

Nickel N-heterocyclic carbene-catalyzed cross-coupling reaction of aryl aldehydes with organozinc reagents to produce aryl ketones

Cheng Jin^a Lijun Gu ^{*b,c} and Minglong Yuan ^{*c}

^a New United Group Company Limited, Changzhou, Jiangsu, 213166, China; ^b Key Laboratory of Chemistry in Ethnic Medicinal Resources, State Ethnic Affairs Commission & Ministry of Education; ^c Engineering Research Center of Biopolymer Functional Materials of Yunnan, Yunnan Minzu University, Kunming, Yunnan, 650500, China

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(A) Materials and equipment

Reagents were obtained commercially and used as received. Solvents were purified and dried by standard methods. Organozinc reagents (0.5M in THF) were purchased from Beijing InnoChem Science & Technology Co., Ltd. All title products were characterized by Infrared (IR), MS, ¹H NMR, ¹³C NMR and High Resolution mass spectrometer (HRMS). IR spectra were reported in frequency of the absorption (cm⁻¹). ¹H NMR spectra were recorded on 400 MHz in CDCl₃, and ¹³C NMR spectra were recorded on 100 MHz in CDCl₃ using tetramethylsilane (TMS) as an internal standard. Chemical shift values (δ) are given in ppm. Coupling constants (*J*) were measured in Hz. Mass spectra were obtained with ionization voltages of 70 eV. HRMS spectra were obtained by ESI on a TOF mass. 200-300 mesh silica gel was used for column chromatography.

(B) Typical experimental procedure

Typical Experimental Procedure for the Synthesis of compounds **3**:

A 10 mL oven-dried Schlenk tube was charged with **1** (0.3 mmol), **2** (0.3 mmol), **5d** (0.45 mmol), MgCl₂ (0.3 mmol), Ni(cod)₂ (8 mol %), IPr (10 mol %), 1,4-dioxane (1.5 mL), THF (1.5 mL). Then the tube was charged with argon, and was stirred at 30 °C for about 8 h. After the reaction was finished, the reaction mixture was diluted in 5 mL EtOAc. The solution was filtered through a celite pad and washed with 15-20 mL of EtOAc. The organic portion was washed with a saturated solution of NH₄Cl (8 mL×2), brine (8 mL×2), dried (Na₂SO₄) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to provide the desired products **3**.

(C) Screening optimal conditions

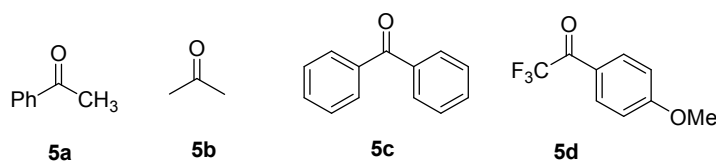
Table S1. Screening optimal conditions

Entry	Ph-CHO + PhZnBr		Solvent, MgCl ₂ hydrogen acceptor Ligand, Ni source		Ph-C(=O)-Ph	Ph-C(=O)-OBn
	1a	2a	hydrogen acceptor	Ligand	3aa	4a
	Ni source	Solvent			Yield ^b (%) (3aa)	Yield ^b (%) (4a)
1	Ni(cod) ₂	1,4-dioxane	5a	IPr	31	38
2	Ni(cod) ₂	1,4-dioxane	5b	IPr	trace	49
3	Ni(cod) ₂	1,4-dioxane	5c	IPr	51	20
4	Ni(cod) ₂	1,4-dioxane	5d	IPr	62	13
5	Ni(cod) ₂	toluene	5d	IPr	trace	34
6	Ni(cod) ₂	THF	5d	IPr	44	28
7	Ni(cod) ₂	EtOEt	5d	IPr	12	19
8	Ni(cod)₂	THF/1,4-	5d	IPr	78	trace

dioxane						
9	NiI ₂ (PPh ₃) ₂	THF/1,4-dioxane	5d	IPr	trace	15
10	NiCl ₂ (dppe) ₂	THF/1,4-dioxane	5d	IPr	9	33
11	Ni(PPh ₃) ₄	THF/1,4-dioxane	5d	IPr	24	36
12	NiCl ₂	THF/1,4-dioxane	5d	IPr	trace	14
13	Ni(cod) ₂	THF/1,4-dioxane	5d	PCy ₃	trace	21
14	Ni(cod) ₂	THF/1,4-dioxane	5d	ItBu	32	10
15	Ni(cod) ₂	THF/1,4-dioxane	5d	SIPr	41	17
16	none	THF/1,4-dioxane	5d	IPr	0	0
17 ^c	Ni(cod) ₂	THF/1,4-dioxane	5d	IPr	46	21

^a Reaction conditions: **1a** (0.3 mmol), **2a** (0.3 mmol), MgCl₂ (0.3 mmol), hydrogen acceptor (1.5 equiv), Ni source (8 mol %), ligands (10 mol %), solvent (3 mL), 30 °C, in Ar atmosphere for 8 h. ^b Isolated yield. ^c Without MgCl₂. cod = 1,5-cyclooctadiene, dppe = 1,2-bis(diphenylphosphino)ethane, IPr = 1,3-bis(2,6-diisopropylphenyl)imidazole-2-ylidene, ItBu = 1,3-Di-tert-butylimidazol-2-ylidene, SIPr = 1,3-bis(2,6-di-isopropylphenyl)imidazolidin-2-ylidene.

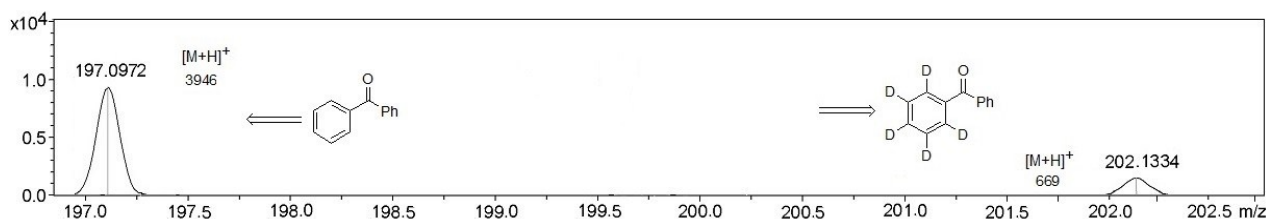
hydrogen acceptor



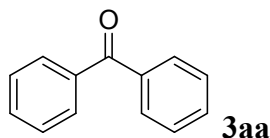
(D) Deuterium-labeling experiment

A 10 mL oven-dried Schlenk tube was charged with **1a** (0.2 mmol), **1a-d₆** (0.2 mmol), **2a** (0.4 mmol), MgCl₂ (0.4 mmol), **5d** (0.6 mmol), Ni(cod)₂ (15 mol %), IPr (20 mol %), 1,4-dioxane (2.0 mL), THF (2.0 mL). Then the tube was charged with argon, and was stirred at 30 °C for about 8 h. After the reaction was finished, the reaction mixture was diluted in 10 mL EtOAc. The solution was filtered through a celite pad and washed with 30 mL of EtOAc. The organic portion was washed with a saturated solution of NH₄Cl (8 mL×2), bine (8 mL×2), dried (Na₂SO₄) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to provide the desired products **3aa** and **3aa-d₅** in a ratio of 5.9:1 (determined by HRMS).

The HRMS spectra of **3aa** and **3aa-d₅**

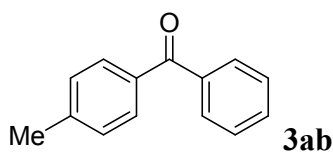


(E) Analytical data



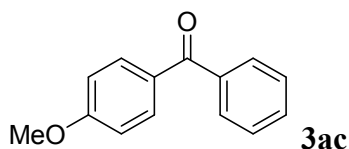
Benzophenone (3aa): ¹

¹H NMR (400 MHz, CDCl₃) δ : 7.83 (dd, $J = 8.0$ Hz, $J = 1.6$ Hz, 4H), 7.61-7.56 (m, 2H), 7.51-7.45 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ : 196.7, 137.8, 132.5, 130.2, 128.4; IR (neat cm⁻¹): 1660 (C=O); LRMS (EI 70 ev) m/z (%): 182 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₃H₁₁O (M+H)⁺ 183.0804, found 183.0801.



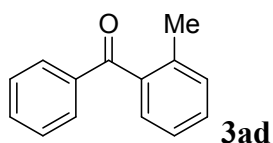
Phenyl(p-tolyl)methanone (3ab): ¹

¹H NMR (400 MHz, CDCl₃) δ : 7.79 (d, $J = 7.2$ Hz, 2H), 7.73 (d, $J = 8.0$ Hz, 2H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.48 (t, $J = 7.6$ Hz, 2H), 7.28 (d, $J = 8.0$ Hz, 2H), 2.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 196.4, 143.2, 137.9, 134.8, 132.1, 130.2, 129.8, 128.9, 128.1, 21.6; IR (neat cm⁻¹): 1658 (C=O); LRMS (EI 70 ev) m/z (%): 196 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₄H₁₃O (M+H)⁺ 197.0960, found 197.0963.



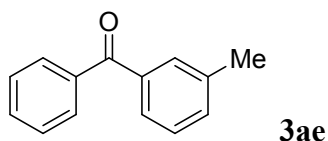
(4-Methoxyphenyl)(phenyl)methanone (3ac): ¹

¹H NMR (400 MHz, CDCl₃) δ : 7.80 (d, $J = 8.4$ Hz, 2H), 7.73 (d, $J = 8.0$ Hz, 2H), 7.51-7.45 (m, 3H), 6.96 (d, $J = 8.4$ Hz, 2H), 3.91 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 197.1, 163.2, 138.2, 132.4, 131.7, 130.0, 129.5, 128.2, 113.6, 55.8; IR (neat cm⁻¹): 1652 (C=O); LRMS (EI 70 ev) m/z (%): 212 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₄H₁₃O₂ (M+H)⁺ 213.0909, found 213.0913.



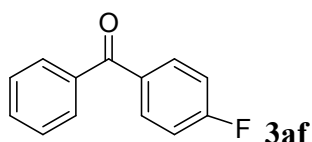
Phenyl(o-tolyl)methanone (3ad): ¹

¹H NMR (400 MHz, CDCl₃) δ : 7.74 (d, J = 7.2 Hz, 2H), 7.54-7.50 (m, 1H), 7.43-7.36 (m, 2H), 7.33-7.26 (m, 1H), 7.25-7.20 (m, 3H); 2.33 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 198.5, 138.8, 138.1, 137.0, 133.5, 131.7, 130.6, 130.3, 129.0, 128.8, 125.4, 20.4; IR (neat cm⁻¹): 1647 (C=O); LRMS (EI 70 ev) m/z (%): 196 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₄H₁₃O (M+H)⁺ 197.0960, found 197.0961.



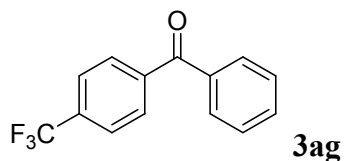
Phenyl(m-tolyl)methanone (3ae): ²

¹H NMR (400 MHz, CDCl₃) δ : 7.81 (dd, J = 1.2 Hz, J = 8.4 Hz, 2H), 7.62-7.57 (m, 3H), 7.46-7.40 (m, 2H), 7.38 (dd, J = 4.4 Hz, J = 4.4 Hz, 2H), 2.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 196.8, 138.1, 137.4, 137.1, 133.0, 132.1, 130.6, 130.1, 128.4, 128.0, 127.2, 21.3; IR (neat cm⁻¹): 1663 (C=O); LRMS (EI 70 ev) m/z (%): 196 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₄H₁₃O (M+H)⁺ 197.0960, found 197.0954.



(4-Fluorophenyl)(phenyl)methanone (3af): ¹

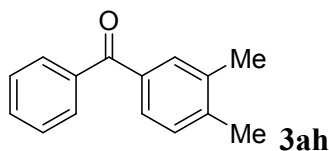
¹H NMR (400 MHz, CDCl₃) δ : 7.86-7.83 (m, 2H), 7.78 (d, J = 4.2 Hz, 2H), 7.62 (dd, J = 7.2 Hz, J = 1.2 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.18 (t, J = 8.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 195.5, 165.5, 162.9, 137.6, 132.7, 132.7, 132.6, 132.4, 132.0, 129.8, 128.3, 115.5, 115.3; IR (neat cm⁻¹): 1661 (C=O); LRMS (EI 70 ev) m/z (%): 200 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₃H₁₀FO (M+H)⁺ 201.0710, found 201.0719.



4-Trifluoromethylbenzophenone (3ag): ¹

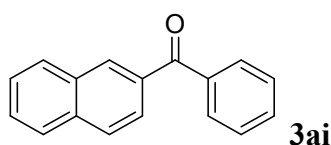
¹H NMR (400 MHz, CDCl₃) δ : 7.90 (d, J = 8.0 Hz, 2H), 7.81 (dd, J = 7.6 Hz, J = 8.0 Hz, 4H),

7.65 (t, $J = 7.2$ Hz, 2H), 7.53 (t, $J = 7.4$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 195.5, 140.6, 136.6, 133.8, 133.5, 133.0, 130.1, 130.0, 128.5, 125.38, 125.34, 125.31, 125.2, 125.0, 122.2; IR (neat cm^{-1}): 1660 (C=O); LRMS (EI 70 ev) m/z (%): 250 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{14}\text{H}_{10}\text{F}_3\text{O}$ ($\text{M}+\text{H}$) $^+$ 251.0672, found 251.0667.



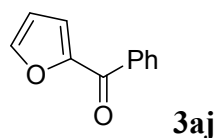
(3,4-Dimethylphenyl)(phenyl)methanone (3ah): ³

^1H NMR (400 MHz, CDCl_3) δ : 7.79 (t, $J = 4.2$ Hz, 2H), 7.61 (s, 1H), 7.59-7.52 (m, 2H), 7.49 (t, $J = 7.6$ Hz, 2H), 7.23 (d, $J = 7.6$ Hz, 1H), 2.35 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 198.3, 141.9, 138.0, 136.7, 135.3, 132.0, 131.1, 129.9, 129.4, 128.1, 128.0, 20.0, 19.7; IR (neat cm^{-1}): 1661 (C=O); LRMS (EI 70 ev) m/z (%): 210 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{15}\text{H}_{15}\text{O}$ ($\text{M}+\text{H}$) $^+$ 211.1116, found 211.1111.



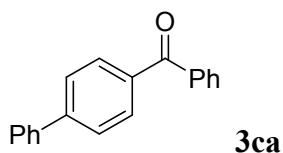
(Naphthalen-6-yl)(phenyl)methanone (3ai): ⁴

^1H NMR (400 MHz, CDCl_3) δ : 8.23 (s, 1H), 7.98-7.84 (m, 6H), 7.63-7.56 (m, 2H), 7.52-7.44 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 196.1, 137.8, 135.1, 134.6, 132.4, 32.1, 131.7, 130.0, 129.4, 128.5, 128.3, 128.1, 127.7, 126.7, 125.6; IR (neat cm^{-1}): 1660 (C=O); LRMS (EI 70 ev) m/z (%): 232 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{17}\text{H}_{13}\text{O}$ ($\text{M}+\text{H}$) $^+$ 233.0959, found 233.0961.



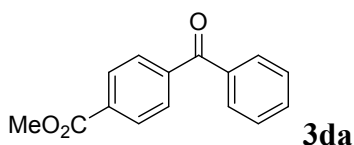
(Furan-2-yl)(phenyl)methanone (3aj): ⁵

^1H NMR (400 MHz, CDCl_3) δ : 7.99-7.94 (m, 2H), 7.72 (dd, $J = 2.4$ Hz, $J = 1.2$ Hz, 1H), 7.64-7.57 (m, 1H), 7.51-7.44 (m, 2H), 7.24 (dd, $J = 4.8$ Hz, $J = 1.2$ Hz, 1H), 6.61 (dd, $J = 4.8$ Hz, $J = 2.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 182.8, 152.2, 147.2, 137.1, 132.2, 129.2, 128.4, 120.7, 112.5; IR (neat cm^{-1}): 1630 (C=O); LRMS (EI 70 ev) m/z (%): 172 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{11}\text{H}_9\text{O}_2$ ($\text{M}+\text{H}$) $^+$ 173.0637, found 173.0648.



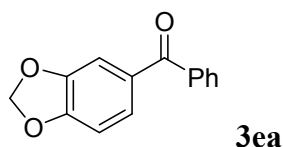
Biphenyl-4-yl(phenyl)methanone (3ca): ⁶

¹H NMR (400 MHz, CDCl₃) δ : 7.90-7.87 (m, 2H), 7.84-7.80 (m, 2H), 7.71-7.59 (m, 5H), 7.50-7.39 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ : 196.4, 144.9, 140.0, 137.5, 136.2, 132.1, 130.7, 139.9, 129.0, 128.4, 128.1, 127.2, 127.7; IR (neat cm⁻¹): 1651 (C=O); LRMS (EI 70 ev) m/z (%): 258 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₉H₁₅O (M+H)⁺ 259.1124, found 259.1127.



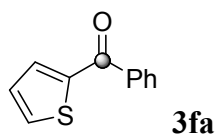
Methyl 4-Benzoylbenzoate (3da): ⁷

¹H NMR (400 MHz, CDCl₃) δ : 8.14 (d, J = 8.4 Hz, 2H), 7.81 (d, J = 8.4 Hz, 2H), 7.78-7.76 (m, 2H), 7.60-7.56 (m, 1H), 7.48 (t, J = 7.6 Hz, 2H), 3.96 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 196.1, 166.2, 141.0, 137.0, 133.1, 132.8, 130.0, 129.6, 129.3, 128.2, 52.1; IR (neat cm⁻¹): 1731 (C=O), 1652 (C=O); LRMS (EI 70 ev) m/z (%): 240 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₅H₁₃O₃ (M+H)⁺ 241.0861, found 241.0855.



(3,4-Methylenedioxyphenyl)phenylmethanone (3ea): ⁸

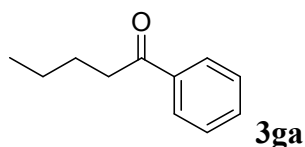
¹H NMR (400 MHz, CDCl₃) δ : 7.76-7.70 (m, 2H), 7.57-7.51 (m, 1H), 7.47-7.42 (m, 2H), 7.37-7.32 (m, 2H), 6.85 (d, J = 8.4 Hz, 1H), 6.04 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 195.2, 151.7, 148.3, 138.4, 132.3, 132.1, 129.9, 128.1, 127.3, 110.2, 108.0, 102.1; IR (neat cm⁻¹): 1663 (C=O); LRMS (EI 70 ev) m/z (%): 226 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₄H₁₁O₃ (M+H)⁺ 227.0703, found 227.0698.



Phenyl(thiophen-2-yl)methanone (3fa): ⁹

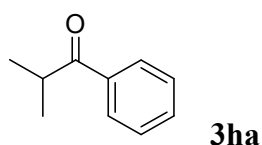
¹H NMR (400 MHz, CDCl₃) δ : 7.87 (d, J = 7.6 Hz, 2H), 7.73 (d, J = 4.8 Hz, 1H), 7.65 (d, J = 3.6

Hz, 1H), 7.61 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.8$ Hz, 2H), 7.17 (t, $J = 4.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 188.2, 143.6, 138.1, 134.8, 134.1, 132.2, 129.1, 128.3, 127.9; IR (neat cm^{-1}): 1638 (C=O); LRMS (EI 70 ev) m/z (%): 188 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{11}\text{H}_9\text{OS}$ ($\text{M} + \text{H}$) $^+$ 189.0368, found 189.0361.



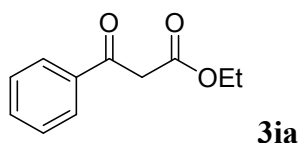
1-phenylpentan-1-one (3ga):⁴

^1H NMR (400 MHz, CDCl_3) δ : 7.97-7.93 (m, 2H), 7.56-7.52 (m, 1H), 7.47-7.43 (m, 2H); 2.93 (t, $J = 7.2$ Hz, 2H), 1.76-1.68 (m, 2H), 1.46-1.36 (m, 2H), 0.90 (t, $J = 7.24$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 200.2, 137.4, 132.5, 128.4, 128.0, 38.4, 26.7, 22.4, 13.9; IR (neat cm^{-1}): 1671 (C=O); LRMS (EI 70 ev) m/z (%): 162 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{11}\text{H}_{15}\text{O}$ ($\text{M} + \text{H}$) $^+$ 163.1117, found 163.1113.



2-Methyl-1-phenylpropan-1-one (3ha):¹

^1H NMR (400 MHz, CDCl_3) δ : 7.96-7.92 (m, 2H), 7.58-7.52 (m, 1H), 7.48-7.42 (m, 2H); 3.69-3.54 (m, 1H), 1.26 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 204.1, 136.0, 132.4, 128.6, 128.1, 35.0, 18.9; IR (neat cm^{-1}): 1677 (C=O); LRMS (EI 70 ev) m/z (%): 148 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{10}\text{H}_{13}\text{O}$ ($\text{M} + \text{H}$) $^+$ 149.0961, found 149.0967.



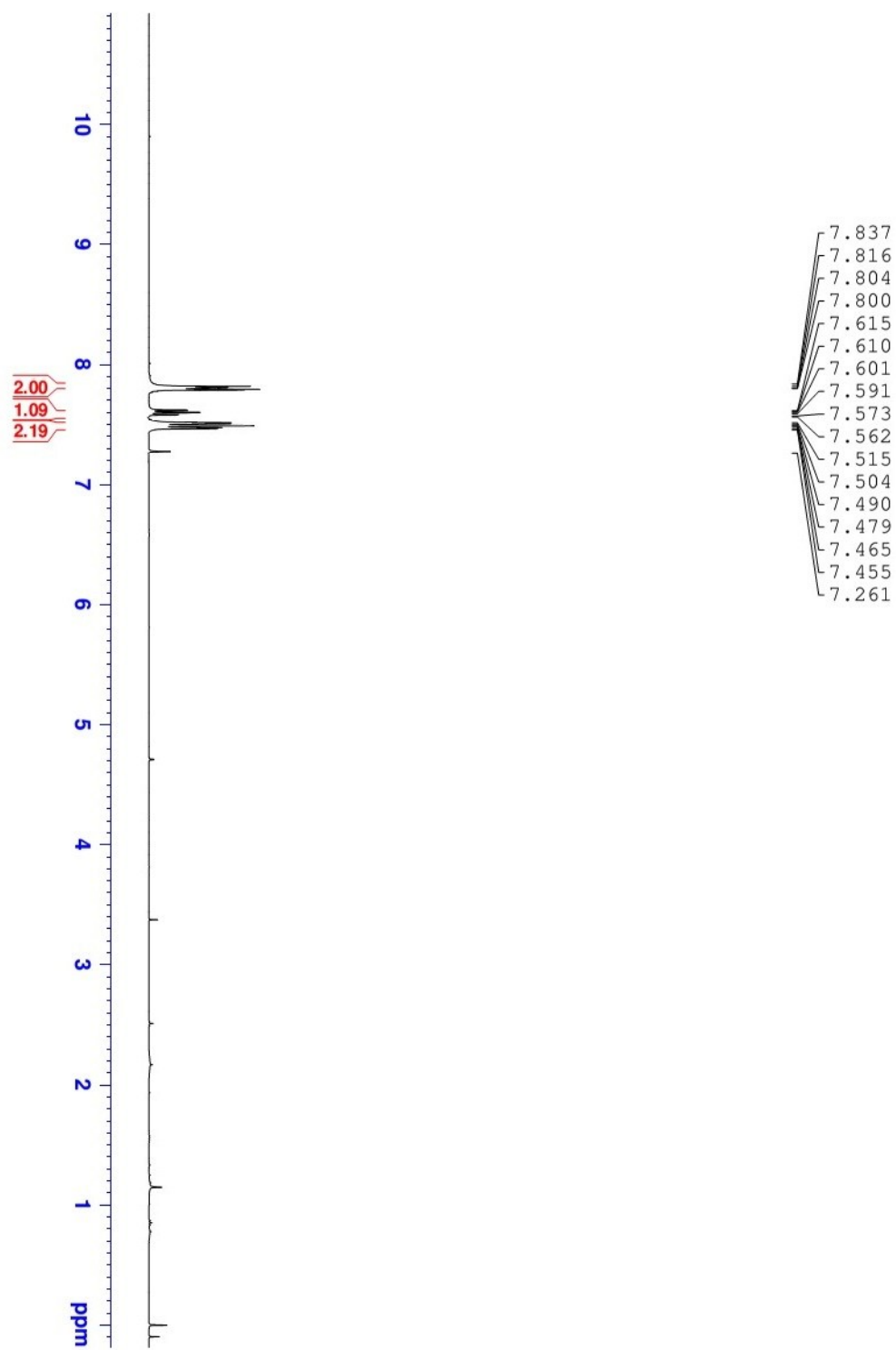
Ethyl 3-oxo-3-phenylpropanoate (3ia):¹⁰

^1H NMR (400 MHz, CDCl_3) δ : 7.89 (d, $J = 7.6$ Hz, 2H), 7.54 (t, $J = 7.4$ Hz, 1H), 7.42-7.32 (m, 2H), 4.22-4.11 (m, 2H), 3.93 (s, 2H), 1.20 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 192.3, 167.3, 135.6, 133.4, 128.5, 128.2, 61.1, 45.6, 13.8; IR (neat cm^{-1}): 1711 (C=O), 1669 (C=O); LRMS (EI 70 ev) m/z (%): 192 (M^+ , 100); HRMS m/z (ESI) calcd for $\text{C}_{11}\text{H}_{13}\text{O}$ ($\text{M} + \text{H}$) $^+$ 193.0859, found 193.0863.

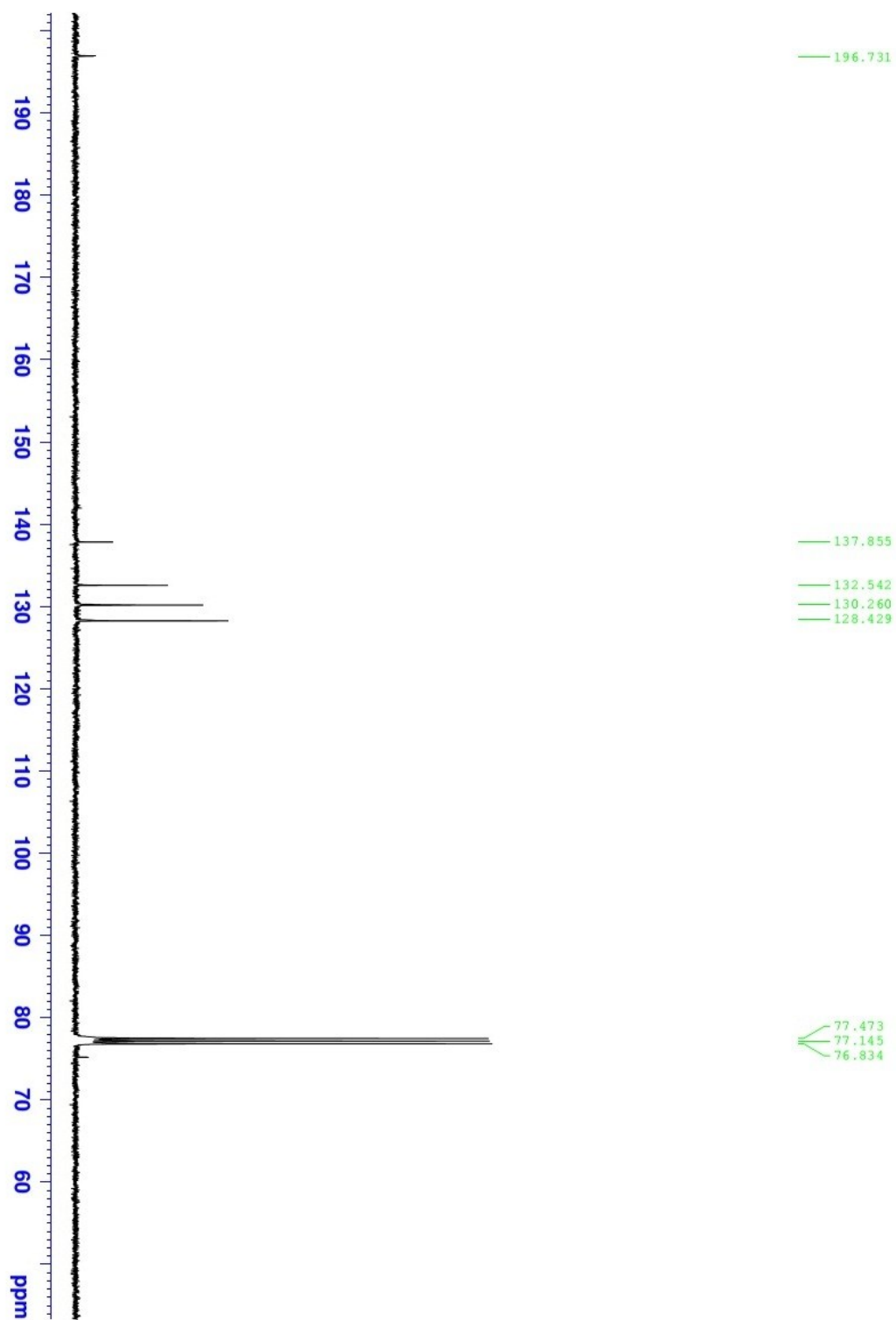
(F) References

- 1) M. Li, C. Wang, H. Ge, *Org. Lett.* **2011**, *13*, 2062.
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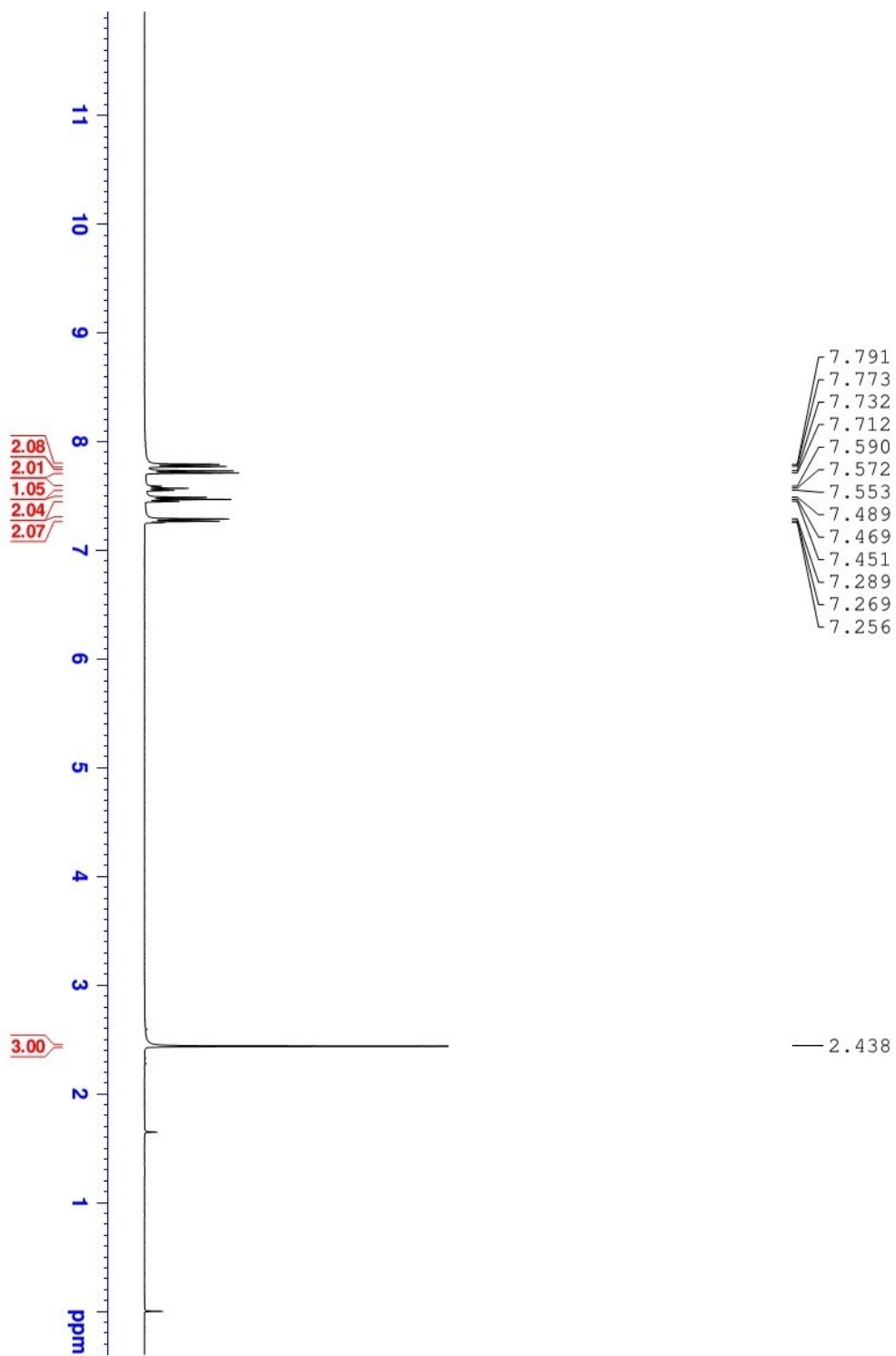
(G) Spectra



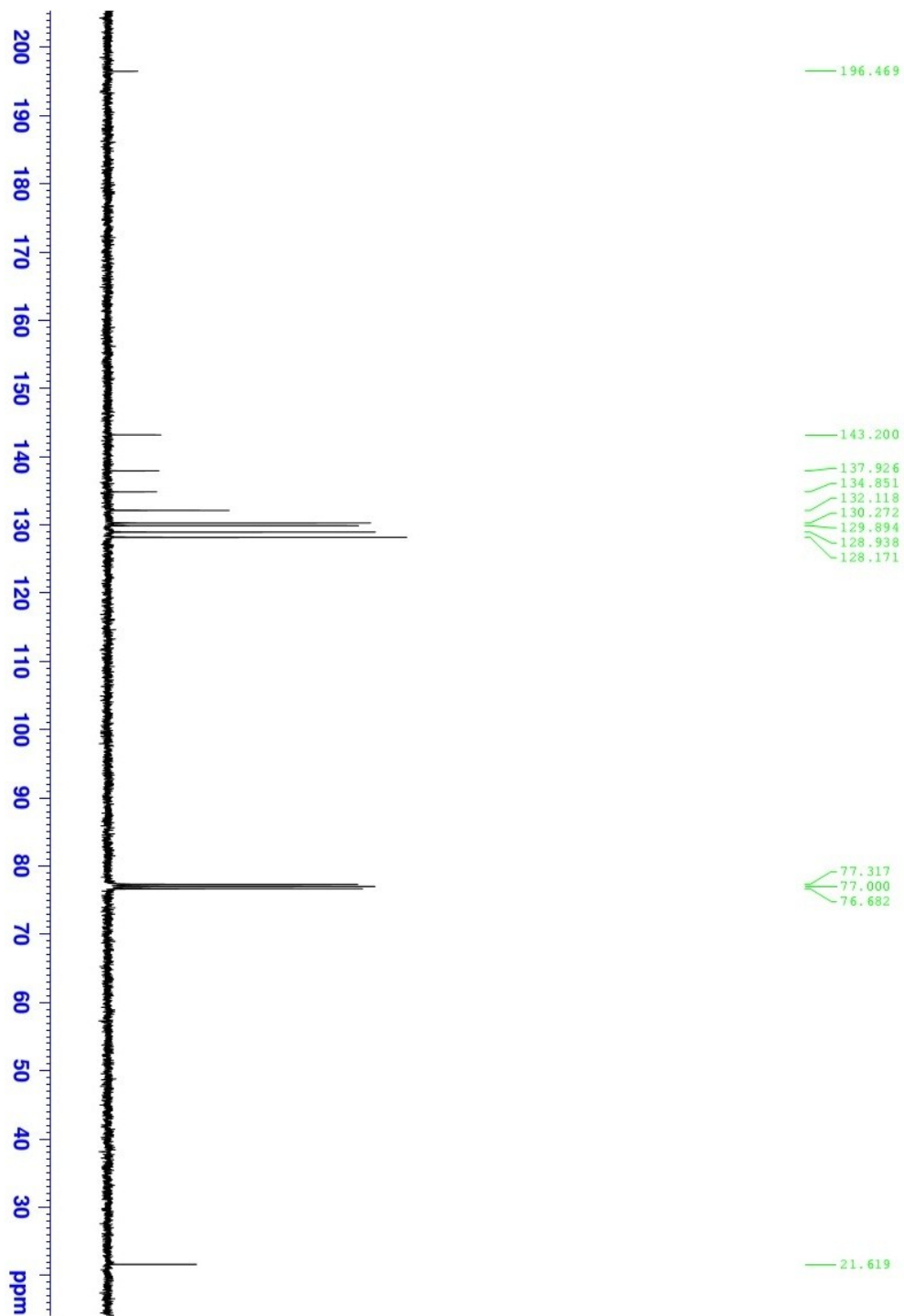
¹H NMR of Compound 3aa



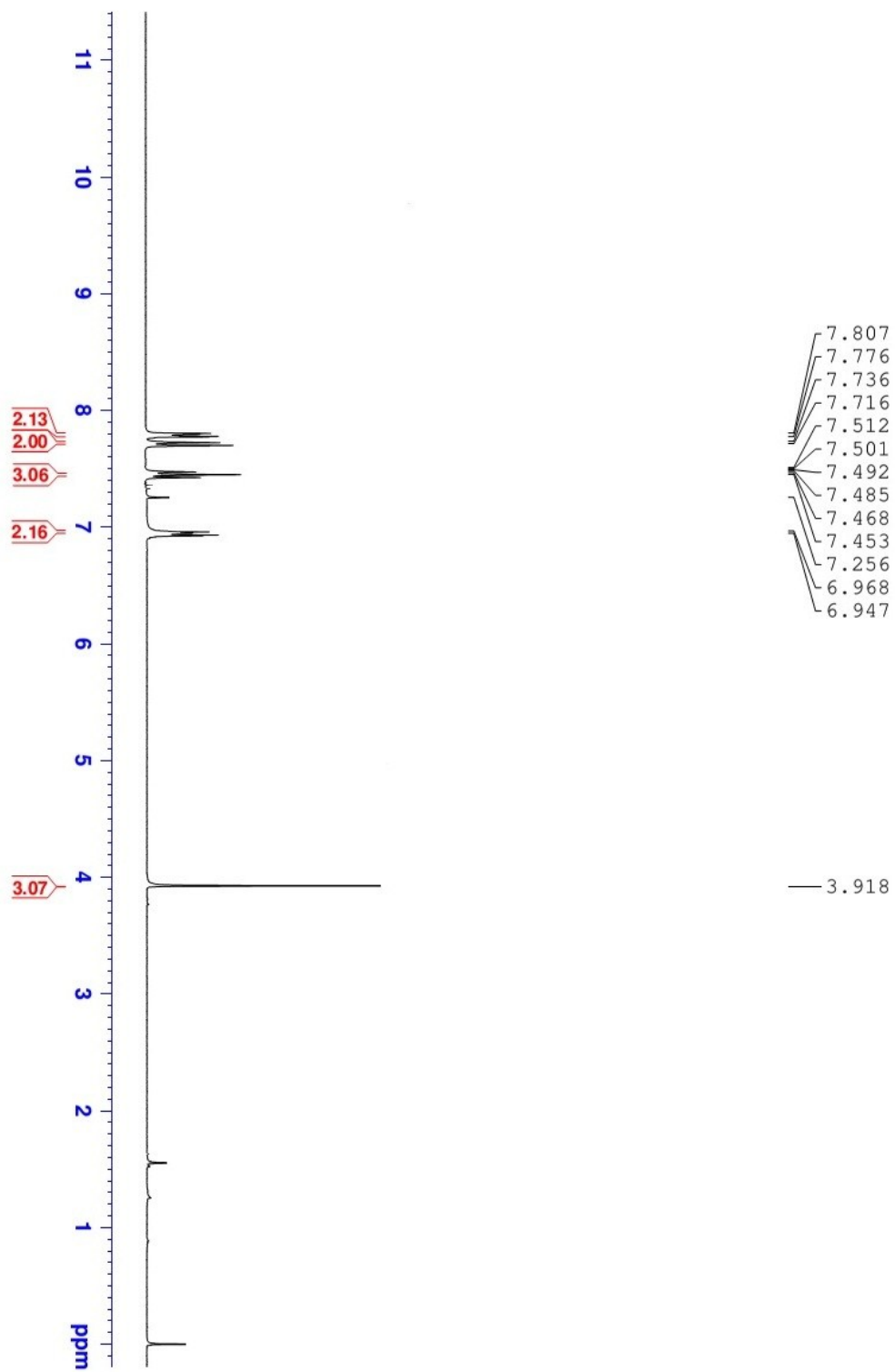
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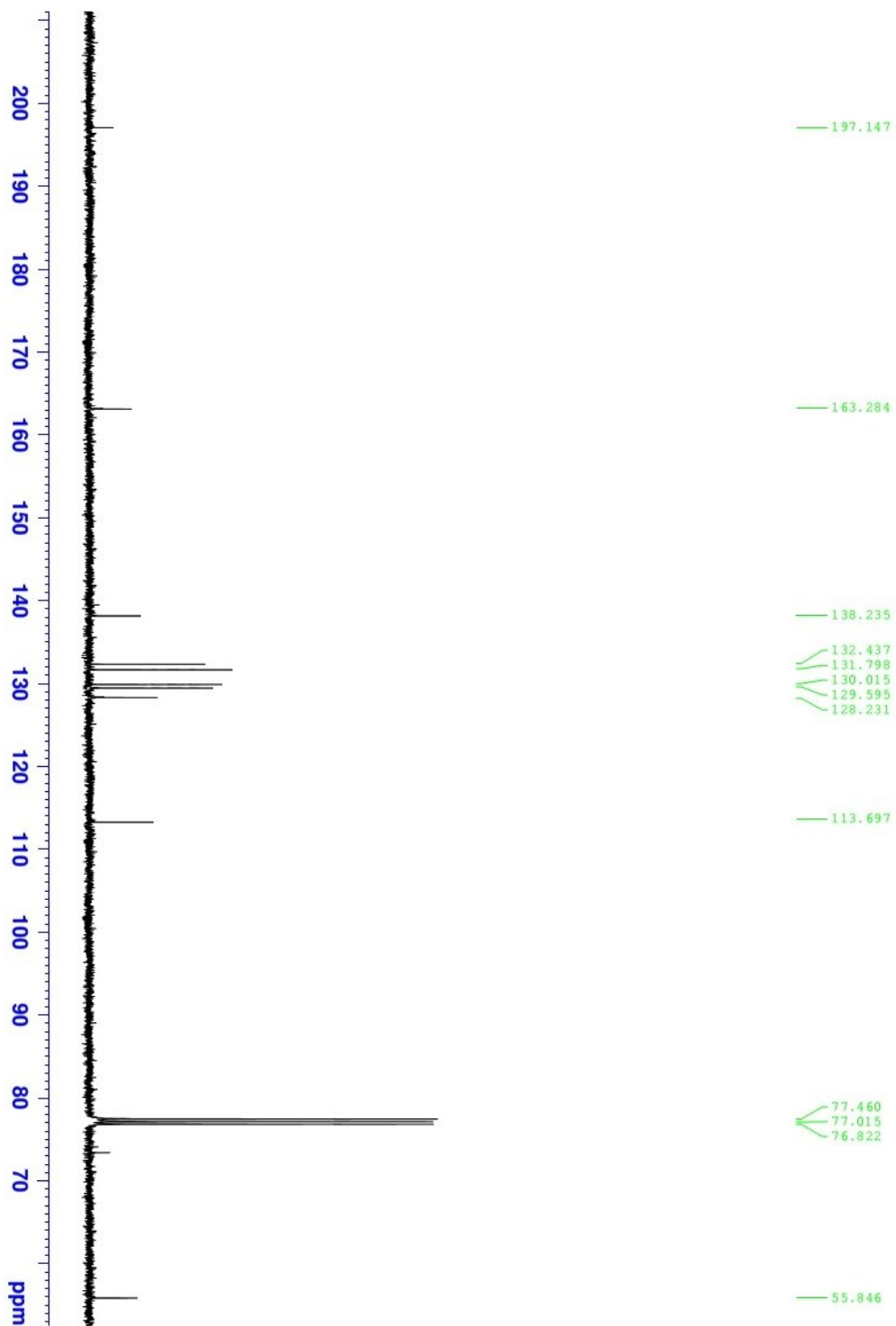
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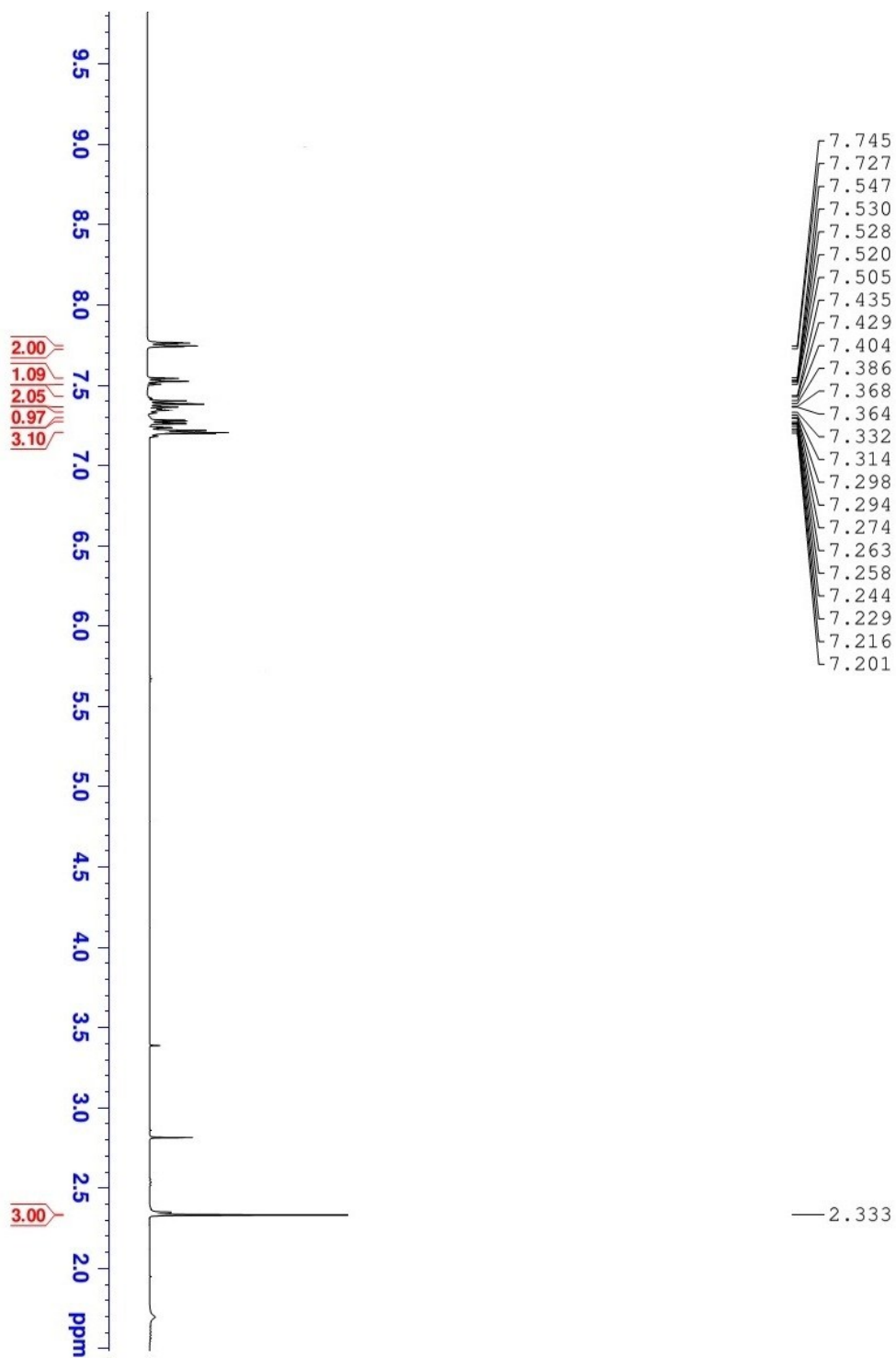
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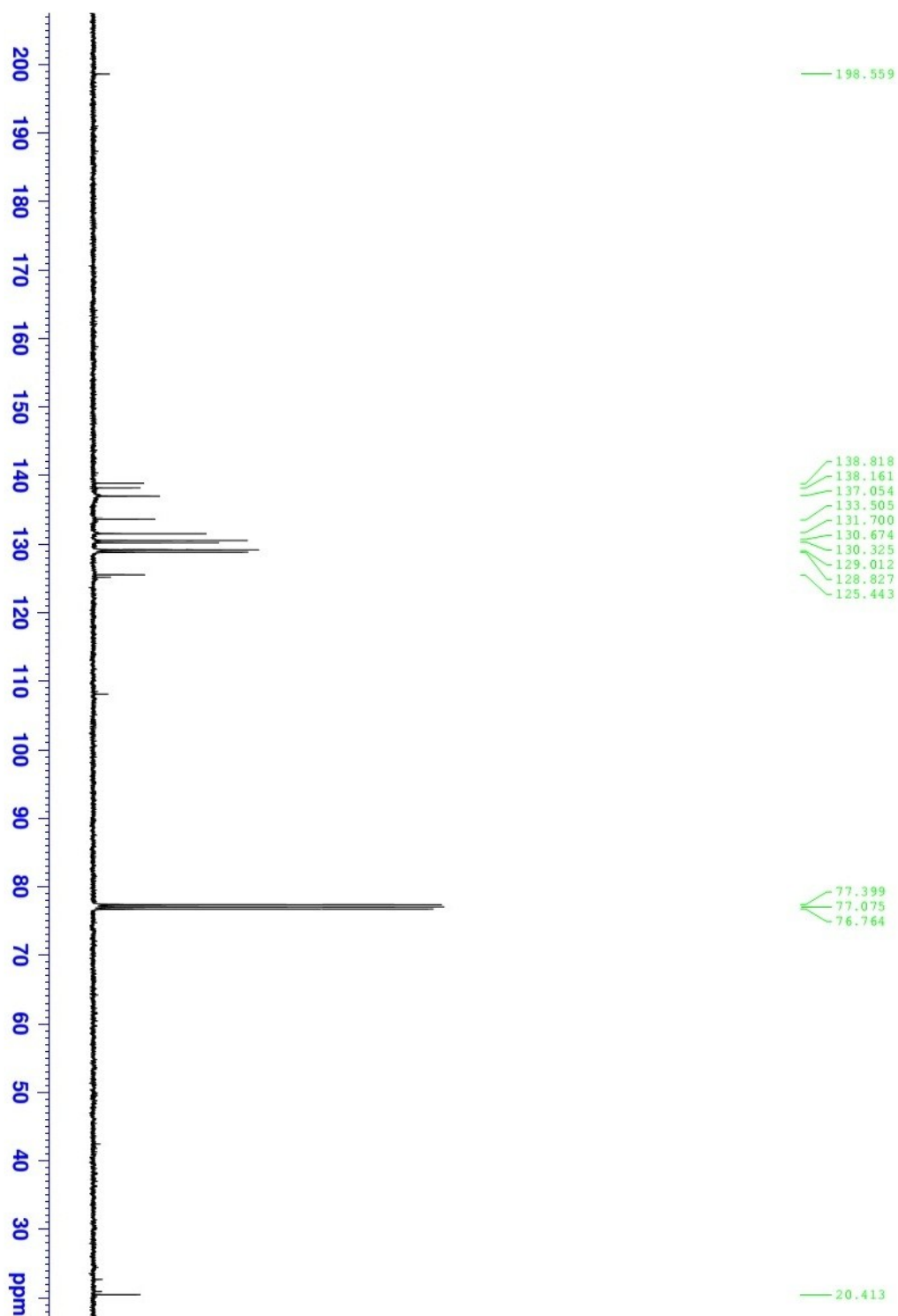
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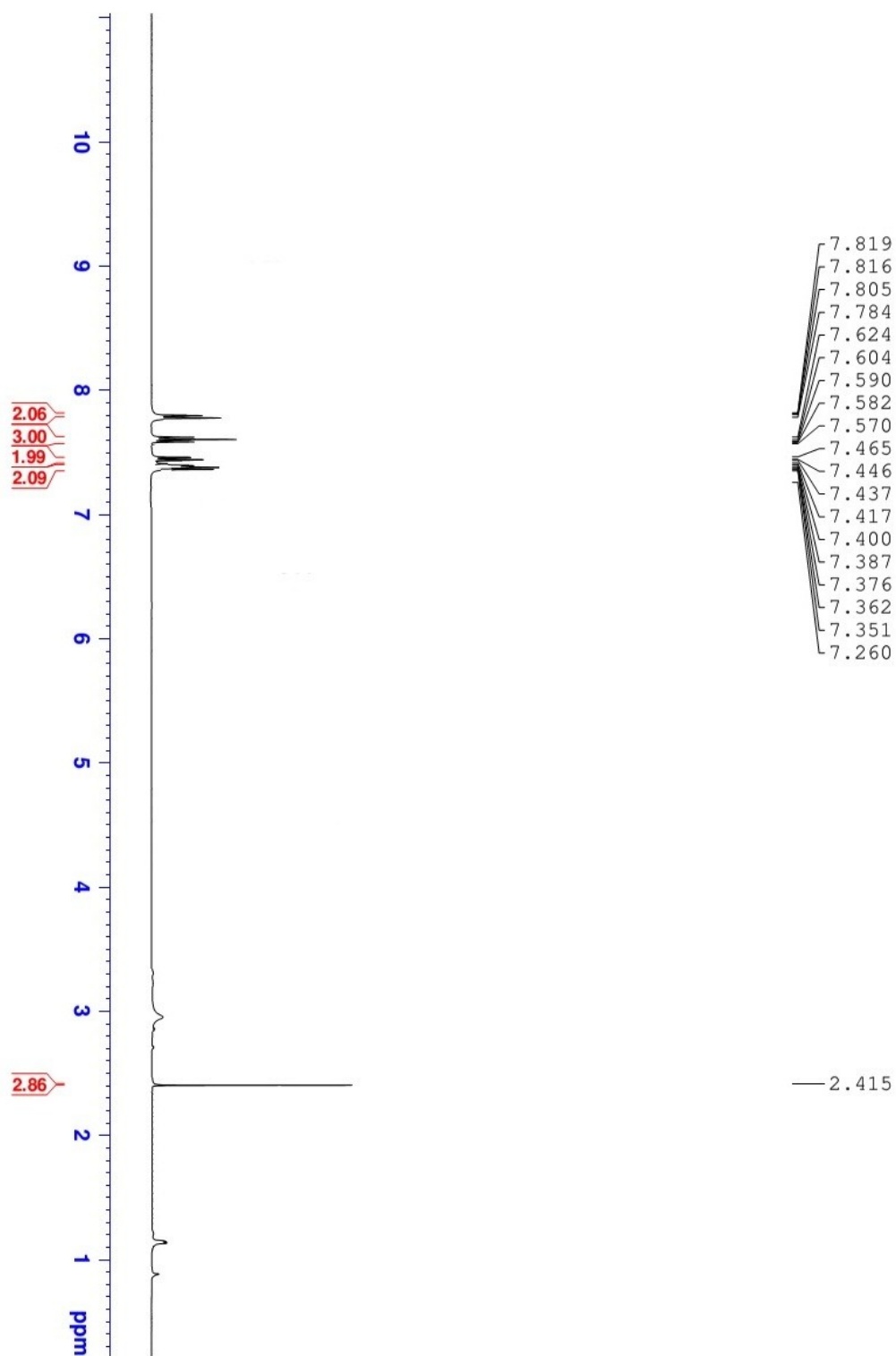
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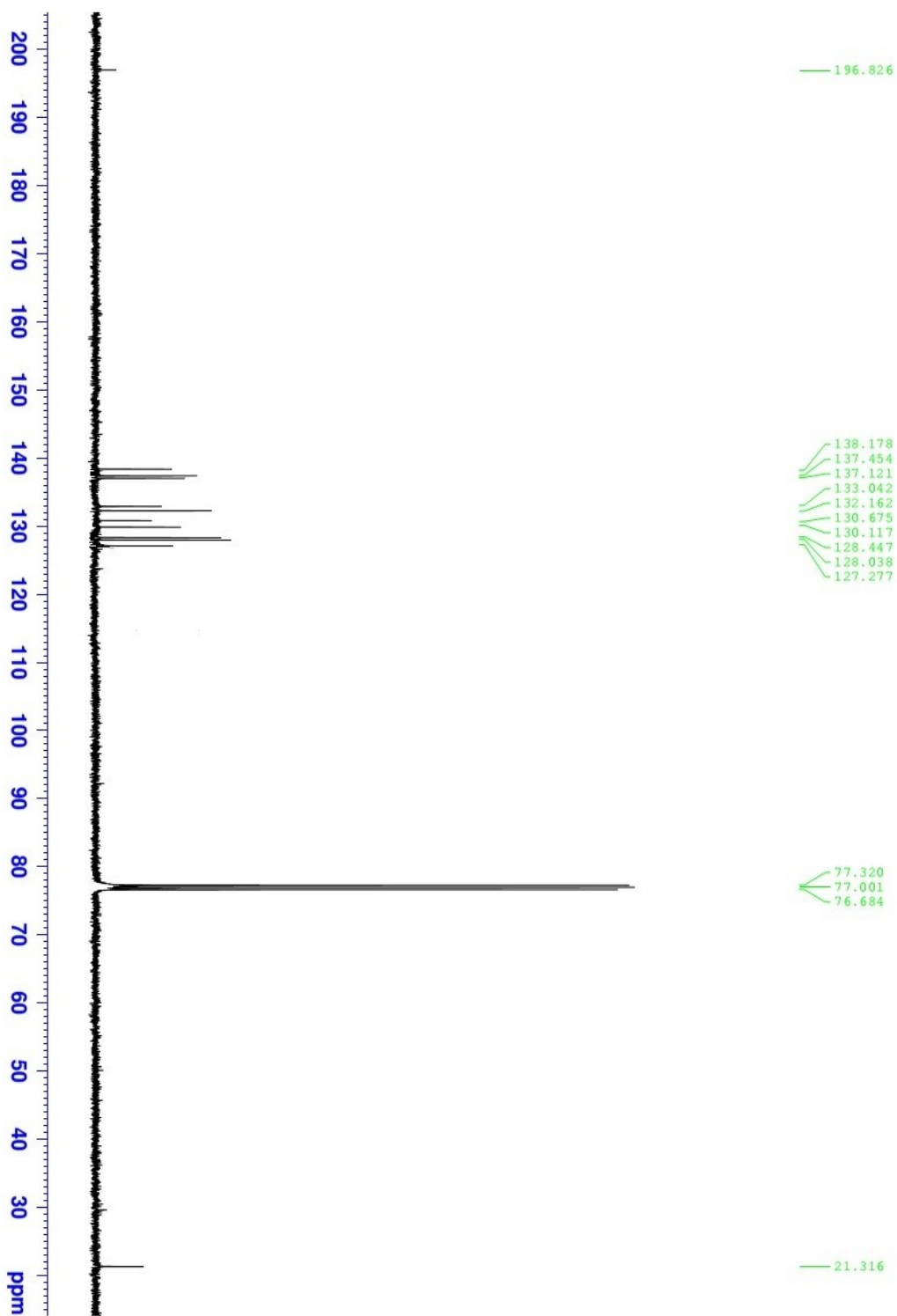
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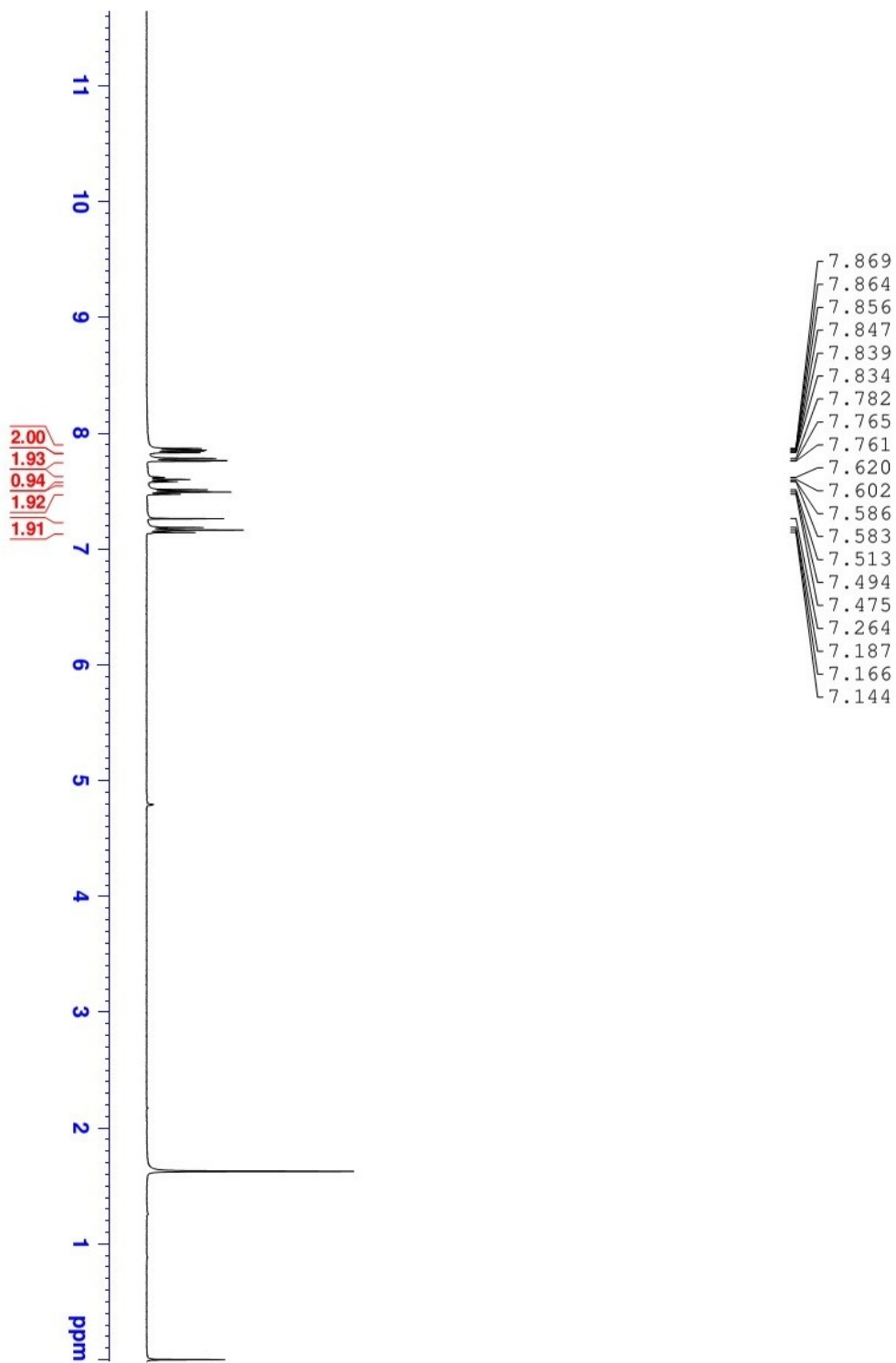
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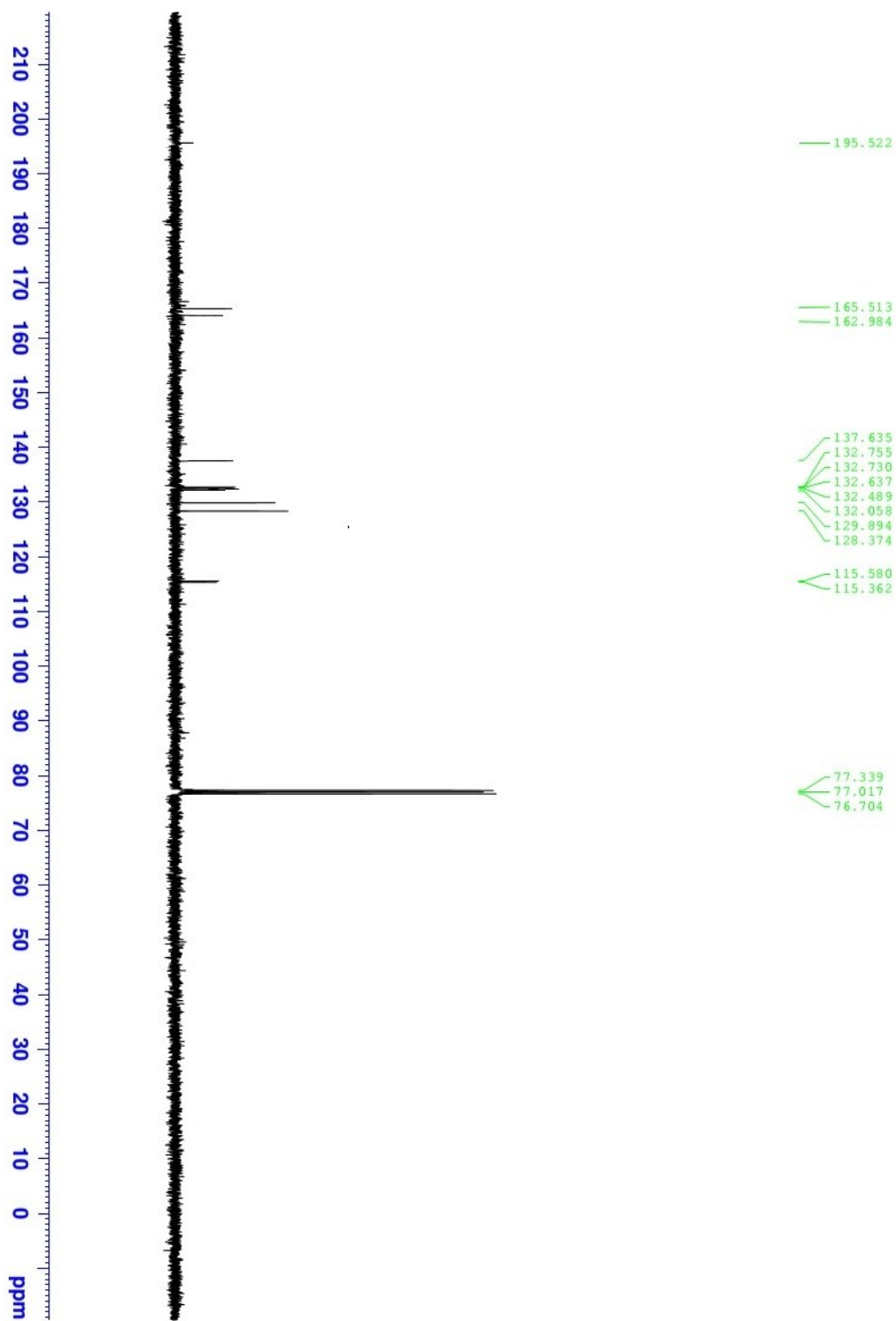
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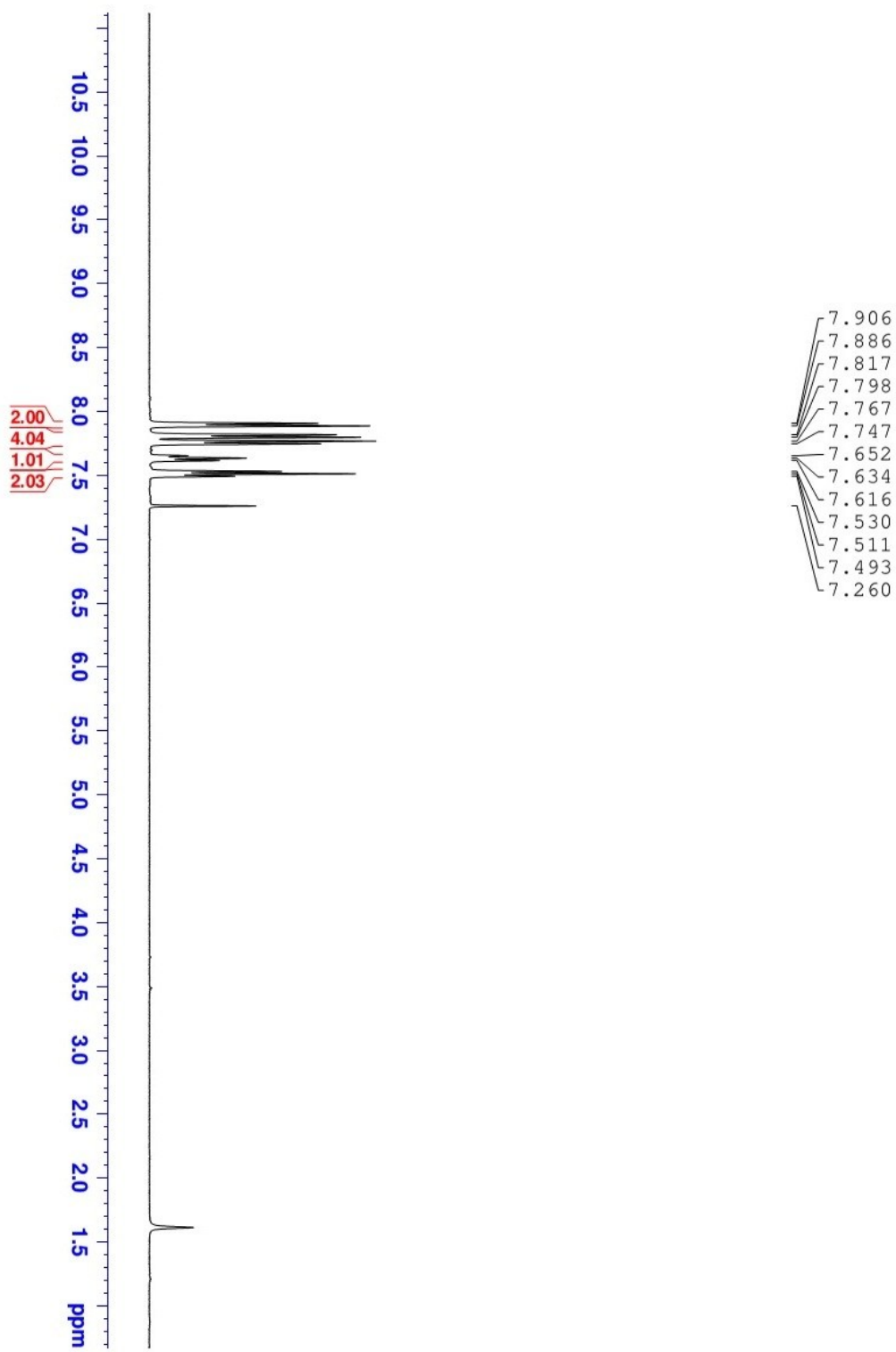
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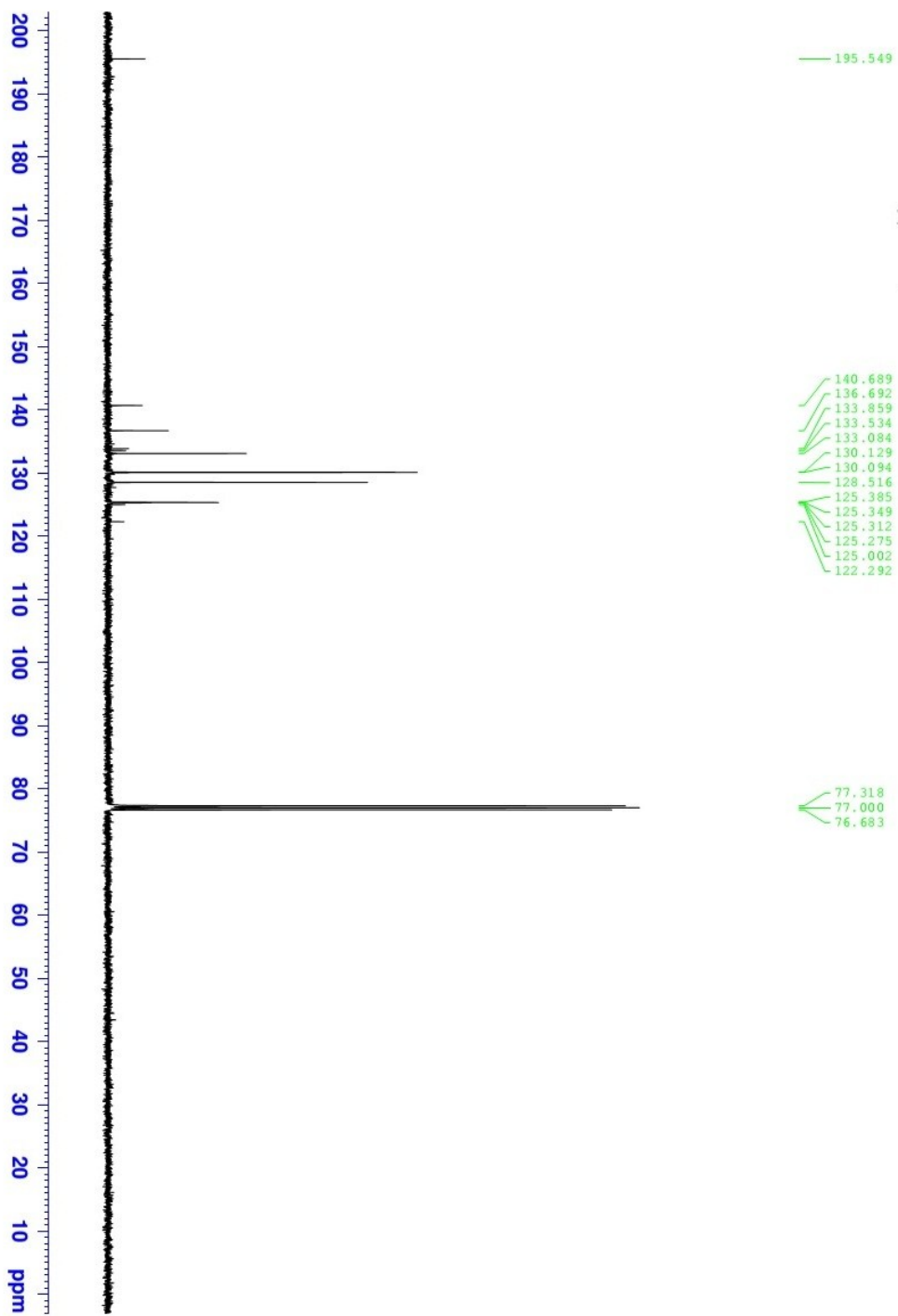
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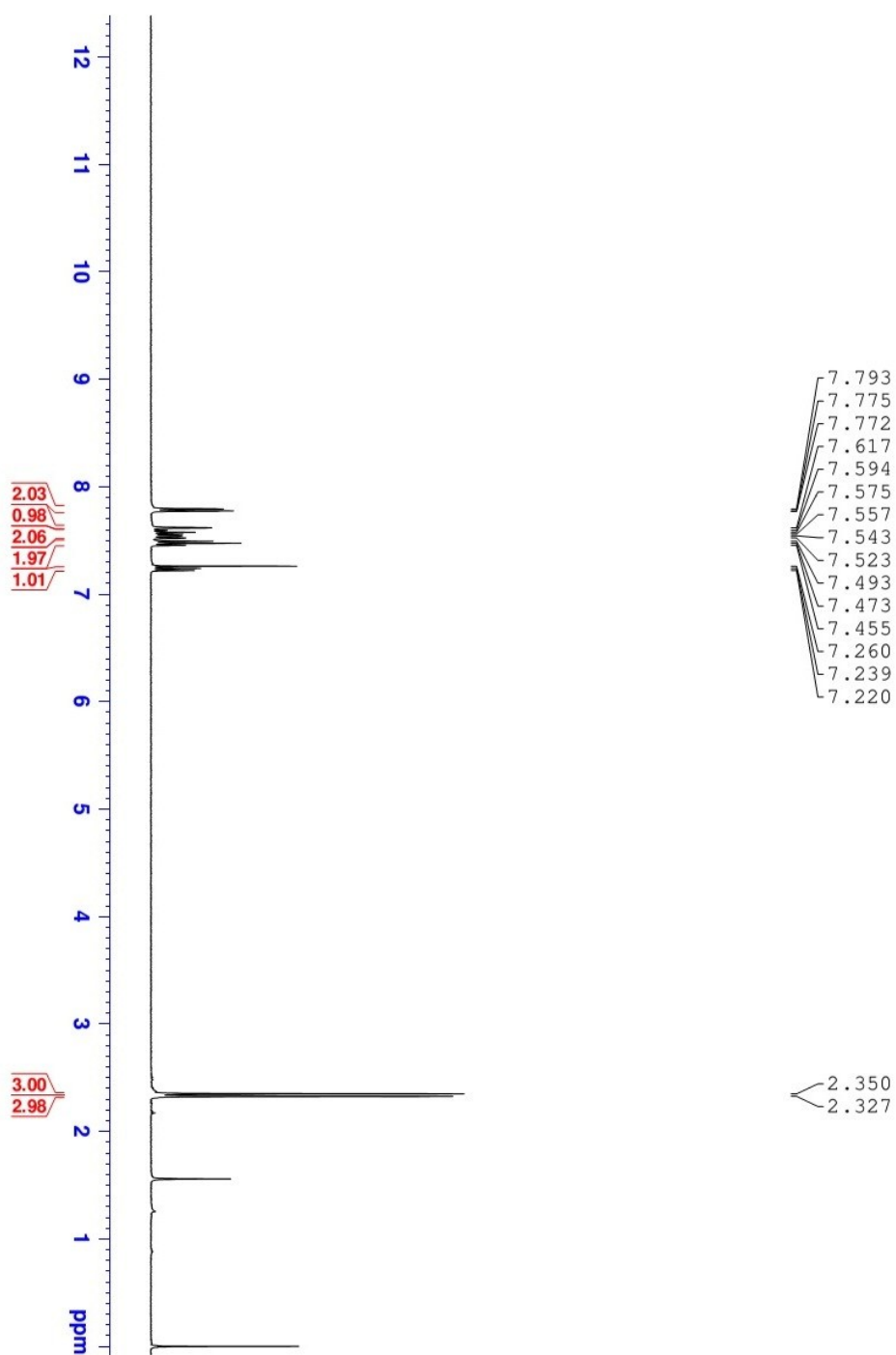
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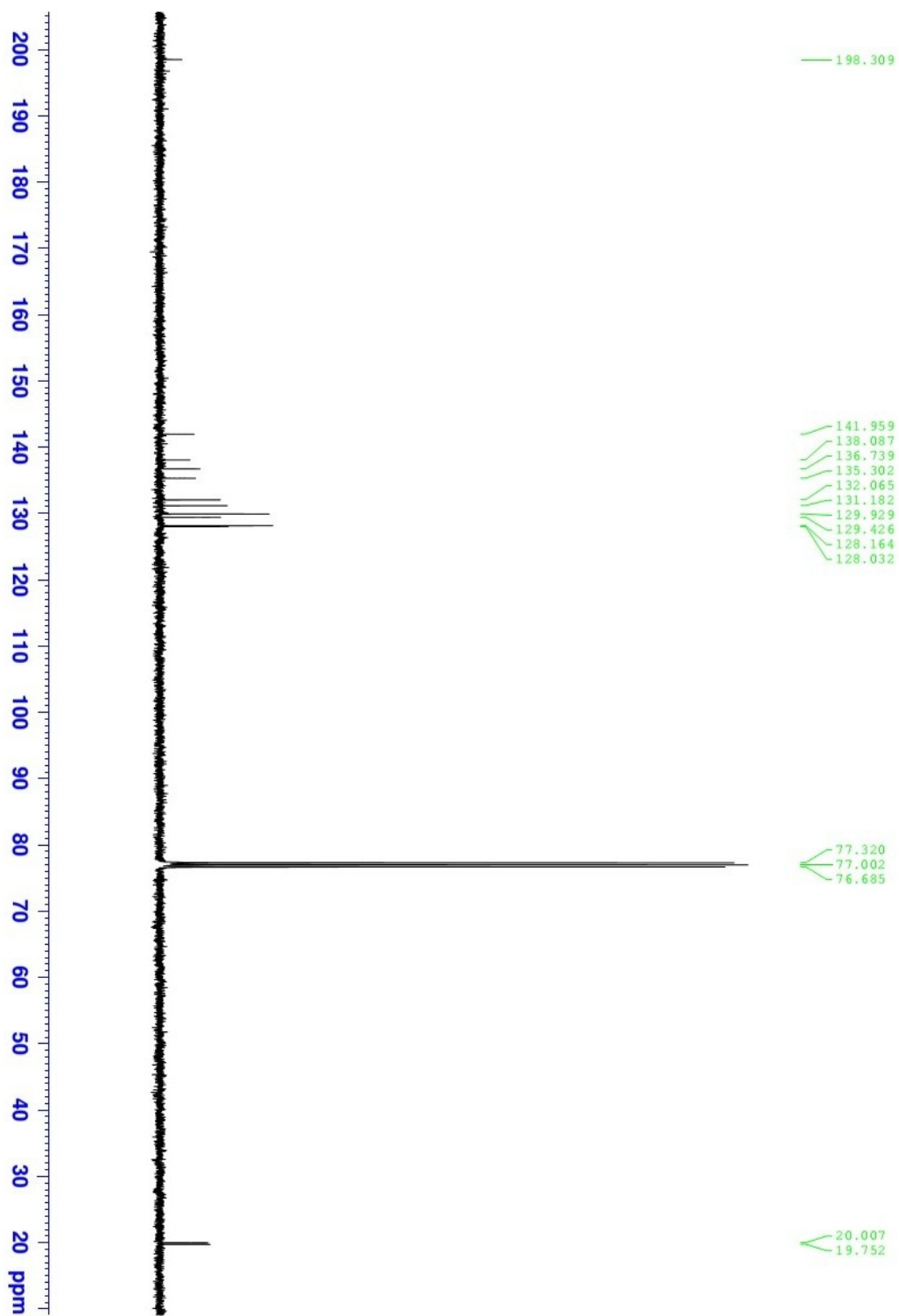
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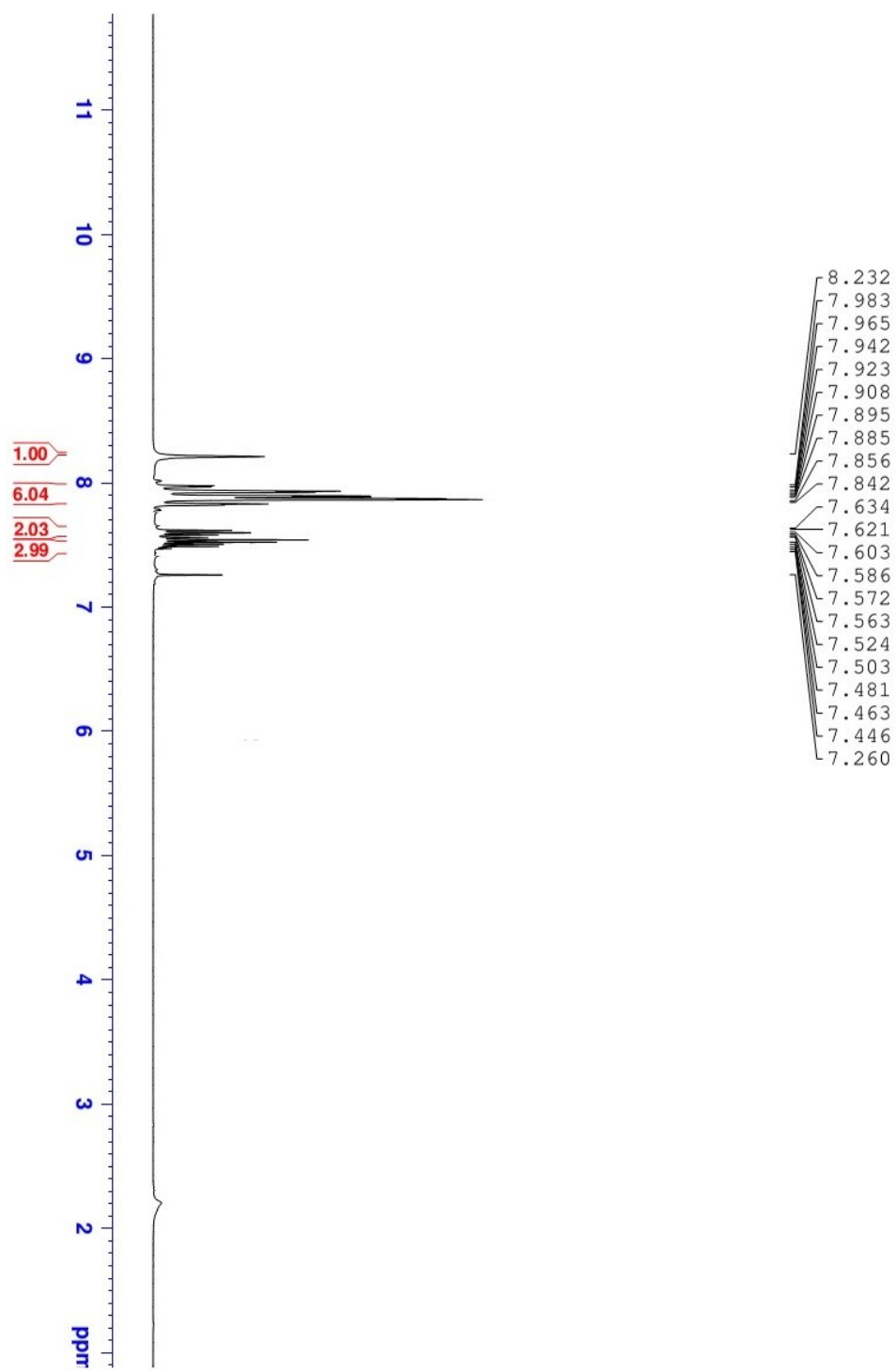
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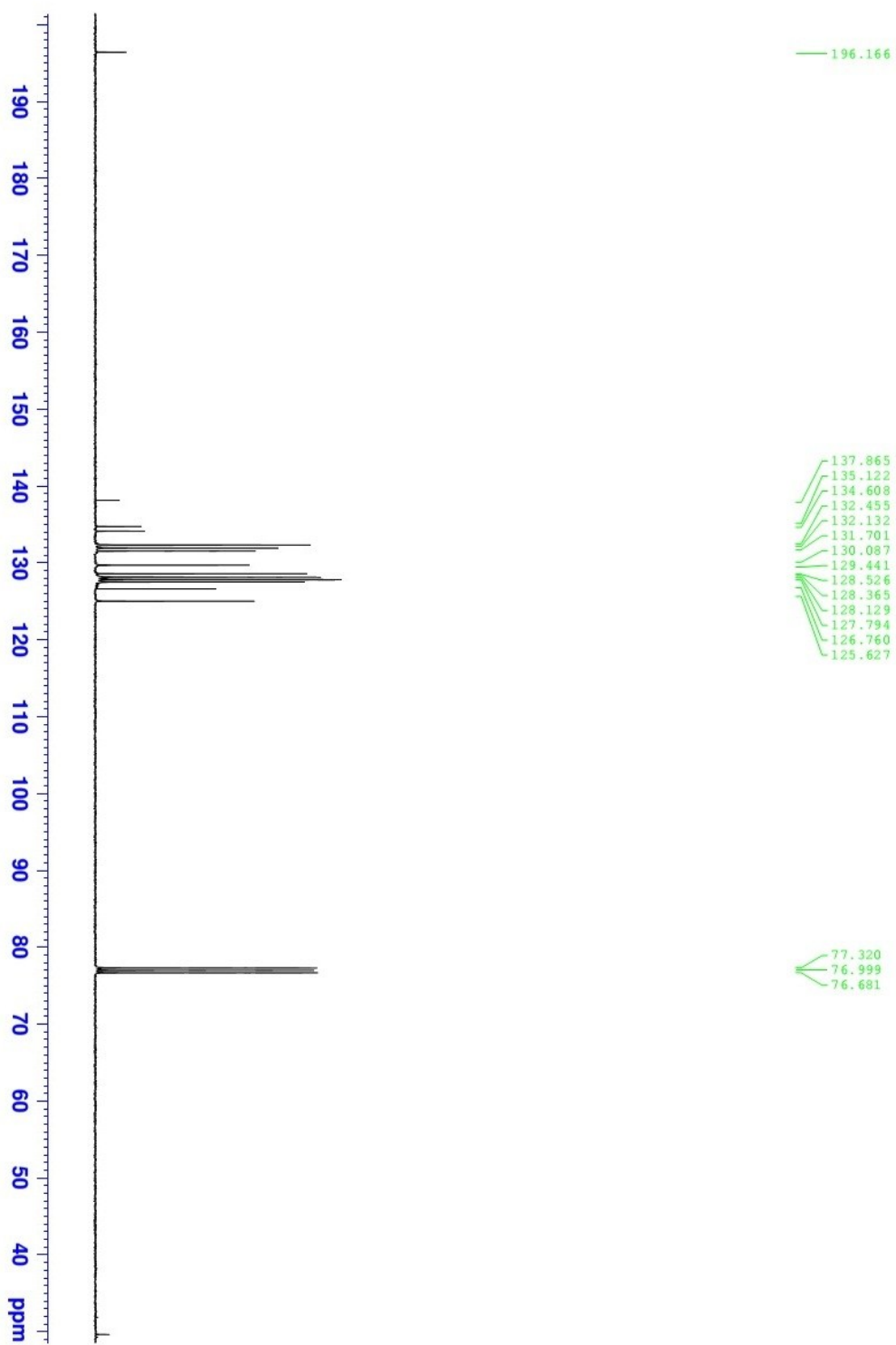
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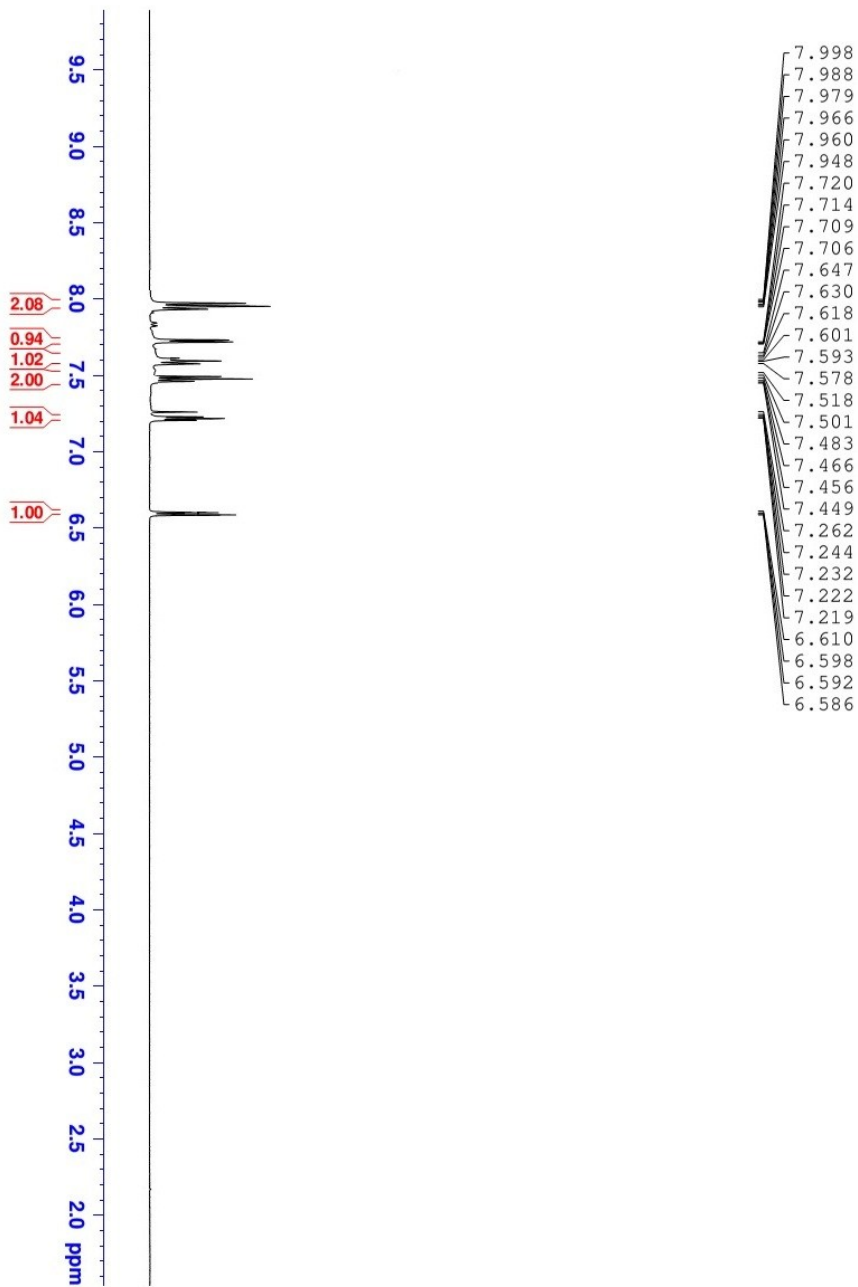
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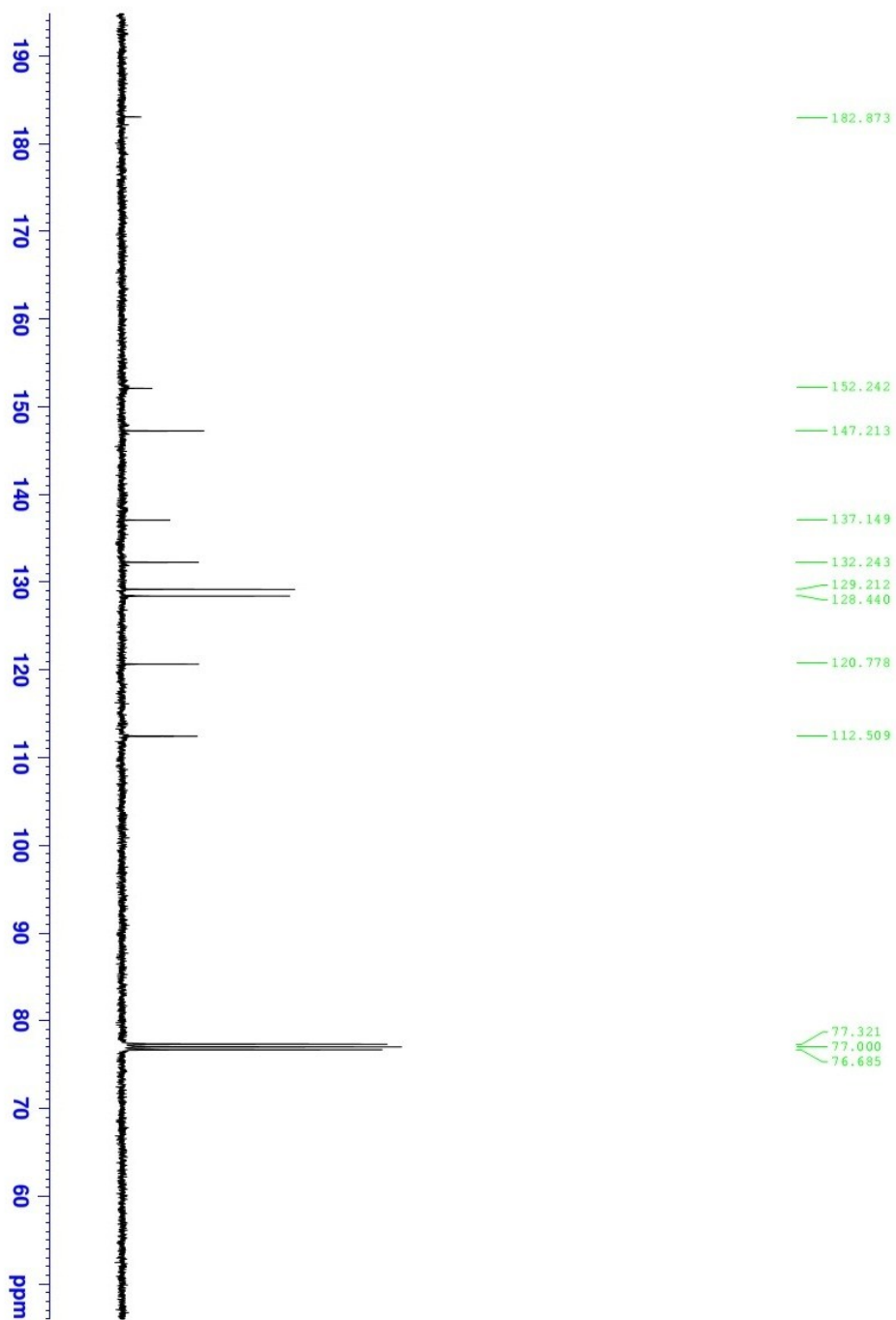
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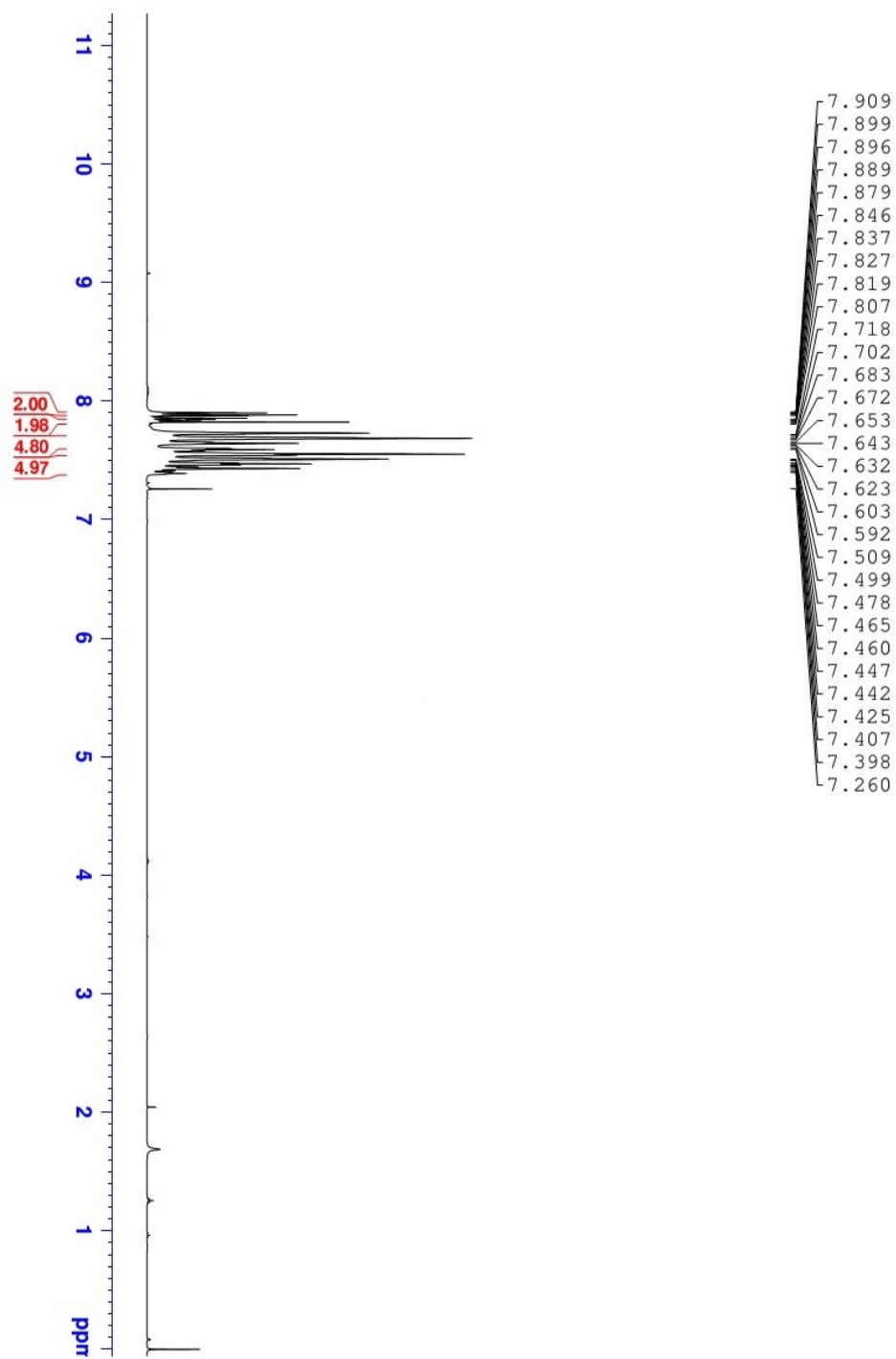
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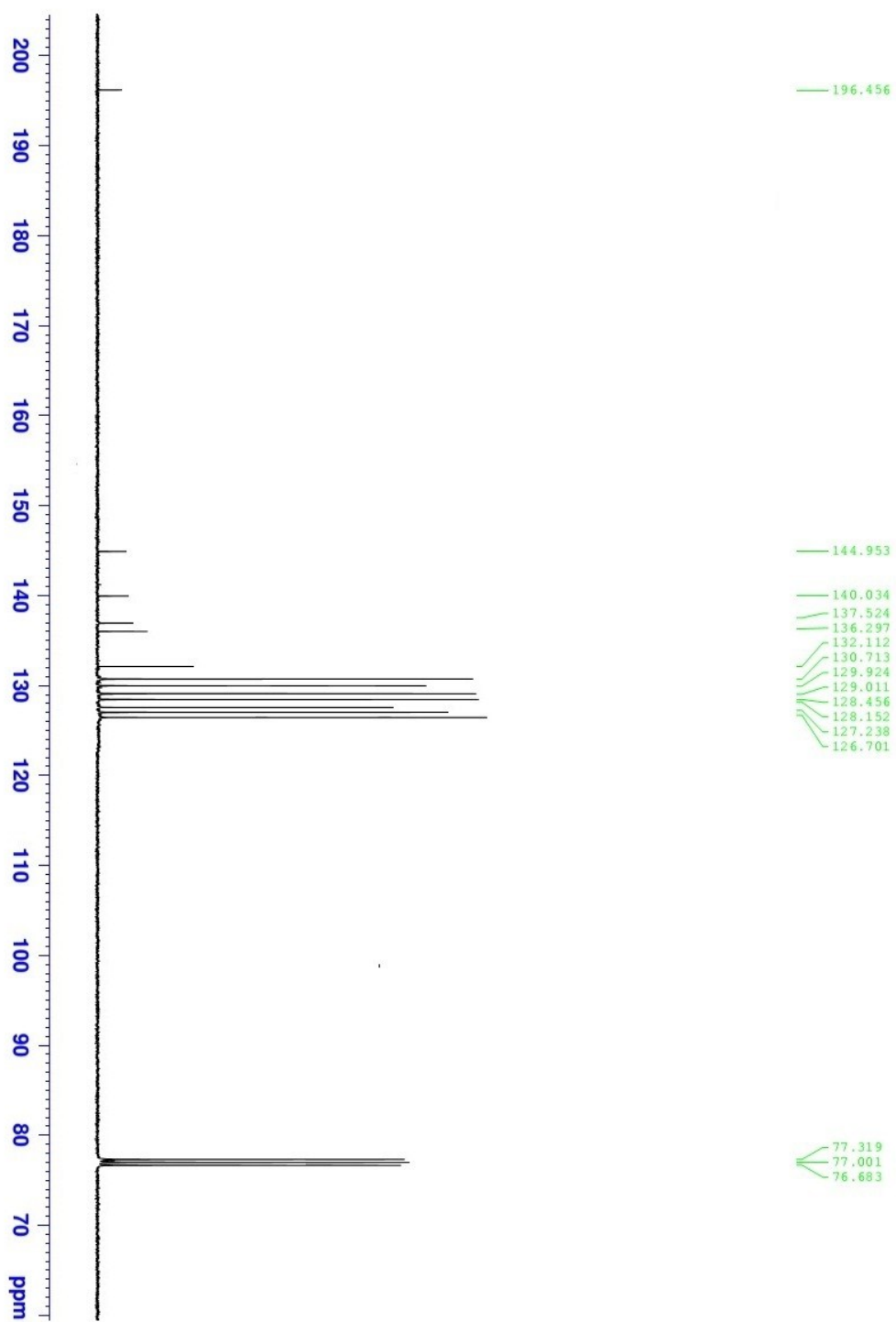
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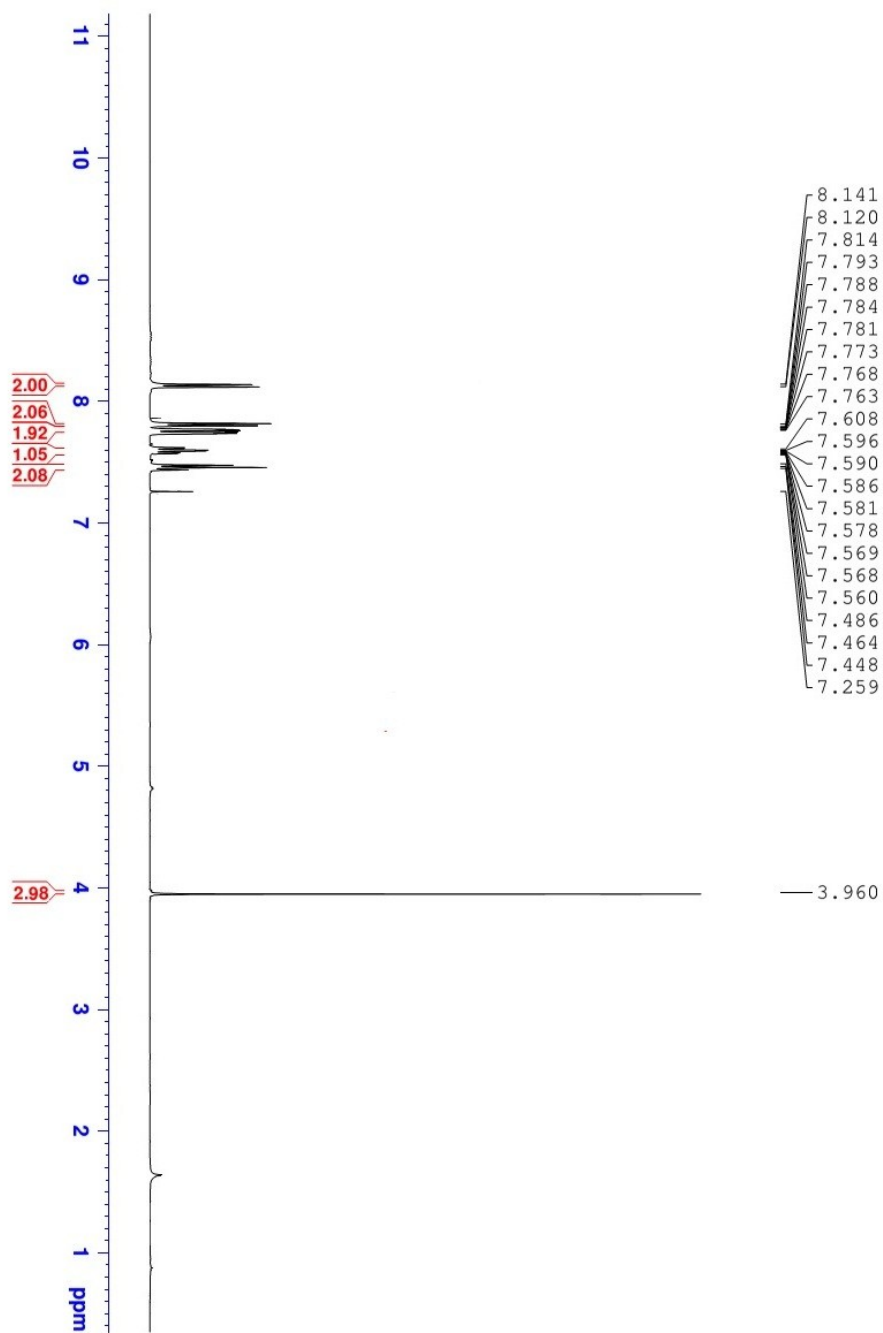
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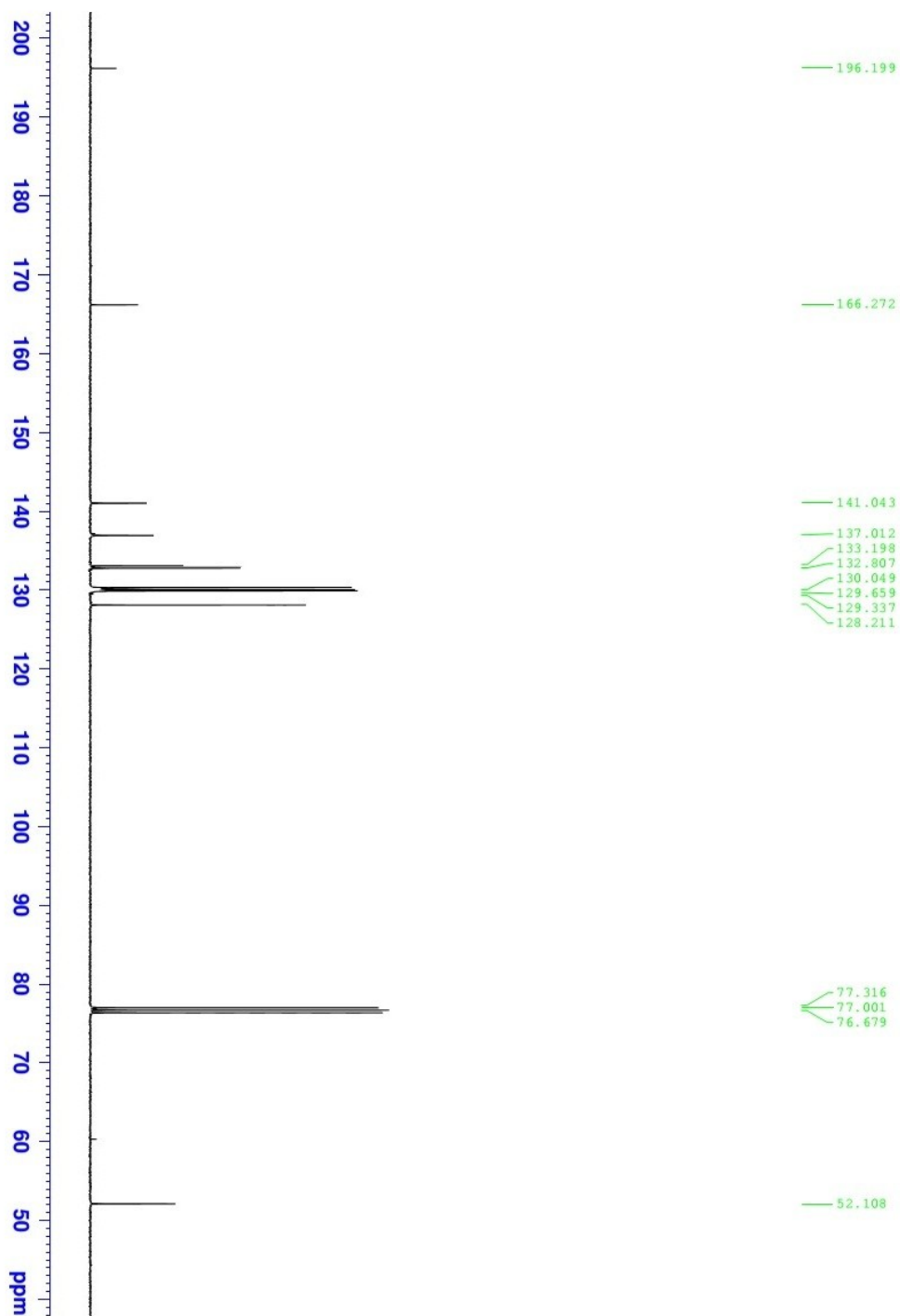
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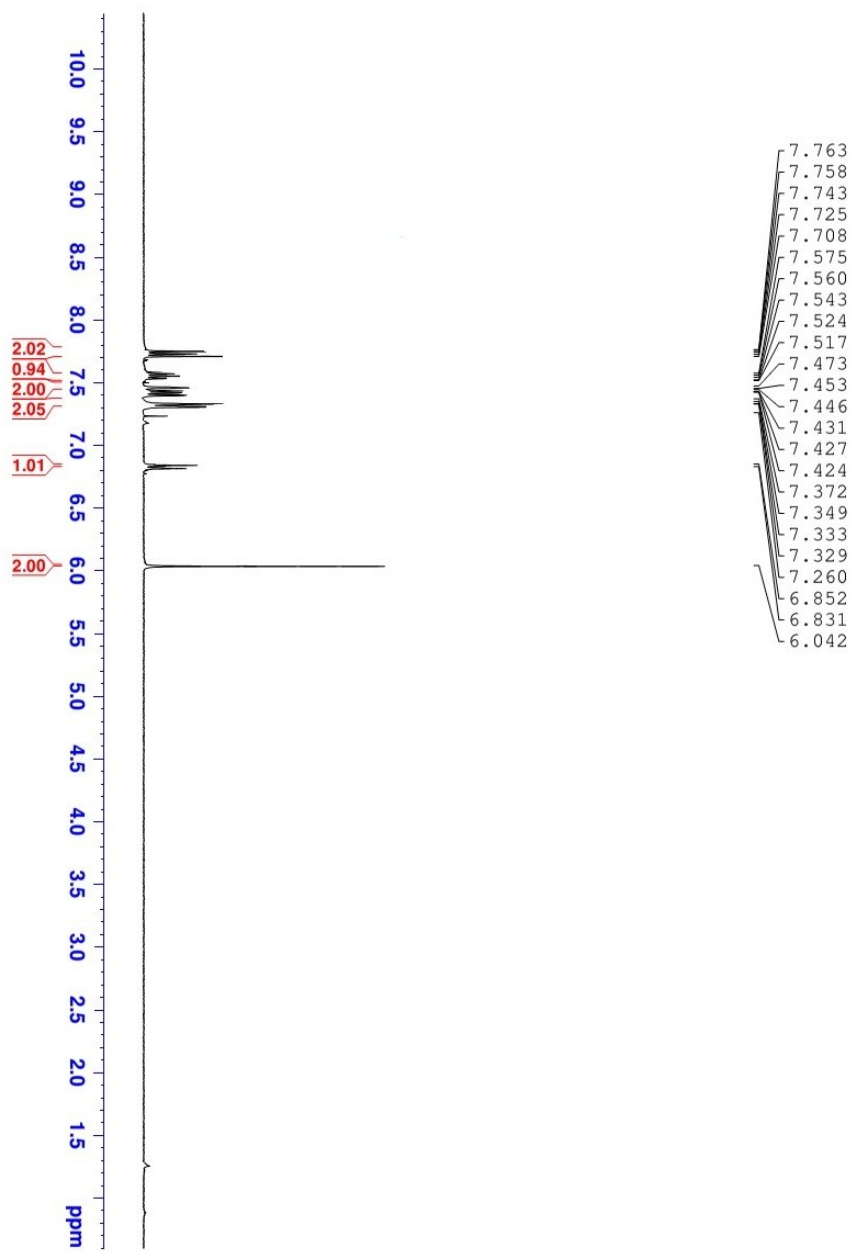
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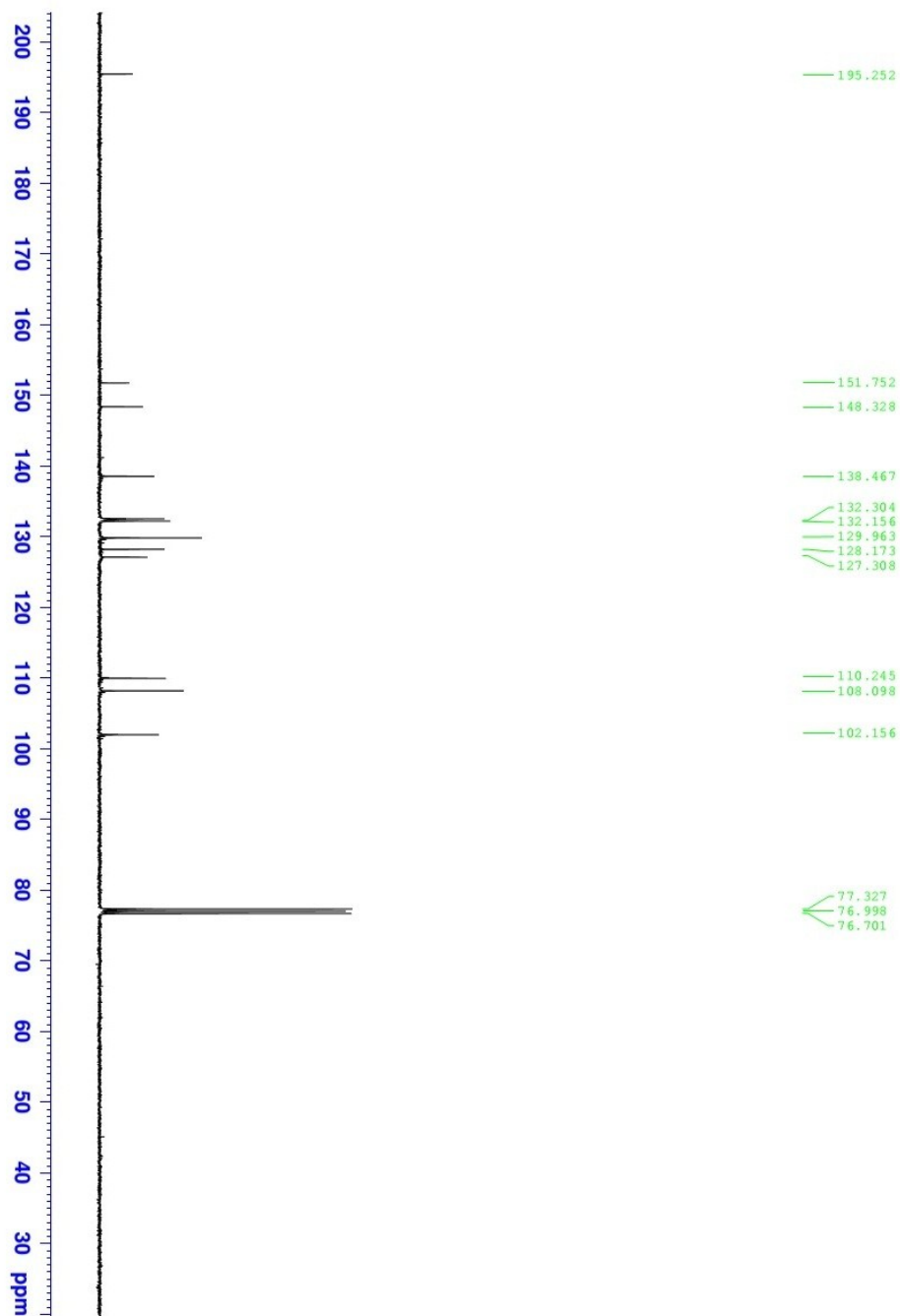
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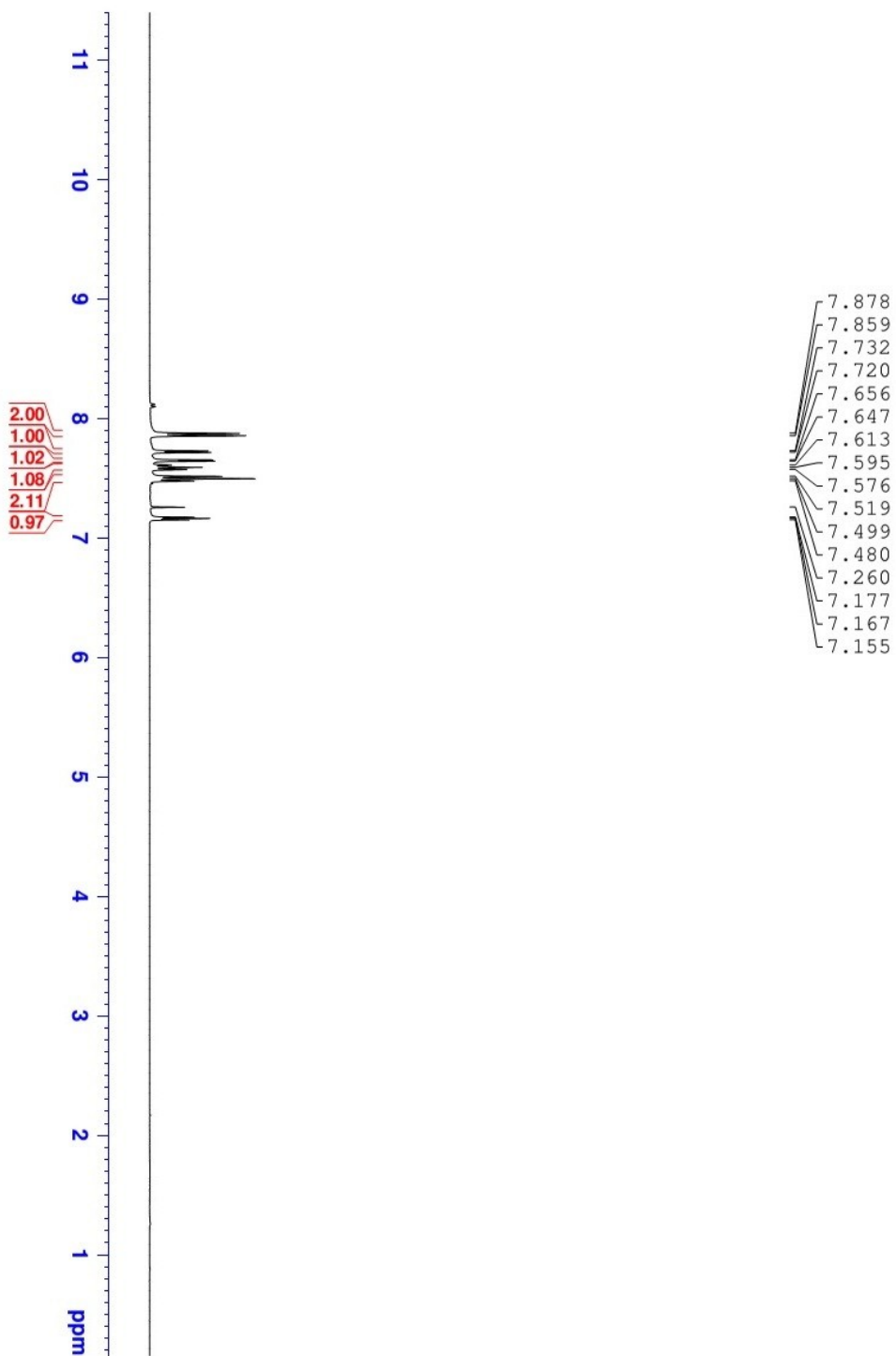
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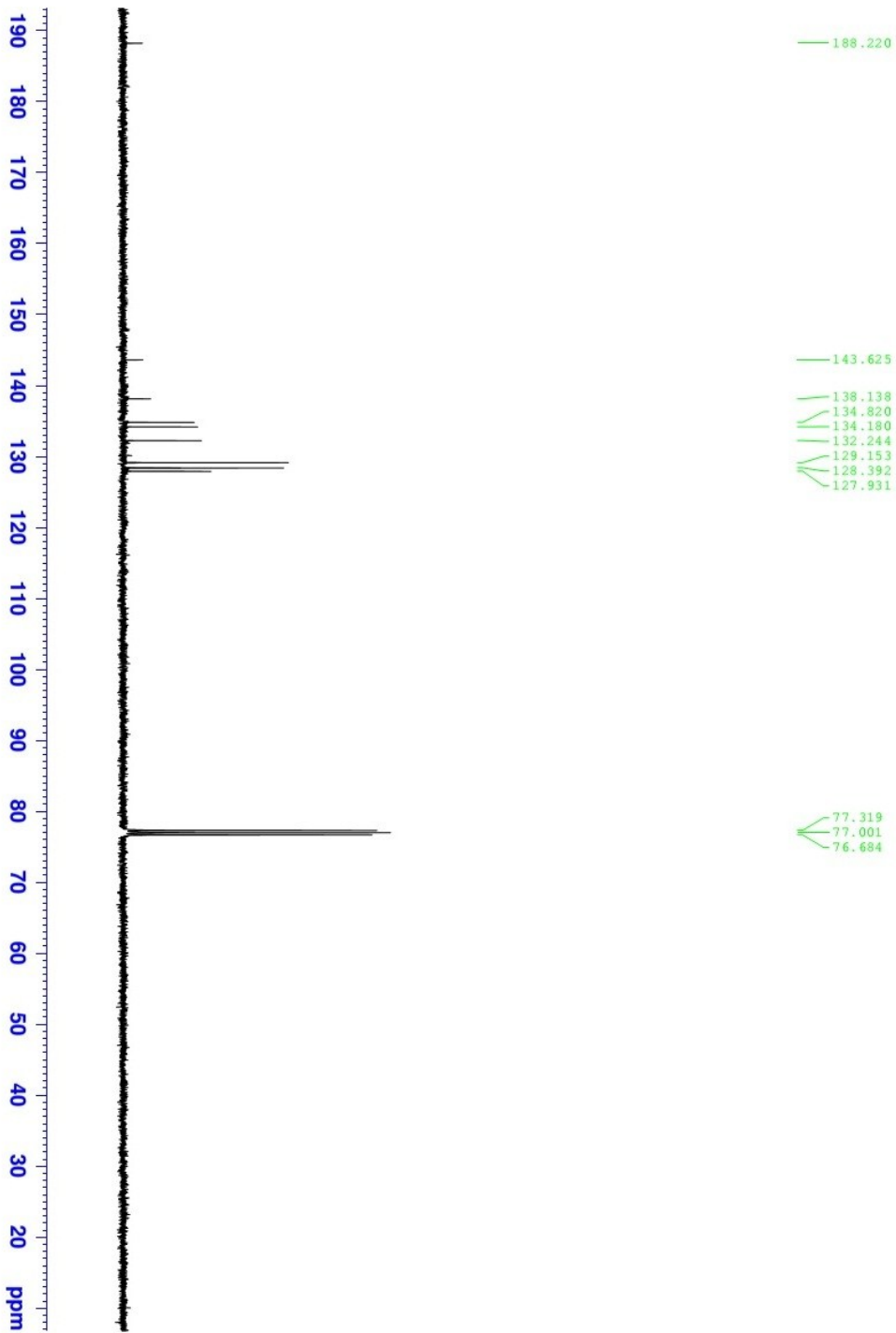
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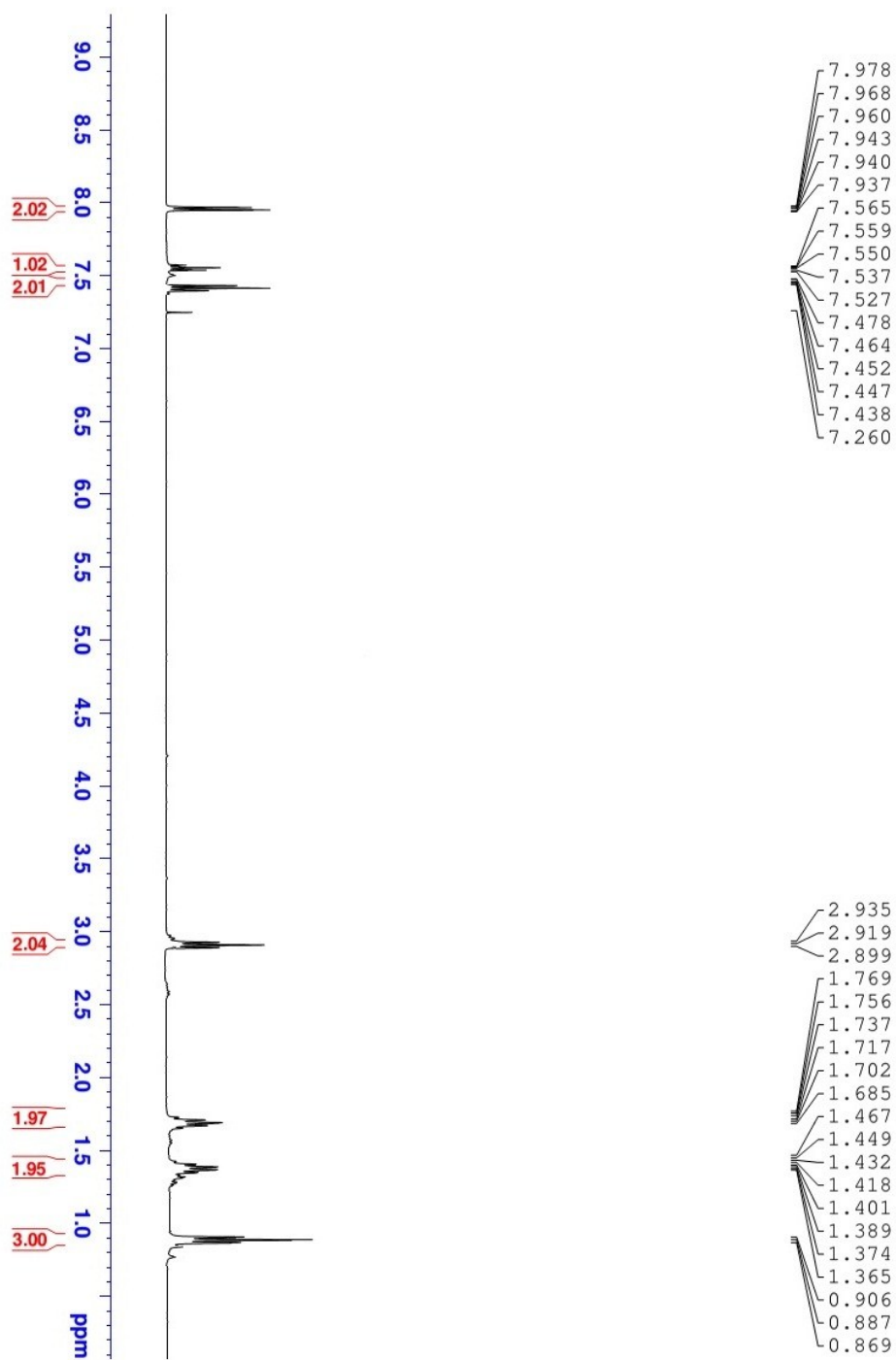
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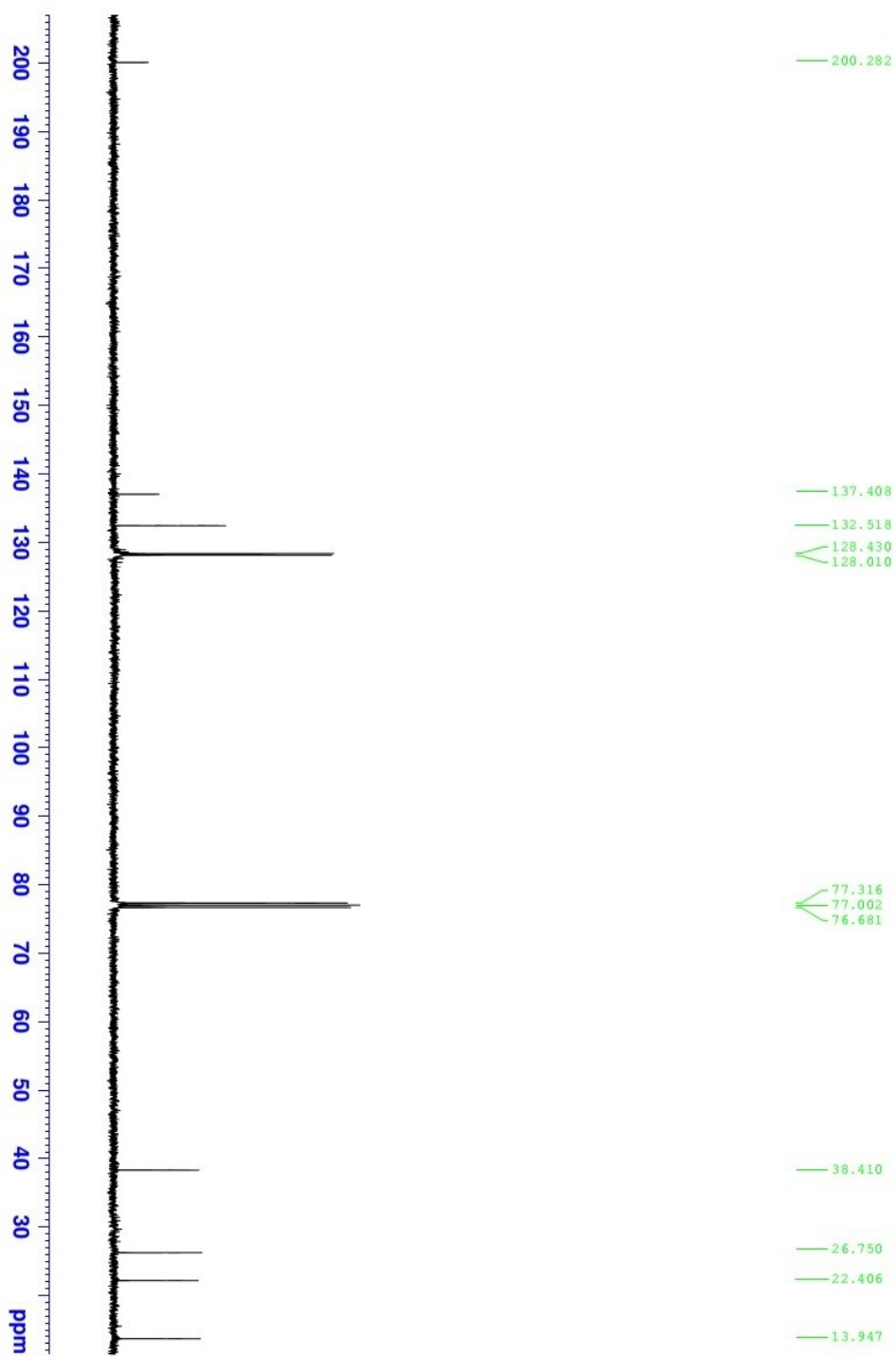
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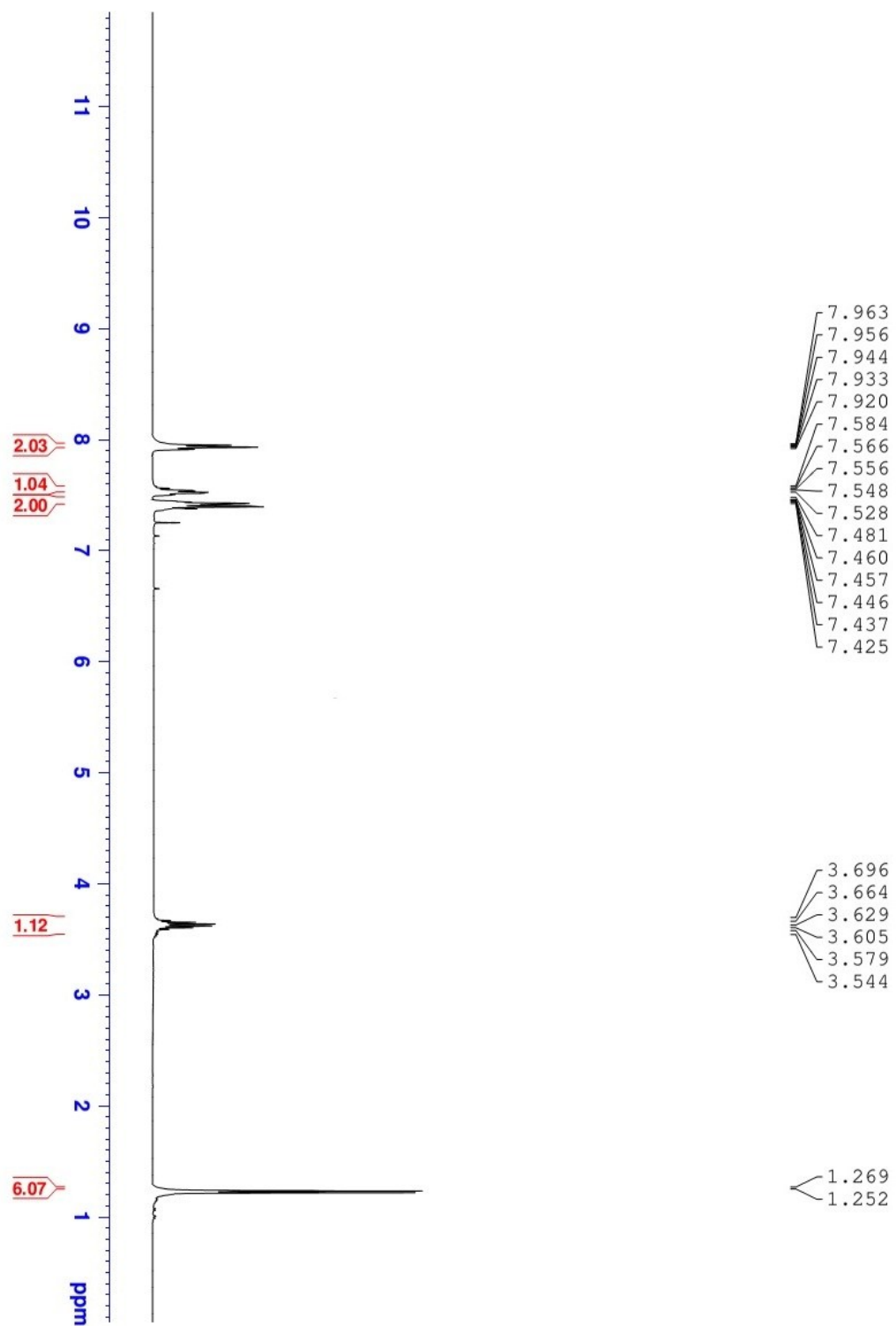
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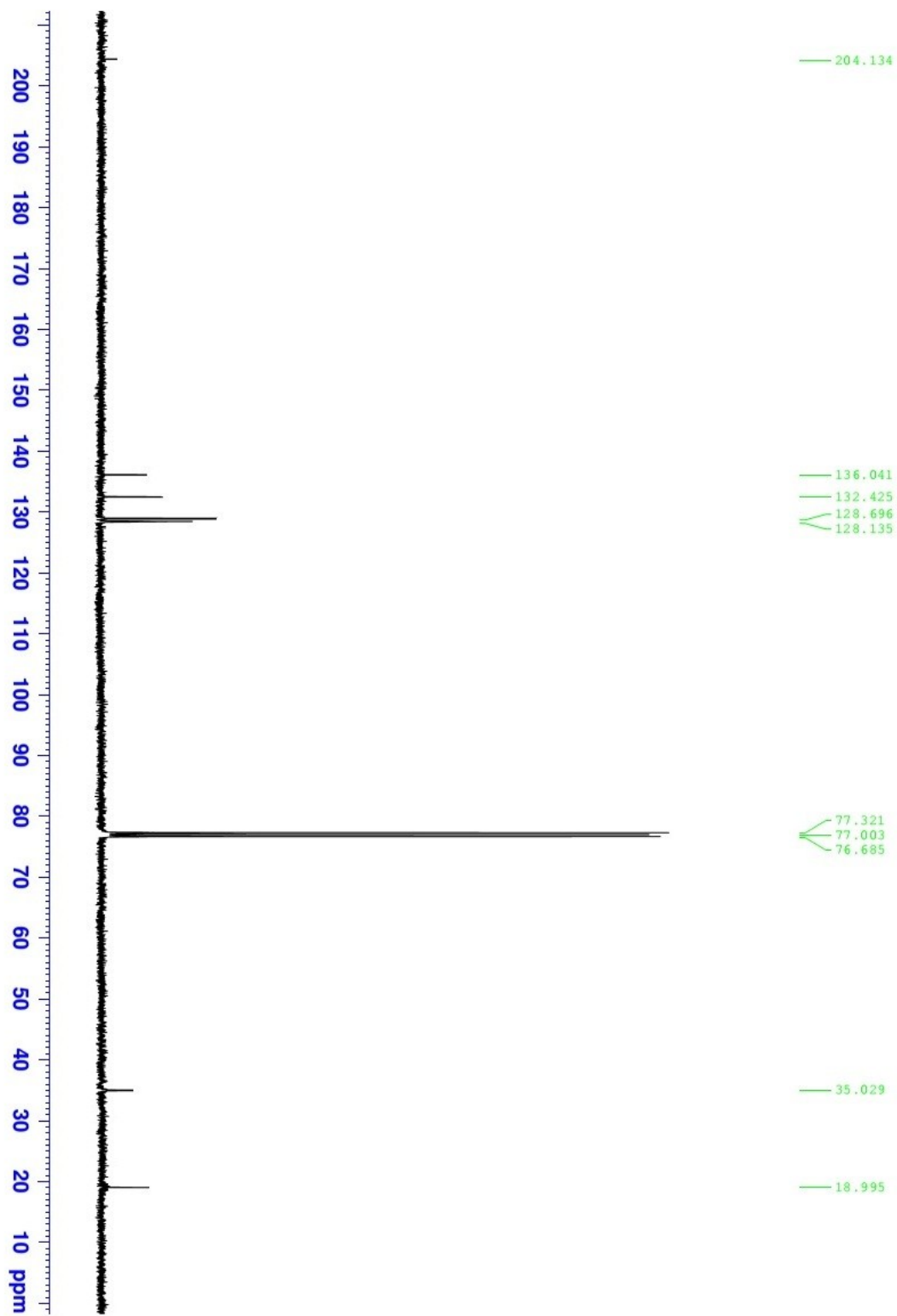
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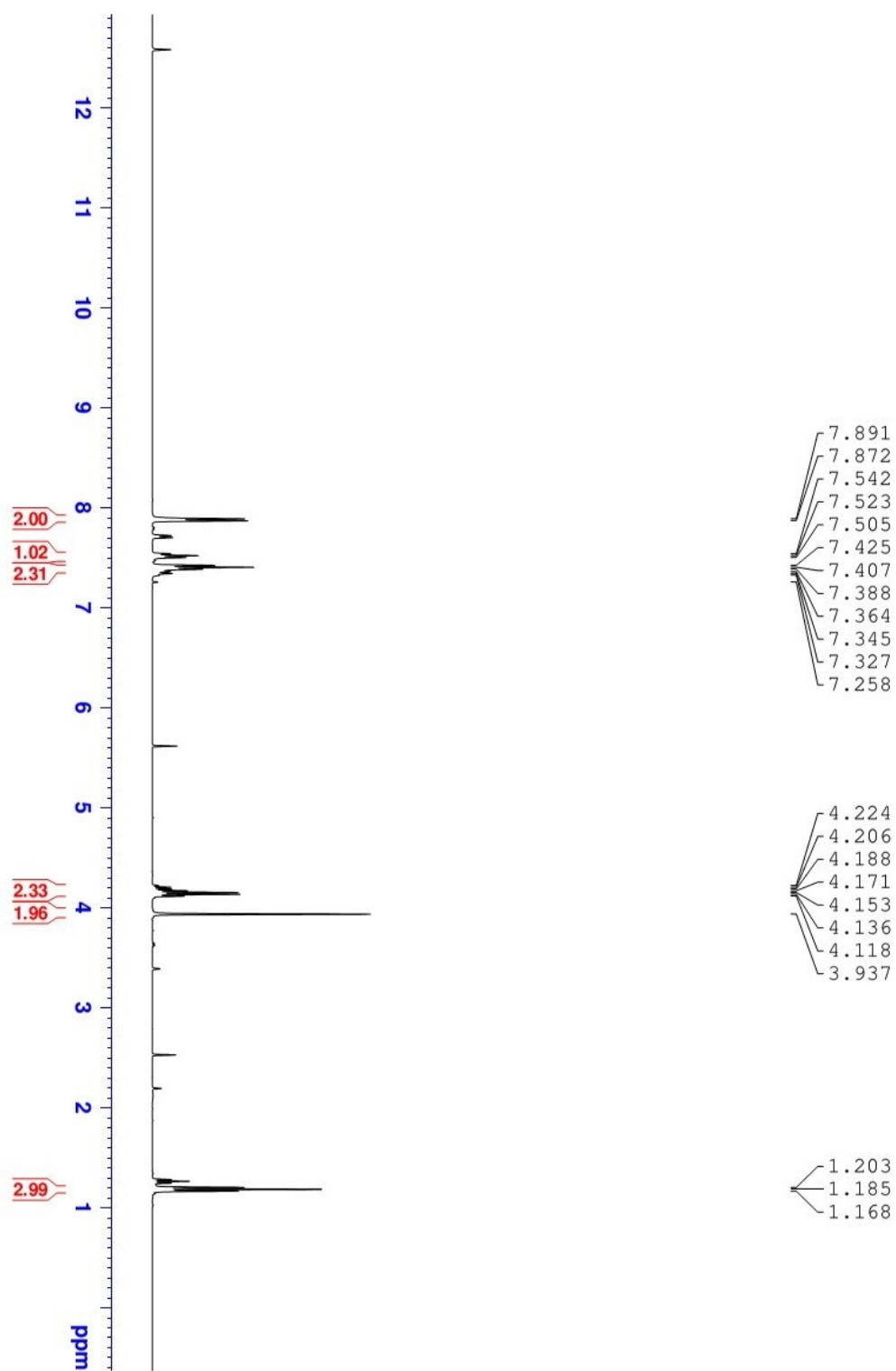
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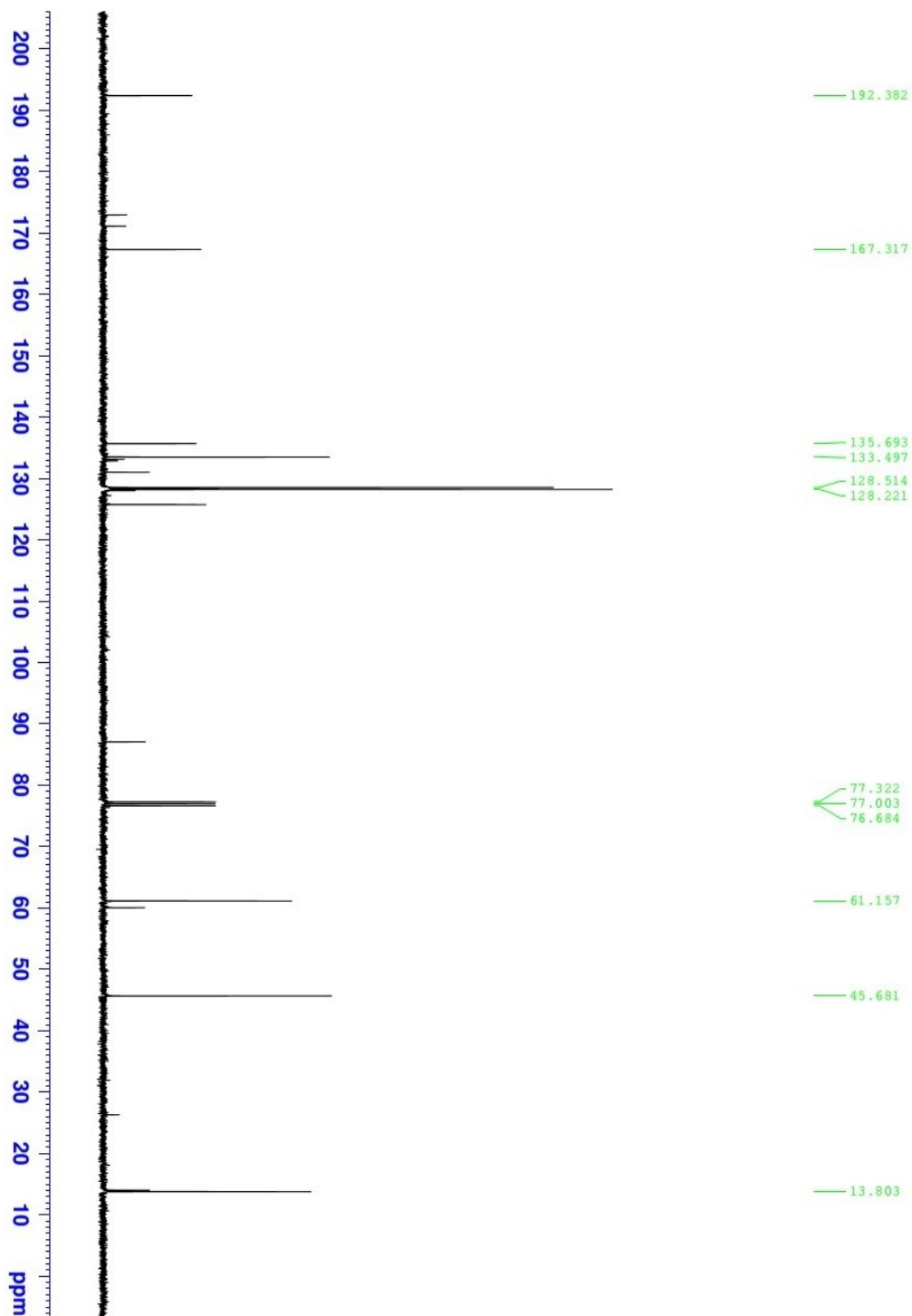
^1H NMR of Compound 3ha



¹³C NMR of Compound 3ha



¹H NMR of Compound 3ia



¹³C NMR of Compound 3ia