Electronic Supplementary Information

For

Amide Bond Formation in Aqueous Solution: Direct Coupling of Metal Carboxylate Salts with Ammonium Salts at Room Temperature

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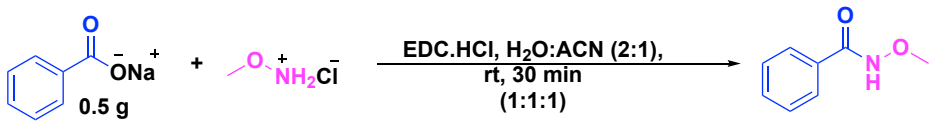
I. General information

Reagents were purchased from commercially available vendors (TCI, Aldrich) and were used without further purification unless otherwise noted. All solvents were used as obtained from commercial sources without further purification unless noted. NMR-spectra were recorded using a 400, 500 and/or 600 MHz Bruker Avance III HD equipped with a cryogenically cooled 5 mm dual probe optimized for ^{1}H and ^{13}C NMR. ^{13}C spectra were recored at 126 MHz, 151 MHz or 101 MHz as indicated. Chemical shifts (δ) are reported in ppm relative to the residual solvent peak (^{1}H NMR) or the solvent peak (^{13}C NMR) as the internal standard. The coupling constant (J) are reported in Hz. HRMS data were obtained on a Thermo Scientific Exactive HRMS coupled to a Thermo Scientific Accela HPLC system using a 2.1 x 50 mm 3.5 µm Waters XTerra C_{18} column eluting with MeCN/H_2O containing 0.1% formic acid.

II. General procedure



Procedure for the synthesis of representative compound *N*-methoxybenzamide:

Our procedure for the synthesis of amide is very simple, follow three steps of solid mixing three salts (carboxylate, ammonium salts, EDC.HCl), add solvent and extraction. An example of synthesis of representative compound *N*-methoxybenzamide as follow:

To a 150 mL RBF, sodium benzoate (0.50 g, 3.47 mmol, 1 equiv.), *O*-methylhydroxylamine (0.29 g, 3.47 mmol, 1 equiv.) and EDC.HCl (0.67 g, 3.47 mmol, 1 equiv.) were added. The solid mixture was stirred, followed by the addition of the mixture

of solvents H_2O:ACN (2:1). The total mixture was stirred at room temperature for 30 min. After that, it was extracted with 2 x 150 mL of ethyl acetate, then washed with 5% $NaHSO_4$(aq.) and 5% $NaHCO_3$(aq.), respectively. The organic layer was concentrated, dried under reduced pressure to afford a clean product as white solid without additional purification. 0.478 g, yield 91%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.03 (s, 1H), 7.81 – 7.73 (m, 2H), 7.46 – 7.37 (m, 1H), 7.35 – 7.26 (m, 2H), 3.74 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.26, 131.89, 131.73, 128.46, 127.30, 63.97. LCMS (ESI) m/z $[M+H]^+$ found 152.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_8H_{10}NO_2^+$ 152.0712, found 152.0713.

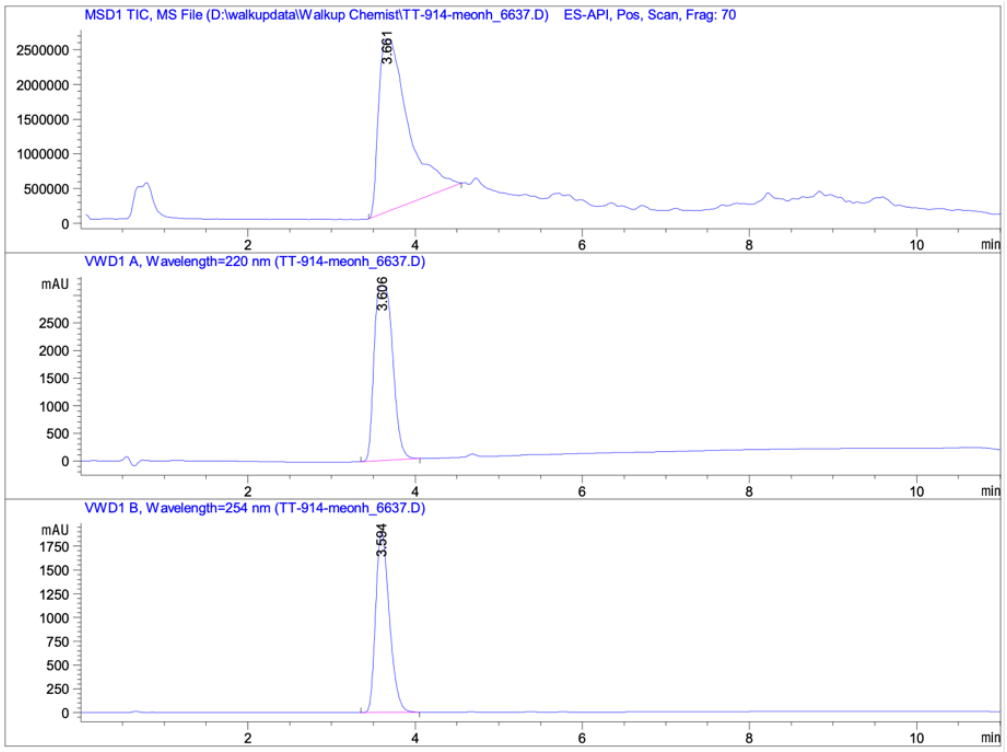


Figure S1. LCMS of *N*-methoxybenzamide after extraction

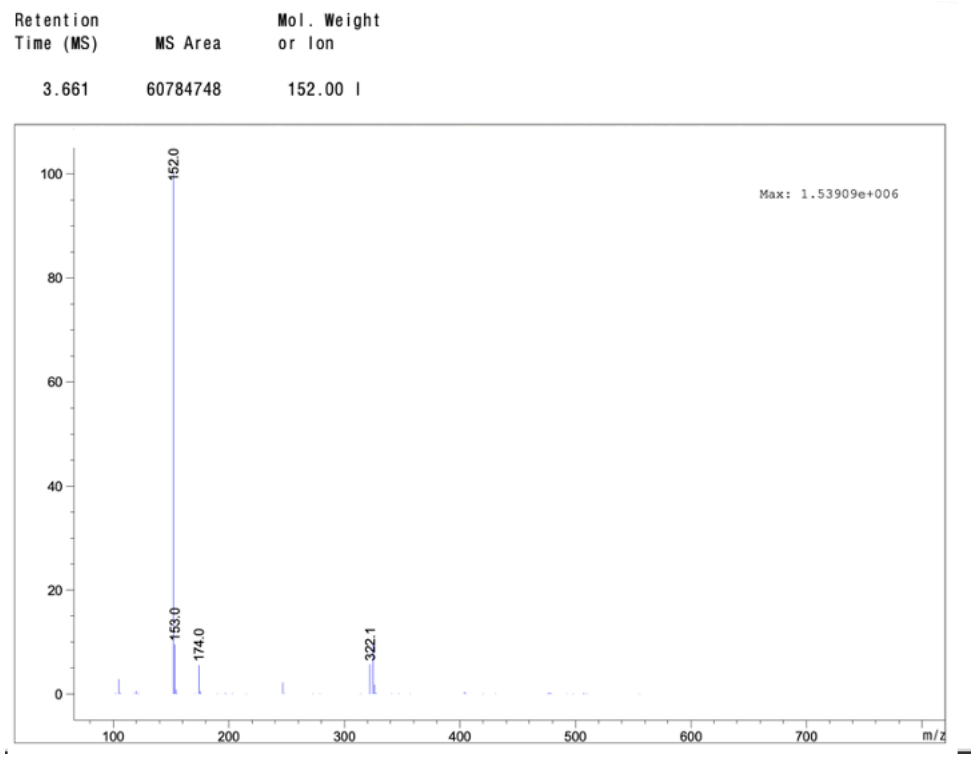


Figure S2. Copy of MS of *N*-methoxybenzamide

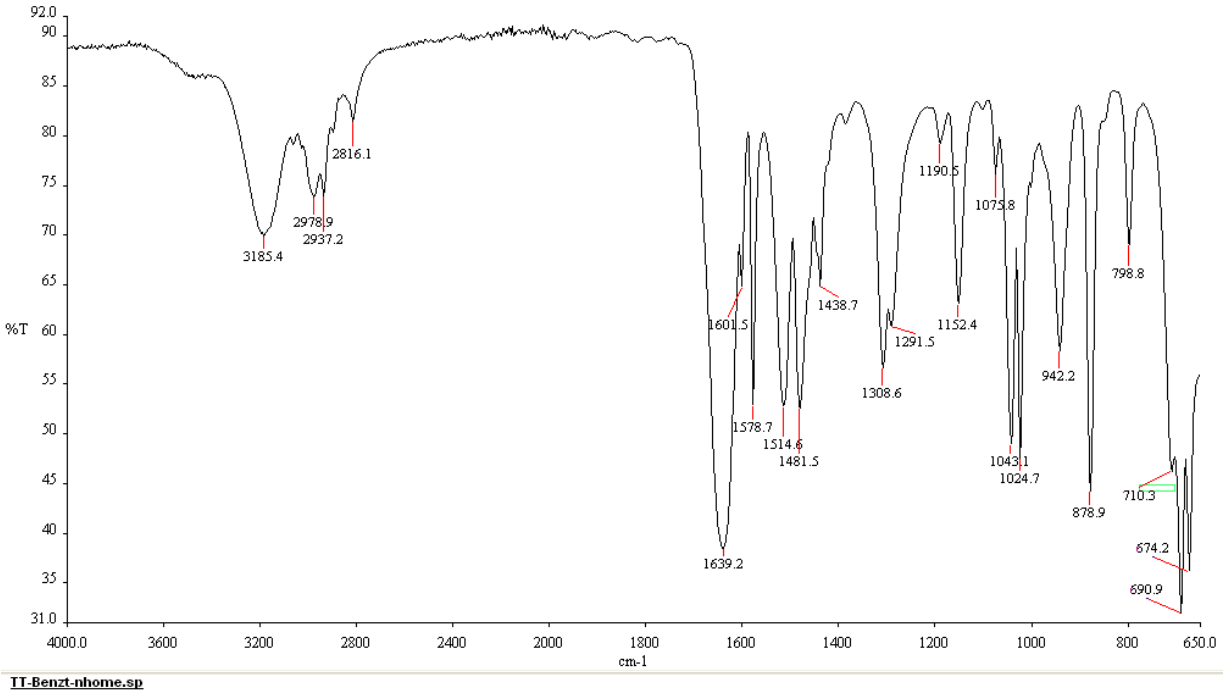
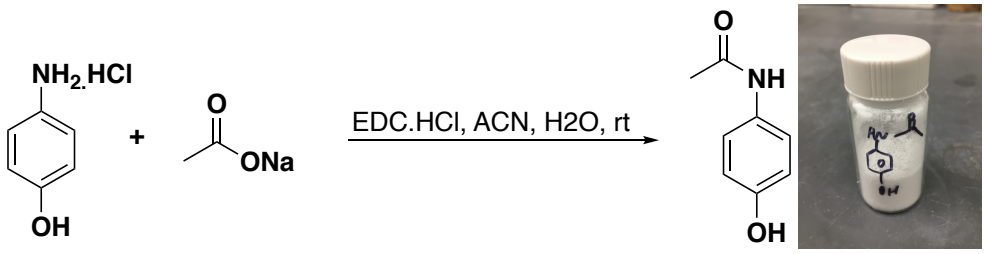


Figure S2. Copy of IR of *N*-methoxybenzamide

III. Gram scale synthesis of drug: acetaminophen

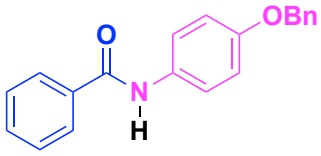


Scheme S1. Gram scale synthesis of drug: acetaminophen

The gram scale synthesis of acetaminophen follows the general procedure as described above, using 4-aminophenol hydrochloride (11.0 g, 76 mmol, 1 equiv.), sodium acetate (6.2 g, 76 mmol, 1 equiv.), EDC.HCl (14.5 g, 76 mmol, 1 equiv.) afford average of 10.28 g of acetaminophen as a white solid, 90% yield. ^{1}H NMR (500 MHz, DMSO) δ 9.68 (s, 1H), 9.19 (s, 1H), 7.53 – 7.21 (m, 2H), 7.02 – 6.60 (m, 2H), 2.03 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 168.32, 153.76, 131.39, 121.65, 115.55, 24.11. LCMS (ESI) m/z $[M+H]^+$ found 152.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_8H_{10}NO_2^+$ 152.0712, found 152.0712.

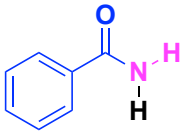
IV. Characterization of the synthesized compounds

The synthesis of the library compounds starts with 0.5 g of corresponding carboxylate salt. The equivalent of ammonium salt and EDC.HCl is 1:1 with respected to the carboxylate salt.

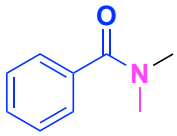


1.052 g, quantitative yield as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 – 7.70 (m, 1H), 7.19 – 7.12 (m, 0H), 7.06 – 6.99 (m, 1H), 6.98 – 6.91 (m, 1H), 6.82 – 6.70 (m, 5H), 6.43 (tt, *J* = 7.3, 1.1 Hz, 2H), 6.36 – 6.30 (m, 4H), 4.48 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.28, 155.03, 150.37, 133.29, 129.74, 129.16, 129.08,

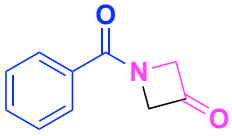
128.88, 128.15, 125.56, 121.22, 120.21, 114.86. LCMS (ESI) m/z $[M+H]^+$ found 304.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{20}H_{18}NO_2^+$ 304.1337, found 304.1336.



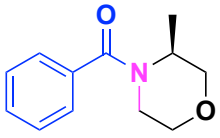
0.193 g, 46 % as a white solid.^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 – 7.79 (m, 2H), 7.60 – 7.48 (m, 1H), 7.48 – 7.39 (m, 2H), 6.38 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.85, 133.39, 132.01, 128.63, 127.38. LCMS (ESI) m/z $[M+H]^+$ found 122.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_7H_8NO^+$ 122.0605, found 122.0606.



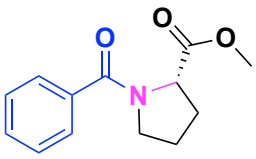
0.450 g, 87 %, as a colorless oil. ^{1}H NMR (500 MHz, DMSO) δ 7.46 – 7.36 (m, 5H), 2.99 (s, 3H), 2.88 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 170.56, 137.03, 129.71, 128.68, 127.36, 39.34, 35.10. LCMS (ESI) m/z $[M+H]^+$ found 150.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_9H_{12}NO^+$ 150.0919, found 150.0918.



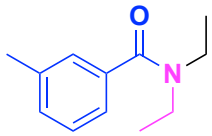
0.607 g, quantitative yield as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.60 (m, 2H), 7.58 – 7.30 (m, 3H), 4.91 (s, 4H). ^{13}C NMR (101 MHz, DMSO) δ 198.51, 170.13, 133.50, 131.83, 128.94, 128.49, 74.07, 71.30. LCMS (ESI) m/z $[M+H]^+$ found 176.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{10}H_{10}NO_2^+$ 176.0712, found 176.0712.



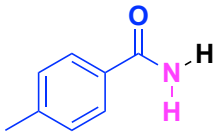
0.647 g, 91 % as a colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 – 7.30 (m, 5H), 4.03-3.34 (br, 7H), 1.35 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.51, 135.91, 129.65, 128.60, 126.66, 70.98, 67.14, 15.42. LCMS (ESI) m/z $[M+H]^+$ found 206.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{12}H_{16}NO_2^+$ 206.1181, found 206.1182.



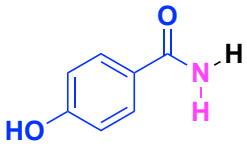
0.720 g, 89 % as a fine white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.50 (m, 2H), 7.45 – 7.32 (m, 3H), 4.66 (dd, *J* = 8.4, 5.2 Hz, 1H), 3.78 (t, *J* = 7.0 Hz, 1H), 3.76 (s, 2H), 3.73 – 3.62 (m, 1H), 3.62 (t, *J* = 5.2 Hz, 1H), 3.56 – 3.48 (m, 1H), 2.32 – 2.17 (m, 1H), 2.00 (tdd, *J* = 16.5, 7.3, 3.0 Hz, 3H), 1.94 – 1.81 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.76, 169.68, 136.13, 130.20, 128.25, 128.23, 127.28, 59.14, 52.25, 49.93, 29.38, 25.37. LCMS (ESI) m/z $[M+H]^+$ found 234.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_{16}NO_3^+$ 234.1130, found 234.11302.



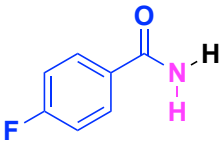
0.604 g, quantitative yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.76 – 6.58 (m, 4H), 2.70-2.98 (br, 4H), 2.70 (s, 1H), 1.80 (s, 4H), 0.54-0.70 (br, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.52, 137.45, 137.00, 129.23, 127.72, 126.45, 122.75, 42.75, 38.72, 20.77, 13.72, 12.46. LCMS (ESI) m/z $[M+H]^+$ found 192.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{12}H_{18}NO^+$ 192.1388, found 192.1387.



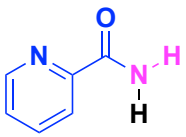
0.248 g, 58 % as a white solid. ^{1}H NMR (500 MHz, DMSO) δ 8.00 (s, 1H), 7.87 – 7.81 (m, 2H), 7.40 (s, 1H), 7.23 (d, *J* = 8.1 Hz, 2H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 168.53, 141.56, 131.94, 129.19, 128.02, 21.35. LCMS (ESI) m/z $[M+H]^+$ found 136.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_8H_{10}NO^+$ 136.0762, found 136.0762.



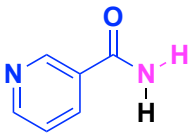
0.274 g, 64 % as a light-pink solid. ^{1}H NMR (500 MHz, DMSO) δ 10.12 (s, 1H), 7.98 (s, 1H), 7.88 (d, *J* = 8.6 Hz, 2H), 7.40 (s, 1H), 6.90 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (126 MHz, DMSO) δ 169.24, 160.88, 130.17, 125.16, 115.45. LCMS (ESI) m/z $[M+H]^+$ found 138.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_7H_8NO_2^+$ 138.0555, found 138.0556.



0.425 g, 99 % as a fine white solid. ^{1}H NMR (500 MHz, DMSO) δ 8.10 (s, 1H), 8.03 – 7.95 (m, 2H), 7.51 (s, 1H), 7.27 – 7.19 (m, 2H). ^{13}C NMR (126 MHz, DMSO) δ 167.61, 164.43 (d, *J* = 248.6 Hz), 131.12 (d, *J* = 2.8 Hz), 130.59 (d, *J* = 8.8 Hz), 115.47 (d, *J* = 21.6 Hz). LCMS (ESI) m/z $[M+H]^+$ found 140.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_7H_7FNO^+$ 140.0512, found 140.0511.

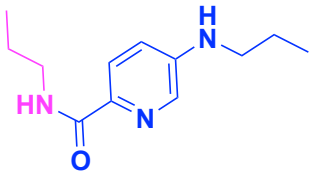


0.265 g, 63 % as a light-yellow oil. ^{1}H NMR (500 MHz, MeOD) δ 8.59 (dt, *J* = 4.9, 1.3 Hz, 1H), 8.11 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.90 (td, *J* = 7.7, 1.7 Hz, 1H), 7.50 (ddd, *J* = 7.6, 4.8, 1.3 Hz, 1H). 4.87 (s, 2H). ^{13}C NMR (126 MHz, MeOD) δ 167.94, 149.65, 148.46, 137.31, 126.46, 121.97. LCMS (ESI) m/z $[M+H]^+$ found 123.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_6H_7N_2O^+$ 123.0558, found 123.0558.

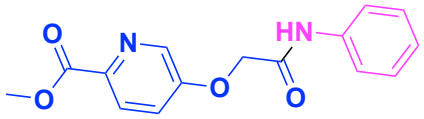


0.252 g, 60 % as a light-yellow oil. ^{1}H NMR (500 MHz, DMSO) δ 9.10 (dd, *J* = 2.4, 1.0 Hz, 1H), 8.67 (dd, *J* = 4.8, 1.8 Hz, 1H), 8.29 (s, 1H), 8.24 (dt, *J* = 8.0, 2.0 Hz,

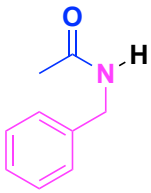
1H), 7.74 (s, 1H), 7.44 (ddd, *J* = 7.9, 4.9, 0.9 Hz, 1H). ^{13}C NMR (126 MHz, DMSO) δ 167.31, 152.29, 149.16, 135.66, 130.10, 123.79. LCMS (ESI) m/z $[M+H]^+$ found 123.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_6H_7N_2O^+$ 123.0558, found 123.0558.



0.454 g, 83 %. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) *δ* 7.99 (d, *J* = 8.6 Hz, 1H), 7.87 (d , *J* = 2.7 Hz, 1H), 7.78 (br, 1H), 6.90 (dd, *J* = 2.8, 2.8 Hz, 2H), 4.09 (br, 1H), 3.41 (dd, *J* = 6.8, 6.4 Hz, 2H), 3.14 (t, *J* = 7.1, 2H) 1.60-1.71 (m, 4H), 0.96-1.03 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) *δ* 164.90, 146.30, 139.10, 133.60, 123.30, 117.70, 45.10, 41.0, 23.10, 22.50, 11.50, 11.50. LCMS (ESI) m/z $[M+H]^+$ found 222.1. HRMS (ESI) m/z [M+H]+ calcd for $C_{12}H_{20}N_3O^+$ 222.1606, found 222.1606.

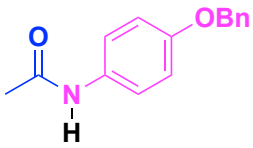


0.540 g, 88 %. yellow solid. ^{1}H NMR (600 MHz, $CDCl_3$) *δ* 8.54 (d, *J* = 2.9 Hz, 1H), 8.18 (d, *J* = 8.8 Hz, 2H), 7.59 (d, *J* = 7.9 Hz, 2H), 7.37 (dd, *J* = 2.6, 6.5 Hz, 3H), 7.18 (t, *J* = 7.4 Hz, 1H), 4.72 (s, 2H), 4.00 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) *δ* 165.01, 164.50, 155.61, 141.90, 138.70, 136.42,129.21, 126.7, 125.30, 120.90, 120.30, 67.60, 52.91. LCMS (ESI) m/z $[M+H]^+$ found 287.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{15}H_{15}N_2O_4^+$ 287.1032, found 287.1030.

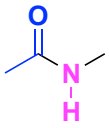


0.91 g, quantitative yield as a colorless solid. ^{1}H NMR (500 MHz, DMSO) δ 8.44 (dd, *J* = 12.2, 6.5 Hz, 1H), 7.39 – 7.23 (m, 5H), 4.32 (dq, *J* = 9.4, 3.9 Hz, 2H), 1.97 – 1.90 (m, 3H). ^{13}C NMR (126 MHz, DMSO) δ 169.76, 140.08, 128.72, 127.77, 127.19,

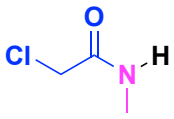
42.73, 23.01. LCMS (ESI) m/z $[M+H]^+$ found 150.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_9H_{12}NO^+$ 150.0919, found 150.0918.



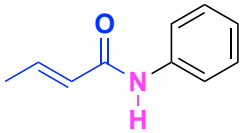
1.34 g, 91 % as a white solid. ^{1}H NMR (500 MHz, DMSO) δ 9.85 (s, 1H), 7.58 – 7.51 (m, 2H), 7.47 – 7.42 (m, 2H), 7.42 – 7.35 (m, 2H), 7.35 – 7.29 (m, 1H), 7.00 – 6.93 (m, 2H), 5.05 (s, 2H), 2.04 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 168.26, 154.59, 137.69, 133.29, 128.85, 128.22, 128.12, 121.02, 115.25, 69.85, 24.29. LCMS (ESI) m/z $[M+H]^+$ found 242.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{15}H_{16}NO_2^+$ 242.1181, found 242.11812.



0.369 g, 83 % as a white needle-like crystals. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.91 (br, 1H), 2.67 (d, *J* = 4.8 Hz, 3H), 1.87 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.30, 26.15, 22.77.

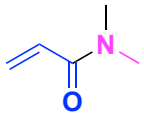


0.351 g, 76 % as a light-yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82 (s, 2H), 2.56 (d, *J* = 4.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.63, 42.43, 26.38.

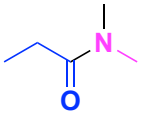


0.746 g, quantitative yield as a white solid. ^{1}H NMR (400 MHz, DMSO) δ 9.97 (s, 1H), 7.74 – 7.67 (m, 2H), 7.36 – 7.27 (m, 2H), 7.04 (td, *J* = 7.4, 1.2 Hz, 1H), 6.84 (dq, *J* = 15.4, 6.9 Hz, 1H), 6.17 (dq, *J* = 15.3, 1.7 Hz, 1H), 1.86 (dd, *J* = 6.9, 1.7 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 163.98, 140.19, 139.82, 129.12, 126.56, 123.58,

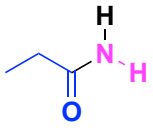
119.74, 17.93. LCMS (ESI) m/z $[M+H]^+$ found 162.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{10}H_{12}NO^+$ 162.0919, found 162.0919.



0.406 g, 77 % as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.97 (dd, *J* = 16.7, 10.5 Hz, 1H), 5.53 (dd, *J* = 16.7, 2.4 Hz, 1H), 4.94 (dd, *J* = 10.4, 2.4 Hz, 1H), 2.40 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.37, 127.48, 126.18, 36.44, 34.63.

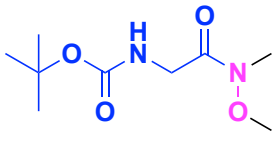


0.416 g, 79 % as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.15 (s, 3H), 2.04 (s, 3H), 1.45 (q, *J* = 7.4 Hz, 2H), 0.21 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (76 MHz, $CDCl_3$) δ 172.53, 36.14, 34.28, 25.57, 8.48.

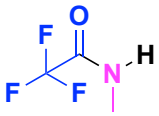


0.266 g, 70 % as a white snowflake. ^{1}H NMR (300 MHz, DMSO) δ 7.29 (s, 1H), 6.83 (s, 1H), 2.06 (q, *J* = 7.6 Hz, 2H), 0.97 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (76 MHz, DMSO) δ 176.31, 28.62, 10.01.

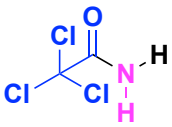
0.221 g, 55 % as a light-yellow oid. ^{1}H NMR (500 MHz, DMSO) δ 7.61 (s, 1H), 7.36 (s, 1H), 4.01 (s, 2H). ^{13}C NMR (126 MHz, DMSO) δ 168.98, 42.91.



0.493 g, 89 % as a white crystal. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.32 (t, *J* = 5.1 Hz, 1H), 3.92 (d, *J* = 5.2 Hz, 2H), 3.58 (s, 3H), 3.05 (s, 3H), 1.30 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.15, 155.82, 79.26, 61.27, 41.53, 32.20, 28.19 (3C).

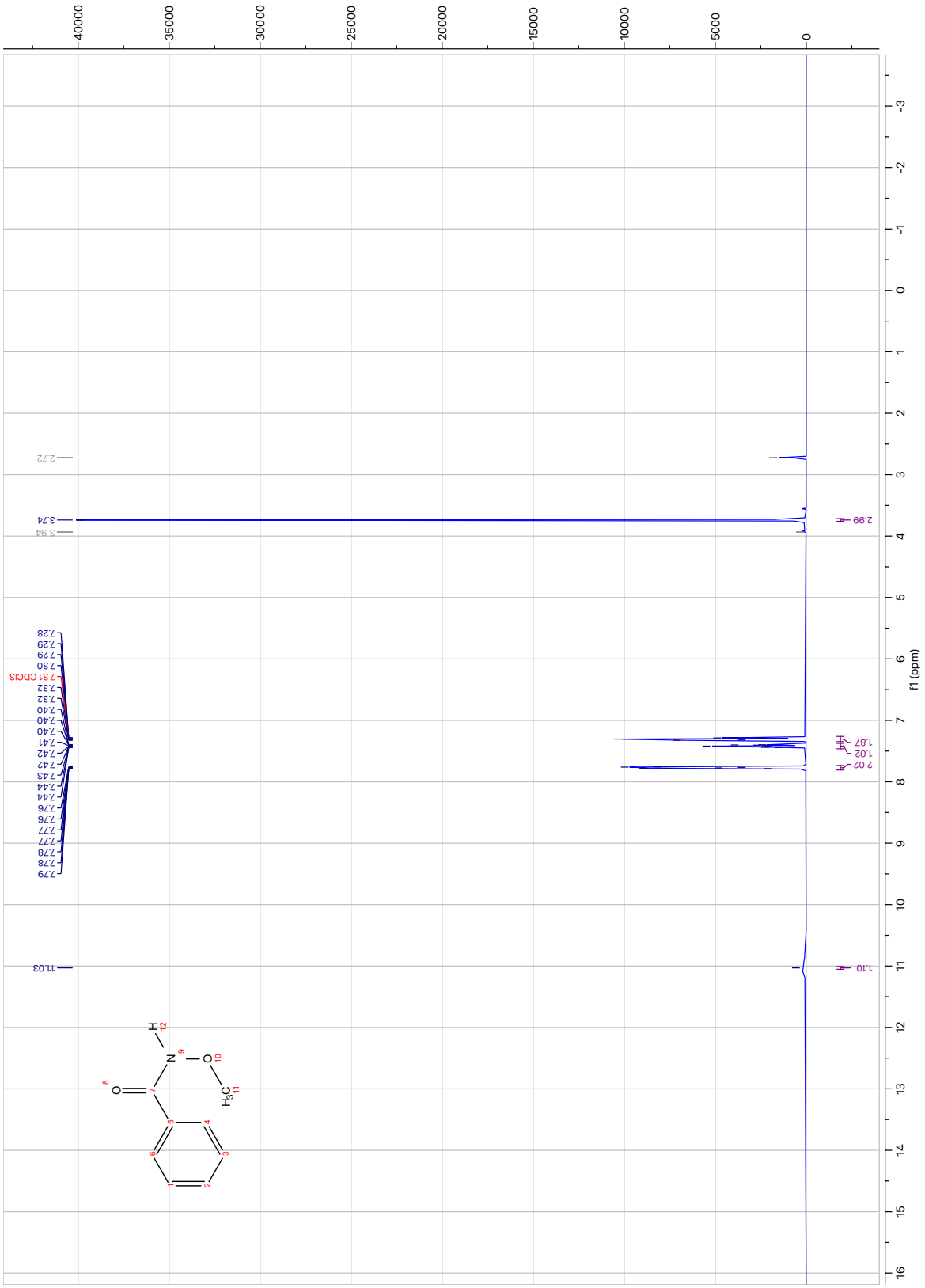


0.425 g, 91 % as a fine white needle-like crystals. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 – 7.73 (m, 1H), 2.86 (d, *J* = 4.9 Hz, 3H). ^{13}C NMR (76 MHz, $CDCl_3$) δ 158.35 (q, *J* = 37.0 Hz), 115.87 (q, *J* = 286.5 Hz), 26.00. ^{19}F NMR (282 MHz, $CDCl_3$) δ -76.61.



0.263 g, 60 % as a deep yellow oil. ^{1}H NMR (400 MHz, DMSO) δ 8.12 (s, 2H). ^{13}C NMR (101 MHz, DMSO) δ 163.39, 93.39.

V. Copy of NMR spectra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7.78
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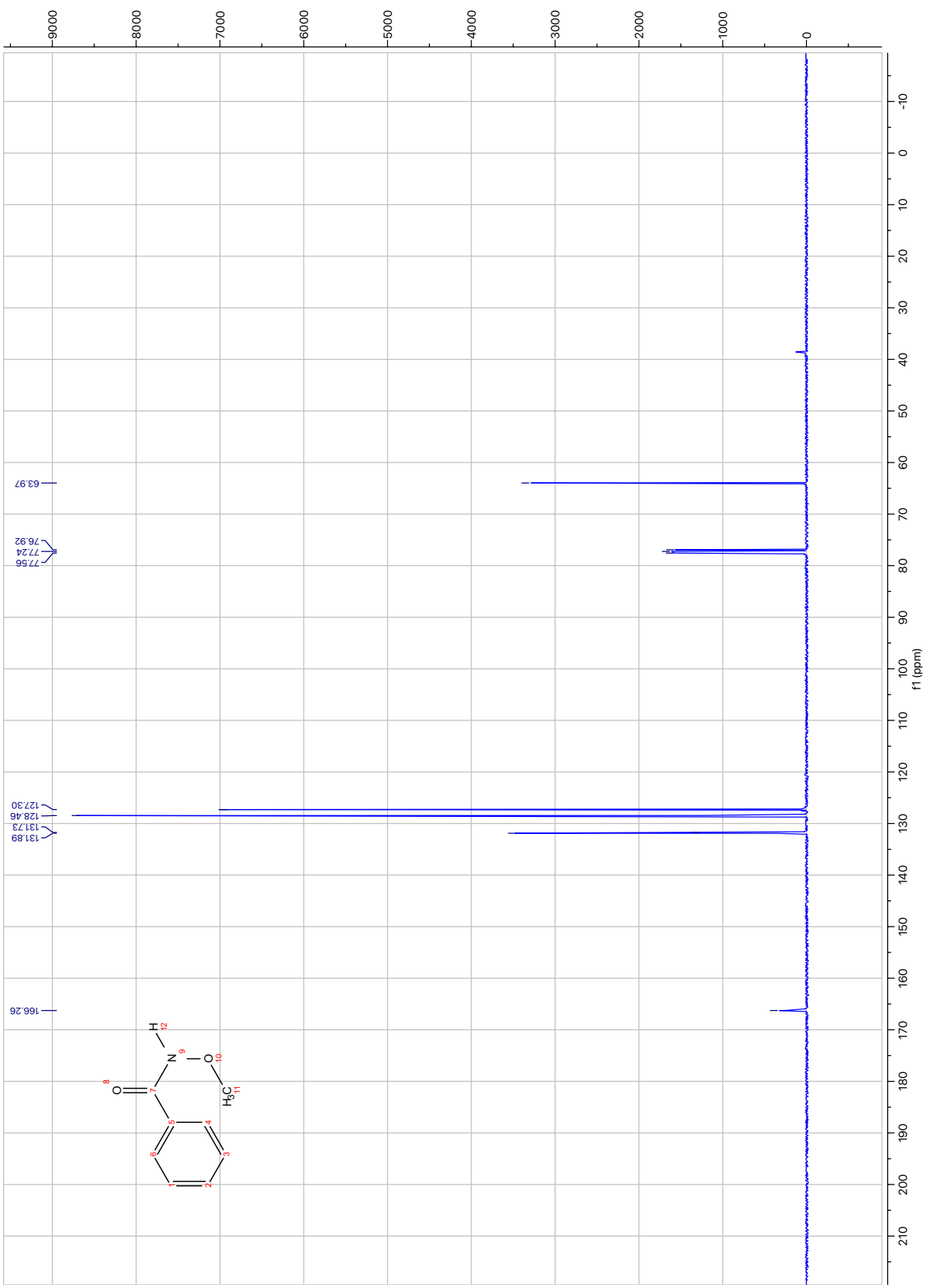
7.43
7.42
7.42
7.41
7.40
7.40
7.40
7.32
7.32
7.31 CDCl3
7.30
7.29
7.29
7.28
3.94
3.74
2.72
2.99
2.02
1.02
1.87
1.10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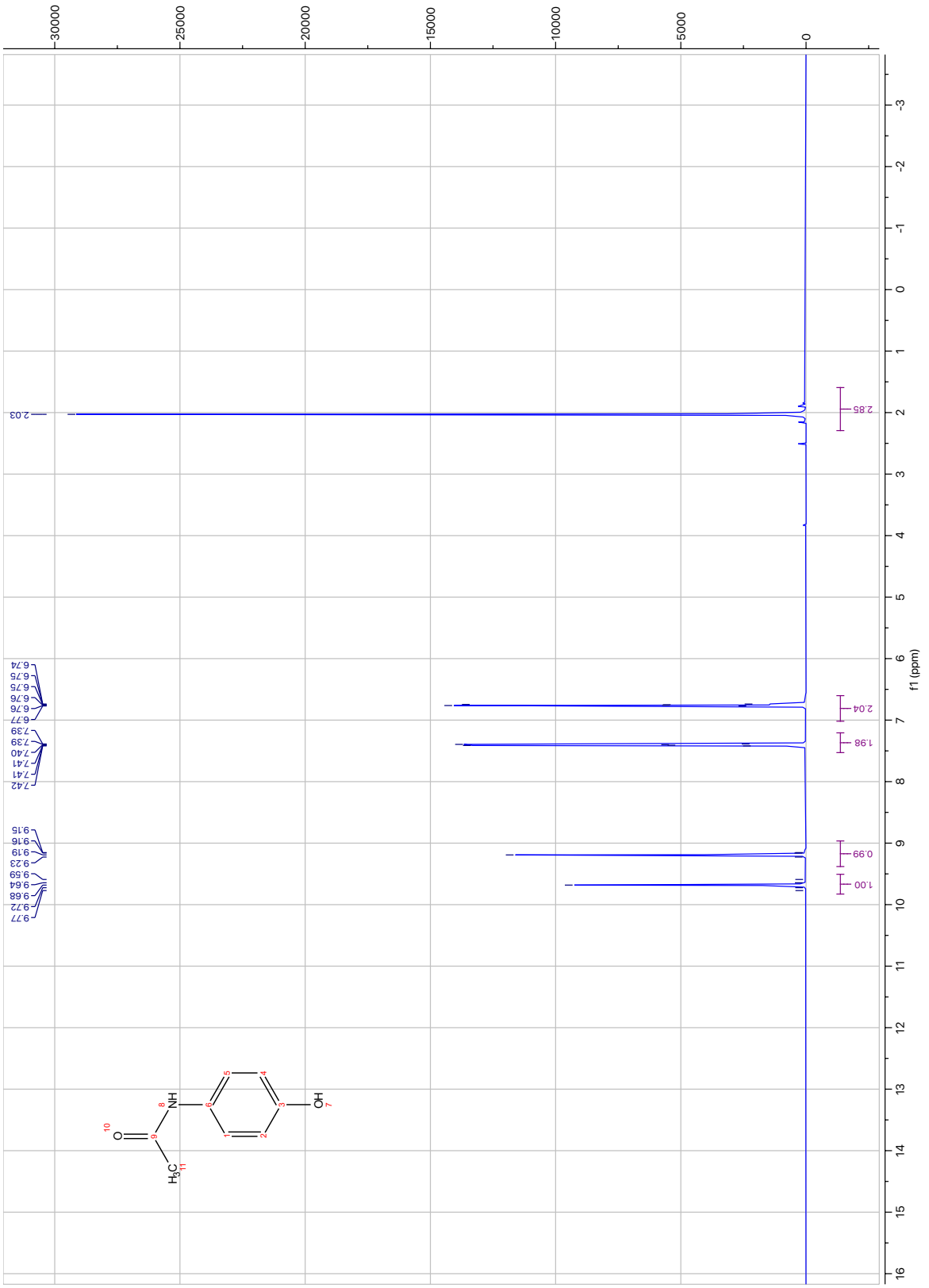
127.30
77.56
77.24
76.92
63.97
f1 (ppm)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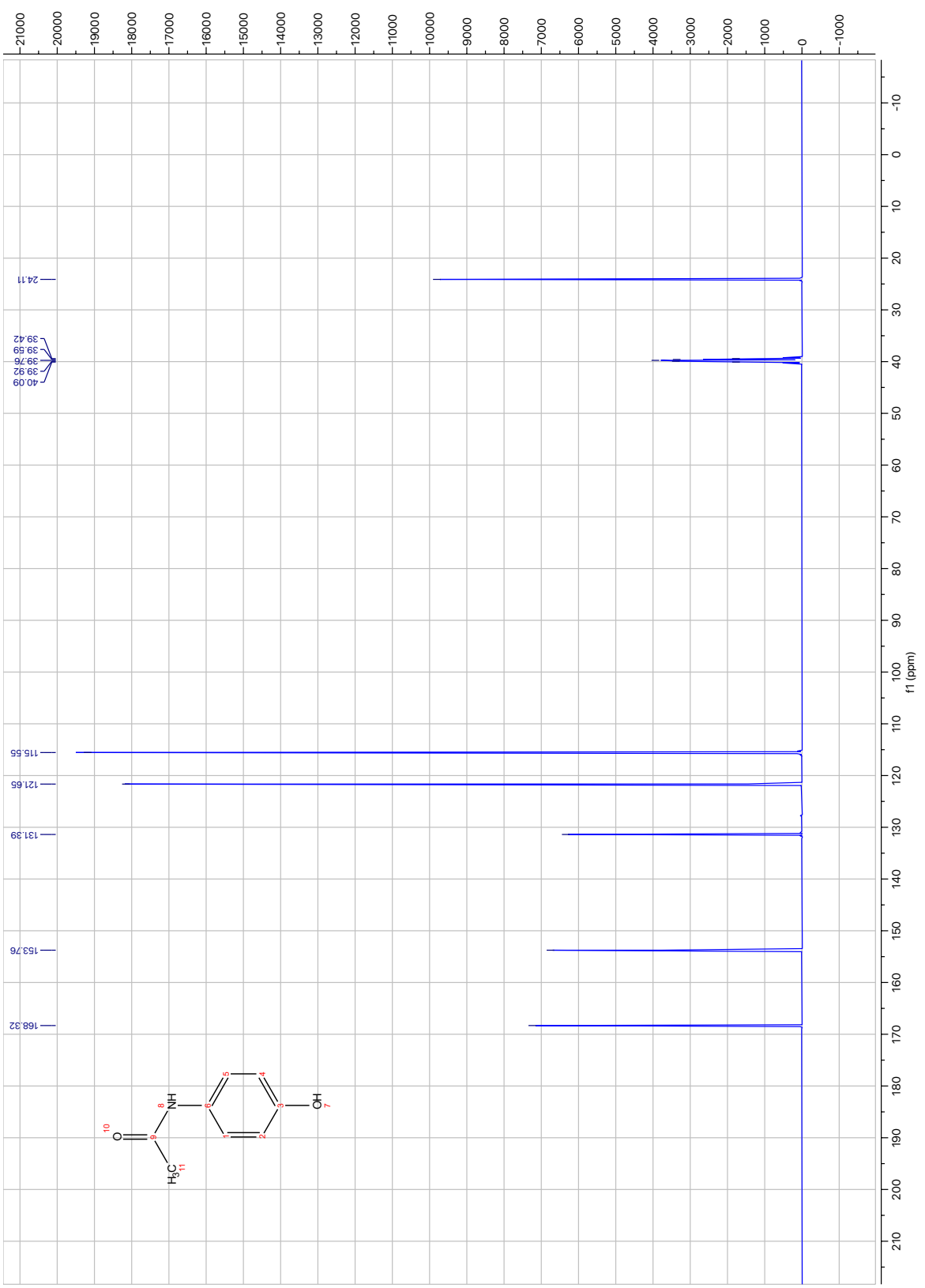
0.99
1.00
9.77
9.72
9.68
9.64
9.59
9.23
9.19
9.16
9.15
7.42
7.41
7.41
7.40
7.39
7.39
6.77
6.76
6.76
6.75
6.75
6.74
NH
OH
O
H3C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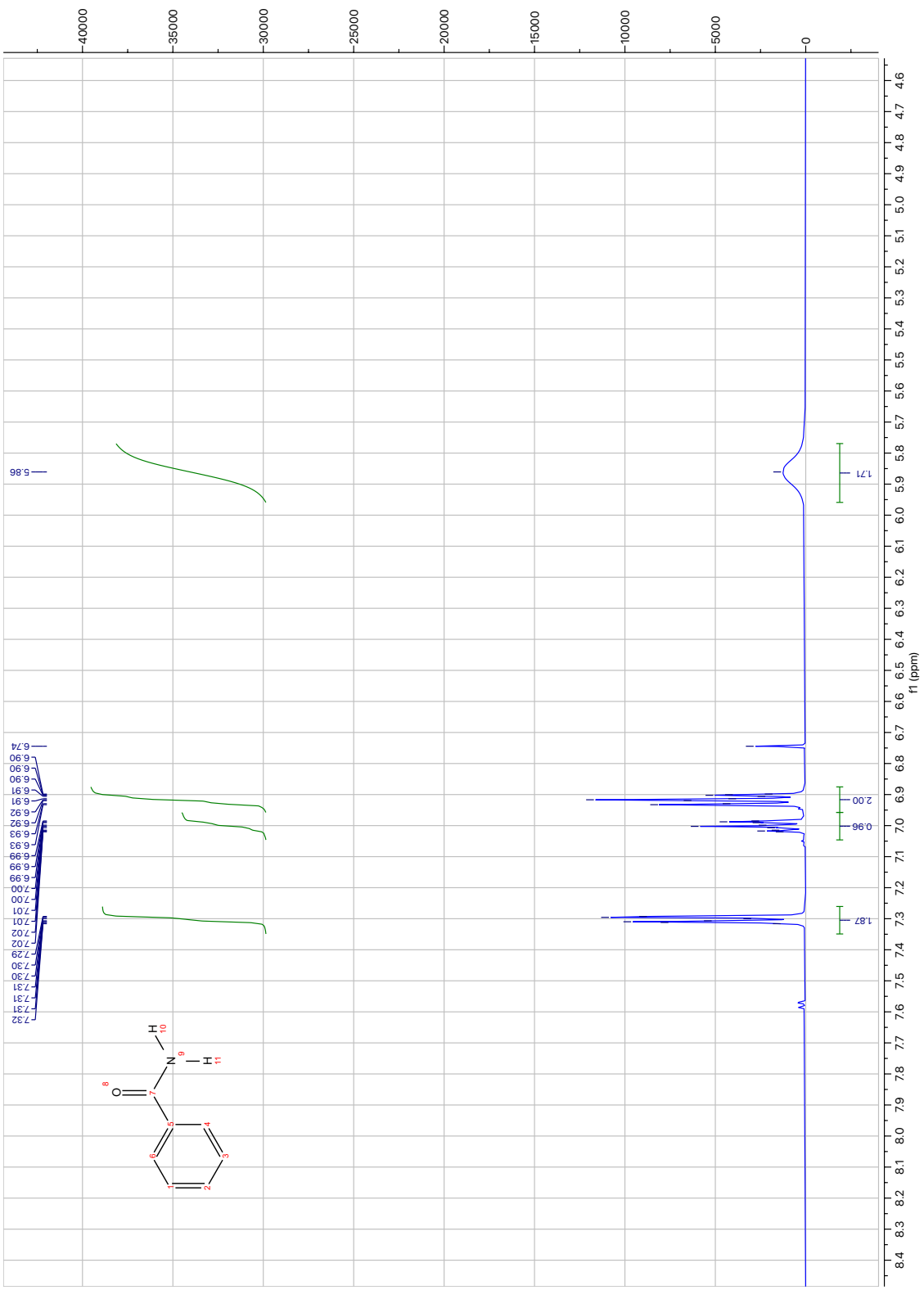
121.65
115.55
40.09
39.92
39.76
39.59
39.42
24.11
f1 (ppm)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6.90
6.90
6.91
6.91
6.92
6.92
6.93
6.93
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7.01
7.01
7.02
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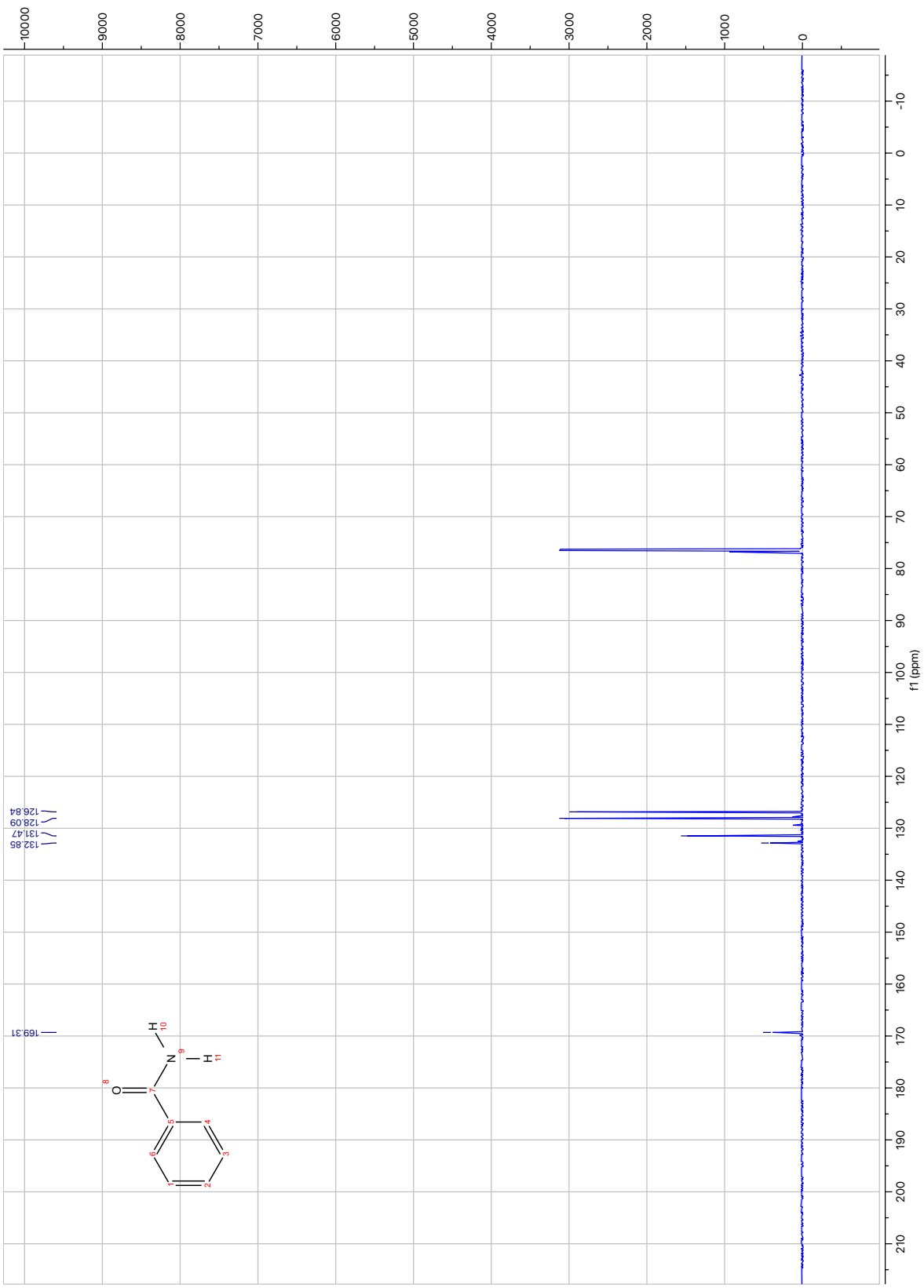
O
N
H
H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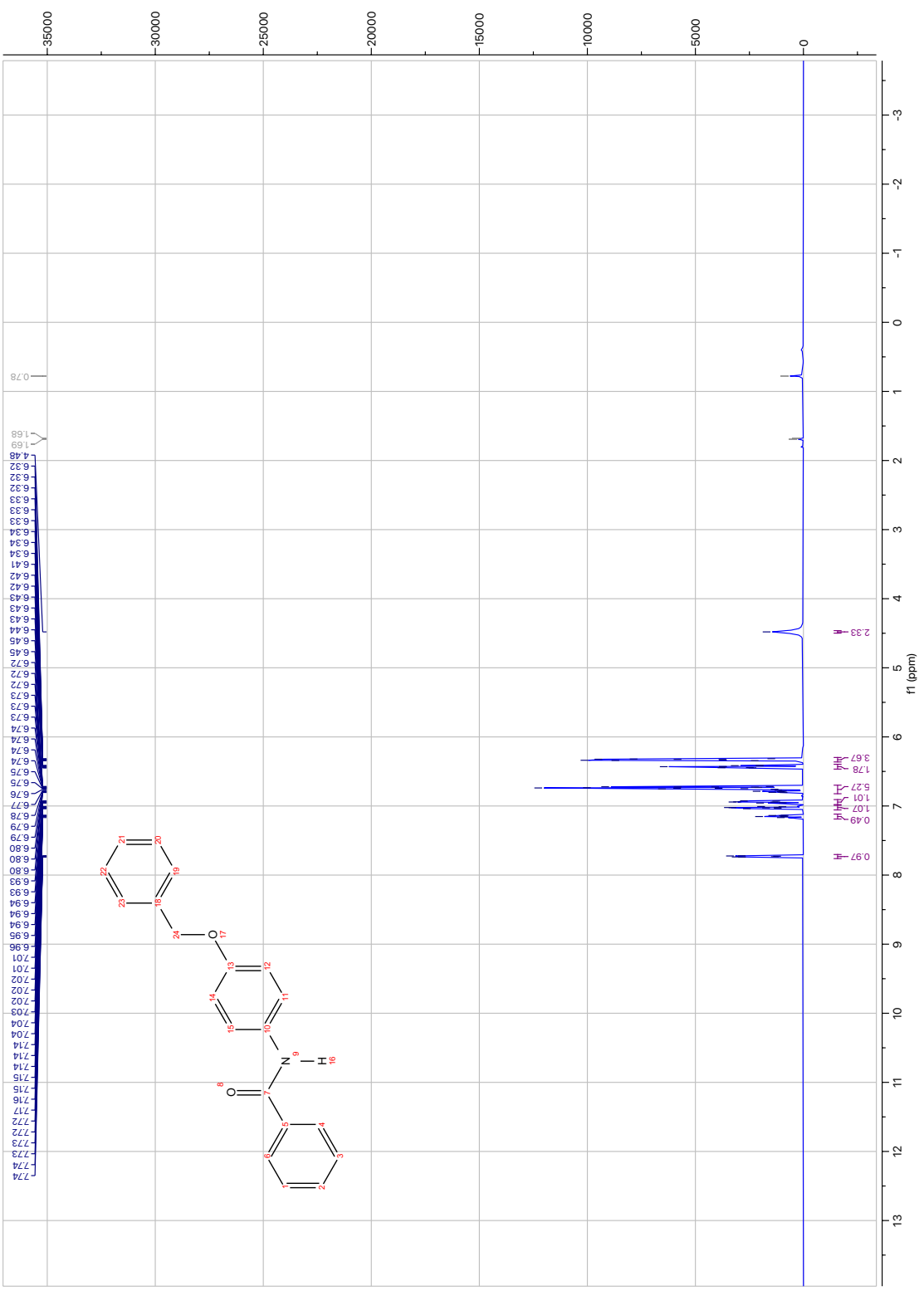
128.84
f1 (ppm)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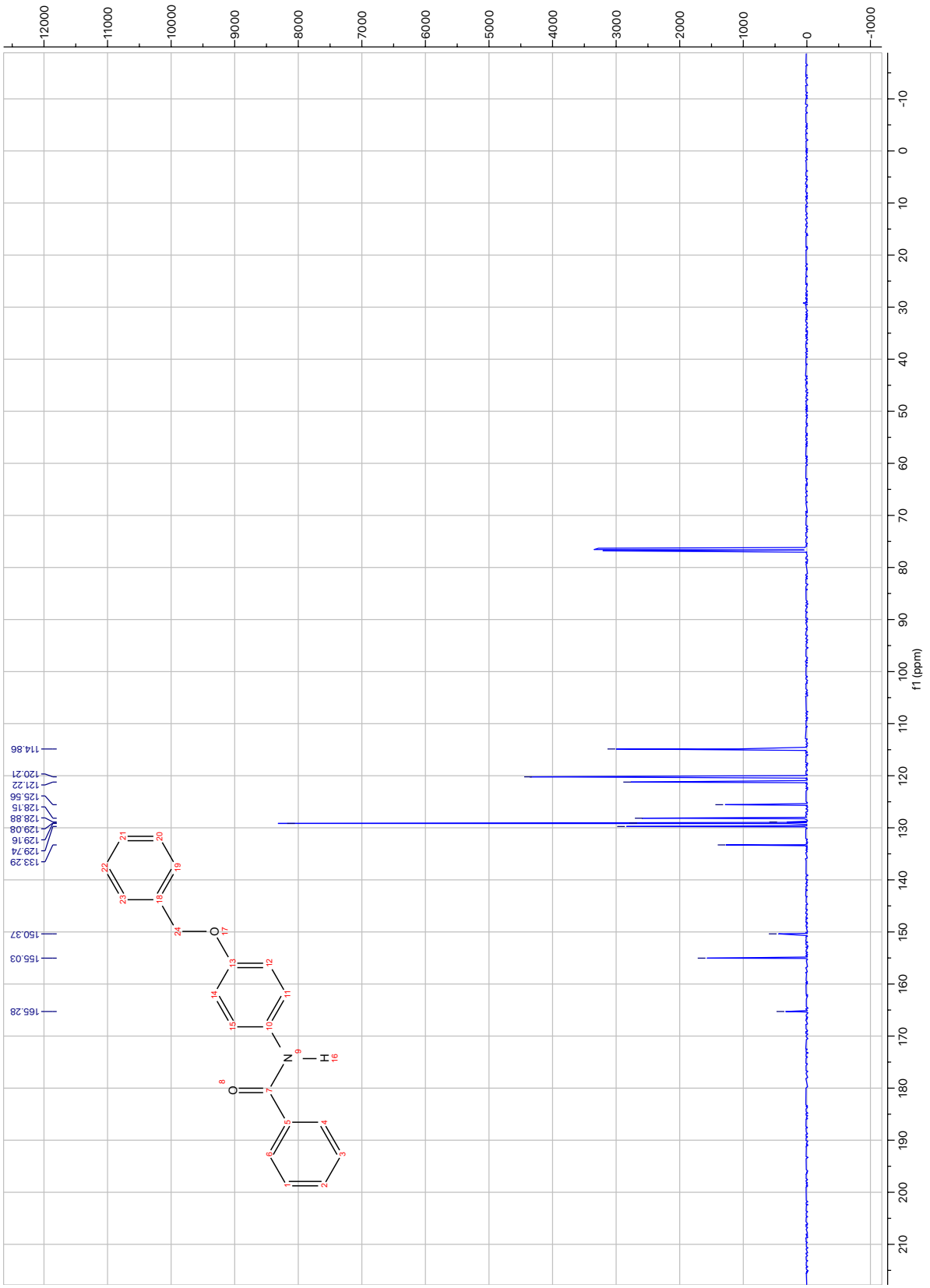
1.68
4.48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129.08
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129.74
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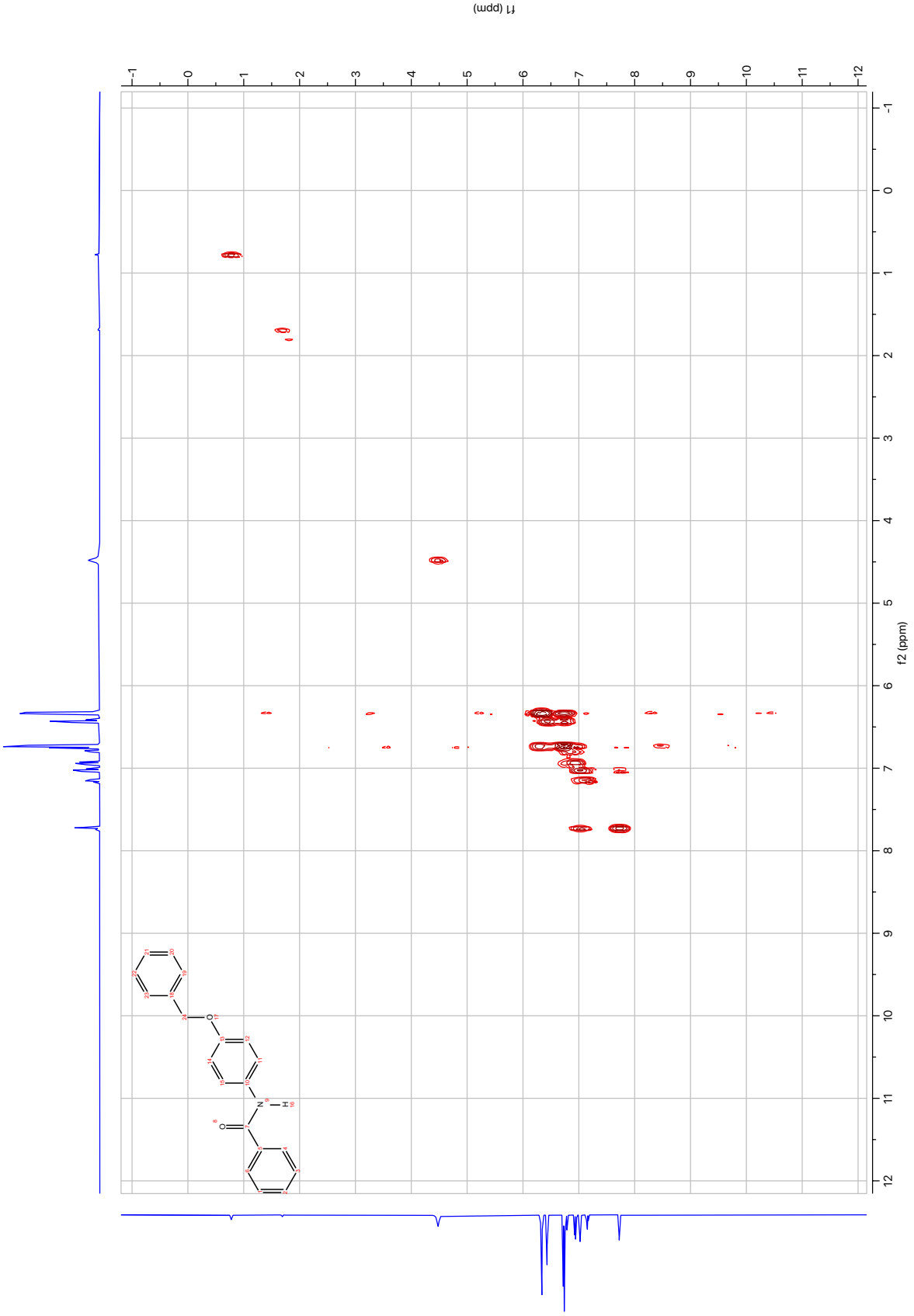
165.28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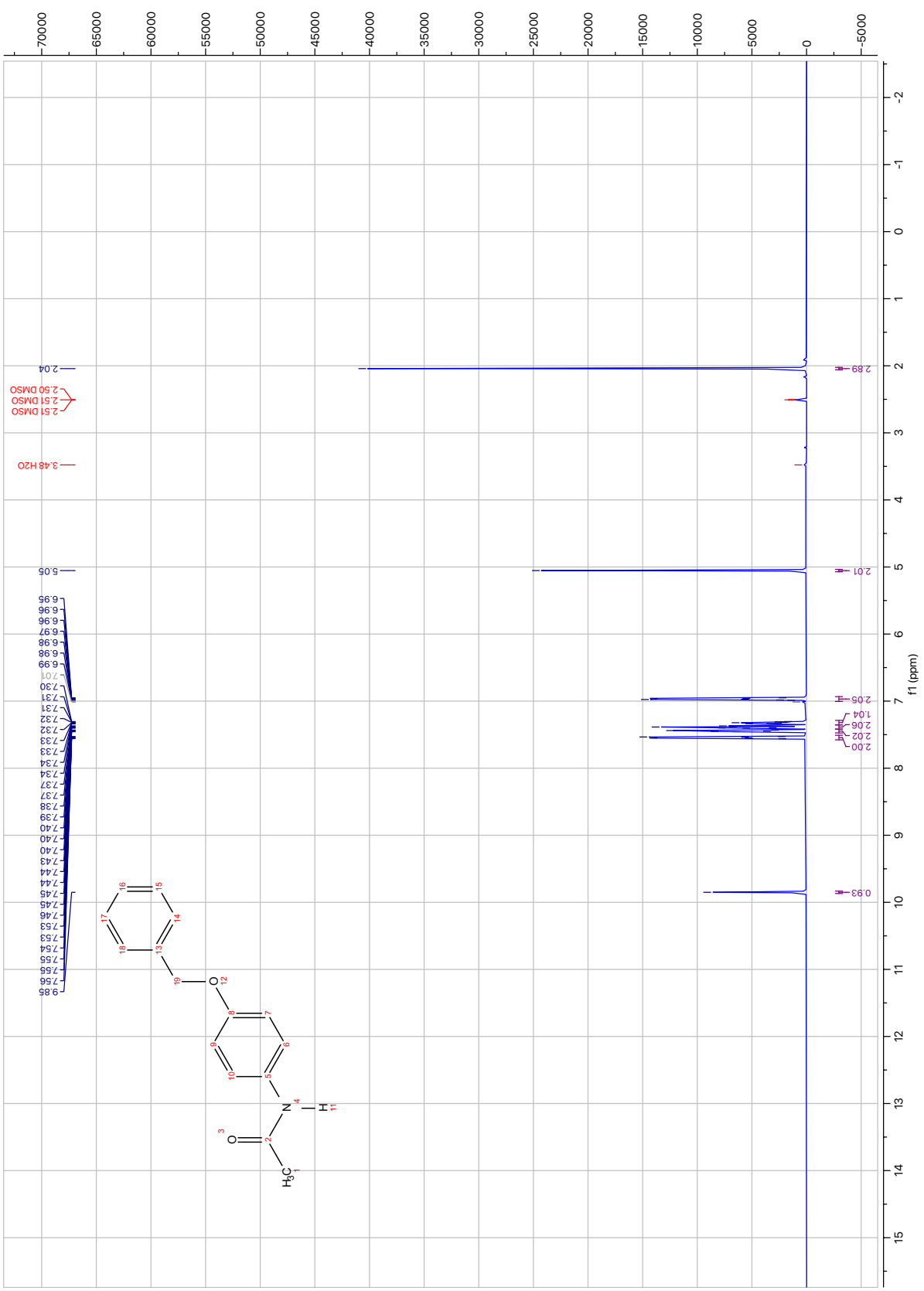
f2 (ppm)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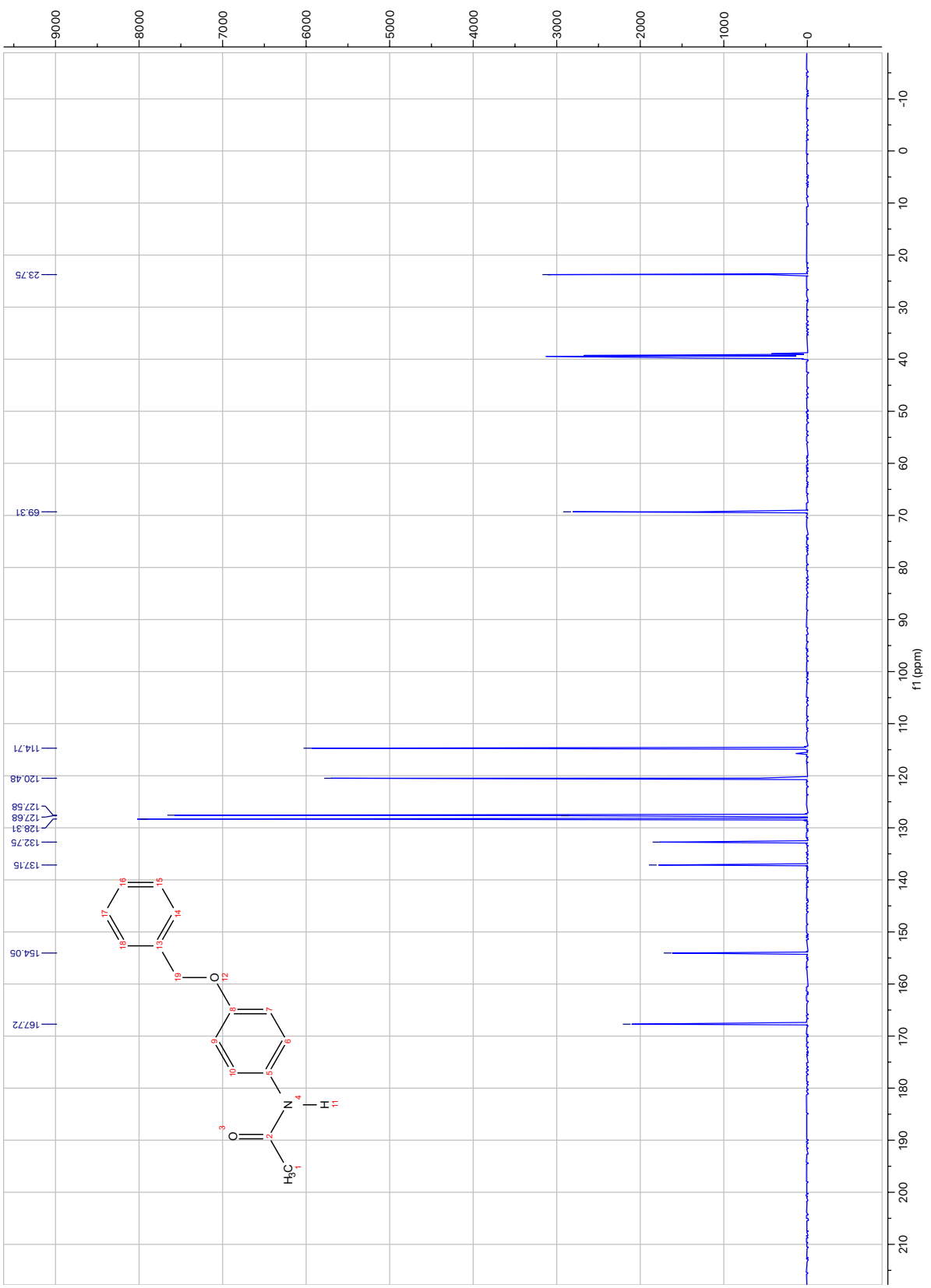
2.05
1.04
2.06
2.02
2.00
0.93
2.04
2.51 DMSO
2.51 DMSO
2.50 DMSO
3.48 H2O
5.05
9.85
H3C
O
N
H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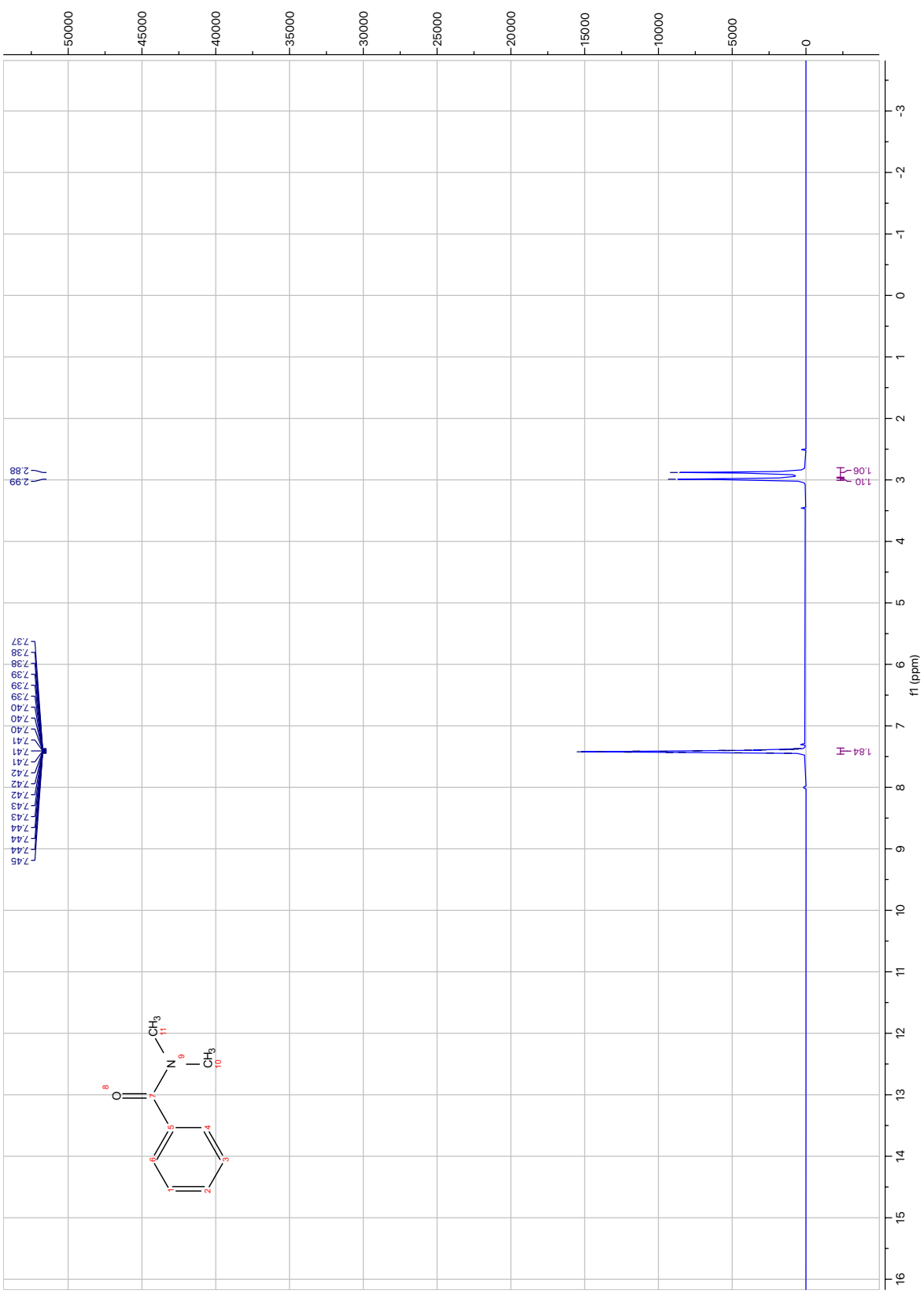
128.31
132.75
137.15
154.05
167.72
f1 (ppm)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15000
10000
5000
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-2
-1
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1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
f1 (ppm)
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1.06
1.10
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2.99
7.37
7.38
7.38
7.39
7.39
7.39
7.40
7.40
7.40
7.41
7.41
7.41
7.42
7.42
7.42
7.43
7.43
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7.44
7.45
CH3
N
CH3
O


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30000
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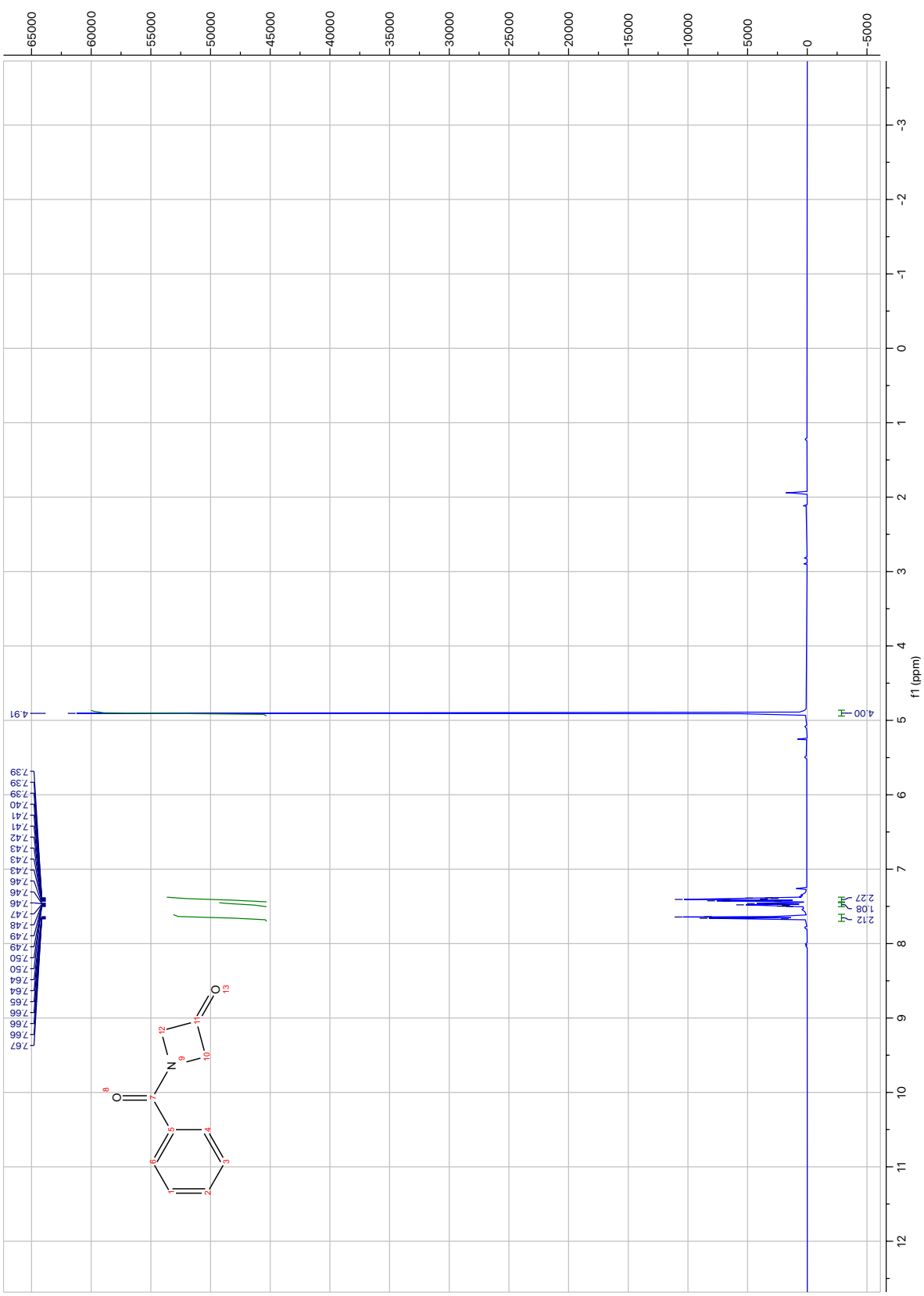
129.71
137.03
170.56
f1 (ppm)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7.46
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7.43
7.42
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7.41
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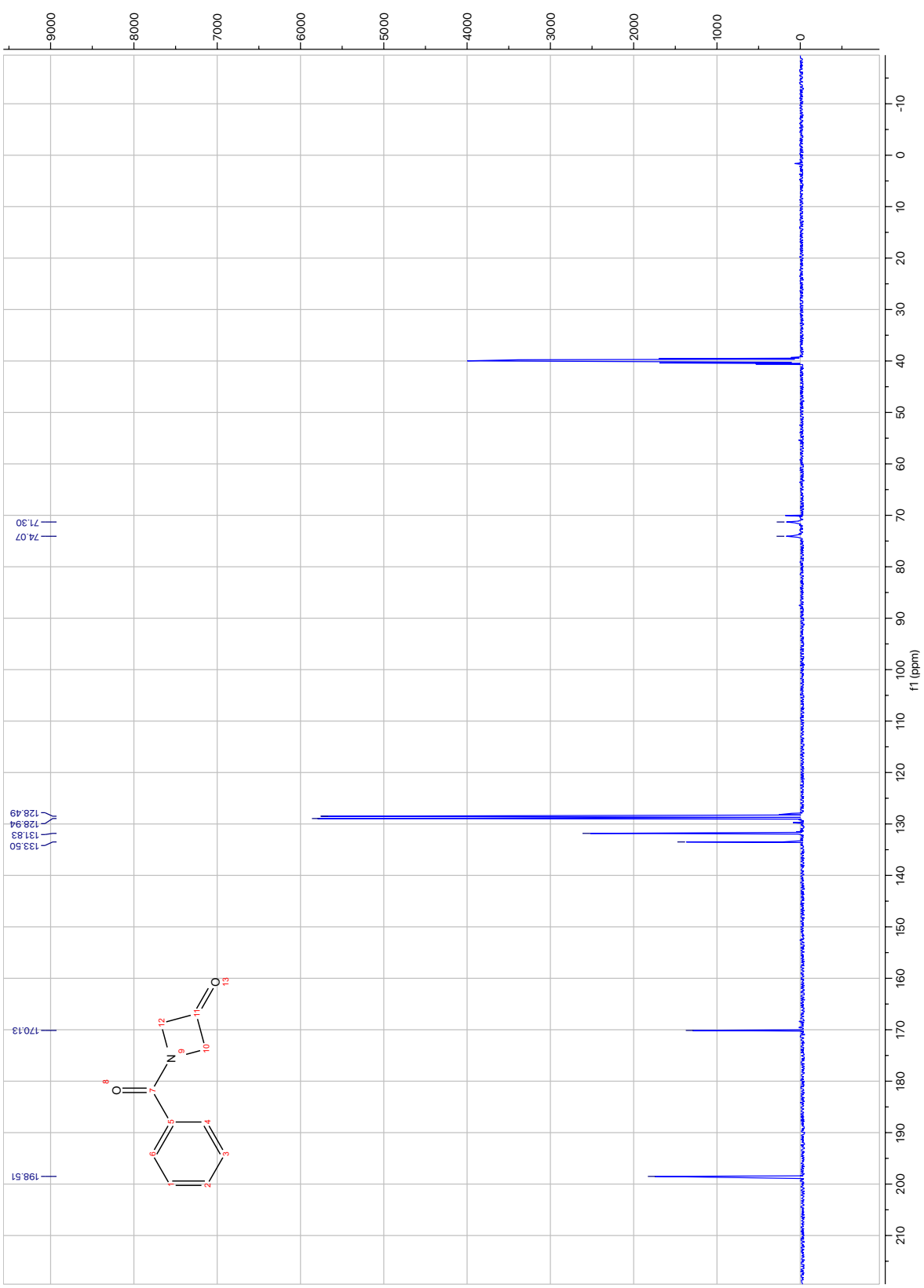
7.39
7.39
7.39
O
O
N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128.49
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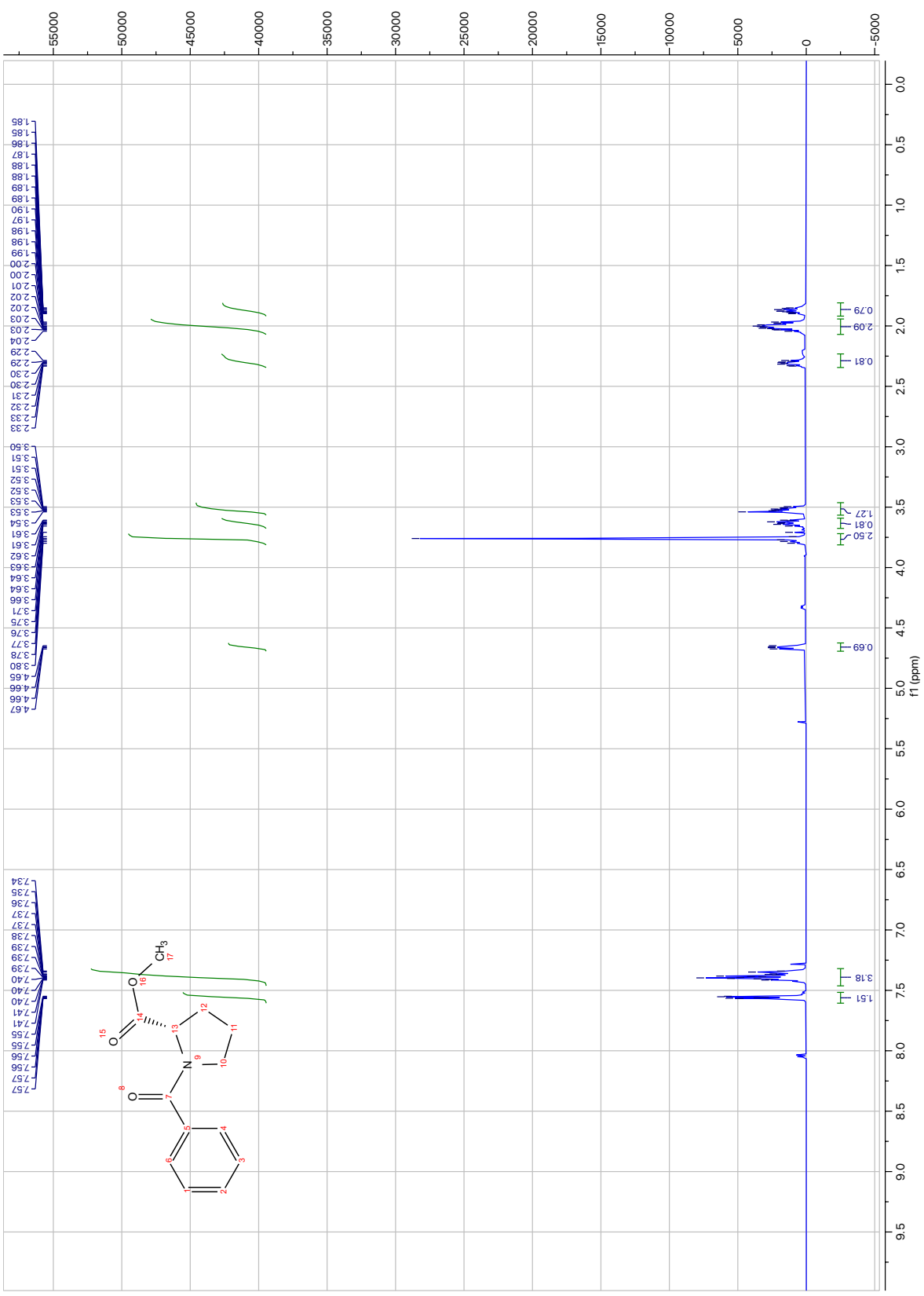
71.30
f1 (ppm)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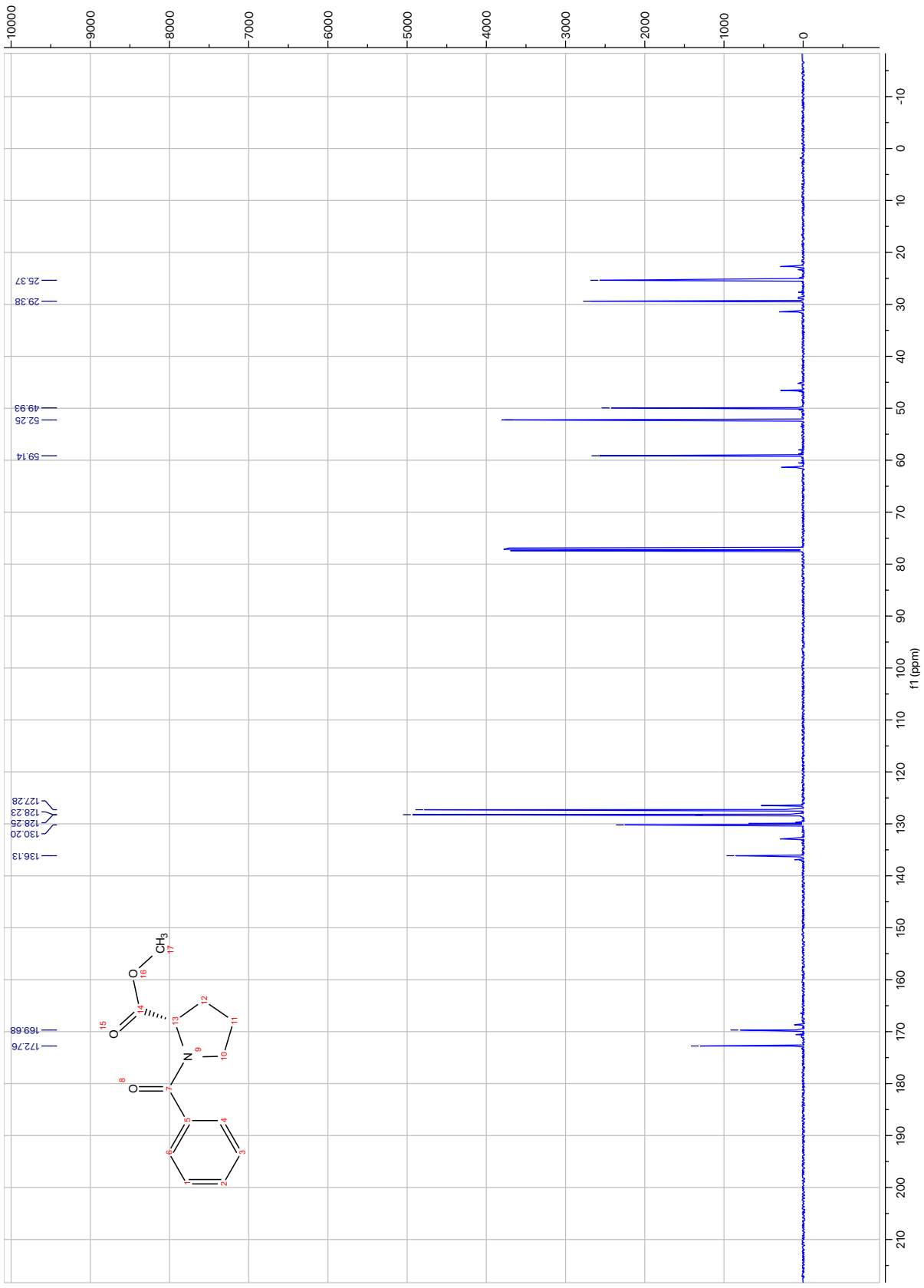
0.81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5000
4000
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2000
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20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
f1 (ppm)
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29.38
49.93
52.25
59.14
127.28
128.23
128.25
130.20
136.13
169.68
172.76
CH3
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16
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14
13
12
11
10
N
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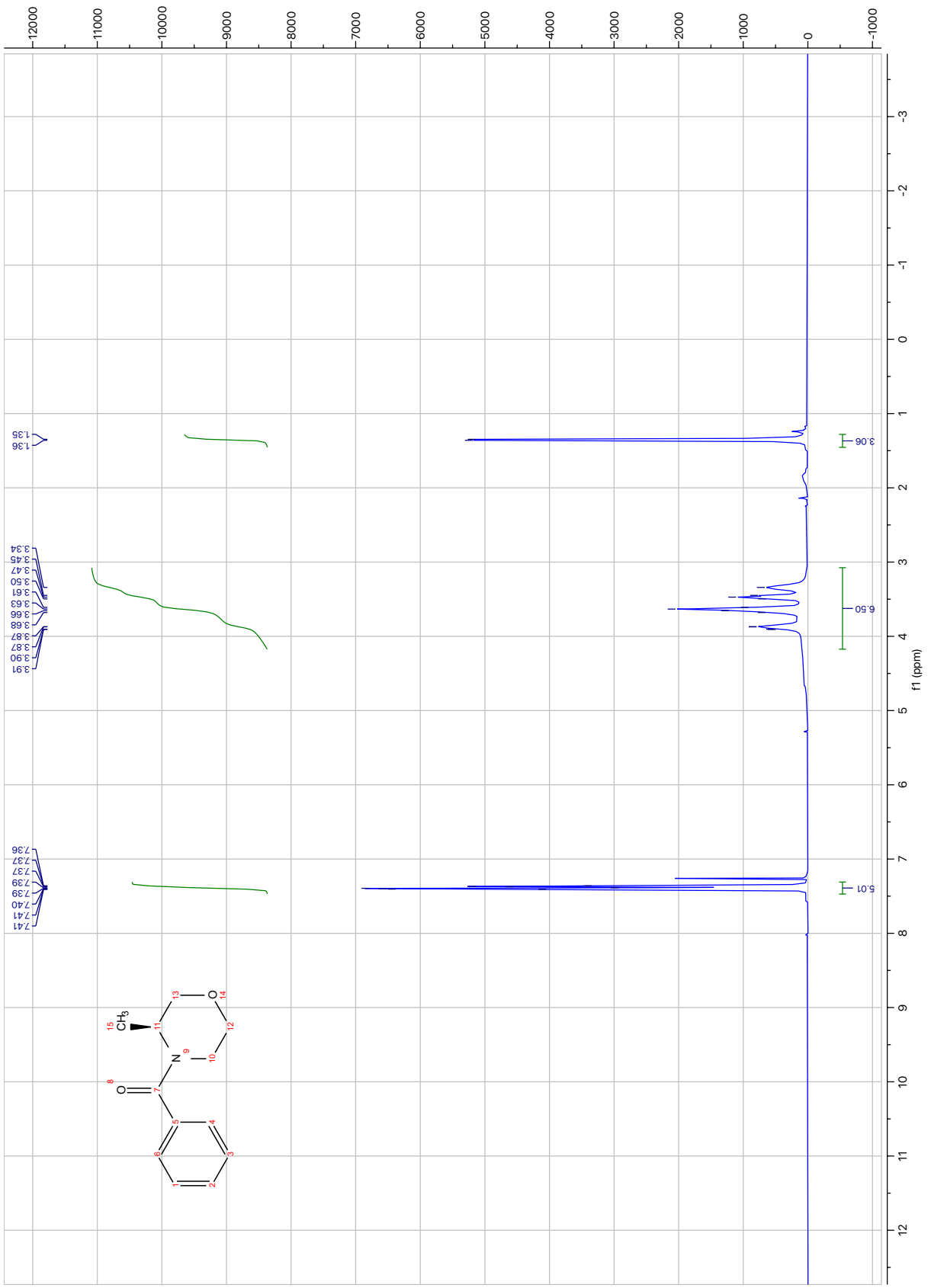
O
8
7
6
5
4
3
2
1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6000
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1000
0
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12
11
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9
8
7
6
5
4
3
2
1
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-1
-2
-3
f1 (ppm)
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7.39
7.39
7.37
7.37
7.36
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3.90
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3.63
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6.50
3.06
O
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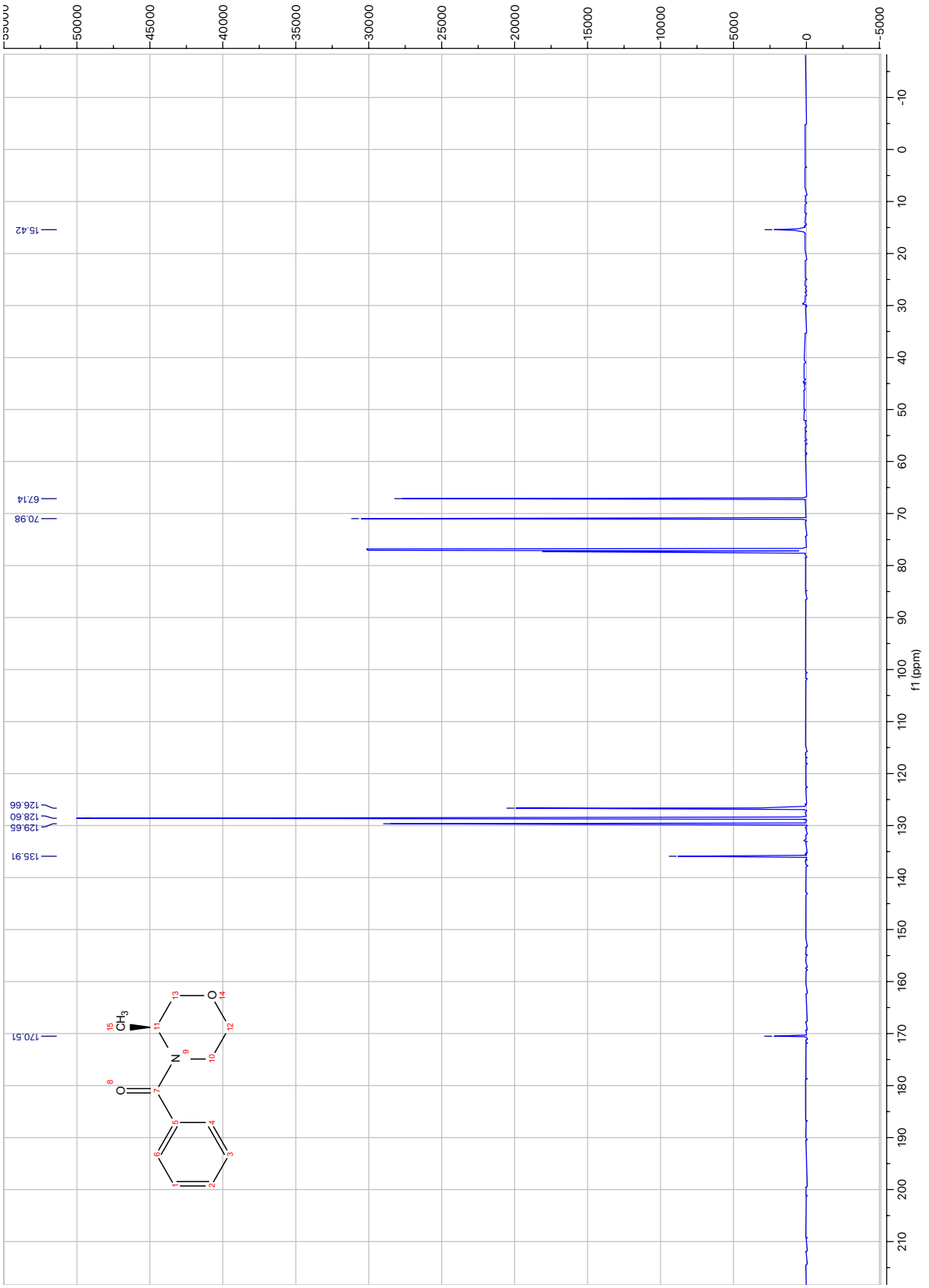
O
CH3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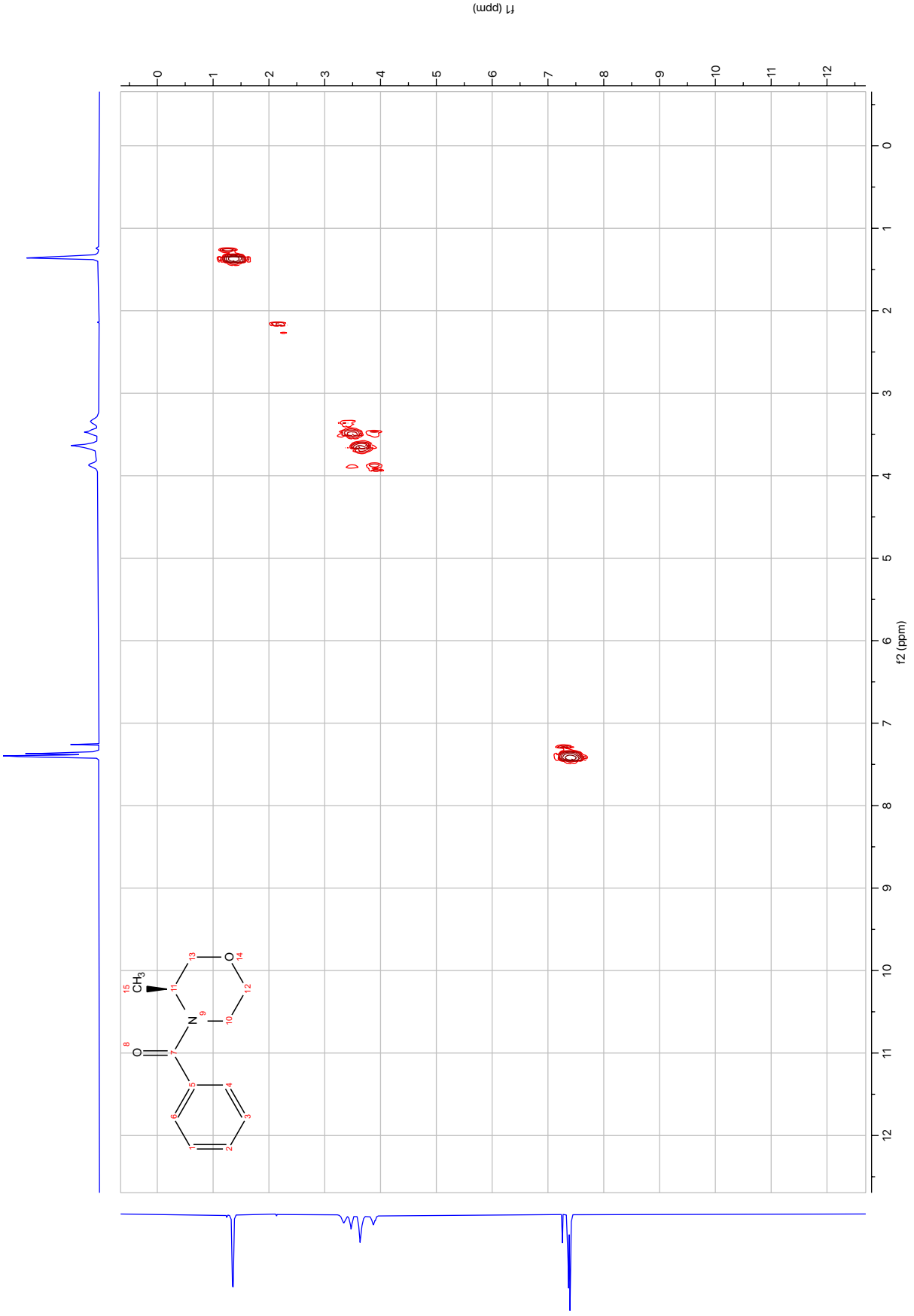
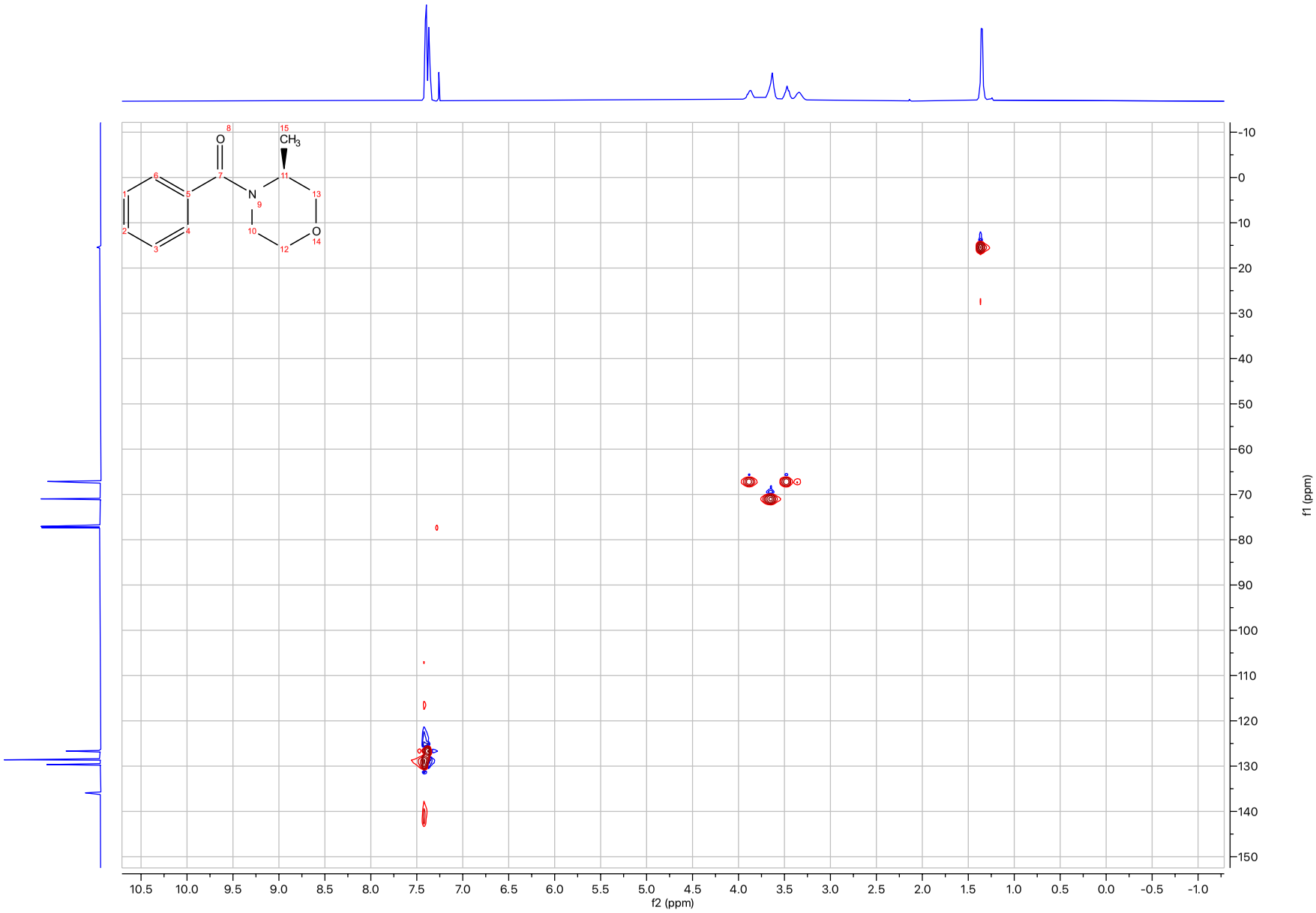
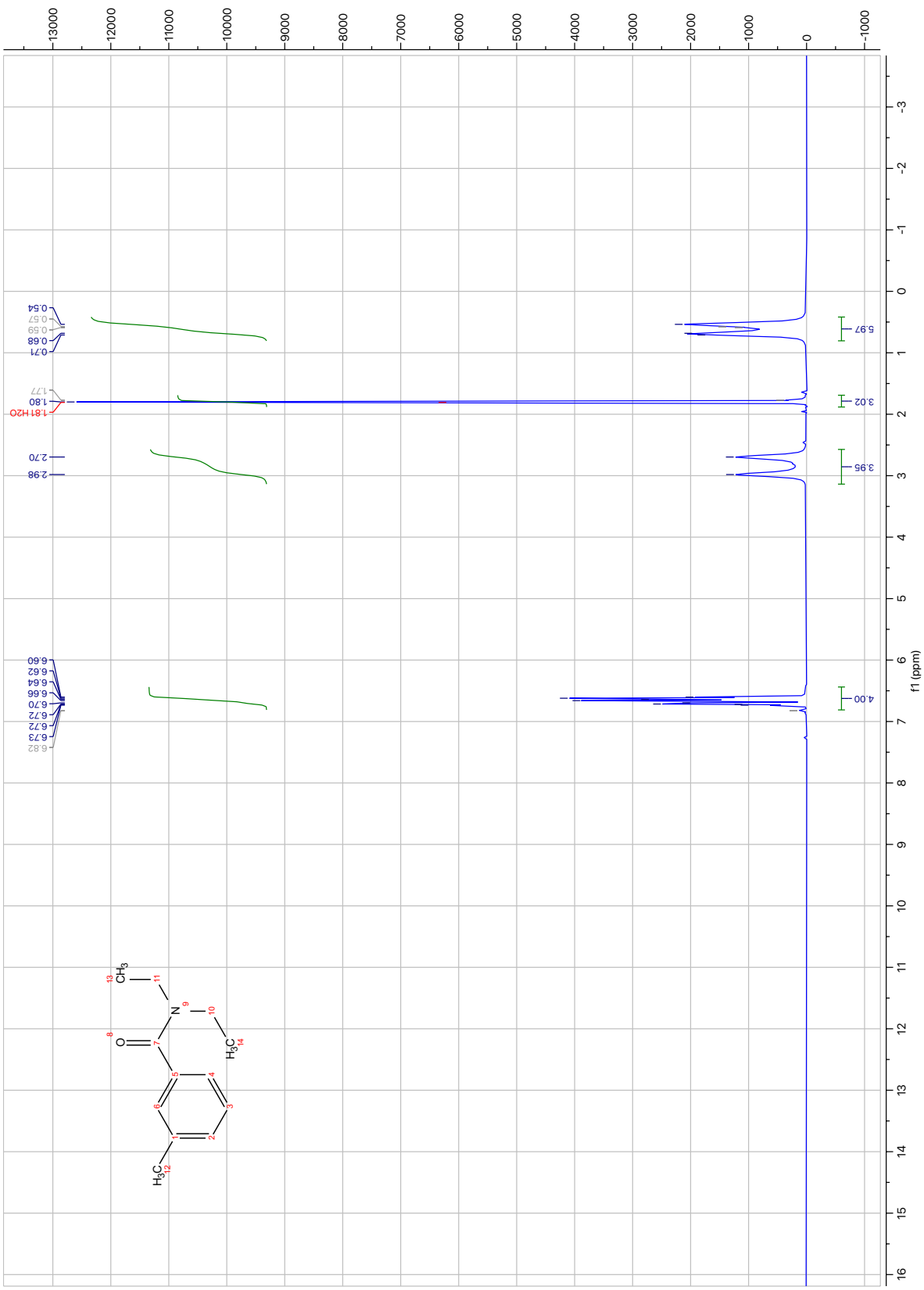
15.42
f1 (ppm)


f1 (ppm)
f2 (ppm)


O
CH3
N
O
f2 (ppm)
f1 (ppm)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3.95
4.00
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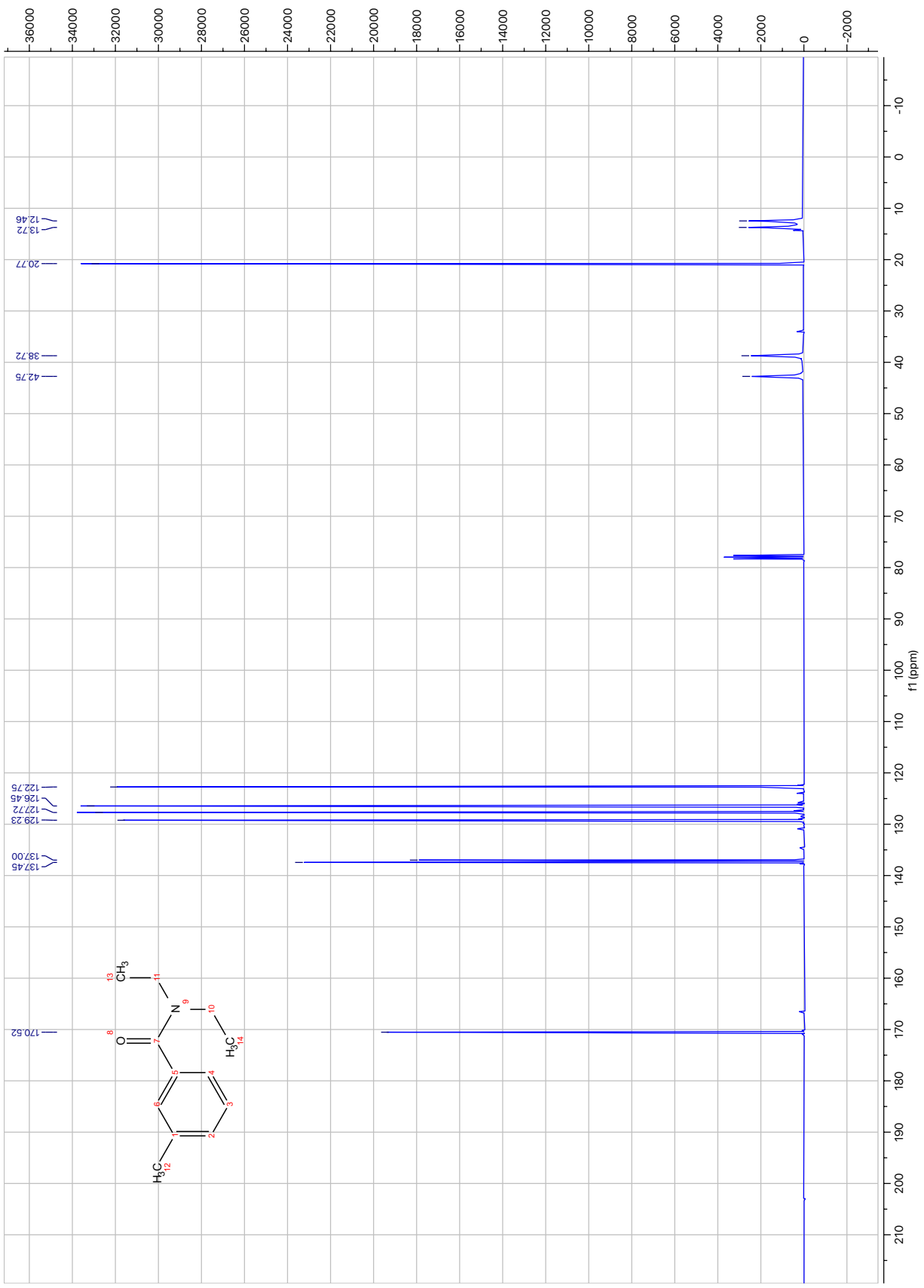
CH3
H3C
H3C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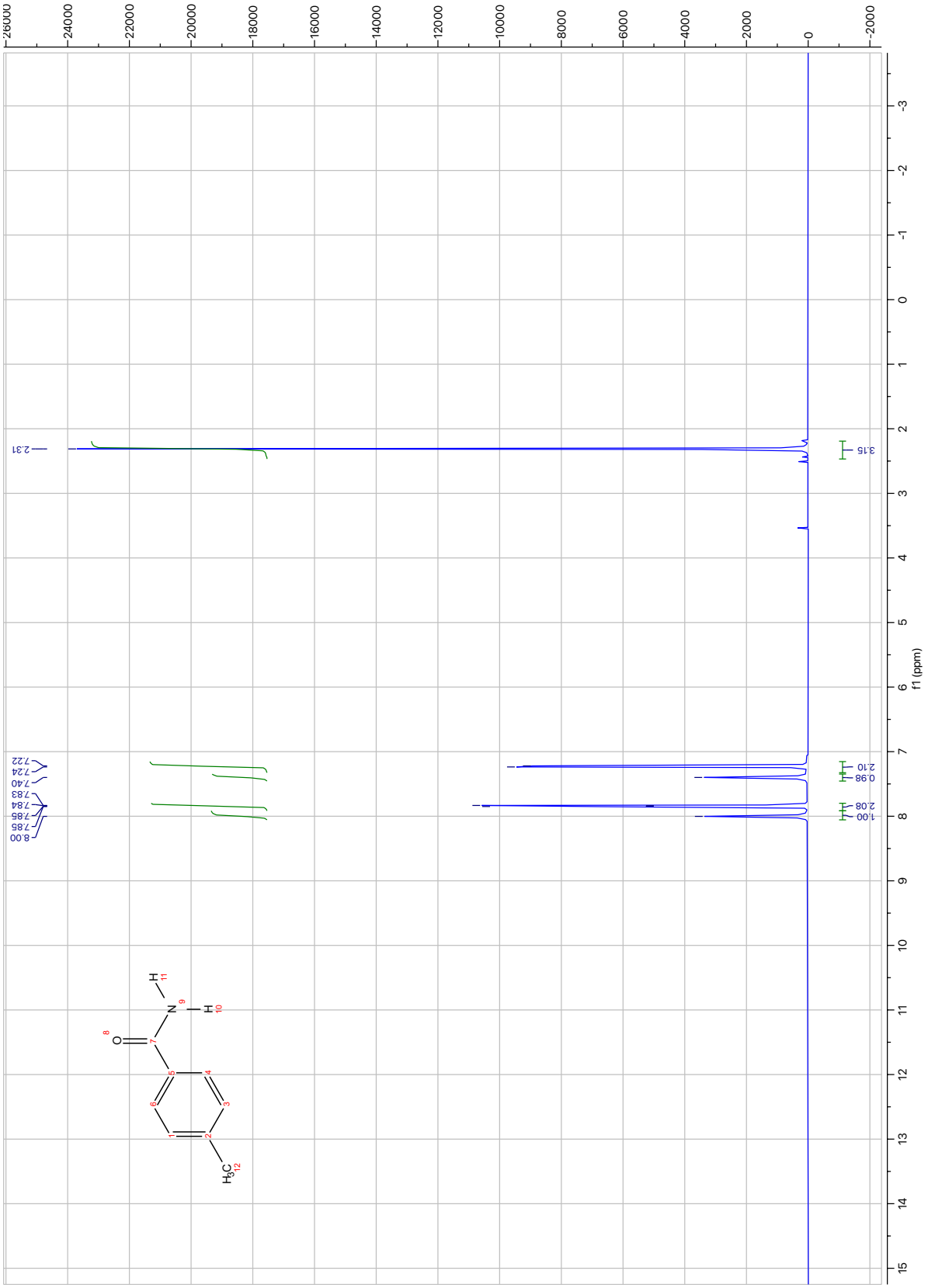
12.46
f1 (ppm)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7.85
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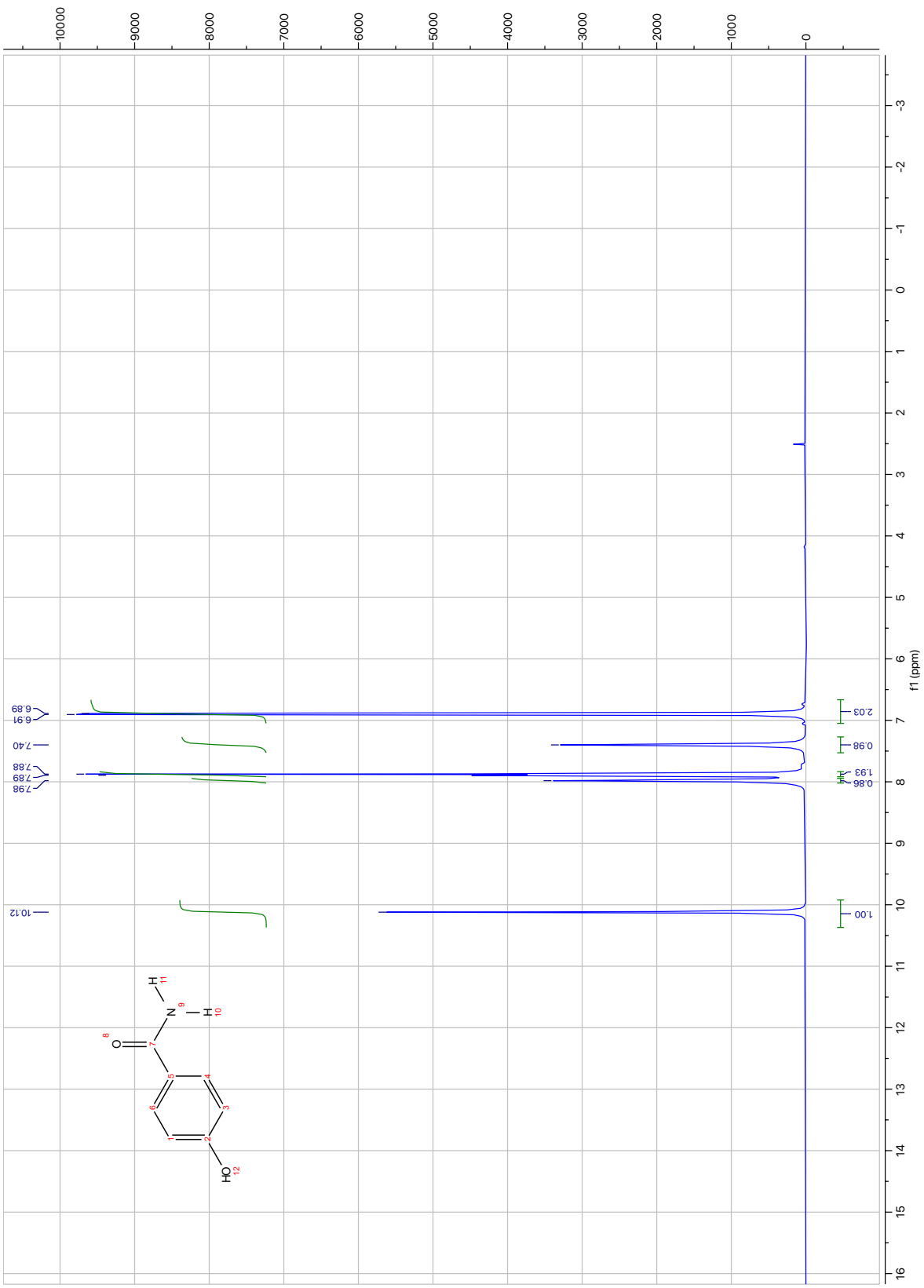
7.24
7.22


168.53
141.56
131.94
129.19
128.02
21.35
f1 (ppm)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7.40
6.91
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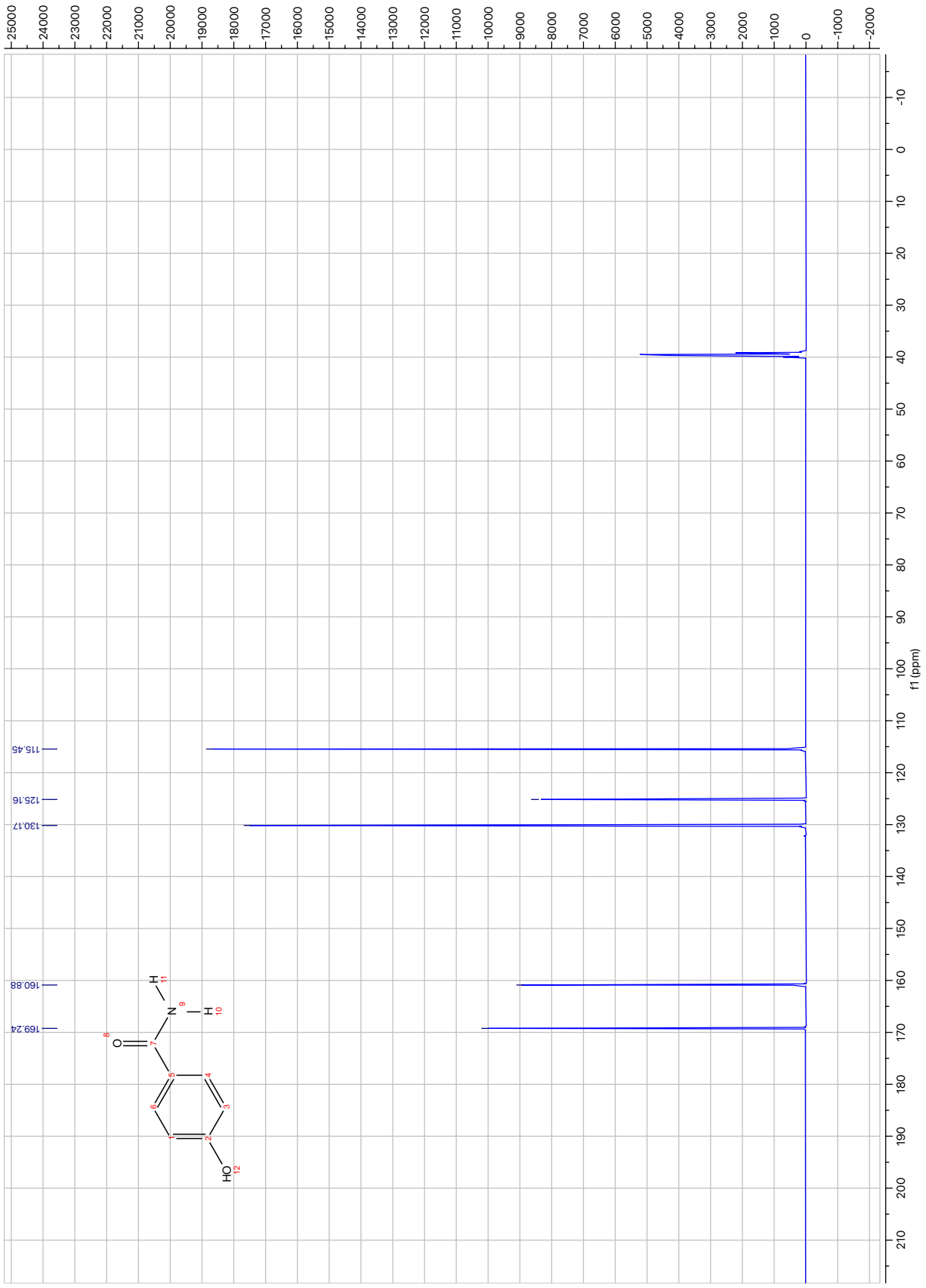
0.86
1.93
0.98
2.03
f1 (ppm)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115.45
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f1 (ppm)
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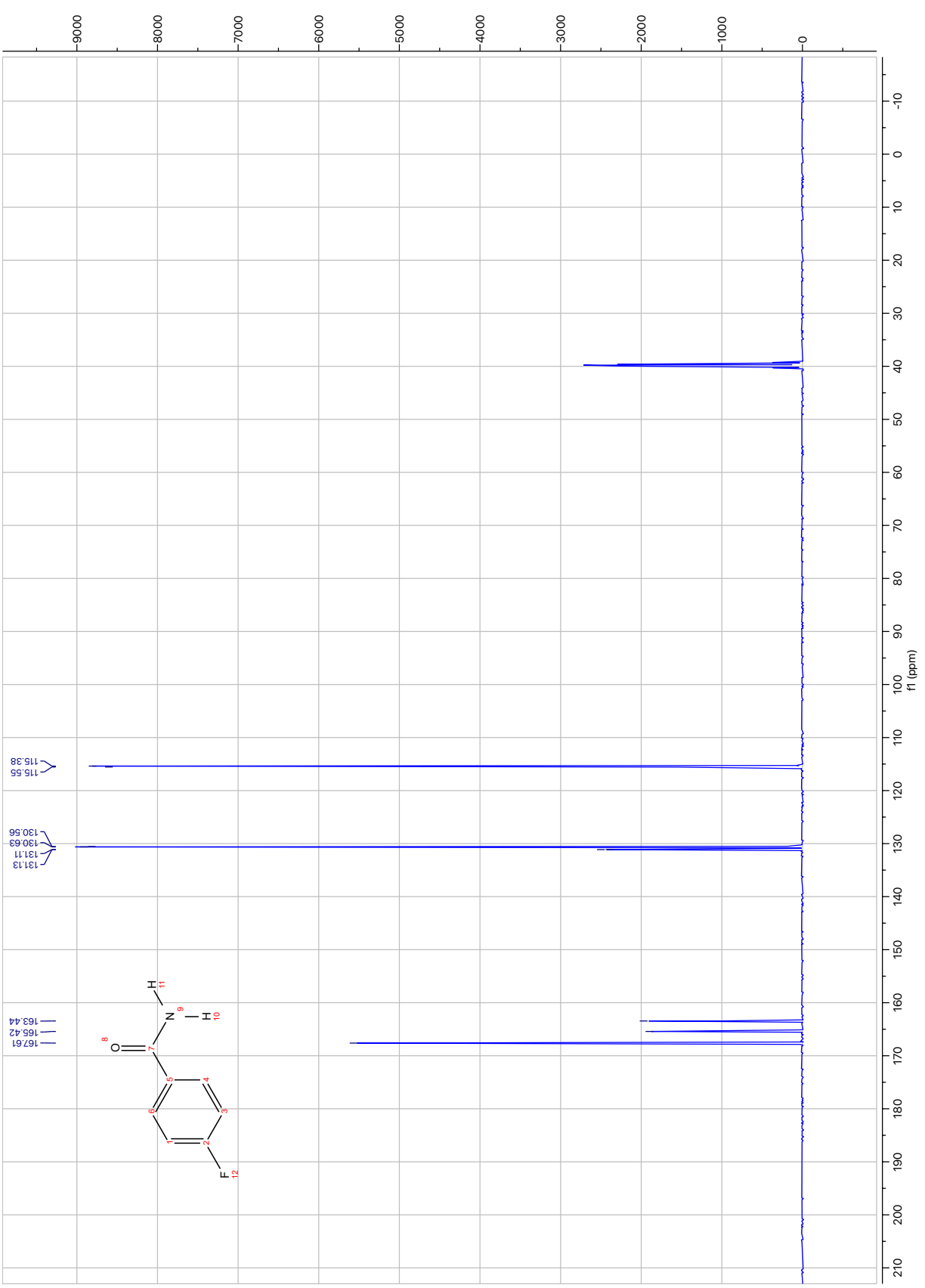
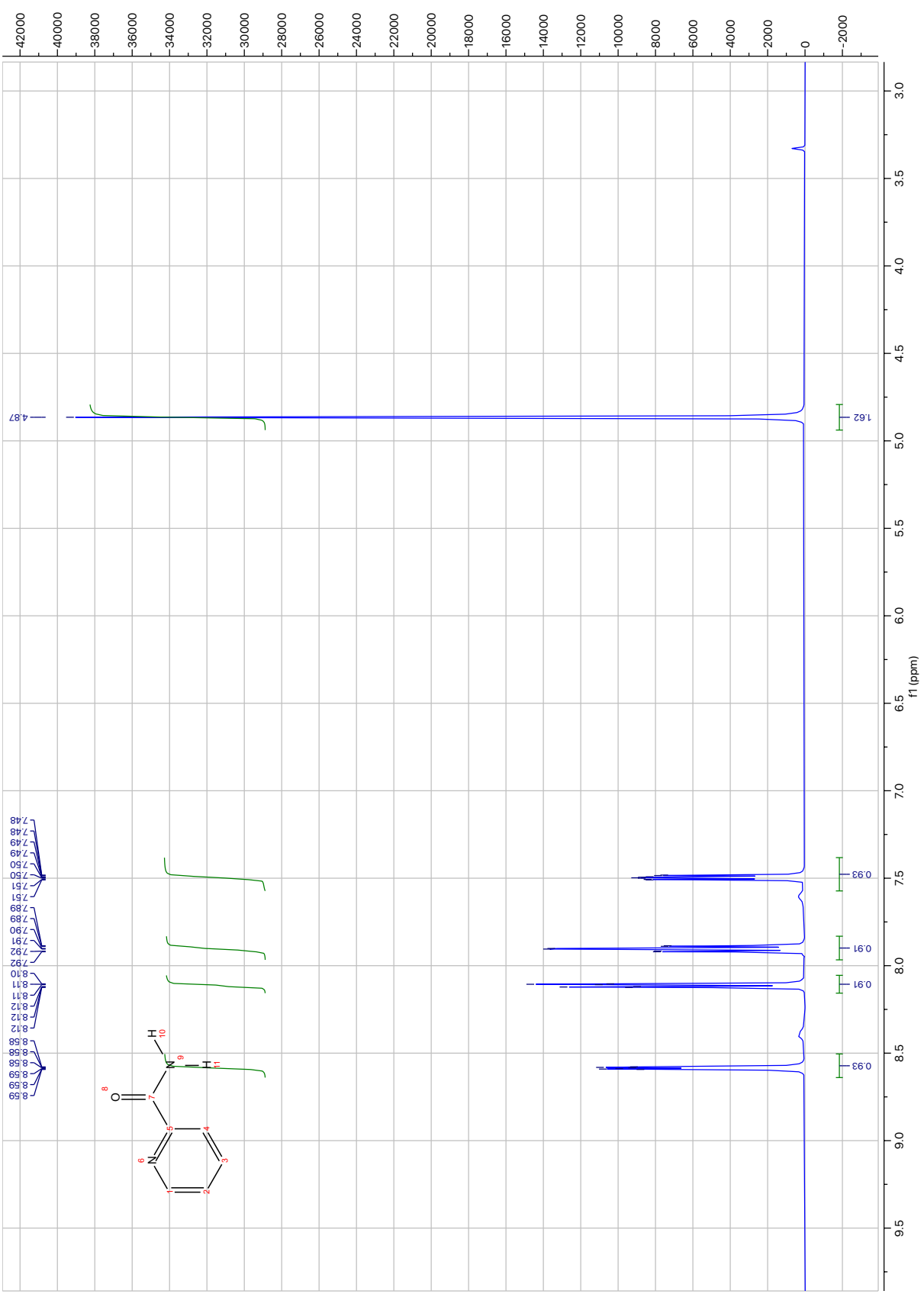
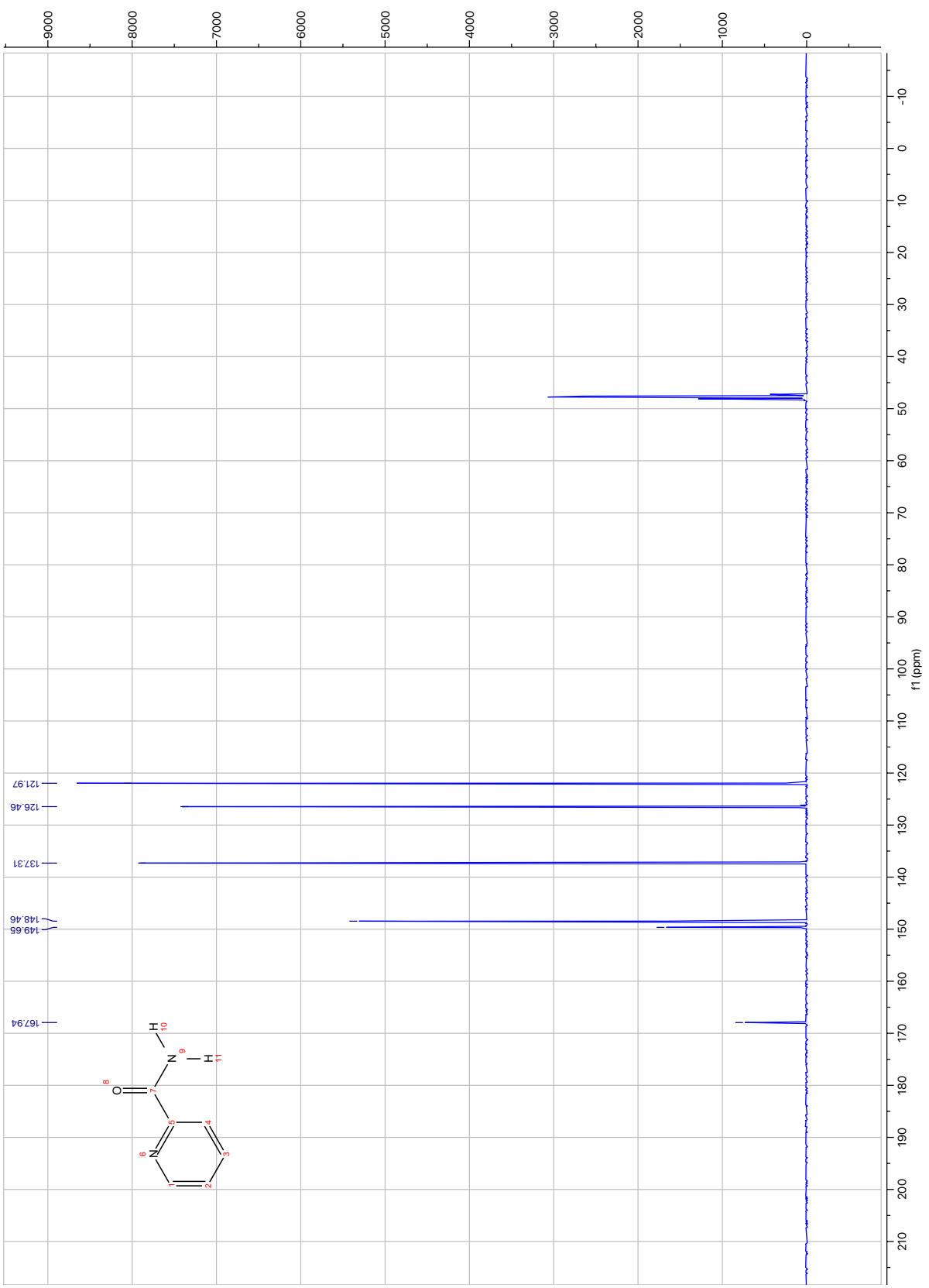
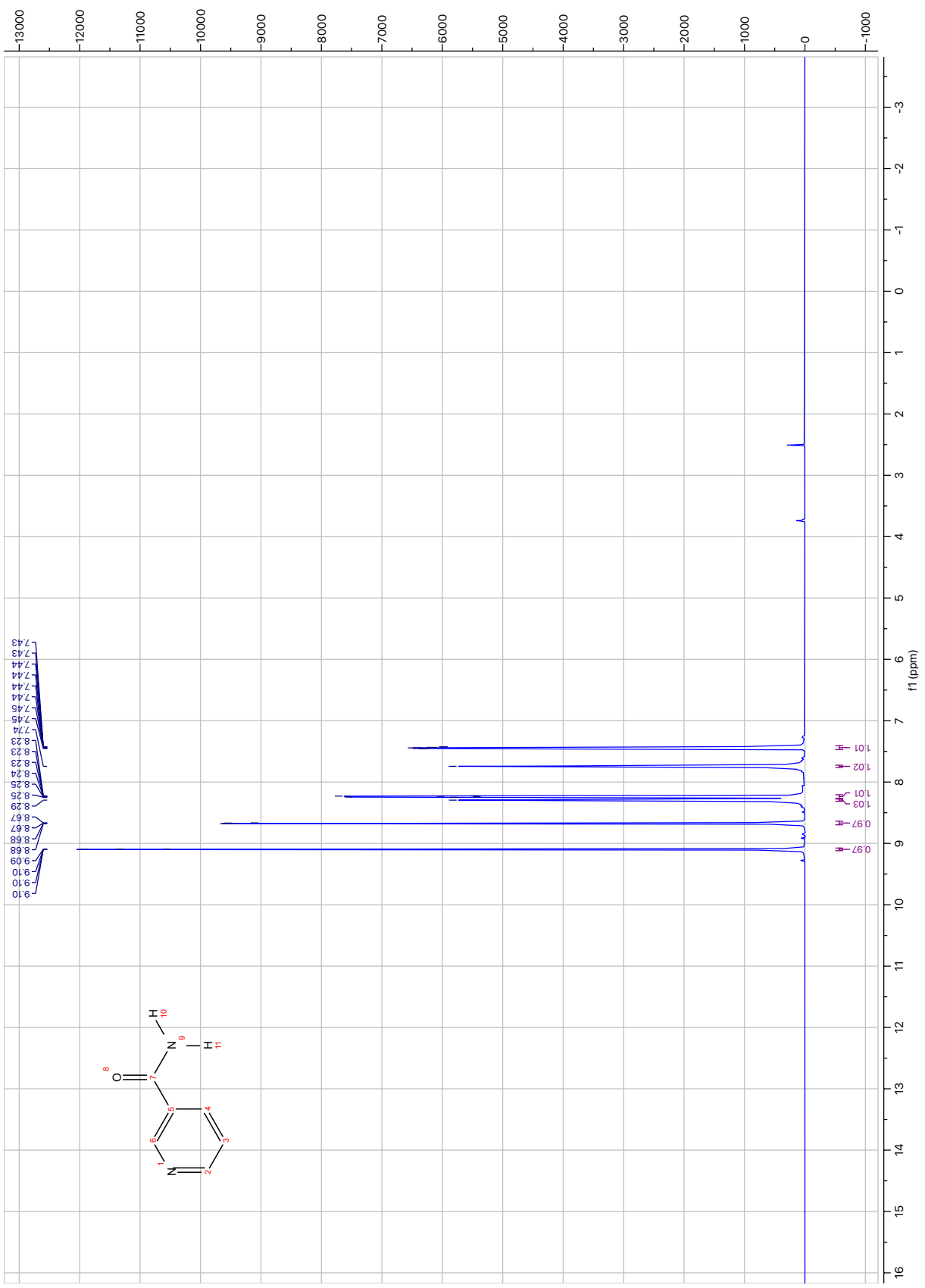
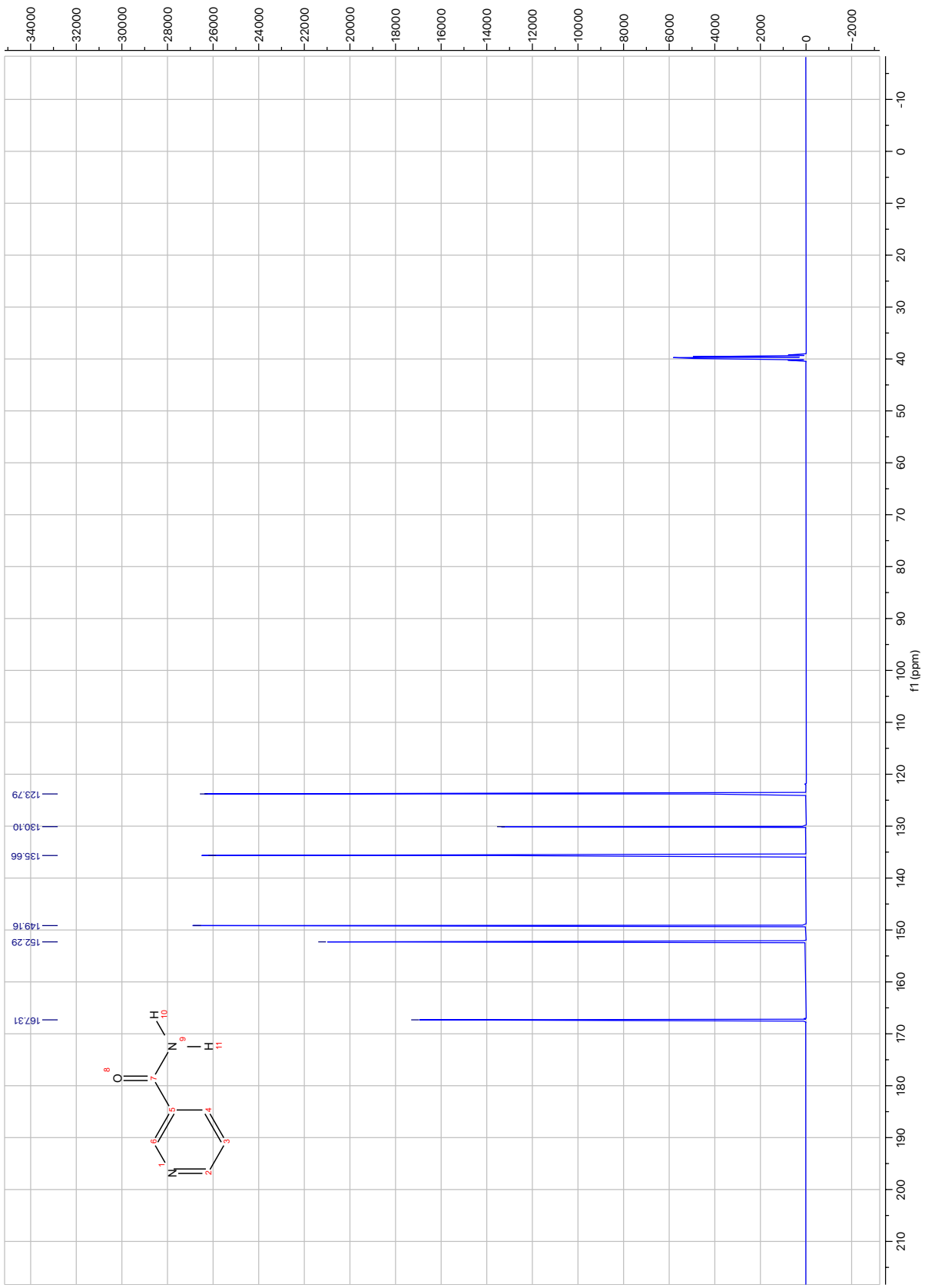
7.22
7.22
7.21
2.14
1.00
2.18
1.00

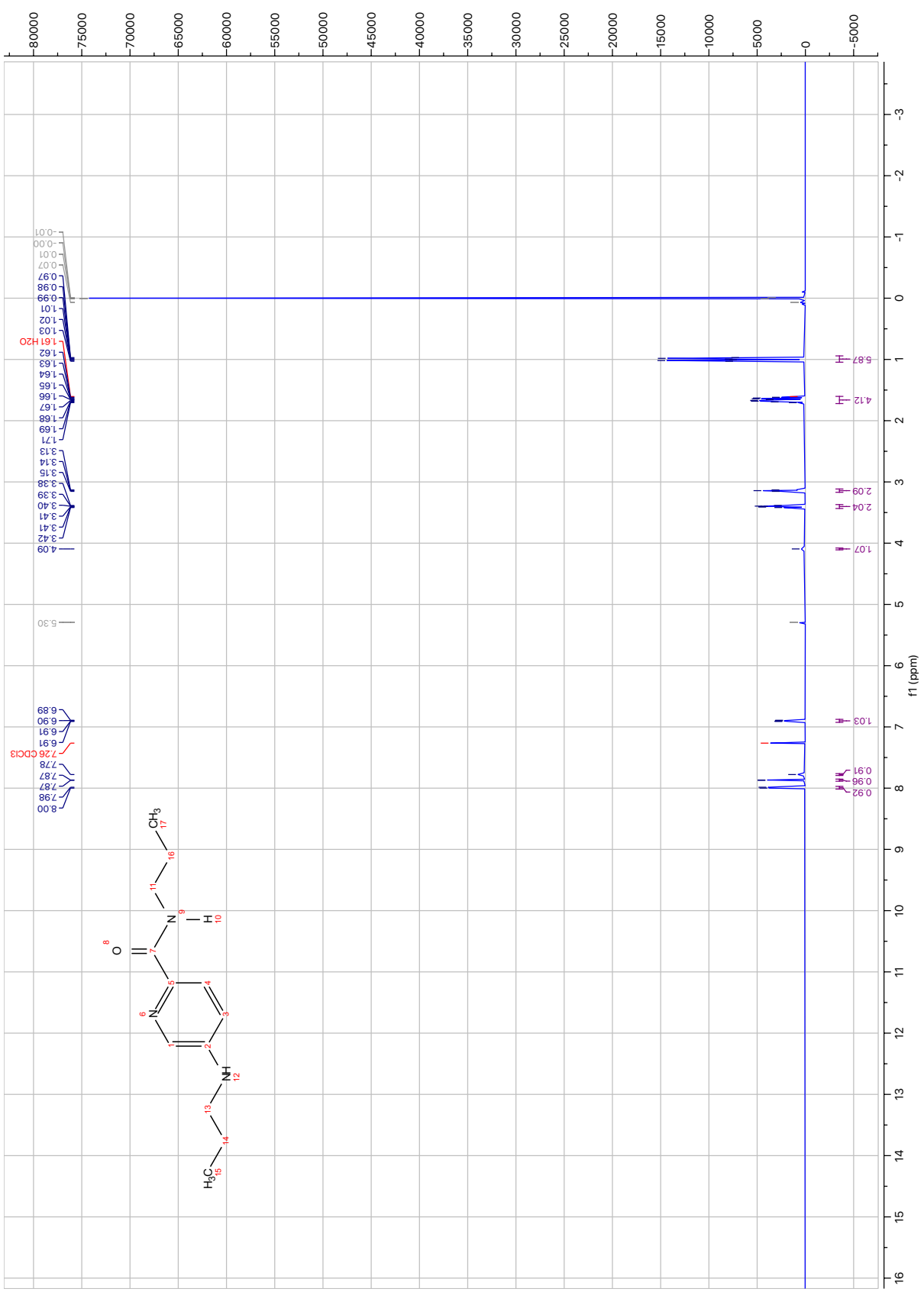

167.61
165.42
163.44
131.13
131.11
130.63
130.56
115.55
115.38
f1 (ppm)

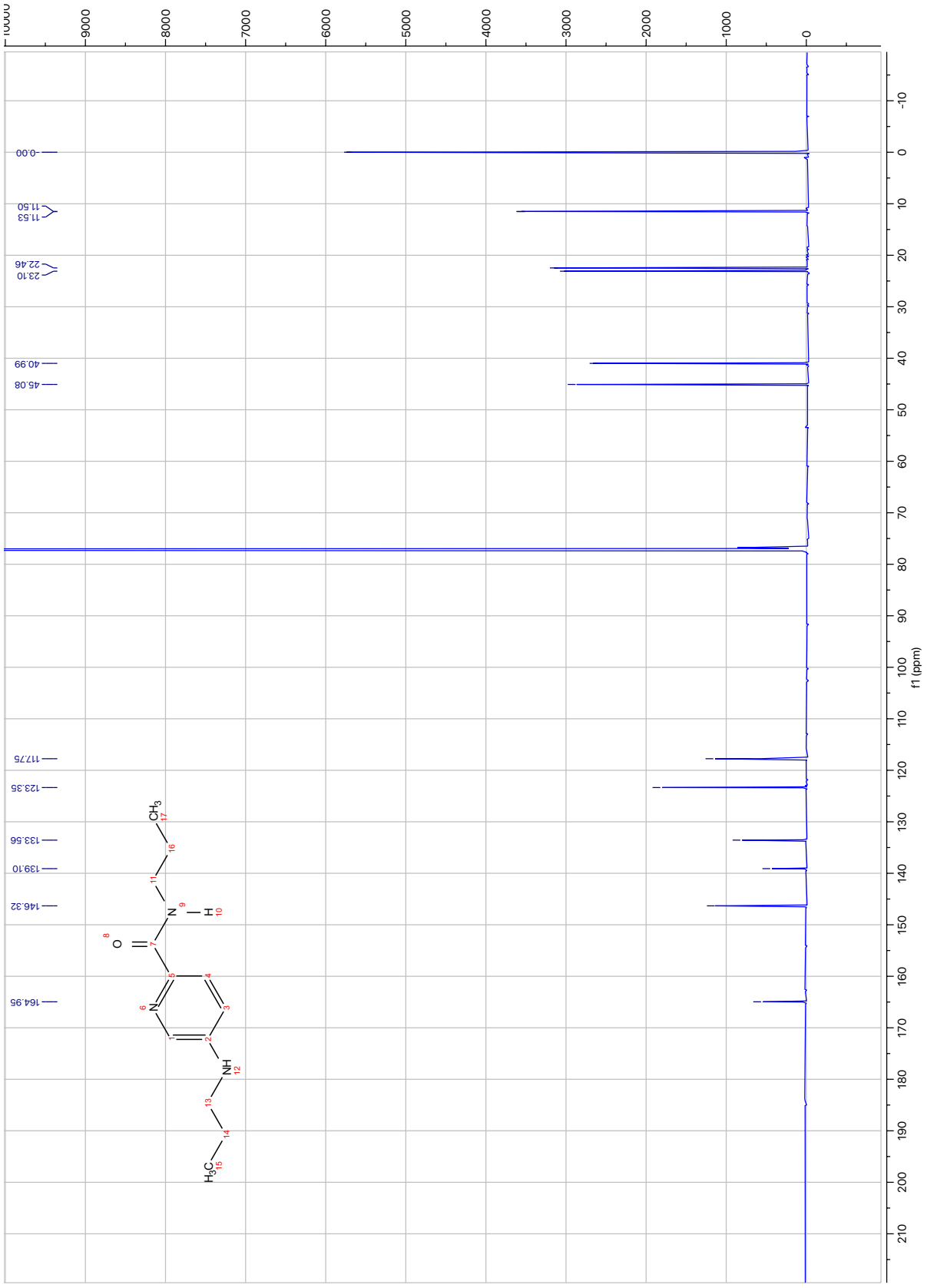

f1 (ppm)
4.87
1.62
0.93
0.91
0.91
0.93

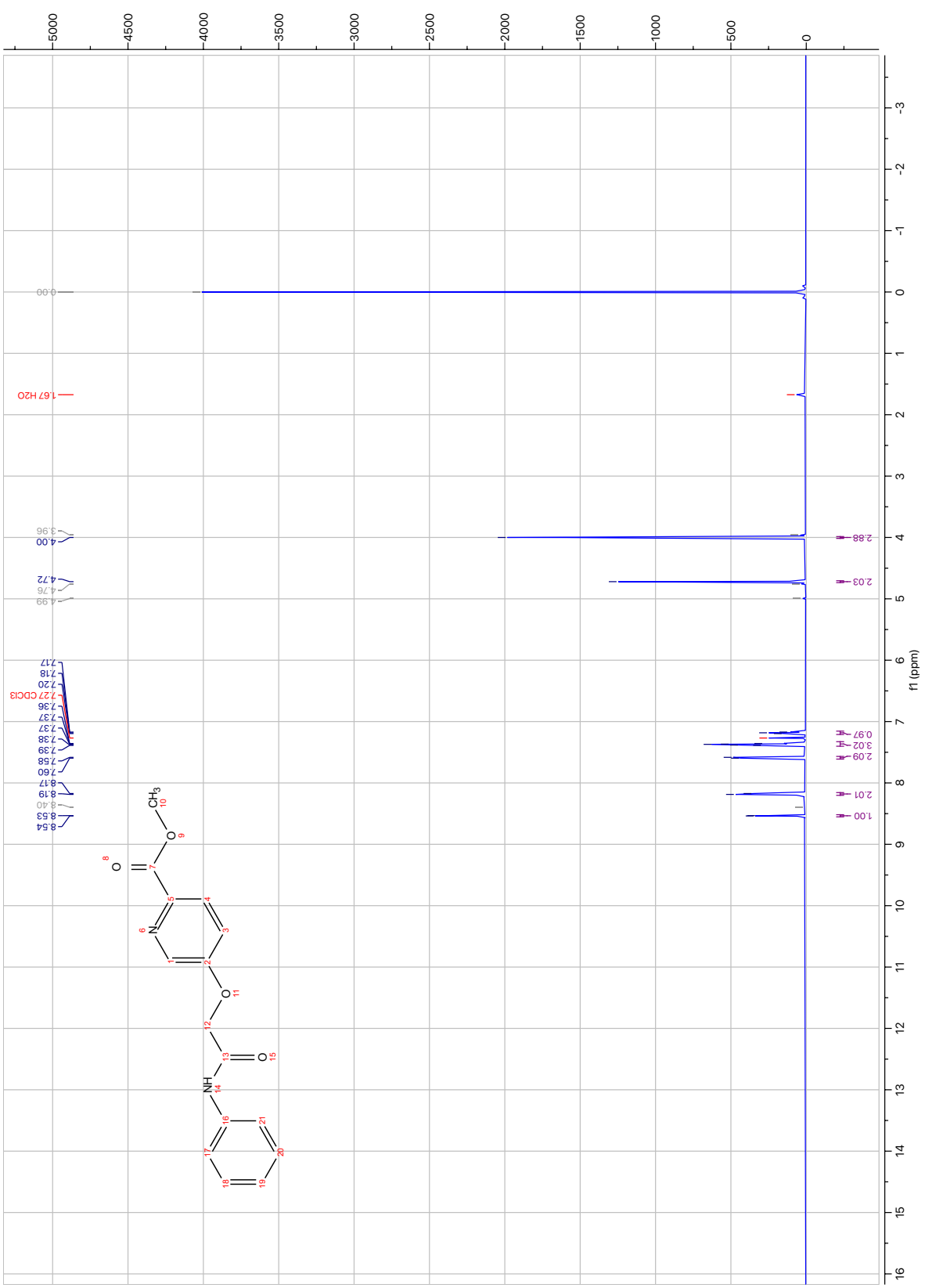

9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
f1 (ppm)
121.97
126.46
137.31
148.46
149.65
167.94
O
N
H
H
N

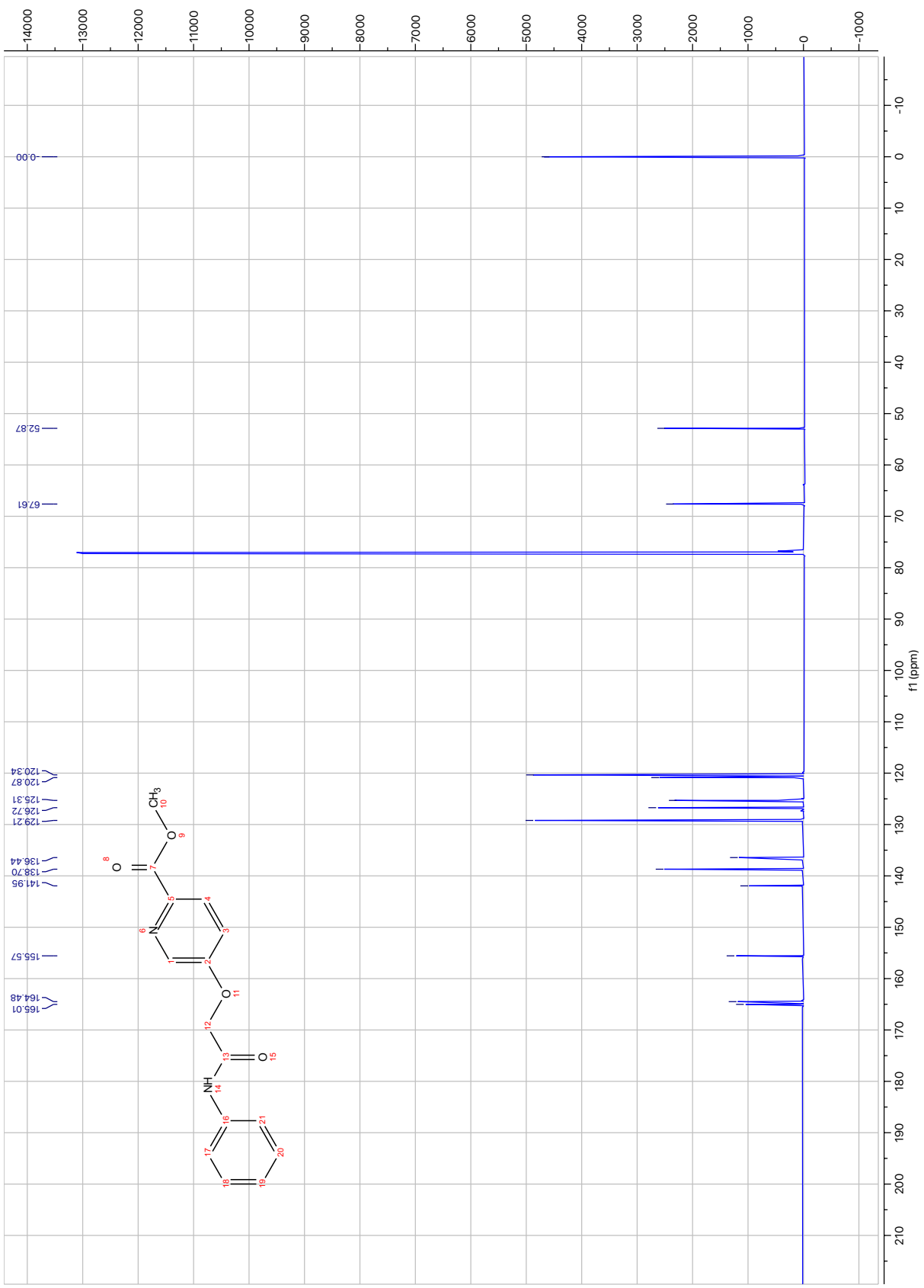

f1 (ppm)
9.10
9.10
9.10
9.09
8.68
8.68
8.67
8.67
8.29
8.25
8.25
8.24
8.23
8.23
7.74
7.46
7.45
7.44
7.44
7.44
7.44
7.43
7.43
0.97
1.03
1.01
0.97
1.02
1.01

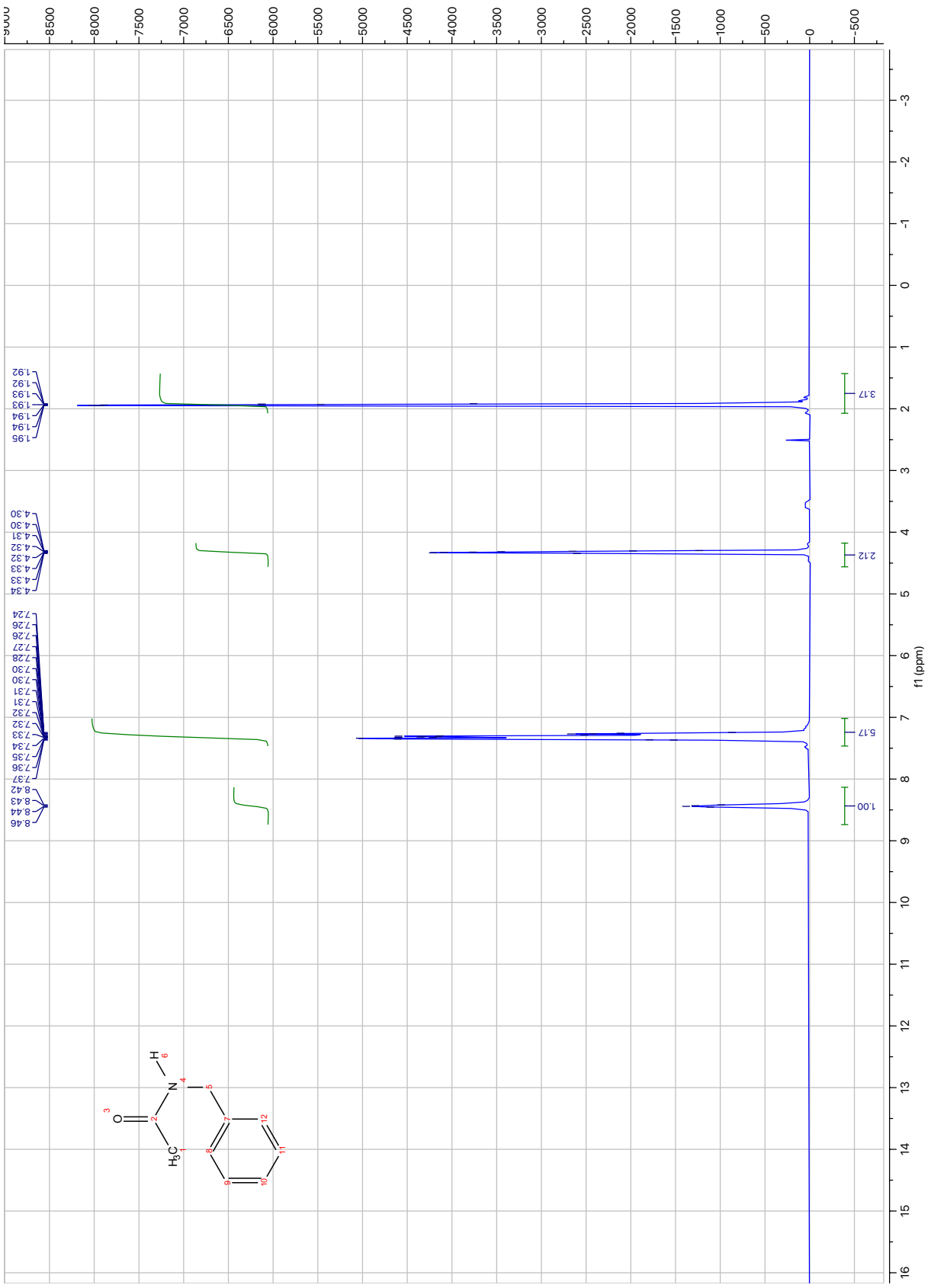

123.79
130.10
135.66
149.16
152.29
167.31
f1 (ppm)


f1 (ppm)
8.00
7.98
7.87
7.87
7.78
7.26 CDCl3
6.91
6.91
6.90
6.89
5.30
4.09
3.42
3.41
3.40
3.39
3.38
3.15
3.14
3.13
1.71
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.62
1.61 H2O
1.03
1.02
1.01
0.99
0.98
0.97
0.07
0.01
0.00
-0.01
5.87
4.12
2.09
2.04
1.07
1.03
0.92
0.96
0.91


-0.00
11.53
11.50
23.10
22.46
40.99
45.08
117.75
123.35
133.56
139.10
146.32
164.95
f1 (ppm)


f1 (ppm)
0.00
1.67 H2O
4.00
3.96
4.72
4.76
4.99
8.54
8.53
8.40
8.19
8.17
7.60
7.58
7.39
7.38
7.37
7.37
7.36
7.27 CDCl3
7.20
7.18
7.17
2.88
2.03
0.97
3.02
2.09
2.01
1.00


0.00
52.87
67.61
120.34
120.87
125.31
126.72
129.21
136.44
138.70
141.95
155.57
164.48
165.01
f1 (ppm)


f1 (ppm)
1.95
1.94
1.94
1.93
1.93
1.92
1.92
4.34
4.33
4.33
4.32
4.32
4.31
4.30
4.30
7.37
7.36
7.35
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.28
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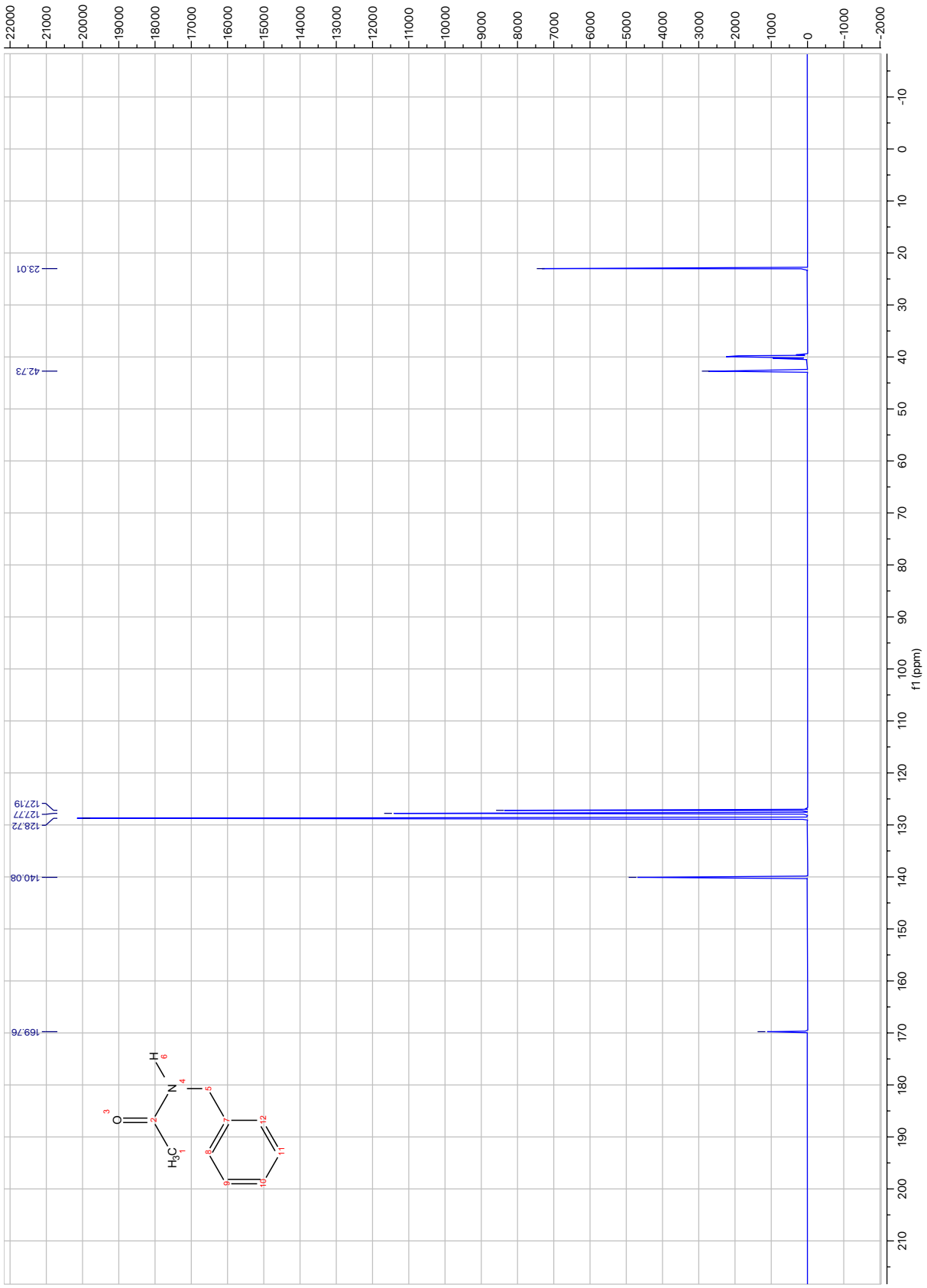
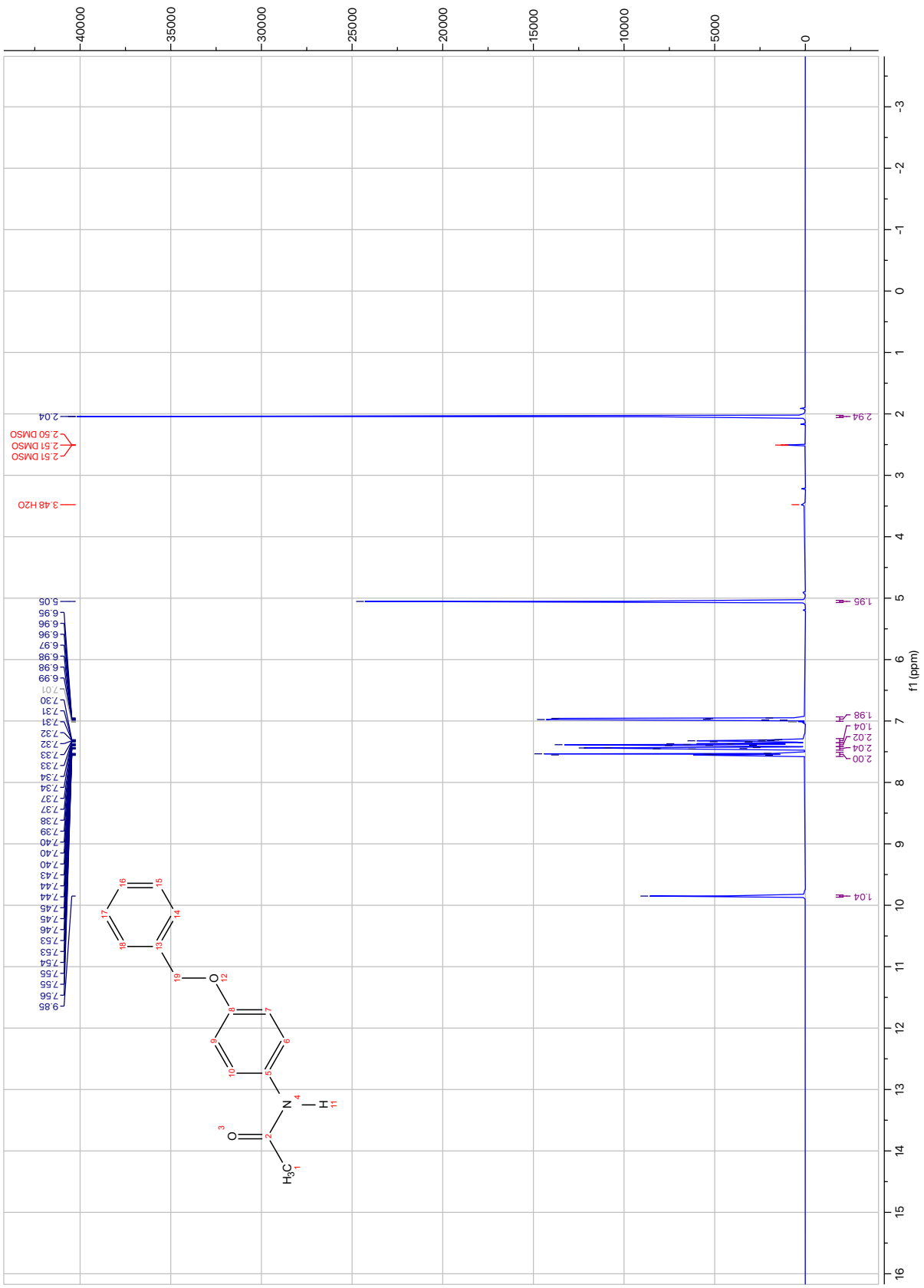
8.43
8.42
3.17
2.12
5.17
1.00


169.76
140.08
128.72
127.77
127.19
42.73
23.01
f1 (ppm)


f1 (ppm)
2.94
1.95
1.98
1.04
2.00
2.04
2.02
1.04
1.04
2.04
2.50 DMSO
2.51 DMSO
2.51 DMSO
3.48 H2O
5.05
6.95
6.96
6.96
6.97
6.98
6.98
6.99
7.01
7.30
7.31
7.31
7.32
7.32
7.33
7.33
7.34
7.34
7.37
7.37
7.38
7.39
7.40
7.40
7.40
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7.45
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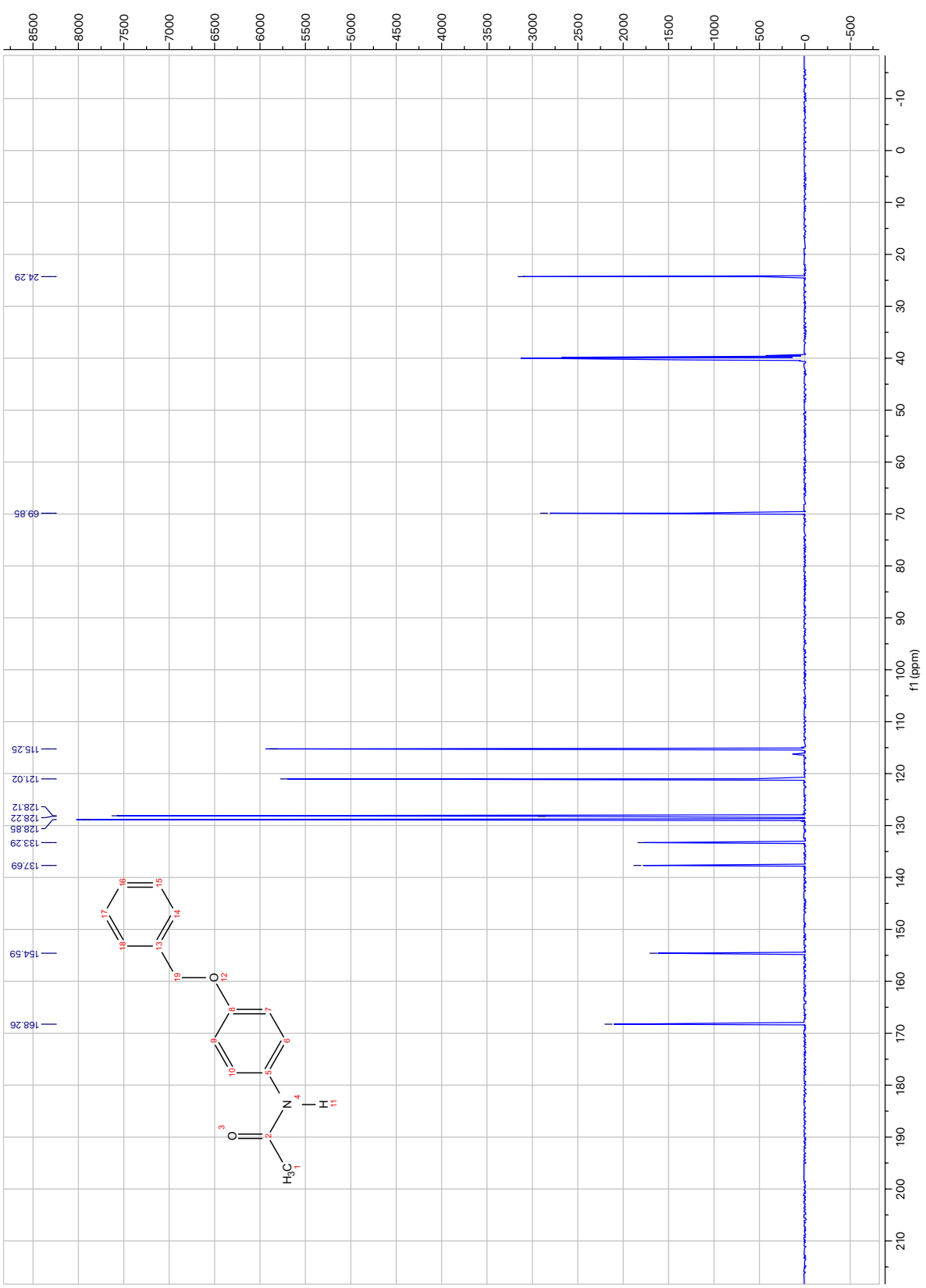
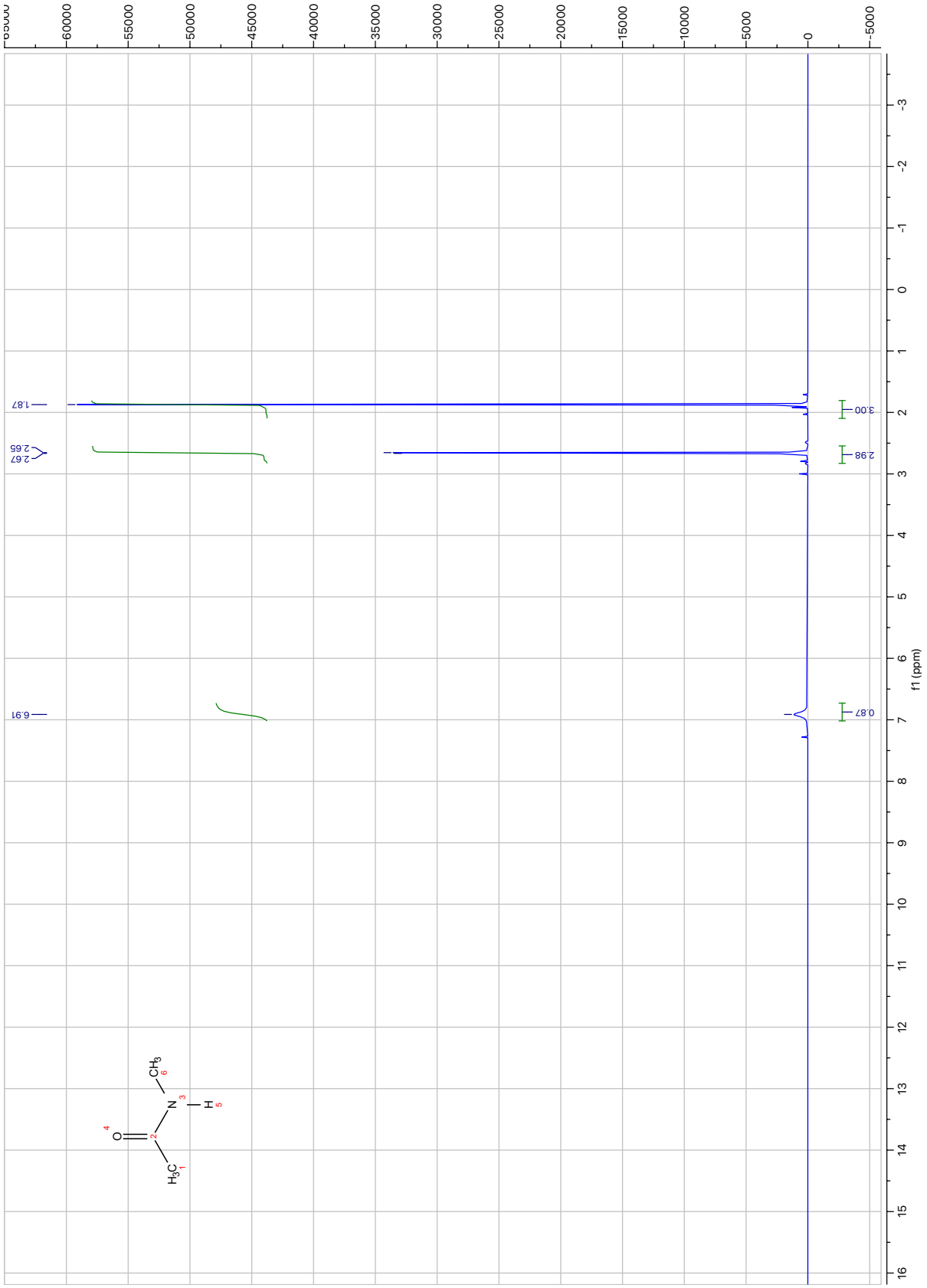
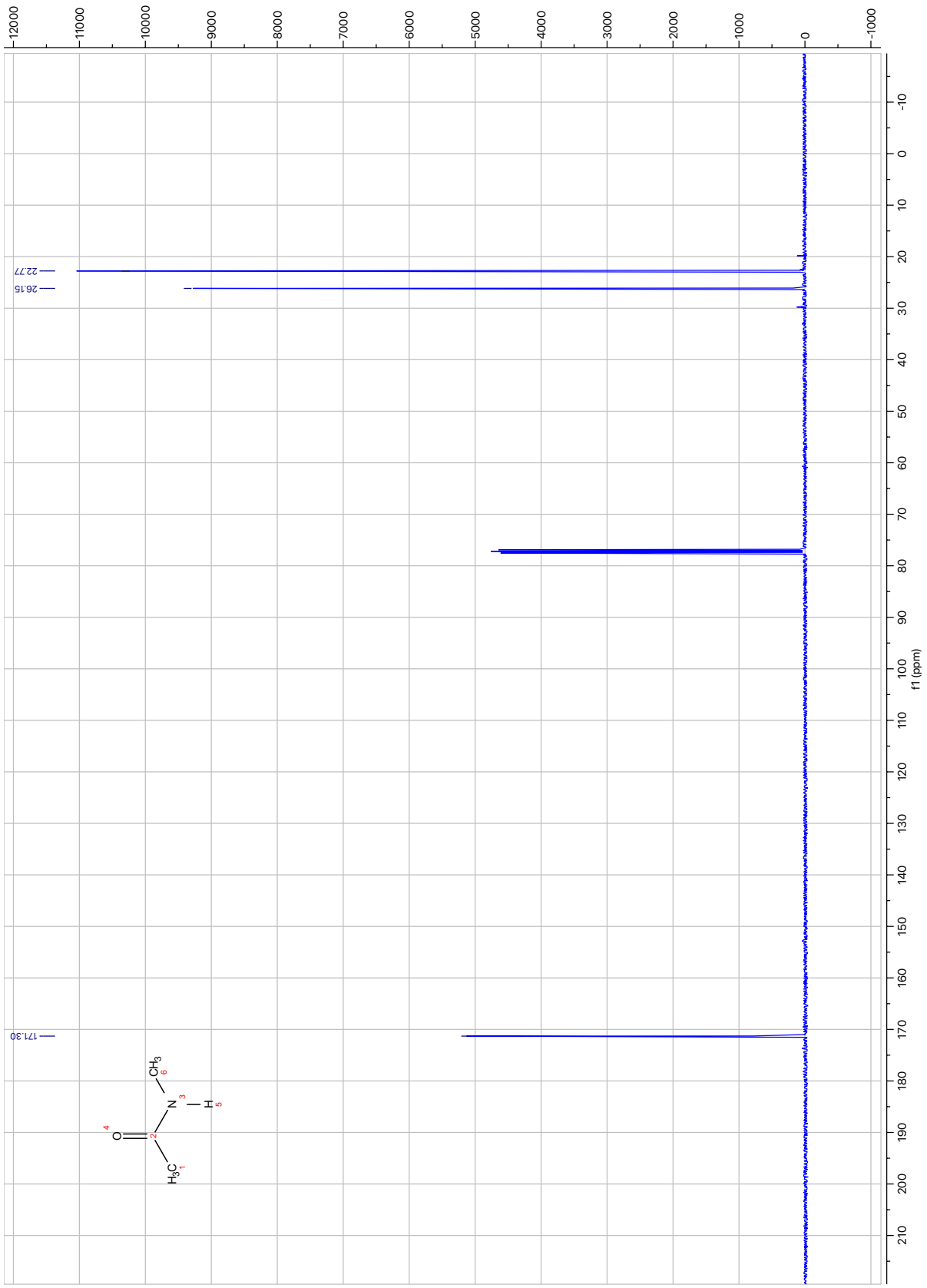
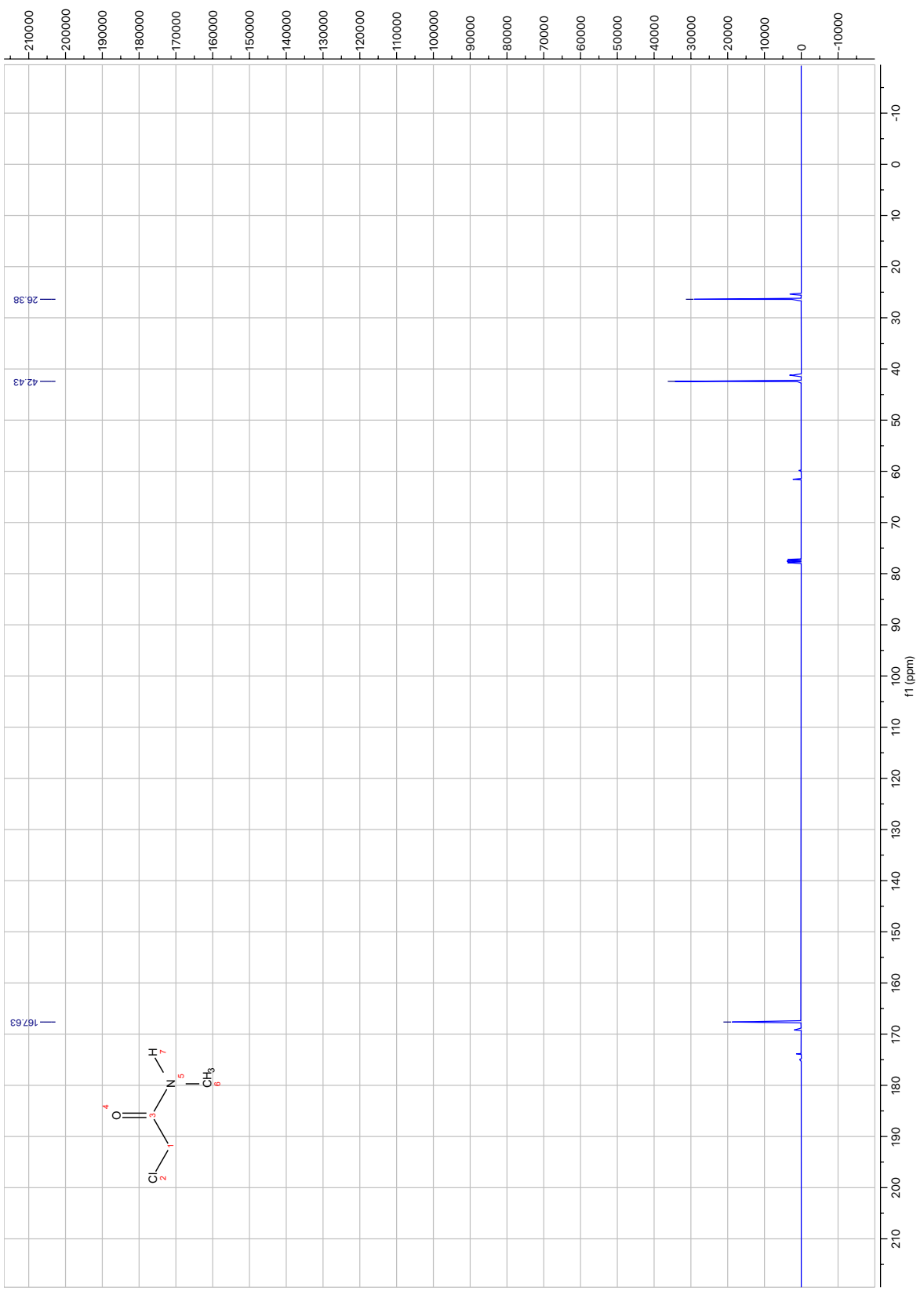
7.46
7.53
7.54
7.55
7.55
7.56
9.85

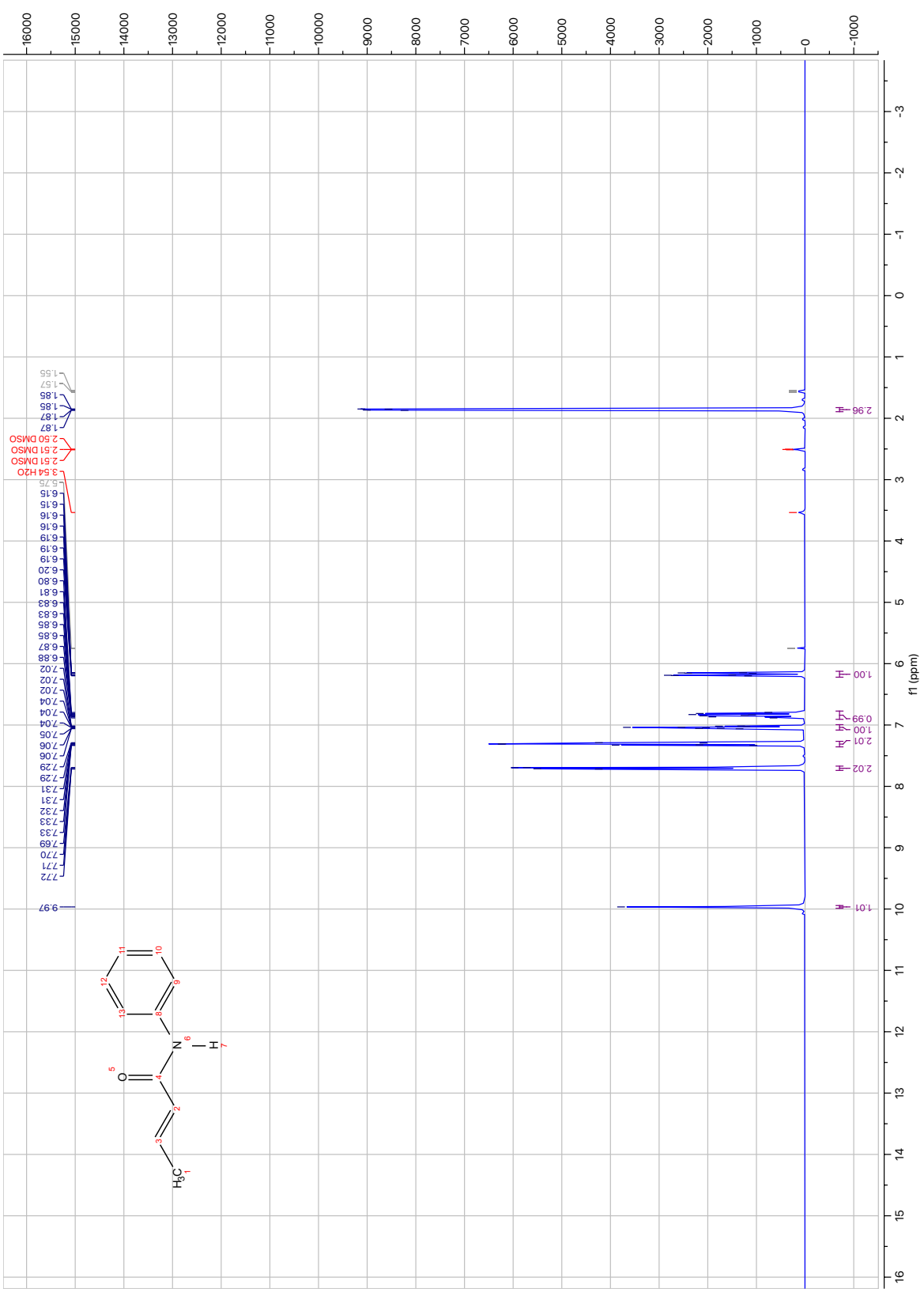

24.29
69.85
115.25
121.02
128.12
128.22
128.85
133.29
137.69
154.59
168.26
f1 (ppm)

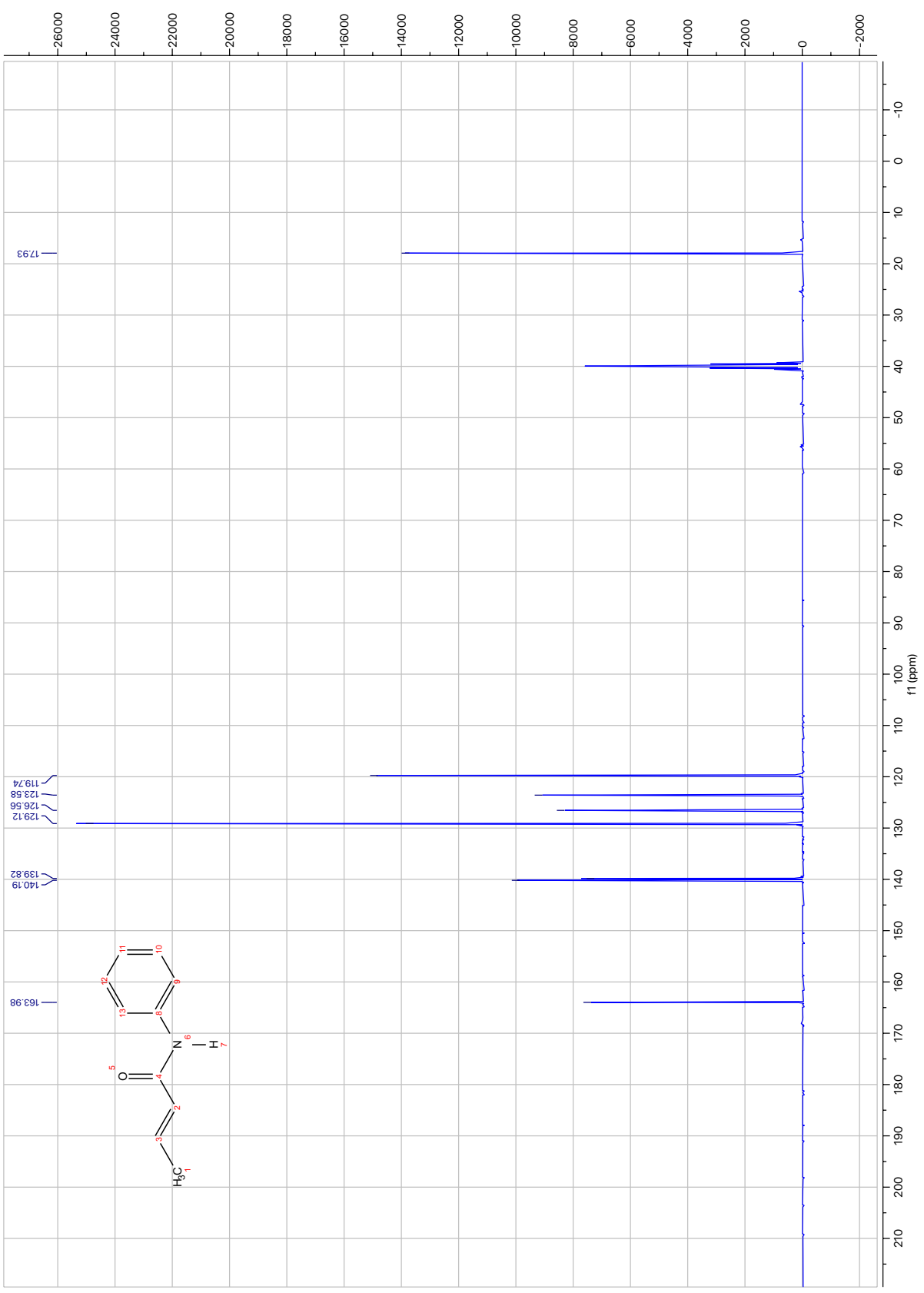

1.87
2.67
2.65
6.91
3.00
2.98
0.87
f1 (ppm)

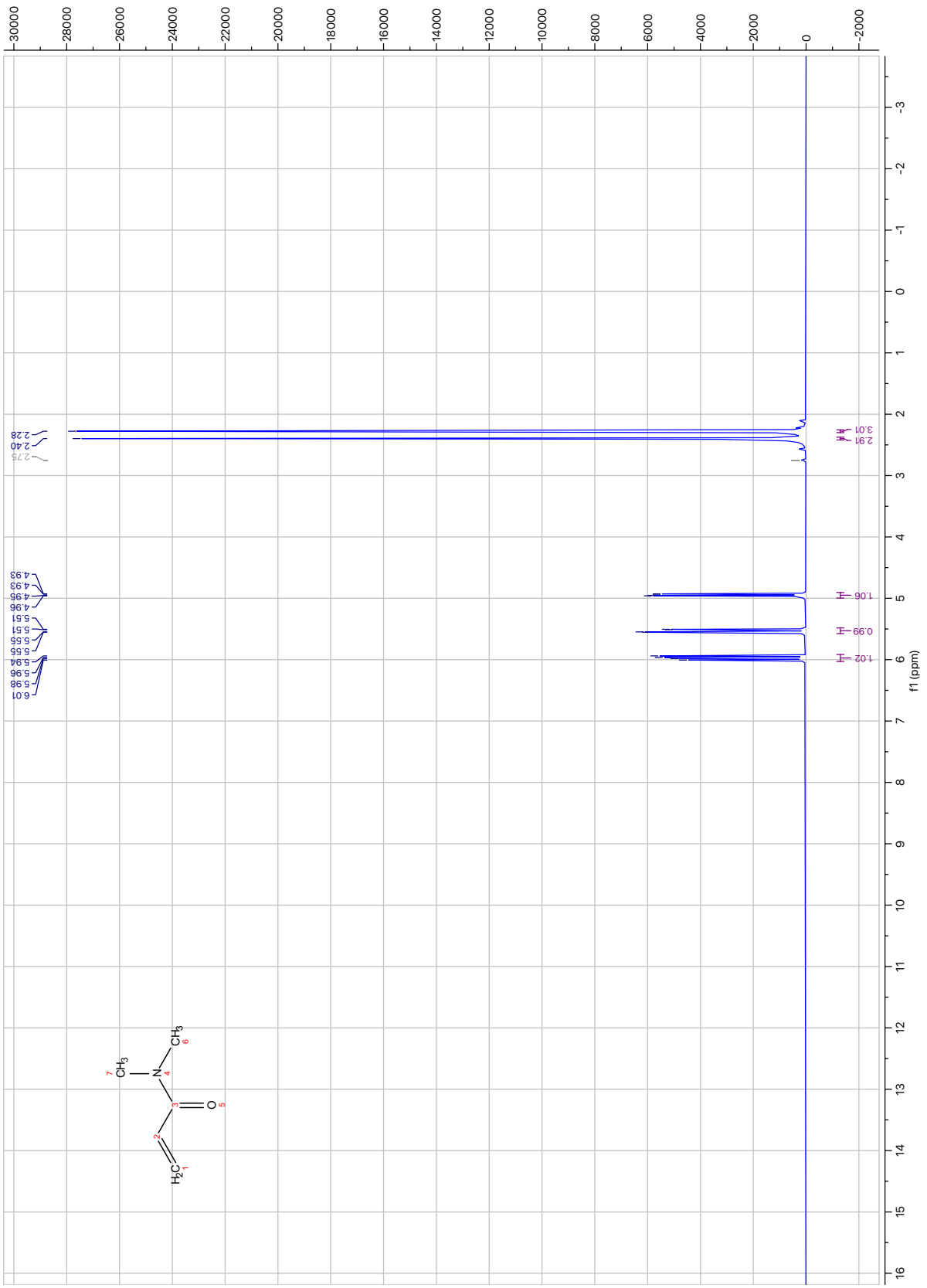

171.30
26.15
22.77
f1 (ppm)


100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
2.57
2.55
3.82
3.00
2.14
f1 (ppm)
-3
-2
-1
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
Cl
O
N
H
CH3


26.38
42.43
167.63
f1 (ppm)


f1 (ppm)
2.96
1.00
0.99
1.00
2.01
2.02
1.01
9.97
2.50 DMSO
2.51 DMSO
3.54 H2O
1.87
1.85
1.85
1.87
1.57
1.55
H3C
O
N
H


17.93
119.74
123.58
126.56
129.12
139.82
140.19
163.98
f1 (ppm)


f1 (ppm)
2.91
3.01
1.06
0.99
1.02
2.28
2.40
2.75
4.93
4.93
4.95
4.96
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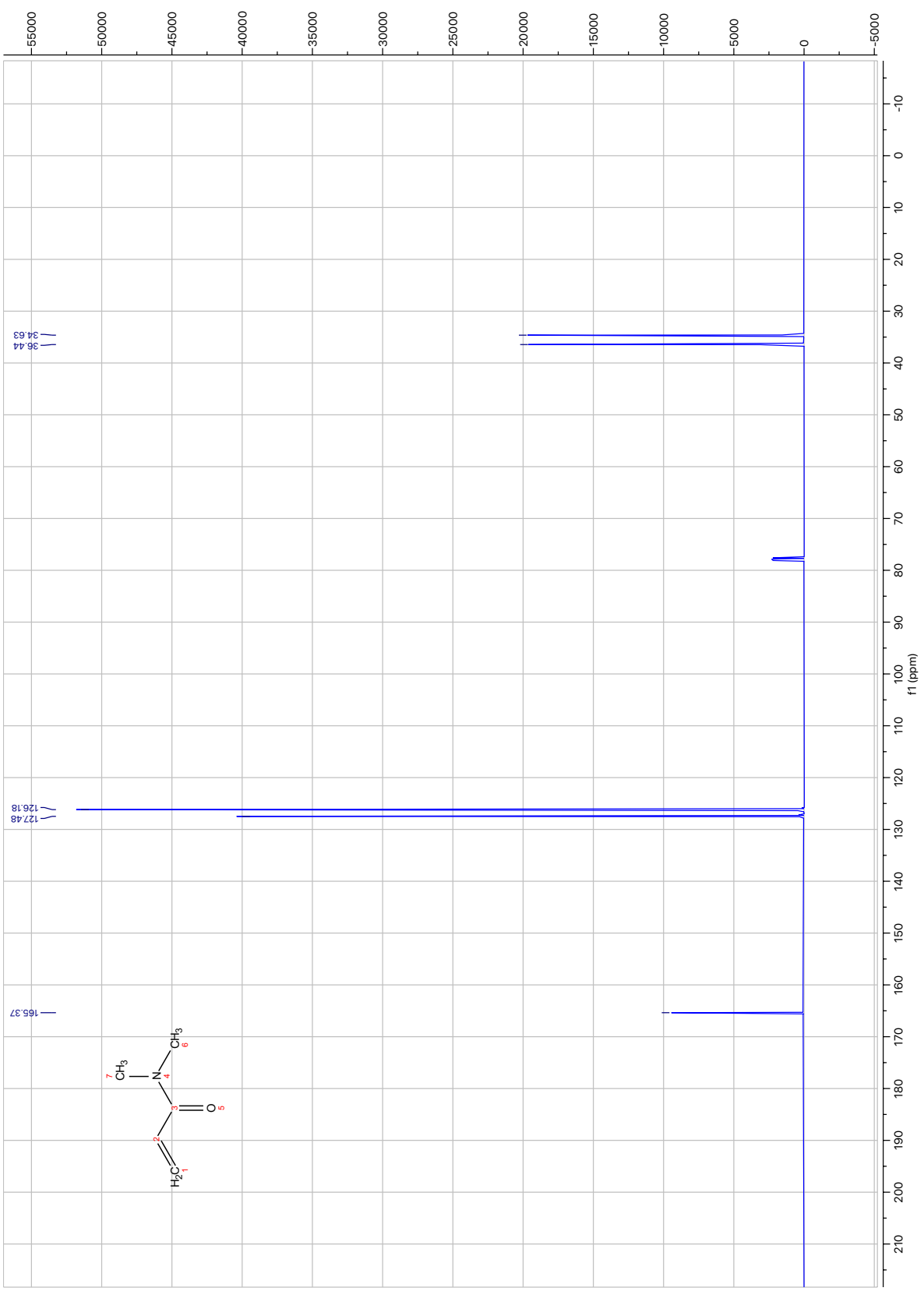
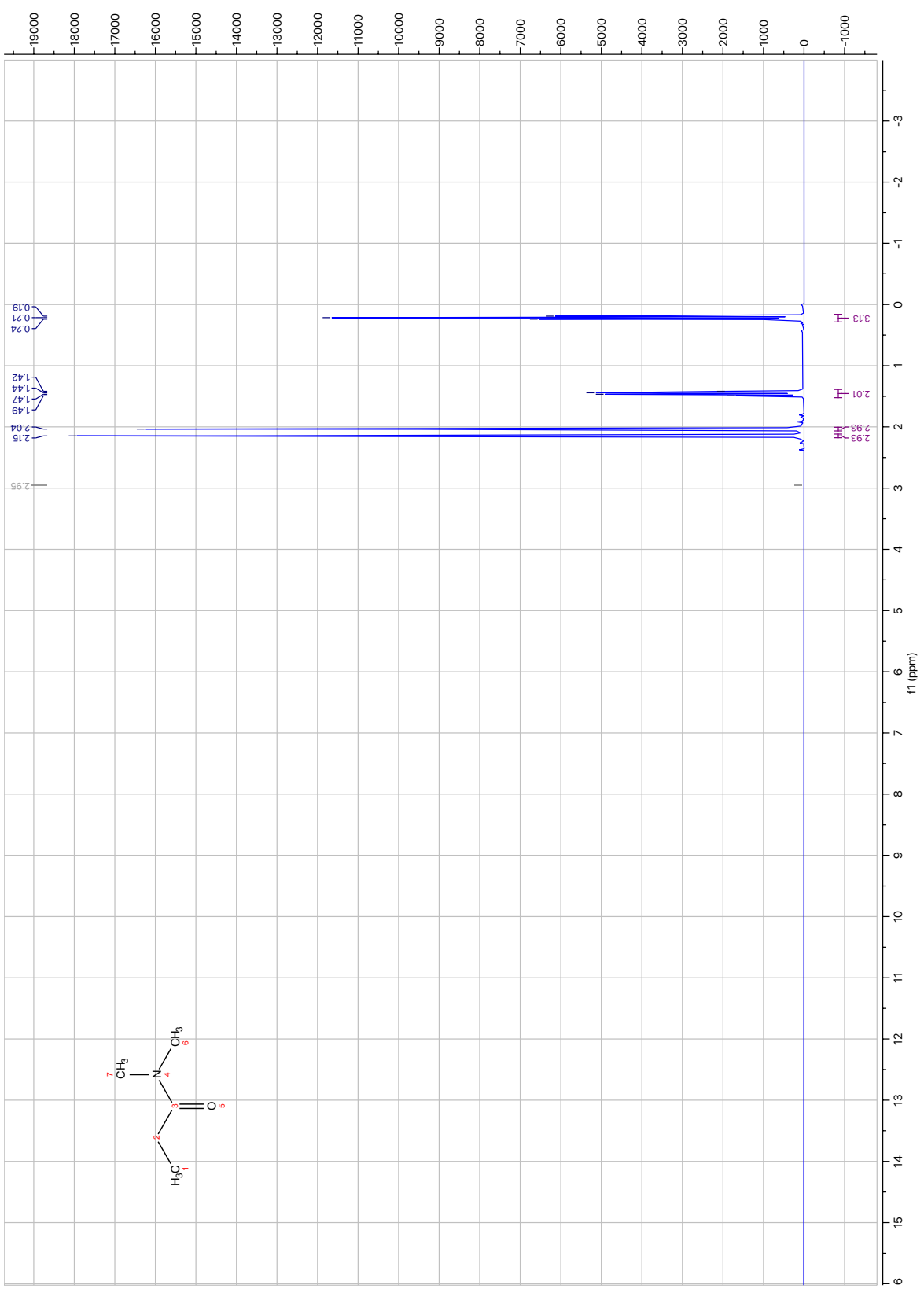
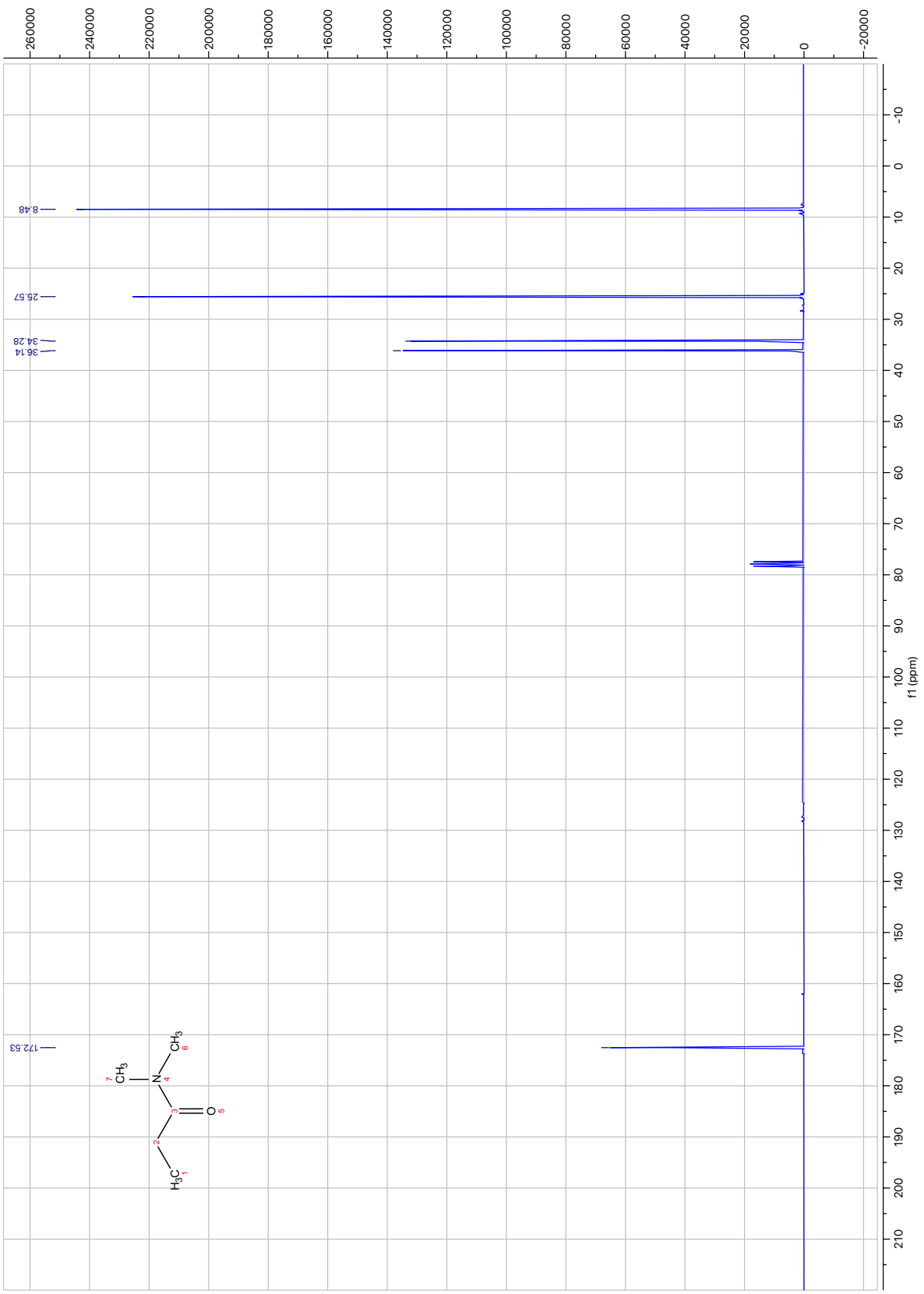
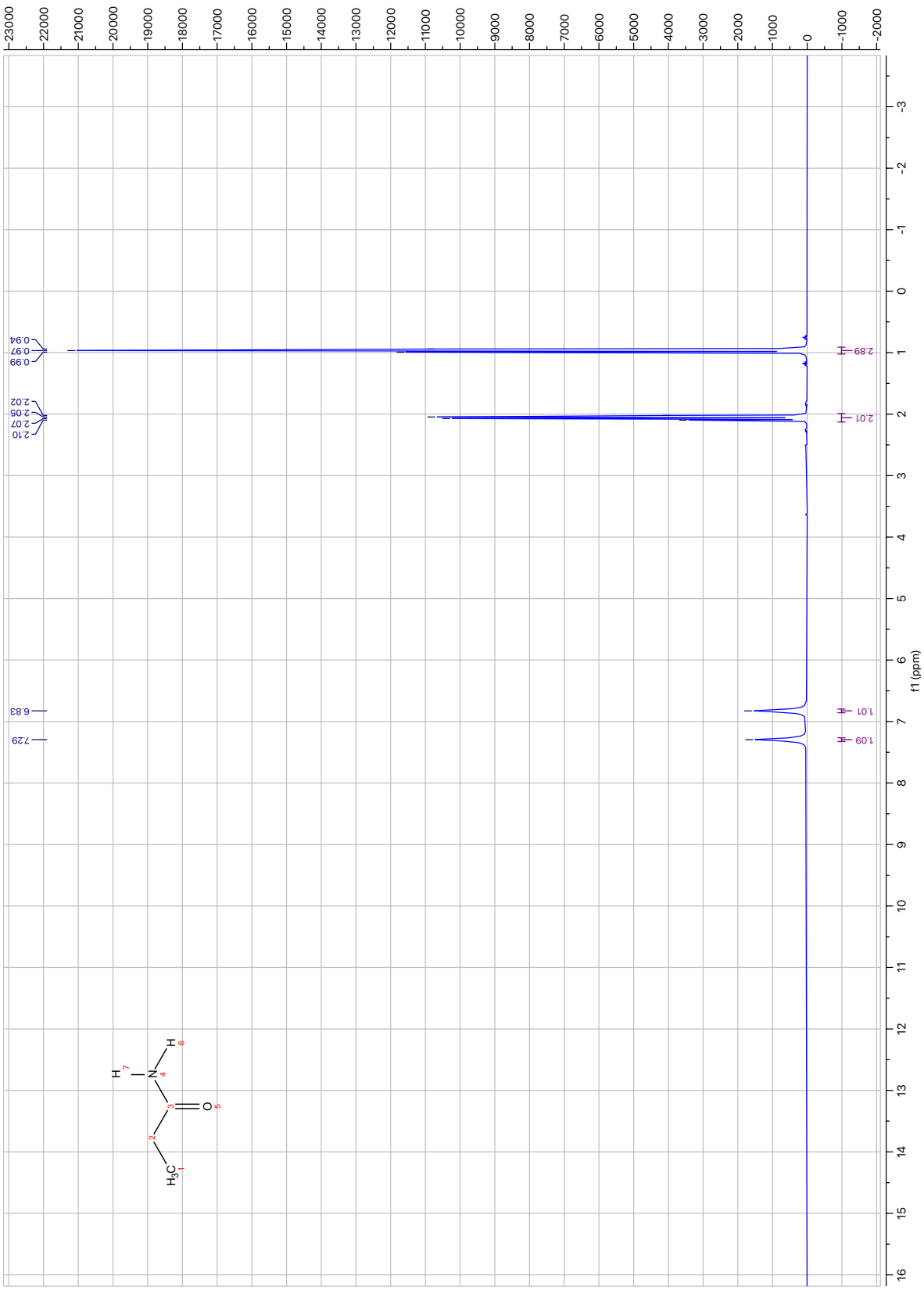
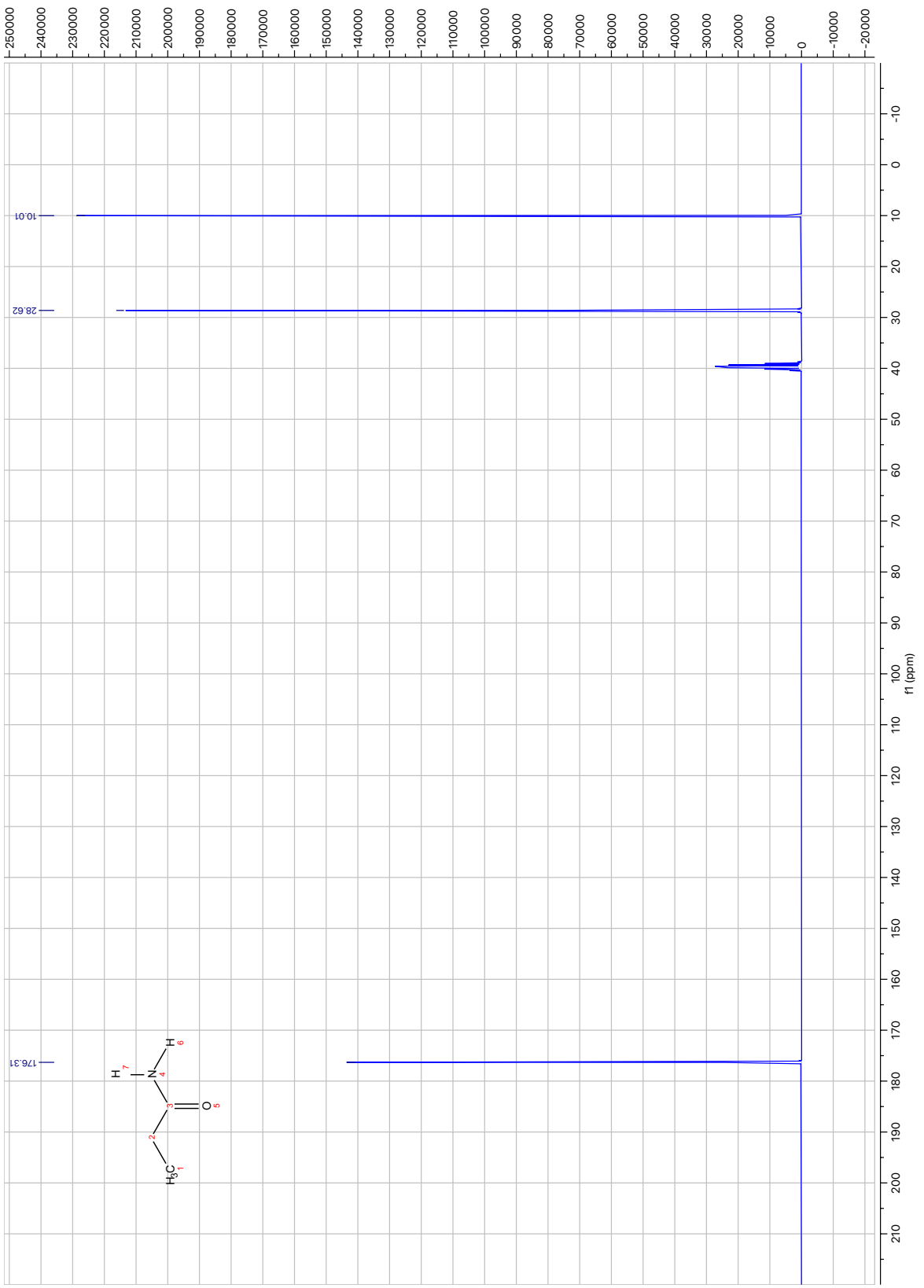
5.51
5.55
5.55
5.94
5.96
5.98
6.01

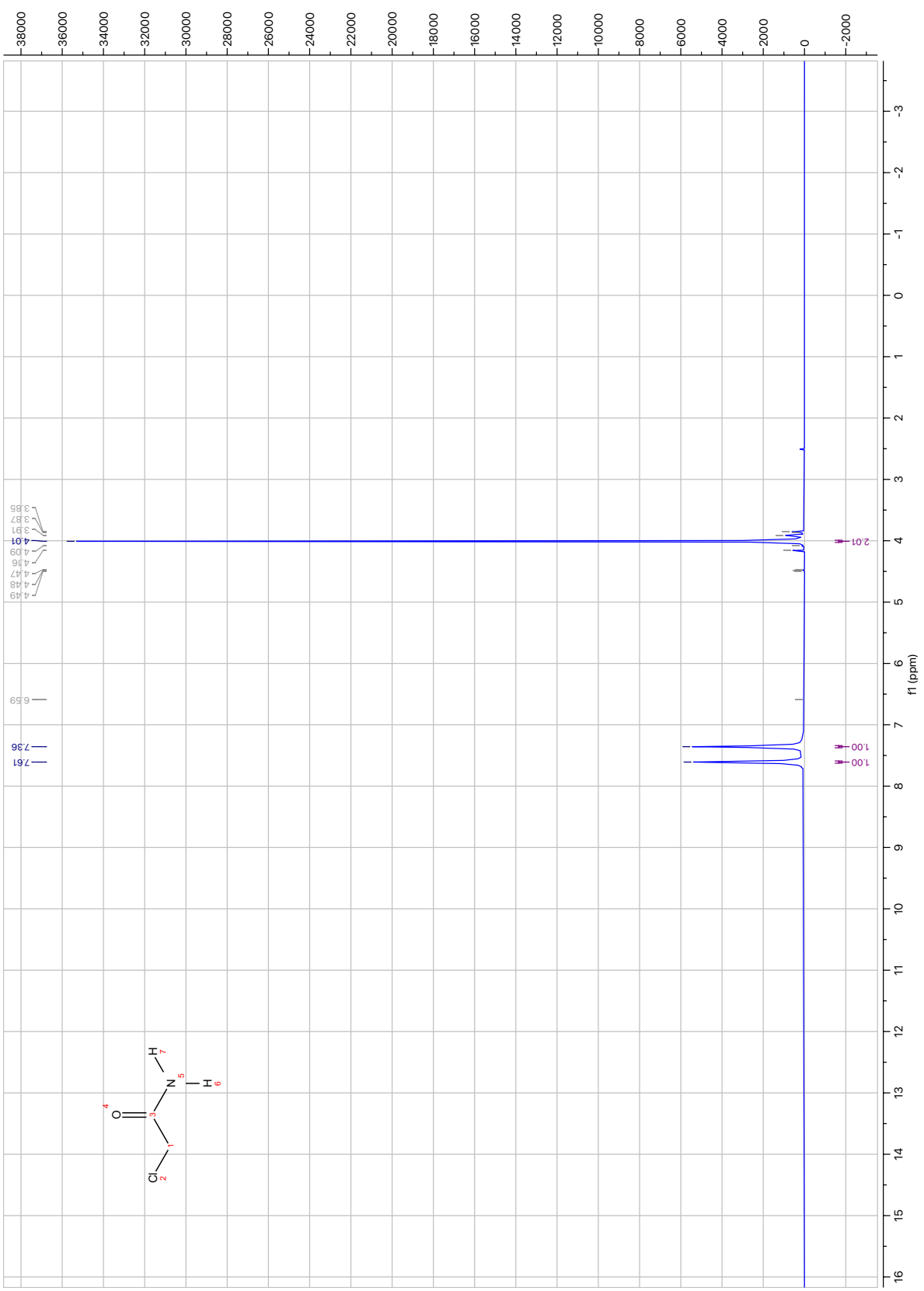

165.37
126.18
127.48
36.44
34.63
f1 (ppm)

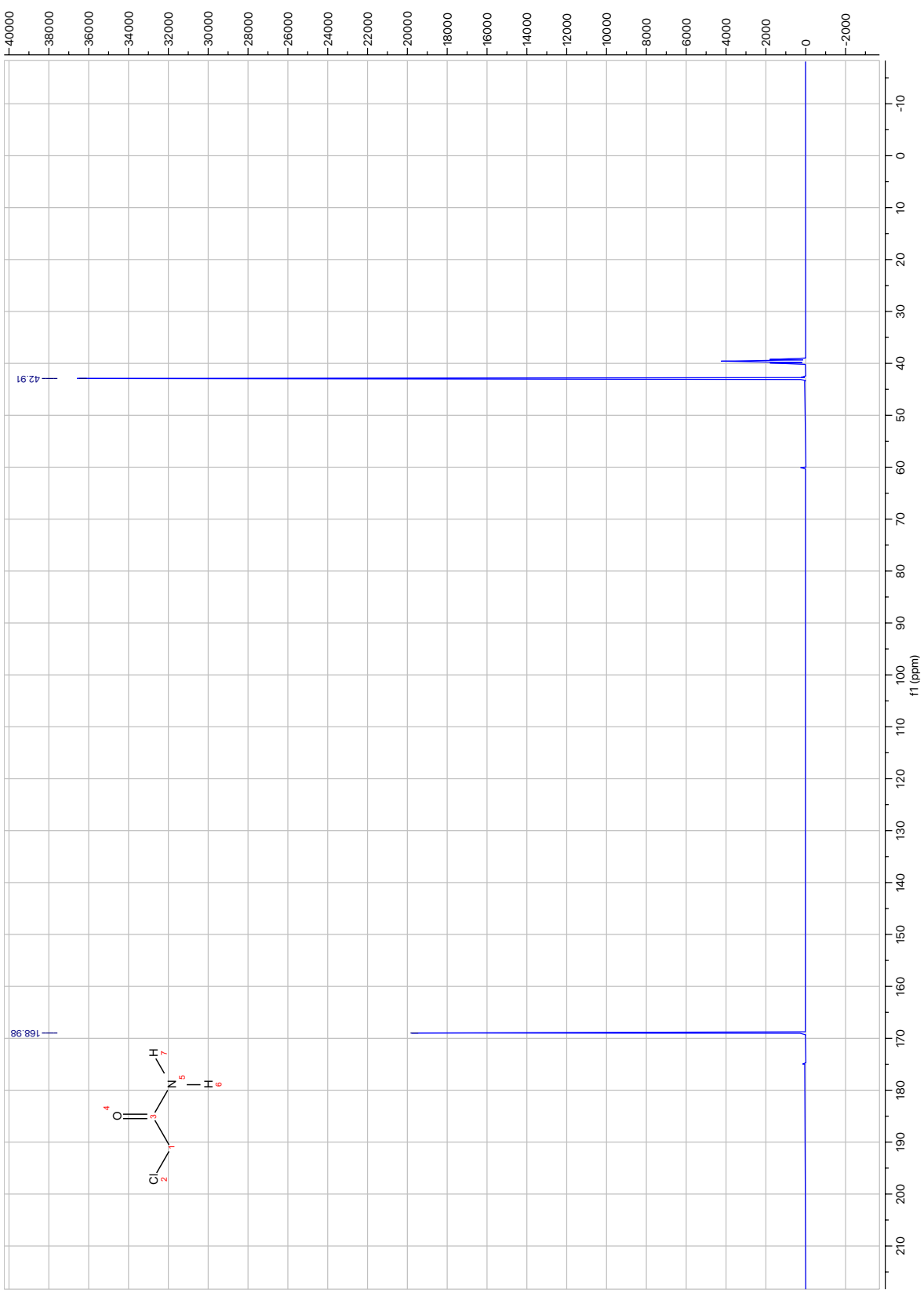

f1 (ppm)
0.19
0.21
0.24
1.42
1.44
1.47
1.49
2.04
2.15
2.95
2.93
2.01
3.13
CH3
CH3
N
O
H3C

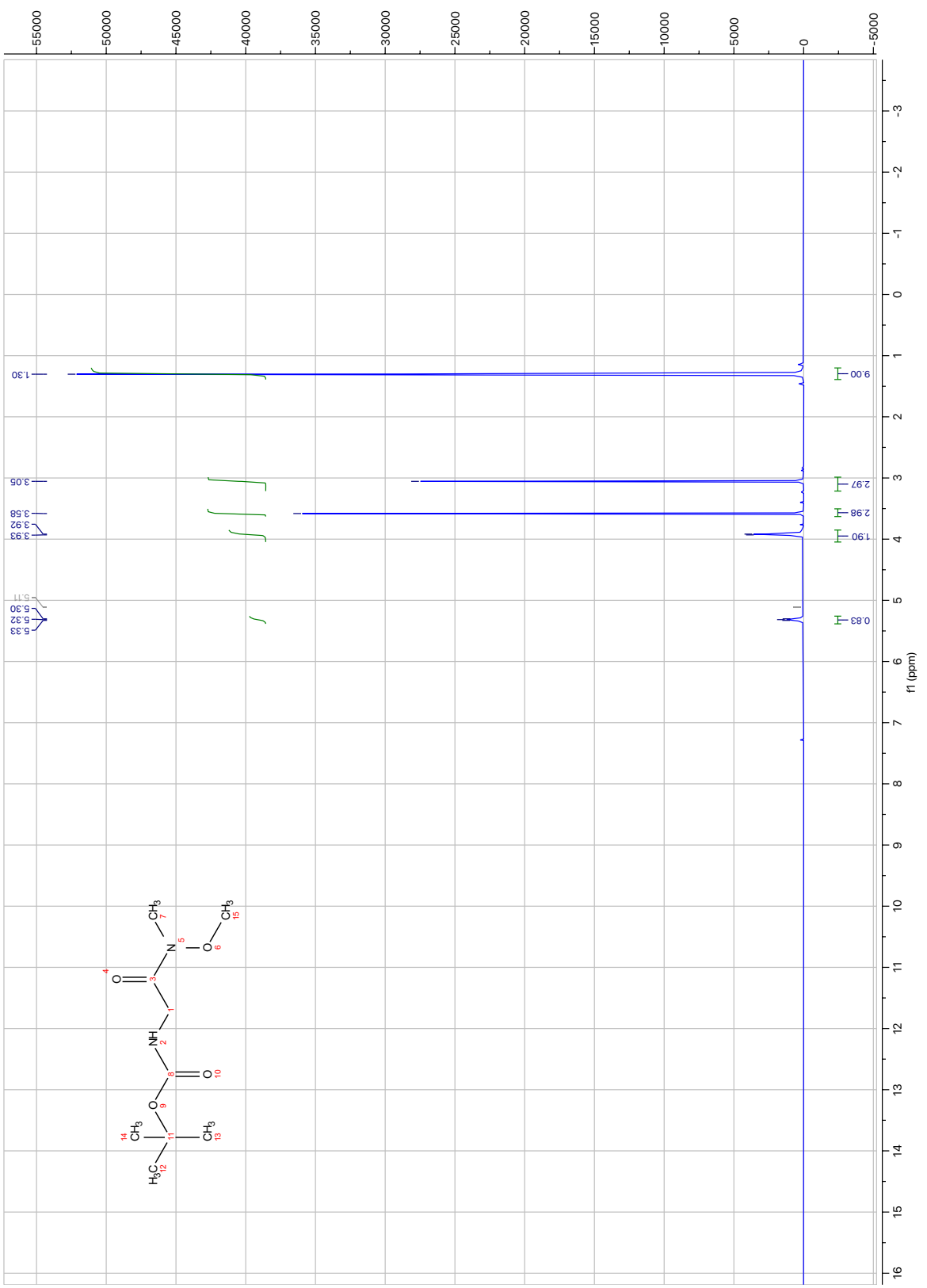

172.53
8.48
25.57
36.14
34.28
f1 (ppm)

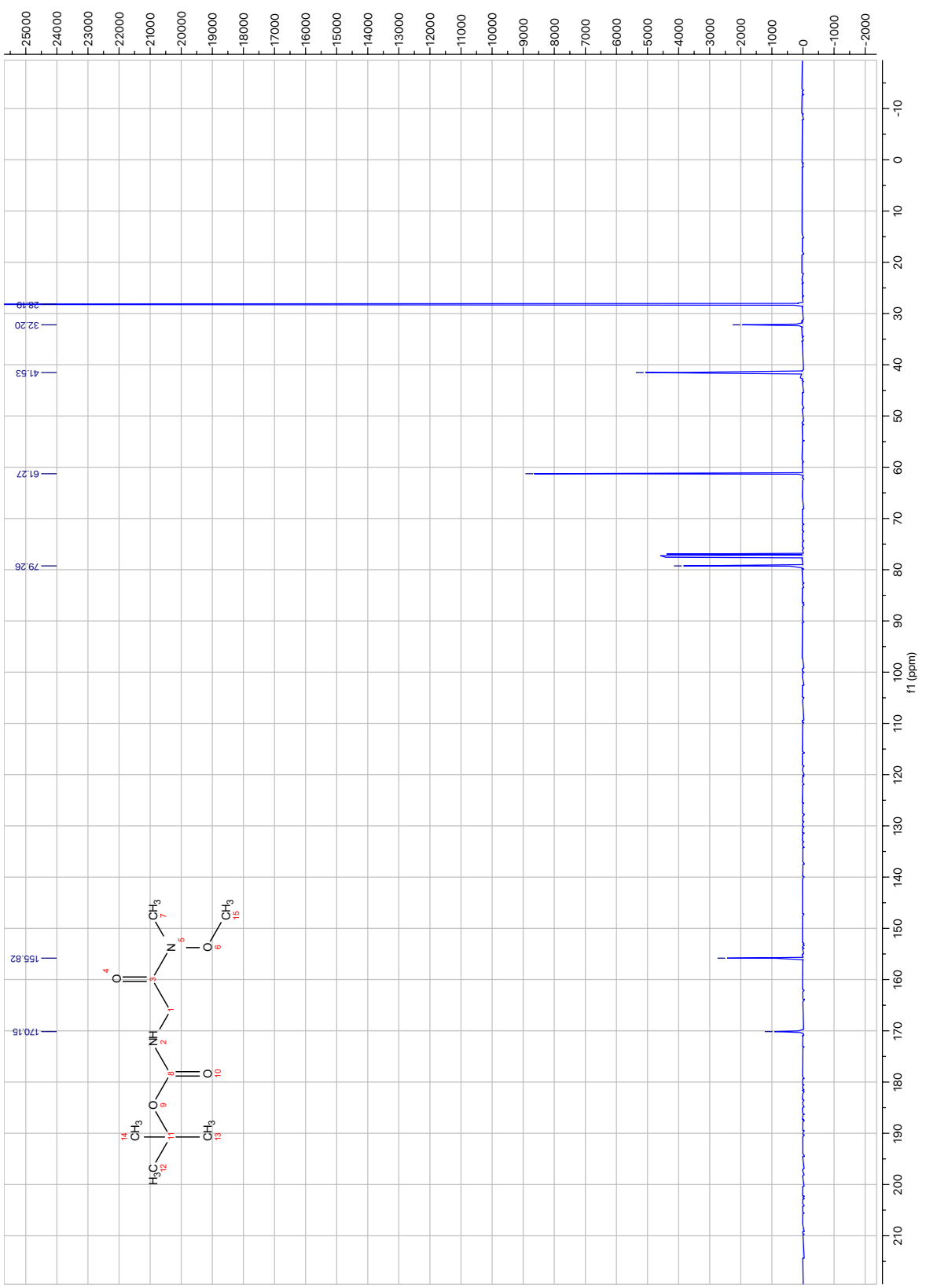

f1 (ppm)
0.94
0.97
0.99
2.02
2.05
2.07
2.10
6.83
7.29
2.89
2.01
1.01
1.09

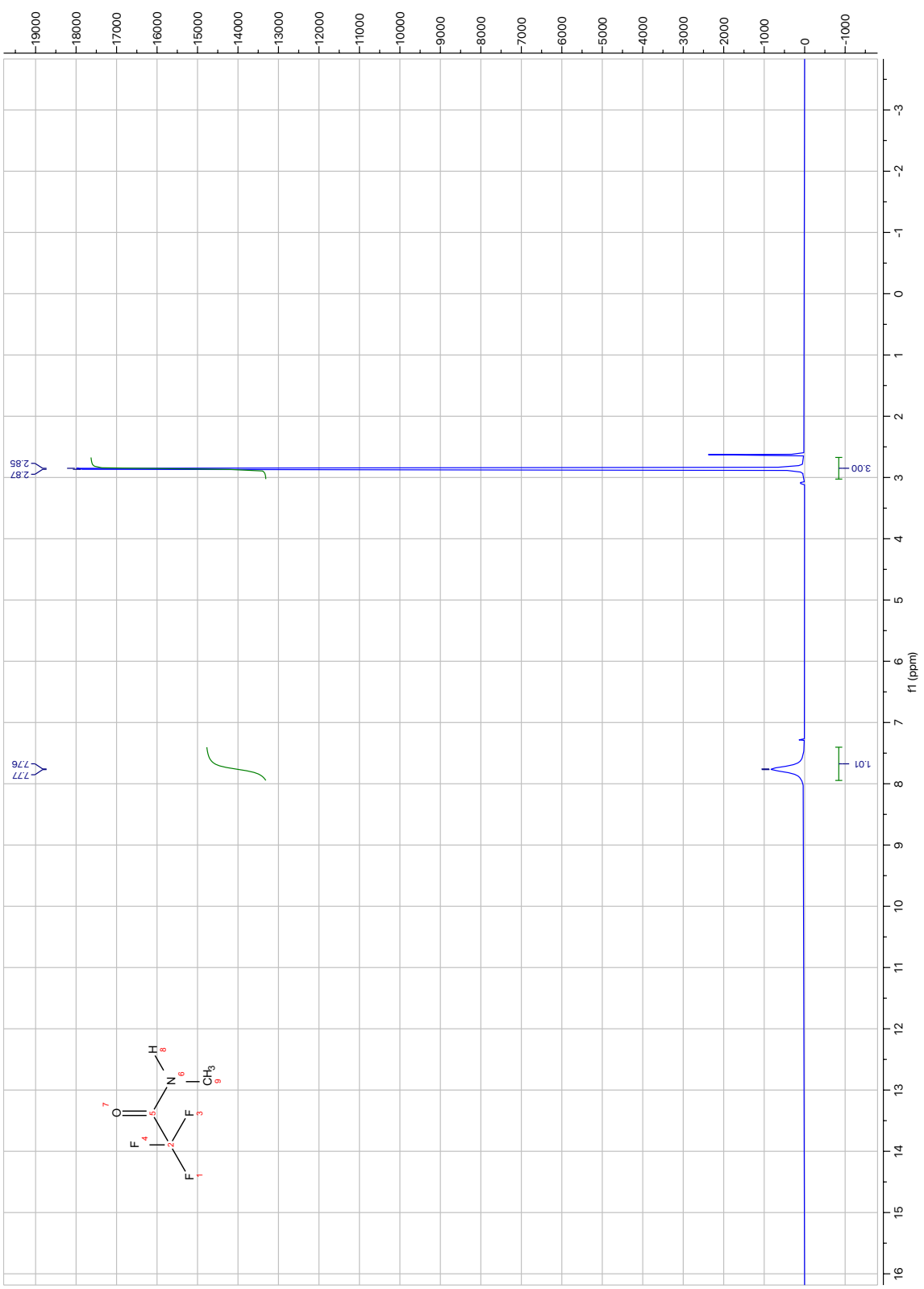

10.01
28.62
176.31
f1 (ppm)


38000
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
3.85
3.87
3.91
4.01
4.09
4.16
4.47
4.48
4.49
6.59
7.36
7.61
2.01
1.00
1.00
f1 (ppm)
-3
-2
-1
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
Cl
O
N
H
H

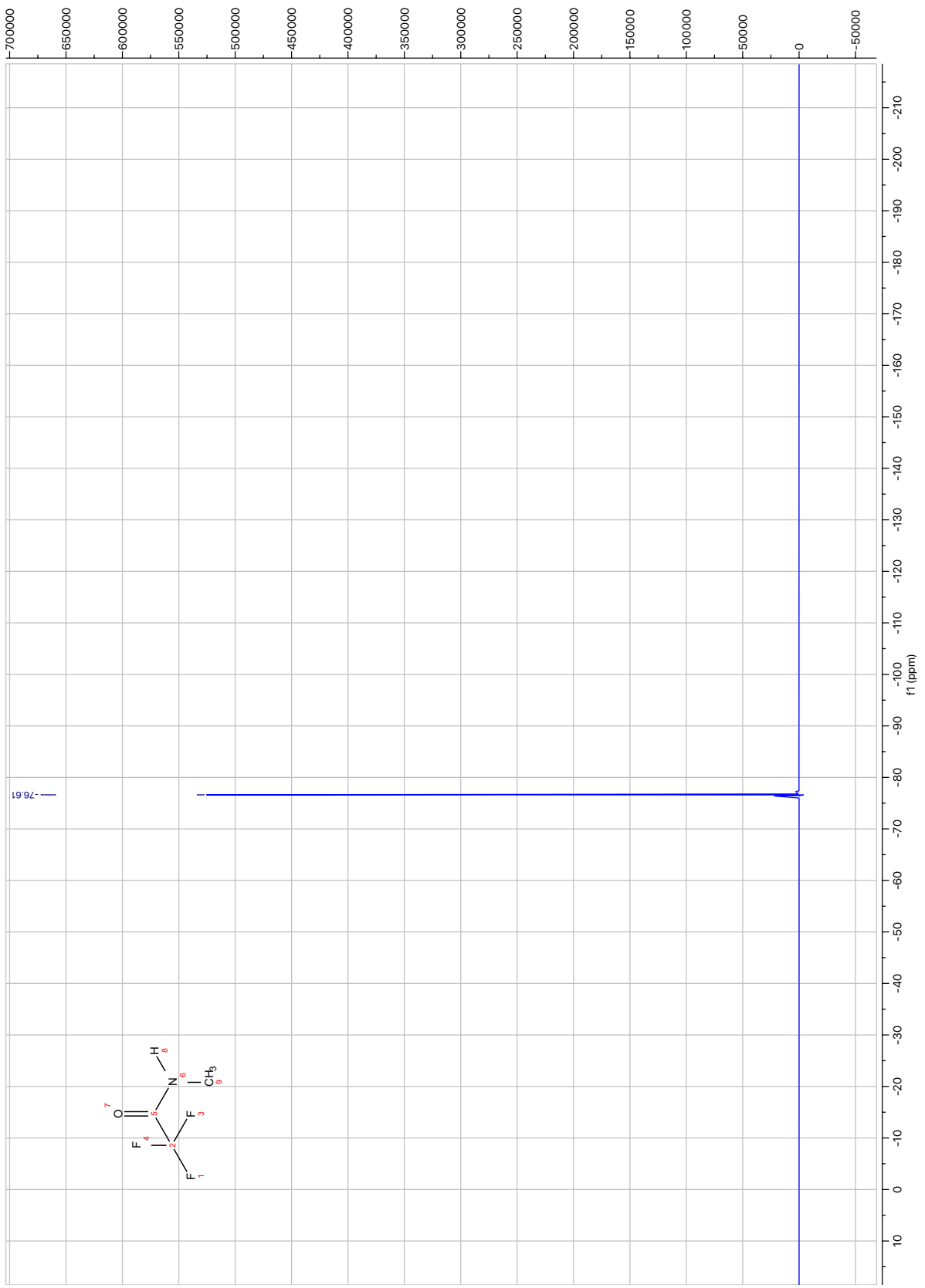

42.97
168.98
f1 (ppm)

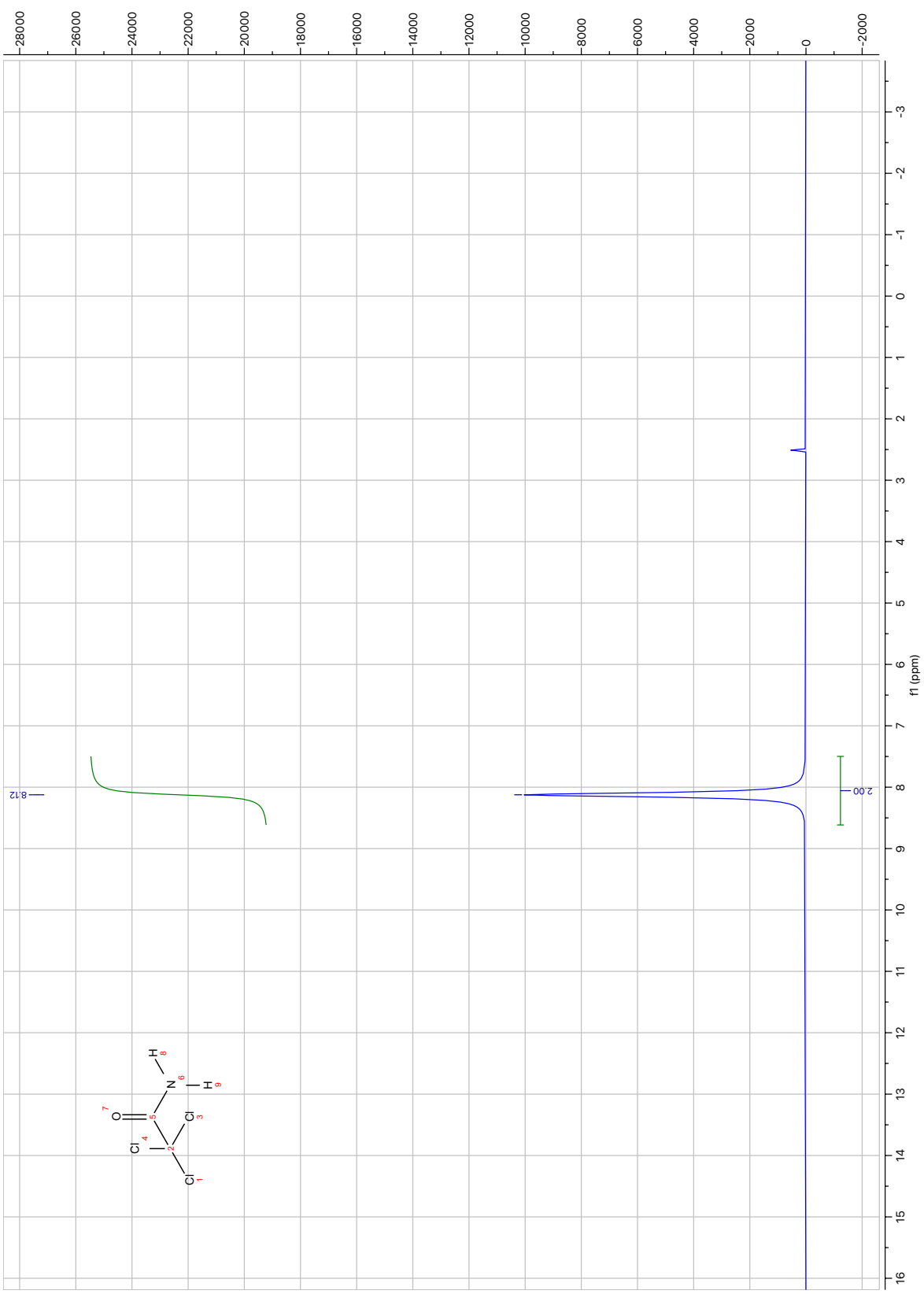

1.30
3.05
3.58
3.92
3.93
5.11
5.30
5.32
5.33
9.00
2.97
2.98
1.90
0.83
f1 (ppm)

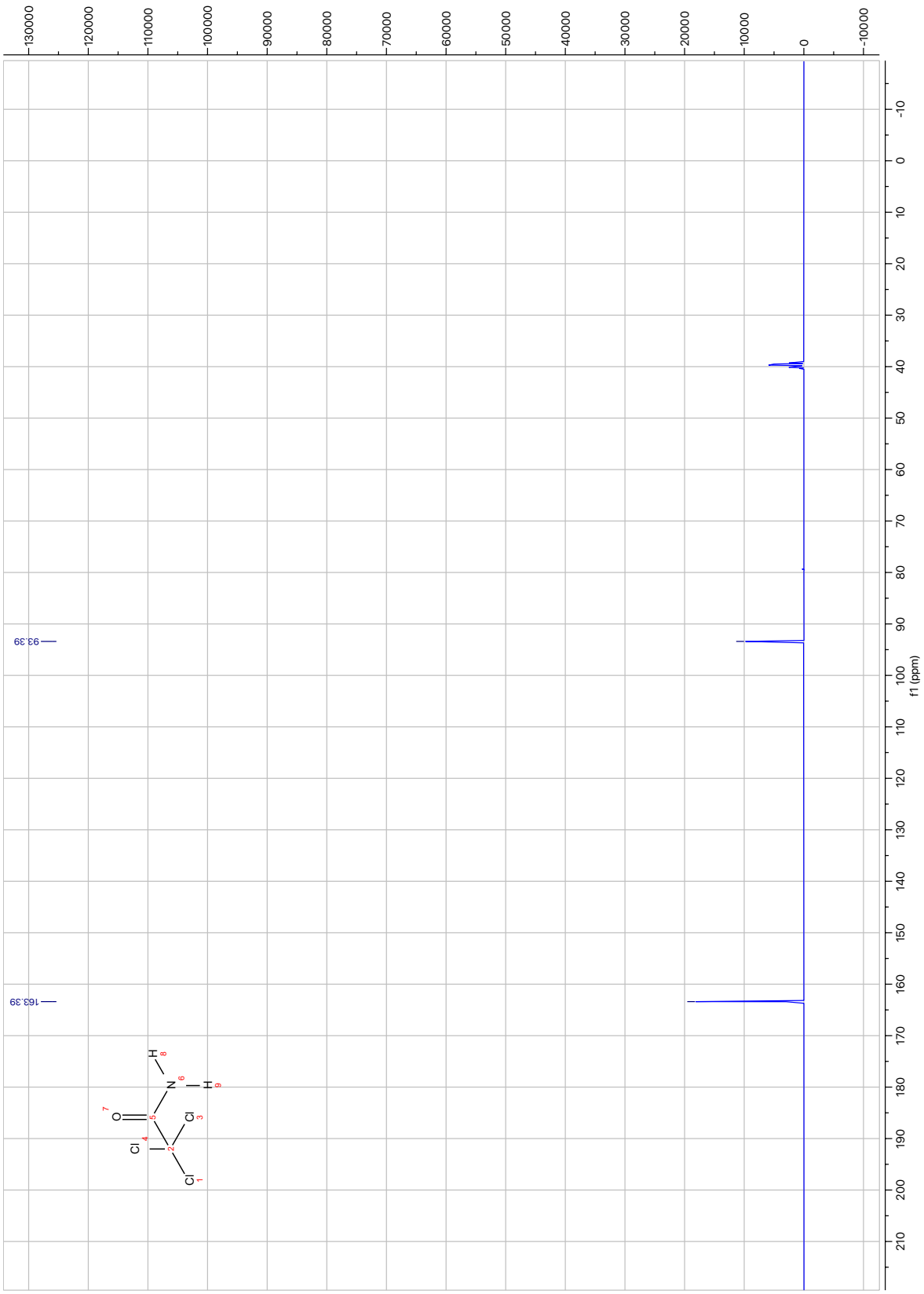

28.19
32.20
41.53
61.27
79.26
155.82
170.15
f1 (ppm)


2.87
2.85
7.77
7.76
3.00
1.01
f1 (ppm)


26.00
110.18
113.98
117.77
121.57
157.62
158.11
158.60
159.09
f1 (ppm)


-76.61
f1 (ppm)


f1 (ppm)
8.12
2.00


130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
f1 (ppm)
93.39
163.39
Cl
Cl
Cl
O
H
N
H