Electronic Supplementary Material (ESI) for ChemComm.

Synthesis of 1-(2,3-Dihydrobenzofuran-3-yl)-methanesulfonohydrazides through Insertion of Sulfur Dioxide

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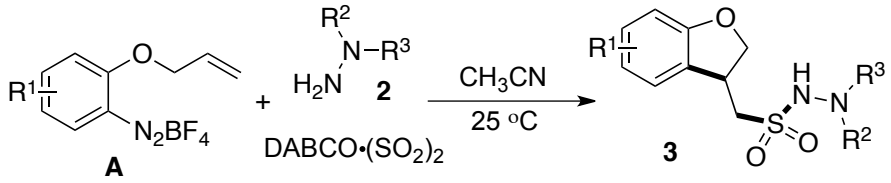
Supporting Information

General Materials and Methods:

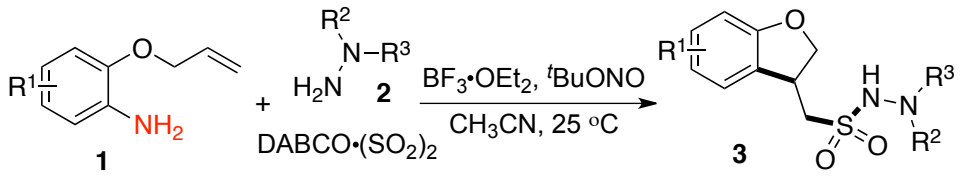
Unless otherwise stated, all commercial reagents were used as received. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63µm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated 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 procedure for the three-component reaction of aryldiazonium ion **A**, DABCO•$(SO_2)_2$ and hydrazines **2**.*

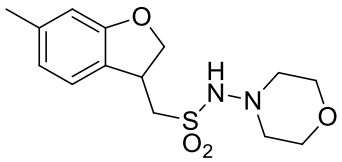


Allyloxyaryldiazonium tetrafluoroborate (0.20 mmol) in CH_3CN (1.0 mL) was added dropwisely to a solution of DABCO•$(SO_2)_2$ (0.12 mmol) and hydrazine (0.24 mmol) in CH_3CN (3.0 mmol) under N_2 in 10 minutes. The mixture was stirred at room temperature for another 10 minutes. Then the solvent was evaporated and the residue was purified directly by flash column chromatograph (EtOAc/n-hexane, 1:2) to give the desired product **3**.

General procedure for the three-component reaction of 2-(allyloxy)anilines, sulfur dioxide and hydrazines.

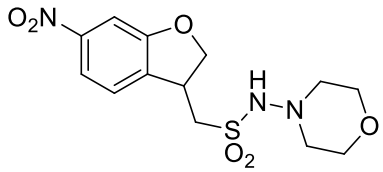


^{t}BuONO (1.8 equiv) was dropwisely added to a solution of 2-(allyloxy)aniline (0.20 mmol) and BF_3•OEt_2 (1.8 equiv) in CH_3CN (1.0 mL) at 0 °C. After 5 min, the above mixture was slowly added into a mixture of DABCO•$(SO_2)_2$ (0.18 mmol) and morpholin-4-amine **2a** (0.30 mmol) in CH_3CN (3.0 mL) under N_2 protection in 10 min. The mixture was stirred at room temperature. After completion of the reaction, the solvent was evaporated and the residue was purified directly by flash column chromatograph (EtOAc/n-hexane, 1: 2) to give the desired product **3**.



1-(6-Methyl-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3a**)

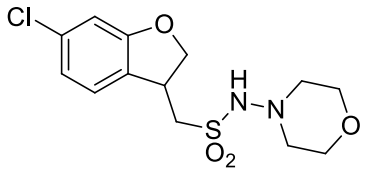
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.31 (s, 3H), 2.85-2.94 (m, 4H), 3.30 (dd, J_1 = 10.9 Hz, J_2 = 14.1 Hz, 1H), 3.57 (dd, J_1 = 3.0 Hz, J_2 = 14.2 Hz, 1H), 3.77 (t, J = 4.5 Hz, 4H), 3.96-4.02 (m, 1H), 4.59 (dd, J_1 = 6.0 Hz, J_2 = 9.6 Hz, 1H), 4.70-4.75 (m, 1H), 5.48 (s, 1H), 6.66 (s, 1H), 6.72 (d, J = 7.6 Hz, 1H), 7.06 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 37.2, 54.3, 57.5, 66.6, 76.2, 110.9, 121.7, 123.8, 124.1, 139.8, 160.1; HRMS (ESI) calcd for $C_{14}H_{20}N_2O_4S$: 313.1217 ($M + H^+$), found: 313.1247.



N-Morpholino-1-(6-nitro-2,3-dihydrobenzofuran-3-yl)methanesulfonamide (**3b**)

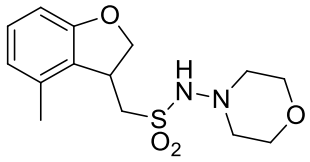
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.90-2.93 (m, 4H), 3.38 (dd, J_1 = 10.4 Hz, J_2 = 14.1 Hz, 1H), 3.59 (dd, J_1 = 3.6 Hz, J_2 = 14.1 Hz, 1H), 3.79 (t, J = 4.6 Hz, 4H), 4.10-4.17(m, 1H), 4.73 (dd, J_1 = 6.4 Hz, J_2 = 9.8 Hz, 1H), 4.87-4.92 (m, 1H), 5.29 (s, 1H), 7.35 (d, J = 8.2 Hz, 1H), 7.63 (d, J = 1.9 Hz, 1H), 7.82 (dd, J_1 = 1.9Hz, J_2 = 8.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$)

δ 37.2, 53.8, 57.6, 66.6, 76.7, 105.5, 116.7, 124.5, 134.6, 149.5, 160.6; HRMS (ESI) calcd for $C_{13}H_{17}N_3O_6S$: 344.0911 ($M + H^+$), found: 344.0912.



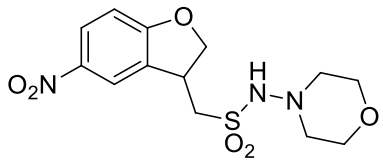
1-(6-Chloro-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3c**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.85-2.93 (m, 4H), 3.31 (dd, J_1 = 10.6 Hz, J_2 = 14.1 Hz, 1H), 3.54 (dd, J_1 = 3.2 Hz, J_2 = 14.1 Hz, 1H), 3.76 (m, 4H), 3.96-4.03 (m, 1H), 4.62 (dd, J_1 = 6.1 Hz, J_2 = 9.7 Hz, 1H), 4.74-4.78 (m, 1H), 5.62 (s, 1H), 6.81 (d, J = 1.3 Hz, 1H), 6.87(dd, J_1 = 1.4Hz, J_2 = 8.0 Hz, 1H), 7.10 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.0, 54.1, 57.5, 66.6, 110.9, 121.1, 124.9, 125.9, 134.8, 160.7; HRMS (ESI) calcd for $C_{13}H_{17}ClN_2O_4S$: 333.0670 ($M + H^+$), found: 333.0666.



1-(4-Methyl-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3d**)

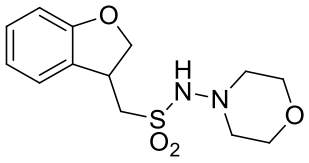
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.33 (s, 3H), 2.88-2.95 (m, 4H), 3.28 (dd, J_1 = 11.5 Hz, J_2 = 14.2 Hz, 1H), 3.45 (d, J = 13.9 Hz, 1H), 3.76 (t, J = 4.6 Hz, 4H), 3.92-3.97 (m, 1H), 4.50-4.55 (m, 1H), 4.87 (dd, J_1 = 2.5 Hz, J_2 = 9.7 Hz, 1H), 5.43 (s, 1H), 6.68-6.72 (m, 2H), 7.10 (t, J = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.3, 36.7, 52.1, 57.6, 66.6, 75.1, 107.7, 122.5, 125.8, 129.4, 134.5, 159.7; HRMS (ESI) calcd for $C_{14}H_{20}N_2O_4S$: 313.1217 ($M + H^+$), found: 313.1213.



N-Morpholino-1-(5-nitro-2,3-dihydrobenzofuran-3-yl)methanesulfonamide (**3e**)

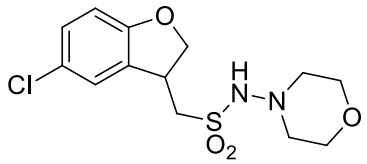
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.89-2.96 (m, 4H), 3.36 (dd, J_1 = 10.8 Hz, J_2 = 14.0 Hz, 1H), 3.67 (dd, J_1 = 3.2 Hz, J_2 = 14.0 Hz, 1H), 3.77-3.81 (m, 4H), 4.11-4.19 (m, 1H) 4.79 (dd, J_1 = 6.5 Hz, J_2 = 10.0 Hz, 1H), 4.93-4.97 (m, 1H), 5.31 (s, 1H), 6.90 (d, J = 8.8 Hz, 1H), 8.13 (s, 1H), 8.18 (dd, J_1 = 2.4Hz, J_2 = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2,

29.7, 36.8, 54.0, 57.4, 57.7, 66.6, 76.7, 110.1, 120.9, 126.9, 128.6; HRMS (ESI) calcd for $C_{13}H_{17}N_3O_6S$: 344.0911(M + H^+), found: 344.0920.



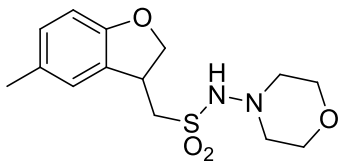
1-(2,3-Dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3f**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.85-2.94 (m, 4H), 3.33 (dd, J_1 = 10.8 Hz, J_2 = 14.0 Hz, 1H), 3.59 (dd, J_1 = 3.2 Hz, J_2 = 14.4 Hz, 1H), 3.77 (t, J = 4.4 Hz, 4H), 4.00-4.07 (m, 1H), 4.60 (dd, J_1 = 6.4 Hz, J_2 = 8.8 Hz, 1H), 4.74 (t, J = 8.8 Hz, 1H), 5.55 (s, 1H), 6.83 (d, J = 8.0 Hz, 1H), 6.90 (t, J = 7.6 Hz, 1H), 7.18 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.4, 54.1, 57.4, 66.6, 75.9, 110.2, 120.9, 124.2, 127.0, 129.4, 159.7; HRMS (ESI) calcd for $C_{13}H_{18}N_2O_4S$: 299.1066 (M + H^+), found: 299.1092



1-(5-Chloro-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3g**)

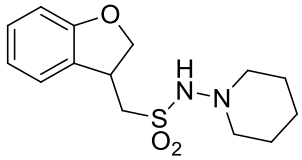
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.89-2.91 (m, 4H), 3.32 (dd, J_1= 10.8 Hz, J_2 = 14.1 Hz, 1H), 3.56 (dd, J_1 = 3.1 Hz, J_2 = 14.2 Hz, 1H), 3.78 (t, J = 4.5 Hz, 4H), 4.04 (s, 1H), 4.62 (dd, J_1 = 6.2 Hz, J_2 = 9.6 Hz, 1H), 4.74-4.79 (m, 1H), 5.42 (s, 1H), 6.74-6.76 (m, 1H), 7.13-7.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.5, 54.0, 57.5, 66.6, 76.5, 111.2, 124.5, 125.6, 129.0, 129.3, 158.5; HRMS (ESI) calcd for $C_{13}H_{17}ClN_2O_4S$: 355.0490 (M + Na^+), found: 355.0483.



1-(5-Methyl-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3h**)

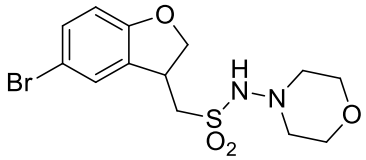
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.29 (s, 3H), 2.89-2.92 (m, 4H), 3.32 (dd, J_1 = 10.9 Hz, J_2 = 14.2 Hz, 1H), 3.58 (dd, J_1 = 3.0 Hz, J_2 = 14.2 Hz, 1H), 3.78 (t, J = 4.5 Hz, 4H), 3.98-4.00 (m, 1H), 4.58 (dd, J_1 = 6.1 Hz, J_2 = 9.6 Hz, 1H), 4.70-4.74 (m, 1H), 5.53 (s, 1H),

6.71-6.73 (m, 1H), 6.97-6.99 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.8, 37.5, 54.2, 57.5, 66.6, 109.7, 124.7, 127.0, 129.8, 130.3, 157.7; HRMS (ESI) calcd for $C_{14}H_{20}N_2O_4S$: 335.1036 (M + Na^+), found: 335.1020.



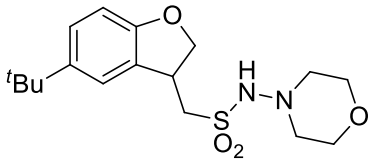
1-(2,3-Dihydrobenzofuran-3-yl)-*N*-(piperidin-1-yl)methanesulfonamide (**3i**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 1.41 (s, 2H), 1.68 (dt, J_1 = 5.6 Hz, J_2 = 11.2 Hz, 4H), 2.81 (s, 4H), 3.30 (dd, J_1 = 10.9 Hz, J_2 = 14.2 Hz, 1H), 3.62 (dd, J_1 = 2.9 Hz, J_2 = 14.2 Hz, 1H), 4.01-4.08 (m, 1H), 4.60 (dd, J_1 = 6.3 Hz, J_2 = 9.7 Hz, 1H), 4.73-4.78 (m, 1H), 5.38 (s, 1H), 6.83 (d, J = 8.0 Hz, 1H), 6.89-6.92 (m, 1H), 7.16-7.21 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.1, 25.7, 37.6, 54.0, 58.5, 76.1, 110.1, 120.9, 124.2, 127.9, 129.3, 159.8; HRMS (ESI) calcd for $C_{14}H_{20}N_2O_3S$: 297.1267 (M + H^+), found: 297.1280.



1-(5-Bromo-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3j**)

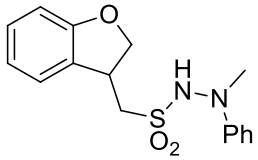
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.88-2.91 (m, 4H), 3.28-3.34 (m, 1H), 3.56 (d, J = 14.1 Hz), 3.78 (s, 4H), 4.04 (s, 1H), 4.59-4.63 (m, 1H), 4.76 (t, J = 9.02 Hz, 1H), 5.40 (s, 1H), 6.71 (d, J = 8.2 Hz), 7.29 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.4, 54.0, 57.5, 66.6, 76.4, 111.8, 112.5, 127.3, 129.5, 132.2, 159.0; HRMS (ESI) calcd for $C_{13}H_{17}BrN_2O_4S$: 398.9985 (M + Na^+), found: 398.9961.



1-(5-(*tert*-Butyl)-2,3-dihydrobenzofuran-3-yl)-*N*-morpholinomethanesulfonamide (**3k**)

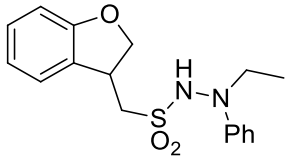
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.30 (s, 9H), 2.87-2.93 (m, 4H), 3.38 (dd, J_1 = 10.9 Hz, J_2 = 14.1 Hz, 1H), 3.60 (dd, J_1 = 3.0 Hz, J_2 = 14.2 Hz, 1H), 3.78 (t, J = 4.6 Hz), 4.00-4.02 (m, 1H), 4.57-4.61 (m, 1 H), 4.71-4.75 (m, 1H), 5.69 (s, 1H), 6.75-6.78 (m, 1H), 7.21-7.22

(m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.7, 34.4, 37.6, 54.3, 57.4, 66.6, 76.2, 109.4, 121.0, 126.4, 126.7, 144.2, 157.6; HRMS (ESI) calcd for $C_{17}H_{26}N_2O_4S$: 377.1505 (M + Na^+), found: 377.1485.



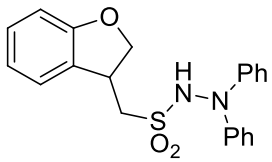
1-(2,3-Dihydrobenzofuran-3-yl)-*N*'-methyl-*N*'-phenylmethanesulfonohydrazide (**3l**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.22-3.29 (m, 4H), 3.53 (dd, J_1 = 2.9 Hz, J_2 = 14.3 Hz, 1H), 4.01-4.08 (m, 1H), 4.54 (dd, J_1 = 6.0 Hz, J_2 = 9.7 Hz, 1H), 4.70-4.75 (m, 1H), 6.13 (s, 1H), 6.78-6.84 (m, 2H), 6.95-6.99 (m, 2H), 7.06 (d, J = 8.2 Hz, 2H), 7.14 (t, J = 7.7 Hz, 1H), 7.25-7.33 (m, 2H); ^{13}C NMR (100 MHz, CDCl3) δ 159.8, 149.7, 129.5, 129.4, 126.8, 124.3, 121.7, 120.9, 114.7, 110.1, 76.1, 55.1, 44.7, 37.4; HRMS (ESI) calcd for $C_{16}H_{18}N_2O_3S$: 319.1111 (M + H^+), found: 319.1104.



1-(2,3-Dihydrobenzofuran-3-yl)-*N*'-ethyl-*N*'-phenylmethanesulfonohydrazide (**3m**)

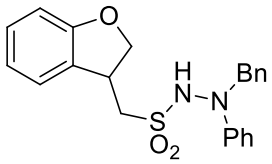
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.11 (t, J = 7.1 Hz, 3H), 3.22 (dd, J_1 = 10.7 Hz, J_2 = 14.2 Hz, 1H), 3.49 (dd, J_1 = 2.8 Hz, J_2 = 14.3 Hz, 1H), 3.72 (d, J = 6.1 Hz, 2H), 3.93-4.06 (m, 1H), 4.52 (dd, J_1 = 6.1 Hz, J_2 = 9.7 Hz, 1H), 4.68-4.73 (m, 1H), 6.46 (s, 1H), 6.77-6.82 (m, 2H), 6.88 (s, 1H) 6.98 (t, J = 7.3 Hz, 1H), 7.07 (d, J = 8.2 Hz, 2H), 7.11-7.15 (m, 1H), 7.30-7.33 (m, 2H); ^{13}C NMR (100 MHz, CDCl3) δ 9.1, 37.5, 50.6, 55.2, 76.1, 110.0, 116.0, 120.9, 121.9, 124.3, 126.9, 129.3, 129.6, 147.9, 159.8; HRMS (ESI) calcd for $C_{17}H_{20}N_2O_3S$: 333.1267 (M + H^+), found: 333.1260.



1-(2,3-Dihydrobenzofuran-3-yl)-*N*', *N*'-diphenylmethanesulfonohydrazide (**3n**)

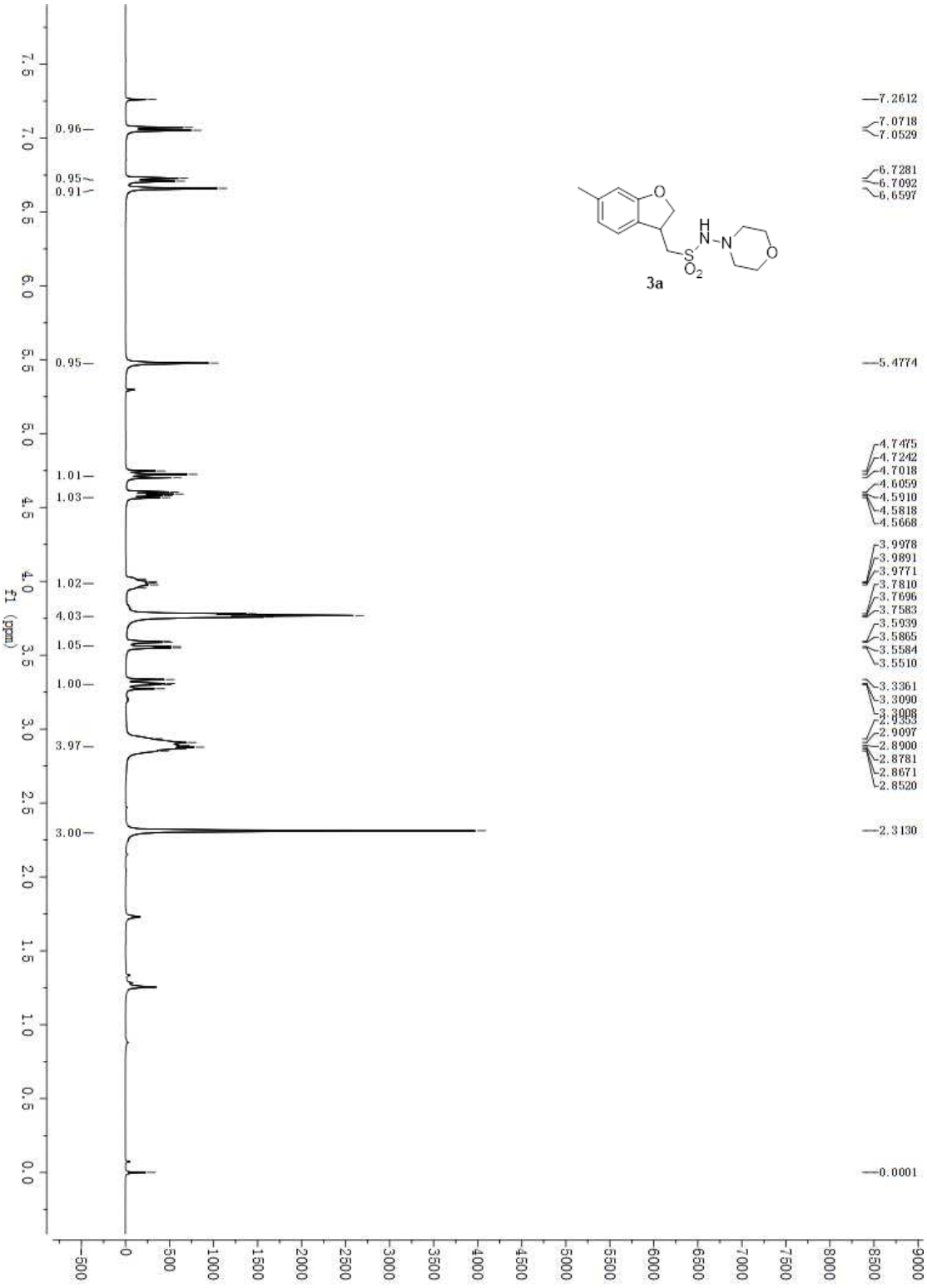
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.08 (dd, J_1 = 10.6 Hz, J_2 = 14.5 Hz, 1H), 3.29 (dd, J_1 = 2.9 Hz, J_2 = 14.6 Hz, 1H), 3.77-3.84 (m, 1H), 4.39 (dd, J_1 = 6.4 Hz, J_2 = 9.7 Hz, 1H), 4.60 (t, J = 9.3 Hz, 1H), 6.59 (d, J = 7.3 Hz, 1H), 6.73-6.78 (m, 2H), 6.96 (s, 1H), 7.08-7.15 (m,

3H), 7.21-7.23 (m, 4H), 7.32-7.35 (m, 4H); ^{13}C NMR (100 MHz, CDCl3) δ 37.5, 56.4, 75.9, 110.0, 120.8, 124.2, 124.7, 126.6, 129.3, 129.6, 147.1, 159.7; HRMS (ESI) calcd for $C_{21}H_{20}N_2O_3S$: 381.1267 (M + H^+), found: 381.1284.



N'-Benzyl-1-(2,3-dihydrobenzofuran-3-yl)-*N*'-phenylmethanesulfonohydrazide (**3o**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.10 (dd, J_1 = 10.7 Hz, J_2 = 14.4 Hz, 1H), 3.39 (dd, J_1 = 2.8 Hz, J_2 = 14.4 Hz, 1H), 3.89-3.96 (m, 1H), 4.46 (dd, J_1 = 6.1 Hz, J_2 = 9.7 Hz, 1H), 4.62-4.67 (m, 1H), 4.76-4.83 (m, 2H), 6.08 (s, 1H), 6.75-6.79 (m, 3H), 7.00 (t, *J* = 7.3 Hz, 1H), 7.09-7.16 (m, 5H), 7.28-7.33 (m, 5H); ^{13}C NMR (100 MHz, CDCl3) δ 37.5, 55.3, 60.2, 76.0, 110.0, 116.5, 120.8, 122.2, 124.3, 126.8, 128.4, 129.0, 129.1, 129.3, 129.6, 133.9, 149.0, 159.7; HRMS (ESI) calcd for $C_{22}H_{22}N_2O_3S$: 395.1424 (M + H^+), found: 395.1415.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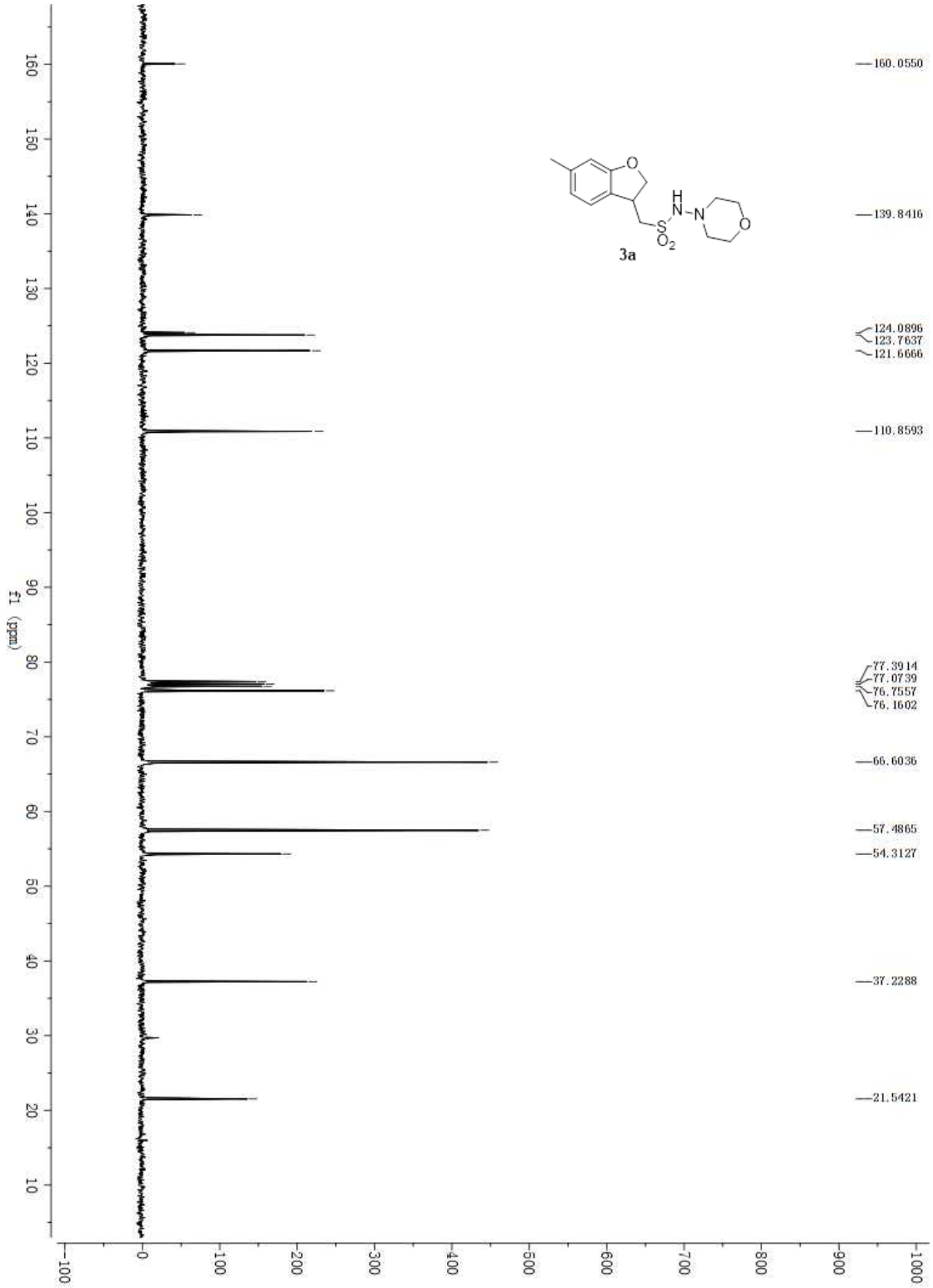
6.7281
6.7092
6.6597
5.4774
4.7475
4.7242
4.7018
4.6059
4.5910
4.5818
4.5668
3.9978
3.9891
3.9771
3.7810
3.7696
3.7583
3.5939
3.5865
3.5584
3.5510
3.3361
3.3090
3.3008
2.9353
2.9097
2.8900
2.8781
2.8671
2.8520
2.3130
0.0001
0.96
0.95
0.91
0.95
1.01
1.03
1.02
4.03
1.05
1.00
3.97
3.00
f1 (ppm)
3a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123.7637
121.6666
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77.3914
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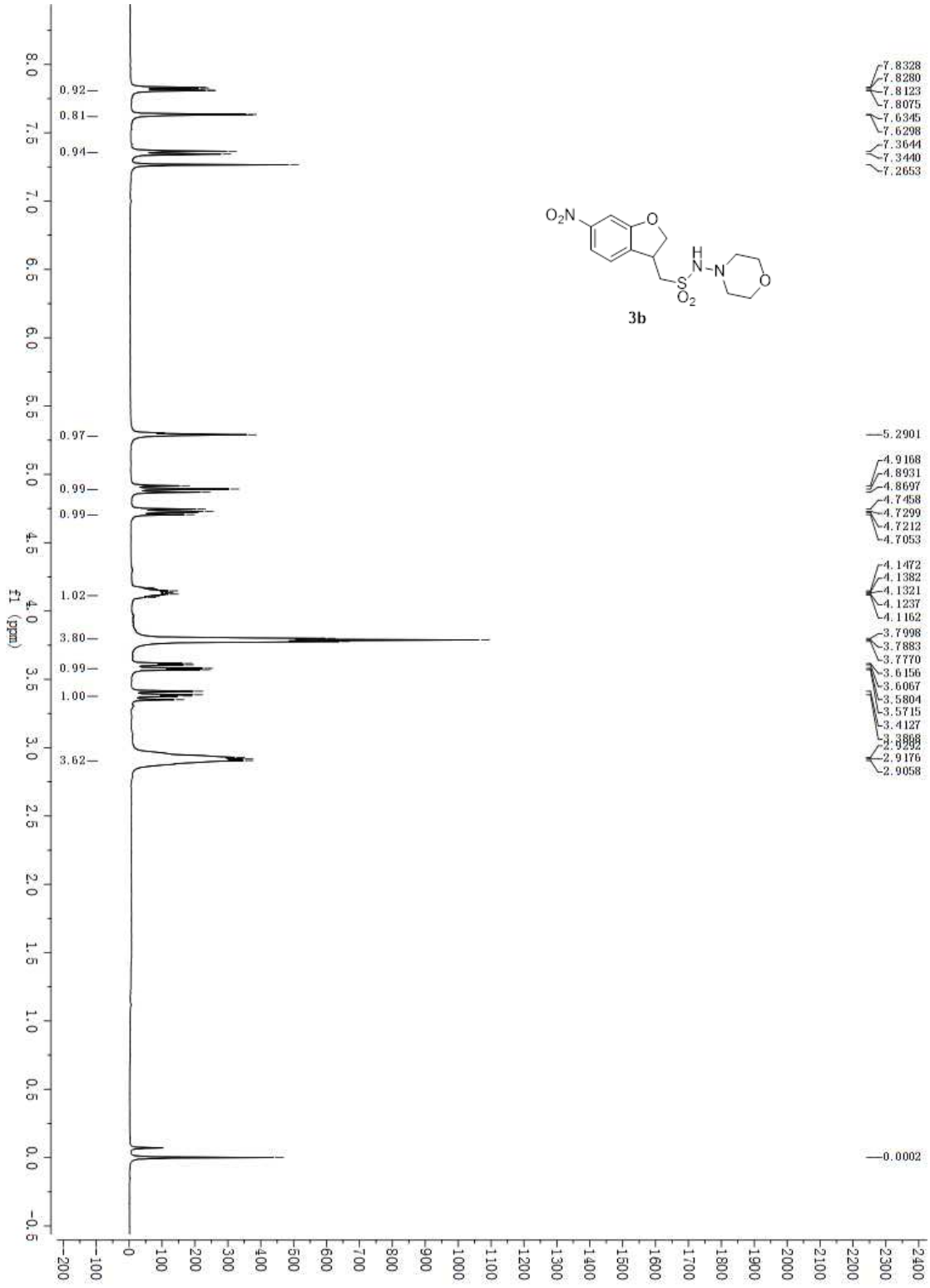
76.1602
66.6036
57.4865
54.3127
37.2288
21.5421
3a
f1 (ppm)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7.8123
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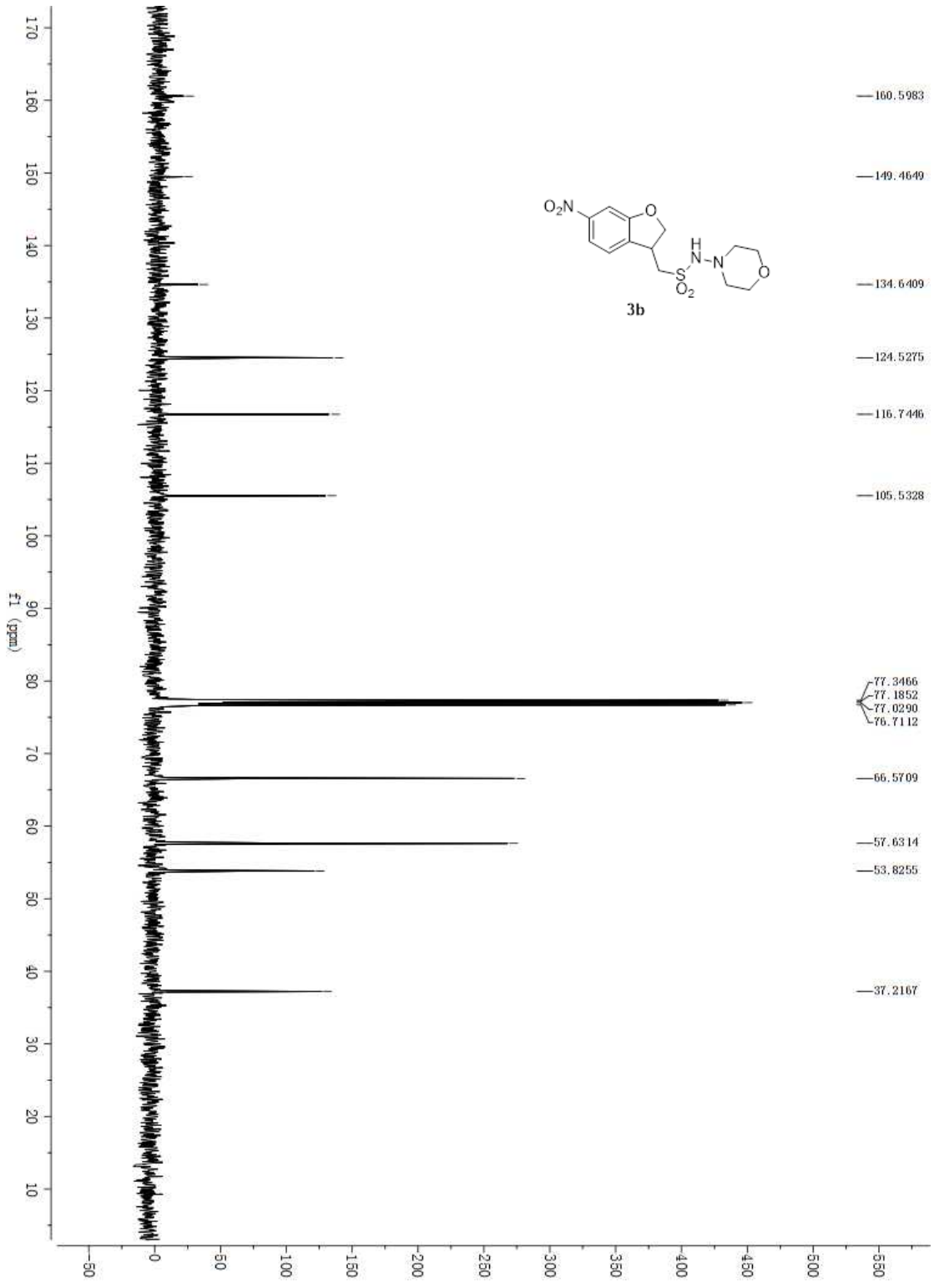
7.6345
7.6298
7.3644
7.3440
7.2653
5.2901
4.9168
4.8931
4.8697
4.7458
4.7299
4.7212
4.7053
4.1472
4.1382
4.1321
4.1237
4.1162
3.7998
3.7883
3.7770
3.6156
3.6067
3.5804
3.5715
3.4127
2.9232
2.9176
2.9058
0.0002
0.92
0.81
0.94
0.97
0.99
0.99
1.02
3.80
0.99
1.00
3.62
f1 (ppm)

3b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134.6409
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116.7446
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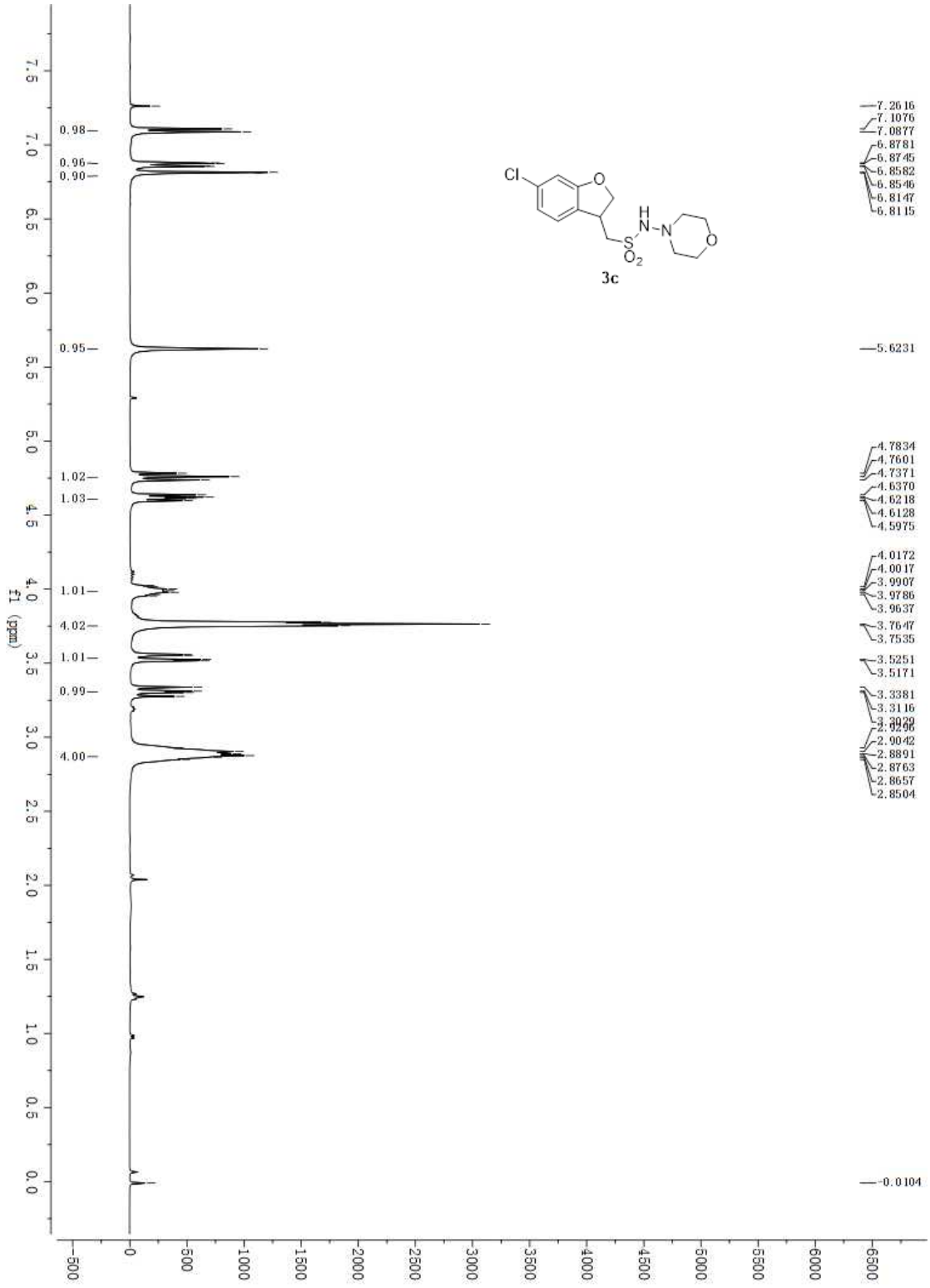
77.0290
76.7112
66.5709
57.6314
53.8255
37.2167
f1 (ppm)
3b


Cl
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H
N
N
O
S
O2
3c
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6.8781
6.8745
6.8582
6.8546
6.8147
6.8115
5.6231
4.7834
4.7601
4.7371
4.6370
4.6218
4.6128
4.5975
4.0172
4.0017
3.9907
3.9786
3.9637
3.7647
3.7535
3.5251
3.5171
3.3381
3.3116
3.2996
2.9042
2.8891
2.8763
2.8657
2.8504
-0.0104
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0.96
0.90
0.95
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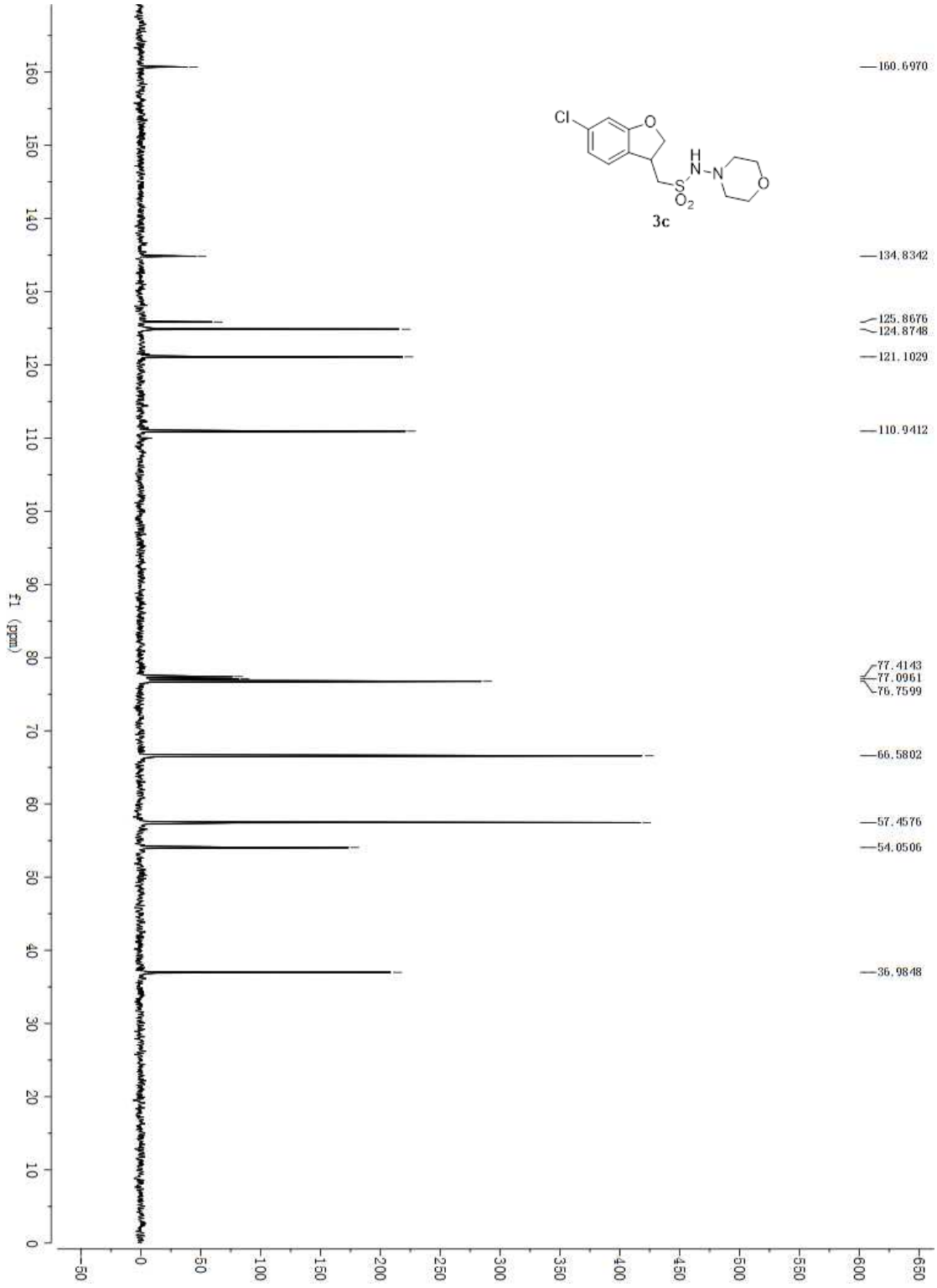
1.03
1.01
4.02
1.01
0.99
4.00
f1 (ppm)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125.8676
124.8748
121.1029
110.9412
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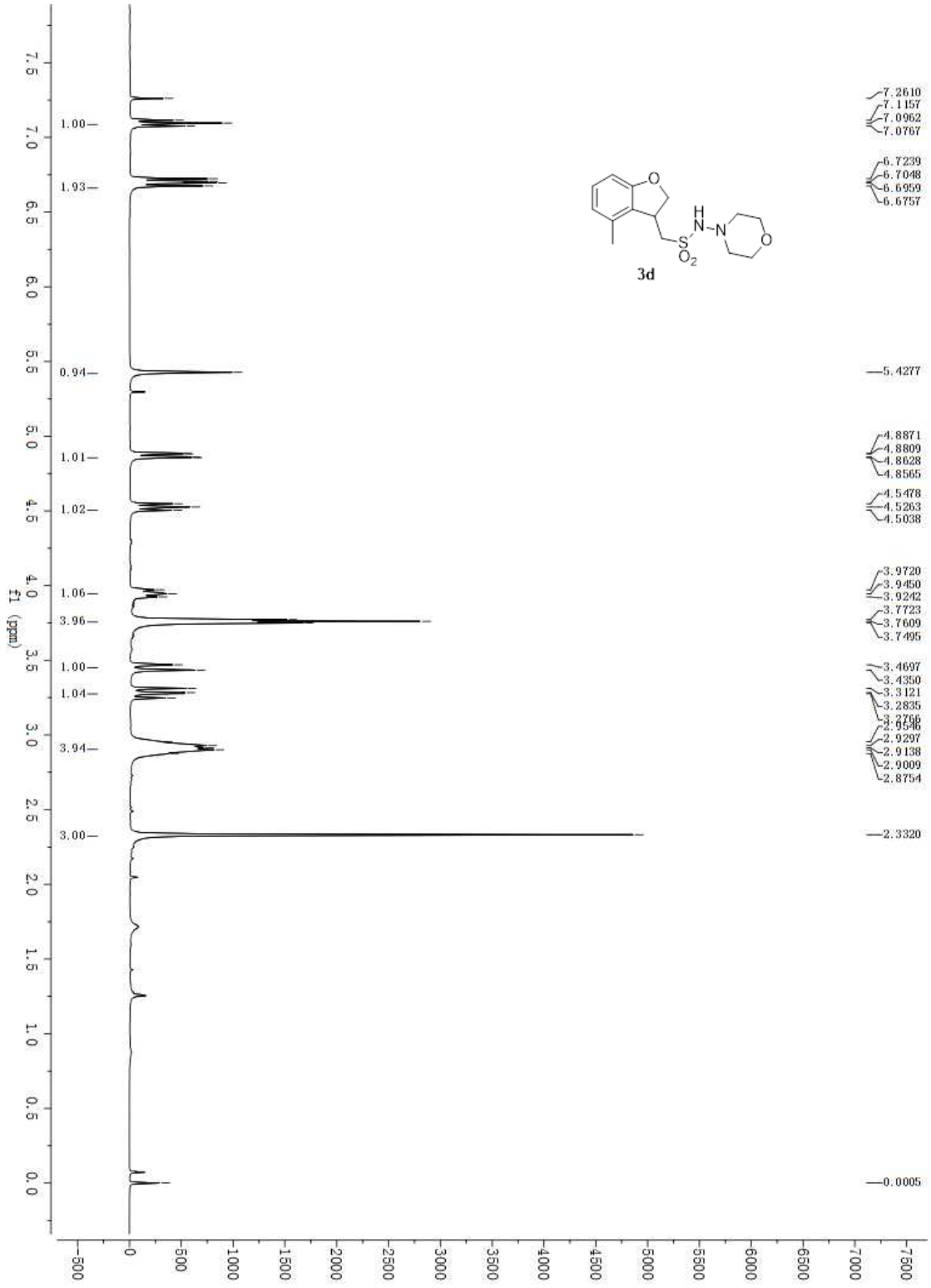
77.0961
76.7599
66.5802
57.4576
54.0506
36.9848
f1 (ppm)
3c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6.6959
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5.4277
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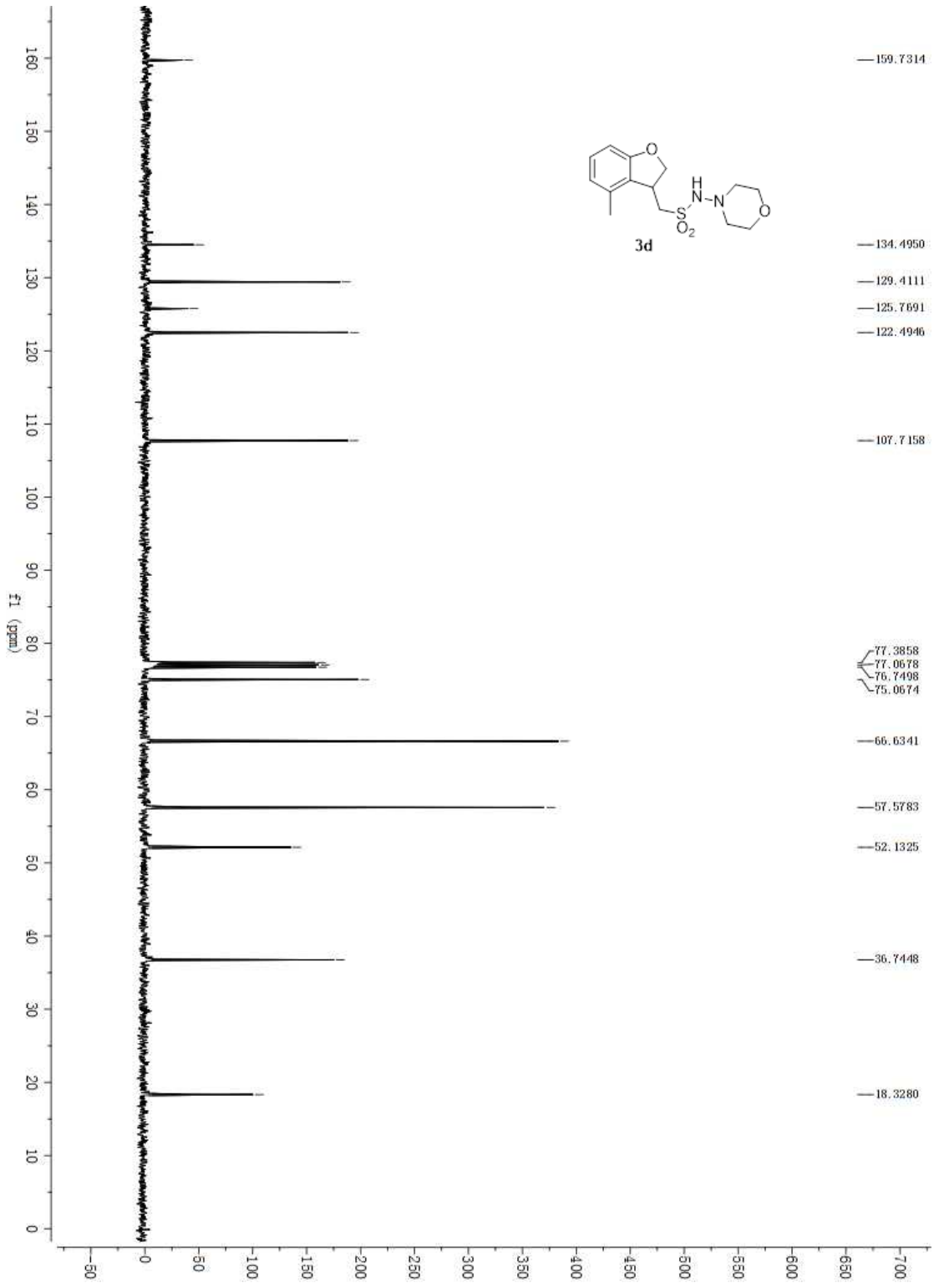
4.8809
4.8628
4.8565
4.5478
4.5263
4.5038
3.9720
3.9450
3.9242
3.7723
3.7609
3.7495
3.4697
3.4350
3.3121
3.2835
3.2766
2.9546
2.9297
2.9138
2.9009
2.8754
2.3320
0.0005
1.00
1.93
0.94
1.01
1.02
1.06
3.96
1.00
1.04
3.94
3.00
f1 (ppm)
3d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122.4946
107.7158
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75.0674
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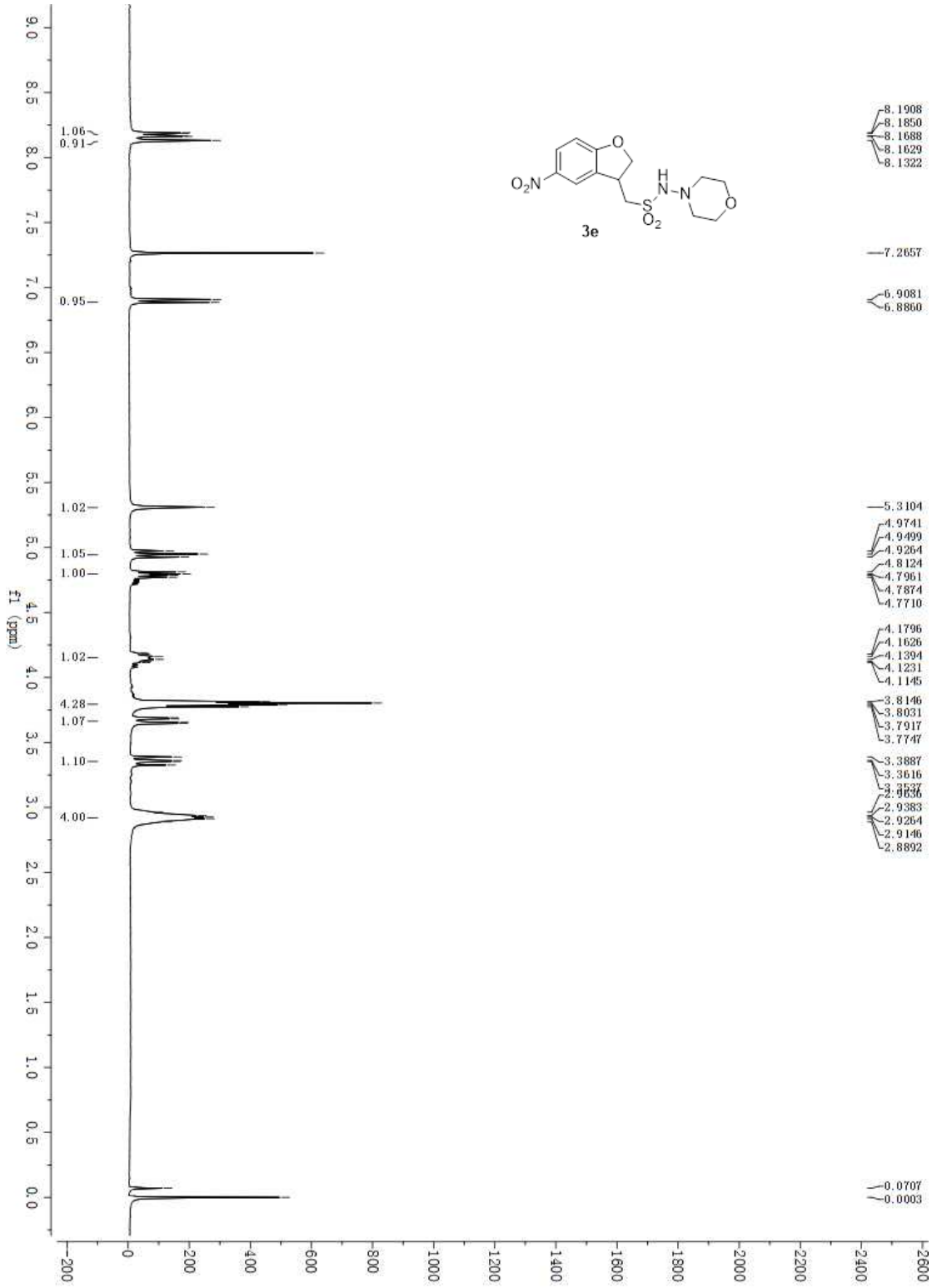
52.1325
36.7448
18.3280
3d
f1 (ppm)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8.1850
8.1688
8.1629
8.1322
7.2657
6.9081
6.8860
5.3104
4.9741
4.9499
4.9264
4.8124
4.7961
4.7874
4.7710
4.1796
4.1626
4.1394
4.1231
4.1145
3.8146
3.8031
3.7917
3.7747
3.3887
3.3616
2.9383
2.9264
2.9146
2.8892
0.0707
0.0003
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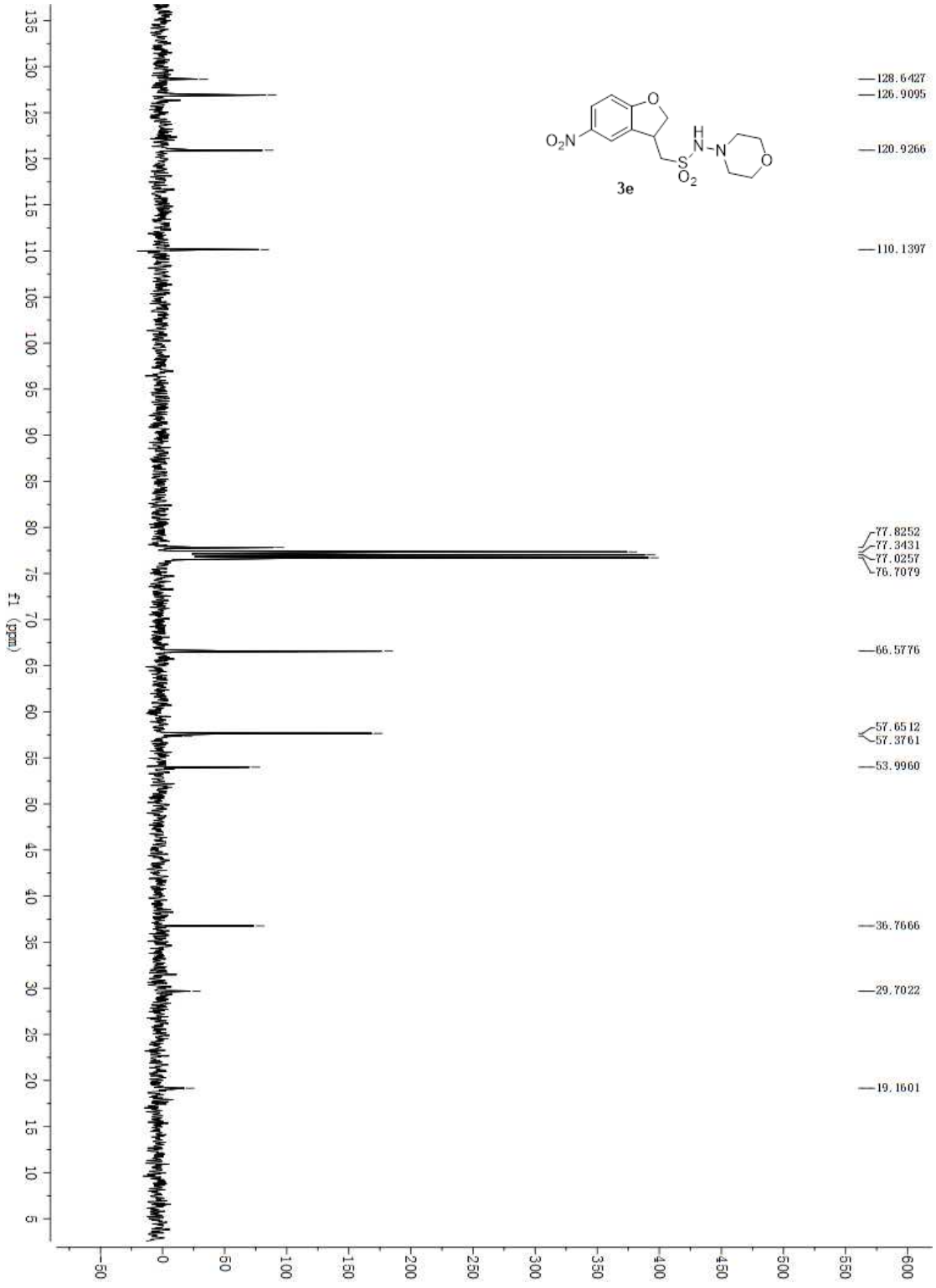
0.91
0.95
1.02
1.05
1.00
1.02
4.28
1.07
1.10
4.00
f1 (ppm)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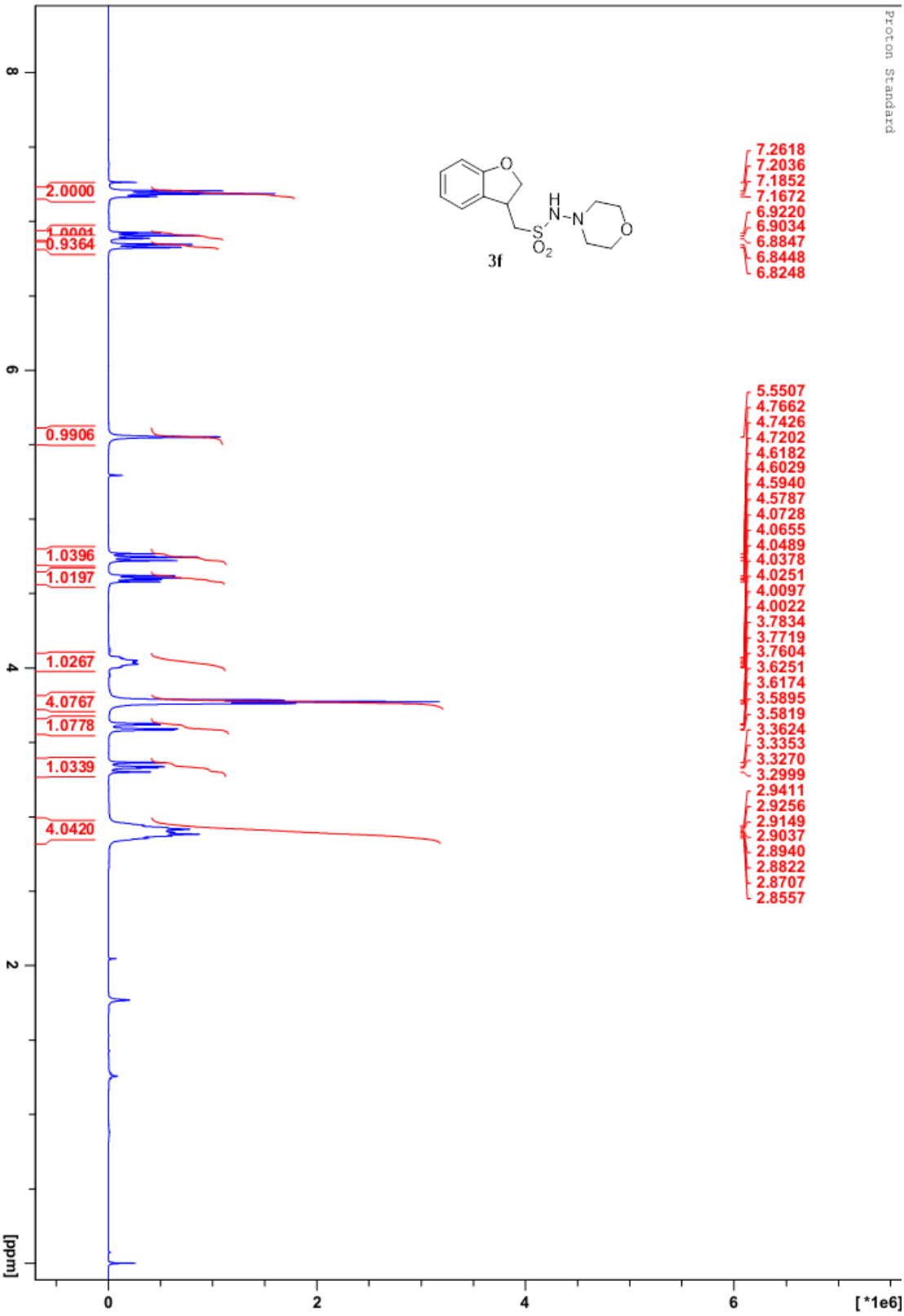
77.3431
77.0257
76.7079
66.5776
57.6512
57.3761
53.9960
36.7666
29.7022
19.1601
f1 (ppm)


proton standard
3f
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7.2036
7.1852
7.1672
6.9220
6.9034
6.8847
6.8448
6.8248
5.5507
4.7662
4.7426
4.7202
4.6182
4.6029
4.5940
4.5787
4.0728
4.0655
4.0489
4.0378
4.0251
4.0097
4.0022
3.7834
3.7719
3.7604
3.6251
3.6174
3.5895
3.5819
3.3624
3.3353
3.3270
3.2999
2.9411
2.9256
2.9149
2.9037
2.8940
2.8822
2.8707
2.8557
2.0000
1.0001
0.9364
0.9906
1.0396
1.0197
1.0267
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4
2
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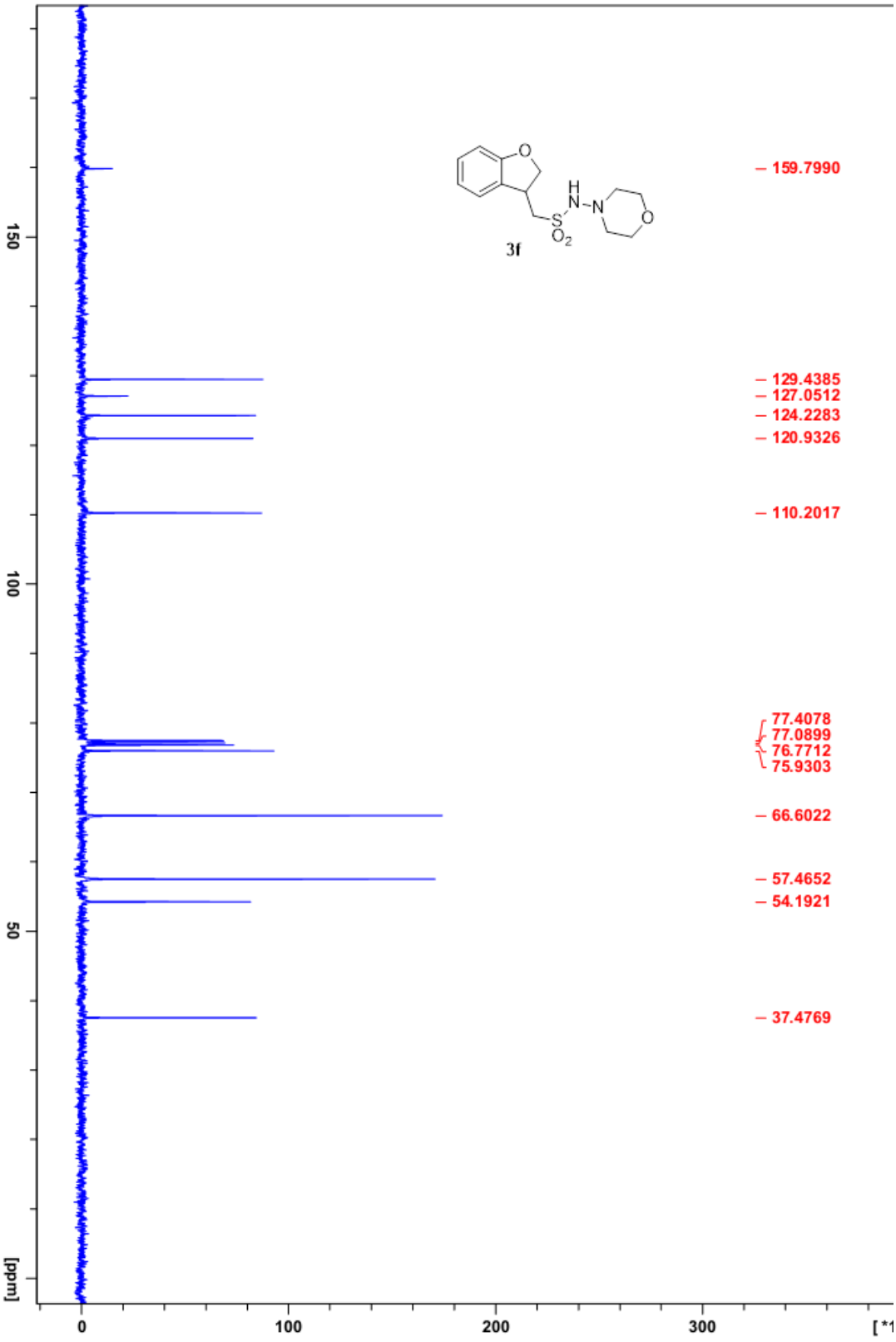
4
6
[*1e6]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76.7712
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57.4652
54.1921
37.4769
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100
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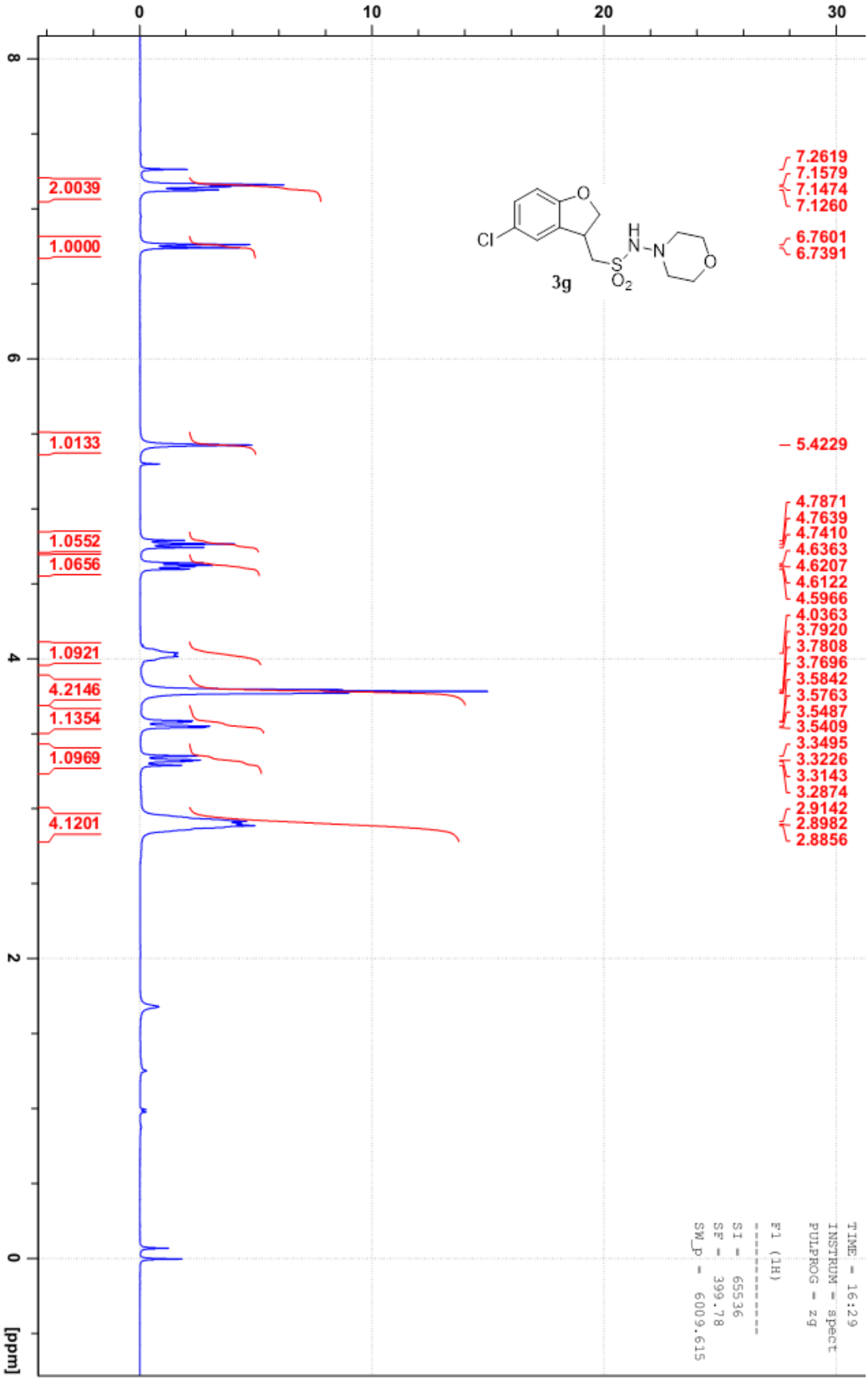
0
100
200
300


0
10
20
30
8
6
4
2
0
[ppm]
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1.0000
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1.0552
1.0656
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4.2146
1.1354
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4.1201
7.2619
7.1579
7.1474
7.1260
6.7601
6.7391
5.4229
4.7871
4.7639
4.7410
4.6363
4.6207
4.6122
4.5966
4.0363
3.7920
3.7808
3.7696
3.5842
3.5763
3.5487
3.5409
3.3495
3.3226
3.3143
3.2874
2.9142
2.8982
2.8856
Cl
O
H
N
N
O
S
O2
3g
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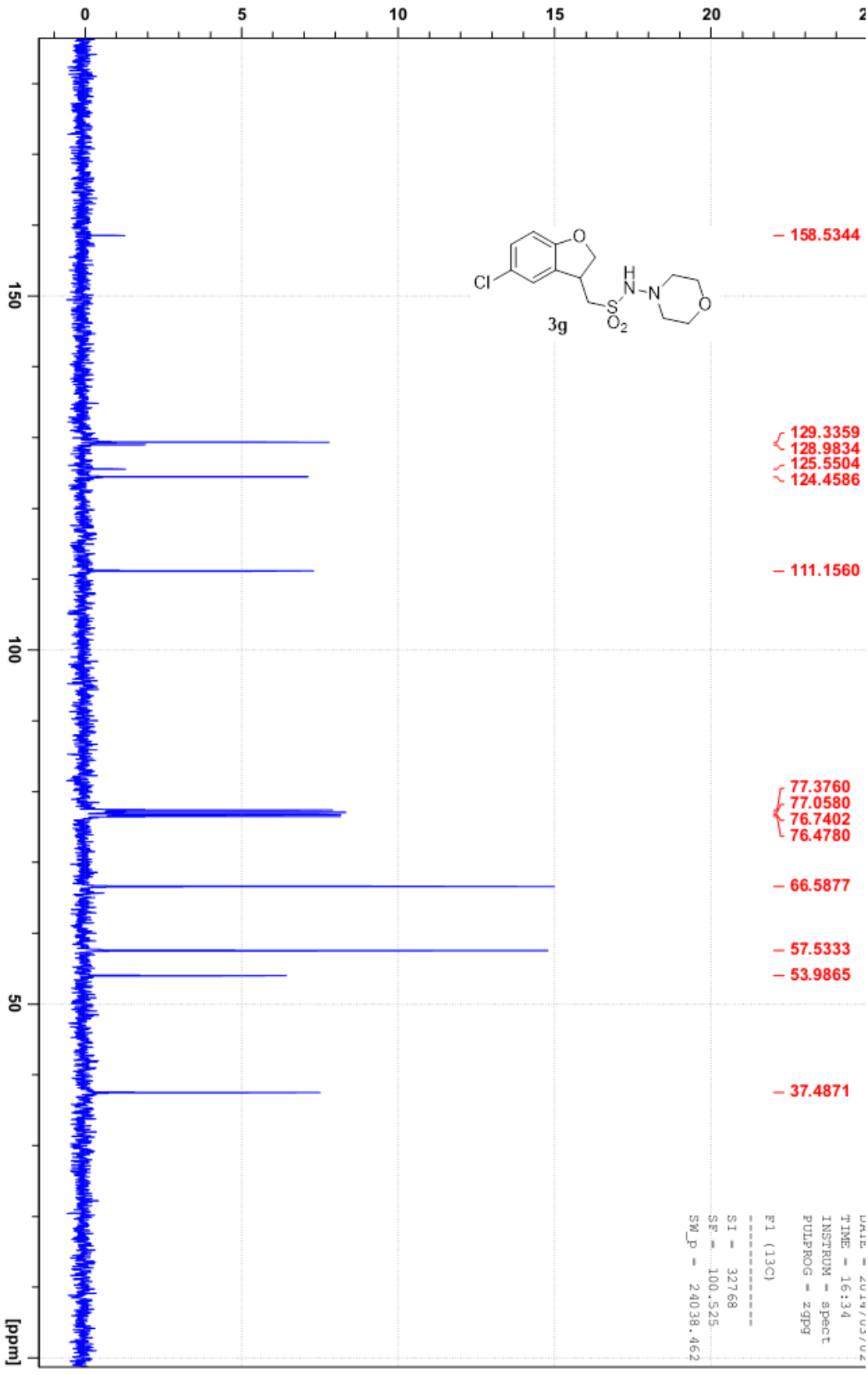
INSTRUM = spect
PULPROG = zg
F1 (1H)
SI = 65536
SF = 399.78
SW_p = 6009.615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10
15
20
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50
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125.5504
124.4586
111.1560
77.3760
77.0580
76.7402
76.4780
66.5877
57.5333
53.9865
37.4871
Cl
O
H
N
N
S
O2
O
3g
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TIME = 16:34
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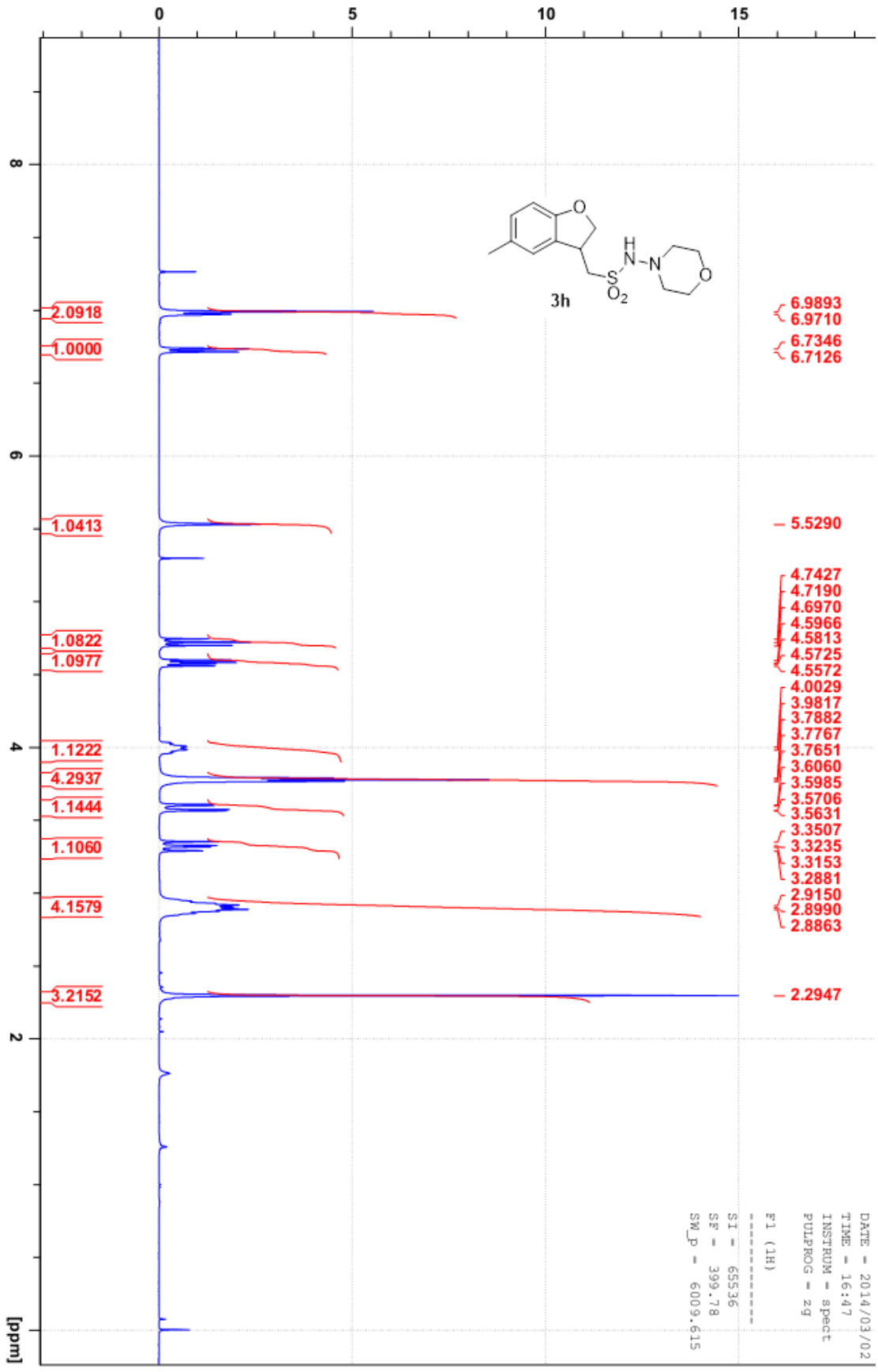
SF = 100.525
SI = 32768
F1 (13C)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10
15
8
6
4
2
[ppm]
3h
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1.0822
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1.1222
4.2937
1.1444
1.1060
4.1579
3.2152
6.9893
6.9710
6.7346
6.7126
5.5290
4.7427
4.7190
4.6970
4.5966
4.5813
4.5725
4.5572
4.0029
3.9817
3.7882
3.7767
3.7651
3.6060
3.5985
3.5706
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3.3507
3.3235
3.3153
3.2881
2.9150
2.8990
2.8863
2.2947
DATE = 2014/03/02
TIME = 16:47
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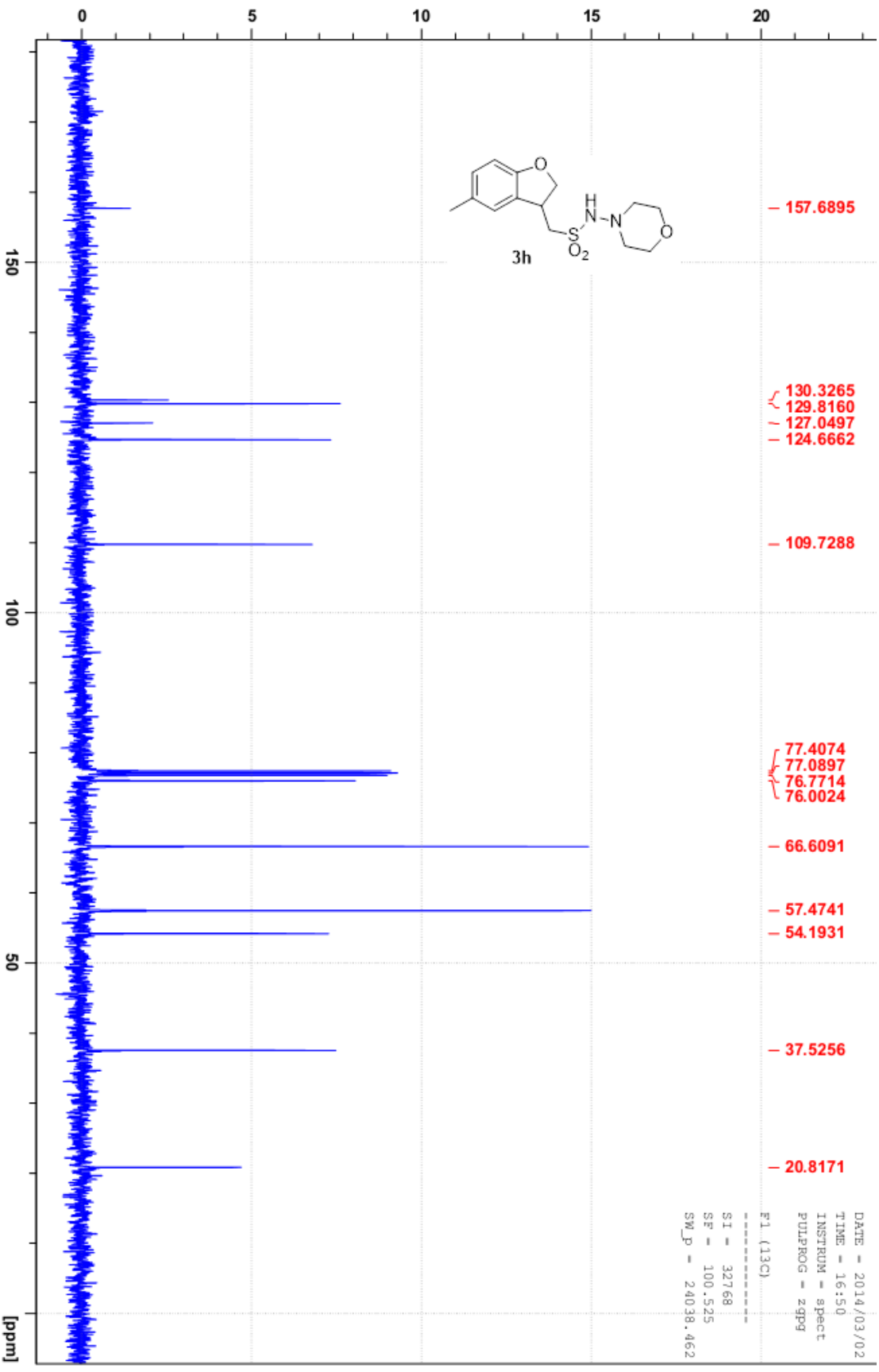
SI = 65536
SF = 399.78
SW_p = 6009.615


0
5
10
15
20
150
100
50
[ppm]
3h
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129.8160
127.0497
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109.7288
77.4074
77.0897
76.7714
76.0024
66.6091
57.4741
54.1931
37.5256
20.8171
DATE - 2014/03/02
TIME - 16:50
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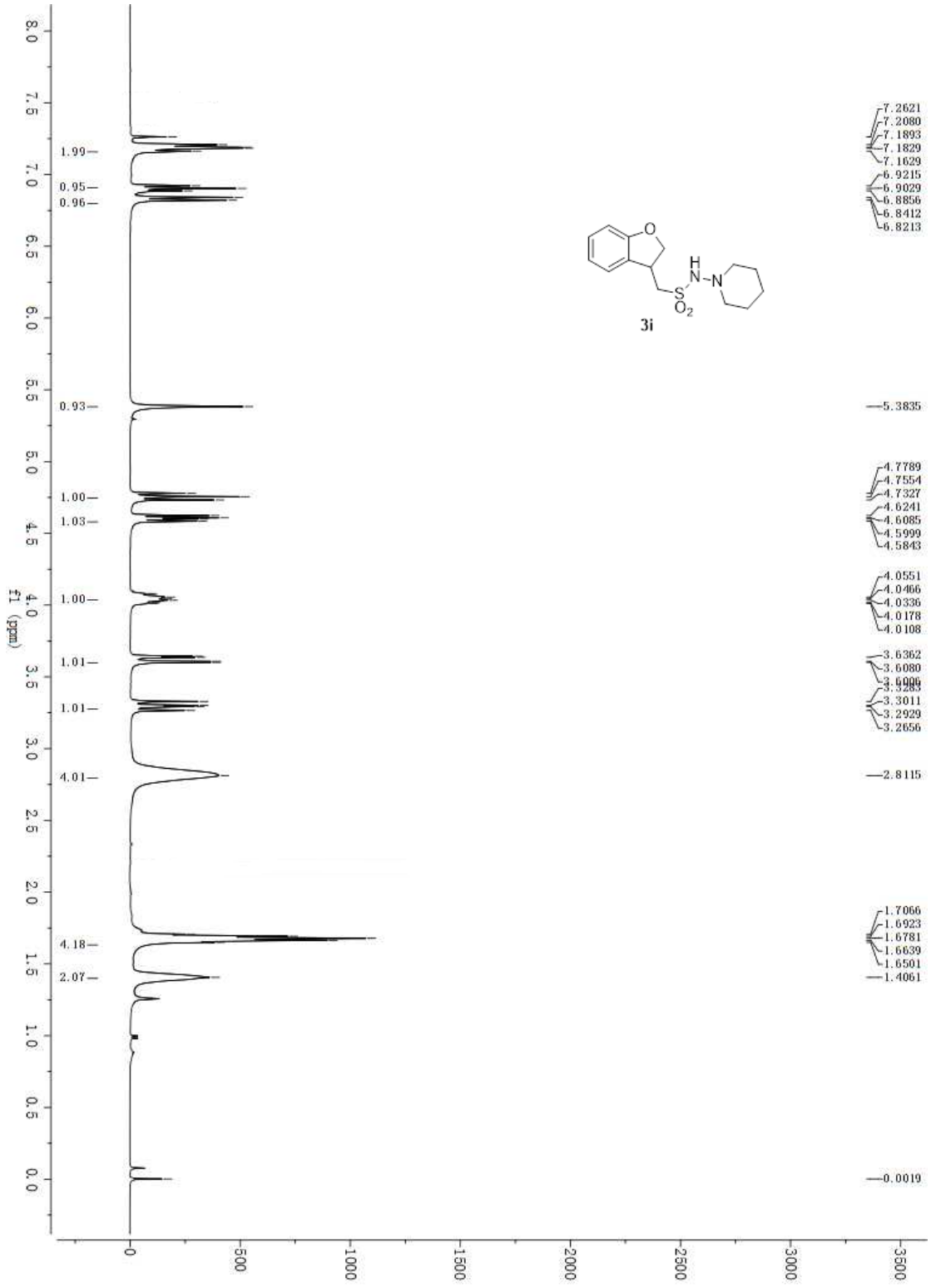
PULPROG - zgpg
F1 (13C)
SI - 32768
SF - 100.525
SW_p - 24038.462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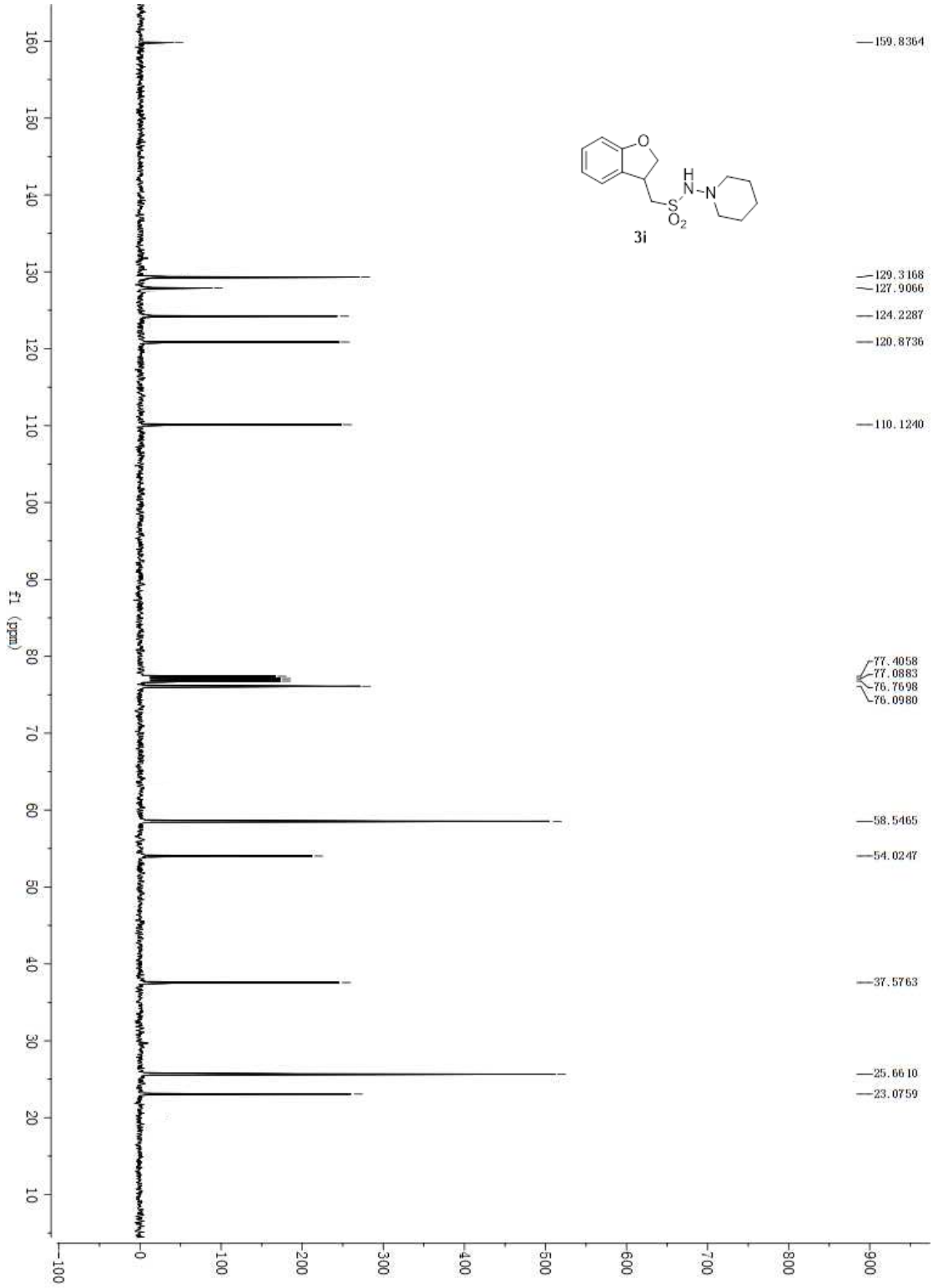
5.3835
4.7789
4.7554
4.7327
4.6241
4.6085
4.5999
4.5843
4.0551
4.0466
4.0336
4.0178
4.0108
3.6362
3.6080
3.3283
3.3011
3.2929
3.2656
2.8115
1.7066
1.6923
1.6781
1.6639
1.6501
1.4061
0.0019
1.99
0.95
0.96
0.93
1.00
1.03
1.00
1.01
1.01
4.01
4.18
2.07
f1 (ppm)
3i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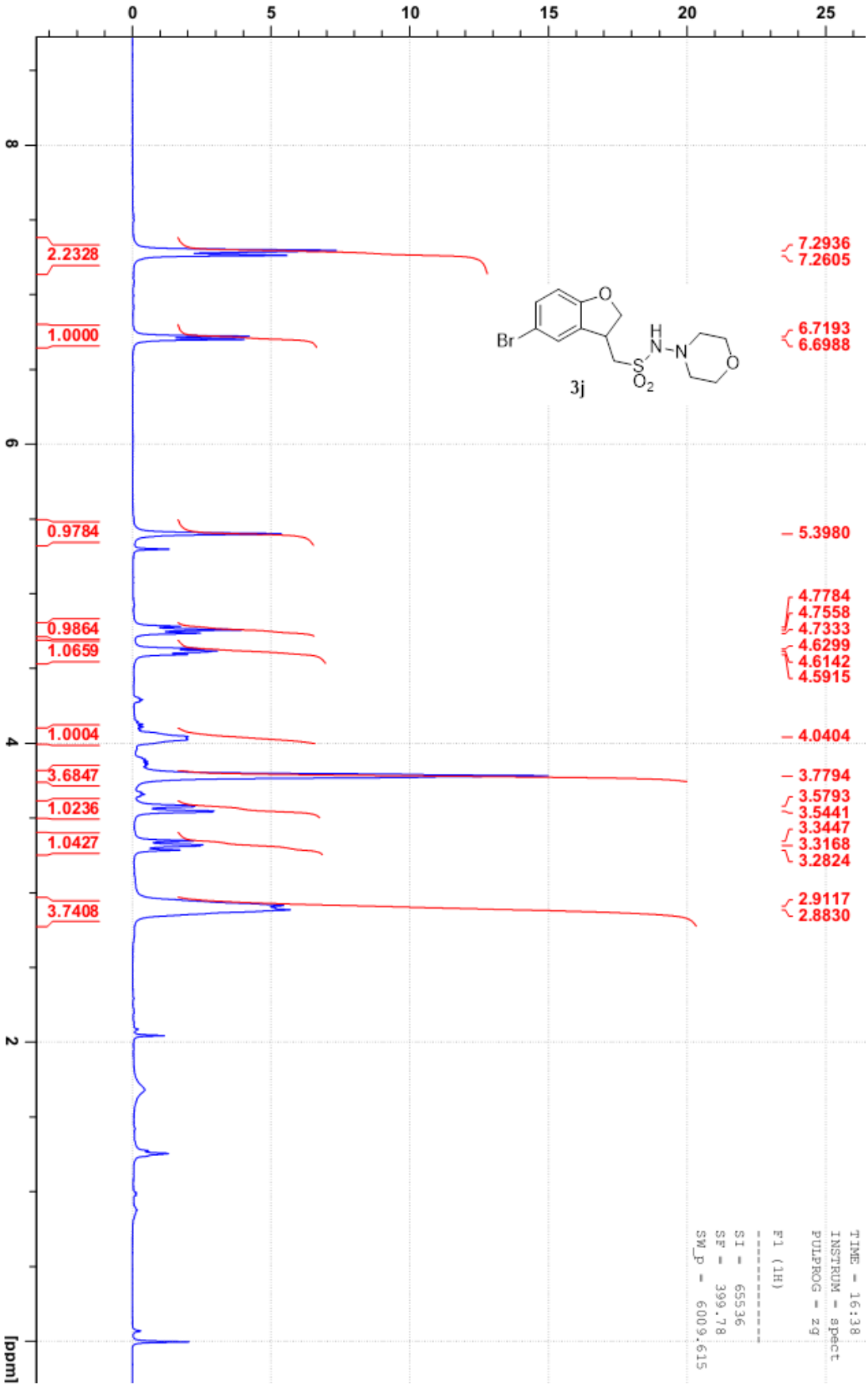
37.5763
25.6610
23.0759
3i
f1 (ppm)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10
15
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25
8
6
4
2
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7.2936
7.2605
6.7193
6.6988
5.3980
4.7784
4.7558
4.7333
4.6299
4.6142
4.5915
4.0404
3.7794
3.5793
3.5441
3.3447
3.3168
3.2824
2.9117
2.8830
Br
O
H
N
N
O
S
O2
3j
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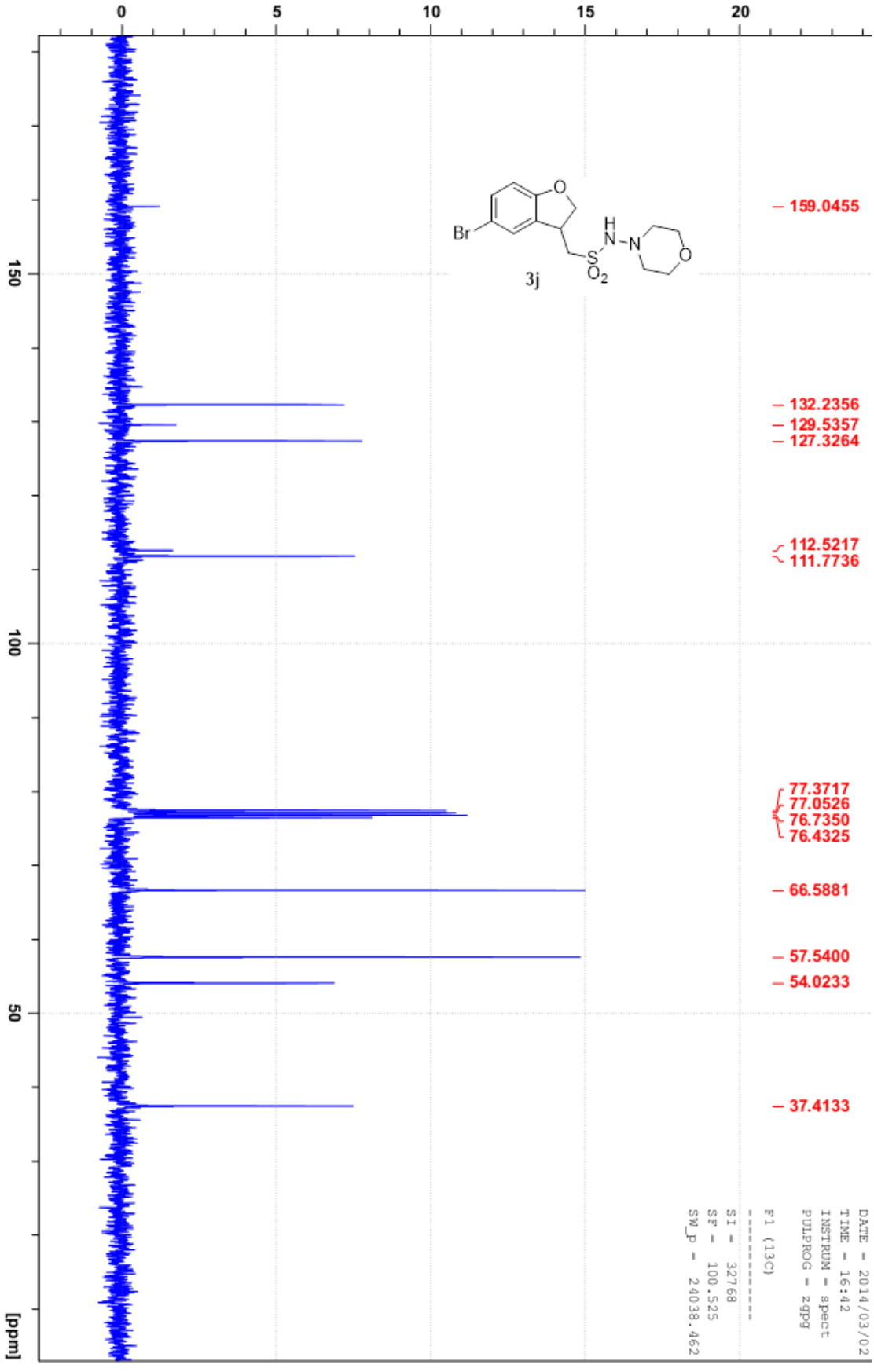
SI = 65536
SF = 399.78
SW_p = 6009.615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10
15
20
150
100
50
[ppm]
Br
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H
N
N
O
S
O2
3j
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129.5357
127.3264
112.5217
111.7736
77.3717
77.0526
76.7350
76.4325
66.5881
57.5400
54.0233
37.4133
DATE = 2014/03/02
TIME = 16:42
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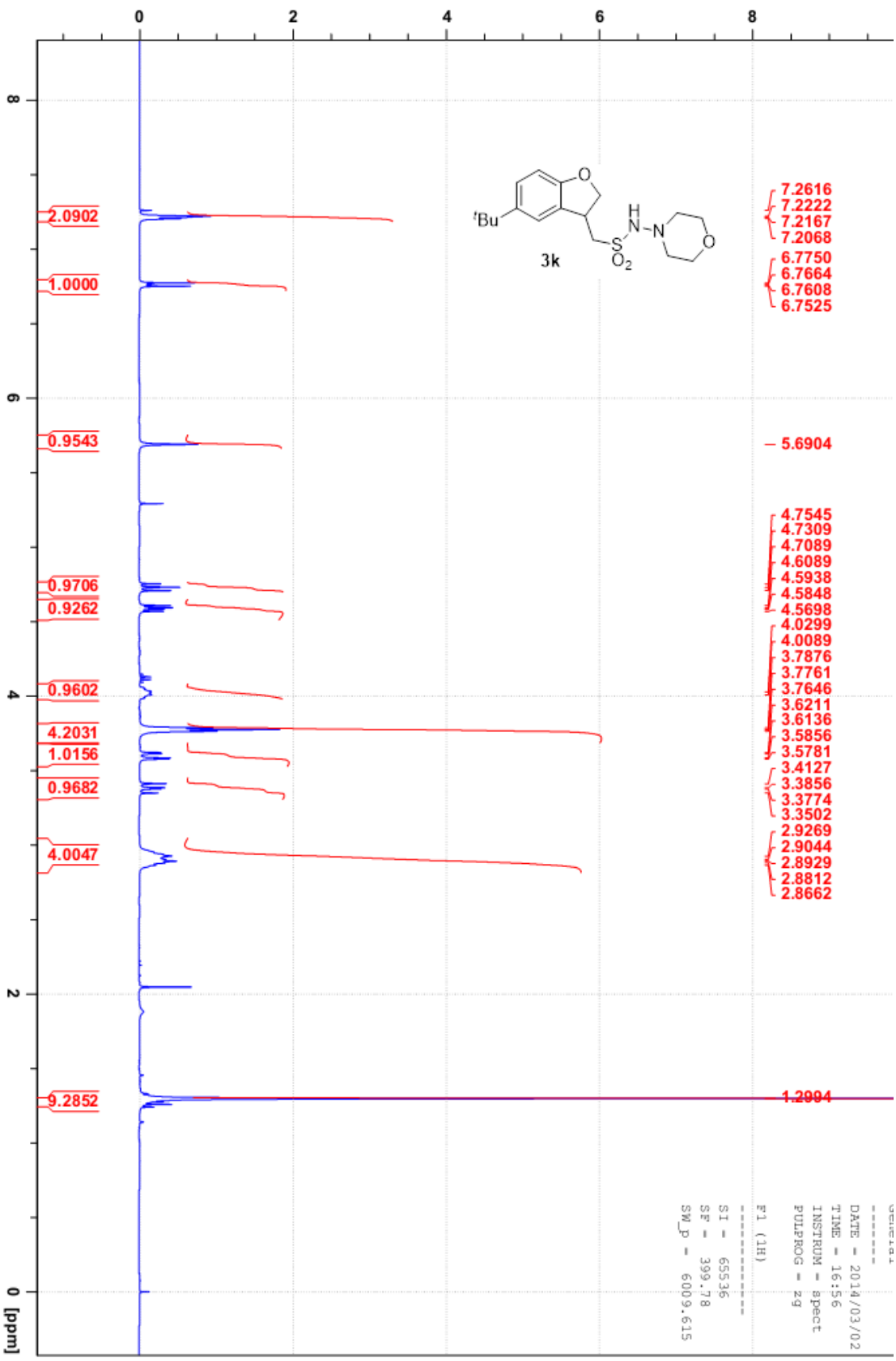
SI = 32768
SF = 100.525
SW_p = 24038.462


3k
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1.0156
0.9682
4.0047
9.2852
7.2616
7.2222
7.2167
7.2068
6.7750
6.7664
6.7608
6.7525
5.6904
4.7545
4.7309
4.7089
4.6089
4.5938
4.5848
4.5698
4.0299
4.0089
3.7876
3.7761
3.7646
3.6211
3.6136
3.5856
3.5781
3.4127
3.3856
3.3774
3.3502
2.9269
2.9044
2.8929
2.8812
2.8662
1.2994
DATE = 2014/03/02
TIME = 16:56
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PULPROG = zg
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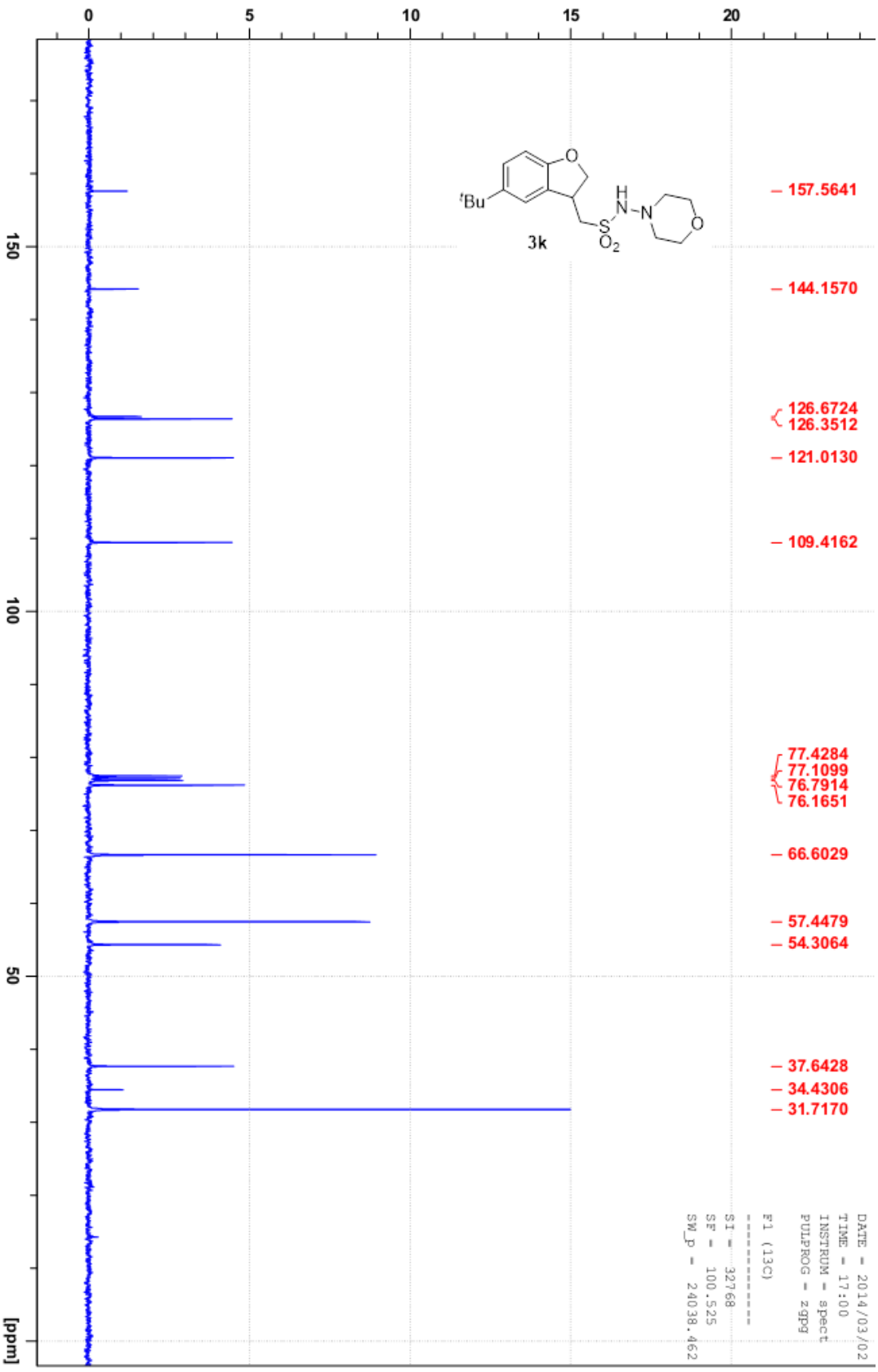
SI = 65536
SF = 399.78
SW_p = 6009.615
[ppm]


0
5
10
15
20
150
100
50
[ppm]
tBu
O
H
N
N
O
S
O2
3k
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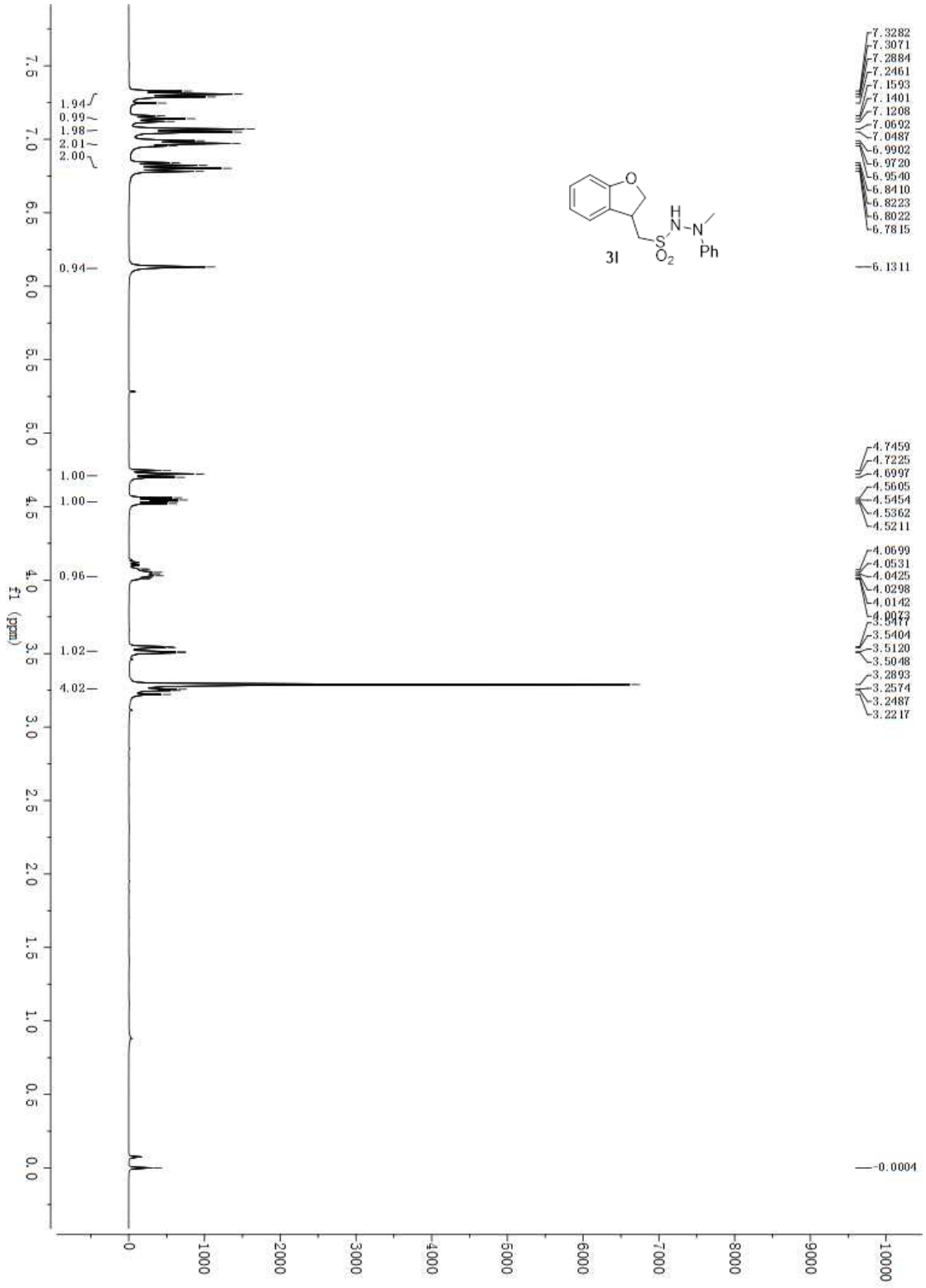
144.1570
126.6724
126.3512
121.0130
109.4162
77.4284
77.1099
76.7914
76.1651
66.6029
57.4479
54.3064
37.6428
34.4306
31.7170
DATE = 2014/03/02
TIME = 17:00
INSTRUM = spect
PULPROG = zgpg
F1 (13C)
SI = 32768
SF = 100.525
SW_p = 24038.462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7.1208
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6.9902
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4.5454
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4.5211
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4.0531
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4.0298
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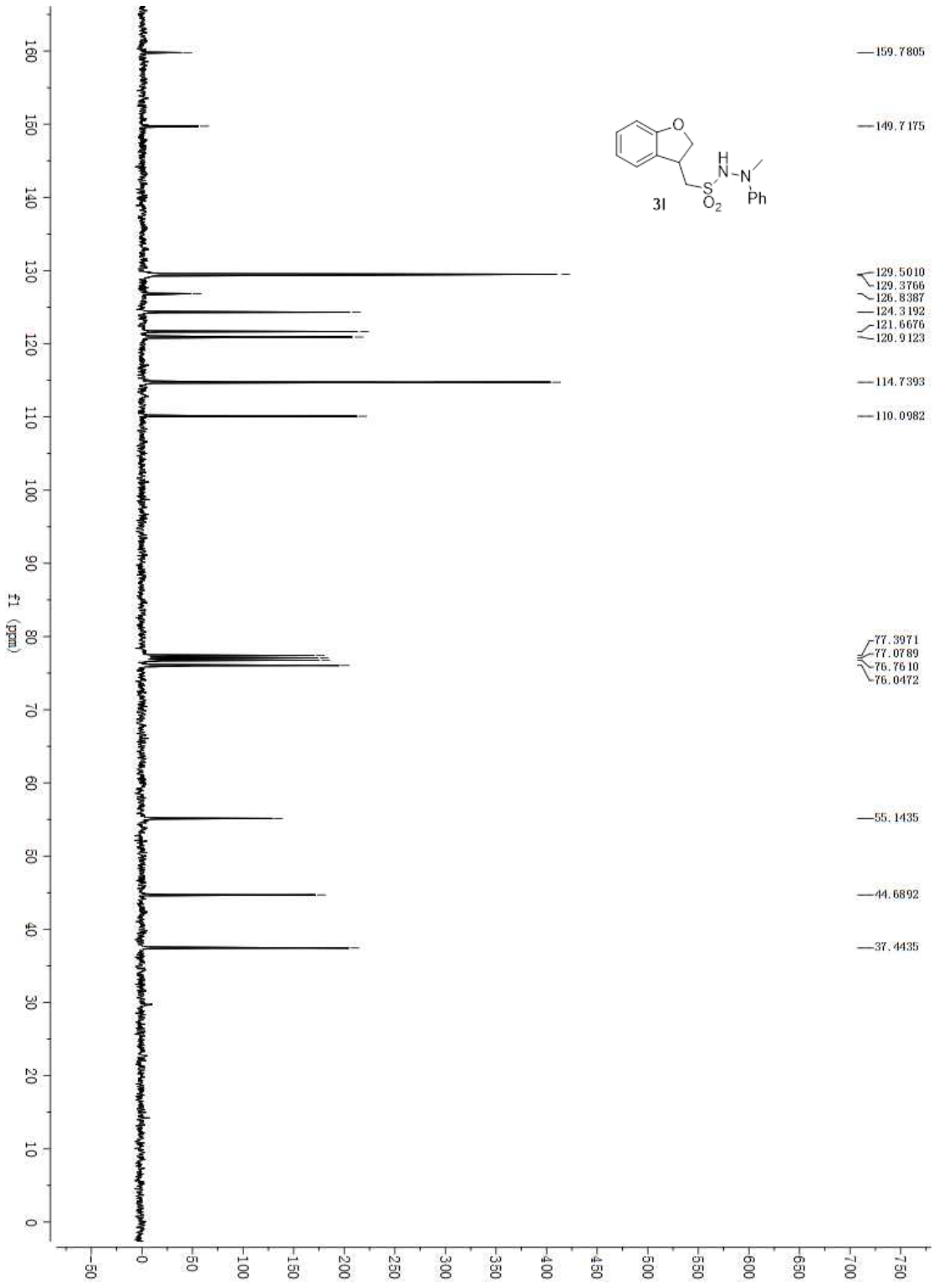
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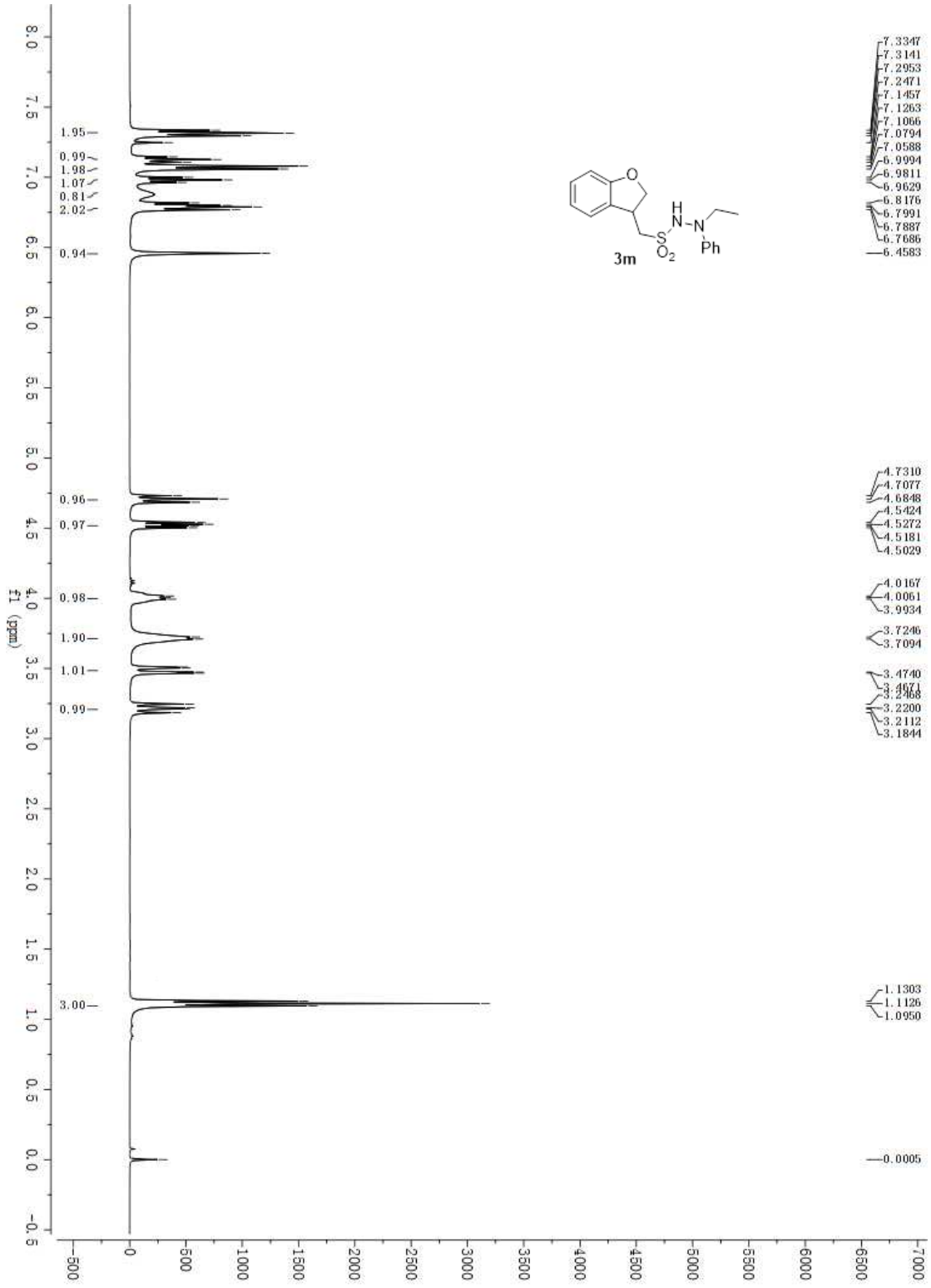
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3.4671
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3.2112
3.1844
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1.1126
1.0950
0.0005
1.95
0.99
1.98
1.07
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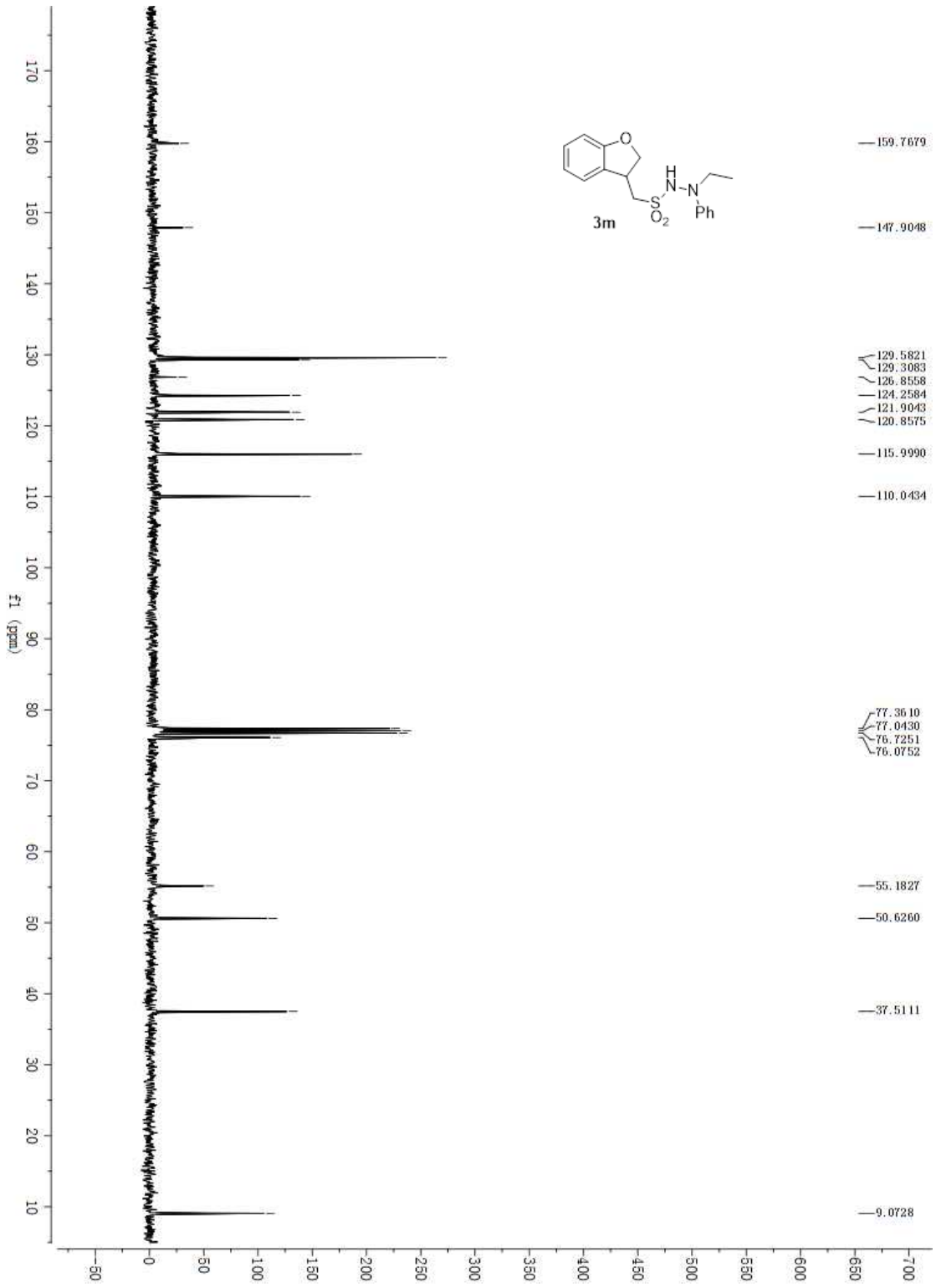
3.00
f1 (ppm)
3m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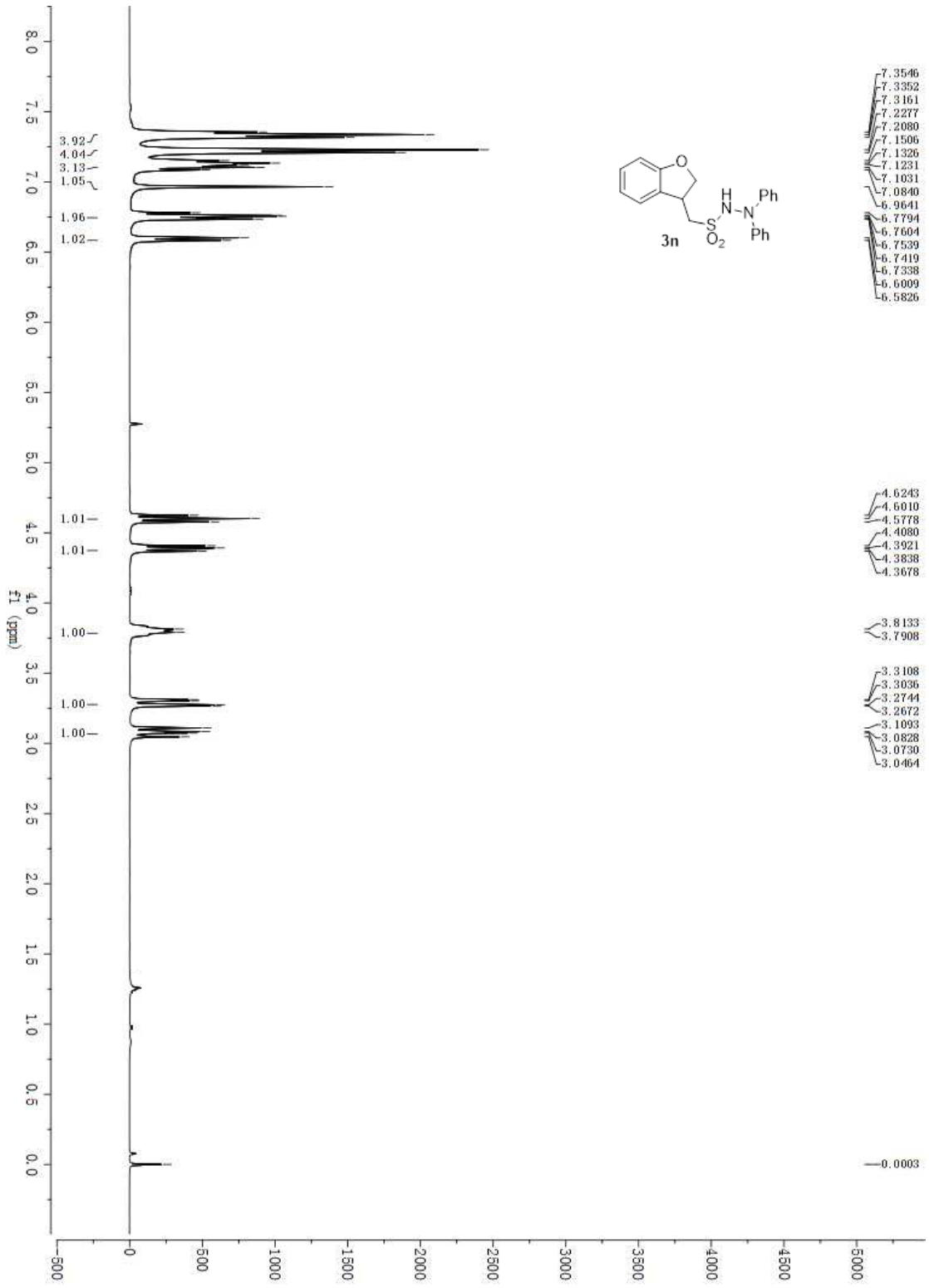
f1 (ppm)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3.2672
3.1093
3.0828
3.0730
3.0464
0.0003
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1.02
1.01
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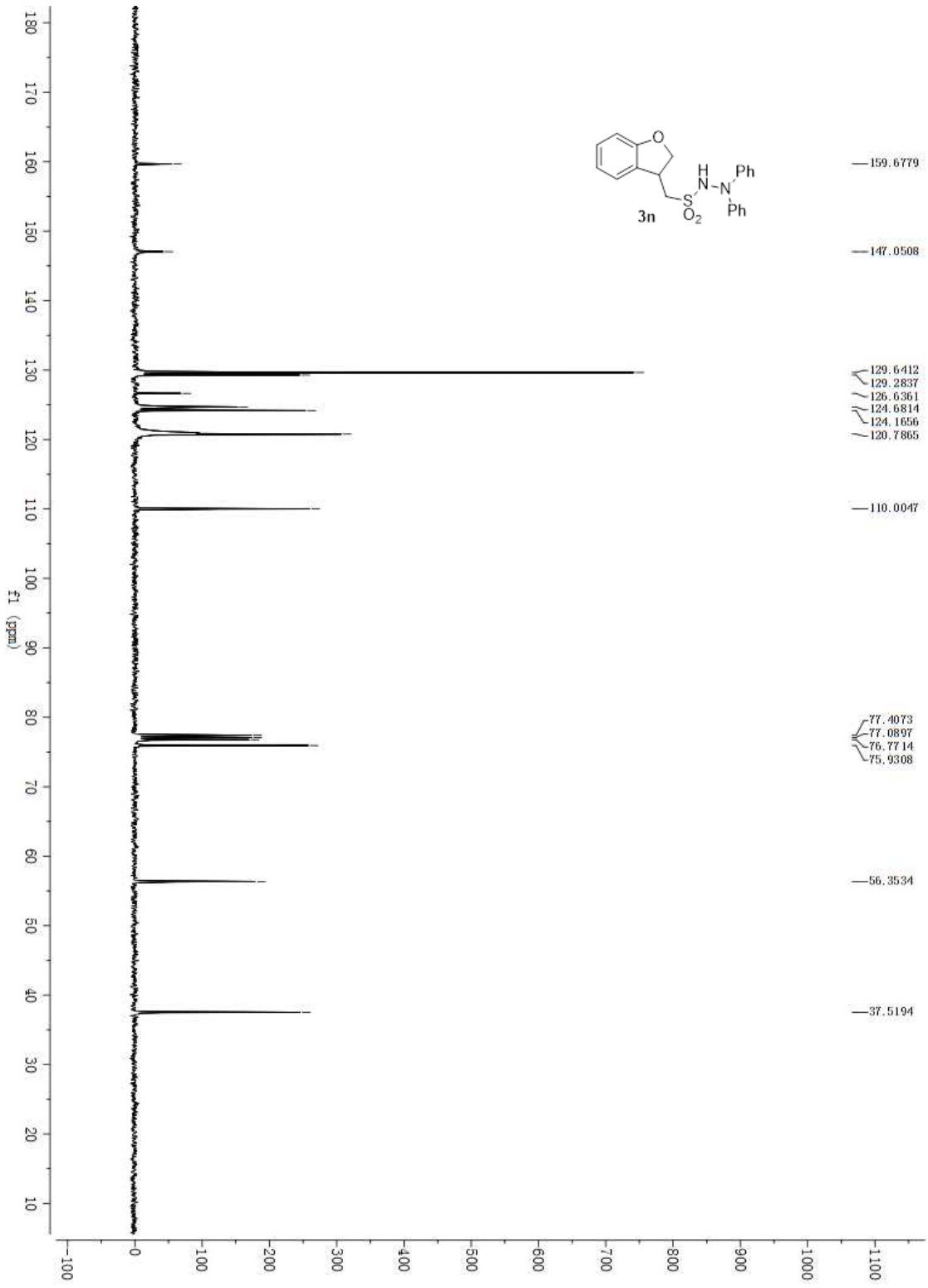
1.00
f1 (ppm)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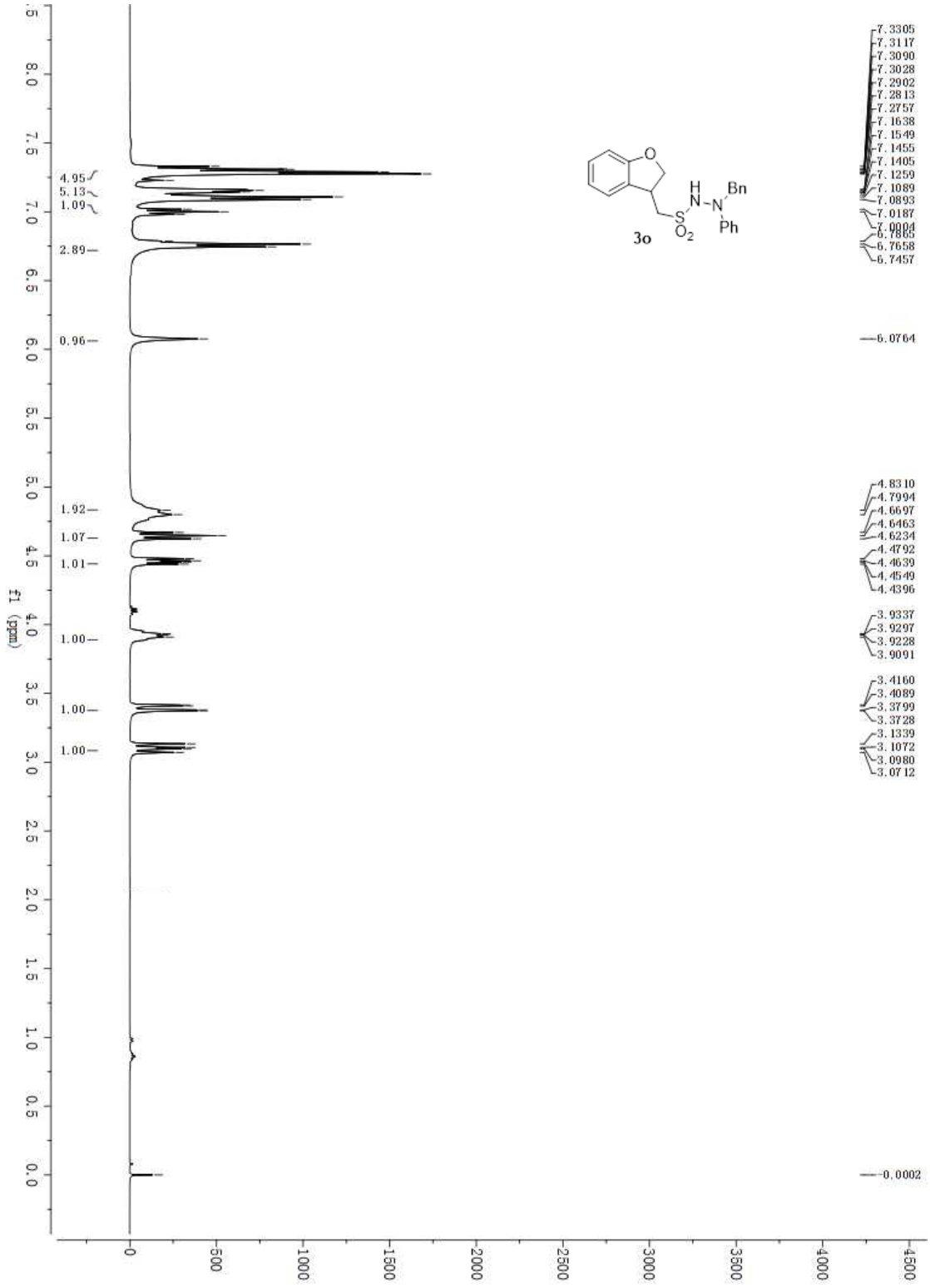
37.5194
f1 (ppm)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7.0893
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7.0004
6.7865
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6.7457
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4.6697
4.6463
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4.4792
4.4639
4.4549
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3.3799
3.3728
3.1339
3.1072
3.0980
3.0712
0.0002
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2.89
0.96
1.92
1.07
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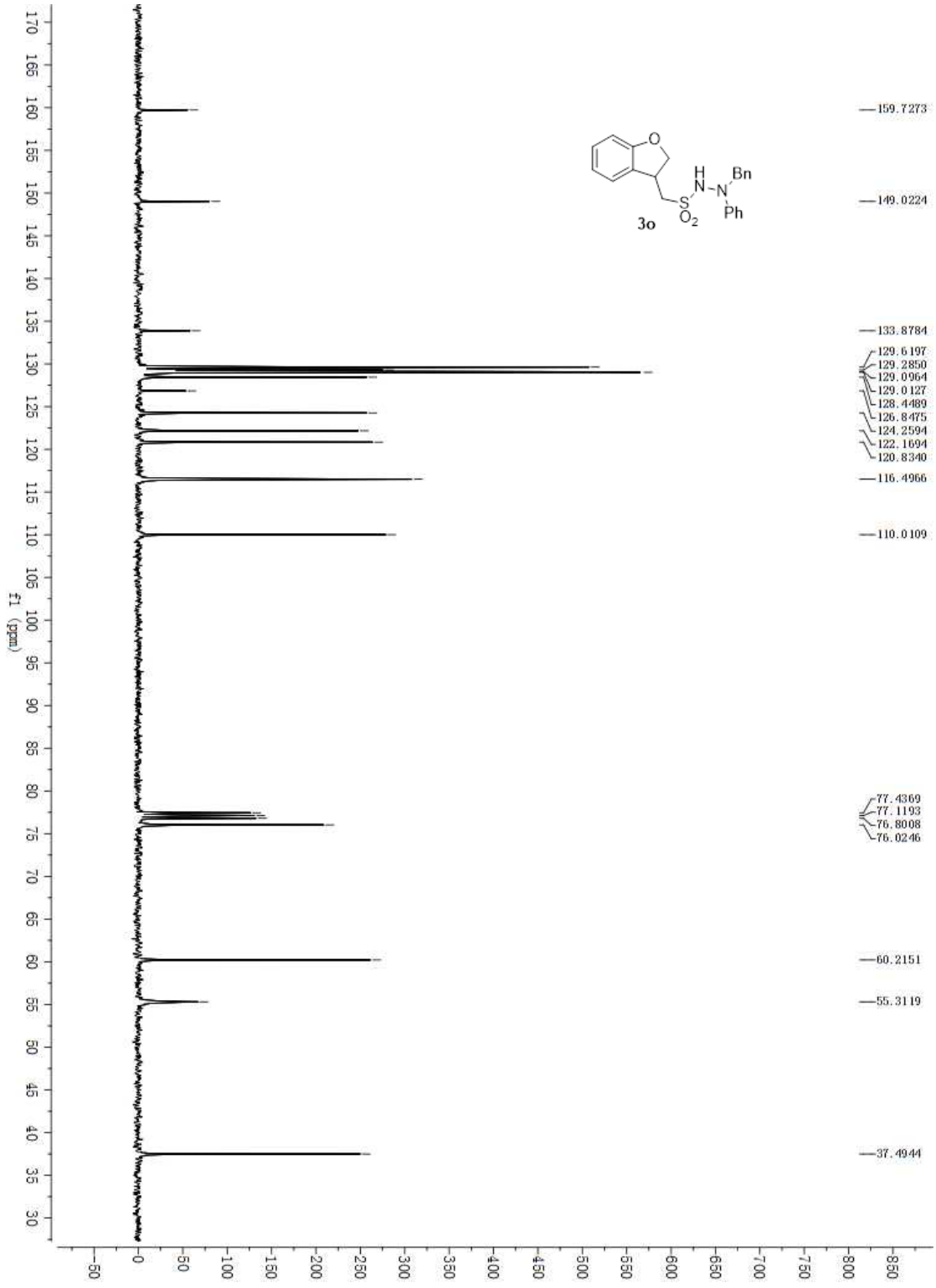
1.00
1.00
1.00
f1 (ppm)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37.4944
3o
f1 (ppm)