Supporting Information

Organophotoredox-Catalyzed C(sp^2)-N Cross Coupling Reaction of Cyclic Aldimines with Cyclic Aliphatic Amines

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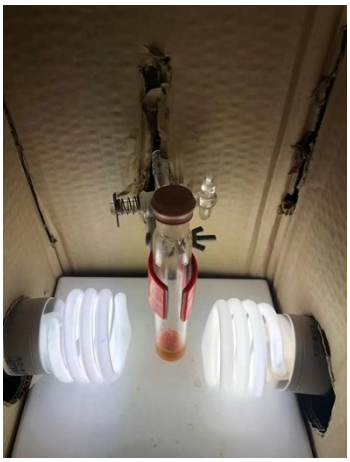
General information:

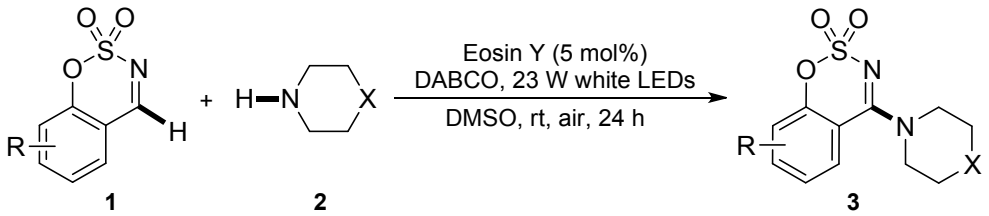
^{1}H, and ^{13}C were recorded at Bruker 400 MHz (^{1}H NMR) and 100 MHz (^{13}C NMR). Chemical shifts were reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm, 77.0 ppm). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br (broad). Coupling constants were reported in Hertz (Hz). Infrared spectra were obtained with a AVATAR 360 FT-IR spectrometer. Melting points were measured with a XT-4 melting point apparatus without correction. X-ray structural analysis was conducted on the XtaLAB mini. The high resolution ESI-MS spectra were obtained with a Bruker APEX IV Fourier transformmass spectrometer.

Materials: All commercially available reagents and solvent were used without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel plates. Silica gel (200-300 mesh) was used for flash chromatography. Cyclic *N*-sulfimines were prepared according to the literatures.[1]

The photos of the reaction set-up：

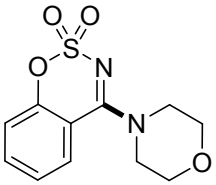

General Procedure for the visible light-induced organophotocatalyzed C(sp^2)–N Cross Coupling Reactions of Cyclic Aldimines with Cyclic Aliphatic Amines:



1 2 3

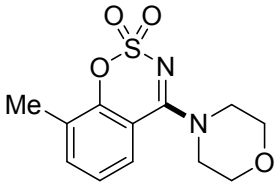
To a 15 mL Schlenk charged with cyclic aldimines **1** (0.2 mmol), Eosin Y (6.5 mg, 0.01 mmol), DABCO (45 mg, 0.4 mmol) and cyclic aliphatic amines **2** (0.4 mmol) was added DSMO (2.0 mL) *via* a syringe. Then, the reaction mixture was open to the air and vigorously stirred under the irradiation of 23 W white LEDs at room temperature for 24 h. After the reaction was complete, the mixture was diluted with water (10 mL) and ethyl acetate (10 mL). The organic layer was washed with saturated brine (3 × 10 mL), dried anhydrous $MgSO_4$, and then concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent) to afford the desired products **3**.

4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3aa):



83% yield, mp 192–193 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.81–3.84 (m, 4H), 3.87–3.89 (m, 4H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.54 (dd, *J* = 8.0 Hz, 1.2 Hz, 1H), 7.64 (dt, *J* = 8.4 Hz, 1.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.1, 154.5, 135.3, 128.0, 124.8, 120.7, 112.8, 66.4, 49.1; **IR** (KBr) ν 2923, 2862, 1588, 1538, 1359, 1197, 1114, 1027, 878 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{13}N_2O_4S$ $[M+H]^+$ 269.0591, found 269.0977.

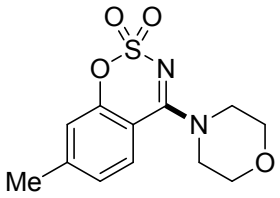
8-methyl-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ba):



65% yield, mp 172–173 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.37 (s, 3H), 3.78–3.80

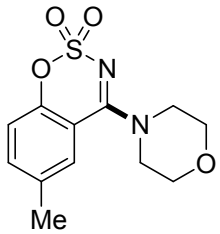
(m, 4H), 3.83–3.86 (m, 4H), 7.20 (t, J = 7.6 Hz, 1H), 7.36 (d, J = 8.0 Hz, 1H), 7.47 (d, J = 7.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.6, 152.6, 136.5, 130.3, 125.5, 124.1, 112.4, 66.3, 49.2, 15.2; **IR** (KBr) ν 2922, 2861, 1594, 1539, 1444, 1361, 1275, 1159, 1116, 879 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_4S$ $[M+H]^+$ 283.0747, found 283.1131.

7-methyl-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ca):



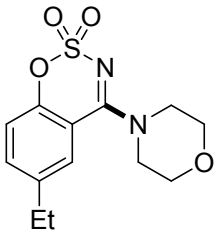
73% yield, mp 210–211 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.44 (s, 3H), 3.79–3.81 (m, 4H), 3.84–3.86 (m, 4H), 7.11 (d, J = 8.4 Hz, 1H), 7.14 (s, 1H), 7.42 (d, J = 8.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.1, 154.4, 147.3, 127.8, 125.8, 120.7, 110.1, 66.3, 49.1, 21.7; **IR** (KBr) ν 2922, 2862, 1618, 1579, 1528, 1450, 1355, 1269, 1168, 1109, 1029, 946, 865 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_4S$ $[M+H]^+$ 283.0747, found 283.1131.

6-methyl-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3da):



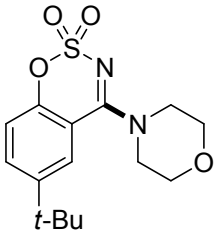
58% yield, mp 224–225 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.42 (s, 3H), 3.82–3.85 (m, 4H), 3.88–3.91 (m, 4H), 7.28 (s, 1H), 7.82 (d, J = 1.2 Hz, 1H), 7.46 (dd, J = 8.4 Hz, 2.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.2, 152.4, 136.1, 134.8, 127.9, 120.4, 112.5, 66.4, 49.1, 21.0; **IR** (KBr) ν 2920, 2861, 1539, 1482, 1357, 1270, 1168, 1114, 881, 830 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_4S$ $[M+H]^+$ 283.0747, found 283.1131.

6-ethyl-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ea):



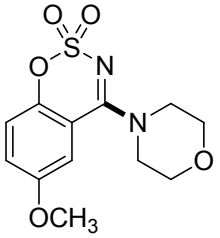
63% yield, mp 167–168 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.25 (d, *J* = 7.6 Hz, 3H), 2.69 (q, *J* = 7.6 Hz, 2H), 3.80–3.83 (m, 4H), 3.86–3.89 (m, 4H), 7.26 (d, *J* = 8.4 Hz, 1H), 7.32 (d, *J* = 2.0 Hz, 1H), 7.46 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.3, 152.4, 141.0, 135.0, 126.8, 120.4, 112.5, 66.3, 49.1, 28.2, 15.3; **IR** (KBr) ν 2975, 2922, 1590, 1538, 1452, 1359, 1272, 1190, 1108, 1028, 880 cm^{-1}; **HRMS** (ESI): calcd for $C_{13}H_{17}N_2O_4S$ $[M+H]^+$ 297.0904, found 297.1301.

6-(tert-butyl)-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3fa):



81% yield, mp 246–247 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.33 (s, 9H), 3.82–3.85 (m, 4H), 3.86–3.89 (m, 4H), 7.28 (d, *J* = 8.8 Hz, 1H), 7.49 (d, *J* = 2.4 Hz, 1H), 7.67 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.6, 152.2, 148.0, 132.8, 124.6, 120.0, 111.9, 66.4, 49.3, 34.7, 31.1; **IR** (KBr) ν 2921, 2863, 1637, 1539, 1364, 1266, 1196, 1116, 1028, 883 cm^{-1}; **HRMS** (ESI): calcd for $C_{15}H_{21}N_2O_4S$ $[M+H]^+$ 325.1217, found 325.1642.

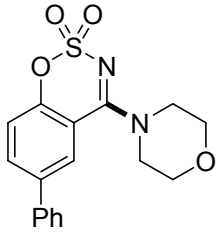
6-methoxy-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ga):



55% yield, mp 203–204 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.80–3.84 (m, 7H), 3.87–3.90 (m, 4H), 6.98 (d, *J* = 3.2 Hz, 1H), 7.17 (dd, *J* = 8.4 Hz, 2.8 Hz, 1H), 7.30 (d, *J* = 9.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.1, 156.0, 147.9, 121.5, 120.5, 113.2, 113.0, 66.3, 56.0, 49.1; **IR** (KBr) ν 2921, 2820, 1622, 1543, 1486, 1343, 1275, 1166,

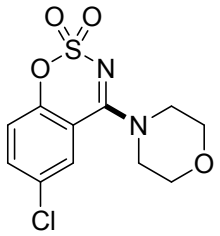
1025, 830, 750 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_5S$ $[M+H]^+$ 299.0696, found 299.1113.

4-morpholino-6-phenylbenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ha):



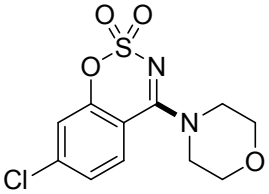
38% yield, mp 219–220 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.82–3.86 (m, 4H), 3.89–3.94 (m, 4H), 7.41–7.46 (m, 2H), 7.47–7.51 (m, 4H), 6.67 (d, *J* = 2.0 Hz, 1H), 7.83 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.1, 153.6, 138.6, 138.5, 134.1, 129.2, 128.4, 126.9, 126.4, 121.0, 113.0, 66.4, 49.0; **IR** (KBr) ν 2920, 2823, 1645, 1542, 1470, 1362, 1195, 1114, 1026, 879 cm^{-1}; **HRMS** (ESI): calcd for $C_{17}H_{17}N_2O_4S$ $[M+H]^+$ 345.0904, found 345.1356.

6-chloro-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ia):



65% yield, mp 197–198 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.16–3.18 (m, 4H), 3.76–3.79 (m, 4H), 6.74 (d, *J* = 8.4 Hz, 1H), 7.12 (d, *J* = 2.4 Hz, 1H), 7.18 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.2, 151.3, 131.7, 127.8, 125.7, 121.6, 119.6, 66.0, 49.6; **IR** (KBr) ν 2923, 2821, 1632, 1542, 1469, 1128, 1110, 870 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{12}ClN_2O_4S$ $[M+H]^+$ 303.0201, found 303.0604.

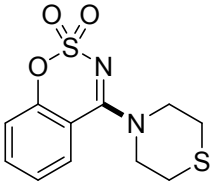
7-chloro-4-morpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ja):



72% yield, mp 244–245 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.81–3.84 (m, 4H), 3.85–3.88 (m, 4H), 7.30 (dd, 8.4 Hz, 2.0 Hz, 1H), 7.38 (d, *J* = 2.0 Hz, 1H), 7.47 (d, *J* = 8.8

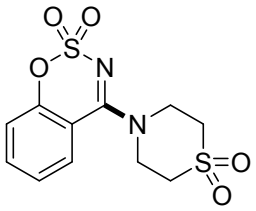
Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.3, 155.0, 141.4, 128.8, 125.4, 121.1, 111.3, 66.3, 49.2; **IR** (KBr) ν 2922, 2821, 1604, 1579, 1532, 1461, 1363, 1196, 1114, 868 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{12}ClN_2O_4S$ $[M+H]^+$ 303.0201, found 303.0604.

4-thiomorpholinobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ab):



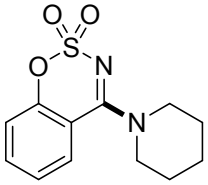
65% yield, mp 203–204 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.81–2.84 (m, 4H), 4.07–4.10 (m, 4H), 7.31–7.37 (m, 2H), 7.52 (dd, 8.0 Hz, 1.2 Hz, 1H), 7.64 (dt, 8.8 Hz, 2.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.7, 154.5, 135.3, 127.9, 124.9, 120.7, 113.0, 51.4, 27.3; **IR** (KBr) ν 2920, 2820, 1610, 1587, 1532, 1467, 1360, 1259, 1188, 955, 863 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{13}N_2O_3S_2$ $[M+H]^+$ 285.0362, found 285.0749.

4-(1,1-dioxidothiomorpholino)benzo[e][1,2,3]oxathiazine 2,2-dioxide (3ac):



85% yield, mp 252–253 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 3.40–3.53 (m, 4H), 4.07–4.21 (m, 4H), 7.47 (t, 8.0 Hz, 1H), 7.57 (d, 8.4 Hz, 1H), 7.80 (dt, 8.4 Hz, 1.2 Hz, 1H), 7.86 (dd, 8.0 Hz, 1.2 Hz, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.3, 153.3, 135.9, 129.1, 125.5, 119.9, 112.7, 50.3, 46.6; **IR** (KBr) ν 2923, 2820, 1583, 1523, 1452, 1362, 1190, 1124, 1040, 953, 753 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{13}N_2O_5S_2$ $[M+H]^+$ 317.0260, found 317.0691.

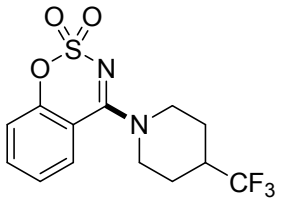
4-(piperidin-1-yl)benzo[e][1,2,3]oxathiazine 2,2-dioxide (3ad):



45% yield, mp 127–128 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.70–1.81 (m, 6H), 3.71–

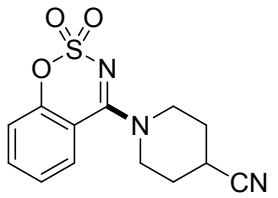
3.82 (m, 4H), 7.28–7.33 (m, 2H), 7.86 (dd, 8.0 Hz, 2.0 Hz, 1H), 7.60 (dt, 8.8 Hz, 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.6, 154.3, 134.9, 128.2, 120.4, 49.6, 25.8, 24.0; **IR** (KBr) ν 2928, 2826, 1637, 1585, 1539, 1467, 1356, 1265, 1185, 1099, 869, 750 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_3S$ $[M+H]^+$ 267.0798, found 267.1150.

4-(4-(trifluoromethyl)piperidin-1-yl)benzo[e][1,2,3]oxathiazine 2,2-dioxide (3ae):



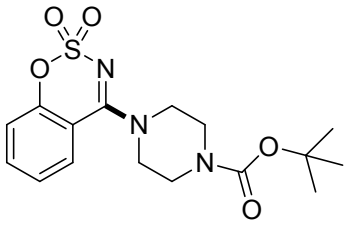
85% yield, mp 201–202 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.73–1.84 (m, 2H), 2.07 (d, 12.8 Hz, 2H), 1.37–2.51 (m, 1H), 3.18 (t, 12.8 Hz, 2H), 4.50 (d, 13.2 Hz, 2H), 7.32–7.37 (m, 2H), 7.54 (d, 7.6 Hz, 1H), 7.65 (t, 7.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.5, 154.4, 135.4, 128.0, 126.6 (q, $J_{C\text{-}F}$ = 276.7 Hz), 124.9, 120.6, 112.9, 47.7, 40.1 (q, $J_{C\text{-}F}$ = 27.9 Hz), 24.4; **IR** (KBr) ν 2918, 2819, 1611, 1588, 1537, 1470, 1363, 1210, 1105, 866, 747 cm^{-1}; **HRMS** (ESI): calcd for $C_{13}H_{14}F_3N_2O_3S$ $[M+H]^+$ 335.0672, found 335.1115.

1-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)piperidine-4-carbonitrile (3af):



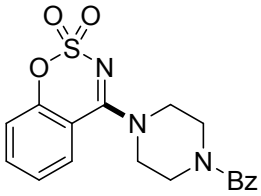
40% yield, mp 189–190 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.00–2.15 (m, 4H), 3.02–3.08 (m, 1H), 3.84–3.90 (m, 2H), 3.93–3.99 (m, 2H), 7.32–7.38 (m, 2H), 7.52 (dd, 8.0 Hz, 1.2 Hz, 1H), 7.66 (dt, 8.8 Hz, 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.6, 154.4, 135.5, 127.9, 125.0, 120.7, 120.0, 112.7, 46.7, 28.3, 26.0; **IR** (KBr) ν 2923, 2821, 1637, 1596, 1510, 1260, 1118, 1089, 799 cm^{-1}; **HRMS** (ESI): calcd for $C_{13}H_{14}N_3O_3S$ $[M+H]^+$ 292.0750, found 292.1138.

tert-butyl 4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)piperazine-1-carboxylate (3ag):



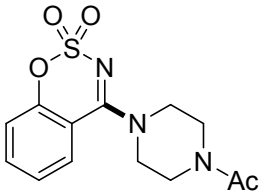
60% yield, mp 171–172 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.47 (s, 9H), 3.58–3.61 (m, 4H), 3.80–3.83 (m, 4H), 7.31–7.36 (m, 2H), 7.54 (dd, 8.0 Hz, 1.6 Hz, 1H), 7.64 (dt, 8.8 Hz, 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.4, 154.4, 154.3, 135.4, 128.0, 124.9, 120.6, 112.9, 80.8, 48.6, 43.1, 28.2; **IR** (KBr) ν 2925, 2824, 1693, 1611, 1588, 1534, 1467, 1421, 1365, 1251, 1162, 1017, 870, 749 cm^{-1}; **HRMS** (ESI): calcd for $C_{16}H_{22}N_3O_5S$ $[M+H]^+$ 368.1275, found 368.1687.

(4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)piperazin-1-yl)(phenyl)methanone (3ah):



65% yield, mp 119–120 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.58–4.04 (m, 8H), 7.32–7.38 (m, 2H), 7.42–7.49 (m, 5H), 7.55 (dd, 8.0 Hz, 1.2 Hz, 1H), 7.66 (dt, 8.8 Hz, 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.6, 163.6, 154.4, 135.6, 134.5, 130.4, 128.7, 128.0, 127.1, 125.0, 120.6, 112.7, 48.7, 41.9; **IR** (KBr) ν 2920, 2850, 1633, 1532, 1464, 1427, 1362, 1264, 1192, 1005, 862, 710 cm^{-1}; **HRMS** (ESI): calcd for $C_{18}H_{18}N_3O_4S$ $[M+H]^+$ 372.1013, found 372.1508.

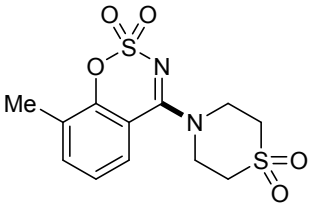
1-(4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)piperazin-1-yl)ethanone (3ai):



50% yield, mp 107–108 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.16 (s, 3H), 3.66–3.69 (m, 2H), 3.76–3.79 (m, 2H), 3.83–3.90 (m, 4H), 7.33–7.38 (m, 2H), 7.56 (dd, 8.0 Hz, 1.6 Hz, 1H), 7.67 (dt, 8.4 Hz, 1.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.3, 163.6, 154.5, 135.6, 128.0, 125.0, 120.7, 112.8, 45.2, 41.0, 21.2; **IR** (KBr) ν 2925,

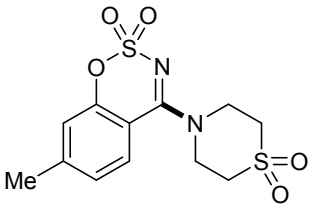
2823, 1645, 1587, 1534, 1425, 1357, 1275, 1190, 856, 750 cm^{-1}; **HRMS** (ESI): calcd for $C_{13}H_{16}N_3O_4S$ $[M+H]^+$ 310.0856, found 310.1267.

4-(1,1-dioxidothiomorpholino)-8-methylbenzo[e][1,2,3]oxathiazine 2,2-dioxide (3bd):



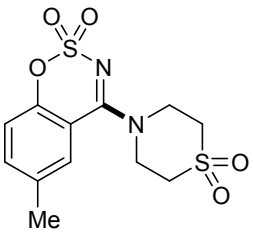
77% yield, mp 300–301 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 2.31 (s, 3H), 3.46 (s, 4H), 4.13 (s, 4H), 7.37 (t, 7.6 Hz, 1H), 7.68 (d, 7.6 Hz, 2H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.8, 151.5, 136.8, 128.8, 126.6, 124.6, 112.5, 50.3, 46.8, 14.6; **IR** (KBr) ν 2928, 1636, 1588, 1455, 1338, 1082, 881, 755 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_5S_2$ $[M+H]^+$ 331.0417, found 331.0850.

4-(1,1-dioxidothiomorpholino)-7-methylbenzo[e][1,2,3]oxathiazine 2,2-dioxide (3cd):



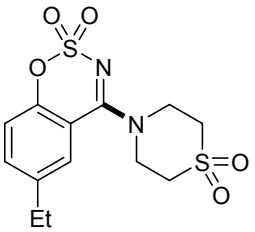
80% yield, mp 280–281 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 2.43 (s, 3H), 3.45 (s, 4H), 4.13 (s, 4H), 7.28 (d, 7.6 Hz, 1H), 7.35 (s, 1H), 7.73 (d, 7.6 Hz, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.4, 153.4, 147.5, 128.9, 126.2, 119.9, 110.0, 50.3, 46.4, 21.1; **IR** (KBr) ν 2922, 1615, 1578, 1453, 1359, 1191, 1122, 863, 763 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_5S_2$ $[M+H]^+$ 331.0417, found 331.0874.

4-(1,1-dioxidothiomorpholino)-6-methylbenzo[e][1,2,3]oxathiazine 2,2-dioxide (3dd):



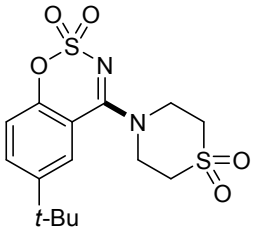
90% yield, mp 307–308 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 2.40 (s, 3H), 3.46 (s, 4H), 4.14 (s, 4H), 7.41 (d, 8.4 Hz, 1H), 7.60 (d, 8.4 Hz, 1H), 7.64 (s, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.5, 151.3, 136.4, 135.1, 128.8, 119.6, 112.4, 50.3, 46.7, 20.3; **IR** (KBr) ν 2952, 1590, 1526, 1474, 1355, 1293, 1123, 1040, 860, 749 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_5S_2$ $[M+H]^+$ 331.0417, found 331.0874.

4-(1,1-dioxidothiomorpholino)-6-ethylbenzo[e][1,2,3]oxathiazine 2,2-dioxide (3ed):



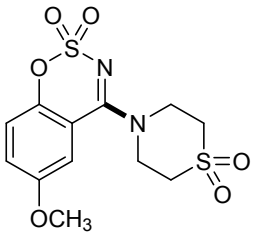
85% yield, mp 309–310 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 1.22 (t, 7.6 Hz, 3H), 2.71 (q, 14.8 Hz, 2H), 3.47 (s, 4H), 4.14 (s, 4H), 7.43 (d, 8.8 Hz, 1H), 7.58–7.75 (m, 2H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.6, 151.4, 141.2, 135.4, 127.8, 119.7, 112.4, 50.3, 46.9, 27.3, 15.3; **IR** (KBr) ν 2923, 1638, 1531, 1485, 1355, 1175, 1124, 880 cm^{-1}; **HRMS** (ESI): calcd for $C_{13}H_{17}N_2O_5S_2$ $[M+H]^+$ 345.0573, found 345.1026.

6-(tert-butyl)-4-(1,1-dioxidothiomorpholino)benzo[e][1,2,3]oxathiazine 2,2-dioxide (3fd):



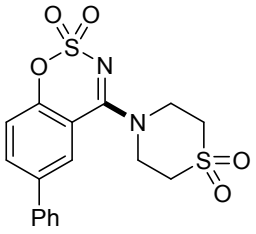
87% yield, mp 266–267 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 1.33 (s, 9H), 3.50 (s, 4H), 4.15 (s, 4H), 7.44 (d, 8.8 Hz, 1H), 7.72 (d, 2.4 Hz, 1H), 7.85 (dd, 8.4 Hz, 2.0 Hz, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.7, 151.3, 147.8, 133.2, 125.4, 119.4, 111.9, 50.2, 46.6, 34.6, 30.7; **IR** (KBr) ν 2920, 1644, 1533, 1471, 1365, 1189, 1124, 1028, 858 cm^{-1}; **HRMS** (ESI): calcd for $C_{15}H_{21}N_2O_5S_2$ $[M+H]^+$ 373.0886, found 373.1372.

4-(1,1-dioxidothiomorpholino)-6-methoxybenzo[e][1,2,3]oxathiazine 2,2-dioxide (3gd):



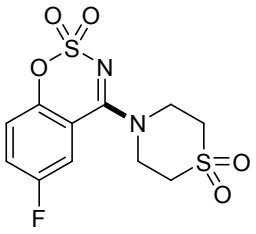
78% yield, mp 280–281 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 3.46 (s, 4H), 3.85 (s, 3H), 4.16 (s, 4H), 7.28 (d, 2.8 Hz, 1H), 7.38 (dd, 8.8 Hz, 2.8 Hz, 1H), 7.47 (d, 9.2 Hz, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.4, 155.7, 146.9, 122.2, 120.9, 113.1, 112.6, 56.0, 50.2, 46.7; **IR** (KBr) ν 2928, 1637, 1531, 1360, 1184, 1123, 1038, 858 cm^{-1}; **HRMS** (ESI): calcd for $C_{12}H_{15}N_2O_6S_2$ $[M+H]^+$ 347.0366, found 347.0816.

4-(1,1-dioxidothiomorpholino)-6-phenylbenzo[e][1,2,3]oxathiazine 2,2-dioxide (3hd):



72% yield, mp 308–309 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 3.50 (s, 4H), 4.21 (s, 4H), 7.44 (t, 7.2 Hz, 1H), 7.53 (t, 7.2 Hz, 2H), 7.62 (d, 8.8 Hz, 1H), 7.76 (d, 7.6 Hz, 2H), 8.08 (t, 8.4 Hz, 2H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.3, 152.7, 137.9, 137.4, 134.0, 129.1, 128.2, 127.0, 120.5, 113.1, 50.2, 46.6; **IR** (KBr) ν 2926, 2821, 1644, 1598, 1455, 1120, 1021, 867 cm^{-1}; **HRMS** (ESI): calcd for $C_{17}H_{17}N_2O_5S_2$ $[M+H]^+$ 393.0573, found 393.1084.

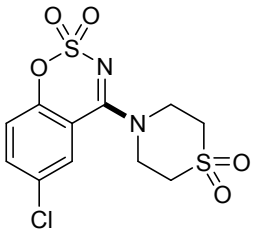
4-(1,1-dioxidothiomorpholino)-6-fluorobenzo[e][1,2,3]oxathiazine 2,2-dioxide (3id):



95% yield, mp 257–258 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 3.46 (s, 4H), 4.15 (s, 4H), 7.63 (dd, 8.8 Hz, 4.4 Hz, 1H), 7.70–7.75 (m, 2H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 162.5, 157.9 (q, $J_{C\text{-}F}$ = 242.5 Hz), 149.5, 123.0 (q, $J_{C\text{-}F}$ = 23.7 Hz), 122.0 (q, $J_{C\text{-}F}$

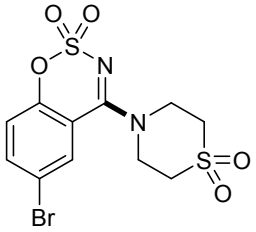
= 8.3 Hz), 115.7 (q, J_{C-F} = 26.3 Hz), 113.8 (q, J_{C-F} = 7.7 Hz), 50.2, 46.7; **IR** (KBr) ν 2923, 1610, 1532, 1453, 1373, 1163, 1123, 1041, 837 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{12}FN_2O_5S_2$ $[M+H]^+$ 335.0166, found 335.0604.

6-chloro-4-(1,1-dioxidothiomorpholino)benzo[e][1,2,3]oxathiazine 2,2-dioxide (3jd):



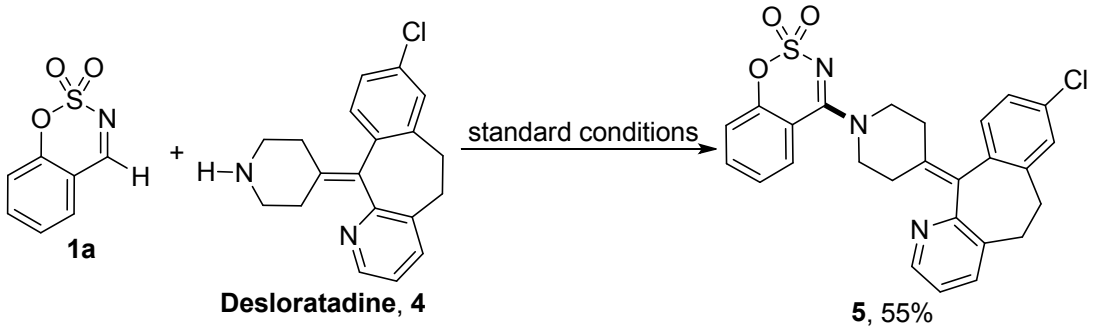
84% yield, mp 312–313 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 3.46 (s, 4H), 4.14 (s, 4H), 7.60 (d, 8.4 Hz, 1H), 7.89 (d, 8.4 Hz, 1H), 7.93 (s, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 162.2, 152.1, 135.6, 129.4, 128.5, 121.9, 114.2, 50.1, 46.4; **IR** (KBr) ν 2928, 2820, 1615, 1541, 1455, 1262, 1114, 1071, 943, 861, 733 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{12}ClN_2O_5S_2$ $[M+H]^+$ 350.9871, found 351.0328.

6-bromo-4-(1,1-dioxidothiomorpholino)benzo[e][1,2,3]oxathiazine 2,2-dioxide (3kd):



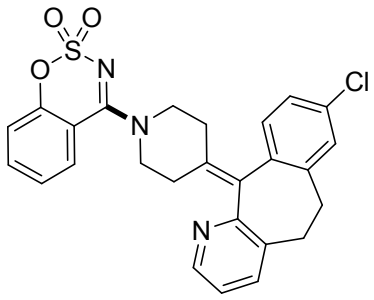
80% yield, mp 310–311 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 3.46 (s, 4H), 4.14 (s, 4H), 7.53 (d, 8.4 Hz, 1H), 8.00 (dd, 8.8 Hz, 2.4 Hz, 1H), 8.05 (d, 2.4 Hz, 1H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 162.1, 152.5, 138.4, 131.3, 122.1, 117.2, 114.6, 50.1, 46.6; **IR** (KBr) ν 2925, 1618, 1597, 1515, 1464, 1364, 1292, 1185, 889, 764 cm^{-1}; **HRMS** (ESI): calcd for $C_{11}H_{12}ClN_2O_5S_2$ $[M+H]^+$ 394.9366, found 394.9798.

The general procedure for the late-stage modification of drug:



To a 15 mL Schlenk charged with cyclic aldimines **1a** (0.3 mmol, 55 mg), Eosin Y (6.5 mg, 0.01 mmol), DABCO (45 mg, 0.4 mmol) and desloratadine **4** (0.2 mmol, 62.2 mg) was added DSMO (2.0 mL) *via* a syringe. Then, the reaction mixture was open to the air and vigorously stirred under the irradiation of 23 W white LEDs at room temperature for 24 h. After the reaction was complete, the mixture was diluted with water (10 mL) and ethyl acetate (10 mL). The organic layer was washed with saturated brine (3 × 10 mL), dried anhydrous $MgSO_4$, and then concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent) to afford the desired products **5**.

4-(4-(8-chloro-5H-benzo[5,6]cyclohepta[1,2-b]pyridin-11(6H)-ylidene)piperidin-1-yl)benzo[e][1,2,3]oxathiazine 2,2-dioxide (5):



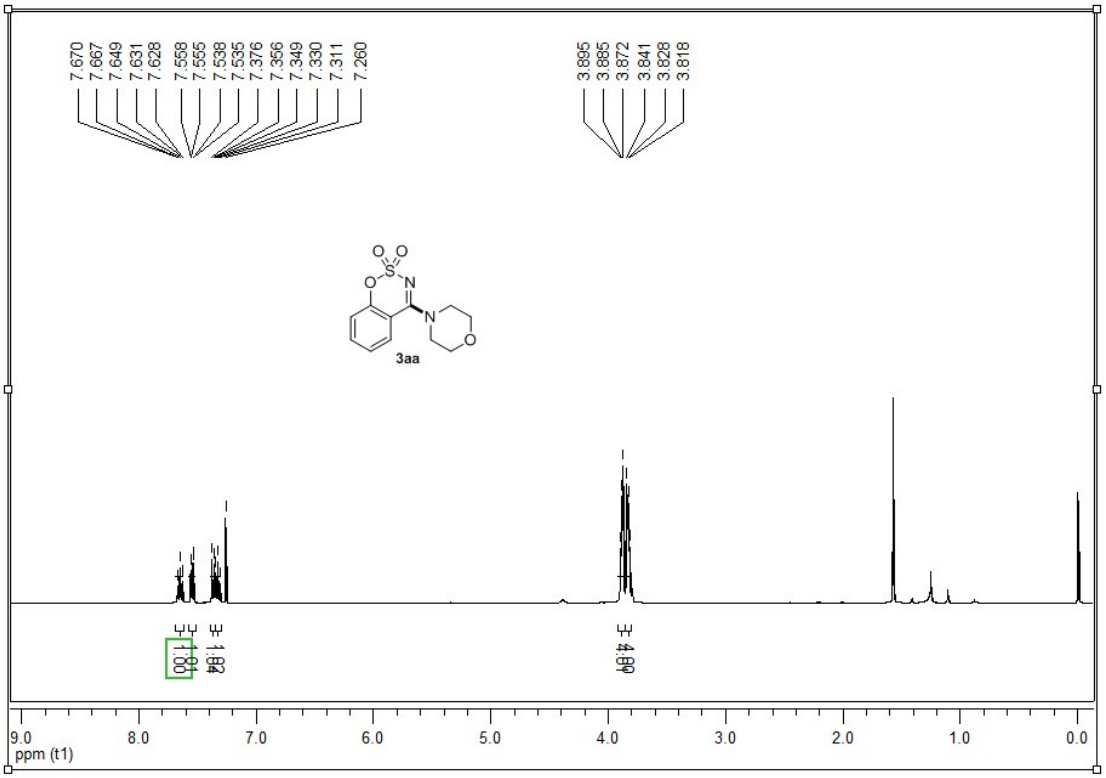
white solid; 55% yield, mp 144–145 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.48–2.53 (m, 2H), 2.61–2.68 (m, 1H), 2.78–2.92 (m, 3H), 3.29–3.42 (m, 2H), 3.56–3.62 (m, 2H), 4.03–4.12 (m, 2H), 7.10–7.17 (m, 3H), 7.19 (s, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.46 (d, *J* = 7.2 Hz, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 8.41 (d, *J* = 4.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.8, 156.2, 154.3, 146.6, 139.6, 137.8, 137.1, 135.7, 135.0, 134.6, 133.3, 133.2, 130.1, 129.0, 128.1, 126.2, 124.6, 122.5, 120.4, 113.2, 49.2, 31.5, 31.4, 30.1; **IR** (KBr) ν 2922, 1610, 1587,

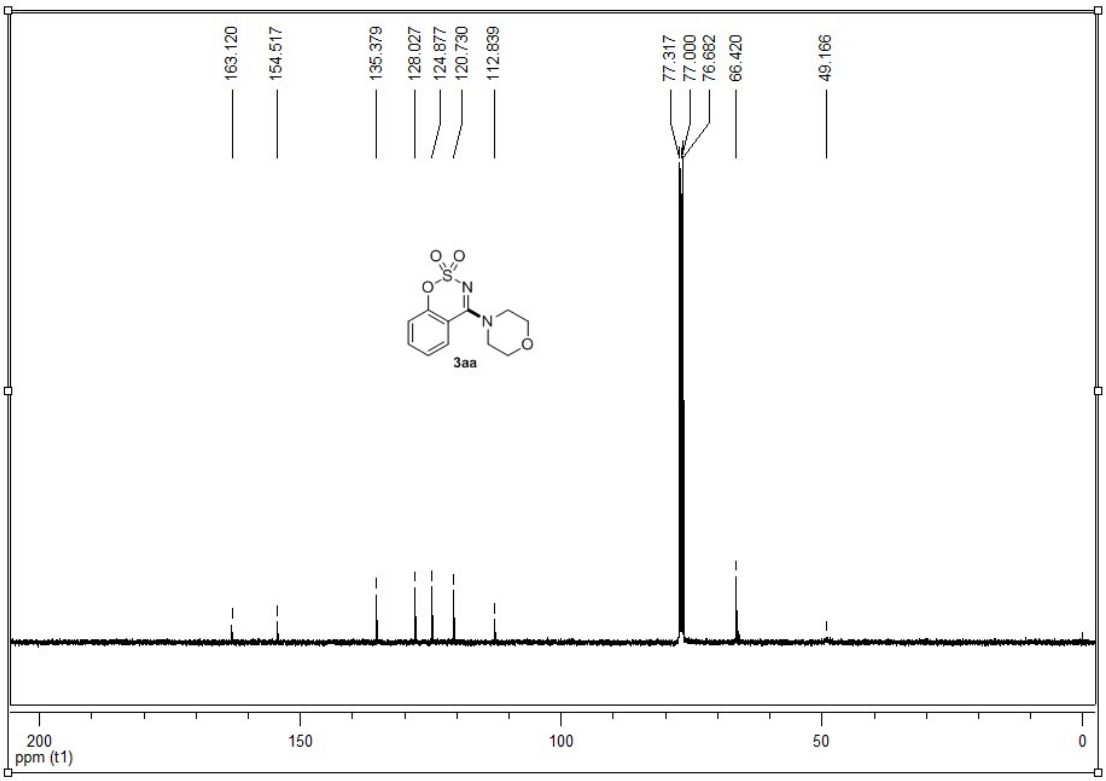
1533, 1469, 1363, 1278, 1190, 1109, 993, 861, 744 cm^{-1}; **HRMS** (ESI): calcd for $C_{26}H_{23}ClN_3O_3S$ $[M+H]^+$ 492.1143, found 492.1574.

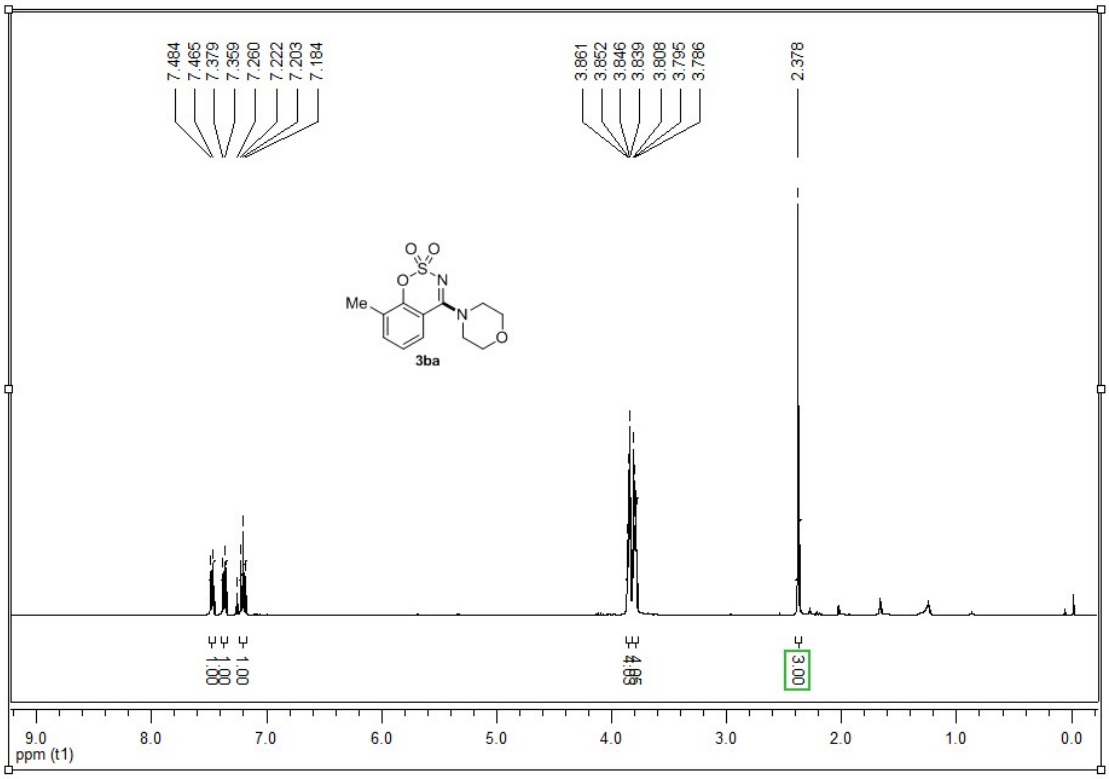
Reference:

[1] (a) N. D. Litvinas, B. H. Brodsky, J. D. Bois, *Angew. Chem. Int. Ed.*, **2009**, *48*, 4513; (b) H. Yu, L. Zhang, Z. Yang, Z. Li, Y. Zhao, Y. Xiao, H. Guo, *J. Org. Chem.*, **2013**, *78*, 8427.

Copies of NMR spectra of the products:



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7.359
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7.184
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3.808
3.795
3.786
2.378
Me
3ba
1.00
1.08
1.00
4.06
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
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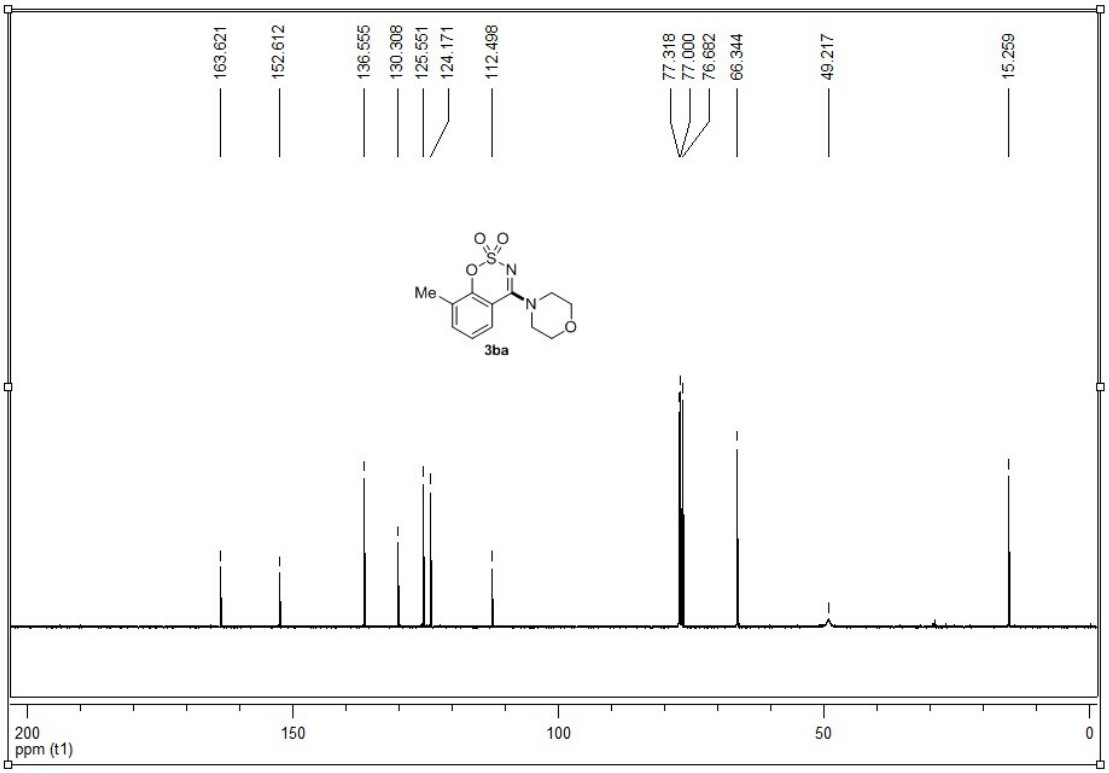
0.0
ppm (t1)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Me
3ba
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50
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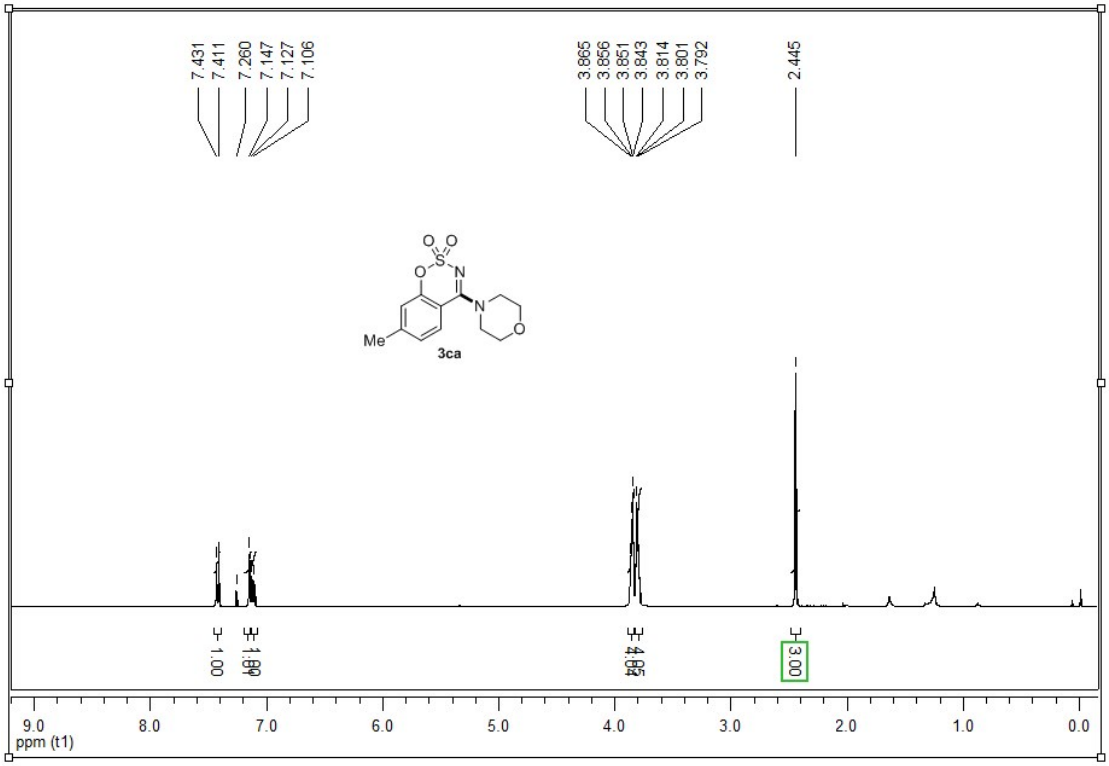
ppm (t1)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2.445
3ca
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1.89
4.66
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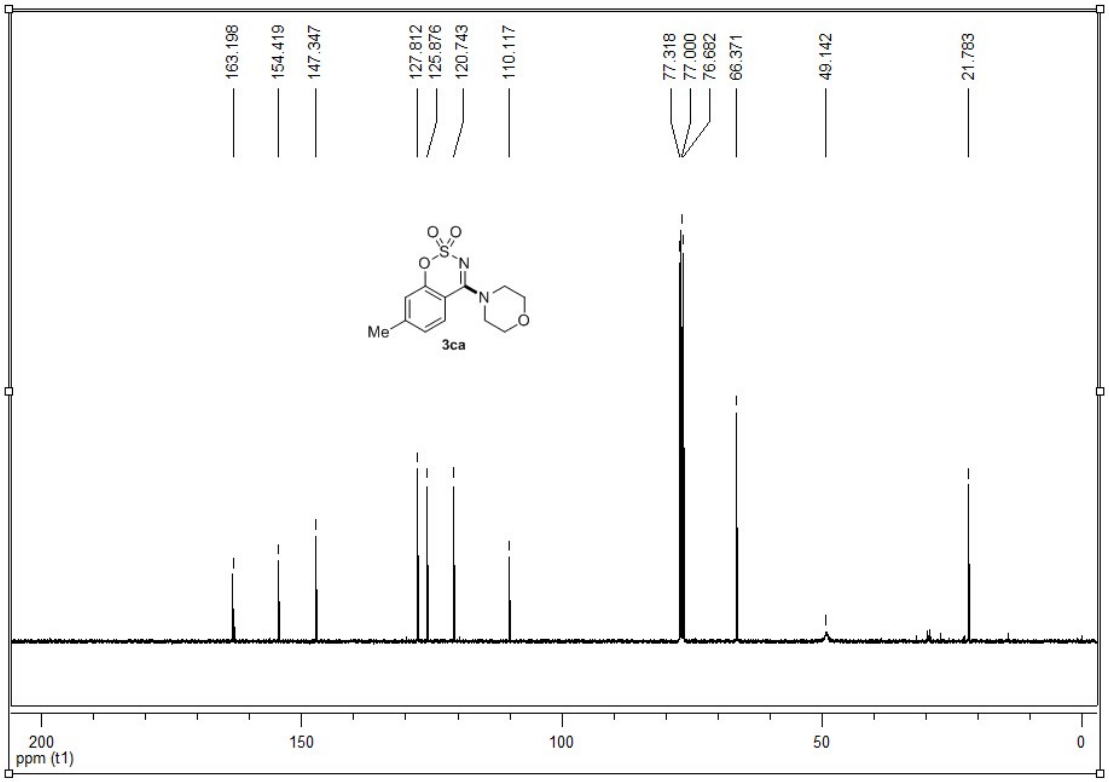
ppm (t1)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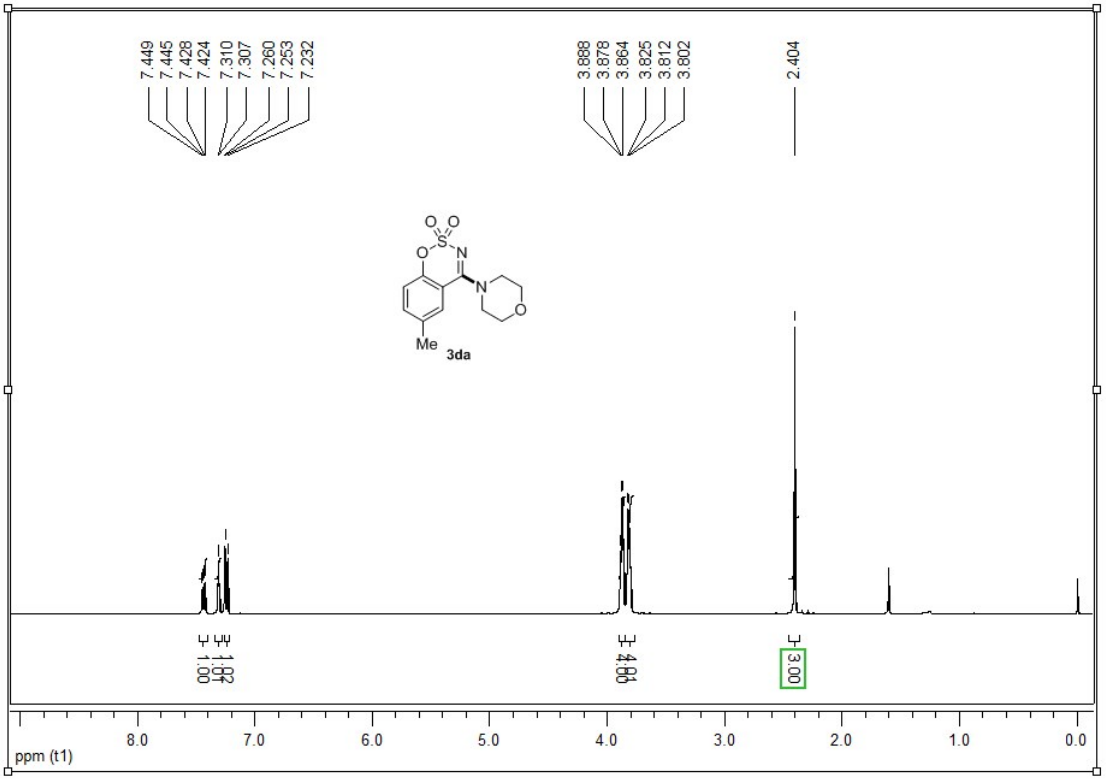
ppm (t1)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3.802
2.404
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1.00
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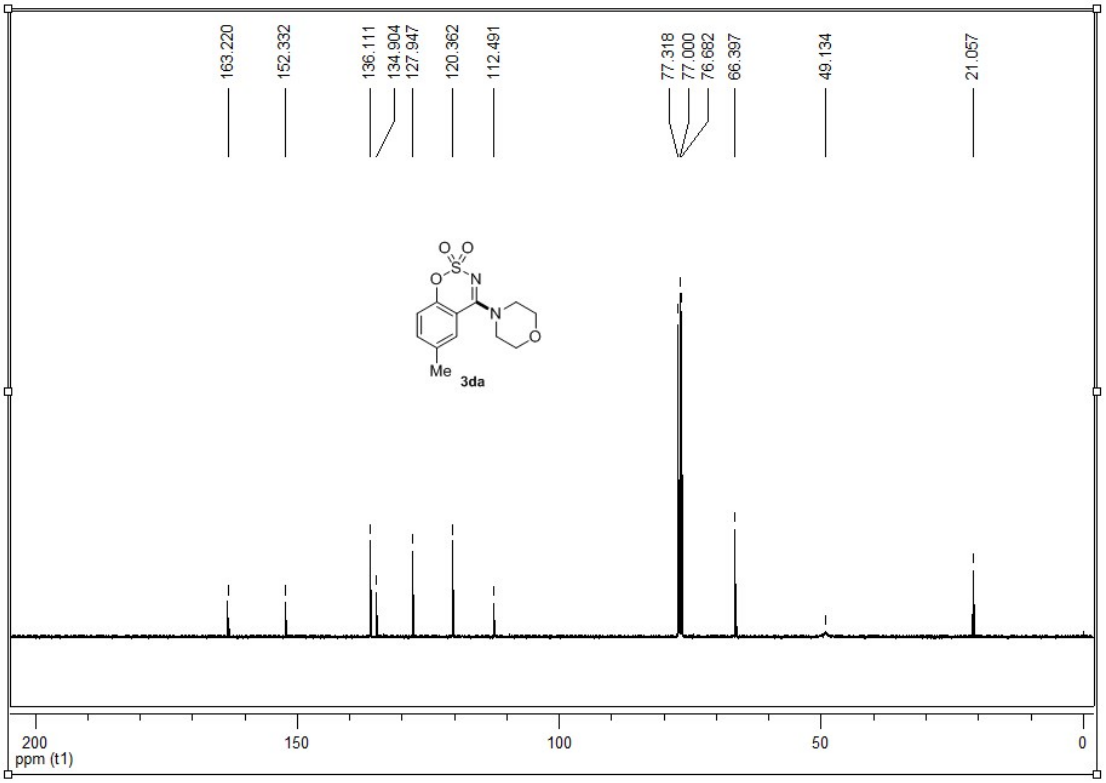
4.08
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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152.332
136.111
134.904
127.947
120.362
112.491
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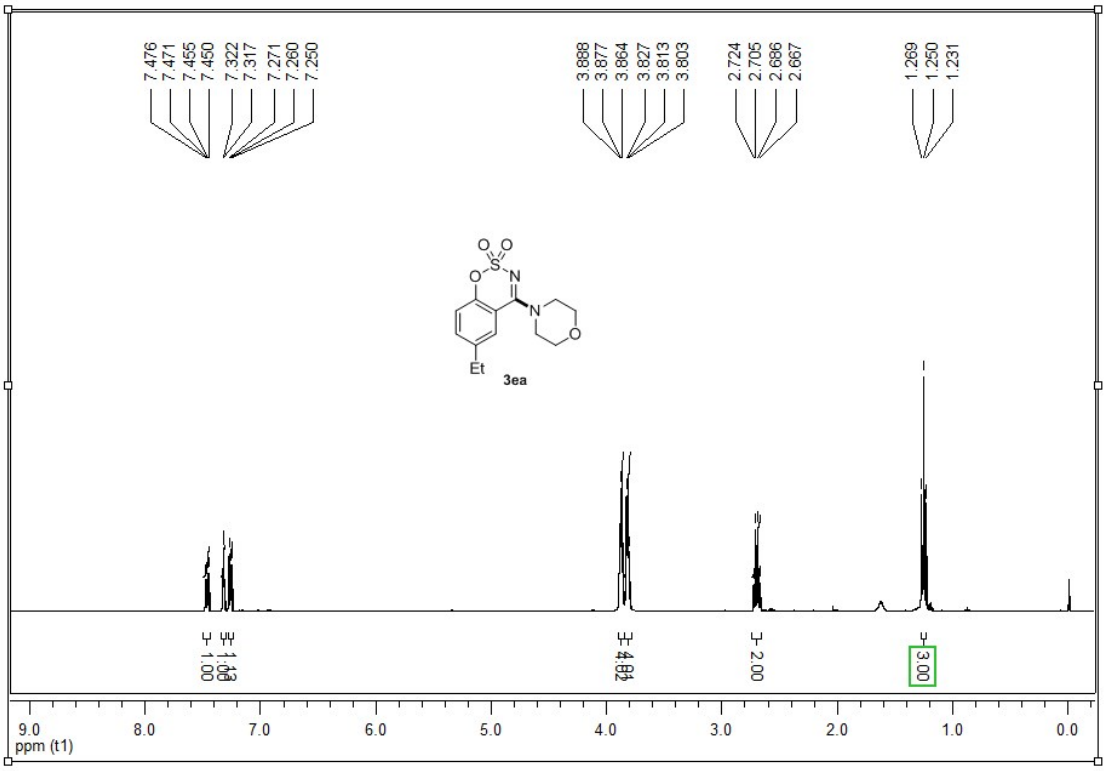
77.000
76.682
66.397
49.134
21.057
Me
3da
200
150
100
50
0
ppm (t1)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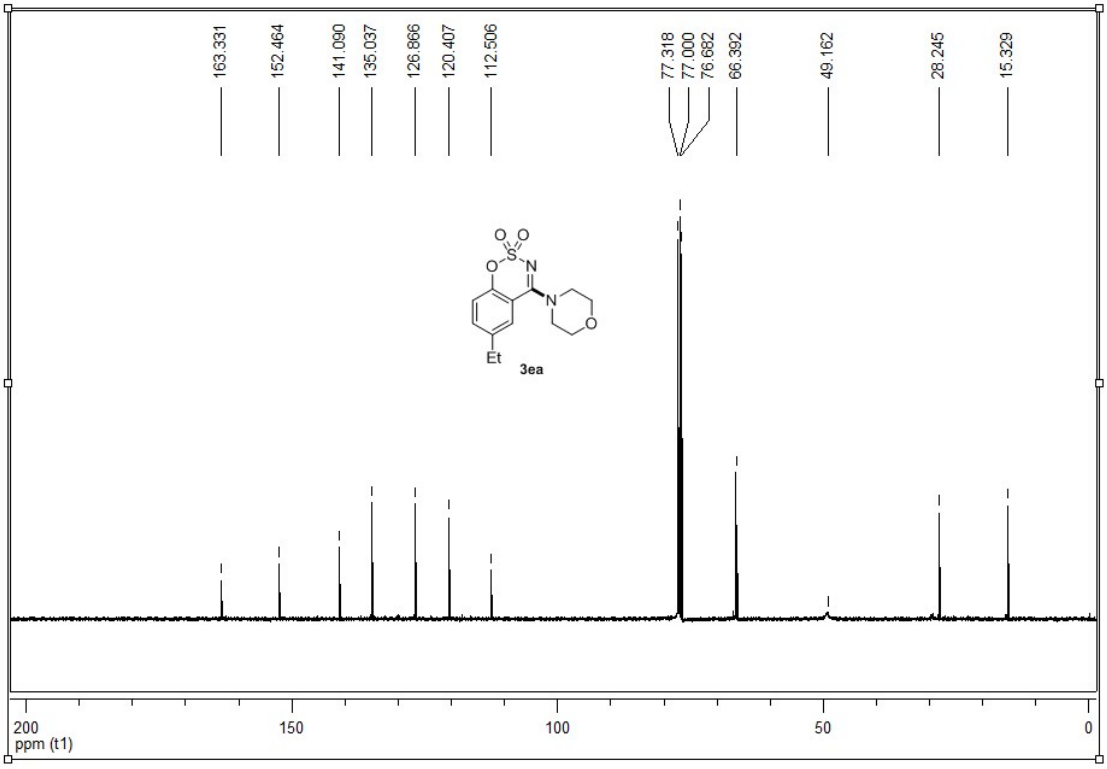
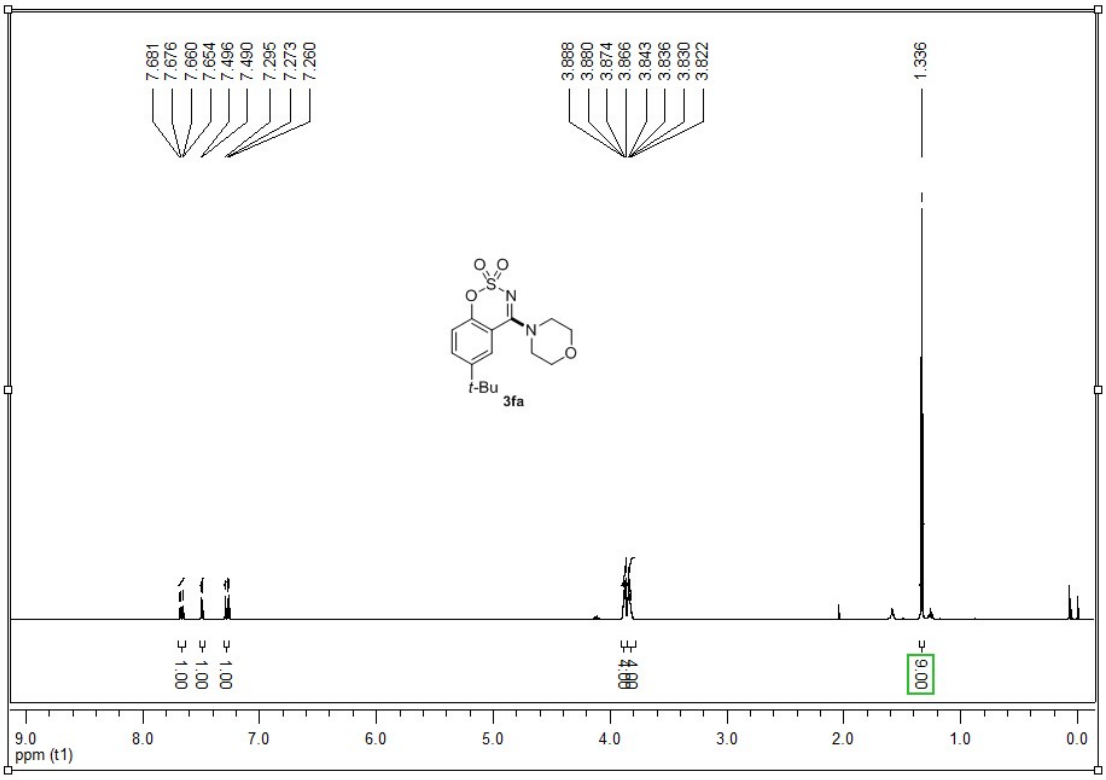
7.471
7.455
7.450
7.322
7.317
7.271
7.260
7.250
3.888
3.877
3.864
3.827
3.813
3.803
2.724
2.705
2.686
2.667
1.269
1.250
1.231
3ea
1.00
4.84
2.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


163.331
152.464
141.090
135.037
126.866
120.407
112.506
77.318
77.000
76.682
66.392
49.162
28.245
15.329
3ea
200
150
100
50
0
ppm (t1)


7.681
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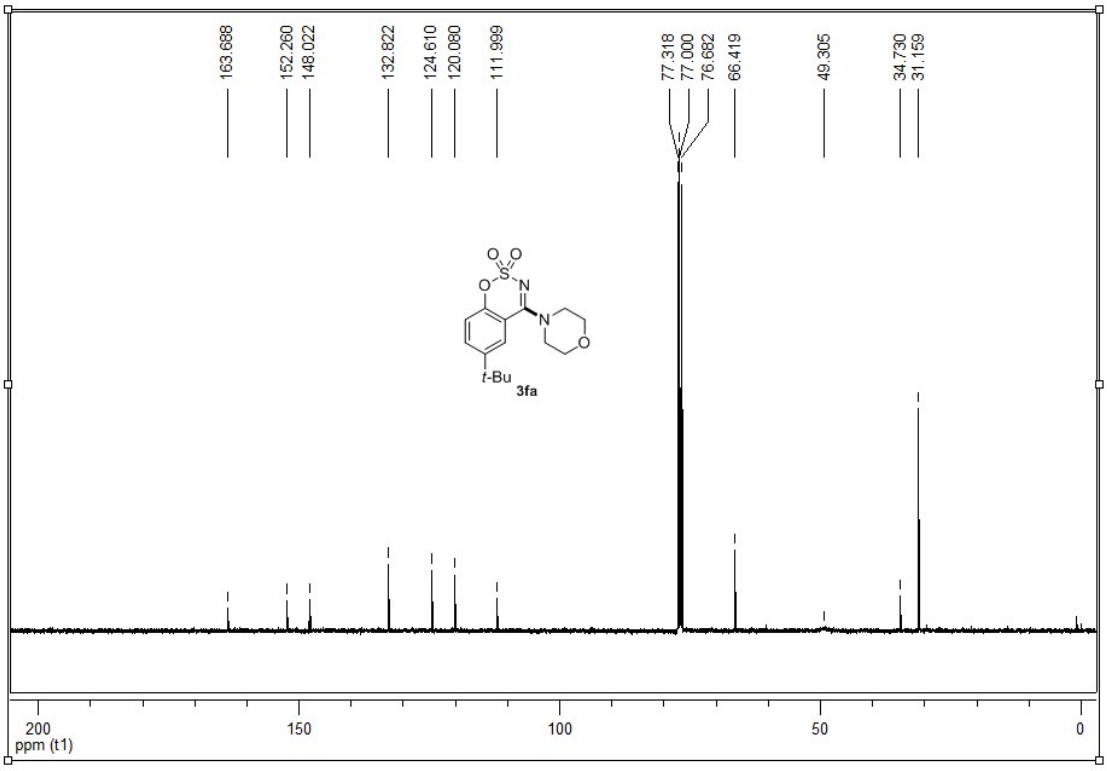
7.660
7.654
7.496
7.490
7.295
7.273
7.260
3.888
3.880
3.874
3.866
3.843
3.836
3.830
3.822
1.336
t-Bu
3fa
1.00
1.00
1.00
4.00
9.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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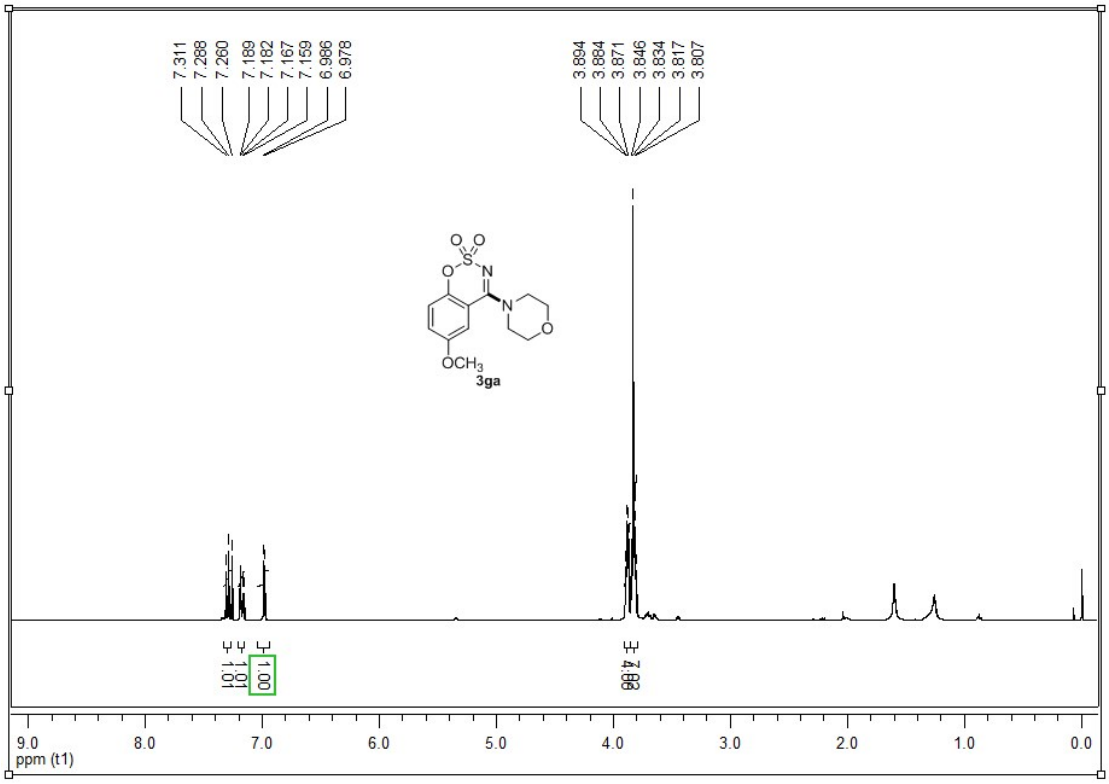
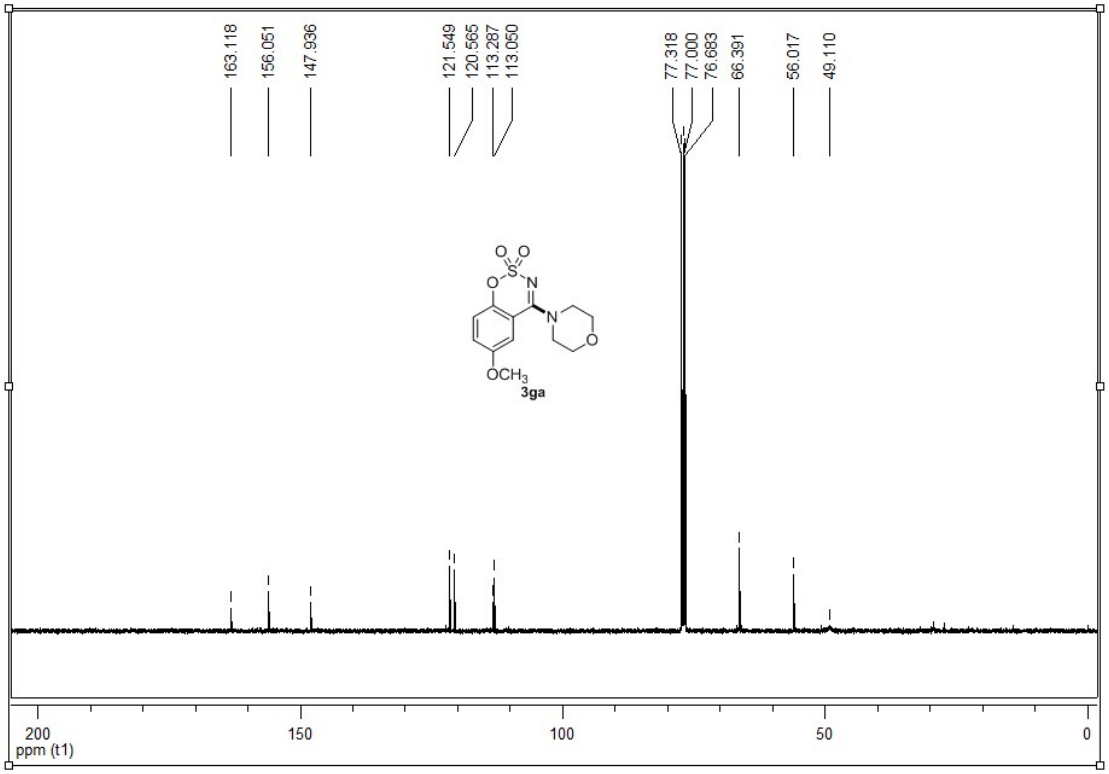
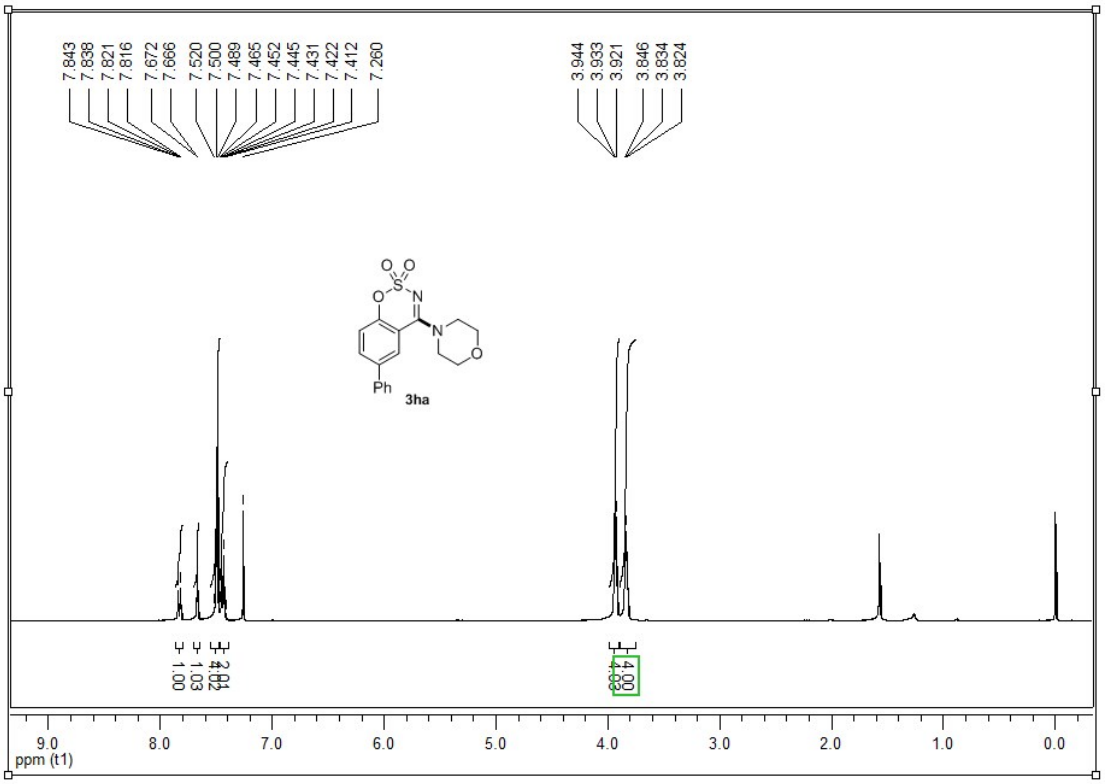
152.260
148.022
132.822
124.610
120.080
111.999
77.318
77.000
76.682
66.419
49.305
34.730
31.159
t-Bu
3fa
200
150
100
50
0
ppm (t1)


7.311
7.288
7.260
7.189
7.182
7.167
7.159
6.986
6.978
3.894
3.884
3.871
3.846
3.834
3.817
3.807
3ga
3.01
3.01
1.00
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


163.118
156.051
147.936
121.549
120.565
113.287
113.050
77.318
77.000
76.683
66.391
56.017
49.110
3ga
200
150
100
50
0
ppm (t1)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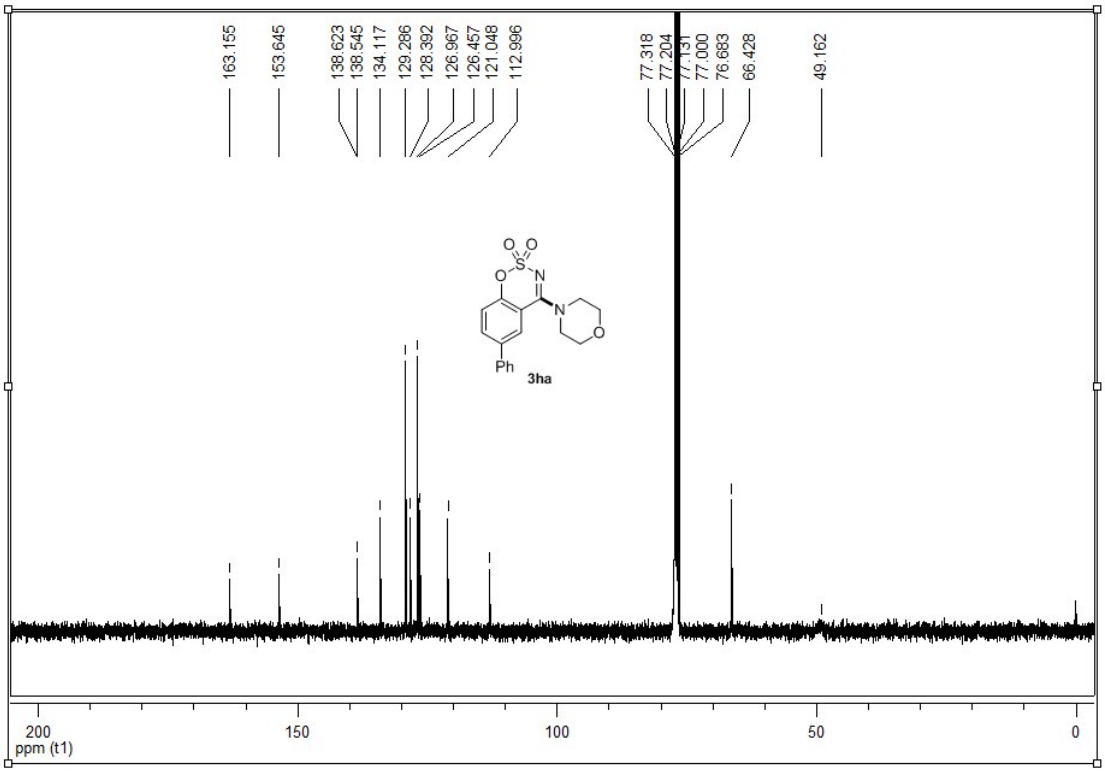
7.821
7.816
7.672
7.666
7.520
7.500
7.489
7.465
7.452
7.445
7.431
7.422
7.412
7.260
3.944
3.933
3.921
3.846
3.834
3.824
3ha
1.00
1.03
2.12
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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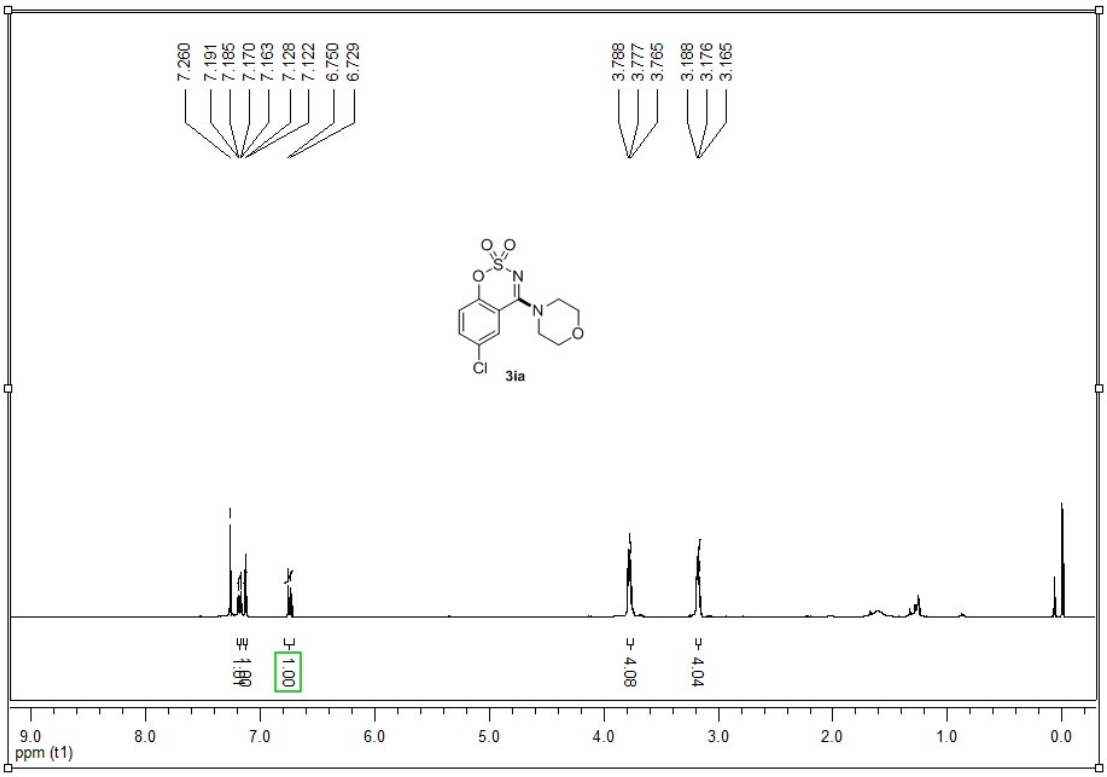
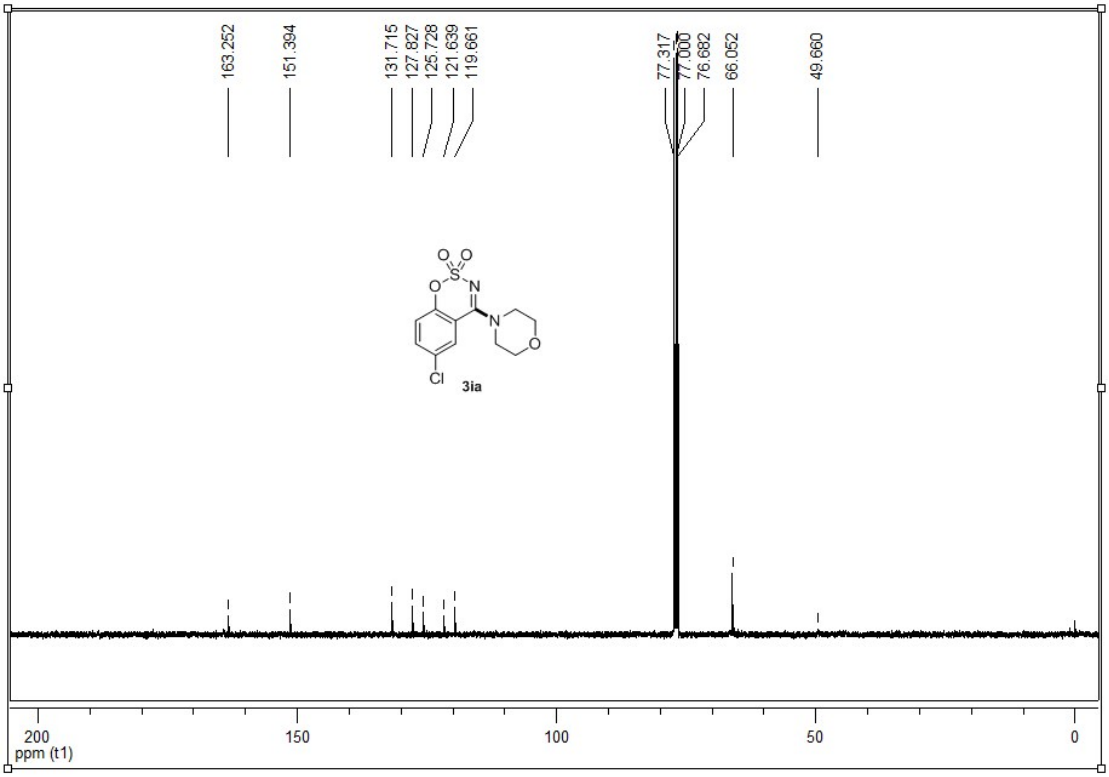
153.645
138.623
138.545
134.117
129.286
128.392
126.967
126.457
121.048
112.996
77.318
77.204
77.131
77.000
76.683
66.428
49.162
3ha
200
150
100
50
0
ppm (t1)


7.260
7.191
7.185
7.170
7.163
7.128
7.122
6.750
6.729
3.788
3.777
3.765
3.188
3.176
3.165
3ia
1.00
4.08
4.04
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


163.252
151.394
131.715
127.827
125.728
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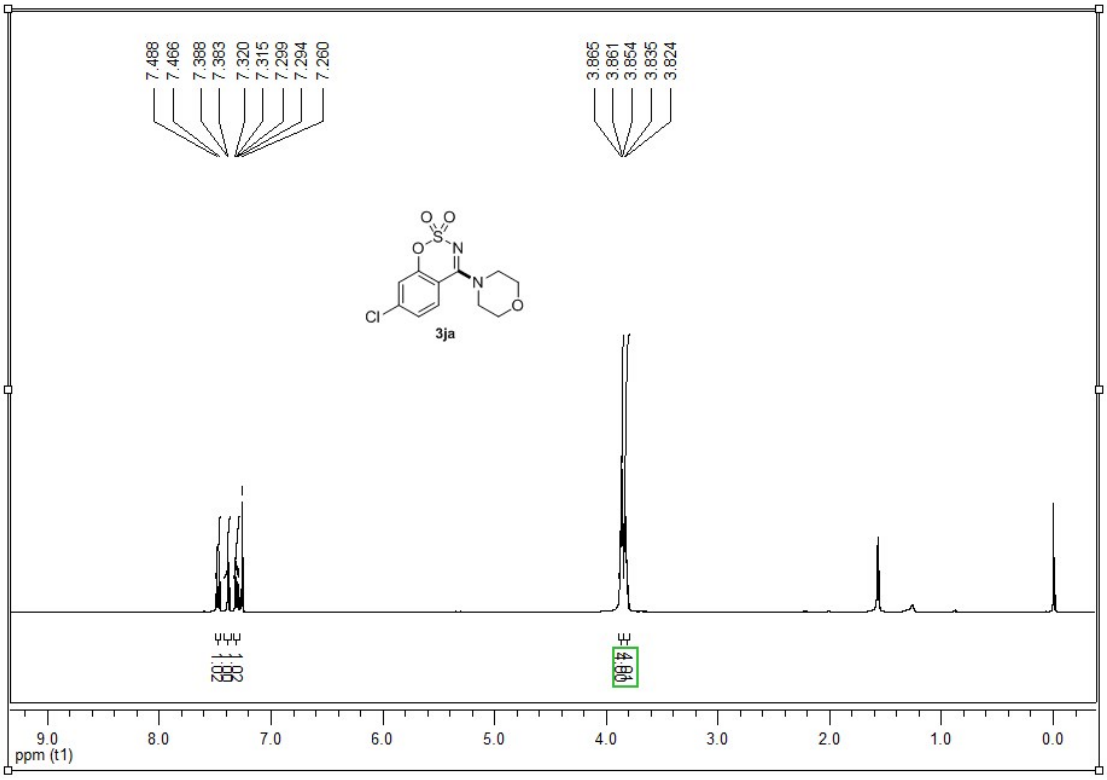
119.661
77.317
77.000
76.682
66.052
49.660
3ia
200
150
100
50
0
ppm (t1)


7.488
7.466
7.388
7.383
7.320
7.315
7.299
7.294
7.260
3.865
3.861
3.854
3.835
3.824
3ja
2.11
4.00
9.0
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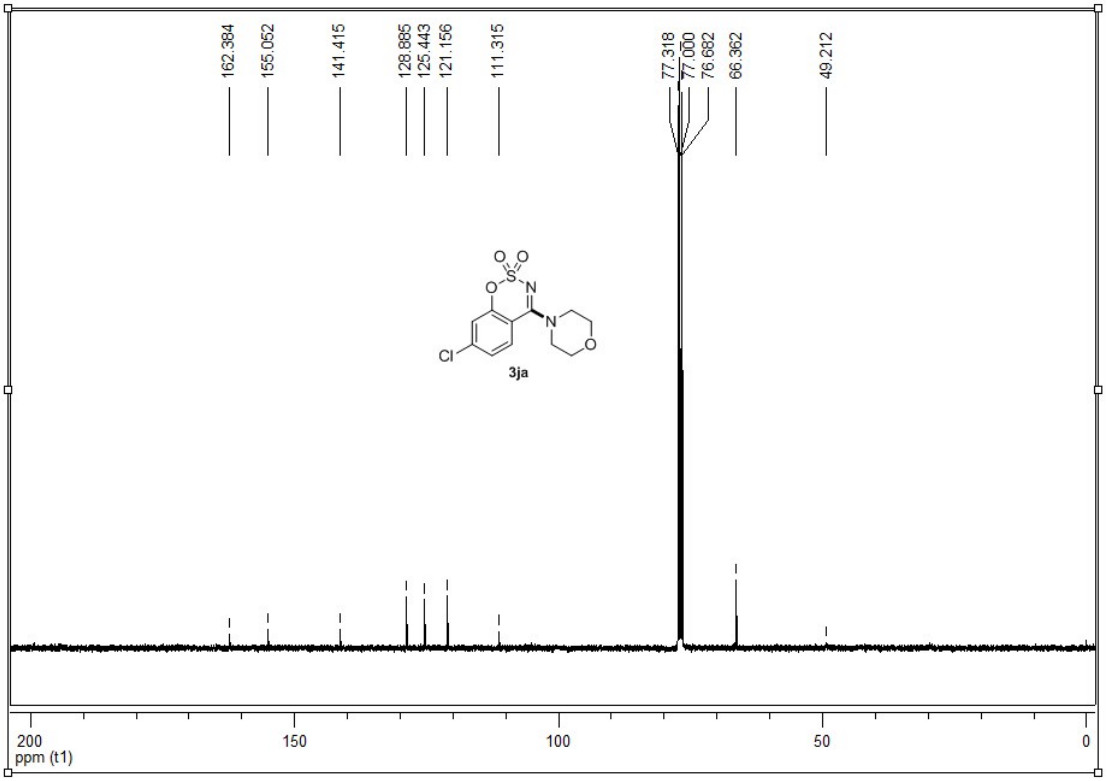
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


162.384
155.052
141.415
128.885
125.443
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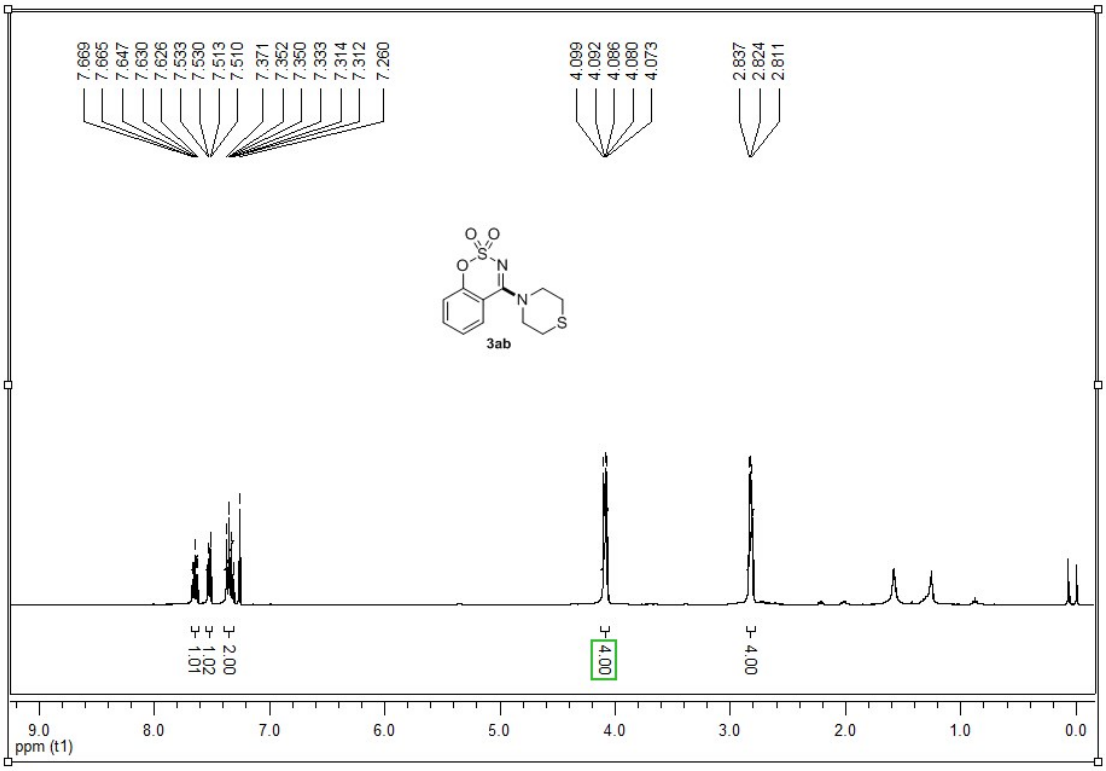
111.315
77.318
77.000
76.682
66.362
49.212
3ja
200
150
100
50
0
ppm (t1)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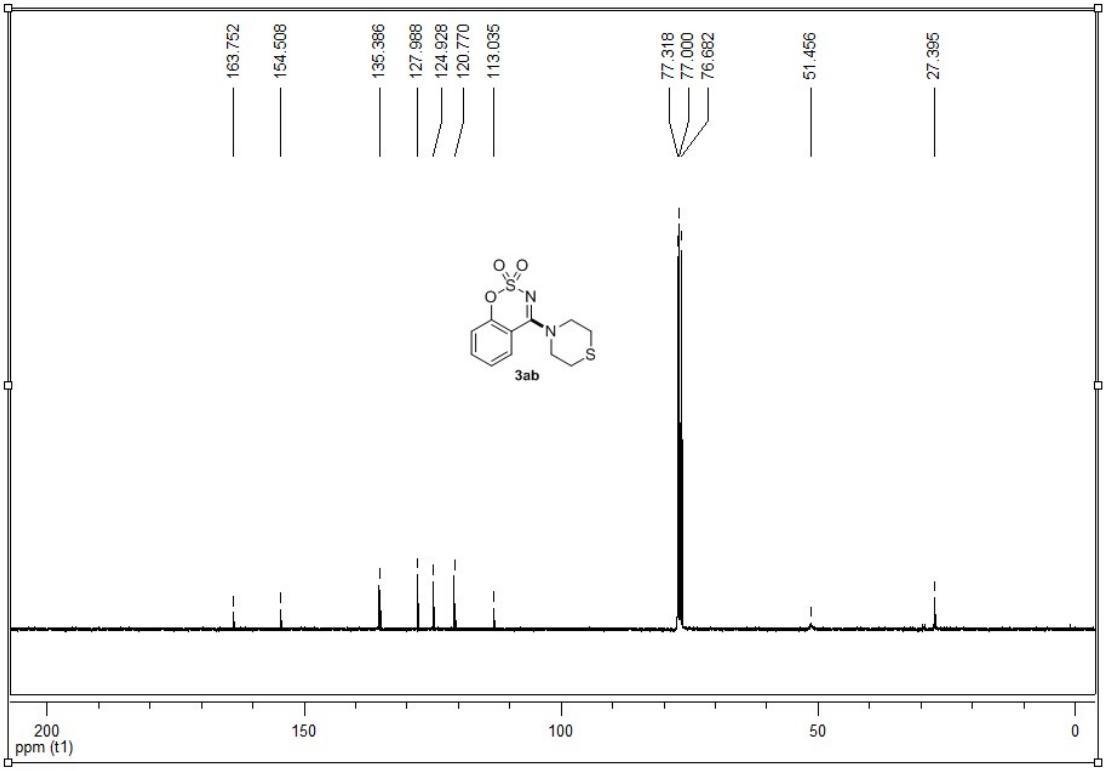
7.665
7.647
7.630
7.626
7.533
7.530
7.513
7.510
7.371
7.352
7.350
7.333
7.314
7.312
7.260
4.099
4.092
4.086
4.080
4.073
2.837
2.824
2.811
3ab
1.01
1.12
2.00
4.00
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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154.508
135.386
127.988
124.928
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77.000
76.682
51.456
27.395
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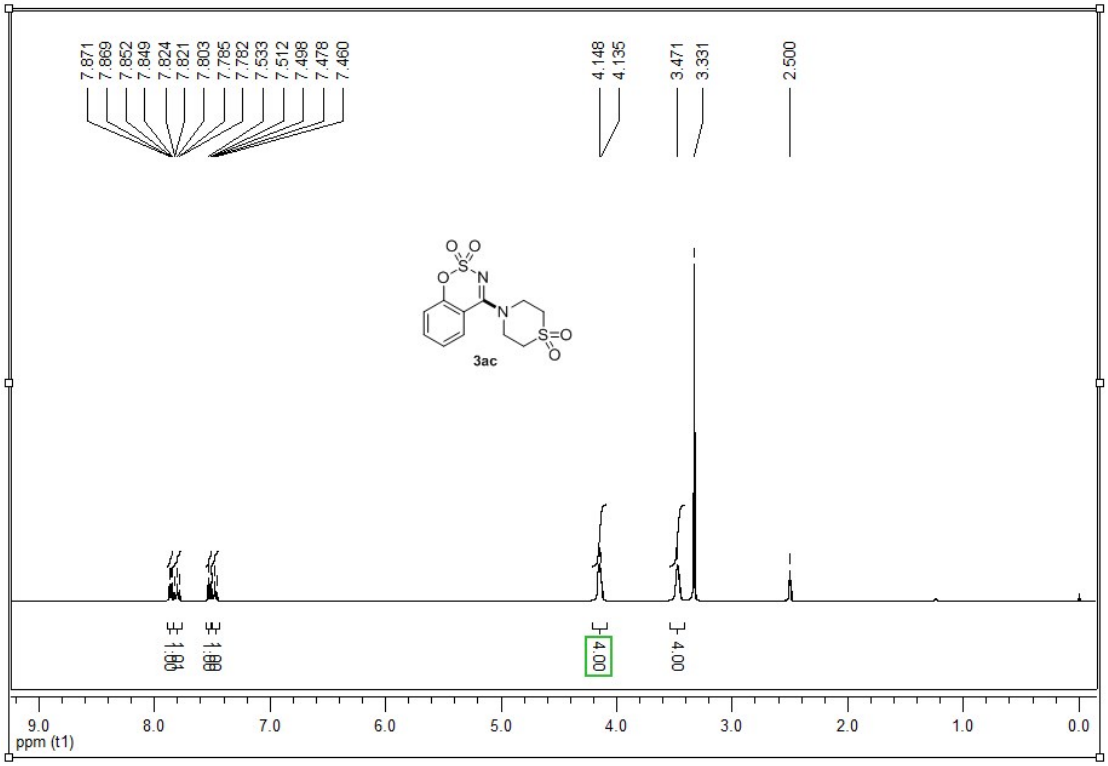
200
150
100
50
0
ppm (t1)


7.871
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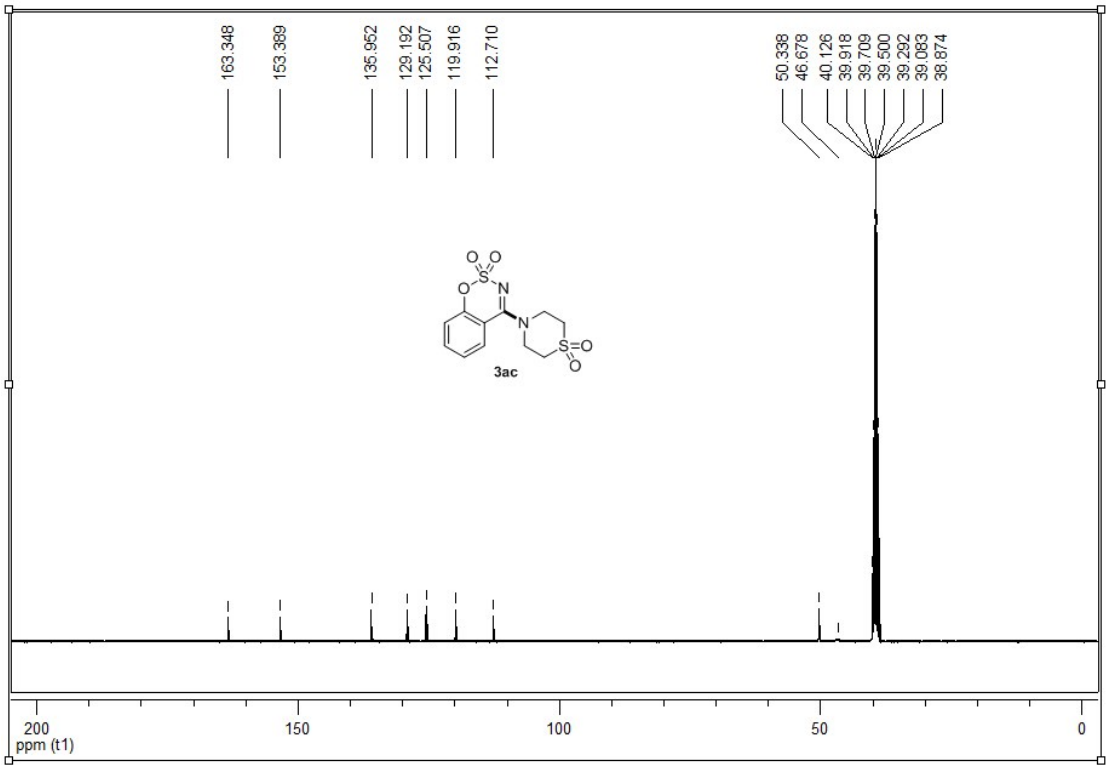
7.852
7.849
7.824
7.821
7.803
7.785
7.782
7.533
7.512
7.498
7.478
7.460
4.148
4.135
3.471
3.331
2.500
3ac
4.00
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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125.507
119.916
112.710
50.338
46.678
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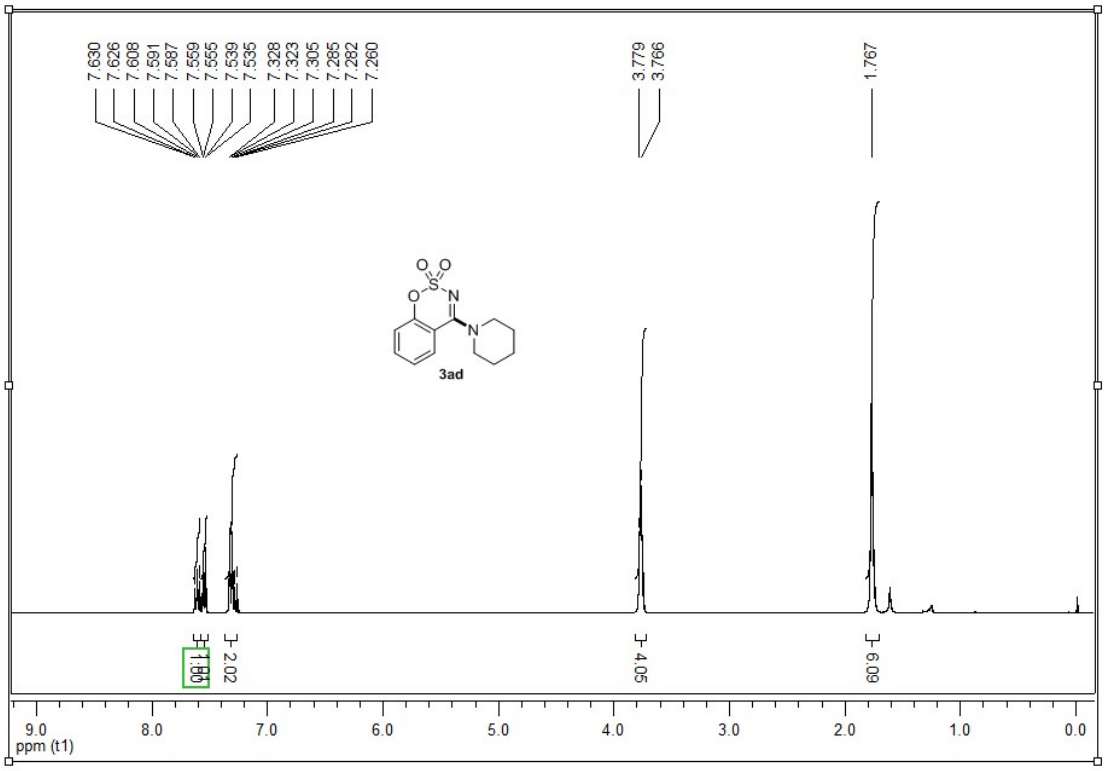
39.918
39.709
39.500
39.292
39.083
38.874
3ac
200
150
100
50
0
ppm (t1)


7.630
7.626
7.608
7.591
7.587
7.559
7.555
7.539
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7.328
7.323
7.305
7.285
7.282
7.260
3.779
3.766
1.767
3ad
1.00
2.02
4.05
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7.0
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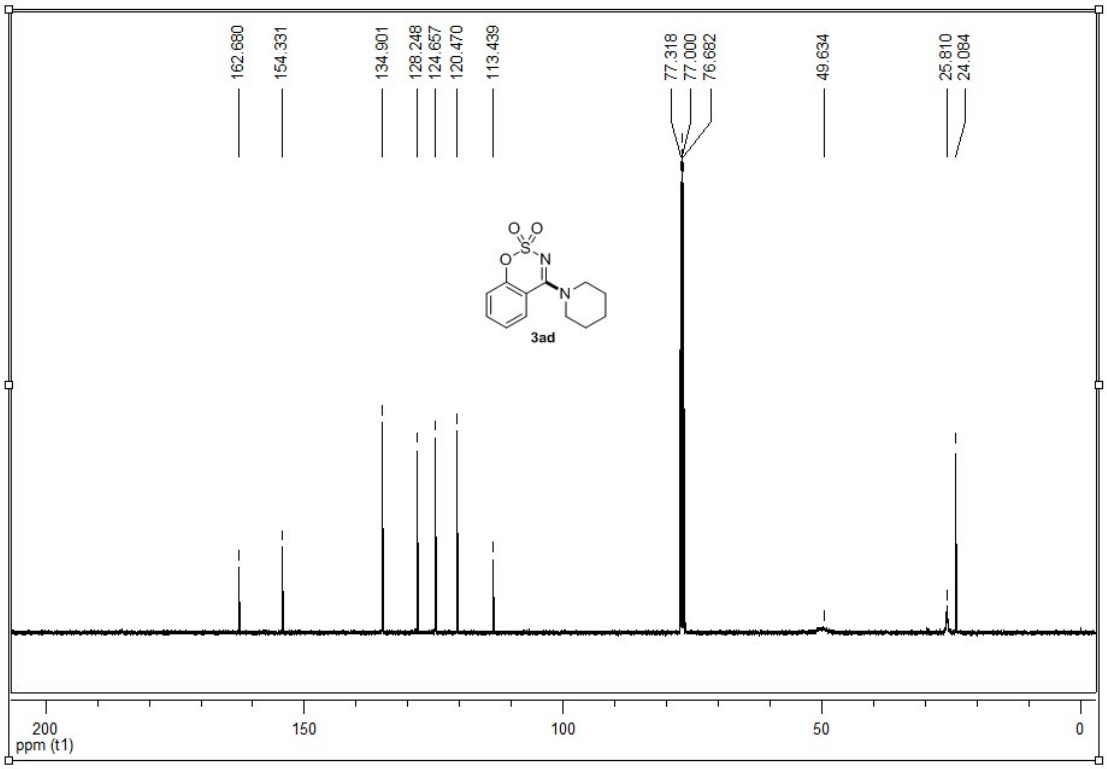
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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128.248
124.657
120.470
113.439
77.318
77.000
76.682
49.634
25.810
24.084
3ad
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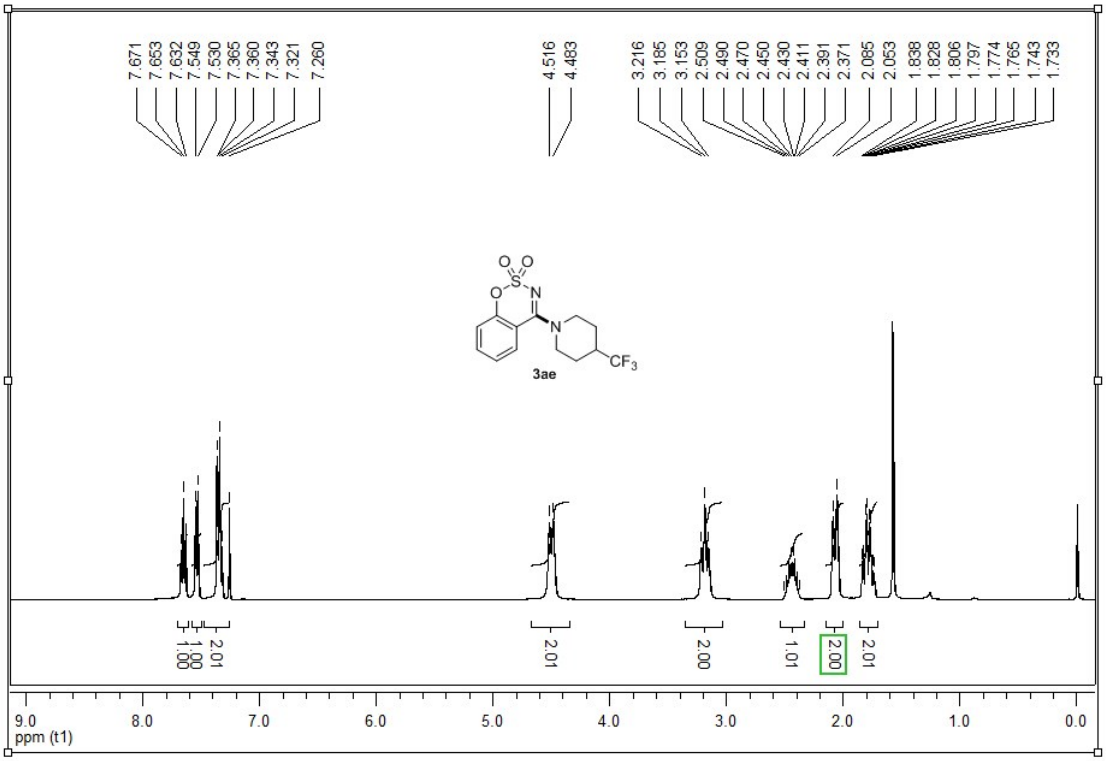
150
100
50
0
ppm (t1)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7.365
7.360
7.343
7.321
7.260
4.516
4.483
3.216
3.185
3.153
2.509
2.490
2.470
2.450
2.430
2.411
2.391
2.371
2.085
2.053
1.838
1.828
1.806
1.797
1.774
1.765
1.743
1.733
3ae
1.00
2.01
2.01
2.00
1.01
2.00
2.01
9.0
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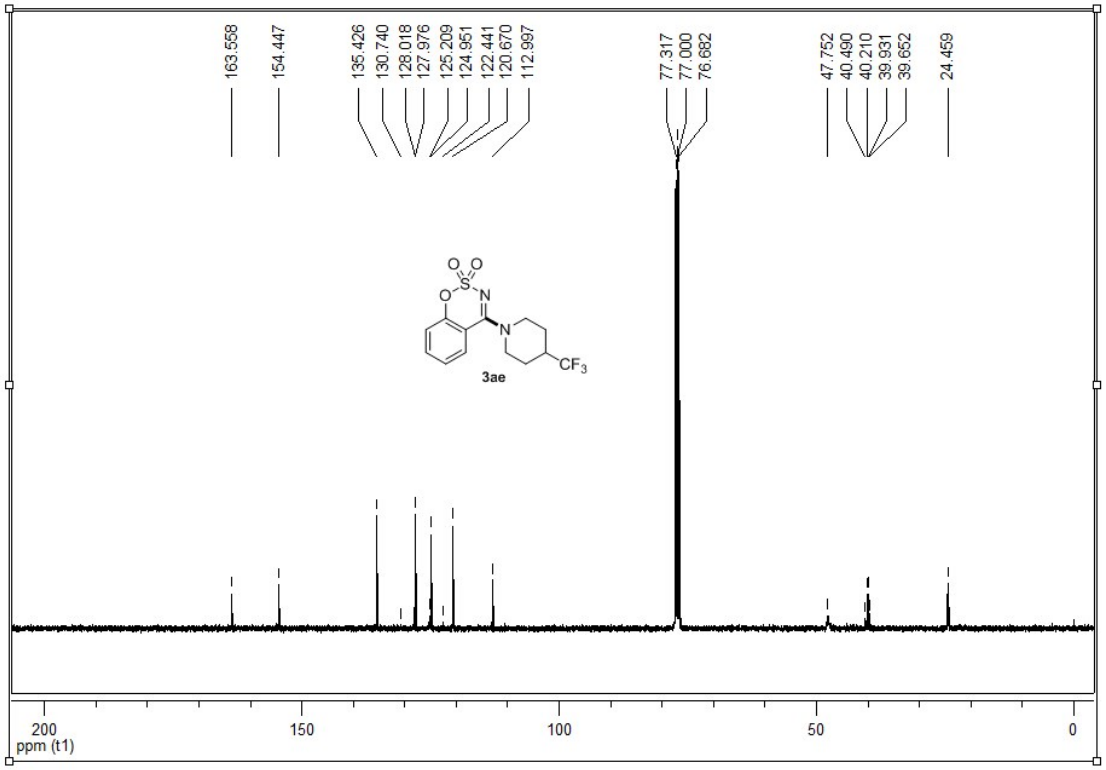
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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127.976
125.209
124.951
122.441
120.670
112.997
77.317
77.000
76.682
47.752
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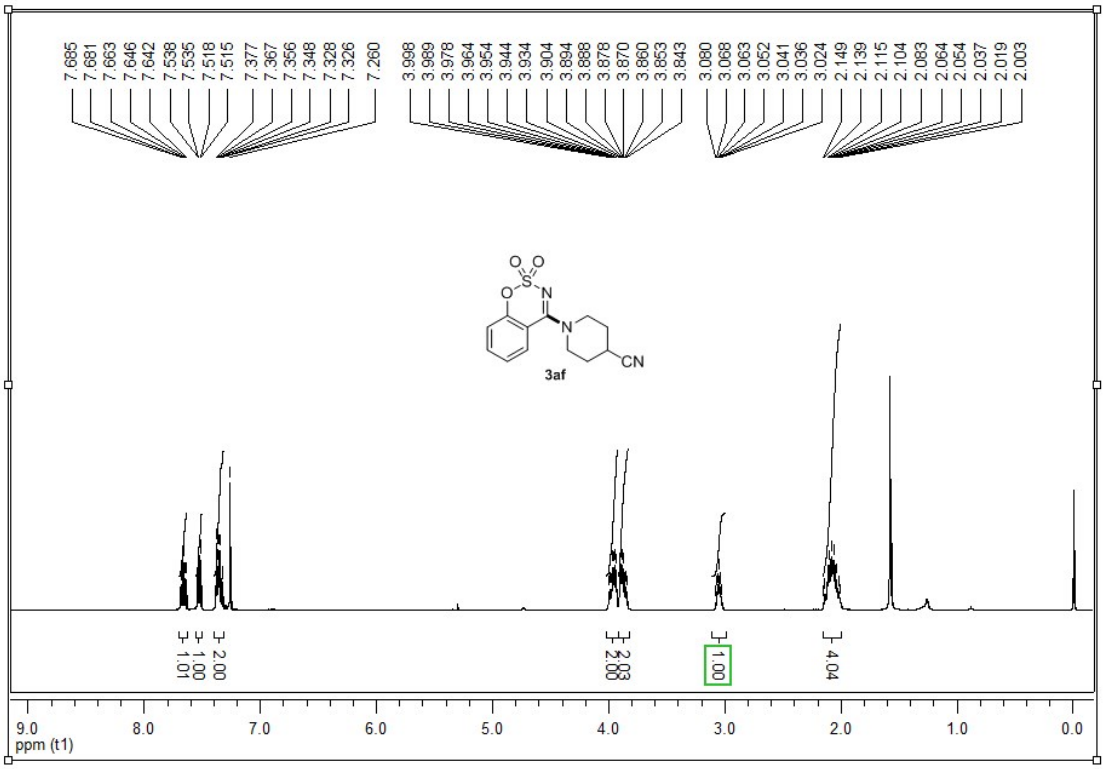
40.210
39.931
39.652
24.459
3ae
200
150
100
50
0
ppm (t1)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7.535
7.518
7.515
7.377
7.367
7.356
7.348
7.328
7.326
7.260
3.998
3.989
3.978
3.964
3.954
3.944
3.934
3.904
3.894
3.888
3.878
3.870
3.860
3.853
3.843
3.080
3.068
3.063
3.052
3.041
3.036
3.024
2.149
2.139
2.115
2.104
2.083
2.064
2.054
2.037
2.019
2.003
3af
1.01
1.00
2.00
2.08
2.28
1.00
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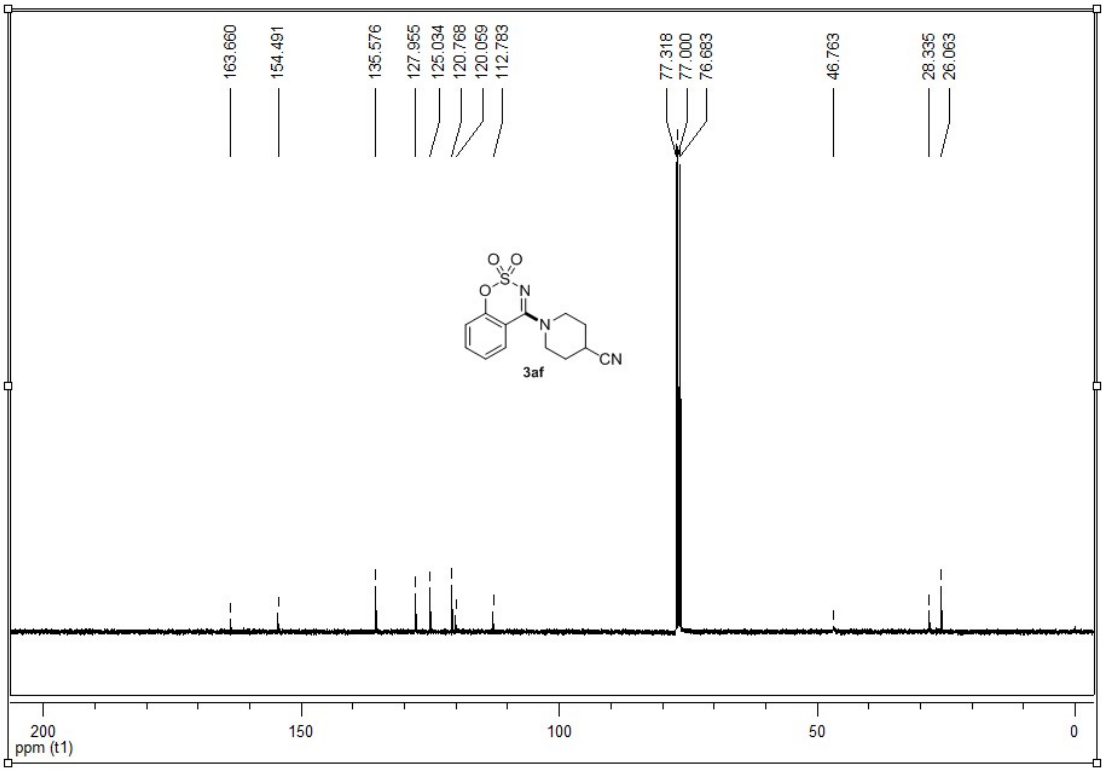
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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76.683
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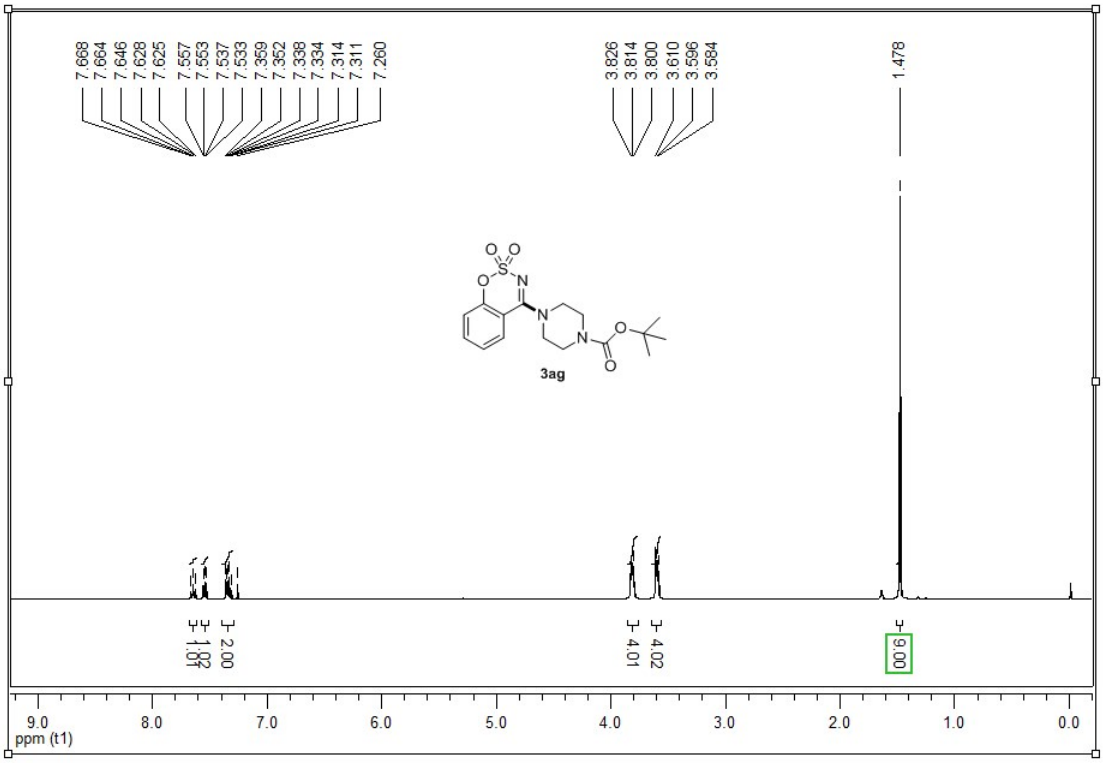
28.335
26.063
3af
200
150
100
50
0
ppm (t1)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7.553
7.537
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7.359
7.352
7.338
7.334
7.314
7.311
7.260
3.826
3.814
3.800
3.610
3.596
3.584
1.478
3ag
2.00
4.01
4.02
9.00
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7.0
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5.0
4.0
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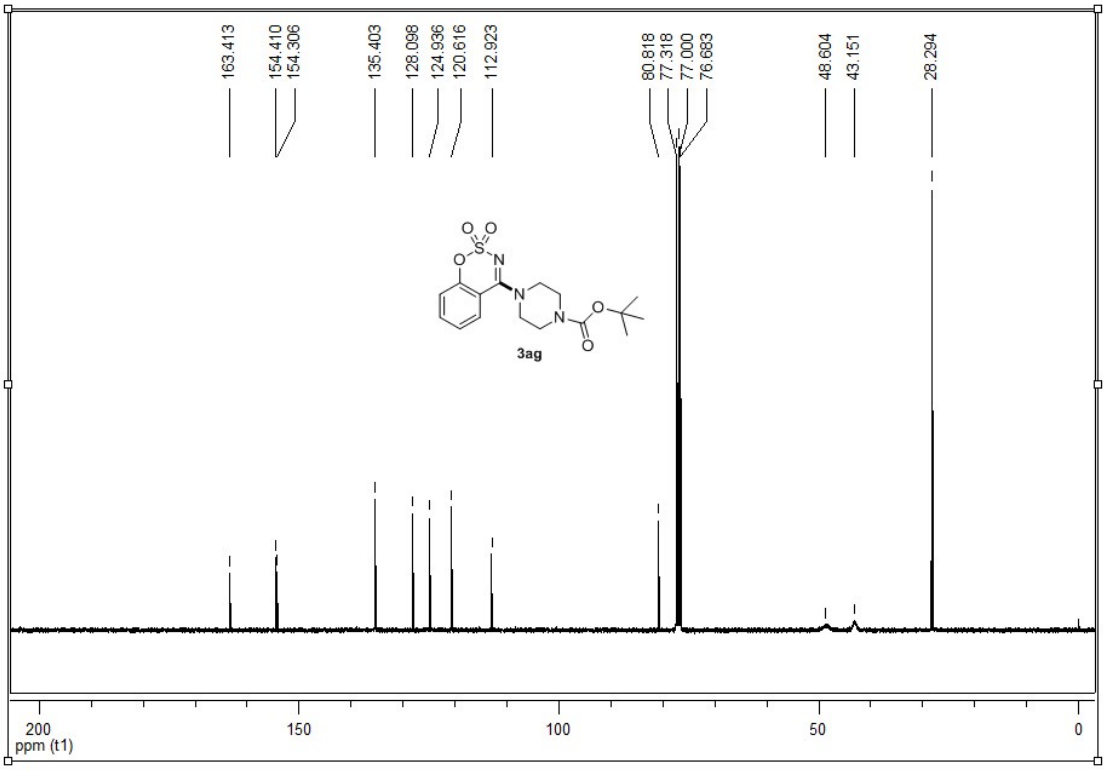
2.0
1.0
0.0
ppm (t1)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120.616
112.923
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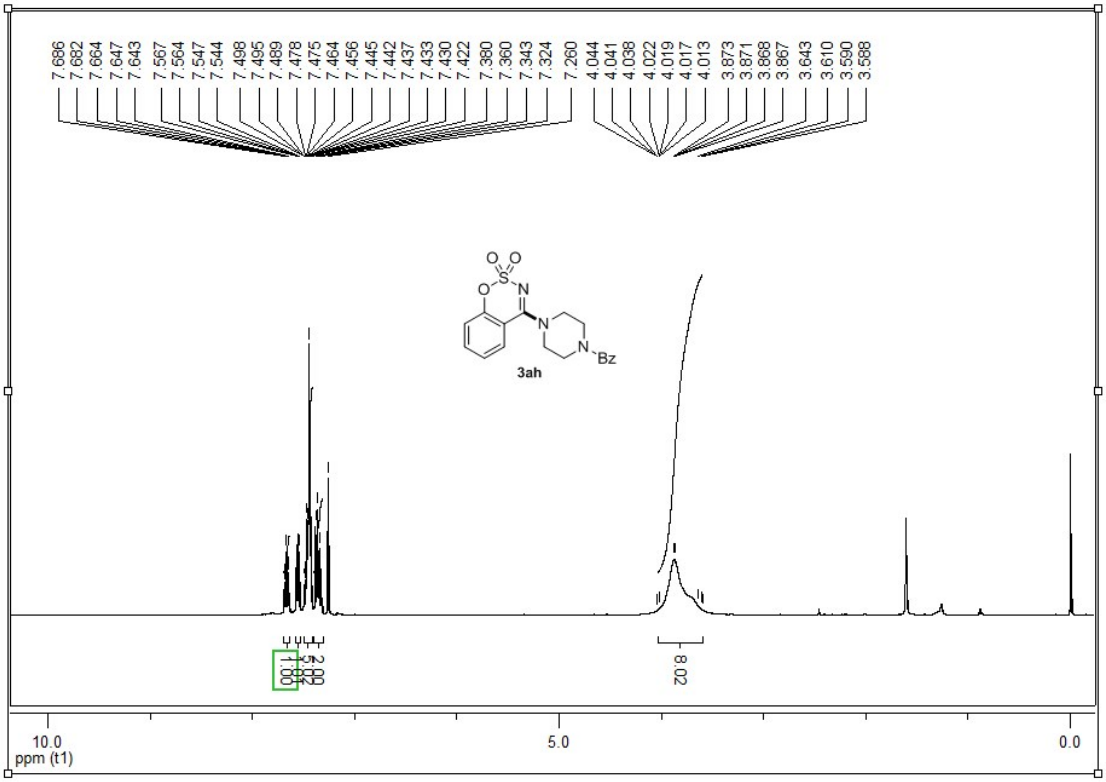
76.683
48.604
43.151
28.294
3ag
200
150
100
50
0
ppm (t1)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7.495
7.489
7.478
7.475
7.464
7.456
7.445
7.442
7.437
7.433
7.430
7.422
7.380
7.360
7.343
7.324
7.260
4.044
4.041
4.038
4.022
4.019
4.017
4.013
3.873
3.871
3.868
3.867
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3.590
3.588
3ah
Bz
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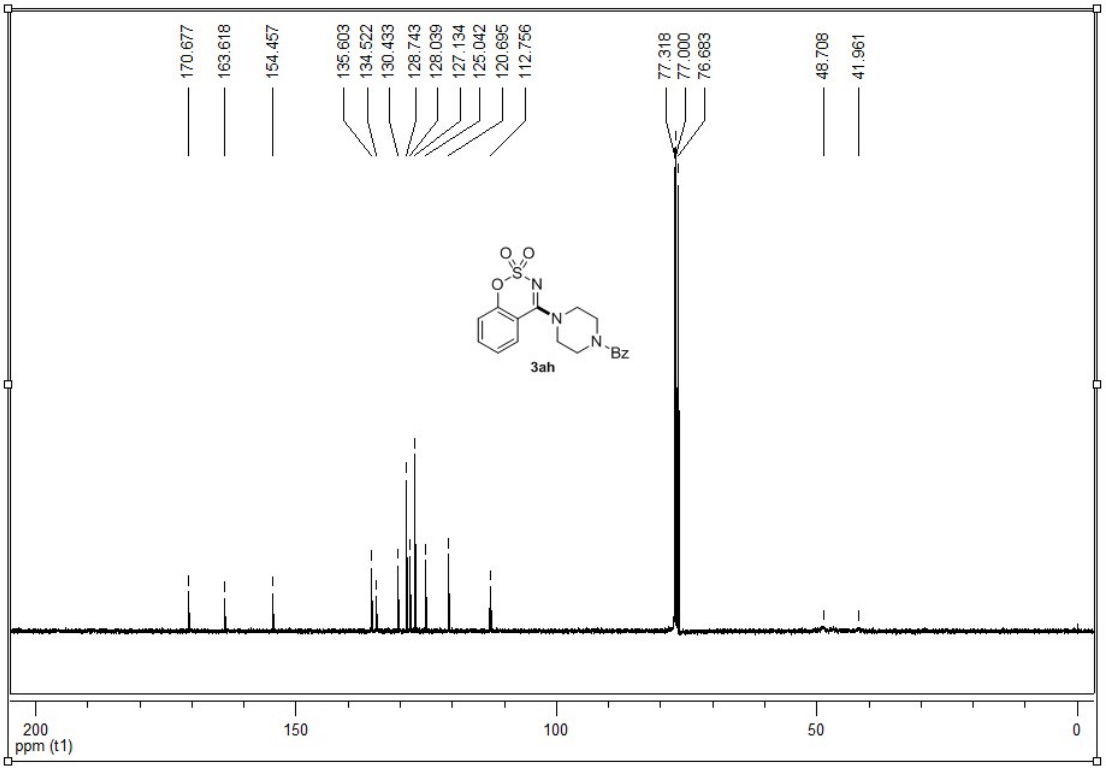
2.08
10.0
ppm (t1)
5.0
0.0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130.433
128.743
128.039
127.134
125.042
120.695
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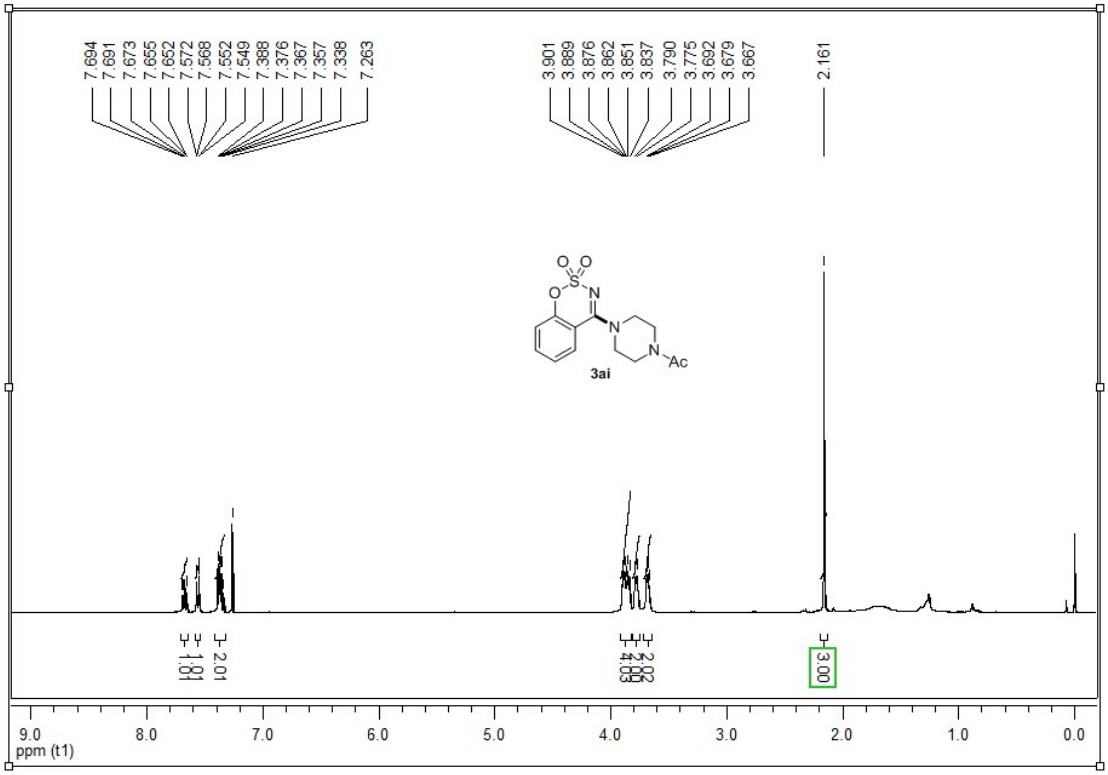
77.000
76.683
48.708
41.961
3ah
Bz
200
ppm (t1)
150
100
50
0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7.376
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7.338
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3.889
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3.862
3.851
3.837
3.790
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3.679
3.667
2.161
3ai
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2.01
4.08
2.02
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5.0
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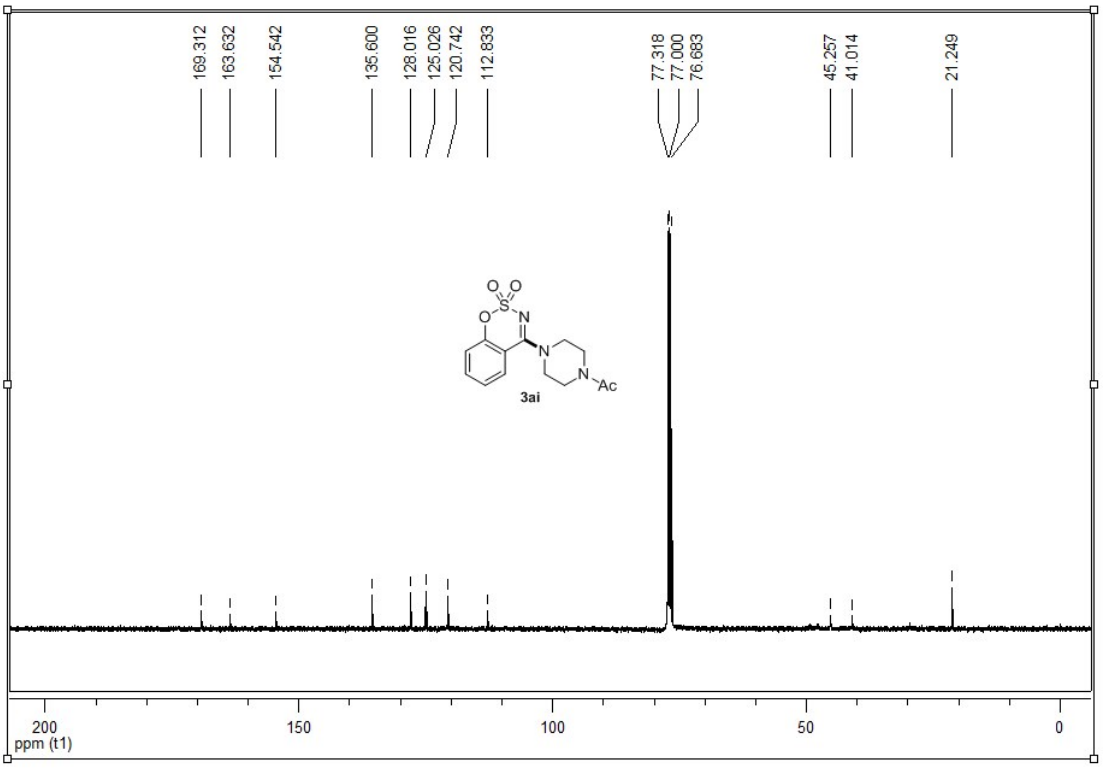
2.0
1.0
0.0
ppm (t1)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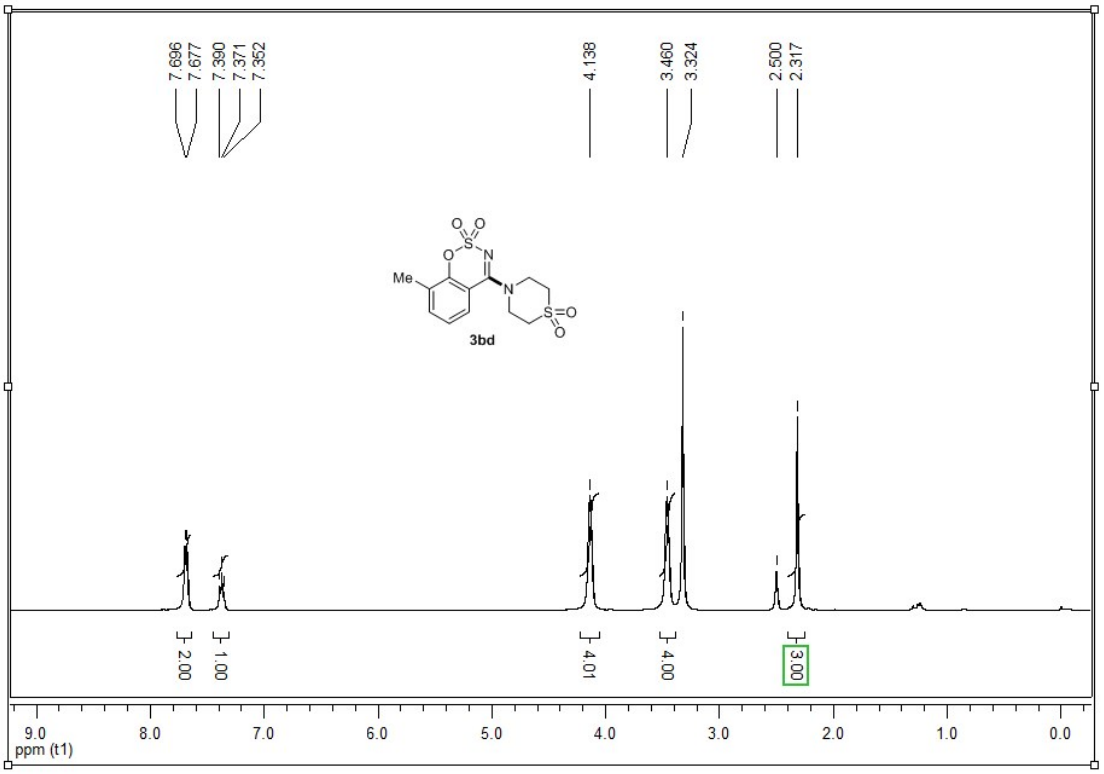
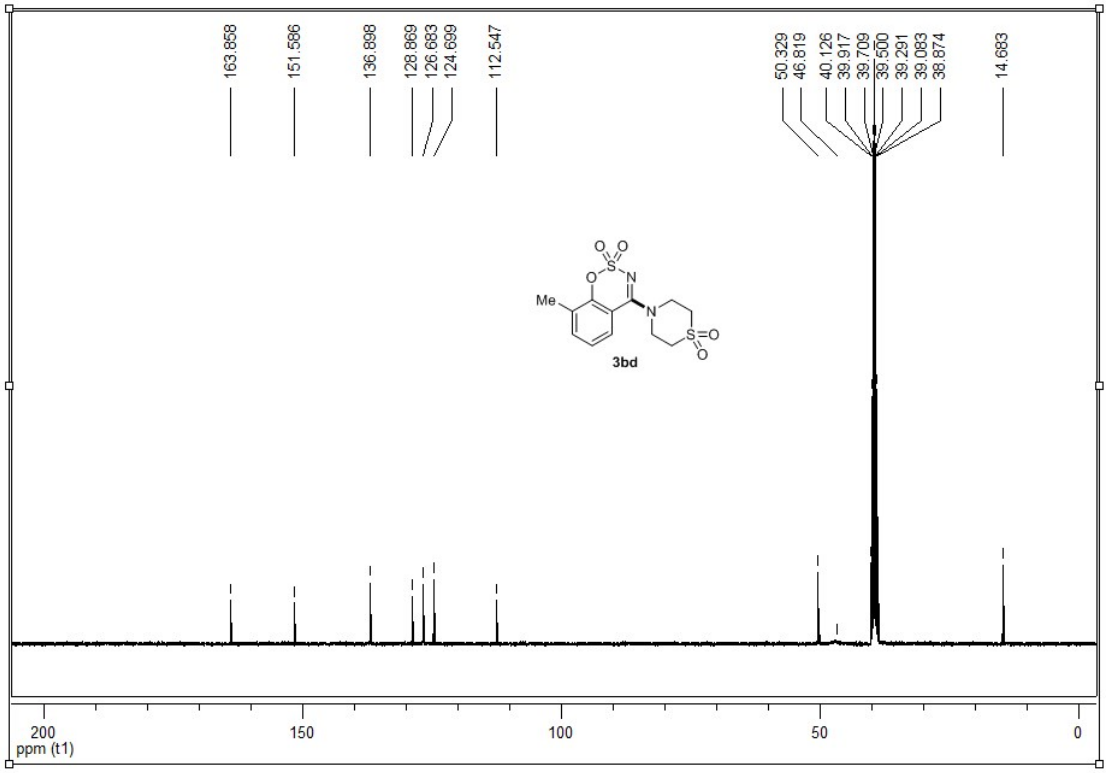
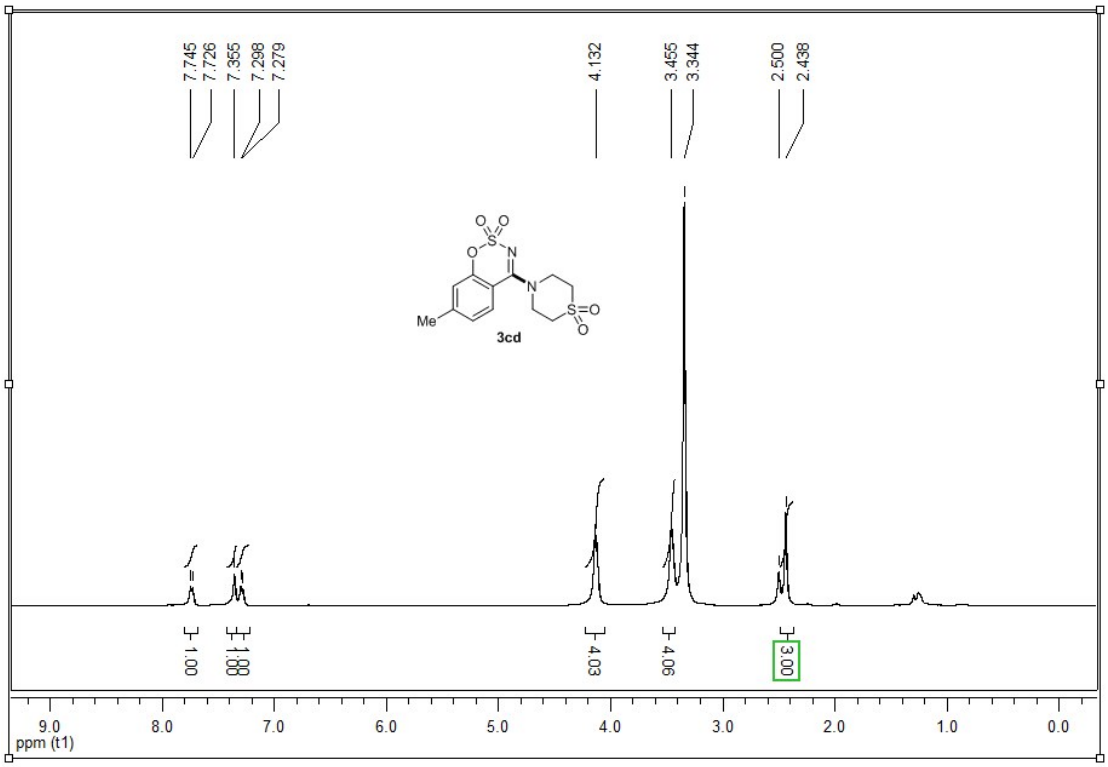
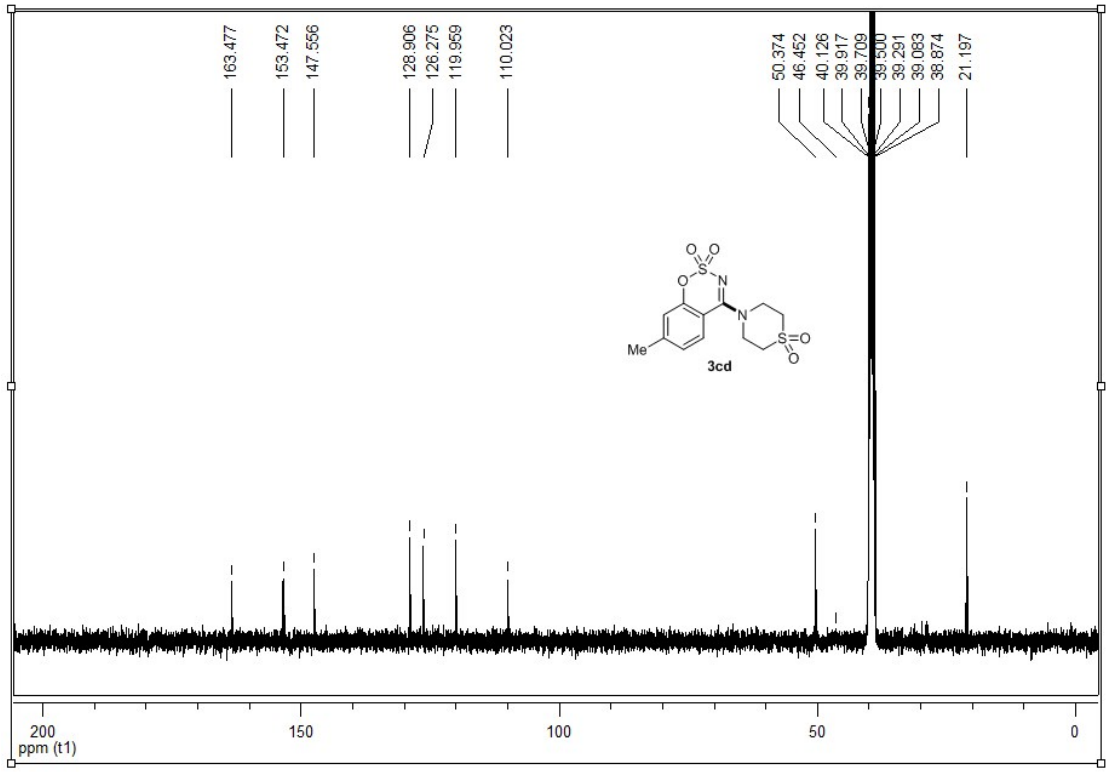
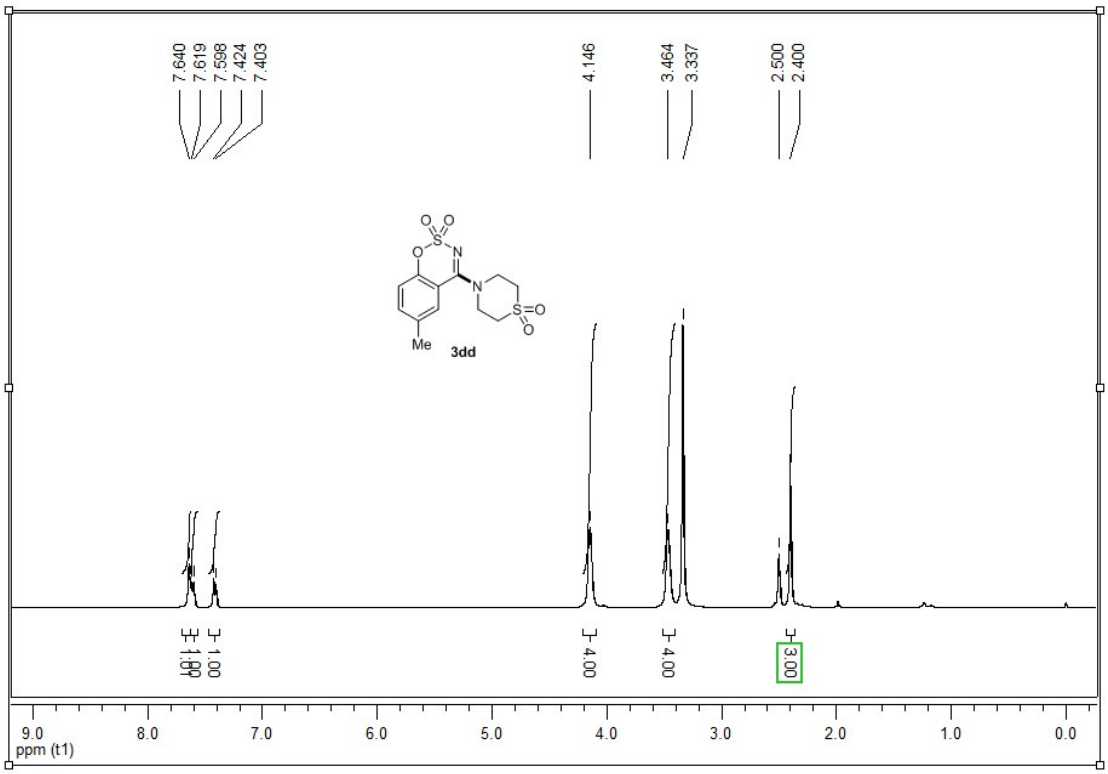
77.000
76.683
45.257
41.014
21.249
3ai
200
150
100
50
0
ppm (t1)

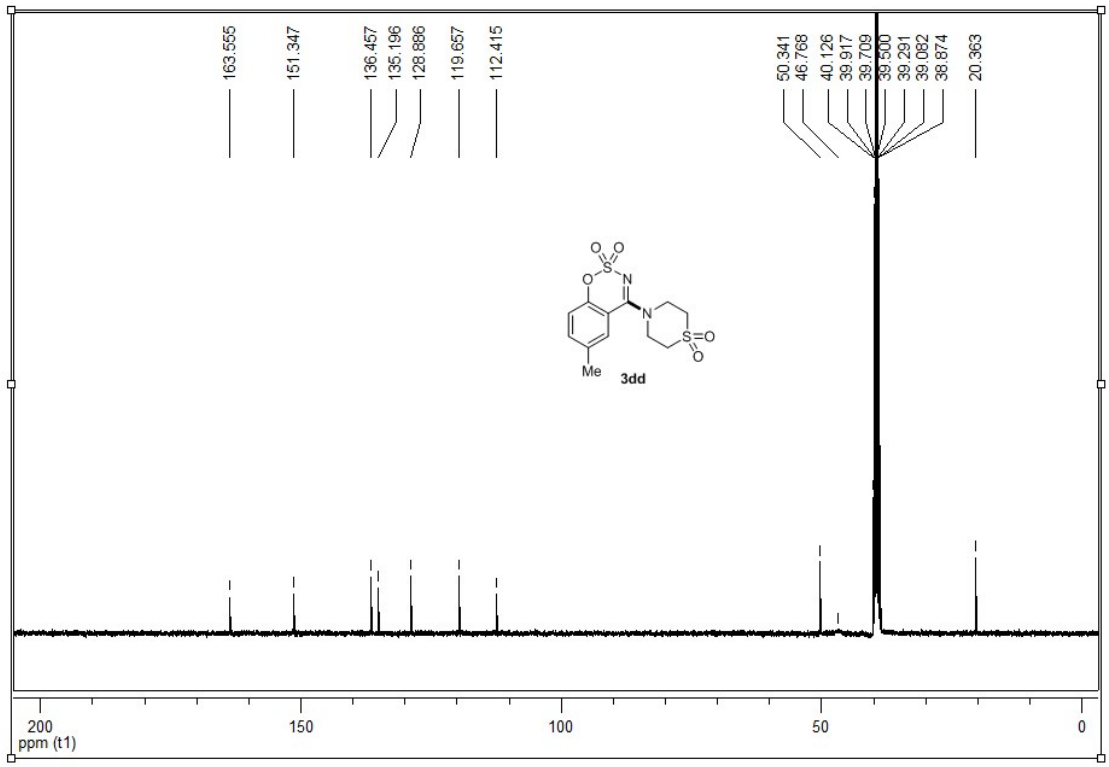

7.696
7.677
7.390
7.371
7.352
4.138
3.460
3.324
2.500
2.317
Me
3bd
2.00
1.00
4.01
4.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

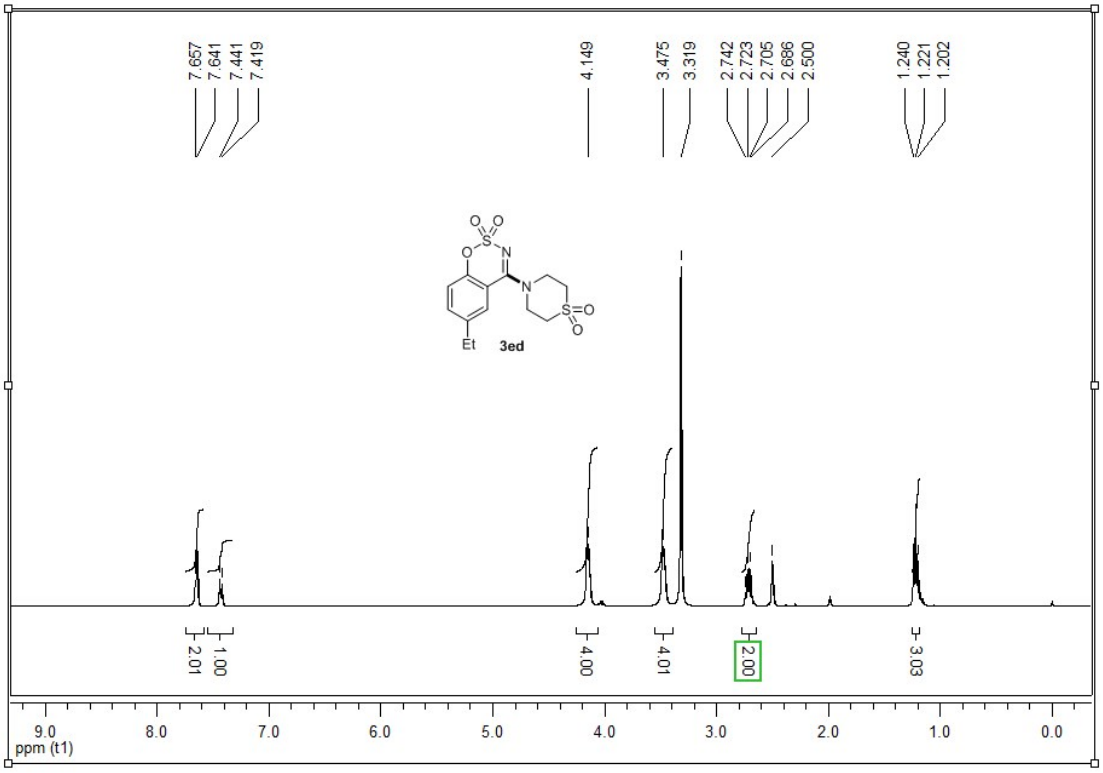

163.858
151.586
136.898
128.869
126.683
124.699
112.547
50.329
46.819
40.126
39.917
39.709
39.500
39.291
39.083
38.874
14.683
Me
3bd
200
150
100
50
0
ppm (t1)

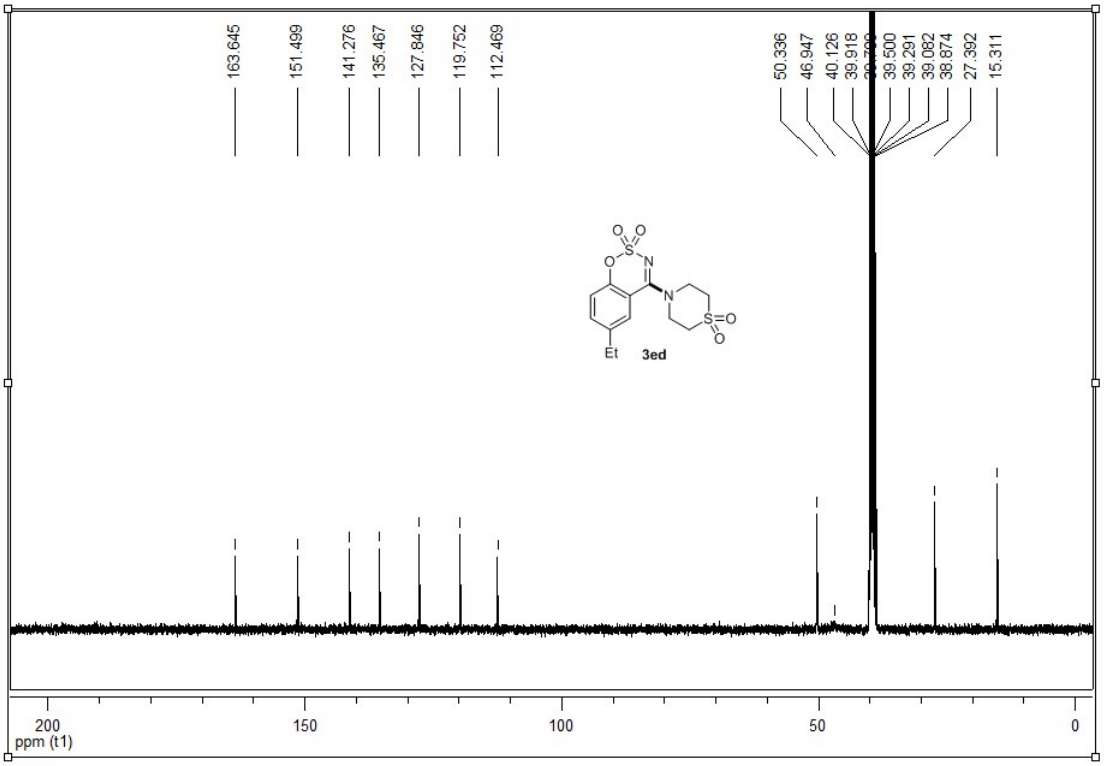

7.745
7.726
7.355
7.298
7.279
4.132
3.455
3.344
2.500
2.438
Me
3cd
1.00
1.00
4.03
4.06
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

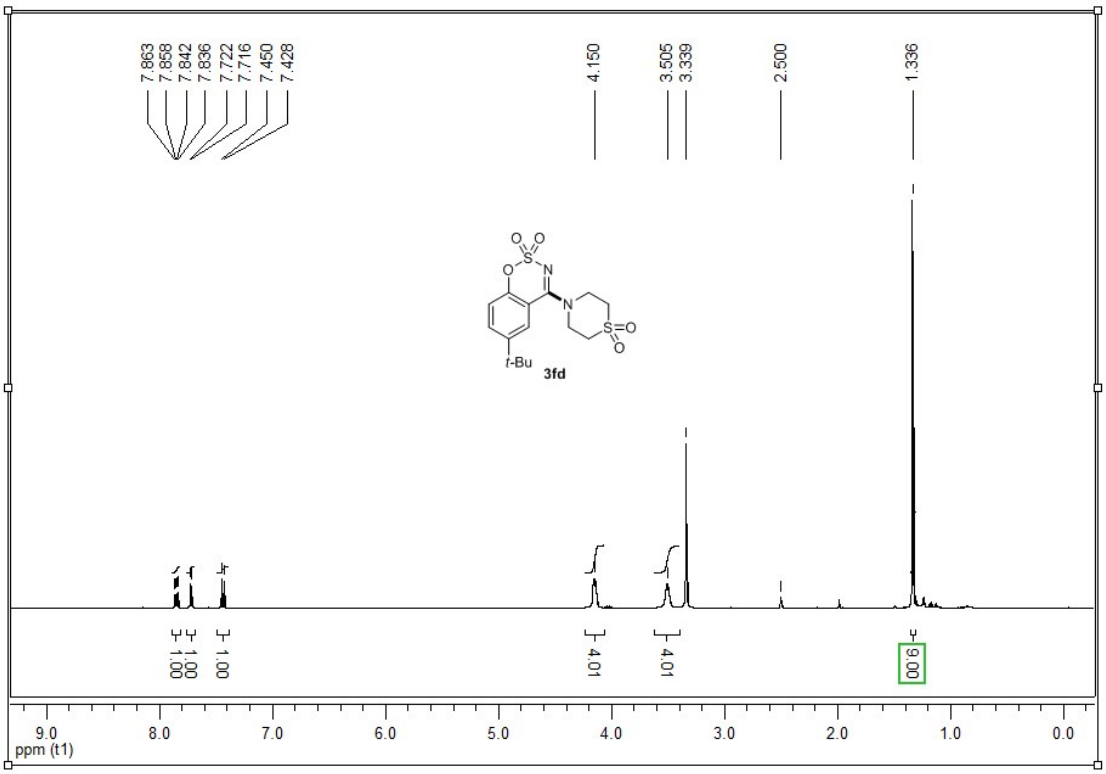

163.477
153.472
147.556
128.906
126.275
119.959
110.023
50.374
46.452
40.126
39.917
39.709
39.500
39.291
39.083
38.874
21.197
Me
3cd
200
150
100
50
0
ppm (t1)

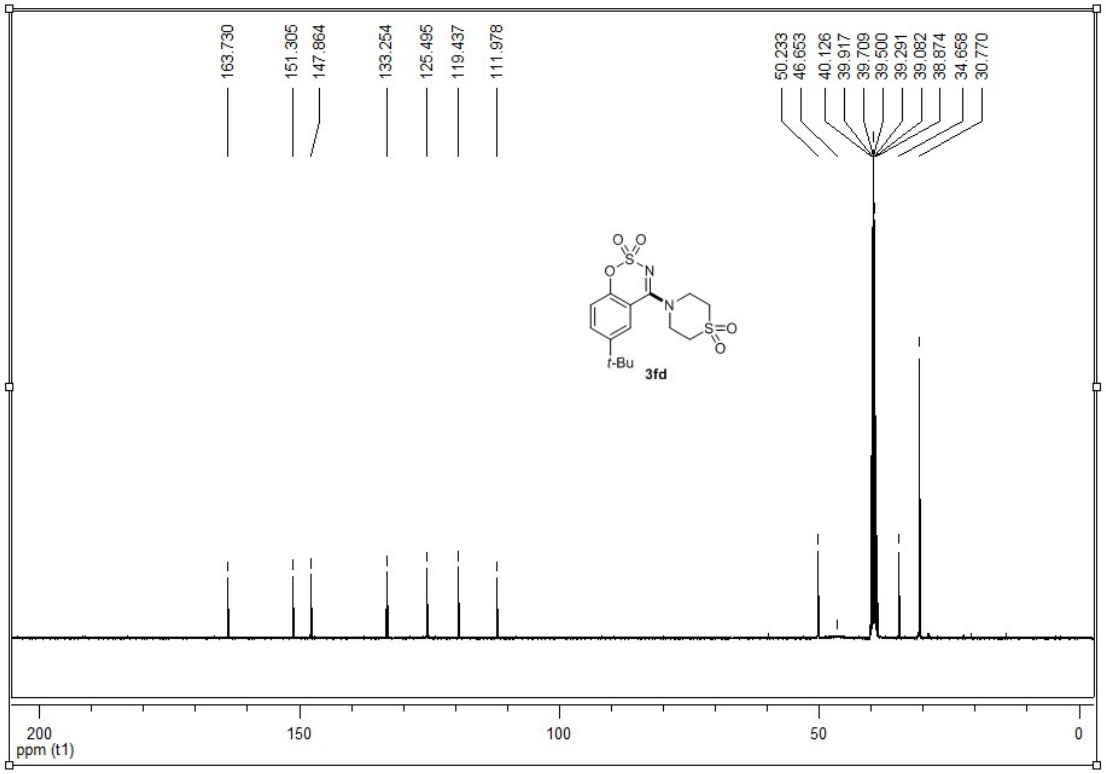

7.640
7.619
7.598
7.424
7.403
4.146
3.464
3.337
2.500
2.400
Me
3dd
1.00
1.00
4.00
4.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

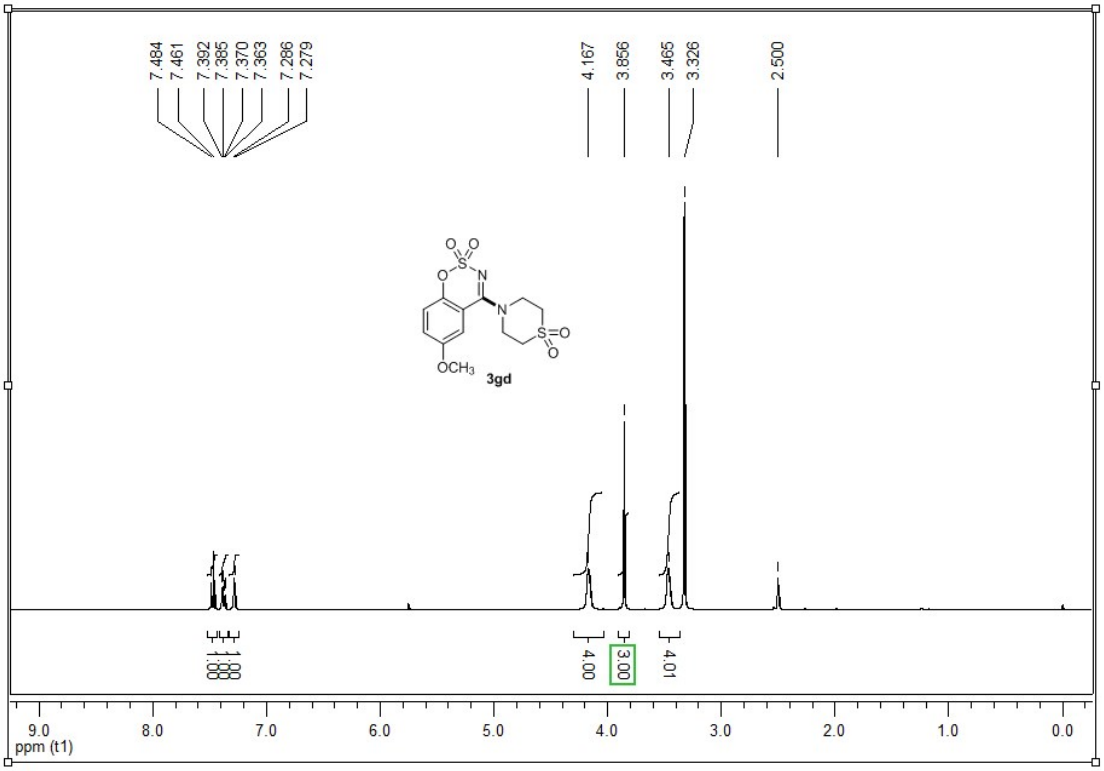

163.555
151.347
136.457
135.196
128.886
119.657
112.415
50.341
46.768
40.126
39.917
39.709
39.500
39.291
39.082
38.874
20.363
Me
3dd
200
150
100
50
0
ppm (t1)

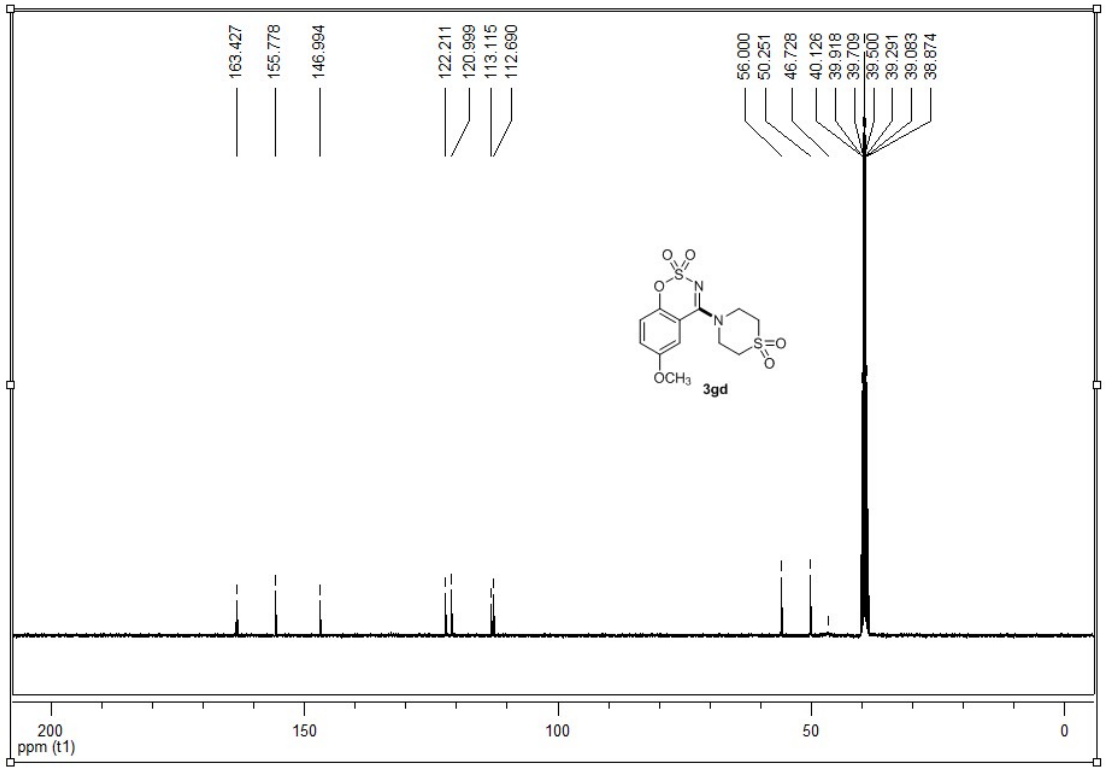

7.657
7.641
7.441
7.419
4.149
3.475
3.319
2.742
2.723
2.705
2.686
2.500
1.240
1.221
1.202
Et
3ed
2.01
1.00
4.00
4.01
2.00
3.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

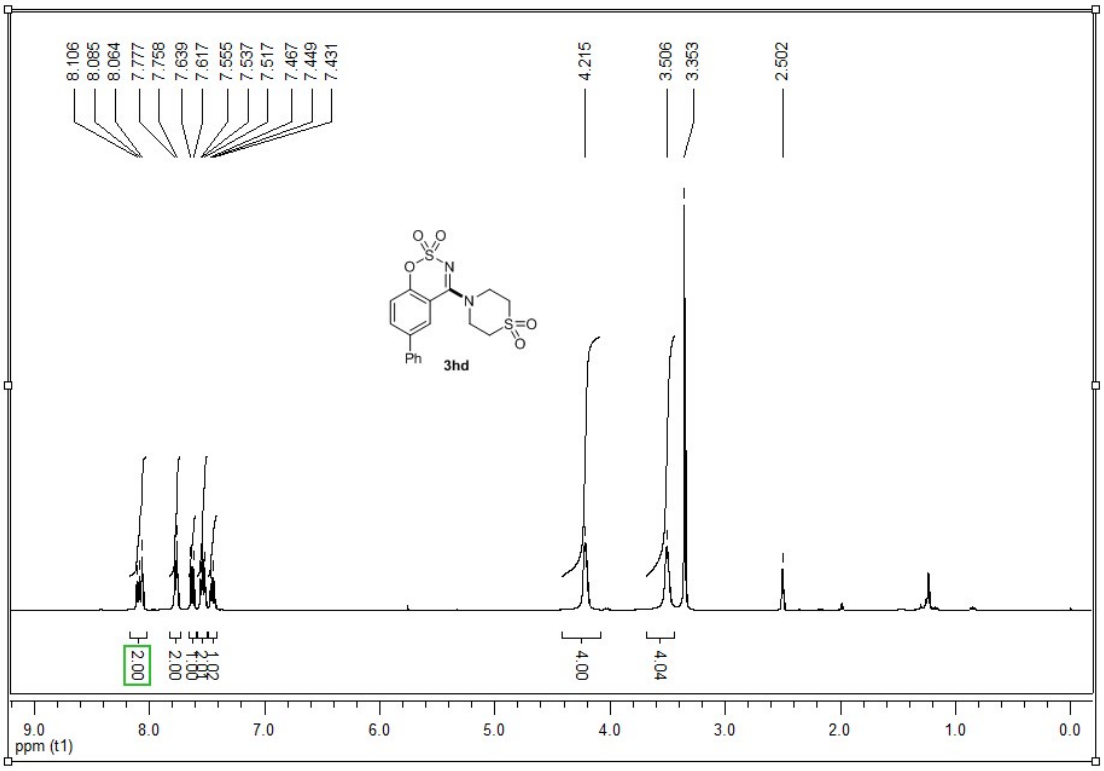

163.645
151.499
141.276
135.467
127.846
119.752
112.469
50.336
46.947
40.126
39.918
39.500
39.291
39.082
38.874
27.392
15.311
Et
3ed
200
150
100
50
0
ppm (t1)

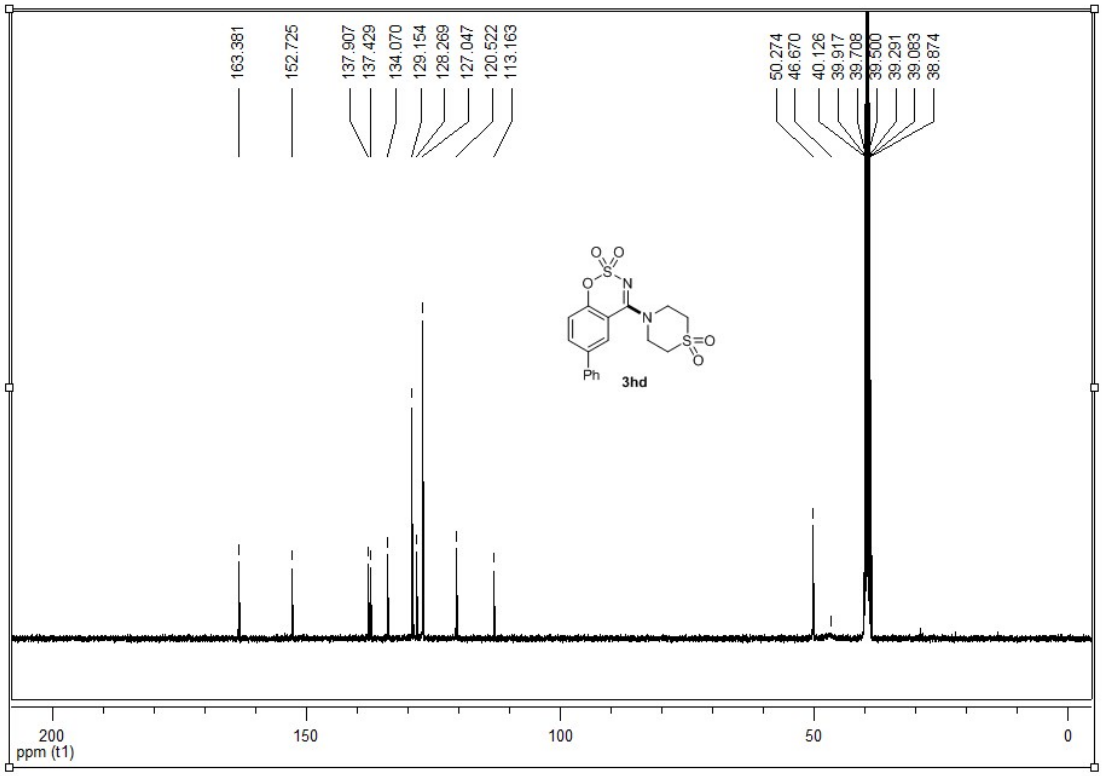

7.863
7.858
7.842
7.836
7.722
7.716
7.450
7.428
4.150
3.505
3.339
2.500
1.336
t-Bu
3fd
1.00
1.00
1.00
4.01
4.01
9.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

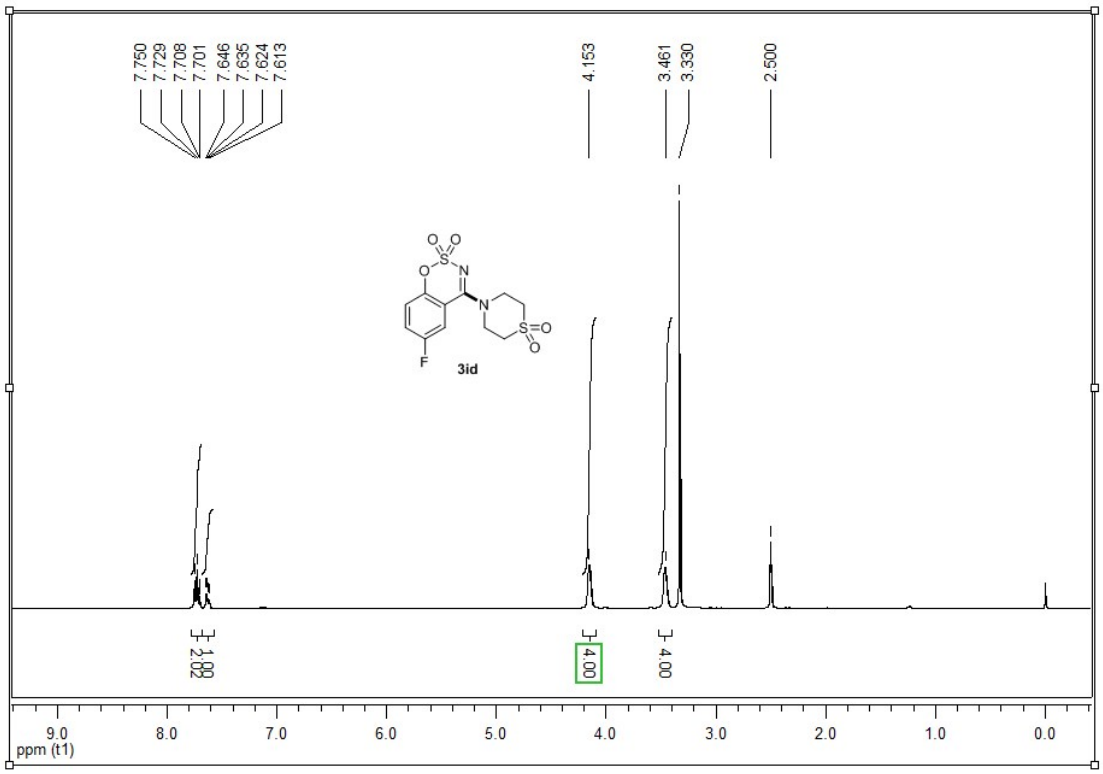

163.730
151.305
147.864
133.254
125.495
119.437
111.978
50.233
46.653
40.126
39.917
39.709
39.500
39.291
39.082
38.874
34.658
30.770
t-Bu
3fd
200
150
100
50
0
ppm (t1)

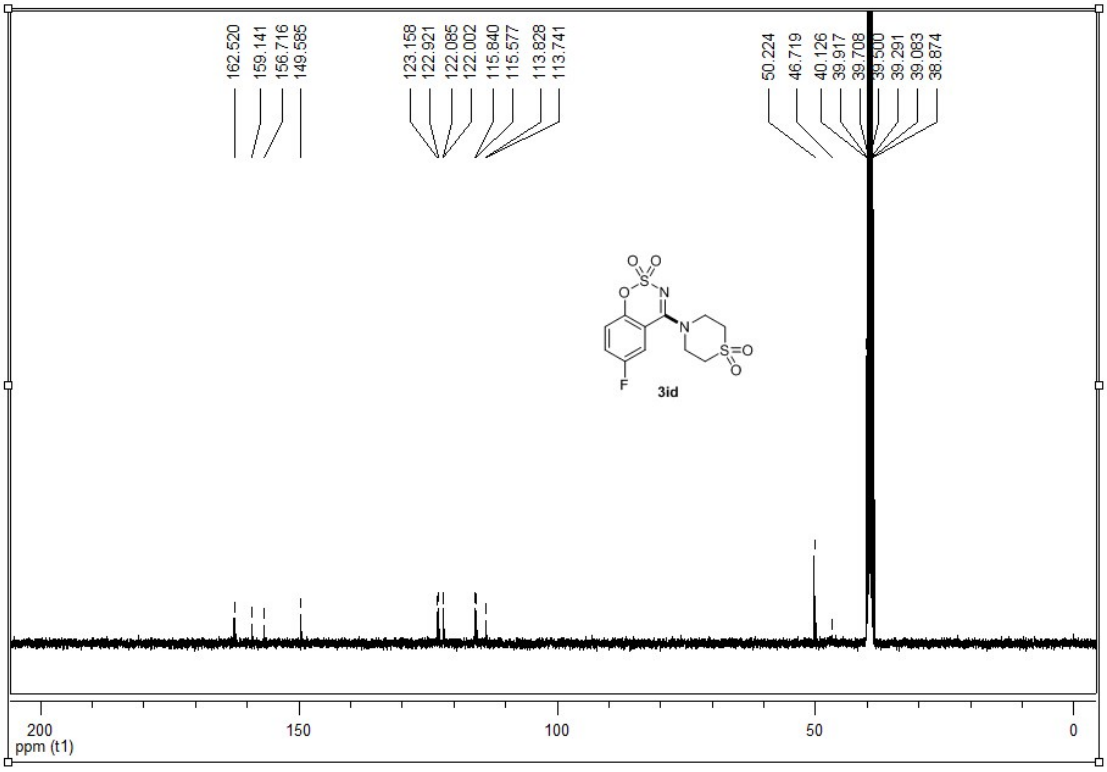

7.484
7.461
7.392
7.385
7.370
7.363
7.286
7.279
4.167
3.856
3.465
3.326
2.500
3gd
1.88
1.88
1.88
4.00
3.00
4.01
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

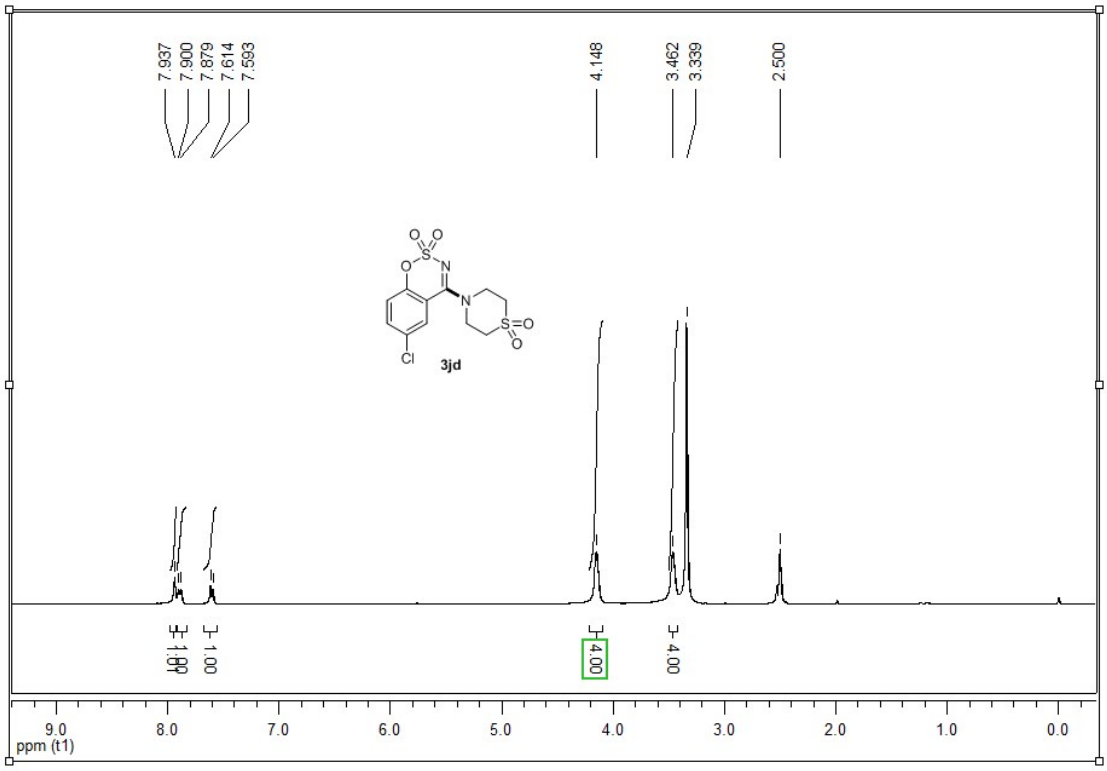

163.427
155.778
146.994
122.211
120.999
113.115
112.690
56.000
50.251
46.728
40.126
39.918
39.709
39.500
39.291
39.083
38.874
3gd
200
150
100
50
0
ppm (t1)

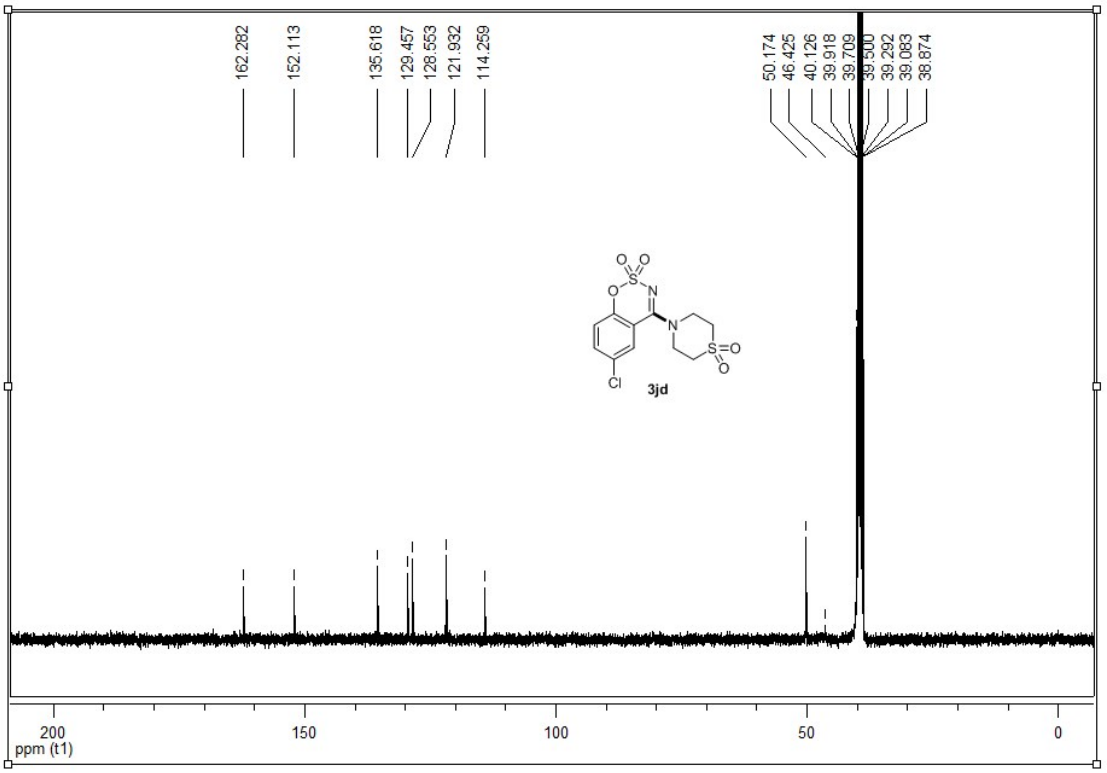

8.106
8.085
8.064
7.777
7.758
7.639
7.617
7.555
7.537
7.517
7.467
7.449
7.431
4.215
3.506
3.353
2.502
3hd
2.00
2.00
4.00
4.04
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

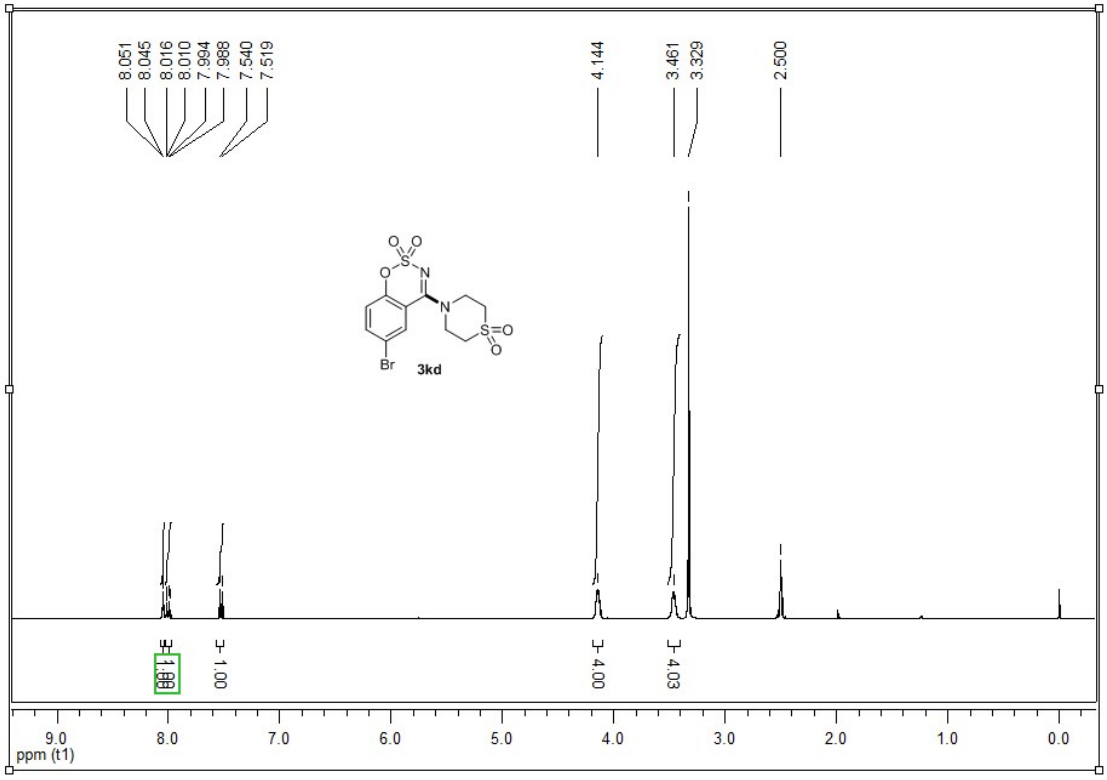

163.381
152.725
137.907
137.429
134.070
129.154
128.269
127.047
120.522
113.163
50.274
46.670
40.126
39.917
39.708
39.500
39.291
39.083
38.874
3hd
200
150
100
50
0
ppm (t1)

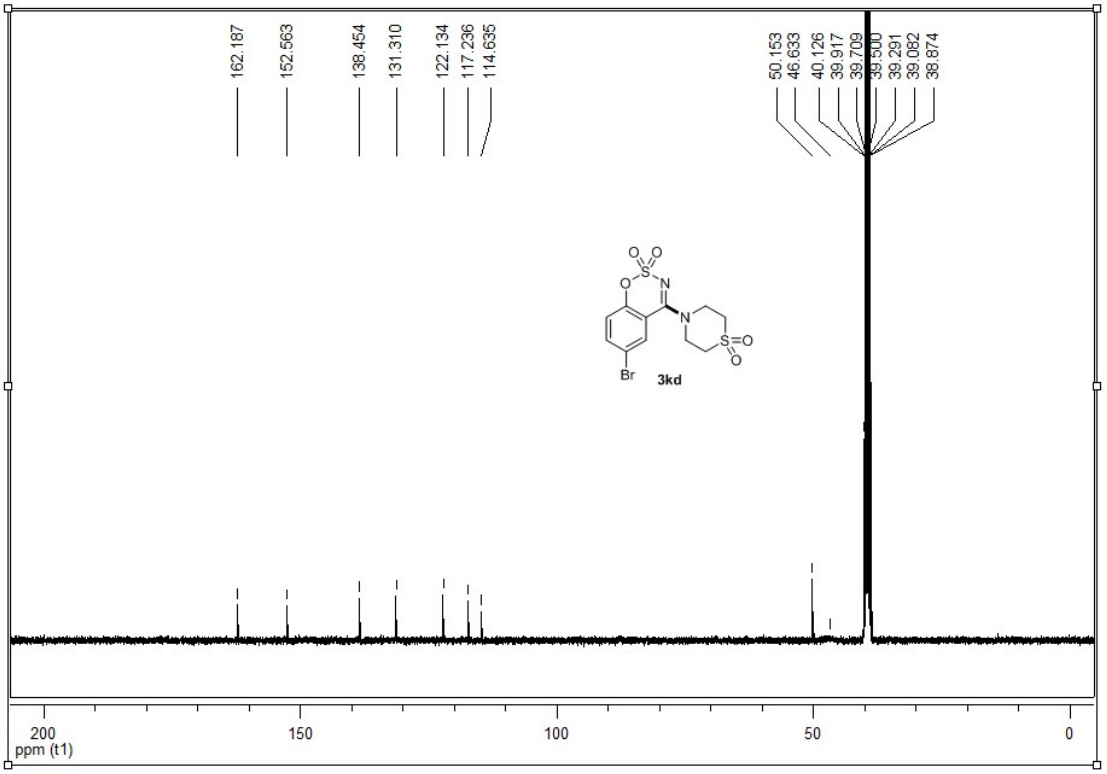

7.750
7.729
7.708
7.701
7.646
7.635
7.624
7.613
4.153
3.461
3.330
2.500
3id
2.02
4.00
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

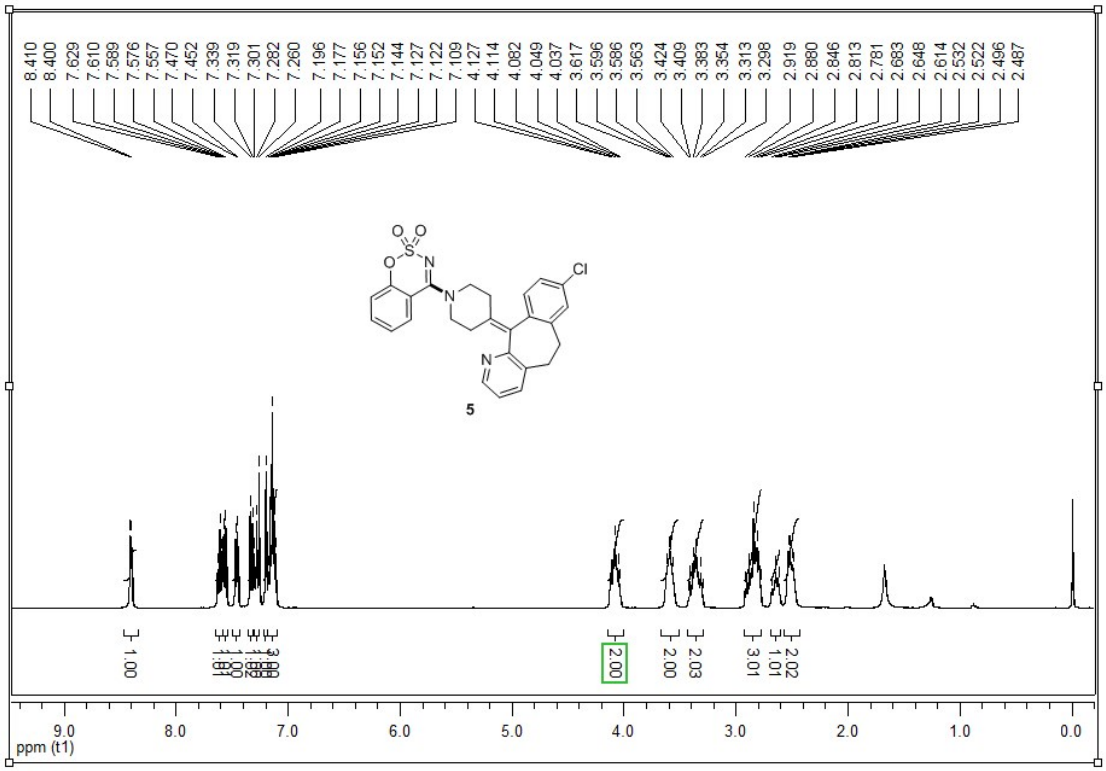

162.520
159.141
156.716
149.585
123.158
122.921
122.085
122.002
115.840
115.577
113.828
113.741
50.224
46.719
40.126
39.917
39.708
39.500
39.291
39.083
38.874
3id
200
150
100
50
0
ppm (t1)

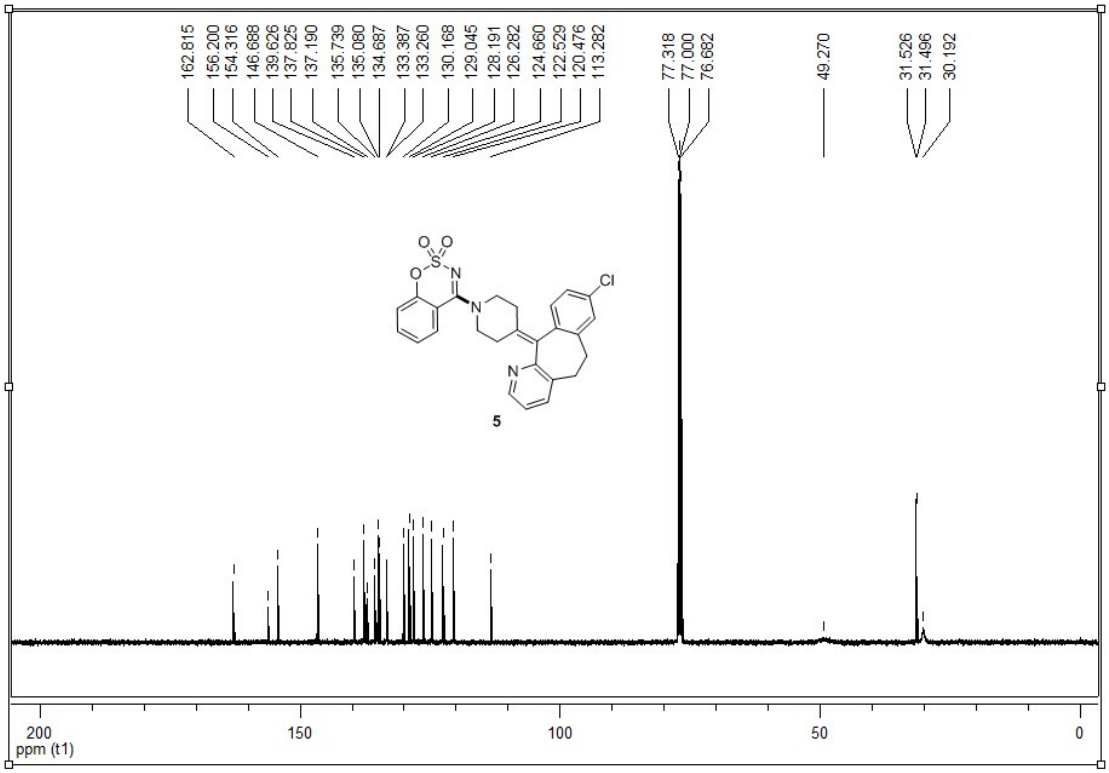

7.937
7.900
7.879
7.614
7.593
4.148
3.462
3.339
2.500
3jd
1.00
1.00
4.00
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


162.282
152.113
135.618
129.457
128.553
121.932
114.259
50.174
46.425
40.126
39.918
39.709
39.500
39.292
39.083
38.874
3jd
200
150
100
50
0
ppm (t1)


8.051
8.045
8.016
8.010
7.994
7.988
7.540
7.519
4.144
3.461
3.329
2.500
3kd
Br
2.00
1.00
4.00
4.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


162.187
152.563
138.454
131.310
122.134
117.236
114.635
50.153
46.633
40.126
39.917
39.709
39.500
39.291
39.082
38.874
3kd
Br
200
150
100
50
0
ppm (t1)


8.410
8.400
7.629
7.610
7.589
7.576
7.557
7.470
7.452
7.339
7.319
7.301
7.282
7.260
7.196
7.177
7.156
7.152
7.144
7.127
7.122
7.109
4.127
4.114
4.082
4.049
4.037
3.617
3.596
3.586
3.563
3.424
3.409
3.383
3.354
3.313
3.298
2.919
2.880
2.846
2.813
2.781
2.683
2.648
2.614
2.532
2.522
2.496
2.487
Cl
N
5
1.00
2.00
2.00
2.03
3.01
1.01
2.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


162.815
156.200
154.316
146.688
139.626
137.825
137.190
135.739
135.080
134.687
133.387
133.260
130.168
129.045
128.191
126.282
124.660
122.529
120.476
113.282
77.318
77.000
76.682
49.270
31.526
31.496
30.192
Cl
N
5
200
150
100
50
0
ppm (t1)

Crystallographic data for the product 3aa:

CCDC 1961174 contains the supplementary crystallographic data for the product **3aa**. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

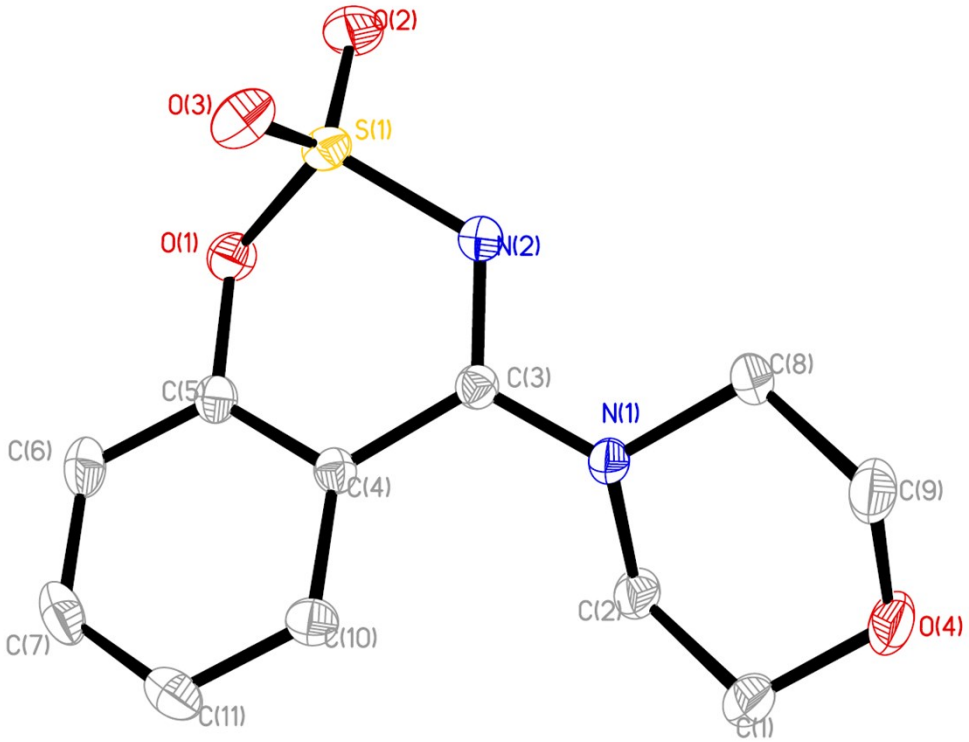


Table 1. Crystal data and structure refinement for 1.

Empirical formula	$C_{11}H_{12}N_2O_4S$
Formula weight	268.29
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 8.4413(11) A alpha = 95.122(5) deg. b = 8.9170(13) A beta = 107.332(5) deg. c = 9.0986(11) A gamma = 113.574(4) deg.
Volume	581.95(13) Å ^{3}
Z, Calculated density	2, 1.531 Mg/m^3
Absorption coefficient	0.287 mm^{-1}
F(000)	280
Crystal size	0.23 x 0.07 x 0.06 mm

Theta range for data collection	2.83 to 27.57 deg.
Limiting indices	-10<=h<=10, -10<=k<=11, -11<=l<=11
Reflections collected / unique	10933 / 2643 [R(int) = 0.0247]
Completeness to theta = 25.03	98.1%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2643 / 0 / 163
Goodness-of-fit on F^2	1.048
Final R indices [I>2sigma(I)]	R1 = 0.0321, wR2 = 0.0832
R indices (all data)	R1 = 0.0421, wR2 = 0.0888
Largest diff. peak and hole	0.268 and -0.413 $e.A^{-3}$