Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

A two-step tandem reaction to prepare hydroxamic acids directly from alcohols

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Experimental Section:

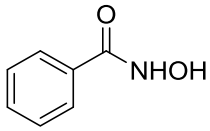
General information

All reagents and solvents were as obtained by commercial source. All the reactions were carried out under Argon atmosphere using standard techniques. Column chromatography was generally performed on silica gel (pore size 60 A°, 32-63 mm particle size) and reactions were monitored by thin-layer chromatography (TLC) analysis was performed with Merck Kieselgel 60 F254 plates and visualized using UV light at 254 nm and $KMnO_4$ staining. ^{1}H NMR and ^{13}C NMR spectra were measured on a Bruker Avance III 400 spectrometer (400 MHz or 100 MHz, respectively) with d^6-DMSO as solvent and reported in ppm relative to d^6-DMSO (^{1}H δ 2.50, ^{13}C δ 39.5). The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet; dd doublet of doublets; br, broad. The coupling constants, J, are reported in Hertz (Hz). The IR spectra were recorded on a Jasco FTIR-480 Plus Fourier Transform spectrometer. High resolution mass spectra HRMS (ESI) were recorded on a Q-Tof mass spectrometer. Melting points were determined in open capillary tubes and are uncorrected.

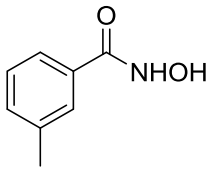
General procedure:

To a solution of *N*-hydroxysuccinimide (0.550 g, 4.77 mmol), iodobenzene diacetate (3.07 g, 9.54 mmol) in MeCN (6 mL) at 0°C under Argon, alcohol **1** (4.34 mmol) was added in one portion. The reaction mixture was stirred for 1h at 0°C, and then at room temperature for 2h. An aqueous solution of 50% NH_2OH (0.53 ml, 8.68 mmol) was slowly added. The reaction mixture was stirred for 12 h. The solvent was removed under vacuum, and the residue purified by flash chromatography (hexanes : EtOAc).

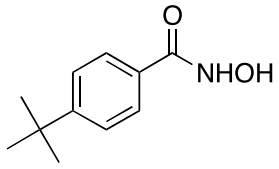
Compound characterizations



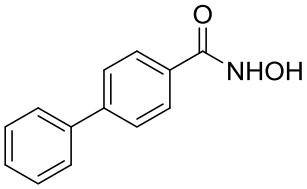
N-hydroxybenzamide (2a). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.31, hexanes : EtOAc, 3:7) in 82% yield (0.488 g, 3.56 mmol) as a white solid, mp 126–128 °C;[1] known compound. The NMR spectroscopic data agree with those described in ref.[1] ^{1}H NMR (400 MHz, DMSO) δ 11.26 (s, 1H), 9.07 (s, 1H), 7.81 – 7.79 (m, 2H), 7.59 - 7.49 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.23, 132.81, 131.13, 128.37, 126.87. IR (nujol) ν = 3297, 2925, 1733, 1646, 1569, 1461, 1377, 1315, 1164, 1077, 1040, 1023, 899, 796.[1]



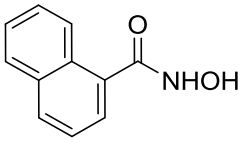
N-hydroxy-3-methylbenzamide (2b). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.36, hexanes : EtOAc, 3:7) in 87% yield (0.570 g, 3.77 mmol) as a white solid, mp 113–115 °C.[2] The ^{1}H NMR agree with those described in ref.[3] ^{1}H NMR (400 MHz, DMSO) δ 11.15 (br, s, 1H), 8.99 (br, s, 1H), 7.58 (s, 1H), 7.53 (t, *J* = 4.3 Hz, 1H), 7.33 (d, *J* = 4.7 Hz, 2H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 164.29, 137.60, 132.74, 131.64, 128.20, 127.43, 123.88, 20.90. IR (nujol) ν = 3302, 3204, 2953, 2924, 1626, 1576, 1541, 1459, 1377, 1286, 1138, 1043, 809, 687.[20] HRMS (ESI) calcd for $C_8H_9NO_2$ $[M+H]^+$: 152,0712, found. 152,0709.



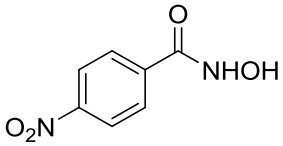
4-tert-butyl-N-hydroxybenzamide (2c). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.307, hexanes : EtOAc, 1:1) in 79% yield (0.662 g, 3.43 mmol) as a white solid, mp 143–145 °C;[4] known compound. The NMR spectroscopic data agree with those described in ref.[5, 21] ^{1}H NMR (400 MHz, DMSO) δ 11.13 (br, s, 1H), 8.97 (br, s, 1H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.47 (d, *J* = 8.3 Hz, 2H), 1.29 (s, 9H). ^{13}C NMR (100 MHz, DMSO) δ 164.20, 153.84, 130.00, 126.66, 125.09, 34.55, 30.89. IR (nujol) ν = 3158, 2924, 1734, 1607, 1542, 1457, 1377, 1325, 1164, 1013, 899, 848, 729.



N-hydroxybiphenyl-4-carboxamide (2d). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.57, hexanes : EtOAc, 3:7) in 69% yield (0.638 g, 2.99 mmol) as a white solid, mp 198–200 °C;[2] known compound. The NMR spectroscopic data agree with those described in ref.[6, 2] ^{1}H NMR (400 MHz, DMSO) δ 11.28 (br, s, 1H), 9.07 (br, s, 1H), 7.86 (d, *J* = 8.2 Hz, 2H), 7.76 (d, *J* = 8.3 Hz, 2H), 7.72 (d, *J* = 7.7 Hz, 2H), 7.45 (dt, *J* = 34.1, 7.3 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 163.86, 142.66, 139.15, 131.57, 129.00, 128.00, 127.49, 126.81, 126.57. . IR (nujol) ν = 3299, 2926, 1650, 1616, 1578, 1556, 1459, 1377, 1164, 1078, 899, 850, 777, 741.[2]

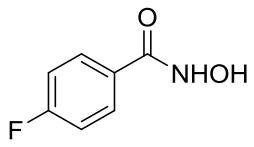


N-hydroxy-1-naphthamide (2e). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.35, hexanes : EtOAc, 4:6) in 67% yield (0.544 g, 2.90 mmol) as a white solid, mp 185–186 °C;[7] known compound. The NMR spectroscopic data agree with those described in ref.[8] ^{1}H NMR (400 MHz, DMSO) δ 11.10 (s, 1H), 9.22 (s, 1H), 8.18 – 8.04 (m, 1H), 8.02 – 7.97 (m, 2H), 7.61 – 7.52 (m, 4H). ^{13}C NMR (100 MHz, DMSO) δ 165.63, 133.10, 132.23, 130.04, 129.97, 128.20, 126.73, 126.27, 125.43, 125.15, 124.95. IR (nujol) ν = 3276, 2924, 1961, 1608, 1460, 1377, 1344, 1319, 1259, 1159, 1064, 898, 867, 778, 741.

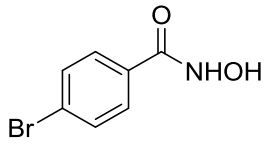


N-hydroxy-4-nitrobenzamide (2f). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.36, hexanes : EtOAc, 3:7) in 79% yield (0.624 g, 3.43 mmol) as a white solid, mp 186–189 °C;[1] known compound. The NMR spectroscopic data agree with those described in ref.[1] ^{1}H NMR (400 MHz, DMSO) δ 11.55 (br, s, 1H), 9.32 (br, s, 1H), 8.31 (d, *J* = 8.7 Hz, 2H), 7.99 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (100 MHz, DMSO) δ 162.29,

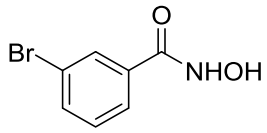
148.94, 138.50, 128.36, 123.58. IR (nujol) ν = 3243, 2954, 2924, 2853, 1656, 1598, 1513, 1461, 1377, 1298, 1154, 1032, 1013, 897, 783.[1]



4-fluoro-N-hydroxybenzamide (2g). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.33, hexanes : EtOAc, 1:1) in 92% yield (0.619 g, 3.99 mmol) as a white solid, mp 163–164 °C;[9] known compound. The NMR spectroscopic data agree with those described in ref.[10] ^{1}H NMR (400 MHz, DMSO) δ 11.23 (br, s, 1H), 9.06 (br, s, 1H), 7.82 (dd, *J* = 8.5, 5.7 Hz, 2H), 7.29 (t, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, DMSO) δ 163.78 (d, *J* = 247 Hz), 163.22, 129.45 (d, *J* = 9 Hz), 129.26 (d, *J* = 3 Hz), 115.35 (d, *J* = 22 Hz). IR (nujol) ν = 3297, 2926, 1613, 1579, 1508, 1462, 1377, 1334, 1245, 1156, 903, 849, 752, 722.[11]

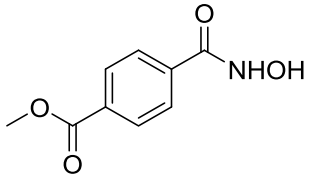


4-bromo-N-hydroxybenzamide (2h). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.33, hexanes : EtOAc, 1:1) in 85% yield (0,937 g, 3.69 mmol) as a white solid, mp 185–186 °C;[12] known compound. The NMR spectroscopic data agree with those described in ref.[12] ^{1}H NMR (400 MHz, DMSO) δ 11.30 (br, s, 1H), 9.11 (br, s, 1H), 7.70 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, DMSO) δ 163.21, 131.87, 131.39, 128.94, 124.77. IR (nujol) ν = 3295, 2923, 1650, 1559, 1459, 1377, 1162, 1075, 901, 844, 741.[12]

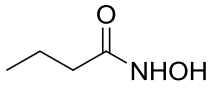


3-bromo-N-hydroxybenzamide (2i). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.33, hexanes : EtOAc, 1:1) in 87% yield (0.815 g, 3.77 mmol) as a white solid, mp 177–179 °C.[13] The ^{1}H NMR agree with those described

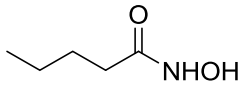
in ref.[3] ^{1}H NMR (400 MHz, DMSO) δ 11.34 (br, s, 1H), 9.17 (br, s, 1H), 7.92 (s, 1H), 7.74 (dd, *J* = 13.5, 8.0 Hz, 2H), 7.43 (t, *J* = 7.9 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 162.54, 134.92, 133.87, 130.66, 129.52, 125.91, 121.66. IR (nujol) ν = 3297, 2899, 1941, 1803, 1622, 1563, 1457, 1377, 1270, 1165, 1045, 997, 896, 723. HRMS (ESI) calcd for $C_7H_6BrNO_2$ $[M+H]^+$: 215,9660, found. 215,9656.



Methyl 4-(hydroxycarbamoyl)benzoate (2j). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.43, hexanes : EtOAc, 3:7) in 93% yield (0.787 g, 3.17 mmol) as a white solid, mp 197–199 °C; known compound. The NMR spectroscopic data agree with those described in ref.[14] ^{1}H NMR (400 MHz, DMSO) δ 11.40 (br, s, 1H), 9.19 (br, s, 1H), 8.03 (d, *J* = 8.3 Hz, 2H), 7.88 (d, *J* = 8.3 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 165.64, 163.20, 136.92, 131.69, 129.16, 127.25, 52.32. IR (nujol) ν = 3297, 2924, 1927, 1726, 1627, 1564, 1456, 1377, 1279, 1108, 1013, 867, 714.

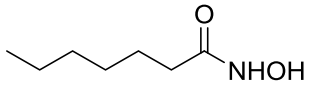


N-hydroxybutyramide (2k). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.35, hexanes : EtOAc, 3:7) in 94% yield (0.42 g, 4.07 mmol) as a yellow oil; known compound. The NMR spectroscopic data agree with those described in ref.[15] ^{1}H NMR (400 MHz, DMSO) δ 10.32 (s, 1H), 8.65 (s, 1H), 1.92 (t, *J* = 7.3 Hz, 2H), 1.53 – 1.48 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 168.92, 34.18, 18.49, 13.51. IR (nujol) ν = 3205, 2924, 1639, 1535, 1458, 1377, 1215, 1107, 1030, 972.[16]

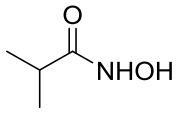


N-hydroxypentanamide (2l). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.273, hexanes : EtOAc, 3:7) in 85% yield (0.432 g, 3.69 mmol) as a yellow solid, mp 52–54 °C;[17] known compound. The NMR spectroscopic data agree with those described in ref.[14] ^{1}H NMR (400 MHz, DMSO) δ 10.33 (br, s, 1H), 8.65 (br, s, 1H), 1.94 (t, *J* = 6.4 Hz, 2H), 1.46 (dd, *J* = 14.0, 7.0 Hz, 2H), 1.25 (dt, *J* = 14.0, 7.0 Hz, 2H), 0.86 (t, *J* =

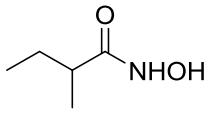
6.4 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 169.09, 31.92, 27.21, 21.67, 13.60. IR (nujol) ν = 3260, 2924, 1667, 1464, 1377, 1116, 1069, 973, 728, 647.



N-hydroxyheptanamide (2m). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.37, hexanes : EtOAc, 3:7) in 87% yield (0.548 g, 3.77 mmol) as a white solid, mp 70–73 °C;[12] known compound. The NMR spectroscopic data agree with those described in ref.[12, 16] ^{1}H NMR (400 MHz, DMSO) δ 10.32 (br, s, 1H), 8.65 (br, s, 1H), 1.93 (t, *J* = 7.4 Hz, 2H), 1.48 – 1.46 (m, 2H), 1.28 – 1.24 (m, 6H), 0.86 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.13, 32.27, 30.98, 28.26, 25.08, 22.01, 13.92. IR (nujol) ν = 3257, 2954, 2925, 2854, 1660, 1464, 1377, 1313, 1120, 1074, 1017, 723, 559.[12]

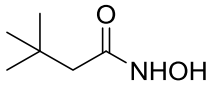


N-hydroxyisobutyramide (2n). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.263, hexanes : EtOAc, 3:7) in 83% yield (0.371 g, 3.59 mmol) as a white solid, mp 118–119 °C;[1] known compound. The NMR spectroscopic data agree with those described in ref.[1] ^{1}H NMR (400 MHz, DMSO) δ 10.35 (br, s, 1H), 8.63 (br, s, 1H), 2.22 (dt, *J* = 13.7, 6.8 Hz, 1H), 0.98, 1.00 (2s, 6H). ^{13}C NMR (100 MHz, DMSO) δ 173.05, 31.11, 19.41. IR (nujol) ν = 3160, 2924, 2853, 1619, 1542, 1460, 1377, 1095, 954, 869.[1]

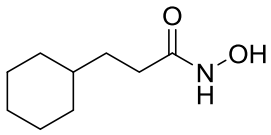


N-hydroxy-2-methylbutanamide (2o). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.33, hexanes : EtOAc, 3:7) in 81% yield (0.411 g, 3.51 mmol) as a white solid, mp 105–106 °C. The ^{1}H NMR agree with those described in ref.[18] ^{1}H NMR (400 MHz, DMSO) δ 10.36 (br, s, 1H), 8.67 (br, s, 1H), 2.03 – 1.94 (m, 1H), 1.51 – 1.41 (m, 1H), 1.35 – 1.22 (m, 1H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.79 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 172.31, 38.35, 26.55, 17.54, 11.75. IR (nujol) ν = 3225, 2944, 1633, 1538, 1459,

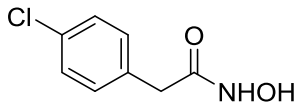
1377, 1289, 1107, 1046, 1008, 940, 845, 675. HRMS (ESI) calcd for $C_5H_{11}NO_2$ $[M+H]^+$: 118,0868, found. 118,0873



N-hydroxy-3,3-dimethylbutanamide (2p). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.18, hexanes : EtOAc, 1:1) in 78% yield (0.443 g, 3.37 mmol) as a white solid, mp 82–84 °C. ^{1}H NMR (400 MHz, DMSO) δ 10.29 (br, s, 1H), 8.67 (br, s, 1H), 1.83 (s, 2H), 0.95 (s, 9H). ^{13}C NMR (100 MHz, DMSO) δ 167.65, 45.49, 30.29, 29.59. IR (nujol) ν = 3308, 2923, 1652, 1463, 1377, 1155, 1065, 1036, 976, 802, 744. HRMS (ESI) calcd for $C_6H_{13}NO_2$ $[M+H]^+$: 132,1025, found. 132,1028.

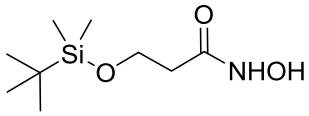


3-cyclohexyl-N-hydroxypropanamide (2q). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.44, hexanes : EtOAc, 3:7) in 67% yield (0.497 g, 2.9 mmol) as a white solid, mp 67–68 °C; known compound. The NMR spectroscopic data agree with those described in ref.[19] ^{1}H NMR (400 MHz, DMSO) δ 10.32 (br, s, 1H), 8.64 (br, s, 1H), 1.95 (t, *J* = 7.7 Hz, 2H), 1.67 -1.60 (m, 3H), 1.41 – 1.09 (m, 6H), 0.88 – 0.80 (m, 2H). ^{13}C NMR (100 MHz, DMSO) δ 169.29, 36.55, 32.59, 32.51, 29.75, 26.08, 25.73. IR (nujol) ν =3250, 2924, 2853, 1786, 1728, 1610, 1577, 1541, 1459, 1377, 1158, 982, 722.



2-(4-chlorophenyl)-N-hydroxyacetamide (2r). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.2, hexanes : EtOAc, 3:7) in 68% yield (0.55 g, 2.95 mmol) as a white solid, mp 146–148 °C.[13] ^{1}H NMR (400 MHz, DMSO) δ 10.66 (br, s, 1H), 8.85 (br, s, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 3.29 (s, 2H). ^{13}C NMR (100 MHz, DMSO) δ 166.61, 135.04, 131.14, 130.75, 128.10, 38.54. IR (nujol) ν = 3175, 2924,

1907, 1732, 1633, 1463, 1376, 1304, 1158, 975, 722. HRMS (ESI) calcd for $C_8H_8ClNO_2$ $[M+H]^+$: 186,0322, found. 186,0319.



3-((tert-butyldimethylsilyl)oxy)-N-hydroxypropanamide (2s). Prepared according to the general procedure. The desired pure product was obtained after column chromatography (R_f = 0.57, hexanes : EtOAc, 1:1) in 73% yield (0.952 g, 3.17 mmol) as a pale yellow solid, mp 40–42 °C. ^{1}H NMR (400 MHz, DMSO) δ 10.26 (br, s, 1H), 8.60 (br, s, 1H), 3.69 (t, *J* = 6.3 Hz, 2H), 2.05 (d, *J* = 6.3 Hz, 2H), 0.76 (s, 9H), -0.07 (s, 6H).^{13}C NMR (100 MHz, DMSO) δ 166.82, 59.19, 36.12, 25.78, 17.95, -5.39. IR (nujol) ν = 3233, 2959, 1728, 1667, 1463, 1377, 1256, 1068, 1007, 933, 838, 773, 721. HRMS (ESI) calcd for $C_9H_{21}NO_3Si$ $[M+H]^+$: 220,1369, found. 220,1365.

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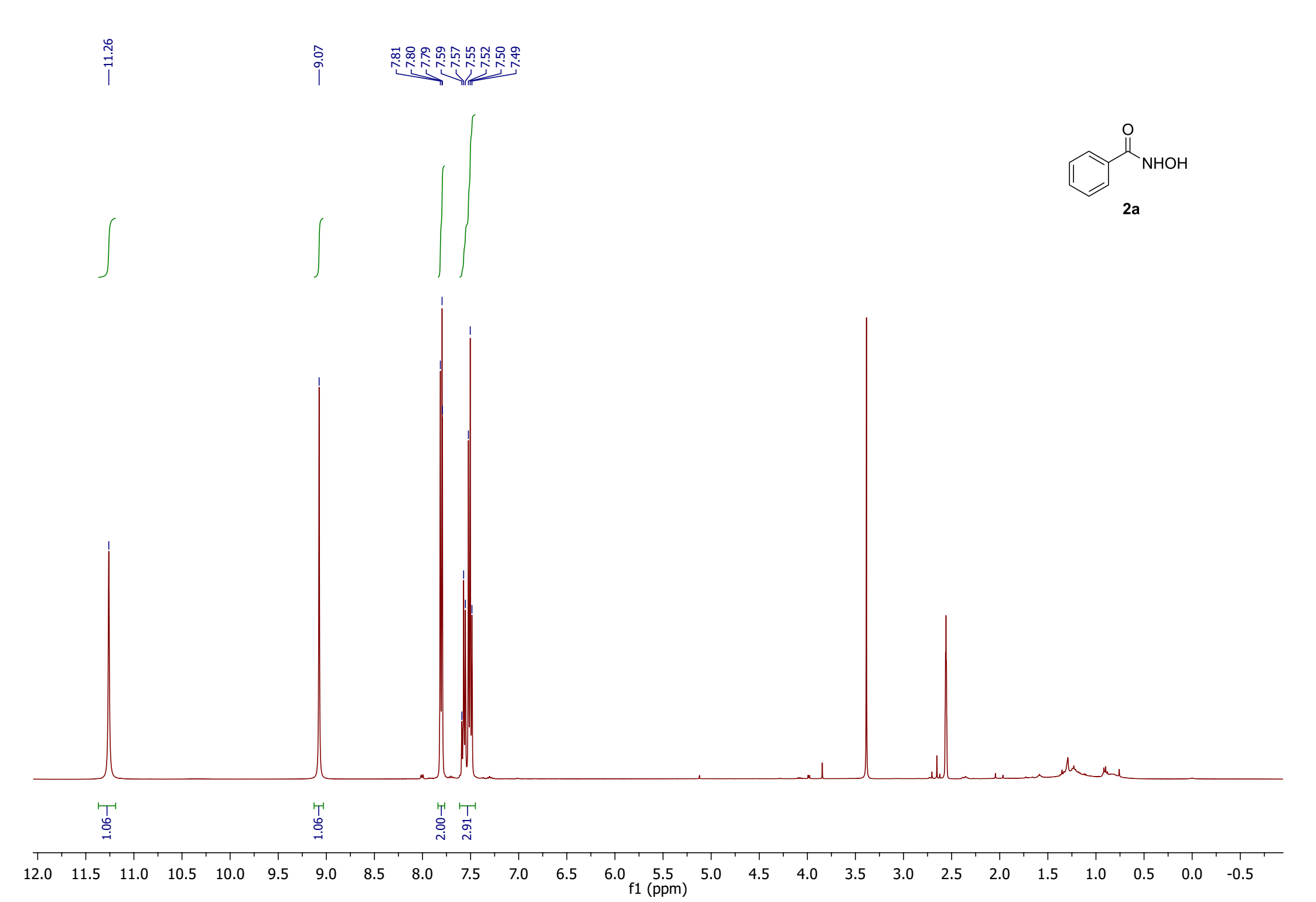
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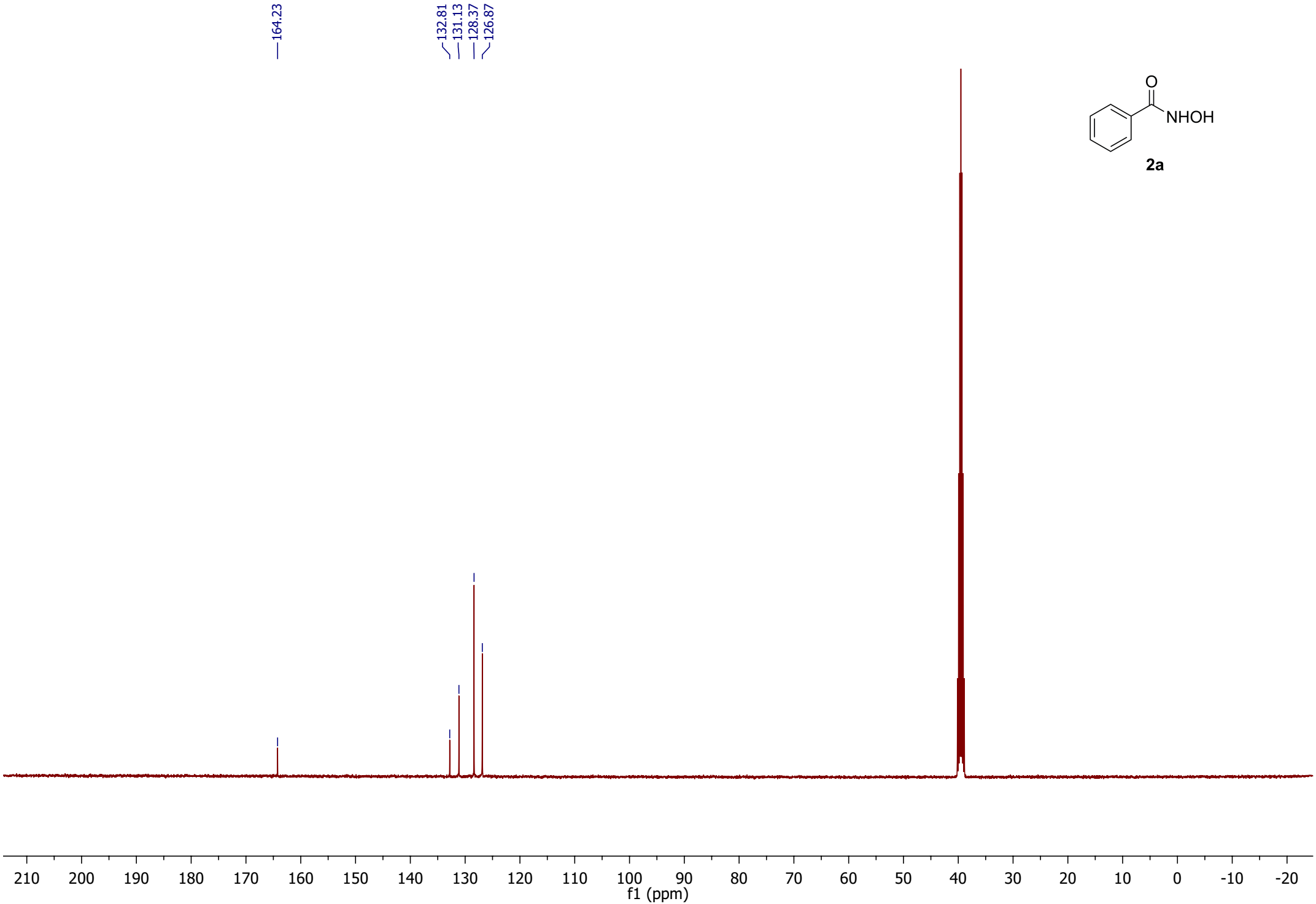
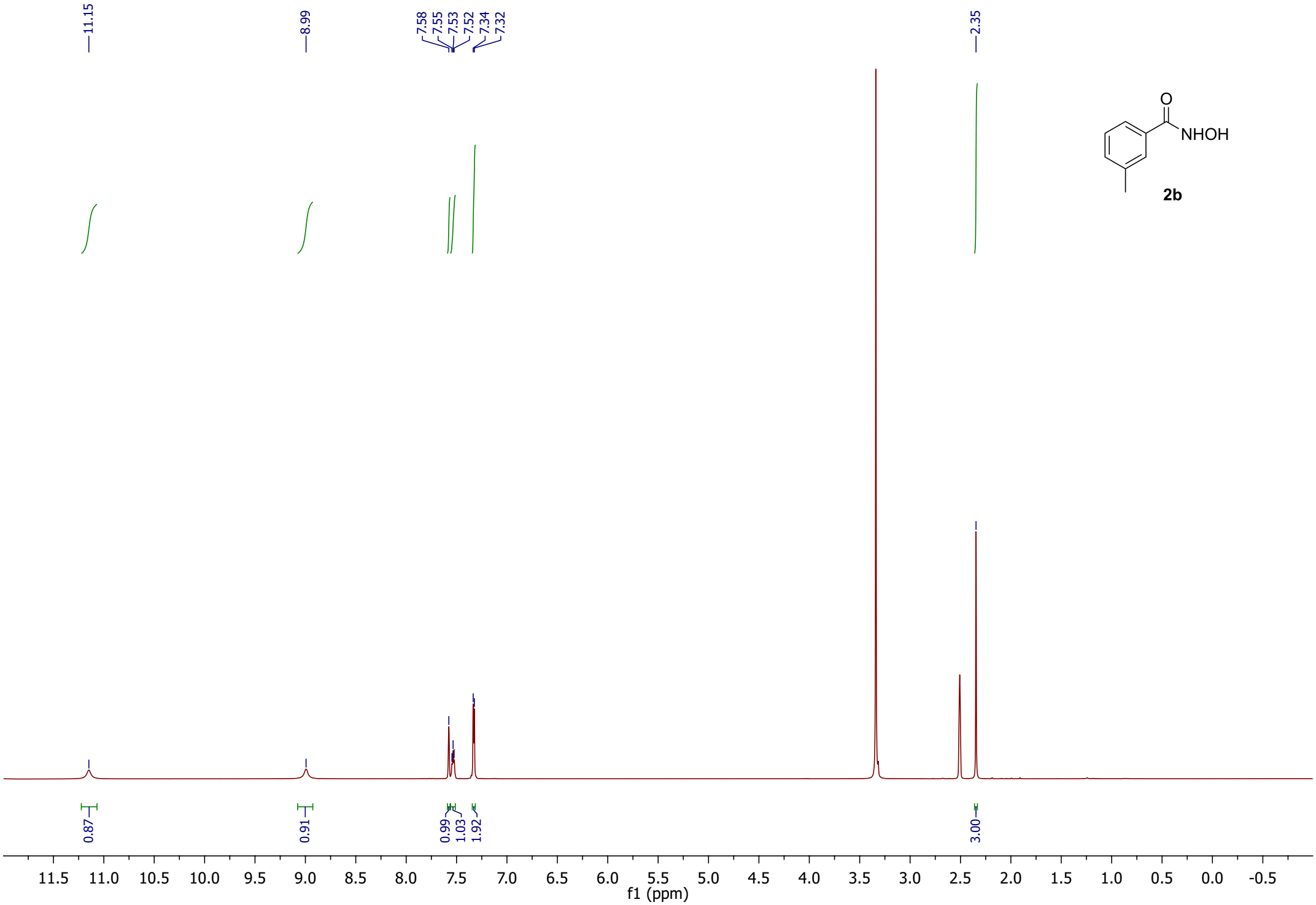
9.07
7.81
7.80
7.79
7.59
7.57
7.55
7.52
7.50
7.49
O
NHOH
2a
1.06
1.06
2.00
2.91
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


164.23
132.81
131.13
128.37
126.87
O
NHOH
2a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)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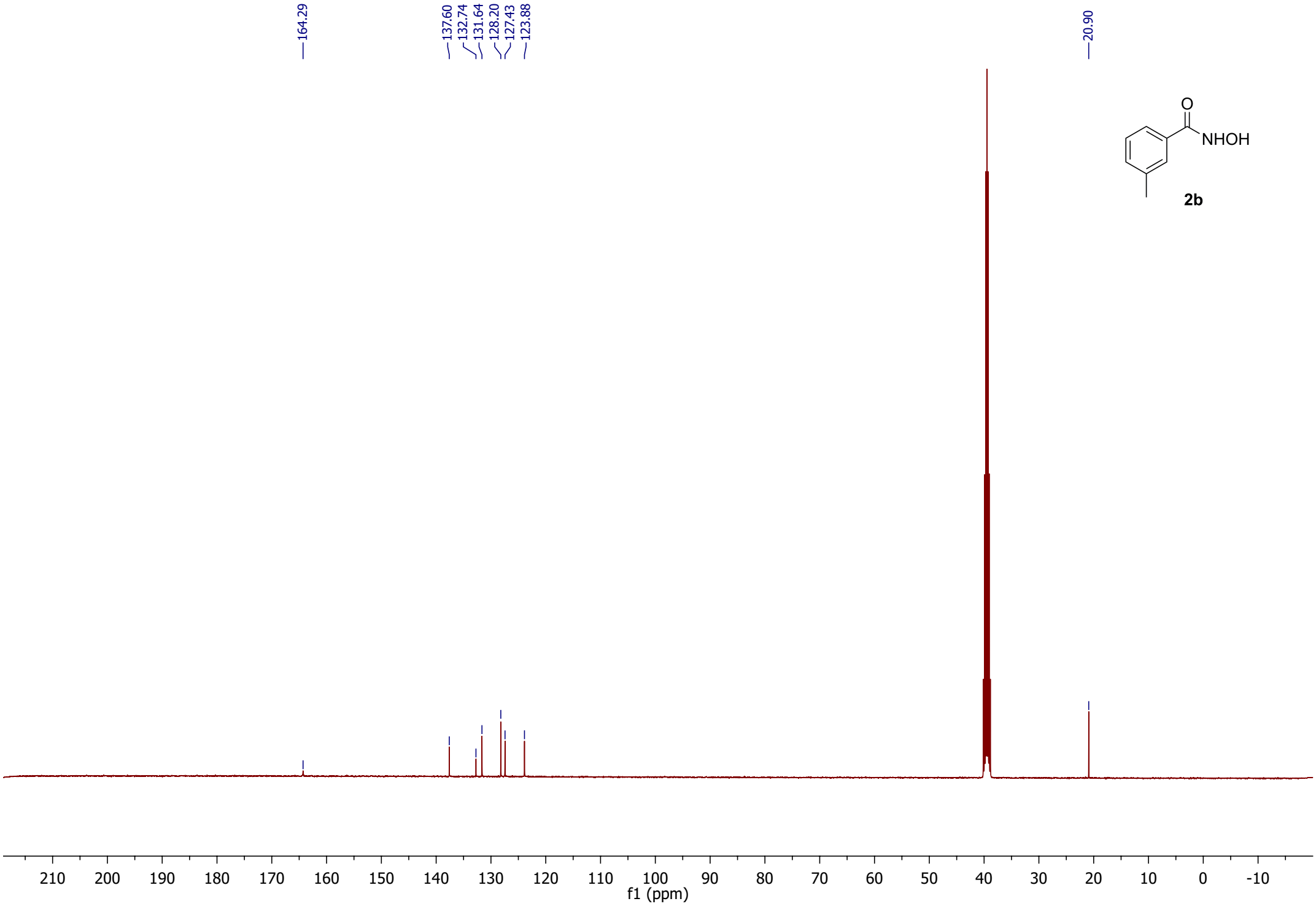
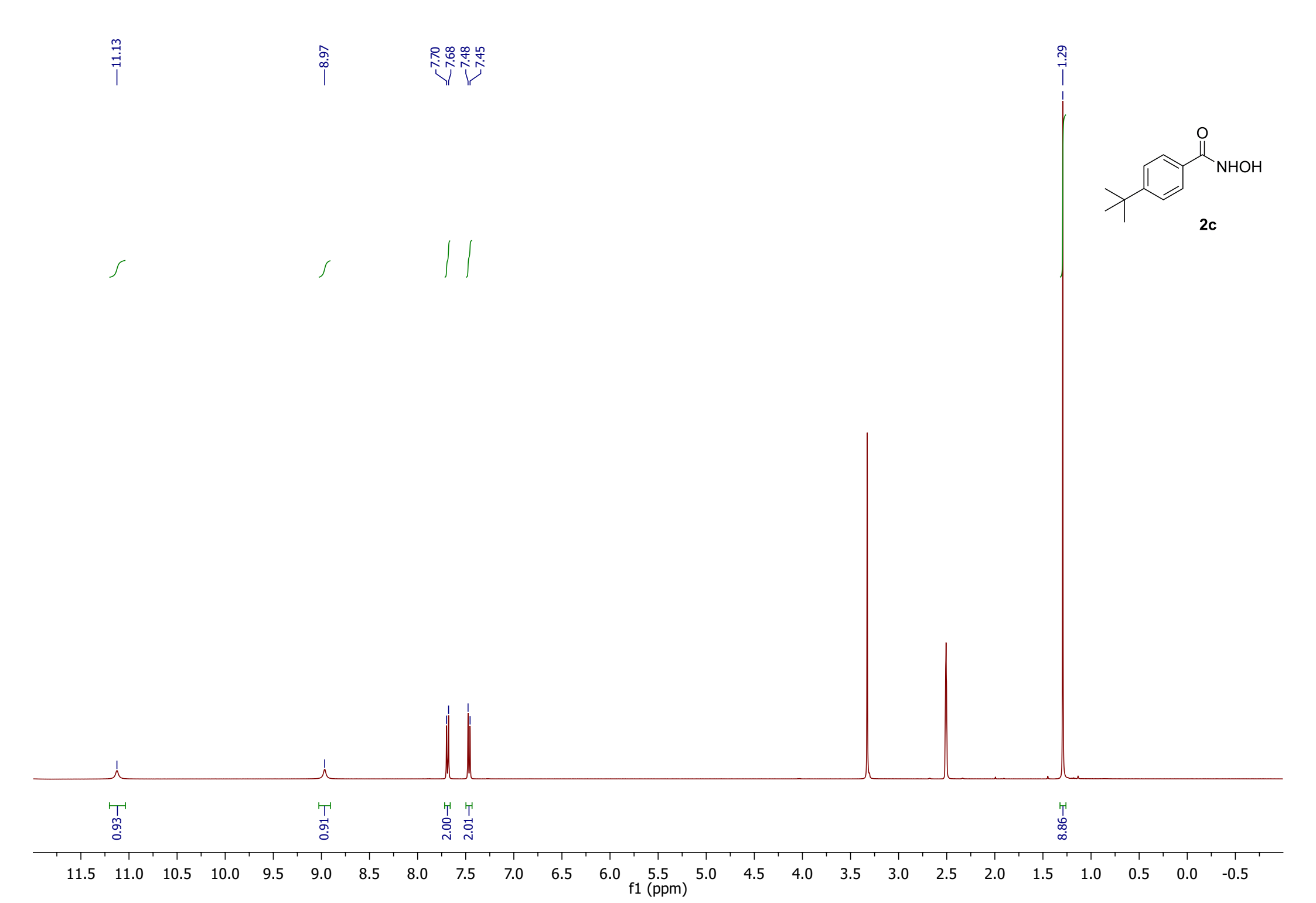
8.99
7.58
7.55
7.53
7.52
7.34
7.32
2.35
O
NHOH
2b
0.87
0.91
0.99
1.03
1.92
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


164.29
137.60
132.74
131.64
128.20
127.43
123.88
20.90
O
NHOH
2b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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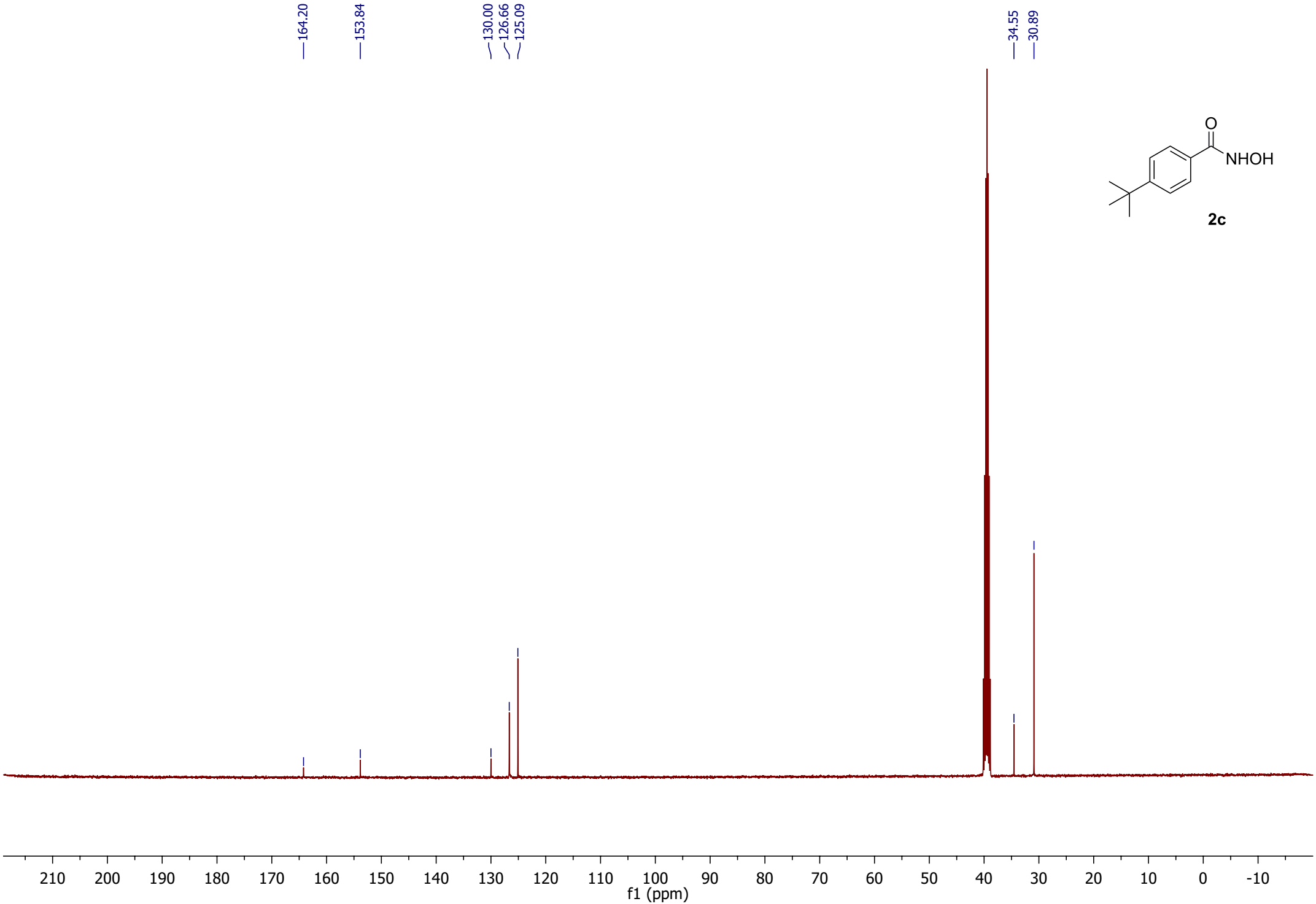
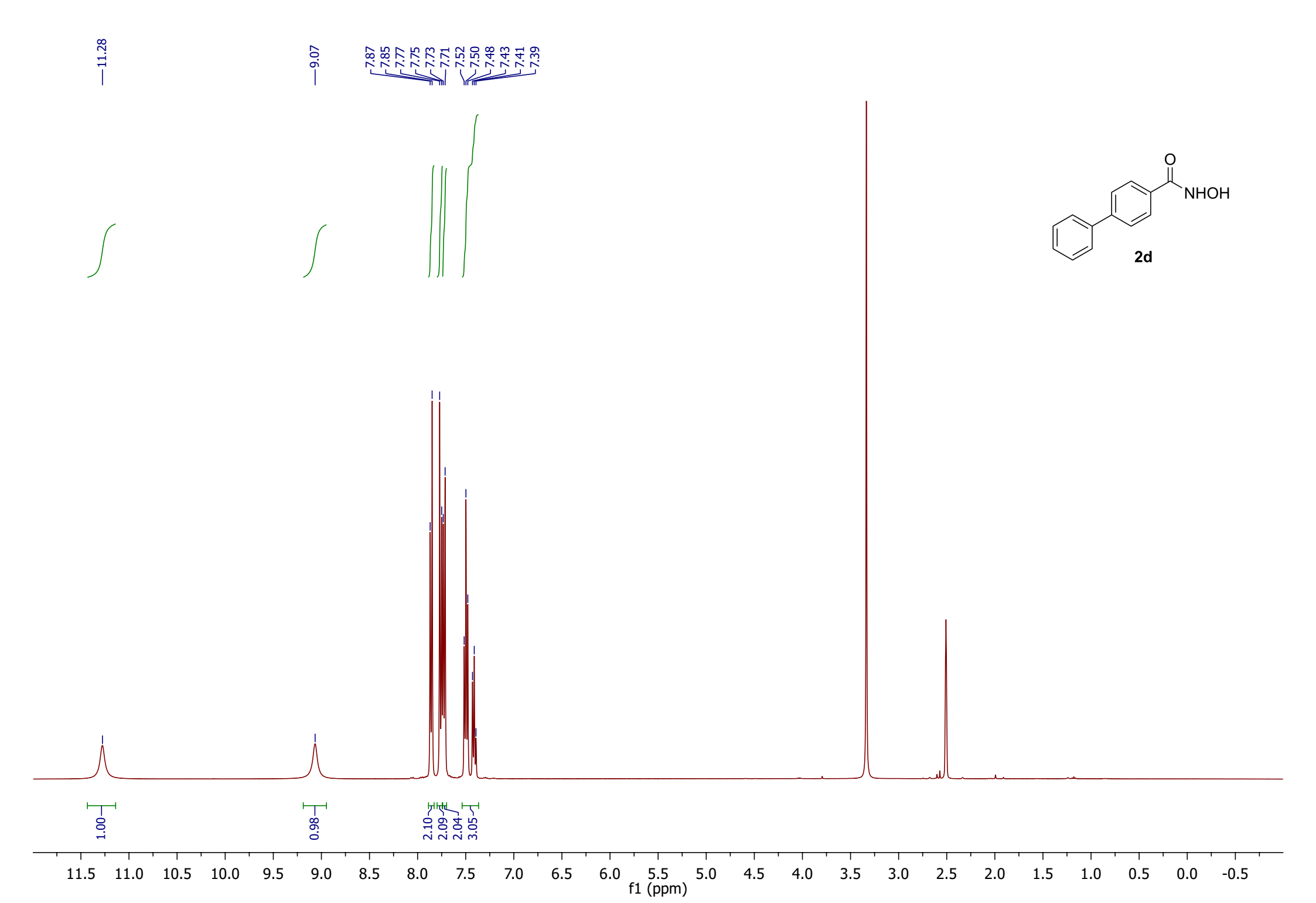
8.97
7.70
7.68
7.48
7.45
1.29
O
NHOH
2c
0.93
0.91
2.00
2.01
8.86
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


164.20
153.84
130.00
126.66
125.09
34.55
30.89
O
NHOH
2c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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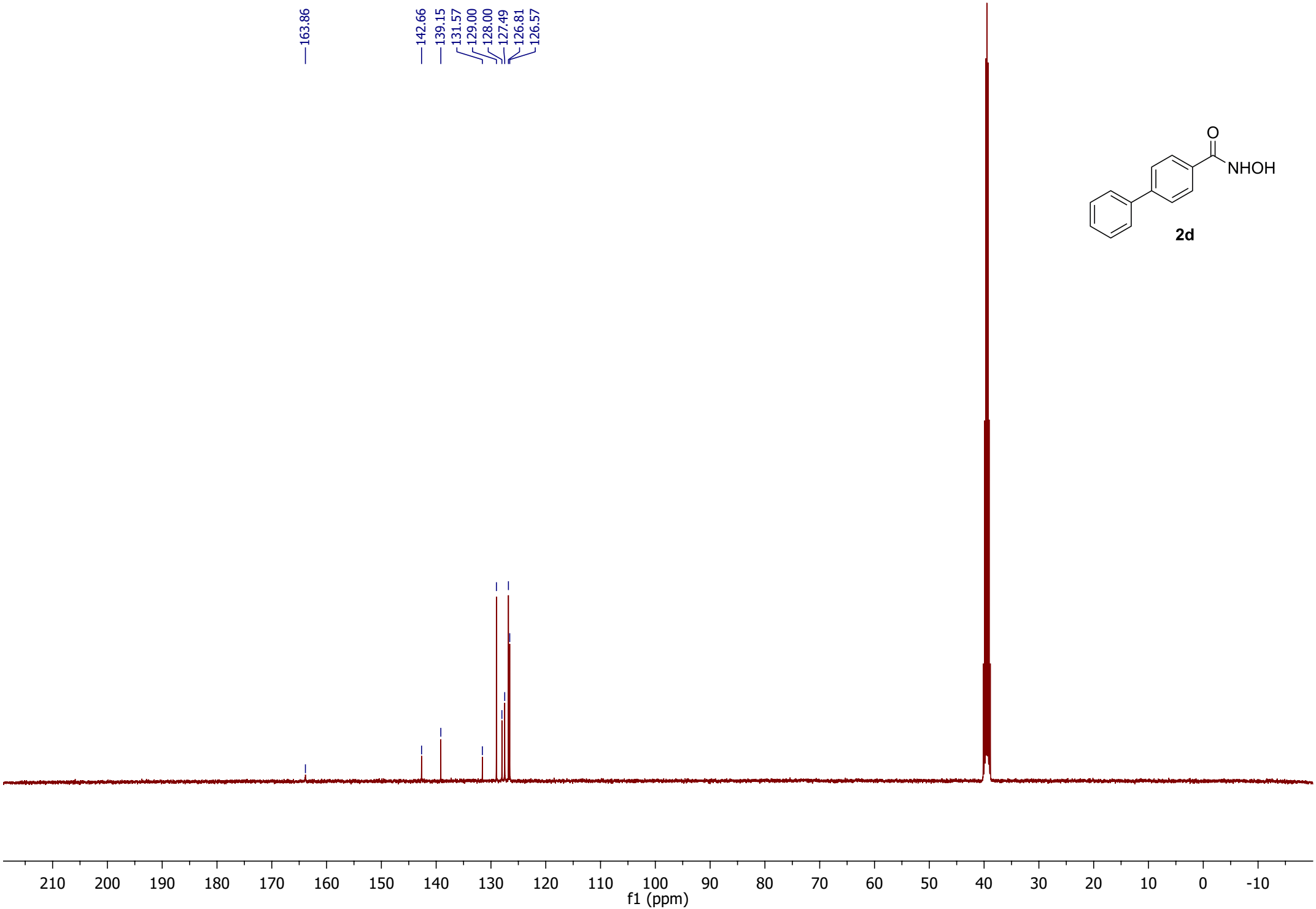
9.07
7.87
7.85
7.77
7.75
7.73
7.71
7.52
7.50
7.48
7.43
7.41
7.39
O
NHOH
2d
1.00
0.98
2.10
2.09
2.04
3.05
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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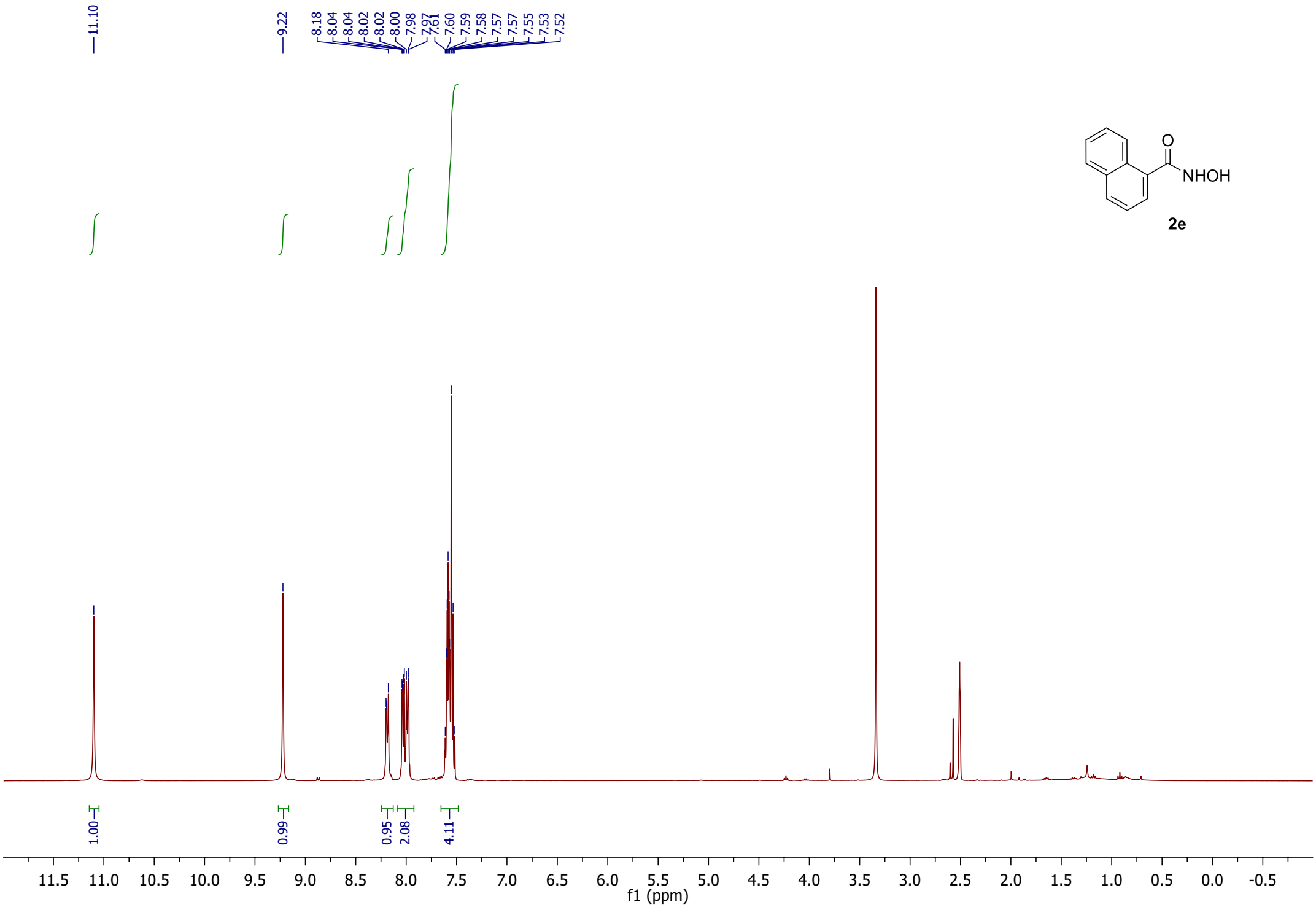
142.66
139.15
131.57
129.00
128.00
127.49
126.81
126.57
O
NHOH
2d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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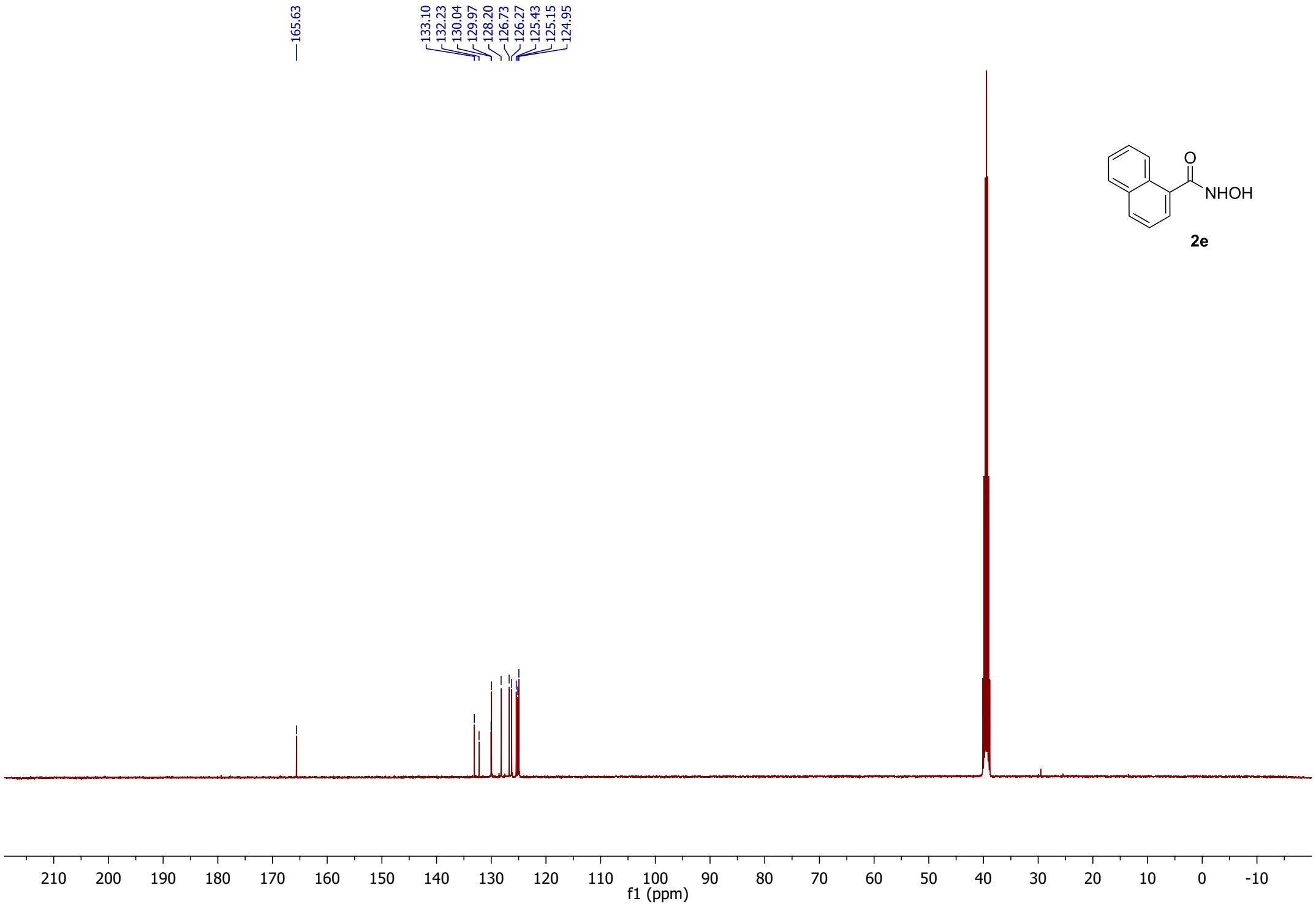
9.22
8.18
8.04
8.04
8.02
8.02
8.00
7.98
7.97
7.61
7.60
7.59
7.58
7.57
7.57
7.55
7.53
7.52
O
NHOH
2e
1.00
0.99
0.95
2.08
4.11
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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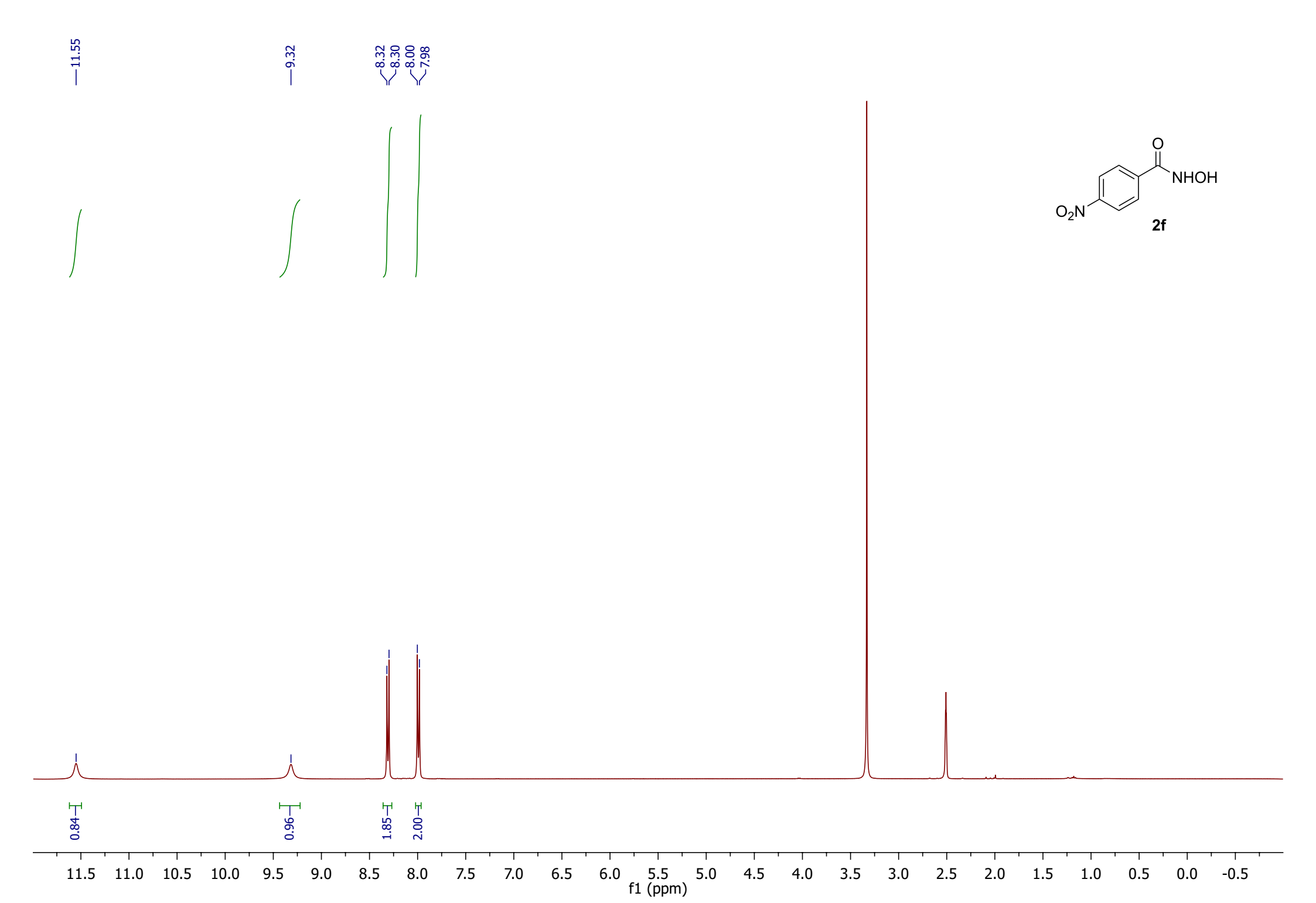
133.10
132.23
130.04
129.97
128.20
126.73
126.27
125.43
125.15
124.95
O
NHOH
2e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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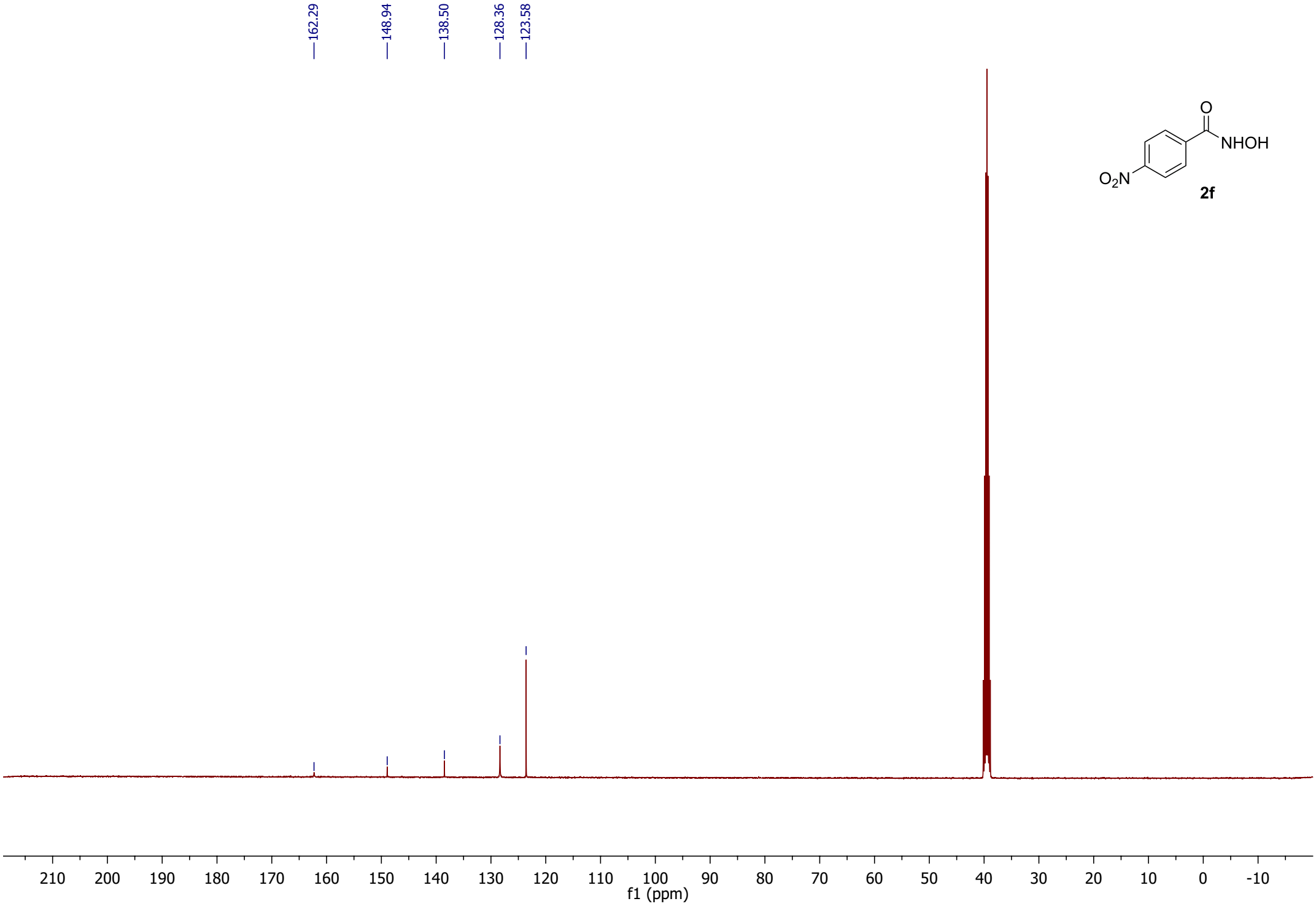
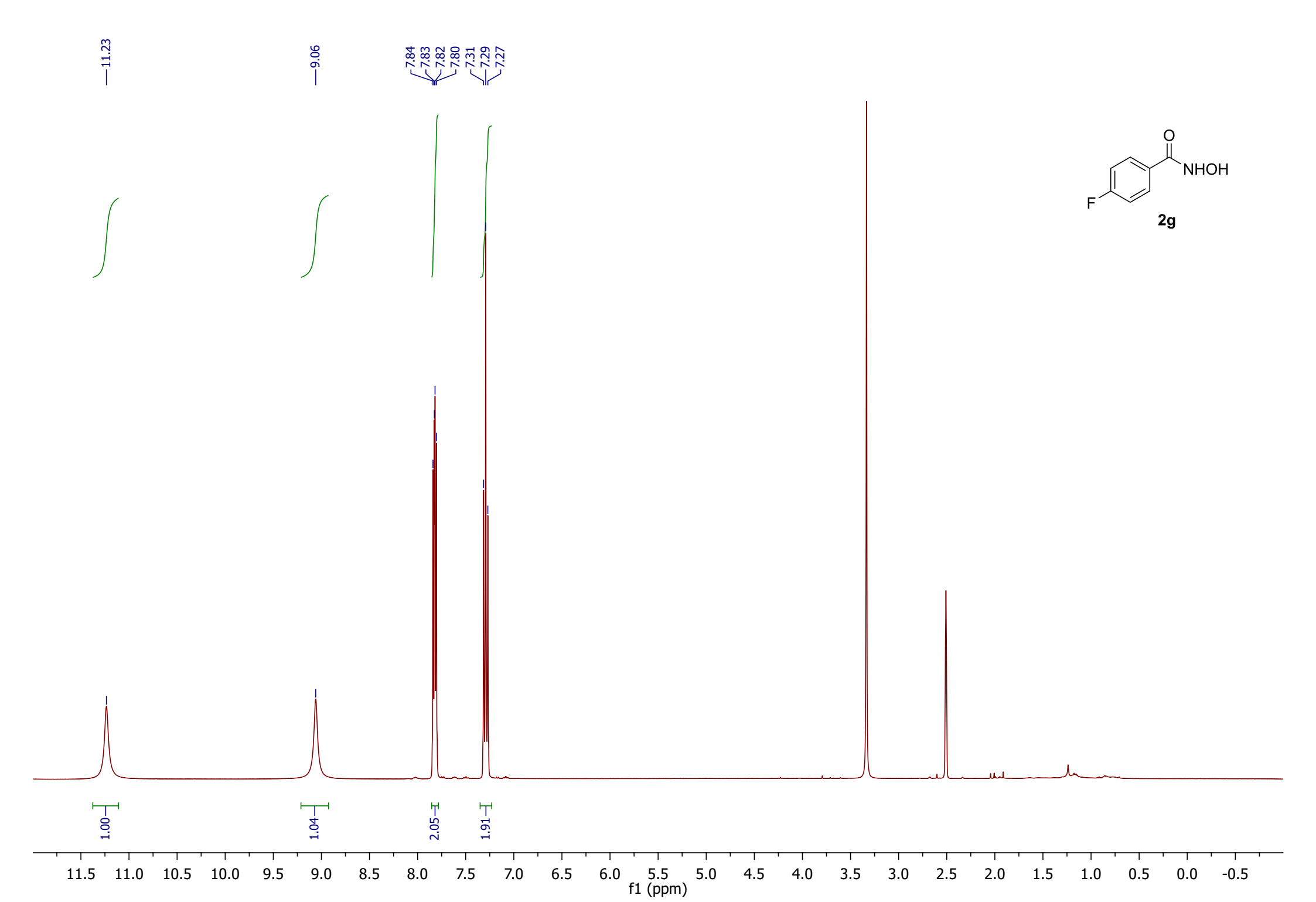
8.32
8.30
8.00
7.98
O
NHOH
O2N
2f
0.84
0.96
1.85
2.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


162.29
148.94
138.50
128.36
123.58
O
NHOH
O2N
2f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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9.06
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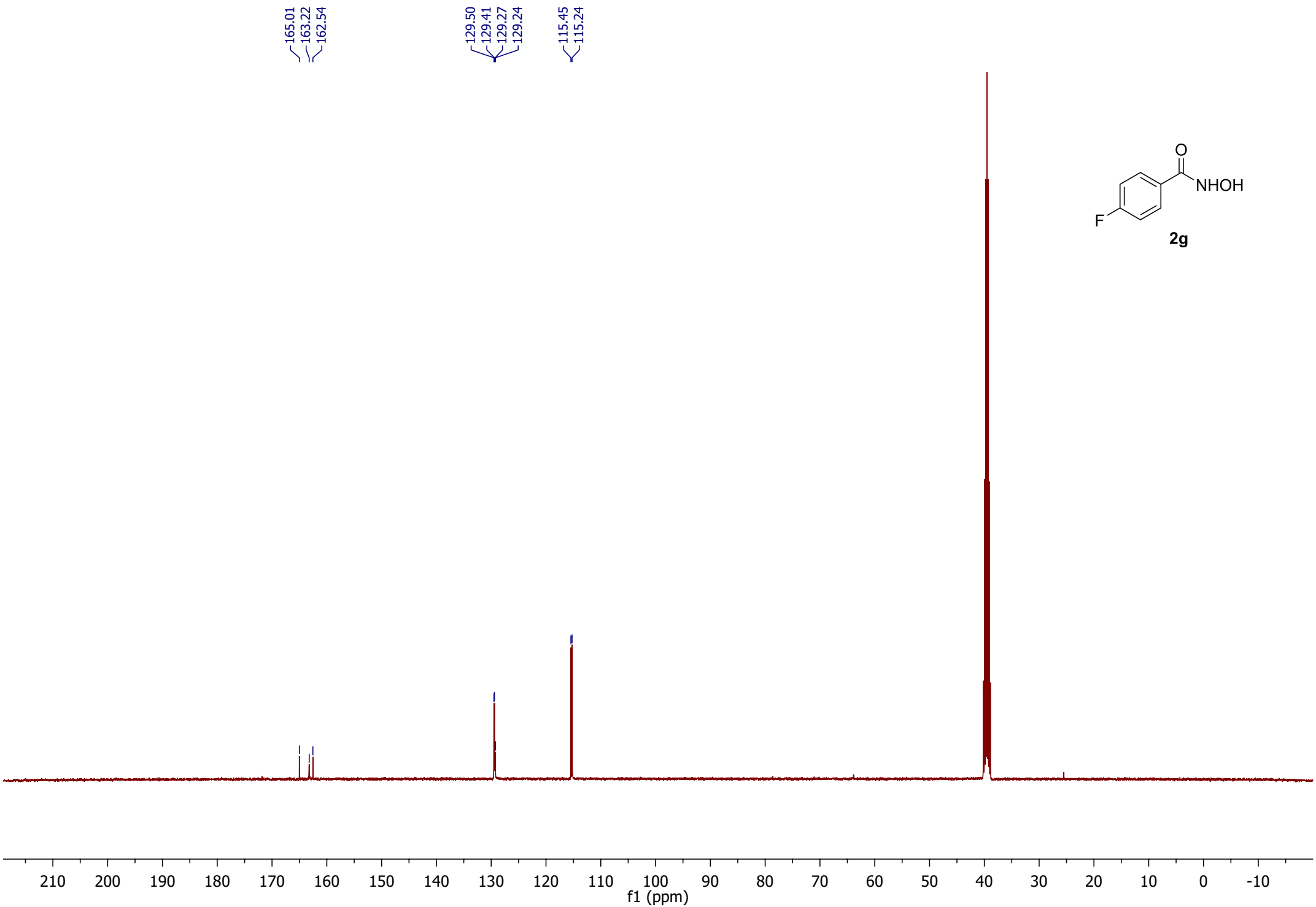
7.83
7.82
7.80
7.31
7.29
7.27
O
NHOH
F
2g
1.00
1.04
2.05
1.91
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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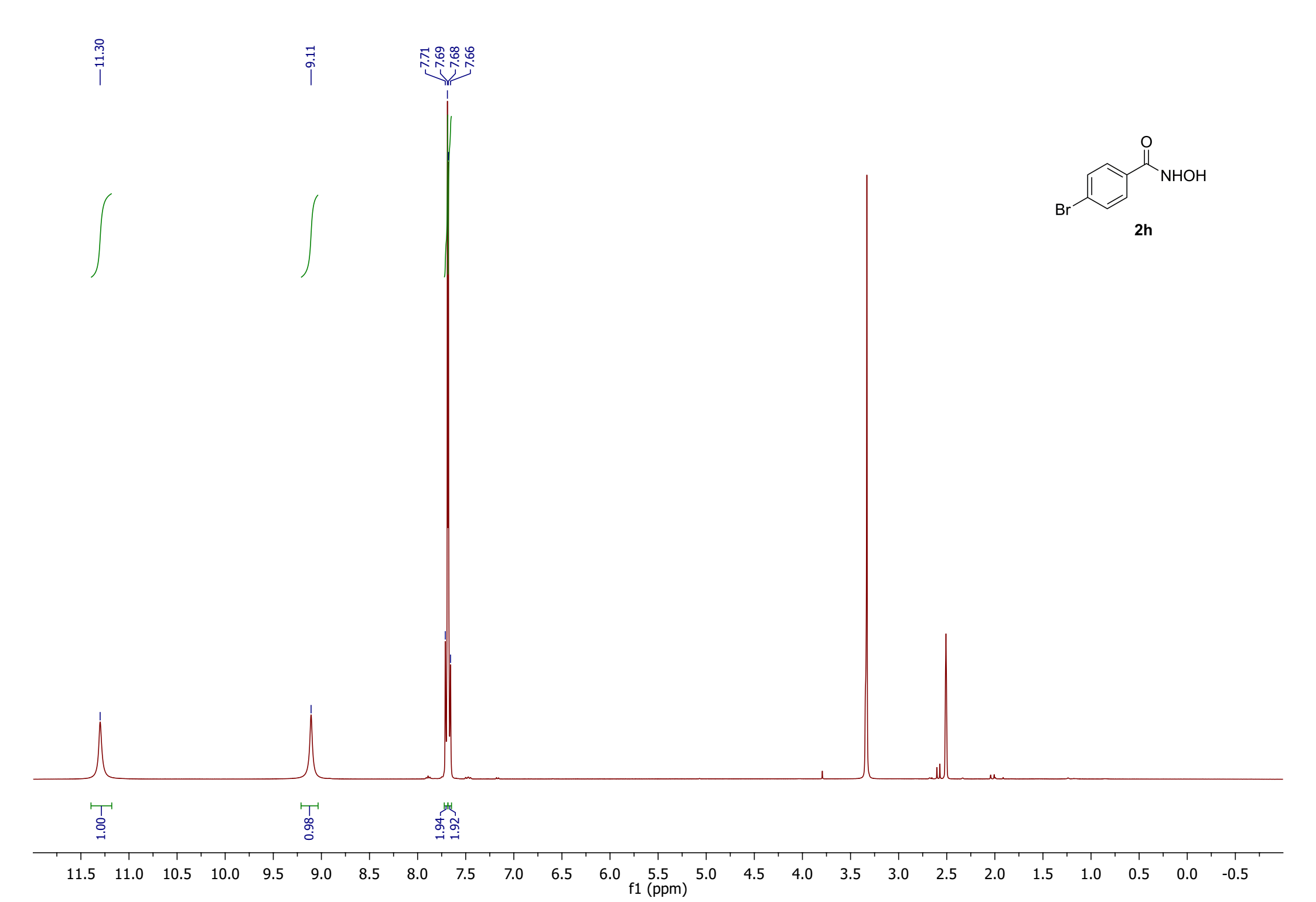
162.54
129.50
129.41
129.27
129.24
115.45
115.24
O
NHOH
F
2g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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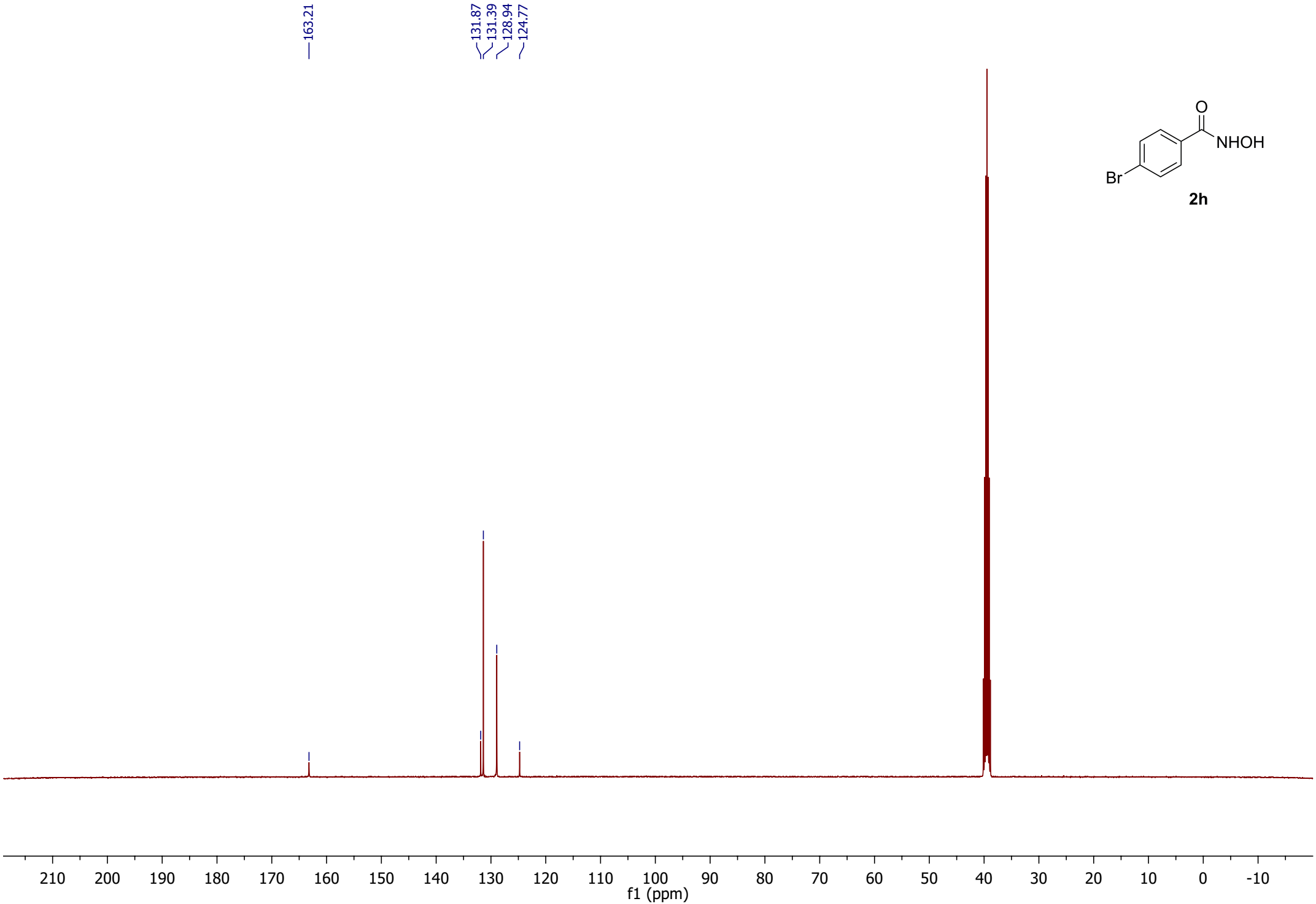
9.11
7.71
7.69
7.68
7.66
O
NHOH
Br
2h
1.00
0.98
1.94
1.92
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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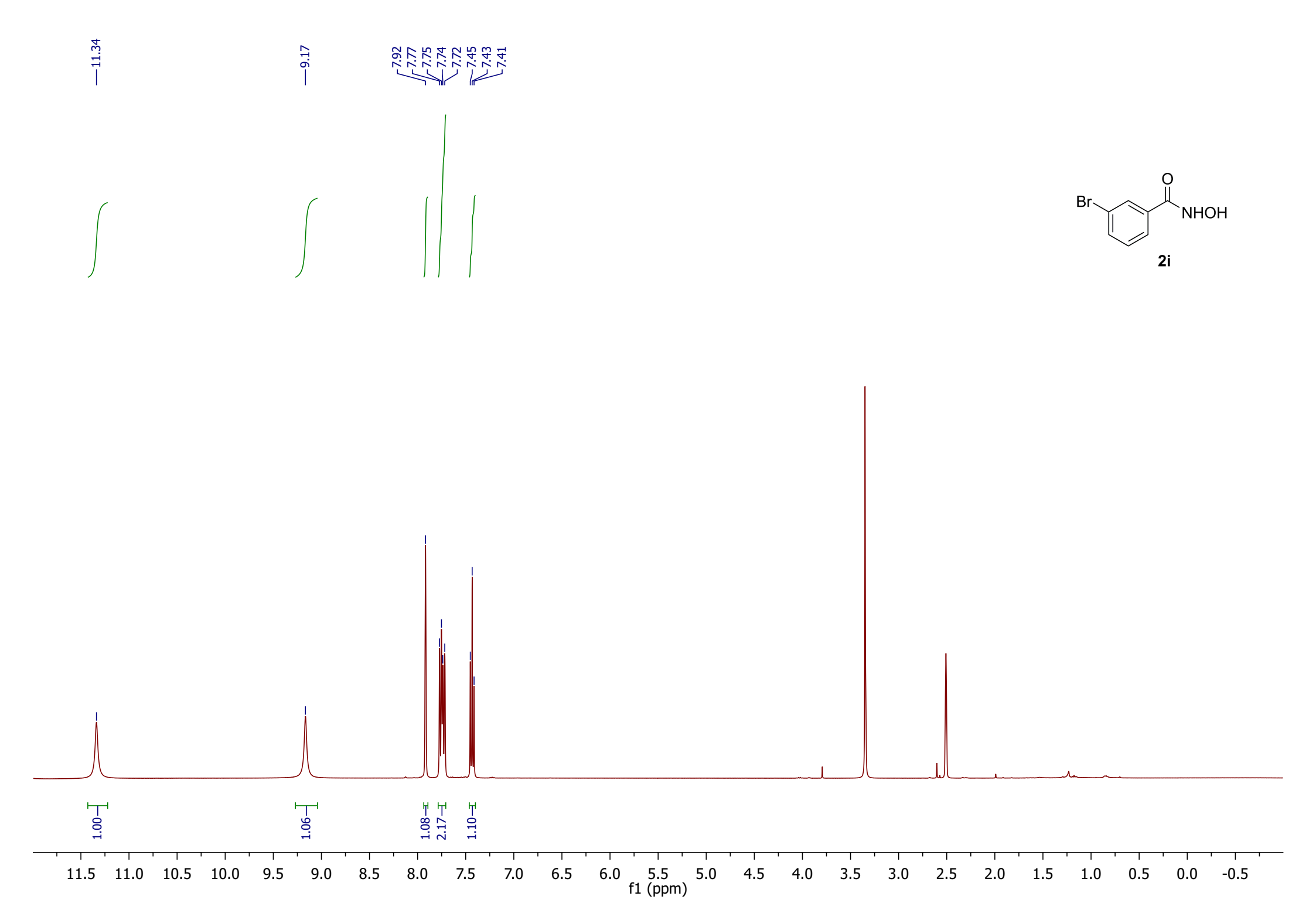
NHOH
Br
2h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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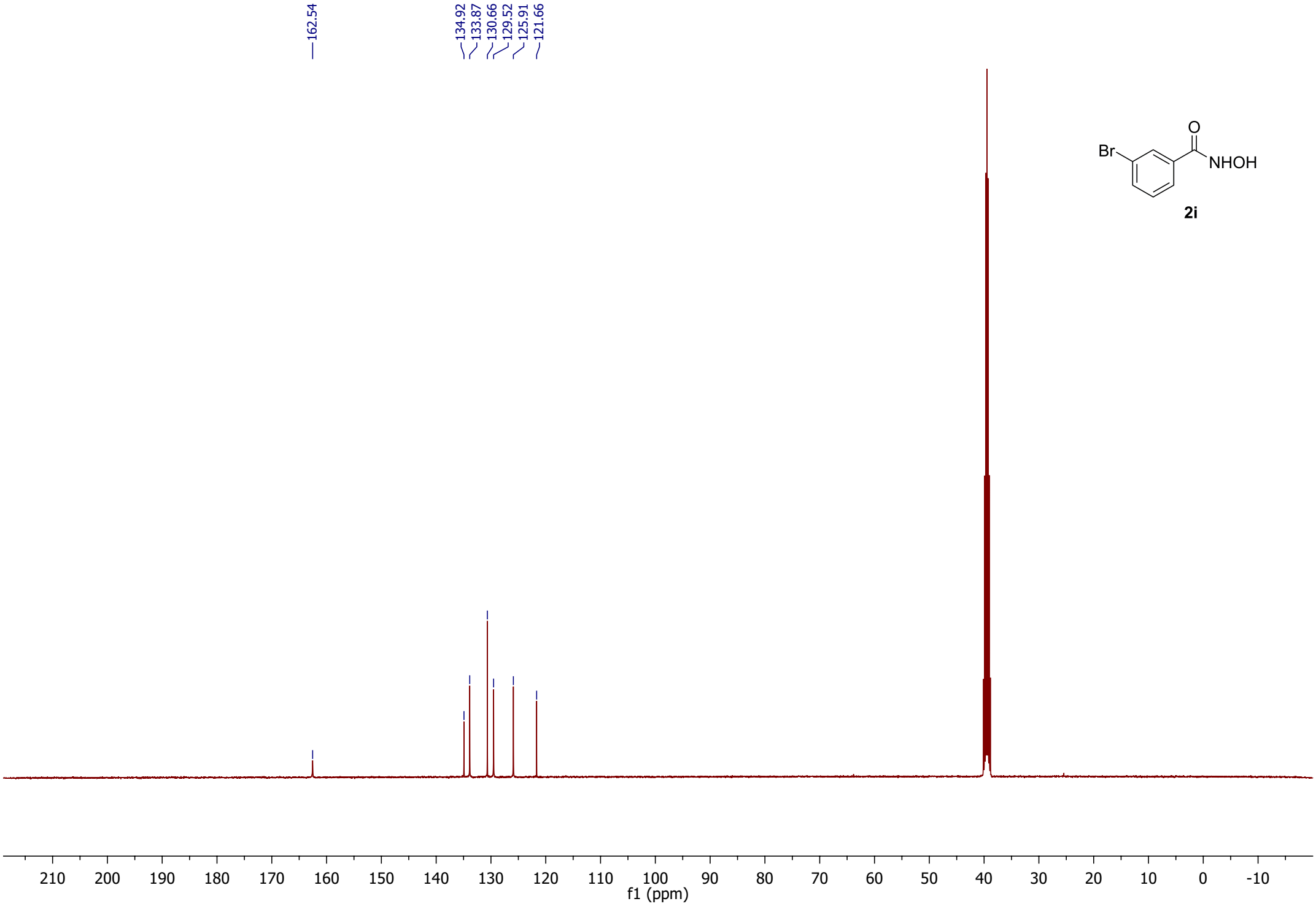
7.74
7.72
7.45
7.43
7.41
Br
O
NHOH
2i
1.00
1.06
1.08
2.17
1.10
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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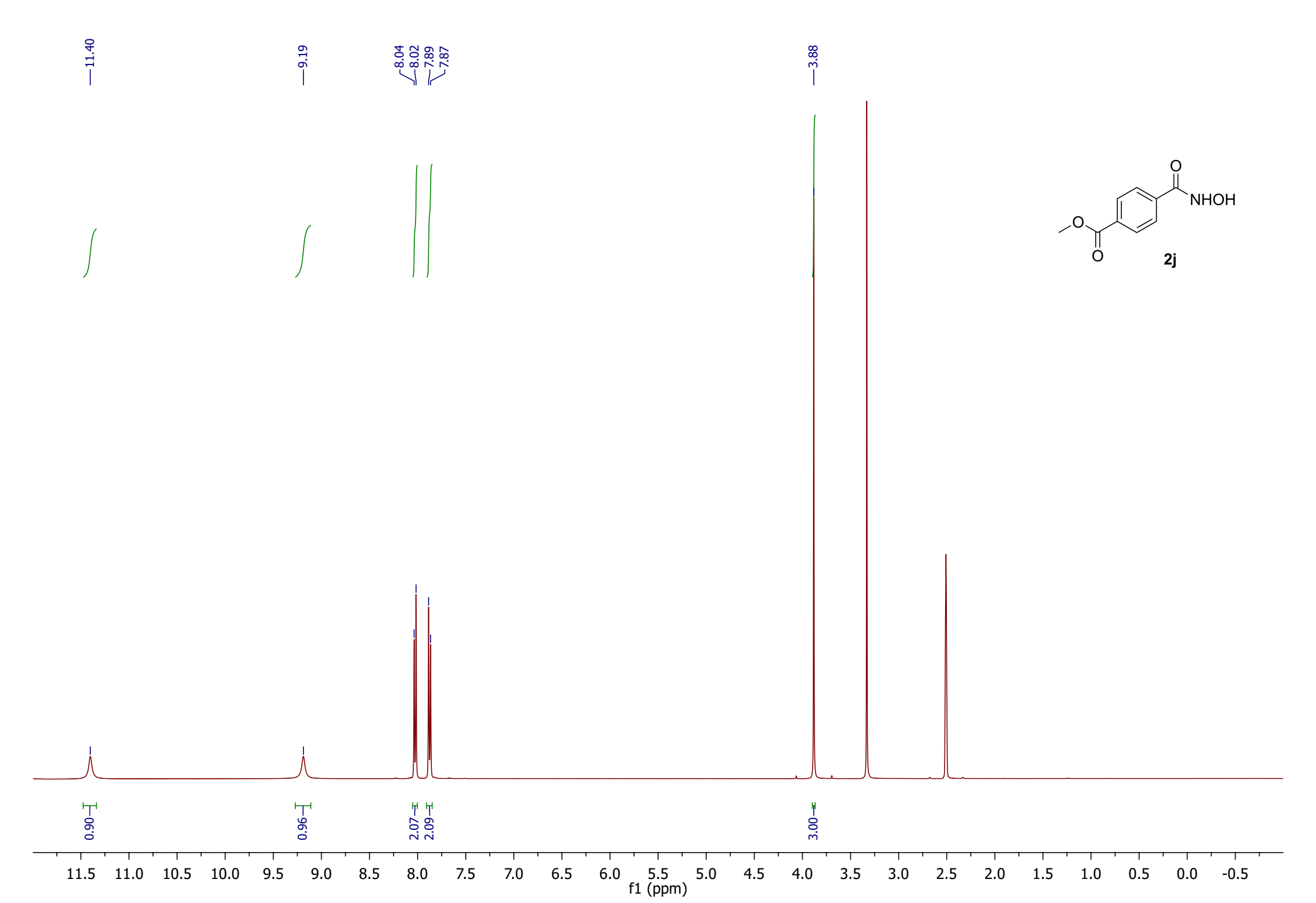
125.91
121.66
Br
O
NHOH
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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O
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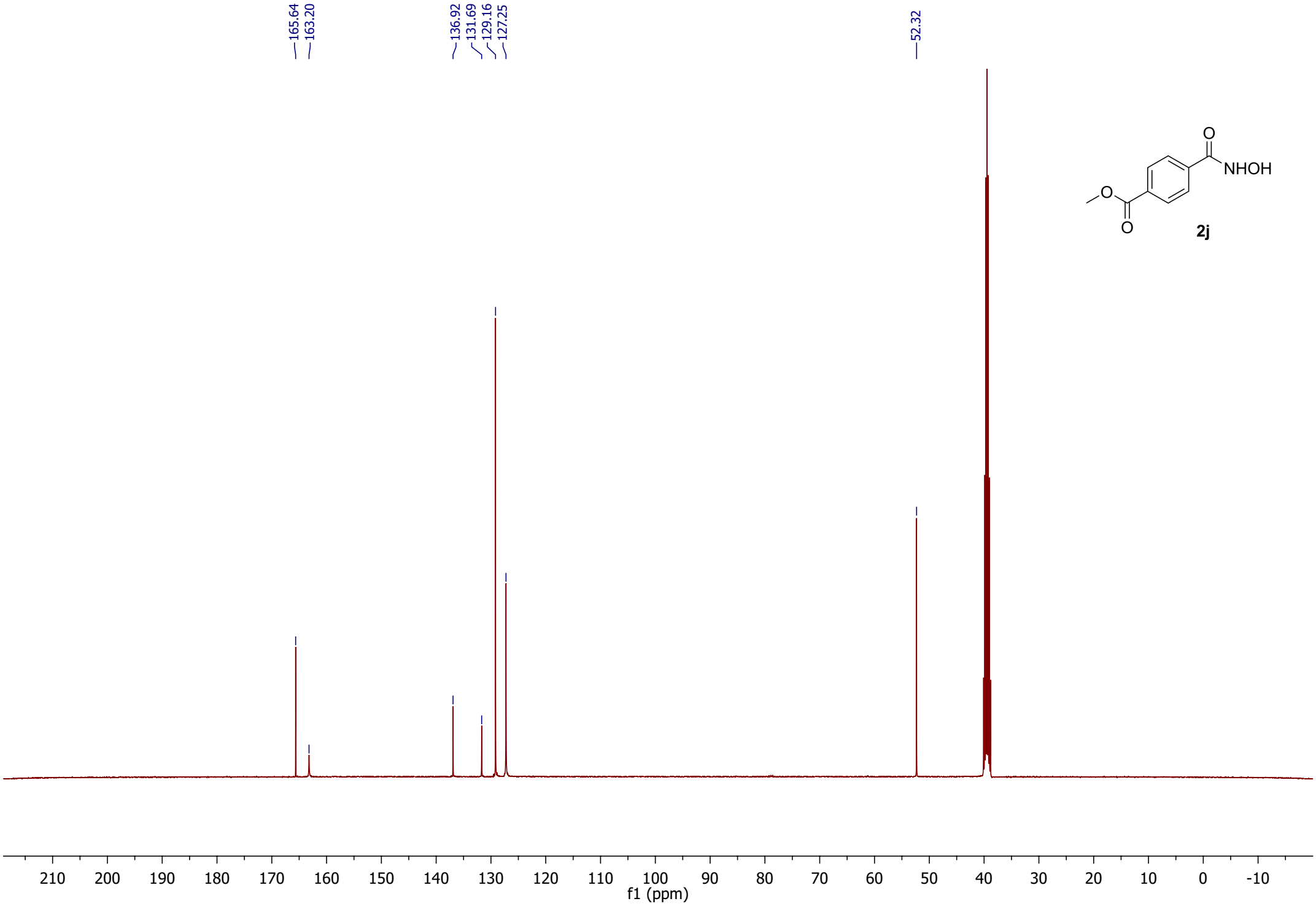
2.07
2.09
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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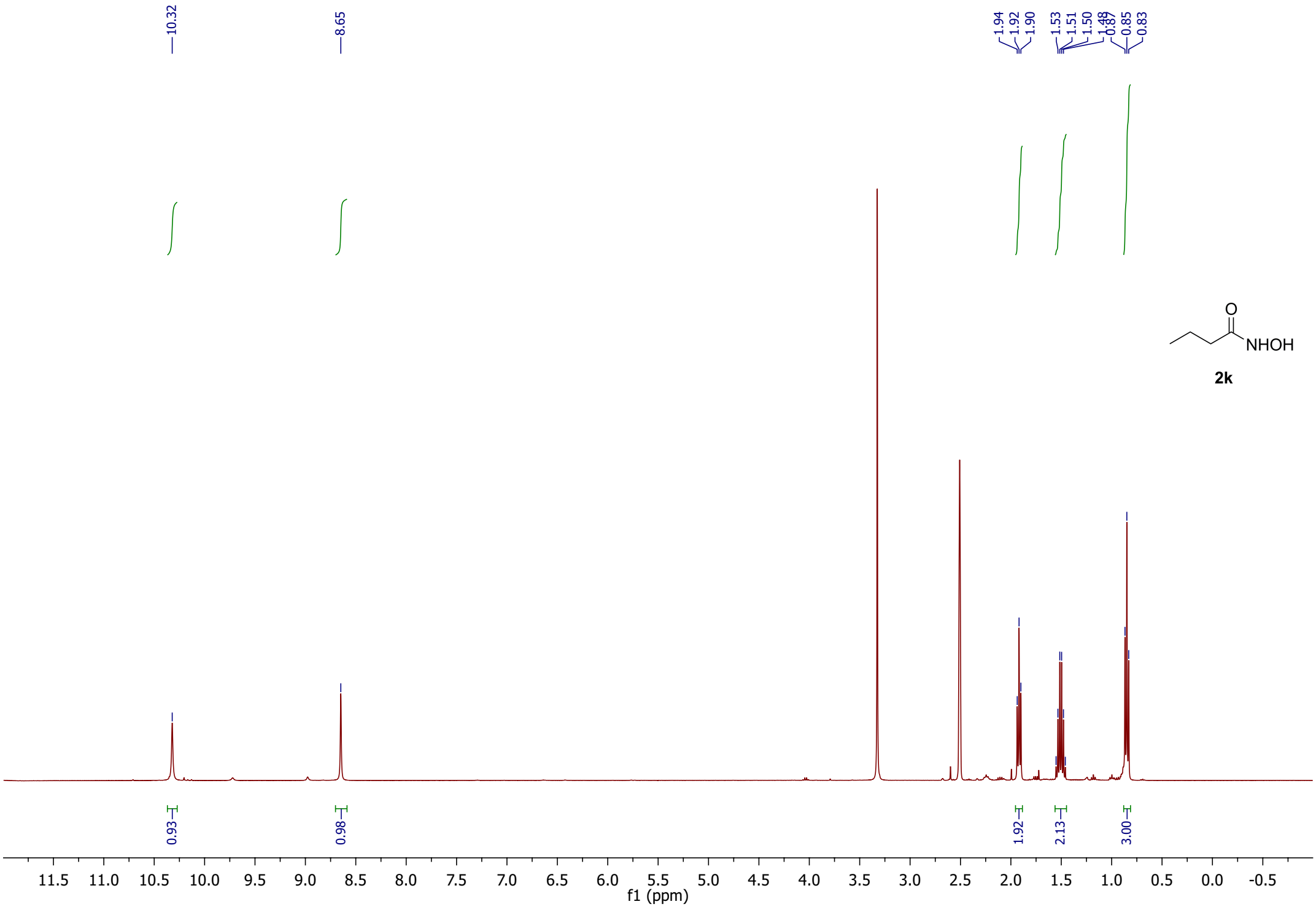
O
NHOH
O
O
2j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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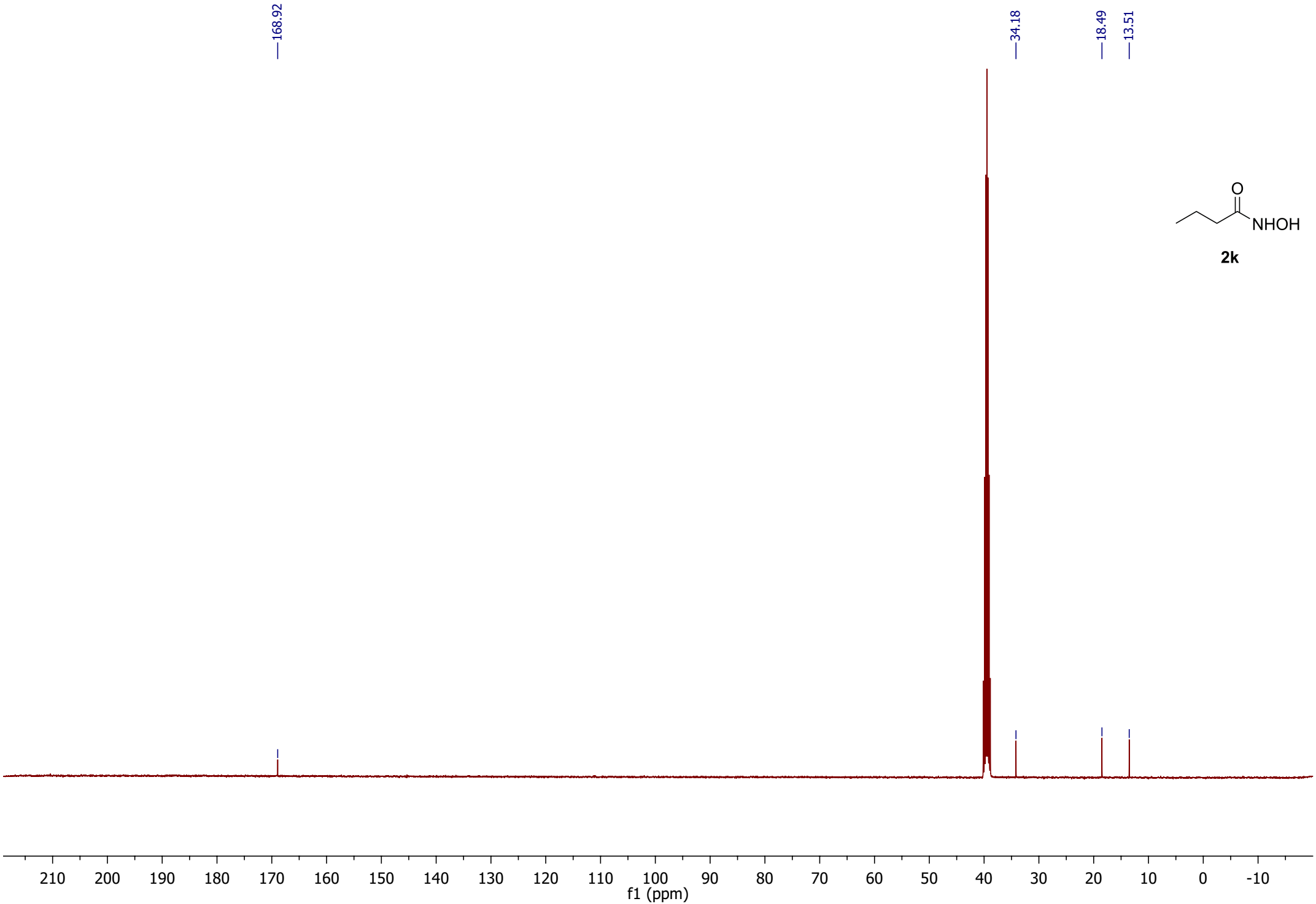
8.65
1.94
1.92
1.90
1.53
1.51
1.50
1.48
0.87
0.85
0.83
O
NHOH
2k
0.93
0.98
1.92
2.13
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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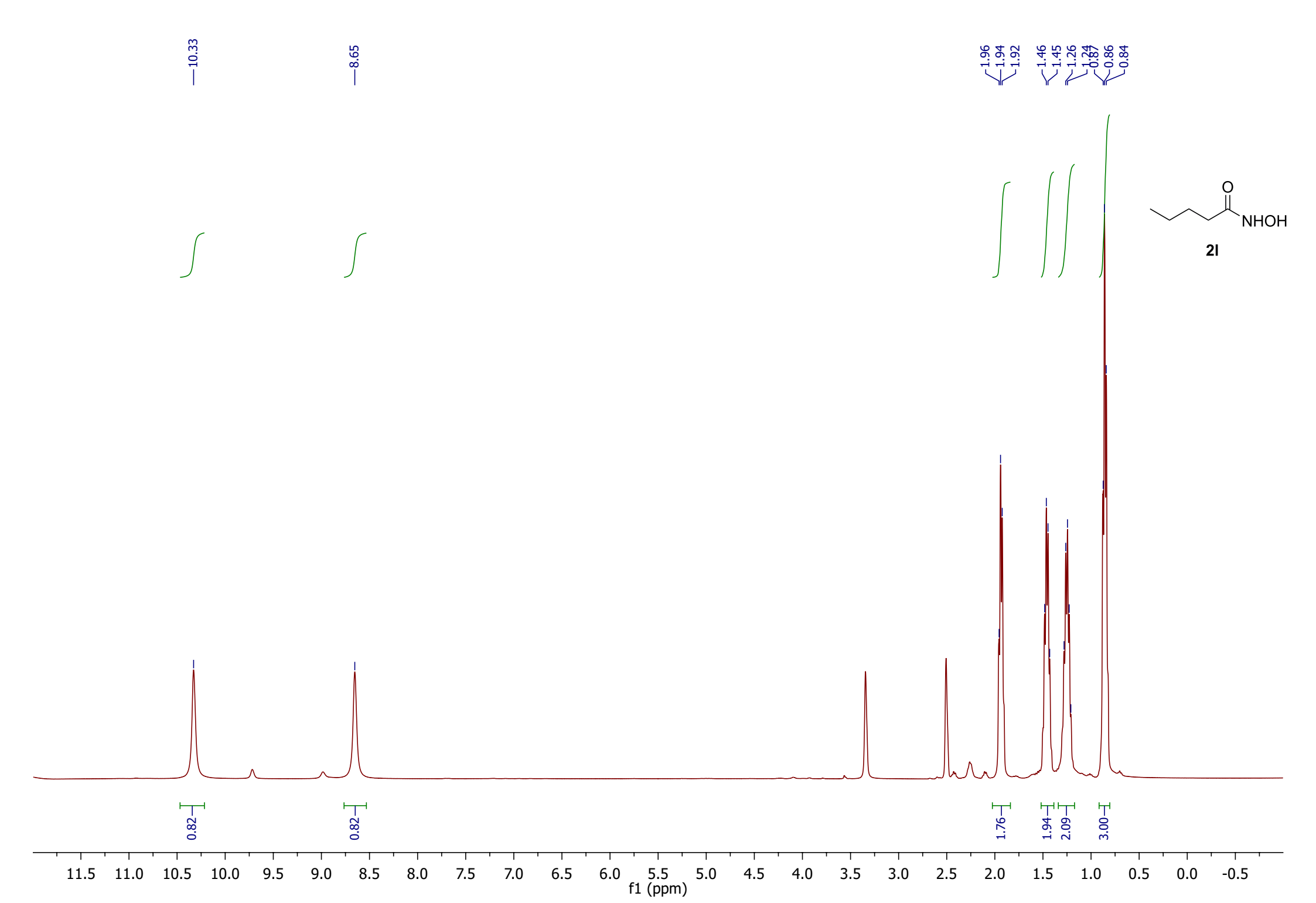
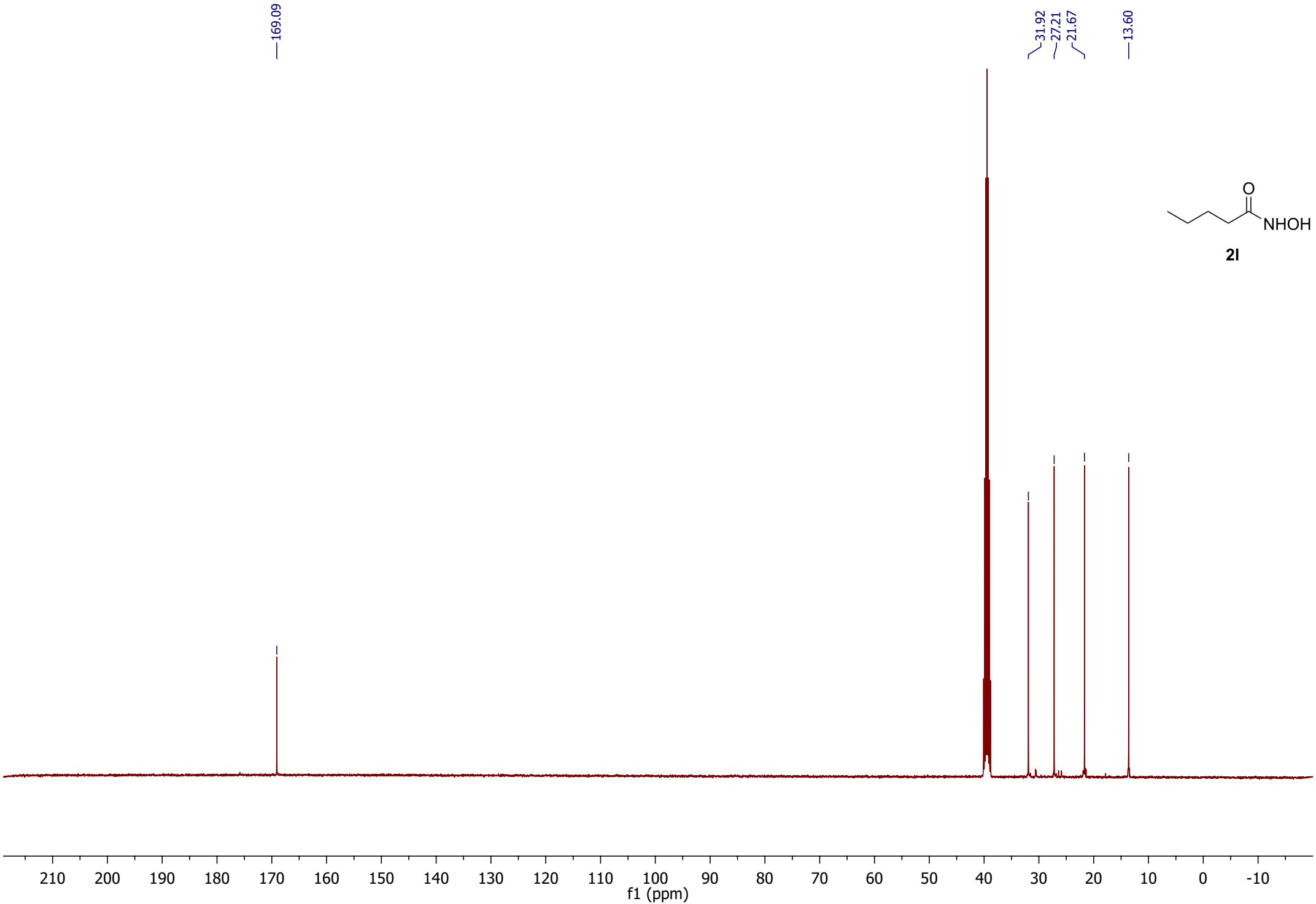
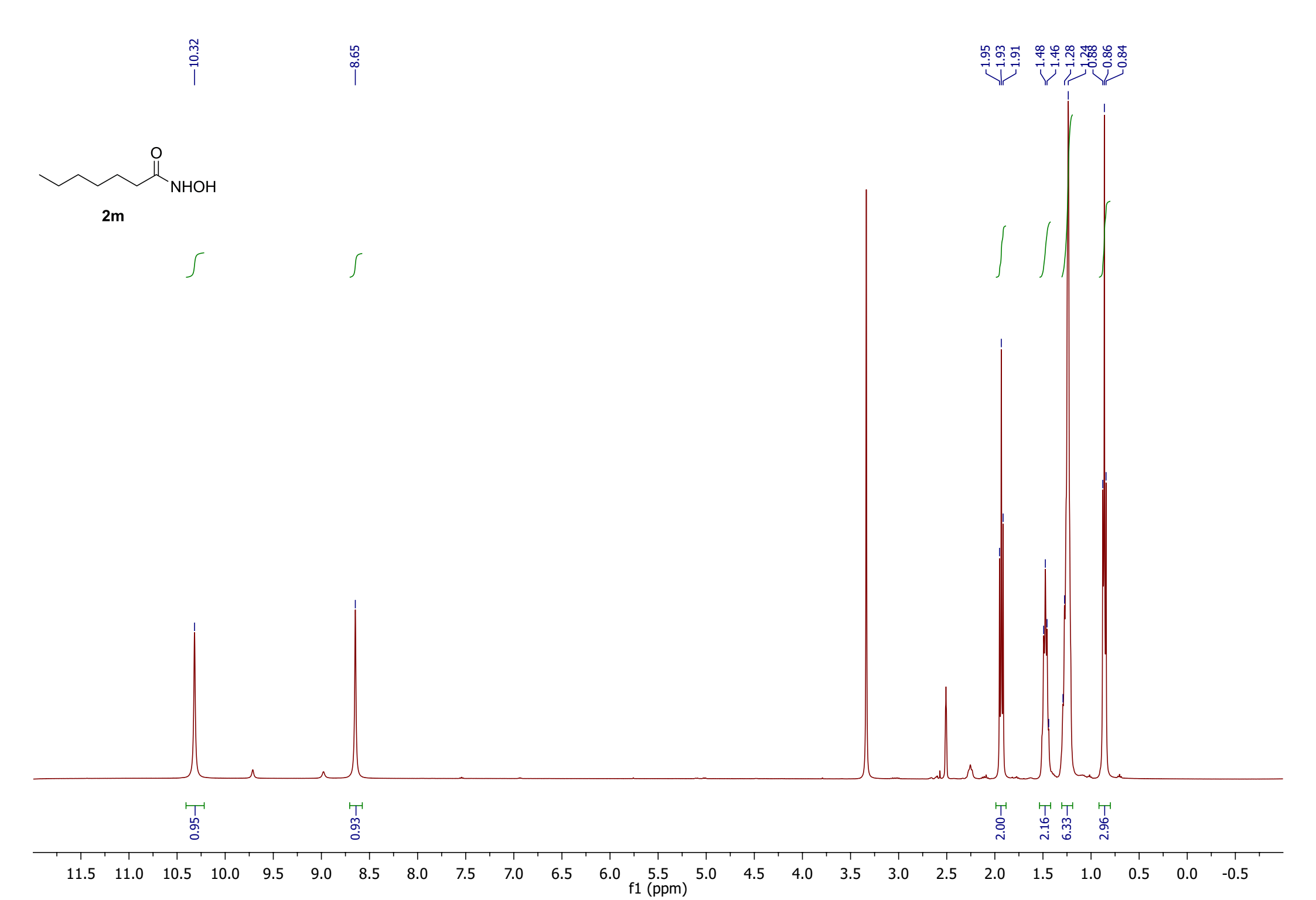
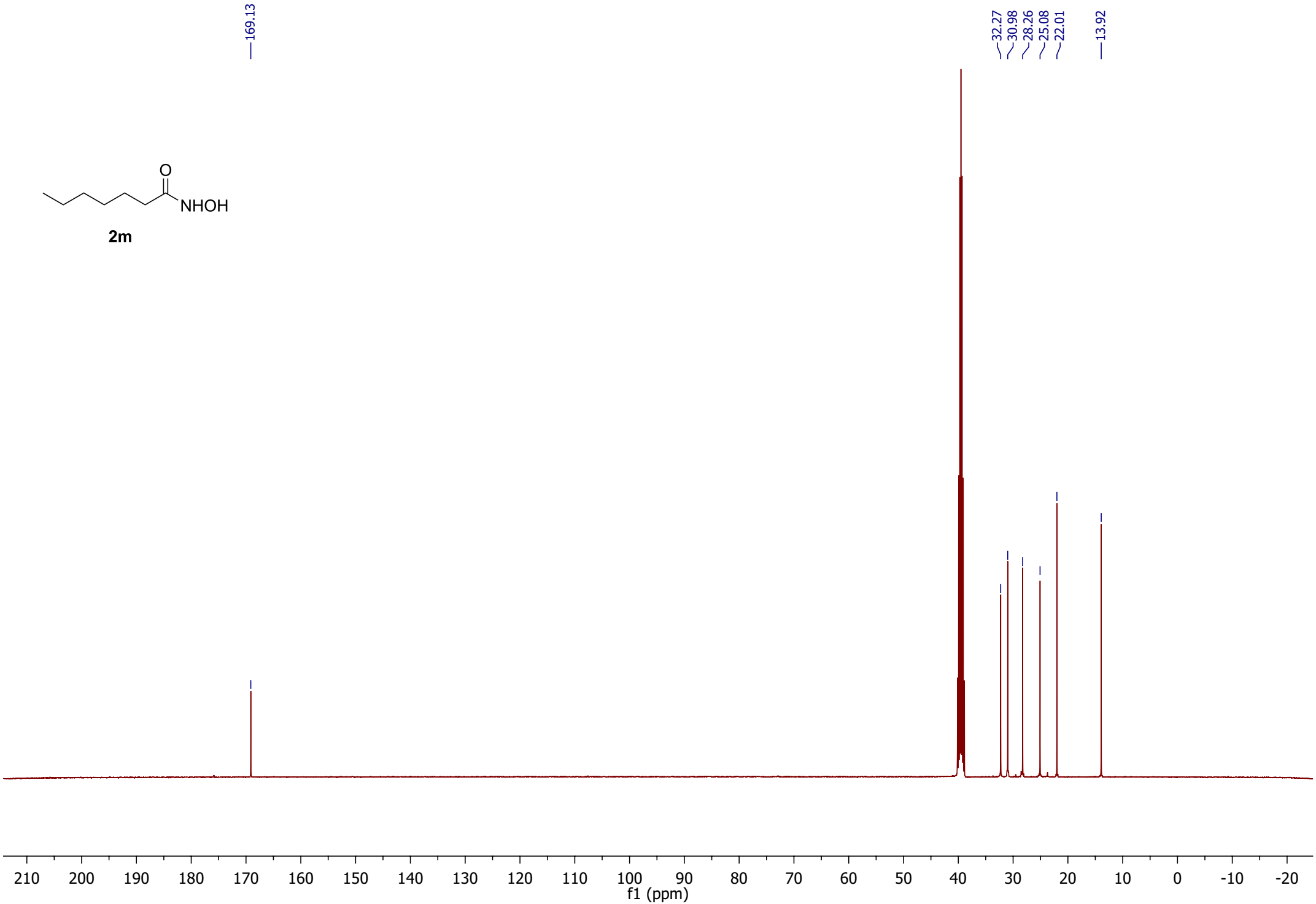
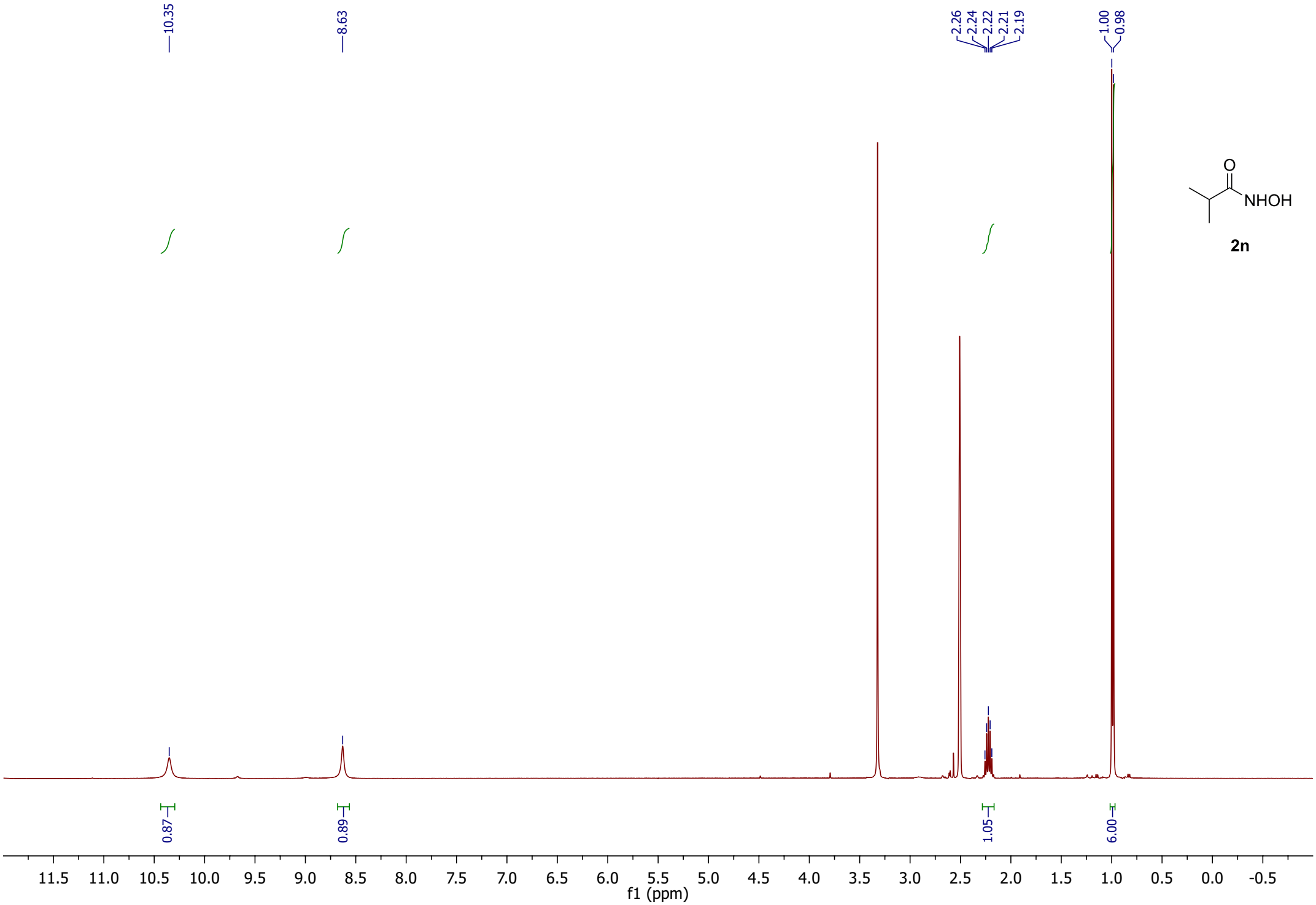
34.18
18.49
13.51
O
NHOH
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

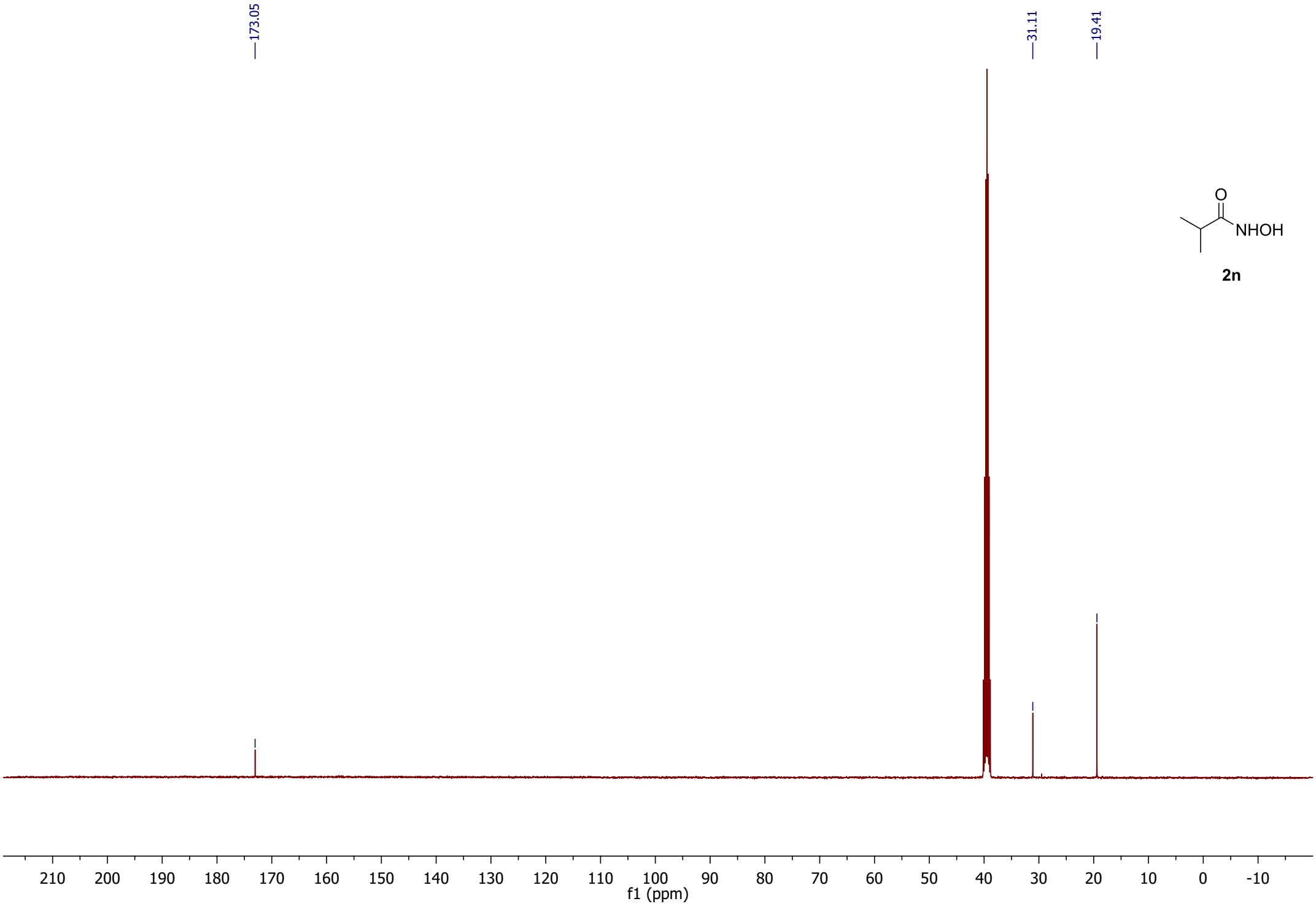

10.33
8.65
1.96
1.94
1.92
1.46
1.45
1.26
1.24
0.87
0.86
0.84
O
NHOH
2l
0.82
0.82
1.76
1.94
2.09
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

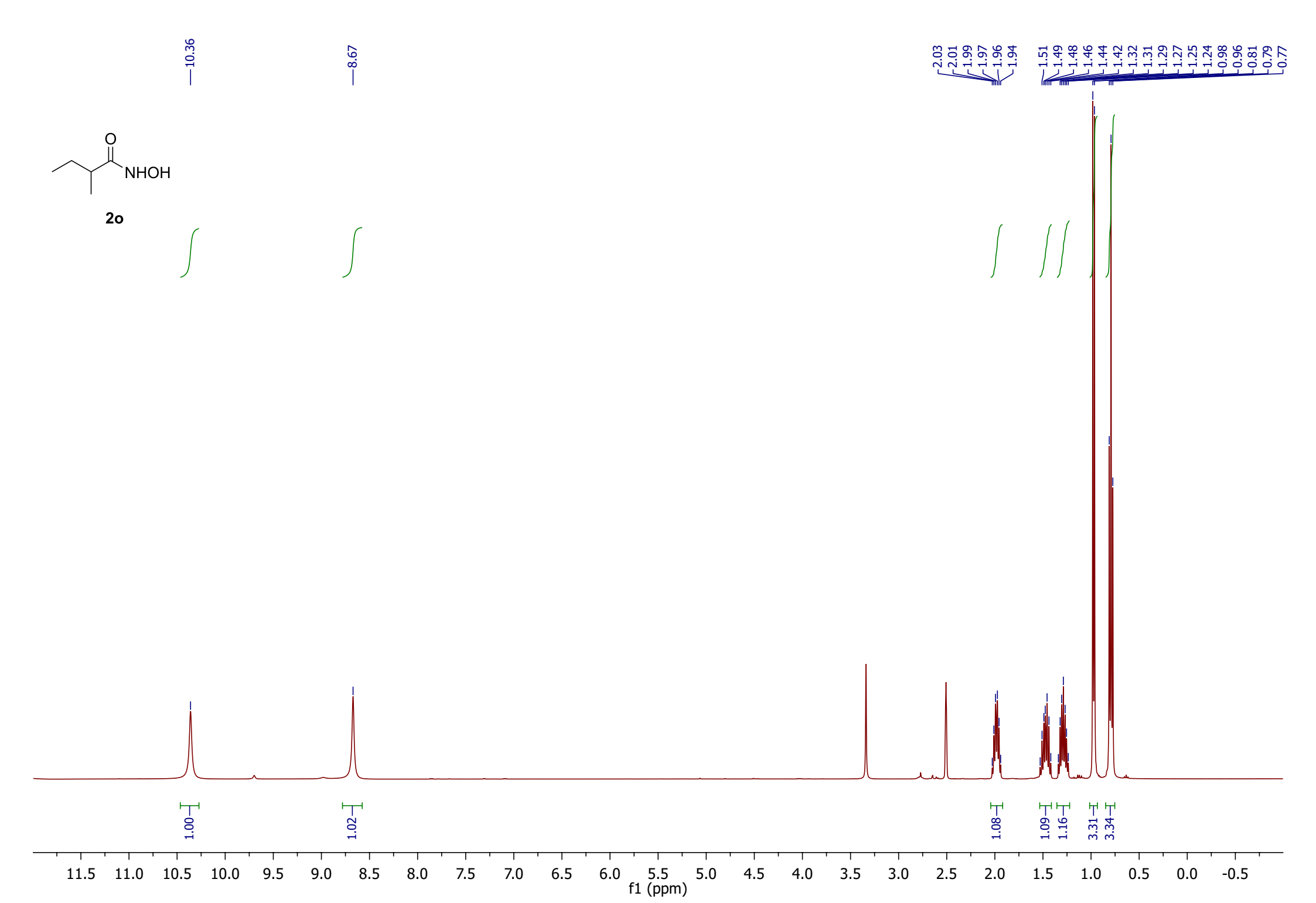

169.09
31.92
27.21
21.67
13.60
O
NHOH
2l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

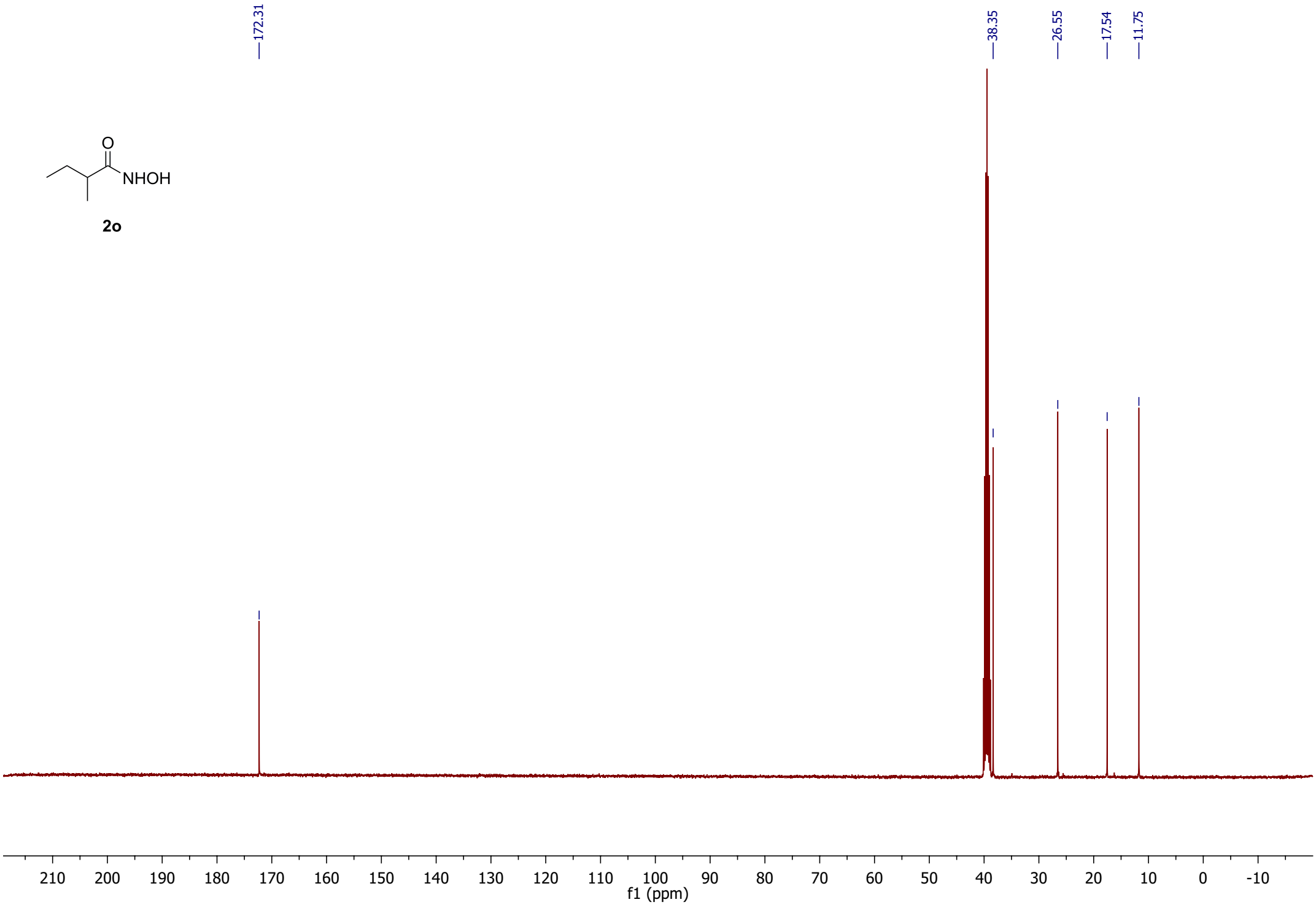

10.32
8.65
1.95
1.93
1.91
1.48
1.46
1.28
1.24
0.88
0.86
0.84
O
NHOH
2m
0.95
0.93
2.00
2.16
6.33
2.96
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

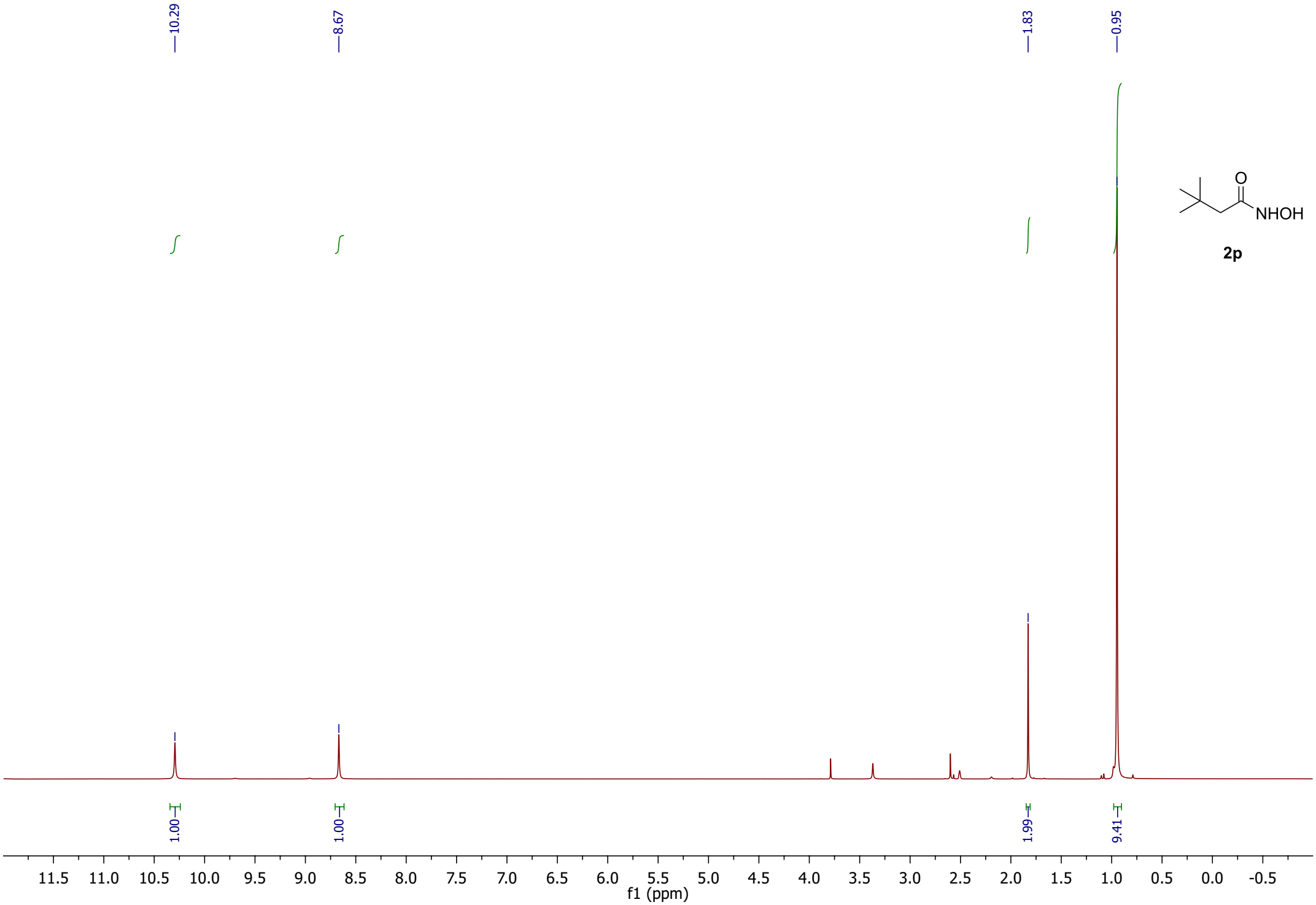

169.13
32.27
30.98
28.26
25.08
22.01
13.92
O
NHOH
2m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

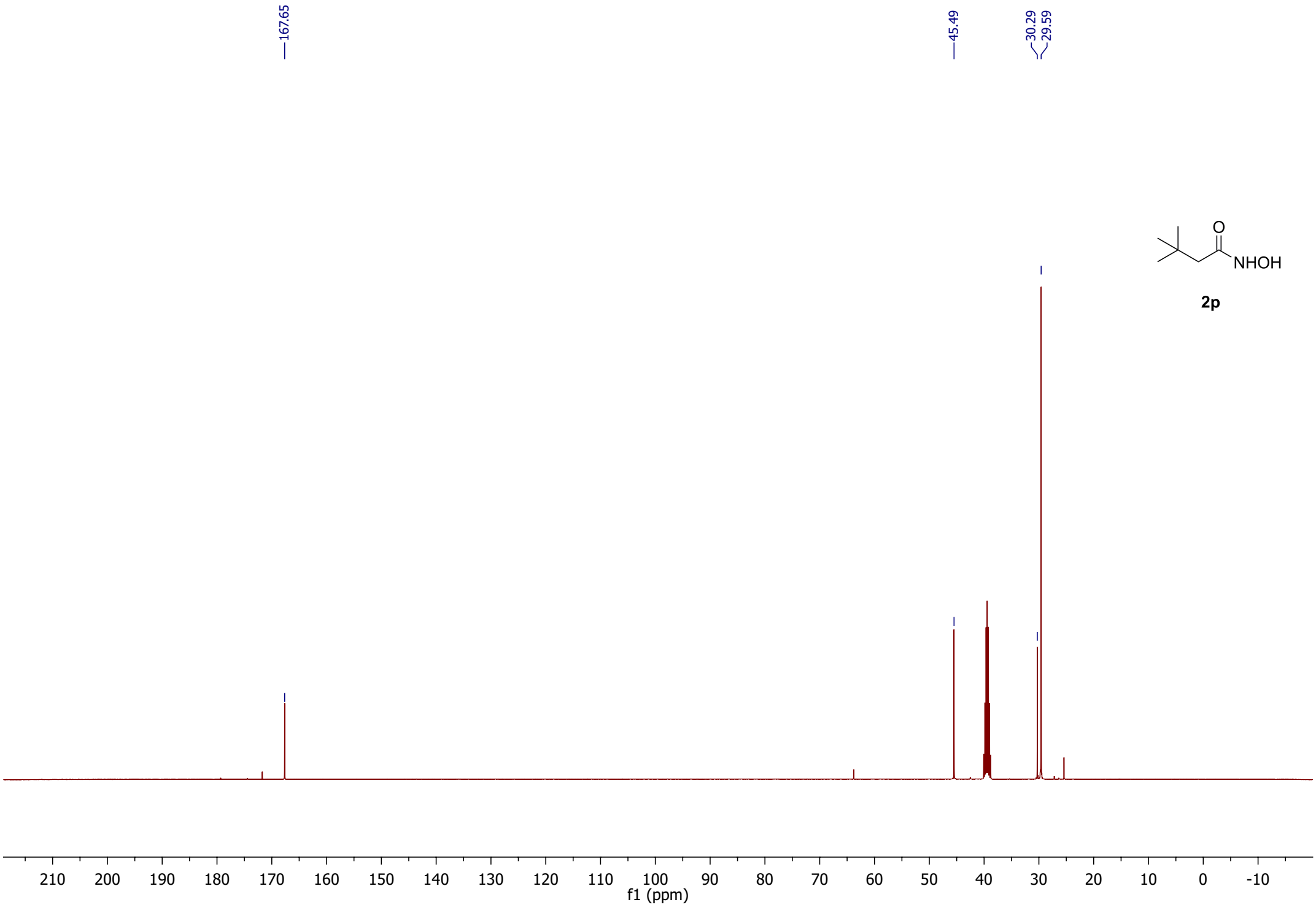

10.35
8.63
2.26
2.24
2.22
2.21
2.19
1.00
0.98
O
NHOH
2n
0.87
0.89
1.05
6.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

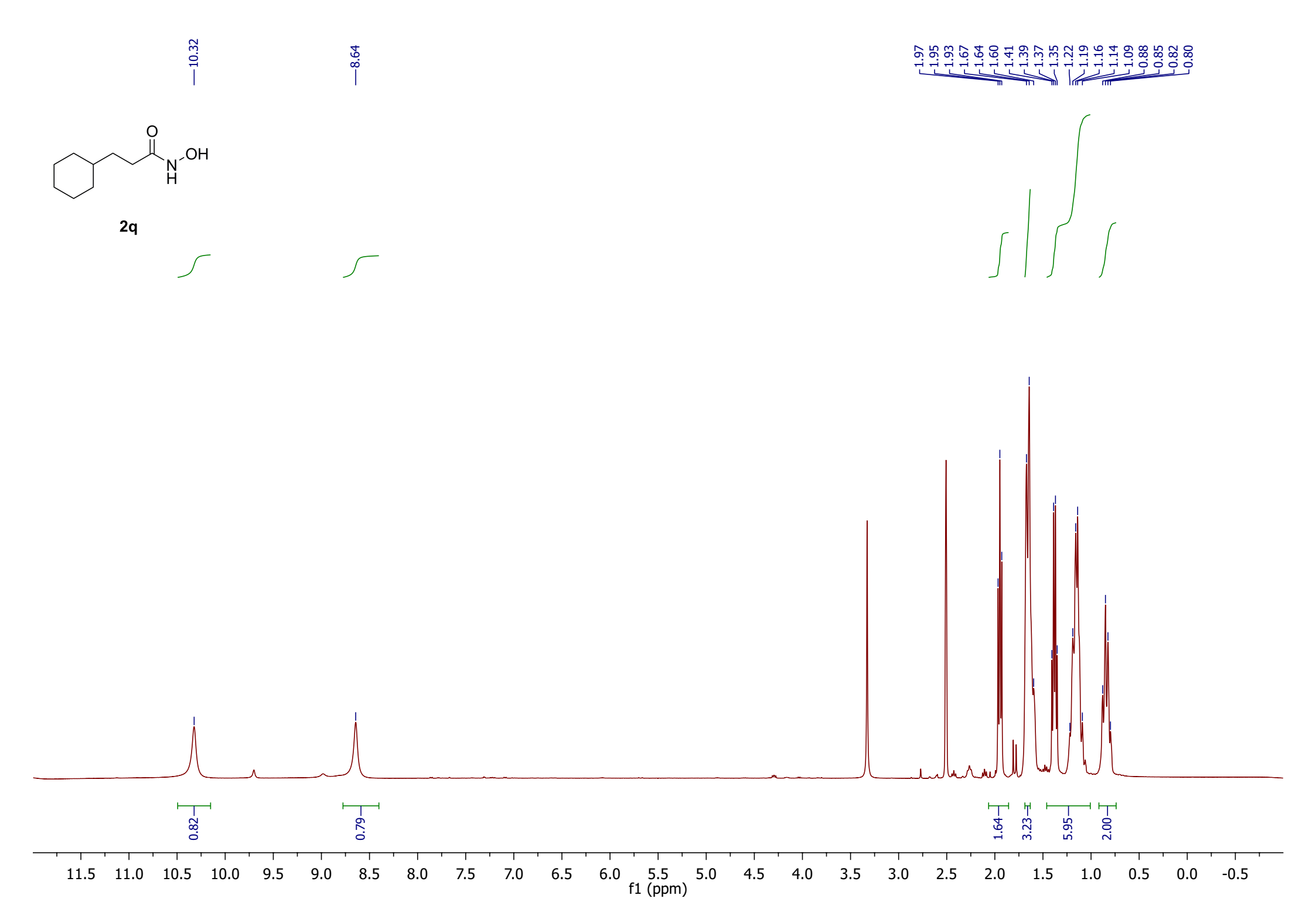

173.05
31.11
19.41
O
NHOH
2n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

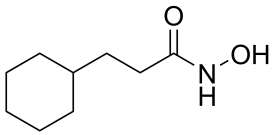

10.36
8.67
2.03
2.01
1.99
1.97
1.96
1.94
1.51
1.49
1.48
1.46
1.44
1.42
1.32
1.31
1.29
1.27
1.25
1.24
0.98
0.96
0.81
0.79
0.77
O
NHOH
2o
1.00
1.02
1.08
1.09
1.16
3.31
3.34
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


172.31
38.35
26.55
17.54
11.75
O
NHOH
2o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


10.29
8.67
1.83
0.95
O
NHOH
2p
1.00
1.00
1.99
9.41
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

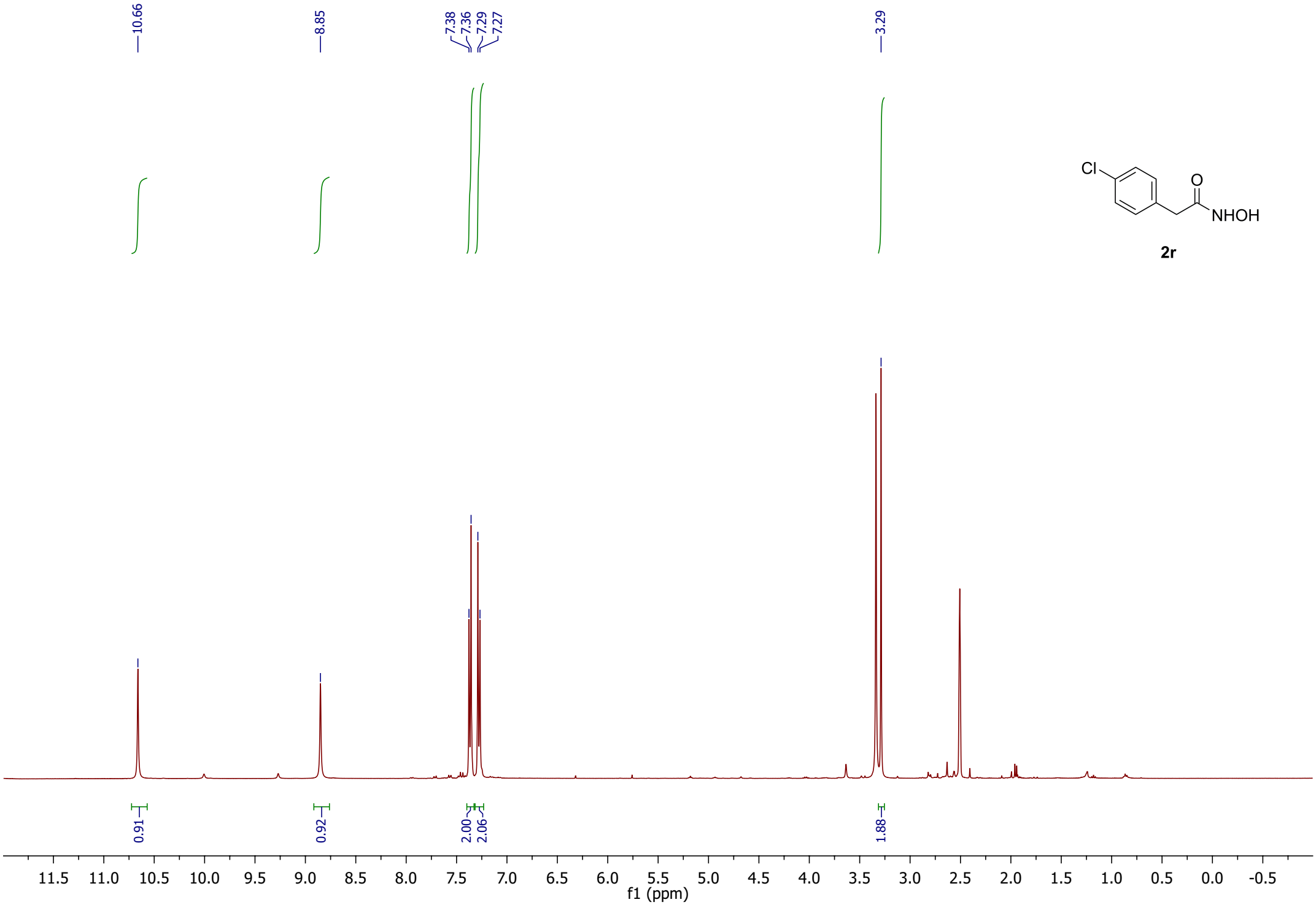

167.65
45.49
30.29
29.59
O
NHOH
2p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

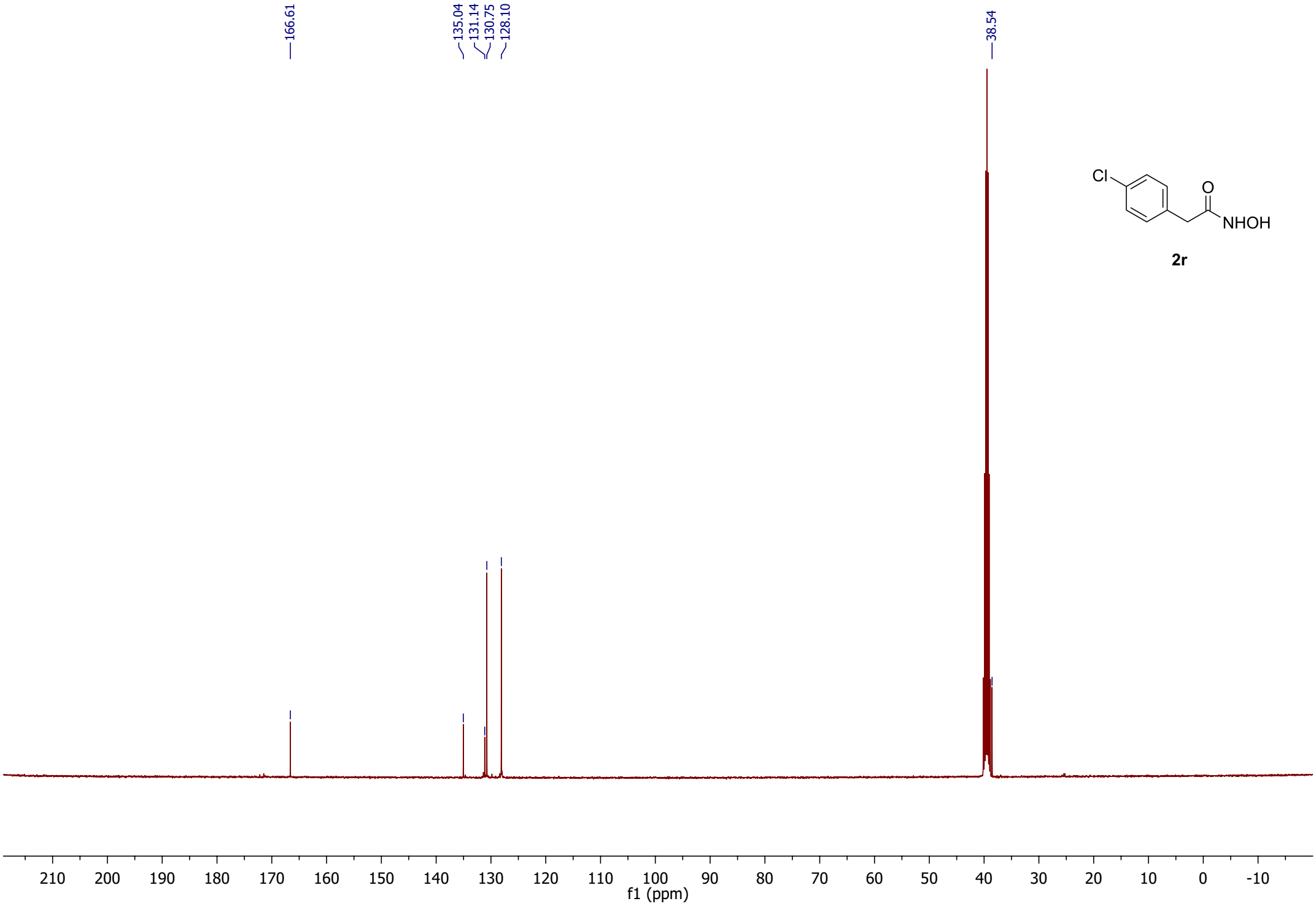

10.32
8.64
1.97
1.95
1.93
1.67
1.64
1.60
1.41
1.39
1.37
1.35
1.22
1.19
1.16
1.14
1.09
0.88
0.85
0.82
0.80
O
N
H
OH
2q
0.82
0.79
1.64
3.23
5.95
2.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

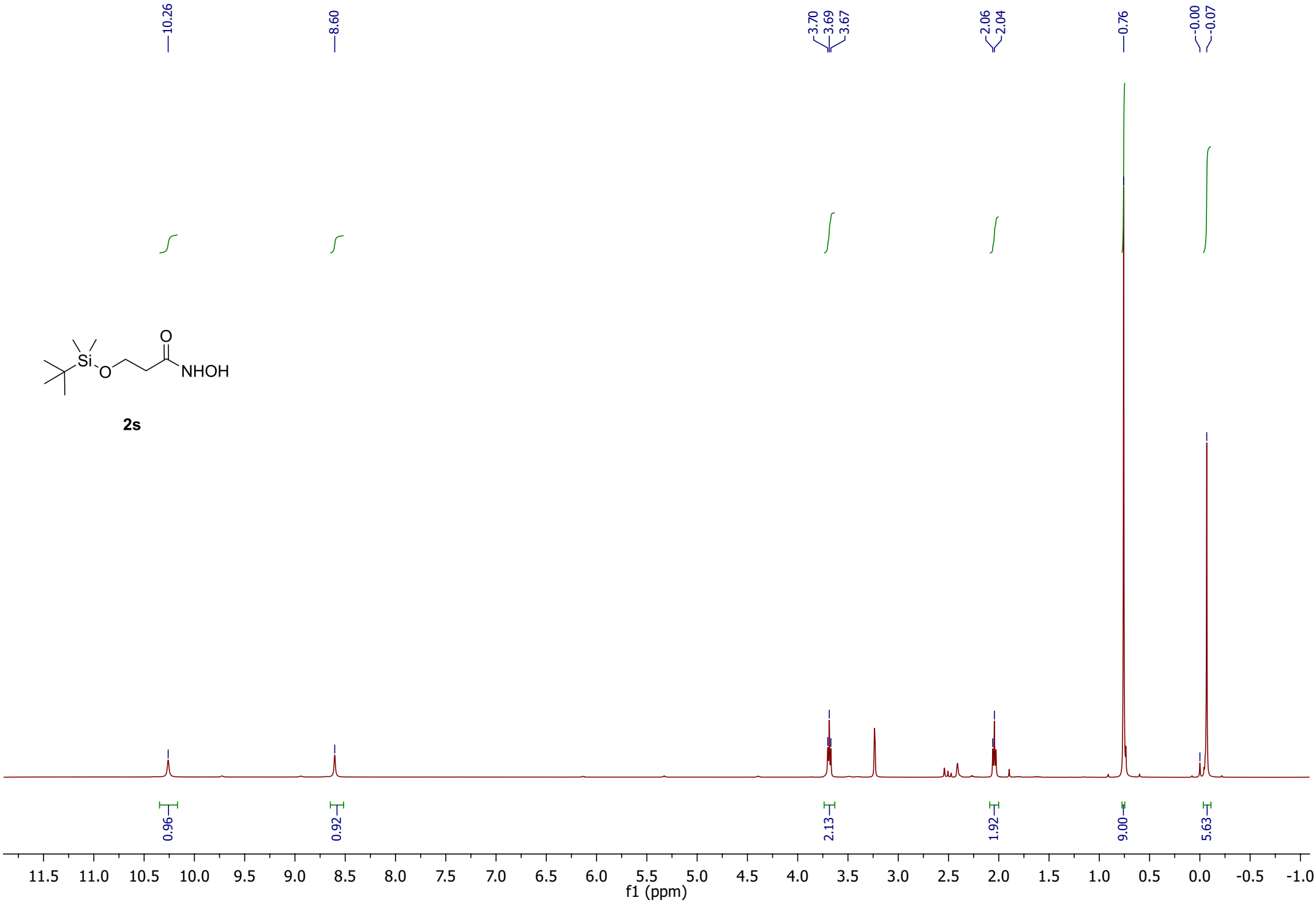

O
N
H
OH

2q

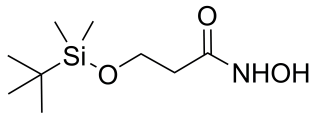
169.29
36.55
32.59
32.51
29.75
26.08
25.73
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


10.66
8.85
7.38
7.36
7.29
7.27
3.29
Cl
O
NHOH
2r
0.91
0.92
2.00
2.06
1.88
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


166.61
135.04
131.14
130.75
128.10
38.54
Cl
O
NHOH
2r
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


10.26
8.60
3.70
3.69
3.67
2.06
2.04
0.76
-0.00
-0.07
Si
O
O
NHOH
0.96
0.92
2.13
1.92
9.00
5.63
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

2s


Si
O
O
NHOH

2s

166.82
59.19
36.12
25.78
17.95
5.39
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)