Supporting Information

Redox-Neutral *Ortho*-C–H Amination of Pinacol Arylborates via Palladium(II)/Norbornene Catalysis for Anilines Synthesis

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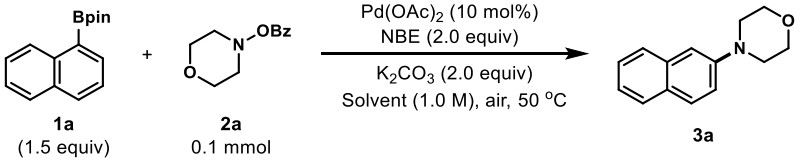
1. General information.

All reactions dealing with moisture-sensitive compounds were performed by standard Schlenk techniques in oven-dried reaction vessels under air atmosphere. Unless otherwise noted, all solvents were dried by JC Meyer Solvent Drying System. Most reagents were purchased from commercial sources and used without further purification, unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC) carried out on 0.2 mm commercial silica gel plates, using UV light as the visualizing agent or basic solution of $KMnO_4$ as a developing agent. ^{1}H NMR spectra were recorded on Bruker 400 MHz instrument and ^{13}C NMR spectra were recorded on Bruker 100 MHz instrument and were calibrated using residual undeuterated solvent as an internal reference ($CDCl_3$ @ 7.26 ppm ^{1}H NMR, 77.16 ppm ^{13}C NMR). The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, ddd = doublet of doublet of doublets, m = multiplet, br = broad. IR spectra were obtained on a Thermo Scientific Spectrum Nicolet iS10 spectrometer using thin films deposited on KBr plates (Thermo Fisher Scientific) and were reported in frequency of absorption (cm^{-1}). Gas chromatography (GC) were recorded on Agilent 7890 instrument and the biphenyl as internal standard. High resolution mass spectra (HRMS) were recorded on DIONEX UltiMate 3000 & Bruker Compact TOF mass spectrometer.

2. Optimization of reaction conditions.

Table S1. Solvent screening.[a]

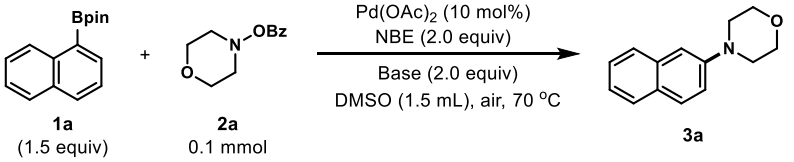


1a (1.5 equiv) + **2a** 0.1 mmol → **3a**

Entry	Solvent	Yield
1	DMA	14%
2	DMF	15%
3	CH_3CN	9%
4	Dioxane	1%
5	Toluene	1%
6	DCE	n.d.
7	DMSO	35%

[a] GC yield, using biphenyl as internal standard.

Table S2. Base screening.[a]

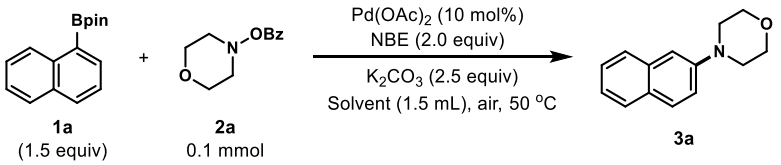


1a (1.5 equiv) + **2a** 0.1 mmol → **3a**

Entry	Base	Yield
1	K_2CO_3	53%
2	Na_2CO_3	33%
3	Cs_2CO_3	17%
4	KOAc	5%
5	$KHCO_3$	58%
6	KOH	67%
7[b]	KOH	70%

[a] GC yield, using biphenyl as internal standard; [b] KOH (2.5 equiv).

Table S3. Co-solvent screening.[a]



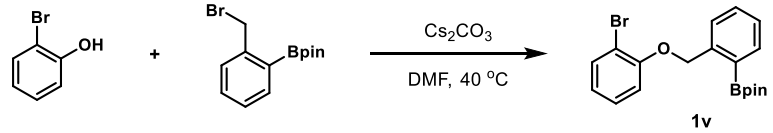
1a (1.5 equiv) **2a** 0.1 mmol **3a**

Entry	Solvent / (v:v)	Yield
1[b]	DMSO:Dioxane (2:5)	70%
2	DMSO:Dioxane (2:5)	82%
3	DMSO:Toluene (2:5)	77%
4	DMSO:THF (2:5)	65%
5	DMSO:DMF (2:5)	68%
6	DMSO:CH_3CN (2:5)	76%
7	DMSO:Dioxane (1:2)	45%
8	DMSO:Dioxane (1:4)	75%

[a] GC yield, using biphenyl as internal standard; [b] KOH instead of K_2CO_3.

3. Preparation of arylboronic esters.

3.1. Preparation of arylboronic ester 1v.



1v

To a 25 mL of oven-dried Schlenk tube equipped with a magnetic stir bar was charged with 2-bromophenol (778.0 mg, 4.5 mmol, 1.5 equiv), 2-(2-(bromomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (888.0 mg, 3.0 mmol, 1.0 equiv), cesium carbonate (1.9 g, 6.0 mmol, 2.0 equiv) and DMF (10.0 mL) at r.t.. The reaction was heated at 40 °C and stirred for 5 h. Then the mixture was diluted with ethyl acetate and sequentially washed with water, brine. The combined organic layers were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (PE:EtOAc = 50:1) to yield the desired product **1v** (673 mg, 59% yield).

Physical state: colorless oil;

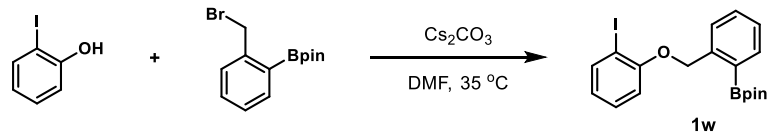
R_f = 0.5 (silica gel, PE:EtOAc = 50:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.4 Hz, 1H), 7.66 (d, *J* = 7.7 Hz, 1H), 7.55 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.26 – 7.20 (m, 1H), 6.97 (d, *J* = 8.2 Hz, 1H), 6.83 (t, *J* = 7.6 Hz, 1H), 5.45 (s, 2H), 1.32 (s, 12H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5, 143.2, 136.2, 133.4, 131.1, 128.5, 127.06, 127.05, 121.2, 113.7, 112.4, 83. 9, 70.3, 25.0;

HRMS (ESI-TOF): calc'd for $C_{19}H_{22}BBrNaO_3^+$ [M+Na^+] 410.0773, found 410.0772.

3.2. Preparation of arylboronic ester 1w.



1w

To a 25 mL of oven-dried Schlenk tube equipped with a magnetic stir bar was charged with 2-iodophenol (330.0 mg, 1.5 mmol, 1.5 equiv), 2-(2-(bromomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (296.0 mg, 1.0 mmol, 1.0 equiv), cesium carbonate (651.5 mg, 2.0 mmol, 2.0 equiv) and DMF (4.0 mL) at r.t.. The reaction was heated at 35 °C and stirred for 3 h. Then the mixture was diluted with ethyl acetate and sequentially washed with water, brine. The combined organic layers were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (PE:EtOAc = 50:1) to yield the desired product **1w** (300 mg, 69% yield).

Physical state: colorless oil;

R_f = 0.5 (silica gel, PE:EtOAc = 50:1);

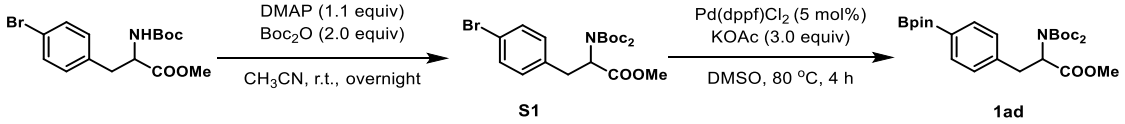
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 – 7.87 (m, 1H), 7.80 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.72 (d, *J* = 7.7 Hz, 1H), 7.52 – 7.48 (m, 1H), 7.32 – 7.27 (m, 2H), 6.90 (dd, *J* = 8.3, 1.2 Hz, 1H), 6.73 – 6.69 (m, 1H), 5.46 (s, 2H), 1.33 (s, 12H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 157.6, 143.2, 139.5, 136.1, 131.5, 129.5, 127.1, 127.0, 122.4, 112.6, 86.8, 83.9, 70.4, 25.1;

HRMS (ESI-TOF): calc'd for $C_{19}H_{22}BNaO_3^+$ [M+Na^+] 459.0599, found 459.0603.

Both the proton and carbon NMR match the literature reported data.[1]

3.3. Preparation of arylboronic ester 1ad.



To a solution of methyl 3-(4-bromophenyl)-2-((*tert*-butoxycarbonyl)amino)propanoate (1.07 g, 3.0 mmol, 1.0 equiv) in CH_3CN (10 mL) were added DMAP (403 mg, 3.3 mmol, 1.1 equiv) and Boc_2O (1.4 mL, 6.0 mmol, 2.0 equiv) at r.t., and the mixture was stirred overnight. Then the reaction mixture was transferred to a separatory funnel containing saturated aqueous solution of NH_4Cl. The aqueous portion was extracted with ethyl acetate (3 x 20 mL) and the combined organic layers were washed with brine (30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo* to yield the crude product **S1**(1.41 g) used in the next step straightly.

To a 50 mL of oven-dried Schlenk tube equipped with a magnetic stir bar was charged with **S1** (457 mg, 1.0 mmol, 1.0 equiv), Bis(pinacolato)diboron (280 mg, 1.1 mmol, 1.1 equiv), $PdCl_2$(dppf) (36 mg, 0.05 mmol, 0.05 equiv), KOAc (290 mg, 3.0 mmol, 3.0 equiv) and DMSO (6 mL) under Ar atmosphere, then heated at 80 ºC and stirred for 4 h. Then the reaction mixture was extracted with ethyl acetate (3 x 20 mL) and the combined organic layers were washed with brine (30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo.* The residue was purified by column chromatography on silica gel (PE:EtOAc = 10:1) to yield the desired product **1ad** (460 mg, 93% yield for two steps).

Physical state: colorless oil;

R_f = 0.4 (silica gel, PE:EtOAc = 10:1);

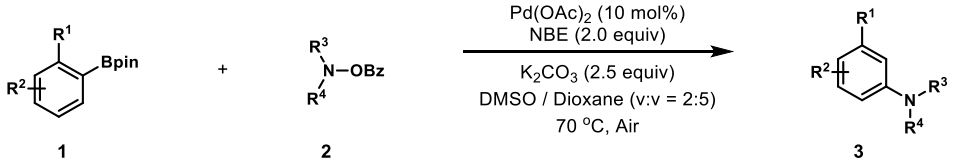
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 5.15 (dd, *J* = 10.1, 5.2 Hz, 1H), 3.73 (s, 3H), 3.44 (dd, *J* = 13.9, 5.2 Hz, 1H), 3.22 (dd, *J* = 13.9, 10.1 Hz, 1H), 1.38 (s, 18H), 1.32 (s, 12H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.0, 151.8, 141.1, 135.0, 129.1, 83.8, 83.1, 59.4, 52.4, 36.5, 28.0, 25.2, 24.9;

IR (KBr pellet): *v* 2979, 2934, 1748, 1701, 1366, 1267, 1142, 1089, 859, 658 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{26}H_{40}BNNaO_8^+$ [M+Na^+] 528.2744, found 528.2754.

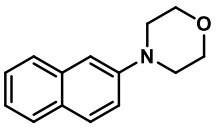
4. General procedure for the synthesis of compounds 3.



To a 25 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with the corresponding pinacol arylborate **1** (0.3 mmol, 1.5 equiv), *O*-benzoyl hydroxylamines **2** (0.2 mmol, 1.0 equiv), $Pd(OAc)_2$ (4.5 mg, 0.02 mmol, 0.1 equiv), K_2CO_3 (69.0 mg, 0.5 mmol, 2.5 equiv), NBE (37.6 mg, 0.4 mmol, 2.0 equiv) and DMSO / Dioxane (2.8 mL, v:v = 2:5). The reaction was stirred at 70 ºC under air (ballon) and monitored by TLC. After completion of the reaction, the mixture was cooled to r.t., then filtered through a thin pad of celite, eluting with ethyl acetate (15 mL). The filtrate was sequentially washed with water, brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel or by PTLC to yield the title product 3.

5. Characterization data for compounds 3.

4-(Naphthalen-2-yl)morpholine (3a, CAS: 7508-21-6)



Physical state: yellow solid;

Yield: 84%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

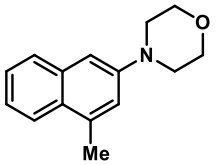
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.70 (m, 3H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.13 (s, 1H), 3.94 – 3.91 (m, 4H), 3.28 – 3.26 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.2, 134.6, 129.0, 128.8, 127.6, 126.9, 126.5, 123.7, 119.1, 110.2, 67.1, 49.9;

HRMS (ESI-TOF): calc'd for $C_{14}H_{16}NO^+$ [M+H$^+$] 214.1226, found 214.1228.

Both the proton and carbon NMR match the literature reported data.[2]

4-(Naphthalen-2-yl)morpholine (3b, CAS: 1174508-56-5)



Physical state: colorless oil;

Yield: 68%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

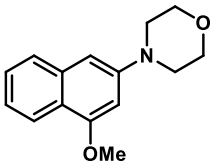
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.3 Hz, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.14 (s, 1H), 7.02 (s, 1H), 3.94 – 3.92 (m, 4H), 3.28 – 3.26 (m, 4H), 2.69 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 148.8, 135.4, 134.9, 128.2, 127.5, 126.2, 124.0, 123.5, 119.8, 108.7, 67.1, 49.9, 19.8;

HRMS (ESI-TOF): calc'd for $C_{15}H_{18}NO^+$ [M+H$^+$] 228.1383, found 228.1382.

Both the proton and carbon NMR match the literature reported data.[3]

4-(4-Methoxynaphthalen-2-yl)morpholine (3c)



Physical state: brown oil;

Yield: 62%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

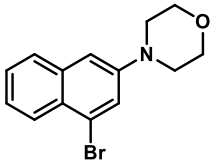
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (d, *J* = 8.3 Hz, 1H), 7.67 (d, *J* = 8.2 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 6.75 (s, 1H), 6.60 (s, 1H), 4.00 (s, 3H), 3.94 – 3.92 (m, 4H), 3.27 – 3.25 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 149.8, 135.3, 127.1, 126.6, 123.1, 121.9, 121.7, 103.2, 98.0, 67.1, 55.5, 50.4;

IR (KBr pellet): ν 3446, 2957, 2359, 1627, 1446, 1409, 1269, 1109, 985, 746 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{15}H_{18}NO_2^+$ [M+H$^+$] 244.1332, found 244.1334.

4-(4-Bromonaphthalen-2-yl)morpholine (3d)



Physical state: light yellow oil;

Yield: 68%;

R_f = 0.3 (silica gel, PE:EtOAc = 30:1);

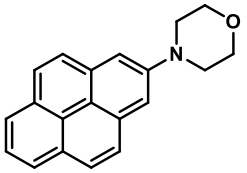
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 (d, *J* = 8.2 Hz, 1H), 7.67 (d, *J* = 7.5 Hz, 1H), 7.58 (d, *J* = 2.4 Hz, 1H), 7.47 – 7.38 (m, 2H), 7.07 (d, *J* = 2.3 Hz, 1H), 3.91 – 3.89 (m, 4H), 3.26 – 3.23 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.2, 135.4, 127.30, 127.28, 127.27, 126.9, 124.9, 123.7, 122.9, 110.2, 66.9, 49.6;

IR (KBr pellet): ν 2960, 2852, 1621, 1595, 1448, 1225, 1121, 983, 942, 763 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{14}H_{15}BrNO^+$ [M+H$^+$] 292.0332, found 292.0332.

4-(Pyren-2-yl)morpholine (3e)



Physical state: yellow solid;

Melting point: 156 – 158 ºC;

Yield: 70%;

R_f = 0.4 (silica gel, PE:EtOAc = 10:1);

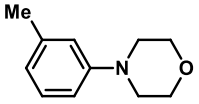
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (d, *J* = 7.6 Hz, 2H), 8.03 – 8.01 (m, 2H), 7.96 – 7.88 (m, 3H), 7.72 (s, 2H), 4.01 – 3.99 (m, 4H), 3.47 – 3.45 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7, 132.4, 130.3, 128.0, 127.1, 125.2, 124.9, 124.8, 119.9, 112.7, 67.2, 50.1;

IR (KBr pellet): *ν* 2960, 2843, 1601, 1448, 1256, 1120, 984, 859, 848, 706 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{20}H_{18}NO^+$ [M+H$^+$] 288.1383, found 288.1385.

4-(*m*-Tolyl)morpholine (3f, CAS:7025-91-4)



Physical state: colorless oil

Yield: 70%;

R_f = 0.5 (silica gel, PE:EtOAc = 20:1);

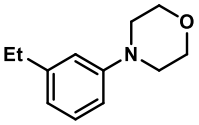
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 – 7.16 (m, 1H), 6.75 – 6.71 (m, 3H), 3.88 – 3.85 (m, 4H), 3.17 – 3.14 (m, 4H), 2.34 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 139.0, 129.2, 121.1, 116.7, 113.0, 67.1, 49.6, 21.9;

HRMS (ESI-TOF): calc'd for $C_{11}H_{16}NO^+$ [M+H$^+$] 178.1226, found 178.1229.

Both the proton and carbon NMR match the literature reported data.[2]

4-(3-Ethylphenyl)morpholine (3g)



Physical state: colorless oil;

Yield: 61%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

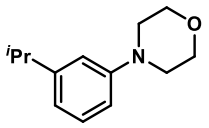
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (t, J = 7.8 Hz, 1H), 6.78 – 6.75 (m, 3H), 3.89 – 3.86 (m, 4H), 3.18 – 3.16 (m, 4H), 2.63 (q, J = 7.6 Hz, 2H), 1.25 (t, J = 7.8 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 145.4, 129.2, 119.9, 115.6, 113.2, 67.1, 49.6, 29.3, 15.8;

HRMS (ESI-TOF): calc'd for $C_{12}H_{18}NO^+$ [M+H$^+$] 192.1383, found 192.1385.

Both the proton and carbon NMR match the literature reported data.[4]

4-(3-Isopropylphenyl)morpholine (3h)



Physical state: colorless oil;

Yield: 44%;

R_f = 0.5 (silica gel, PE:EtOAc = 20:1);

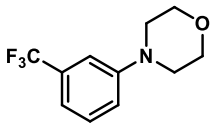
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 (t, *J* = 7.8 Hz, 1H), 6.81 – 6.74 (m, 3H), 3.89 – 3.86 (m, 4H), 3.18 – 3.16 (m, 4H), 2.87 (hept, *J* = 6.9 Hz, 1H), 1.26 (s, 3H), 1.25 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 150.1, 129.2, 118.5, 114.4, 113.3, 67.1, 49.7, 34.6, 24.2;

HRMS (ESI-TOF): calc'd for $C_{13}H_{20}NO^+$ [M+H$^+$] 206.1539, found 206.1543.

Both the proton and carbon NMR match the literature reported data.[4]

4-(3-(Trifluoromethyl)phenyl)morpholine (3i)



Physical state: colorless oil;

Yield: 39%;

R_f = 0.4 (silica gel, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (t, *J* = 8.2 Hz, 1H), 7.12 – 7.05 (m, 3H), 3.88 – 3.86 (m, 4H), 3.21 – 3.19 (m, 4H);

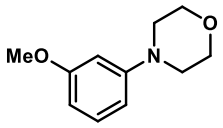
^{13}C NMR (100 MHz, $CDCl_3$): δ 151.52, 131.63 (q, *J* = 31.8 Hz), 129.76, 124.40 (q, *J* = 272.4 Hz), 118.58, 118.57, 116.37 (q, *J* = 3.9 Hz), 112.00 (q, *J* = 3.9 Hz), 66.86, 48.97;

^{19}F NMR (376 MHz, $CDCl_3$) δ –62.7;

HRMS (ESI-TOF): calc'd for $C_{11}H_{13}F_3NO^+$ [M+H$^+$] 232.0944, found 232.0946.

Both the proton and carbon NMR match the literature reported data.[2]

4-(3-Methoxyphenyl)morpholine (3j, CAS: 32040-09-8)



Physical state: colorless oil;

Yield: 67%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

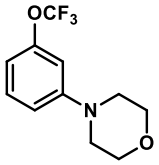
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 – 7.17 (m, 1H), 6.55 – 6.52 (m, 1H), 6.46 – 6.44(m, 2H), 3.87 – 3.84 (m, 4H), 3.80 (s, 3H), 3.17 – 3.14 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7, 152.8, 123.0, 108.6, 104.8, 102.3, 67.0, 55.3, 49.4;

HRMS (ESI-TOF): calc'd for $C_{11}H_{16}NO_2^+$ [M+H$^+$] 194.1176, found 194.1177.

Both the proton and carbon NMR match the literature reported data.[2]

4-(3-(Trifluoromethoxy)phenyl)morpholine (3k)



Physical state: colorless oil;

Yield: 38%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 – 7.24 (m, 1H), 6.83 – 6.80 (m, 1H), 6.73 – 6.71 (m, 2H), 3.87 – 3.85 (m, 4H), 3.18 – 3.16 (m, 4H);

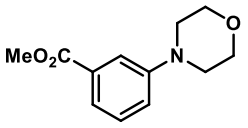
^{13}C NMR (100 MHz, $CDCl_3$): δ 152.7, 150.4, 130.2, 120.6 (q, J = 255.2 Hz), 113.6, 111.8, 108.2, 66.8, 48.9;

^{19}F NMR (376 MHz, $CDCl_3$) δ –57.47;

HRMS (ESI-TOF): calc'd for $C_{11}H_{13}F_3NO_2^+$ [M+H$^+$] 248.0893, found 248.0895.

Both the proton and carbon NMR match the literature reported data.[5]

Methyl 3-morpholinobenzoate (3l)



Physical state: light yellow oil;

Yield: 75%;

R_f = 0.4 (silica gel, PE:EtOAc = 10:1);

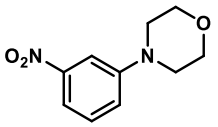
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (s, 1H), 7.53 (d, *J* = 7.7 Hz, 1H), 7.32 (t, *J* = 7.9 Hz, 1H), 7.10 – 7.07 (m, 1H), 3.89 (s, 3H), 3.87 – 3.85 (m, 4H), 3.20 – 3.18 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 167.4, 151.3, 131.1, 129.3, 121.1, 120.1, 116.4, 66.9, 52.2, 49.2;

HRMS (ESI-TOF): calc'd for $C_{12}H_{16}NO_3^+$ [M+H$^+$] 222.1125, found 222.1125.

Both the proton and carbon NMR match the literature reported data.[2]

4-(3-Nitrophenyl)morpholine (3m, CAS: 116922-22-6)



Physical state: yellow solid;

Yield: 77%;

R_f = 0.3 (silica gel, PE:EtOAc = 10:1);

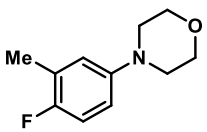
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 – 7.66 (m, 2H), 7.40 (t, *J* = 8.1 Hz, 1H), 7.18 (dd, *J* = 8.3, 2.5 Hz, 1H), 3.89 – 3.87 (m, 4H), 3.26 – 3.24 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.0, 149.4, 129.9, 121.0, 114.3, 109.6, 66.7, 48.6;

HRMS (ESI-TOF): calc'd for $C_{10}H_{12}N_2NaO_3^+$ [M+Na$^+$] 231.0740, found 231.0737.

Both the proton and carbon NMR match the literature reported data.[2]

4-(4-Fluoro-3-methylphenyl)morpholine (3n)



Physical state: light yellow oil;

Yield: 54%;

R_f = 0.35 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.91 (t, *J* = 9.0 Hz, 1H), 6.74 – 6.67 (m, 2H), 3.86 – 3.84 (m, 4H), 3.08 – 3.05 (m, 4H), 2.25 (s, 3H);

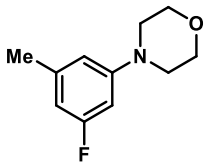
^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1 (d, *J* = 236.6 Hz), 147.7 (d, *J* = 2.6 Hz), 125.2 (d, *J* = 17.8 Hz), 119.3 (d, *J* = 4.6 Hz), 115.3 (d, *J* = 22.9 Hz), 114.9 (d, *J* = 7.5 Hz), 67.1, 50.5, 15.1 (d, *J* = 3.3 Hz);

^{19}F NMR (376 MHz, $CDCl_3$) δ –128.3;

IR (KBr pellet): *v* 2960, 2854, 1505, 1261, 1223, 1121, 880, 803, 759, 701 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{11}H_{15}FNO^+$ [M+H$^+$] 196.1132, found 196.1133.

4-(3-Fluoro-5-methylphenyl)morpholine (3o)



Physical state: light yellow oil;

Yield: 56%;

R_f = 0.5 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.49 (s, 1H), 6.41 (s, 1H), 6.39 (s, 1H), 3.85 – 3.83 (m, 4H), 3.15 – 3.13 (m, 4H), 2.30 (s, 3H);

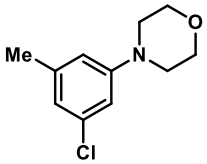
^{13}C NMR (100 MHz, $CDCl_3$): δ 163.9 (d, *J* = 242.6 Hz), 152.8 (d, *J* = 10.5 Hz), 140.7 (d, *J* = 9.7 Hz), 111.7 (d, *J* = 2.2 Hz), 107.3 (d, *J* = 21.3 Hz), 99.8 (d, *J* = 25.3 Hz), 66.9, 49.1, 21.9 (d, *J* = 2.3 Hz);

^{19}F NMR (376 MHz, $CDCl_3$) δ –113.4;

IR (KBr pellet): *v* 2961, 2855, 1614, 1589, 1450, 1268, 1194, 1134, 1123, 1019 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{11}H_{15}FNO^+$ [M+H$^+$] 196.1132, found 196.1135.

4-(3-Chloro-5-methylphenyl)morpholine (3p)



Physical state: light yellow oil;

Yield: 62%;

R_f = 0.4 (silica gel, PE:EtOAc = 30:1);

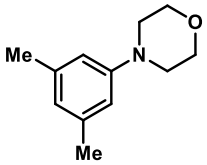
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.69 – 6.68 (m, 2H), 6.59 (s, 1H), 3.85 – 3.83 (m, 4H), 3.15 – 3.12 (m, 4H), 2.29 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.3, 140.4, 134.8, 120.7, 114.6, 112.9, 66.9, 49.1, 21.7;

IR (KBr pellet): *v* 2961, 2854, 1600, 1572, 1449, 1250, 1122, 992, 890, 828 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{11}H_{15}ClNO^+$ [M+H$^+$] 212.0837, found 212.0839.

4-(3,5-Dimethylphenyl)morpholine (3q, CAS: 16800-77-4)



Physical state: light yellow oil;

Yield: 60%;

R_f = 0.5 (silica gel, PE:EtOAc = 20:1);

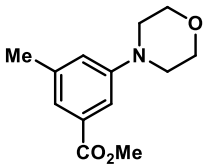
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.57 (s, 3H), 3.87 – 3.85 (m, 4H), 3.16 – 3.14 (m, 4H), 2.30 (s, 6H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 138.8, 122.1, 113.8, 67.1, 49.7, 21.8;

HRMS (ESI-TOF): calc'd for $C_{12}H_{18}NO^+$ [M+H$^+$] 192.1383, found 192.1387.

Both the proton and carbon NMR match the literature reported data.[6]

Methyl 3-methyl-5-morpholinobenzoate (3r)



Physical state: colorless oil;

Yield: 74%;

R_f = 0.3 (silica gel, PE:EtOAc = 10:1);

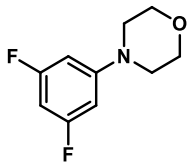
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 – 7.66 (m, 2H), 7.23 (d, *J* = 7.6 Hz, 1H), 3.89 (s, 3H), 3.86 – 3.84 (m, 4H), 2.94 – 2.92 (m, 4H), 2.36 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 151.4, 138.5, 131.3, 128.8, 124.7, 120.2, 67.4, 52.3, 52.1, 18.3;

IR (KBr pellet): *v* 2952, 2852, 1720, 1435, 1270, 1251, 1117, 1000, 941, 762 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{13}H_{18}NO_3^+$ [M+H$^+$] 236.1281, found 236.1283.

4-(3,5-Difluorophenyl)morpholine (3s, CAS: 736991-31-4)



Physical state: light yellow oil;

Yield: 69%;

R_f = 0.6 (silica gel, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.39 – 6.32 (m, 2H), 6.28 (tt, *J* = 8.8, 2.2 Hz, 1H), 3.84 – 3.82 (m, 4H), 3.15 – 3.13 (m, 4H);

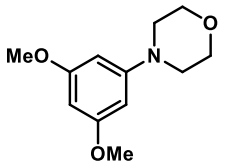
^{13}C NMR (100 MHz, $CDCl_3$): δ164.1 (dd, *J* = 244.3, 15.8 Hz), 153.4 (t, *J* = 12.2 Hz), 98.0 – 97.7 (m), 94.6 (t, *J* = 26.1 Hz), 66.6, 48.4;

^{19}F NMR (376 MHz, $CDCl_3$): δ -119.0 (s);

HRMS (ESI-TOF): calc'd for $C_{10}H_{12}F_2NO^+$ [M+H$^+$] 200.0881, found 200.0877.

Both the proton and carbon NMR match the literature reported data.[7]

4-(3,5-Dimethoxyphenyl)morpholine (3t, CAS: 102739-73-1)



Physical state: colorless oil;

Yield: 52%;

R_f = 0.3 (silica gel, PE:EtOAc = 10:1);

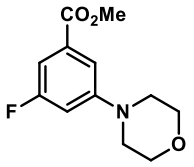
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.084 – 6.079 (m, 2H), 6.05 – 6.04 (m, 1H), 3.86 – 3.83 (m, 4H), 3.78 (s, 6H), 3.15 – 3.12 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 161.6, 153.4, 94.8, 91.9, 67.0, 55.4, 49.5;

HRMS (ESI-TOF): calc'd for $C_{12}H_{18}NO_3^+$ [M+H$^+$] 224.1281, found 224.1276.

Both the proton and carbon NMR match the literature reported data.[4]

Methyl 3-fluoro-5-morpholinobenzoate (3u)



Physical state: colorless oil;

Yield: 50%;

R_f = 0.4 (silica gel, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (s, 1H), 7.20 – 7.17 (m, 1H), 6.74 (dt, *J* = 11.6, 2.3 Hz, 1H), 3.90 (s, 3H), 3.87 – 3.84 (m, 4H), 3.21 – 3.19 (m, 4H);

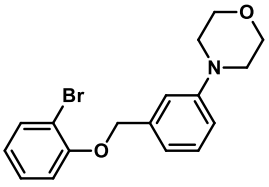
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5 (d, *J* = 3.7 Hz), 163.6 (d, *J* = 244.1 Hz), 152.8 (d, *J* = 9.9 Hz), 132.5 (d, *J* = 9.8 Hz), 112.0 (d, *J* = 2.3 Hz), 107.2 (d, *J* = 23.8 Hz), 106.5 (d, *J* = 25.5 Hz), 66.7, 52.5, 48.6;

^{19}F NMR (376 MHz, $CDCl_3$): δ –111.4;

IR (KBr pellet): *v* 2957, 2855, 1723, 1613, 1588, 1448, 1277, 1122, 1008, 766 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{12}H_{15}FNO_3^+$ [M+H$^+$] 240.1030, found 240.1024.

4-(3-((2-Bromophenoxy)methyl)phenyl)morpholine (3v)



Physical state: colorless oil;

Yield: 55%;

***R**f* = 0.3 (silica gel, PE:EtOAc = 6:1);

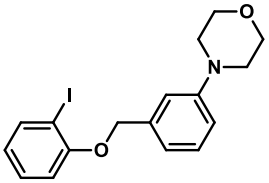
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.31 – 7.21 (m, 2H), 7.09 (s, 1H), 6.97 – 6.93 (m, 2H), 6.88 – 6.83 (m, 2H), 5.13 (s, 2H), 3.88 – 3.86 (m, 4H), 3.20 – 3.18 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 155.1, 151.7, 137.7, 133.5, 129.5, 128.6, 122.3, 118.5, 115.2, 114.1, 114.0, 112.6, 71.0, 67.0, 49.3;

IR (KBr pellet): *v* 2919, 2850, 1603, 1585, 1477, 1441, 1243, 1121, 1029c, 747 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{17}H_{19}BrNO_2^+$ [M+H$^+$] 348.0594, found 348.0589.

4-(3-((2-Iodophenoxy)methyl)phenyl)morpholine (3w)



Physical state: colorless oil;

Yield: 51%;

***R**f* = 0.4 (silica gel, PE:EtOAc = 5:1);

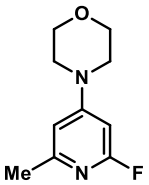
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.30 – 7.25 (m, 2H), 7.14 (t, *J* = 1.9 Hz, 1H), 7.00 – 6.95 (m, 1H), 6.88 – 6.84 (m, 2H), 6.72 (td, *J* = 7.6, 1.3 Hz, 1H), 5.12 (s, 2H), 3.88 – 3.86 (m, 4H), 3.21 – 3.18 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 157.3, 151.7, 139.6, 137.7, 129.6, 129.4, 123.0, 118.4, 115.2, 114.2, 112.9, 87.0, 71.0, 67.0, 49.4;

IR (KBr pellet): *v* 2958, 2852, 2360, 1603, 1583, 1470, 1244, 1121, 1017, 748 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{17}H_{19}INO_2^+$ [M+H$^+$] 396.0455, found 396.0454.

4-(2-Fluoro-6-methylpyridin-4-yl)morpholine (3x)



Physical state: colorless oil;

Yield: 43%;

R_f = 0.4 (silica gel, PE:EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (s, 1H), 7.04 (dd, *J* = 10.1, 1.9 Hz, 1H), 3.87 – 3.85 (m, 4H), 3.10 – 3.07 (m, 4H), 2.28 (s, 3H);

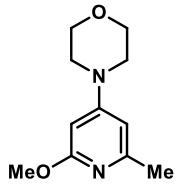
^{13}C NMR (100 MHz, $CDCl_3$): δ 154.5 (d, *J* = 236.8 Hz), 138.2 (d, *J* = 14.3 Hz), 134.5 (d, *J* = 23.8 Hz), 131.6 (d, *J* = 4.6 Hz), 128.2 (d, *J* = 4.8 Hz), 66.9, 50.4 (d, *J* = 3.7 Hz), 17.8;

^{19}F NMR (376 MHz, $CDCl_3$): δ –78.1;

IR (KBr pellet): *v* 2965, 2857, 1740, 1451, 1249, 1102, 1048, 1008, 858, 709 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{10}H_{14}FN_2O^+$ [M+Na^+] 197.1085, found 197.1086.

4-(2-Methoxy-6-methylpyridin-4-yl)morpholine (3y)



Physical state: colorless oil;

Yield: 44%;

R_f = 0.2 (silica gel, PE:EtOAc = 20:1);

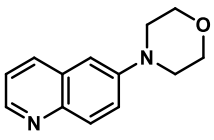
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.25 (d, *J* = 1.9 Hz, 1H), 5.88 (d, *J* = 2.0 Hz, 1H), 3.88 (s, 3H), 3.81 – 3.79 (m, 4H), 3.23 – 3.21 (m, 4H), 2.36 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 165.6, 158.6, 156.6, 102.9, 90.3, 66.6, 53.4, 46.9, 24.7;

IR (KBr pellet): *v* 2961, 2854, 1606, 1556, 1453, 1205, 1123, 1080, 1051, 818 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{11}H_{17}N_2O_2^+$ [M+H^+] 209.1285, found 209.1289.

4-(Quinolin-6-yl)morpholine (3z, CAS: 1275591-19-9)



Physical state: yellow solid;

Melting point: 93 – 95 °C;

Yield: 74%;

$\mathbf{R}_f$ = 0.2 (silica gel, PE:EtOAc = 3:1);

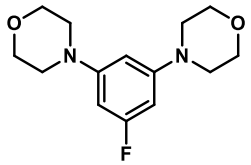
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.72 (dd, *J* = 4.3, 1.6 Hz, 1H), 8.01 – 7.98 (m, 2H), 7.47 (dd, *J* = 9.3, 2.8 Hz, 1H), 7.31 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.02 (d, *J* = 2.7 Hz, 1H), 3.92 – 3.90 (m, 4H), 3.29 – 3.27 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.4, 147.9, 144.1, 134. 9, 130.2, 129.5, 122.2, 121.57, 109.1, 66.9, 49.5;

HRMS (ESI-TOF): calc'd for $C_{13}H_{15}N_2O^+$ [M+H$^+$] 215.1179, found 215.1183.

Both the proton and carbon NMR match the literature reported data.[4]

4,4'-(5-Fluoro-1,3-phenylene)dimorpholine (3aa)



Physical state: white solid;

Boiling point: 98 – 100 °C;

Yield: 42%;

$\boldsymbol{R_f}$ = 0.3 (silica gel, PE:EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.18 – 6.16 (m, 2H), 6.13 (d, *J* = 2.1 Hz, 1H), 3.85 – 3.82 (m, 8H), 3.14 – 3.12 (m, 8H);

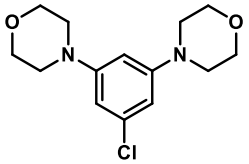
^{13}C NMR (100 MHz, $CDCl_3$): δ 164.8 (d, *J* = 240.1 Hz), 153.5 (d, *J* = 12.1 Hz), 98.4 (d, *J* = 2.0 Hz), 94.9 (d, *J* = 25.7 Hz), 66.9, 49.3;

^{19}F NMR (376 MHz, $CDCl_3$): δ -109.7;

IR (KBr pellet): *v* 2959, 2853, 1614, 1581, 1449, 1267, 1203, 1121, 1005, 814 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{14}H_{20}FN_2O_2^+$ [M+H$^+$] 267.1503, found 267.1502.

4,4'-(5-Chloro-1,3-phenylene)dimorpholine (3ab, CAS: 55086-81-2)



Physical state: colorless oil

Yield: 47%;

R_f = 0.3 (silica gel, PE:EtOAc = 5:1);

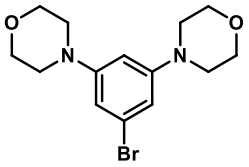
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.43 (s, 1H), 6.42 (s, 1H), 6.28 (t, *J* = 2.1 Hz, 1H), 3.84 – 3.82 (m, 8H), 3.14 – 3.12 (m, 8H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 153.1, 135.8, 108.0, 101.5, 66.9, 49.3;

IR (KBr pellet): *v* 2959, 2853, 1591, 1565, 1447, 1204, 1121, 1002, 940, 817 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{14}H_{20}ClN_2O_2^+$ [M+H$^+$] 283.1208, found 283.1207.

4,4'-(5-Bromo-1,3-phenylene)dimorpholine (3ac)



Physical state: colorless oil;

Yield: 36%;

R_f = 0.4 (silica gel, PE:EtOAc = 5:1);

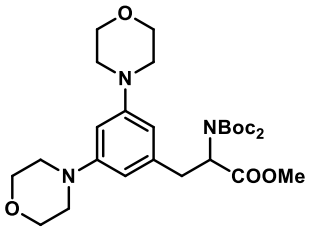
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.57 (s, 1H), 6.57 (s, 1H), 6.32 (t, *J* = 2.1 Hz, 1H), 3.84 – 3.81 (m, 8H), 3.14 – 3.11 (m, 8H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 153.3, 124.1, 111.0, 102.1, 66.9, 49.4;

IR (KBr pellet): *v* 2958, 2852, 1594, 1557, 1446, 1203, 1120, 1001, 929, 763 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{14}H_{20}BrN_2O_2^+$ [M+H$^+$] 327.0703, found 327.0704.

Methyl 2-(bis(*tert*-butoxycarbonyl)amino)-3-(3,5-dimorpholinophenyl)propanoate (3ad)



Physical state: colorless oil;

Yield: 26%;

R_f = 0.3 (silica gel, PE:EtOAc = 2:1);

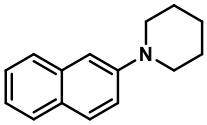
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.30 – 6.28 (m, 3H), 5.13 (dd, *J* = 10.5, 4.8 Hz, 1H), 3.84 – 3.81 (m, 8H), 3.74 (s, 3H), 3.32 (dd, *J* = 14.0, 4.8 Hz, 1H), 3.17 – 3.12 (m, 1H), 3.12 – 3.09 (m, 8H), 1.39 (s, 18H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 152.5, 151.9, 139.2, 109.9, 102.7, 83.0, 67.1, 59.4, 52.4, 49.9, 36.8, 28.0;

IR (KBr pellet): *v* 2920, 2851, 1744, 1594, 1367, 1253, 1141, 1121, 1004, 872 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{28}H_{43}N_3NaO_8^+$ [M+Na^+] 572.2941, found 572.2941.

1-(Naphthalen-2-yl)piperidine (3B, CAS: 5465-85-0)



Physical state: white solid;

Yield: 57%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

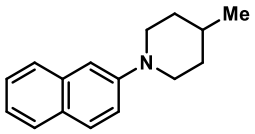
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 – 7.64 (m, 3H), 7.39 – 7.35 (m, 1H), 7.30 – 7.23 (m, 2H), 7.12 (d, *J* = 2.5 Hz, 1H), 3.28 – 3.21 (m, 4H), 1.79 – 1.73 (m, 4H), 1.65 – 1.58 (m, 2H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2, 134.8, 128.6, 128.4, 127.5, 126.8, 126.2, 123.2, 120.3, 110.5, 51.2, 26.0, 24.5;

HRMS (ESI-TOF): calc'd for $C_{15}H_{18}N^+$ [M+H^+] 212.1434, found 212.1432.

Both the proton and carbon NMR match the literature reported data.[8]

4-Methyl-1-(naphthalen-2-yl)piperidine (3C)



Physical state: yellow solid;

Melting point: 44 – 46 ºC;

Yield: 70%;

$\mathbf{R}_f$ = 0.5 (silica gel, PE:EtOAc = 15:1);

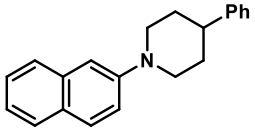
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 – 7.59(m, 3H), 7.33 – 7.29 (m, 1H), 7.22 – 7.18 (m, 2H), 7.06 (s, 1H), 3.72 – 3.68 (m, 2H), 2.73 – 2.66 (m, 2H), 1.73 – 1.69 (m, 2H), 1.55 – 1.42 (m, 2H), 1.39 – 1.29 (m, 2H), 0.93 (d, *J* = 6.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9, 134.8, 128.6, 128.4, 127.5, 126.8, 126.2, 123.2, 120.3, 110.5, 50.5, 34.3, 30.9, 22.1;

IR (KBr pellet): *v* 2948, 2922, 1627, 1597, 1508, 1338, 1214, 1135, 844, 744 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{16}H_{20}N^+$ [M+H$^+$] 226.1590, found 226.1592.

1-(Naphthalen-2-yl)-4-phenylpiperidine (3D, CAS: 1185746-63-7)



Physical state: yellow solid;

Melting point: 116 – 118 ºC;

Yield: 72%;

$\mathbf{R}_f$ = 0.5 (silica gel, PE:EtOAc = 15:1);

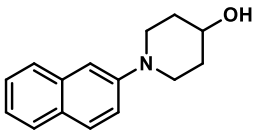
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 – 7.69 (m, 3H), 7.42 – 7.37 (m, 1H), 7.35 – 7.18 (m, 8H), 3.95 – 3.92 (m, 2H), 2.93 – 2.86 (m, 2H), 2.74 – 2.66 (m, 1H), 2.02 – 1.91 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.8, 146.2, 134. 8, 128.8, 128.7, 128.5, 127.5, 127.01, 126.8, 126.5, 126.3, 123.4, 120.3, 110.7, 51.0, 42.7, 33.5;

HRMS (ESI-TOF): calc'd for $C_{21}H_{22}N^+$ [M+H$^+$] 288.1747, found 288.1747.

Both the proton and carbon NMR match the literature reported data.[9]

1-(Naphthalen-2-yl)piperidin-4-ol (3E)



Physical state: white solid;

Melting point: 94 – 96 °C;

Yield: 73%;

R_f = 0.3 (silica gel, PE:EtOAc = 2:1);

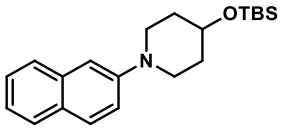
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 – 7.66 (m, 3H), 7.41 – 7.37 (m, 1H), 7.30 – 7.24 (m, 2H), 7.13 (d, *J* = 2.4 Hz, 1H), 3.89 – 3.83 (m, 1H), 3.68 – 3.63 (m, 2H), 3.02 – 2.95 (m, 2H), 2.07 – 2.01 (m, 2H), 1.78 – 1.69 (m, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.2, 134.7, 128.8, 128.5, 127.5, 126.8, 126.3, 123.4, 120.1, 110.7, 68.0, 47.8, 34.3;

IR (KBr pellet): *v* 2939, 2922, 1628, 1508, 1382, 1209, 1062, 833, 738, 475 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{15}H_{18}NO^+$ [M+H$^+$] 228.1383, found 228.1378.

4-((*tert*-Butyldimethylsilyl)oxy)-1-(naphthalen-2-yl)piperidine (3F)



Physical state: white solid;

Melting point: 74 – 76 °C;

Yield: 45%;

R_f = 0.7 (silica gel, PE:EtOAc = 20:1);

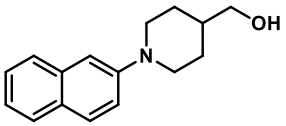
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 – 7.66 (m, 3H), 7.40 – 7.36 (m, 1H), 7.30 – 7.24 (m, 2H), 7.14 (d, *J* = 1.9 Hz, 1H), 3.94 – 3.88 (m, 1H), 3.57 – 3.51 (m, 2H), 3.14 – 3.08 (m, 2H), 1.96 – 1.90 (m, 2H), 1.77 – 1.69 (m, 2H), 0.91 (s, 9H), 0.09 (s, 6H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.5, 134.8, 128.7, 128.4, 127.5, 126.8, 126.3, 123.3, 120.0, 110.5, 67.6, 47.1, 34.5, 26.0, 18.5, -4.5;

IR (KBr pellet): *v* 2950, 2927, 2854, 1253, 1114, 844, 836, 823, 774, 754 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{21}H_{32}NOSi^+$ [M+H$^+$] 342.2248, found 342.2243.

(1-(Naphthalen-2-yl)piperidin-4-yl)methanol (3G)



Physical state: purple solid;

Yield: 83%;

R$_f$ = 0.4 (silica gel, PE:EtOAc = 2:1);

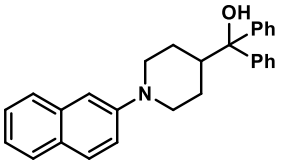
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 – 7.66 (m, 3H), 7.40 – 7.36 (m, 1H), 7.30 – 7.25 (m, 2H), 7.14 (s, 1H), 3.79 – 3.75 (m, 1H), 3.66 – 3.53 (m, 3H), 2.83 – 2.77 (m, 1H), 2.66 – 2.60 (m, 1H), 2.01 – 1.90 (m, 1H), 1.87 – 1.67 (m, 4H), 1.26 – 1.11 (m, 1H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9, 134.7, 128.7, 128.5, 127.5, 126.8, 126.3, 123.4, 120.4, 110.8, 66.2, 53.8, 51.0, 38.7, 27.2, 24.8;

IR (KBr pellet): *v* 2928, 2853, 1627, 1597, 1508, 1390, 1216, 1117, 1030, 746 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{16}H_{20}NO^+$ [M+H$^+$] 242.1539, found 242.1547.

(1-(Naphthalen-2-yl)piperidin-4-yl)diphenylmethanol (3H)



Physical state: yellow solid;

Melting point: 110 – 112 ºC;

Yield: 53%;

R$_f$ = 0.6 (silica gel, PE:EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 – 7.65 (m, 3H), 7.53 – 7.51 (m, 4H), 7.40 – 7.19 (m, 9H), 7.10 (s, 1H), 3.86 – 3.81 (m, 2H), 2.84 – 2.77 (m, 2H), 2.66 – 2.58 (m, 1H),

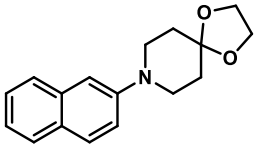
2.13 (s, 1H), 1.69 – 1.60 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.5, 145.9, 134.7, 128.7, 128.5, 128.4, 127.5, 126.8, 126.3, 125.9, 123.4, 120.2, 110.6, 79.7, 50.7, 44.3, 26.6;

IR (KBr pellet): *v* 2956, 1626, 1596, 1508, 1446, 1387, 1180, 1064, 746, 703 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{28}H_{28}NO^+$ [M+H^+] 394.2165, found 394.2169.

8-(Naphthalen-2-yl)-1,4-dioxa-8-azaspiro[4.5]decane (3I)



Physical state: yellow solid;

Melting point: 90 – 92 °C;

Yield: 57%;

R_f = 0.6 (silica gel, PE:EtOAc = 5:1);

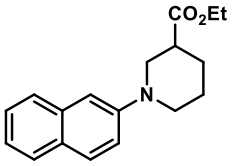
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 – 7.68 (m, 3H), 7.43 – 7.39 (m, 1H), 7.32 – 7.27 (m, 2H), 7.16 (d, *J* = 2.4 Hz, 1H), 4.02 (s, 4H), 3.47 – 3.42 (m, 4H), 1.95 – 1.89 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 148.9, 134.7, 128.8, 128.5, 127.5, 126.8, 126.3, 123.4, 120.1, 110.8, 107.3, 64.5, 48.2, 34.7;

IR (KBr pellet): *v* 2957, 1627, 1597, 1508, 1210, 1142, 1099, 1035, 842, 747 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{17}H_{20}NO_2^+$ [M+H^+] 270.1489, found 270.1489.

Ethyl 1-(naphthalen-2-yl)piperidine-3-carboxylate (3J)



Physical state: yellow oil;

Yield: 71%;

R_f = 0.5 (silica gel, PE:EtOAc = 8:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 – 7.69 (m, 3H), 7.43 – 7.39 (m, 1H), 7.32 – 7.29

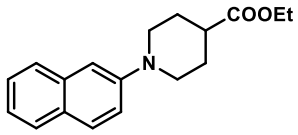
(m, 2H), 7.17 (s, 1H), 4.21 (q, J = 7.1 Hz, 2H), 3.85 – 3.81 (m, 1H), 3.63 – 3.59 (m, 1H), 3.17 – 3.11 (m, 1H), 2.95 – 2.88 (m, 1H), 2.79 – 2.72 (m, 1H), 2.09 – 2.06 (m, 1H), 1.92 – 1.67 (m, 3H), 1.31 (t, J = 7.1 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 174.0, 149.4, 134.7, 128.8, 128.6, 127.5, 126.8, 126.3, 123.5, 120.4, 110.9, 60.7, 52.6, 50.3, 41.6, 27.1, 24.4, 14.4;

IR (KBr pellet): v 2936, 1728, 1627, 1598, 1508, 1389, 1178, 1034, 838, 746 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{18}H_{22}NO_2^+$ [M+H$^+$] 284.1645, found 284.1639.

Ethyl 1-(naphthalen-2-yl)piperidine-4-carboxylate (3K)



Physical state: yellow oil;

Yield: 59%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

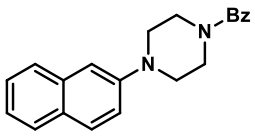
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 – 7.66 (m, 3H), 7.41-7.37 (m, 1H), 7.30 – 7.25 (m, 2H), 7.12 (d, J = 2.3 Hz, 1H), 4.17 (q, J = 7.1 Hz, 2H), 3.75 (dt, J = 12.4, 3.2 Hz, 2H), 2.86 (td, J = 12.3, 2.8 Hz, 2H), 2.51 – 2.43 (m, 1H), 2.15 – 1.86 (m, 4H), 1.28 (t, J = 7.2 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 175.0, 149.5, 134.7, 128.8, 128.6, 127.5, 126.8, 126.3, 123.5, 120.2, 110.8, 60.6, 49.7, 41.2, 28.2, 14.4;

IR (KBr pellet): v 2952, 1729, 1627, 1597, 1508, 1389, 1173, 1044, 959, 746 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{18}H_{22}NO_2^+$ [M+H$^+$] 284.1645, found 284.1646.

(4-(Naphthalen-2-yl)piperazin-1-yl)(phenyl)methanone (3L)



Physical state: yellow oil;

Yield: 43%;

R_f = 0.4 (silica gel, PE:EtOAc = 2:1);

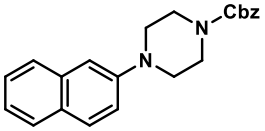
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.68 (m, 3H), 7.49 – 7.39 (m, 6H), 7.35 – 7.29 (m, 1H), 7.26 – 7.24 (m, 1H), 7.12 (s, 1H), 3.99 (brs, 2H), 3.64 (brs, 2H), 3.35 (brs, 2H), 3.21 (brs, 2H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 148.8, 135.7, 134.5, 130.0, 129.1, 129.0, 128.7, 127.6, 127.2, 126.9, 126.6, 124.0, 119.8, 111.2, 50.3, 50.1, 47.7, 42.2;

IR (KBr pellet): *v* 2859, 1699, 1627, 1597, 1429, 1242, 1223, 1120, 963, 747 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{21}H_{21}N_2O^+$ [M+H^+] 317.1648, found 317.1649.

Benzyl 4-(naphthalen-2-yl)piperazine-1-carboxylate (3M)



Physical state: yellow solid;

Melting point: 66 – 68 ºC;

Yield: 50%;

R_f = 0.5 (silica gel, PE:EtOAc = 4:1);

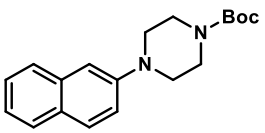
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.66 (m, 3H), 7.44 – 7.28 (m, 7H), 7.27 – 7.22 (m, 1H), 7.11 (s, 1H), 5.18 (s, 2H), 3.71 (t, *J* = 5.1 Hz, 4H), 3.24 (s, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4, 149.0, 136.7, 134.5, 129.0, 128.9, 128.7, 128.2, 128.1, 127.6, 126.9, 126.5, 123.9, 119.9, 111.1, 67.4, 49.9, 43.9;

IR (KBr pellet): *v* 2820, 1702, 1627, 1597, 1429, 1243, 1223, 1120, 963, 697 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{22}H_{23}N_2O_2^+$ [M+H^+] 347.1754, found 347.1751.

***tert*-Butyl 4-(naphthalen-2-yl)piperazine-1-carboxylate (3N, CAS: 684249-11-4)**



Physical state: white solid;

Melting point: 79 – 81 ºC;

Yield: 60%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

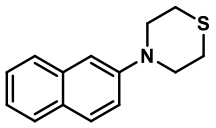
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.67 (m, 3H), 7.41 (t, *J* = 7.1 Hz, 1H), 7.34 – 7.24 (m, 2H), 7.12 (d, *J* = 2.3 Hz, 1H), 3.63 (t, *J* = 5.1 Hz, 4H), 3.23 (t, *J* = 5.2 Hz, 4H), 1.50 (s, 9H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 154.9, 149.2, 134.6, 129.0, 128.9, 127.6, 126.9, 126.5, 123.8, 119.9, 111.0, 80.1, 49.9, 28.6;

IR (KBr pellet): *v* 2975, 1695, 1628, 1420, 1365, 1247, 1169, 1122, 961, 746 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{19}H_{24}N_2NaO_2^+$ [M+Na^+] 335.1730, found 335.1731.

4-(Naphthalen-2-yl)thiomorpholine (3O)



Physical state: white solid;

Yield: 56%;

R_f = 0.6 (silica gel, PE:EtOAc = 20:1);

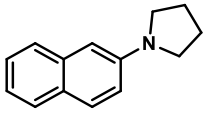
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 – 7.68 (m, 3H), 7.44 – 7.40 (m, 1H), 7.33 – 7.29 (m, 1H), 7.22 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.13 (m, 1H), 3.65 – 3.63 (m, 4H), 2.83 – 2.81 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 149.2, 134.7, 129.0, 128.6, 127.6, 126.8, 126.5, 123.7, 120.3, 111.5, 52.5, 27.1;

HRMS (ESI-TOF): calc'd for $C_{14}H_{16}NS^+$ [M+H^+] 230.0998, found 230.0993.

Both the proton and carbon NMR match the literature reported data.[10]

1-(Naphthalen-2-yl)pyrrolidine (3P, CAS: 13672-14-5)



Physical state: white solid;

Yield: 42%;

R_f = 0.8 (silica gel, PE:EtOAc = 50:1);

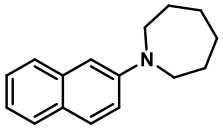
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 – 7.62 (m, 3H), 7.36 – 7.32 (m, 1H), 7.17 – 7.14 (m, 1H), 7.00 (dd, *J* = 9.0, 2.5 Hz, 1H), 6.76 (d, *J* = 2.4 Hz, 1H), 3.43 – 3.40 (m, 4H), 2.08 – 2.04 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 146.0, 135.4, 128.9, 127.7, 126.4, 126.3, 125.9, 121.3, 115.8, 104.8, 48.0, 25.7;

HRMS (ESI-TOF): calc'd for $C_{14}H_{16}N^+$ [M+H$^+$] 198.1277, found 198.1276.

Both the proton and carbon NMR match the literature reported data.[8]

1-(Naphthalen-2-yl)azepane (3Q, CAS:55045-05-1)



Physical state: yellow oil;

Yield: 33%;

R_f = 0.6 (silica gel, PE:EtOAc = 50:1);

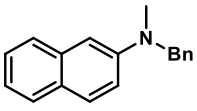
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 – 7.65 (m, 2H), 7.61 (d, *J* = 8.3 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.17 – 7.10 (m, 2H), 6.88 (d, *J* = 2.5 Hz, 1H), 3.60 – 3.57 (m, 4H), 1.88 – 1.84 (m, 4H), 1.60 – 1.56 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9, 135.5, 129.0, 127.5, 126.2, 126.0, 121.4, 115.3, 104.6, 49.6, 28.0, 27.2;

HRMS (ESI-TOF): calc'd for $C_{16}H_{20}N^+$ [M+H$^+$] 226.1590, found 226.1591.

Both the proton and carbon NMR match the literature reported data.[8]

***N*-Benzyl-*N*-methylnaphthalen-2-amine (3R, CAS: 447407-87-6)**



Physical state: yellow oil;

Yield: 35%;

R$_f$ = 0.7 (silica gel, PE:EtOAc = 5:1);

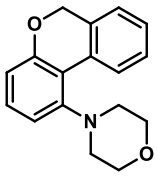
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 – 7.61 (m, 3H), 7.37 – 7.29 (m, 3H), 7.26 – 7.24 (m, 3H), 7.21 – 7.14 (m, 2H), 6.95 (d, *J* = 2.6 Hz, 1H), 4.64 (s, 2H), 3.09 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 147.8, 139.0, 135.2, 129.0, 128.7, 127.6, 127.1, 127.0, 126.9, 126.4, 126.3, 122.1, 116.3, 106.3, 56.9, 38.8;

HRMS (ESI-TOF): calc'd for $C_{18}H_{18}N^+$ [M+H$^+$] 248.1434, found 248.1427.

Both the proton and carbon NMR match the literature reported data.[9]

4-(6*H*-Benzo[*c*]chromen-1-yl)morpholine (5)



Physical state: colorless oil;

Yield: 12%;

R_f = 0.3 (silica gel, PE:EtOAc = 20:1);

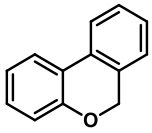
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.91 (d, *J* = 7.9 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.27 – 7.23 (m, 1H), 7.19 – 7.14 (m, 2H), 6.73 – 6.67 (m, 2H), 4.91 (s, 2H), 3.85 – 3.79 (m, 6H), 3.18 (s, 1H), 2.88 (s, 1H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 157.4, 151.0, 131.6, 130.7, 129.7, 128.1, 127.3, 124.9, 124.3, 115.5, 111.9, 111.1, 68.8, 67.0, 52.1;

IR (KBr pellet): *v* 2851, 1593, 1446, 1248, 1118, 1029, 977, 798, 741, 722 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{17}H_{18}NO_2^+$ [M+H$^+$] 268.1332, found 268.1326.

6*H*-Benzo[*c*]chromene (6, CAS: 229-95-8)



Physical state: colorless oil;

Yield: 84%;

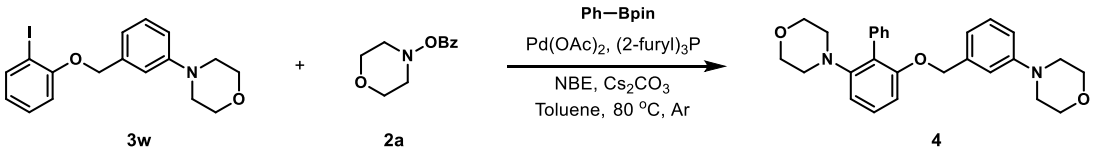
R_f = 0.8 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.3 Hz, 1H), 7.71 (d, *J* = 7.6 Hz, 1H), 7.38

(t, *J* = 7.1 Hz, 1H), 7.29 – 7.25 (m, 2H), 7.16 (d, *J* = 7.0 Hz, 1H), 7.06 (t, *J* = 7.2 Hz, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 5.13 (s, 2H).

Both the proton and carbon NMR match the literature reported data.[9]

6. Procedure for the synthesis of compound 4.[11]



3w **2a** **4**

To a 25 mL of Schlenk tube equipped with a magnetic stir bar was charged with aryl iodide **3w** (0.16 mmol; 63.0 mg, 1.0 equiv), **2a** (0.24 mmol, 49.7 mg, 1.5 equiv), phenylboronic acid pinacol ester (0.24 mmol, 49.0 mg, 1.5 equiv), $Pd(OAc)_2$ (0.008 mmol, 1.8 mg, 0.05 equiv), $(2\text{-furyl})_3P$ (0.032 mmol, 7.4 mg, 0.2 equiv), Cs_2CO_3 (0.48 mmol, 156.4 mg, 3.0 equiv), norbornene (0.08 mmol, 7.5 mg, 0.5 equiv), and dry toluene (1.6 mL). The resulting reaction mixture was stirred at r.t. for 10 min under Ar and then heated at 80 °C for 18 h. The reaction was monitored by TLC, after completion of the reaction, the mixture was cooled to r.t., then the mixture was filtered through a thin pad of celite eluting with ethyl acetate (15 mL), and the filtrate was sequentially washed with water, brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (PE:EtOAc = 4:1) to yield the desired product **4** (49.0 mg, 71% yield).

Physical state: colorless oil;

R_f = 0.4 (silica gel, PE:EtOAc = 4:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 – 7.48 (m, 2H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.23 (m, 2H), 7.17 (t, *J* = 8.1 Hz, 1H), 6.80 – 6.67 (m, 5H), 4.97 (s, 2H), 3.86 – 3.79 (m, 4H), 3.50 – 3.44 (m, 4H), 3.04 – 2.96 (m, 4H), 2.81 – 2.73 (m, 4H);

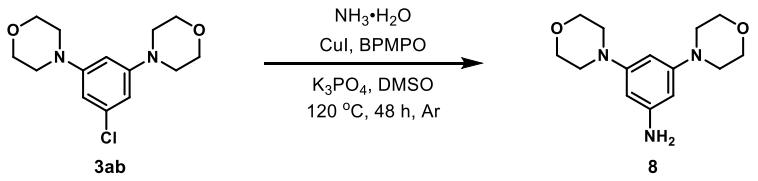
^{13}C NMR (100 MHz, $CDCl_3$): δ 156.6, 151.9, 151.4, 138.6, 136.3, 131.3, 129.1, 128.9, 127.8, 126.5, 125.1, 117.7, 114.4, 113.5, 111.7, 107.9, 70.2, 67.1, 67.0, 51.7, 49.2;

IR (KBr pellet): *v* 2958, 2852, 1740, 1601, 1583, 1448, 1260, 1117, 792, 700 cm^{-1};

HRMS (ESI-TOF): calc'd for $C_{27}H_{31}N_2O_3^+$ [M+H$^+$] 431.2329, found 431.2330.

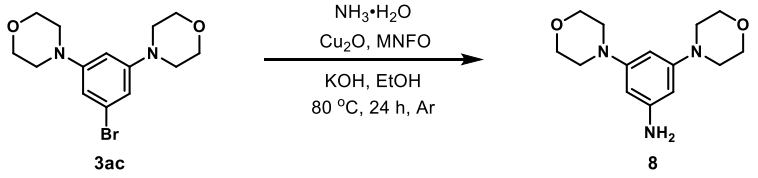
7. Procedure for the synthesis of compound 8.

Compound **8** was prepared according to the literature reported procedure.[12,13]



3ab **8**

Method A: In an argon-filled glove box, an 4.0 mL vial equipped with a magnetic stir bar was charged with CuI (0.005 mmol, 1.0 mg, 0.05 equiv), BPMPO (0.01 mmol, 4.0 mg, 0.1 equiv), K_3PO_4 (0.11 mmol, 23.3 mg, 1.1 equiv), **3ab** (0.1 mmol, 28.0 mg, 1.0 equiv), then the vial was tightly sealed and transferred out of glovebox. Ammonia aqueous solution (w/w 25%, 0.3 mmol, 46 μL, 3.0 equiv) and DMSO (0.2 mL) were then added into the reaction vessel via syringe. The reaction mixture was stirred at 120 °C for 48 hours. After the reaction vessel was cooled to r.t., then the mixture was directly concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (PE:EtOAc = 2:1) to yield the desired product **8** (16.0 mg, 61% yield, 90% brsm) as a white solid and recovered substrate **3ab** (10 mg, 32% yield) as a colorless oil.



3ac **8**

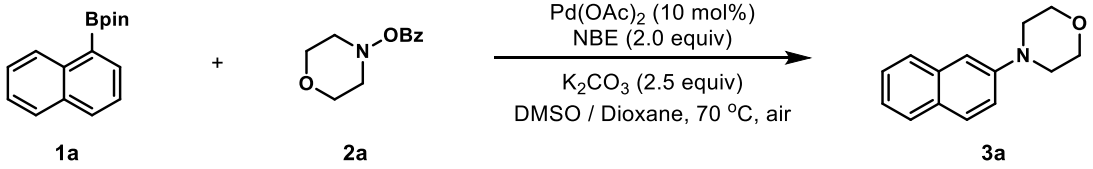
Method B: In an argon-filled glove box, an 4.0 mL vial equipped with a magnetic stir bar was charged with Cu_2O (0.008 mmol, 1.2 mg, 0.05 equiv), MNFO (0.008 mmol, 2.32 mg, 0.05 equiv), KOH (0.21 mmol, 11.8 mg, 1.3 equiv), **3ac** (0.16 mmol, 53.0 mg, 1.0 equiv), then the vial was tightly sealed and transferred out of glovebox. Ammonia aqueous solution (w/w 25%, 0.8 mmol, 64 μL, 5.0 equiv) and EtOH (0.2 mL) were then added into the reaction vessel via syringe. The reaction mixture was stirred at 80 °C for 24 hours. After the reaction vessel was cooled to r.t., then the mixture was concentrated directly *in vacuo*. The residue was purified by column chromatography on silica gel (PE:EtOAc = 2:1) to yield the desired product **8** (27.0 mg, 64% yield) as a white solid. **^{1}H NMR** (400 MHz, CDCl3): δ 5.92 (t, *J* = 2.1 Hz, 1H), 5.84 (d, *J* = 1.9 Hz, 2H), 3.83 – 3.81 (m, 8H), 3.12 – 3.09 (m, 8H);

^{13}C NMR (100 MHz, CDCl3): δ 153.5, 148.1, 95. 8, 95.3, 67.1, 49.7.

HRMS (ESI-TOF): calc'd for $C_{14}H_{22}N_3O_2^+$ [M+H$^+$] 264.1706, found 264.1701.

The proton NMR match the literature reported data.[14]

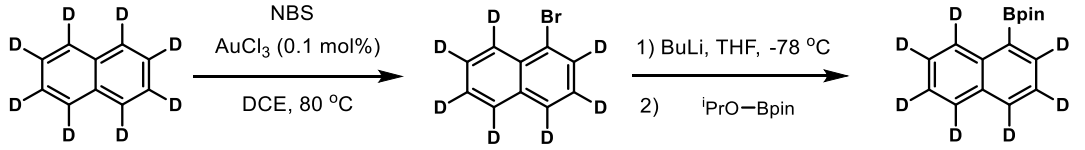
8. Experimental procedure for gram-scale synthesis of compound 3a.



To a 250 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with 4,4,5,5-tetramethyl-2-(naphthalen-1-yl)-1,3,2-dioxaborolane **1a** (2.29 g, 9.0 mmol, 1.5 equiv), $Pd(OAc)_2$ (135.0 mg, 0.6 mmol, 0.1 equiv), K_2CO_3 (2.07 g, 15.0 mmol, 2.5 equiv), morpholino benzoate **2a** (1.24 g, 6.0 mmol, 1.0 equiv), norbornene (1.13 g, 12.0 mmol, 2.0 equiv), DMSO (20.0 mL) and Dioxane (50 mL) under air. The reaction was stirred at 70 ºC for 16 h. The reaction mixture was allowed to cool down to r.t. and extracted with ethyl acetate (3 × 40 mL), and the combined organic extracts were washed with water and brine, dried over Na_2SO_4. After filtered and concentrated *in vacuo*, the residue was directly purified by column chromatography on silica gel (PE:EtOAc = 20:1) to yield the desired product **3a** (0.92 g, 72%yield) as a yellow solid.

9. Mechanistic study.

9.1. Preparation of 1-naphthylboronic ester-d_7 (1a-*d*).

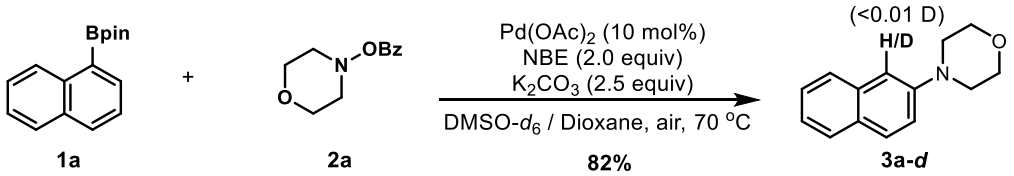


1-Bromonaphthalene-d_7 was prepared according to the literature reported procedure.[15] To a 25 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with NBS (356.0 mg, 2.0 mmol, 1.0 equiv), naphthalene-d_8 (272.4 mg, 2.0 mmol, equiv) and DCE (4 mL). $AuCl_3$ (0.003 mmol, 1 mg) was dissolved in another 1 mL DCE. 0.07 mL of this $AuCl_3$ solution was added to the reaction mixture via syringe. The reaction was stirred at 80 ºC for 16 h. Upon completion of bromination, the solvent was removed

under reduced pressure. Then 4.0 mL dry THF was added to the reaction mixture under Ar atmosphere and cooled down to -78 °C. ^{n}BuLi (0.8 mL, 2.0 mmol, 2.5 M in hexane) was added dropwise to the reaction mixture. The temperature was maintained at -78 °C for 1 h and afterwards ^{i}PrOBpin (446.0 mg, 2.4 mmol) was added. The resulting reaction mixture was allowed to warm to r.t. and stirred for 40 min. The reaction was quenched by saturated NH_4Cl solution and extracted with ethyl acetate (3 × 20 mL), washed with water and brine, dried over Na_2SO_4. After filtered and concentrated *in vacuo*, the residue was directly purified by column chromatography on silica gel (PE:EtOAc = 20:1) to yield the desired product **1a-*d*** (207 mg, 40%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.43 (s, 12H).

9.2. Deuterium labelling study with DMSO-d_6.



To a 25 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with **1a** (38.1 mg, 0.15 mmol, 1.5 equiv), **2a** (20.7 mg, 0.2 mmol, 1.0 equiv), $Pd(OAc)_2$ (2.25 mg, 0.01 mmol, 0.1 equiv), K_2CO_3 (34.5 mg, 0.25 mmol, 2.5 equiv), NBE (18.8 mg, 0.2 mmol, 2.0 equiv) and DMSO-d_6 / Dioxane (1.4 mL, v:v=2:5) under air. The reaction was stirred at 70 °C. The reaction was monitored by TLC, after completion of the reaction, the mixture was cooled to r.t., then the mixture was filtered through a thin pad of celite eluting with ethyl acetate (15 mL), and the filtrate was sequentially washed with water, brine, dried over Na_2SO_4. After concentrated *in vacuo*, the residue was purified by column chromatography on silica gel to yield the desired product **3a-*d***.

Physical state: yellow solid;

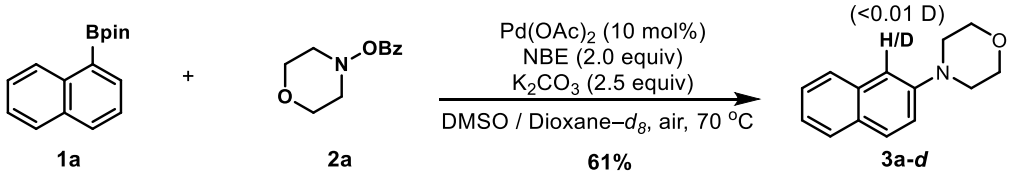
Yield: 82%;

***R*$_f$** = 0.4 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.70 (m, 3H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.13 (s, 1H), 3.94 – 3.91 (m, 4H), 3.28 – 3.26 (m,

4H).

9.3. Deuterium labelling study with Dioxane-d_8.



To a 10 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with **1a** (38.1 mg, 0.15 mmol, 1.5 equiv), **2a** (20.7 mg, 0.2 mmol, 1.0 equiv), $Pd(OAc)_2$ (2.25 mg, 0.01 mmol, 0.1 equiv), K_2CO_3 (34.5 mg, 0.25 mmol, 2.5 equiv), NBE (18.8 mg, 0.2 mmol, 2.0 equiv) and DMSO / Dioxane-d_8 (0.7 mL, v:v=2:5) under air. The reaction was stirred at 70 °C. The reaction was monitored by TLC, after completion of the reaction, the mixture was cooled to r.t., then the mixture was filtered through a thin pad of celite eluting with ethyl acetate (15 mL), and the filtrate was sequentially washed with water, brine, dried over Na_2SO_4. After concentrated *in vacuo*, the residue was purified by column chromatography on silica gel to yield the desired product **3a-*d***.

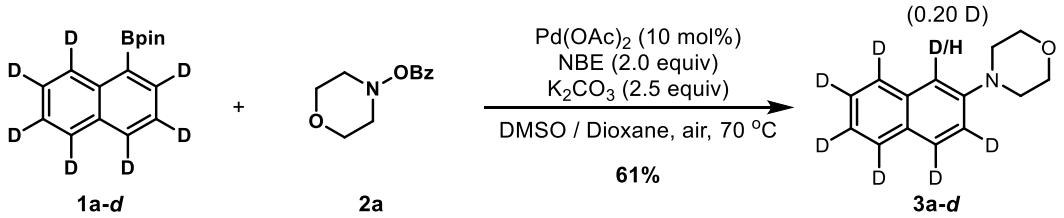
Physical state: yellow solid;

Yield: 61%;

***R*$_f$** = 0.4 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.70 (m, 3H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.13 (s, 1H), 3.94 – 3.91 (m, 4H), 3.28 – 3.26 (m, 4H).

9.4. Deuterium labelling study with 1-naphthylboronic ester-d_7.



To a 25 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with **1a-*d*** (39.2 mg, 0.15 mmol, 1.5 equiv), **2a** (20.7 mg, 0.2 mmol, 1.0 equiv),

$Pd(OAc)_2$ (2.25 mg, 0.01 mmol, 0.1 equiv), K_2CO_3 (34.5 mg, 0.25 mmol, 2.5 equiv), NBE (18.8 mg, 0.2 mmol, 2.0 equiv) and DMSO / Dioxane (1.4 mL, v:v=2:5) under air. The reaction was stirred at 70 °C. The reaction was monitored by TLC, after completion of the reaction, the mixture was cooled to r.t., then the mixture was filtered through a thin pad of celite eluting with ethyl acetate (15 mL), and the filtrate was sequentially washed with water, brine, dried over Na_2SO_4. After concentrated *in vacuo*, the residue was purified by column chromatography on silica gel to yield the desired product **3a-*d***.

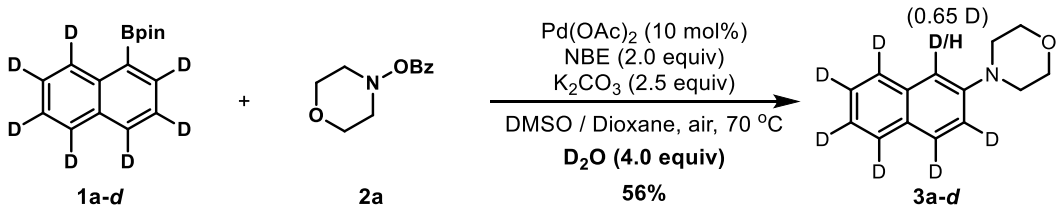
Physical state: yellow solid;

Yield: 61%;

R_f = 0.4 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.13 (s, 0.8H), 3.94 – 3.91 (m, 4H), 3.28 – 3.26 (m, 4H).

9.5. Deuterium labelling study with D_2O.



To a 25 mL oven-dried Schlenk tube equipped with a magnetic stir bar was charged with **1a-*d*** (39.2 mg, 0.15 mmol, 1.5 equiv), **2a** (20.7 mg, 0.2 mmol, 1.0 equiv), $Pd(OAc)_2$ (2.25 mg, 0.01 mmol, 0.1 equiv), K_2CO_3 (34.5 mg, 0.25 mmol, 2.5 equiv), NBE (18.8 mg, 0.2 mmol, 2.0 equiv), DMSO / Dioxane (1.4 mL, v:v=2:5) and D_2O (8 mg, 0.4 mmol, 4.0 equiv) under air. The reaction was stirred at 70 °C. The reaction was monitored by TLC, after completion of the reaction, the mixture was cooled to r.t., then the mixture was filtered through a thin pad of celite eluting with ethyl acetate (15 mL), and the filtrate was sequentially washed with water, brine, dried over Na_2SO_4. After concentrated *in vacuo*, the residue was purified by column chromatography on silica gel to yield the desired product **3a-*d***.

Physical state: yellow solid;

Yield: 56%;

***R**f* = 0.4 (silica gel, PE:EtOAc = 20:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.13 (s, 0.35H), 3.94 – 3.91 (m, 4H), 3.28 – 3.26 (m, 4H).

10. Reference.

1. S. Chen, Z.-S. Liu, T. Yang, Y. Hua, Z. Zhou, H.-G. Cheng, Q. Zhou, *Angew. Chem. Int. Ed.* **2018**, *57*, 7161–7165.
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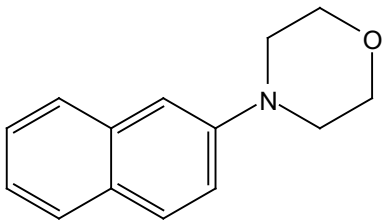
11. NMR spectra of the new compounds.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N

3a

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7.73
7.72
7.70
7.44
7.42
7.40
7.33
7.31
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7.28
7.27
7.26
7.25
7.25
7.13
3.94
3.92
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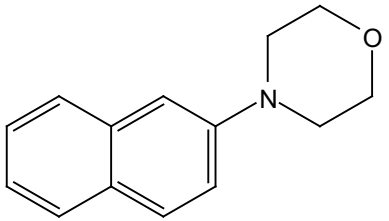
1.19
1.00
4.00
4.06
f1 (ppm)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N

3a

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128.96
128.79
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126.90
126.48
123.68
119.05
110.22
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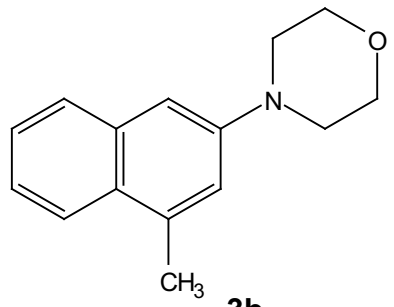
77.16
76.84
67.08
49.94
f1 (ppm)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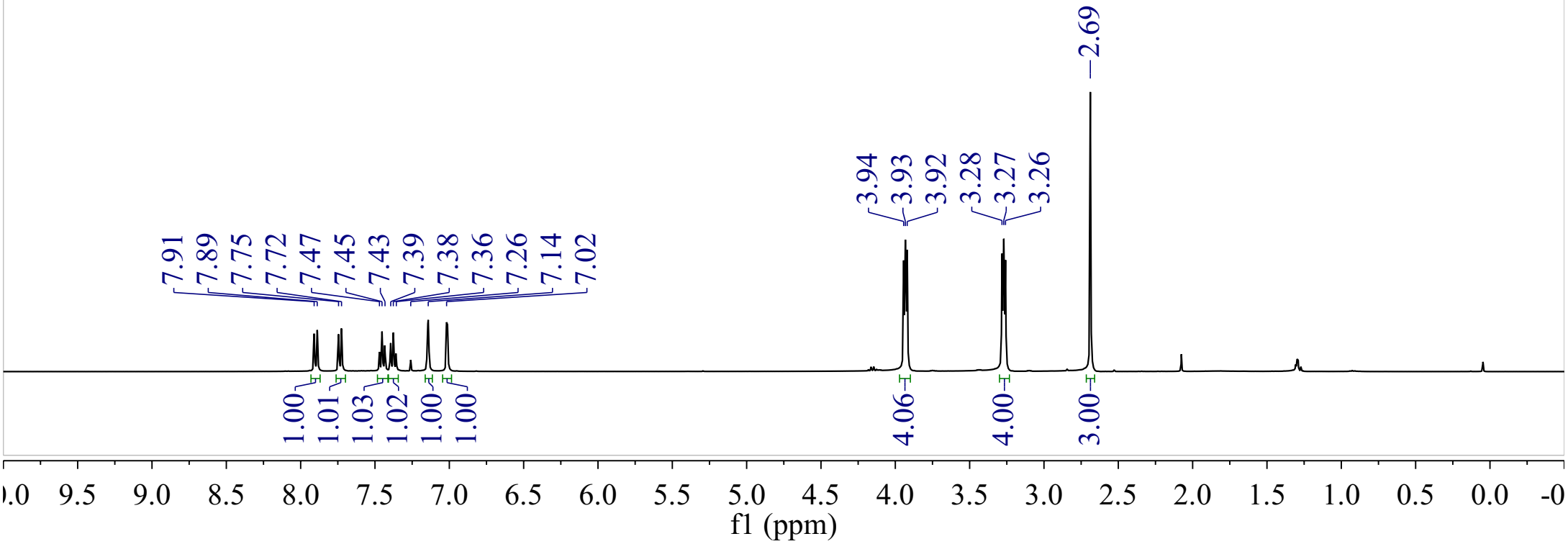
N
CH3

3b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7.39
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7.36
7.26
7.14
7.02
3.94
3.93
3.92
3.28
3.27
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1.02
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1.00
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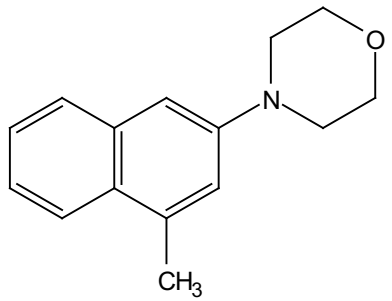
f1 (ppm)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N
CH3

3b

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108.69
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67.06
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19.76
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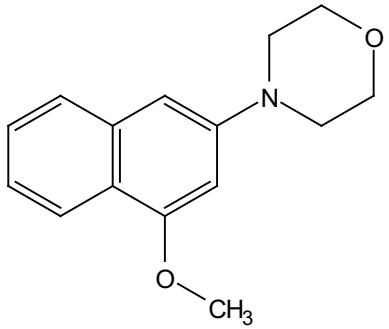
f1 (ppm)



3c

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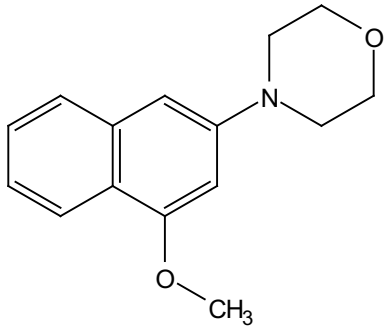
7.42
7.33
7.31
7.30
7.26
6.75
6.60
4.00
3.94
3.93
3.92
3.27
3.26
3.25
1.02
1.00
1.07
1.02
1.00
1.02
3.09
4.05
4.01

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

f1 (ppm)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N
O
CH3

3c

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121.90
121.68
103.22
98.02
77.48
77.16
76.84
67.08
55.53
50.36
190
180
170
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110
100
90
80
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50
40
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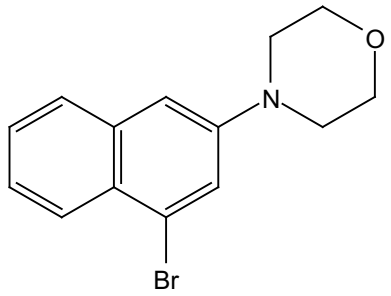
f1 (ppm)


O
N
Br

3d

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8.07
7.68
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7.58
7.58
7.47
7.45
7.45
7.43
7.43
7.42
7.42
7.40
7.40
7.39
7.26
7.08
7.07
3.91
3.90
3.89
3.26
3.25
3.23
1.00
1.04
1.02
2.12
1.04
4.17
4.10
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
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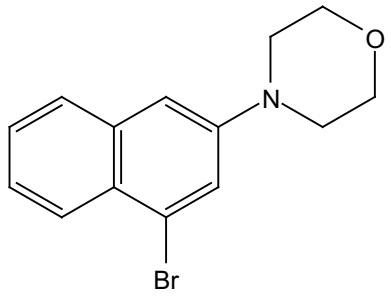
0.0
f1 (ppm)


O
N
Br

3d

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77.16
76.84
66.92
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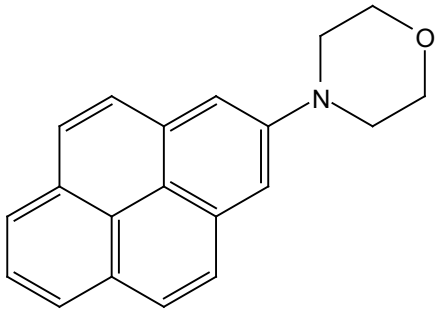
f1 (ppm)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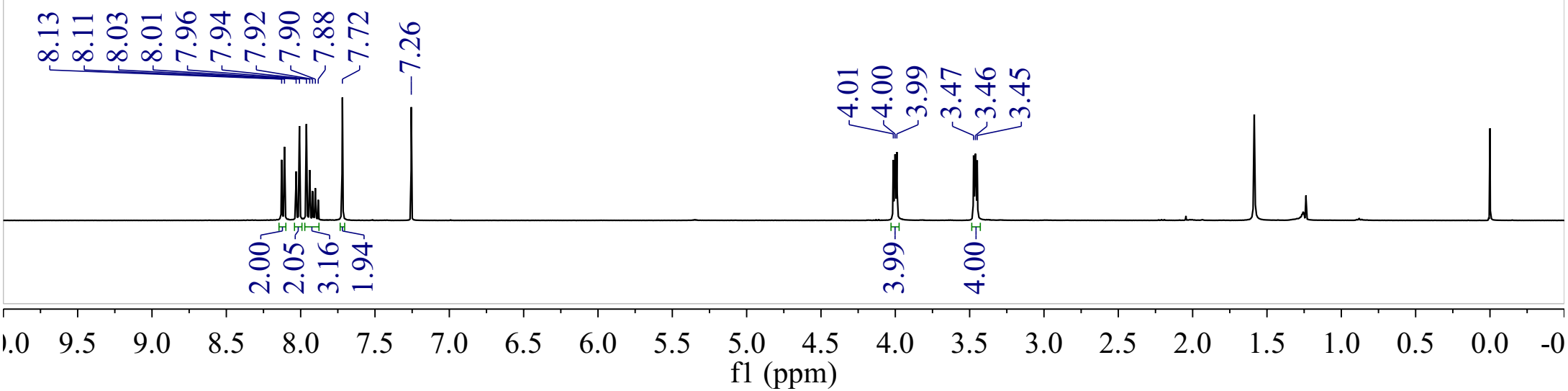
N

3e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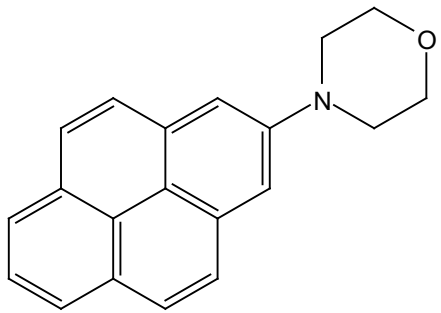
f1 (ppm)


O
N

3e

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119.87
112.67
67.23
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190
180
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50
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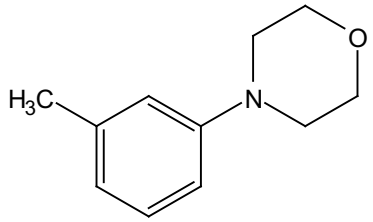
0
f1 (ppm)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N
O

3f

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6.73
6.72
6.71
3.88
3.87
3.86
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3.16
3.14
2.34
1.00
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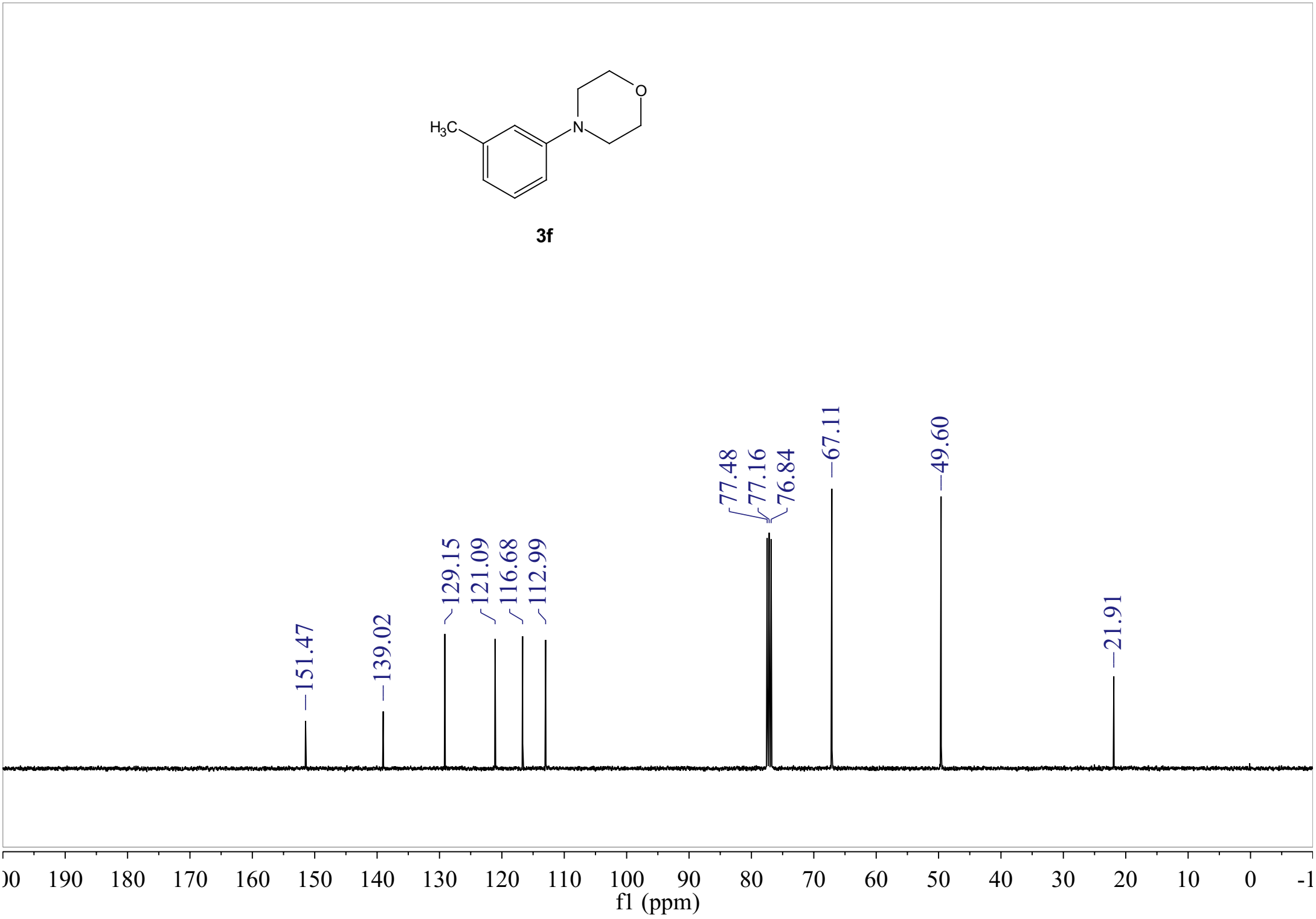
4.01
4.07
3.03
f1 (ppm)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N
O
3f
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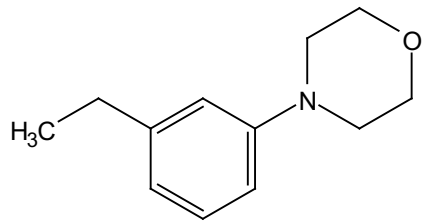
H3C
N
O

3g

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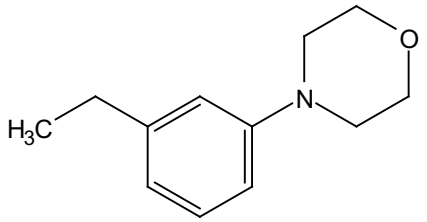
1.25
1.23
1.00
3.06
4.06
4.07
2.09
3.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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N
O

3g

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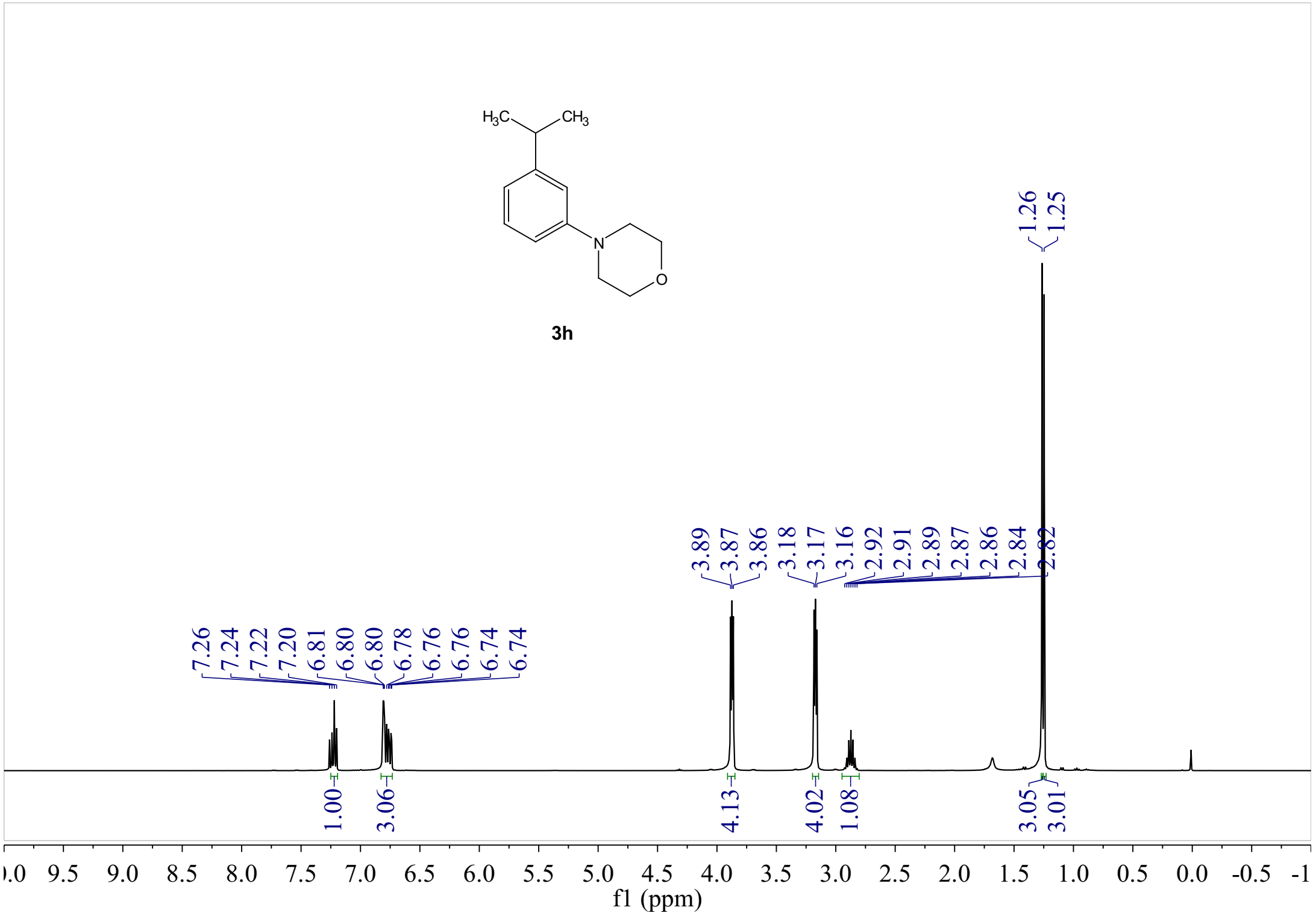
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N
O
3h
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6.80
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6.76
6.76
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6.74
3.89
3.87
3.86
3.18
3.17
3.16
2.92
2.91
2.89
2.87
2.86
2.84
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3.06
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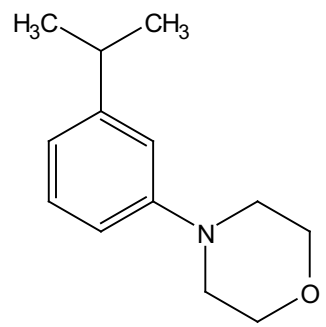
1.08
3.05
3.01
f1 (ppm)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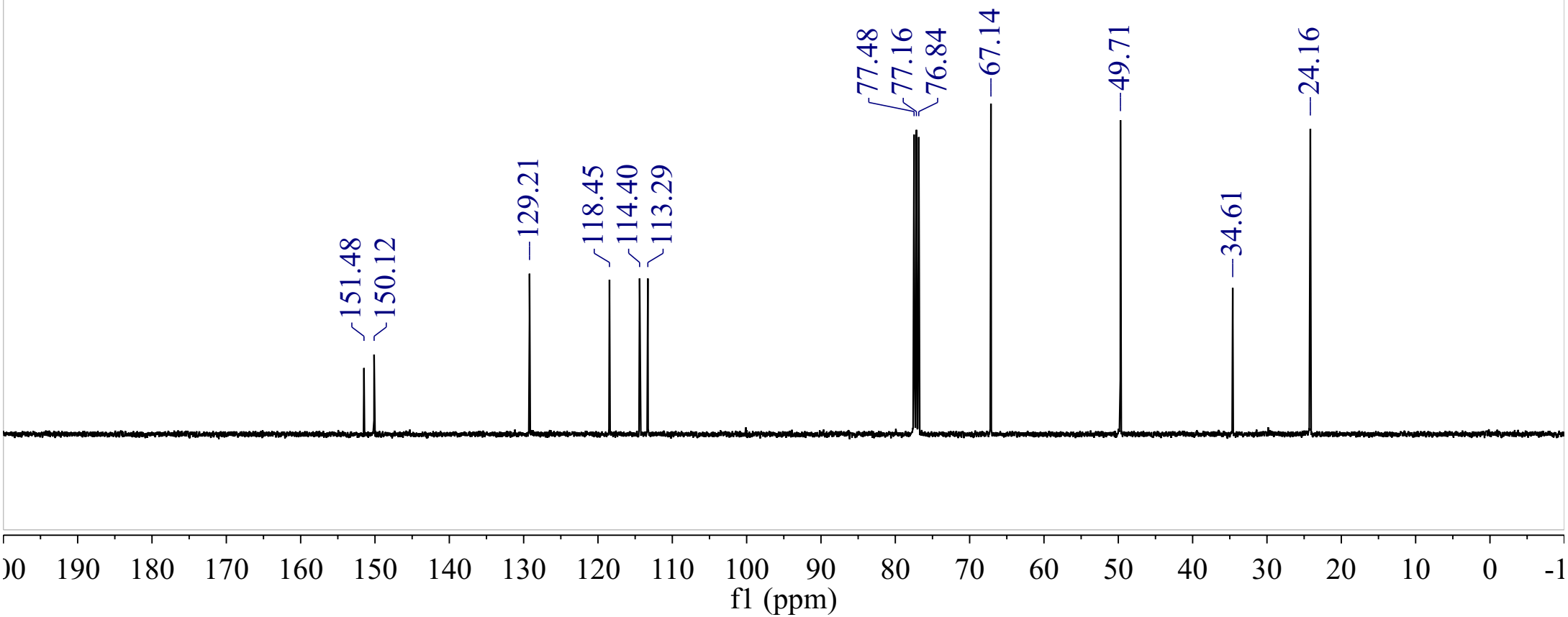
CH3
N
O

3h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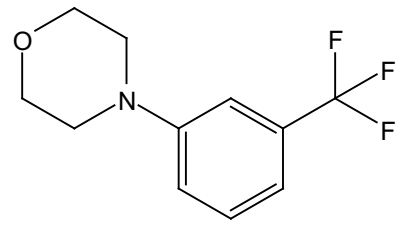
24.16
f1 (ppm)


O
N
F
F
F

3i

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7.06
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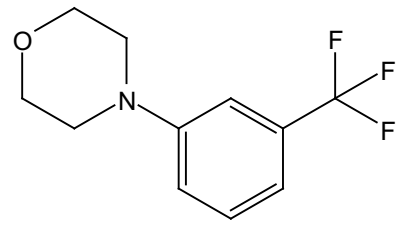
4.07
f1 (ppm)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N
F
F
F

3i

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123.05
120.34
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118.57
116.43
116.39
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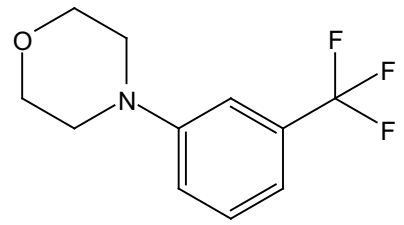
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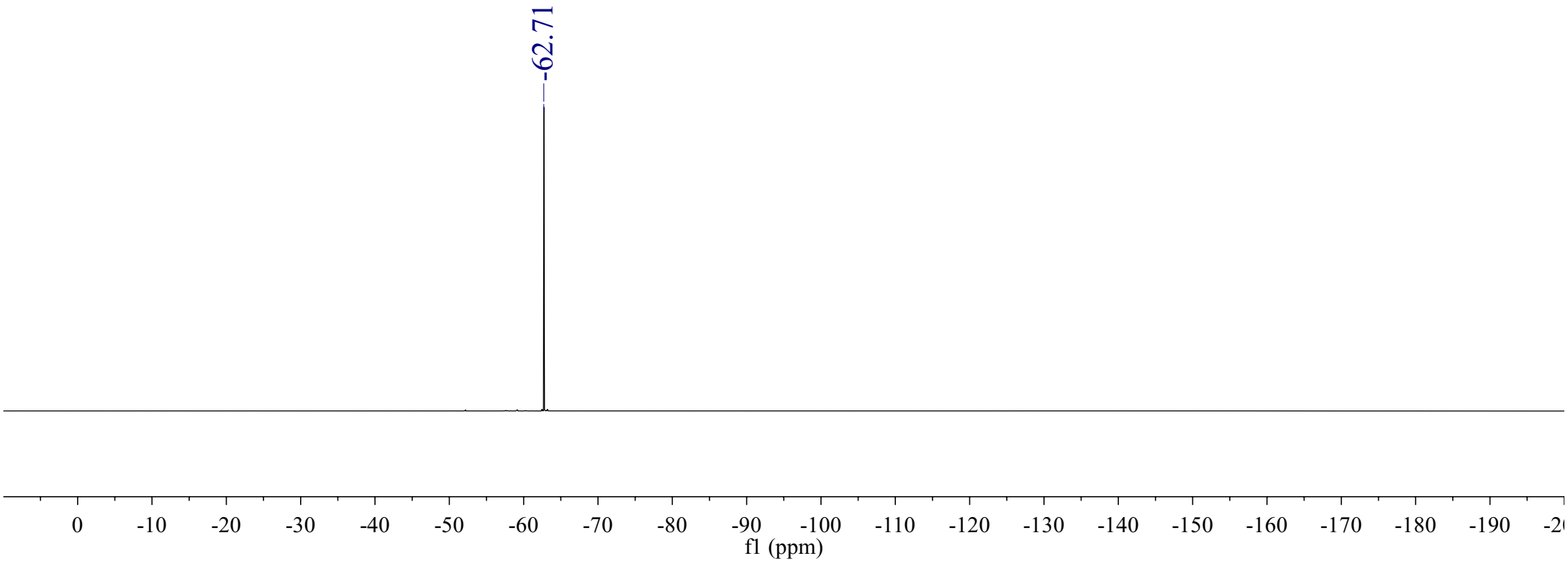
N
F
F
F

3i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-140
-150
-160
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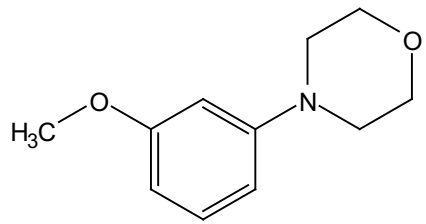
-180
-190
f1 (ppm)


O
N
O
H3C

3j

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7.21
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7.20
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7.19
7.18
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6.46
6.46
6.45
6.44
3.87
3.87
3.85
3.85
3.84
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3.15
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4.06
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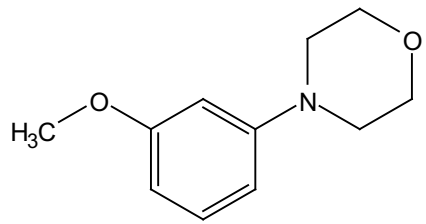
4.03
f1 (ppm)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N
O
H3C

3j

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76.84
67.01
55.31
49.40
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50
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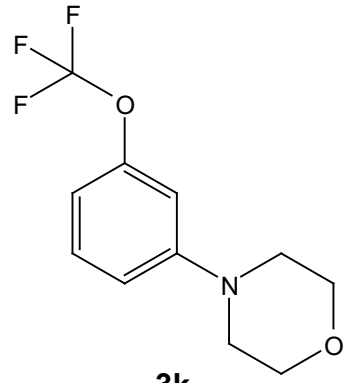
f1 (ppm)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F
F
O
N
O

3k

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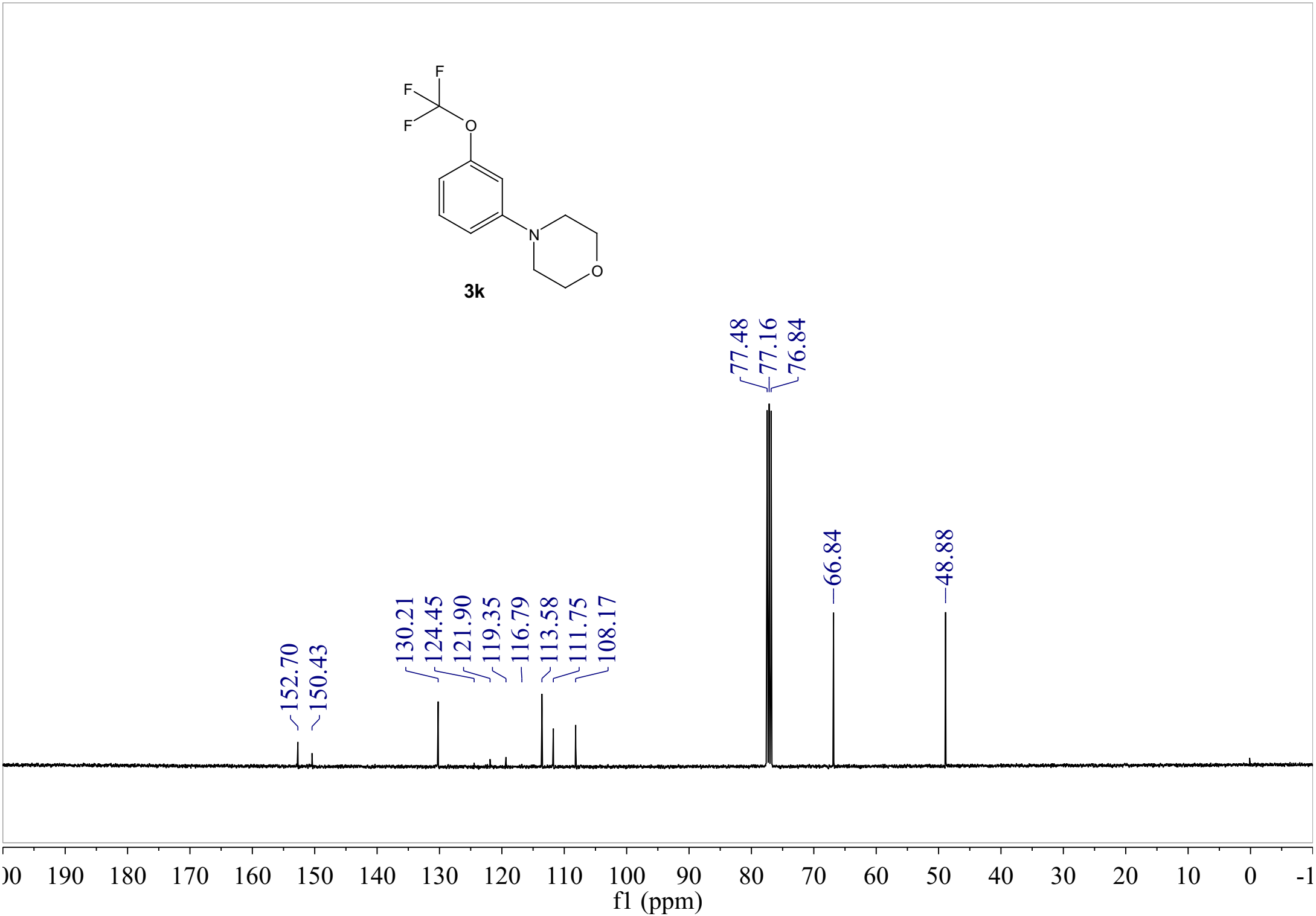
6.71
3.87
3.86
3.85
3.18
3.17
3.16
1.3
1.0
2.0
4.1
4.1
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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F
F
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N
O
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124.45
121.90
119.35
116.79
113.58
111.75
108.17
190
180
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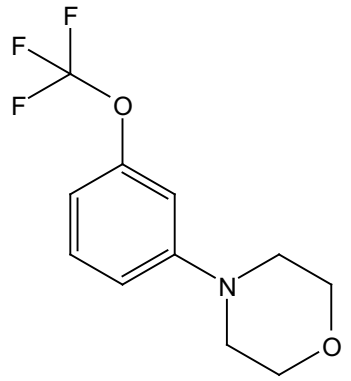
0
f1 (ppm)



3k

-57.47

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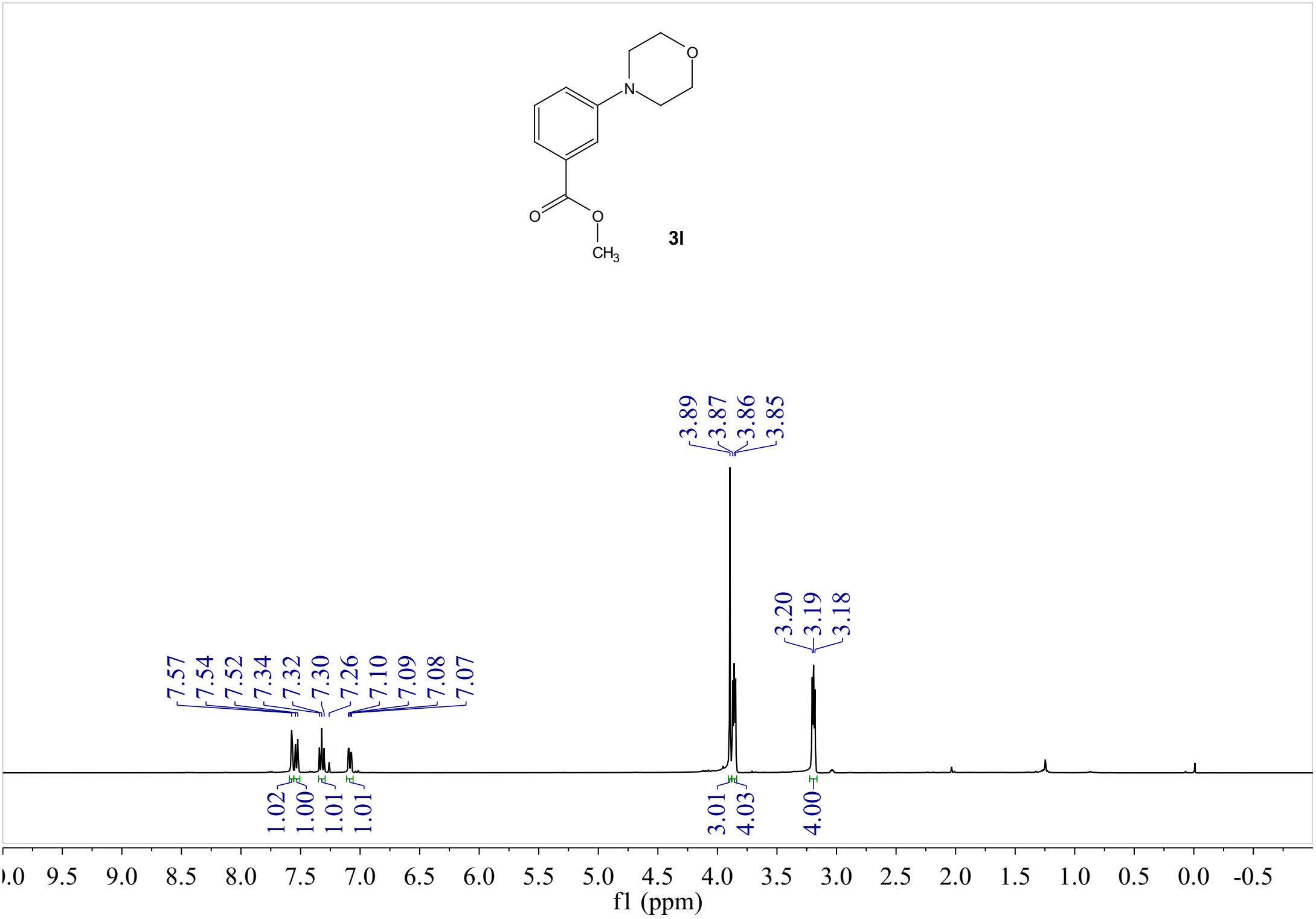
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3.18
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7.10
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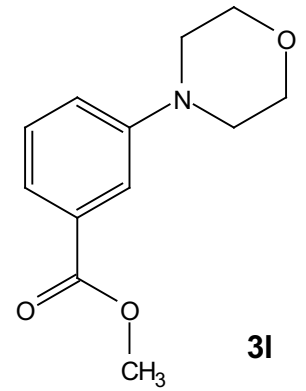
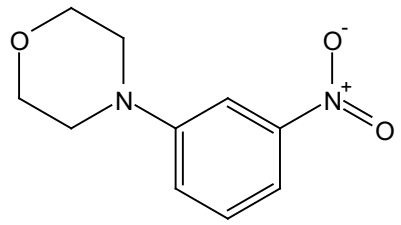
7.08
7.07
1.02
1.00
1.01
1.01
3.01
4.03
4.00
f1 (ppm)


O
N
O
O
CH3

3l

167.41
151.33
131.08
129.25
121.05
120.08
116.43
77.48
77.16
76.84
66.89
52.22
49.17
f1 (ppm)


O
N
O-
N+
O

3m

7.72
7.71
7.70
7.70
7.70
7.70
7.68
7.68
7.68
7.67
7.42
7.40
7.38
7.26
7.19
7.19
7.17
7.17
3.89
3.88
3.87
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3.25
3.24
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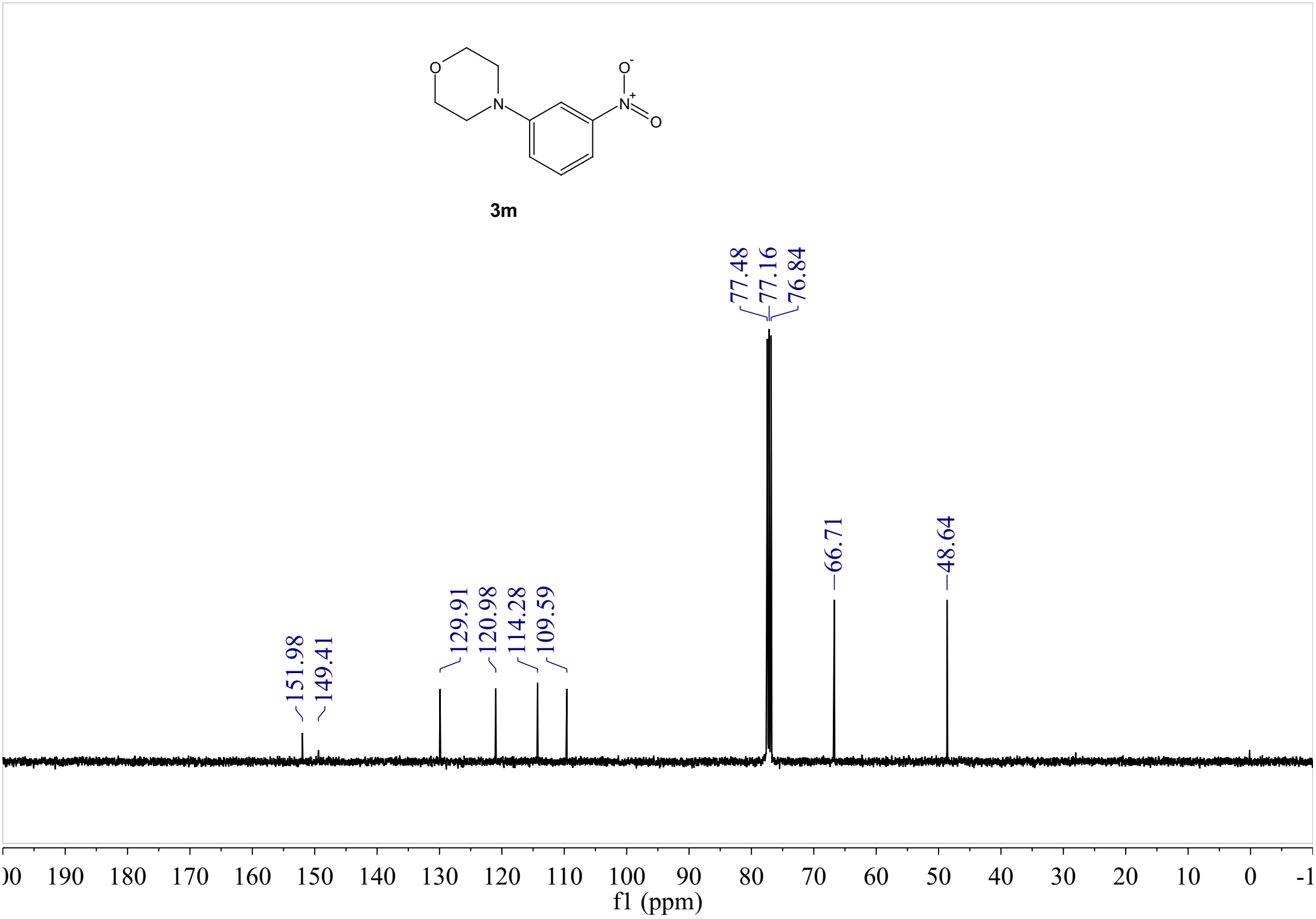
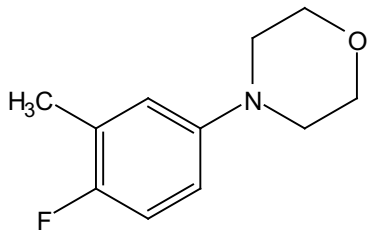
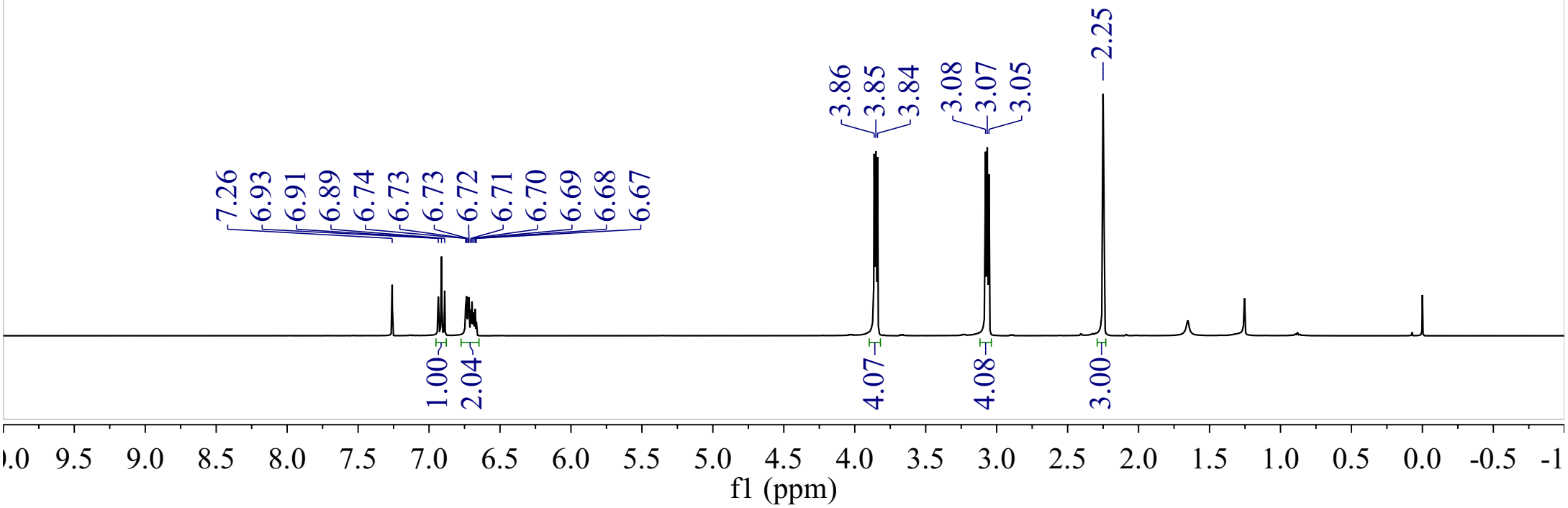
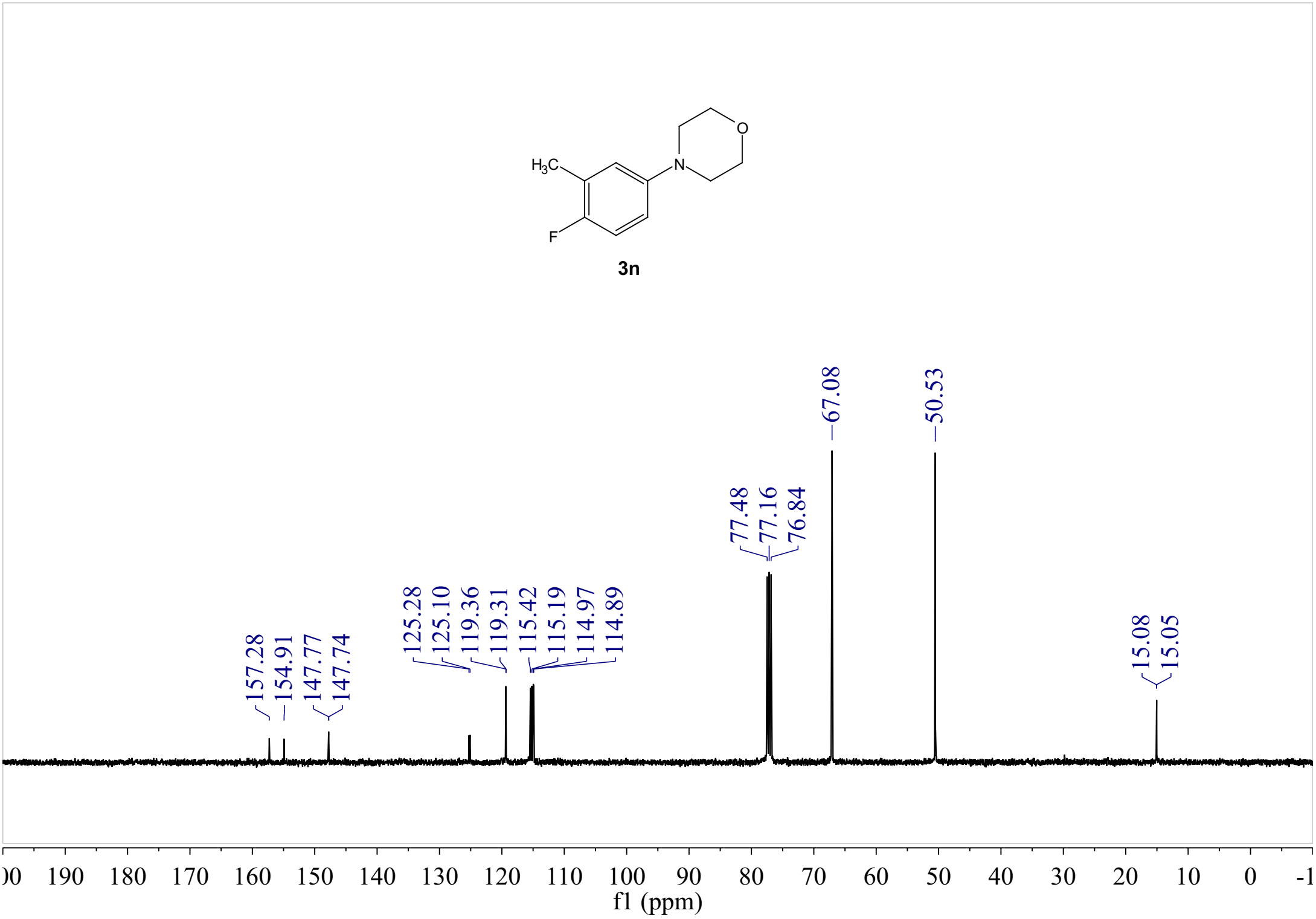
2.00
1.00
1.02
4.02
4.00
f1 (ppm)

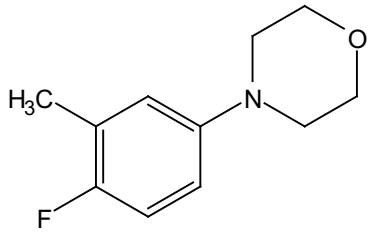

3m
77.48
77.16
76.84
66.71
48.64
151.98
149.41
129.91
120.98
114.28
109.59
f1 (ppm)


H3C
N
O
F

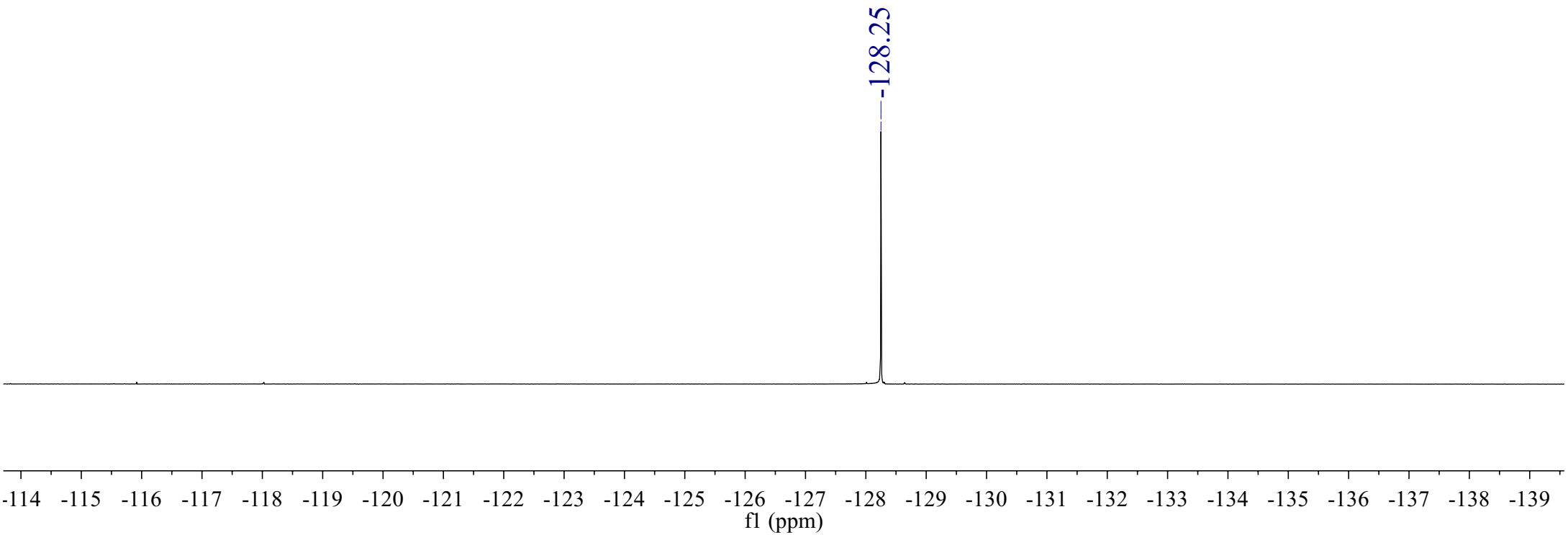
3n

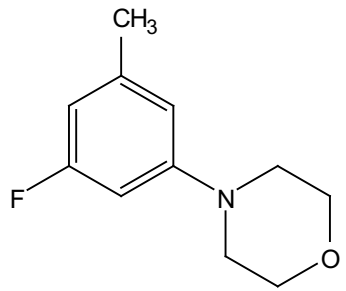

7.26
6.93
6.91
6.89
6.74
6.73
6.73
6.72
6.71
6.70
6.69
6.68
6.67
3.86
3.85
3.84
3.08
3.07
3.05
2.25
1.00
2.04
4.07
4.08
3.00
f1 (ppm)


H3C
F
N
O
3n
157.28
154.91
147.77
147.74
125.28
125.10
119.36
119.31
115.42
115.19
114.97
114.89
77.48
77.16
76.84
67.08
50.53
15.08
15.05
f1 (ppm)


H3C
N
O
F

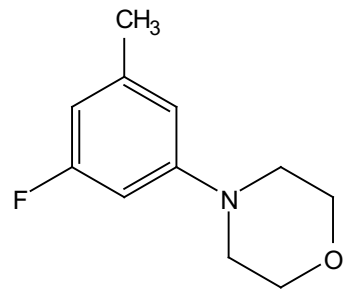
3n


-128.25
-114 -115 -116 -117 -118 -119 -120 -121 -122 -123 -124 -125 -126 -127 -128 -129 -130 -131 -132 -133 -134 -135 -136 -137 -138 -139
f1 (ppm)


CH3
F
N
O

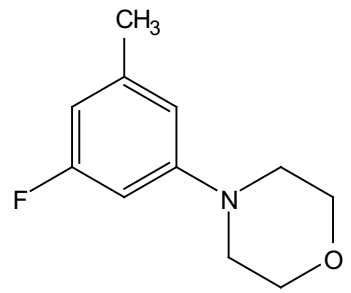
3o

7.26
6.49
6.41
6.39
3.85
3.84
3.83
3.15
3.14
3.13
2.30
1.00
1.00
1.06
4.04
4.03
3.02
f1 (ppm)

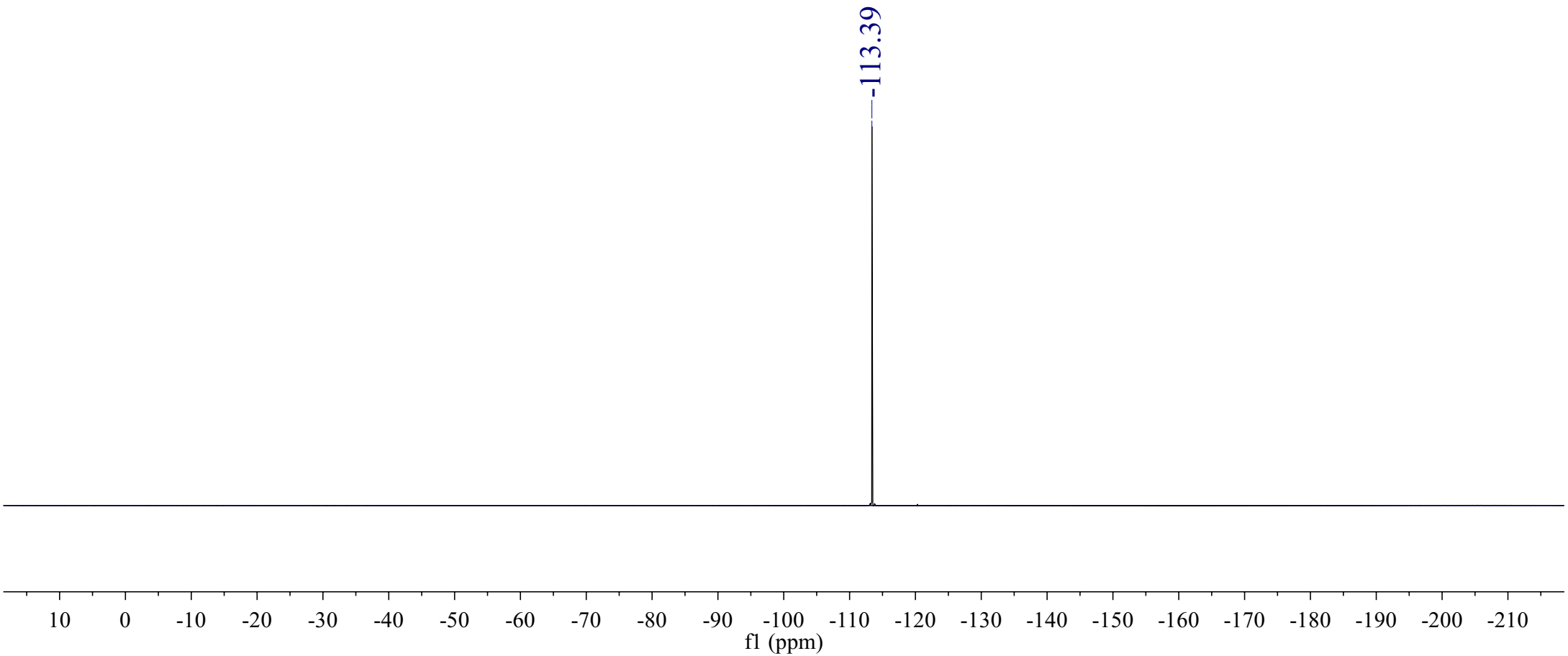

CH3
F
N
O

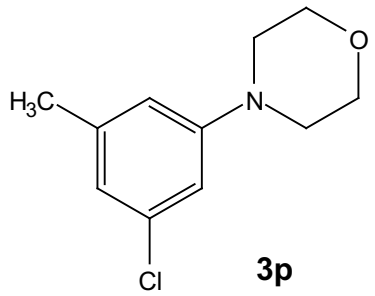
3o

165.11
162.70
152.85
152.74
140.78
140.68
111.71
111.69
107.40
107.18
99.91
99.66
77.48
77.16
76.84
66.89
49.06
21.95
21.93
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


CH3
F
N
O

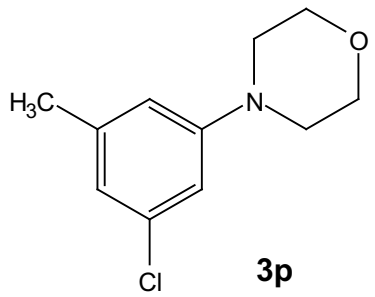
3o


-113.39
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


H3C
N
O
Cl

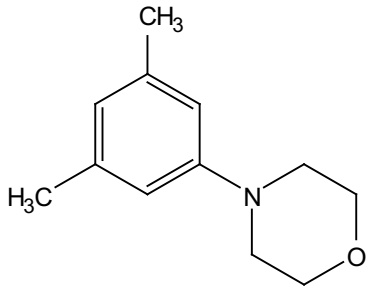
3p

7.26
6.69
6.68
6.59
3.85
3.84
3.83
3.15
3.13
3.12
2.29
2.00
1.00
4.08
4.07
3.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

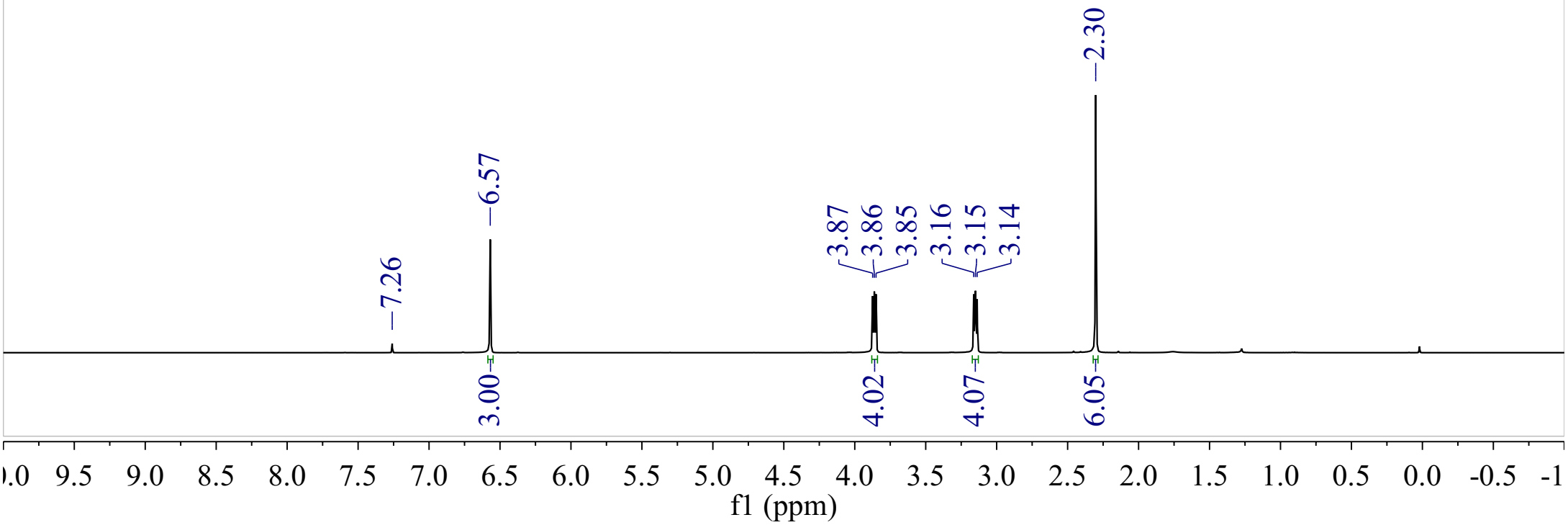

H3C
N
O
Cl

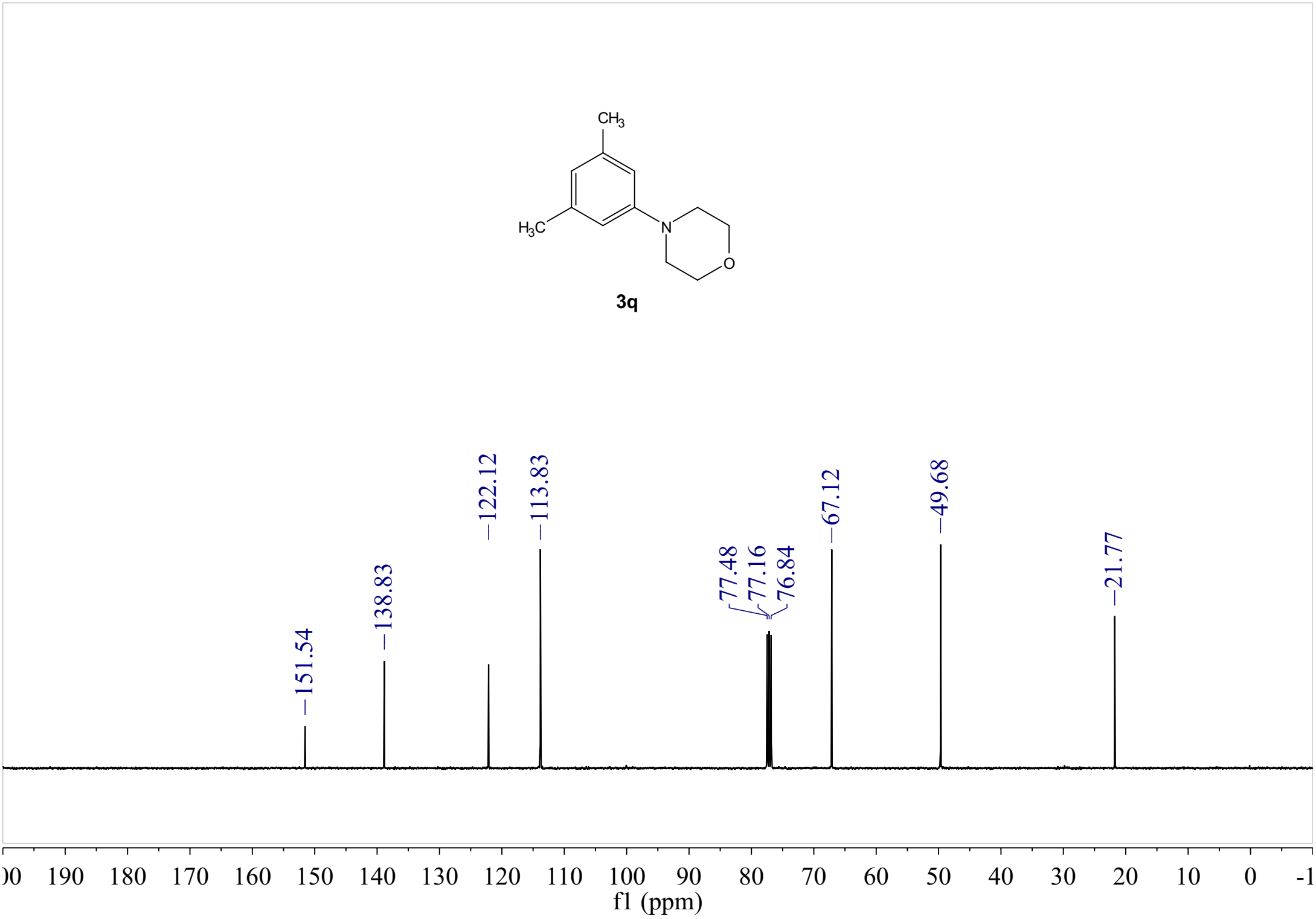
3p

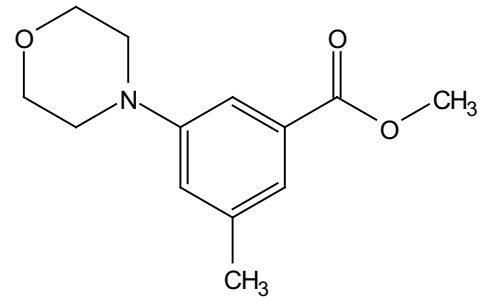
152.34
140.40
134.82
120.69
114.60
112.88
77.48
77.16
76.84
66.89
49.12
21.74
f1 (ppm)


CH3
H3C
N
O

3q

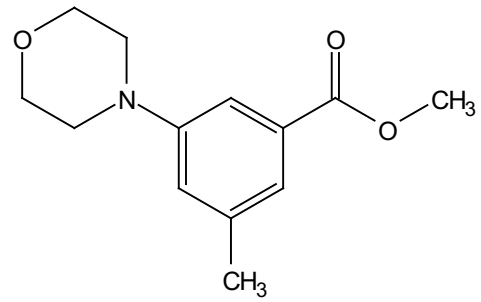

7.26
6.57
3.87
3.86
3.85
3.16
3.15
3.14
2.30
3.00
4.02
4.07
6.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


CH3
H3C
N
O
3q
151.54
138.83
122.12
113.83
77.48
77.16
76.84
67.12
49.68
21.77
f1 (ppm)


O
N
O
O
CH3
CH3

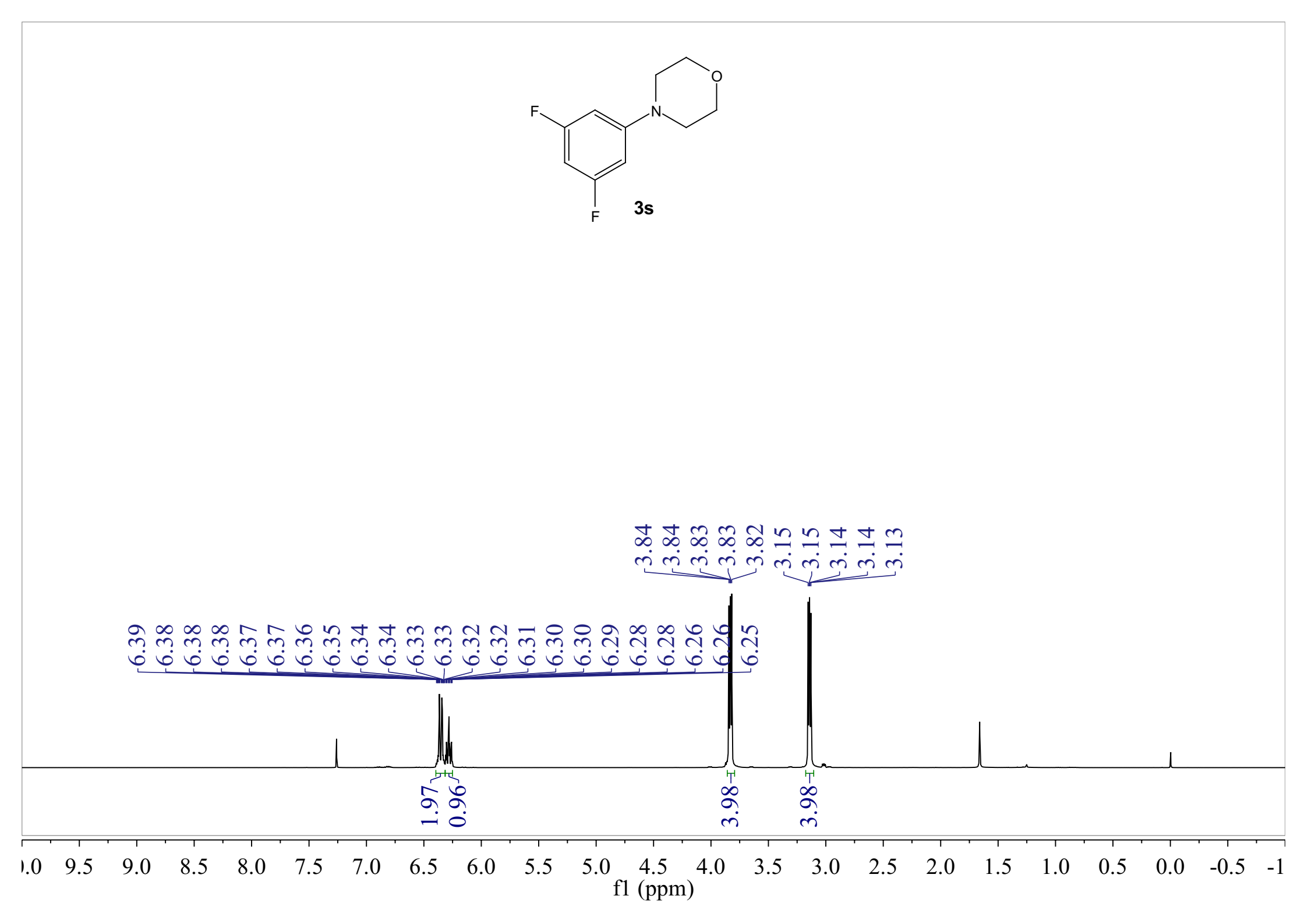
3r

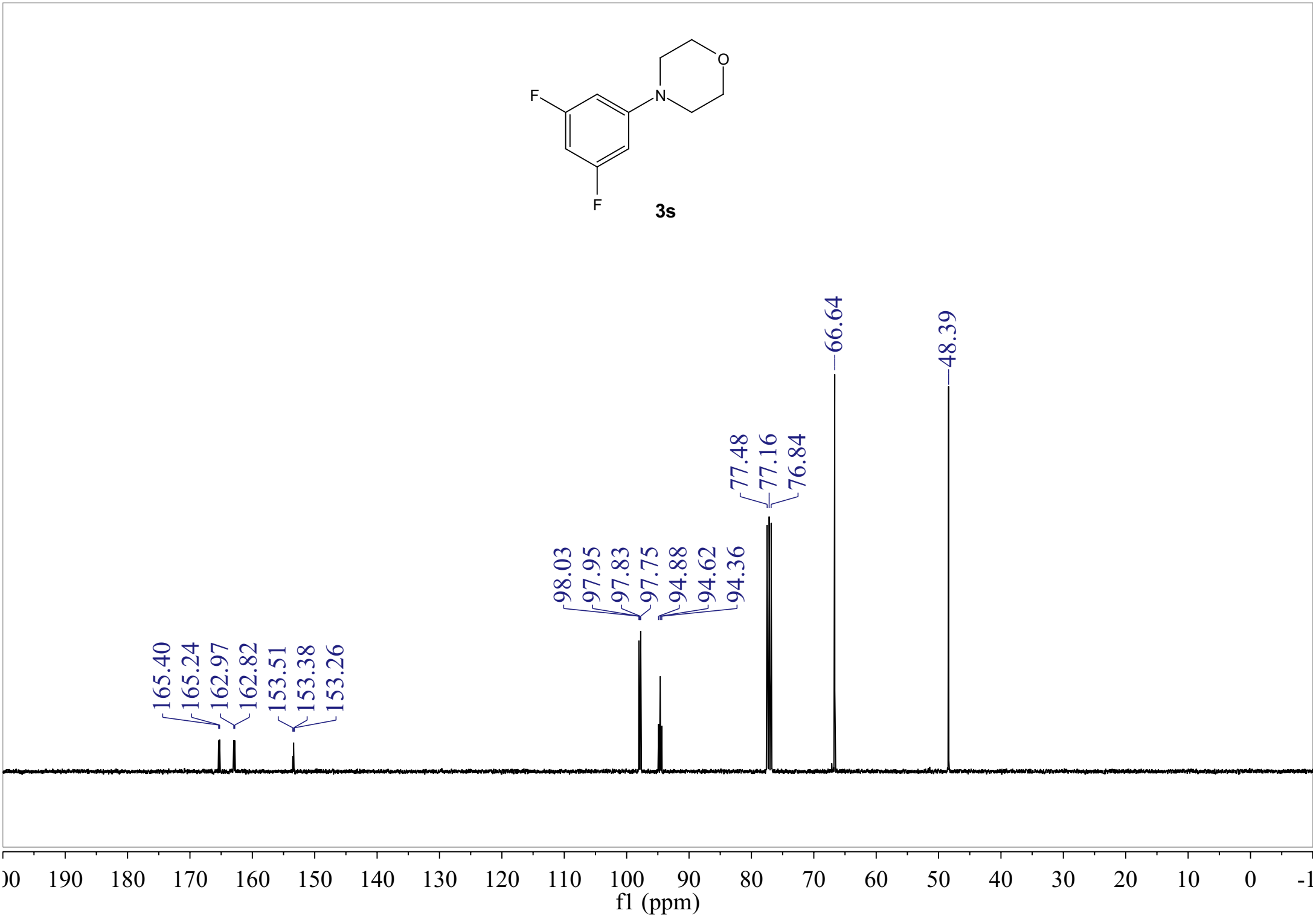
7.68
7.66
7.26
7.24
7.22
3.89
3.86
3.85
3.84
2.94
2.93
2.92
2.36
2.03
1.00
3.09
4.09
4.06
3.01
f1 (ppm)

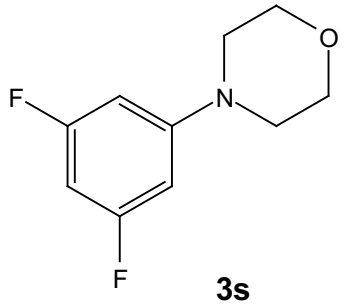

O
N
O
O
CH3
CH3

3r

167.29
151.40
138.46
131.28
128.77
124.73
120.20
77.48
77.16
76.84
67.43
52.26
52.13
18.32
f1 (ppm)


F
N
O
F
3s
3.84
3.84
3.83
3.83
3.82
3.15
3.15
3.14
3.14
3.13
6.39
6.38
6.38
6.38
6.37
6.37
6.36
6.35
6.34
6.34
6.33
6.33
6.32
6.32
6.31
6.30
6.30
6.29
6.28
6.28
6.26
6.26
6.25
1.97
0.96
3.98
3.98
f1 (ppm)


F
N
O
F
3s
165.40
165.24
162.97
162.82
153.51
153.38
153.26
98.03
97.95
97.83
97.75
94.88
94.62
94.36
77.48
77.16
76.84
66.64
48.39
f1 (ppm)

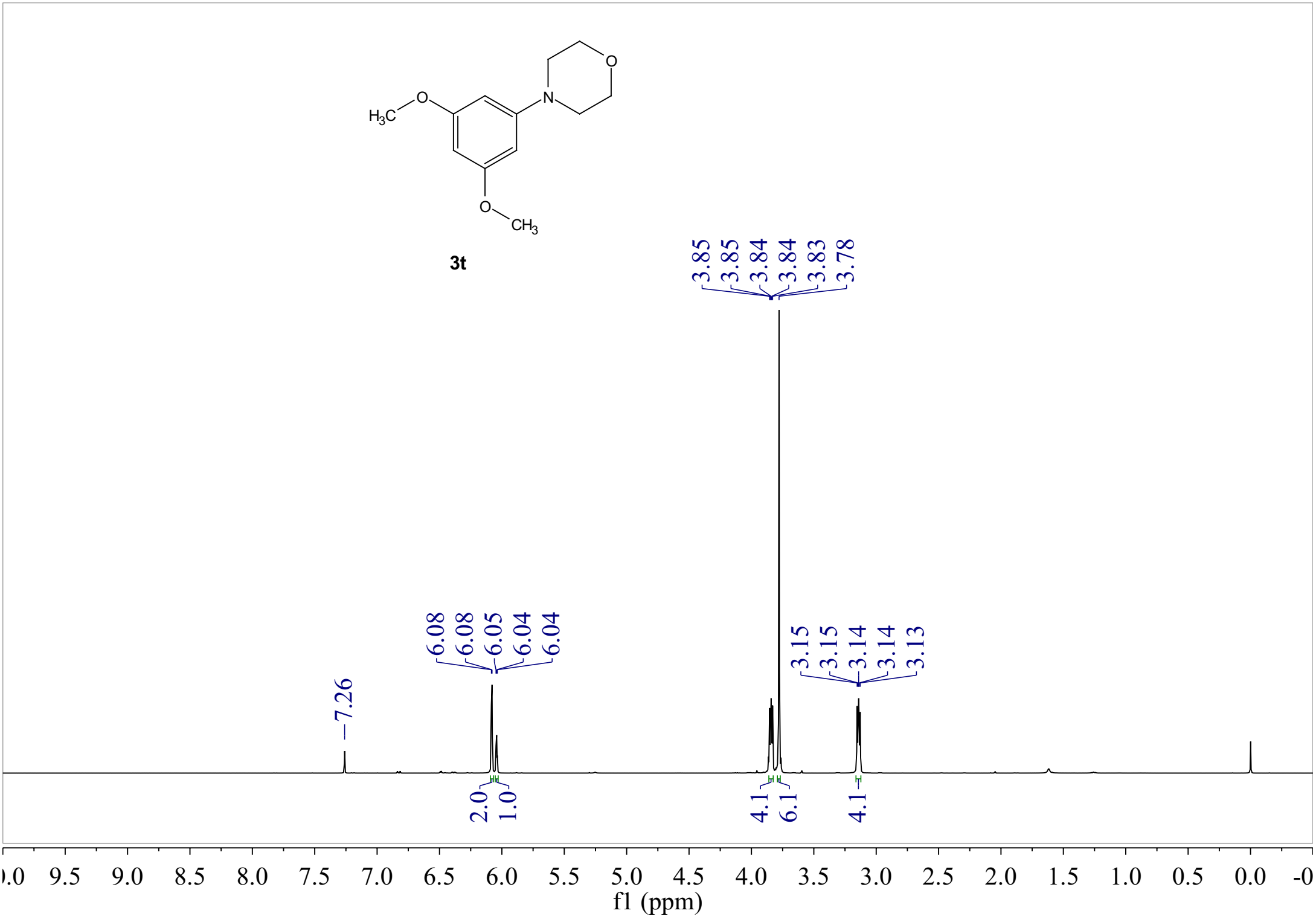


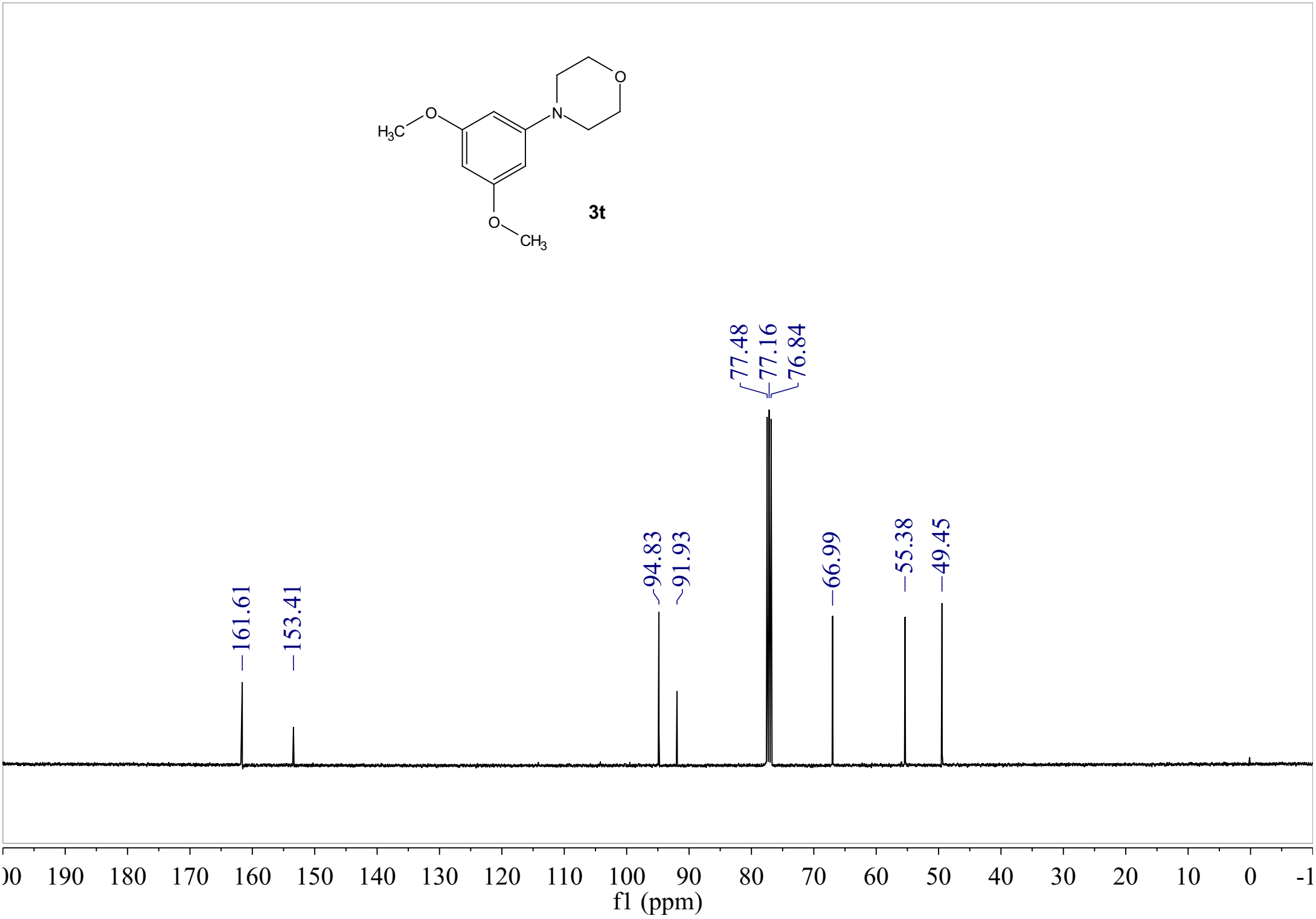
3s

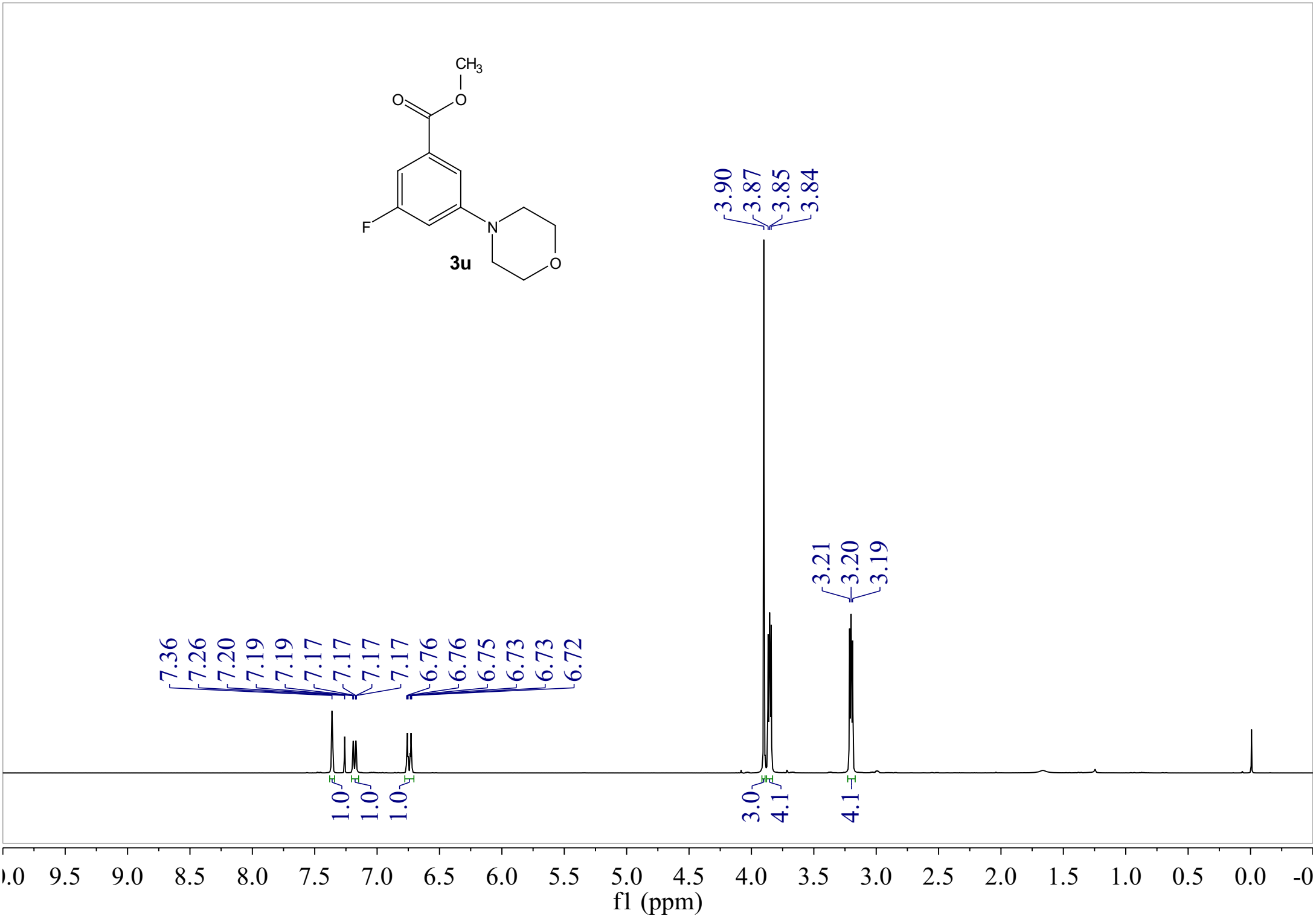
-118.99

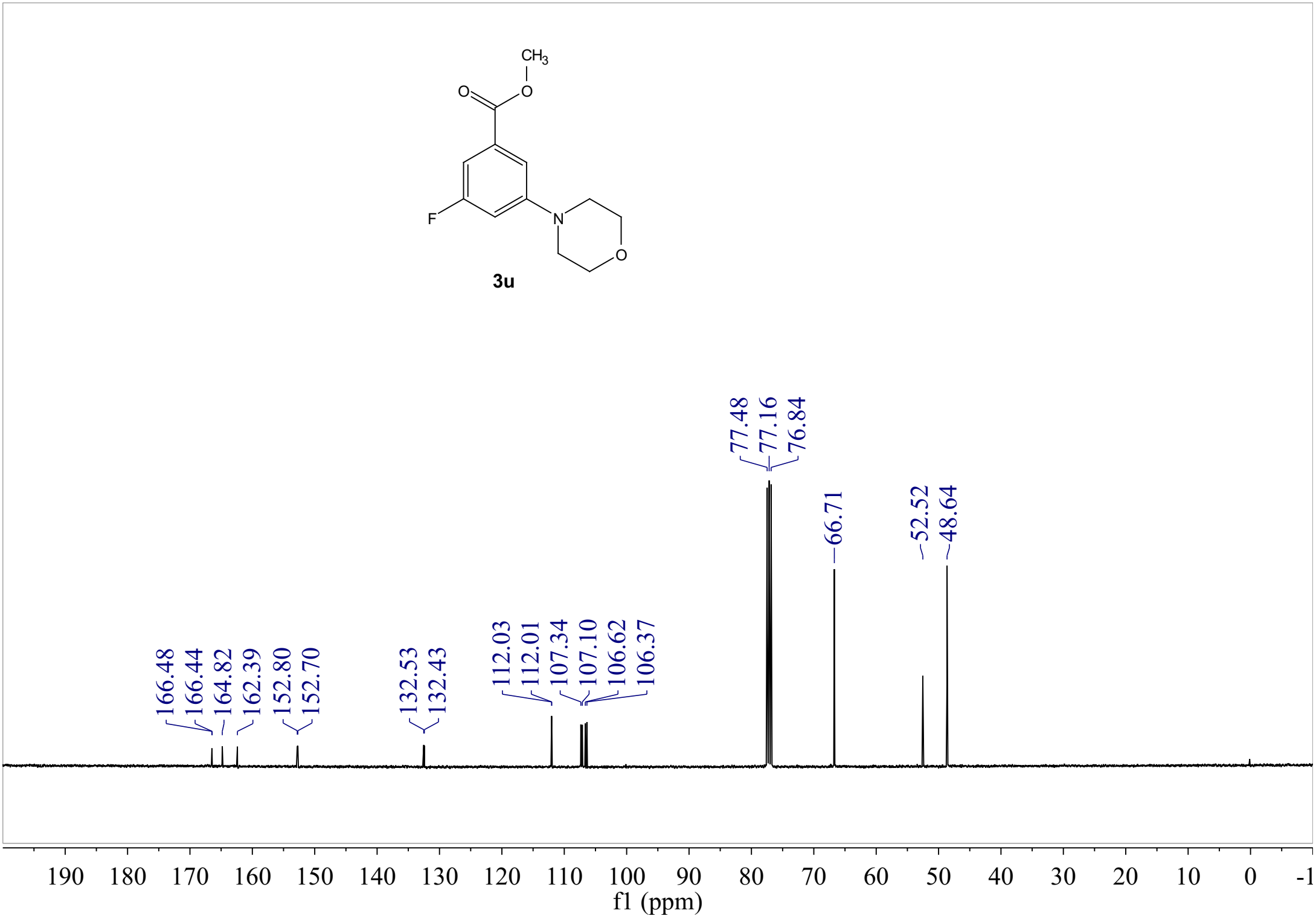
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

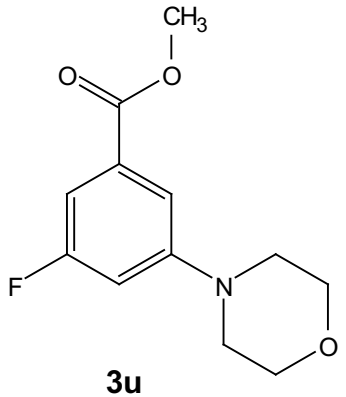
f1 (ppm)


3t
3.85
3.85
3.84
3.84
3.83
3.78
6.08
6.08
6.05
6.04
6.04
7.26
3.15
3.15
3.14
3.14
3.13
2.0
1.0
4.1
6.1
4.1
f1 (ppm)


O
N
O
H3C
O
CH3
3t
161.61
153.41
94.83
91.93
77.48
77.16
76.84
66.99
55.38
49.45
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


CH3
O
O
F
N
O
3u
3.90
3.87
3.85
3.84
3.21
3.20
3.19
7.36
7.26
7.20
7.19
7.19
7.17
7.17
7.17
7.17
6.76
6.76
6.75
6.73
6.73
6.72
1.0
1.0
1.0
3.0
4.1
4.1
f1 (ppm)


CH3
O
O
F
N
O
3u
166.48
166.44
164.82
162.39
152.80
152.70
132.53
132.43
112.03
112.01
107.34
107.10
106.62
106.37
77.48
77.16
76.84
66.71
52.52
48.64
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

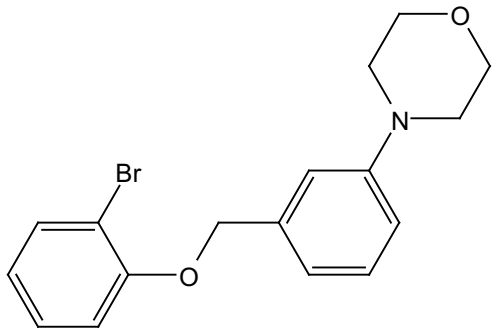


3u

-111.37

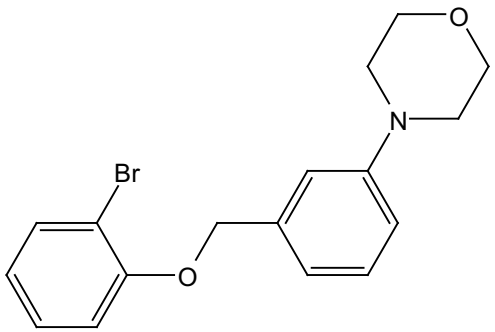
100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300

f1 (ppm)


Br
O
N
O

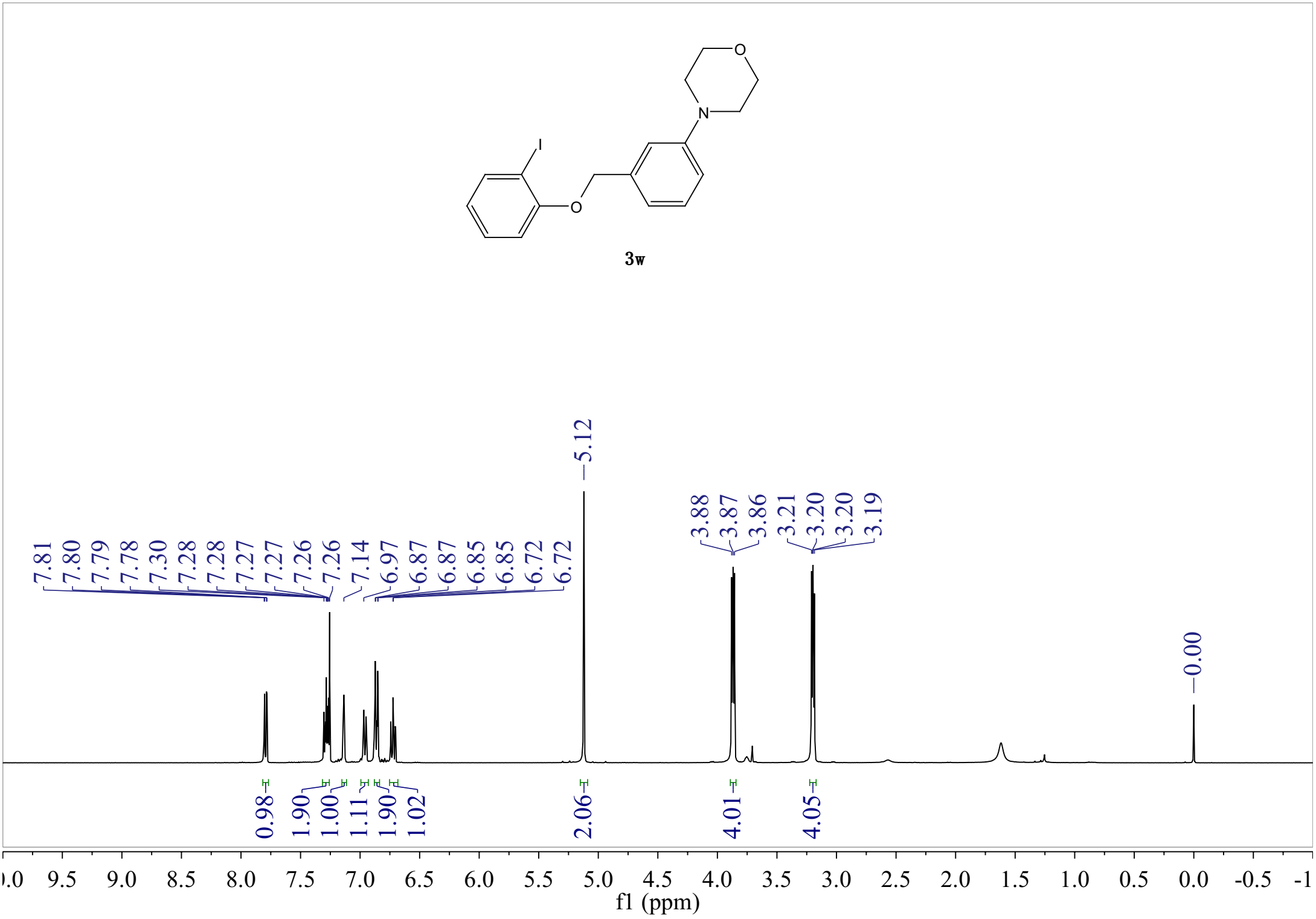
3v

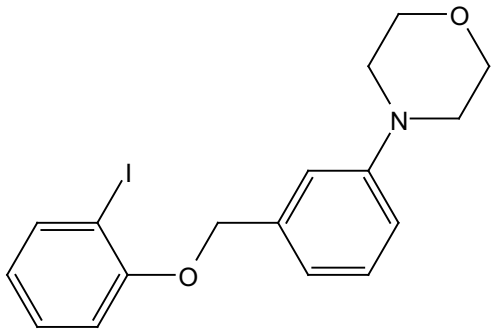
7.57
7.57
7.55
7.55
7.29
7.27
7.26
7.22
7.09
6.95
6.95
6.93
6.93
6.86
6.85
6.85
6.85
5.13
3.88
3.87
3.86
3.20
3.19
3.18
1.00
2.50
1.04
2.03
2.07
2.07
4.02
4.03
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


O
N
Br
O

3v

155.12
151.65
137.74
133.51
129.47
128.57
122.27
118.52
115.23
114.10
114.03
112.60
77.48
77.16
76.84
70.99
67.03
49.32
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
N
I
O
3w
7.81
7.80
7.79
7.78
7.30
7.28
7.28
7.27
7.27
7.26
7.26
7.14
6.97
6.87
6.87
6.85
6.85
6.72
6.72
5.12
3.88
3.87
3.86
3.21
3.20
3.20
3.19
0.00
0.98
1.90
1.00
1.11
1.90
1.02
2.06
4.01
4.05
f1 (ppm)

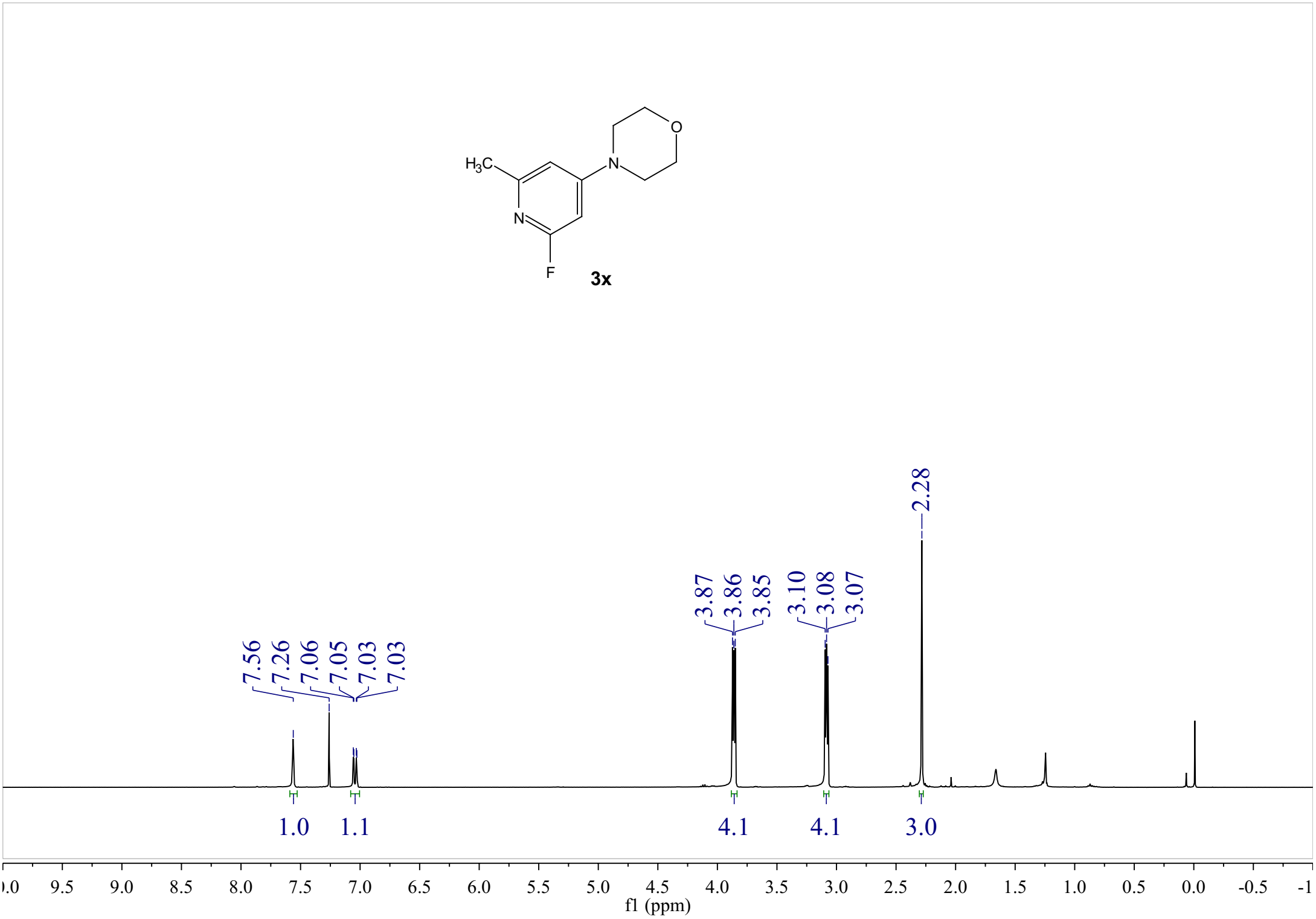


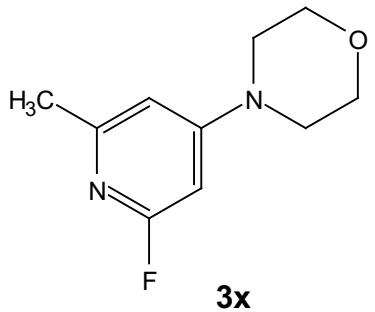
3w

157.27
151.65
139.62
137.72
129.58
129.42
122.93
118.44
115.20
114.15
112.88
86.96
77.48
77.16
76.84
70.98
67.04
49.38

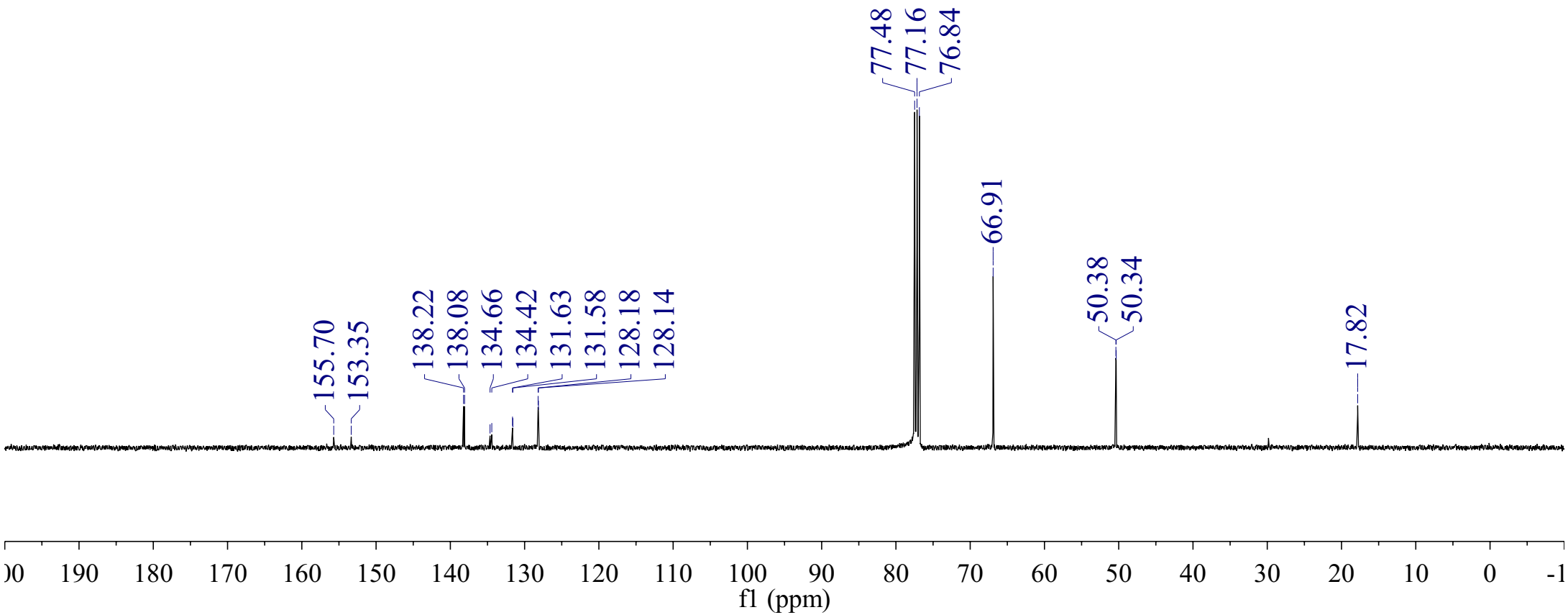
)0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

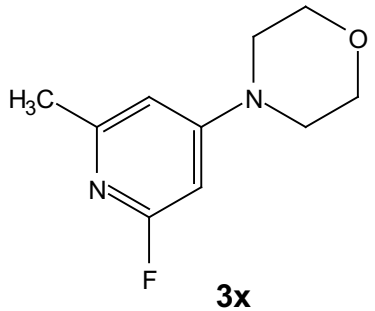
f1 (ppm)


H3C
N
O
N
F
3x
7.56
7.26
7.06
7.05
7.03
7.03
3.87
3.86
3.85
3.10
3.08
3.07
2.28
1.0
1.1
4.1
4.1
3.0
f1 (ppm)

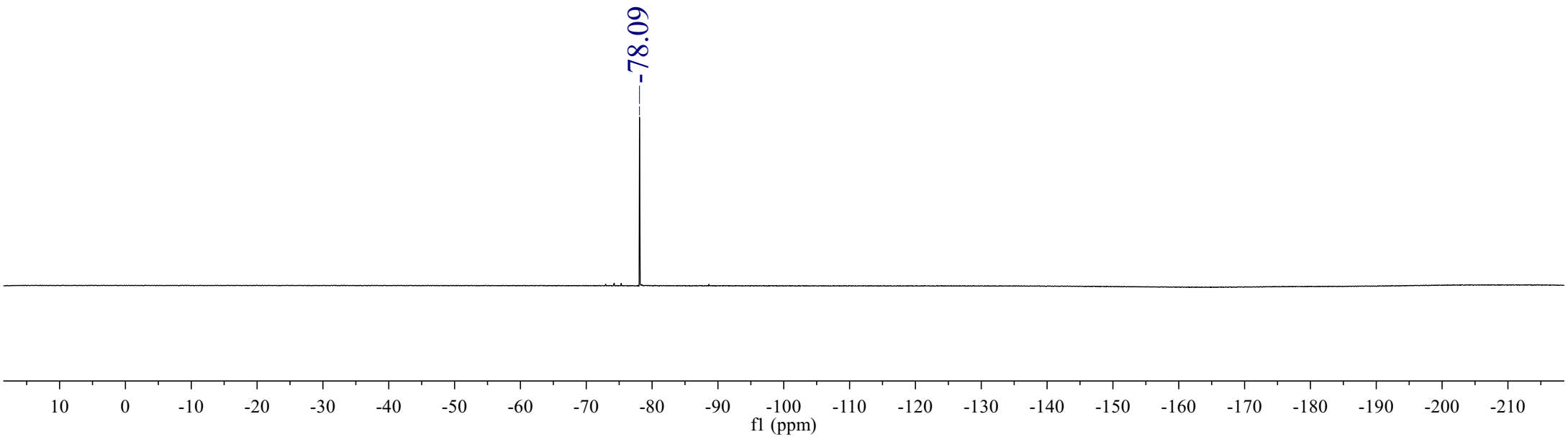

H3C
N
O
N
F

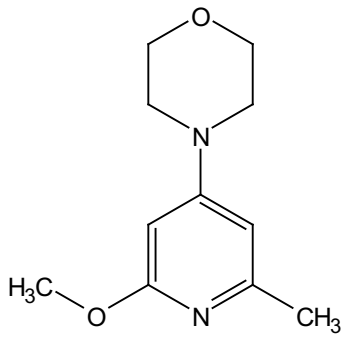
3x


155.70
153.35
138.22
138.08
134.66
134.42
131.63
131.58
128.18
128.14
77.48
77.16
76.84
66.91
50.38
50.34
17.82
f1 (ppm)


H3C
N
O
N
F

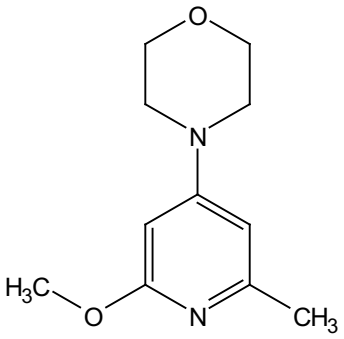
3x


-78.09
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


O
N
H3C
O
N
CH3

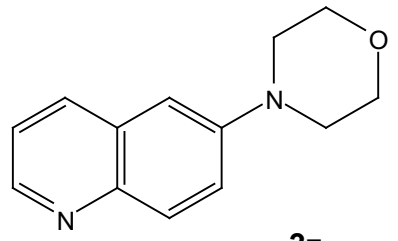
3y

7.26
6.25
6.25
5.89
5.88
3.88
3.81
3.80
3.79
3.23
3.22
3.21
2.36
1.0
1.0
3.0
4.0
4.1
3.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


O
N
H3C
O
N
CH3

3y

165.56
158.57
156.58
102.90
90.34
77.48
77.16
76.84
66.61
53.42
46.89
24.73
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

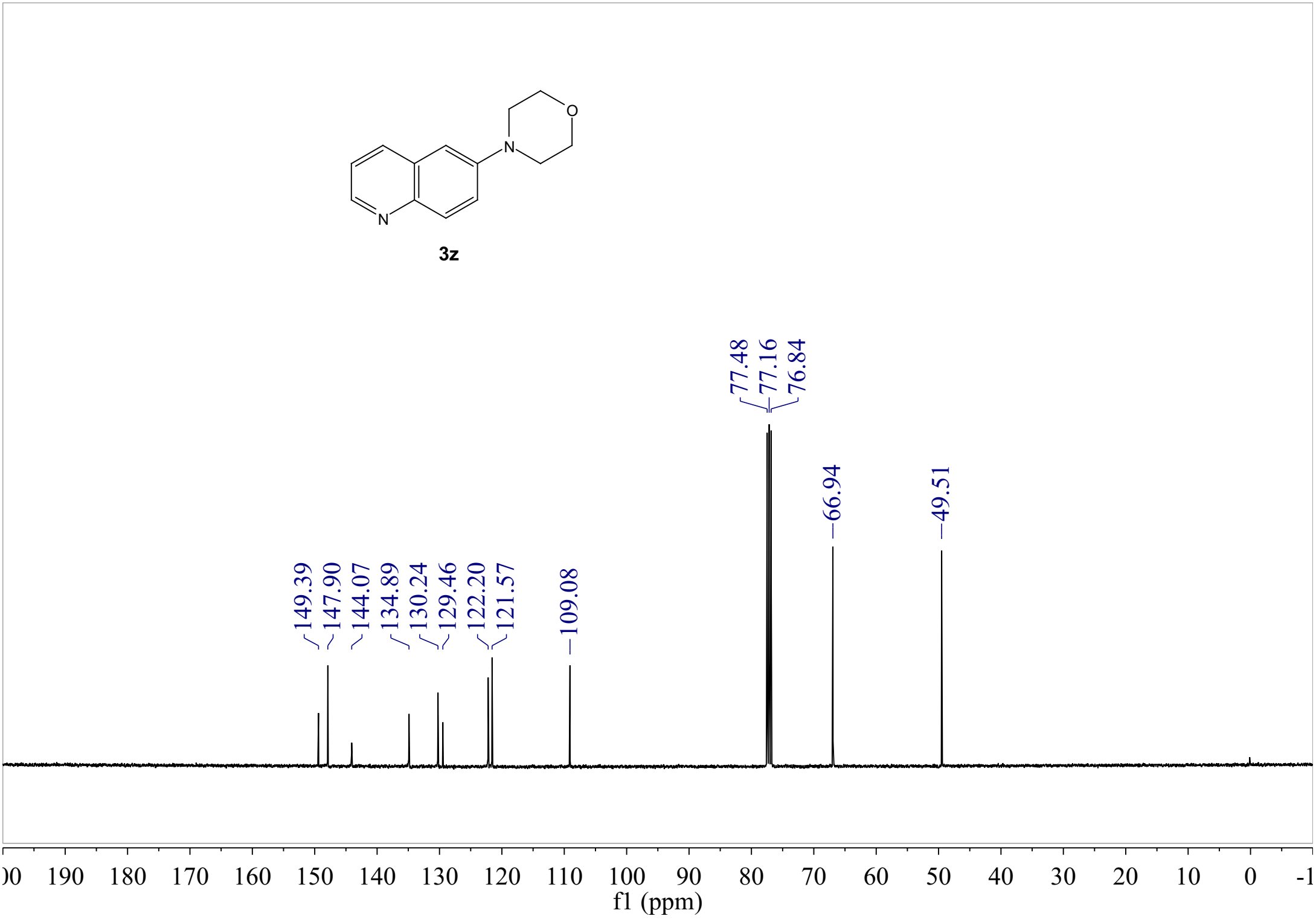


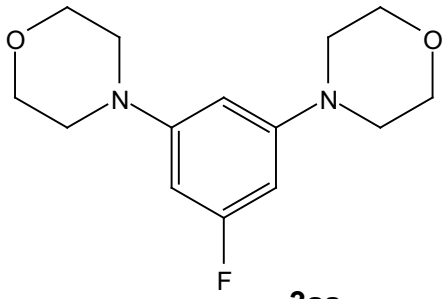
3z

8.73
8.73
8.72
8.72
8.01
8.00
7.99
7.98
7.98
7.49
7.48
7.46
7.46
7.33
7.32
7.31
7.30
7.26
7.02
7.01
3.92
3.91
3.90
3.29
3.28
3.27

1.0
2.0
1.0
1.0
1.0
4.0
4.0

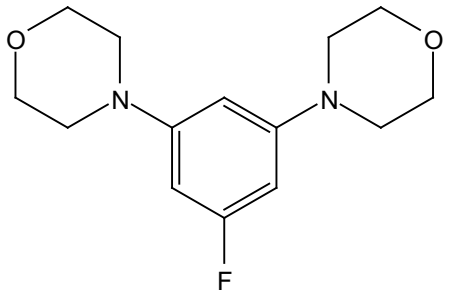
f1 (ppm)


O
N
N
3z
149.39
147.90
144.07
134.89
130.24
129.46
122.20
121.57
109.08
77.48
77.16
76.84
66.94
49.51
)0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


O
N
N
O
F

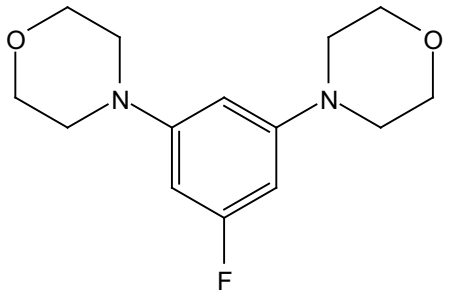
3aa

7.26
6.18
6.17
6.17
6.16
6.16
6.13
6.13
3.85
3.84
3.83
3.83
3.82
3.14
3.14
3.13
3.12
2.0
1.0
8.2
8.2
f1 (ppm)


O
N
N
O
F

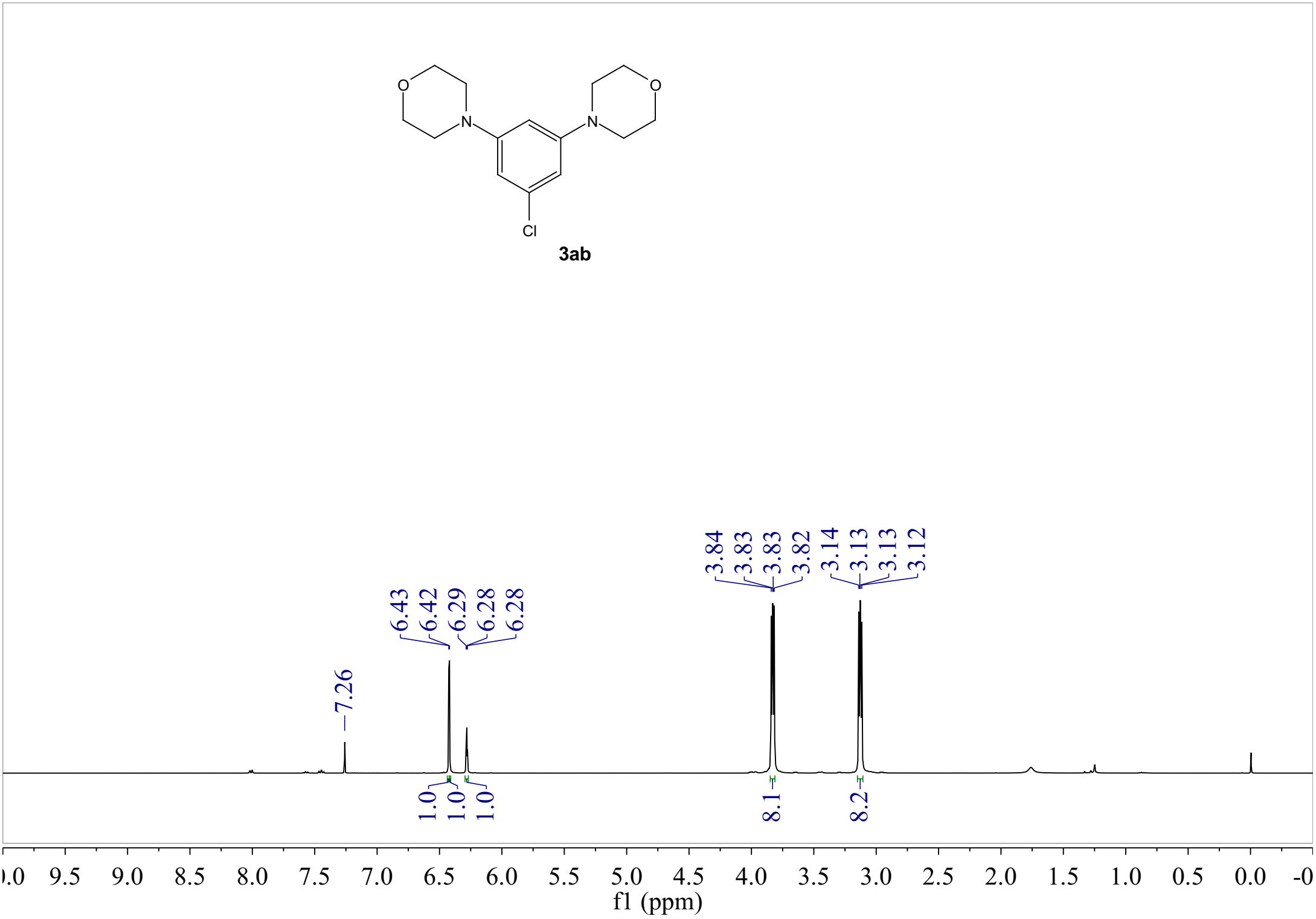
3aa

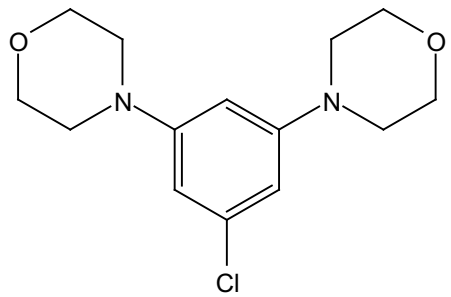
166.02
163.63
153.58
153.46
98.41
98.39
95.00
94.74
77.48
77.16
76.84
66.90
49.30
f1 (ppm)


O
N
N
O
F

3aa

-109.71
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


O
N
N
O
Cl
3ab
7.26
6.43
6.42
6.29
6.28
6.28
3.84
3.83
3.83
3.82
3.14
3.13
3.13
3.12
1.0
1.0
1.0
8.1
8.2
f1 (ppm)

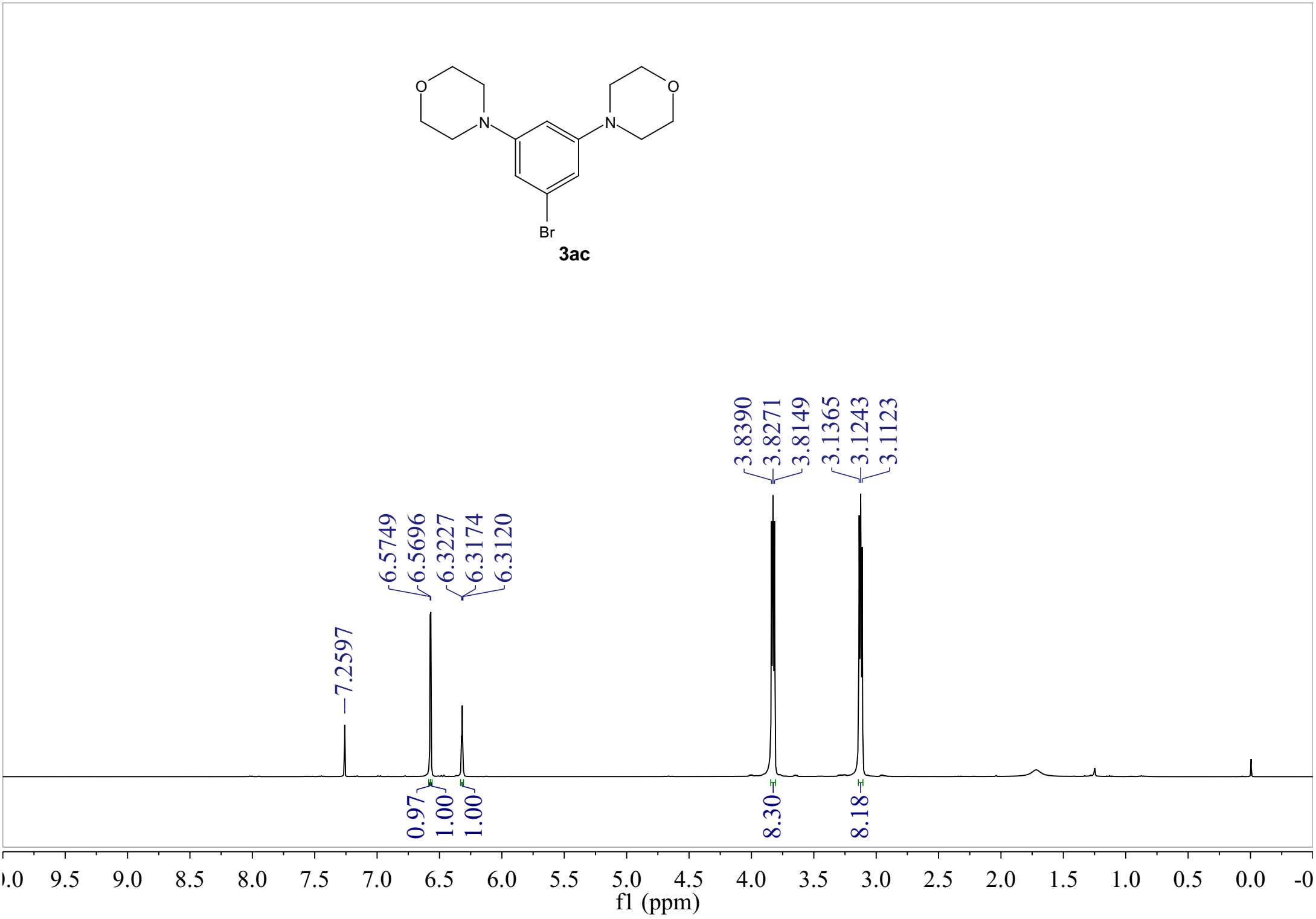


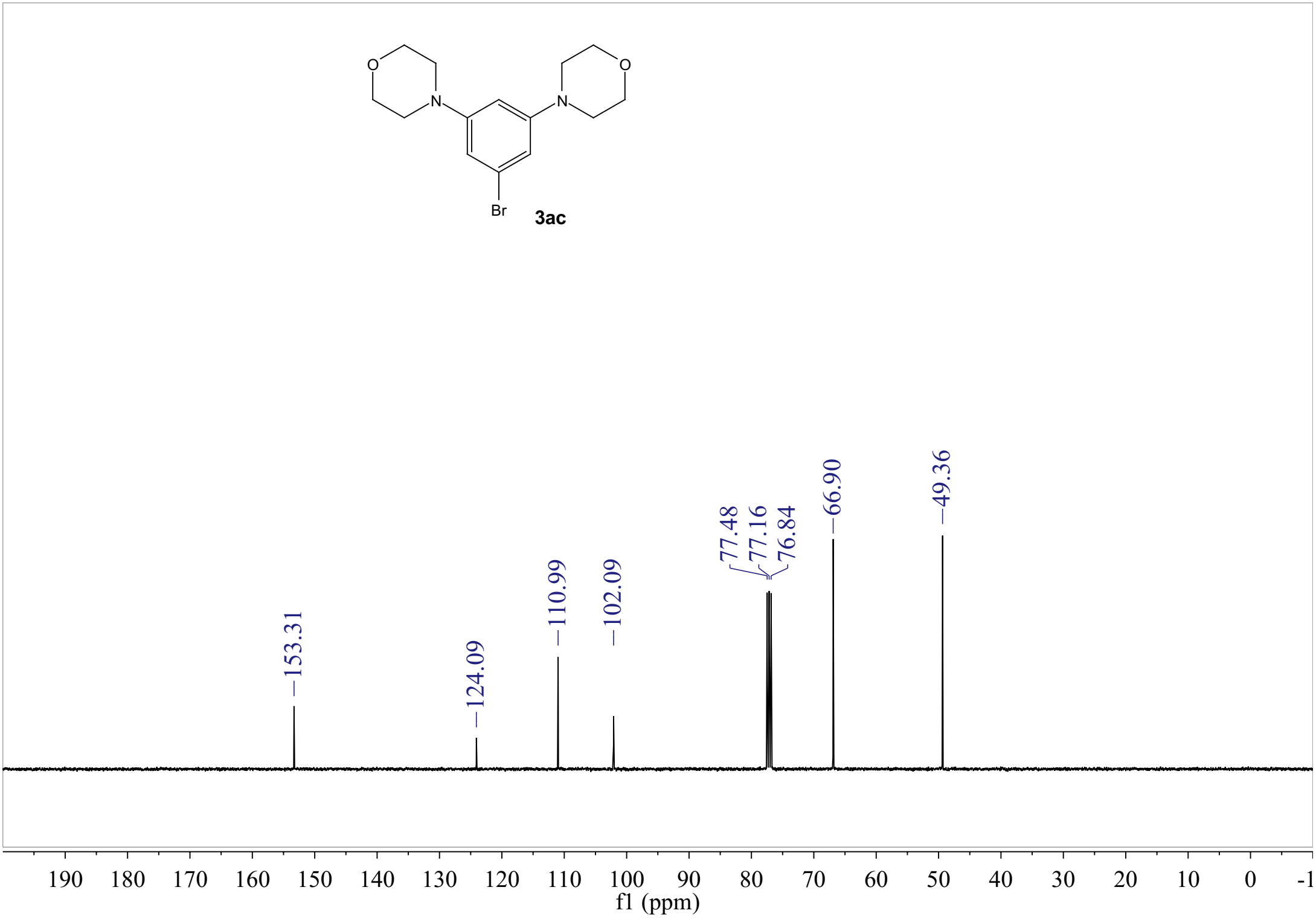
3ab

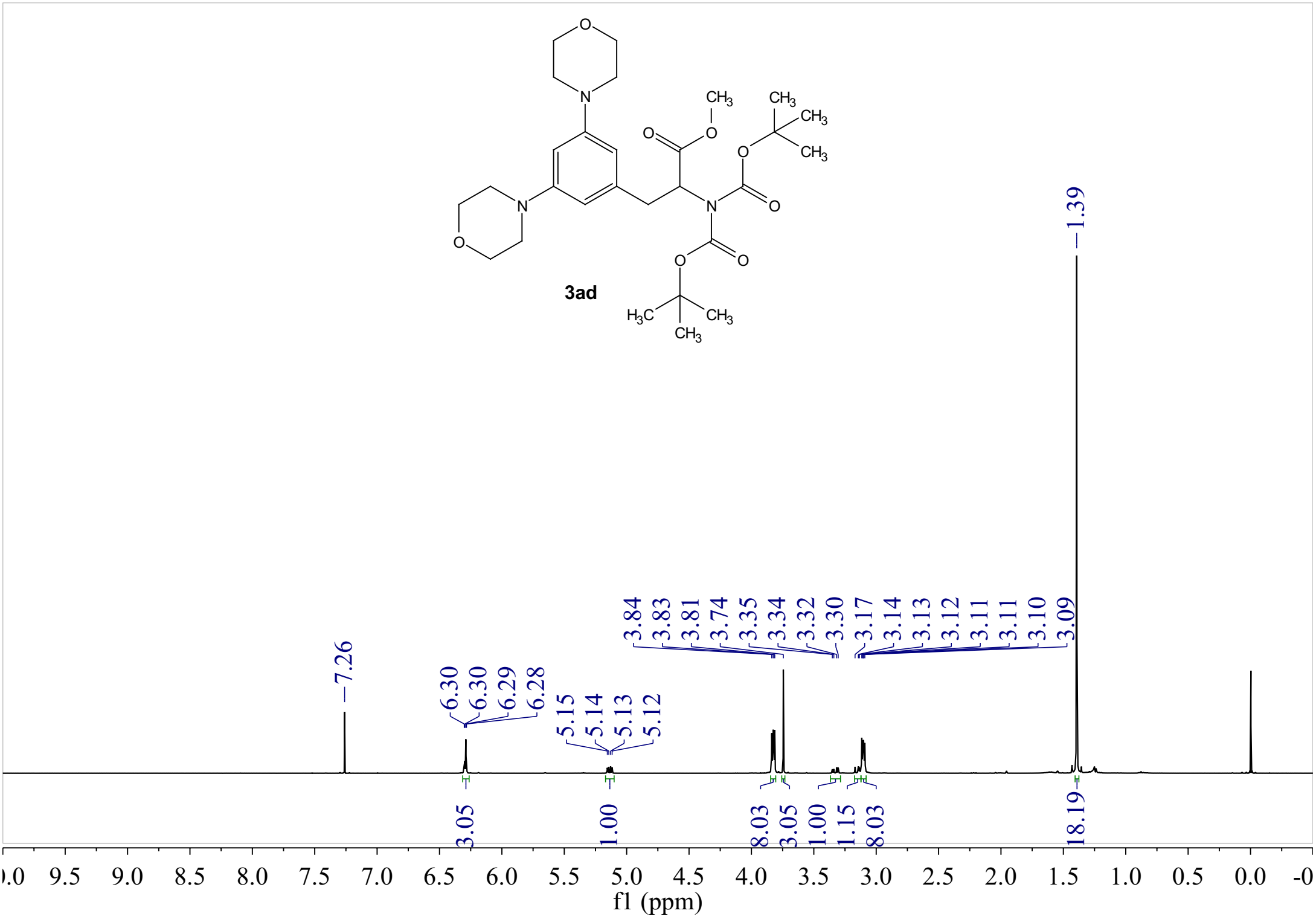
153.13
135.78
108.01
101.51
77.48
77.16
76.84
66.88
49.33

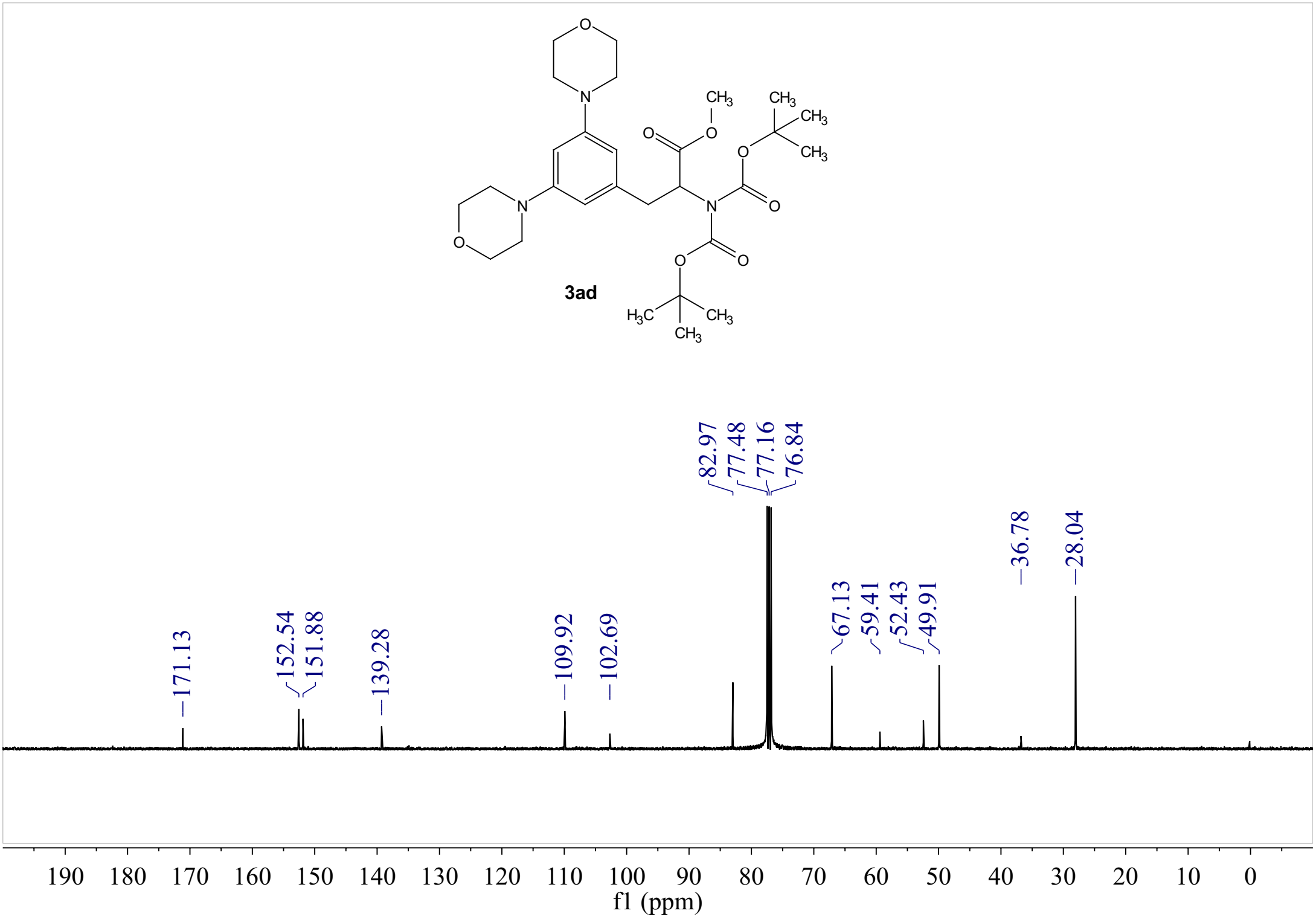
)0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

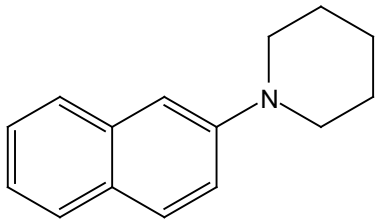
f1 (ppm)


O
N
N
O
Br
3ac
7.2597
6.5749
6.5696
6.3227
6.3174
6.3120
3.8390
3.8271
3.8149
3.1365
3.1243
3.1123
0.97
1.00
1.00
8.30
8.18
f1 (ppm)


O
N
N
O
Br
3ac
153.31
124.09
110.99
102.09
77.48
77.16
76.84
66.90
49.36
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

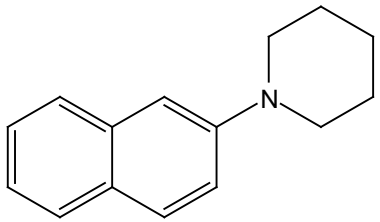

3ad
7.26
6.30
6.30
6.29
6.28
5.15
5.14
5.13
5.12
3.84
3.83
3.81
3.74
3.35
3.34
3.32
3.30
3.17
3.14
3.13
3.12
3.11
3.11
3.10
3.09
1.39
3.05
1.00
8.03
3.05
1.00
1.15
8.03
18.19
f1 (ppm)


3ad
171.13
152.54
151.88
139.28
109.92
102.69
82.97
77.48
77.16
76.84
67.13
59.41
52.43
49.91
36.78
28.04
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


N

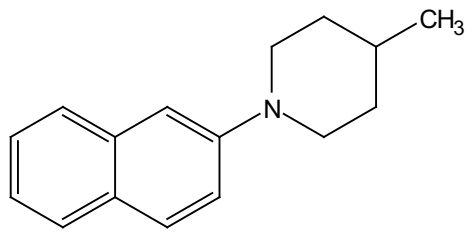
3B

7.70
7.68
7.66
7.66
7.38
7.37
7.36
7.35
7.29
7.28
7.28
7.28
7.27
7.26
7.26
7.26
7.24
7.12
7.11
3.26
3.24
3.23
1.77
1.76
1.74
1.73
1.62
1.62
1.61
0.00
3.0
1.0
2.1
1.0
4.0
4.2
2.1
f1 (ppm)


N

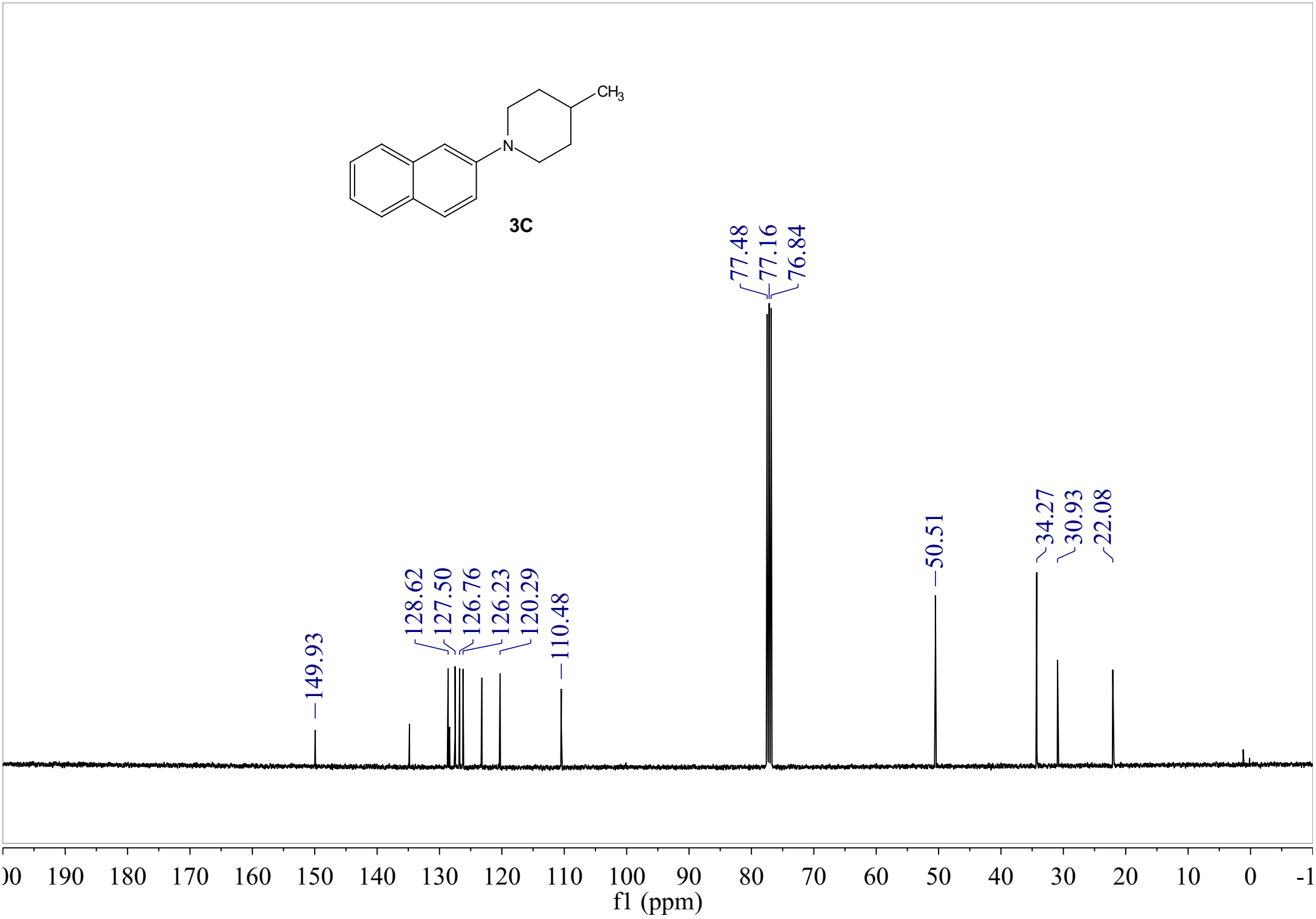
3B

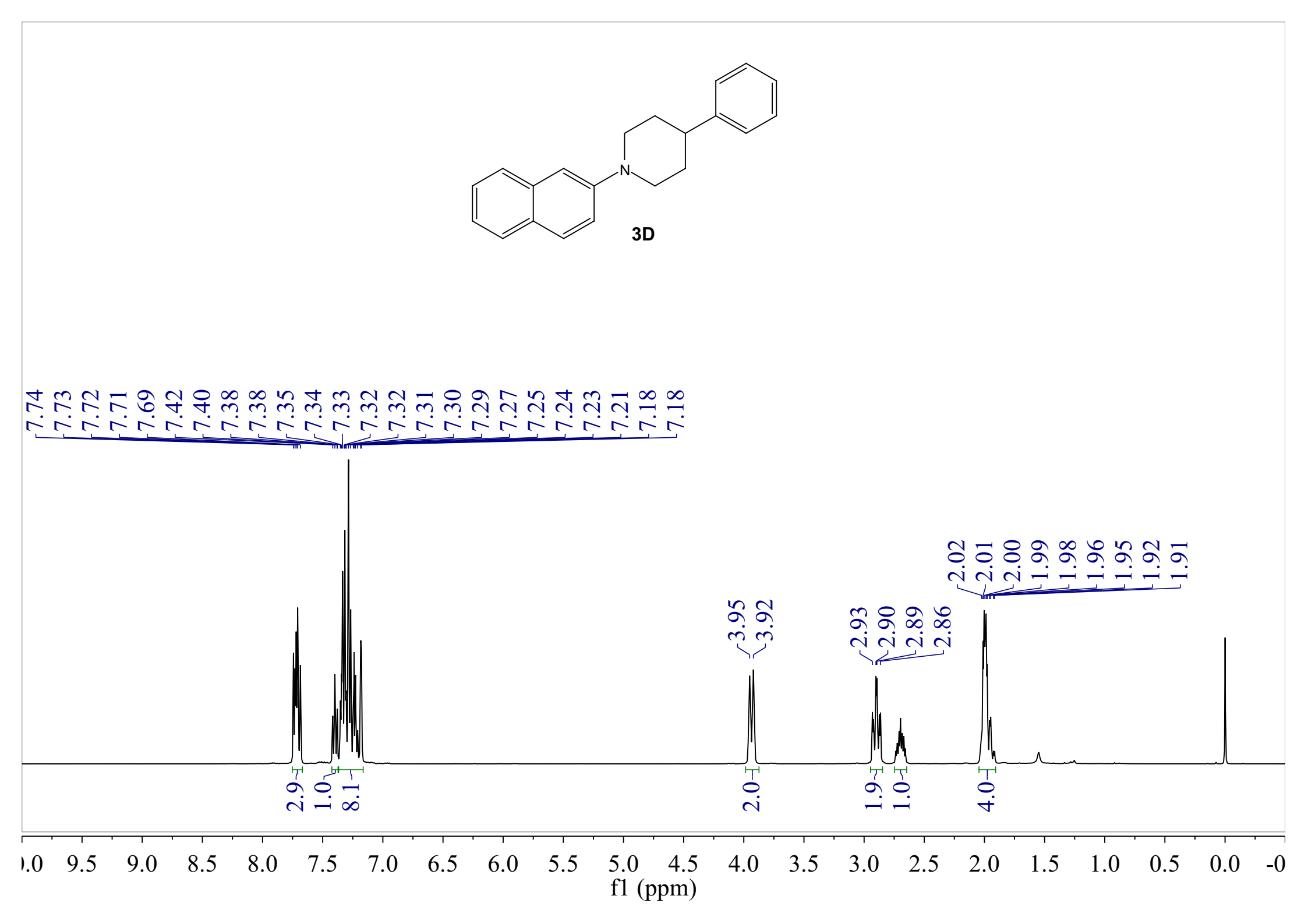
150.23
128.61
127.50
126.78
126.21
120.34
110.49
77.48
77.16
76.84
51.15
26.02
24.50
f1 (ppm)

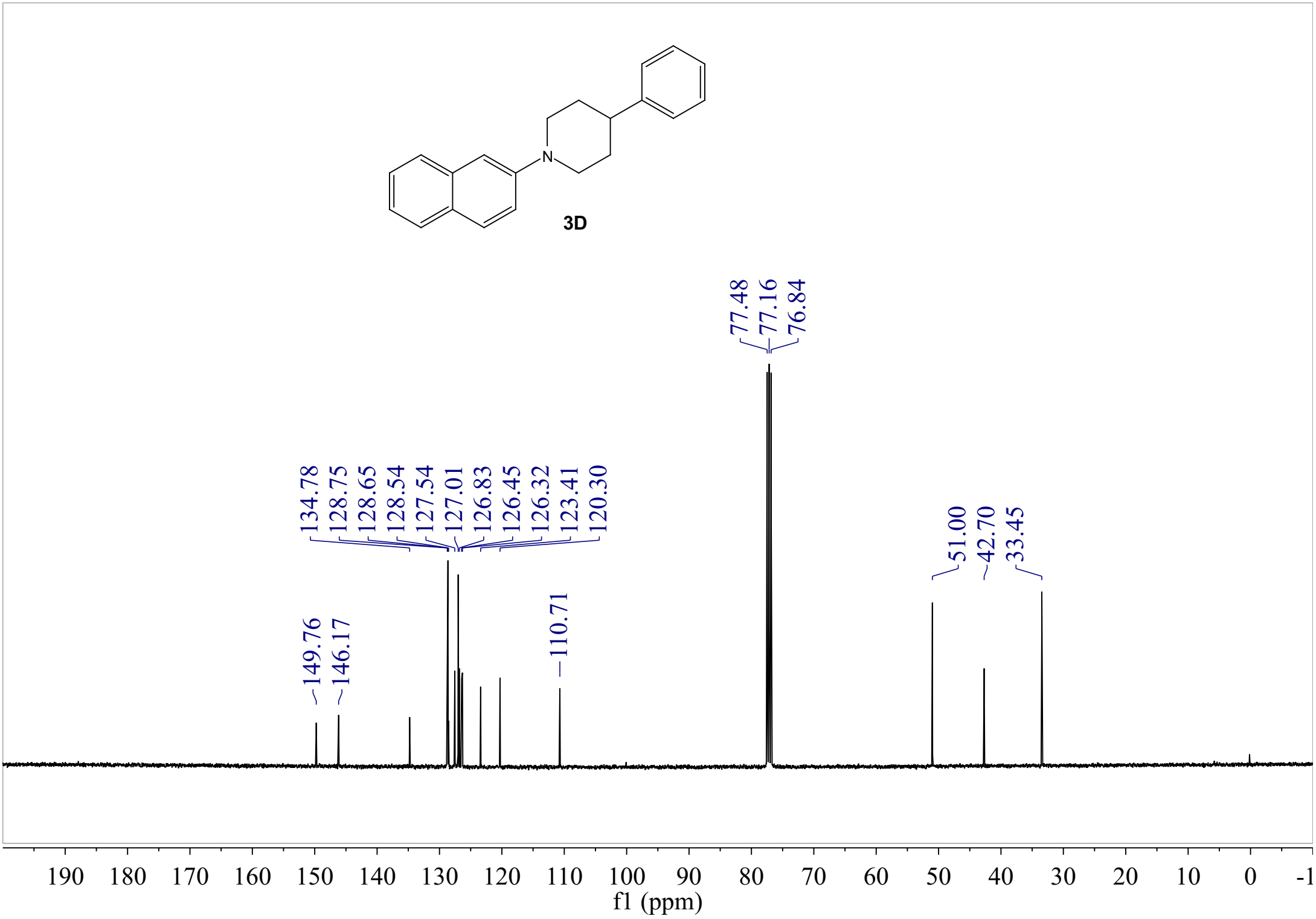

CH3
N

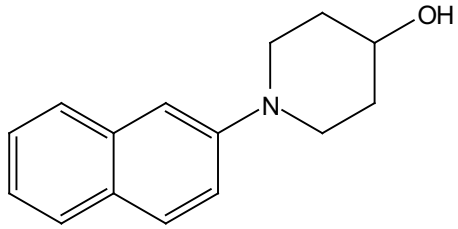
3C

1.73
1.69
1.54
1.53
1.52
1.51
1.51
1.50
1.49
1.48
1.48
1.47
1.46
1.45
1.44
1.44
1.39
1.38
1.36
1.35
1.33
1.32
1.30
1.29
0.93
0.92
7.64
7.62
7.61
7.59
7.33
7.32
7.31
7.29
7.29
7.23
7.22
7.21
7.20
7.19
7.18
7.17
7.17
7.06
3.72
3.68
2.73
2.72
2.70
2.69
2.67
2.66
3.0
1.0
2.4
1.0
2.1
2.0
2.1
1.8
2.1
3.0
f1 (ppm)

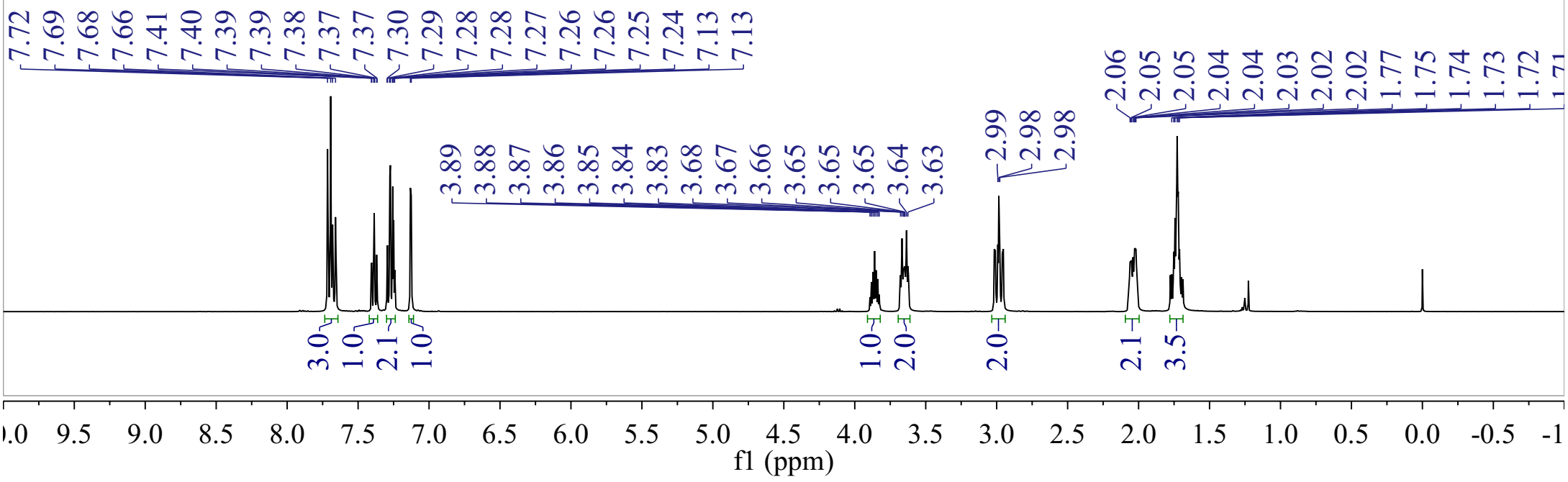

CH3
N
3C
77.48
77.16
76.84
149.93
128.62
127.50
126.76
126.23
120.29
110.48
50.51
34.27
30.93
22.08
f1 (ppm)

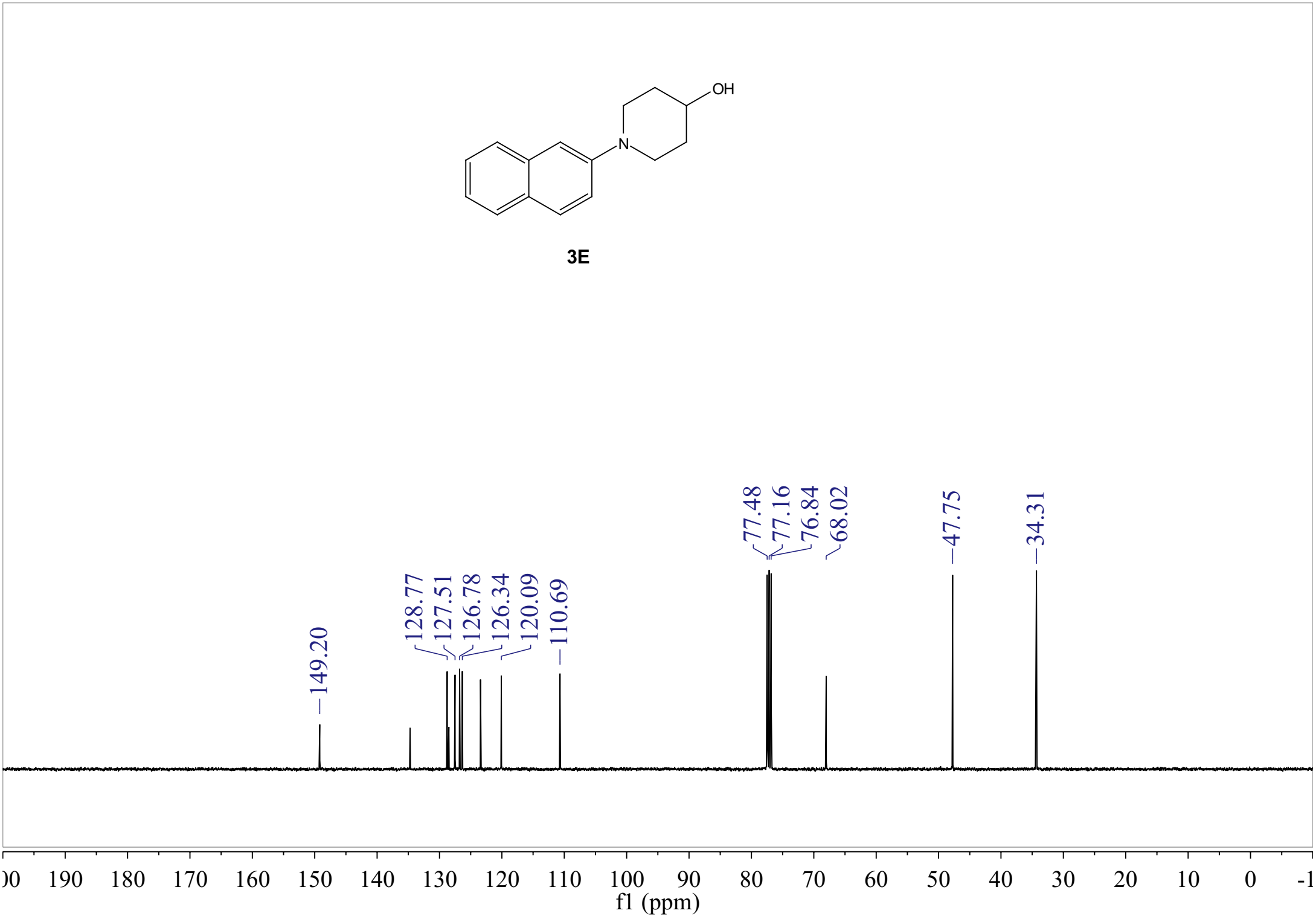

N
3D
7.74
7.73
7.72
7.71
7.69
7.42
7.40
7.38
7.38
7.35
7.34
7.33
7.32
7.32
7.31
7.30
7.29
7.27
7.25
7.24
7.23
7.21
7.18
7.18
3.95
3.92
2.93
2.90
2.89
2.86
2.02
2.01
2.00
1.99
1.98
1.96
1.95
1.92
1.91
2.9
1.0
8.1
2.0
1.9
1.0
4.0
f1 (ppm)

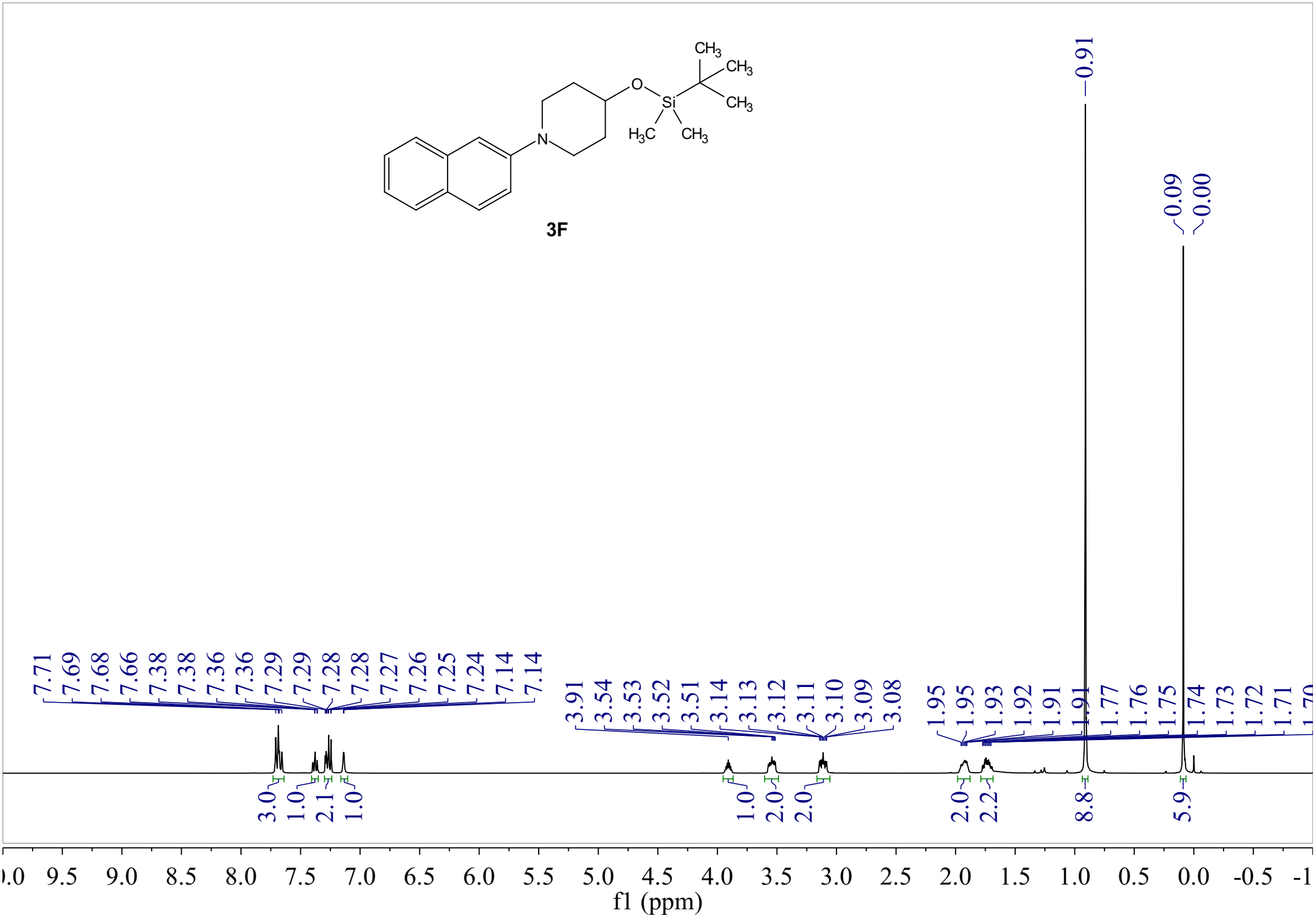

N
3D
77.48
77.16
76.84
134.78
128.75
128.65
128.54
127.54
127.01
126.83
126.45
126.32
123.41
120.30
149.76
146.17
110.71
51.00
42.70
33.45
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

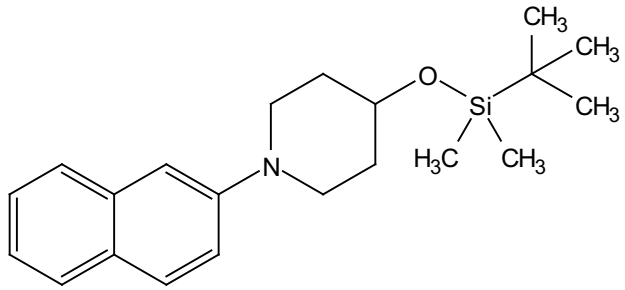

OH
N

3E


7.72
7.69
7.68
7.66
7.41
7.40
7.39
7.39
7.38
7.37
7.37
7.30
7.29
7.28
7.28
7.27
7.26
7.26
7.25
7.24
7.13
7.13
3.89
3.88
3.87
3.86
3.85
3.84
3.83
3.68
3.67
3.66
3.65
3.65
3.65
3.64
3.63
2.99
2.98
2.98
2.06
2.05
2.05
2.04
2.04
2.03
2.02
2.02
1.77
1.75
1.74
1.73
1.72
3.0
1.0
2.1
1.0
1.0
2.0
2.0
2.1
3.5
f1 (ppm)

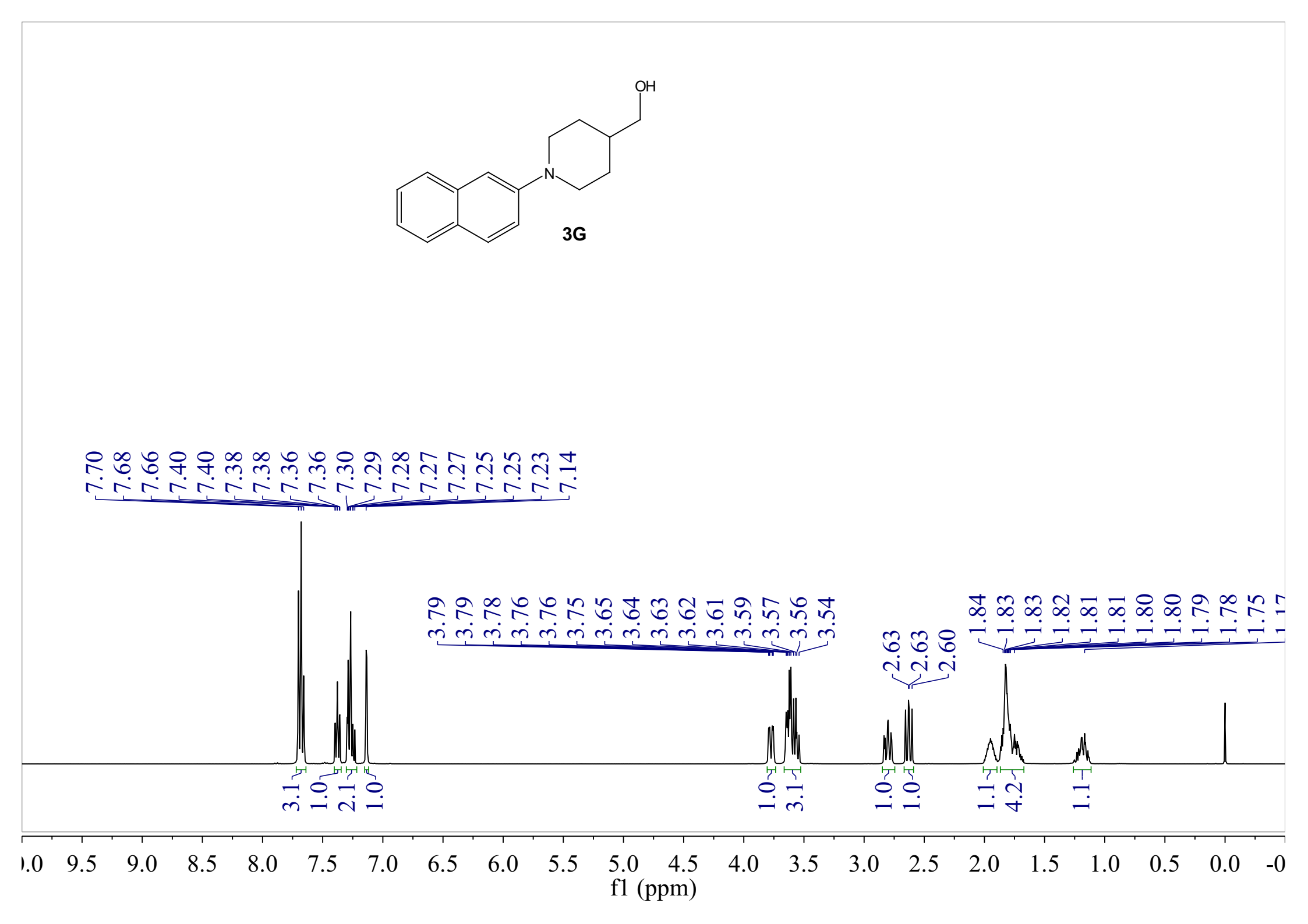

OH
N
3E
149.20
128.77
127.51
126.78
126.34
120.09
110.69
77.48
77.16
76.84
68.02
47.75
34.31
f1 (ppm)

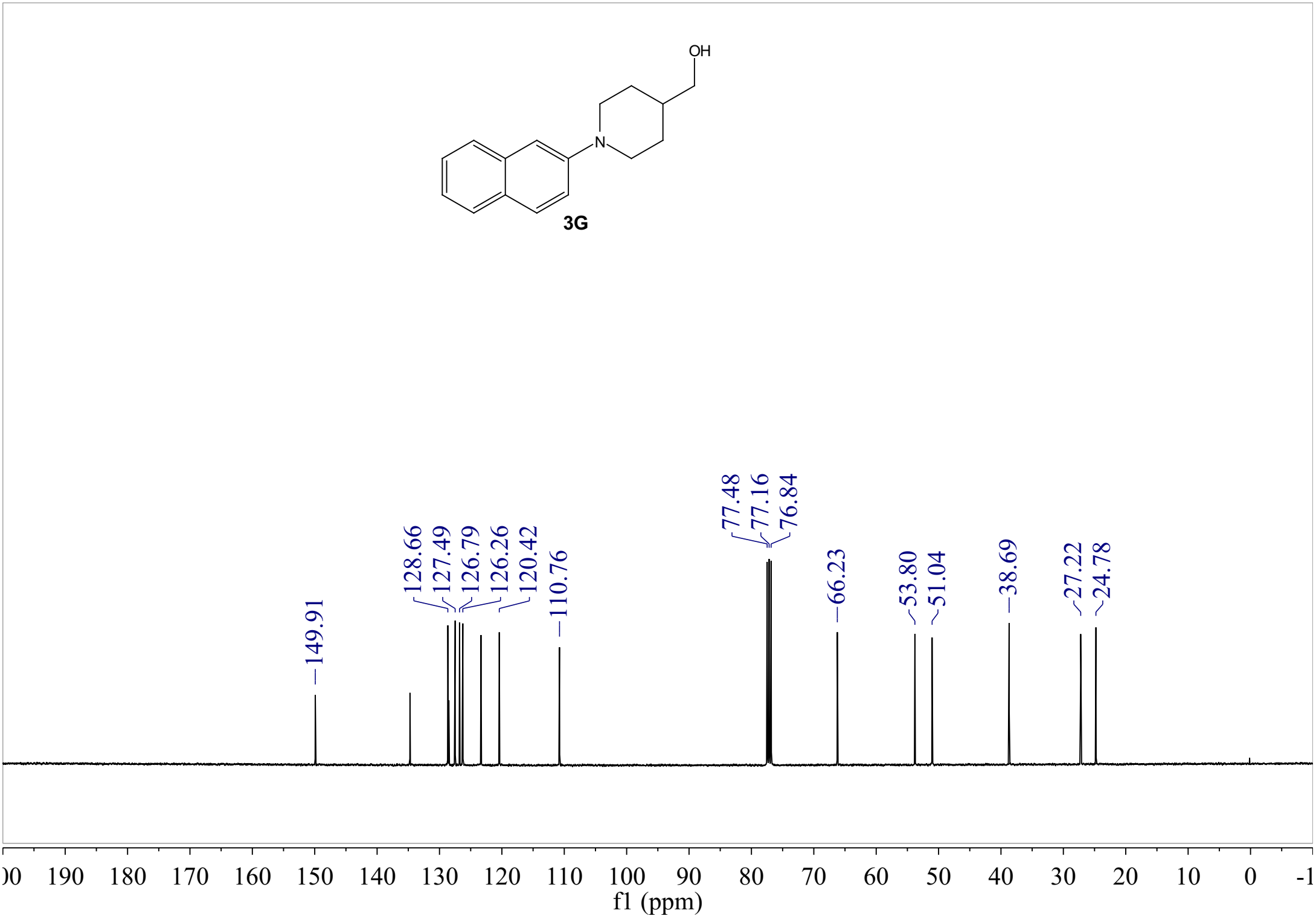

CH3
CH3
CH3
O
Si
H3C
CH3
N
3F
0.91
0.09
0.00
7.71
7.69
7.68
7.66
7.38
7.38
7.36
7.36
7.29
7.29
7.28
7.28
7.27
7.26
7.25
7.24
7.14
7.14
3.91
3.54
3.53
3.52
3.51
3.14
3.13
3.12
3.11
3.10
3.09
3.08
1.95
1.95
1.93
1.92
1.91
1.91
1.77
1.76
1.75
1.74
1.73
1.72
1.71
3.0
1.0
2.1
1.0
1.0
2.0
2.0
2.0
2.2
8.8
5.9
f1 (ppm)

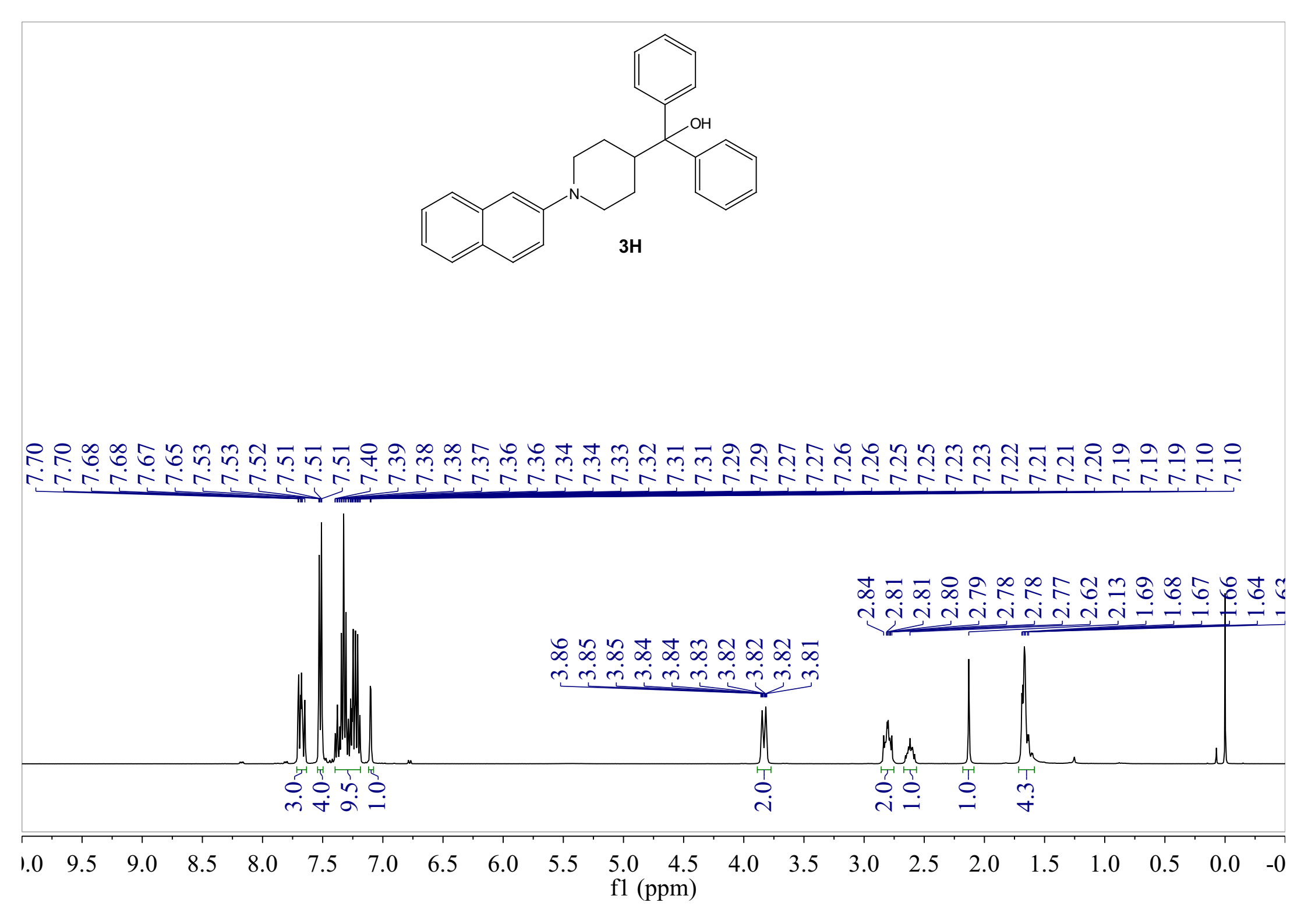

CH3
CH3
CH3
O
Si
H3C
CH3
N

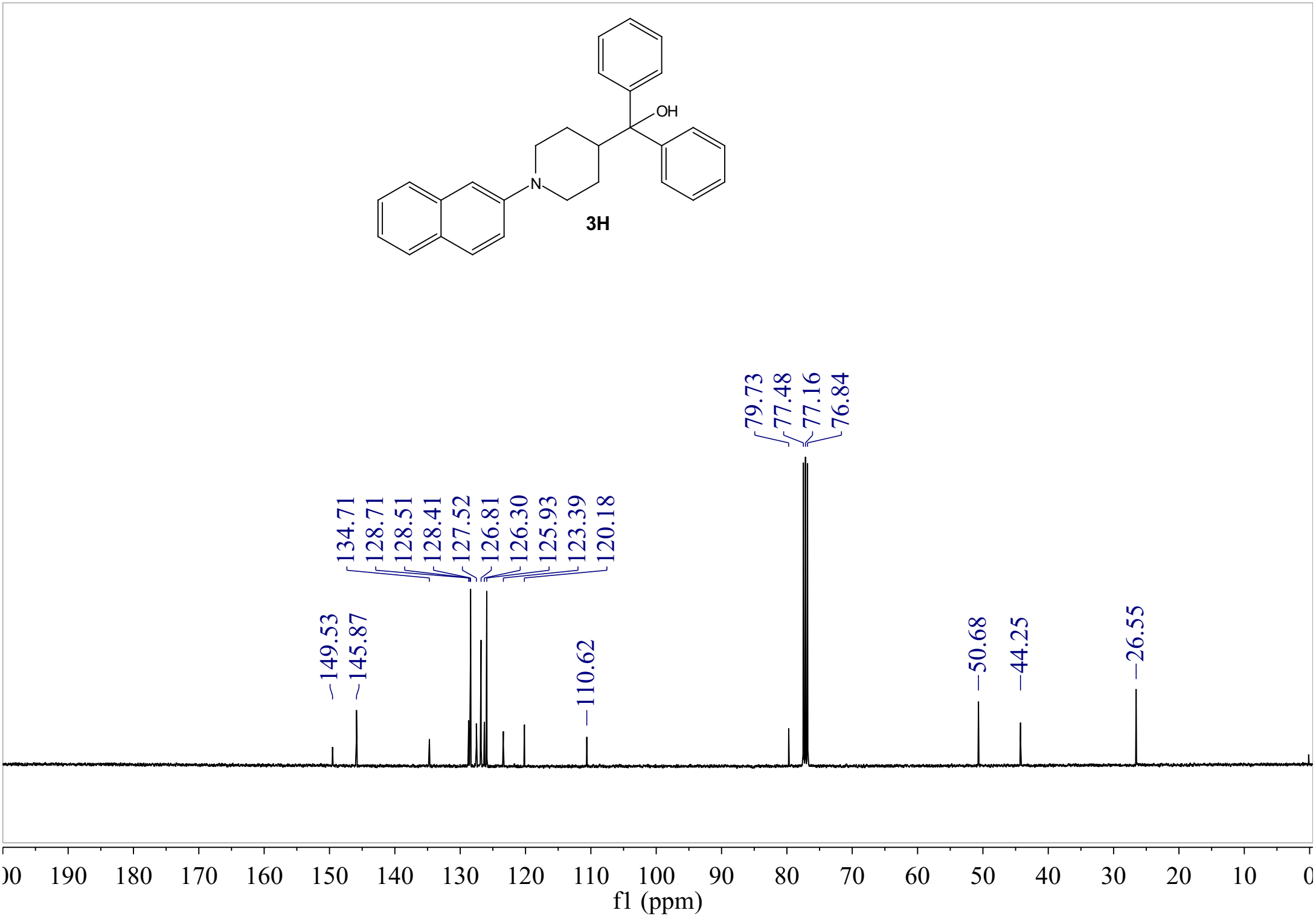
3F

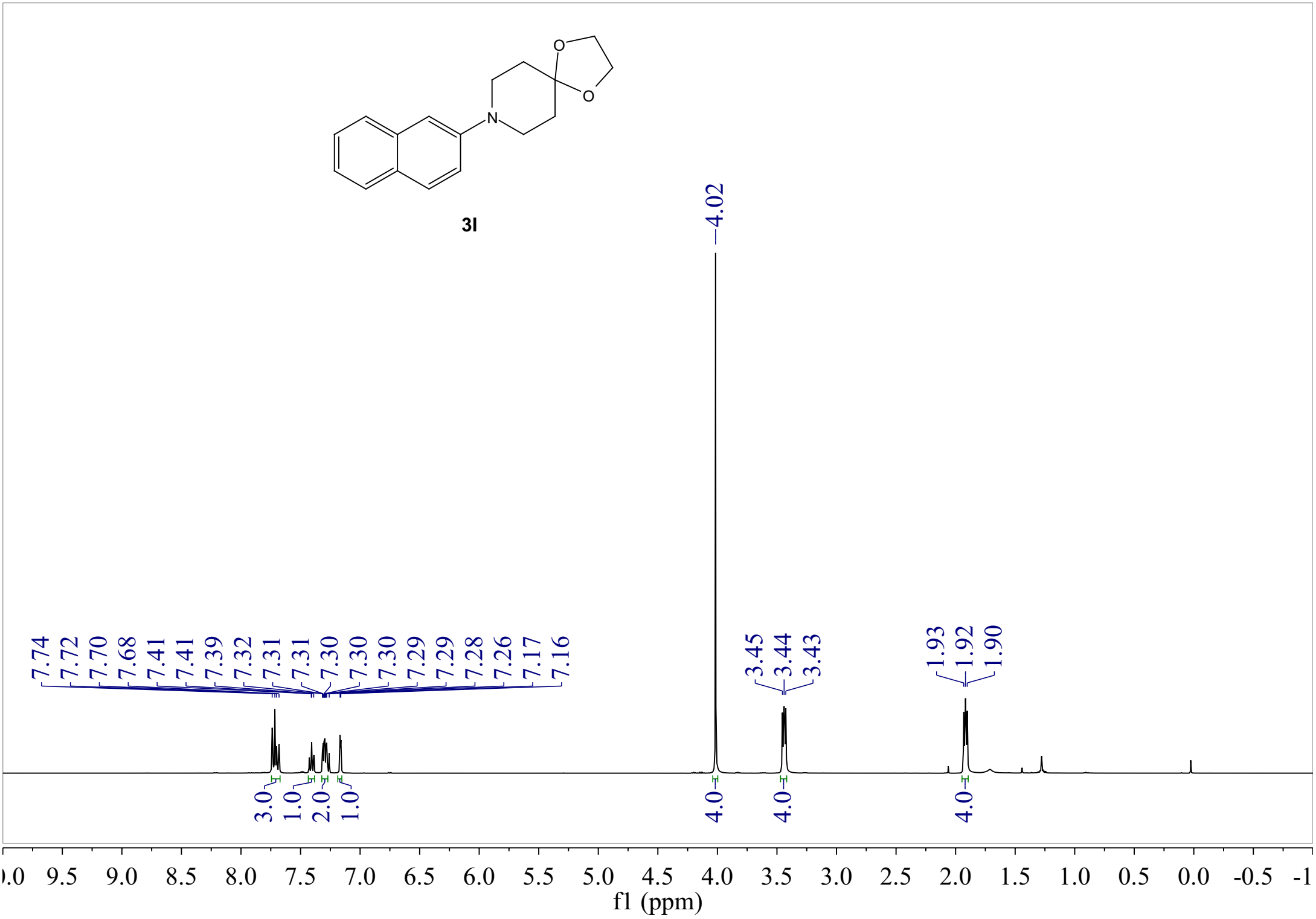
149.49
128.70
127.51
126.77
126.28
120.04
110.47
77.48
77.16
76.84
67.59
47.14
34.46
26.00
18.29
-4.49
f1 (ppm)

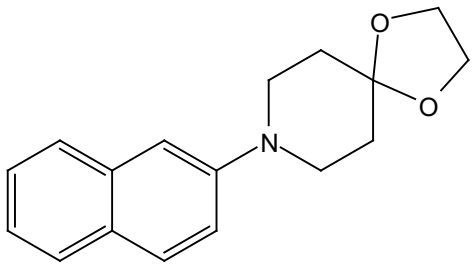

OH
N
3G
7.70
7.68
7.66
7.40
7.40
7.38
7.38
7.36
7.36
7.30
7.29
7.28
7.27
7.27
7.25
7.25
7.23
7.14
3.79
3.79
3.78
3.76
3.76
3.75
3.65
3.64
3.63
3.62
3.61
3.59
3.57
3.56
3.54
2.63
2.63
2.60
1.84
1.83
1.83
1.82
1.81
1.81
1.80
1.80
1.79
1.78
1.75
3.1
1.0
2.1
1.0
1.0
3.1
1.0
1.0
1.1
4.2
1.1
f1 (ppm)


OH
N
3G
149.91
128.66
127.49
126.79
126.26
120.42
110.76
77.48
77.16
76.84
66.23
53.80
51.04
38.69
27.22
24.78
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OH
N
3H
7.70 7.70 7.68 7.68 7.67 7.65 7.53 7.53 7.52 7.51 7.51 7.51 7.40 7.39 7.38 7.38 7.37 7.36 7.36 7.34 7.34 7.33 7.32 7.31 7.31 7.29 7.29 7.27 7.27 7.26 7.26 7.25 7.25 7.23 7.23 7.22 7.21 7.21 7.20 7.19 7.19 7.19 7.10 7.10
3.86 3.85 3.85 3.84 3.84 3.83 3.82 3.82 3.82 3.81
2.84 2.81 2.81 2.80 2.79 2.78 2.78 2.77 2.62 2.13 1.69 1.68 1.67 1.66 1.64
3.0 4.0 9.5 1.0 2.0 2.0 1.0 1.0 4.3
f1 (ppm)


OH
N
3H
149.53
145.87
134.71
128.71
128.51
128.41
127.52
126.81
126.30
125.93
123.39
120.18
110.62
79.73
77.48
77.16
76.84
50.68
44.25
26.55
f1 (ppm)


O
O
N
3l
4.02
7.74
7.72
7.70
7.68
7.41
7.41
7.39
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.29
7.28
7.26
7.17
7.16
3.45
3.44
3.43
1.93
1.92
1.90
3.0
1.0
2.0
1.0
4.0
4.0
4.0
f1 (ppm)

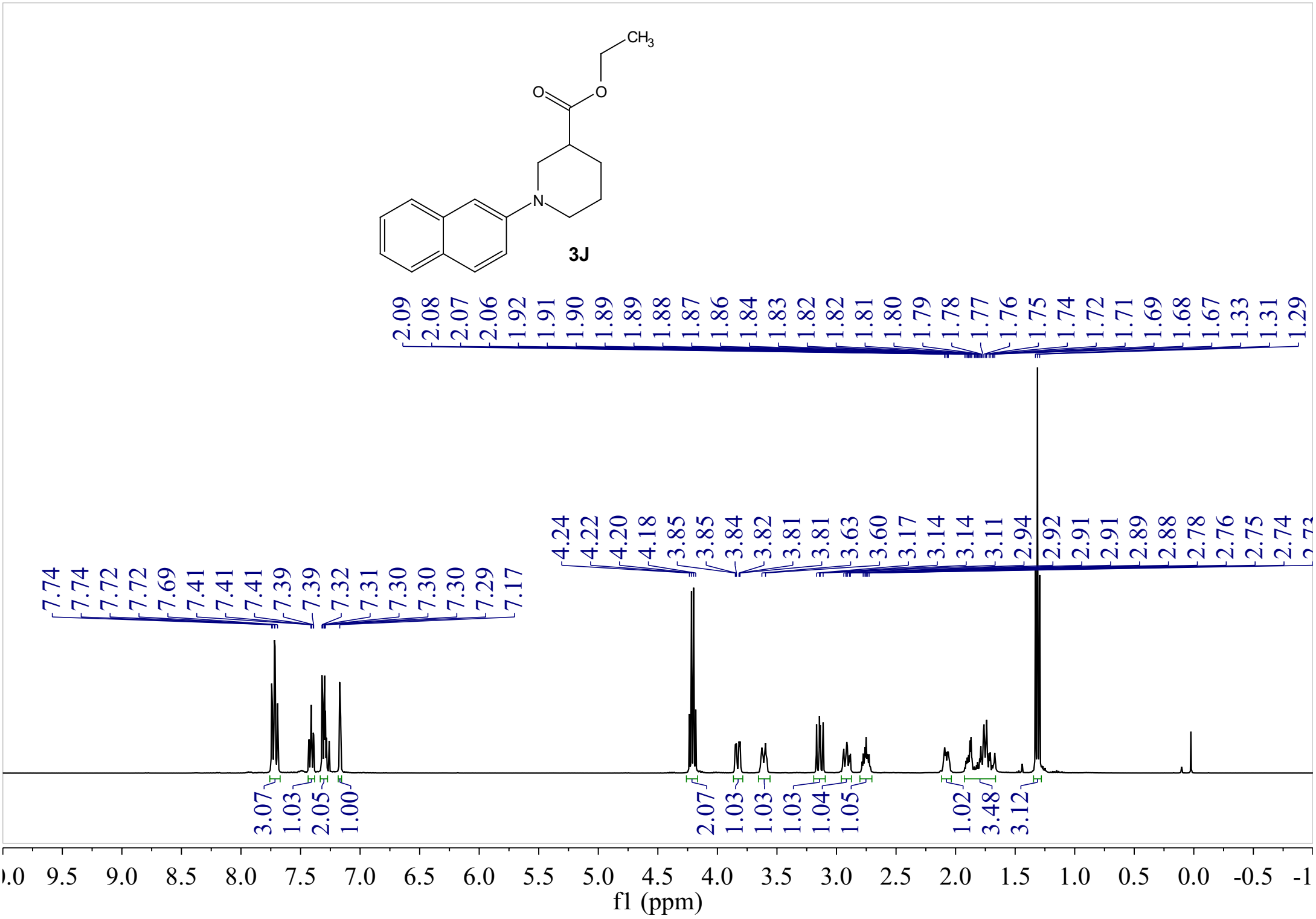


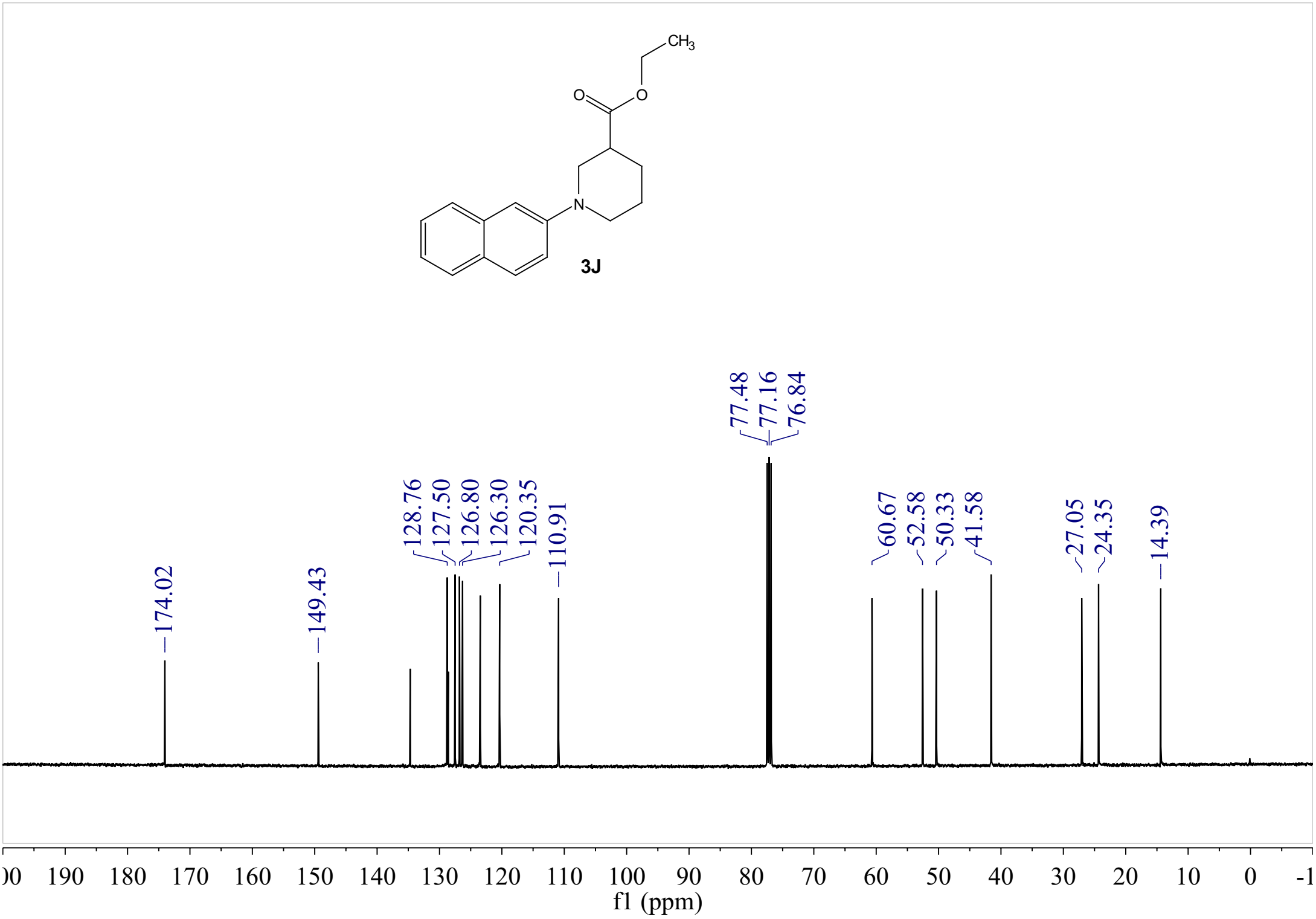
3l

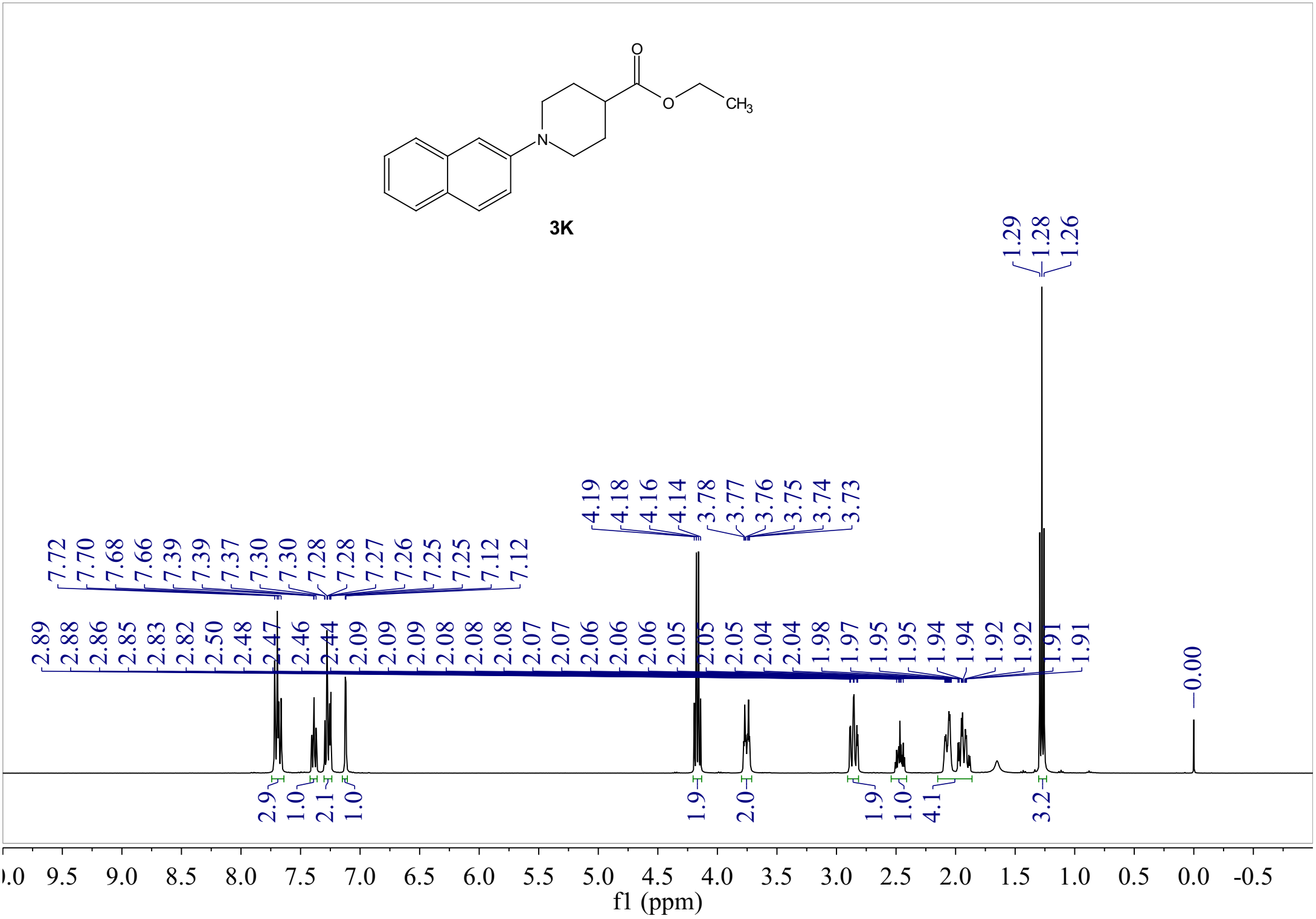
148.91
128.77
127.49
126.77
126.31
120.08
110.80
107.27
77.48
77.16
76.84
64.46
48.16
34.72

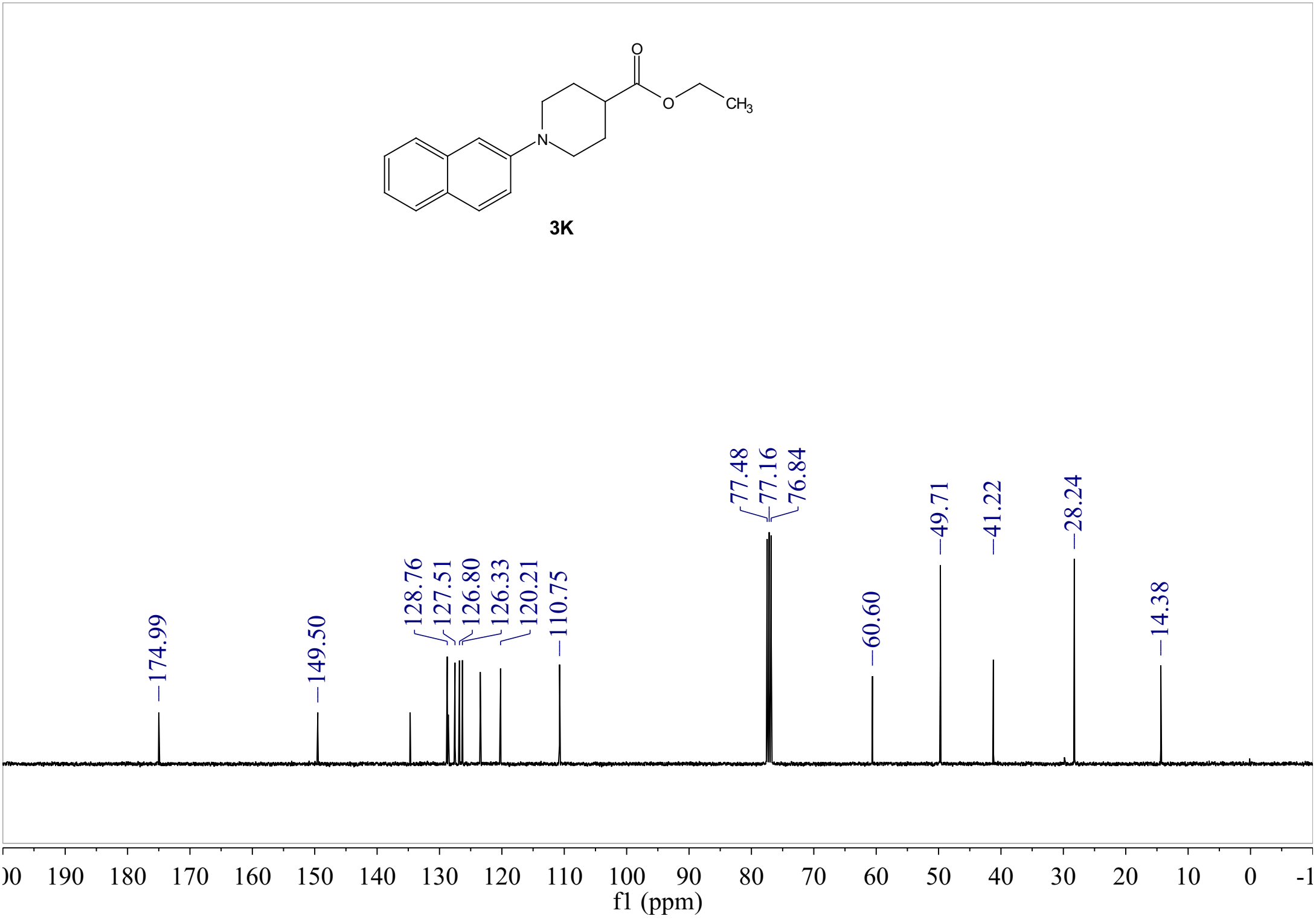
)0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

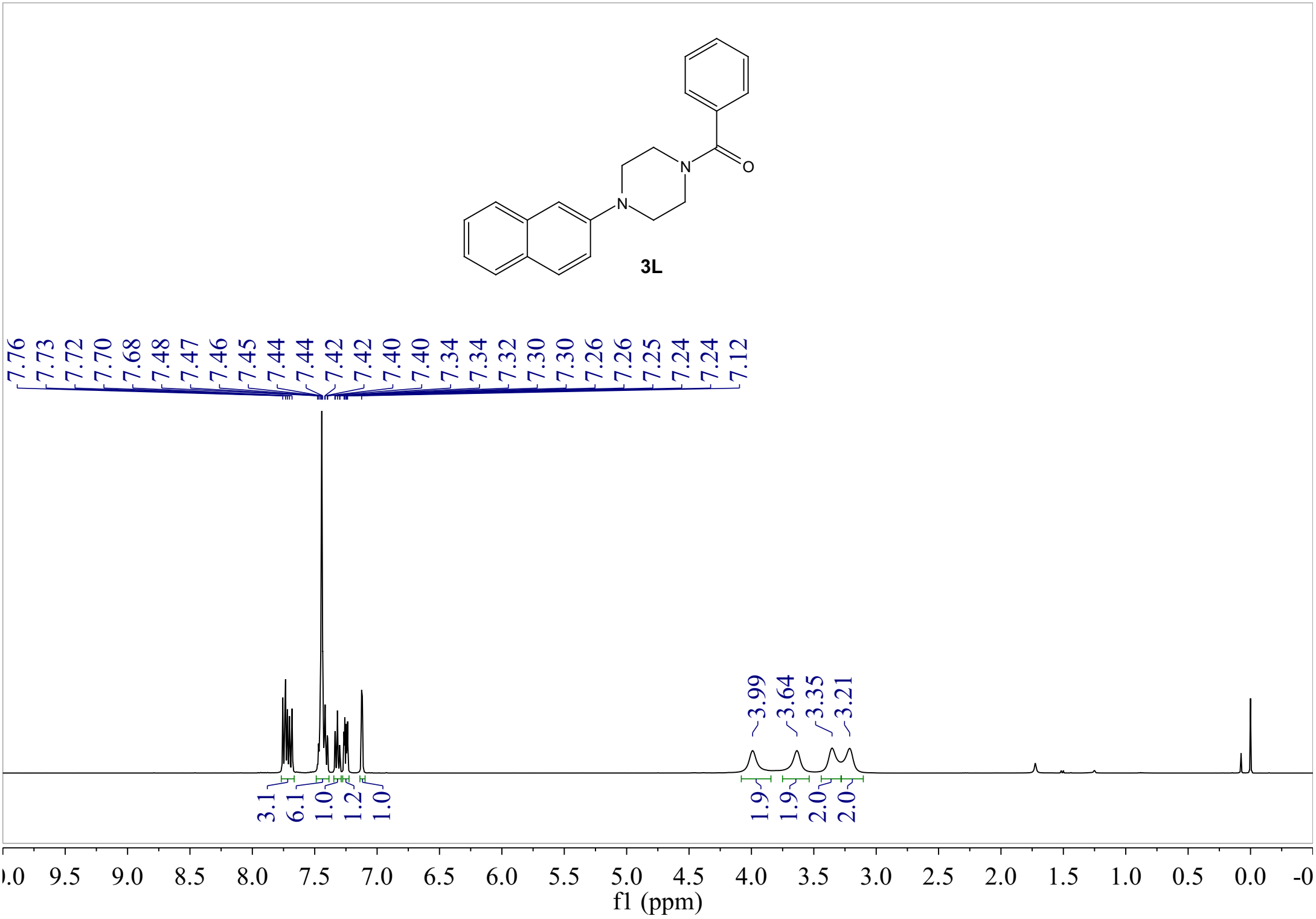
f1 (ppm)

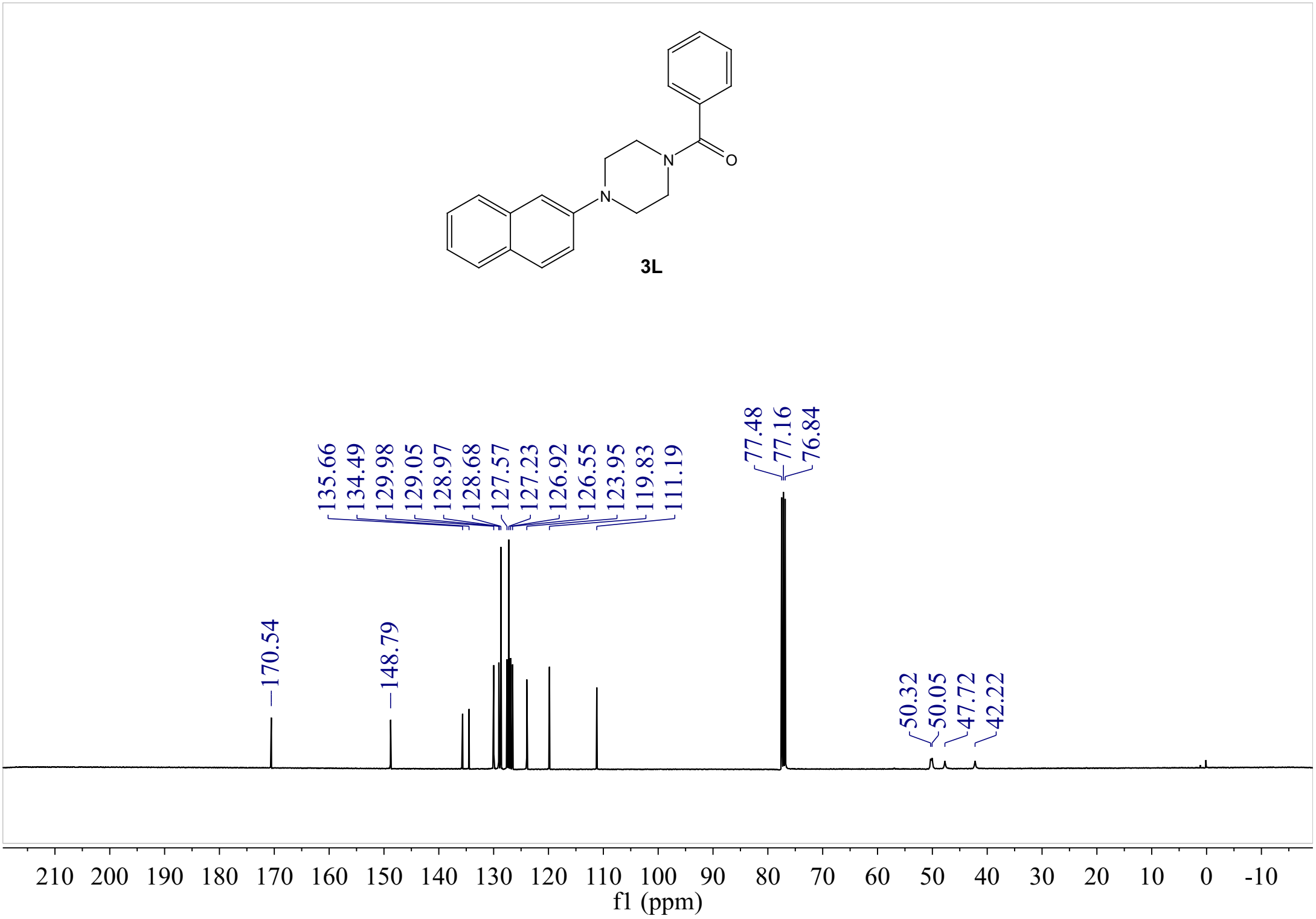

CH3
O
O
N
3J
2.09 2.08 2.07 2.06 1.92 1.91 1.90 1.89 1.89 1.88 1.87 1.86 1.84 1.83 1.82 1.82 1.81 1.80 1.79 1.78 1.77 1.76 1.75 1.74 1.72 1.71 1.69 1.68 1.67 1.33 1.31 1.29
4.24 4.22 4.20 4.18 3.85 3.85 3.84 3.82 3.81 3.81 3.63 3.60 3.17 3.14 3.14 3.11 2.94 2.92 2.91 2.91 2.89 2.88 2.78 2.76 2.75 2.74
7.74 7.74 7.72 7.72 7.69 7.41 7.41 7.41 7.39 7.39 7.32 7.31 7.30 7.30 7.30 7.29 7.17
3.07 1.03 2.05 1.00
2.07 1.03 1.03 1.03 1.04 1.05 1.02 3.48 3.12
f1 (ppm)

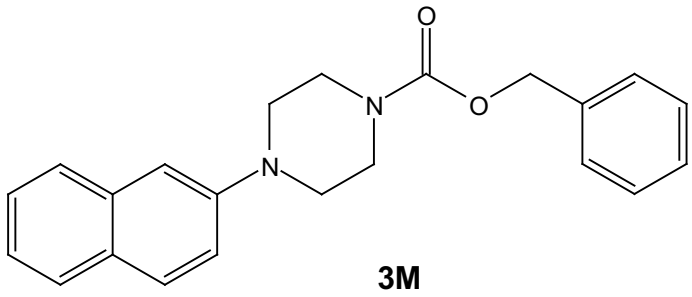

CH3
O
O
N
3J
174.02
149.43
128.76
127.50
126.80
126.30
120.35
110.91
77.48
77.16
76.84
60.67
52.58
50.33
41.58
27.05
24.35
14.39
f1 (ppm)


O
O
CH3
N
3K
7.72
7.70
7.68
7.66
7.39
7.39
7.37
7.30
7.30
7.28
7.28
7.27
7.26
7.25
7.25
7.12
7.12
4.19
4.18
4.16
4.14
3.78
3.77
3.76
3.75
3.74
3.73
2.89
2.88
2.86
2.85
2.83
2.82
2.50
2.48
2.47
2.46
2.44
2.09
2.09
2.09
2.08
2.08
2.08
2.07
2.07
2.06
2.06
2.06
2.05
2.05
2.05
2.04
2.04
1.98
1.97
1.95
1.95
1.94
1.94
1.92
1.92
1.91
1.91
1.29
1.28
1.26
0.00
2.9
1.0
2.1
1.0
1.9
2.0
1.9
1.0
4.1
3.2
f1 (ppm)


O
O
CH3
N
3K
174.99
149.50
128.76
127.51
126.80
126.33
120.21
110.75
77.48
77.16
76.84
60.60
49.71
41.22
28.24
14.38
f1 (ppm)


N
N
O
3L
7.76
7.73
7.72
7.70
7.68
7.48
7.47
7.46
7.45
7.44
7.44
7.42
7.42
7.40
7.40
7.34
7.34
7.32
7.30
7.30
7.26
7.26
7.25
7.24
7.24
7.12
3.99
3.64
3.35
3.21
3.1
6.1
1.0
1.2
1.0
1.9
1.9
2.0
2.0
f1 (ppm)

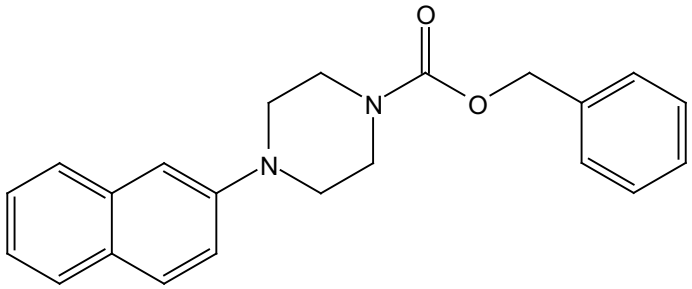

N
N
O
3L
135.66
134.49
129.98
129.05
128.97
128.68
127.57
127.23
126.92
126.55
123.95
119.83
111.19
77.48
77.16
76.84
170.54
148.79
50.32
50.05
47.72
42.22
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



3M

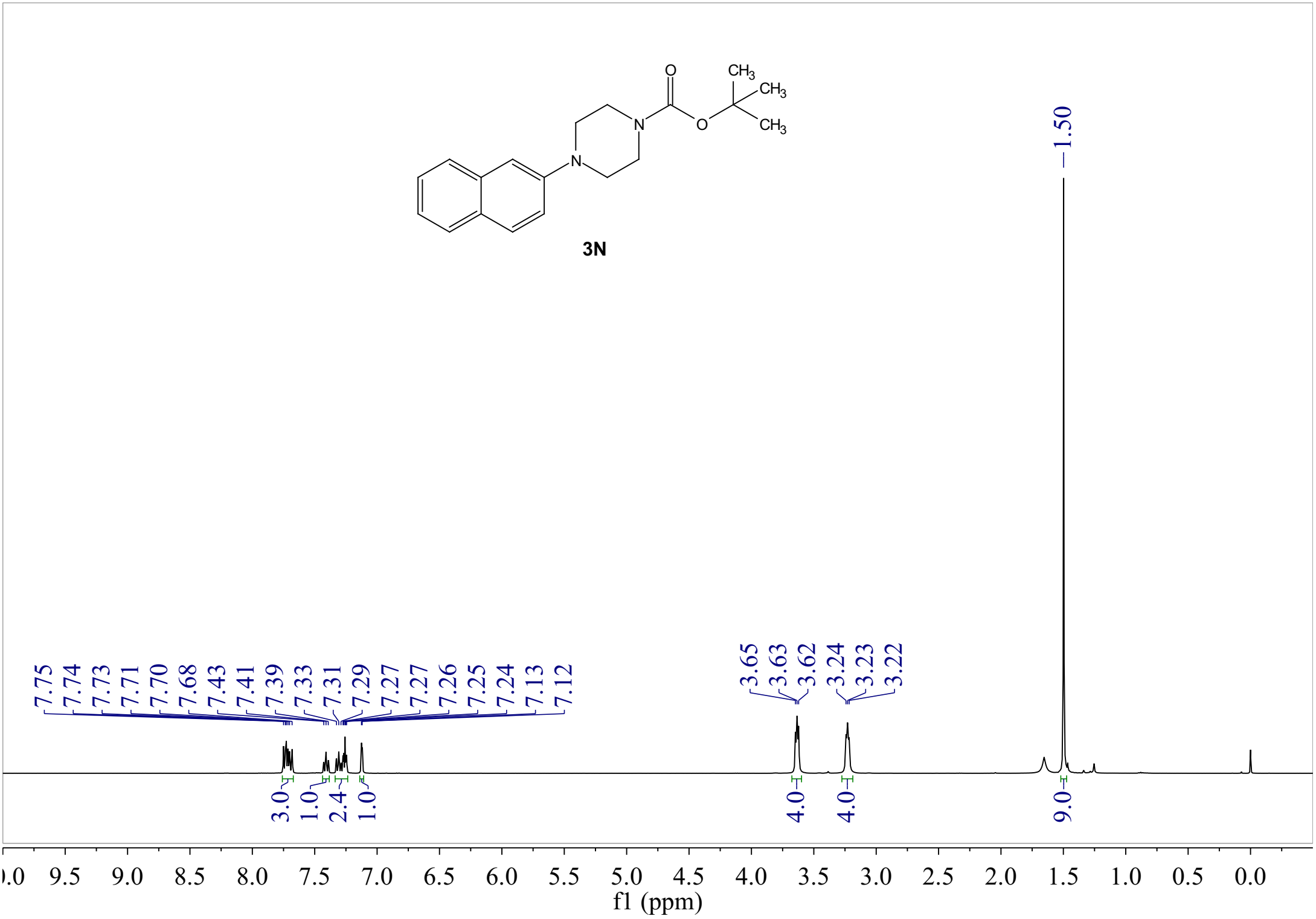
7.75
7.73
7.72
7.71
7.70
7.68
7.41
7.39
7.38
7.33
7.31
7.26
7.25
7.24
7.23
7.11
5.18
3.73
3.71
3.70
3.24
3.0
7.1
1.3
1.0
2.0
4.0
4.0

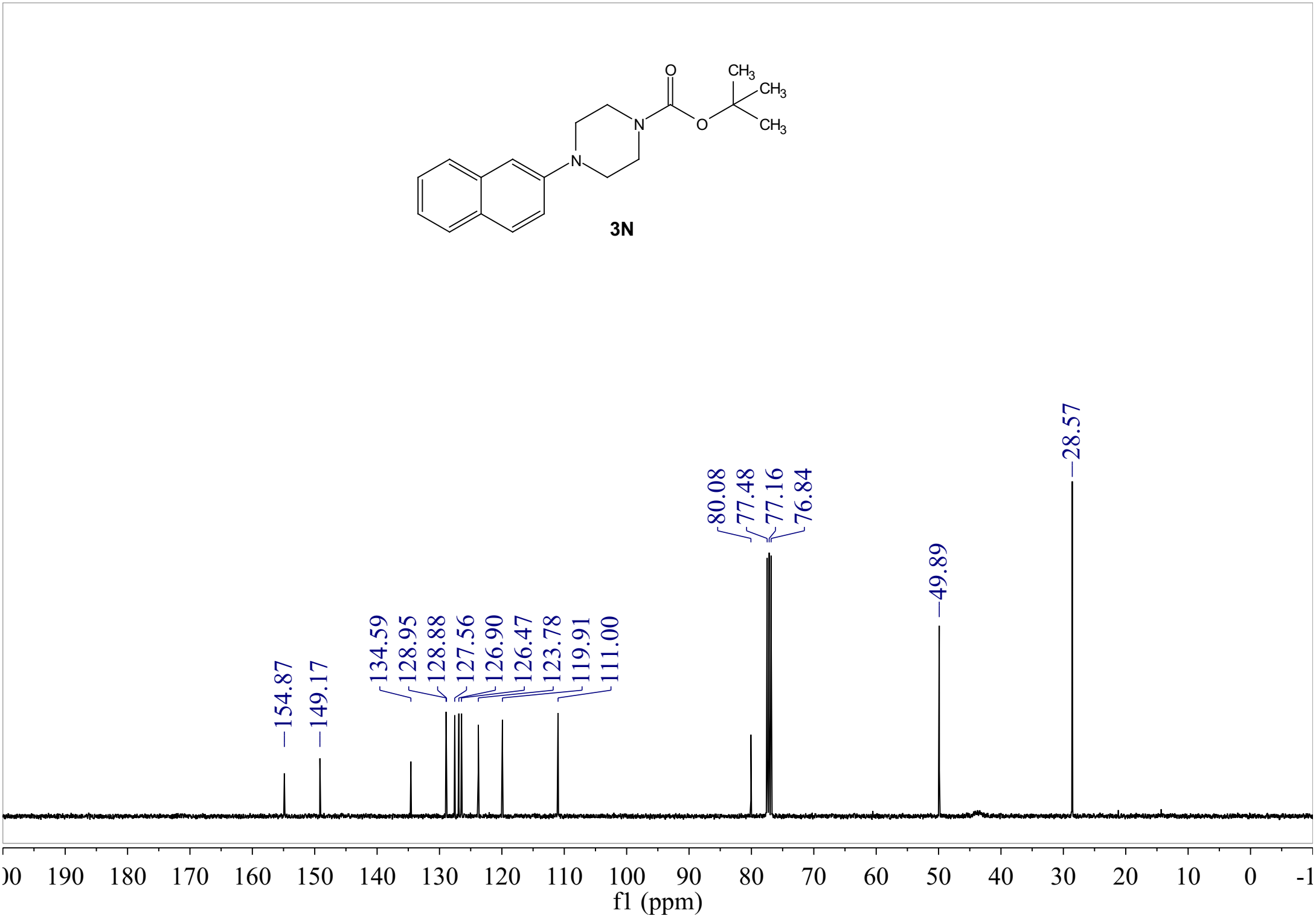
f1 (ppm)

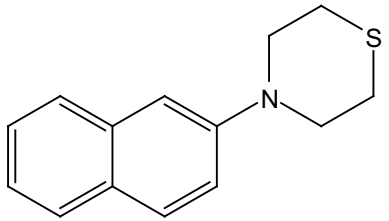

O
N
O
N

3M

77.48
77.16
76.84
136.72
134.54
129.00
128.93
128.67
128.24
128.11
127.57
126.92
126.52
123.87
119.94
111.11
155.35
149.04
67.42
49.88
43.89
f1 (ppm)

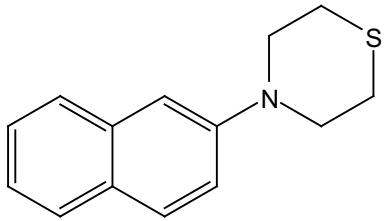

O
CH3
CH3
CH3
N
N
O
3N
1.50
7.75
7.74
7.73
7.71
7.70
7.68
7.43
7.41
7.39
7.33
7.31
7.29
7.27
7.27
7.26
7.25
7.24
7.13
7.12
3.65
3.63
3.62
3.24
3.23
3.22
3.0
1.0
2.4
1.0
4.0
4.0
9.0
f1 (ppm)


O
CH3
CH3
CH3
N
O
N
3N
154.87
149.17
134.59
128.95
128.88
127.56
126.90
126.47
123.78
119.91
111.00
80.08
77.48
77.16
76.84
49.89
28.57
f1 (ppm)


S
N

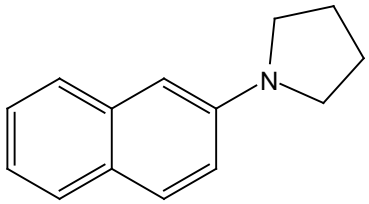
3O

7.75
7.74
7.73
7.72
7.70
7.68
7.42
7.42
7.40
7.33
7.33
7.31
7.31
7.26
7.23
7.23
7.21
7.20
7.13
7.13
3.65
3.64
3.63
2.83
2.83
2.82
2.81
2.81
3.0
1.0
1.0
1.0
1.0
4.0
4.1
f1 (ppm)


S
N

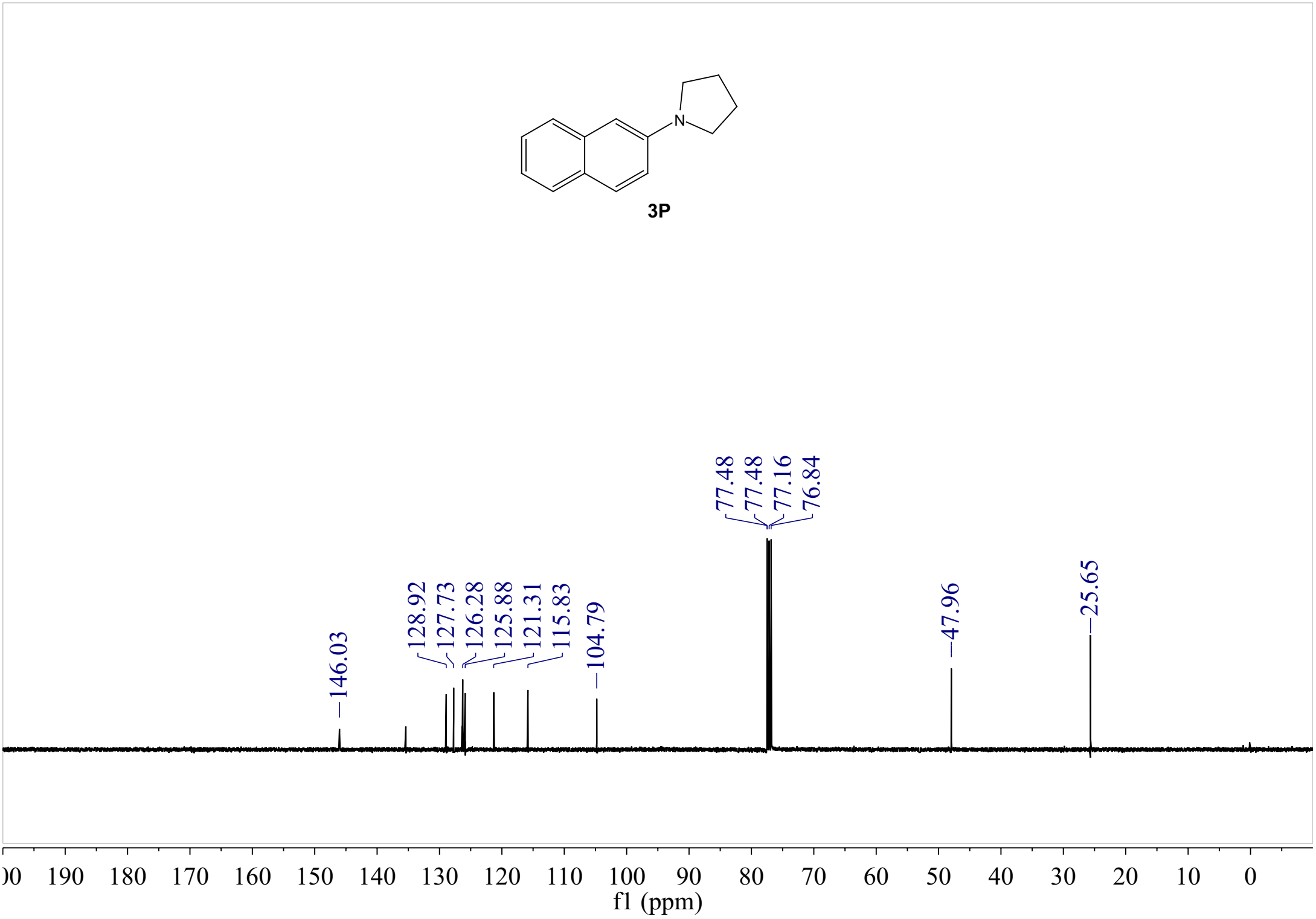
3O

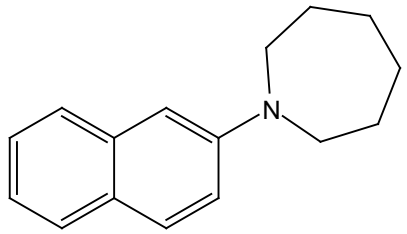
149.24
134.68
129.04
128.64
127.55
126.82
126.46
123.68
120.33
111.45
77.48
77.16
76.84
52.53
27.11
f1 (ppm)


N

3P

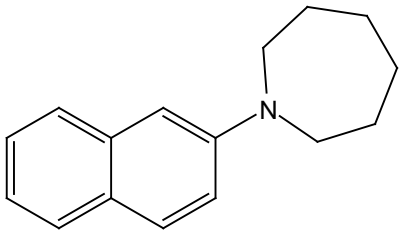
7.70
7.68
7.67
7.64
7.62
7.36
7.36
7.35
7.34
7.34
7.33
7.32
7.26
7.17
7.17
7.16
7.15
7.15
7.14
7.13
7.02
7.01
6.99
6.99
6.76
6.76
3.43
3.42
3.41
3.41
3.40
2.08
2.07
2.06
2.05
2.04
3.12
1.05
1.00
1.00
1.00
4.03
4.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


N
3P
146.03
128.92
127.73
126.28
125.88
121.31
115.83
104.79
77.48
77.48
77.16
76.84
47.96
25.65
f1 (ppm)


N

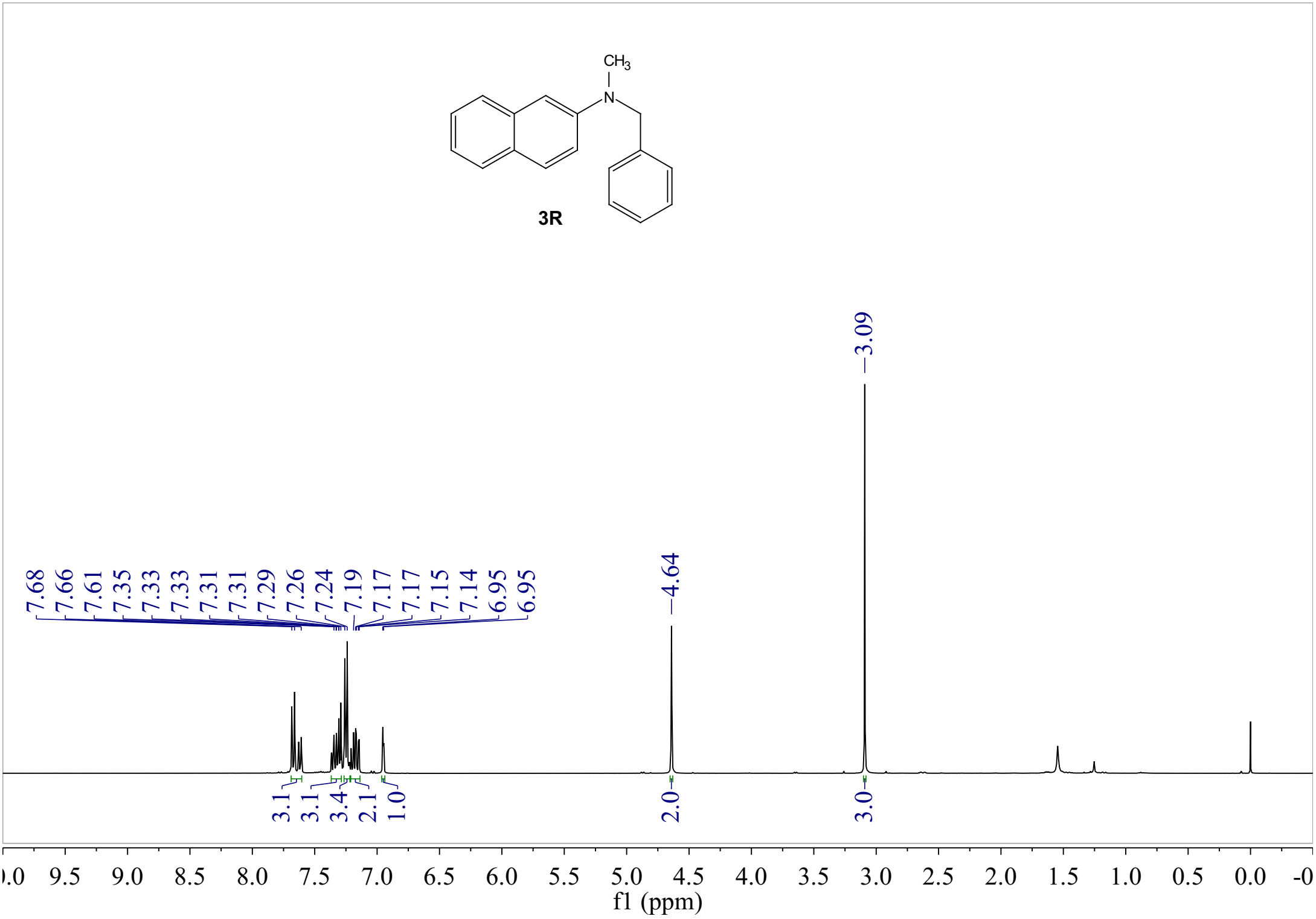
3Q

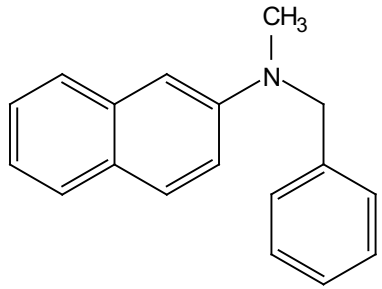
7.69
7.67
7.65
7.62
7.60
7.36
7.36
7.34
7.34
7.33
7.32
7.32
7.26
7.17
7.17
7.16
7.15
7.15
7.13
7.13
7.11
7.10
6.88
6.87
3.60
3.58
3.57
1.88
1.87
1.87
1.86
1.86
1.86
1.85
1.84
1.84
1.60
1.59
1.58
1.57
1.56
1.56
2.0
1.0
1.0
2.1
1.0
4.1
4.1
4.1
f1 (ppm)



3Q

146.90
128.96
127.53
126.22
125.95
121.39
115.28
104.57
77.48
77.16
76.84
49.64
27.98
27.16
f1 (ppm)

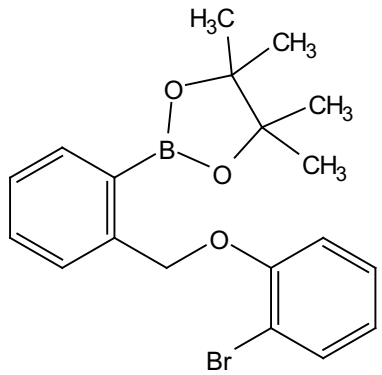

CH3
N
3R
7.68
7.66
7.61
7.35
7.33
7.33
7.31
7.31
7.29
7.26
7.24
7.19
7.17
7.17
7.15
7.14
6.95
6.95
4.64
3.09
3.1
3.1
3.4
2.1
1.0
2.0
3.0
f1 (ppm)



3R

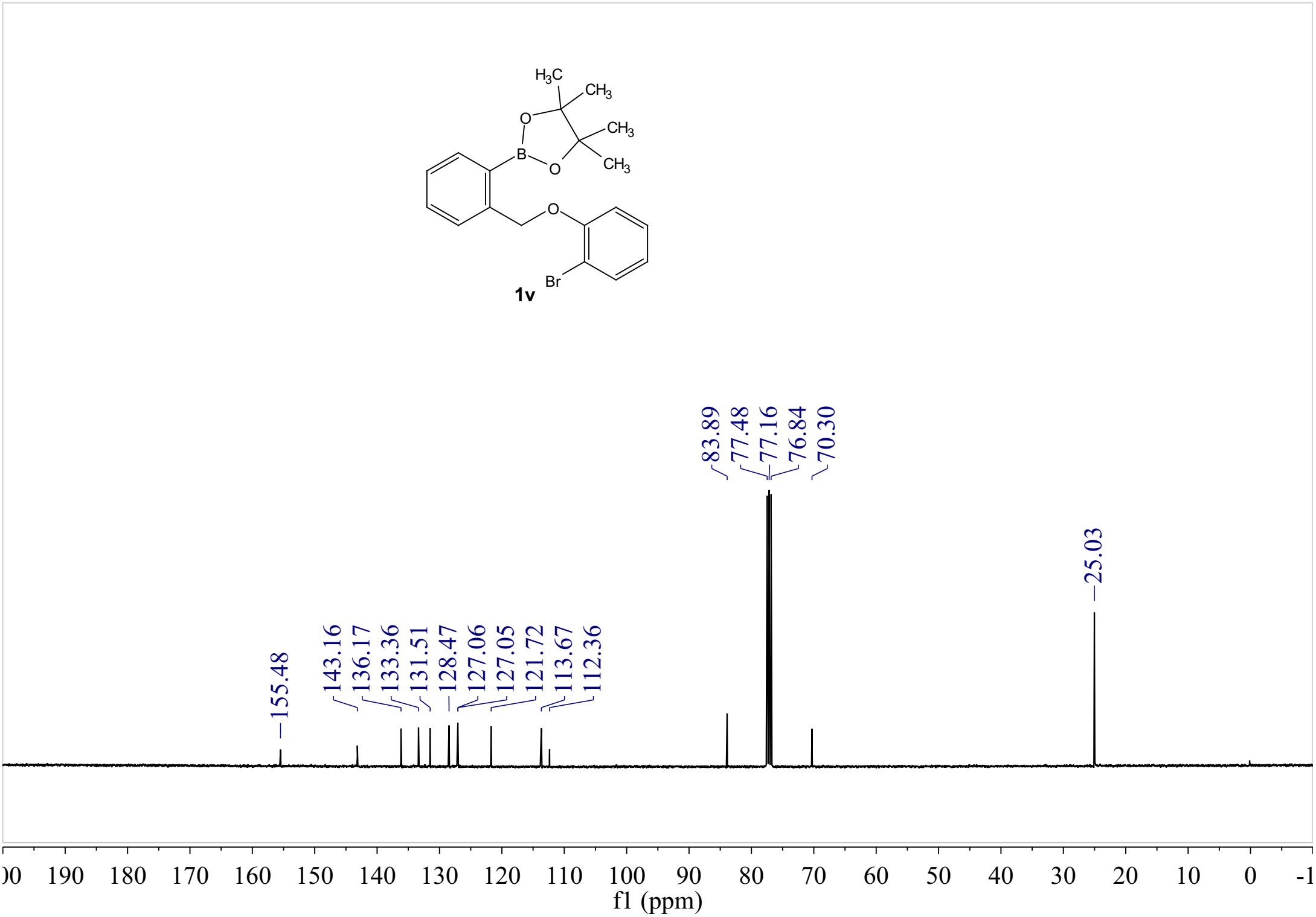
147.83
138.95
135.16
129.00
128.72
127.57
127.09
126.98
126.94
126.37
126.26
122.13
116.33
106.28
77.48
77.16
76.84
56.87
38.80

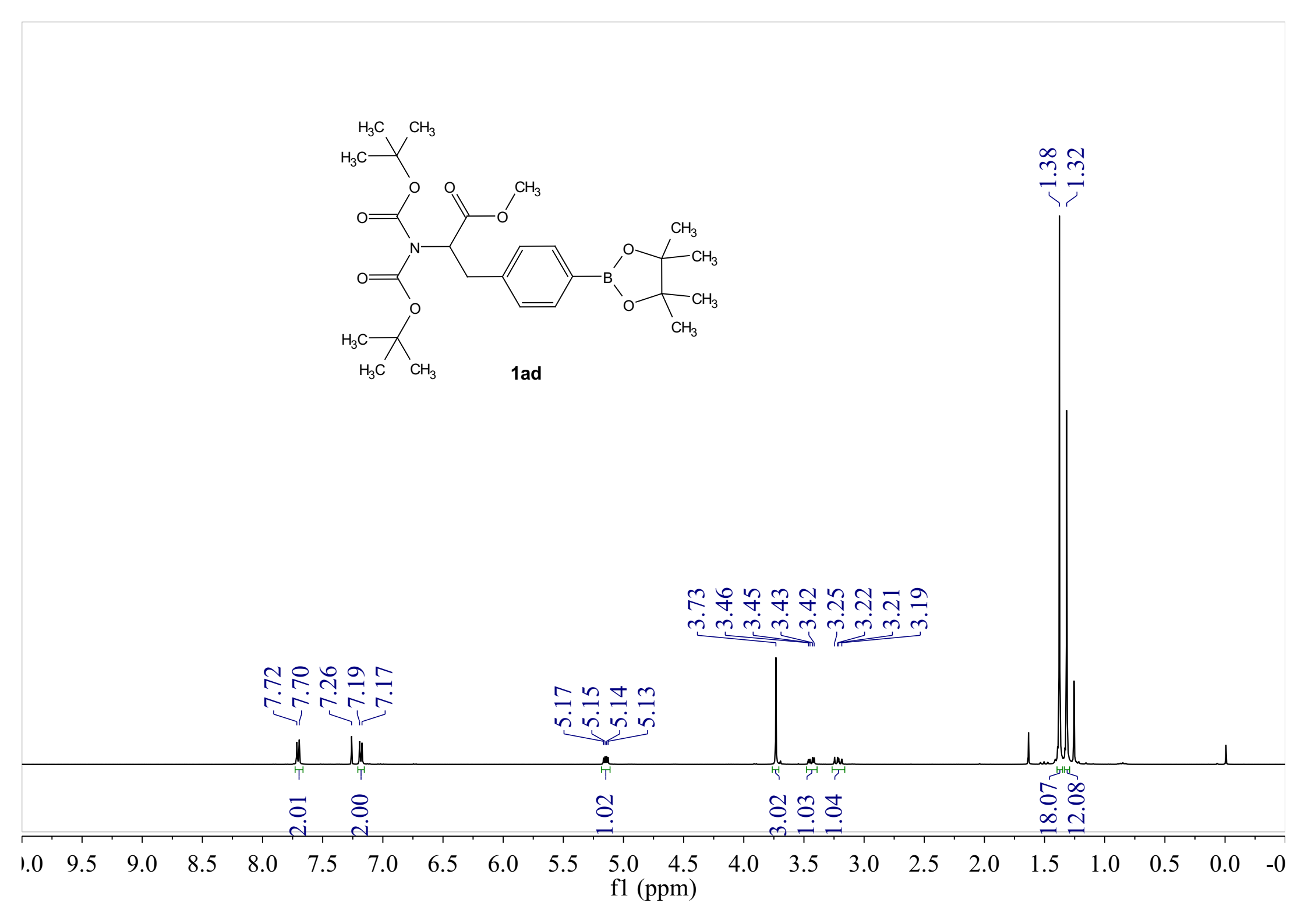
f1 (ppm)

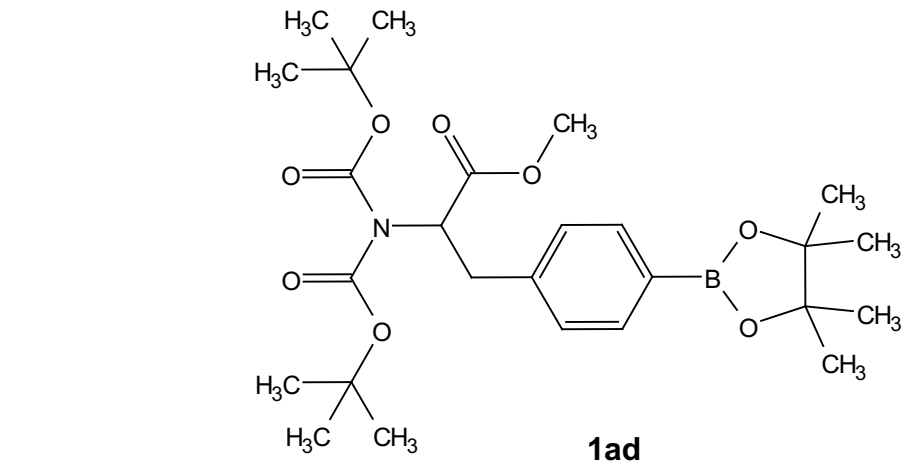

H3C
CH3
O
CH3
B
O
CH3
O
Br

1v

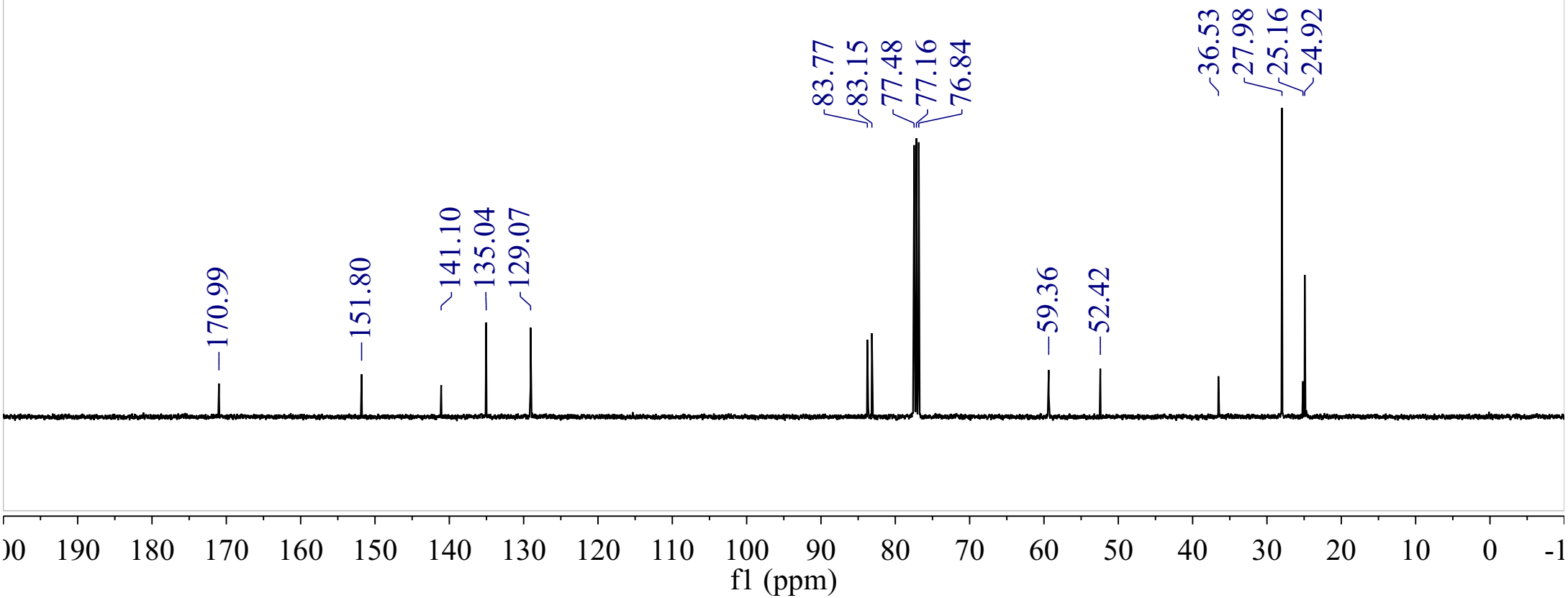
1.32
5.45
7.88
7.87
7.67
7.65
7.56
7.56
7.54
7.54
7.48
7.32
7.26
7.24
7.22
6.98
6.96
6.83
1.00
1.01
0.99
1.02
1.03
1.19
1.02
1.01
2.06
12.25
f1 (ppm)

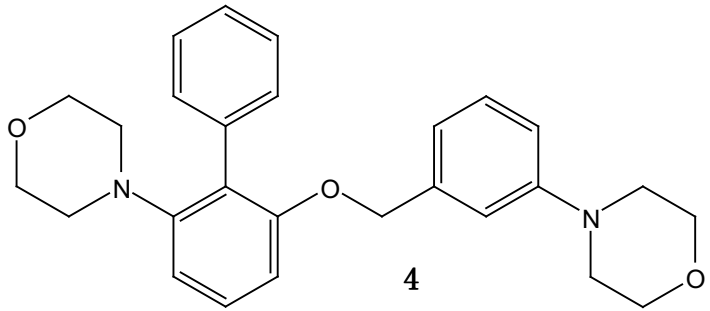

H3C
CH3
CH3
CH3
O
B
O
O
Br
1v
155.48
143.16
136.17
133.36
131.51
128.47
127.06
127.05
121.72
113.67
112.36
83.89
77.48
77.16
76.84
70.30
25.03
f1 (ppm)


1ad
7.72
7.70
7.26
7.19
7.17
5.17
5.15
5.14
5.13
3.73
3.46
3.45
3.43
3.42
3.25
3.22
3.21
3.19
1.38
1.32
2.01
2.00
1.02
3.02
1.03
1.04
18.07
12.08
f1 (ppm)



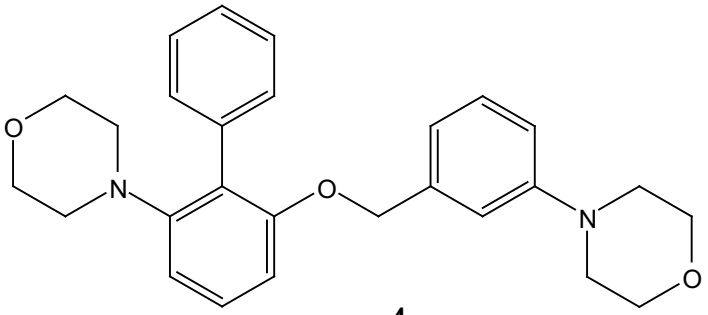
1ad


170.99
151.80
141.10
135.04
129.07
83.77
83.15
77.48
77.16
76.84
59.36
52.42
36.53
27.98
25.16
24.92
f1 (ppm)

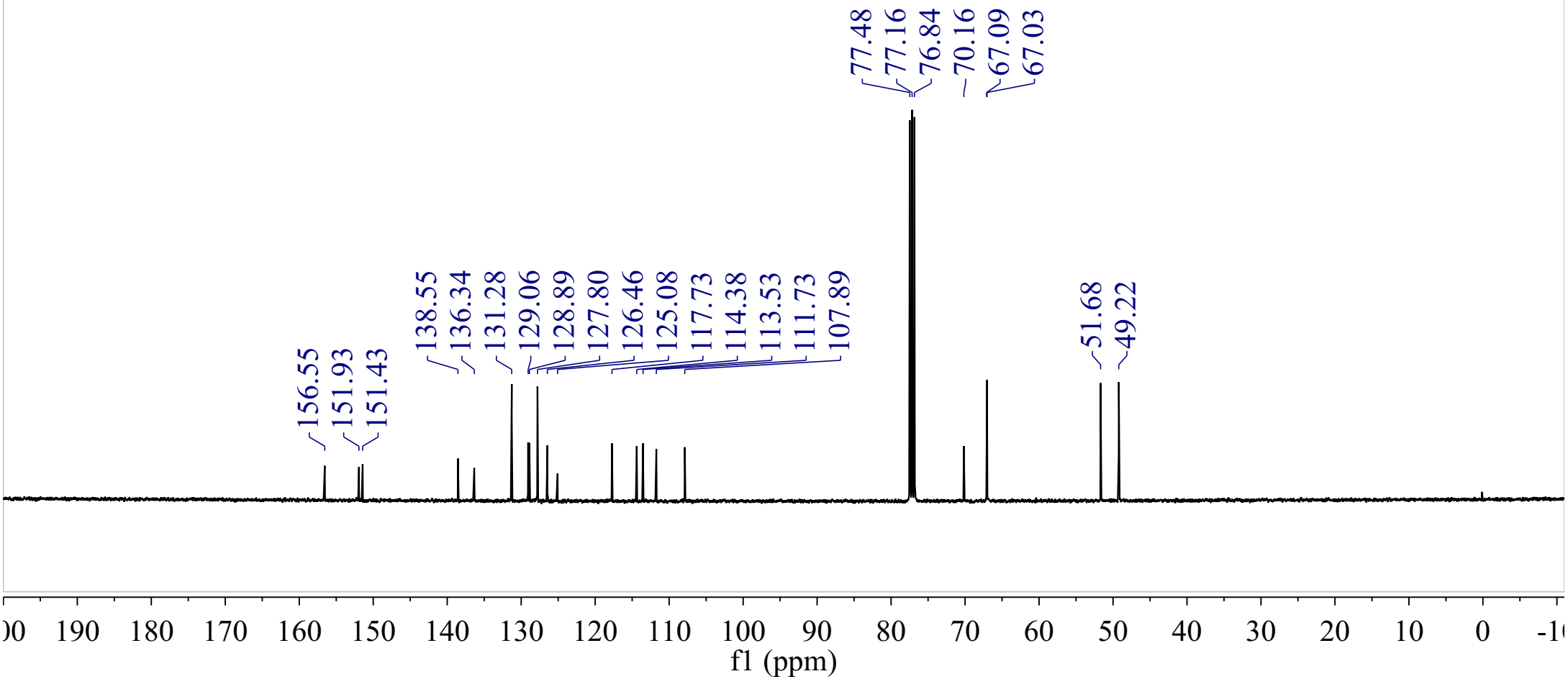

O
N
O
N
O

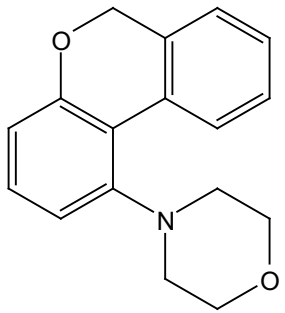
4

7.50
7.50
7.48
7.48
7.40
7.38
7.36
7.28
7.28
7.26
7.25
7.24
7.17
7.15
6.79
6.76
6.73
6.71
6.69
4.97
3.84
3.83
3.82
3.48
3.47
3.46
3.01
2.99
2.98
2.78
2.77
2.76
2.00
2.01
2.36
1.03
5.05
2.00
4.01
4.02
4.03
4.07
f1 (ppm)

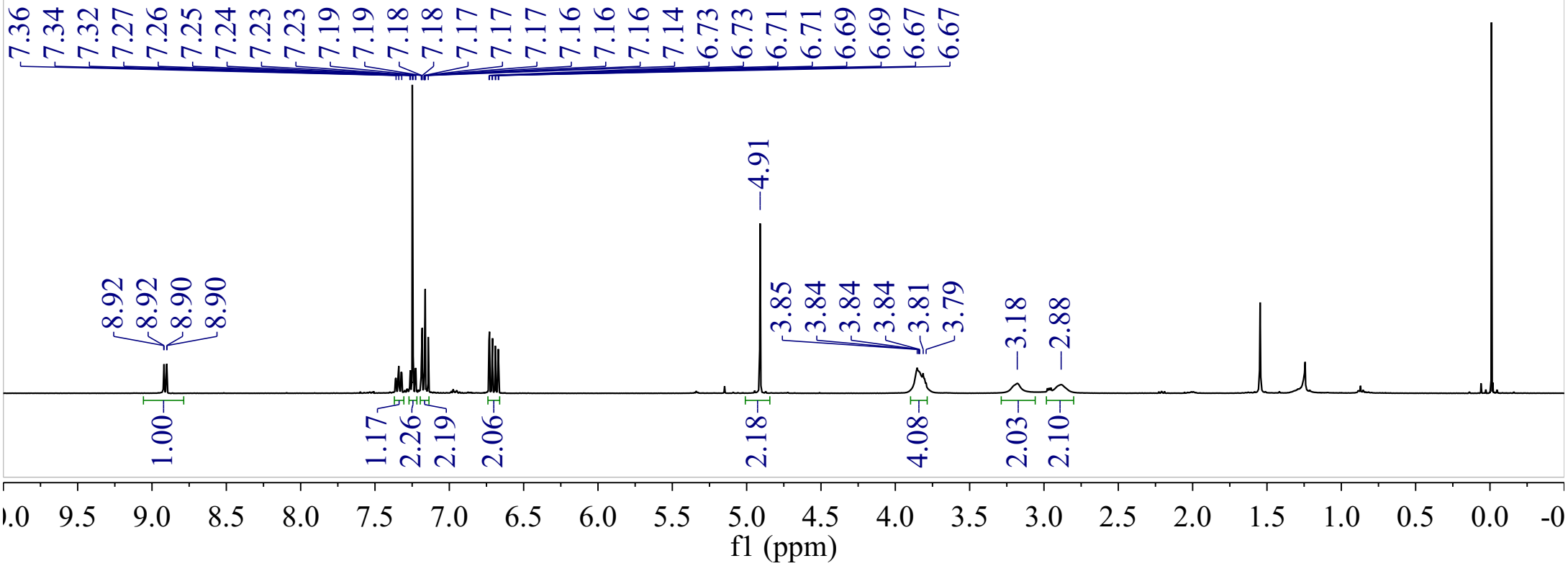

O
N
O
N
O

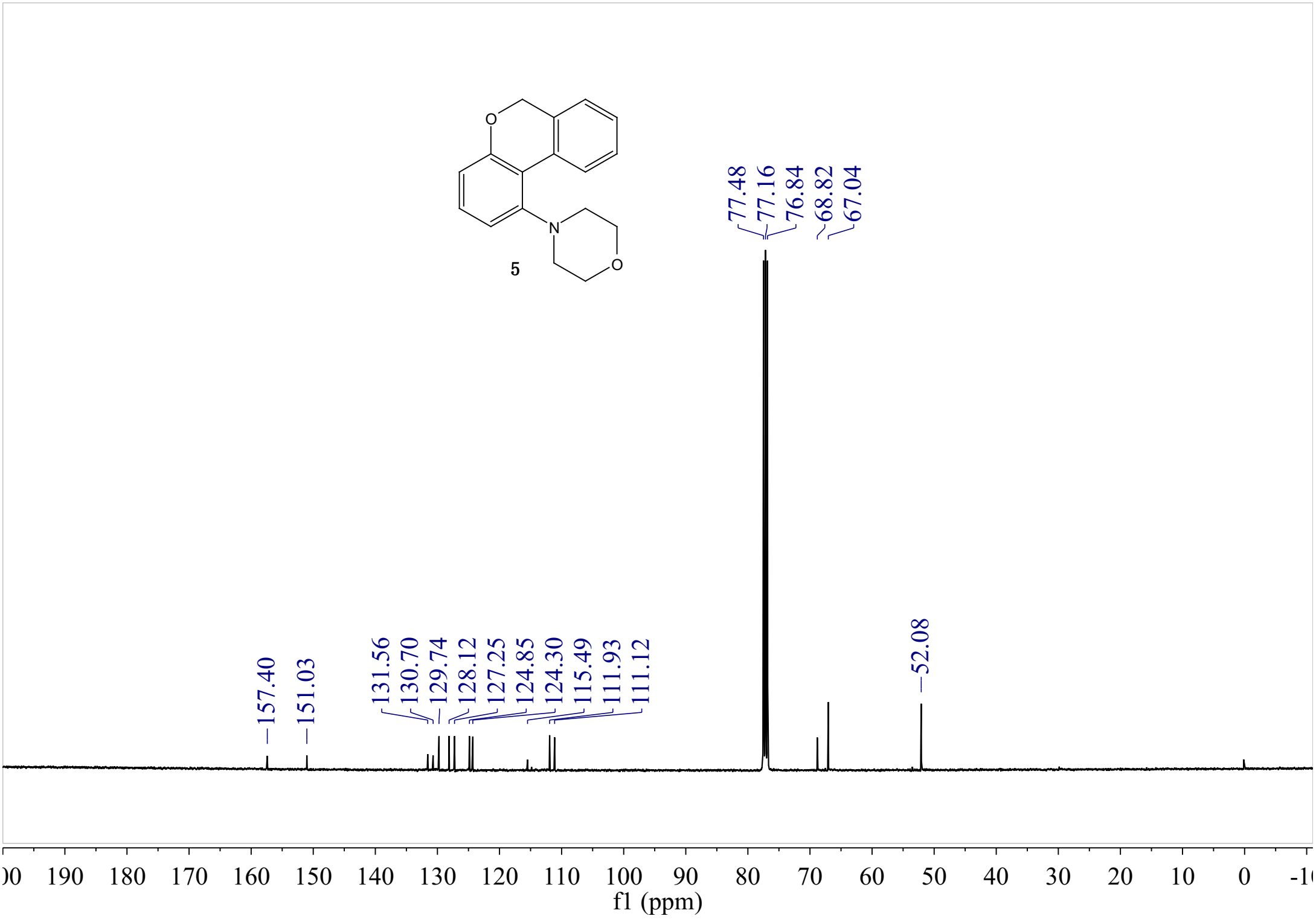
4

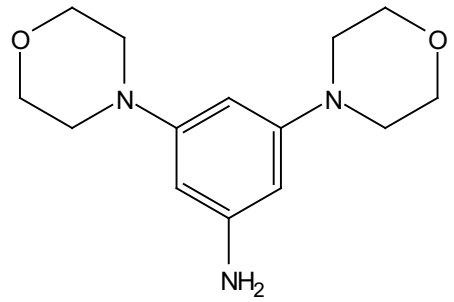

77.48
77.16
76.84
70.16
67.09
67.03
156.55
151.93
151.43
138.55
136.34
131.28
129.06
128.89
127.80
126.46
125.08
117.73
114.38
113.53
111.73
107.89
51.68
49.22
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
N
O

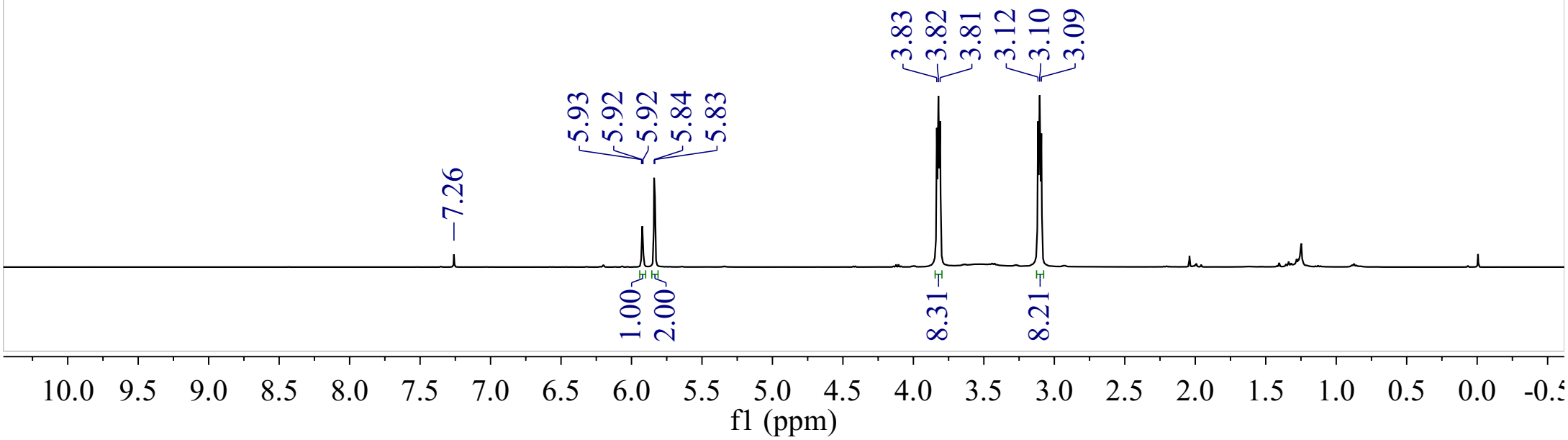
5

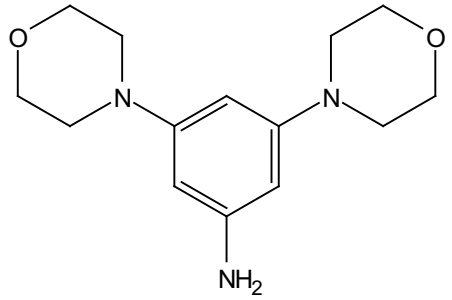

7.36
7.34
7.32
7.27
7.26
7.25
7.24
7.23
7.23
7.19
7.19
7.18
7.18
7.17
7.17
7.17
7.16
7.16
7.16
7.14
6.73
6.73
6.71
6.71
6.69
6.69
6.67
6.67
8.92
8.92
8.90
8.90
4.91
3.85
3.84
3.84
3.84
3.81
3.79
3.18
2.88
1.00
1.17
2.26
2.19
2.06
2.18
4.08
2.03
2.10
f1 (ppm)


O
N
O
5
77.48
77.16
76.84
68.82
67.04
157.40
151.03
131.56
130.70
129.74
128.12
127.25
124.85
124.30
115.49
111.93
111.12
52.08
f1 (ppm)


O
N
N
O
NH2

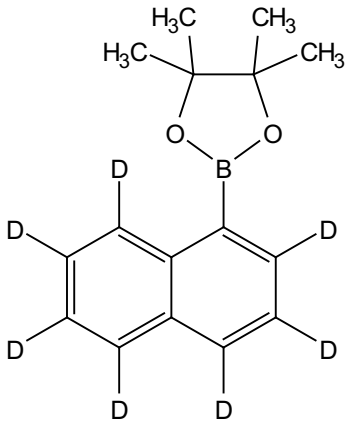
8


7.26
5.93
5.92
5.92
5.84
5.83
3.83
3.82
3.81
3.12
3.10
3.09
1.00
2.00
8.31
8.21
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

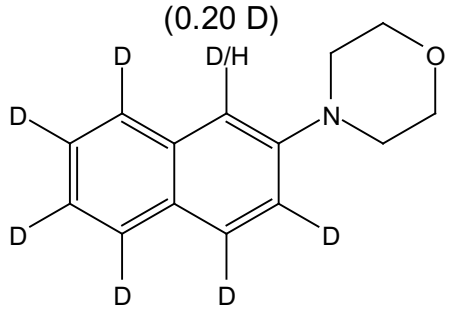

O
N
N
O
NH2

8

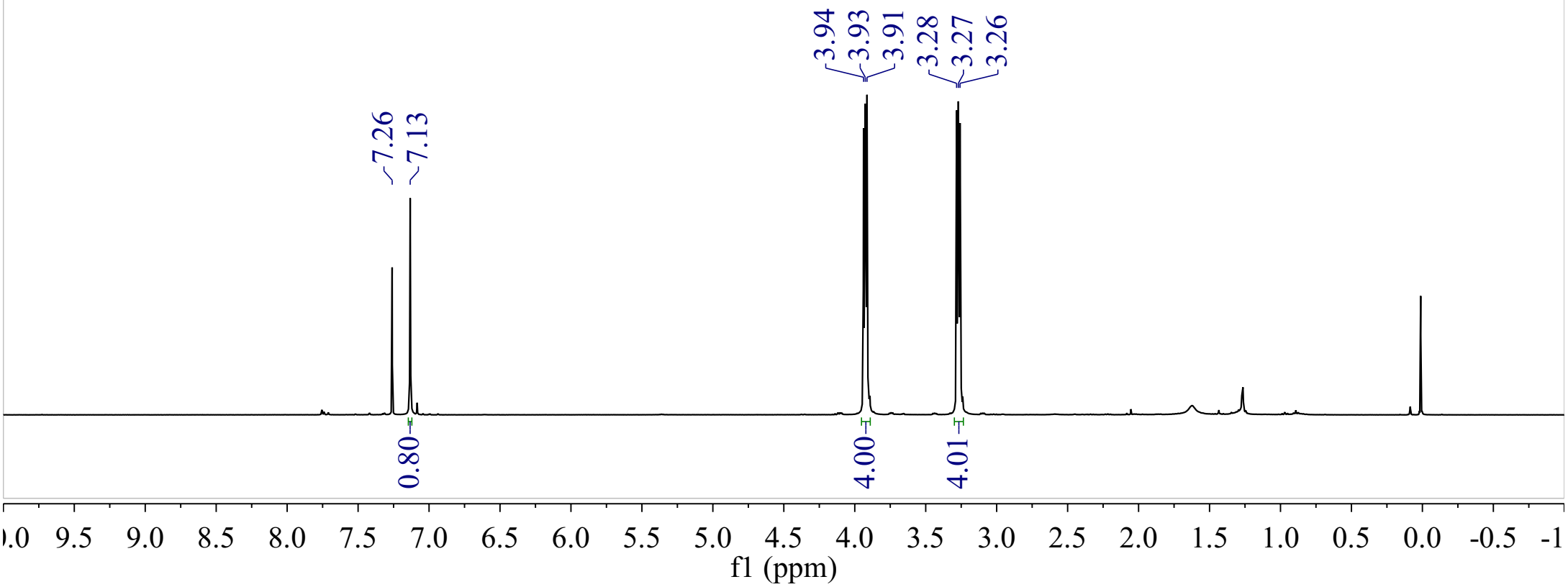
153.50
148.06
95.78
95.31
77.48
77.16
76.84
67.06
49.73
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

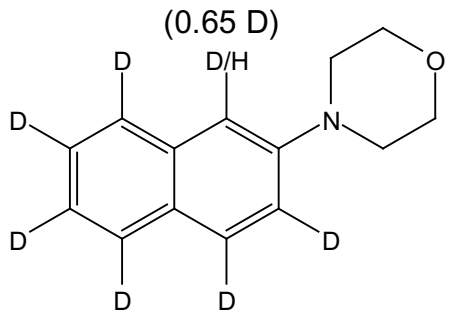


1a-*d*


(0.20 D)
D
D/H
O
N
D
D
D
D
D

3a-*d*


7.26
7.13
3.94
3.93
3.91
3.28
3.27
3.26
0.80
4.00
4.01
f1 (ppm)



3a-*d*