SUPPORTING INFORMATION

Rh-catalyzed nitrene alkyne metathesis/formal C–N bond insertion cascade: synthesis of 3-iminoindolines

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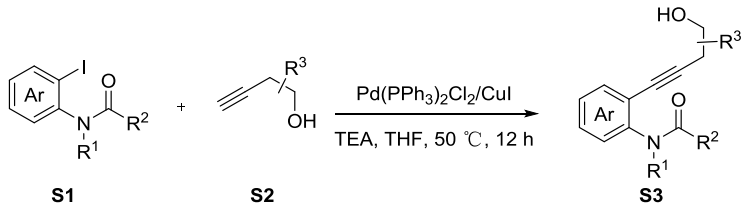
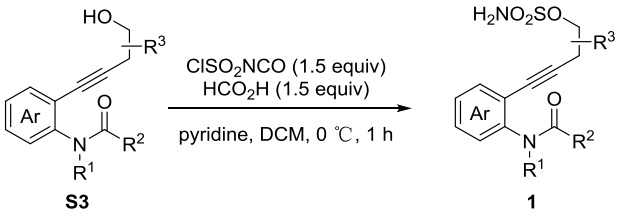
General Information

All reactions were carried out in oven-dried glassware. Solvents were purified by following the standard methods. Flash column chromatography was performed using silica gel (300-400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 200-300 mesh silica gel impregnated with a fluorescent indicator (254 nm). ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$, DMSO-d_6 and C_6D_6 on 400 MHz and 500 MHz spectrometer; chemical shifts were reported in ppm with the solvent signal as reference, and coupling constants (*J*) were given in Hertz. The peak information was described as: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, comp = composite. Enantioselectivity was determined on HPLC using Daicel Chiralpak AD-H column. High-resolution mass spectra (HRMS) were recorded on a commercial apparatus (ESI Source) and (CI Source).

General Procedure for the Synthesis of Sulfamate Esters 1.[1]

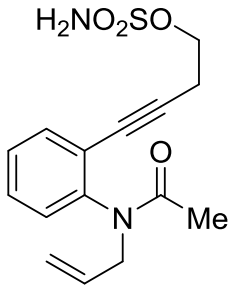


Synthesis of S3: To a 50-mL oven-dried flask containing a magnetic stirring bar, **S1** (5.0 mmol), Et_3N (10 mL), THF (10 mL), CuI (9.5 mg, 1.0 mol%), $Pd(PPh_3)_2Cl_2$ (52.7 mg, 1.5 mol%), and **S2** (5.5 mmol, 1.1 equiv.) were added in sequence under argon atmosphere. The reaction mixture was stirred at 50 °C for 12 h. Then the reaction mixture was filtered through a short pad of Celite and the solid was washed with ethyl acetate. The combined organic layer was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to give **S3** in >75% yields.

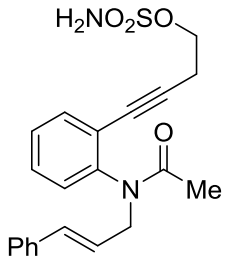


Synthesis of 1: To a 50-mL oven-dried round bottom flask equipped with stirring bar, and $ClSO_2NCO$ (0.65 mL, 7.5 mmol, 1.5 equiv.), was added formic acid (0.29 mL, 7.5 mmol, 1.5 equiv.) drop wise under an argon atmosphere at 0 °C. The above resulting white solid was dissolved in CH_2Cl_2 (5.0 mL). The solution was warmed to 25 °C and allowed to stir overnight. Then a solution of **S3** (5.0 mmol) and pyridine (0.6 mL, 7.5 mmol, 1.5 equiv.) in CH_2Cl_2 (4.0 mL) was added drop wise at 0 °C. The reaction mixture was warmed to 25 °C and stirred until complete consumption of starting material (15 min~1 h, monitored by thin layer chromatography). Then the reaction was quenched by addition of H_2O (10 mL) and extracted with EtOAc (3 x 20 mL). The combined organic extract was washed with brine (50 mL), dried over anhydrous

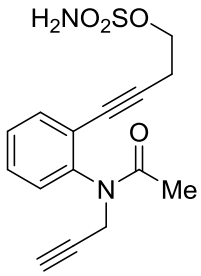
Na_2SO_4, and concentrated under reduced pressure after filtration. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 3:1 to 1:1) to afford pure sulfamate ester **1** in high yields.



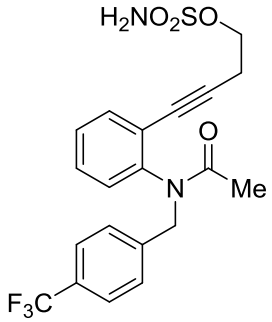
4-[2-(*N*-Allylacetamido)phenyl]but-3-yn-1-yl sulfamate (1a). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.48 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.37-7.29 (comp, 2H), 7.16 (dd, *J* = 7.7, 1.3 Hz, 1H), 6.09 (s, 2H), 5.91-5.81 (m, 1H), 5.10-5.04 (comp, 2H), 4.58 (dd, *J* = 14.8, 6.0 Hz, 1H), 4.30-4.25 (m, 2H), 4.01 (dd, *J* = 14.8, 7.0 Hz, 1H), 2.87 (t, *J* = 5.7 Hz, 2H), 1.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 172.0, 144.0, 133.1, 132.9, 129.4, 128.8, 128.4, 123.1, 118.4, 91.1, 78.5, 67.3, 51.9, 22.4, 20.5; HRMS (TOF MS ESI$^+$) calculated for $C_{15}H_{19}N_2O_4S$ $[M+H]^+$: 323.1060, found 323.1060.



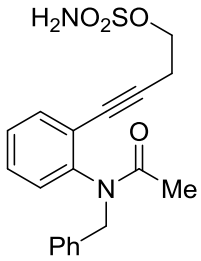
4-[2-(*N*-Cinnamylacetamido)phenyl]but-3-yn-1-yl sulfamate (1b). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.48 (d, *J* = 7.3 Hz, 1H), 7.35-7.26 (comp, 6H), 7.19 (dd, *J* = 18.8, 7.3 Hz, 2H), 6.36 (d, *J* = 15.9 Hz, 1H), 6.30-6.24 (comp, 3H), 4.62 (dd, *J* = 14.5, 6.0 Hz, 1H), 4.30-4.23 (comp, 3H), 2.86-2.75 (m, 2H), 1.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 172.0, 143.8, 136.8, 133.6, 133.1, 129.5, 128.7, 128.6, 128.4, 127.7, 125.5, 124.1, 123.2, 91.1, 78.5, 67.3, 51.4, 22.3, 20.3; HRMS (TOF MS ESI$^+$) calculated for $C_{21}H_{23}N_2O_4S$ $[M+H]^+$: 399.1373, found 399.1368.



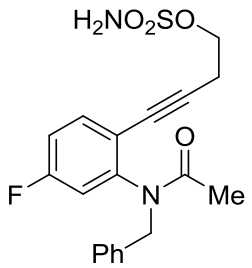
4-{2-[*N*-(Prop-2-yn-1-yl)acetamido]phenyl}but-3-yn-1-yl sulfamate (1c). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.50-7.48 (m, 1H), 7.41-7.32 (comp, 3H), 6.06 (s, 2H), 4.96 (dd, *J* = 17.4, 2.5 Hz, 1H), 4.26 (t, *J* = 5.8 Hz, 2H), 4.01 (dd, *J* = 17.4, 2.5 Hz, 1H), 2.85 (dd, *J* = 6.2, 5.4 Hz, 2H), 2.20 (t, *J* = 2.5 Hz, 1H), 1.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 171.8, 143.1, 133.1, 129.5, 129.1, 128.9, 122.8, 91.3, 78.8, 77.9, 72.7, 67.4, 37.7, 22.2, 20.4; HRMS (TOF MS ESI$^+$) calculated for $C_{15}H_{17}N_2O_4S$ $[M+H]^+$: 321.0904, found 321.0893.



4-{2-[*N*-(4-(Trifluoromethyl)benzyl)acetamido]phenyl}but-3-yn-1-yl sulfamate (1d). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.52 (d, *J* = 7.6 Hz, 2H), 7.47 (d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.7 Hz, 2H), 7.31 – 7.27 (m, 2H), 6.91 (d, *J* = 7.4 Hz, 1H), 6.14 (s, 2H), 5.30 (d, *J* = 13.3 Hz, 1H), 4.53 (d, *J* = 14.6 Hz, 1H), 4.25 – 4.24 (m, 2H), 2.84 – 2.75 (m, 2H), 1.93 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 172.4, 143.7, 140.9, 133.4, 129.8 (dd, *J* = 64.6, 32.3 Hz), 129.6, 129.5, 128.6 (d, *J* = 9.9 Hz), 125.4 (q, *J* = 3.7 Hz), 124.2 (dd, *J* = 1615.4, 799.7 Hz), 123.2, 123.0, 91.2, 78.1, 67.3, 52.2, 22.4, 20.4; ^{19}F NMR (471 MHz, $CDCl_3$) (δ, ppm) -62.45; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{20}F_3N_2O_4S$ $[M+H]^+$: 441.1090, found 441.1096.

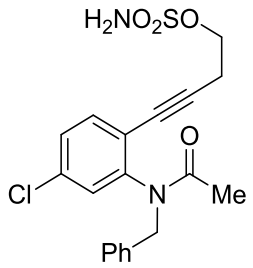


4-[2-(*N*-Benzylacetamido)phenyl]but-3-yn-1-yl sulfamate (1e). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.46 (dd, *J* = 7.5, 1.7 Hz, 1H), 7.30-7.18 (comp, 7H), 6.87 (dd, *J* = 7.7, 1.3 Hz, 1H), 6.19 (s, 2H), 5.31 (d, *J* = 14.3 Hz, 1H), 4.43 (d, *J* = 14.3 Hz, 1H), 4.29-4.20 (m, 2H), 2.87-2.74 (m, 2H), 1.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 172.2, 143.8, 136.9, 133.1, 129.3, 129.2, 128.8, 128.4, 128.4, 127.6, 123.0, 91.0, 78.2, 67.3, 52.6, 22.4, 20.4; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{21}N_2O_4S$ $[M+H]^+$: 373.1217, found 373.1214.

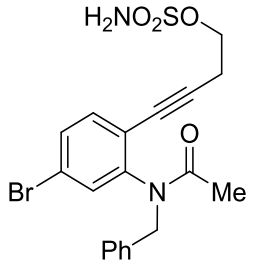


4-[2-(*N*-Benzylacetamido)-4-fluorophenyl]but-3-yn-1-yl sulfamate (1f).

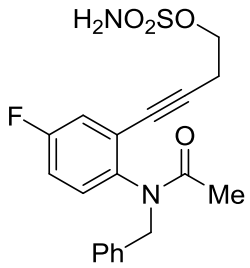
Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.46 – 7.43 (m, 1H), 7.29 – 7.21 (comp, 5H), 7.01 (t, *J* = 8.3 Hz, 1H), 6.65 (d, *J* = 8.8 Hz, 1H), 6.45 (s, 2H), 5.29 (d, *J* = 14.5 Hz, 1H), 4.51 (d, *J* = 14.5 Hz, 1H), 4.32 – 4.25 (m, 2H), 2.86 – 2.77 (m, 2H), 1.97 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.6, 161.7 (d, *J* = 252.3 Hz), 145.0 (d, *J* = 9.9 Hz), 136.3, 134.2 (d, *J* = 9.2 Hz), 129.0, 128.2, 127.5, 119.1 (d, *J* = 3.7 Hz), 116.1 (d, *J* = 22.6 Hz), 115.6 (d, *J* = 21.7 Hz), 90.6, 77.0, 67.2, 52.1, 22.1, 20.0; ^{19}F NMR (471 MHz, $CDCl_3$) (δ, ppm) -108.5; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{20}FN_2O_4S$ $[M+H]^+$: 391.1122, found 391.1121.



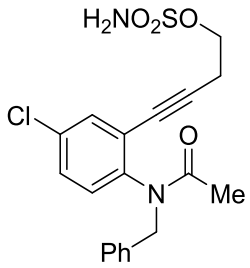
4-[2-(*N*-Benzylacetamido)-4-chlorophenyl]but-3-yn-1-yl sulfamate (1g). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.39 (d, *J* = 8.4 Hz, 1H), 7.29 – 7.21 (comp, 6H), 6.94 (s, 1H), 6.49 (s, 2H), 5.22 (d, *J* = 14.4 Hz, 1H), 4.56 (d, *J* = 14.4 Hz, 1H), 4.32 – 4.26 (m, 2H), 2.86 – 2.76 (m, 2H), 1.96 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.5, 144.3, 136.1, 134.0, 133.7, 128.9, 128.7, 128.4, 128.1, 127.5, 121.5, 92.0, 77.0, 67.1, 52.1, 22.0, 20.0; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{20}ClN_2O_4S$ $[M+H]^+$: 407.0827, found 407.0826.



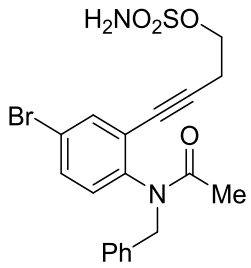
4-[2-(*N*-Benzylacetamido)-4-bromophenyl]but-3-yn-1-yl sulfamate (1h). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.40 (d, *J* = 8.1 Hz, 1H), 7.32 – 7.23 (comp, 4H), 7.21 (d, *J* = 6.5 Hz, 2H), 7.09 (s, 1H), 6.45 (s, 2H), 5.19 (d, *J* = 14.4 Hz, 1H), 4.55 (d, *J* = 14.4 Hz, 1H), 4.30 – 4.24 (m, 2H), 2.84 – 2.73 (m, 2H), 1.95 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.5, 144.4, 136.1, 133.8, 131.6, 131.3, 129.0, 128.2, 127.5, 122.0, 121.9, 92.2, 77.1, 67.1, 52.2, 22.1, 20.0; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{20}BrN_2O_4S$ $[M+H]^+$: 451.0322, found 451.0328.



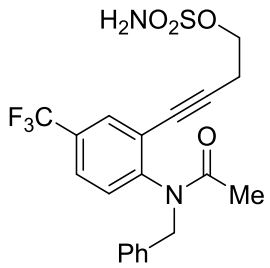
4-[2-(*N*-Benzylacetamido)-5-fluorophenyl]but-3-yn-1-yl sulfamate (1i). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.30 – 7.22 (m, 3H), 7.16 (ddd, *J* = 11.5, 8.1, 2.9 Hz, 3H), 7.05 – 6.84 (m, 1H), 6.80 (dd, *J* = 8.8, 5.3 Hz, 1H), 6.23 (s, 2H), 5.32 (d, *J* = 14.3 Hz, 1H), 4.37 (d, *J* = 14.3 Hz, 1H), 4.32 – 4.17 (m, 2H), 2.92 – 2.65 (m, 2H), 1.92 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 172.2, 161.5 (d, *J* = 249.3 Hz), 139.9 (d, *J* = 3.4 Hz), 136.6, 130.5 (d, *J* = 9.2 Hz), 129.4, 128.4, 127.7, 124.8 (d, *J* = 9.9 Hz), 119.7 (d, *J* = 24.1 Hz), 116.5 (d, *J* = 22.4 Hz), 92.2, 77.3, 67.1, 52.4, 22.4, 20.3; ^{19}F NMR (376 MHz, $CDCl_3$) (δ, ppm) -112.29; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{20}FN_2O_4S$ $[M+H]^+$: 391.1122, found 391.1121.



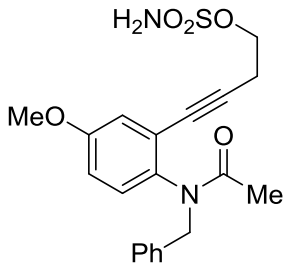
4-[2-(*N*-Benzylacetamido)-5-chlorophenyl]but-3-yn-1-yl sulfamate (1j). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.42 (s, 1H), 7.31 – 7.17 (comp, 6H), 6.81 (d, *J* = 8.5 Hz, 1H), 6.46 (s, 2H), 5.29 (d, *J* = 14.4 Hz, 1H), 4.44 (d, *J* = 14.4 Hz, 1H), 4.31 – 4.24 (m, 2H), 2.87 – 2.76 (m, 2H), 1.94 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.7, 142.0, 136.3, 133.7, 132.6, 129.9, 129.2, 129.1, 128.2, 127.5, 124.4, 92.4, 76.8, 67.1, 52.1, 22.1, 20.1; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{20}ClN_2O_4S$ $[M+H]^+$: 407.0827, found 407.0828.



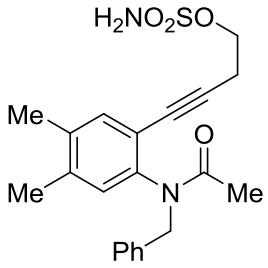
4-[2-(*N*-Benzylacetamido)-5-bromophenyl]but-3-yn-1-yl sulfamate (1k). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.59 (s, 1H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.25 – 7.18 (comp, 5H), 6.71 (d, *J* = 8.3 Hz, 1H), 6.14 (s, 2H), 5.28 (d, *J* = 14.3 Hz, 1H), 4.38 (d, *J* = 14.3 Hz, 1H), 4.24 – 4.23 (m, 2H), 2.85 – 2.76 (m, 2H), 1.92 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.9, 142.8, 136.6, 135.8, 132.4, 130.3, 129.4, 128.5, 127.8, 125.0, 121.9, 92.6, 77.0, 67.1, 52.4, 22.41, 20.4; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{20}BrN_2O_4S$ $[M+H]^+$: 451.0322, found 451.0327.



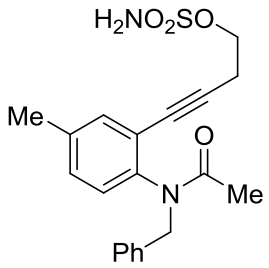
4-[2-(*N*-Benzylacetamido)-5-(trifluoromethyl)phenyl]but-3-yn-1-yl sulfamate (1l). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.74 (s, 1H), 7.49 (d, *J* = 7.9 Hz, 1H), 7.27 – 7.20 (comp, 5H), 7.03 (d, *J* = 8.3 Hz, 1H), 6.33 (s, 2H), 5.32 (d, *J* = 14.4 Hz, 1H), 4.50 (d, *J* = 14.4 Hz, 1H), 4.32 – 4.26 (m, 2H), 2.89 – 2.79 (m, 2H), 1.95 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.6, 146.6, 136.3, 130.5 (d, *J* = 33.3 Hz), 130.1 (d, *J* = 3.1 Hz), 129.5 (d, *J* = 3.3 Hz), 129.2, 128.4, 127.7, 125.8 (d, *J* = 3.5 Hz), 124.0, 123.2 (q, *J* = 272.8 Hz), 93.0, 76.9, 67.1, 52.3, 22.3, 20.2; ^{19}F NMR (376 MHz, $CDCl_3$) (δ, ppm) -62.91; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{19}F_3N_2O_4SNa$ $[M+Na]^+$: 463.0910, found 463.0902.



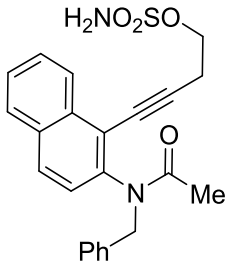
4-[2-(*N*-Benzylacetamido)-5-methoxyphenyl]but-3-yn-1-yl sulfamate (1m). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.27 – 7.18 (comp, 5H), 6.95 (s, 1H), 6.74 (s, 2H), 6.20 (s, 2H), 5.28 (d, *J* = 14.2 Hz, 1H), 4.37 (d, *J* = 14.1 Hz, 1H), 4.29 – 4.22 (m, 2H), 3.78 (s, 3H), 2.84 – 2.75 (m, 2H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 172.7, 158.9, 137.0, 136.8, 129.7, 129.4, 128.3, 127.5, 123.7, 117.4, 115.3, 90.7, 78.2, 67.3, 55.6, 52.6, 22.3, 20.3; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{23}N_2O_5S$ $[M+H]^+$: 403.1322, found 403.1320.



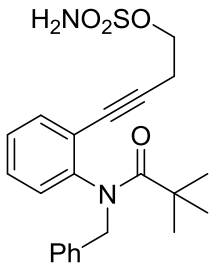
4-[2-(*N*-Benzylacetamido)-4,5-dimethylphenyl]but-3-yn-1-yl sulfamate (1n). Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.29 – 7.21 (comp, 6H), 6.67 (s, 1H), 6.30 (s, 2H), 5.20 (d, *J* = 14.3 Hz, 1H), 4.50 (d, *J* = 14.3 Hz, 1H), 4.28 – 4.18 (m, 2H), 2.84 – 2.70 (m, 2H), 2.21 (s, 3H), 2.15 (s, 3H), 1.94 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 172.3, 141.5, 138.3, 137.0, 136.9, 133.7, 129.4, 129.2, 128.2, 127.4, 119.8, 89.6, 78.3, 67.4, 52.6, 22.2, 20.2, 19.6, 19.2; HRMS (TOF MS ESI$^+$) calculated for $C_{21}H_{25}N_2O_4S$ $[M+H]^+$: 401.1530, found 401.1533.



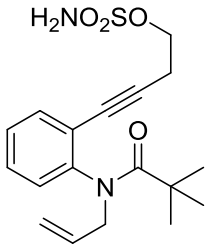
4-[2-(*N*-Benzylacetamido)-5-methylphenyl]but-3-yn-1-yl sulfamate (1o). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.29 – 7.20 (comp, 6H), 7.02 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 8.1 Hz, 1H), 6.39 (s, 2H), 5.30 (d, *J* = 14.4 Hz, 1H), 4.42 (d, *J* = 14.3 Hz, 1H), 4.29 – 4.22 (m, 2H), 2.85 – 2.74 (m, 2H), 2.30 (s, 3H), 1.93 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 172.2, 141.1, 138.2, 136.9, 133.4, 129.9, 129.2, 128.3, 128.2, 127.4, 122.4, 90.4, 78.2, 67.3, 52.4, 22.2, 20.8, 20.2; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{23}N_2O_4S$ $[M+H]^+$: 387.1373, found 387.1370.



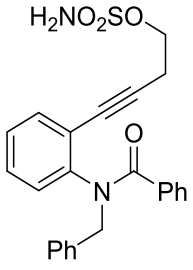
4-[2-(*N*-Benzylacetamido)naphthalen-1-yl]but-3-yn-1-yl sulfamate (1p). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 8.31 (d, *J* = 8.3 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.72 (d, *J* = 8.6 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.23 (s, 5H), 6.98 (d, *J* = 8.6 Hz, 1H), 6.32 (s, 2H), 5.36 (d, *J* = 14.3 Hz, 1H), 4.61 (d, *J* = 14.3 Hz, 1H), 4.34 – 4.31 (m, 2H), 3.03 – 2.92 (m, 2H), 1.96 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 172.2, 142.3, 136.9, 133.7, 132.3, 129.5, 129.4, 128.3, 128.2, 127.6, 127.6, 127.3, 125.7, 125.9, 120.3, 96.3, 76.5, 67.4, 52.6, 22.5, 20.7; HRMS (TOF MS ESI$^+$) calculated for $C_{23}H_{23}N_2O_4S$ $[M+H]^+$: 423.1373, found 423.1370.



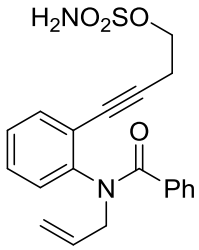
4-[2-(*N*-Benzylpivalamido)phenyl]but-3-yn-1-yl sulfamate (1q) Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.48 – 7.46 (m, 1H), 7.29 – 7.20 (comp, 4H), 7.17 – 7.12 (comp, 3H), 6.80 (dd, *J* = 7.9, 0.9 Hz, 1H), 6.02 (s, 2H), 5.84 (d, *J* = 14.5 Hz, 1H), 4.34 – 4.27 (m, 2H), 3.93 (d, *J* = 14.5 Hz, 1H), 2.90 (t, *J* = 5.7 Hz, 2H), 1.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 178.7, 144.6, 137.6, 132.9, 130.8, 128.9, 128.4, 128.3, 128.2, 127.4, 124.1, 91.7, 79.0, 67.5, 55.2, 41.3, 29.1, 20.5; HRMS (TOF MS ESI$^+$) calculated for $C_{22}H_{27}N_2O_4S$ $[M+H]^+$: 415.1686, found 415.1690.



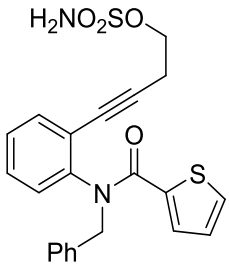
4-[2-(*N*-Allylpivalamido)phenyl]but-3-yn-1-yl sulfamate (1r). Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.43 (m, 1H), 7.32 (ddd, *J* = 5.0, 4.2, 2.9 Hz, 2H), 7.24 – 7.14 (m, 1H), 6.01 (s, 2H), 5.93 – 5.78 (m, 1H), 5.09 (dd, *J* = 10.2, 1.2 Hz, 1H), 5.07 – 4.89 (m, 2H), 4.37 – 4.23 (m, 2H), 3.53 (dd, *J* = 14.5, 7.1 Hz, 1H), 2.89 (dd, *J* = 6.5, 5.0 Hz, 2H), 1.04 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.5, 144.8, 133.0, 132.9, 130.6, 128.39, 128.36, 124.1, 117.9, 91.6, 79.0, 67.4, 55.0, 41.2, 29.1, 20.5; HRMS (TOF MS ESI$^+$) calculated for $C_{18}H_{25}N_2O_4S$ $[M+H]^+$: 365.1530, found 365.1530.



4-(2-(N-benzylbenzamido)phenyl)but-3-yn-1-yl sulfamate (1s). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.38 – 7.36 (m, 2H), 7.32 – 7.25 (comp, 6H), 7.22 – 7.19 (m, 1H), 7.16 – 7.13 (m, 2H), 7.11 – 7.05 (m, 2H), 6.84 – 6.82 (m, 1H), 5.84 (s, 2H), 5.65 (d, *J* = 14.4 Hz, 1H), 4.57 (d, *J* = 14.4 Hz, 1H), 4.33 (t, *J* = 5.8 Hz, 2H), 2.90 – 2.86 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.7, 144.3, 137.0, 136.0, 133.0, 129.8, 129.6, 129.3, 128.8, 128.5, 128.1, 127.8, 127.7, 123.0, 91.3, 79.0, 67.5, 53.3, 20.6; HRMS (TOF MS ESI$^+$) calculated for $C_{24}H_{23}N_2O_4S$ $[M+H]^+$: 435.1373, found 435.1373.

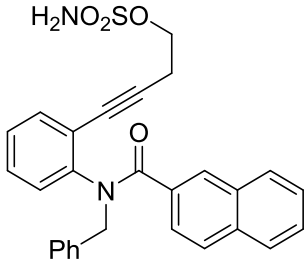


4-[2-(N-allylbenzamido)phenyl]but-3-yn-1-yl sulfamate (1t). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.37 – 7.35 (m, 2H), 7.29 – 7.27 (m, 1H), 7.24 – 7.20 (m, 2H), 7.17 – 7.12 (comp, 4H), 6.05 – 5.92 (comp, 3H), 5.19 – 5.15 (m, 2H), 4.83 (dd, *J* = 14.8, 5.9 Hz, 1H), 4.35 (t, *J* = 5.8 Hz, 2H), 4.24 (dd, *J* = 14.8, 6.9 Hz, 1H), 2.91 – 2.88 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 171.4, 144.3, 135.9, 133.0, 132.7, 129.9, 129.4, 128.9, 128.2, 127.8, 127.7, 123.1, 118.6, 91.3, 79.1, 67.5, 52.9, 20.6; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{21}N_2O_4S$ $[M+H]^+$: 385.1217, found 385.1218.



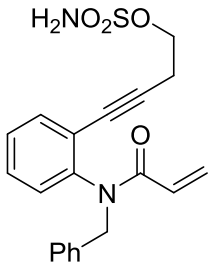
4-[2-(*N*-Benzylthiophene-2-carboxamido)phenyl]but-3-yn-1-yl sulfamate (1u). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.47 – 7.45 (m, 1H), 7.35 – 7.23 (comp,

8H), 6.99 – 6.97 (m, 1H), 6.87 (d, *J* = 3.2 Hz, 1H), 6.80 – 6.78 (m, 1H), 6.04 (s, 2H), 5.65 (d, *J* = 14.4 Hz, 1H), 4.52 (d, *J* = 14.5 Hz, 1H), 4.27 – 4.17 (m, 2H), 2.77 – 2.73 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 163.2, 143.3, 137.5, 136.6, 133.3, 132.6, 131.2, 129.8, 129.3, 129.1, 128.8, 128.4, 127.6, 125.9, 123.7, 91.2, 78.1, 67.4, 54.0, 20.3; HRMS (TOF MS ESI$^+$) calculated for $C_{22}H_{21}N_2O_4S_2$ $[M+H]^+$: 441.0937, found 441.0939.



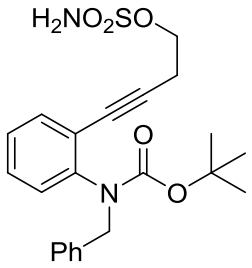
4-[2-(*N*-Benzyl-2-naphthamido)phenyl]but-3-yn-1-yl sulfamate (1v). White oil.

^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 8.01 (s, 1H), 7.76 (d, *J* = 6.3 Hz, 1H), 7.67 (d, *J* = 6.3 Hz, 1H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.40 (d, *J* = 6.7 Hz, 4H), 7.34 – 7.28 (comp, 3H), 7.21 (d, *J* = 7.3 Hz, 1H), 7.05 – 6.95 (comp, 3H), 6.33 (s, 2H), 5.67 (d, *J* = 14.4 Hz, 1H), 4.77 (d, *J* = 14.4 Hz, 1H), 4.35 – 4.32 (m, 2H), 2.85 – 2.75 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 171.3, 143.9, 136.7, 133.4, 133.0, 132.9, 132.0, 129.3, 129.1, 128.8, 128.5, 128.2, 127.42, 127.36, 127.1, 127.0, 125.2, 124.9, 122.6, 91.2, 78.7, 67.3, 53.2, 20.2; HRMS (TOF MS ESI$^+$) calculated for $C_{28}H_{24}N_2O_4S$ $[M+H]^+$: 485.1530, found 485.1529.

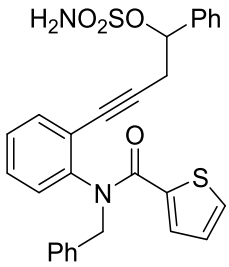


4-[2-(*N*-Benzylacrylamido)phenyl]but-3-yn-1-yl sulfamate (1w). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.47 – 7.45 (m, 1H), 7.30 – 7.20 (comp, 7H), 6.87 – 6.85 (m,

1H), 6.41 (dd, *J* = 16.8, 1.7 Hz, 1H), 6.10 (s, 2H), 5.94 (dd, *J* = 16.8, 10.4 Hz, 1H), 5.57 (dd, *J* = 10.4, 1.7 Hz, 1H), 5.45 (d, *J* = 14.4 Hz, 1H), 4.46 (d, *J* = 14.4 Hz, 1H), 4.20 (dd, *J* = 8.9, 3.4 Hz, 2H), 2.78 (t, *J* = 5.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 166.6, 142.7, 136.7, 133.0, 129.3, 129.2, 129.11, 129.08 128.44, 128.37, 127.9, 127.6, 123.2, 91.3, 78.2, 67.4, 52.7, 20.3; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{21}N_2O_4S$ $[M+H]^+$: 385.1217, found 385.1218.

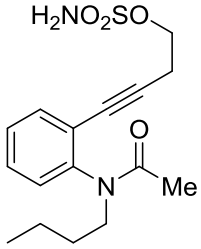


4-{2-[Benzyl(*tert*-butoxycarbonyl)amino]phenyl}but-3-yn-1-yl sulfamate (1x). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.41 (d, *J* = 6.7 Hz, 1H), 7.26 – 7.24 (comp, 5H), 7.17 – 7.13 (m, 2H), 6.80 (d, *J* = 7.0 Hz, 1H), 5.78 (s, 2H), 5.28 (d, *J* = 14.3 Hz, 1H), 4.29 (s, 3H), 2.93 – 2.80 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 155.9, 143.9, 138.0, 132.5, 129.1, 128.7, 128.5, 128.4, 127.4, 127.2, 122.4, 89.7, 80.8, 79.3, 67.7, 53.0, 28.4, 20.5; HRMS (TOF MS ESI$^+$) calculated for $C_{22}H_{26}N_2O_5SNa$ $[M+Na]^+$: 453.1455, found 453.1450.



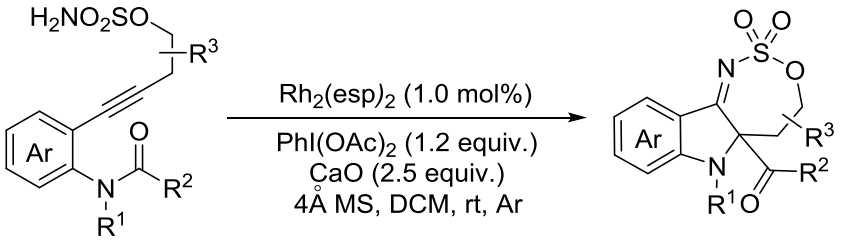
4-[2-(*N*-Benzylthiophene-2-carboxamido)phenyl]-1-phenylbut-3-yn-1-yl sulfamate (1y). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.44 (t, *J* = 7.9 Hz, 1H), 7.40 – 7.38 (m, 1H), 7.36 – 7.25 (comp, 10H), 7.22 – 7.18 (m, 2H), 6.90 – 6.79 (comp, 3H), 5.82 – 5.76 (m, 3H), 5.64 – 5.57 (m, 1H), 4.40 – 4.32 (m, 1H), 3.25 – 2.92 (m, 2H); ^{13}C NMR

(100 MHz, $CDCl_3$) (δ, ppm) 162.8, 143.1, 137.7, 137.5, 136.7, 133.6, 132.7, 131.4, 130.2, 129.3, 129.0, 128.8, 128.5, 128.3, 127.5, 127.0, 125.6, 123.6, 90.4, 79.9, 79.1, 53.8, 28.3; HRMS (TOF MS ESI$^+$) calculated for $C_{28}H_{25}N_2O_4S_2$ $[M+H]^+$: 517.1250, found 517.1255.

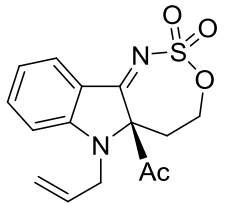


4-[2-(*N*-Butylacetamido)phenyl]but-3-yn-1-yl sulfamate (1z). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.49 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.39 – 7.36 (m, 1H), 7.33 – 7.29 (m, 1H), 7.17 (dd, *J* = 7.8, 0.9 Hz, 1H), 6.42 (s, 2H), 4.28 (t, *J* = 5.9 Hz, 2H), 3.89-3.83 (m, 1H), 3.53-3.47 (m, 1H), 2.86 (t, *J* = 5.9 Hz, 2H), 1.85 (s, 3H), 1.52-1.45 (m, 2H), 1.34-1.27 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 171.8, 144.0, 133.0, 129.3, 128.4, 128.1, 122.9, 90.9, 78.2, 67.2, 60.4, 48.7, 29.6, 22.2, 20.0, 13.7; HRMS (TOF MS ESI$^+$) calculated for $C_{16}H_{23}N_2O_4S$ $[M+H]^+$: 339.1373, found 339.1369.

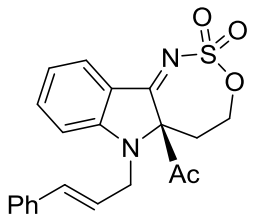
General Procedure for the Cascade Reaction



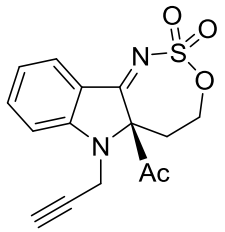
To a 10-mL oven-dried vial containing a magnetic stirring bar, $Rh_2(esp)_2$ (1.5 mg, 1.0 mol%), $PhI(OAc)_2$ (77.3 mg, 1.2 equiv.), CaO (28.0 mg, 2.5 equiv.), and 4Å MS (50.0 mg) in DCM (1.0 mL), was added as a solution of sulfamate ester **1** (0.2 mmol) in the DCM (1.0 mL) *via* a syringe at room temperature under argon atmosphere. After addition, the reaction mixture was stirred for additional 12 h under these conditions. Until consumption of the material (monitored by TLC). Then the reaction mixture was purified by column chromatography on silica gel without any additional treatment (Hexanes : EtOAc = 4:1 to 2:1) to give the pure products **2** in good to high yields.



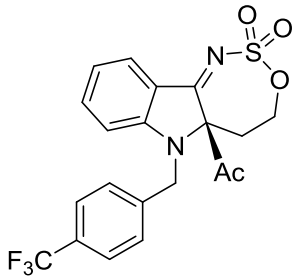
1-[6-Allyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2a). Yellow oil. 51.3 mg, 80% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.70 – 7.67 (m, 2H), 7.00 – 6.93 (m, 2H), 5.76 – 5.70 (m, 1H), 5.24 – 5.17 (m, 2H), 4.75 (t, *J* = 11.9 Hz, 1H), 4.60 (d, *J* = 12.4 Hz, 1H), 4.06 – 4.02 (m, 1H), 3.81 – 3.77 (m, 1H), 2.61 (d, *J* = 14.4 Hz, 1H), 2.06 – 2.00 (m, 1H), 1.90 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.9, 180.2, 158.3, 140.1, 132.3, 125.2, 120.1, 119.8, 117.9, 111.0, 80.1, 69.8, 43.5, 25.8, 24.7; HRMS (TOF MS ESI$^+$) calculated for $C_{15}H_{16}N_2O_4SNa$ $[M+Na]^+$: 343.0723, found 343.0723.



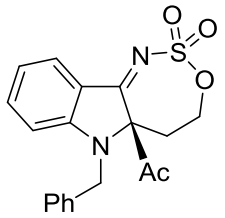
1-[6-Cinnamyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2b). Yellow oil. 60.3 mg, 76% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.73 – 7.67 (m, 2H), 7.41 (d, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.3 Hz, 2H), 7.23 (t, *J* = 7.2 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.20 – 6.14 (m, 1H), 4.78 (t, *J* = 11.7 Hz, 1H), 4.62 (d, *J* = 12.5 Hz, 1H), 4.22 (dd, *J* = 17.3, 6.2 Hz, 1H), 3.97 (dd, *J* = 17.3, 5.2 Hz, 1H), 2.70 (d, *J* = 14.4 Hz, 1H), 2.17 – 2.11 (m, 1H), 1.93 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 194.0, 180.2, 158.3, 140.2, 136.0, 132.4, 128.6, 127.8, 125.4, 125.2, 123.8, 120.1, 119.9, 111.0, 80.2, 69.9, 43.2, 25.8, 24.7; HRMS (TOF MS ESI^{+}) calculated for $C_{21}H_{20}N_2O_4SNa$ $[M+Na]^{+}$: 419.1036, found 419.1030.



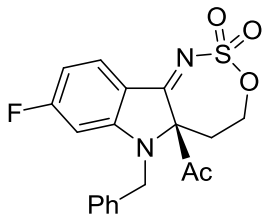
1-[2,2-Dioxido-6-(prop-2-yn-1-yl)-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2c). Yellow oil. 43.3 mg, 68% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.78 – 7.73 (m, 2H), 7.16 (d, *J* = 8.4 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 4.74 (t, *J* = 11.3 Hz, 1H), 4.63 (d, *J* = 12.3 Hz, 1H), 4.28 – 4.20 (m, 2H), 3.27 (s, 1H), 2.77 (d, *J* = 14.6 Hz, 1H), 2.01 – 1.94 (m, 1H), 1.92 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.8, 180.1, 157.3, 140.1, 125.2, 120.8, 120.2, 111.1, 79.7, 77.7, 75.0, 69.7, 30.6, 25.8, 25.7; HRMS (TOF MS ESI^{+}) calculated for $C_{15}H_{14}N_2O_4SNa$ $[M+Na]^{+}$: 341.0566, found 341.0559.



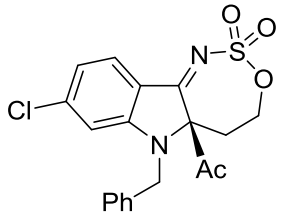
1-{2,2-Dioxido-6-[4-(trifluoromethyl)benzyl]-4,5-dihydro [1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl}ethan-1-one (2d). Yellow oil. 53.5 mg, 61% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.77 (d, *J* = 8.0 Hz, 1H), 7.69 – 7.63 (comp, 3H), 7.46 (d, *J* = 7.9 Hz, 2H), 6.99 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 8.4 Hz, 1H), 4.81 (d, *J* = 17.8 Hz, 1H), 4.75 – 4.70 (m, 1H), 4.58 (d, *J* = 12.5 Hz, 1H), 4.48 (d, *J* = 17.8 Hz, 1H), 2.54 (d, *J* = 14.4 Hz, 1H), 2.22 – 2.16 (m, 1H), 1.93 (s, 3H) ; ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 194.0, 179.9, 158.5, 141.6, 140.3, 128.0 (q, *J* = 31.8 Hz), 127.3, 125.5 (q, *J* = 3.5 Hz), 125.3, 128.0 (q, *J* = 31.8 Hz), 120.6, 120.2, 110.9, 80.5, 69.7, 44.5, 26.1, 24.9; ^{19}F NMR (471 MHz, DMSO-d6) (δ, ppm) -60.93; HRMS (TOF MS ESI^{+}) calculated for $C_{20}H_{17}F_3N_2O_4SNa$ [M+Na]$^{+}$: 461.0753, found 461.0758.



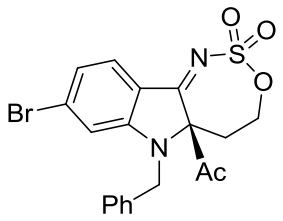
1-[6-Benzyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]eth an-1-one (2e). Yellow oil. 60.0 mg, 81% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.74 (d, *J* = 7.9 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.1 Hz, 2H), 7.28 – 7.26 (m, 1H), 7.23 (d, *J* = 7.3 Hz, 2H), 6.96 (t, *J* = 7.3 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 4.76 – 4.66 (m, 2H), 4.57 (d, *J* = 12.5 Hz, 1H), 4.38 (d, *J* = 17.2 Hz, 1H), 2.56 (d, *J* = 14.3 Hz, 1H), 2.16 – 2.10 (m, 1H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 194.0, 180.0, 158.7, 140.2, 136.6, 128.7, 127.4, 125.6, 125.3, 120.4, 120.1, 111.0, 80.5, 69.8, 44.9, 26.1, 24.8; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{18}N_2O_4SNa$ [M+Na]$^{+}$: 393.0879, found 393.0878.



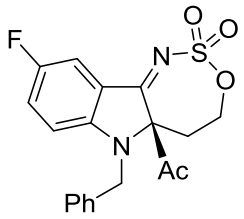
1-[6-Benzyl-8-fluoro-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2f). Yellow oil. 40.4 mg, 52% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.84 – 7.82 (m, 1H), 7.35 – 7.32 (m, 2H), 7.29 – 7.27 (m, 1H), 7.23 (d, *J* = 7.4 Hz, 2H), 6.81 (t, *J* = 8.9 Hz, 1H), 6.75 (d, *J* = 10.2 Hz, 1H), 4.72 – 4.67 (m, 2H), 4.56 (d, *J* = 12.4 Hz, 1H), 4.39 (d, *J* = 17.2 Hz, 1H), 2.54 (d, *J* = 14.6 Hz, 1H), 2.16 – 2.10 (m, 1H), 1.92 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.9, 178.6, 170.2 (d, *J* = 257.1 Hz), 160.6 (d, *J* = 15.1 Hz), 136.2, 128.7, 128.4 (d, *J* = 13.2 Hz), 127.5, 125.8, 117.0, 109.4 (d, *J* = 25.6 Hz), 97.4 (d, *J* = 27.6 Hz), 81.3, 69.6, 45.1, 26.2, 24.9; ^{19}F NMR (471 MHz, DMSO-d6) (δ, ppm) -95.12; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{17}FN_2O_4SNa$ $[M+Na]^{+}$: 411.0785, found 411.0780.



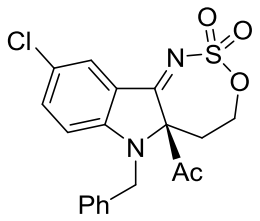
1-[6-Benzyl-8-chloro-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2g). Yellow oil. 44.5 mg, 55% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.76 (d, *J* = 9.0 Hz, 1H), 7.36 – 7.32 (m, 2H), 7.30 – 7.26 (m, 1H), 7.22 (d, *J* = 7.2 Hz, 2H), 6.99 – 6.97 (m, 2H), 4.75 – 4.67 (m, 2H), 4.60 – 4.54 (m, 1H), 4.38 (d, *J* = 17.3 Hz, 1H), 2.54 (d, *J* = 14.7 Hz, 1H), 2.20 – 2.12 (m, 1H), 1.93 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 193.8, 178.9, 159.0, 145.0, 136.2, 128.7, 127.5, 125.9, 125.6, 120.8, 119.0, 110.6, 81.0, 69.7, 44.9, 26.2, 25.0; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{17}ClN_2O_4SNa$ $[M+Na]^{+}$: 427.0490, found 427.0491.



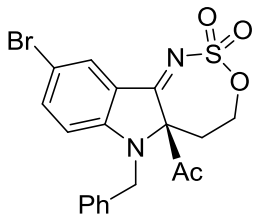
1-[6-Benzyl-8-bromo-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2h). Yellow oil. 51.2 mg, 57% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.67 (d, *J* = 8.2 Hz, 1H), 7.36 – 7.33 (m, 2H), 7.30 – 7.27 (m, 1H), 7.22 (d, *J* = 7.1 Hz, 2H), 7.11 (d, *J* = 10.3 Hz, 2H), 4.75 – 4.68 (m, 2H), 4.58 – 4.56 (m, 1H), 4.38 (d, *J* = 17.2 Hz, 1H), 2.54 (d, *J* = 14.7 Hz, 1H), 2.19 – 2.13 (m, 1H), 1.94 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.7, 179.1, 158.9, 136.2, 134.6, 128.7, 127.5, 125.7, 125.6, 123.6, 119.3, 113.6, 80.9, 69.7, 44.9, 26.1, 25.0; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{17}BrN_2O_4SNa$ $[M+Na]^{+}$: 470.9985, found 470.9982.



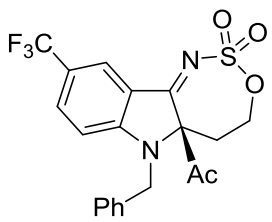
1-[6-Benzyl-9-fluoro-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2i). Yellow oil. 41.9 mg, 54% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.59 – 7.52 (m, 2H), 7.35 – 7.32 (m, 2H), 7.29 – 7.26 (m, 1H), 7.23 (d, *J* = 7.5 Hz, 2H), 6.84 (dd, *J* = 9.0, 2.9 Hz, 1H), 4.76 – 4.66 (m, 2H), 4.60 – 4.57 (m, 1H), 4.37 (d, *J* = 17.2 Hz, 1H), 2.56 (d, *J* = 14.4 Hz, 1H), 2.20 – 2.14 (m, 1H), 1.93 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.7, 179.8, 155.9, 156.3 (d, *J* = 239.2 Hz), 136.4, 128.7, 128.5, 127.4, 125.6, 120.2 (d, *J* = 9.1 Hz), 112.6 (d, *J* = 8.1 Hz), 109.5 (d, *J* = 23.5 Hz), 81.2, 69.9, 45.1, 26.1, 24.9; ^{19}F NMR (471 MHz, DMSO-d6) (δ, ppm) -122.97; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{17}FN_2O_4SNa$ $[M+Na]^{+}$: 411.0785, found 411.0780.



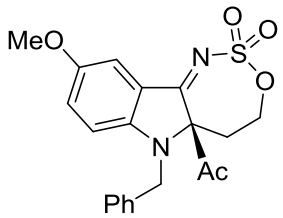
1-[6-Benzyl-9-chloro-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2j). Yellow oil. 40.5 mg, 50% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.74 (s, 1H), 7.65 (d, *J* = 8.9 Hz, 1H), 7.34 – 7.32 (m, 2H), 7.29 – 7.26 (m, 1H), 7.22 (d, *J* = 7.5 Hz, 2H), 6.85 (d, *J* = 8.9 Hz, 1H), 4.75 – 4.68 (m, 2H), 4.60 – 4.57 (m, 1H), 4.38 (d, *J* = 17.3 Hz, 1H), 2.56 (d, *J* = 14.5 Hz, 1H), 2.22 – 2.16 (m, 1H), 1.94 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.7, 179.0, 157.3, 139.6, 136.2, 128.7, 127.5, 125.6, 124.2, 123.9, 121.1, 112.8, 81.1, 69.9, 45.0, 26.0, 25.0; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{17}ClN_2O_4SNa$ [M+Na]$^{+}$: 427.0490, found 427.0490.



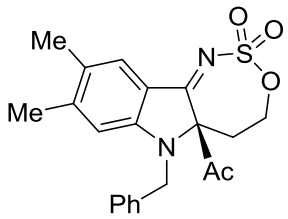
1-[6-Benzyl-9-bromo-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2k). Yellow oil. 47.6 mg, 53% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.86 (d, *J* = 2.0 Hz, 1H), 7.75 (dd, *J* = 8.9, 2.1 Hz, 1H), 7.35 – 7.27 (comp, 3H), 7.22 (d, *J* = 7.1 Hz, 2H), 6.80 (d, *J* = 8.9 Hz, 1H), 4.76 – 4.67 (m, 2H), 4.61 – 4.56 (m, 1H), 4.38 (d, *J* = 17.3 Hz, 1H), 2.56 (d, *J* = 14.6 Hz, 1H), 2.22 – 2.14 (m, 1H), 1.94 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 193.7, 178.8, 157.5, 142.1, 136.2, 128.7, 127.5, 127.0, 125.6, 121.7, 113.2, 111.5, 81.0, 69.8, 45.0, 26.0, 25.0; HRMS (TOF MS ESI^{+}) calculated for $C_{19}H_{17}BrN_2O_4SNa$ [M+Na]$^{+}$: 470.9985, found 470.9990.



1-[6-Benzyl-2,2-dioxido-9-(trifluoromethyl)-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2l). Yellow oil. 30.7 mg, 35% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 8.01 (s, 1H), 7.92 (d, *J* = 8.7 Hz, 1H), 7.35 – 7.26 (comp, 3H), 7.24 (d, *J* = 7.5 Hz, 2H), 6.99 (d, *J* = 8.9 Hz, 1H), 4.80 – 4.70 (m, 2H), 4.63 – 4.58 (m, 1H), 4.45 (d, *J* = 17.4 Hz, 1H), 2.61 (d, *J* = 14.8 Hz, 1H), 2.29 – 2.21 (m, 1H), 1.97 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.7, 179.4, 159.8, 135.9, 135.7, 128.8, 127.5, 125.6, 124.0 (q, *J* = 271.4 Hz), 122.7 (q, *J* = 3.8 Hz), 120.4 (q, *J* = 33.2 Hz), 119.8, 112.0, 81.2, 69.8, 45.0, 26.0, 25.2; ^{19}F NMR (376 MHz, DMSO-d6) (δ, ppm) -60.34; HRMS (TOF MS ESI^{+}) calculated for $C_{20}H_{17}F_3N_2O_4SNa$ [M+Na]$^{+}$: 461.0753, found 461.0764.

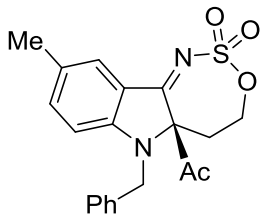


1-[6-Benzyl-9-methoxy-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2m). Red oil. 60.9 mg, 76% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.34 – 7.32 (m, 3H), 7.28 – 7.25 (m, 1H), 7.22 (d, *J* = 7.1 Hz, 2H), 7.10 (s, 1H), 6.80 (d, *J* = 8.9 Hz, 1H), 4.74 (t, *J* = 11.9 Hz, 1H), 4.65 (d, *J* = 17.1 Hz, 1H), 4.56 (d, *J* = 12.1 Hz, 1H), 4.36 (d, *J* = 17.1 Hz, 1H), 3.78 (s, 3H), 2.54 (d, *J* = 14.5 Hz, 1H), 2.10 – 2.06 (m, 1H), 1.90 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 193.9, 179.4, 155.2, 153.7, 136.8, 131.8, 128.7, 127.4, 125.6, 120.1, 112.6, 103.7, 81.1, 69.8, 55.7, 45.1, 26.2, 24.7; HRMS (TOF MS ESI^{+}) calculated for $C_{20}H_{20}N_2O_5SNa$ [M+Na]$^{+}$: 423.0985, found 423.0982.

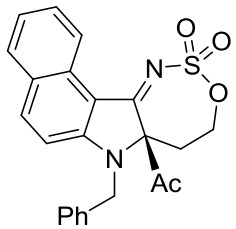


1-[6-Benzyl-8,9-dimethyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2n). Yellow solid, mp = 200 – 201 ^{o}C. 56.6 mg, 71% yield;

^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.56 (s, 1H), 7.39 – 7.36 (m, 2H), 7.33 – 7.29 (m, 1H), 7.25 (d, *J* = 7.1 Hz, 2H), 6.80 (s, 1H), 4.78 – 4.70 (m, 2H), 4.59 – 4.54 (m, 1H), 4.37 (d, *J* = 17.3 Hz, 1H), 2.55 – 2.54 (m, 1H), 2.29 (s, 3H), 2.25 (s, 3H), 2.07 – 2.00 (m, 1H), 1.91 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 194.2, 179.0, 158.2, 152.3, 136.8, 129.9, 128.7, 127.3, 125.5, 124.3, 118.2, 111.1, 80.7, 69.6, 44.7, 26.4, 24.6, 21.4, 18.7. HRMS (TOF MS ESI$^+$) calculated for $C_{21}H_{22}N_2O_4SNa$ [M+Na]$^+$: 421.1192, found 421.1198.

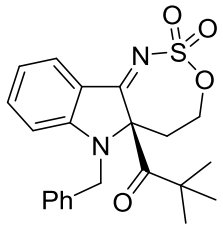


1-[6-Benzyl-9-methyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]ethan-1-one (2o). Yellow oil. 53.8 mg, 70% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.54 (s, 1H), 7.49 – 7.46 (m, 1H), 7.34 – 7.31 (m, 2H), 7.28 – 7.25 (m, 1H), 7.22 – 7.20 (m, 2H), 6.75 (d, *J* = 8.6 Hz, 1H), 4.76 – 4.69 (m, 1H), 4.65 (d, *J* = 17.2 Hz, 1H), 4.58 – 4.53 (m, 1H), 4.35 (d, *J* = 17.2 Hz, 1H), 2.54 (d, *J* = 14.7 Hz, 1H), 2.27 (s, 3H), 2.11 – 2.06 (m, 1H), 1.89 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 194.0, 179.7, 157.4, 141.9, 136.7, 129.8, 128.7, 127.4, 125.6, 124.1, 120.1, 110.9, 80.7, 69.7, 44.9, 26.2, 24.7, 19.9; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{20}N_2O_4SNa$ [M+Na]$^+$: 407.1036, found 407.1035.

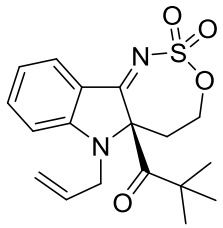


1-[7-Benzyl-11,11-dioxido-8,9-dihydrobenzo[*e*][1,2,3]oxathiazepino[5,4-*b*]indol-7a(7*H*)-yl]ethan-1-one (2p). Yellow oil. 16.8 mg, 20% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 8.90 (d, *J* = 8.1 Hz, 1H), 8.23 (d, *J* = 9.2 Hz, 1H), 7.94 (d, *J* = 7.7 Hz, 1H), 7.79 – 7.75 (m, 1H), 7.52 – 7.48 (m, 1H), 7.36 – 7.28 (comp, 3H), 7.25 – 7.23(m, 2H), 7.17 (d,

J = 9.2 Hz, 1H), 4.93 (d, *J* = 17.5 Hz, 1H), 4.78 – 4.71 (m, 1H), 4.56 – 4.52 (m, 2H), 2.56 – 2.53 (m, 1H), 2.15 – 2.07 (m, 1H), 1.95 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 194.5, 162.3, 143.3, 136.2, 131.0, 129.9, 129.4, 128.7, 128.3, 127.5, 125.5, 125.2, 123.1, 112.1, 110.6, 81.6, 69.3, 44.8, 26.3, 24.6; HRMS (TOF MS ESI$^+$) calculated for $C_{23}H_{20}N_2O_4SNa$ $[M+Na]^+$: 443.1036, found 443.1031.

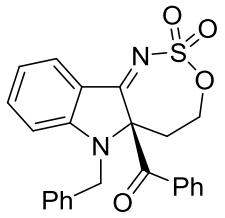


1-[6-Benzyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]-2,2-dimethylpropan-1-one (2q). Yellow oil. 64.4 mg, 78% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.75 (d, *J* = 7.9 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.32 – 7.29 (m, 2H), 7.26 – 7.23 (m, 1H), 7.18 (d, *J* = 7.3 Hz, 2H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 8.3 Hz, 1H), 4.86 (d, *J* = 17.7 Hz, 1H), 4.62 – 4.58 (m, 1H), 4.49 – 4.48 (m, 1H), 4.16 (d, *J* = 17.7 Hz, 1H), 2.43 – 2.40 (m, 1H), 1.98 – 1.91 (m, 1H), 1.06 (s, 9H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 202.3, 179.8, 157.7, 139.9, 136.5, 128.7, 127.2, 125.5, 125.3, 120.5, 120.2, 110.6, 80.0, 69.0, 46.4, 44.8, 28.8, 28.3; HRMS (TOF MS ESI$^+$) calculated for $C_{22}H_{24}N_2O_4SNa$ $[M+Na]^+$: 435.1349, found 435.1345.

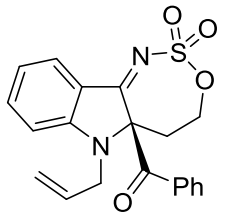


1-[6-Allyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]-2,2-dimethylpropan-1-one (2r). Yellow oil. 49.9 mg, 69% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.71 – 7.64 (m, 2H), 6.98 – 6.92 (m, 2H), 5.77 – 5.67 (m, 1H), 5.25 – 5.17 (m, 2H), 4.73 – 4.67 (m, 1H), 4.59 – 4.54 (m, 1H), 4.14 (dd, *J* = 17.5, 5.9 Hz, 1H), 3.54 (dd, *J* = 17.5, 4.7 Hz, 1H), 2.63 – 2.59 (m, 1H), 1.92 – 1.84 (m, 1H), 1.02 (s, 9H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 202.3, 180.4, 157.2, 139.7, 132.6, 125.2,

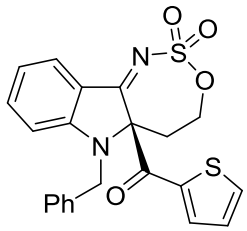
120.6, 120.0, 118.2, 110.9, 79.9, 69.4, 46.2, 43.8, 28.3, 28.1. HRMS (TOF MS ESI$^+$) calculated for $C_{18}H_{22}N_2O_4SNa$ [M+Na]$^+$: 385.1192, found 385.1190.



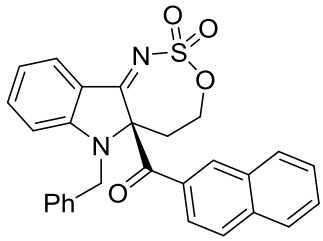
[6-Benzyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl](phenyl)methanone (2s). Yellow oil. 64.0 mg, 74% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.80 (d, *J* = 7.9 Hz, 1H), 7.58 – 7.54 (m, 1H), 7.52 – 7.49 (comp, 3H), 7.34 – 7.29 (m, 2H), 7.25 – 7.20 (comp, 3H), 7.13 – 7.11 (m, 2H), 6.96 (t, *J* = 7.5 Hz, 1H), 6.71 (d, *J* = 8.5 Hz, 1H), 4.94 – 4.87 (m, 1H), 4.68 – 4.60 (m, 2H), 4.48 (d, *J* = 17.2 Hz, 1H), 2.79 – 2.75 (m, 1H), 2.20 – 2.12 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 189.7, 180.2, 158.1, 140.2, 136.3, 135.0, 133.3, 128.5, 128.5, 127.6, 127.3, 125.6, 125.3, 120.52, 120.48, 110.9, 79.8, 69.9, 45.1, 27.5; HRMS (TOF MS ESI$^+$) calculated for $C_{24}H_{21}N_2O_4S$ [M+H]$^+$: 433.1217, found 433.1215.



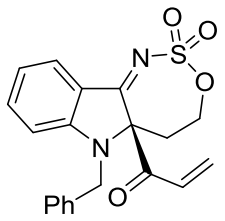
[6-Allyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl](phenyl)methanone (2t). Yellow oil, 54.5 mg, 71% yield; ^{1}H NMR (400 MHz, C_6D_6) (δ, ppm) 7.75 – 7.70 (comp, 3H), 6.93 – 6.85 (m, 2H), 6.80 – 6.76 (m, 2H), 6.43 (t, *J* = 7.5 Hz, 1H), 6.11 (d, *J* = 8.4 Hz, 1H), 5.17 – 5.08 (m, 1H), 4.85 (s, 1H), 4.75 – 4.64 (m, 2H), 3.93 – 3.85 (m, 1H), 3.36 – 3.21 (m, 2H), 2.08 (d, *J* = 14.0 Hz, 1H), 1.08 – 1.01 (m, 1H); ^{13}C NMR (100 MHz, C_6D_6) (δ, ppm) 190.1, 179.4, 156.9, 138.6, 135.9, 133.2, 132.6, 128.6, 126.2, 121.8, 120.1, 117.4, 110.0, 79.6, 68.9, 44.0, 28.4; HRMS (TOF MS ESI$^+$) calculated for $C_{20}H_{19}N_2O_4S$ [M+H]$^+$: 383.1060, found 383.1065.



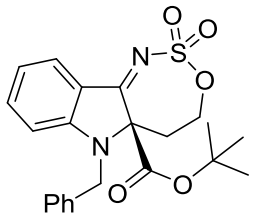
[6-Benzyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl](thiophen-2-yl)methanone (2u). Yellow oil, 81.6 mg, 93% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.98 – 7.97 (m, 1H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.64 – 7.60 (m, 1H), 7.28 – 7.26 (m, 1H), 7.21 – 7.19 (comp, 3H), 7.09 – 6.99 (comp, 4H), 6.76 (d, *J* = 8.5 Hz, 1H), 4.88 – 4.82 (m, 1H), 4.66 – 4.61 (m, 2H), 4.52 (d, *J* = 17.2 Hz, 1H), 2.84 – 2.80 (m, 1H), 2.19 – 2.12 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 181.3, 179.4, 157.9, 140.3, 139.7, 136.4, 136.2, 131.1, 128.6, 128.4, 127.2, 125.5, 125.2, 120.6, 120.4, 111.1, 78.9, 69.6, 45.0, 27.4; HRMS (TOF MS ESI^{+}) calculated for $C_{22}H_{19}N_2O_4S_2$ $[M+H]^{+}$: 439.0781, found 439.0782.



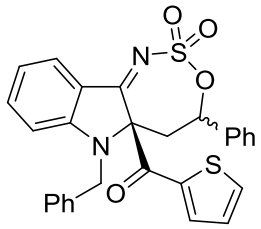
[6-Benzyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl](naphthalen-2-yl)methanone (2v). Yellow oil. 74.3 mg, 77% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 8.04 (s, 1H), 7.91 – 7.85 (m, 3H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.63 – 7.52 (comp, 4H), 7.18 (s, 3H), 7.15 (s, 2H), 6.99 (t, *J* = 7.3 Hz, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 4.95 (t, *J* = 11.6 Hz, 1H), 4.71 – 4.65 (m, 2H), 4.54 (d, *J* = 17.2 Hz, 1H), 2.86 – 2.83 (m, 1H), 2.25 – 2.19 (m, 1H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 189.5, 180.3, 158.1, 140.2, 136.3, 134.8, 132.3, 131.4, 129.1, 128.9, 128.7, 128.5, 128.2, 127.6, 127.3, 127.2, 125.7, 125.2, 123.8, 120.6, 120.6, 111.0, 79.9, 70.0, 45.2, 27.6; HRMS (TOF MS ESI^{+}) calculated for $C_{28}H_{22}N_2O_4SNa$ $[M+Na]^{+}$: 505.1192, found 505.1195.



1-[6-Benzyl-2,2-dioxido-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl]prop-2-en-1-one (2w). Yellow oil. 65.8 mg, 86% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.76 (d, *J* = 7.8 Hz, 1H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.32 – 7.30 (m, 2H), 7.26 (d, *J* = 6.8 Hz, 1H), 7.23 – 7.22 (m, 2H), 6.97 (t, *J* = 7.1 Hz, 1H), 6.79 (d, *J* = 8.2 Hz, 1H), 6.37 (d, *J* = 16.7 Hz, 1H), 6.03 – 5.98 (m, 1H), 5.82 (d, *J* = 10.2 Hz, 1H), 4.84 (t, *J* = 11.7 Hz, 1H), 4.66 – 4.59 (m, 2H), 4.38 (d, *J* = 17.2 Hz, 1H), 2.64 – 2.61 (m, 1H), 2.21 – 2.16 (m, 1H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 185.1, 179.4, 158.8, 140.1, 136.4, 133.1, 130.3, 128.6, 127.4, 125.7, 125.3, 120.4, 110.9, 79.8, 69.7, 45.0, 26.2; HRMS (TOF MS ESI^{+}) calculated for $C_{20}H_{18}N_2O_4SNa$ $[M+Na]^{+}$: 405.0879, found 405.0880.

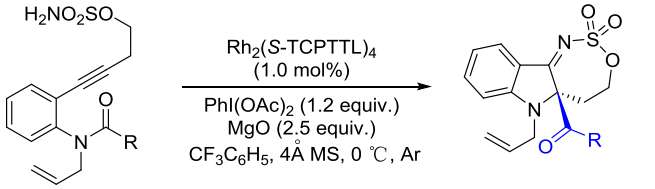


***tert*-Butyl 6-benzyl-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indole-5a(6*H*)-carboxylate 2,2-dioxide (2x).** Yellow oil. 42.0 mg, 49% yield; ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.54 – 7.50 (m, 2H), 7.35 – 7.15 (m, 1H), 7.05 – 6.94 (comp, 6H), 5.00 – 4.94 (m, 1H), 4.72 – 4.66 (m, 1H), 4.04 (d, *J* = 13.6 Hz, 1H), 3.71 (d, *J* = 13.6 Hz, 1H), 3.24 – 3.21 (m, 1H), 2.48 – 2.41 (m, 1H), 1.67 (s, 9H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 180.0, 151.0, 149.5, 138.5, 133.4, 129.1, 127.8, 127.1, 123.7, 123.6, 123.1, 115.5, 83.8, 78.9, 75.8, 68.2, 27.9, 27.6; HRMS (TOF MS ESI^{+}) calculated for $C_{22}H_{24}N_2O_5SNa$ $[M+Na]^{+}$: 451.1298, found 451.1297.



[6-Benzyl-2,2-dioxido-4-phenyl-4,5-dihydro-[1,2,3]oxathiazepino[5,4-*b*]indol-5a(6*H*)-yl](thiophen-2-yl)methanone (2y). Yellow oil. 67.9 mg, 66% yield, > 95:5 *dr*. ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.99 – 7.98 (m, 1H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.65 – 7.62 (m, 1H), 7.53 – 7.51 (m, 2H), 7.45 – 7.42 (comp, 3H), 7.28 – 7.27 (m, 1H), 7.15 – 7.13 (comp, 3H), 7.08 – 7.05 (m, 1H), 7.04 – 7.01 (comp, 3H), 6.72 (d, *J* = 8.5 Hz, 1H), 6.12 (d, *J* = 10.9 Hz, 1H), 4.57 (s, 2H), 2.87 (d, *J* = 13.6 Hz, 1H), 2.59 (dd, *J* = 14.6, 11.7 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d6) (δ, ppm) 181.7, 180.7, 158.2, 140.6, 139.9, 137.6, 136.3, 136.1, 130.9, 129.5, 128.8, 128.6, 128.3, 127.1, 125.9, 125.4, 125.2, 121.0, 120.7, 111.3, 84.7, 78.5, 45.0, 32.7; HRMS (TOF MS ESI^{+}) calculated for $C_{28}H_{22}N_2O_4S_2Na$ $[M+Na]^{+}$: 537.0913, found 537.0919.

General Procedure for the Asymmetric Cascade Reaction:



1a, R = Me
1r, R = ^{t}Bu

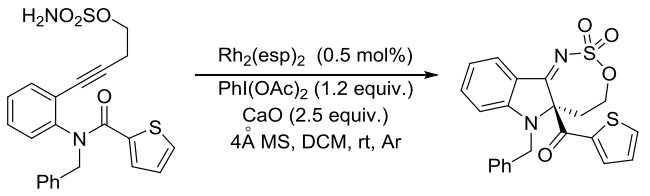
2a, 64% yield, 83% *ee*
2r, 59% yield, 63% *ee*

To a 10-mL oven-dried vial containing a magnetic stirring bar, Rh_2(*S*-TCPTTL)$_4$ (1.8 mg, 1.0 mol%), PhI(OAc)$_2$ (38.7 mg, 1.2 equiv.), MgO (10.0 mg, 2.5 equiv.), and 4Å MS (50.0 mg) in trifluoromethyl benzene (1.0 mL), was added as a solution of sulfamate ester **1** (0.1 mmol) in trifluoromethyl benzene (1.0 mL) *via* a syringe at 0 °C under argon atmosphere. After addition, the reaction mixture was stirred under these conditions until consumption of the material (monitored by TLC, about 4 days). Then the reaction mixture was purified by column chromatography on silica gel without any additional treatment (Hexanes : EtOAc = 4:1 to 2:1) to give the pure products **2** as yellow oil.

2a, 20.5 mg, 64% yield. 83% *ee*; HPLC conditions for determination of enantiomeric excess: Daicel Chiralpak AD-H, λ = 254 nm, hexane : 2-propanol = 85:15, flow rate = 1.0 mL/min, t_{major} = 30.8 min, t_{minor} = 42.2 min.

2r, 21.4 mg, 59% yield. 63% *ee*; HPLC conditions for determination of enantiomeric excess: Daicel Chiralpak AD-H, λ = 254 nm, hexane : 2-propanol = 85:15, flow rate = 1.0 mL/min, t_{major} = 13.0 min, t_{minor} = 16.8 min.

General Procedure of the Gram Scale Reaction

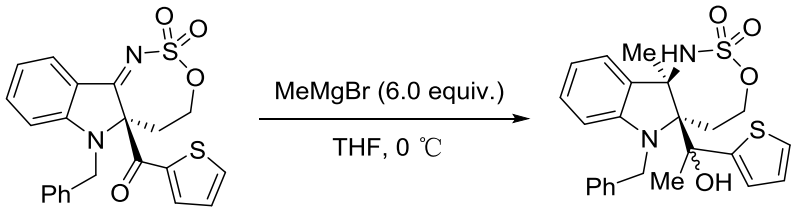


1u, 3.5 mmol, 1.54 g

2u, 90% yield, 1.38 g

To a 50-mL oven-dried vial containing a magnetic stirring bar, $Rh_2(esp)_2$ (13.3 mg, 0.5 mol%), $PhI(OAc)_2$ (1.35 g, 4.2 mmol, 1.2 equiv.), CaO (490 mg, 8.75 mmol, 2.5 equiv.), and 4Å MS (1.0 g) in DCM (10 mL), was added as a solution of sulfamate ester **1u** (1.54 g, 3.5 mmol) in DCM (5 mL) *via* a syringe under argon atmosphere at room temperature. After addition, the reaction mixture was stirred for additional 12 h. Then most of the solvent was evaporated in vacuo (about 2 mL left), the residue was purified by column chromatography on silica gel (Hexanes : EtOAc = 4:1 to 2:1) to give 1.38 g of pure **2u** in 90% yield.

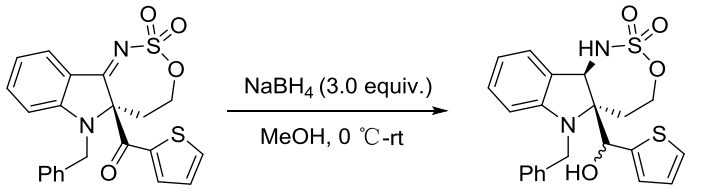
General Procedure for the Synthesis of 3



2u **3**, 77% yield, > 95:5 *dr*

To a 10-mL oven-dried vial containing a magnetic stirring bar, a solution of methyl magnesium bromide (3.0 M in 2-methyl-THF, 0.4 mL, 1.2 mmol) in THF (1.0 mL), was added as a solution of **2u** (87.6 mg, 0.2 mmol, 1.0 equiv.) in THF (1.0 mL) *via* a syringe pump over 2 h under argon atmosphere at 0 °C. After addition, the reaction mixture was stirred under these conditions until consumption of the material (monitored by TLC, about 1 h). The reaction mixture was quenched with saturated aqueous NH_4Cl solution (10 mL), and extracted with Et_2O (3 X 10 mL). The combined extract was washed with brine (10 mL), dried with Na_2SO_4, and then concentrated under reduced pressure after filtration. The crude product was purified by flash chromatography on silica gel (Hexanes : EtOAc = 5:1 to 3:1) to afford 72.4 mg pure product **3** in 77% yield. Colorless oil, > 95:5 *dr*. ^{1}H NMR (400 MHz, DMSO-d6) (δ, ppm) 7.97 (s, 1H), 7.54 (d, *J* = 4.9 Hz, 1H), 7.27 – 7.26 (comp, 4H), 7.22 – 7.17 (m, 1H), 7.12 – 7.09 (m, 2H), 7.08 – 7.05 (m, 1H), 7.03 – 7.00 (m, 1H), 6.97 (d, *J* = 7.1 Hz, 1H), 6.65 (t, *J* = 7.3 Hz, 1H), 6.48 (d, *J* = 7.7 Hz, 1H), 5.86 (t, *J* = 12.3 Hz, 1H), 4.48 (dd, *J* = 30.1, 14.2 Hz, 2H), 3.62 (d, *J* = 15.9 Hz, 1H), 2.54 – 2.50 (m, 1H), 2.41 – 2.39 (m, 1H), 1.63 (s, 3H), 1.21 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 152.8, 147.4, 138.9, 136.2, 128.4, 128.2, 127.8, 127.1, 125.8, 125.7, 125.5, 118.5, 117.9, 107.3, 83.6, 75.6, 69.1, 68.8, 47.7, 31.0, 29.6, 27.1; HRMS (TOF MS ESI$^+$) calculated for $C_{24}H_{27}N_2O_4S_2$ $[M+H]^+$: 471.1407, found 471.1410.

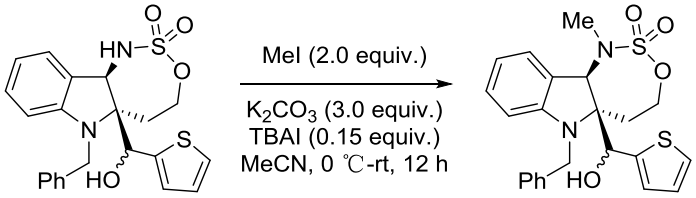
General Procedure for the Synthesis of 4



2u **4**, 85% yield, > 95:5 *dr*

To a 10-mL oven-dried vial containing a magnetic stirring bar, **2u** (87.6 mg, 0.2 mmol) in anhydrous MeOH (2.0 mL), was added $NaBH_4$ (22.7 mg, 0.6 mmol, 3.0 equiv.) at 0 °C. The reaction mixture was warmed to room temperature and stirred for additional 1 h under these conditions until consumption of the material (monitored by TLC). The reaction mixture was quenched with saturated aqueous NH_4Cl solution (10 mL), and extracted with Et_2O (2 X 10 mL). The combined extract was washed with brine (10 mL), dried with Na_2SO_4, and then concentrated under reduced pressure after filtration. The crude product was purified by flash chromatography on silica gel (Hexanes : EtOAc = 4:1 to 2:1) to give 75.1 mg pure product **4** in 85% yield. White solid, mp = 158 – 160 ^{o}C, > 95:5 *dr*. ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.89 (d, *J* = 10.8 Hz, 1H), 7.50 (d, *J* = 5.0 Hz, 1H), 7.25 – 7.22 (m, 2H), 7.18 – 7.14 (comp, 3H), 7.05 (d, *J* = 3.3 Hz, 1H), 7.01 – 6.99 (m, 1H), 6.91 (d, *J* = 7.1 Hz, 1H), 6.79 (t, *J* = 7.6 Hz, 1H), 6.47 (t, *J* = 7.3 Hz, 1H), 5.90 (d, *J* = 7.7 Hz, 1H), 5.78 (d, *J* = 4.7 Hz, 1H), 5.50 – 5.44 (m, 2H), 4.88 – 4.82 (m, 1H), 4.78 – 4.73 (m, 1H), 4.19 (d, *J* = 17.1 Hz, 1H), 3.69 (d, *J* = 17.1 Hz, 1H), 2.48 – 2.45 (m, 1H), 2.25 – 2.19 (m, 1H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 149.6, 145.8, 139.1, 128.4, 127.8, 127.6, 125.6, 125.6, 125.3, 125.4, 125.1, 119.7, 116.1, 104.5, 73.9, 71.6, 68.8, 63.0, 46.8, 34.1; HRMS (TOF MS ESI^{+}) calculated for $C_{22}H_{23}N_2O_4S_2$ $[M+H]^{+}$: 443.1094, found 443.1094.

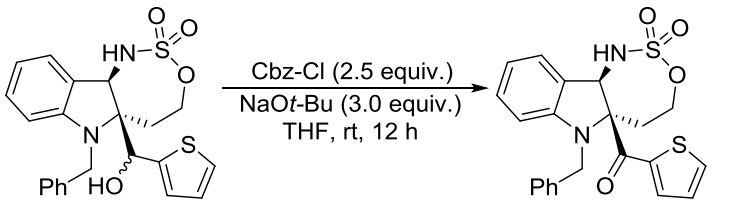
General Procedure for the Synthesis of 5



4 **5**, 88% yield, > 95:5 *dr*

To a 10-mL oven-dried vial containing a magnetic stirring bar, **4** (88.4 mg, 0.2 mmol) in MeCN (2.0 mL), was added MeI (24.9 µL, 0.4 mmol, 2.0 equiv.), K_2CO_3 (82.8 mg, 0.6 mmol, 3.0 equiv.), and *n*-Bu_4NI (11.1 mg, 0.03 mmol, 0.15 equiv.) in sequence at 0 ^oC, and the mixture was stirred at room temperature for 12 h. Then the reaction was quenched with H_2O (15 mL) and extracted with ethyl acetate (3 X 10 mL). The combined organic layer was washed successively with water and brine, dried over anhydrous Na_2SO_4, and then concentrated under reduced pressure after filtration. The crude product was purified by flash chromatography on silica gel (Hexanes : EtOAc = 10:1 to 5:1) to give 80.3 mg pure product **5** in 88% yield. White solid, mp = 137 – 139 ^oC, > 95:5 *dr*. ^1H NMR (500 MHz, DMSO-d6) (δ, ppm) 7.54 (d, *J* = 4.9 Hz, 1H), 7.25 – 7.22 (m, 2H), 7.17 – 7.15 (m, 2H), 7.09 – 7.05 (m, 2H), 6.95 (d, *J* = 7.1 Hz, 1H), 6.84 (t, *J* = 7.7 Hz, 1H), 6.50 (t, *J* = 7.3 Hz, 1H), 6.06 (d, *J* = 4.1 Hz, 1H), 5.99 (d, *J* = 7.8 Hz, 1H), 5.76 (s, 1H), 5.41 (d, *J* = 4.1 Hz, 1H), 4.85 (t, *J* = 11.3 Hz, 1H), 4.72 – 4.68 (m, 1H), 4.35 (d, *J* = 17.0 Hz, 1H), 3.71 (d, *J* = 17.0 Hz, 1H), 3.21 (s, 3H), 2.33 – 2.23 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 150.4, 146.8, 139.0, 128.4, 127.6, 125.8, 125.7, 125.3, 125.9, 125.34, 125.26, 121.0, 115.9, 104.9, 73.4, 70.0, 69.3, 67.5, 47.8, 34.5, 31.5; HRMS (TOF MS ESI$^+$) calculated for $C_{23}H_{24}N_2O_4S_2Na$ $[M+Na]^+$: 479.1070, found 479.1062.

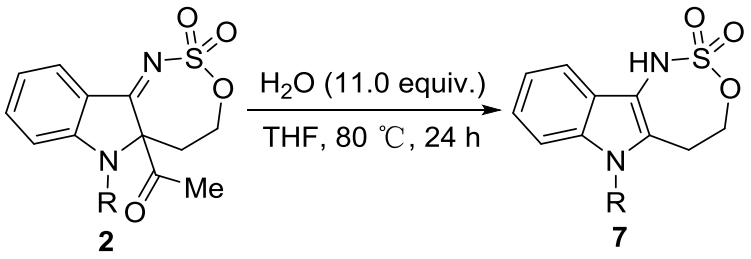
General Procedure for the Synthesis of 6



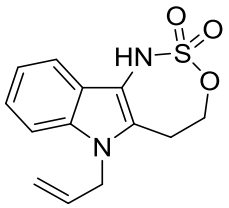
4 **6**, 51% yield, > 95:5 *dr*

To a 10-mL oven-dried vial containing a magnetic stirring bar, NaO*t*-Bu (57.6 mg, 0.6 mmol, 3.0 equiv.) in anhydrous THF (1.0 mL), was added as a solution of **4** (88.4 mg, 0.2 mmol) and Cbz-Cl (85.3 mg, 0.5 mmol, 2.5 equiv.) in THF (1.0 mL) at 0 ^{o}C, and the reaction mixture was stirred at room temperature for 12 h. Then the reaction was quenched with H_2O (15 mL) and extracted with ethyl acetate (3 X 10 mL). The combined organic layer was washed successively with water and brine, dried over anhydrous Na_2SO_4, and then concentrated under reduced pressure after filtration. The crude product was purified by flash chromatography on silica gel (Hexanes : EtOAc = 10:1 to 4:1) to give 44.9 mg pure product **6** in 51% yield. Yellow solid, mp = 214 – 216 ^{o}C, > 95:5 *dr*. ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.68 – 7.66 (m, 1H), 7.32 – 7.26 (comp, 6H), 7.11 – 7.06 (m, 2H), 6.98 – 6.97 (m, 1H), 6.89 – 6.86 (m, 1H), 6.29 (d, *J* = 7.5 Hz, 1H), 5.57 (dd, *J* = 30.9, 12.1 Hz, 2H), 4.71 – 4.60 (m, 2H), 4.25 (dd, *J* = 73.0, 16.5 Hz, 2H), 3.07 – 3.04 (m, 1H), 2.55 – 2.50 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 189.6, 149.0, 142.9, 137.9, 135.8, 132.6, 129.9, 129.0, 128.6, 127.7, 125.7, 125.6, 122.8, 120.4, 109.3, 80.2, 68.7, 64.8, 49.1, 35.9; HRMS (TOF MS ESI^{+}) calculated for $C_{22}H_{21}N_2O_4S_2$ $[M+H]^{+}$: 441.0937, found 441.0940.

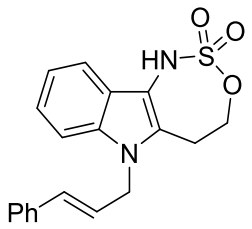
General Procedure for the Synthesis of 7



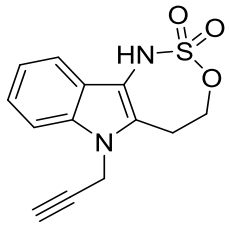
To a 10-mL oven-dried vial containing a magnetic stirring bar, **2** (0.1 mmol) in THF (2.0 mL), was added H_2O (20.0 μL, 1.1 mmol, 11.0 equiv.) at room temperature, and the reaction mixture was stirred at 80 °C for 24 h. Then the reaction was quenched with saturated aqueous $NaHCO_3$ solution (10 mL) and extracted with ethyl acetate (3 X 10 mL). The combined organic layer was washed successively with water and brine, dried over anhydrous Na_2SO_4, and then concentrated under reduced pressure after filtration. The crude product was purified by flash chromatography on silica gel (Hexanes : EtOAc = 5:1 to 3:1) to give pure products **7** in good to high yields.



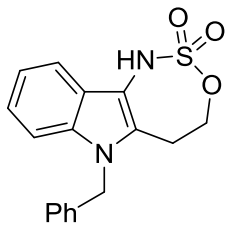
6-Allyl-1,4,5,6-tetrahydro-[1,2,3]oxathiazepino[5,4-*b*]indole 2,2-dioxide (7a). Colorless solid, mp = 151 – 153 °C. 18.9 mg, 68% yield; ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.59 (d, *J* = 7.8 Hz, 1H), 7.28 – 7.23 (m, 2H), 7.18 (t, *J* = 7.2 Hz, 1H), 6.38 (s, 1H), 5.97 – 5.90 (m, 1H), 5.17 (d, *J* = 10.4 Hz, 1H), 4.81 (d, *J* = 17.1 Hz, 1H), 4.69 – 4.66 (comp, 4H), 3.20 (t, *J* = 5.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 135.0, 132.9, 132.7, 124.4, 122.8, 120.9, 117.6, 116.9, 109.6, 109.4, 70.0, 45.3, 26.7; HRMS (TOF MS ESI$^+$) calculated for $C_{13}H_{14}N_2O_3SNa$ $[M+Na]^+$: 301.0617, found 301.0617.



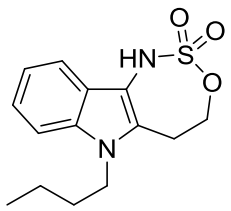
6-Cinnamyl-1,4,5,6-tetrahydro-[1,2,3]oxathiazepino[5,4-*b*]indole 2,2-dioxide (7b). Colorless oil. 26.2 mg, 74% yield; ^{1}H NMR (500 MHz, $CDCl_3$) (δ, ppm) 7.59 (d, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 8.2 Hz, 1H), 7.27 – 7.21 (comp, 6H), 7.17 (t, *J* = 7.4 Hz, 1H), 6.32 (s, 1H), 6.26 – 6.14 (m, 2H), 4.84 (d, *J* = 4.4 Hz, 2H), 4.64 (t, *J* = 5.0 Hz, 2H), 3.24 (t, *J* = 5.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 135.8, 135.1, 132.7, 131.8, 128.8, 128.3, 125.6, 124.5, 124.1, 123.0, 121.0, 117.6, 109.7, 109.6, 69.9, 45.0, 26.9; HRMS (TOF MS ESI$^+$) calculated for $C_{19}H_{19}N_2O_3S$ $[M+H]^+$: 355.1111, found 355.1116.



6-(Prop-2-yn-1-yl)-1,4,5,6-tetrahydro-[1,2,3]oxathiazepino[5,4-*b*]indole 2,2-dioxide (7c). Colorless solid, mp = 205 – 207 ^{o}C. 19.6 mg, 71% yield; ^{1}H NMR (500 MHz, DMSO-d6) (δ, ppm) 9.79 (s, 1H), 7.56 (d, *J* = 8.2 Hz, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 5.10 (s, 2H), 4.69 (t, *J* = 5.1 Hz, 2H), 3.35 – 3.32 (m, 3H); ^{13}C NMR (125 MHz, DMSO-d6) (δ, ppm) 134.2, 132.5, 124.2, 122.0, 120.2, 116.9, 110.0, 109.7, 79.2, 75.1, 68.6, 32.1, 26.2; HRMS (TOF MS ESI$^+$) calculated for $C_{13}H_{13}N_2O_3S$ $[M+H]^+$: 277.0641, found 277.0640.



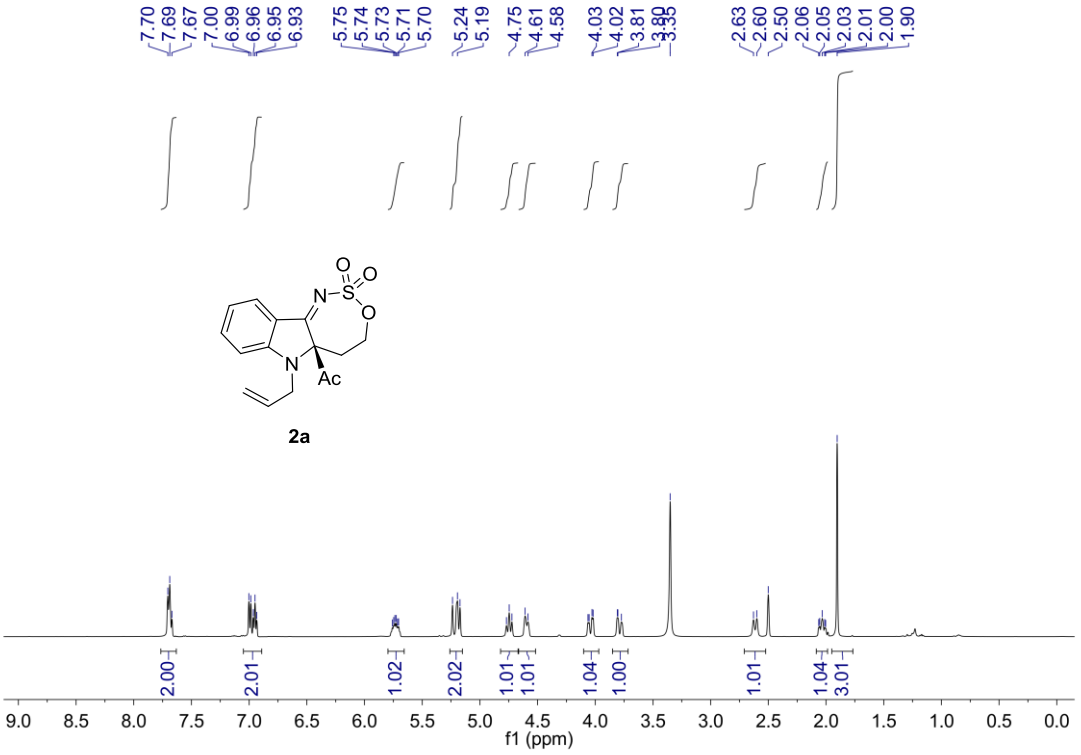
6-Benzyl-1,4,5,6-tetrahydro-[1,2,3]oxathiazepino[5,4-*b*]indole 2,2-dioxide (7e). Colorless oil. 23.6 mg, 72% yield; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.62 – 7.60 (m, 1H), 7.29 – 7.19 (comp, 6H), 6.94 (d, *J* = 7.0 Hz, 2H), 6.36 (s, 1H), 5.31 (s, 2H), 4.58 – 4.55 (m, 2H), 3.15 – 3.12 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 137.0, 135.5, 132.8, 129.2, 128.0, 125.8, 124.5, 123.1, 121.1, 117.7, 109.80, 109.75, 69.9, 46.7, 26.9; HRMS (TOF MS ESI$^+$) calculated for $C_{17}H_{17}N_2O_3S$ $[M+H]^+$: 329.0954, found 329.0957.



6-Butyl-1,4,5,6-tetrahydro-[1,2,3]oxathiazepino[5,4-*b*]indole 2,2-dioxide (7z) Colorless solid, mp = 58 – 60 ^{o}C. 18.2 mg, 62% yield; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.56 (d, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.2 Hz, 1H), 7.24 – 7.20 (m, 1H), 7.17 – 7.13 (m, 1H), 6.29 (s, 1H), 4.70 – 4.67 (m, 2H), 4.05 (t, *J* = 7.4 Hz, 2H), 3.26 – 3.23 (m, 2H), 1.73 – 1.65 (m, 1H), 1.40 – 1.29 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) (δ, ppm) 134.8, 132.6, 124.4, 122.5, 120.7, 117.5, 109.7, 108.9, 69.9, 43.2, 32.7, 26.8, 20.3, 13.9; HRMS (TOF MS ESI$^+$) calculated for $C_{14}H_{19}N_2O_3S$ $[M+H]^+$: 295.1111, found 295.1111.

References:

1 (a) D. Zhu, J. Ma, K. Luo, H. G. Fu, L. Zhang and S. F. Zhu, Enantioselective intramolecular C-H insertion of donor and donor/donor carbenes by a nondiazo approach. *Angew. Chem., Int. Ed.,* 2016, **55**, 8452-8456; (b) A. R. Thornton and S. B. Blakey, Catalytic metallonitrene/alkyne metathesis: a powerful cascade process for the synthesis of nitrogen-containing molecules. *J. Am. Chem. Soc.,* 2008, **130**, 5020-5021.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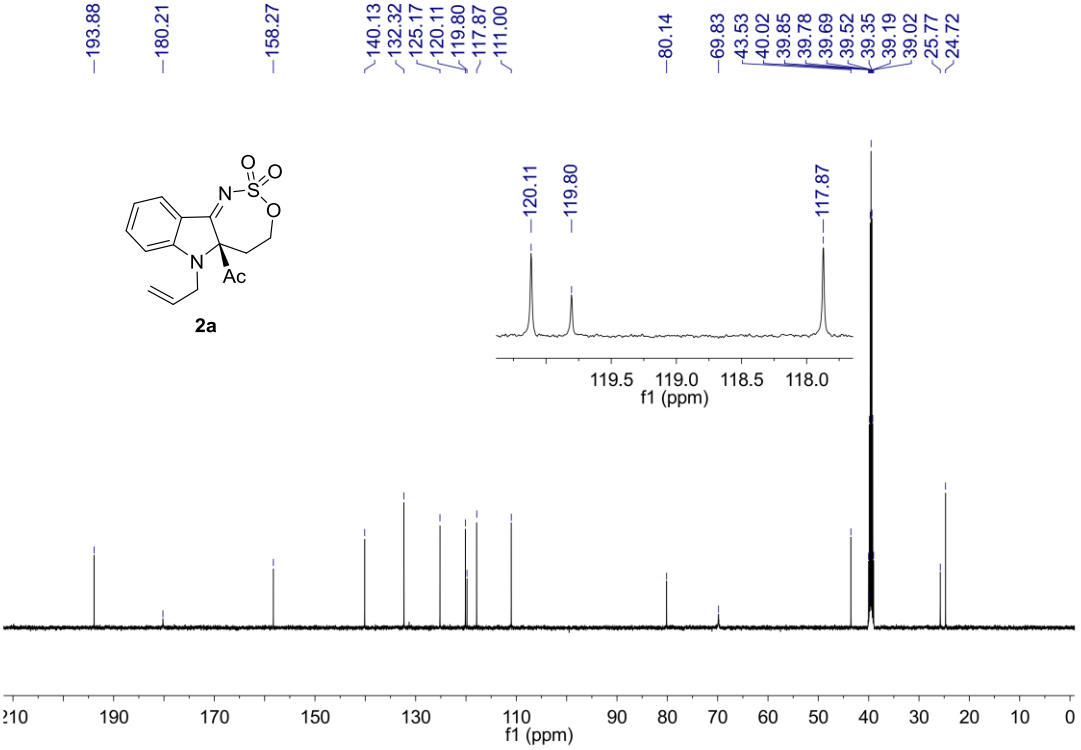
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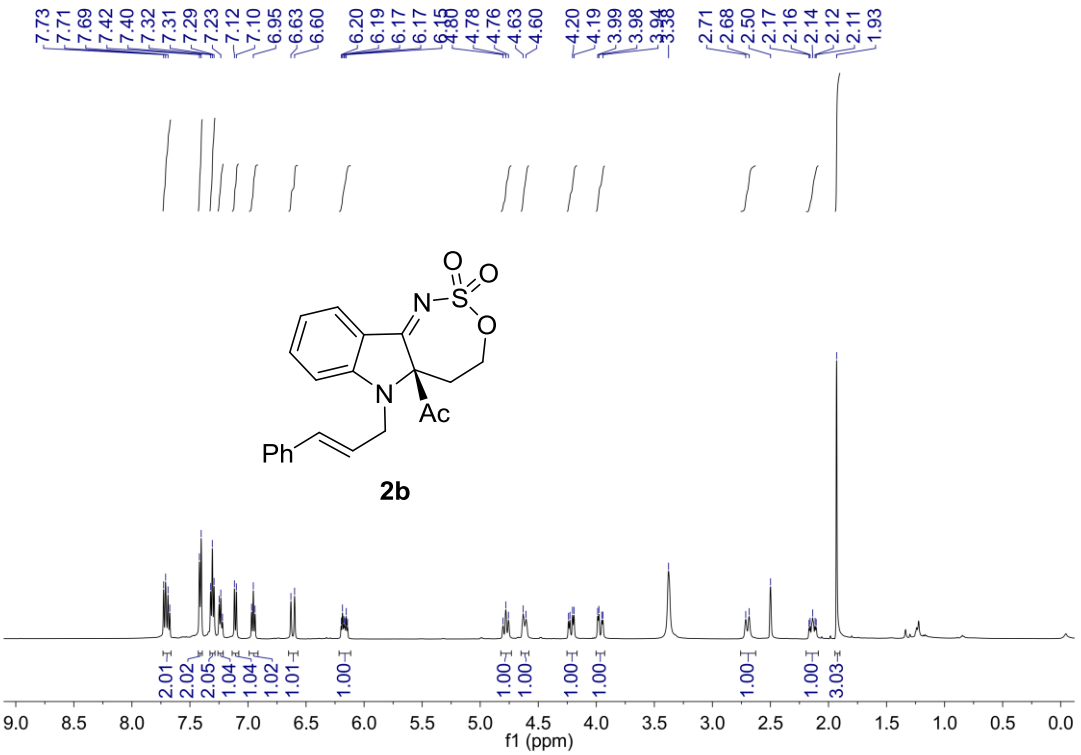
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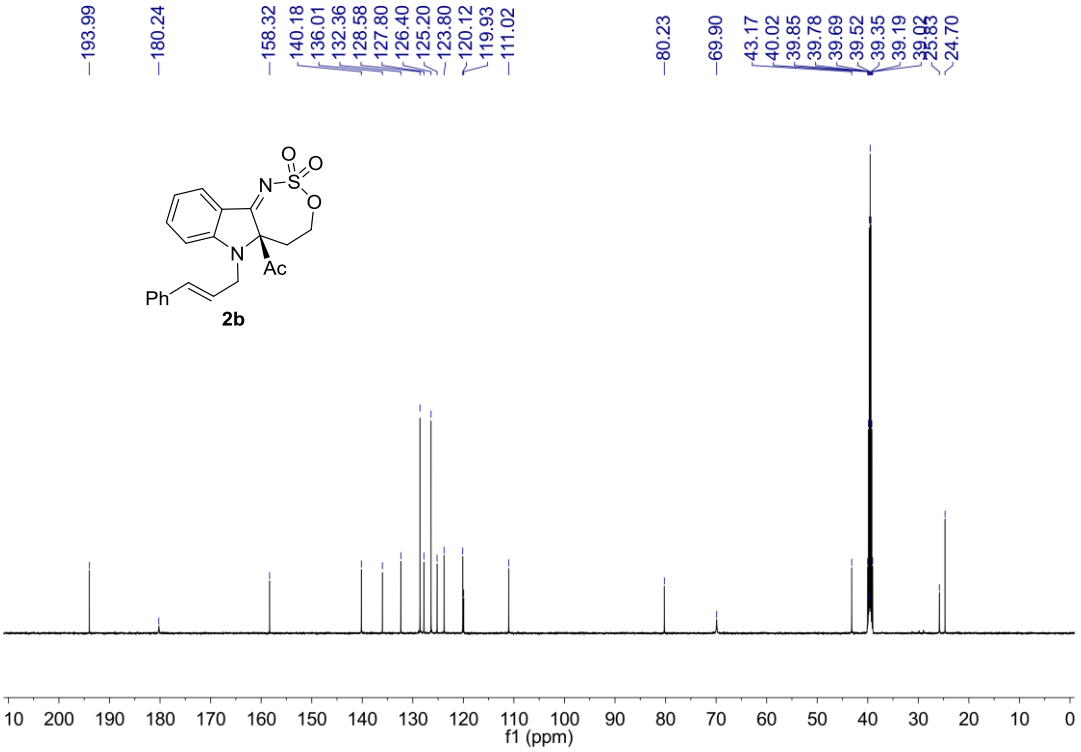
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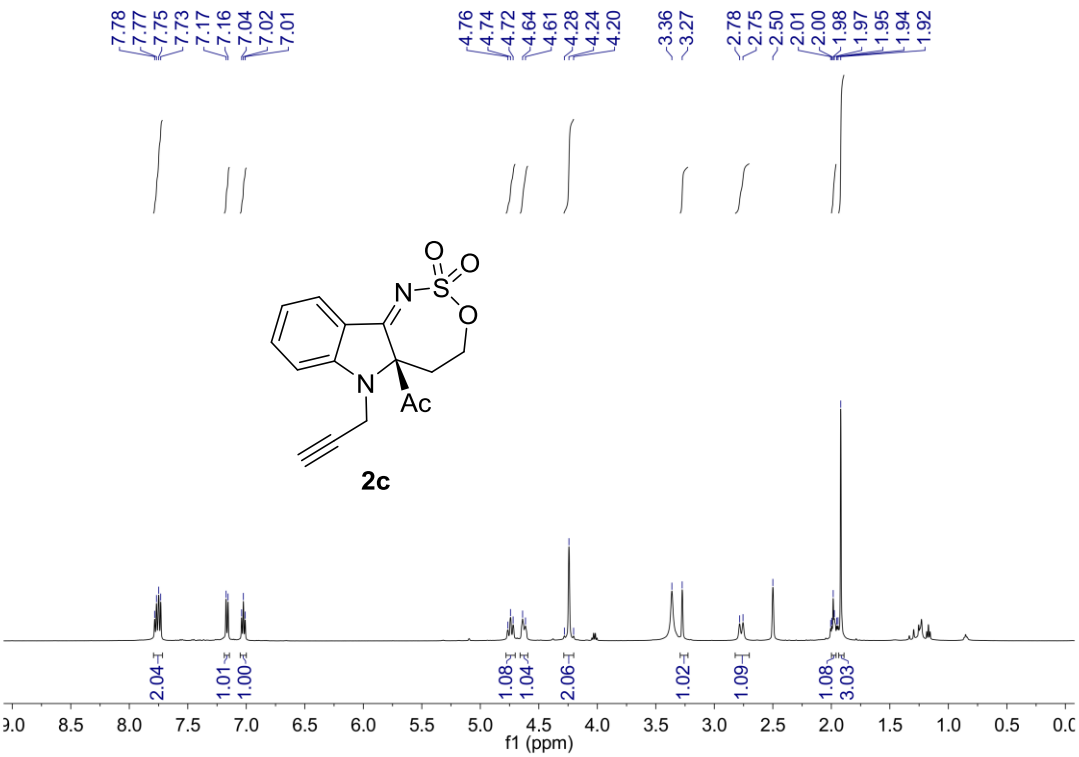
f1 (ppm)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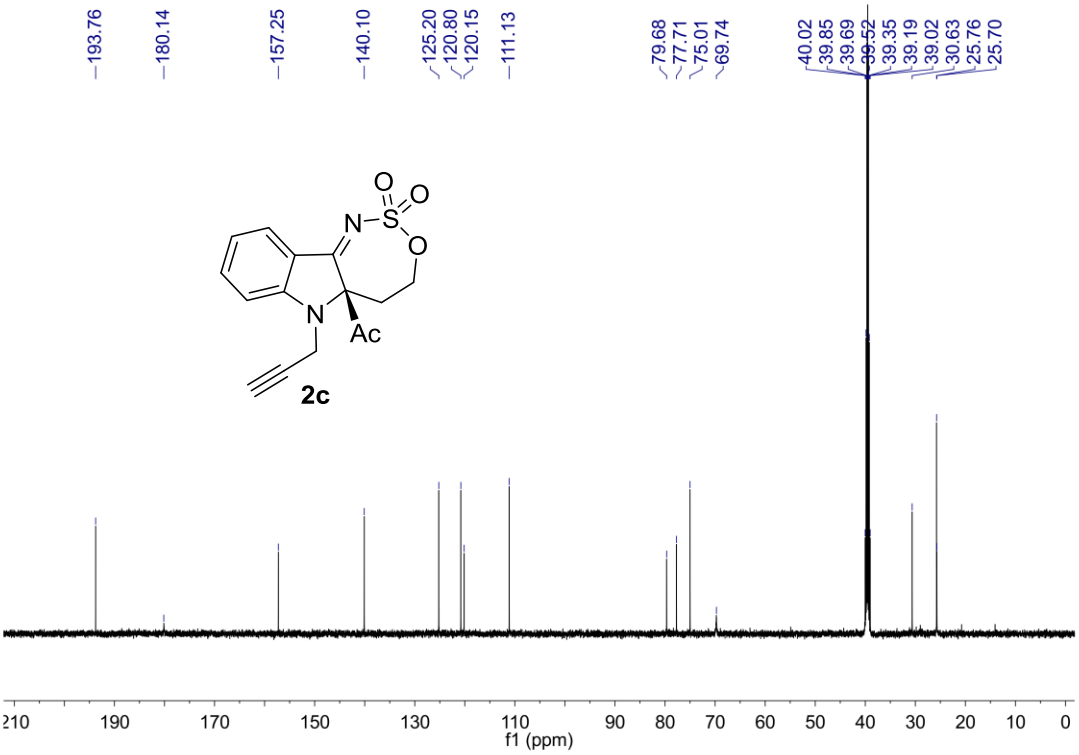
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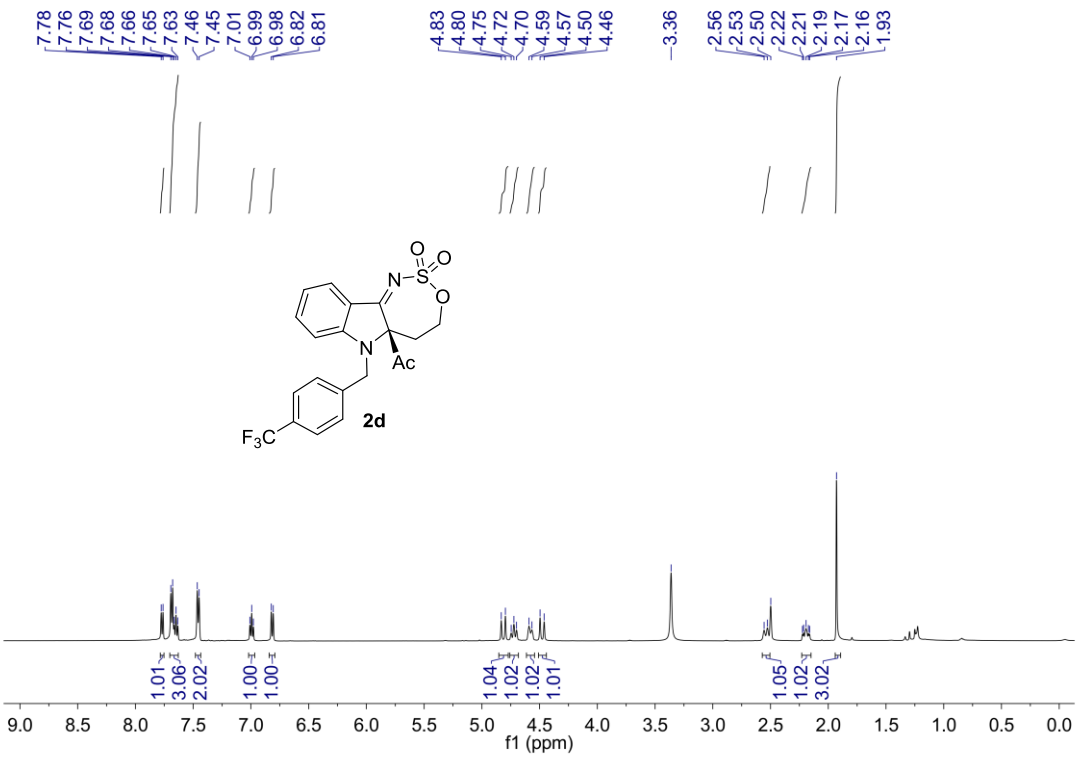
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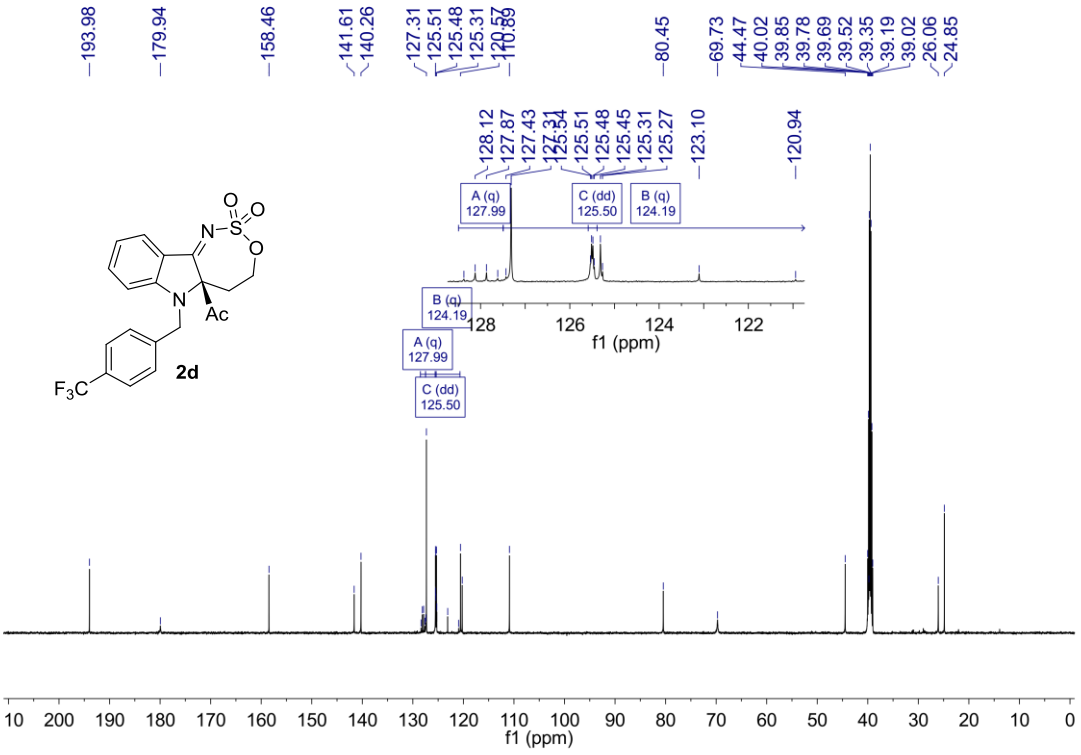
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C (dd)
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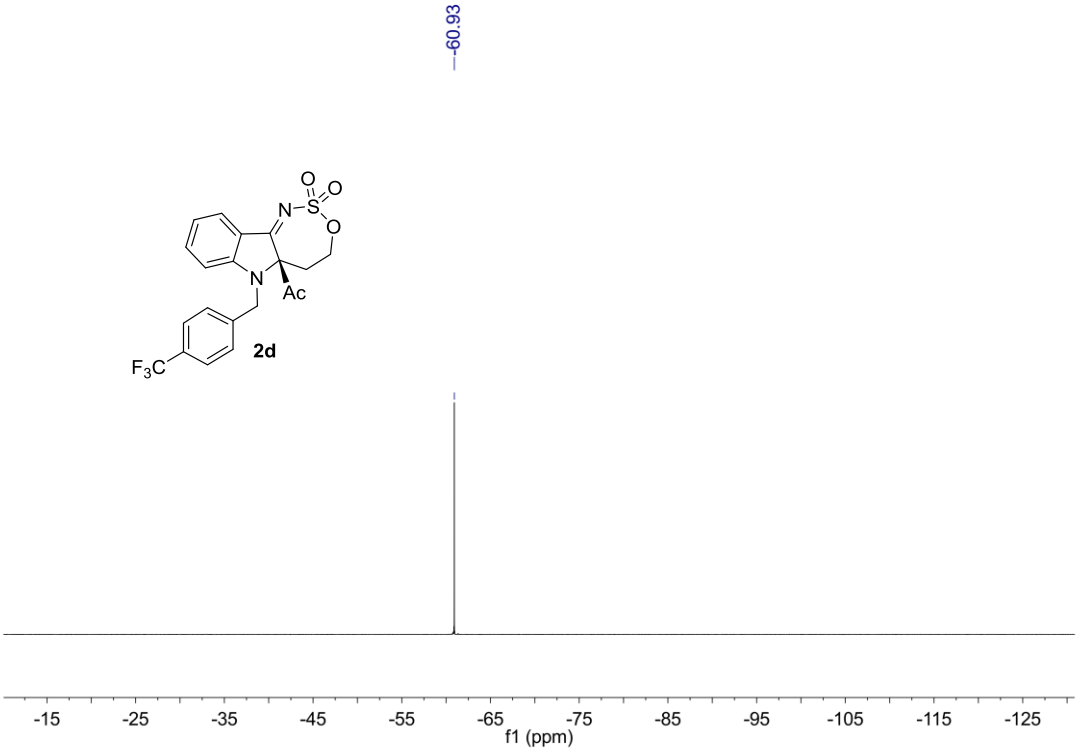
B (q)
124.19
2d
f1 (ppm)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S
O
N
Ac
F3C
2d
-15
-25
-35
-45
-55
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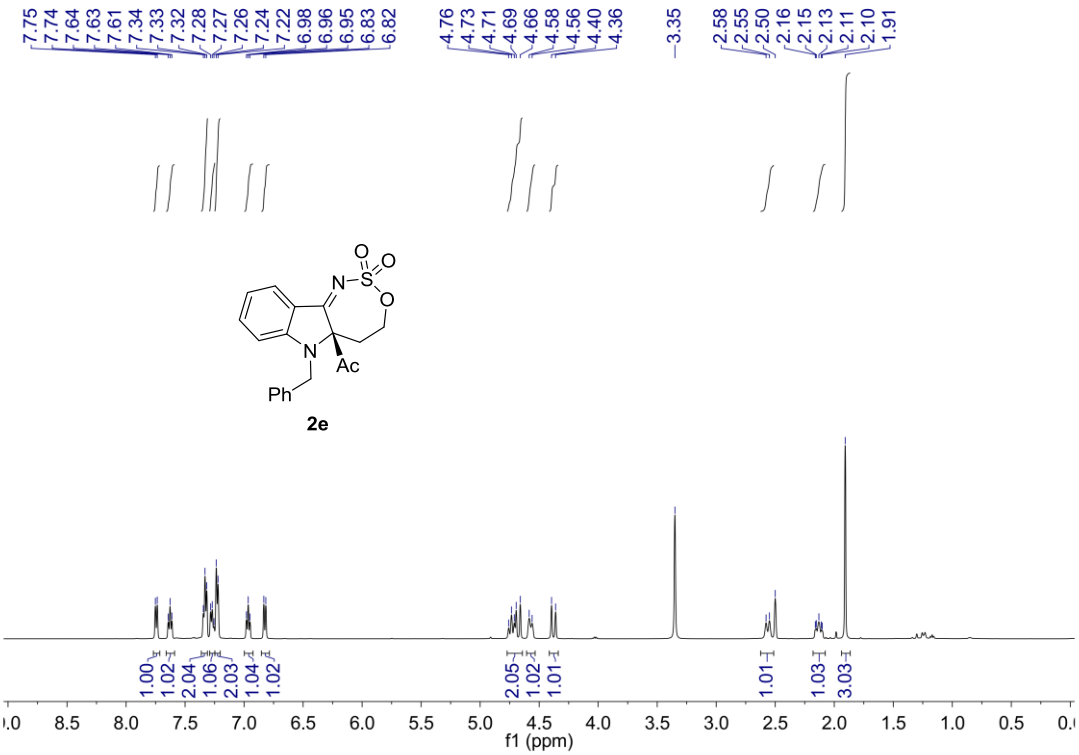
-85
-95
-105
-115
-125
f1 (ppm)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7.22
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6.96
6.95
6.83
6.82
4.76
4.73
4.71
4.69
4.66
4.58
4.56
4.40
4.36
3.35
2.58
2.55
2.50
2.16
2.15
2.13
2.11
2.10
1.91
O
O
N
S
O
N
Ac
Ph
2e
1.00
1.02
2.04
1.06
2.03
1.04
1.02
2.05
1.02
1.01
1.01
1.03
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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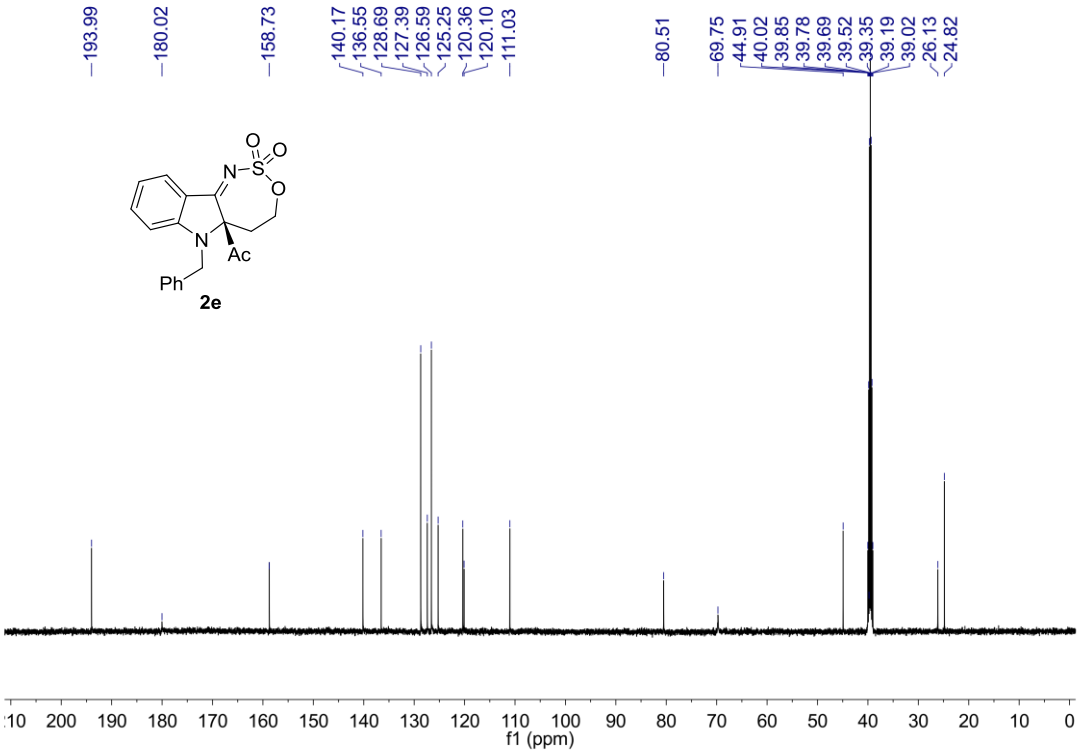
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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128.69
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126.59
125.25
120.36
120.10
111.03
80.51
69.75
44.91
40.02
39.85
39.78
39.69
39.52
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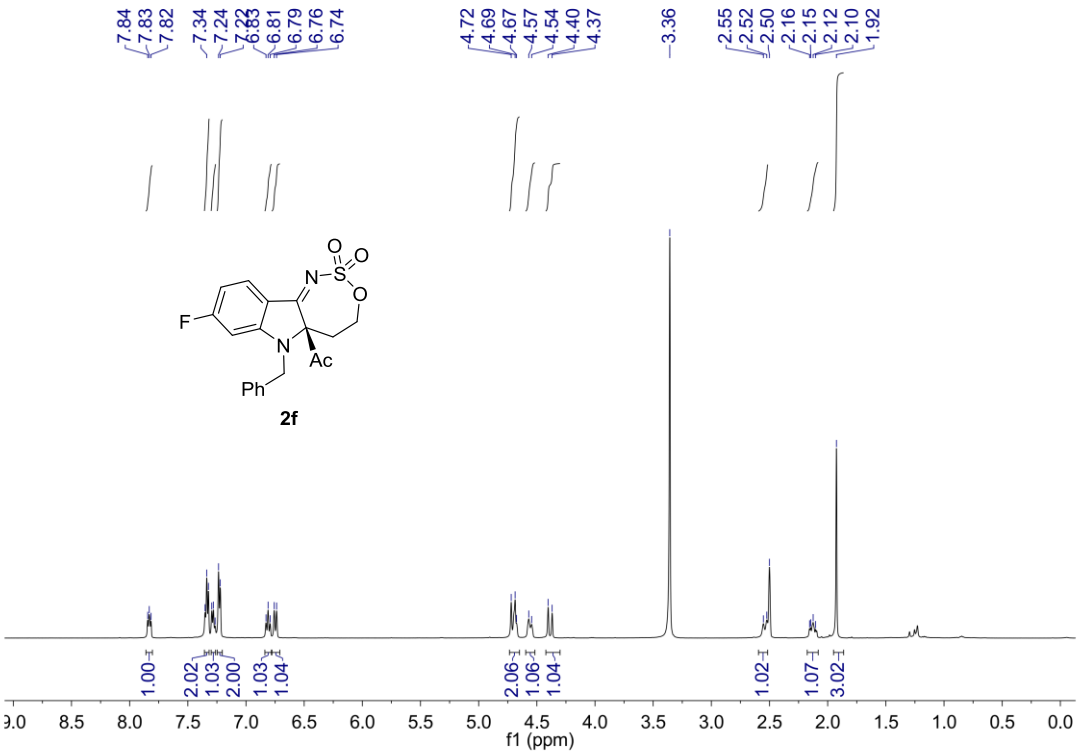
39.02
26.13
24.82
O
O
N
S
O
N
Ac
Ph
2e
f1 (ppm)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6.76
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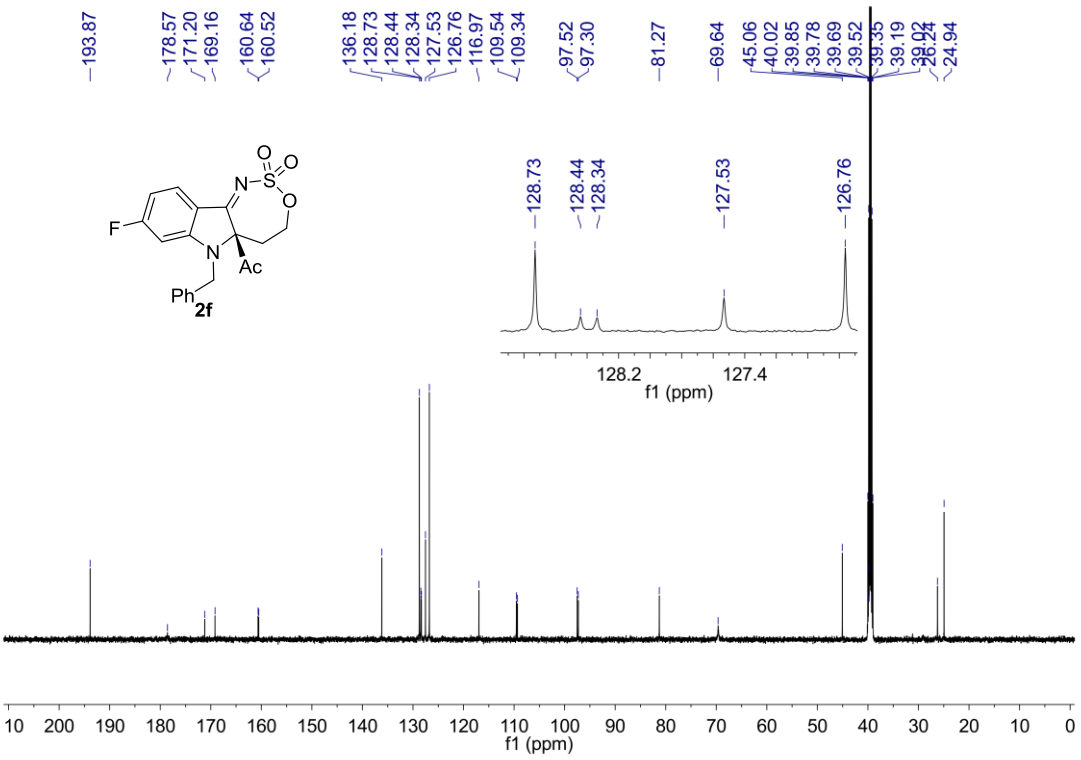
4.69
4.67
4.57
4.54
4.40
4.37
3.36
2.55
2.52
2.50
2.16
2.15
2.12
2.10
1.92
F
O
O
N
S
O
N
Ac
Ph
2f
1.00
2.02
1.03
2.00
1.03
1.04
2.06
1.06
1.04
1.02
1.07
3.02
f1 (ppm)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160.52
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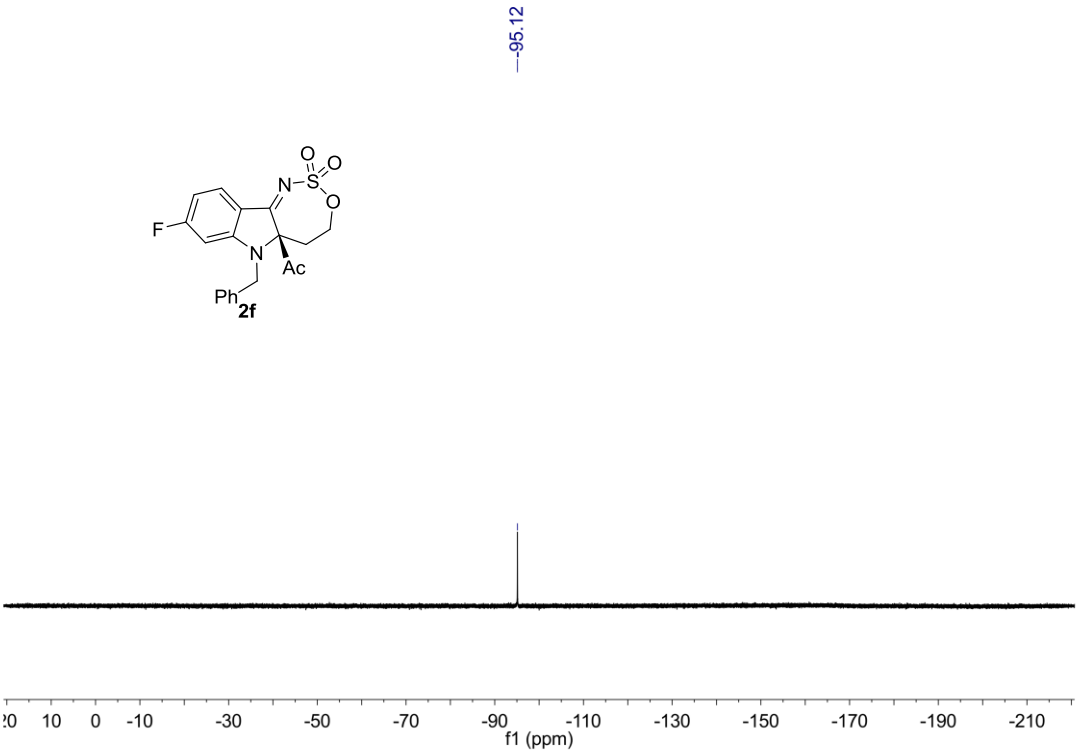
128.34
127.53
126.76
116.97
109.54
109.34
97.52
97.30
81.27
69.64
45.06
40.02
39.85
39.78
39.69
39.52
39.35
39.19
39.02
26.24
24.94
F
N
S
O
O
O
N
Ac
Ph
2f
128.73
128.44
128.34
127.53
126.76
128.2
127.4
f1 (ppm)
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


95.12
F
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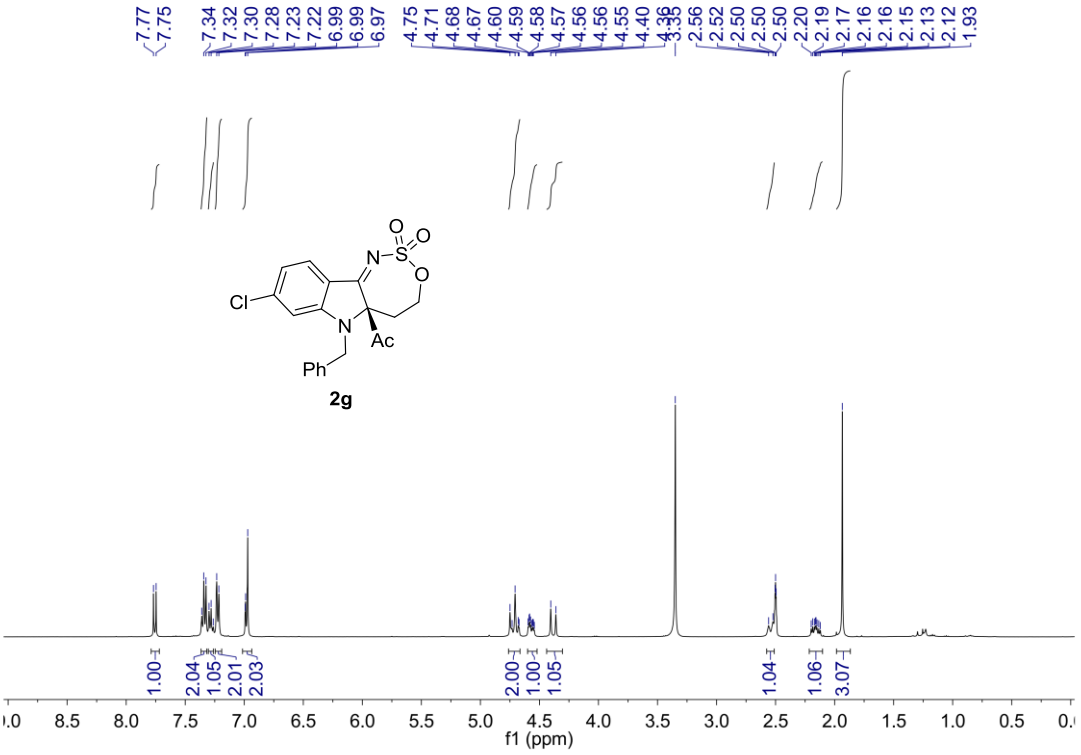
O
N
Ac
Ph
2f
20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)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6.99
6.97
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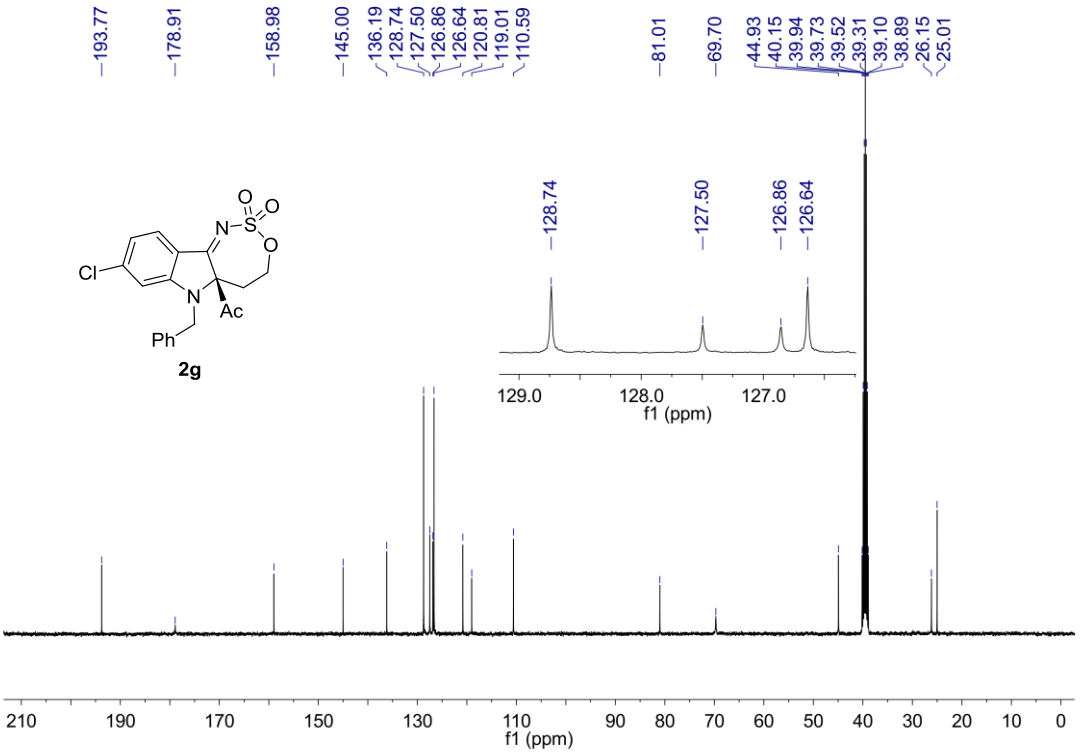
4.68
4.67
4.60
4.59
4.58
4.57
4.56
4.56
4.55
4.40
2.56
2.52
2.50
2.50
2.50
2.20
2.19
2.17
2.16
2.16
2.15
2.13
2.12
1.93
Cl
N
S
O
O
O
N
Ac
Ph
2g
1.00
2.04
1.05
2.01
2.03
2.00
1.00
1.05
1.04
1.06
3.07
f1 (ppm)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128.74
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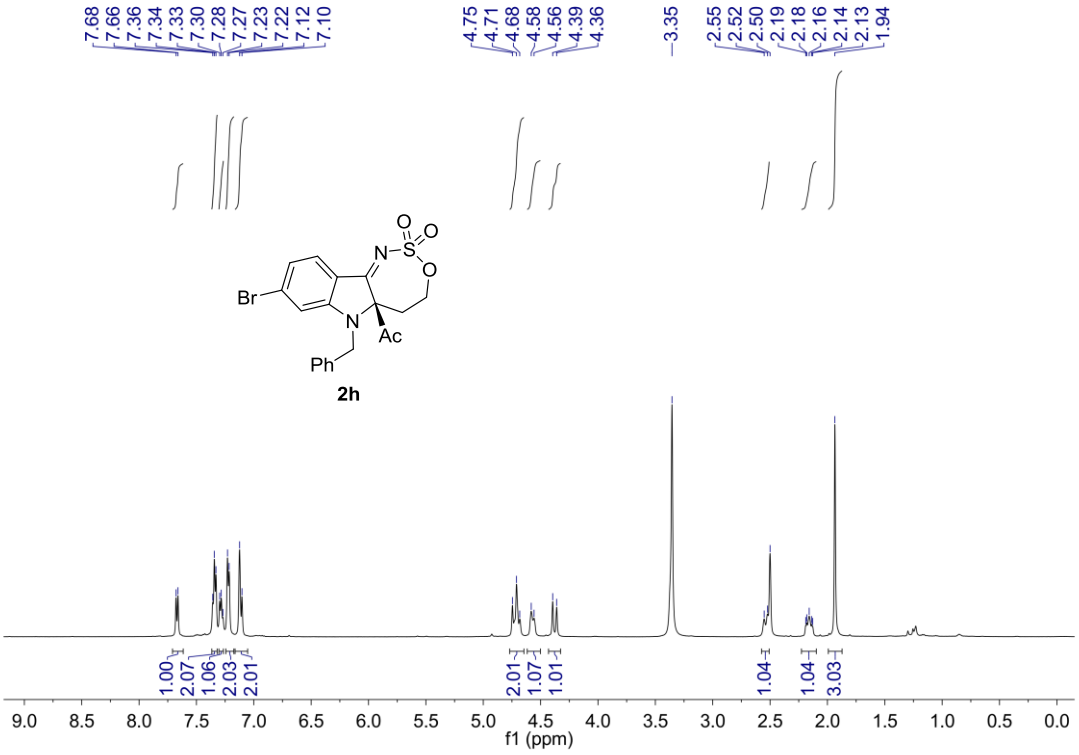
126.86
126.64
120.81
119.01
110.59
81.01
69.70
44.93
40.15
39.94
39.73
39.52
39.31
39.10
38.89
26.15
25.01
128.74
127.50
126.86
126.64
Cl
N
S
O
O
O
N
Ac
Ph
2g
f1 (ppm)
f1 (ppm)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7.30
7.28
7.27
7.23
7.22
7.12
7.10
4.75
4.71
4.68
4.58
4.56
4.39
4.36
3.35
2.55
2.52
2.50
2.19
2.18
2.16
2.14
2.13
1.94
Br
N
S
O
O
O
N
Ac
Ph
2h
1.00
2.07
1.06
2.03
2.01
2.01
1.07
1.01
1.04
1.04
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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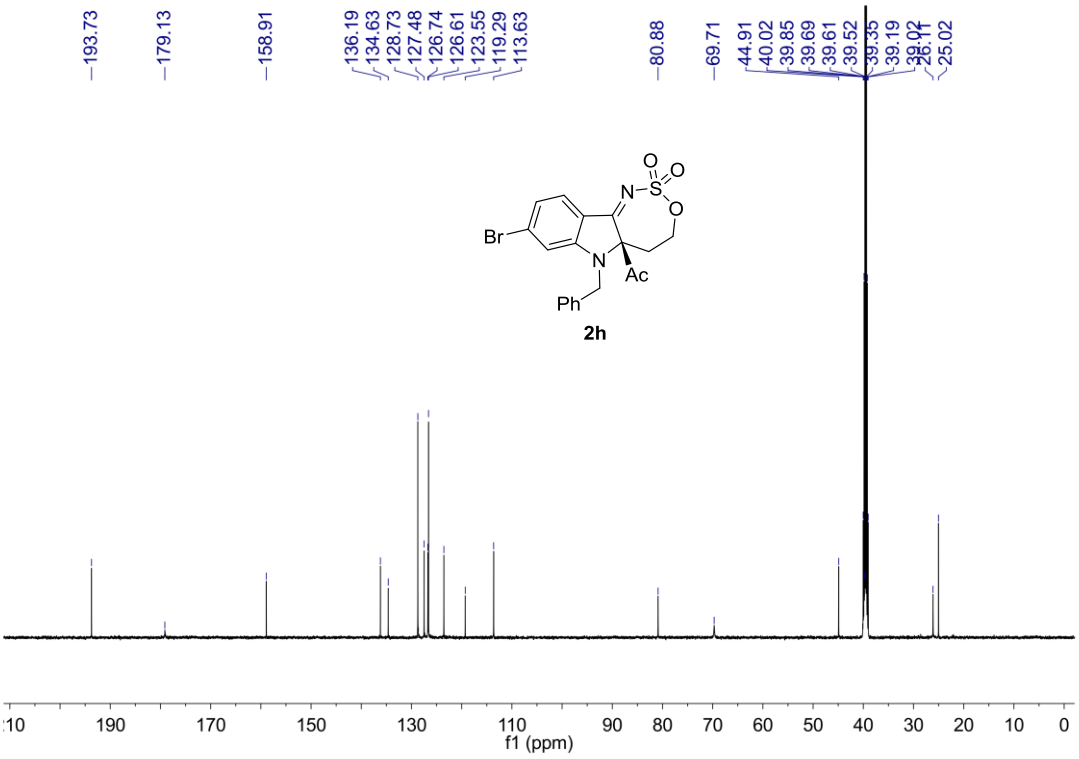
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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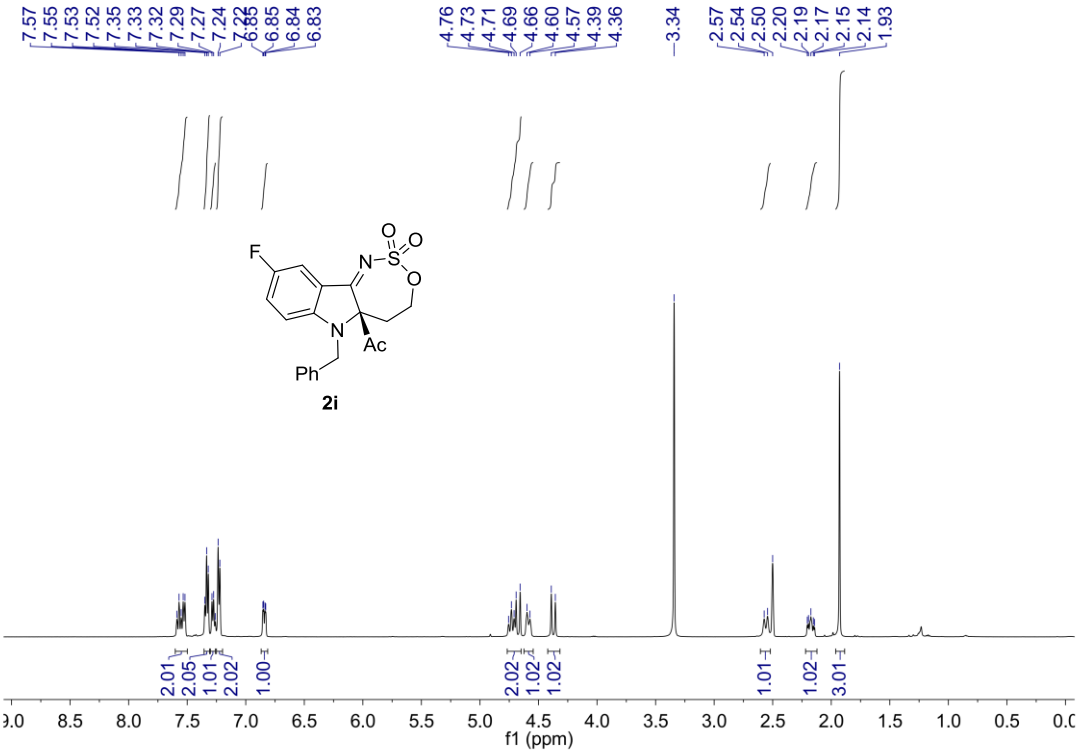
126.74
126.61
123.55
119.29
113.63
80.88
69.71
44.91
40.02
39.85
39.69
39.61
39.52
39.35
39.19
25.02
Br
N
S
O
O
O
N
Ac
Ph
2h
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.83
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4.73
4.71
4.69
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4.57
4.39
4.36
3.34
2.57
2.54
2.50
2.20
2.19
2.17
2.15
2.14
1.93
F
O
O
N
S
O
N
Ac
Ph
2i
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2.05
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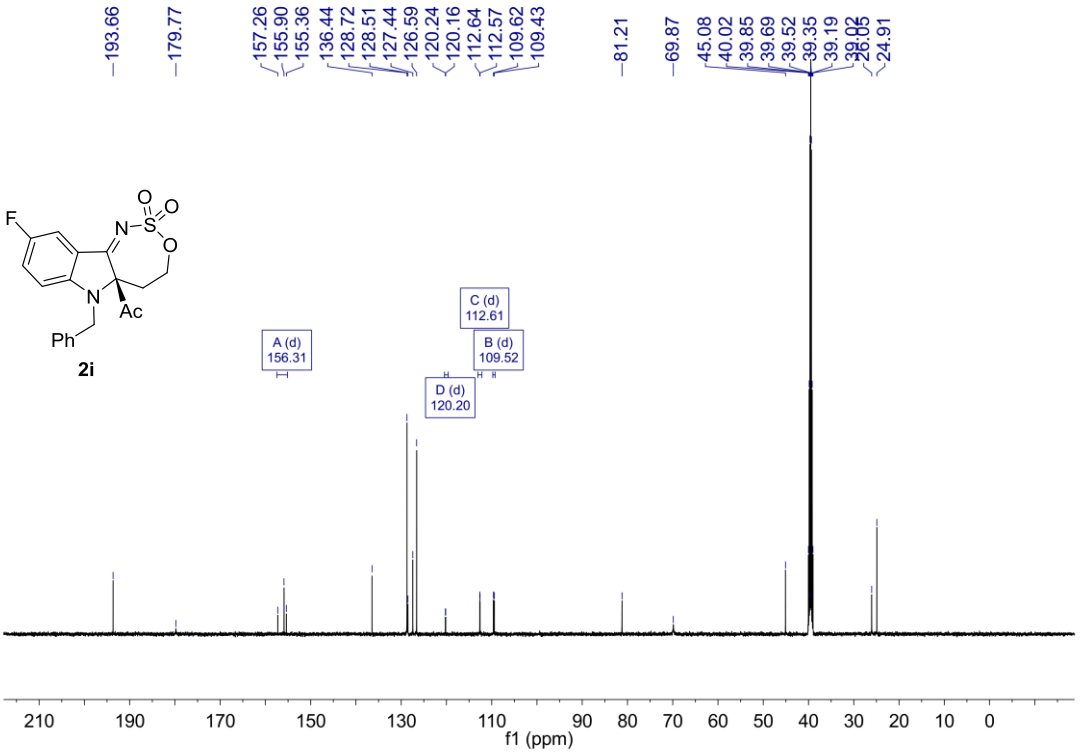
2.02
1.00
2.02
1.02
1.02
1.01
1.02
3.01
f1 (ppm)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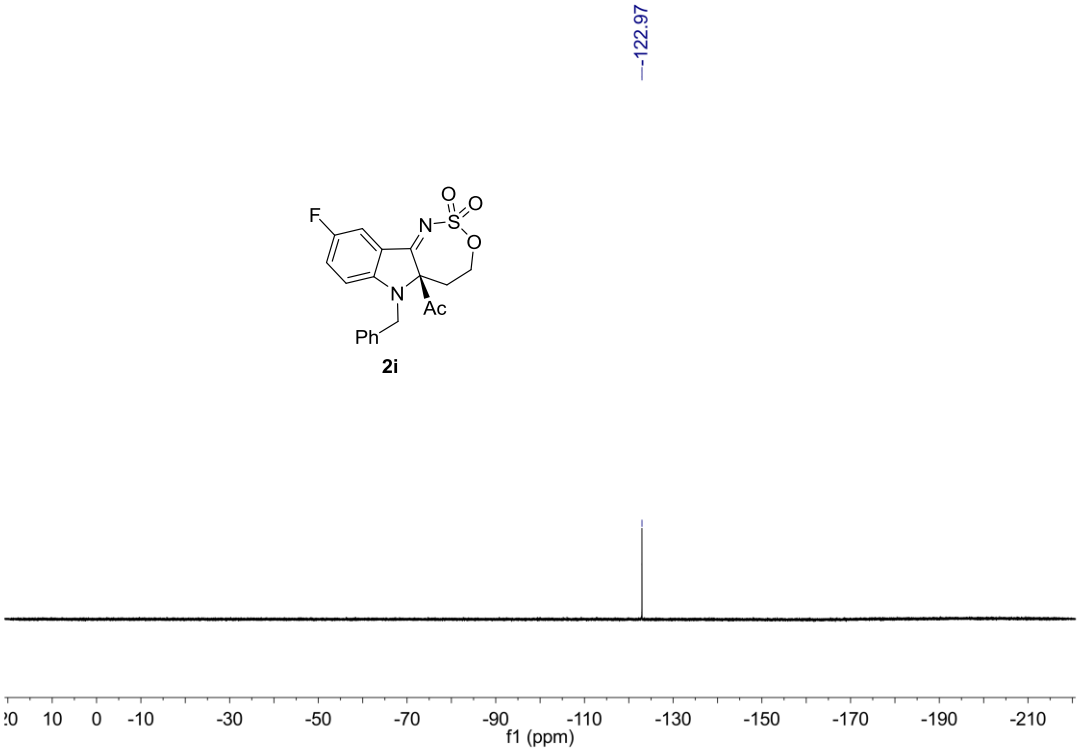
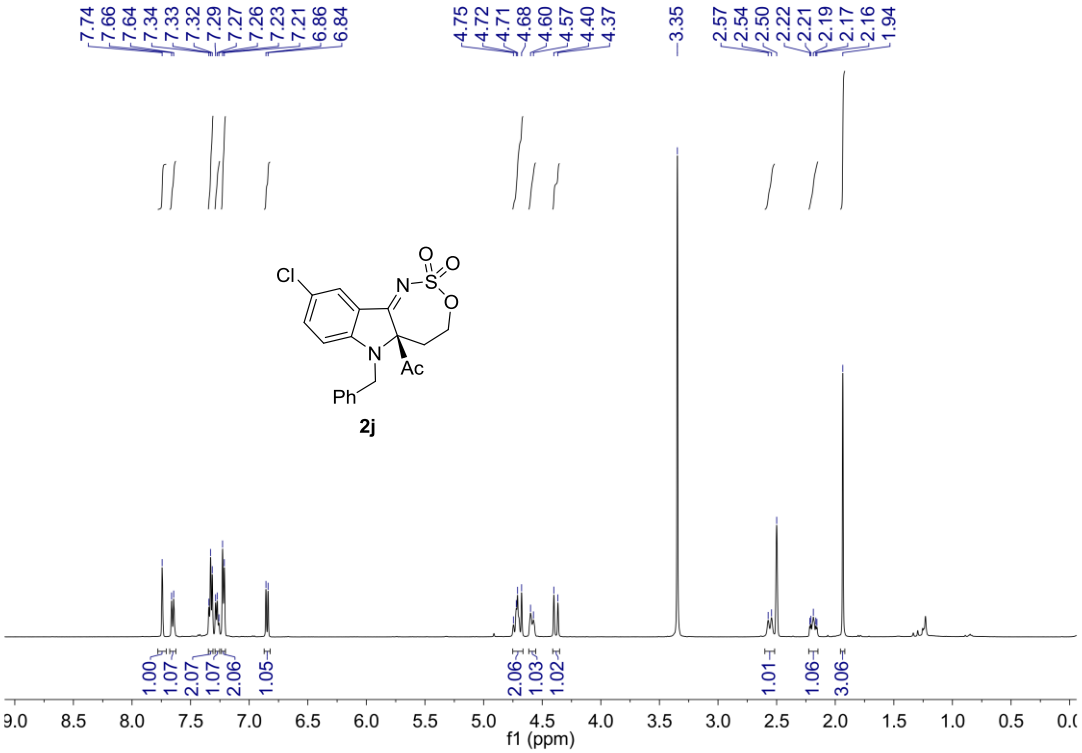
120.16
112.64
112.57
109.62
109.43
81.21
69.87
45.08
40.02
39.85
39.69
39.52
39.35
39.19
24.91
F
O
O
N
S
O
N
Ac
Ph
2i
A (d)
156.31
C (d)
112.61
B (d)
109.52
D (d)
120.20
f1 (ppm)


-122.97
F
O
O
N
S
O
N
Ac
Ph
2i
f1 (ppm)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4.72
4.71
4.68
4.60
4.57
4.40
4.37
3.35
2.57
2.54
2.50
2.22
2.21
2.19
2.17
2.16
1.94
Cl
O
O
N
S
O
N
Ac
Ph
2j
1.00
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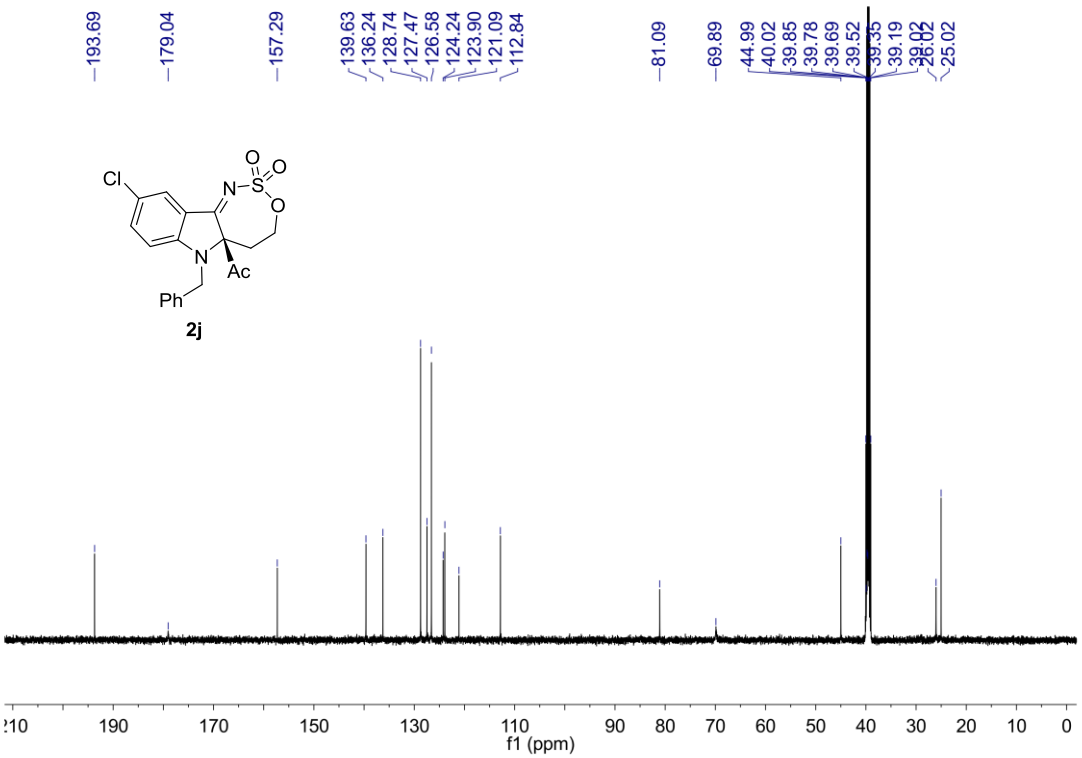
2.06
1.05
2.06
1.03
1.02
1.01
1.06
3.06
f1 (ppm)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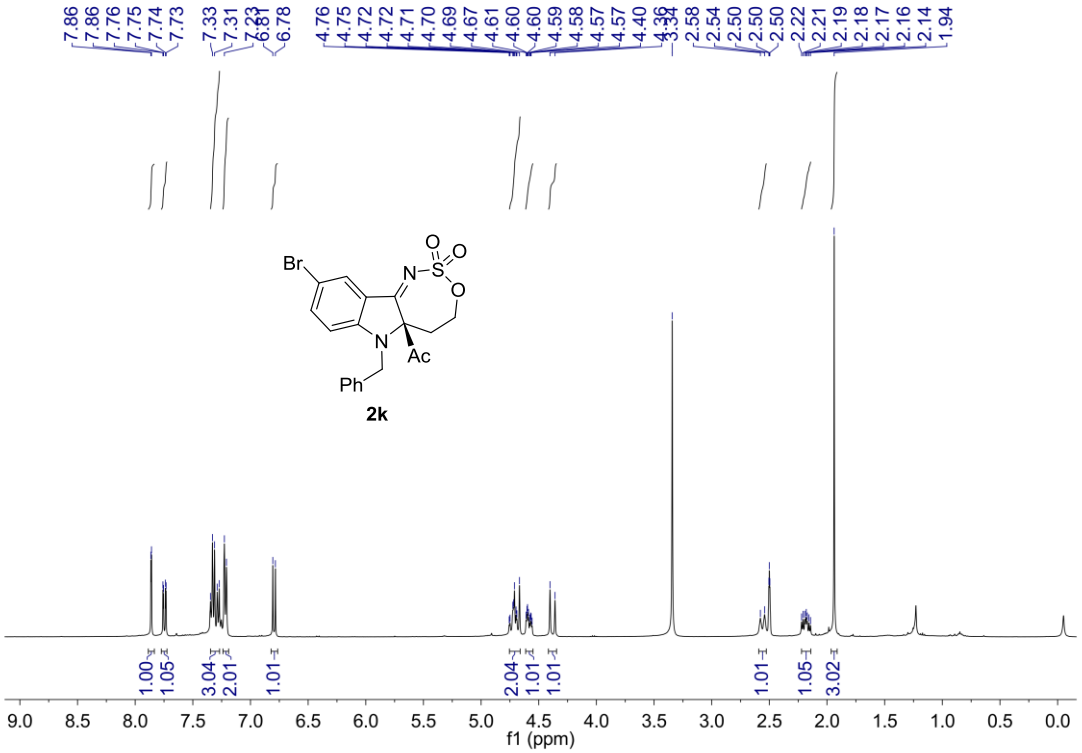
124.24
123.90
121.09
112.84
81.09
69.89
44.99
40.02
39.85
39.78
39.69
39.52
39.35
39.19
39.02
26.02
25.02
Cl
O
O
N
S
O
N
Ac
Ph
2j
f1 (ppm)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4.71
4.70
4.69
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4.60
4.60
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4.58
4.57
4.57
4.40
2.58
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2.21
2.19
2.18
2.17
2.16
2.14
1.94
Br
O
O
N
S
O
N
Ac
Ph
2k
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1.01
2.04
1.01
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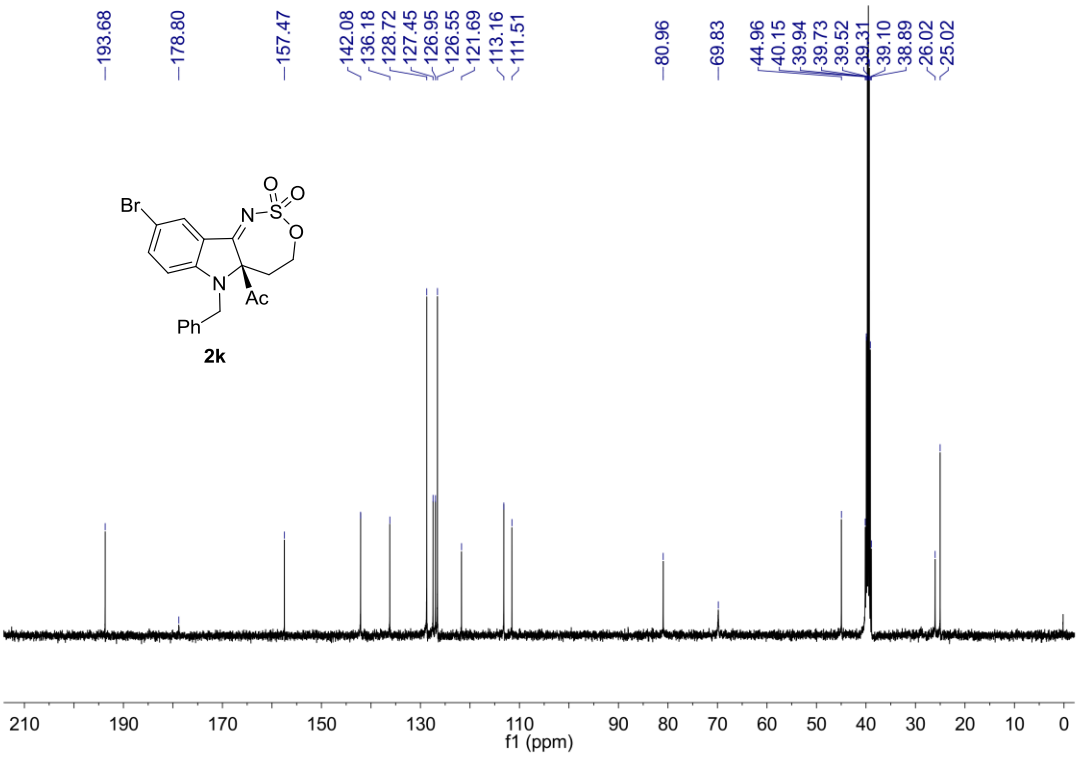
1.01
1.05
3.02
f1 (ppm)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113.16
111.51
80.96
69.83
44.96
40.15
39.94
39.73
39.52
39.31
39.10
38.89
26.02
25.02
Br
O
O
N
S
O
N
Ac
Ph
2k
210
190
170
150
130
110
90
80
70
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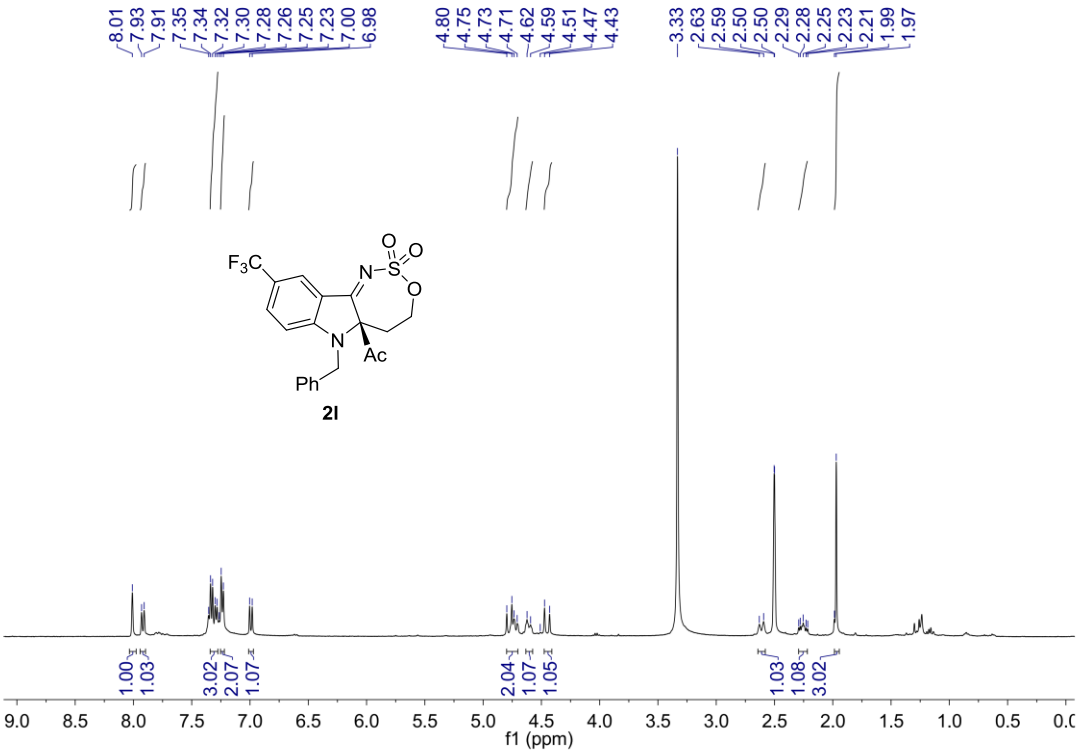
50
40
30
20
10
0
f1 (ppm)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7.23
7.00
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4.75
4.73
4.71
4.62
4.59
4.51
4.47
4.43
3.33
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2.59
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2.28
2.25
2.23
2.21
1.99
1.97
F3C
O
O
N
S
O
N
Ac
Ph
2l
1.00
1.03
3.02
2.07
1.07
2.04
1.07
1.05
1.03
1.08
3.02
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8.0
7.5
7.0
6.5
6.0
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3.0
2.5
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1.5
1.0
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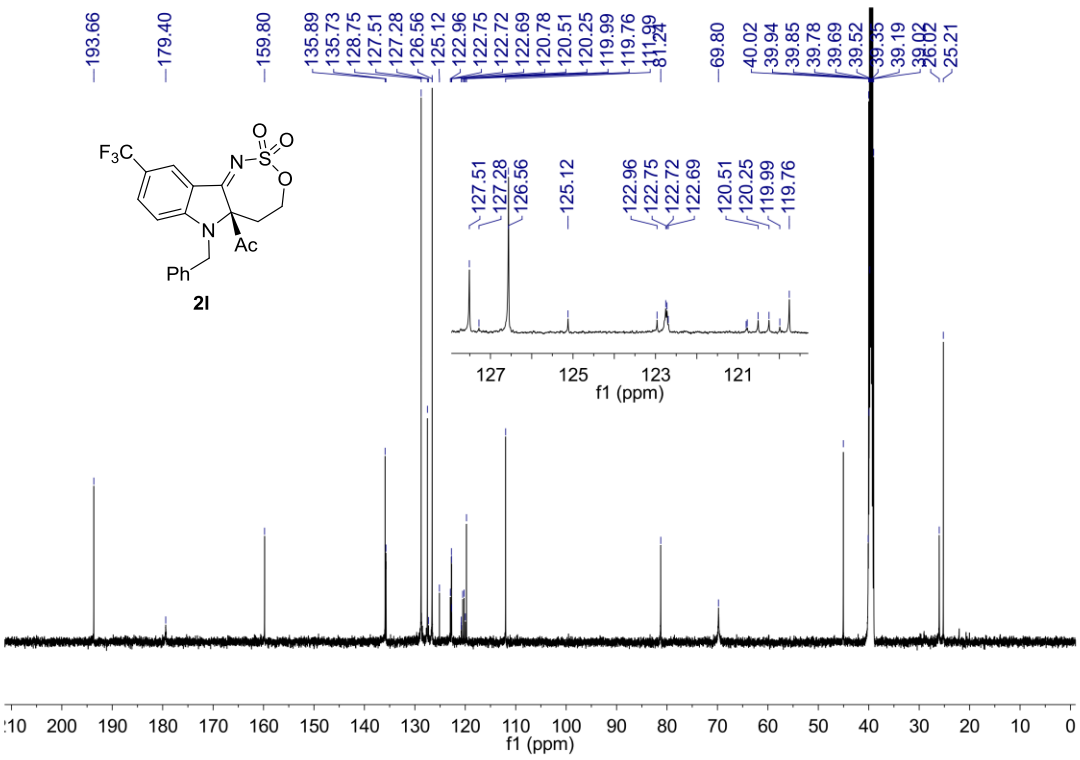
f1 (ppm)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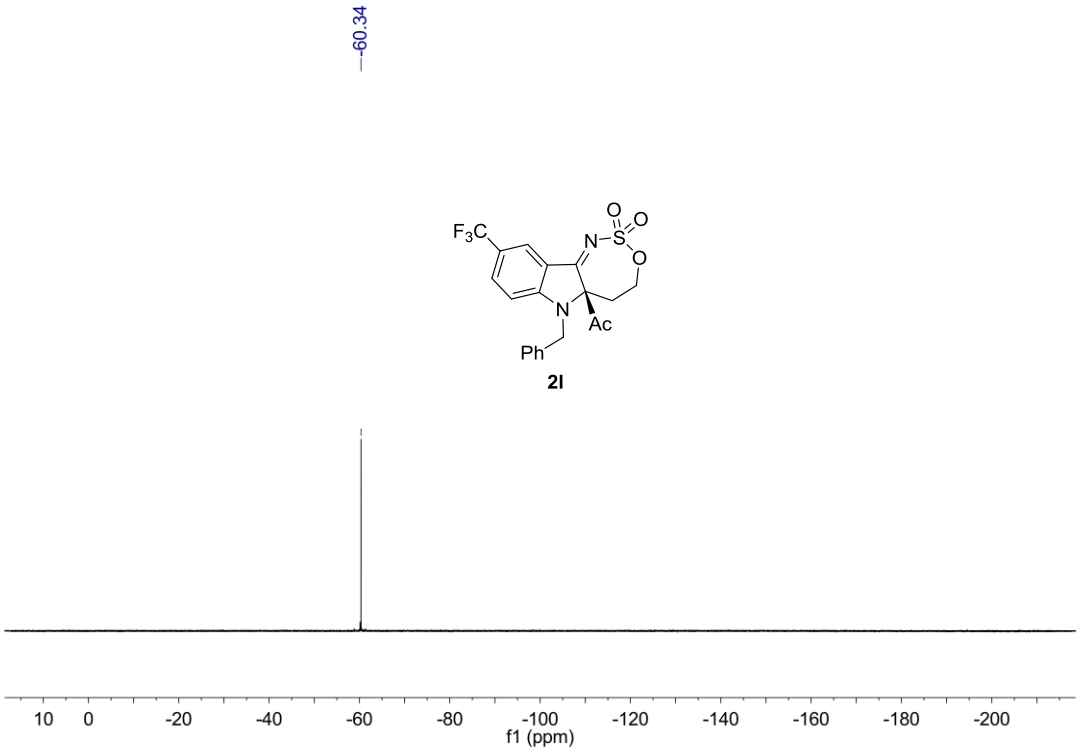
122.72
122.69
120.78
120.51
120.25
119.99
119.76
111.99
81.24
69.80
40.02
39.94
39.85
39.78
39.69
39.52
39.35
39.19
39.02
26.02
25.21
F3C
N
S
O
O
O
N
Ac
Ph
2l
127.51
127.28
126.56
125.12
122.96
122.75
122.72
122.69
120.51
120.25
119.99
119.76
127
125
123
121
f1 (ppm)
f1 (ppm)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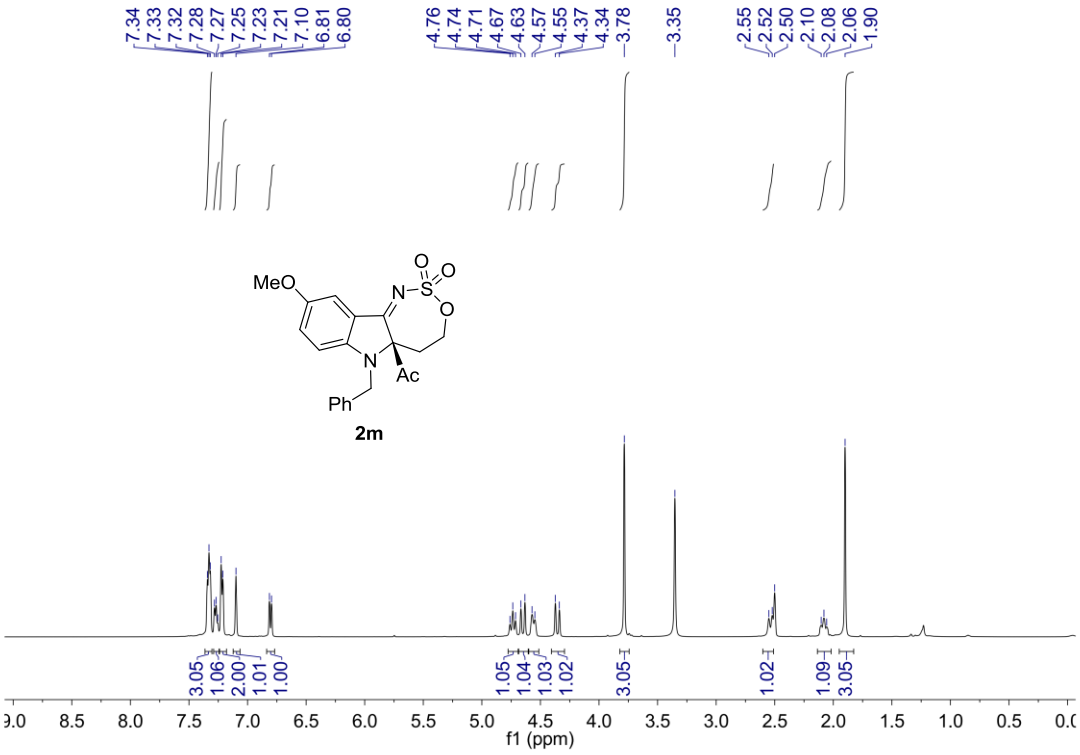
O
O
N
Ac
Ph
2l
f1 (ppm)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6.80
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4.74
4.71
4.67
4.63
4.57
4.55
4.37
4.34
3.78
3.35
2.55
2.52
2.50
2.10
2.08
2.06
1.90
MeO
N
S
O
O
O
N
Ac
Ph
2m
3.05
1.06
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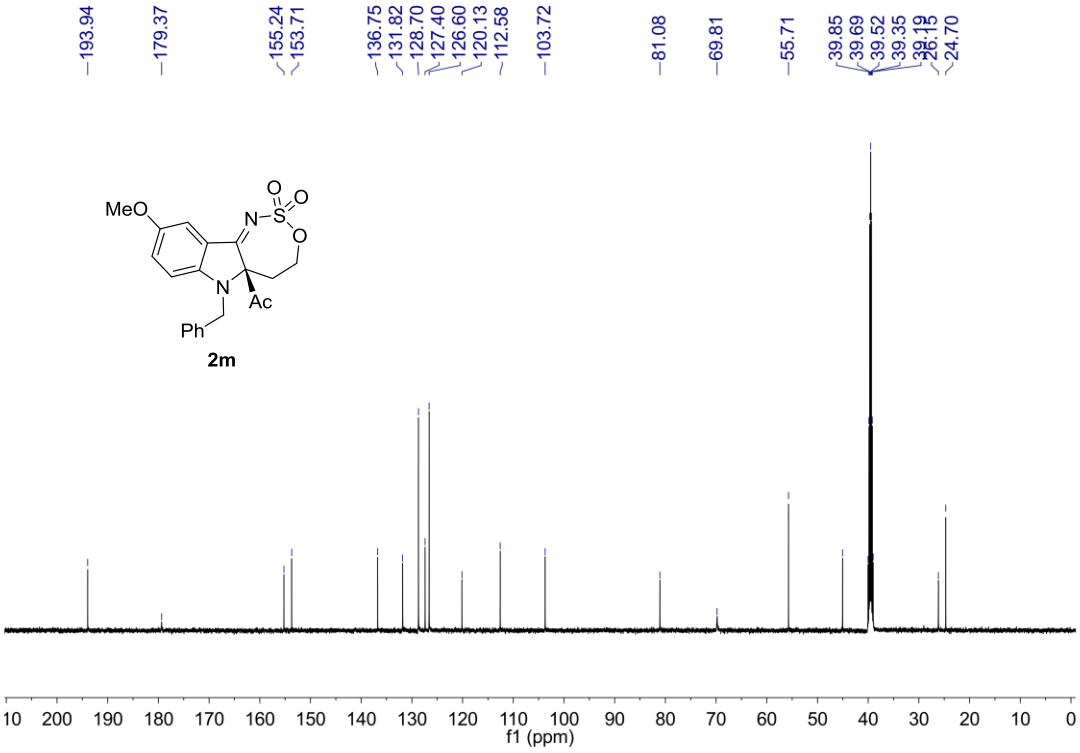
1.05
1.04
1.03
1.02
3.05
1.02
1.09
3.05
f1 (ppm)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81.08
69.81
55.71
39.85
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24.70
MeO
N
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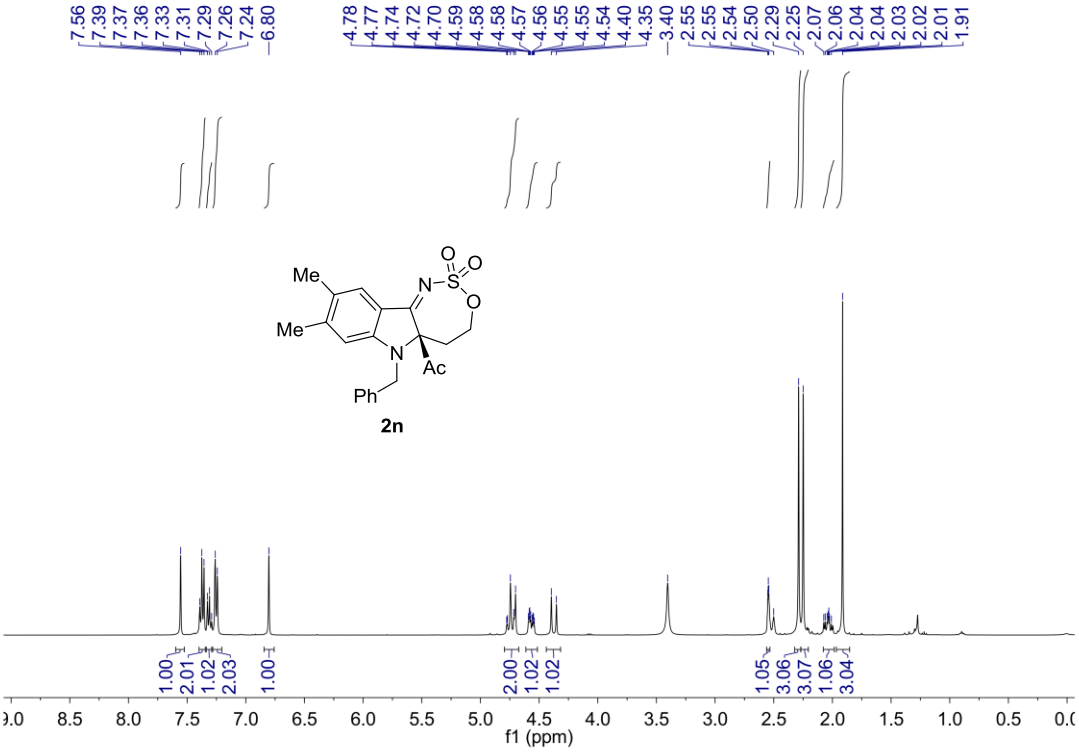
O
O
N
Ac
Ph
2m
f1 (ppm)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4.56
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4.35
3.40
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2.54
2.50
2.29
2.25
2.07
2.06
2.04
2.04
2.03
2.02
2.01
1.91
Me
Me
N
S
O
O
O
N
Ac
Ph
2n
1.00
2.01
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1.02
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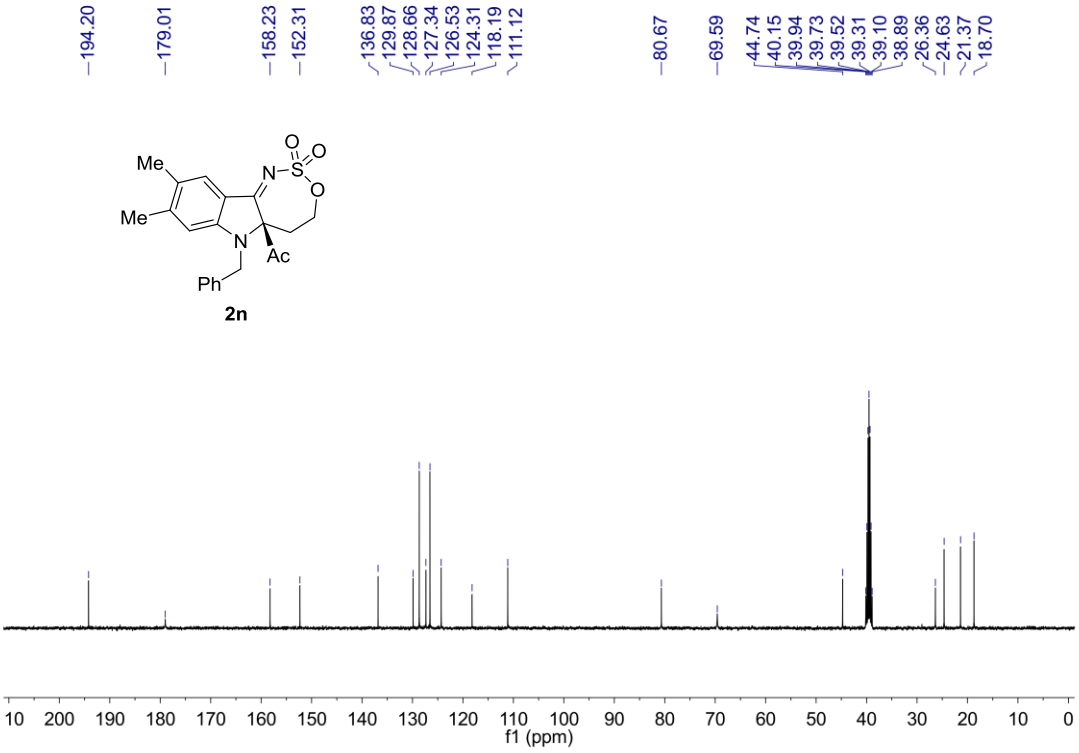
3.06
3.07
1.06
3.04
f1 (ppm)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129.87
128.66
127.34
126.53
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111.12
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69.59
44.74
40.15
39.94
39.73
39.52
39.31
39.10
38.89
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Me
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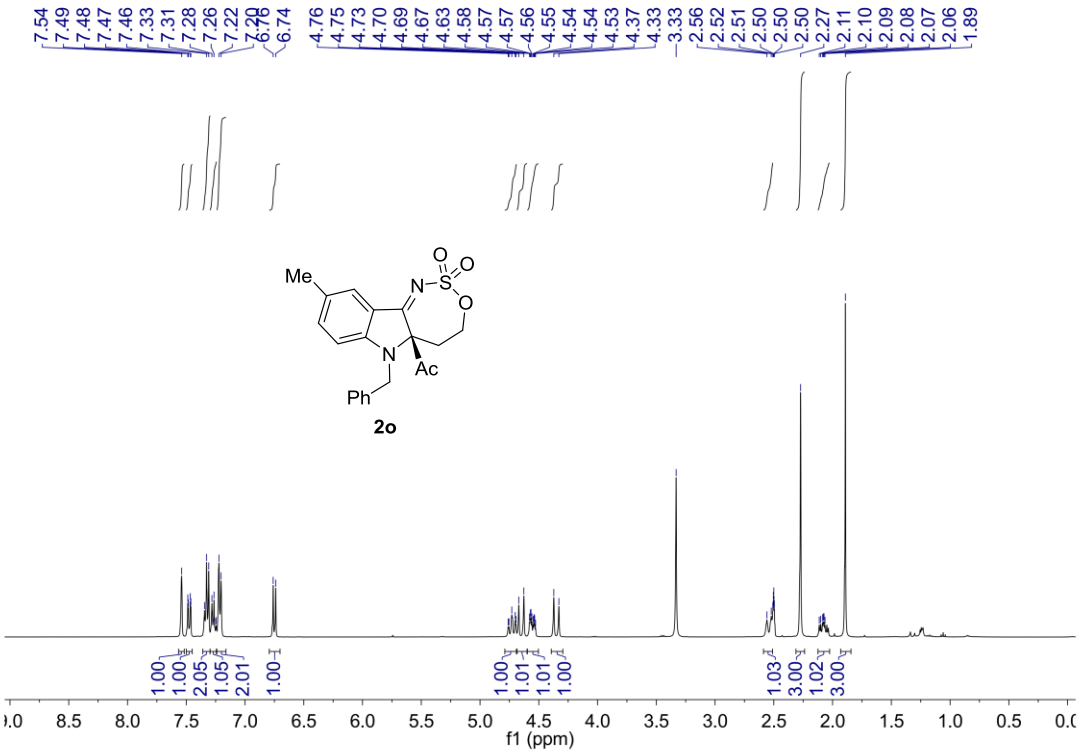
O
N
Ac
Ph
2n
f1 (ppm)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4.76 4.75 4.73 4.70 4.69 4.67 4.63 4.58 4.57 4.57 4.56 4.55 4.54 4.54 4.53 4.37 4.33
3.33
2.56 2.52 2.51 2.50 2.50 2.50 2.27 2.11 2.10 2.09 2.08 2.07 2.06 1.89
Me
N
S
O
O
O
N
Ac
Ph
2o
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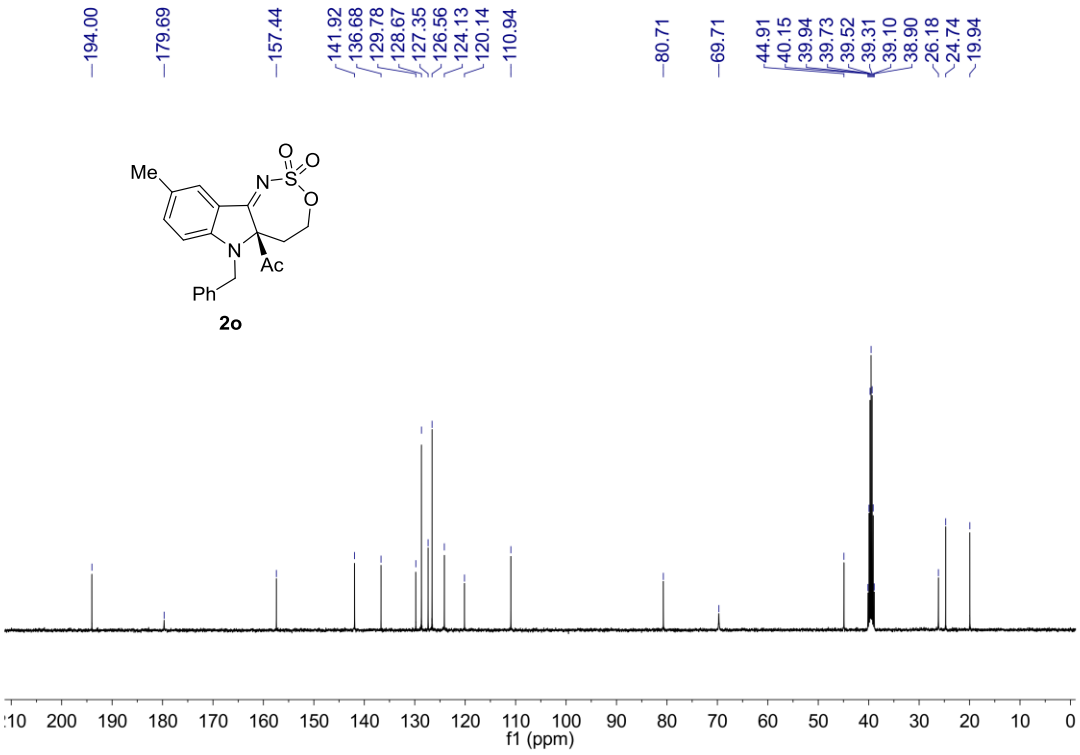
f1 (ppm)


194.00 179.69 157.44 141.92 136.68 129.78 128.67 127.35 126.56 124.13 120.14 110.94 80.71 69.71 44.91 40.15 39.94 39.73 39.52 39.31 39.10 38.90 26.18 24.74 19.94
Me
N
S
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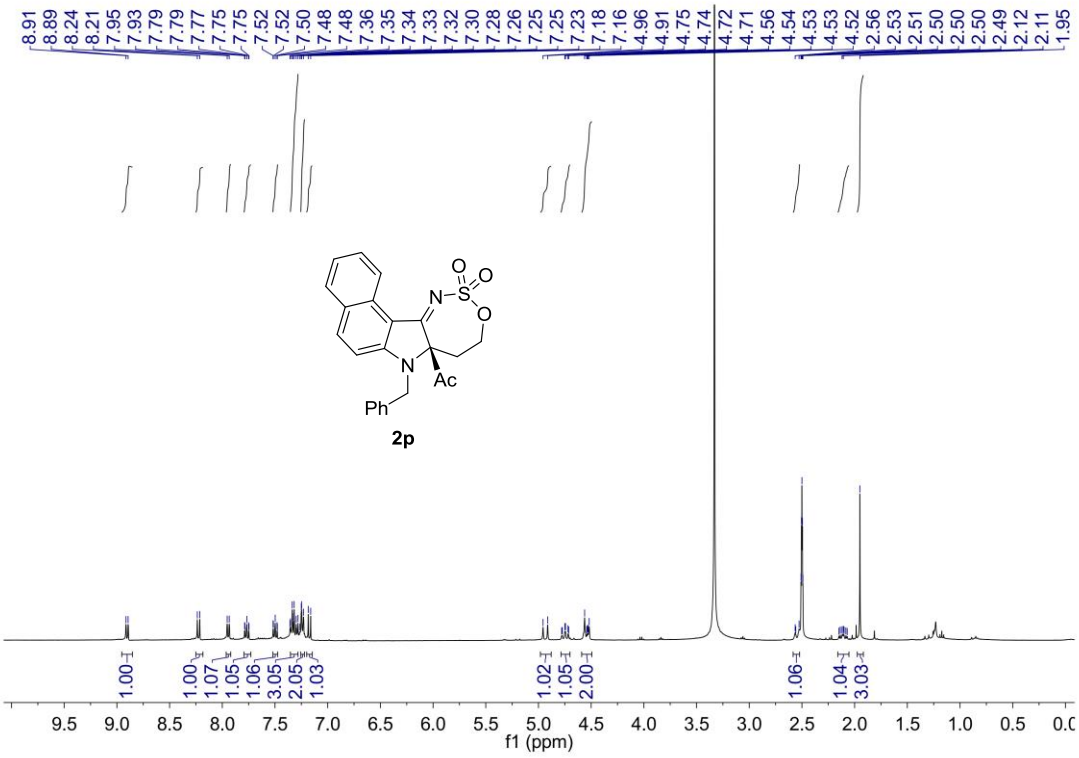
O
O
N
Ac
Ph
2o
f1 (ppm)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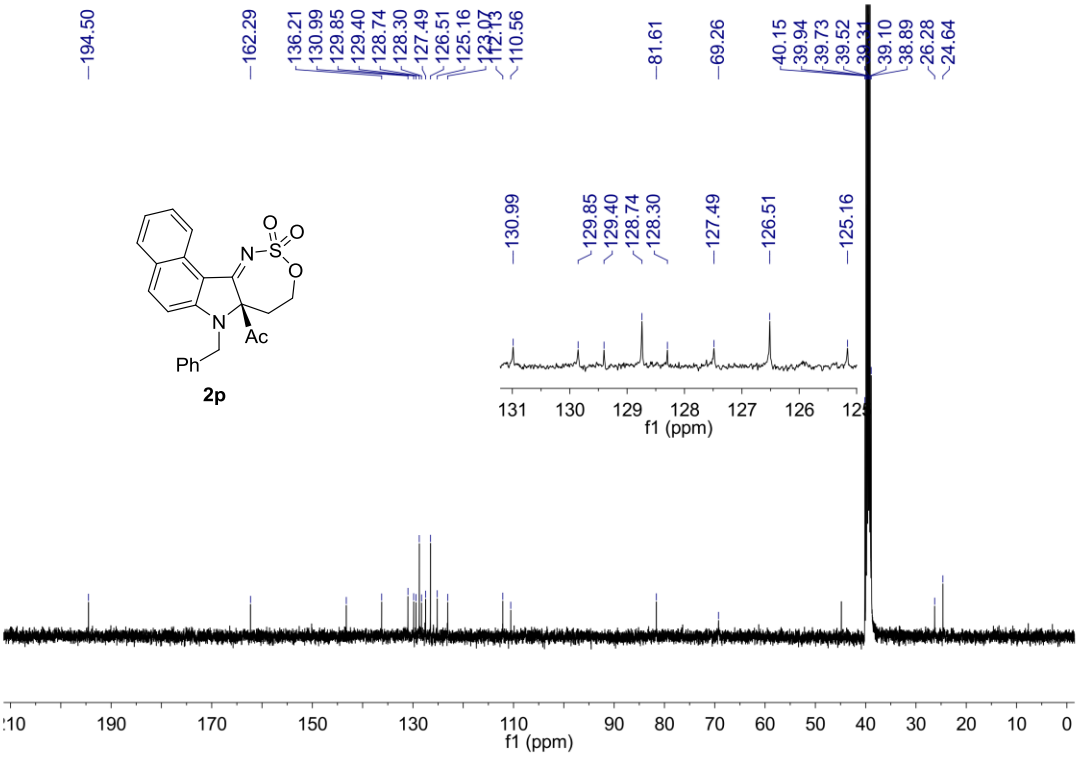
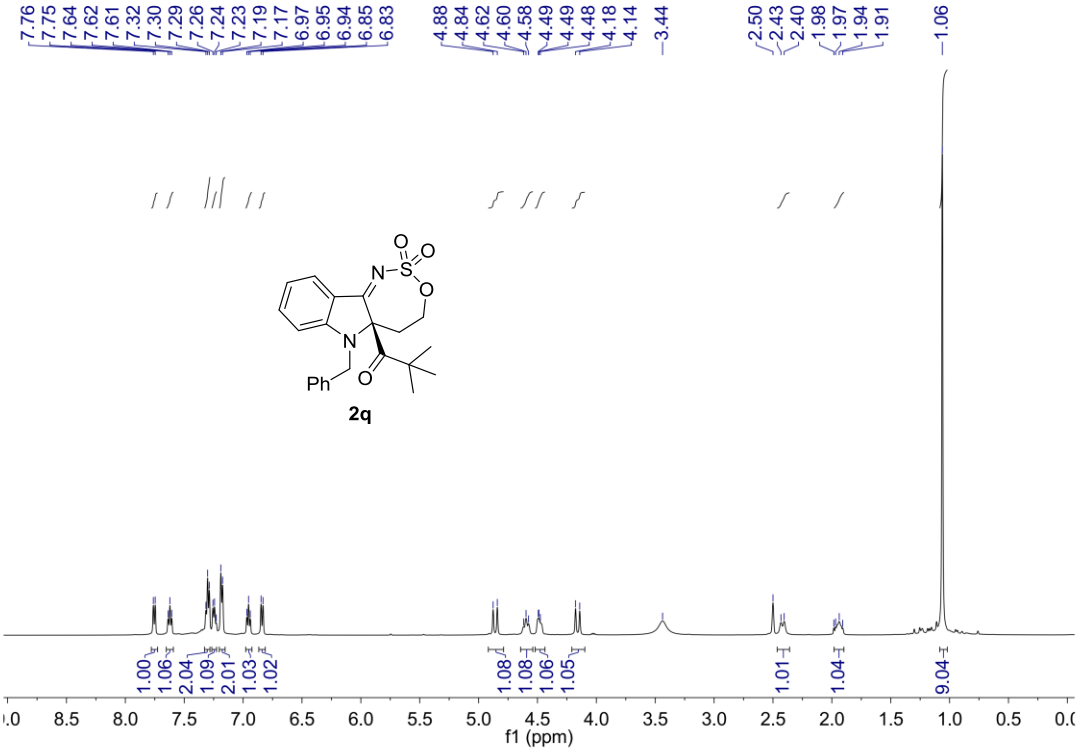
O
O
N
S
O
N
Ac
Ph
2p
1.00 1.00 1.07 1.05 1.06 3.05 2.05 1.03 1.02 1.05 2.00 1.06 1.04 3.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


194.50 162.29 136.21 130.99 129.85 129.40 128.74 128.30 127.49 126.51 125.16 123.07 112.13 110.56 81.61 69.26 40.15 39.94 39.73 39.52 39.10 38.89 26.28 24.64
O
O
N
S
O
N
Ac
Ph
2p
130.99 129.85 129.40 128.74 128.30 127.49 126.51 125.16
131 130 129 128 127 126
f1 (ppm)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.29
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7.24
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7.19
7.17
6.97
6.95
6.94
6.85
6.83
4.88
4.84
4.62
4.60
4.58
4.49
4.49
4.48
4.18
4.14
3.44
2.50
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1.91
1.06
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1.06
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1.09
2.01
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1.08
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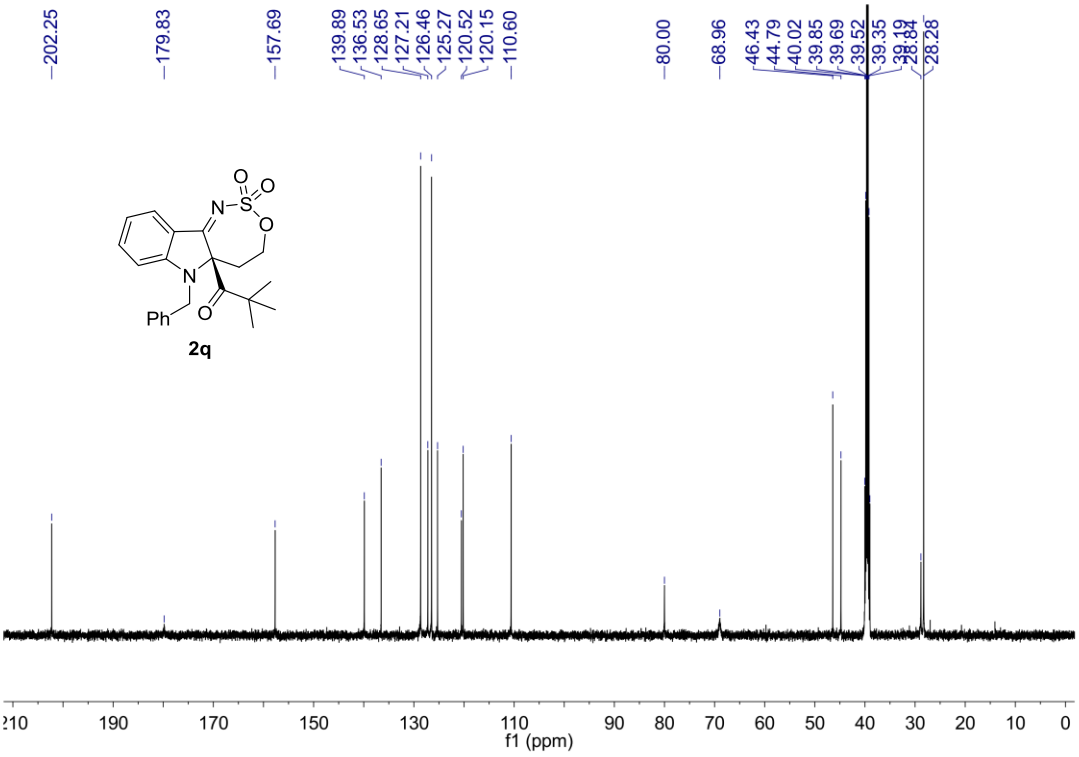
1.05
1.01
1.04
9.04
f1 (ppm)
Ph
2q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136.53
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127.21
126.46
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68.96
46.43
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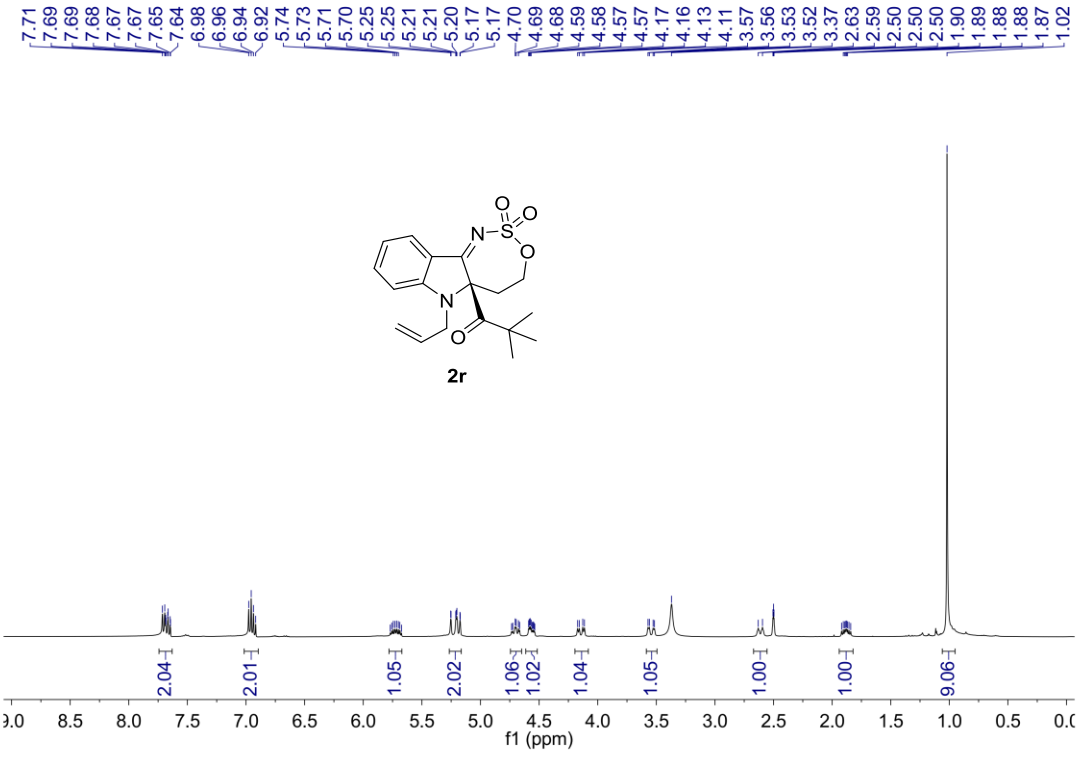
39.19
28.84
28.28
f1 (ppm)
Ph
2q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7.67
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7.64
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6.96
6.94
6.92
5.74
5.73
5.71
5.70
5.25
5.21
5.21
5.20
5.17
5.17
4.70
4.69
4.68
4.59
4.58
4.57
4.57
4.17
4.16
4.13
4.11
3.57
3.56
3.53
3.52
3.37
2.63
2.59
2.50
2.50
2.50
1.90
1.89
1.88
1.88
1.87
1.02
2r
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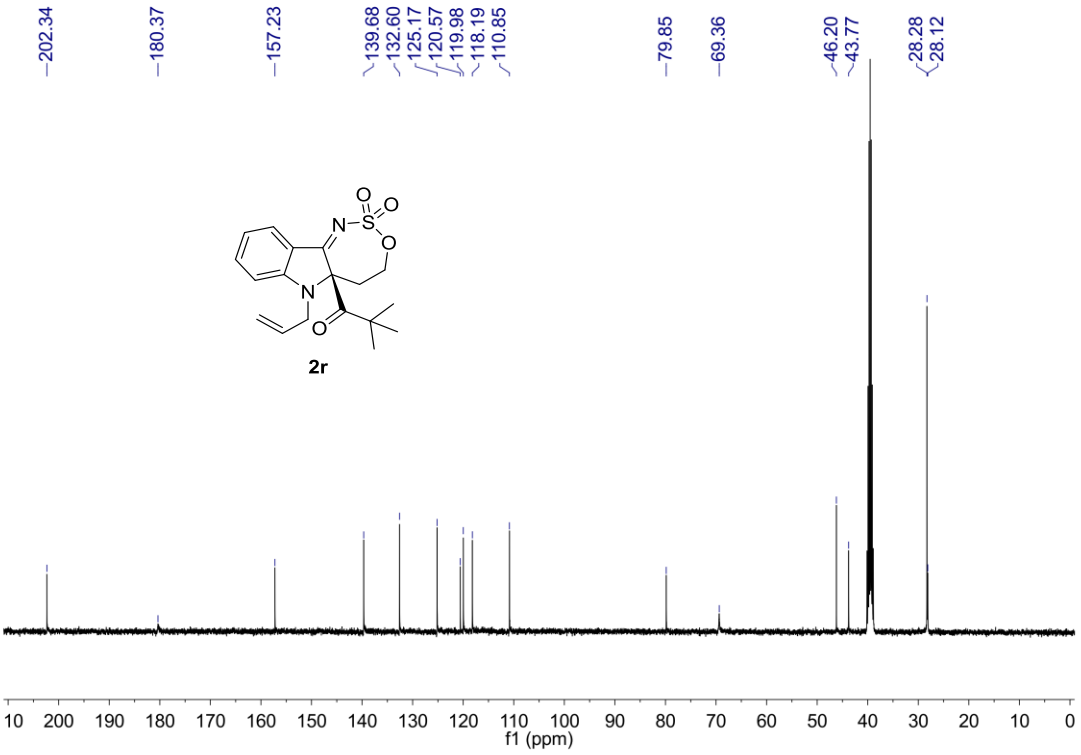
1.05
1.00
1.00
9.06
f1 (ppm)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139.68
132.60
125.17
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119.98
118.19
110.85
79.85
69.36
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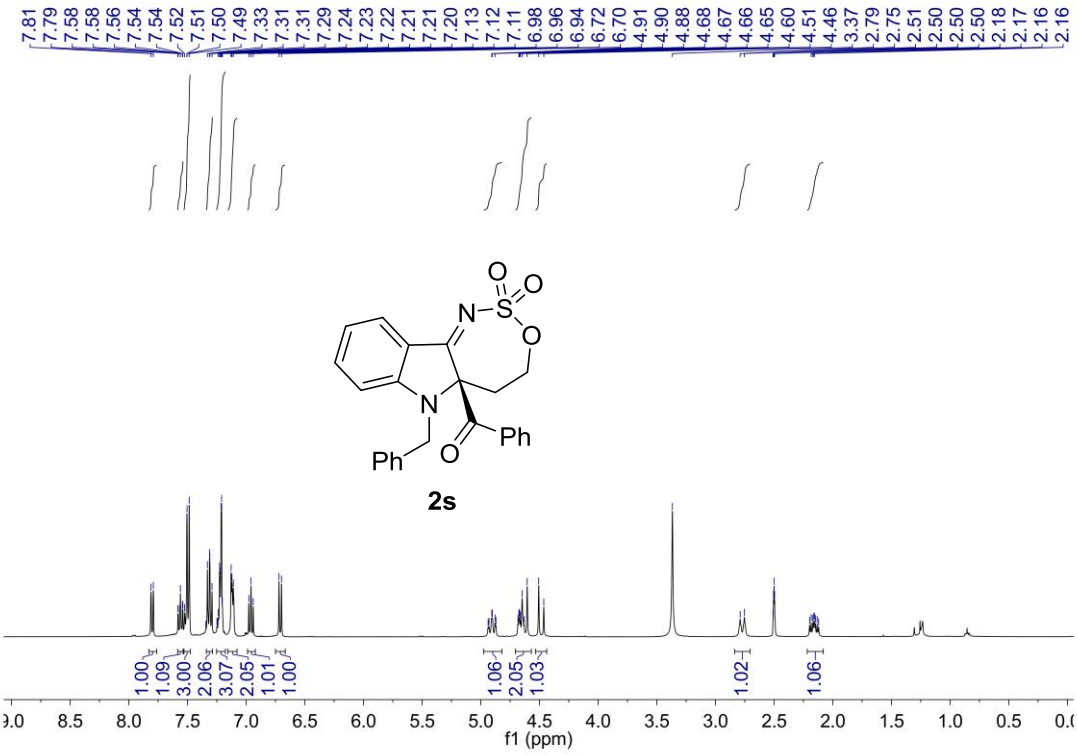
43.77
28.28
28.12
2r
f1 (ppm)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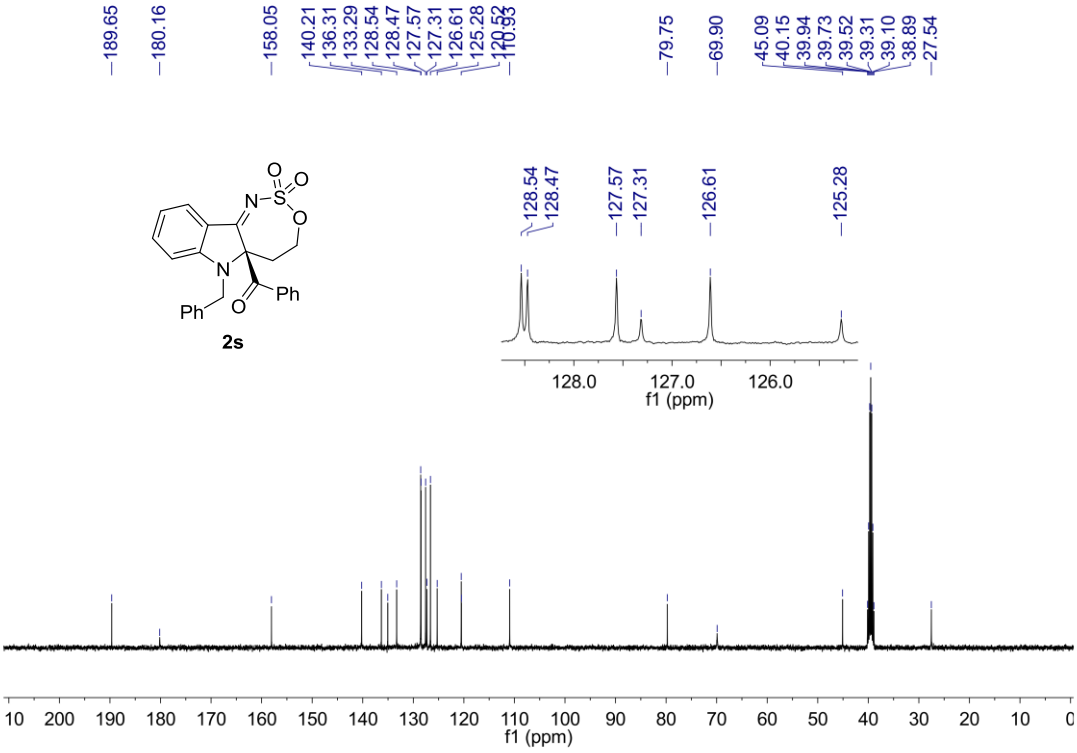
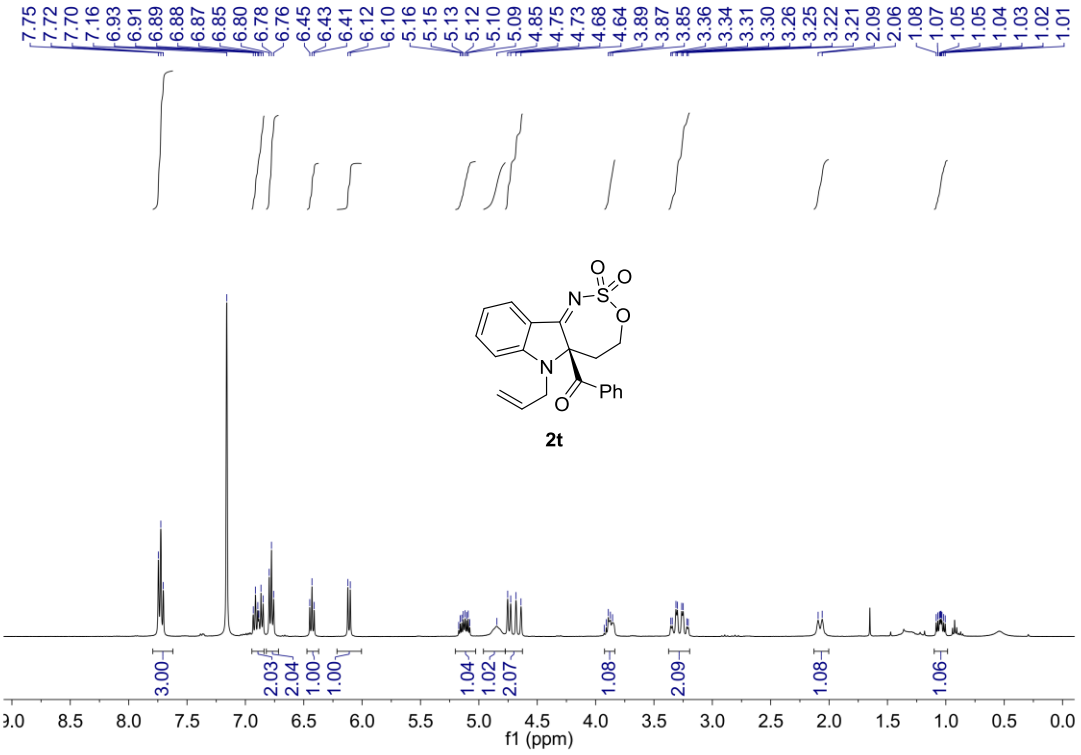
2s
1.00 1.09 3.00 2.06 3.07 2.05 1.01 1.00 1.06 2.05 1.03 1.02 1.06
f1 (ppm)


189.65 180.16 158.05 140.21 136.31 133.29 128.54 128.47 127.57 127.31 126.61 125.28 79.75 69.90 45.09 40.15 39.94 39.73 39.52 39.31 39.10 38.89 27.54
2s
128.54 128.47 127.57 127.31 126.61 125.28
f1 (ppm)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6.41
6.12
6.10
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5.15
5.13
5.12
5.10
5.09
4.85
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4.73
4.68
4.64
3.89
3.87
3.85
3.36
3.34
3.31
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3.26
3.25
3.22
3.21
2.09
2.06
1.08
1.07
1.05
1.05
1.04
1.03
1.02
1.01
O
O
N
S
O
N
O
Ph
2t
3.00
2.03
2.04
1.00
1.00
1.04
1.02
2.07
1.08
2.09
1.08
1.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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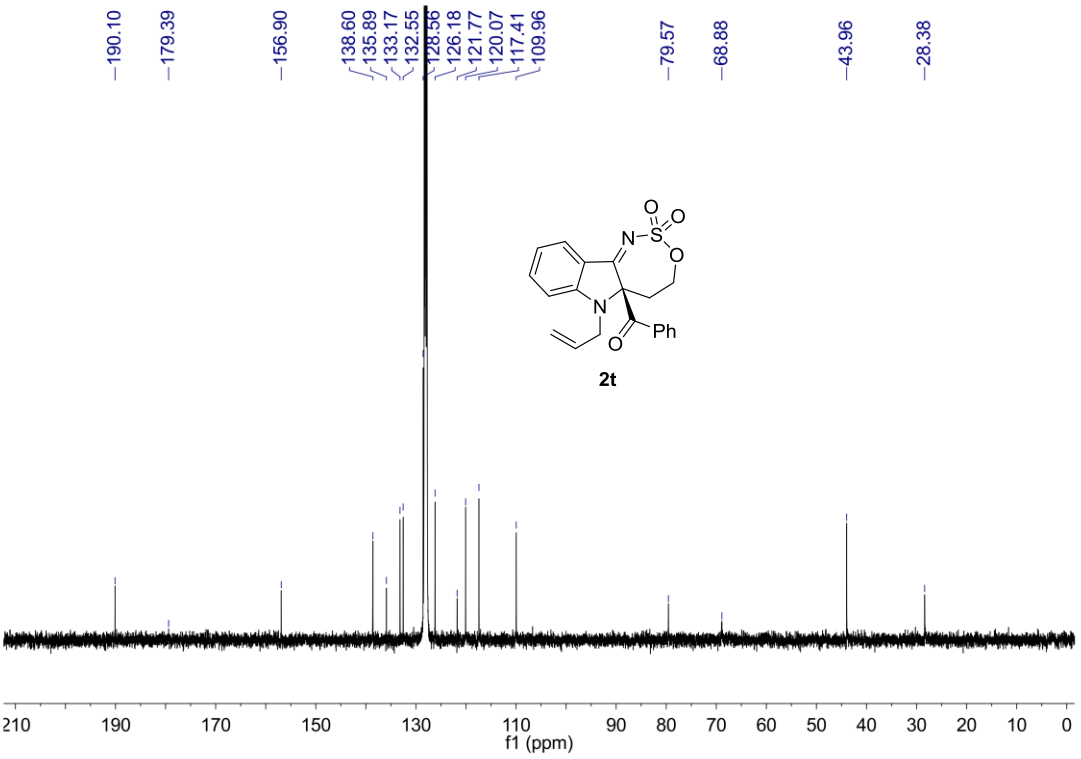
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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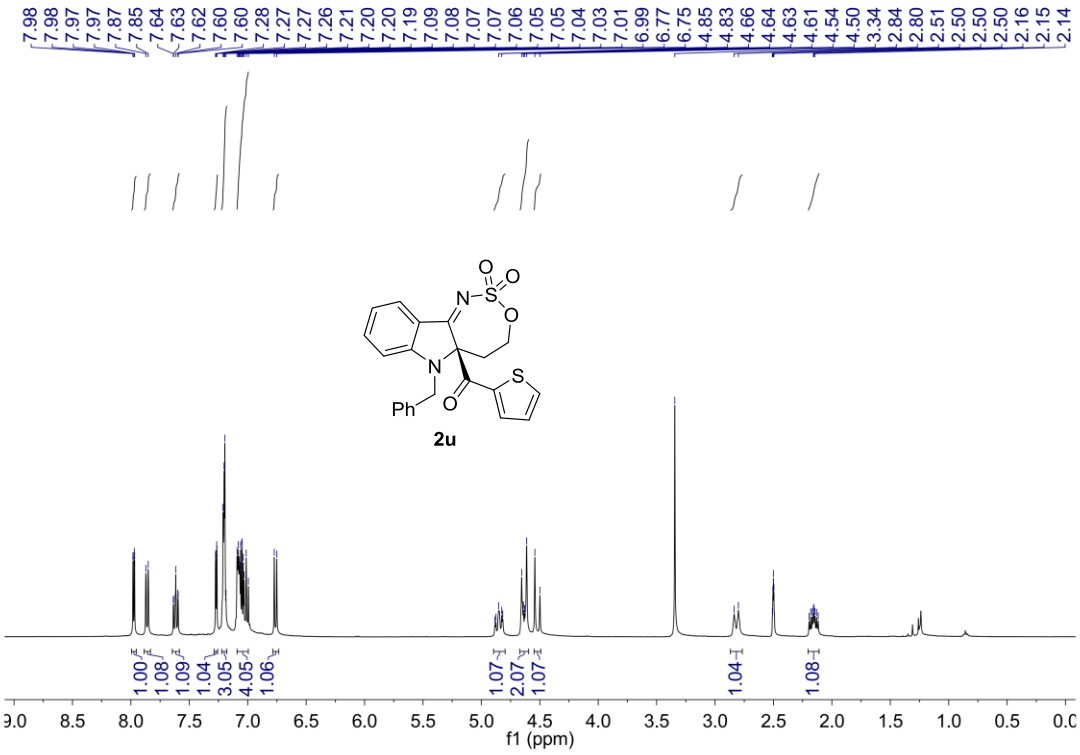
133.17
132.55
128.56
126.18
121.77
120.07
117.41
109.96
79.57
68.88
43.96
28.38
O
O
N
S
O
N
O
Ph
2t
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.97
7.87
7.85
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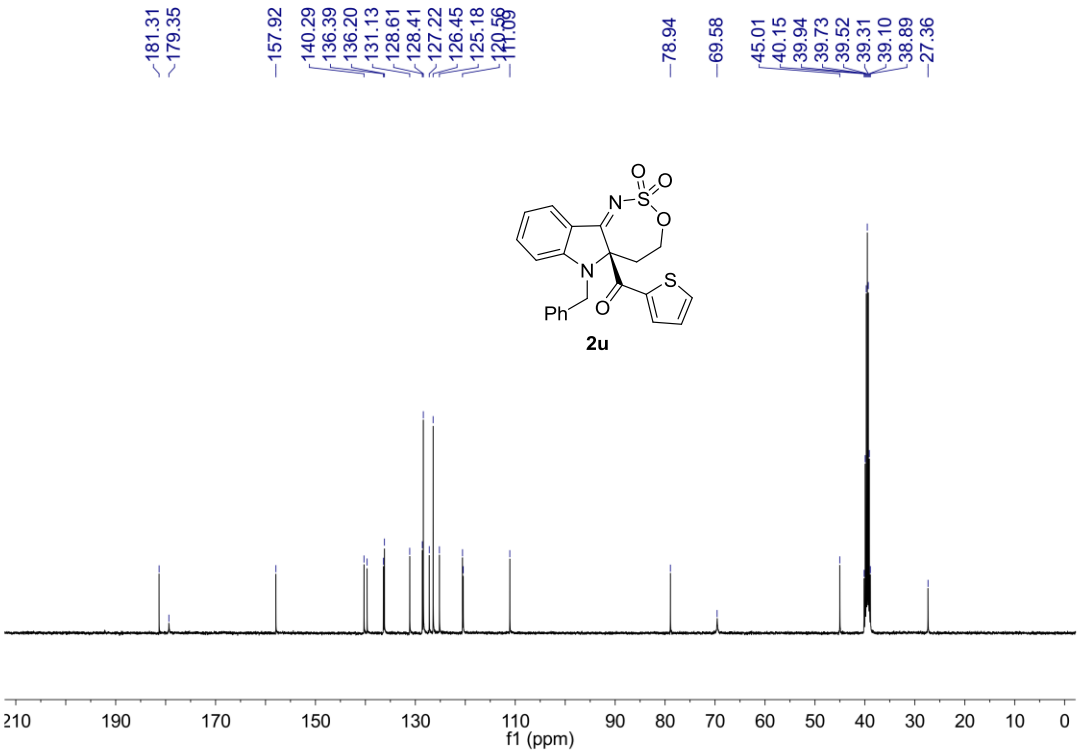
7.63
7.62
7.60
7.60
7.28
7.27
7.27
7.26
7.21
7.20
7.20
7.19
7.09
7.08
7.07
7.07
7.06
7.05
7.05
7.04
7.03
7.01
6.99
6.77
6.75
4.85
4.83
4.66
4.64
4.63
4.61
4.54
4.50
3.34
2.84
2.80
2.51
2.50
2.50
2.50
2.16
2.15
2.14
O
O
N
S
O
N
Ph
O
S
2u
1.00
1.08
1.09
1.04
3.05
4.05
1.06
1.07
2.07
1.07
1.04
1.08
f1 (ppm)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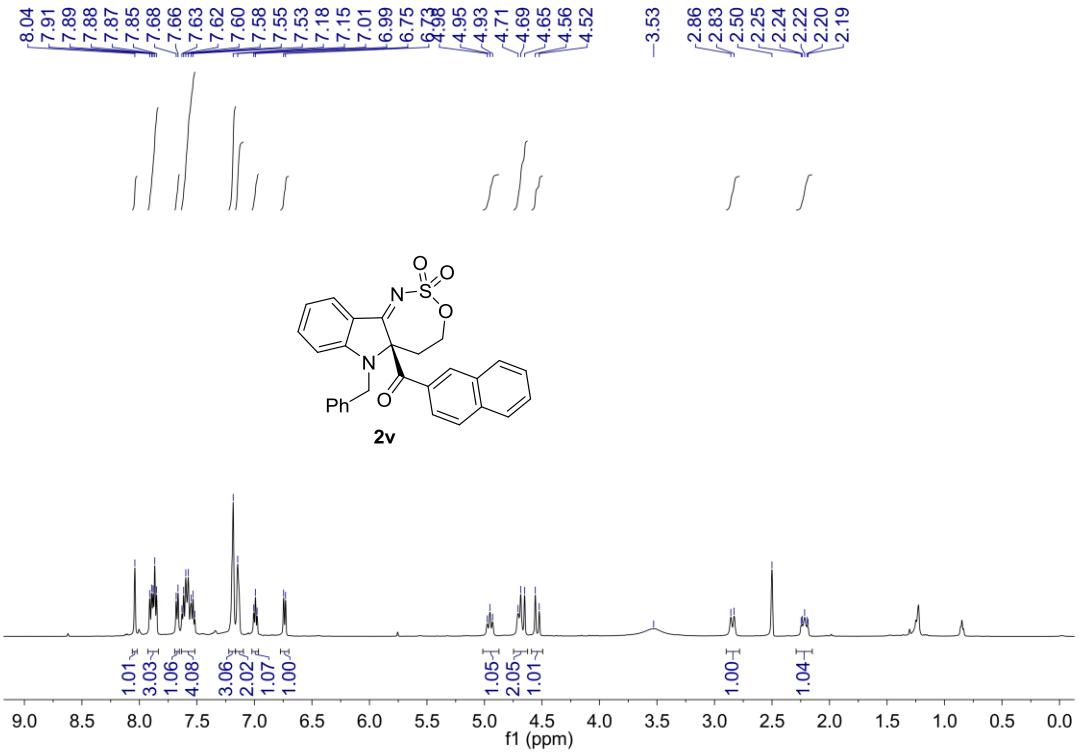
131.13
128.61
128.41
127.22
126.45
125.18
78.94
69.58
45.01
40.15
39.94
39.73
39.52
39.31
39.10
38.89
27.36
O
O
N
S
O
N
Ph
O
S
2u
f1 (ppm)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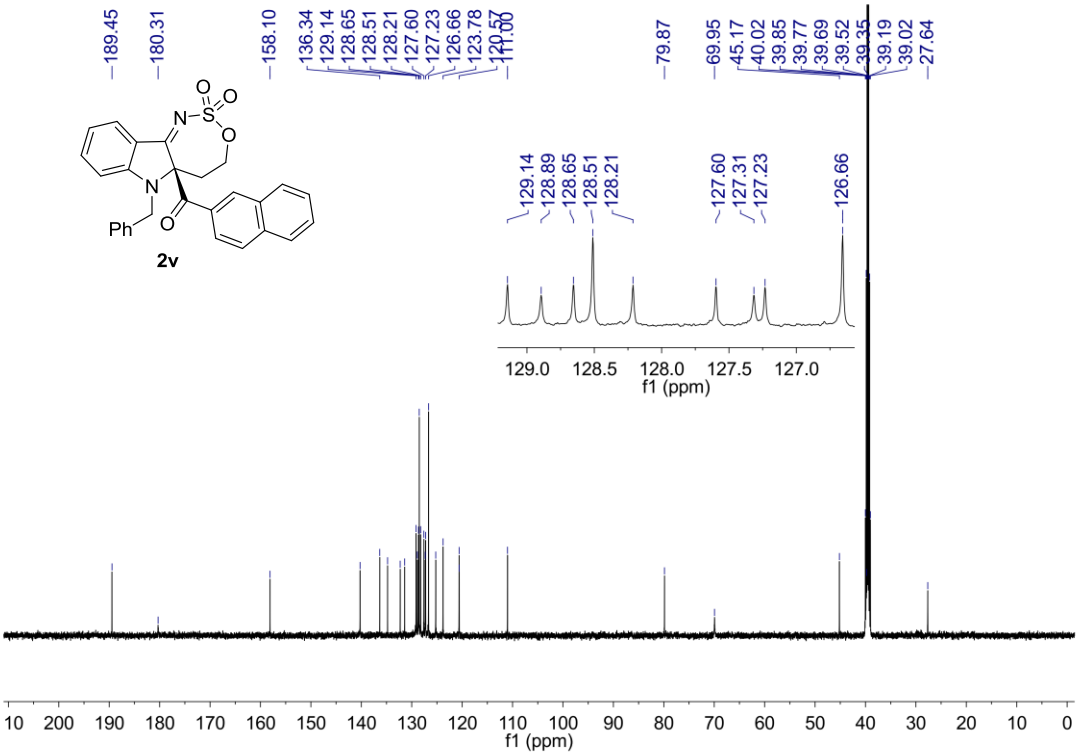
7.62
7.60
7.58
7.55
7.53
7.18
7.15
7.01
6.99
6.75
4.95
4.93
4.71
4.69
4.65
4.56
4.52
3.53
2.86
2.83
2.50
2.25
2.24
2.22
2.20
2.19
Ph
2v
1.01
3.03
1.06
4.08
3.06
2.02
1.07
1.00
1.05
2.05
1.01
1.00
1.04
f1 (ppm)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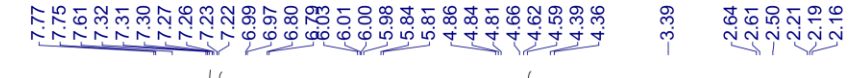
127.23
126.66
123.78
79.87
69.95
45.17
40.02
39.85
39.77
39.69
39.52
39.35
39.19
39.02
27.64
Ph
2v
129.14
128.89
128.65
128.51
128.21
127.60
127.31
127.23
126.66
f1 (ppm)
f1 (ppm)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7.22
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6.80
6.79
6.03
6.01
6.00
5.98
5.84
5.81
4.86
4.84
4.81
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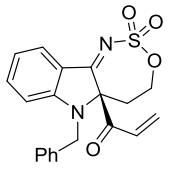
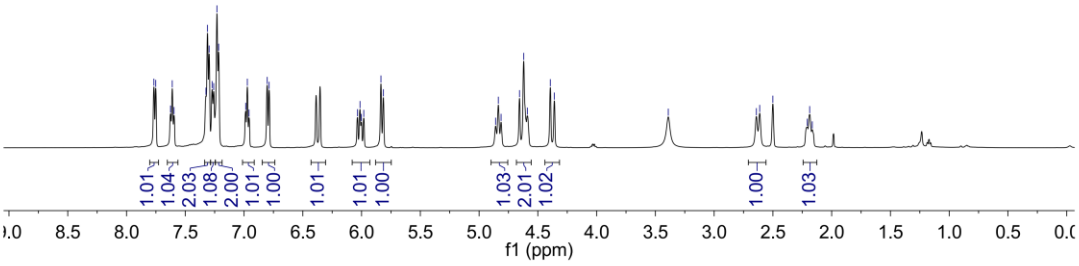
2.21
2.19
2.16


O
O
N
S
O
N
O
Ph

2w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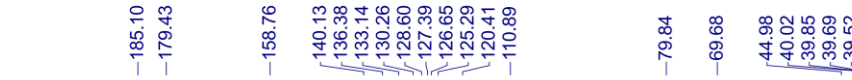
1.01
1.01
1.00
1.03
2.01
1.02
1.00
1.03
f1 (ppm)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126.65
125.29
120.41
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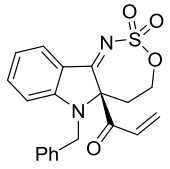
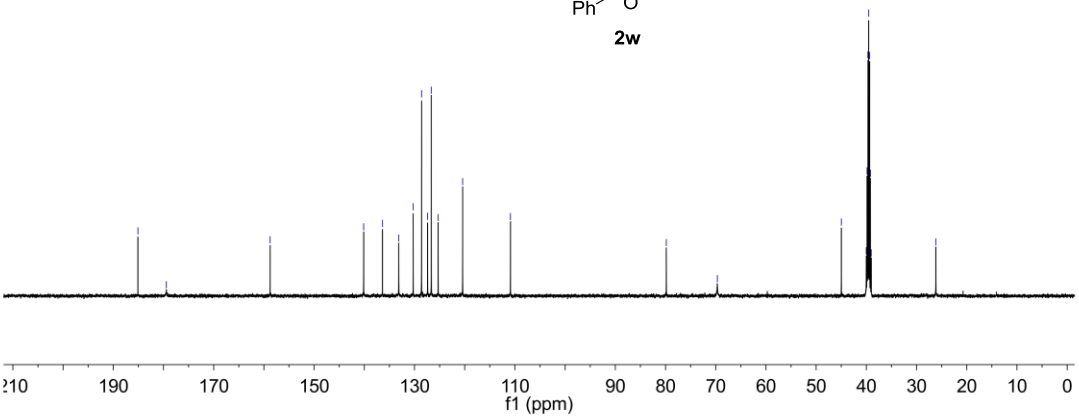
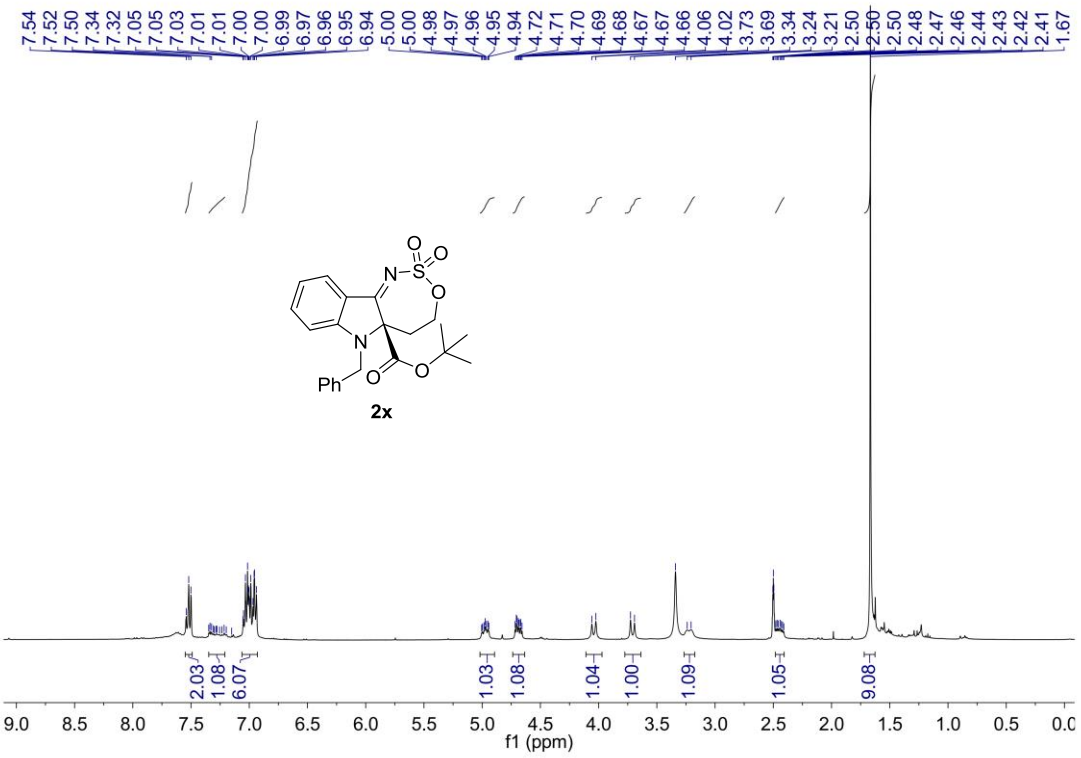
39.69
39.52
39.35
39.19
39.02
26.15


O
O
N
S
O
N
O
Ph

2w


f1 (ppm)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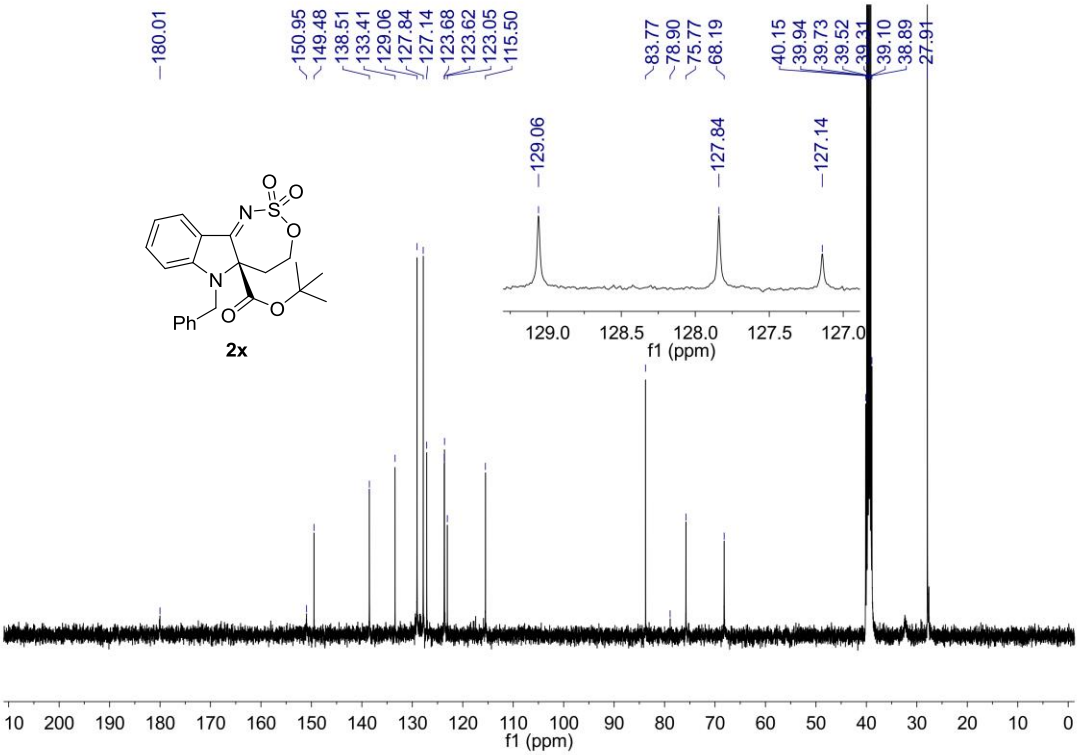
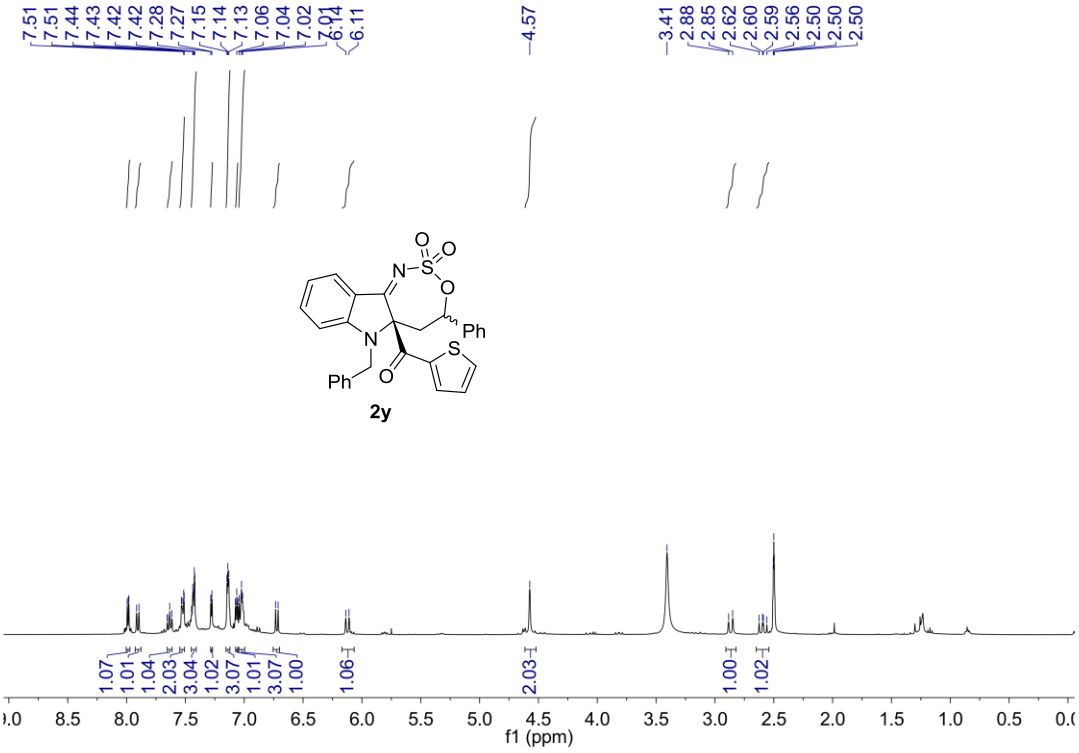
2x
2.03 1.08 6.07 1.03 1.08 1.04 1.00 1.09 1.05 9.08
f1 (ppm)


180.01 150.95 149.48 138.51 133.41 129.06 127.84 127.14 123.68 123.62 123.05 115.50 83.77 78.90 75.77 68.19 40.15 39.94 39.73 39.52 39.31 39.10 38.89 27.91
129.06 127.84 127.14
2x
f1 (ppm)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7.06
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6.11
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2.62
2.60
2.59
2.56
2.50
2.50
2.50
Ph
Ph
2y
1.07
1.01
1.04
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3.04
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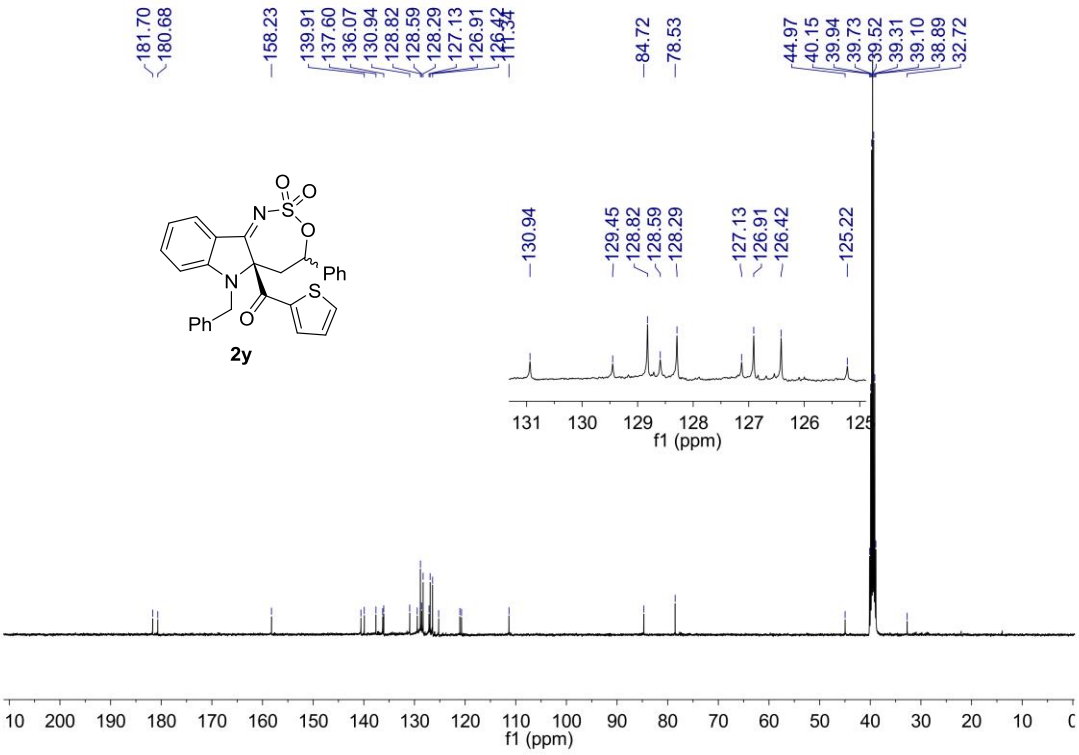
3.07
1.01
3.07
1.00
1.06
2.03
1.00
1.02
f1 (ppm)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39.10
38.89
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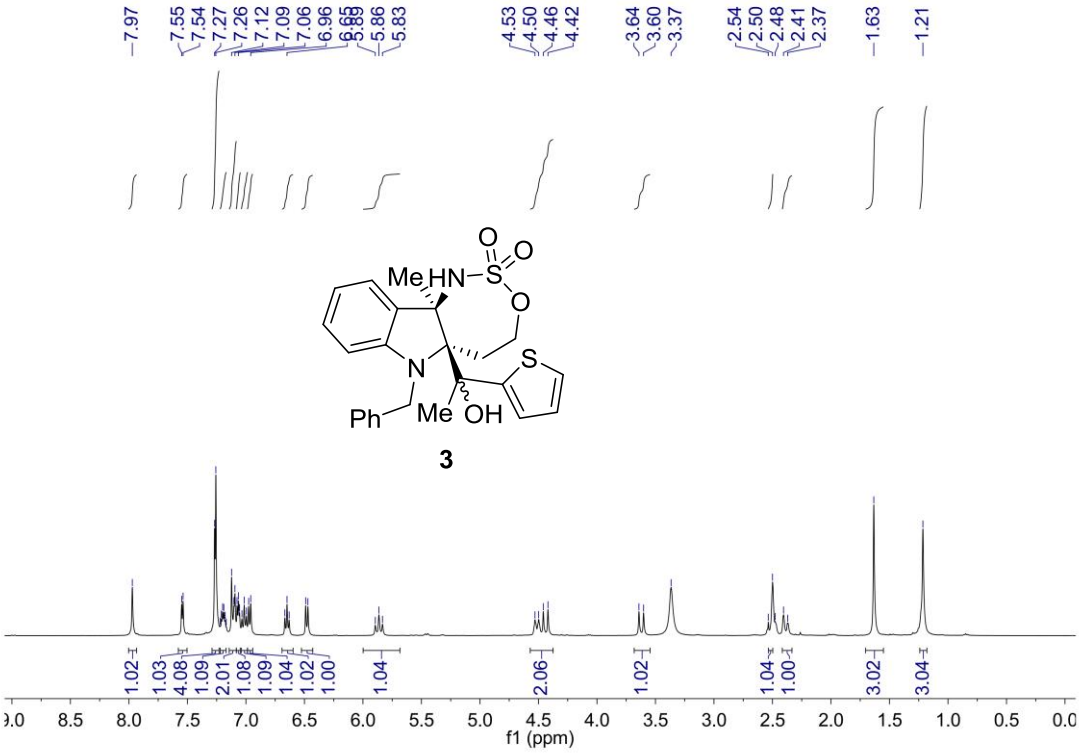
128.59
128.29
127.13
126.91
126.42
125.22
Ph
Ph
2y
f1 (ppm)
f1 (ppm)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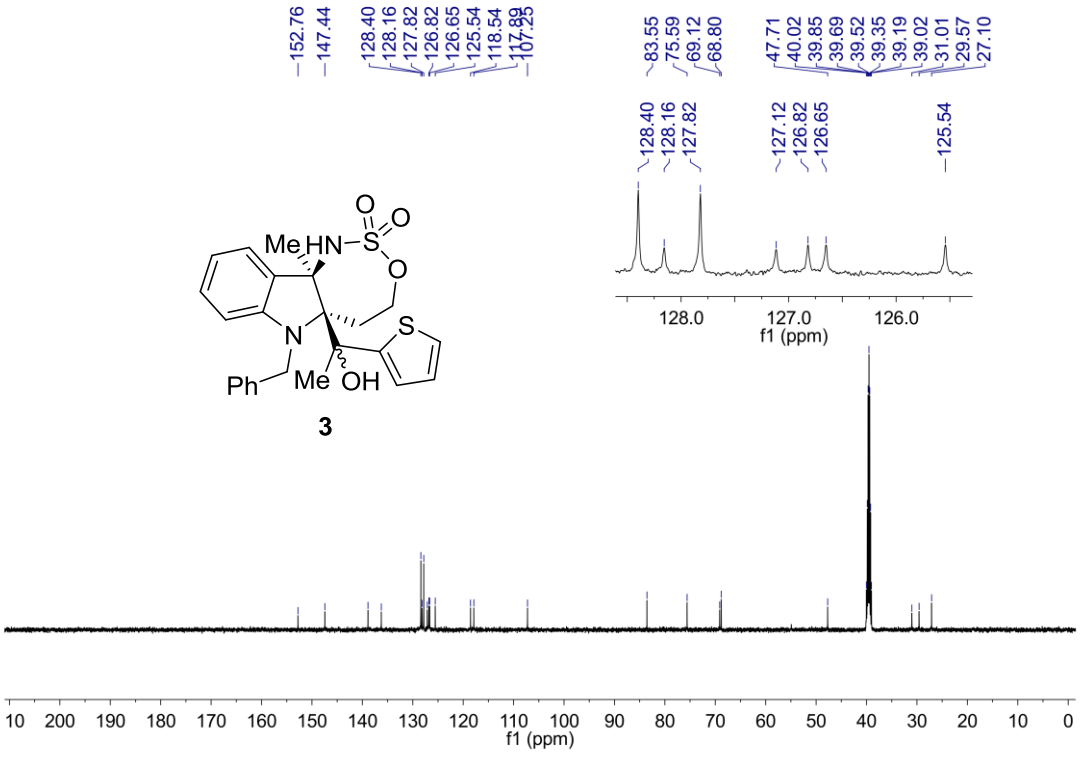
7.55
7.54
7.27
7.26
7.12
7.09
7.06
6.96
5.86
5.83
4.53
4.50
4.46
4.42
3.64
3.60
3.37
2.54
2.50
2.48
2.41
2.37
1.63
1.21
MeHN
S
O
N
S
Ph
Me
OH
1.02
1.03
4.08
1.09
2.01
1.08
1.09
1.04
1.02
1.00
1.04
2.06
1.02
1.04
1.00
3.02
3.04
f1 (ppm)

3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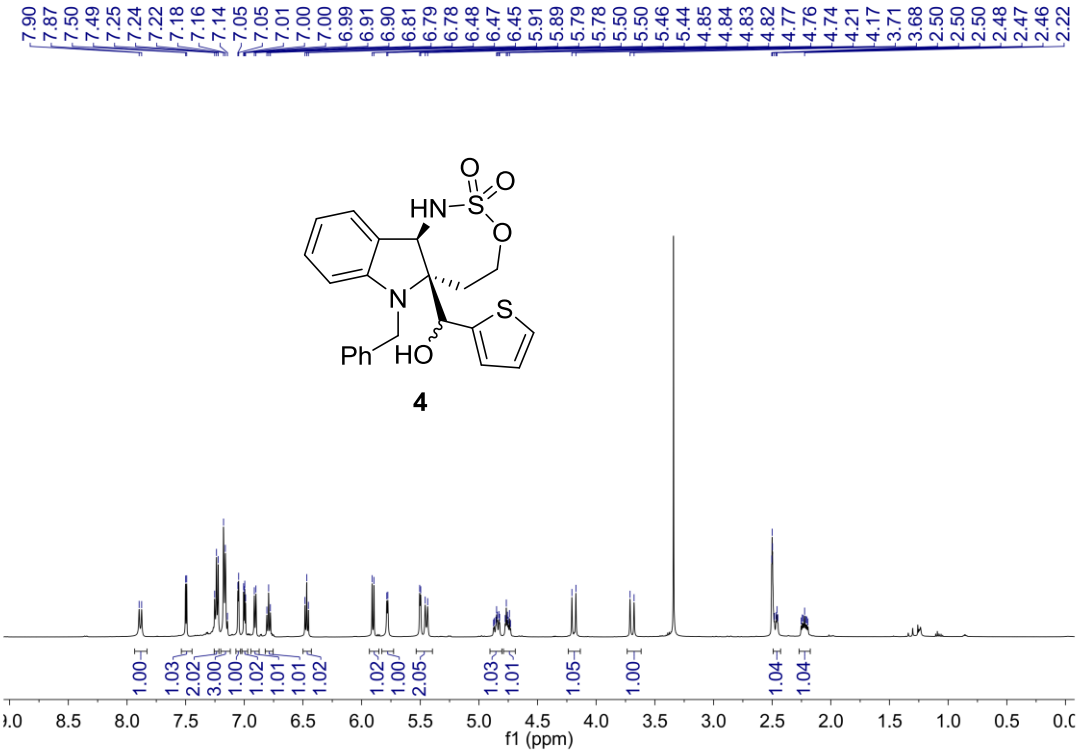
147.44
128.40
128.16
127.82
126.82
126.65
125.54
118.54
83.55
75.59
69.12
68.80
47.71
40.02
39.85
39.69
39.52
39.35
39.19
39.02
31.01
29.57
27.10
128.40
128.16
127.82
127.12
126.82
126.65
125.54
128.0
127.0
126.0
f1 (ppm)
MeHN
S
O
N
S
Ph
Me
OH
f1 (ppm)

3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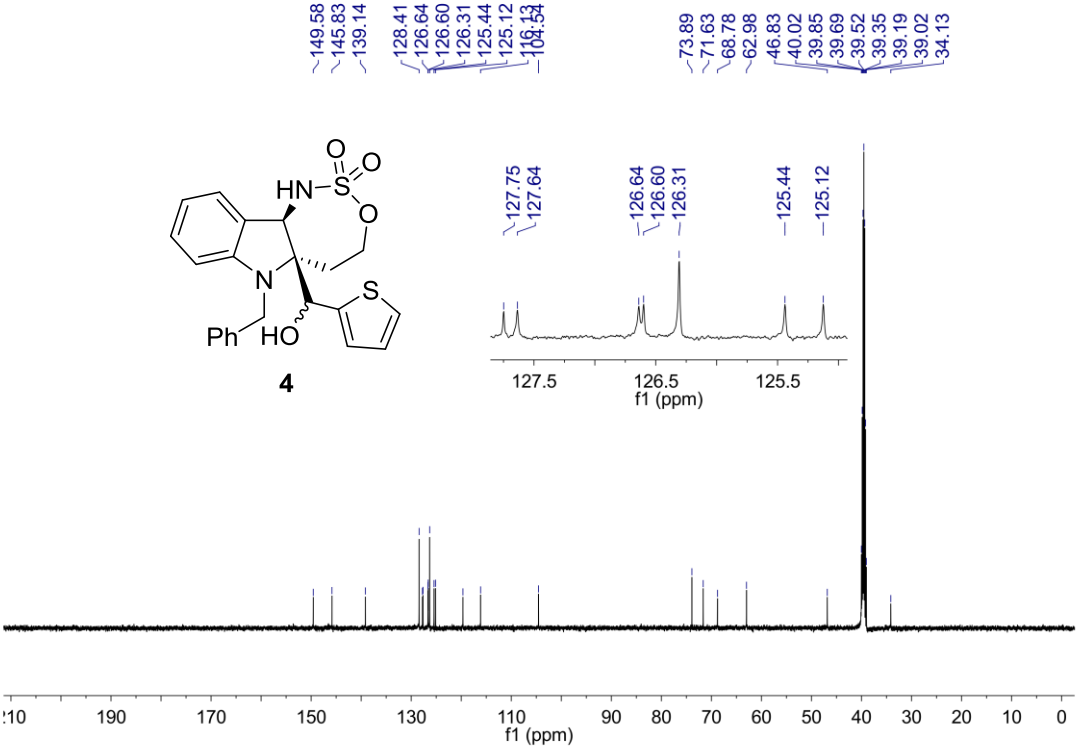
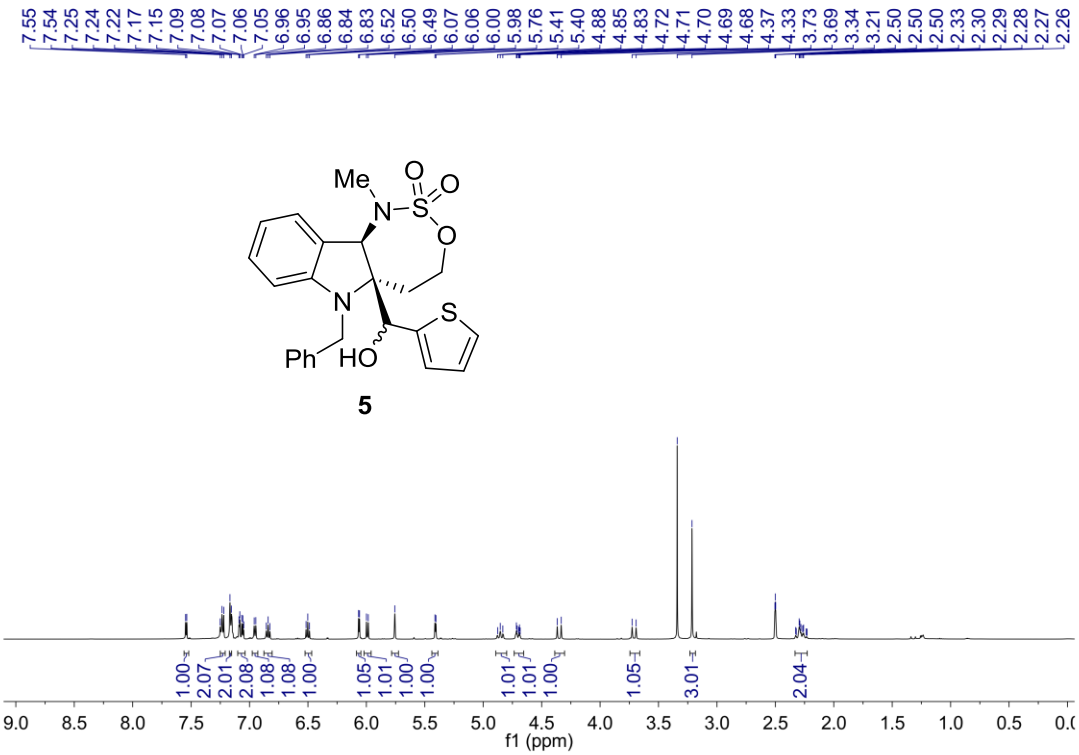
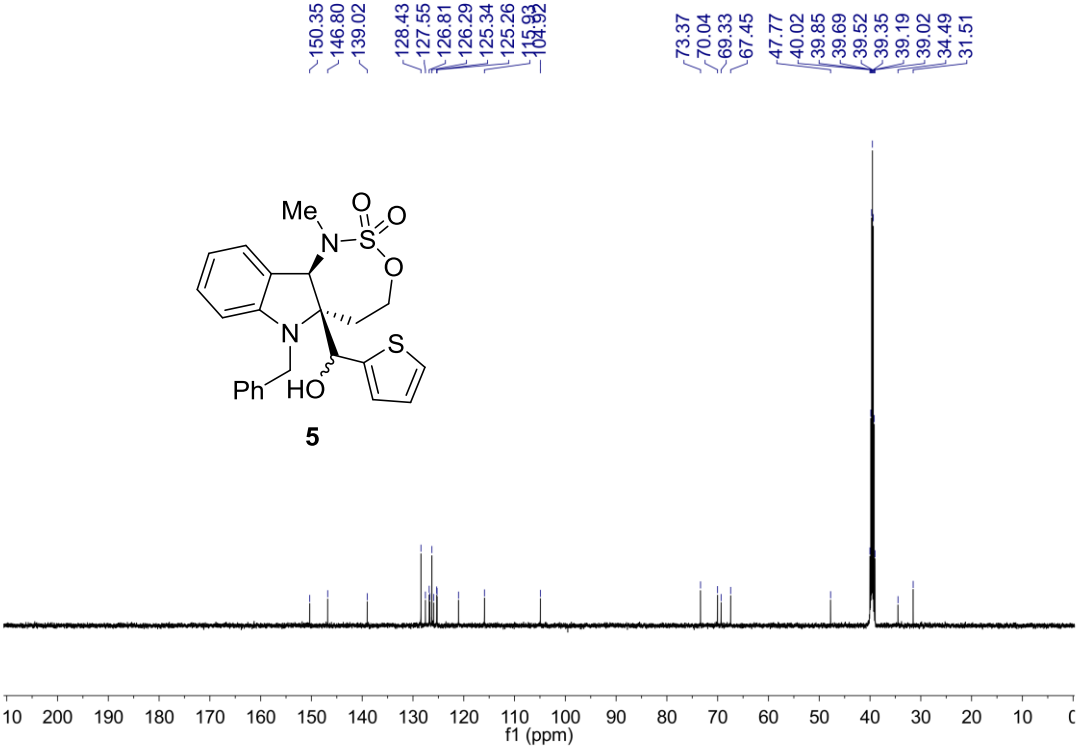
7.87
7.50
7.49
7.25
7.24
7.22
7.18
7.16
7.14
7.05
7.05
7.01
7.00
7.00
6.99
6.91
6.90
6.81
6.79
6.78
6.48
6.47
6.45
5.91
5.89
5.79
5.78
5.50
5.50
5.46
5.44
4.85
4.84
4.83
4.82
4.77
4.76
4.74
4.21
4.17
3.71
3.68
2.50
2.50
2.50
2.48
2.47
2.46
2.22
HN
S
O
O
O
N
S
Ph
HO
4
1.00
1.03
2.02
3.00
1.00
1.02
1.01
1.01
1.02
1.02
1.00
2.05
1.03
1.01
1.05
1.00
1.04
1.04
8.5
8.0
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
0.5
f1 (ppm)

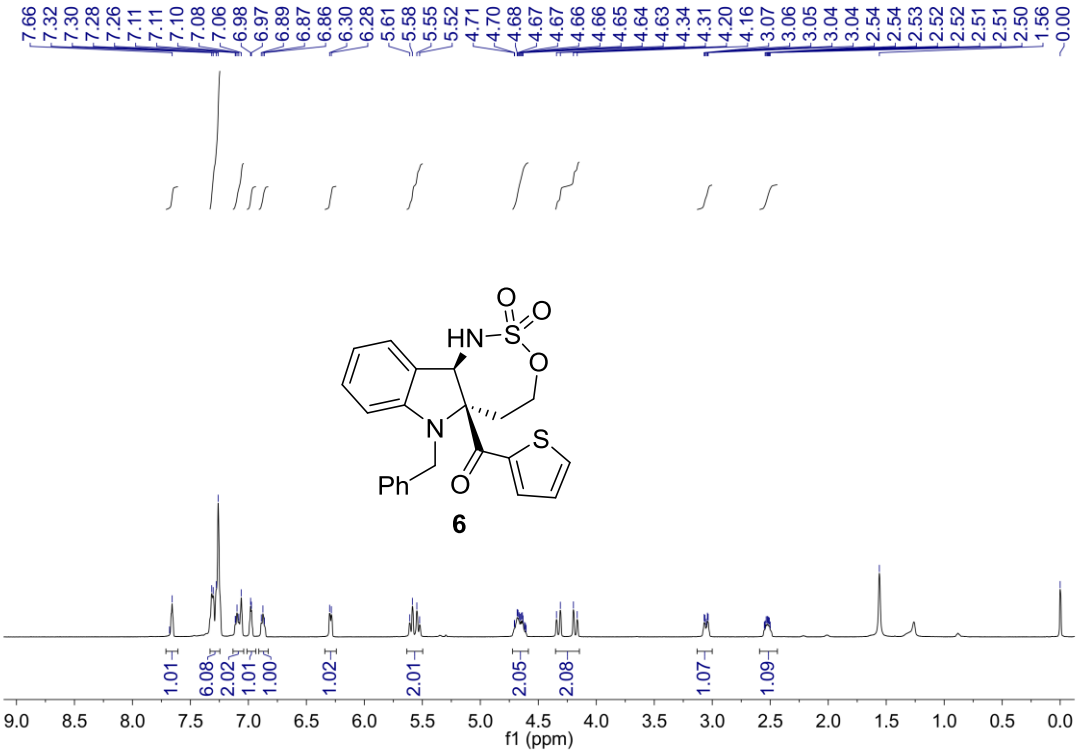

149.58
145.83
139.14
128.41
126.64
126.60
126.31
125.44
125.12
73.89
71.63
68.78
62.98
46.83
40.02
39.85
39.69
39.52
39.35
39.19
39.02
34.13
HN
S
O
O
O
N
S
Ph
HO
4
127.75
127.64
126.64
126.60
126.31
125.44
125.12
127.5
126.5
125.5
f1 (ppm)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

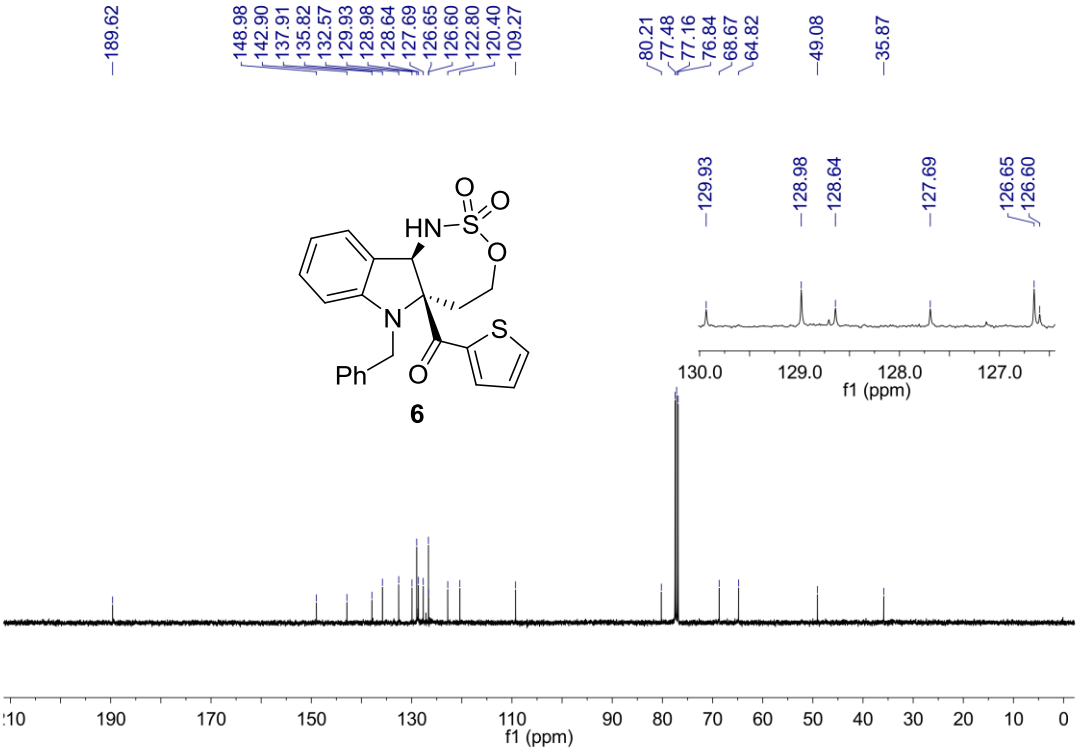

7.55 7.54 7.25 7.24 7.22 7.17 7.15 7.09 7.08 7.07 7.06 7.05 6.96 6.95 6.86 6.84 6.83 6.52 6.50 6.49 6.07 6.06 6.00 5.98 5.76 5.41 5.40 4.88 4.85 4.83 4.72 4.71 4.70 4.69 4.68 4.37 4.33 3.73 3.69 3.34 3.21 2.50 2.50 2.50 2.33 2.30 2.29 2.28 2.27 2.26
Me
O
O
N
S
O
N
S
Ph
HO
1.00 2.07 2.01 2.08 1.08 1.08 1.00 1.05 1.01 1.00 1.00 1.01 1.01 1.00 1.05 3.01 2.04
9.0 8.5 8.0 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 0.5
f1 (ppm)

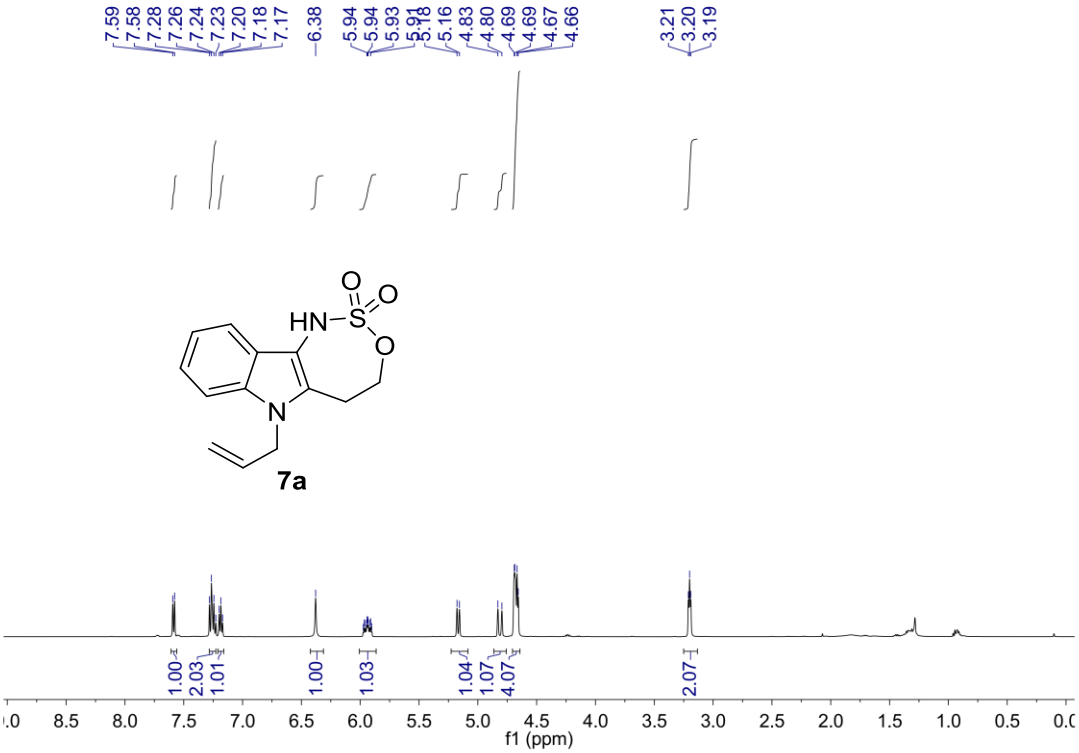
5

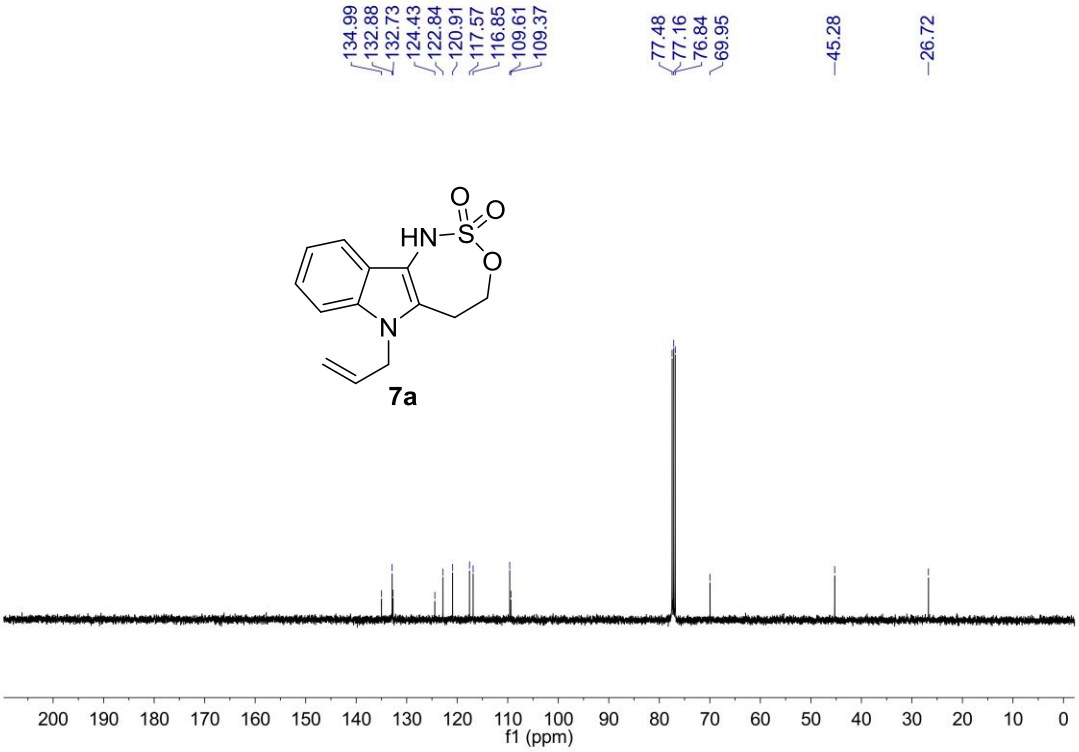

150.35 146.80 139.02 128.43 127.55 126.81 126.29 125.34 125.26 115.93 104.92 73.37 70.04 69.33 67.45 47.77 40.02 39.85 39.69 39.52 39.35 39.19 39.02 34.49 31.51
Me
O
O
N
S
O
N
S
Ph
HO
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

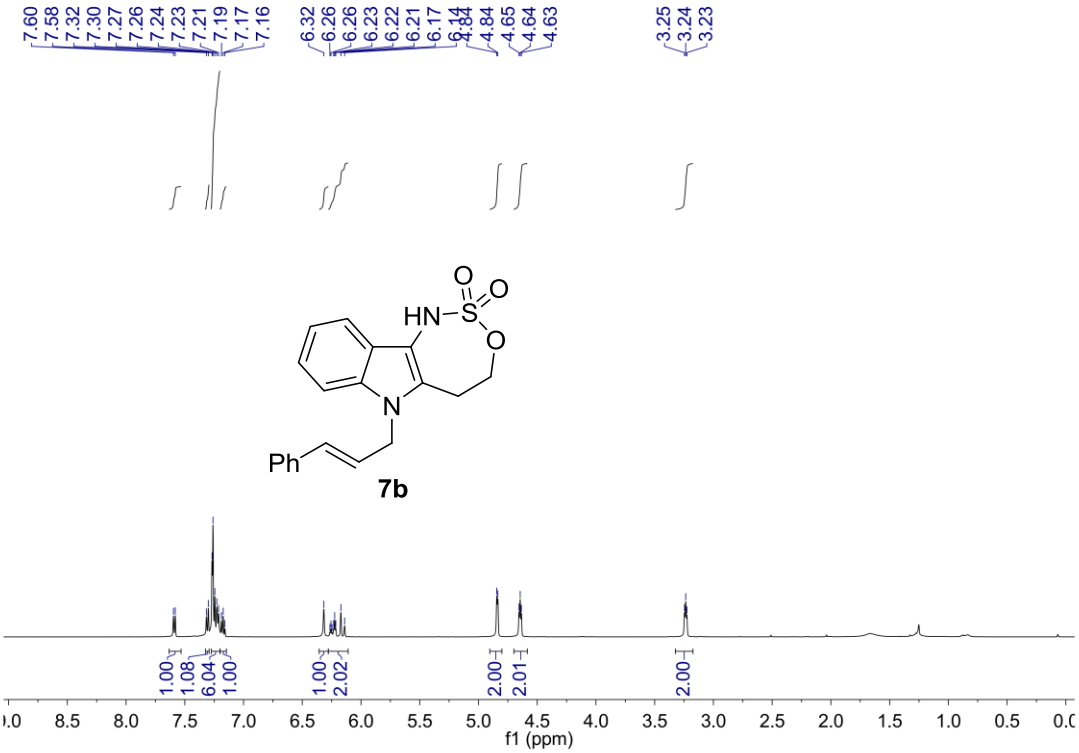
5

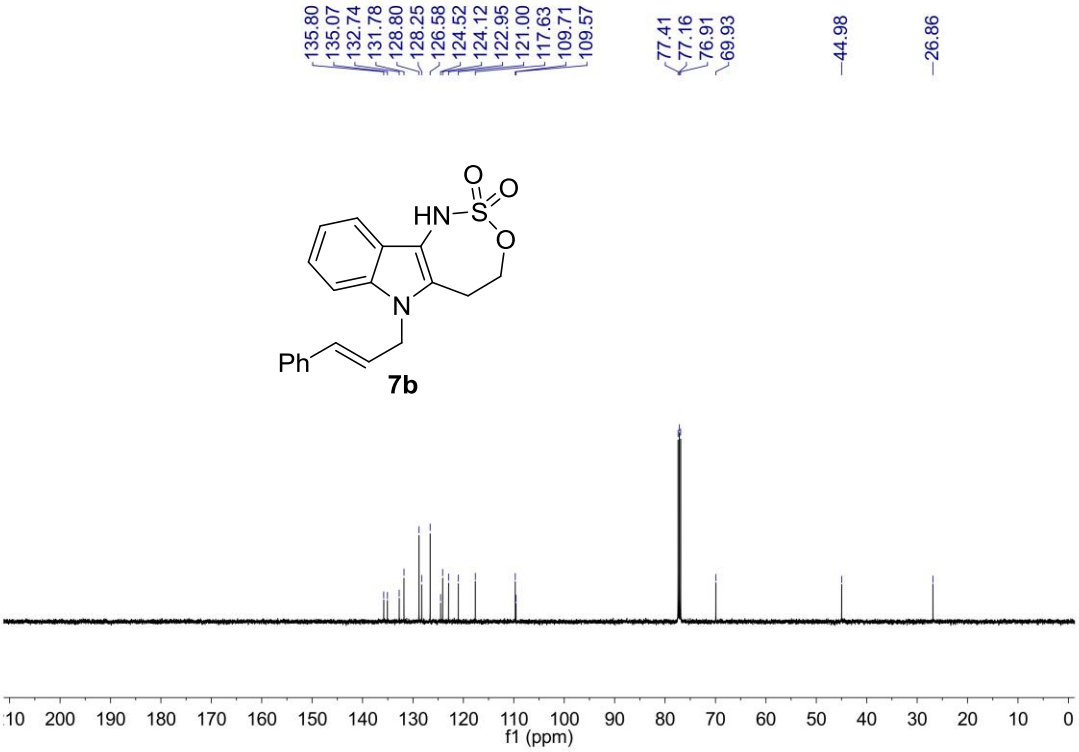

7.66
7.32
7.30
7.28
7.26
7.11
7.11
7.10
7.08
7.06
6.98
6.97
6.89
6.87
6.86
6.30
6.28
5.61
5.58
5.55
5.52
4.71
4.70
4.68
4.67
4.67
4.66
4.66
4.65
4.64
4.63
4.34
4.31
4.20
4.16
3.07
3.06
3.05
3.04
3.04
2.54
2.54
2.53
2.52
2.52
2.51
2.51
2.50
1.56
0.00
HN
S
O
O
O
N
S
Ph
O
6
1.01
6.08
2.02
1.01
1.00
1.02
2.01
2.05
2.08
1.07
1.09
9.0
8.5
8.0
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
0.5
0.0
f1 (ppm)

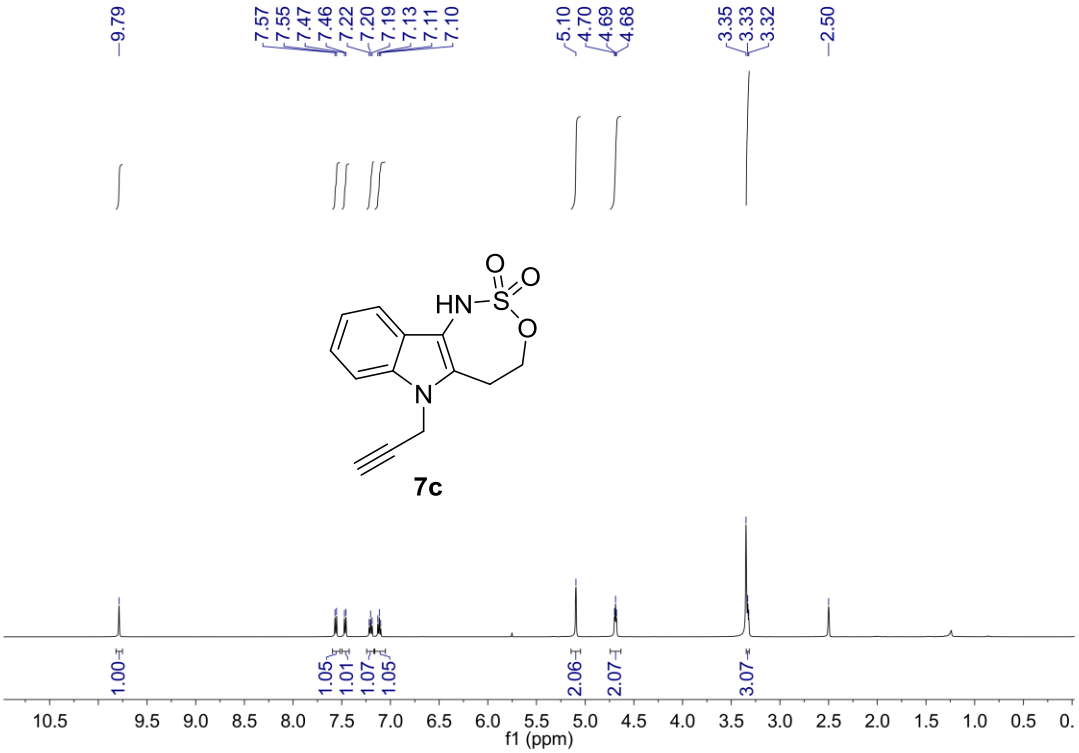

189.62
148.98
142.90
137.91
135.82
132.57
129.93
128.98
128.64
127.69
126.65
126.60
122.80
120.40
109.27
80.21
77.48
77.16
76.84
68.67
64.82
49.08
35.87
HN
S
O
O
O
N
S
Ph
O
6
129.93
128.98
128.64
127.69
126.65
126.60
130.0
129.0
128.0
127.0
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

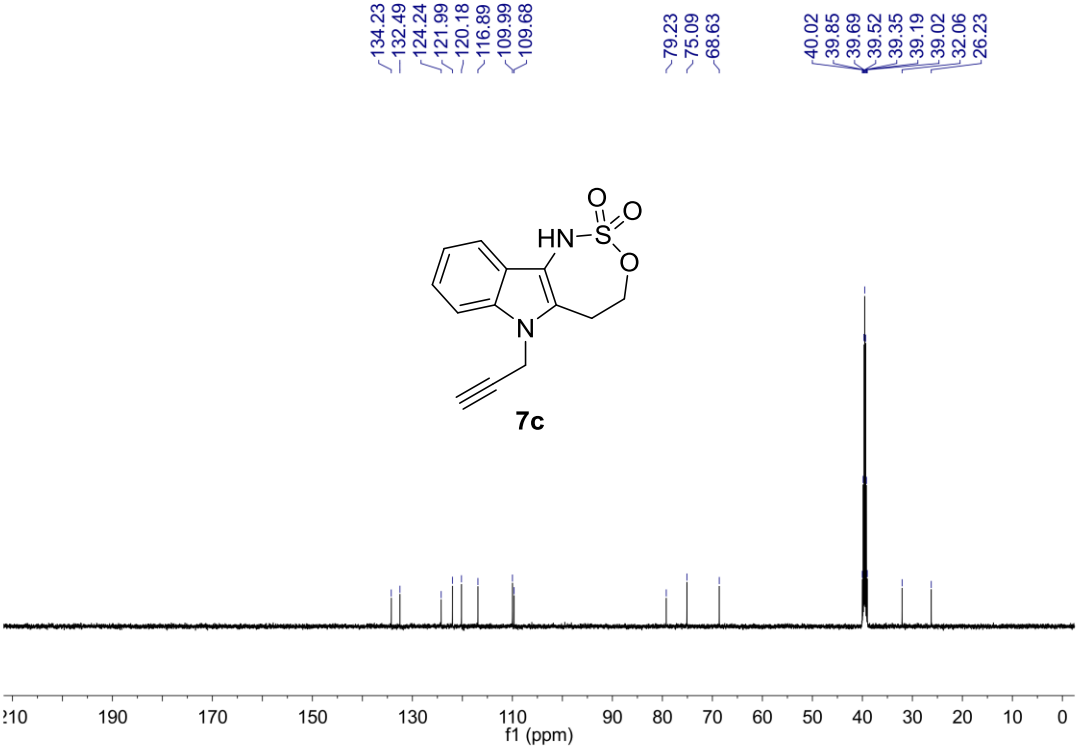

7.59
7.58
7.28
7.26
7.24
7.23
7.20
7.18
7.17
6.38
5.94
5.94
5.93
5.91
5.16
4.83
4.80
4.69
4.69
4.67
4.66
3.21
3.20
3.19
HN
S
O
O
O
N
7a
1.00
2.03
1.01
1.00
1.03
1.04
1.07
4.07
2.07
f1 (ppm)

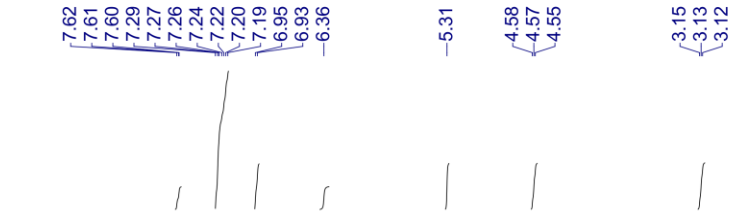

134.99
132.88
132.73
124.43
122.84
120.91
117.57
116.85
109.61
109.37
77.48
77.16
76.84
69.95
45.28
26.72
HN
S
O
O
O
N
7a
f1 (ppm)


7.60
7.58
7.32
7.30
7.27
7.26
7.24
7.23
7.21
7.19
7.17
7.16
6.32
6.26
6.26
6.23
6.22
6.21
6.17
4.84
4.65
4.64
4.63
3.25
3.24
3.23
HN
S
O
O
O
N
Ph
7b
1.00
1.08
6.04
1.00
1.00
2.02
2.00
2.01
2.00
f1 (ppm)


135.80
135.07
132.74
131.78
128.80
128.25
126.58
124.52
124.12
122.95
121.00
117.63
109.71
109.57
77.41
77.16
76.91
69.93
44.98
26.86
HN
S
O
O
O
N
Ph
7b
f1 (ppm)


9.79
7.57
7.55
7.47
7.46
7.22
7.20
7.19
7.13
7.11
7.10
5.10
4.70
4.69
4.68
3.35
3.33
3.32
2.50
HN
S
O
O
O
N
7c
1.00
1.05
1.01
1.07
1.05
2.06
2.07
3.07
f1 (ppm)


134.23
132.49
124.24
121.99
120.18
116.89
109.99
109.68
79.23
75.09
68.63
40.02
39.85
39.69
39.52
39.35
39.19
39.02
32.06
26.23
HN
S
O
O
O
N
7c
f1 (ppm)


7.62
7.61
7.60
7.29
7.27
7.26
7.24
7.22
7.20
7.19
6.95
6.93
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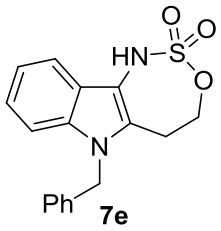
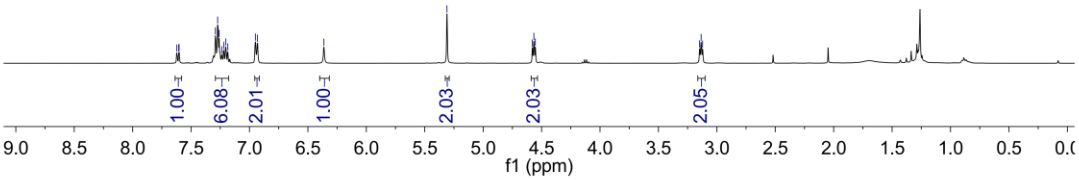
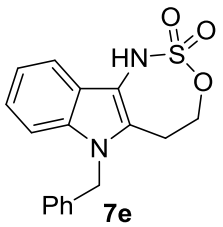
4.58
4.57
4.55
3.15
3.13
3.12


O
O
HN
S
O
N
Ph

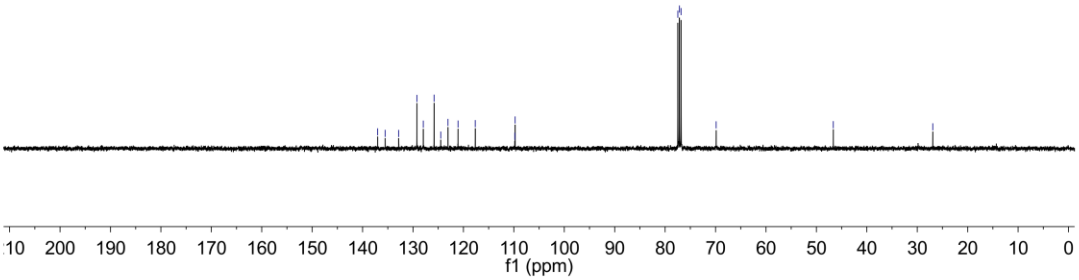
7e

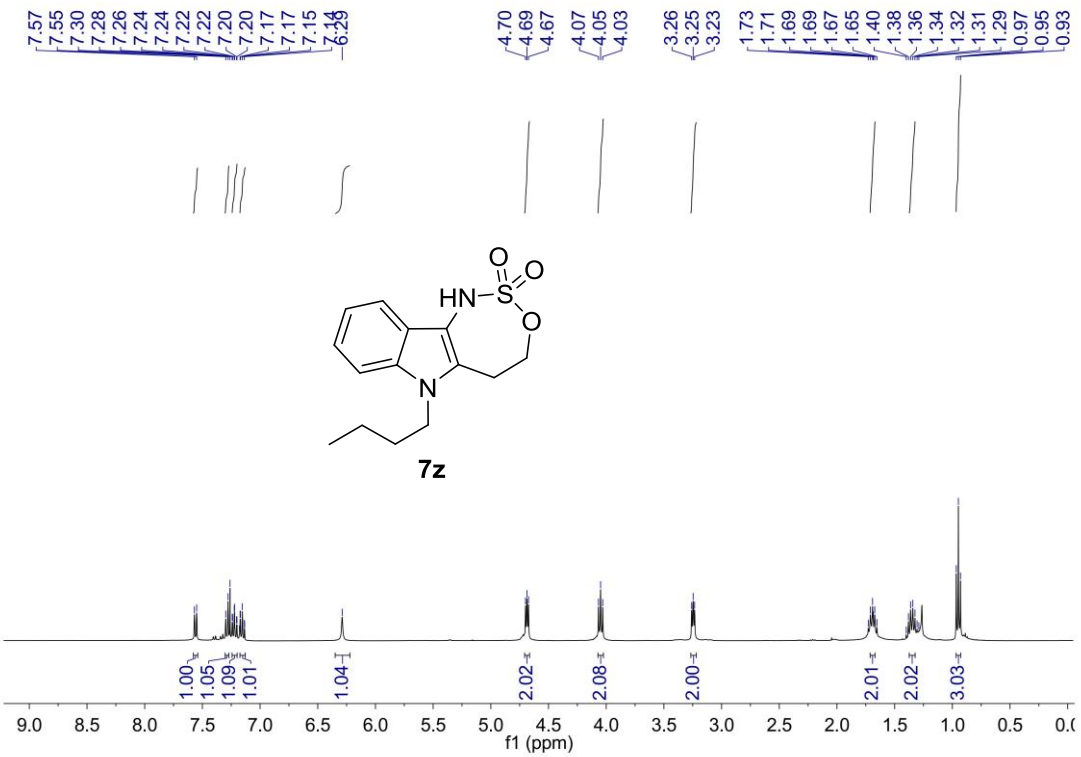

1.00
6.08
2.01
1.00
2.03
2.03
2.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

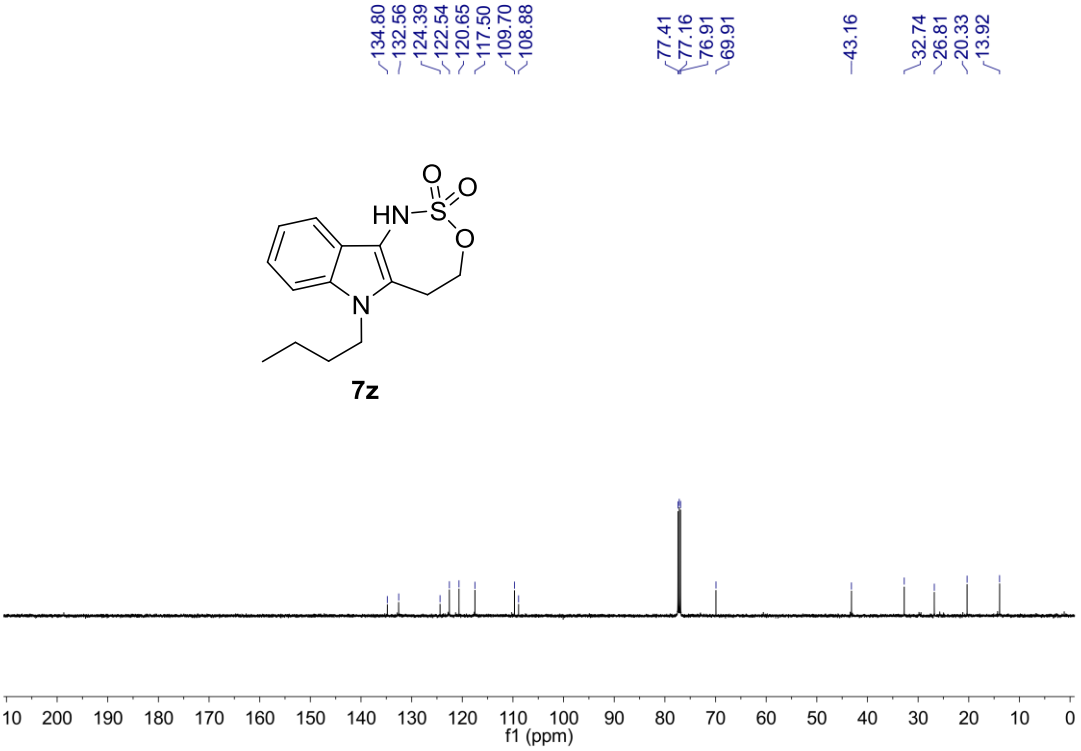

137.01
135.51
132.84
129.23
127.96
125.82
124.50
123.07
121.07
117.66
109.80
109.75
77.48
77.16
76.84
69.88
46.65
26.92


O
O
HN
S
O
N
Ph

7e

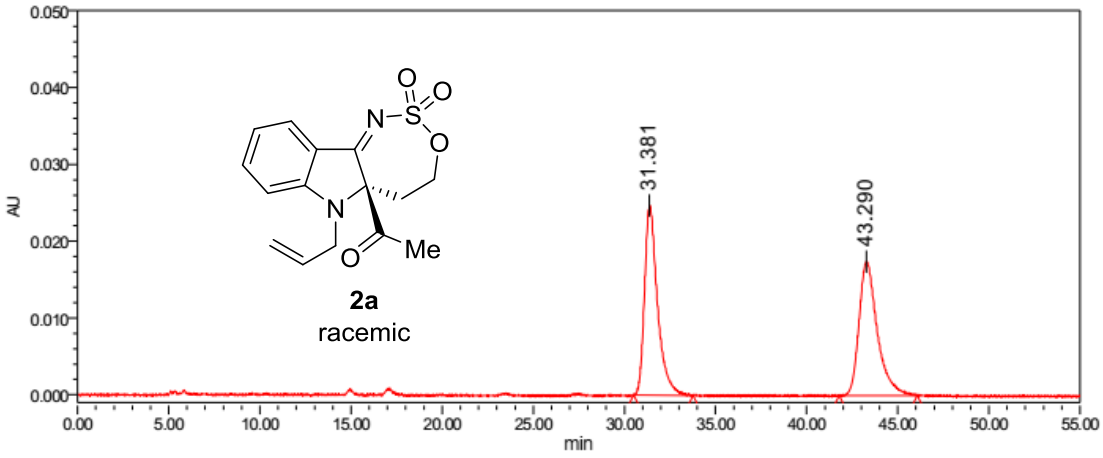

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.57
7.55
7.30
7.28
7.26
7.24
7.24
7.22
7.22
7.20
7.20
7.17
7.17
7.15
7.14
6.23
4.70
4.69
4.67
4.07
4.05
4.03
3.26
3.25
3.23
1.73
1.71
1.69
1.69
1.67
1.65
1.40
1.38
1.36
1.34
1.32
1.31
1.29
0.97
0.95
0.93
HN
S
O
O
O
N
7z
1.00
1.05
1.09
1.01
1.04
2.02
2.08
2.00
2.01
2.02
3.03
9.0
8.5
8.0
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
0.5
f1 (ppm)


134.80
132.56
124.39
122.54
120.65
117.50
109.70
108.88
77.41
77.16
76.91
69.91
43.16
32.74
26.81
20.33
13.92
HN
S
O
O
O
N
7z
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

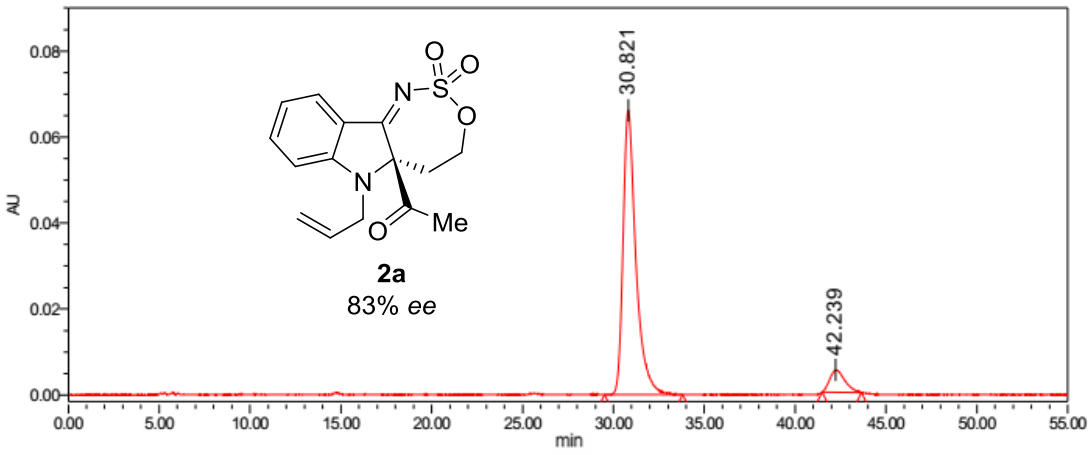
Condition: hexane : 2-propanol = 85:15

Flow rate = 1.0 mL/min, λ = 254 nm, Daicel Chiralpak AD-H.



PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	31.381	1232322	24685	50.21
2	43.290	1222042	17435	49.79
Total		2454364	42120	100

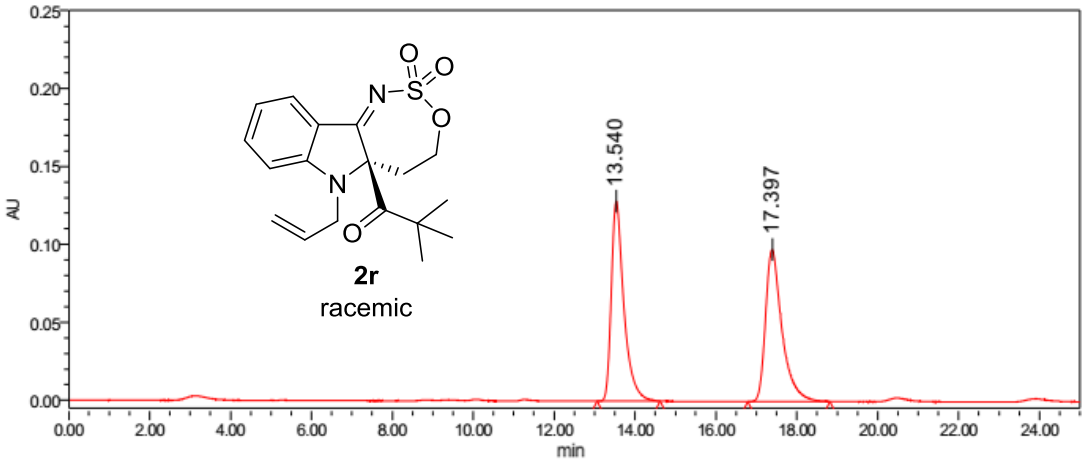


PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	30.821	3242254	66043	91.56
2	42.239	298836	5171	8.44
Total		3541090	71214	100

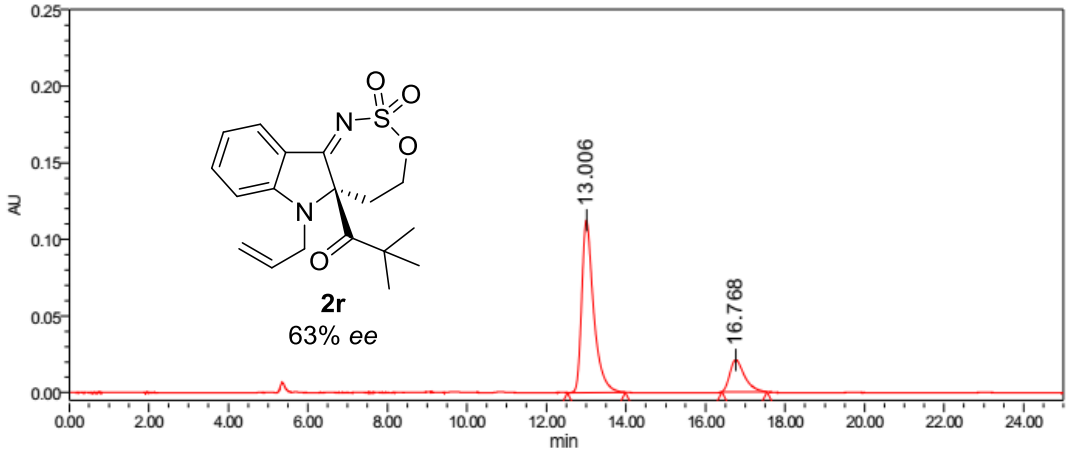
Condition: hexane : 2-propanol = 85:15

Flow rate = 1.0 mL/min, λ = 254 nm, Daicel Chiralpak AD-H.



PDA Ch1 254nm

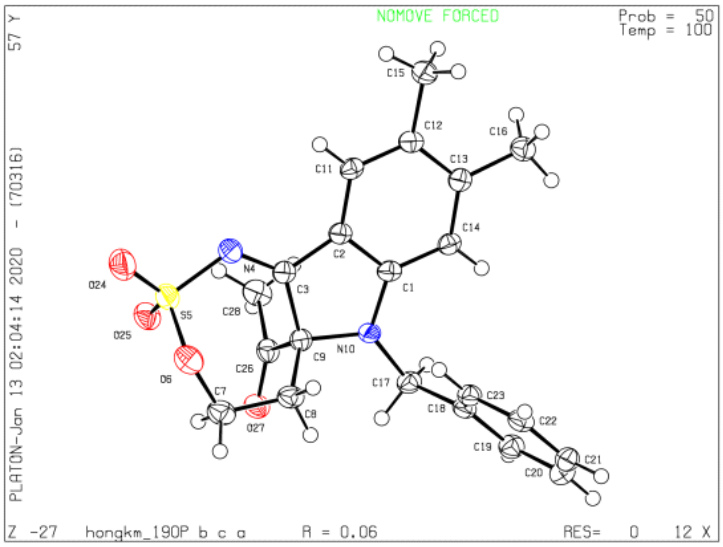
Peak#	Ret. Time	Area	Height	Area%
1	13.540	2772193	128275	50.08
2	17.397	2762888	97523	49.92
Total		5535081	225798	100

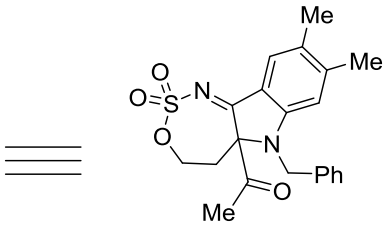


PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	13.006	2343745	112504	81.50
2	16.768	531993	20864	18.50
Total		2875738	133368	100

Crystallographic Data for 2n.





2n

CCDC: 1957861

Datablock: hongkm_190926

Bond precision: C-C = 0.0030 A Wavelength=1.54184

Cell: a=13.8567(3) b=14.0776(3) c=19.5063(3)
alpha=90 beta=90 gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	3805.08(13)	3805.06(13)
Space group	P b c a	P b c a
Hall group	-P 2ac 2ab	-P 2ac 2ab
Moiety formula	C21 H22 N2 O4 S	C21 H22 N2 O4 S
Sum formula	C21 H22 N2 O4 S	C21 H22 N2 O4 S
Mr	398.47	398.46
Dx,g cm-3	1.391	1.391
Z	8	8
Mu (mm-1)	1.773	1.773
F000	1680.0	1680.0
F000'	1687.54	
h,k,lmax	17,17,24	17,17,24
Nref	4138	3840
Tmin,Tmax	0.737,0.701	0.405,1.000
Tmin'	0.668	

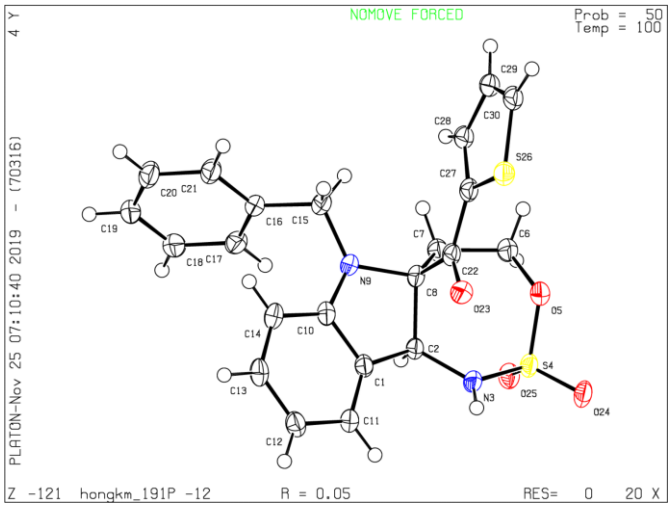
Correction method= # Reported T Limits: Tmin=0.405 Tmax=1.000
AbsCorr = MULTI-SCAN

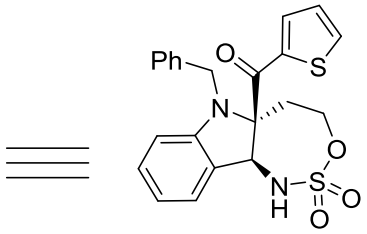
Data completeness= 0.928 Theta(max)= 79.616

R(reflections)= 0.0592(3368) wR2(reflections)= 0.1650(3840)

S = 1.067 Npar= 256

Crystallographic Data for 6.





6

CCDC 1968979

Datablock: hongkm_191118_2

Bond precision: C-C = 0.0032 A Wavelength=1.54184

Cell: a=9.7354(3) b=10.1100(4) c=10.7275(3)
alpha=99.198(3) beta=96.818(2) gamma=107.990(3)
Temperature: 100 K

	Calculated	Reported
Volume	975.31(6)	975.30(6)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C22 H20 N2 O4 S2	C22 H20 N2 O4 S2
Sum formula	C22 H20 N2 O4 S2	C22 H20 N2 O4 S2
Mr	440.52	440.52
Dx,g cm-3	1.500	1.500
Z	2	2
Mu (mm-1)	2.767	2.767
F000	460.0	460.0
F000'	462.59	
h,k,lmax	12,12,13	12,12,13
Nref	4137	3986
Tmin,Tmax	0.847,0.871	0.872,1.000
Tmin'	0.758	

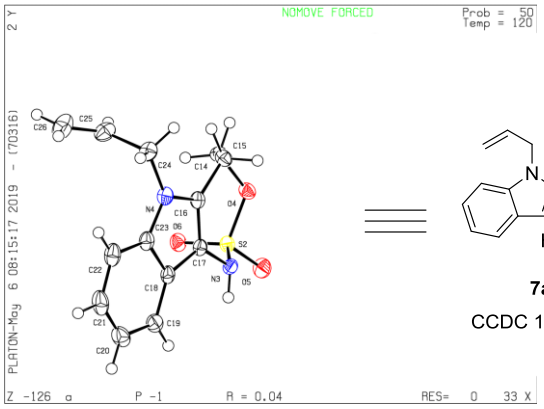
Correction method= # Reported T Limits: Tmin=0.872 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.964 Theta(max)= 77.202

R(reflections)= 0.0457(3438) wR2(reflections)= 0.1307(3986)

S = 1.098 Npar= 272

Crystallographic Data for 7a.



Datablock: a

```
Bond precision:  C-C = 0.0031 A          Wavelength=0.71075

Cell:         a=9.6651(3)        b=10.7492(4)      c=13.2113(4)
              alpha=93.4901(10)  beta=98.4723(10)  gamma=106.3564(10)
Temperature: 120 K
```

	Calculated	Reported
Volume	1295.03(7)	1295.03(7)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C13 H14 N2 O3 S	C13 H14 N2 O3 S
Sum formula	C13 H14 N2 O3 S	C13 H14 N2 O3 S
Mr	278.32	278.32
Dx,g cm-3	1.428	1.428
Z	4	4
Mu (mm-1)	0.255	0.255
F000	584.0	584.0
F000'	584.75	
h,k,lmax	12,13,17	12,13,17
Nref	5928	5920
Tmin,Tmax	0.955,0.975	0.673,0.746
Tmin'	0.950	

```
Correction method= # Reported T Limits: Tmin=0.673 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.999          Theta(max)= 27.466

R(reflections)= 0.0428( 4914)     wR2(reflections)= 0.1326( 5920)

S = 1.084                  Npar= 343
```
