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Catalytic Aerobic Synthesis of Quaternary α-Hydroxy phosphonates via Direct Hydroxylation of Phosphonate Compounds

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Supporting Information

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(A) Materials and equipment

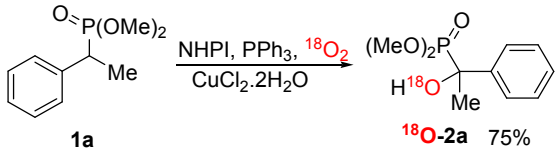
Reagents were obtained commercially and used as received. Solvents were purified and dried by standard methods. For substrates **1** were prepared according the literature methods.[1] ^{1}H NMR spectra were recorded on a Bruker-400 NMR spectrometer using TMS as an internal standard. Chemical shift values (δ) are given in ppm. Coupling constants (J) were measured in Hz. GC-MS analyses were performed on a SHIMADZU QP2010. High Resolution mass spectrometer (HRMS) spectra were recorded on a Bruker micrOTOF-Q II analyzer. 200-300 mesh silica gel was used for column chromatography.

(B) Typical experimental procedure

Typical experimental procedure for the synthesis of quaternary α-hydroxy phosphonates

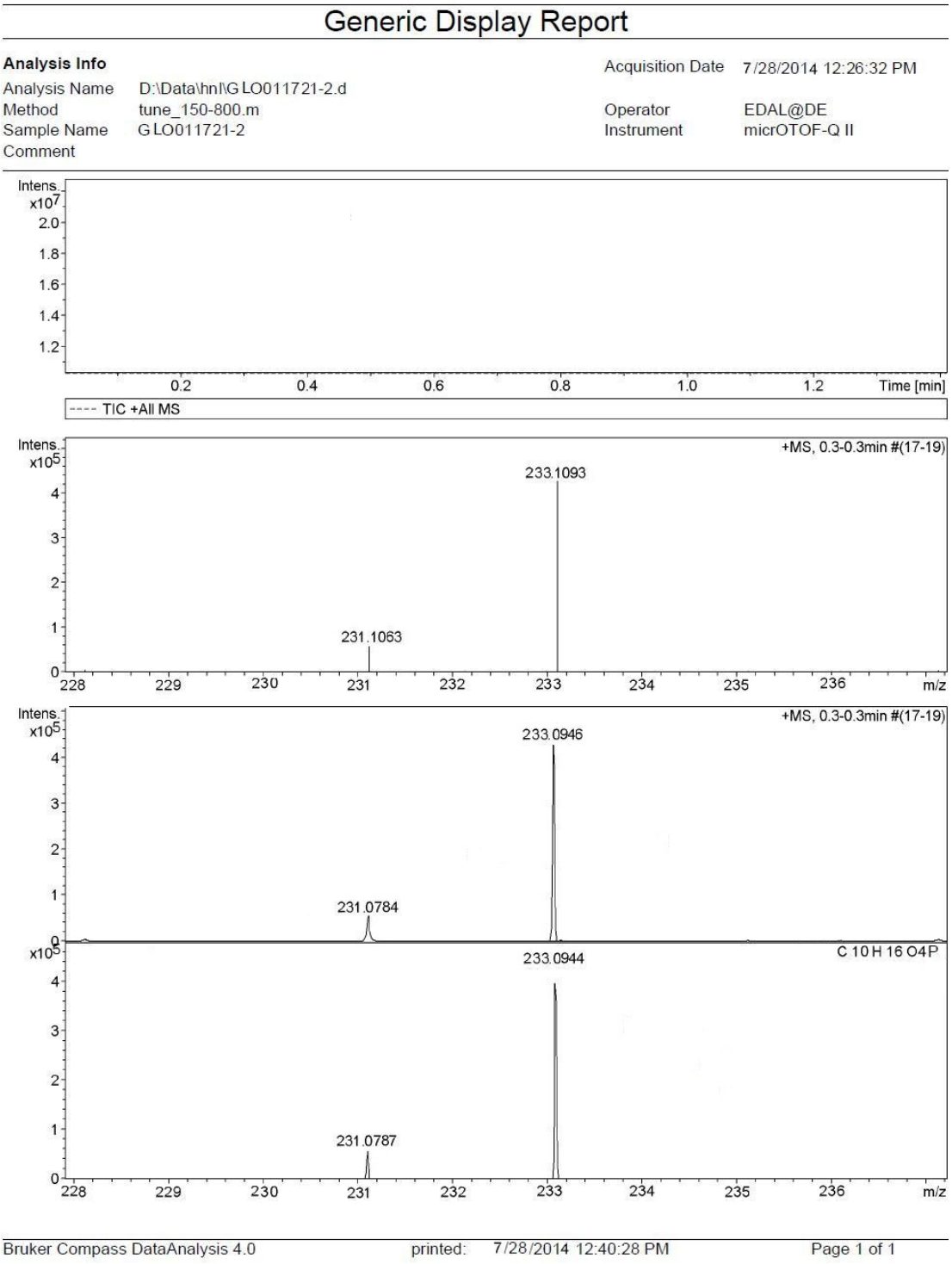
An oven-dried Schlenk tube was charged with a magnetic stir-bar, phosphonates **1** (0.3 mmol), $CuCl_2 \cdot 2H_2O$ (0.045 mmol), NHPI (0.03 mmol), PPh_3 (0.36 mmol), CH_3CN (2 mL), The tube was sealed, and oxygen was purged through syringe. Reaction was stirred at 100 ˚C for 8-10 h. After the reaction was finished, the reaction mixture was diluted in 30 mL ethyl acetate, filtered on celite pad. The organic portion was washed with a saturated solution of brine (8 mL×3), dried (Na_2SO_4) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **2**.

(C) **Labeling experiments**

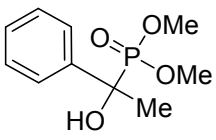


Reaction conditions: **1a** (0.3 mmol), $CuCl_2 \cdot 2H_2O$ (5 mol%), NHPI (10 mol%), CH_3CN (2.0 mL), 100 ˚C in $^{18}O_2$ atmosphere (1 atm) for 10 h.

The HRMS spectra of 2a for the reaction under $^{18}O_2$ (95%).



(D) Analytical data

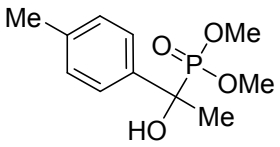


2a

Dimethyl 1-hydroxy-1-phenylethylphosphonate (2a): [2]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.59-7.55 (m, 2H), 7.40-7.28 (m, 3H), 4.41 (br, 1H), 3.71 (d, J = 10.4 Hz, 3H), 3.58 (d, J = 10.0 Hz, 3H), 1.79 (d, J = 15.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:

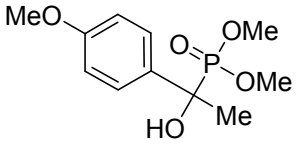
141.2 (d, J = 0.8 Hz), 128.3 (d, J = 2.0 Hz), 127.1 (d, J = 2.6 Hz), 125.4 (d, J = 4.1 Hz), 73.5 (d, J = 159.7 Hz), 54.3 (d, J = 7.3 Hz), 53.4 (d, J = 7.4 Hz), 26.2 (d, J = 3.2 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 26.21; IR (neat cm^{-1}): 3251, 3031, 2921,1226, 1021, 990, 766; LRMS (EI 70 ev) *m/z* (%): 230 (M^+, 100); HRMS m/z (ESI) calcd for $C_{10}H_{16}O_4P$ $(M+H)^+$ 231.0787, found 231.0784.



2b

Dimethyl 1-hydroxy-1-p-tolylethylphosphonate (2b): [2]

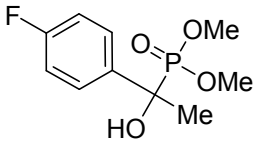
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48 (dd, J = 8.0 Hz, J = 2.0 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 4.46 (br, 1H), 3.77 (d, J = 10.0 Hz, 3H), 3.63 (d, J = 10.0 Hz, 3H), 2.32 (d, J = 1.2 Hz, 3H), 1.83 (d, J = 15.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 138.3 (d, J = 8.0 Hz), 136.4, 128.1, 125.2 (d, J = 4.2 Hz), 73.3 (d, J = 163.4 Hz), 54.1 (d, J = 7.7 Hz), 53.5 (d, J = 7.7 Hz), 25.7 (d, J = 3.3 Hz), 21.0; IR (neat cm^{-1}): 3269, 2937, 1447, 1221, 1020, 941; ^{31}P NMR (161 MHz, $CDCl_3$) δ: 26.64; LRMS (EI 70 ev) *m/z* (%): 244 (M^+, 100); HRMS m/z (ESI) calcd for $C_{11}H_{18}O_4P$ $(M+H)^+$ 245.0943, found 245.0939.



2c

Dimethyl 1-hydroxy-1-(4-methoxyphenyl)ethylphosphonate (2c): [2]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (dd, J = 2.0 Hz, J = 8.0 Hz, 2H), 6.83 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 3.69 (d, J = 10.0 Hz, 3H), 3.57 (d, J = 10.0 Hz, 3H), 1.76 (d, J = 15.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.2 (d, J = 2.5 Hz), 132.1, 127.4 (d, J = 4.3 Hz), 113.4 (d, J = 2.0 Hz), 73.2 (d, J = 159.2 Hz), 55.4, 54.4 (d, J = 7.3 Hz), 53.2 (d, J = 7.3 Hz), 25.5 (d, J = 4.1 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 26.51; LRMS (EI 70 ev) *m/z* (%): 260 (M^+, 100); HRMS m/z (ESI) calcd for $C_{11}H_{18}O_5P$ $(M+H)^+$ 261.0893, found 261.0890.

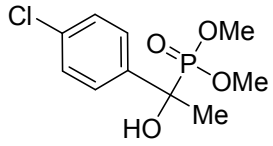


2d

Dimethyl 1-(4-fluorophenyl)-1-hydroxyethylphosphonate (2d): [2]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.60-7.53 (m, 2H), 7.04 (t, J = 10.0 Hz, 2H), 4.47 (br, 1H), 3.77 (d, J = 10.4 Hz, 3H), 3.64 (d, J = 10.4 Hz, 3H), 1.83 (d, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.6, 159.8, 159.1, 137.18, 137.15, 137.13, 137.12, 127.4, 127.39, 127.37, 127.32, 114.3, 114.1, 114.05, 114.03, 73.9 (d, J = 159.8 Hz), 54.7 (d, J = 7.6 Hz), 53.6 (d, J = 7.4 Hz), 26.1 (d, J = 4.4 Hz);

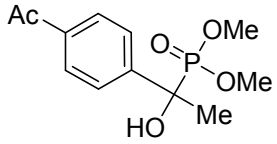
^{31}P NMR (161 MHz, $CDCl_3$) δ: 25.76; LRMS (EI 70 ev) *m/z* (%): 248 (M^+, 100); HRMS m/z (ESI) calcd for $C_{10}H_{15}FO_4P$ $(M+H)^+$ 249.0693, found 249.0699.



2e

Dimethyl 1-(4-chlorophenyl)-1-hydroxyethylphosphonate (2e): [2]

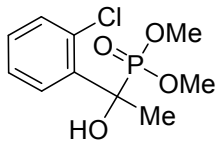
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (dd, J = 2.0 Hz, J = 8.4 Hz, 2H), 7.23 (d, J = 8.4 Hz, 2H), 4.49 (br, 1H), 3.69 (d, J = 10.0 Hz, 3H), 3.58 (d, J = 10.0 Hz, 3H), 1.71 (d, J = 15.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.1, 133.0 (d, J = 3.1 Hz), 128.6 (d, J = 2.2 Hz), 127.9 (d, J = 4.0 Hz), 73.1 (d, J = 159.5 Hz), 54.7 (d, J = 7.8 Hz), 53.5 (d, J = 7.6 Hz), 25.7 (d, J = 7.8 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 25.80; LRMS (EI 70 ev) *m/z* (%): 264 (M^+, 78); HRMS m/z (ESI) calcd for $C_{10}H_{15}ClO_4P$ $(M+H)^+$ 265.0397, found 265.0399.



2f

Dimethyl 1-(4-acetylphenyl)-1-hydroxyethylphosphonate (2f):

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (dd, J = 7.2 Hz, J = 1.2 Hz, 2H), 7.29 (d, J = 7.2 Hz, 2H), 4.47 (br, 1H), 3.73 (s, 3H), 3.69 (s, 3H), 2.61 (s, 3H), 1.78 (d, J = 15.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 198.7, 138.0 (d, J = 4.4 Hz), 133.4 (d, J = 2.4 Hz), 129.0 (d, J = 2.6 Hz), 128.6 (d, J = 3.7 Hz), 73.6 (d, J = 157.3 Hz), 54.5 (d, J = 7.1 Hz), 53.5 (d, J = 7.2 Hz), 26.3, 24.6 (d, J = 3.3 Hz). ^{31}P NMR (161 MHz, $CDCl_3$) δ: 24.59; IR (neat cm^{-1}): 3262, 2963, 1657, 1604, 1221, 1063, 1027, 979; LRMS (EI 70 ev) *m/z* (%): 272 (M^+, 100); HRMS m/z (ESI) calcd for $C_{12}H_{18}O_5P$ $(M+H)^+$ 273.0893, found 273.0891.

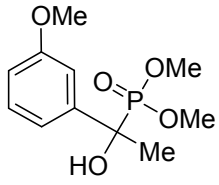


2g

Dimethyl 1-(2-chlorophenyl)-1-hydroxyethylphosphonate (2g): [2]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66-7.62 (m, 1H), 7.24 (dd, J = 1.2 Hz, J = 7.6 Hz, 1H), 7.19-7.14 (m, 2H), 4.46 (br, 1H), 3.68 (d, J = 10.4 Hz, 6H), 1.88 (d, J = 15.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.1 (d, J = 3.1 Hz), 131.8 (d, J = 5.3 Hz), 131.0 (d, J = 1.9 Hz), 129.4 (d, J = 3.5 Hz), 128.6 (d, J = 2.6 Hz), 126.3 (d, J = 1.7 Hz), 74.7 (d, J = 158.7 Hz), 54.4 (d, J = 7.0 Hz), 53.5 (d, J = 6.8 Hz), 25.3; ^{31}P NMR (161 MHz, $CDCl_3$) δ: 25.31; LRMS (EI 70 ev) *m/z* (%): 264 (M^+, 86); HRMS m/z

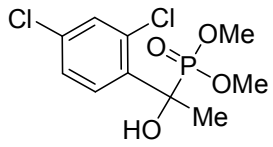
(ESI) calcd for $C_{10}H_{15}ClO_4P$ $(M+H)^+$ 265.0397, found 265.0400.



2h

Dimethyl 1-hydroxy-1-(3-methoxyphenyl)ethylphosphonate **(2h):** [3]

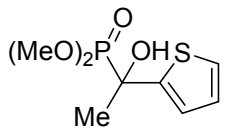
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.31-7.28 (m, 1H), 7.23-7.19 (m, 2H), 6.88-6.85 (m, 1H), 4.44 (br, 1H), 3.87 (s, 3H), 3.79 (d, J = 10.4 Hz, 3H), 3.66 (d, J = 10.4 Hz, 3H), 1.81 (d, J = 15.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.1 (d, J = 2.0 Hz), 142.3 (d, J = 1.2 Hz), 129.1 (d, J = 2.6 Hz), 118.7 (d, J = 4.6 Hz), 113.2 (d, J = 2.8 Hz), 110.9 (d, J = 4.3 Hz), 73.5 (d, J = 158.2 Hz), 55.7, 54.3 (d, J = 7.7 Hz), 53.2 (d, J = 7.7 Hz), 25.1 (d, J = 3.5 Hz); IR (neat cm^{-1}): 3291, 2921, 1441, 1231, 1129, 1044, 789; ^{31}P NMR (161 MHz, $CDCl_3$) δ: 25.99; LRMS (EI 70 ev) *m/z* (%): 260 (M^+, 100); HRMS m/z (ESI) calcd for $C_{11}H_{18}O_5P$ $(M+H)^+$ 261.0893, found 261.0898.



2i

Dimethyl 1-(2,4-dichlorophenyl)-1-hydroxyethylphosphonate **(2i):** [4]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (d, J = 7.2 Hz, 1H), 7.49 (d, J = 8.8 Hz, 1H), 7.40 (d, J = 6.4 Hz, 1H), 4.475(br, 1H), 3.69 (d, J = 10.4 Hz, 3H), 3.57 (d, J = 10.8 Hz, 3H), 1.85 (d, J = 15.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.3 (d, J = 4.4 Hz), 133.4 (d, J = 3.9 Hz), 132.5 (d, J = 2.0 Hz), 131.8 (d, J = 5.8 Hz), 129.9 (d, J = 3.1 Hz), 127.1 (d, J = 2.6 Hz), 73.4 (d, J = 161.9 Hz), 54.4 (d, J = 7.1 Hz), 53.6 (d, J = 7.4 Hz), 24.8 (d, J = 3.3 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 25.33; LRMS (EI 70 ev) *m/z* (%): 298 (M^+, 31); IR (neat cm^{-1}): 3277, 2966, 1441, 1227, 1033, 853, 790; HRMS m/z (ESI) calcd for $C_{10}H_{14}Cl_2O_4P$ $(M+H)^+$ 299.0007, found 299.0011.

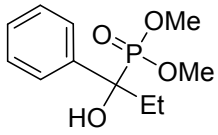


2j

Dimethyl 1-hydroxy-1-(thiophen-2-yl)ethylphosphonate (2j): [4]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.31-7.27 (m, 1H), 7.12-7.08 (m, 1H), 6.95-6.92 (m, 1H), 4.51 (br, 1H), 3.66 (d, J = 10.4 Hz, 3H), 3.58 (d, J = 10.4 Hz, 3H), 1.74 (d, J = 15.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.1, 142.3 (d, J = 10.1 Hz), 111.0, 108, 71.1 (d, J = 162.9 Hz), 54.3 (d, J = 7.5 Hz), 53.1 (d, J = 7.1 Hz), 23.1 (d, J = 2.8 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 24.67; LRMS (EI 70 ev) *m/z*

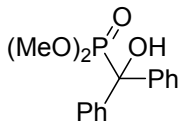
(%): 236 (M^+, 100); HRMS m/z (ESI) calcd for $C_8H_{14}O_4PS$ $(M+H)^+$ 237.0350, found 237.0354.



2k

Dimethyl 1-hydroxy-1-phenylpropylphosphonate (2k): [3]

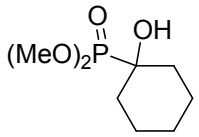
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52-7.49 (m, 2H), 7.31 (t, J = 7.8 Hz, 2H), 7.23-7.20 (m, 1H), 4.43 (br, 1H), 3.69 (d, J = 10.0 Hz, 6H), 2.61-2.27 (m, 2H), 0.87 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 138.7, 127.8 (d, J = 3.0 Hz), 127.0 (d, J = 3.3 Hz), 125.8 (d, J = 4.3 Hz), 75.7 (d, J = 158.6 Hz), 54.6 (d, J = 7.5 Hz), 53.1 (d, J = 7.3 Hz), 29.6 (d, J = 4.3 Hz), 9.1 (d, J = 8.9 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 25.29; LRMS (EI 70 ev) *m/z* (%): 244 (M^+, 100); HRMS m/z (ESI) calcd for $C_{11}H_{18}O_4P$ $(M+H)^+$ 245.0943, found 245.0944.



2l

Dimethyl hydroxydiphenylmethylphosphonate (2l): [4]

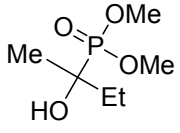
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, J = 7.6 Hz, 4H), 7.37-7.31 (m, 4H), 7.23-7.20 (m, 2H), 4.51 (br, 1H), 3.71 (d, J = 10.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.4 (d, J = 1.4 Hz), 127.7 (d, J = 2.3 Hz), 127.0 (d, J = 3.1 Hz), 126.5 (d, J = 4.2 Hz), 75.8 (d, J = 254.6 Hz), 54.8 (d, J = 7.0 Hz), 53.4 (d, J = 7.8 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 23.53; LRMS (EI 70 ev) *m/z* (%): 292 (M^+, 100); HRMS m/z (ESI) calcd for $C_{15}H_{18}O_4P$ $(M+H)^+$ 293.0944, found 293.0947.



2m

Dimethyl 1-hydroxycyclohexylphosphonate (2m): [5]

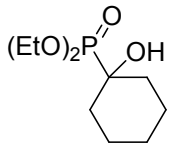
^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.48 (br, 1H), 3.71 (s, 3H), 3.68 (s, 3H), 1.85 (t, J = 10.2 Hz, 2H), 1.68-1.31 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 71.8 (d, J = 164.0 Hz), 53.3 (d, J = 7.5 Hz), 31.1 (d, J = 2.8 Hz), 25.1, 19.7 (d, J = 11.3 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 26.43; LRMS (EI 70 ev) *m/z* (%): 208 (M^+, 100); HRMS m/z (ESI) calcd for $C_8H_{18}O_4P$ $(M+H)^+$ 209.0945, found 209.0947.



2n

Dimethyl 2-hydroxybutan-2-ylphosphonate (2n): [2]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.49 (br, 1H), 3.74 (s, 3H), 3.71 (s, 3H), 1.85-1.81 (m, 1H), 1.69-1.62 (m, 1H), 1.33 (d, J = 15.6 Hz, 3H), 0.97 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 73.3 (d, J = 161.2 Hz), 54.5 (d, J = 6.8 Hz), 53.1 (d, J = 5.9 Hz), 29.2 (d, J = 5.0 Hz), 21.5 (d, J = 4.4 Hz), 7.1 (d, J = 8.0 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 30.10; IR (neat cm^{-1}): 3300, 2939, 1452, 1227, 1132, 1020; LRMS (EI 70 ev) *m/z* (%): 182 (M^+, 100); HRMS m/z (ESI) calcd for $C_6H_{16}O_4P$ $(M+H)^+$ 183.0791, found 183.0797.



2o

Diethyl 1-hydroxycyclohexylphosphonate (2o): [6]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.16-4.09 (m, 4H), 3.37 (br, 1H), 1.86 (t, J = 9.5 Hz, 2H), 1.68-1.60 (m, 5H), 1.52-1.49 (m, 2H), 1.31-1.27 (m, 6H), 1.20-1.14 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 71.7 (d, J = 163.9 Hz), 62.6 (d, J = 7.4 Hz), 31.4, 25.2, 20.0 (d, J = 11.0 Hz), 16.4 (d, J = 5.4 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ: 26.73; LRMS (EI 70 ev) *m/z* (%): 236 (M^+, 100); HRMS m/z (ESI) calcd for $C_{10}H_{22}O_4P$ $(M+H)^+$ 237.1257, found 237.1261.

(E) References

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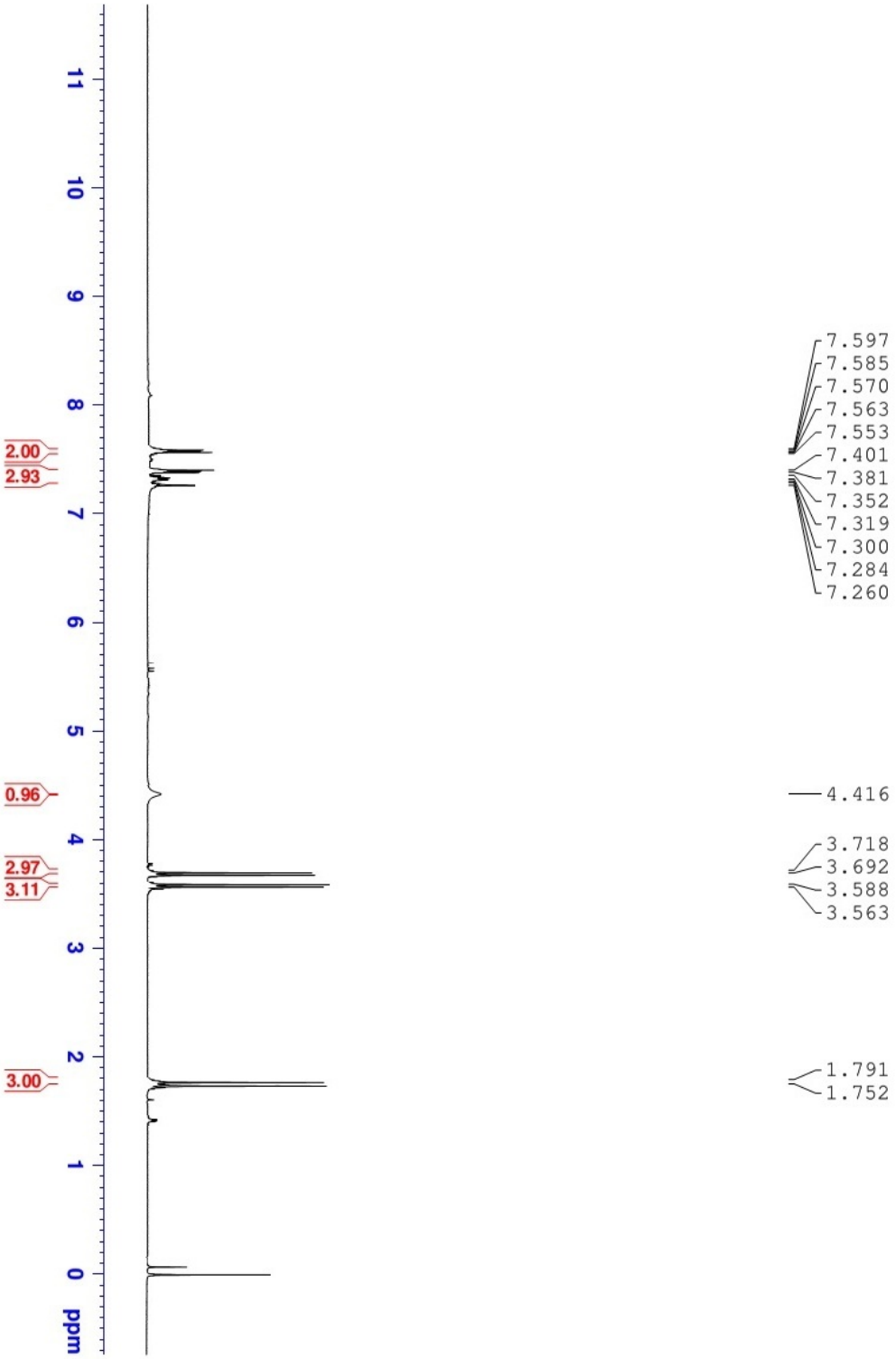
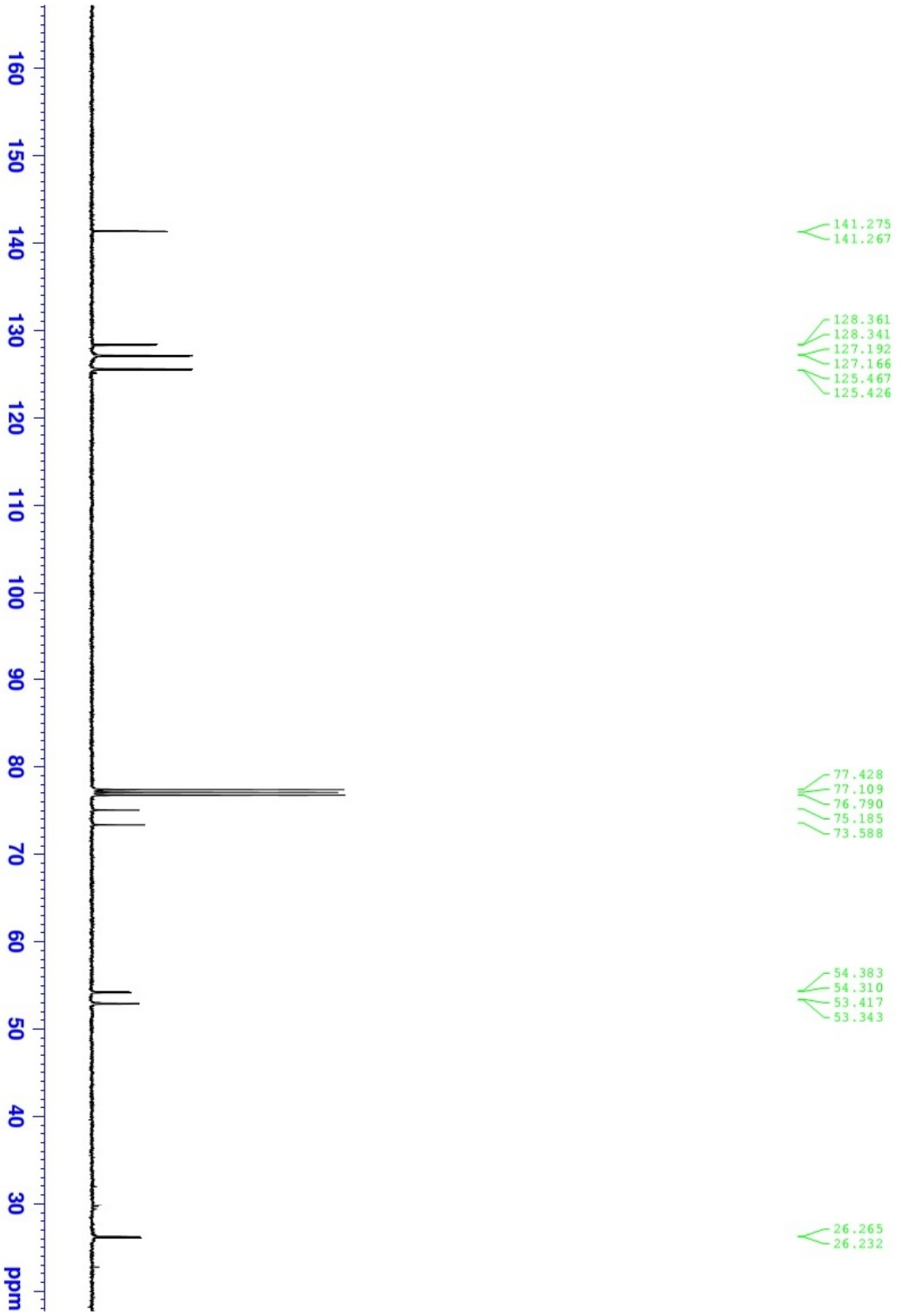
(F) Spectra



^{1}H NMR of Compound 2a



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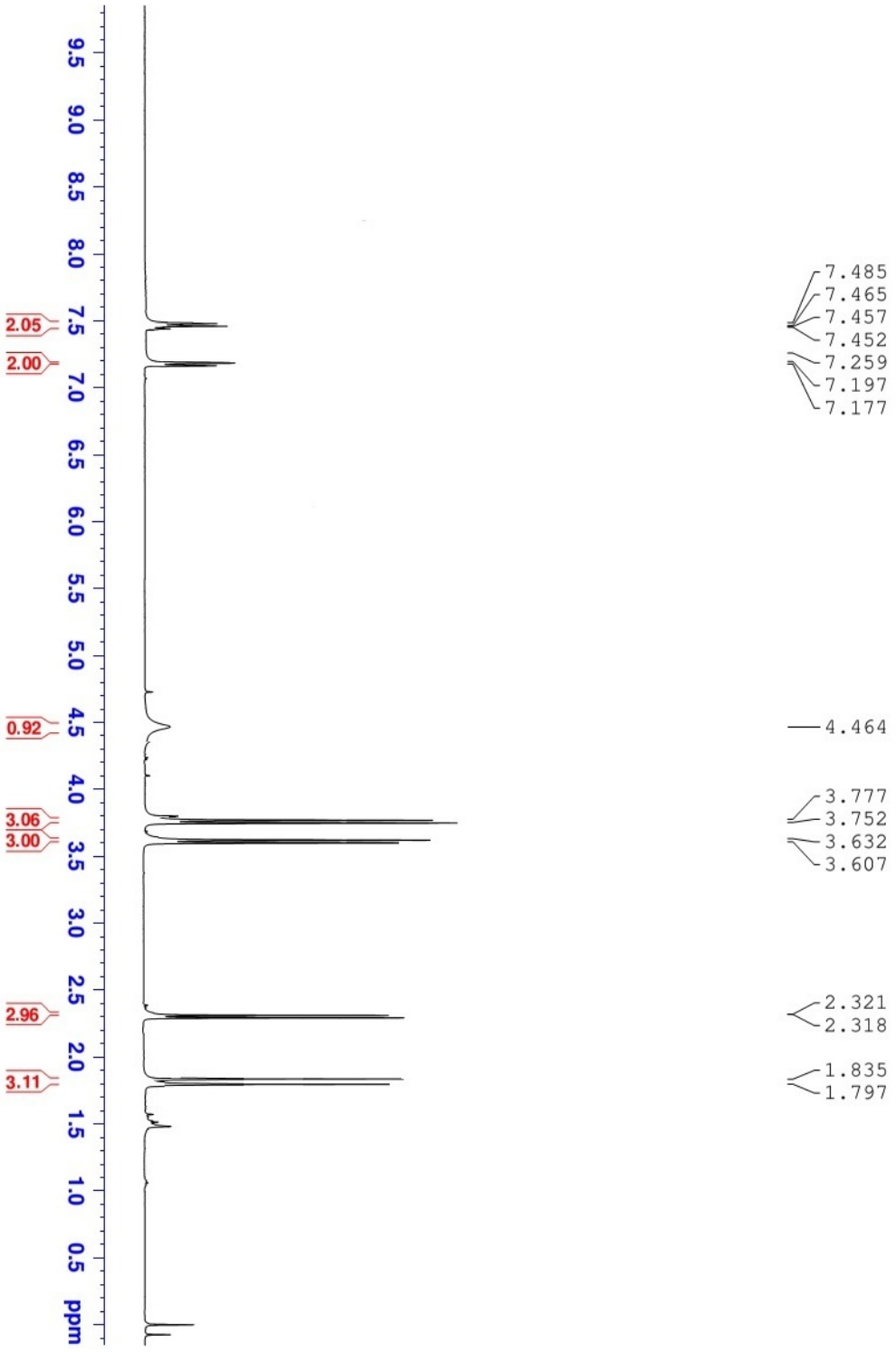

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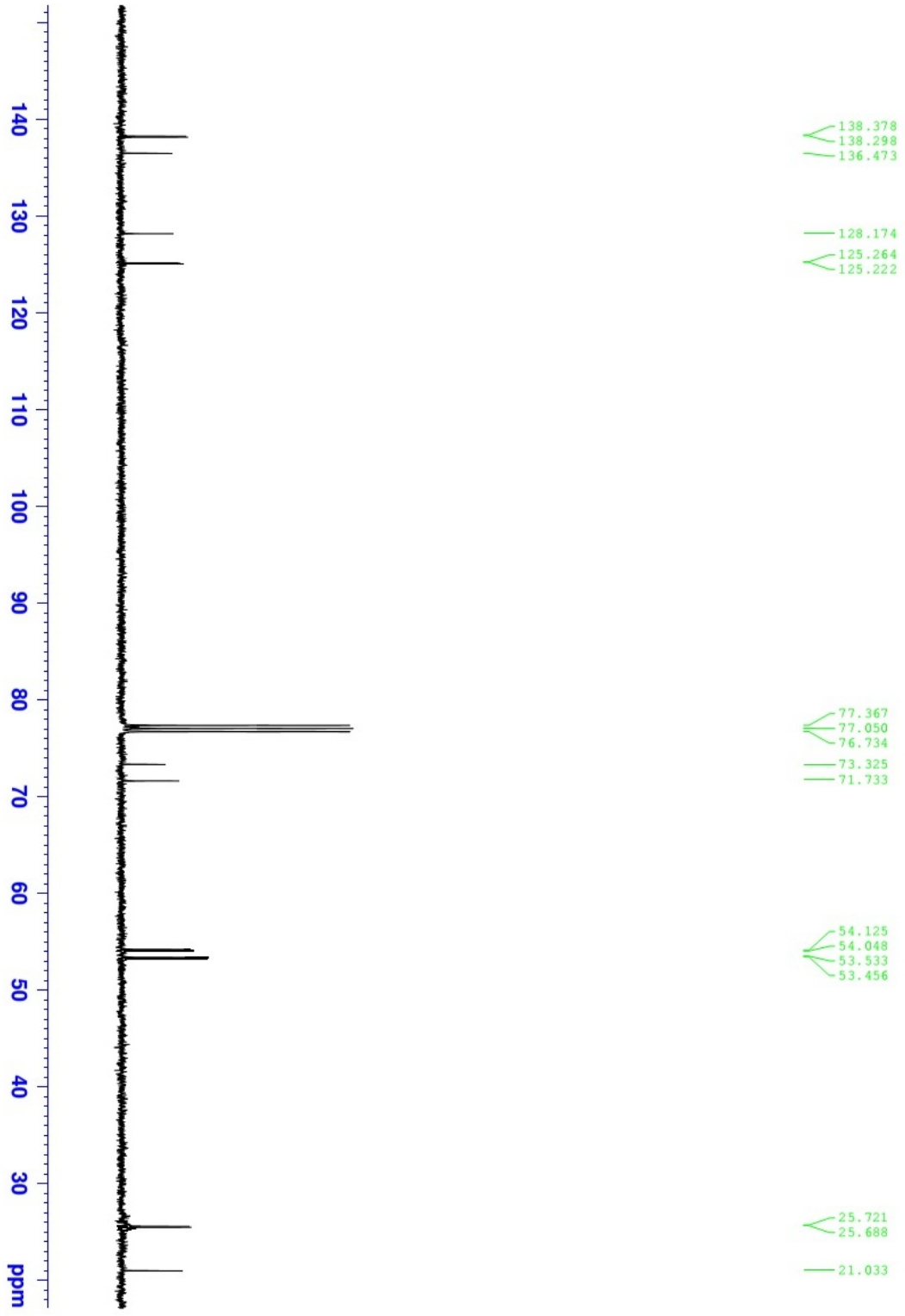

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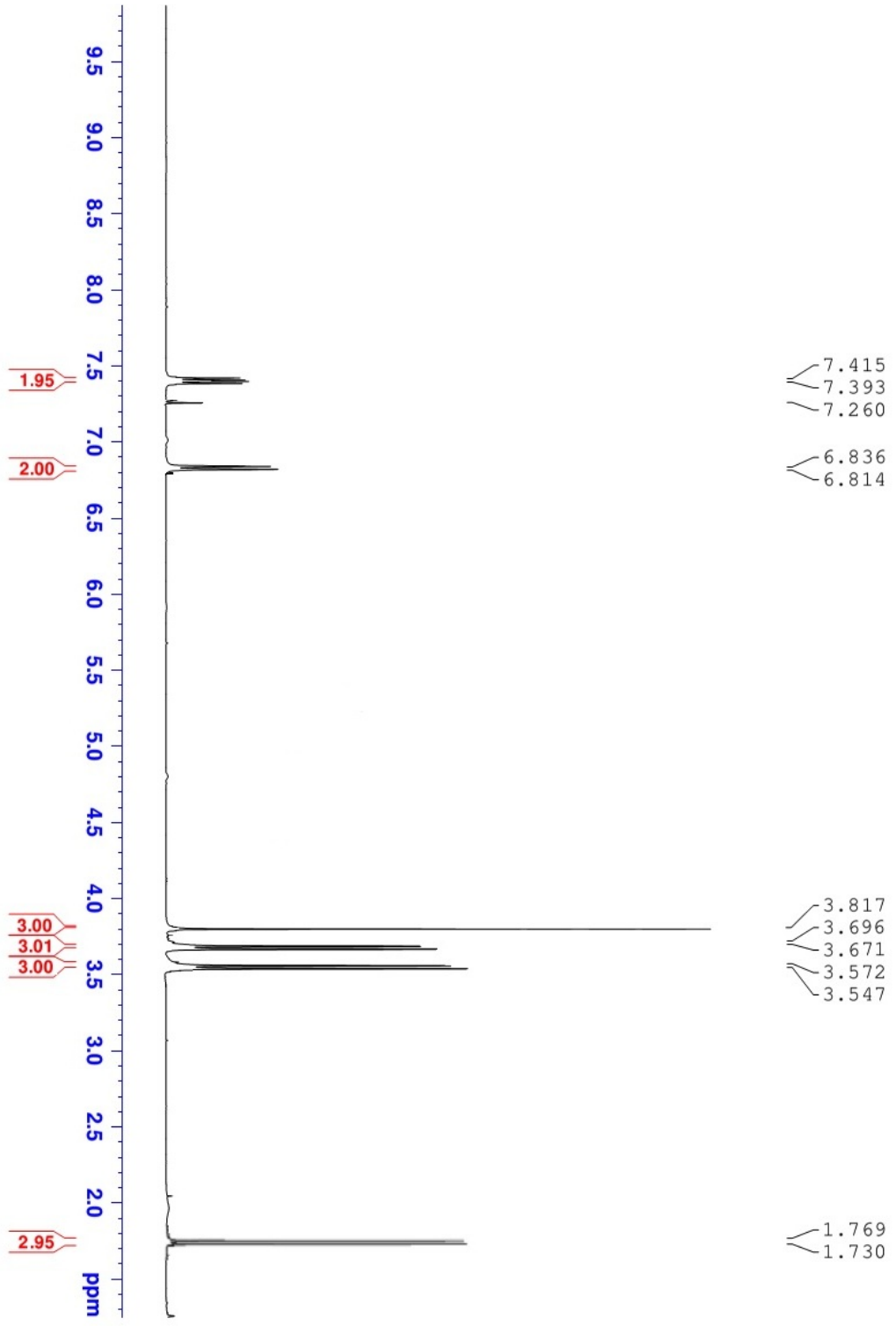

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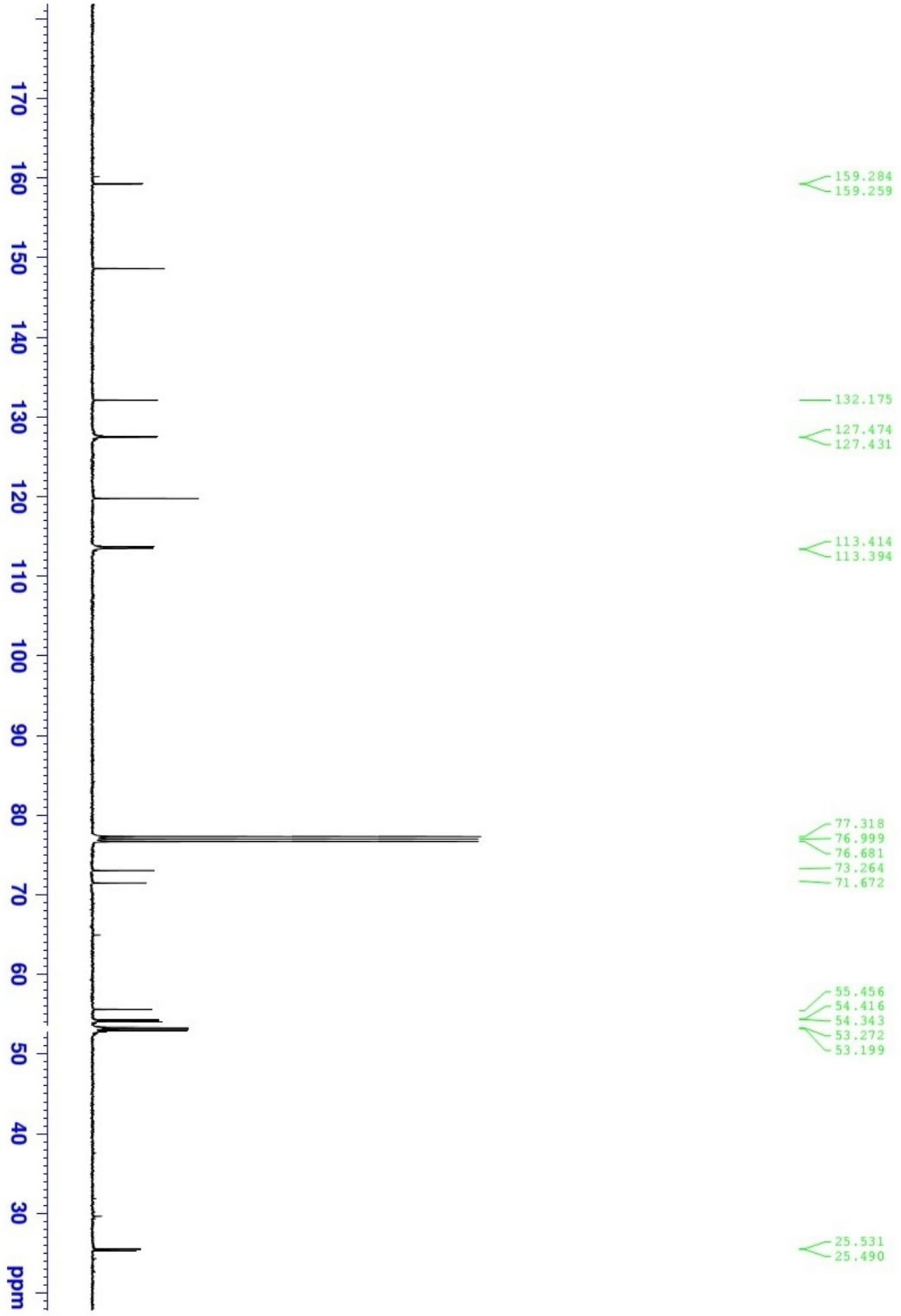

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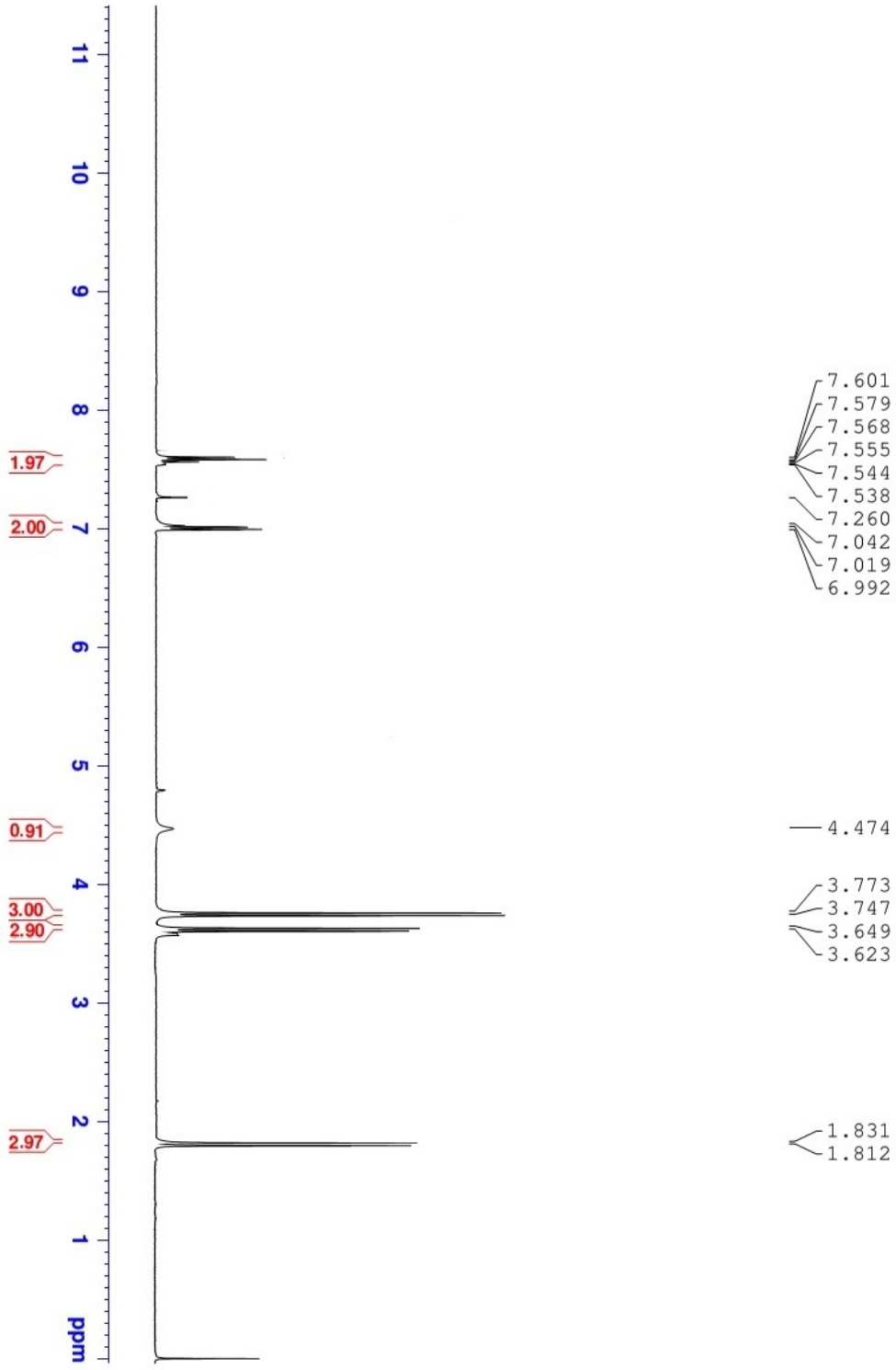

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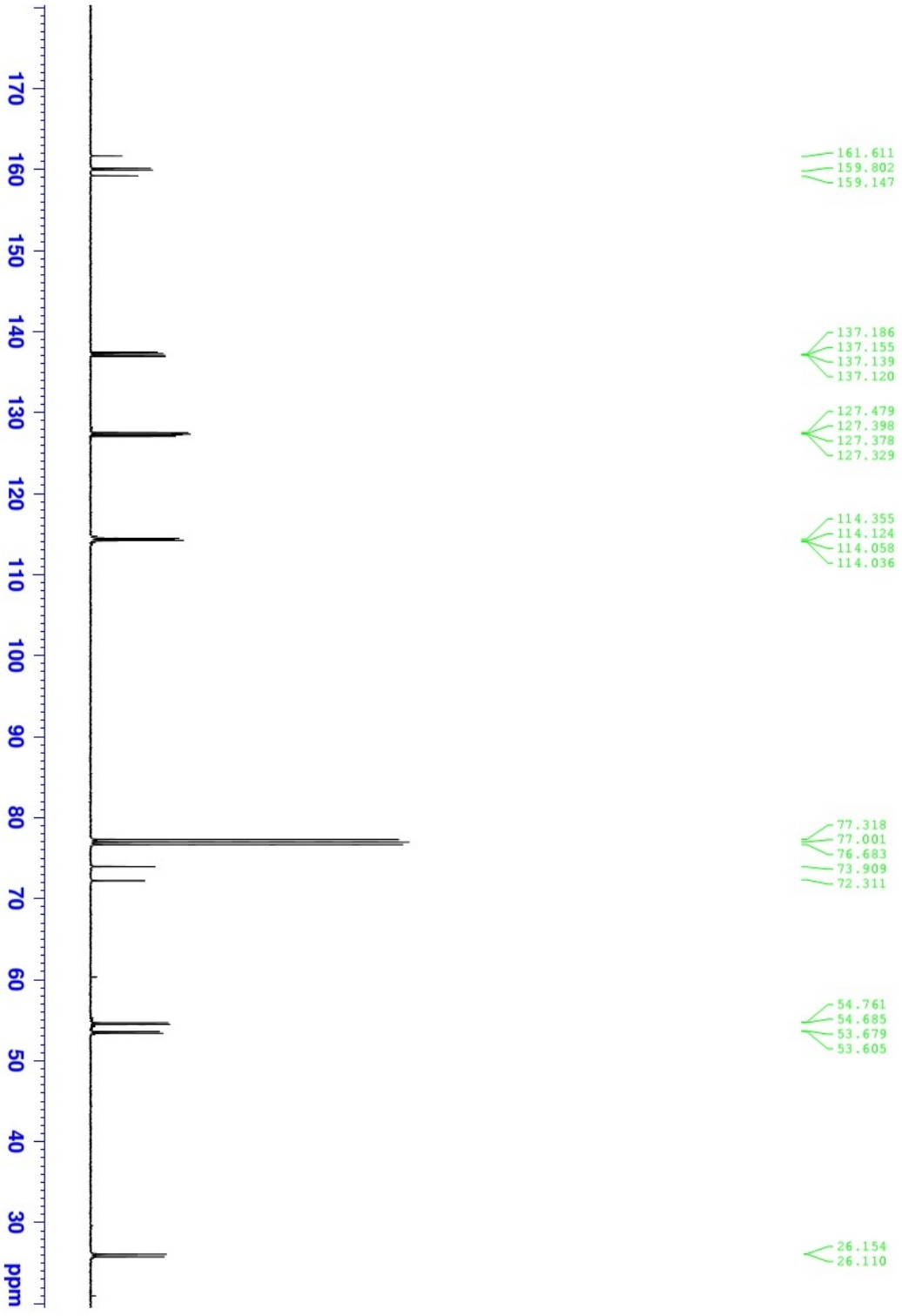

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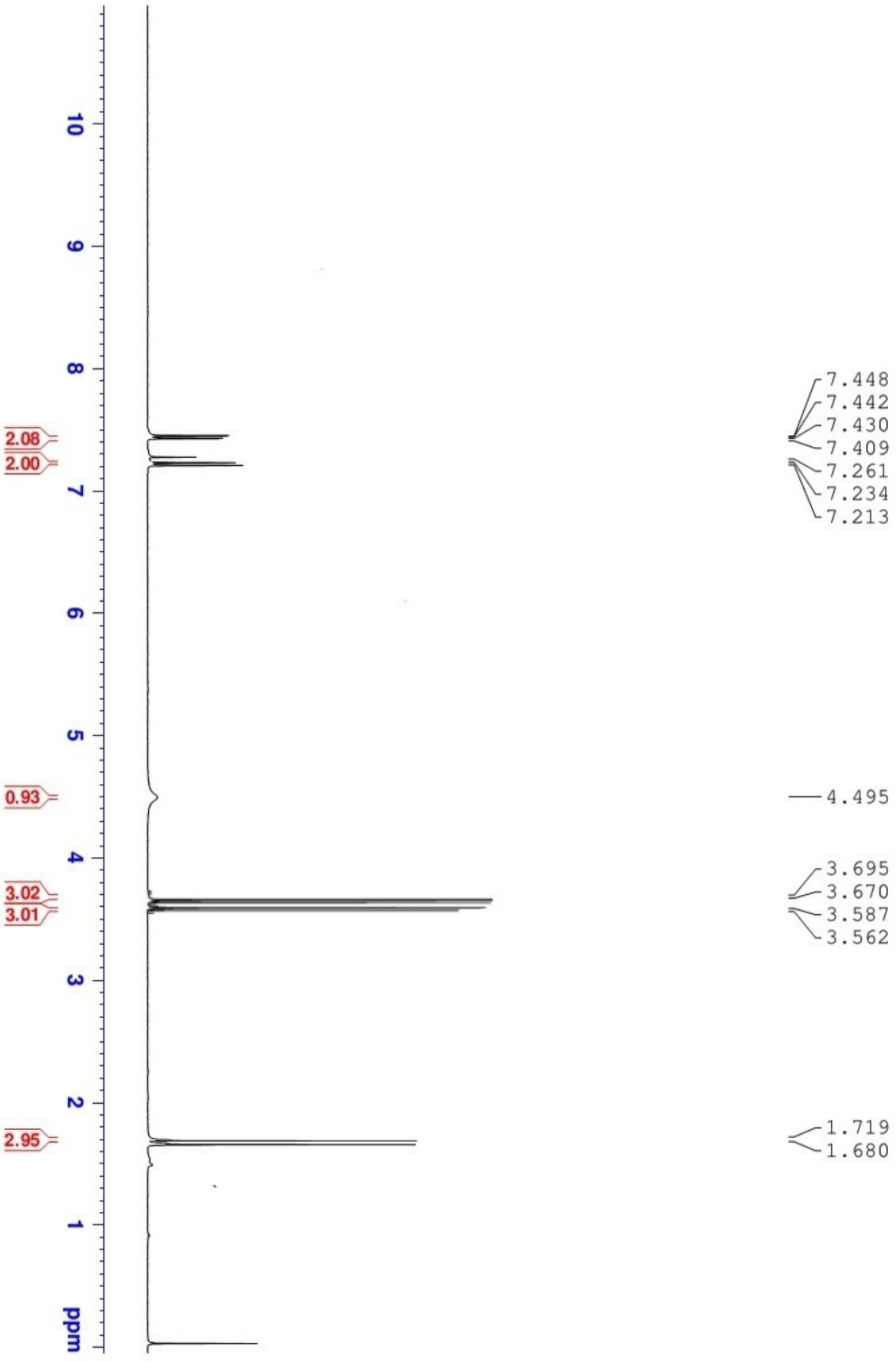

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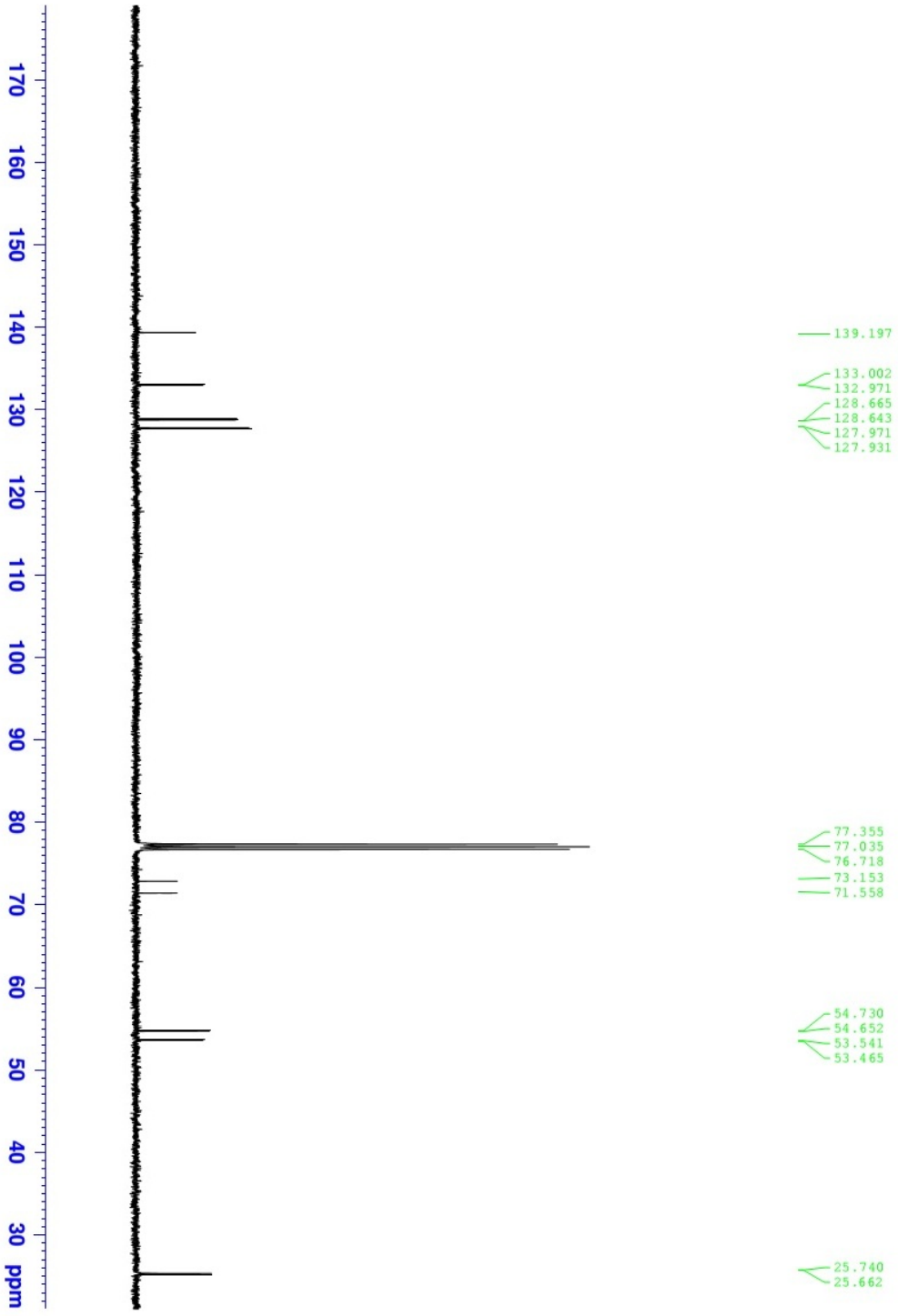

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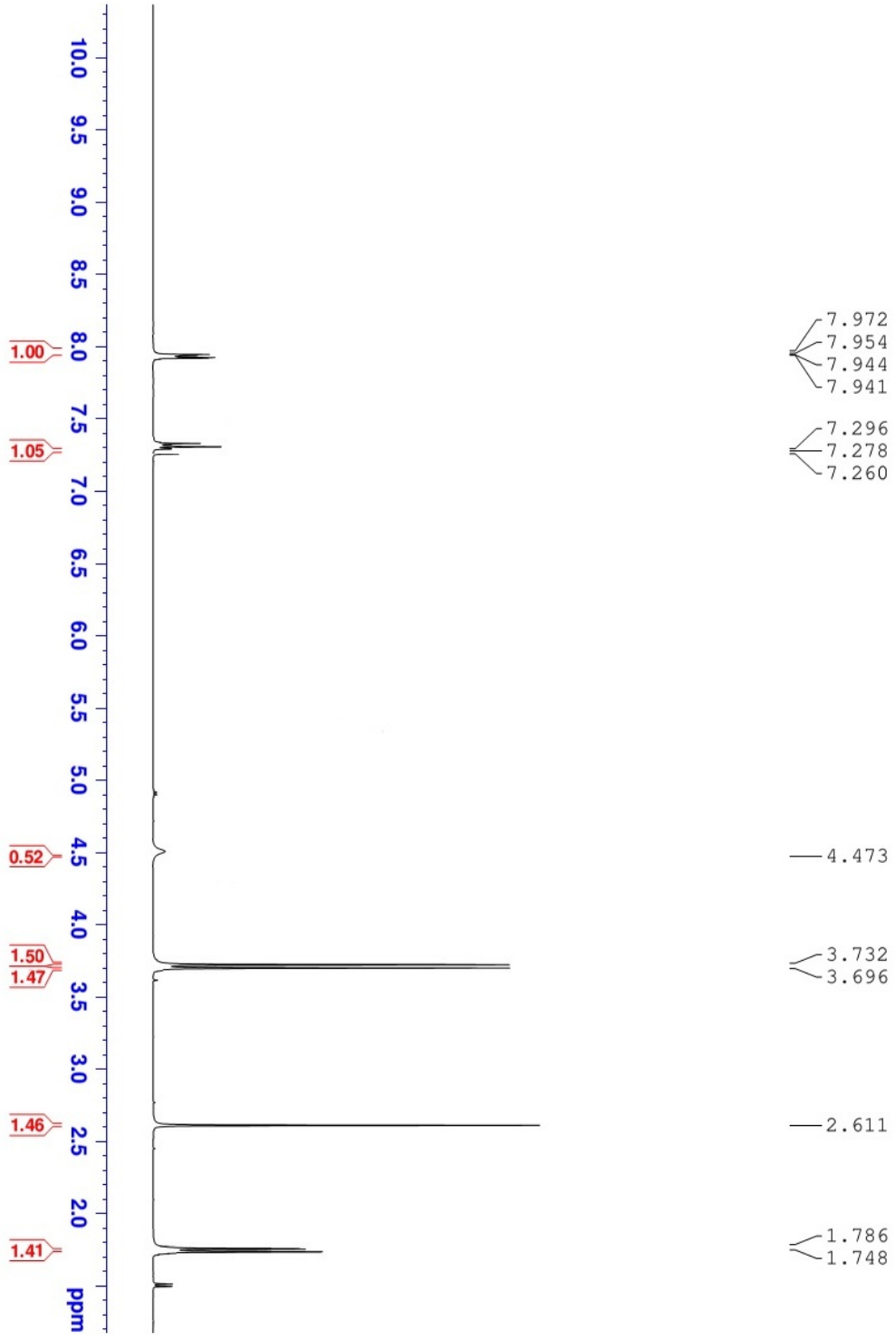

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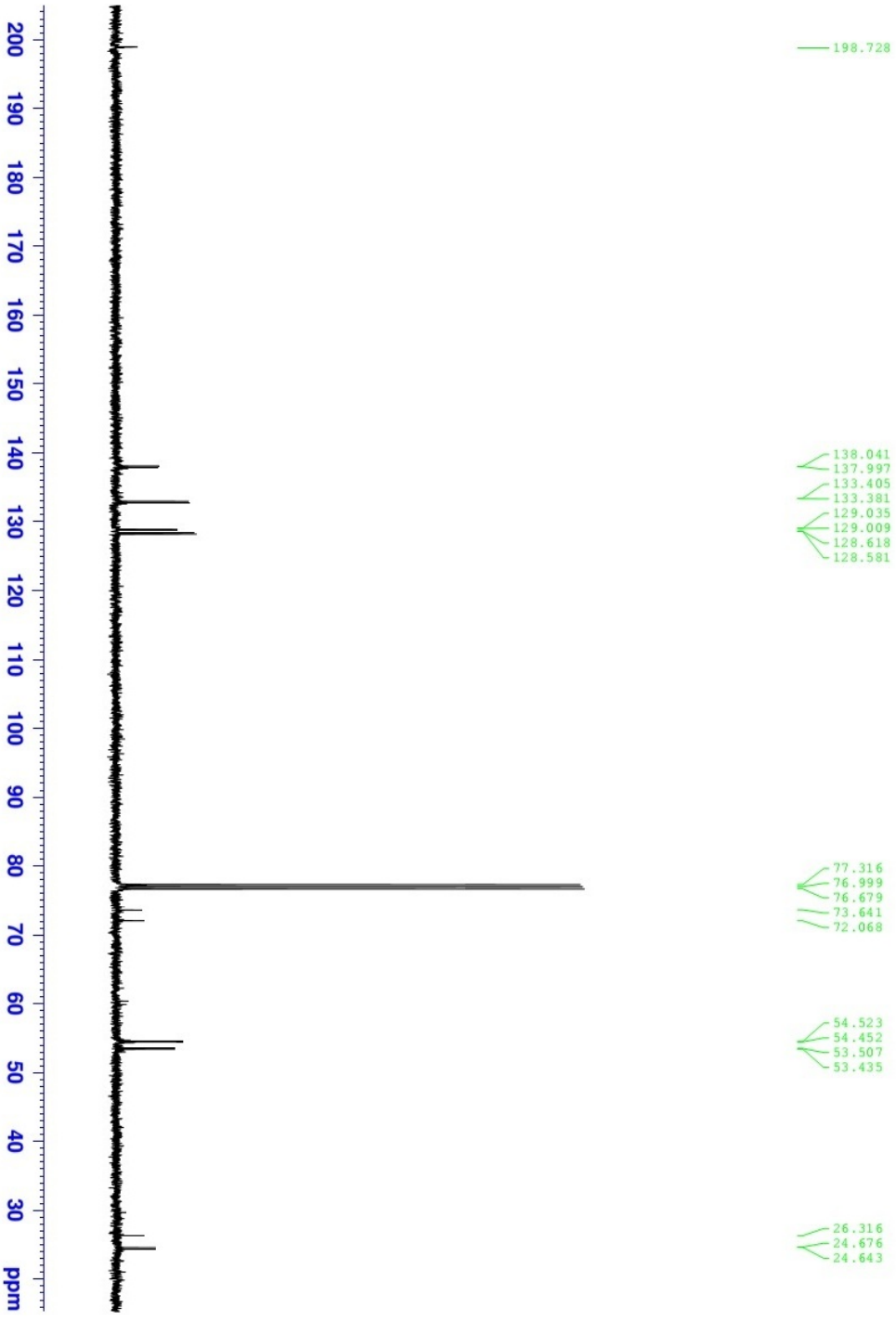

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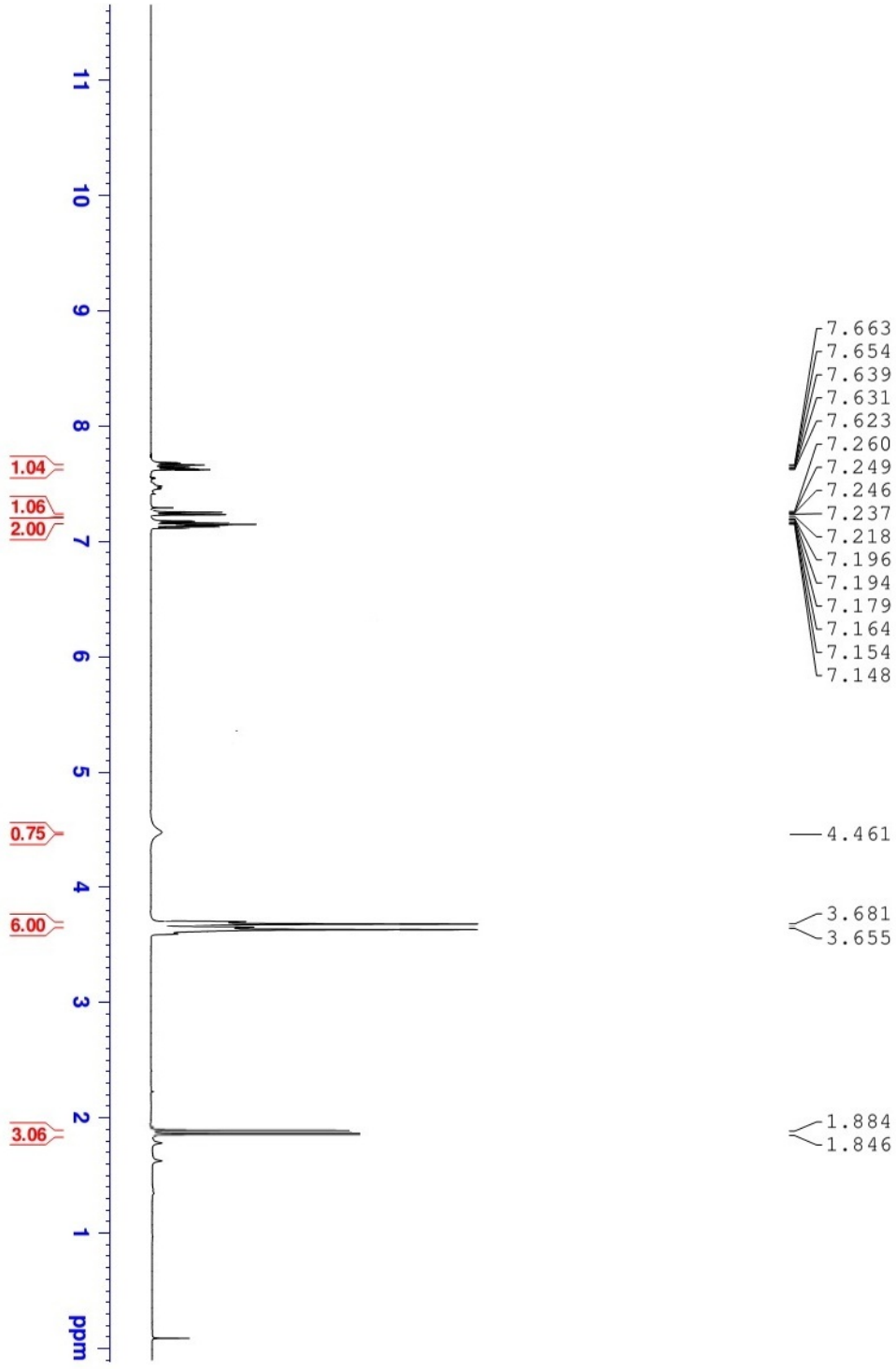

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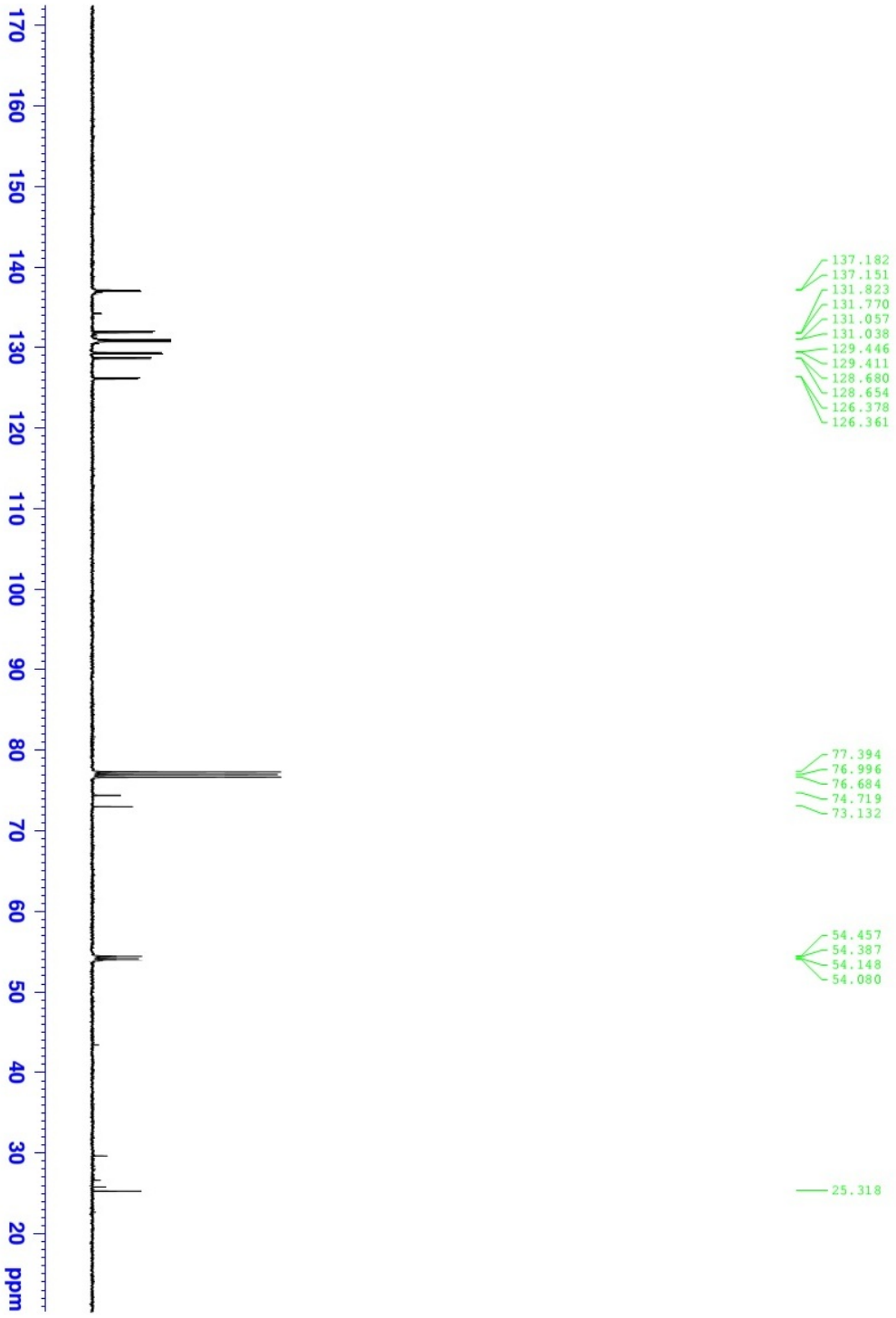

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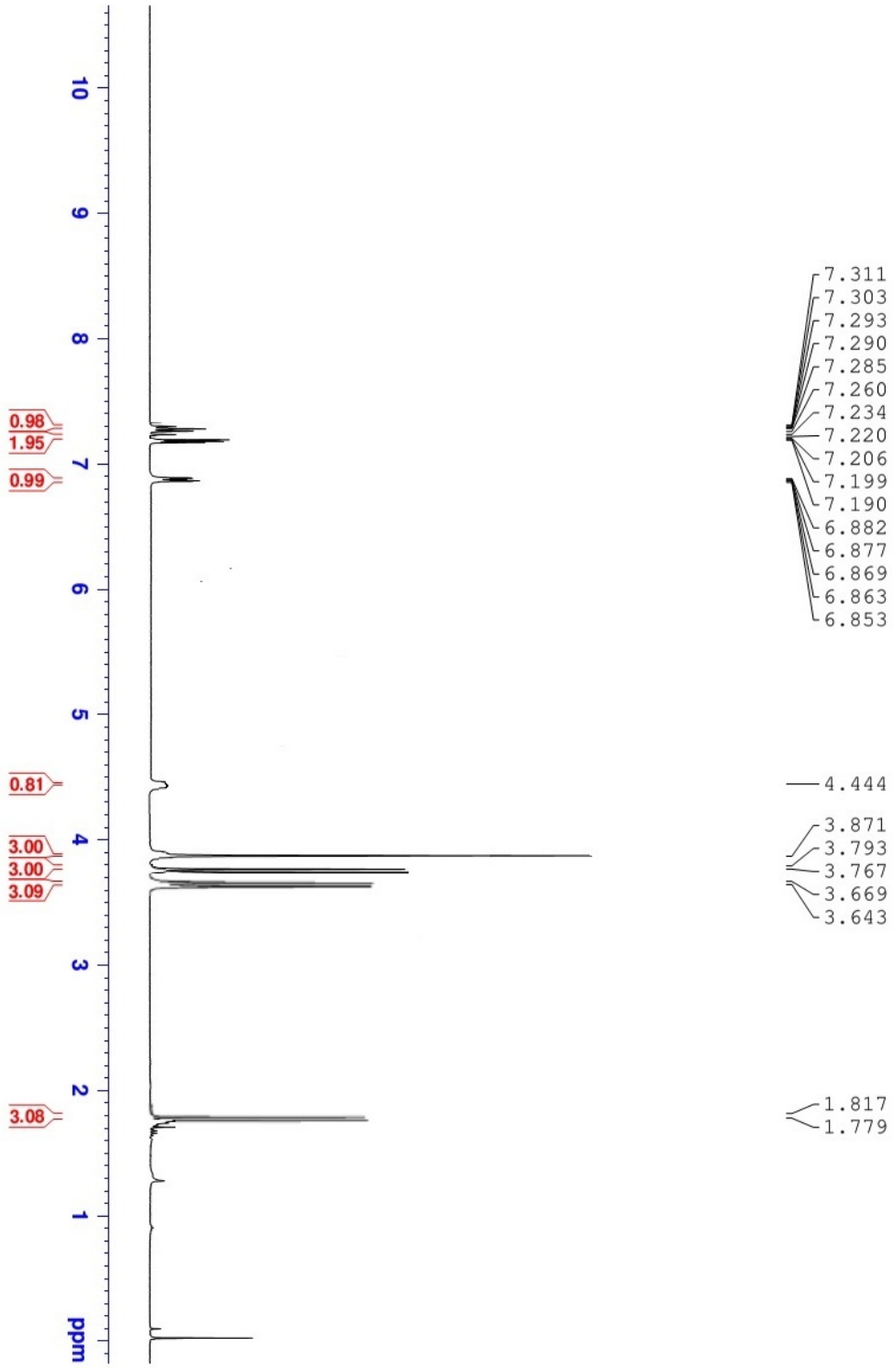

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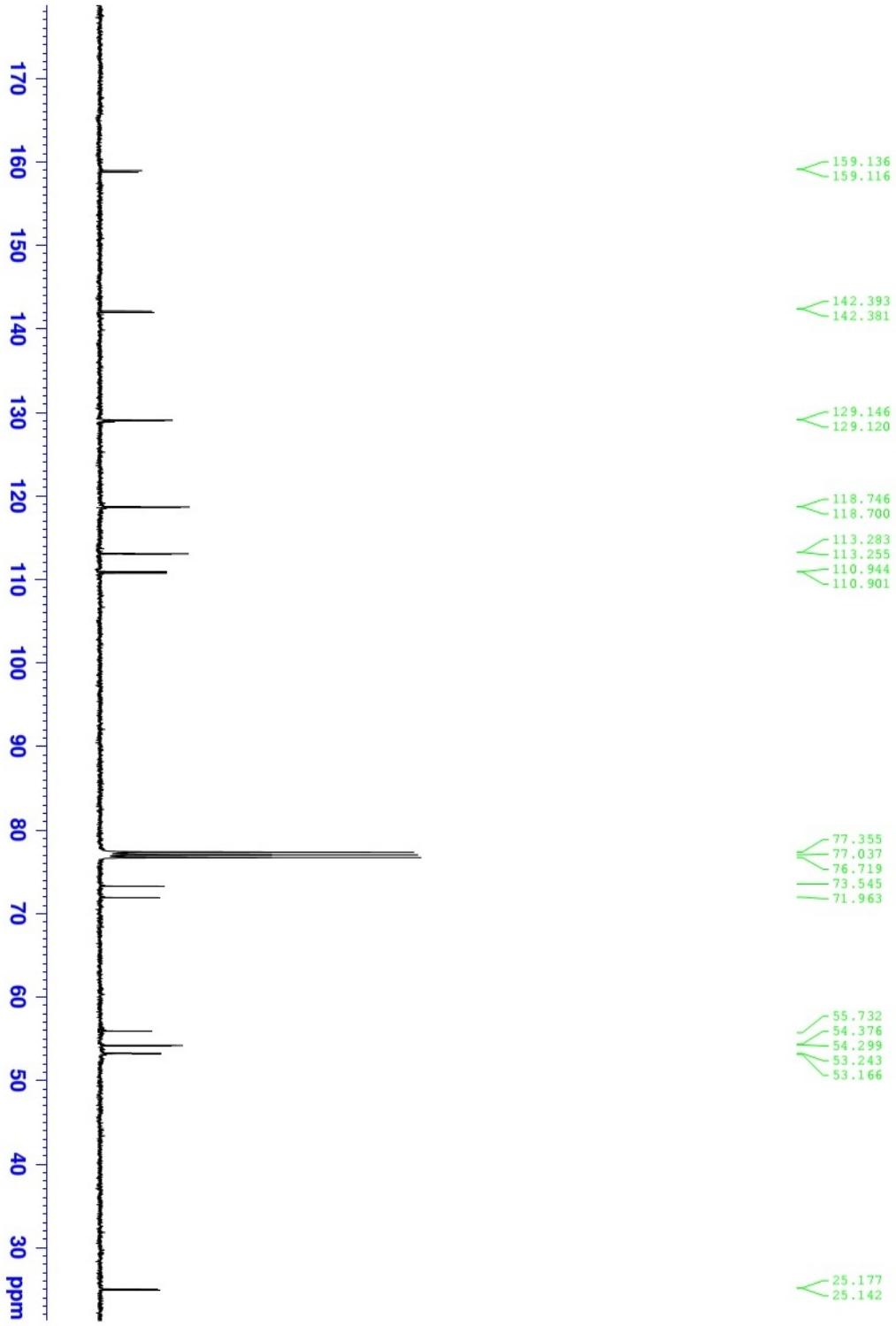

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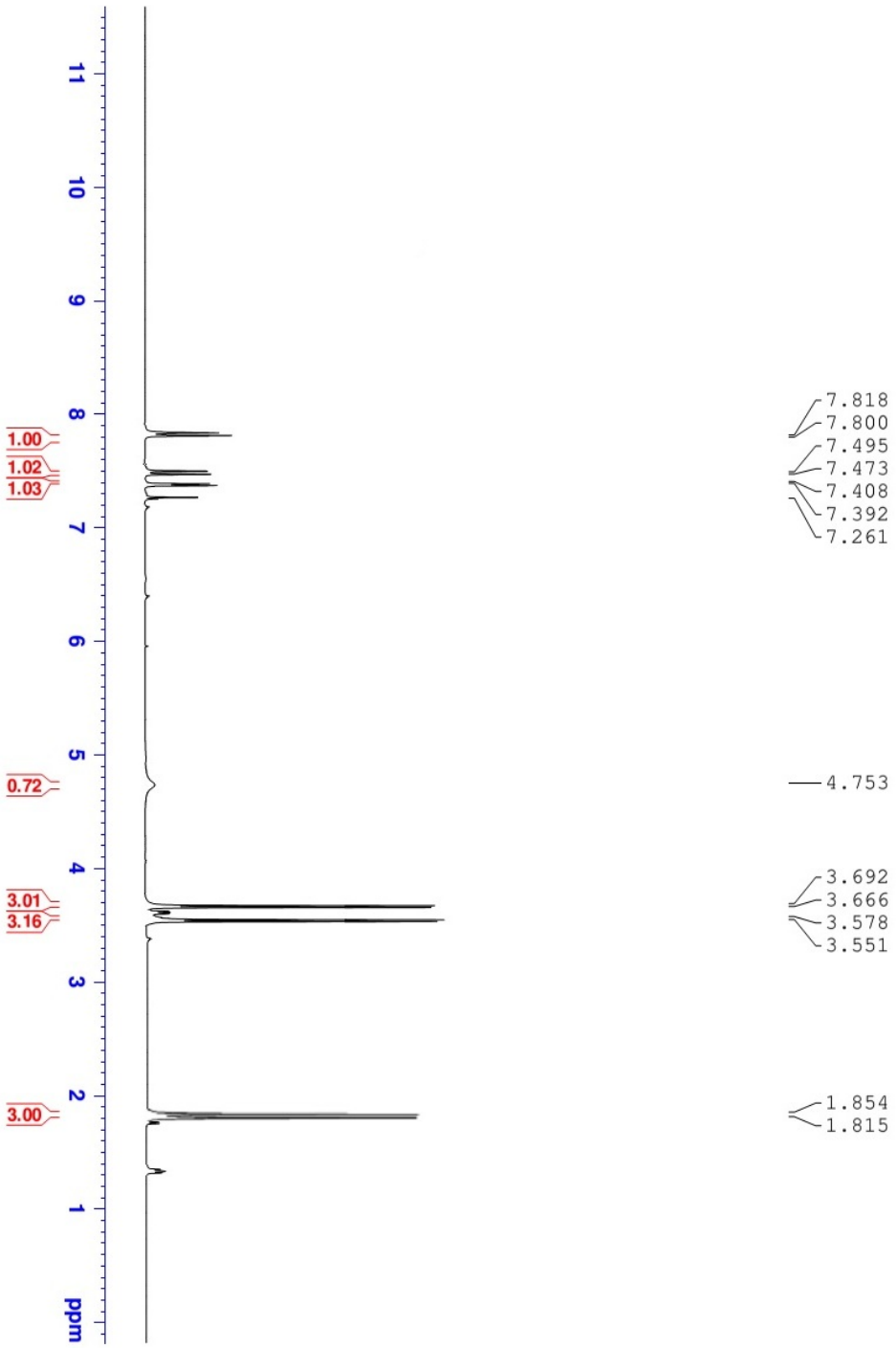

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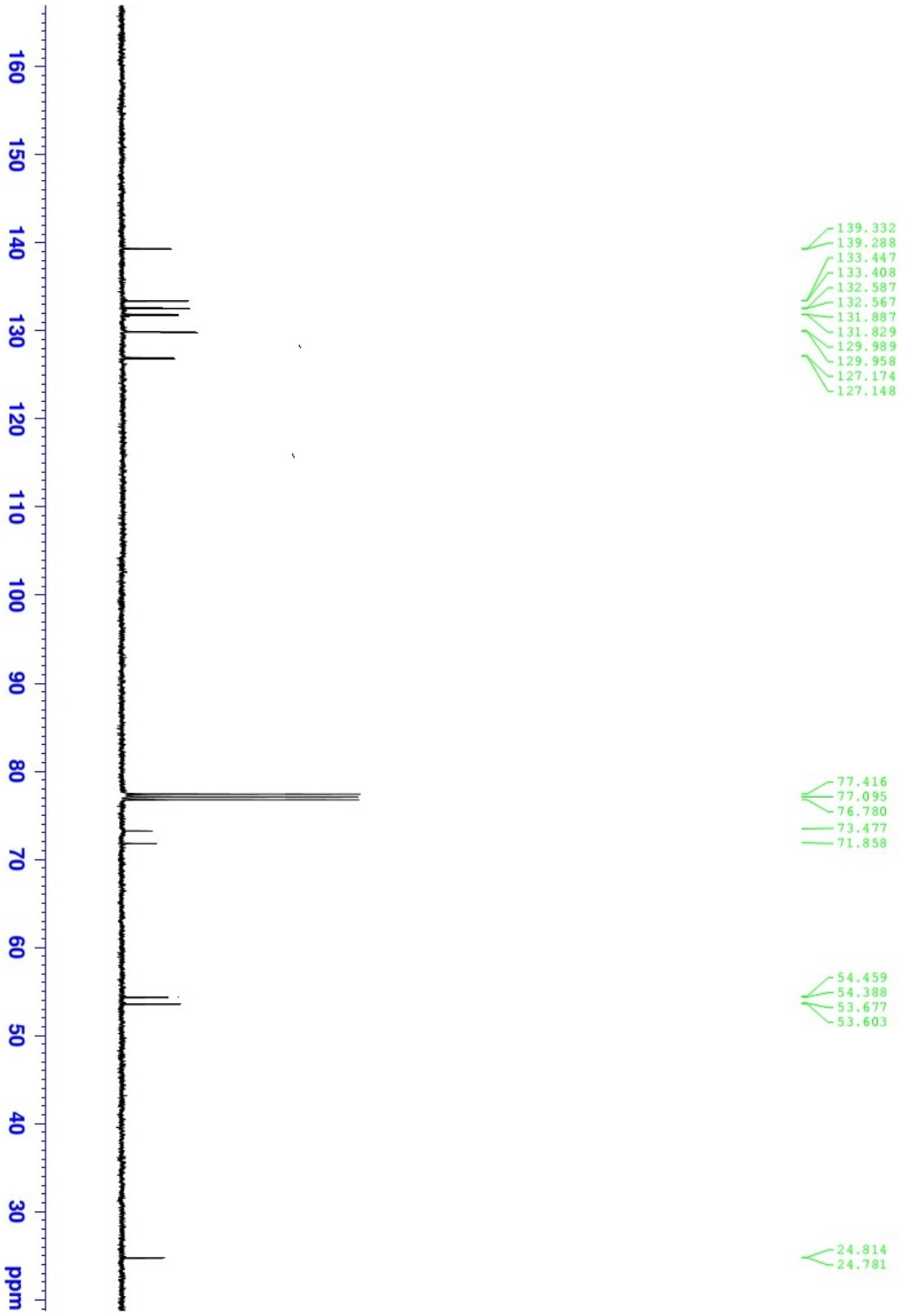

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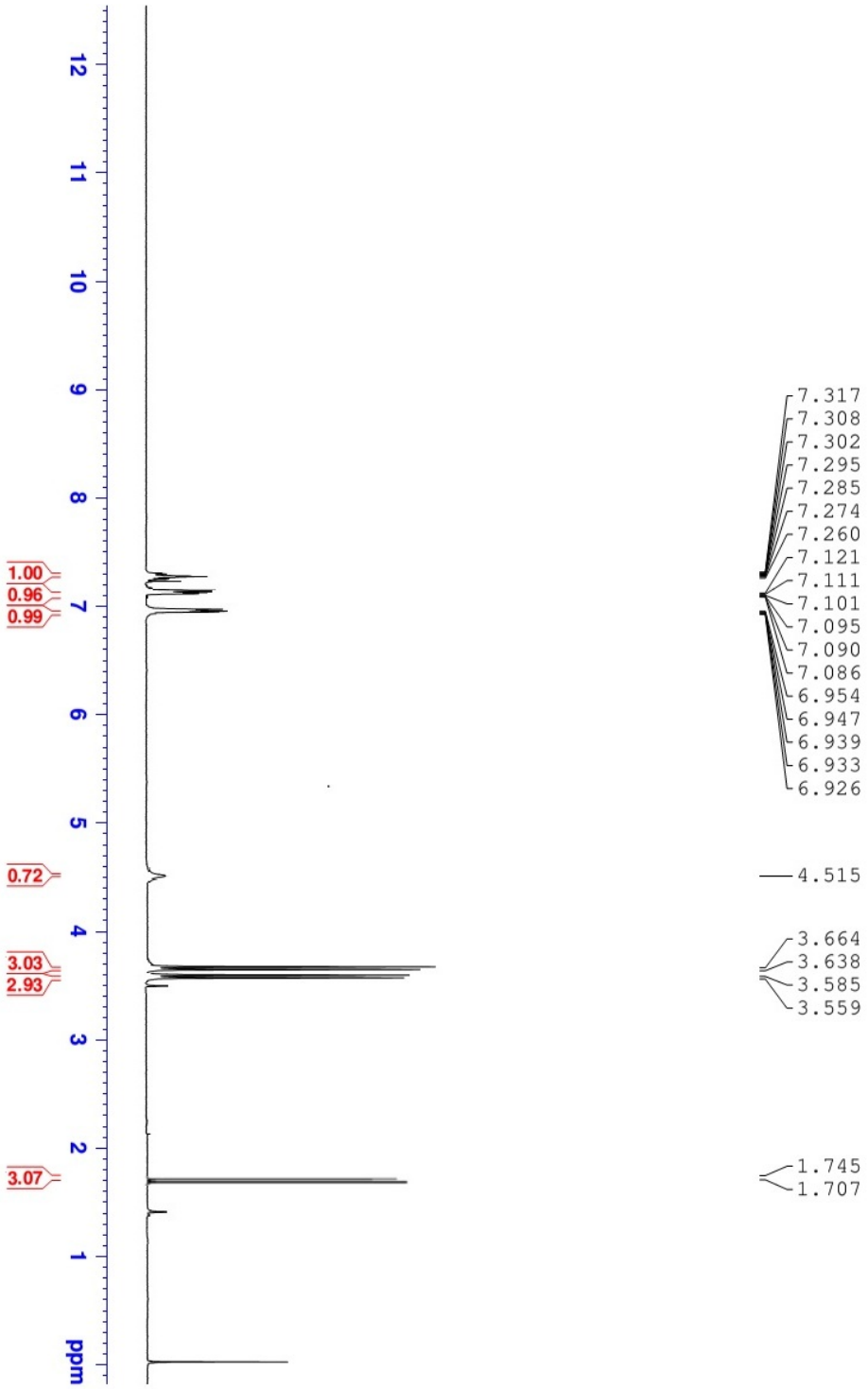

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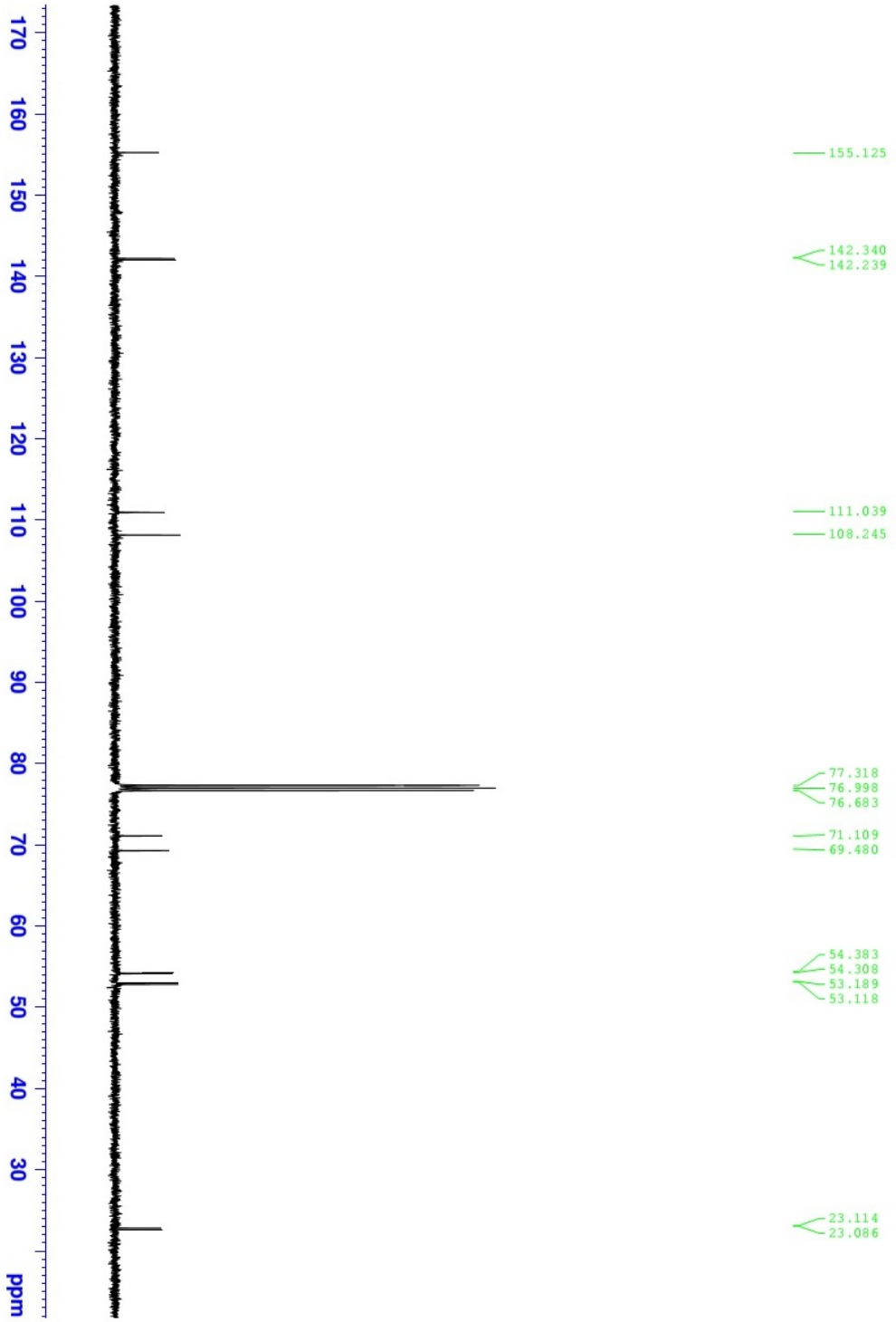

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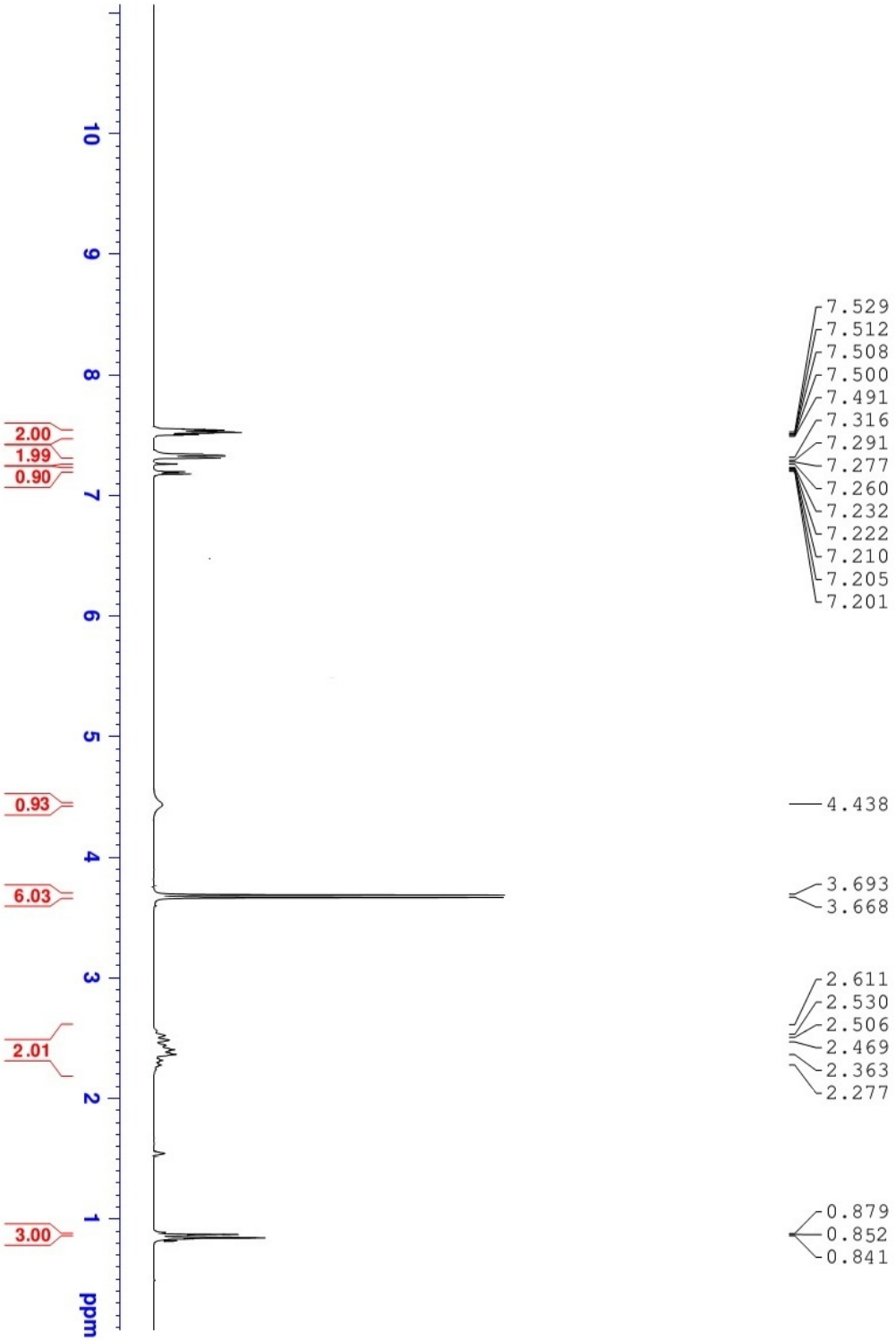

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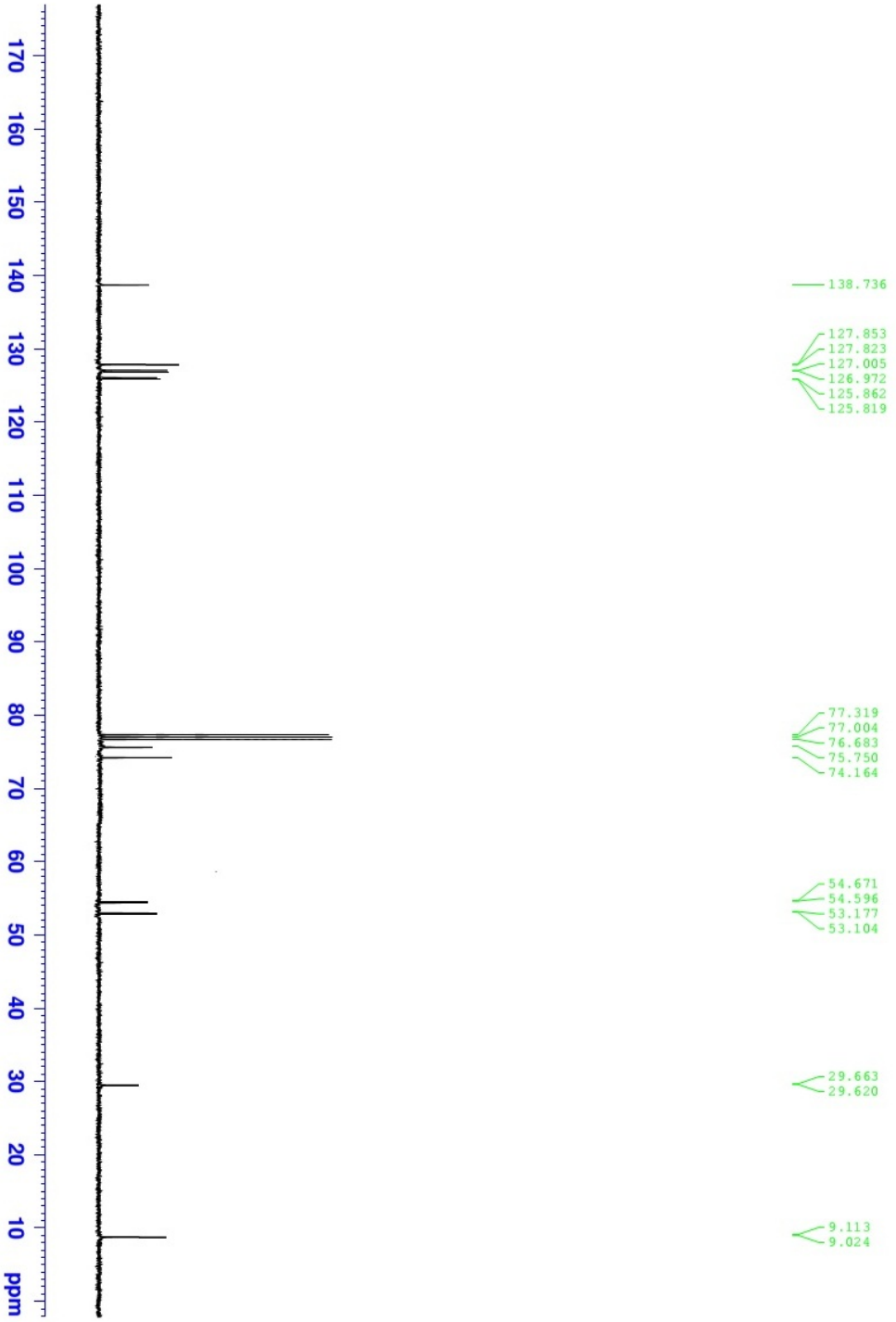

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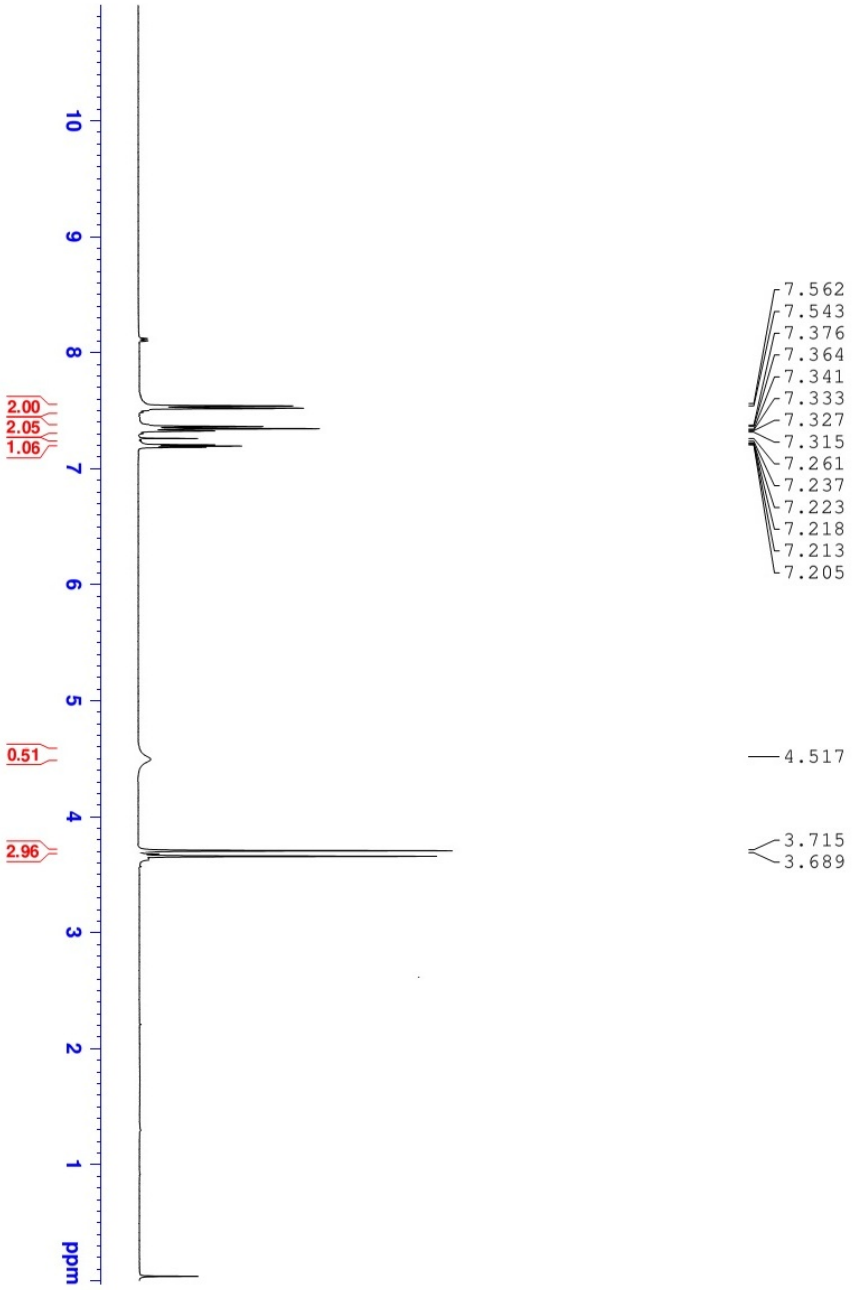

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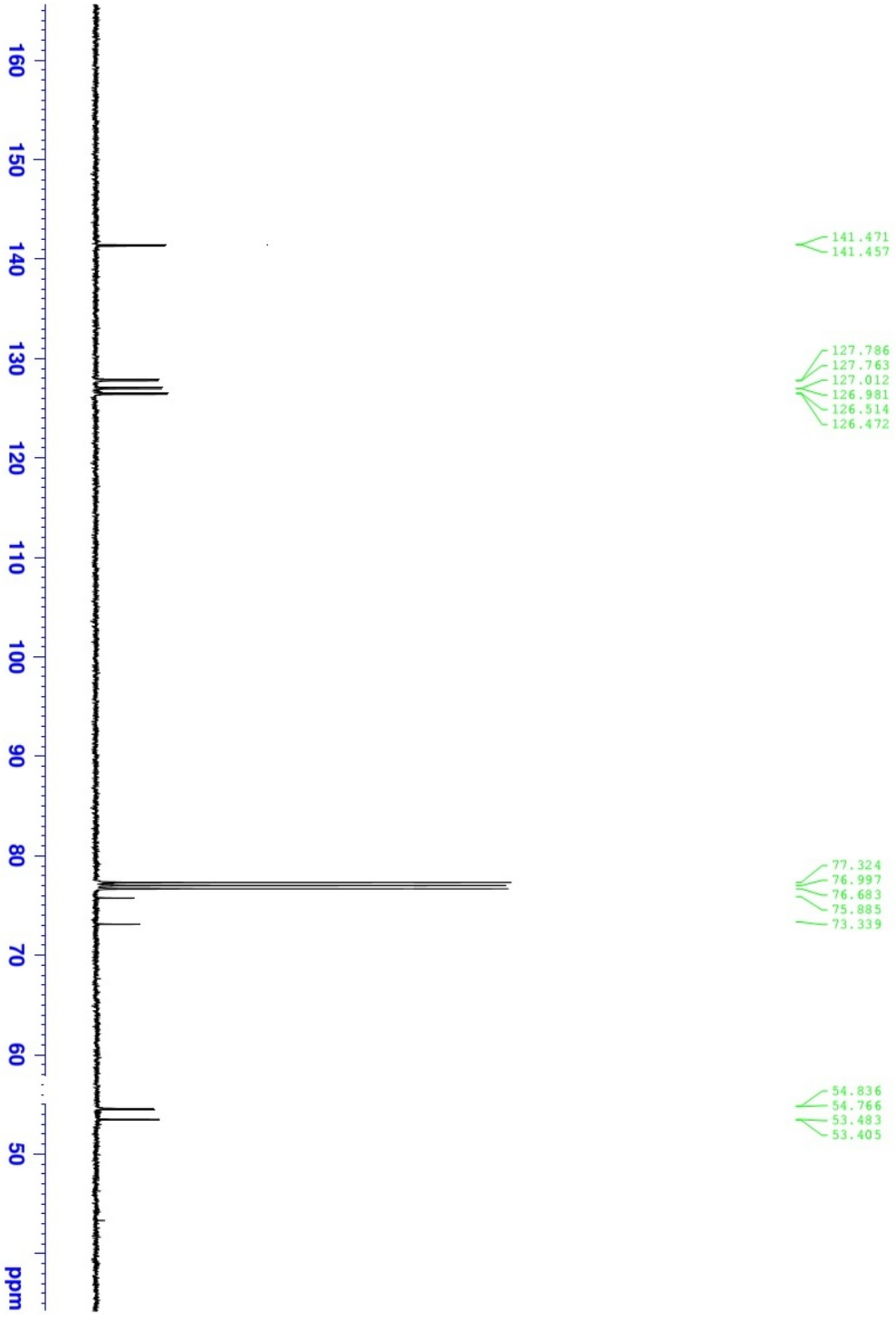

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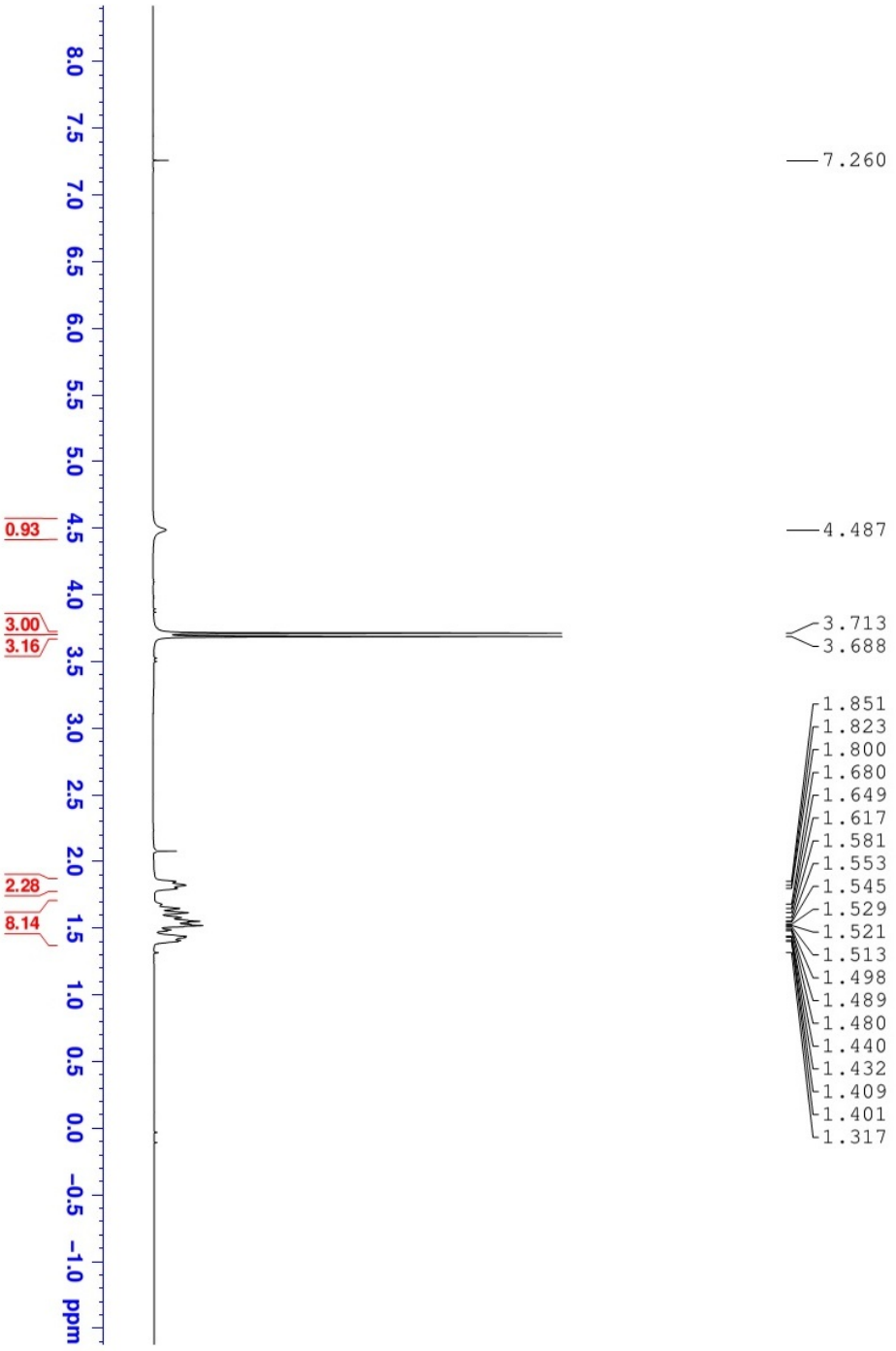

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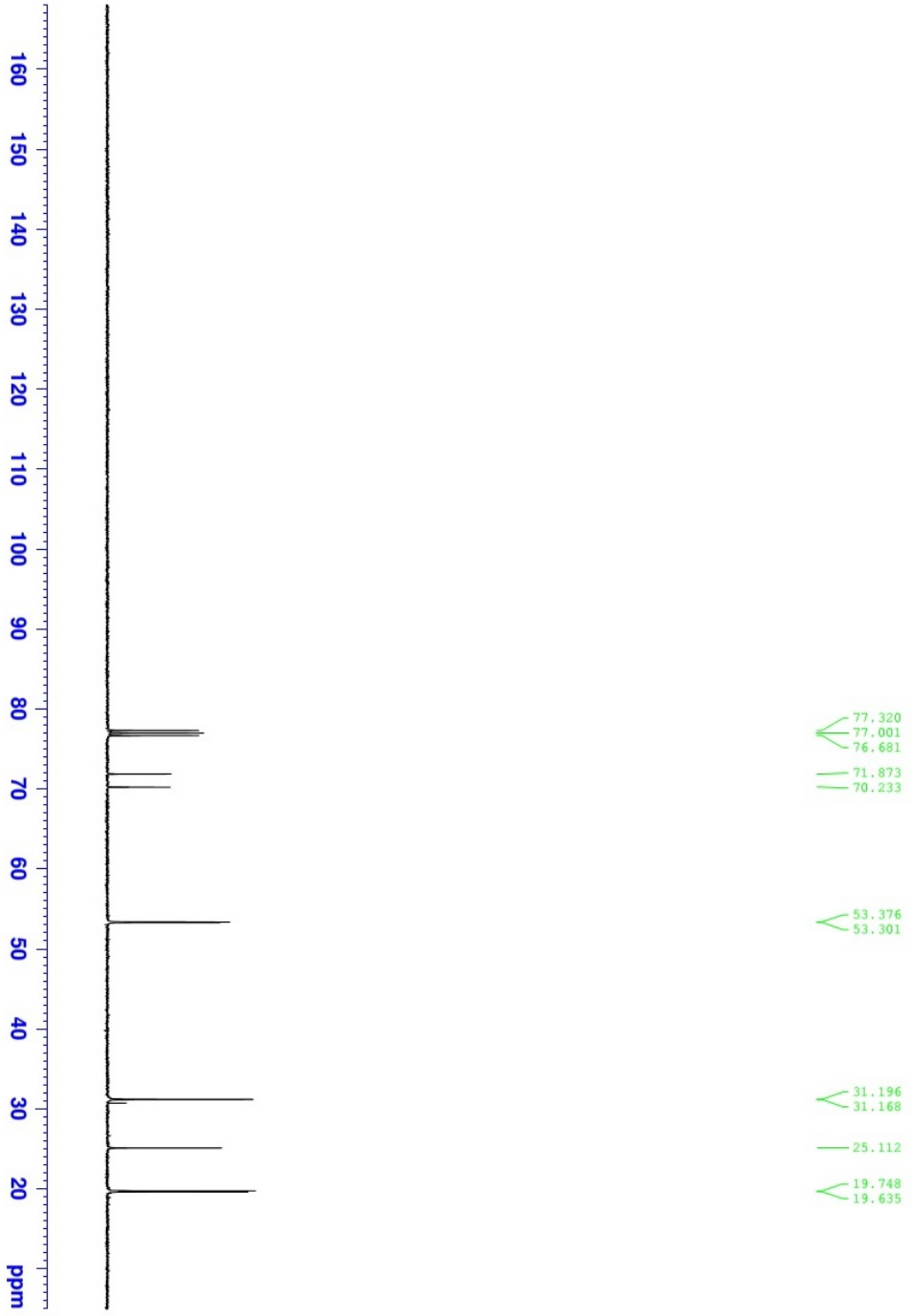

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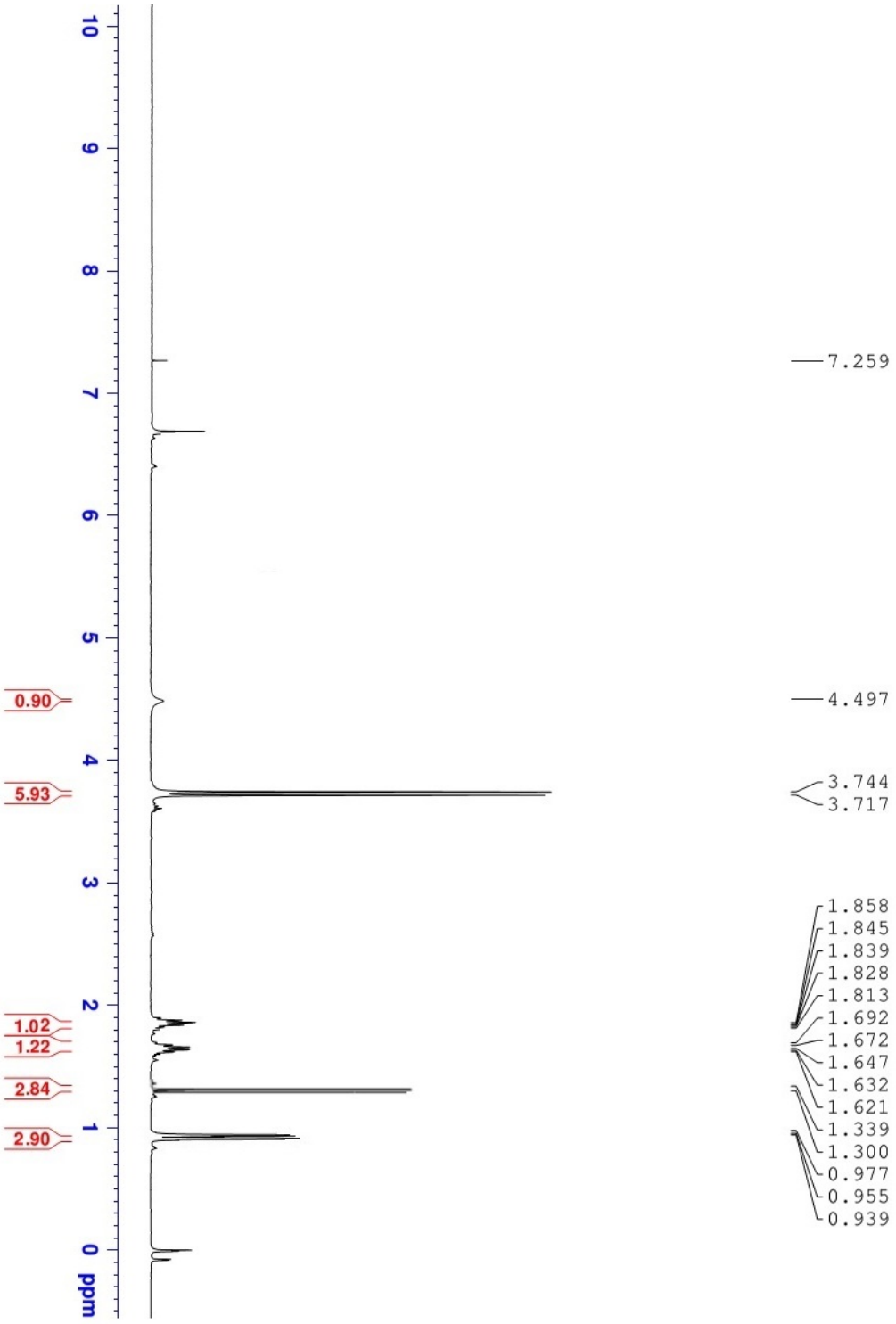

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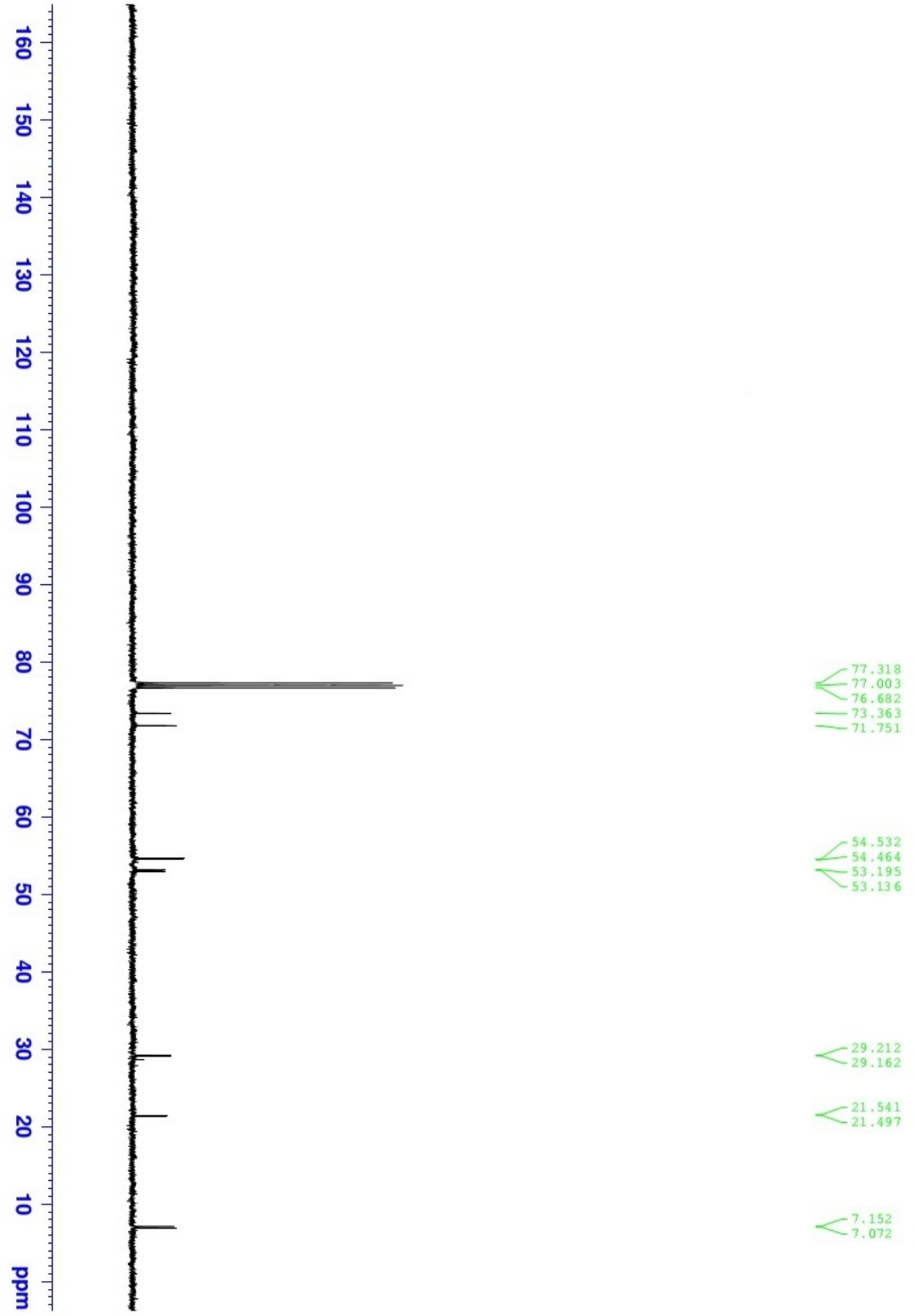

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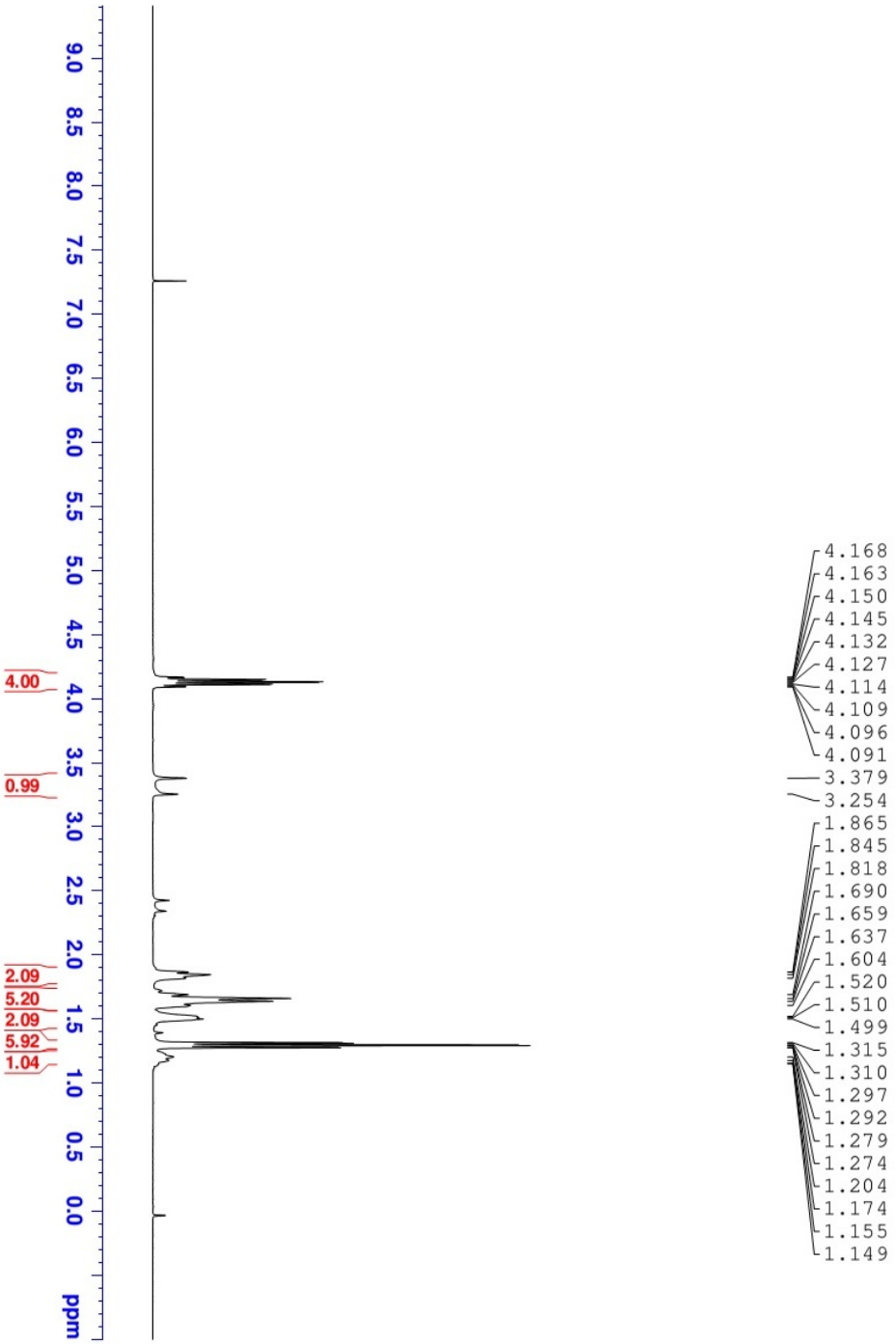

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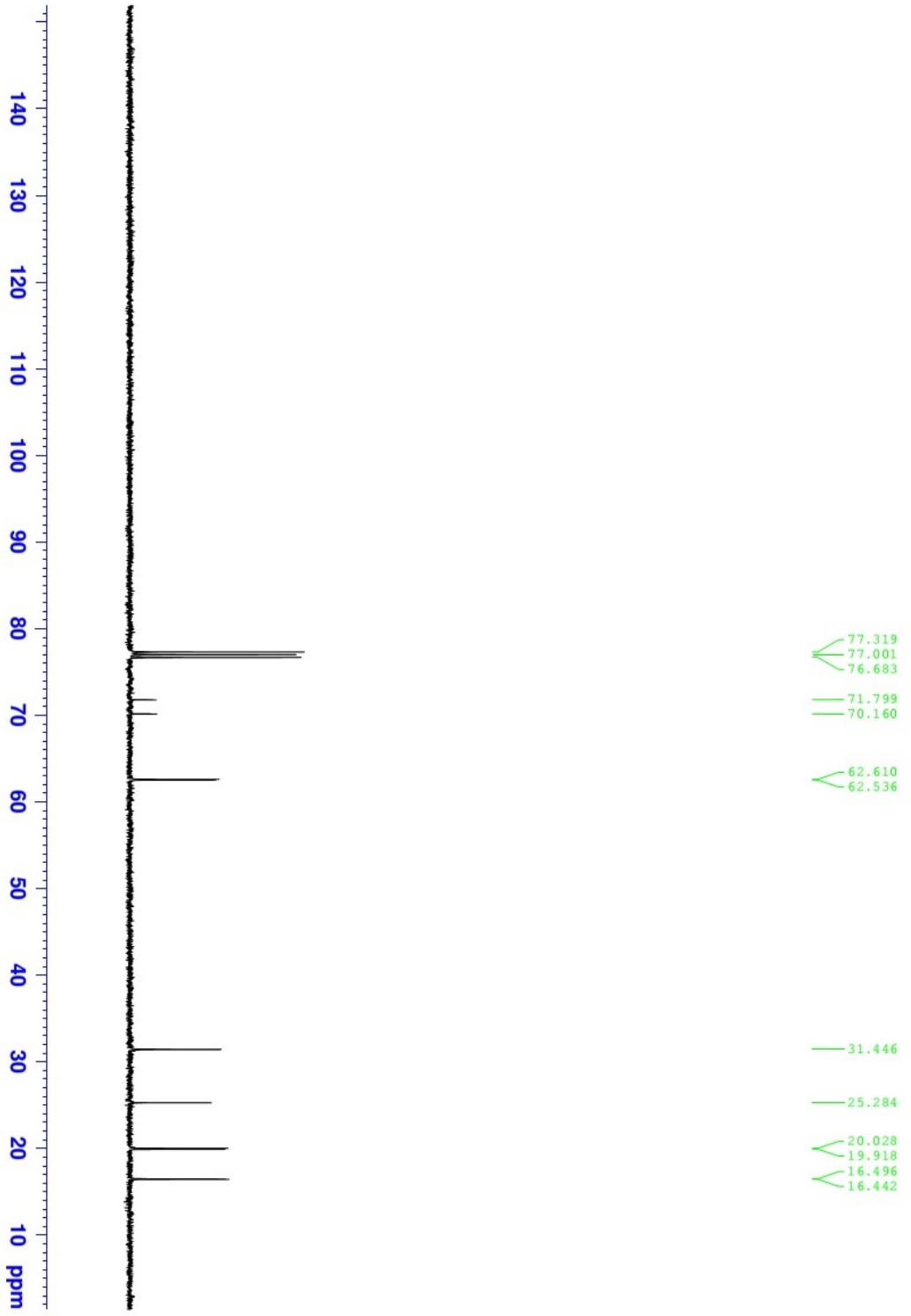

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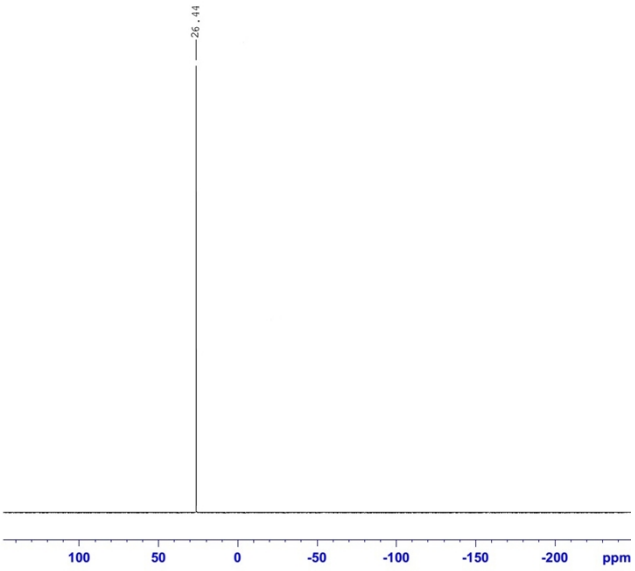

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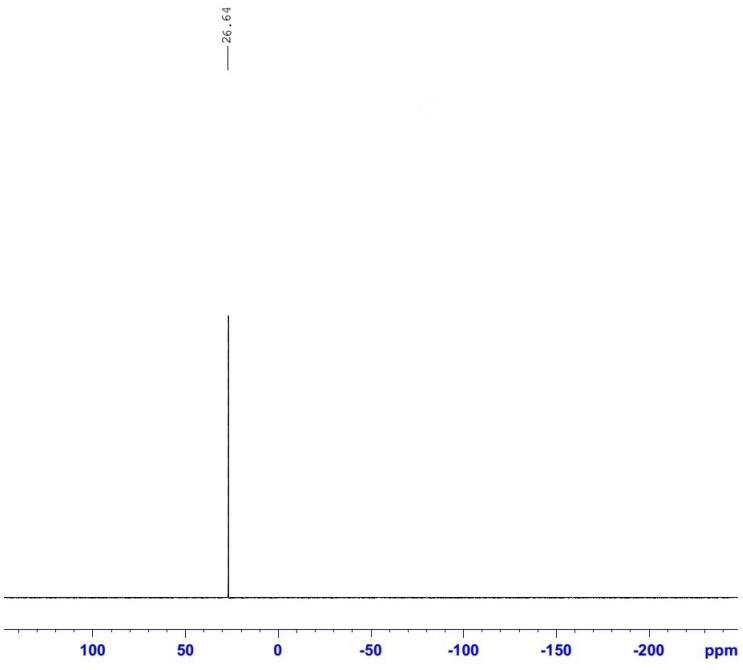

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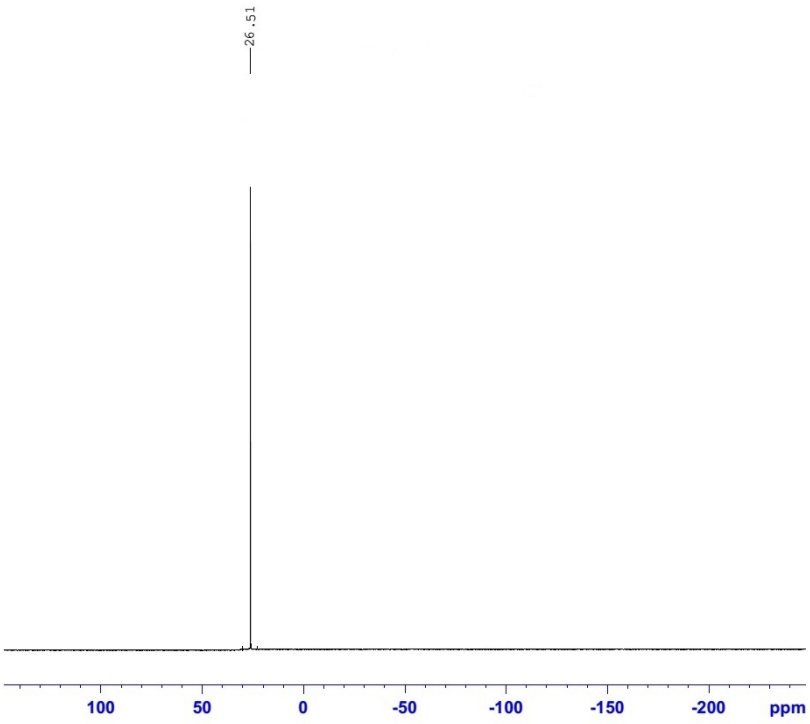

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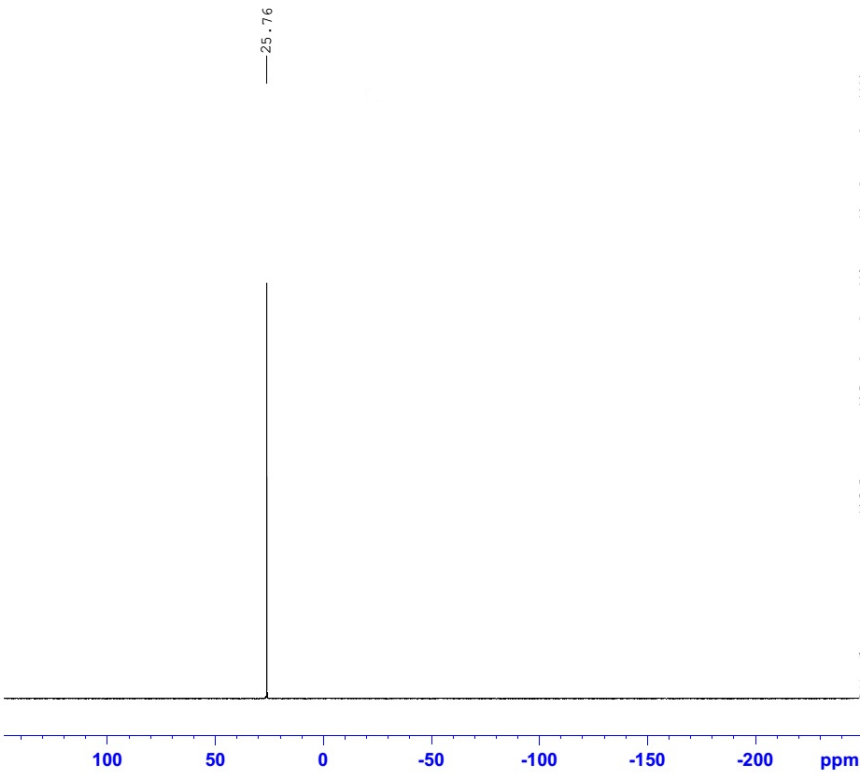

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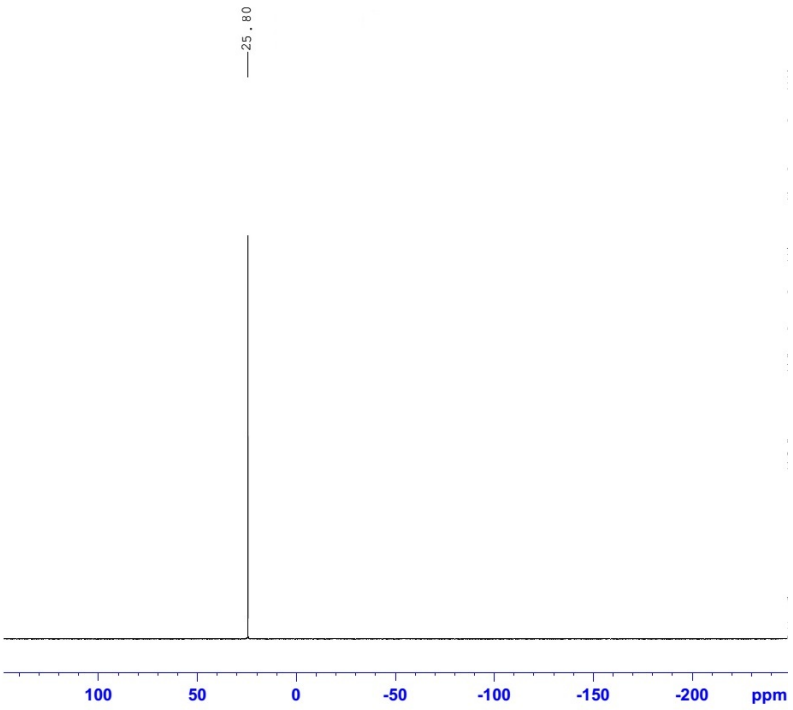

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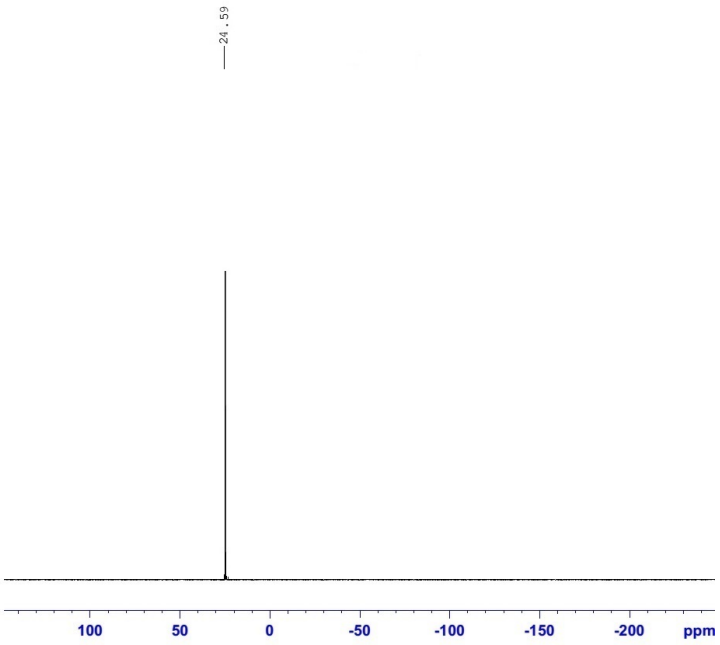

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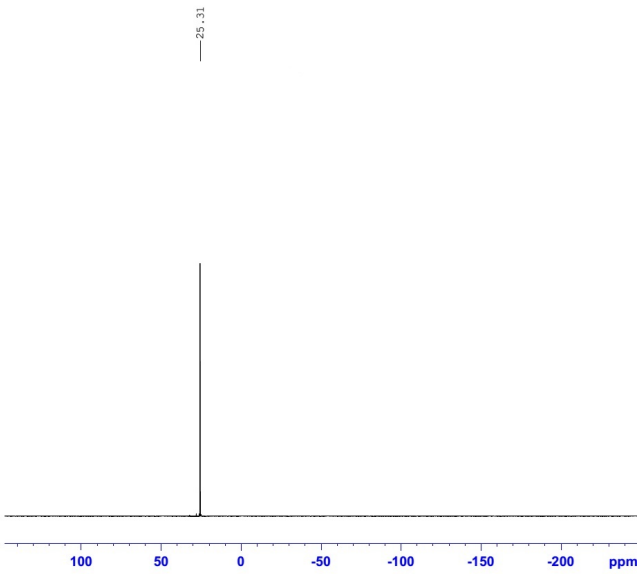

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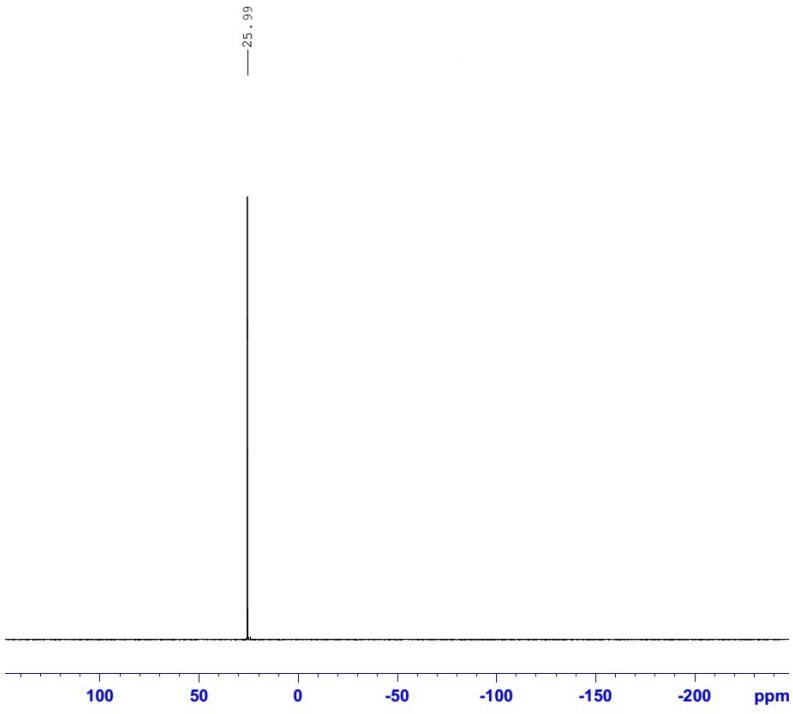

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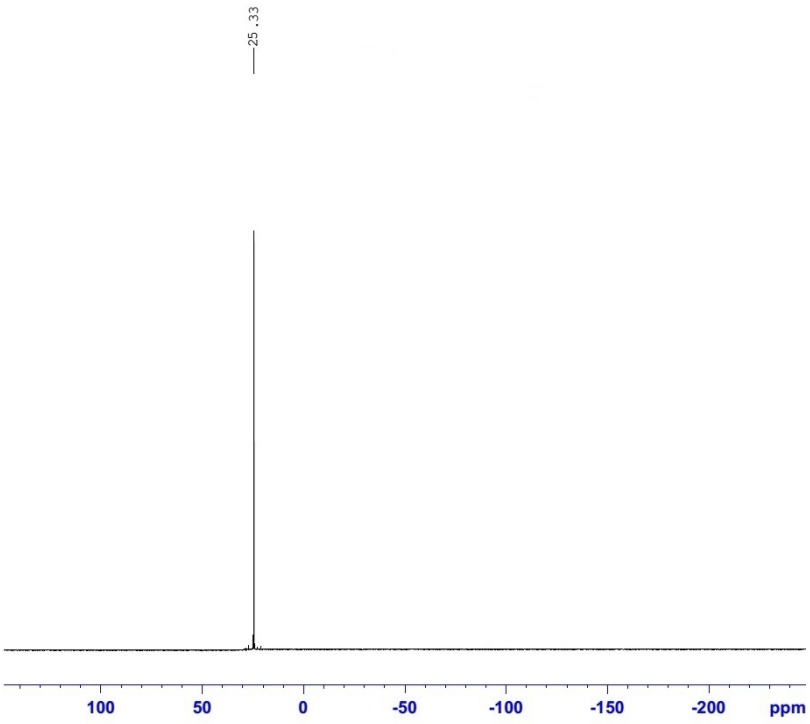

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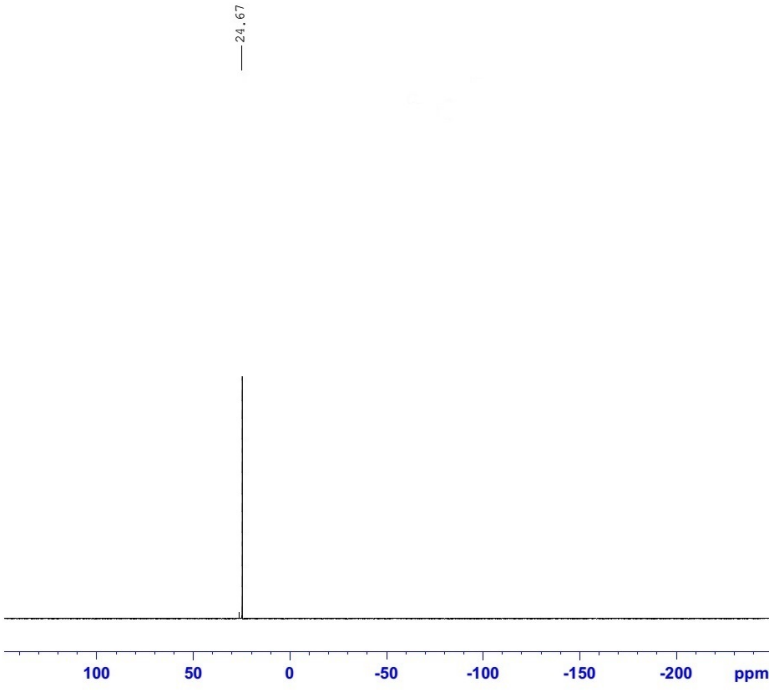

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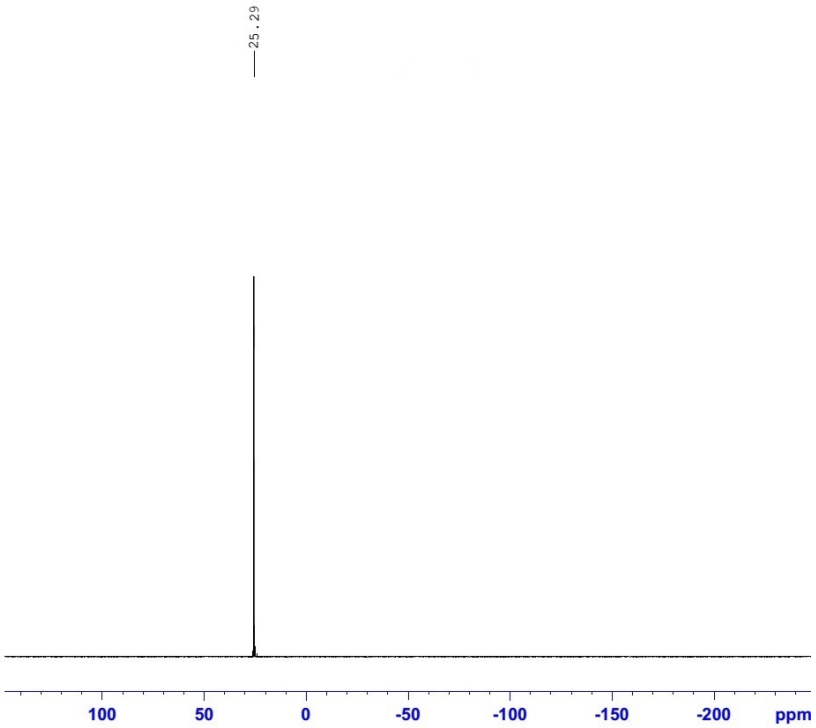

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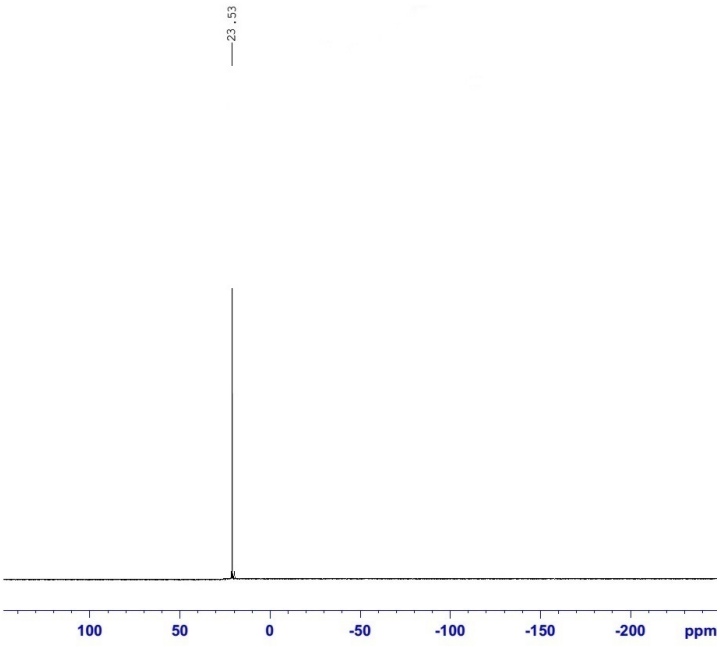

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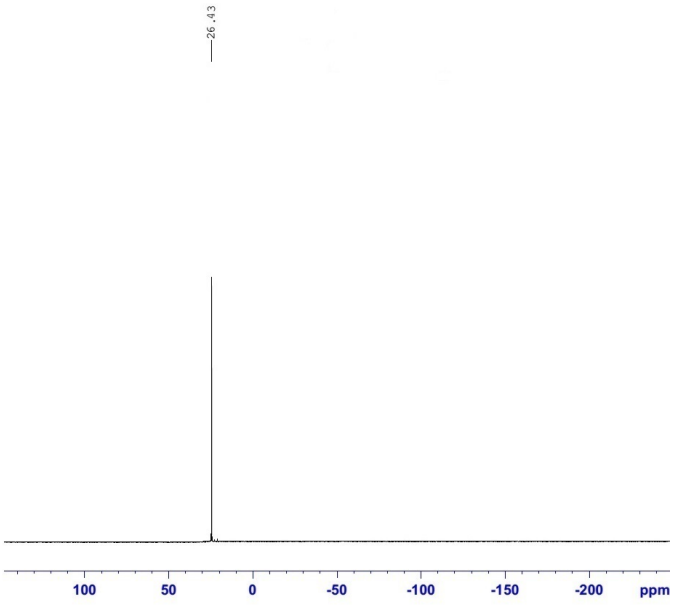

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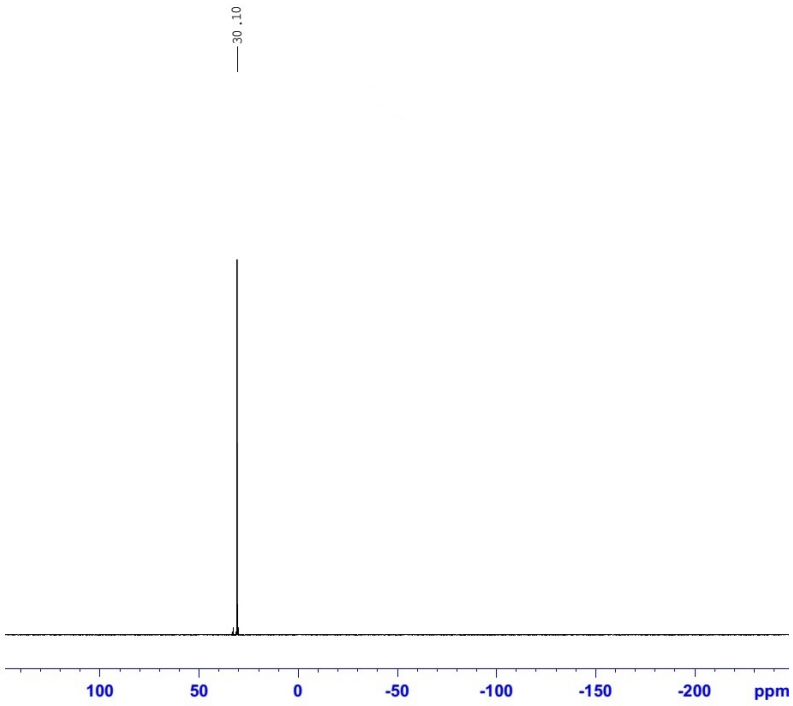

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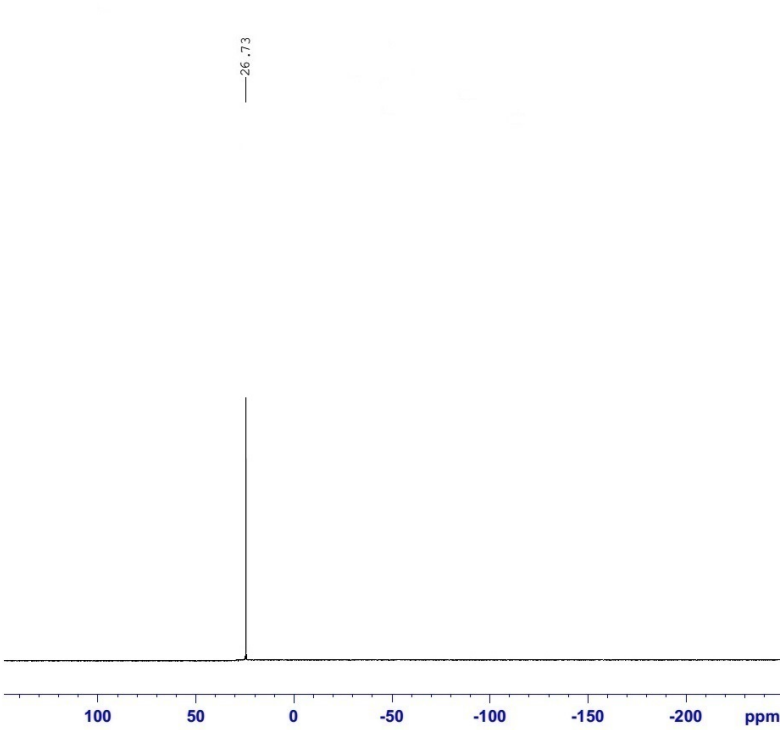

^{31}P NMR of Compound 2o