[\text{M}+\text{H}]^+$  358.1403, found 358.1377.



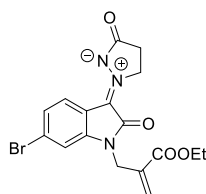
[Reaction time: 8 min]; **3d**: 169 mg, 49%, a yellow solid, m.p. 135.9-138.2 °C; IR (thin film):  $\nu_{\max}$  3671, 2984, 1705, 1615, 1578, 1262, 1115, 659  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.17 (dd,  $J$  = 8.2, 2.6 Hz, 1 H), 7.08 (m, 1 H), 6.82 (dd,  $J$  = 8.4, 4.0 Hz, 1 H), 6.36 (s, 1 H), 5.62 (s, 1 H), 5.03 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H), 4.64 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 2.90 (t,  $J$  = 7.4 Hz, 2 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.3, 160.8, 157.9, 137.5 (d), 133.8, 126.9, 123.7, 119.9 (d), 117.7 (d), 114.8 (d),

110.0 (d), 61.4, 57.7, 40.8, 28.3, 14.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  -118.7; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{FN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  346.1203, found 346.1131.



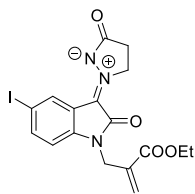
[Reaction time: 14 min]; **3e**: 173 mg, 50%, a yellow solid, m.p. 164.1-165.9 °C; IR (thin film):  $\nu_{\text{max}}$  3410, 3101, 2989, 1710, 1643, 1592, 1265, 1148, 776, 694  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.39 (d,  $J$  = 2.0 Hz, 1 H), 7.32 (dd,  $J$  = 8.4, 2.0 Hz, 1 H), 6.81 (d,  $J$  = 8.4 Hz, 1 H), 6.36 (s, 1 H), 5.62 (s, 1 H), 5.03 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H), 4.64 (s, 2 H), 4.26 (q,  $J$  = 7.2 Hz, 2 H), 2.90 (t,  $J$  = 7.4 Hz, 2 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.6, 165.3, 160.8, 160.2, 137.5, 133.8, 126.9, 123.7,

119.9, 117.7, 114.8, 110.0, 61.4, 57.7, 40.8, 28.3, 14.2;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.3, 160.6, 139.8, 133.7, 132.0, 129.0, 127.1, 127.0, 123.1, 118.0, 110.2, 61.4, 57.8, 40.8, 28.3, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{ClN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  362.0908, found 362.0905.



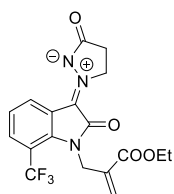
[Reaction time: 12 min]; **3f**: 329 mg, 81%, a yellow solid, m.p. 166.7-168.3 °C; IR (thin film):  $\nu_{\text{max}}$  3406, 2930, 1709, 1644, 1599, 1374, 1153, 1025, 783, 511  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.29 (d,  $J$  = 8.0 Hz, 1 H), 7.30 (dd,  $J$  = 8.0, 1.6 Hz, 1 H), 7.04 (d,  $J$  = 1.2 Hz, 1 H), 6.38 (s, 1 H), 5.60 (s, 1 H), 4.98 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H), 4.64 (s, 2 H), 4.28 (q,  $J$  = 7.2 Hz, 2 H), 2.89 (t,  $J$  = 7.4 Hz, 2 H), 1.34 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$

NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.6, 165.2, 160.7, 142.3, 133.5, 128.4, 127.0, 126.7, 126.5, 123.3, 115.8, 112.7, 61.5, 57.7, 40.8, 28.3, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  406.0402, found 406.0427.



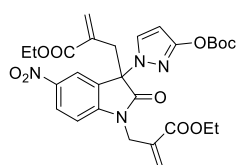
[Reaction time: 12 min]; **3g**: 353 mg, 78%, a yellow solid, m.p. 204.1-206.6 °C; IR (thin film):  $\nu_{\text{max}}$  3425, 1705, 1694, 1600, 1384, 1163, 1127, 778, 518  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.74 (s, 1 H), 7.70 (d,  $J$  = 8.0 Hz, 1 H), 6.68 (d,  $J$  = 8.4 Hz, 1 H), 6.35 (s, 1 H), 5.60 (s, 1 H), 5.03 ( $\psi$ t,  $J$  = 2.8 Hz, 2 H), 4.64 (s, 2 H), 4.26 (q,  $J$  = 6.8 Hz, 2 H), 2.90 (t,  $J$  = 6.4 Hz, 2 H), 1.34 (t,  $J$  = 6.8 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):

$\delta$  186.6, 165.3, 160.4, 140.9, 140.8, 135.3, 133.7, 126.9, 122.8, 118.6, 111.2, 86.1, 61.4, 57.8, 40.7, 28.3, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{IN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  454.0264, found 454.0259.



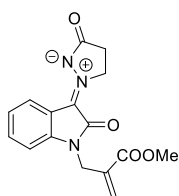
[Reaction time: 20 min]; **3h**: 229 mg, 58%, a yellow solid, m.p. 127.8-128.4 °C; IR (thin film):  $\nu_{\text{max}}$  3355, 2984, 1716, 1599, 1369, 1330, 1124, 750  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.17 (dd,  $J$  = 8.2, 2.6 Hz, 1 H), 7.08 (m, 1 H), 6.82 (dd,  $J$  = 8.4, 4.0 Hz, 1 H), 6.36 (s, 1 H), 5.62 (s, 1 H), 5.03 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H), 4.64 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 2.90 (t,  $J$  = 7.4 Hz, 2 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  -55.7;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.1, 161.6, 138.8, 134.6, 130.9, 129.8

(q), 124.3, 123.0, 122.9, 121.6, 119.4, 112.9 (q), 61.2, 58.5, 42.8 (d), 28.0, 14.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  -55.7; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{18}\text{H}_{17}\text{F}_3\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  396.1171, found 396.1164.



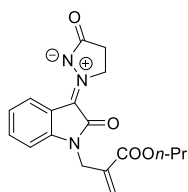
[Reaction time: 1 min]; **3'i**: 275 mg, 47%, a white solid, m.p. 159.0-161.7 °C; IR (thin film):  $\nu_{\text{max}}$  3164, 1772, 1751, 1709, 1617, 1375, 1150, 759  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  8.33 (dd,  $J$  = 8.8, 2.4 Hz, 1 H), 8.18 (dd,  $J$  = 7.8, 2.6 Hz, 2 H), 7.34 (dd,  $J$  = 8.8 Hz, 1 H), 6.27 (d,  $J$  = 2.8 Hz, 1 H), 6.20 (s, 1 H), 6.02 (s, 1 H), 5.63 (s, 1 H), 5.55 (s, 1 H), 4.63 (s, 2 H), 4.22-4.13 (m, 2 H), 3.91-3.75 (m, 3 H), 3.43

(d,  $J$  = 12.8 Hz, 1 H), 1.45 (s, 9 H), 1.23 (t,  $J$  = 7.0 Hz, 3 H), 1.02 (d,  $J$  = 7.0 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  172.9, 166.1, 165.1, 155.7, 150.1, 148.6, 143.3, 133.7, 133.3, 131.8, 131.4, 127.7, 127.5, 126.4, 121.7, 111.1, 97.6, 84.5, 68.7, 61.3, 61.2, 41.4, 35.2, 27.5, 14.4, 14.1; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{28}\text{H}_{32}\text{N}_4\text{NaO}_{10}$   $[\text{M}+\text{Na}]^+$  607.2016, found 607.1944.



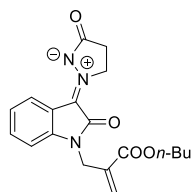
[Reaction time: 5 min]; **3j**: 250 mg, 80%, a yellow solid, m.p. 199.1-201.2 °C; IR (thin film):  $\nu_{\max}$  3417, 3103, 2950, 1730, 1713, 1573, 1350, 1267, 1113, 761  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.44 (d,  $J$  = 7.6 Hz, 1 H), 7.39 (m, 1 H), 7.16 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.86 (d,  $J$  = 8.0 Hz, 1 H), 6.35 (s, 1 H), 5.61 (s, 1 H), 5.01 ( $\psi$ t,  $J$  = 7.4 Hz, 2 H), 4.65 (s, 2 H), 3.82 (s, 3 H), 2.89 ( $\psi$ t,  $J$  = 7.6 Hz, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.8, 160.9, 141.5, 133.5, 132.7, 127.9, 126.8, 124.5, 123.6, 116.9,

109.2, 57.4, 52.3, 40.6, 28.4;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.8, 160.9, 141.5, 133.5, 132.7, 127.9, 126.8, 124.5, 123.6, 116.9, 109.2, 57.4, 52.3, 40.6, 28.4; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{16}\text{H}_{16}\text{N}_3\text{O}_4$  [ $\text{M}+\text{H}$ ] $^+$  314.1141, found 314.1138.



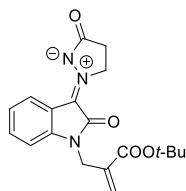
[Reaction time: 9 min]; **3k**: 239 mg, 70%, a white solid, m.p. 159.4-162.4 °C; IR (thin film):  $\nu_{\max}$  3410, 3118, 2964, 2885, 1708, 1604, 1694, 1265, 1139, 762  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.43 (d,  $J$  = 7.6 Hz, 1 H), 7.39 (m, 1 H), 7.15 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.85 (d,  $J$  = 8.0 Hz, 1 H), 6.34 (s, 1 H), 5.59 (s, 1 H), 5.01 (t,  $J$  = 7.4 Hz, 2 H), 4.65 (s, 2H), 4.17 (t,  $J$  = 6.8 Hz, 2 H), 2.89 (t,  $J$  = 7.4 Hz, 2 H), 1.76-1.69 (m, 2 H), 0.89 (t,  $J$  =

7.4 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.4, 160.9, 141.6, 133.8, 132.6, 127.8, 126.4, 124.5, 123.6, 116.9, 109.2, 66.9, 57.4, 40.6, 28.4, 21.9, 10.4; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_4\text{Na}$  [ $\text{M}+\text{Na}$ ] $^+$  364.1273, found 364.1266.



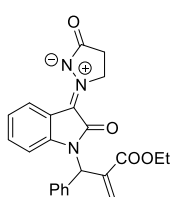
[Reaction time: 15 min]; **3l**: 142 mg, 40%, a yellow solid, m.p. 144.6-146.2 °C; IR (thin film):  $\nu_{\max}$  3437, 2962, 2937, 2873, 1730, 1693, 1604, 1344, 1151, 755  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.44 (d,  $J$  = 7.6 Hz, 1 H), 7.38 (d,  $J$  = 8.0 Hz, 1 H), 7.15 ( $\psi$ t,  $J$  = 7.4 Hz, 1 H), 6.86 (d, 1 H), 6.33 (s, 1 H), 5.59 (s, 1 H), 5.01 (t,  $J$  = 7.4 Hz, 2 H), 4.65 (s, 2 H), 4.22 (t,  $J$  = 6.6 Hz, 2 H), 2.89 (t,  $J$  = 7.6 Hz, 2 H), 1.71-1.64 (m, 2 H),

1.46-1.37 (m, 2H), 0.95 (t,  $J$  = 7.4 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.4, 160.9, 141.6, 133.8, 132.7, 127.9, 126.4, 124.6, 123.6, 116.9, 109.2, 65.2, 57.4, 40.6, 30.6, 28.5, 19.2, 13.7; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{19}\text{H}_{22}\text{N}_3\text{O}_4$  [ $\text{M}+\text{H}$ ] $^+$  356.1610, found 356.1601.



[Reaction time: 23 min]; **3m**: 199 mg, 56%, a yellow solid, m.p. 177.1-185.4 °C; IR (thin film):  $\nu_{\max}$  3381, 3110, 3008, 2977, 2930, 1698, 1605, 1372, 1257, 1143, 758  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.44 (d,  $J$  = 7.6 Hz, 1 H), 7.39 (td,  $J$  = 7.8, 1.2 Hz, 1 H), 7.15 (m, 1 H), 6.85 (d,  $J$  = 7.6 Hz, 1 H), 6.22 (s, 1 H), 5.45 (s, 1 H), 5.01 (t,  $J$  = 7.6 Hz, 2 H), 4.61 (s, 2 H), 2.89 (t,  $J$  = 7.6 Hz, 2 H), 1.52 (s, 9 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):

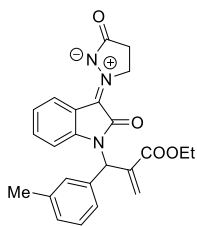
$\delta$  186.7, 164.6, 160.9, 141.6, 135.1, 132.7, 127.9, 125.1, 124.6, 123.6, 116.9, 109.2, 81.9, 57.4, 40.6, 28.5, 28.1 (3 C). HRMS (ESI):  $m/z$  calcd for  $\text{C}_{19}\text{H}_{22}\text{N}_3\text{O}_4$  [ $\text{M}+\text{H}$ ] $^+$  356.1610, found 356.1602.



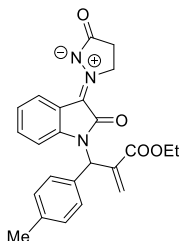
[Reaction time: 5 min]; **6a**: 169 mg, 42%, a yellow solid, m.p. 103.1-106.6 °C; IR (thin film):  $\nu_{\max}$  3424, 2980, 1708, 1602, 1368, 1261, 1149, 750  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.49 (d,  $J$  = 7.2 Hz, 1 H), 7.39-7.36 (m, 1 H), 7.35-7.32 (m, 2 H), 7.30 (s, 1 H), 7.25 (dd,  $J$  = 7.8, 1.0 Hz, 1 H), 7.11 (m, 1 H), 6.81 (d,  $J$  = 8.0 Hz, 1 H), 6.62 (s, 1 H), 6.59 (d,  $J$  = 1.2 Hz, 1 H), 6.64 (d,  $J$  = 2.0 Hz, 1 H), 5.01 (ddd,  $J$  = 32.0, 16.4, 7.2 Hz, 2 H), 4.20-4.13 (m, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 1.16 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$

NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.4, 161.0, 141.9, 138.4, 136.0, 132.4, 129.3, 128.9, 128.3, 128.0, 127.9, 124.5, 123.3, 117.2, 110.9, 61.4, 57.5, 56.1, 28.4, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{22}\text{N}_3\text{O}_4$  [ $\text{M}+\text{H}$ ] $^+$  404.1610, found 404.1566.

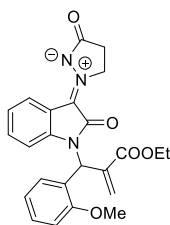




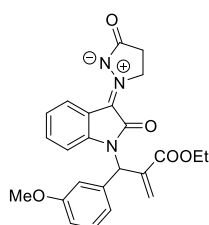
[Reaction time: 5 min]; **6c**: 213 mg, 56%, a yellow solid, m.p. 173.4-175.2 °C; IR (thin film):  $\nu_{\text{max}}$  3426, 2928, 1714, 1634, 1346, 1368, 1600, 1255, 745, 668  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.48 (d,  $J$  = 7.2 Hz, 1 H), 7.28-7.22 (m, 2 H), 7.14-7.08 (m, 4 H), 6.83 (d,  $J$  = 8.0 Hz, 1 H), 6.57 (s, 2 H), 5.62 (d,  $J$  = 1.6 Hz, 1 H), 5.09-4.95 (m, 2 H), 4.21-4.12 (m, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 2.23 (s, 3 H), 1.16 (t,  $J$  = 7.0 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.5, 161.0, 142.0, 138.7, 138.5, 136.0, 132.4, 129.1, 128.8, 128.6, 127.9, 124.9, 124.6, 123.3, 117.2, 111.0, 61.3, 57.4, 56.2, 29.7, 28.5, 21.5, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  418.1767, found 418.1698.



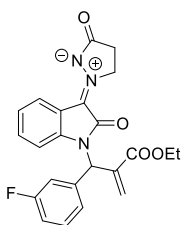
[Reaction time: 5 min]; **6d**: 154 mg, 37%, a yellow solid, m.p. 162.7-165.4 °C; IR (thin film):  $\nu_{\text{max}}$  3405, 2982, 1715, 1688, 1604, 1368, 1152, 746, 700  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.48 (d,  $J$  = 7.2 Hz, 1 H), 7.28-7.22 (m, 2 H), 7.14-7.08 (m, 4 H), 6.83 (d,  $J$  = 8.0 Hz, 1 H), 6.57 (s, 2 H), 5.62 (d,  $J$  = 1.6 Hz, 1 H), 5.09-4.95 (m, 2 H), 4.21-4.12 (m, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 2.32 (s, 3 H), 1.16 (t,  $J$  = 7.0 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.5, 161.0, 142.0, 138.7, 138.5, 136.0, 132.4, 129.1, 128.8, 128.6, 127.9, 124.9, 124.6, 123.2, 117.2, 111.0, 61.3, 57.4, 56.2, 28.4, 28.1, 21.5, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  418.1767, found 418.1744.



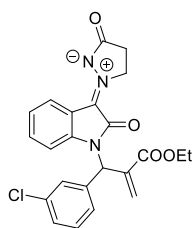
[Reaction time: 2h]; **6e**: 71 mg, 16%, a yellow solid, m.p. 153.4-155.7 °C; IR (thin film):  $\nu_{\text{max}}$  3408, 2978, 1709, 1567, 1461, 1258, 1154, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.48 (dd,  $J$  = 7.6, 0.8 Hz, 1 H), 7.34-7.28 (m, 2 H), 7.25 (m, 1 H), 7.08 (td,  $J$  = 7.8, 0.8 Hz, 1 H), 6.94 (m, 1 H), 6.90 (d,  $J$  = 8.0 Hz, 1 H), 6.85 (d,  $J$  = 8.0 Hz, 1 H), 6.77 (s, 1 H), 6.51 (d,  $J$  = 1.2 Hz, 1 H), 5.56 (d,  $J$  = 1.2 Hz, 1 H), 5.66 (s, 1 H), 5.07-4.93 (m, 2 H), 4.19-4.09 (m, 2 H), 3.79 (s, 3 H), 2.87 (t,  $J$  = 7.2 Hz, 2 H), 1.15 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.5, 161.0, 156.9, 142.5, 137.6, 132.5, 129.8, 128.6, 127.9, 124.9, 124.2, 123.1, 120.6, 117.1, 110.8, 111.0, 61.2, 57.3, 55.6, 51.7, 28.5, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{23}\text{N}_3\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  456.1535, found 456.1527.



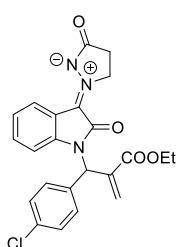
[Reaction time: 5 min]; **6f**: 277 mg, 64%, a yellow solid, m.p. 92.8-93.9 °C; IR (thin film):  $\nu_{\text{max}}$  3418, 2979, 2837, 1711, 1602, 1368, 1263, 750, 670  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.48 (dd,  $J$  = 8.0, 0.8 Hz, 1 H), 7.30-7.24 (m, 2 H), 7.10 (td,  $J$  = 7.8, 0.6 Hz, 1 H), 6.91-6.82 (m, 4 H), 6.59 (s, 2 H), 5.66 (s, 1 H), 5.08-4.94 (m, 2 H), 4.21-4.12 (m, 2 H), 3.76 (s, 3 H), 2.87 (t,  $J$  = 7.6 Hz, 1 H), 1.17 (t,  $J$  = 7.0 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.4, 161.0, 160.0, 141.9, 138.3, 137.6, 132.4, 130.0, 129.3, 127.9, 124.5, 123.3, 120.2, 117.2, 114.2, 113.2, 111.0, 61.3, 57.5, 56.0, 55.3, 28.4, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_5$   $[\text{M}+\text{H}]^+$  434.1716, found 434.1710.



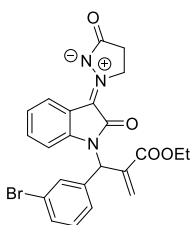
[Reaction time: 5 min]; **6i**: 328 mg, 71%, a yellow solid, m.p. 106.3-109.1 °C; IR (thin film):  $\nu_{\text{max}}$  3398, 2979, 2943, 1712, 1593, 1388, 1145, 751, 689  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.49 (d,  $J$  = 7.2 Hz, 1 H), 7.35-7.33 (m, 1 H), 7.29-7.25 (m, 1 H), 7.15-7.10 (m, 2 H), 7.05-7.01 (m, 2 H), 6.81 (d,  $J$  = 8.0 Hz, 1 H), 6.62 (s, 2 H), 5.67 (s, 1 H), 5.09-5.50 (m, 2 H), 4.23-4.12 (m, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 1.17 (t,  $J$  = 7.0 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.2, 161.0, 141.5, 138.7 (d), 137.8, 132.4, 130.6 (d), 129.5, 128.0, 124.2, 123.6 (d), 123.5, 117.3, 115.5, 115.3 (d), 115.0, 110.7, 61.5, 57.5, 55.6, 28.4, 14.0;  $^{19}\text{F}$  NMR (376 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  -111.5; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{FN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  422.1516, found 422.1546.



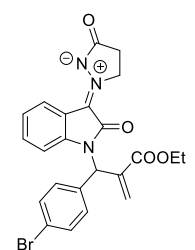
[Reaction time: 8 min]; **6l**: 167 mg, 38%, a yellow solid, m.p. 96.1-98.3 °C; IR (thin film):  $\nu_{\max}$  3426, 2980, 1710, 1593, 1385, 1149, 1026, 750, 686  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.50 (d,  $J$  = 7.2 Hz, 1 H), 7.32-7.28 (m, 4 H), 7.22-7.19 (m, 1 H), 7.14 (m, 1 H), 6.81 (d,  $J$  = 8.0 Hz, 1 H), 6.62 (d,  $J$  = 1.6 Hz, 1 H), 6.59 (s, 1 H), 5.66 (d,  $J$  = 1.6 Hz, 1 H), 5.09-4.95 (m, 2 H), 4.23-4.11 (m, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 1.17 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.2, 161.0, 141.4, 138.2, 137.7, 134.9, 132.4, 130.2, 129.6, 128.6, 128.1, 128.0, 126.1, 124.2, 123.5, 117.3, 110.7, 61.5, 57.5, 55.6, 28.4, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{ClN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  438.1221, found 438.1222.



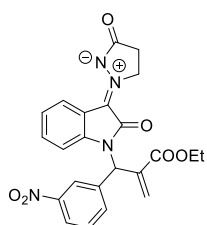
[Reaction time: 5 min]; **6m**: 188 mg, 43%, a yellow solid, m.p. 111.4-111.5 °C; IR (thin film):  $\nu_{\max}$  3423, 2926, 1712, 1603, 1262, 1150, 1014, 750, 678  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.49 (d,  $J$  = 7.6 Hz, 1 H), 7.35-7.33 (m, 2 H), 7.29-7.24 (m, 4 H), 7.13 ( $\psi$ t,  $J$  = 7.2 Hz, 1 H), 6.78 (d,  $J$  = 8.0 Hz, 1 H), 6.59 (s, 1 H), 5.65 (d,  $J$  = 1.6 Hz, 1 H), 5.06-4.96 (m, 2 H), 4.21-4.11 (m, 2 H), 2.88 ( $\psi$ t,  $J$  = 7.4 Hz, 2 H), 1.17 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.2, 161.0, 141.5, 138.0, 134.6, 134.3, 132.4, 129.3, 129.2, 129.0, 128.0, 123.5, 117.3, 110.7, 61.5, 57.5, 55.5, 29.7, 28.4, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{20}\text{ClN}_3\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  460.1040, found 460.1024.



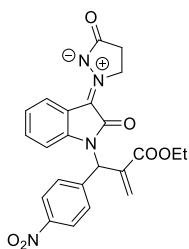
[Reaction time: 10 min]; **6o**: 265 mg, 55%, a yellow solid, m.p. 101.1-104.3 °C; IR (thin film):  $\nu_{\max}$  3421, 2979, 1709, 1593, 1368, 1149, 771, 682  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.50 (d,  $J$  = 7.6 Hz, 1 H), 7.48-7.46 (m, 2 H), 7.31-7.22 (m, 4 H), 7.13 ( $\psi$ t,  $J$  = 7.8 Hz, 1 H), 6.81 (d,  $J$  = 8.0 Hz, 1 H), 6.62 (s, 1 H), 6.58 (s, 1 H), 5.66 (d,  $J$  = 1.2 Hz, 1 H), 5.09-4.95 (m, 2 H), 4.23-4.11 (m, 2 H), 2.88 (t,  $J$  = 7.4 Hz, 2 H), 1.17 (t,  $J$  = 7.0 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.2, 161.0, 141.4, 138.5, 137.7, 132.4, 131.6, 131.0, 130.4, 129.6, 128.1, 126.5, 124.2, 123.5, 123.0, 117.3, 110.7, 61.5, 57.6, 55.5, 28.4, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{BrN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  482.0715, found 482.0714.



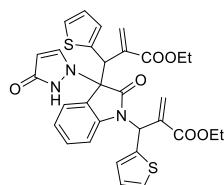
[Reaction time: 5 min]; **6p**: 216 mg, 45%, a yellow solid, m.p. 112.7-114.8 °C; IR (thin film):  $\nu_{\max}$  3568, 3425, 2979, 1704, 1596, 1371, 1270, 1071, 749, 682  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.49 (d,  $J$  = 7.2 Hz, 1 H), 7.49 (d,  $J$  = 8.4 Hz, 2 H), 7.27 (m, 1 H), 7.19 (d,  $J$  = 8.4 Hz, 2 H), 7.12 (m, 1 H), 6.78 (d,  $J$  = 8.0 Hz, 1 H), 6.60 (d,  $J$  = 1.2 Hz, 1 H), 6.58 (s, 1 H), 5.56 (d,  $J$  = 1.6 Hz, 1 H), 5.08-4.94 (m, 2 H), 4.23-4.11 (m, 2 H), 2.88 (t,  $J$  = 7.6 Hz, 2 H), 1.17 (t,  $J$  = 27.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.7, 165.2, 161.0, 141.5, 137.9, 135.2, 132.4, 132.1, 129.6, 129.4, 128.0, 124.2, 123.5, 122.4, 117.3, 110.7, 61.5, 57.5, 55.6, 28.4, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{BrN}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  482.0715, found 482.0715.



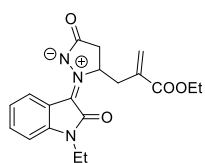
[Reaction time: 2 min]; **6r**: 211 mg, 47%, a yellow solid, m.p. 99.6-101.7 °C; IR (thin film):  $\nu_{\max}$  3400, 2979, 2927, 1709, 1567, 1349, 1150, 748  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.51 (d,  $J$  = 7.6 Hz, 1 H), 8.21 (d,  $J$  = 8.8 Hz, 1 H), 8.20 (s, 1 H), 7.68 (d,  $J$  = 8.0 Hz, 1 H), 7.58 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 7.30 (m, 1 H), 7.15 (t,  $J$  = 7.6 Hz, 1 H), 6.80 (d,  $J$  = 8.0 Hz, 1 H), 6.72 (s, 1 H), 6.68 (d,  $J$  = 1.2 Hz, 1 H), 5.70 (d,  $J$  = 1.6 Hz, 1 H), 5.04-4.99 (m, 2 H), 4.24-4.15 (m, 2 H), 2.89 (t,  $J$  = 7.6 Hz, 2 H), 1.2 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.6, 165.0, 161.0, 148.6, 140.9, 138.5, 137.1, 134.0, 132.4, 130.0, 129.9, 128.1, 123.8, 123.4, 123.0, 117.4, 110.3, 61.7, 57.7, 55.4, 28.3, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{N}_4\text{O}_6$   $[\text{M}+\text{H}]^+$  449.1461, found 449.1450.



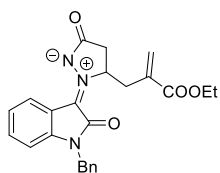
[Reaction time: 2 min]; **6s**: 242 mg, 54%, a yellow solid, m.p. 199.7-122.4 °C; IR (thin film):  $\nu_{\max}$  3418, 2982, 1716, 1633, 1606, 1299, 1147, 750, 696  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.51 (d,  $J$  = 7.6 Hz, 1 H), 8.23 (d,  $J$  = 8.8 Hz, 2 H), 7.51 (d,  $J$  = 8.8 Hz, 2 H), 7.32-7.27 (m, 1 H), 7.16 (t,  $J$  = 7.6 Hz, 1 H), 6.79 (d,  $J$  = 7.6 Hz, 1 H), 6.73 (s, 1 H), 6.68 (s, 1 H), 5.69 (d,  $J$  = 1.6 Hz, 1 H), 5.08-4.95 (m, 2 H), 4.24-4.15 (m, 2 H), 2.89 (t,  $J$  = 7.6 Hz, 2 H), 1.20 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  186.6, 165.0, 161.0, 147.7, 143.5, 141.0, 137.1, 132.4, 130.0, 129.0, 128.9, 128.1, 124.1, 123.8, 123.7, 117.4, 110.4, 61.7, 57.7, 55.4, 29.7, 28.3, 14.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{N}_4\text{O}_6$   $[\text{M}+\text{H}]^+$  449.1461, found 449.1450



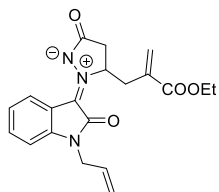
[Reaction time: 5 min]; **6't**: 156 mg, 34%, a white solid, m.p. 199.7-200.9 °C,  $dr > 20:1$ ; IR (thin film):  $\nu_{\max}$  3410, 3126, 3923, 2851, 1734, 1713, 1610, 1300, 1157, 760  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  9.84 (s, 1H), 7.63 (d,  $J$  = 7.2 Hz, 1 H), 7.46 (dd,  $J$  = 4.8, 2.8 Hz, 1 H), 7.40 ( $\psi$ s, 1 H), 7.30 (dd,  $J$  = 4.8, 2.8 Hz, 1 H), 7.25 (d,  $J$  = 7.2 Hz, 1 H), 7.15-7.11 (m, 2 H), 7.00 ( $\psi$ t,  $J$  = 7.6-7.2 Hz, 1 H), 6.89 (dd,  $J$  = 5.2, 0.8 Hz, 1 H), 6.86 (dd,  $J$  = 4.8, 0.8 Hz, 1 H), 6.57-6.53 (m, 3 H), 6.46 (s, 1 H), 5.78 (s, 1 H), 5.57 (d,  $J$  = 2.4 Hz, 1 H), 4.12-4.01 (m, 2 H), 3.83 (d,  $J$  = 12.8 Hz, A of AB, 1 H), 3.80-3.72 (m, 1 H), 3.55-3.47 (m, 1 H), 3.42 (d,  $J$  = 12.8 Hz, B of AB, 1H), 1.06 (t,  $J$  = 7.2 Hz, 3H), 0.90 (t,  $J$  = 7.2 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  172.5, 168.3, 165.5, 162.0, 142.5, 137.6, 136.2, 136.1, 133.2, 130.2, 129.9, 129.5, 128.2, 127.9, 127.8, 127.5, 127.1, 126.7, 125.7, 124.3, 124.1, 122.6, 122.0, 92.3, 79.7, 68.2, 61.5, 61.0, 51.9, 14.1, 13.7; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{31}\text{H}_{30}\text{N}_3\text{O}_6\text{S}_2$   $[\text{M}+\text{H}]^+$  604.1576, found 604.1461.



[Reaction time: 12 h]; **8b**: 171 mg, 48%, a yellow solid, m.p. 202.1-204.0 °C; IR (thin film):  $\nu_{\max}$  3404, 2984, 2941, 1705, 1687, 1604, 1373, 1156, 1127, 779, 667  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.46 (d,  $J$  = 7.6 Hz, 1 H), 7.42 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 7.13 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.88 (d,  $J$  = 8.0 Hz, 1 H), 6.36 (s, 1 H), 6.07 (dd,  $J$  = 15.6, 6.4 Hz, 1 H), 5.74 (s, 1 H), 4.23-4.15 (m, 2 H), 3.89-3.79 (m, 2 H), 2.96-2.90 (m, 3 H), 2.68 (dd,  $J$  = 16.8, 1.2 Hz, 1 H), 1.33-1.27 (m, 6 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.3, 166.1, 160.0, 142.0, 135.2, 132.8, 129.1, 128.6, 123.3, 122.5, 117.3, 68.2, 61.3, 37.3, 35.0, 34.6, 14.1, 13.0; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{19}\text{H}_{22}\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  356.1610, found 356.1612.



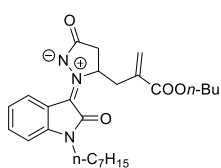
[Reaction time: 7 h]; **8c**: 313 mg, 74%, a red solid, m.p. 163.2-164.8 °C; IR (thin film):  $\nu_{\max}$  3419, 2979, 2931, 1703, 1692, 1608, 1595, 1386, 1158, 1140, 756, 661  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 Hz,  $\text{CDCl}_3$ ):  $\delta$  8.45 (d,  $J$  = 7.2 Hz, 1 H), 7.36-7.27 (m, 6 H), 7.10 (td,  $J$  = 8.0-7.6 Hz, 1 H), 6.77 (d,  $J$  = 8.0 Hz, 1 H), 6.37 (s, 1 H), 6.15-6.09 (m, 1 H), 5.74 (s, 1 H), 5.02 (d,  $J$  = 16.0 Hz, A of AB, 1 H), 4.97 (d,  $J$  = 16.0 Hz, B of AB, 1 H), 4.22-4.14 (m, 2 H), 3.06-2.92 (m, 3 H), 2.70 (dd,  $J$  = 16.8, 1.6 Hz, 1 H), 1.27 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.3, 166.1, 160.5, 142.1, 135.1, 132.8, 129.3, 129.0, 128.7, 128.5, 128.0, 127.2, 125.7, 123.5, 117.3, 109.2, 68.3, 61.3, 43.9, 37.4, 34.7, 14.1; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  418.1767, found 418.1760.



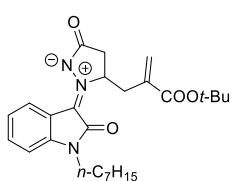
[Reaction time: 8 h]; **8d**: 323 mg, 88%, a yellow solid, m.p. 124.2-125.6 °C; IR (thin film):  $\nu_{\max}$  3402, 2981, 2925, 1703, 1607, 1589, 1384, 1261, 1026, 759, 687  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.46 (d,  $J$  = 7.6 Hz, 1 H), 7.39 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 7.13 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.86 (d,  $J$  = 7.6 Hz, 1 H), 6.36 (s, 1 H), 6.07 (dd,  $J$  = 7.6, 6.8 Hz, 1 H), 5.91-5.81 (m, 1 H), 5.74 (s, 1 H), 5.28 (d,  $J$  = 2.8 Hz, 1 H), 5.25 (s, 1 H), 4.42 (d,  $J$  = 4.0 Hz, 2 H), 4.24-4.12 (m, 2 H), 3.02-2.91 (m, 3 H), 2.68 (d,  $J$  = 16.8 Hz, B of AB, 1 H), 1.28 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.3, 166.1, 160.0, 142.1, 135.1, 132.8, 130.8,



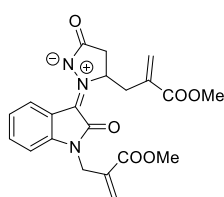
34.6, 31.7, 28.9, 27.7, 26.9, 22.6, 14.0; HRMS (ESI):  $m/z$  calcd for  $C_{23}H_{30}N_3O_4$   $[M+H]^+$  412.2236, found 412.2233.



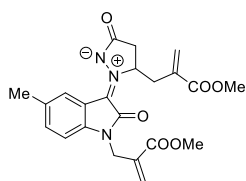
[Reaction time: 8 h]; **8j**: 349 mg, 77%, a yellow solid, m.p. 96.2-97.7 °C; IR (thin film):  $\nu_{\max}$  3418, 2955, 2871, 1718, 1694, 1607, 1294, 1130, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.46 (d,  $J$  = 7.6 Hz, 1 H), 7.41 (m, 1 H), 7.12 (m, 1 H), 6.87 (d,  $J$  = 8.0 Hz, 1 H), 6.36 (s,  $J$  = 7.2 Hz, 1 H), 6.09-6.03 (m, 1 H), 5.74 (s, 1 H), 4.18-4.08 (m, 2 H), 3.84-3.70 (m, 2 H), 2.96-2.90 (m, 3 H), 2.68 (dd,  $J$  = 16.8, 1.6 Hz, 1 H), 1.76-1.60 (m, 5 H), 1.43-1.26 (m, 10 H), 0.93 (t,  $J$  = 7.6-7.2 Hz, 3 H), 0.88 (t,  $J$  = 6.8 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.2, 166.2, 160.2, 142.4, 135.2, 132.8, 129.1, 128.6, 125.9, 123.2, 117.3, 108.5, 68.2, 65.2, 40.3, 37.3, 34.5, 31.7, 30.5, 28.9, 27.7, 26.9, 22.6, 19.2, 14.0, 13.7; HRMS (ESI):  $m/z$  calcd for  $C_{26}H_{36}N_3O_4$   $[M+H]^+$  454.2706, found 454.2704.



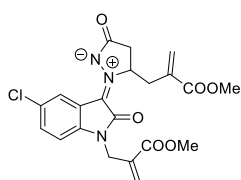
[Reaction time: 8 h]; **8k**: 358 mg, 79%, a yellow solid, m.p. 106.9-108.2 °C; IR (thin film):  $\nu_{\max}$  3374, 3242, 2955, 2859, 1696, 1592, 1294, 1030, 757  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.45 (d,  $J$  = 8.0 Hz, 1 H), 7.40 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 7.11 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.86 (d,  $J$  = 8.0 Hz, 1 H), 6.26 (s, 1 H), 6.04-5.99 (m, 1 H), 5.67 (s, 1 H), 3.83-3.70 (m, 2 H), 3.01-2.84 (m, 3 H), 2.69 (d,  $J$  = 16.4 Hz, B of AB, 1 H), 1.72-1.60 (m, 2 H), 1.45 (s, 9 H), 1.35-1.28 (m, 8 H), 0.87 (t,  $J$  = 6.6 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.3, 165.3, 160.2, 142.4, 136.4, 132.7, 128.6, 128.3, 125.9, 123.1, 117.3, 108.4, 81.5, 77.2, 68.4, 40.3, 36.8, 34.5, 31.7, 28.9, 28.0, 27.9, 27.7, 26.9, 22.6, 14.0; HRMS (ESI):  $m/z$  calcd for  $C_{26}H_{36}N_3O_4$   $[M+H]^+$  454.2706, found 454.2701.



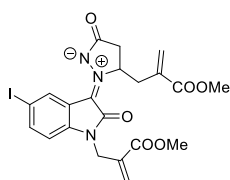
[Reaction time: 8 h]; **4a**: 175 mg, 44%, a yellow solid, m.p. 130.7-131.6 °C; IR (thin film):  $\nu_{\max}$  2999, 2951, 1708, 1628, 1593, 1306, 1167, 685  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.47 (d,  $J$  = 7.2 Hz, 1 H), 7.39 (m, 1 H), 7.15 (m, 1 H), 6.84 (d,  $J$  = 7.6 Hz, 1 H), 6.35 (s, 2 H), 6.09-6.05 (m, 1 H), 5.75 (s, 1 H), 5.64 (s, 1 H), 4.67 (s, 2 H), 3.83 (s, 3 H), 3.69 (s, 3 H), 3.05 (dd,  $J$  = 14.0, 8.0 Hz, 1 H, A of AB), 2.98-2.88 (m, 2 H), 2.70 (dd,  $J$  = 16.8, 1.6 Hz, 1 H, B of AB);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.2, 166.5, 165.8, 160.4, 141.6, 134.6, 133.4, 132.9, 129.7, 128.4, 126.6, 125.4, 123.6, 117.3, 109.1, 68.3, 52.3 (2C), 40.5, 37.2, 34.5; HRMS (ESI):  $m/z$  calcd for  $C_{21}H_{21}N_3NaO_6$   $[M+Na]^+$  434.1328, found 434.1310.



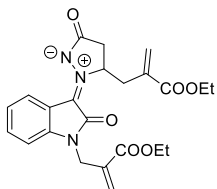
[Reaction time: 6 h]; **4b**: 172 mg, 41%, a yellow solid, m.p. 132.4-133.7 °C; IR (thin film):  $\nu_{\max}$  3403, 2996, 2949, 1710, 1612, 1596, 1147, 686  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.32 (s, 1 H), 7.21 (d,  $J$  = 8.0 Hz, 1 H), 6.73 (d,  $J$  = 8.0 Hz, 1 H), 6.36 (s, 1 H), 6.35 (s, 1 H), 6.12-6.07 (m, 1 H), 5.76 (s, 1 H), 5.62 (s, 1 H), 4.65 (s, 2 H), 3.83 (s, 3 H), 3.71 (s, 3 H), 3.06-2.88 (s, 3 H), 2.69 (dd,  $J$  = 16.8, 1.2 Hz, 1 H, B of AB), 2.37 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.3, 166.5, 165.9, 160.4, 139.5, 134.7, 133.5 (2C), 133.4, 129.6, 128.9, 126.5, 125.9, 117.2, 108.9, 68.2, 52.3 (2C), 40.5, 37.3, 34.6, 21.0; HRMS (ESI):  $m/z$  calcd for  $C_{22}H_{23}N_3NaO_6$   $[M+Na]^+$  448.1485, found 448.4179.



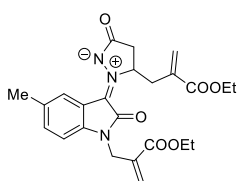
[Reaction time: 7 h]; **4c**: 163 mg, 37%, a yellow solid, m.p. 129.7-131.6 °C; IR (thin film):  $\nu_{\max}$  3408, 2950, 1712, 1632, 1605, 1149, 1123, 679  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.48 (d,  $J$  = 2.0 Hz, 1 H), 7.36 (dd,  $J$  = 10.4, 8.4 Hz, 1 H), 6.82 (d,  $J$  = 8.4 Hz, 1 H), 6.38 (s, 1 H), 6.37 (s, 1 H), 6.12-6.07 (m, 1 H), 5.77 (s, 1 H), 5.66 (s, 1 H), 4.67 (s, 2 H), 3.83 (s, 3 H), 3.71 (s, 3 H), 3.08 (dd,  $J$  = 14.0, 7.6 Hz, 1 H, A of AB), 3.00-2.87 (m, 2 H), 2.71 (dd,  $J$  = 16.8, 1.2 Hz, 1 H, B' of A'B');  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.2, 166.5, 165.8, 160.1, 139.8, 134.4, 133.2, 132.2, 129.9, 129.2, 127.7, 127.0, 124.1, 118.4, 110.1, 68.7, 52.4 (2C), 40.7, 37.2, 34.3; HRMS (ESI):  $m/z$  calcd for  $C_{21}H_{20}ClN_3NaO_6$   $[M+Na]^+$  468.0938, found 468.0922.



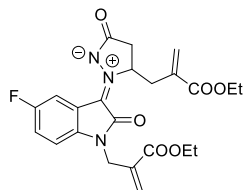
[Reaction time: 6 h]; **4d**: 263 mg, 49%, a red solid, m.p. 157.2-158.7 °C; IR (thin film):  $\nu_{\text{max}}$  3395, 3004, 2956, 1712, 1630, 1599, 1178, 1137, 675, 640  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.77 (d,  $J$  = 1.6 Hz, 1 H), 7.71 (dd,  $J$  = 8.0, 1.6 Hz, 1 H), 6.67 (d,  $J$  = 8.0 Hz, 1 H), 6.37 (s, 1 H), 6.36 (s, 1 H), 6.13-6.07 (m, 1 H), 5.77 (s, 1 H), 5.66 (s, 1 H), 4.66 (s, 2 H), 3.83 (s, 3 H), 3.72 (s, 3 H), 3.06-2.89 (s, 3 H), 2.71 (dd,  $J$  = 16.8, 1.6 Hz, 1 H, B of AB);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.2, 166.5, 165.8, 159.8, 141.1, 140.9, 135.8, 134.4, 133.2, 129.9, 127.0, 123.7, 119.0, 111.1, 86.2, 68.7, 52.4, 40.6, 37.2, 34.4; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{21}\text{H}_{20}\text{IN}_3\text{NaO}_6$   $[\text{M}+\text{Na}]^+$  560.0294, found 560.0277.



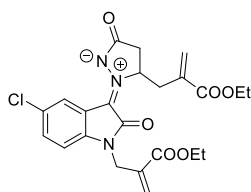
[Reaction time: 7 h]; **4e**: 338 mg, 77%, a yellow oil; IR (thin film):  $\nu_{\text{max}}$  2988, 2928, 1713, 1600, 1303, 1153, 683  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.47 (d,  $J$  = 7.2 Hz, 1 H), 7.39 (m, 1 H), 7.14 ( $\psi$ t,  $J$  = 7.6 Hz, 1 H), 6.85 (d,  $J$  = 7.6 Hz, 1 H), 6.35 (s, 1 H), 6.34 (s, 1 H), 6.10-6.05 (m, 1 H), 5.74 (s, 1 H), 5.61 (s, 1 H), 4.67 (s, 2 H), 4.28 (q,  $J$  = 7.2 Hz, 2 H), 4.20-4.10 (m, 2 H), 3.04 (dd,  $J$  = 14.0, 8.0 Hz, A of AB, 1 H), 2.95 (dd,  $J$  = 16.8, 8.4 Hz, A' of A'B', 1 H), 2.91 (dd,  $J$  = 14.0, 4.4 Hz, B of AB, 1 H), 2.69 (dd,  $J$  = 16.8, 1.2 Hz, B' of A'B', 1 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H), 1.27 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.2, 166.1, 165.4, 160.4, 141.7, 135.0, 133.7, 132.8, 129.3, 128.4, 126.3, 125.3, 123.6, 117.3, 109.1, 77.2, 68.4, 61.3, 40.5, 37.2, 34.5, 14.2, 14.1; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{26}\text{N}_3\text{O}_6$   $[\text{M}+\text{H}]^+$  440.1822, found 440.1823.



[Reaction time: 7 h]; **4f**: 225 mg, 50%, a yellow oil; IR (thin film):  $\nu_{\text{max}}$  2927, 2856, 1607, 1562, 1480, 1366, 1299, 1131, 696  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.32 (s, 1 H), 7.19 (d,  $J$  = 7.6 Hz, 1 H), 6.73 (d,  $J$  = 8.0 Hz, 1 H), 6.35 (s, 1 H), 6.33 (s, 1 H), 6.10-6.05 (m, 1 H), 5.74 (s, 1 H), 5.59 (s, 1 H), 4.64 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 4.21-4.13 (m, 2 H), 3.01 (dd,  $J$  = 14.6, 8.2 Hz, A of AB, 1 H), 2.93 (dd,  $J$  = 17.0, 8.4 Hz, A' of A'B', 1 H), 2.91 (dd,  $J$  = 14.6, 4.0 Hz, B of AB, 1 H), 2.69 (dd,  $J$  = 17.2-16.8, 1.2-0.8 Hz, B' of A'B', 1 H), 2.36 (s, 3 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H), 1.27 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.3, 166.1, 165.4, 160.4, 139.6, 135.1, 133.8, 133.5, 133.4, 129.2, 128.9, 126.1, 125.8, 117.3, 108.9, 68.3, 61.3, 40.5, 37.2, 34.6, 29.7, 21.0, 14.2, 14.1; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{27}\text{N}_3\text{O}_6\text{Na}$   $[\text{M}+\text{Na}]^+$  476.1798, found 476.1790.

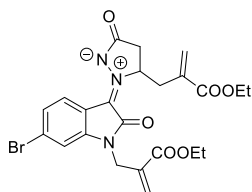


[Reaction time: 5 h]; **4g**: 146 mg, 31%, a yellow oil; IR (thin film):  $\nu_{\text{max}}$  3406, 2989, 2925, 1171, 1565, 1371, 1137, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.23 (dd,  $J$  = 8.4, 2.4 Hz, 1 H), 7.09 (td,  $J$  = 8.4, 2.8 Hz, 1 H), 6.80 (dd,  $J$  = 8.4, 4.0 Hz, 1 H), 6.36 (s, 1 H), 6.35 (s, 1 H), 6.10-6.05 (m, 1 H), 5.75 (s, 1 H), 5.63 (s, 1 H), 4.66 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 4.15 (qd,  $J$  = 7.2, 1.6 Hz, 2 H), 3.06 (dd,  $J$  = 14.0, 7.6 Hz, A of AB, 1 H), 2.94 (dd,  $J$  = 17.2, 8.4 Hz, A' of A'B', 1 H), 2.90 (dd,  $J$  = 14.0, 4.4 Hz, B of AB, 1 H), 2.70 (dd,  $J$  = 17.2, 1.1 Hz, B' of A'B', 1 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H), 1.27 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.1, 166.1, 165.4, 160.4, 160.3, 137.6, 134.8, 133.7, 129.5, 126.6, 119.1, 118.9, 118.2 (d), 115.4 (d), 109.8 (d), 77.2, 68.7, 61.4, 40.7, 37.1, 34.3, 14.2, 14.1;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  -118.7; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{25}\text{FN}_3\text{O}_6$   $[\text{M}+\text{H}]^+$  458.1727, found 458.1729.

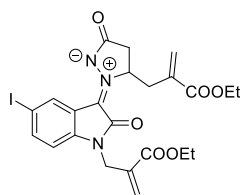


[Reaction time: 6 h]; **4h**: 256 mg, 54%, a yellow solid; m.p. 65.3-67.7 °C; IR (thin film):  $\nu_{\text{max}}$  3409, 2985, 2930, 1713, 1161, 1369, 1298, 1251, 1151, 697  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.48 (d,  $J$  = 2.0 Hz, 1 H), 7.35 (dd,  $J$  = 8.4, 2.0 Hz, 1 H), 6.81 (d,  $J$  = 8.4 Hz, 1 H), 6.36 (s, 1 H), 6.33 (s, 1 H), 6.28 (s, 1 H), 5.83 (ddd,  $J$  = 8.0, 4.8, 0.8 Hz, 1 H), 5.75 (s, 1 H), 5.64 (d,  $J$  = 8.0 Hz, 2 H), 4.69 (d,  $J$  = 17.6 Hz, A of AB, 1 H), 4.63 (d,  $J$  = 17.6 Hz, B of AB, 1 H), 4.28 (q, 7.2-6.8 Hz, 1 H), 4.19 (qd,  $J$  = 7.2, 1.2 Hz, 1 H), 4.15 (q,  $J$  = 7.2 Hz, 2 H), 3.05-2.97 (m, 2 H), 2.87 (td,  $J$  = 14.6, 4.6 Hz, 1 H), 2.48 (dd,  $J$  = 14.0, 9.6 Hz,

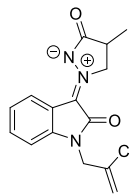
B' of A'B', 1 H), 1.33 (t,  $J$  = 7.2 Hz, 3H), 1.27 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  187.1, 166.3, 165.9, 165.4, 160.0, 139.9, 136.7, 134.6, 132.2, 129.7, 128.6, 126.6, 124.2, 118.4, 110.1, 73.5, 61.3, 61.2, 40.6, 36.8, 33.9, 14.2, 14.1; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{25}\text{ClN}_3\text{O}_6$   $[\text{M}+\text{H}]^+$  474.1432, found 474.1441.



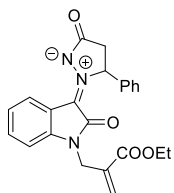
[Reaction time: 12 h]; **4i**: 352 mg, 68%, a yellow oil; IR (thin film):  $\nu_{\text{max}}$  3420, 2982, 2933, 1721, 1603, 1370, 1300, 1062, 750  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.30 (d,  $J$  = 8.4 Hz, 1 H), 7.28 (dd,  $J$  = 8.0, 1.6 Hz, 1 H), 7.02 (d,  $J$  = 1.6 Hz, 1 H), 6.37 (s, 1 H), 6.35 (s, 1 H), 6.06-6.00 (m, 1 H), 5.74 (s, 1 H), 5.62 (s, 1 H), 4.65 (s, 2 H), 4.29 (q,  $J$  = 7.6 Hz, 2 H), 4.14 (qd,  $J$  = 7.2, 0.8 Hz, 2 H), 3.08 (dd,  $J$  = 14.0, 7.6 Hz, A of AB, 1 H), 2.95 (dd,  $J$  = 16.8, 8.4 Hz, A' of A'B', 1 H), 2.87 (dd,  $J$  = 14.2, 3.8 Hz, B of AB, 1 H), 2.69 (dd,  $J$  = 17.2, 1.6 Hz, B' of A'B', 1 H), 1.34 (t,  $J$  = 7.2 Hz, 3 H), 1.26 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.1, 166.0, 165.2, 160.2, 142.4, 134.8, 133.4, 129.5, 129.1, 127.0, 126.6 (2C), 124.2, 116.2, 112.6, 77.2, 68.6, 61.4, 61.3, 40.7, 37.0, 34.3, 14.2 (d); HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{25}\text{BrN}_3\text{O}_6$   $[\text{M}+\text{H}]^+$  518.0927, found 518.0936.



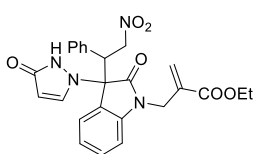
[Reaction time: 8 h]; **4j**: 311 mg, 55%, a yellow oil; IR (thin film):  $\nu_{\text{max}}$  3452, 3342, 1723, 1694, 1621, 1519, 1348, 1109, 977, 809, 749, 686  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.75 (d,  $J$  = 1.6 Hz, 1 H), 7.69 (dd,  $J$  = 8.4, 1.6 Hz, 1 H), 6.65 (d,  $J$  = 8.4 Hz, 1 H), 6.35 (s, 2 H), 6.10-6.05 (m, 1 H), 5.74 (s, 1 H), 5.62 (s, 1 H), 4.65 (s, 2 H), 4.27 (q,  $J$  = 7.2 Hz, 1 H), 4.19-4.12 (m, 2 H), 3.05 (dd,  $J$  = 14.0, 7.6 Hz, A of AB, 1 H), 2.95 (dd,  $J$  = 17.2, 8.4 Hz, A' of A'B', 1 H), 2.88 (dd,  $J$  = 14.2, 4.2 Hz, B of AB, 1 H), 2.70 (dd,  $J$  = 16.8, 1.6 Hz, 1 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H), 1.27 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.2, 166.0, 165.3, 159.8, 141.0, 140.9, 135.8, 134.7, 133.5, 129.5, 126.6, 123.6, 119.1, 111.1, 86.1, 77.2, 68.8, 61.4, 40.6, 37.1, 34.3, 14.2 (d); HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{25}\text{IN}_3\text{O}_6$   $[\text{M}+\text{H}]^+$  566.0788, found 566.0744.



[Reaction time: 2 min]; **10**: 143 mg, 84%, a yellow solid, m.p. 157.2-158.7  $^{\circ}\text{C}$ ; IR (thin film):  $\nu_{\text{max}}$  3684, 3987, 1710, 1694, 1573, 1383, 1267, 1128, 761  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.46 (d,  $J$  = 7.6 Hz, 1 H), 7.39 (m, 1 H), 7.16 (t,  $J$  = 7.6 Hz, 1 H), 6.87 (d,  $J$  = 8.0 Hz, 1 H), 6.35 (s, 1 H), 5.60 (s, 1 H), 5.27 (dd,  $J$  = 16.0, 7.2 Hz, 1 H), 4.63 (s, 2 H), 4.57 (dd,  $J$  = 15.6, 5.6 Hz, 1 H), 4.27 (q,  $J$  = 7.2 Hz, 2 H), 3.04-2.95 (m, 1 H), 1.49 (d,  $J$  = 7.2 Hz, 3 H), 1.33 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  189.7, 165.4, 160.9, 141.5, 133.8, 132.6, 127.9, 126.5, 124.4, 123.6, 116.9, 109.2, 77.2, 64.2, 61.4, 40.6, 34.4, 16.1, 14.2; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{18}\text{H}_{20}\text{N}_3\text{O}_4$   $[\text{M}+\text{H}]^+$  342.1454, found 1455.

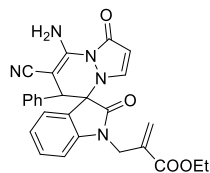


[Reaction time: 30 min]; **12**: 154 mg, 77%, a yellow solid, m.p. 175.3-177.2  $^{\circ}\text{C}$ ; IR (thin film):  $\nu_{\text{max}}$  3384, 2935, 2934, 1604, 1182, 1162, 661, 648  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.60 (d,  $J$  = 7.6 Hz, 1 H), 7.38-7.29 (m, 6 H), 7.13 ( $\psi$ t,  $J$  = 8.0 Hz, 1 H), 6.92-6.89 (m, 1 H), 6.52 (d,  $J$  = 1.2 Hz, 1 H), 5.93 (d,  $J$  = 1.6 Hz, 1 H), 5.36 (q,  $J$  = 7.2 Hz, 1 H), 3.89-3.74 (m, 2 H), 3.3.1 (dd,  $J$  = 17.2, 9.6 Hz, 1 H), 2.71 (dd,  $J$  = 16.8, 1.6 Hz, 1 H), 1.58 (t,  $J$  = 7.2 Hz, 1 H), 0.83 (t,  $J$  = 7.2 Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  185.4, 165.3, 159.7, 141.8, 138.7, 133.7, 133.0, 129.1, 128.8, 126.1, 125.7, 125.1, 123.5, 117.3, 109.0, 72.2, 61.3, 40.4, 38.9, 14.1, HRMS (ESI):  $m/z$  calcd for  $\text{C}_{23}\text{H}_{21}\text{N}_3\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  426.1430, found 426.1423.



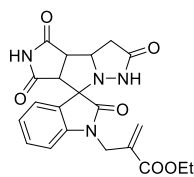
[Reaction time: 2 h]; **13**: 89 mg, 37%, a light yellow solid, m.p. 179.6-183.5  $^{\circ}\text{C}$ ; IR (thin film):  $\nu_{\text{max}}$  2854, 1715, 1663, 1495, 1377, 1303, 1142, 755  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  10.16 (s, 1 H), 7.70 (d,  $J$  = 7.2 Hz, 1 H), 7.11 (d,  $J$  = 2.0 Hz, 1 H), 7.17-7.04 (m, 7 H), 6.71 (d,  $J$  = 8.0 Hz, 1 H), 6.01 (s, 1 H), 5.63 (d,  $J$  = 2.4 Hz, 1 H), 5.47 (dd,  $J$  = 13.2, 11.6 Hz, 1 H), 5.05 (dd,  $J$  = 11.2, 3.6 Hz, 1 H),

4.91 (s, 1 H), 4.86 (dd,  $J=13.6, 3.6$  Hz, 1 H), 4.45 (s, 2 H), 4.19-4.11 (m, 2 H), 1.21 (t,  $J=3.2$  Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  171.4, 165.2, 162.9, 141.3, 133.5, 133.2, 131.3, 130.2, 129.6, 128.7, 128.5, 127.5, 126.4, 125.1, 123.4, 110.2, 93.35, 79.6, 74.9, 70.1, 61.3, 48.2, 41.0, 14.4; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{25}\text{H}_{24}\text{N}_4\text{O}_6\text{Na}$   $[\text{M}+\text{Na}]^+$  499.1594, found 499.1583.



[Reaction time: 20 min]; **14**: 145 mg, 60%, a white solid m.p. 206.0-207.3 °C; IR (thin film):  $\nu_{\text{max}}$  3370, 2981, 2189, 1725, 1683, 1492, 1382, 1172, 1027, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  7.85 (d,  $J=7.2$  Hz, 1 H), 7.72 (s, 2 H), 7.40 (m, 1 H), 7.32 (d,  $J=4.0$  Hz, 1 H), 7.28 (ψt,  $J=7.4$  Hz, 1 H), 7.20-7.30 (m, 3 H), 6.91 (br s, 2 H), 6.77 (d,  $J=7.8$  Hz, 1 H), 5.76 (s, 1 H), 5.67 (d,  $J=4.0$  Hz, 1 H), 4.77 (s, 1 H),

4.20 (d,  $J=16.8$  Hz, B of AB, 1 H), 4.14-4.04 (m, 2 H), 1.18 (t,  $J=7.2$  Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  169.3, 165.4, 165.0, 150.2, 143.3, 142.8, 134.4, 133.3, 132.0, 130.2, 128.6, 128.4, 126.0, 125.2, 124.1, 122.0, 119.5, 110.6, 101.4, 67.1, 61.2, 58.5, 46.4, 14.4; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{27}\text{H}_{24}\text{N}_5\text{O}_4$   $[\text{M}+\text{H}]^+$  482.1828, found 482.1689.



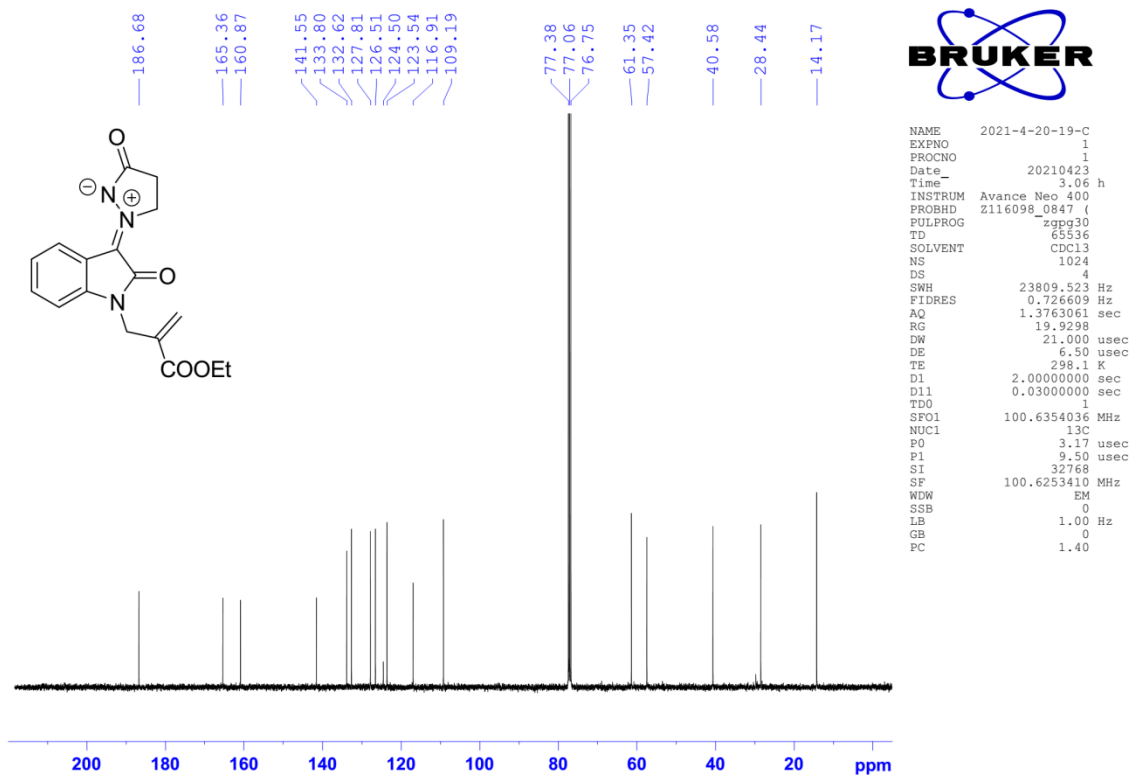
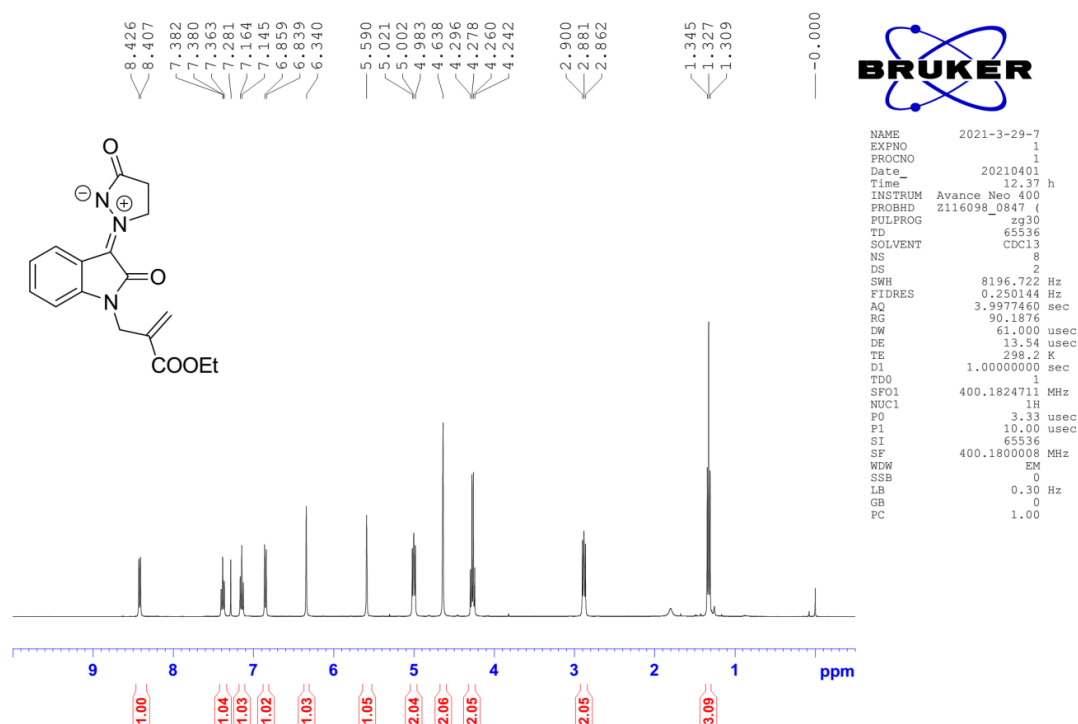
[Reaction time: 2 h]; **15**: 192 mg, 91%, a white solid, m.p. 245.7-247.9 °C; IR (thin film):  $\nu_{\text{max}}$  2982, 2924, 1726, 1702, 1611, 1376, 1152, 1024, 767  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  11.37 (s, 1 H), 9.10 (s, 1 H), 7.35 (t,  $J=8.0$  Hz, 1 H), 7.19 (d,  $J=7.6$  Hz, 1 H), 7.06 (t,  $J=7.6$  Hz, 1 H), 6.99 (d,  $J=8.0$  Hz, 1 H), 6.17 (s, 1 H), 5.50 (s, 1 H), 4.73 (td,  $J=9.2, 4.8$  Hz, 1 H), 4.52 (s, 2 H), 4.21 (q,  $J=7.2$  Hz, 2 H), 3.86 (t,  $J=$

9.4 Hz, 1 H), 3.66 (d,  $J=8.8$  Hz, 1 H), 2.86 (dd,  $J=17.2, 4.8$  Hz, 1 H, A of AB), 2.63 (dd,  $J=17.2, 9.2$  Hz, 1 H, B of AB), 1.26 (t,  $J=7.2$  Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  178.2, 176.4, 175.1, 174.5, 165.4, 143.5, 134.2, 130.6, 128.7, 125.5, 122.6, 122.4, 109.8, 74.9, 63.1, 61.3, 53.6, 49.2, 40.5, 33.9, 14.5; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{21}\text{H}_{21}\text{N}_4\text{O}_6$   $[\text{M}+\text{H}]^+$  425.1461, found 425.1451.

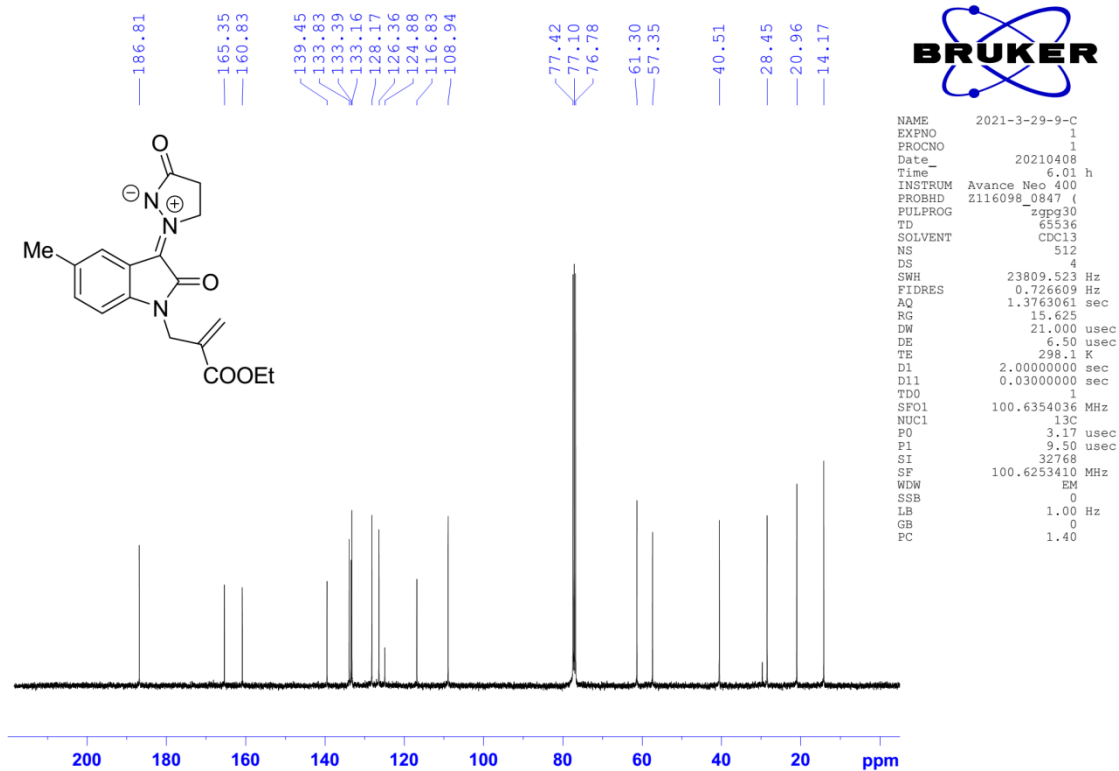
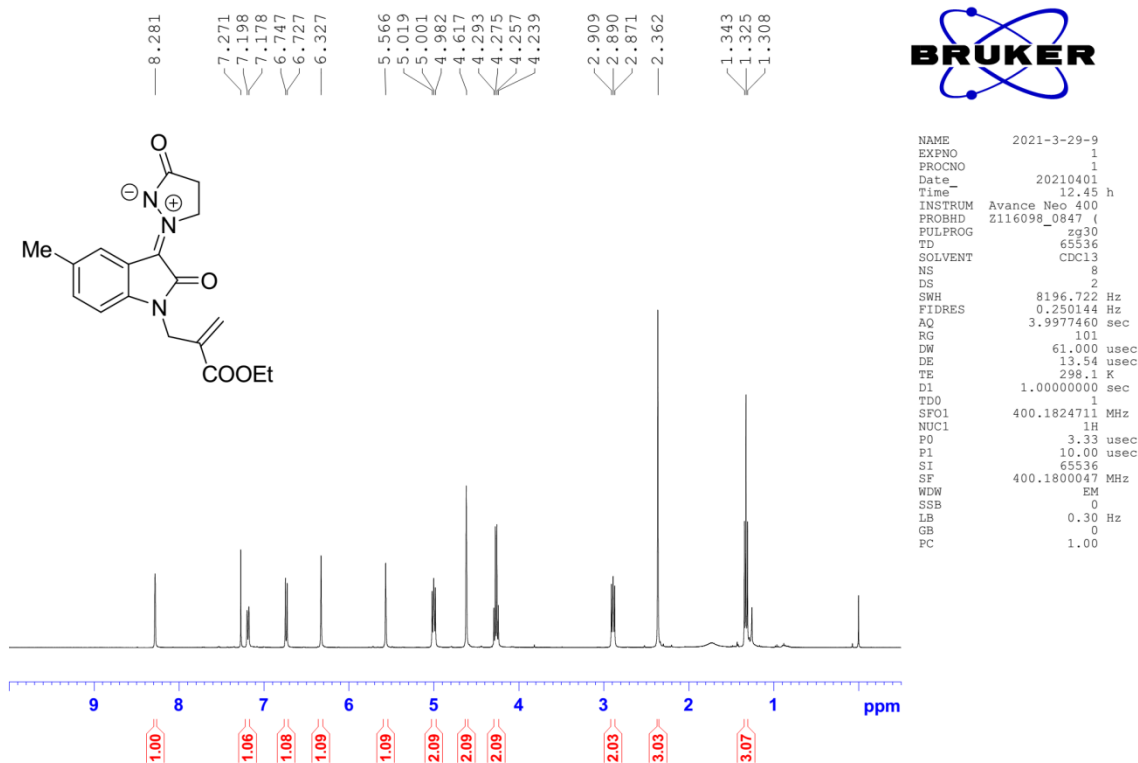


### 3. Copies of NMR for all new compounds.

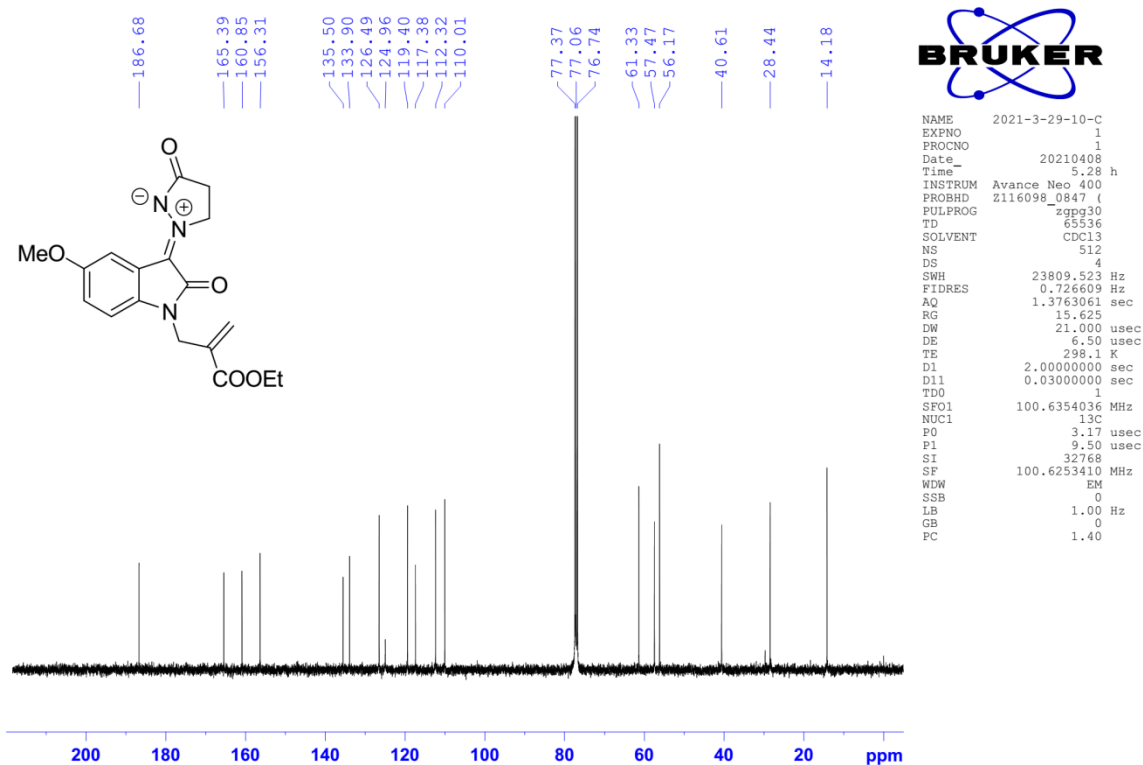
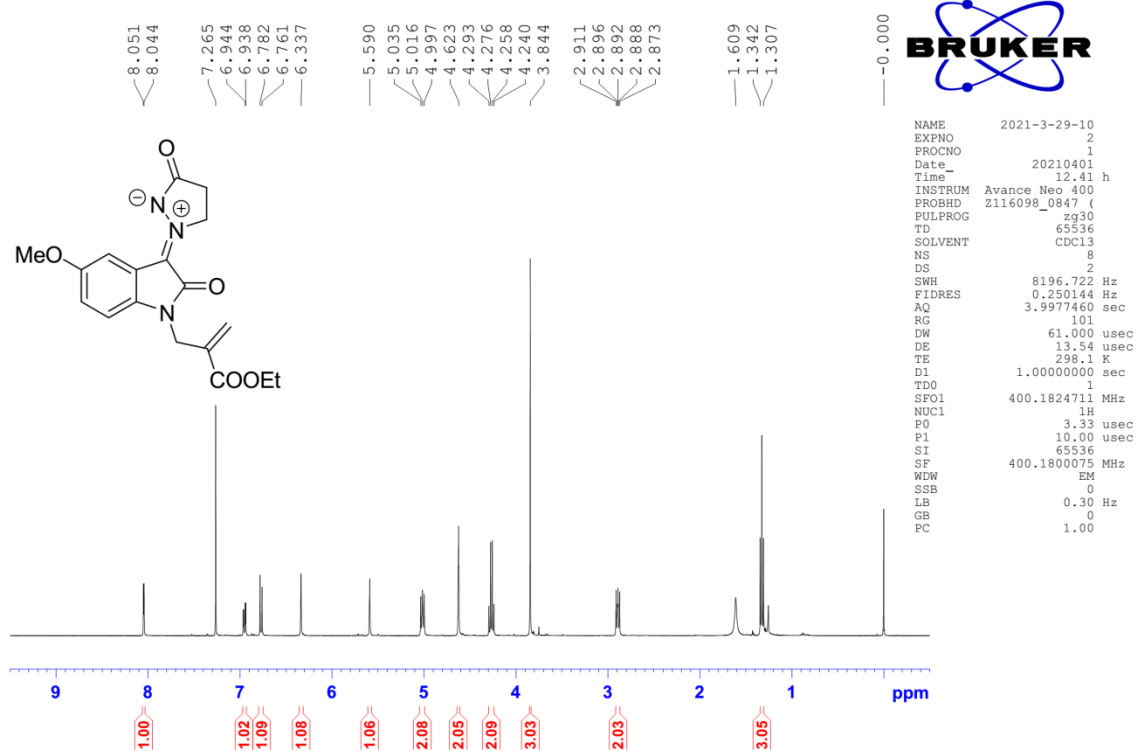
#### <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3a



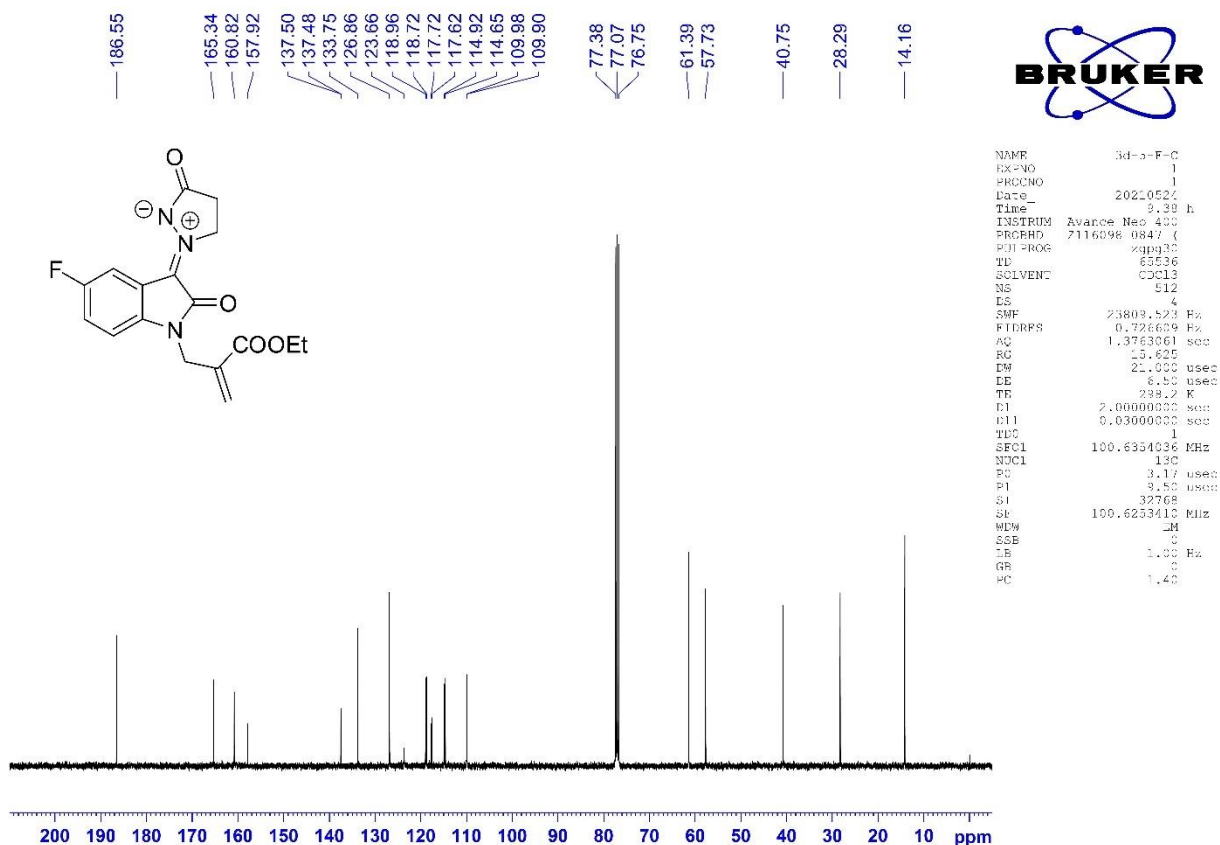
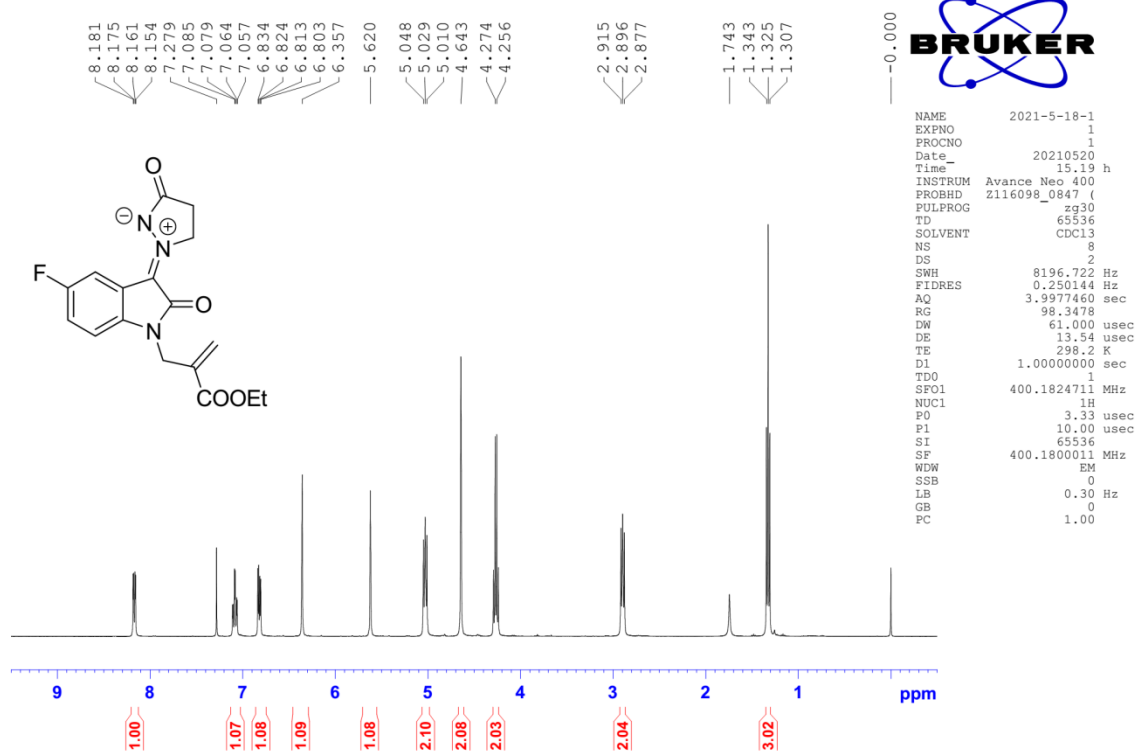
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3b

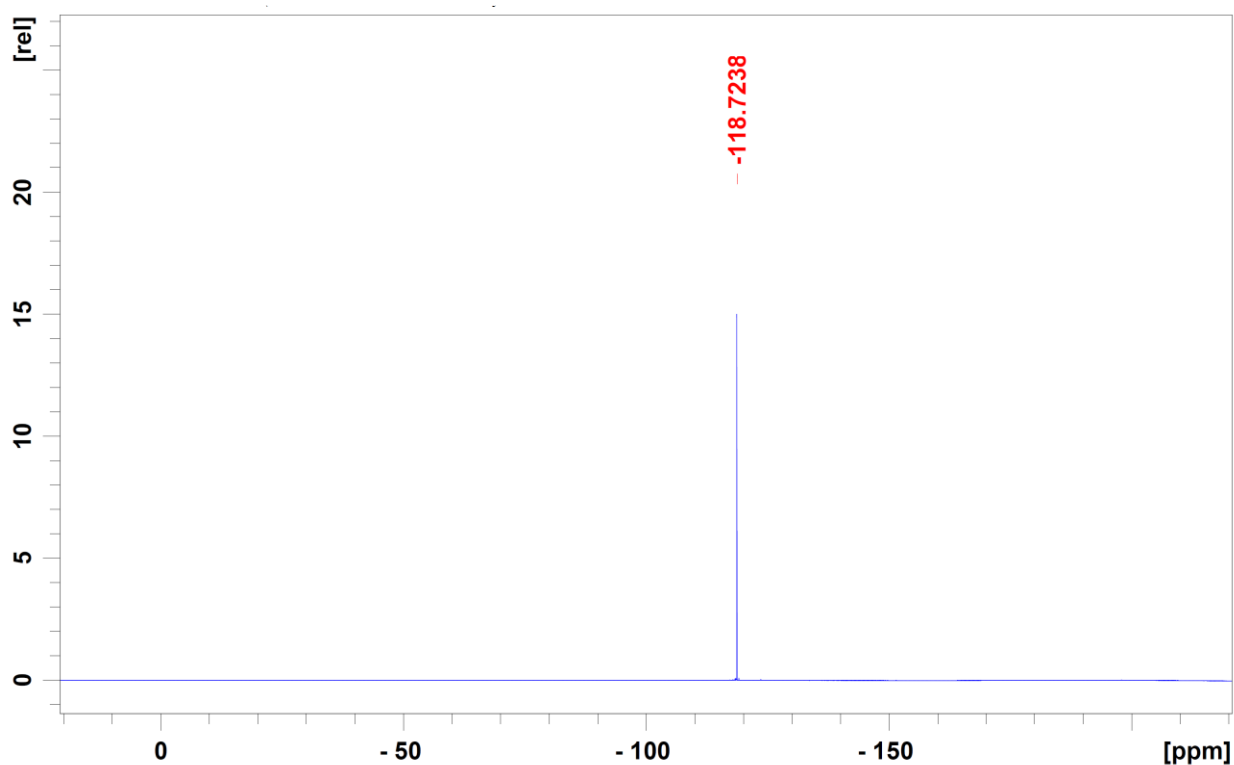


# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3c

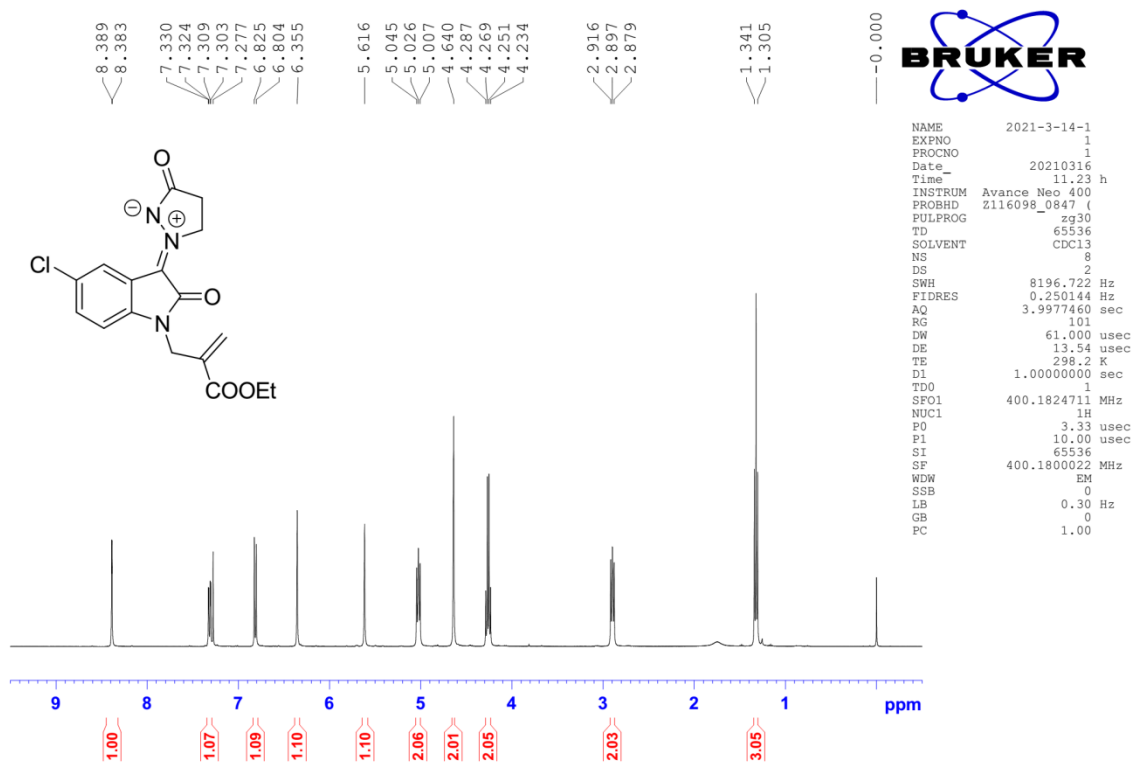


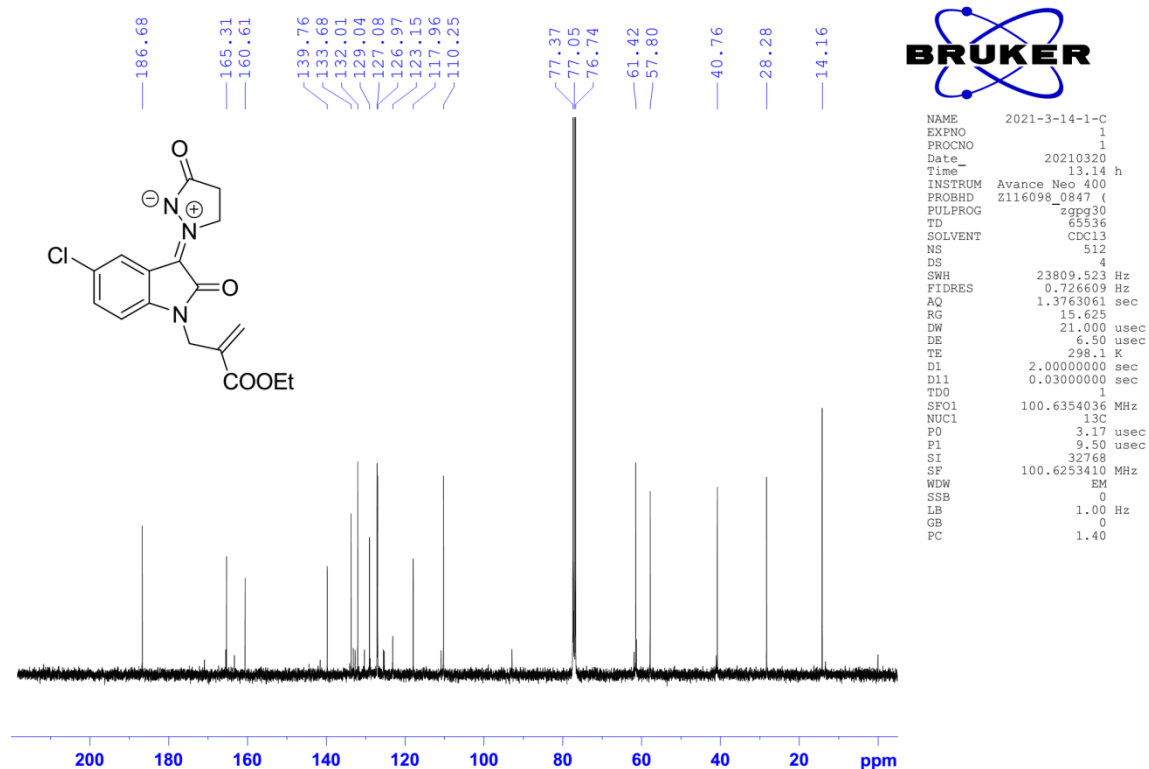
$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR Spectra for Compound **3d**



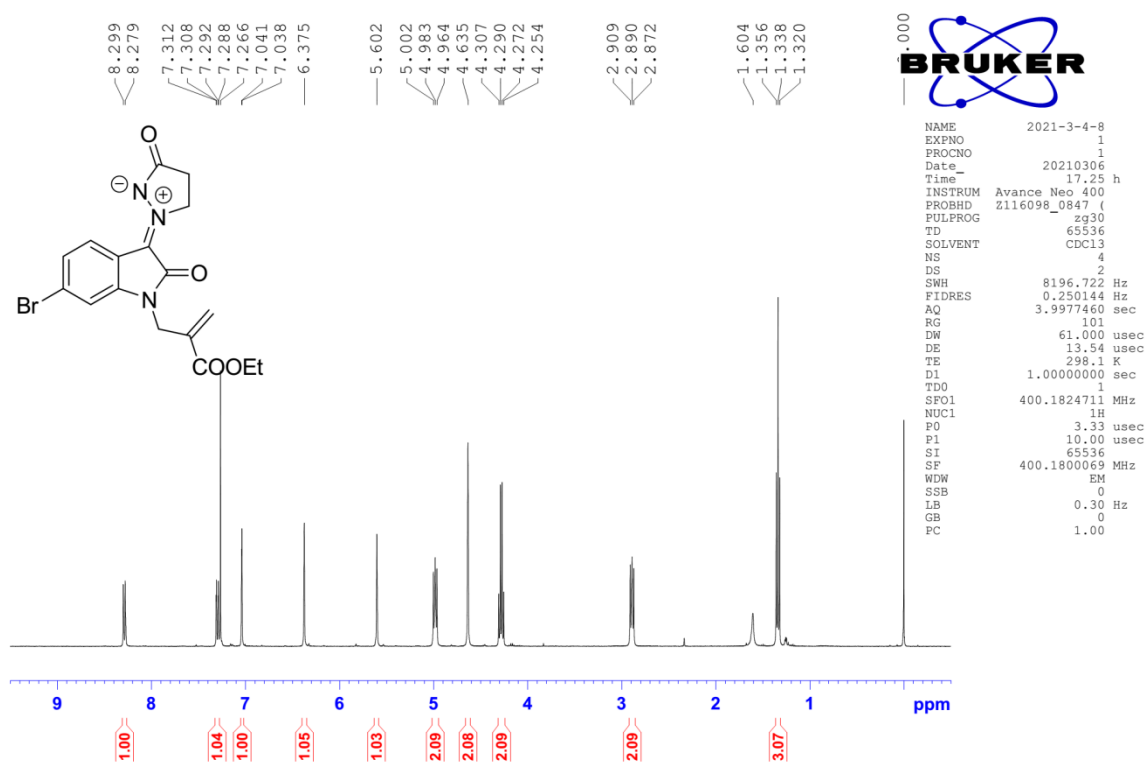


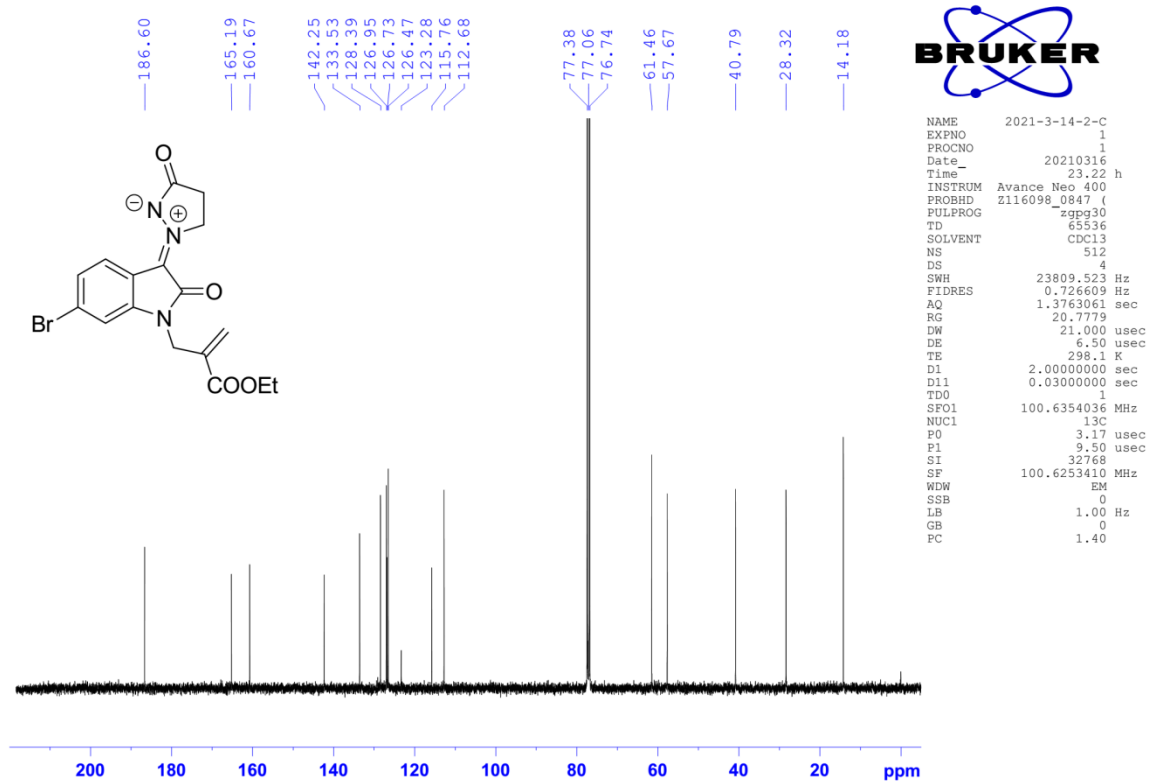
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound **3e**



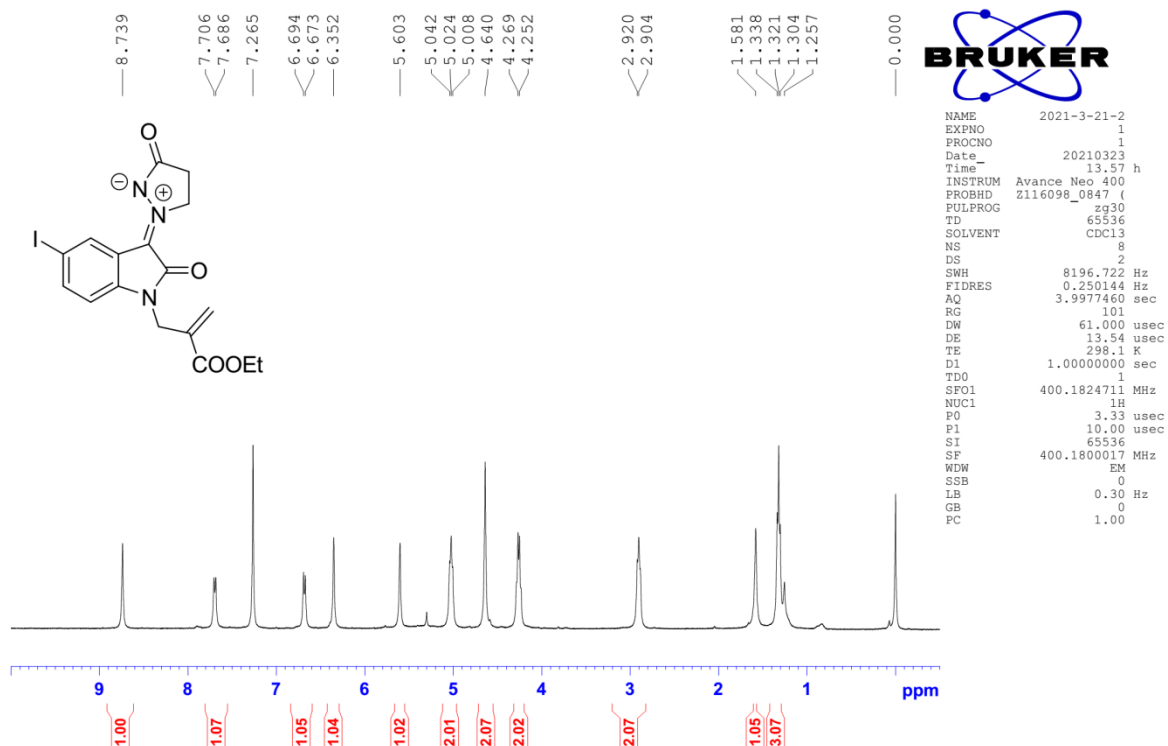


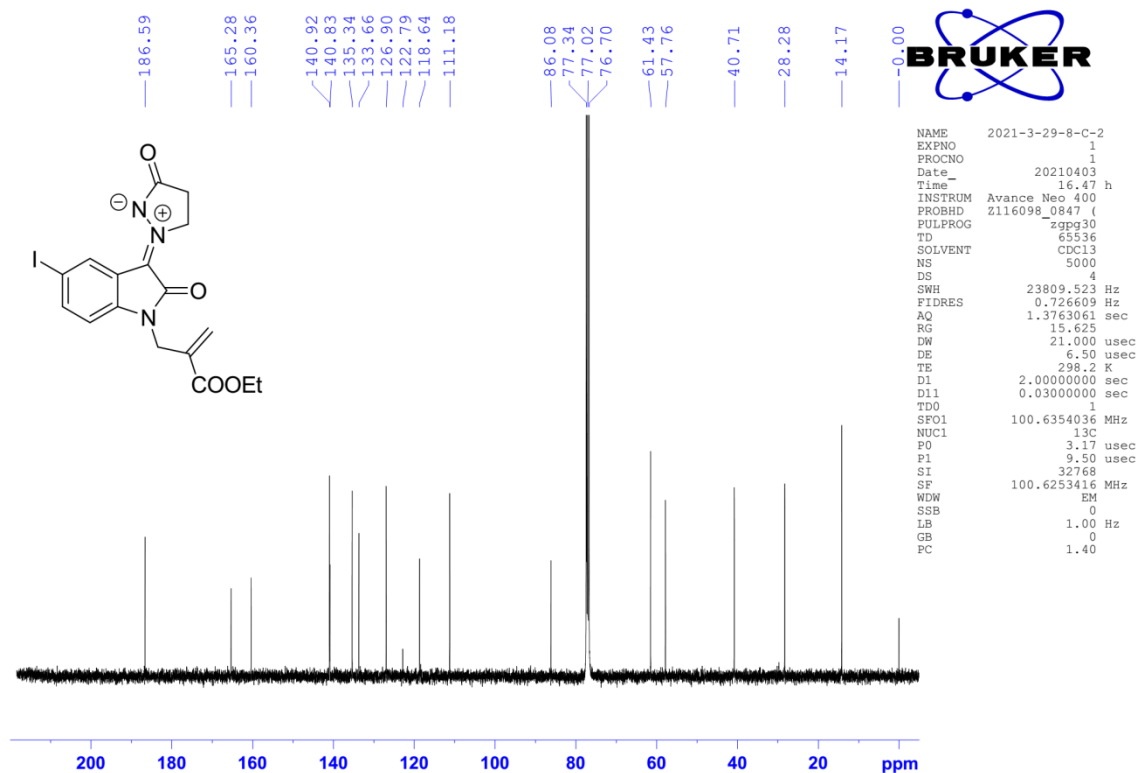
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3f



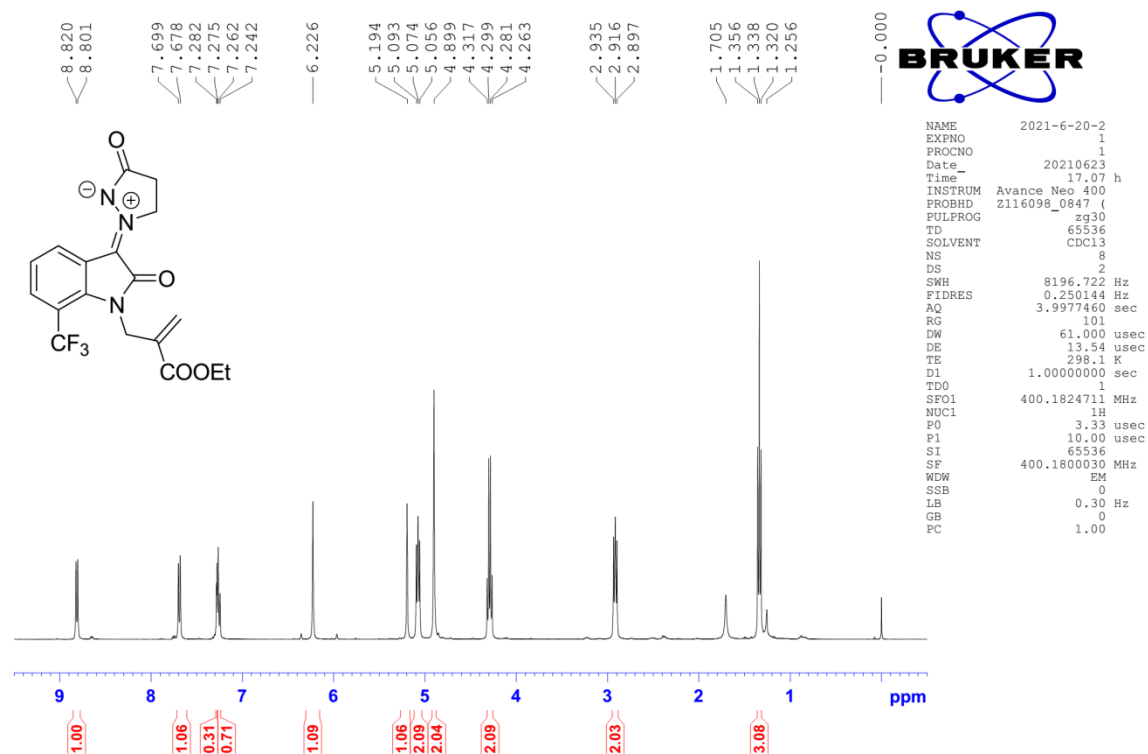


<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3g

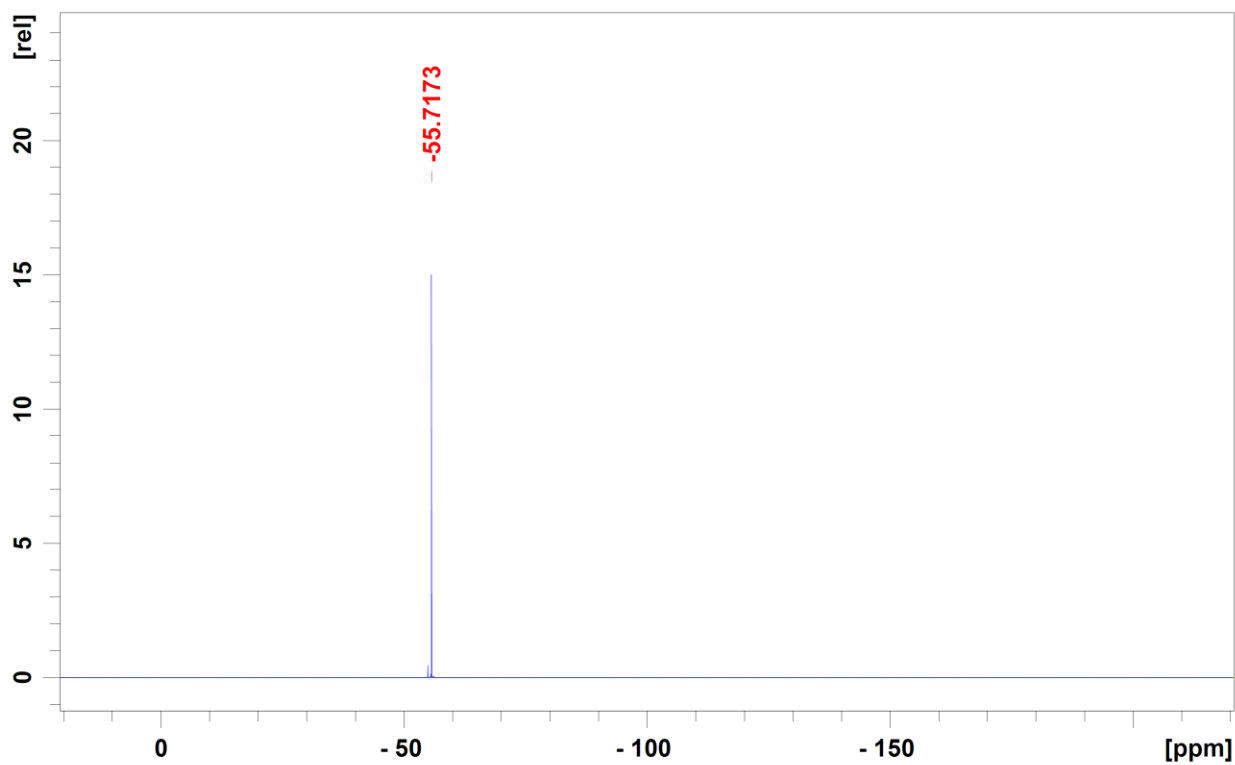
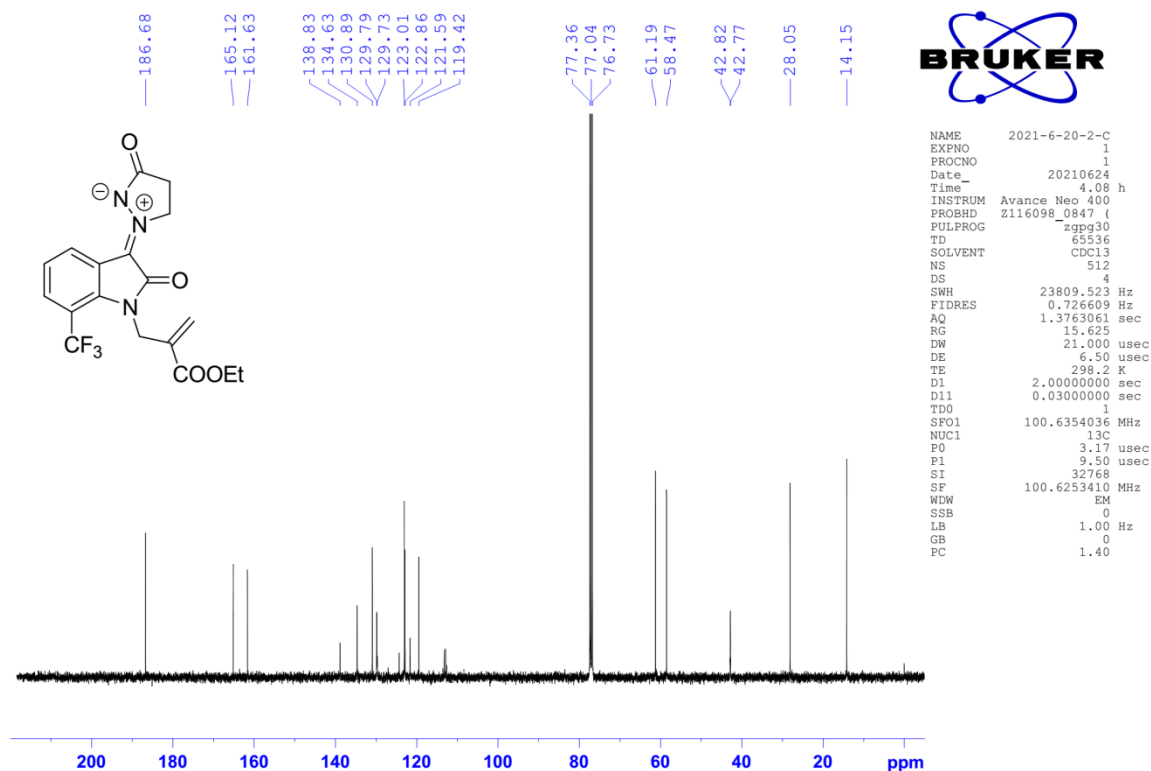




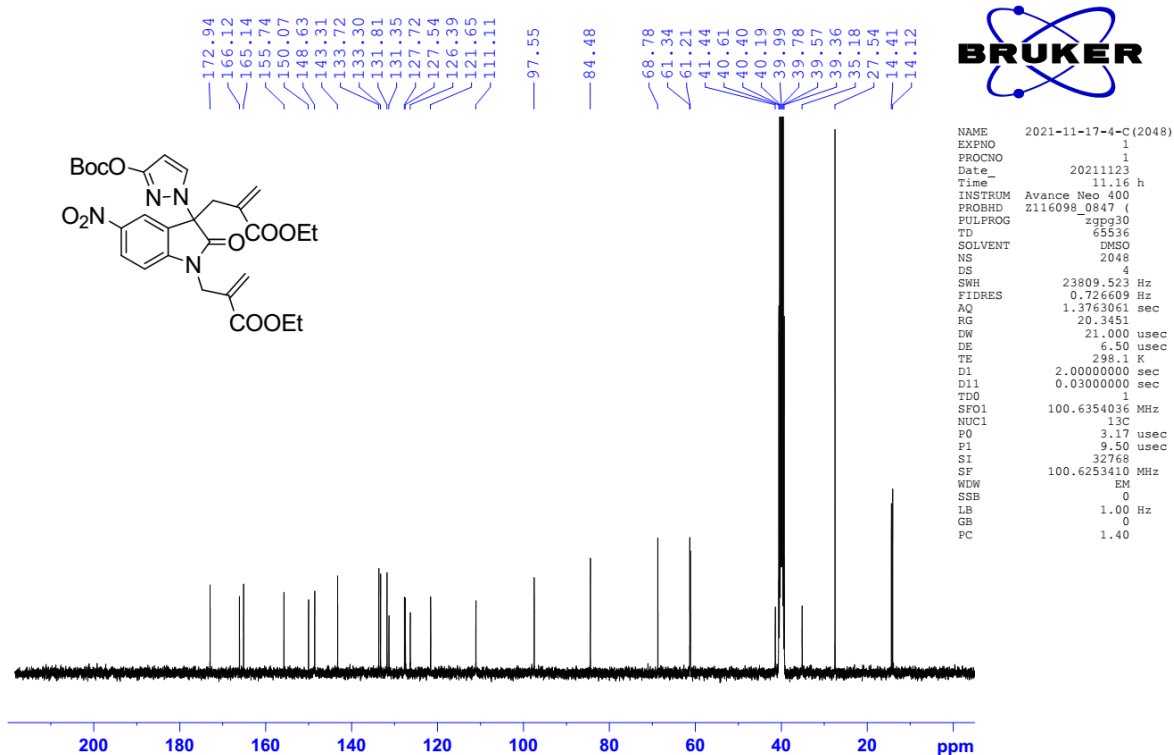
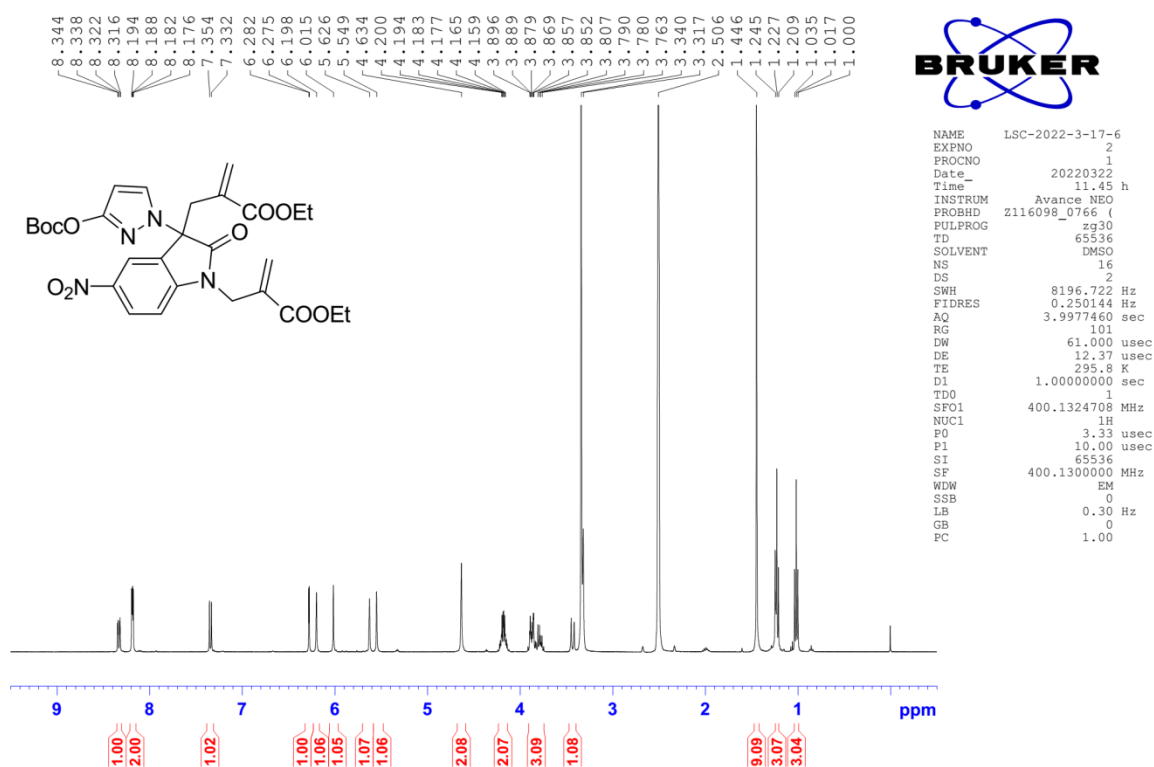
<sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F NMR Spectra for Compound 3h



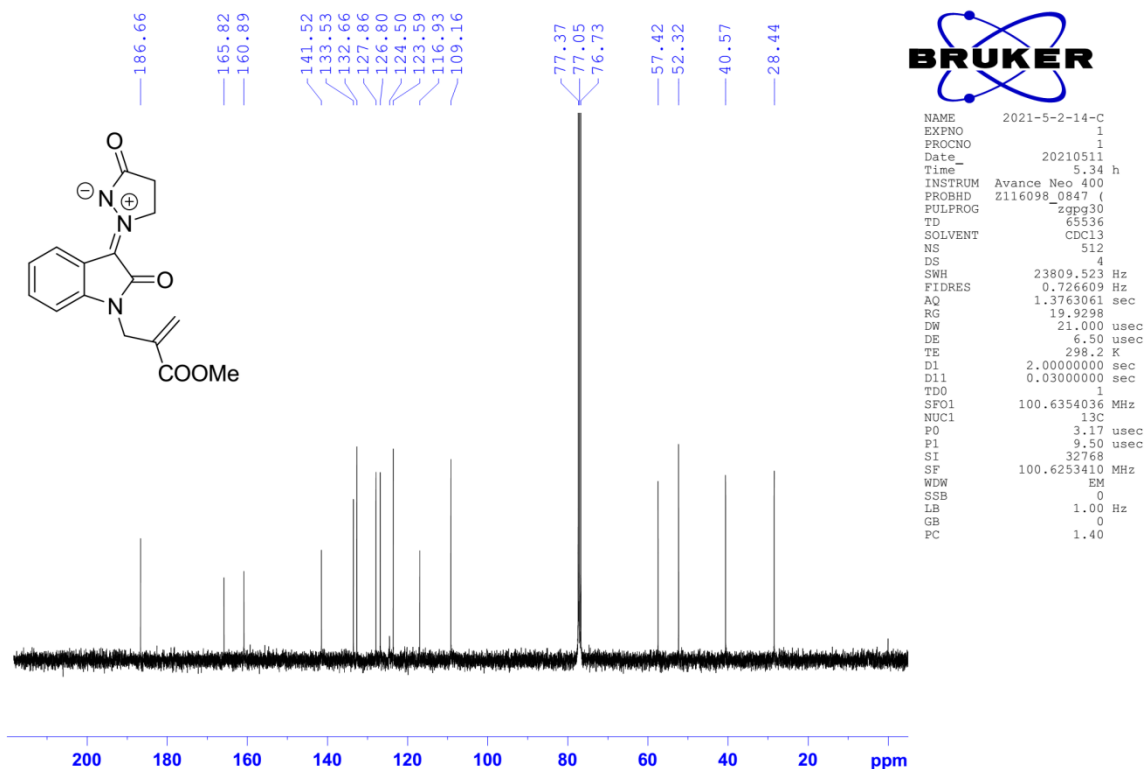
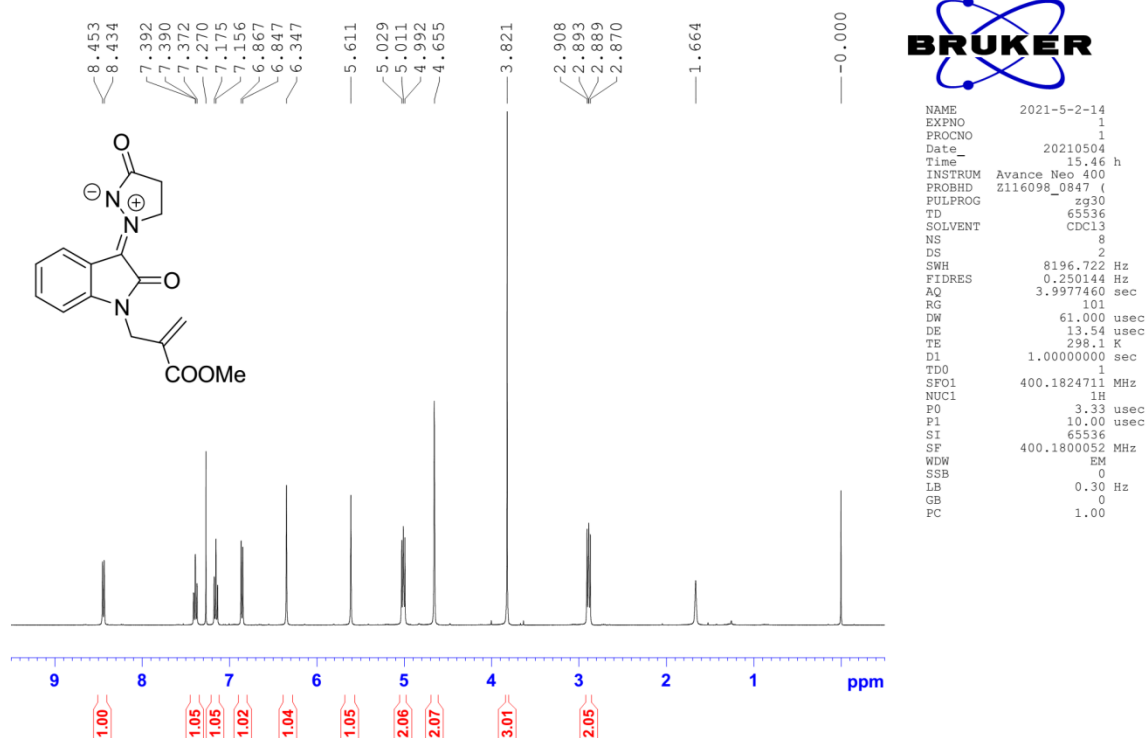




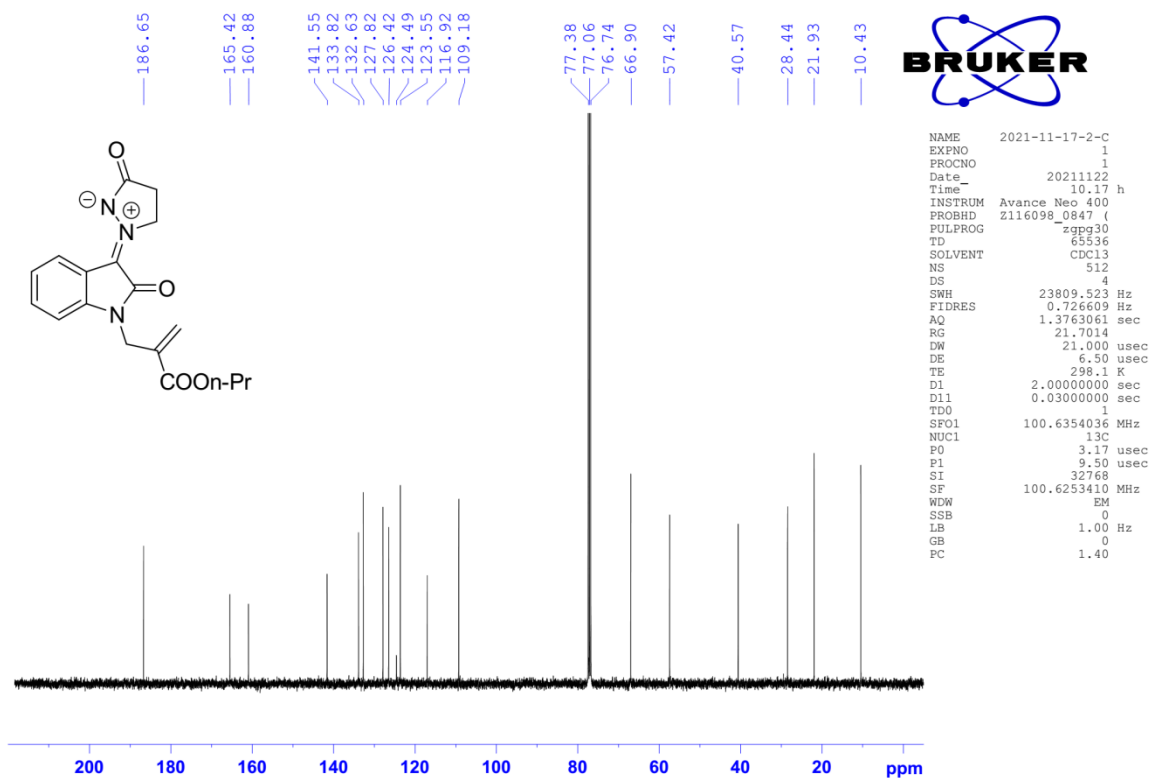
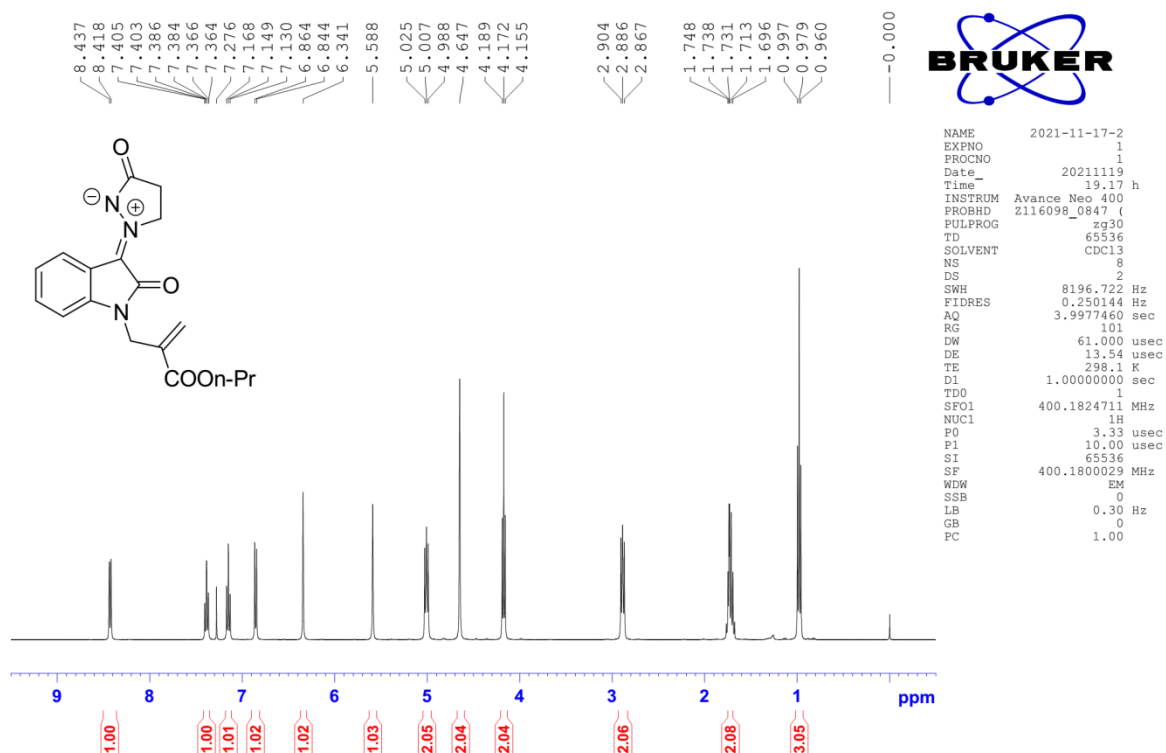
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3'i



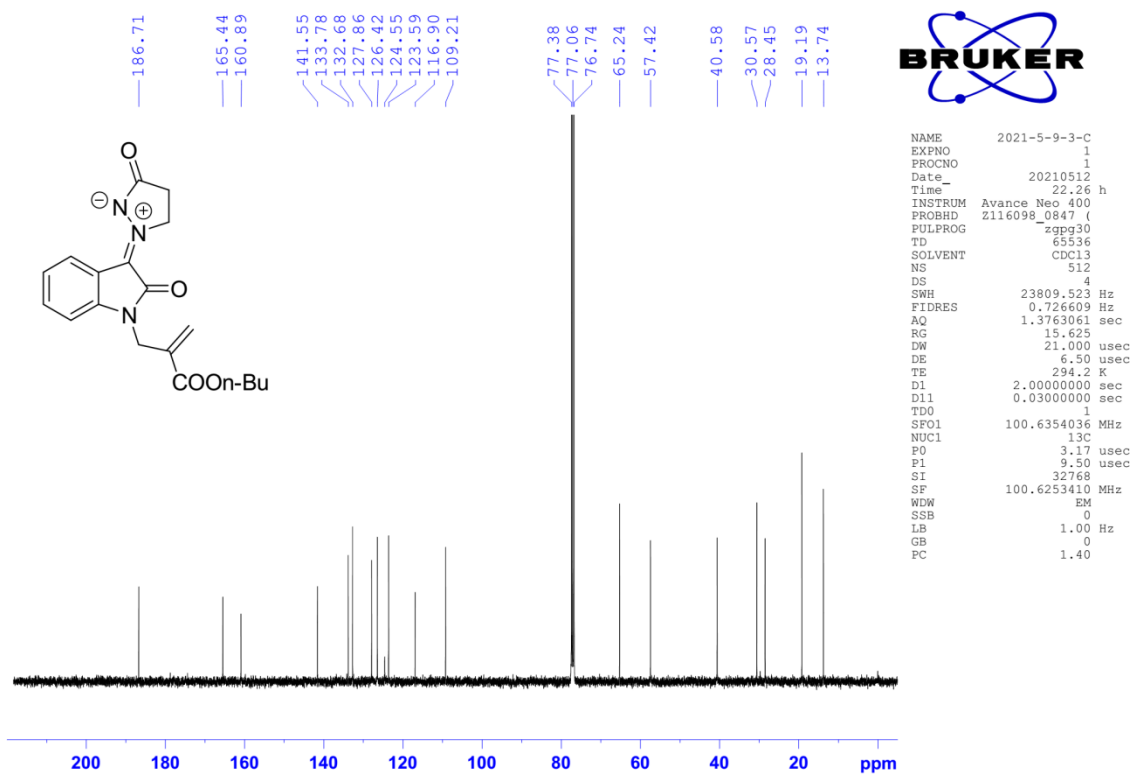
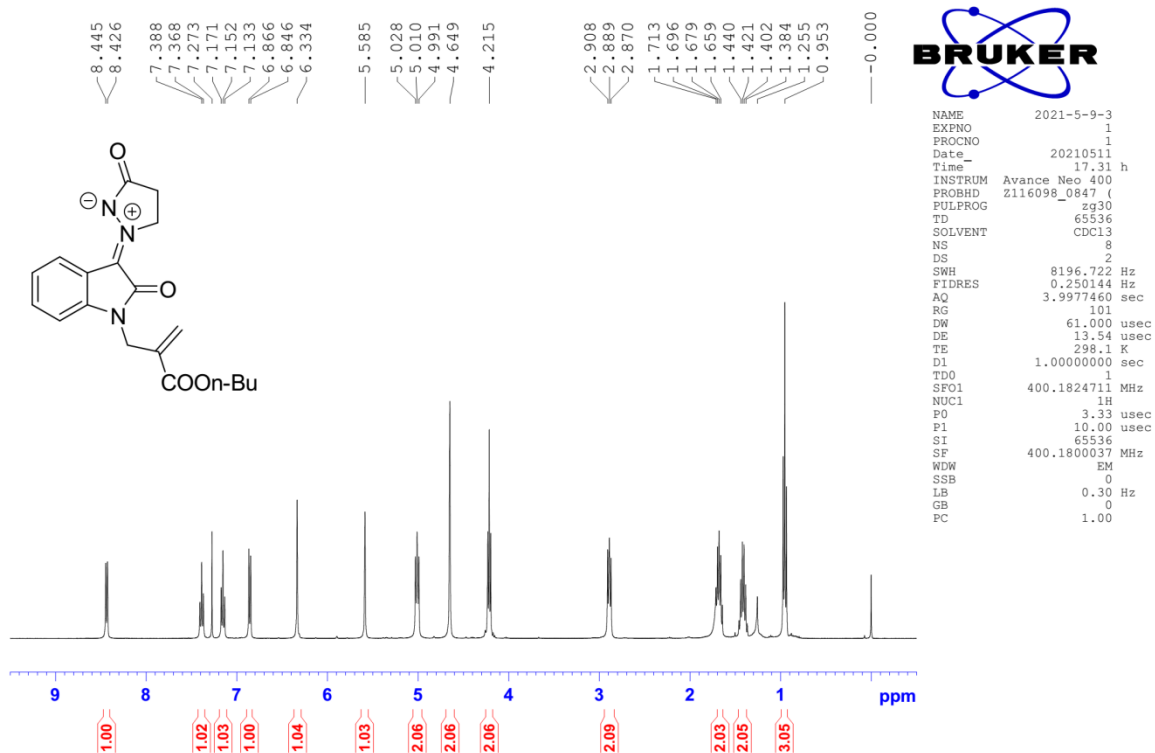
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3j



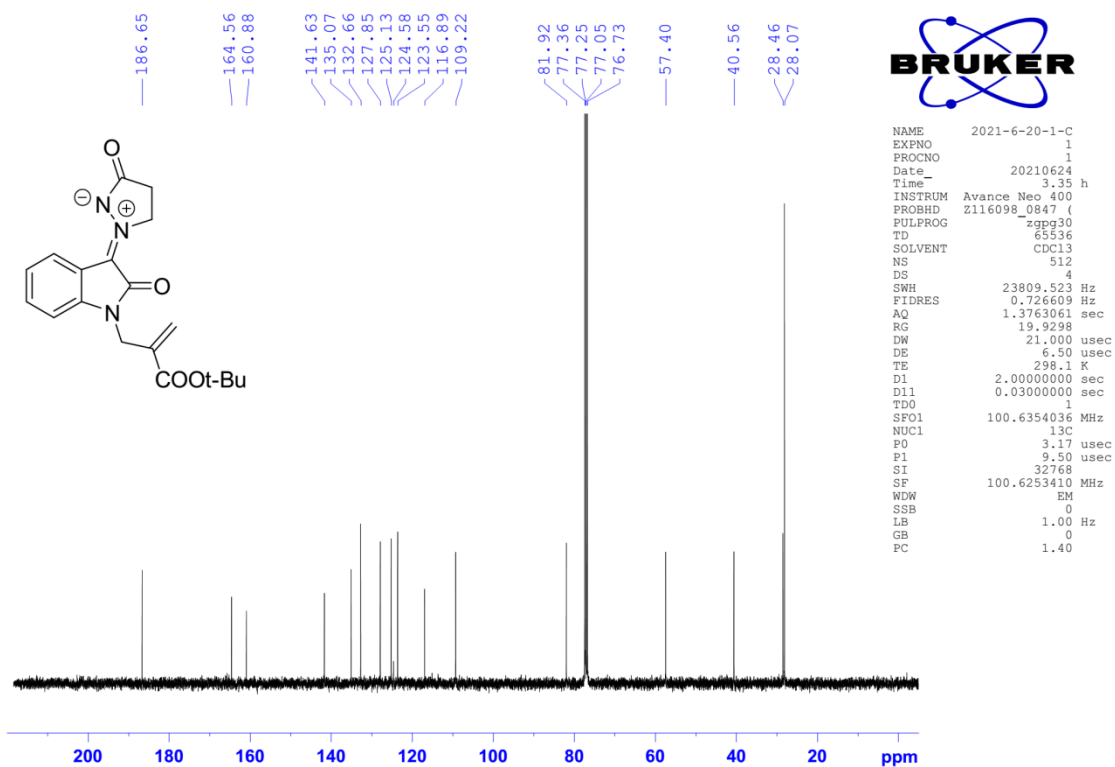
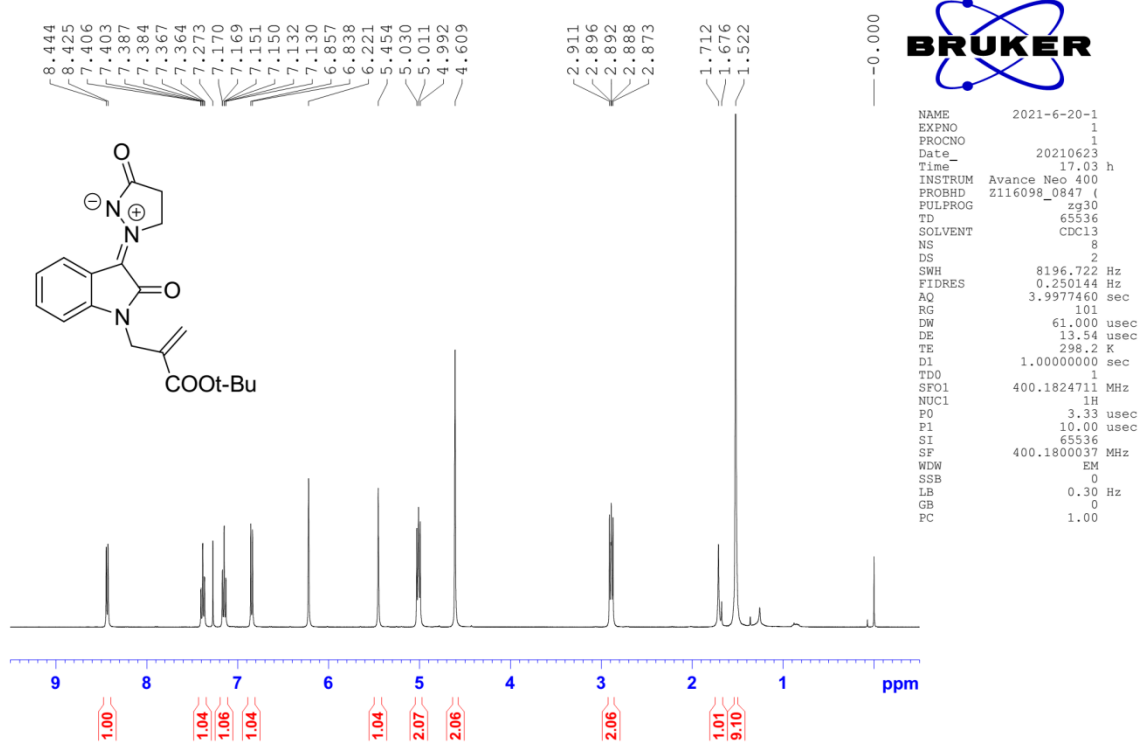
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3k



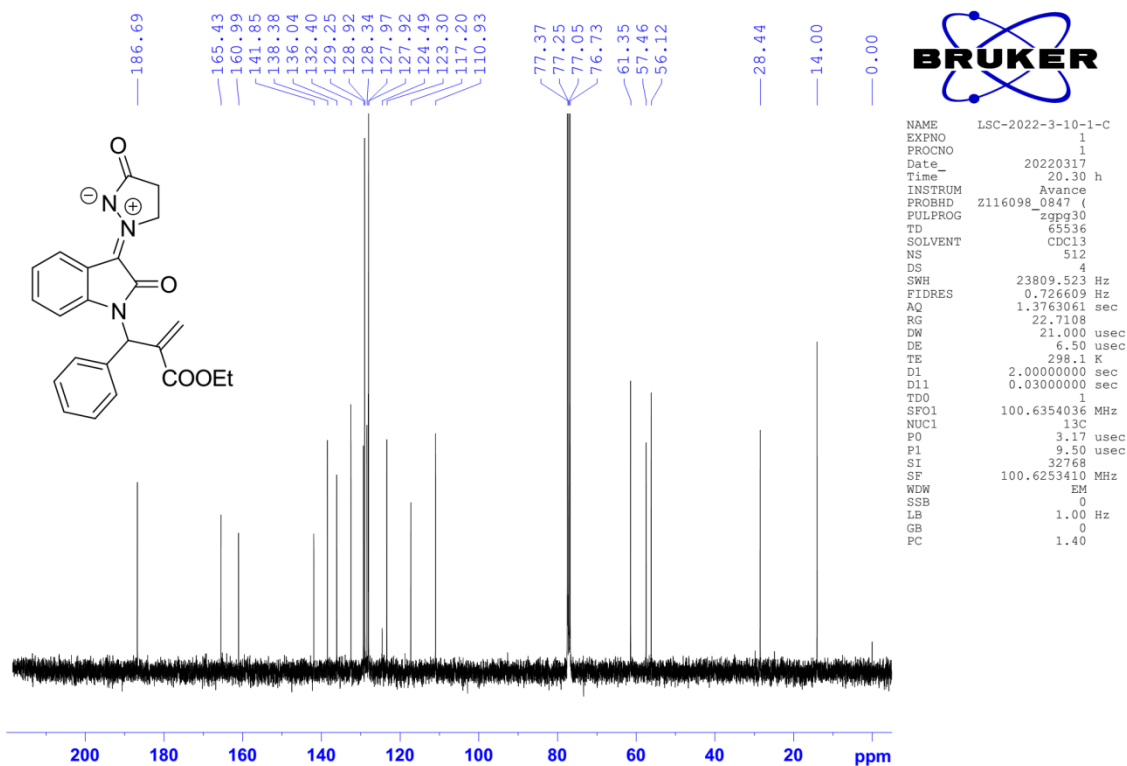
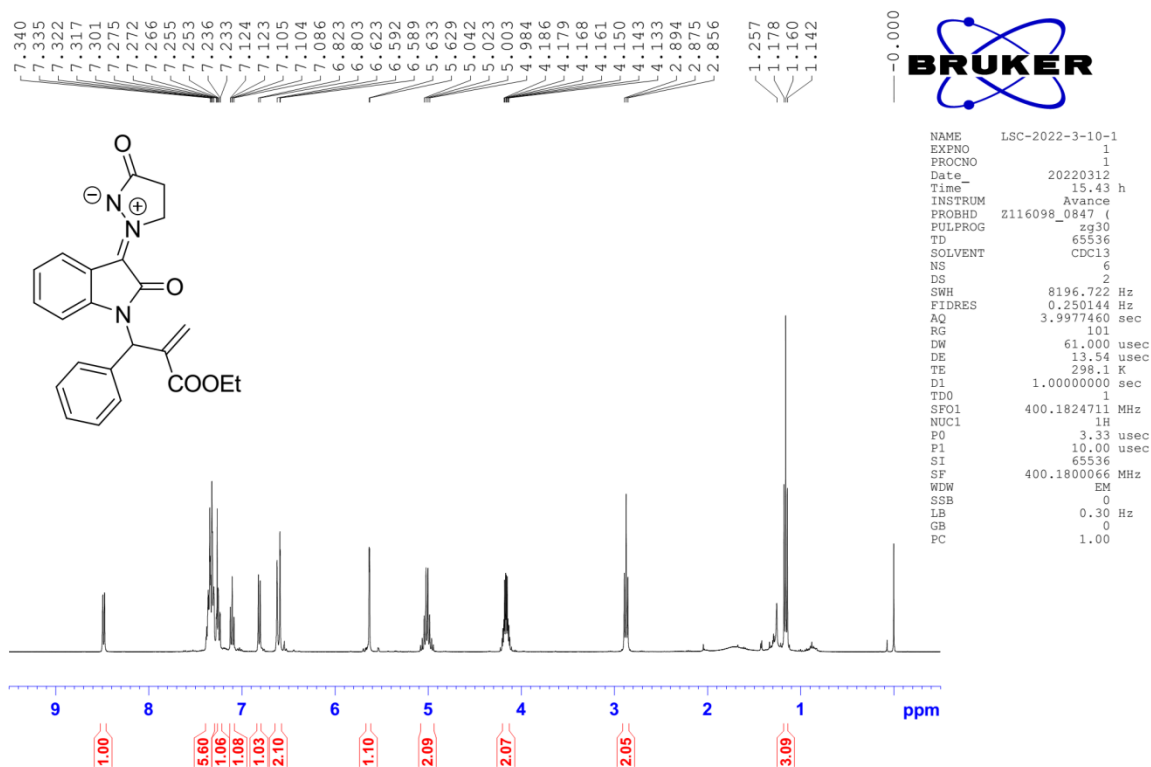
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 31



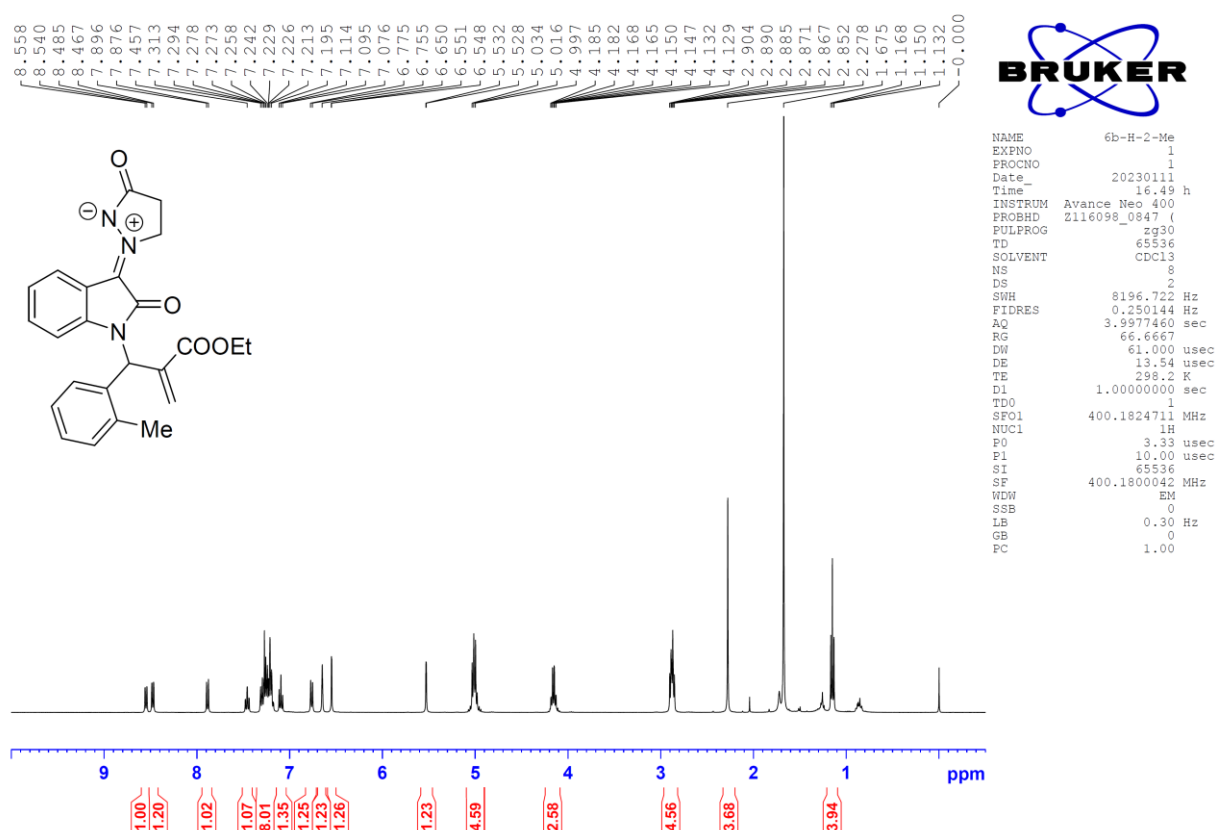
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 3m



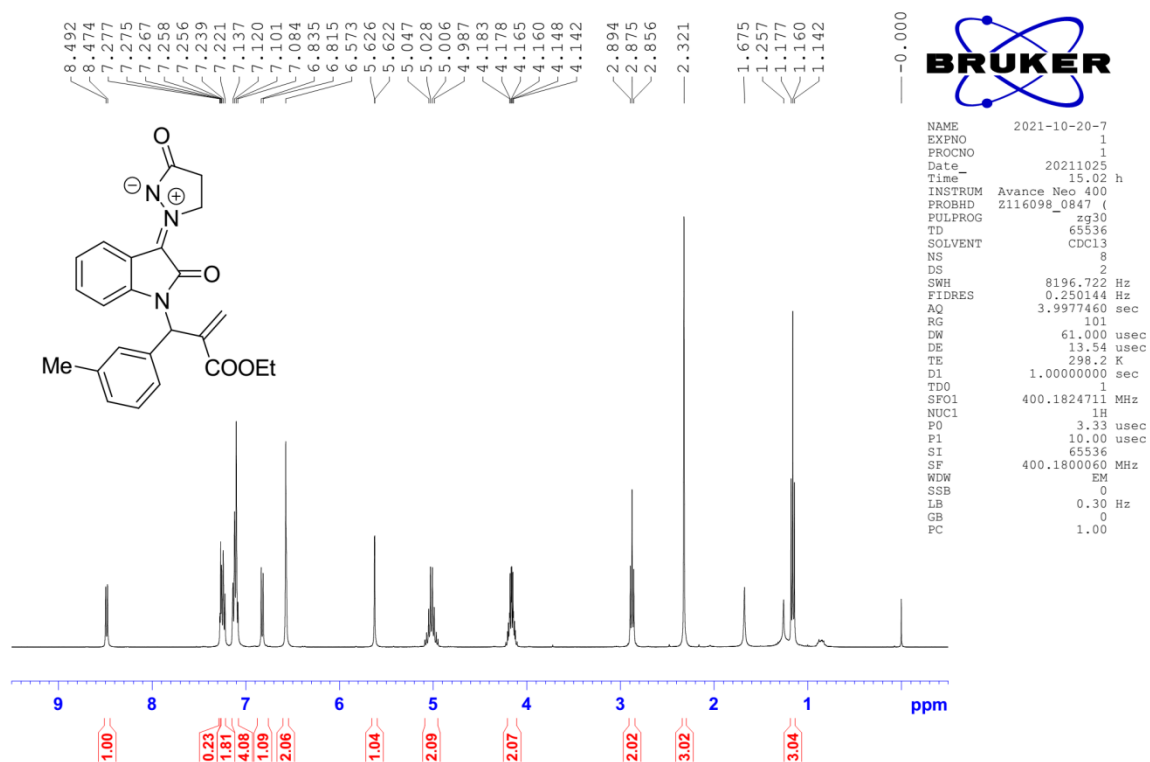
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6a



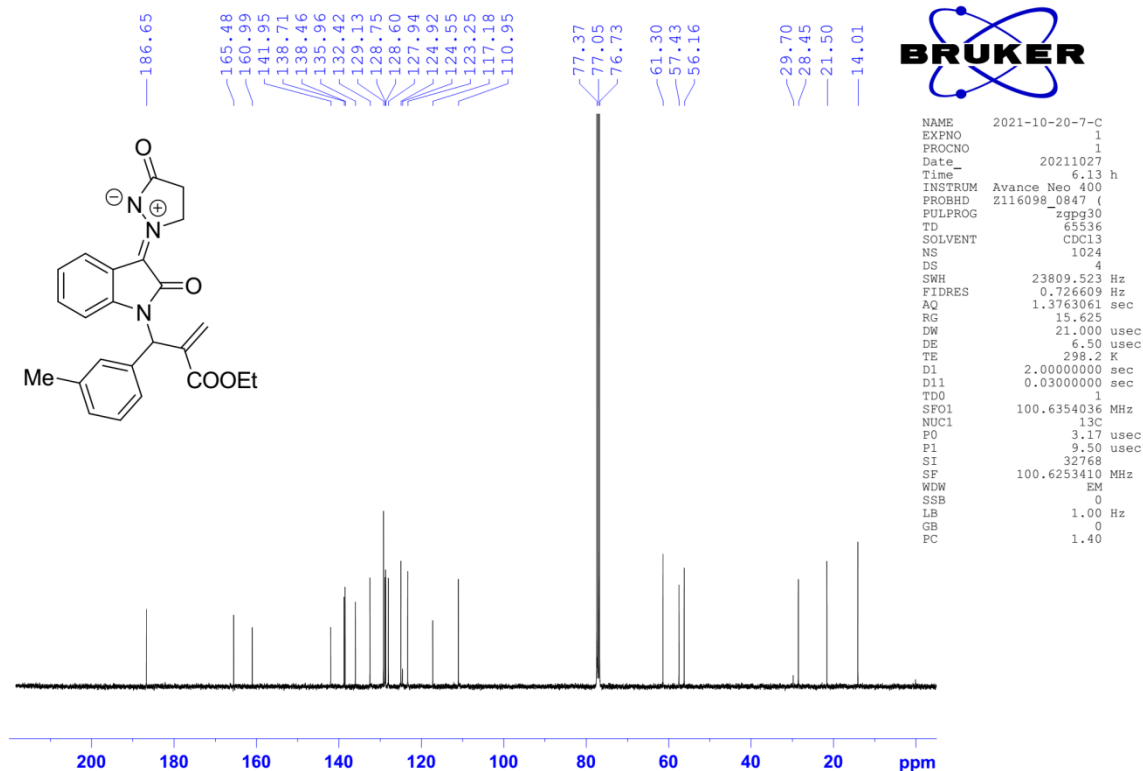
<sup>1</sup>H NMR Spectra for Compound **6b** (mixed with an inseparable by-product)



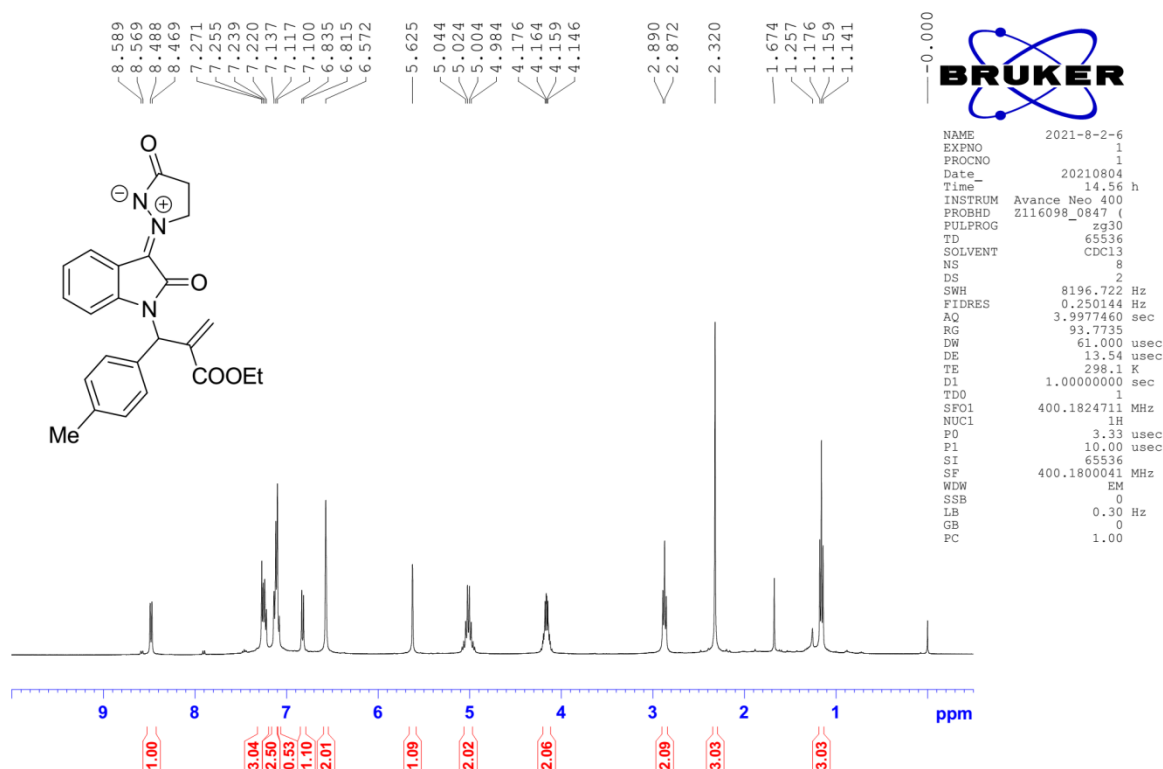
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound **6c**

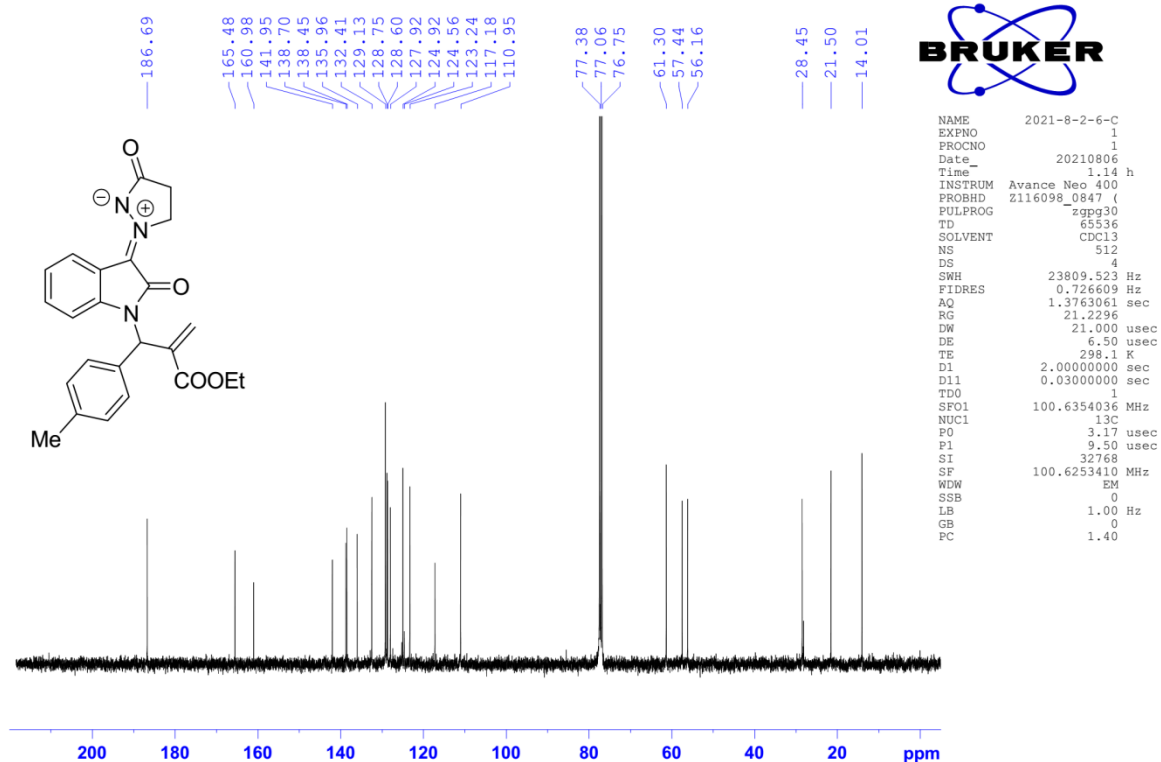




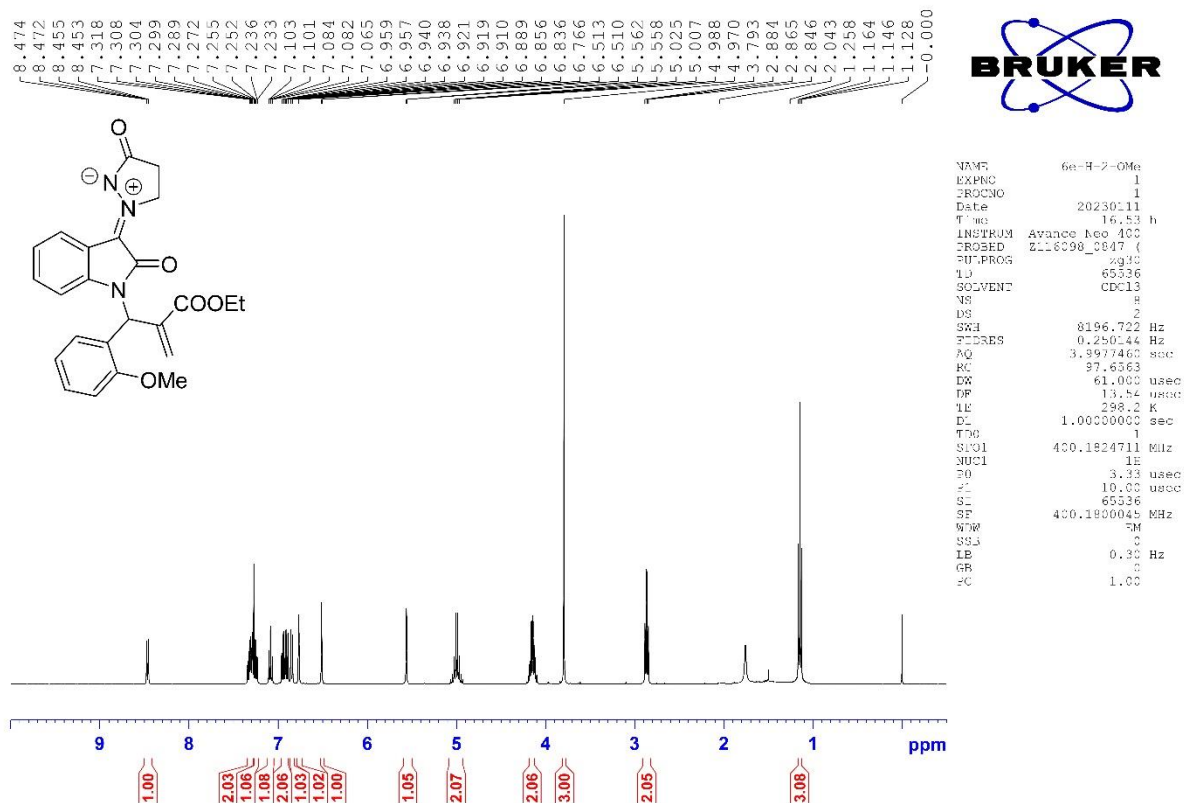


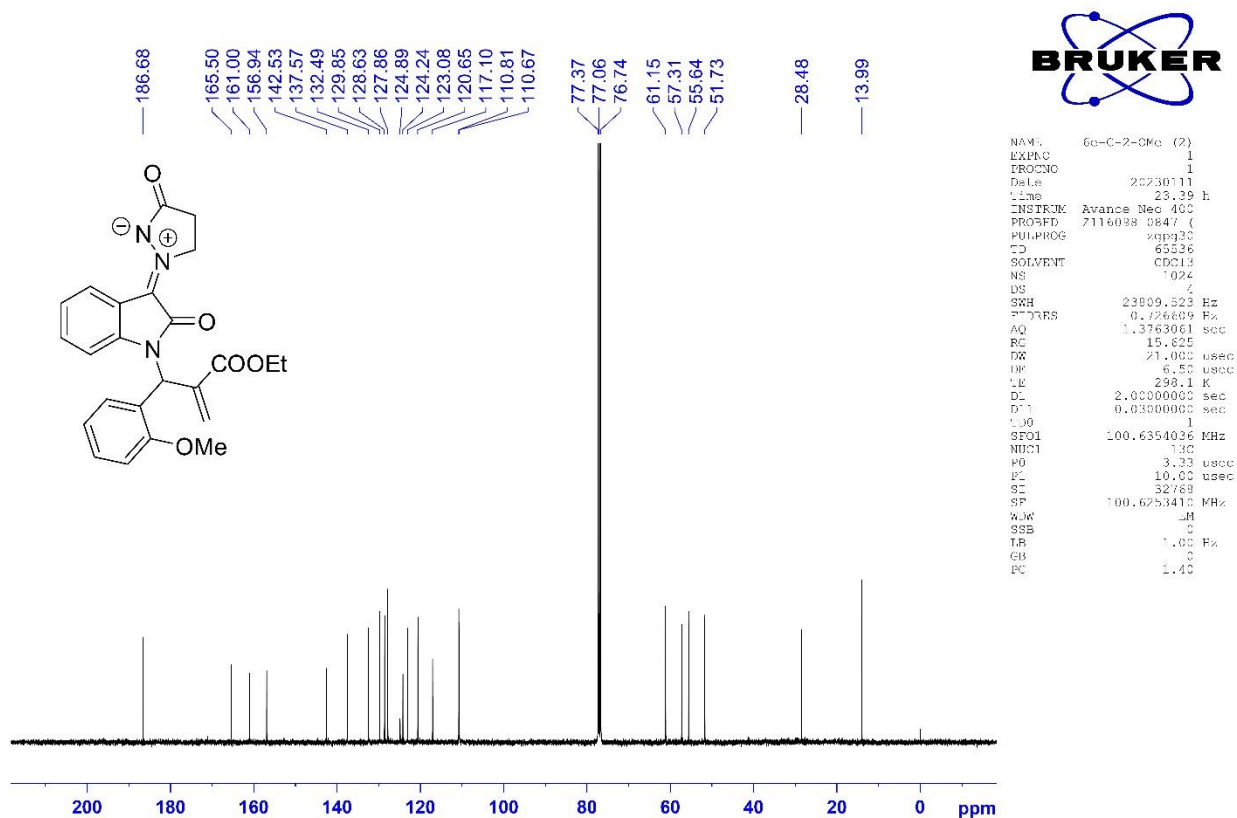
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6d



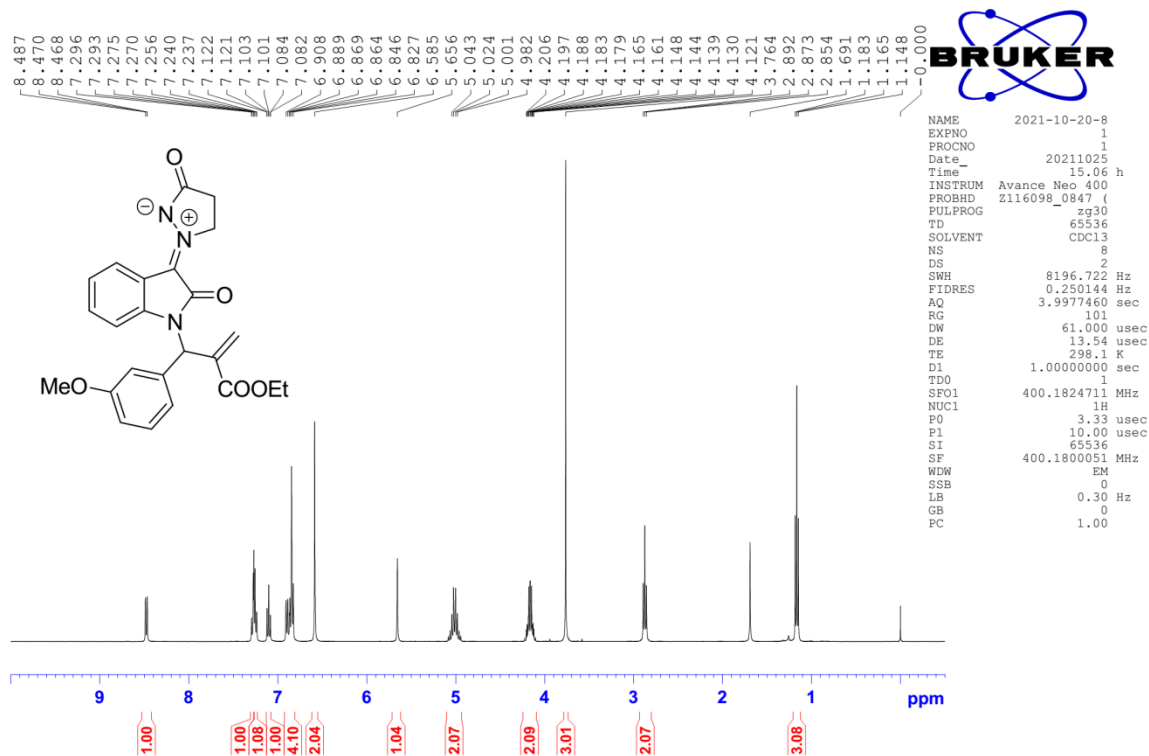


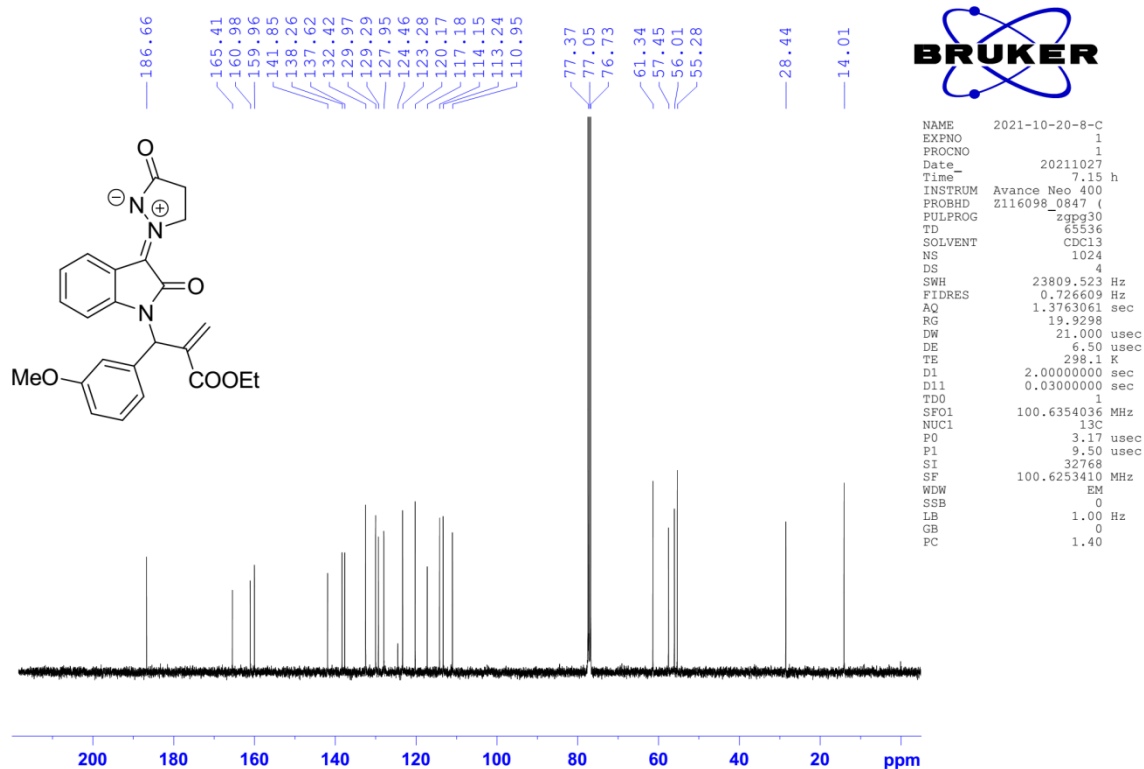
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6e



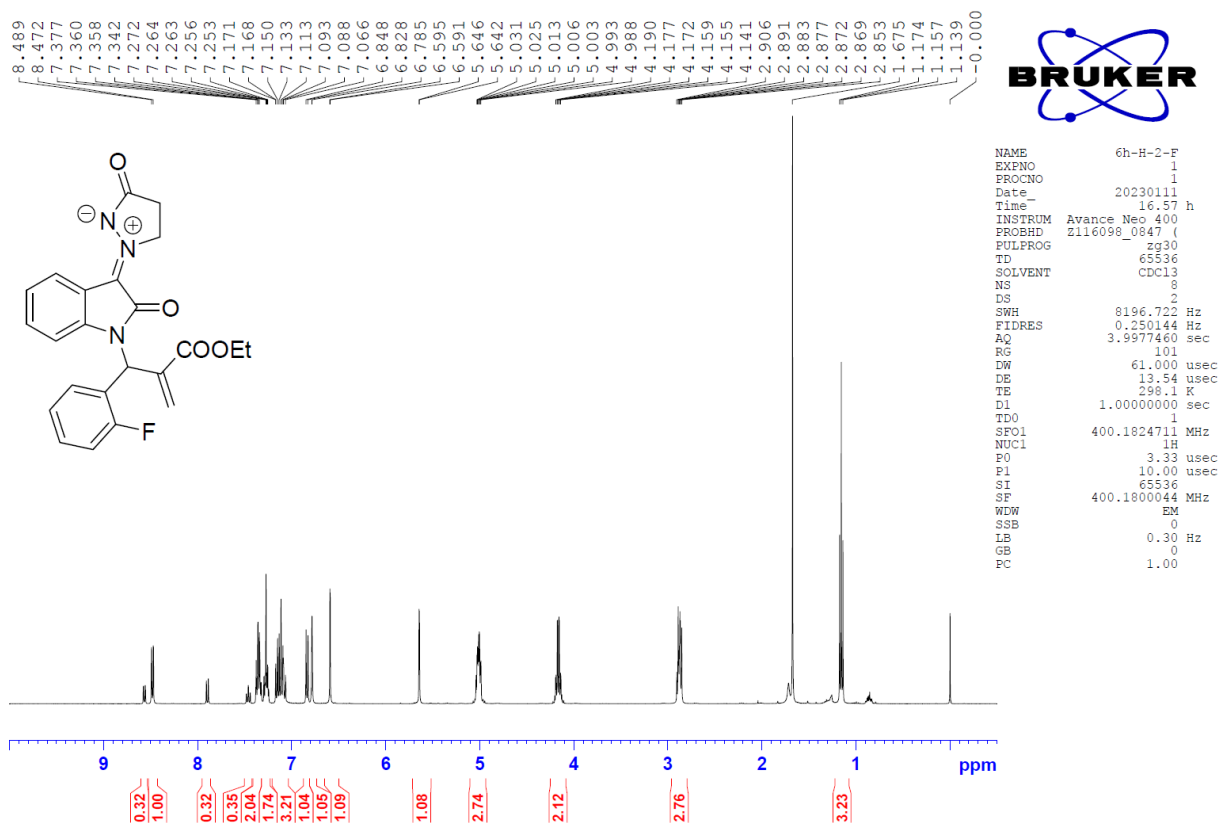


<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6f

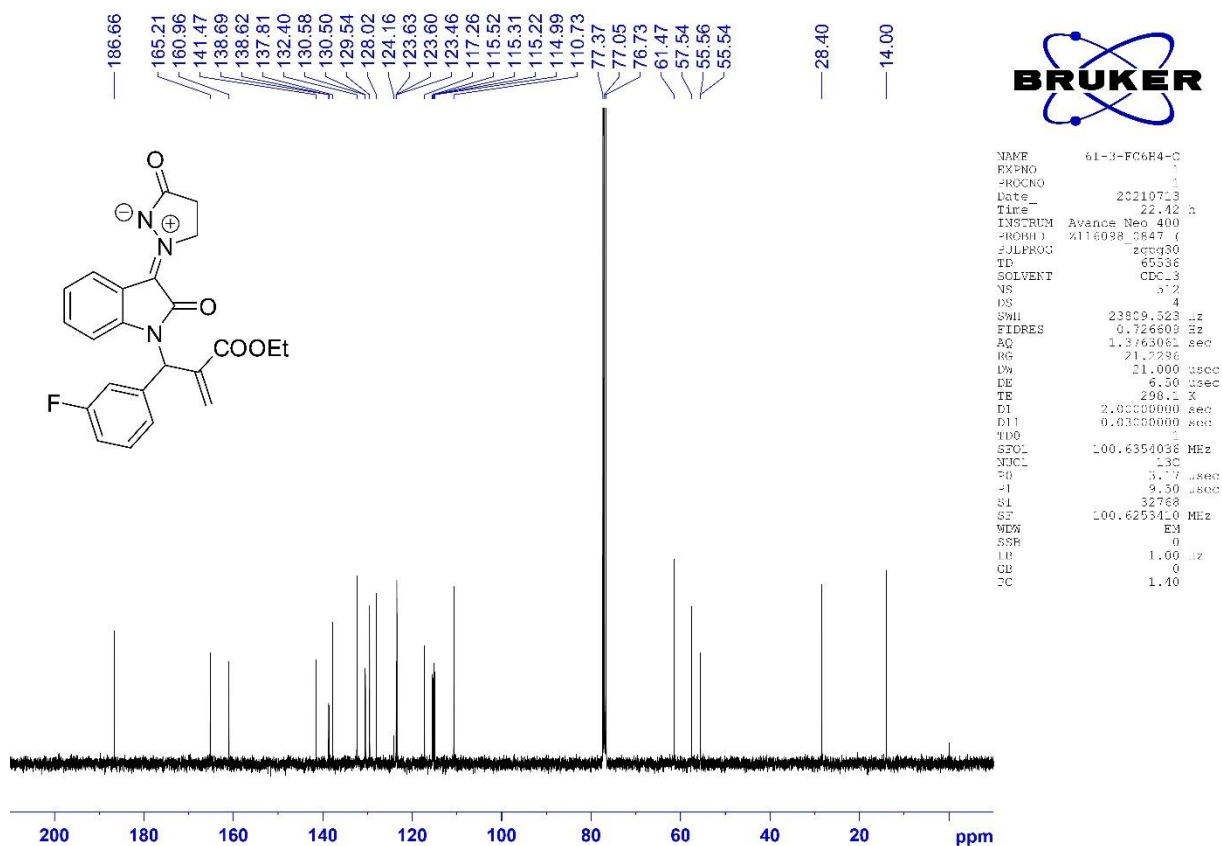
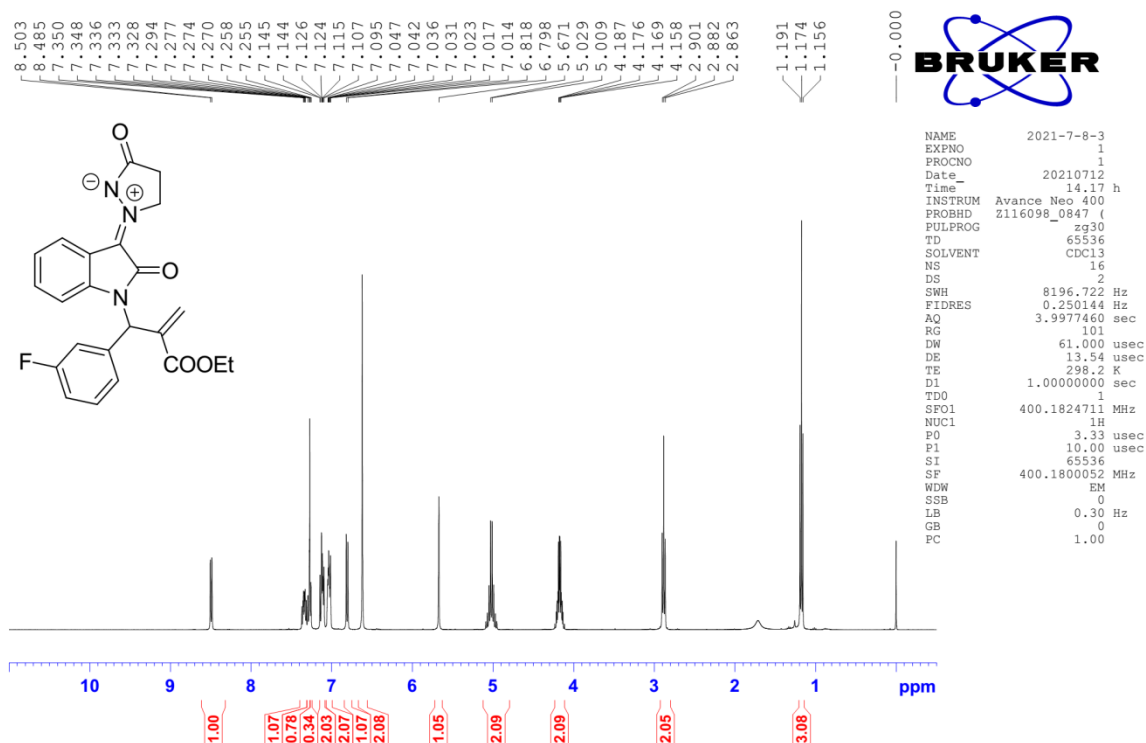


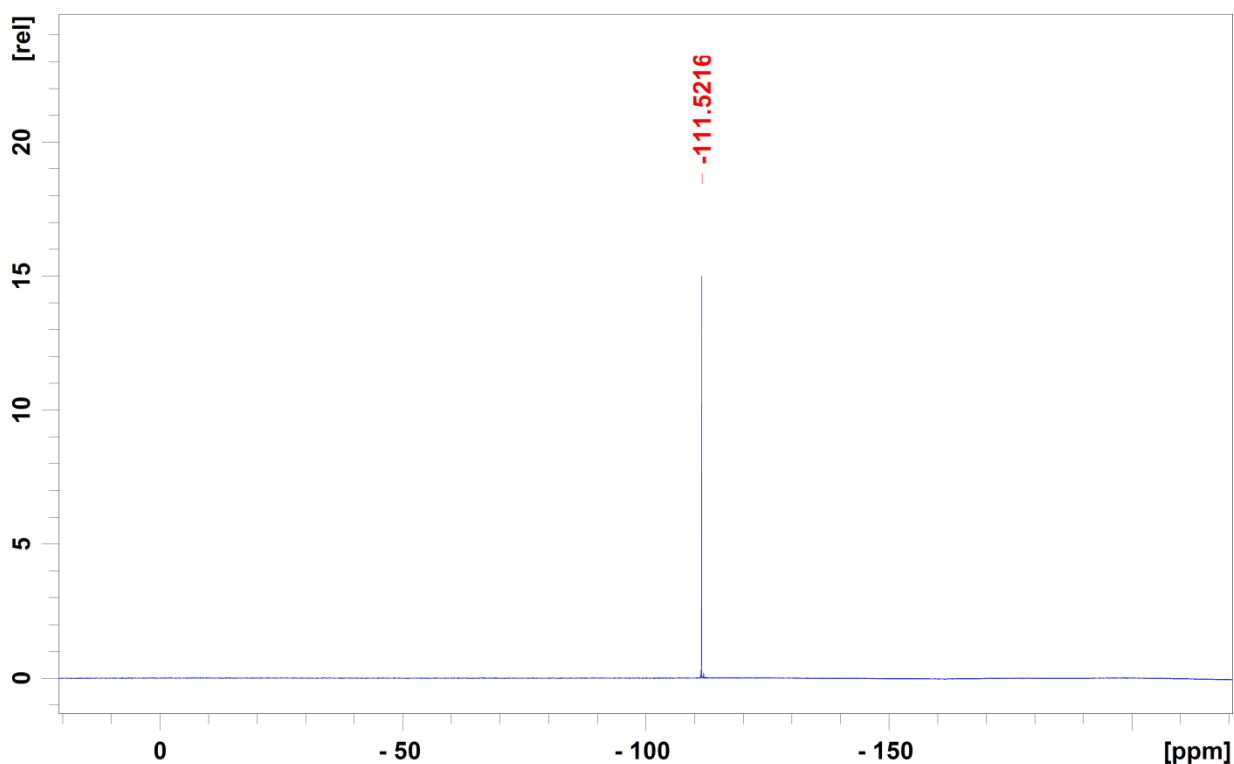


<sup>1</sup>H NMR Spectra for Compound 6h (mixed with an inseparable by-product)

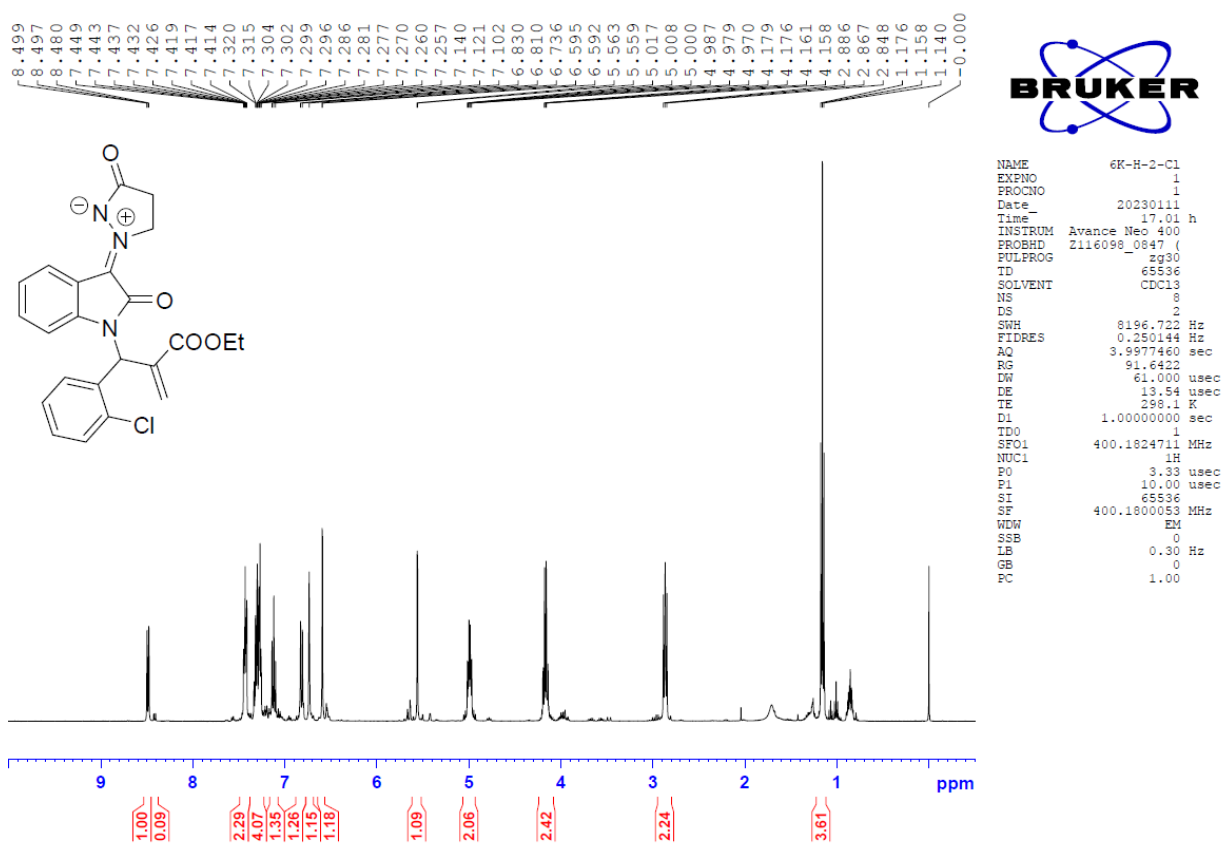


$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR Spectra for Compound **6i**

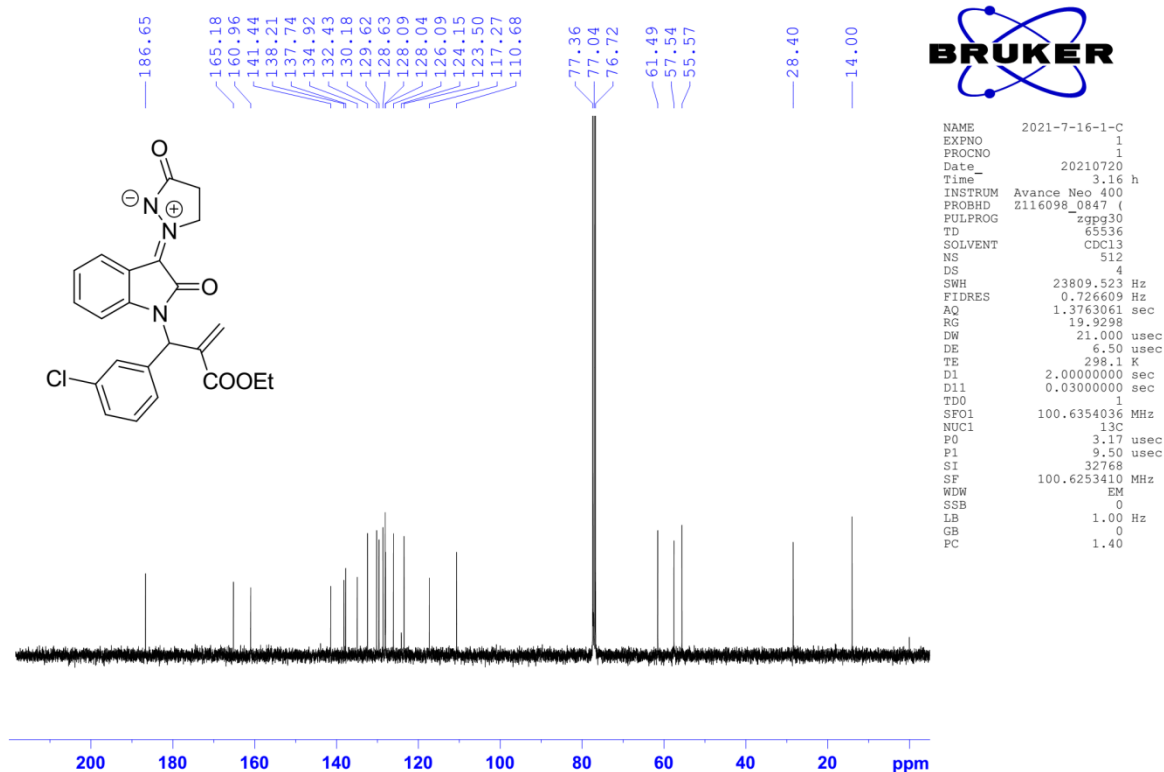
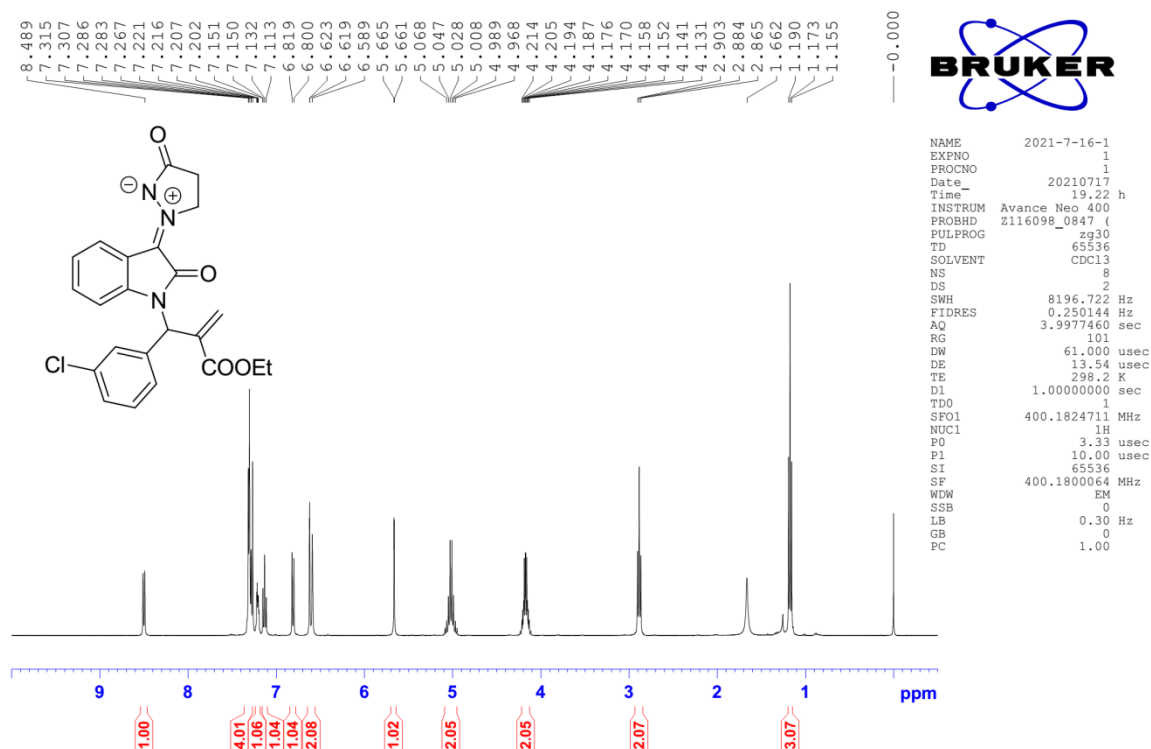




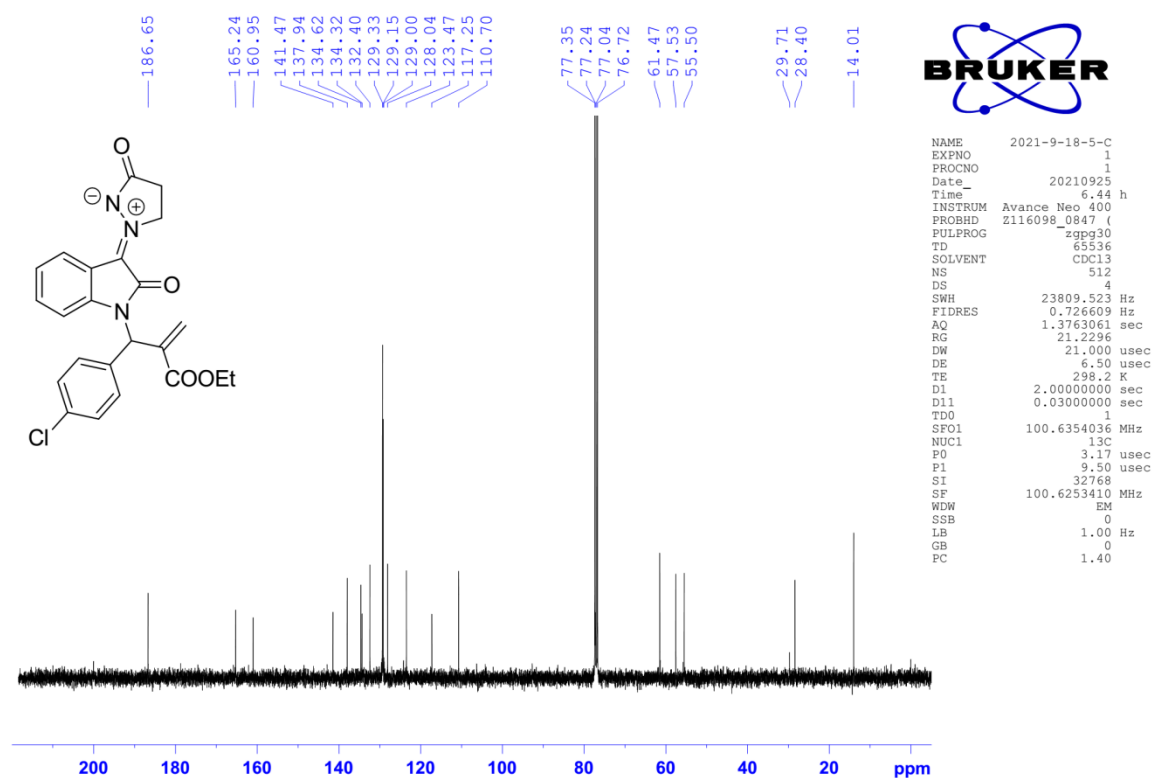
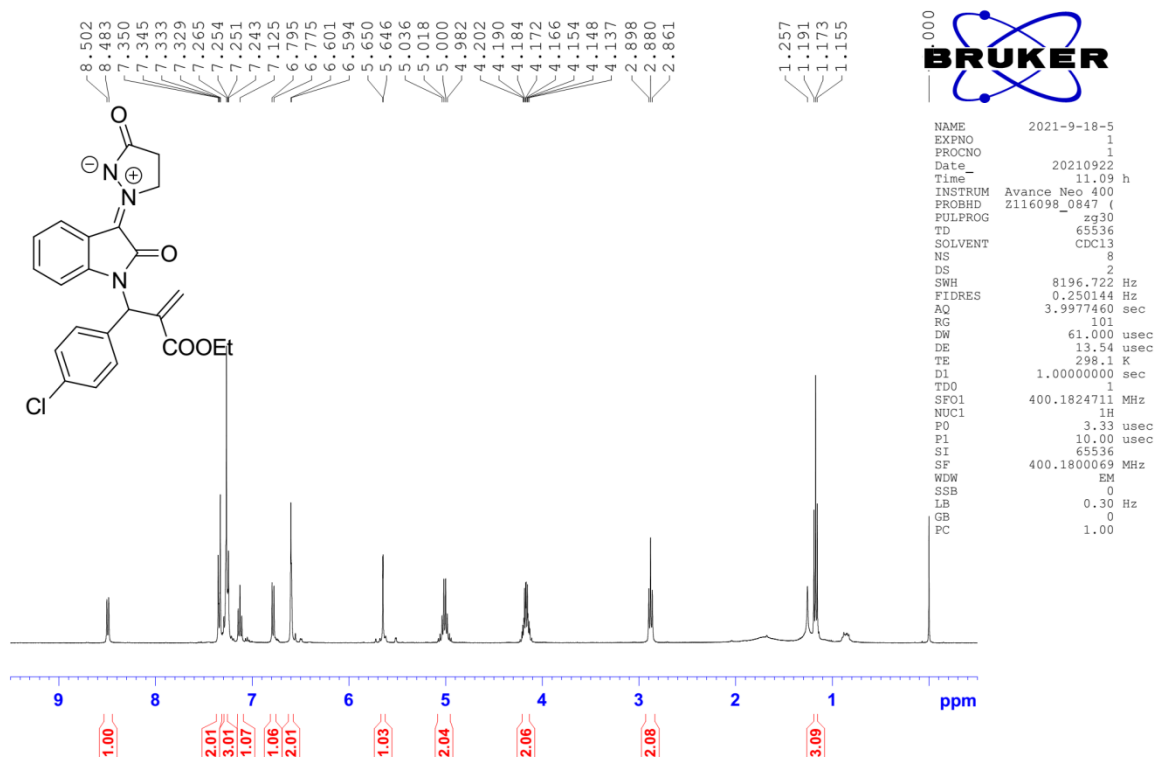
<sup>1</sup>H NMR Spectra for Compound 6k (mixed with an inseparable by-product)



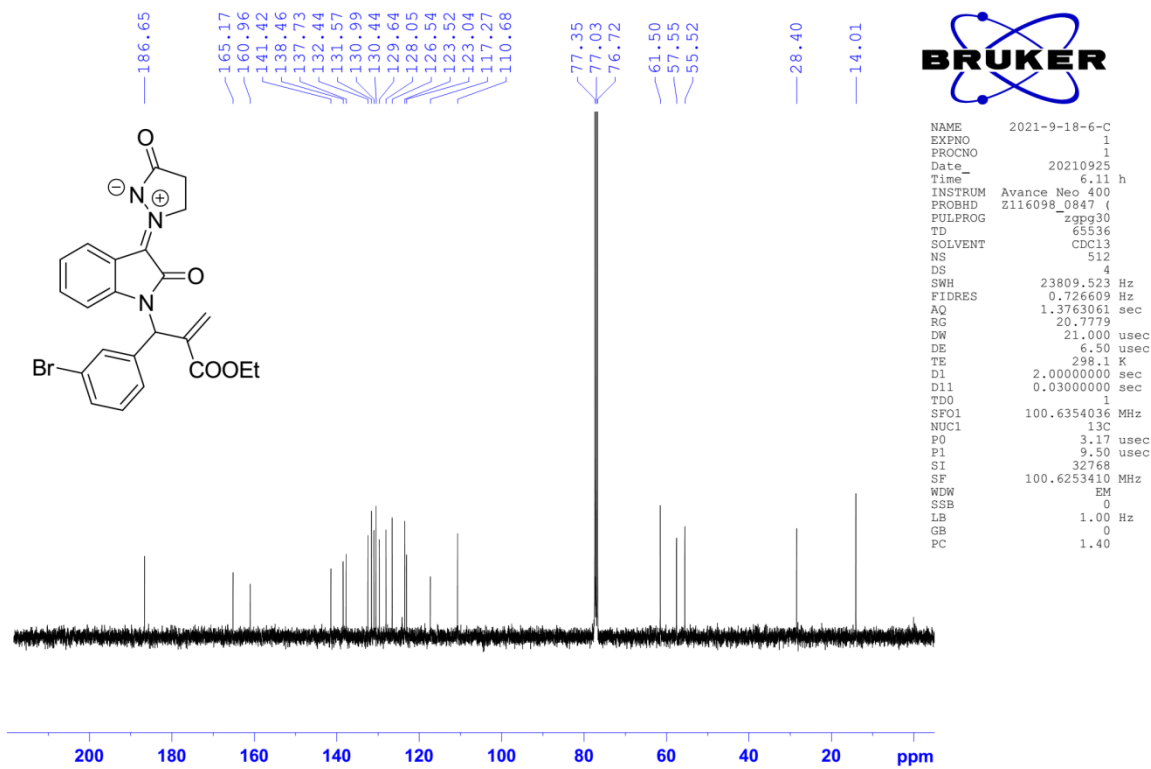
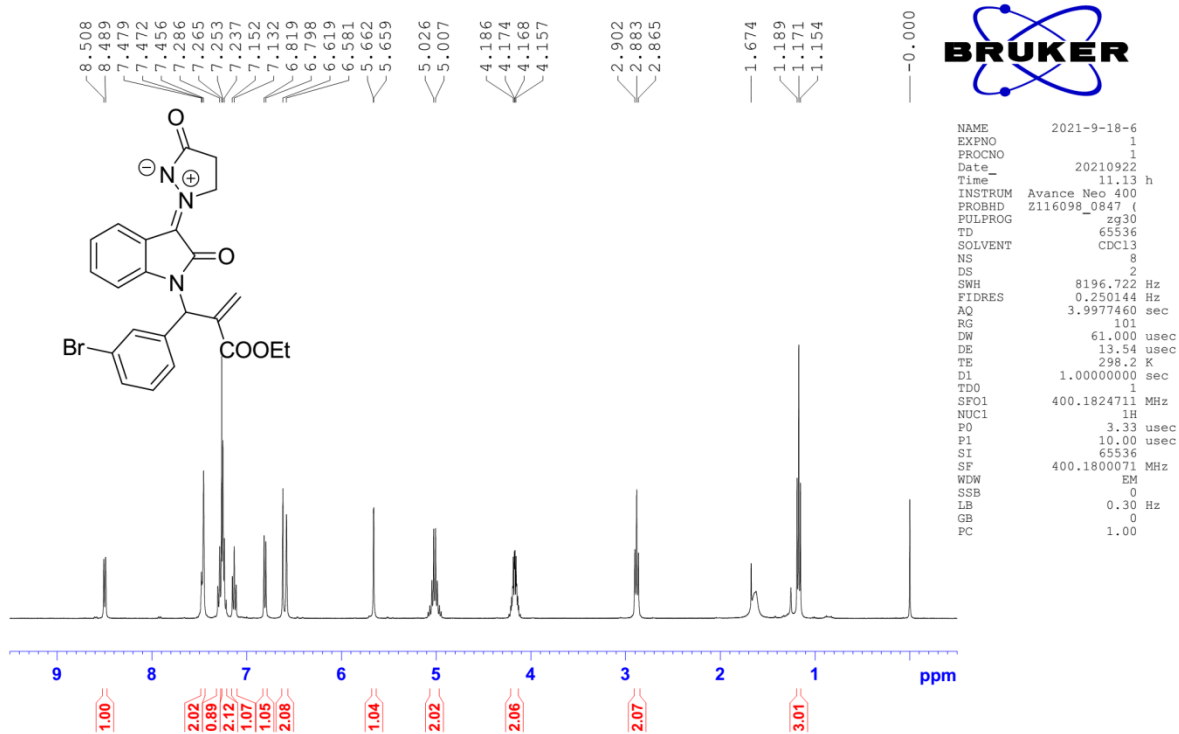
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 61



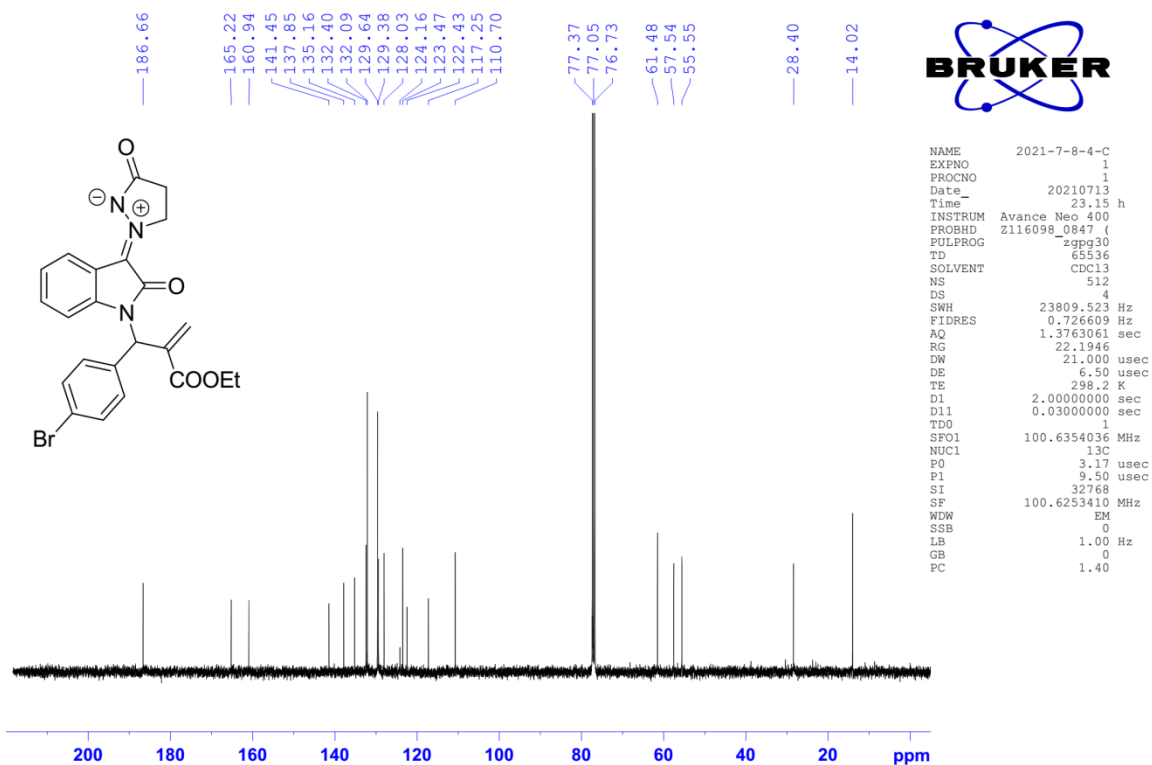
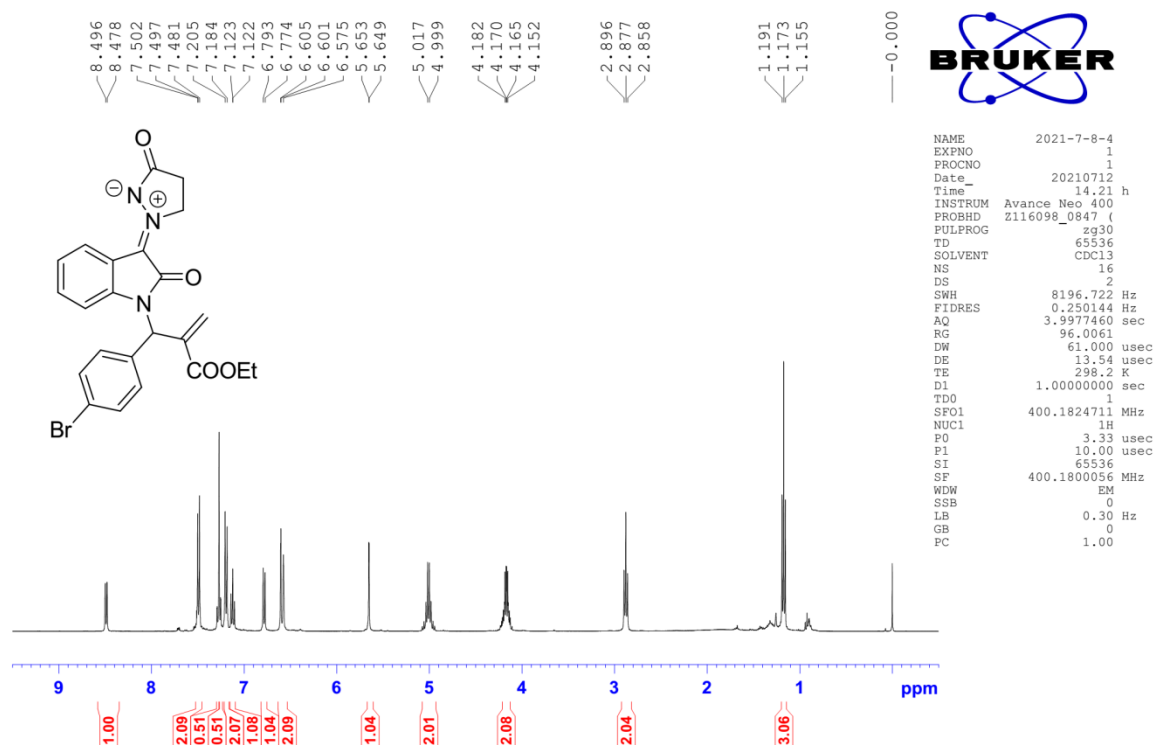
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound **6m**



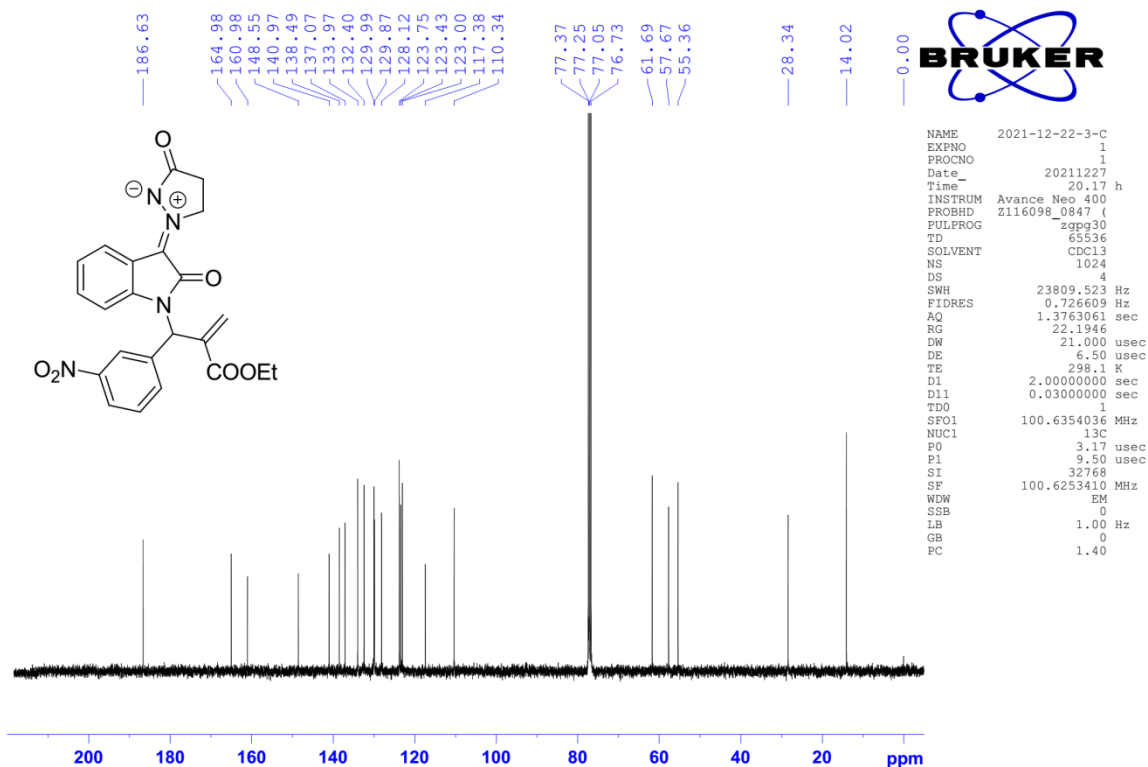
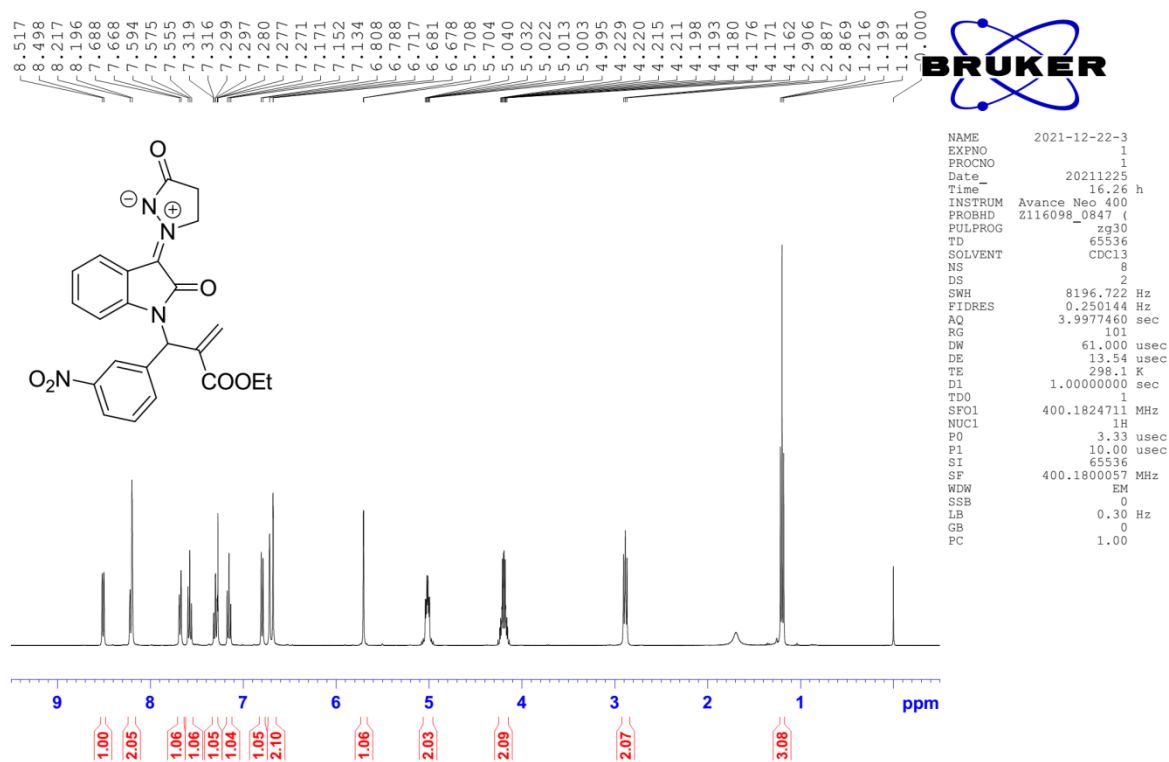


<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound **6o**

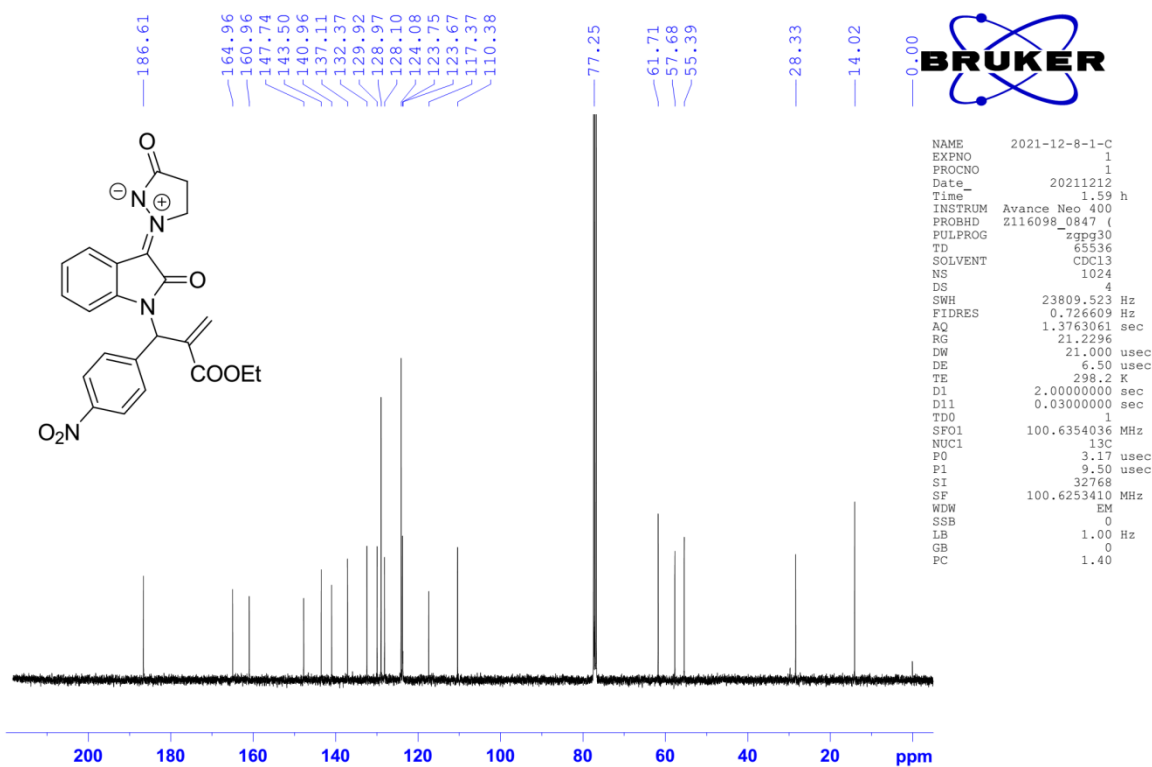
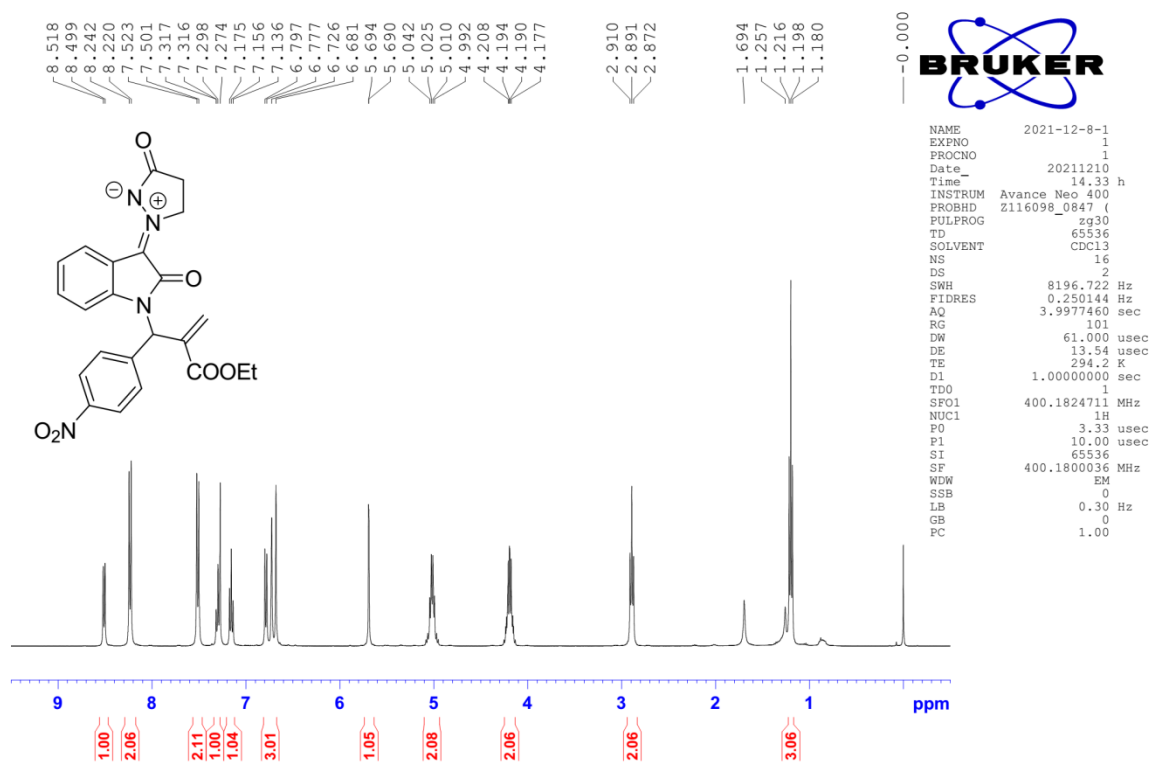
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6p

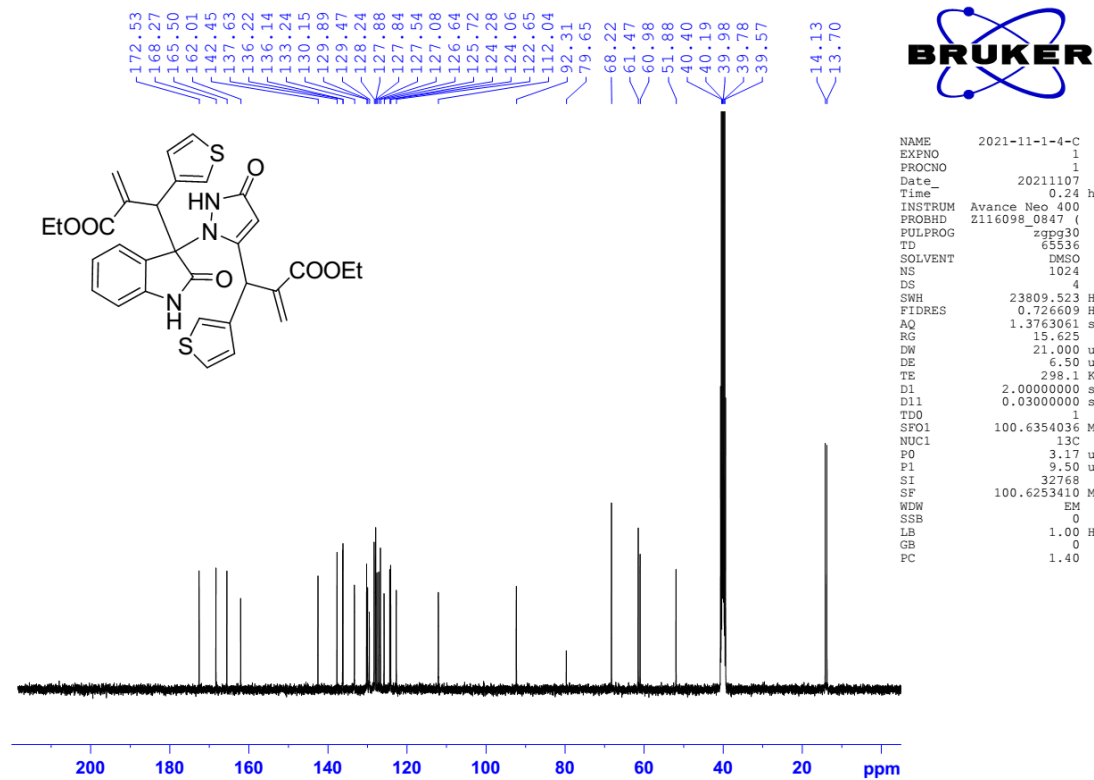
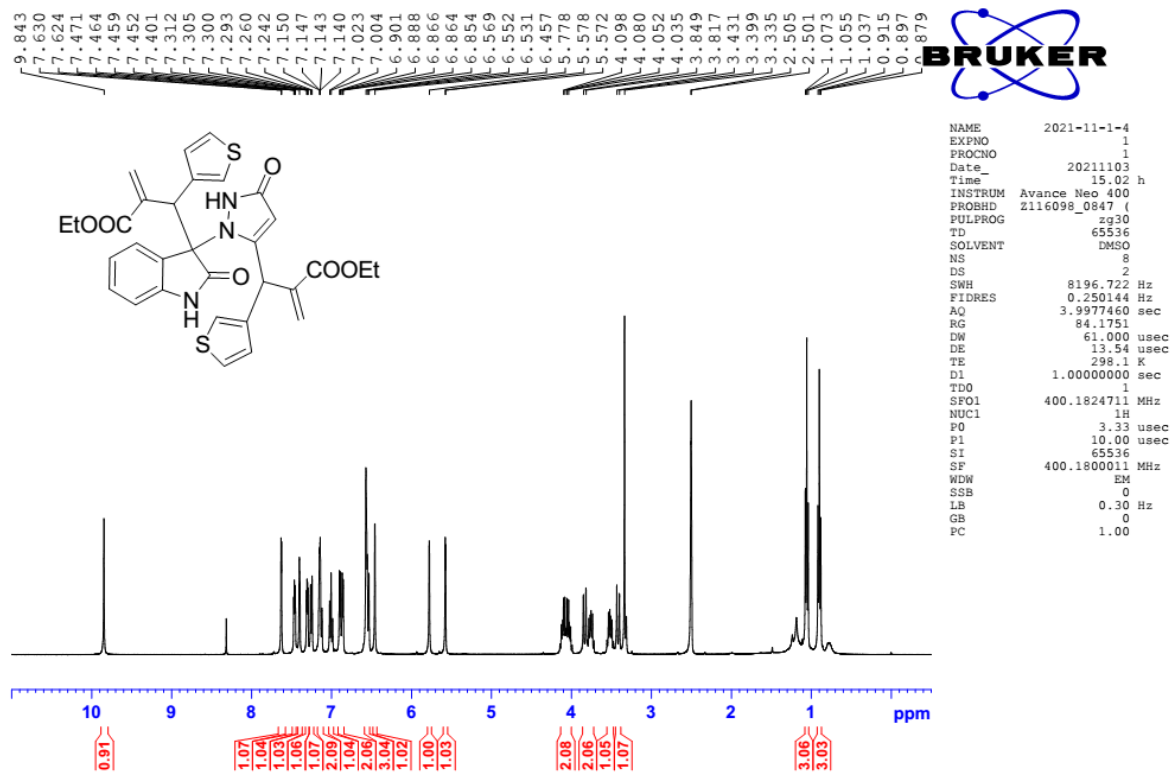


# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6r

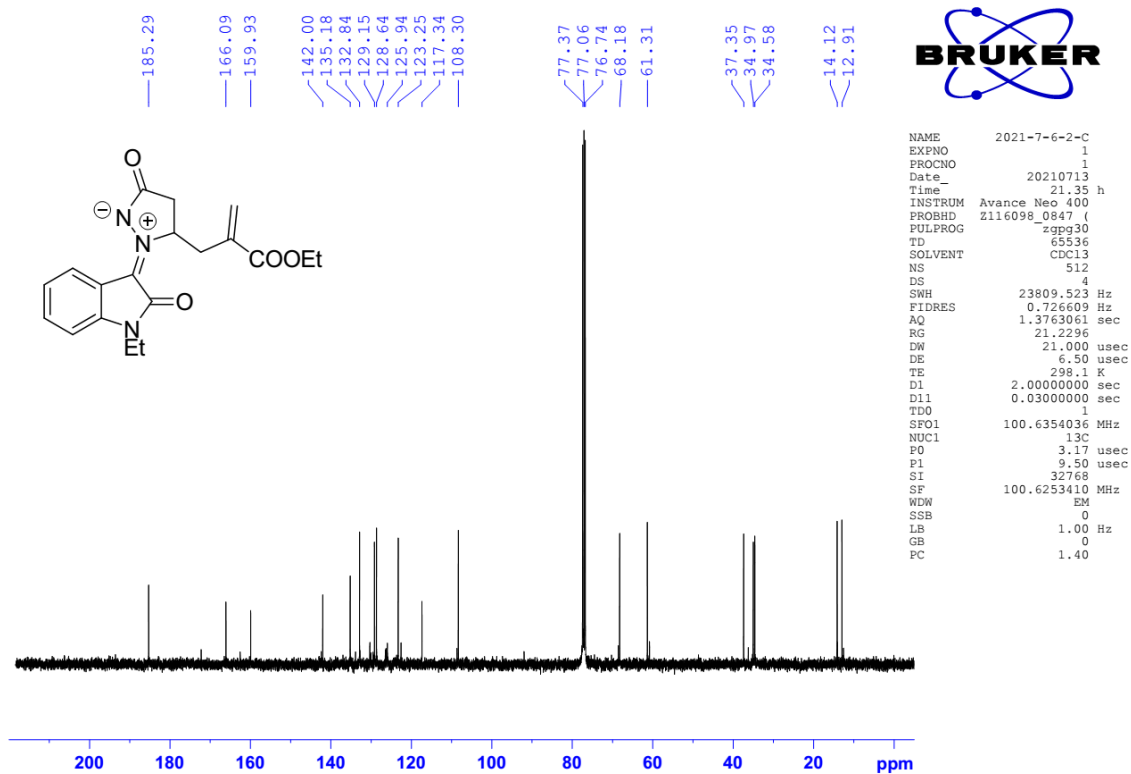
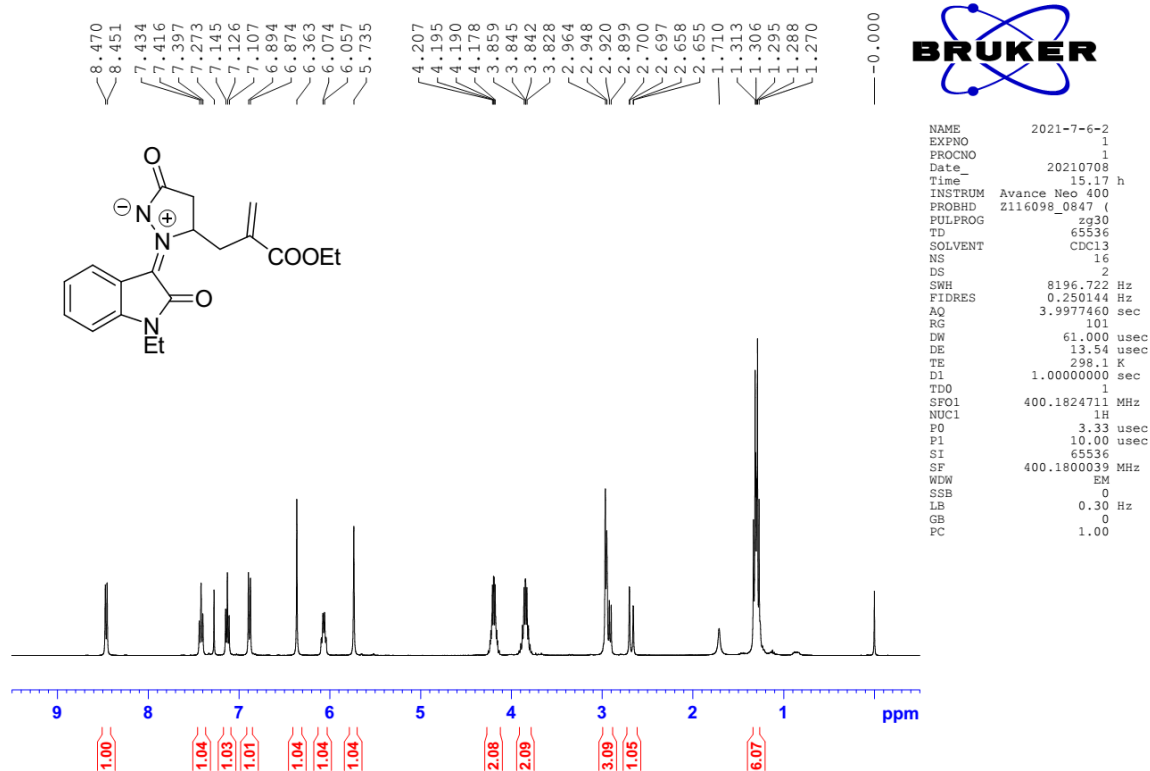


# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6s

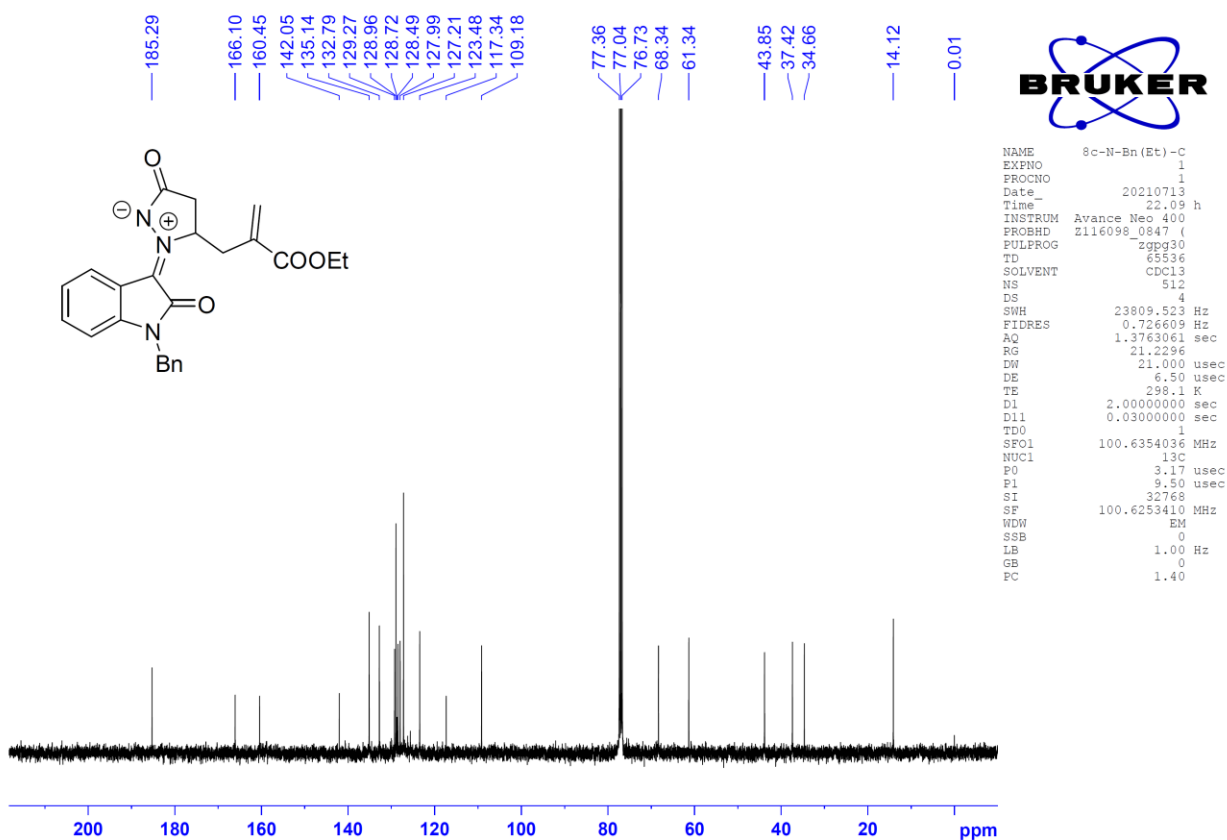
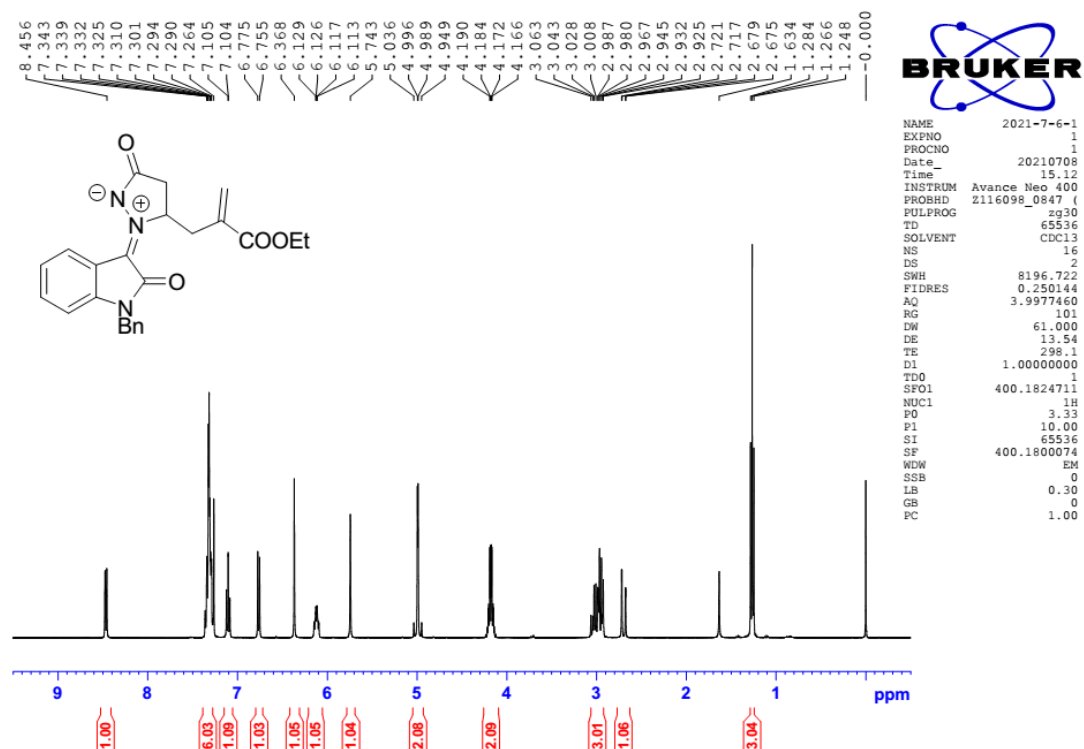


<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 6't

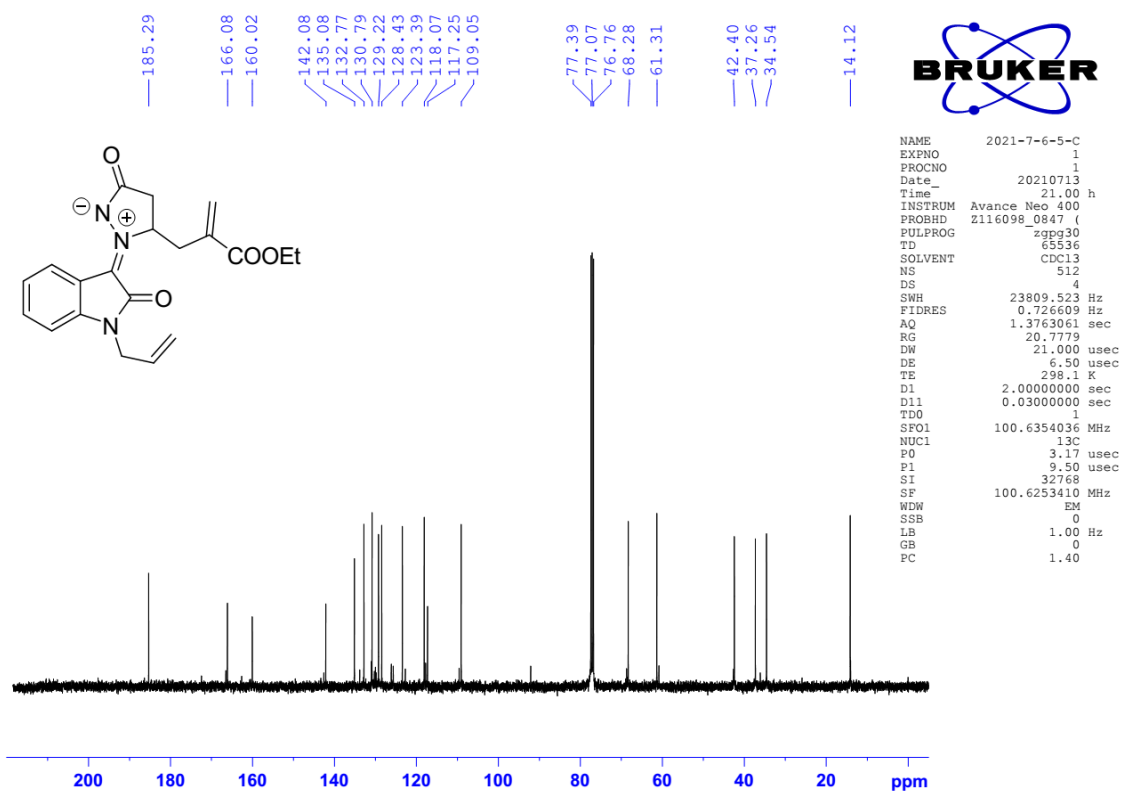
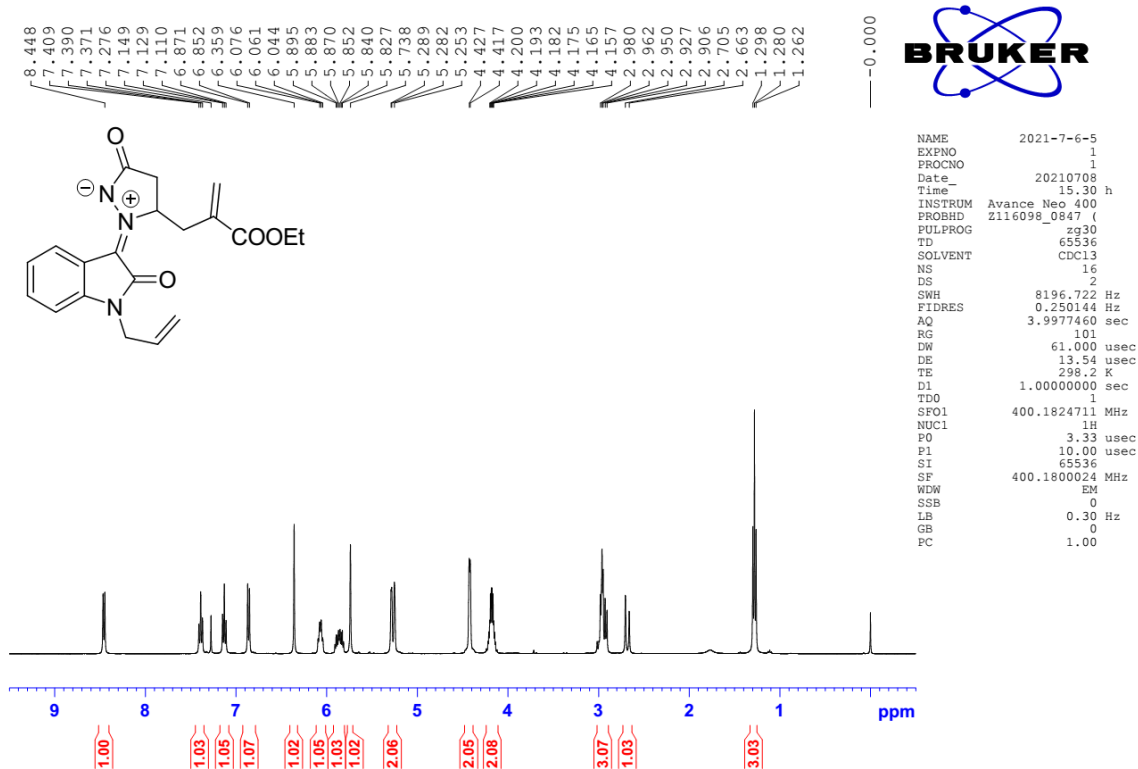
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8b



<sup>1</sup>H NMR Spectra for Compound 8c (a known compound)

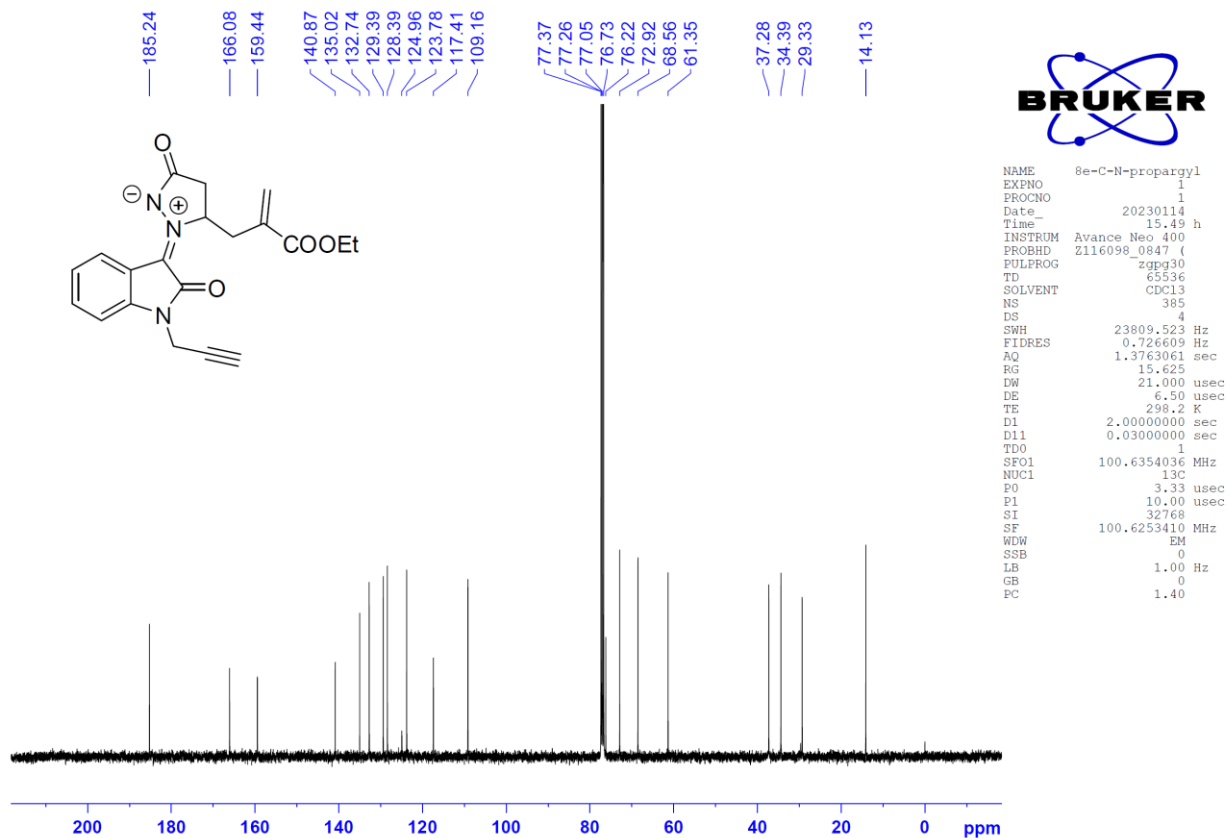
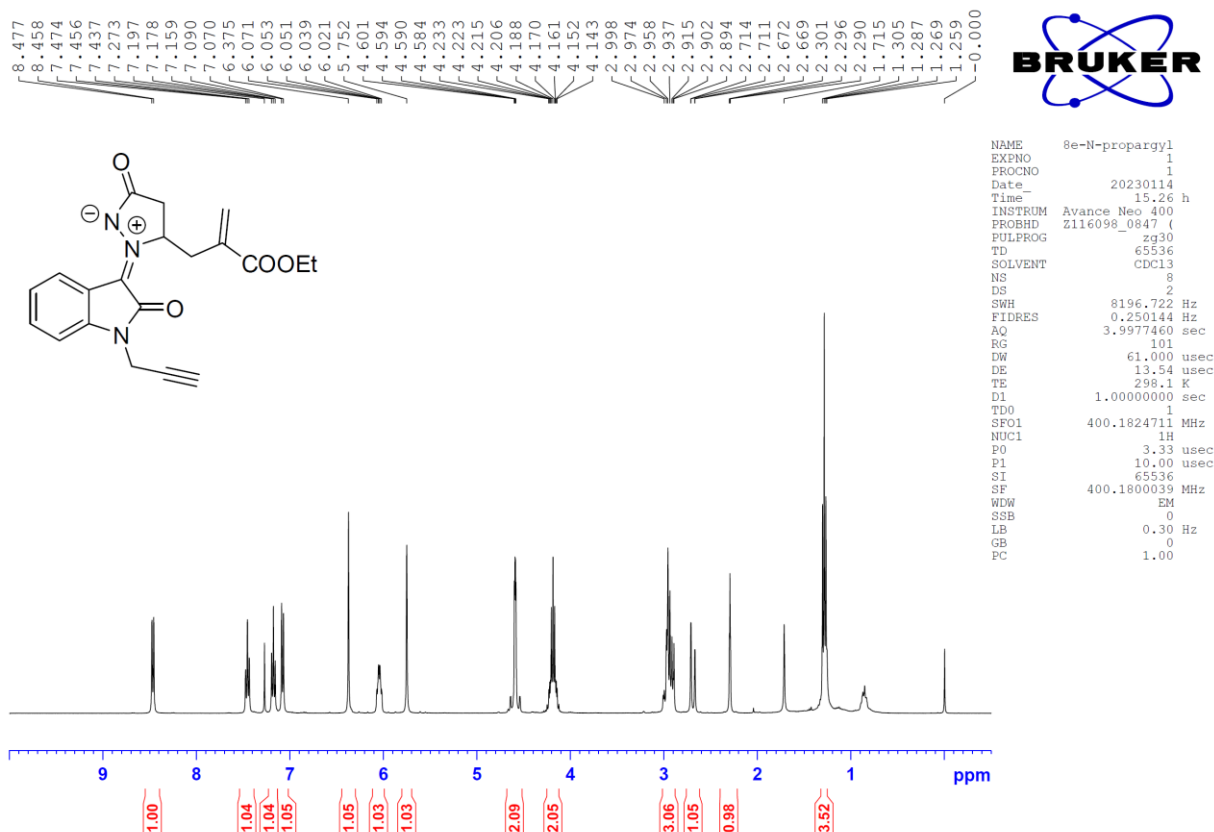


# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8d

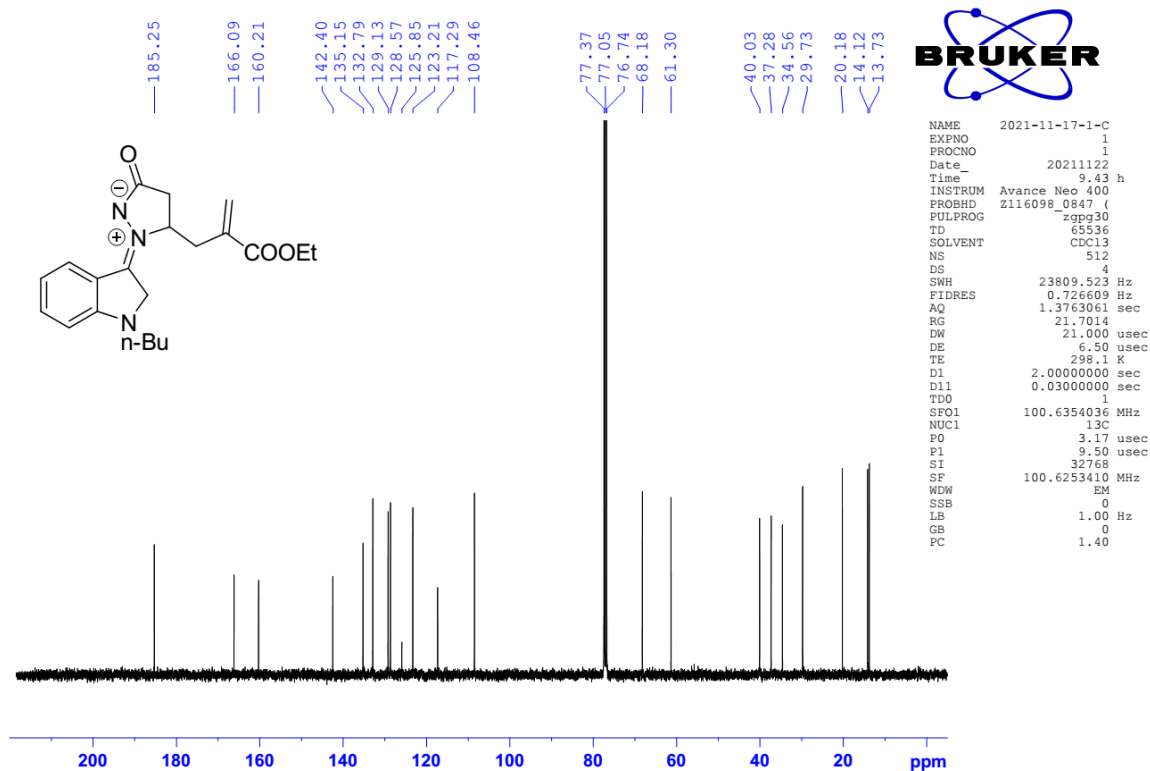
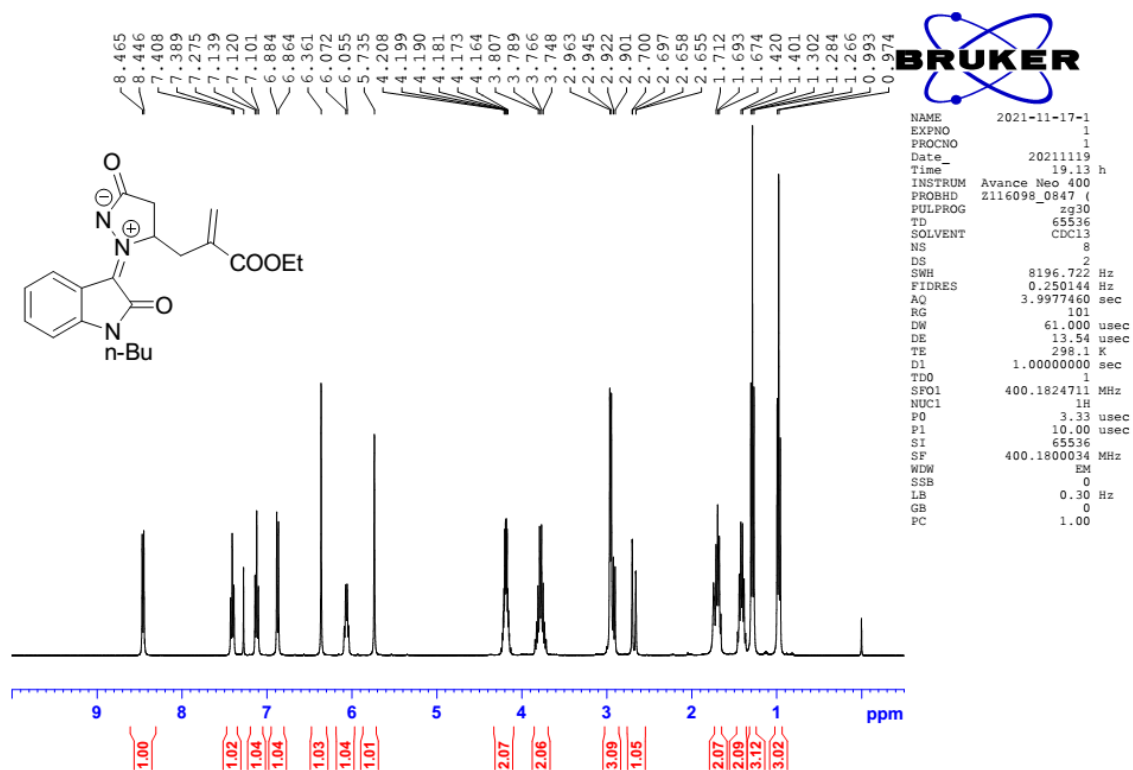




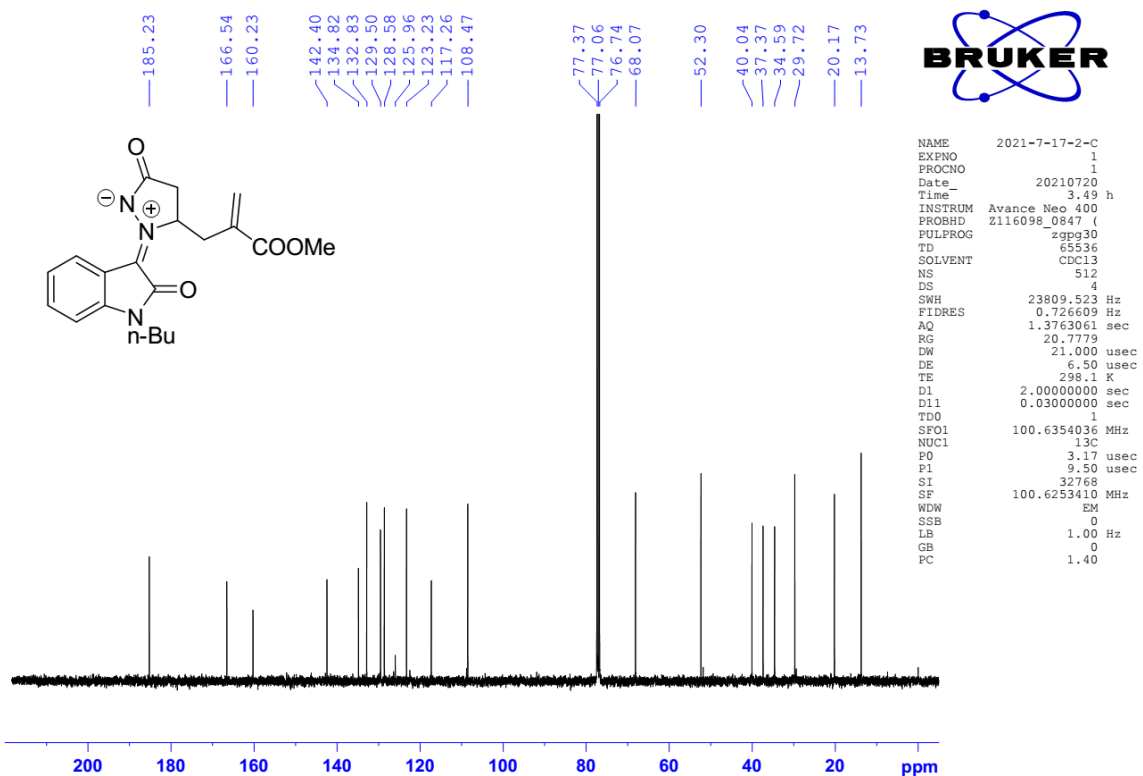
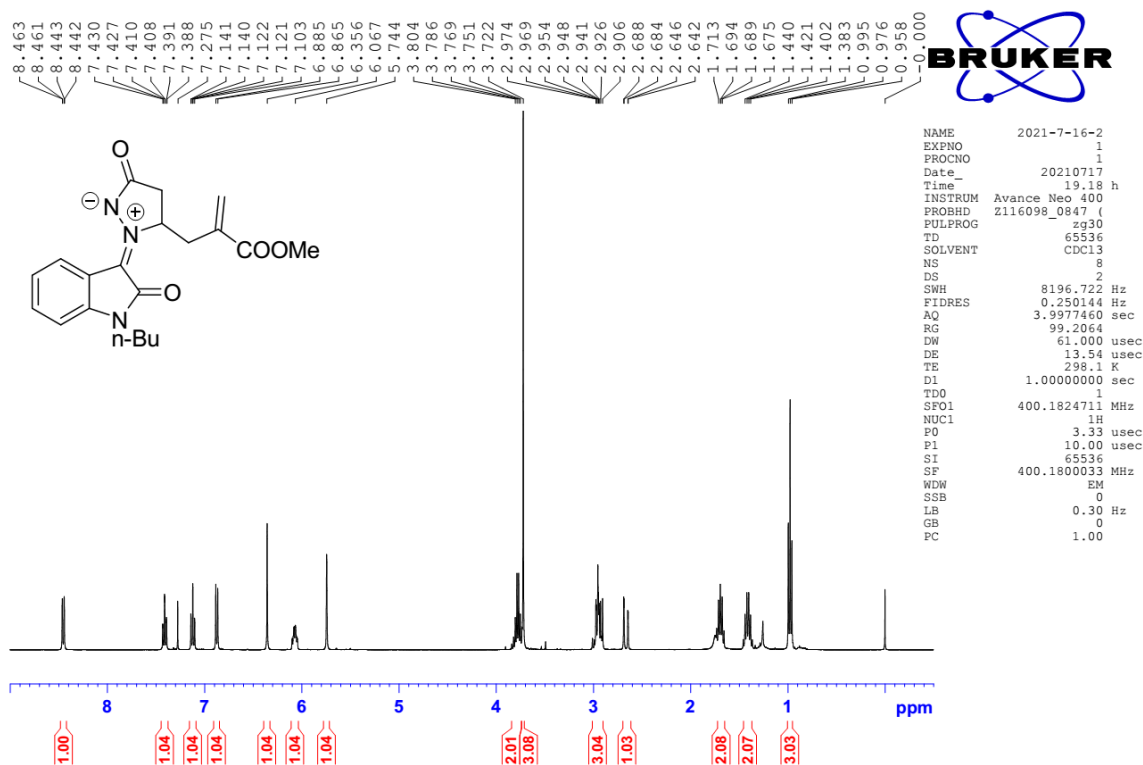
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8e



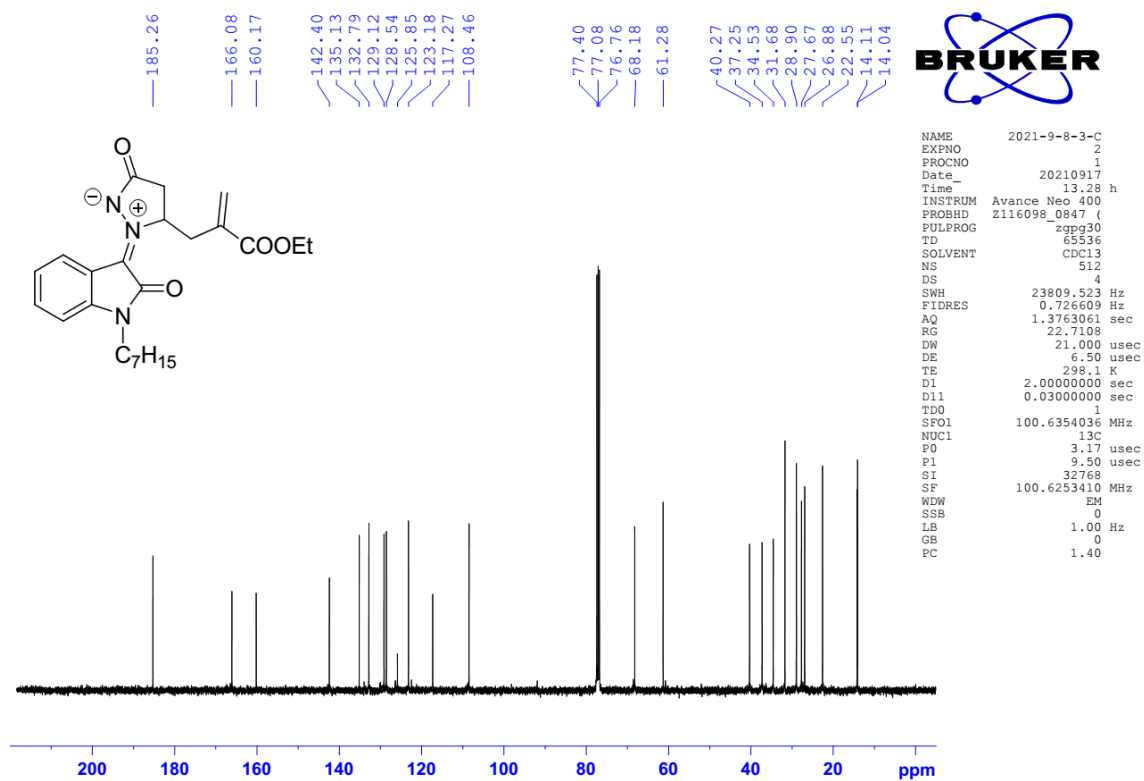
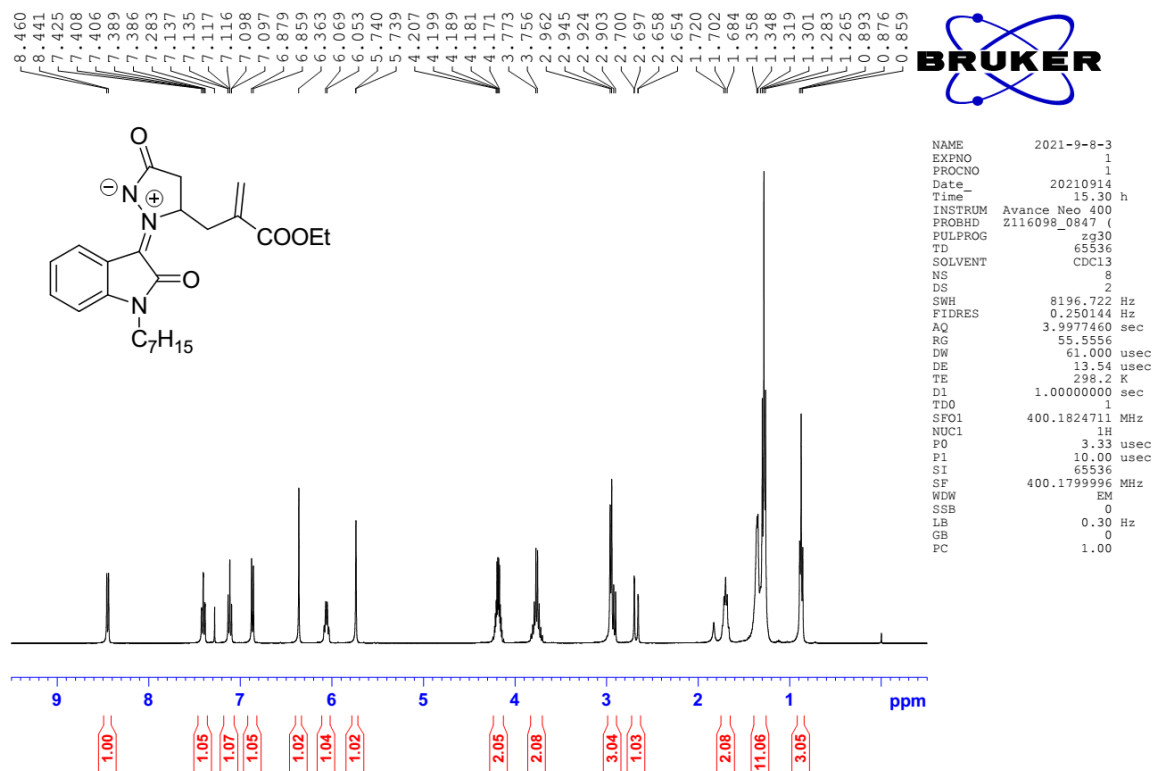
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8f



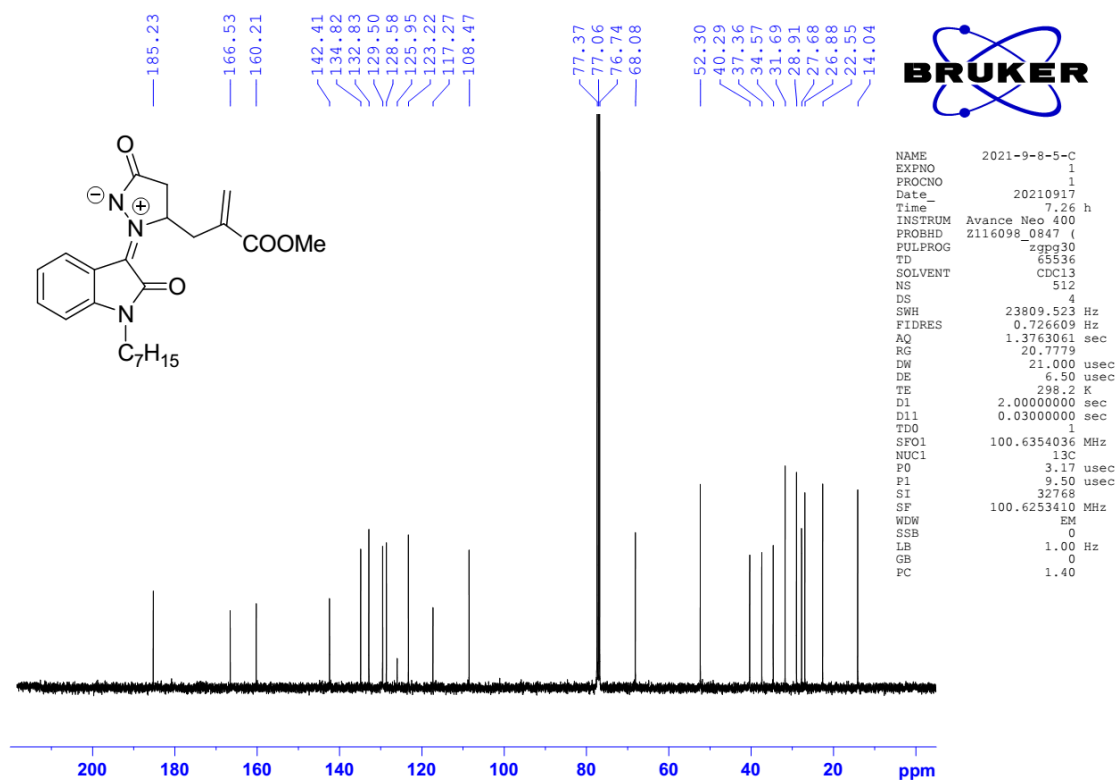
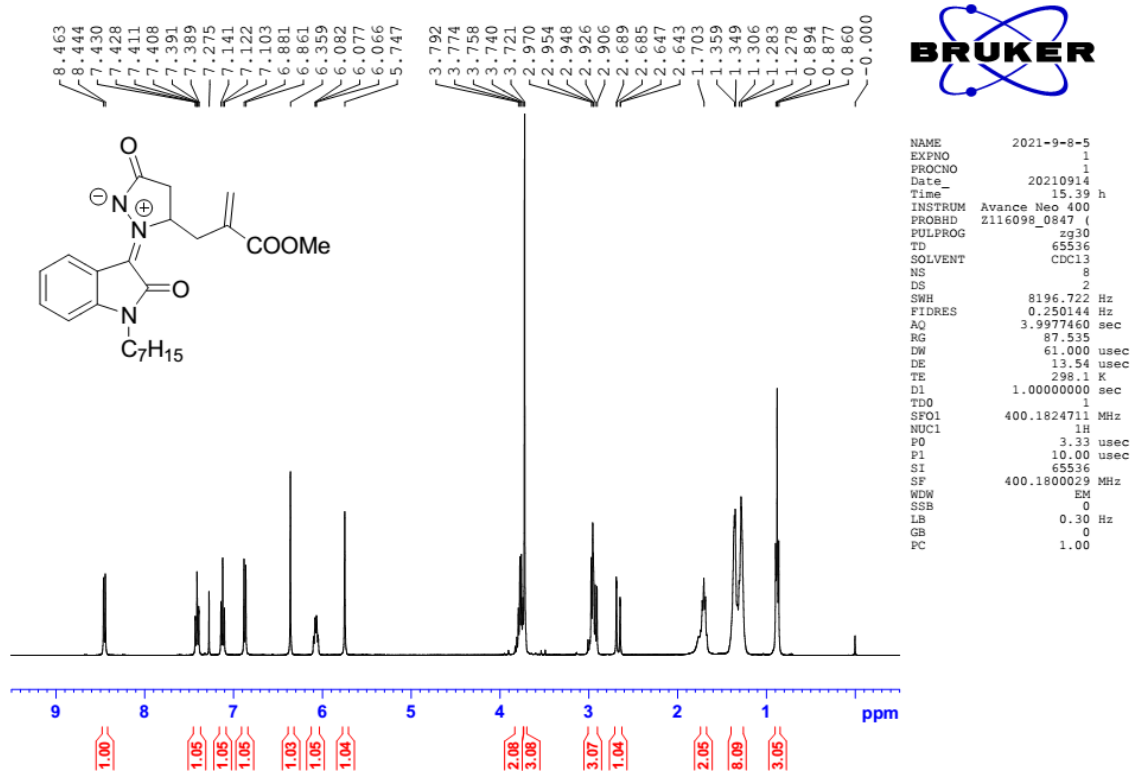
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8g



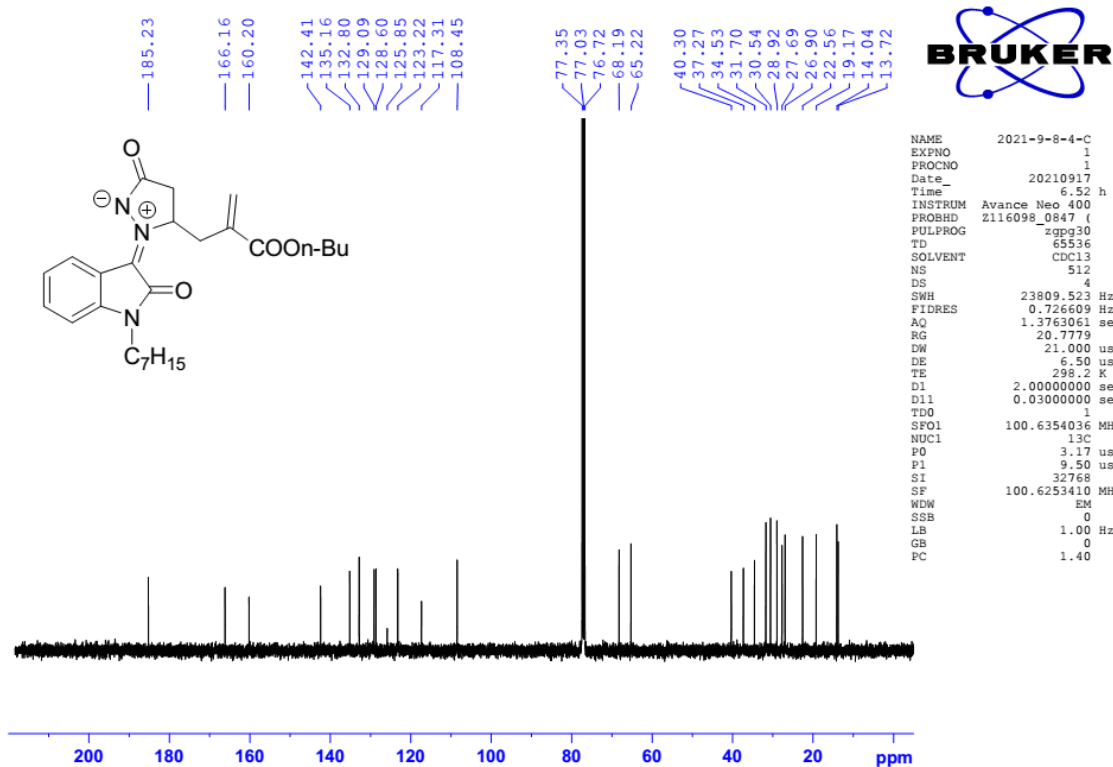
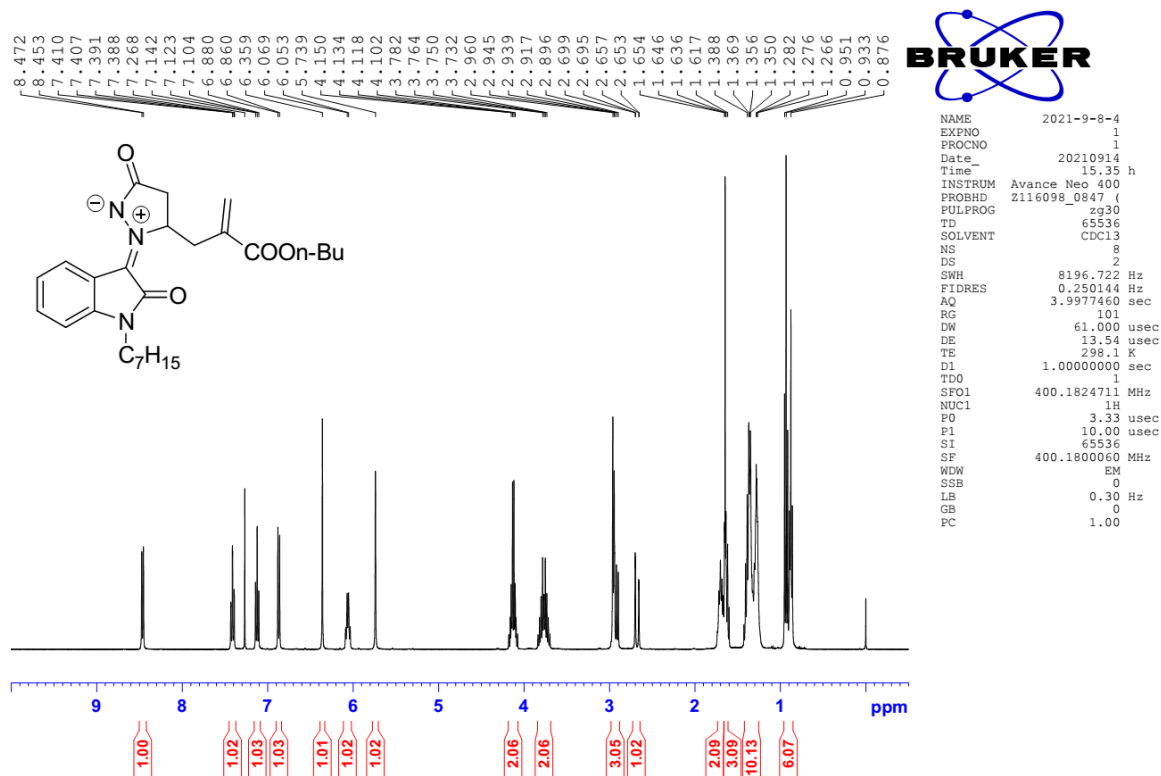
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8h



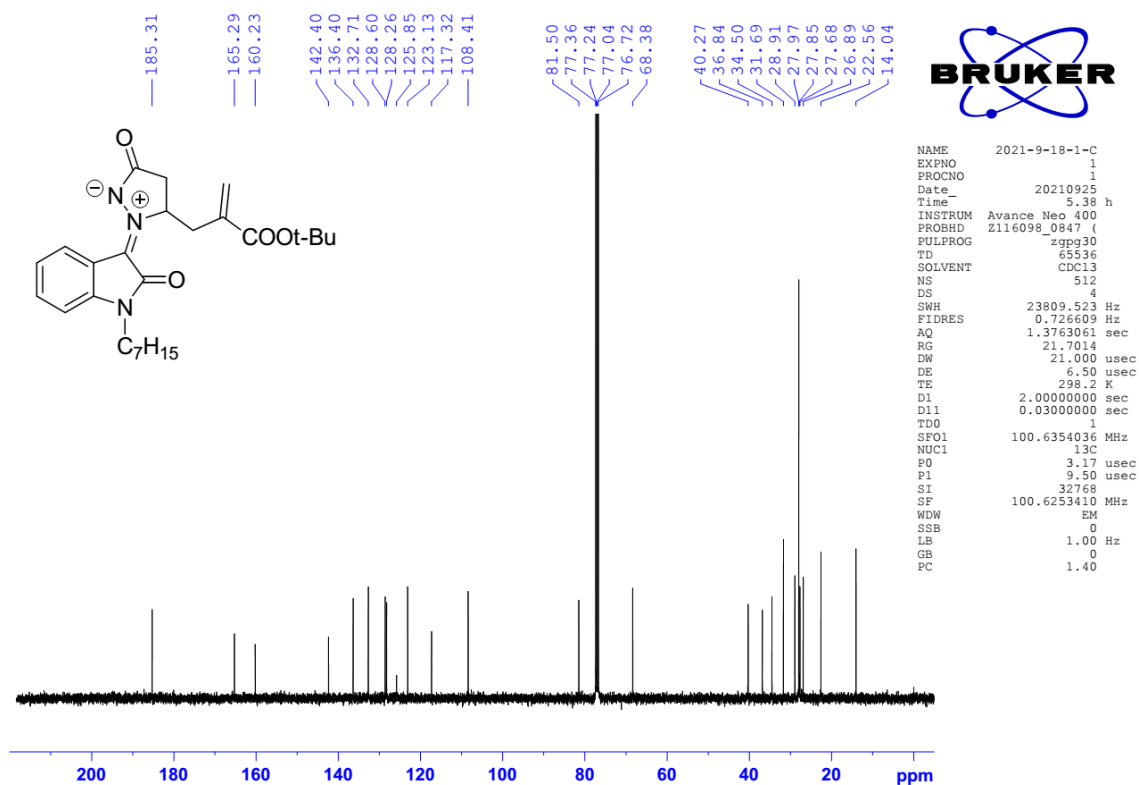
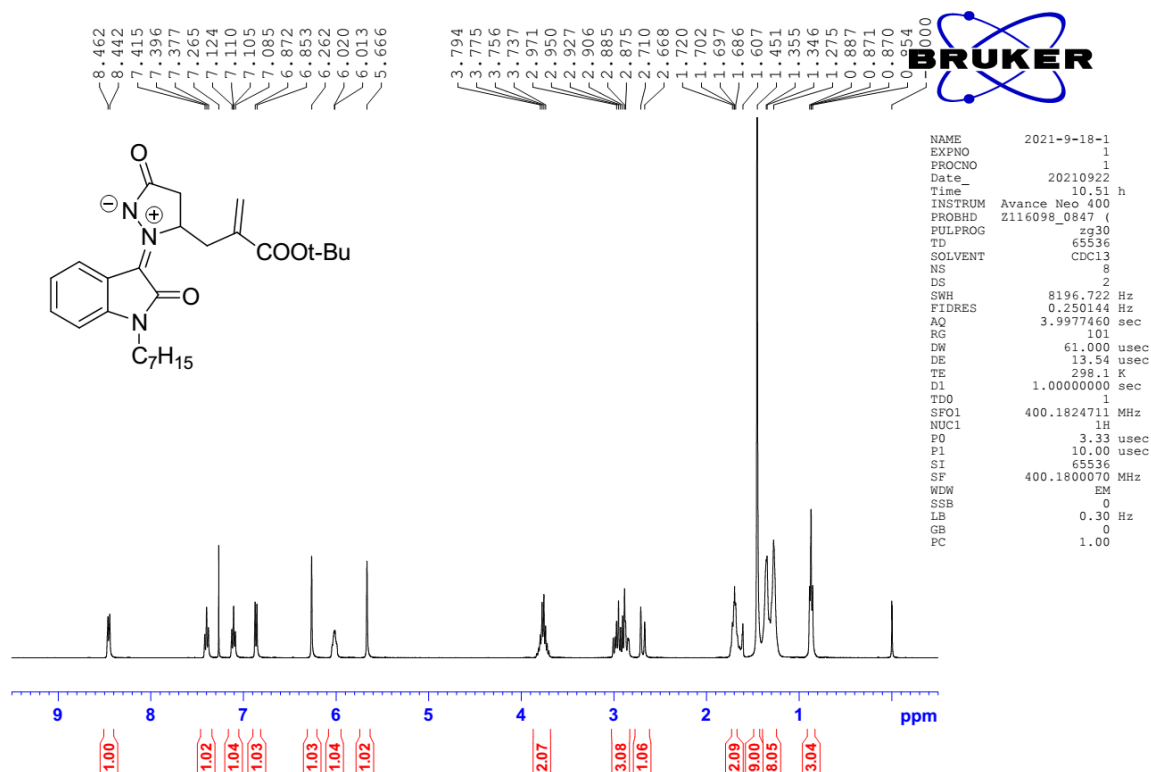
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8i



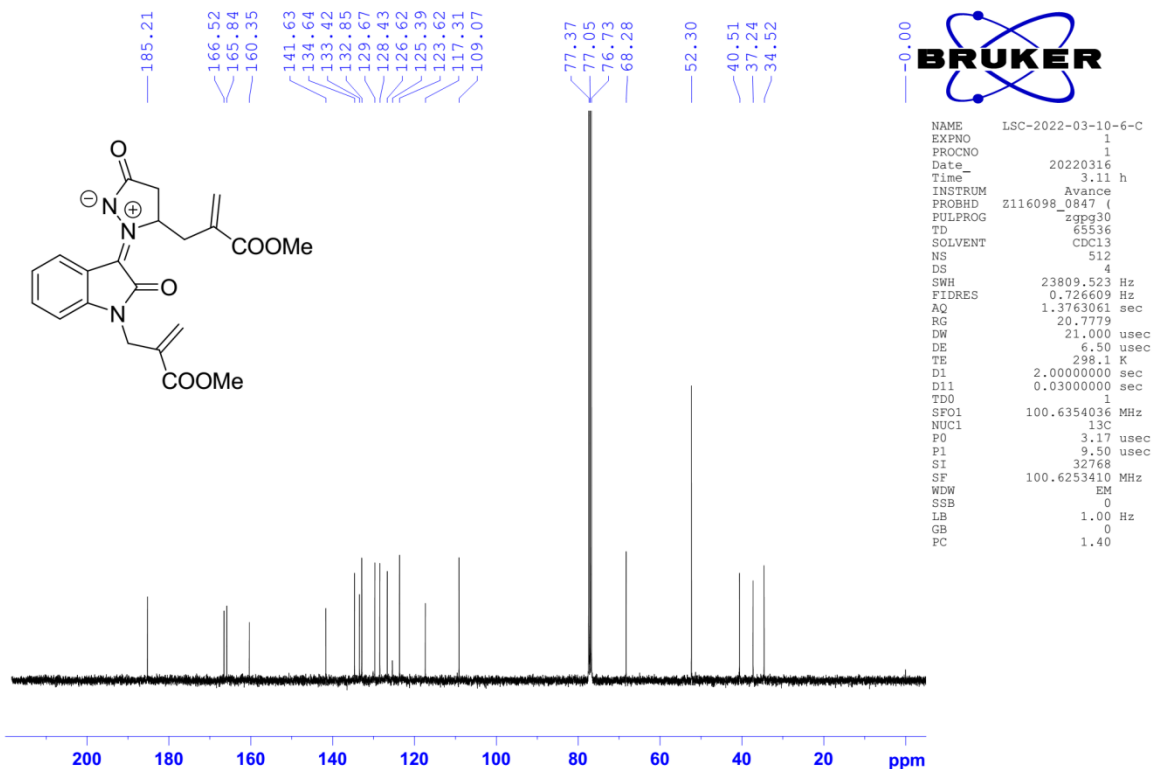
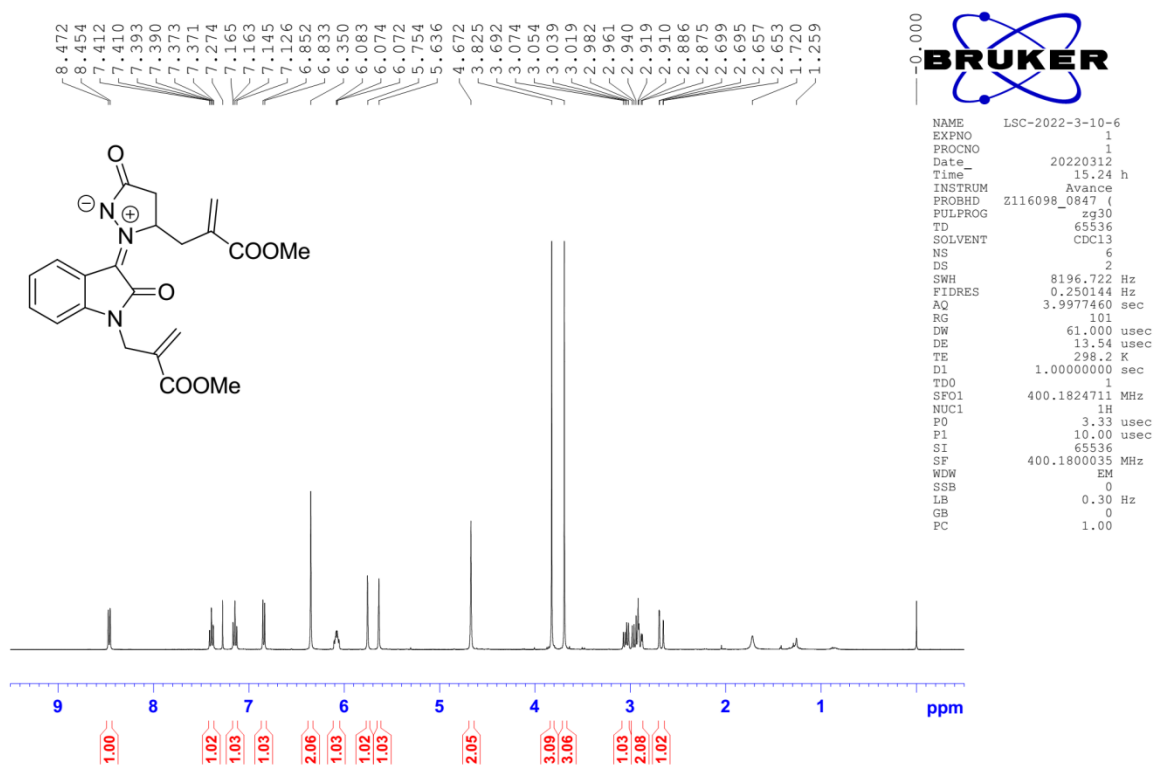
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8j



# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 8k

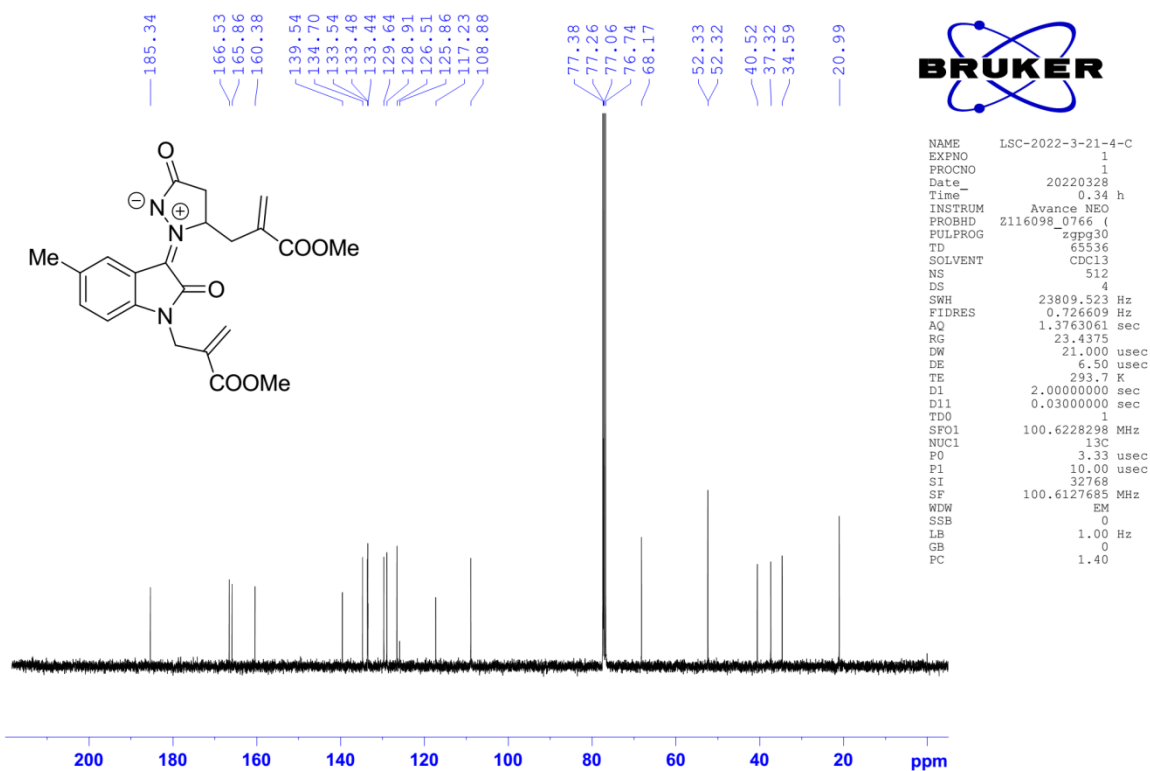
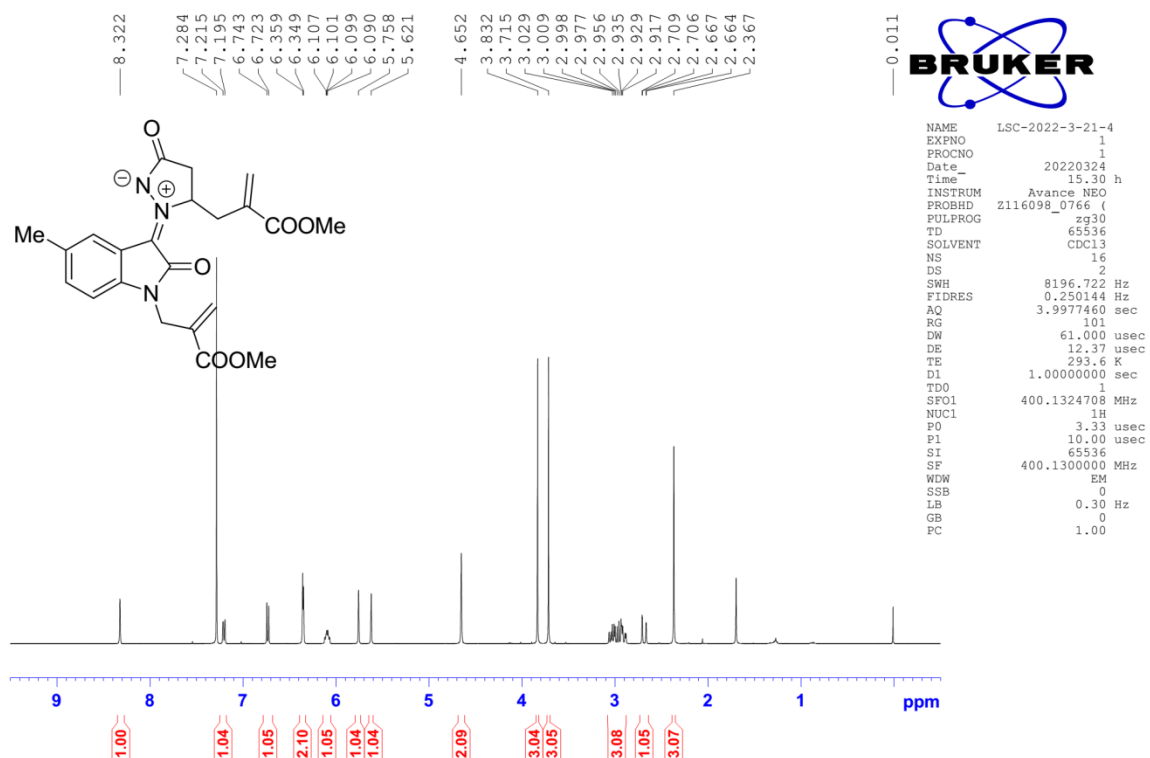


# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4a

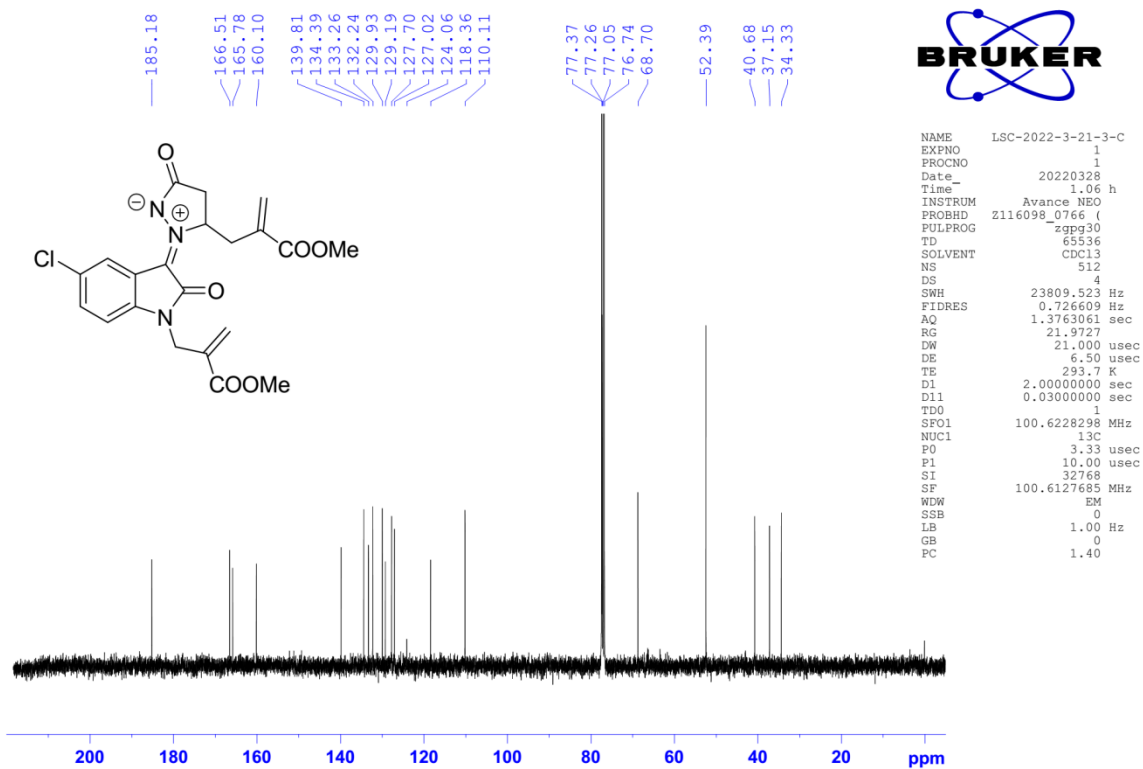
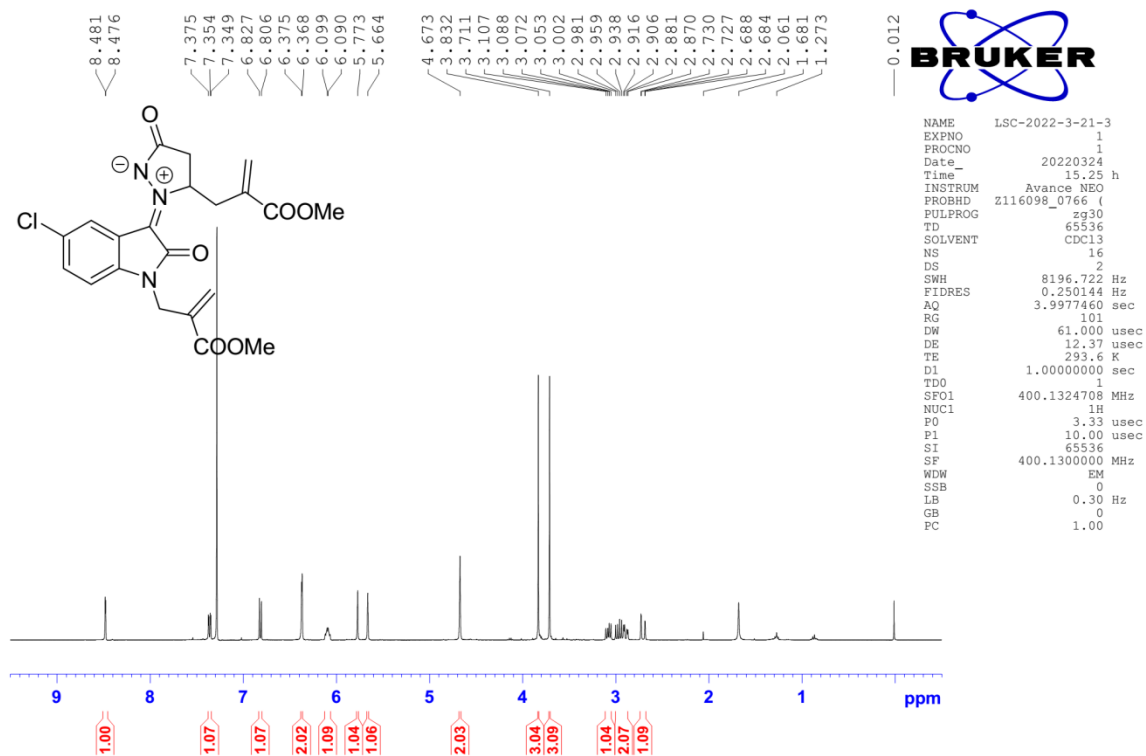




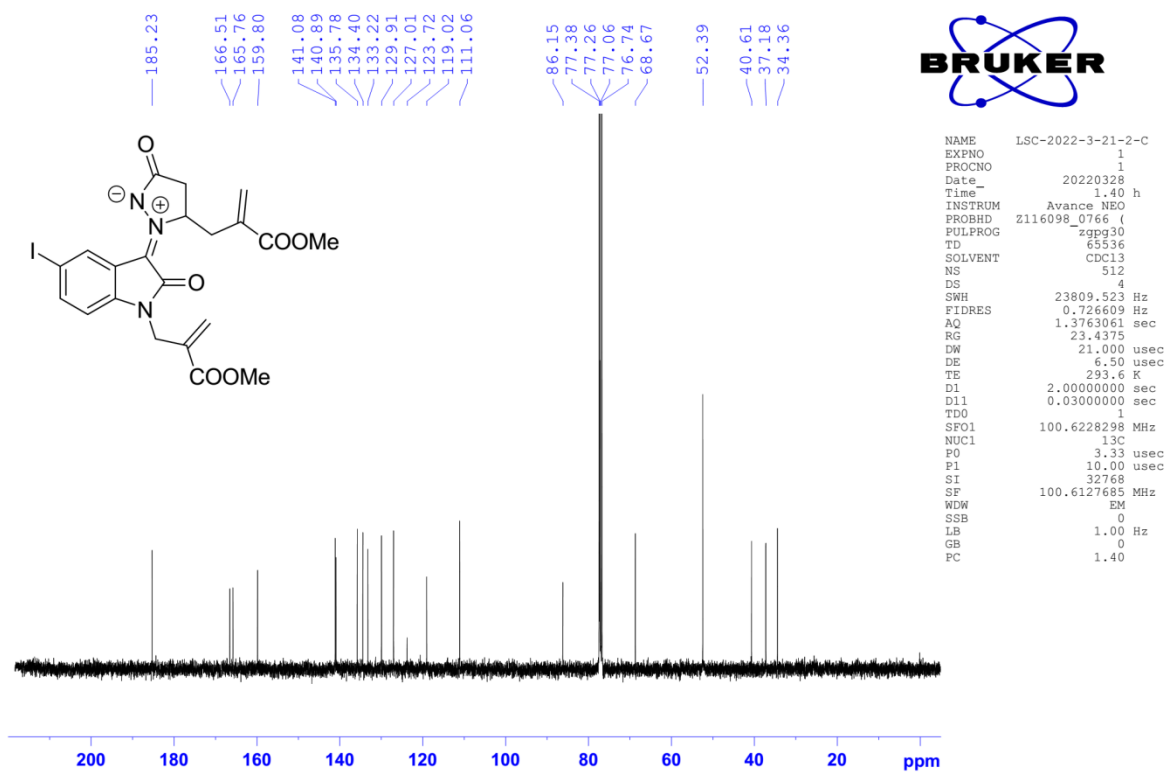
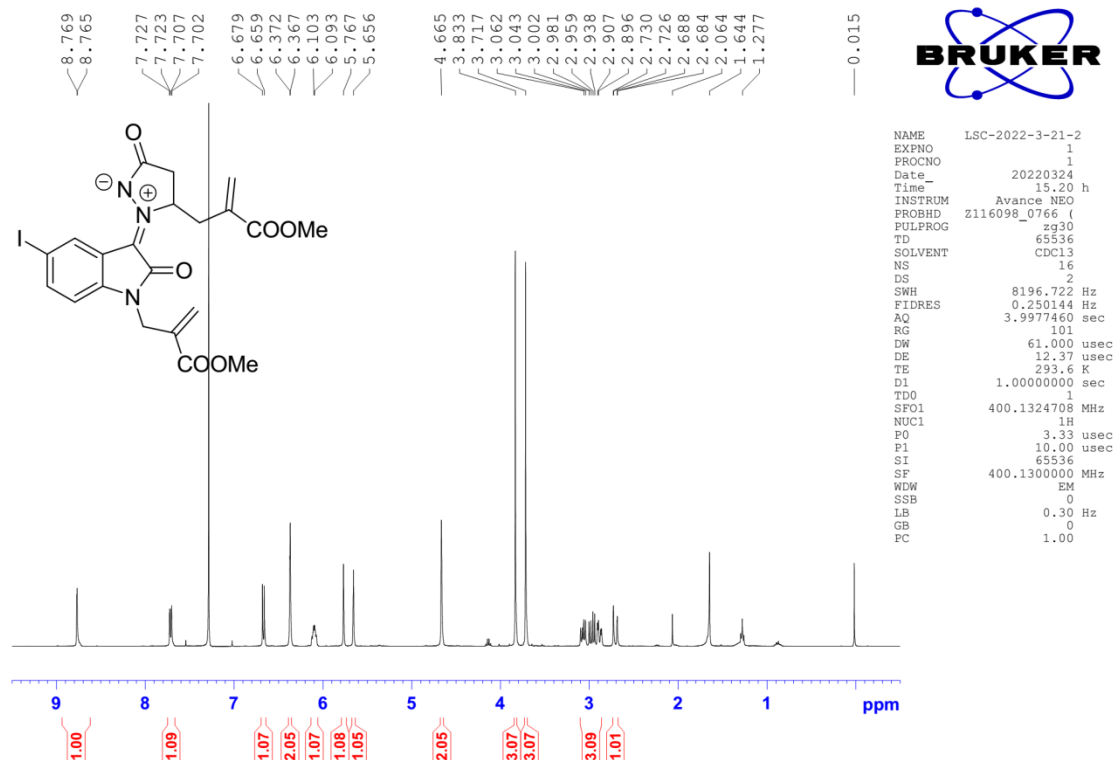
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4b



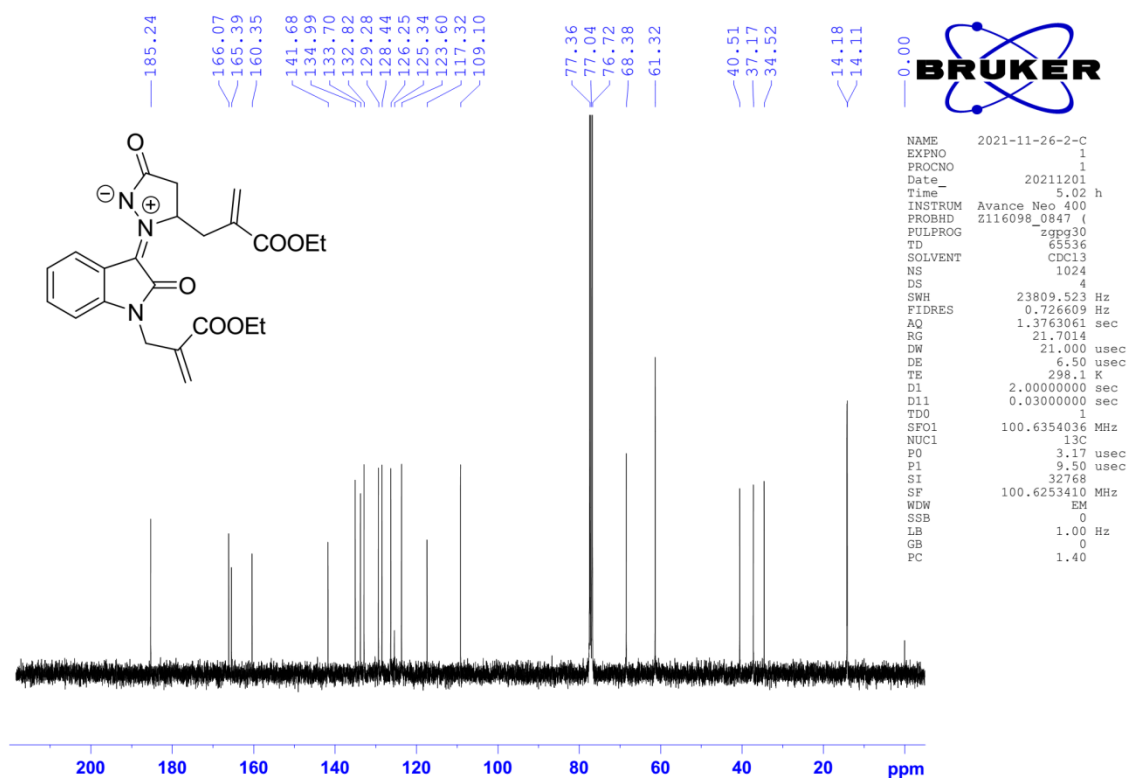
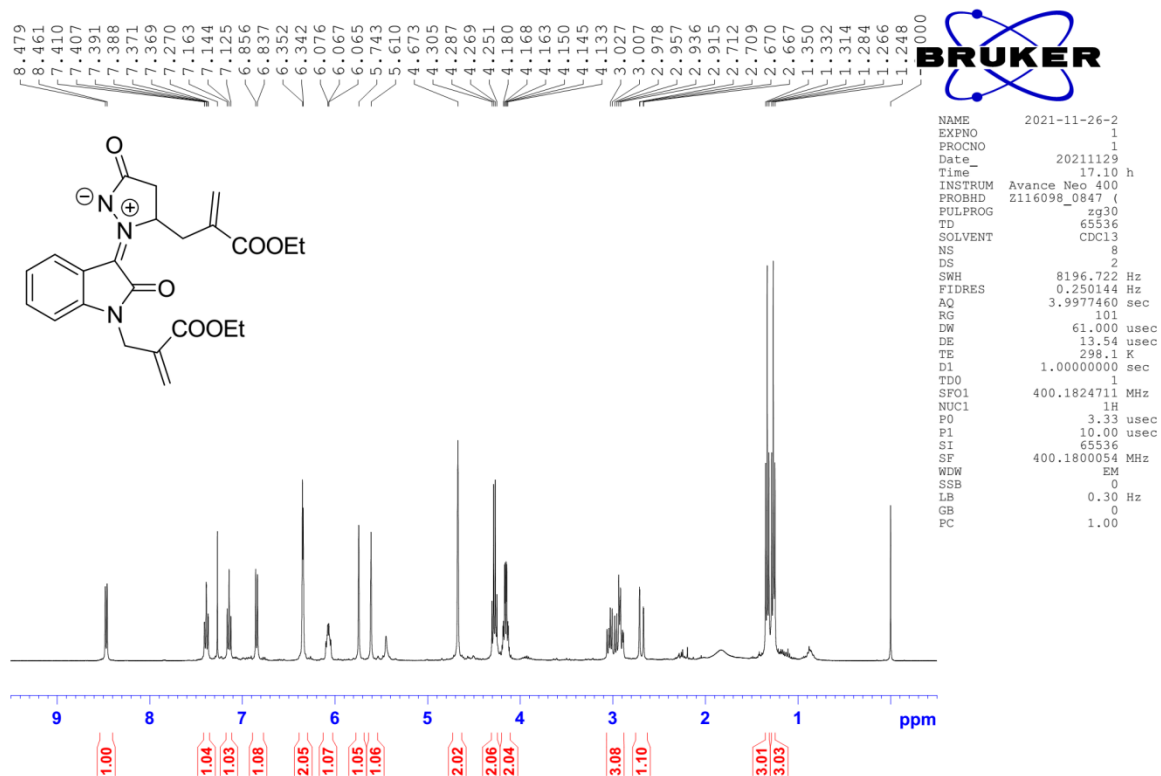
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4c



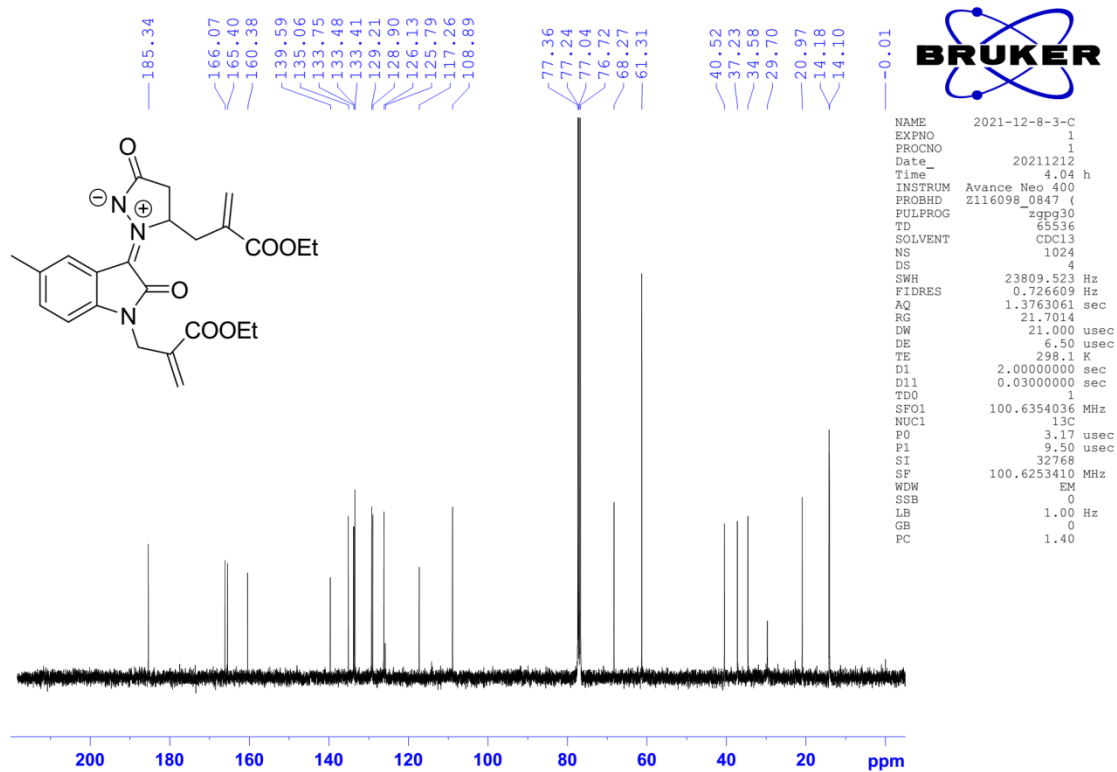
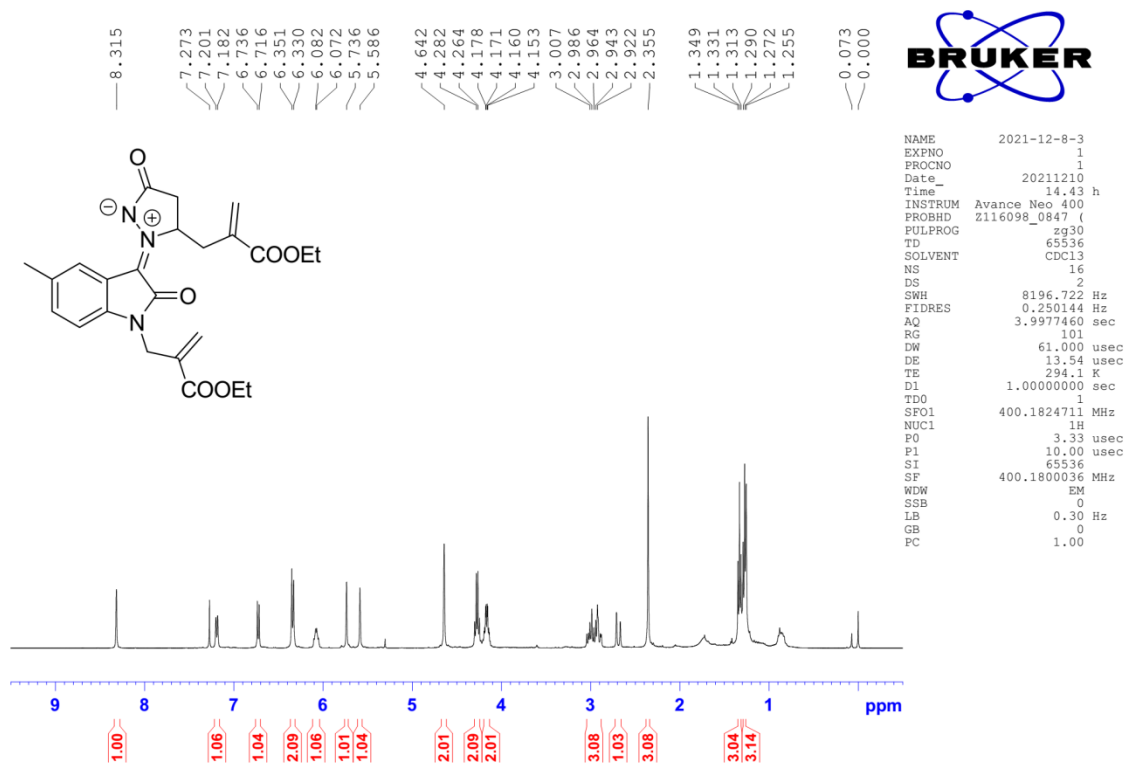
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4d



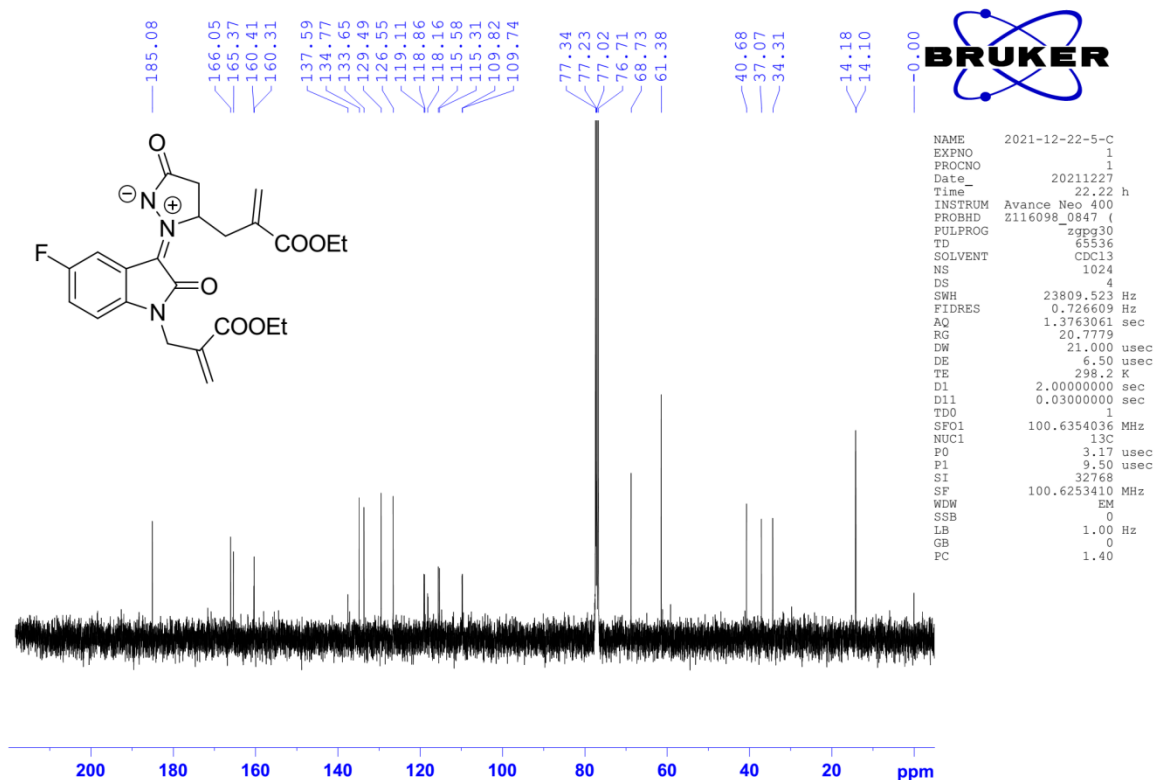
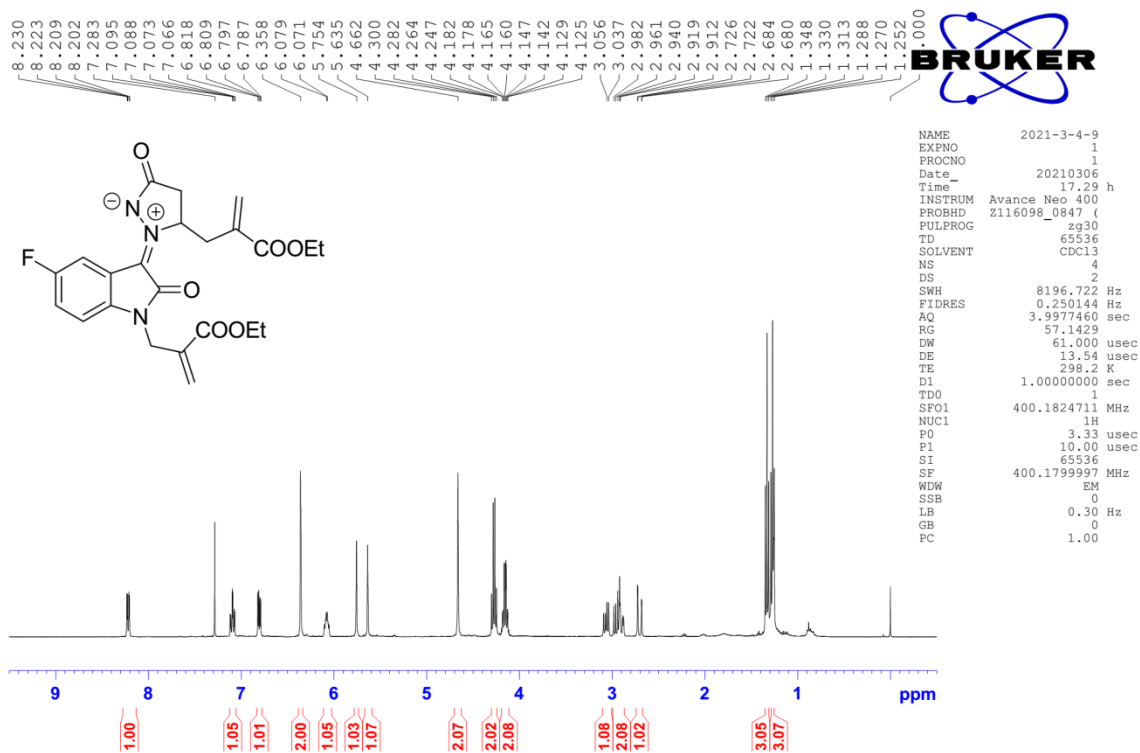
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4e



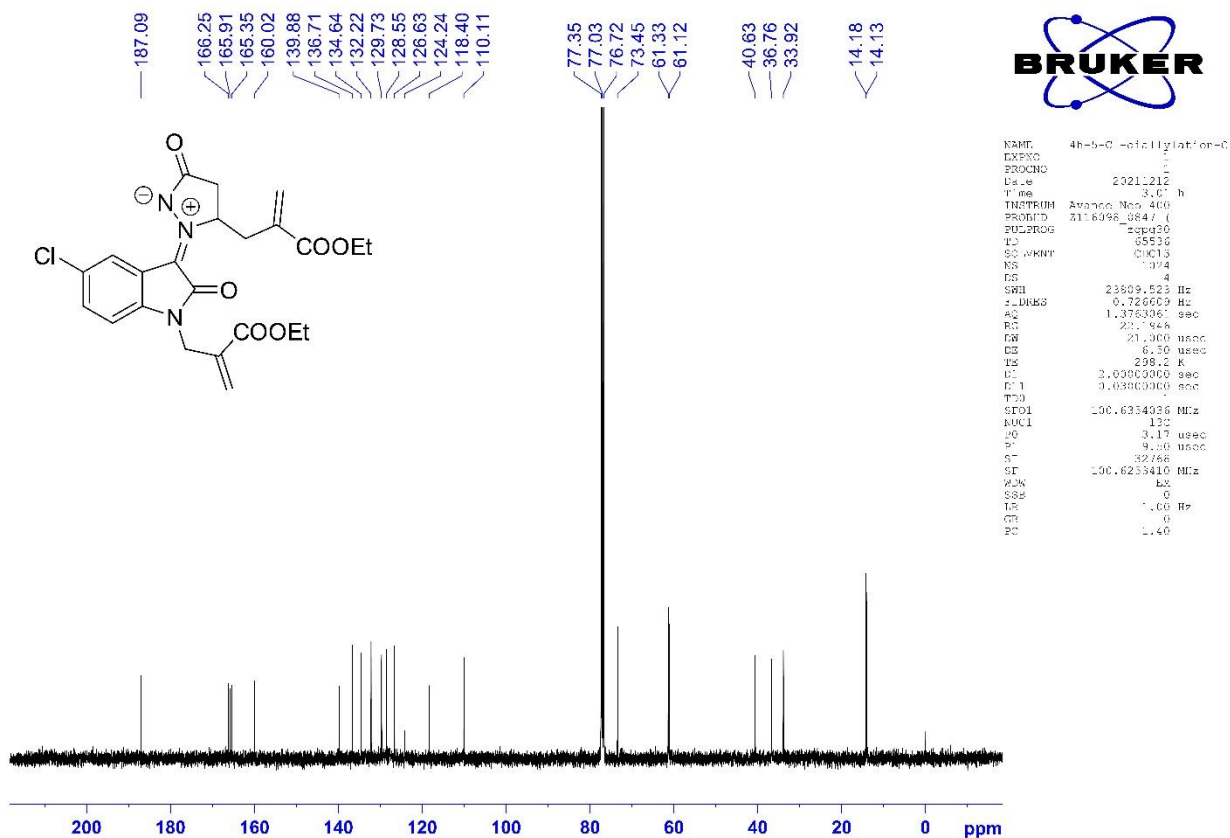
# <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4f



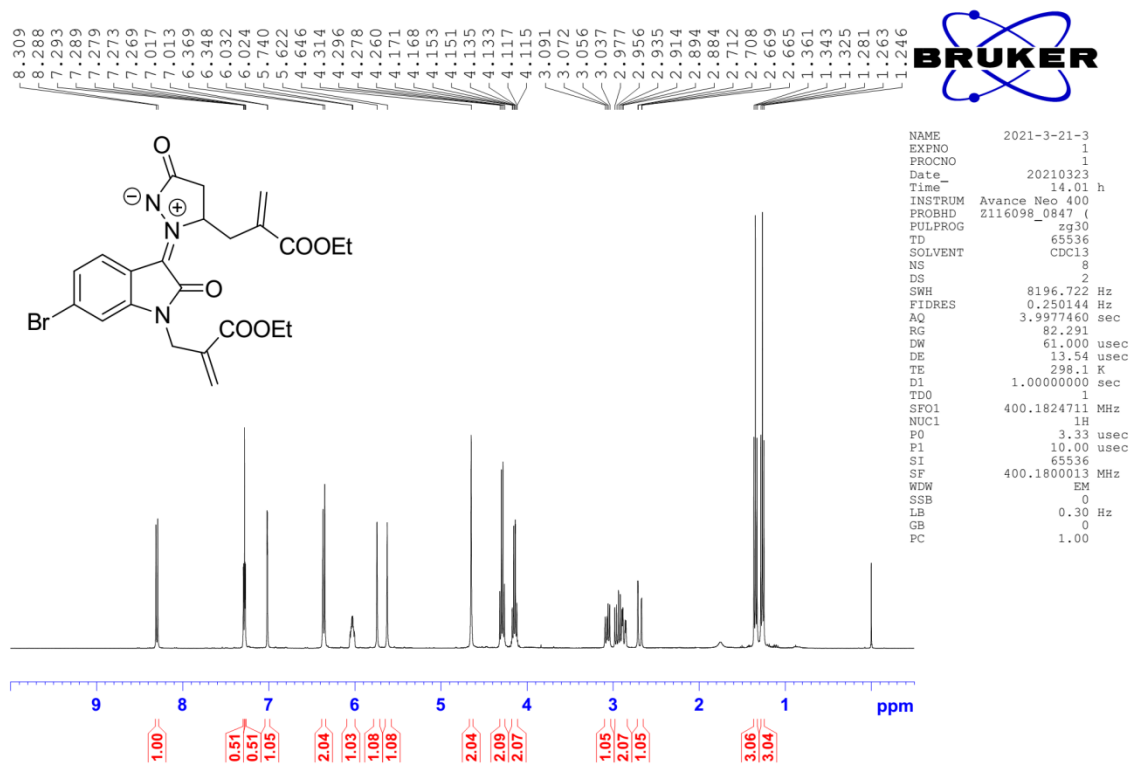
$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR Spectra for Compound **4g**



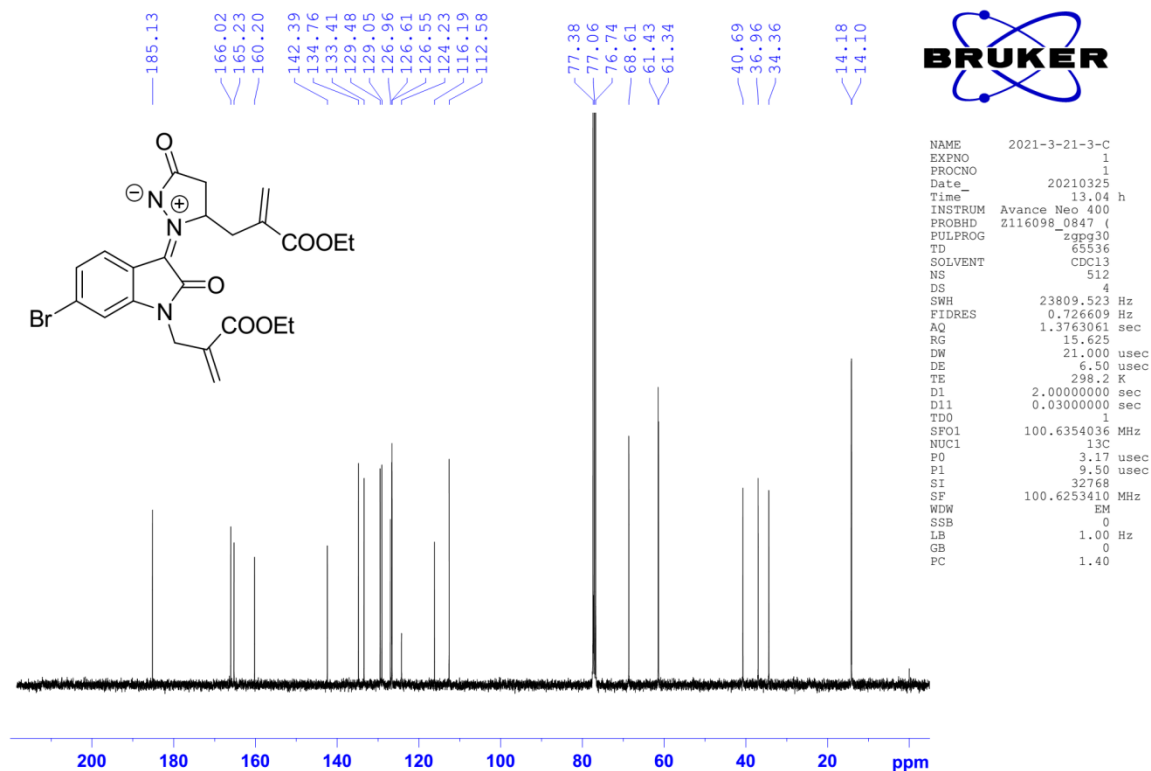




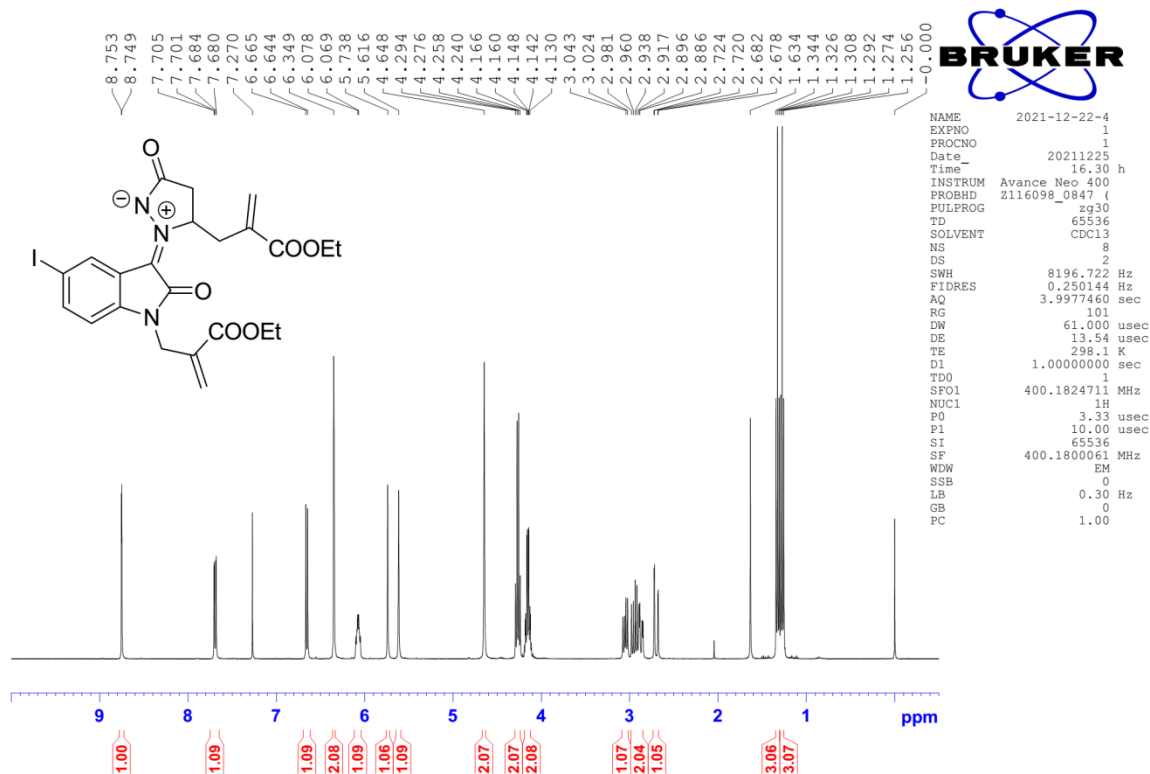
<sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra for Compound 4i

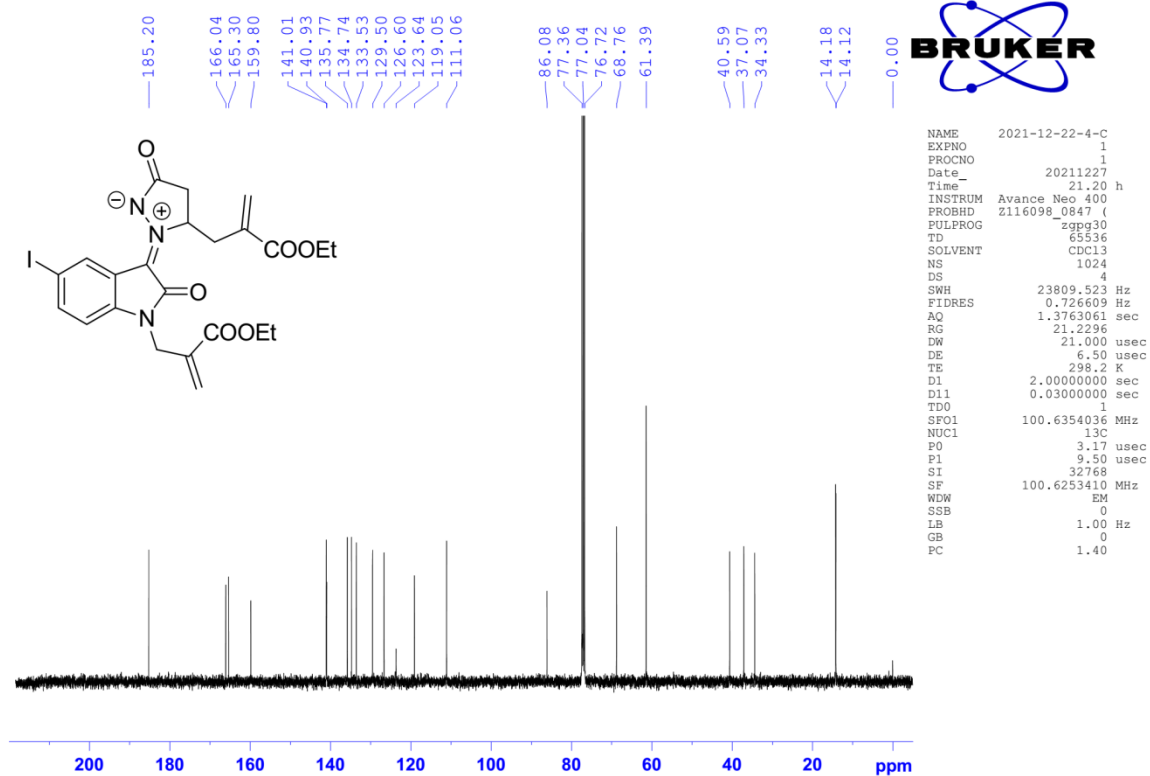




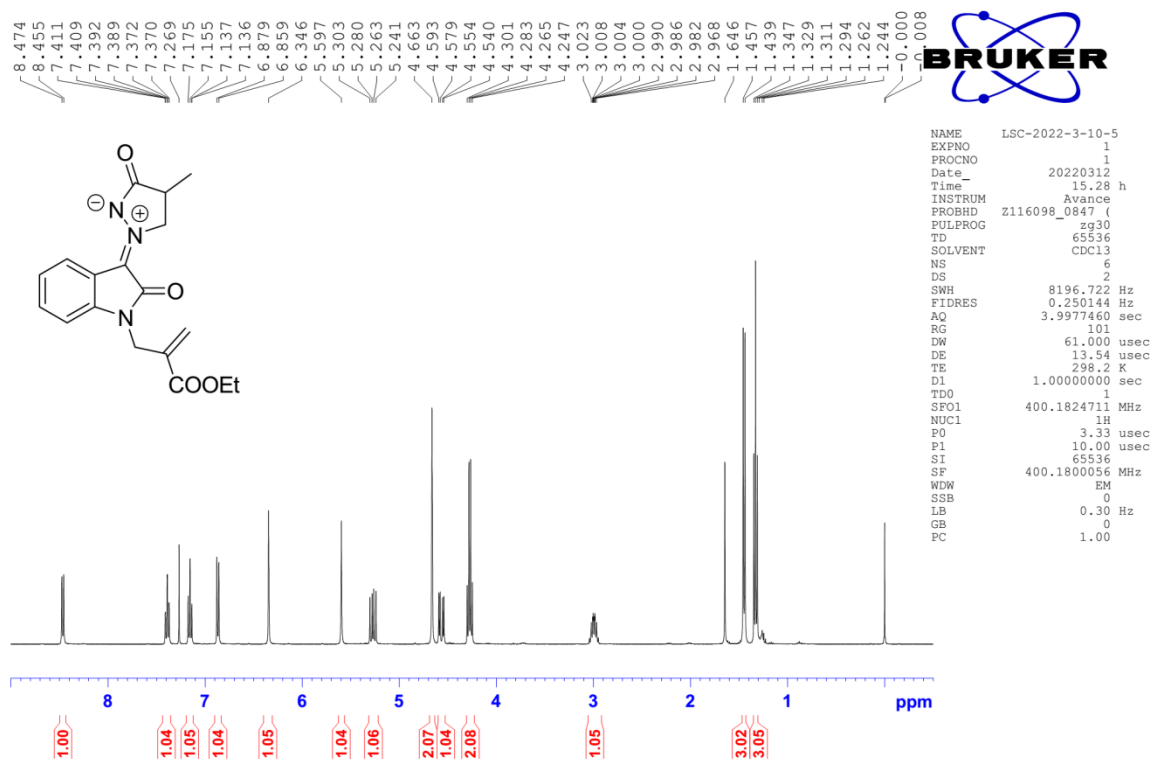


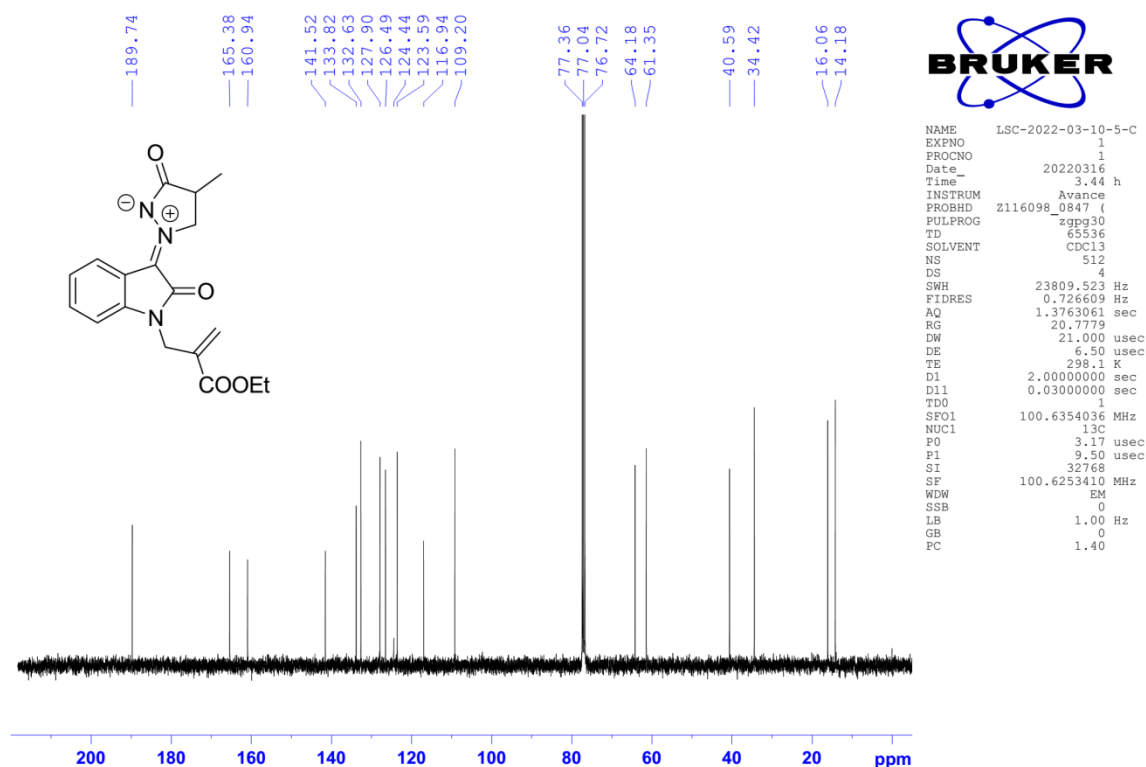
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 4j



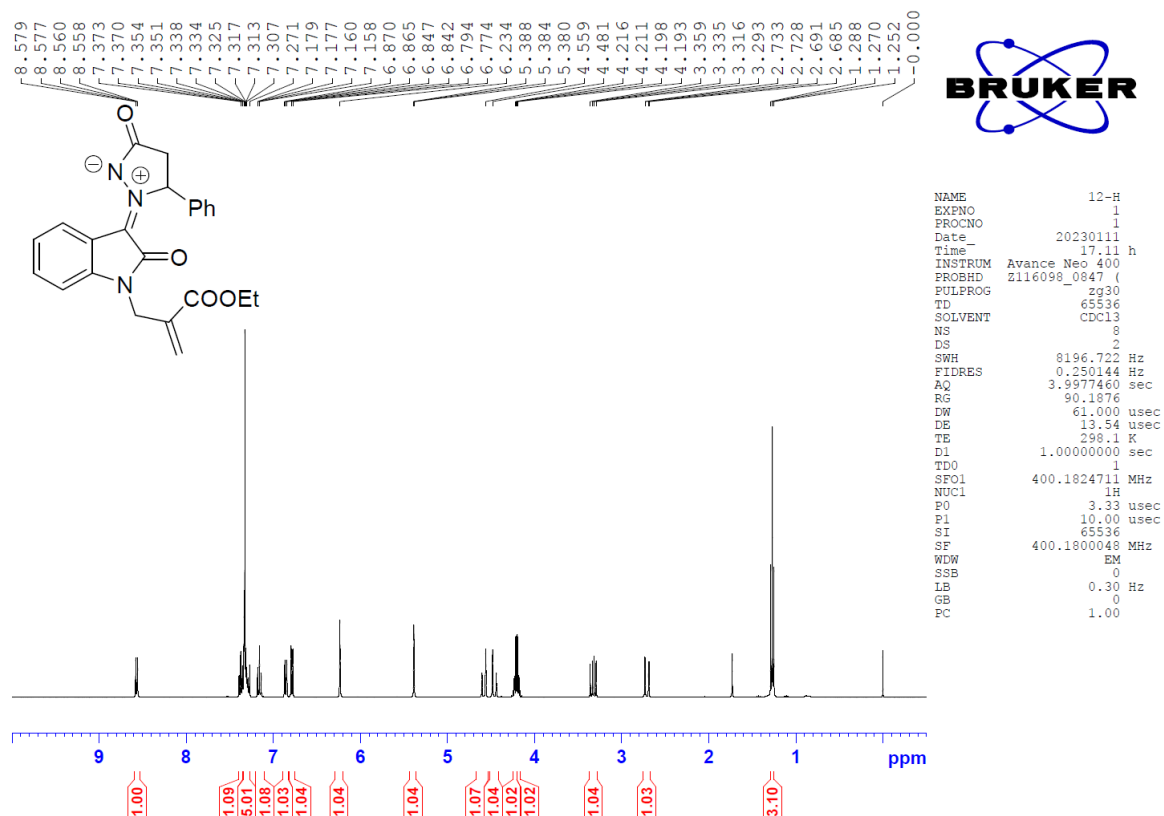


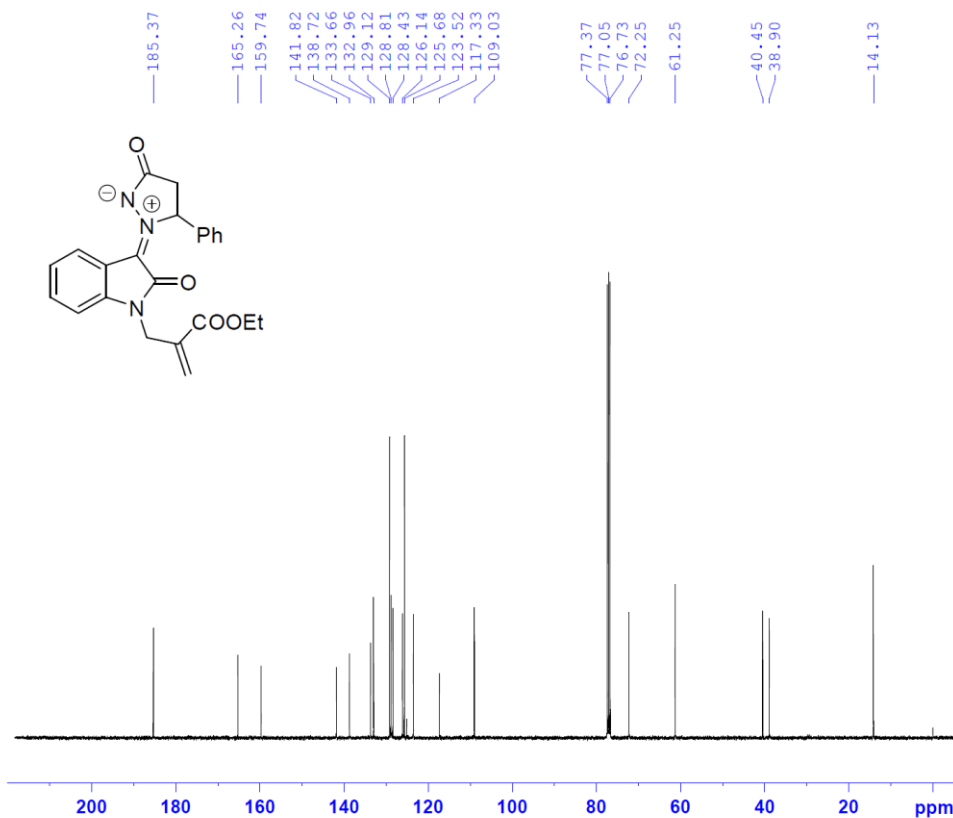
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 10





<sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra for Compound 12

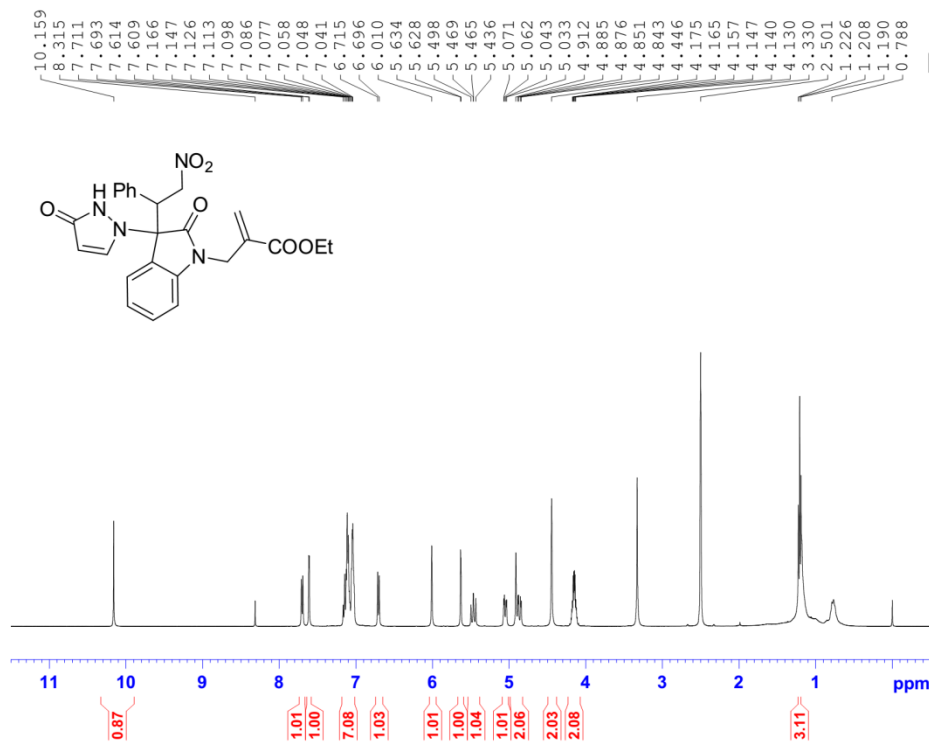




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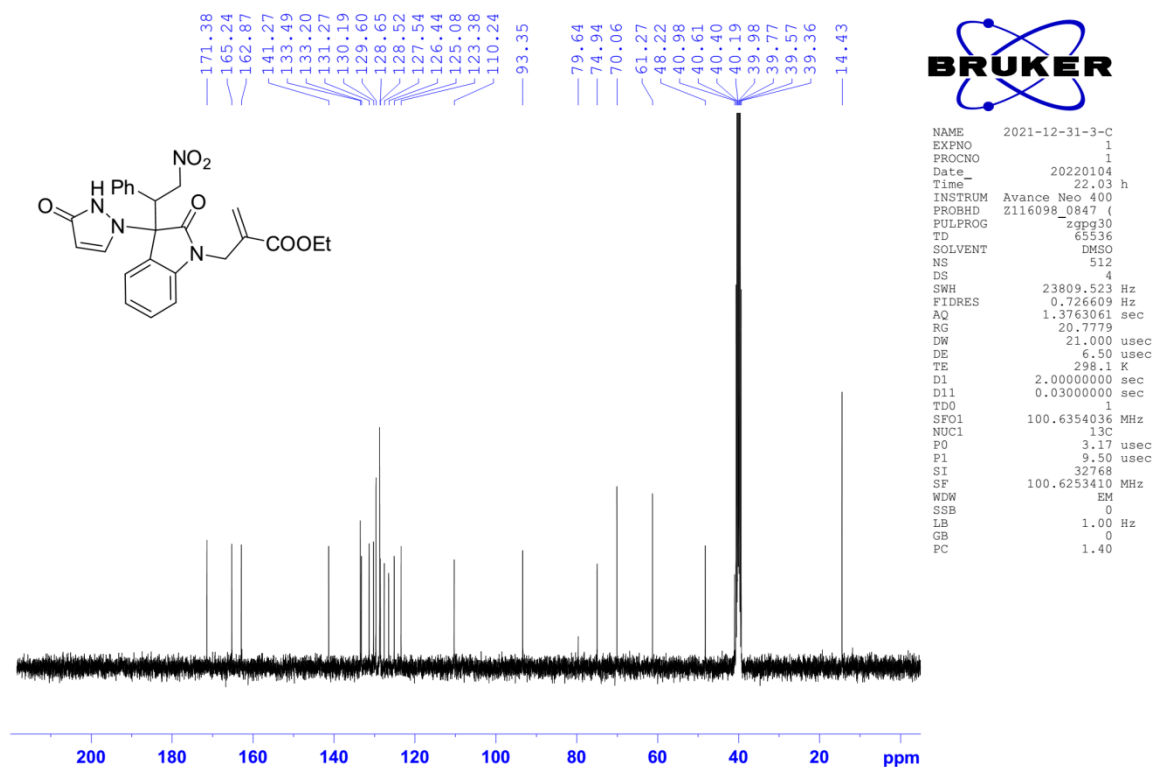
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RG         15.625
DW         21.000 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
SF01       100.6354036 MHz
NUC1       13C
P0         3.33 usec
P1         10.00 usec
SI         32768
SF         100.6253422 MHz
WDW        EM
SSB        0
LB         1.00 Hz
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PC         1.40
  
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<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 13

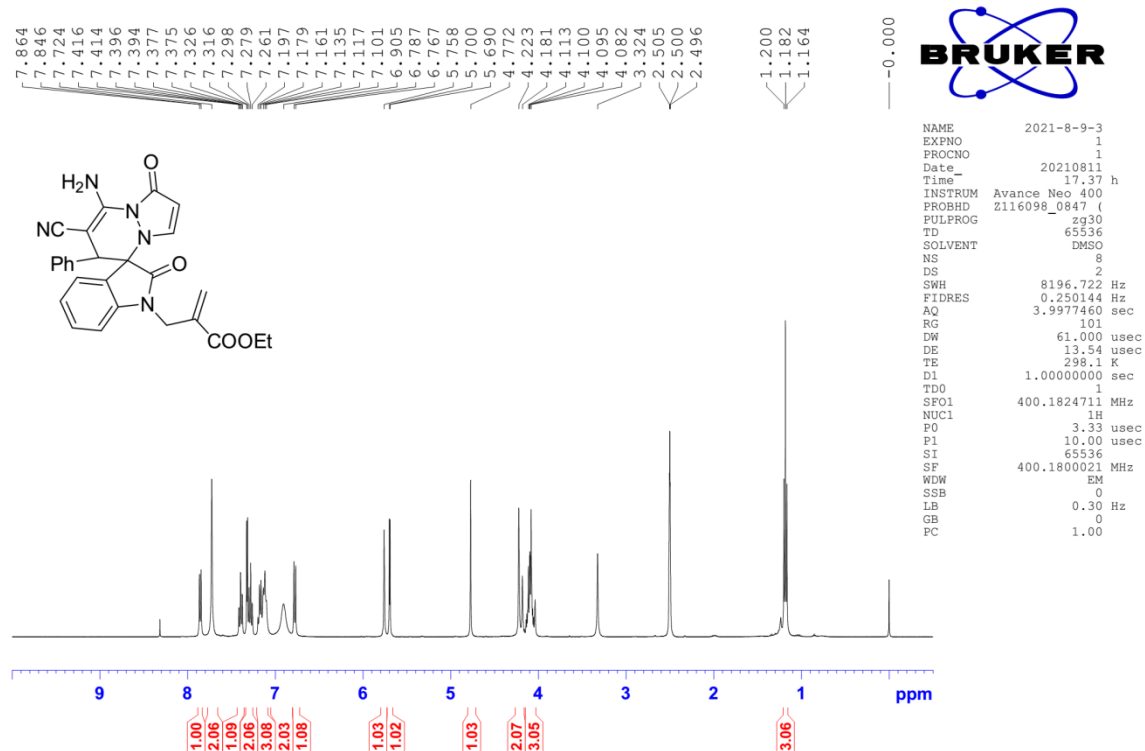


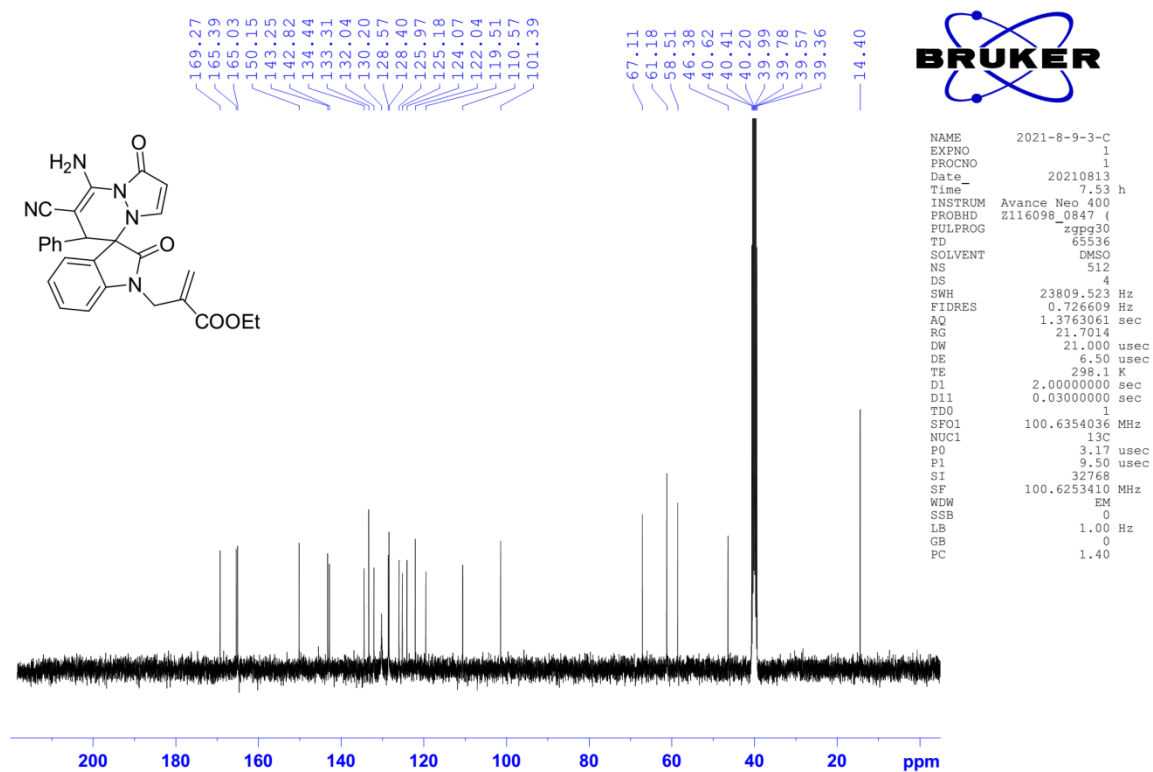
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AQ         3.9977460 sec
RG         99.2064
DW         61.000 usec
DE         13.54 usec
TE         298.1 K
D1         1.00000000 sec
TD0        1
SF01       400.1824711 MHz
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P0         3.33 usec
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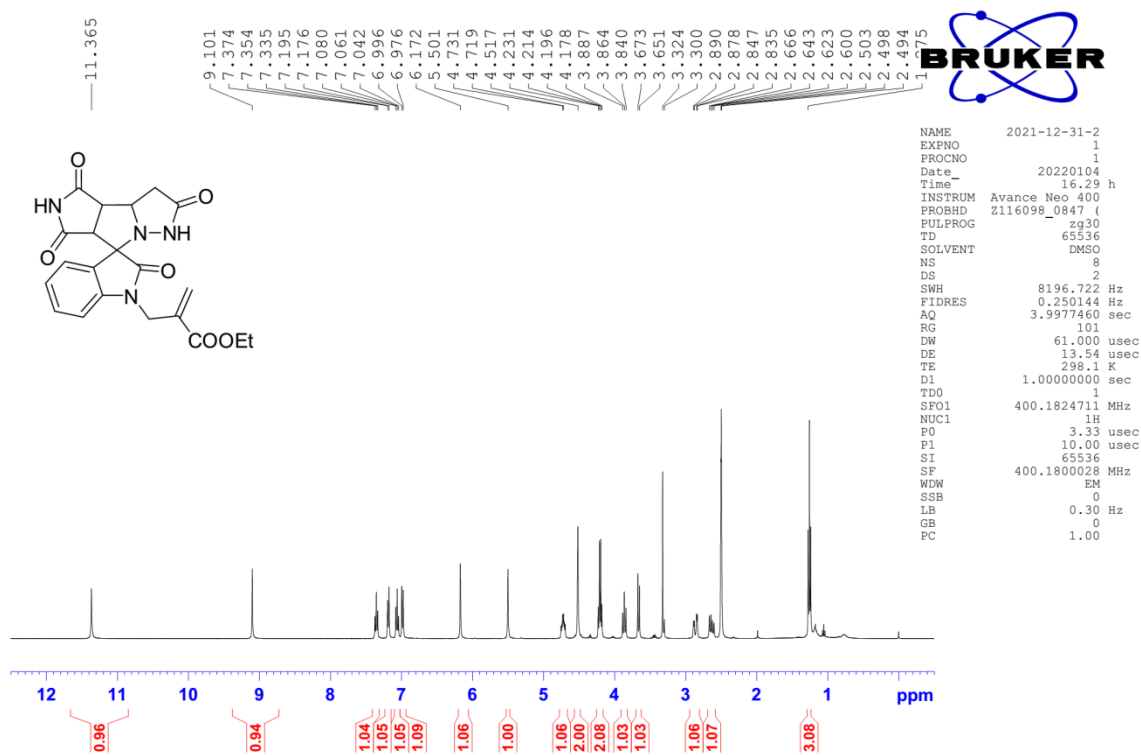


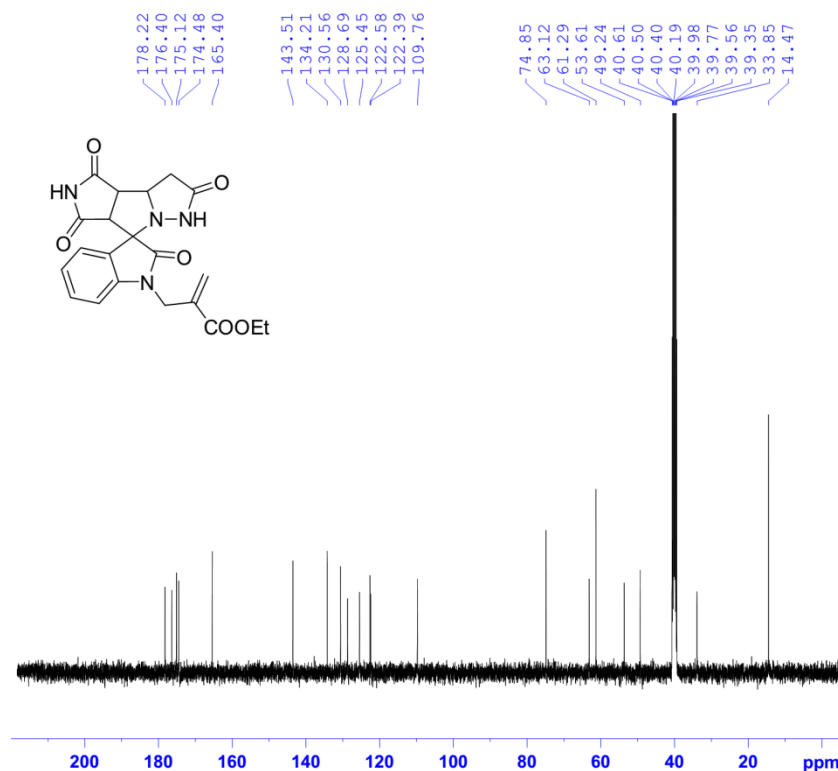
<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 14





<sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compound 15





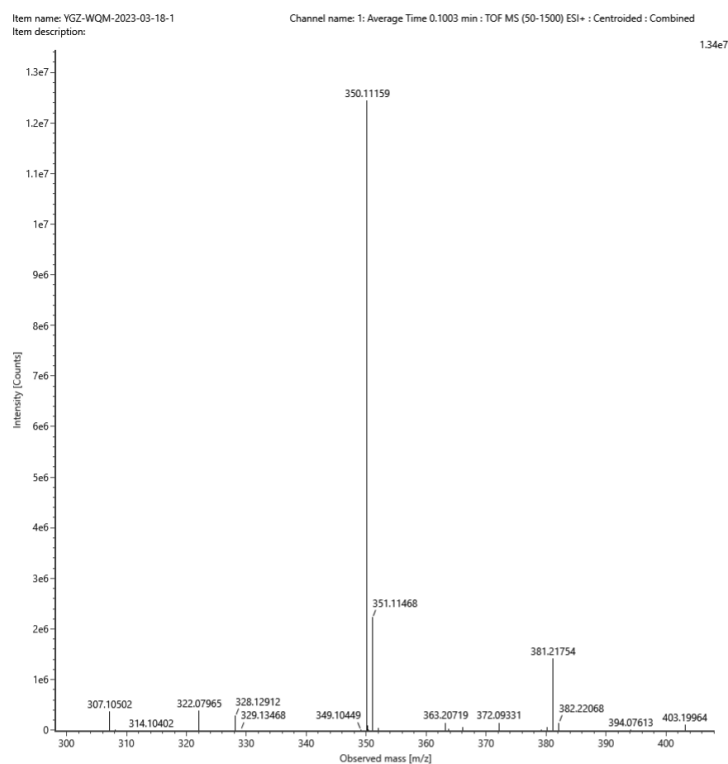
```

NAME      2021-12-31-2-C
EXPNO     1
PROCNO    1
Date_     20220104
Time      22.35 h
INSTRUM   Avance Neo 400
PROBHD    Z116098_0847 (
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         512
DS         4
SWH        23809.523 Hz
FIDRES     0.726609 Hz
AQ         1.3763061 sec
RG         20.3451
DW         21.000 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
SFO1       100.6354036 MHz
NUC1       13C
P0         3.17 usec
PI         9.50 usec
SI         32768
SF         100.6253410 MHz
WDW         EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40

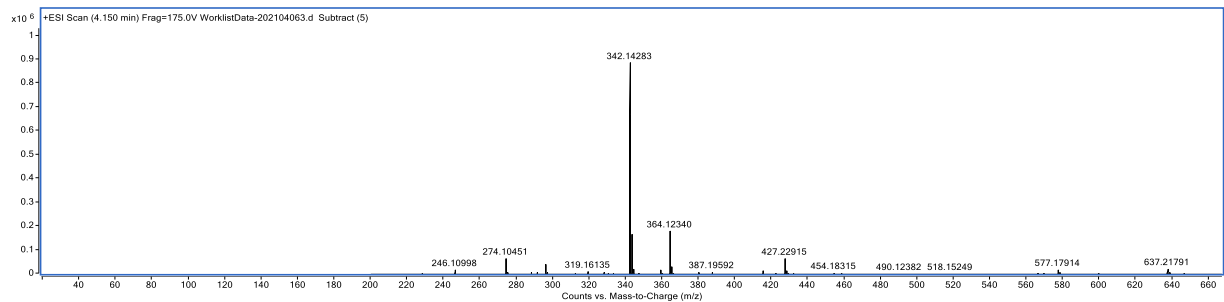
```

#### 4. Copes of HRMS for all new compounds

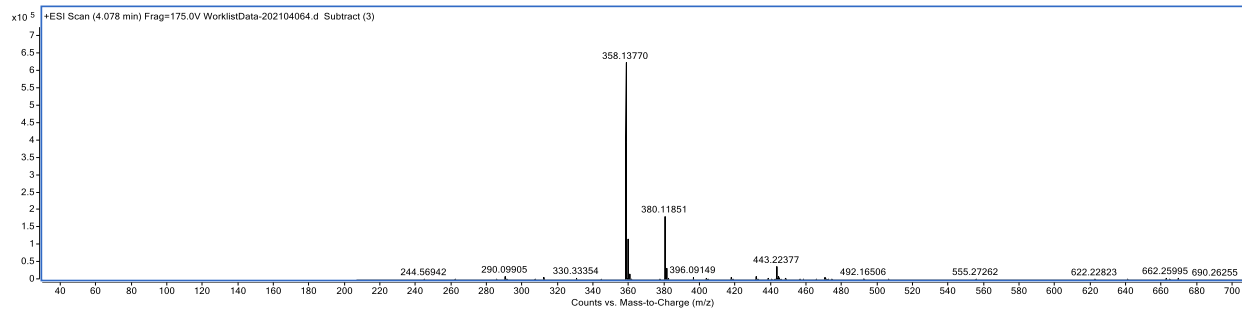
##### HRMS for Compound **3a**



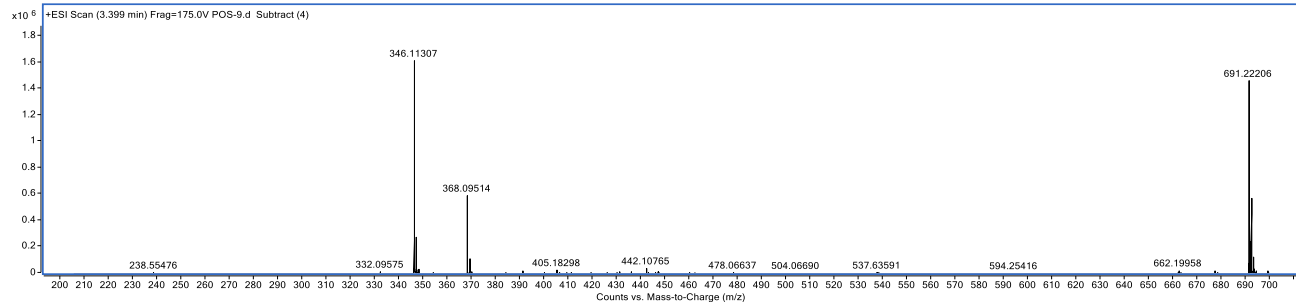
HRMS for Compound **3b**



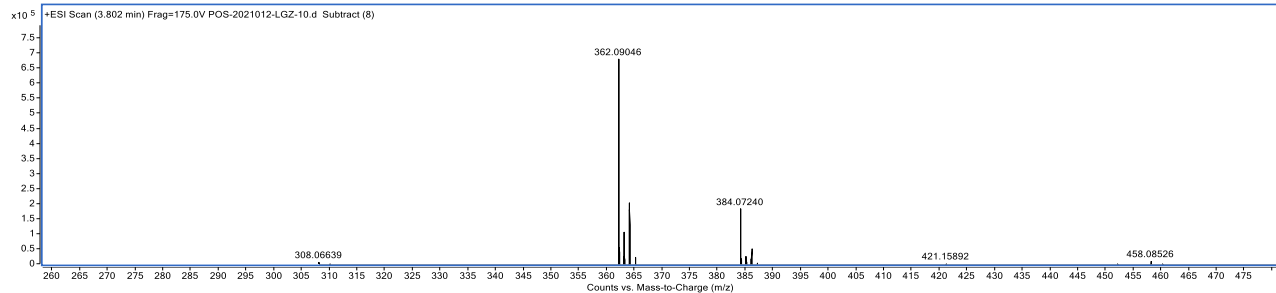
HRMS for Compound **3c**



HRMS for Compound **3d**

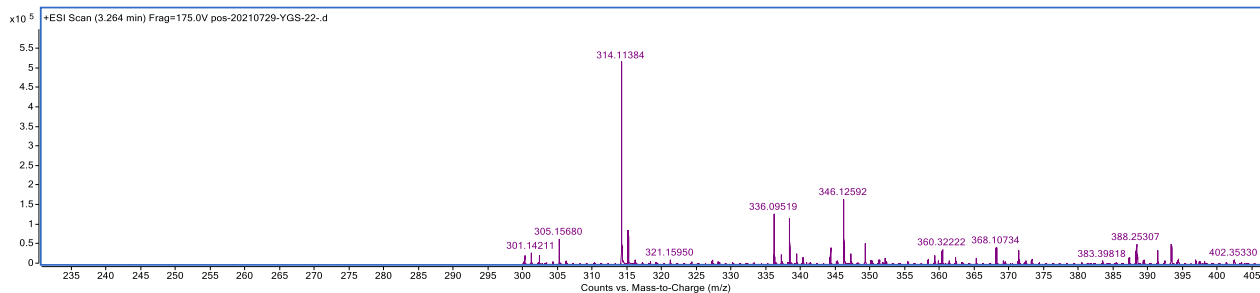
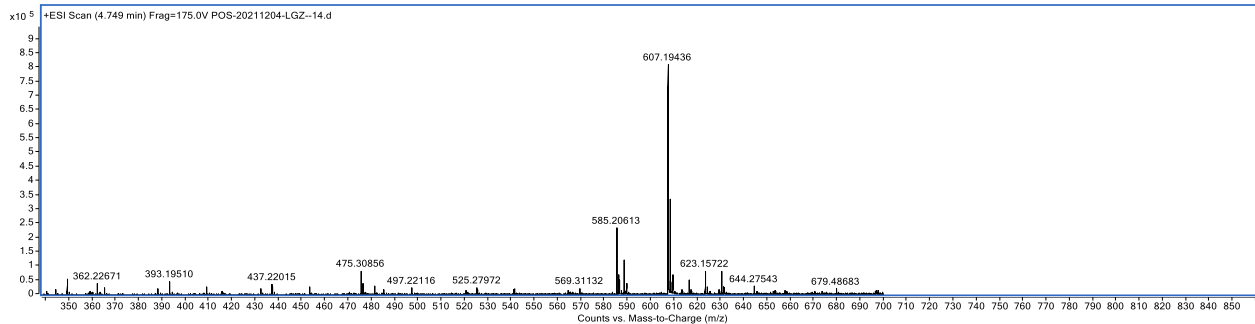
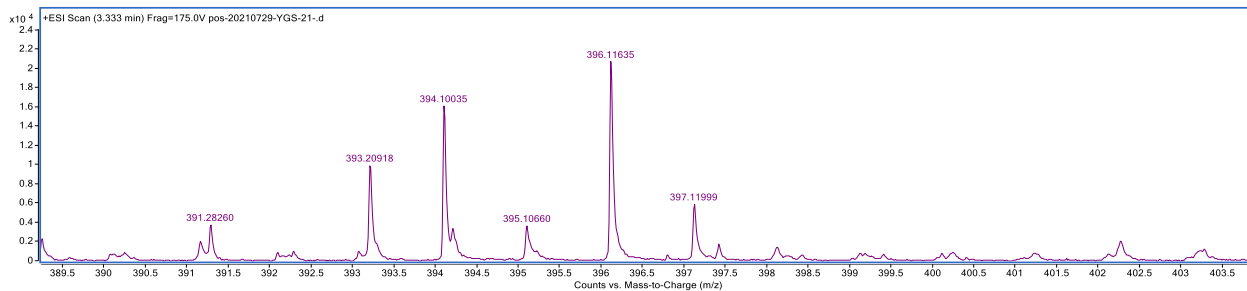
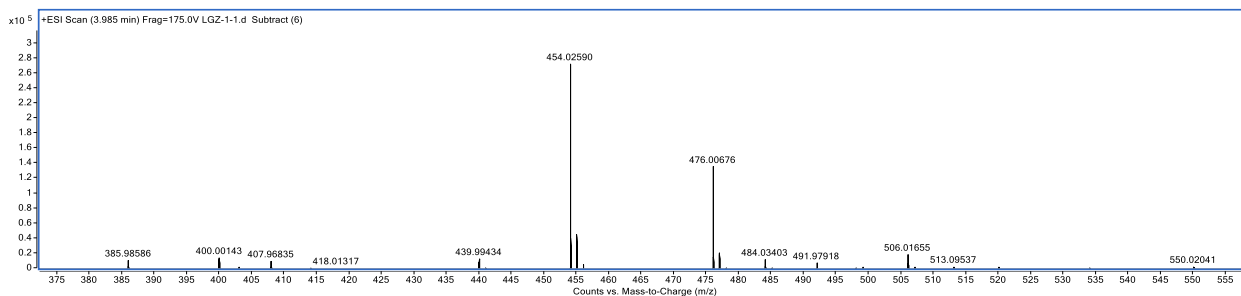
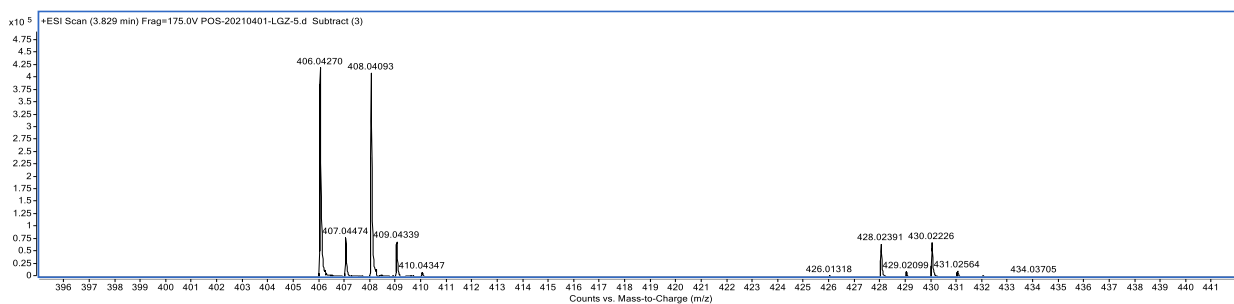


HRMS for Compound **3e**

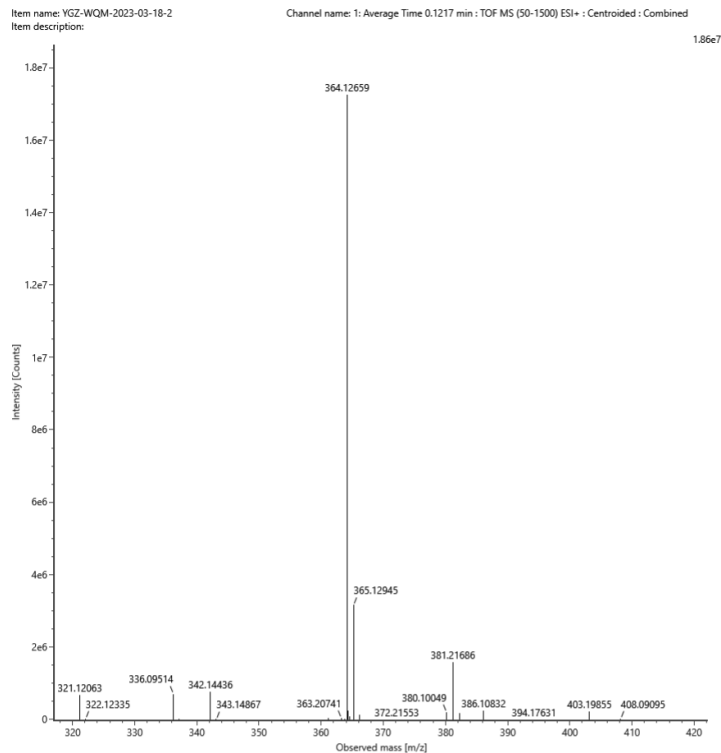


HRMS for Compound **3f**

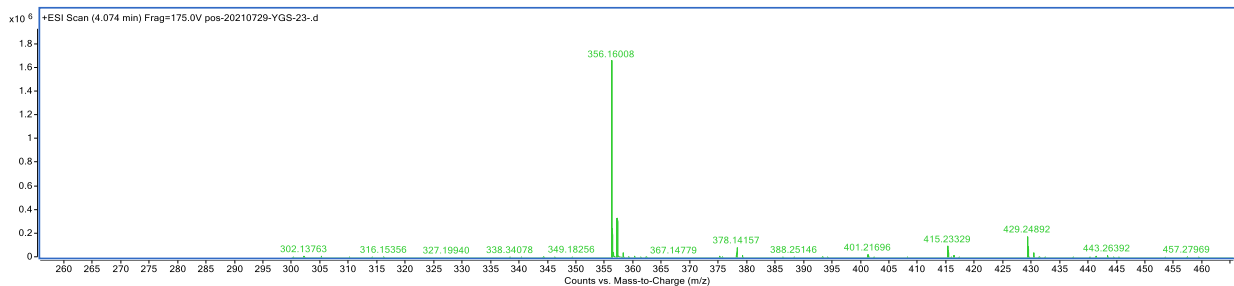




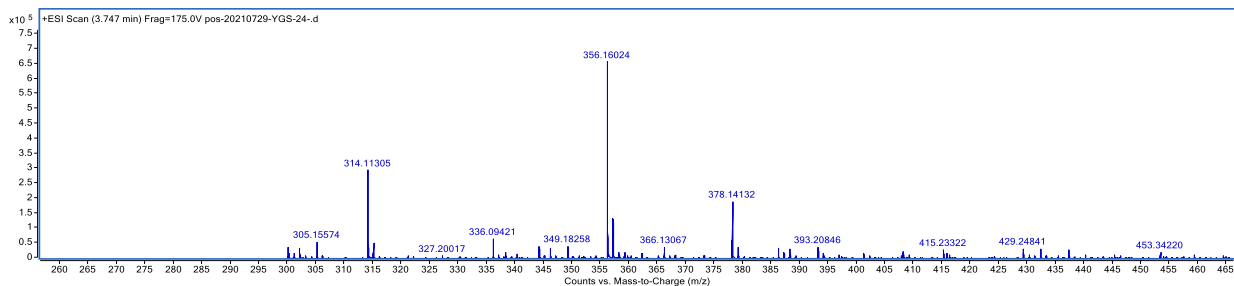
HRMS for Compound **3k**



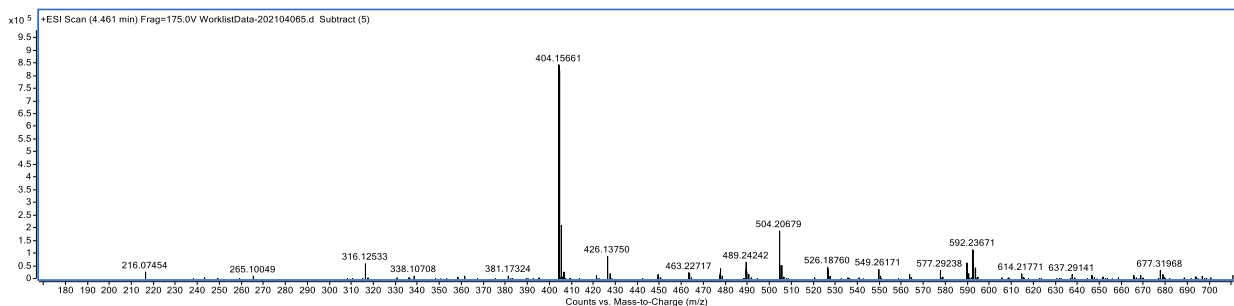
HRMS for Compound **3l**



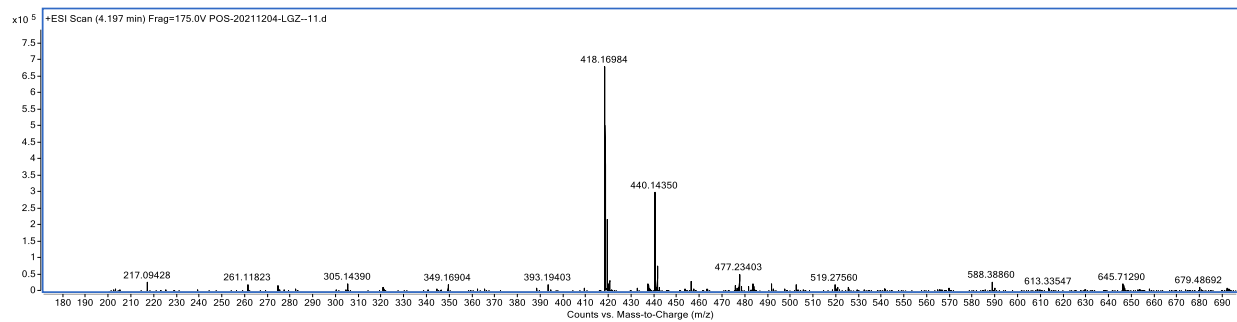
HRMS for Compound **3m**



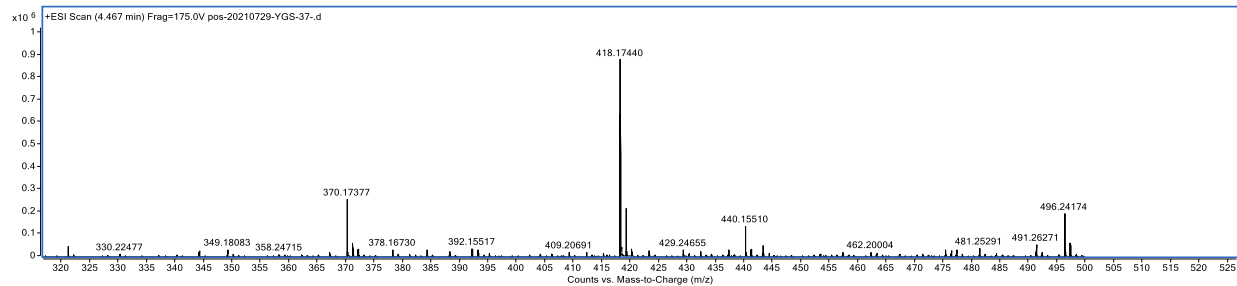
HRMS for Compound **6a**



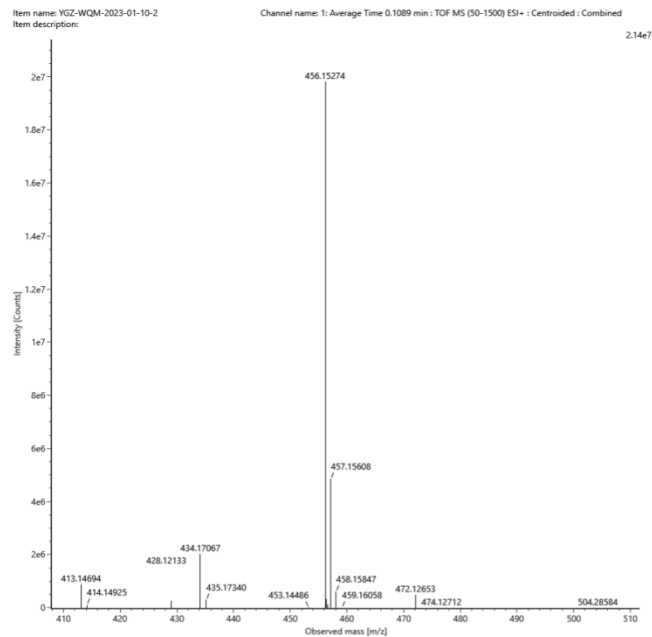
HRMS for Compound **6c**



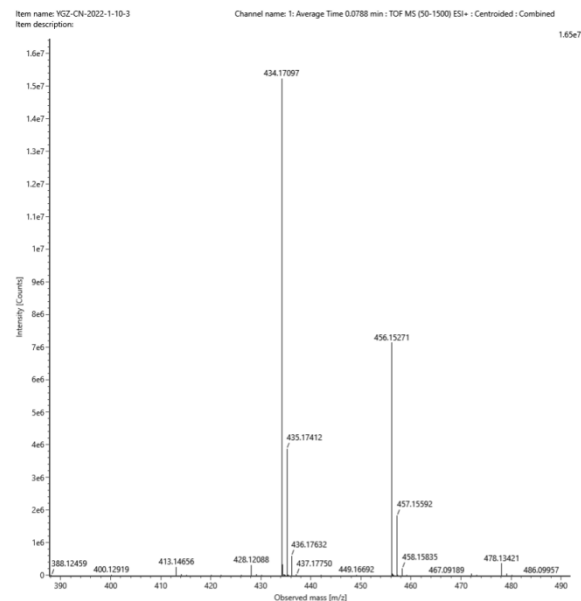
HRMS for Compound **6d**



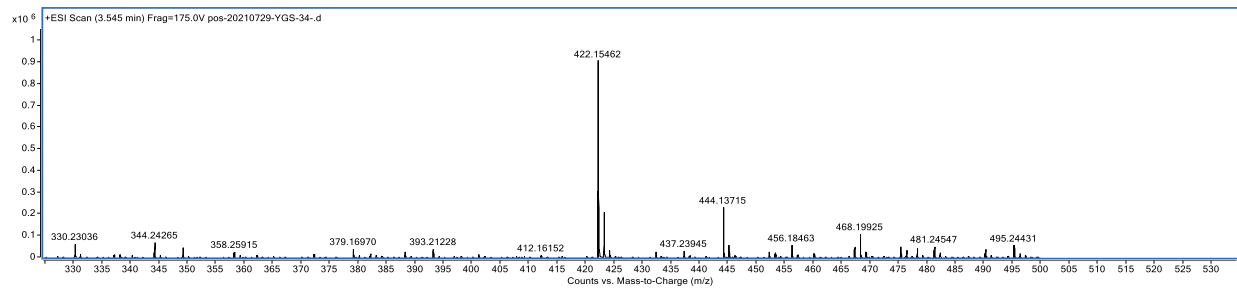
HRMS for Compound **6e**



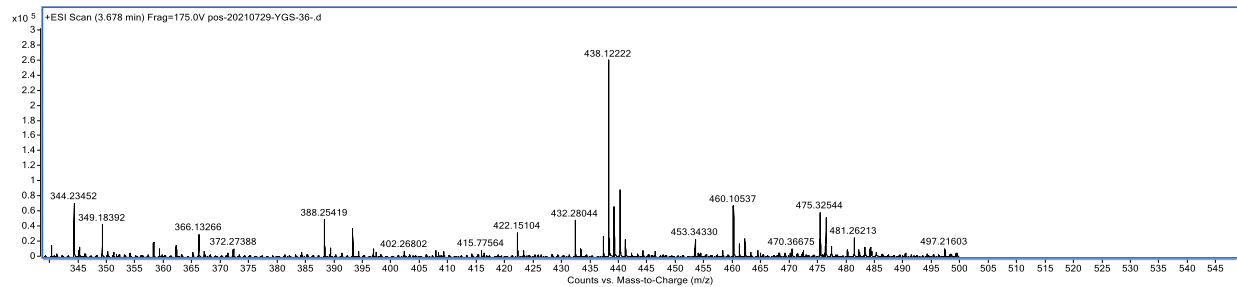
HRMS for Compound **6f**



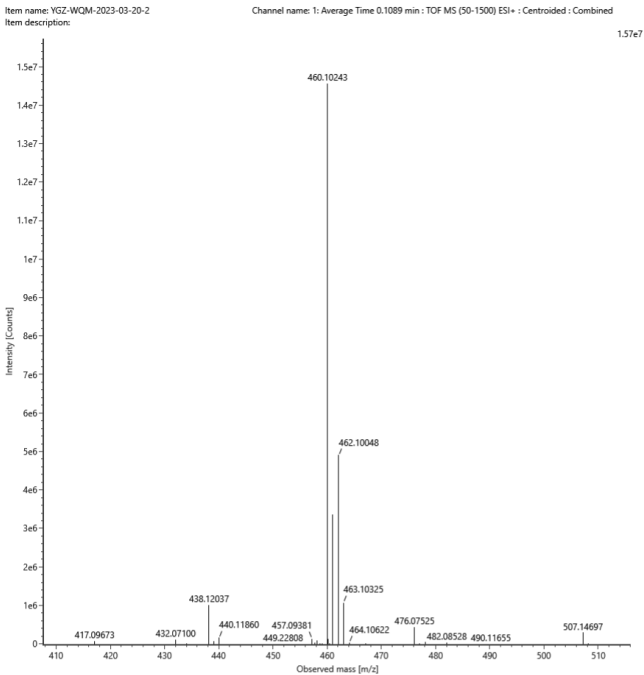
HRMS for Compound **6i**



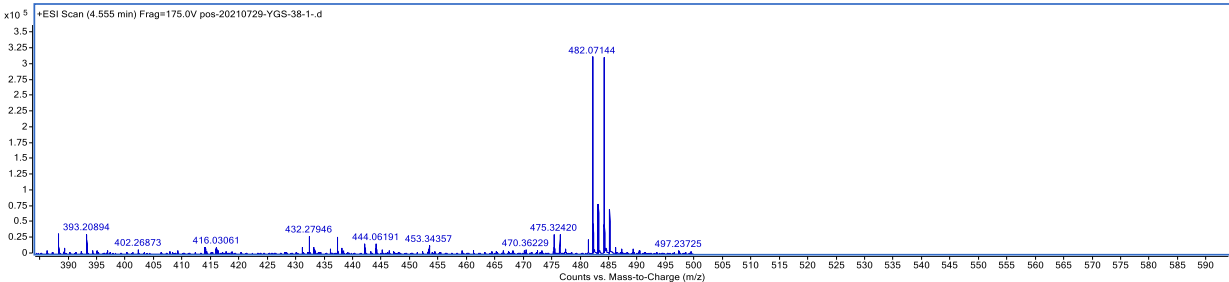
HRMS for Compound **6l**



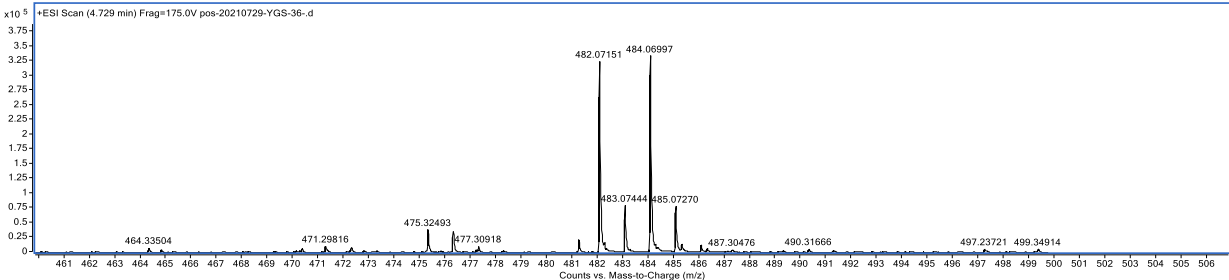
HRMS for Compound **6m**



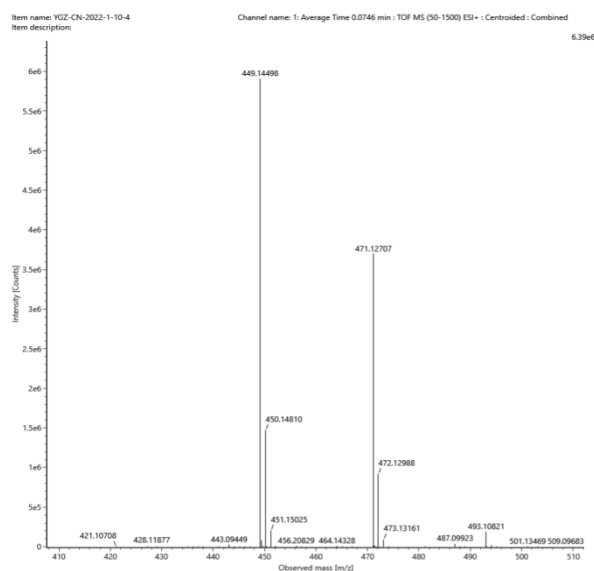
HRMS for Compound **6o**



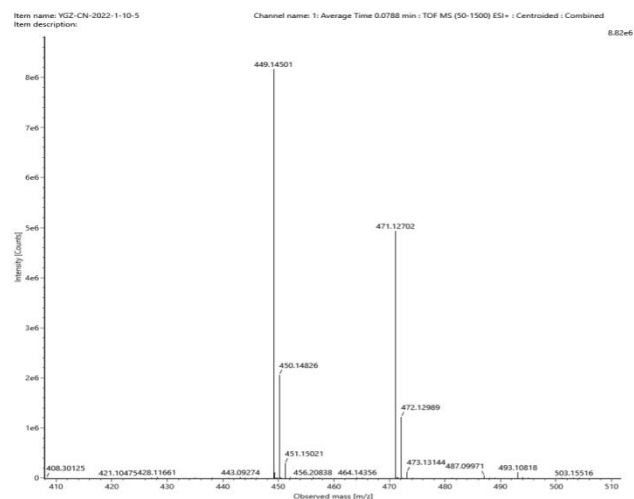
HRMS for Compound **6p**



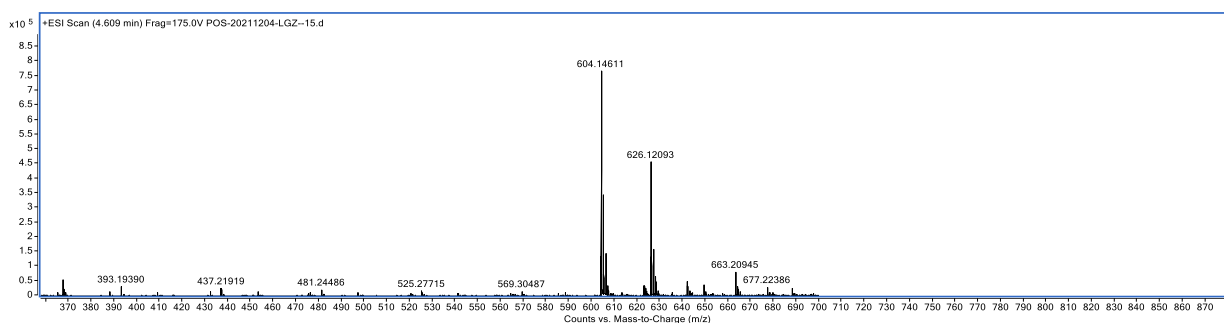
HRMS for Compound **6r**



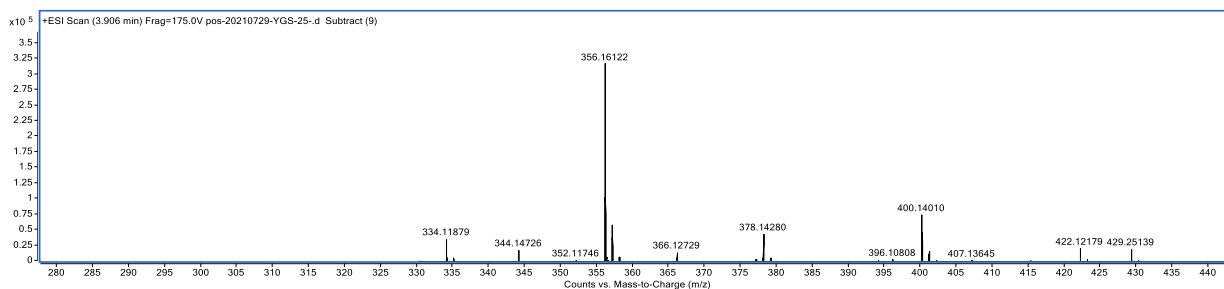
HRMS for Compound **6s**



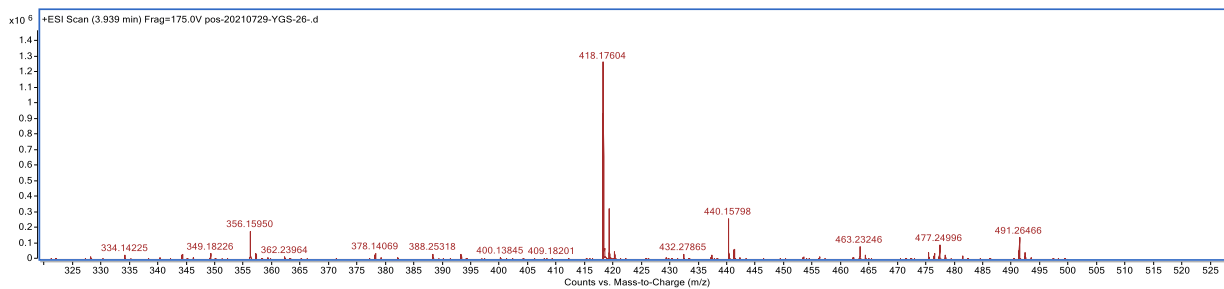
HRMS for Compound **6't**



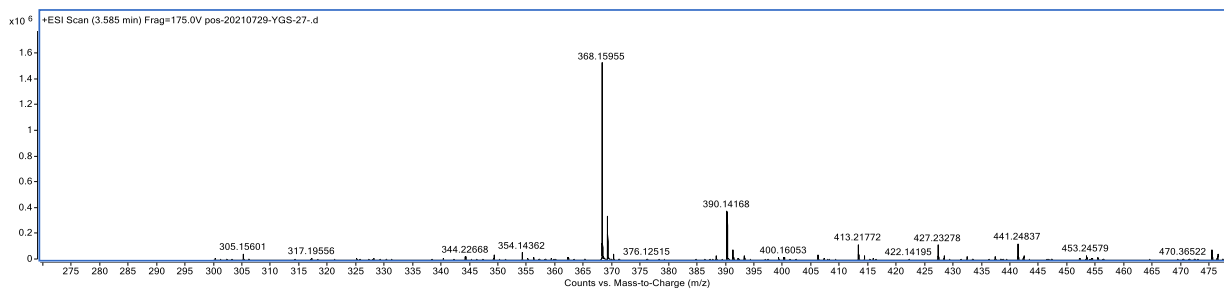
HRMS for Compound **8b**



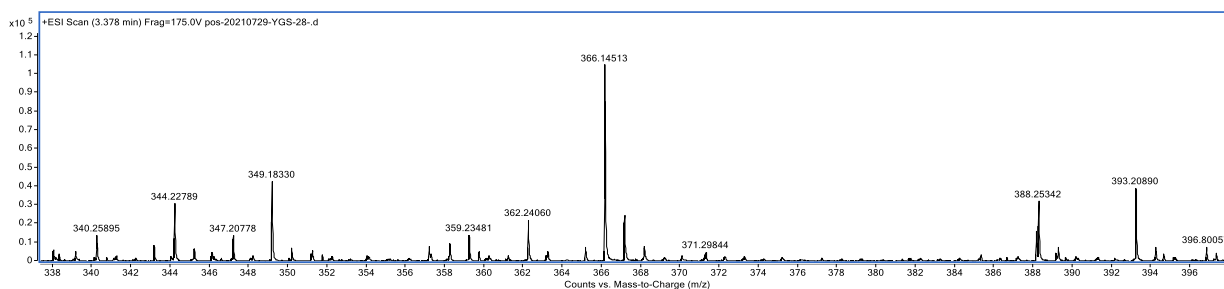
## HRMS for Compound 8c



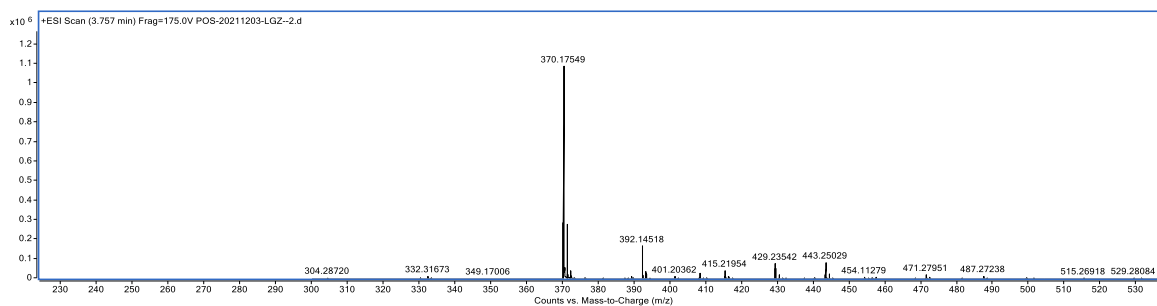
## HRMS for Compound 8d



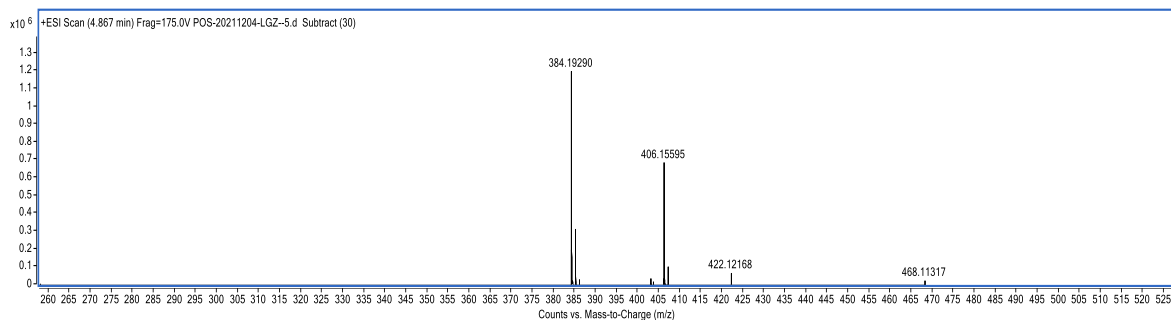
## HRMS for Compound 8e



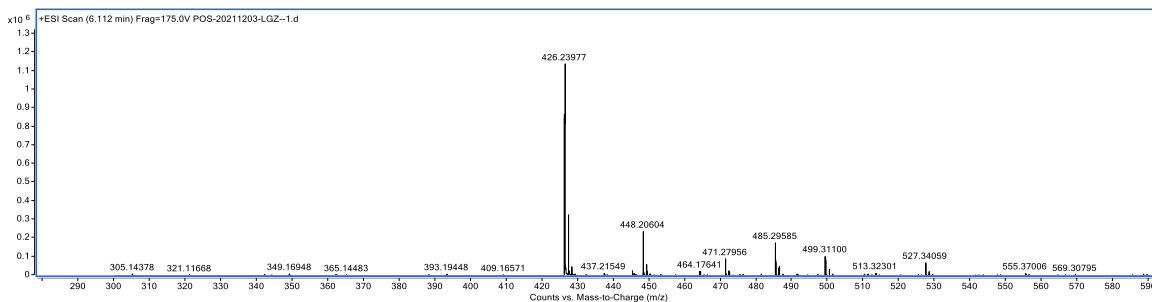
## HRMS for Compound 8f



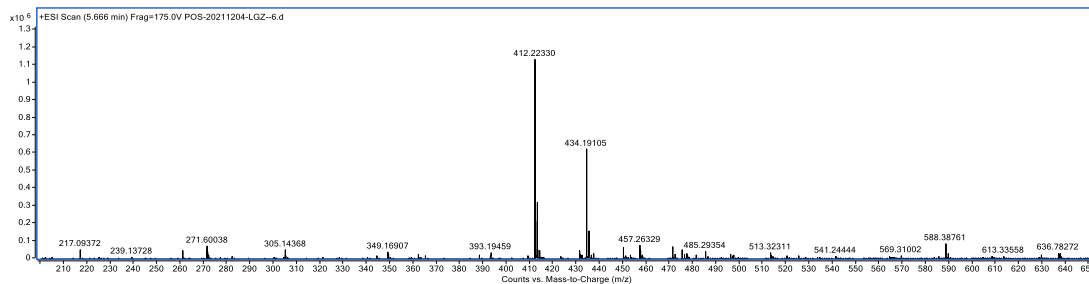
## HRMS for Compound 8g



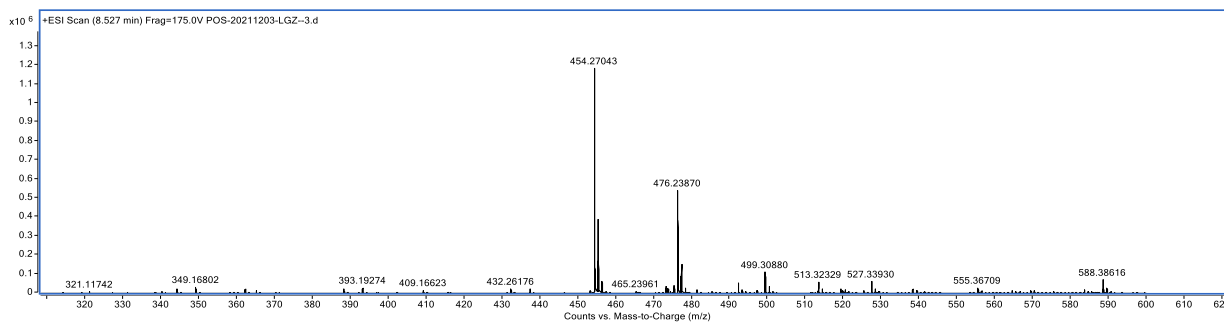
## HRMS for Compound 8h



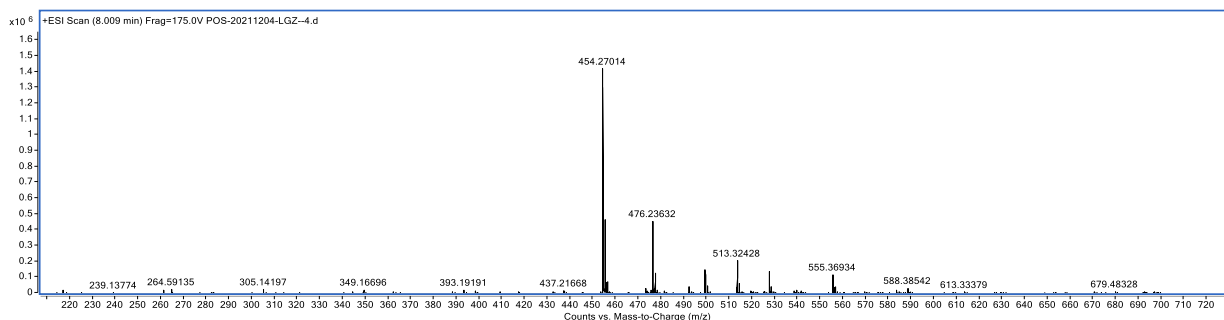
### HRMS for Compound 8i



### HRMS for Compound 8j

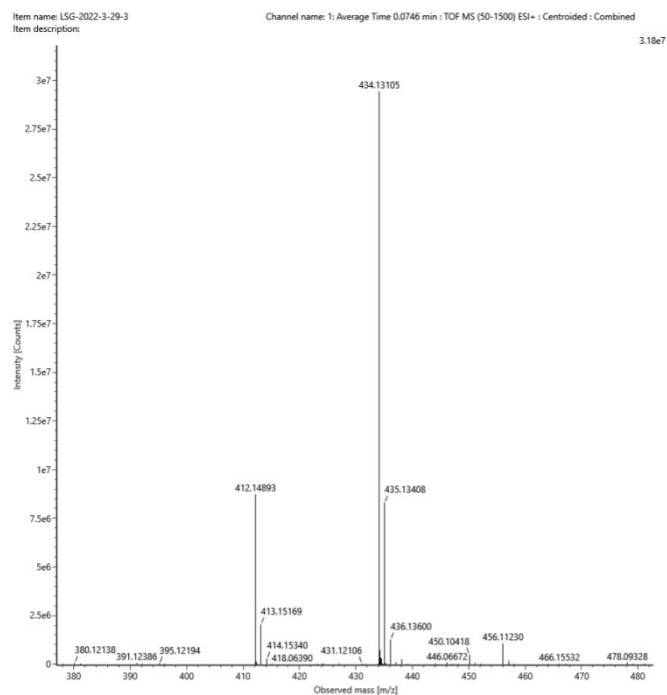


### HRMS for Compound 8k

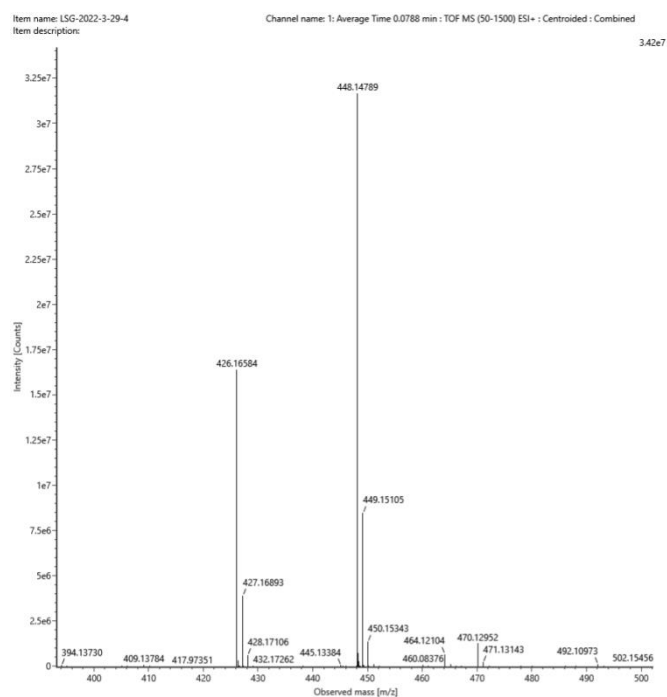


### HRMS for Compound 4a

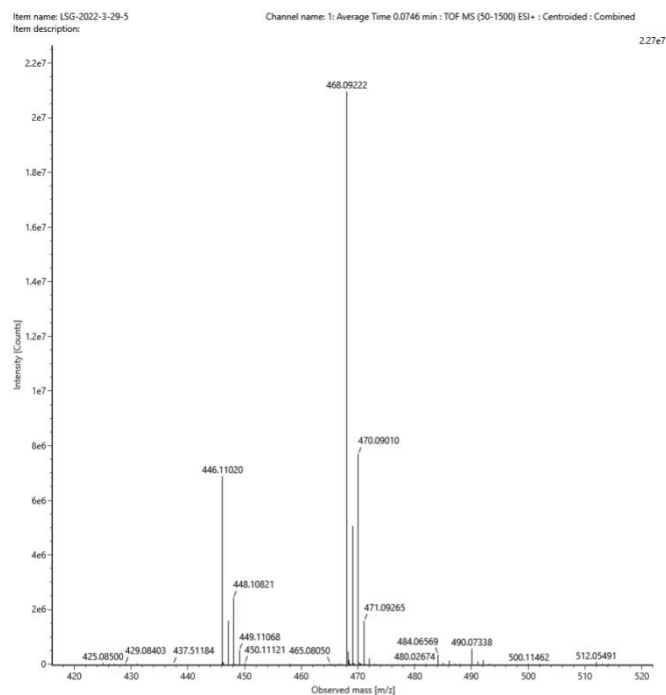




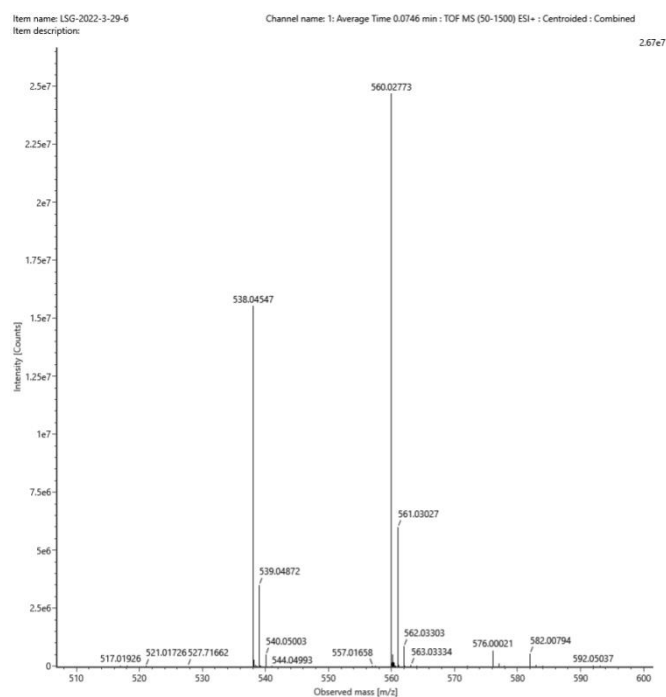
### HRMS for Compound **4b**



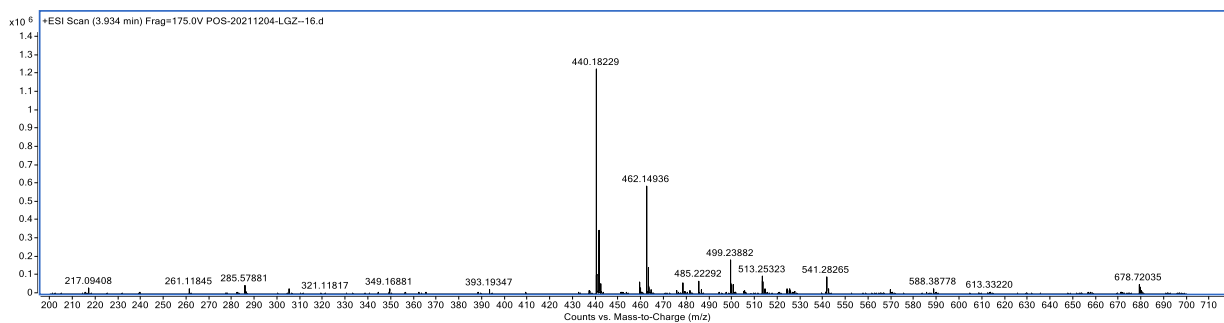
### HRMS for Compound **4c**

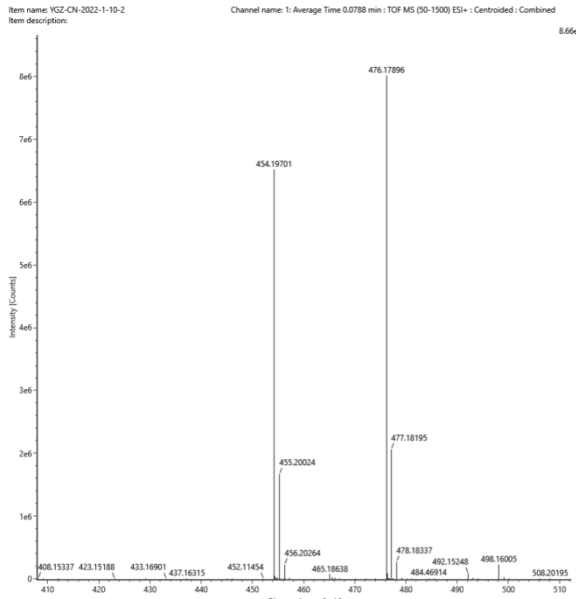


## HRMS for Compound 4d

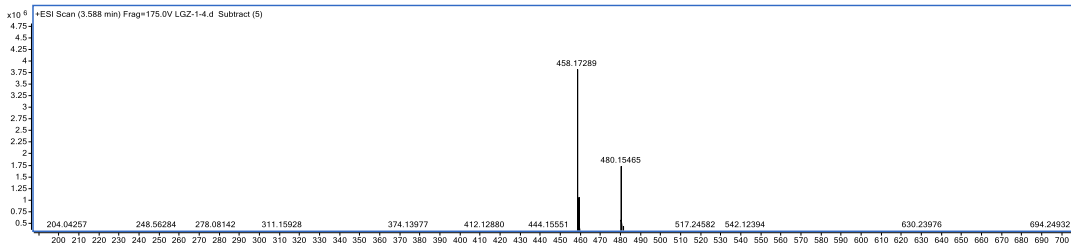
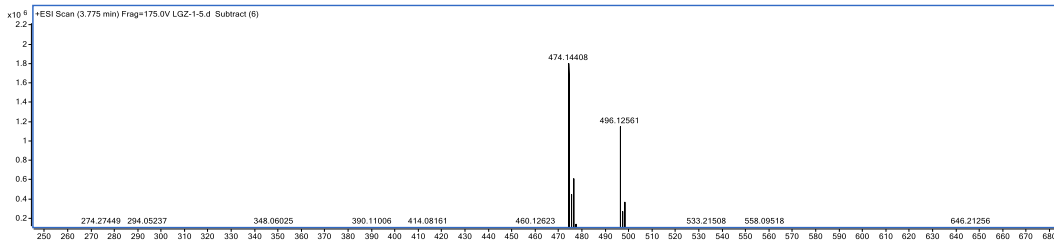
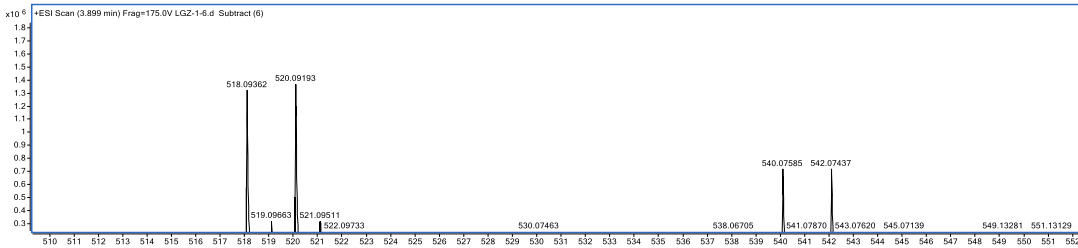


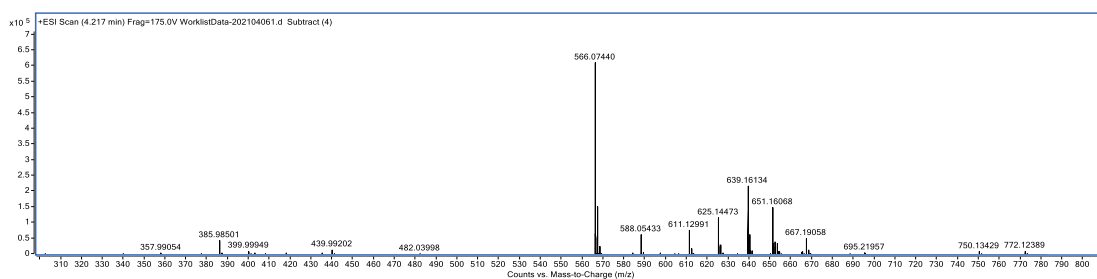
## HRMS for Compound 4e



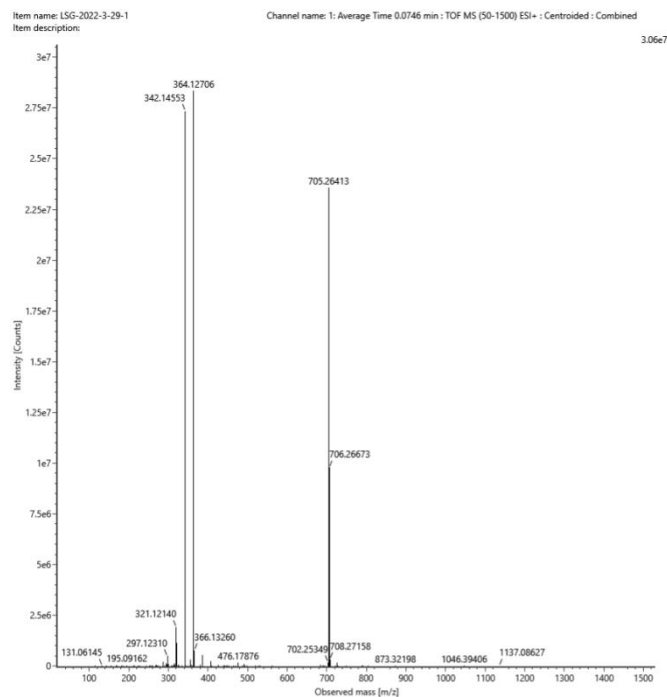
HRMS for Compound **4f**

## HRMS for Compound 4g

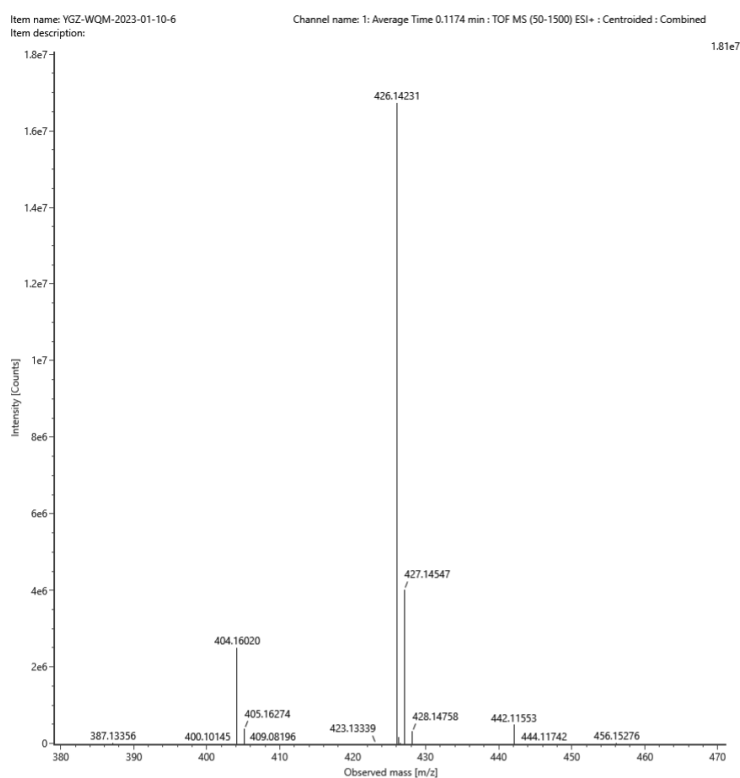
HRMS for Compound **4h**HRMS for Compound **4i**HRMS for Compound **4j**



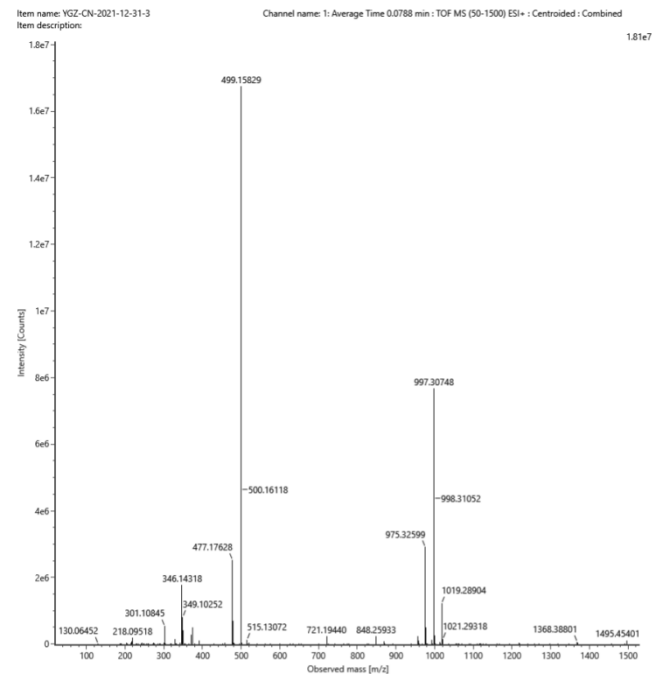
## HRMS for Compound 10



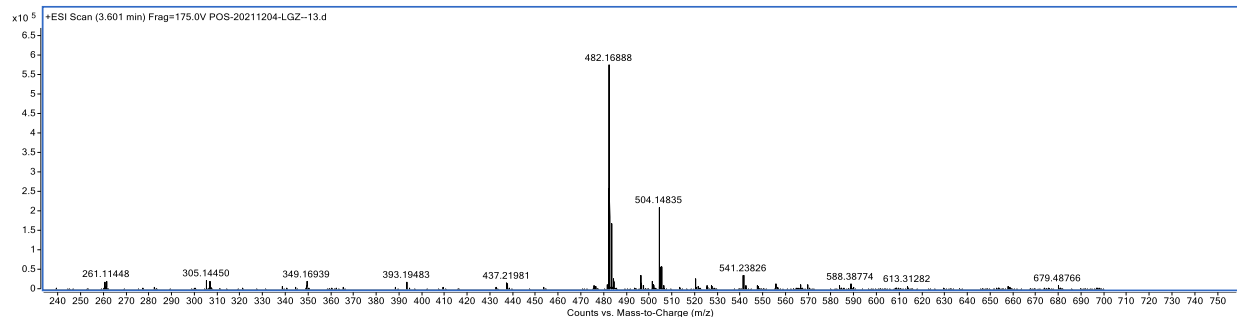
## HRMS for Compound 12



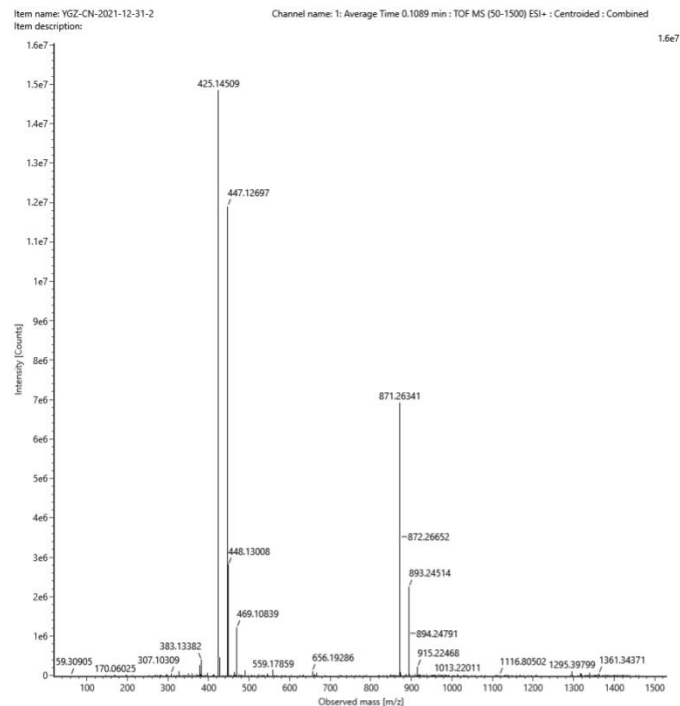
HRMS for Compound 13



HRMS for Compound 14

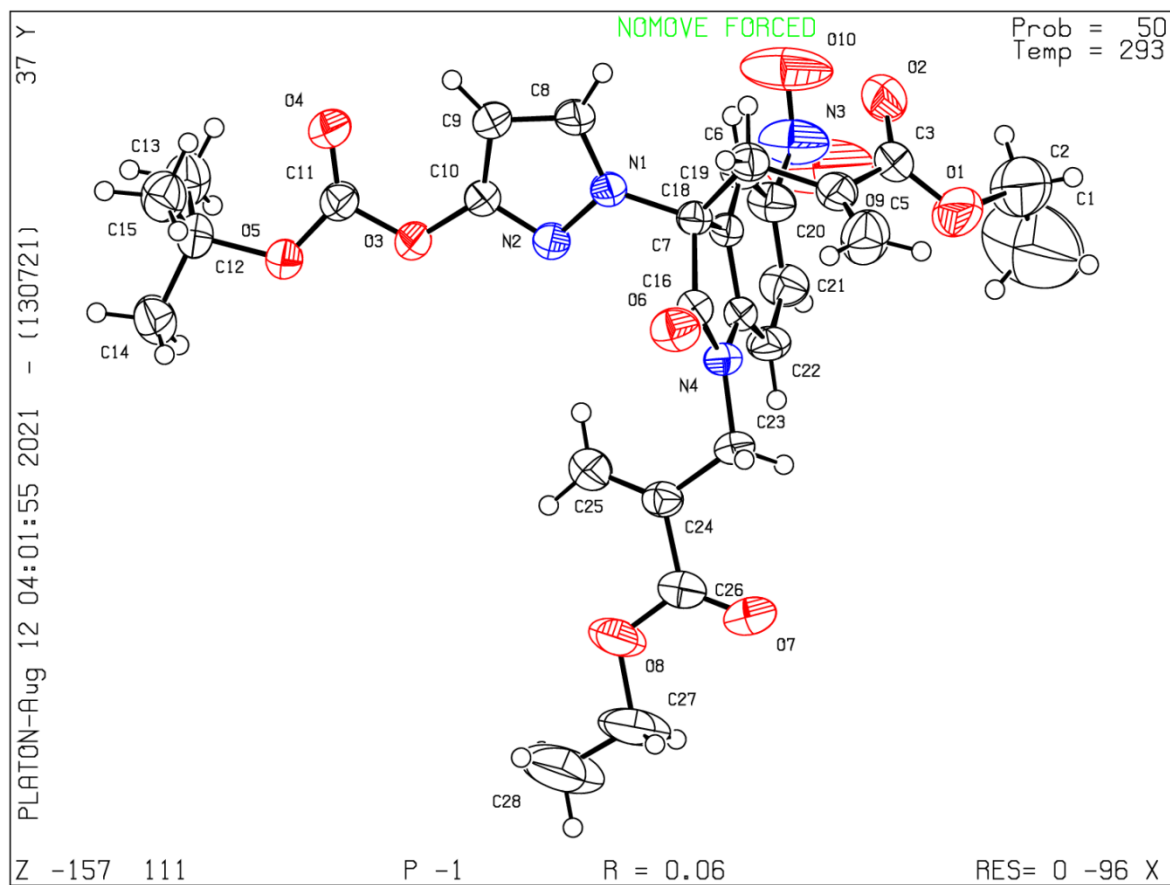


HRMS for Compound 15



## 5. Copes of Data of X-ray crystal structure for 3'i (CCDC 2108657).

Datablock 111 - ellipsoid plot



## checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found.      CIF dictionary      Interpreting this report

### Datablock: 111

---

|                 |                  |                                       |
|-----------------|------------------|---------------------------------------|
| Bond precision: | C-C = 0.0034 Å   | Wavelength=0.71073                    |
| Cell:           | a=10.738 (2)     | b=10.755 (2)      c=14.284 (3)        |
|                 | alpha=67.993 (6) | beta=88.754 (8)      gamma=79.339 (6) |
| Temperature:    | 293 K            |                                       |
|                 | Calculated       | Reported                              |
| Volume          | 1501.0 (5)       | 1501.0 (5)                            |
| Space group     | P -1             | P -1                                  |
| Hall group      | -P 1             | -P 1                                  |
| Moiety formula  | C28 H32 N4 O10   | C28 H32 N4 O10                        |
| Sum formula     | C28 H32 N4 O10   | C28 H32 N4 O10                        |
| Mr              | 584.58           | 584.57                                |
| Dx, g cm-3      | 1.293            | 1.293                                 |
| Z               | 2                | 2                                     |
| Mu (mm-1)       | 0.099            | 0.099                                 |
| F000            | 616.0            | 616.0                                 |
| F000'           | 616.34           |                                       |
| h,k,lmax        | 14,14,18         | 14,14,18                              |
| Nref            | 7035             | 6994                                  |
| Tmin,Tmax       | 0.965,0.981      | 0.708,0.746                           |
| Tmin'           | 0.965            |                                       |

Correction method= # Reported T Limits: Tmin=0.708 Tmax=0.746  
AbsCorr = MULTI-SCAN

Data completeness= 0.994      Theta(max)= 27.728

R(reflections)= 0.0601( 4671)      wR2(reflections)= 0.1755( 6994)

S = 1.027      Npar= 384

---

The following ALERTS were generated. Each ALERT has the format  
**test-name\_ALERT\_alert-type\_alert-level.**  
Click on the hyperlinks for more details of the test.

## checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found.      CIF dictionary      Interpreting this report

### Datablock: 2\_sq

---

|                 |                                 |                    |              |
|-----------------|---------------------------------|--------------------|--------------|
| Bond precision: | C-C = 0.0094 A                  | Wavelength=0.71073 |              |
| Cell:           | a=8.1785 (14)                   | b=25.516 (4)       | c=12.111 (2) |
|                 | alpha=90                        | beta=90            | gamma=90     |
| Temperature:    | 273 K                           |                    |              |
|                 | Calculated                      | Reported           |              |
| Volume          | 2527.4 (7)                      | 2527.3 (7)         |              |
| Space group     | P n a 21                        | P n a 21           |              |
| Hall group      | P 2c -2n                        | P 2c -2n           |              |
| Moiety formula  | C26 H20 Br N3 O3 [+<br>solvent] | C26 H20 Br N3 O3   |              |
| Sum formula     | C26 H20 Br N3 O3 [+<br>solvent] | C26 H20 Br N3 O3   |              |
| Mr              | 502.35                          | 502.36             |              |
| Dx, g cm-3      | 1.320                           | 1.320              |              |
| Z               | 4                               | 4                  |              |
| Mu (mm-1)       | 1.657                           | 1.657              |              |
| F000            | 1024.0                          | 1024.0             |              |
| F000'           | 1023.25                         |                    |              |
| h,k,lmax        | 10,33,15                        | 10,33,15           |              |
| Nref            | 5758 [ 3016]                    | 5674               |              |
| Tmin,Tmax       | 0.667,0.806                     | 0.410,0.746        |              |
| Tmin'           | 0.654                           |                    |              |

Correction method= # Reported T Limits: Tmin=0.410 Tmax=0.746  
AbsCorr = MULTI-SCAN

Data completeness= 1.88/0.99      Theta(max)= 27.413

R(reflections)= 0.0573 ( 3876)      wR2(reflections)= 0.1471 ( 5674)

S = 1.017      Npar= 298

---



|  |  |                            |                                 |                     |       |      |        |
|--|--|----------------------------|---------------------------------|---------------------|-------|------|--------|
| <b>Alert level B</b>   |  |                            |                                 |                     |       |      |        |
| PLAT220_ALERT_2_B  | NonSolvent                             | Resd 1                     | C                               | Ueq(max)/Ueq(min)   | Range | 7.1  | Ratio  |
| <b>Alert level C</b>   |  |                            |                                 |                     |       |      |        |
| PLAT220_ALERT_2_C  | NonSolvent                             | Resd 1                     | O                               | Ueq(max)/Ueq(min)   | Range | 3.4  | Ratio  |
| PLAT222_ALERT_3_C  | NonSolvent                             | Resd 1                     | H                               | Uiso(max)/Uiso(min) | Range | 7.4  | Ratio  |
| PLAT242_ALERT_2_C  | Low                                    | 'MainMol'                  | Ueq as Compared to Neighbors of |                     |       | N3   | Check  |
| PLAT242_ALERT_2_C  | Low                                    | 'MainMol'                  | Ueq as Compared to Neighbors of |                     |       | C2   | Check  |
| PLAT242_ALERT_2_C  | Low                                    | 'MainMol'                  | Ueq as Compared to Neighbors of |                     |       | C3   | Check  |
| PLAT242_ALERT_2_C  | Low                                    | 'MainMol'                  | Ueq as Compared to Neighbors of |                     |       | C26  | Check  |
| PLAT360_ALERT_2_C  | Short                                  | C(sp3)-C(sp3)              | Bond C1                         | - C2                | .     | 1.34 | Ang.   |
| PLAT360_ALERT_2_C  | Short                                  | C(sp3)-C(sp3)              | Bond C27                        | - C28               | .     | 1.38 | Ang.   |
| <b>Alert level G</b>   |  |                            |                                 |                     |       |      |        |
| PLAT003_ALERT_2_G  | Number of Uiso or Uij                  | Restrained non-H Atoms ... |                                 |                     |       | 3    | Report |
| PLAT177_ALERT_4_G  | The CIF-Embedded .res File             | Contains DELU Records      |                                 |                     |       | 1    | Report |
| PLAT178_ALERT_4_G  | The CIF-Embedded .res File             | Contains SIMU Records      |                                 |                     |       | 1    | Report |
| PLAT199_ALERT_1_G  | Reported _cell_measurement_temperature | ..... (K)                  |                                 |                     |       | 293  | Check  |
| PLAT200_ALERT_1_G  | Reported _diffrn_ambient_temperature   | ..... (K)                  |                                 |                     |       | 293  | Check  |
| PLAT793_ALERT_4_G  | Model has Chirality at C7              | (Centro SPGR)              |                                 |                     |       | R    | Verify |
| PLAT860_ALERT_3_G  | Number of Least-Squares                | Restraints .....           |                                 |                     |       | 15   | Note   |
| PLAT941_ALERT_3_G  | Average HKL Measurement                | Multiplicity .....         |                                 |                     |       | 4.6  | Low    |
| <p>0 <b>ALERT level A</b> = Most likely a serious problem - resolve or explain</p> <p>1 <b>ALERT level B</b> = A potentially serious problem, consider carefully</p> <p>8 <b>ALERT level C</b> = Check. Ensure it is not caused by an omission or oversight</p> <p>8 <b>ALERT level G</b> = General information/check it is not something unexpected</p> <p>2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data</p> <p>9 ALERT type 2 Indicator that the structure model may be wrong or deficient</p> <p>3 ALERT type 3 Indicator that the structure quality may be low</p> <p>3 ALERT type 4 Improvement, methodology, query or suggestion</p> <p>0 ALERT type 5 Informative message, check</p> |  |                            |                                 |                     |       |      |        |

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special\_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

### **Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

### **Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

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