

Supporting Information

The stable “F–SO₂” donor provides a mild and efficient approach to nitrile and amide

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Experimental Section

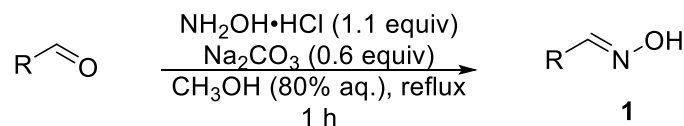
1. General information

All starting materials and reagents were purchased from commercial sources and used as received unless otherwise noted. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates. Visualization of the developed plates was performed under UV light (254nm). Flash column chromatography was performed on silica gel. GC analysis of determination of conversion was performed on the instrument of Agilent 7890 GC. NMR spectra were recorded on Bruker AVANCE III 400MHz (^1H NMR) and 101MHz (^{13}C NMR) instrument in Chloroform-*d* or DMSO-*d*₆ with TMS as internal standard.

2. General procedure for the synthesis of Substrates and fluorosulfonyl imidazolium salt A

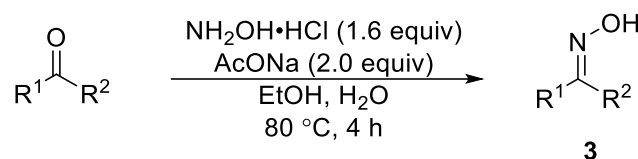
imidazolium salt A

General procedure for the synthesis of Substrates 1



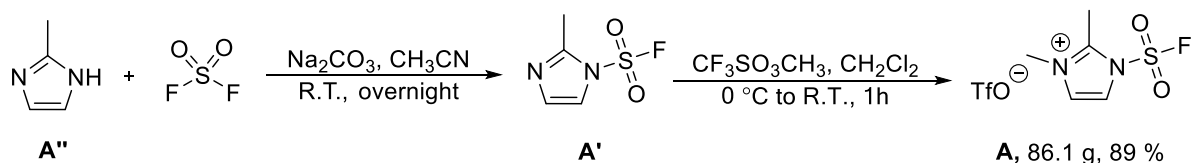
Aldehydes (20 mmol, 1.0 equiv), hydroxylamine hydrochloride (1.53 g, 22 mmol) and Na₂CO₃ (1.27 g, 12 mmol) were dissolved in 50 mL CH₃OH (80 % aq.). The reaction mixture was heated under reflux for 0.5-1.0 h. After the reaction was complete by TLC, the reaction was cooled to room temperature, diluted with water (50 mL) and extracted with ethyl acetate (3×50 mL). And then the combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated to dryness. This crude aldoximes **1** was used without further purification.

General procedure for the synthesis of Substrates 3



Ketones (20 mmol, 1.0 equiv), hydroxylamine hydrochloride (32 mmol, 1.6 equiv) and AcONa (40 mmol, 2.0 equiv) were dissolved in the mixture of EtOH/H₂O (v/v, 4:1, 50 mL). The reaction mixture was stirred at 80 °C until the consumption of the starting material was observed by TLC. After that, the reaction was cooled to room temperature, diluted with water (100 mL), extracted with ethyl acetate (150 mL ×3), dried with anhydrous Na₂SO₄, and concentrated. The residue was purified by recrystallization or flash column chromatography on silica gel to afford the desired substrates **3**.

General procedure for the synthesis of fluorosulfonyl imidazolium salt A

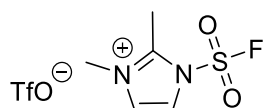


2-Methyl-1H-imidazole (24.7 g, 300 mmol), acetonitrile (600 mL) and sodium carbonate (79.6

g, 750 mmol) were added into a 1L round-bottom flask. Round-bottom flask equipped with magnetic stirrer and rubber stopper. Then the SO₂F₂ gas was introduced into the stirring reaction mixture by slow bubbling through a SO₂F₂ balloon, and the reaction mixture was stirred at room temperature for overnight. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was diluted with 600 mL of DCM and washed with water (3000 mL × 3), saturated NaCl solution (600 mL) and dried over anhydrous Na₂SO₄ and the evaporation of the solvent gave crude intermediate **A'** with some residual acetonitrile and dichloromethane. This crude mixture was used directly in the next step. Crude intermediate **A'** and dichloromethane (300mL) were added into a 500mL round-bottom flask. Round-bottom flask equipped with magnetic stirrer. Then methyl trifluoromethanesulfonate (34 mL, 296 mmol) added slowly at 0 °C under N₂. The mixture allowed to warm to room temperature and stirred for 4 hours, while monitoring by TLC. After that time, the mixture was concentrated under rotary evaporation to give a white viscous crude product, to which methyl tertbutyl ether (300 mL) was added. With vigorous stirring, a solid precipitate was formed. The precipitate was washed with methyl tert-butyl ether (300mL × 3) and dried in vacuo to yield the compound **A** as a white solid (86.1 g, overall yield 89 %).

Analytic Data of fluorosulfonyl imidazolium salt **A**

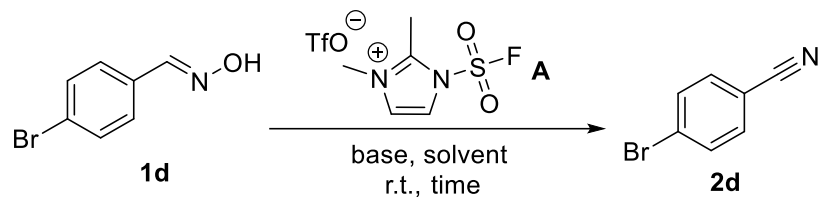
1-(Fluorosulfonyl)-2,3-dimethyl-1H-imidazol-3-ium trifluoromethanesulfonate (**A**)¹



¹H NMR (400 MHz, DMSO-*d*₆) δ 7.44 (d, *J* = 2.0 Hz, 1H), 7.41 (d, *J* = 1.9 Hz, 1H), 3.68 (s, 3H), 2.50 (s, 3H). ¹⁹F NMR (377 MHz, DMSO-*d*₆) δ 39.97 (s, 1F), -77.96 (s, 3F).

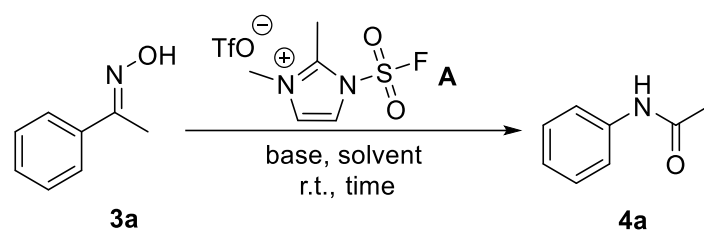
3. Optimization of reaction conditions

Table 1 Optimization of converting aldoxime to nitrile^a



Entry	A (eq.)	Base (eq.)	Solvent (mL)	Yield (%) ^b
1	1.0	Et ₃ N (2.0)	CH ₃ CN	70
2	1.5	Et₃N (2.0)	CH₃CN	98
3	2.0	Et ₃ N (2.0)	CH ₃ CN	97
4	1.5	DIPEA (2.0)	CH ₃ CN	87
5	1.5	DBU(2.0)	CH ₃ CN	81
6	1.5	Pyridine (2.0)	CH ₃ CN	24
7	1.5	Na ₂ CO ₃ (2.0)	CH ₃ CN	42
8	1.5	K ₂ CO ₃ (2.0)	CH ₃ CN	64
9	1.5	CsF (2.0)	CH ₃ CN	48
10	1.5	NaOH (2.0)	CH ₃ CN	50
11	1.5	Et ₃ N (1.0)	CH ₃ CN	21
12	1.5	Et ₃ N (1.5)	CH ₃ CN	53
13 ^c	1.5	Et ₃ N (2.0)	CH ₃ CN	98
14	1.5	Et ₃ N (2.0)	CH ₂ Cl ₂	62
15	1.5	Et ₃ N (2.0)	H ₂ O	49
16	1.5	Et ₃ N (2.0)	EtOAc	38
17	1.5	Et ₃ N (2.0)	DMF	52
18	1.5	Et ₃ N (2.0)	CH ₃ OH	37
19	1.5	Et ₃ N (2.0)	THF	35
20	1.5	Et ₃ N (2.0)	Heptane	47
21	No A	Et ₃ N (2.0)	CH ₃ CN	No reaction
22	1.5	No base	CH ₃ CN	No reaction

^a Reaction conditions: 4-bromobenzaldehyde oxime **1d** (0.5 mmol), fluorosulfuryl imidazolium salt **A**, base, CH₃CN (2.0 mL, 0.25 M), room temperature, 10 min. ^b Isolated yield. ^c 15 min.

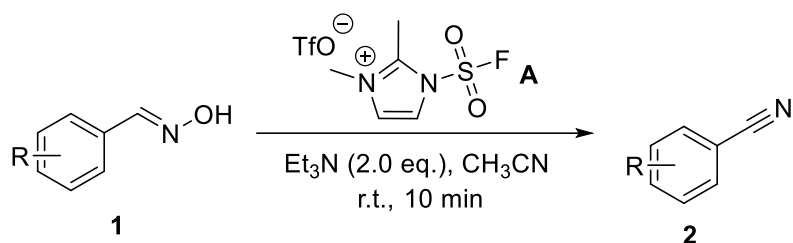
Table 2 Optimization of converting ketoxime to amide^a

Entry	A (eq.)	Base	Solvent	Yield ^b (%)
1	1.0	Et ₃ N (2.0)	CH ₃ CN	64
2	1.5	Et₃N (2.0)	CH₃CN	98
3	2.0	Et ₃ N (2.0)	CH ₃ CN	98
4	1.5	DIPEA (2.0)	CH ₃ CN	90
5	1.5	DBU(2.0)	CH ₃ CN	70
6	1.5	Pyridine (2.0)	CH ₃ CN	72
7	1.5	Na ₂ CO ₃ (2.0)	CH ₃ CN	78
8	1.5	K ₂ CO ₃ (2.0)	CH ₃ CN	88
9	1.5	CsCO ₃ (2.0)	CH ₃ CN	83
10	1.5	Et ₃ N (1.0)	CH ₃ CN	80
11	1.5	Et ₃ N (1.5)	CH ₃ CN	91
12 ^c	1.5	Et ₃ N (2.0)	CH ₃ CN	98
13	1.5	Et ₃ N (2.0)	DMF	80
14	1.5	Et ₃ N (2.0)	CH ₂ Cl ₂	81
15	1.5	Et ₃ N (2.0)	THF	83
16	1.5	Et ₃ N (2.0)	H ₂ O	35
17	1.5	Et ₃ N (2.0)	EtOAc	62
18	1.5	Et ₃ N (2.0)	CH ₃ OH	31
19	1.5	Et ₃ N (2.0)	n-hexane	46
20	No A	Et ₃ N (2.0)	CH ₃ CN	No reaction
21	1.5	No base	CH ₃ CN	44

^a Reaction conditions: acetophenone oxime **3a** (0.5 mmol), fluorosulfonyl imidazolium salt **A**, base, CH₃CN (2.0 mL, 0.25 M), room temperature, 10 min. ^b Isolated yield. ^c 15 min.

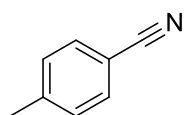
4. General procedure for the synthesis of Products 2

General procedure for the synthesis of Products 2



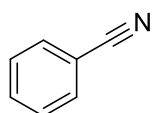
Aldoxime **1** (0.5 mmol), CH₃CN (1.0 mL) and Et₃N (139 μ L, 1.0 mmol) were added into a 15 mL Schlenk flask equipped with magnetic stirrer and rubber stopper. Then the fluorosulfonyl imidazolium salt **A** (246 mg, 0.75 mmol) was dissolved in acetonitrile (1.0 mL) and that was injected slowly with a syringe into Schlenk flask, and the reaction mixture was stirred at room temperature for 10 minutes. The progress of the reaction was monitored by TLC or GC. After completion of the reaction, the reaction mixture was diluted with 10 mL of DCM and washed with 5% HCl (2 \times 8 mL), 5% NaHCO₃ (2 \times 8 mL), saturated NaCl solution (2 \times 8 mL) and dried over anhydrous Na₂SO₄ and the evaporation of the solvent gave a residue that was purified on silica gel column chromatography using petroleum ether and ethyl acetate.

Analytic Data of Products 2



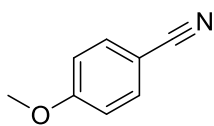
4-Methylbenzonitrile (**2a**)²

White solid (43.9 mg, 75%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.44 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 143.72, 132.05, 129.85, 119.17, 109.32, 21.84.



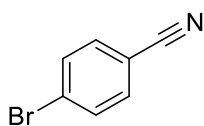
Benzonitrile (**2b**)²

Colorless oil (40.2 mg, 78%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.65 – 7.52 (m, 3H), 7.43 (t, *J* = 7.8 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 132.83, 132.09, 129.16, 118.84, 112.33.



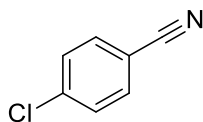
4-Methoxybenzonitrile (2c)²

White solid (63.9 mg, 96%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.38 (m, 2H), 6.88 – 6.77 (m, 2H), 3.76 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 162.83, 133.85, 119.20, 114.76, 103.65, 55.51.



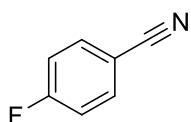
4-Bromobenzonitrile (2d)³

White solid (89.2 mg, 98%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.54 (m, 2H), 7.52 – 7.45 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 133.46, 132.62, 127.97, 118.07, 111.19.



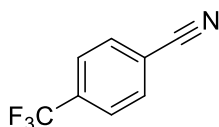
4-Chlorobenzonitrile (2e)²

White solid (68.0 mg, 99%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.6 Hz, 2H), 7.43 (d, *J* = 8.7 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 139.47, 133.41, 129.68, 117.96, 110.77.



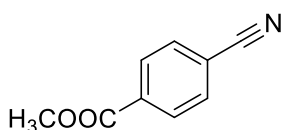
1,3-bis(4-bromophenyl)urea (2f)³

White solid (45.4 mg, 75%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.60 (ddd, *J* = 9.5, 5.0, 2.4 Hz, 2H), 7.18 – 7.01 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 164.92 (d, *J* = 255.9 Hz), 134.69 (d, *J* = 9.4 Hz), 117.98, 116.76 (d, *J* = 22.6 Hz), 108.47 (d, *J* = 3.6 Hz).



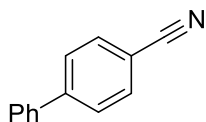
4-Fluorobenzonitrile (2g)⁴

Colorless oil (63.4 mg, 84%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.89 – 7.71 (m, 4H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 134.70 , 134.37 , 132.71 , 126.21 , 126.18 , 126.14 , 124.42 , 121.71 , 117.46 , 116.05 .



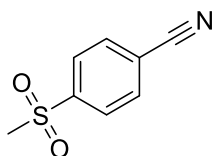
Methyl 4-cyanobenzoate (2h)²

Colorless oil (79.7 mg, 99%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 7.96 (m, 2H), 7.71 – 7.57 (m, 2H), 3.86 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 165.27 , 133.81 , 132.18 , 129.99 , 117.88 , 116.23 , 52.63 .



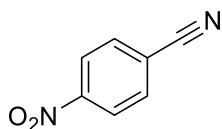
[1,1'-biphenyl]-4-Carbonitrile (2i)³

White solid (88.7 mg, 99%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.61 (q, *J* = 8.5, 7.7 Hz, 4H), 7.56 (d, *J* = 7.2 Hz, 2H), 7.51 – 7.37 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 145.41 , 138.98 , 132.61 , 129.23 , 128.81 , 127.66 , 127.24 , 119.06 , 110.85 .



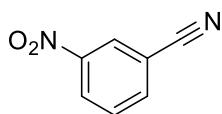
4-(methylsulfonyl)benzonitrile (2j)³

White solid (88.8 mg, 98%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.12 – 8.01 (m, 2H), 7.98 – 7.83 (m, 2H), 3.10 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 144.44 , 133.27 , 128.22 , 117.51 , 117.16 , 44.19 .



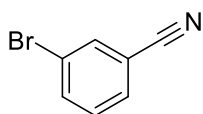
4-Nitrobenzonitrile (2k)²

Yellow solid (73.3 mg, 99%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.36 (dt, *J* = 9.0, 2.0 Hz, 2H), 7.91 (dt, *J* = 9.0, 2.0 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 150.03 , 133.58 , 124.31 , 118.28 , 116.90 .



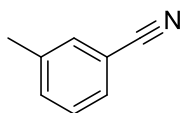
3-Nitrobenzonitrile (2l)³

White solid (72.5 mg, 98%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.59 – 8.53 (m, 1H), 8.50 (ddd, *J* = 8.4, 2.1, 1.0 Hz, 1H), 8.03 (d, *J* = 7.7 Hz, 1H), 7.77 (t, *J* = 8.0 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 148.23 , 137.68 , 130.74 , 127.57 , 127.24 , 116.58 , 114.11 .



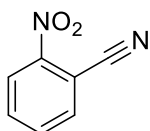
3-Bromobenzonitrile (2m)³

White solid (85.5 mg, 94%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.80 – 7.69 (m, 2H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.36 (t, *J* = 7.9 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 136.14 , 134.72 , 130.75 , 130.70 , 122.89 , 117.30 , 114.18 .



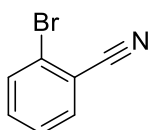
3-Methylbenzonitrile (2n)⁵

White solid (55.6 mg, 95%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.42 – 7.26 (m, 4H), 2.33 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 139.24 , 133.67 , 132.36 , 129.17 , 129.02 , 118.96 , 112.13 , 21.04 .



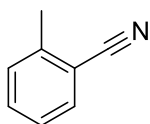
2-Nitrobenzonitrile (2o)⁶

White solid (73.3 mg, 99%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 7.9 Hz, 1H), 7.47 – 7.39 (m, 1H), 7.30 – 7.19 (m, 2H), 2.49 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 141.80, 132.67, 132.40, 130.23, 126.24, 118.07, 112.66, 20.35.



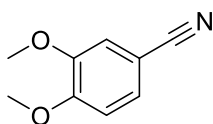
2-Bromobenzonitrile (2p)³

White solid (87.3 mg, 96%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.75 – 7.65 (m, 2H), 7.53 – 7.41 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 134.35, 133.95, 133.23, 127.69, 125.32, 117.16, 115.86.



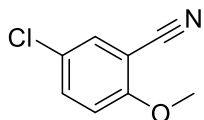
2-Methylbenzonitrile (2q)⁵

Colorless oil (56.8 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 7.9 Hz, 1H), 7.47 – 7.39 (m, 1H), 7.30 – 7.19 (m, 2H), 2.49 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 141.80, 132.67, 132.40, 130.23, 126.24, 118.07, 112.66, 20.35.



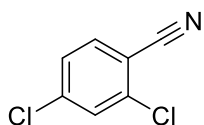
3,4-Dimethoxybenzonitrile (2r)⁵

White solid (79.1 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.25 (d, *J* = 8.3 Hz, 1H), 7.04 (s, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 3.90 (s, 3H), 3.86 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 152.86, 149.15, 126.46, 119.24, 113.88, 111.27, 103.76, 56.12.



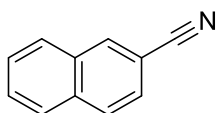
5-Chloro-2-methoxybenzonitrile (2s)⁷

White solid (84.4 mg, 96%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.50 (dd, *J* = 7.4, 2.5 Hz, 2H), 7.01 – 6.86 (m, 1H), 3.94 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 159.93 , 134.40 , 132.97 , 125.64 , 115.14 , 112.73 , 103.10 , 56.44 .



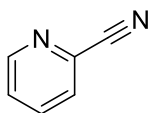
2,4-Dichlorobenzonitrile (2t)⁶

White solid (81.7 mg, 95%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.63 (d, *J* = 8.4 Hz, 1H), 7.56 (d, *J* = 1.9 Hz, 1H), 7.39 (dd, *J* = 8.4, 2.0 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 140.11 , 137.82 , 134.62 , 130.28 , 127.89 , 115.24 , 111.90 .



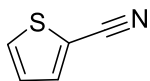
2-Naphthonitrile (2u)²

White solid (75.8 mg, 99%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.15 (s, 1H), 7.85 (t, *J* = 9.1 Hz, 3H), 7.67 – 7.51 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 134.60 , 134.11 , 132.18 , 129.20 , 129.09 , 128.40 , 128.06 , 127.69 , 126.27 , 119.31 , 109.27 .



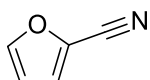
Picolinonitrile (2v)³

Yellow oil (50.5 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.64 (d, *J* = 4.8 Hz, 1H), 7.83 (td, *J* = 7.8, 1.7 Hz, 1H), 7.69 – 7.63 (m, 1H), 7.51 (ddd, *J* = 7.8, 4.8, 1.1 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 151.10 , 137.26 , 133.70 , 128.62 , 127.16 , 117.27 .



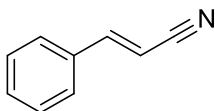
Thiophene-2-carbonitrile (2w)³

Colorless oil (45.3 mg, 83%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.74 – 7.51 (m, 2H), 7.13 (dd, *J* = 4.9, 3.9 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 137.51 , 132.76 , 127.75 , 114.30 , 109.78 .



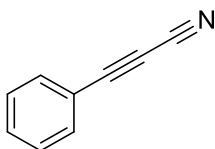
Furan-2-carbonitrile (2x)⁶

Colorless oil (36.8 mg, 79%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.56 (d, *J* = 1.8 Hz, 1H), 7.08 (d, *J* = 3.7 Hz, 1H), 6.51 (dd, *J* = 3.7, 1.9 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 147.58 , 126.04 , 122.18 , 111.57 , 111.47 .



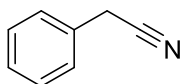
Cinnamitrile (2aa)²

Colorless oil (60.7 mg, 94%) ¹H NMR (400 MHz, Chloroform-*d*) δ 7.42 (q, *J* = 5.3, 4.8 Hz, 5H), 7.34 (d, *J* = 16.6 Hz, 1H), 5.85 (d, *J* = 16.7 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 150.54 , 133.55 , 131.27 , 129.16 , 127.47 , 118.33 , 96.36 .



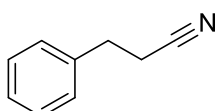
3-Phenylpropionitrile (2ab)³

White solid (54.6 mg, 86%) ¹H NMR (400 MHz, Chloroform-*d*) δ 7.64 – 7.57 (m, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 133.49 , 131.94 , 128.90 , 117.45 , 105.48 , 83.06 , 63.03 .



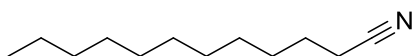
2-Phenylacetonitrile (2ac)²

Colorless oil (51.5 mg, 88%) ¹H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.30 (m, 5H), 3.73 (s, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 130.12 , 129.17 , 128.06 , 128.00 , 118.10 , 23.55 .



3-Phenylpropanenitrile (2ad)⁶

Colorless oil (59.0 mg, 90%) ¹H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.24 (m, 5H), 2.95 (t, *J* = 7.4 Hz, 2H), 2.60 (t, *J* = 7.4 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 138.28 , 128.91 , 128.40 , 127.25 , 119.38 , 31.51 , 19.28.

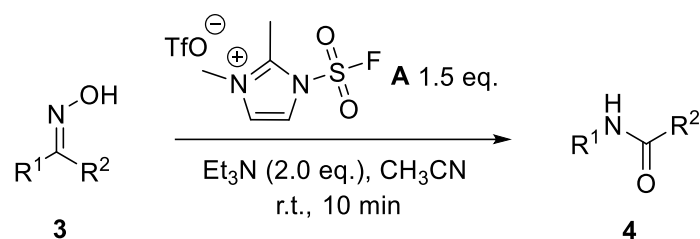


Dodecanenitrile (2ae)⁸

White solid (82.5 mg, 91%) ¹H NMR (400 MHz, Chloroform-*d*) δ 2.30 (t, *J* = 7.1 Hz, 2H), 1.62 (p, *J* = 7.2 Hz, 2H), 1.49 – 1.35 (m, 2H), 1.35 – 1.11 (m, 14H), 0.85 (t, *J* = 6.8 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 119.73 , 31.86 , 29.52 , 29.48 , 29.28 , 28.74 , 28.62 , 25.36 , 22.63 , 17.02 , 14.03 .

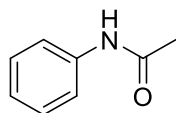
5. General procedure for the synthesis of Products 4

General procedure for the synthesis of Products 4



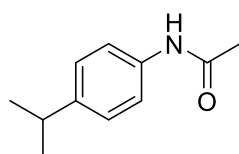
Aldoxime **1** (0.5 mmol), CH₃CN (1.0 mL) and Et₃N (139 μL, 1.0 mmol) were added into a 15 mL Schlenk flask equipped with magnetic stirrer and rubber stopper. Then the fluorosulfonyl imidazolium salt **A** (246 mg, 0.75 mmol) was dissolved in acetonitrile (1.0 mL) and that was injected slowly with a syringe into Schlenk flask, and the reaction mixture was stirred at room temperature for 10 minutes. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was diluted with 10 mL of DCM and washed with 5% HCl (2 × 8 mL), 5% NaHCO₃ (2 × 8 mL), saturated NaCl solution (2 × 8 mL) and dried over anhydrous Na₂SO₄ and the evaporation of the solvent gave a residue that was purified on silica gel column chromatography using petroleum ether and ethyl acetate.

Analytic Data of Products 4



N-phenylacetamide (**4a**)⁹

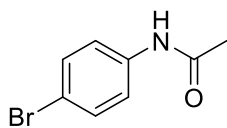
White solid (66.2 mg, 98%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.49 (s, 1H), 7.53 (d, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.11 (t, *J* = 7.4 Hz, 1H), 2.17 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 169.63, 137.88, 128.91, 124.54, 120.45, 24.15.



N-(4-isopropylphenyl)acetamide (**4b**)¹⁰

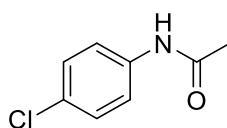
White solid (85.9, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.81 (s, 1H), 7.43 (d, *J* = 8.5 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 2.89 (p, *J* = 6.9 Hz, 1H), 2.16 (s, 3H), 1.24 (d, *J* = 6.9 Hz, 6H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 168.83, 145.02, 135.67, 126.82, 120.36, 33.60,

24.38 , 24.04 .



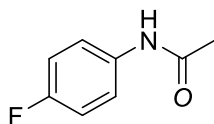
***N*-(4-bromophenyl)acetamide (4c)¹¹**

White solid (103.8 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.48 (s, 1H), 7.43 (s, 4H), 2.19 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 168.48 , 136.97 , 131.96 , 121.42 , 116.88 , 24.61 .



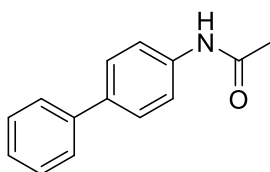
***N*-(4-chlorophenyl)acetamide (4d)¹²**

White solid (81.4 mg, 96%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.08 (s, 1H), 7.64 – 7.56 (m, 2H), 7.38 – 7.29 (m, 2H), 2.05 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.91, 138.74, 129.03, 126.94, 120.91, 24.45.



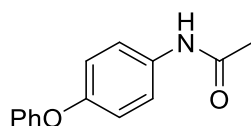
***N*-(4-fluorophenyl)acetamide (4e)¹³**

White solid (91.8 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.71 (s, 1H), 7.47 (dd, *J* = 8.7, 4.8 Hz, 2H), 7.01 (t, *J* = 8.5 Hz, 2H), 2.17 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 168.61, 159.39 (d, *J* = 243.5 Hz), 135.10 – 132.25 (m), 121.91 (d, *J* = 7.9 Hz), 115.61 (d, *J* = 22.4 Hz), 24.43.



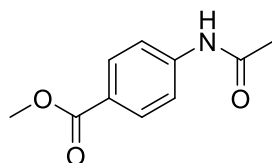
***N*-([1,1'-biphenyl]-4-yl)acetamide (4f)¹⁴**

White solid (97.2 mg, 92%). ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.05 (s, 1H), 7.68 (d, $J = 8.6$ Hz, 2H), 7.61 (t, $J = 8.5$ Hz, 4H), 7.43 (t, $J = 7.6$ Hz, 2H), 7.31 (t, $J = 7.3$ Hz, 1H), 2.07 (s, 3H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 168.82 , 140.20 , 139.28 , 135.10 , 129.35 , 127.42 , 127.34 , 126.67 , 119.77 , 24.51 .



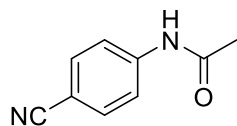
***N*-(4-phenoxyphenyl)acetamide (4g)¹⁰**

White solid (106.8 mg, 94%). ^1H NMR (400 MHz, $\text{Chloroform-}d$) δ 7.78 (d, $J = 9.8$ Hz, 1H), 7.48 (d, $J = 8.9$ Hz, 2H), 7.33 (t, $J = 7.9$ Hz, 2H), 7.10 (t, $J = 7.4$ Hz, 1H), 6.99 (dd, $J = 8.2, 4.8$ Hz, 4H), 2.18 (s, 3H). ^{13}C NMR (101 MHz, $\text{Chloroform-}d$) δ 168.70 , 157.49 , 153.51 , 133.43 , 129.76 , 123.13 , 121.88 , 119.55 , 118.46 , 24.38 .



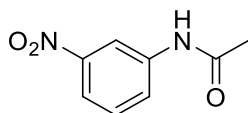
Methyl 4-acetamidobenzoate (4h)¹⁵

White solid (91.7 mg, 95%). ^1H NMR (400 MHz, $\text{Chloroform-}d$) δ 8.39 (s, 1H), 7.98 (d, $J = 8.1$ Hz, 2H), 7.64 (d, $J = 8.4$ Hz, 2H), 3.90 (s, 3H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, $\text{Chloroform-}d$) δ 169.21 , 166.84 , 142.44 , 130.77 , 125.38 , 118.95 , 52.11 , 24.67 .



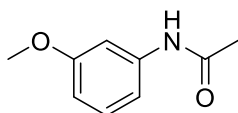
***N*-(4-cyanophenyl)acetamide (4i)¹⁶**

White solid (64.8 mg, 81%). ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.39 (s, 1H), 7.76 (s, 4H), 2.09 (s, 3H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 169.66 , 143.94 , 133.71 , 119.57 , 119.37 , 105.14 , 24.67 .



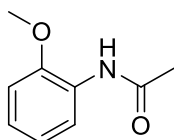
***N*-(3-nitrophenyl)acetamide (4j)**¹³

Yellow solid (85.6 mg, 95%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.44 (s, 1H), 8.62 (t, *J* = 2.0 Hz, 1H), 7.88 (dt, *J* = 8.1, 2.5 Hz, 2H), 7.59 (t, *J* = 8.2 Hz, 1H), 2.10 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 169.55 , 148.40 , 140.85 , 130.59 , 125.32 , 117.99 , 113.42 , 24.52 .



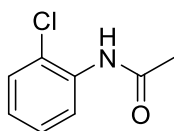
***N*-(3-methoxyphenyl)acetamide (4k)**¹³

White solid (80.1 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.12 (s, 1H), 7.19 (t, *J* = 8.1 Hz, 1H), 7.02 (d, *J* = 7.9 Hz, 1H), 6.71 – 6.59 (m, 1H), 3.77 (s, 3H), 2.16 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 169.00 , 160.06 , 139.26 , 129.64 , 112.25 , 109.98 , 105.89 , 55.28 , 24.56 .



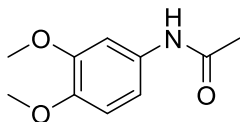
***N*-(2-methoxyphenyl)acetamide (4l)**¹⁵

White solid (79.3 mg, 96%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 9.4 Hz, 1H), 7.82 (s, 1H), 7.04 (td, *J* = 7.8, 1.6 Hz, 1H), 6.96 (td, *J* = 7.7, 1.3 Hz, 1H), 6.87 (dd, *J* = 8.1, 1.1 Hz, 1H), 3.87 (s, 3H), 2.20 (s, 3H). ¹³C NMR (101 MHz,) δ 168.27 , 147.71 , 127.69 , 123.64 , 121.04 , 119.80 , 109.89 , 55.64 , 24.93 .



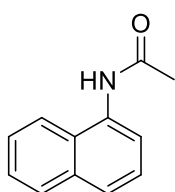
***N*-(2-chlorophenyl)acetamide (4m)**¹³

White solid (81.5 mg, 95%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.37 (d, *J* = 8.1 Hz, 1H), 7.67 (s, 1H), 7.43 – 7.34 (m, 1H), 7.32 – 7.24 (m, 1H), 7.05 (t, *J* = 7.3 Hz, 1H), 2.26 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 168.32 , 134.60 , 128.99 , 127.75 , 124.65 , 122.57 , 121.68 , 24.90 .



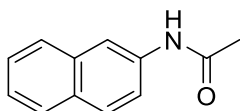
***N*-(3,4-dimethoxyphenyl)acetamide (4n)¹³**

White solid (94.6 mg, 97%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.82 (s, 1H), 7.29 (d, *J* = 2.3 Hz, 1H), 7.09 (dd, *J* = 8.7, 2.4 Hz, 1H), 6.86 (d, *J* = 8.7 Hz, 1H), 3.71 (s, 3H), 3.70 (s, 3H), 2.01 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.37, 148.92, 145.09, 133.44, 112.43, 111.40, 104.73, 56.11, 55.75, 24.30.



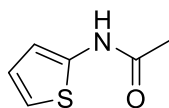
***N*-(naphthalen-1-yl)acetamide (4o)¹⁰**

White solid (86.1 mg, 93%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.96 (s, 1H), 8.13 – 8.05 (m, 1H), 7.94 (dd, *J* = 6.2, 3.2 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 1H), 7.71 (d, *J* = 7.3 Hz, 1H), 7.55 (td, *J* = 6.9, 6.0, 3.6 Hz, 2H), 7.52 – 7.45 (m, 1H), 2.20 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 169.42, 134.17, 128.58, 128.14, 126.44, 126.21, 126.04, 125.51, 123.22, 122.01, 23.97.



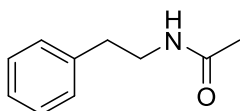
***N*-(naphthalen-2-yl)acetamide (4p)¹²**

White solid (88.0 mg, 95%). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.21 (s, 1H), 7.95 (s, 1H), 7.77 (dt, *J* = 8.5, 4.3 Hz, 3H), 7.51 – 7.38 (m, 3H), 2.23 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 168.99, 135.44, 133.81, 130.65, 128.73, 127.67, 127.56, 126.49, 125.04, 120.05, 116.84, 24.66.



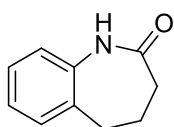
***N*-(thiophen-2-yl)acetamide (4q)¹⁷**

White solid (56.5 mg, 80%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.14 (s, 1H), 6.94 – 6.86 (m, 1H), 6.83 (dd, *J* = 5.5, 3.7 Hz, 1H), 6.63 (dd, *J* = 3.7, 1.4 Hz, 1H), 2.06 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 166.67, 140.36, 124.32, 117.03, 110.69, 22.98.



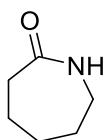
N-phenethylacetamide (4r)¹⁵

White solid (71.8 mg, 88 %). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.33 (t, $J = 7.3$ Hz, 2H), 7.24 (dd, $J = 19.8, 7.2$ Hz, 3H), 5.66 (s, 1H), 3.53 (q, $J = 6.4$ Hz, 2H), 2.84 (t, $J = 6.9$ Hz, 2H), 1.96 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 170.22 , 138.88 , 128.77 , 128.68 , 126.55 , 40.72 , 35.63 , 23.37 .



1,3,4,5-tetrahydro-2H-1-benzazepin-2-one (4s)¹⁵

White solid (62.8 mg, 78%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.85 (s, 1H), 7.25 (d, $J = 8.3$ Hz, 2H), 7.20 – 7.12 (m, 1H), 7.01 (d, $J = 7.7$ Hz, 1H), 2.83 (t, $J = 7.2$ Hz, 2H), 2.39 (t, $J = 7.2$ Hz, 2H), 2.27 (q, $J = 7.0$ Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 175.24 , 137.76 , 134.39 , 129.91 , 127.50 , 125.79 , 121.84 , 32.74 , 30.33 , 28.49 .

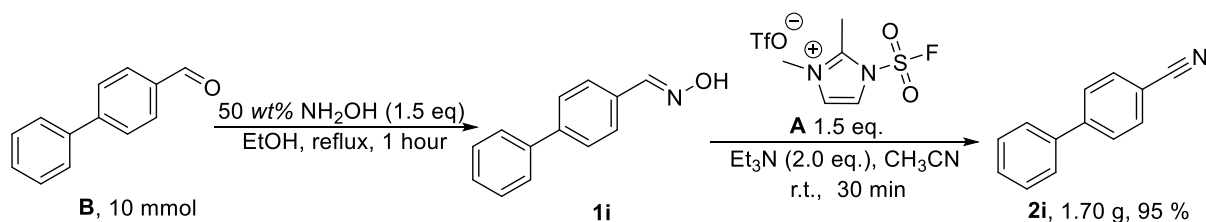


ϵ -caprolactam (4t)¹²

White solid (40.7 mg, 72%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.17 (s, 1H), 3.25 – 3.04 (m, 2H), 2.40 (d, $J = 5.4$ Hz, 2H), 1.73 – 1.54 (m, 6H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 179.53 , 42.72 , 36.73 , 30.58 , 29.67 , 23.19 .

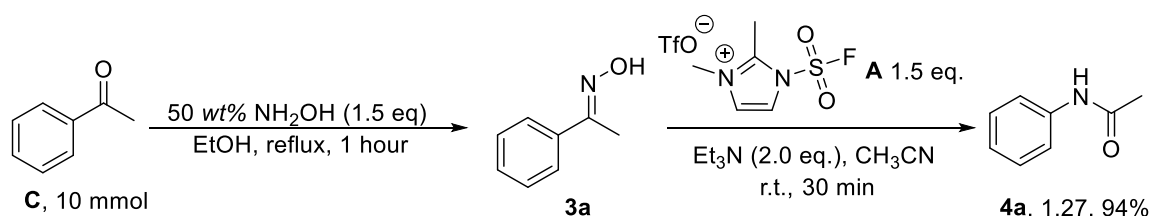
6. Gram-scale production of 2i and 4a by crystallization

Procedure for the synthesis of 2i



4-phenylbenzaldehyde **B** (1.82 g, 10 mmol), EtOH (100 mL, 0.1 M) and 50 wt% aqueous hydroxylamine solution (0.77 g, 15 mmol) were added into a 250 mL round-bottom flask equipped with magnetic stirrer, then the mixture was heated at reflux for an hour. After 4-phenylbenzaldehyde **B** was completely consumed (monitored by TLC) and the reaction mixture was concentrated under reduced pressure to give the crude 4-phenylbenzaldoxime **1i**. The crude 4-phenylbenzaldoxime **1i** (without any purification), CH₃CN (50 mL) and Et₃N (2.8 mL, 20 mmol) were added into a 250 mL round-bottom flask equipped with magnetic stirrer and rubber stopper. Then the fluorosulfonyl imidazolium salt **A** (4.92 g, 15 mmol) was dissolved in acetonitrile (50 mL) and that was injected slowly with a syringe into flask, and the reaction mixture was stirred at room temperature for 30 minutes. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was concentrated under reduced pressure to remove acetonitrile and diluted with 100 mL of DCM and washed with 5% HCl (100 mL), saturated NaCl solution (100 mL) and dried over anhydrous Na₂SO₄ and the evaporation of the appropriate amount solvent. While white solid was crystallized and washed with petroleum ether to give 4-phenylbenzonitrile **2i** (1.70 g, 95 %).

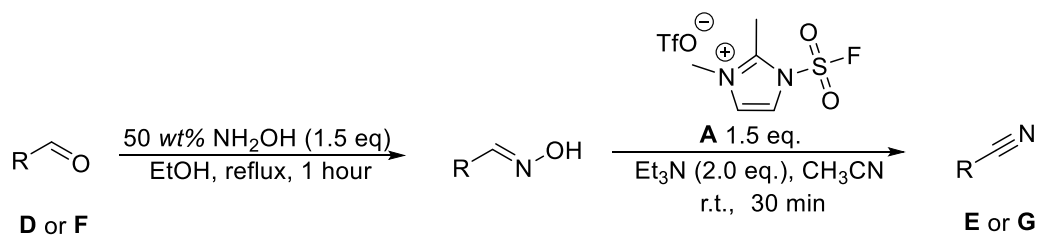
Procedure for the synthesis of 4a



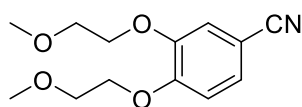
Acetophenone **C** (1.20 g, 10 mmol), EtOH (100 mL, 0.1 M) and 50 wt% aqueous hydroxylamine solution (0.77 g, 15 mmol) were added into a 250 mL round-bottom flask equipped with magnetic stirrer, then the mixture was heated at reflux for an hour. After Acetophenone **C** was completely consumed (monitored by TLC) and the reaction mixture was

concentrated under reduced pressure to give the crude acetophenone oxime **3a**. The crude acetophenone oxime **3a** (without any purification), CH₃CN (50 mL) and Et₃N (2.8 mL, 20 mmol) were added into a 250 mL round-bottom flask equipped with magnetic stirrer and rubber stopper. Then the fluorosulfonyl imidazolium salt **A** (4.92 g, 15 mmol) was dissolved in acetonitrile (50 mL) and that was injected slowly with a syringe into flask, and the reaction mixture was stirred at room temperature for 30 minutes. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was concentrated under reduced pressure to remove appropriate amount acetonitrile. While white solid was crystallized and washed with petroleum ether to give acetanilide **4a** (1.70 g, 94 %).

7. Synthesis of precursors for drugs

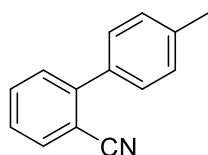


Aldehyde **D** or **F** (0.5 mmol), EtOH (5 mL, 0.1 M) and 50 wt% aqueous hydroxylamine solution (24.7 μL , 0.75 mmol) were added into a 10 mL round-bottom flask equipped with magnetic stirrer, then the mixture was heated at reflux for an hour. After aldehyde was completely consumed (monitored by TLC) and the reaction mixture was concentrated under reduced pressure to give the crude aldoxime. The crude aldoxime (without any purification), CH_3CN (3 mL) and Et_3N (139 μL , 1 mmol) were added into a 10 mL round-bottom flask equipped with magnetic stirrer and rubber stopper. Then the fluorosulfonyl imidazolium salt **A** (246 mg, 0.75 mmol) was dissolved in acetonitrile (2 mL) and that was injected slowly with a syringe into flask, and the reaction mixture was stirred at room temperature for 30 minutes. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was diluted with 20 mL of DCM and washed with 5% HCl (2 \times 20 mL), 5% NaHCO_3 (2 \times 20 mL), saturated NaCl solution (2 \times 20 mL) and dried over anhydrous Na_2SO_4 and the evaporation of the solvent gave a residue that was purified on silica gel column chromatography using petroleum ether and ethyl acetate.



3,4-Bis(2-methoxyethoxy)benzaldehyde (**E**)³

yellow oil (119.3 mg, 95%). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.25 (m, 1H), 7.16 (d, $J = 1.7$ Hz, 1H), 6.94 (d, $J = 8.4$ Hz, 1H), 4.19 (dt, $J = 15.5, 4.7$ Hz, 4H), 3.80 (q, $J = 5.5$ Hz, 4H), 3.46 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 152.95, 148.92, 126.85, 119.18, 117.08, 113.53, 104.20, 70.82, 70.71, 69.08, 68.64, 59.32, 59.28.



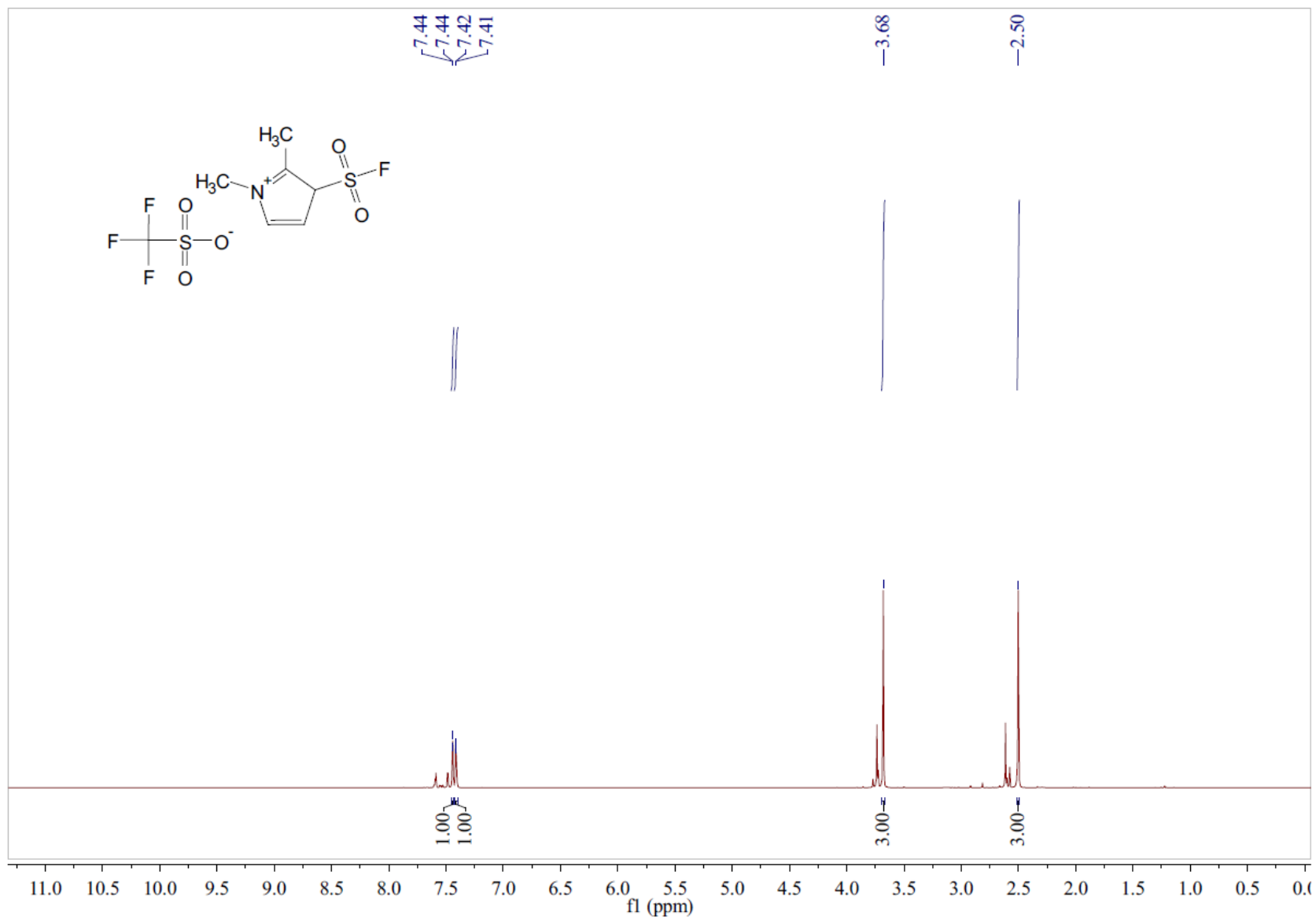
4'-methyl-2-biphenylcarbonitrile (G)¹⁸

White solid (93.7 mg, 97%). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.81 – 7.74 (m, 1H), 7.66 (td, *J* = 7.7, 1.4 Hz, 1H), 7.52 (ddd, *J* = 14.5, 7.2, 1.2 Hz, 3H), 7.44 (td, *J* = 7.6, 1.2 Hz, 1H), 7.33 (d, *J* = 7.9 Hz, 2H), 2.45 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 145.56 , 138.73 , 135.30 , 133.75 , 132.81 , 130.01 , 129.48 , 128.65 , 127.32 , 118.93 , 111.21 , 21.29 .

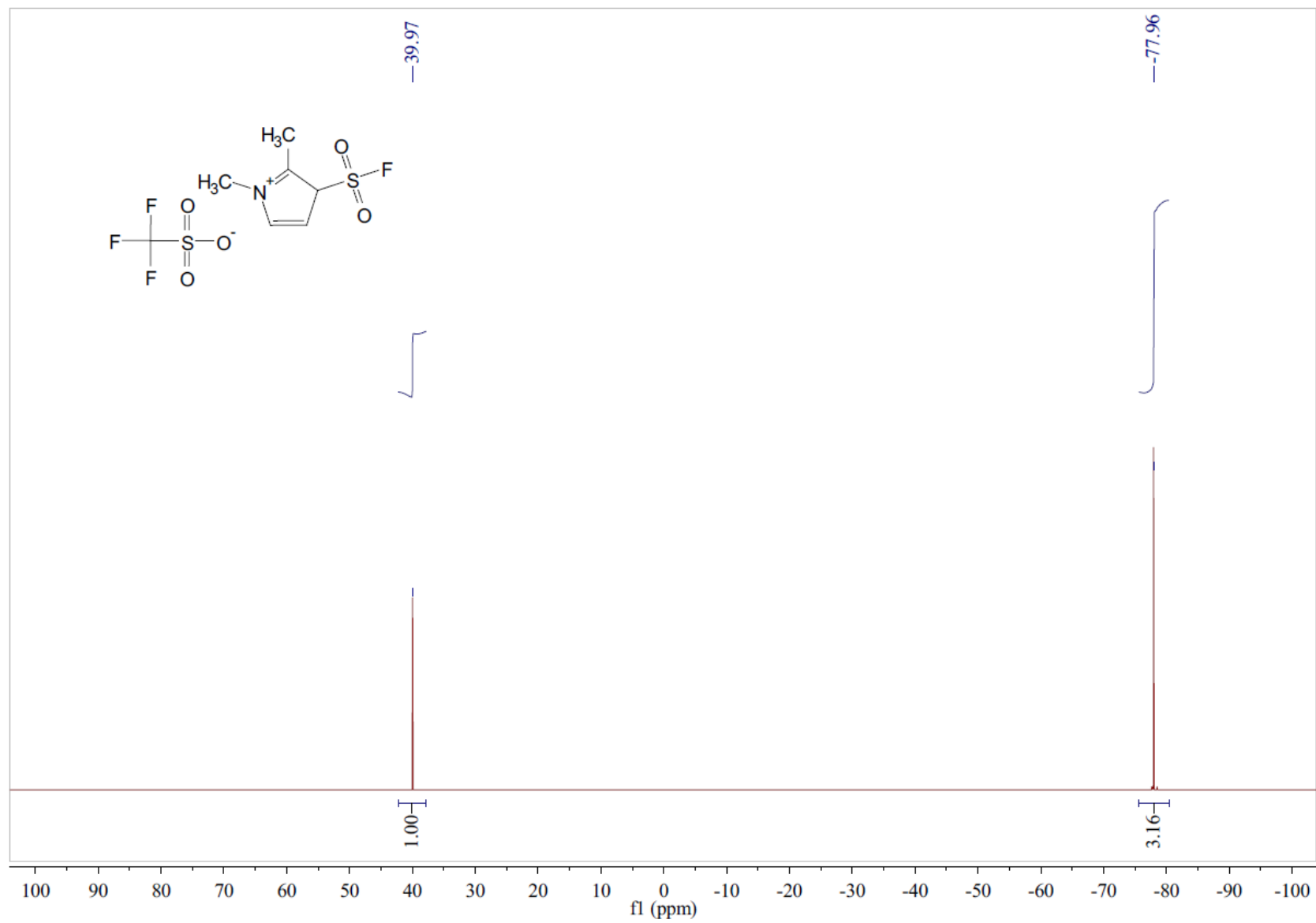
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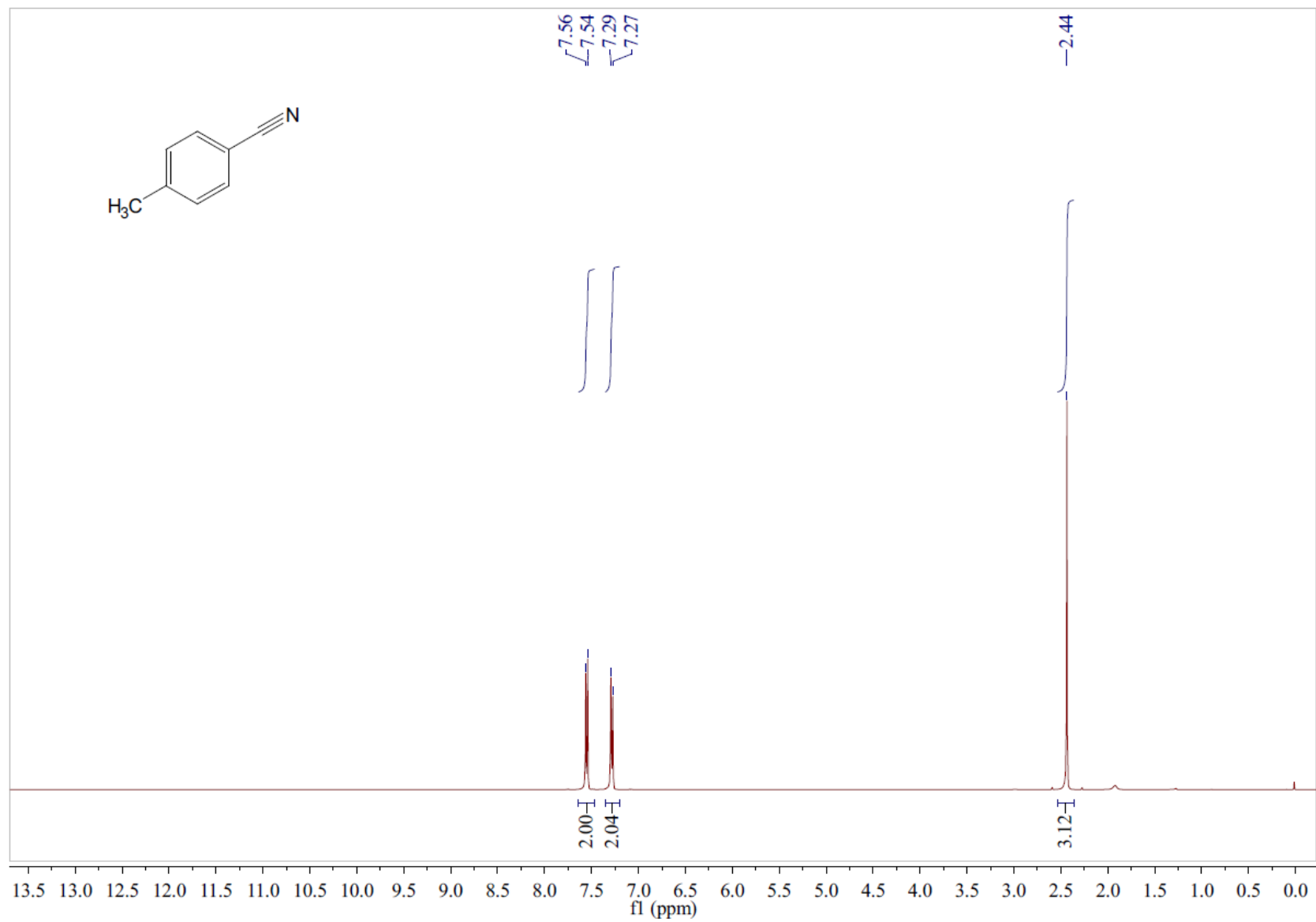
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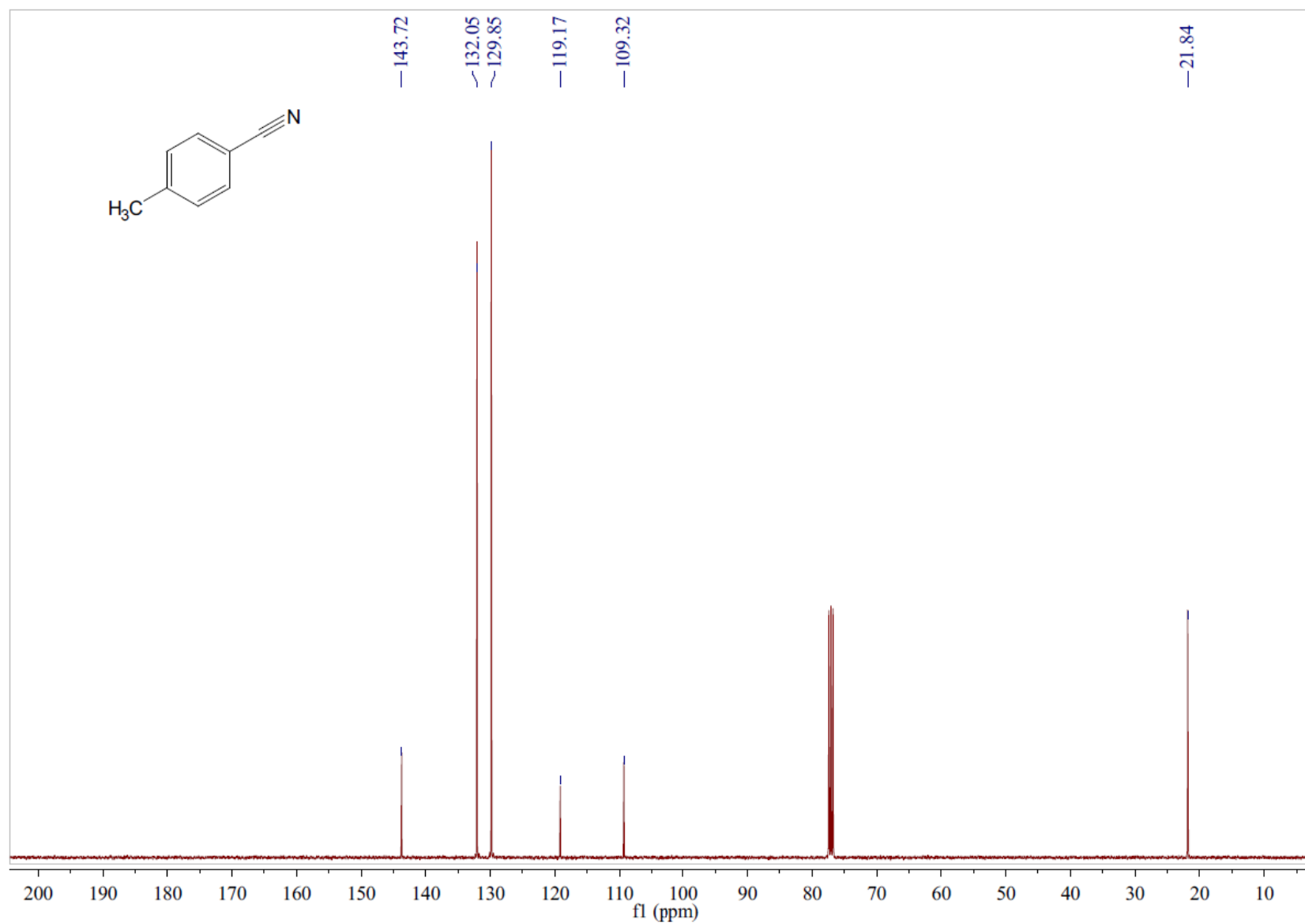
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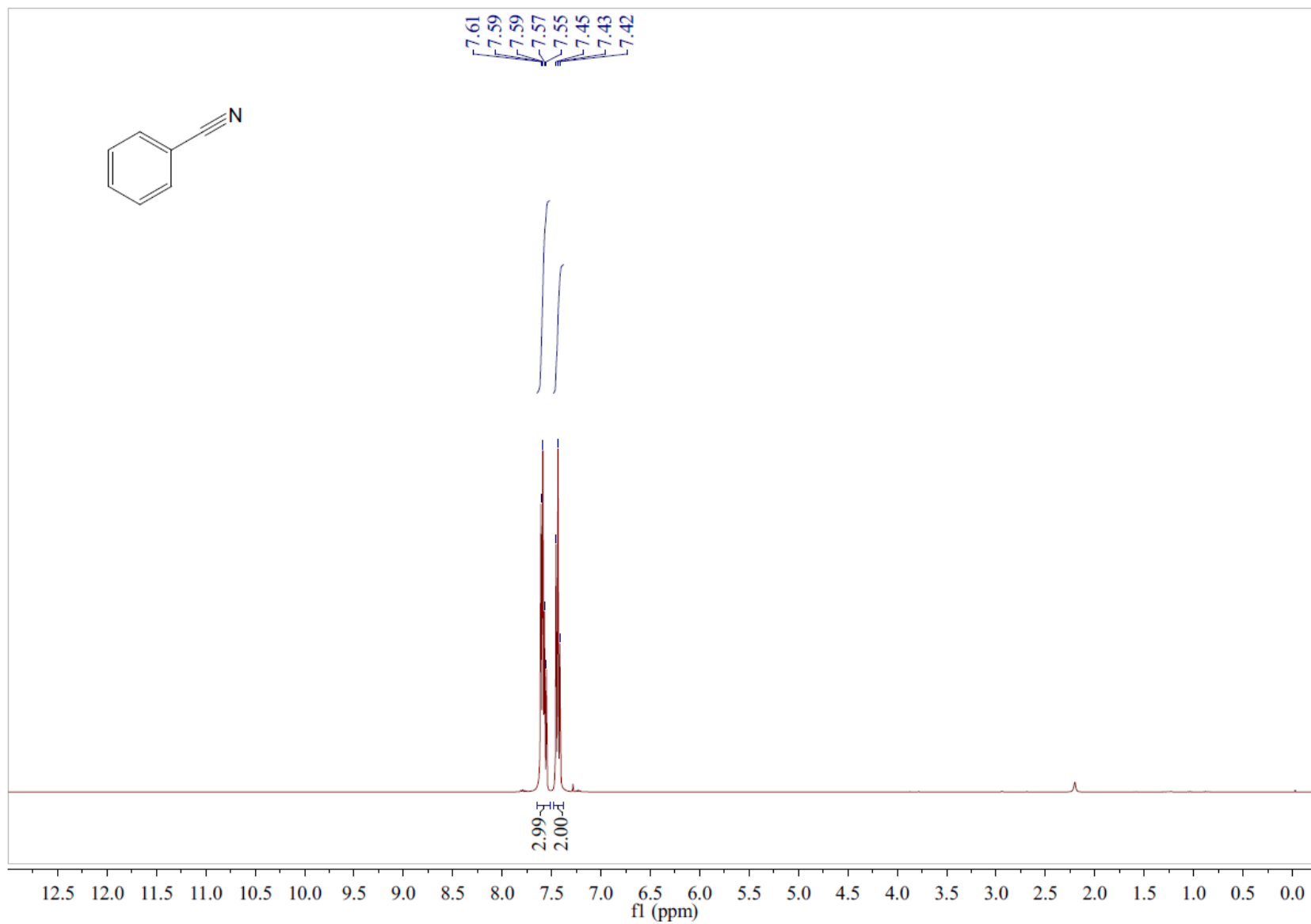
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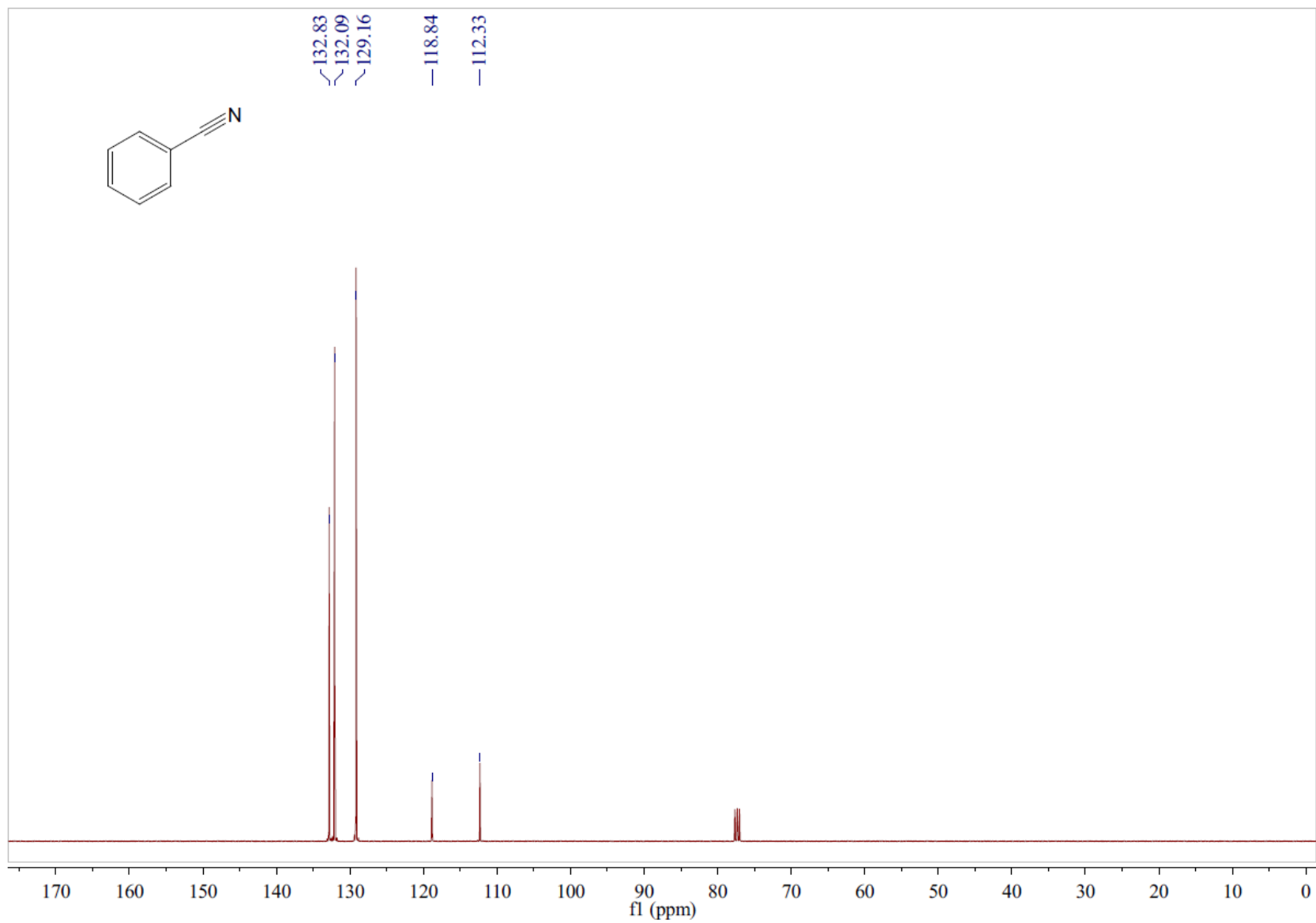
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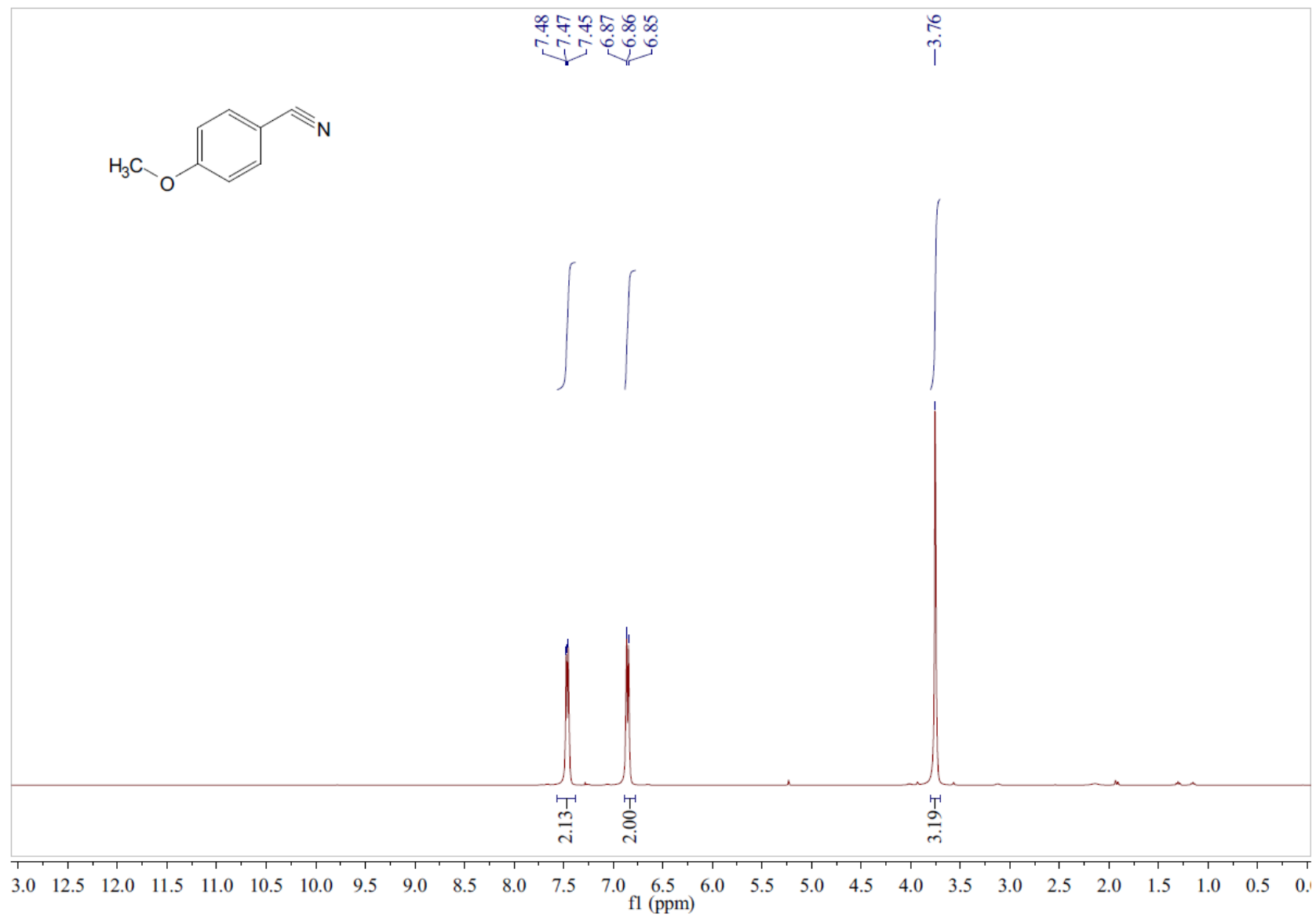
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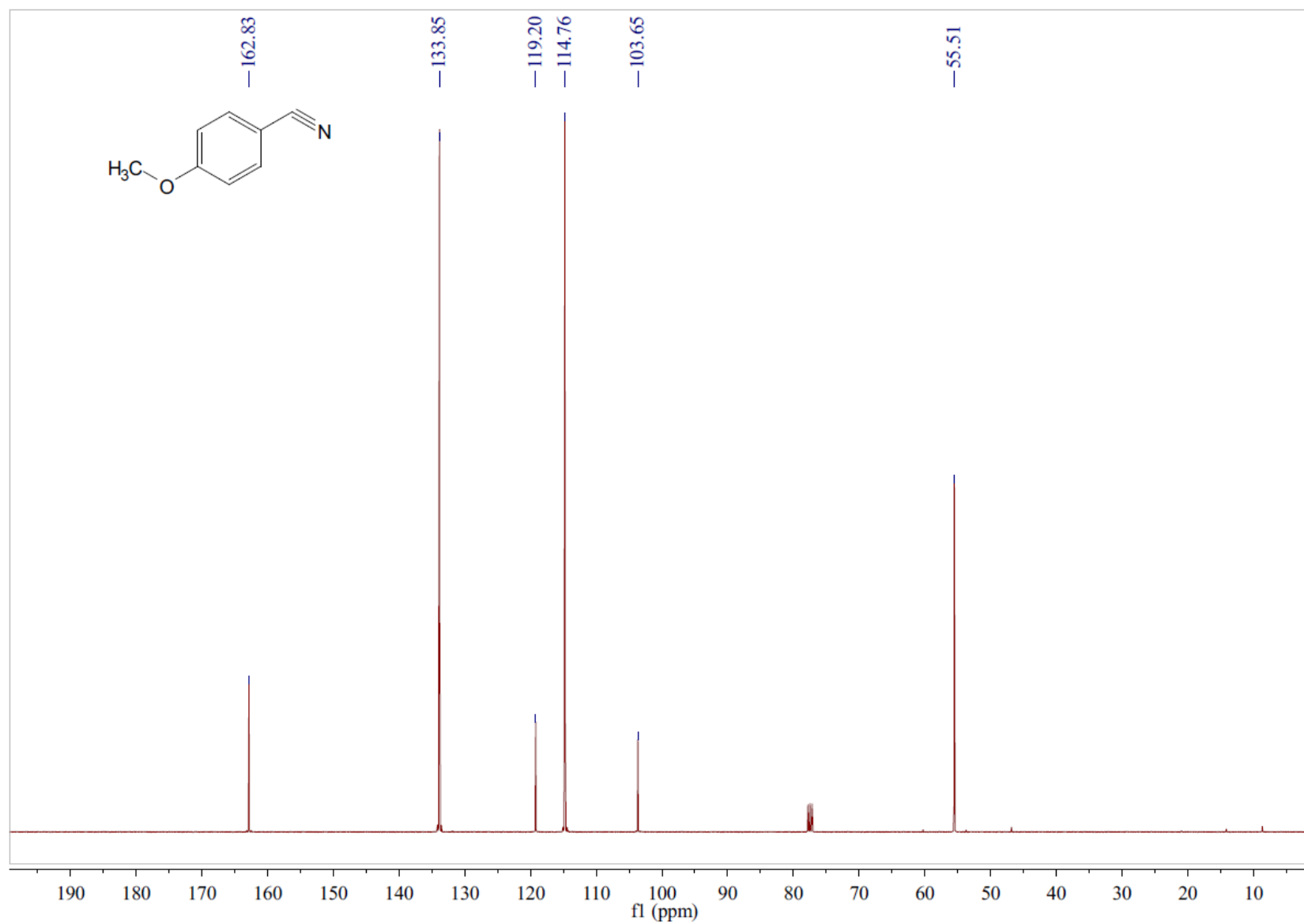
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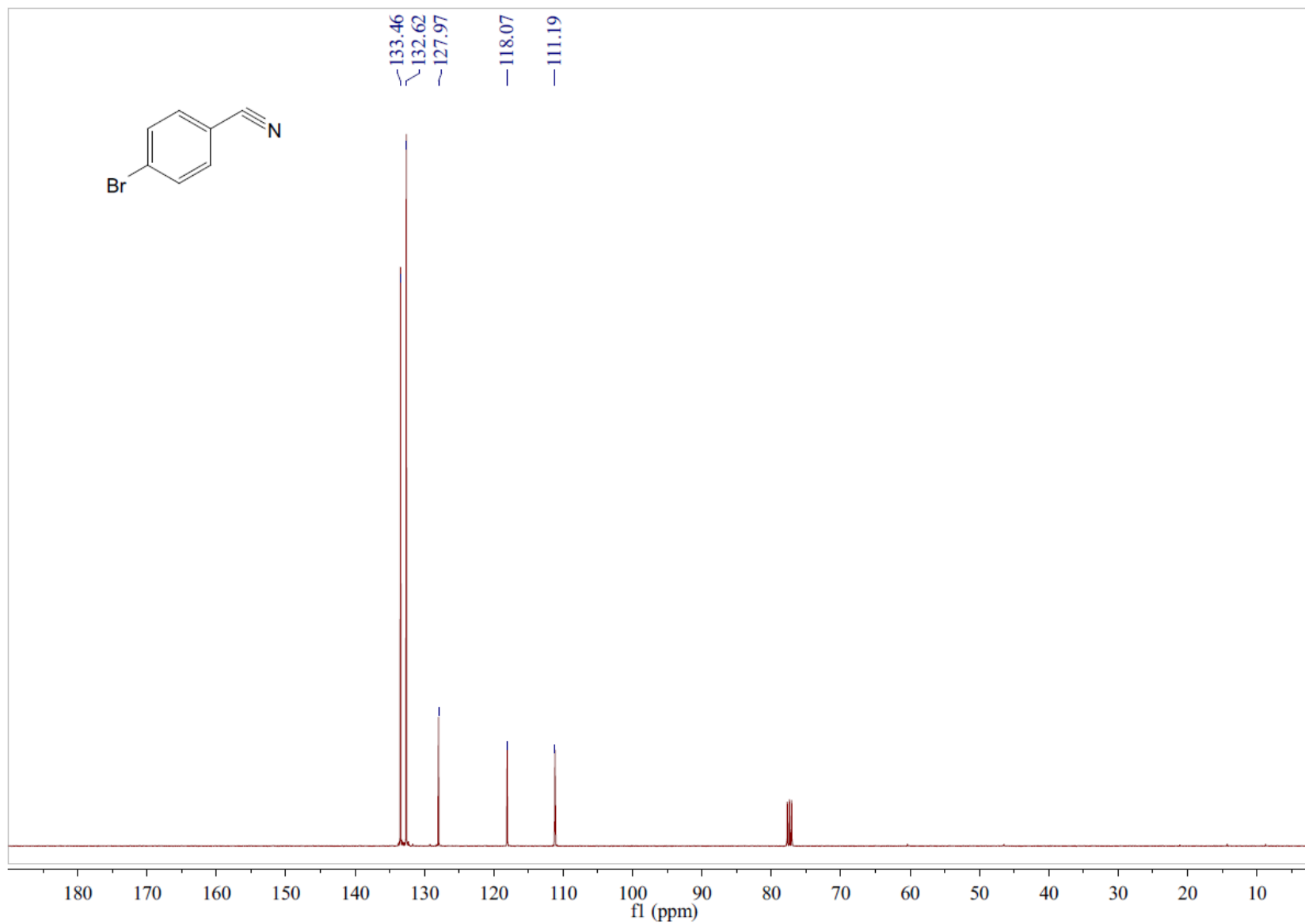
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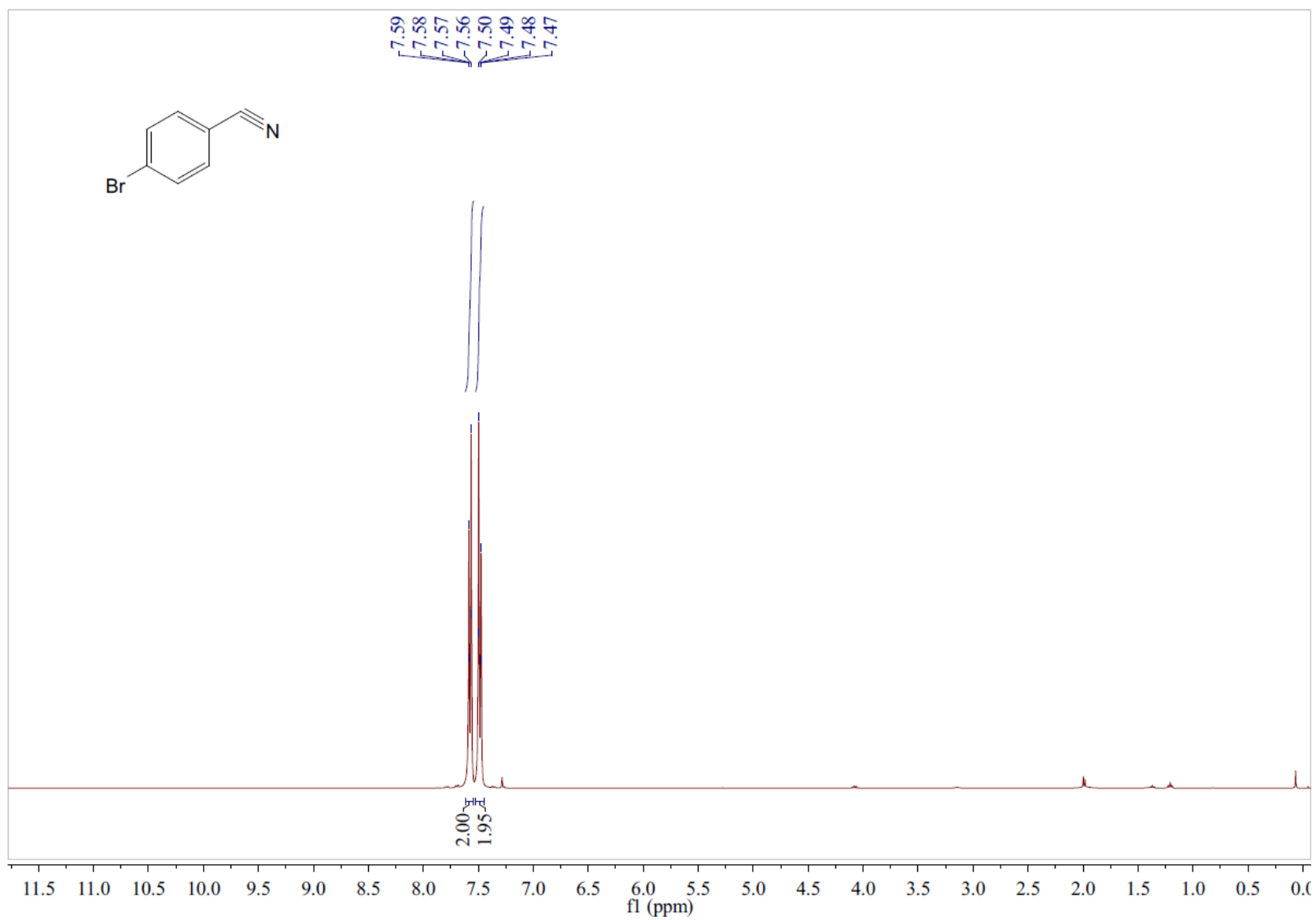
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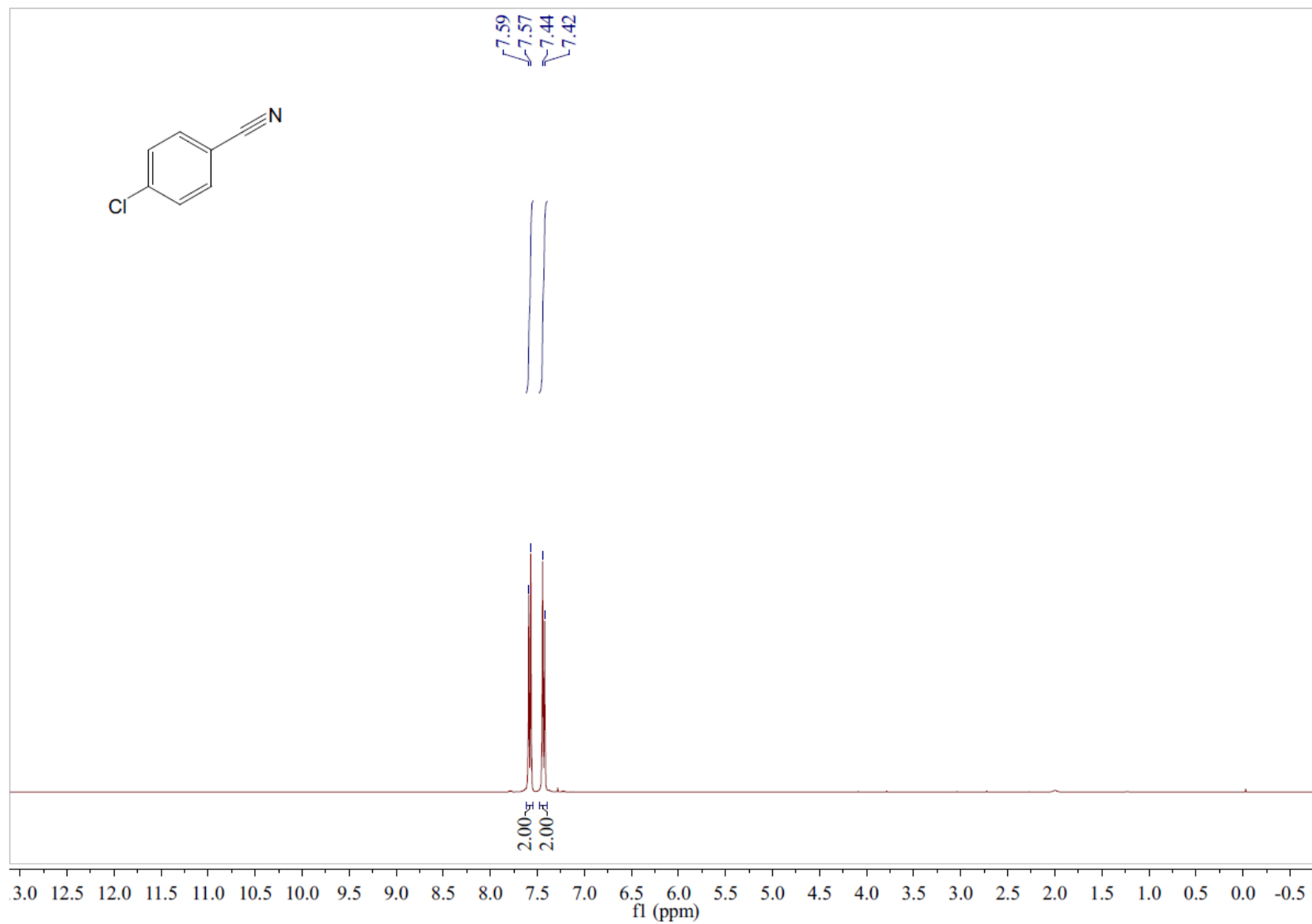
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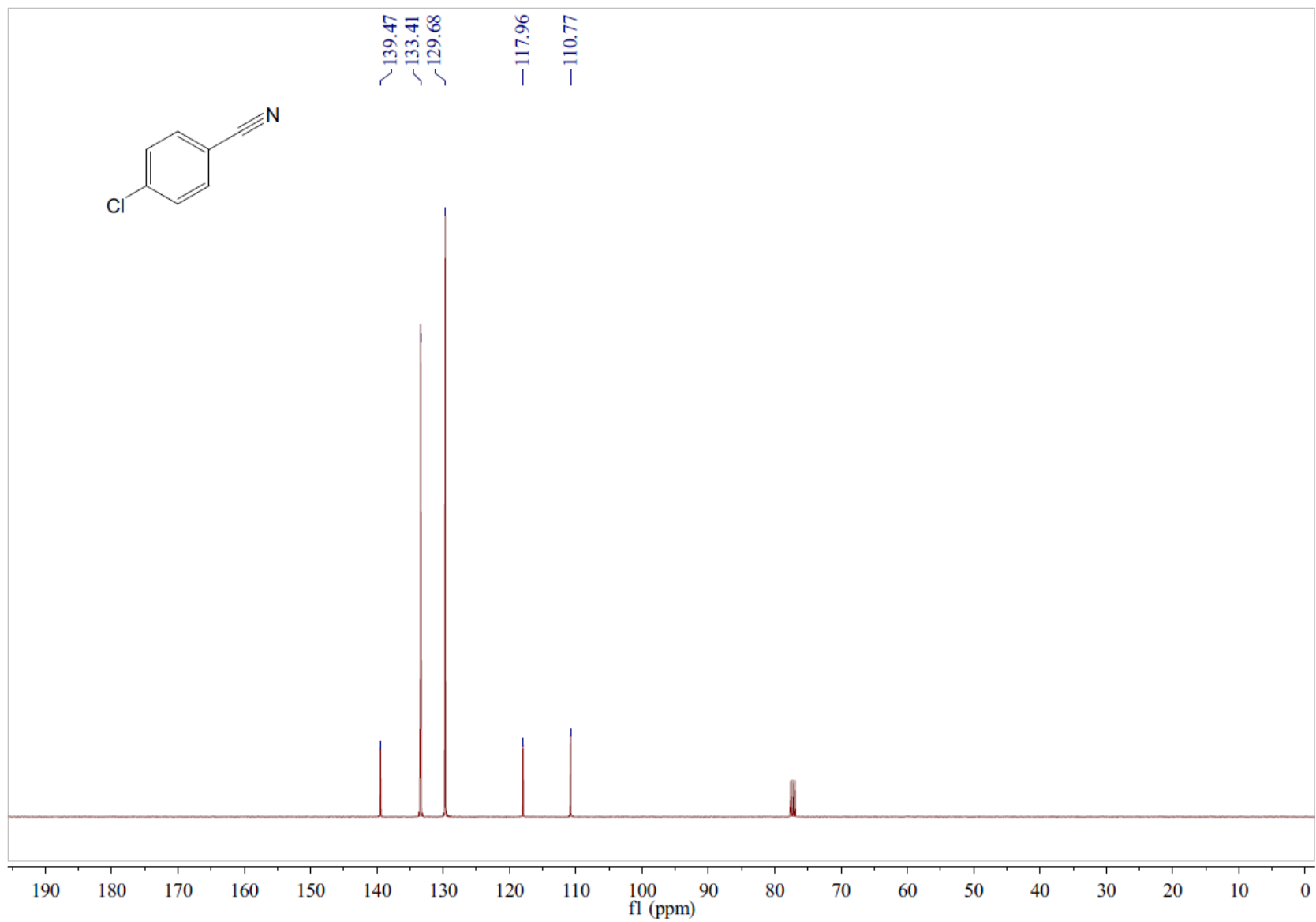
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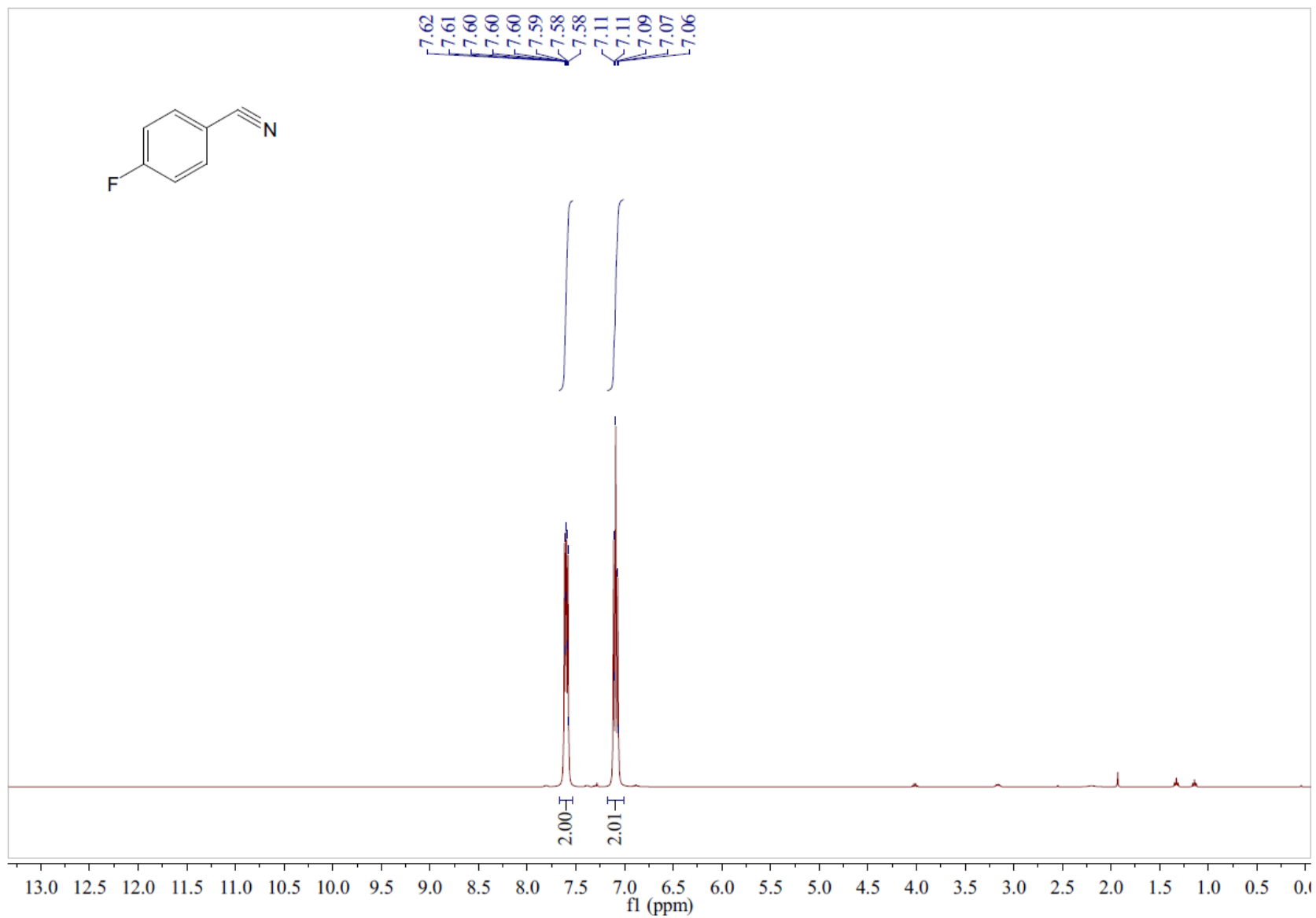
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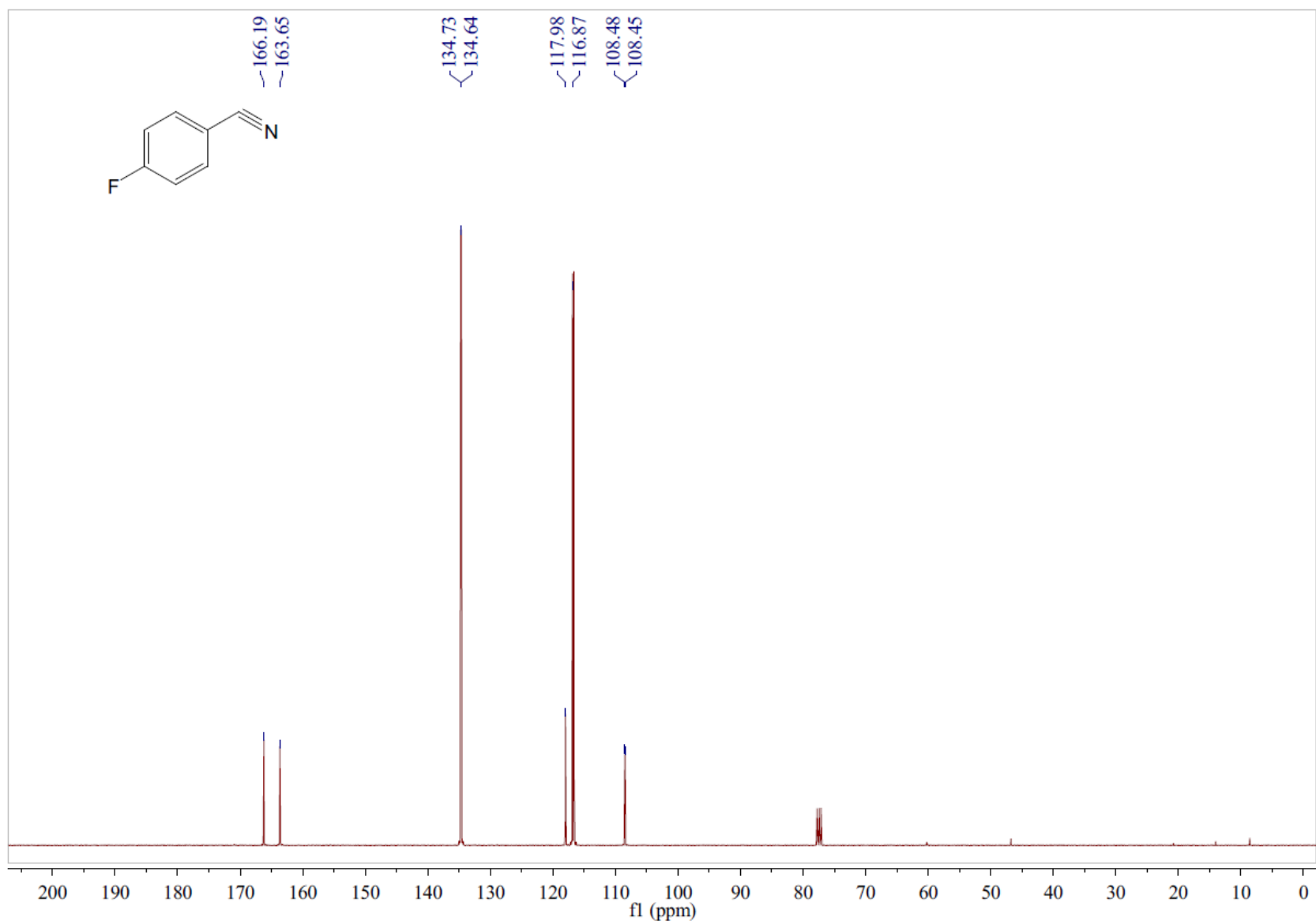
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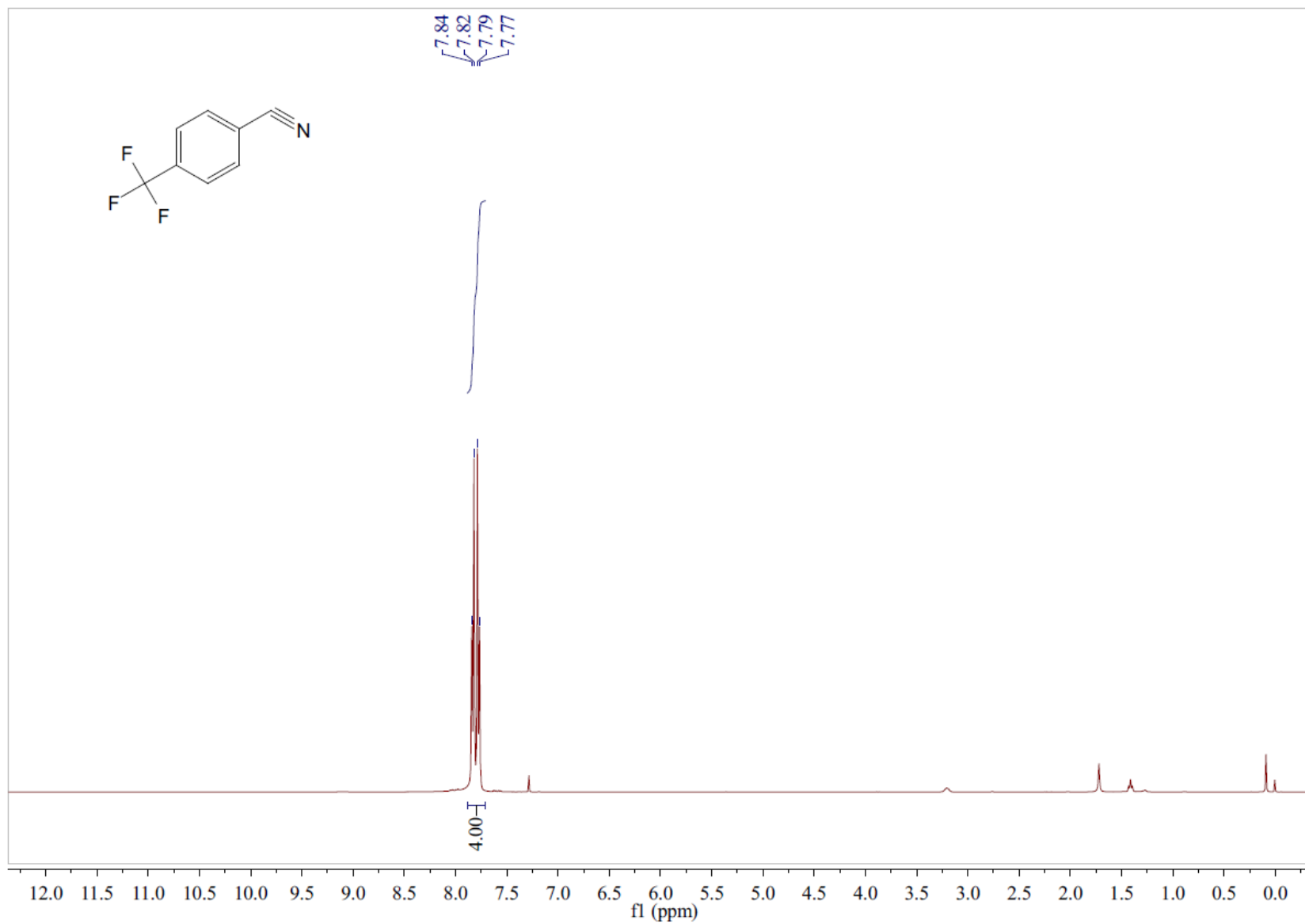
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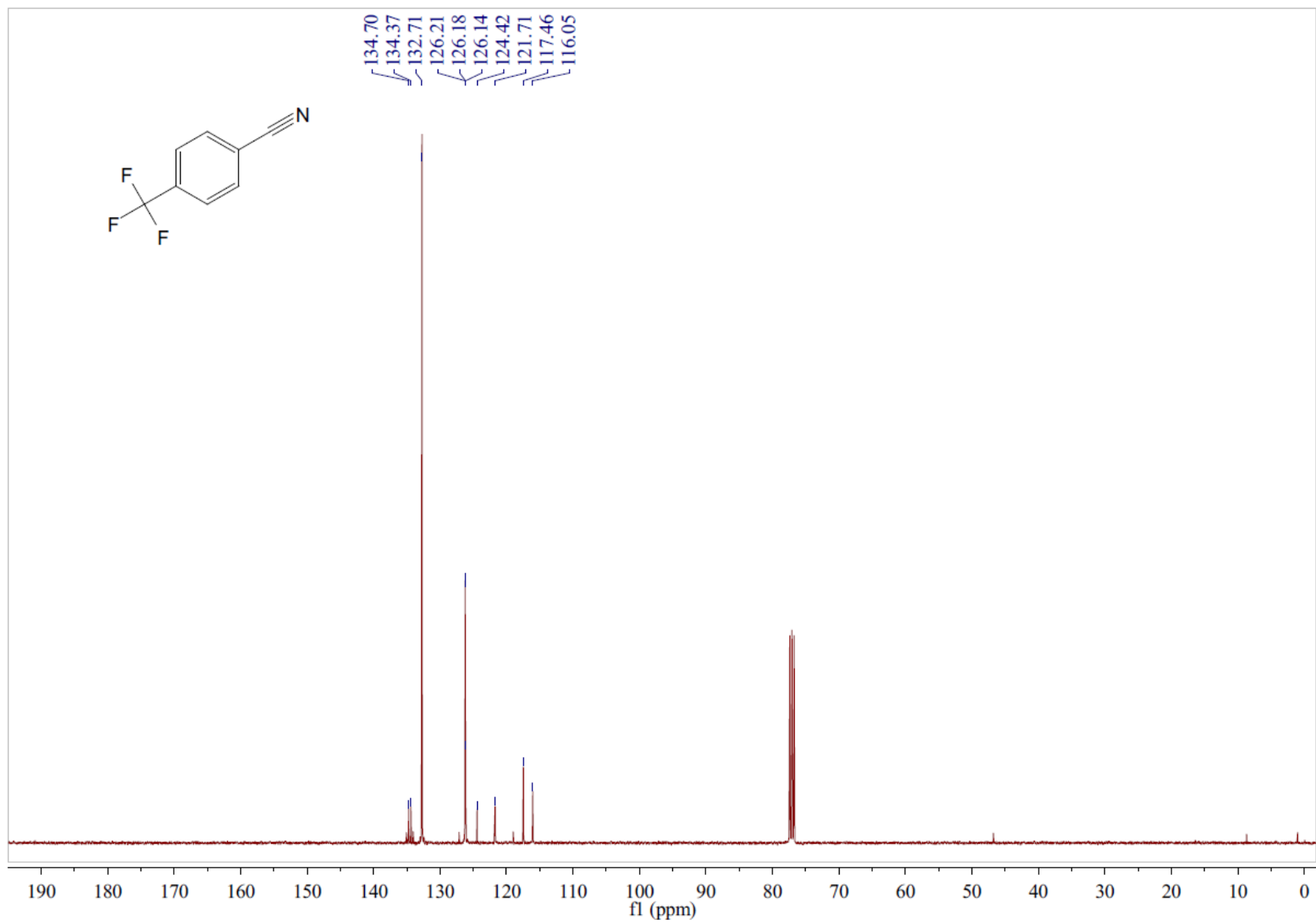
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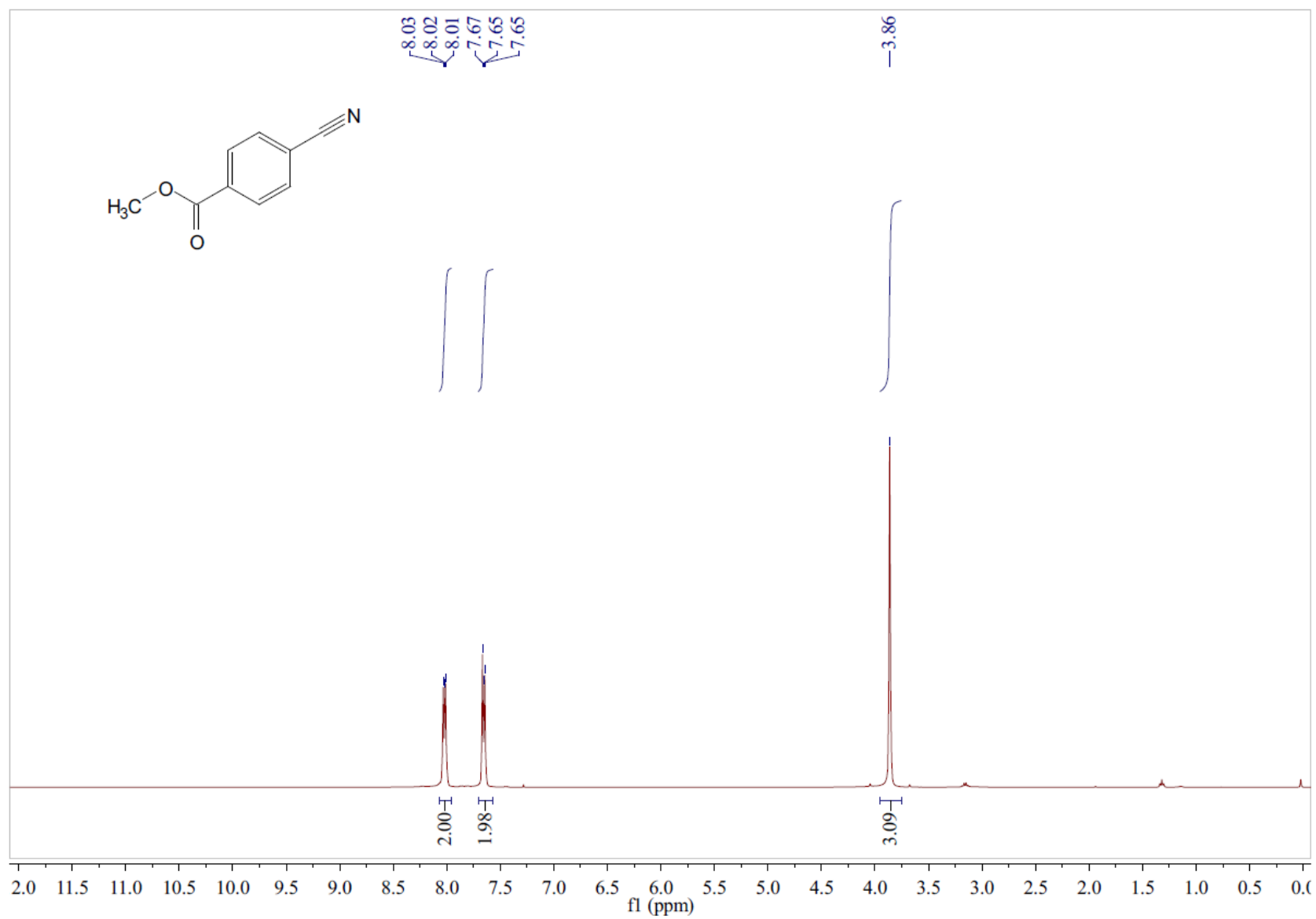
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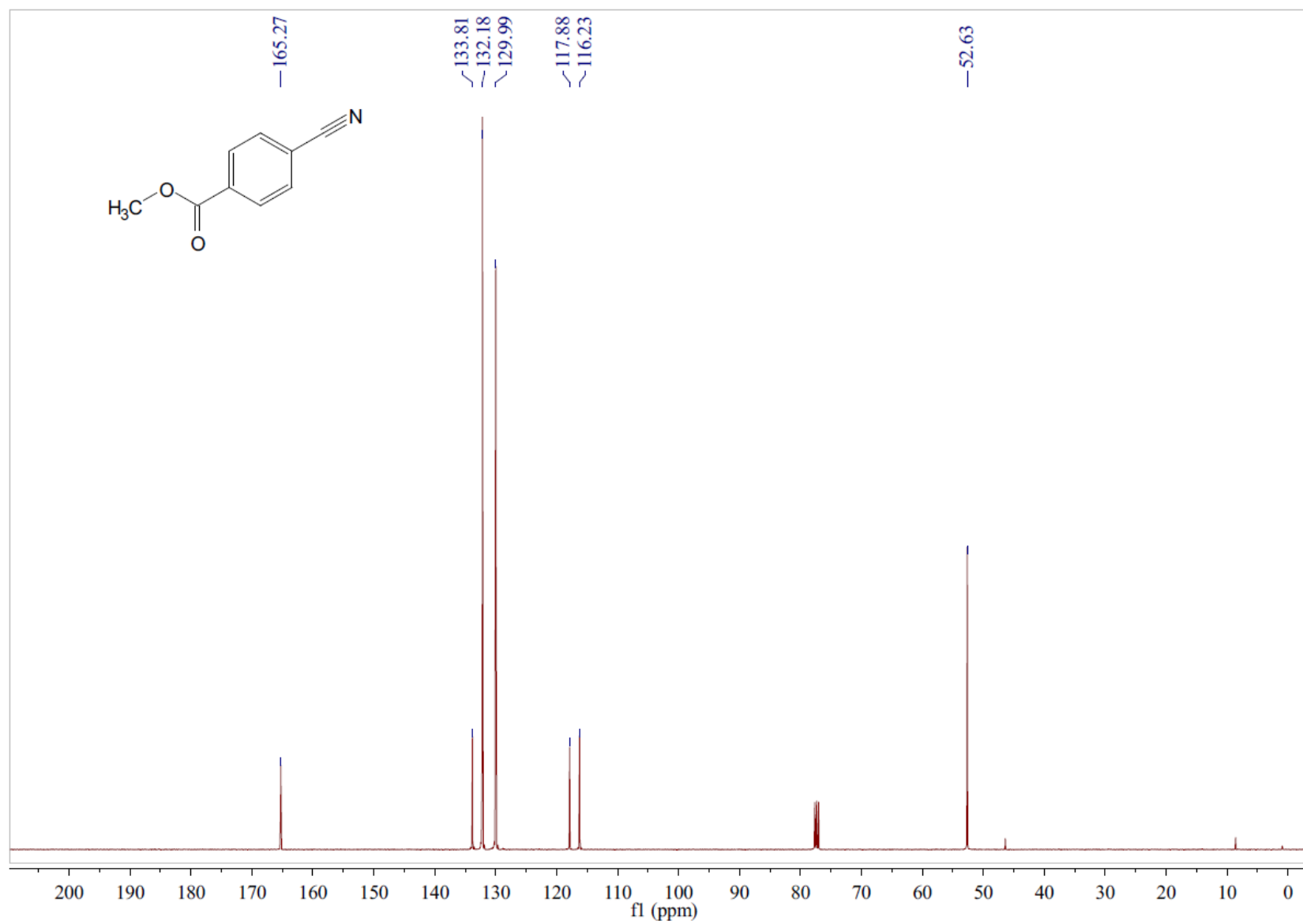
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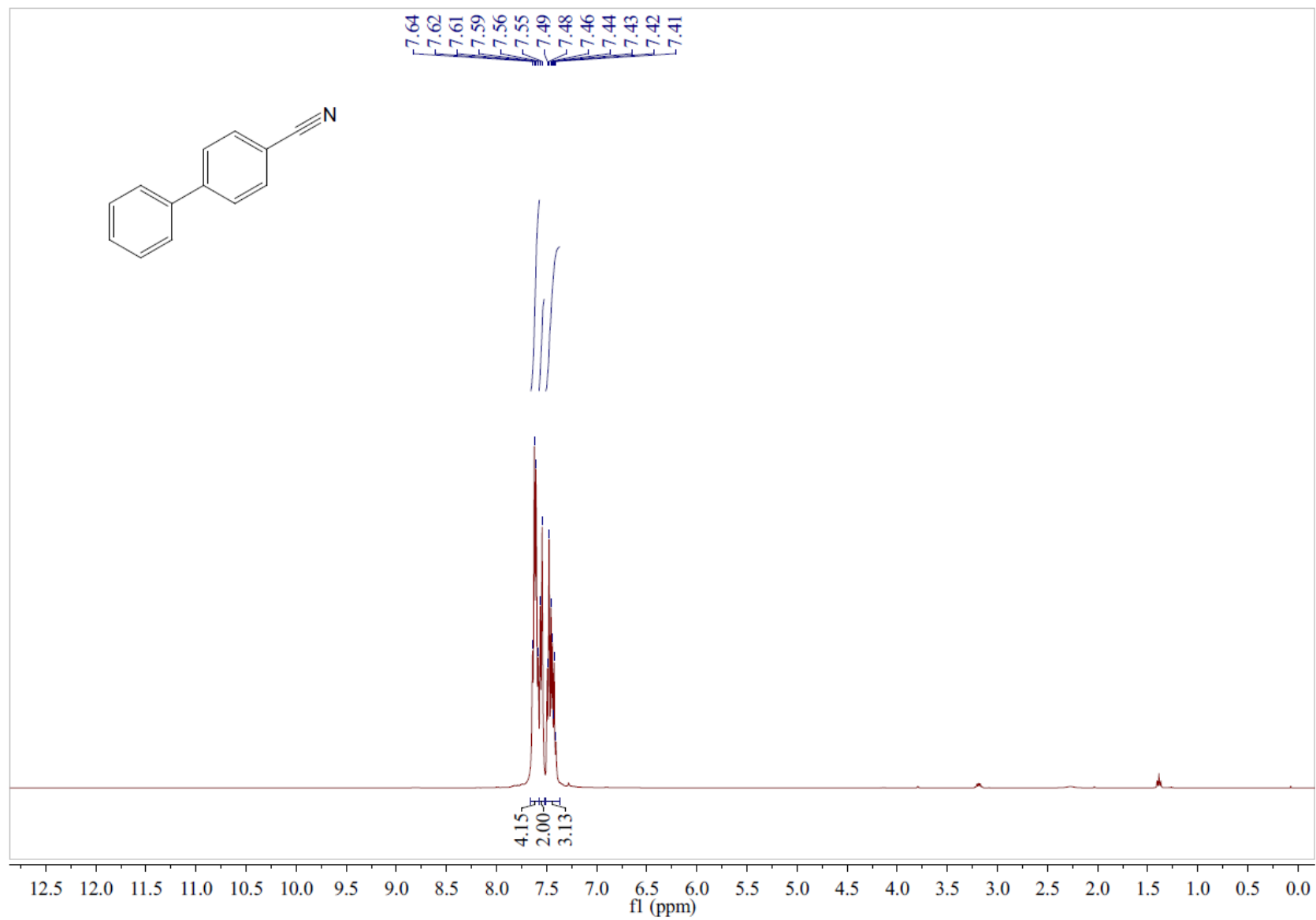
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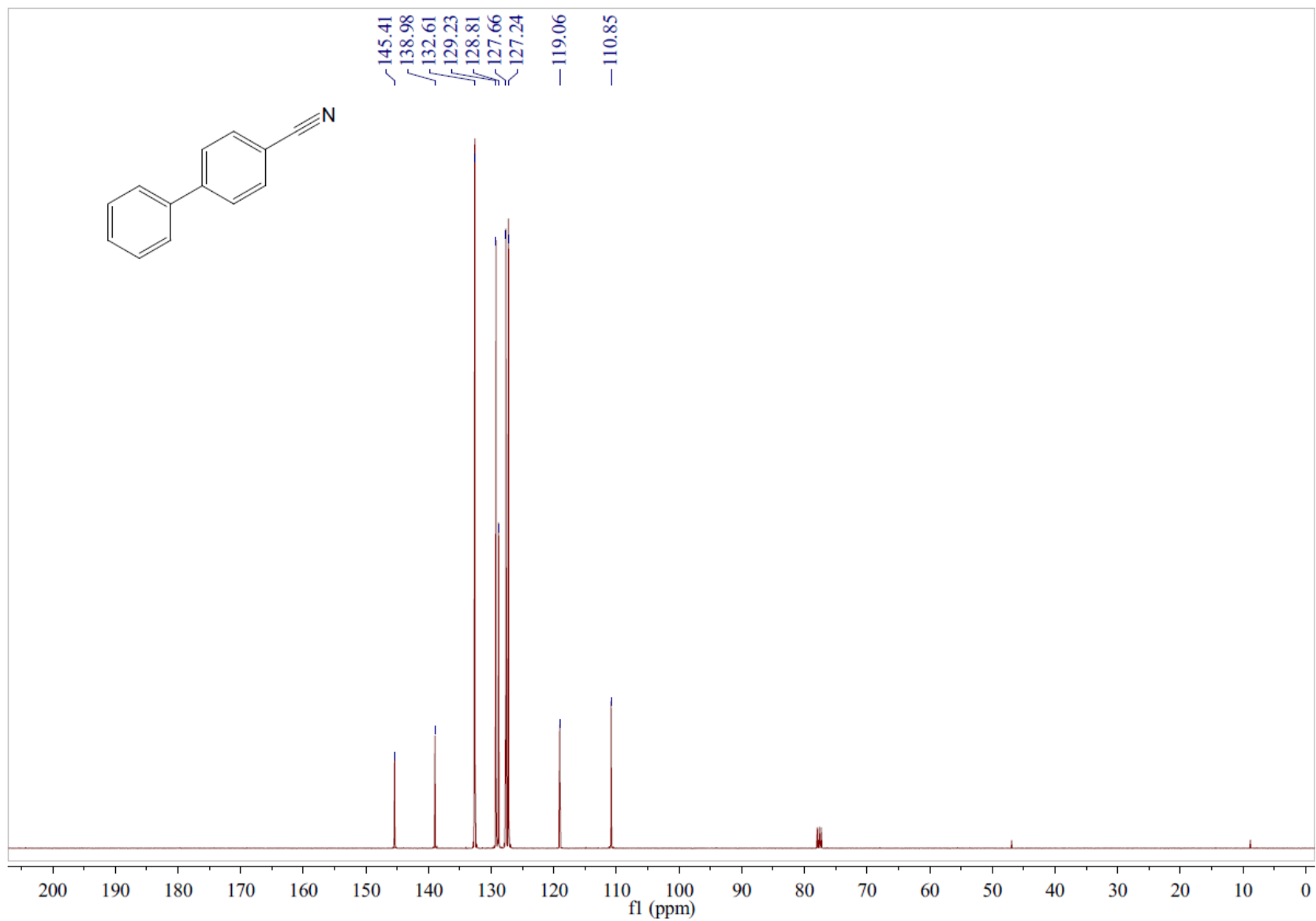
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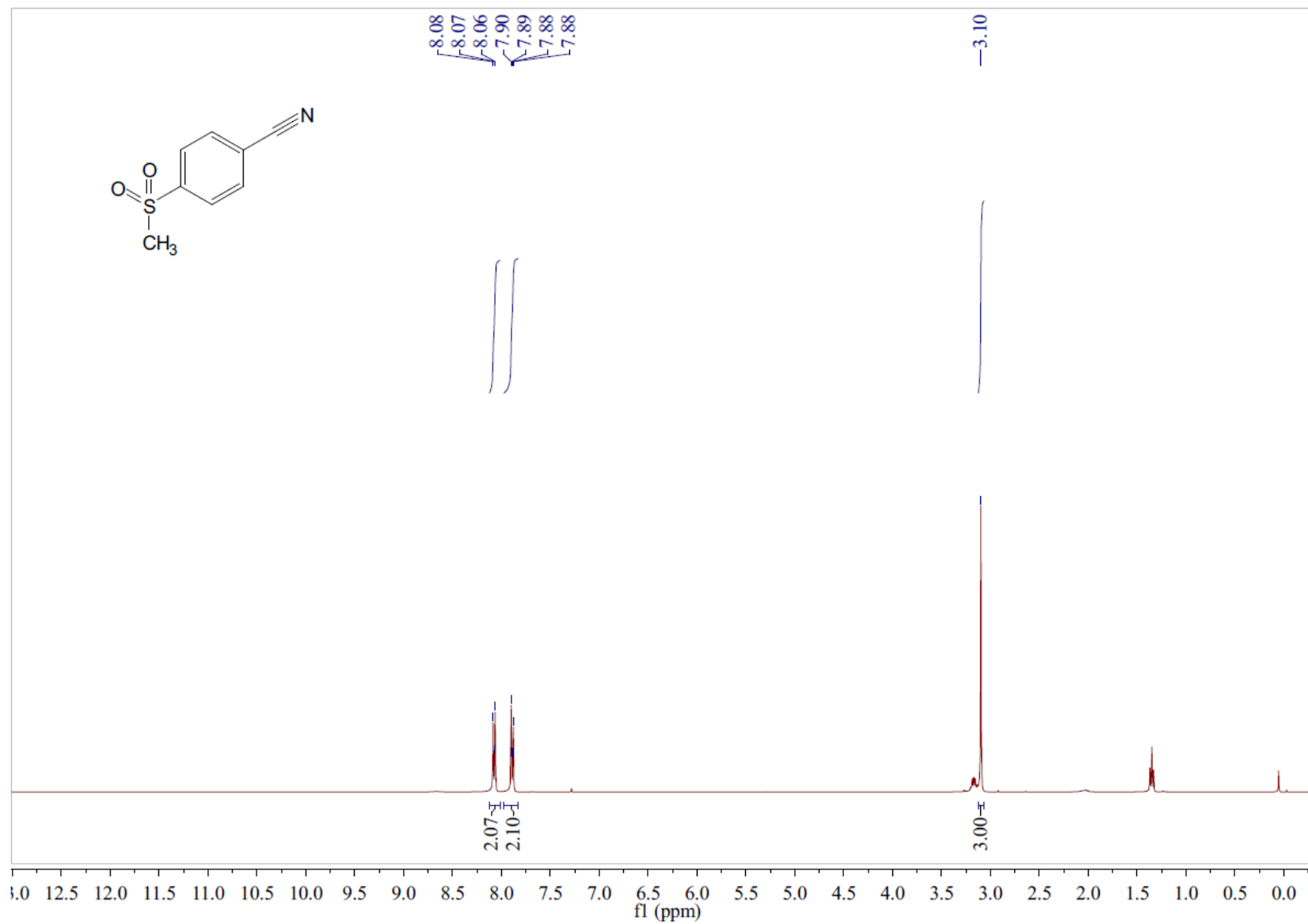
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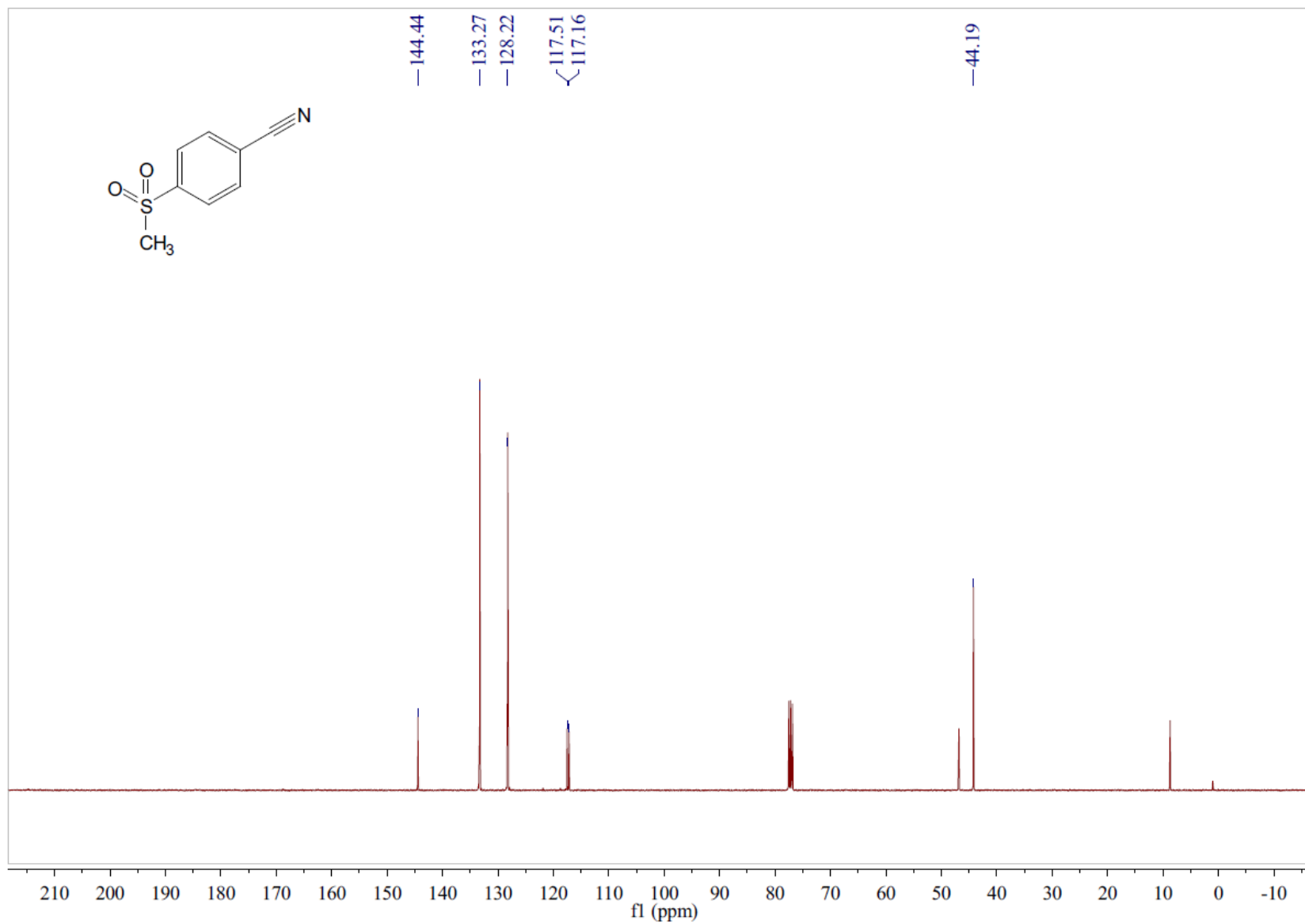
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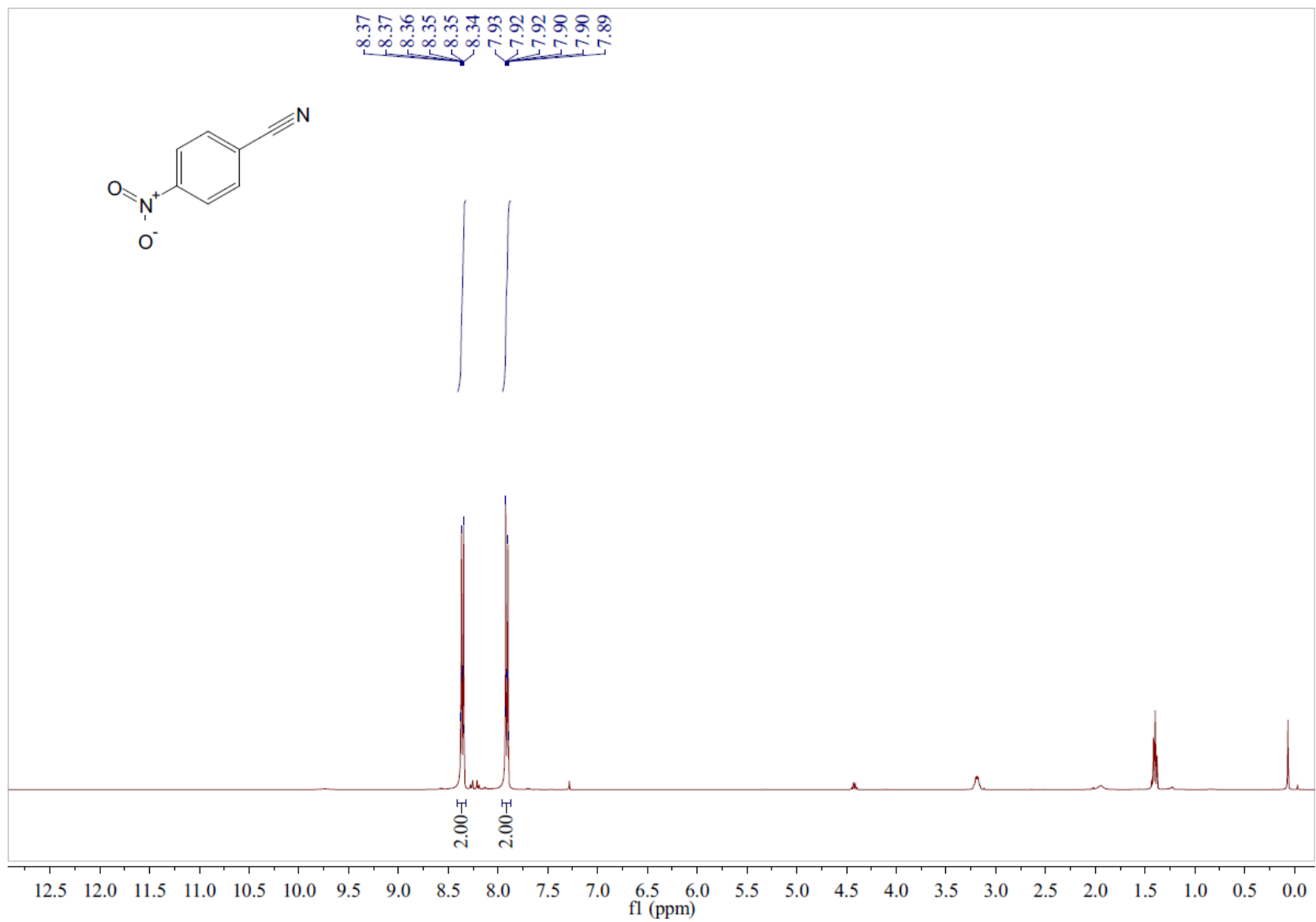
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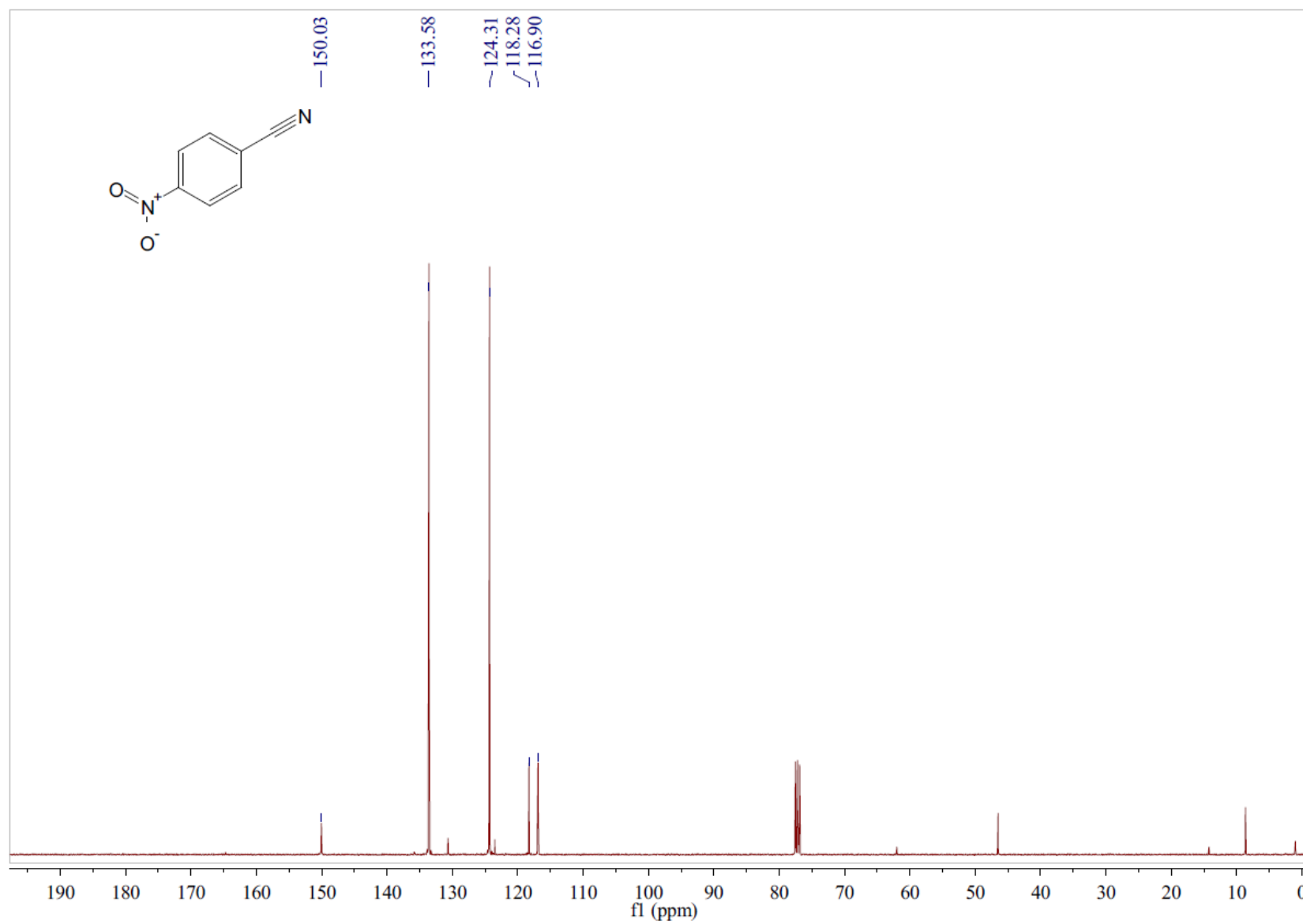
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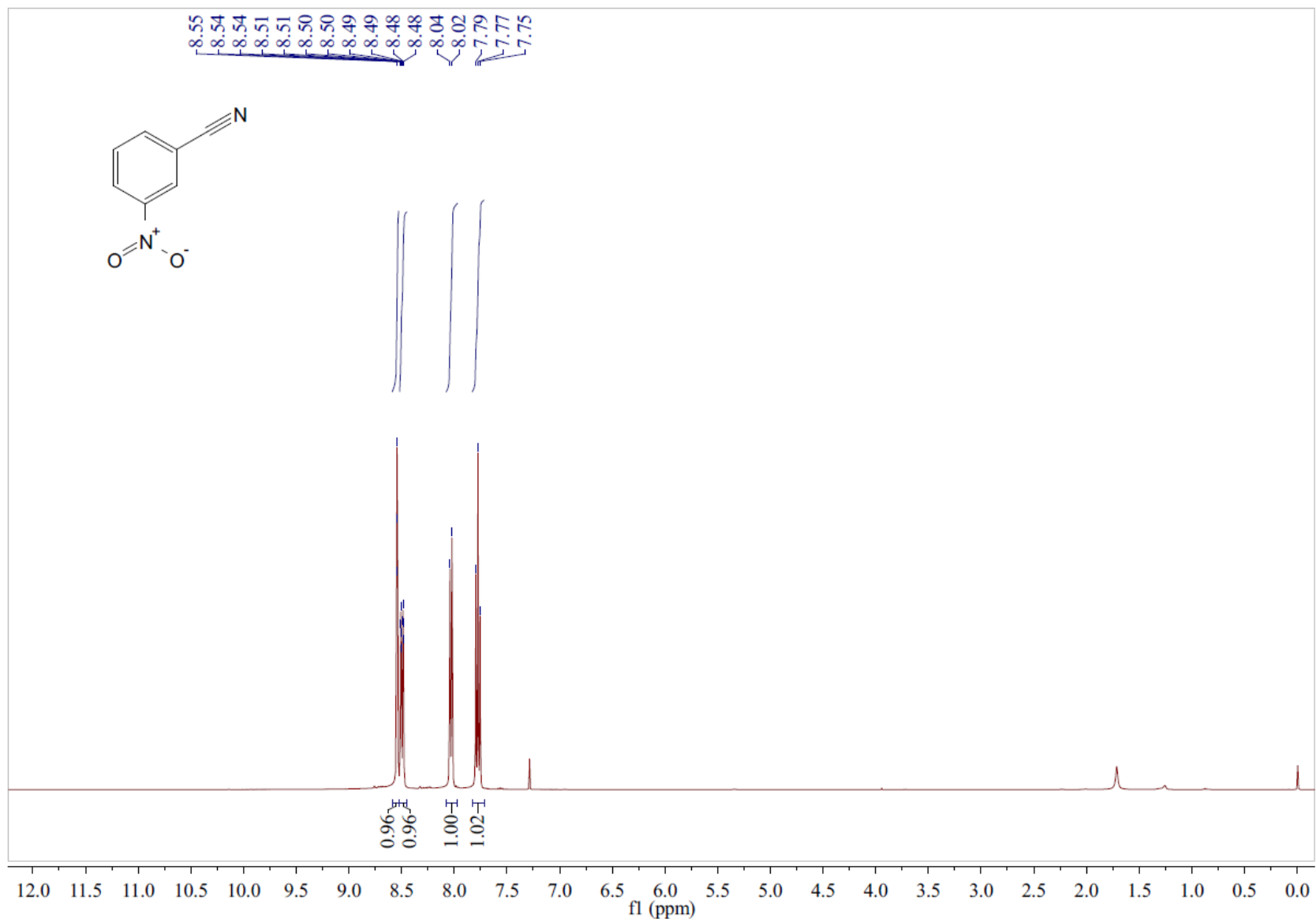
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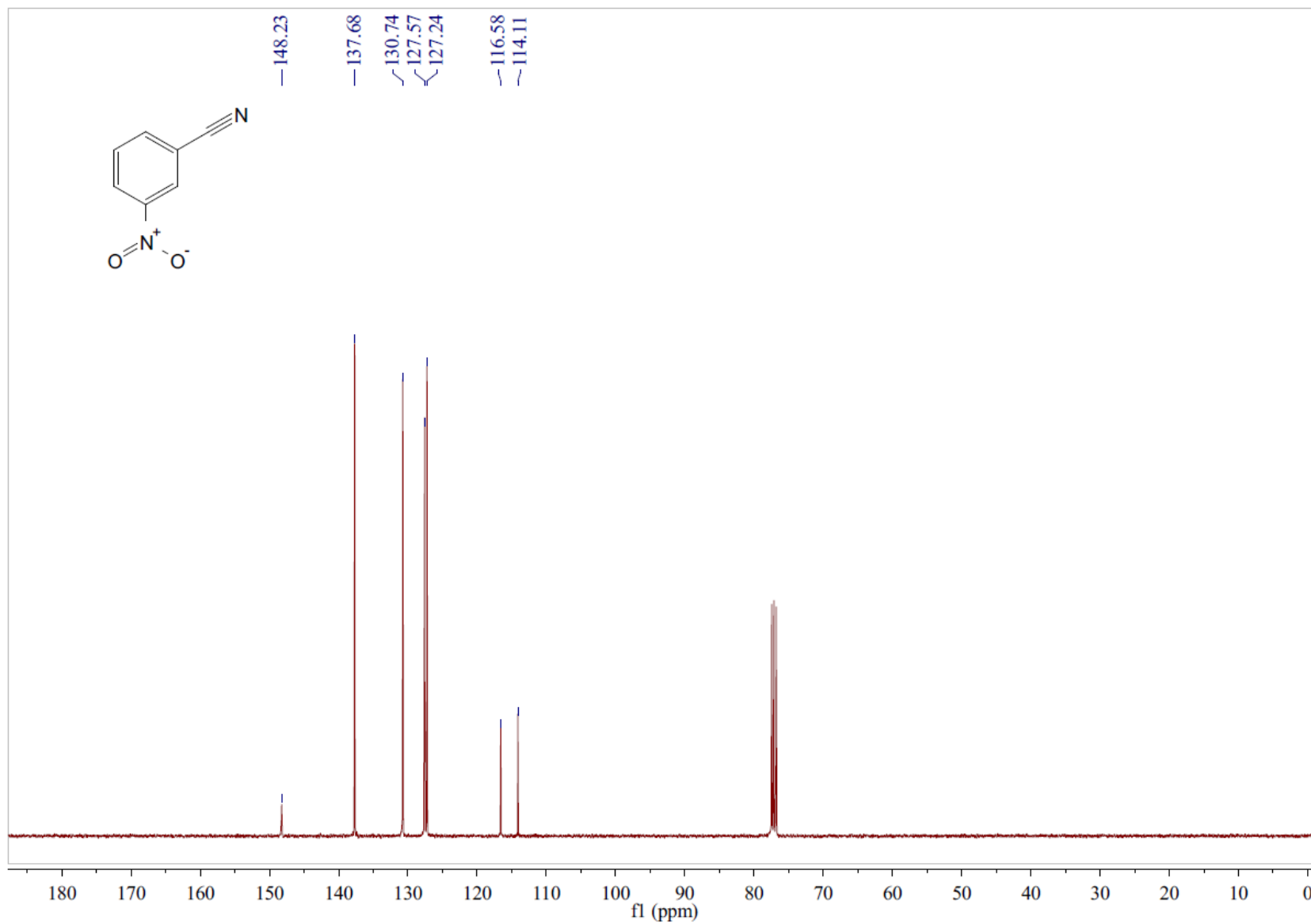
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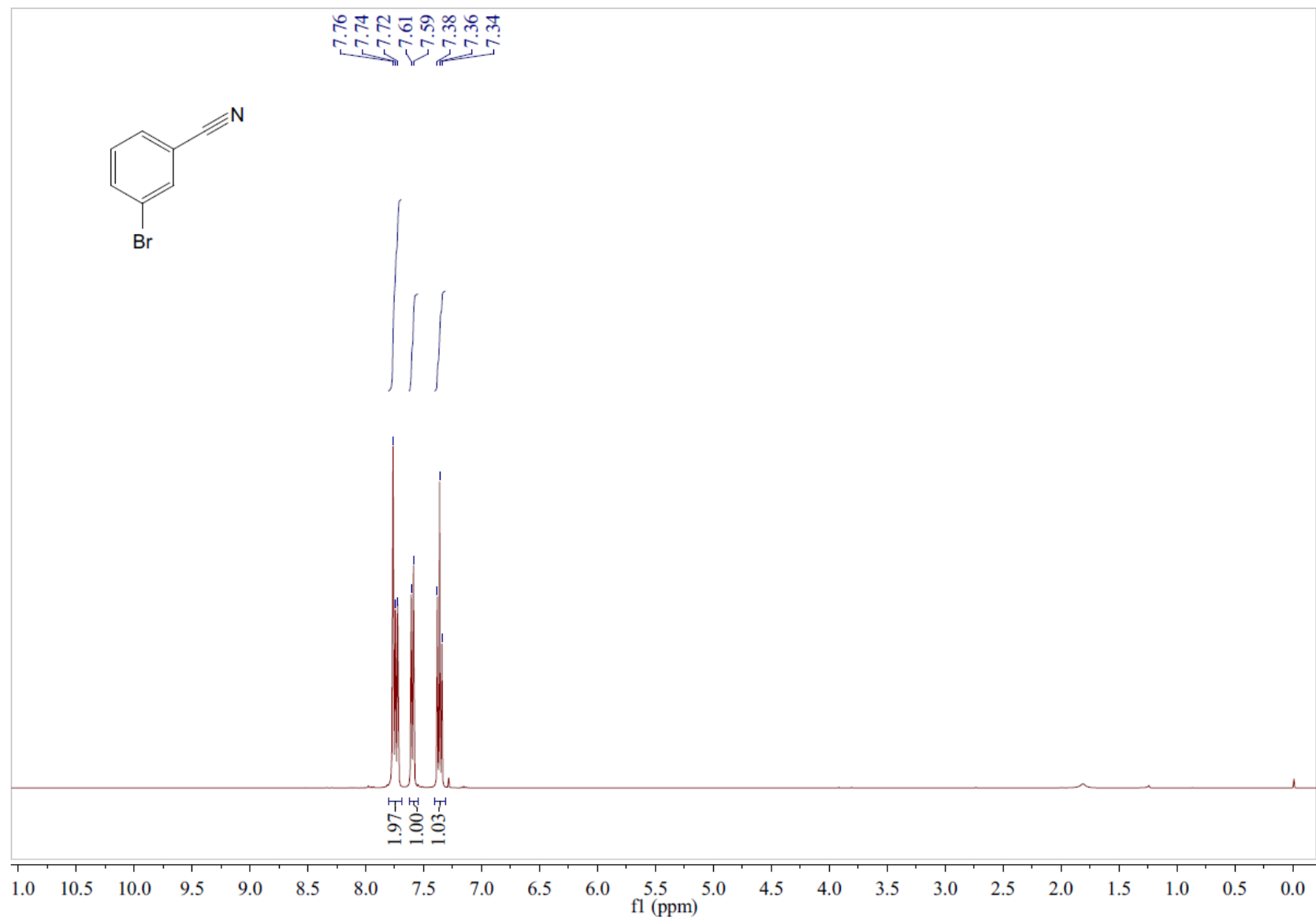
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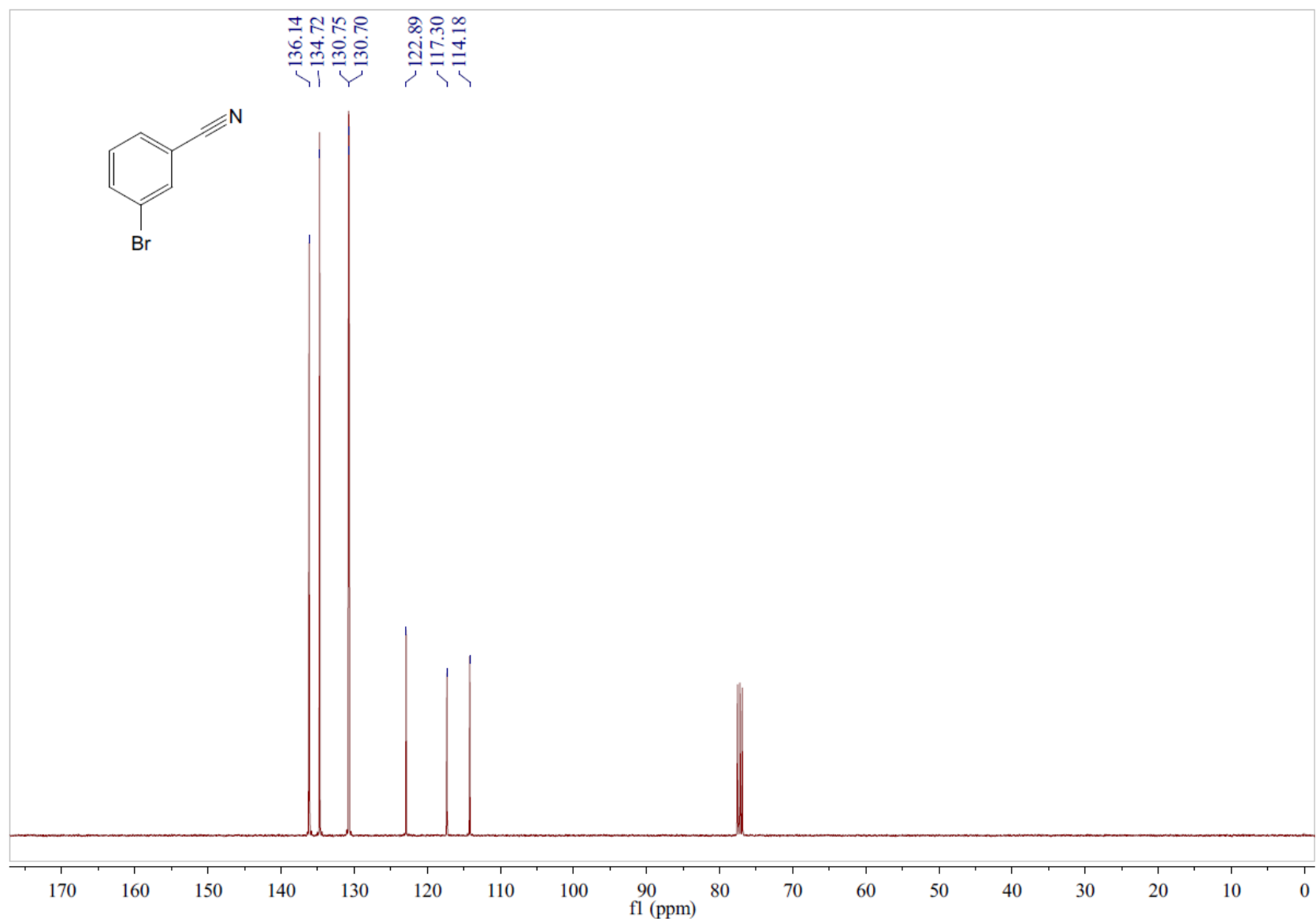
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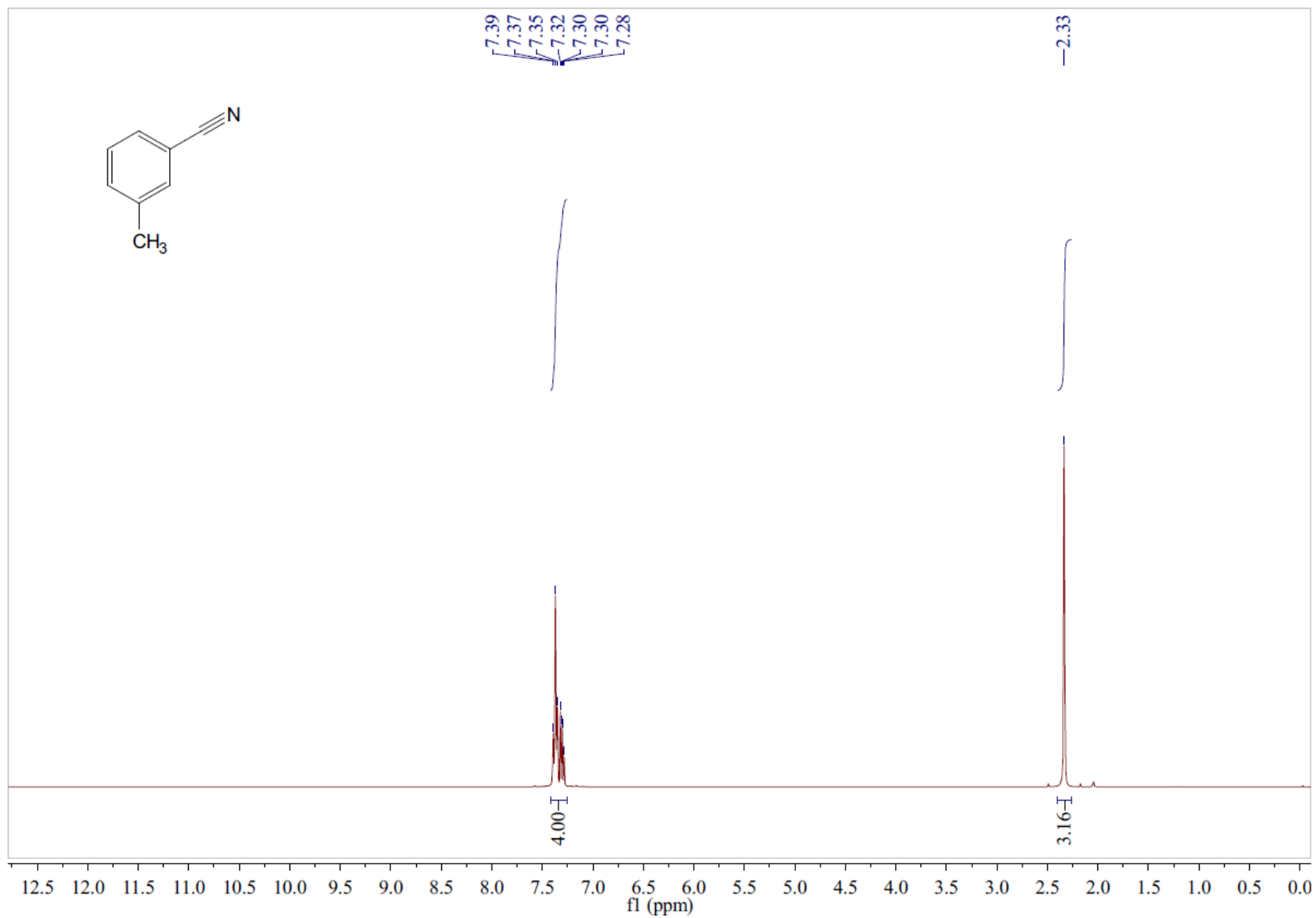
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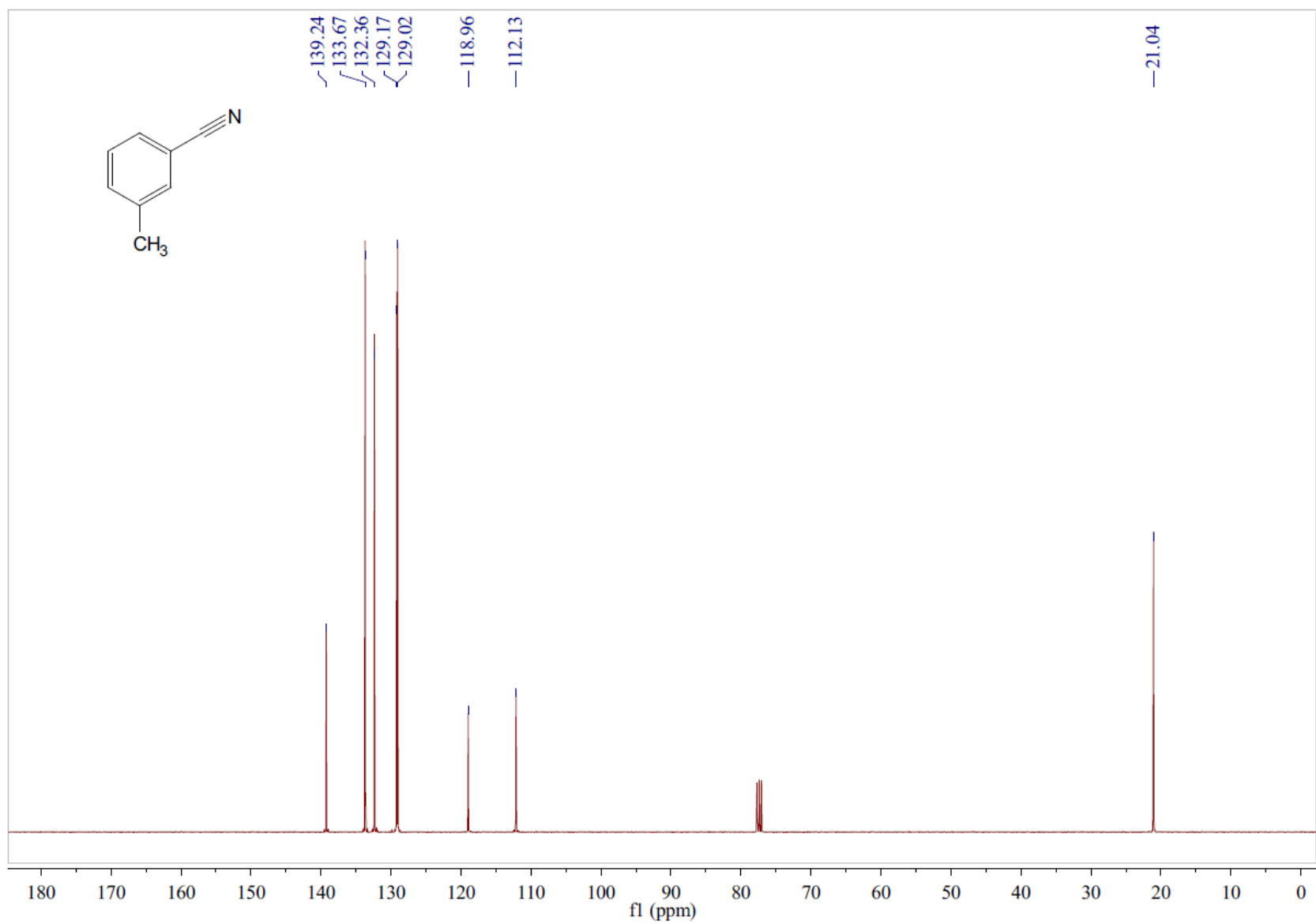
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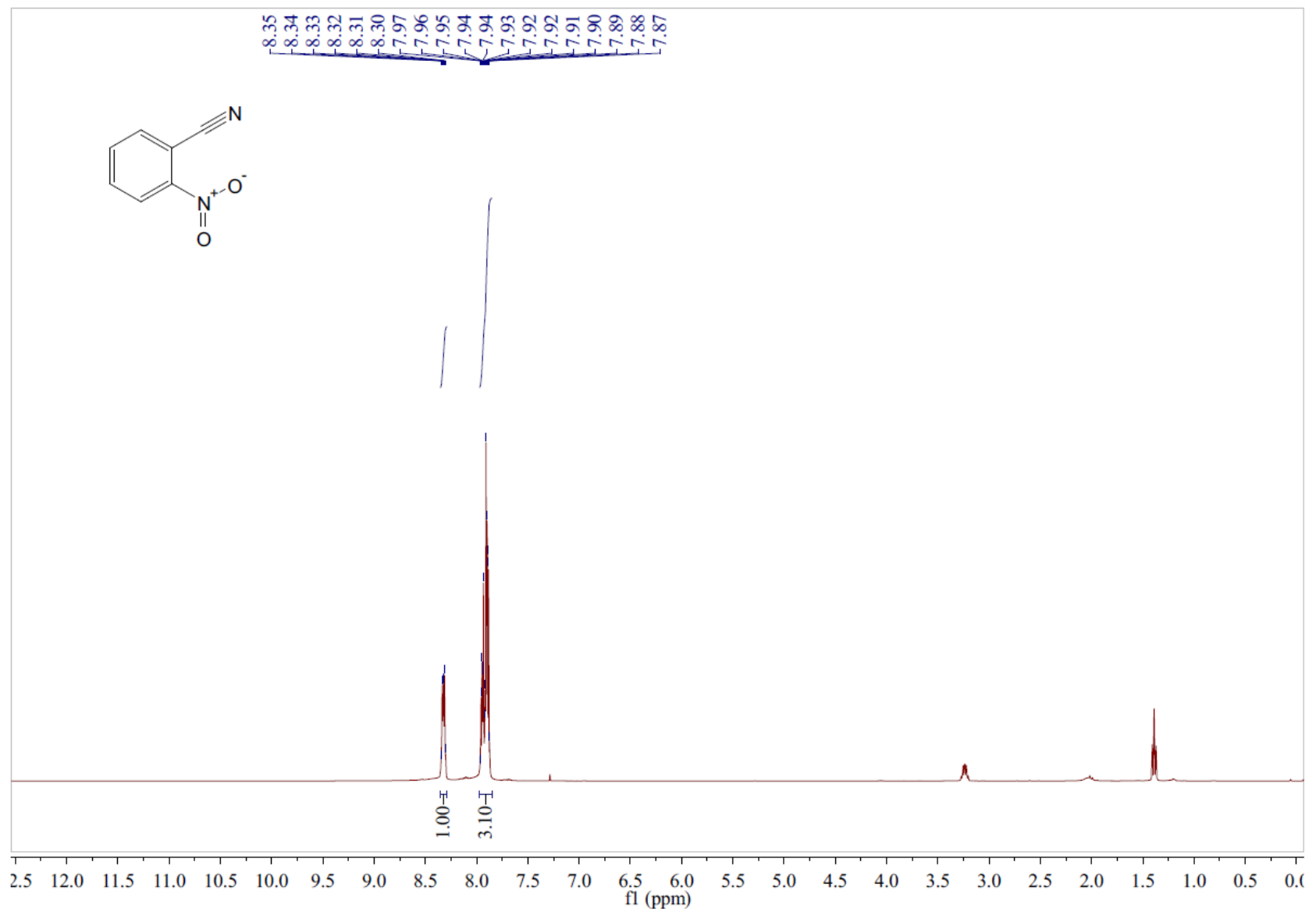
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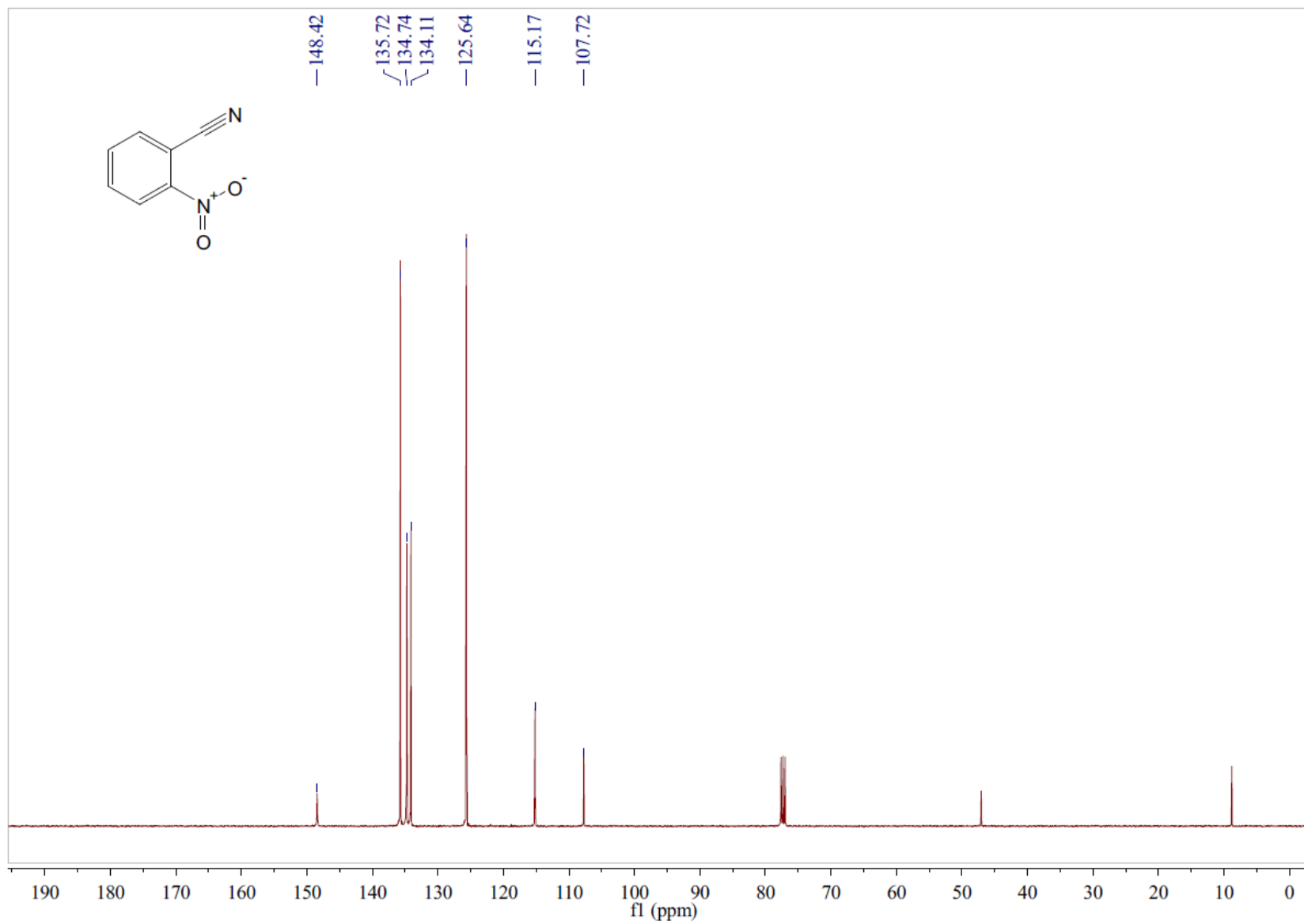
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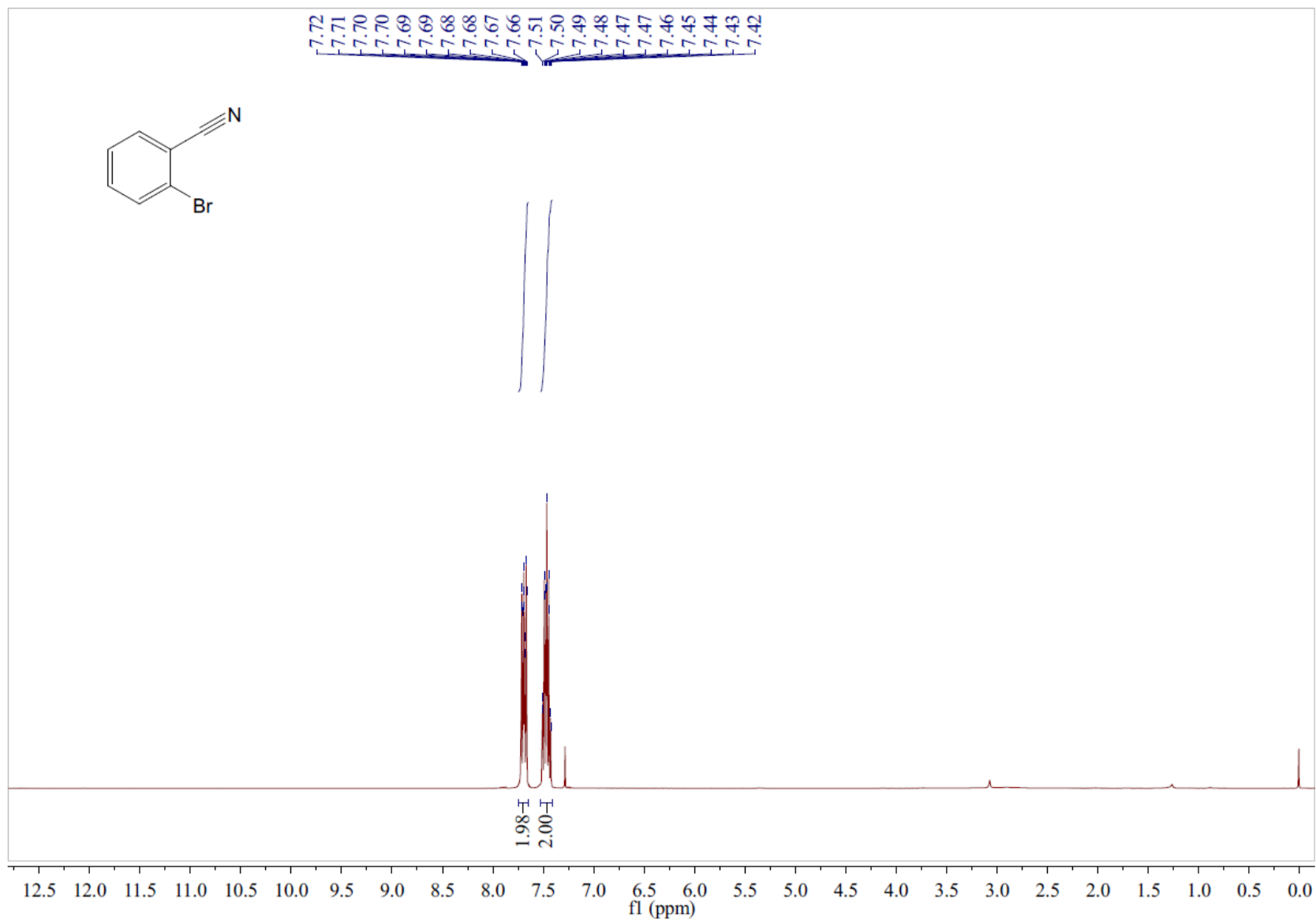
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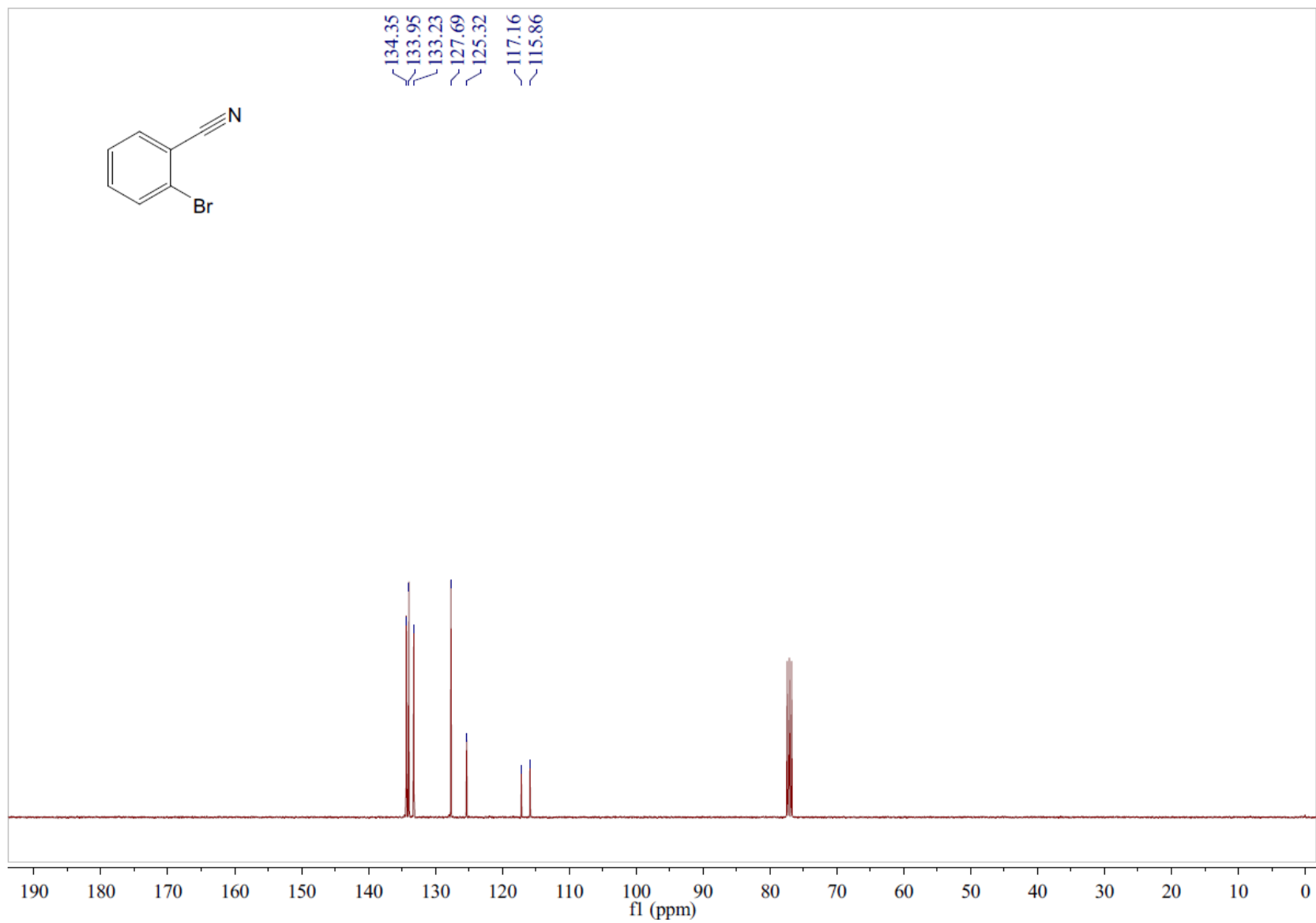
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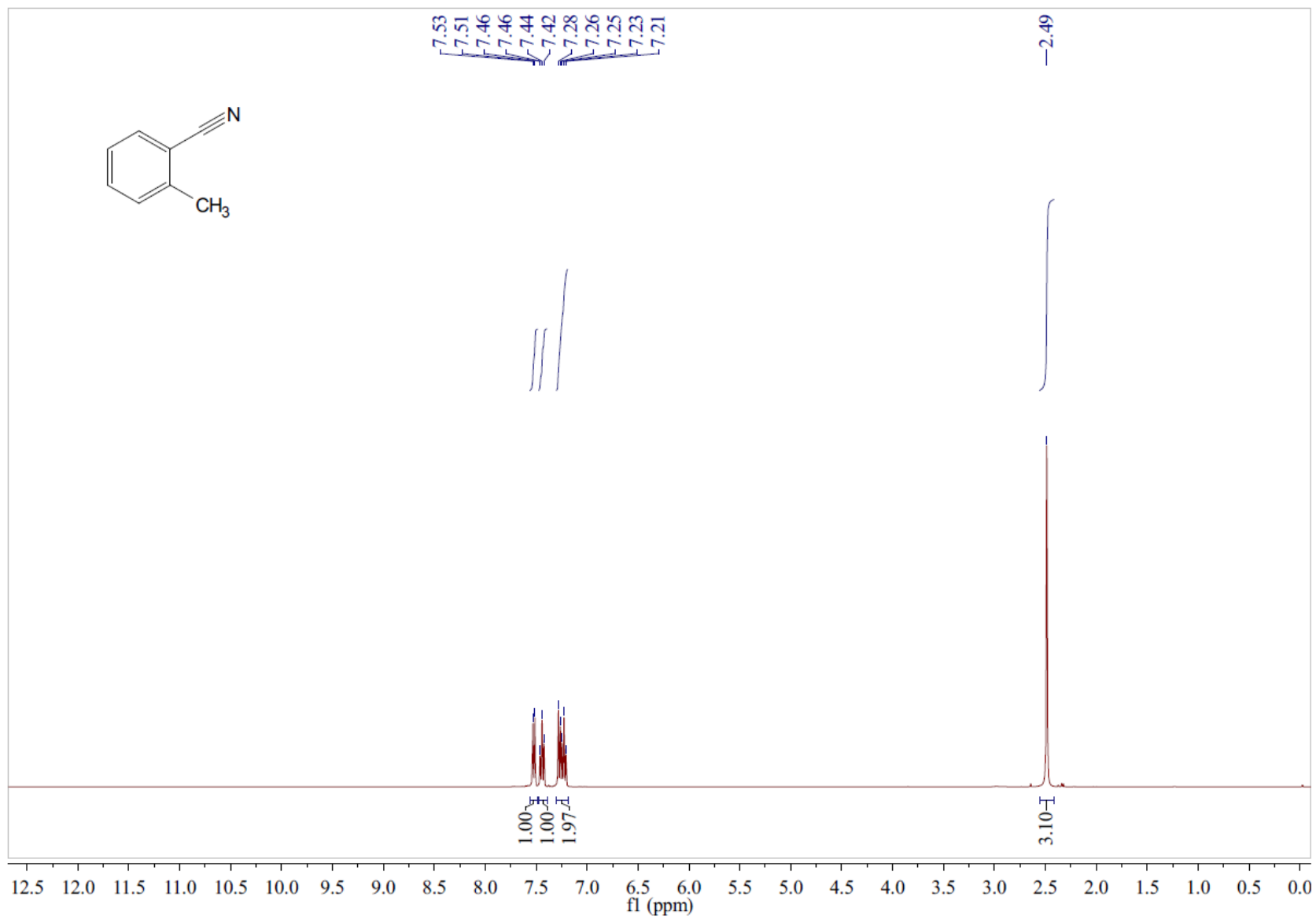
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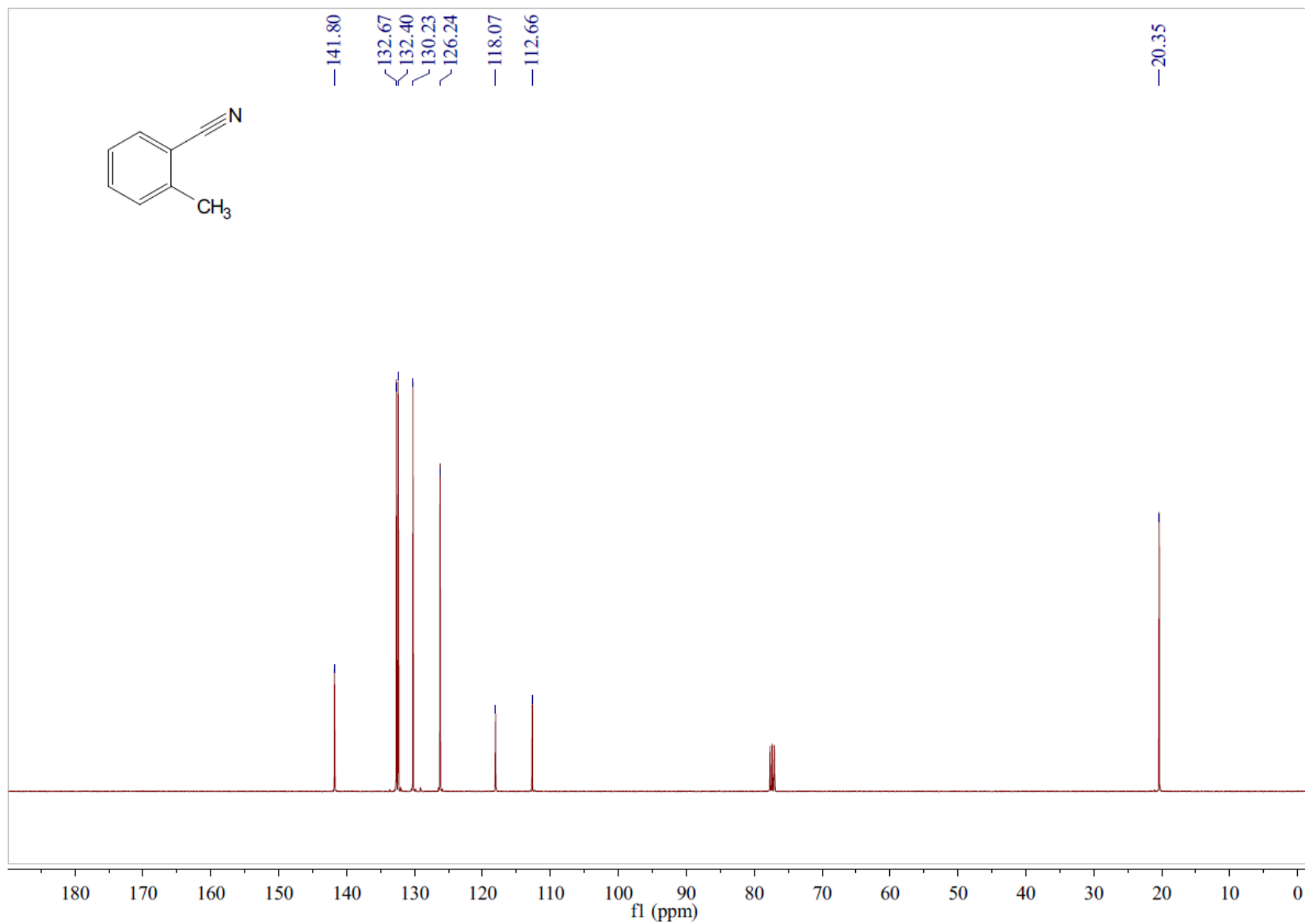
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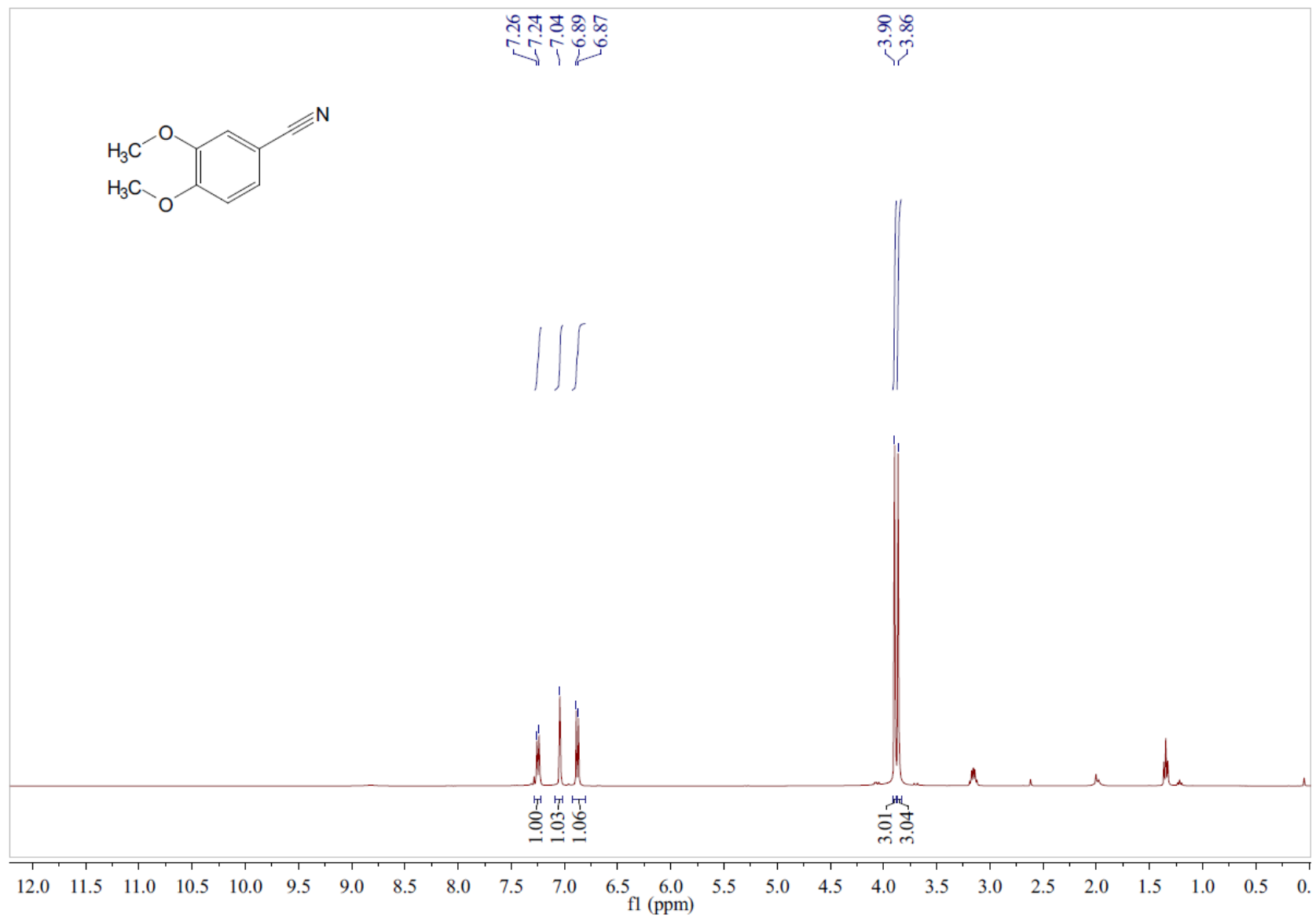
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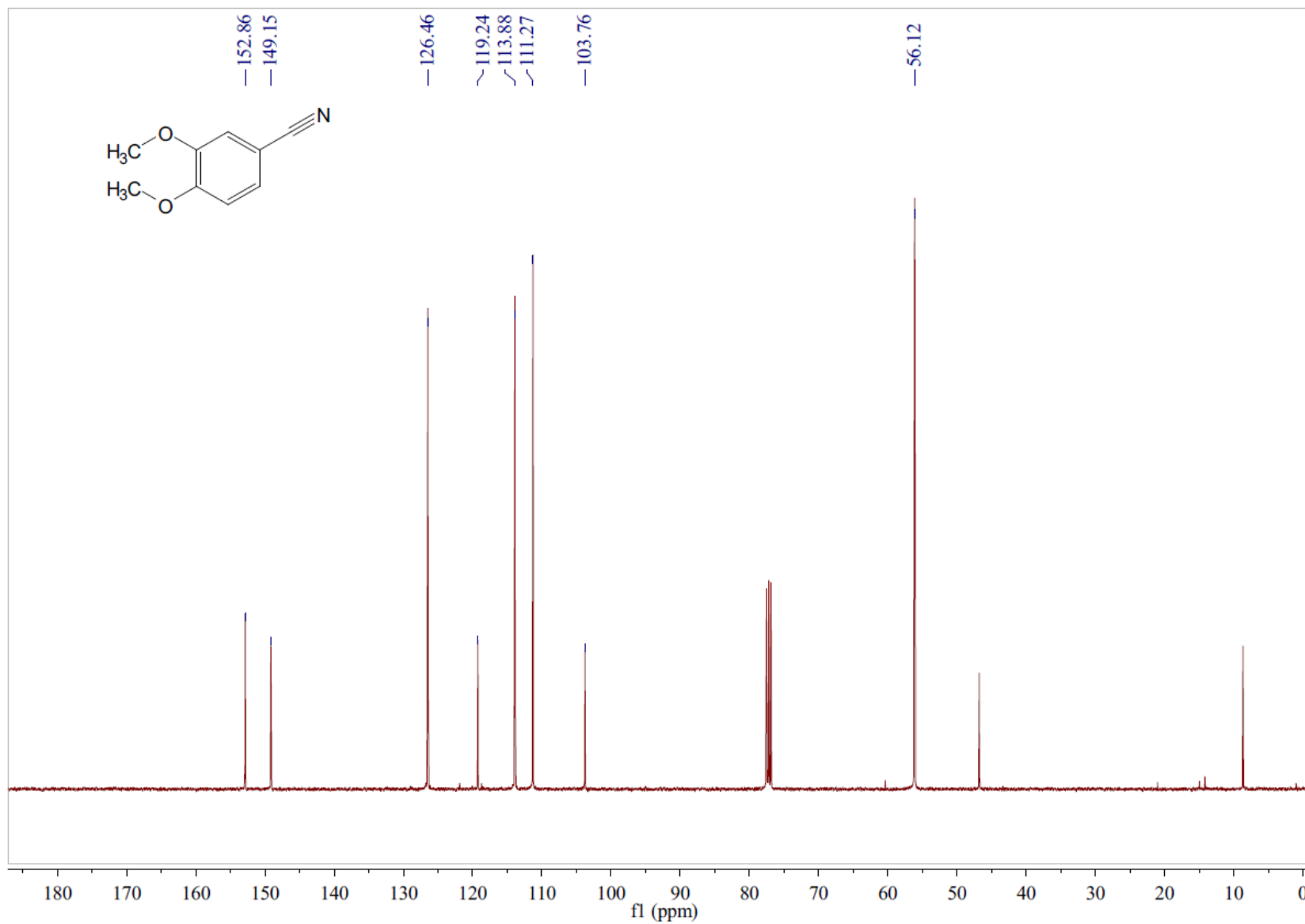
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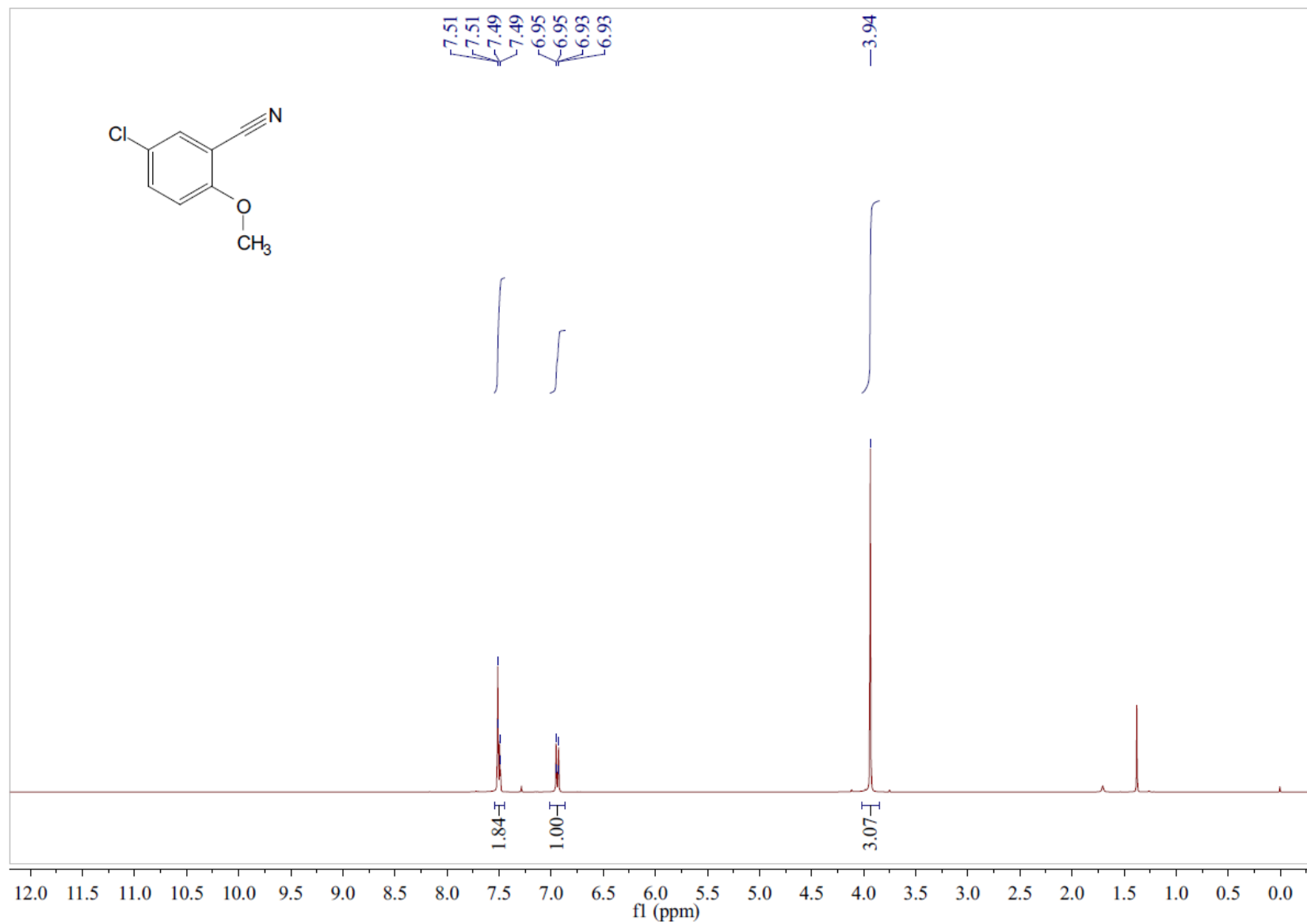
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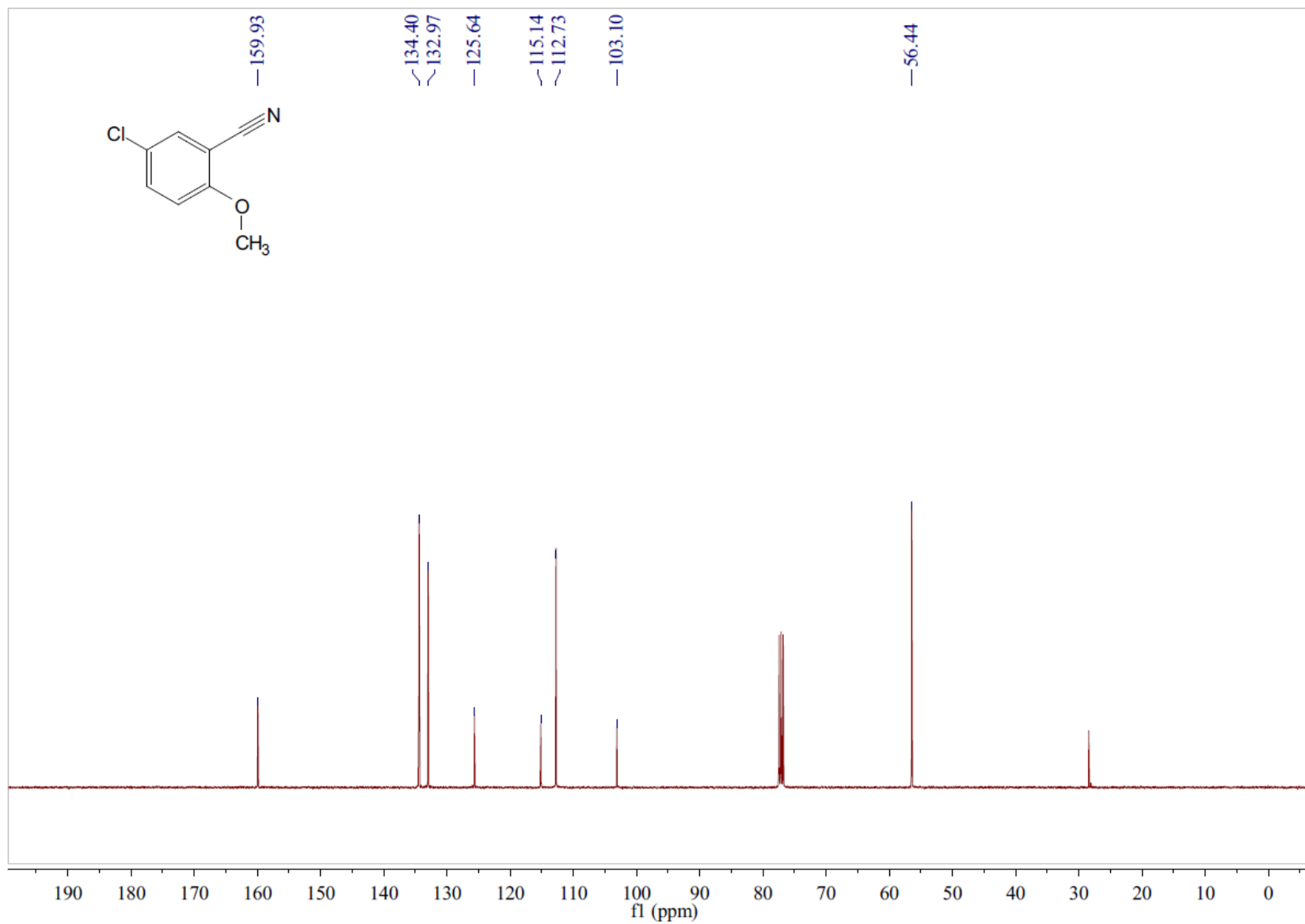
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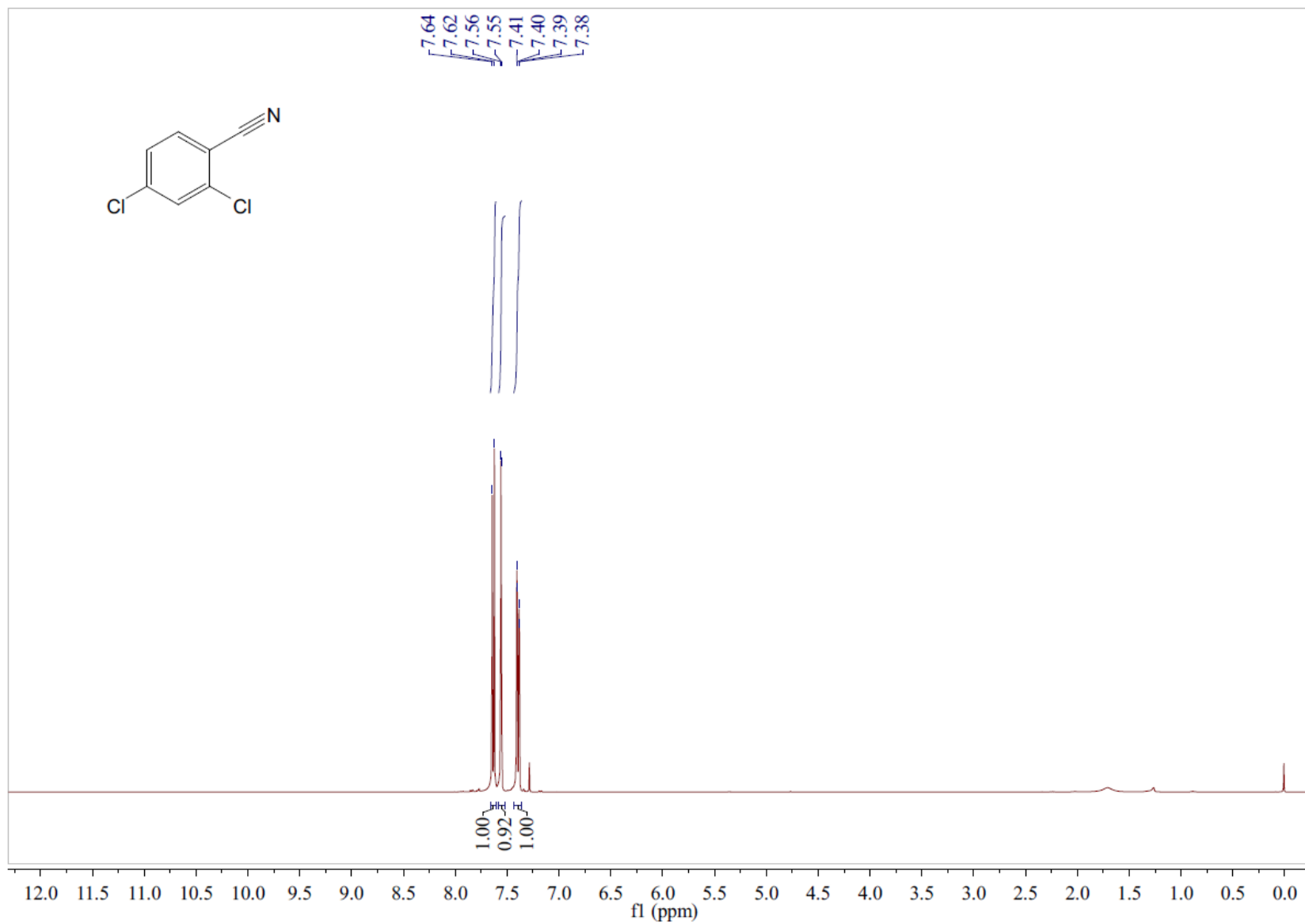
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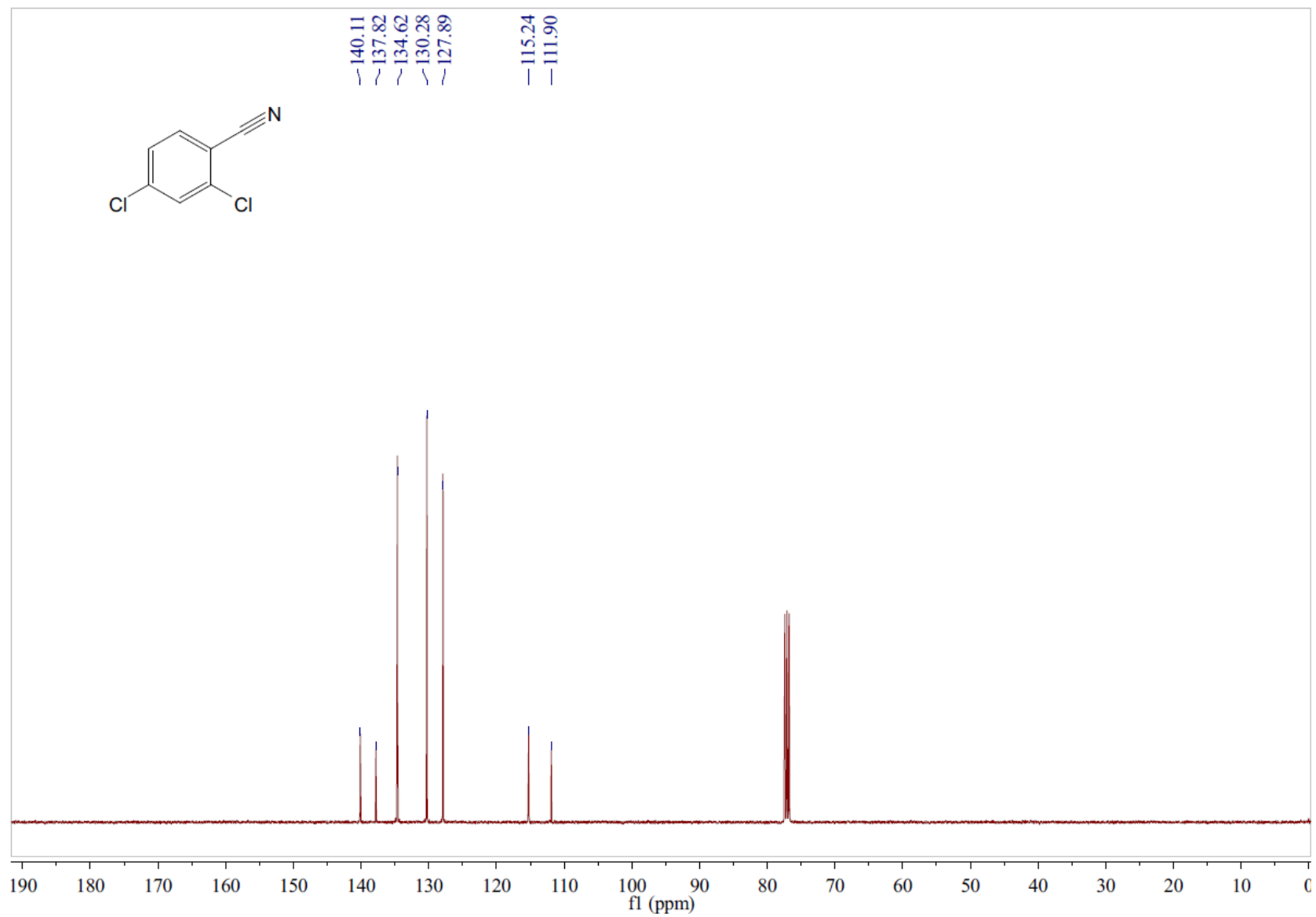
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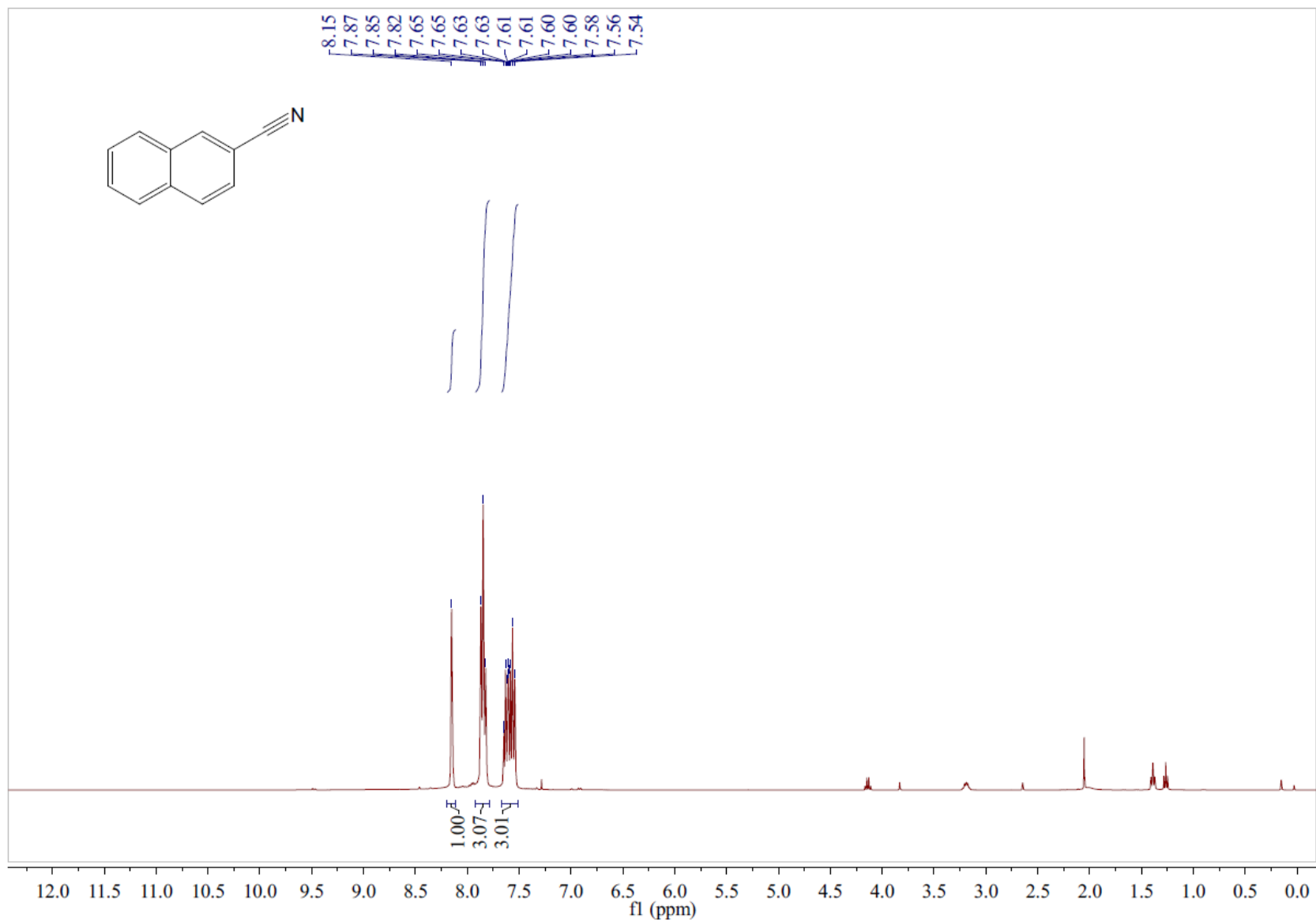
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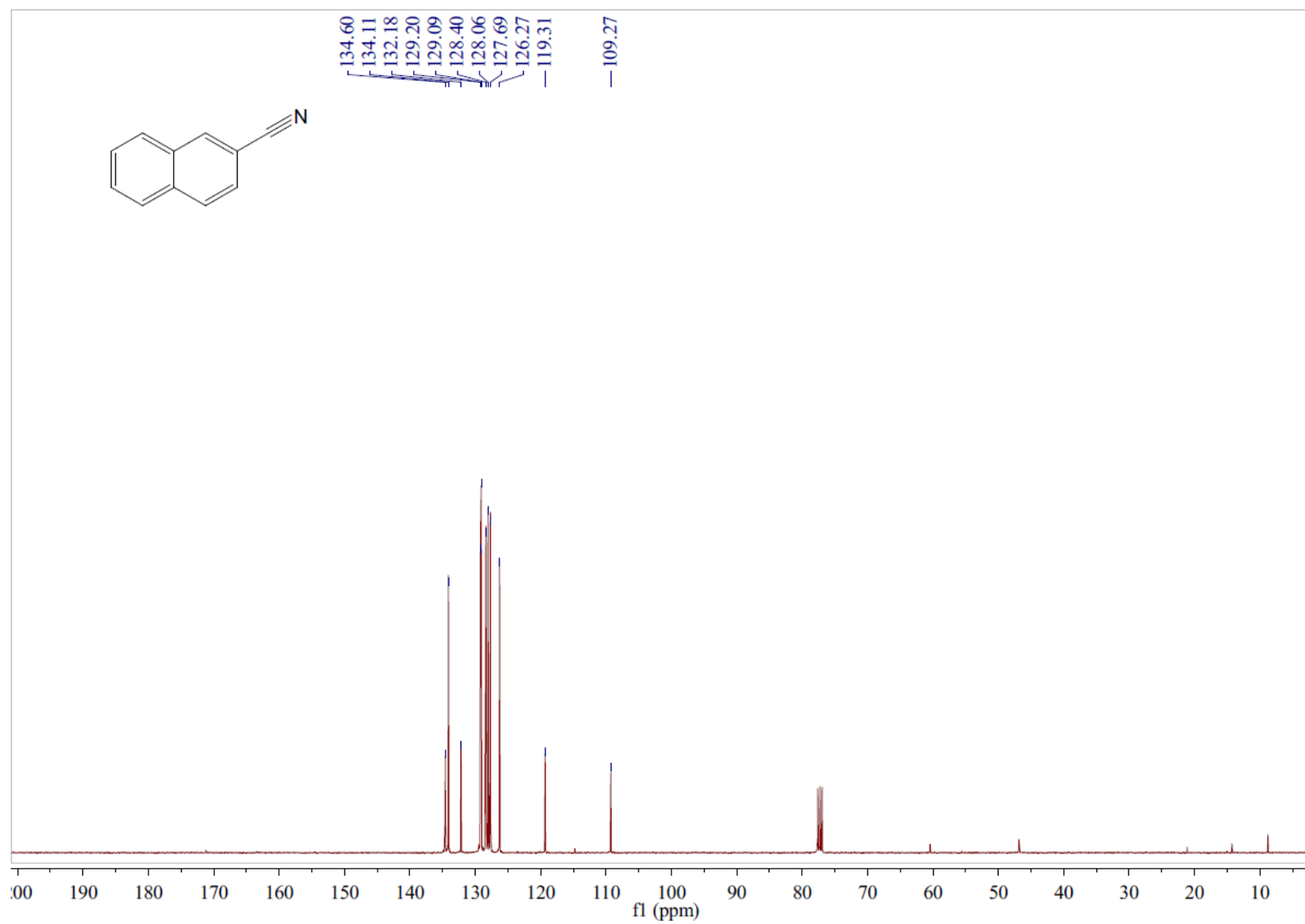
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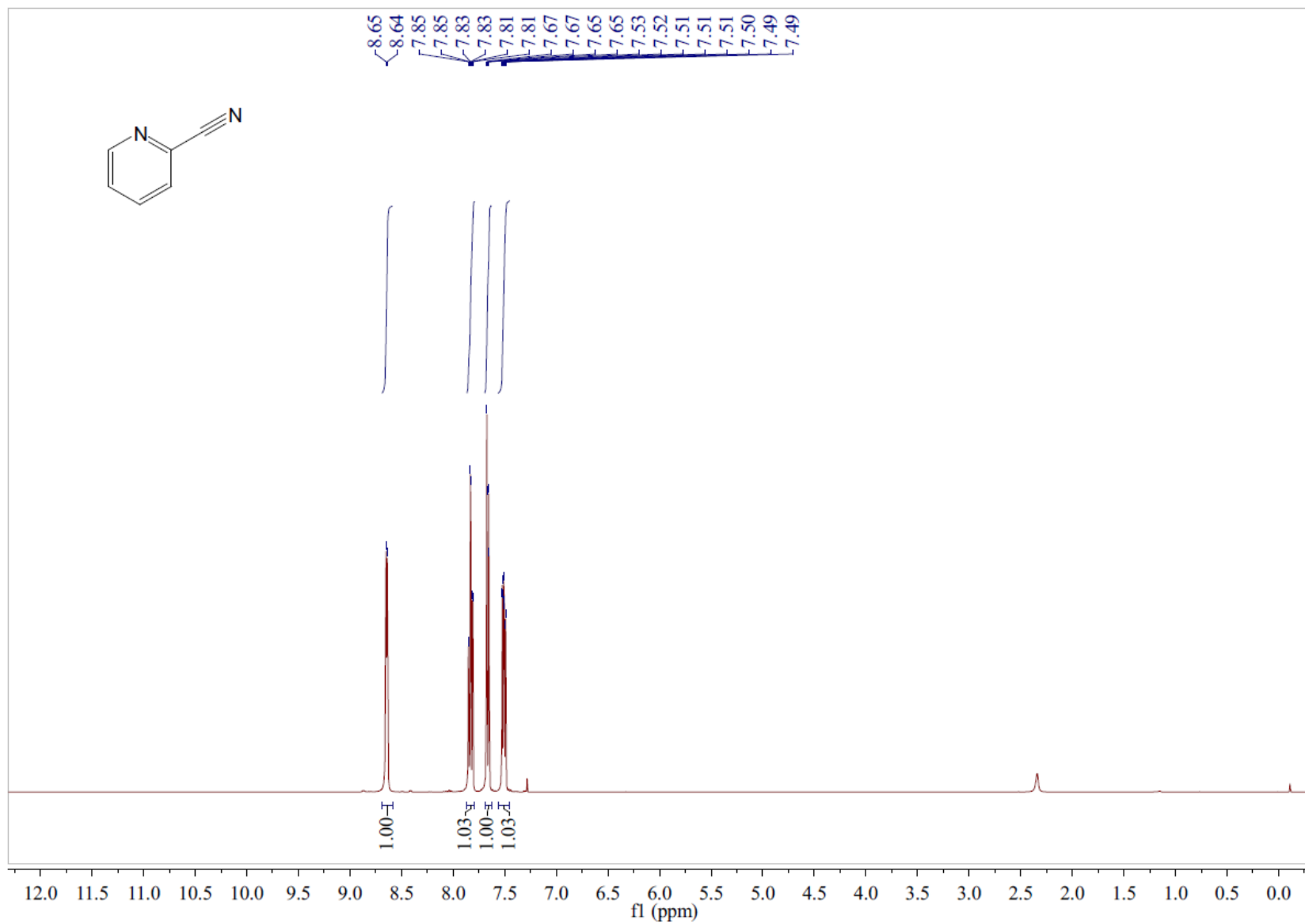
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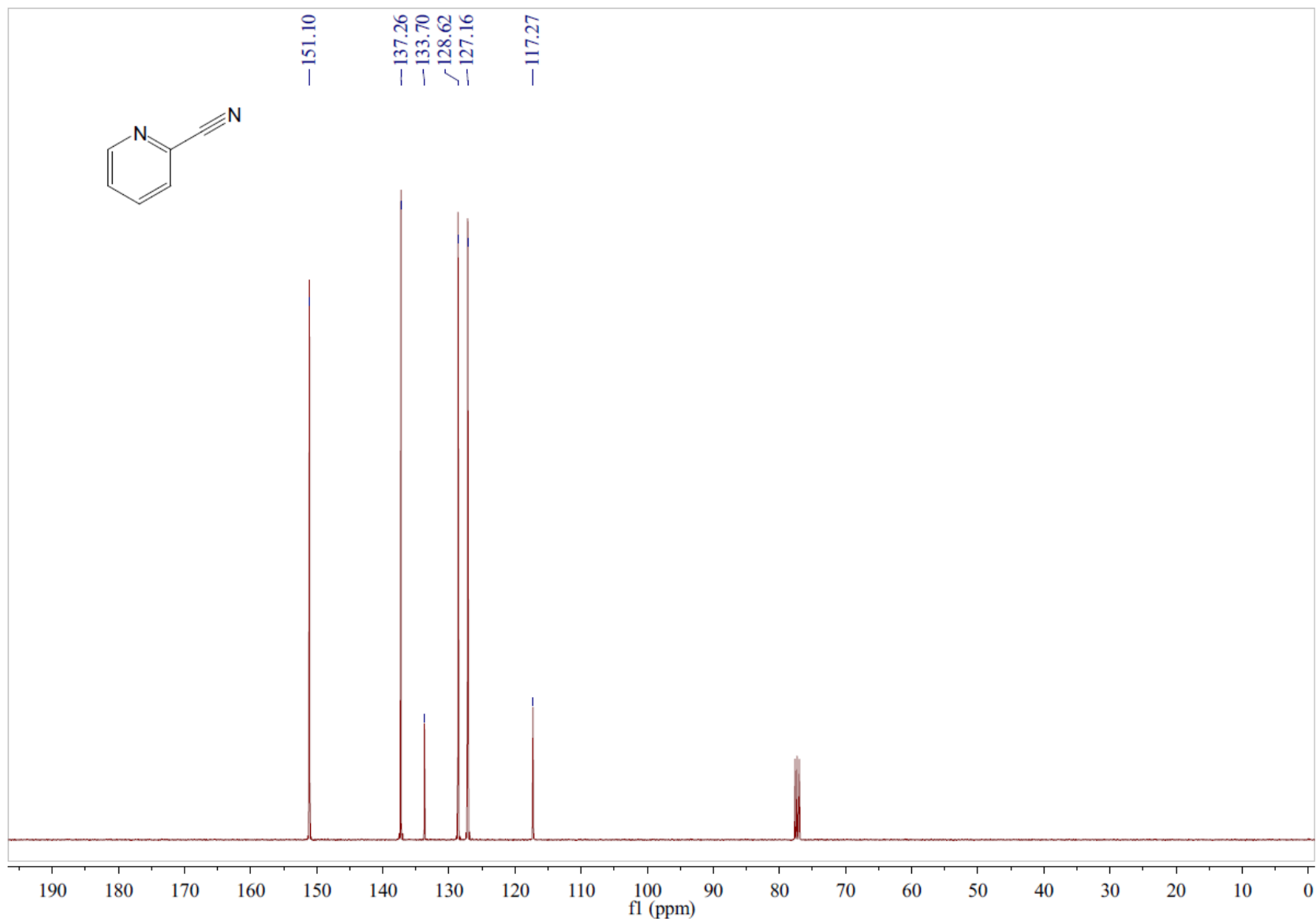
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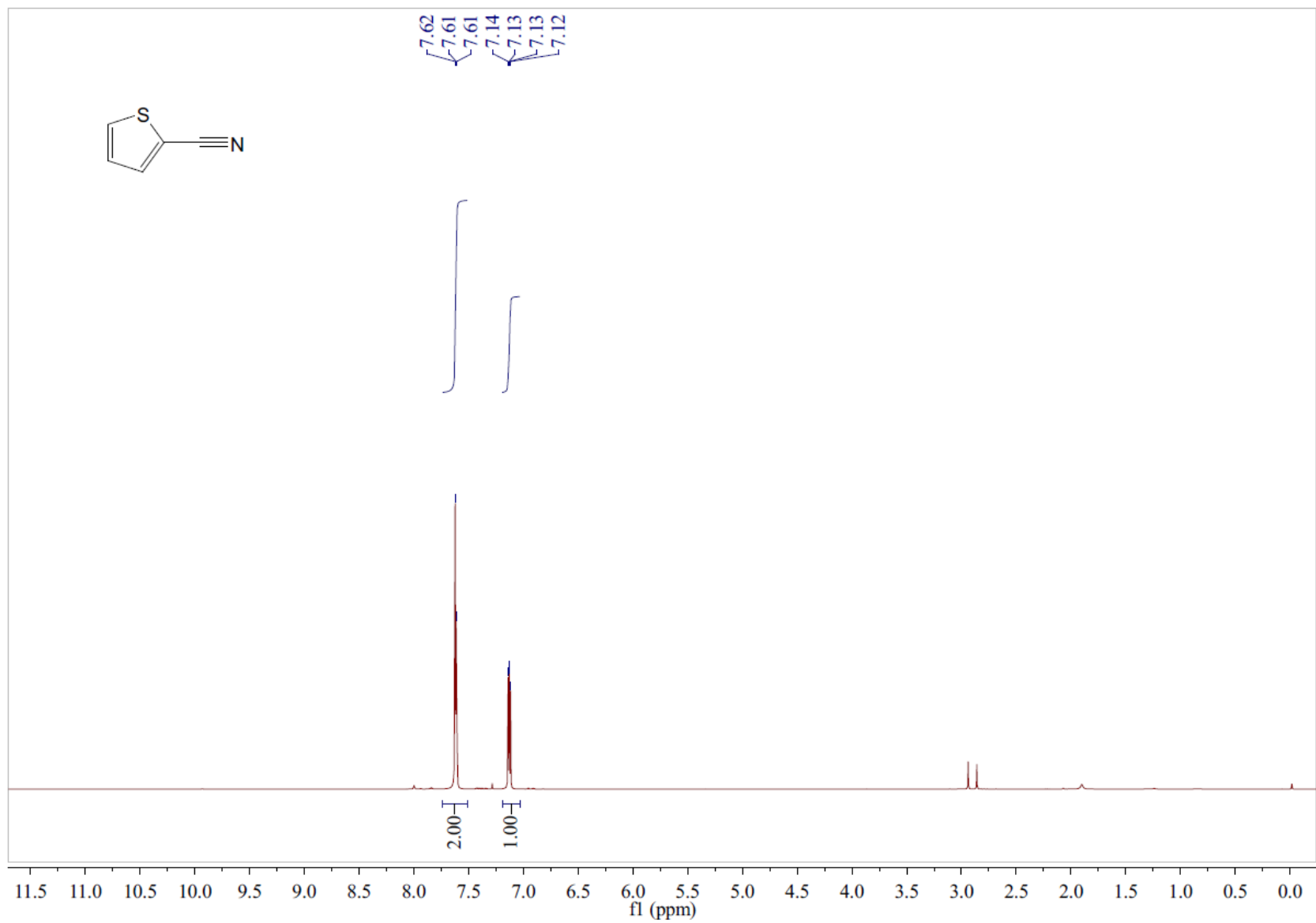
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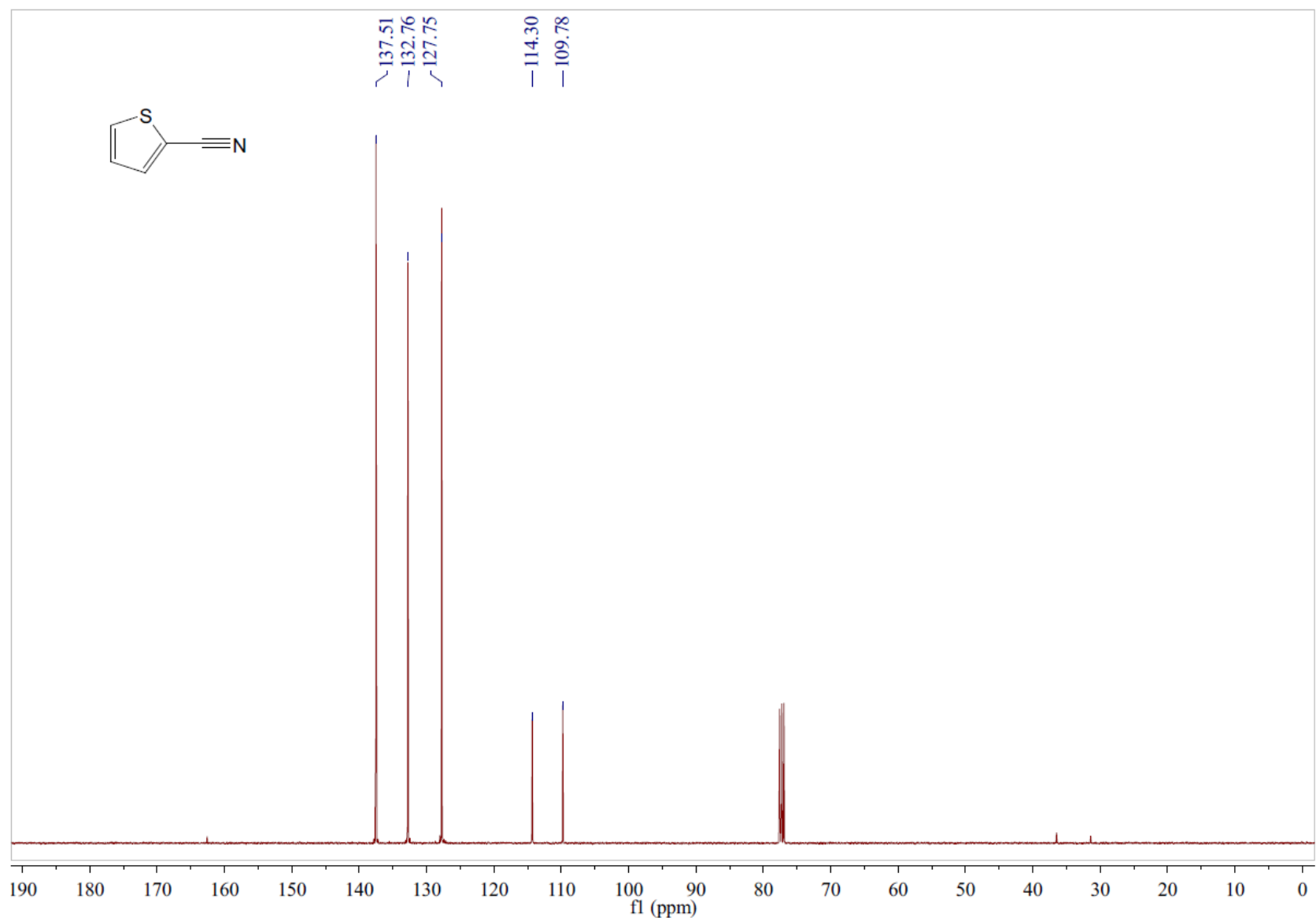
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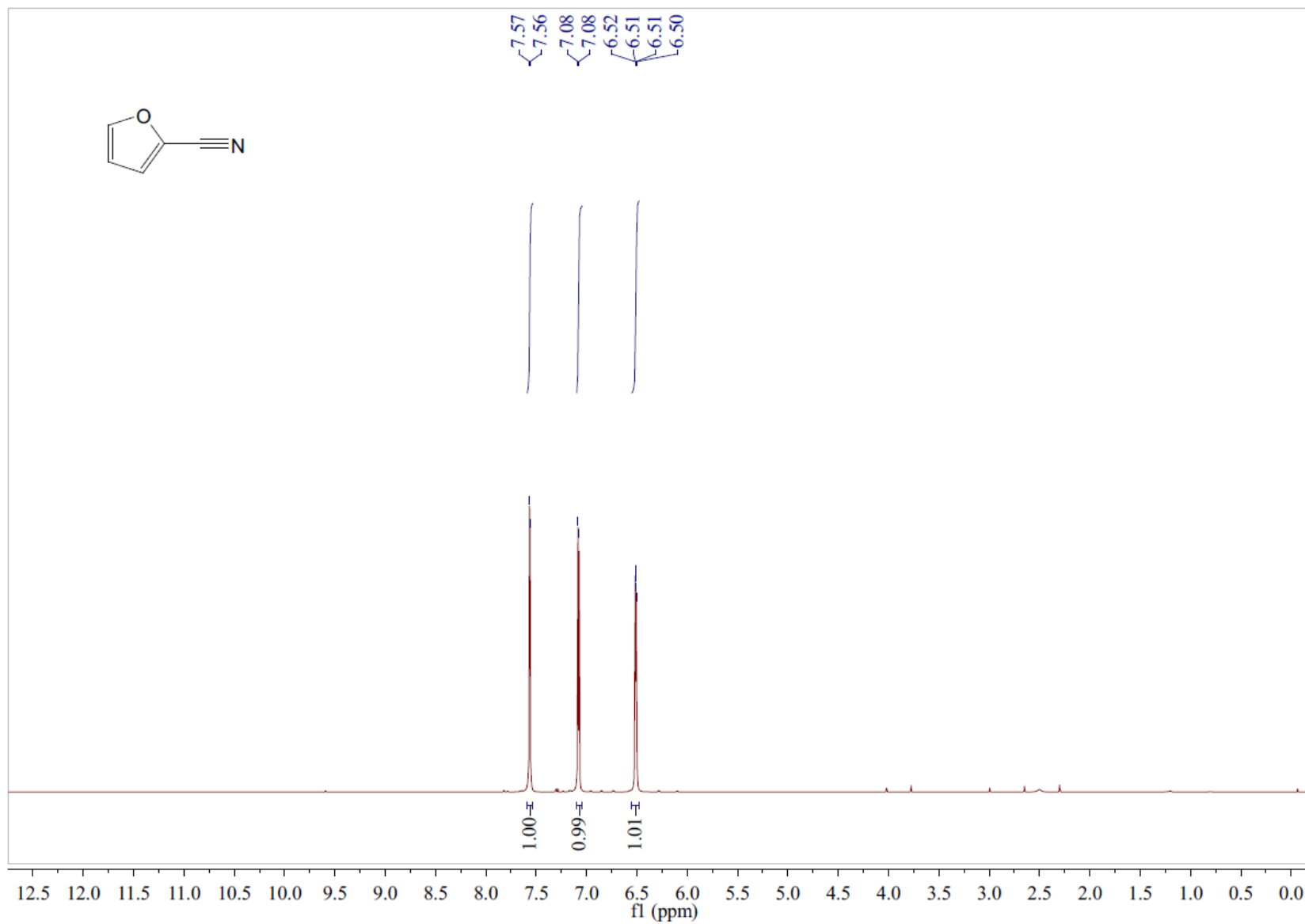
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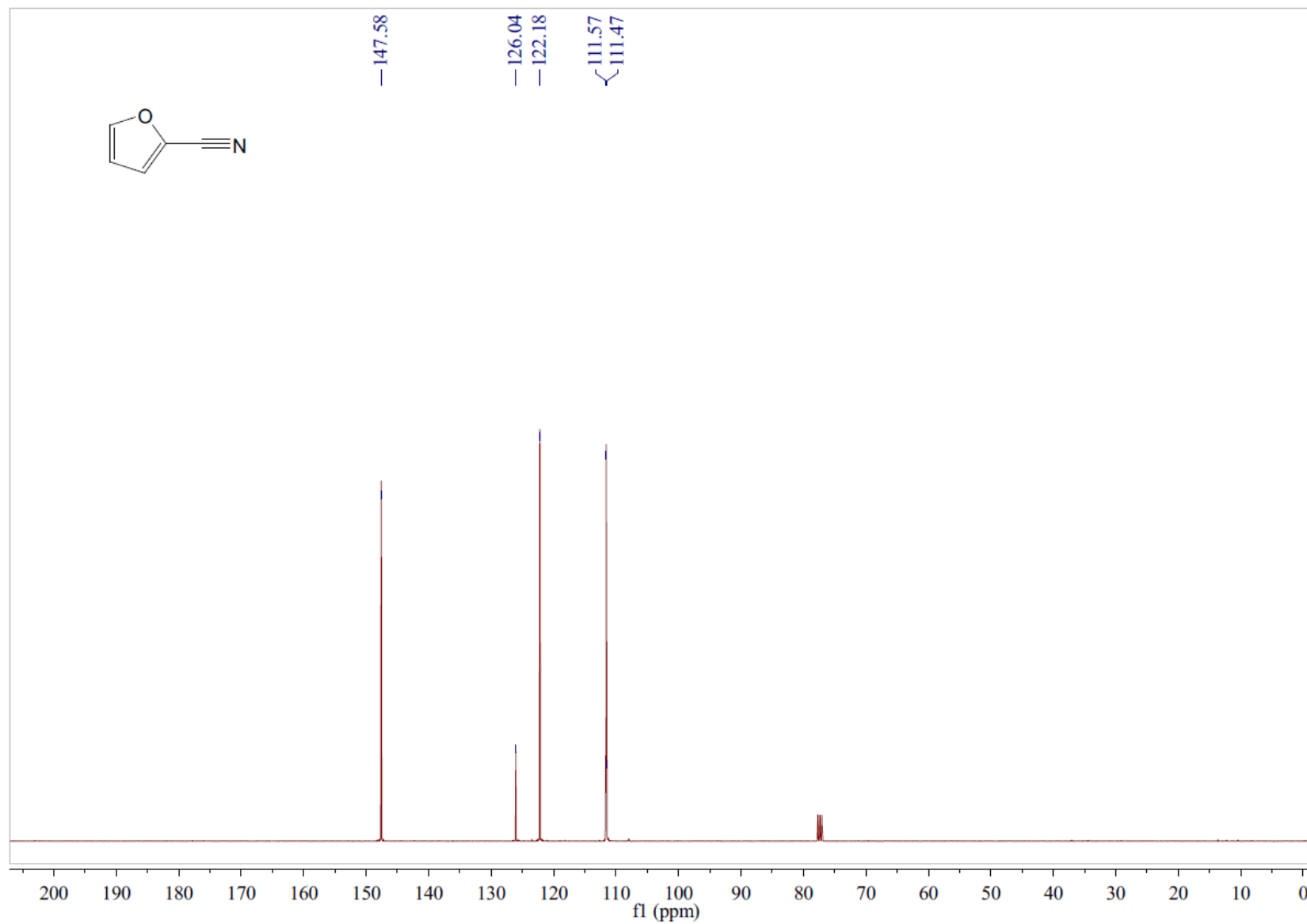
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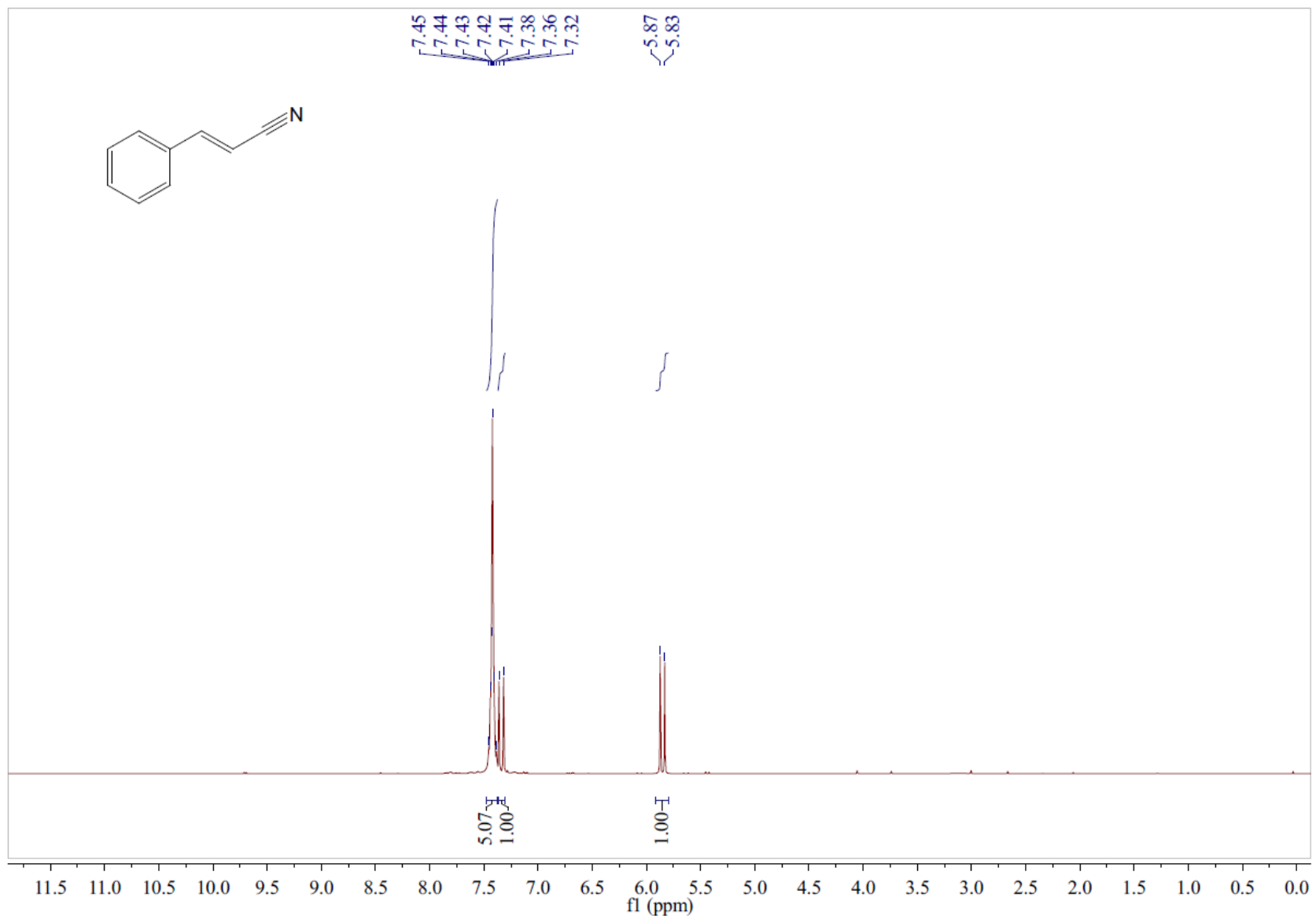
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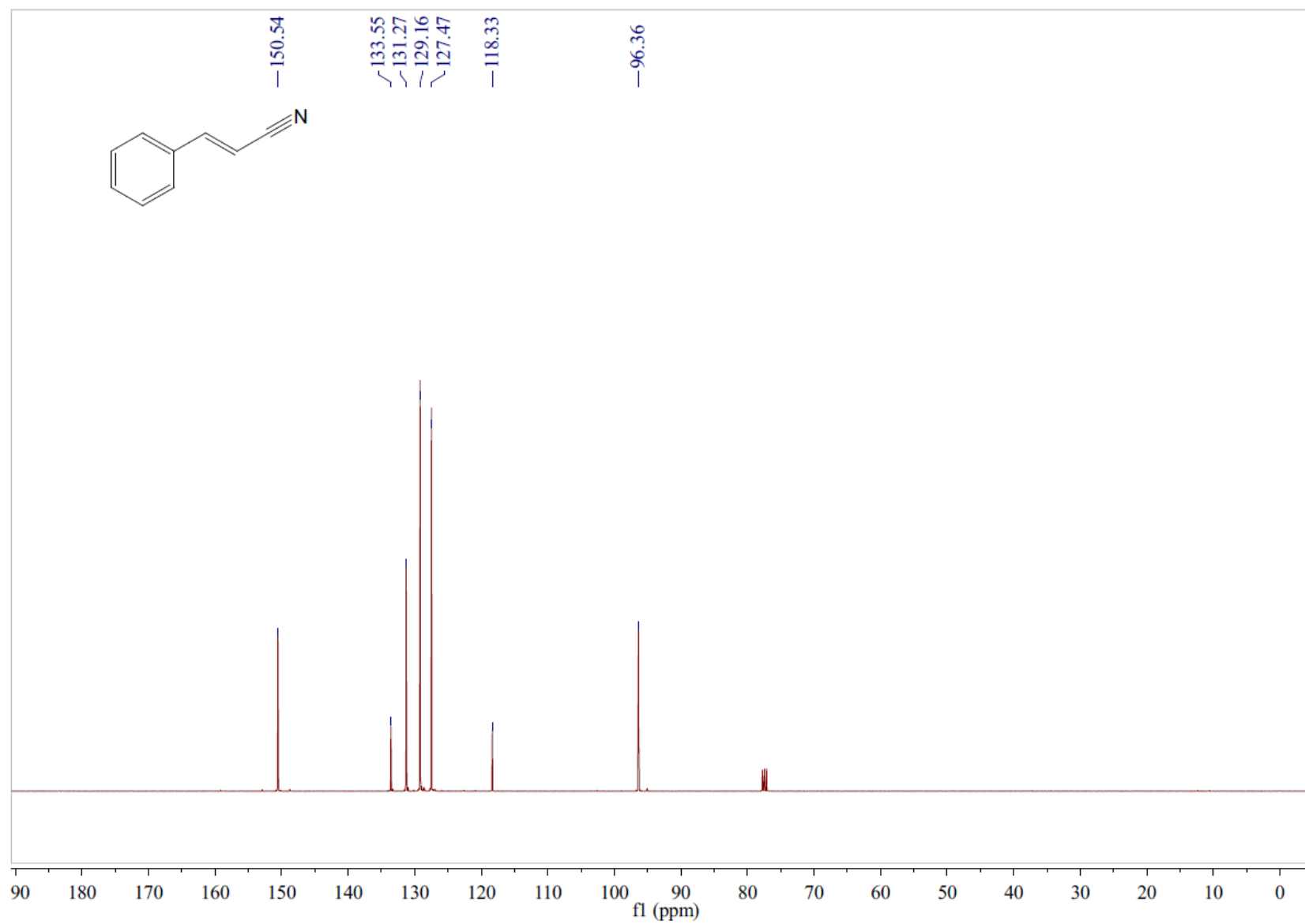
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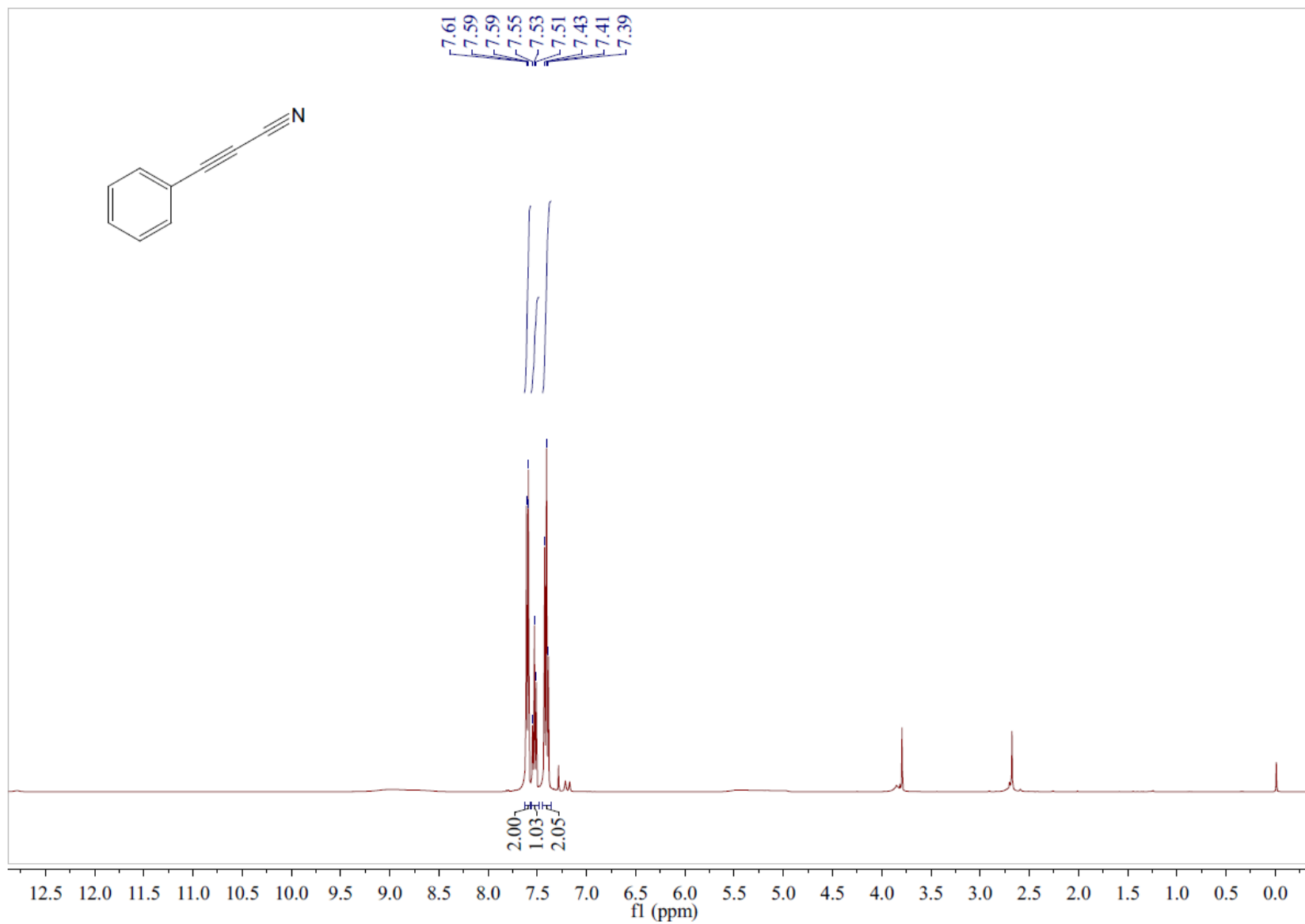
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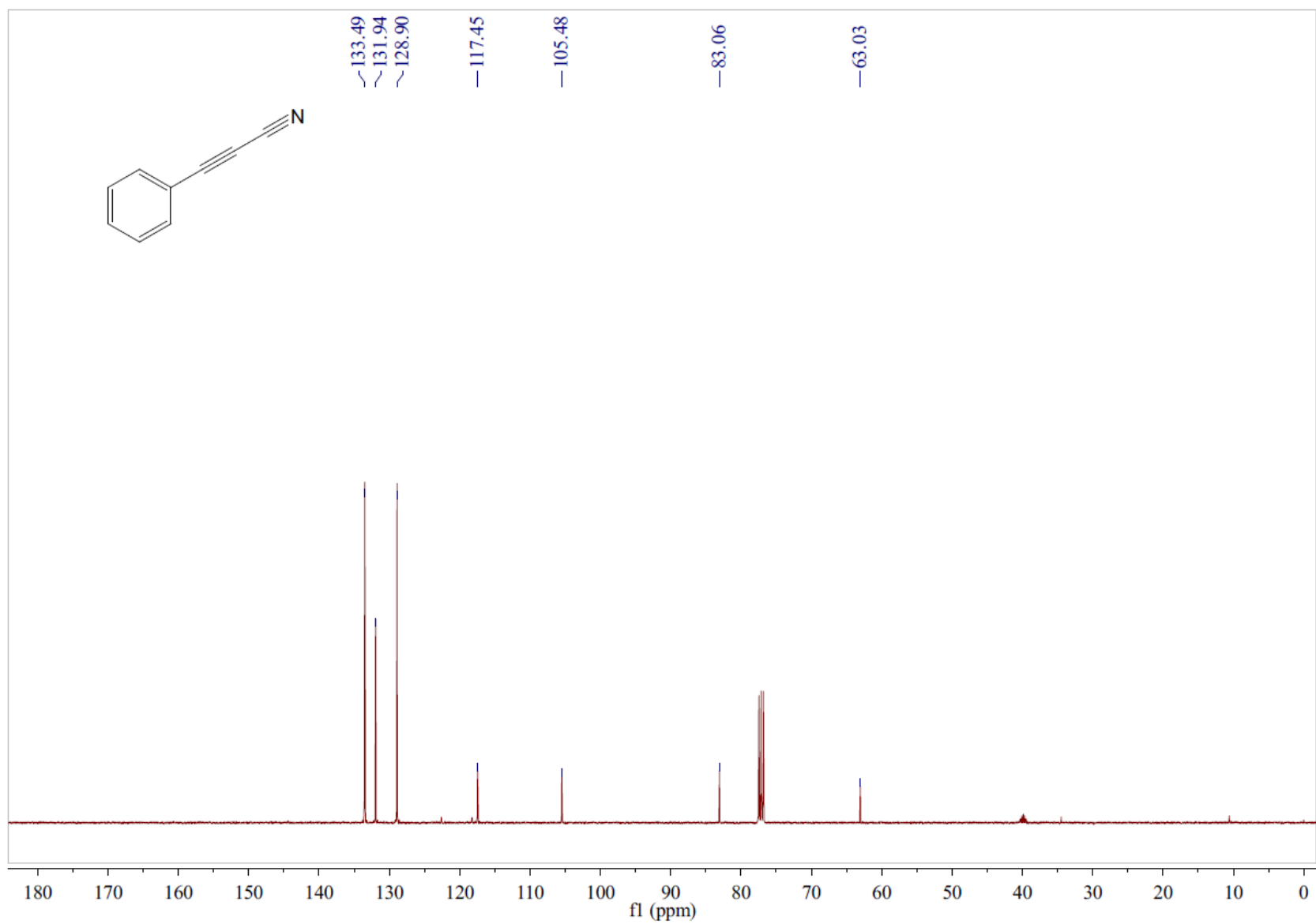
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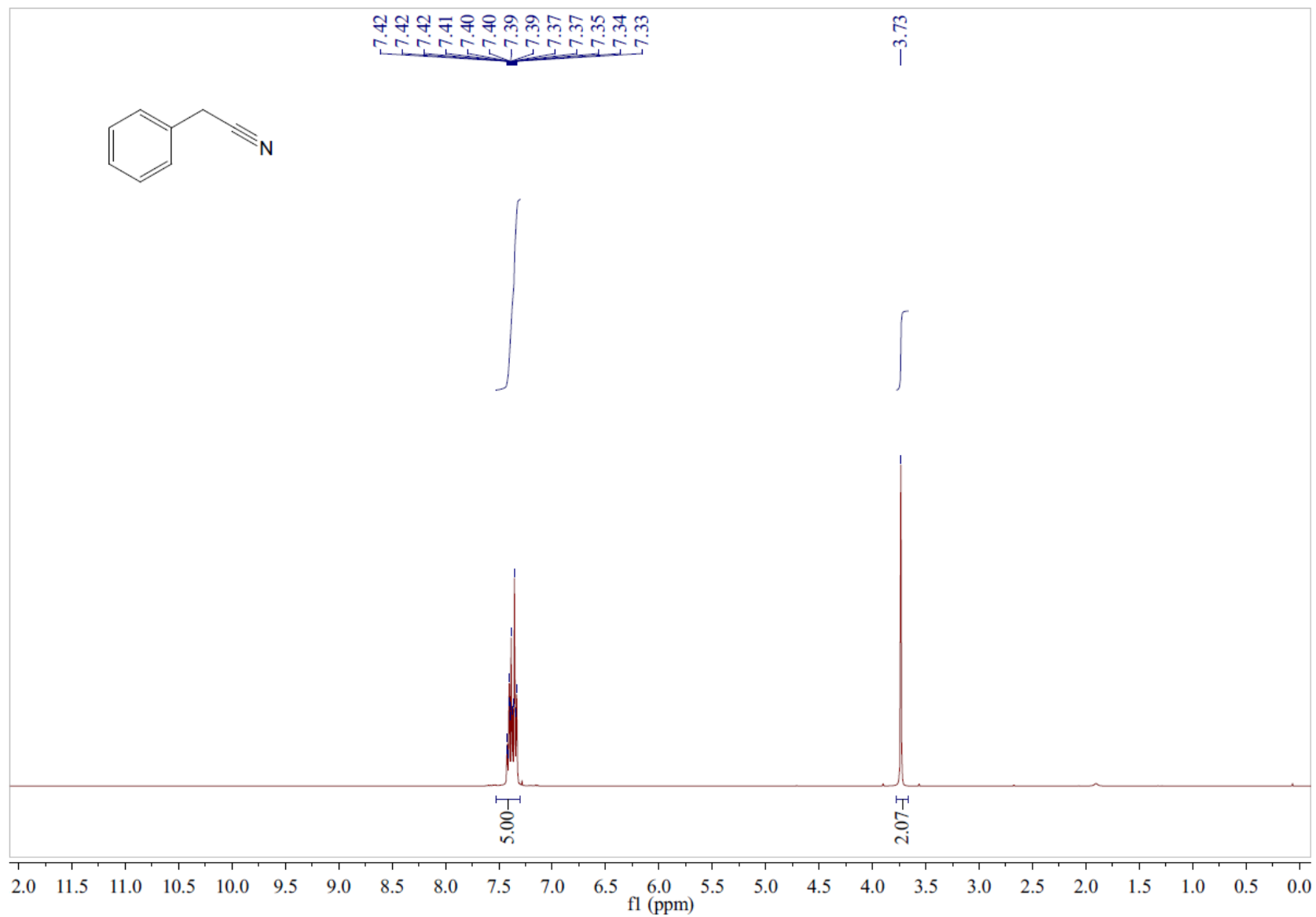
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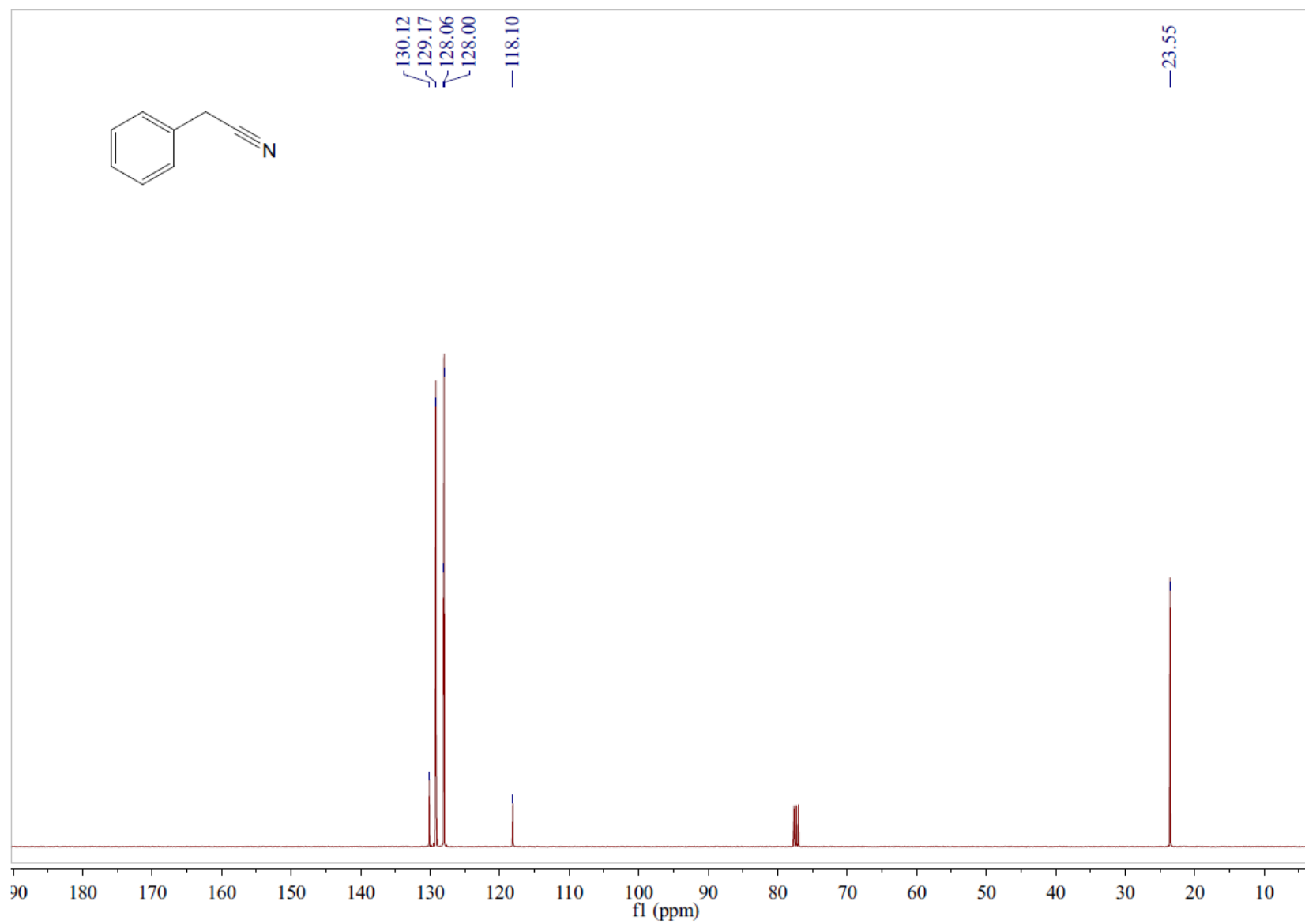
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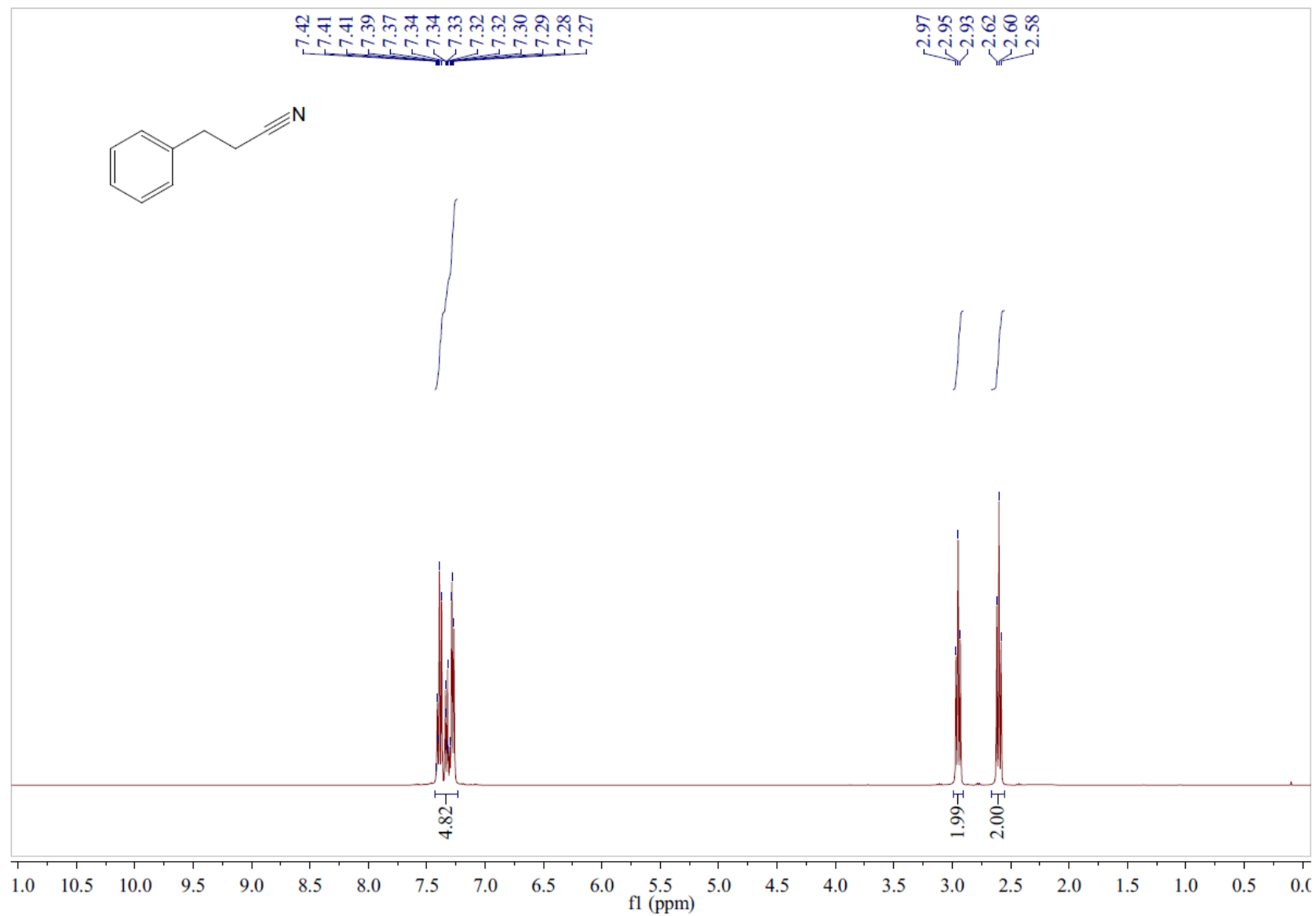
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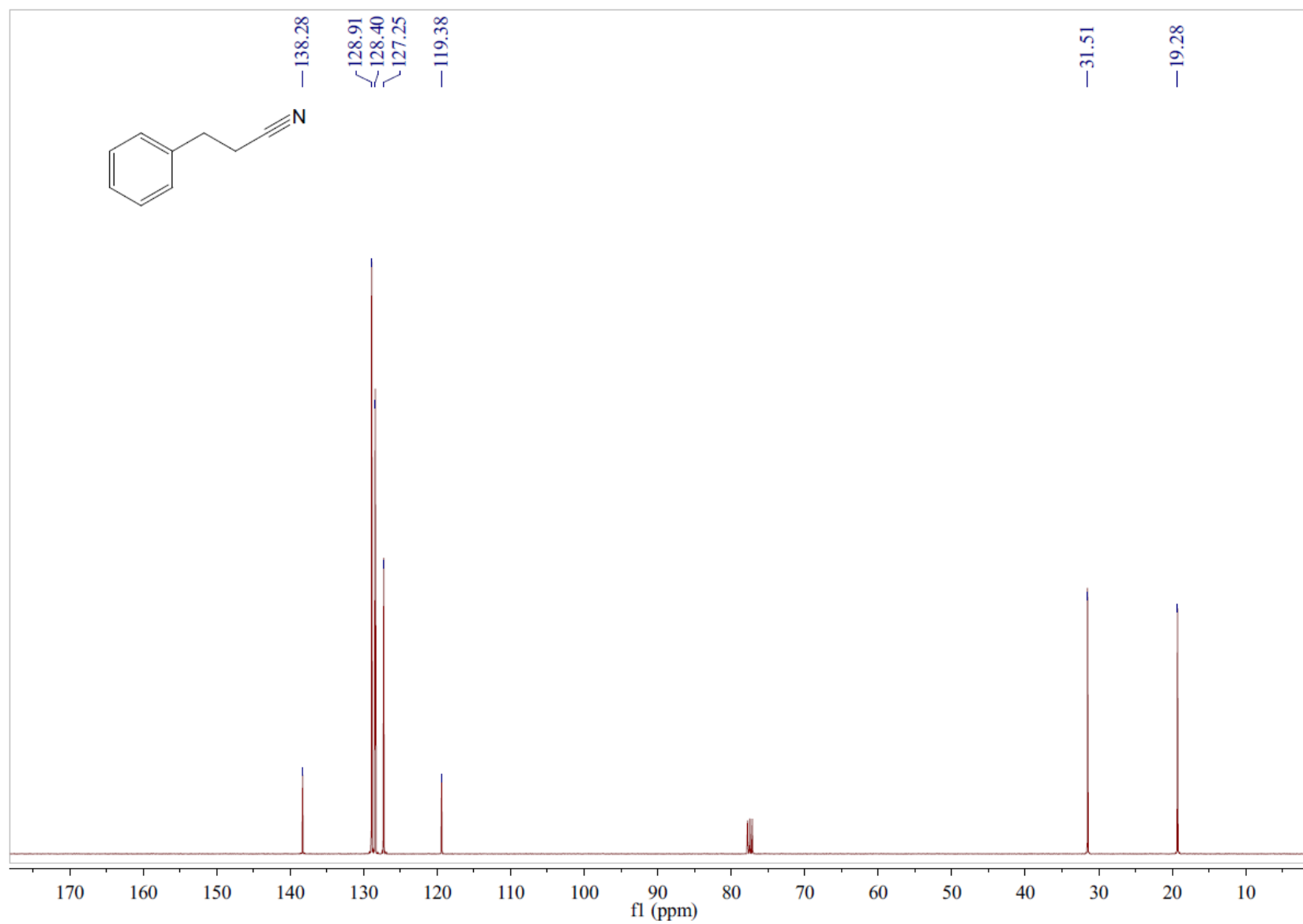
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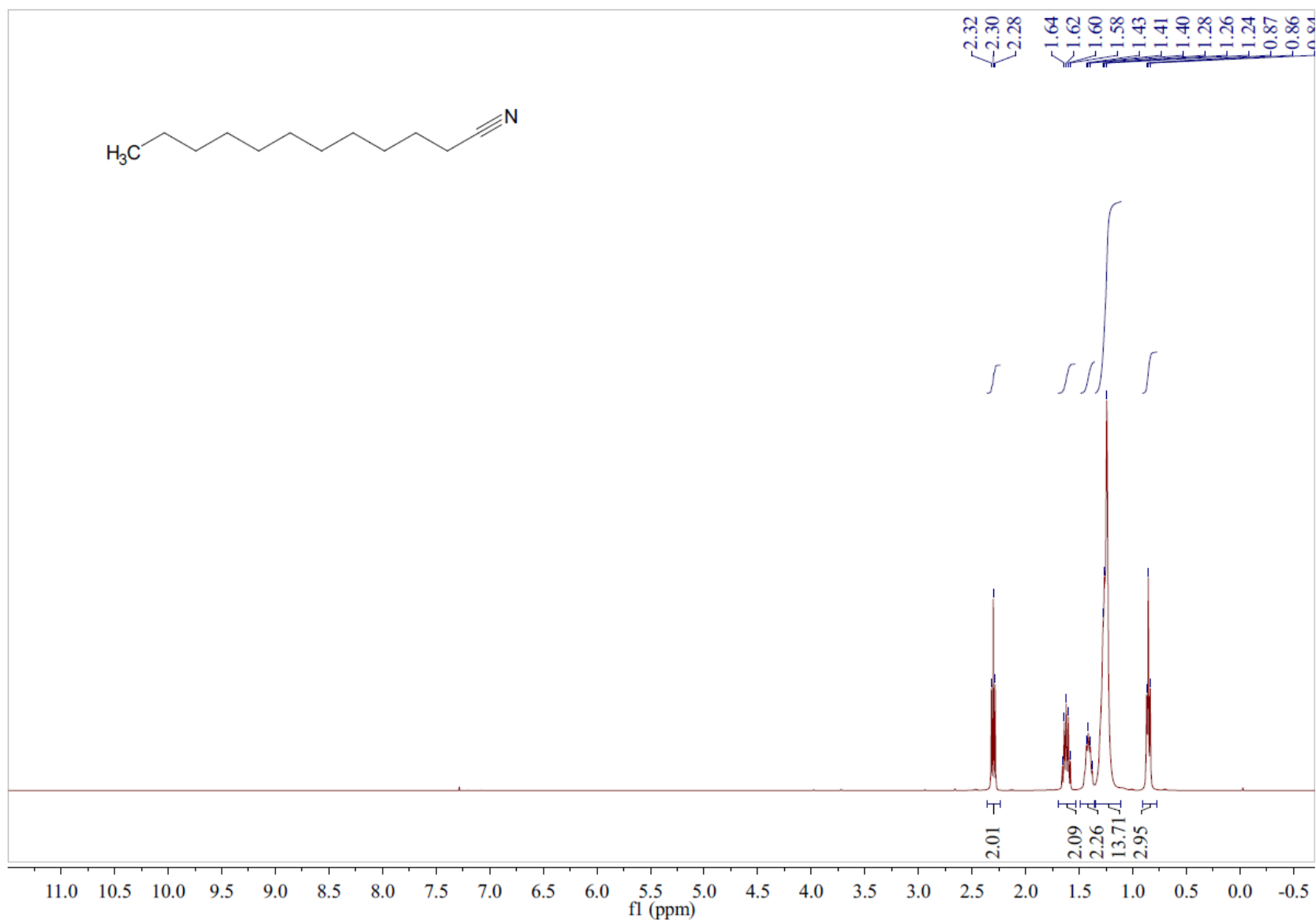
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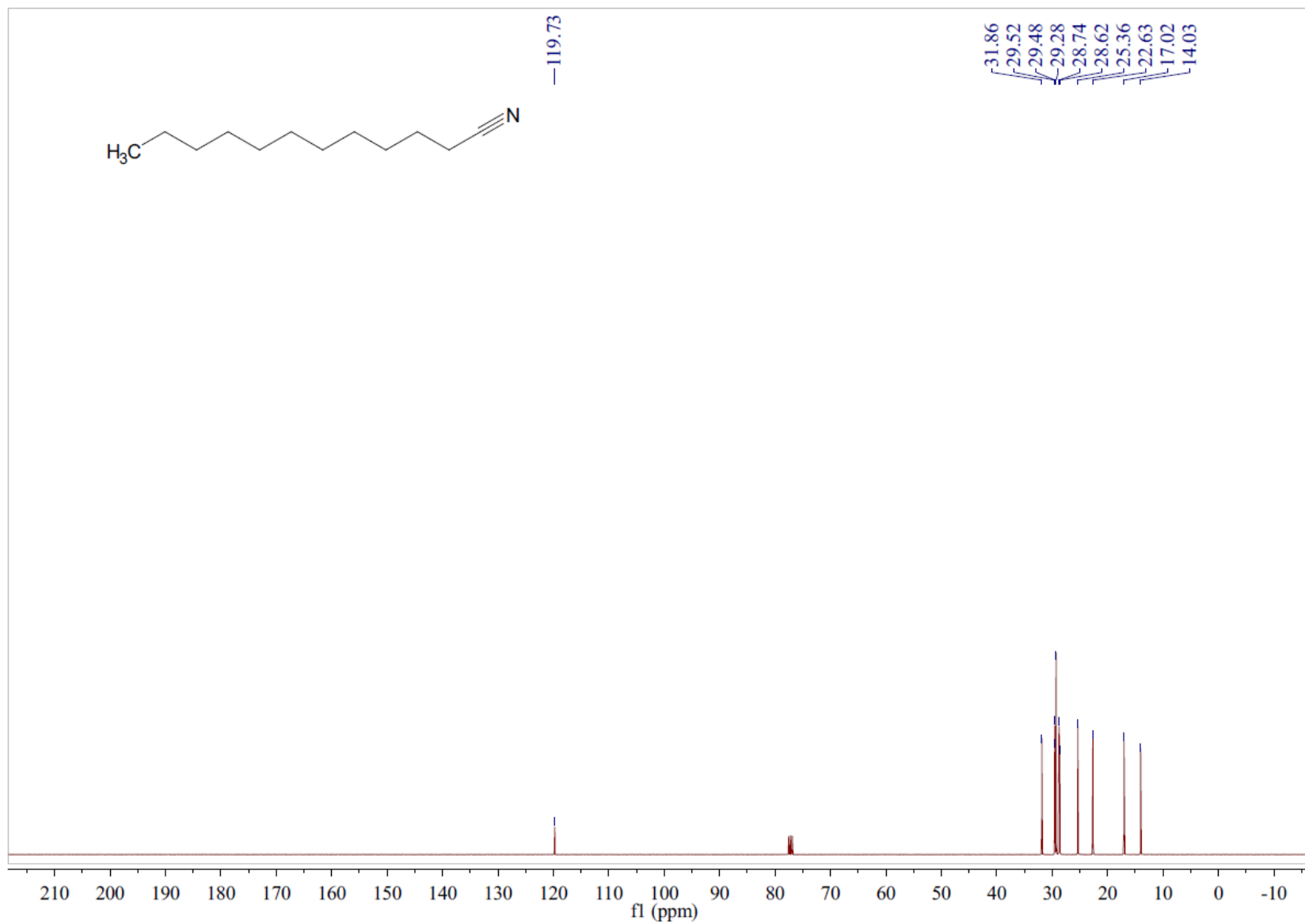
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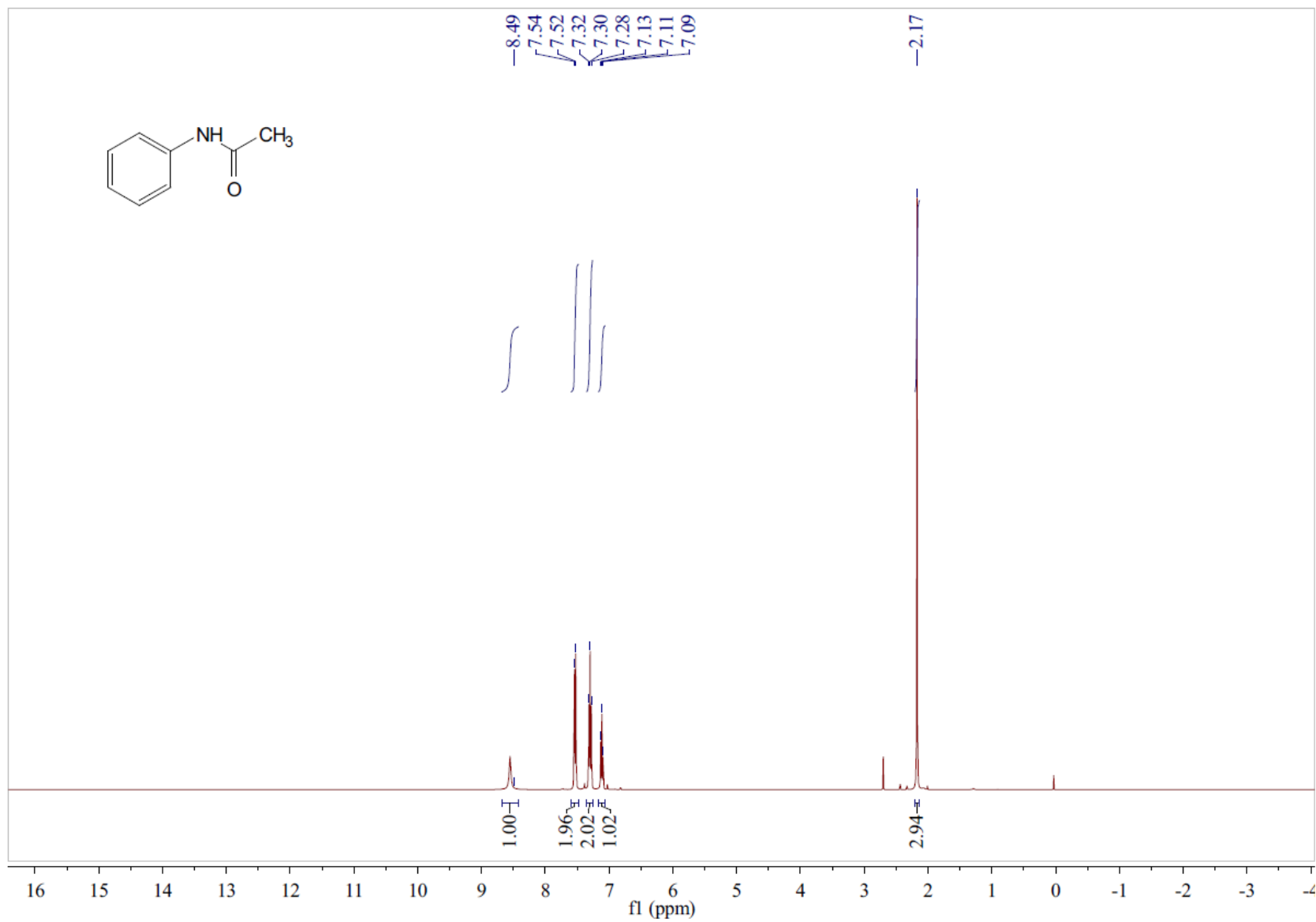
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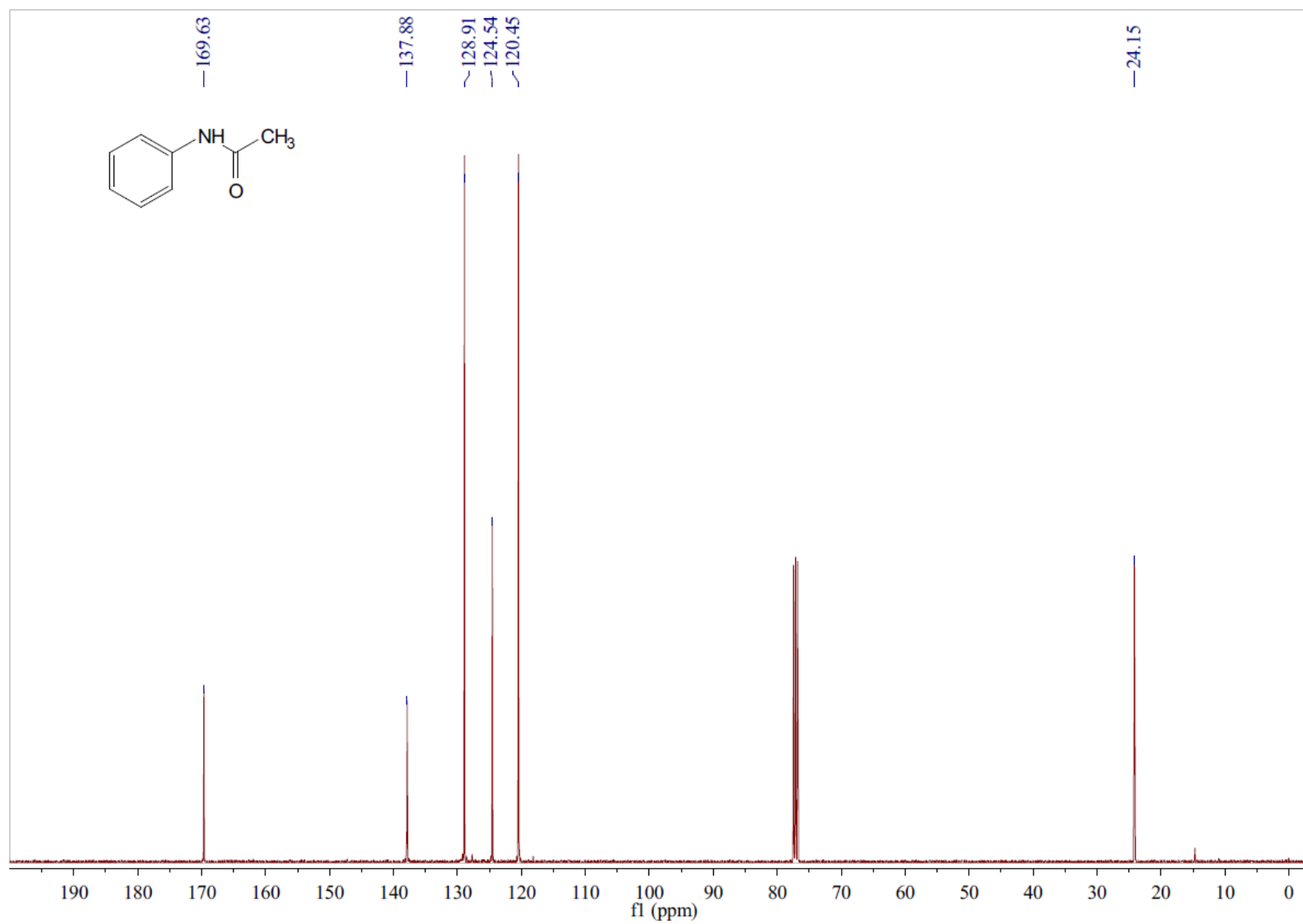
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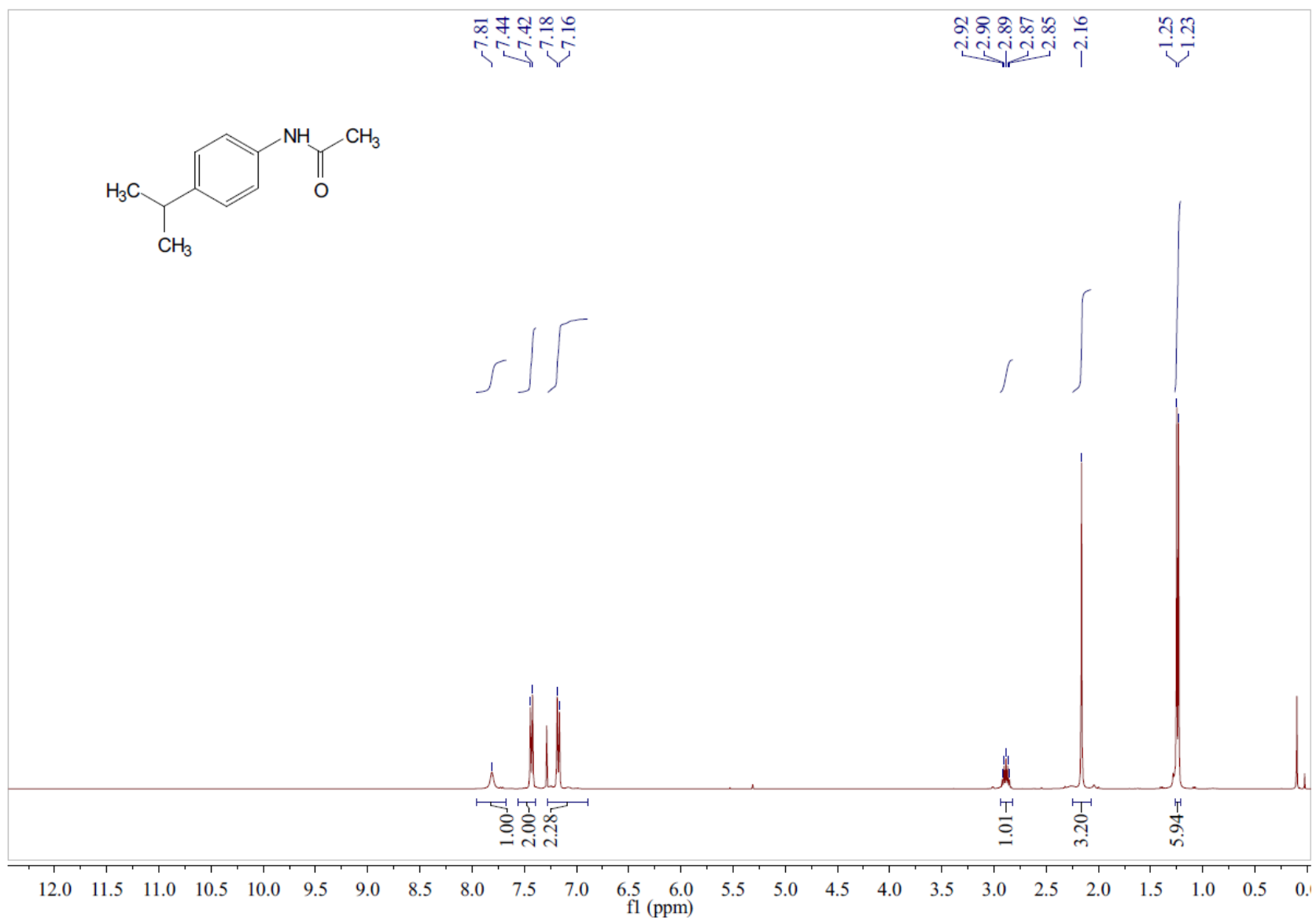
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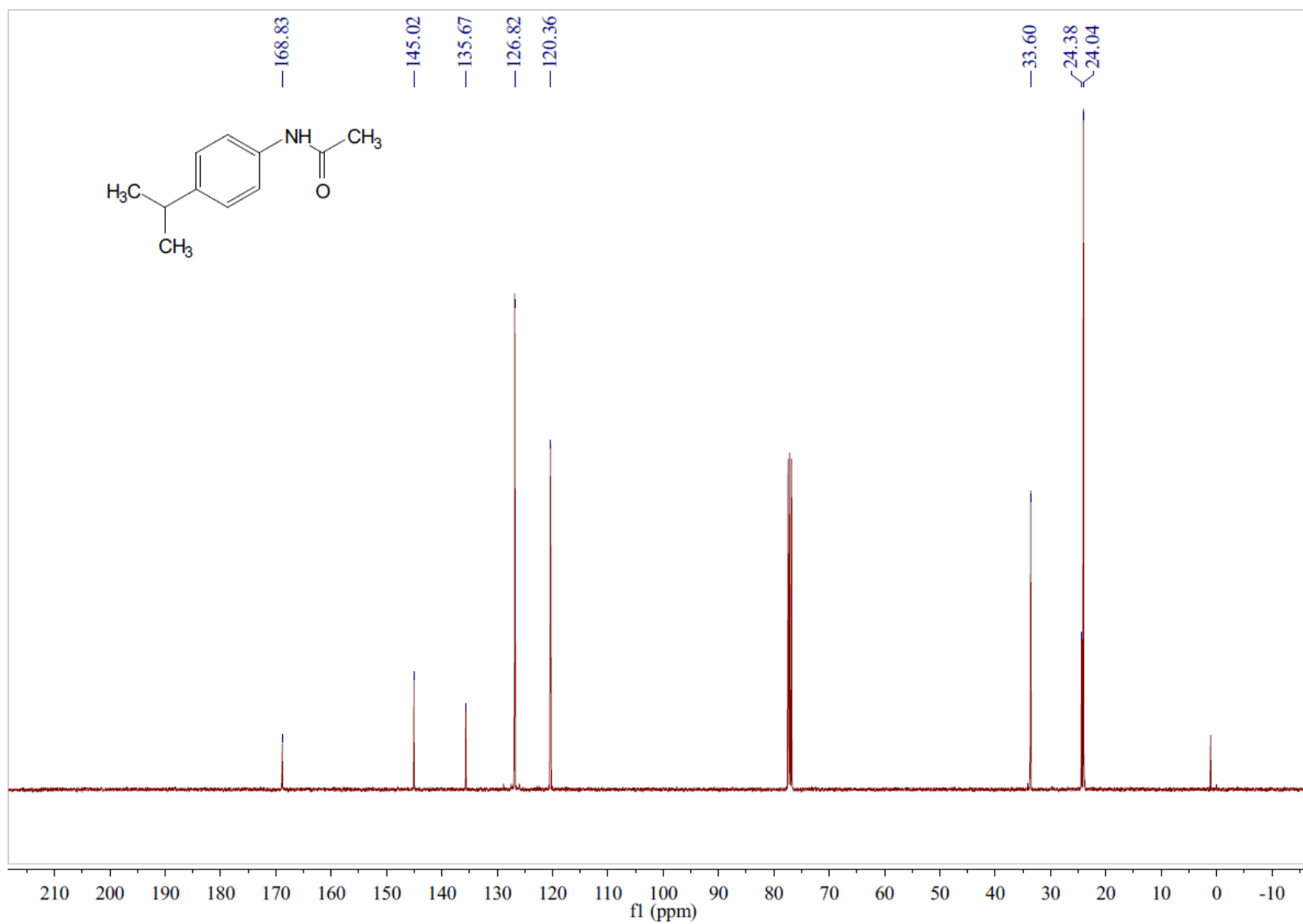
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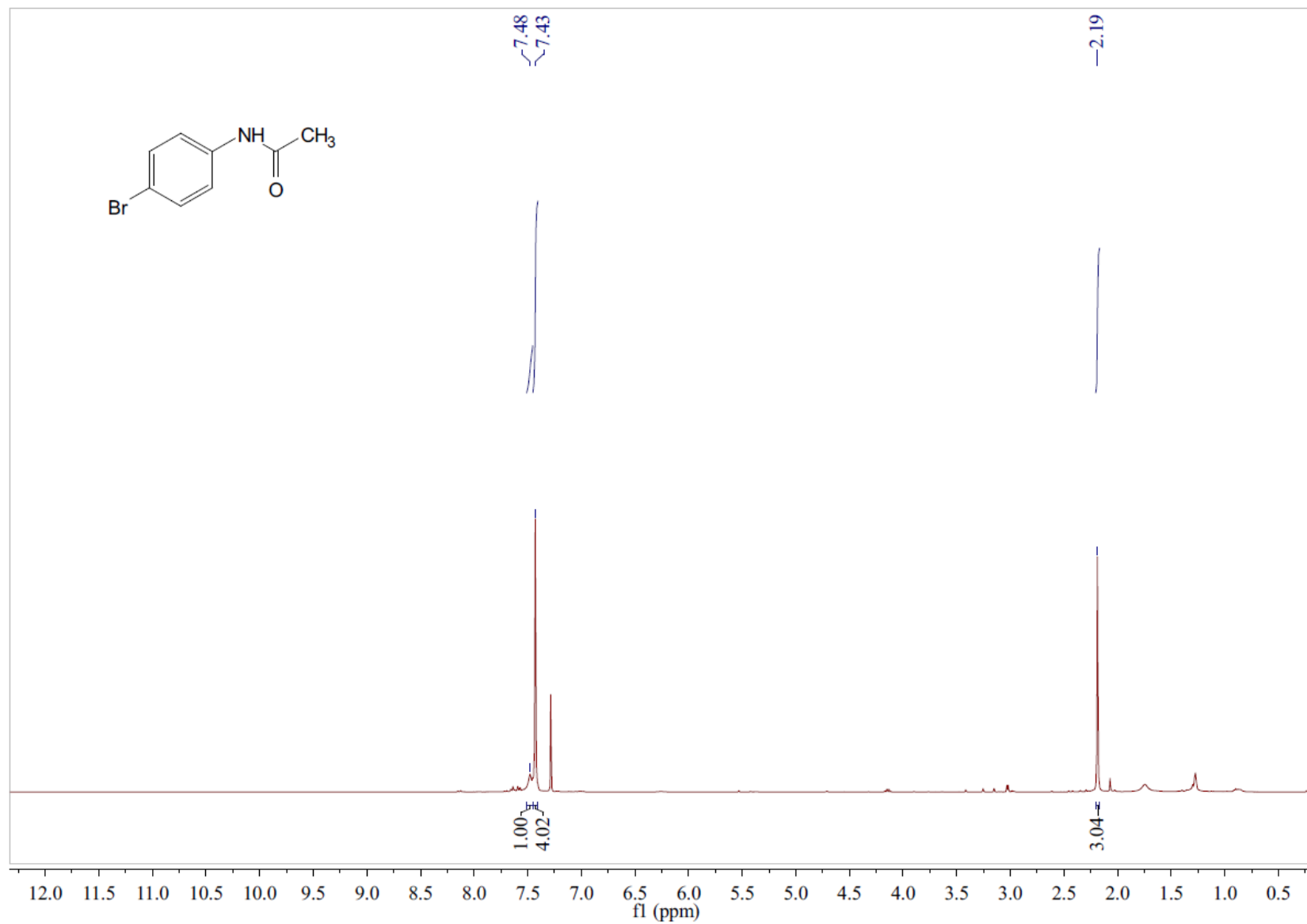
¹H NMR Spectra of 4b



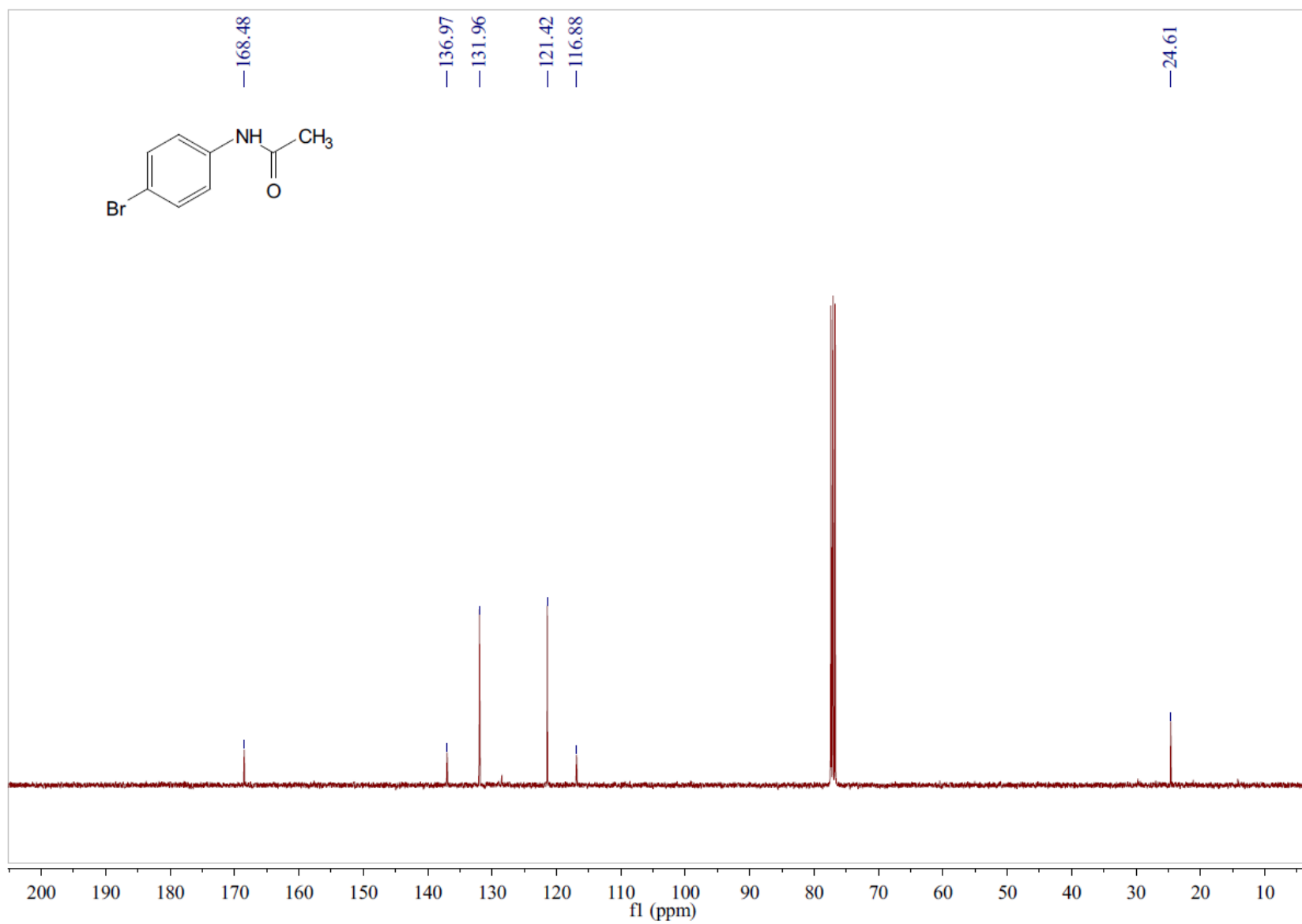
¹³C NMR Spectra of 4b



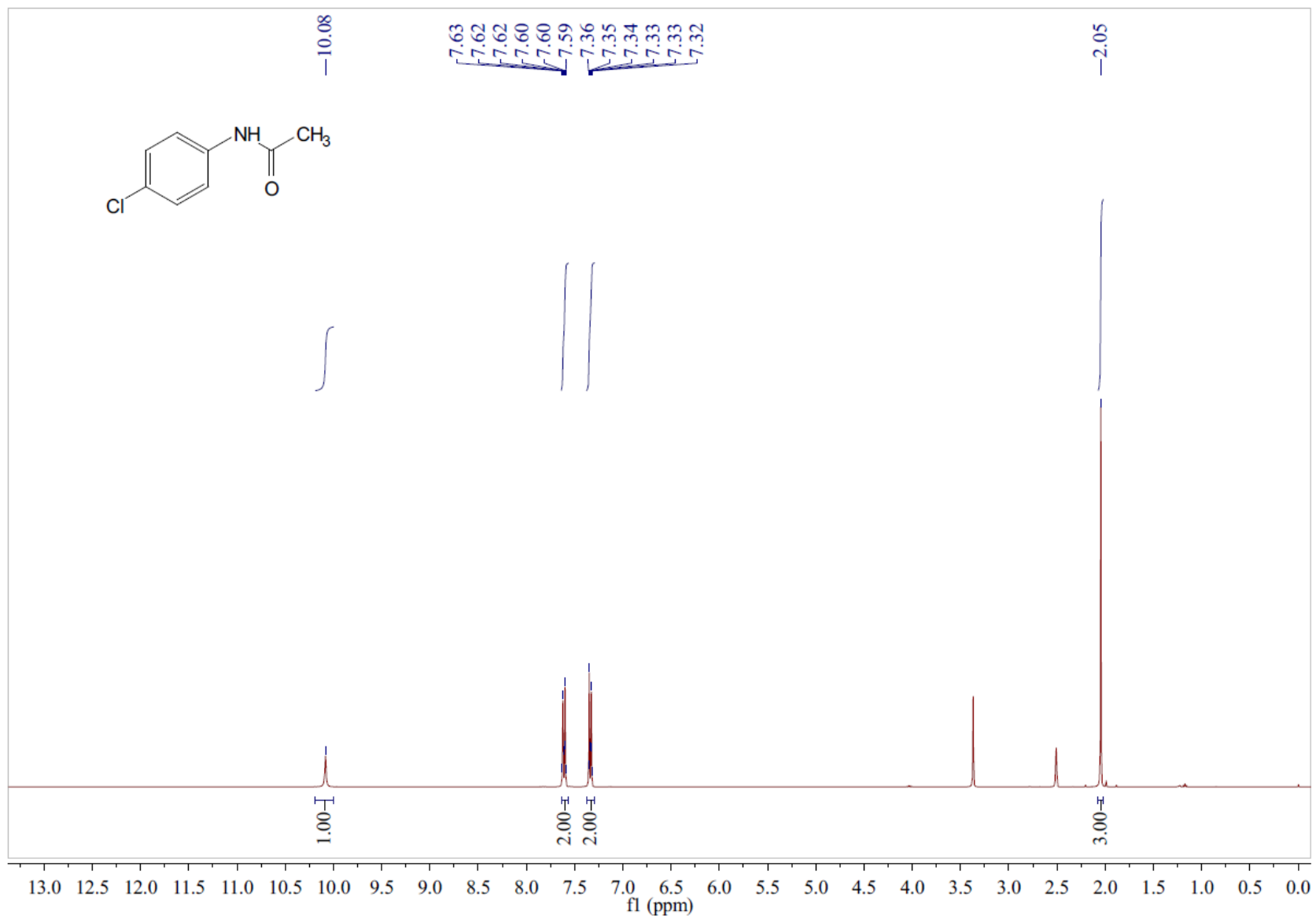
¹H NMR Spectra of 4c



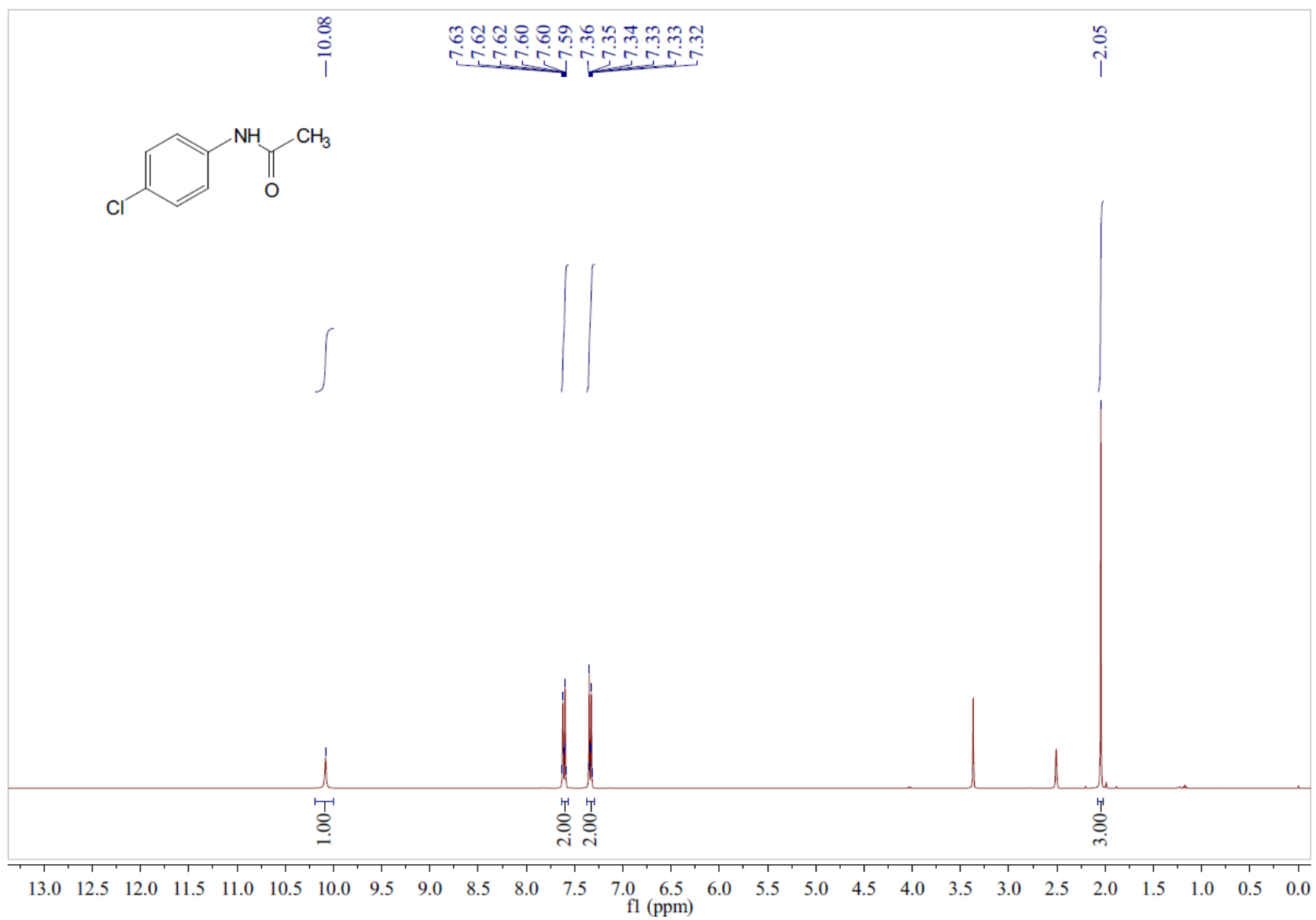
¹³C NMR Spectra of 4c



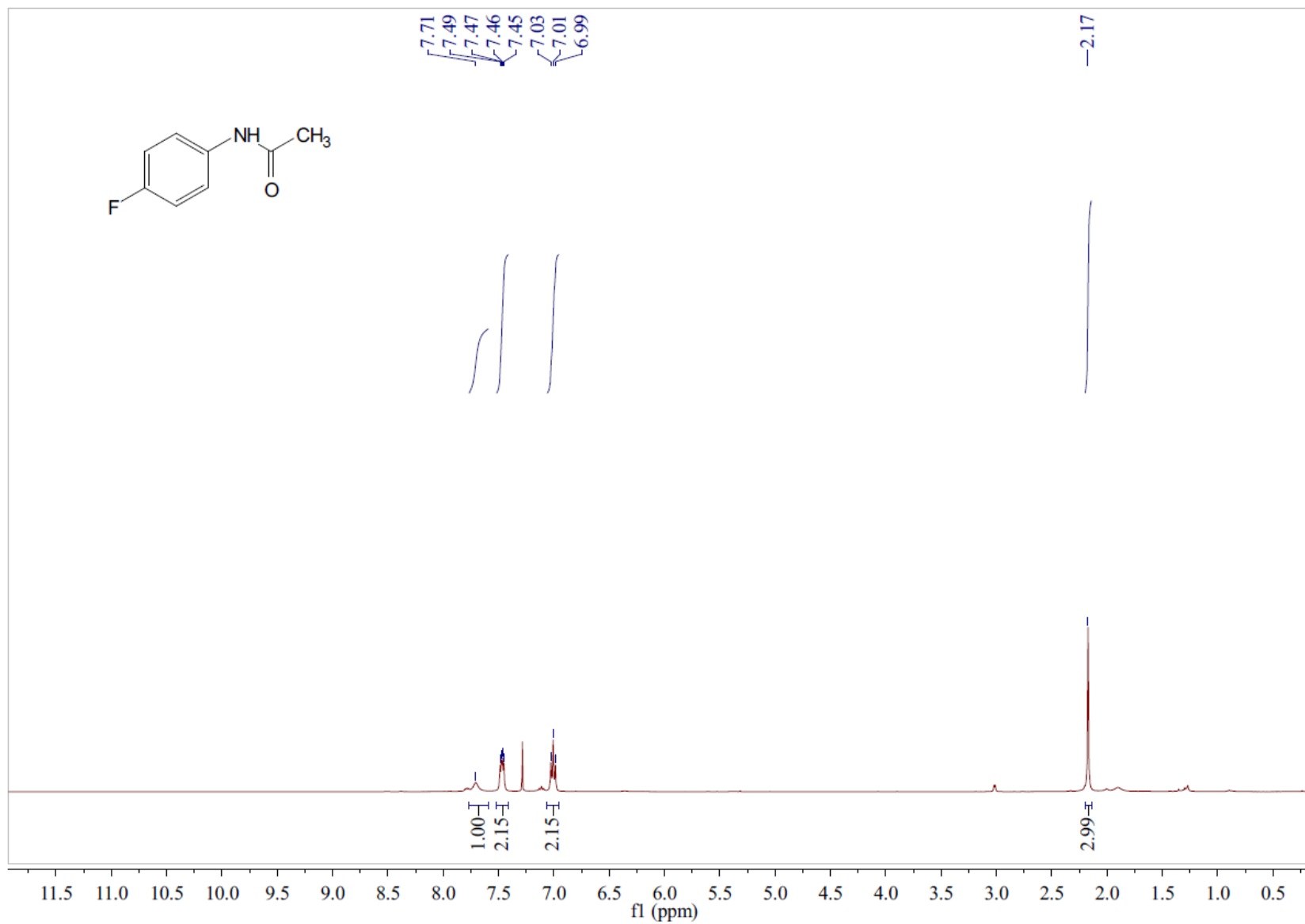
¹H NMR Spectra of 4d



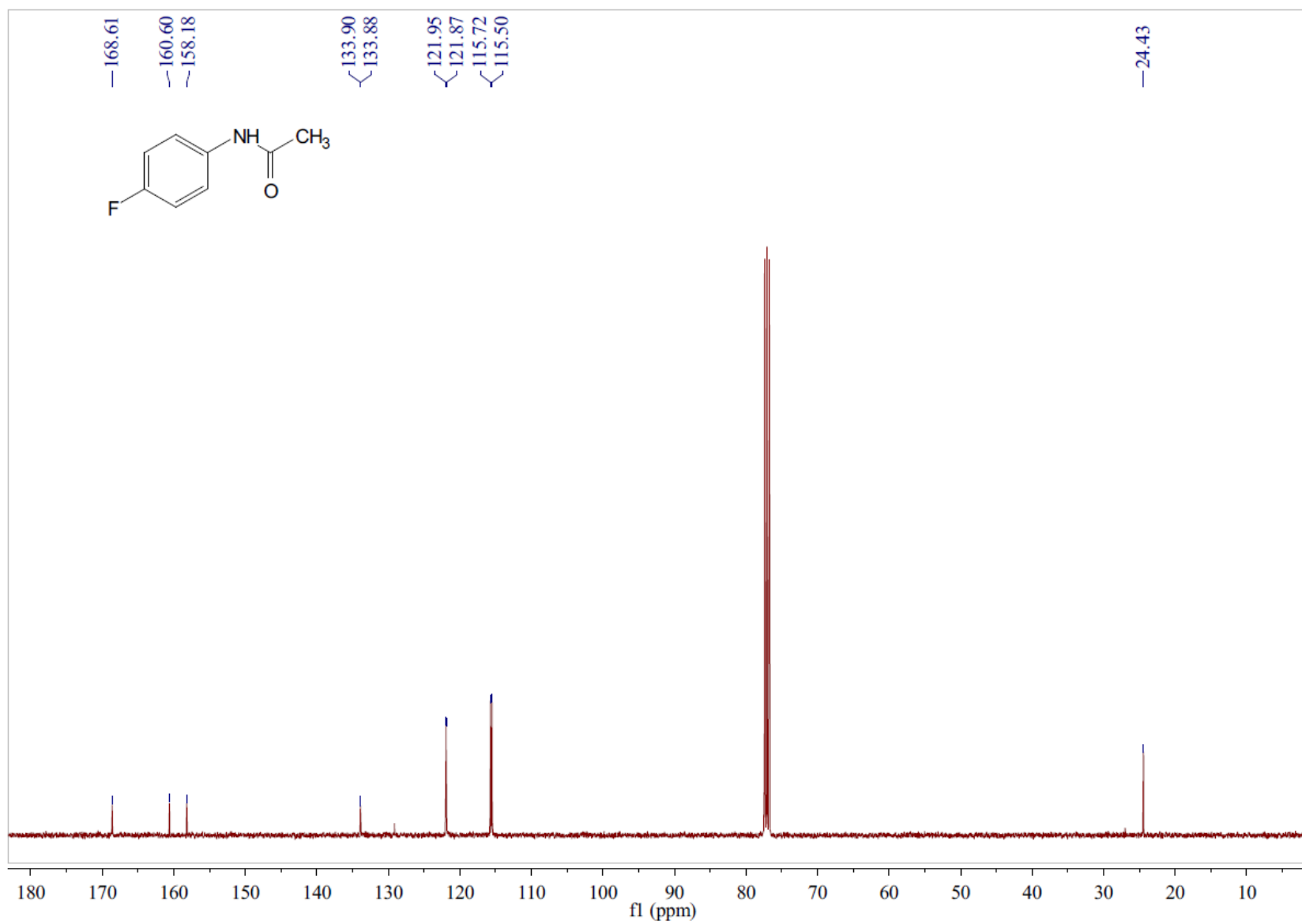
¹³C NMR Spectra of 4d



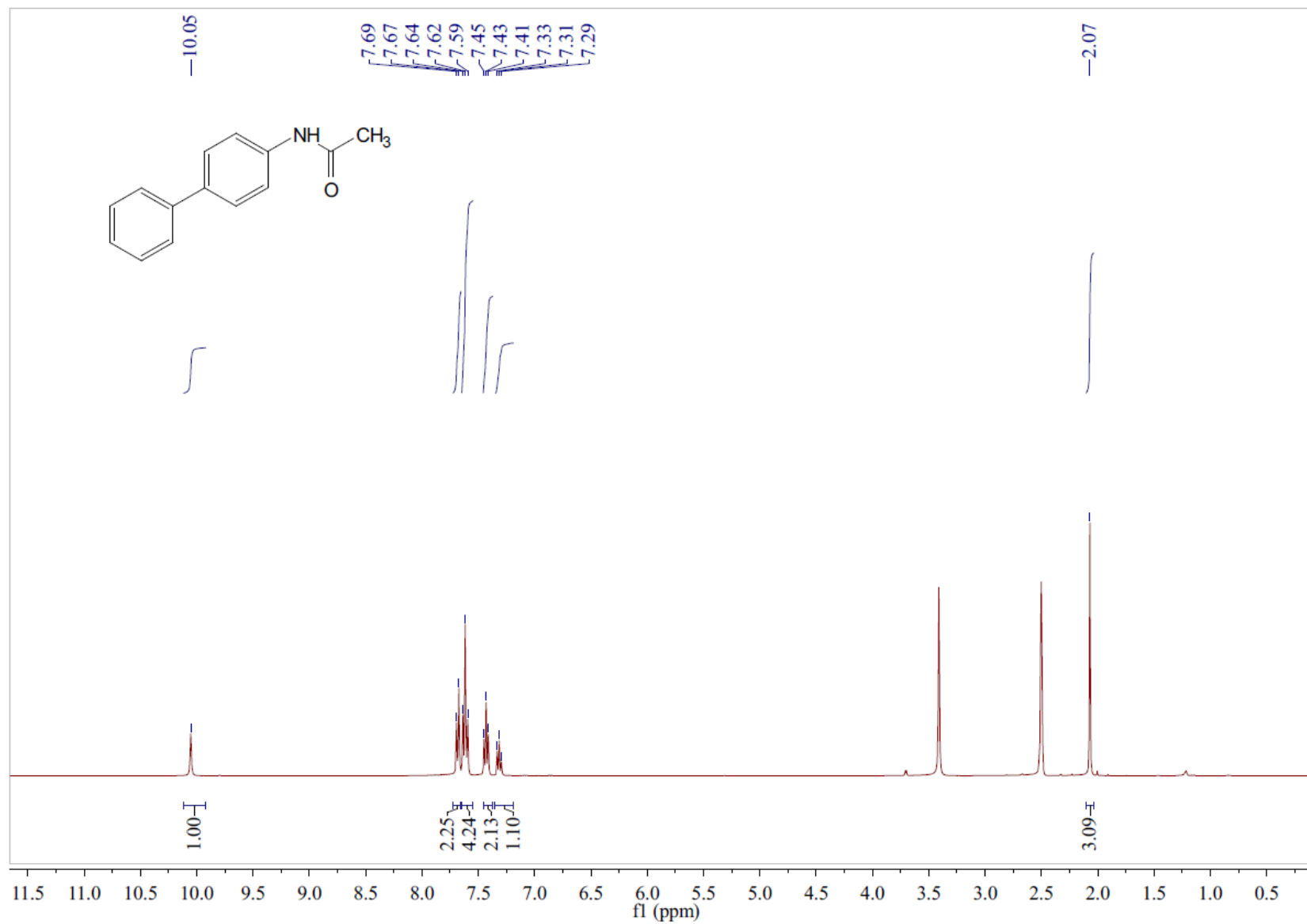
¹H NMR Spectra of 4e



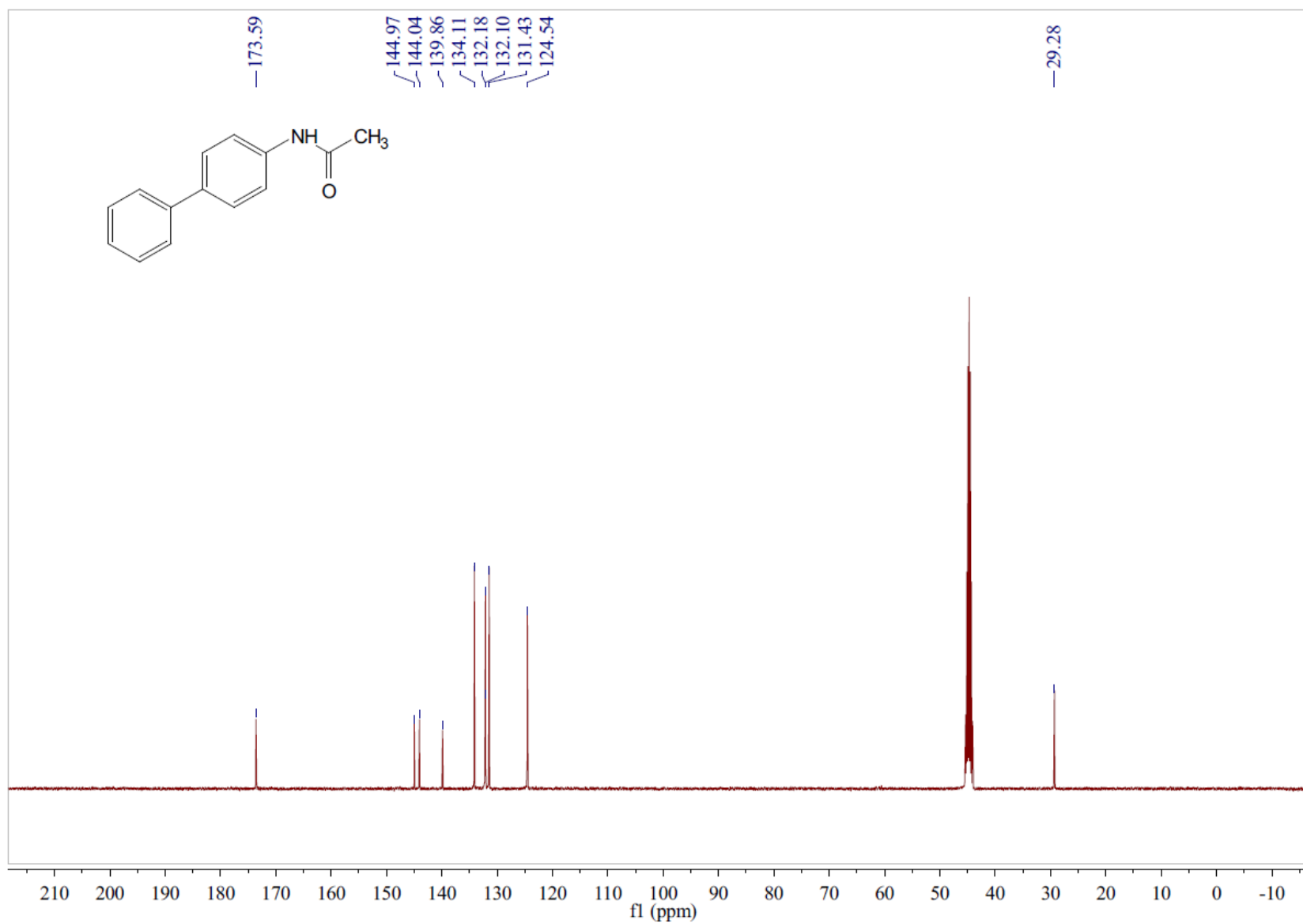
¹³C NMR Spectra of 4e



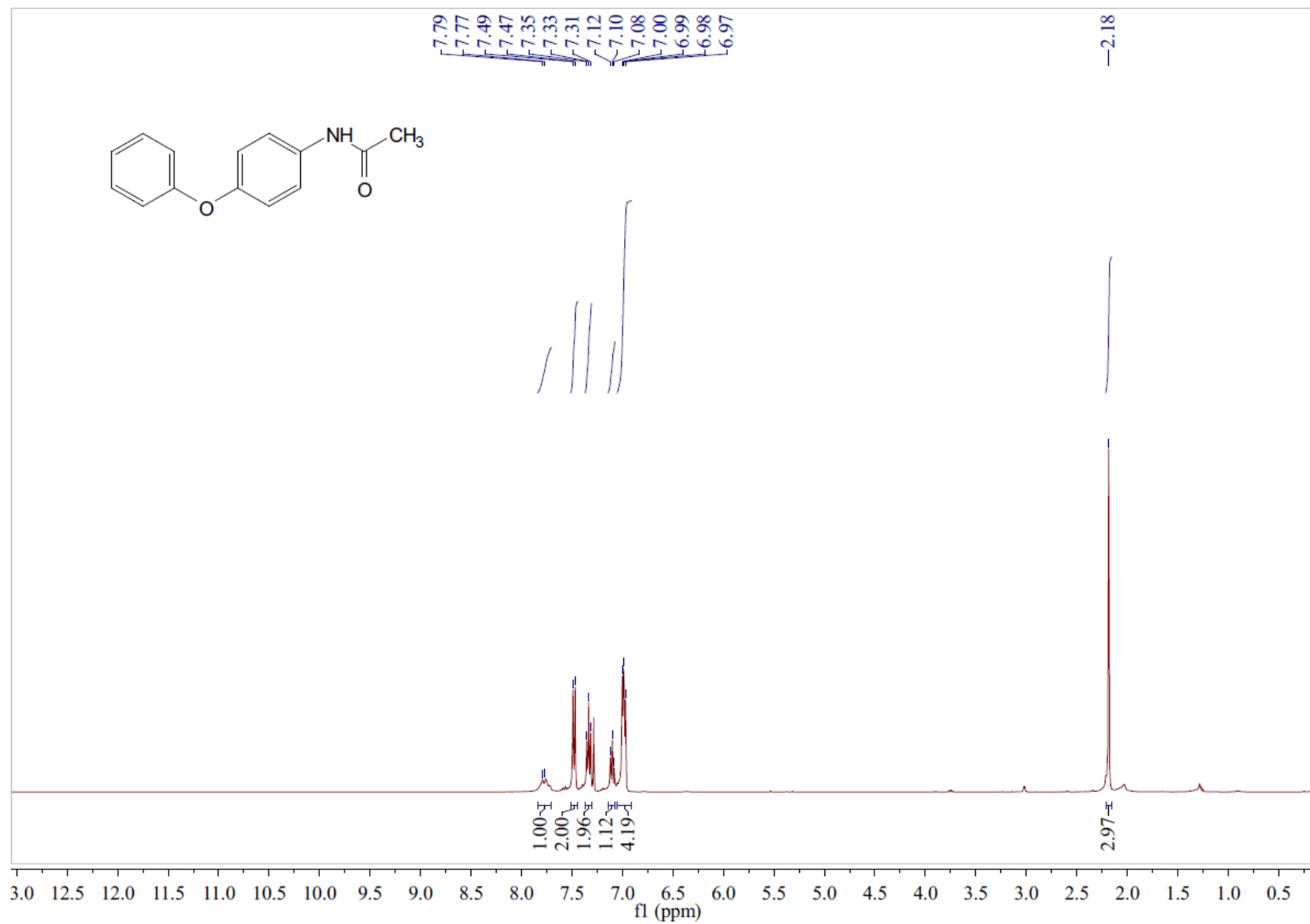
¹H NMR Spectra of 4f



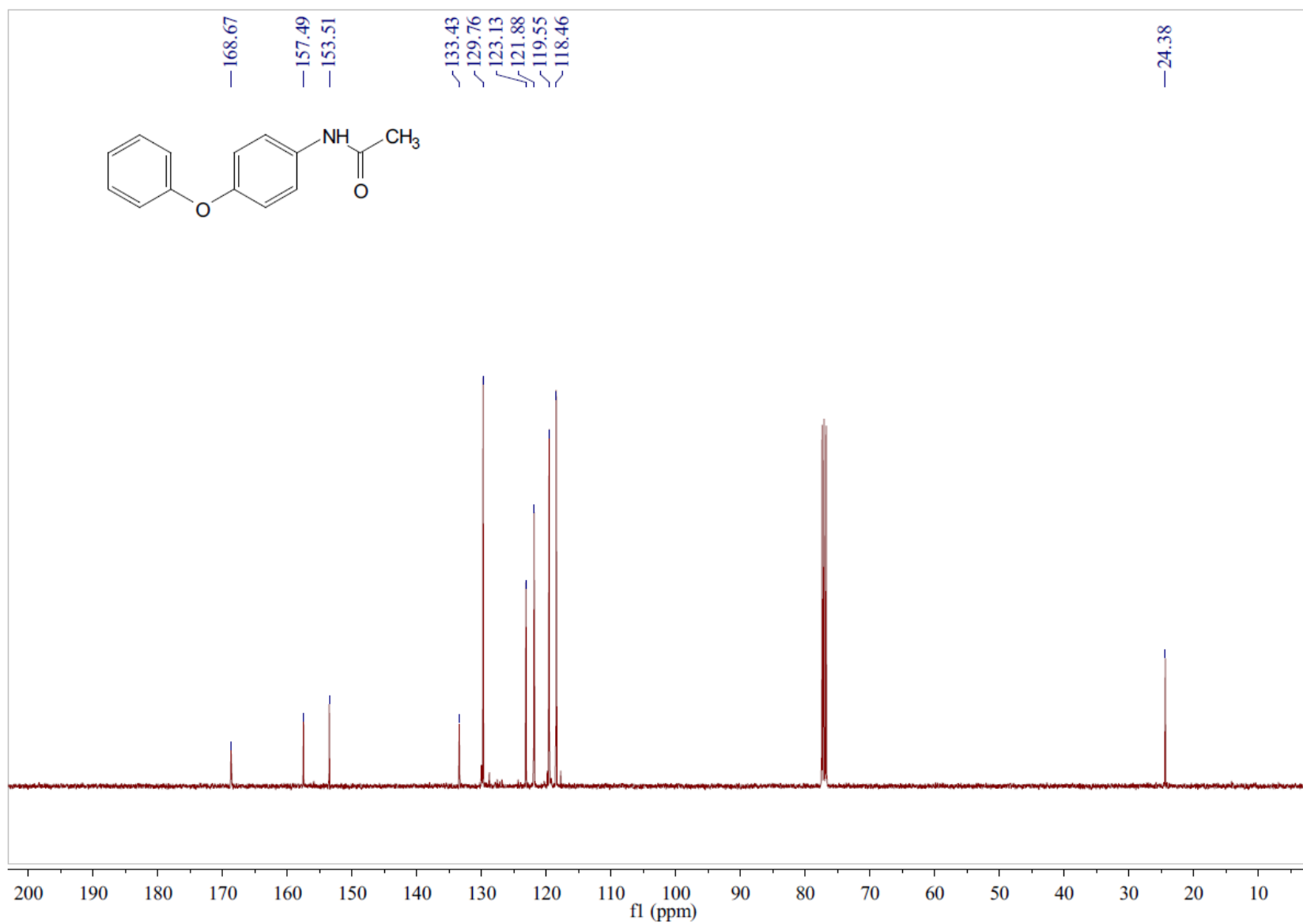
¹³C NMR Spectra of 4f



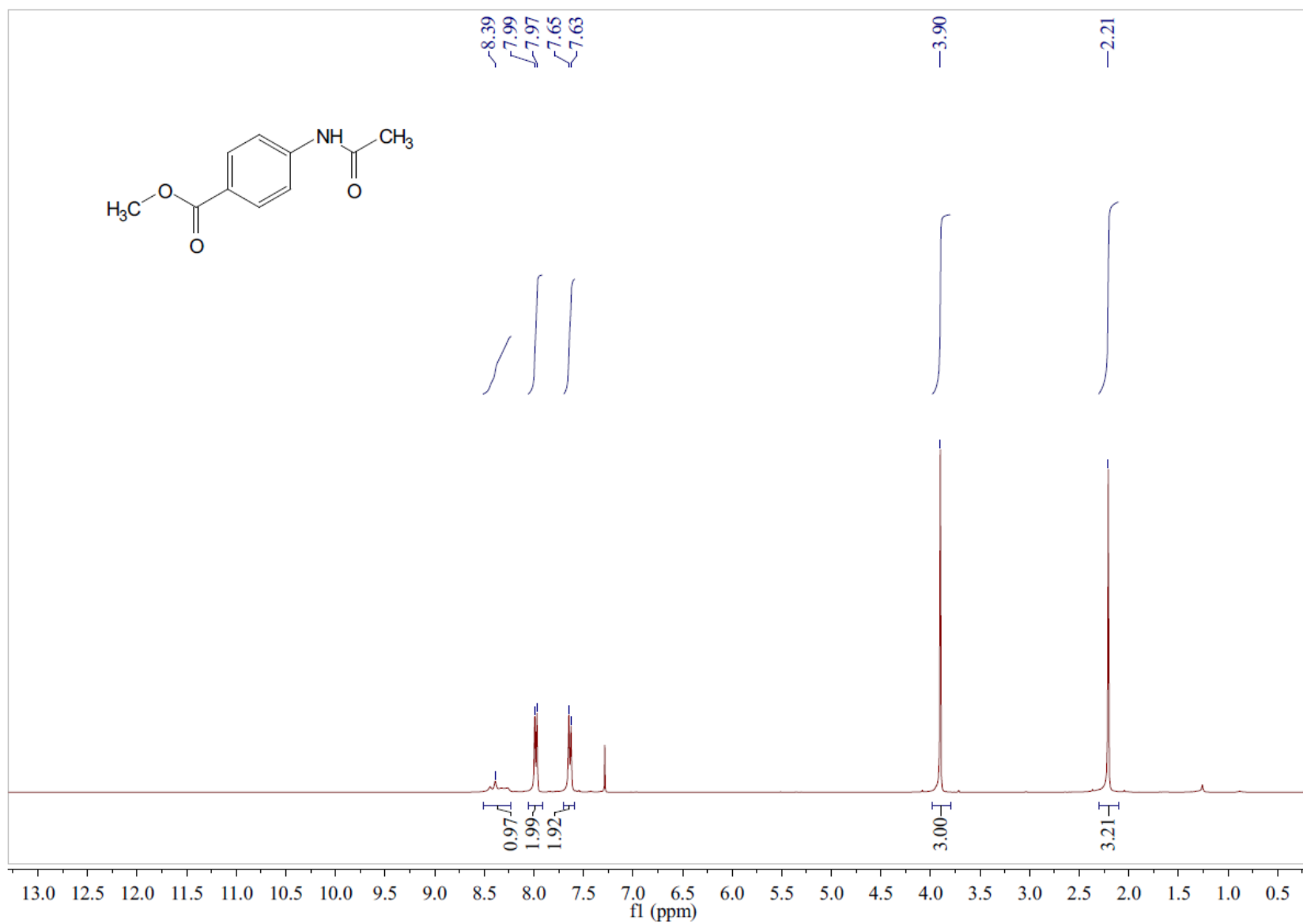
¹H NMR Spectra of 4g



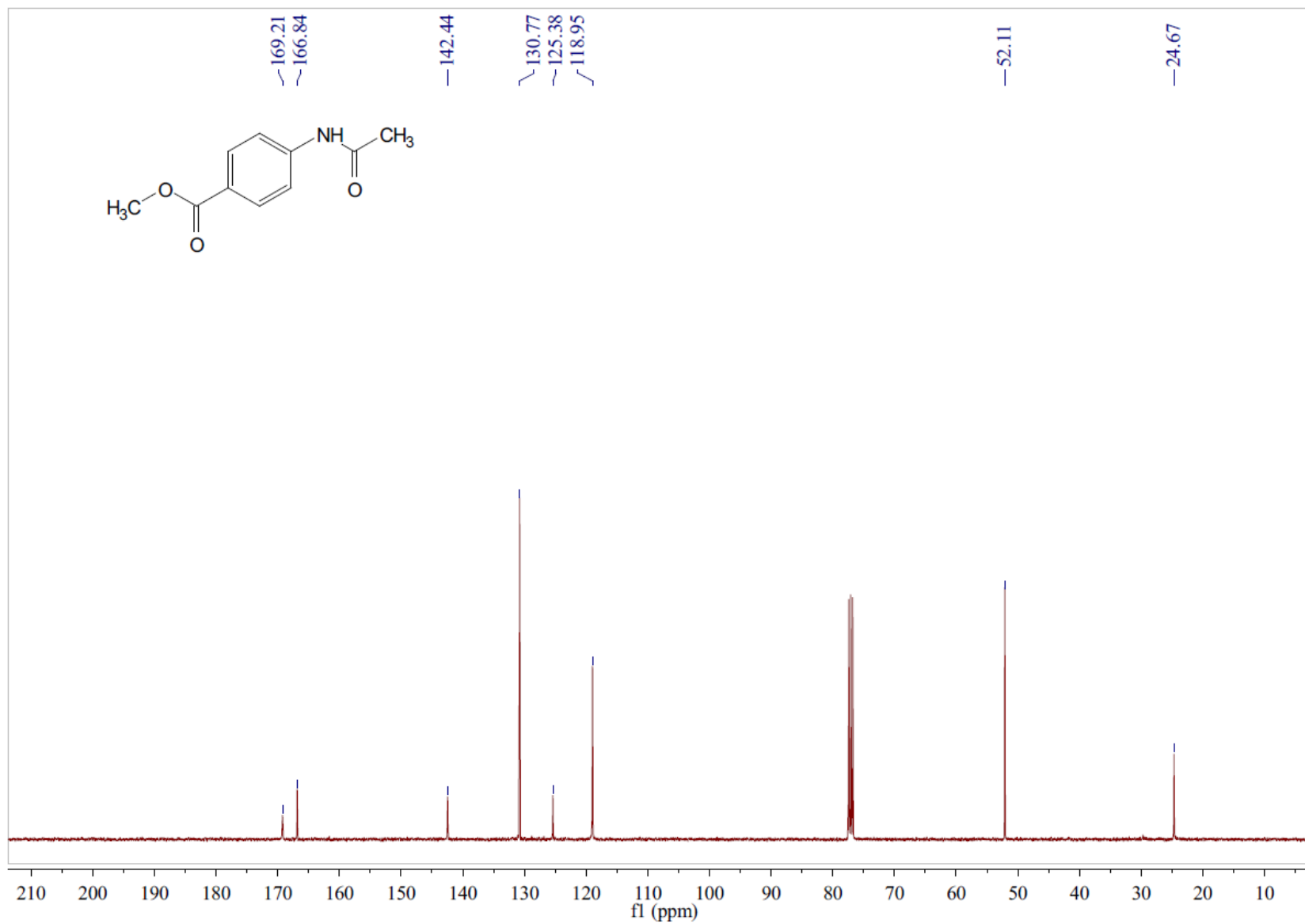
¹³C NMR Spectra of 4g



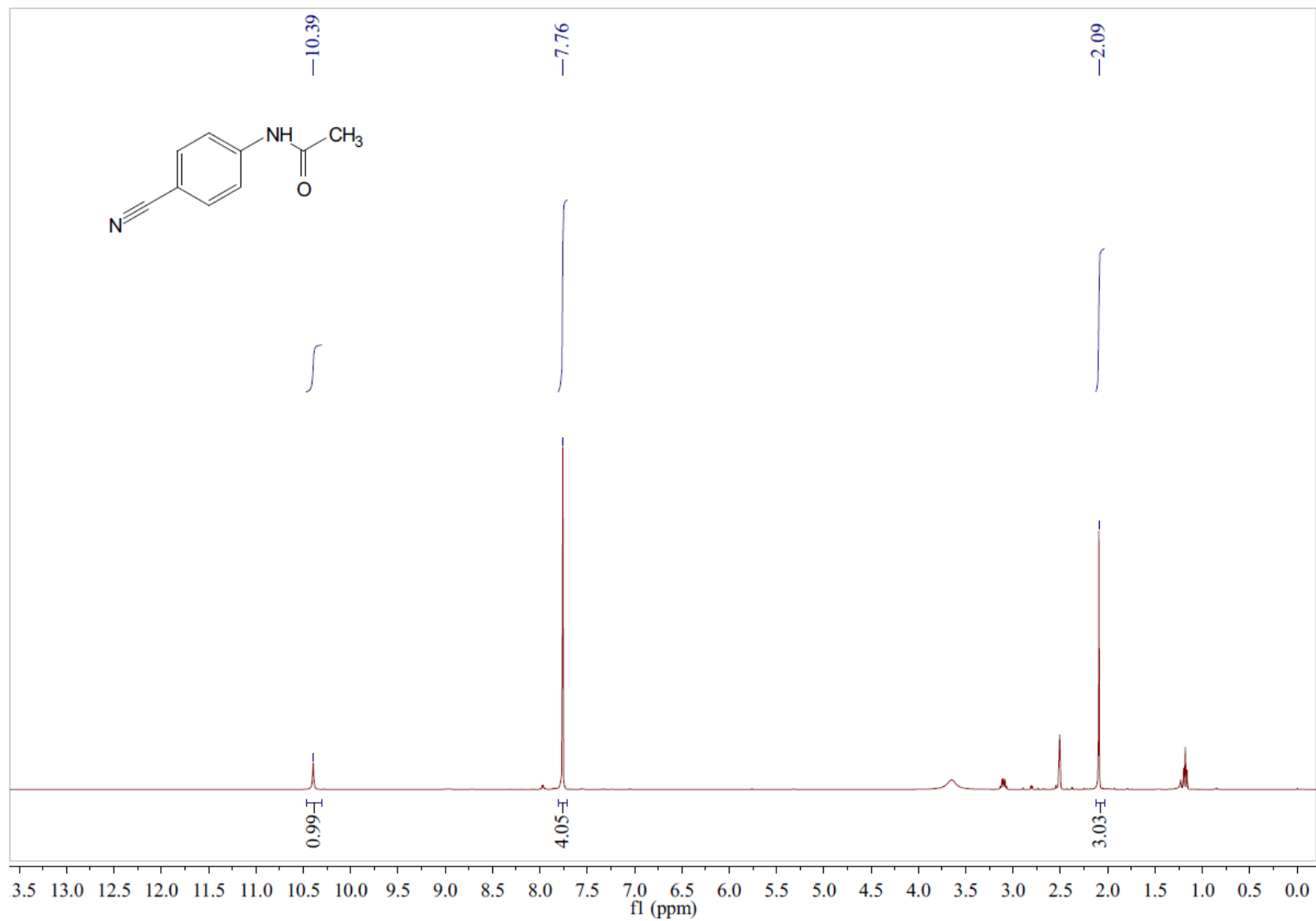
¹H NMR Spectra of 4h



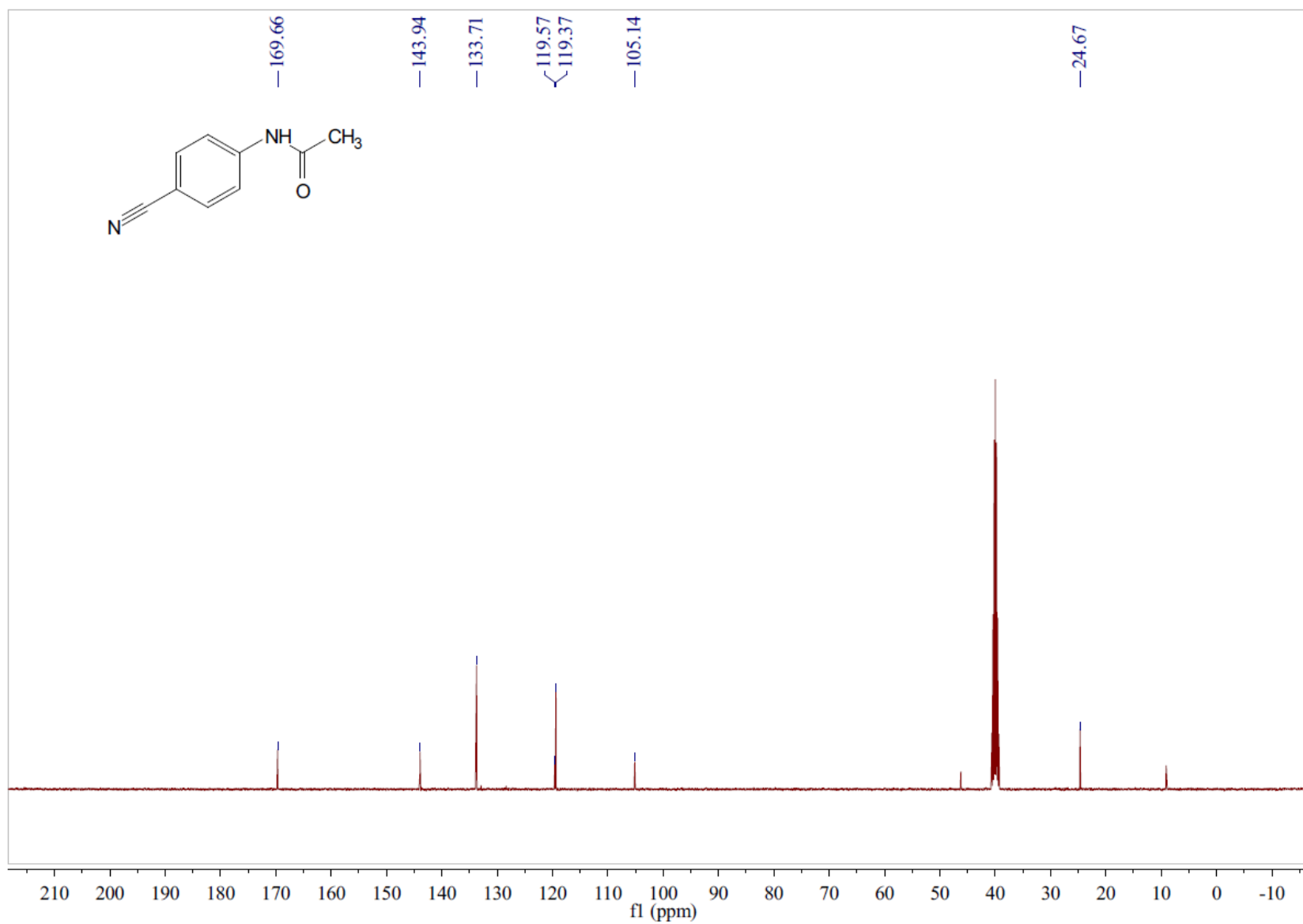
¹³C NMR Spectra of 4h



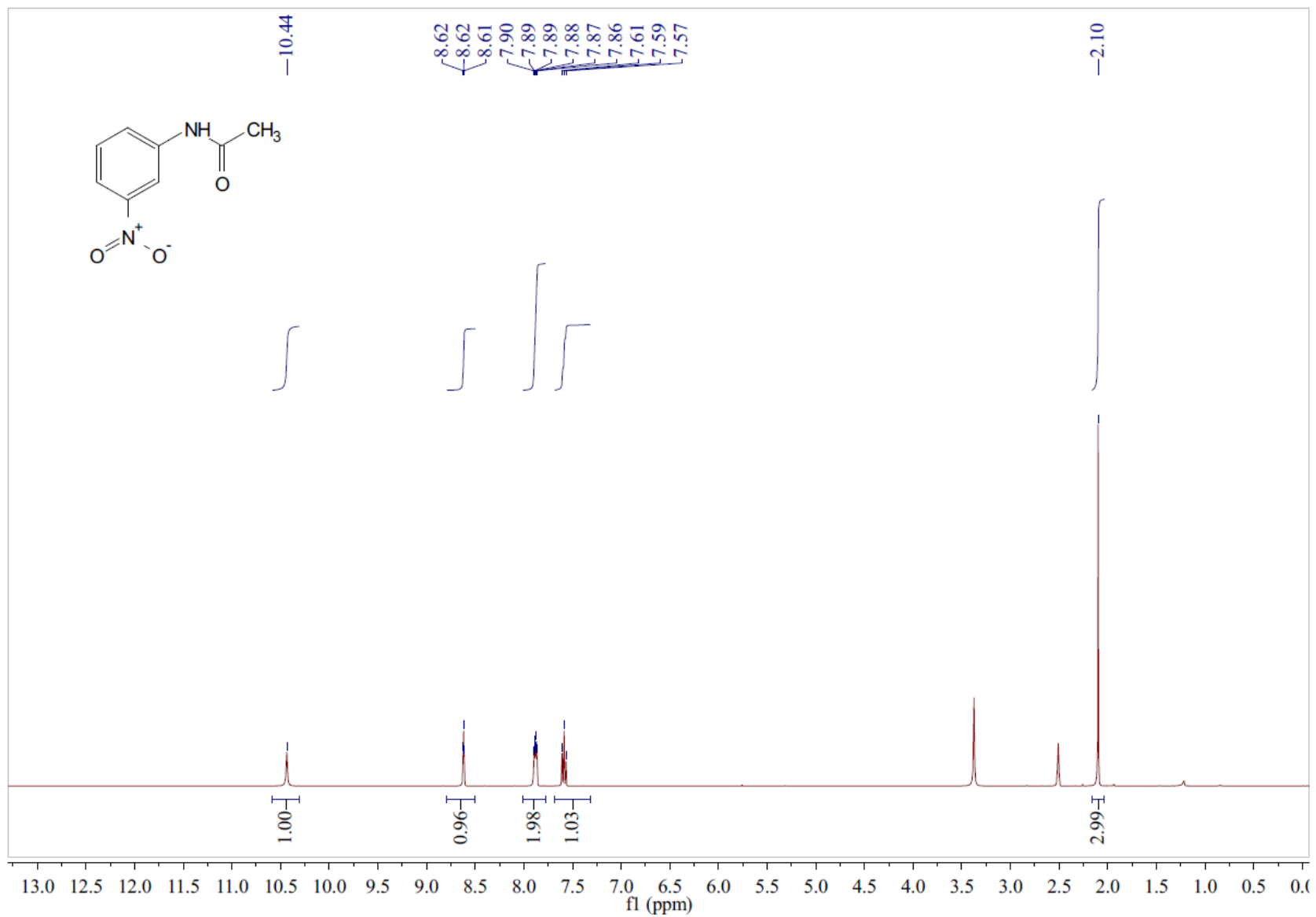
¹H NMR Spectra of 4i



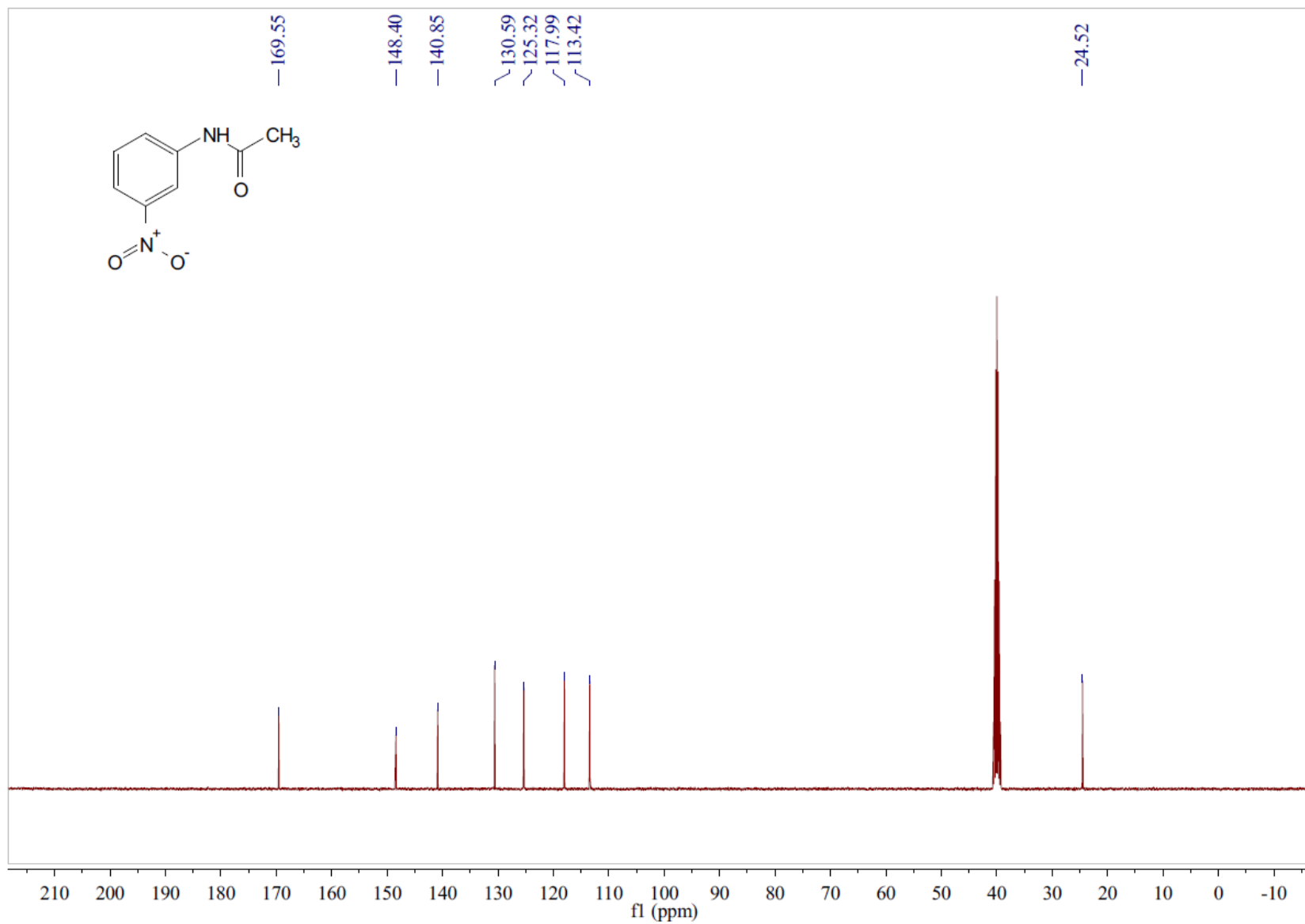
¹³C NMR Spectra of 4i



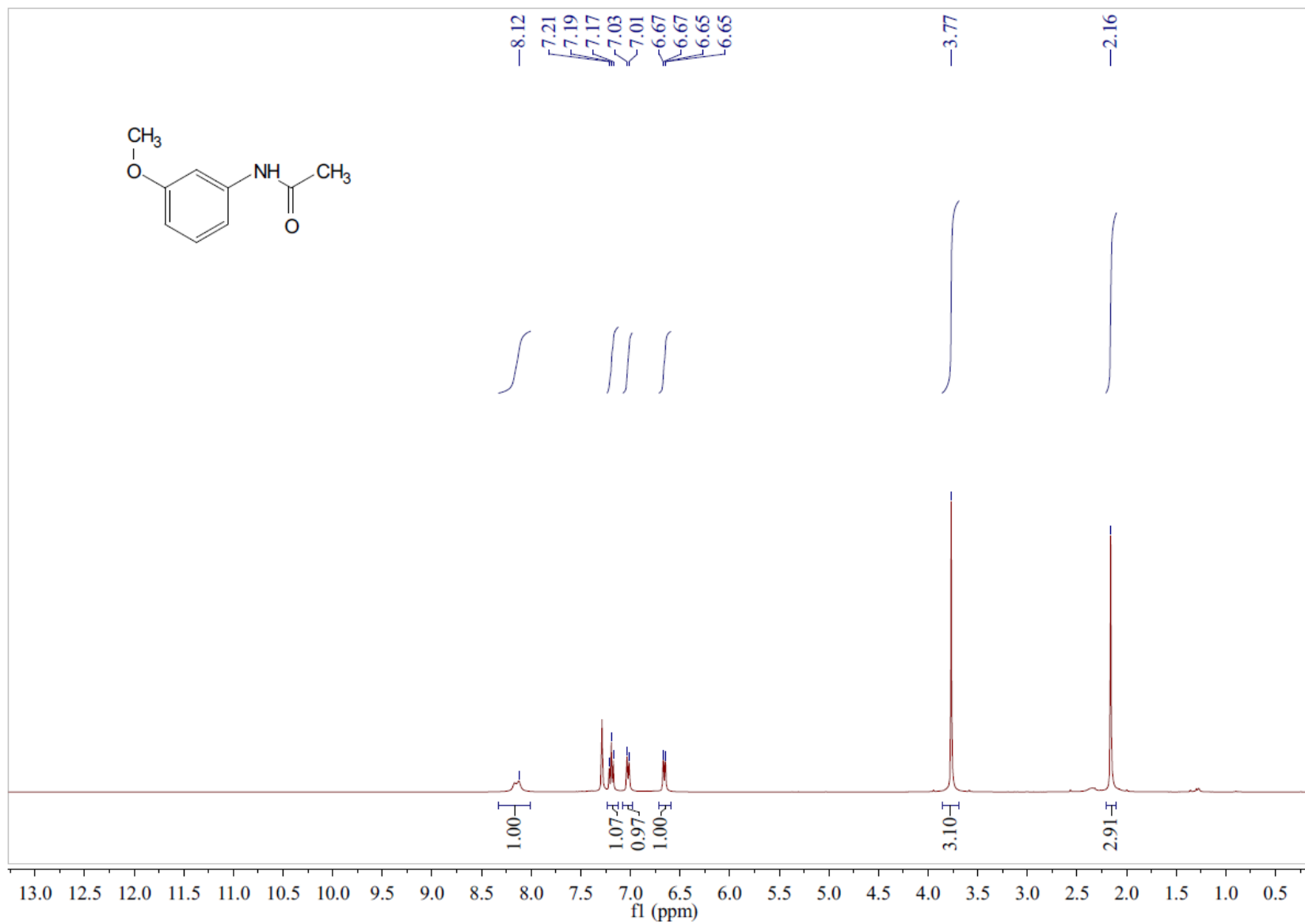
¹H NMR Spectra of 4j



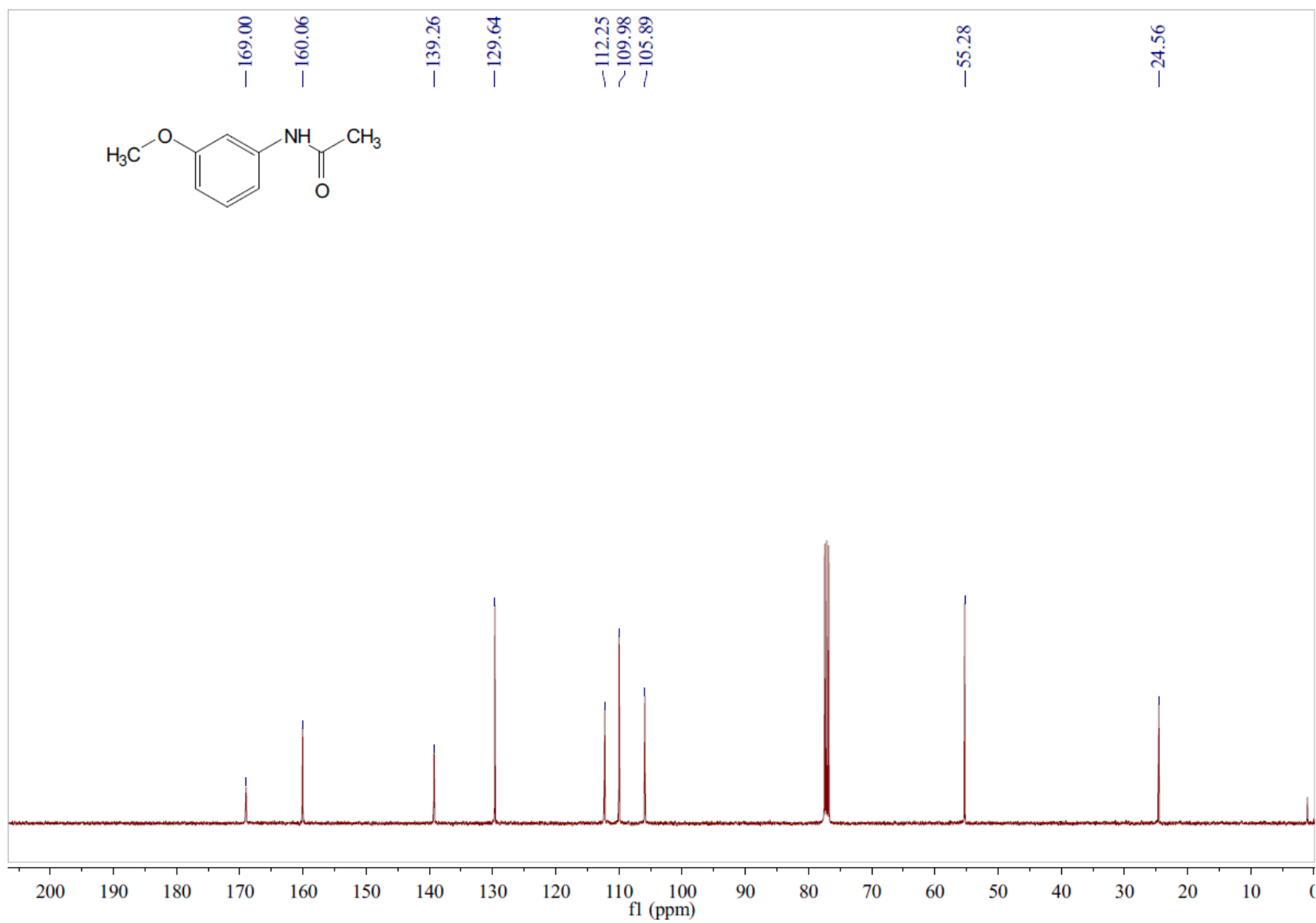
¹³C NMR Spectra of 4j



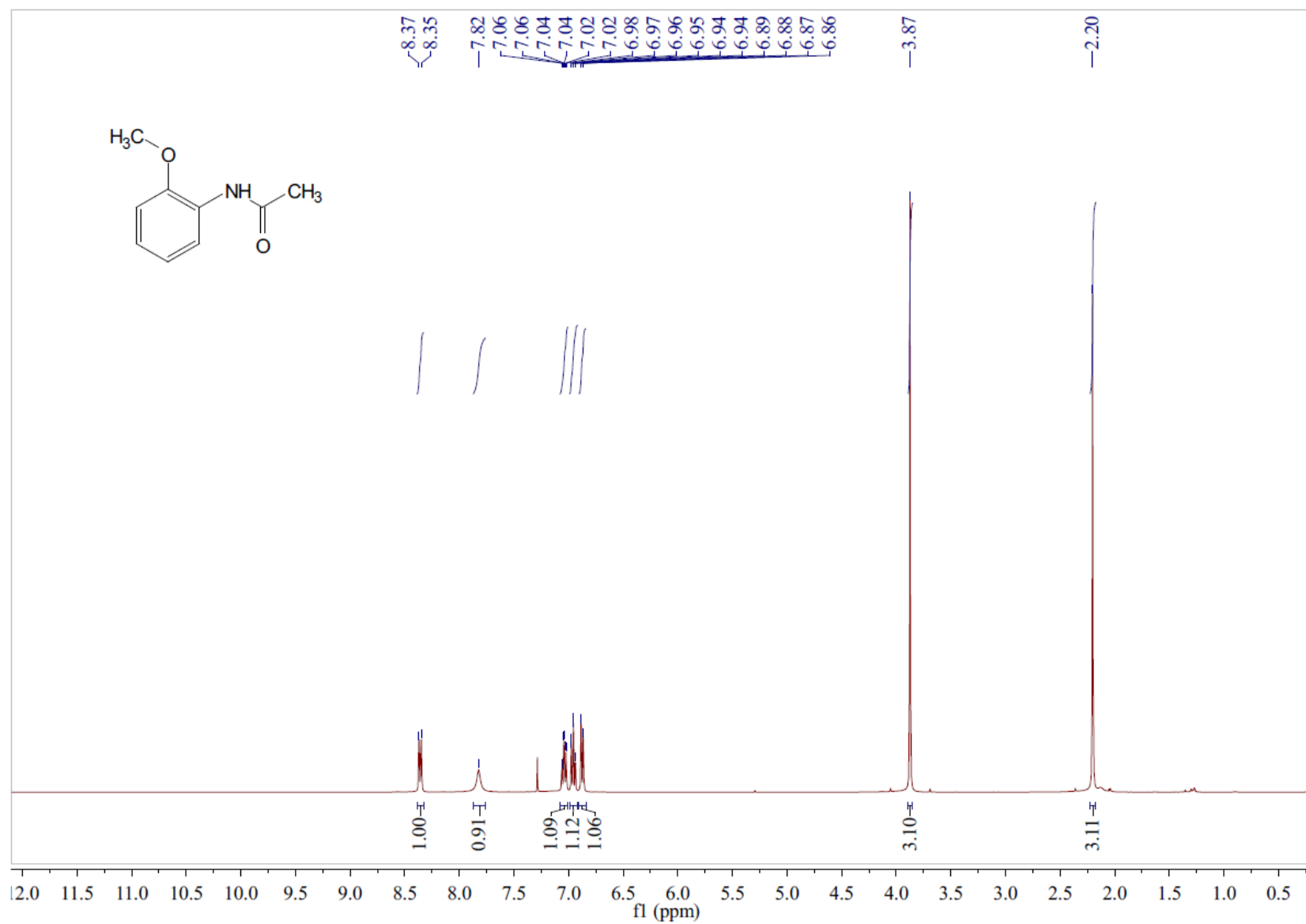
¹H NMR Spectra of 4k



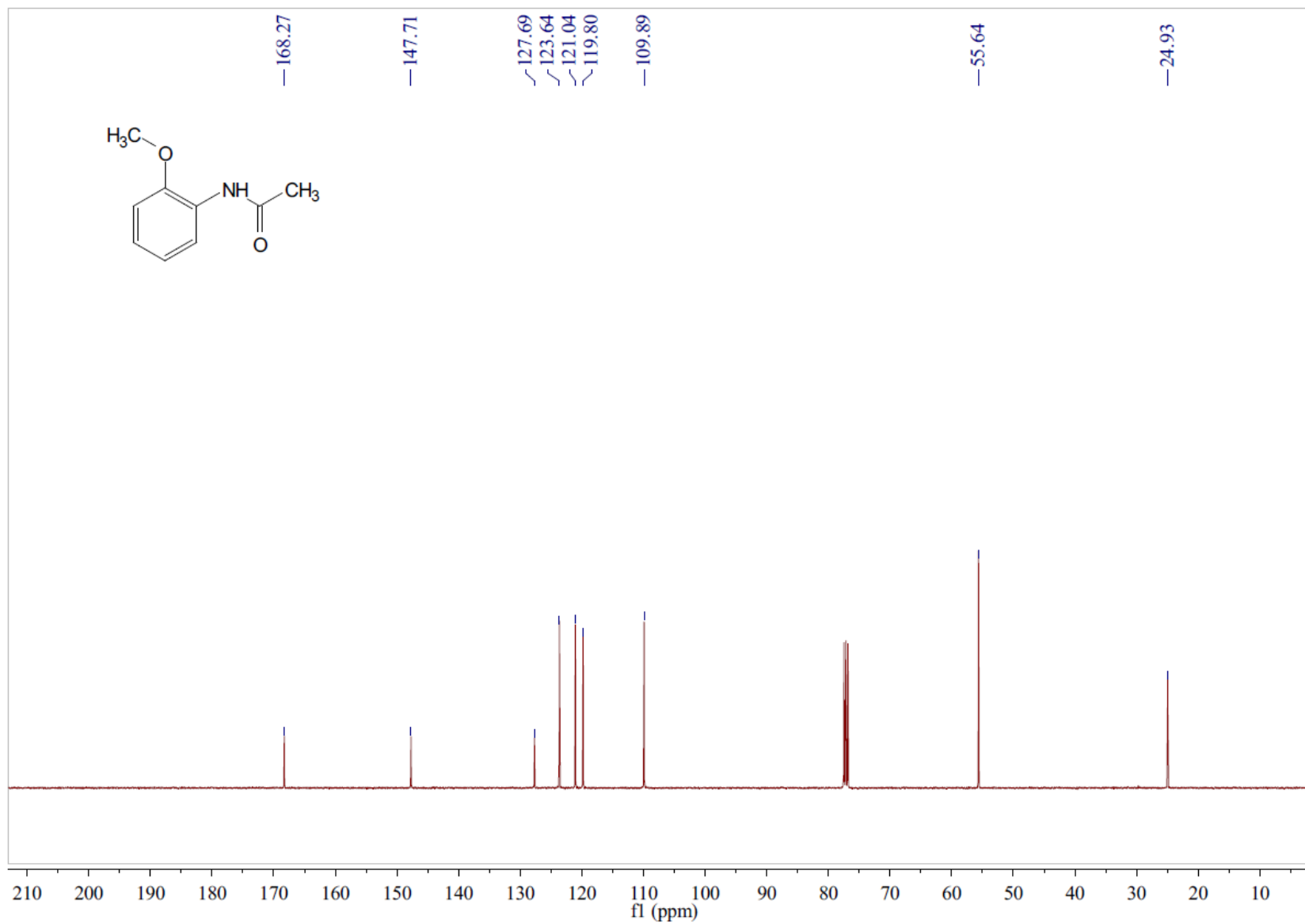
¹³C NMR Spectra of 4k



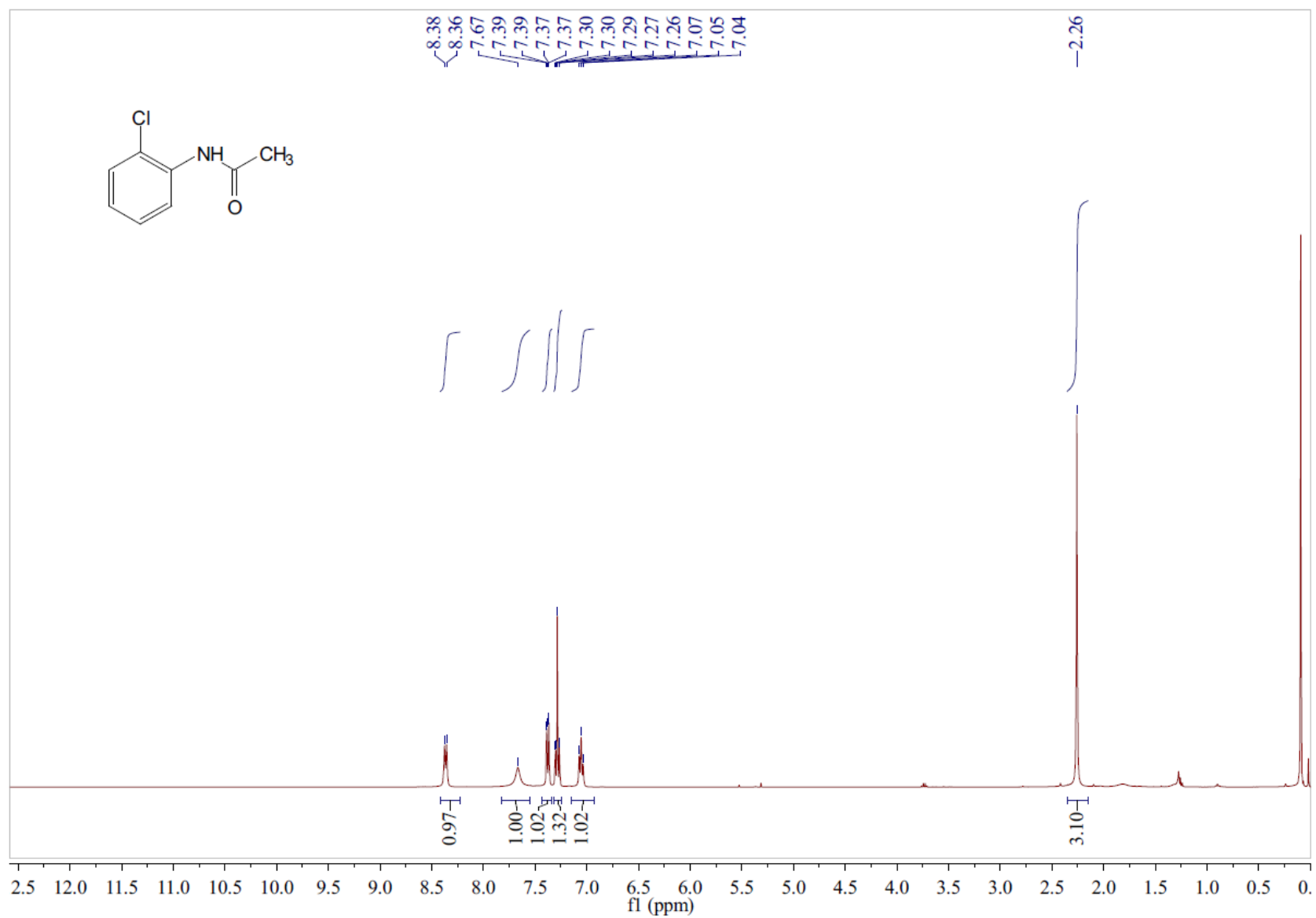
¹H NMR Spectra of 4l



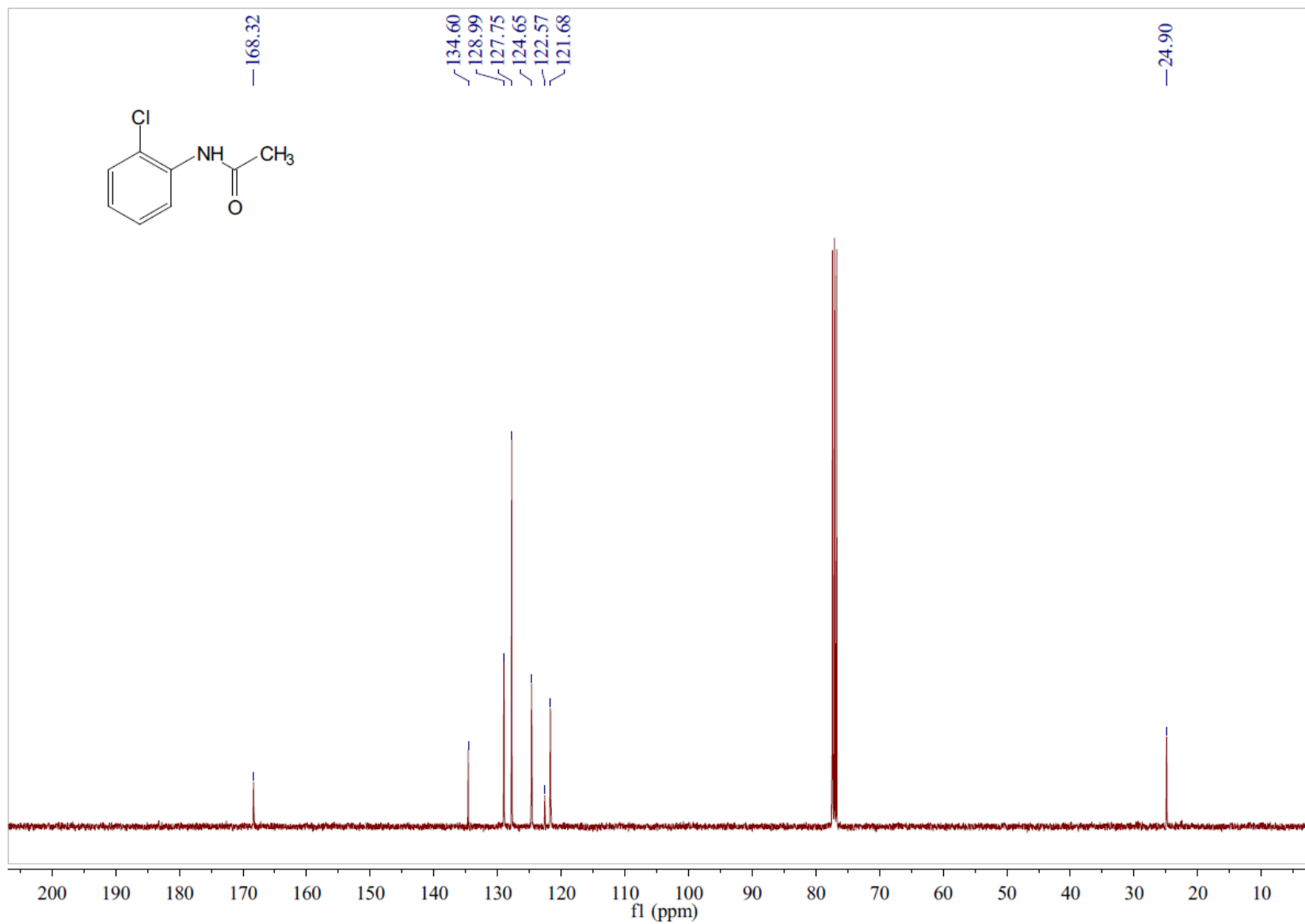
¹³C NMR Spectra of 4l



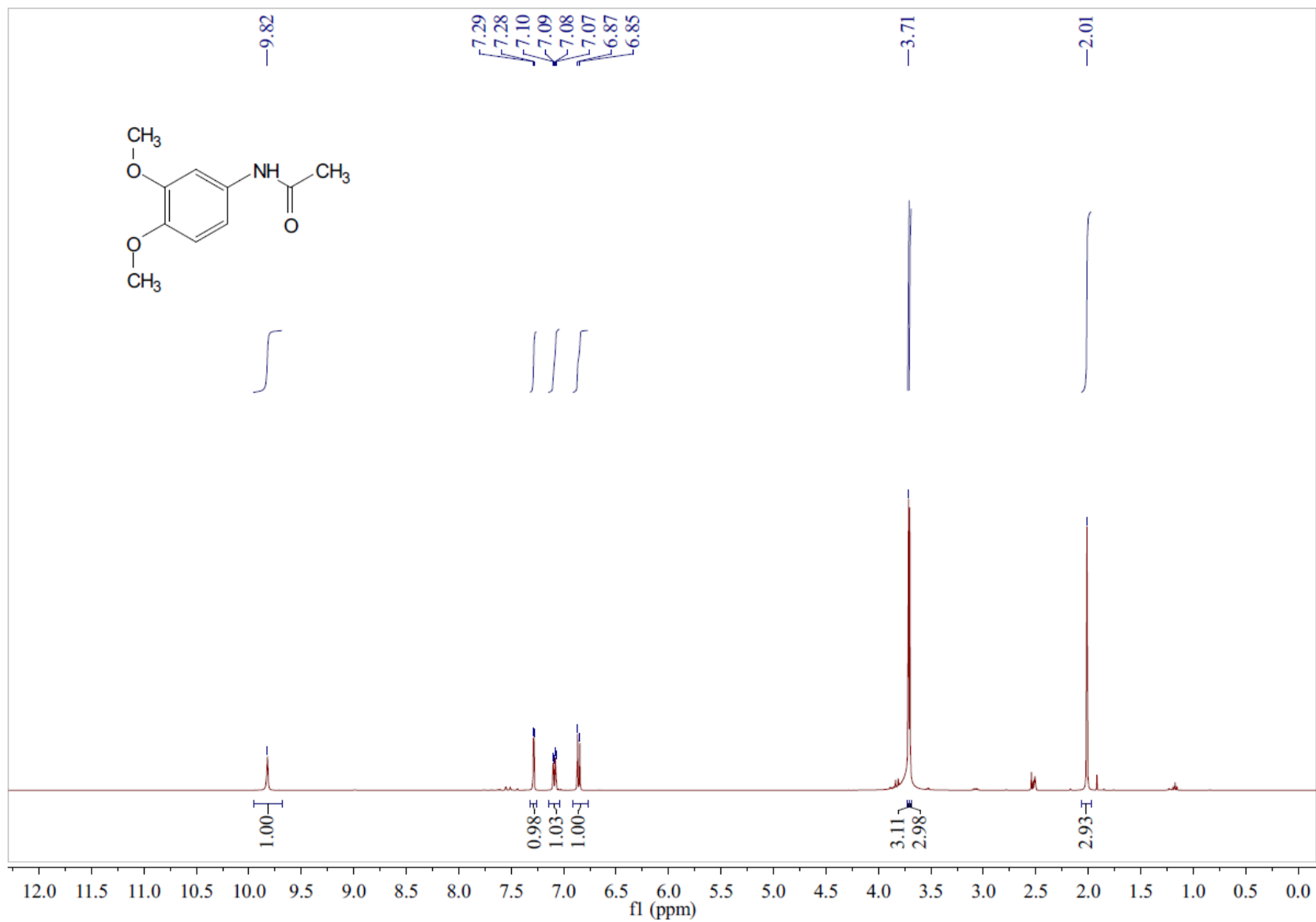
¹H NMR Spectra of 4m



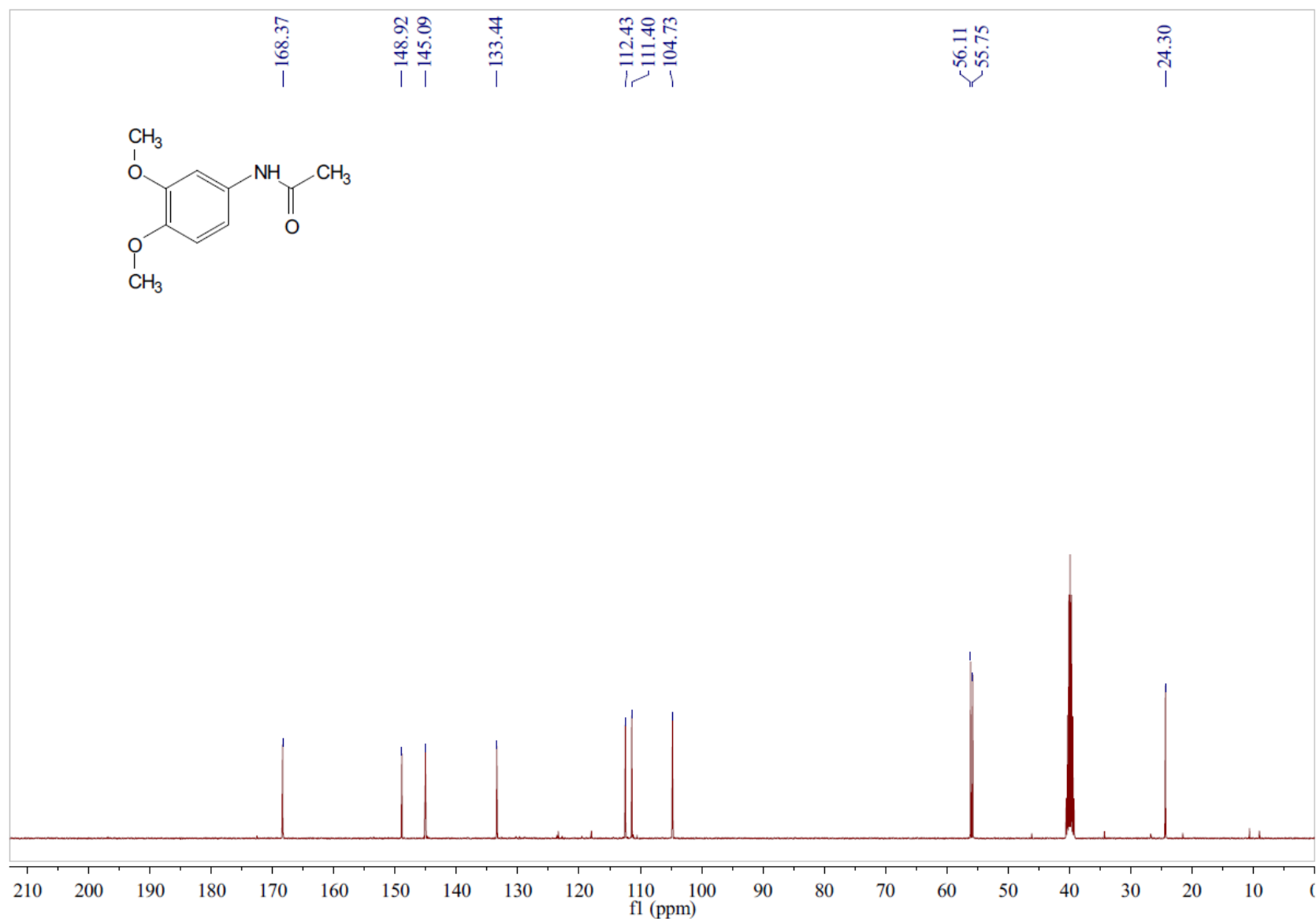
¹³C NMR Spectra of 4m



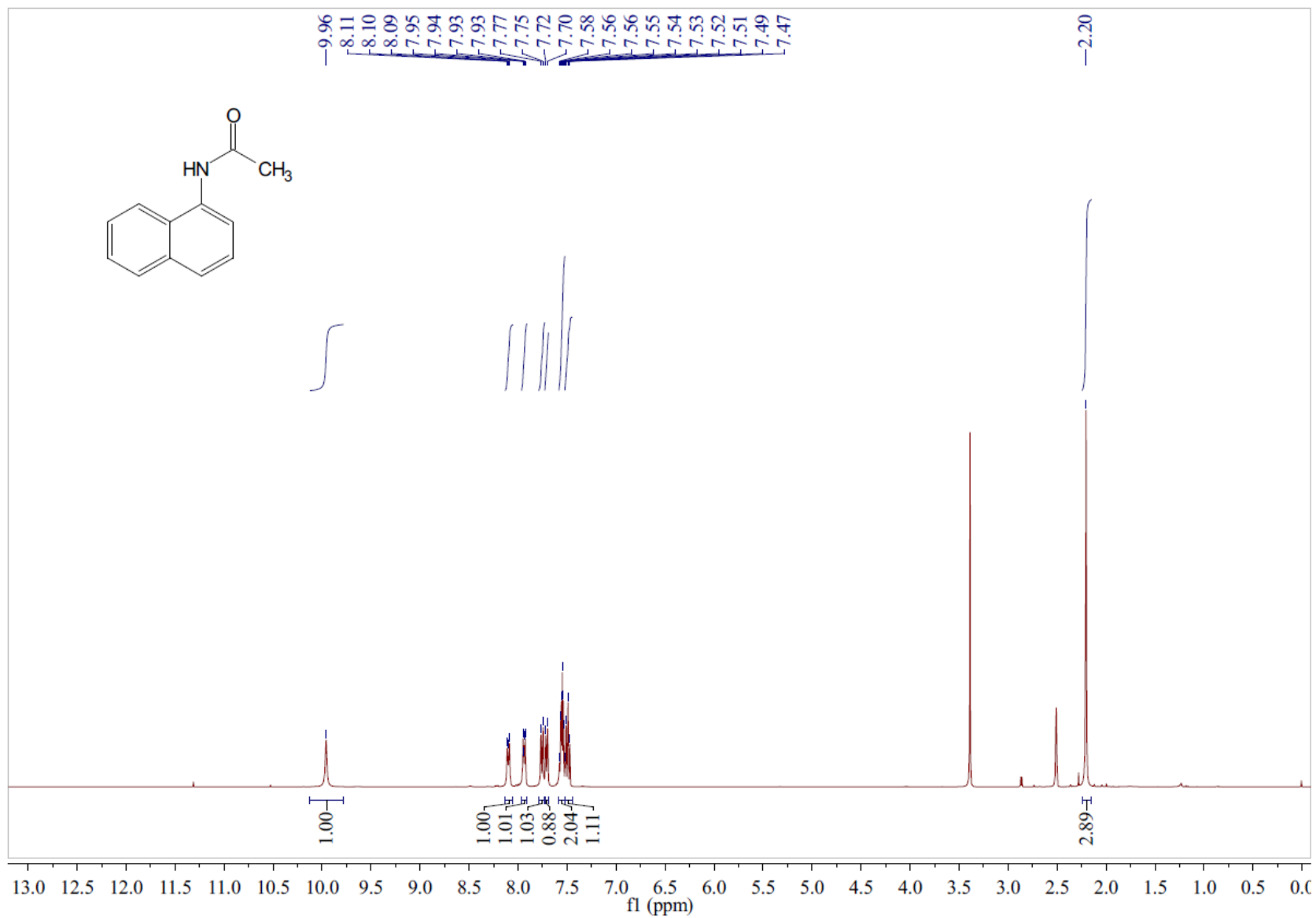
¹H NMR Spectra of 4n



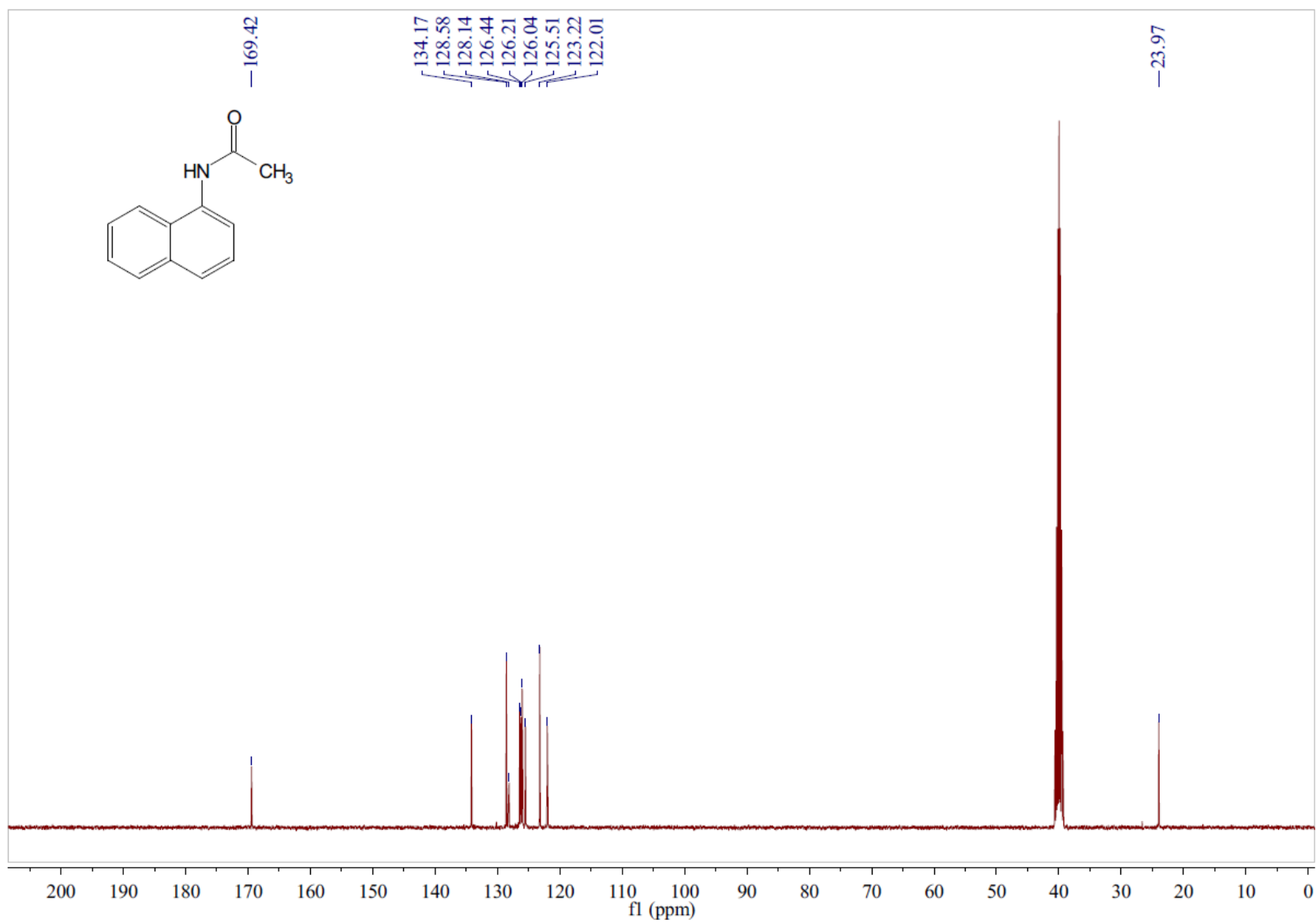
¹³C NMR Spectra of 4n



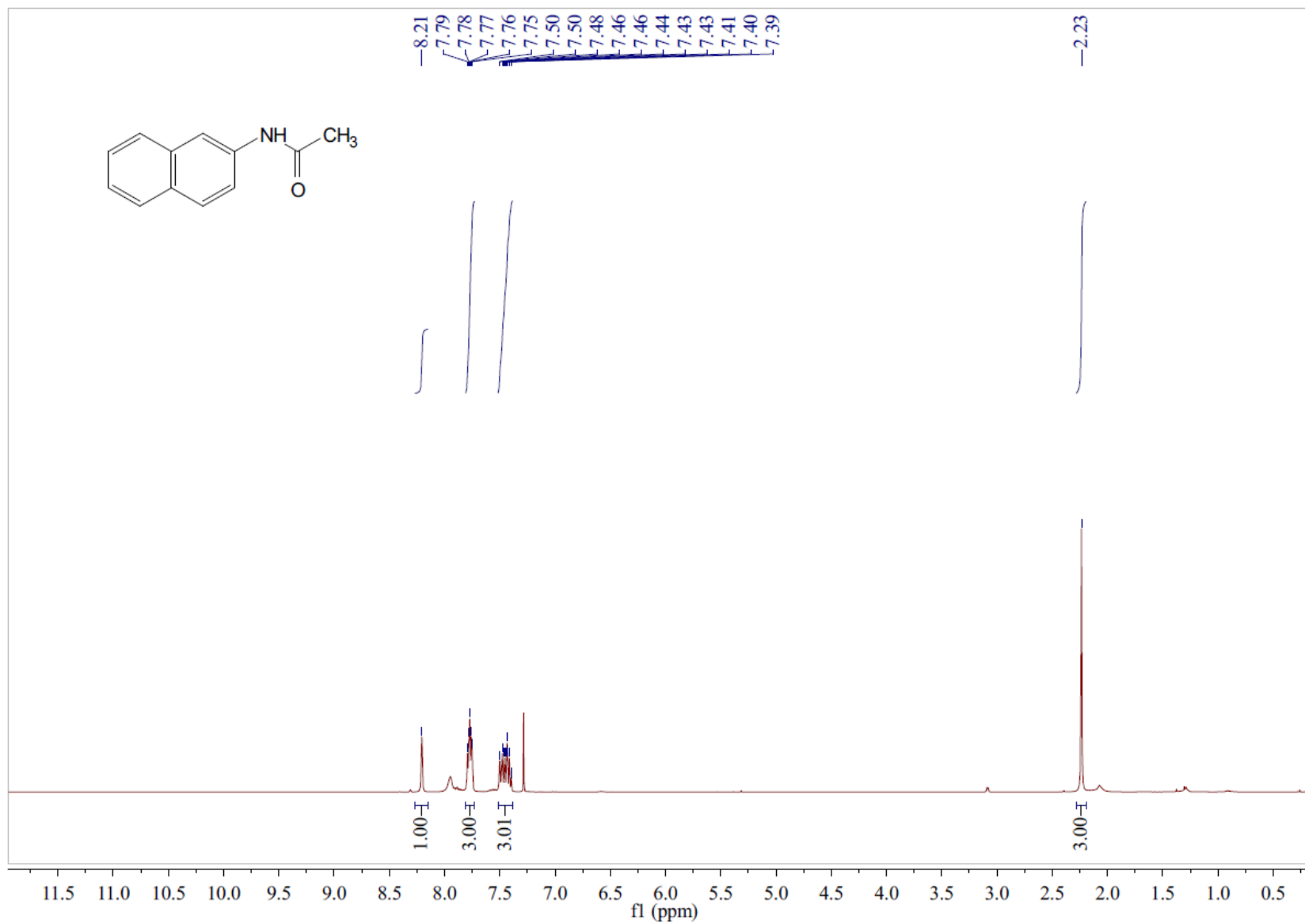
¹H NMR Spectra of 4o



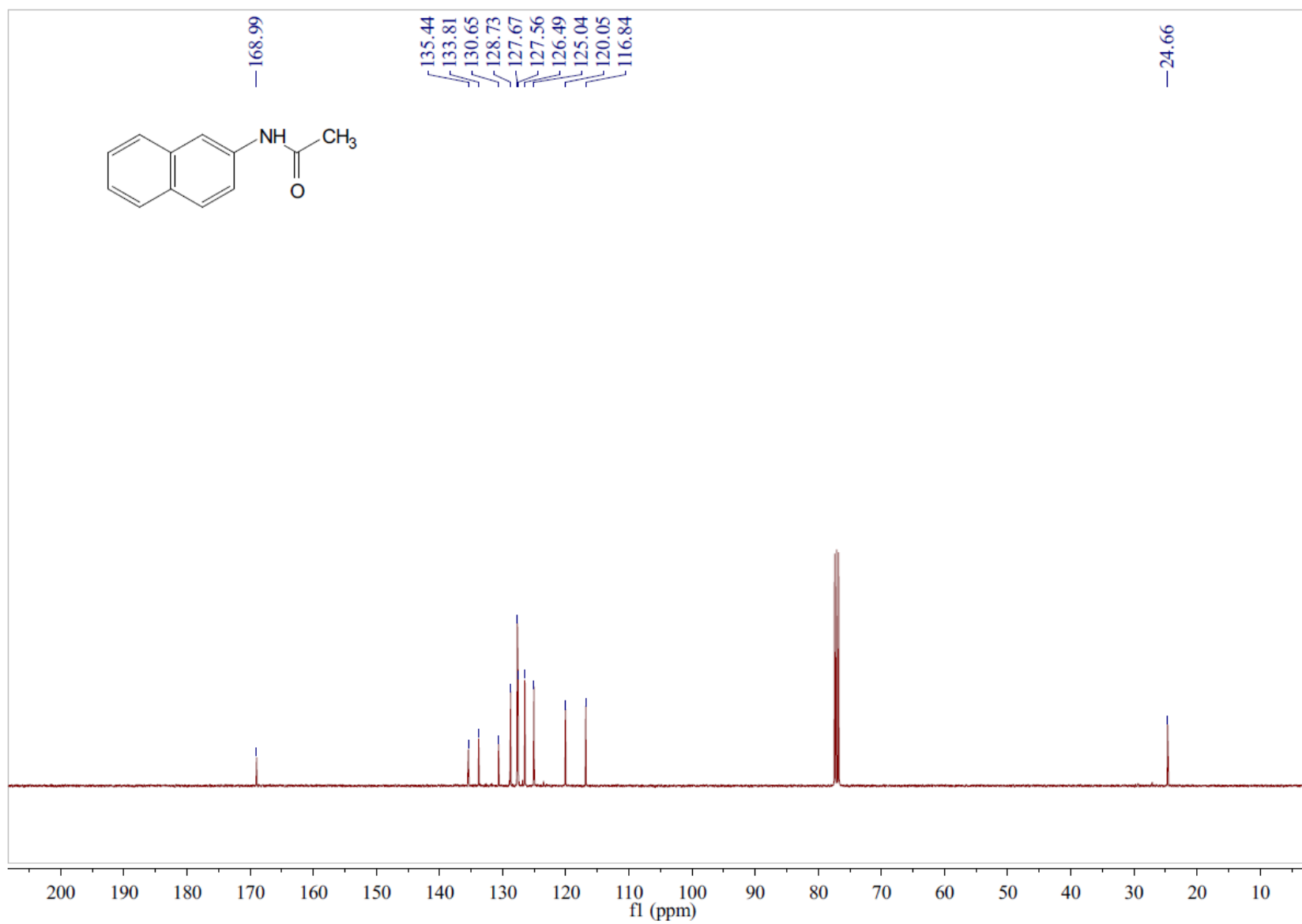
¹³C NMR Spectra of 4o



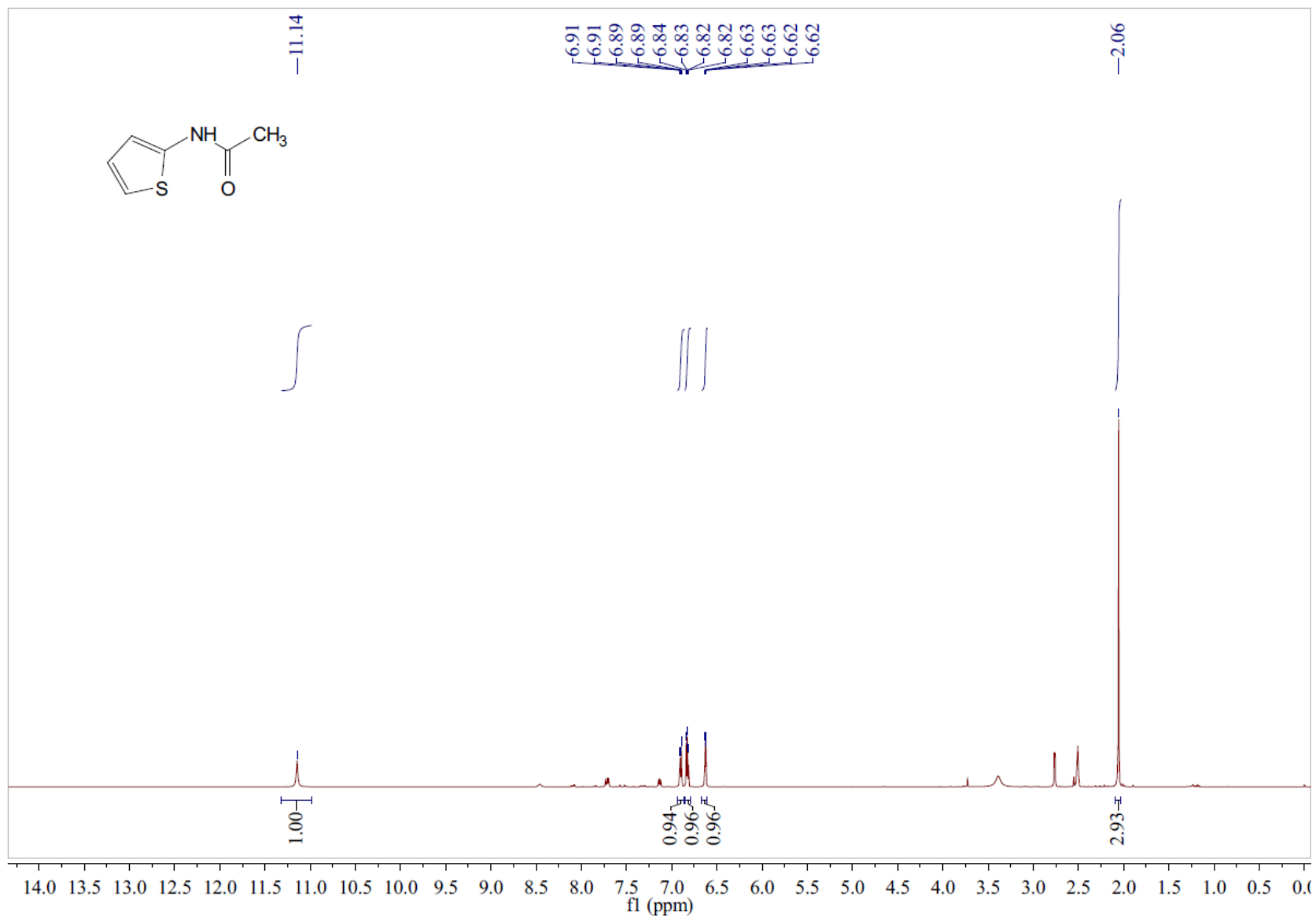
¹H NMR Spectra of 4p



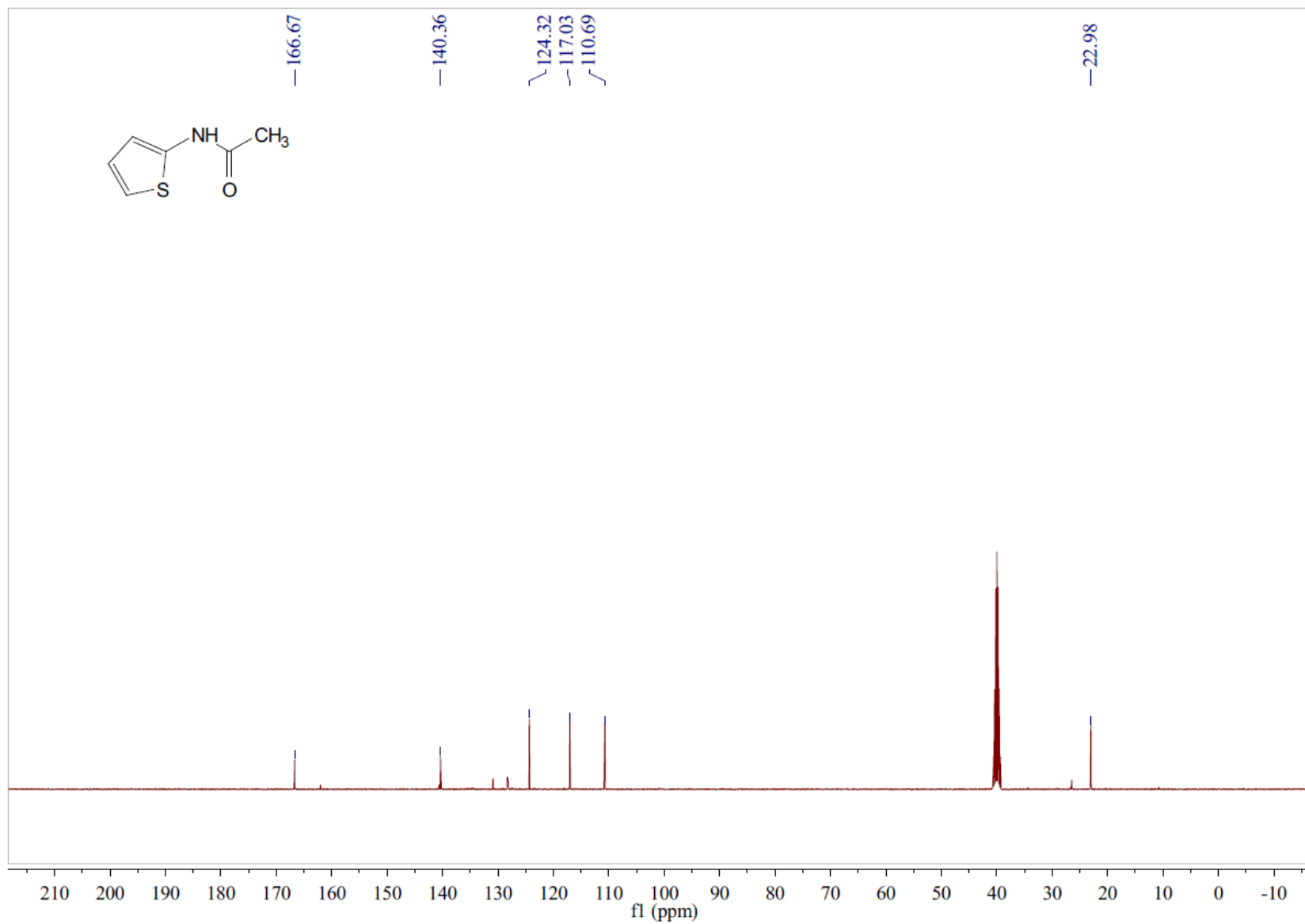
¹³C NMR Spectra of 4p



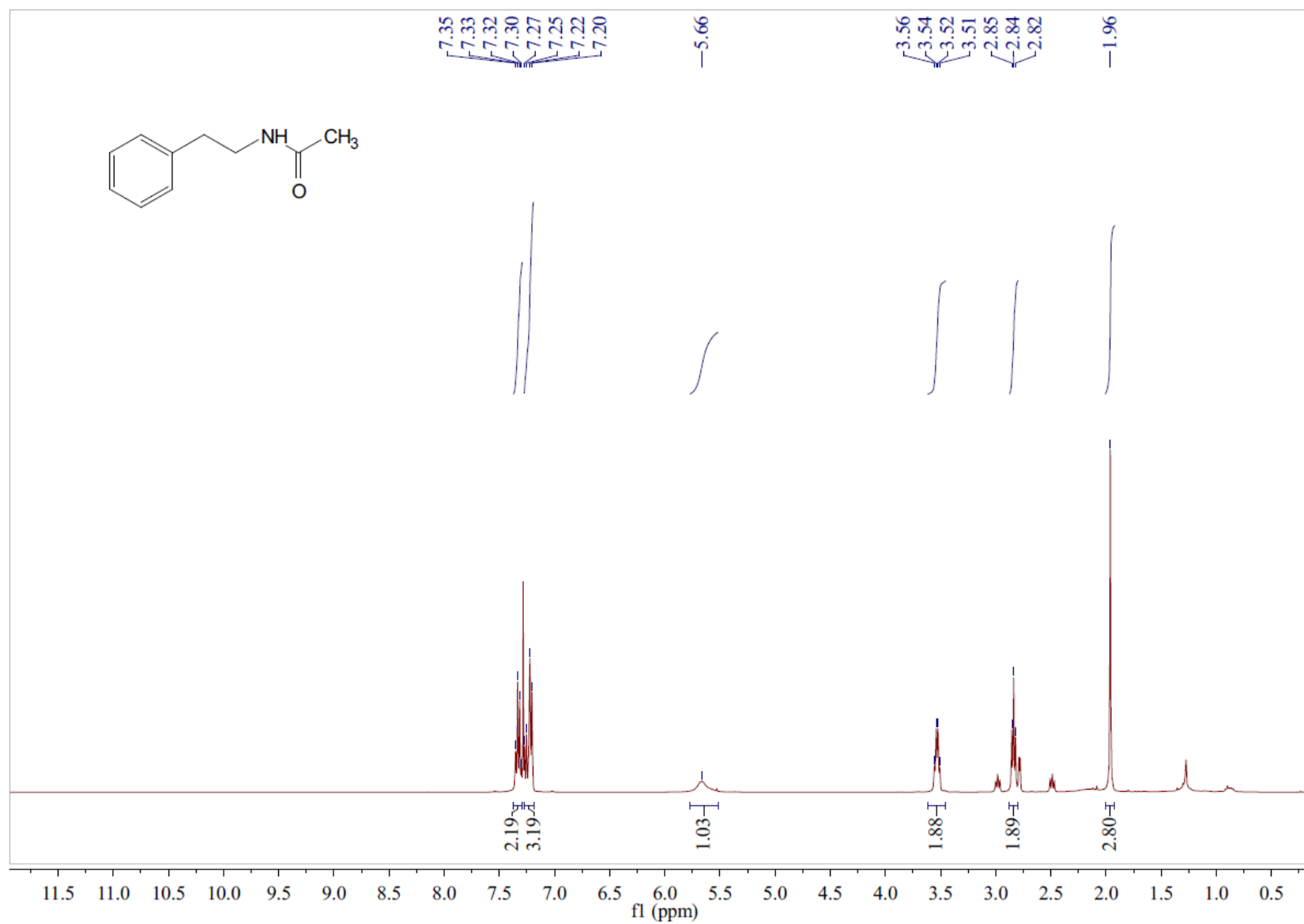
¹H NMR Spectra of 4q



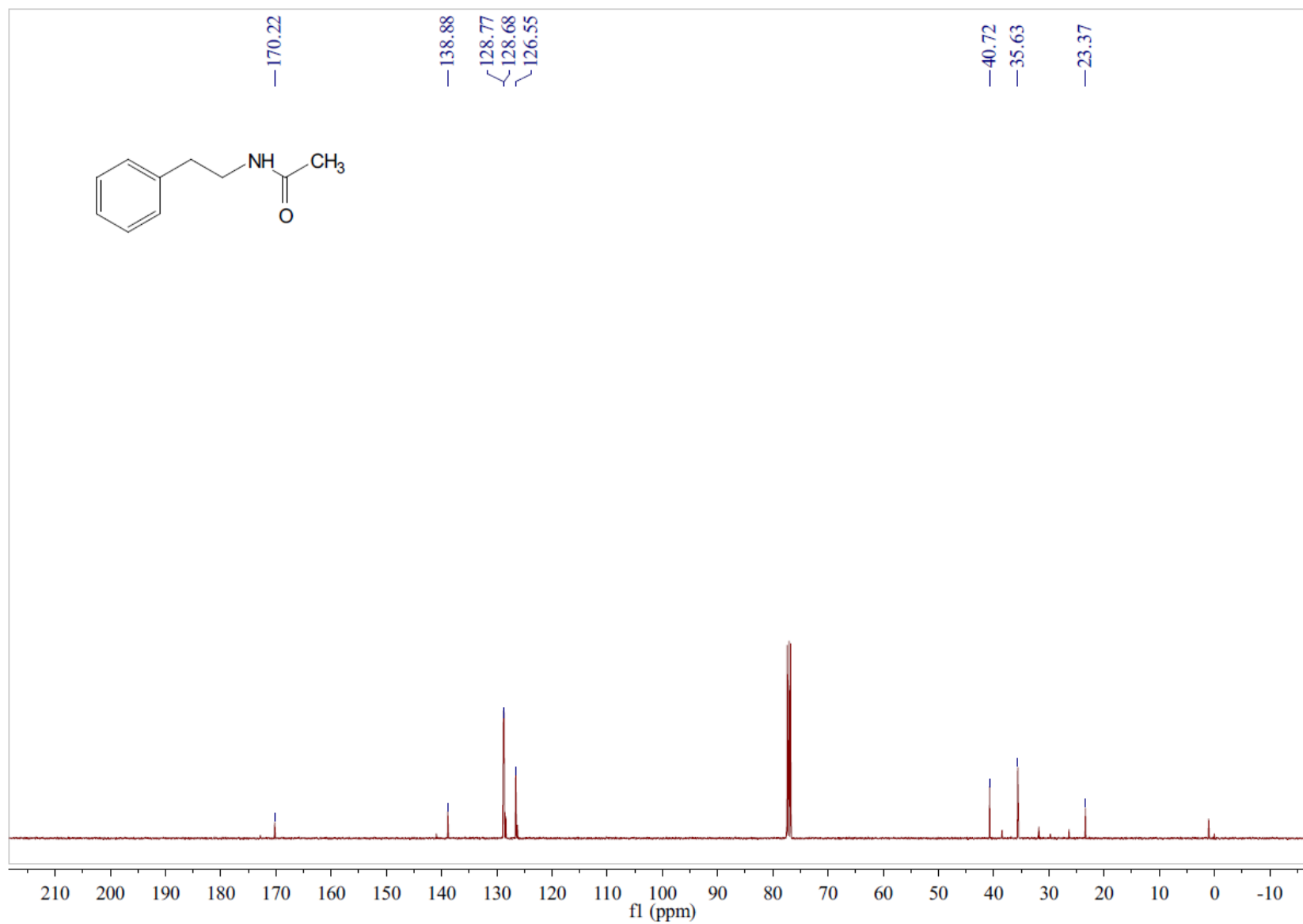
¹³C NMR Spectra of 4q



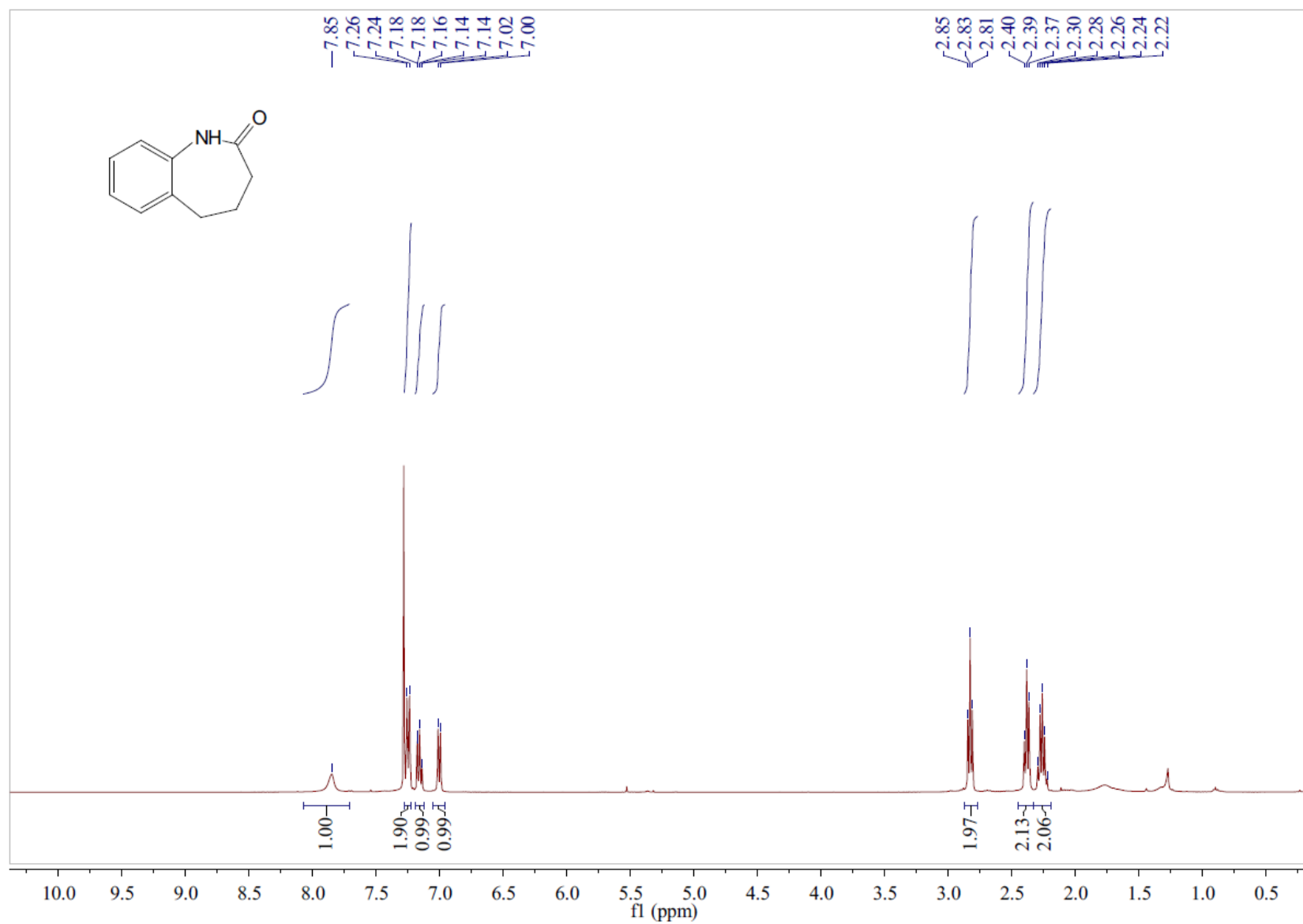
¹H NMR Spectra of 4r



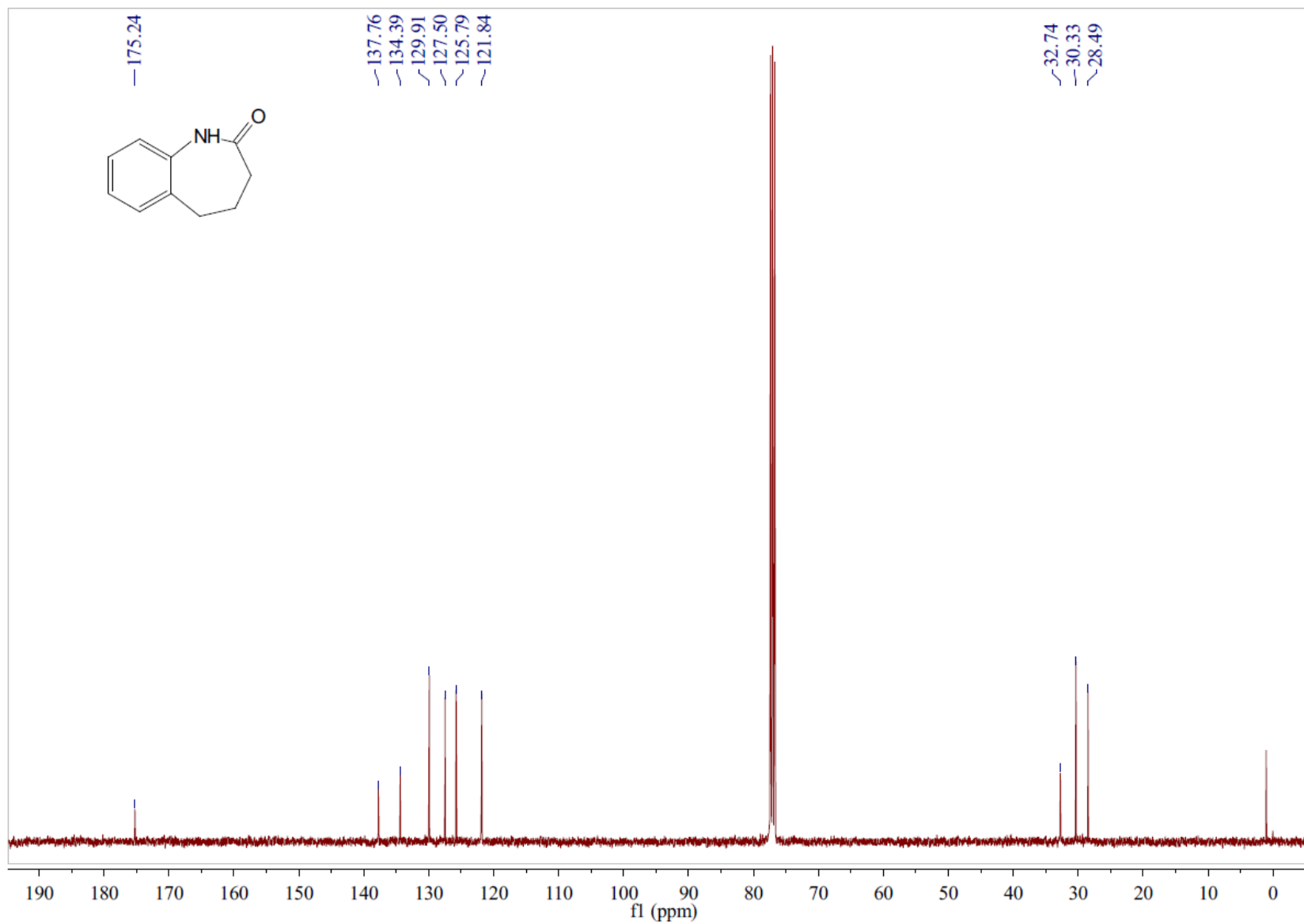
¹³C NMR Spectra of 4r



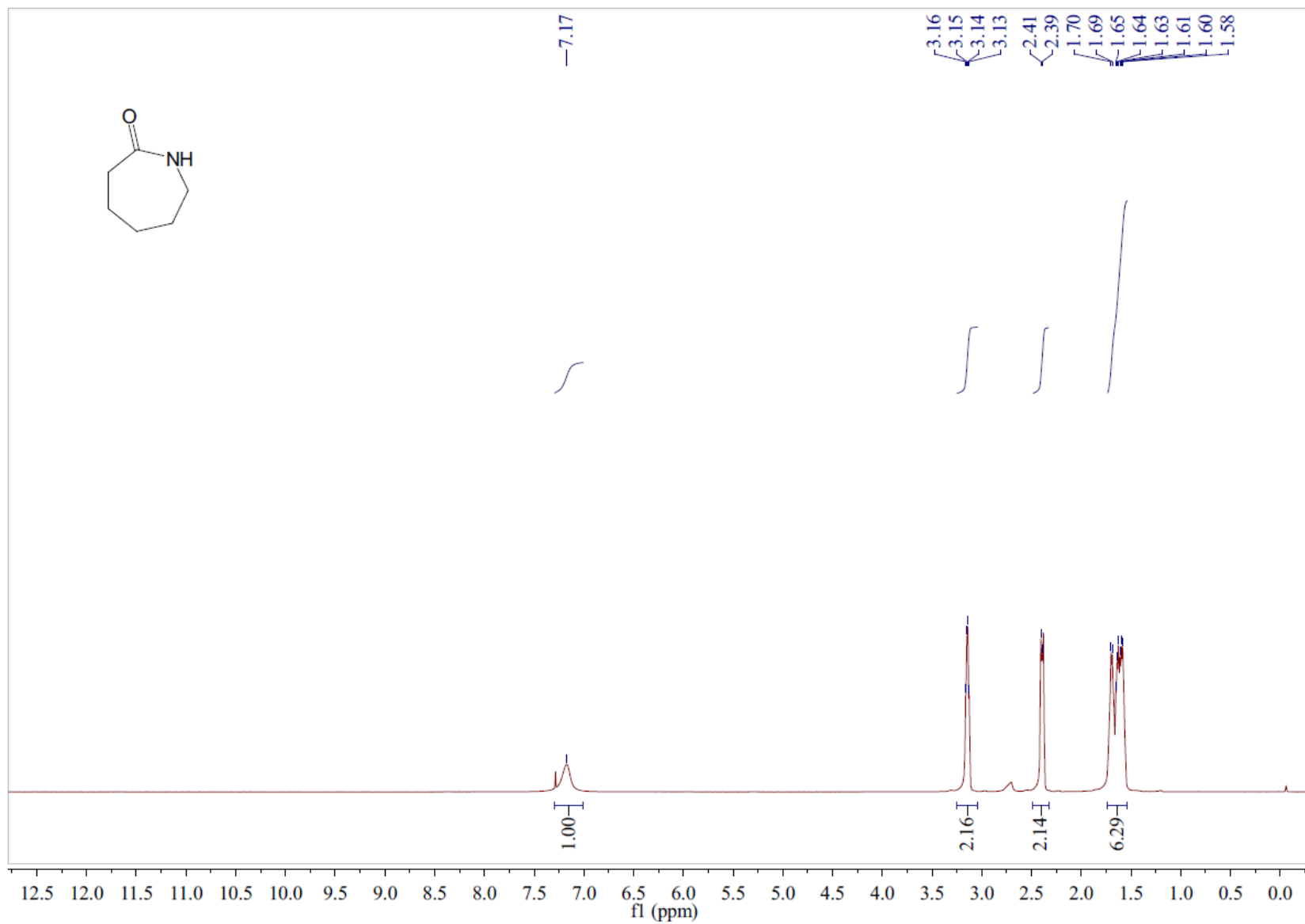
¹H NMR Spectra of 4s



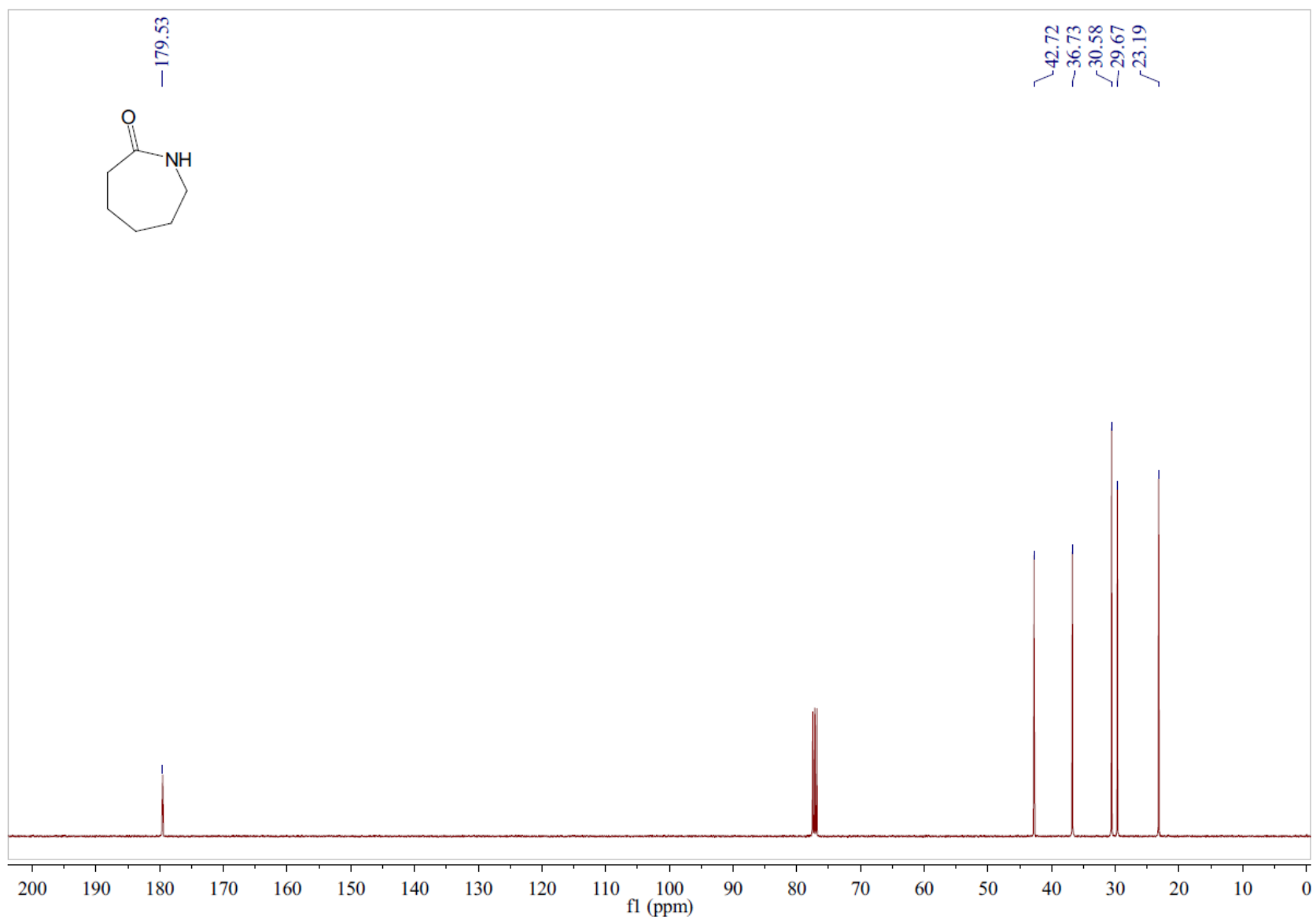
¹³C NMR Spectra of 4s



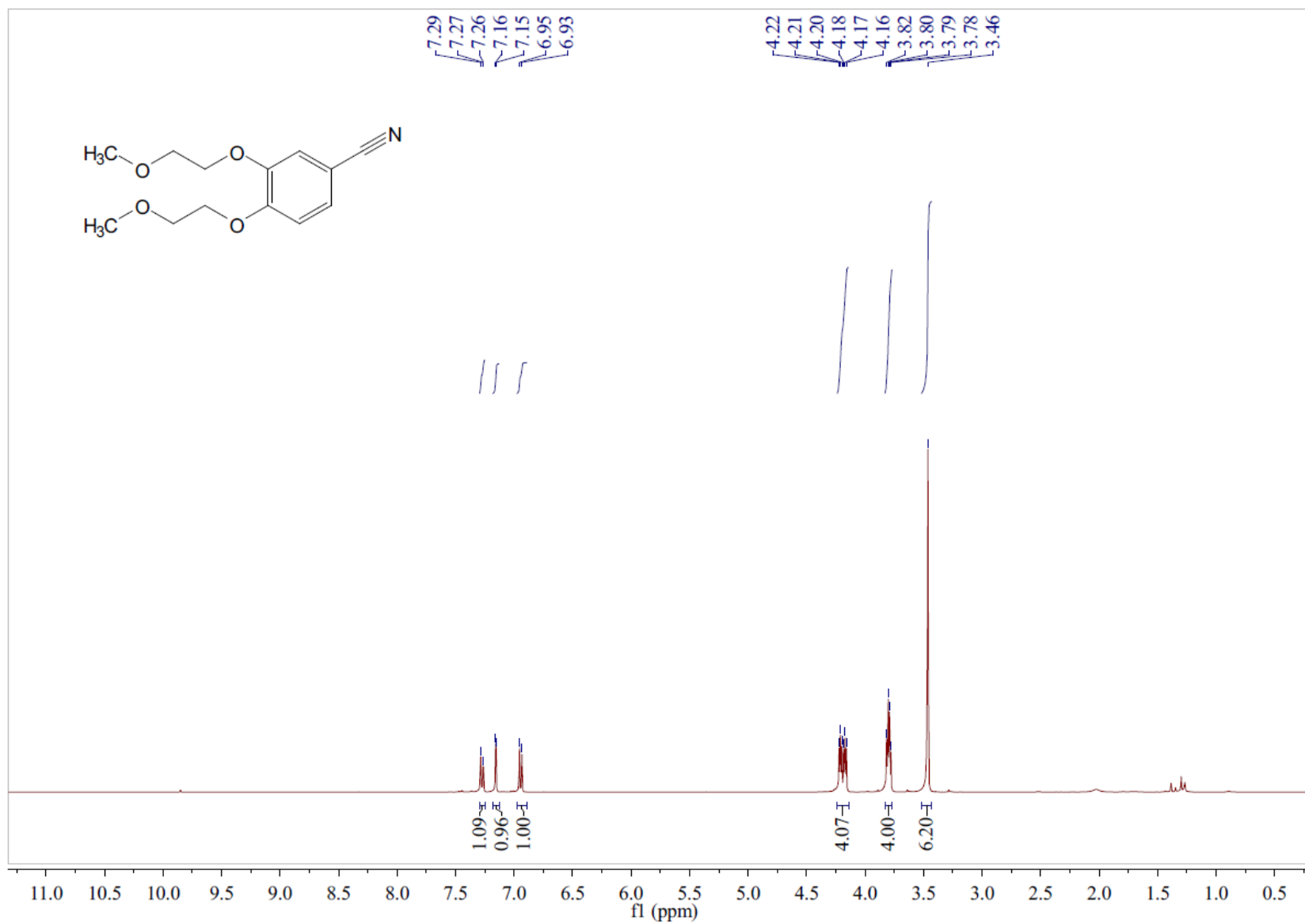
¹H NMR Spectra of 4t



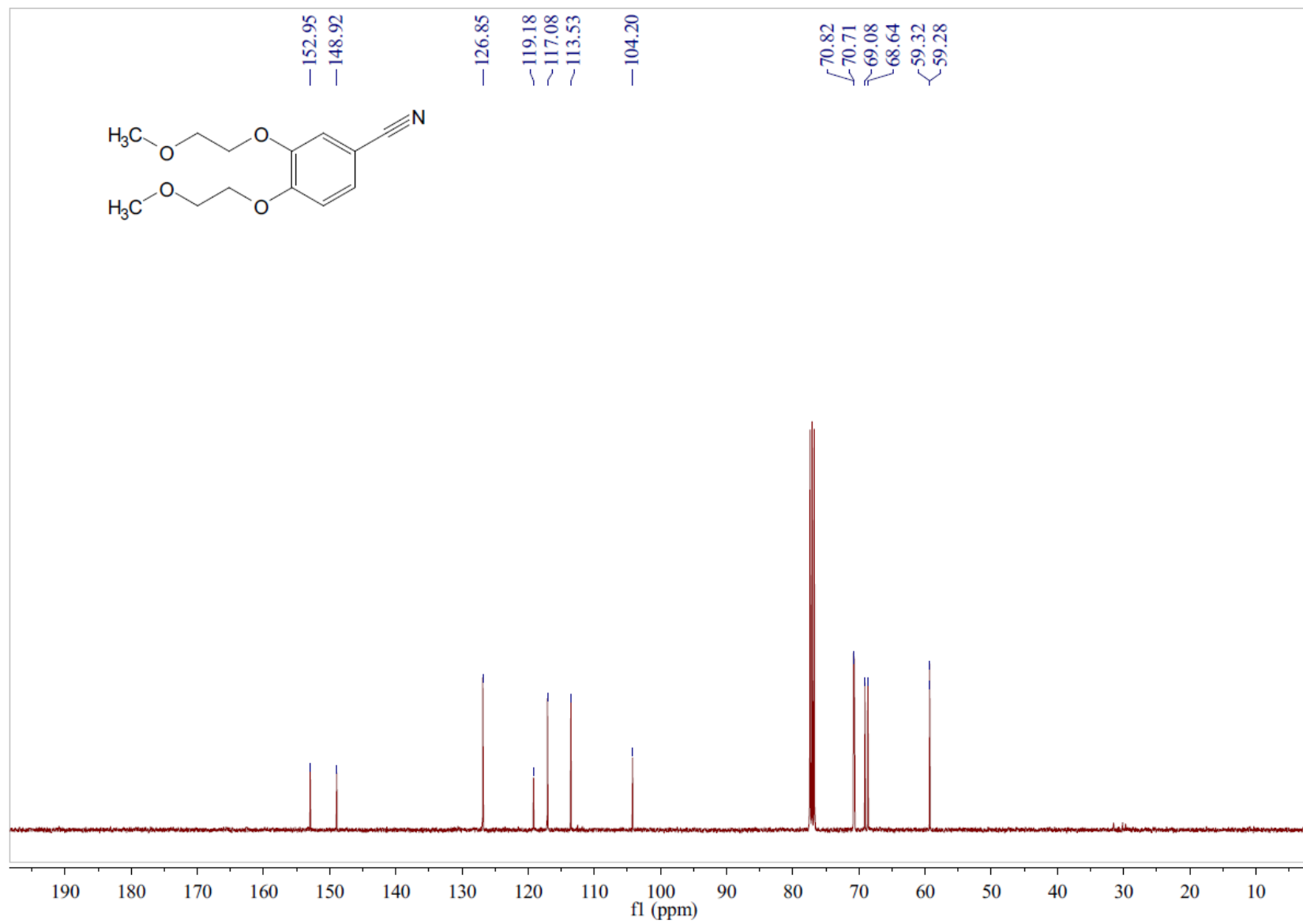
¹³C NMR Spectra of 4t



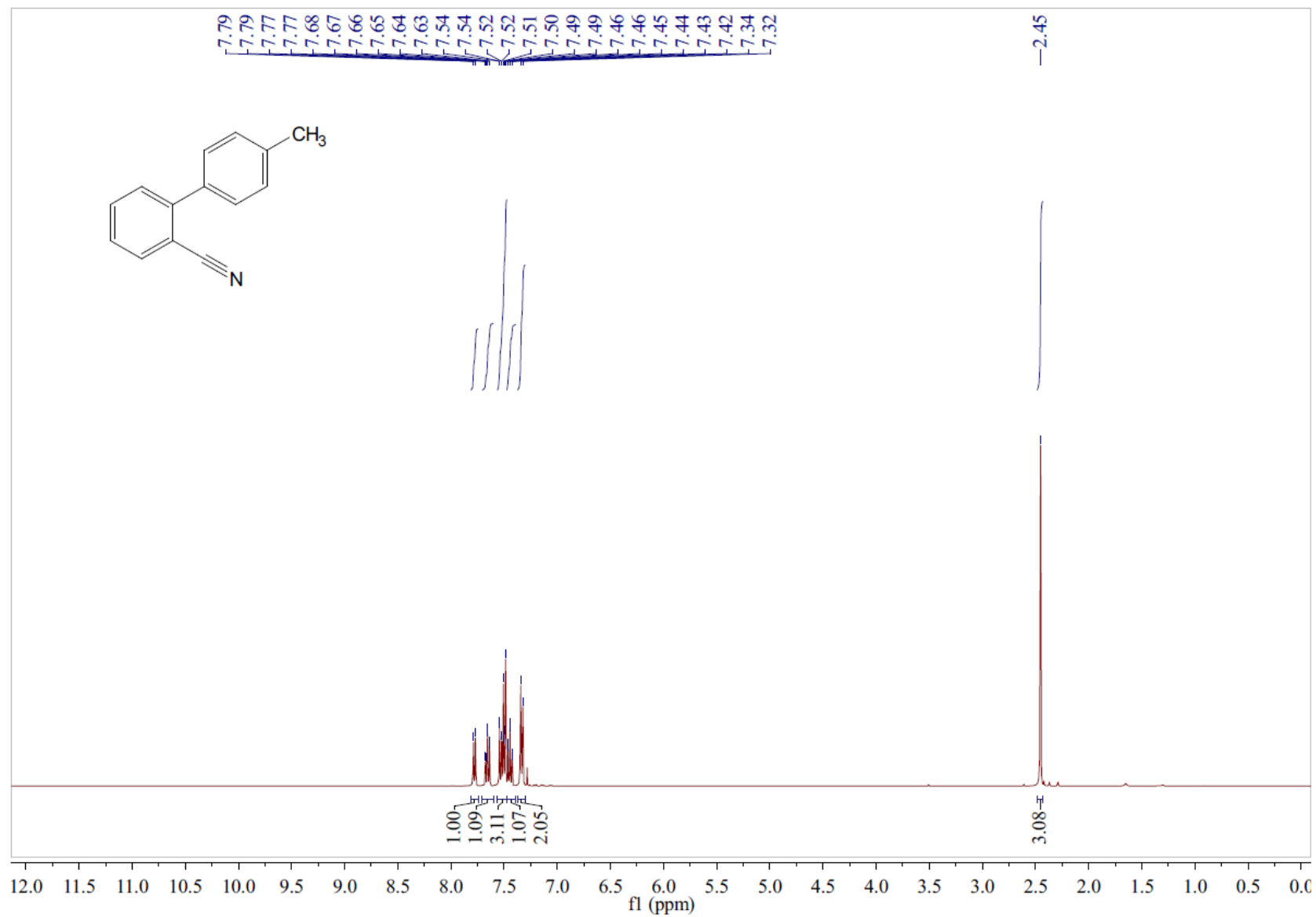
¹H NMR Spectra of E



¹³C NMR Spectra of E



¹H NMR Spectra of G



¹³C NMR Spectra of G

