Electronic Supplementary Material (ESI) for ChemComm.

C-H Bond sulfonylation of anilines with the insertion of sulfur dioxide under metal-free conditions

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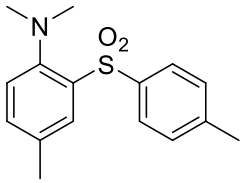
Supporting Information

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.

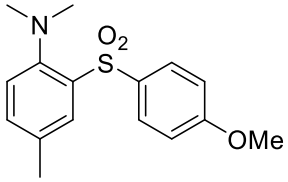
General experimental procedure for the C-H Bond sulfonylation of anilines with the insertion of sulfur dioxide under metal-free conditions

DABCO·$(SO_2)_2$ (0.3 mmol), aryldiazonium tetrafluoroborate **2** (0.3 mmol) were combined in a tube. The tube was evacuated and backfilled with N_2 three times before the addition of MeCN (2.0 mL). The mixture was then placed in oil bath at 65 ^{o}C. Then aniline **1** (0.2 mmol) was added dropwisely during 5 min (if aniline is solid, dissolve it in 0.1 mL of MeCN) and stirred for 8 hours. After the conversion was completed as indicated by TLC, the solvent was evaporated under reduced pressure. The residue was purified directly by flash column chromatography (EtOAc/*n*-hexane, 1:10) to give the corresponding product **3**.



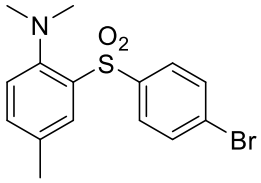
N,N,4-Trimethyl-2-tosylaniline (**3a**)[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.01 (s, 1H), 7.75 (d, *J* = 8.1, 2H), 7.33 – 7.29 (m, 1H), 7.19 (d, *J* = 8.1, 2H), 7.12 (d, *J* = 8.1, 1H), 2.37 (s, 3H), 2.35 (s, 3H), 2.34 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.4, 143.2, 139.7, 138.1, 135.4, 129.9, 129.9, 128.8, 128.4, 124.4, 45.6, 21.8, 21.1. HRMS (ESI) calcd for $C_{16}H_{19}N_1O_2S$: 290.1209 (M + H^+), found: 290.1208.



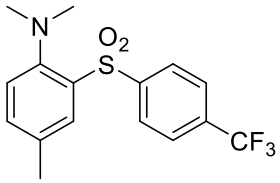
2-((4-Methoxyphenyl)sulfonyl)-*N,N*,4-trimethylaniline (**3b**)[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.01 (s, 1H), 7.82 (d, *J* = 8.9, 2H), 7.31 (d, *J* = 8.0, 1H), 7.13 (d, *J* = 8.1, 1H), 6.88 (d, *J* = 8.9, 2H), 3.82 (s, 3H), 2.38 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 163.0, 151.3, 139.3, 135.4, 134.3, 130.8, 129.8, 126.8, 124.4, 114.0, 113.4, 55.8, 45.8, 21.1. HRMS (ESI) calcd for $C_{16}H_{19}N_1O_3S$: 306.1158 (M + H^+), found: 306.1150.]



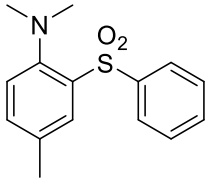
2-((4-Bromophenyl)sulfonyl)-*N,N*,4-trimethylaniline (**3c**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.00 (s, 1H), 7.75 – 7.70 (m, 2H), 7.57 – 7.53 (m, 2H), 7.35 (d, *J* = 8.1, 1H), 7.16 -7.12 (m, 1H), 2.38 (s, 3H), 2.35 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.3, 141.7, 137.4, 135.9, 135.7, 131.4, 129.9, 129.9, 127.6, 124.5, 45.6, 21.2. HRMS (ESI) calcd for $C_{15}H_{16}BrN_1O_2S$: 354.0158 (M + H^+), found: 354.0156.



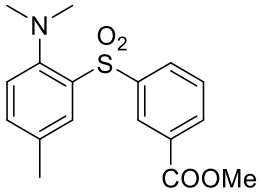
N,N,4-Trimethyl-2-((4-(trifluoromethyl)phenyl)sulfonyl)aniline (**3d**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.04 (s, 1H), 7.98 (d, *J* = 8.2, 2H), 7.68 (d, *J* = 8.3, 2H), 7.38 (d, *J* = 8.1, 1H), 7.15 (d, *J* = 8.1, 1H), 2.41 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.3, 146.3, 137.1, 136.2, 135.9, 135.1 (d, J = 216 Hz), 130.1 (d, *J* = 3.9 Hz), 128.7, 124.9 (d, *J* = 69 Hz), 45.5, 21.1. HRMS (ESI) calcd for $C_{16}H_{16}F_3N_1O_2S$: 344.0927 ($M + H^+$), found: 344.0929.



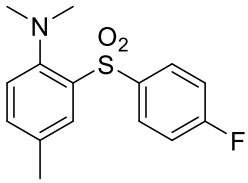
N,N,4-Trimethyl-2-(phenylsulfonyl)aniline (**3e**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.04 (d, *J* = 1.6, 1H), 7.88 – 7.84 (m, 2H), 7.51 – 7.46 (m, 1H), 7.43 - 7.38 (m, 2H), 7.36 – 7.31 (m, 1H), 7.13 (d, *J* = 8.1, 1H), 2.39 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.4, 142.7, 137.9, 135.5, 132.5, 129.9, 128.2, 124.5, 109.9, 45.4, 21.1. HRMS (ESI) calcd for $C_{15}H_{17}N_1O_2S$: 276.1053 ($M + H^+$), found: 276.1050.



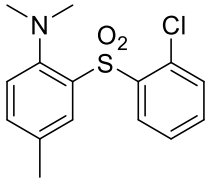
Methyl 3-((2-(dimethylamino)-5-methylphenyl)sulfonyl)benzoate (**3f**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.54 (s, 1H), 8.18 – 8.14 (m, 1H), 8.11 – 8.06 (m, 1H), 8.03 (s, 1H), 7.51 (t, *J* = 7.8, 1H), 7.37 – 7.32 (m, 1H), 7.13 (d, *J* = 8.1, 1H), 3.90 (s, 3H), 2.40 (s, 3H), 2.33 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 166.0, 151.2, 143.2, 137.4, 135.8, 133.5, 132.6, 130.5, 130.0, 129.7, 128.4, 124.6, 52.8, 45.6, 21.2. HRMS (ESI) calcd for $C_{17}H_{19}N_1O_4S$: 334.1108 ($M + H^+$), found: 334.1118.



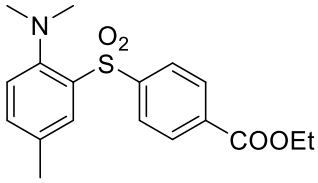
2-((4-Fluorophenyl)sulfonyl)-*N,N*,4-trimethylaniline (**3g**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.01 (s, 1H), 7.91 – 7.86 (m, 2H), 7.36 – 7.31 (m, 1H), 7.14 (d, *J* = 8.1 Hz, 1H), 7.11 – 7.05 (m, 2H), 2.38 (s, 3H), 2.35 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 165.2 (d, *J* = 252 Hz), 151.3, 138.6, 137.7, 135.7, 131.3, 130.5 (d, *J* = 126 Hz), 124.5, 115.4 (d, *J* = 15.4 Hz), 45.7, 45.6, 21.1. HRMS (ESI) calcd for $C_{15}H_{16}FN_1O_2S$: 294.0959 ($M + H^+$), found: 294.0959.



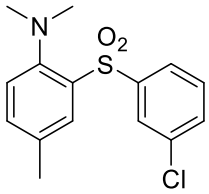
2-((2-Chlorophenyl)sulfonyl)-*N,N*,4-trimethylaniline (**3h**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.32 – 8.27 (m, 1H), 8.05 (d, *J* = 1.8 Hz, 1H), 7.46 – 7.40 (m, 2H), 7.38 – 7.35 (m, 1H), 7.34 – 7.31 (m, 1H), 7.15 (d, *J* = 8.1 Hz, 1H), 2.41 (s, 3H), 2.20 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 135.8, 135.7, 133.4, 131.6, 131.2, 130.7, 126.5, 124.4, 45.1, 21.2. HRMS (ESI) calcd for $C_{15}H_{16}ClN_1O_2S$: 310.0663 ($M + H^+$), found: 310.0663.



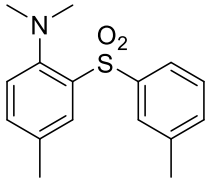
Ethyl 4-((2-(dimethylamino)-5-methylphenyl)sulfonyl)benzoate (**3i**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.07 (d, *J* = 8.5 Hz, 2H), 8.03 (d, *J* = 1.4, 1H), 7.90 (d, *J* = 8.6 Hz, 2H), 7.38 – 7.33 (m, 1H), 7.13 (d, *J* = 8.1 Hz, 1H), 4.36 (q, *J* = 7.1 Hz, 2H), 2.40 (s, 3H), 2.30 (s, 6H), 1.37 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 165.6, 151.3, 146.7, 137.3, 136.0, 135.8, 133.9, 130.1, 129.4, 128.1, 124.6, 61.8, 45.4, 21.2, 14.5. HRMS (ESI) calcd for $C_{18}H_{21}N_1O_4S$: 348.1264 ($M + H^+$), found: 348.1265.



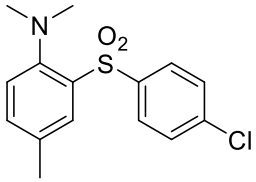
2-((3-Chlorophenyl)sulfonyl)-*N,N*,4-trimethylaniline (**3j**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.00 (d, *J* = 1.6 Hz, 1H), 7.89 (t, *J* = 1.8 Hz, 1H), 7.75 – 7.71 (m, 1H), 7.47 – 7.43 (m, 1H), 7.38 – 7.32 (m, 2H), 7.15 (d, *J* = 8.1 Hz, 1H), 2.38 (s, 3H), 2.35 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.3, 144.3, 137.2, 136.0, 135.8, 134.2, 132.6, 130.0, 129.5, 128.5, 126.4, 124.6, 45.4, 21.1. HRMS (ESI) calcd for $C_{15}H_{16}ClN_1O_2S$: 310.0663 (M + H^+), found: 310.0692.



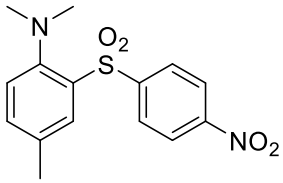
N,N,4-Trimethyl-2-(m-tolylsulfonyl)aniline (**3k**)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.02 (d, *J* = 1.6 Hz, 1H), 7.70 – 7.65 (m, 2H), 7.35 - 7.31 (m, 1H), 7.31 – 7.27 (m, 2H), 7.14 (d, *J* = 8.1 Hz, 1H), 2.39 (s, 3H), 2.36 (s, 3H), 2.34 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.4, 142.5, 138.3, 138.0, 135.6, 133.4, 130.0, 128.5, 128.0, 125.5, 124.6, 45.6, 21.2. HRMS (ESI) calcd for $C_{16}H_{19}N_1O_2S$: 290.1209 (M + H^+), found: 290.1206.



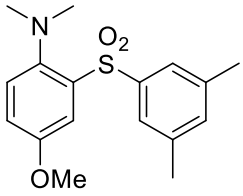
2-((4-Chlorophenyl)sulfonyl)-*N,N*,4-trimethylaniline (**3l**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.01 (s, 1H), 7.81 (d, *J* = 8.5 Hz, 2H), 7.41 – 7.33 (m, 3H), 7.14 (d, *J* = 8.1 Hz, 1H), 2.39 (s, 3H), 2.36 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.3, 139.1, 135.9, 135.7, 130.0, 129.9, 129.9, 128.4, 124.6, 120.2, 45.6, 21.2. HRMS (ESI) calcd for $C_{15}H_{16}ClN_1O_2S$: 332.0482 (M + Na^+), found: 332.0487.



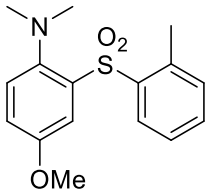
N,N,4-Trimethyl-2-((4-nitrophenyl)sulfonyl)aniline (**3m**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.26 (d, *J* = 8.7 Hz, 2H), 8.02 (d, *J* = 8.7 Hz, 3H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.16 (d, *J* = 8.1 Hz, 1H), 2.42 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.2, 150.0, 148.6, 136.5, 136.2, 130.2, 129.3, 124.6, 123.4, 109.9, 45.5, 30.8. HRMS (ESI) calcd for $C_{15}H_{16}N_2O_4S$: 343.0723 (M + Na^+), found: 343.0722.



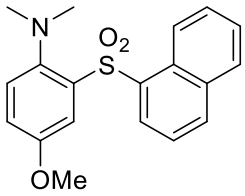
2-((3,5-Dimethylphenyl)sulfonyl)-4-methoxy-*N,N*-dimethylaniline (**3n**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.72 (d, *J* = 3.0 Hz, 1H), 7.50 (s, 2H), 7.19 (d, *J* = 8.7 Hz, 1H), 7.11 (s, 1H), 7.08 – 7.04 (m, 1H), 3.85 (s, 3H), 2.32 (s, 6H), 2.30 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.0, 146.6, 142.1, 139.6, 138.1, 134.3, 126.0, 125.9, 121.4, 113.2, 113.2, 56.0, 45.5, 21.3. HRMS (ESI) calcd for $C_{17}H_{21}N_1O_3S$: 320.1315 (M + H^+), found: 320.1314.



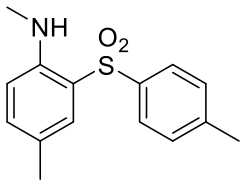
4-Methoxy-*N,N*-dimethyl-2-(*o*-tolylsulfonyl)aniline (**3o**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.17 – 8.13 (m, 1H), 7.73 (d, *J* = 3.0 Hz, 1H), 7.40 – 7.29 (m, 2H), 7.19 (d, *J* = 8.7 Hz, 1H), 7.14 – 7.05 (m, 2H), 3.85 (s, 3H), 2.25 (s, 3H), 2.15 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.0, 146.4, 140.6, 139.5, 136.9, 132.6, 131.8, 130.4, 126.1, 125.6, 121.5, 113.4, 56.2, 45.1, 20.0. HRMS (ESI) calcd for $C_{16}H_{19}N_1O_3S$: 306.1158 (M + H^+), found: 306.1163.



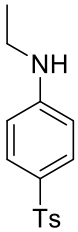
4-Methoxy-*N,N*-dimethyl-2-(naphthalen-1-ylsulfonyl)aniline (**3p**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.48 (d, *J* = 7.4 Hz, 1H), 8.28 (d, *J* = 7.5 Hz, 1H), 8.01 (d, *J* = 8.1 Hz, 1H), 7.92 (d, *J* = 2.7 Hz, 1H), 7.87 – 7.81 (m, 1H), 7.59 (t, *J* = 7.8 Hz, 1H), 7.48 – 7.38 (m, 2H), 7.11 – 7.02 (m, 2H), 3.91 (s, 3H), 2.02 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.2, 146.4, 140.1, 137.3, 134.0, 133.9, 130.4, 128.9, 128.6, 128.0, 126.1, 124.2, 121.4, 112.9, 112.8, 56.3, 45.0. HRMS (ESI) calcd for $C_{119}H_{19}N_1O_3S$: 342.1158 ($M + H^+$), found: 342.1174.



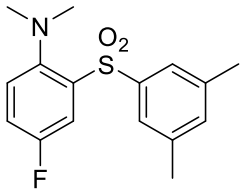
N,4-Dimethyl-2-tosylaniline (**3q**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.75 (d, *J* = 8.3 Hz, 2H), 7.64 (s, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 1H), 6.54 (d, *J* = 8.5 Hz, 1H), 6.13 (s, 1H), 2.79 (d, *J* = 4.7 Hz, 3H), 2.36 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 146.1, 143.9, 139.4, 136.4, 130.1, 129.8, 127.0, 125.4, 112.1, 109.9, 30.4, 21.8. HRMS (ESI) calcd for $C_{15}H_{17}N_1O_2S$: 298.0872 ($M + Na^+$), found: 298.0862.



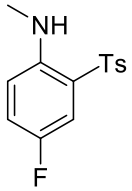
N-Ethyl-4-tosylaniline (**3r'**)

^{1}H NMR (400 MHz, $cdcl_3$) δ (ppm) 7.81 (d, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 6.63 (d, *J* = 8.8 Hz, 2H), 4.03 (s, 1H), 3.22 (q, *J* = 7.1 Hz, 2H), 2.40 (s, 3H), 1.27 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 151.4, 151.0, 144.8, 140.0, 129.8, 127.9, 125.3, 122.4, 112.3, 38.4, 21.6, 15.0. HRMS (ESI) calcd for $C_{15}H_{17}N_1O_2S$: 276.1053 ($M + H^+$), found: 276.1056.



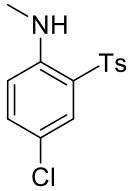
2-((3,5-Dimethylphenyl)sulfonyl)-4-fluoro-*N,N*-dimethylaniline (**3s**)

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.18 (s, 1H), 7.14 (s, 2H), 7.08 (s, 1H), 6.93 (s, 2H), 2.29 (s, 6H), 2.24 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 142.9, 139.1 (d, *J* = 31.8 Hz), 135.2, 134.5, 134.3 (d, *J* = 202 Hz), 133.2, 127.6, 125.5, 125.4, 109.9, 40.9, 21.3. HRMS (ESI) calcd for $C_{16}H_{18}F_1N_1O_2S$: 308.1115 ($M + H^+$), found: 308.1112.



4-Fluoro-*N*-methyl-2-tosylaniline (**3t**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.43 (s, 1H), 7.23 (s, 1H), 7.21 (d, *J* = 2.7 Hz, 2H), 7.18 (s, 1H), 7.13 (s, 1H), 7.11 (s, 1H), 2.40 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 143.5 (d, *J* = 253 Hz), 140.6, 136.7, 130.4, 129.5, 127.8, 124.8, 123.8, 21.9, 21.7.

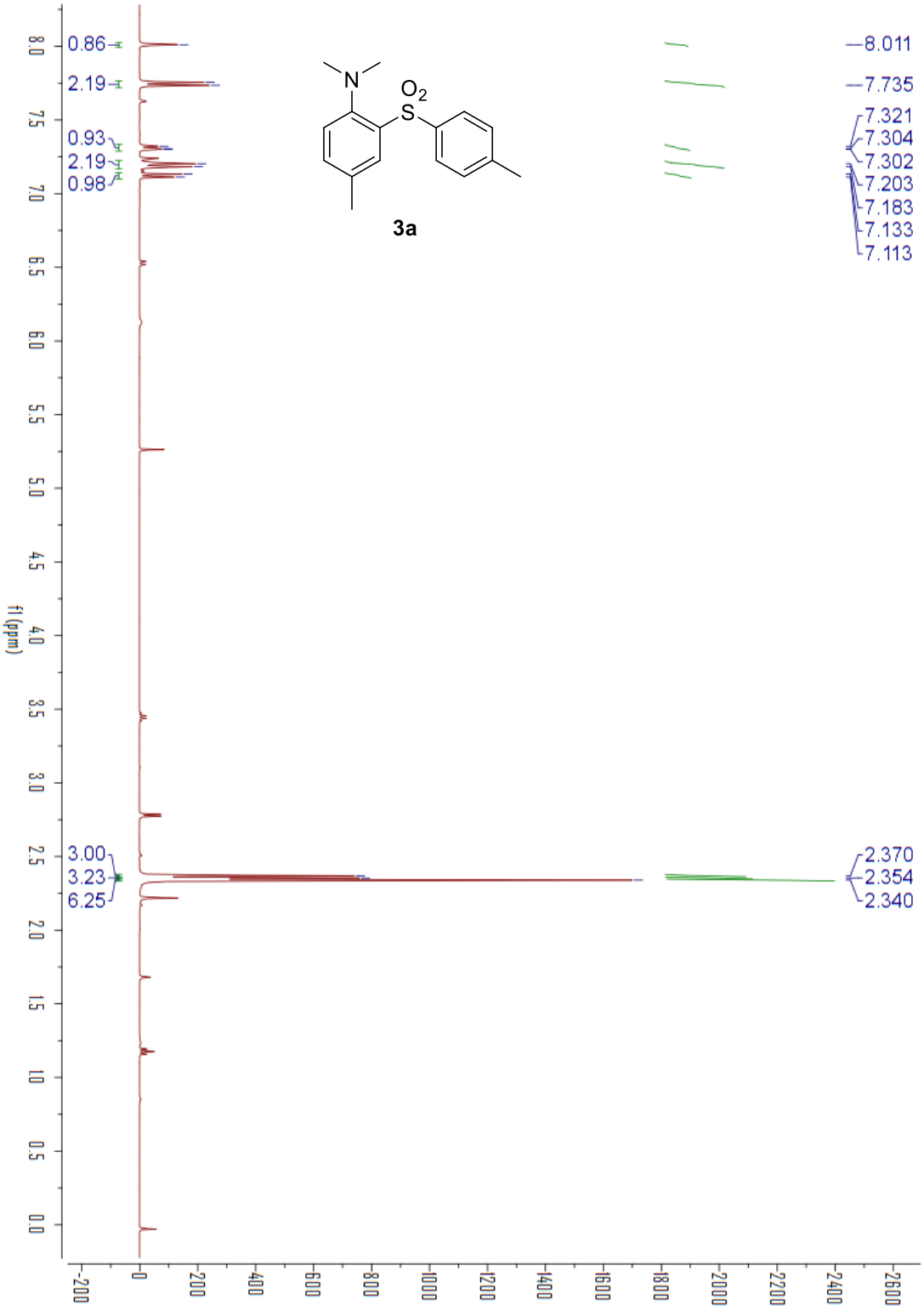


4-Chloro-*N*-methyl-2-tosylaniline (**3u**)

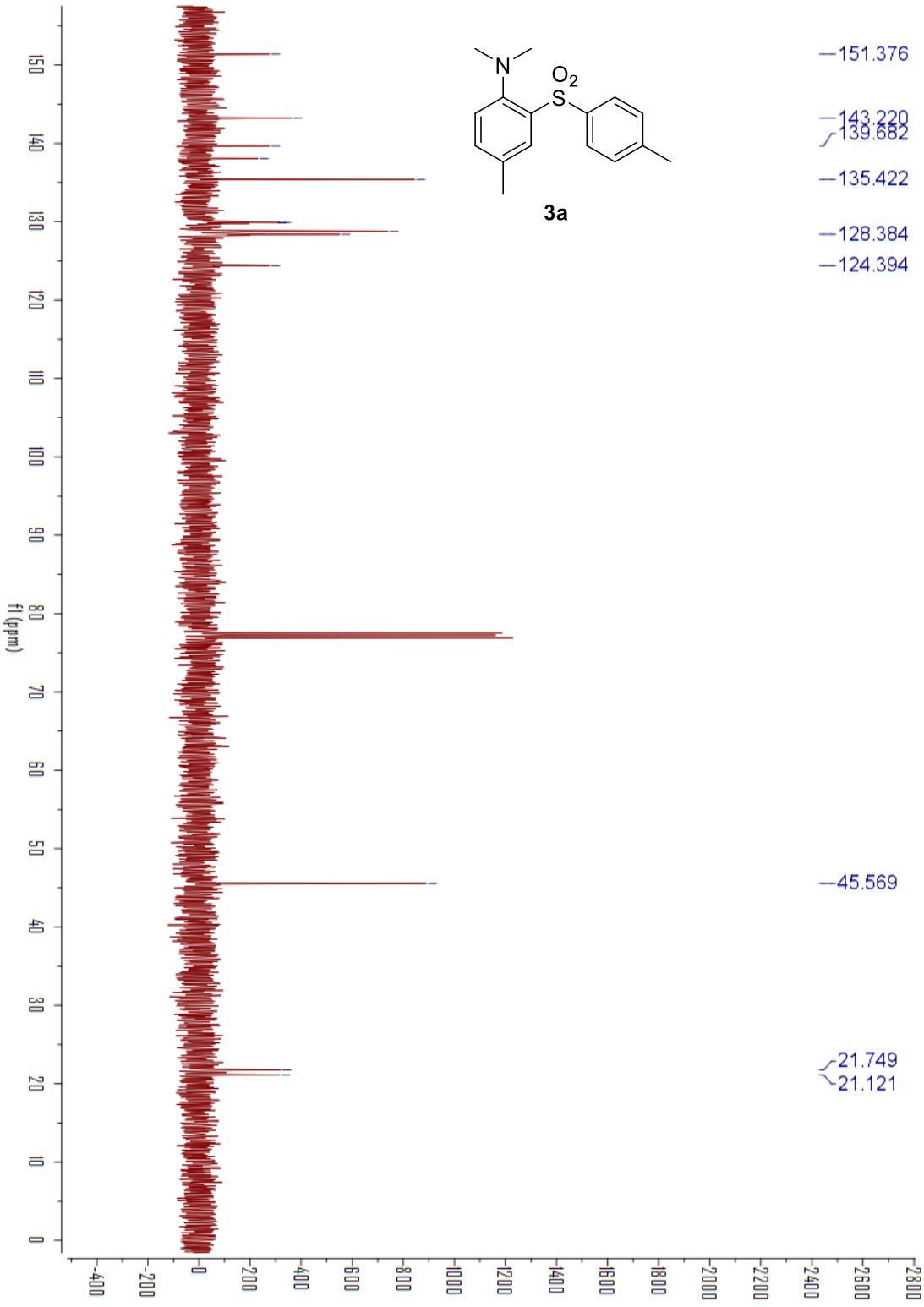
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.43 (s, 1H), 7.23 (s, 1H), 7.21 (d, *J* = 2.4 Hz, 2H), 7.18 (s, 1H), 7.13 (s, 1H), 7.11 (s, 1H), 2.40 (s, 3H), 2.36 (s, 3H), ^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 144.8, 142.3, 136.7, 130.4, 129.5, 127.8, 124.8, 109.9, 29.6, 22.0.

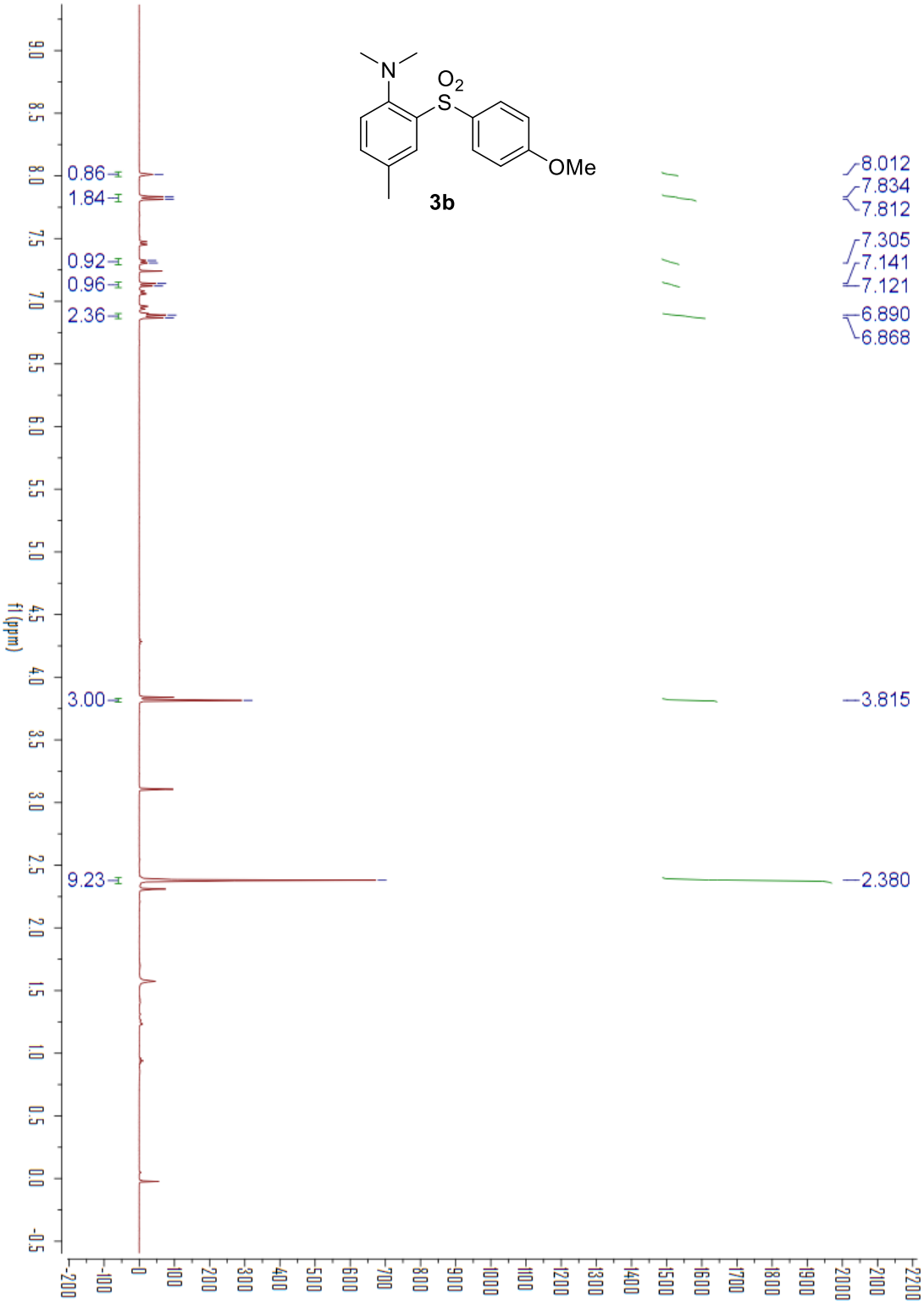
Reference：

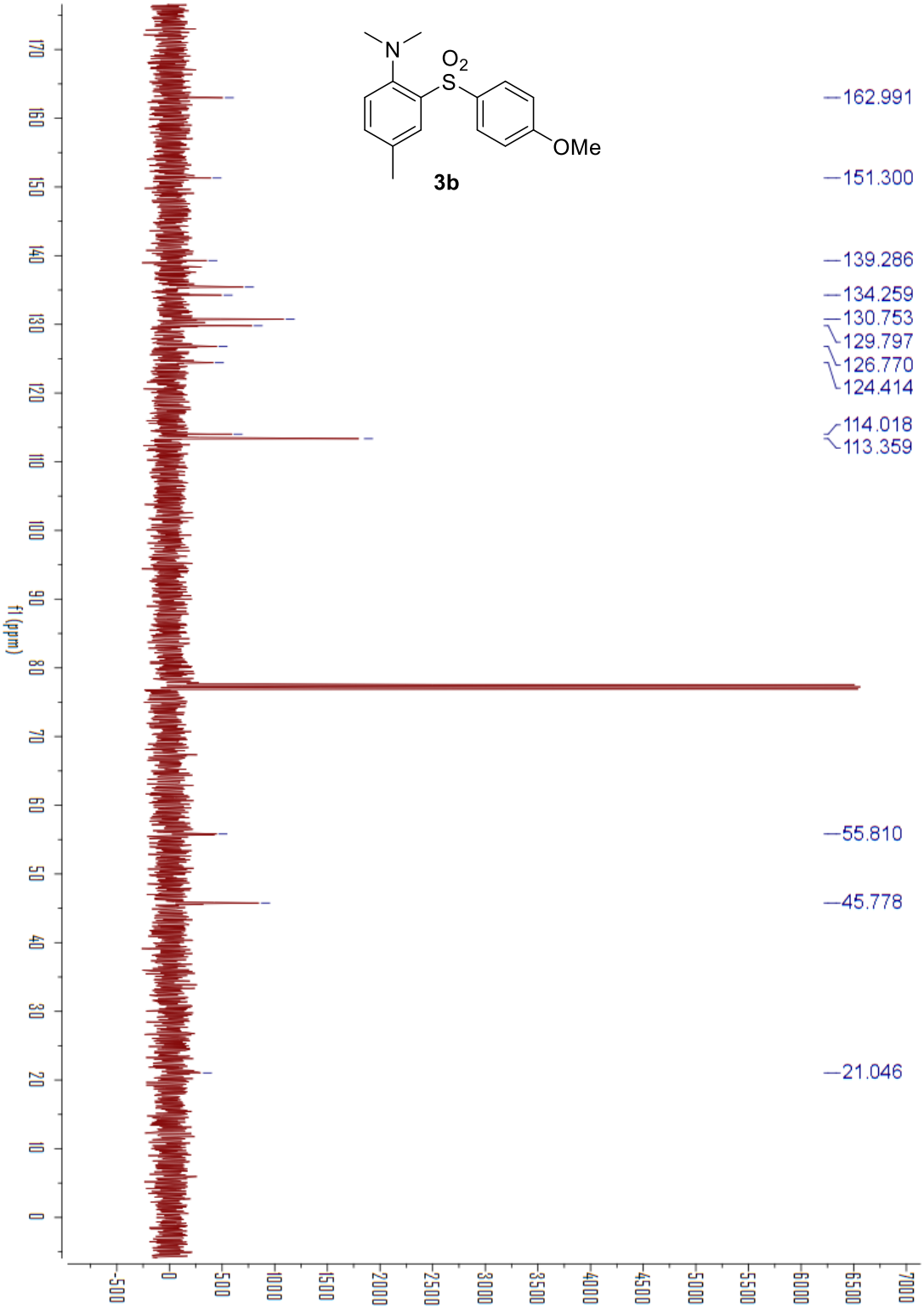
(1) T. C. Johnson, B. L. Elbert, A. J. Farley, T. W. Gorman, C. Genicot, B. Lallemand, P. Pasau, J. Flasz, J. L. Castro, M. MacCoss, D. J. Dixon, R. S. Paton, C. J. Schofield, M. D. Smith and M. C. Willis, *Chem. Sci.*, 2018, **9**, 629.

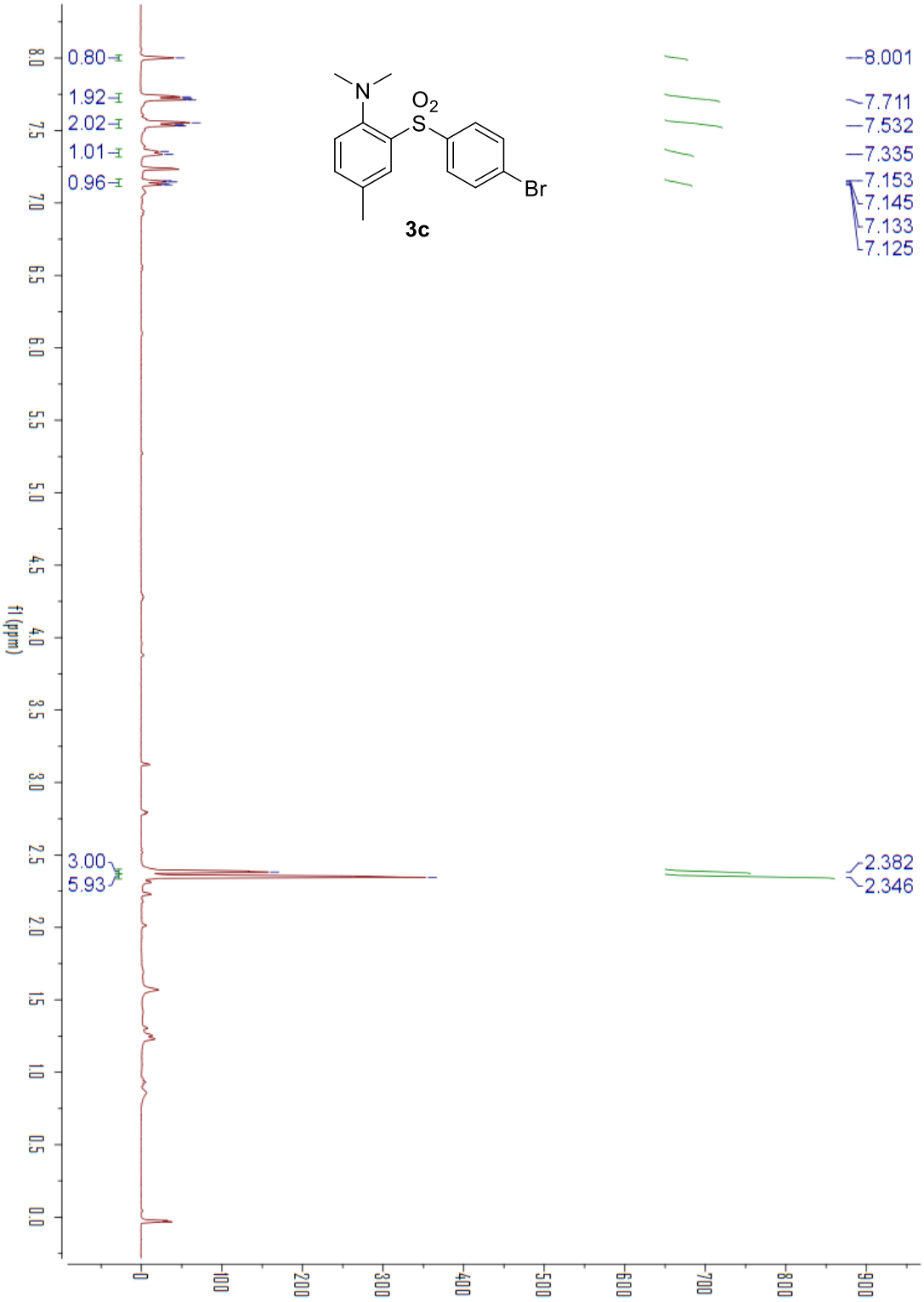

0.86
2.19
0.93
2.19
0.98
3.00
3.23
6.25
8.011
7.735
7.321
7.304
7.302
7.203
7.183
7.133
7.113
2.370
2.354
2.340
f1 (ppm)
N
O2
S

3a


151.376
143.220
139.682
135.422
128.384
124.394
45.569
21.749
21.121
3a
f1 (ppm)


N
O2
S
OMe
3b
0.86
1.84
0.92
0.96
2.36
3.00
9.23
8.012
7.834
7.812
7.305
7.141
7.121
6.890
6.868
3.815
2.380
f1 (ppm)


N
O2
S
OMe
3b
162.991
151.300
139.286
134.259
130.753
129.797
126.770
124.414
114.018
113.359
55.810
45.778
21.046
f1 (ppm)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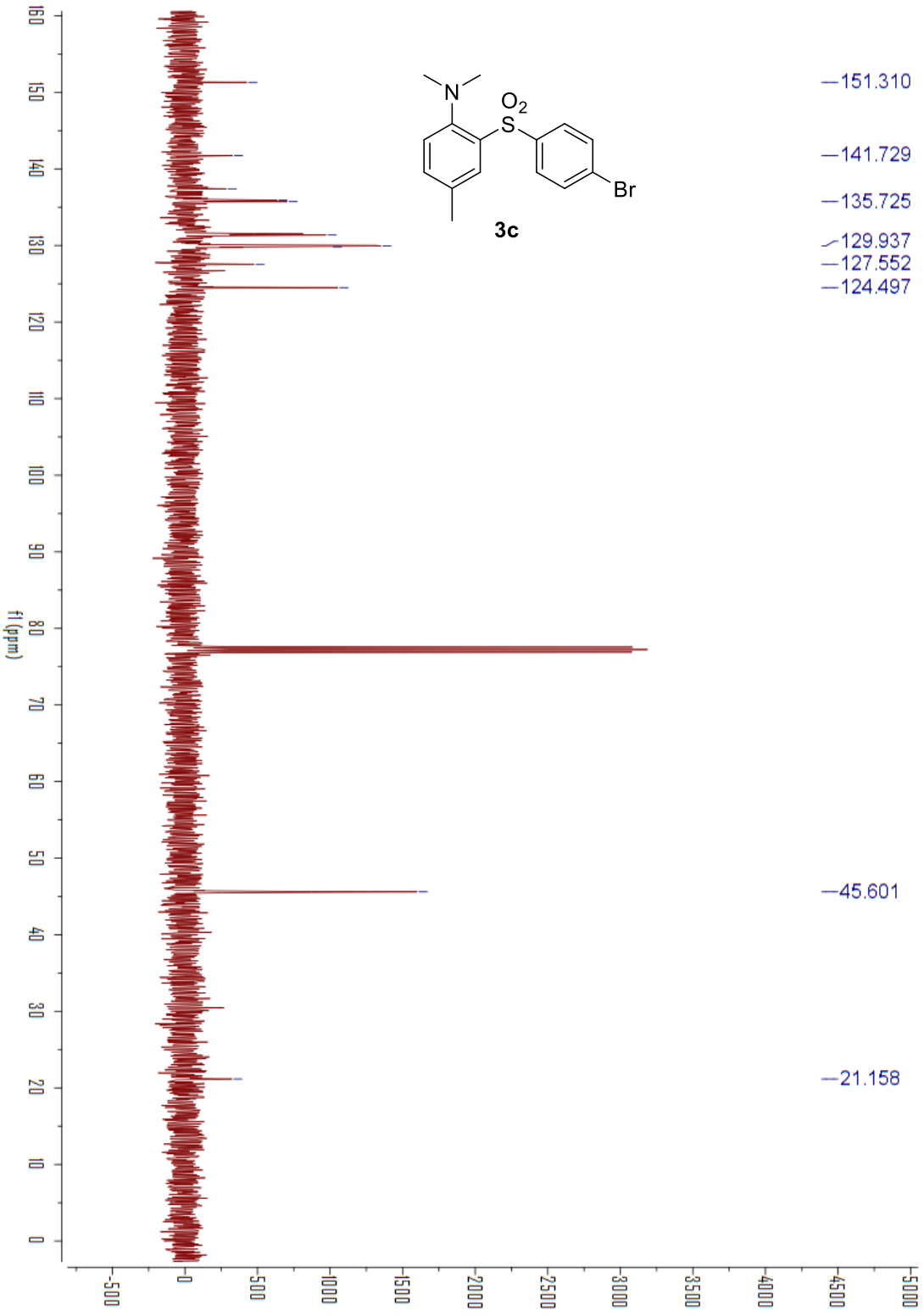
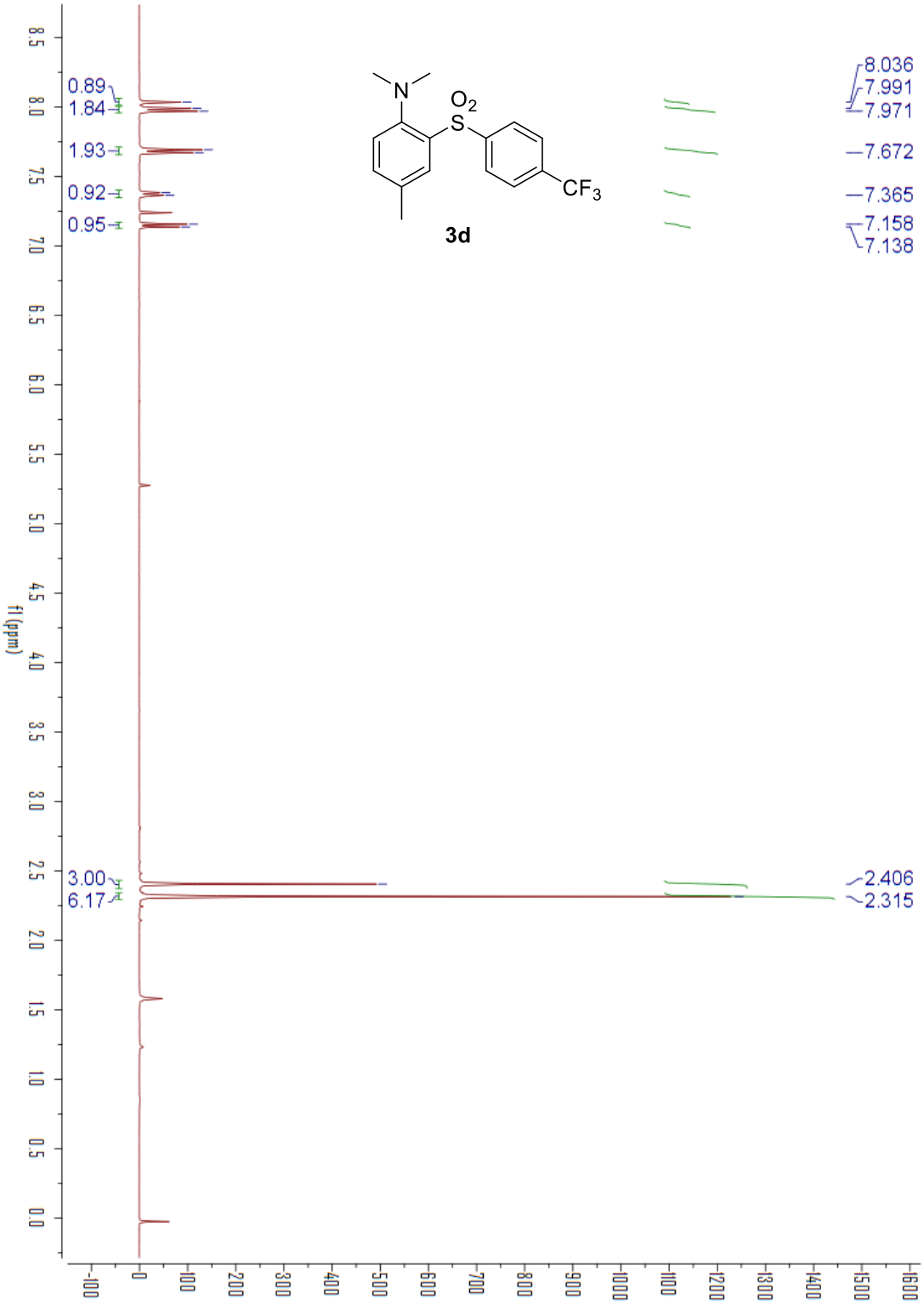
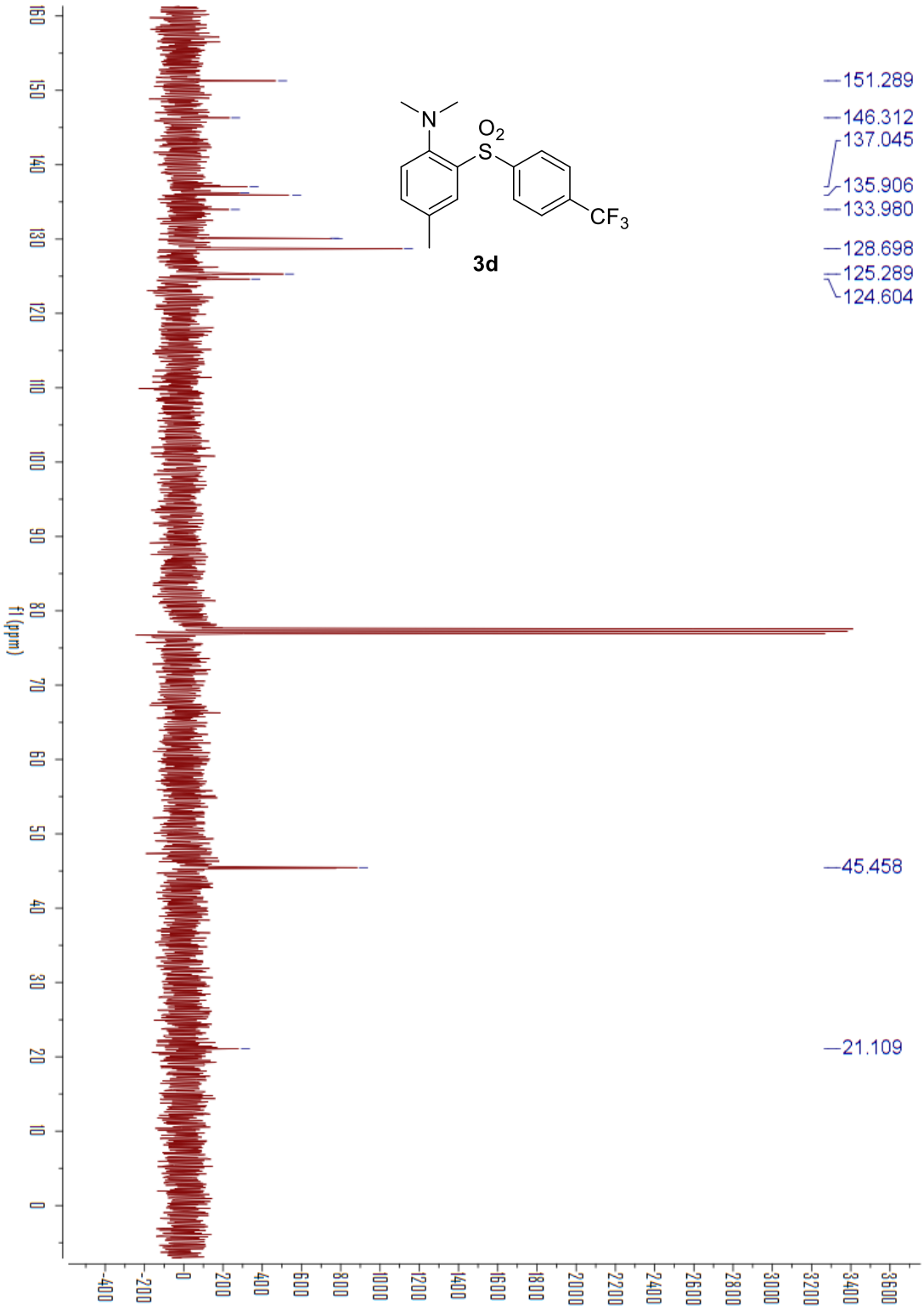
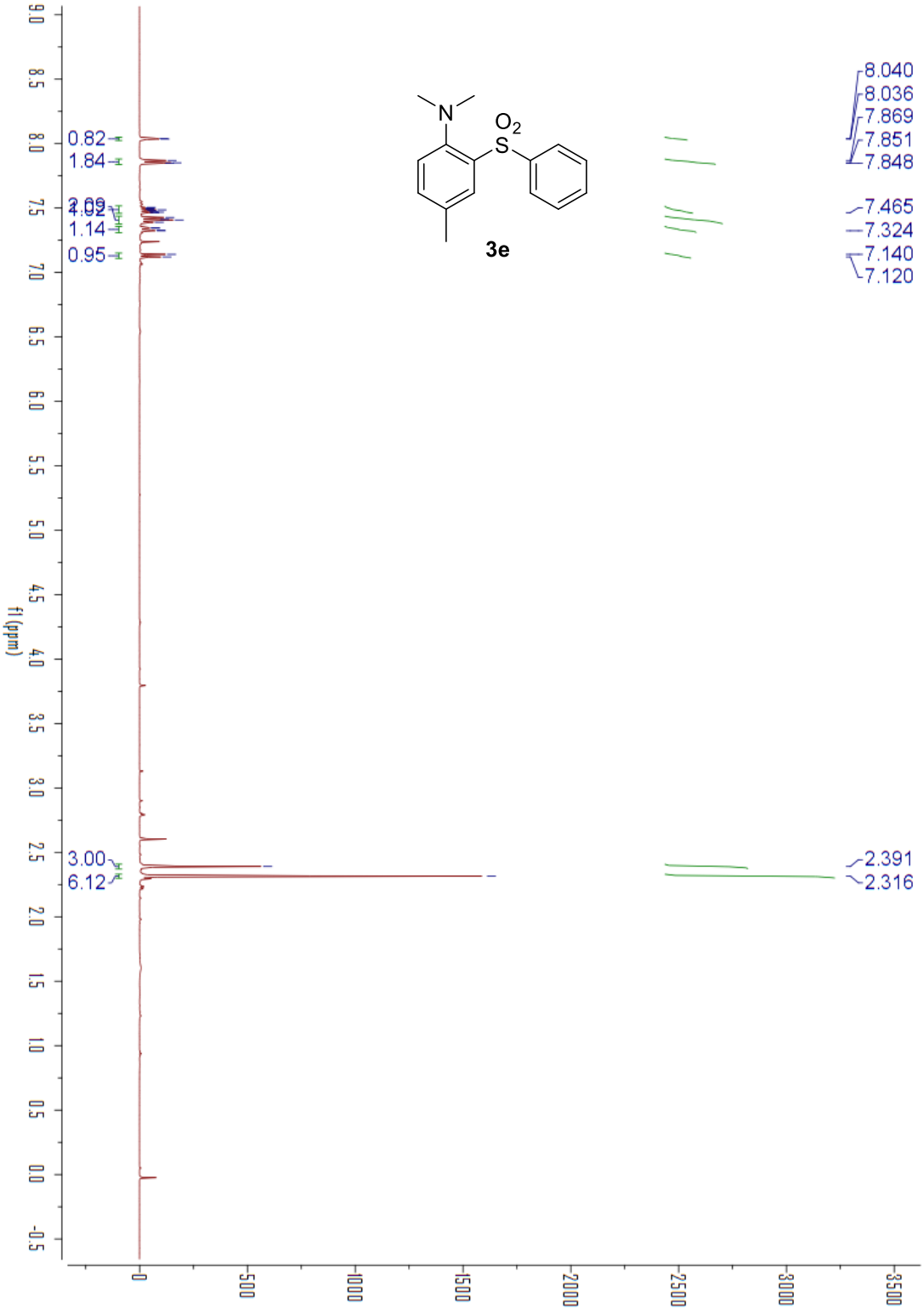
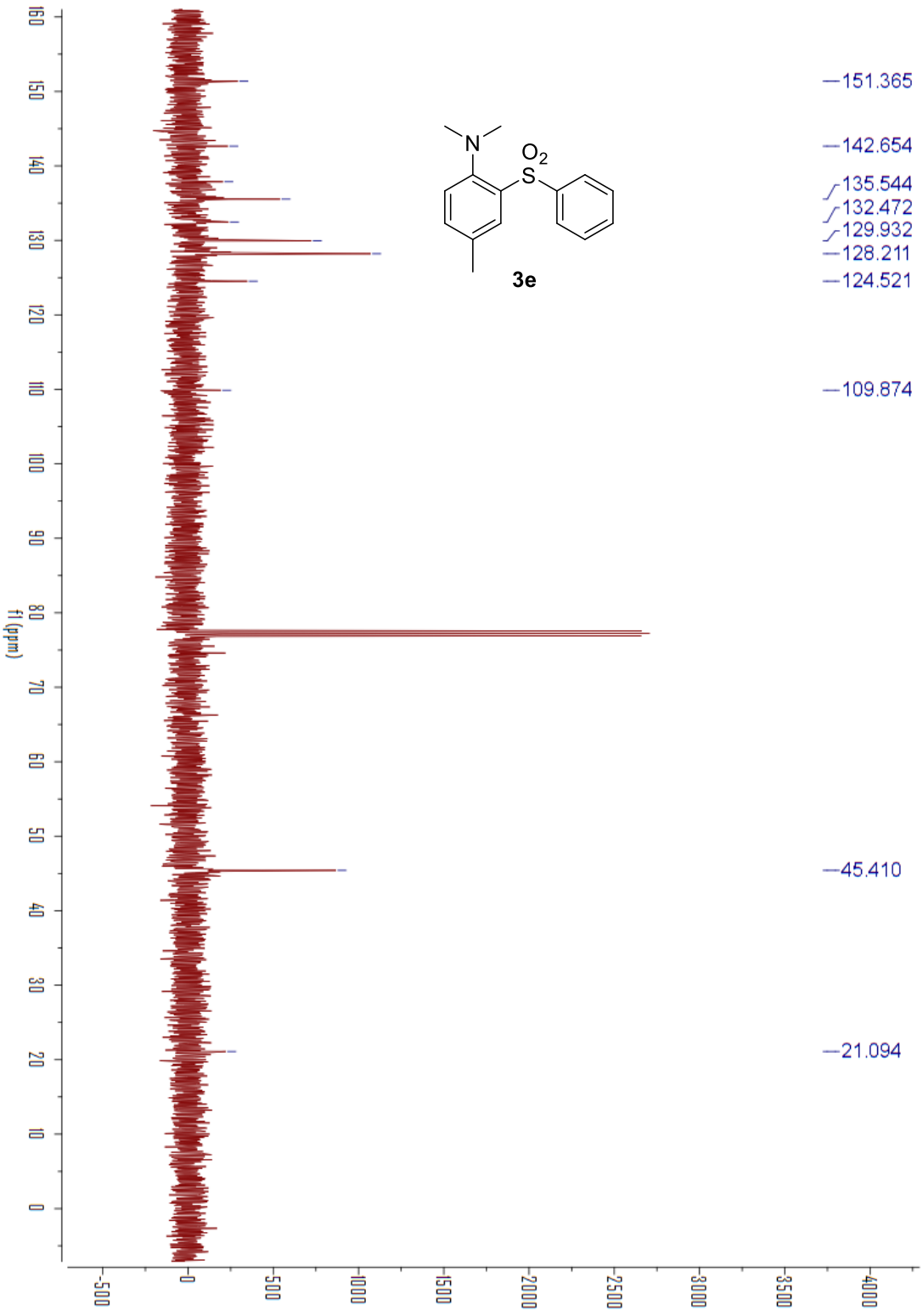
1.92
2.02
1.01
0.96
3.00
5.93
2.382
2.346
8.001
7.711
7.532
7.335
7.153
7.145
7.133
7.125
N
O2
S
Br
3c
f1 (ppm)


N
O2
S
Br
3c
151.310
141.729
135.725
129.937
127.552
124.497
45.601
21.158
f1 (ppm)


0.89
1.84
1.93
0.92
0.95
3.00
6.17
8.036
7.991
7.971
7.672
7.365
7.158
7.138
2.406
2.315
N
O2
S
CF3
3d
f1 (ppm)


151.289
146.312
137.045
135.906
133.980
128.698
125.289
124.604
45.458
21.109
f1 (ppm)
3d


3e
8.040
8.036
7.869
7.851
7.848
7.465
7.324
7.140
7.120
2.391
2.316
0.82
1.84
2.09
4.92
1.14
0.95
3.00
6.12
f1 (ppm)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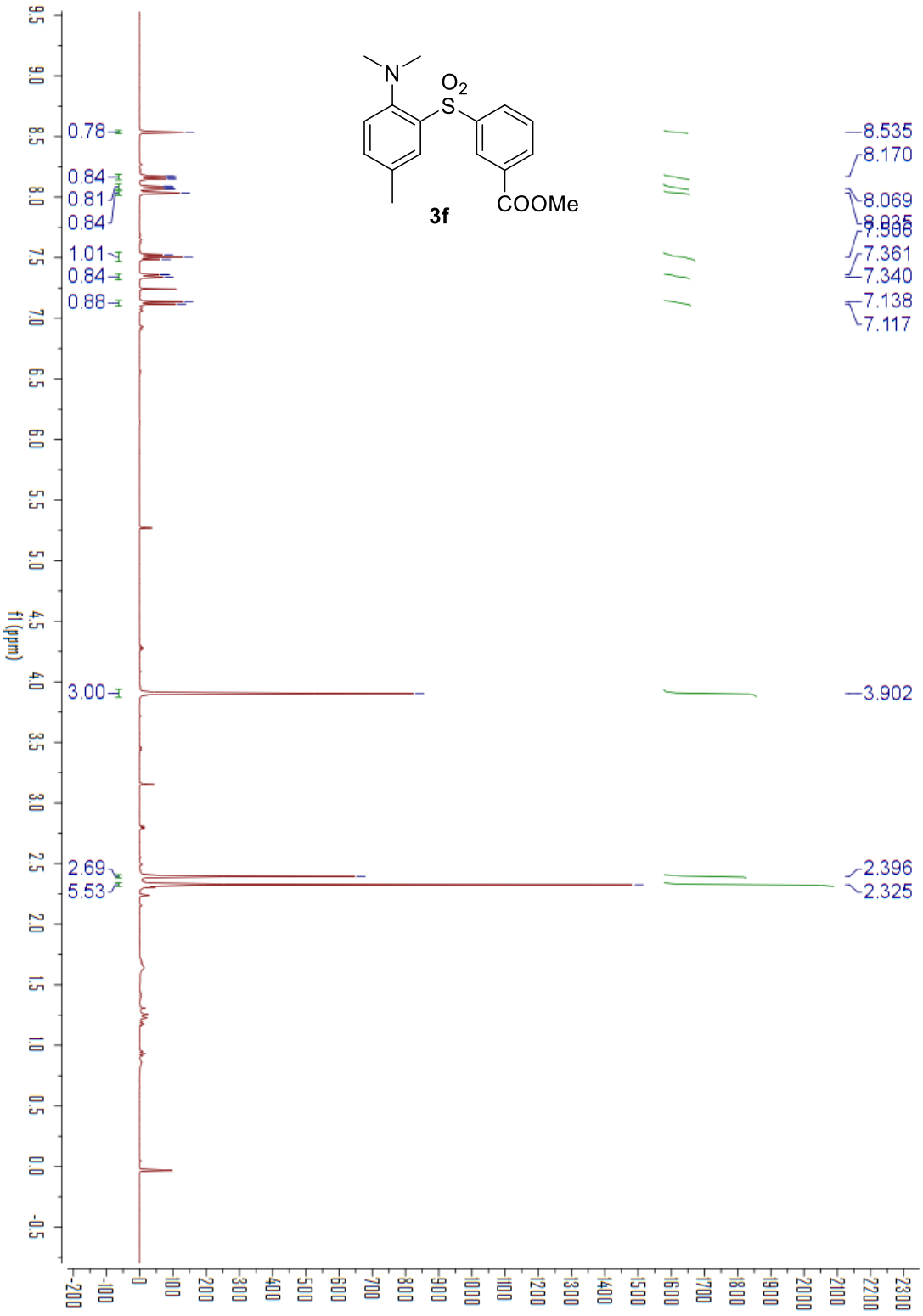
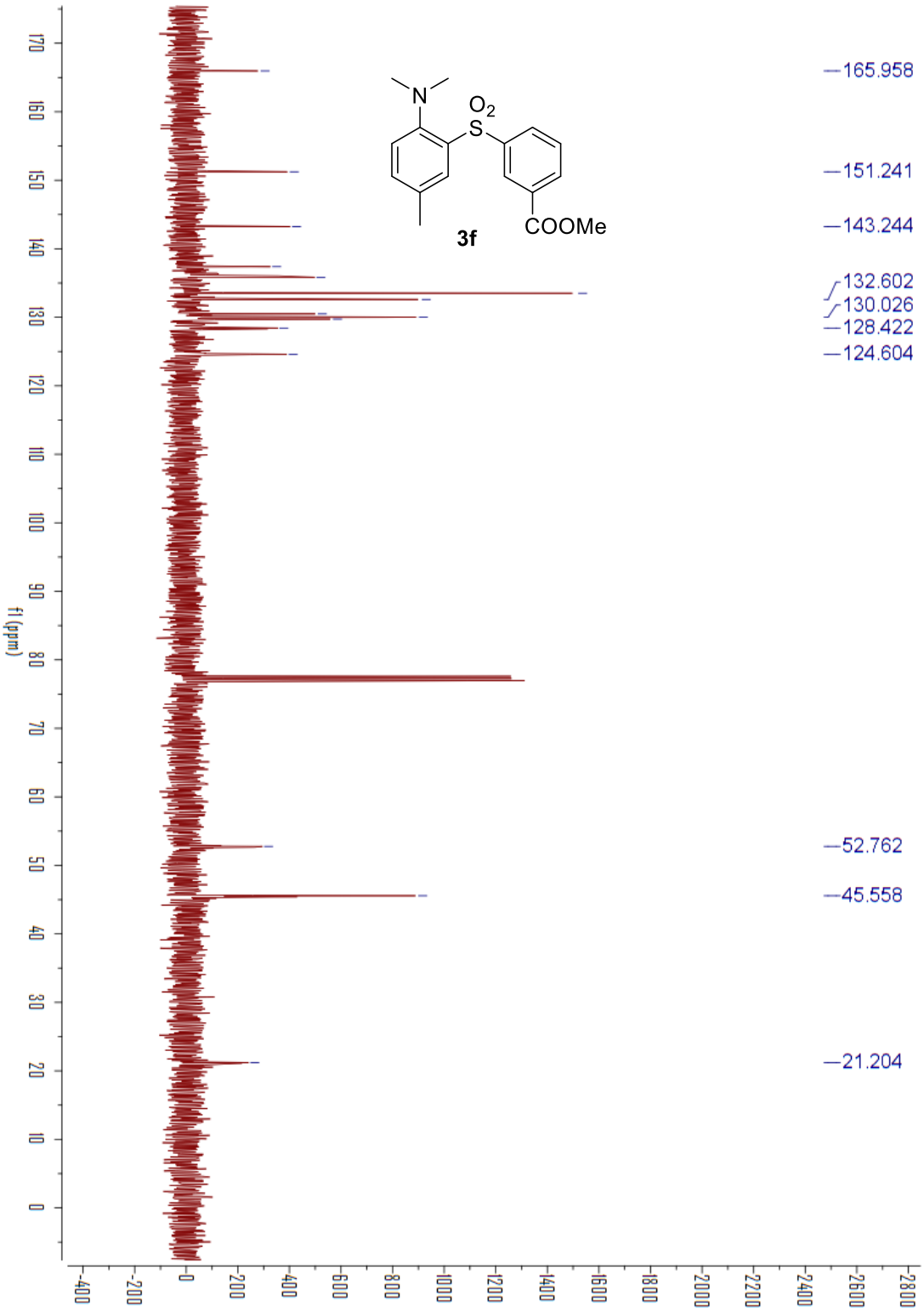
142.654
135.544
132.472
129.932
128.211
124.521
109.874
45.410
21.094
f1 (ppm)
3e


N
O2
S
COOMe
3f
0.78
0.84
0.81
0.84
1.01
0.84
0.88
3.00
2.69
5.53
8.535
8.170
8.069
7.361
7.340
7.138
7.117
3.902
2.396
2.325
f1 (ppm)


N
O2
S
COOMe
3f
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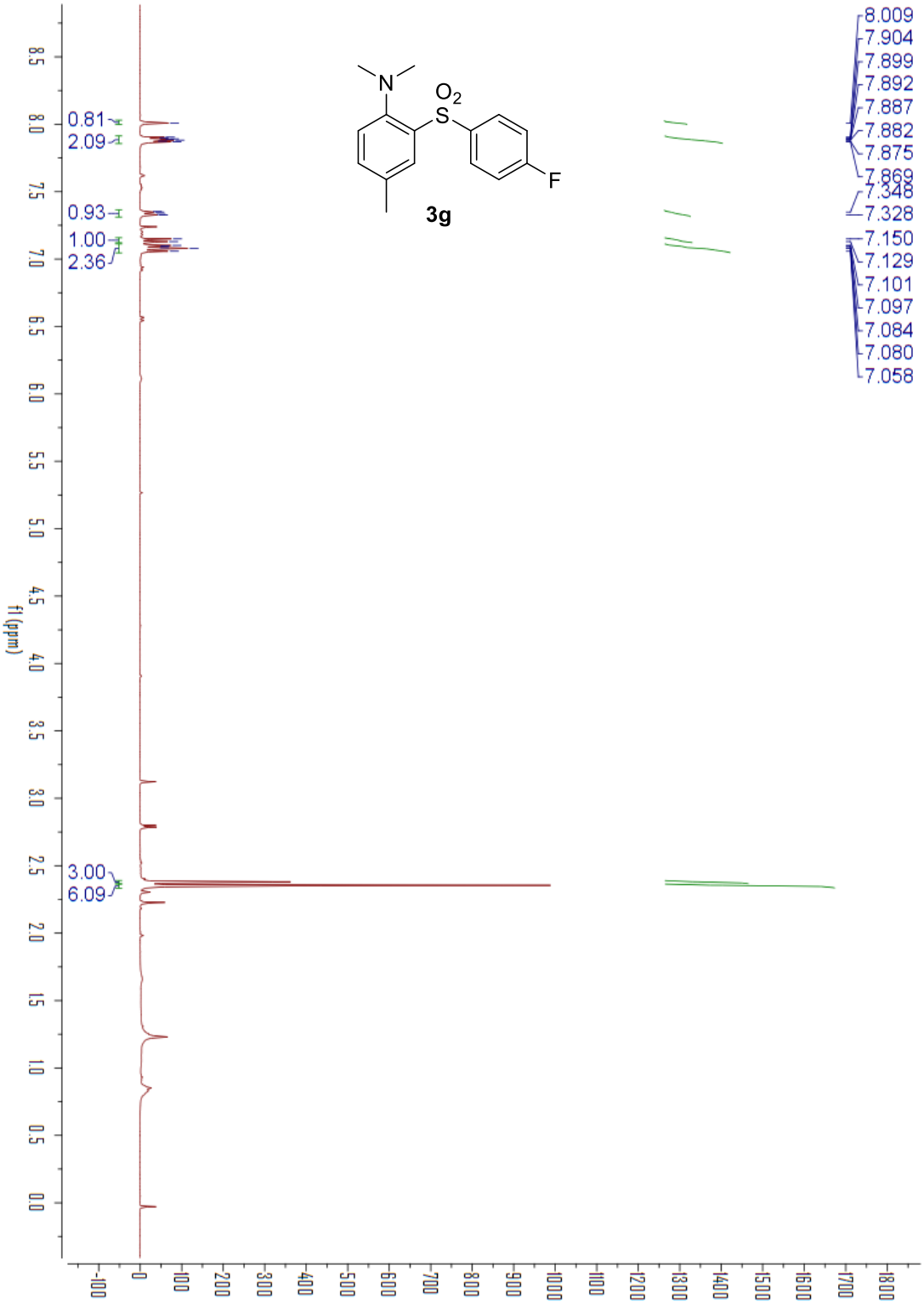
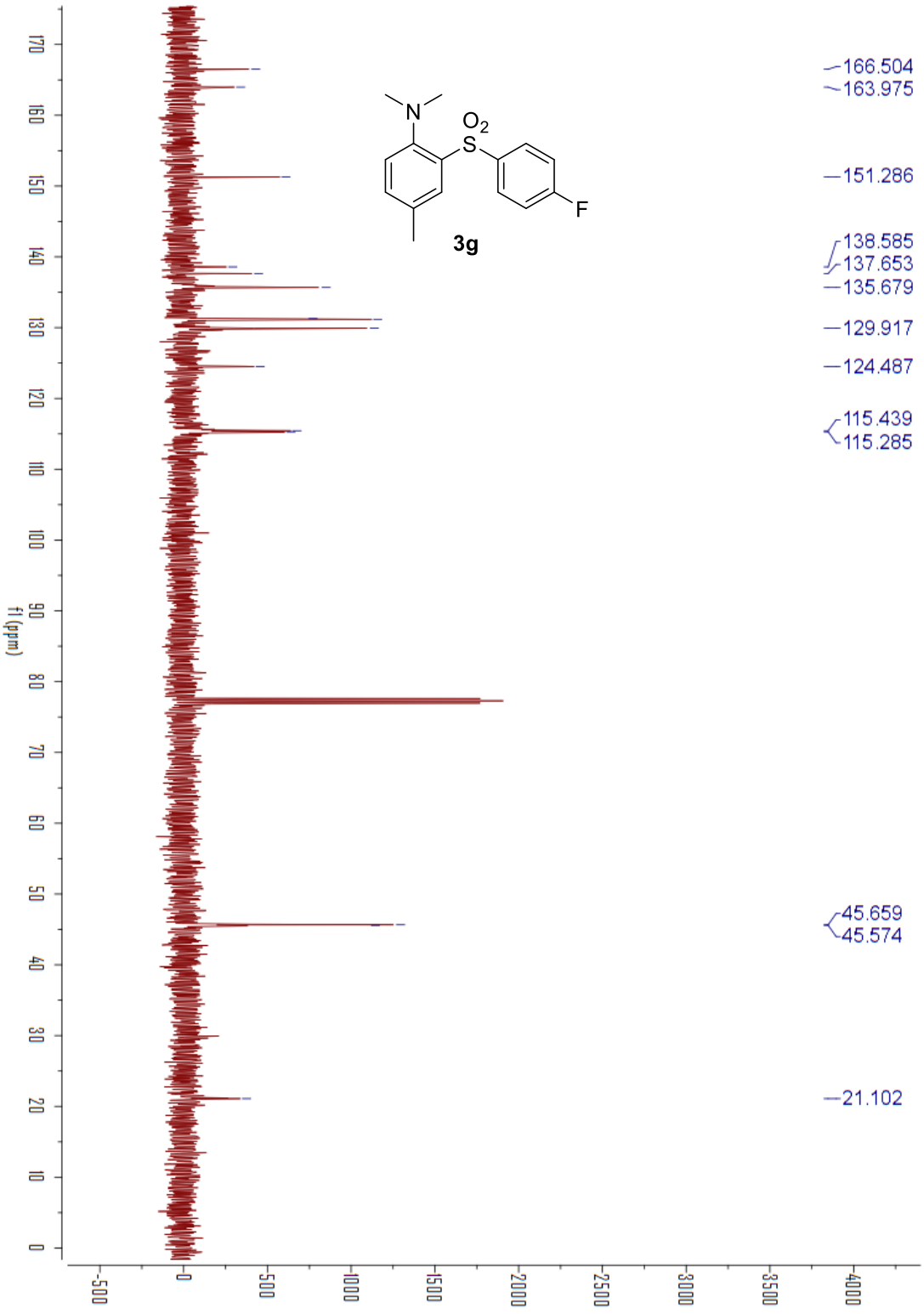
143.244
132.602
130.026
128.422
124.604
52.762
45.558
21.204
f1 (ppm)


8.009
7.904
7.899
7.892
7.887
7.882
7.875
7.869
7.348
7.328
7.150
7.129
7.101
7.097
7.084
7.080
7.058
0.81
2.09
0.93
1.00
2.36
3.00
6.09
N
O2
S
F
3g
f1 (ppm)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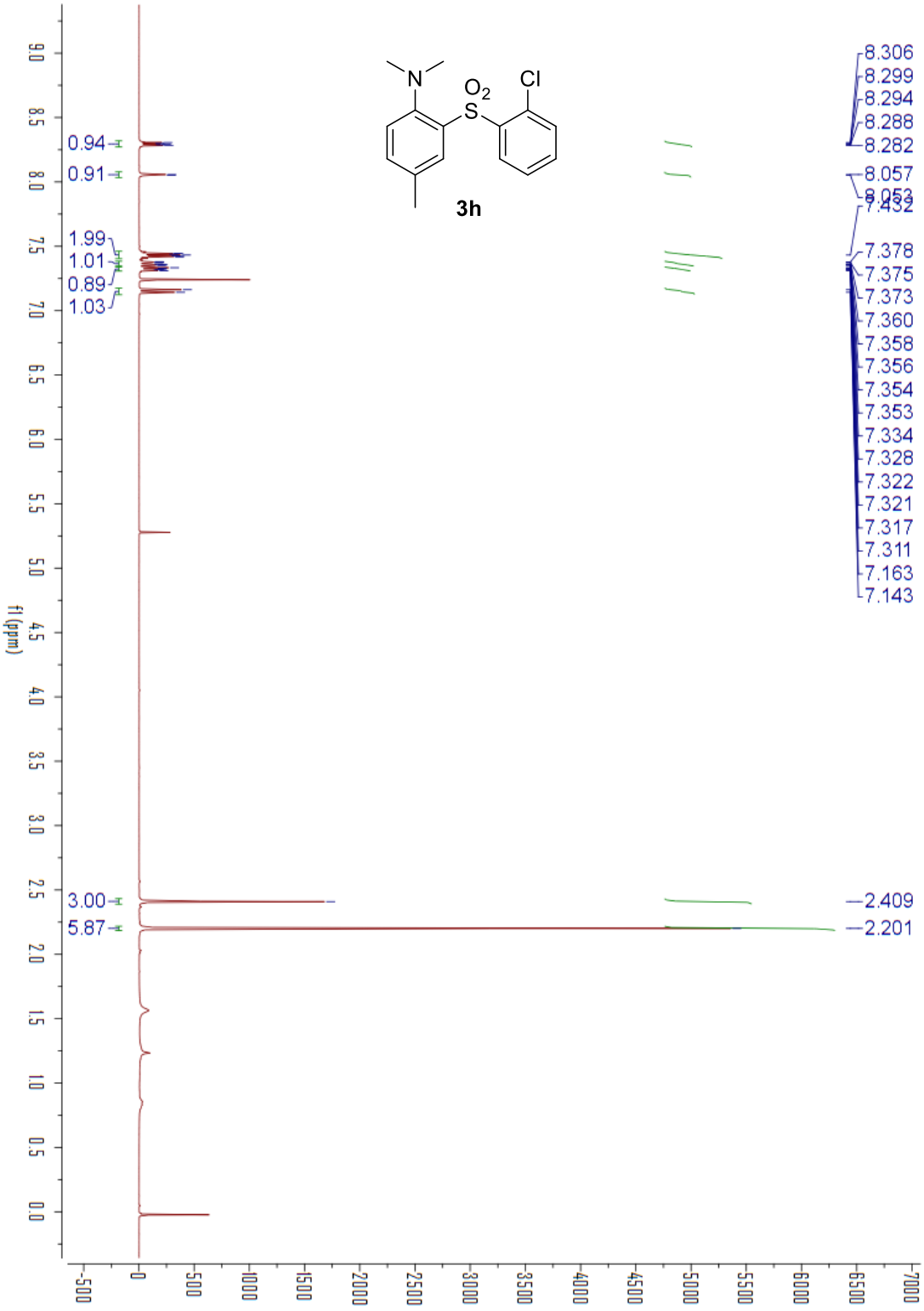
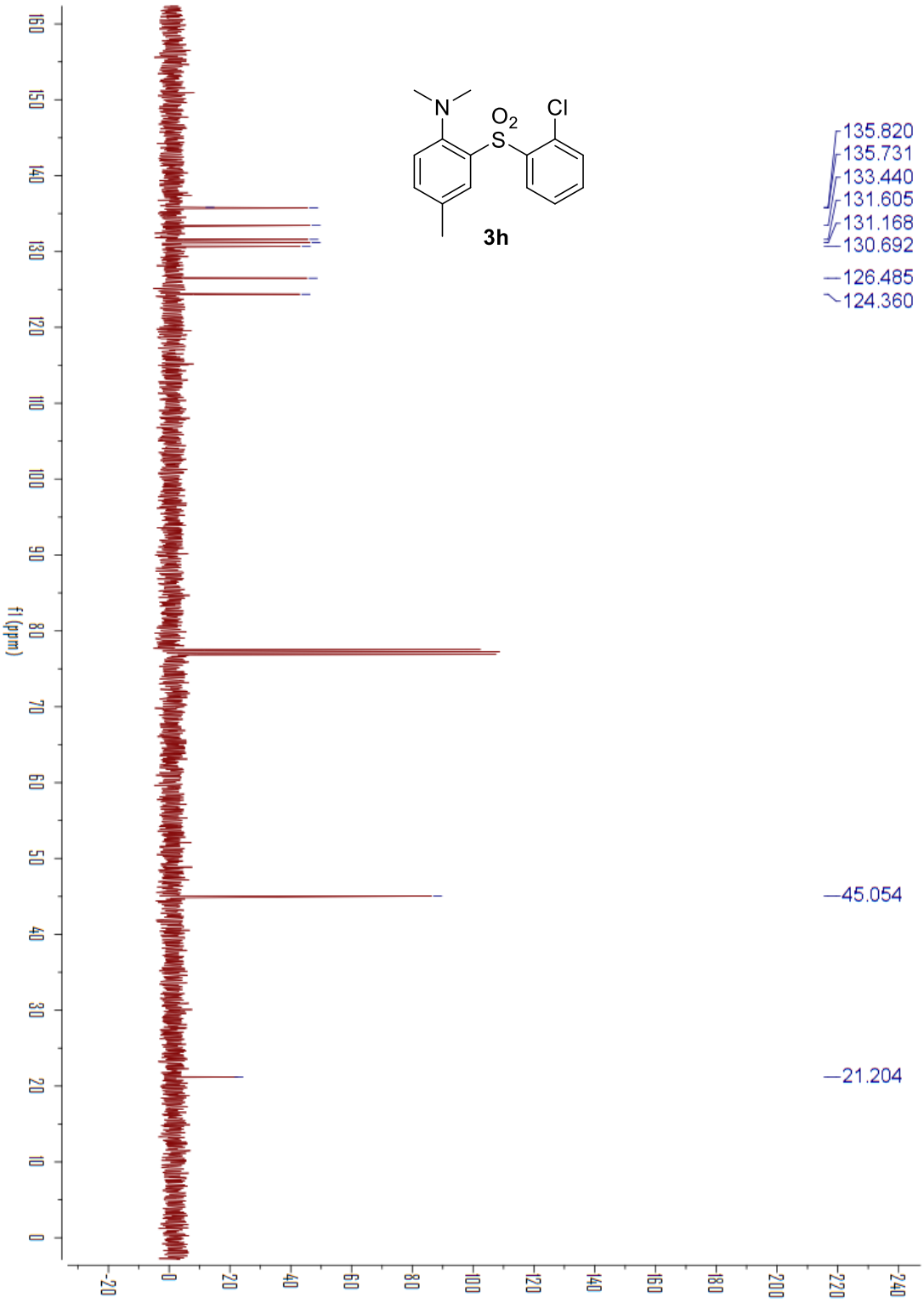
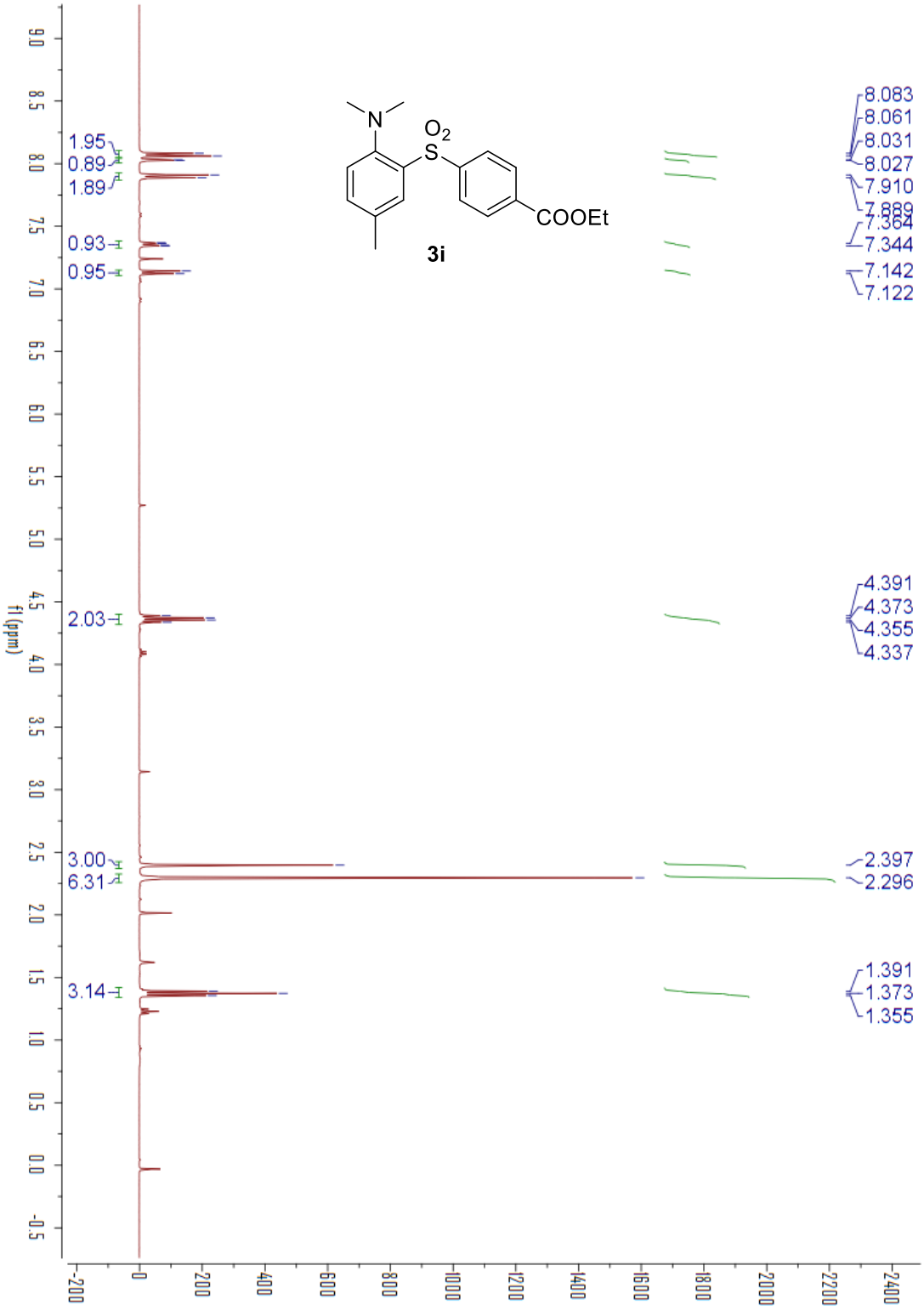
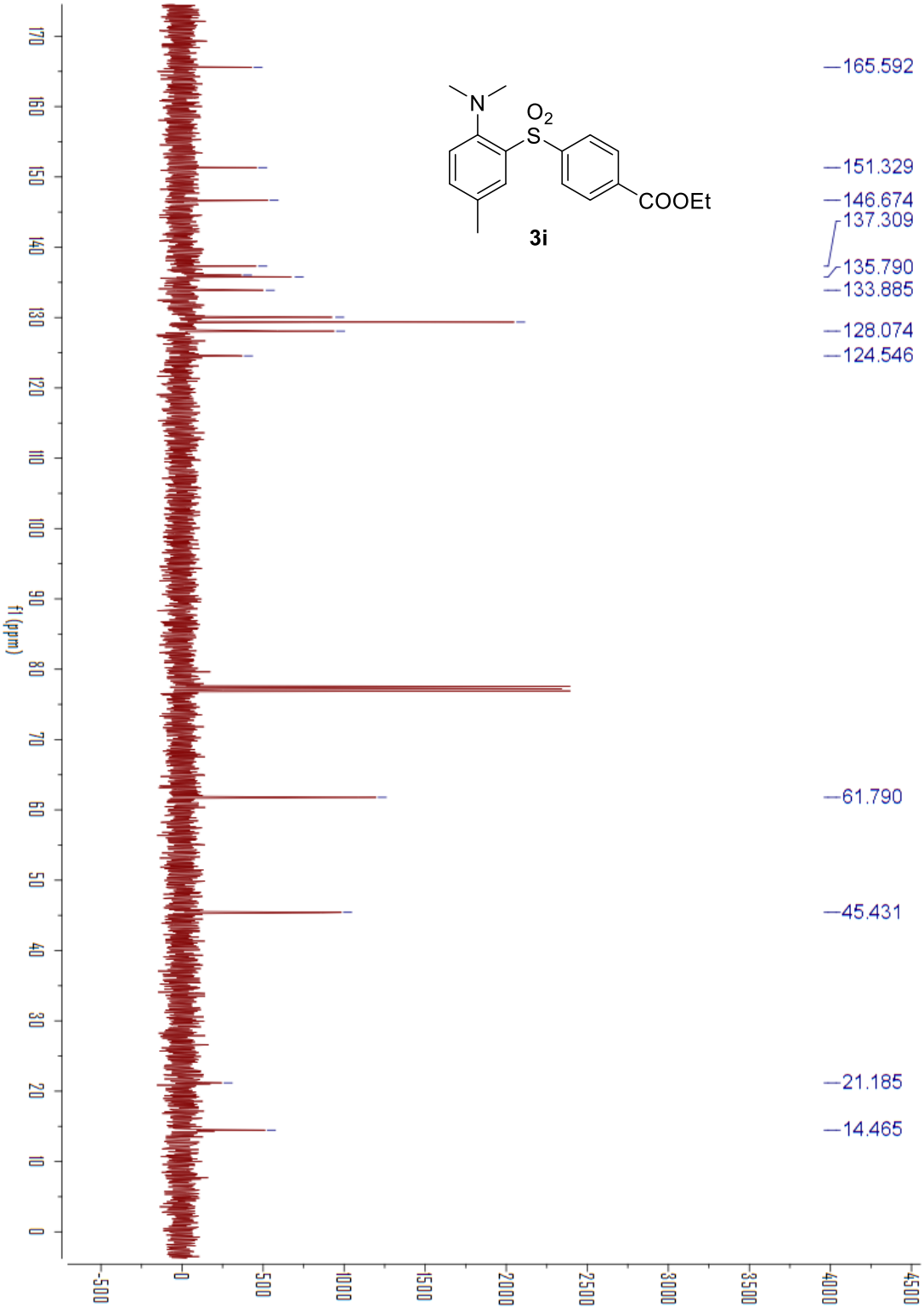
163.975
151.286
138.585
137.653
135.679
129.917
124.487
115.439
115.285
45.659
45.574
21.102
N
O2
S
F
3g
f1 (ppm)


N
O2
S
Cl
3h
0.94
0.91
1.99
1.01
0.89
1.03
3.00
5.87
8.306
8.299
8.294
8.288
8.282
8.057
8.053
7.432
7.378
7.375
7.373
7.360
7.358
7.356
7.354
7.353
7.334
7.328
7.322
7.321
7.317
7.311
7.163
7.143
2.409
2.201
f1 (ppm)


N
O2
S
Cl
3h
135.820
135.731
133.440
131.605
131.168
130.692
126.485
124.360
45.054
21.204
f1 (ppm)


N
O2
S
COOEt
3i
1.95
0.89
1.89
0.93
0.95
2.03
3.00
6.31
3.14
8.083
8.061
8.031
8.027
7.910
7.889
7.364
7.344
7.142
7.122
4.391
4.373
4.355
4.337
2.397
2.296
1.391
1.373
1.355
f1 (ppm)


N
O2
S
COOEt
3i
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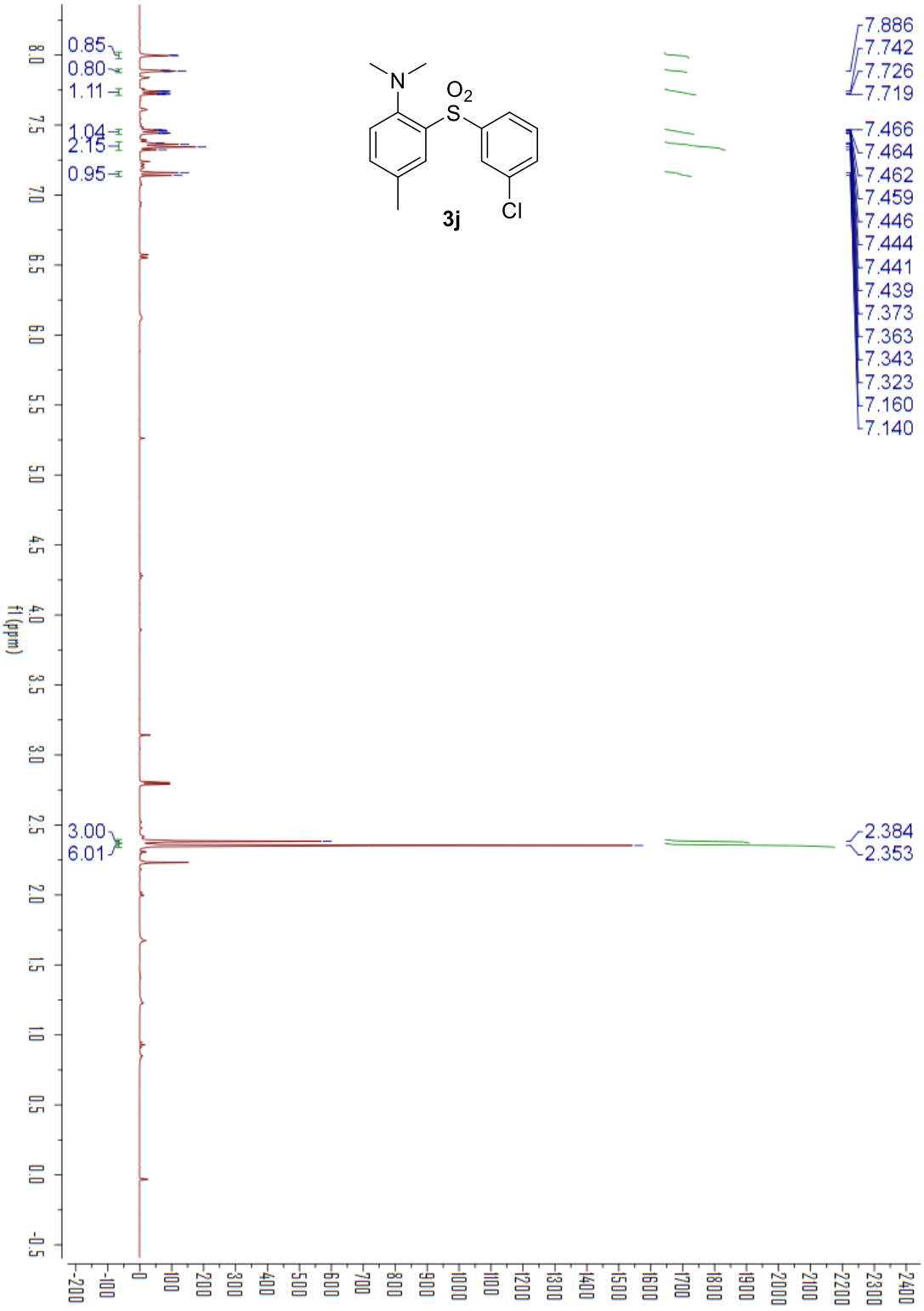
151.329
146.674
137.309
135.790
133.885
128.074
124.546
61.790
45.431
21.185
14.465
f1 (ppm)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0.80
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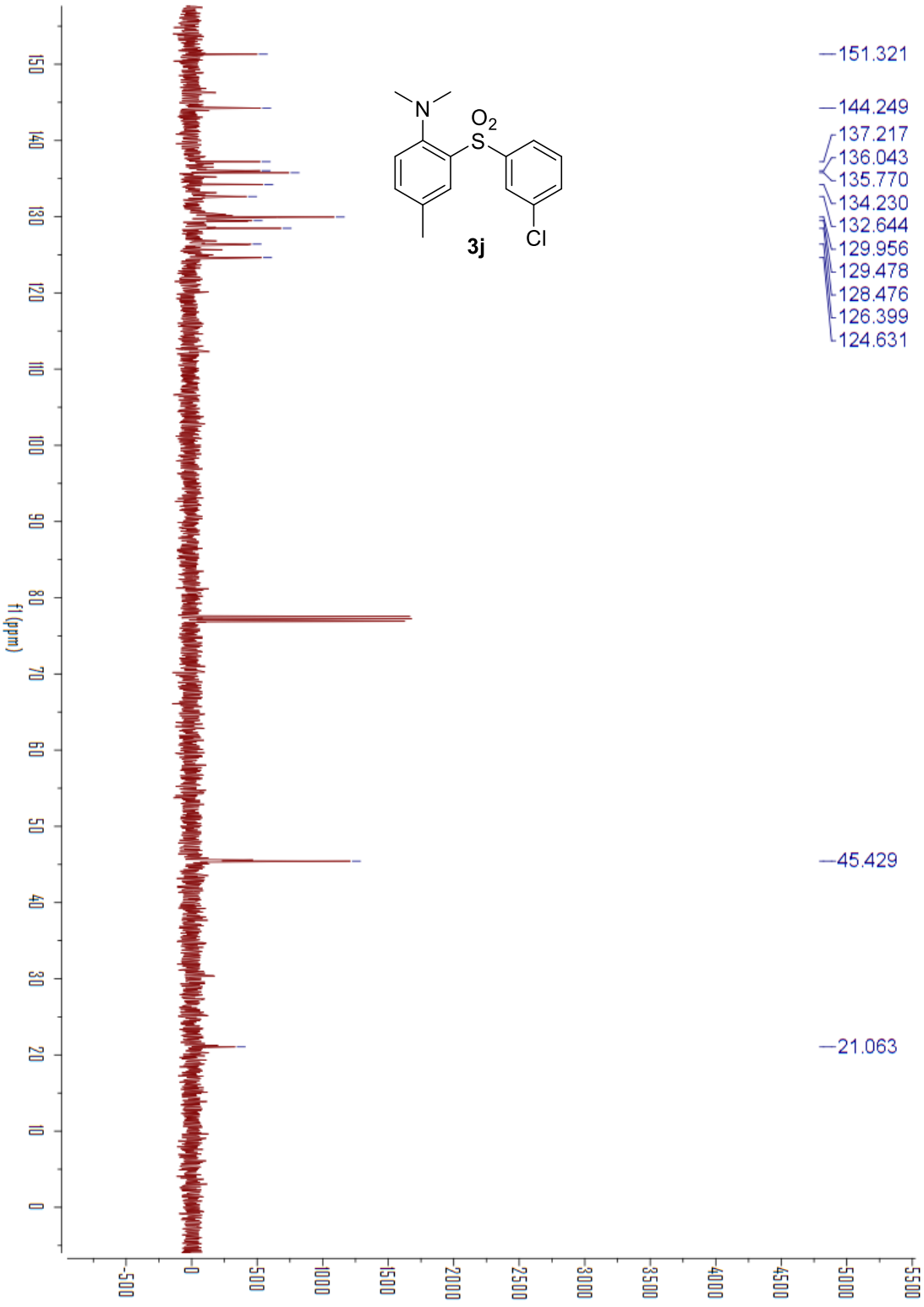
1.04
2.15
0.95
3.00
6.01
7.886
7.742
7.726
7.719
7.466
7.464
7.462
7.459
7.446
7.444
7.441
7.439
7.373
7.363
7.343
7.323
7.160
7.140
2.384
2.353
N
O2
S
Cl
3j
f1 (ppm)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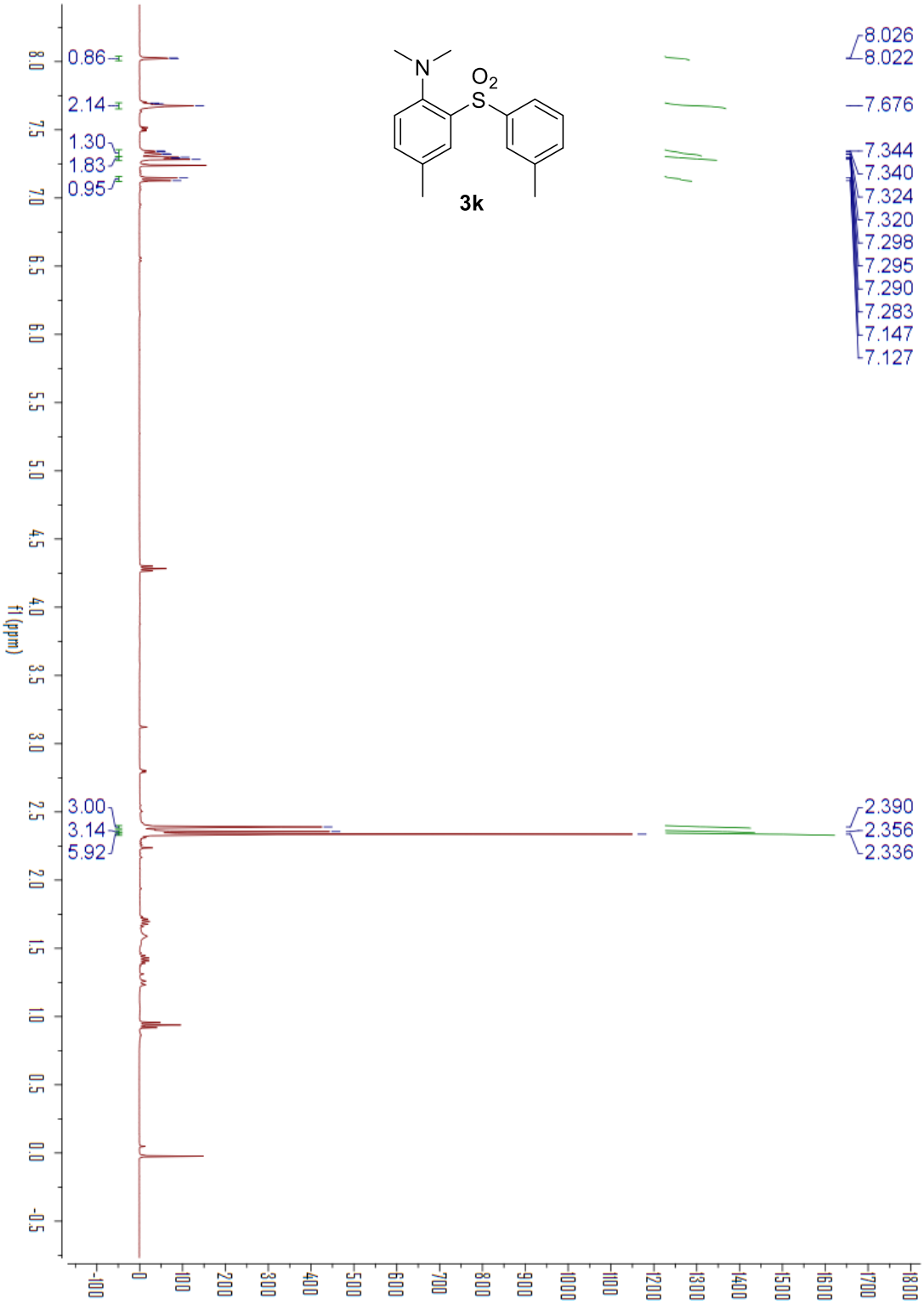
137.217
136.043
135.770
134.230
132.644
129.956
129.478
128.476
126.399
124.631
45.429
21.063
N
O2
S
Cl
3j
f1 (ppm)


0.86
2.14
1.30
1.83
0.95
3.00
3.14
5.92
8.026
8.022
7.676
7.344
7.340
7.324
7.320
7.298
7.295
7.290
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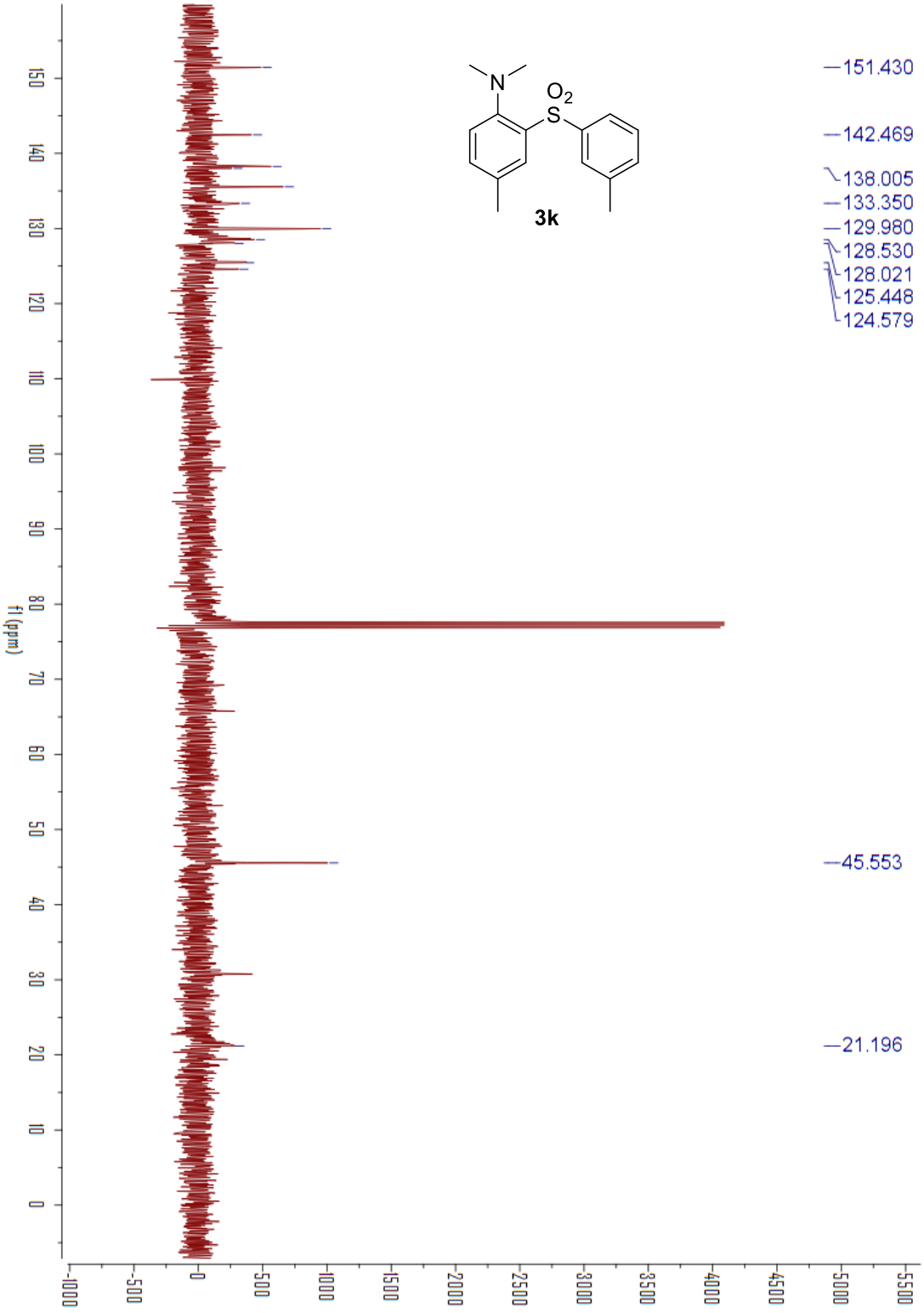
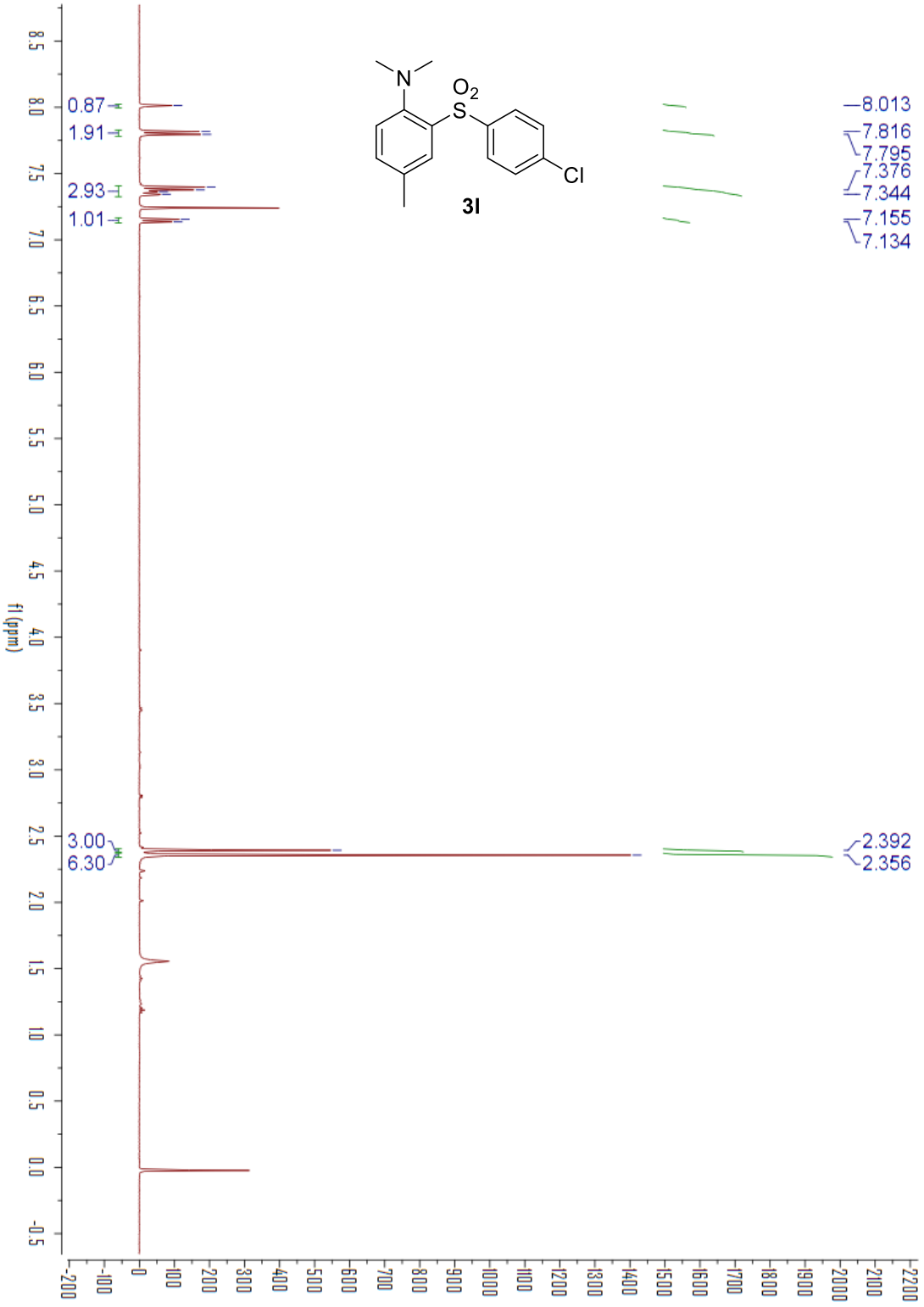
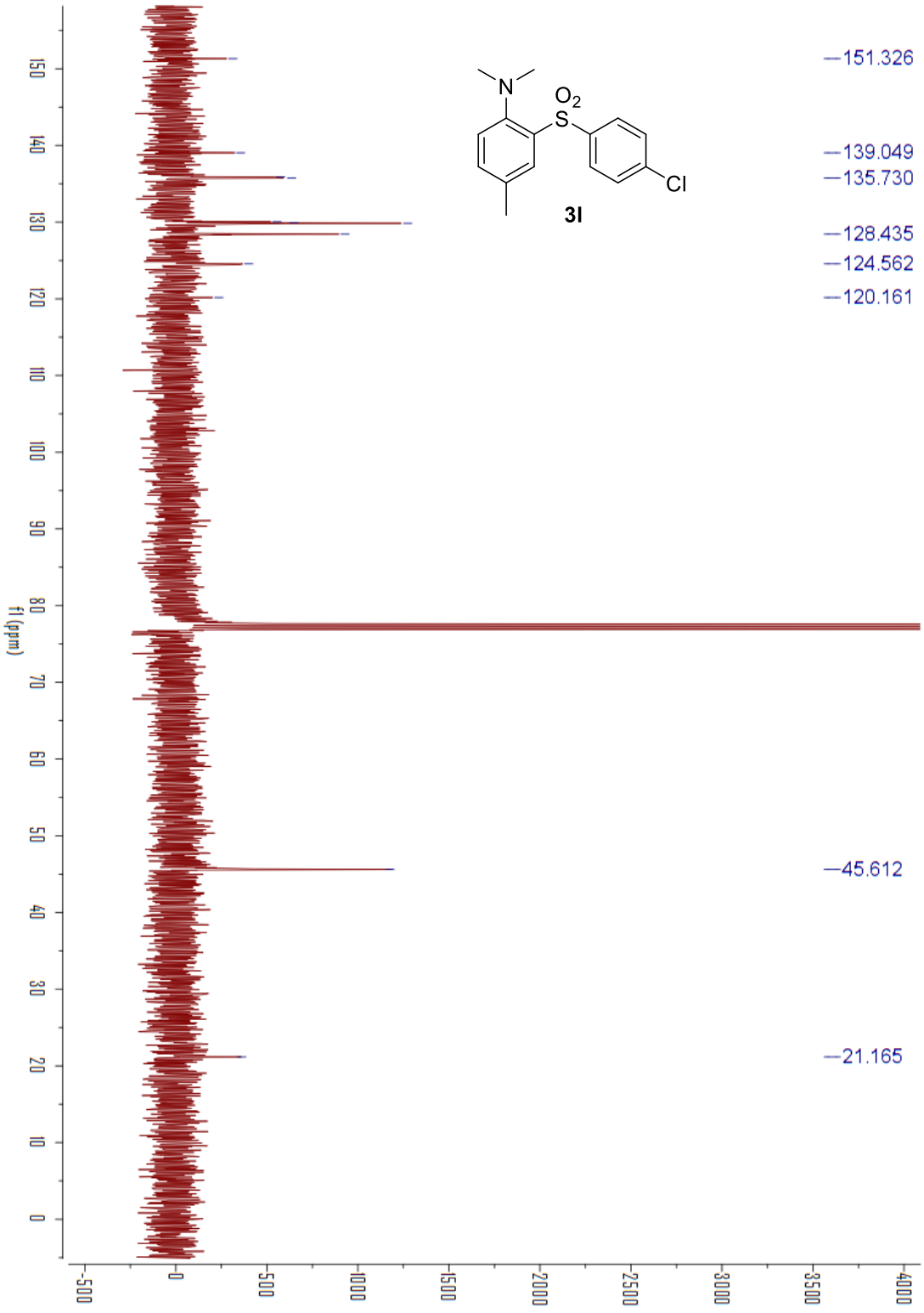
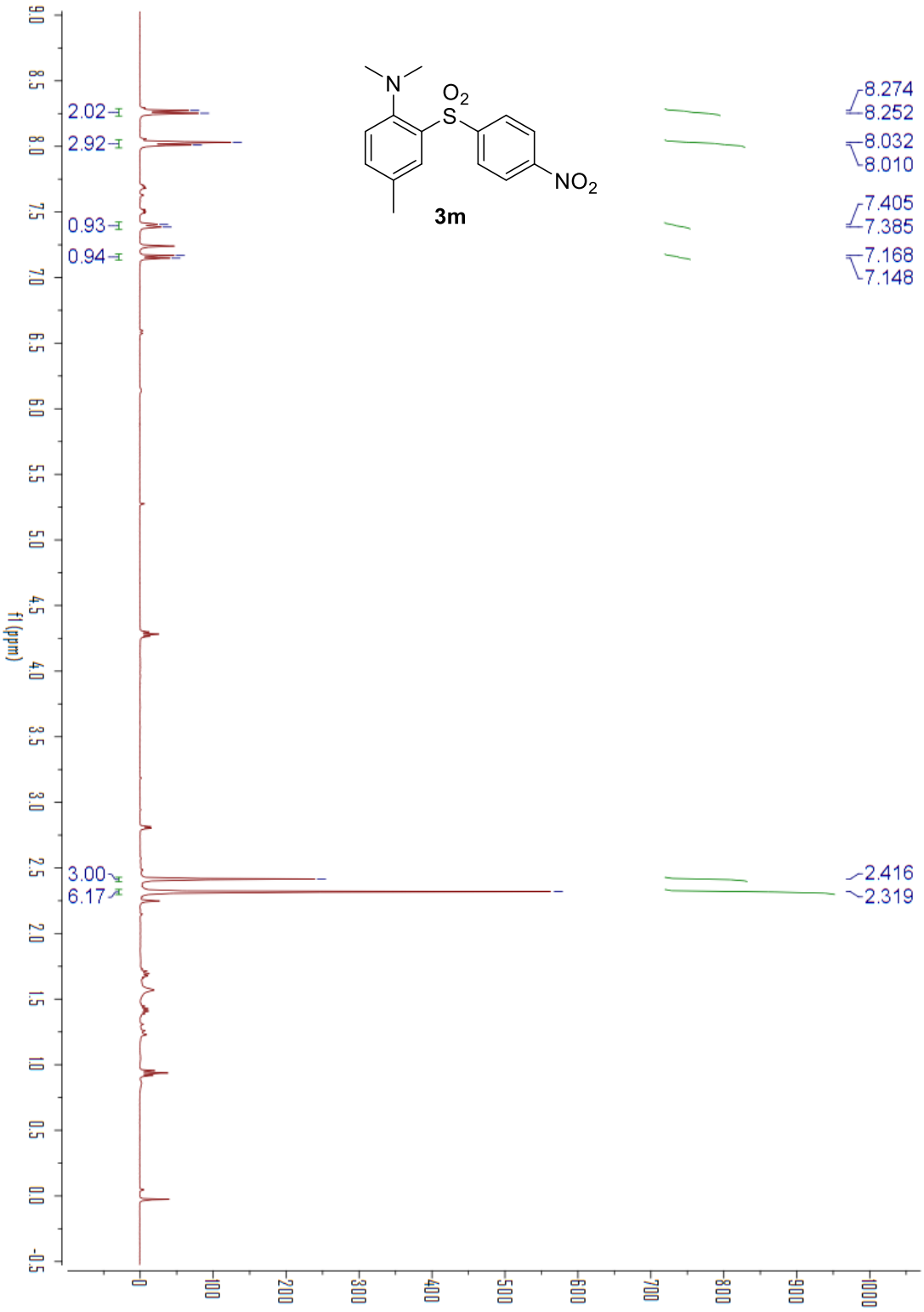
2.336
N
O2
S
3k
f1 (ppm)


151.430
142.469
138.005
133.350
129.980
128.530
128.021
125.448
124.579
45.553
21.196
f1 (ppm)
3k


0.87
1.91
2.93
1.01
3.00
6.30
8.013
7.816
7.795
7.376
7.344
7.155
7.134
2.392
2.356
f1 (ppm)
3I


151.326
139.049
135.730
128.435
124.562
120.161
45.612
21.165
N
O2
S
Cl
3l
f1 (ppm)


2.02
2.92
0.93
0.94
3.00
6.17
8.274
8.252
8.032
8.010
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7.385
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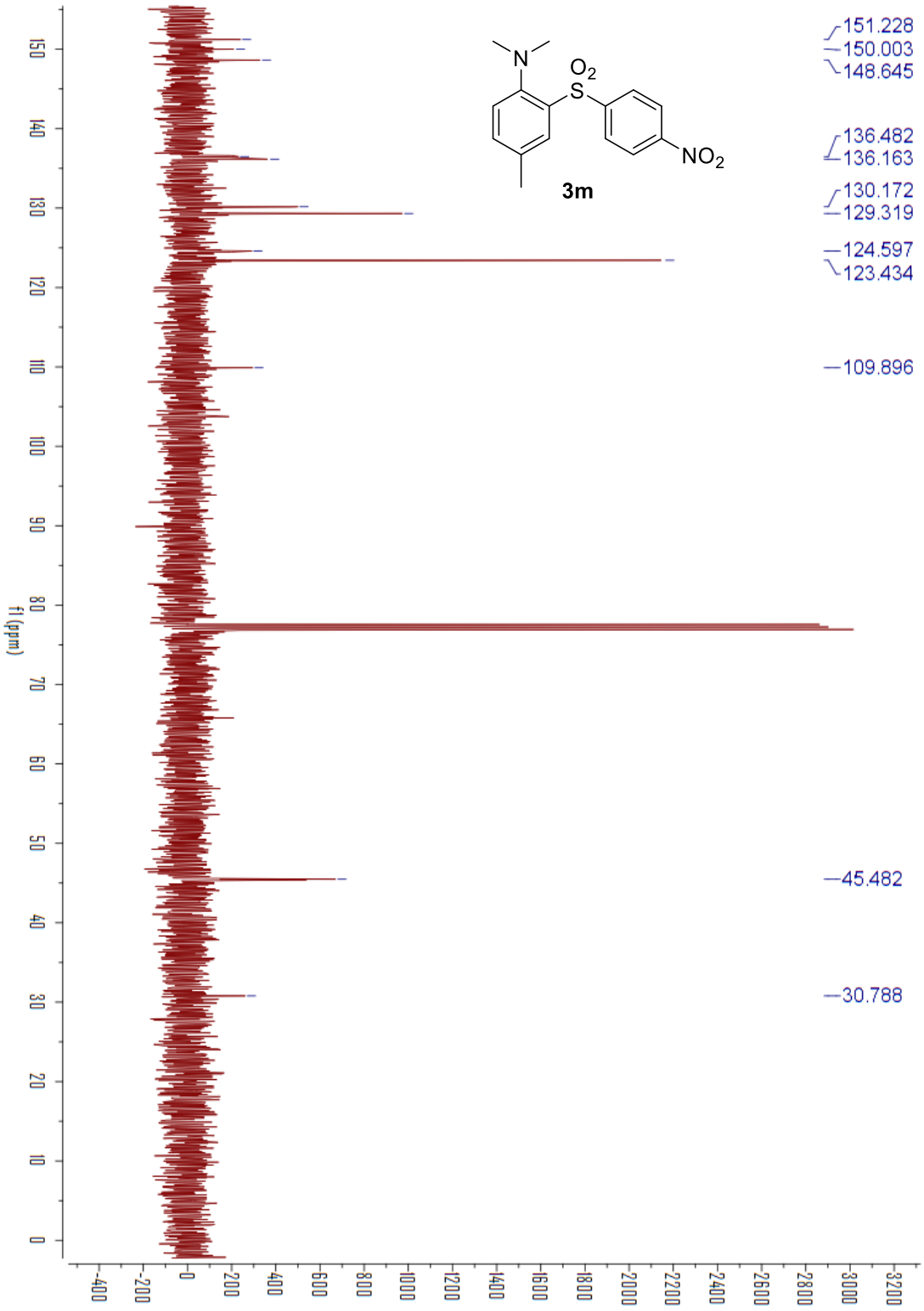
2.319
N
O2
S
NO2
3m
f1 (ppm)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150.003
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129.319
124.597
123.434
109.896
45.482
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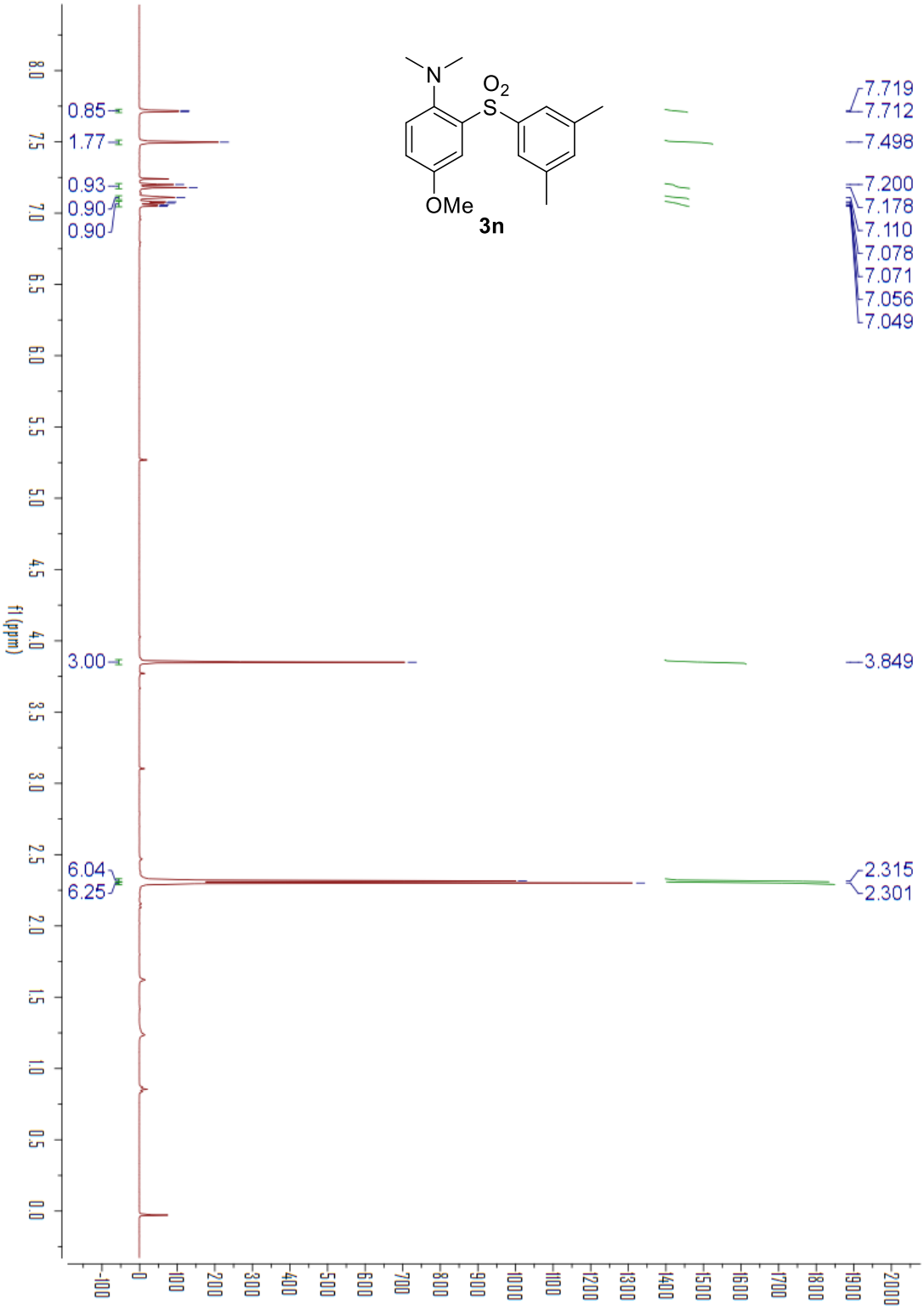
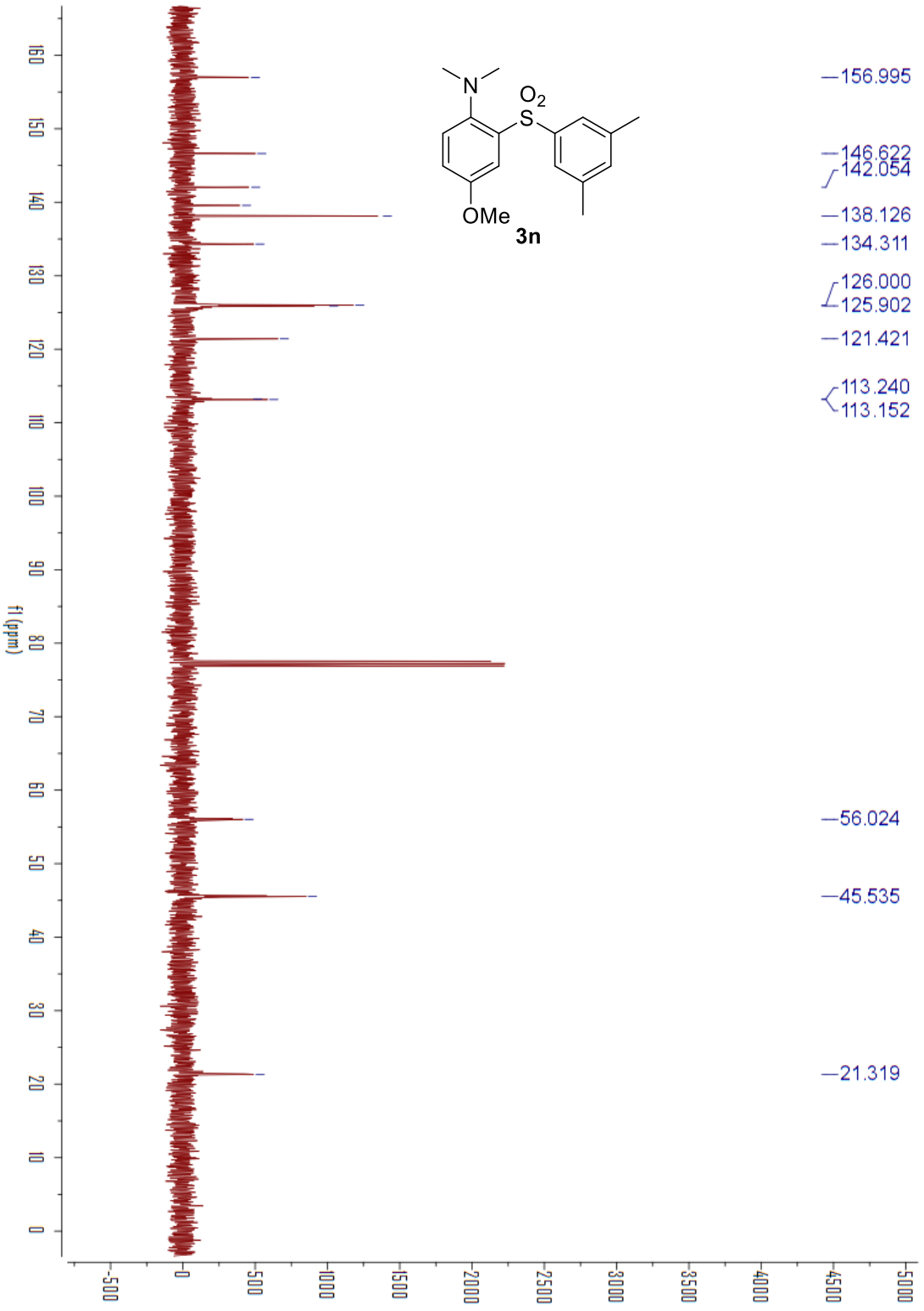
3m
f1 (ppm)


N
O2
S
OMe
3n
0.85
1.77
0.93
0.90
0.90
3.00
6.04
6.25
7.719
7.712
7.498
7.200
7.178
7.110
7.078
7.071
7.056
7.049
3.849
2.315
2.301
f1 (ppm)


N
O2
S
OMe
3n
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146.622
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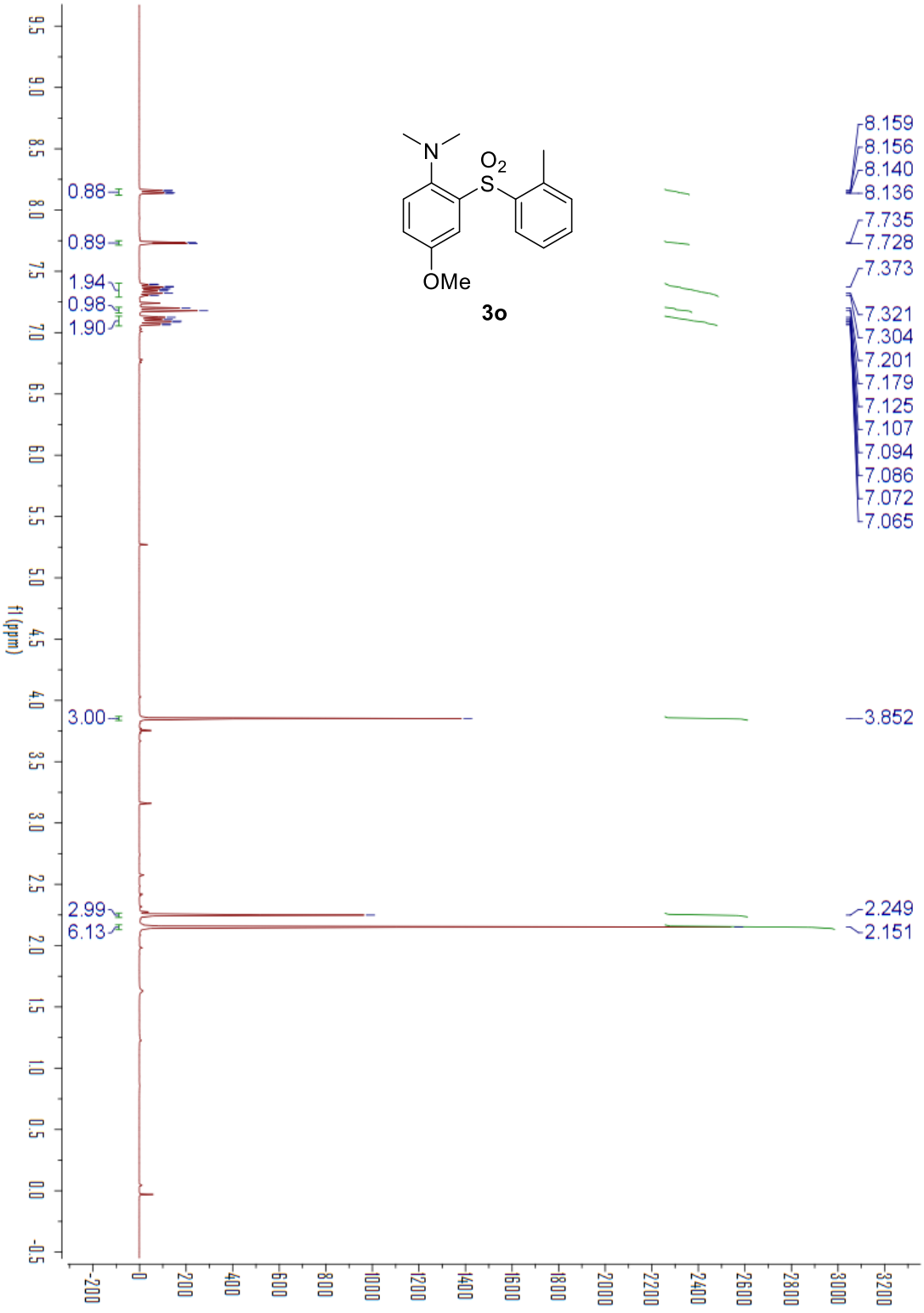
138.126
134.311
126.000
125.902
121.421
113.240
113.152
56.024
45.535
21.319
f1 (ppm)


N
O2
S
OMe
3o
0.88
0.89
1.94
0.98
1.90
3.00
2.99
6.13
8.159
8.156
8.140
8.136
7.735
7.728
7.373
7.321
7.304
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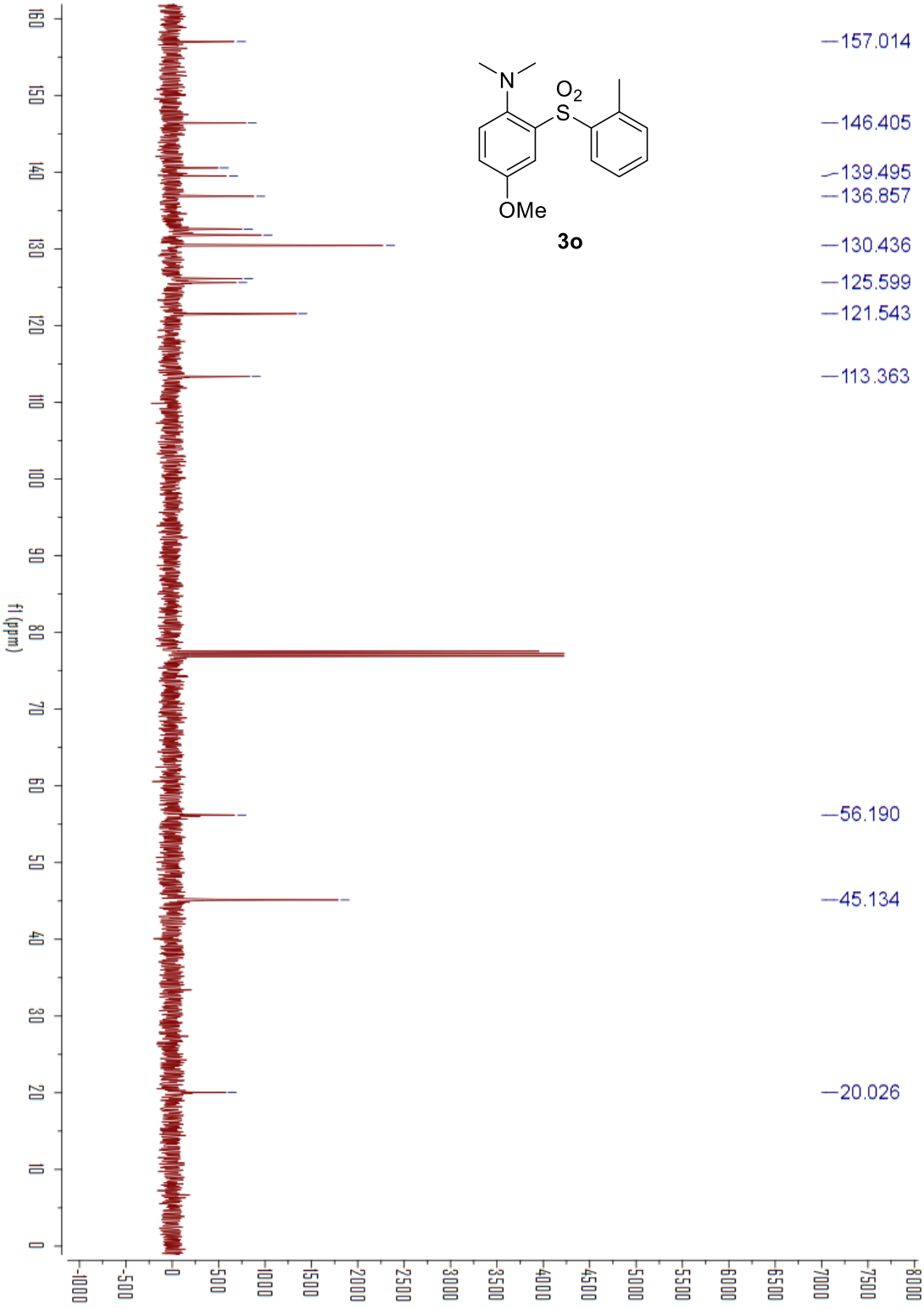
7.065
3.852
2.249
2.151
f1 (ppm)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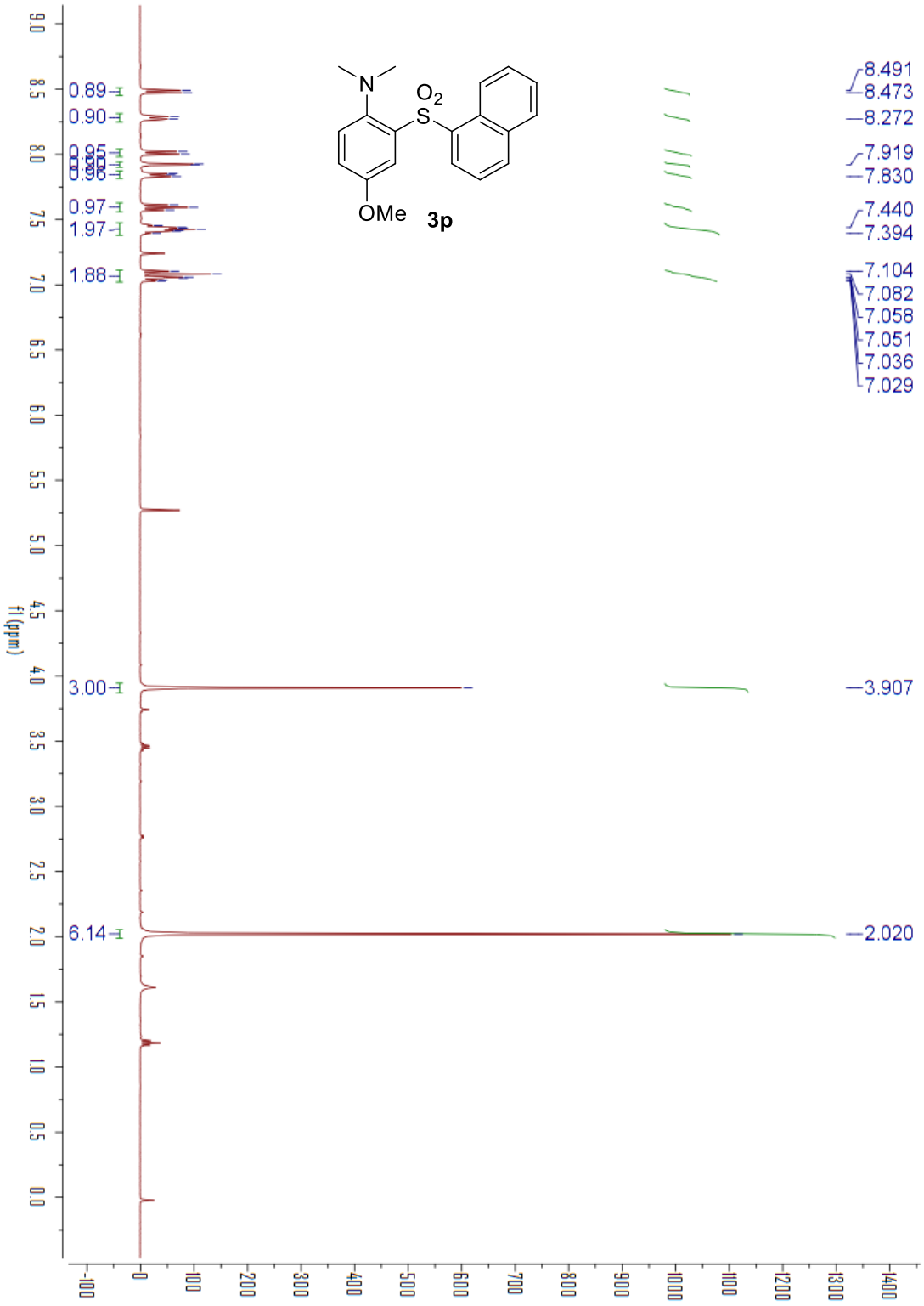
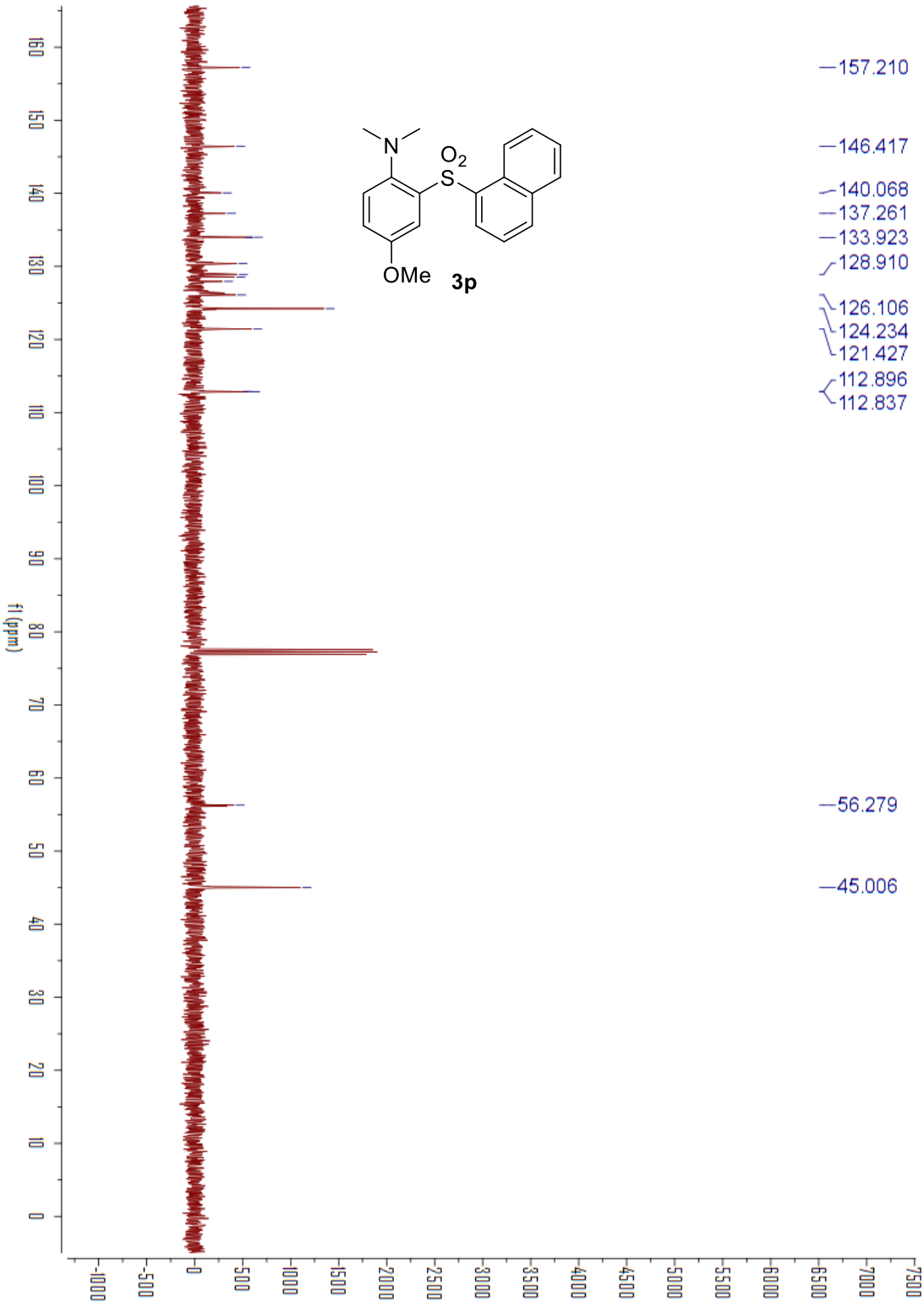
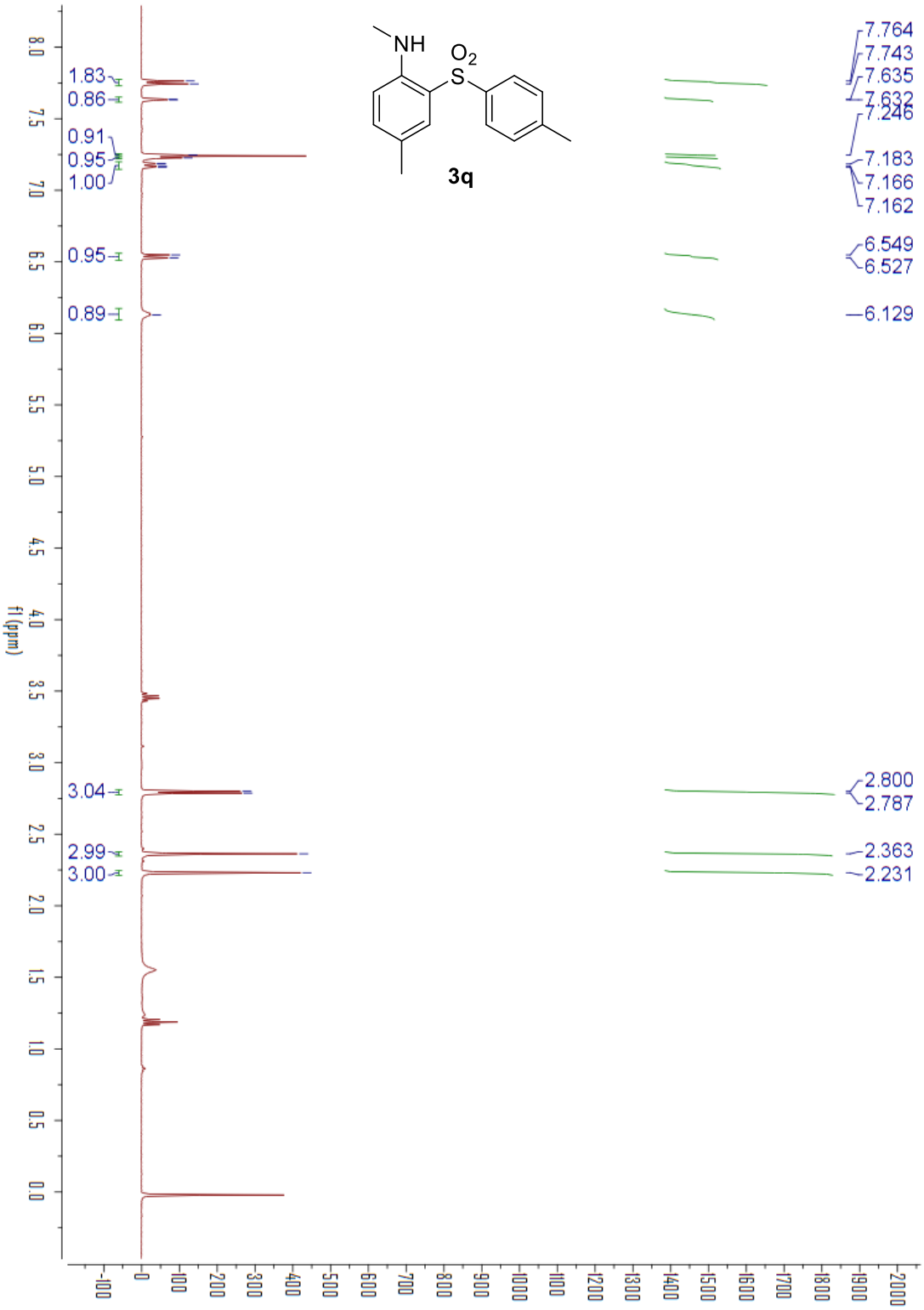
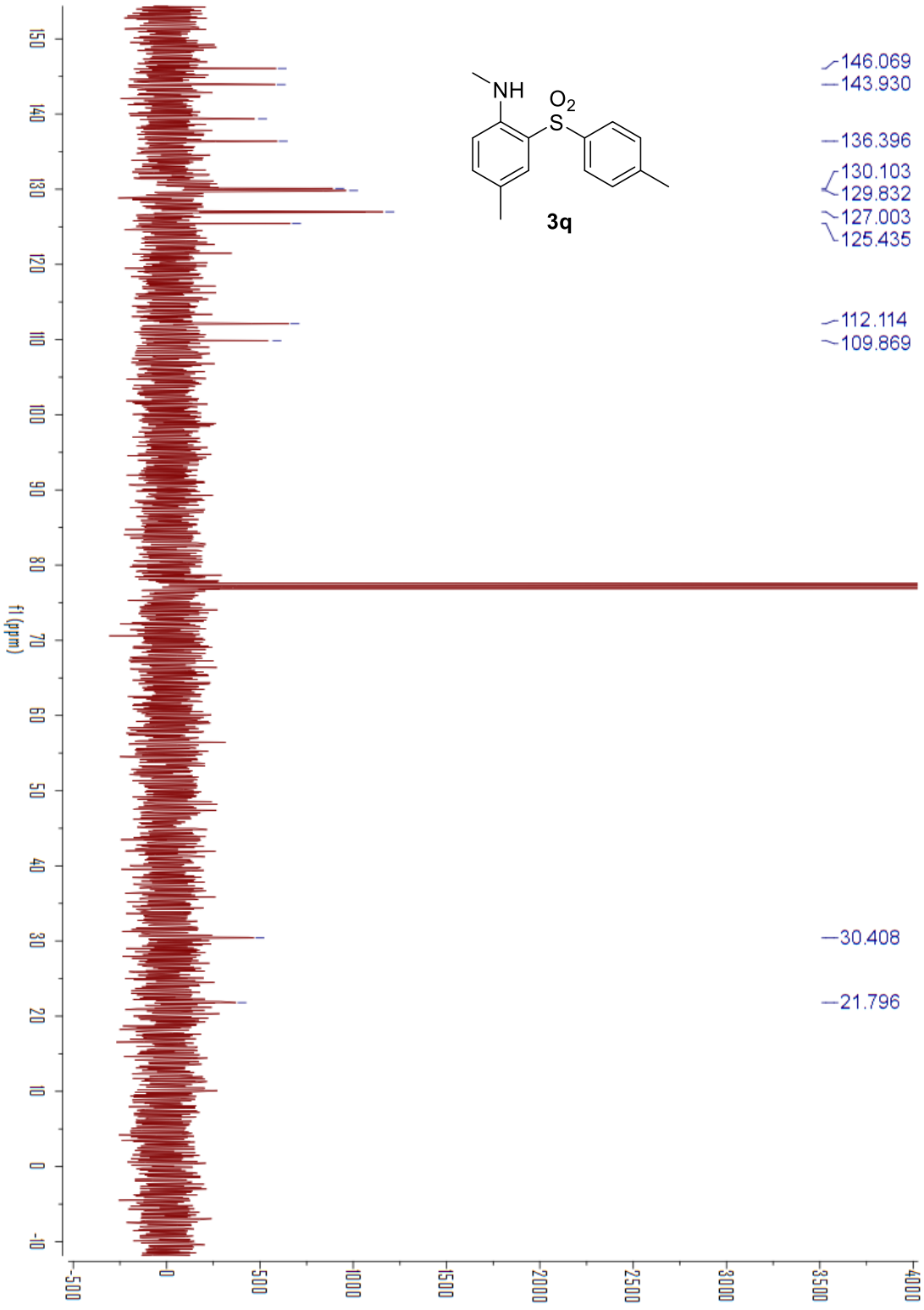
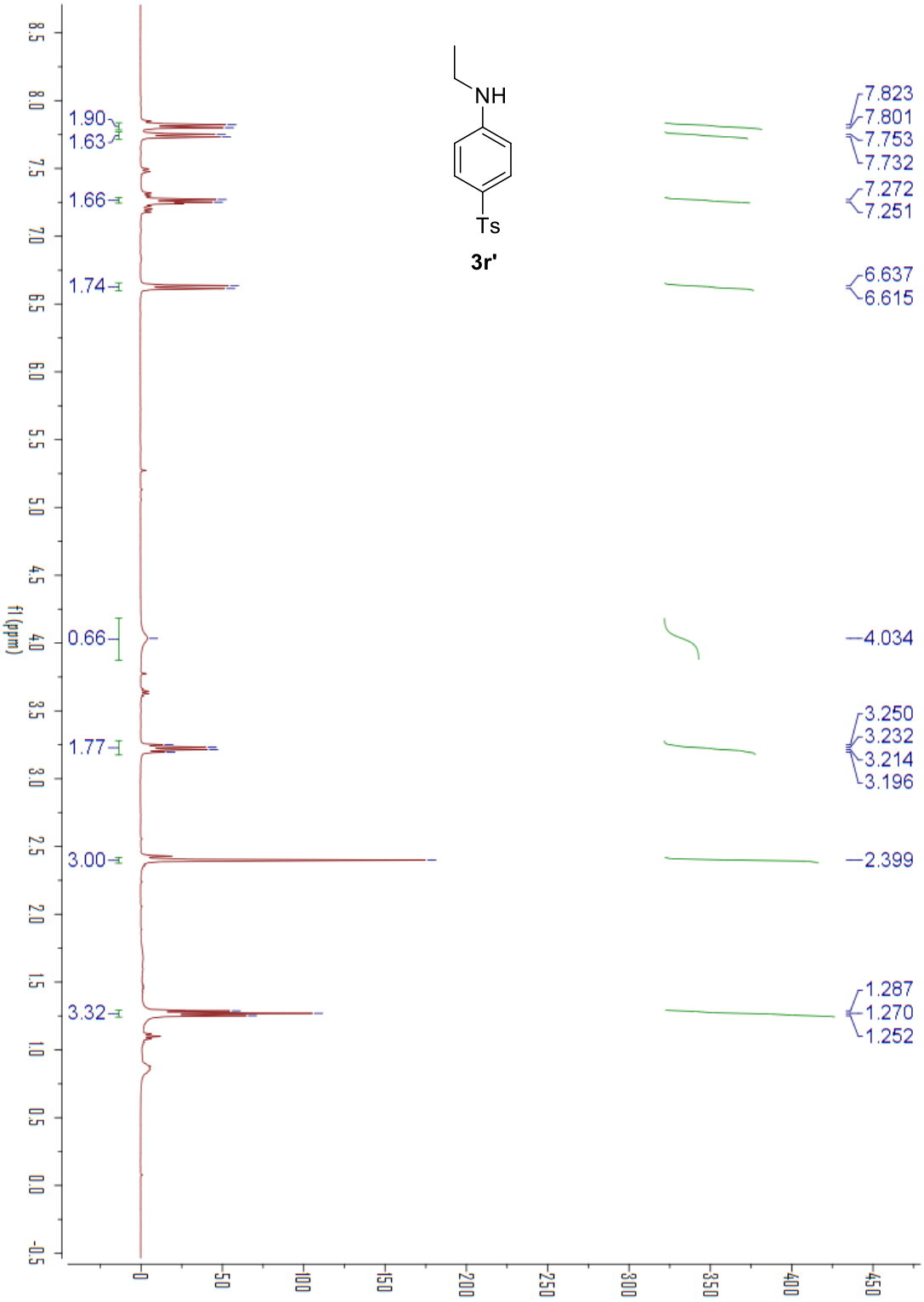
136.857
130.436
125.599
121.543
113.363
56.190
45.134
20.026
N
O2
S
OMe
3o
f1 (ppm)

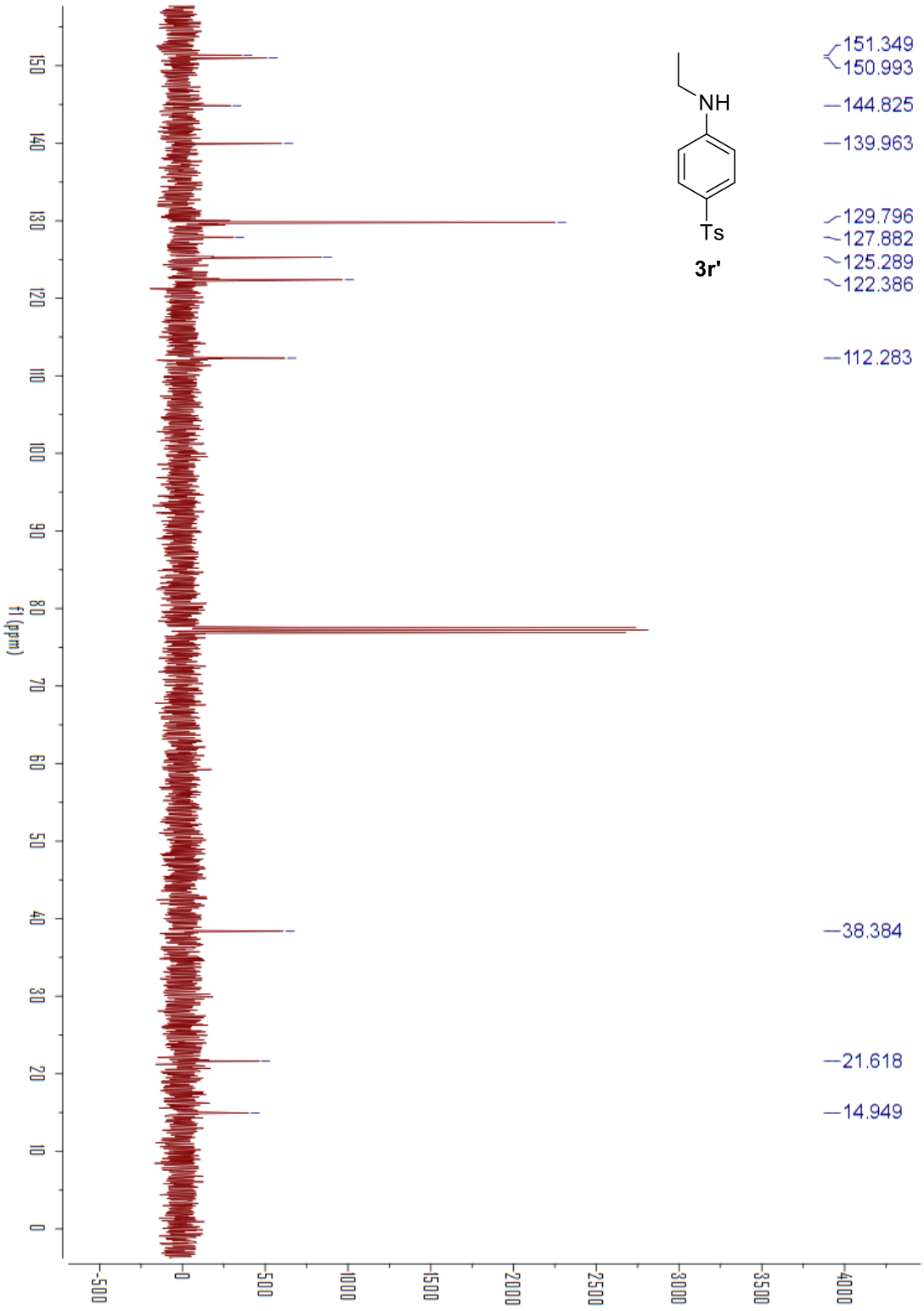

N
O2
S
OMe
3p
0.89
0.90
0.95
0.96
0.97
1.97
1.88
3.00
6.14
8.491
8.473
8.272
7.919
7.830
7.440
7.394
7.104
7.082
7.058
7.051
7.036
7.029
3.907
2.020
f1 (ppm)

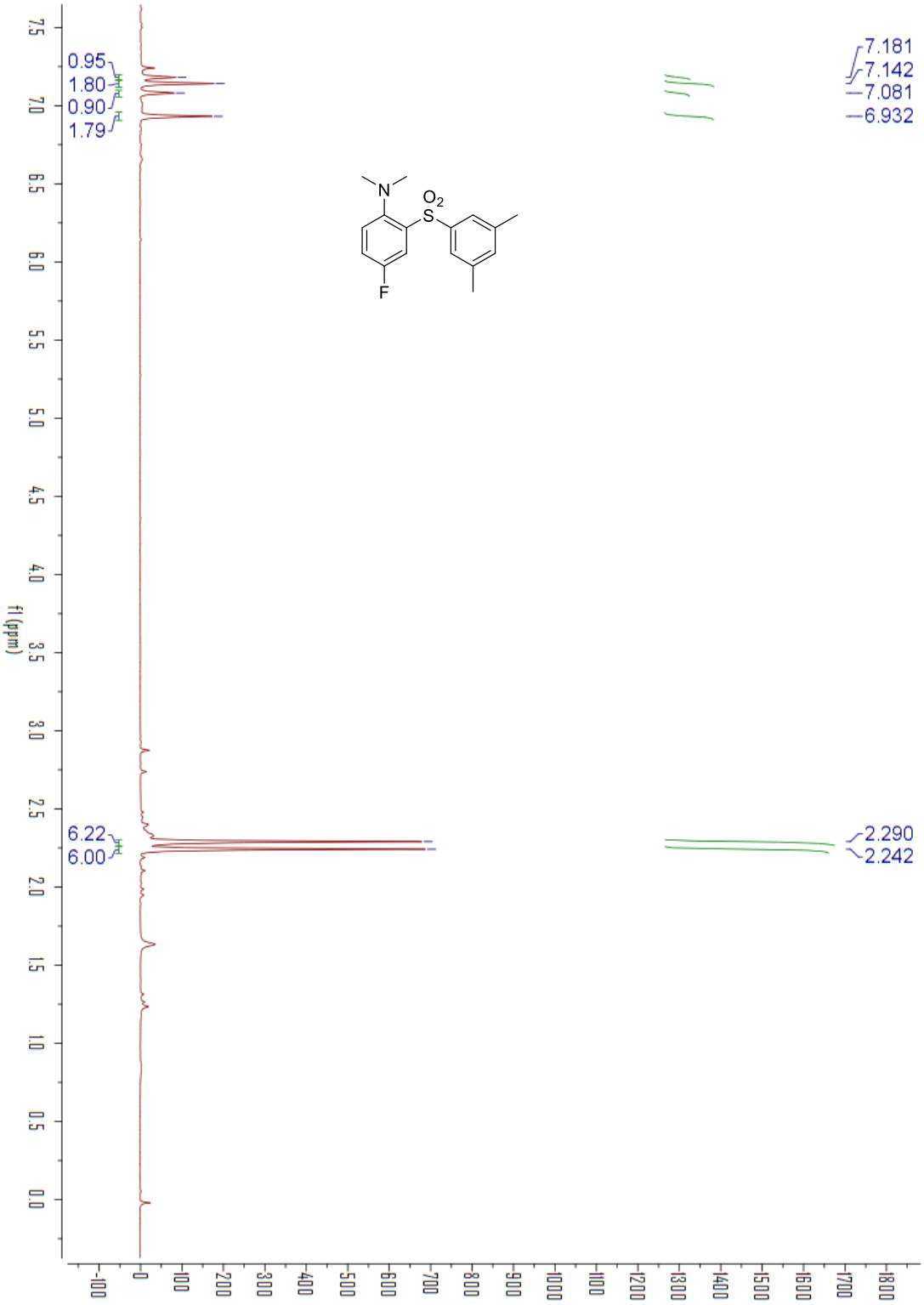

157.210
146.417
140.068
137.261
133.923
128.910
126.106
124.234
121.427
112.896
112.837
56.279
45.006
N
O2
S
OMe
3p
f1 (ppm)

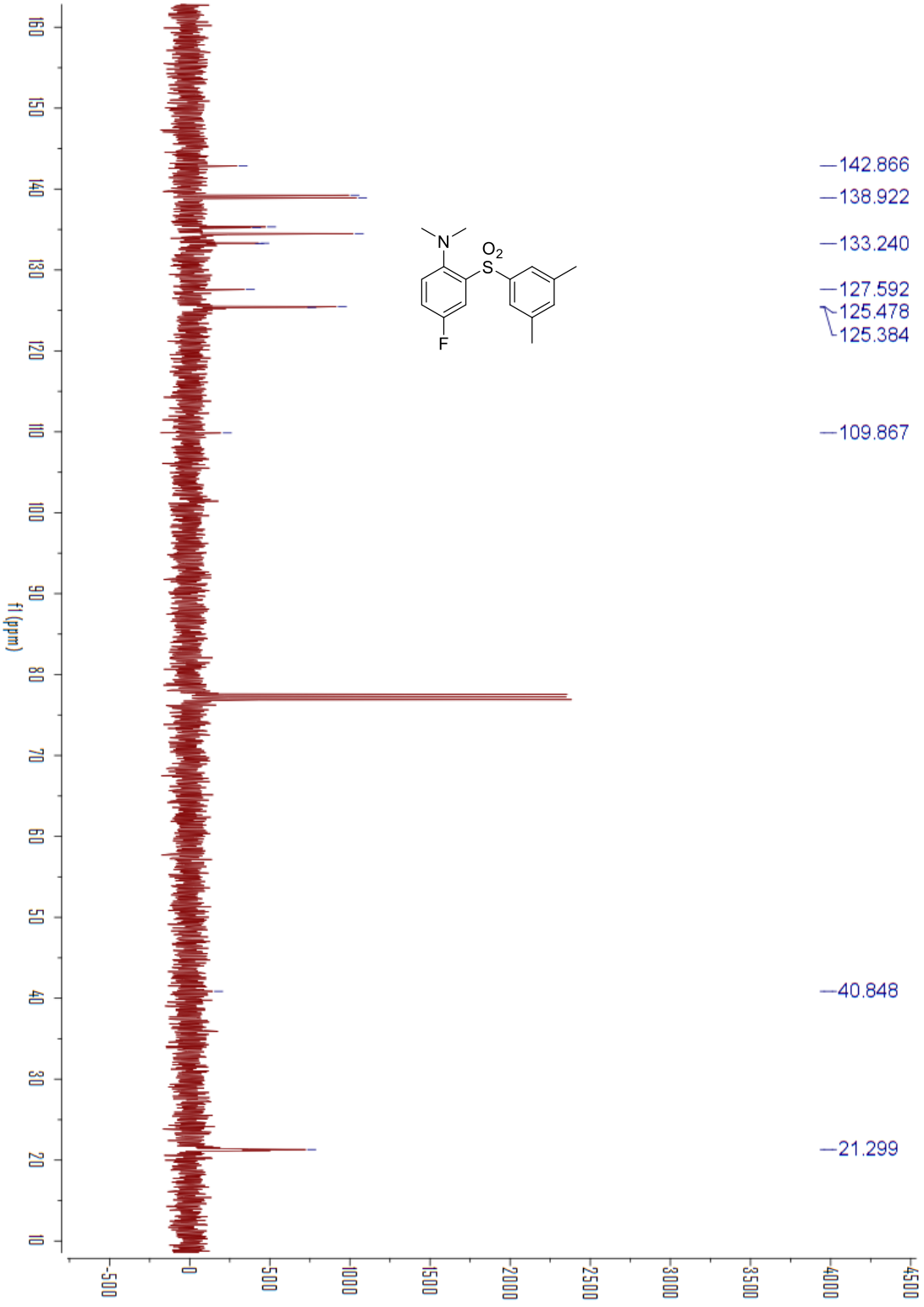

NH
O2
S
3q
7.764
7.743
7.635
7.632
7.246
7.183
7.166
7.162
6.549
6.527
6.129
2.800
2.787
2.363
2.231
1.83
0.86
0.91
0.95
1.00
0.95
0.89
3.04
2.99
3.00
f1 (ppm)

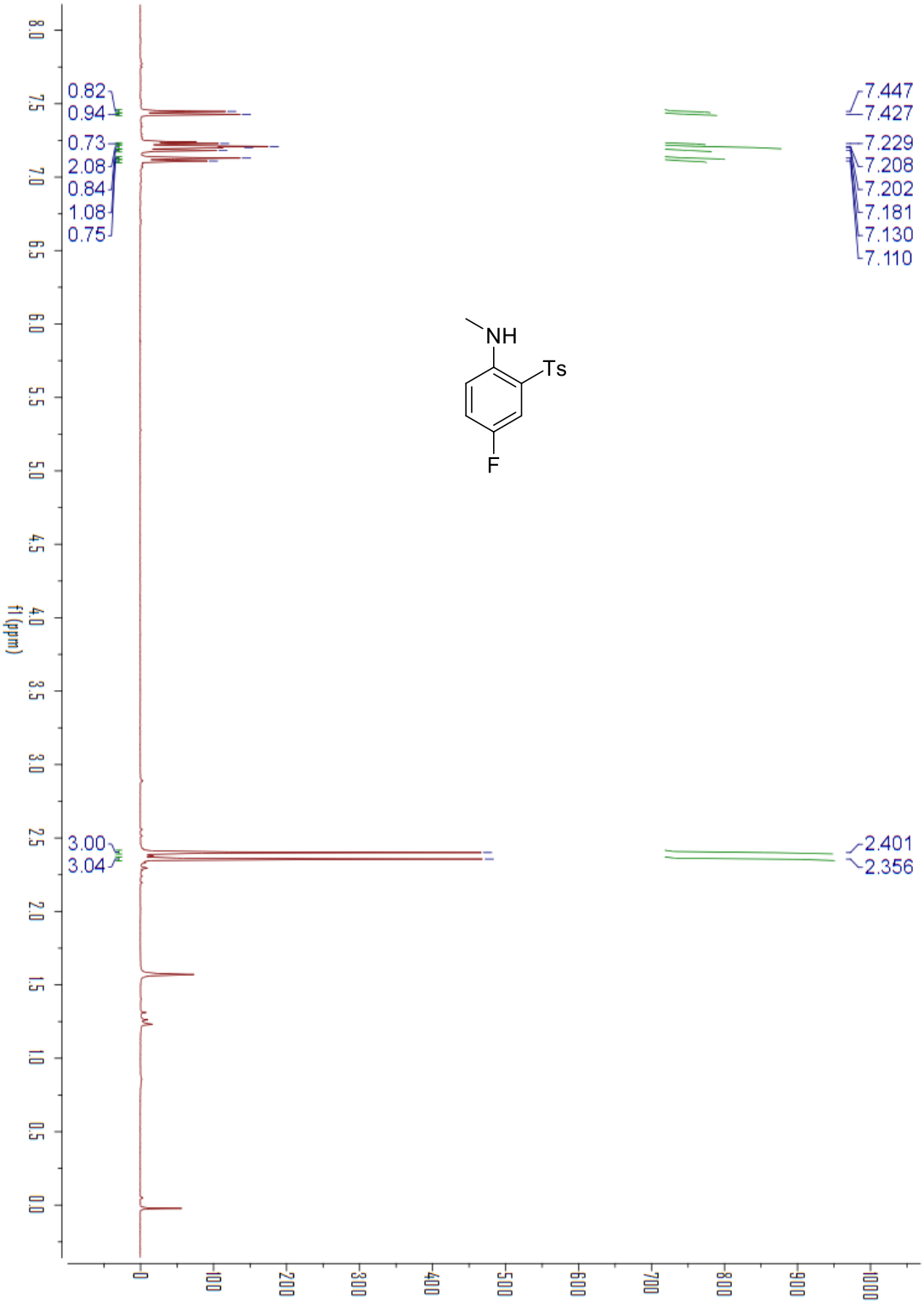

NH
O2
S
3q
146.069
143.930
136.396
130.103
129.832
127.003
125.435
112.114
109.869
30.408
21.796
f1 (ppm)

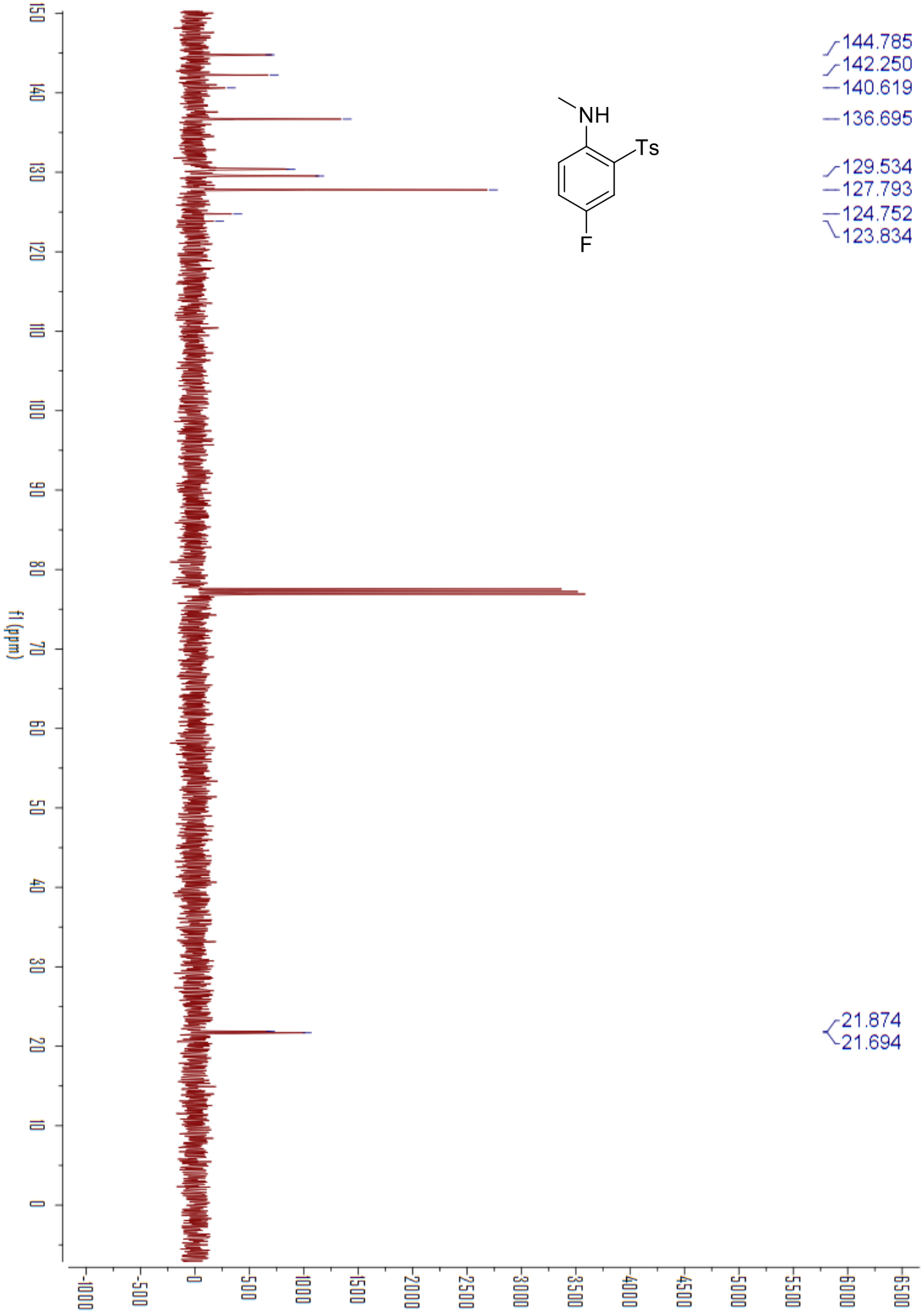

NH
Ts
3r'
7.823
7.801
7.753
7.732
7.272
7.251
6.637
6.615
4.034
3.250
3.232
3.214
3.196
2.399
1.287
1.270
1.252
1.90
1.63
1.66
1.74
0.66
1.77
3.00
3.32
f1 (ppm)

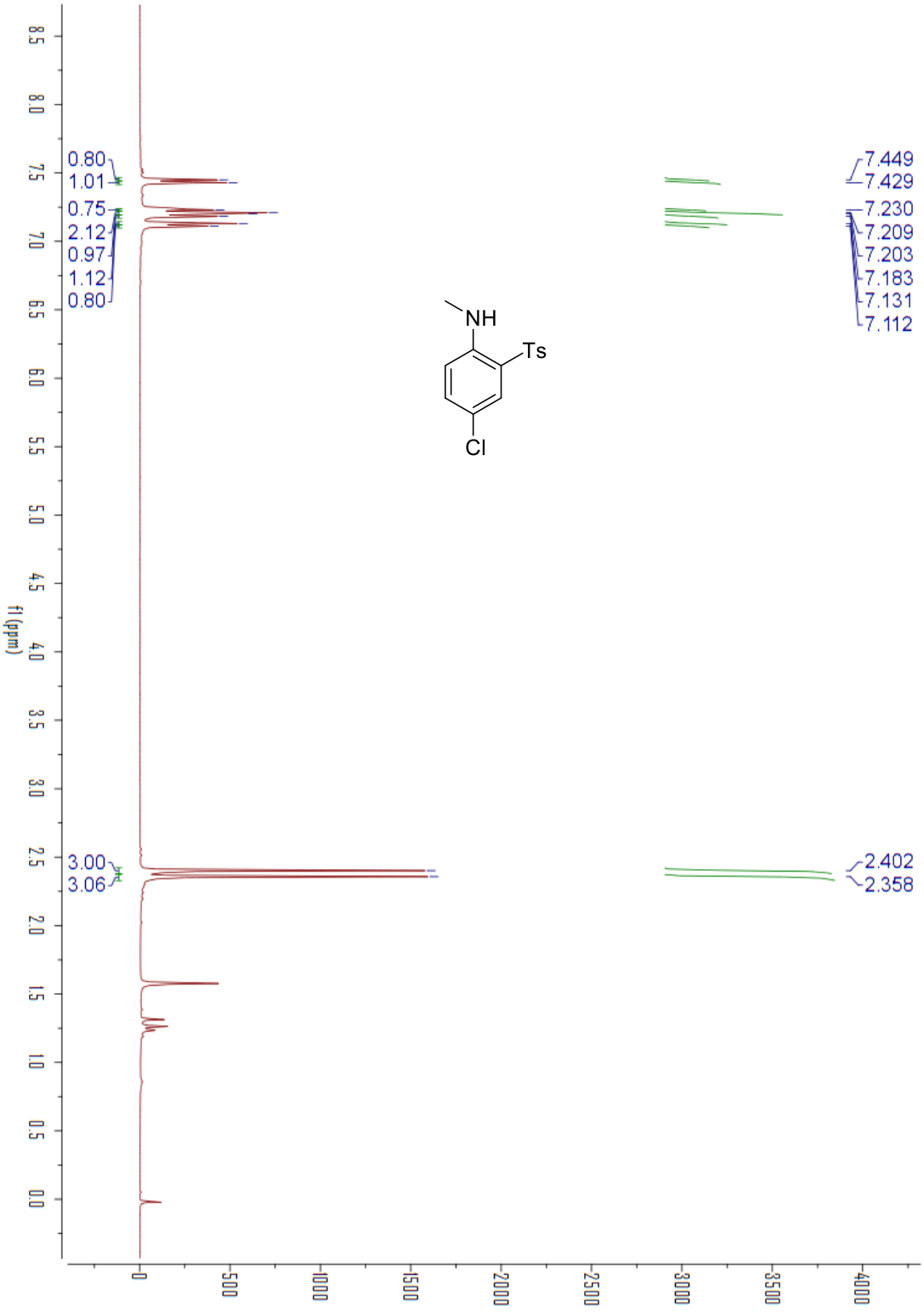

151.349
150.993
144.825
139.963
129.796
127.882
125.289
122.386
112.283
38.384
21.618
14.949
NH
Ts
3r'
f1 (ppm)

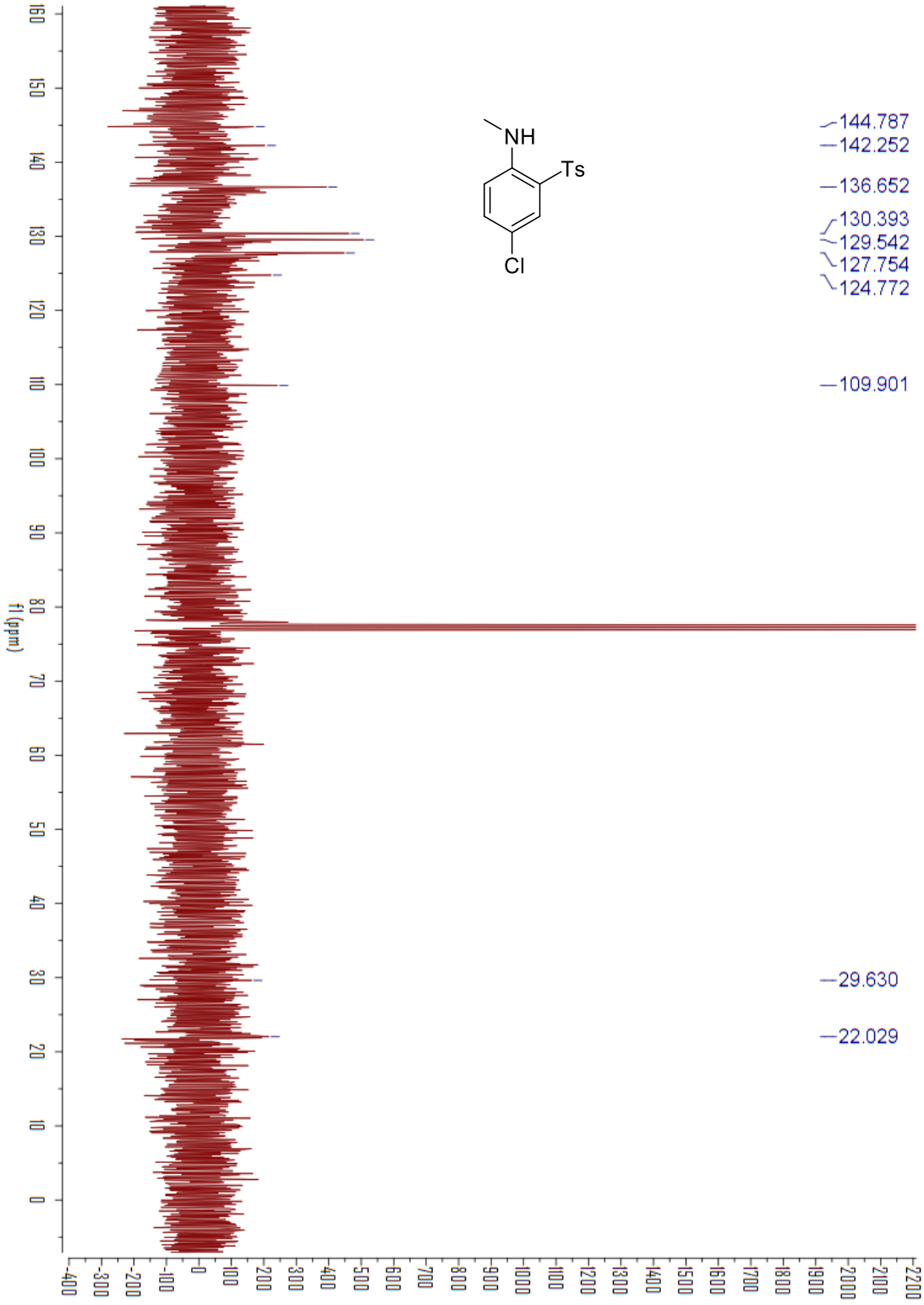

0.95
1.80
0.90
1.79
6.22
6.00
7.181
7.142
7.081
6.932
2.290
2.242
N
O2
S
F
f1 (ppm)


142.866
138.922
133.240
127.592
125.478
125.384
109.867
40.848
21.299
f1 (ppm)


0.82
0.94
0.73
2.08
0.84
1.08
0.75
3.00
3.04
7.447
7.427
7.229
7.208
7.202
7.181
7.130
7.110
2.401
2.356
NH
Ts
F
f1 (ppm)


144.785
142.250
140.619
136.695
129.534
127.793
124.752
123.834
21.874
21.694
NH
Ts
F
f1 (ppm)


0.80
1.01
0.75
2.12
0.97
1.12
0.80
3.00
3.06
7.449
7.429
7.230
7.209
7.203
7.183
7.131
7.112
2.402
2.358
NH
Ts
Cl
f1 (ppm)


NH
Ts
Cl
144.787
142.252
136.652
130.393
129.542
127.754
124.772
109.901
29.630
22.029
f1 (ppm)