Supporting Information

Transition-metal free alkylarylation of acrylamides initiated by radical C−C bond cleavage of the tertiary cycloalkanols

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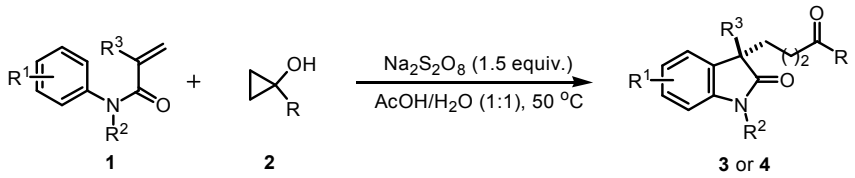
General Information

All reactions were carried out in Schlenk-tubes filled with nitrogen. Column chromatography was carried out on silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance III-400 in solvents as indicated. Chemical shift are reported in ppm from TMS with the solvent resonance as internal standard ($CDCl_3$: ^{1}H-NMR: δ = 7.26; ^{13}C-NMR: δ = 77.0). IR spectra were recorded on a Bruker Tensor 27 spectrometer and only major peaks are reported in cm^{-1}. HRMS were obtained on a Q-TOF micro spectrometer. Melting points were measured on a microscopic apparatus and are uncorrected.

Starting Materials

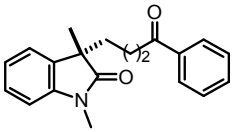
All of acrylamides **1** were synthesized according to the literature, and the NMR spectroscopies were in full accordance with the data in the literature.[1] Tertiary cyclopropanols **4** were prepared by the addition of Grignard reagent to the precursor esters according to the reported procedure.[2] Tertiary cyclobutanols were prepared by the addition of Grignard reagent to cyclobutanone according to the reported procedure.[3] All of the NMR spectroscopy were in full accordance with the data in the literatures.

General Procedure for the Cyclization of Acrylamides with Cyclopropanols

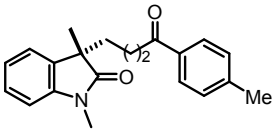


Acrylamides **1** (0.2 mmol, 1.0 equiv), and $Na_2S_2O_8$ (71.4 mg, 1.5 equiv) were added into an oven-dried Schlenk-tube. The tube was evacuated and backfilled with nitrogen (3 times). Then, a solution of tertiary cyclopropanols **2** (0.3 mmol, 1.5 equiv) in HOAc/H_2O (1:1, 2 mL) was injected into the tube by syringe. The tube was then sealed with a Teflon lined cap and the mixture was stirred at 50 ^oC for 24 h. The resulting mixture was diluted with EtOAc, and the organic phase was washed successively with H_2O, $NaHCO_3$ (3 times) and brine then dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (gradient eluent of EtOAc/petroleum ether: 1/10 to 1/5) to give the corresponding products **3** and **4** in yields listed in Table 2 and Table 3.

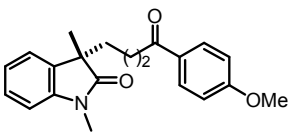
Characterization of Products 3



3a: Colorless liquid (82%, 50.3 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.26 (t, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.07 (t, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.22 (s, 3H), 2.90-2.77 (m, 2H), 2.01-1.84 (m, 2H), 1.43-1.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.5, 180.6, 143.2, 136.8, 133.7, 132.9, 128.5, 127.9, 127.8, 122.6, 108.0, 48.4, 38.3, 37.8, 26.2, 23.9, 19.2 ppm; IR (KBr): υ_{max} 1711, 1608, 1460, 1346, 1246 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{21}NNaO_2$ $[M+Na]^+$ 330.1464, found 330.1460.

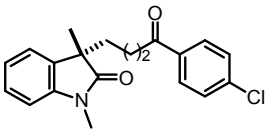


3b: Pale yellow liquid (80%, 51.4 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.75 (d, *J* = 8.0 Hz, 2H), 7.27-7.19 (m, 4H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 2.89-2.74 (m, 2H), 2.38 (s, 3H), 2.00-1.84 (m, 2H), 1.42-1.36 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.2, 180.5, 143.6, 143.2, 134.3, 133.7, 129.1, 128.0, 127.7, 122.6, 122.5, 108.0, 48.3, 38.2, 37.8, 26.1, 23.9, 21.5, 19.3 ppm; IR (KBr): υ_{max} 1712, 1610, 1462, 1347, 1256 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_2$ $[M+Na]^+$ 344.1621, found 344.1615.

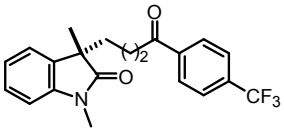


3c: Pale yellow liquid (70%, 47.2 mg); R_f 0.3 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85-7.82 (m, 2H), 7.27-7.19 (m, 2H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.90-6.86 (m, 2H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.84 (s, 3H), 3.21 (s, 3H), 2.86-2.71 (m, 2H), 2.00-1.83 (m, 2H), 1.41-1.32 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.1, 180.6, 163.3, 143.2, 133.7, 130.1, 129.9, 127.7, 122.6, 122.5, 113.6, 108.0,

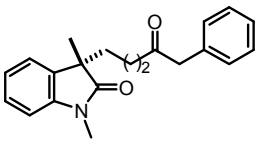
55.4, 48.4, 38.0, 37.8, 26.1, 23.9, 19.4 ppm; IR (KBr): υ_{max} 1711, 1604, 1462, 1347, 1254, 1172 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_3$ $[M+Na]^+$ 360.1570, found 360.1561.



3d: Colorless liquid (73%, 49.8 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.77 (d, *J* = 8.8 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.27-7.23 (m, 1H), 7.19 (d, *J* = 7.2 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 2.87-2.72 (m, 2H), 1.99-1.82 (m, 2H), 1.41-1.31 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.2, 180.4, 143.2, 139.3, 135.0, 133.6, 129.3, 128.8, 127.8, 122.5, 108.0, 48.3, 38.3, 37.6, 26.1, 23.9, 19.1 ppm; IR (KBr): υ_{max} 1711, 1611, 1464, 1347, 1247 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{20}ClKNO_2$ $[M+K]^+$ 380.0814, found 380.0806.

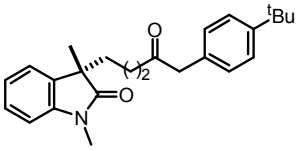


3e: Colorless liquid (54%, 40.5 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.94 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.20 (d, *J* = 6.8 Hz, 1H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.85 (d, *J* = 7.6 Hz, 1H), 3.22 (s, 3H), 2.93-2.79 (m, 2H), 2.02-1.84 (m, 2H), 1.44-1.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.5, 180.4, 143.2, 139.4, 134.2 (q, *J* = 32.5 Hz), 128.2, 127.8, 125.6 (q, *J* = 3.6 Hz), 123.5 (q, *J* = 271.2 Hz), 122.6, 122.5, 108.1, 48.3, 38.6, 37.6, 26.1, 24.0, 19.0 ppm; IR (KBr): υ_{max} 1711, 1612, 1469, 1325, 1169, 1130, 1067 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{20}F_3NNaO_2$ $[M+Na]^+$ 398.1338, found 398.1330.

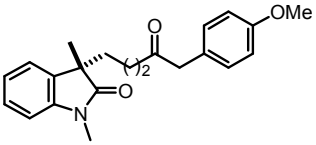


3f: Pale yellow liquid (80%, 51.4 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.31-7.23 (m, 4H), 7.16-7.11 (m, 3H), 7.05 (td, *J* = 7.6, 0.8 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 3.57 (s, 2H), 3.19 (s, 3H), 2.41-2.27 (m, 2H),

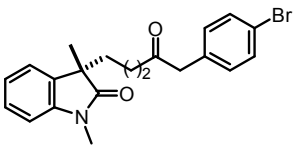
1.84-1.73 (m, 2H), 1.31 (s, 3H), 1.22-1.13 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 207.7, 180.5, 143.2, 134.1, 133.6, 129.3, 128.7, 127.7, 126.9, 122.5, 108.0, 50.0, 48.3, 41.7, 37.5, 26.1, 23.8, 18.7 ppm; IR (KBr): υ_{max} 1711, 1610, 1462, 1346 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_2$ $[M+Na]^+$ 344.1621, found 344.1611.



3g: Pale yellow liquid (68%, 51.3 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32 (d, *J* = 8.4 Hz, 2H), 7.27-7.23 (m, 1H), 7.15 (d, *J* = 7.2 Hz, 1H), 7.07-7.03 (m, 3H), 6.83 (d, *J* = 8.0 Hz, 1H), 3.54 (s, 2H), 3.19 (s, 3H), 2.40-2.25 (m, 2H), 1.84-1.68 (m, 2H), 1.32 (s, 3H), 1.30 (s, 9H), 1.22-1.14 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 208.0, 180.5, 149.8, 143.2, 133.6, 131.0, 128.9, 127.7, 125.6, 122.5, 108.0, 49.5, 48.3, 41.6, 37.5, 34.4, 31.3, 26.1, 23.8, 18.7 ppm; IR (KBr): υ_{max} 1713, 1612, 1468, 1373, 1261 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{31}KNO_2$ $[M+K]^+$ 416.1986, found 416.1975.

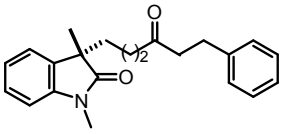


3h: Pale yellow liquid (66%, 46.3 mg); R_f 0.3 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.27-7.23 (m, 1H), 7.15 (d, *J* = 7.2 Hz, 1H), 7.07-7.02 (m, 3H), 6.85-6.82 (m, 3H), 3.78 (s, 3H), 3.50 (s, 2H), 3.19 (s, 3H), 2.37-2.23 (m, 2H), 1.83-1.67 (m, 2H), 1.31 (s, 3H), 1.21-1.12 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 208.1, 180.5, 158.5, 143.2, 130.3, 127.7, 126.2, 122.5, 114.1, 108.0, 55.2, 49.1, 48.2, 41.5, 37.5, 26.1, 23.8, 18.7 ppm; IR (KBr): υ_{max} 1711, 1611, 1512, 1465, 1348, 1252 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{25}KNO_3$ $[M+K]^+$ 390.1466, found 390.1457.

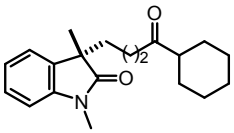


3i: Pale yellow liquid (62%, 49.5 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43 (d, *J* = 8.0 Hz, 2H), 7.30-7.26 (m, 1H), 7.17 (d, *J* = 6.8 Hz, 1H), 7.08 (t, *J* = 7.2 Hz, 1H), 7.01 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.0 Hz,

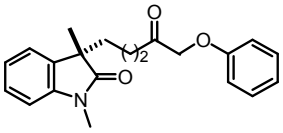
1H), 3.55 (s, 2H), 3.22 (s, 3H), 2.41-2.27 (m, 2H), 1.84-1.73 (m, 2H), 1.34 (s, 3H), 1.24-1.17 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.9, 180.4, 143.1, 133.5, 133.0, 131.7, 131.0, 127.8, 122.5, 121.0, 108.0, 49.0, 48.2, 41.9, 37.4, 26.1, 23.8, 18.7 ppm; IR (KBr): υ_{max} 1711, 1612, 1490, 1469, 1376, 1348; HRMS (ESI) calcd for $C_{21}H_{22}BrNNaO_2$ $[M+Na]^+$ 422.0726, found 422.0713.



3j: Colorless liquid (73%, 48.9 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.28-7.24 (m, 3H), 7.19-7.12 (m, 4H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 2.82 (t, *J* = 7.6 Hz, 2H), 2.62 (t, *J* = 7.6 Hz, 2H), 2.33-2.19 (m, 2H), 1.87-1.69 (m, 2H), 1.33 (s, 3H), 1.24-1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 209.4, 180.4, 143.1, 140.9, 133.6, 128.4, 128.2, 127.7, 126.0, 122.5, 108.0, 48.2, 44.0, 42.6, 37.5, 29.5, 26.1, 23.8, 18.7 ppm; IR (KBr): υ_{max} 1711, 1612, 1493, 1469, 1375, 1348 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{25}NNaO_2$ $[M+Na]^+$ 358.1777, found 358.1782.



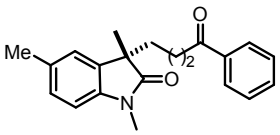
3k: Colorless liquid (40%, 25 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.25 (t, *J* = 7.6 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 7.06 (t, *J* = 7.2 Hz, 1H), 6.83 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 2.38-2.21 (m, 3H), 1.86-1.71 (m, 6H), 1.34 (s, 3H), 1.25-1.13 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 213.6, 180.6, 143.2, 133.7, 127.7, 122.6, 122.5, 108.0, 50.6, 48.3, 40.3, 37.7, 28.4, 26.1, 25.8, 25.6, 23.9, 18.6 ppm; IR (KBr): υ_{max} 1711, 1612, 1492, 1452, 1375, 1347 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{27}NNaO_2$ $[M+Na]^+$ 336.1934, found 336.1936.



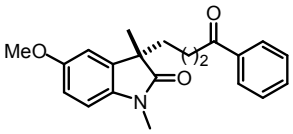
3l: Colorless liquid (80%, 53.9 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30-7.24 (m, 3H), 7.18 (d, *J* = 6.8 Hz, 1H), 7.06 (t, *J* = 7.6

Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.85-6.81 (m, 3H), 4.44 (s, 2H), 3.20 (s, 3H), 2.48 (td, *J* = 7.6, 1.2 Hz, 2H), 1.92-1.75 (m, 2H), 1.34 (s, 3H), 1.30-1.21 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 207.2, 180.4, 157.6, 143.2, 133.5, 129.6, 127.8, 122.5, 121.6, 114.4, 108.0, 72.6, 48.2, 38.8, 37.5, 26.1, 23.9, 18.1 ppm; IR (KBr): υ_{max} 1707, 1608, 1491, 1463, 1375, 1241 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_3$ $[M+Na]^+$ 360.1570, found 360.1573.

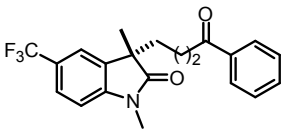
Characterization of Products 4



4b: Pale yellow solid (73%, 46.9 mg), mp = 93-95 ^{o}C; R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86-7.84 (m, 2H), 7.55-7.51 (m, 1H), 7.44-7.40 (m, 2H), 7.06-7.01 (m, 2H), 6.73 (d, *J* = 7.6 Hz, 1H), 3.20 (s, 3H), 2.88-2.78 (m, 2H), 2.33 (s, 3H), 2.01-1.82 (m, 2H), 1.42-1.35 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.7, 180.5, 140.9, 136.8, 133.8, 132.9, 132.1, 128.5, 128.0, 127.9, 123.4, 107.8, 48.4, 38.4, 37.8, 26.2, 24.0, 21.1, 19.3 ppm; IR (KBr): υ_{max} 1710, 1601, 1498, 1454, 1351, 1260 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_2$ $[M+Na]^+$ 344.1621, found 344.1624.

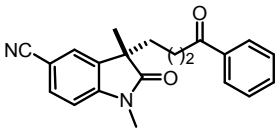


4c: Pale yellow solid (60%, 40.4 mg), mp = 87-89 ^{o}C; R_f 0.3 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.43-7.39 (m, 2H), 6.82-6.73 (m, 3H), 3.79 (s, 3H), 3.19 (s, 3H), 2.92-2.77 (m, 2H), 2.01-1.80 (m, 2H), 1.41-1.35 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.5, 180.1, 156.1, 136.8, 135.1, 132.9, 128.5, 127.9, 111.7, 110.3, 108.2, 55.7, 48.8, 38.3, 37.8, 26.2, 24.0, 19.3 ppm; IR (KBr): υ_{max} 1707, 1600, 1496, 1356, 1287, 1214 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_3$ $[M+Na]^+$ 360.1570, found 360.1572.

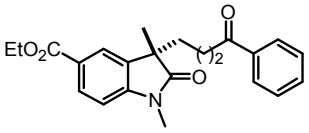


4d: Pale yellow liquid (80%, 60 mg); R_f 0.2 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.56-7.50 (m, 2H), 7.43-7.39 (m, 3H), 6.91 (d, *J* = 8.0 Hz, 1H), 3.25 (s, 3H), 2.90-2.83 (m, 2H), 2.06-1.85 (m, 2H), 1.43-1.30 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.2, 180.4, 146.3, 136.7, 134.3, 133.0, 128.5, 127.8, 125.7 (q, J_{C-F} = 4.0 Hz), 124.8 (q, J_{C-F} = 32.5 Hz), 124.4 (q,

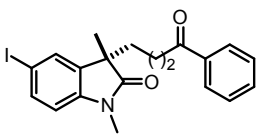
$J_{C\text{-}F}$ = 271.2 Hz), 119.5 (q, J = 3.6 Hz), 107.8, 48.4, 38.2, 37.7, 26.3, 23.8, 19.0 ppm; IR (KBr): υ_{max} 1723, 1619, 1455, 1332, 1279, 1118 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{20}F_3NNaO_2$ $[M+Na]^+$ 398.1338, found 398.1337.



4e: Pale yellow liquid (68%, 45.2 mg); R_f 0.2 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, J = 7.2, 1.2 Hz, 2H), 7.60 (dd, J = 8.0, 1.6 Hz, 1H), 7.56-7.52 (m, 1H), 7.45-7.41 (m, 3H), 6.91 (d, J = 8.0 Hz, 1H), 3.25 (s, 3H), 2.91-2.85 (m, 2H), 2.01-1.83 (m, 2H), 1.41-1.29 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.1, 180.1, 147.2, 136.7, 134.8, 133.3, 133.1, 128.6, 127.9, 125.9, 119.2, 108.5, 105.7, 48.3, 38.1, 37.6, 26.4, 23.8, 19.0 ppm; IR (KBr): υ_{max} 2221, 1722, 1611, 1496, 1453, 1344, 1256 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{20}N_2NaO_2$ $[M+Na]^+$ 355.1417, found 355.1412.

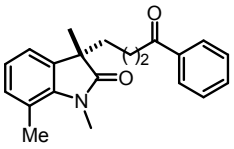


4f: Colorless liquid (83%, 62.9 mg); R_f 0.2 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 8.02 (dd, J = 8.4, 1.6 Hz, 1H), 7.86-7.83 (m, 3H), 7.52 (t, J = 7.6 Hz, 1H), 7.41(t, J = 7.6 Hz, 2H), 6.87 (d, J = 8.4 Hz, 1H), 4.42-4.31 (m, 2H), 3.25 (s, 3H), 2.93-2.77 (m, 2H), 2.06-1.86 (m, 2H), 1.44-1.27 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.3, 180.8, 166.5, 147.4, 136.7, 133.6, 133.0, 130.6, 128.5, 127.9, 124.9, 123.7, 107.5, 60.9, 48.3, 38.3, 37.7, 26.4, 23.9, 19.1, 14.4 ppm; IR (KBr): υ_{max} 1709, 1615, 1498, 1454, 1372, 1276 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{25}NNaO_4$ $[M+Na]^+$ 402.1676, found 402.1671.

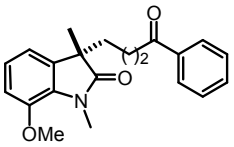


4g: Pale yellow liquid (87%, 75.3 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.87 (dd, J = 7.2, 1.6 Hz, 2H), 7.59-7.54 (m, 2H), 7.52-7.41 (m, 3H), 6.63 (d, J = 8.0 Hz, 1H), 3.19 (s, 3H), 2.90-2.83 (m, 2H), 1.99-1.79 (m,

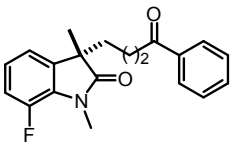
2H), 1.41-1.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.4, 179.7, 143.0, 136.8, 136.7, 136.2, 133.0, 131.4, 128.6, 127.9, 110.1, 85.2, 48.5, 38.3, 37.7, 26.2, 23.9, 19.1 ppm; IR (KBr): υ_{max} 1714, 1602, 1484, 1341 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{20}INNaO_2$ $[M+Na]^+$ 456.0431, found 456.0423.



4h: White solid (62%, 39.8 mg), mp = 97-99 °C; R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.55-7.51 (m, 1H), 7.42 (td, *J* = 7.6, 1.6 Hz, 2H), 7.04-6.92 (m, 3H), 3.50 (s, 3H), 2.88-2.81 (m, 2H), 2.58 (s, 3H), 1.97-1.83 (m, 2H), 1.39-1.31 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 181.3, 141.0, 136.8, 134.3, 132.9, 131.5, 128.5, 127.9, 122.5, 120.5, 119.6, 47.7, 38.4, 38.1, 29.5, 24.5, 19.3, 19.1 ppm; IR (KBr): υ_{max} 1708, 1600, 1459, 1364, 1337 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_2$ $[M+Na]^+$ 344.1621, found 344.1614.

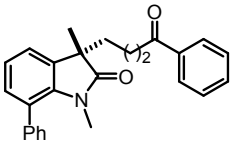


4i: Colorless liquid (45%, 30.3 mg); R_f 0.3 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 (dd, *J* = 7.2, 1.6 Hz, 2H), 7.54-7.50 (m, 1H), 7.44-7.40 (m, 2H), 7.02-6.98 (m, 1H), 6.82 (dd, *J* = 8.0, 0.8 Hz, 2H), 3.86 (s, 3H), 3.49 (s, 3H), 2.88-2.81 (m, 2H), 1.97-1.83 (m, 2H), 1.39-1.32 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 180.8, 145.3, 136.8, 135.4, 132.9, 131.0, 128.5, 127.9, 123.1, 115.3, 111.6, 55.8, 48.4, 38.4, 38.0, 29.4, 24.3, 19.3 ppm; IR (KBr): υ_{max} 1707, 1605, 1464, 1336, 1249 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_3$ $[M+Na]^+$ 360.1570, found 360.1565.

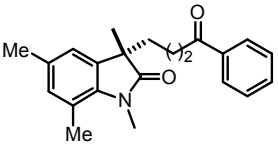


4j: Colorless liquid (63%, 41 mg); R_f 0.3 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.55-7.51 (m, 1H), 7.44-7.41 (m, 2H), 6.99-6.96 (m, 3H), 3.43 (d, *J* = 2.8 Hz, 3H), 2.94-2.79 (m, 2H), 2.01-1.83 (m,

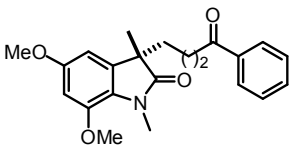
2H), 1.42-1.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.4, 180.1, 147.7 (d, $J_{C\text{-}F}$ = 242.0 Hz), 146.8, 136.7 (d, $J_{C\text{-}F}$ = 3.0 Hz), 133.0, 129.8 (d, $J_{C\text{-}F}$ = 7.7 Hz), 128.5, 127.9, 123.1 (d, $J_{C\text{-}F}$ = 3.0 Hz), 118.4 (d, $J_{C\text{-}F}$ = 3.2 Hz), 115.8 (d, $J_{C\text{-}F}$ = 19.2 Hz), 48.8 (d, $J_{C\text{-}F}$ = 1.4 Hz), 38.2, 37.9, 28.6 (d, $J_{C\text{-}F}$ = 5.6 Hz), 24.2, 19.1 ppm; IR (KBr): υ_{max} 1718, 1630, 1483, 1371, 1336, 1237 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{20}FNNaO_2$ $[M+Na]^+$ 348.1370, found 348.1365.



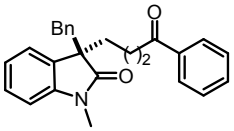
4k: White solid (72%, 55.2 mg), mp = 128-130 °C; R_f 0.3 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.54 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45-7.35 (m, 7H), 7.22-7.20 (m, 1H), 7.10-7.05 (m, 2H), 2.91-2.87 (m, 2H), 2.85 (s, 3H), 2.02-1.91 (m, 2H), 1.49-1.26 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 181.5, 140.1, 139.0, 136.8, 134.7, 132.9, 130.8, 130.0, 128.5, 127.9, 127.7, 127.5, 125.4, 121.9, 121.6, 47.6, 38.3, 38.1, 30.1, 24.4, 19.2 ppm; IR (KBr): υ_{max} 1712, 1598, 1454, 1367, 1336, 1065 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{25}NNaO_2$ $[M+Na]^+$ 406.1777, found 406.1773.



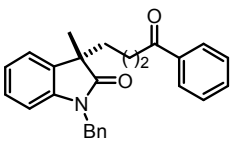
4l: Pale yellow liquid (67%, 44.9 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.43-7.39 (m, 2H), 6.65 (s, 1H), 6.52 (s, 1H), 3.19 (s, 3H), 2.87-2.79 (m, 2H), 2.35 (s, 3H), 2.33 (s, 3H), 2.17-1.96 (m, 2H), 1.41 (s, 3H), 1.35-1.19 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 180.9, 143.6, 137.6, 136.8, 134.0, 132.9, 128.5, 127.9, 127.2, 125.7, 106.8, 49.2, 38.2, 35.9, 26.2, 22.6, 21.5, 19.7, 18.0 ppm; IR (KBr): υ_{max} 1711, 1619, 1452, 1378, 1334, 1240 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{25}NNaO_2$ $[M+Na]^+$ 358.1777, found 358.1768.



4m: Solid (29%, 21.3 mg), mp = 123-125 ^{o}C; R_f 0.35 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 6.8, 1.6 Hz, 2H), 7.55-7.51 (m, 1H), 7.44-7.40 (m, 2H), 6.41-6.39 (m, 2H), 3.83 (s, 3H), 3.79 (s, 3H), 3.44 (s, 3H), 2.92-2.78 (m, 2H), 1.98-1.77 (m, 2H), 1.39-1.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.7, 180.4, 156.7, 145.9, 136.8, 136.0, 132.9, 128.5, 127.9, 124.5, 100.4, 98.9, 55.8, 49.0, 38.4, 38.0, 29.3, 24.4, 19.3 ppm; IR (KBr): υ_{max} 1702, 1605, 1496, 1456, 1363, 1270,1210, 1153 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{25}NNaO_4$ $[M+Na]^+$ 390.1676, found 390.1671.

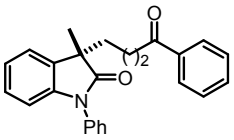


4n: White solid (75%, 57.5 mg), mp = 154-156 ^{o}C; R_f 0.35 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.2, 1.6 Hz, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.2 Hz, 2H), 7.17 (t, *J* = 7.2 Hz, 2H), 7.06-6.99 (m, 4H), 6.82-6.80 (m, 2H), 6.58 (d, *J* = 7.6 Hz, 1H), 3.13 (d, *J* = 12.8 Hz, 1H), 3.02 (d, *J* = 12.8 Hz, 1H), 2.95-2.80 (m, 5H), 2.18-1.99 (m, 2H), 1.46-1.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.5, 179.0, 143.8, 136.8, 135.7, 132.9, 130.8, 129.8, 128.5, 127.9, 127.8, 127.4, 126.4, 123.5, 122.2, 107.8, 54.7, 44.5, 38.4, 36.3, 25.8, 19.2 ppm; IR (KBr): υ_{max} 1703, 1611, 1493, 1450, 1375, 1261 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{25}NNaO_2$ $[M+Na]^+$ 406.1777, found 406.1763.



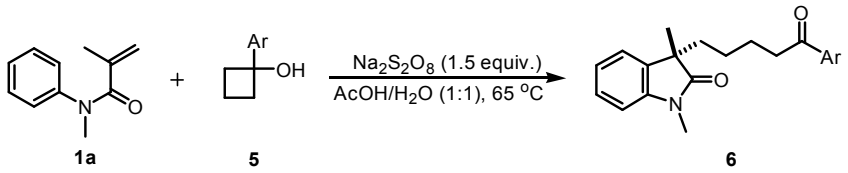
4o: Colorless liquid (70%, 53.6 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (d, *J* = 7.6 Hz, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.29-7.21 (m, 6H), 7.14 (td, *J* = 7.6, 0.8 Hz, 1H), 7.04 (t, *J* = 7.6 Hz, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 4.96 (d, *J* = 15.6 Hz, 1H), 4.91 (d, *J* = 16.0 Hz, 1H), 2.94-2.79 (m, 2H), 2.08-1.91 (m, 2H), 1.54-1.38 (m, 5H); ^{13}C NMR (100 MHz,

$CDCl_3$): δ = 199.5, 180.6, 142.3, 136.8, 136.0, 133.7, 132.9, 128.7, 128.5, 127.9, 127.7, 127.5, 127.2, 122.7, 122.6, 109.1, 48.4, 43.6, 38.4, 37.8, 24.3, 19.5 ppm; IR (KBr): υ_{max} 1711, 1610, 1489, 1451, 1353, 1174 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{25}NNaO_2$ $[M+Na]^+$ 406.1777, found 406.1766.



4p: White solid (76%, 56.1 mg), mp = 125-126 °C; R_f 0.25 (EtOAc/petroleum ether = 1:5); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.87 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.55-7.50 (m, 3H), 7.44-7.38 (m, 5H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.19 (td, *J* = 7.6, 1.2 Hz, 1H), 7.11 (t, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 2.93-2.87 (m, 2H), 2.08-1.97 (m, 2H), 1.57-1.50 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.5, 180.0, 143.2, 136.8, 134.6, 133.5, 132.9, 129.5, 128.5, 127.9, 127.7, 126.6, 123.1, 122.9, 109.4, 48.5, 38.3, 38.2, 24.2, 19.3 ppm; IR (KBr): υ_{max} 1721, 1603, 1497, 1456, 1371, 1203 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{23}NNaO_2$ $[M+Na]^+$ 392.1621, found 392.1607.

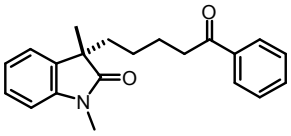
General Procedure for the Cyclization of Acrylamide 1a with Cyclobutanols



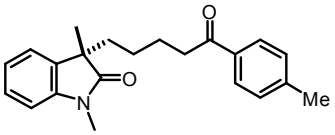
1a **5** **6**

Acrylamide **1a** (0.2 mmol, 1.0 equiv), and $Na_2S_2O_8$ (71.4 mg, 1.5 equiv) were added into an oven-dried Schlenk-tube. The tube was evacuated and backfilled with nitrogen (3 times). Then, a solution of tertiary cyclobutanols **5** (0.4 mmol, 2.0 equiv) in $HOAc/H_2O$ (1:1, 2 mL) was injected into the tube by syringe. The tube was then sealed with a Teflon lined cap and the mixture was stirred at 65 °C for 24 h. The resulting mixture was diluted with EtOAc, and the organic phase was washed successively with H_2O, $NaHCO_3$ (2 times) and brine then dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (gradient eluent of EtOAc/petroleum ether: 1/10 to 1/5) to give the corresponding products **6** in yields listed in equation 1.

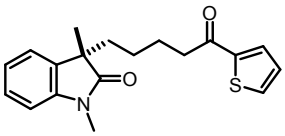
Characterization of Products 6



6a: Colorless liquid (40%, 25.7 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89-7.87 (m, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.44-7.40 (m, 2H), 7.26 (td, *J* = 7.6, 1.2 Hz, 1H), 7.17 (dd, *J* = 7.2, 0.8 Hz, 1H), 7.06 (td, *J* = 7.6, 0.4 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 3.21 (s, 3H), 2.87-2.76 (m, 2H), 2.00-1.74 (m, 2H), 1.65-1.58 (m, 2H), 1.35 (s, 3H), 1.10-0.93 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 180.6, 143.2, 136.8, 134.0, 132.8, 128.5, 127.9, 127.6, 122.5, 122.4, 107.9, 48.3, 38.2, 26.1, 24.2, 23.8 ppm; IR (KBr): υ_{max} 1712, 1610, 1468, 1375, 1253, 1125 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}NNaO_2$ $[M+Na]^+$ 344.1621, found 344.1624.



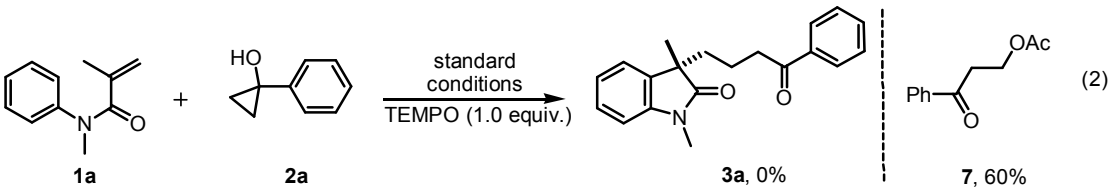
6b: Colorless liquid (40%, 26.8 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 8.0 Hz, 2H), 7.28-7.21 (m, 3H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.06 (t, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 2.84-2.69 (m, 2H), 2.39 (s, 3H),1.99-1.74 (m, 2H), 1.64-1.56 (m, 2H), 1.35 (s, 3H), 1.09-0.92 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.7, 180.7, 143.6, 143.2, 134.3, 134.0, 129.2, 128.1, 127.6, 122.5, 107.9, 48.3, 38.2, 26.1, 24.3, 23.8, 21.6 ppm; IR (KBr): υ_{max} 1712, 1608, 1461, 1366, 1252, 1185, 1021 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{25}NNaO_2$ $[M+Na]^+$ 358.1777, found 358.1780.



6c: Pale yellow liquid (44%, 28.8 mg); R_f 0.25 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.63 (d, *J* = 4.0 Hz, 1H), 7.59 (d, *J* = 4.8 Hz, 1H), 7.26 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 6.8 Hz, 1H), 7.10-7.04 (m, 2H), 6.84 (d, *J* = 7.6 Hz,

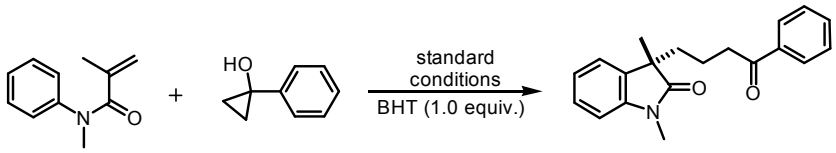
1H), 3.21 (s, 3H), 2.84-2.69 (m, 2H), 1.99-1.74 (m, 2H), 1.65-1.58 (m, 2H), 1.35 (s, 3H), 1.09-0.95 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.0, 180.6, 144.2, 143.2, 133.9, 133.4, 131.7, 128.0, 127.7, 122.5, 122.4, 107.9, 48.3, 39.0, 38.1, 26.1, 24.5, 24.2, 23.8 ppm; IR (KBr): υ_{max} 1706, 1608, 1465, 1356, 1248, 1080 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{21}NNaO_2S$ $[M+Na]^+$ 350.1185, found 350.1187.

Investigation of the Reaction Mechanism



1a **2a** **3a**, 0% **7**, 60% (2)

When 1.0 equiv of TEMPO was added to the reaction of **1a** with **2a** under the standard conditions, no desired product **3a** was detected along with a coupling product **7** isolated in 60%. The result indicates that the radical intermediate probably be involved in the catalytic cycle of the reaction.



1a **2a** **3a**, 52%

When 1.0 equiv of BHT was added to the reaction of **1a** with **2a** under the standard conditions, 52% yield of desired product **3a** was isolated along with 38% **1a** recovered. The result indicates that the radical intermediate probably be involved in the catalytic cycle of the reaction.

Isotope Labeling Experiment

a) Intramolecular Kinetic Isotope Effect (KIE) Experiment:

An oven-dried Schlenk-tube was charged with [D_1]-**1a** (0.1 mmol, 1.0 equiv) and $Na_2S_2O_8$ (35.7 mg, 1.5 equiv). The tube was evacuated and backfilled with nitrogen (3 times). Then, a solution of tertiary cycloalkanol **2a** (0.15 mmol, 1.5 equiv) in HOAc/H_2O (1:1, 1 mL) was injected into the tube by syringe. The tube was then sealed with a Teflon lined cap and the mixture was stirred at 50 °C for 3.5 h. The resulting mixture was diluted with EtOAc, and the organic phase was washed successively with H_2O (1 time), $NaHCO_3$ (3 times) and brine (1 time) then dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give the corresponding product in 60% yield. The product was analyzed by ^{1}H NMR (Figure 1).

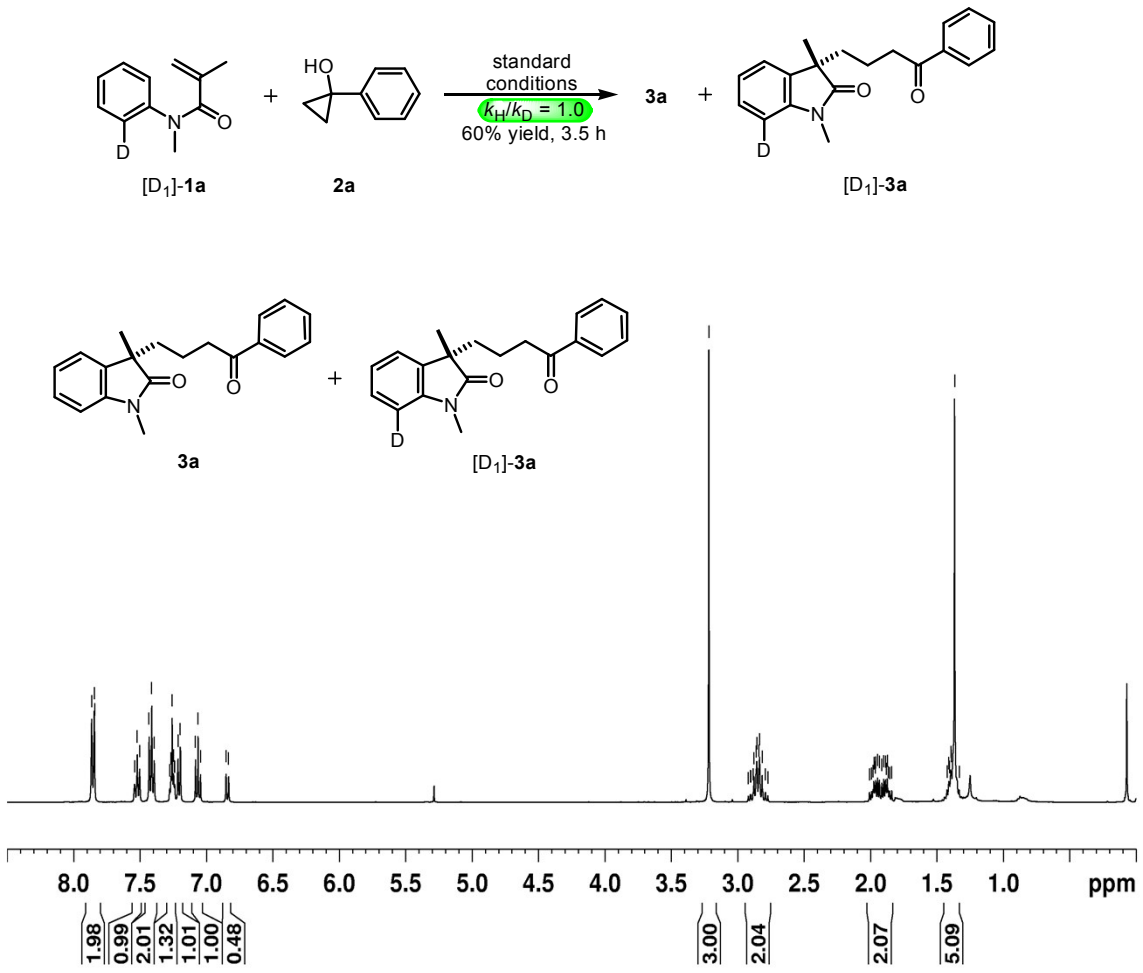


Figure 1. ^{1}H NMR spectra of the mixture of the product **3a** and [D_1]-**3a**.

b) Intermolecular Kinetic Isotope Effect (KIE) Experiment:

An oven-dried Schlenk-tube was charged with **1a** (0.05 mmol), [D_5]-**1a** (0.05 mmol) and $Na_2S_2O_8$ (35.7 mg, 1.5 equiv). The tube was evacuated and backfilled with nitrogen (3 times). Then, a solution of tertiary cycloalkanol **2a** (0.15 mmol, 1.5 equiv) in HOAc/H_2O (1:1, 1 mL) was injected into the tube by syringe. The tube was then sealed with a Teflon lined cap and the mixture was stirred at 50 °C for 3.5 h. The resulting mixture was diluted with EtOAc, and the organic phase was washed successively with H_2O (1 time), $NaHCO_3$ (2 times) and brine (1 time) then dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give the corresponding product in 65% yield. The product was analyzed by ^1H NMR (Figure 2).

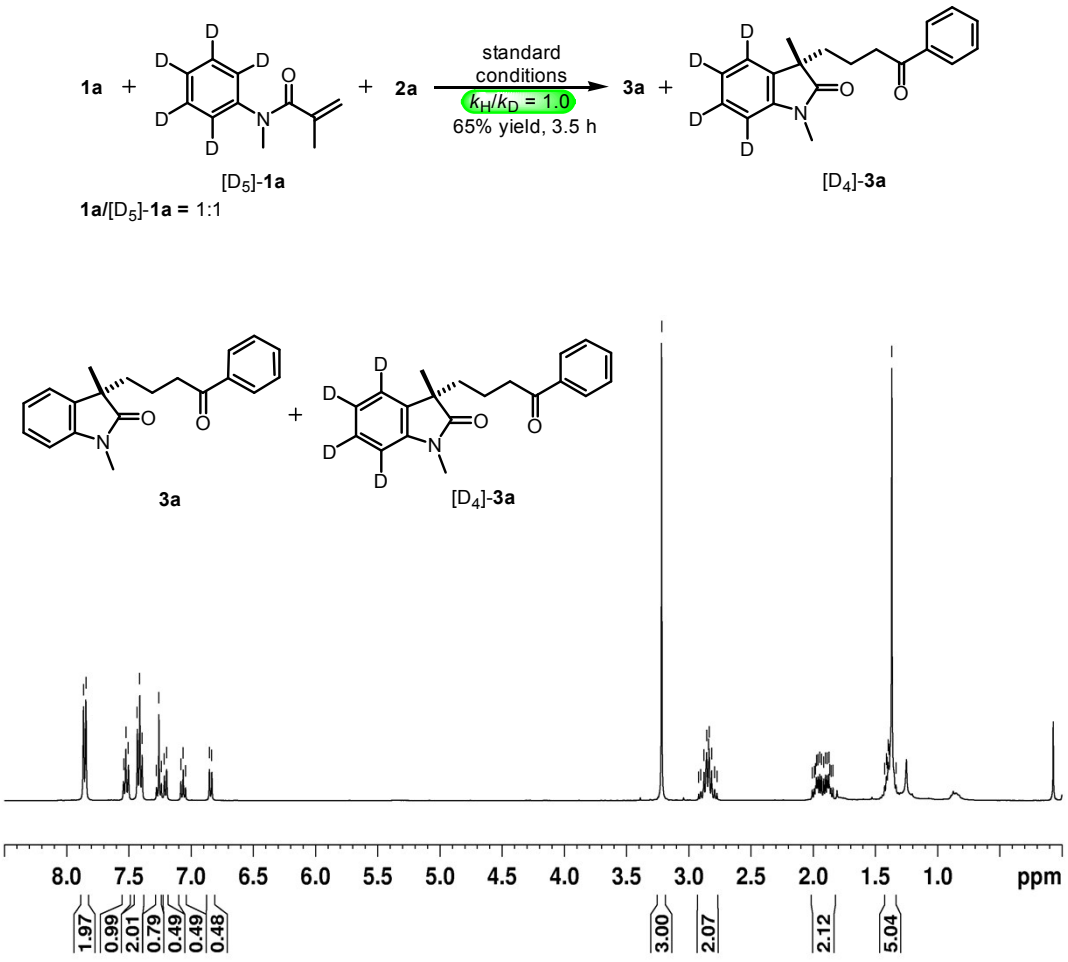


Figure 2. ^1H NMR spectra of the mixture of the product **3a** and [D_4]-**3a**.

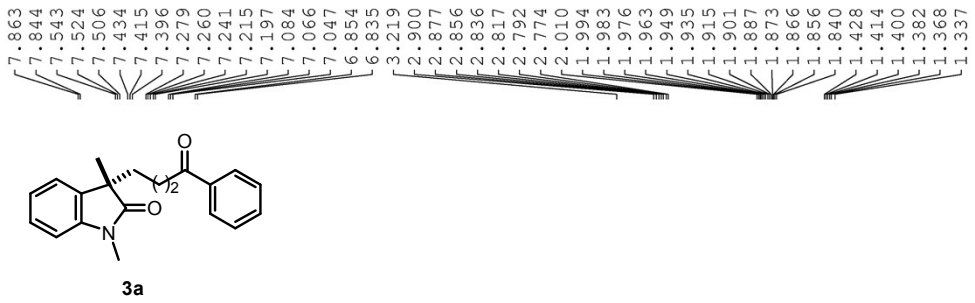
References

1 (a) T. Wu, X. Mu and G. Liu, *Angew. Chem.*, *Int. Ed.*, 2011, **50**, 12578; (b) X. Mu, T. Wu, H.-Y. Wang, Y.-L. Guo and G. Liu, *J. Am. Chem. Soc.*, 2012, **134**, 878; (c) D. C. Fabry, M. Stodulski, S. Hoerner and T. Gulder, *Chem. Eur. J.*, 2012, **18**, 10834.

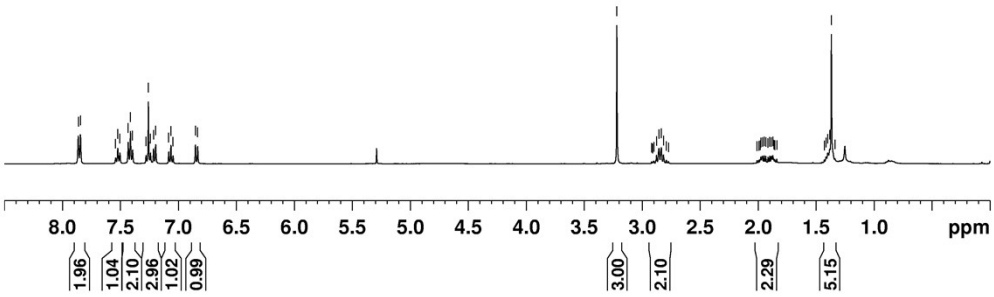
2 (a) A. Ilangovan, S. Saravanakumar and S. Malayappasamy, *Org. Lett.*, 2013, **15**, 4968; (b) J. Jiao, L. X. Nguyen, D. R. Patterson and R. A. Flowers II, *Org. Lett.*, 2007, **9**, 1323; (c) D. Rosa and A. Orellana, *Chem. Commun.*, 2013, **49**, 5420.

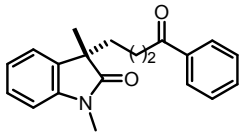
3 (a) B. M. Casey, C. A. Eakin and R. A. Flowers II, *Tetrahedron Lett.*, 2009, **50**, 1264; (b) H.-J. Xu, F.-F. Zhu, Y.-Y. Shen, X. Wan and Y.-S. Feng, *Tetrahedron*, 2012, **68**, 4145.

^{1}H NMR and ^{13}C NMR Spectra of Products 3



3a





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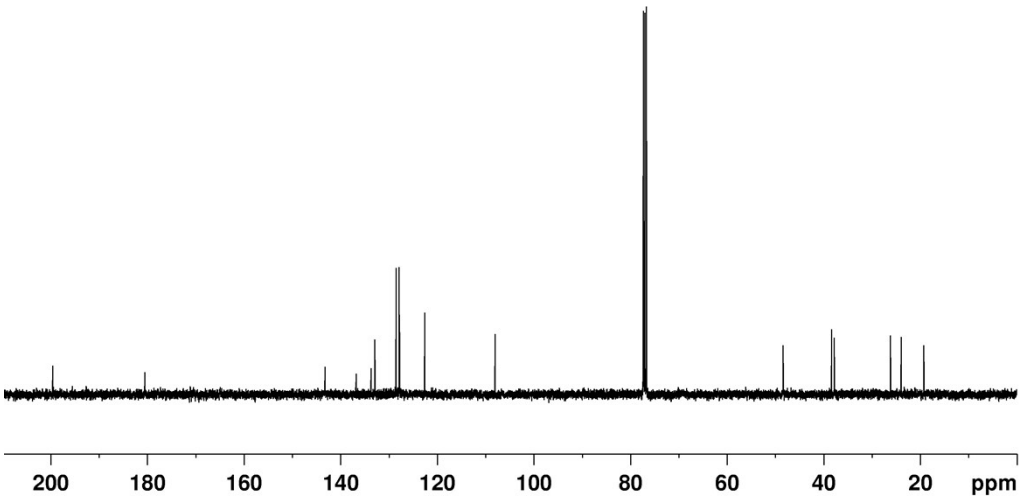

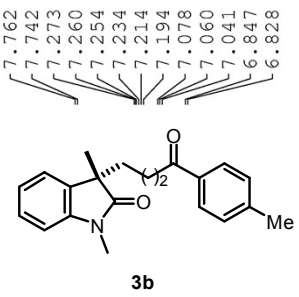
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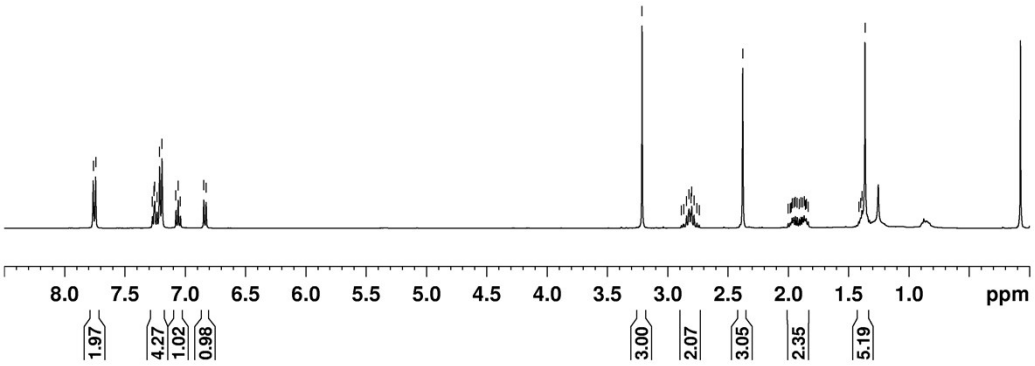
2
O
N
Me

3b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3.05
2.35
5.19


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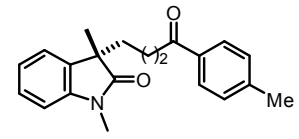
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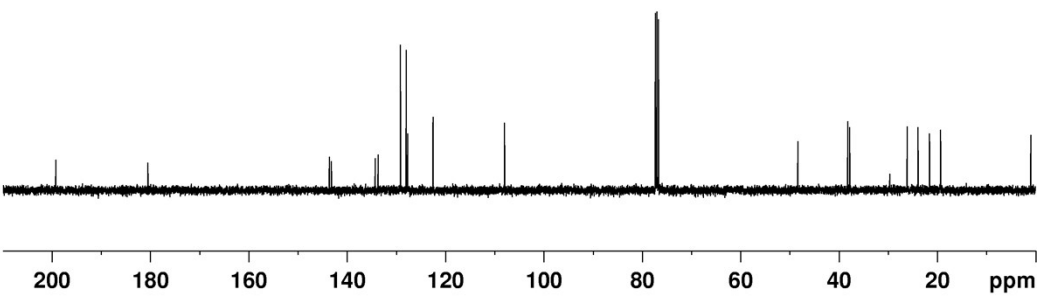
3b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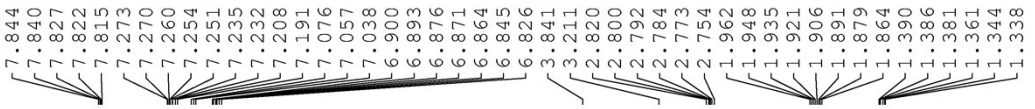
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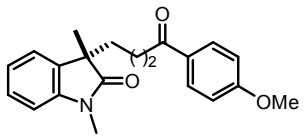
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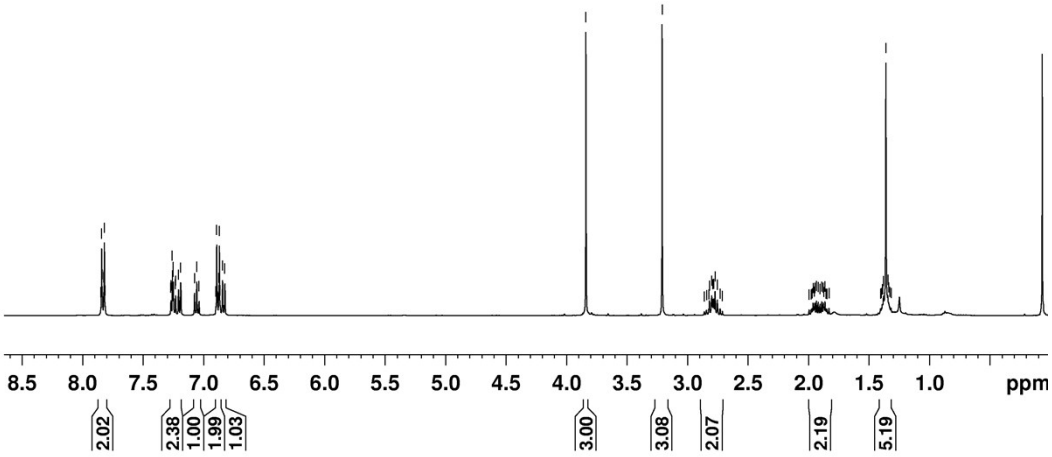
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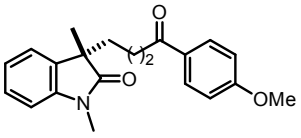
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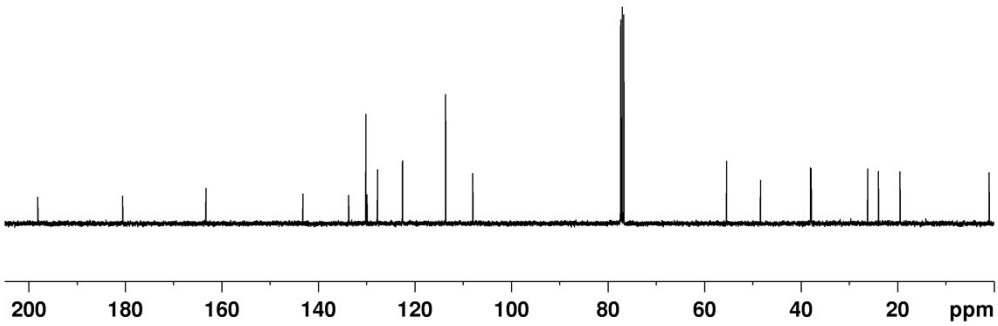
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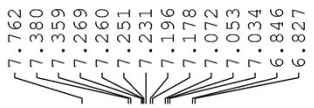
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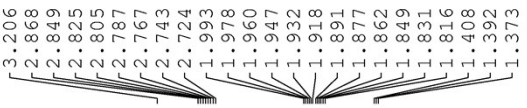
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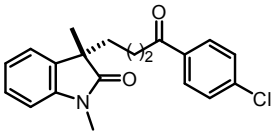
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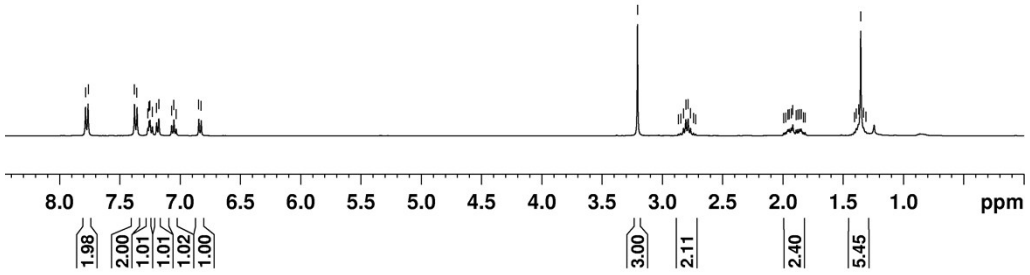
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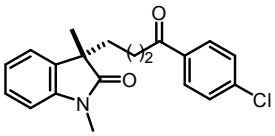
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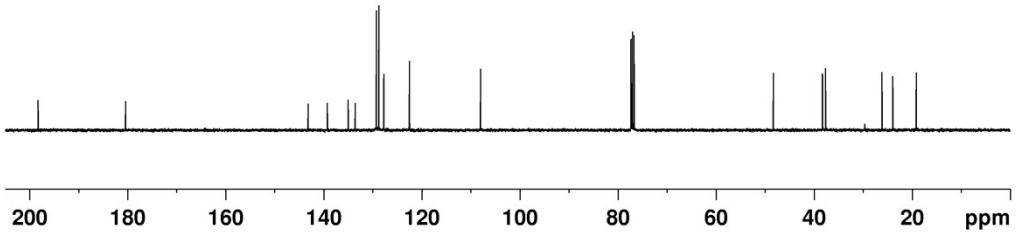
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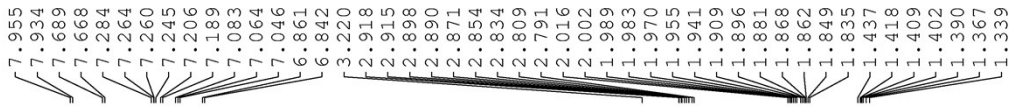
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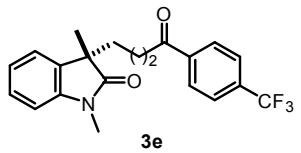
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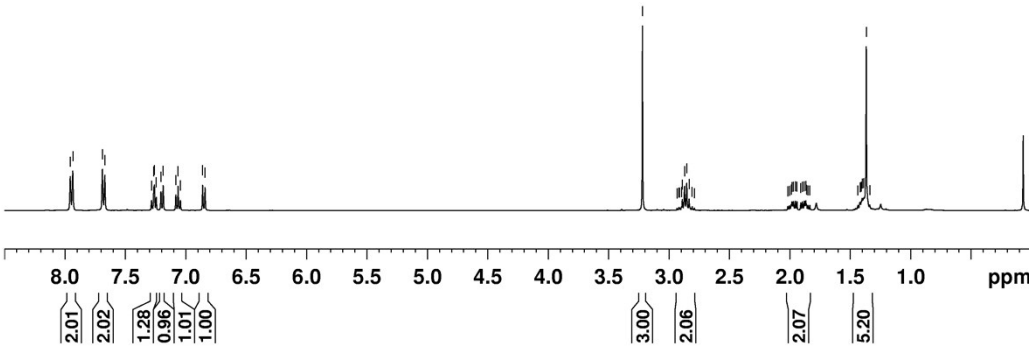
3e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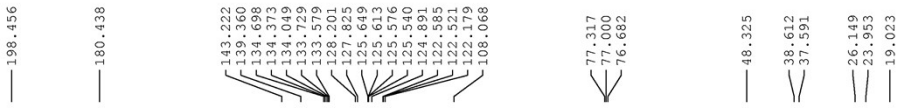
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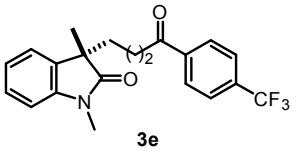
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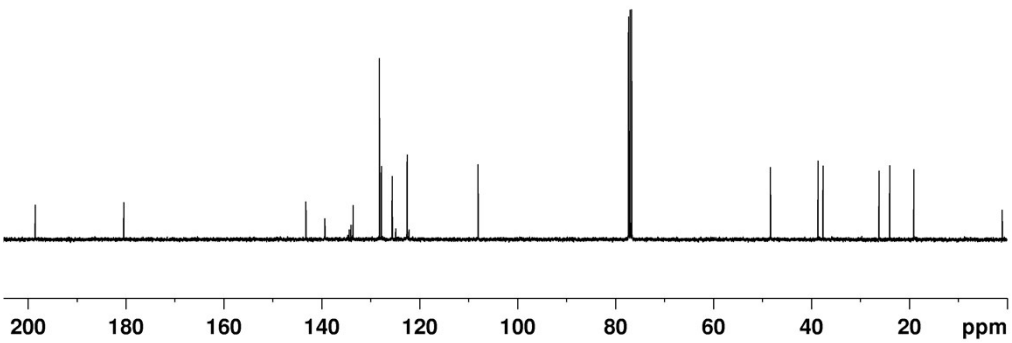
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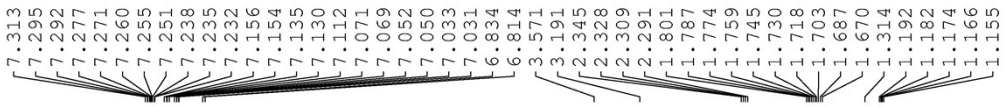
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7.271
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7.255
7.251
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7.235
7.232
7.156
7.154
7.135
7.130
7.112
7.071
7.069
7.052
7.050
7.033
7.031
6.834
6.814
3.571
3.191
2.345
2.328
2.309
2.291
1.801
1.787
1.774
1.759
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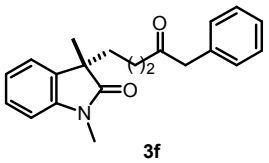
1.166
1.155



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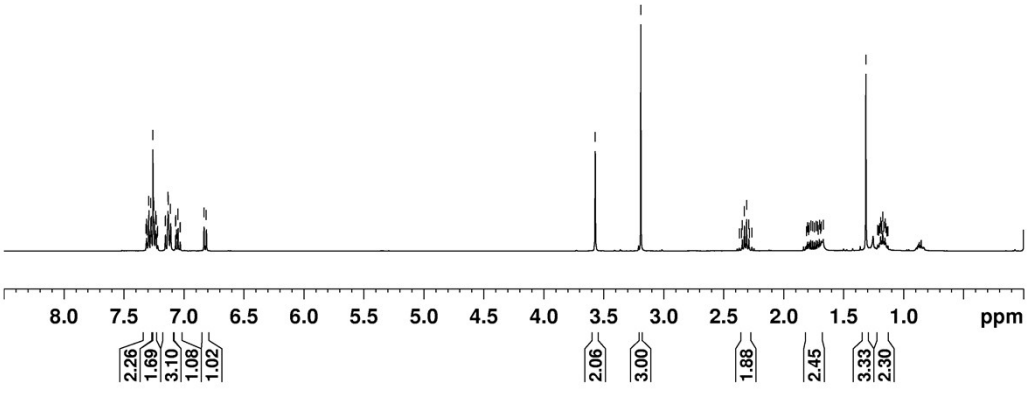
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2.06
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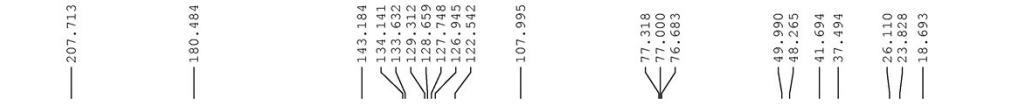
3.33
2.30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77.000
76.683
49.990
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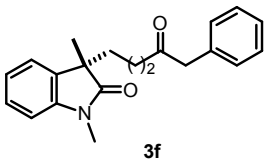
37.494
26.110
23.828
18.693



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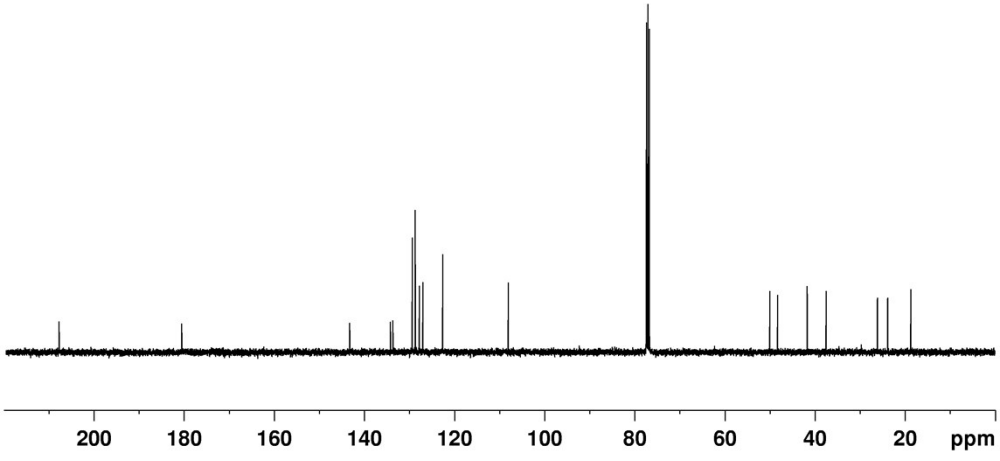
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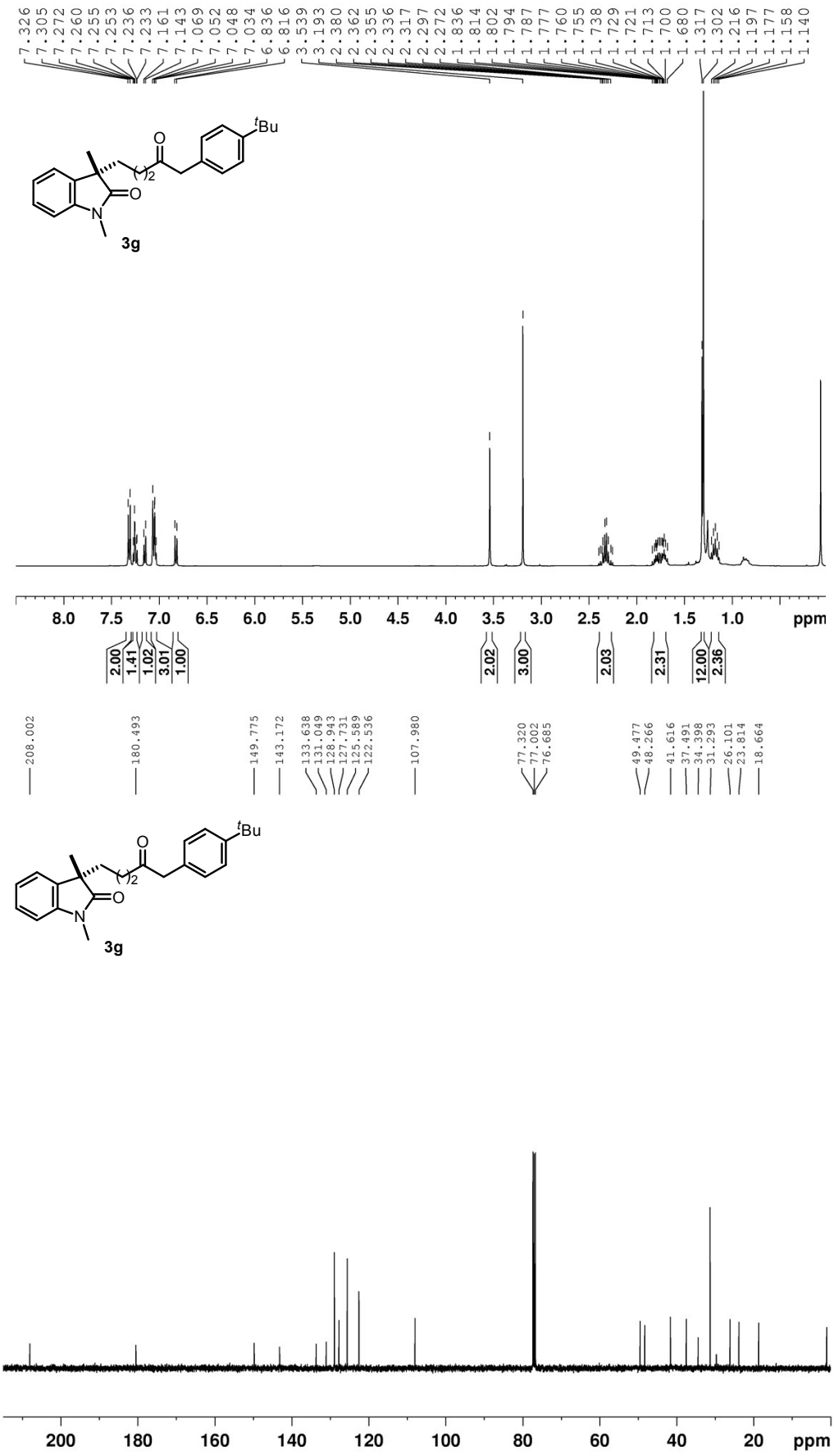
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7.034
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6.816
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3.193
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2.362
2.355
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2.317
2.297
2.272
1.836
1.814
1.802
1.794
1.787
1.777
1.760
1.755
1.738
1.729
1.721
1.713
1.700
1.680
1.317
1.302
1.216
1.197
1.177
1.158
1.140
tBu
O
O
N
3g
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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1.02
3.01
1.00
2.02
3.00
2.03
2.31
12.00
2.36
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180.493
149.775
143.172
133.638
131.049
128.943
127.731
125.589
122.536
107.980
77.320
77.002
76.685
49.477
48.266
41.616
37.491
34.398
31.293
26.101
23.814
18.664
tBu
O
O
N
3g
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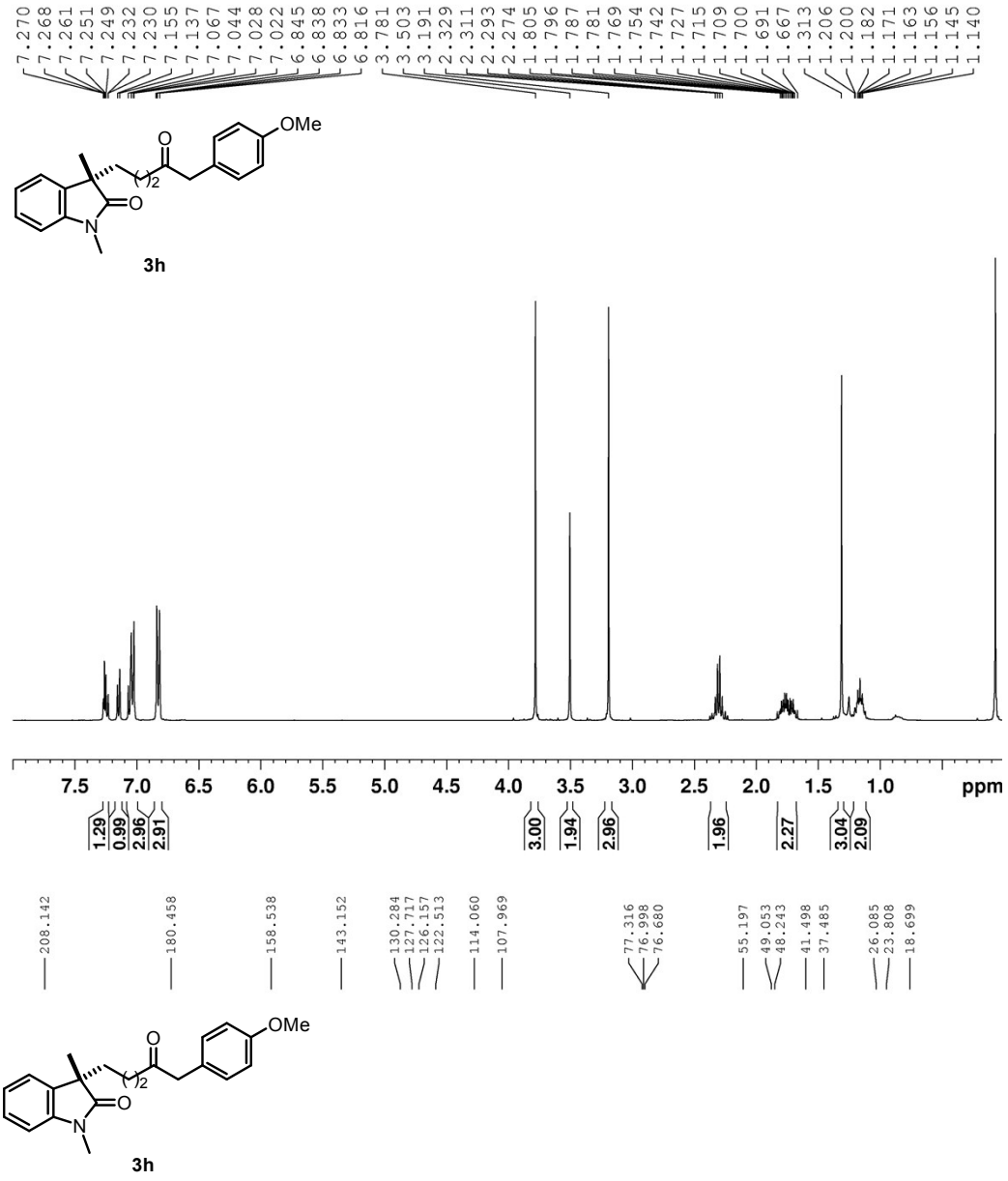
140
120
100
80
60
40
20
ppm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7.067
7.044
7.028
7.022
6.845
6.838
6.833
6.816
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3.503
3.191
2.329
2.311
2.293
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1.796
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1.781
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1.709
1.700
1.691
1.667
1.313
1.206
1.200
1.182
1.171
1.163
1.156
1.145
1.140
OMe
O
O
N
3h
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5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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2.96
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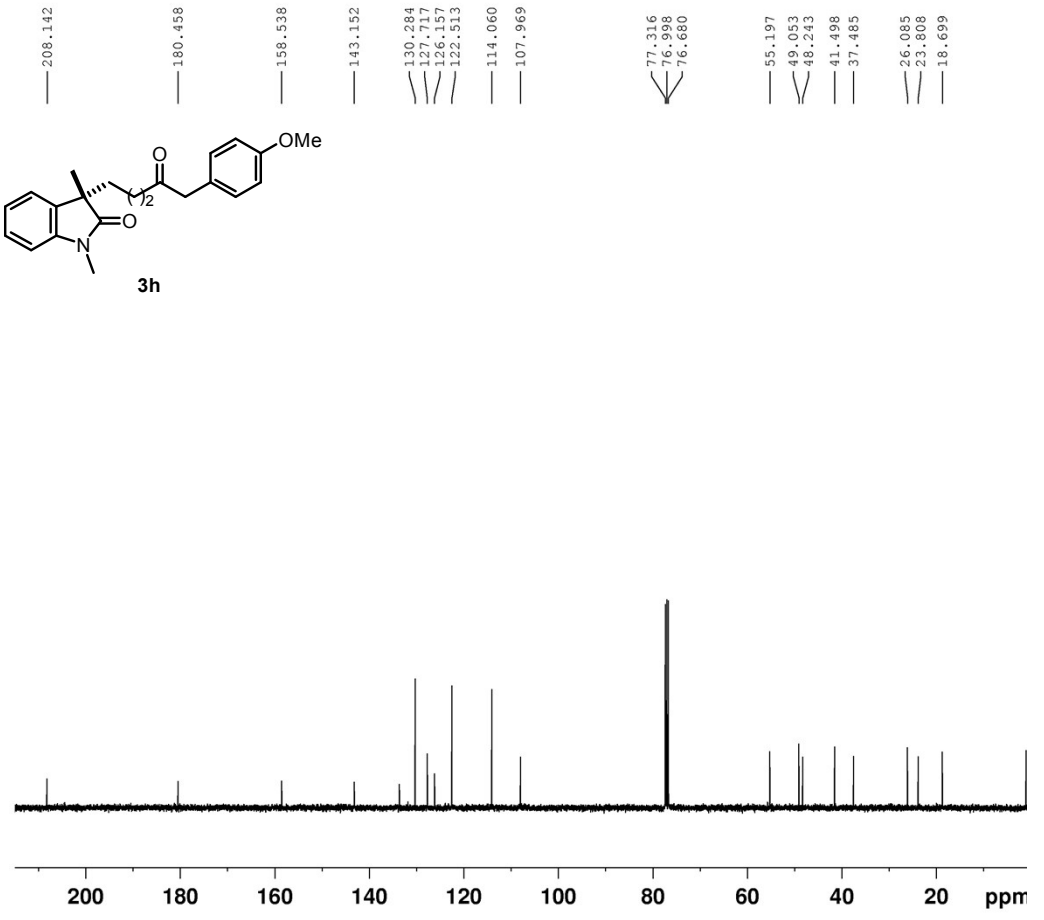
2.27
3.04
2.09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127.717
126.157
122.513
114.060
107.969
77.316
76.998
76.680
55.197
49.053
48.243
41.498
37.485
26.085
23.808
18.699
OMe
O
O
N
3h
200
180
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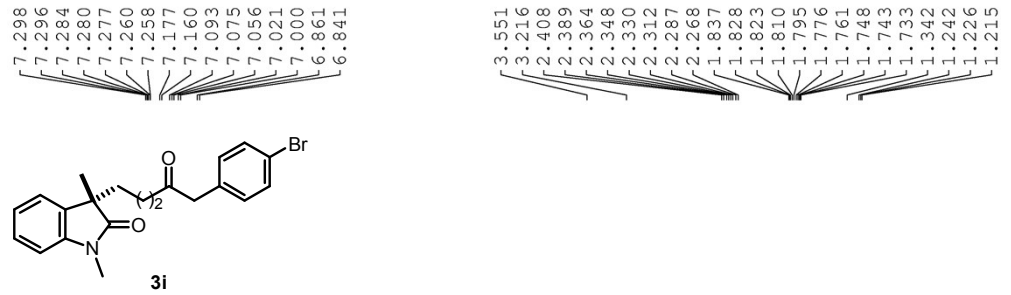
140
120
100
80
60
40
20
ppm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7.260
7.258
7.177
7.160
7.093
7.075
7.056
7.021
7.000
6.861
6.841
3.551
3.216
2.408
2.389
2.364
2.348
2.330
2.312
2.287
2.268
1.837
1.828
1.823
1.810
1.795
1.776
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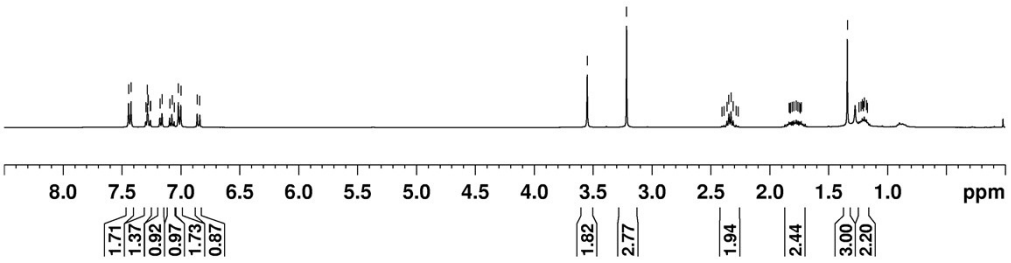
1.743
1.733
1.342
1.242
1.226
1.215
Br
O
O
N

3i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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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0.97
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0.87
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2.77
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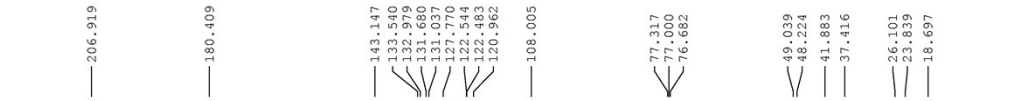
2.44
3.00
2.20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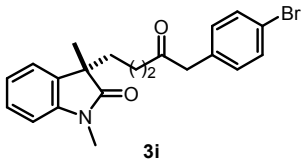
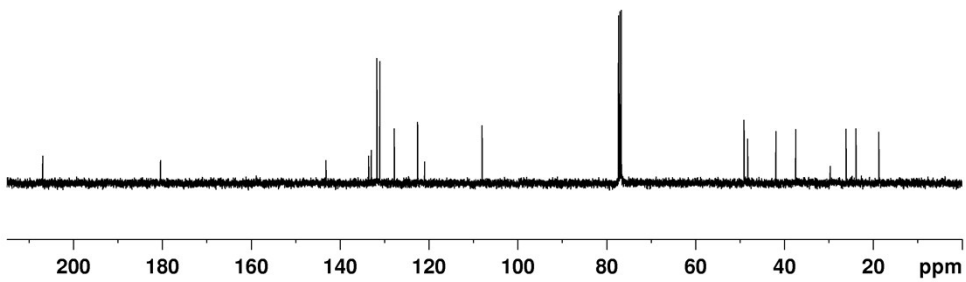

Br
O
O
N

3i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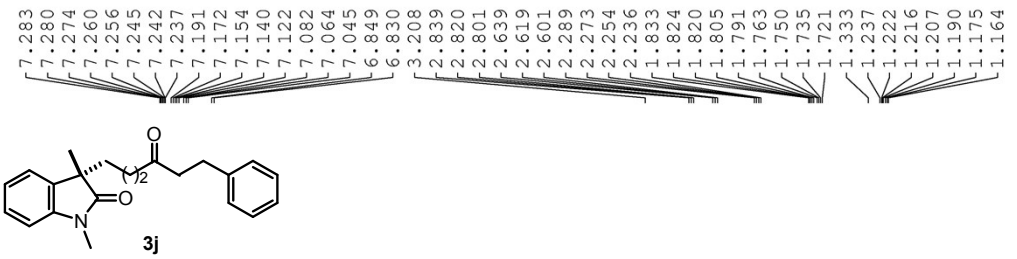
160
140
120
100
80
60
40
20
ppm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7.260
7.256
7.245
7.242
7.237
7.191
7.172
7.154
7.140
7.122
7.082
7.064
7.045
6.849
6.830
3.208
2.839
2.820
2.801
2.639
2.619
2.601
2.289
2.273
2.254
2.236
1.833
1.824
1.820
1.805
1.791
1.763
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1.721
1.333
1.237
1.222
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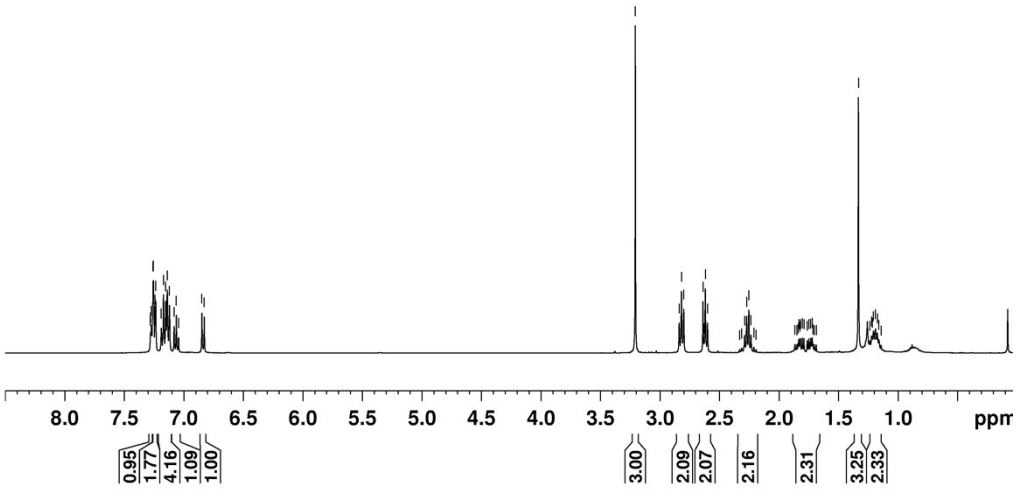
1.207
1.190
1.175
1.164

3j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5.0
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3.0
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2.0
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1.0
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3.00
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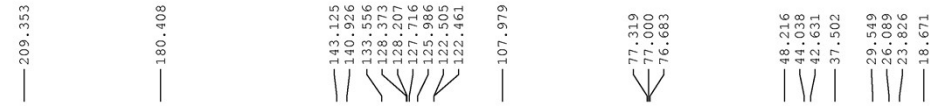
3.25
2.33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122.461
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76.683
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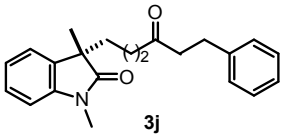
23.826
18.671



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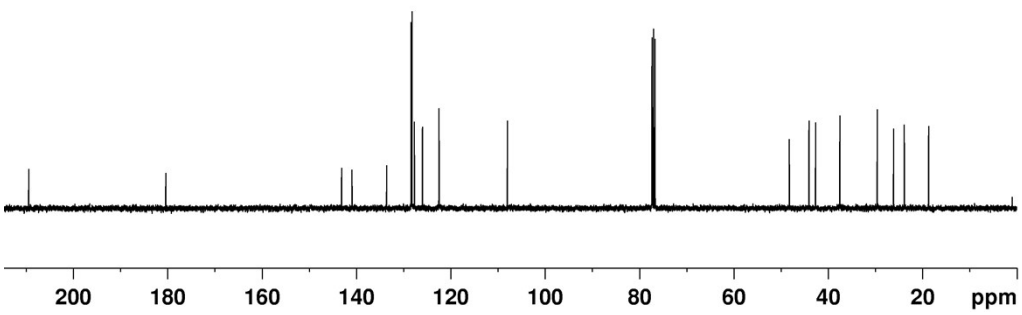
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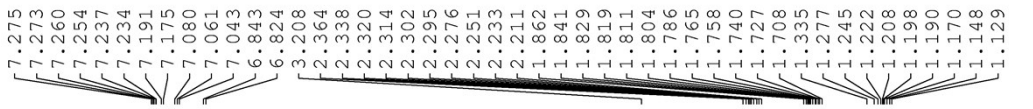
160
140
120
100
80
60
40
20
ppm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6.824
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2.338
2.320
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2.302
2.295
2.276
2.251
2.233
2.211
1.862
1.841
1.829
1.819
1.811
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1.786
1.765
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1.740
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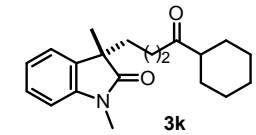
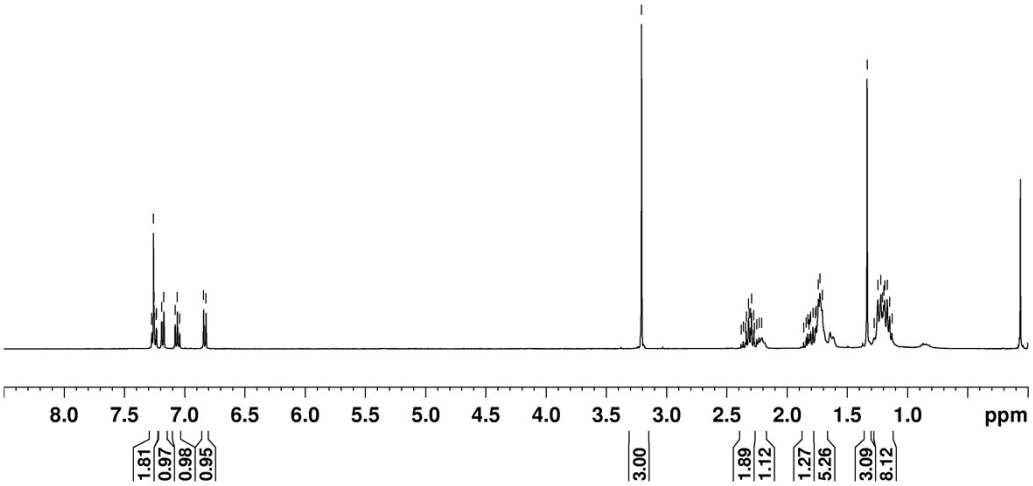
1.148
1.129


O
O
N
3k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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.81
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0.98
0.95
3.00
1.89
1.12
1.27
5.26
3.09
8.12


213.588
180.581
143.186
133.691
127.723
122.576
122.548
107.994
77.321
77.004
76.685
50.610
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37.687
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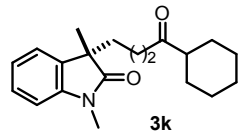
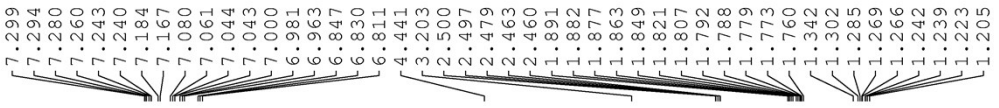
26.140
25.770
25.596
25.572
23.860
18.624


O
O
N
3k

200
180
160
140
120
100
80
60
40
20
ppm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7.061
7.044
7.043
7.000
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6.963
6.847
6.830
6.811
4.441
3.203
2.500
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1.882
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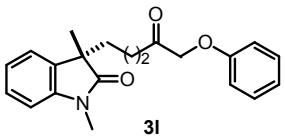
1.205



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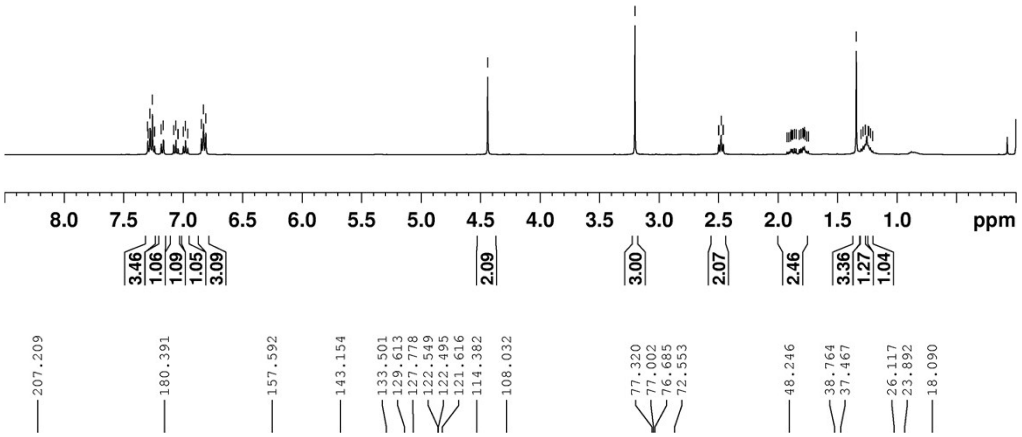
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ppm
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1.06
1.09
1.05
3.09
2.09
3.00
2.07
2.46
3.36
1.27
1.04

207.209
180.391
157.592
143.154
133.501
129.613
127.778
122.549
122.495
121.616
114.382
108.032
77.320
77.002
76.685
72.553
48.246
38.764
37.467
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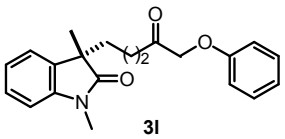
23.892
18.090



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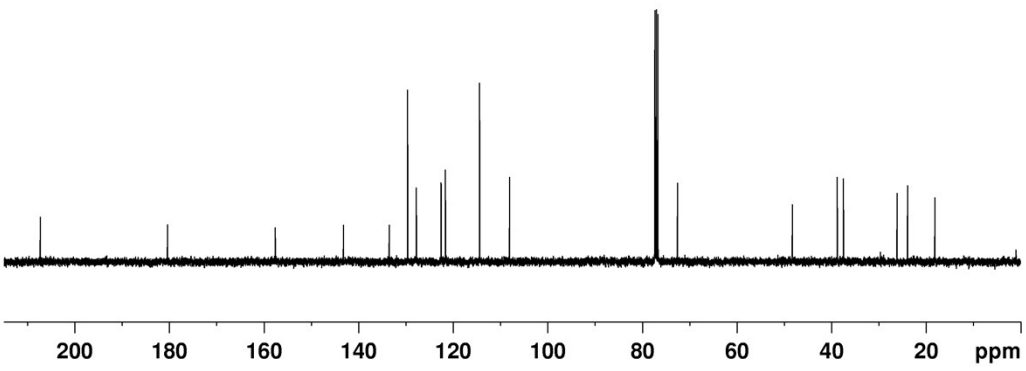
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180
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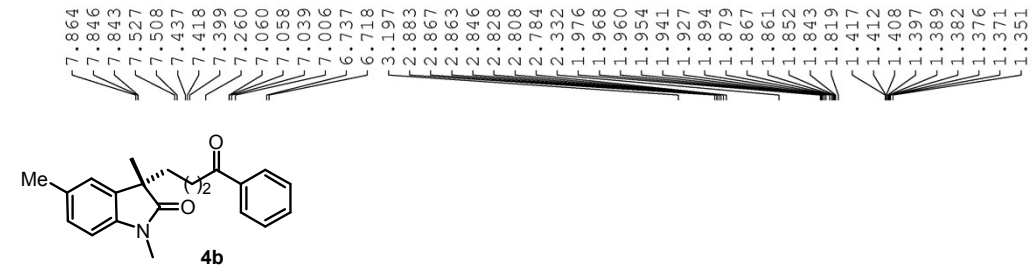
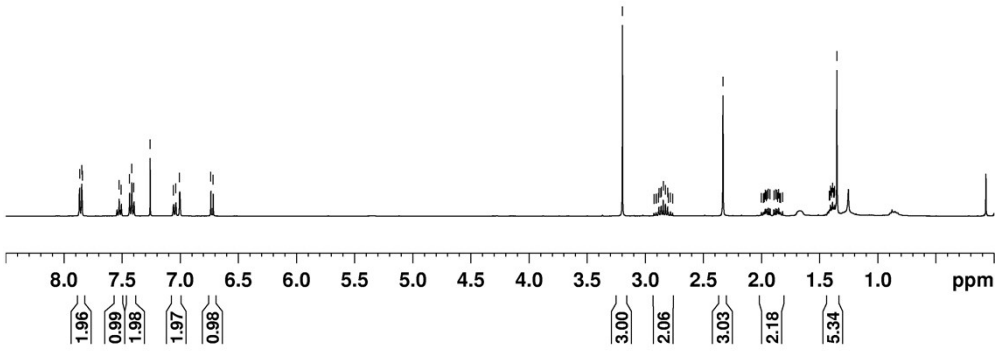
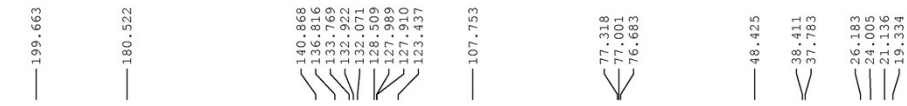
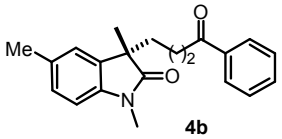
140
120
100
80
60
40
20
ppm

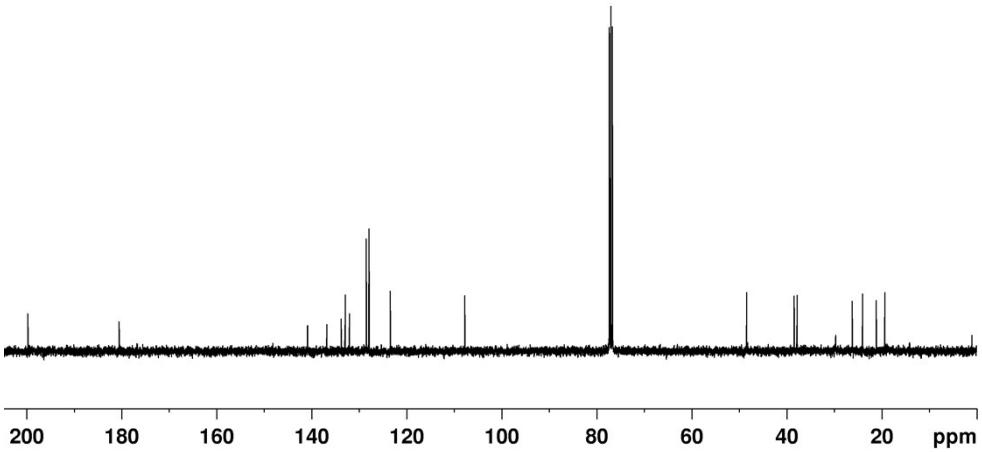
^{1}H NMR and ^{13}C NMR Spectra of Products 4

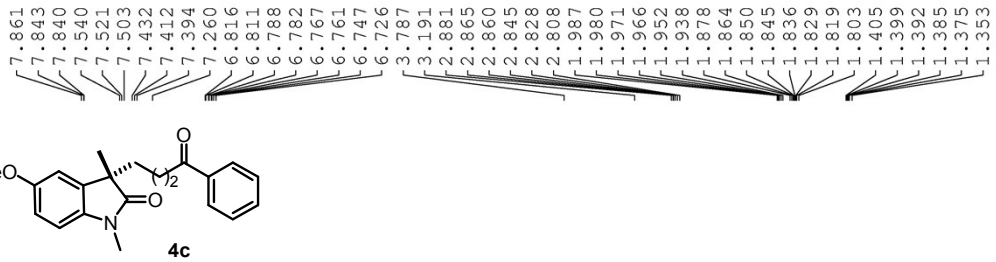








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1.850
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1.836
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1.375
1.353

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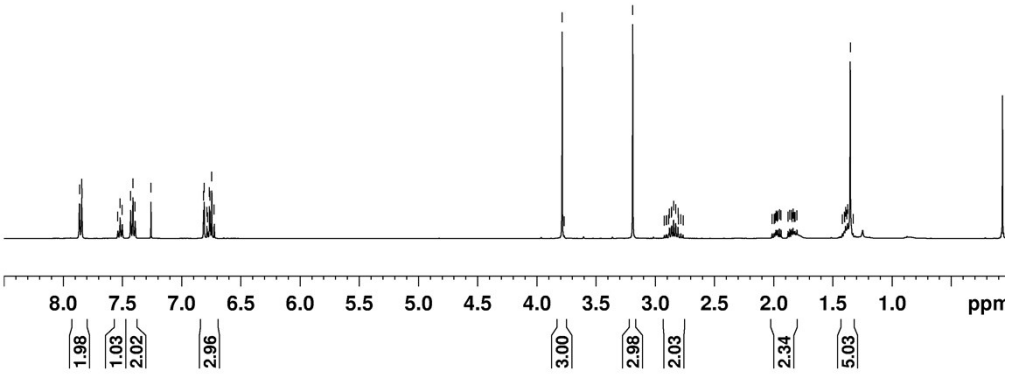
O
O
N

4c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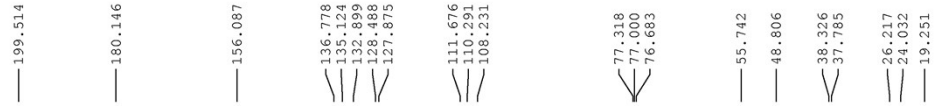
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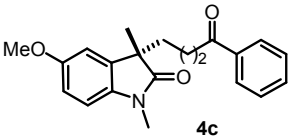
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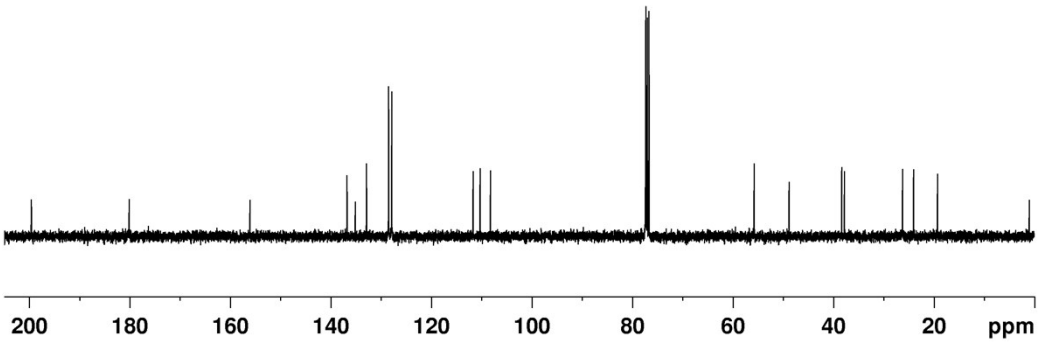
N

4c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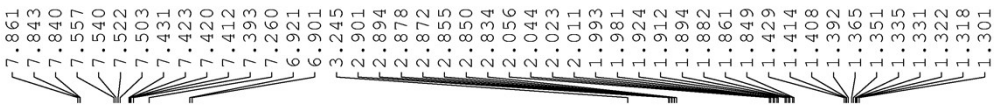
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6.901
3.245
2.901
2.894
2.878
2.872
2.855
2.850
2.834
2.056
2.044
2.023
2.011
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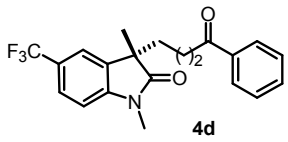
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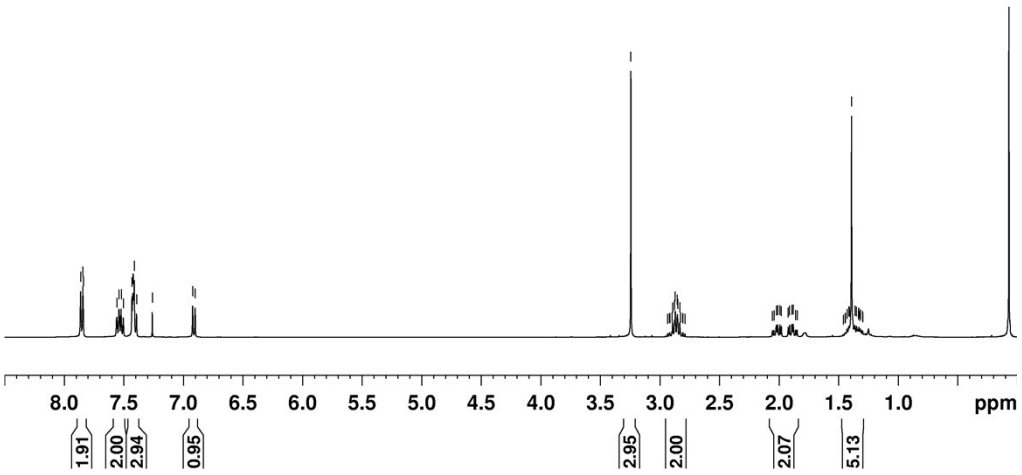
O
N

4d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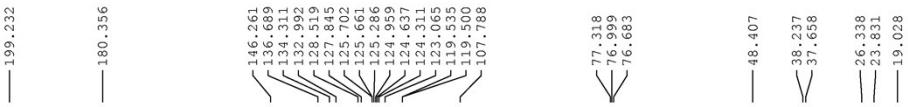
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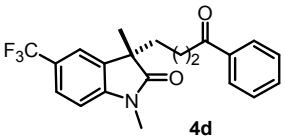
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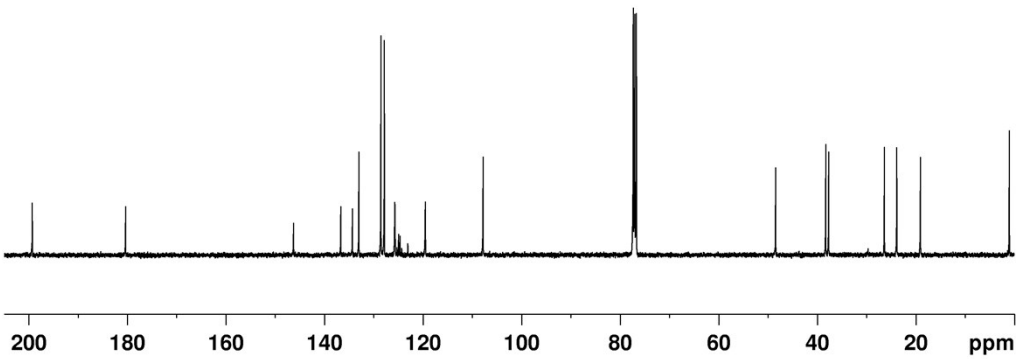
O
O
N

4d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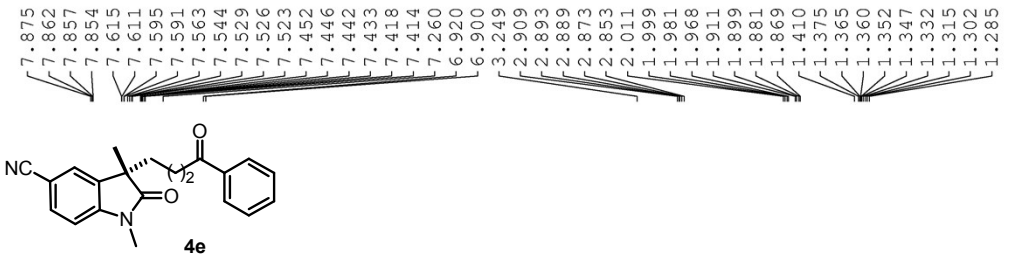
ppm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7.526
7.523
7.452
7.446
7.442
7.433
7.418
7.414
7.260
6.920
6.900
3.249
2.909
2.893
2.889
2.873
2.853
2.011
1.999
1.981
1.968
1.911
1.899
1.881
1.869
1.410
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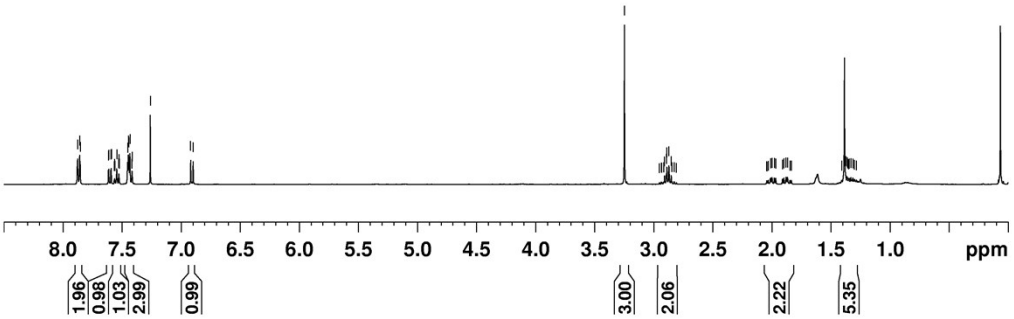
1.347
1.332
1.315
1.302
1.285
NC
O
O
N
4e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4.0
3.5
3.0
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0.99
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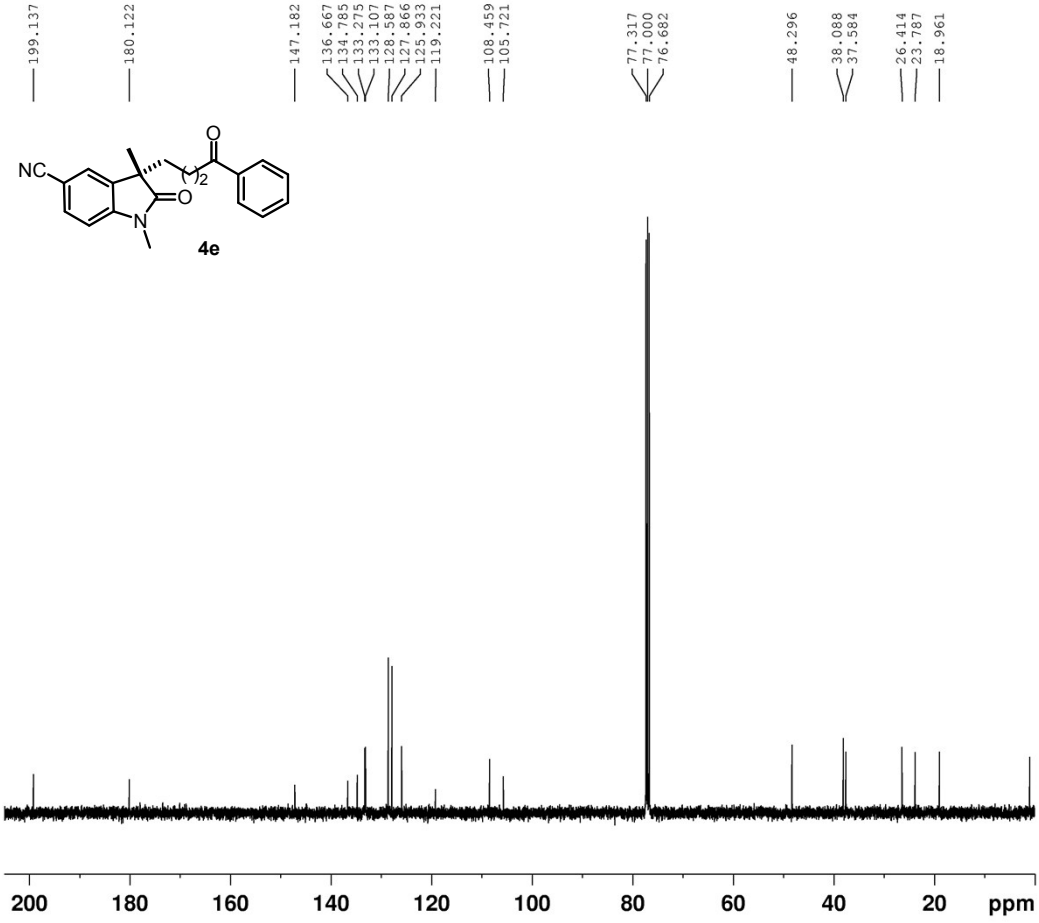
2.22
5.35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77.317
77.000
76.682
48.296
38.088
37.584
26.414
23.787
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NC
O
O
N
4e
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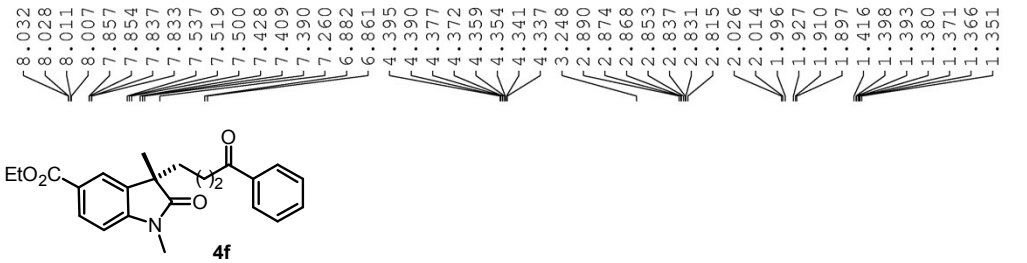
140
120
100
80
60
40
20
ppm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7.409
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7.260
6.882
6.861
4.395
4.390
4.377
4.372
4.359
4.354
4.341
4.337
3.248
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2.874
2.868
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2.837
2.831
2.815
2.026
2.014
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1.927
1.910
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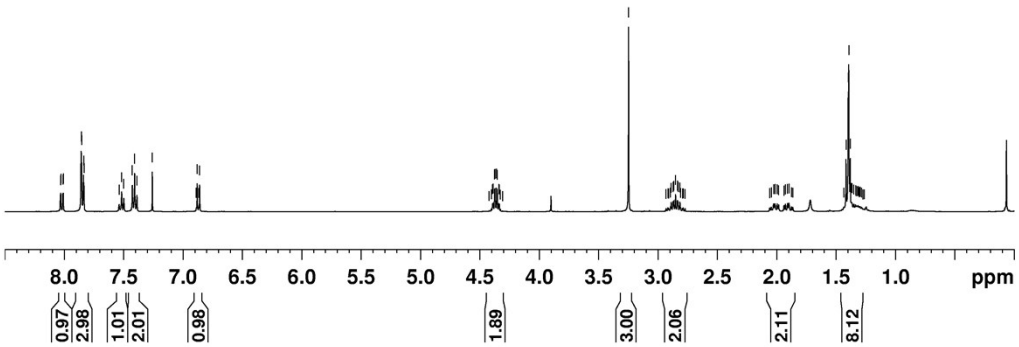
1.393
1.380
1.371
1.366
1.351

EtO2C
O
O
N

4f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3.0
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2.0
1.5
1.0
ppm
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2.98
1.01
2.01
0.98
1.89
3.00
2.06
2.11
8.12


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132.954
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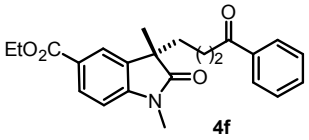
EtO2C
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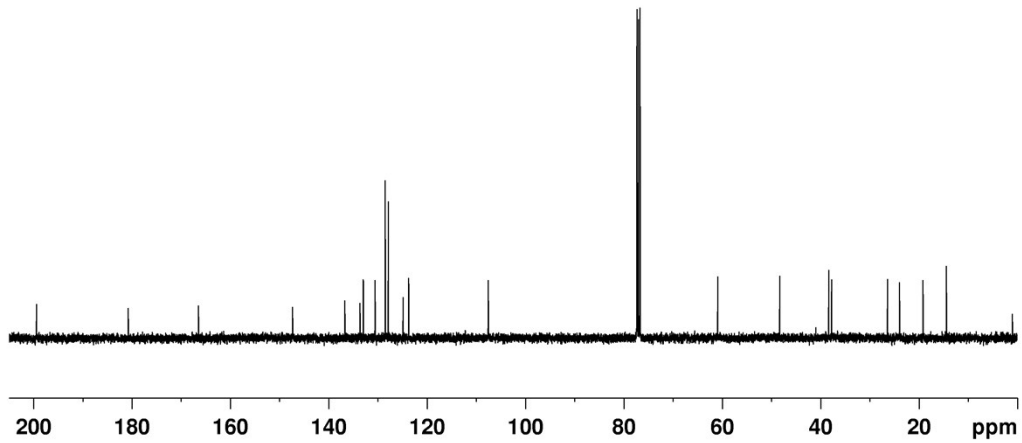
O
N

4f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80
60
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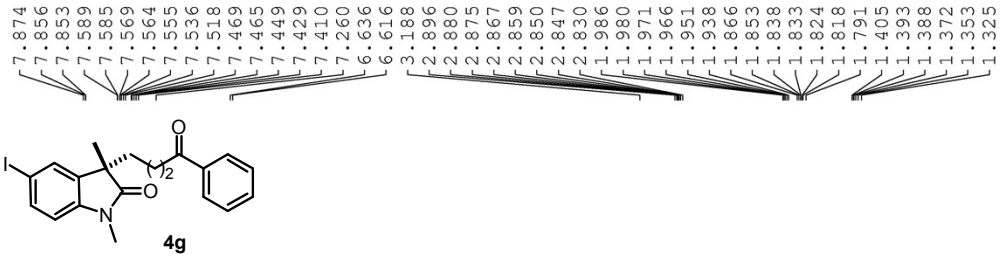
20
ppm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7.449
7.429
7.410
7.260
6.636
6.616
3.188
2.896
2.880
2.875
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2.850
2.847
2.830
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1.980
1.971
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1.938
1.866
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1.838
1.833
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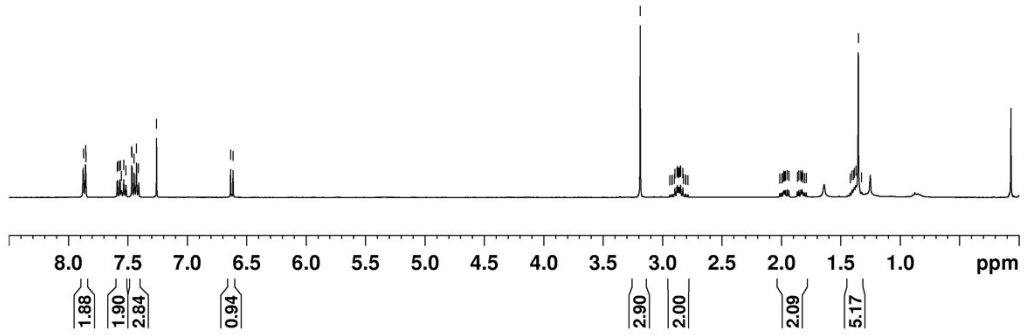
1.388
1.372
1.353
1.325

I
O
O
N

4g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4.0
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2.0
1.5
1.0
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0.94
2.90
2.00
2.09
5.17


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136.227
133.000
131.386
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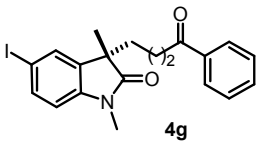
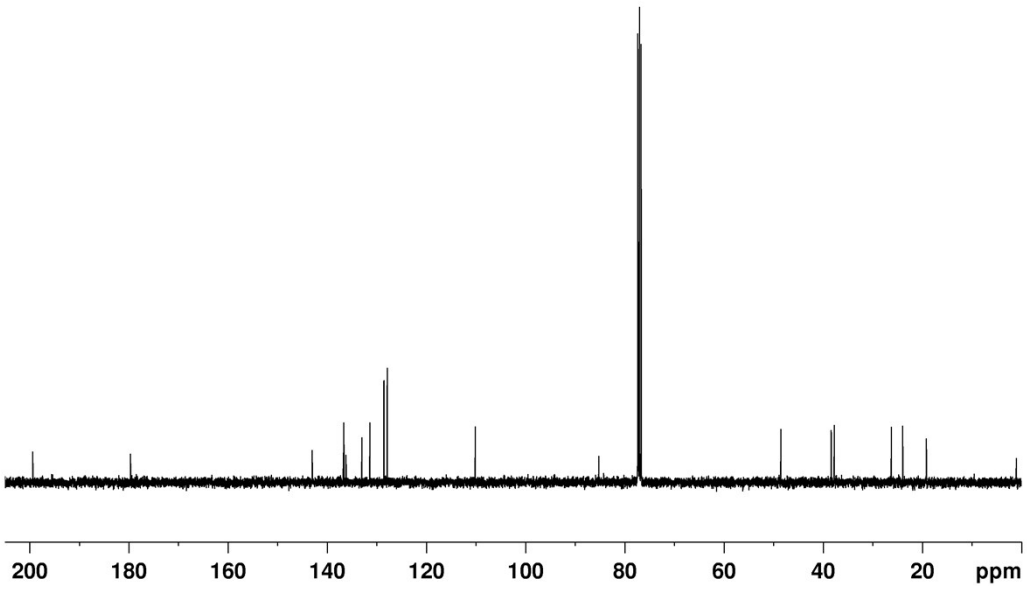
23.905
19.132


I
O
O
N

4g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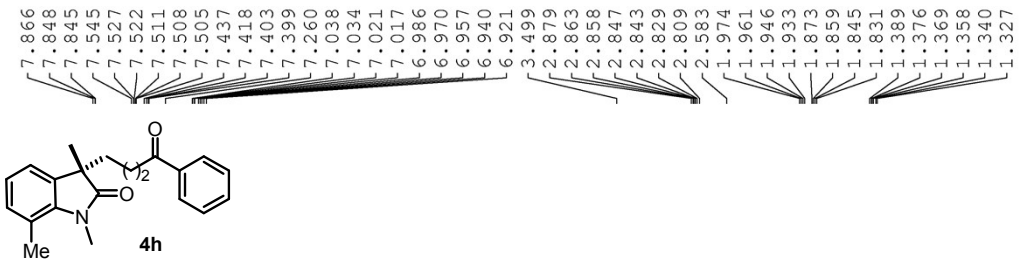
ppm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7.399
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7.038
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7.021
7.017
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6.940
6.921
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2.829
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1.327

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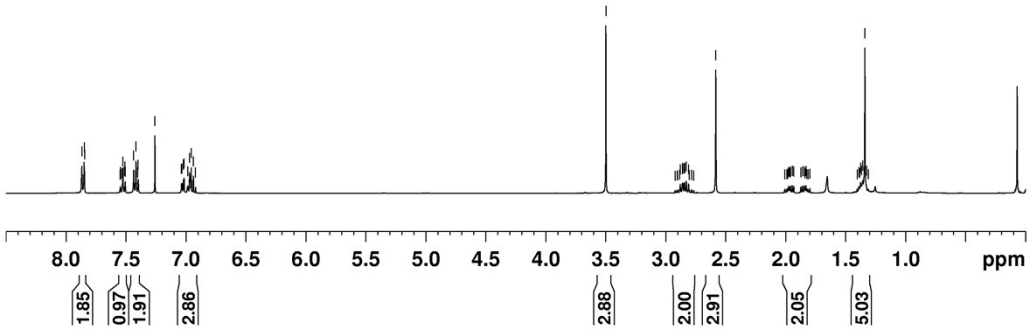
2
O
N
Me

4h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2.00
2.91
2.05
5.03


199.618
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140.994
136.826
134.340
132.917
131.489
128.512
127.908
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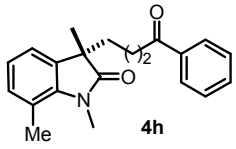
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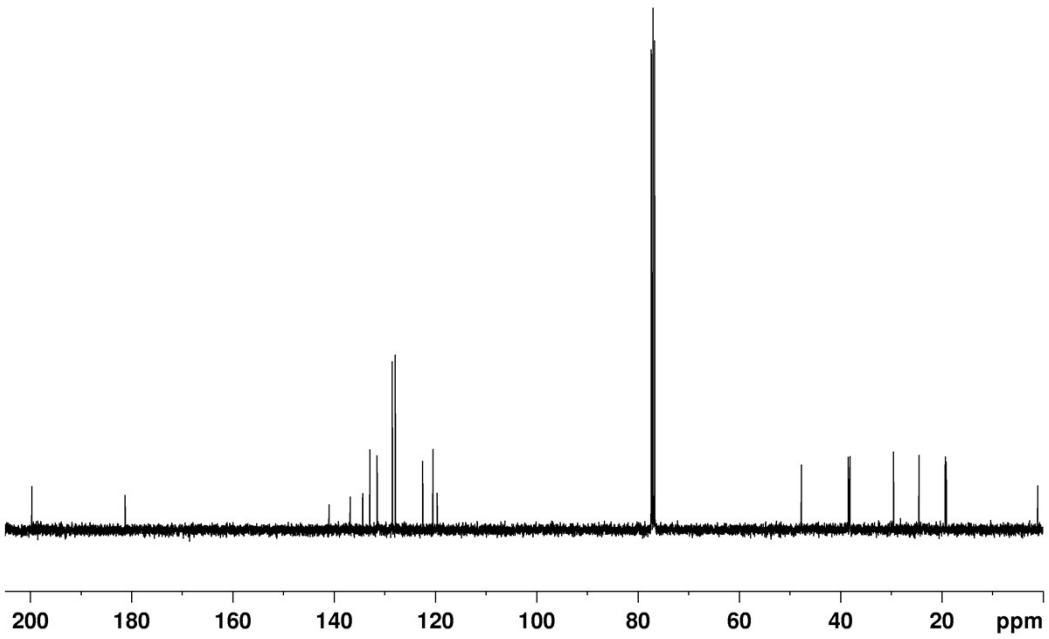
O
N
Me

4h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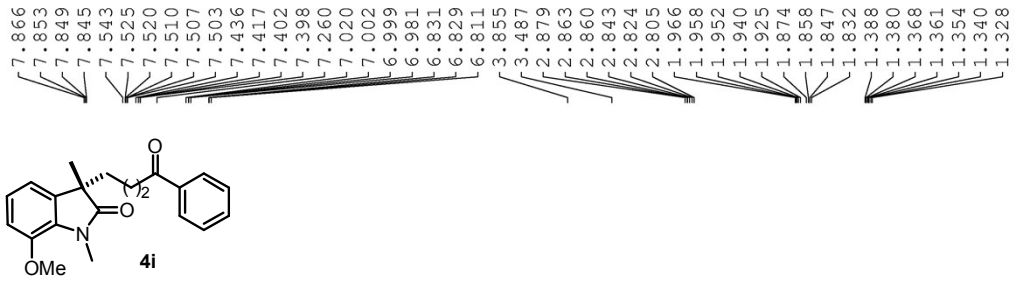
ppm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2
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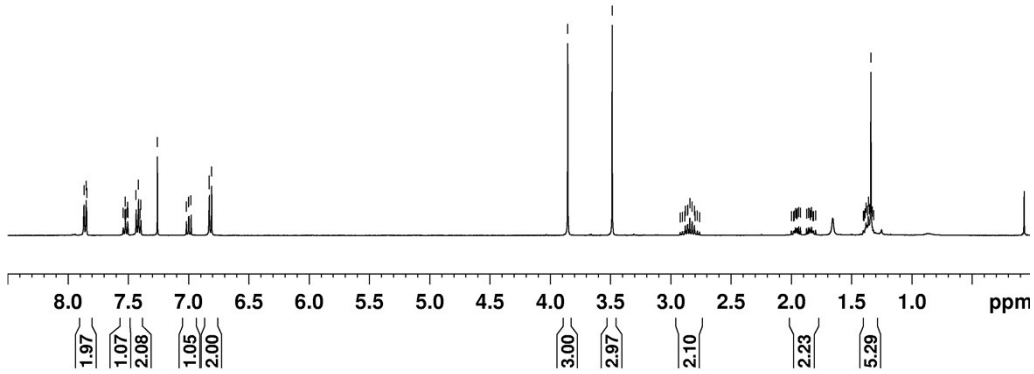
N
OMe

4i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3.00
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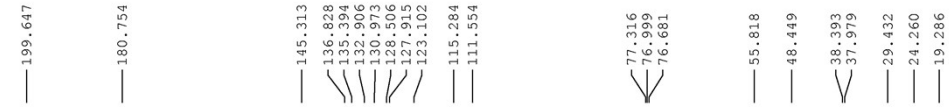
2.23
5.29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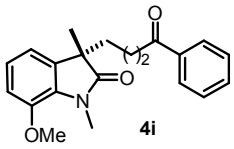
O
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O
N
OMe

4i

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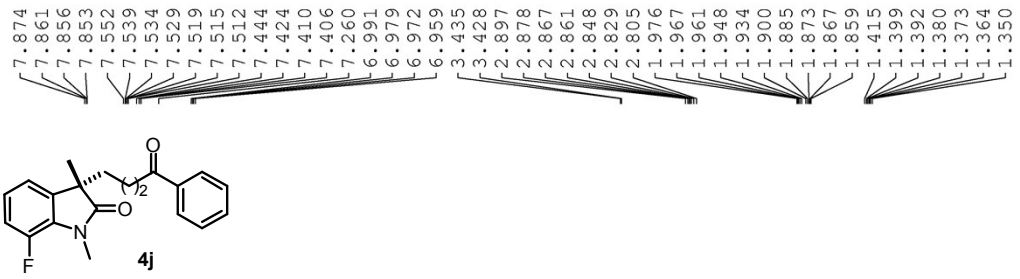
140
120
100
80
60
40
20
ppm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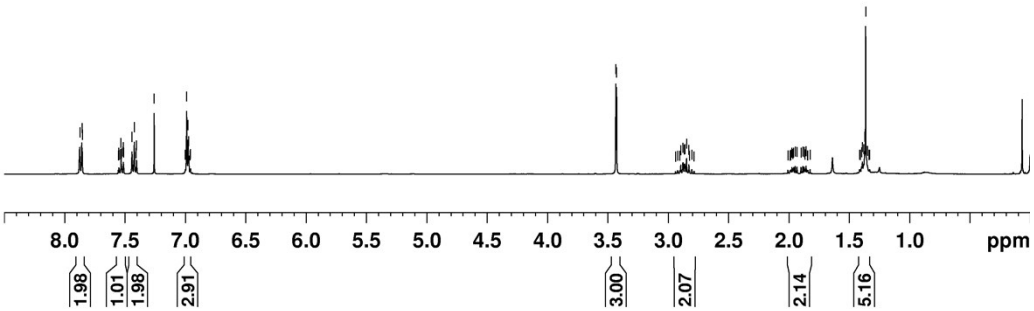
O
N
F

4j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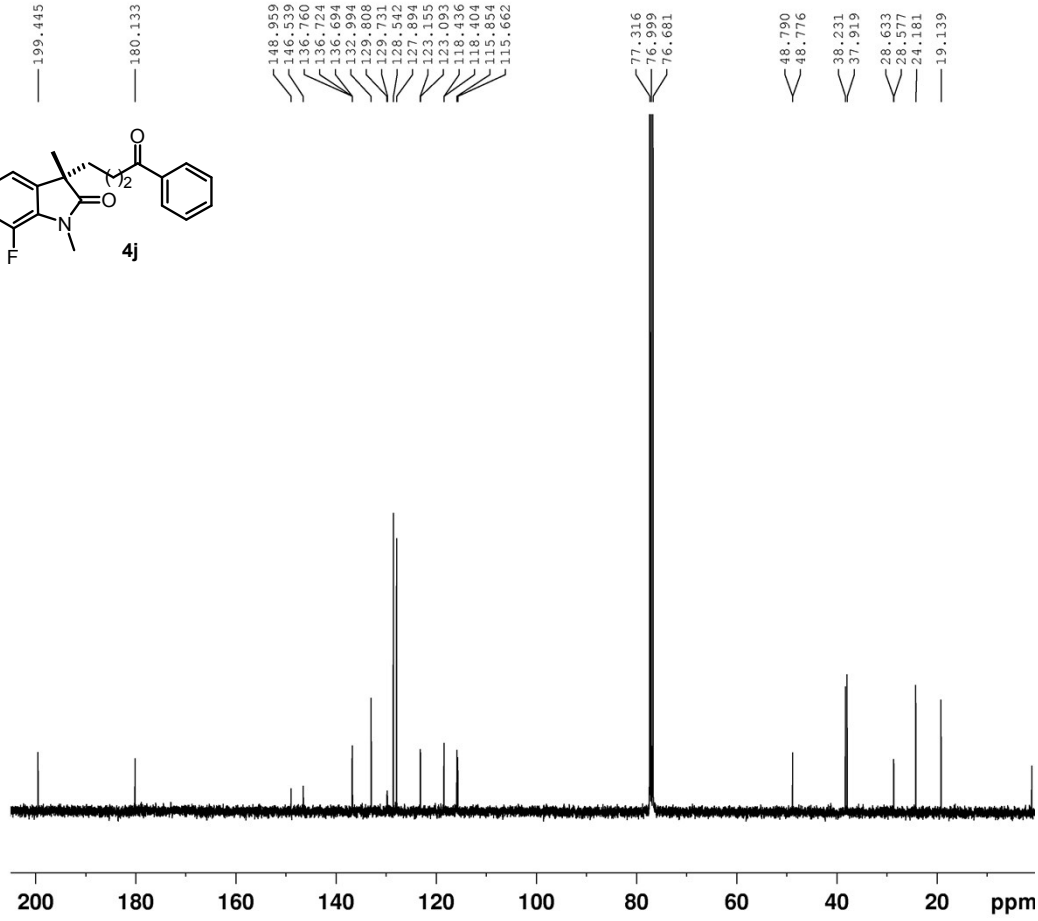
2.14
5.16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132.994
129.808
129.731
128.542
127.894
123.155
123.093
118.436
118.404
115.854
115.662
77.316
76.999
76.681
48.790
48.776
38.231
37.919
28.633
28.577
24.181
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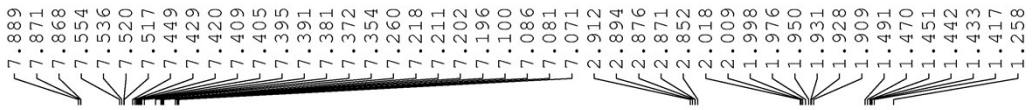
160
140
120
100
80
60
40
20
ppm

O
2
O
N
F

4j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7.391
7.381
7.372
7.354
7.260
7.218
7.211
7.202
7.196
7.100
7.086
7.081
7.071
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2.894
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2.871
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2.018
2.009
1.998
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1.451
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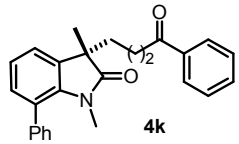
1.258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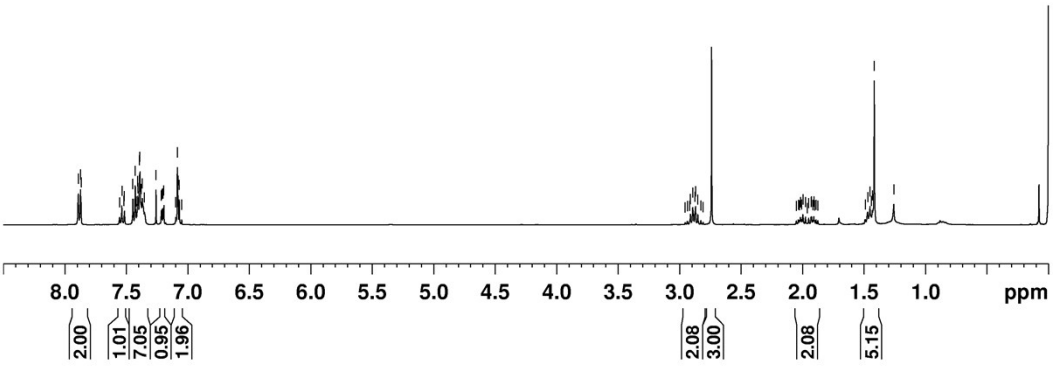
O
N
Ph

4k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4.5
4.0
3.5
3.0
2.5
2.0
1.5
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ppm
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7.05
0.95
1.96
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3.00
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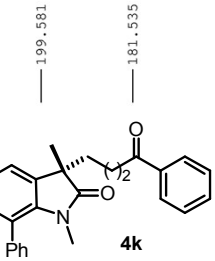
5.15


O
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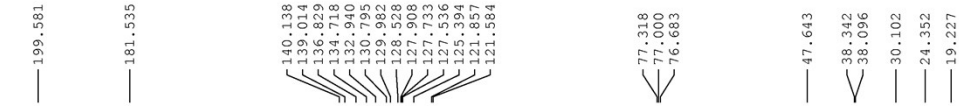
O
N
Ph

4k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130.795
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128.528
127.908
127.733
127.536
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77.318
77.000
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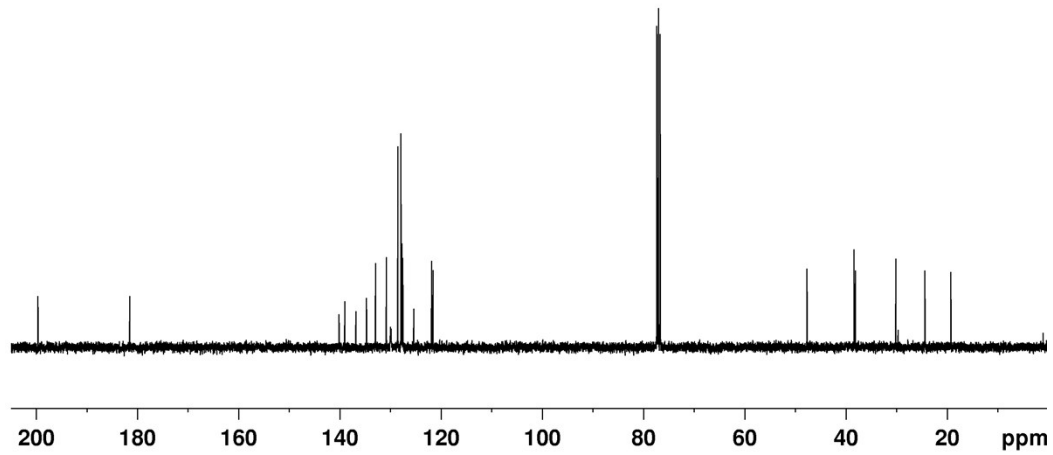
47.643
38.342
38.096
30.102
24.352
19.227


200
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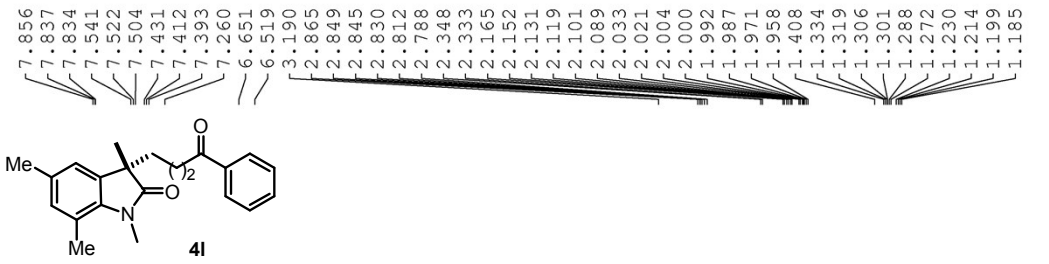
100
80
60
40
20
ppm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7.834
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7.504
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7.412
7.393
7.260
6.651
6.519
3.190
2.865
2.849
2.845
2.830
2.812
2.788
2.348
2.333
2.165
2.152
2.131
2.119
2.101
2.089
2.033
2.021
2.004
2.000
1.992
1.987
1.971
1.958
1.408
1.334
1.319
1.306
1.301
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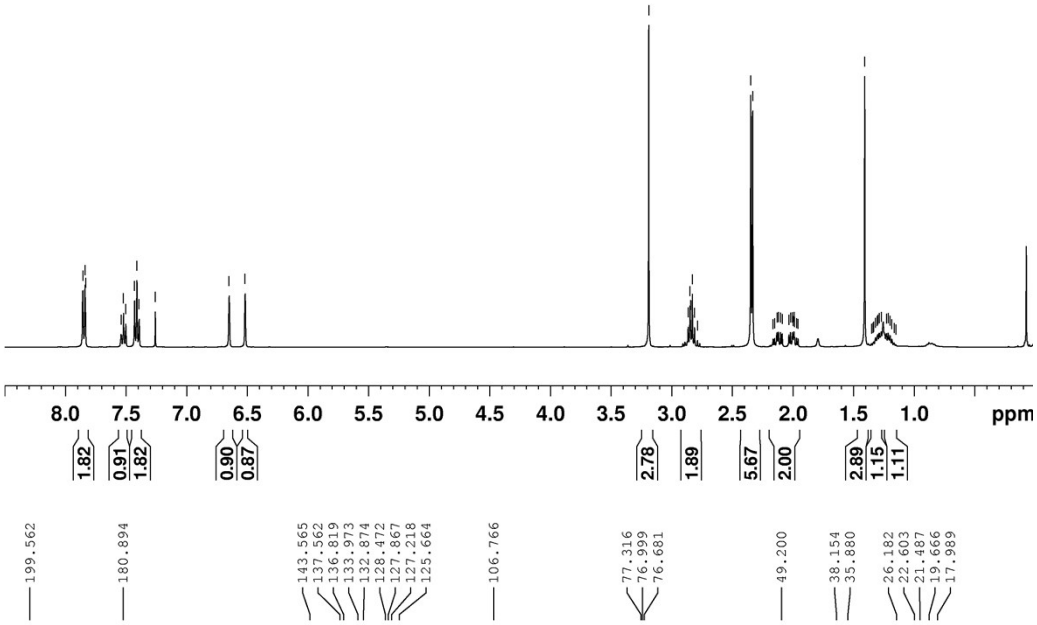
1.272
1.230
1.214
1.199
1.185

Me
O
)2
O
N
Me

4l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4.0
3.5
3.0
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2.0
1.5
1.0
ppm
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0.91
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0.90
0.87
2.78
1.89
5.67
2.00
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1.15
1.11

199.562
180.894
143.565
137.562
136.819
133.973
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128.472
127.867
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125.664
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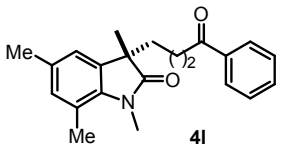
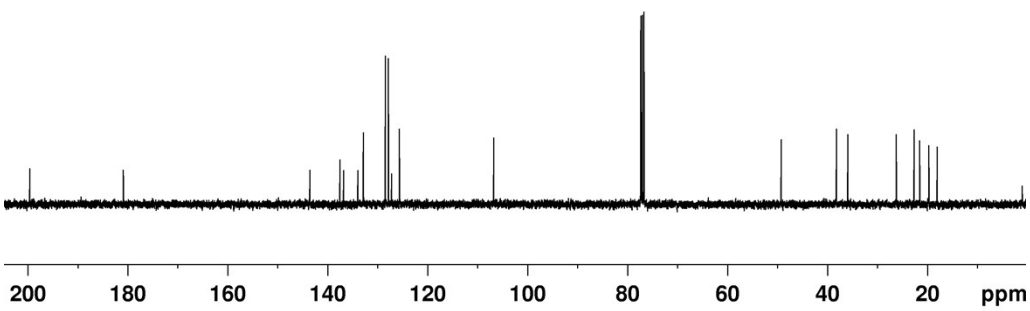
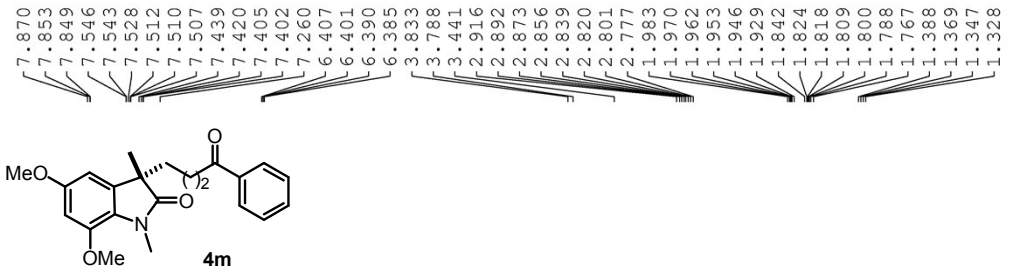
49.200
38.154
35.880
26.182
22.603
21.487
19.666
17.989


Me
O
)2
O
N
Me

4l


200
180
160
140
120
100
80
60
40
20
ppm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7.512
7.510
7.507
7.439
7.420
7.405
7.402
7.260
6.407
6.401
6.390
6.385
3.833
3.788
3.441
2.916
2.892
2.873
2.856
2.839
2.820
2.801
2.777
1.983
1.970
1.962
1.953
1.946
1.929
1.842
1.824
1.818
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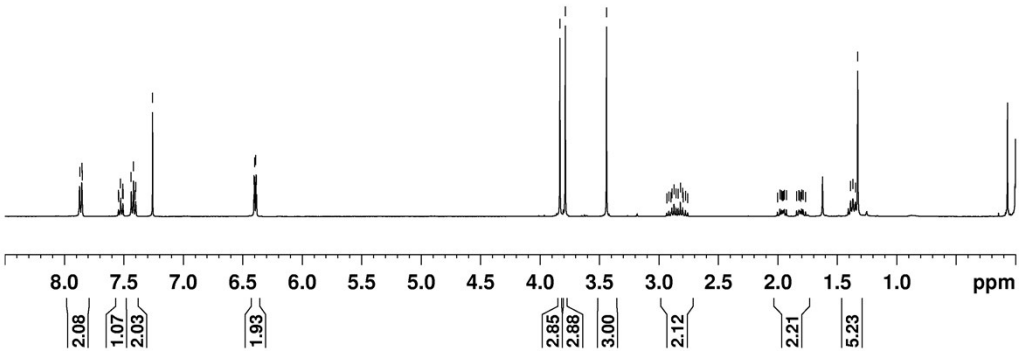
1.800
1.788
1.767
1.388
1.369
1.347
1.328

MeO
O
N
OMe

4m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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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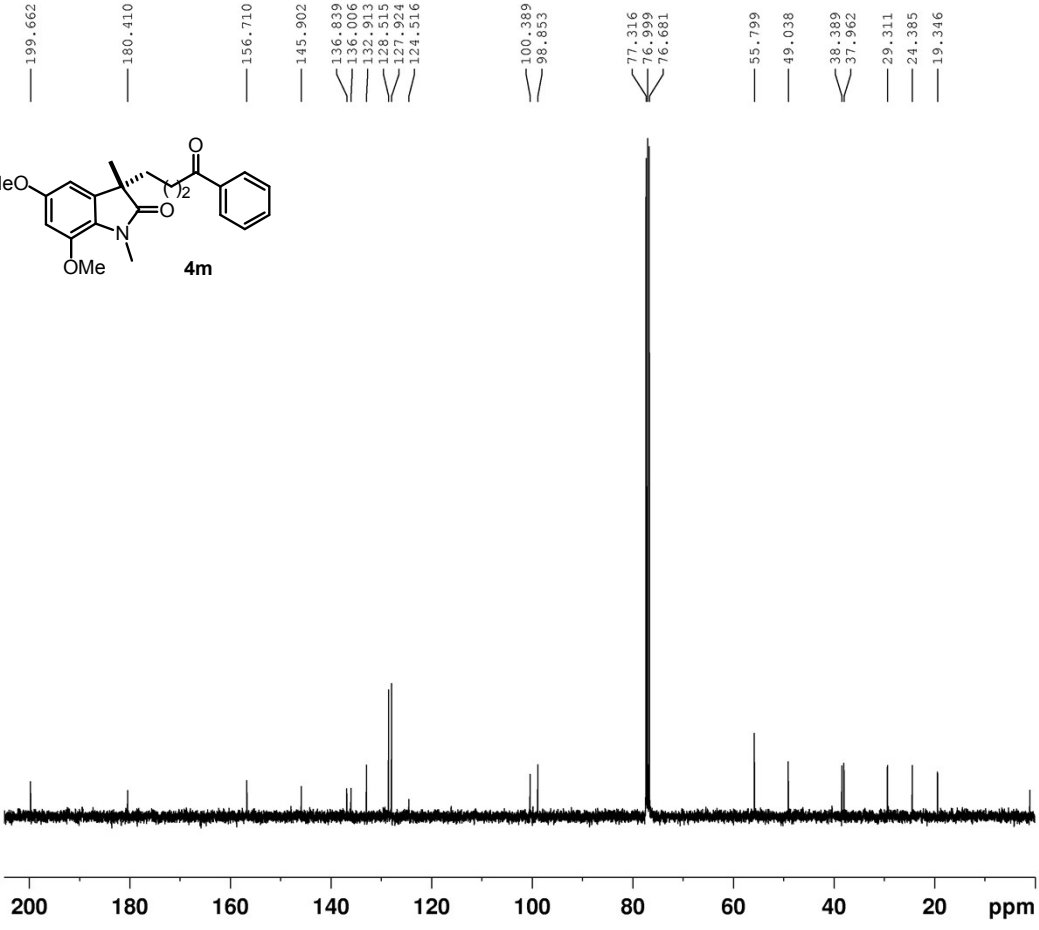
1.07
2.03
1.93
2.85
2.88
3.00
2.12
2.21
5.23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156.710
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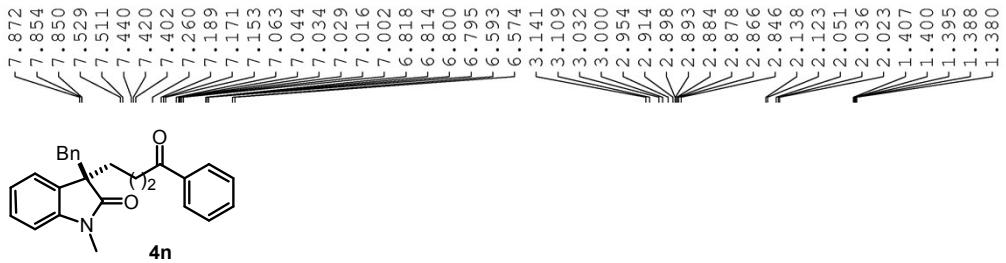
136.839
136.006
132.913
128.515
127.924
124.516
100.389
98.853
77.316
76.999
76.681
55.799
49.038
38.389
37.962
29.311
24.385
19.346
200
180
160
140
120
100
80
60
40
20
ppm

MeO
O
N
OMe

4m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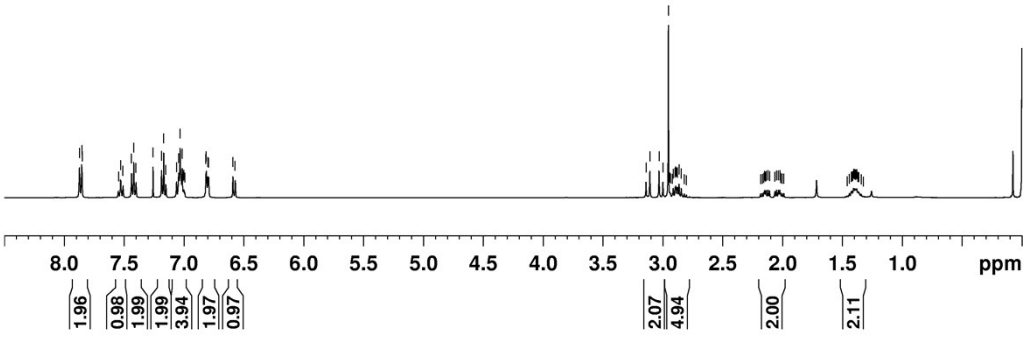
7.850
7.529
7.511
7.440
7.420
7.402
7.260
7.189
7.171
7.153
7.063
7.044
7.034
7.029
7.016
7.002
6.818
6.814
6.800
6.795
6.593
6.574
3.141
3.109
3.032
3.000
2.954
2.914
2.898
2.893
2.884
2.878
2.866
2.846
2.138
2.123
2.051
2.036
2.023
1.407
1.400
1.395
1.388
1.380

Bn
O
2
O
N

4n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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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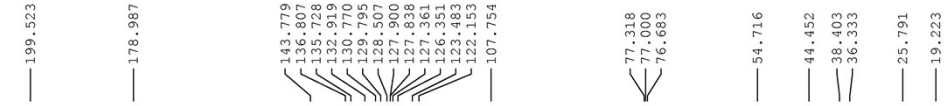
0.98
1.99
1.99
3.94
1.97
0.97
2.07
4.94
2.00
2.11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143.779
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135.728
132.919
130.770
129.795
128.507
127.900
127.838
127.361
126.351
123.483
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107.754
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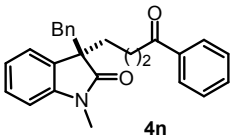
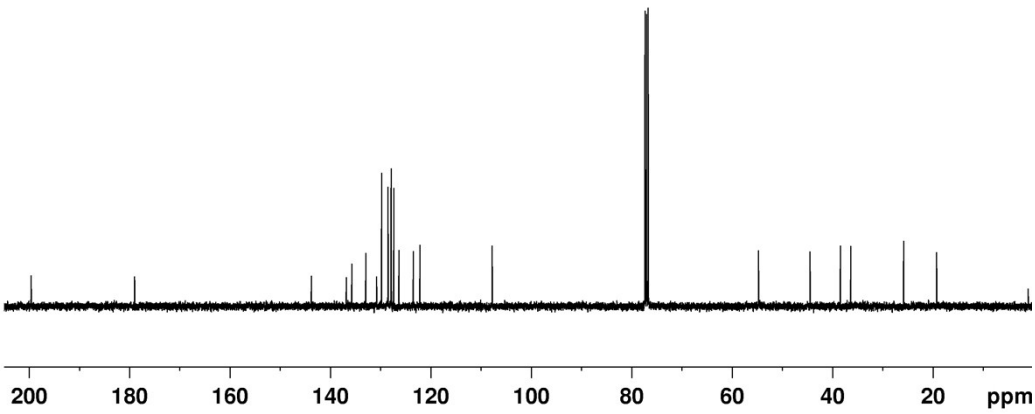
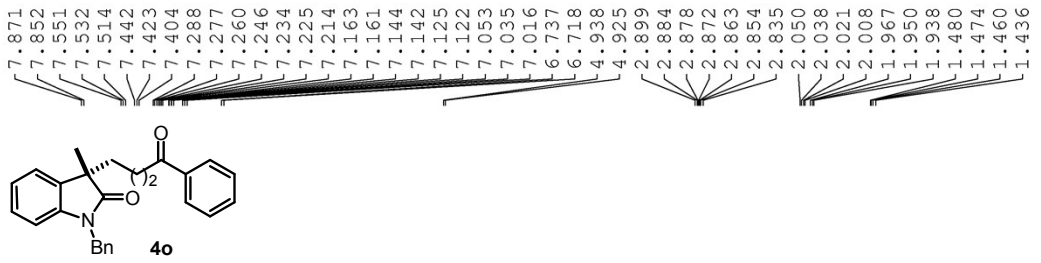
77.000
76.683
54.716
44.452
38.403
36.333
25.791
19.223


Bn
O
2
O
N

4n


200
180
160
140
120
100
80
60
40
20
ppm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7.852
7.551
7.532
7.514
7.442
7.423
7.404
7.288
7.277
7.260
7.246
7.234
7.225
7.214
7.163
7.161
7.144
7.142
7.125
7.122
7.053
7.035
7.016
6.737
6.718
4.938
4.925
2.899
2.884
2.878
2.872
2.863
2.854
2.835
2.050
2.038
2.021
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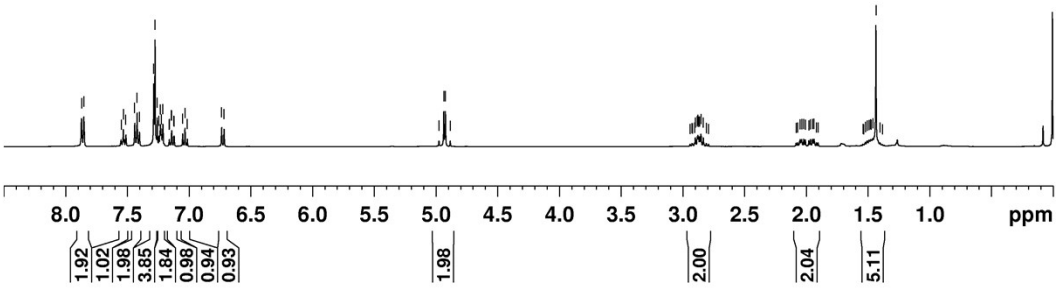
1.950
1.938
1.480
1.474
1.460
1.436
O
O
N
Bn
4o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7.0
6.5
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5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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3.85
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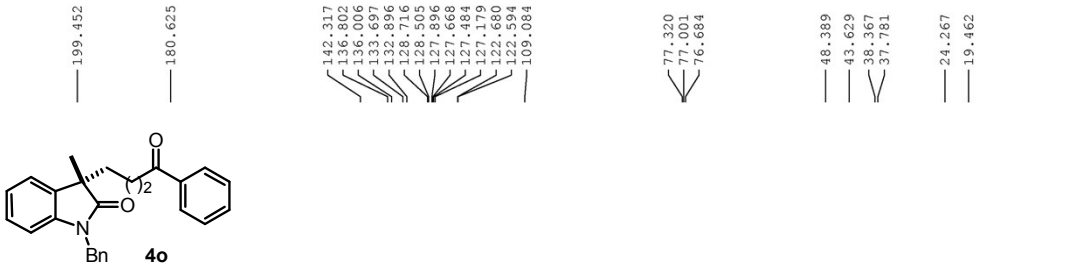
0.93
1.98
2.00
2.04
5.11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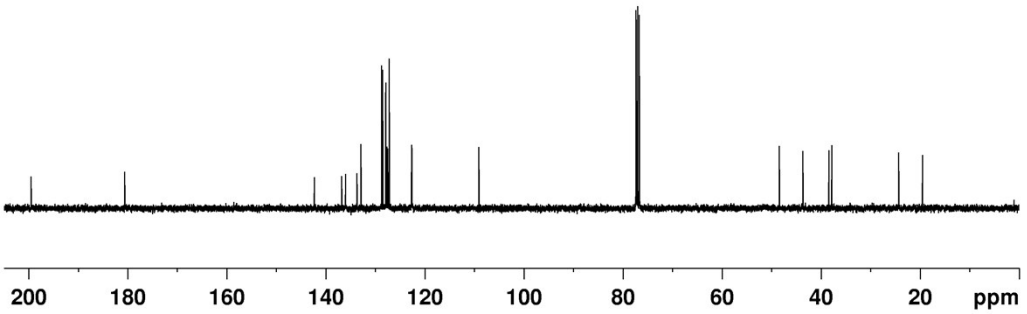
128.716
128.505
127.896
127.668
127.484
127.179
122.680
122.594
109.084
77.320
77.001
76.684
48.389
43.629
38.367
37.781
24.267
19.462
O
O
N
Bn
4o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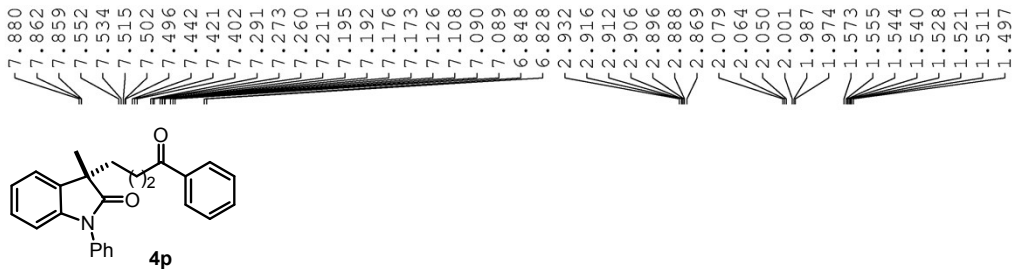
180
160
140
120
100
80
60
40
20
ppm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7.515
7.502
7.496
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7.421
7.402
7.291
7.273
7.260
7.211
7.195
7.192
7.176
7.173
7.126
7.108
7.090
7.089
6.848
6.828
2.932
2.916
2.912
2.906
2.896
2.888
2.869
2.079
2.064
2.050
2.001
1.987
1.974
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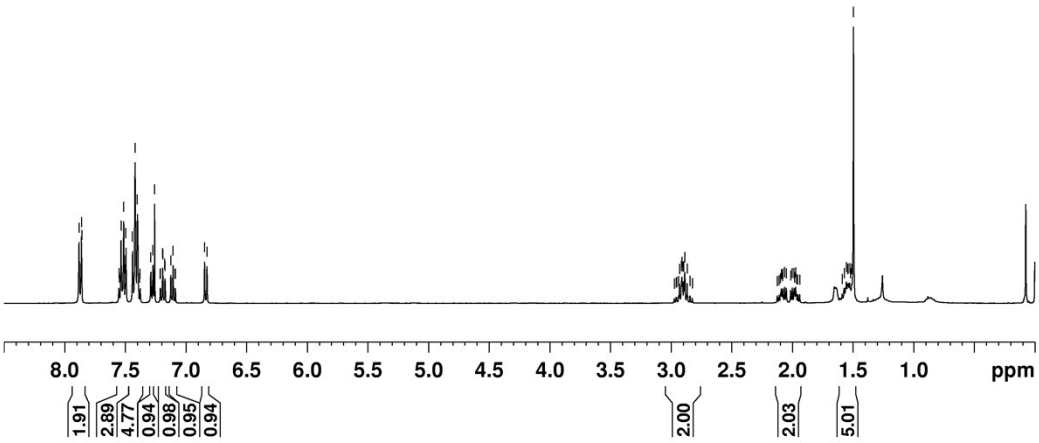
1.544
1.540
1.528
1.521
1.511
1.497

O
2
O
N
Ph

4p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7.0
6.5
6.0
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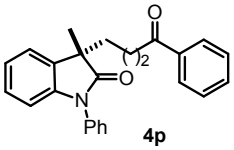
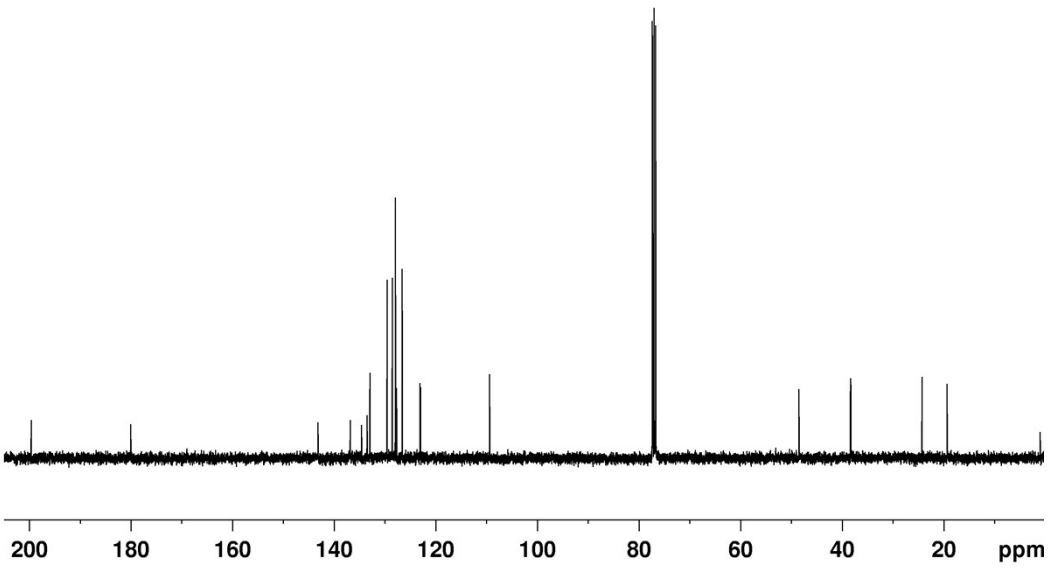
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.91
2.89
4.77
0.94
0.98
0.95
0.94
2.00
2.03
5.01

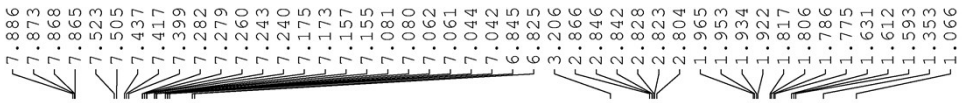

199.522
180.032
143.161
136.808
134.576
133.484
132.947
129.535
128.534
127.914
127.686
126.581
123.072
122.937
109.367
77.319
77.001
76.683
48.475
38.321
38.241
24.234
19.312

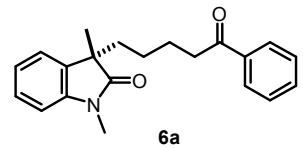

O
2
O
N
Ph

4p

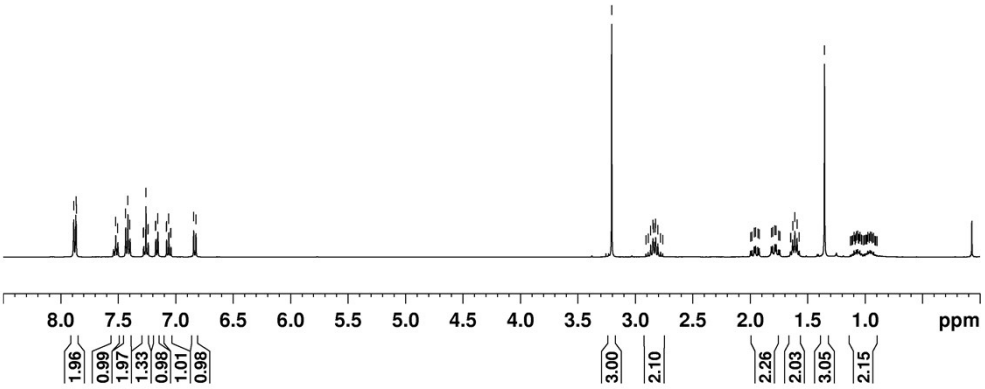

200
180
160
140
120
100
80
60
40
20
ppm

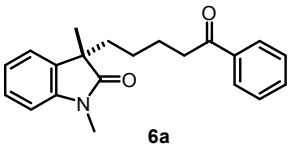
^{1}H NMR and ^{13}C NMR Spectra of Products 6



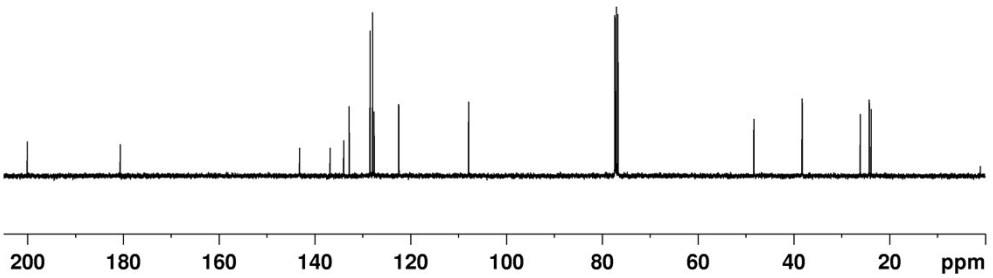


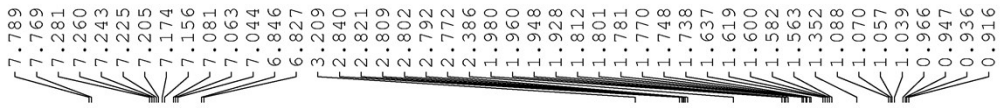
6a





6a

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7.081
7.063
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6.846
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2.840
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2.809
2.802
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2.772
2.386
1.980
1.960
1.948
1.928
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1.738
1.637
1.619
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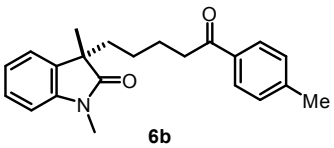
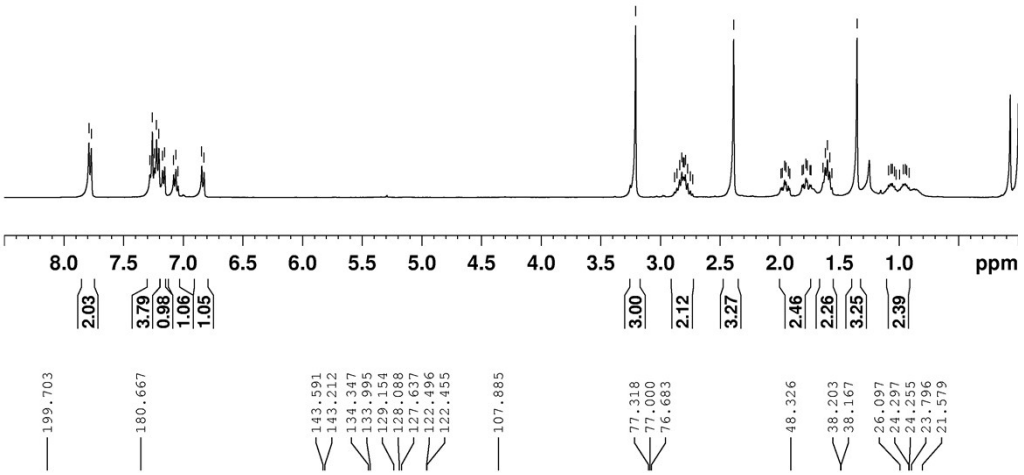
0.936
0.916


O
O
N
Me

6b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2.0
1.5
1.0
ppm
2.03
3.79
0.98
1.06
1.05
3.00
2.12
3.27
2.46
2.26
3.25
2.39

199.703
180.667
143.591
143.212
134.347
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128.088
127.637
122.496
122.455
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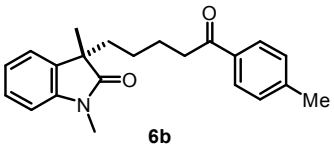
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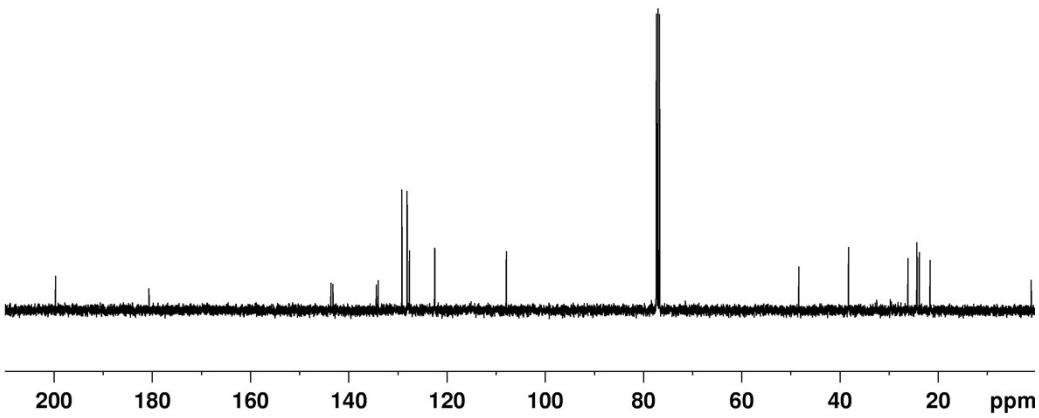
6b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100
80
60
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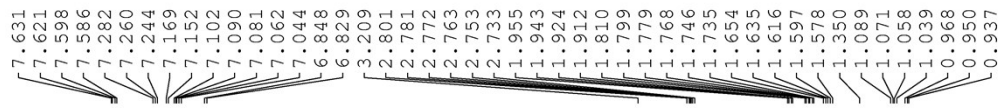
20
ppm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3.209
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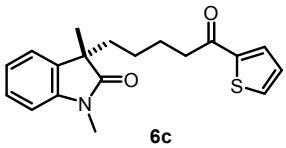

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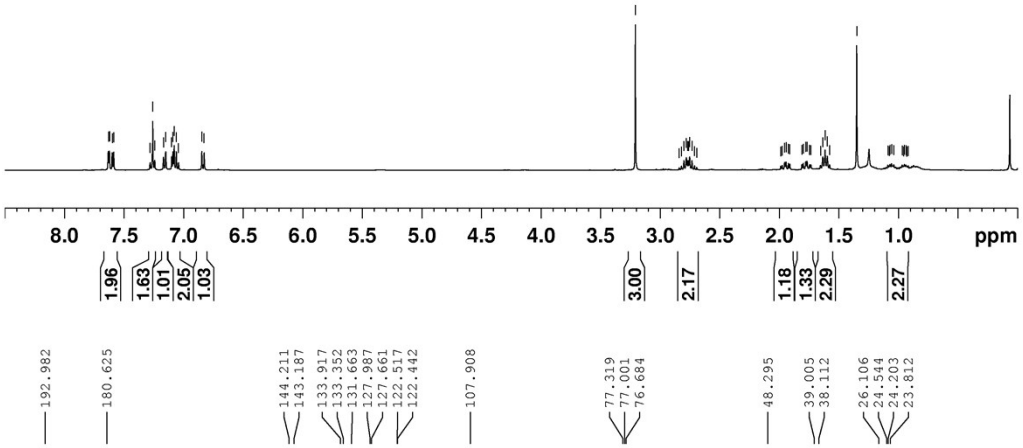
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4.5
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1.5
1.0
ppm
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1.01
2.05
1.03
3.00
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1.33
2.29
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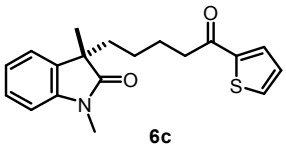

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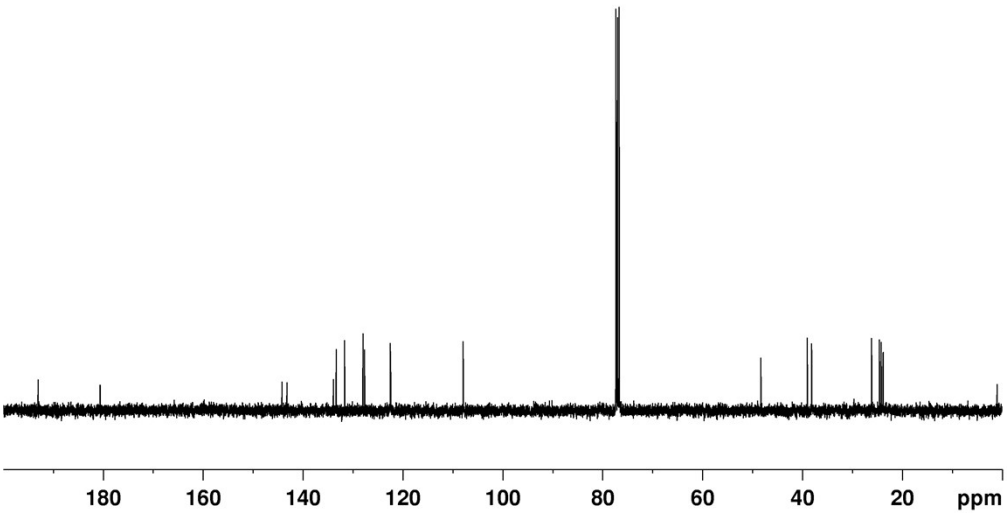
180
160
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20
ppm