Supporting Information

Direct Carbonylative Amidation of Benzylic Alcohols with Alkylamines via Palladium Catalyzed C-O Bond Activation

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Contents

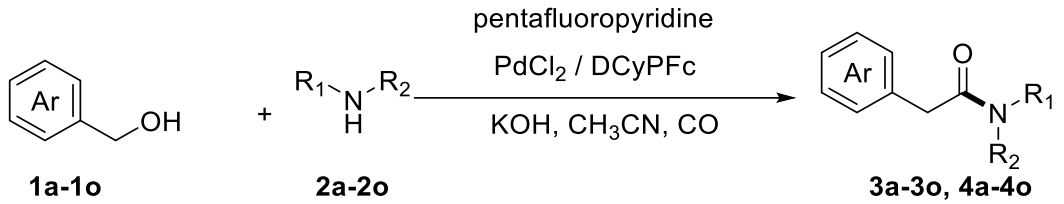
1. General comments

Chemicals were purchased from Enery Chemical, Adamas, Bidepharm., TCI, Aladdin and used as such unless stated otherwise. All solvents like acetonitrile, tetrahydrofuran, N, N-dimethylmethanamide, 1,4-dioxane were purchased from Adamas (Water ≤ 30 ppm (by K.F.), 99.9%, SafeDry, with molecular sieves, Safeseal). NMR spectra were recorded on Bruker AV 400 or Bruker Fourier 300 spectrometer. Chemical shifts (ppm) are given relative to TMS (0.00 ppm) for 1H and $CDCl_3$ (77.0 ppm), DMSO-d6 (39.5 ppm) for 13C solvent. Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublet), m (multiplet) and br.s (broad singlet). High-resolution mass spectra HRMS spectra were recorded on a Thermo Scientific Exactive Orbitrap Mass Spectrometer under Electron Spray Ionization conditions preparing sample solution in methanol. The data are given as mass units per charge (m/z). GC yields were calculated using dodecane as an internal standard. Gas chromatography analysis was performed on an Agilent 6820 instrument with an FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d. 0.25 μm film thickness) using nitrogen as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel., 54-74 μm, 200-300 mesh (Yucheng Chemical CO., LTD, Shanghai).

NOTE: Because of the high toxicity of carbon monoxide, all the reactions should be performed in an autoclave. The laboratory should be well-equipped with a CO detector and alarm system.

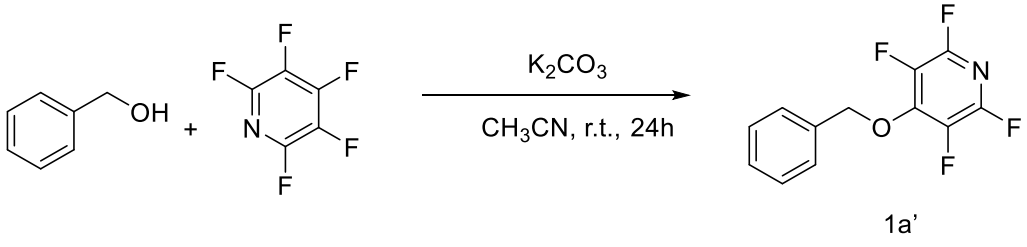
2. General Procedures

2.1 General Procedures for the palladium-catalyzed carbonylation of benzyl alcohols with amines:



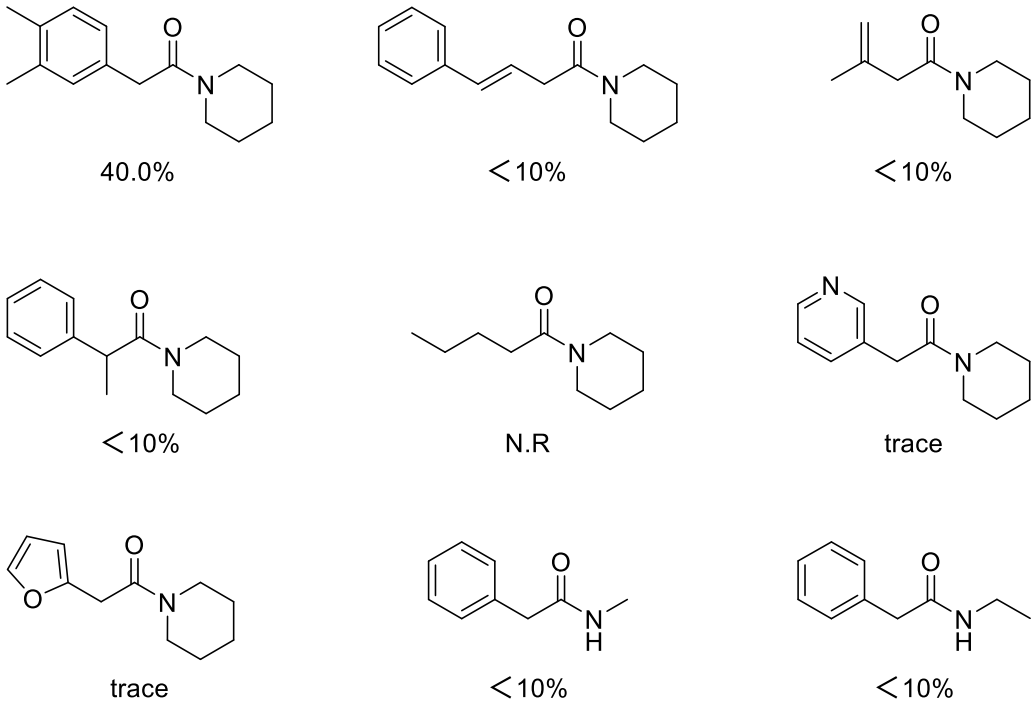
A flame-dried glass pressure tube (5 mL) was charged with KOH (0.5 mmol, 2.5 eq, 28 mg), CH_3CN (2 mL), then was added pentafluoropyridine (0.3 mmol, 1.5 equiv, 33 μL), aromatic benzyl alcohol compounds (1a-1o, 0.2 mmol, 1.0 equiv) and CH_3CN (2 mL) were added through the septum and stirred at room temperature for 5 h. After that $PdCl_2$ (5 mol%, 1.8 mg) and DCyPFc (1,1'-Bis(dicyclohexylphosphino)ferrocene, 10 mol %, 11.6 mg) were added to the tube. Then the glass pressure tube was capped with a PTFE septum, evacuated, and backfilled with nitrogen three times, amine compounds (2a-2o, 0.5 mmol, 2.5 equiv.) was added via the septum. The glass pressure tube was placed in an aluminum heating block, and a needle was inserted through the septum of each tube to maintain pressure equilibrium during subsequent CO exposure. The aluminum heating block was then transferred to an autoclave. The autoclave was purged three times with nitrogen and once with CO. After sealing, the system was pressurized with CO (5 bar), heated to 110 °C and stirred for 20 h. Upon completion, the autoclave was cooled to room temperature, carefully vented in a fume hood to release CO, and then purged three times with nitrogen to remove residual gas. The reaction mixture was diluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (eluent: PE/EA = 4:1 - 3:1) to afford the desired product 3a-3o and 4a-4o.

2.2 General Procedures for the synthesis of substrate 1a' [1]:



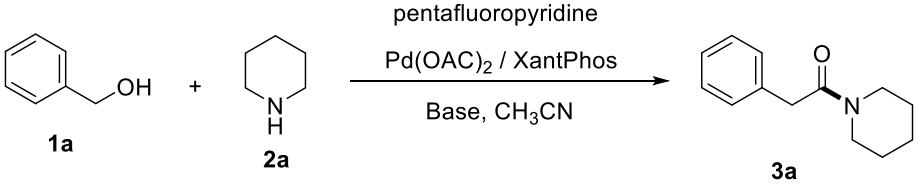
To a 100 mL round-bottomed flask with a stir bar was added potassium carbonate (3.46g, 2.5 eq, 25 mmol, 3.46g), CH_3CN (30 mL), then was added benzyl alcohol (1.0 eq, 10.0 mmol, 1.04 mL), followed by the dropwise addition of pentafluoropyridine (1.2 eq, 12.0 mmol, 1.32 mL). The reaction mixture was stirred for 24 h at room temperature. After the reaction was complete, the reaction mixture was filtered through fret funnel and the filtrate obtained was concentrated on rotavapor. The crude reaction mixture obtained was then purified by flash chromatography on a silica-gel column to obtain corresponding substrate.

2.3 Failed examples



3. Optimization of reaction conditions

3.1 Optimization of the base in one-pot reactions

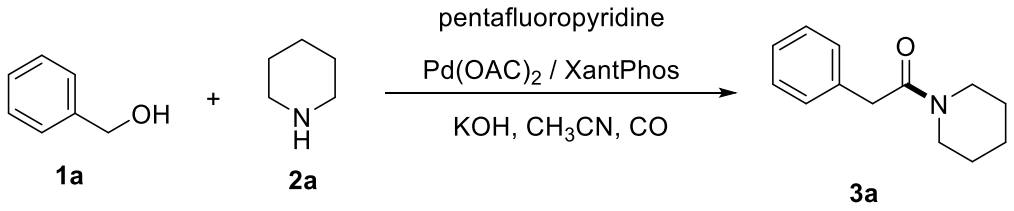


Entry	Base	Yield (%)

1	**KOH 2.5 eq**	**37.4**
2	NaOH 2.5 eq	26.3
3	NaH 2.5 eq	Trace
4	LiOH 2.5 eq	Trace
5	t-BuOK 2.5 eq	8.1
6	t-BuONa 2.5 eq	Trace
7	K_2CO_3 2.5 eq	33.2
8	K_3PO_4 2.5 eq	Trace
9	$KHCO_3$ 2.5 eq	Trace
10	$NaHCO_3$ 2.5 eq	Trace
11	Et_3N 2.5 eq	Trace
12	Piperidine 2.5 eq	Trace
13	KOH 1.5 eq	27.6
14	KOH 3.5 eq	10.1

Reaction conditions: Step 1: 1a (0.1 mmol, 1.0 equivalent, 10 μL), pentafluoropyridine (0.2 mmol, 2.0 equivalent, 22 μL), Base, CH_3CN (1 mL), N_2, r.t. and 5h, Step 2: 2a (0.2 mmol, 2.0 equivalent, 20 μL), $Pd(OAC)_2$ (5 mol%, 1.1 mg), Xphos (10 mol%, 5.8 mg), CO(5 bar), 110 °C and 20 h. N.R. = no reaction.

3.2 Optimization of reactant equivalents



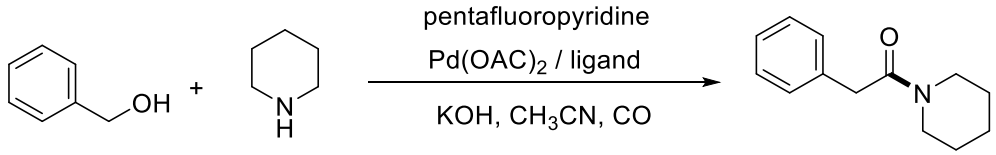
1a **2a** **3a**

Entry	Variable	Yield (%)
1	1a : pentafluoropyridine : 2a = 1.0 : 2.0 : 2.0 eq	37.4
2	1a : pentafluoropyridine : 2a = 1.0 : 1.5 : 3.0 eq	45.5
3	**1a : pentafluoropyridine : 2a = 1.0 : 1.5 :2.5 eq**	**55.6**
4	1a : pentafluoropyridine : 2a = 1.0 : 1.5 :2.0 eq	35.8
5	1a : pentafluoropyridine : 2a = 2.0 :1.5 : 1.5 eq	43.7

6	1a : pentafluoropyridine : 2a = 2.0 :1.5 : 1.0 eq	21.3
7	1a : pentafluoropyridine : 2a = 2.0 :1.5 : 2.0 eq	36.8
8	1a : pentafluoropyridine : 2a = 2.0: 1.0 :1.5 eq	52.3
9	1a : pentafluoropyridine: 2a =2.0: 1.0 : 2.0 eq	28.2
10	1a : pentafluoropyridine : 2a =2.0: 1.0 : 4.0 eq	48.3

Reaction conditions: 1.0 equivalent = 0.1 mmol, Step 1: 1a , pentafluoropyridine, KOH (2.5 equivalent, 14 mg), CH_3CN (1 mL), N_2, r.t. and 5h, Step 2: 2a, $Pd(OAC)_2$ (5 mol%, 1.1 mg), Xphos (10 mol%, 5.8 mg), CO(5 bar), 110 °C and 20 h. N.R. = no reaction.

3.3 Optimization of ligands



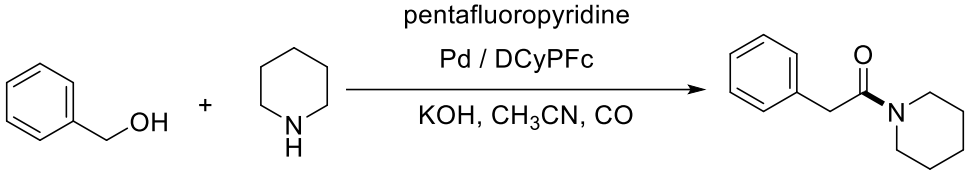
1a **2a** **3a**

Entry	Ligand	Yield(%)
1[a]	XantPhos	55.6
2[a]	DPEPhos	62.6
3[a]	N-Xantphos	40.5
4[a]	[t-Bu_3PH]BF_4	N.R.
5[a]	PPh_3	N.R.
6[a]	IMes • HCl	N.R.
7[a]	TMPP	28.2
8[a]	TFP	Trace
9[a]	SPhos	N.R.
10[a]	t-Bu XPhos	N.R.
11[a]	DtBPF	N.R.
12[a]	**DCyPFc**	**85.5**
13[a]	（$BuPAd_2$）*2	N.R.
14[a]	BrettPhos	42.2
15[a]	DPPF	79.4

16[a]	DCyPE	38.4
17[b]	DCyPFc	84.1

[a] Reaction conditions: Step 1: 1a (0.1 mmol, 1.0 equivalent, 10 μL), pentafluoropyridine (0.15 mmol, 1.5 equivalent, 16 μL), KOH (2.5 equivalent, 14 mg), CH_3CN (1 mL), N_2, r.t. and 5h, Step 2: 2a (0.25 mmol, 2.5 equivalent, 24 μL), $Pd(OAC)_2$ (5 mol%, 1.1 mg), ligand (10 mol%), CO(5 bar), 110 °C and 20 h. N.R. = no reaction. [b] DCyPFc (20 mol %, 23.1 mg)

3.4 Optimization of palladium catalysts

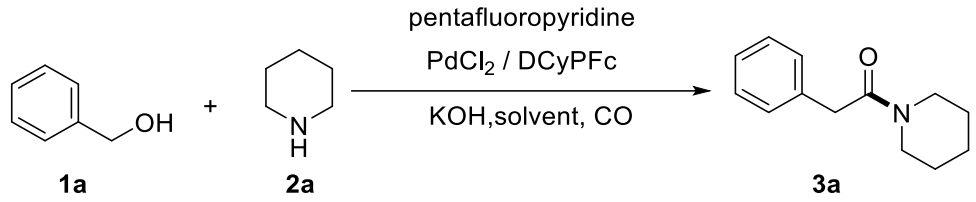


1a **2a** **3a**

Entry	Pd catalysts	Yield (%)
1	$Pd(OAc)_2$	85.5
2	**$PdCl_2$**	**91.2**
3	$Pd(PPh_3)_4$	85.8
4	K_2PdCl_4	87.1
5	$Pd(bipy)Cl_2$	85.9
6	$Pd(PhCN)_2Cl_2$	71.1
7	K_2PdCl_4	64.7
8	$Pd(PPh_3)_2Cl_2$	65.5
9	$Pd(PhCN)_2Cl_2$	87.6
10	$Pd(PPh_3)_2Cl_2$	77.1

Reaction conditions: Stesp 1: 1a (0.1 mmol, 1.0 equivalent, 10 μL), pentafluoropyridine (0.15 mmol, 1.5 equivalent, 16 μL), KOH(2.5 equivalent, 14 mg), CH_3CN (1 mL), N_2, r.t. and 5h, Step 2: 2a (0.25 mmol, 2.5 equivalent, 24 μL), palladium catalysts (5 mol%), DCyPFc (10 mol %, 11.6 mg), CO (5 bar), 110 °C and 20 h. N.R. = no reaction.

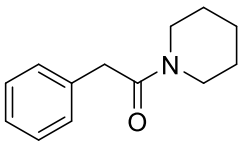
3.5 Optimization of reaction solvents



Entry	Solvent	Yield (%)
1	**CH_3CN**	**91.2**
2	dioxane	40.8
3	DMF	31.8
4	DMAc	42.0
5	Ph-CF_3	47.2
6	DMSO	36.3
7	THF	75.5
8	DCE	Trace
9	toluene	Trace

Reaction conditions: Stesp 1: 1a (0.1 mmol, 1.0 equivalent, 10 μL), pentafluoropyridine (0.15 mmol, 1.5 equivalent, 16 μL), KOH(2.5 equivalent, 14 mg), CH_3CN (1 mL), N_2, r.t. and 5h, Step 2: 2a (0.25 mmol, 2.5 equivalent, 24 μL), palladium catalysts (5 mol%), DCyPFc (10 mol %, 11.6 mg), CO (5 bar), 110 °C and 20 h. N.R. = no reaction.

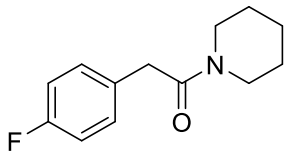
4. Characterization data of products



2-phenyl-1-(piperidin-1-yl)ethan-1-one (3a): (63.4 mg, light yellow oil, yield: 78%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [2]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.14 (m, 5H), 3.71 (s, 2H), 3.54 (t, J = 5.5 Hz, 2H), 3.34 (t, J = 5.5 Hz, 2H), 1.62 – 1.42 (m, 4H), 1.38 – 1.25 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 135.3, 128.6, 128.5, 126.6, 47.2, 42.8, 41.1, 26.1, 25.4, 24.3.

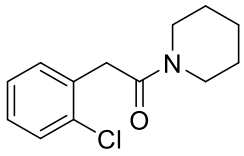


2-(4-fluorophenyl)-1-(piperidin-1-yl)ethan-1-one(3b): (56.6 mg, light yellow oil, yield: 64%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 – 7.12 (m, 2H), 6.97 (t, *J* = 8.5 Hz, 2H), 3.66 (s, 2H), 3.54 (t, *J* = 5.4 Hz, 2H), 3.35 (t, *J* = 5.5 Hz, 2H), 1.61 – 1.45 (m, 4H), 1.41 – 1.31 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.9, 161.6 (d, *J* = 244.9 Hz), 131.1 (d, *J* = 3.2 Hz), 130.1 (d, *J* = 7.9 Hz), 115.3 (d, *J* = 21.5 Hz), 47.1, 42.8, 40.0, 26.2, 25.4, 24.3.

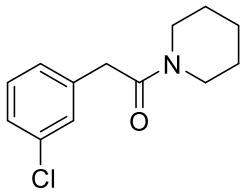
^{19}F NMR (565 MHz, $CDCl_3$) δ -116.4.



(2-chlorophenyl)-1-(piperidin-1-yl)ethan-1-one(3c): (46.6 mg, light yellow oil, yield: 49%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [4]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.22 – 7.15 (m, 2H), 3.80 (s, 2H), 3.61 – 3.54 (m, 2H), 3.39 – 3.35 (m, 2H), 1.63 – 1.57 (m, 2H), 1.54 (t, *J* = 5.9 Hz, 2H), 1.44 (t, 2H).

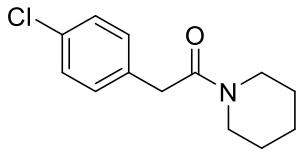
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.3, 130.5, 129.3, 128.8, 128.1, 126.9, 125.2, 47.0, 43.0, 38.0, 26.2, 25.5, 24.4.



2-(3-chlorophenyl)-1-(piperidin-1-yl)ethan-1-one(3d): (81.8 mg, white solid, m.p. = 52-53 ℃, yield: 86%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 3H), 7.12 (d, *J* = 6.7 Hz, 1H), 3.68 (s, 2H), 3.55 (t, *J* = 5.4 Hz, 2H), 3.35 (t, *J* = 5.4 Hz, 2H), 1.61 – 1.48 (m, 4H), 1.44 – 1.33 (m, 2H).

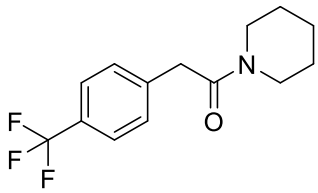
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.4, 137.3, 134.3, 129.7, 128.8, 126.8, 126.8, 47.2, 42.9, 40.4, 26.2, 25.4, 24.3.



2-(4-chlorophenyl)-1-(piperidin-1-yl)ethan-1-one(3e): (76.1 mg, light yellow solid, m.p. = 83-84 ℃, yield: 80%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [5]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 3.66 (s, 2H), 3.54 (t, *J* = 5.4 Hz, 2H), 3.34 (t, *J* = 5.4 Hz, 2H), 1.52 (dt, *J* = 17.4, 5.1 Hz, 4H), 1.36 (p, *J* = 5.6 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.7, 133.8, 132.4, 130.0, 128.6, 47.1, 42.8, 40.1, 26.2, 25.4, 24.3.

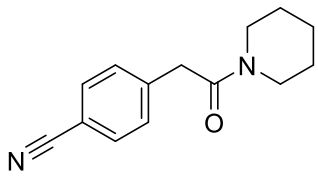


1-(piperidin-1-yl)-2-(4-(trifluoromethyl)phenyl)ethan-1-one(3f): (21.7 mg, white solid, m.p. = 100-102 ℃, yield: 20%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [6]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.9 Hz, 2H), 7.35 (d, *J* = 7.9 Hz, 2H), 3.75 (s, 2H), 3.56 (t, *J* = 5.6 Hz, 2H), 3.37 (t, *J* = 5.6 Hz, 2H), 1.61 – 1.56 (m, 2H), 1.53 – 1.48 (m, 2H), 1.42 – 1.37 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.2, 139.5, 129.1, 129.0 (q, *J* = 32.5 Hz), 125.4 (q, *J* = 3.9 Hz), 124.3 (q, *J* = 272.0 Hz), 47.1, 42.9, 40.5, 26.2, 25.4, 24.3.

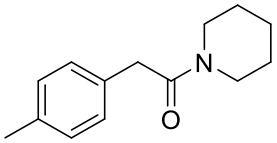
^{19}F NMR (565 MHz, Chloroform-*d*) δ -62.5.



4-(2-oxo-2-(piperidin-1-yl)ethyl)benzonitrile(3g): (29.2 mg, light yellow oil, yield: 32%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [3]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 3.73 (s, 2H), 3.53 (t, *J* = 5.6 Hz, 2H), 3.36 (t, *J* = 5.6 Hz, 2H), 1.61 – 1.56 (m, 2H), 1.52 – 1.47 (m, 2H), 1.44 – 1.38 (m, 2H).

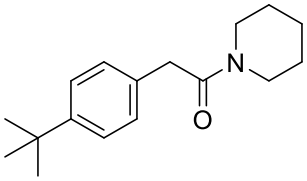
^{13}C NMR (151 MHz, $CDCl_3$) δ 167.6, 140.9, 132.1, 129.7, 118.7, 110.5, 47.0, 42.9, 40.5, 26.2, 25.3, 24.2.



1-(piperidin-1-yl)-2-(p-tolyl)ethan-1-one(3h): (43.5 mg, white solid, m.p. = 59-60 ℃ yield: 50%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.15 – 7.06 (m, 4H), 3.66 (s, 2H), 3.54 (t, *J* = 5.4 Hz, 2H), 3.34 (t, *J* = 5.6 Hz, 2H), 2.29 (s, 3H), 1.60 – 1.42 (m, 4H), 1.38 – 1.26 (m, 2H).

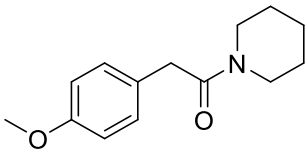
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.2, 135.9, 132.2, 129.1, 128.2, 47.0, 42.6, 40.6, 26.0, 25.3, 24.3, 20.8.



2-(4-(tert-butyl)phenyl)-1-(piperidin-1-yl)ethan-1-one(3i): (83.0 mg, light yellow oil, yield: 80%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [7]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.3 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 3.68 (s, 2H), 3.56 (t, *J* = 5.6 Hz, 2H), 3.38 (t, *J* = 5.6 Hz, 2H), 1.60 – 1.56 (m, 2H), 1.54 – 1.49 (m, 2H), 1.40 – 1.35 (m, 2H), 1.30 (s, 9H).

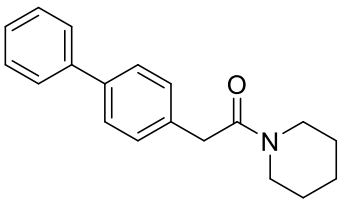
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.4, 149.4, 132.3, 128.2, 125.4, 47.2, 42.8, 40.4, 34.3, 31.3, 26.1, 25.4, 24.4.



2-(4-methoxyphenyl)-1-(piperidin-1-yl)ethan-1-onesss(3j): (31.7 mg, an pale yellow oil, yield: 34%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.2 Hz, 2H), 6.82 (d, *J* = 8.3 Hz, 2H), 3.76 (s, 3H), 3.63 (s, 2H), 3.53 (t, *J* = 5.4 Hz, 2H), 3.34 (t, *J* = 5.6 Hz, 2H), 1.60 – 1.43 (m, 4H), 1.37 – 1.28 (m, 2H).

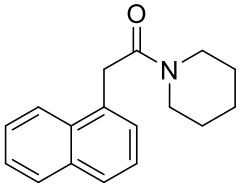
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.4, 158.2, 129.5, 127.4, 113.9, 55.1, 47.1, 42.7, 40.1, 26.1, 25.4, 24.3.



2-([1,1'-biphenyl]-4-yl)-1-(piperidin-1-yl)ethan-1-one(3k): (90.5 mg, white solid, m.p. = 57-59 ℃, yield: 81%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [6]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 – 7.53 (m, 4H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.37 – 7.29 (m, 3H), 3.77 (s, 2H), 3.59 (t, *J* = 5.3 Hz, 2H), 3.41 (t, *J* = 5.6 sHz, 2H), 1.63 – 1.49 (m, 4H), 1.44 – 1.35 (m, 2H).

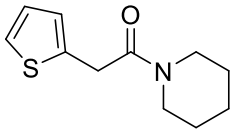
^{13}C NMR (75 MHz, $CDCl_3$) δ 169.1, 140.7, 139.5, 134.4, 129.0, 128.7, 127.3, 127.1, 126.9, 47.2, 42.8, 40.6, 26.2, 25.4, 24.3.



2-(naphthalen-1-yl)-1-(piperidin-1-yl)ethan-1-one(3l): (33.4 mg, light yellow soild, m.p. = 42-43 ℃, yield: 33%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [3]

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.3 Hz, 1H), 7.86 (d, *J* = 7.9 Hz, 1H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 7.1 Hz, 1H), 4.15 (s, 2H), 3.64 (t, *J* = 5.4 Hz, 3H), 3.36 (t, *J* = 5.4 Hz, 3H), 1.63 – 1.53 (m, 4H), 1.44 – 1.33 (m, 2H).

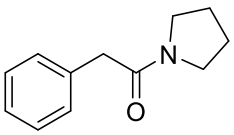
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.2, 133.7, 131.8, 131.5, 128.6, 127.4, 126.1, 126.0, 125.6, 125.3, 123.3, 47.1, 42.8, 38.3, 26.2, 25.5, 24.3.



1-(piperidin-1-yl)-2-(thiophen-2-yl)ethan-1-one(3m): (40.2 mg, light yellow oil, yield: 48%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [8]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 (d, *J* = 5.2 Hz, 1H), 6.92 (t, *J* = 3.9 Hz, 2H), 6.88 (d, *J* = 2.4 Hz, *J* = 2.1 Hz, 1H), 3.88 (s, 2H), 3.56 (t, *J* = 5.4 Hz, 2H), 3.42 (t, *J* = 5.2 Hz, 2H), 1.62 – 1.57 (m, 2H), 1.54 – 1.49 (m, 2H), 1.47 – 1.41 (m, 2H).

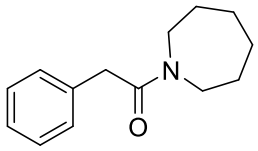
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.0, 136.9, 126.6, 125.8, 124.5, 47.3, 42.9, 35.1, 26.2, 25.3, 24.3.



2-phenyl-1-(pyrrolidin-1-yl)ethan-1-one(4a): (71.2 mg, light yellow oil, yield: 94%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [9]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 – 7.25 (m, 4H), 7.24 – 7.20 (m, 1H), 3.64 (s, 2H), 3.47 (t, *J* = 6.9 Hz, 2H), 3.40 (t, *J* = 6.8 Hz, 2H), 1.93 – 1.86 (m, 2H), 1.85 – 1.78 (m, 2H).

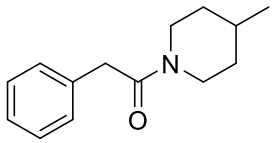
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.4, 134.8, 128.8, 128.5, 126.6, 46.8, 45.8, 42.2, 26.0, 24.2.



1-(azepan-1-yl)-2-phenylethan-1-one(4b): (40.0 mg, light yellow oil, yield: 46%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [10]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 (d, *J* = 14.9 Hz, 2H), 7.23 (d, *J* = 7.4 Hz, 2H), 7.18 (t, *J* = 7.1 Hz, 1H), 3.67 (s, 2H), 3.49 (t, *J* = 6.1 Hz, 2H), 3.38 (t, *J* = 6.1 Hz, 2H), 1.69 – 1.64 (m, 2H), 1.58 – 1.54 (m, 2H), 1.50 – 1.46 (m, 2H), 1.46 – 1.41 (m, 2H).

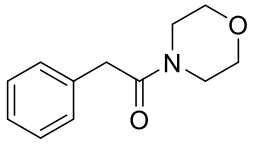
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.3, 135.2, 128.5, 128.4, 126.4, 48.1, 45.8, 40.9, 28.9, 27.2, 27.0, 26.4.



1-(4-methylpiperidin-1-yl)-2-phenylethan-1-one(4c): (54.8 mg, light yellow oil, yield: 63%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [6]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.6 Hz, 2H), 7.20 (dd, *J* = 18.4, 7.5 Hz, 3H), 4.56 (d, *J* = 13.4 Hz, 1H), 3.79 (d, *J* = 13.7 Hz, 1H), 3.69 (s, 2H), 2.89 (t, *J* = 12.7 Hz, 1H), 2.53 (t, *J* = 12.4 Hz, 1H), 1.61 (d, *J* = 13.0 Hz, 1H), 1.50 (d, *J* = 10.5 Hz, 2H), 1.07 – 0.98 (m, 1H), 0.86 (d, *J* = 6.2 Hz, 3H), 0.81 (d, *J* = 10.5 Hz, 1H).

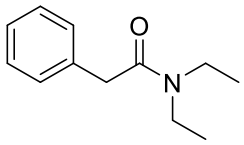
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.0, 135.2, 128.4, 128.4, 126.4, 46.3, 42.0, 41.0, 34.1, 33.5, 30.7, 21.4.



1-morpholino-2-phenylethan-1-one(4d): (36.1mg, white solid, m.p. = 60-63 ℃, yield:: 44%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [11]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.27 (m, 2H), 7.23 (t, *J* = 6.4 Hz, 3H), 3.71 (s, 2H), 3.62 (s, 4H), 3.43 (dt, *J* = 5.8, 4.8 Hz, 4H).

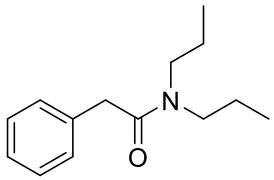
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.5, 134.7, 128.7, 128.4, 126.8, 66.7, 66.3, 46.4, 42.0, 40.7.



N,N-diethyl-2-phenylacetamide(4e): (46.7 mg, light yellow oil, yield: 61%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [12]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.26 – 7.18 (m, 3H), 3.68 (s, 2H), 3.38 (q, *J* = 7.1 Hz, 2H), 3.28 (q, *J* = 7.1 Hz, 2H), 1.11 (t, *J* = 7.1 Hz, 3H), 1.07 (t, *J* = 7.1 Hz, 3H).

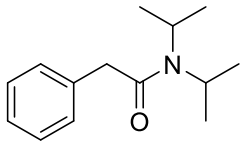
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.0, 135.4, 128.5, 128.5, 126.5, 42.2, 40.8, 40.0, 14.1, 12.8.



2-phenyl-N,N-dipropylacetamide(4f): (54.4 mg, light yellow oil, yield: 62%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [13]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.4 Hz, 2H), 7.26 – 7.19 (m, 3H), 3.69 (s, 2H), 3.28 (t, *J* = 7.6 Hz, 2H), 3.17 (t, *J* = 7.8 Hz, 2H), 1.59 – 1.46 (m, 4H), 0.88 – 0.82 (m, 6H).

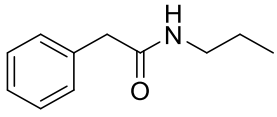
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.4, 135.5, 128.6, 128.4, 126.5, 49.8, 47.4, 40.8, 22.1, 20.7, 11.2, 11.0.



N,N-diisopropyl-2-phenylacetamide(4g): (52.6 mg, clourless oil, yield: 60%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [14]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.15 (m, 5H), 4.04 – 3.87 (m, 1H), 3.67 (s, 2H), 3.35 (s, 1H), 1.41 (d, *J* = 6.8 Hz, 7H), 0.99 (d, *J* = 6.6 Hz, 7H).

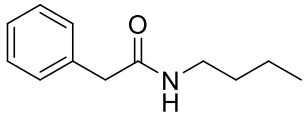
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.8, 135.8, 128.5, 128.4, 126.4, 49.3, 45.7, 43.4, 20.5, 20.4.



2-phenyl-N-propylacetamide(4h): (66.6 mg, white solid, m.p. = 66-69 ℃, yield: 94%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [15]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.5 Hz, 2H), 7.28 – 7.20 (m, 3H), 5.64 (s, 1H), 3.54 (s, 2H), 3.14 (q, *J* = 6.7 Hz, 2H), 1.46 – 1.39 (m, 2H), 0.82 (t, *J* = 7.4 Hz, 3H).

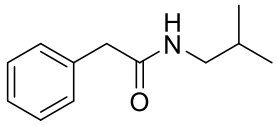
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.9, 135.0, 129.3, 128.8, 127.1, 43.7, 41.2, 22.6, 11.1.



N-butyl-2-phenylacetamide(4i): (70.4 mg, white solid, m.p. = 49-50 ℃, yield: 92%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [16]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.4 Hz, 2H), 7.30 – 7.26 (m, 1H), 7.26 – 7.22 (m, 2H), 5.57 (s, 1H), 3.54 (s, 2H), 3.22 – 3.15 (m, 2H), 1.42 – 1.35 (m, 2H), 1.28 – 1.20 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

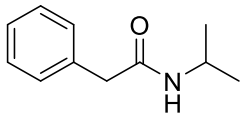
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.9, 135.1, 129.4, 129.0, 127.3, 43.8, 39.4, 31.5, 20.0, 13.7.



N-isobutyl-2-phenylacetamide(4j): (70.4 mg, a brown solid, m.p. = 67-69 ℃, yield: 92%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [17]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 – 7.20 (m, 5H), 5.65 (s, 1H), 3.55 (s, 2H), 3.01 (t, *J* = 6.5 Hz, 2H), 1.79 – 1.55 (m, 1H), 0.80 (d, *J* = 6.7 Hz, 6H).

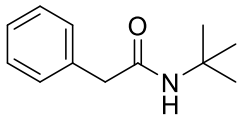
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.8, 135.1, 129.3, 128.9, 127.2, 46.8, 43.8, 28.3, 19.8.



N-isopropyl-2-phenylacetamide(4k): (63.1 mg, white solid, m.p. = 104-106 ℃, yield: 89%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [18]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.5 Hz, 2H), 7.27 – 7.21 (m, 3H), 5.61 (s, 1H), 4.08 – 4.00 (m, 1H), 3.49 (s, 2H), 1.06 (d, *J* = 6.8 Hz, 6H).

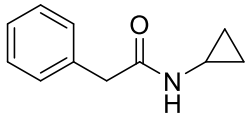
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.0, 135.1, 129.1, 128.7, 127.0, 43.6, 41.3, 22.4.



N-(tert-butyl)-2-phenylacetamide(4l): (60.4 mg, white solid, m.p. = 112-114℃, yield: 79%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [19]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.5 Hz, 2H), 7.29 – 7.26 (m, 1H), 7.25 – 7.22 (m, 2H), 5.21 (s, 1H), 3.48 (s, 2H), 1.28 (s, 9H).

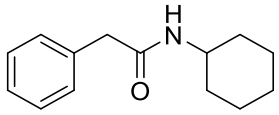
^{13}C NMR (151 MHz, $CDCl_3$) δ 170.2, 135.4, 129.2, 128.9, 127.1, 51.2, 44.8, 28.6.



N-cyclopropyl-2-phenylacetamide(4m): (40.0 mg, white solid, m.p. = 101-103 ℃, yield: 46%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [20]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.4 Hz, 2H), 7.31 – 7.25 (m, 2H), 7.25 – 7.20 (m, 2H), 5.52 (s, 1H), 3.53 (s, 2H), 2.66 (tq, *J* = 7.1, 3.6 Hz, 1H), 0.73 – 0.69 (m, 2H), 0.41 – 0.37 (m, 2H).

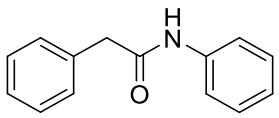
^{13}C NMR (151 MHz, $CDCl_3$) δ 172.4, 134.8, 129.3, 129.0, 127.3, 43.7, 22.7, 6.5.



N-cyclohexyl-2-phenylacetamide(4n): (83.4 mg, white solid, m.p. = 133-134 ℃, yield: 96%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [21]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.3 Hz, 2H), 7.27 – 7.18 (m, 3H), 5.49 (s, 1H), 3.72 (q, *J* = 9.9, 9.3 Hz, 1H), 3.50 (s, 2H), 1.80 (d, *J* = 10.1 Hz, 2H), 1.64 – 1.48 (m, 3H), 1.25 (d, *J* = 10.0 Hz, 2H), 1.11 – 0.91 (m, 3H).

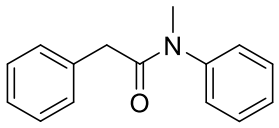
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.9, 135.2, 129.2, 128.8, 127.0, 48.1, 43.8, 32.8, 25.3, 24.6.



N,2-diphenylacetamide(4o): (52.4 mg, white solid, m.p. = 111-114℃, yield: 62%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [22]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 3H), 7.35 – 7.31 (m, 3H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H), 3.71 (s, 2H).

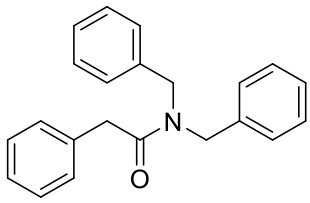
^{13}C NMR (151 MHz, $CDCl_3$) δ 169.3, 137.7, 134.6, 129.5, 129.2, 128.9, 127.6, 124.5, 120.0, 44.8.



N-methyl-N,2-diphenylacetamide(4p): (63.1 mg, light yellow oil, yield: 70%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [23]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 – 7.33 (m, 3H), 7.24 – 7.17 (m, 3H), 7.12 (d, *J* = 7.7 Hz, 2H), 7.06 (d, *J* = 7.5 Hz, 2H), 3.47 (s, 2H), 3.28 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.0, 144.0, 135.5, 129.7, 129.1, 128.3, 127.9, 127.6, 126.6, 40.9, 37.6.



N,N-dibenzyl-2-phenylacetamide(4q): (88.3 mg, white solid, m.p. = 56-58℃, yield: 70%, Petroleum Ether/Ethyl acetate = 5:1-3:1) [24]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 11H), 7.23 (d, *J* = 7.3 Hz, 2H), 7.14 (d, *J* = 7.2 Hz, 2H), 4.66 (s, 2H), 4.47 (s, 2H), 3.83 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.4, 137.2, 136.3, 134.9, 128.8, 128.7, 128.6, 128.4, 128.2, 127.5, 127.3, 126.7, 126.3, 50.1, 48.1, 40.8.

5. References

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6. Copies of NMR Spectra of products

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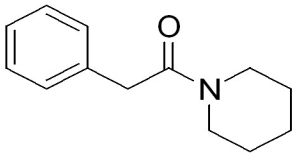


7.31 7.30 7.28 7.28 7.26 CDCl3 7.24 7.23 7.21 7.20 7.17

3.71 3.56 3.54 3.53 3.36 3.34 3.32

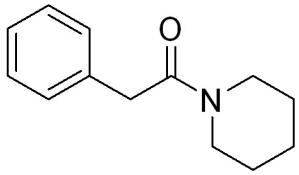
1.59 1.58 1.57 1.57 1.55 1.54 1.52 1.49 1.46 1.45 1.35 1.33 1.31 1.31 1.30

5.00 2.00 1.97 2.01 4.01 2.01

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

f1 (ppm)

1.fid

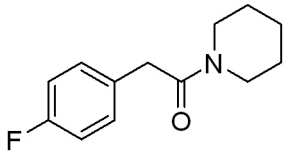


169.28
135.30
128.59
128.50
126.57
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
47.21
42.85
41.08
26.09
25.41
24.33

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

f1 (ppm)

1.1.fid



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3.66
3.56
3.54
3.52
3.37
3.35
3.34
1.61
1.59
1.57
1.55
1.53
1.50
1.49
1.47
1.45
1.39
1.38
1.36
1.34
1.32

2.00
2.00
2.00
2.00
2.01
4.09
2.04

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)

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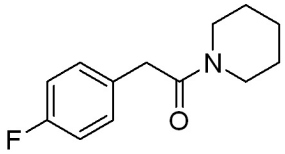
F
O
N

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160.80
131.07
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130.13
130.08
115.38
115.24
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
47.09
42.82
39.96
26.16
25.38
24.30
H
H
190
180
170
160
150
140
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100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

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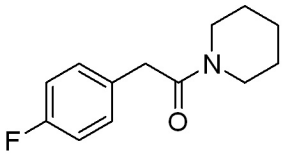

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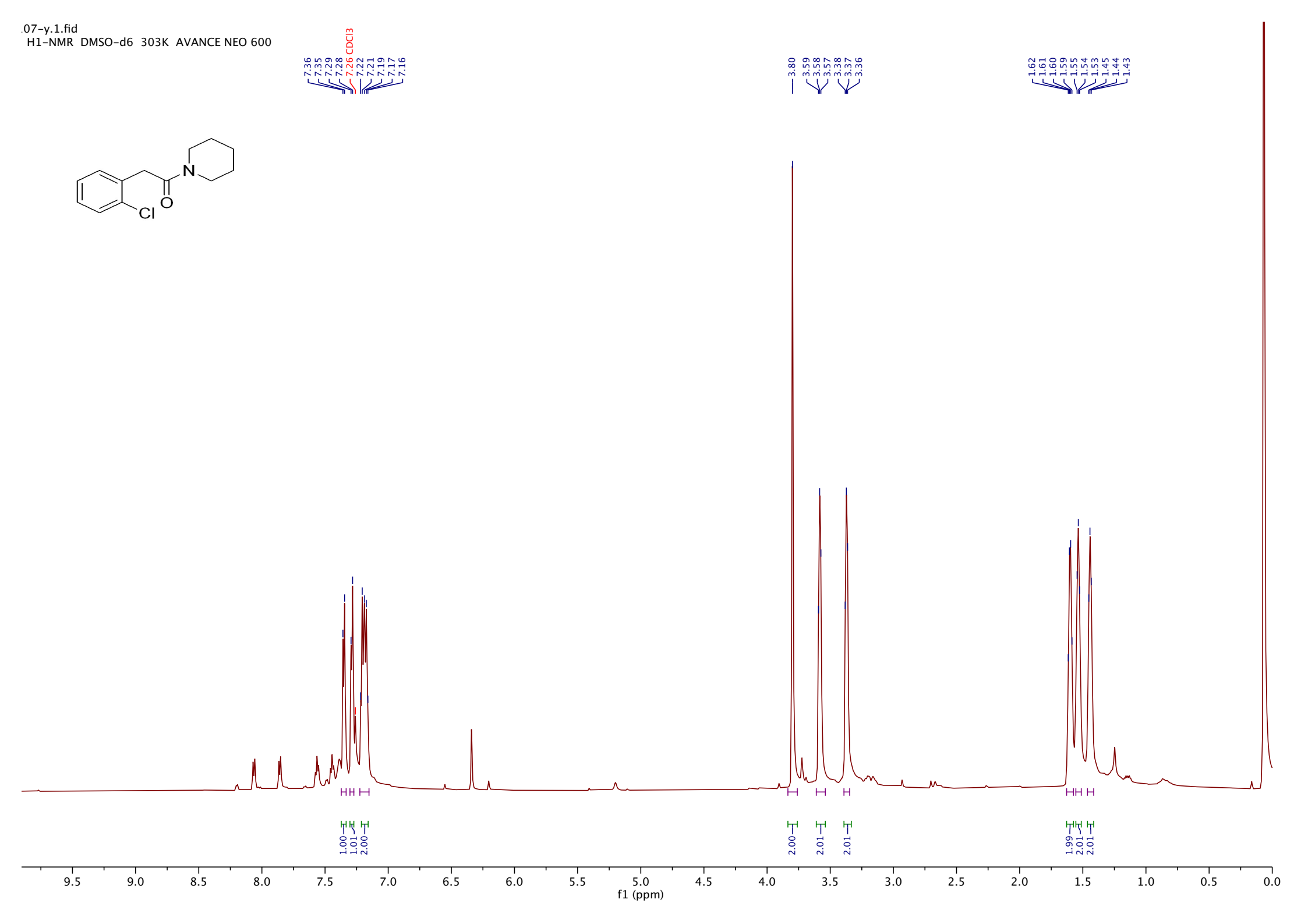
105 -106 -107 -108 -109 -110 -111 -112 -113 -114 -115 -116 -117 -118 -119 -120 -121 -122 -123 -124 -125 -126 -127 -128 -129 -130 -131

f1 (ppm)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7.29
7.28
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7.22
7.21
7.19
7.17
7.16
3.80
3.59
3.58
3.57
3.38
3.37
3.36
1.62
1.61
1.60
1.59
1.55
1.54
1.53
1.45
1.44
1.43
N
O
Cl
1.00
1.01
2.00
2.00
2.01
2.01
1.99
2.01
2.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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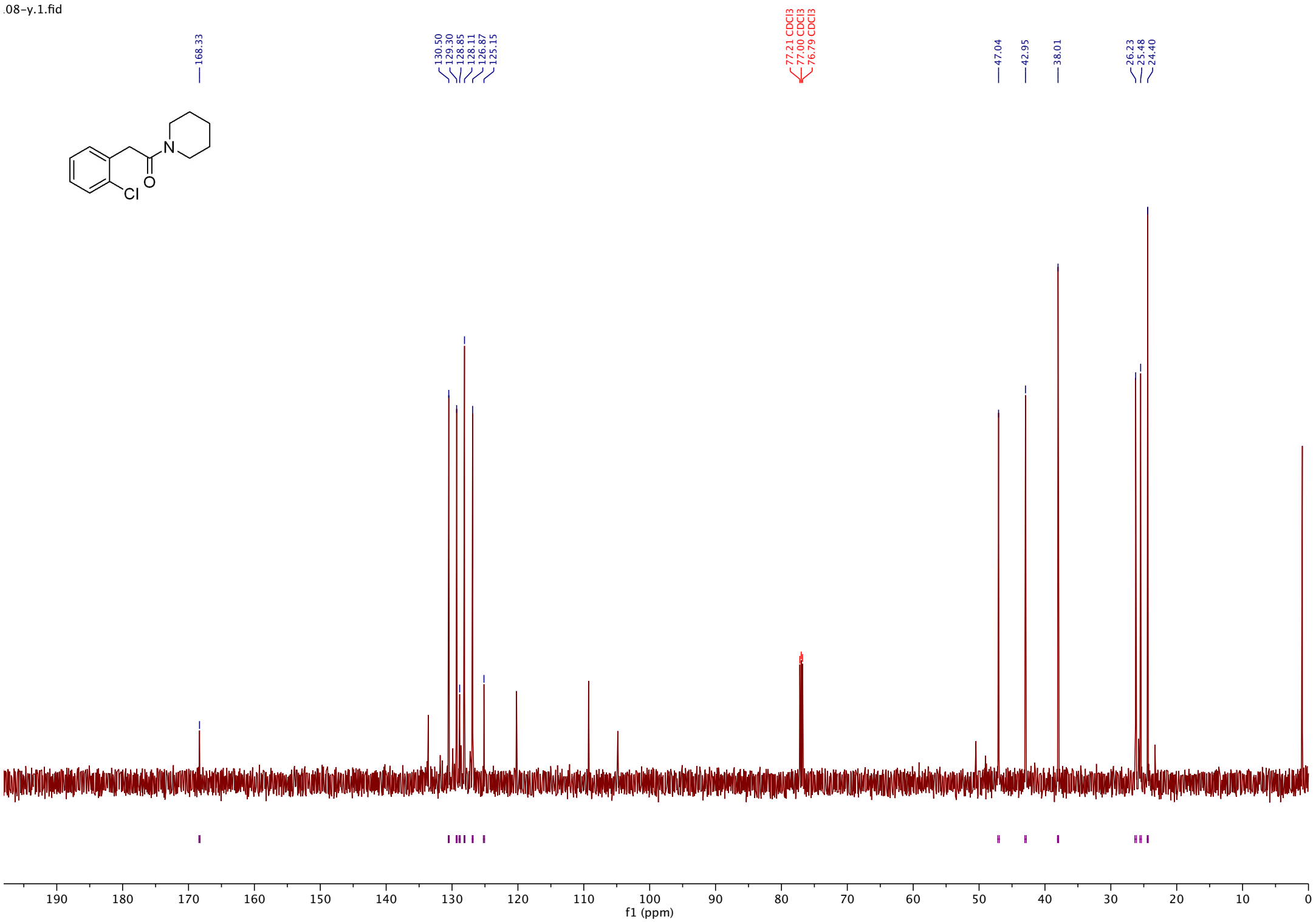
f1 (ppm)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125.15
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
47.04
42.95
38.01
26.23
25.48
24.40
N
O
Cl
190
180
170
160
150
140
130
120
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90
80
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50
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30
20
10
0
f1 (ppm)

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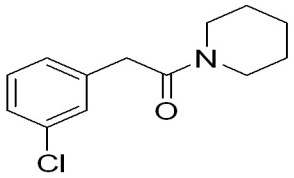
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Cl

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7.23
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7.20
7.20
7.13
7.11
3.68
3.57
3.55
3.54
3.37
3.35
3.34
1.61
1.60
1.60
1.58
1.57
1.54
1.53
1.52
1.51
1.49
1.42
1.41
1.39
1.37
1.35
3.00
1.00
2.00
2.00
2.00
4.00
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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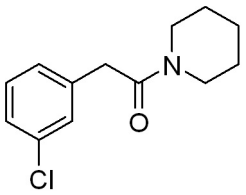
0.0
f1 (ppm)

)23-46.1.fid



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126.84
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77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
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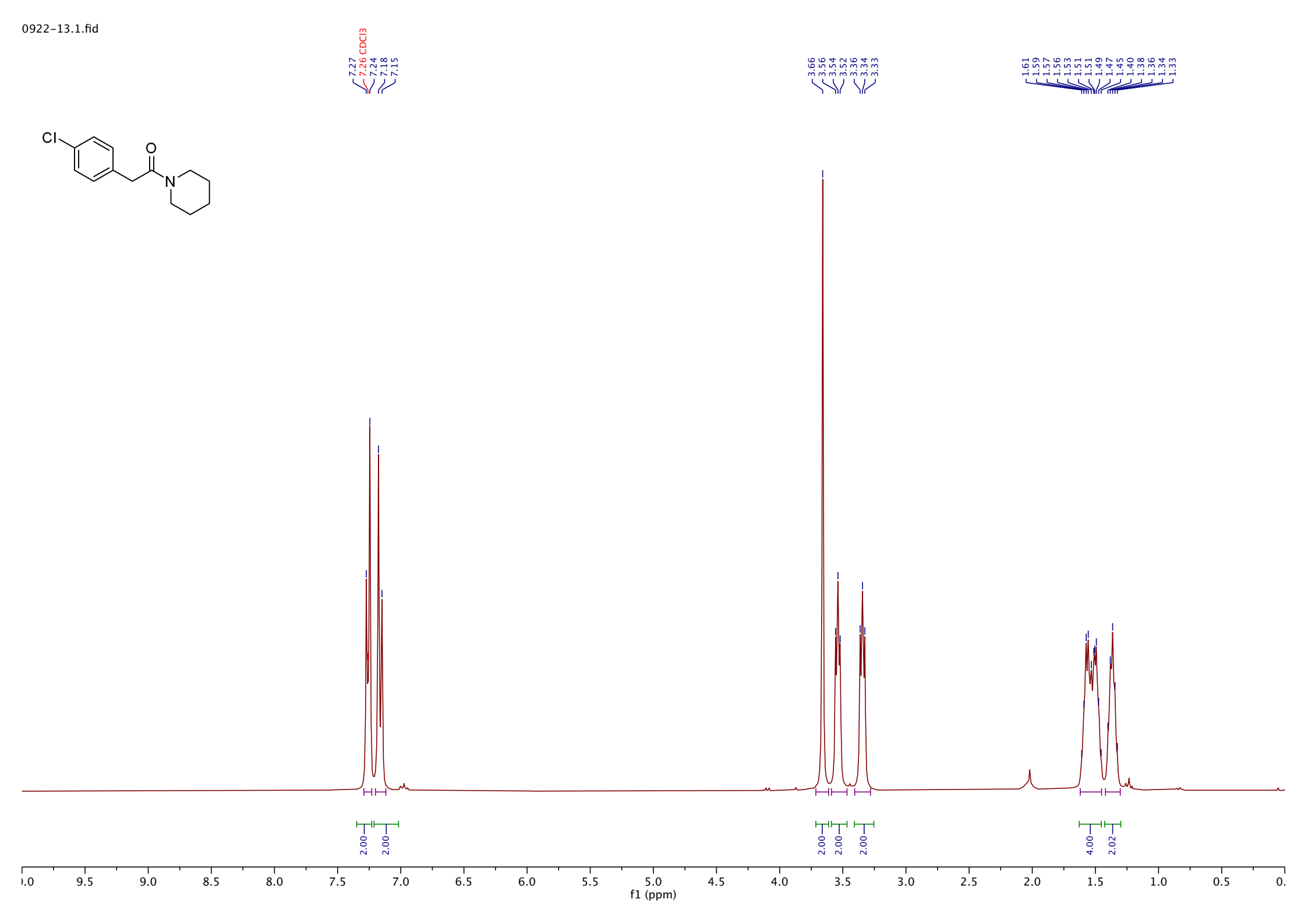
42.89
40.41
26.18
25.38
24.30

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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3.56
3.54
3.52
3.36
3.34
3.33
1.61
1.59
1.57
1.56
1.53
1.51
1.51
1.49
1.47
1.45
1.40
1.38
1.36
1.34
1.33
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2.00
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4.00
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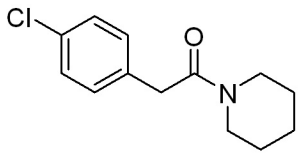
f1 (ppm)

13.1.fid



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76.58 CDCl3
47.10
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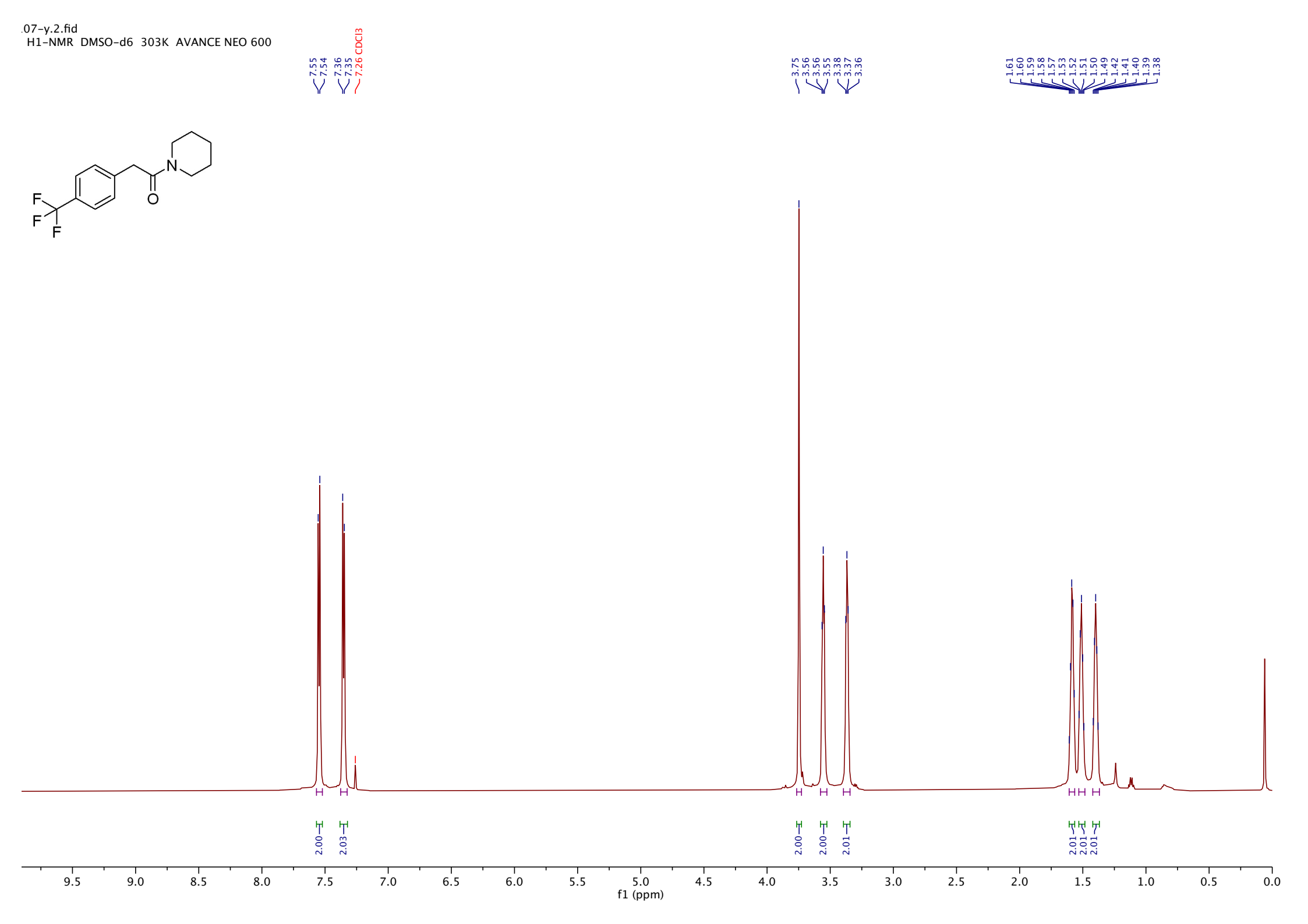
40.15
26.15
25.35
24.27

f1 (ppm)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3.56
3.55
3.38
3.37
3.36
1.61
1.60
1.59
1.58
1.57
1.53
1.52
1.51
1.50
1.49
1.42
1.41
1.40
1.39
1.38
F
F
F
O
N
2.00
2.03
2.00
2.00
2.01
2.01
2.01
2.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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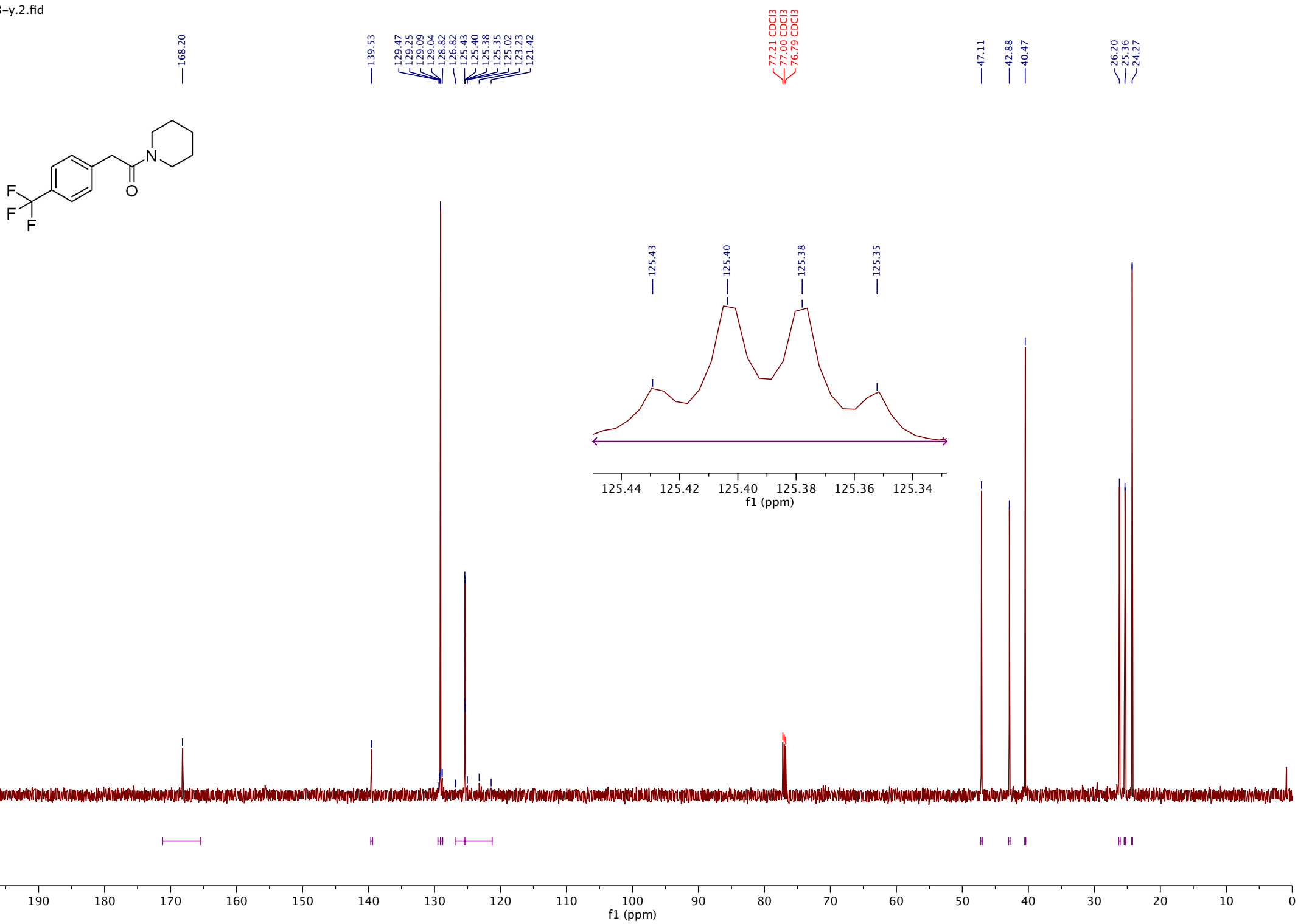
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

.08-y.2.fid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129.04
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125.40
125.38
125.35
125.02
123.23
121.42
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
47.11
42.88
40.47
26.20
25.36
24.27
F
F
F
N
O
125.43
125.40
125.38
125.35
125.44 125.42 125.40 125.38 125.36 125.34
f1 (ppm)
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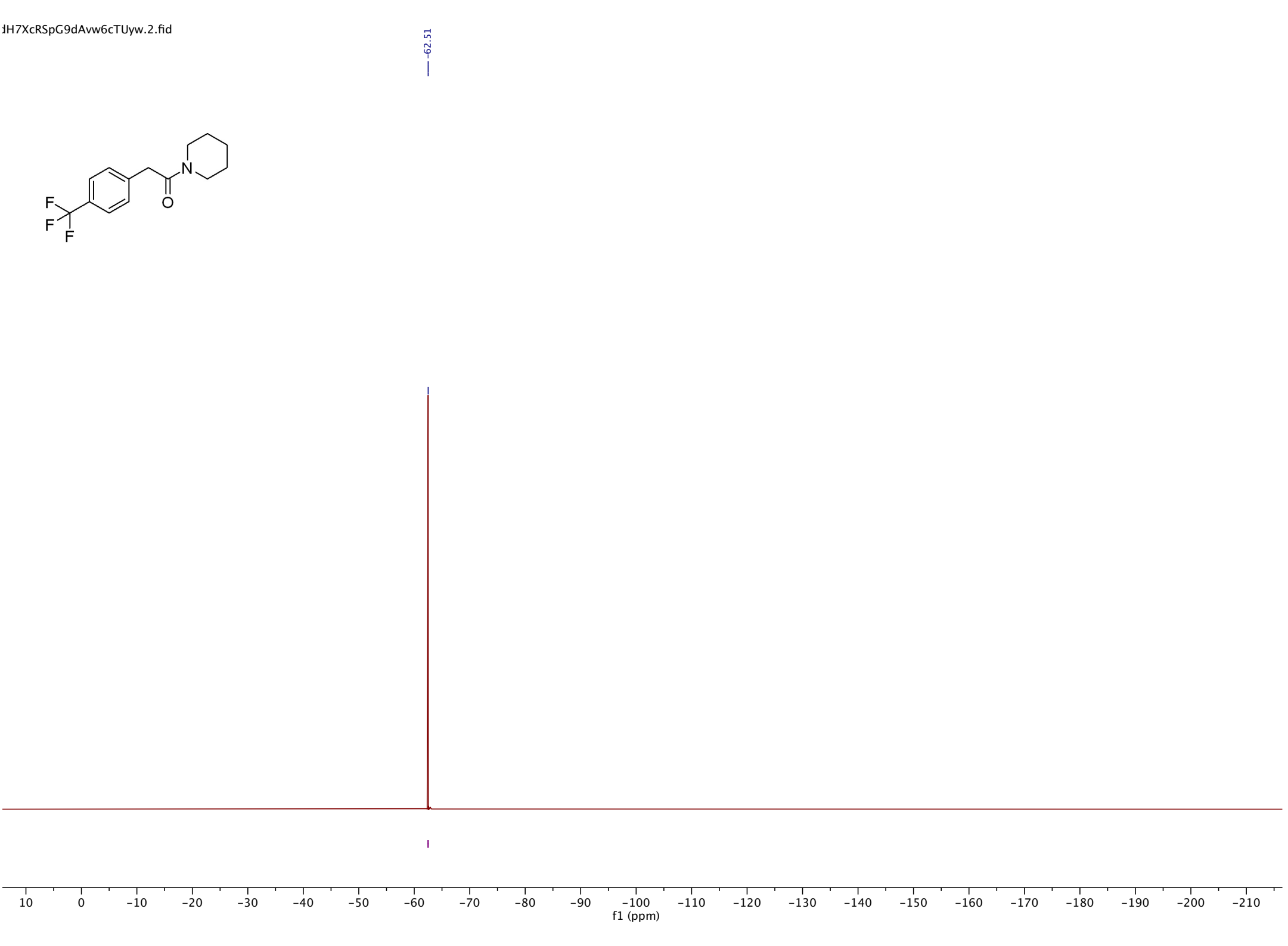
f1 (ppm)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F
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F
N
O
10
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-10
-20
-30
-40
-50
-60
-70
-80
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-130
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-150
-160
-170
-180
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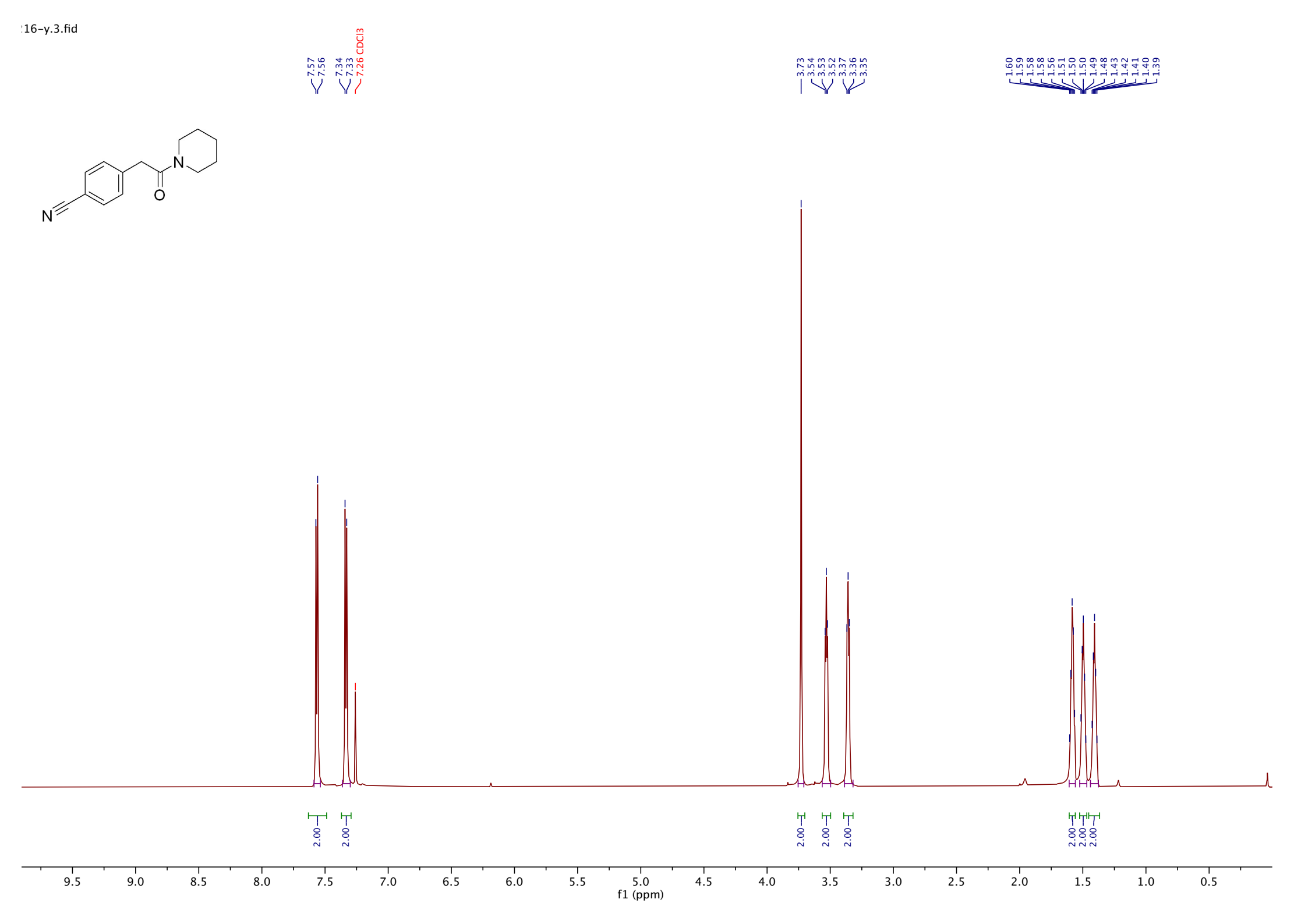
-200
-210
f1 (ppm)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3.36
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1.58
1.58
1.56
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1.40
1.39
N
O
N
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2.00
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2.00
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2.00
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8.0
7.5
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6.0
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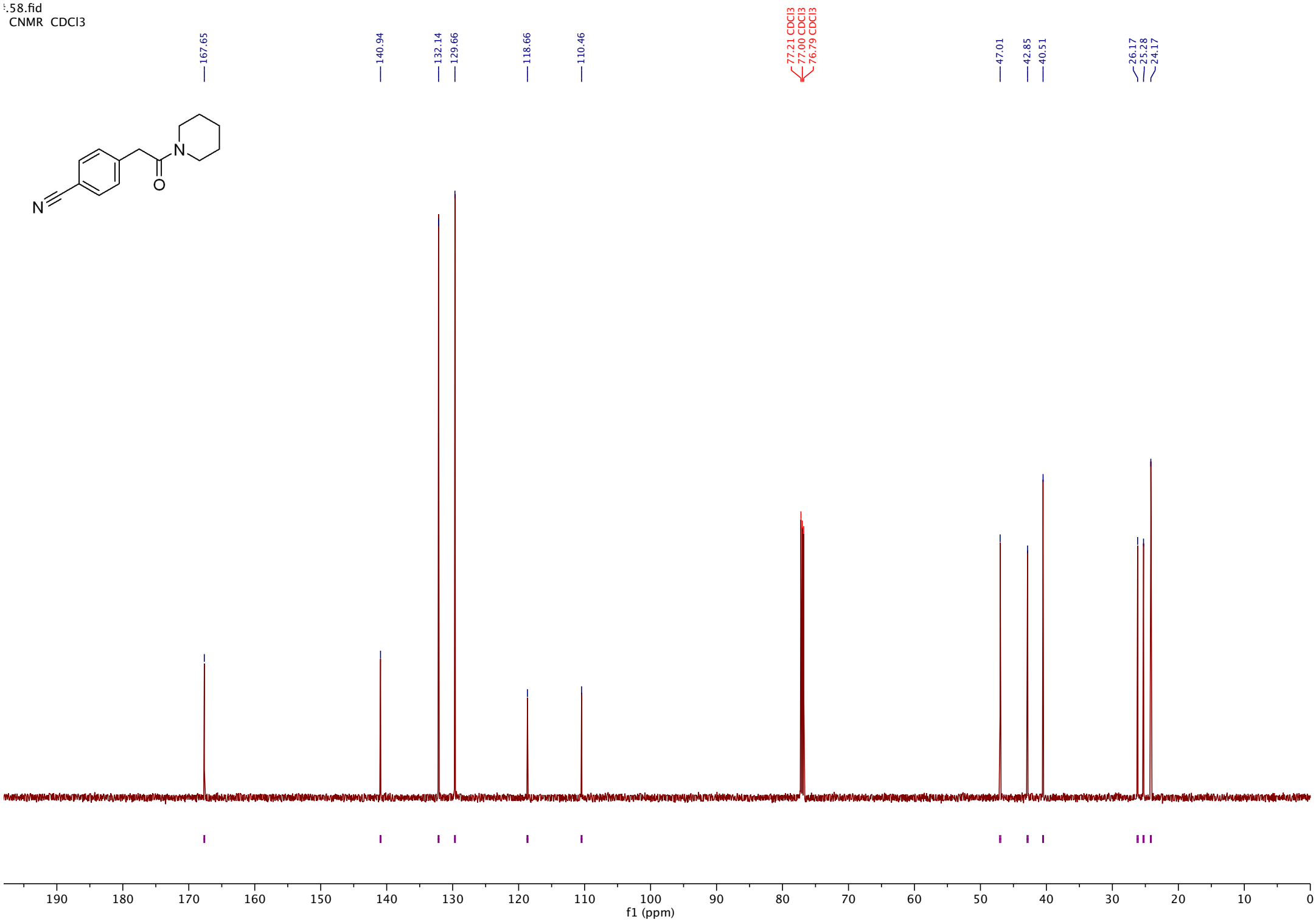
f1 (ppm)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77.00 CDCl3
76.79 CDCl3
47.01
42.85
40.51
26.17
25.28
24.17
N
O
N
190
180
170
160
150
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130
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100
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70
60
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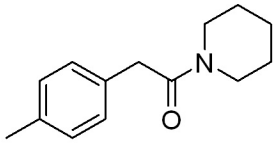
30
20
10
0
f1 (ppm)

3.1.fid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O

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3.34
3.32
2.29
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1.56
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1.53
1.51
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1.48
1.46
1.44
1.36
1.34
1.32
1.30
1.28
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2.00
2.01
2.00
3.01
4.01
2.00
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9.0
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8.0
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7.0
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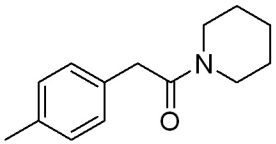
f1 (ppm)

3.1.fid



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128.24
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77.00 CDCl3
76.79 CDCl3
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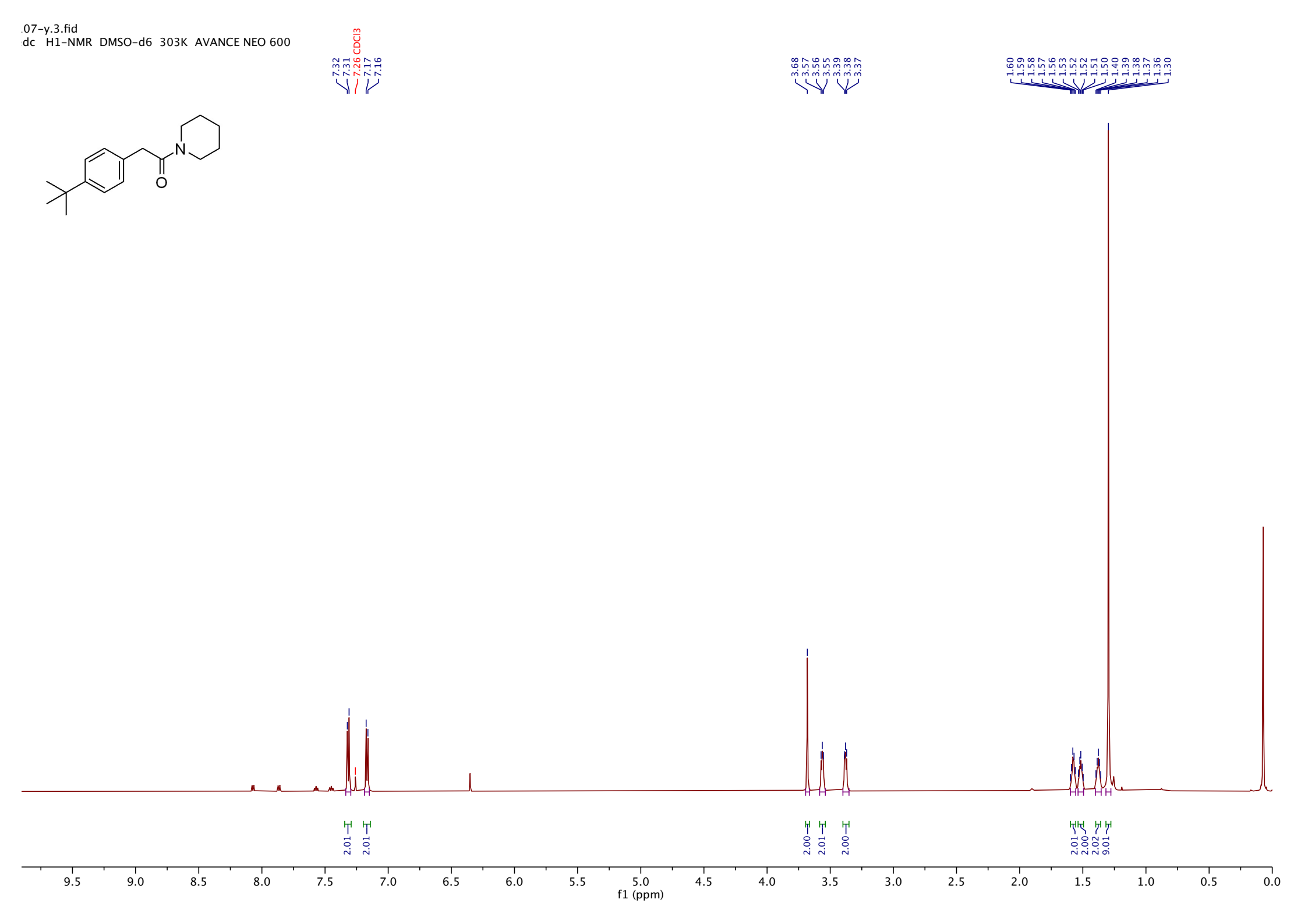
26.01
25.31
24.26
20.83

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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7.26 CDCl3
7.17
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1.59
1.58
1.57
1.56
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1.52
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1.50
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1.39
1.38
1.37
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1.30
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O
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2.01
2.00
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2.00
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2.00
2.02
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f1 (ppm)

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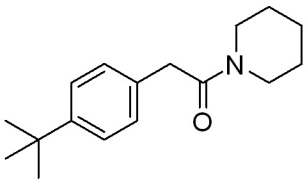
N
O

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77.00 CDCl3
76.79 CDCl3
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40.43
34.33
31.29
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24.41
190
180
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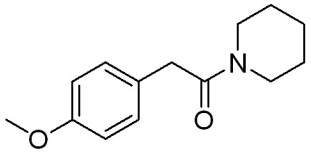
20
10
0
f1 (ppm)

2.1.fid



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6.81
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3.32
1.59
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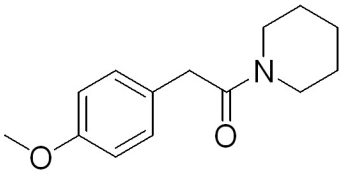
2.01
3.00
2.00
2.00
2.00
4.02
2.01

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
f1 (ppm)

2.1.fid

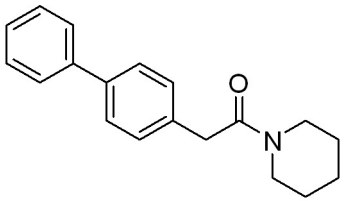


169.41
158.21
129.46
127.35
113.94
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
55.11
47.08
42.72
40.08
26.09
25.37
24.31

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

f1 (ppm)

)22-14.1.fid



7.60 7.60 7.57 7.57 7.54 7.46 7.43 7.41 7.36 7.35 7.34 7.33 7.31 7.26 CDCl3

3.77 3.61 3.59 3.58 3.43 3.41 3.39

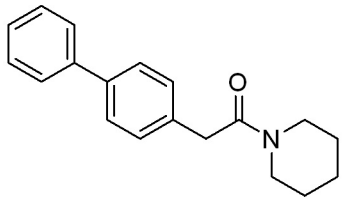
1.63 1.62 1.61 1.59 1.58 1.56 1.54 1.51 1.50 1.43 1.42 1.40 1.38 1.36

4.00 2.00 3.00 2.00 2.01 2.00 4.01 2.00

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)

14.1.fid



169.13

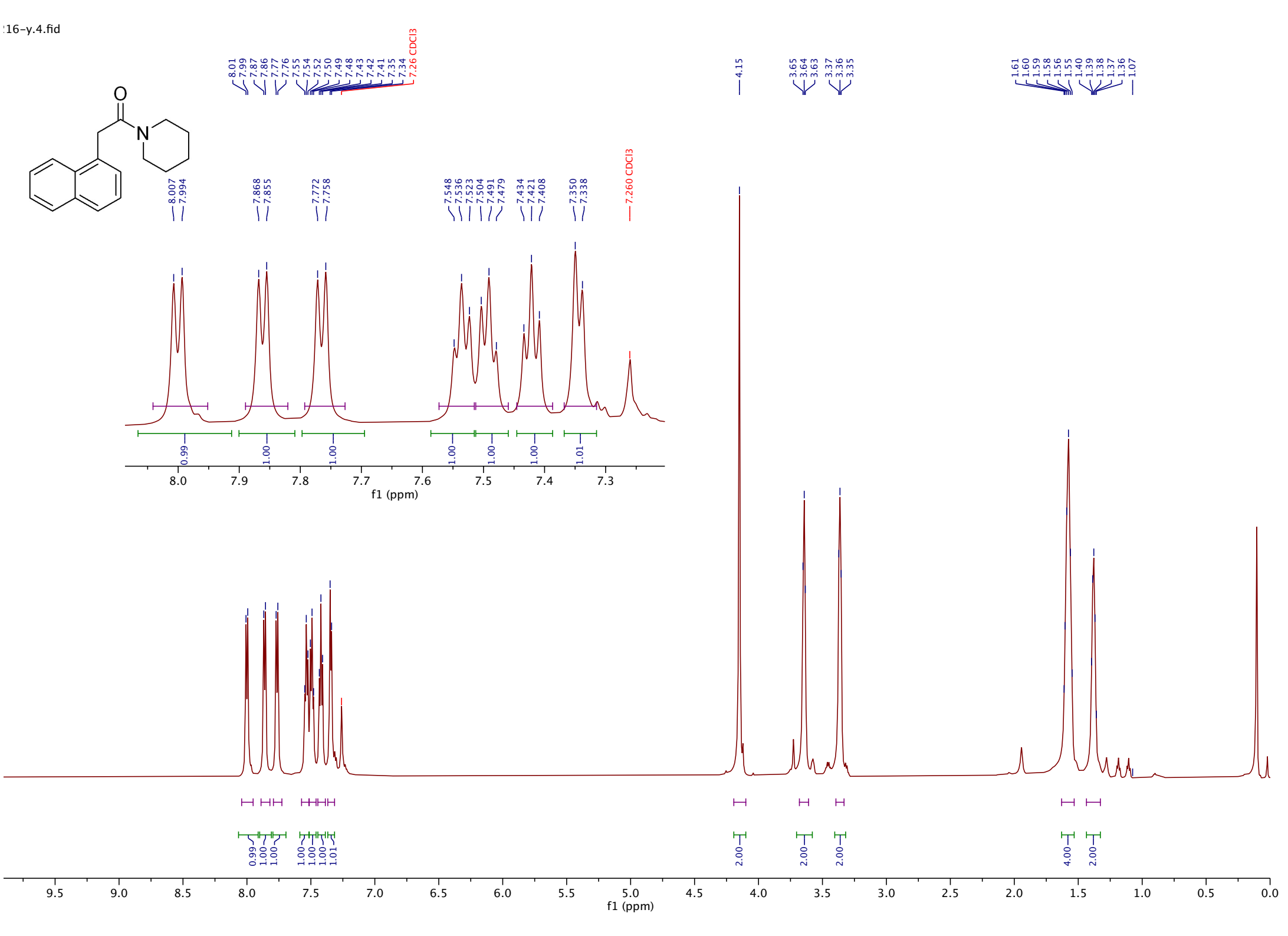
140.69
139.45
134.40
128.98
128.66
127.25
127.12
126.91

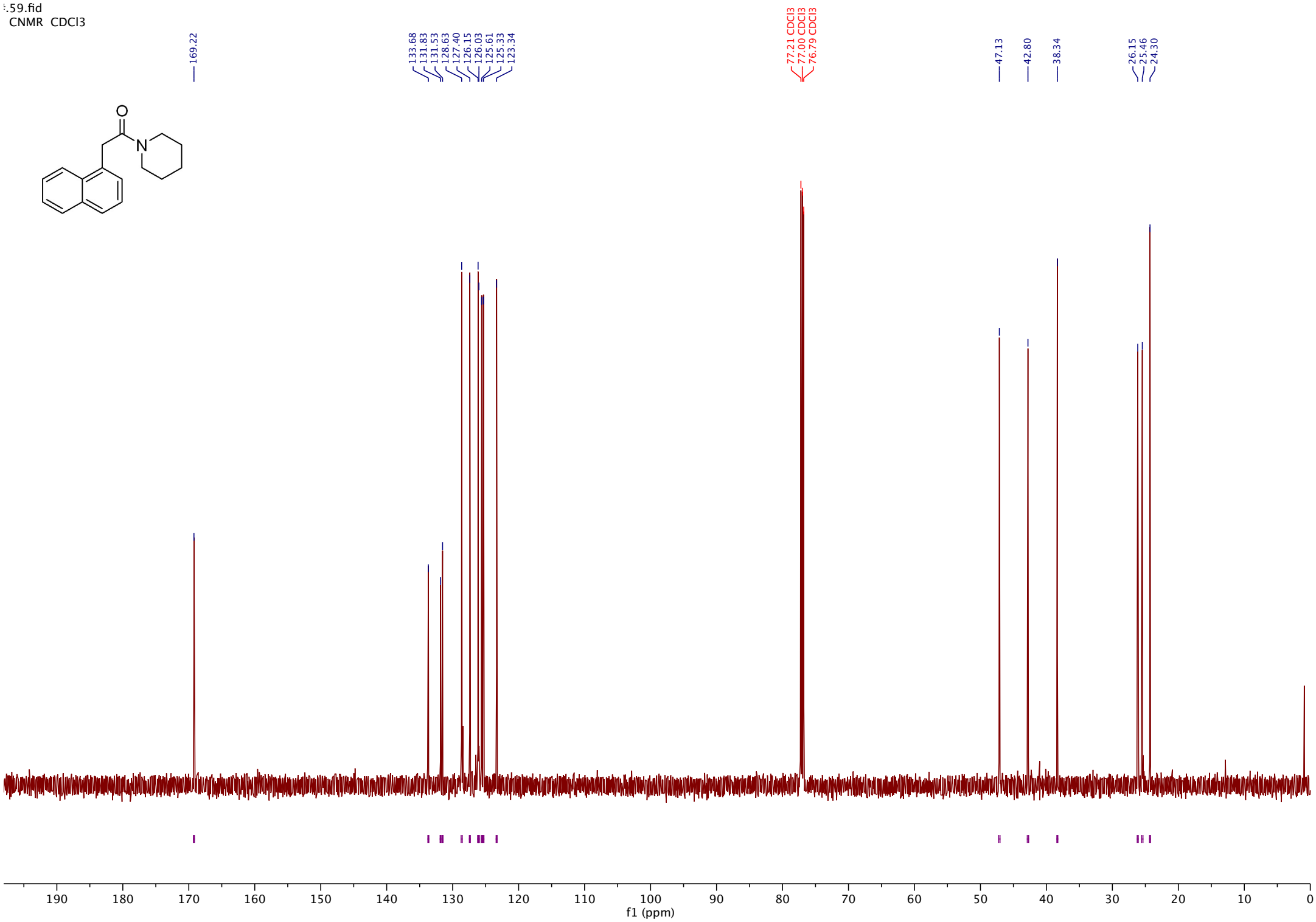
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3

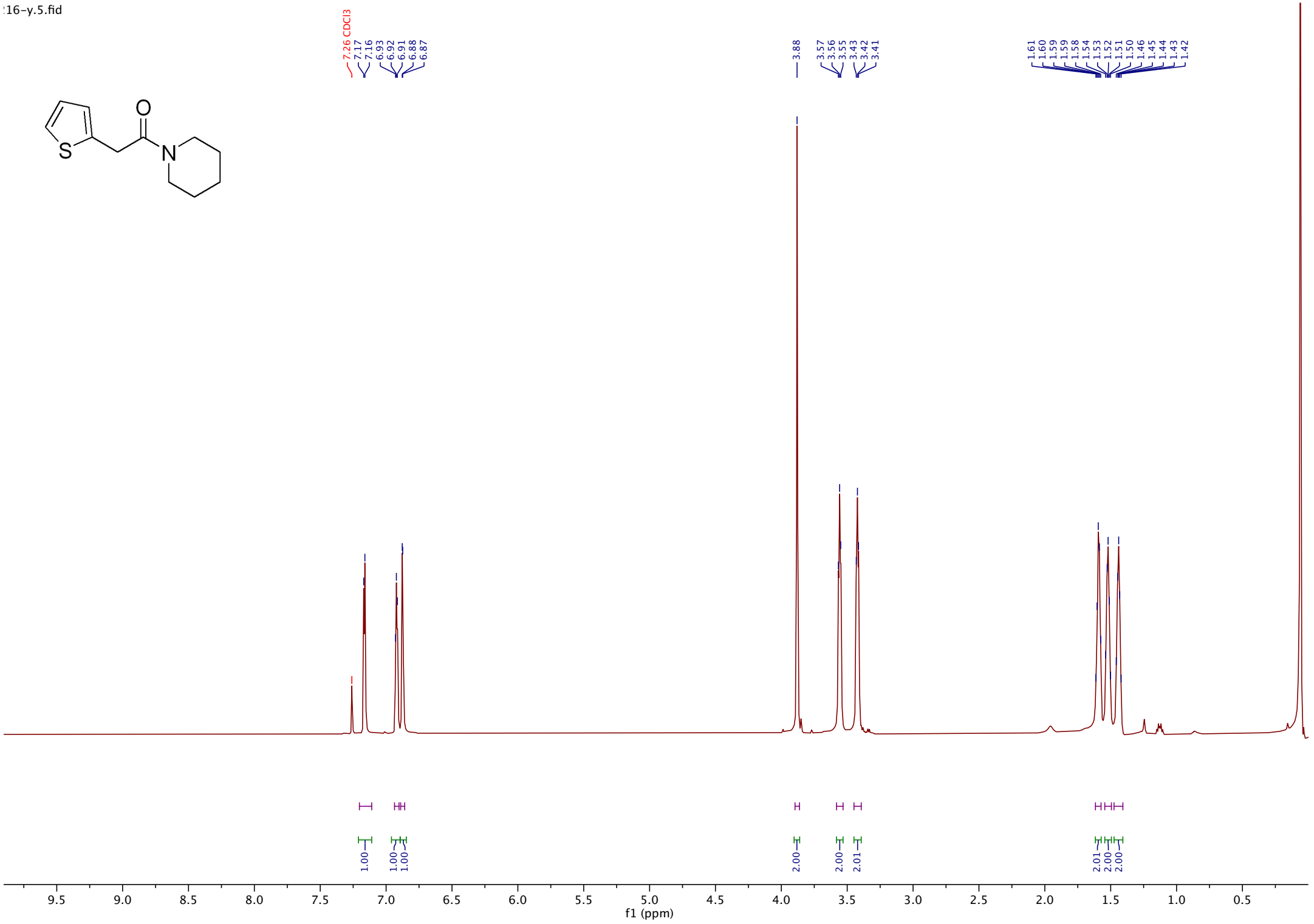
47.19
42.84
40.62

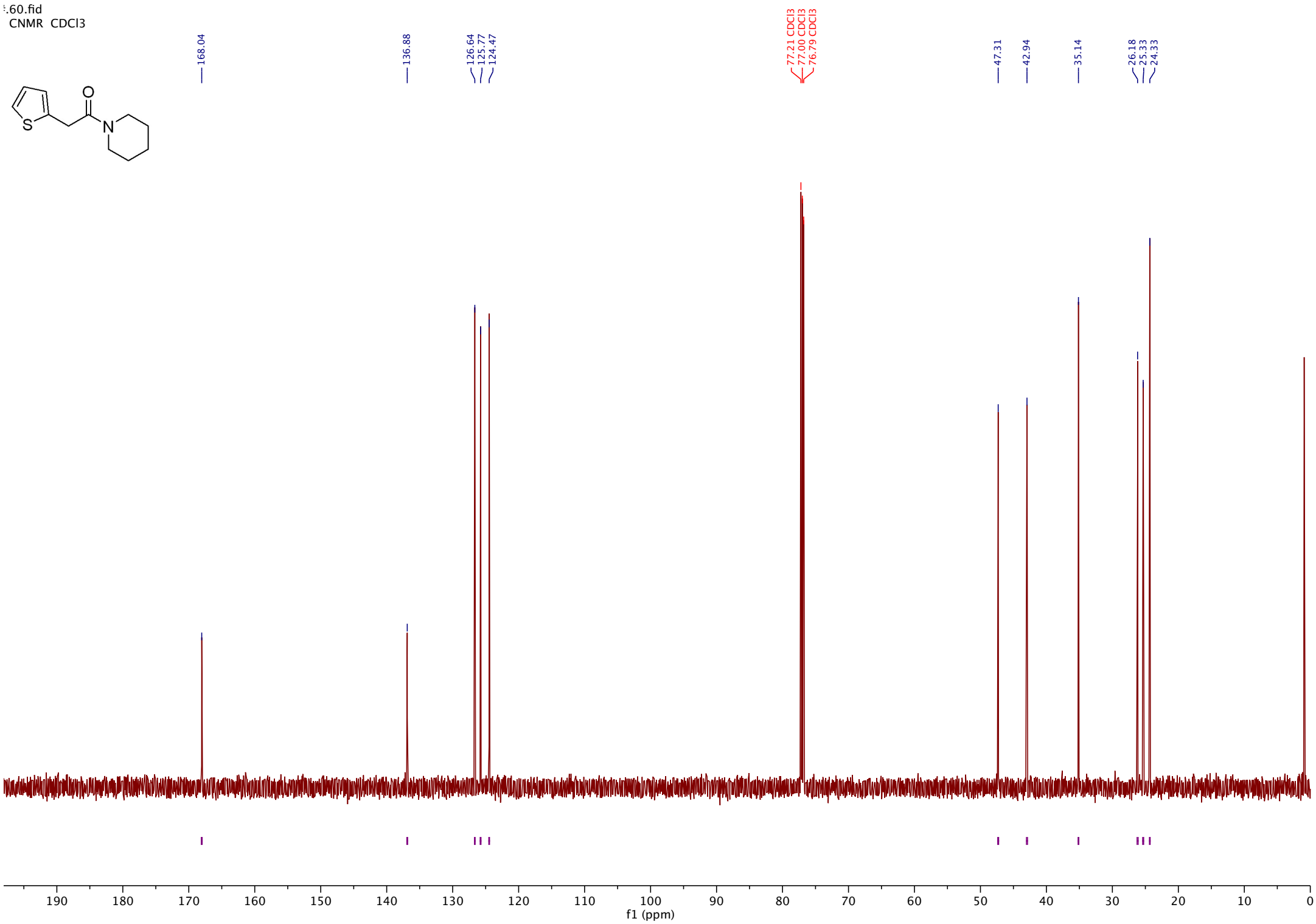
26.16
25.42
24.34

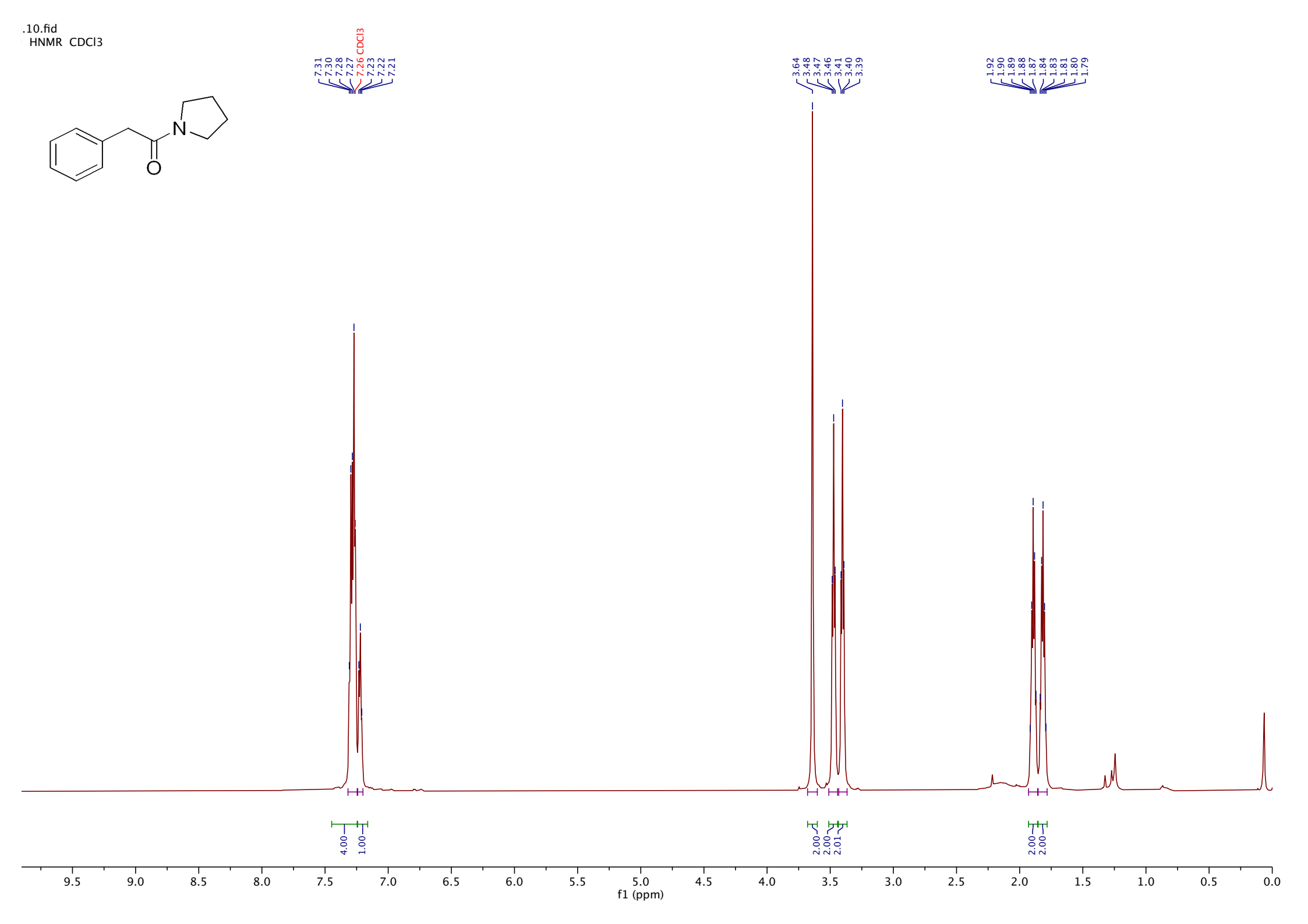
f1 (ppm)


16-y.4.fid
8.01
7.99
7.87
7.86
7.77
7.76
7.55
7.54
7.52
7.50
7.49
7.48
7.43
7.42
7.41
7.35
7.34
7.26 CDCl3
4.15
3.65
3.64
3.63
3.37
3.36
3.35
1.61
1.60
1.59
1.58
1.56
1.55
1.40
1.39
1.38
1.37
1.36
1.07
O
N
8.007
7.994
7.868
7.855
7.772
7.758
7.548
7.536
7.523
7.504
7.491
7.479
7.434
7.421
7.408
7.350
7.338
7.260 CDCl3
0.99
1.00
1.00
1.00
1.00
1.00
1.01
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
f1 (ppm)
0.99
1.00
1.00
1.00
1.00
1.00
1.01
2.00
2.00
2.00
4.00
2.00
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)

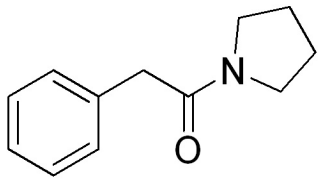

59.fid
CNMR CDCl3
169.22
133.68
131.83
131.53
128.63
127.40
126.15
126.03
125.61
125.33
123.34
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
47.13
42.80
38.34
26.15
25.46
24.30
O
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

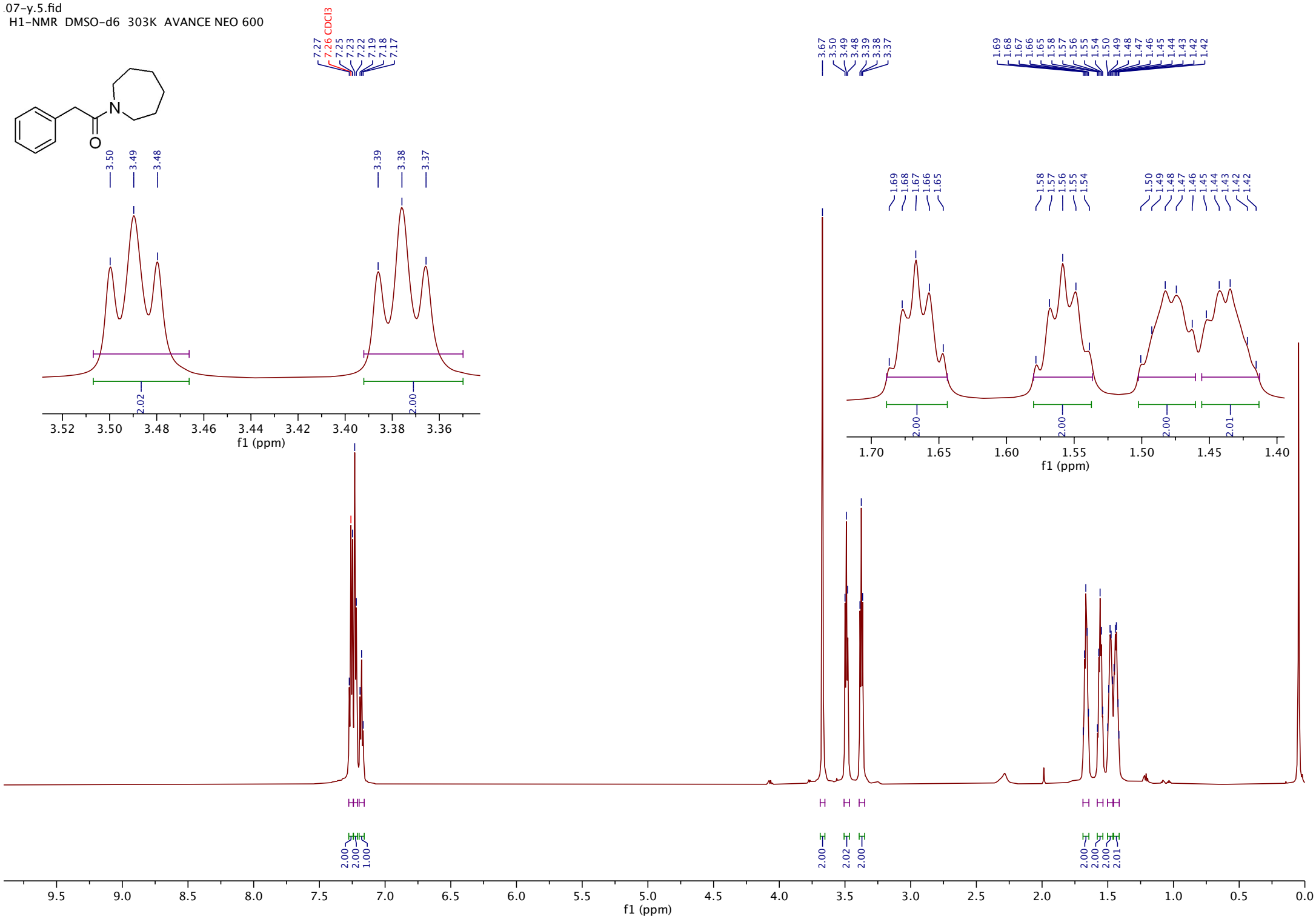

16-y.5.fid
7.26 CDCl3
7.17
7.16
6.93
6.92
6.91
6.88
6.87
3.88
3.57
3.56
3.55
3.43
3.42
3.41
1.61
1.60
1.59
1.59
1.58
1.54
1.53
1.52
1.51
1.50
1.46
1.45
1.44
1.43
1.42
O
S
N
1.00
1.00
1.00
2.00
2.00
2.01
2.01
2.00
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


.60.fid
CNMR CDCl3
168.04
136.88
126.64
125.77
124.47
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
47.31
42.94
35.14
26.18
25.33
24.33
O
S
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

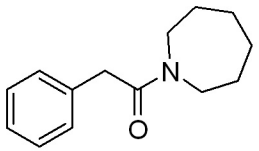

.10.fid
HNMR CDCl3
7.31
7.30
7.28
7.27
7.26 CDCl3
7.23
7.22
7.21
3.64
3.48
3.47
3.46
3.41
3.40
3.39
1.92
1.90
1.89
1.88
1.87
1.84
1.83
1.81
1.80
1.79
N
O
4.00
1.00
2.00
2.00
2.01
2.00
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

.33.fid
CNMR CDCl3



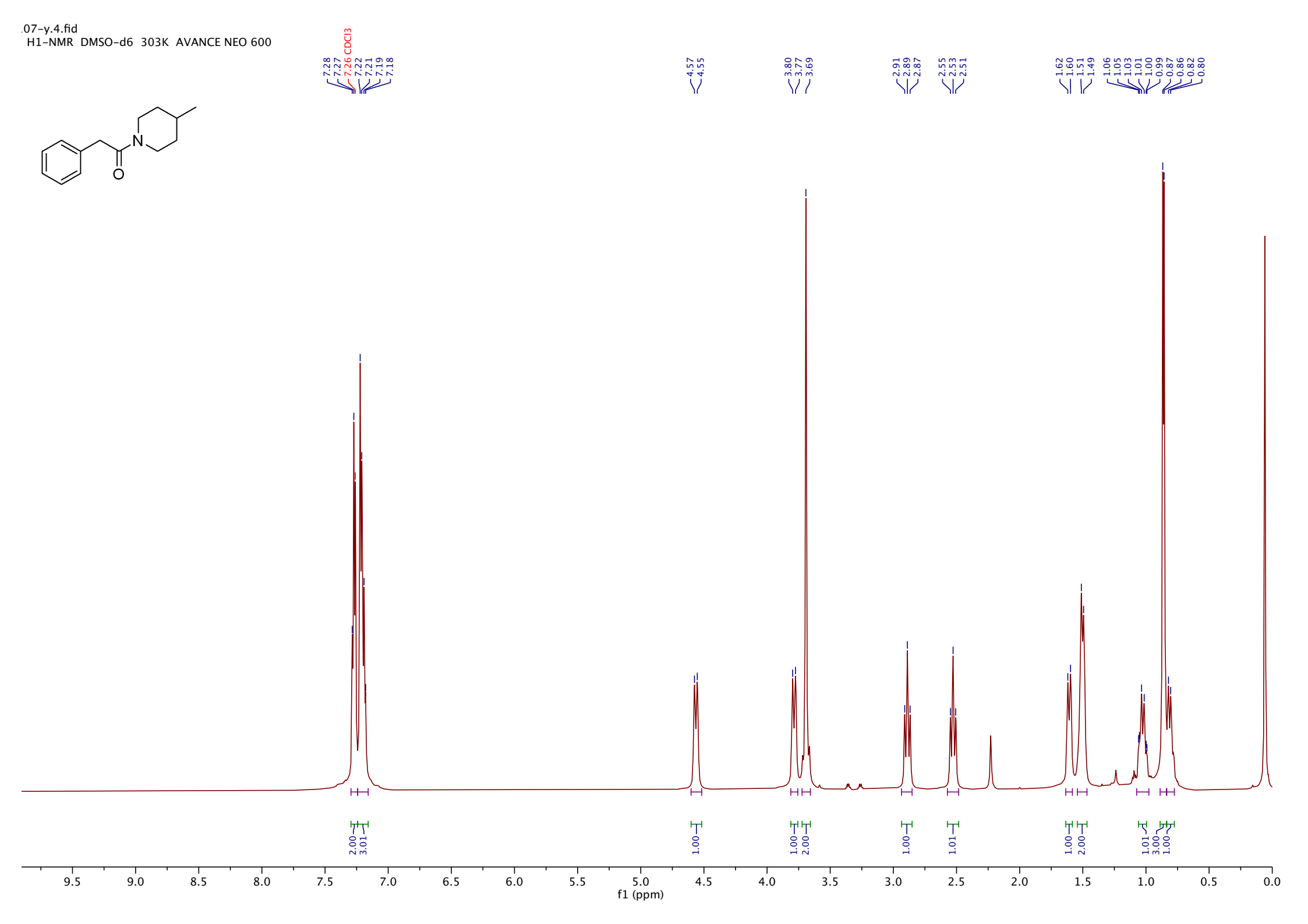

.07-y.5.fid
H1-NMR DMSO-d6 303K AVANCE NEO 600
7.27
7.26 CDCl3
7.25
7.23
7.22
7.19
7.18
7.17
3.67
3.50
3.49
3.48
3.39
3.38
3.37
1.69
1.68
1.67
1.66
1.65
1.58
1.57
1.56
1.55
1.54
1.50
1.49
1.48
1.47
1.46
1.45
1.44
1.43
1.42
1.42
N
O
2.02
2.00
f1 (ppm)
2.00
2.00
2.00
2.01
f1 (ppm)
2.00
2.00
1.00
2.00
2.02
2.00
2.00
2.00
2.00
2.01
f1 (ppm)

.08-y.5.fid

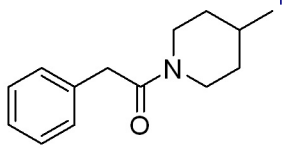


170.35
135.24
128.53
128.35
126.40
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
48.09
45.84
40.86
28.90
27.23
26.98
26.36

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


.07-y.4.fid
H1-NMR DMSO-d6 303K AVANCE NEO 600
7.28
7.27
7.26 CDCl3
7.22
7.21
7.19
7.18
4.57
4.55
3.80
3.77
3.69
2.91
2.89
2.87
2.55
2.53
2.51
1.62
1.60
1.51
1.49
1.06
1.05
1.03
1.01
1.00
0.99
0.87
0.86
0.82
0.80
N
O
2.00
3.01
1.00
1.00
2.00
1.00
1.01
1.00
2.00
1.01
3.00
1.00
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)

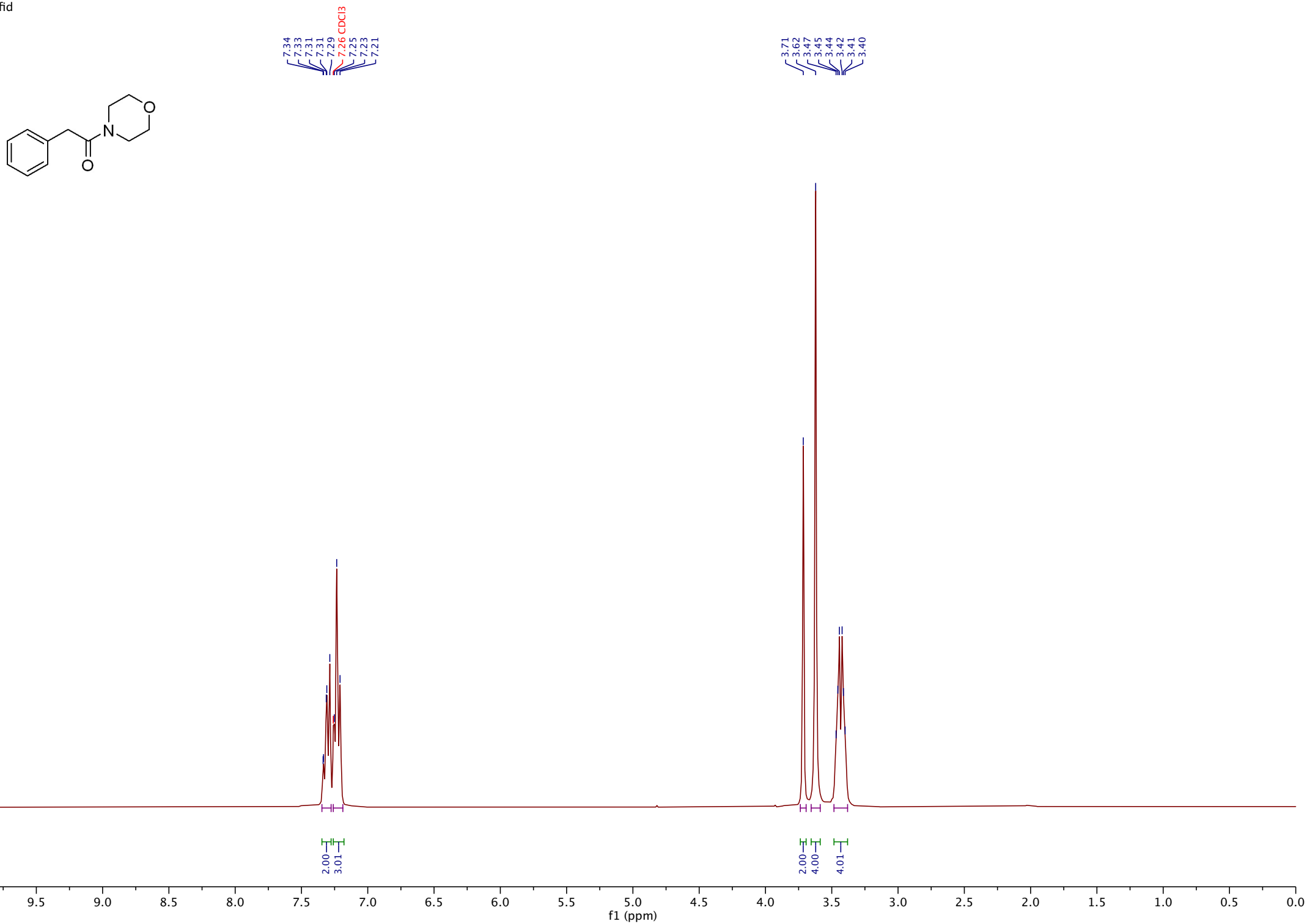
.08-y.4.fid



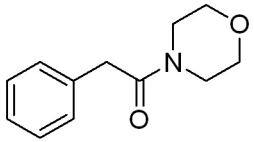
169.02
135.23
128.41
128.38
126.41
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
46.29
42.01
40.95
34.15
33.50
30.74
21.44

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

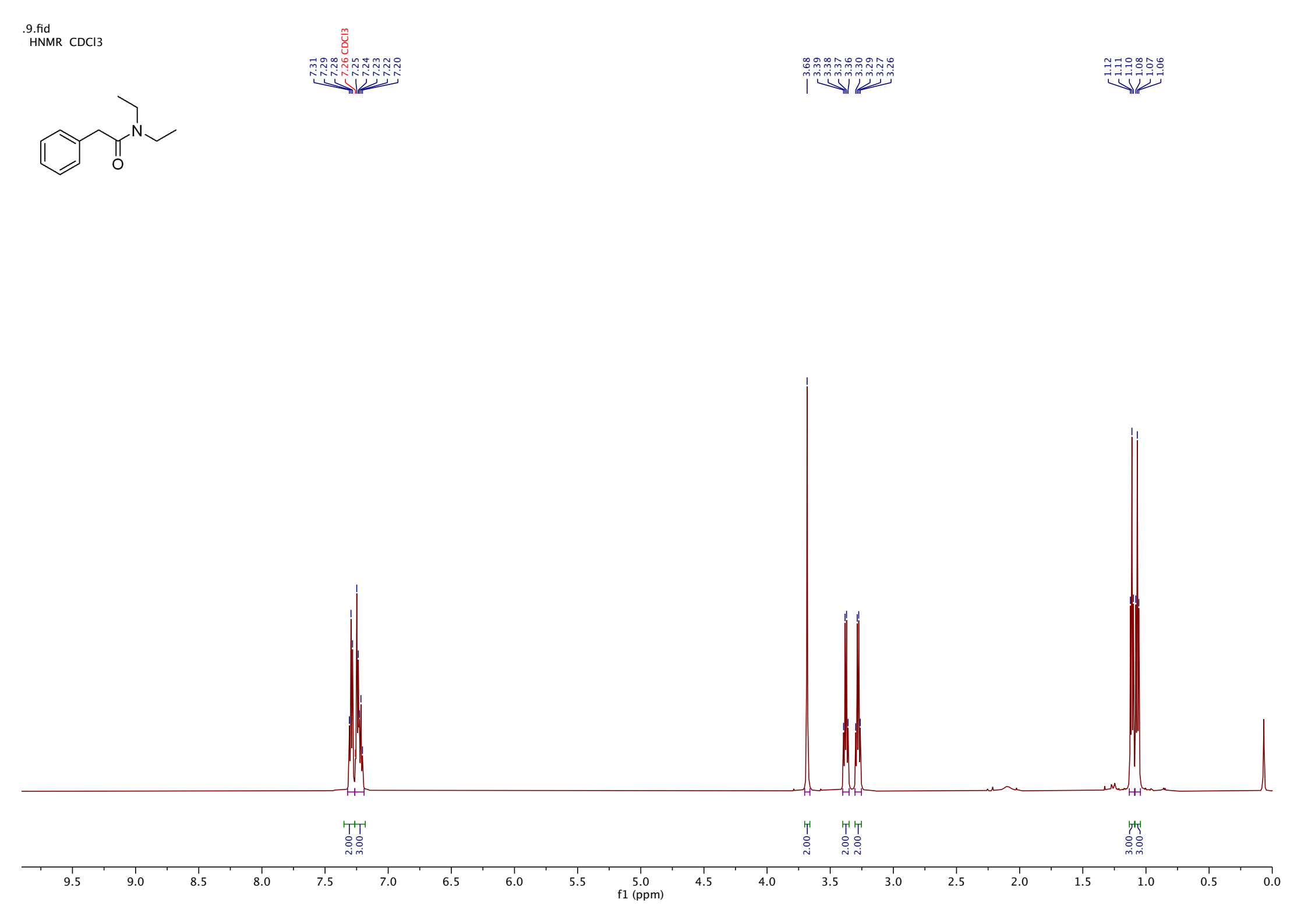
.1.fid


7.34
7.33
7.31
7.31
7.29
7.26 CDCl3
7.25
7.23
7.21
3.71
3.62
3.47
3.45
3.44
3.42
3.41
3.40
O
N
O
2.00
3.01
2.00
4.00
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
f1 (ppm)

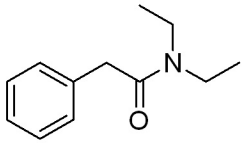
.1.fid



169.49
134.73
128.68
128.43
126.78
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
66.67
66.34
46.42
42.04
40.71
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


.9.fid
HNMR CDCl3
7.31
7.29
7.28
7.26 CDCl3
7.25
7.24
7.23
7.22
7.20
3.68
3.39
3.38
3.37
3.36
3.30
3.29
3.27
3.26
1.12
1.11
1.10
1.08
1.07
1.06
N
O
2.00
3.00
2.00
2.00
2.00
3.00
3.00
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)

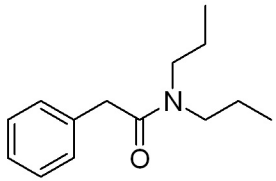
.32.fid
CNMR CDCl3

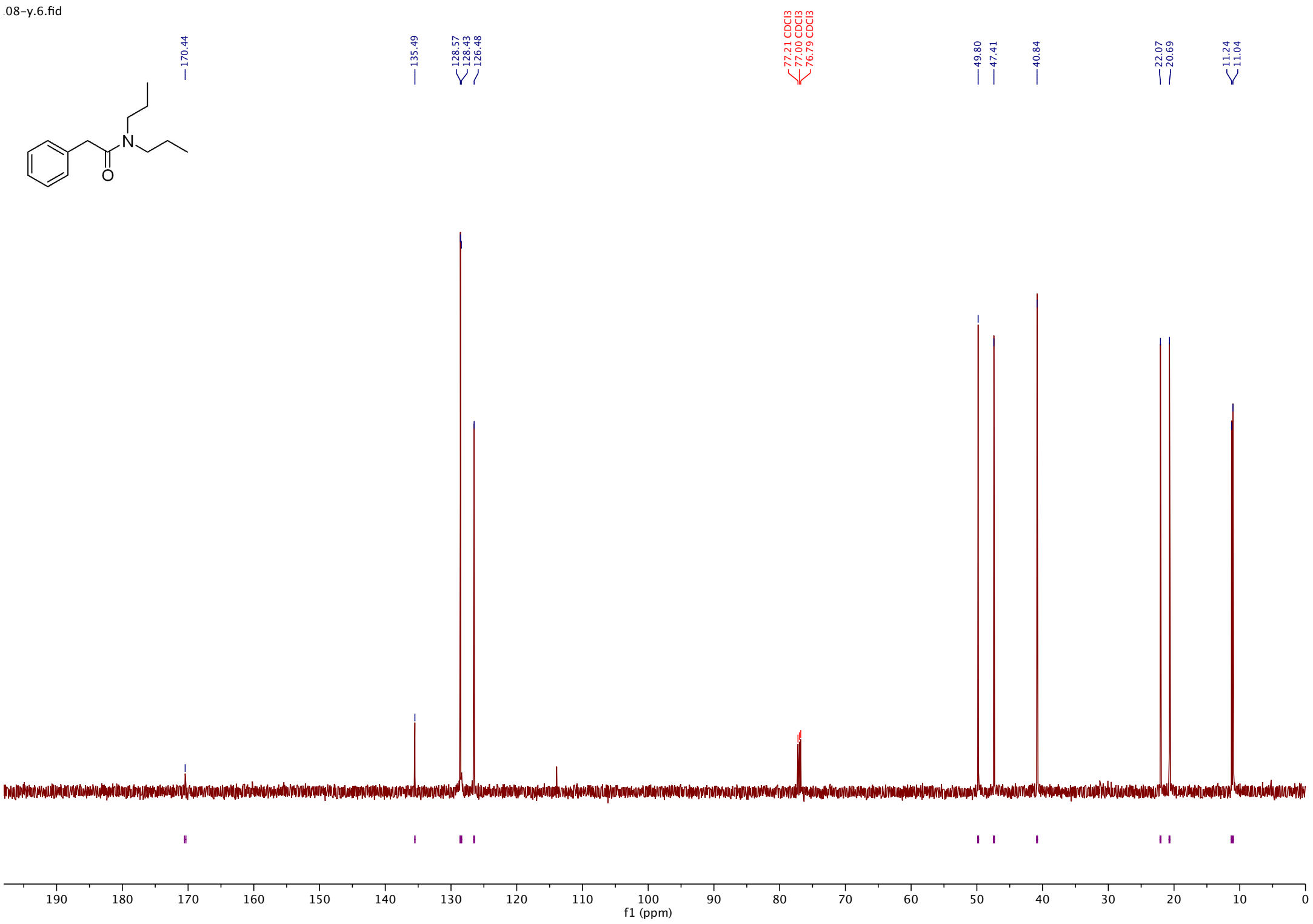


170.01
135.40
128.54
128.49
126.52
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
42.24
40.79
40.02
14.07
12.81

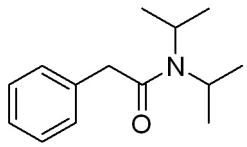
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

.07-y.6.fid
H1-NMR DMSO-d6 303K AVANCE NEO 600




.08-y.6.fid
170.44
135.49
128.57
128.43
126.48
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
49.80
47.41
40.84
22.07
20.69
11.24
11.04
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

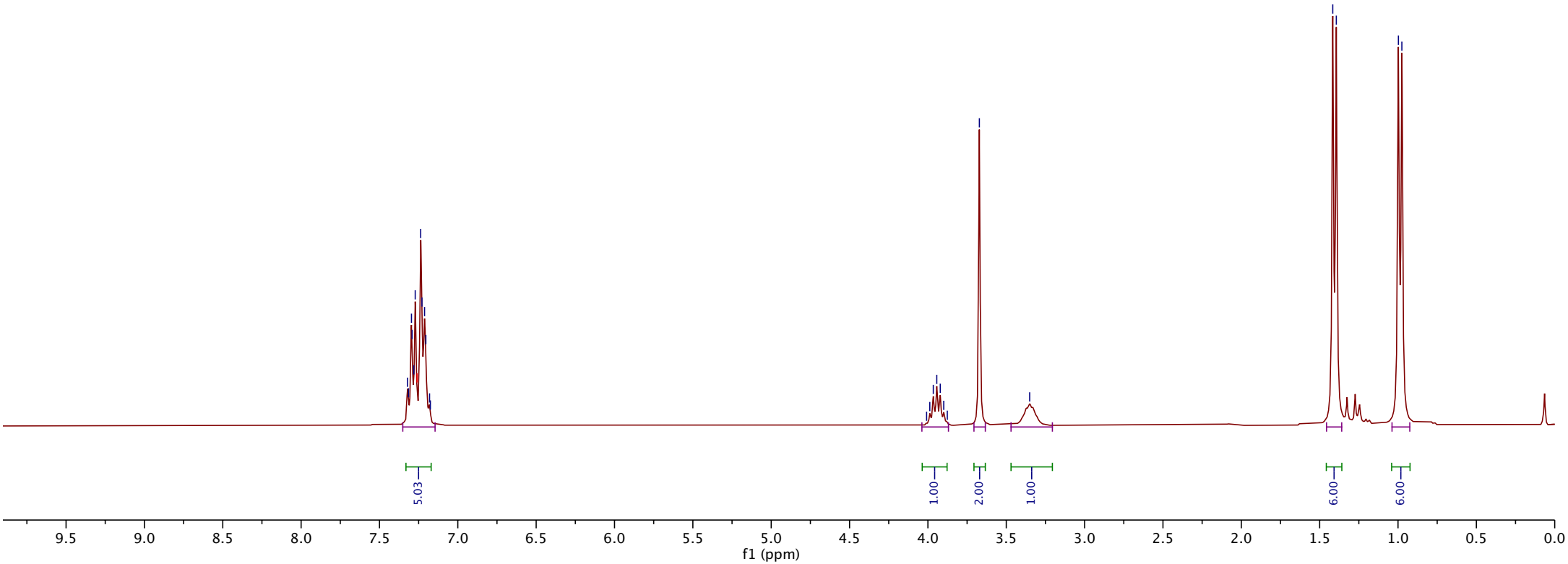
.1.fid



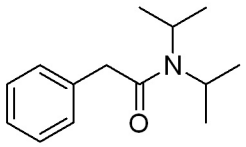
7.32 7.31 7.30 7.29 7.28 7.27 7.26 CDCl3 7.24 7.23 7.21 7.20 7.18 7.18

4.01 3.99 3.97 3.94 3.92 3.90 3.88 3.67 3.35

1.42 1.39 1.00 0.98



.1.fid

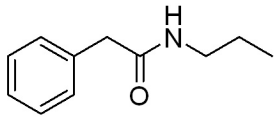


169.78
135.80
128.51
128.37
126.42
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
49.31
45.69
43.41
20.50
20.43

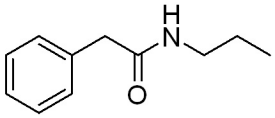
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

.8.fid
HNMR CDCl3

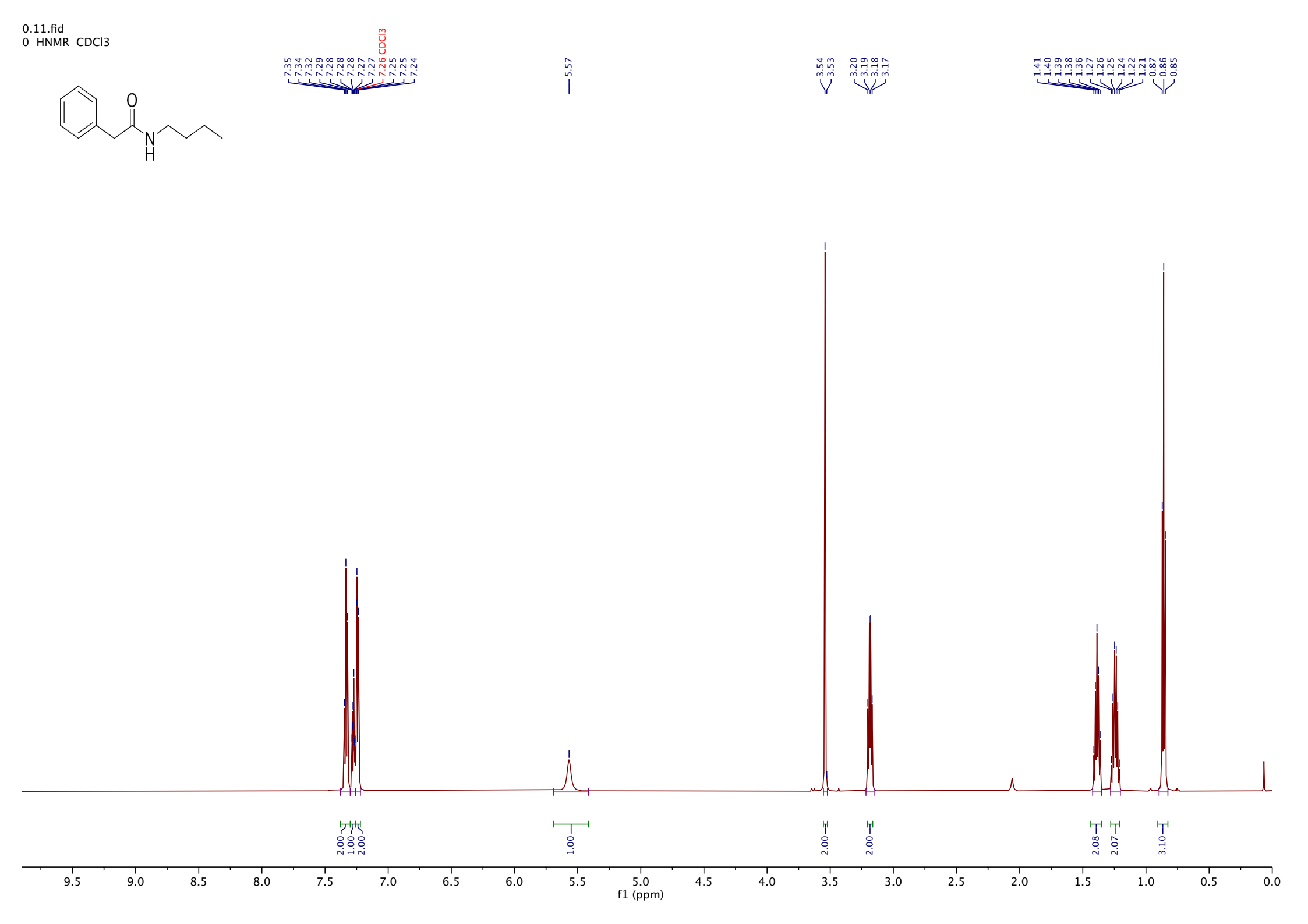


.31.fid
CNMR CDCl3

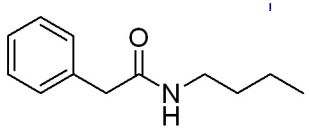


170.89
135.04
129.29
128.83
127.14
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
43.71
41.21
22.59
11.11

f1 (ppm)

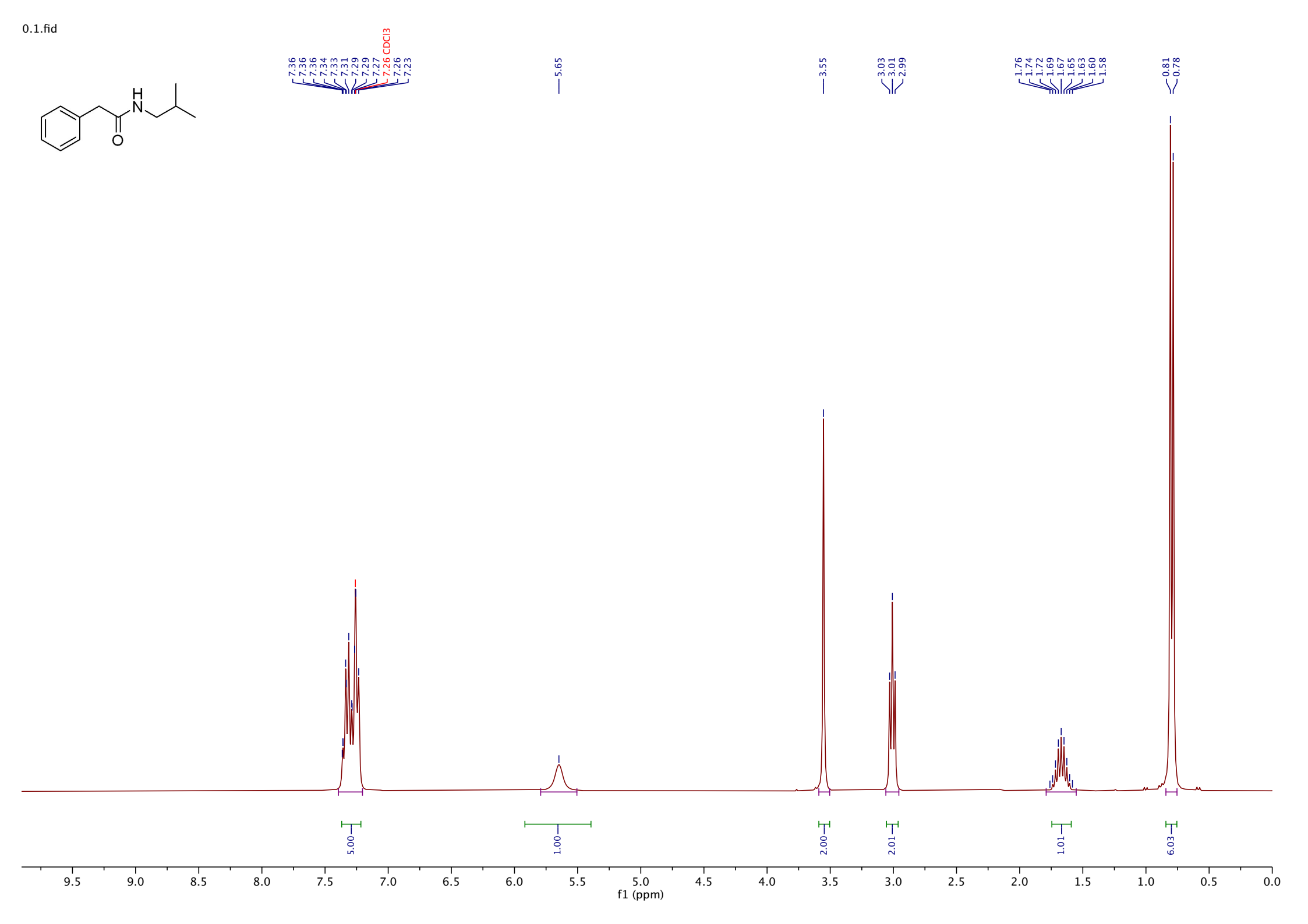

0.11.fid
0 HNMR CDCl3
O
N
H
7.35
7.34
7.32
7.29
7.28
7.28
7.28
7.27
7.27
7.26 CDCl3
7.25
7.25
7.24
5.57
3.54
3.53
3.20
3.19
3.18
3.17
1.41
1.40
1.39
1.38
1.36
1.27
1.26
1.25
1.24
1.22
1.21
0.87
0.86
0.85
2.00
1.00
2.00
1.00
2.00
2.00
2.08
2.07
3.10
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

0.34.fid
0 CNMR CDCl3

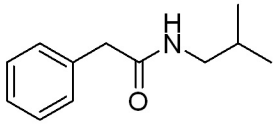


170.83
135.04
129.31
128.86
127.16
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
43.74
39.30
31.43
19.87
13.60

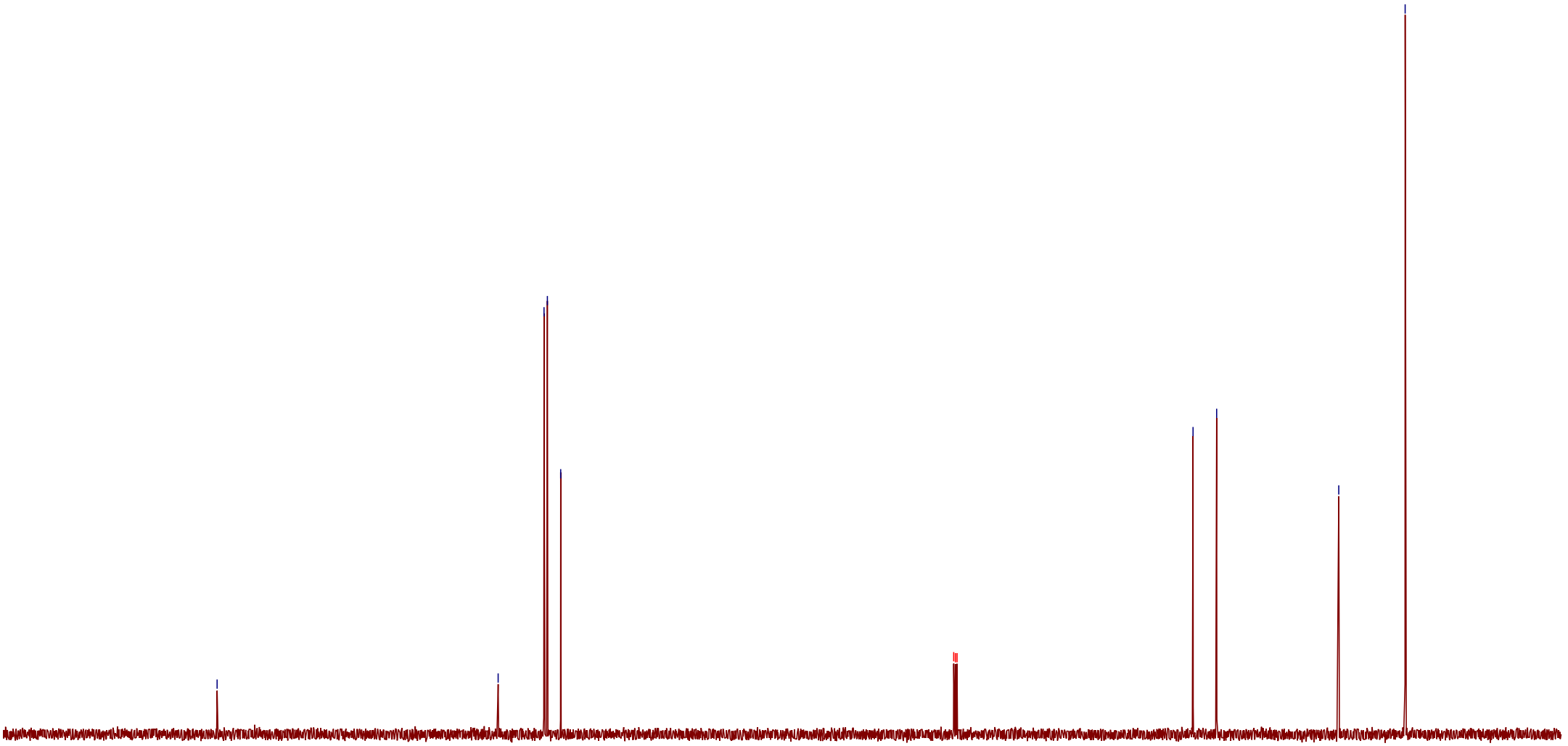
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

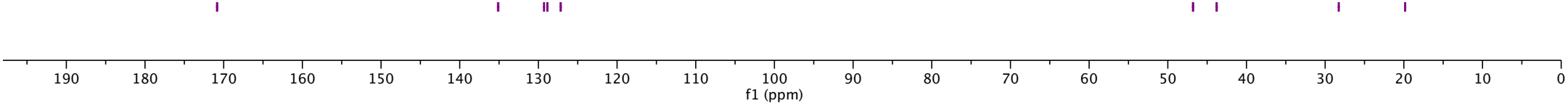

0.1.fid
7.36
7.36
7.36
7.34
7.33
7.31
7.29
7.29
7.27
7.26 CDCl3
7.26
7.23
5.65
3.55
3.03
3.01
2.99
1.76
1.74
1.72
1.69
1.67
1.65
1.63
1.60
1.58
0.81
0.78
H
N
O
5.00
1.00
2.00
2.01
1.01
6.03
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

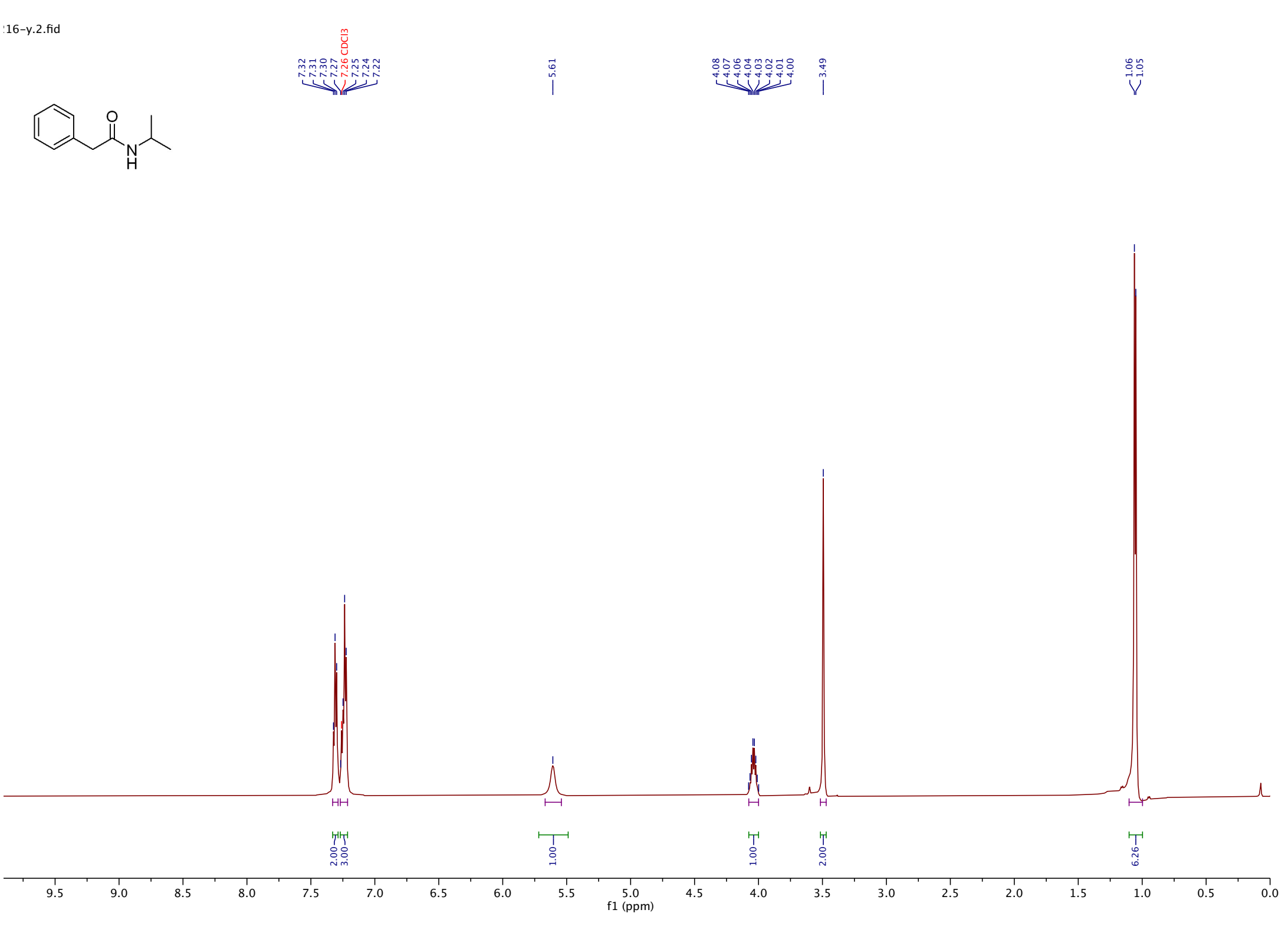
0.1.fid

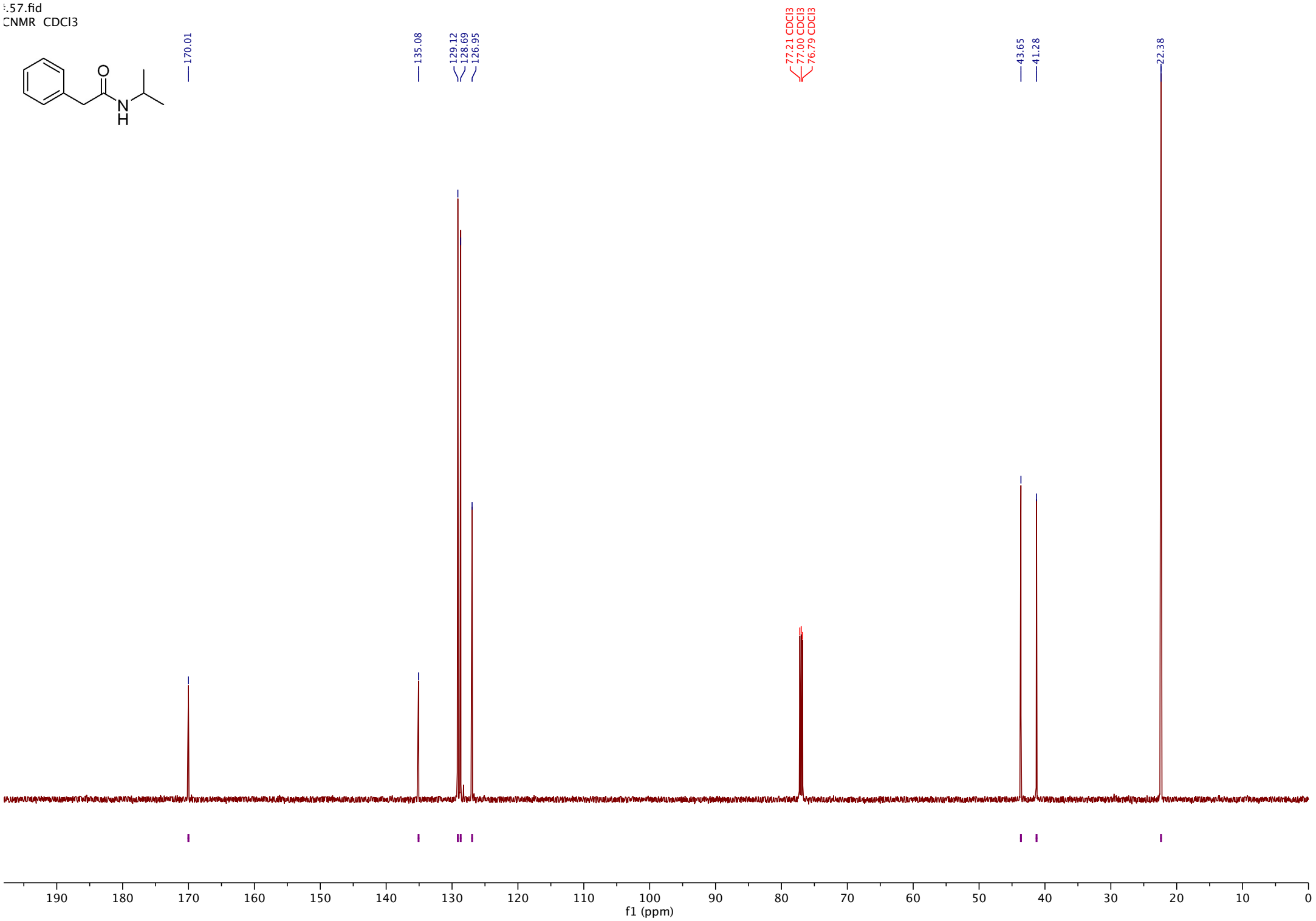


135.12
129.29
128.86
127.17
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
46.79
43.79
28.27
19.83

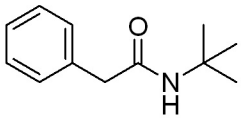




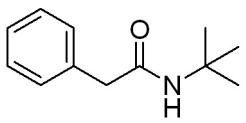

16-y.2.fid
7.32
7.31
7.30
7.27
7.26 CDCl3
7.25
7.24
7.22
5.61
4.08
4.07
4.06
4.04
4.03
4.02
4.01
4.00
3.49
1.06
1.05
O
N
H
2.00
3.00
1.00
1.00
2.00
6.26
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

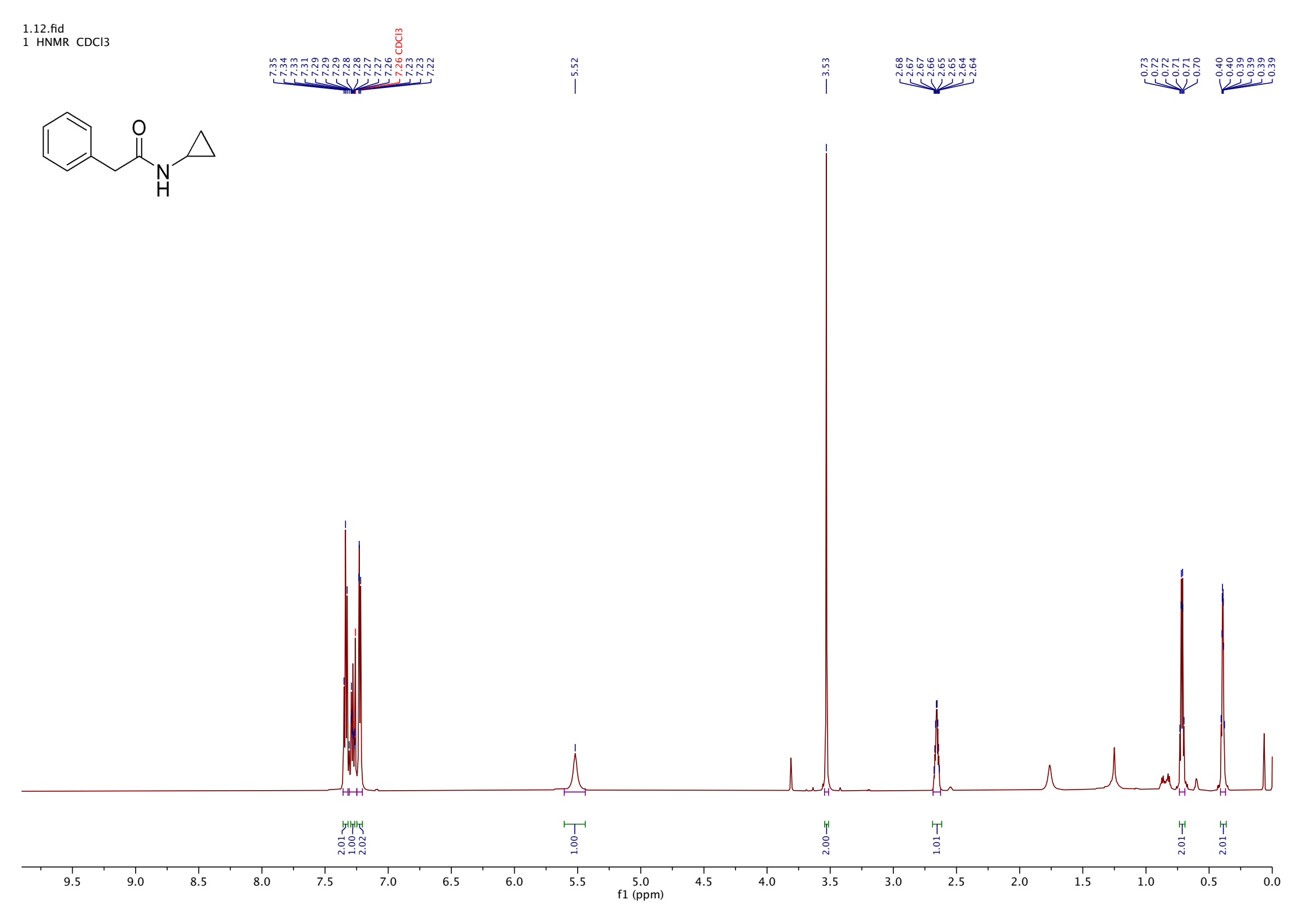

.57.fid
CNMR CDCl3
O
N
H
170.01
135.08
129.12
128.69
126.95
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
43.65
41.28
22.38
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

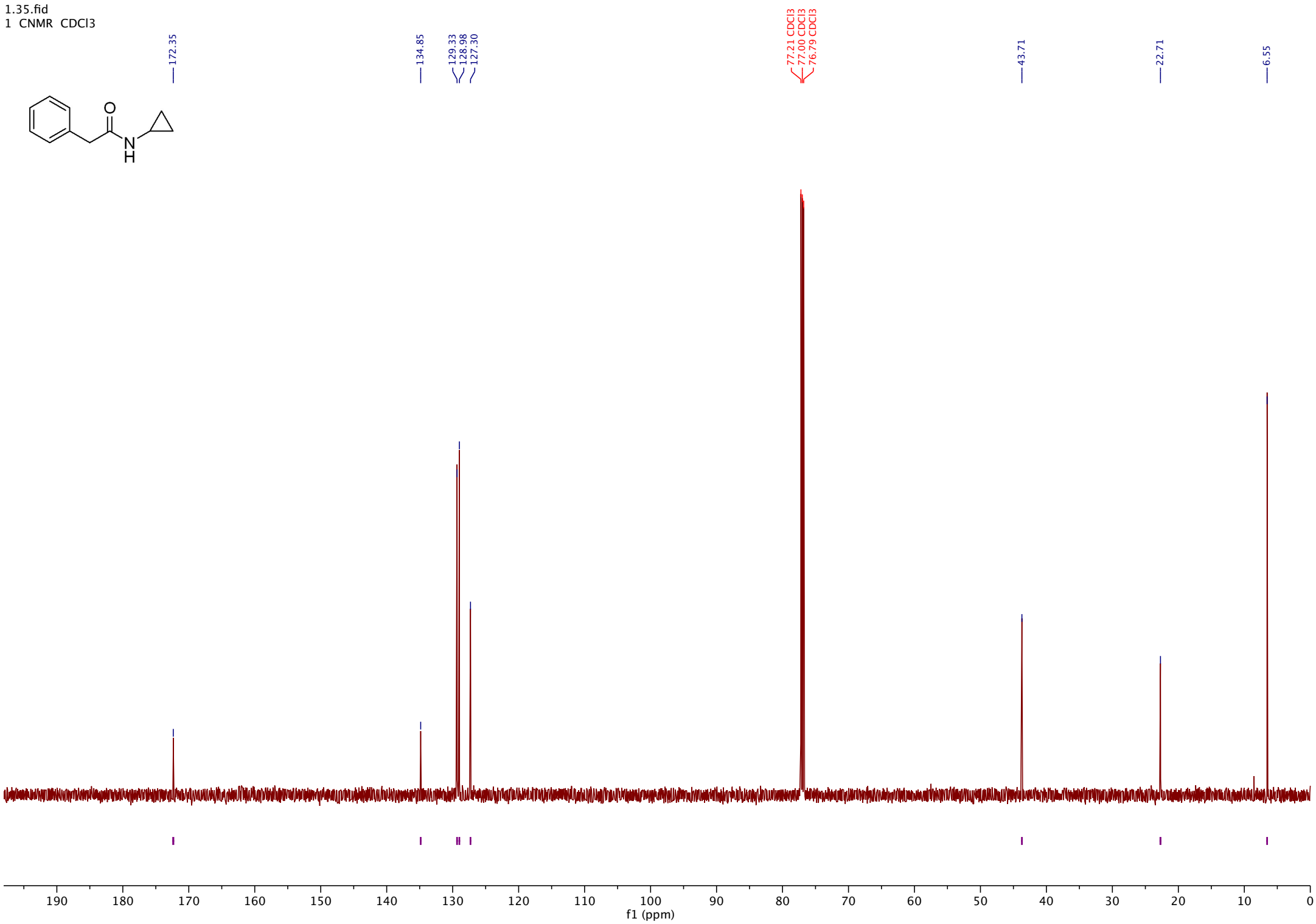
2.13.fid
2 HNMR CDCl3



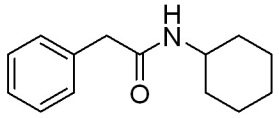
2.36.fid
2 CNMR CDCl3




1.12.fid
1 HNMR CDCl3
7.35
7.34
7.33
7.31
7.29
7.29
7.29
7.28
7.28
7.27
7.27
7.26
7.26 CDCl3
7.23
7.23
7.22
5.52
3.53
2.68
2.67
2.67
2.66
2.65
2.65
2.64
2.64
0.73
0.72
0.72
0.71
0.71
0.70
0.40
0.40
0.39
0.39
0.39
0.39
O
N
H
2.01
1.00
2.02
1.00
2.00
1.01
2.01
2.01
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
f1 (ppm)


1.35.fid
1 CNMR CDCl3
172.35
134.85
129.33
128.98
127.30
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
43.71
22.71
6.55
O
N
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

.1.fid

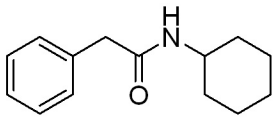


7.33
7.31
7.28
7.26 CDCl3
7.26
7.23
7.21
5.49
3.76
3.73
3.70
3.67
3.50
1.81
1.78
1.61
1.56
1.51
1.26
1.23
1.08
1.05
1.01
0.98
0.94

2.00
3.01
1.00
1.00
2.00
2.00
3.01
2.02
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
f1 (ppm)

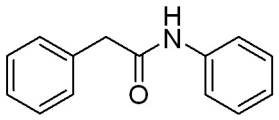
.1.fid



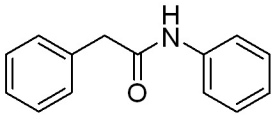
135.18
129.17
128.75
127.03
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
48.06
43.82
32.76
25.35
24.59

f1 (ppm)

.07-y.7.fid
H1-NMR DMSO-d6 303K AVANCE NEO 600



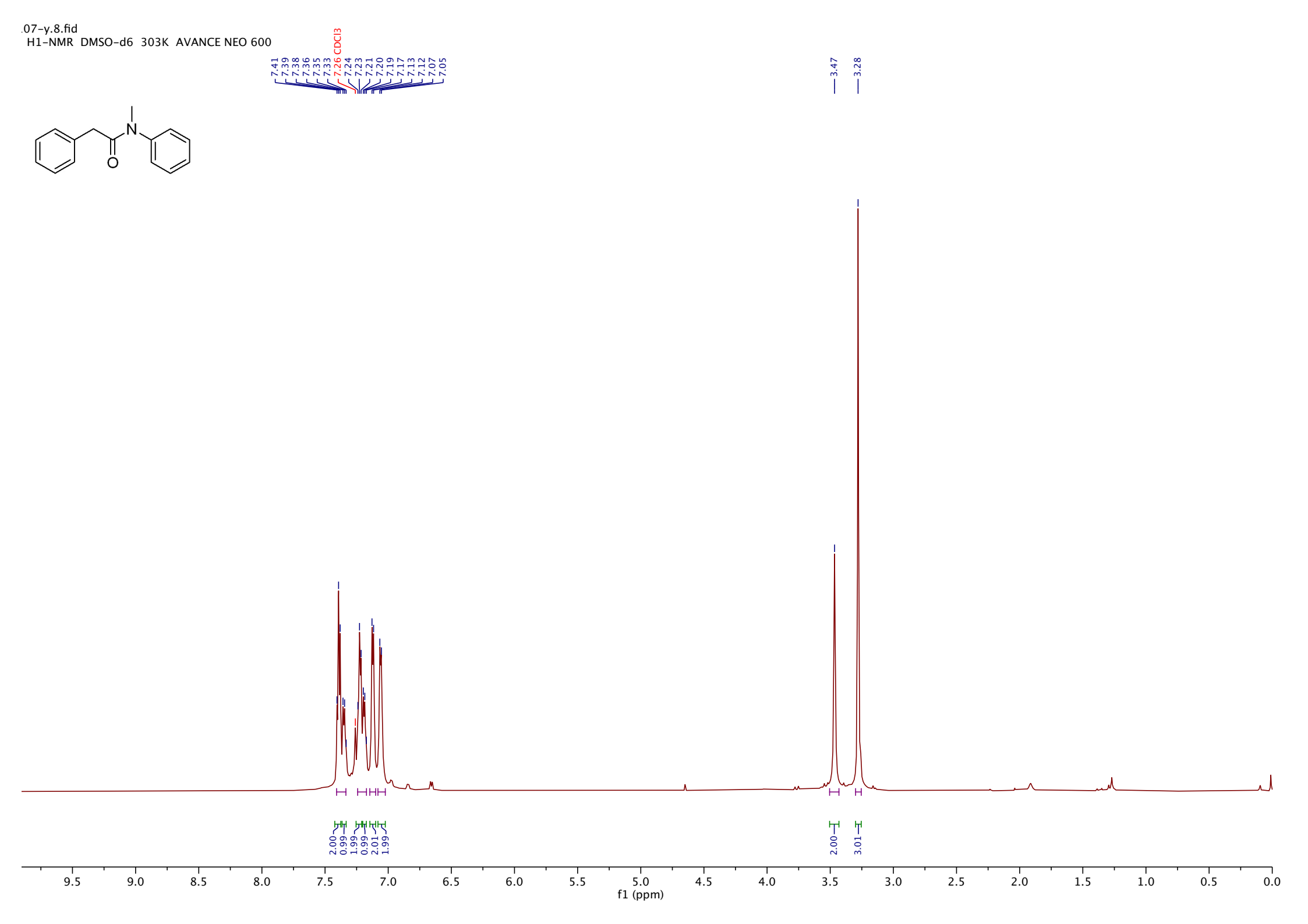
2.fid



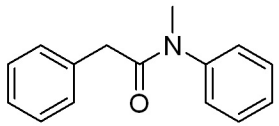
169.18
137.66
134.48
129.43
129.10
128.86
127.54
124.38
119.88
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
44.70

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

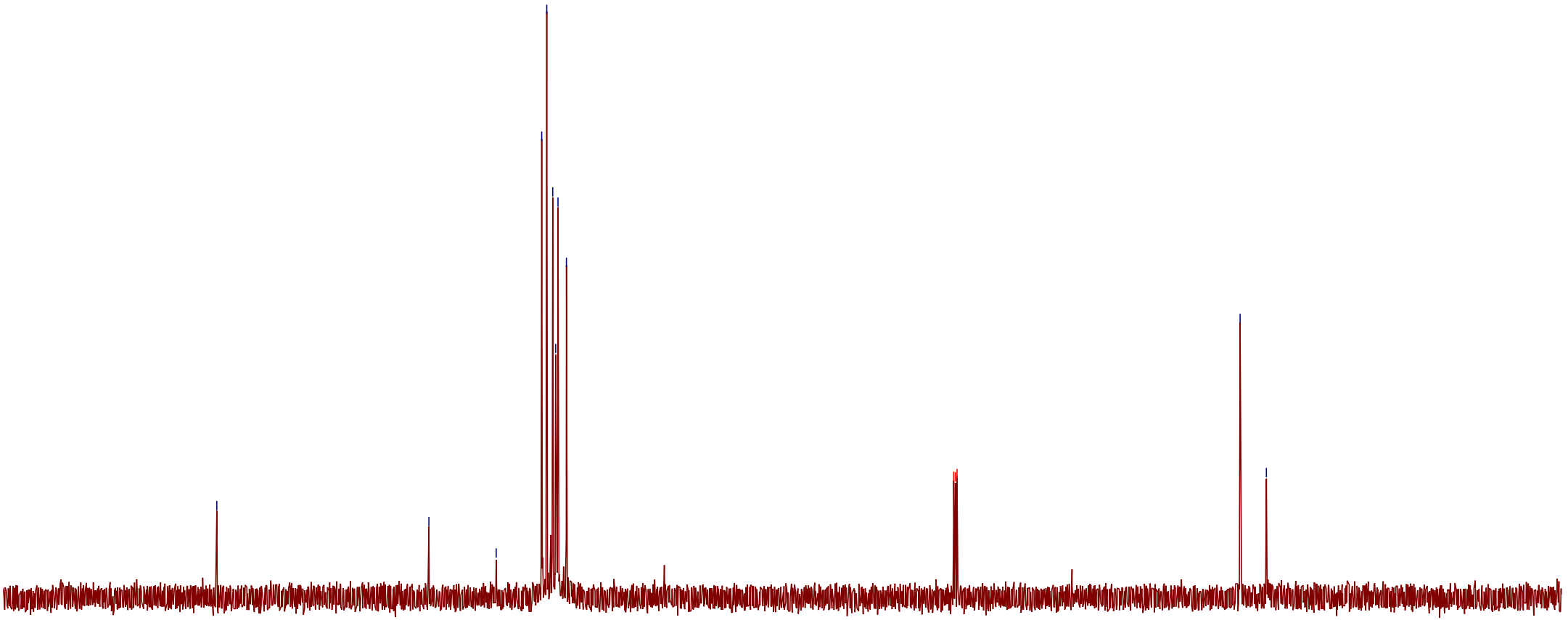
f1 (ppm)

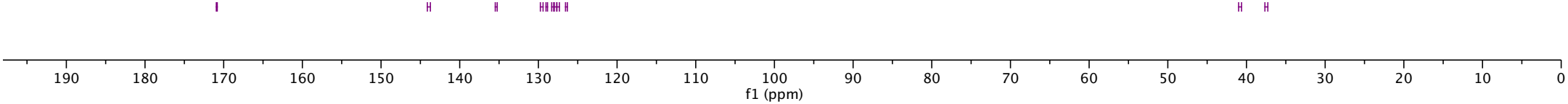

.07-y.8.fid
H1-NMR DMSO-d6 303K AVANCE NEO 600
7.41
7.39
7.38
7.36
7.35
7.33
7.26 CDCl3
7.24
7.23
7.21
7.20
7.19
7.17
7.13
7.12
7.07
7.05
3.47
3.28
N
O
2.00
0.99
1.99
0.99
2.01
1.99
2.00
3.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

.08-y.8.fid

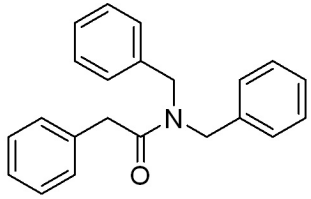


170.87
143.92
135.36
129.58
128.94
128.18
127.80
127.51
126.44
77.21 CDCl3
77.00 CDCl3
76.79 CDCl3
40.81
37.47





.1.fid



7.40 7.39 7.37 7.35 7.34 7.33 7.32 7.32 7.30 7.30 7.28 7.26 CDCl3 7.24 7.22 7.15 7.13

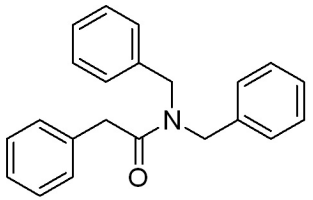
4.66 4.47 3.83

11.03 2.00 2.00 2.00 2.00 2.13

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)

.1.fid



171.43

137.17
136.29
134.90
128.82
128.70
128.56
128.42
128.18
127.52
127.26
126.74
126.33

77.21 CDCl3
77.00 CDCl3
76.79 CDCl3

50.10
48.12

40.83

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

f1 (ppm)