Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Generation of (2-oxoindolin-3-yl)methanesulfonohydrazides via a photo-induced reaction of *N*-(2-iodoaryl)acrylamide, DABSO, and hydrazine

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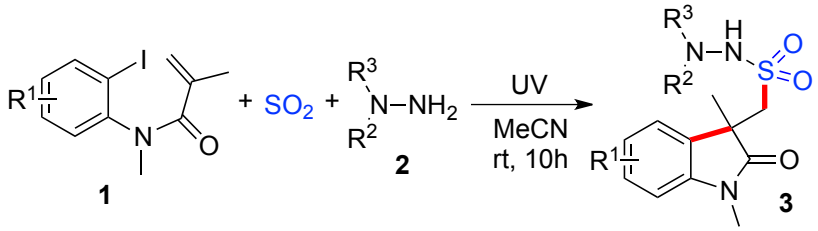
Supporting Information

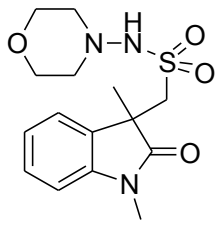
General experimental methods:

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

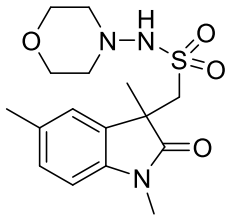
*General experimental procedure for the photo-induced three-component reaction ofN-(2-iodoaryl)acrylamide **1**, sulfur dioxide, and hydrazine **2***



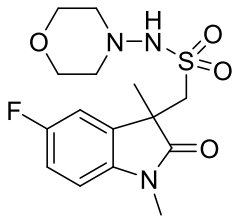
In a quartz tube, TBAI (0.3 mmol)and hydrazine **2** (0.3mmol) was added to a mixture of *N*-(2-iodoaryl)acrylamide **1** (0.2 mmol)and $DABCO^{\cdot}(SO_2)_2$(0.16 mmol) in MeCN (4.0 mL) under N_2 atmosphere. The mixture, placed around the mercury lamp (purchased from Yuming, Shanghai) with a distance of 10 centimeters, was stirred under UV irradiation (0.67W cm^{-1}) for 10 hours at room temperature. After the conversion was completed as indicated by TLC, the solvent was evaporated under reduced pressure. The residue was purified directly by flash column chromatograph to give the corresponding product **3**.



1-(1,3-Dimethyl-2-oxoindolin-3-yl)-*N*-morpholinomethanesulfonamide (**3a**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.4 Hz, 1H), 7.30 (dd, *J* = 11.2, 4.3 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 5.51 (s, 1H), 3.75 (d, *J* = 14.4 Hz, 1H), 3.72 (t, *J* = 4.6 Hz, 4H), 3.61 (d, *J* = 14.4 Hz, 1H), 3.22 (s, 3H), 2.83 – 2.80 (m, 4H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 143.2, 131.0, 129.0, 124.4, 122.9, 108.8, 66.8, 57.4, 55.9, 45.9, 26.8, 24.8. HRMS (ESI) calcd for $C_{15}H_{22}N_3O_4S^+$: 340.1326 (M + H^+), found: 340.1310. IR (KBr) 3481, 3198, 1699, 1614, 1472, 1309, 1153 cm^{-1}.

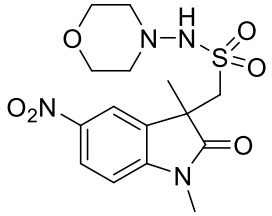


N-Morpholino-1-(1,3,5-trimethyl-2-oxoindolin-3-yl)methanesulfonamide (**3b**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (s, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 6.75 (d, *J* = 7.9 Hz, 1H), 5.37 (s, 1H), 3.76 (d, *J* = 14.4 Hz, 1H), 3.73 (t, *J* = 4.3 Hz, 4H), 3.60 (d, *J* = 14.5 Hz, 1H), 3.20 (s, 3H), 2.88 – 2.79 (m, 4H), 2.33 (s, 3H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 140.8, 132.4, 131.1, 129.2, 125.0, 108.5, 66.8, 57.4, 55.8, 46.1, 26.8, 24.8, 21.4. HRMS (ESI) calcd for$C_{16}H_{24}N_3O_4S^+$: 354.1482 (M + H^+), found: 354.1482. IR (KBr) 3445, 3164, 1694, 1606, 1456, 1335, 1154 cm^{-1}.

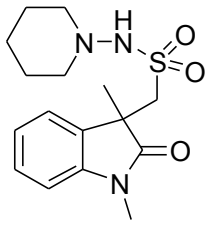


1-(5-Fluoro-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-morpholinomethanesulfonamide (**3c**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (dd, *J* = 7.9, 2.5 Hz, 1H), 7.00 (td, *J* = 8.9, 2.5 Hz, 1H),

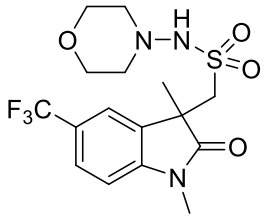
6.78 (dd, *J* = 8.5, 4.1 Hz, 1H), 5.37 (s, 1H), 3.77 (d, *J* = 12.1 Hz, 1H), 3.73 (t, *J* = 3.3 Hz, 4H), 3.60 (d, *J* = 14.4 Hz, 1H), 3.21 (s, 3H), 2.91 – 2.76 (m, 4H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 159.5(d, *J* = 240.9 Hz), 139.1, 132.6(d, *J* = 7.6 Hz), 115.2(d, *J* = 23.5 Hz), 112.7(d, *J* = 25.1 Hz), 109.2(d, *J* = 8.1 Hz), 66.8, 57.5, 55.8, 46.4, 27.0, 24.7. HRMS (ESI) calcd for $C_{15}H_{21}FN_3O_4S^+$: 358.1231(M + H^+), found: 358.1231. IR (KBr) 3238, 3184, 1698, 1622, 1471, 1329, 1150 cm^{-1}.



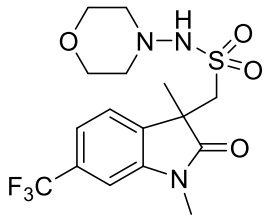
1-(1,3-Dimethyl-5-nitro-2-oxoindolin-3-yl)-*N*-morpholinomethanesulfonamide (**3d**). ^{1}H NMR (400 MHz, DMSO) δ 8.52 (d, *J* = 2.3 Hz, 1H), 8.40 (s, 1H), 8.27 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.25 (d, *J* = 8.7 Hz, 1H), 4.07 (d, *J* = 14.3 Hz, 1H), 3.66 (d, *J* = 14.3 Hz, 1H), 3.59 (t, *J* = 4.4Hz, 4H), 3.20 (s, 3H), 2.74 – 2.72 (m, 4H), 1.39 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 178.2, 149.4, 142.3, 131.9, 125.5, 120.5, 108.7, 66.0, 56.2, 55.1, 45.1, 26.8, 24.1. HRMS (ESI) calcd for$C_{15}H_{21}N_4O_6S^+$: 385.1176 (M + H^+), found: 385.1177. IR (KBr) 3446, 3232, 1717, 1615, 1523, 1456, 1335, 1303, 1160 cm^{-1}.



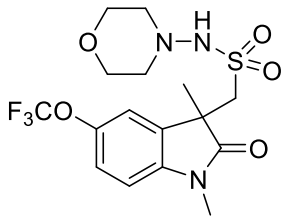
1-(1,3-Dimethyl-2-oxoindolin-3-yl)-*N*-(piperidin-1-yl)methanesulfonamide (**3e**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.4 Hz, 1H), 7.32 - 7.26 (m, 1H), 7.08 (t, *J* = 7.5, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 5.18 (s, 1H), 3.71 (d, *J* = 14.3 Hz, 1H), 3.62 (d, *J* = 14.3 Hz, 1H), 3.21 (s, 3H), 2.72 (d, *J* = 4.3 Hz, 4H), 1.64 – 1.58 (m, 4H), 1.47 (s, 3H), 1.37 – 1.30 (m, 2H). ^{13}C NMR (100MHz, $CDCl_3$) δ 178.2, 143.0, 130.9, 128.6, 124.3, 122.6, 108.4, 58.2, 55.6, 45.7, 26.5, 25.5, 24.3, 23.1. HRMS (ESI) calcd for $C_{16}H_{24}N_3O_3S^+$: 338.1533 (M + H^+), found: 338.1545. IR (KBr) 3479, 3219, 1705, 1613, 1472, 1314, 1156 cm^{-1}.



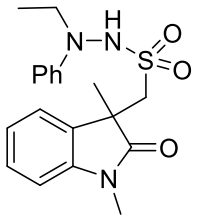
1-(1,3-Dimethyl-2-oxo-5-(trifluoromethyl)indolin-3-yl)-*N*-morpholinomethanesulfonamide (**3f**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 10.8 Hz, 2H), 6.93 (d, *J* = 8.1 Hz, 1H), 5.25 (s, 1H), 3.81 (d, *J* = 14.5 Hz, 1H), 3.73 (t, *J* = 4.6 Hz, 4H), 3.65 (d, *J* = 14.5 Hz, 1H), 3.25 (s, 3H), 2.83 – 2.77 (m, 4H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 146.3, 131.4, 127.2 (q, *J* = 271.0 Hz), 126.7 (q, *J* = 3.6 Hz), 121.6 (q, *J* = 3.3 Hz), 108.6, 66.8, 66.5, 57.5, 55.9, 45.9, 27.0, 24.8.HRMS (ESI) calcd for$C_{16}H_{21}F_3N_3O_4S^+$: 408.1199 ($M + H^+$), found: 408.1217. IR (KBr) 3439, 3251, 1713, 1625, 1461, 1326, 1144 cm^{-1}.



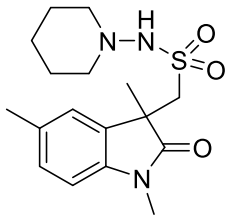
1-(1,3-Dimethyl-2-oxo-6-(trifluoromethyl)indolin-3-yl)-*N*-morpholinomethanesulfonamide (**3g**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 7.7 Hz, 1H), 7.37 (d, *J* = 7.7 Hz, 1H), 7.07 (s, 1H), 5.36 (s, 1H), 3.81 (d, *J*= 14.4 Hz, 1H), 3.74 (t, *J* = 4.4 Hz, 4H), 3.64 (d, *J* = 14.4 Hz, 1H), 3.26 (s, 3H), 2.85 – 2.82 (m, 4H), 1.47 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.0, 143.8, 134.8, 130.9 (q, *J* = 32.4 Hz), 124.7, 120.5 (q, *J* = 271.0 Hz), 119.9 (q, *J* = 4.1 Hz), 105.5 (q, *J* = 3.7 Hz), 66.8, 57.4, 55.7, 45.9, 29.9, 27.0, 24.8.HRMS (ESI) calcd for$C_{16}H_{21}F_3N_3O_4S^+$: 408.1199 ($M + H^+$), found: 408.1207. IR (KBr) 3515, 3182, 1716, 1627, 1458, 1321, 1165 cm^{-1}.



1-(1,3-Dimethyl-2-oxo-5-(trifluoromethoxy)indolin-3-yl)-*N*-morpholinomethanesulfonamide (**3h**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (s, 1H), 7.19 (d, *J* = 8.5 Hz, 1H), 6.84 (d, *J* = 8.5 Hz, 1H), 5.37 (s, 1H), 3.76 (d, *J* = 14.4 Hz, 1H), 3.73 (t, *J* = 4.6 Hz, 4H), 3.61 (d, *J* = 14.4 Hz, 1H), 3.22 (s, 3H), 2.82 – 2.78 (m, 4H), 1.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 145.0, 141.9, 132.4, 122.0, 120.4 (q, *J* = 256.4 Hz), 118.7, 109.1, 66.8, 57.5, 55.8, 46.2, 27.0, 24.6. HRMS (ESI) calcd for $C_{16}H_{21}F_3N_3O_5S^+$: 424.1149 ($M + H^+$), found: 424.1135. IR (KBr) 3446, 3192, 1712, 1622, 1470, 1328, 1158 cm^{-1}.

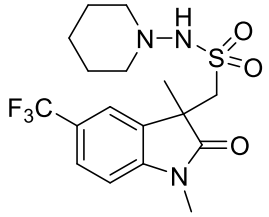


1-(1,3-Dimethyl-2-oxoindolin-3-yl)-*N*'-ethyl-*N*'-phenylmethanesulfonohydrazide (**3i**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.4 Hz, 1H), 7.32 – 7.25 (m, 3H), 7.07 (td, *J* = 7.6, 0.8 Hz, 1H), 6.97 (d, *J* = 7.9 Hz, 2H), 6.92 (t, *J* = 7.3 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 6.27 (s, 1H), 3.68 (d, *J* = 14.1 Hz, 1H), 3.63 – 3.58 (m, 2H), 3.56 (d, *J* = 14.1 Hz, 1H), 3.20 (s, 3H), 1.41 (s, 3H), 1.03 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 148.1, 143.2, 130.7, 129.6, 129.0, 124.6, 122.9, 121.4, 115.8, 108.7, 57.7, 50.2, 45.9, 26.8, 24.7, 9.5. HRMS (ESI) calcd for $C_{19}H_{24}N_3O_3S^+$: 374.1533 ($M + H^+$), found: 374.1527. IR (KBr) 3439, 3177, 1701, 1615, 1472, 1329, 1159 cm^{-1}.

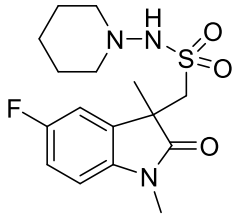


N-(Piperidin-1-yl)-1-(1,3,5-trimethyl-2-oxoindolin-3-yl)methanesulfonamide (**3j**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (s, 1H), 7.08 (d, *J* = 7.8 Hz, 1H), 6.74 (d, *J* = 7.9 Hz, 1H), 5.23 (s, 1H), 3.69 (d, *J* = 14.4 Hz, 1H), 3.61 (d, *J* = 14.4 Hz, 1H), 3.19 (s, 3H), 2.74 (d, *J* = 4.0 Hz, 4H), 2.33 (s, 3H), 1.67 – 1.58 (m, 4H), 1.45 (s, 3H), 1.37 – 1.32 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 140.8, 132.3, 131.3, 129.1, 125.2, 108.4, 58.4, 55.8,

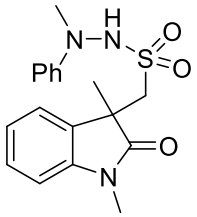
46.1, 26.8, 25.7, 24.7, 23.3, 21.4. HRMS (ESI) calcd for $C_{17}H_{26}N_3O_3S^+$: 352.1689 (M + H^+), found: 352.1684. IR (KBr) 3475, 3210, 1704, 1620, 1473, 1310, 1158 cm^{-1}.



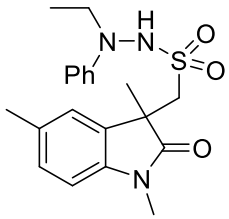
1-(1,3-Dimethyl-2-oxo-5-(trifluoromethyl)indolin-3-yl)-*N*-(piperidin-1-yl)methanesulfonamide (**3k**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.2 Hz, 1H), 6.85 (s, 1H), 6.53 (d, *J* = 8.2 Hz, 1H), 4.81 (s, 1H), 3.37 (d, *J* = 14.4 Hz, 1H), 3.26 (d, *J* = 14.4 Hz, 1H), 2.85 (s, 3H), 2.31 (d, *J* = 3.9 Hz, 4H), 1.26 – 1.20 (m, 4H), 1.08 (s, 3H), 0.97 – 0.93 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 146.1, 131.3, 126.4 (q, *J* = 3.9 Hz), 124.6 (q, *J* = 32.6 Hz), 124.4 (q, *J* = 270.0 Hz), 121.5 (q, *J* = 3.6 Hz), 108.2, 58.2, 55.5, 45.6, 26.8, 25.5, 24.5, 23.0. HRMS (ESI) calcd for $C_{17}H_{23}F_3N_3O_3S^+$: 406.1407 (M + H^+), found: 406.1401. IR (KBr) 3401, 3210, 1715, 1606, 1475, 1328, 1169 cm^{-1}.



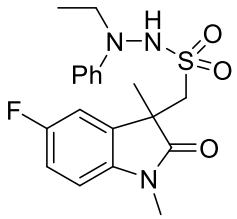
1-(5-Fluoro-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-(piperidin-1-yl)methanesulfonamide (**3l**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 6.1 Hz, 1H), 6.99 (t, *J* = 7.7 Hz, 1H), 6.77 (dd, *J* = 8.4, 4.0 Hz, 1H), 5.22 (s, 1H), 3.70 (d, *J* = 14.3 Hz, 1H), 3.60 (d, *J* = 14.3 Hz, 1H), 3.20 (s, 3H), 2.74 (d, *J* = 4.4 Hz, 4H), 1.64 –1.61 (m, 4H), 1.47 (s, 3H), 1.38 – 1.33 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 159.2 (d, *J* = 240.7 Hz), 138.9, 132.5 (d, *J* = 8.2 Hz), 114.9 (d, *J* = 23.5 Hz), 112.7 (d, *J* = 25.2 Hz), 108.9 (d, *J* = 8.3 Hz), 58.3, 55.5, 46.2, 26.7,25.5, 24.3, 23.1. HRMS (ESI) calcd for $C_{16}H_{23}FN_3O_3S^+$: 356.1439 (M + H^+), found: 356.1436. IR (KBr) 3479, 3221, 1707, 1610, 1473, 1305, 1157 cm^{-1}.



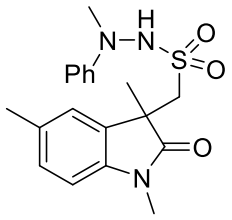
1-(1,3-Dimethyl-2-oxoindolin-3-yl)-*N*'-methyl-*N*'-phenylmethanesulfonohydrazide (**3m**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 7.4, 0.6 Hz, 1H), 7.32 – 7.25 (m, 3H), 7.07 (td, *J* = 7.6, 0.9 Hz, 1H), 6.98 (dd, *J* = 8.8, 0.9 Hz, 2H), 6.92 (t, *J* = 7.3 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 6.05 (s, 1H), 3.74 (d, *J* = 14.0 Hz, 1H), 3.61 (d, *J* = 14.1 Hz, 1H), 3.21 (s, 3H), 3.19 (s, 3H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 150.0, 143.3, 130.6, 129.5, 129.1, 124.6, 122.9, 121.4, 114.7, 108.8, 57.7, 45.9, 44.3, 26.8, 24.7. HRMS (ESI) calcd for $C_{18}H_{22}N_3O_3S^+$: 360.1376 (M + H^+), found: 360.1365. IR (KBr) 3412, 3189, 1713, 1599, 1471, 1309, 1154 cm^{-1}.



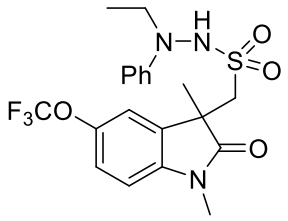
N'-Ethyl-*N*'-phenyl-1-(1,3,5-trimethyl-2-oxoindolin-3-yl)methanesulfonohydrazide (**3n**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (dd, *J* = 8.7, 7.4 Hz, 2H), 7.19 (s, 1H), 7.09 (dd, *J* = 7.9, 0.8 Hz, 1H), 6.99 (d, *J* = 7.9 Hz, 2H), 6.92 (t, *J* = 7.3 Hz, 1H), 6.74 (d, *J* = 7.9 Hz, 1H), 6.39 (s, 1H), 3.68 (d, *J* = 14.2 Hz, 1H), 3.64 – 3.58 (m, 2H), 3.55 (d, *J* = 14.2 Hz, 1H), 3.18 (s, 3H), 2.32 (s, 3H), 1.41 (s, 3H), 1.04 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 148.1, 140.9, 132.4, 130.8, 129.6, 129.3, 125.3, 121.5, 115.7, 108.5, 57.8, 50.2, 46.1, 26.8, 24.8, 21.4, 9.5. HRMS (ESI) calcd for $C_{20}H_{26}N_3O_3S^+$: 388.1689 (M + H^+), found: 388.1673. IR (KBr) 3380, 3137, 1699, 1601, 1454, 1302, 1160 cm^{-1}.



N'-Ethyl-1-(5-fluoro-1,3-dimethyl-2-oxoindolin-3-yl)-*N*'-phenylmethanesulfonohydrazide (**3o**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 8.5, 7.5 Hz, 2H), 7.14 (dd, *J* = 8.0, 2.5 Hz, 1H), 6.98 (dd, *J* = 8.9, 2.5 Hz, 3H), 6.93 (t, *J* = 7.3 Hz, 1H), 6.76 (dd, *J* = 8.5, 4.1 Hz, 1H), 6.40 (s, 1H), 3.67 (d, *J* = 14.1 Hz, 1H), 3.64 – 3.58 (m, 2H), 3.54 (d, *J* = 14.1 Hz, 1H), 3.18 (s, 3H), 1.41 (s, 3H), 1.03 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 159.4 (d, *J* = 239.6 Hz), 148.1, 139.2, 132.3 (d, *J* = 8.6 Hz), 129.7, 121.6, 115.9, 115.3 (d, *J* = 23.6 Hz), 113.0 (d, *J* = 25.2 Hz), 109.1 (d, *J* = 8.1 Hz), 57.5, 50.4, 46.4, 27.0, 24.6, 9.5. HRMS (ESI) calcd for $C_{19}H_{23}FN_3O_3S^+$: 392.1439 (M + H^+), found: 392.1425. IR (KBr) 3390, 3187, 1705, 1599, 1443, 1328, 1158 cm^{-1}.

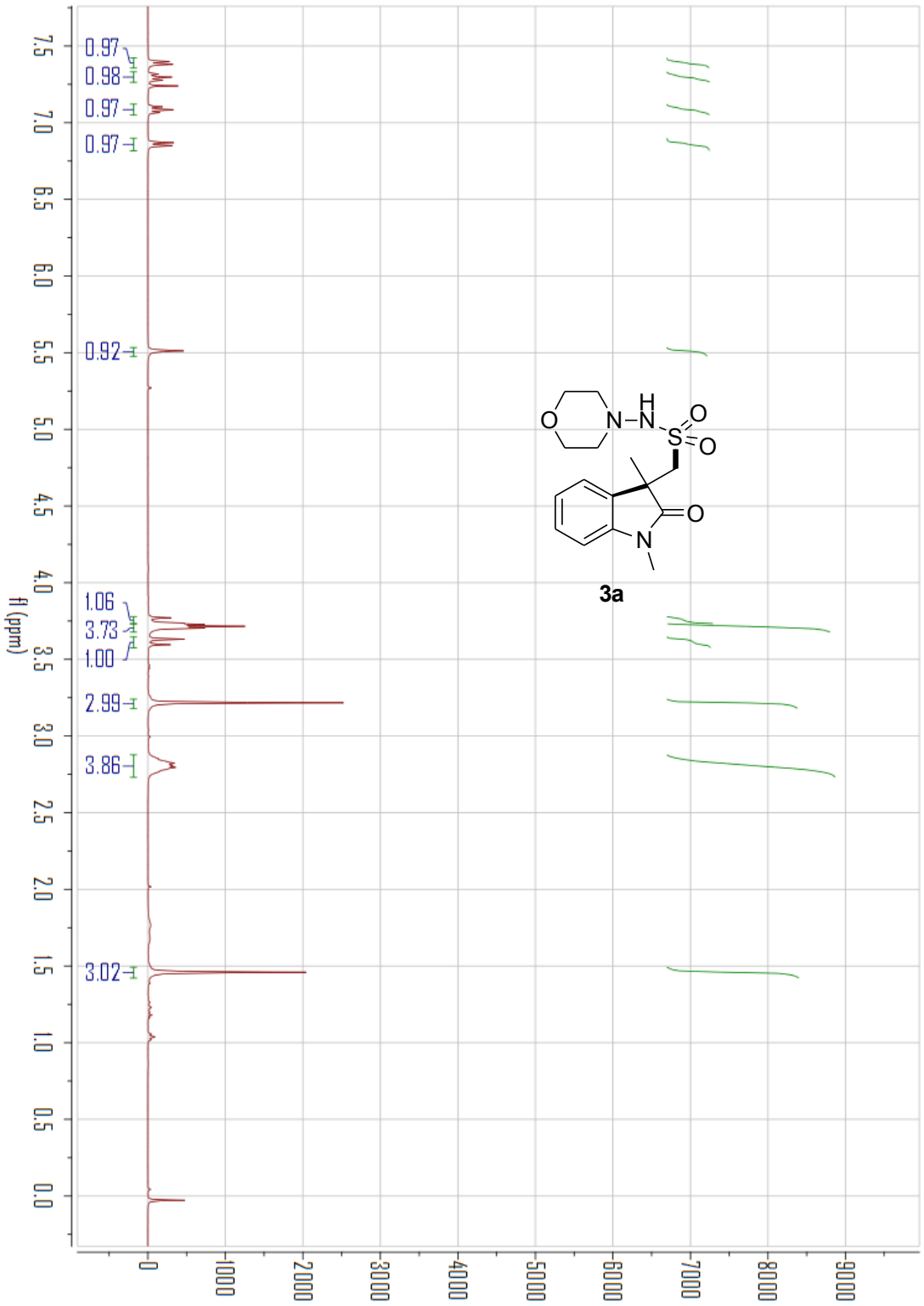


N'-Methyl-*N*'-phenyl-1-(1,3,5-trimethyl-2-oxoindolin-3-yl)methanesulfonohydrazide (**3p**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 - 7.24 (m, 2H), 7.20 (s, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 6.99 (d, *J* = 8.1 Hz, 2H), 6.92 (t, *J* = 7.3 Hz, 1H), 6.74 (d, *J* = 7.9 Hz, 1H), 6.35 (s, 1H), 3.73 (d, *J* = 14.1 Hz, 1H), 3.59 (d, *J* = 14.1 Hz, 1H), 3.18 (s, 3H), 3.17 (s, 3H), 2.31 (s, 3H), 1.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 150.1, 140.8, 132.4, 130.7, 129.4, 129.3, 125.3, 121.2, 114.7, 108.5, 57.7, 46.0, 44.1, 26.8, 24.8, 21.4. HRMS (ESI) calcd for $C_{19}H_{24}N_3O_3S^+$: 374.1533 (M + H^+), found: 374.1542. IR (KBr) 3486, 3168, 1711, 1601, 1453, 1295, 1154 cm^{-1}.



1-(1,3-Dimethyl-2-oxo-5-(trifluoromethoxy)indolin-3-yl)-*N*'-ethyl-*N*'-phenylmethanesulfonohydrazide (**3q**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.25 (m, 3H), 7.17 (d, *J* = 8.5 Hz, 1H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.92 (d, *J* = 7.3 Hz, 1H), 6.83 (d, *J* = 8.5 Hz, 1H), 6.36 (s,

1H), 3.67 (d, *J* = 14.1 Hz, 1H), 3.63 – 3.57 (m, 2H), 3.54 (d, *J* = 14.1 Hz, 1H), 3.19 (s, 3H), 1.43 (s, 3H), 1.02 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 148.1, 144.9, 141.9, 132.2, 129.6, 123.4 (q, *J* = 231.4 Hz), 122.1, 121.6, 118.9, 115.8, 109.0, 57.2, 50.4, 46.2, 26.9, 24.5, 9.5. HRMS (ESI) calcd for $C_{20}H_{23}F_3N_3O_4S^+$: 458.1356 (M + H^+), found: 458.1355. IR (KBr) 3393, 3188, 1704, 1600, 1453, 1328, 1158 cm^{-1}.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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
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3000
4000
5000
6000
7000
8000
9000
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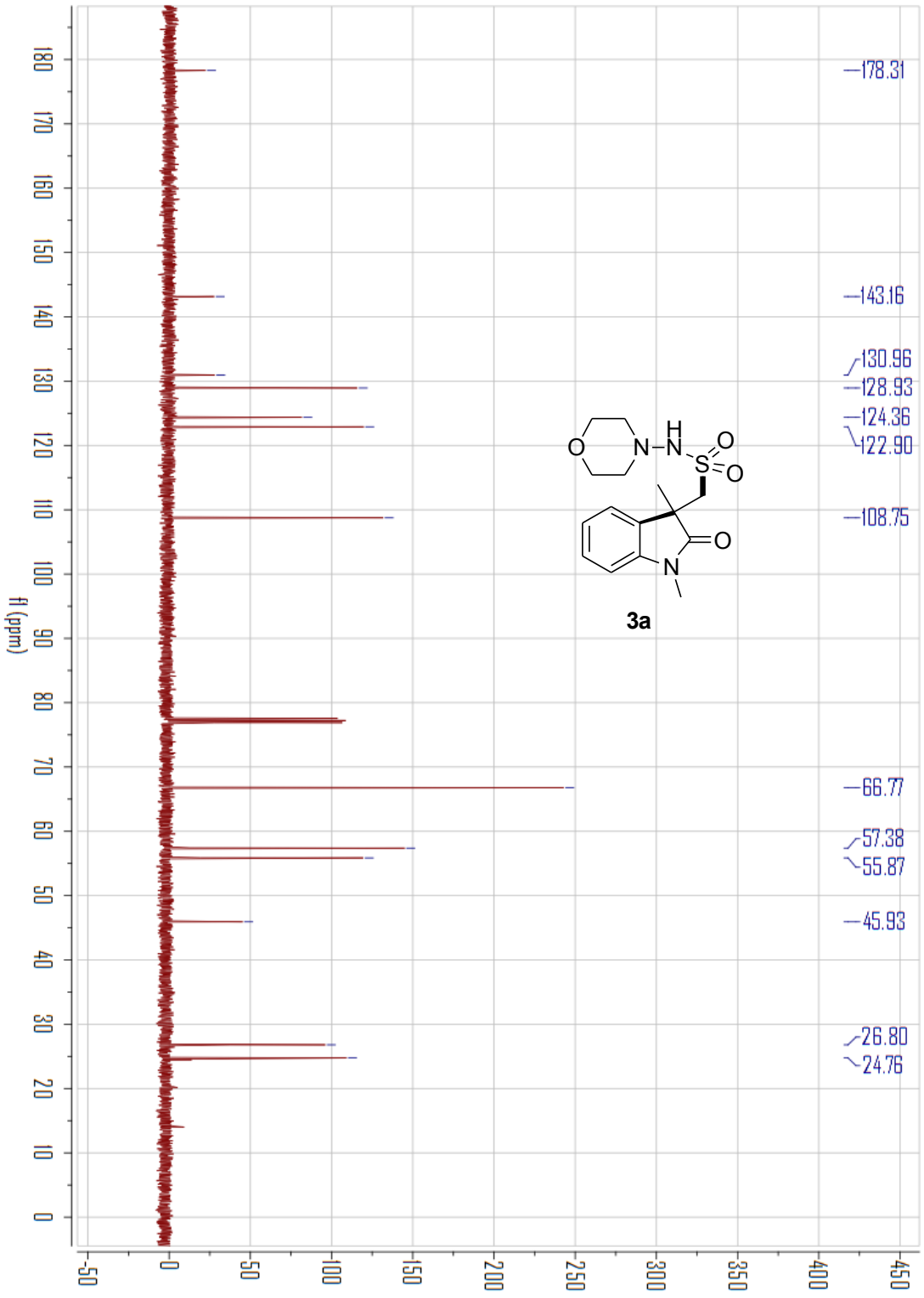
H
N
S
O
O
O
N

3a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24.76
f1 (ppm)
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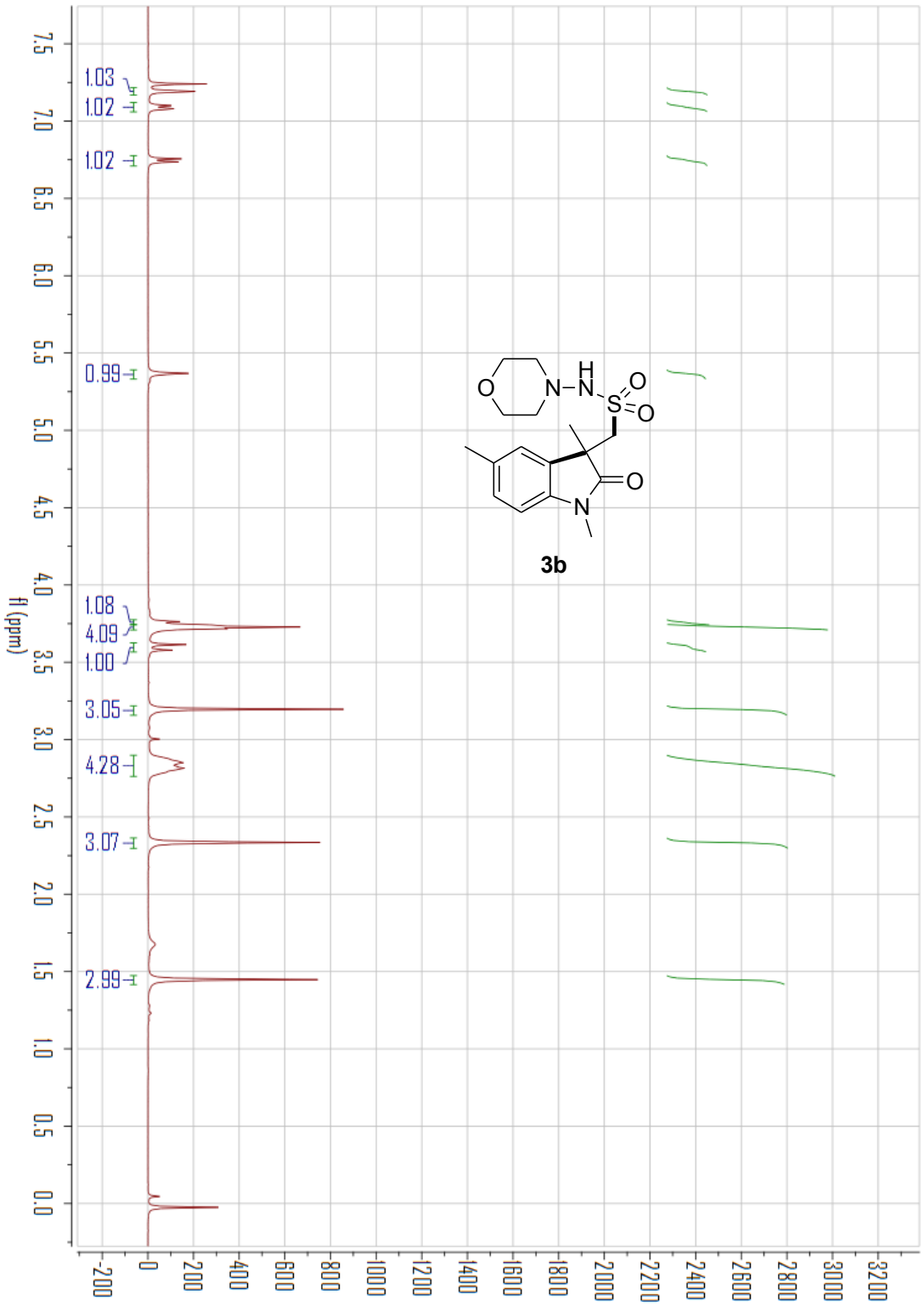
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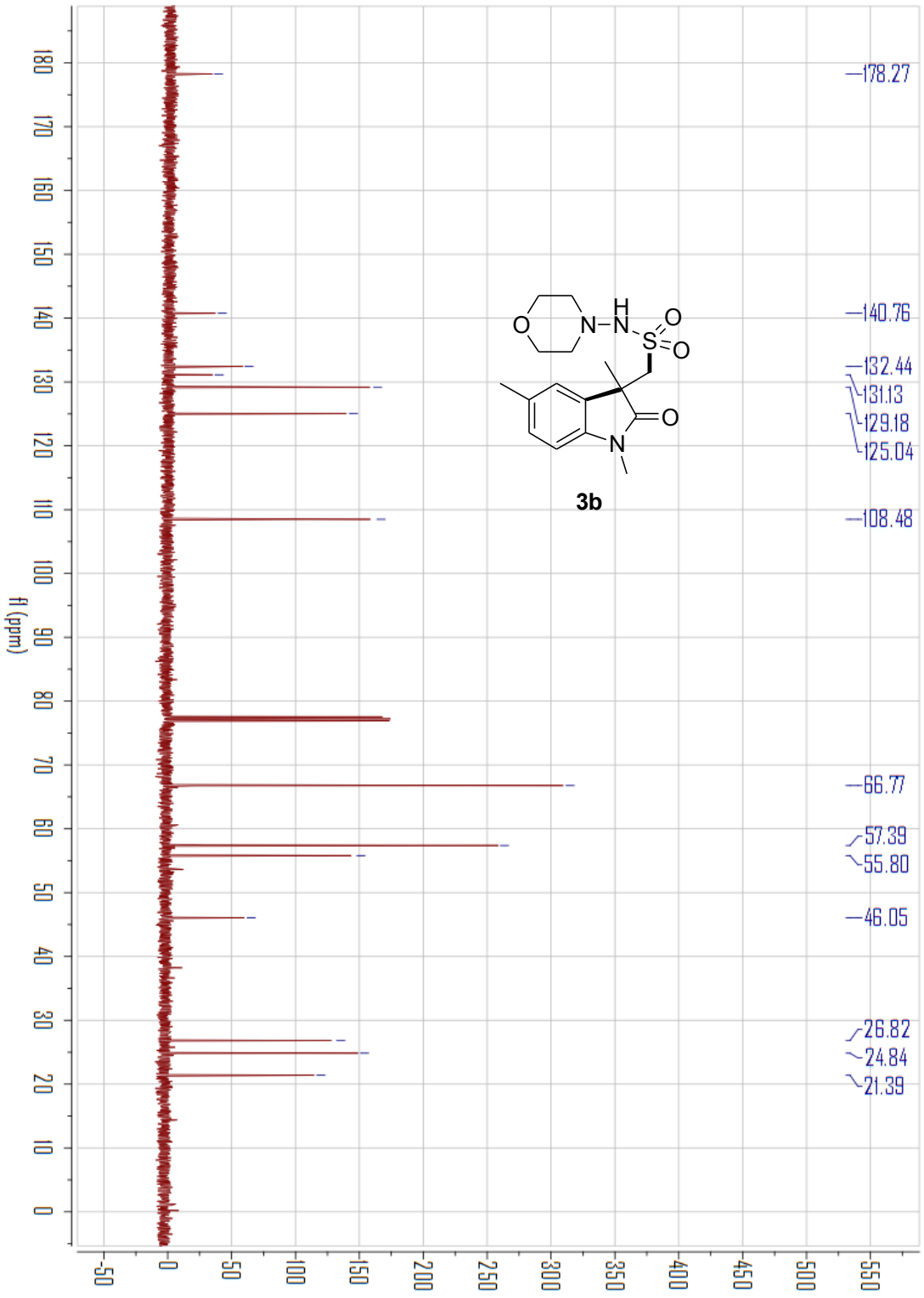
H
N
S
O
O
O
N
3b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f1 (ppm)
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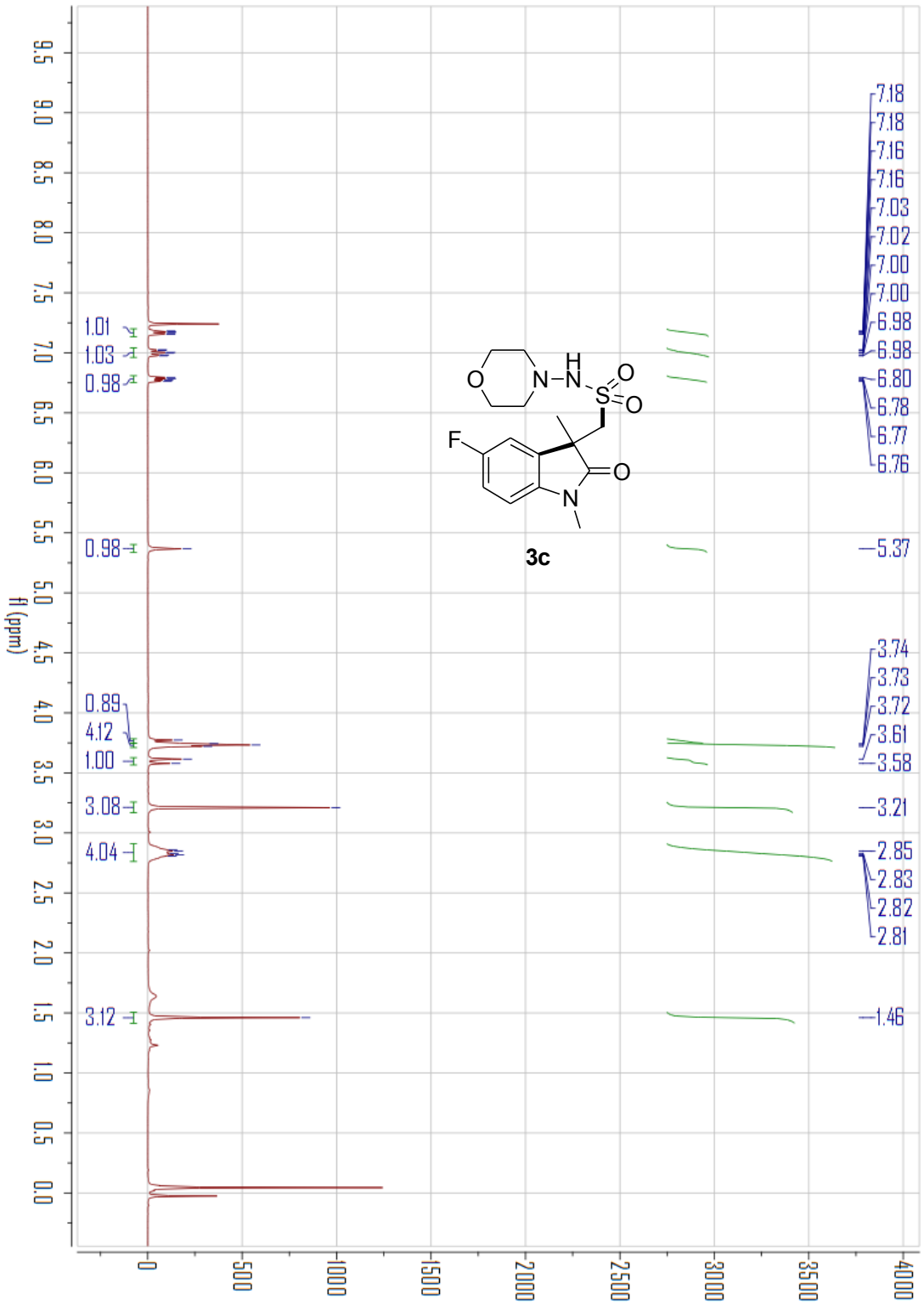
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3.61
3.58
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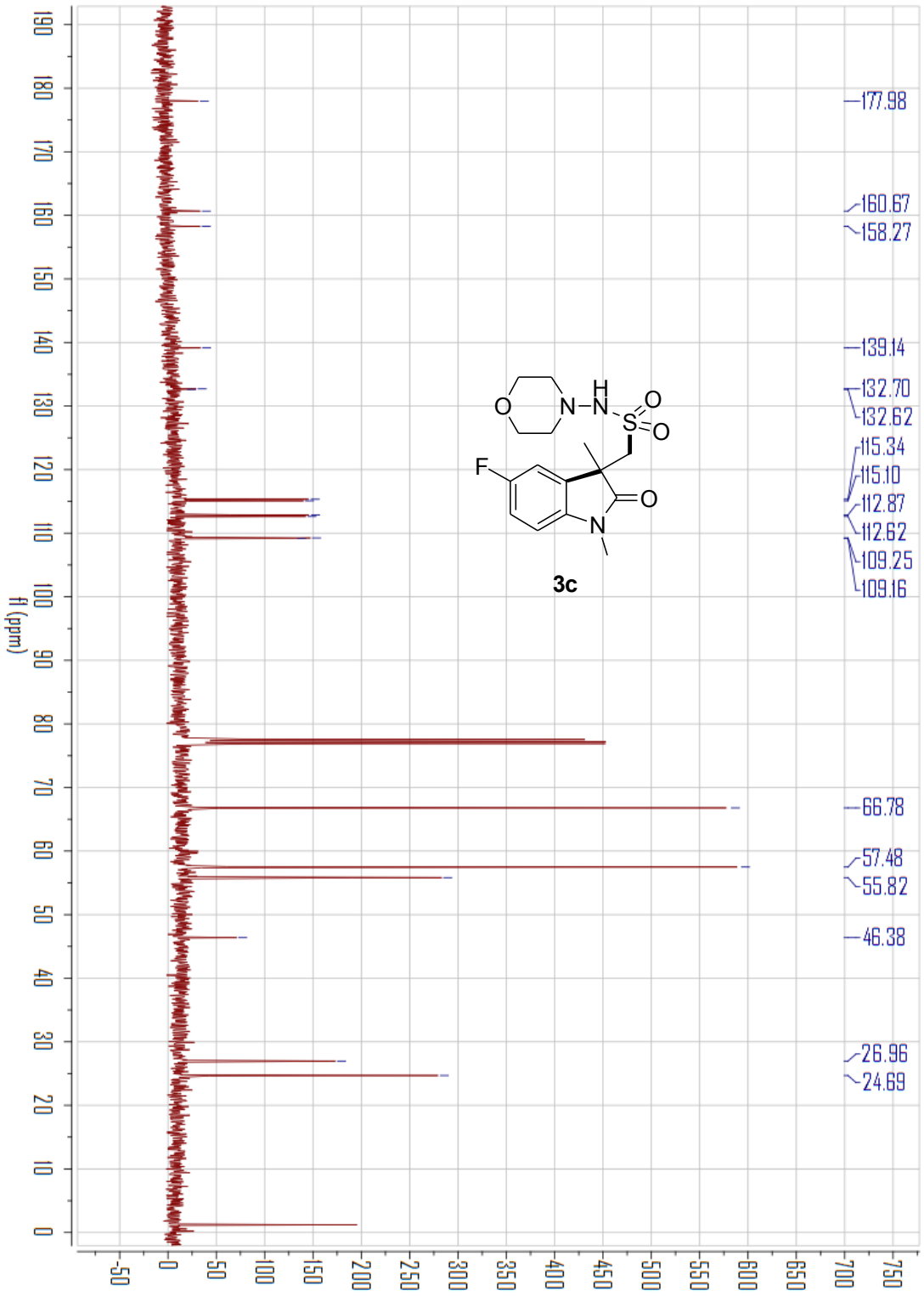
N
S
O
O
F
O
N

3c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55.82
46.38
26.96
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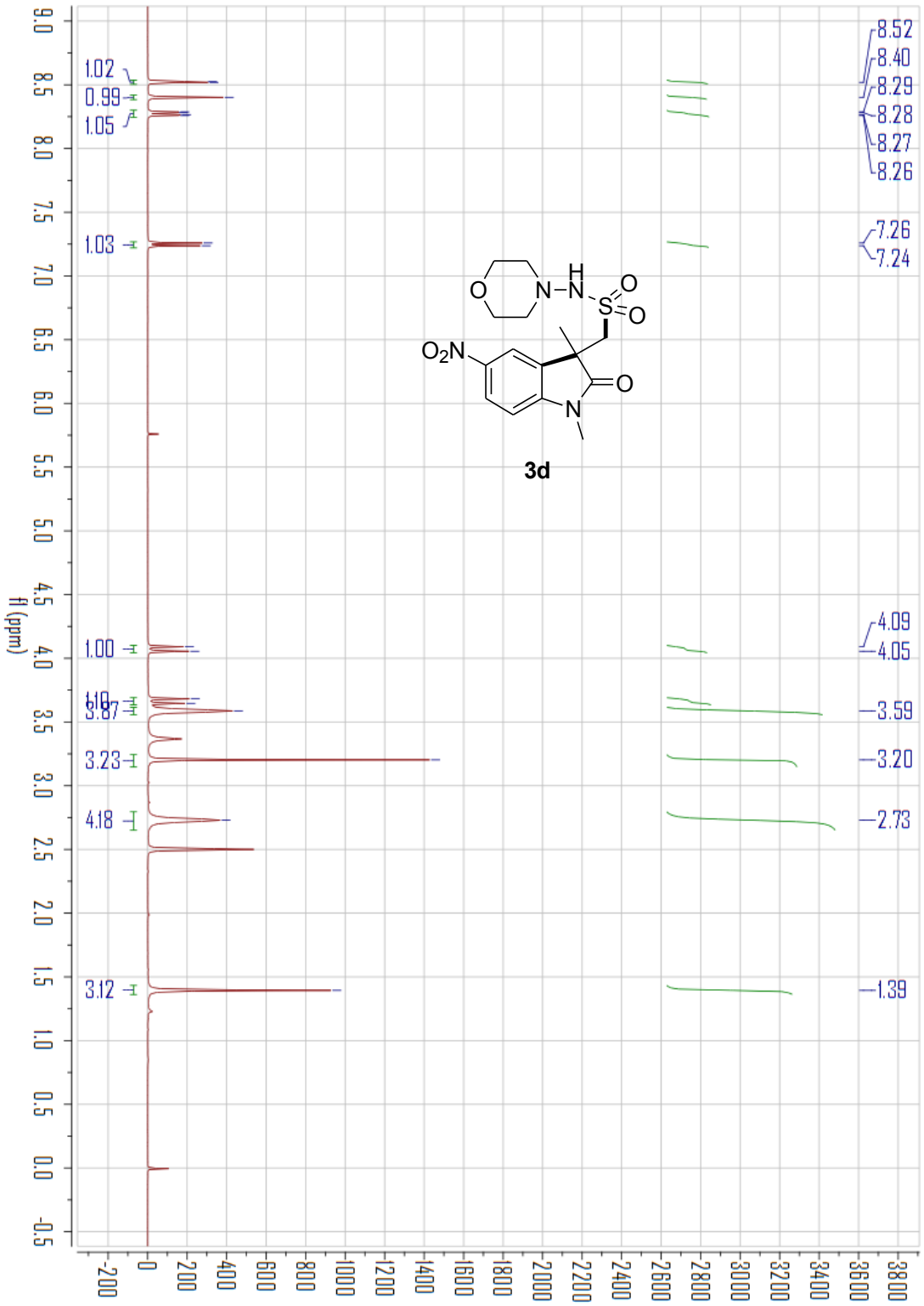
f1 (ppm)
3c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8.28
8.27
8.26
7.26
7.24
4.09
4.05
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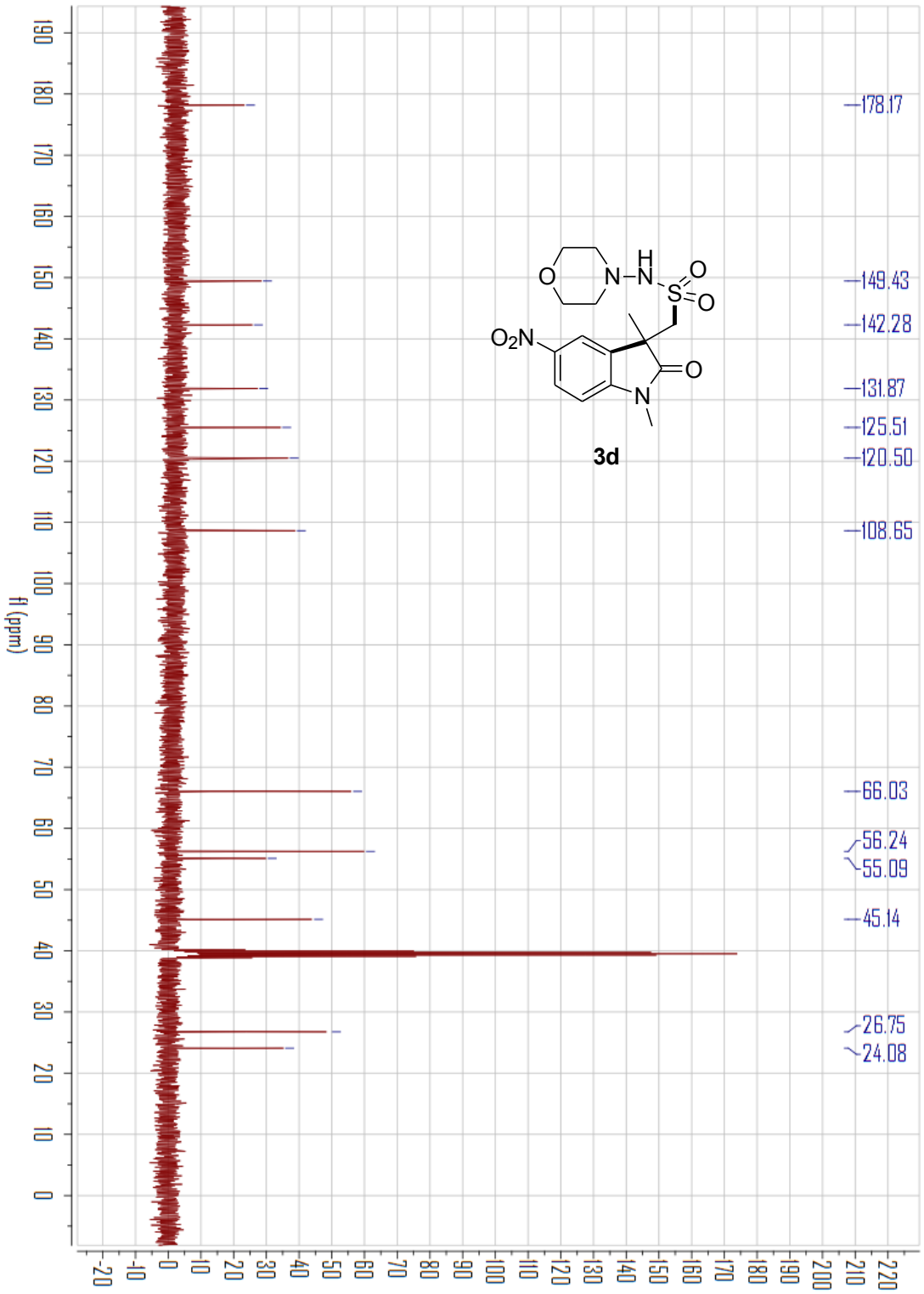
N
H
N
S
O
O
O2N
O
N
3d
f1 (ppm)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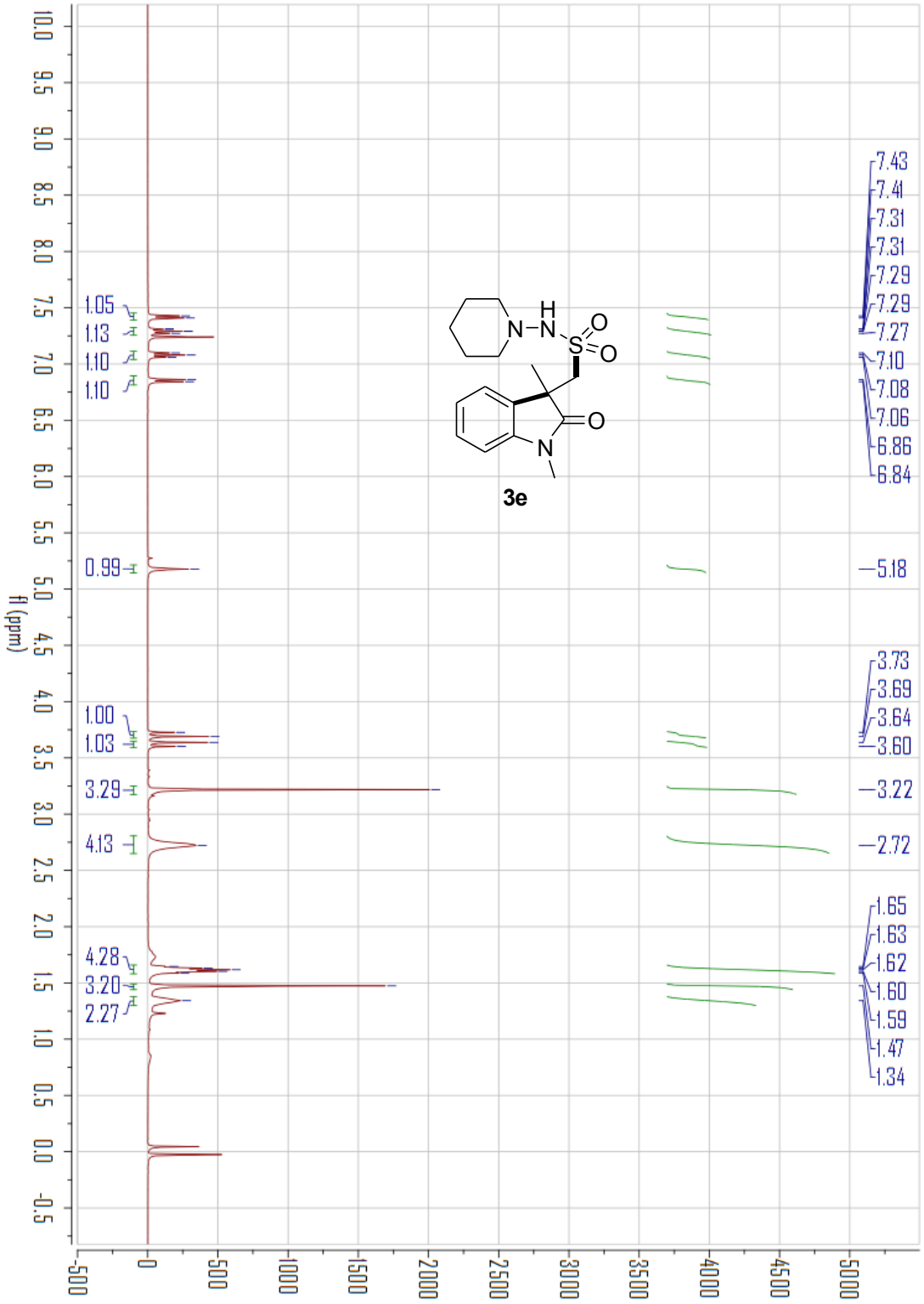
24.08
f1 (ppm)
3d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7.31
7.29
7.29
7.27
7.10
7.08
7.06
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6.84
5.18
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3.69
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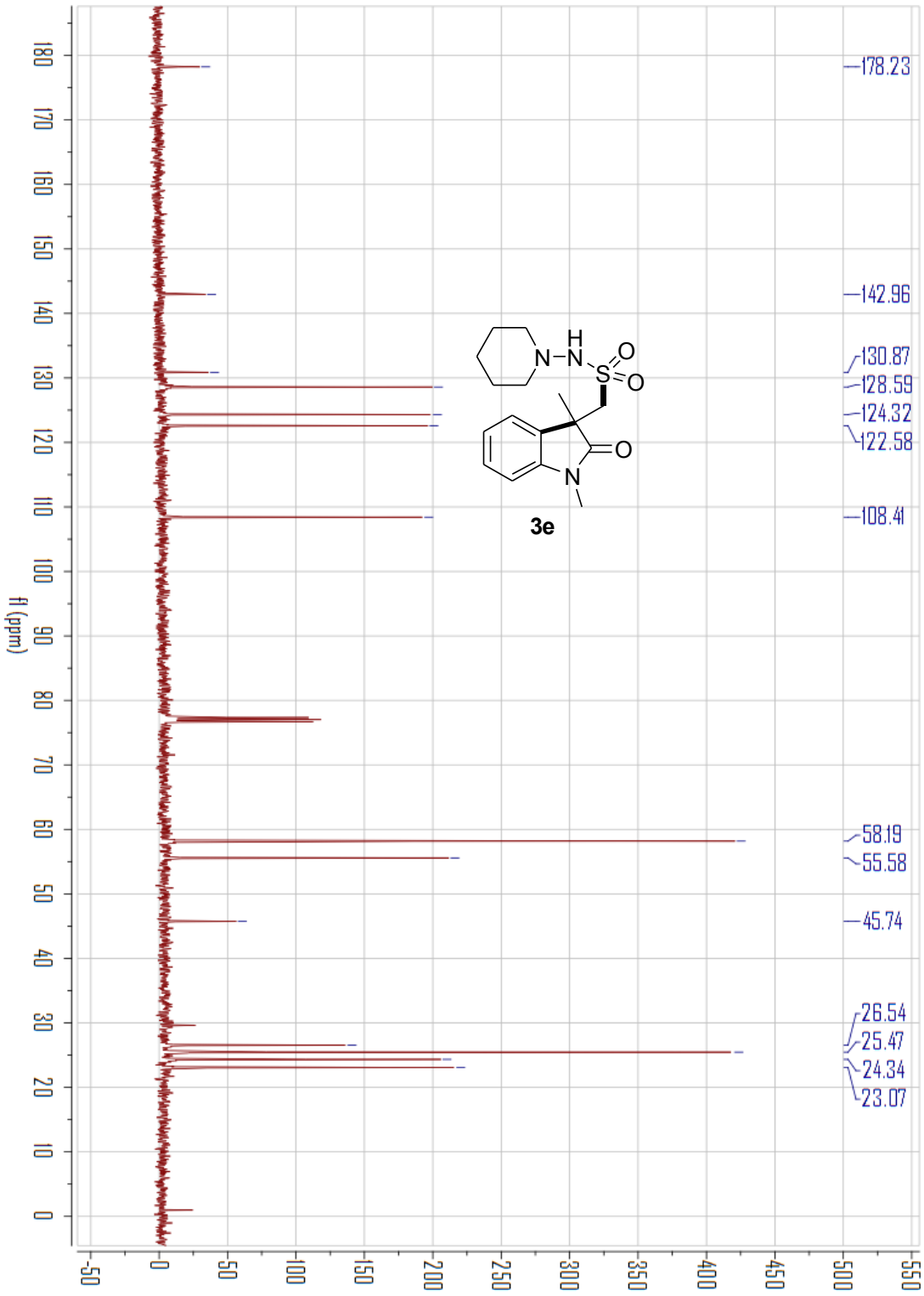
1.62
1.60
1.59
1.47
1.34
3e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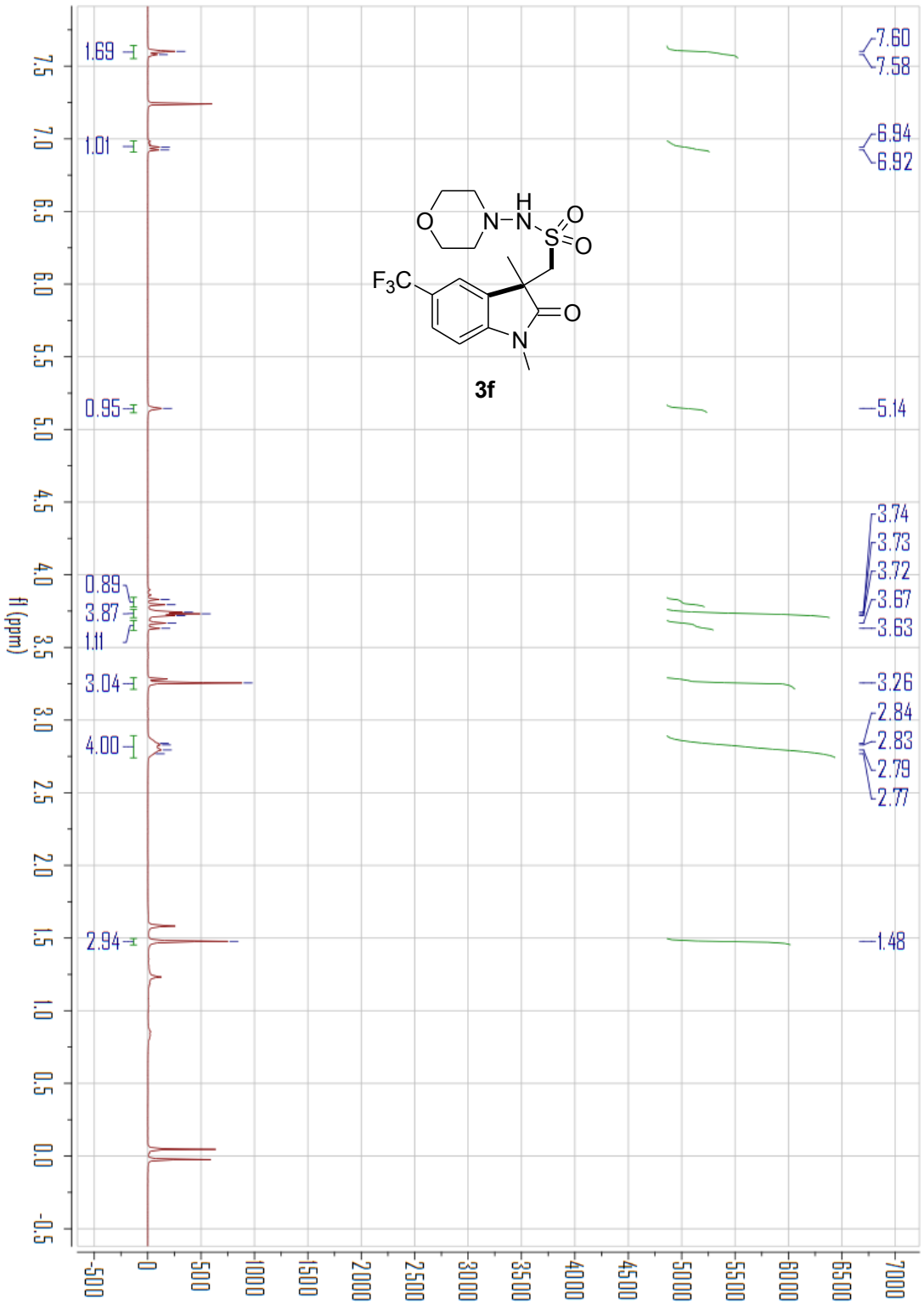
25.47
24.34
23.07
f1 (ppm)
3e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3.72
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2.77
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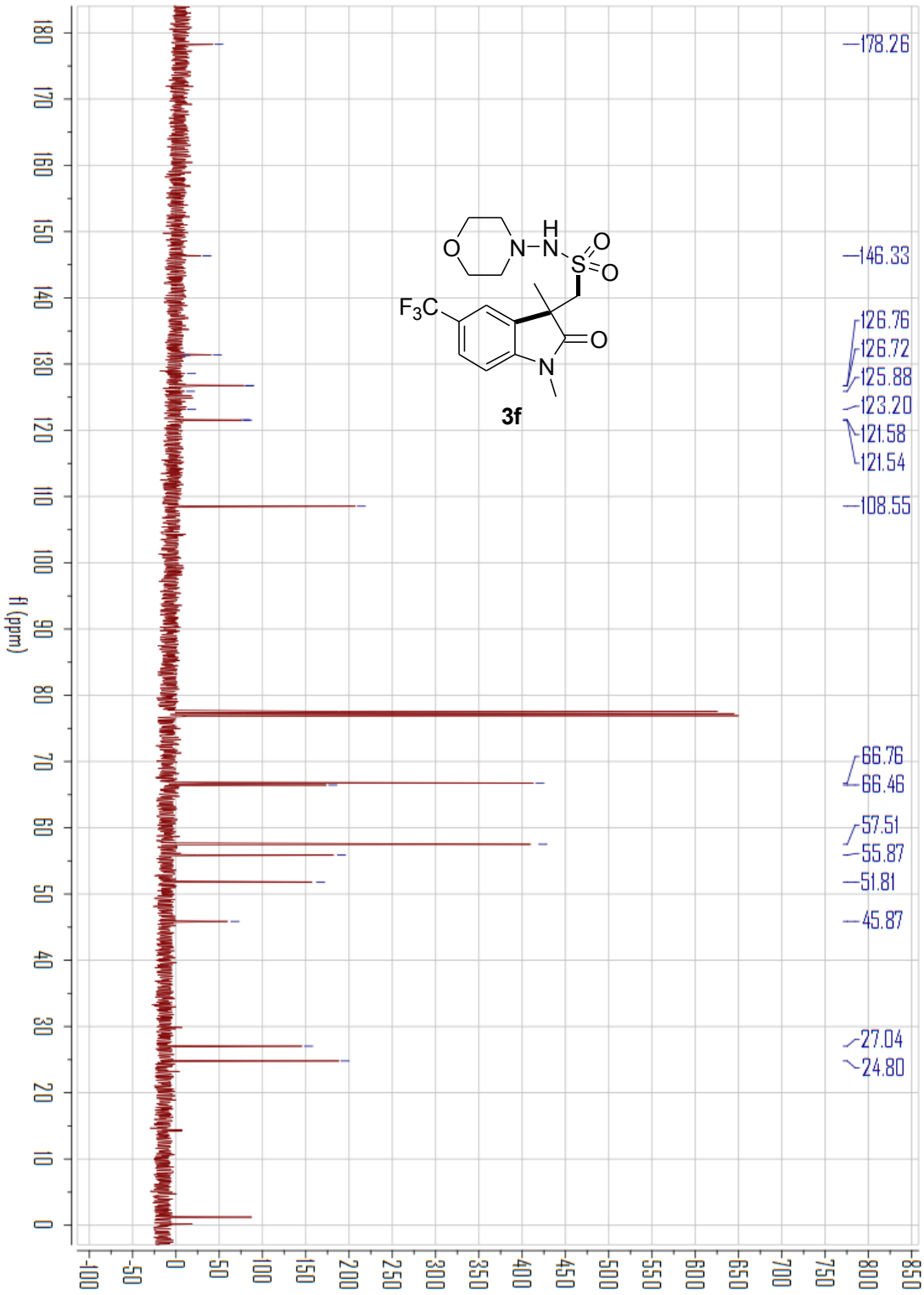
f1 (ppm)
3f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45.87
27.04
24.80
f1 (ppm)
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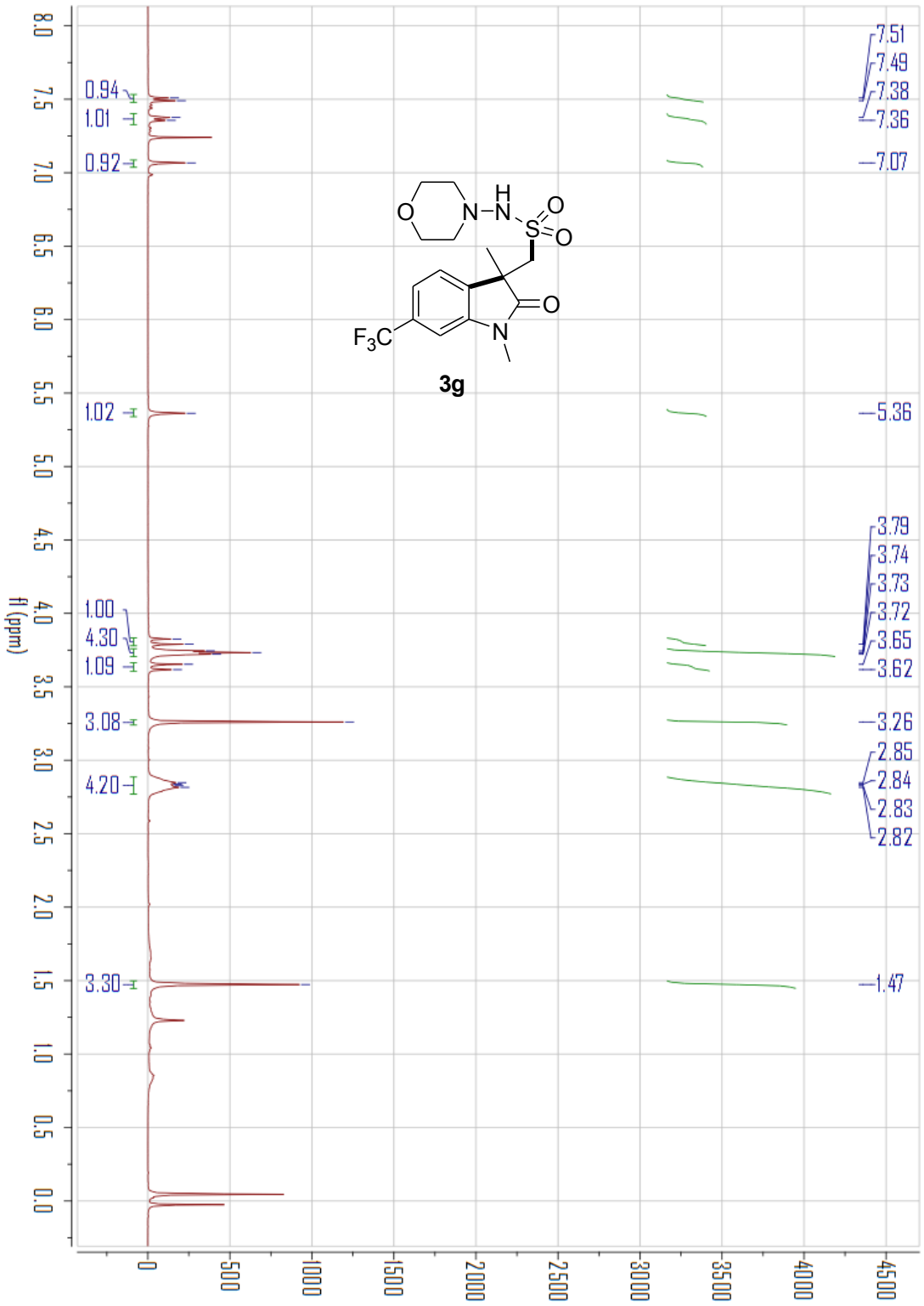
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2.85
2.84
2.83
2.82
1.47
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4.30
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3.08
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f1 (ppm)
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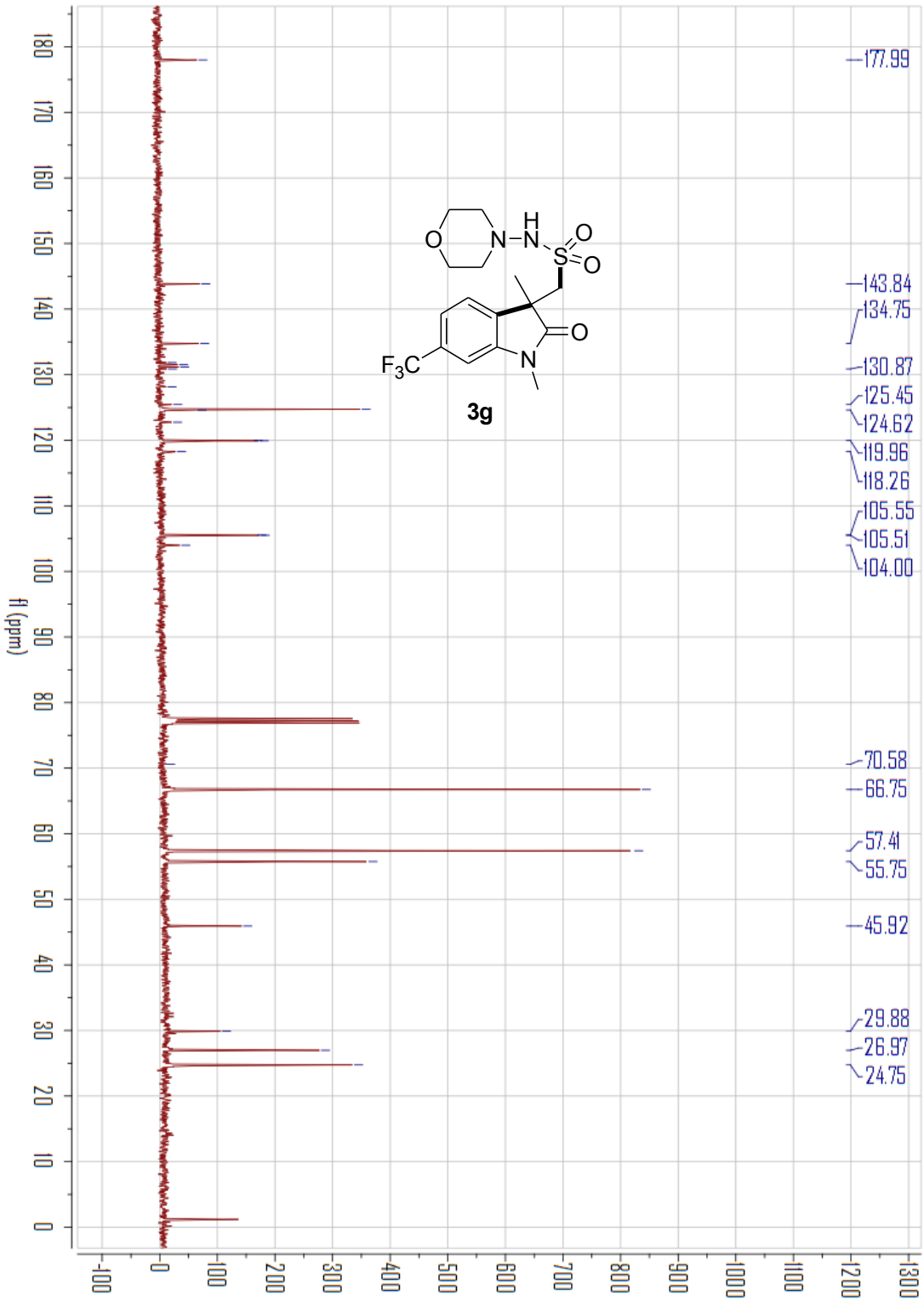
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f1 (ppm)
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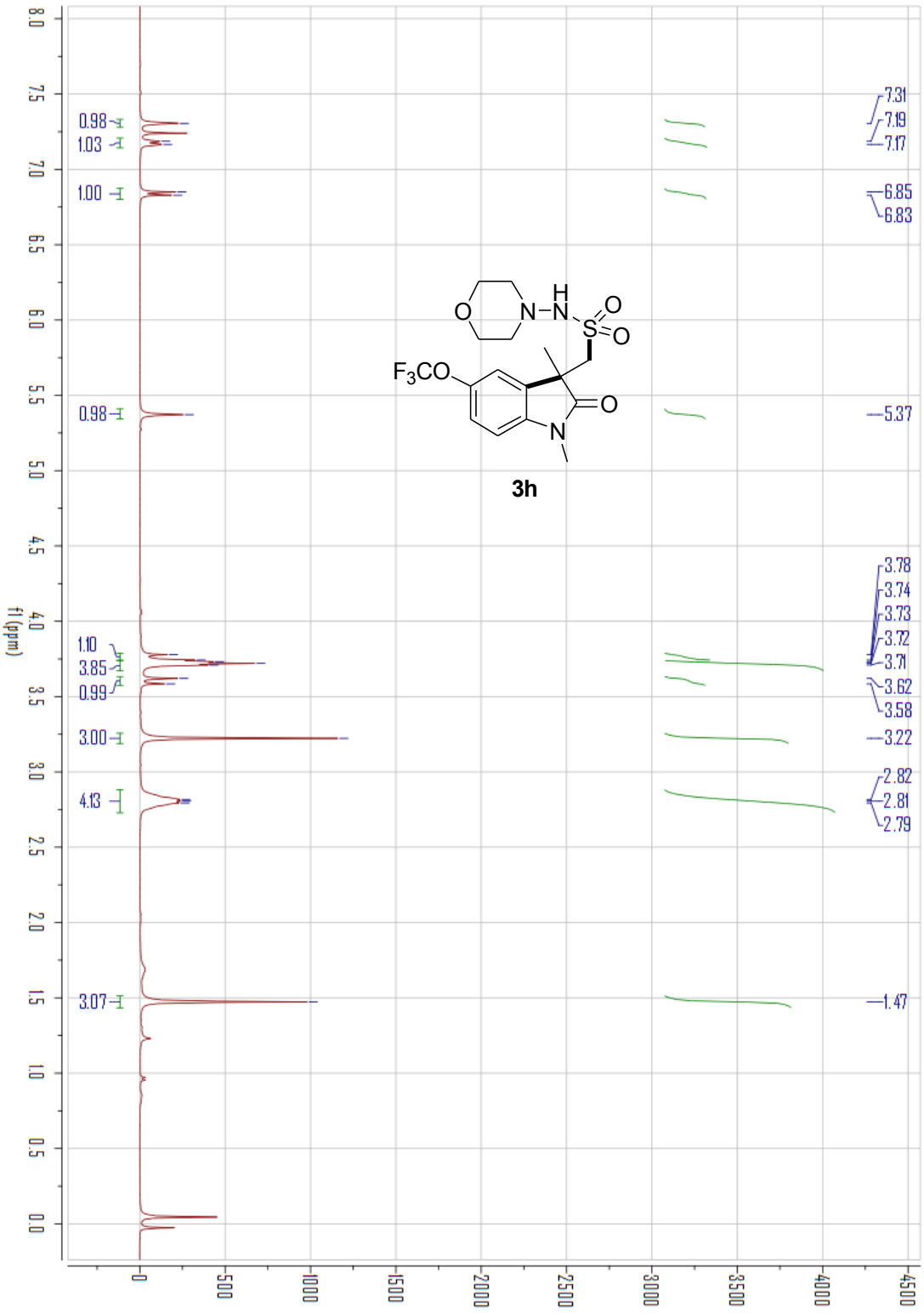
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6.83
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3.73
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2.79
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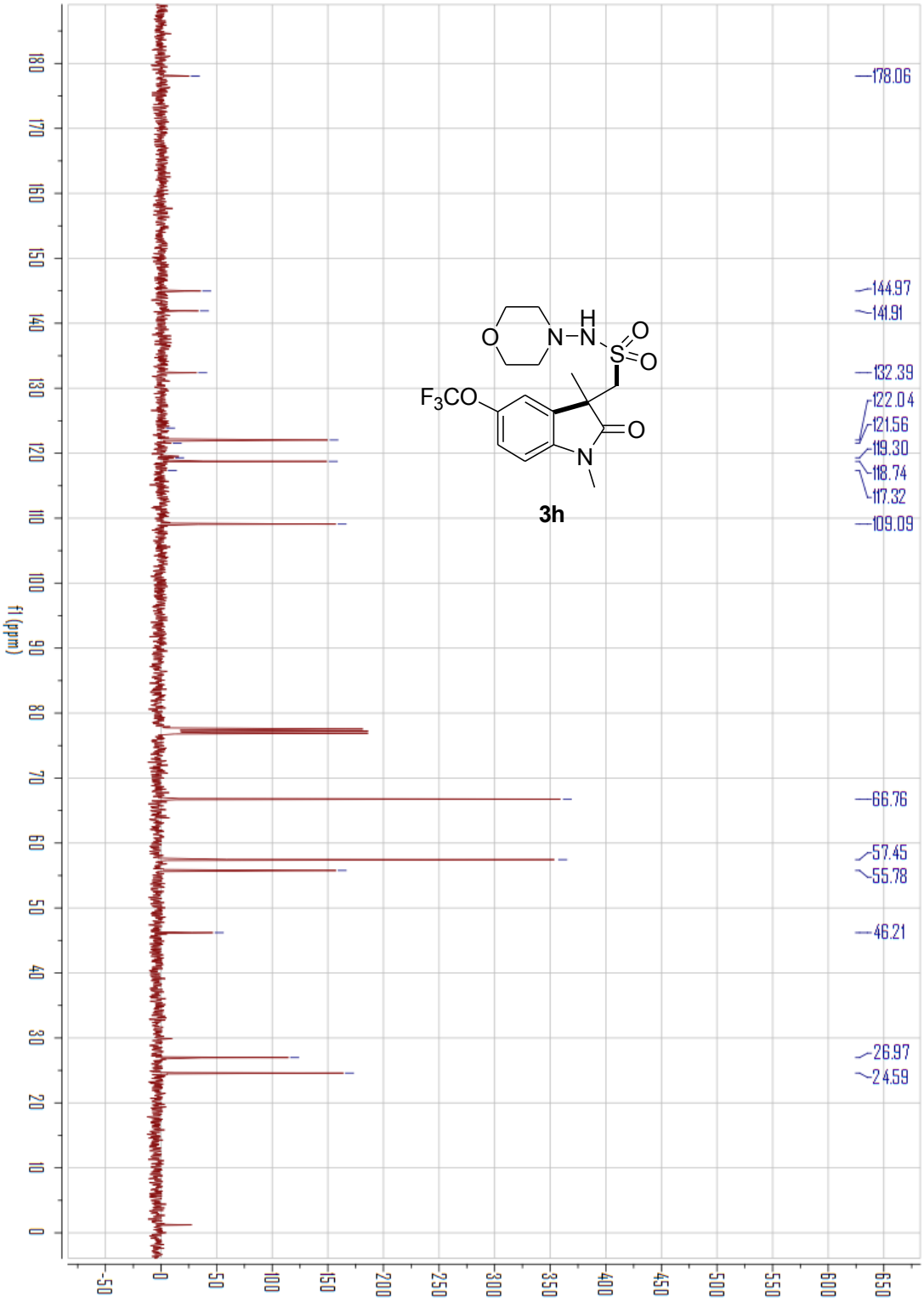
f1 (ppm)
F3CO
3h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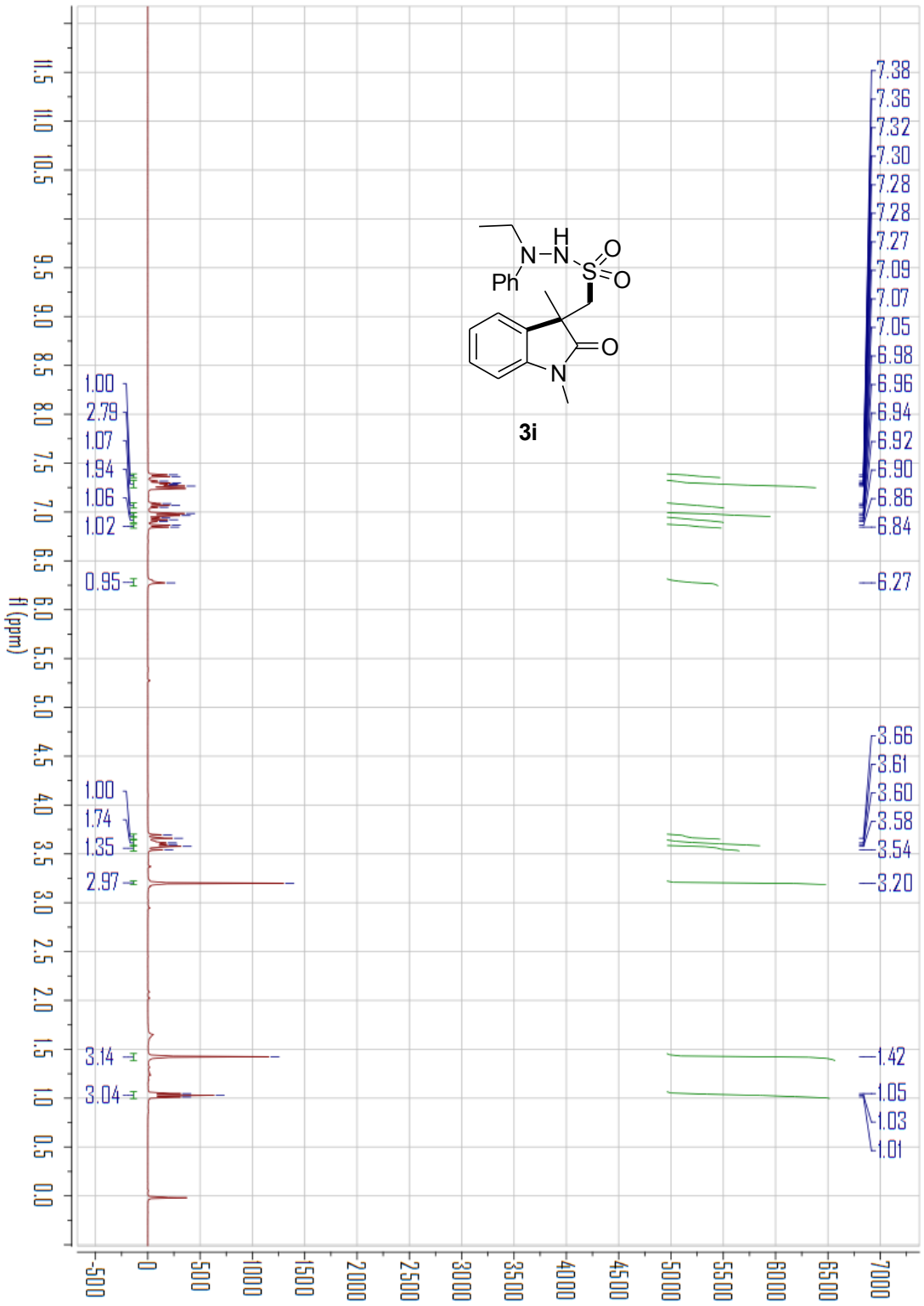
f1 (ppm)
3h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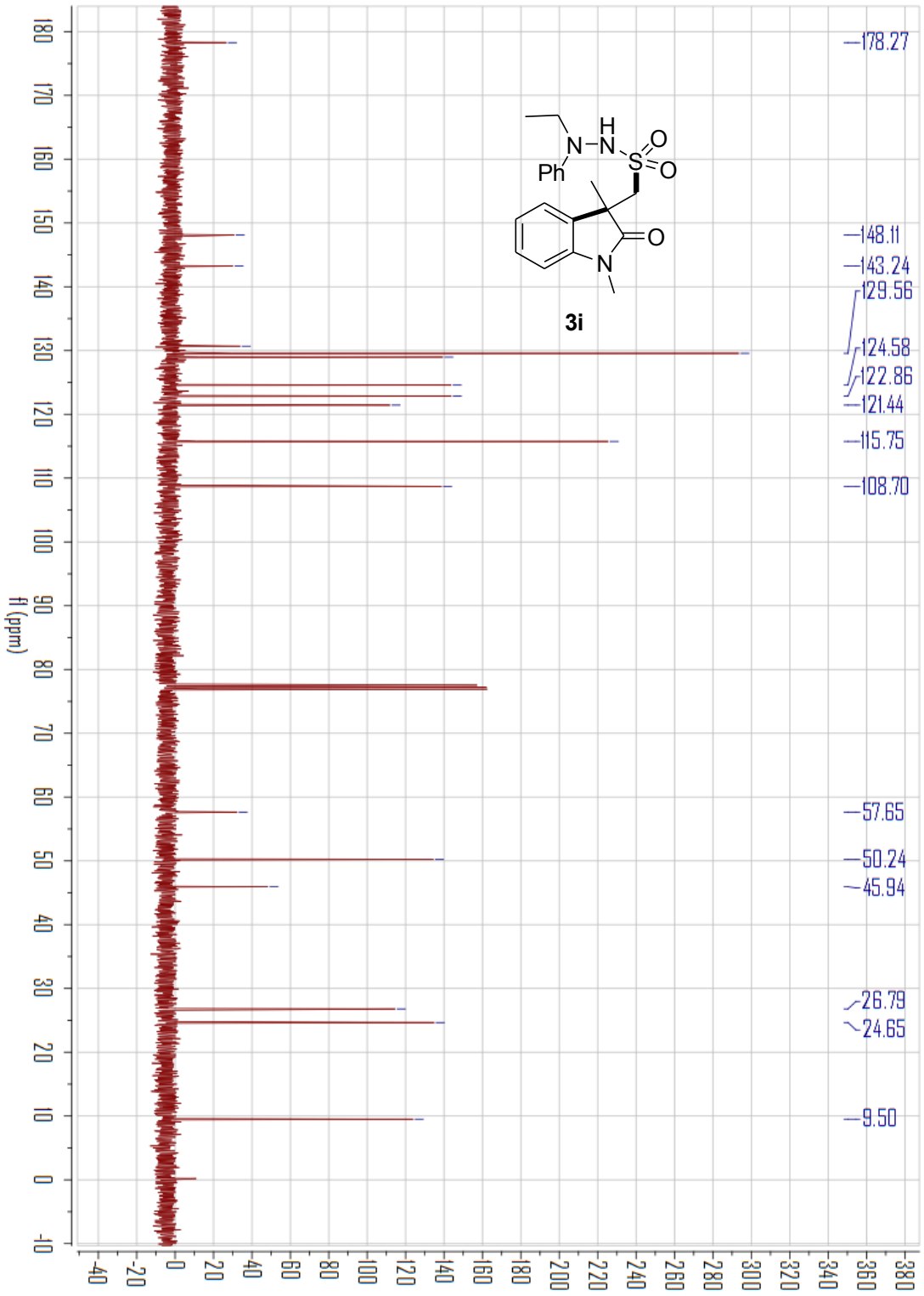
f1 (ppm)
3i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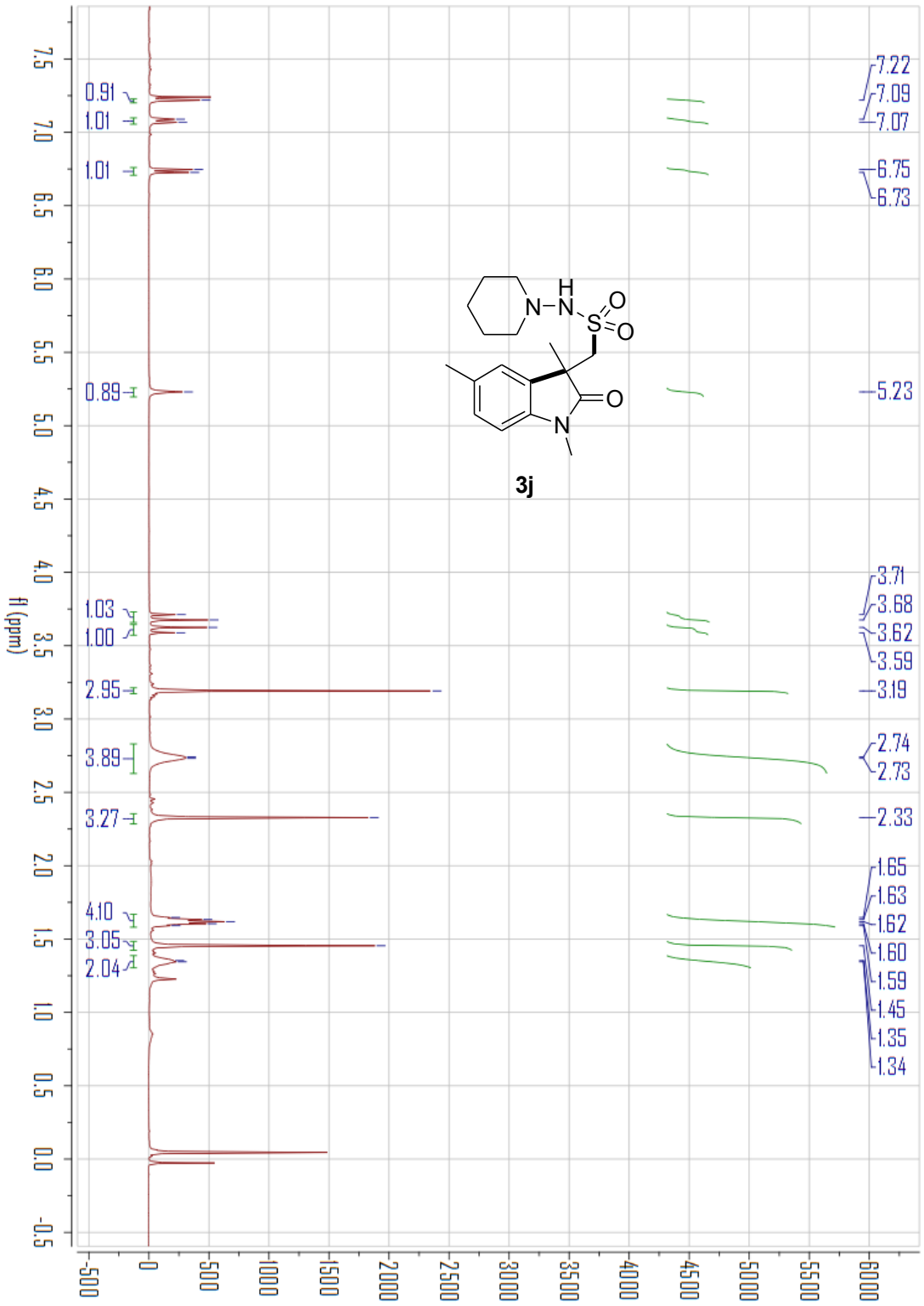
3i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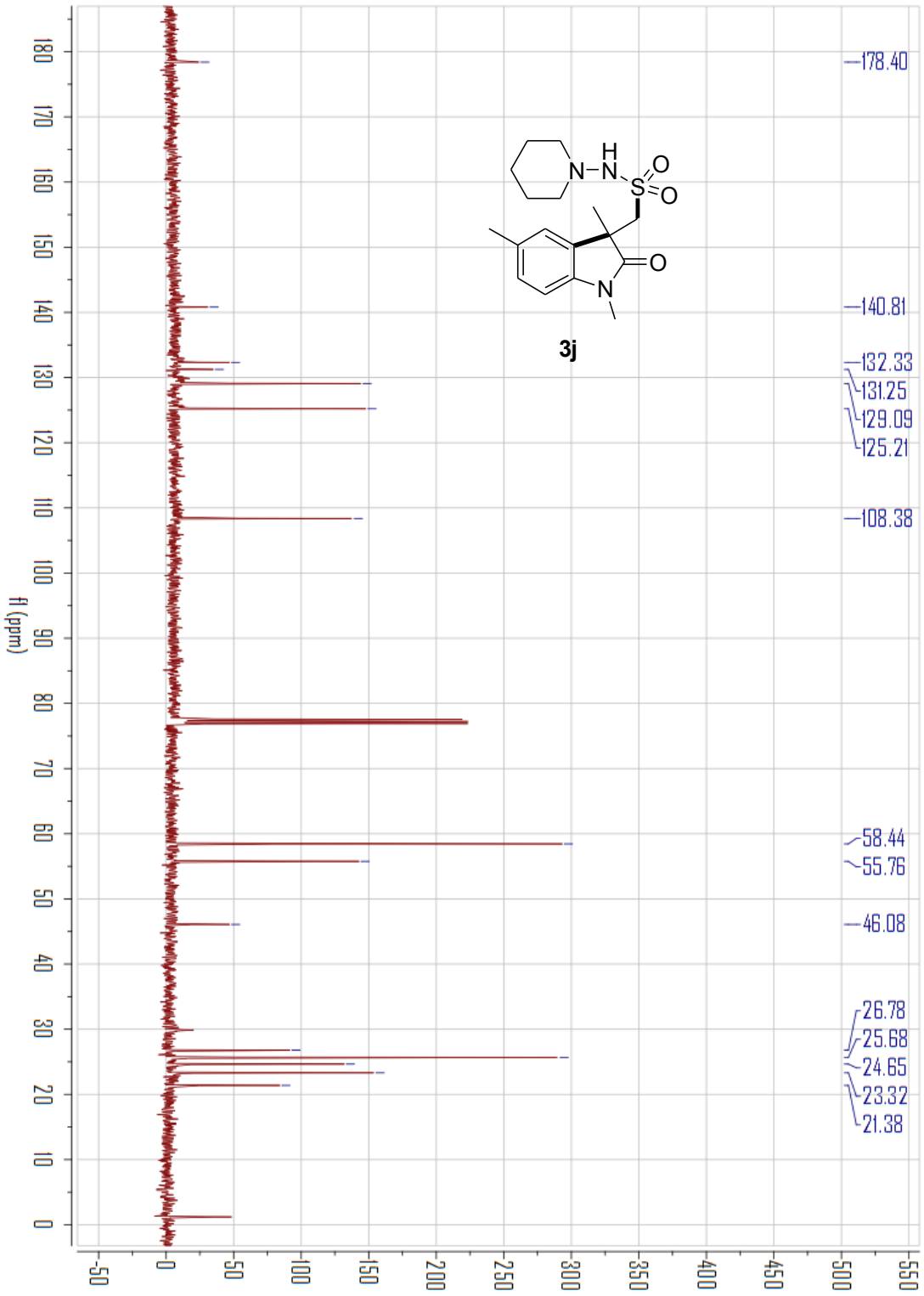
1.45
1.35
1.34
f1 (ppm)
3j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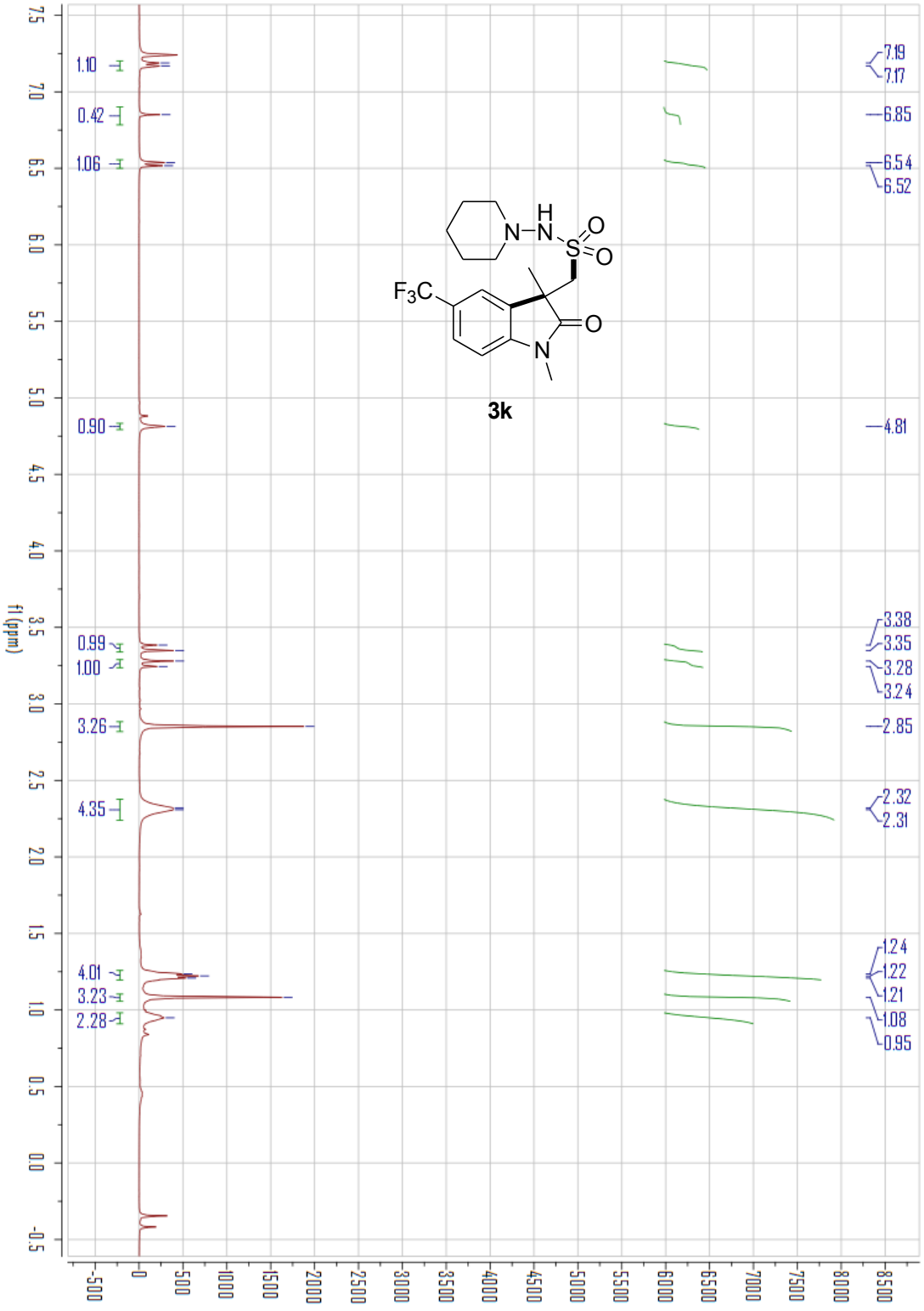
23.32
21.38
f1 (ppm)
3j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3.35
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1.21
1.08
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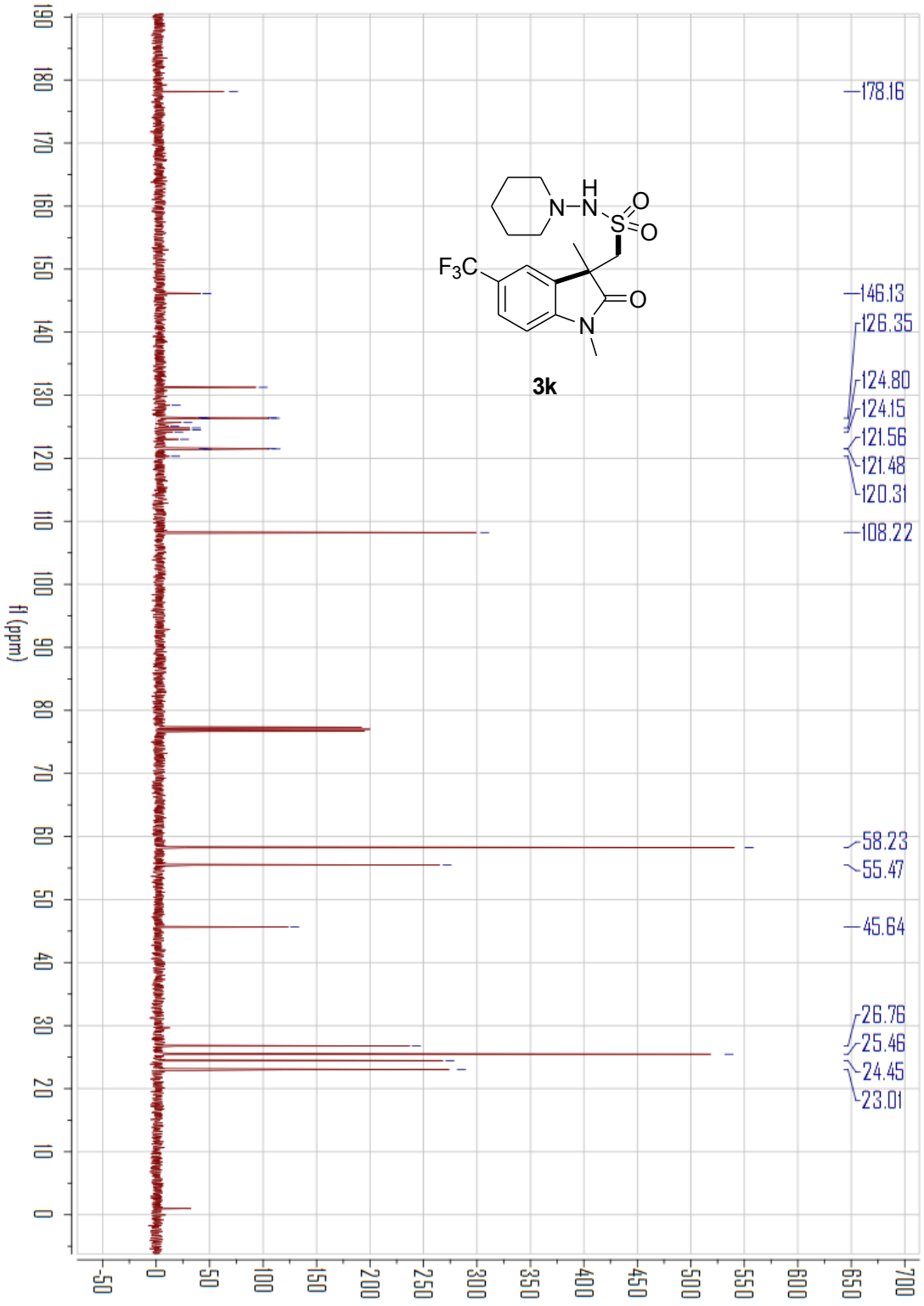
f1 (ppm)

3k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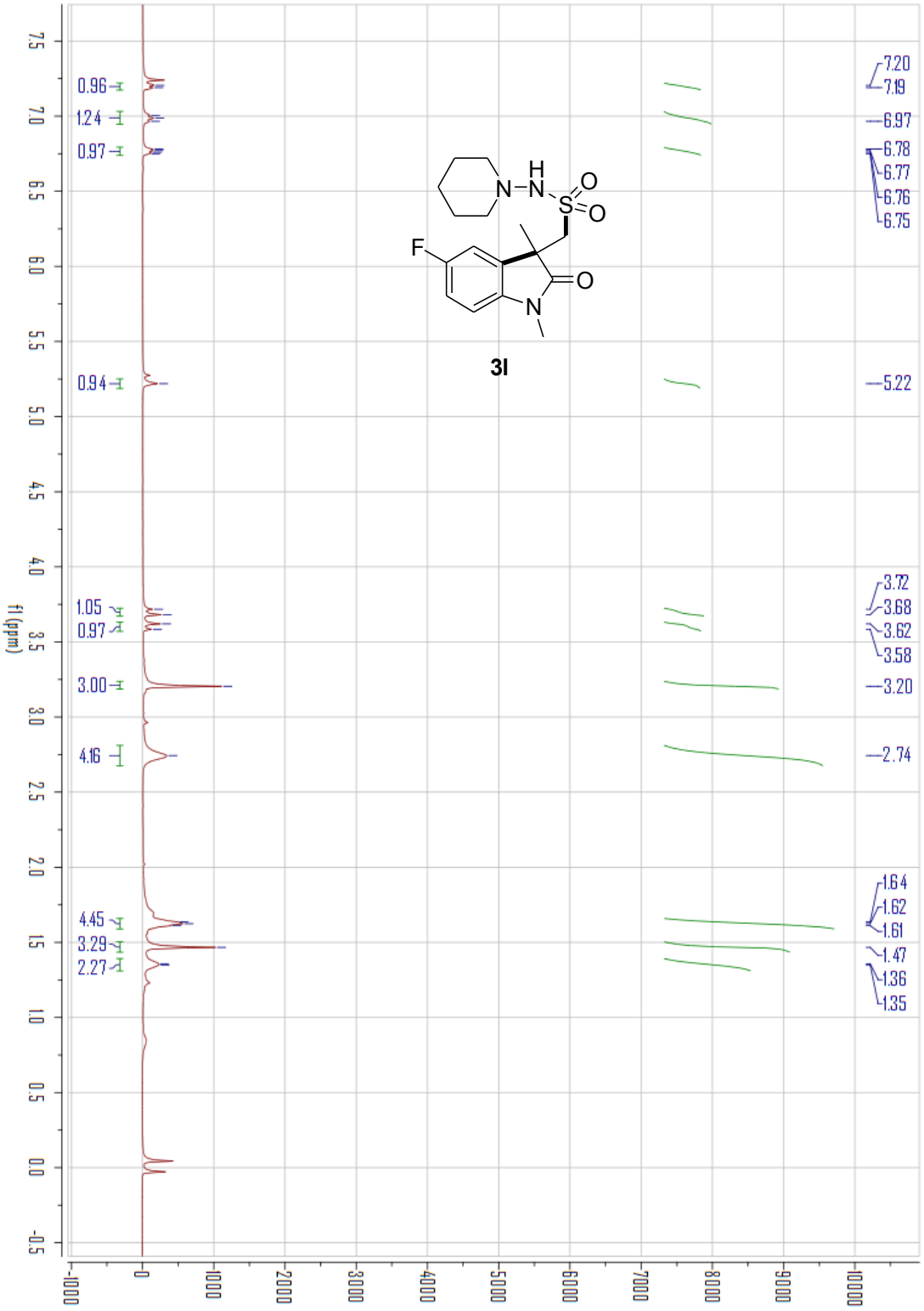
3k
F3C
N
H
N
S
O
O
O
N
f1 (ppm)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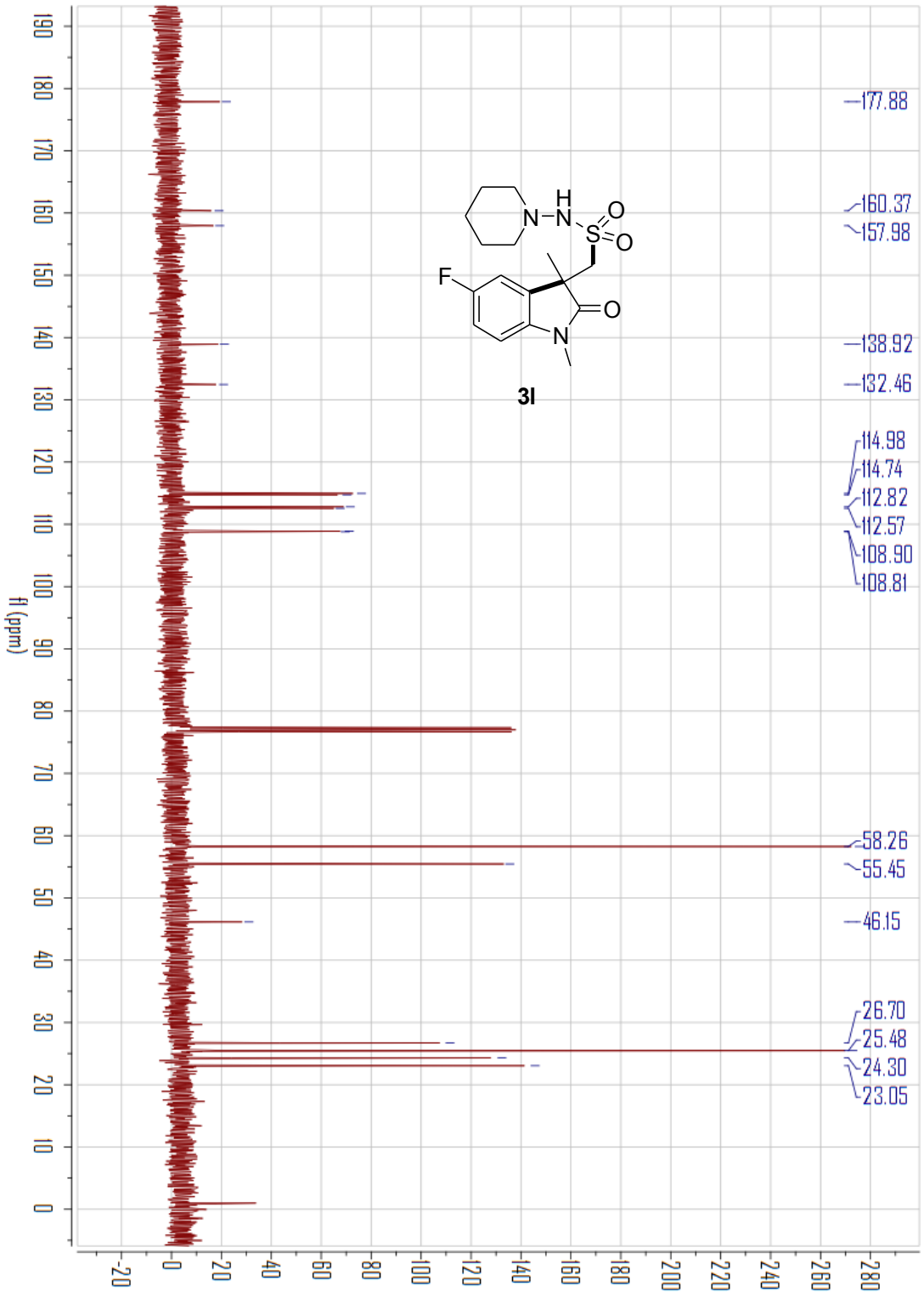
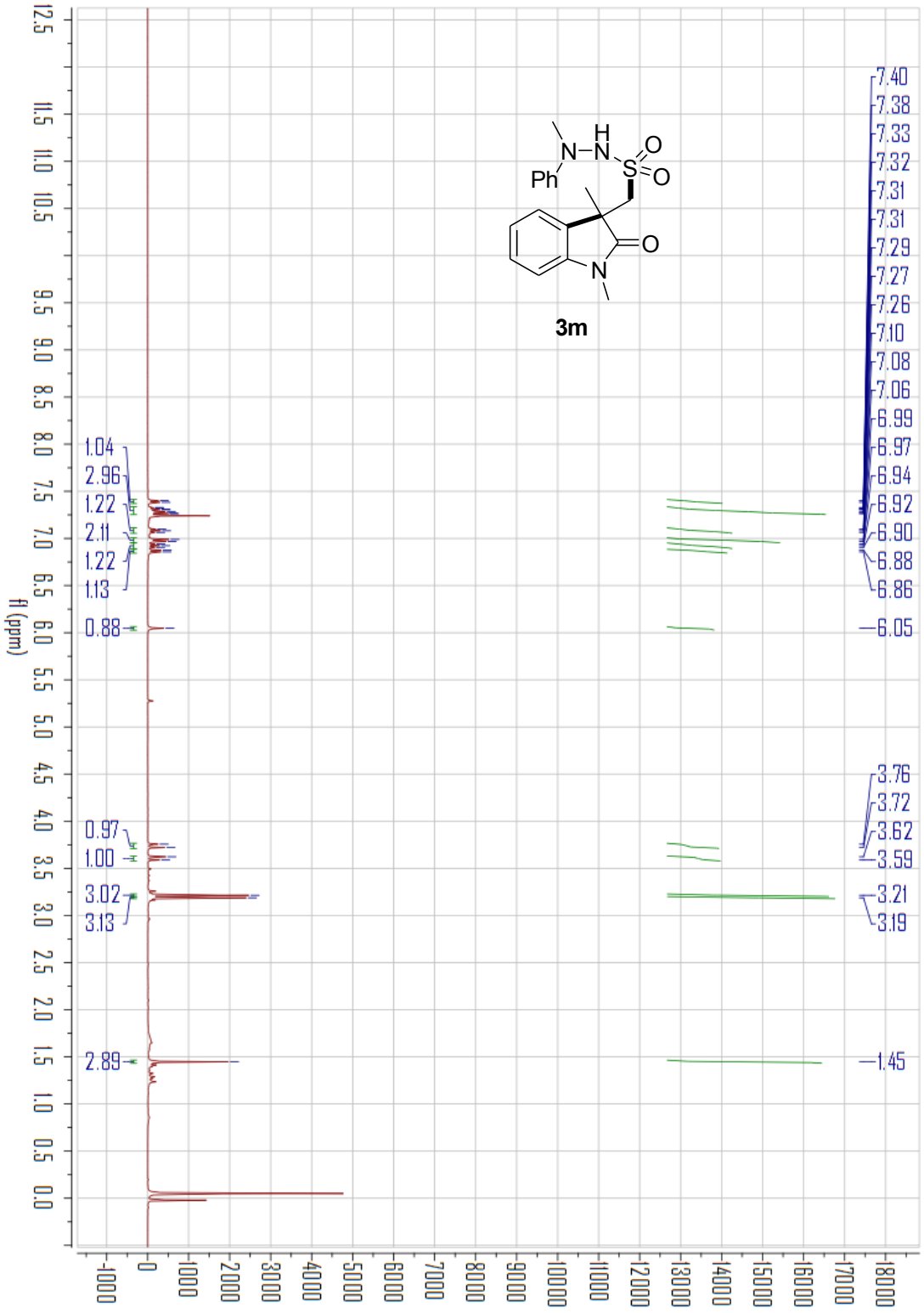
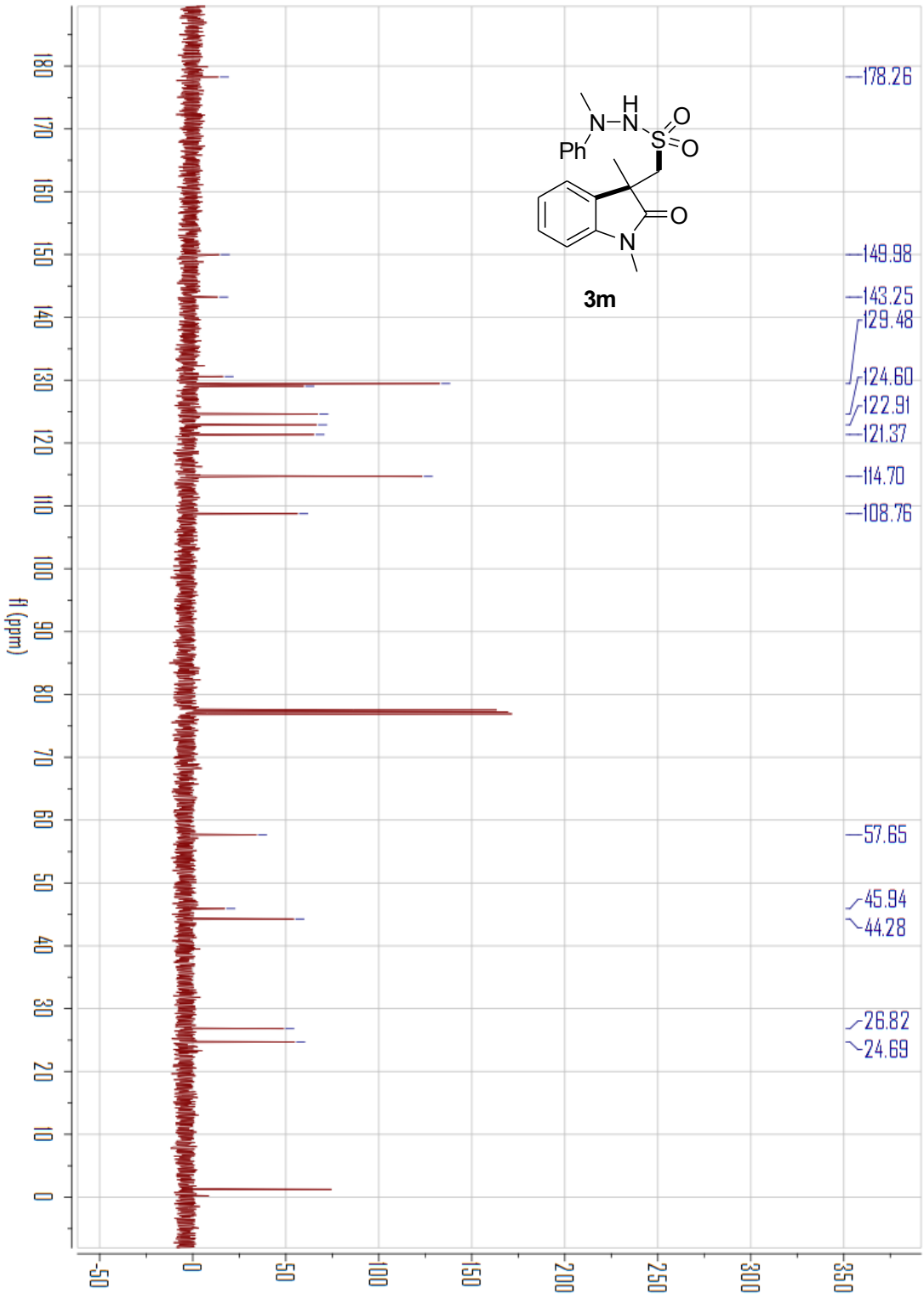
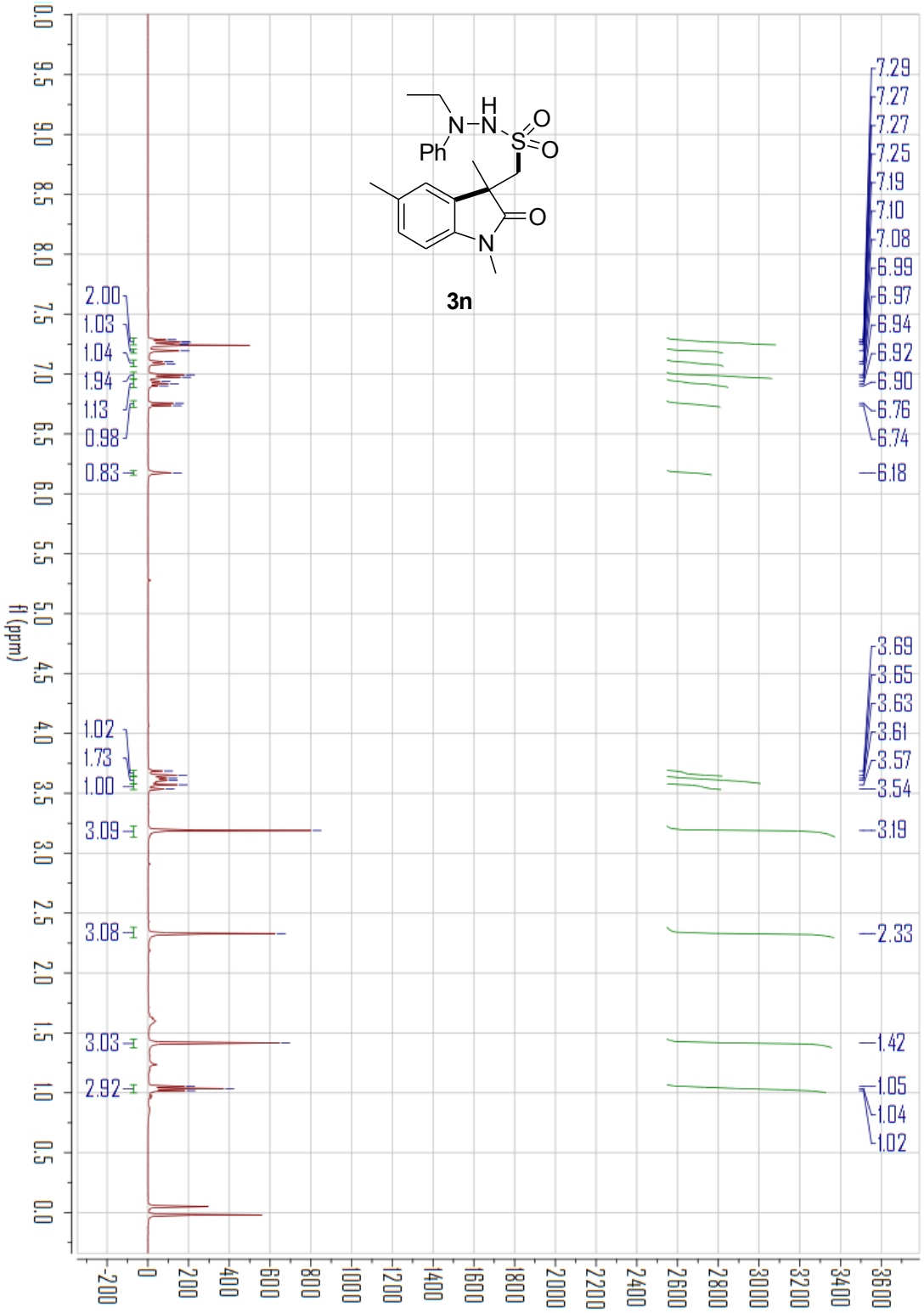
1.36
1.35
f1 (ppm)

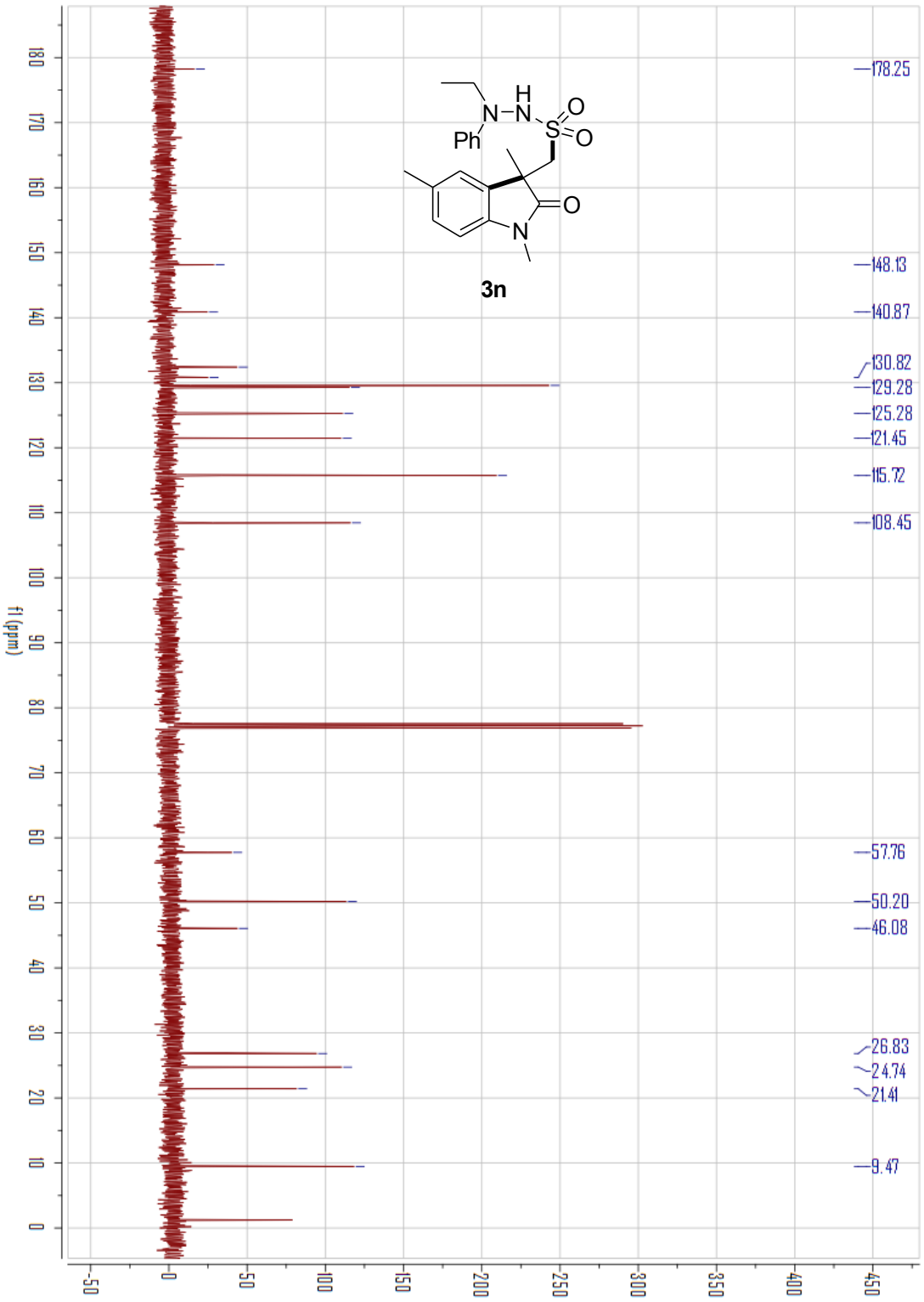
3l

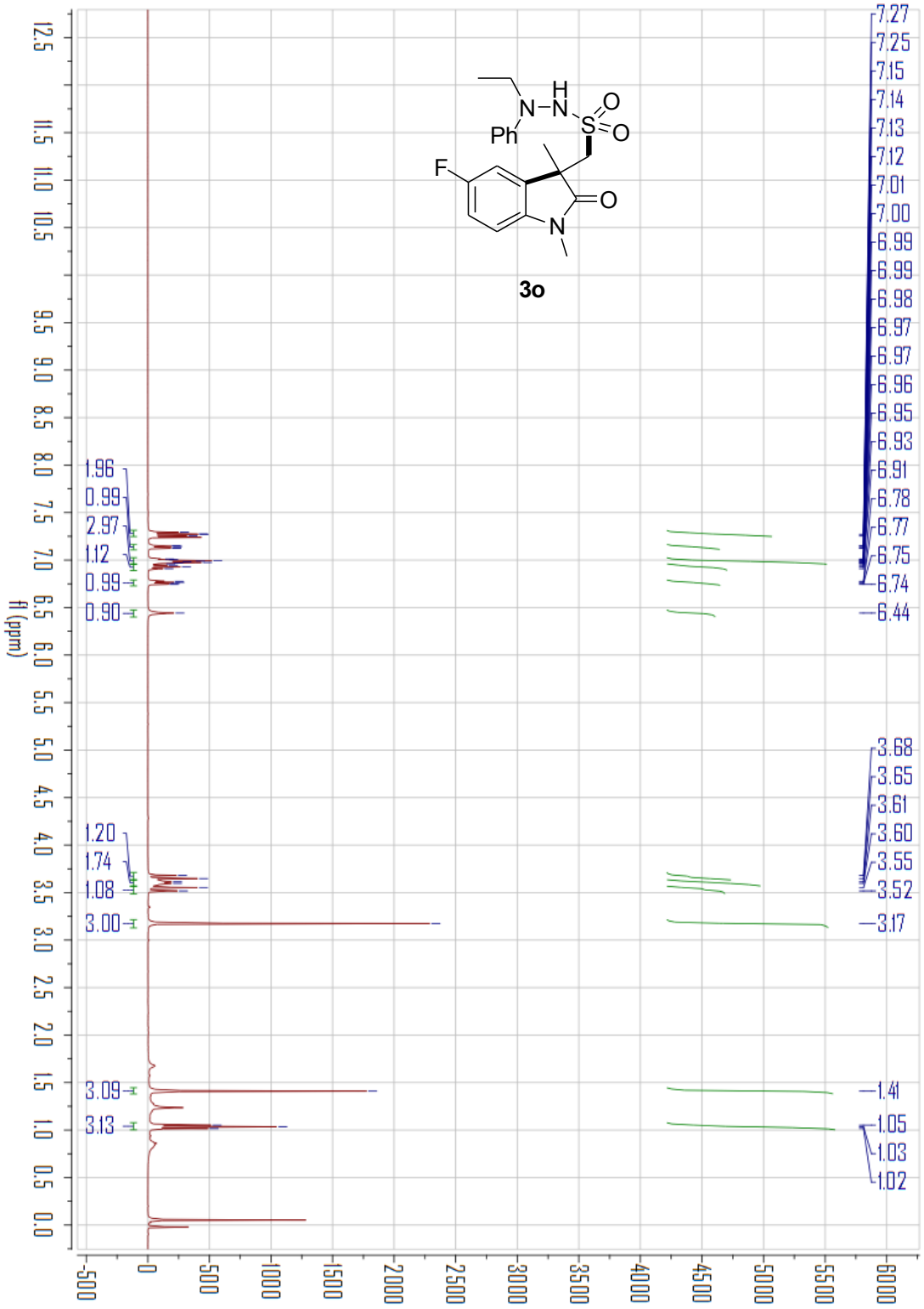

177.88
160.37
157.98
138.92
132.46
114.98
114.74
112.82
112.57
108.90
108.81
58.26
55.45
46.15
26.70
25.48
24.30
23.05
f1 (ppm)
3l

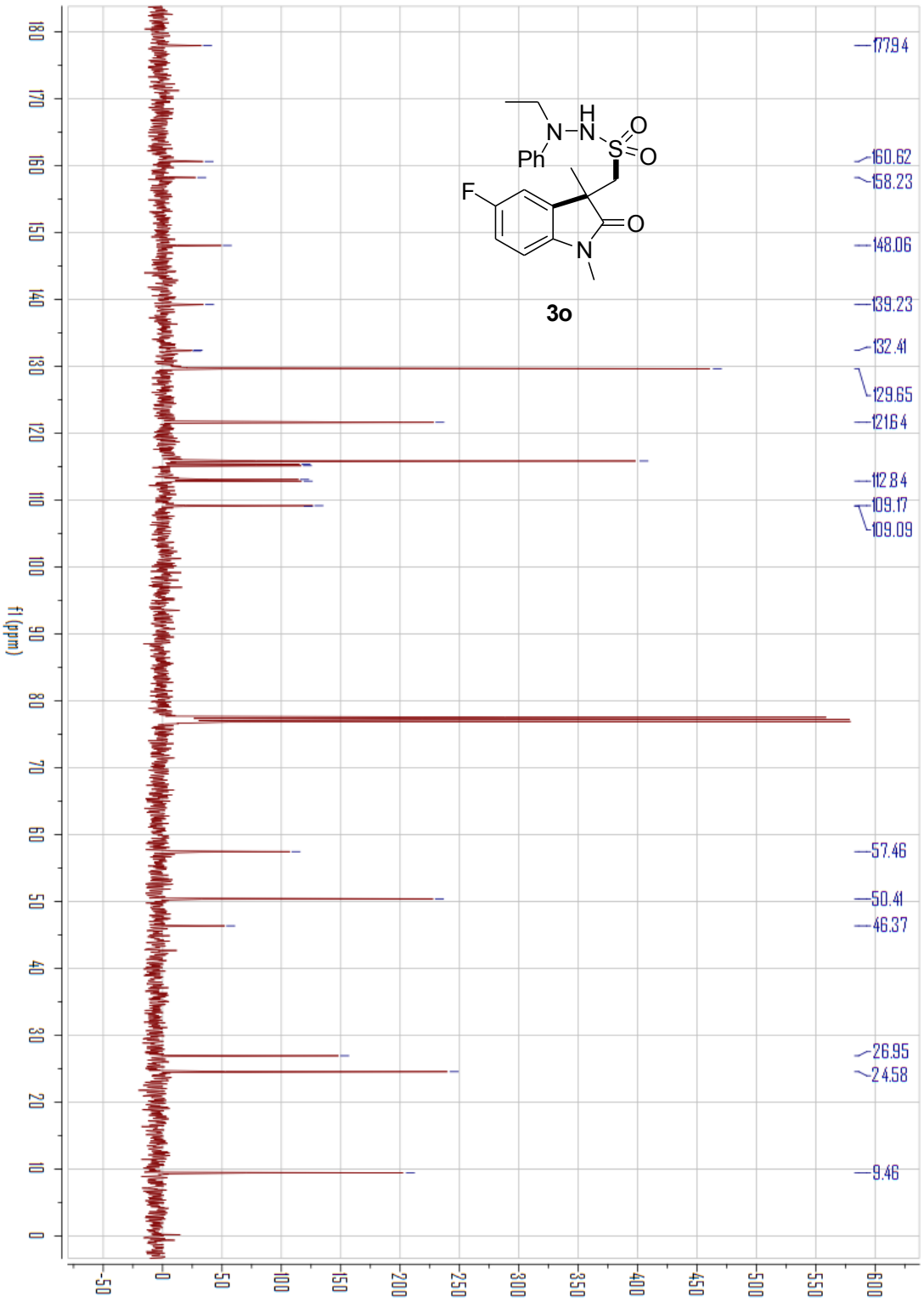

3m
f1 (ppm)
7.40
7.38
7.33
7.32
7.31
7.31
7.29
7.27
7.26
7.10
7.08
7.06
6.99
6.97
6.94
6.92
6.90
6.88
6.86
6.05
3.76
3.72
3.62
3.59
3.21
3.19
1.45
1.04
2.96
1.22
2.11
1.22
1.13
0.88
0.97
1.00
3.02
3.13
2.89

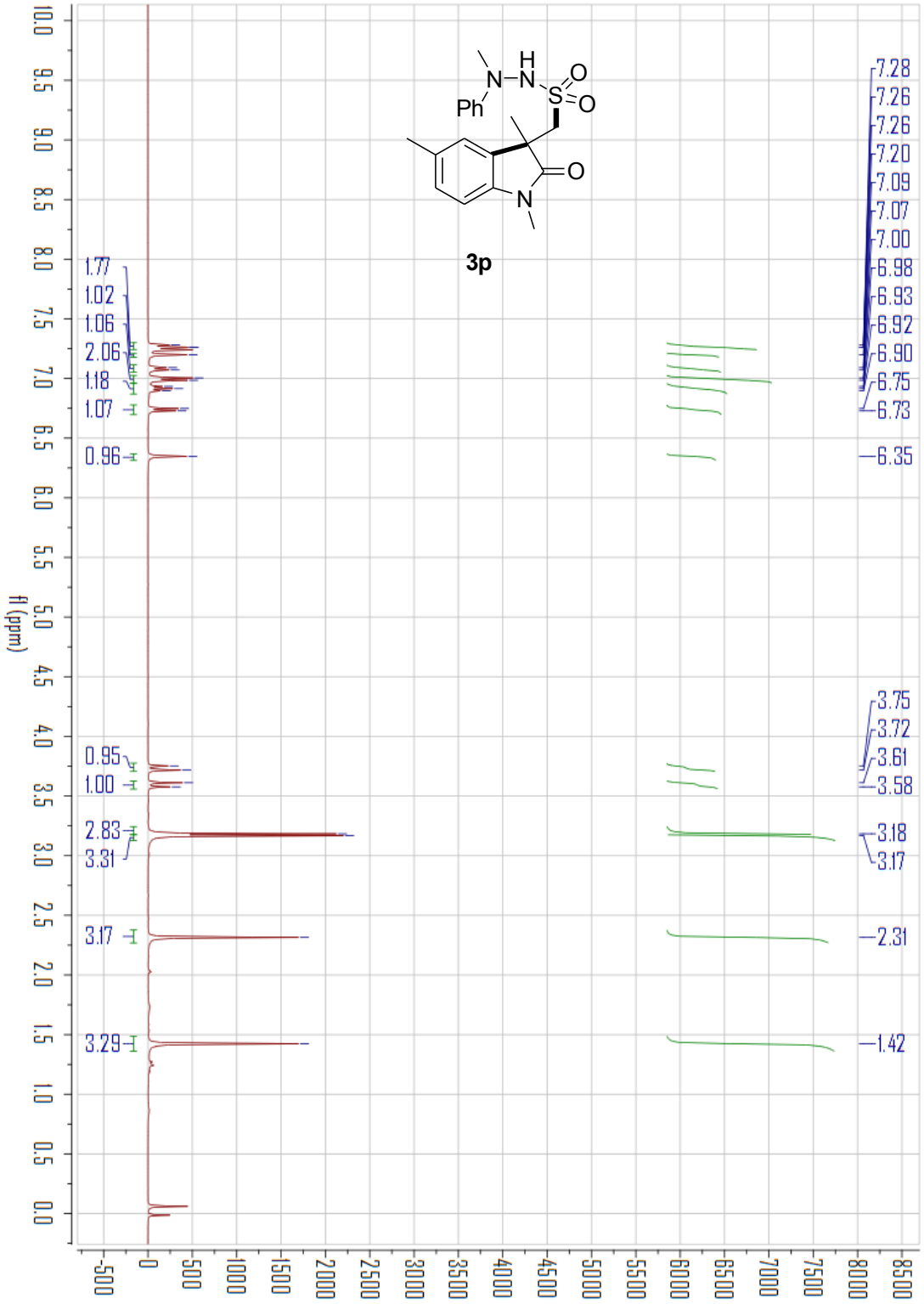

178.26
149.98
143.25
129.48
124.60
122.91
121.37
114.70
108.76
57.65
45.94
44.28
26.82
24.69
f1 (ppm)
3m

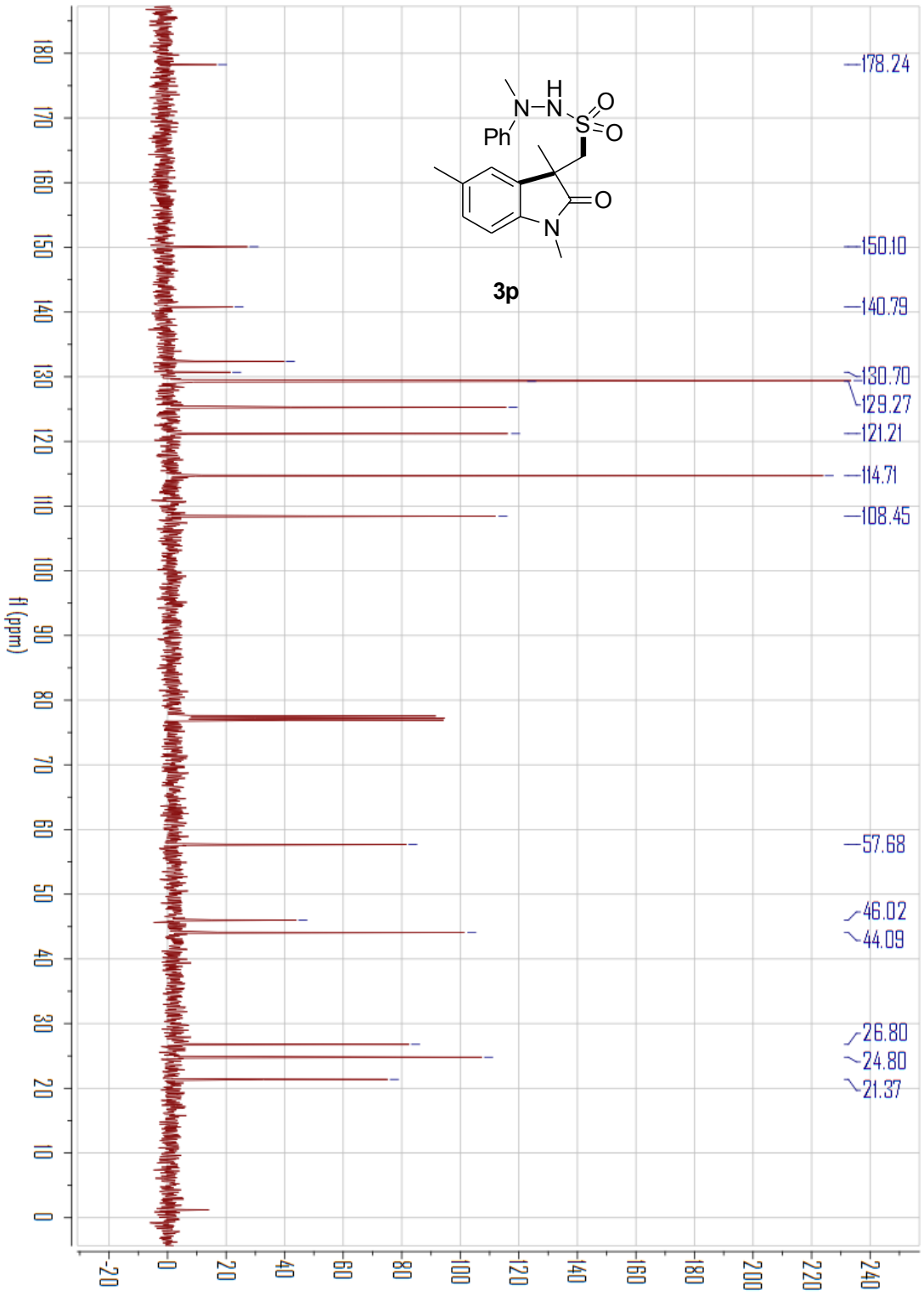

3n
f1 (ppm)
7.29
7.27
7.27
7.25
7.19
7.10
7.08
6.99
6.97
6.94
6.92
6.90
6.76
6.74
6.18
3.69
3.65
3.63
3.61
3.57
3.54
3.19
2.33
1.42
1.05
1.04
1.02
2.00
1.03
1.04
1.94
1.13
0.98
0.83
1.02
1.73
1.00
3.09
3.08
3.03
2.92

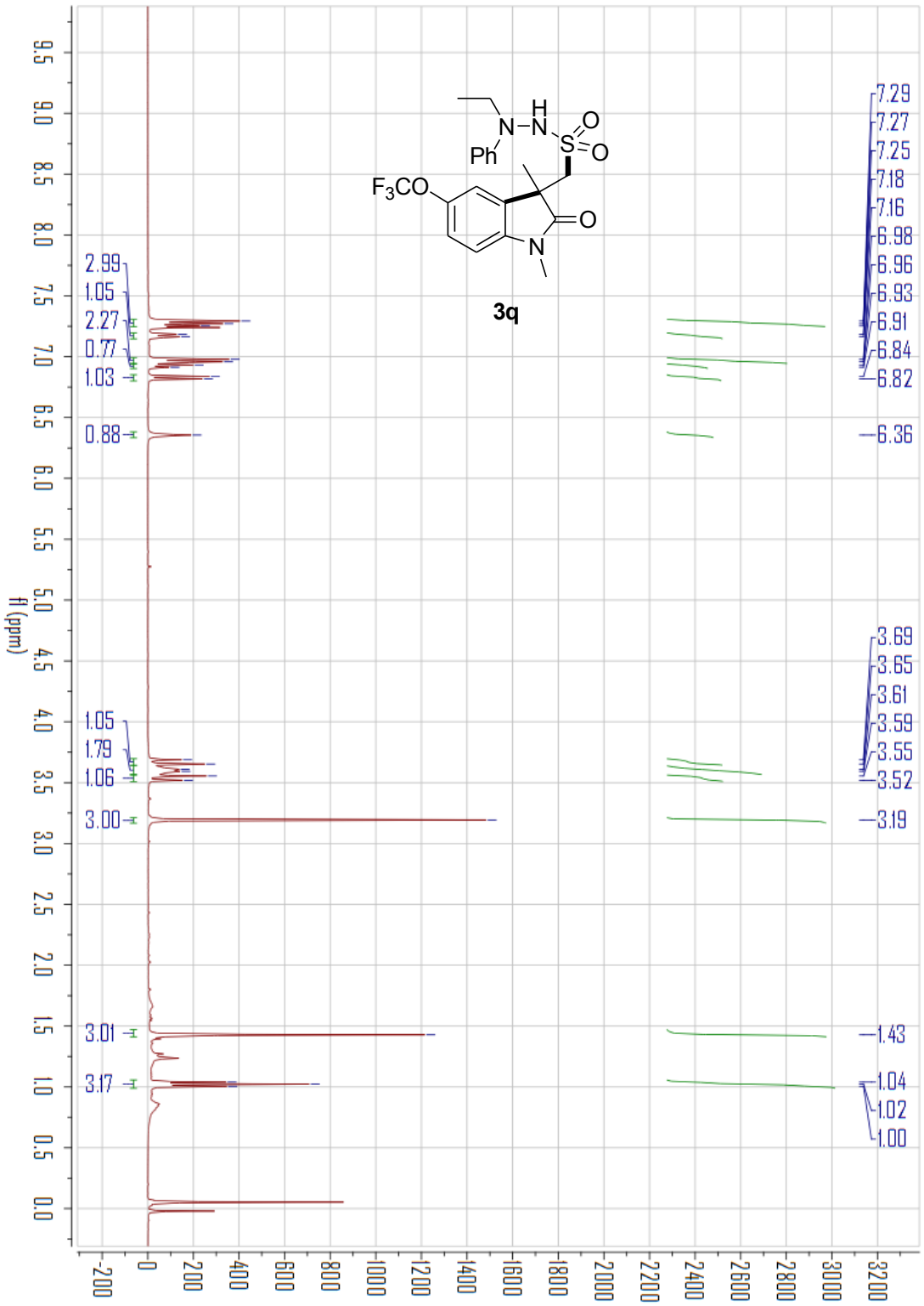

178.25
148.13
140.87
130.82
129.28
125.28
121.45
115.72
108.45
57.76
50.20
46.08
26.83
24.74
21.41
9.47
f1 (ppm)
3n

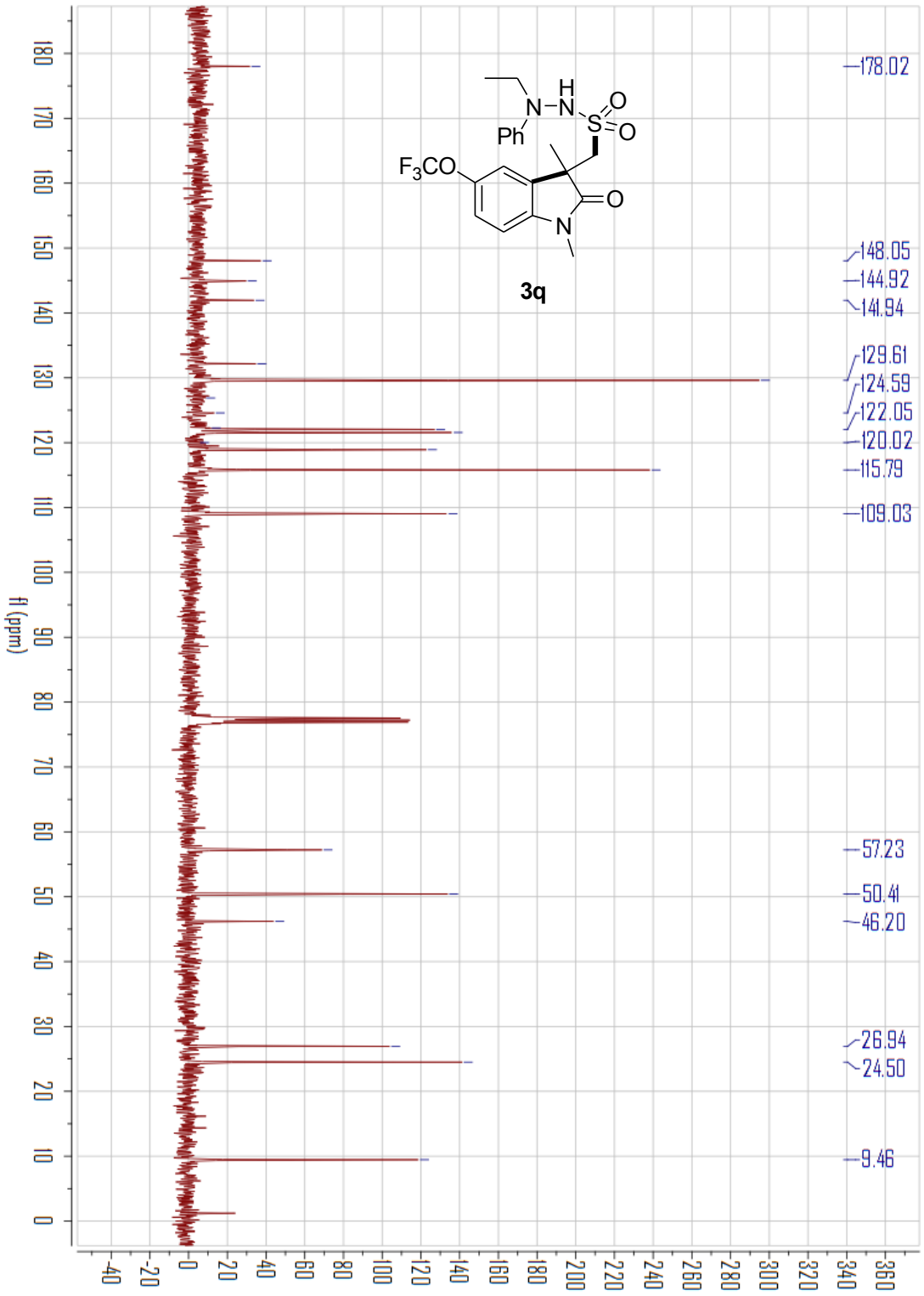

3o
f1 (ppm)
1.96
0.99
2.97
1.12
0.99
0.90
1.20
1.74
1.08
3.00
3.09
3.13
7.27
7.25
7.15
7.14
7.13
7.12
7.01
7.00
6.99
6.99
6.98
6.97
6.97
6.96
6.95
6.93
6.91
6.78
6.77
6.75
6.74
6.44
3.68
3.65
3.61
3.60
3.55
3.52
3.17
1.41
1.05
1.03
1.02


177.94
160.62
158.23
148.06
139.23
132.41
129.65
121.64
112.84
109.17
109.09
57.46
50.41
46.37
26.95
24.58
9.46
f1 (ppm)
3o


3p
f1 (ppm)
1.77
1.02
1.06
2.06
1.18
1.07
0.96
0.95
1.00
2.83
3.31
3.17
3.29
7.28
7.26
7.26
7.20
7.09
7.07
7.00
6.98
6.93
6.92
6.90
6.75
6.73
6.35
3.75
3.72
3.61
3.58
3.18
3.17
2.31
1.42


178.24
150.10
140.79
130.70
129.27
121.21
114.71
108.45
57.68
46.02
44.09
26.80
24.80
21.37
f1 (ppm)
3p


3q
f1 (ppm)
2.99
1.05
2.27
0.77
1.03
0.88
1.05
1.79
1.06
3.00
3.01
3.17
7.29
7.27
7.25
7.18
7.16
6.98
6.96
6.93
6.91
6.84
6.82
6.36
3.69
3.65
3.61
3.59
3.55
3.52
3.19
1.43
1.04
1.02
1.00


178.02
148.05
144.92
141.94
129.61
124.59
122.05
120.02
115.79
109.03
57.23
50.41
46.20
26.94
24.50
9.46
f1 (ppm)
3q