Supporting information

Palladium-catalyzed Selective C-H Acyloxylation using Sodium Perborate as Oxidant

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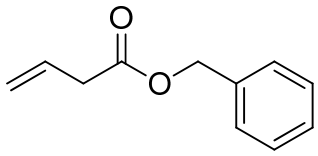
General information

Starting materials **1a,g,h** were prepared analogously to literature procedures.[1] Starting material **1c** was prepared according to literature procedures from 4-Phenyl-1-buten-4-ol.[2] Starting material **1d** was synthesized analogously to literature procedures.[3] All other chemicals were obtained from commercial suppliers and used without further preparations. All reactions were performed under air. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded in $CDCl_3$ (internal standard: 7.26 ppm ^{1}H, 77.16 ppm ^{13}C, external standard α,α,α-trifluorotoluene (-63.73 ppm) ^{19}F) using a 400 MHz instrument. A small peak at 1.5 ppm in some of the ^{1}H NMR spectra is due to residual water in the NMR sample. Stoichiometric NMR studies were performed on a 500 MHz instrument. HRMS data were recorded on an instrument using ESI technique. All column chromatography was performed using silica gel (35-70 microns).

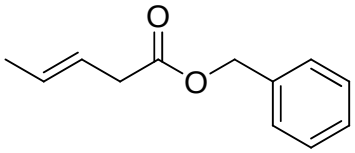
Compound characterization

Benzyl but-3-enoate (1a)



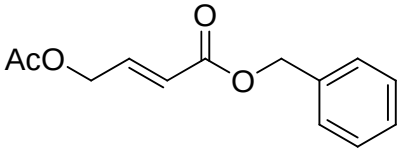
This product was isolated according to general procedure C. Isolated by column chromatography (Et_2O/pentane 1:9) as a colorless oil (82%). The NMR data obtained for **1a** are in agreement with literature values.[4] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.15 (2H, dt, $^{3}J_{HH}$ = 7.0 Hz, $^{4}J_{HH}$ = 1.4 Hz), 5.14 (2H, s), 5.15-5.21 (2H, m), 5.95 (1H, ddt, $^{3}J_{HH}$ = 17.4, 9.8, 7.0 Hz), 7.30-7.40 (5H, m). ^{13}C NMR (100 MHz, $CDCl_3$): δ 39.3, 66.6, 118.9, 128.4, 128.4, 128.7, 130.3, 136.0, 171.5.

Benzyl pent-3-enoate (1g)



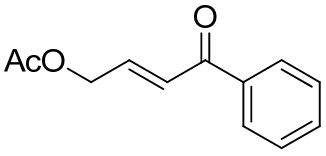
This product was isolated according to general procedure C. Isolated by column chromatography (Et_2O/pentane 1:20) as a colorless oil (76%). The NMR data obtained for **1g** are in agreement with literature values.[4] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.70 (3H, m), 3.07 (2H, m), 5.13 (2H, s), 5.50-5.65 (2H, m), 7.29-7.40 (5H, m). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 18.1, 38.2, 66.5, 122.7, 128.3, 128.7, 129.7, 136.1, 172.2.

(*E*)-Benzyl 4-acetoxybut-2-enoate (4a)



Synthesized according to general method A. Isolated by column chromatography (EtOAc/pentane 1:5) as a colorless oil (57%). The NMR data obtained for **4a** are in agreement with literature values.[5] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.11 (3H, s), 4.74 (2H, dd, $^3J_{HH}$ = 4.5 Hz, $^4J_{HH'}$ = 2.0), 5.19 (2H, s), 6.08 (1H, dt, $^3J_{HH}$ = 15.8 Hz, 2.0 Hz), 6.98 (1H, dt, $^3J_{HH}$ = 15.8 Hz, 4.5 Hz), 7.30-7.39 (5H, m). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 20.8, 62.6, 66.6, 122.0, 128.4, 128.5, 128.7, 135.9, 141. 9, 165.7, 170.4. HRMS (ESI): *m*/*z* calcd. for $[C_{13}H_{14}O_4+Na]^+$ 257.0784, found 257.0775.

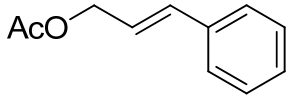
(*E*)-4-(Acetoxy)-1-phenyl-2-buten-1-one (4c)



Synthesized according to general method A. Isolated by column chromatography (Et_2O/pentane 1:2) as a colorless oil (56%). The NMR data obtained for **4c** are in agreement with literature values.[6] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.10 (3H, s), 4.85 (2H,

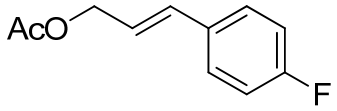
dd, $^{3}J_{HH}$ = 4.4 Hz, $^{4}J_{HH'}$ = 1.5), 7.00 (1H, dt, $^{3}J_{HH}$ = 15.5 Hz, 4.4 Hz), 7.10 (1H, dt, $^{3}J_{HH}$ = 15.5 Hz, 1.5 Hz), 7.48 (2H, tm, $^{3}J_{HH}$ = 7.8 Hz), 7.56 (1H, tt, $^{3}J_{HH}$ = 7.4 Hz, $^{4}J_{HH}$ = 2.0 Hz), 7.91-7.97 (2H, m). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 20.9, 63.3, 126.2, 128.8, 128.8, 133.2, 137.5, 141.2, 170.53. HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{13}O_3+Na]^+$ 227.0679, found: 227.0668.

(*E*)-3-Phenyl-2-propenyl acetate (4e)



Synthesized according to general method A. Isolated by column chromatography (Et_2O/pentane 1:9) as a colorless oil (57%). The NMR data obtained for **4e** are in agreement with literature values.[7] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.10 (3H, s), 4.71 (2H, dd, $^{3}J_{HH}$ = 6.5 Hz, $^{4}J_{HH'}$ = 1.3), 6.28 (1H, dt, $^{3}J_{HH}$ = 15.9 Hz, 6.5 Hz), 6.65 (1H, d, $^{3}J_{HH}$ = 15.9 Hz), 7.26 (1H, tt, $^{3}J_{HH}$ = 7.3 Hz, $^{4}J_{HH}$ = 1.4 Hz), 7.32 (2H, tm, $^{3}J_{HH}$ = 7.3 Hz), 7.37-7.41 (2H, m). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.1, 65.2, 123.3, 126.7, 128.2, 128.7, 134.4, 136.3, 171.0. HRMS (ESI): *m*/*z* calcd. for $[C_{11}H_{12}O_2+Na]^+$ 199.0735, found: 199.0729.

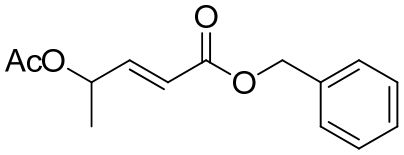
(*E*)-3-(-4-Fluoro)phenyl-2-propenyl acetate (4f)



Synthesized according to general method A. Isolated by column chromatography (Et_2O/pentane 1:9) as a colorless oil (59%). The NMR data obtained for **4f** are in agreement with literature values.[7] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.10 (3H, s), 4.71 (2H, dd, $^{3}J_{HH}$ = 6.4 Hz, $^{4}J_{HH'}$ = 1.2), 6.20 (1H, dt, $^{3}J_{HH}$ = 15.9 Hz, 6.4 Hz), 6.61 (1H, d, $^{3}J_{HH}$ = 15.9), 7.01(2H, dd, $^{3}J_{HH}$ = 8.7 Hz, $^{3}J_{HF}$ = 8.7 Hz), 7.36 (2H, dd, $^{3}J_{HH}$ = 8.7, $^{4}J_{HF}$ = 5.3 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.1, 65.1, 115.7 (d, $^{2}J_{CF}$ = 22.0 Hz), 123.1, 128.3 (d, $^{3}J_{CF}$ = 8.0 Hz), 132.5 (d, $^{4}J_{CF}$ = 3.2 Hz), 133.2, 162.8 (d, $^{1}J_{CF}$ = 262.7 Hz), 171.0. ^{19}F

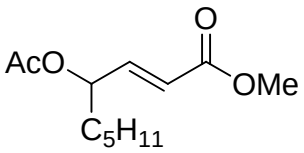
NMR (300 MHz, $CDCl_3$): δ -113.68 (s). HRMS (ESI): *m*/*z* calcd. for $[C_{11}H_{11}FO_2+Na]^+$ 217.0635, found: 217.0638.

(*E*)-Benzyl 4-acetoxypent-2-enoate (4g)



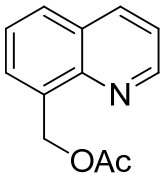
Synthesized according to general method A. Isolated by column chromatography (Et_2O/pentane 1:3) as a colorless oil (71%). The NMR data obtained for **4g** are in agreement with literature values.[5] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.35 (3H, d, $^3J_{HH}$ = 6.6 Hz), 2.07 (3H, s), 5.18 (2H, s), 5.48 (1H, dqd, $^3J_{HH}$ = 6.8 Hz, 4.9 Hz, $^4J_{HH}$ =1.6 Hz), 6.00 (1H, dd, $^3J_{HH}$ = 15.8 Hz, $^4J_{HH}$ =1.6 Hz), 6.90 (1H, dd, $^3J_{HH}$ = 15.8 Hz, 4.9 Hz), 7.30-7.39 (5H, m). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 19.7, 21.2, 66.6, 69.0, 120.8, 128.5, 128.7, 135.9, 147.02, 166.0, 170.1. HRMS (ESI): *m*/*z* calcd. for $[C_{14}H_{16}O_4+Na]^+$ 271.0941, found: 271.0935.

(*E*)-Methyl 4-acetoxynon-2-enoate (4i)



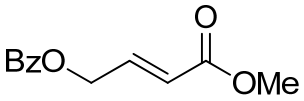
Synthesized according to general method A. Isolated by column chromatography (Et_2O/pentane 1:5) as a colorless oil (62%). The NMR data obtained for **4i** are in agreement with literature values.[8] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.88 (3H, t, $^3J_{HH}$ = 6.77 Hz), 1.20-1.38 (6H, m, $(CH_2)_3$), 1.60-1.70 (2H, m), 2.09 (s, 3H), 3.74 (s, 3H), 5.38 (1H, dtd, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 6.0 Hz, $^4J_{HH}$ = 1.6 Hz), 5.93 (1H, dd, $^3J_{HH}$ = 15.7 Hz, $^4J_{HH}$ = 1.5 Hz), 6.85 (1H, dd, $^3J_{HH}$ = 15.7 Hz, 5.38, Hz). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.9, 21.0, 22.4, 24.6, 31.5, 33.7, 51.7, 72.5, 121.1, 145.8, 166.5, 170.1. HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{20}O_4+Na]^+$ 251.1254, found: 251.1264.

Quinolin-8-ylmethyl acetate (4k)



Synthesized according to general method A except acetic acid (0.5 ml) was used as solvent and the reaction was run for 22 hours. Isolated by column chromatography (Et_2O/pentane 1:10) as a colorless oil (52%). The NMR data obtained for **4k** are in agreement with literature values.[9] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.16 (3H, s), 5.85 (2H, s), 7.44 (1H, dd, $^3J_{HH'}$ = 4.27, 8.36), 7.54 (1H, t, $^3J_{HH'}$ = 7.17), 7.77 (1H, d, $^3J_{HH'}$ = 7.17), 7.80 (1H, d, $^3J_{HH'}$ = 8.36), 8.17 (1H, dd, $^3J_{HH'}$ = 1.74, 8.36), 8.96 (1H, dd, $^3J_{HH'}$ = 1.74, 4.20). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.3, 62.9, 121.4, 126.3, 128.28, 128.31, 128.9, 134.4, 136.3, 146.3, 150.1, 171.2. HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{11}NO_2+Na]^+$ 224.0682; found: 224.0675.

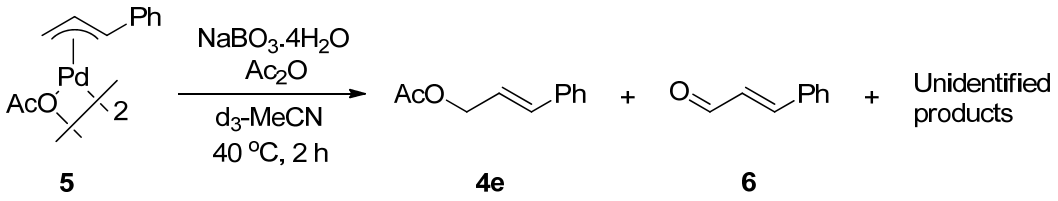
(*E*)-Methyl 4-benzoyloxybut-2-enoate (4o)



Synthesized according to general method B. Isolated by column chromatography (Et_2O/pentane 1:4) as a colorless oil (62%). The NMR data obtained for **4o** are in agreement with literature values.[8] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.76 (3H, s), 4.99 (2H, dd, $^3J_{HH}$ = 4.6 Hz, $^4J_{HH}$ = 2.0 Hz), 6.14 (1H, dt, $^3J_{HH}$ = 15.8 Hz, $^4J_{HH}$ = 2.0 Hz*),* 7.07 (1H, dt, $^3J_{HH}$ = 15.8 Hz, 4.6 Hz), 7.46 (2H, tm, $^3J_{HH}$ = 7.6 Hz), 7.59 (1H, tm, $^3J_{HH}$ = 7.6 Hz), 8.07 (2H, dm, $^3J_{HH}$ = 7.6 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 51.9, 63.1, 121.9, 128.6, 129.6, 129.8, 133.5, 141. 7, 166.0, 166.4. HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{12}O_4+Na]^+$ 243.0628, found 243.0624.

Stoichiometric studies

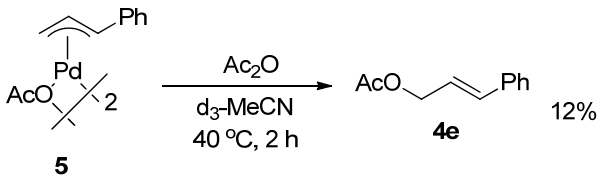
Oxidation of 5 with SPB



5 **4e** **6**

A mixture of complex **5** (8.0 mg, 0.014 mmol), sodium perborate (43.8 mg, 0.28 mmol, 10 equiv. wrt Pd), and acetic anhydride (57.7 mg, 0.57 mmol, 20 equiv. wrt Pd) in d_3-MeCN (0.5 ml), was stirred at 40 °C for 2 hours. ^{1}H NMR analysis of the crude mixture revealed complete consumption of **5**, and the formation of a complex mixture of compounds which included **4e**, and **6** which formed in a 0.6:1 ratio.

Reaction between 5 and Ac_2O without SPB



5 **4e**

A mixture of complex **5** (8.0 mg, 0.028 mmol wrt Pd and acetic anhydride (58 mg, 0.57 mmol, 20 equiv. per Pd) in d_3-MeCN (0.5 ml), was stirred at 40 °C for 2 h min/hours. ^{1}H NMR analysis of the crude reaction mixture revealed a 12% conversion of complex **5** to product **4e**, with the remainder unreacted.

References

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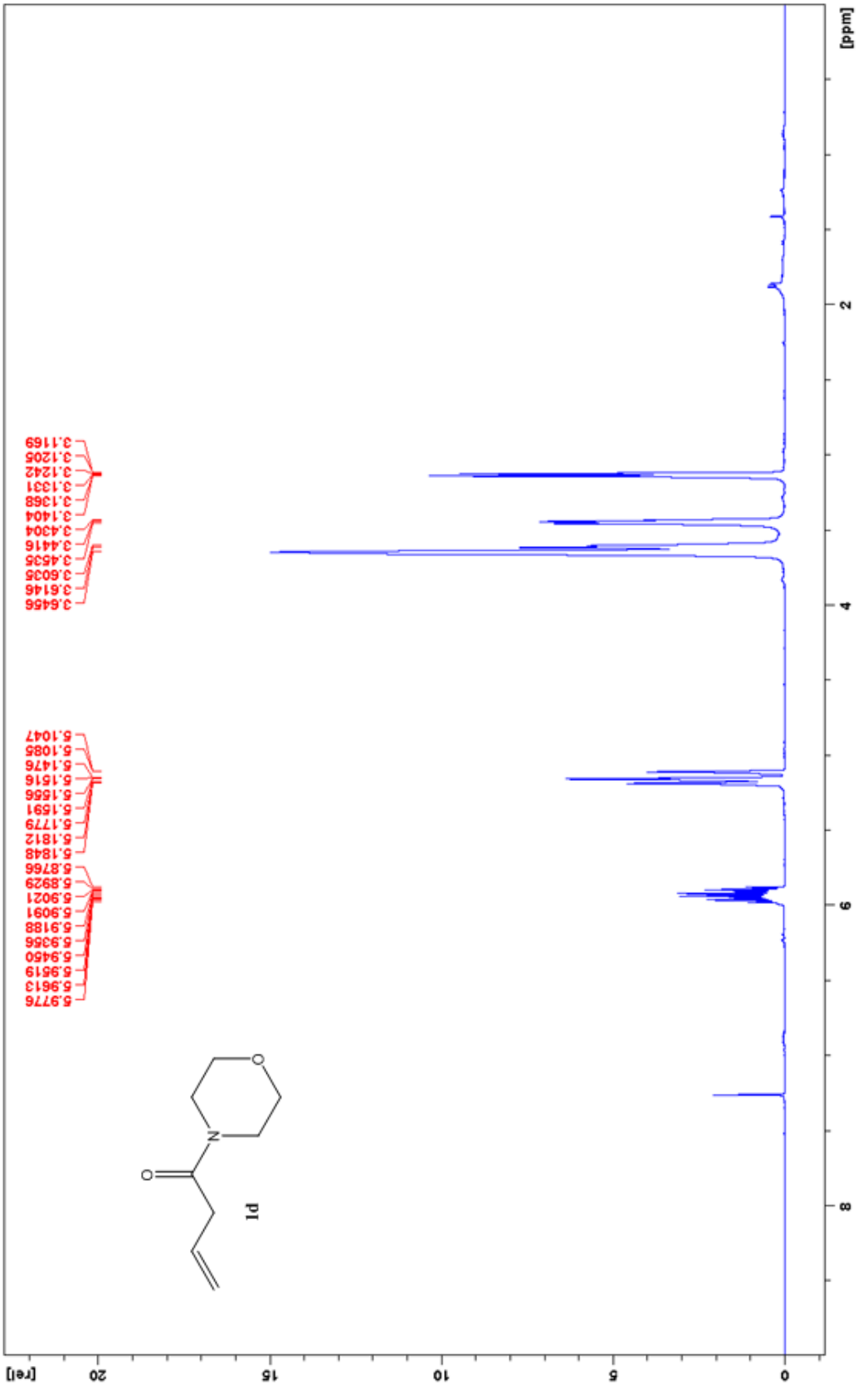
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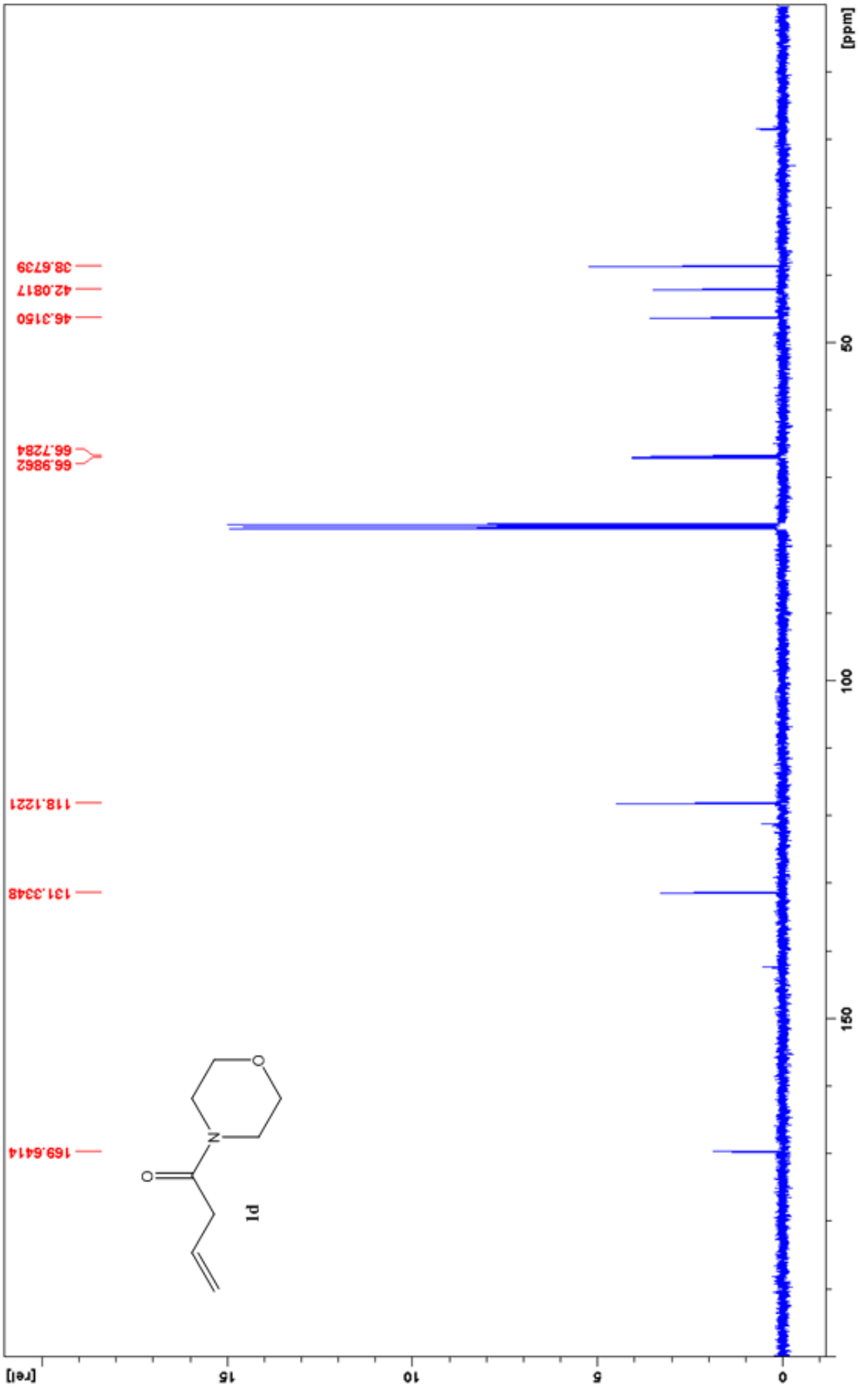
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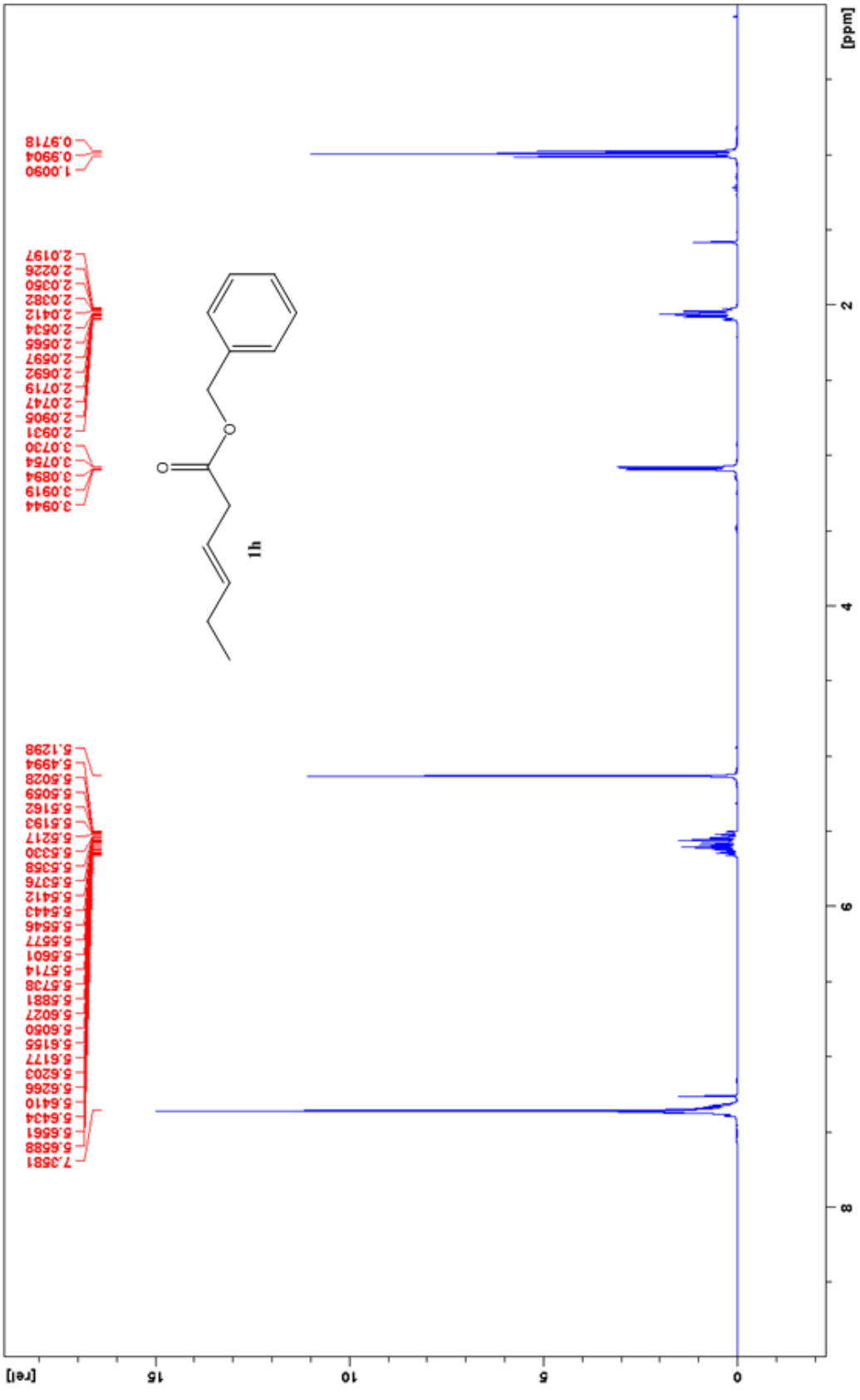
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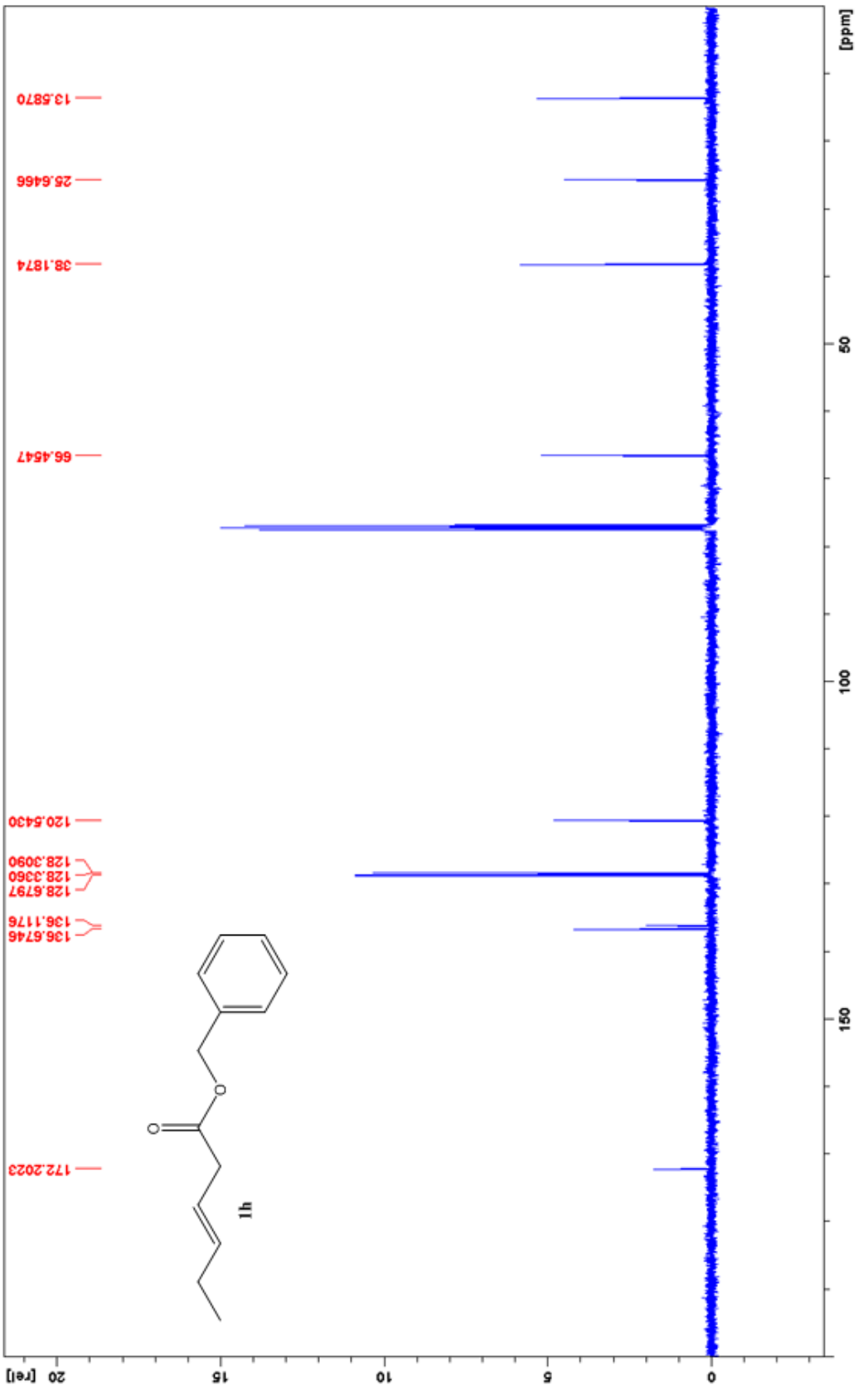
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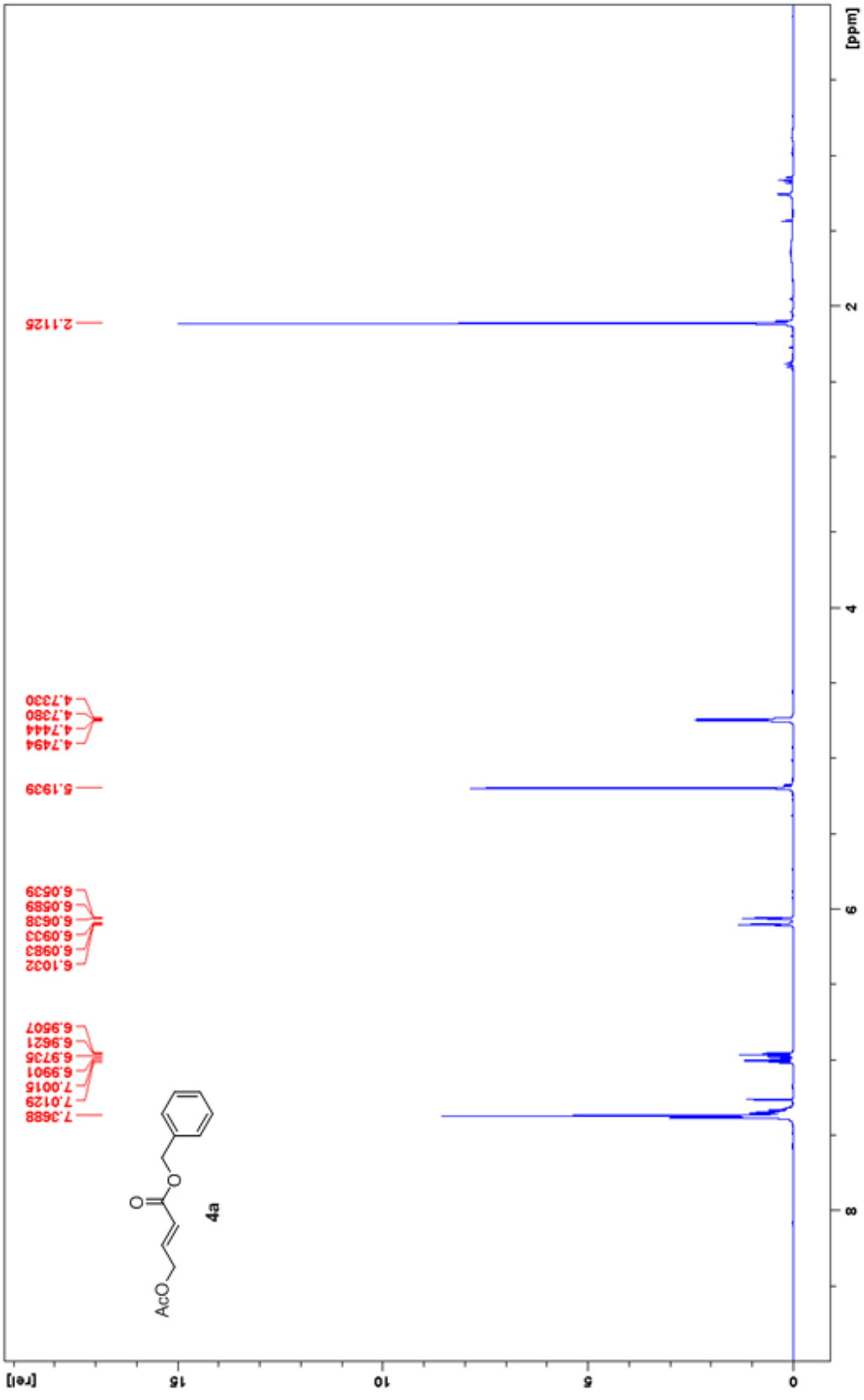
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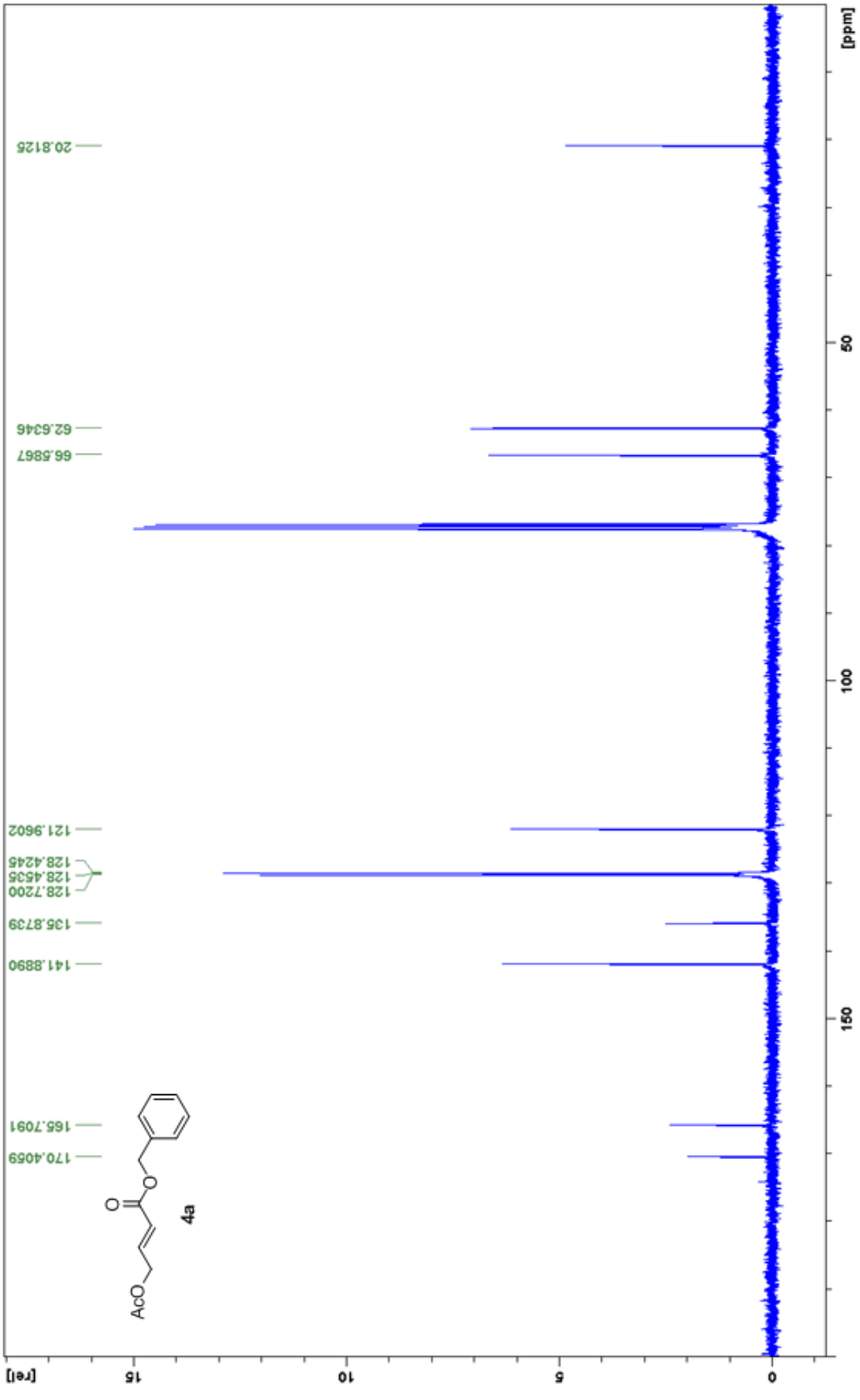
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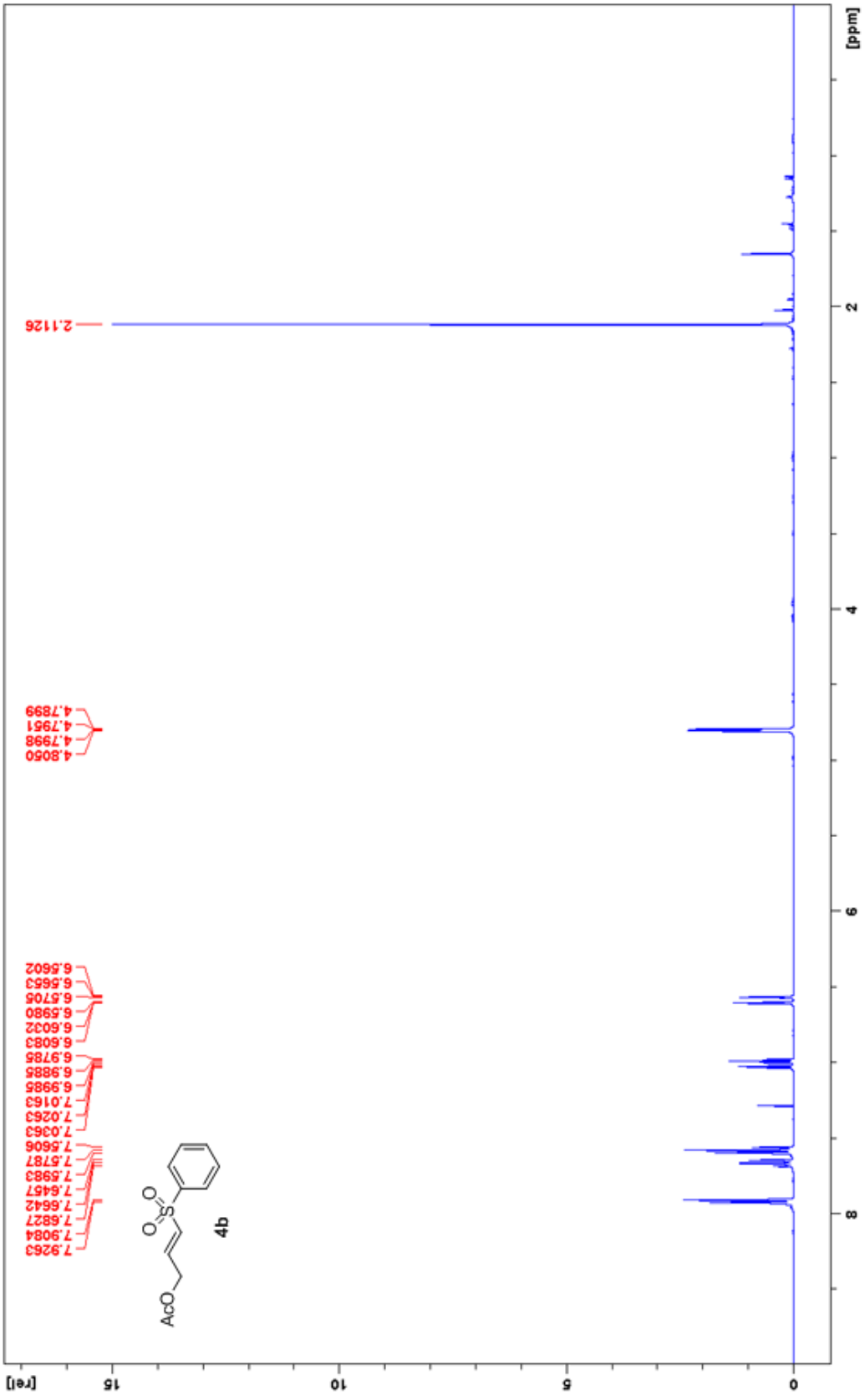
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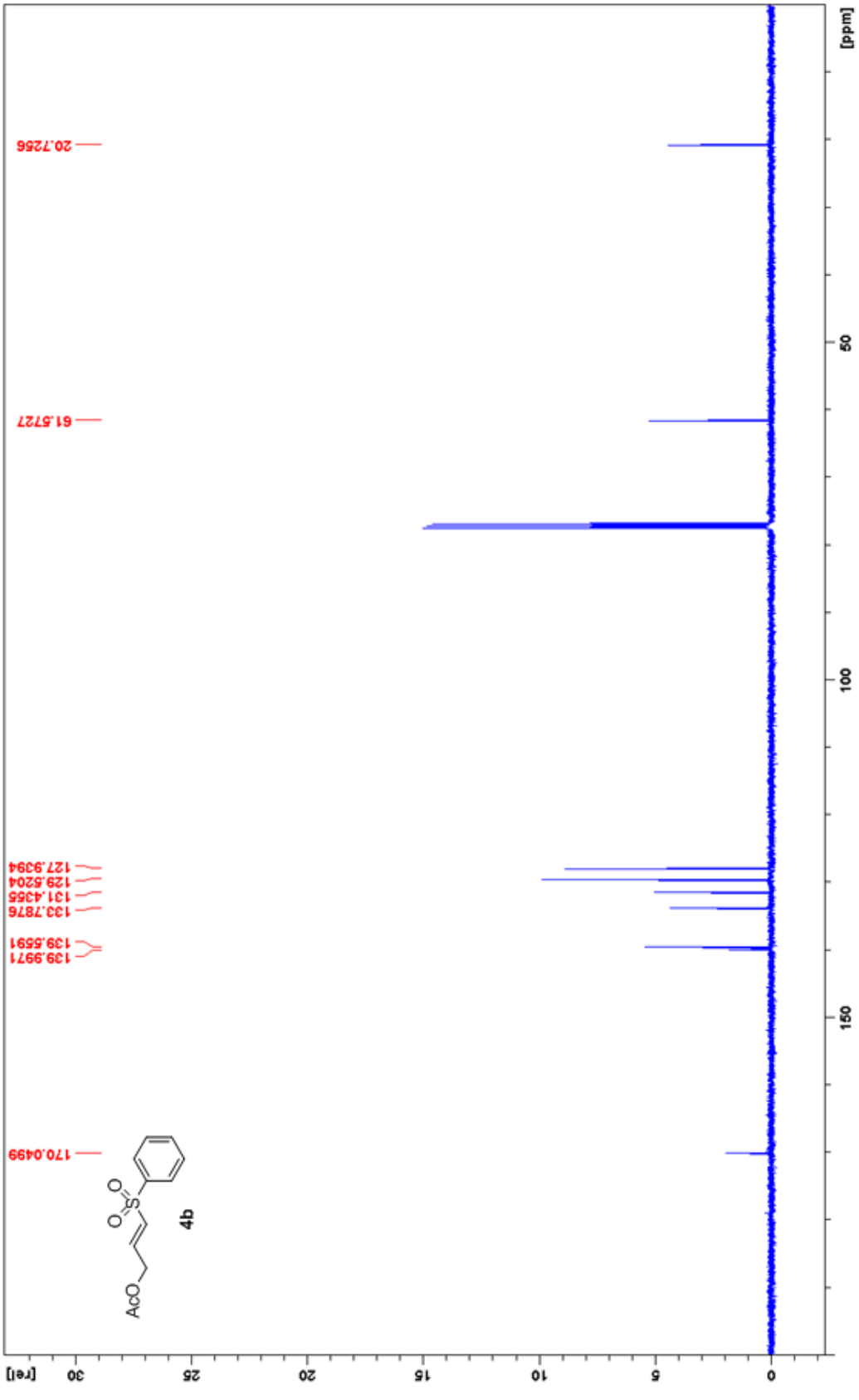
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61.5727
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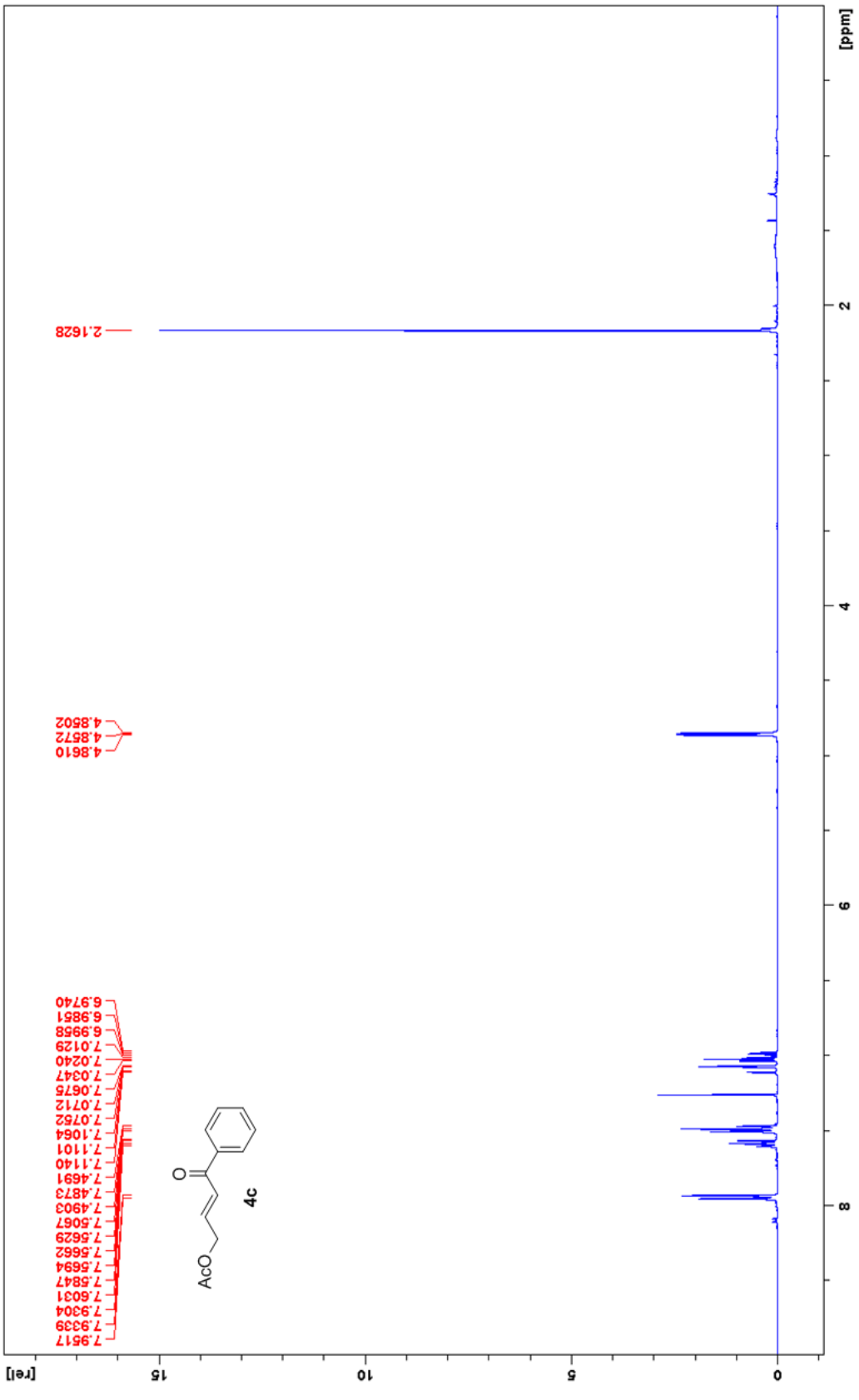
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7.5662
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7.4873
7.4691
7.1140
7.1101
7.1064
7.0752
7.0712
7.0675
7.0347
7.0240
7.0129
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6.9740
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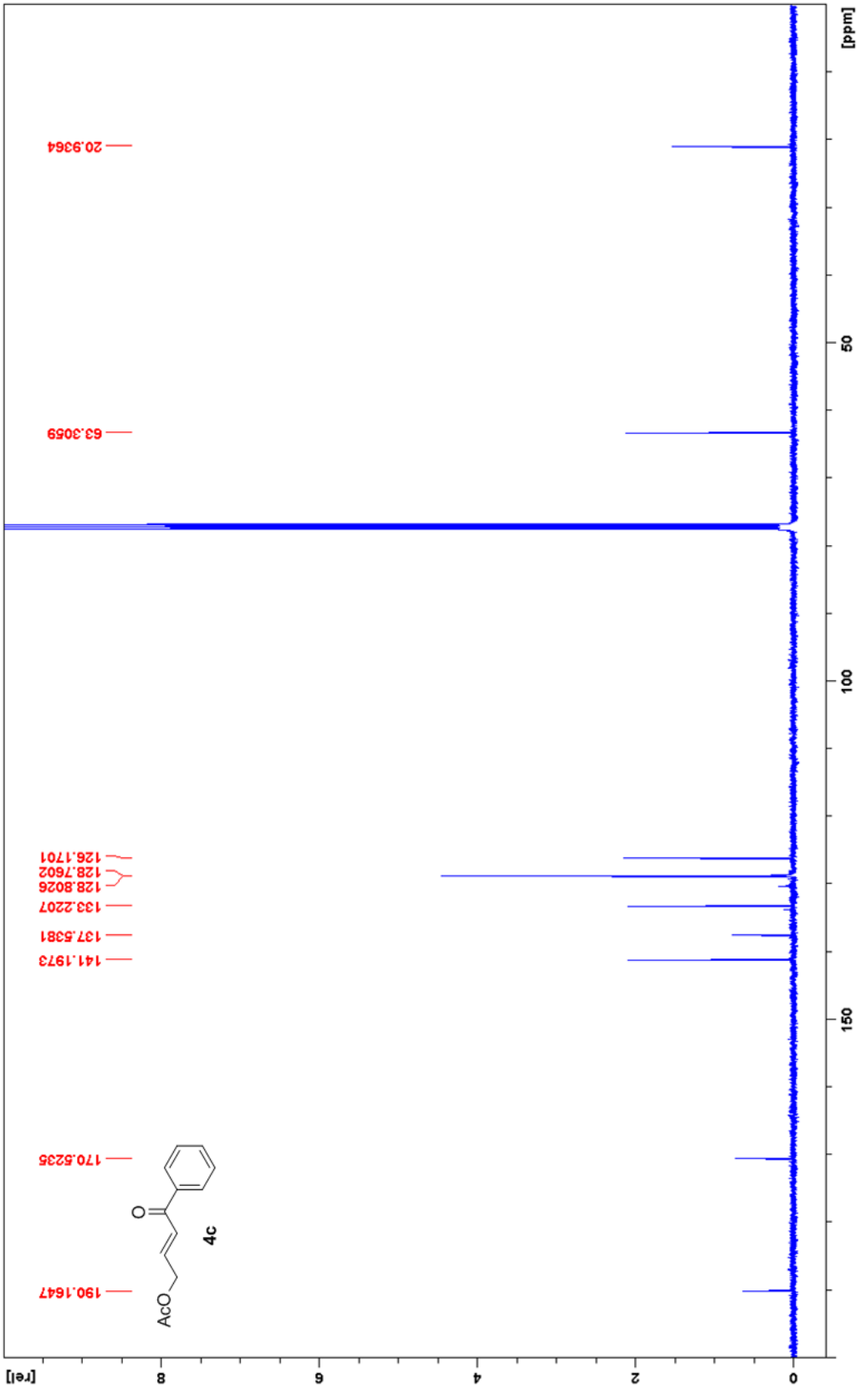
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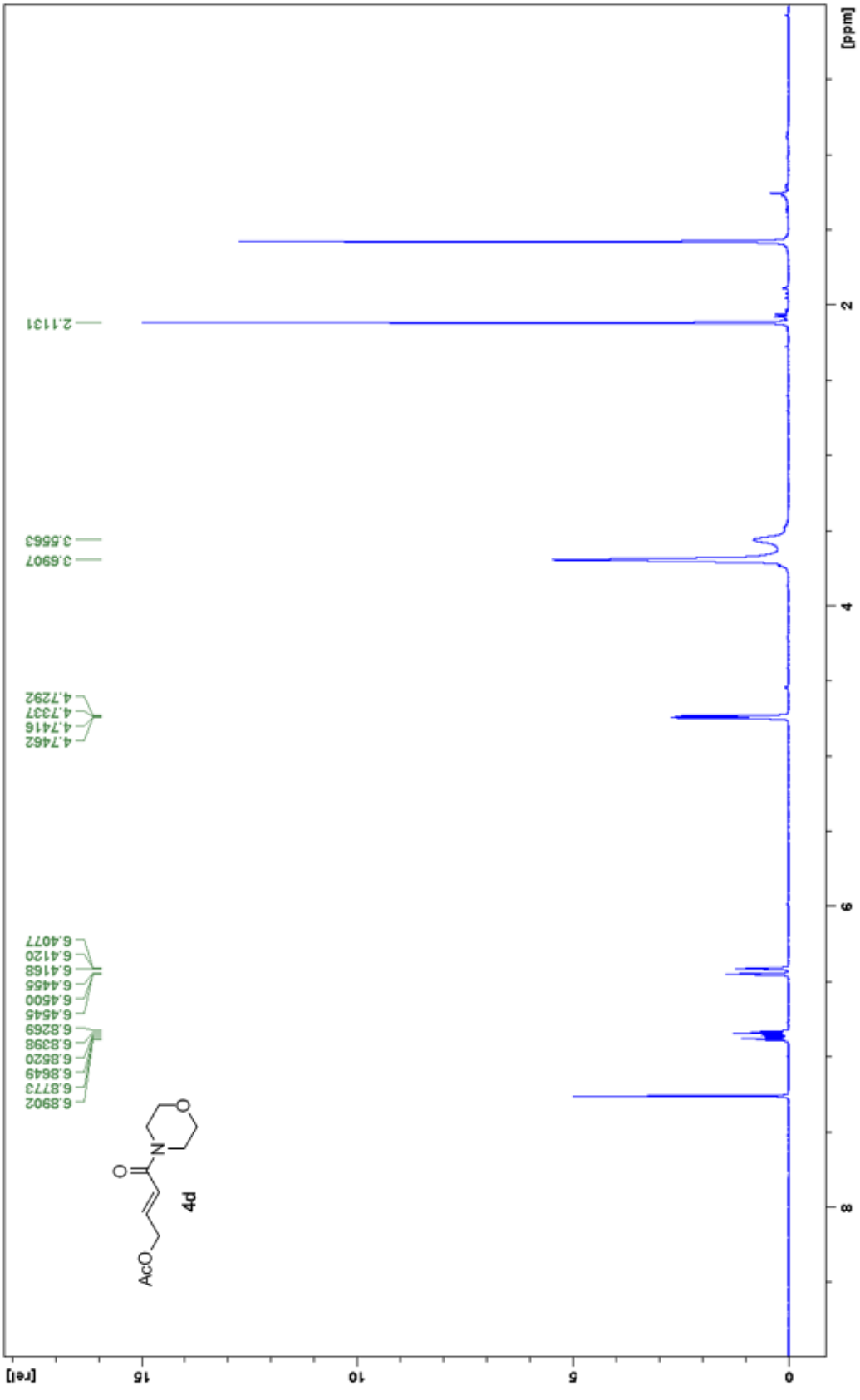
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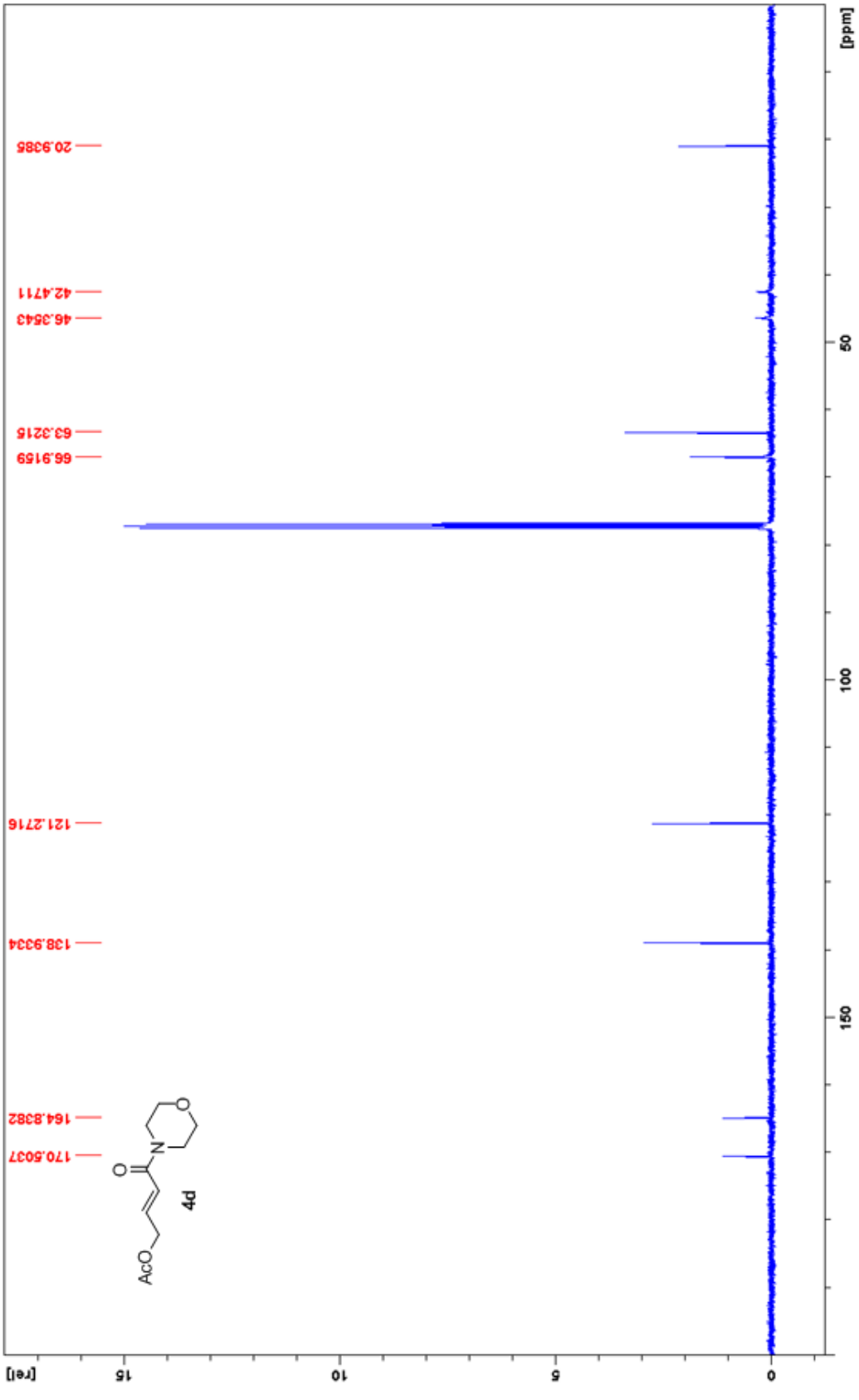
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AcO
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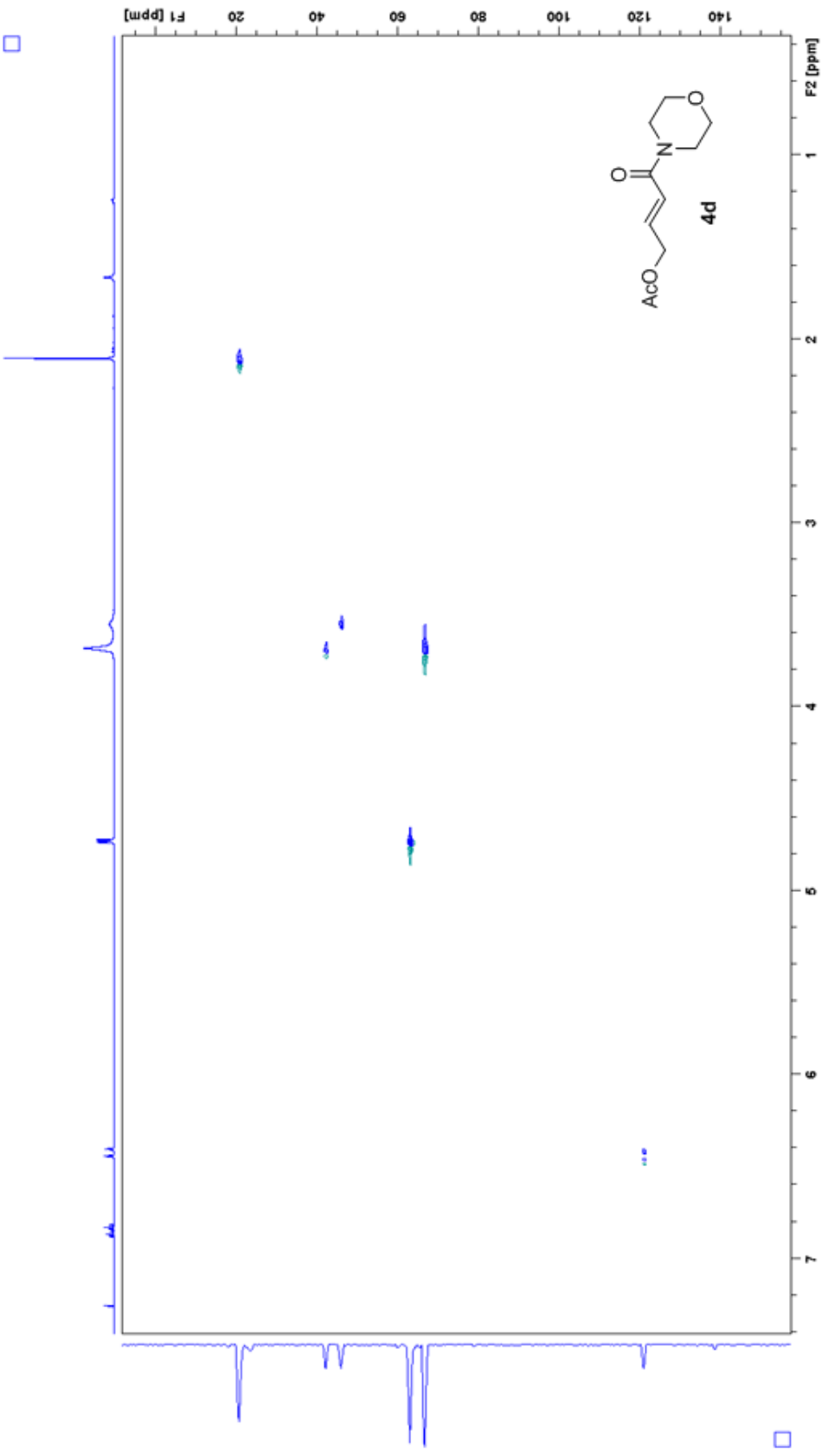
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6
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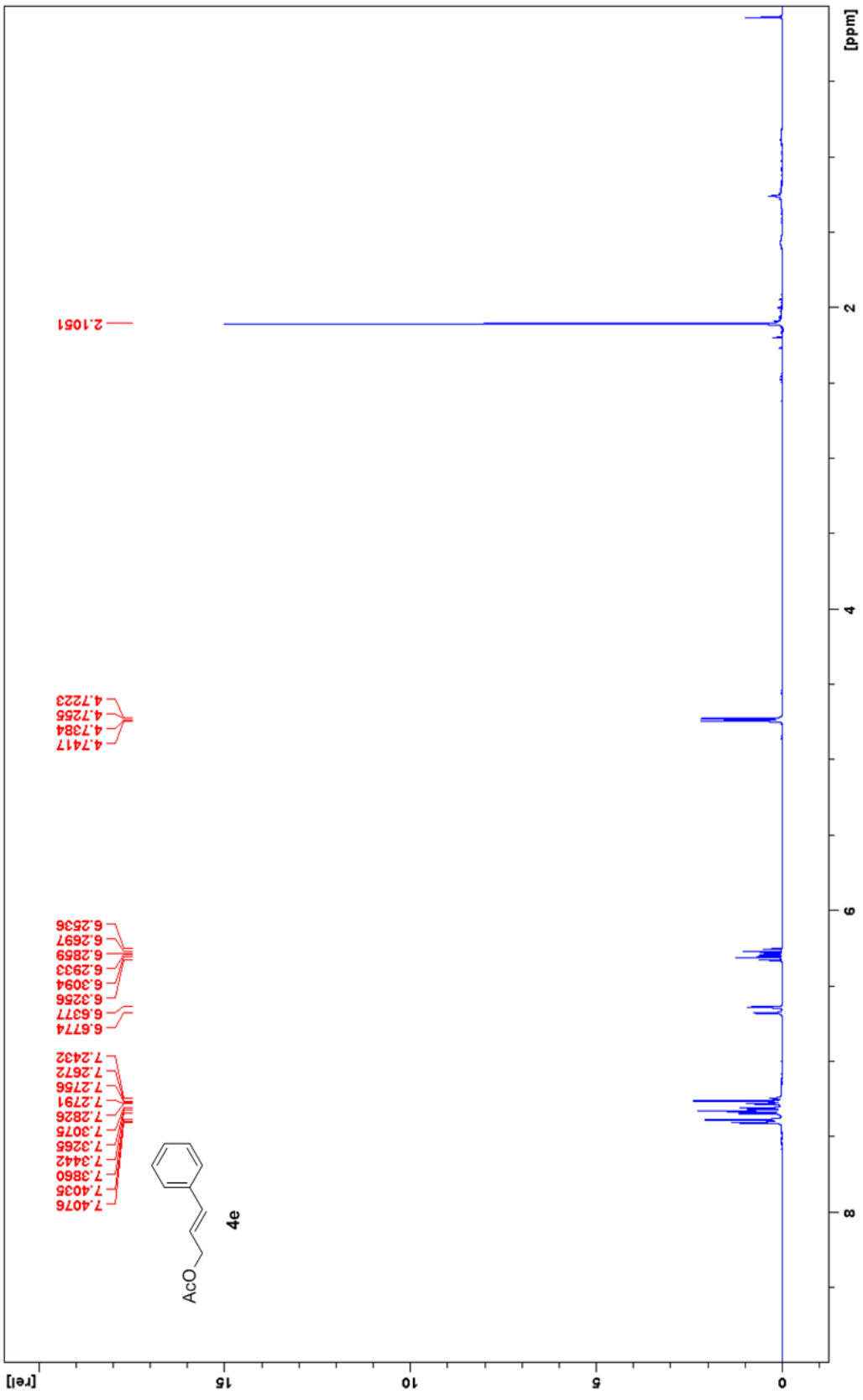
AcO
4d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7.3075
7.2826
7.2791
7.2756
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7.2432
6.5774
6.5377
6.3256
6.3094
6.2933
6.2859
6.2697
6.2536
4.7417
4.7384
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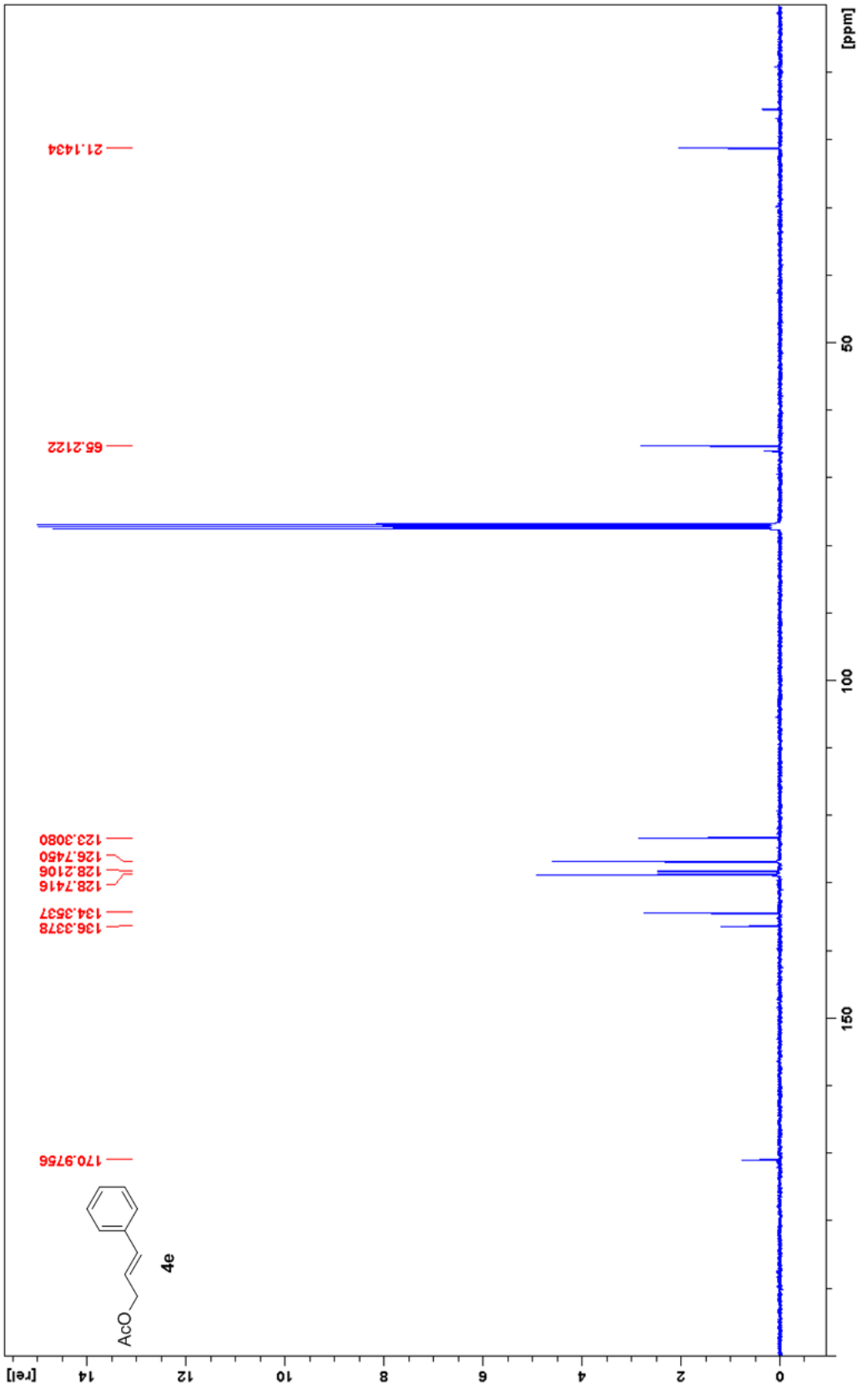
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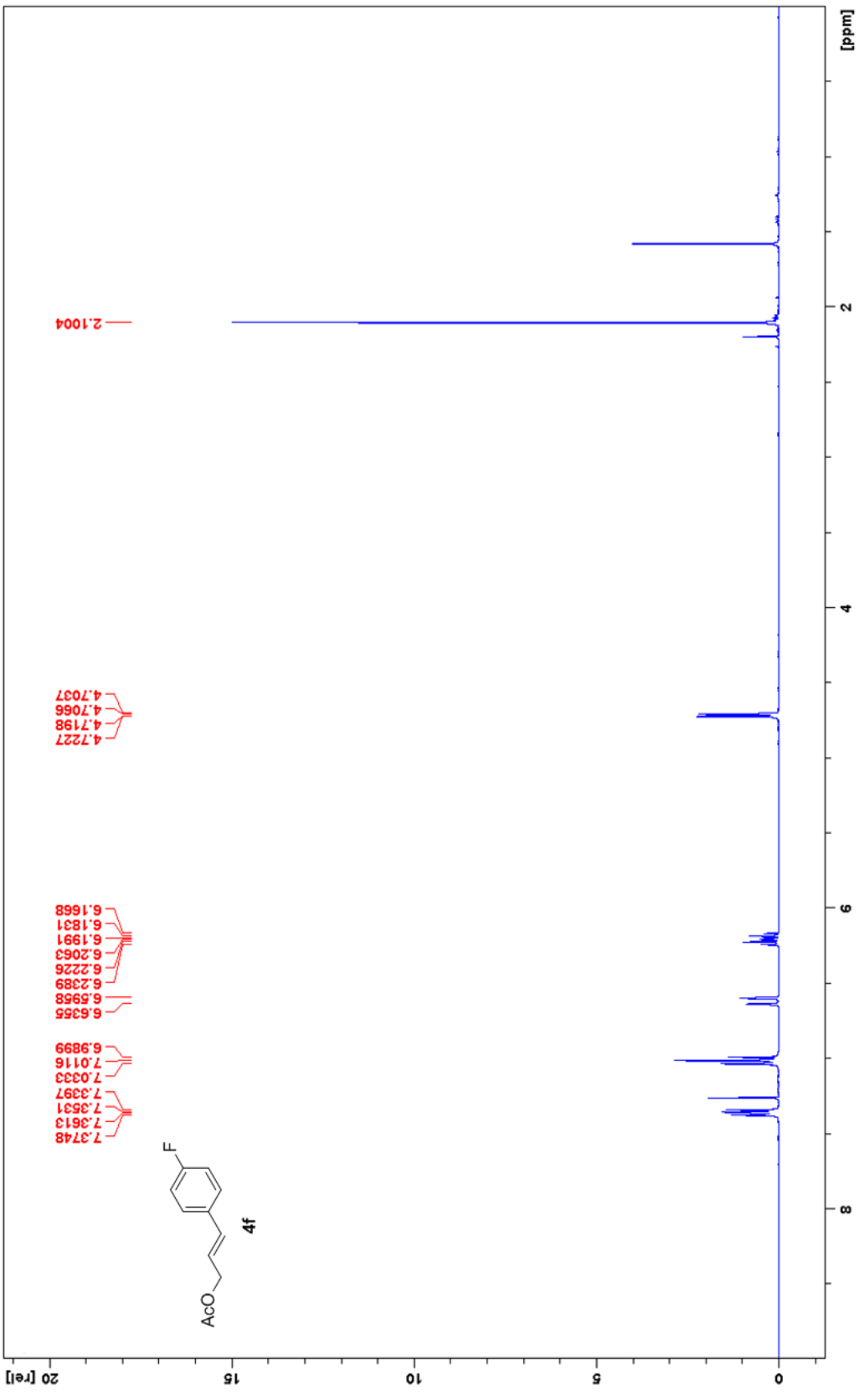
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6.5958
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6.2226
6.2063
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4.7037
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AcO
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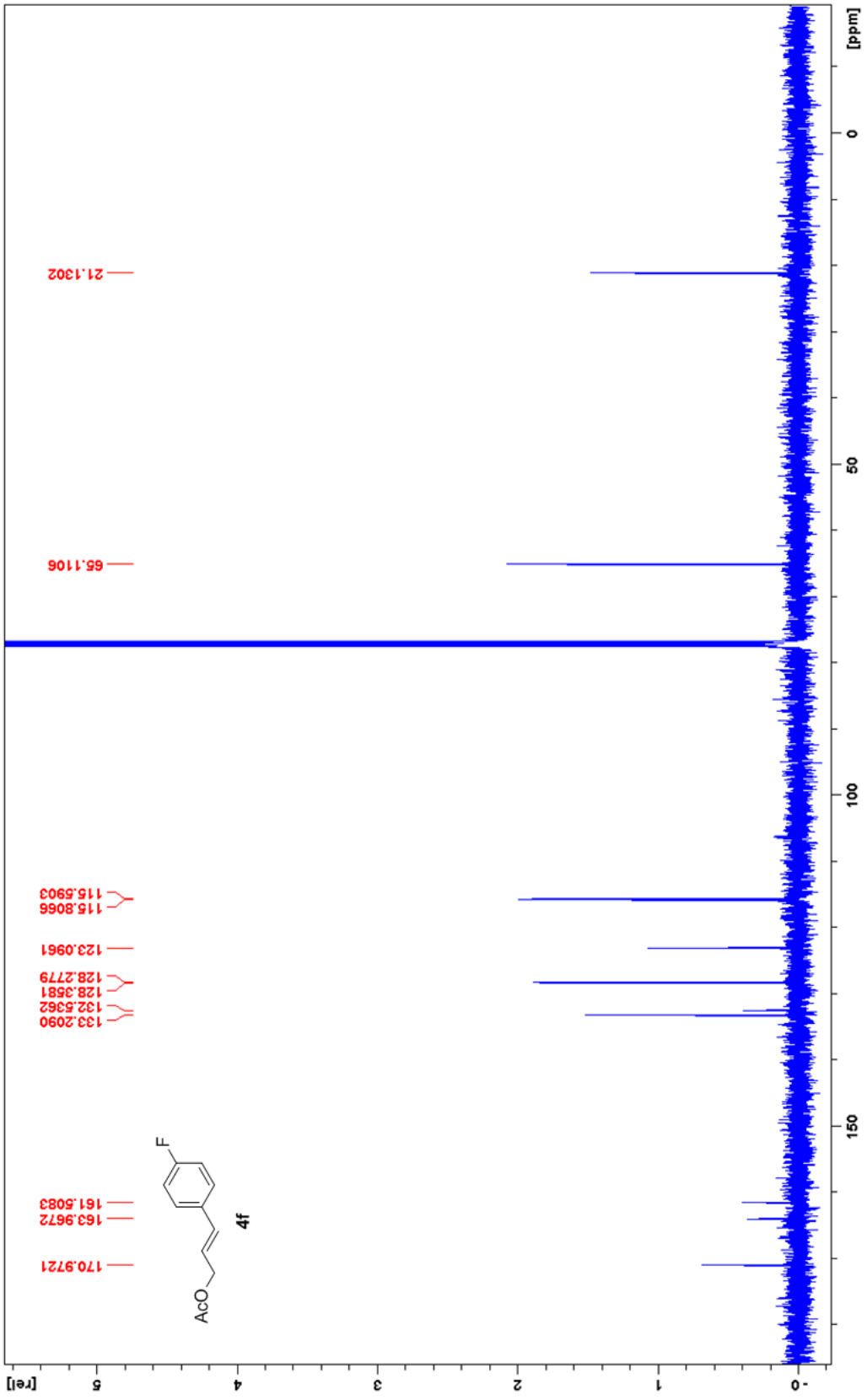
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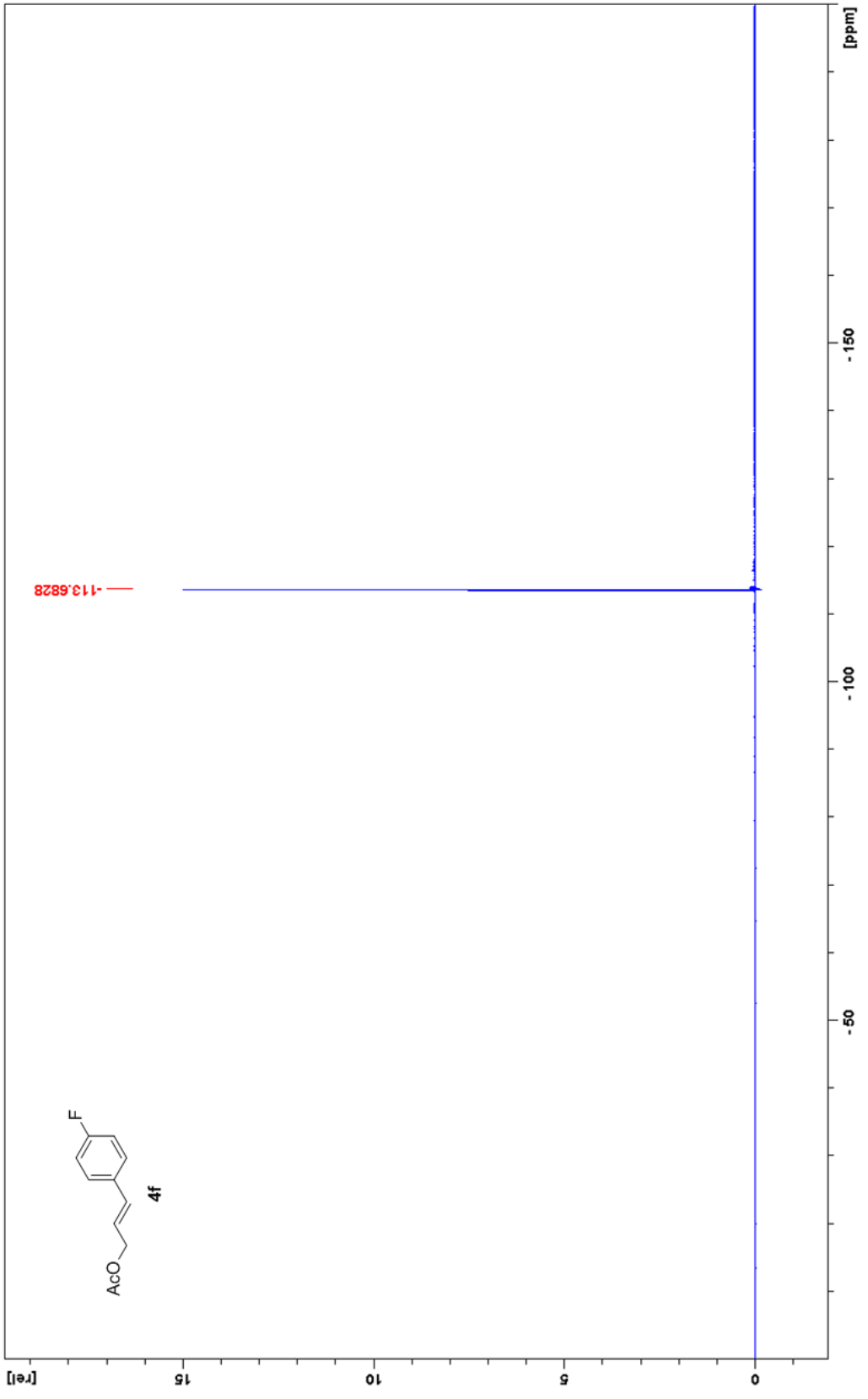
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- 100
- 150
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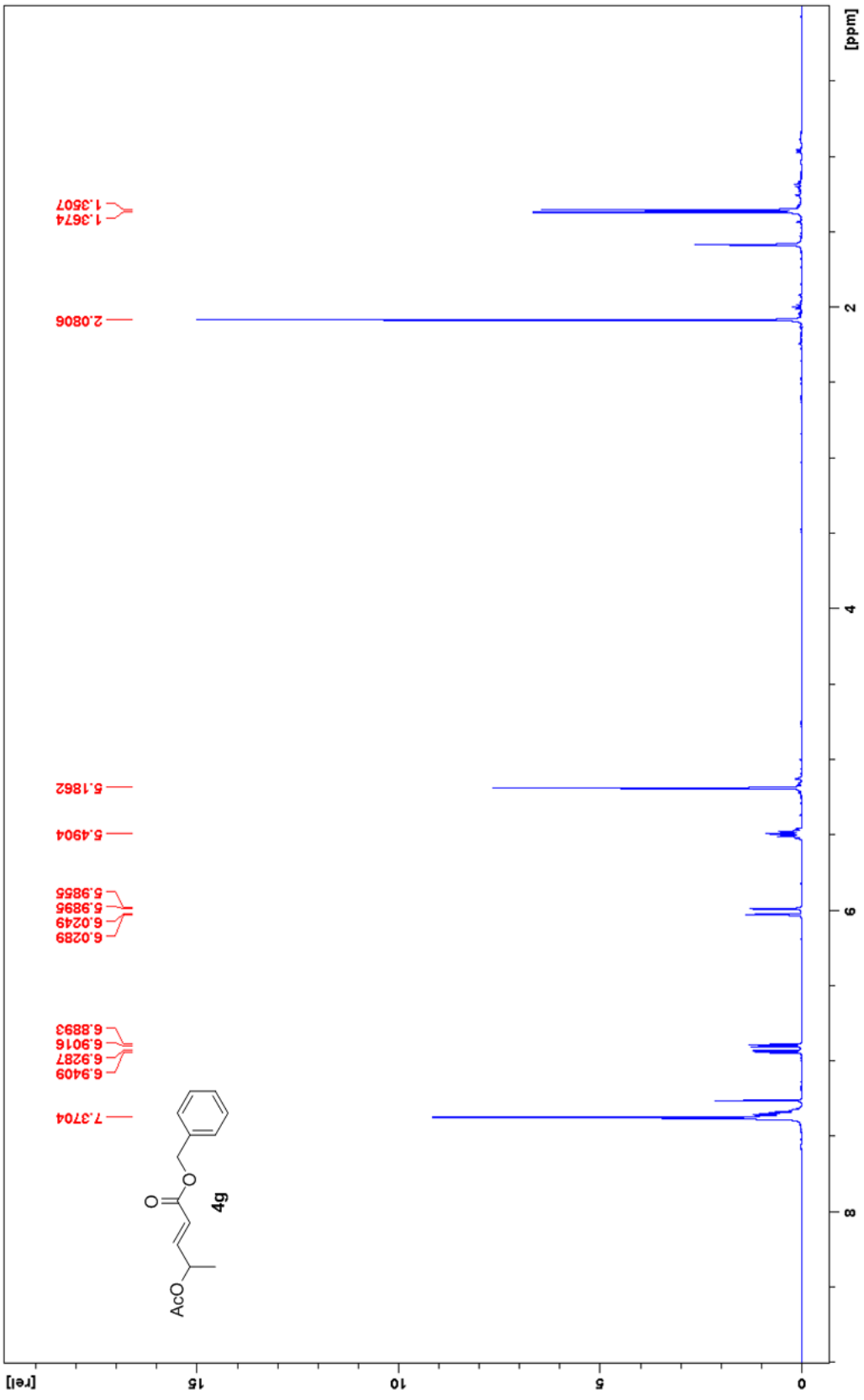
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5.9855
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5.1862
2.0806
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4g
AcO
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O
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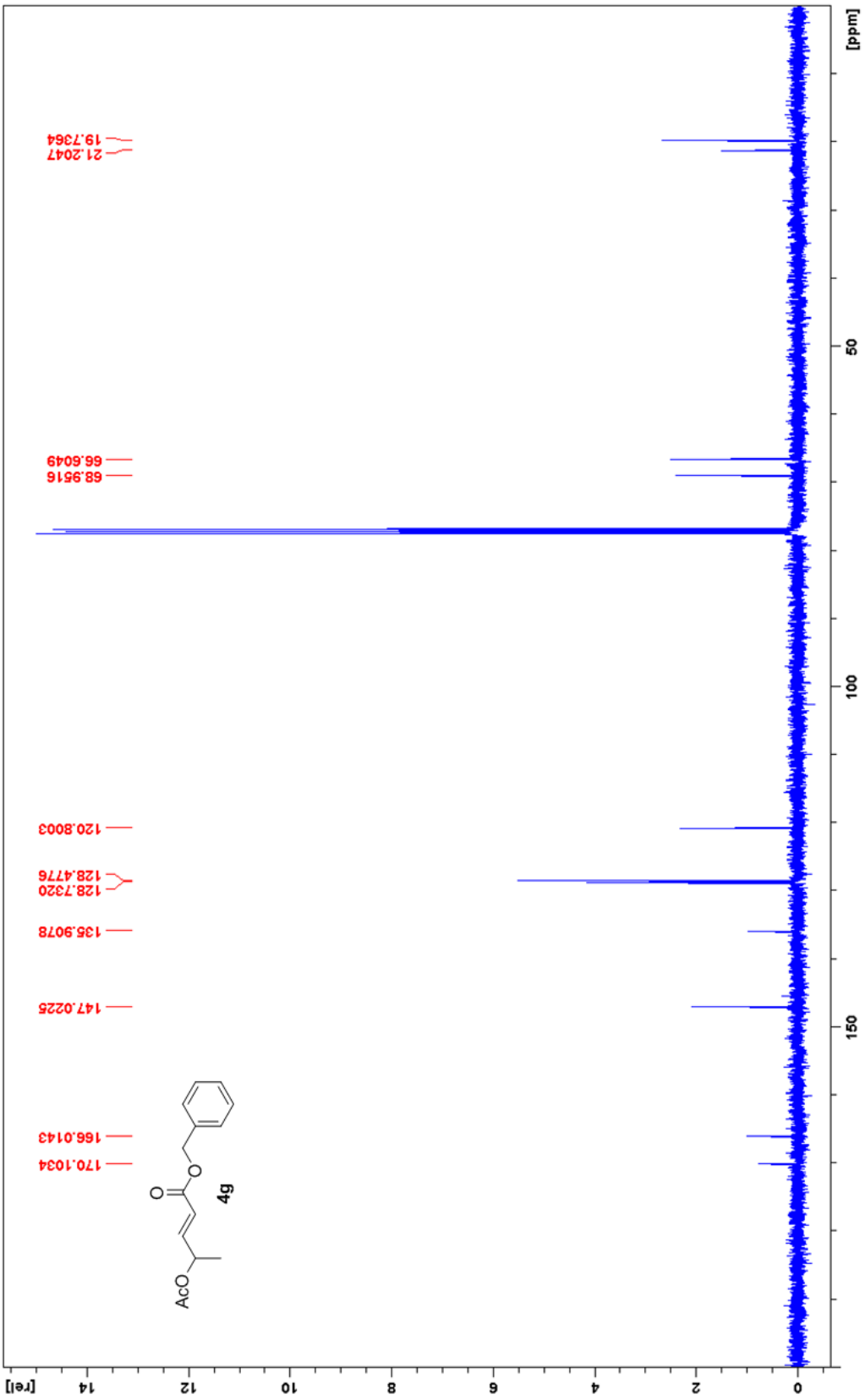
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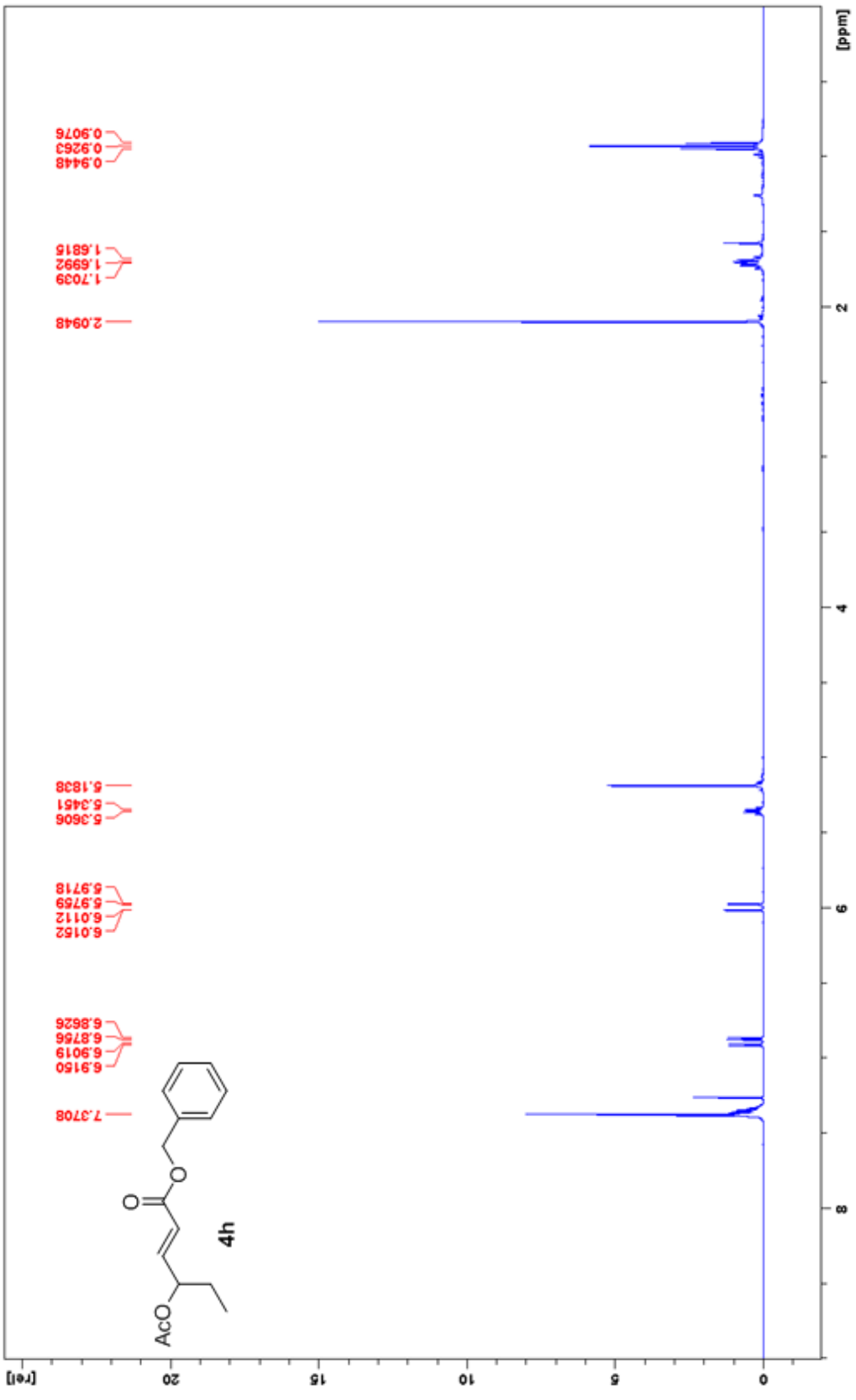
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5.9759
5.9718
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5.3451
5.1838
2.0948
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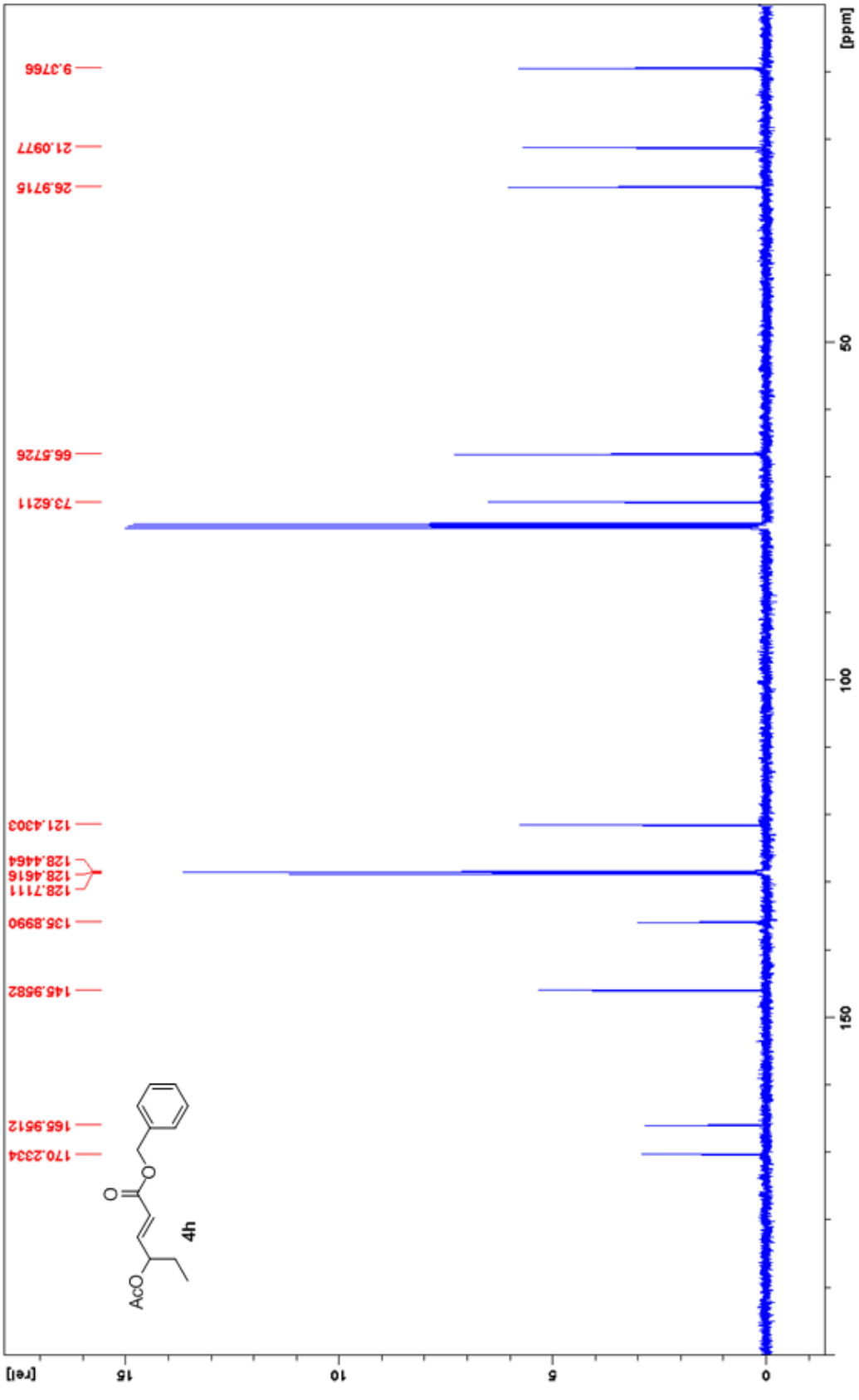
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4h
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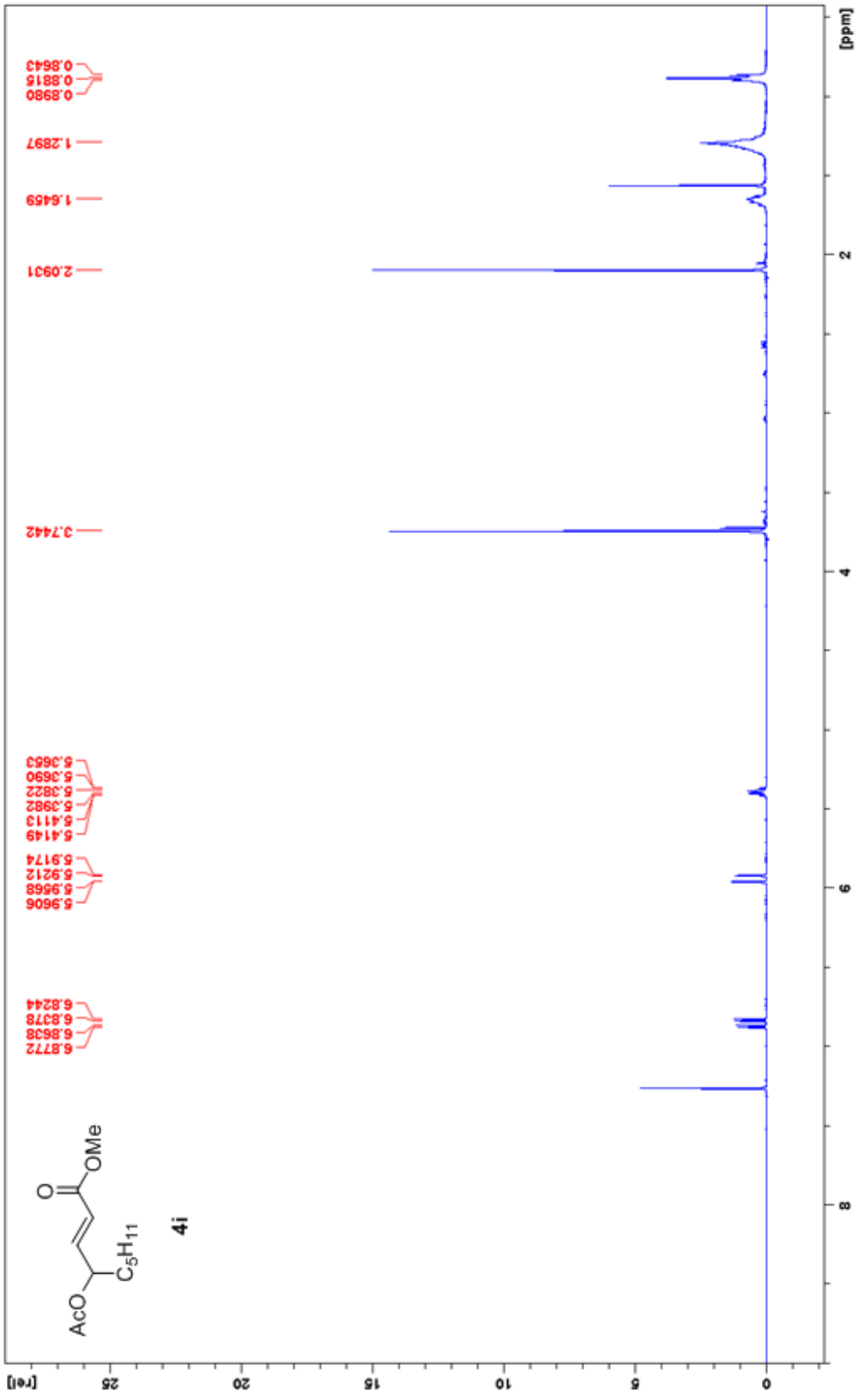
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5.4113
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5.9174
5.9212
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C5H11
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OMe
4i
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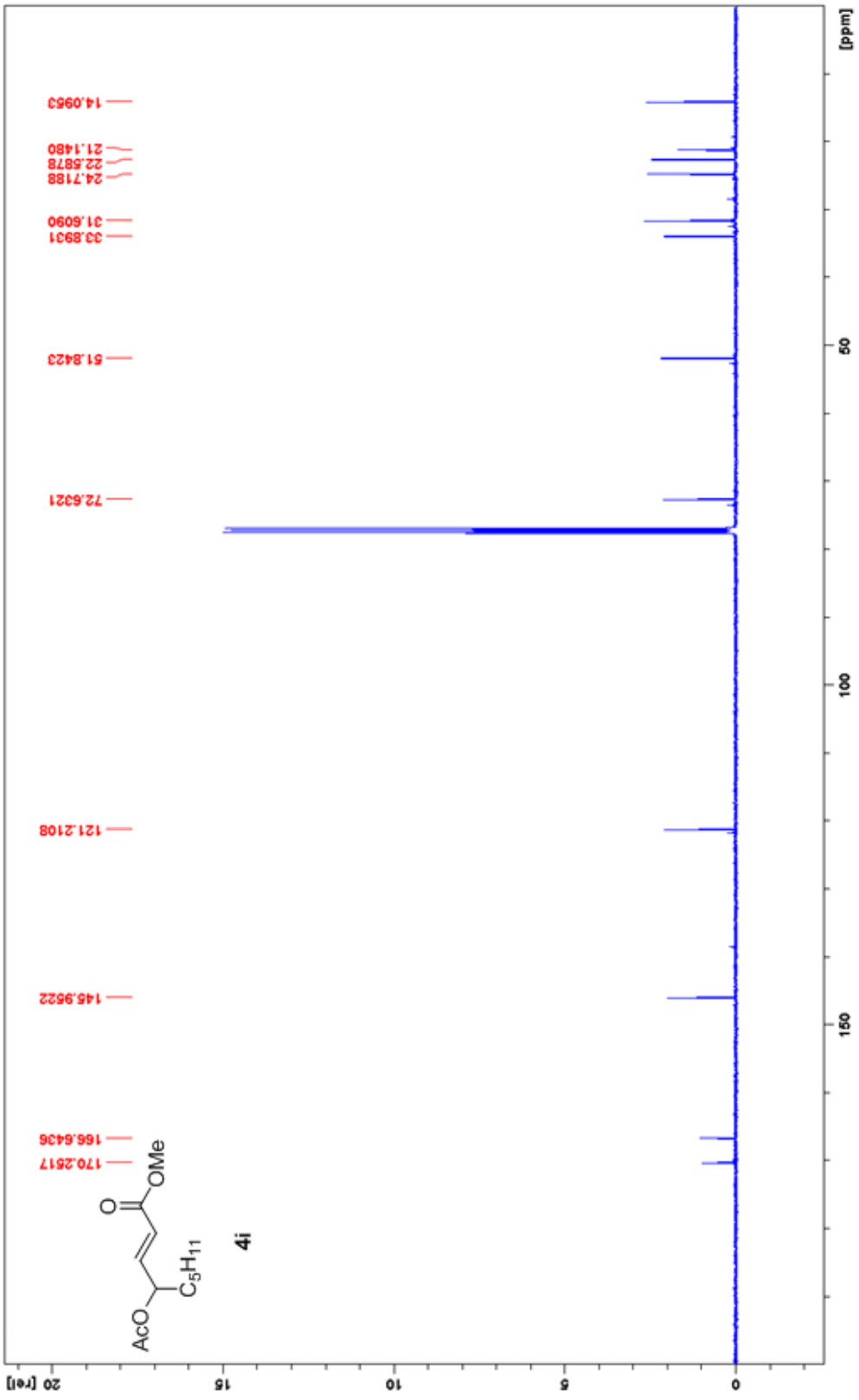
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C_5H_{11}
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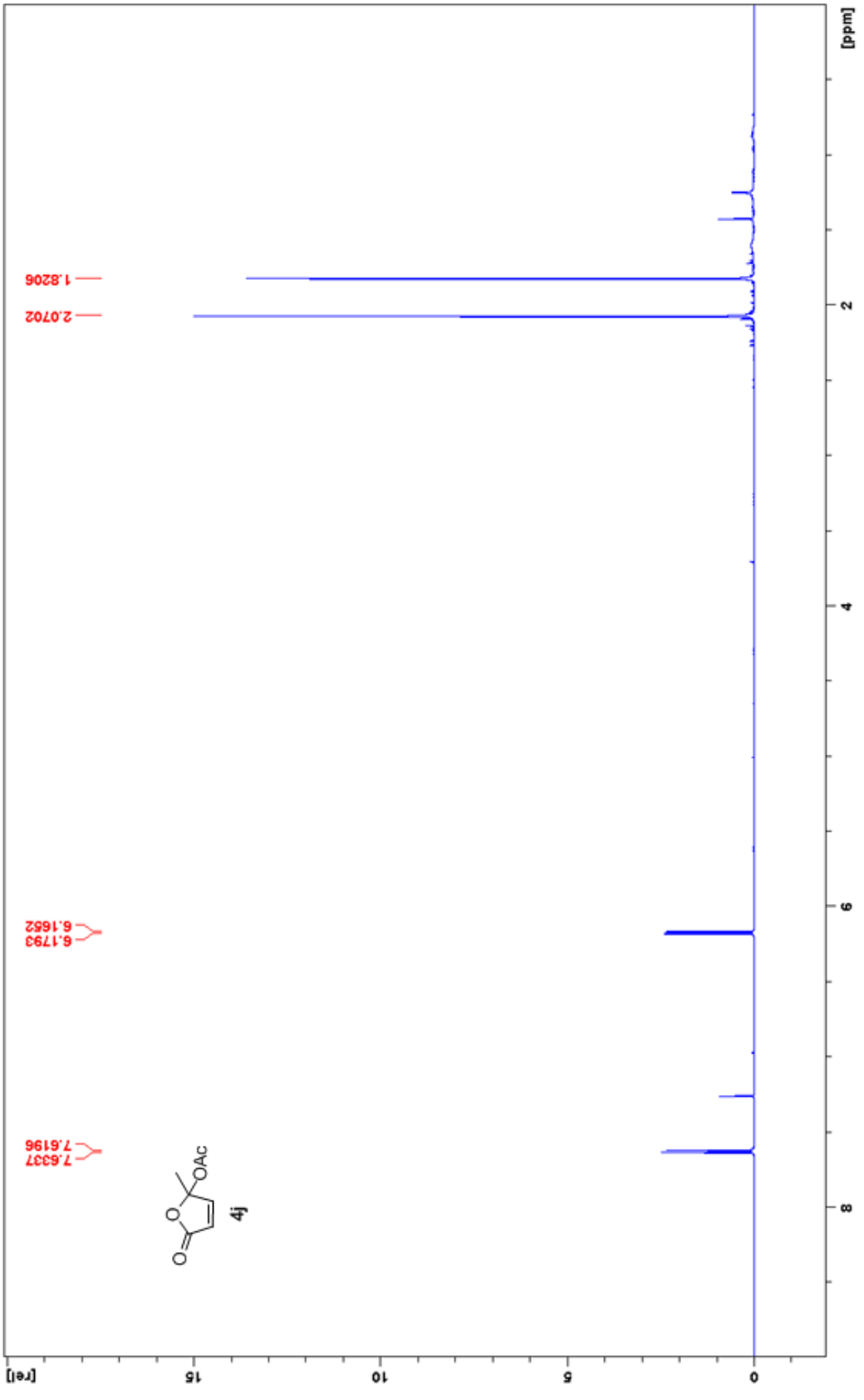
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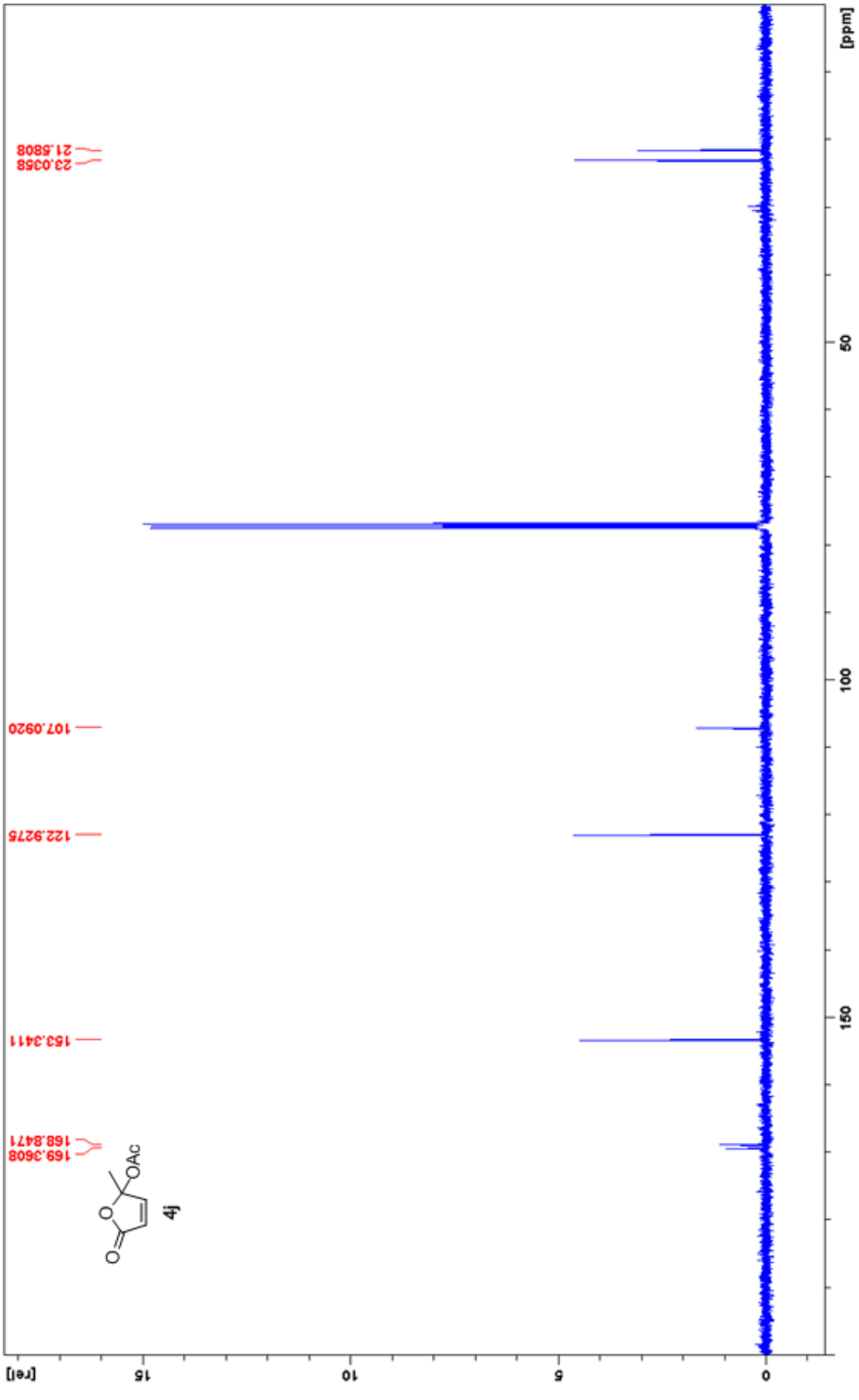
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4j
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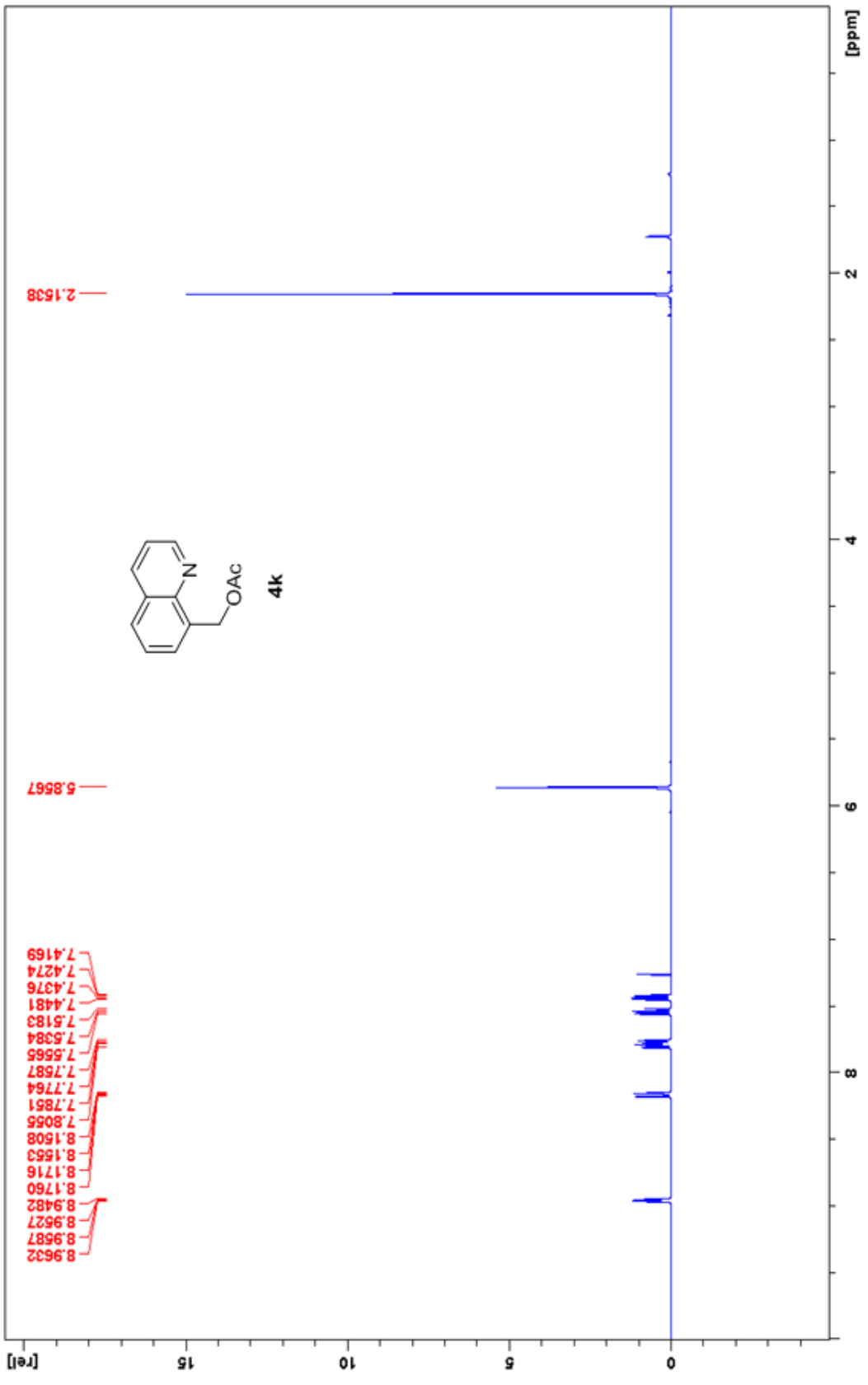
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7.7851
7.8055
8.1508
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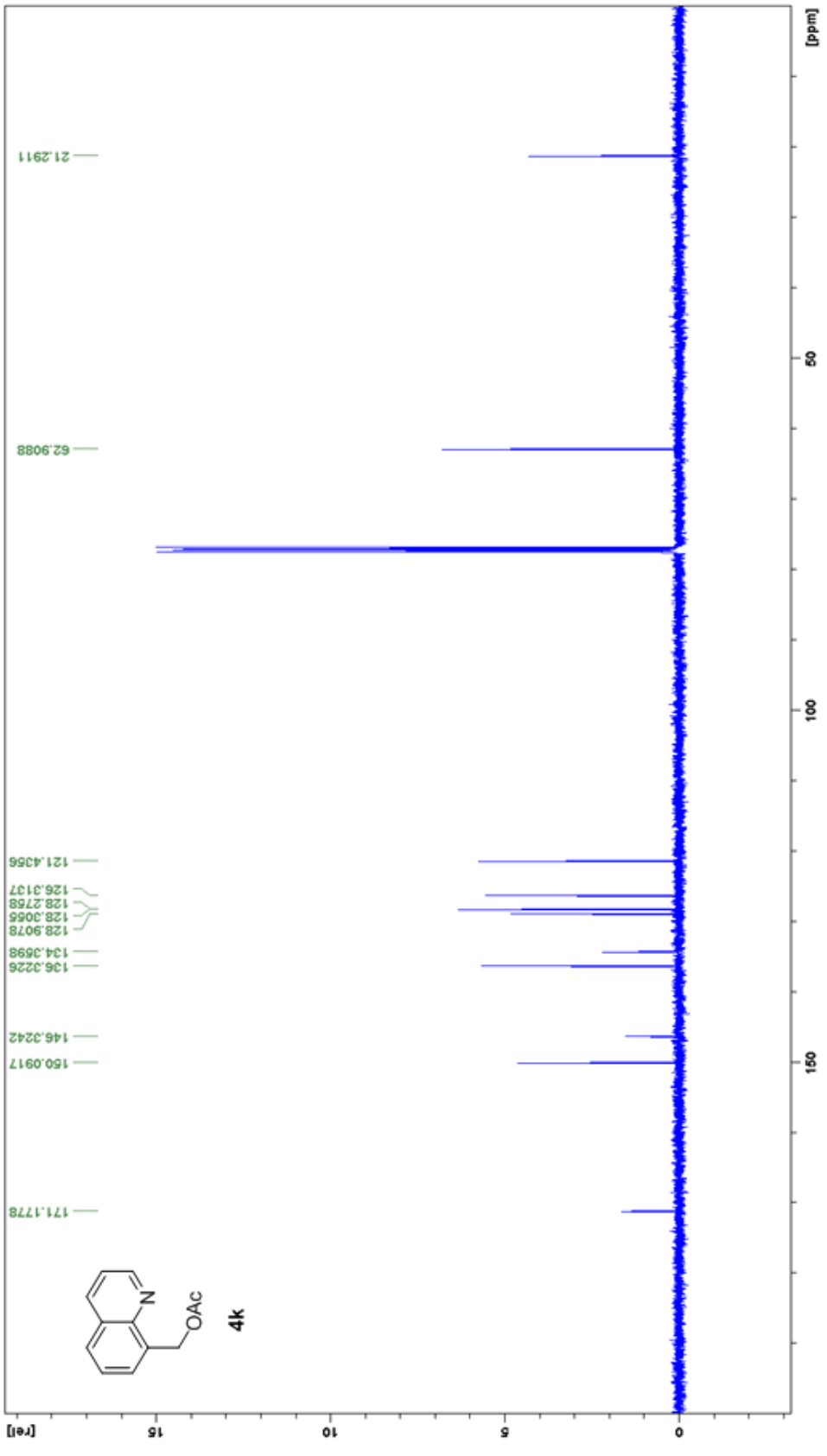
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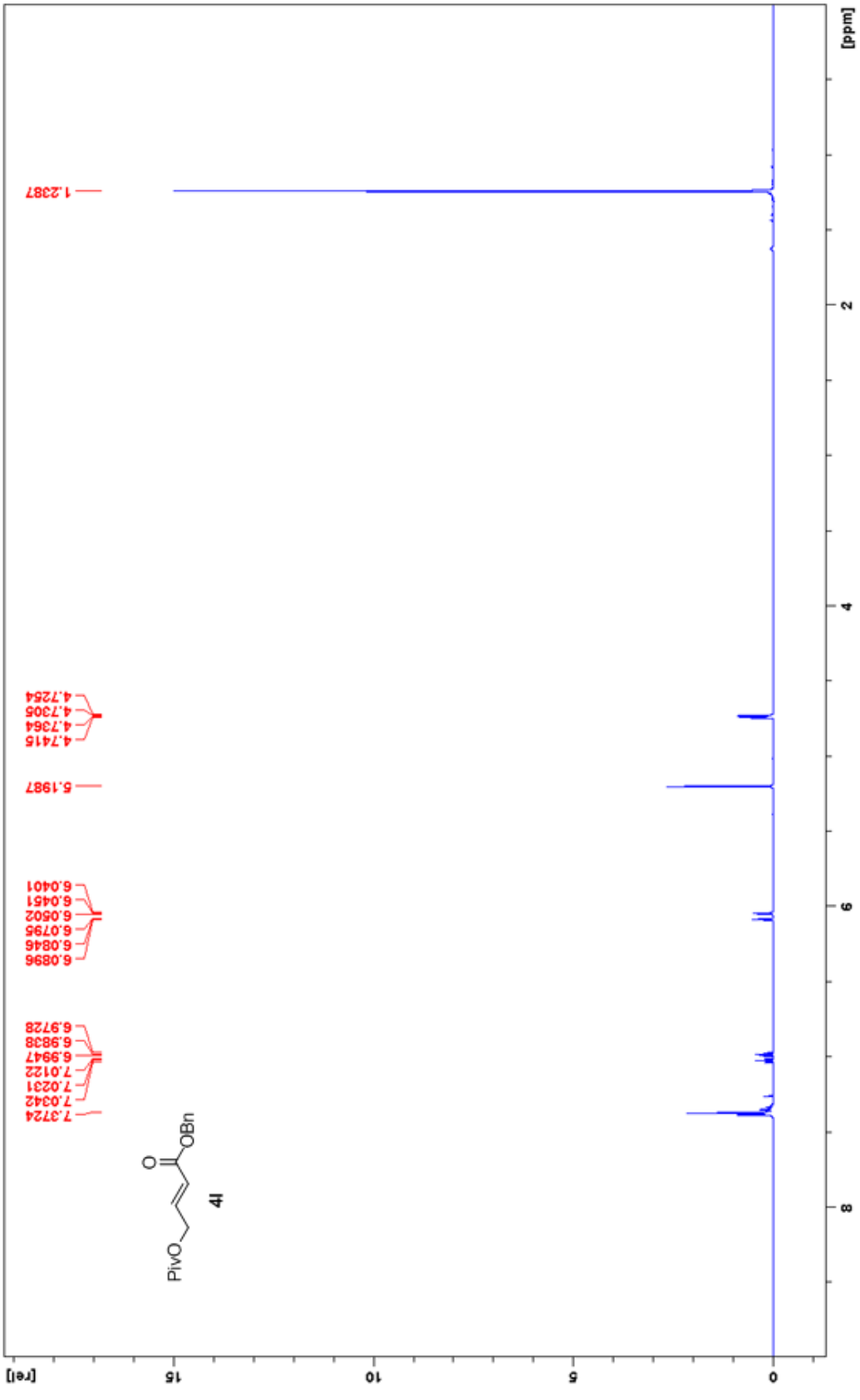
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4.7364
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OBn
4l
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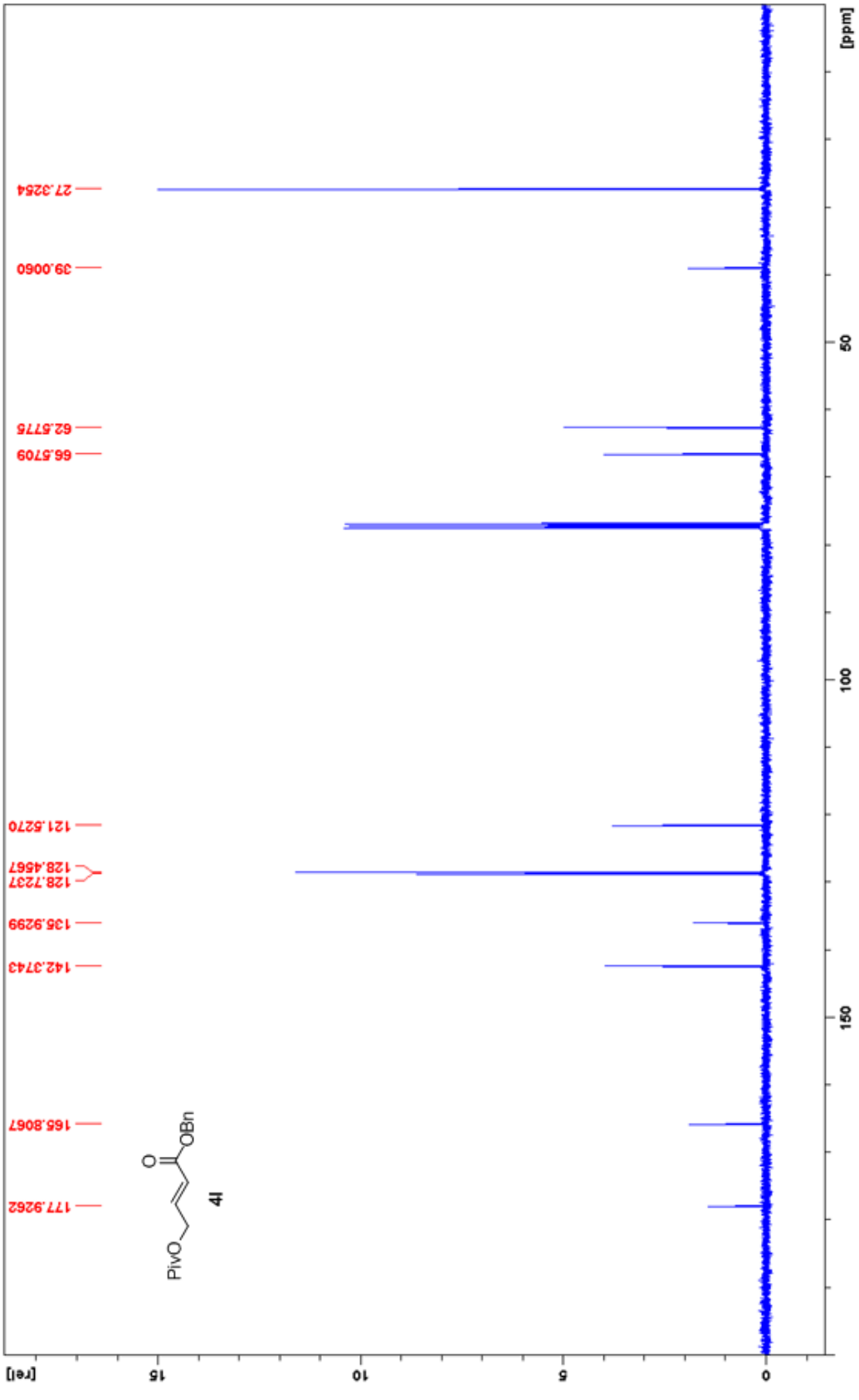
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4l
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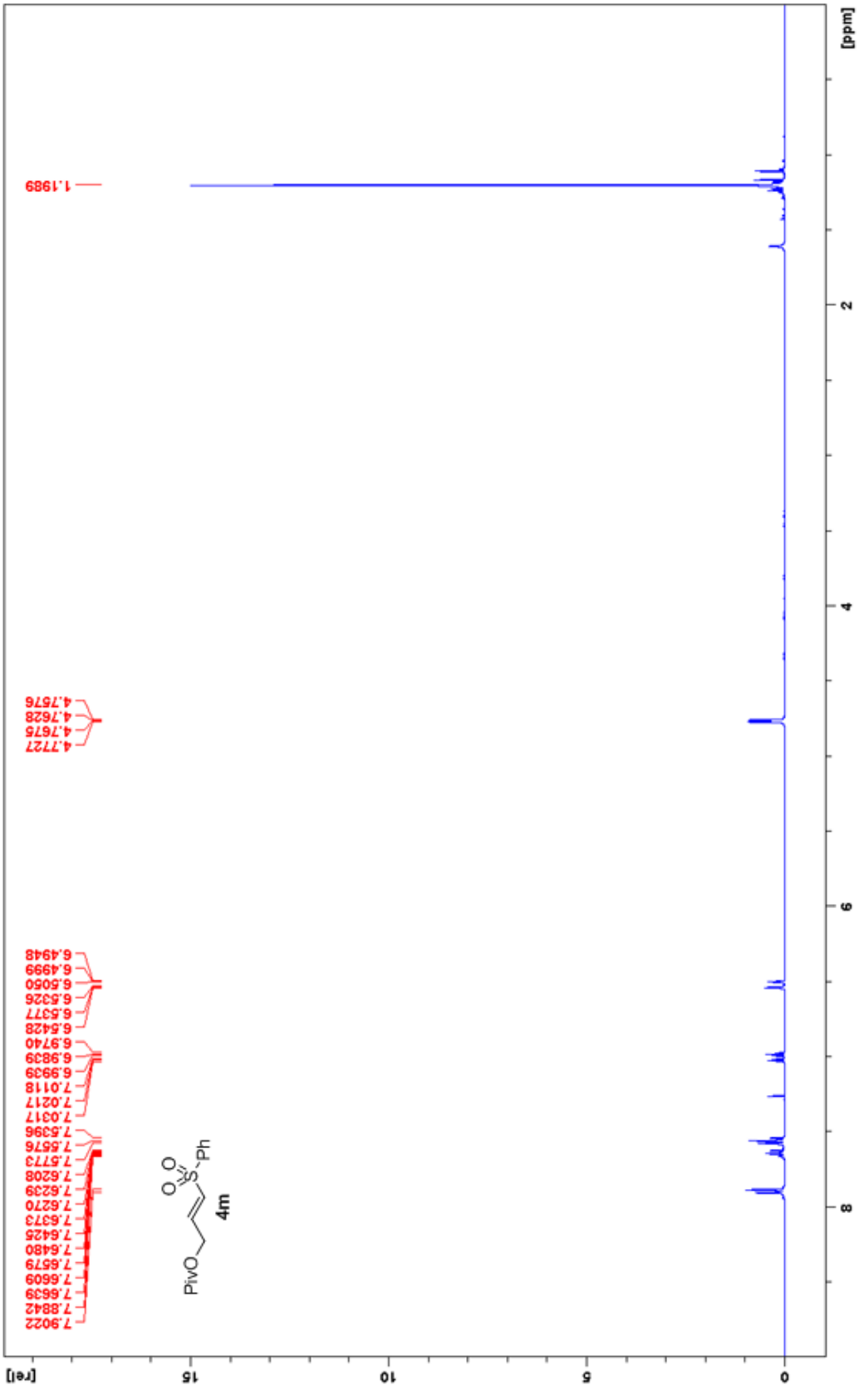
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Ph
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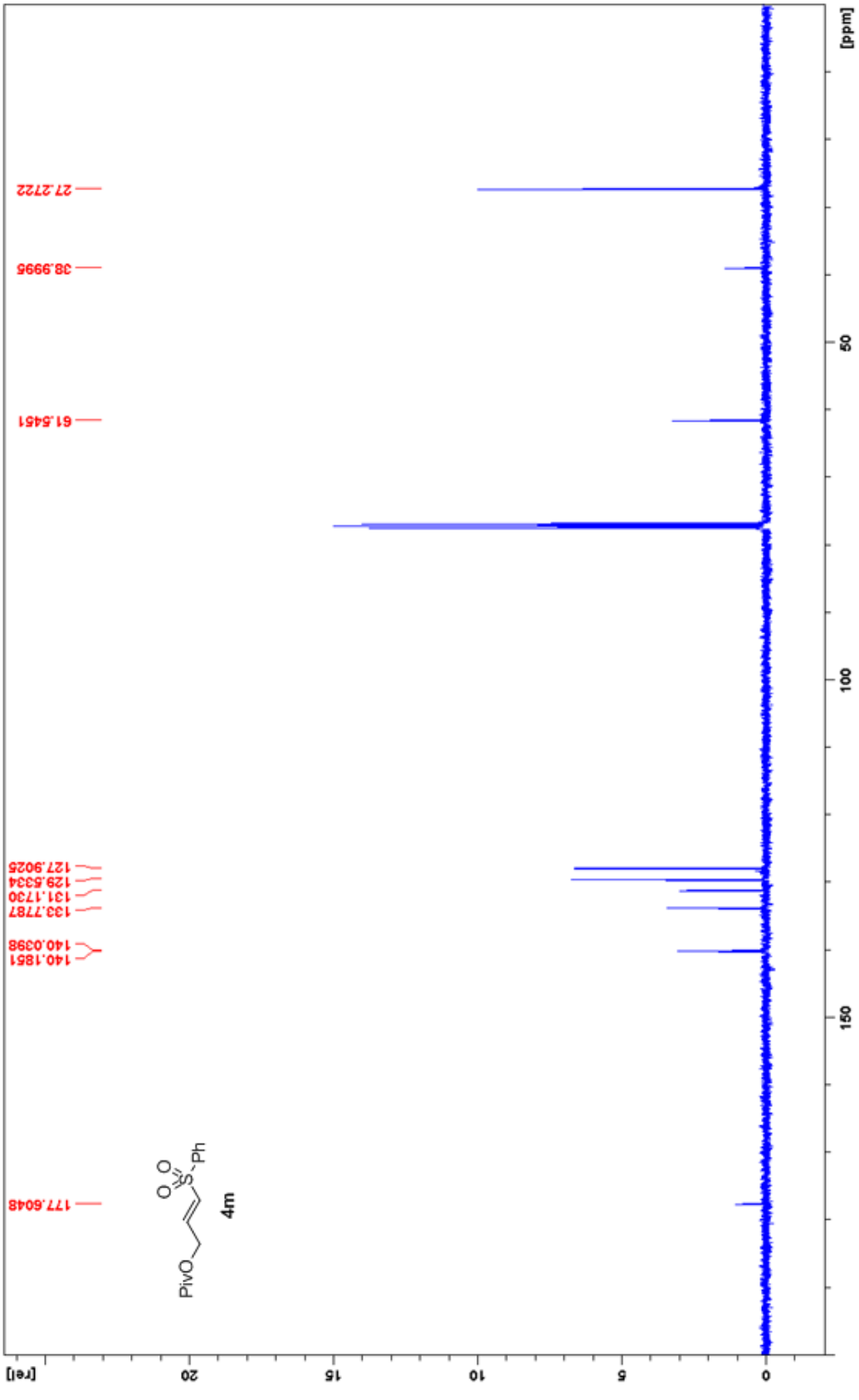
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O
S
Ph
4m
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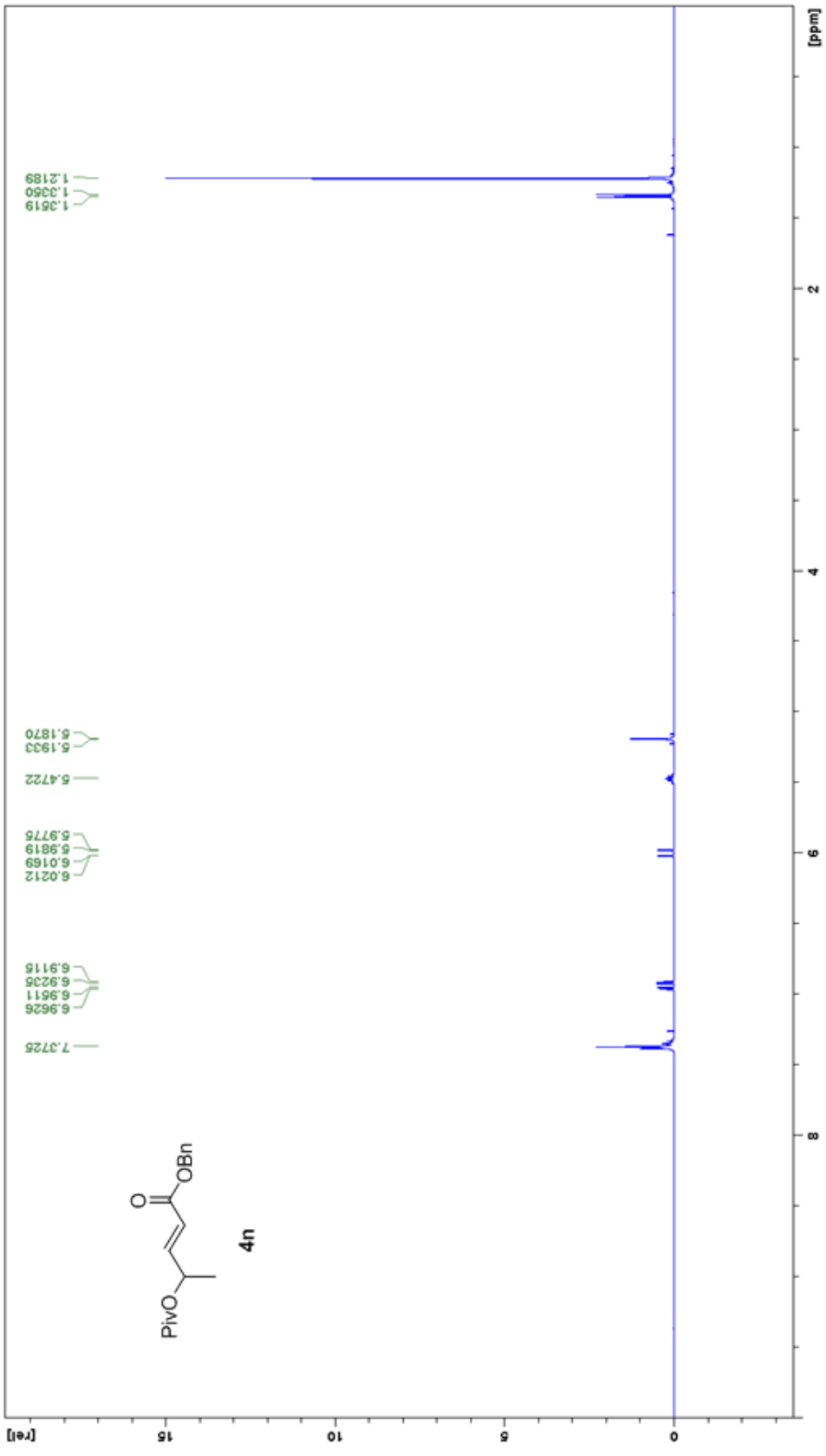
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PivO
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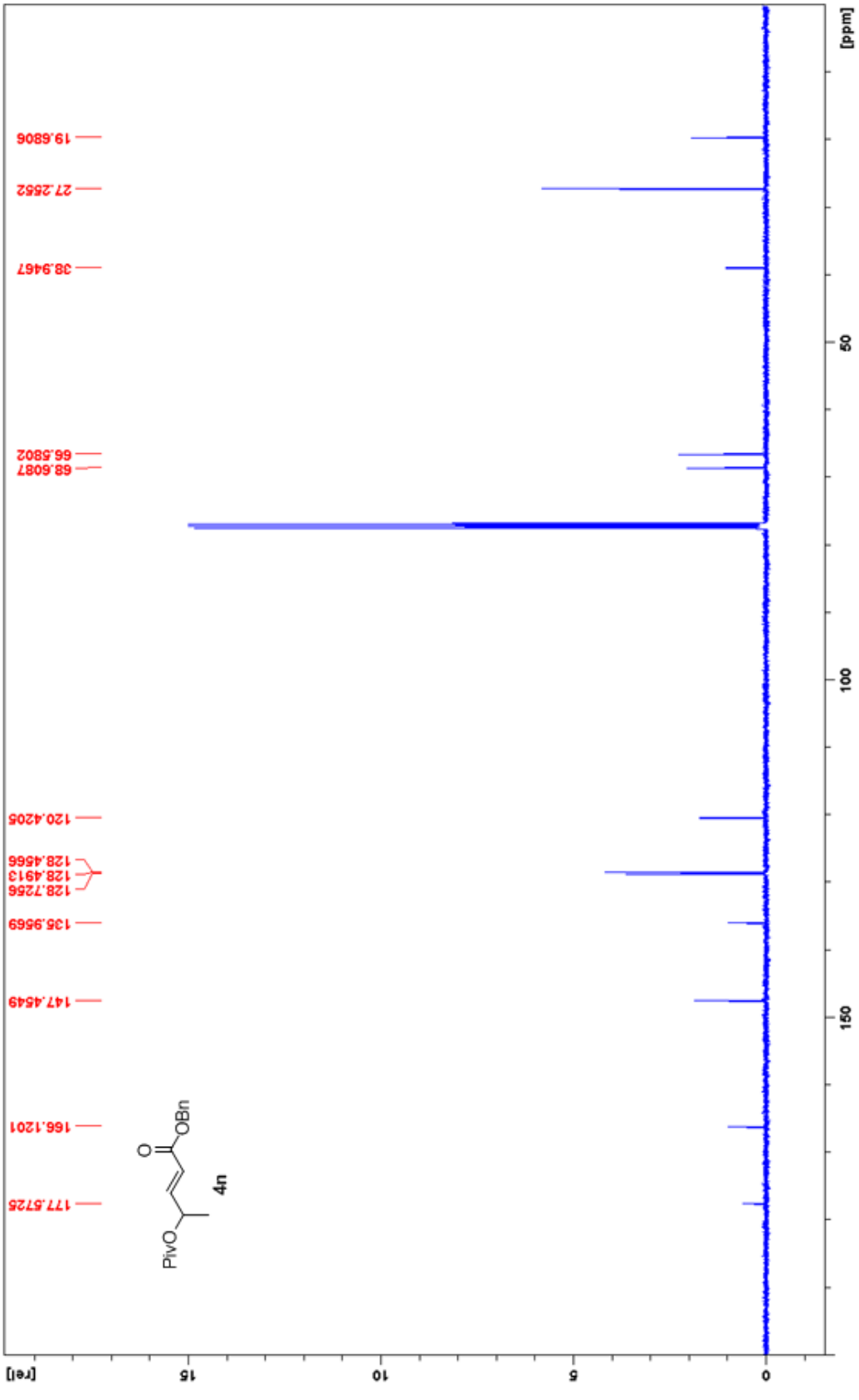
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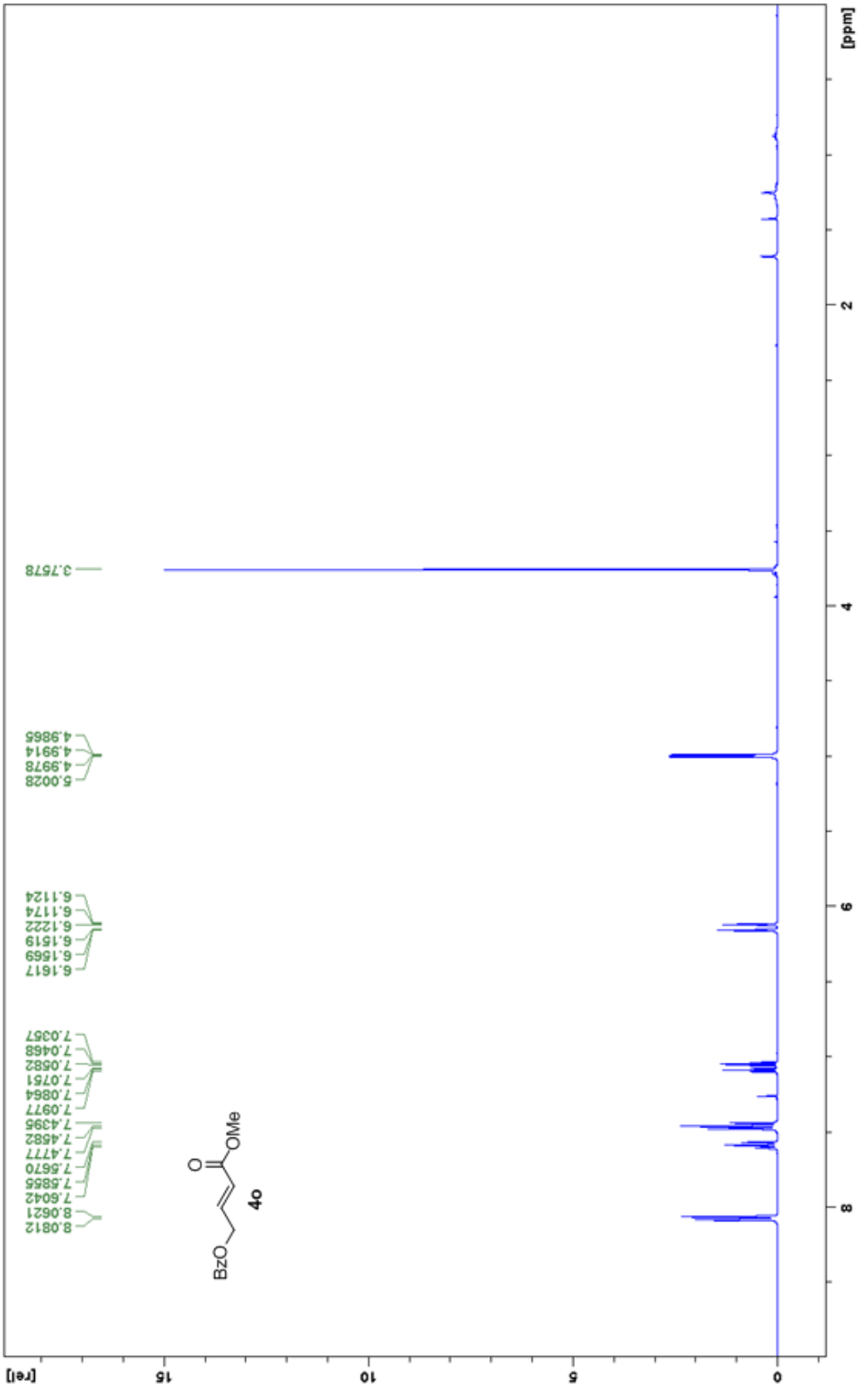
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7.0468
7.0357
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OMe
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4o
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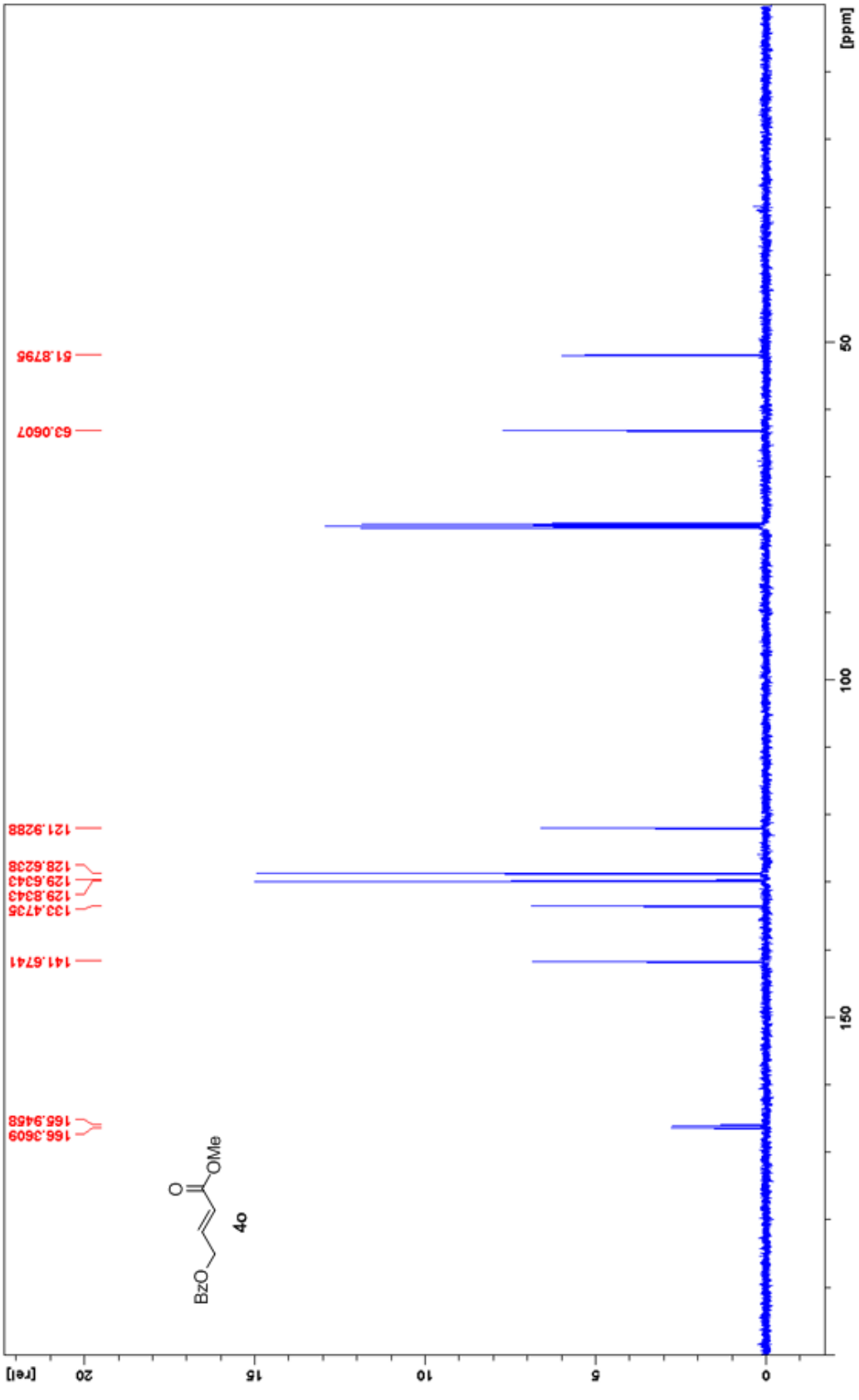
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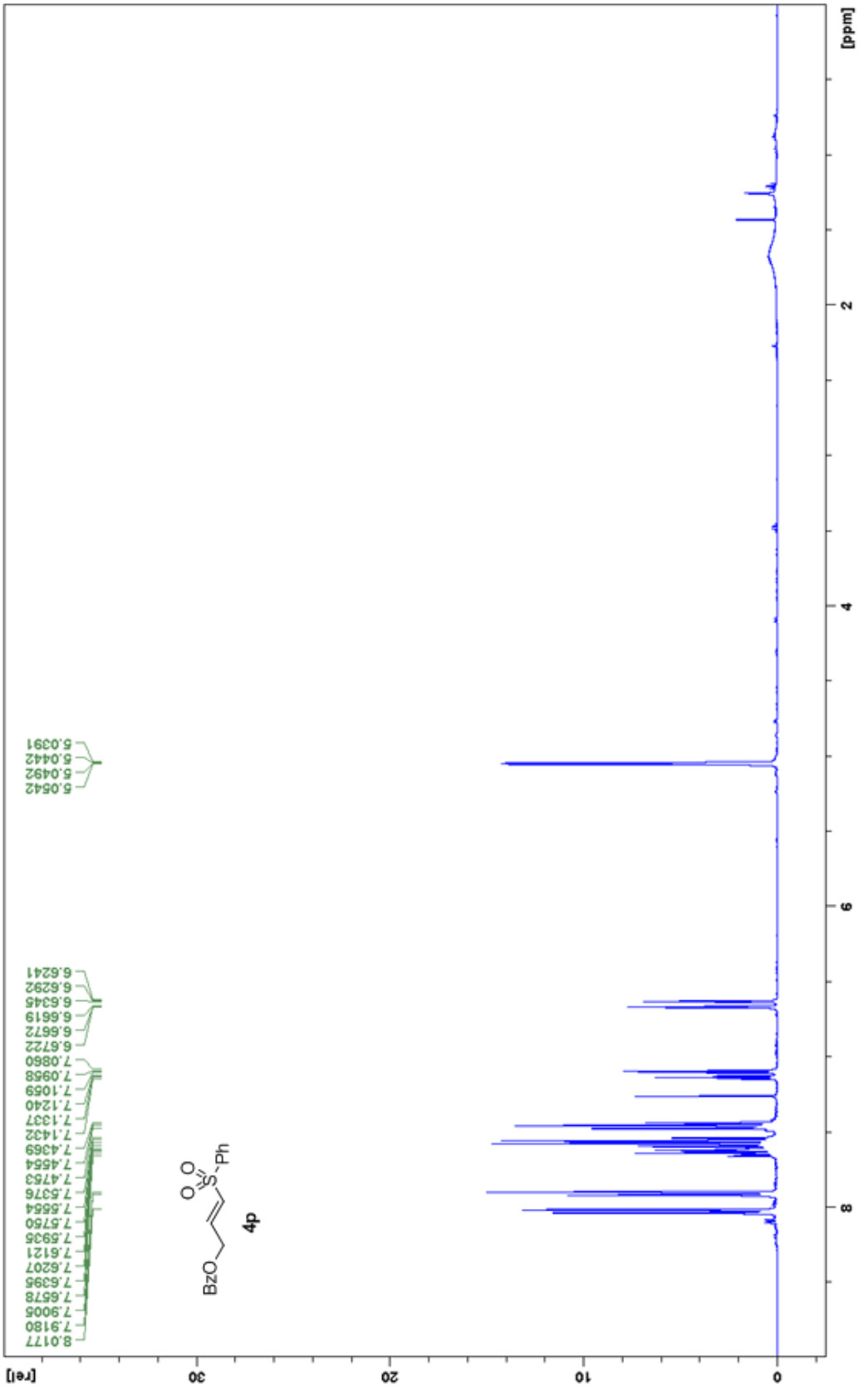
BzO
OMe
O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7.4753
7.4564
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7.1337
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7.1059
7.0958
7.0860
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6.6619
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6.6292
6.6241
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5.0492
5.0442
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O
S
Ph
4p
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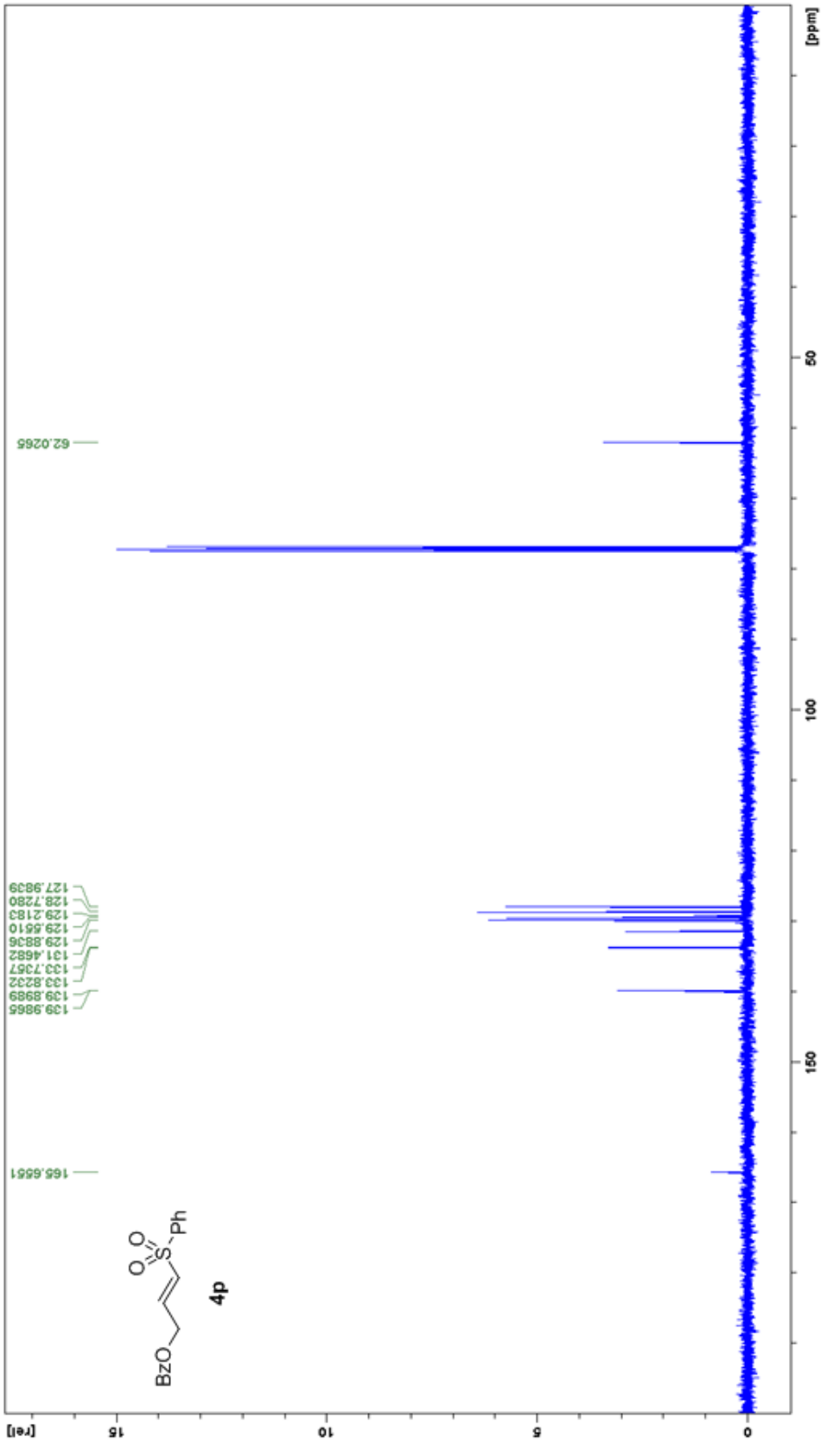
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BzO
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S
Ph
4p