SUPPORTING INFORMATION

Manganese-catalyzed nucleophilic addition of aldehydes to carbonyl compounds via hydrazone umpolung on water

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1. General Experimental Information:

Reaction Setup: All reactions were carried out in oven-dried microwave reaction vials which were covered by aluminum seals with PTFE-faced silicone septa unless otherwise stated. Reactions in organic solvents were done under an atmosphere of nitrogen and were charged and sealed inside the glovebox unless otherwise stated. Reactions done under air and open-air conditions were charged and sealed outside the glovebox, with open-air having a needle pierced through the silicone septa during the reaction. All reaction temperatures correspond to either oil bath or heating block temperatures. All air and moisture sensitive catalysts, ligands, and reagents were stored in MBRAUN UNIlab Pro Glove Box Workstation unless otherwise stated.

Purifications: All work-up and purification procedures were carried out with reagent-grade solvents. Short-packed column chromatography was performed with Silicycle SiliaFlash silica gel F60 (230–400 mesh) or Biotage Sfär silica HC D 20 μm. Flash column chromatography was performed with Isolera One Prime advanced automatic flash purification system. Thin layer chromatography (TLC) was performed using Silicycle 60Å 250 μm glass backed plates. Visualization was accomplished with UV light and/or *p*-anisaldehyde solution.

Solvents and Chemicals: Solvents and reagents were purchased from Sigma-Aldrich and Fisher Scientific chemical companies and were used without further purification unless otherwise specified. Bis(2-(diphenylphosphino)ethyl)amine hydrochloride was purchased from Ambeed and Strem Chemicals, while manganese(0) carbonyl from Ambeed. Dry solvents (e.g. toluene, dioxane, THF) were taken directly from the *Pure Solvent MD-7* purification system (Innovative Technology).

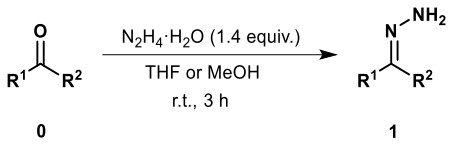
NMR Spectroscopy: ^{1}H (500 MHz), ^{13}C (126 MHz), ^{19}F (471 MHz) NMR spectra were recorded on Bruker AV500 and Varian MERCURY plus-500 spectrometers. Chemical shifts for both ^{1}H and ^{13}C NMR spectra are expressed in parts per million (ppm) units downfield from TMS, with the solvent residue peak as the chemical shift standard ($CDCl_3$: δ 7.26 ppm in ^{1}H NMR; δ 77.16 ppm in ^{13}C NMR). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, dd = doublet of doublets, etc.), coupling constant *J* (Hz), and integration. All NMR spectra were recorded at room temperature.

Mass Spectrometry: High-resolution mass spectrometry was conducted by using electro-spraying ionization (ESI) performed by McGill University on a Thermo-Scientific Exactive Orbitrap. Protonated/deprotonated molecular ions or sodium adducts were used for empirical formula confirmation.

Characterization of Products: The products are known compounds which were noted with references in characterization data section and reported with NMR data.

2. Experimental Procedures

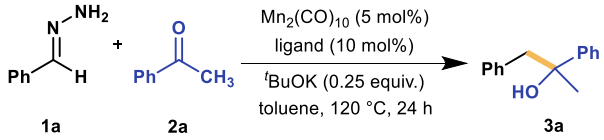
2.1 General procedure for preparation of hydrazone



0 1

Hydrazones were prepared based on reported literature.[1] To a round bottom flask with a magnetic stir bar, solvent (THF or MeOH, 5 mL) and hydrazine monohydrate (0.68 mL, 1.4 equiv.) were added. While stirring, **0** (10 mmol) was added dropwise into the solution for 5 min. Then, a proper amount of anhydrous Na_2SO_4 was added to remove water. After stirring for another 3 h, Na_2SO_4 was filtered out and the mixture was concentrated under vacuum.

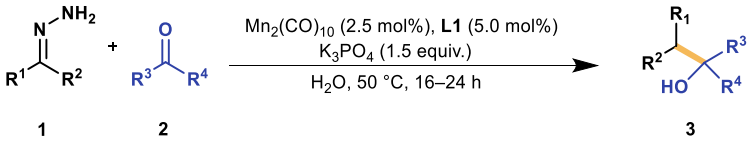
2.2 General procedure A: addition of 1a to 2a in organic solvents (for Scheme 2)



1a 2a 3a

To an oven-dried microwave reaction vial with magnetic stir bar, ^{t}BuOK (7.0 mg, 0.25 equiv.), $Mn_2(CO)_{10}$ (4.9 mg, 0.050 equiv.), and 0.10 equiv. of ligand were introduced. The mixture was diluted with dry toluene (0.50 mL), covered with a rubber septum, and stirred for 1.5 h at room temperature. Afterwards, 0.25 mmol (29.2 µL) of **2a** was added followed by 1.0 equiv. of **1a**. The inside wall of the vial was then washed with another 0.50 mL dry toluene and the mixture was stirred for 24 h at 120 °C while being covered by an aluminum seal with PTFE-faced silicone septa. Once complete, the mixture was passed through a celite plug and concentrated under vacuum. Then, 1,3,5-trimethoxybenzene was added as an NMR internal standard before diluting with $CDCl_3$ and analyzing by ^{1}H NMR.

2.3 General procedure B: addition of 1 to 2 on water (for Table 1 and Scheme 3)



1 2 3

To an oven-dried microwave reaction vial with magnetic stir bar, K_3PO_4 (79.6 mg, 1.5 equiv.), $Mn_2(CO)_{10}$ (2.4 mg, 0.025 equiv.), and bis(2-(diphenylphosphino)ethyl)amine hydrochloride (**L1**, 6.0 mg, 0.05 equiv.) were introduced. The mixture was diluted with distilled H_2O (0.50 mL), covered with a rubber septum, and stirred for 5 minutes at room temperature. Afterwards, 0.25 mmol of **2** was added followed by 3.0 equiv. of **1**. The inside wall of the vial was then washed with another 0.50 mL distilled H_2O and the mixture was stirred for 16–24 h at 50 °C while being covered by an aluminum seal with PTFE-faced silicone septa. Once complete, the reaction was cooled down to room temperature, extracted with ethyl acetate (3 × 5 mL), and dried over anhydrous Na_2SO_4. The solution was filtered, concentrated under vacuum, and the residue was

purified by either column chromatography or preparative TLC (using hexanes and ethyl acetate as eluent) to give alcohol **3**.

For substrates **2k** and **2l**: 0.050 equiv. (4.9 mg) of $Mn_2(CO)_{10}$ and 0.10 equiv. (11.9 mg) of **L1** were added while everything else remained the same.

3. Additional Reaction Optimization Data

3.1 Ligand screening

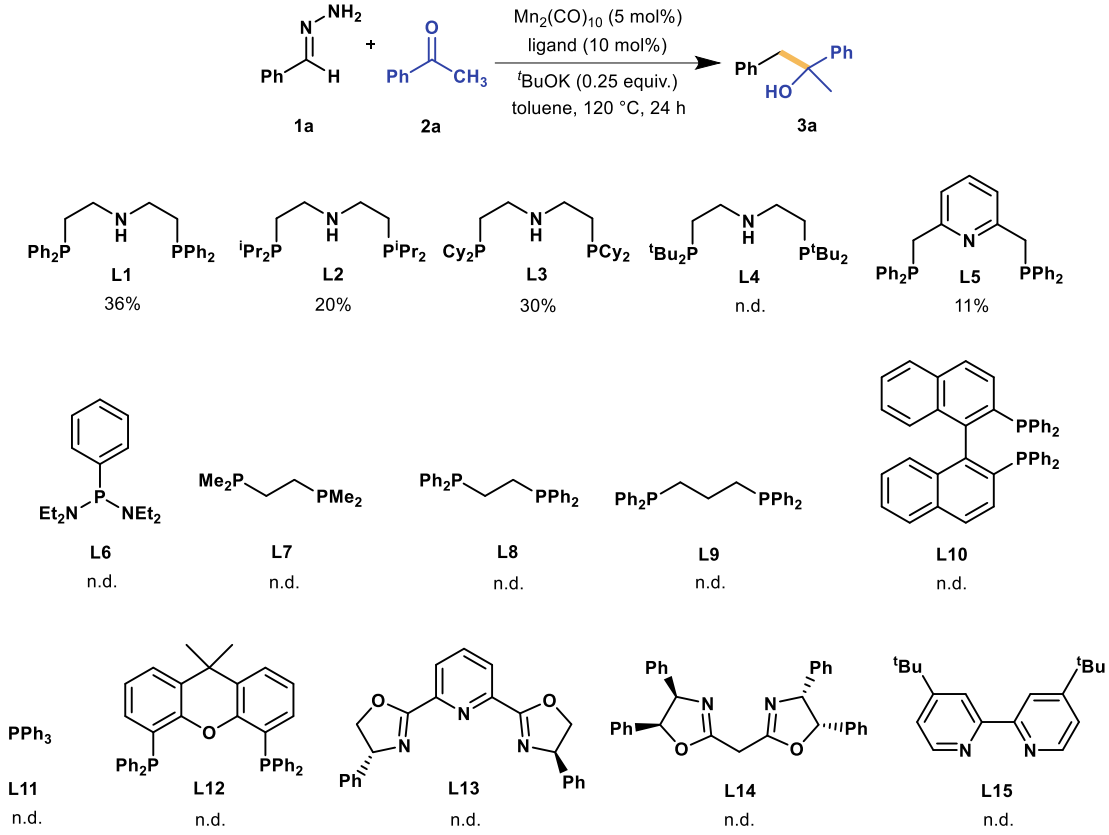


Figure S1. List of ligands screened with their corresponding product **3a** NMR yields. Reactions were done following general procedure A. n.d.: product **3a** not detected.

3.2 Reaction variations

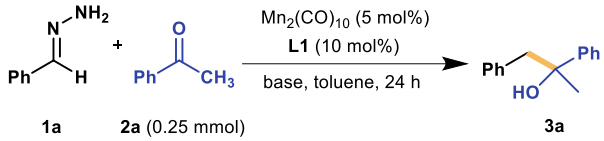


Table S1. Condition deviations from general procedure A.

Entry	Temperature (°C)	Base	1a (equiv.)	3a NMR Yield (%)
1	120	0.25 equiv. ^{t}BuOK	1.0	36
2	120	2.0 equiv. ^{t}BuOK	1.0	0
3	70	0.25 equiv. ^{t}BuOK	1.0	36
4	70	2.0 equiv. ^{t}BuOK	1.0	0
5	r.t.	0.25 equiv. ^{t}BuOK	1.0	10
6	r.t.	2.0 equiv. ^{t}BuOK	1.0	0
7	70	0.25 equiv. ^{t}BuOK	3.0	45
8	70	2.0 equiv. K_3PO_4	1.0	33
9	70	2.0 equiv. K_3PO_4	3.0	80
10	50	2.0 equiv. K_3PO_4	3.0	80
11	r.t.	2.0 equiv. K_3PO_4	3.0	13

3.3 Base screening

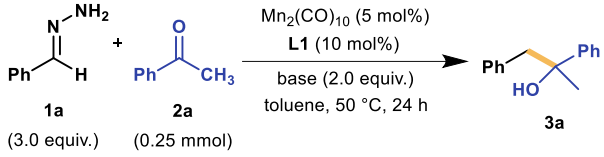


Table S2. List of bases screened and corresponding product **3a** NMR yields.

Entry	Base	3a NMR Yield (%)
1	^{t}BuOK	0
2	KOH	47
3	K_3PO_4	80
4	K_2CO_3	46
5	$NaHCO_3$	0
6	TBD	0
7	DBU	0
8	imidazole	0
9	aniline	0
10	Ph_2NH	0

3.4 Solvent experiments

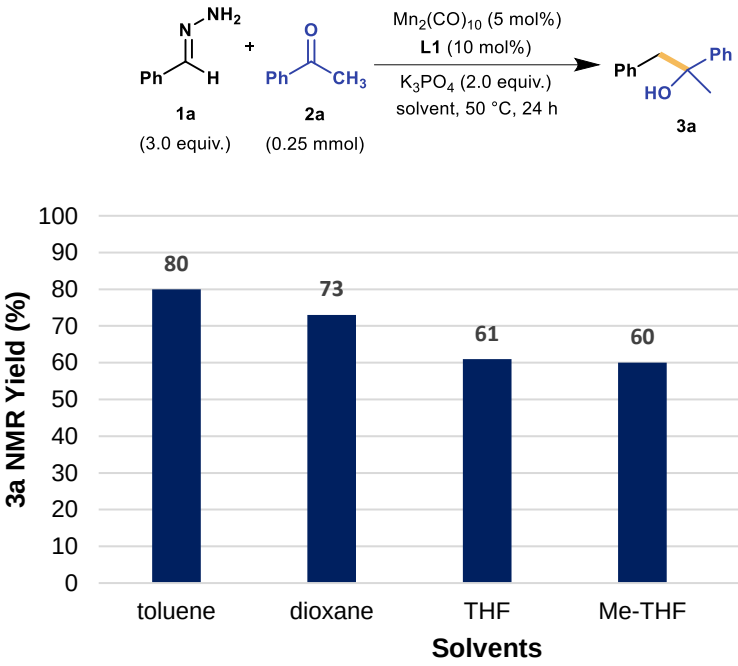


Figure S2. List of solvents screened and corresponding product **3a** NMR yields.

3.5 Catalyst loading vs. solvent experiments

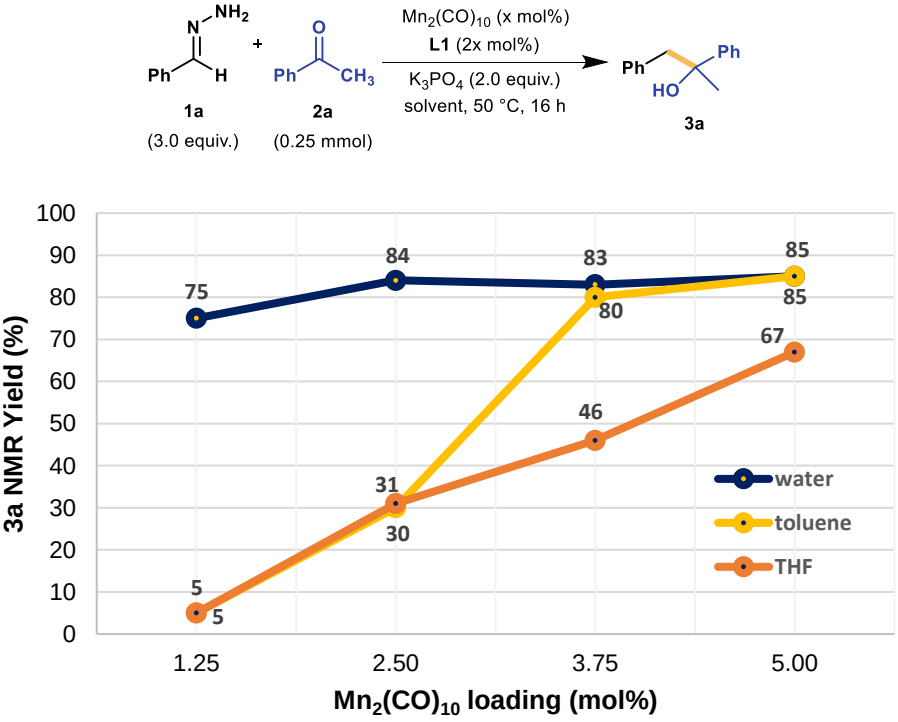


Figure S3. Pre-catalyst loading against solvent optimization with corresponding **3a** NMR yields. Reactions in THF and toluene were done under air and were also added with 0.4 equivalents of 18-crown-6 additive.

3.6 Base equivalence vs. catalyst loading experiments

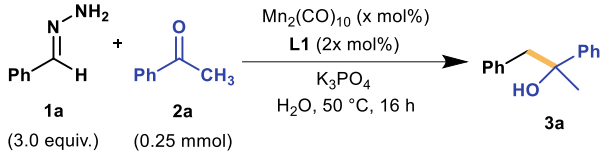


Table S3. Base optimization against pre-catalyst loading with corresponding **3a** NMR yields (%).

Entry	K_3PO_4 equivalence				$Mn_2(CO)_{10}$ loading
	0.5	**1.0**	**1.5**	**2.0**	
1	76	78	87	70	**2.5 mol%**
2	72	41	61	66	**3.75 mol%**

3.7 Hydrazone equivalence optimization

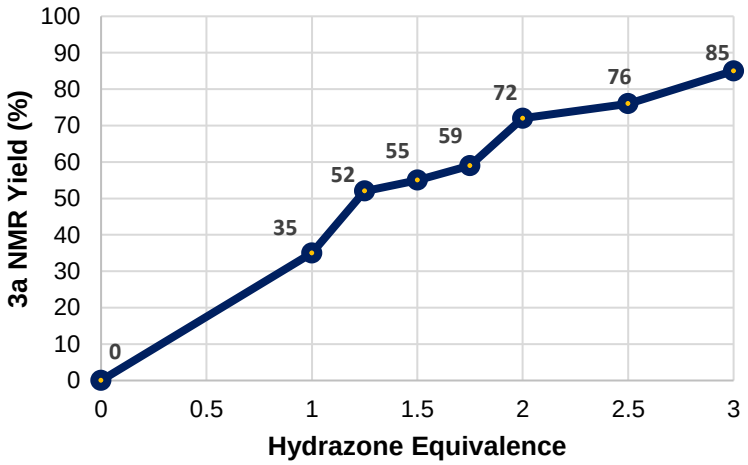


Figure S4. Hydrazone equivalence optimization in 1.0 mL H_2O with corresponding **3a** NMR yields after 16 h reaction time.

Table S4. Additional hydrazone equivalence optimization in either H_2O or neat with corresponding **3a** NMR yields after 4 h reaction time.

Entry	1a equiv.	Solvent	3a NMR Yield (%)
1	1.25	0.2 mL H_2O	84
2	1.25	neat	34
3	1.50	neat	65
4	2.00	neat	76
5	3.00	neat	82

3.8 Pre-mixing time optimization

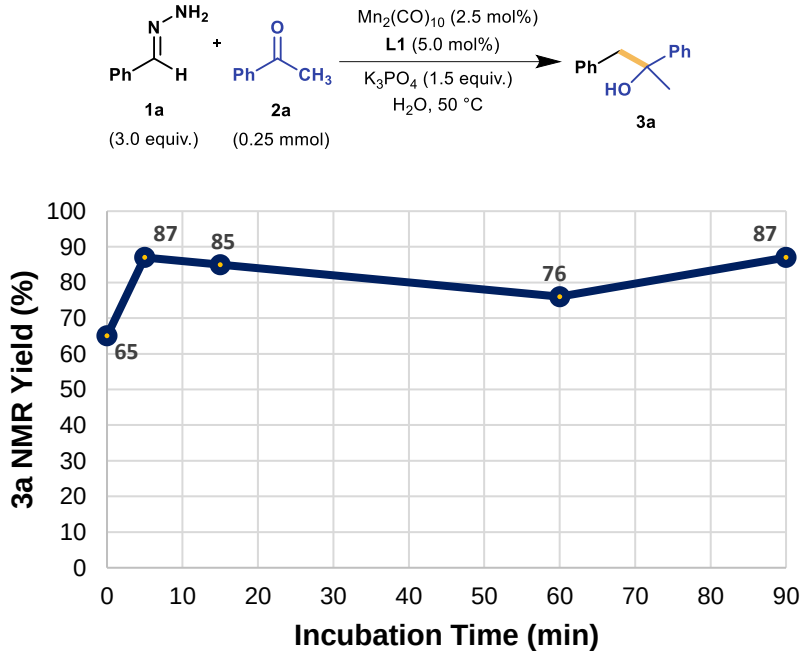


Figure S5. Mixing time optimization for $Mn_2(CO)_{10}$, **L1**, and base with H_2O at room temperature before adding the substrates. Total reaction time is 16 h for 50 °C.

3.9 Total reaction time for addition of 1a to 2a

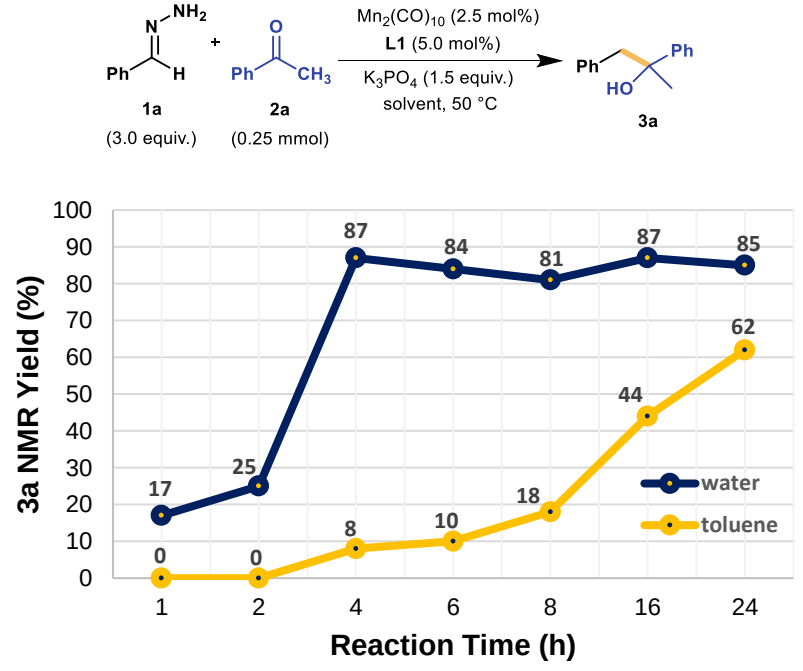


Figure S6. Total reaction time optimization with 5 minutes of pre-mixing at room temperature.

4. Green Metrics Calculation

Table S5. Values used to calculate the quantitative green metrics for the nucleophilic addition of **1a** to **2a** via hydrazone umpolung. These estimated values are based either on reported literature (Ru and Fe) or from the current method (Mn).

	Ru THF[2]	**Fe THF**[1]	**Ru water**[3]	**Mn water**
Scale (mmol)	0.4	0.2	0.2	0.2
Acetophenone (mg)	48.1	24.0	24.0	24.0
Benzaldehyde hydrazone (mg)	60.1	30.0	36.0	30.0
Base (mg)	21.2	21.2	31.8	63.7
Pre-catalyst (mg)	1.8	4.3	1.8	2.0
Ligand (mg)	0.9	0	3.1	4.8
Additives (mg)	30.0	15.2	11.1	0
Reaction Solvent (mg)	177.6	177.6	244.4	200.0
Work-up Solvents (mg)	1804.0	2706.0	23630.0	12000.0
Purification Solvents (mg)	218923.2	109461.6	109461.6	109461.6
Isolated Product (mg)	79.4	43.0	30.1	35.6
Yield (%)	94	>99	71	84
AE (%)	88	88	88	88
RME (%)	73	80	50	66
PMI (total)	2.8E+03	2.6E+03	4.4E+03	3.4E+03

4.1 Atom economy

$$AE\ (\%) = \frac{molecular\ weight\ of\ product}{total\ molecular\ weight\ of\ reactants} \times 100$$

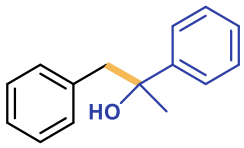
4.2 Reaction mass efficiency

$$RME\ (\%) = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

4.3 Process mass intensity

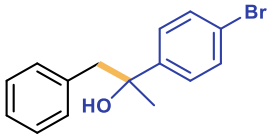
$$PMI = \frac{total\ mass\ in\ a\ process}{mass\ of\ product}$$

5. Characterization Data of Compounds



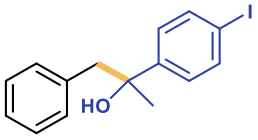
3a

1,2-Diphenylpropan-2-ol (3a): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 84%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.39 (m, 2H), 7.37 – 7.31 (m, 2H), 7.28 – 7.19 (m, 4H), 7.05 – 6.97 (m, 2H), 3.15 (d, *J* = 13.4 Hz, 1H), 3.04 (d, *J* = 13.3 Hz, 1H), 1.88 (s, 1H), 1.58 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 147.69, 136.86, 130.74, 128.19, 126.79, 126.78, 125.11, 74.58, 50.63, 29.53. Data is consistent with literature report.[2]



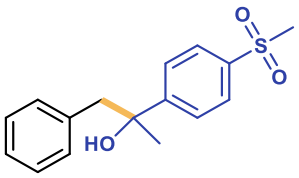
3b

2-(4-Bromophenyl)-1-phenylpropan-2-ol (3b): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 86%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.6 Hz, 2H), 7.25 – 7.19 (m, 3H), 7.02 – 6.95 (m, 2H), 3.09 (d, *J* = 13.4 Hz, 1H), 3.00 (d, *J* = 13.3 Hz, 1H), 1.83 (s, 1H), 1.54 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 146.77, 136.41, 131.23, 130.70, 128.35, 127.09, 126.98, 120.75, 74.38, 50.48, 29.60. Data is consistent with literature report.[2]



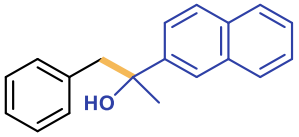
3c

2-(4-Iodophenyl)-1-phenylpropan-2-ol (3c): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 77%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 – 7.60 (m, 2H), 7.27 – 7.20 (m, 3H), 7.19 – 7.11 (m, 2H), 7.04 – 6.96 (m, 2H), 3.09 (d, *J* = 13.4 Hz, 1H), 3.00 (d, *J* = 13.4 Hz, 1H), 1.87 (s, 1H), 1.54 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.48, 137.19, 136.39, 130.68, 128.32, 127.36, 126.95, 92.31, 74.38, 50.40, 29.51. Data is consistent with literature report.[2]



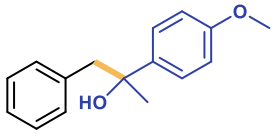
3d

2-(4-Methylsulfonyl)phenyl)-1-phenylpropan-2-ol (3d): synthesized utilizing general procedure B. Purified with a 0–60% EtOAc/hexanes gradient over silica. White solid. Yield: 60%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.6 Hz, 2H), 7.61 (d, *J* = 8.6 Hz, 2H), 7.31 – 7.22 (m, 3H), 7.04 – 6.95 (m, 2H), 3.19 – 2.93 (m, 5H), 2.01 (s, 1H), 1.58 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.11, 138.89, 135.87, 130.63, 128.49, 127.33, 127.19, 126.30, 74.51, 50.34, 44.70, 29.58. Data is consistent with literature report.[1]



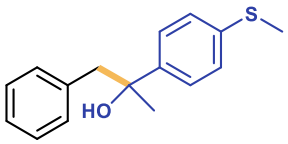
3e

2-(Naphthalen-2-yl)-1-phenylpropan-2-ol (3e): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 91%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 – 7.77 (m, 4H), 7.58 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.51 – 7.44 (m, 2H), 7.24 – 7.16 (m, 3H), 7.06 – 6.98 (m, 2H), 3.26 (d, *J* = 13.4 Hz, 1H), 3.13 (d, *J* = 13.4 Hz, 1H), 1.98 (s, 1H), 1.66 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.09, 136.75, 133.27, 132.42, 130.75 (2C), 128.34, 128.28 (2C), 127.89, 127.61, 126.85, 126.16, 125.85, 123.96, 123.56, 74.77, 50.35, 29.74. Data is consistent with literature report.[4]



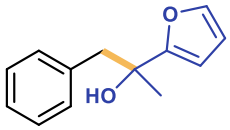
3f

2-(4-Methoxyphenyl)-1-phenylpropan-2-ol (3f): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 63%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.8 Hz, 2H), 7.25 – 7.19 (m, 3H), 7.03 – 6.96 (m, 2H), 6.86 (d, *J* = 8.9 Hz, 2H), 3.82 (s, 3H), 3.11 (d, *J* = 13.3 Hz, 1H), 3.01 (d, *J* = 13.3 Hz, 1H), 1.81 (s, 1H), 1.55 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.43, 139.91, 137.03, 130.76, 128.17, 126.73, 126.33, 113.47, 74.34, 55.39, 50.76, 29.57. Data is consistent with literature report.[1]



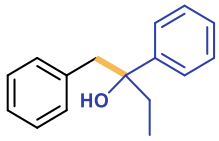
3g

2-(4-Methylthio)phenyl)-1-phenylpropan-2-ol (3g): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. White solid. Yield: 63%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.30 (m, 2H), 7.25 – 7.19 (m, 5H), 7.05 – 6.97 (m, 2H), 3.11 (d, *J* = 13.3 Hz, 1H), 3.01 (d, *J* = 13.4 Hz, 1H), 2.49 (s, 3H), 1.88 (s, 1H), 1.55 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.73, 136.73, 136.61, 130.72, 128.22, 126.80, 126.45, 125.73, 74.36, 50.52, 29.51, 16.05. Data is consistent with literature report.[1]



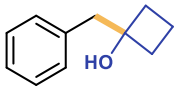
3h

1-Phenyl-2-furylpropan-2-ol (3h): synthesized utilizing general procedure B. Purified with a 0–40% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 55%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.26 – 7.18 (m, 3H), 7.01 – 6.90 (m, 2H), 6.29 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.06 (dd, *J* = 3.3, 0.9 Hz, 1H), 3.22 (d, *J* = 13.2 Hz, 1H), 3.08 (d, *J* = 13.2 Hz, 1H), 2.07 (s, 1H), 1.54 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.01, 141.48, 136.65, 130.39, 128.23, 126.83, 110.37, 105.41, 72.06, 48.17, 26.68. Data is consistent with literature report.[2]



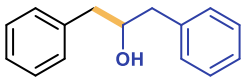
3i

1,2-Diphenylbutan-2-ol (3i): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 71%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 4H), 7.26 – 7.16 (m, 4H), 7.00 – 6.92 (m, 2H), 3.16 (d, *J* = 13.3 Hz, 1H), 3.06 (d, *J* = 13.4 Hz, 1H), 1.99 (dq, *J* = 14.1, 7.4 Hz, 1H), 1.83 (dq, *J* = 14.1, 7.2 Hz, 1H), 1.76 (s, 1H), 0.76 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.62, 136.60, 130.82, 128.19, 128.09, 126.75, 126.55, 125.73, 77.10, 49.59, 34.64, 7.99. Data is consistent with literature report.[2]



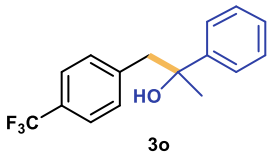
3j

1-Benzylcyclobutanol (3j): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 50%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 2H), 7.29 – 7.23 (m, 3H), 2.91 (s, 2H), 2.23 – 2.09 (m, 2H), 2.07 – 1.95 (m, 2H), 1.86 – 1.76 (m, 1H), 1.67 – 1.56 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.60, 130.17, 128.50, 126.71, 75.15, 45.61, 35.54, 12.28. Data is consistent with literature report.[5]



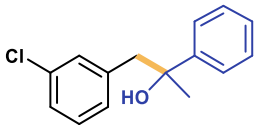
3k

1,3-Diphenylpropan-2-ol (3k): synthesized utilizing general procedure B. Purified with a 0–10% EtOAc/hexanes gradient over silica. Colorless oil. Yield: 42%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.27 (m, 4H), 7.27 – 7.12 (m, 6H), 4.08 (tt, *J* = 8.2, 4.7 Hz, 1H), 2.87 (dd, *J* = 13.6, 4.6 Hz, 2H), 2.77 (dd, *J* = 13.6, 8.2 Hz, 2H), 1.63 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.58, 129.56, 128.70, 126.65, 73.73, 43.53. Data is consistent with literature report.[6]



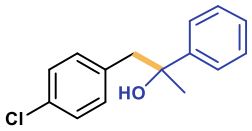
3o

1-(4-(Trifluoromethyl)phenyl)-2-phenylpropan-2-ol (3o): synthesized utilizing general procedure B. Purified with a 10% EtOAc/hexanes solvent system over preparative TLC. Light yellow oil. Yield: 81%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.9 Hz, 2H), 7.40 – 7.30 (m, 4H), 7.30 – 7.23 (m, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 3.17 (d, *J* = 13.3 Hz, 1H), 3.08 (d, *J* = 13.3 Hz, 1H), 1.79 (s, 1H), 1.59 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.14, 141.23, 131.02, 128.96 (q, *J* = 32.3 Hz), 128.35, 127.11, 125.06, 124.90 (q, *J* = 3.7 Hz), 124.47 (q, *J* = 272.0 Hz), 74.69, 50.41, 29.55. ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.39. Data is consistent with literature report.[1]



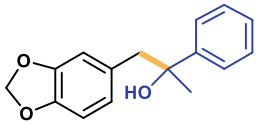
3p

1-(3-Chlorophenyl)-2-phenylpropan-2-ol (3p): synthesized utilizing general procedure B. Purified with a 10% EtOAc/hexanes solvent system over preparative TLC. Colorless oil. Yield: 67%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.36 (m, 2H), 7.36 – 7.30 (m, 2H), 7.29 – 7.23 (m, 1H), 7.18 (ddd, *J* = 8.0, 2.1, 1.2 Hz, 1H), 7.13 (t, *J* = 7.8 Hz, 1H), 7.01 (t, *J* = 1.9 Hz, 1H), 6.86 (dt, *J* = 7.5, 1.4 Hz, 1H), 3.08 (d, *J* = 13.4 Hz, 1H), 2.99 (d, *J* = 13.4 Hz, 1H), 1.79 (s, 1H), 1.57 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.30, 139.08, 133.89, 130.81, 129.27, 128.89, 128.30, 127.04, 126.90, 125.04, 74.60, 50.30, 29.42. Data is consistent with literature report.[2]



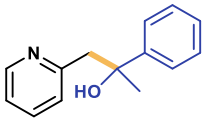
3q

1-(4-Chlorophenyl)-2-phenylpropan-2-ol (3q): synthesized utilizing general procedure B. Purified with a 10% EtOAc/hexanes solvent system over preparative TLC. Colorless oil. Yield: 40%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 4H), 7.27 – 7.23 (m, 2H), 7.19 – 7.13 (m, 2H), 6.92 – 6.87 (m, 2H), 3.09 (d, *J* = 13.4 Hz, 1H), 2.99 (d, *J* = 13.4 Hz, 1H), 1.76 (s, 1H), 1.57 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.27, 135.44, 132.70, 132.02, 128.29, 128.23, 126.99, 125.10, 74.63, 49.97, 29.53. Data is consistent with literature report.[2]



3r

1-(1,3-Benzodioxole-5-yl)-2-phenylpropan-2-ol (3r): synthesized utilizing general procedure B. Purified with 10% EtOAc/hexanes solvent system over preparative TLC. Colorless oil. Yield: 72%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.37 (m, 2H), 7.36 – 7.30 (m, 2H), 7.27 – 7.22 (m, 1H), 6.67 (d, *J* = 7.8 Hz, 1H), 6.50 – 6.42 (m, 2H), 5.90 (s, 2H), 3.05 (d, *J* = 13.6 Hz, 1H), 2.93 (d, *J* = 13.5 Hz, 1H), 1.86 (s, 1H), 1.56 (s, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.65, 147.45, 146.50, 130.50, 128.24, 126.84, 125.08, 123.75, 111.02, 108.04, 100.97, 74.53, 50.28, 29.56. Data is consistent with literature report.[2]



3s

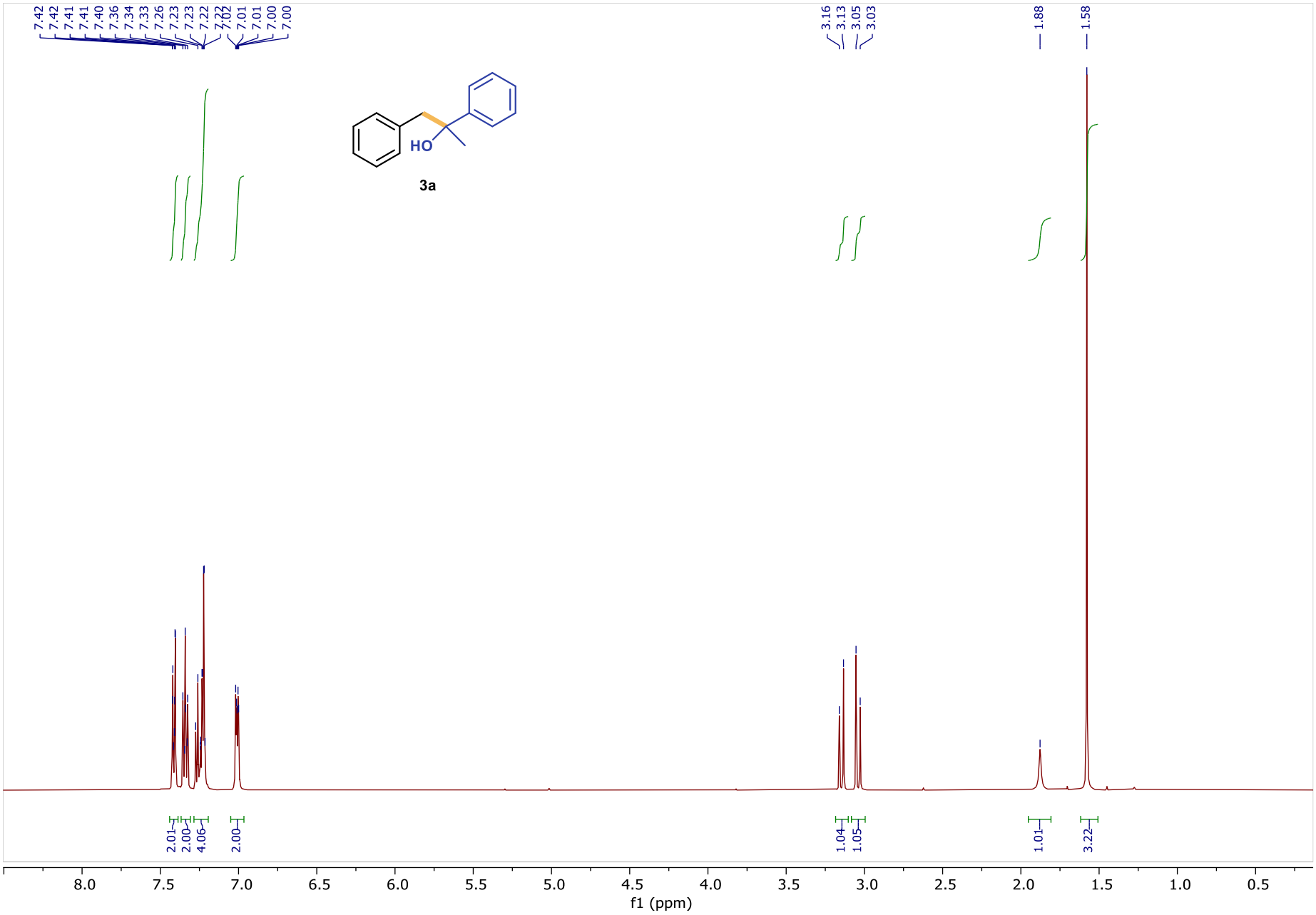
2-Phenyl-1-(pyridin-2-yl)propan-2-ol (3s): synthesized utilizing general procedure B. Purified with a 50% EtOAc/hexanes solvent system over preparative TLC. Light yellow oil. Yield: 62%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.44 (d, *J* = 4.9 Hz, 1H), 7.50 (td, *J* = 7.7, 1.9 Hz, 1H), 7.47 – 7.41 (m, 2H), 7.28 – 7.21 (m, 2H), 7.17 – 7.11 (m, 1H), 7.10 – 7.05 (m, 1H), 6.96 (d, *J* = 7.8 Hz, 1H), 6.70 (s, 1H), 3.28 (d, *J* = 14.5 Hz, 1H), 3.23 (d, *J* = 14.5 Hz, 1H), 1.56 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.48, 148.35, 148.29, 136.89, 128.00, 126.27, 125.02, 124.50, 121.65, 74.74, 49.09, 30.82. Data is consistent with literature report.[2]

References:

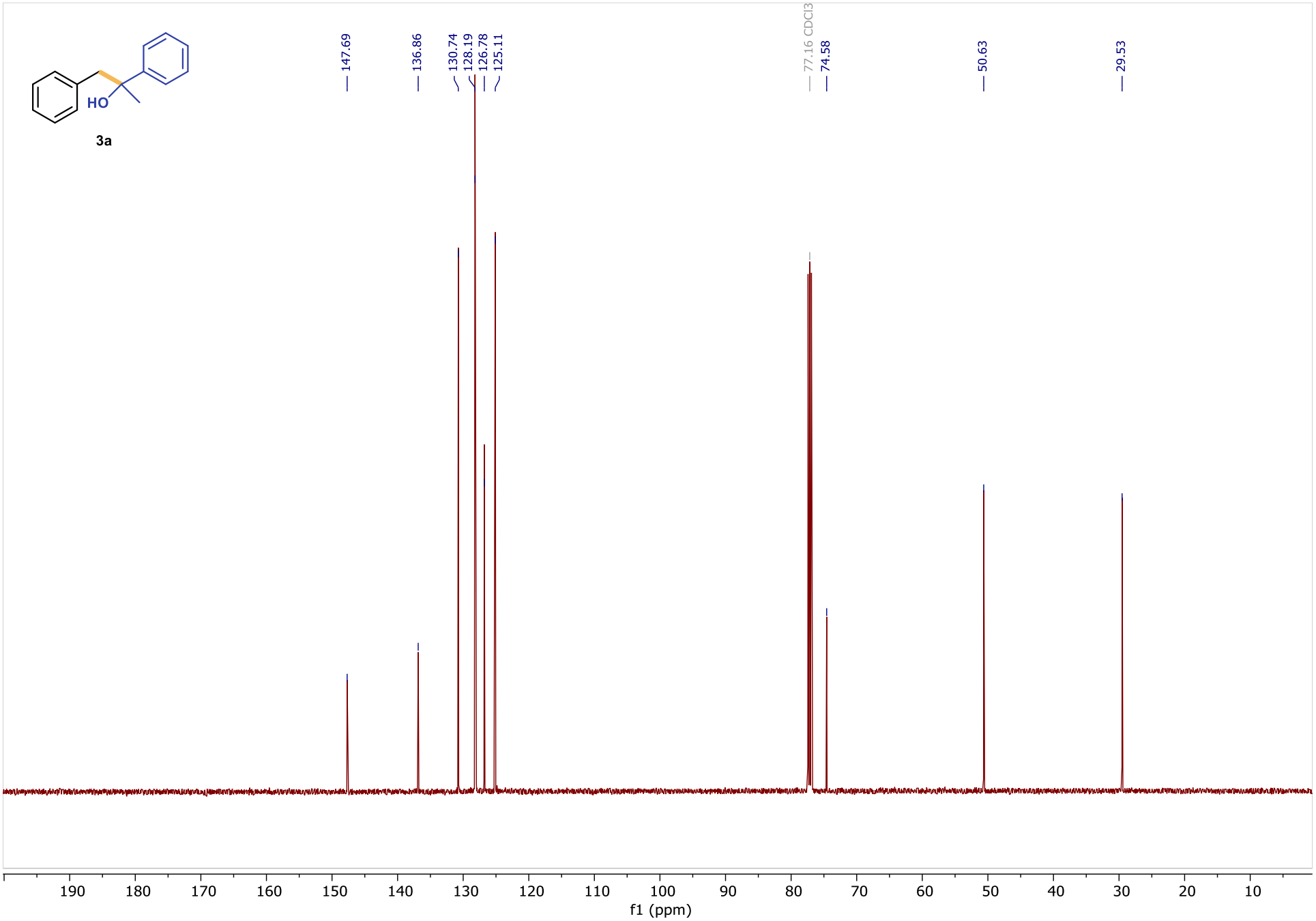
1 C.-C. Li, X.-J. Dai, H. Wang, D. Zhu, J. Gao and C.-J. Li, *Org. Lett.*, 2018, **20**, 3801–3805.
2 H. Wang, X.-J. Dai and C.-J. Li, *Nat. Chem.*, 2017, **9**, 374–378.
3 Y.-Z. Wang, Q. Liu, L. Cheng, S.-C. Yu, L. Liu and C.-J. Li, *Tetrahedron*, 2021, **80**, 131889.
4 J. Varma Nallaparaju, T. Nikonovich, T. Jarg, D. Merzhyievskyi, R. Aav and D. G. Kananovich, *Angew. Chem. Int. Ed.*, 2023, **62**, e202305775.
5 B. Xu, D. Wang, Y. Hu and Q. Shen, *Org. Chem. Front.*, 2018, **5**, 1462–1465.
6 G. Zhang, H. Zeng, J. Wu, Z. Yin, S. Zheng and J. C. Fettinger, *Angew. Chem. Int. Ed.*, 2016, **55**, 14369–14372.

Spectra Collection:

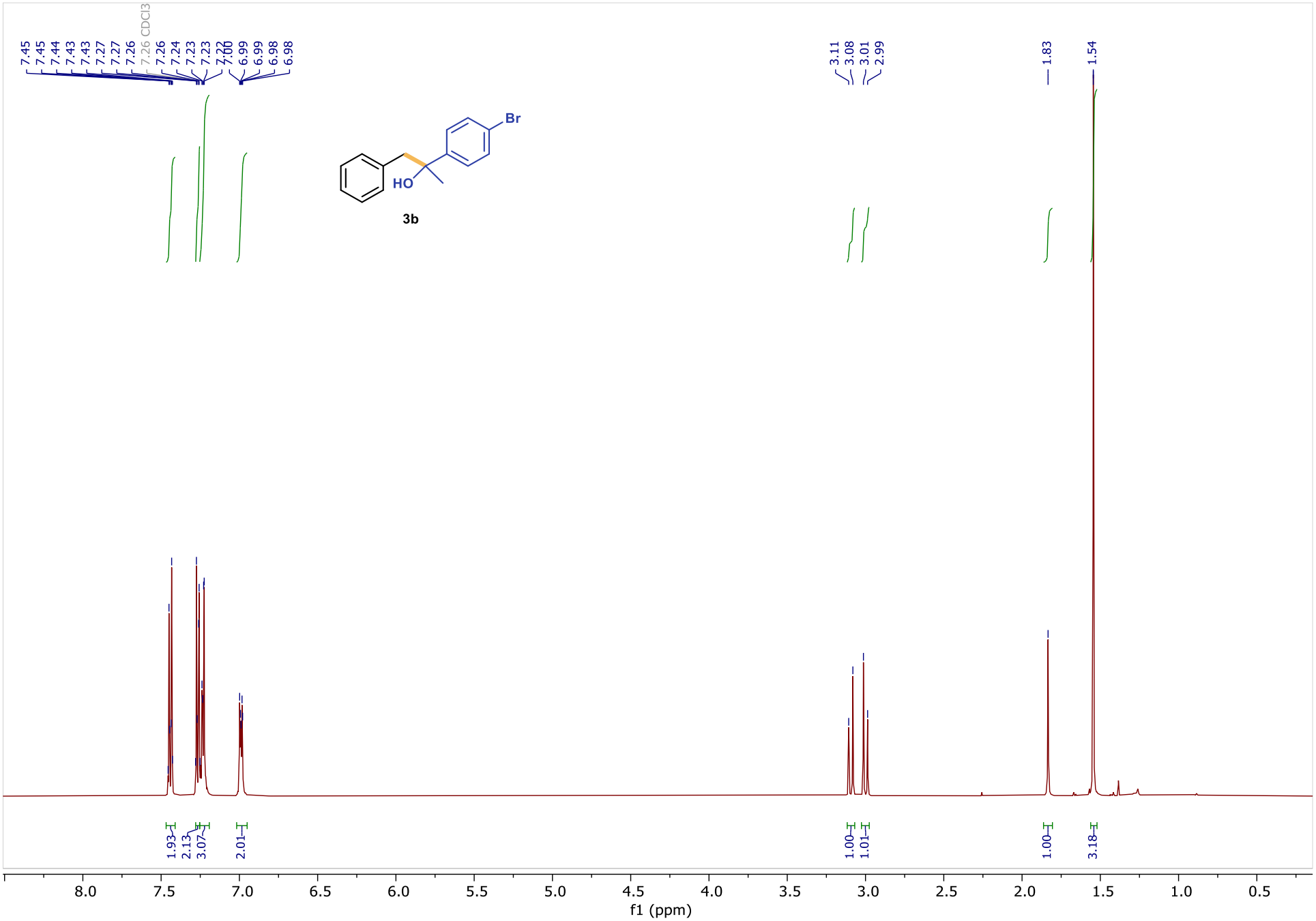
^{1}H NMR of 3a, $CDCl_3$, 500 MHz



^{13}C NMR of 3a, $CDCl_3$, 126 MHz



^{1}H NMR of 3b, $CDCl_3$, 500 MHz



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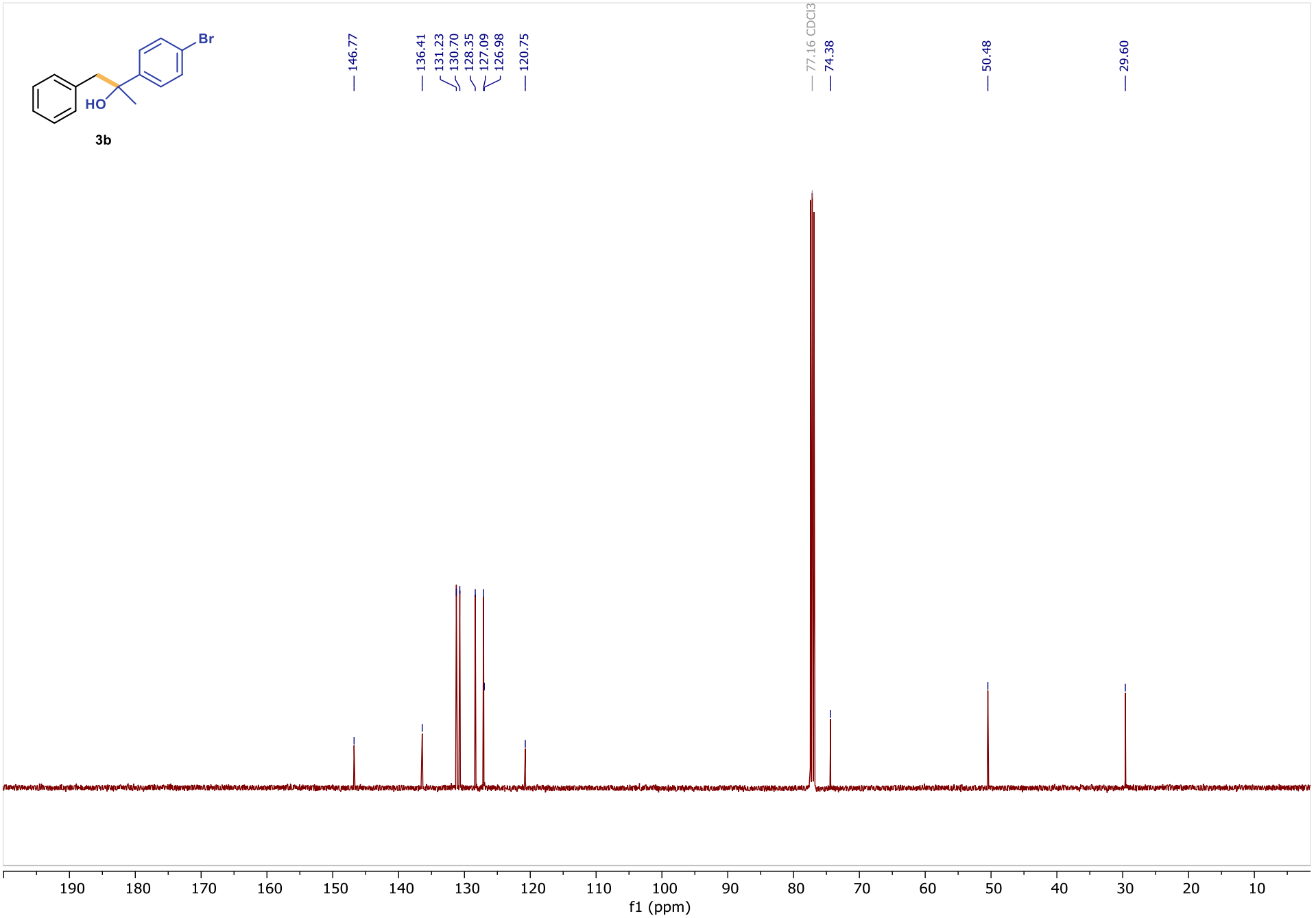
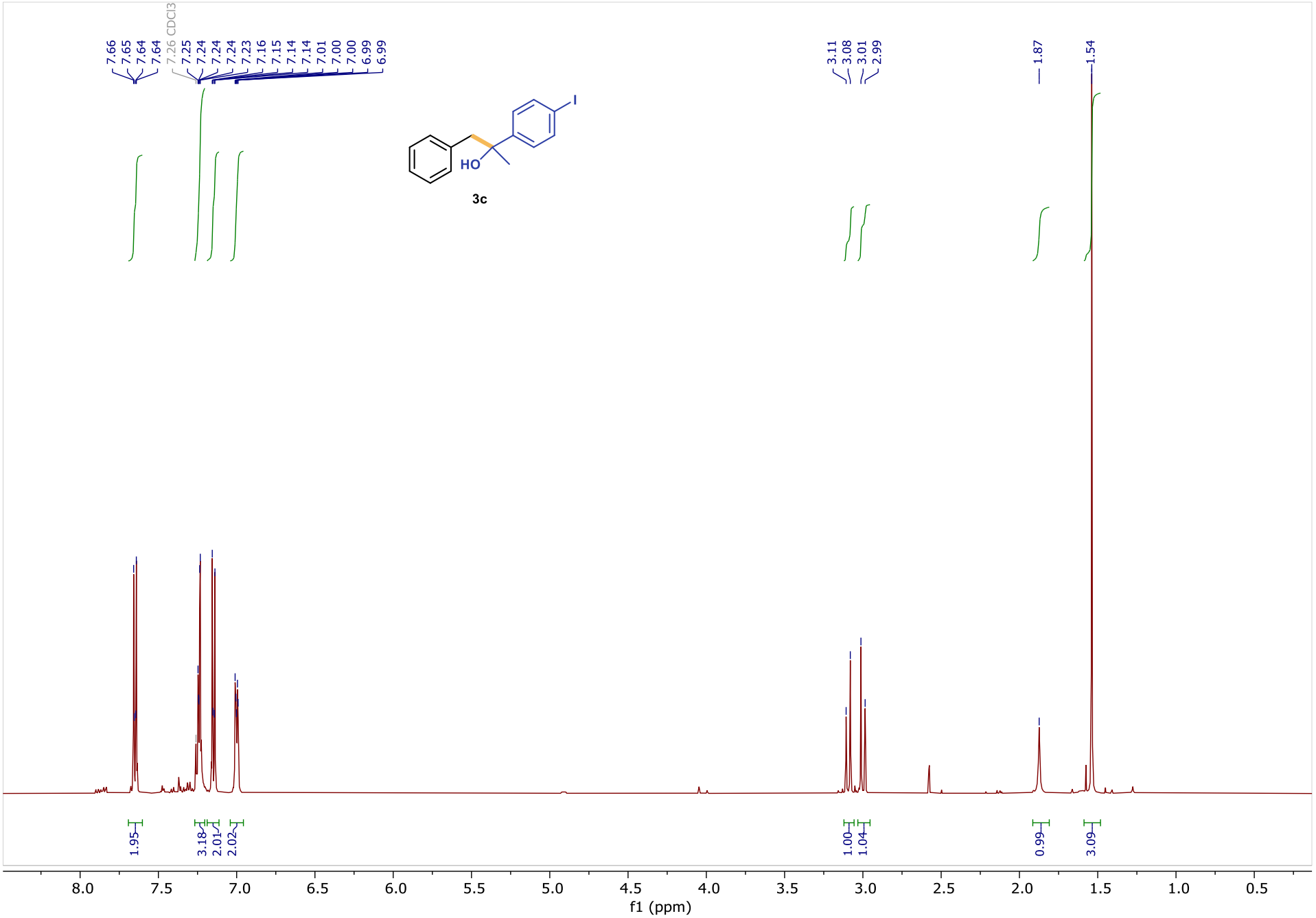


^{1}H NMR of 3c, $CDCl_3$, 500 MHz



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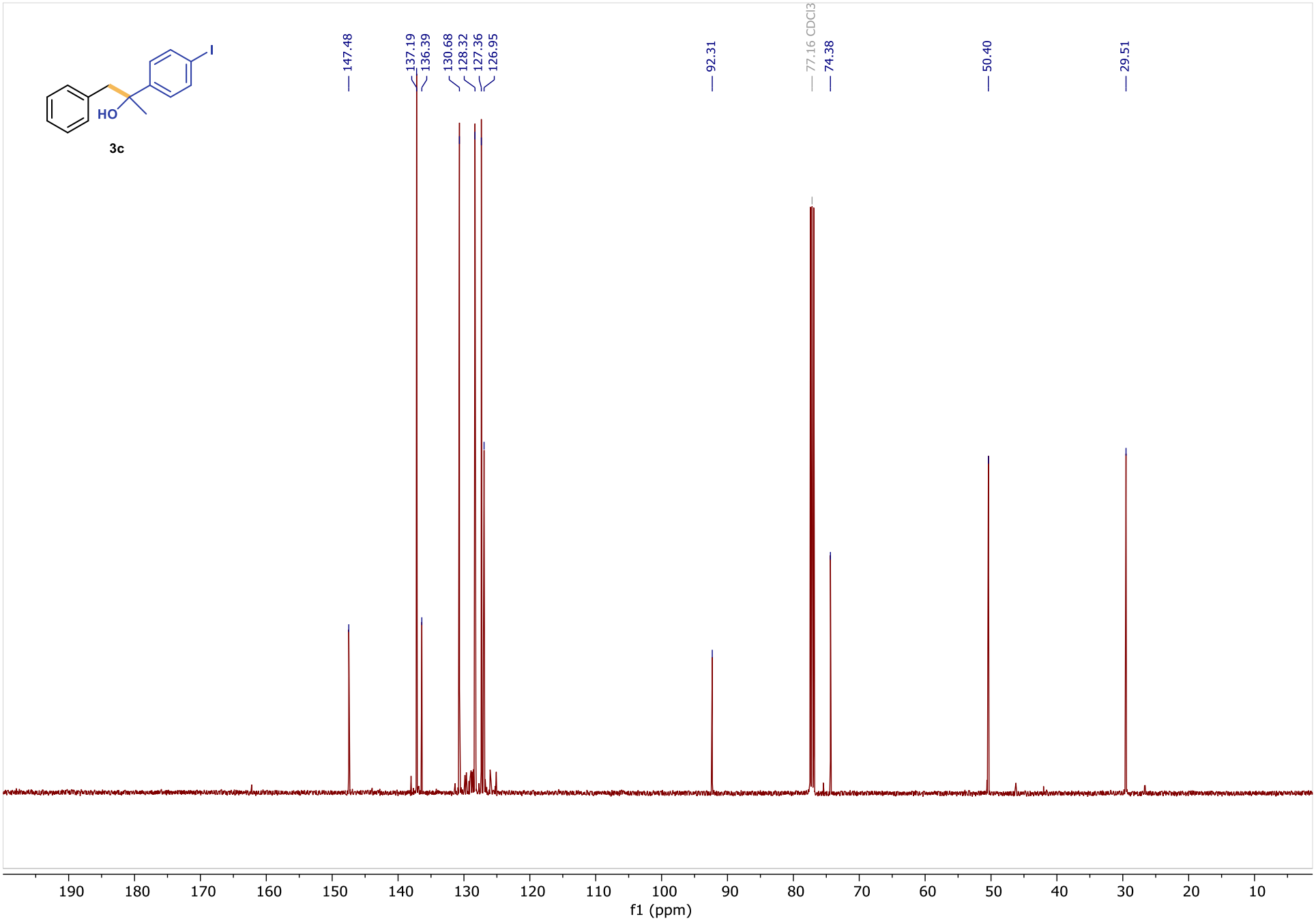
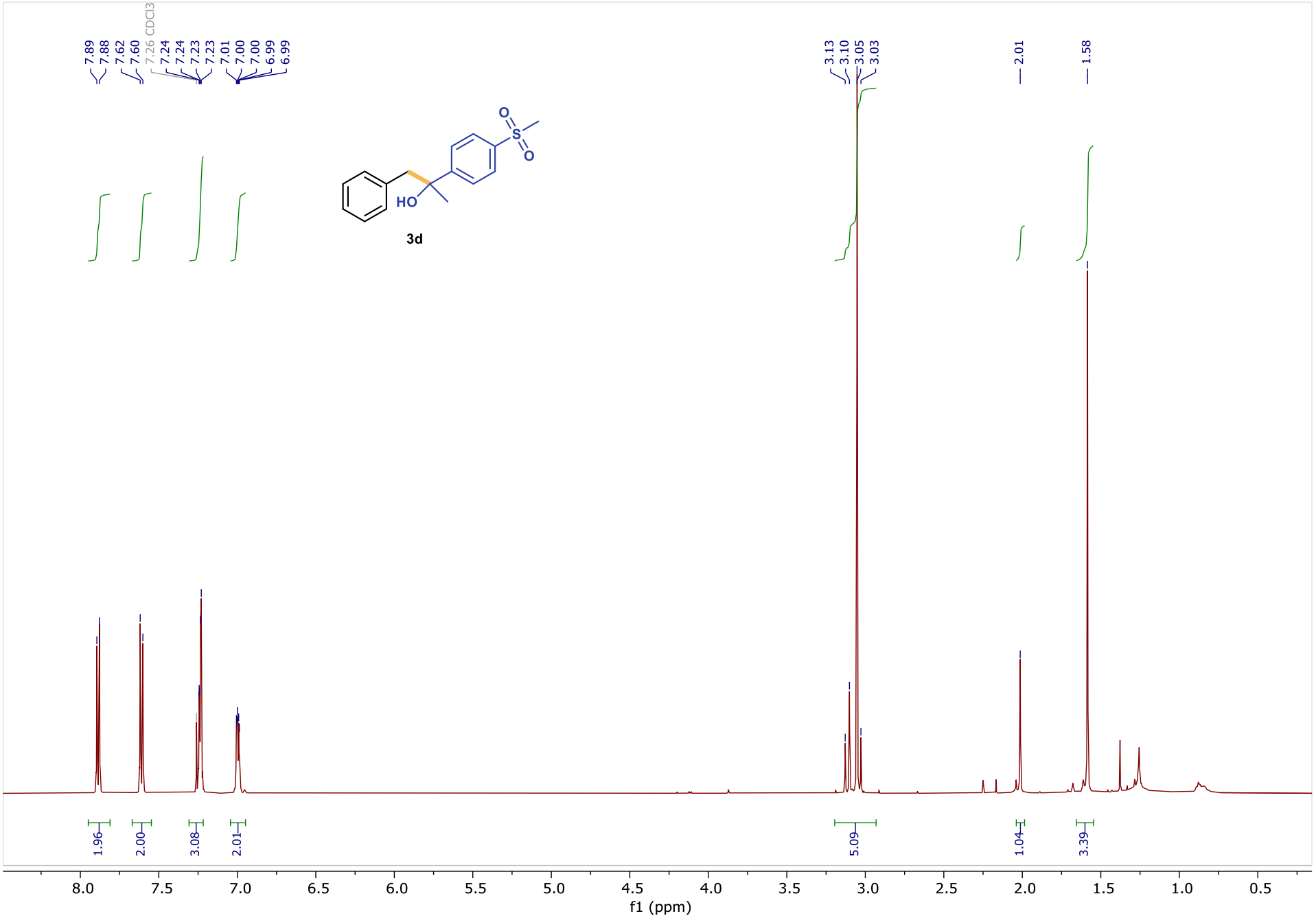
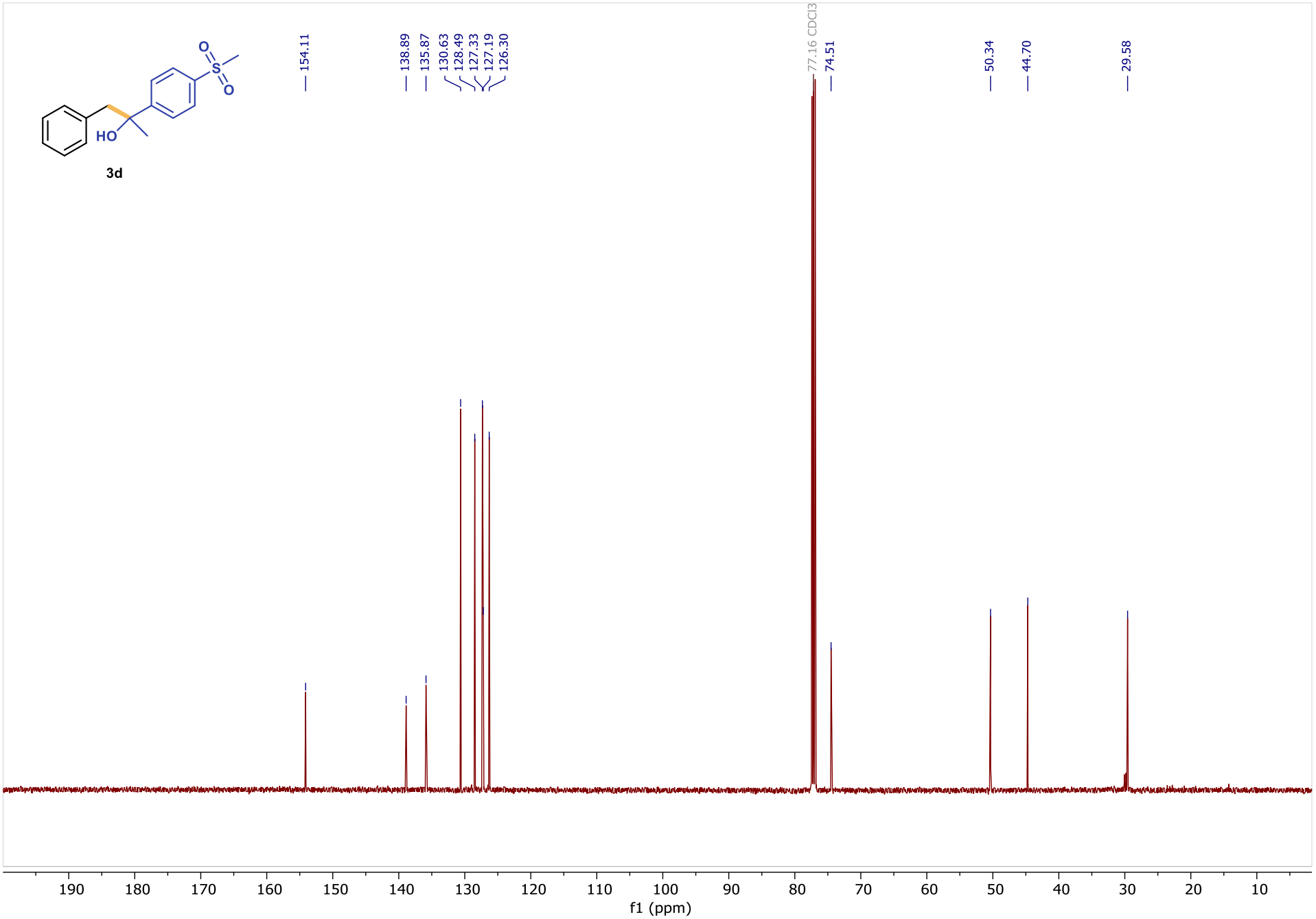


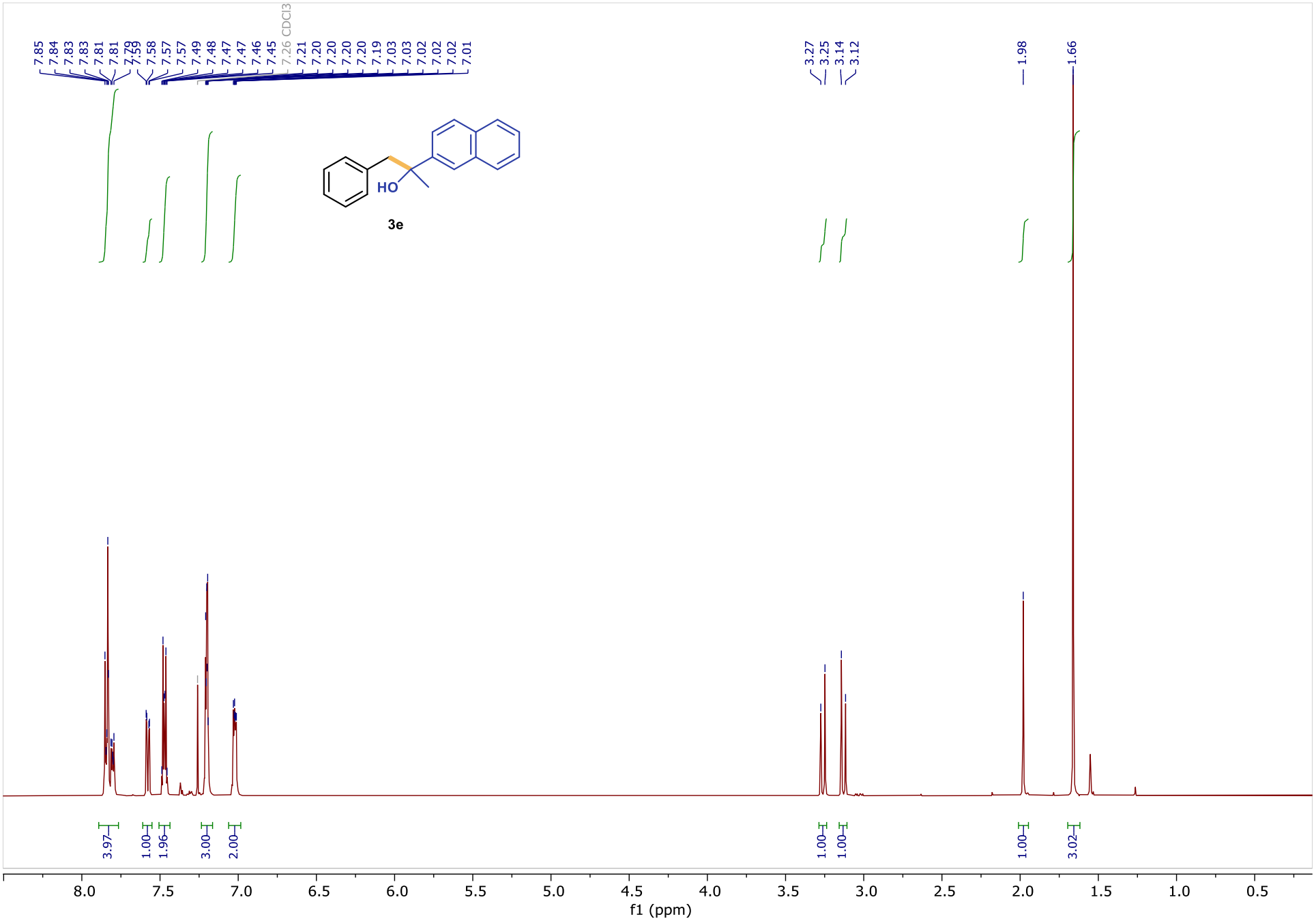
^{1}H NMR of 3d, $CDCl_3$, 500 MHz



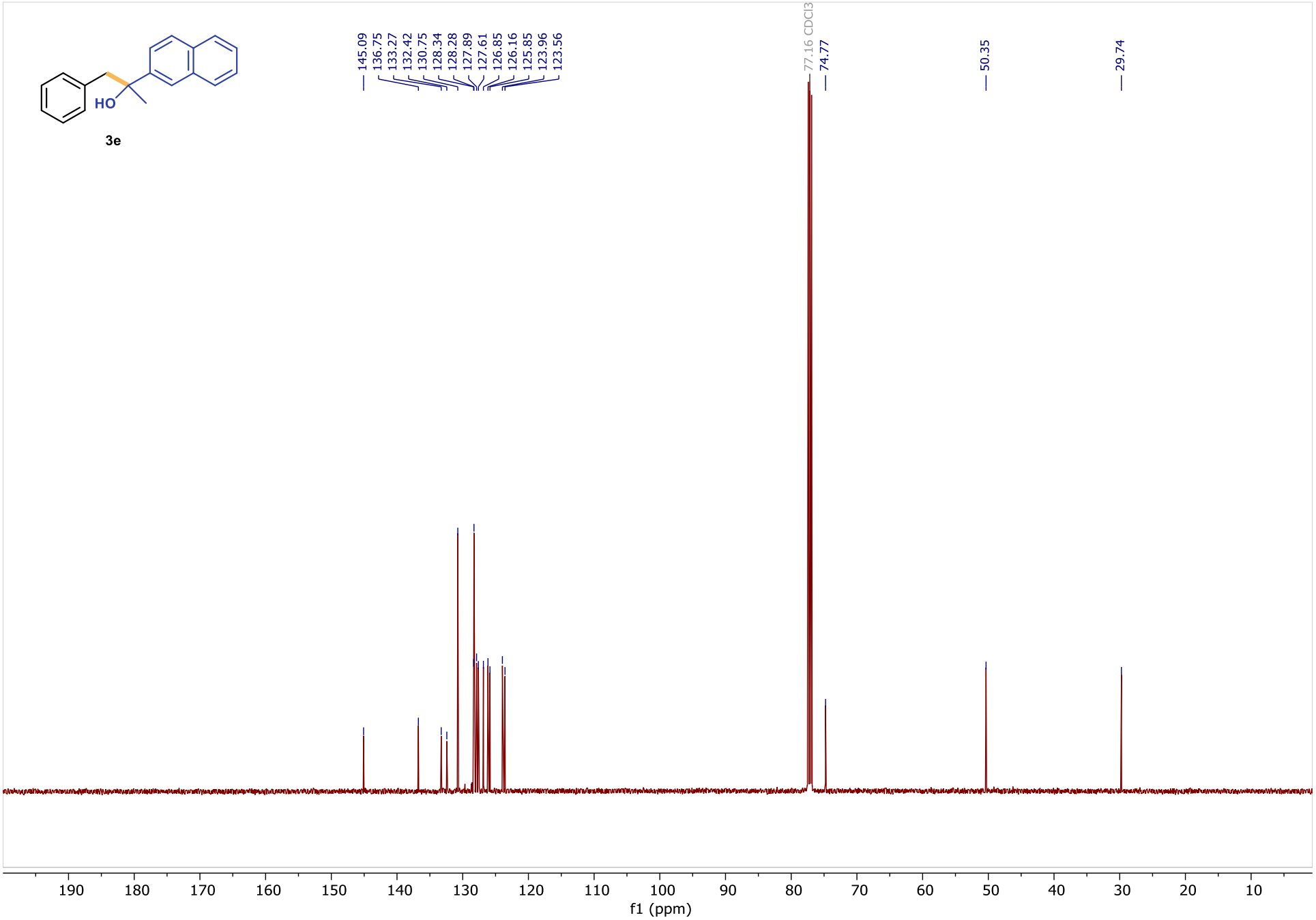
^{13}C NMR of 3d, $CDCl_3$, 126 MHz



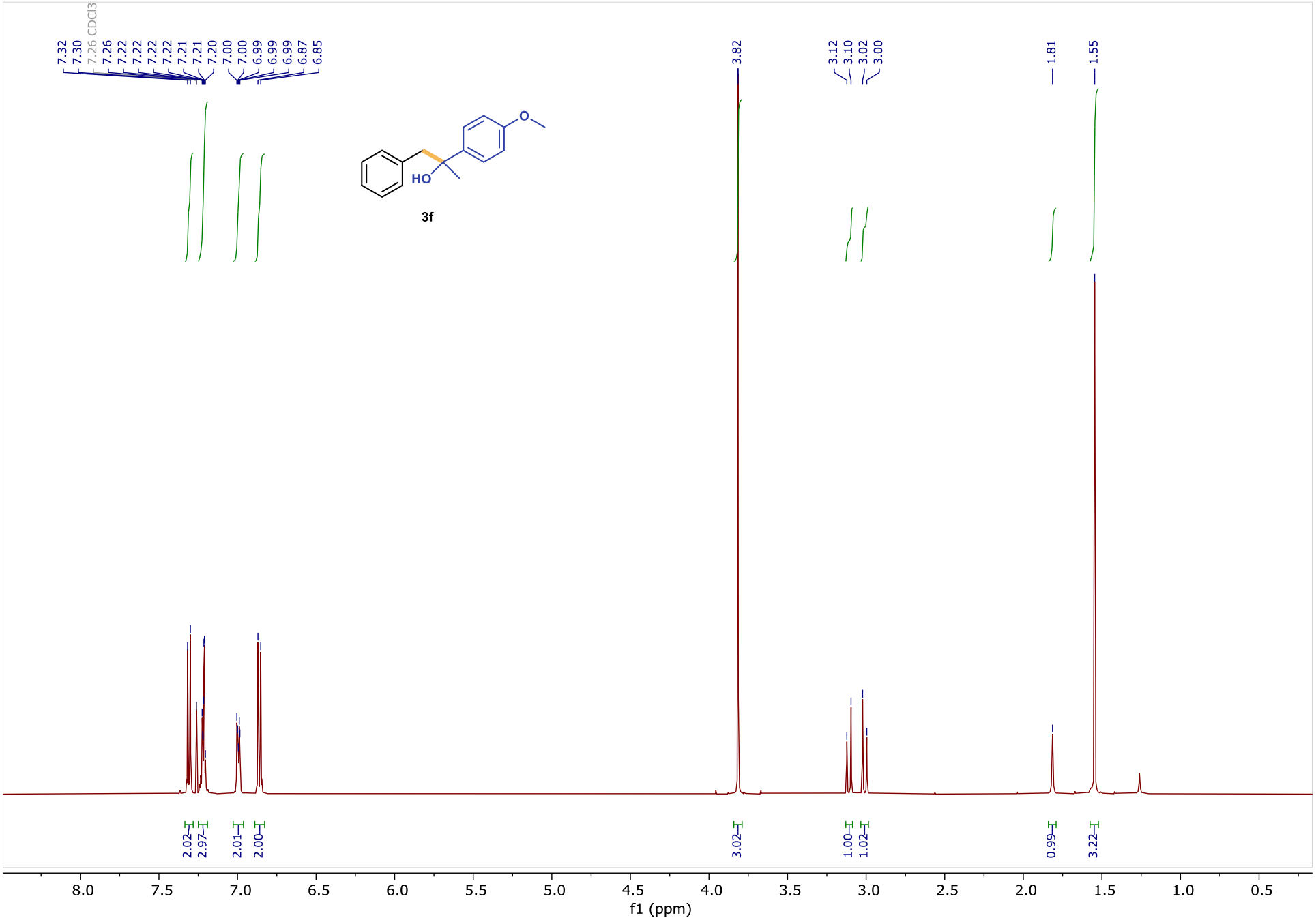
^{1}H NMR of 3e, $CDCl_3$, 500 MHz



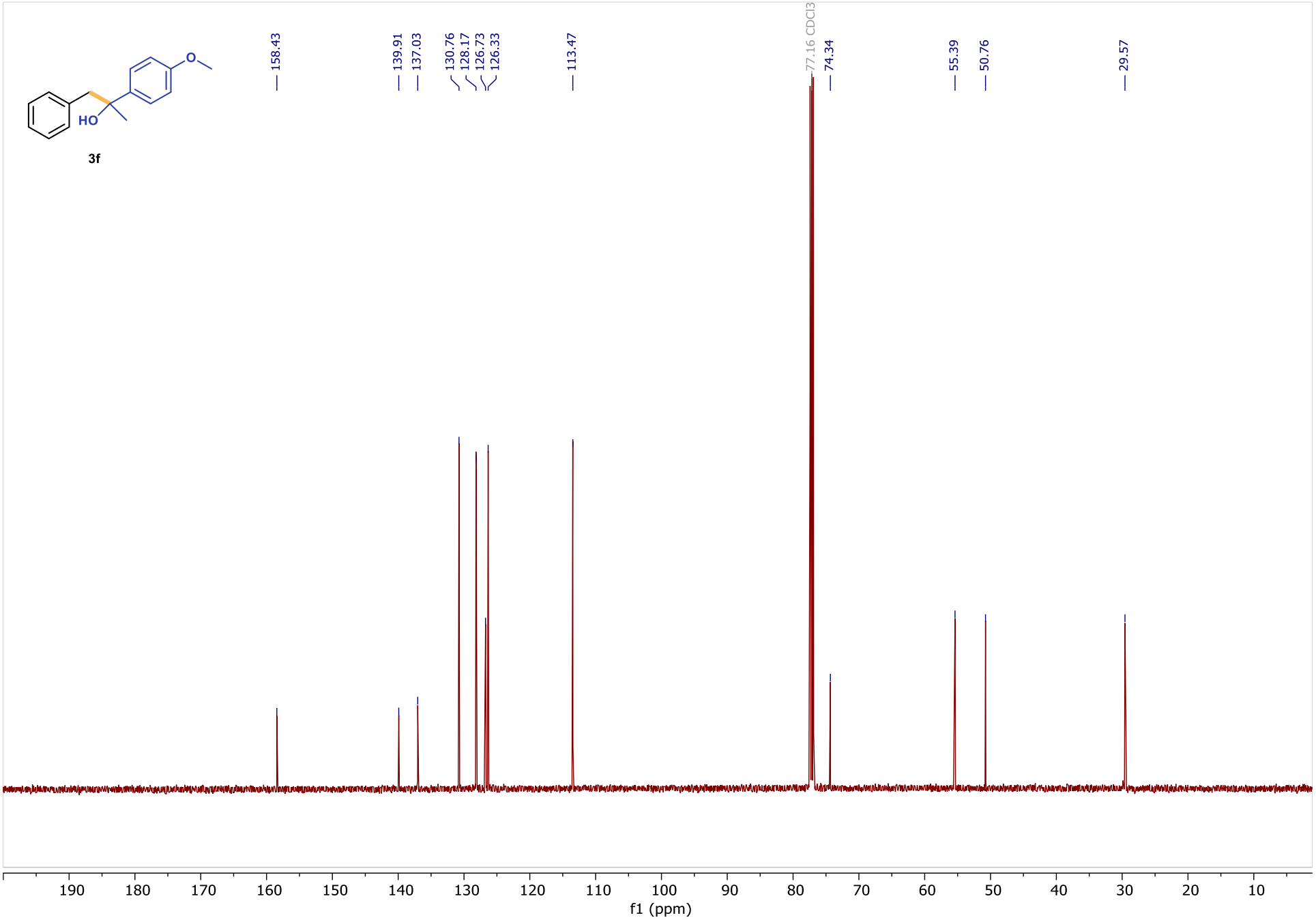
^{13}C NMR of 3e, $CDCl_3$, 126 MHz



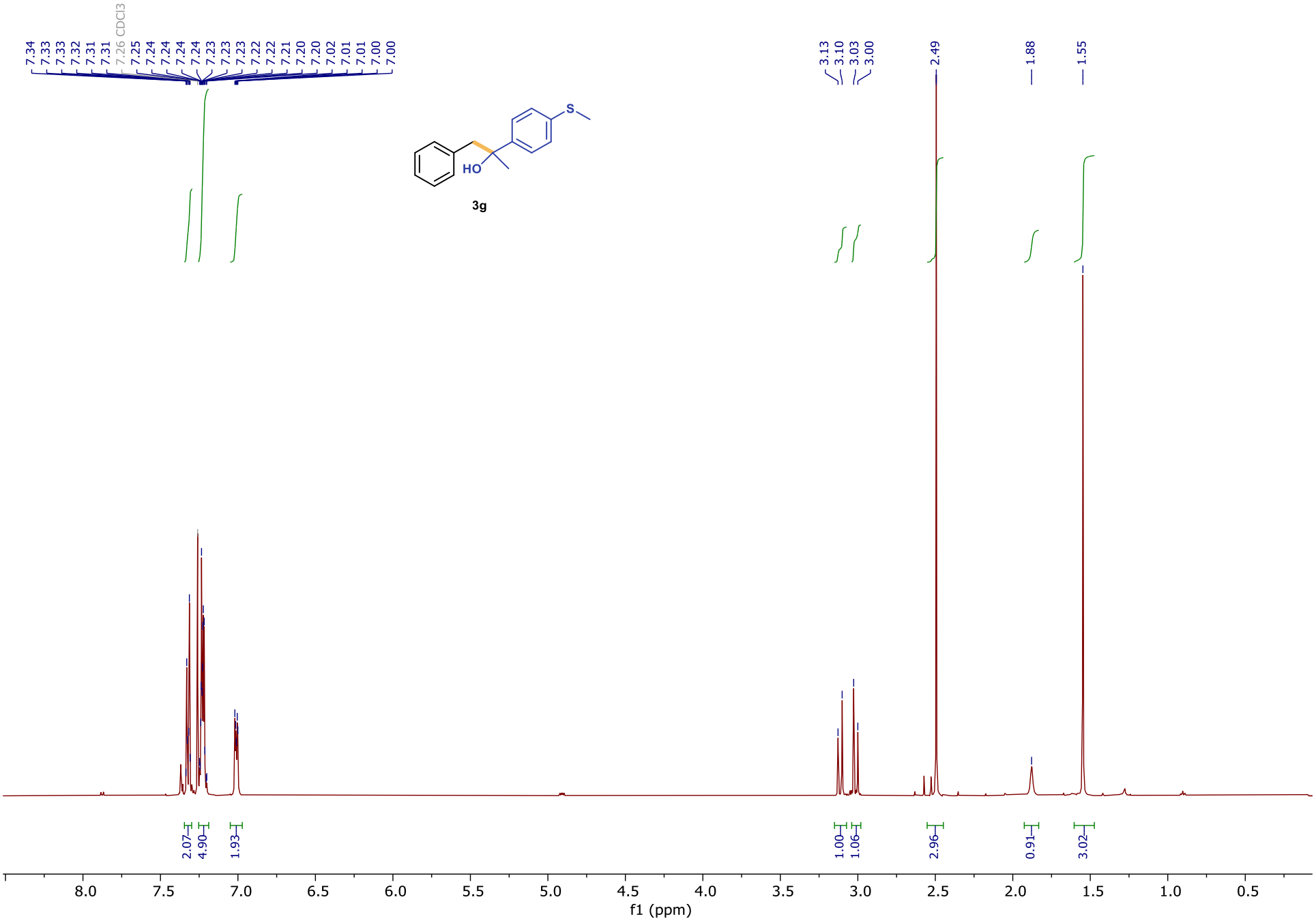
^{1}H NMR of 3f, $CDCl_3$, 500 MHz



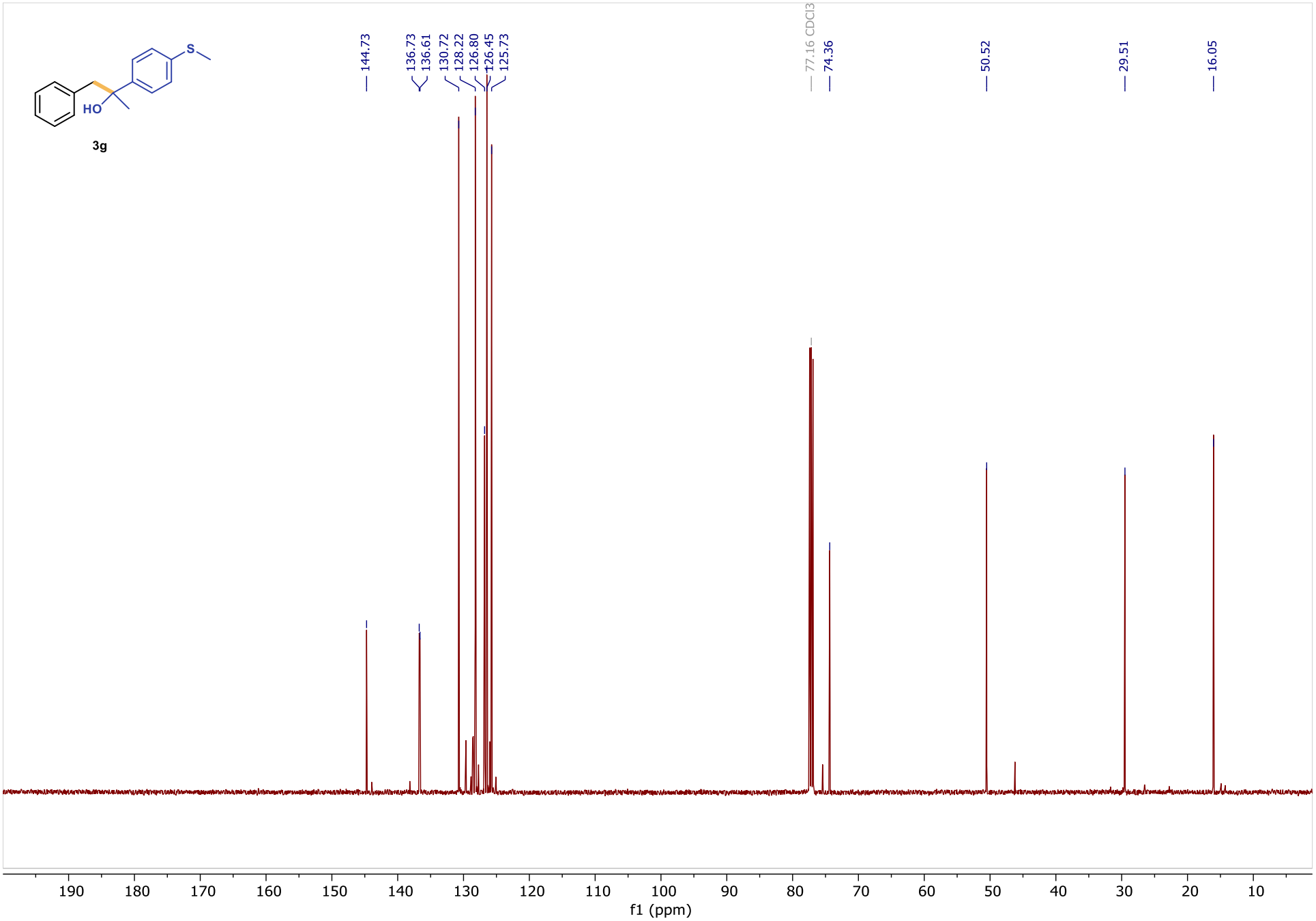
^{13}C NMR of 3f, $CDCl_3$, 126 MHz



^{1}H NMR of 3g, $CDCl_3$, 500 MHz



^{13}C NMR of 3g, $CDCl_3$, 126 MHz



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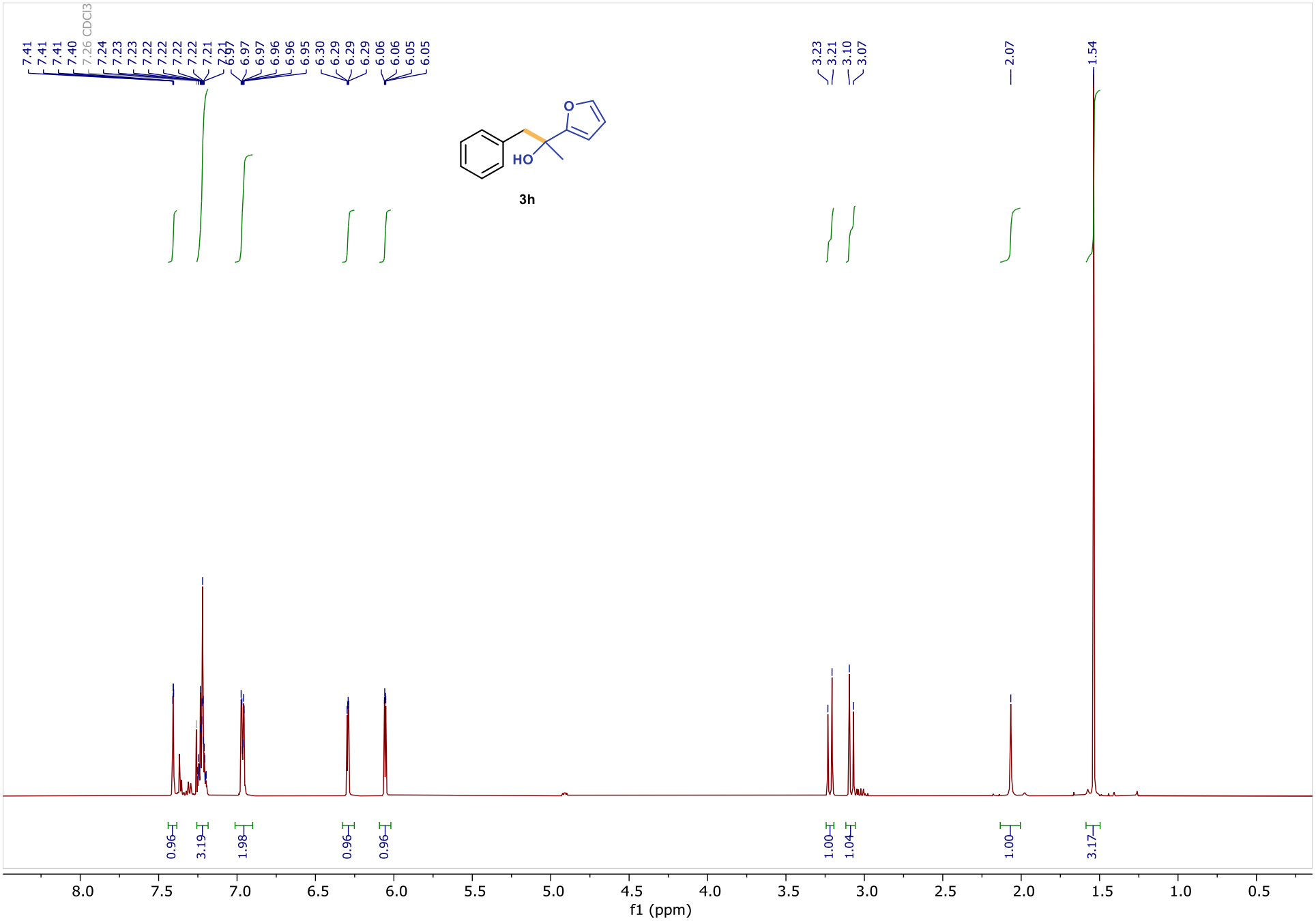
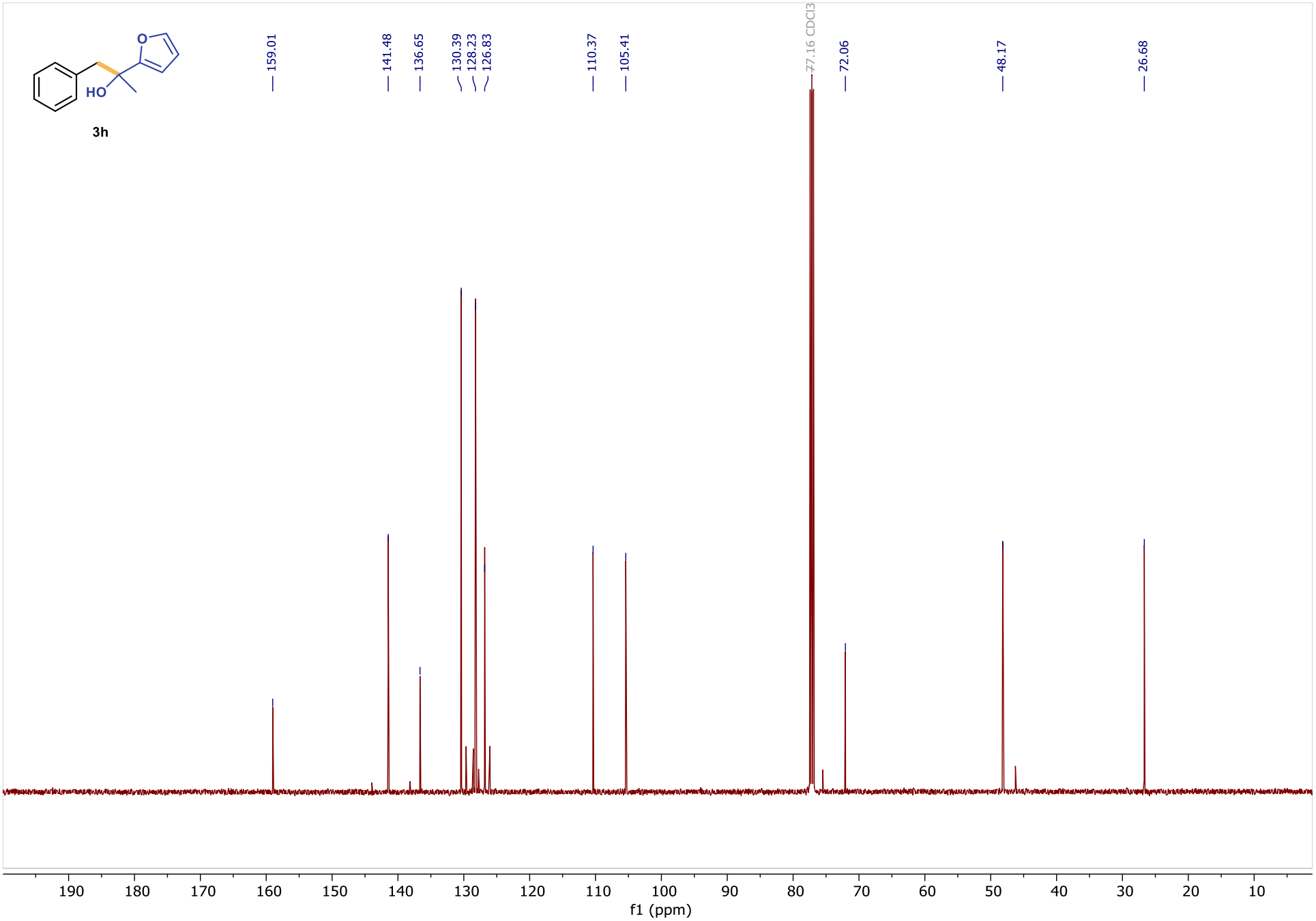
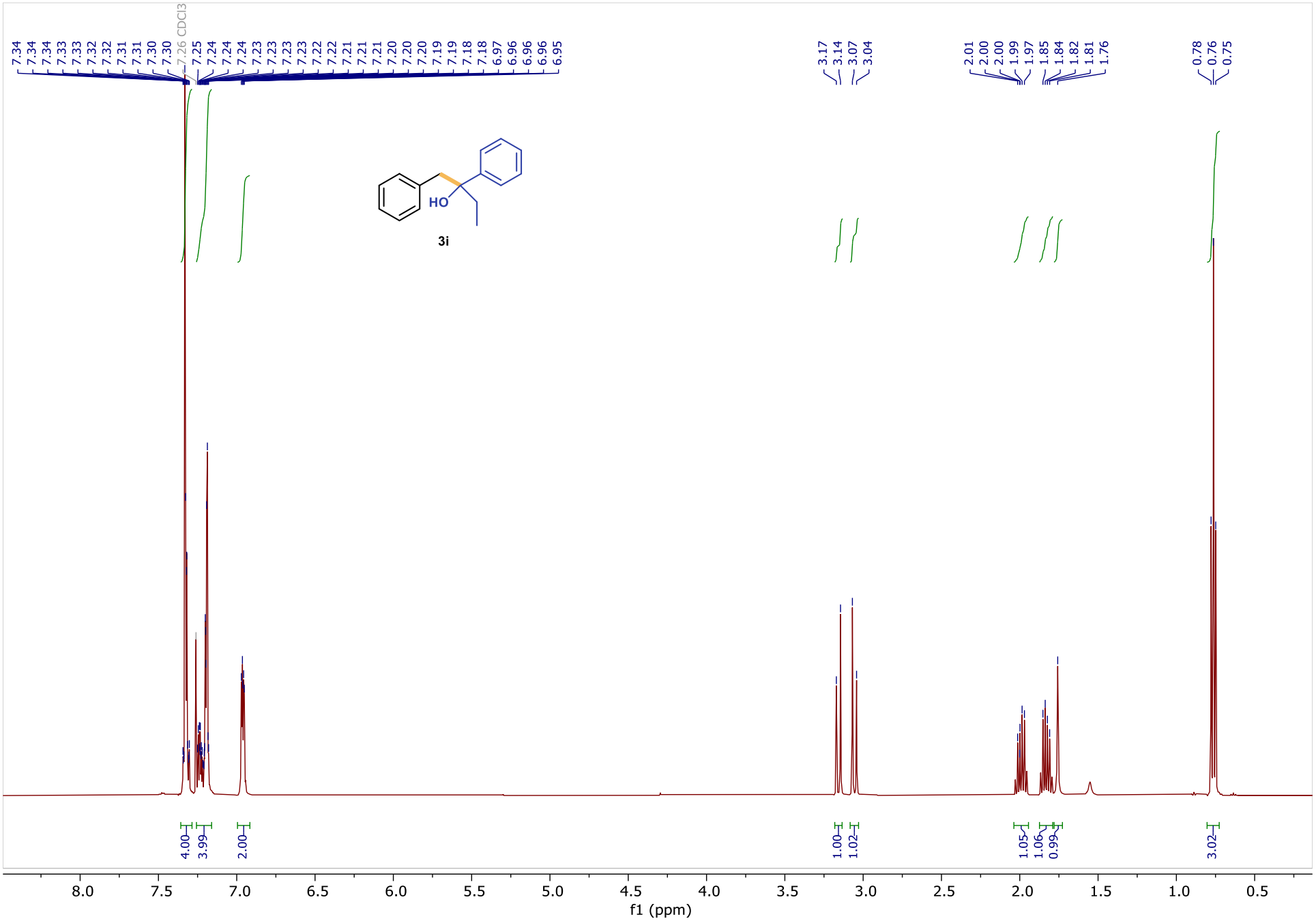


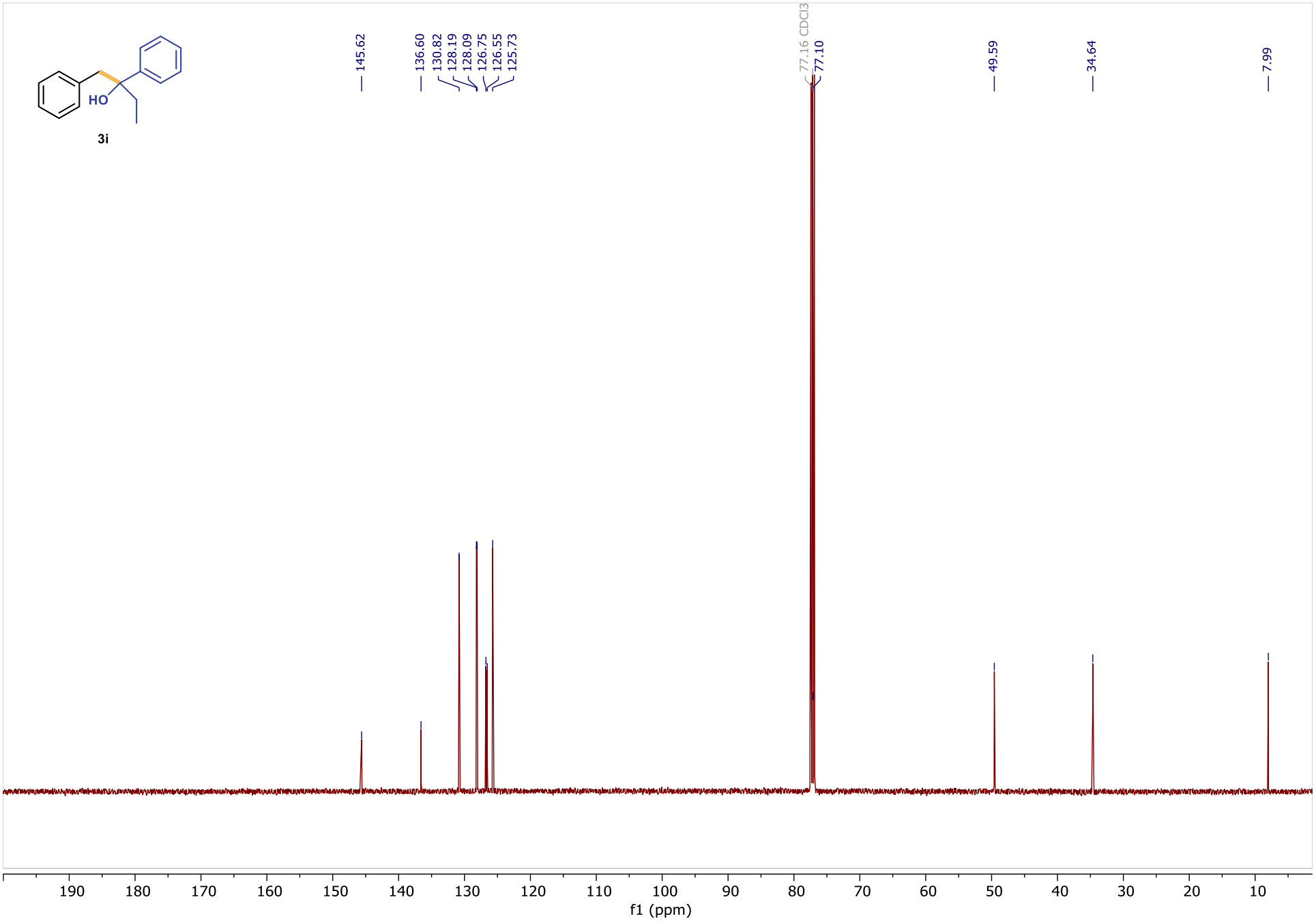
^{13}C NMR of 3h, $CDCl_3$, 126 MHz



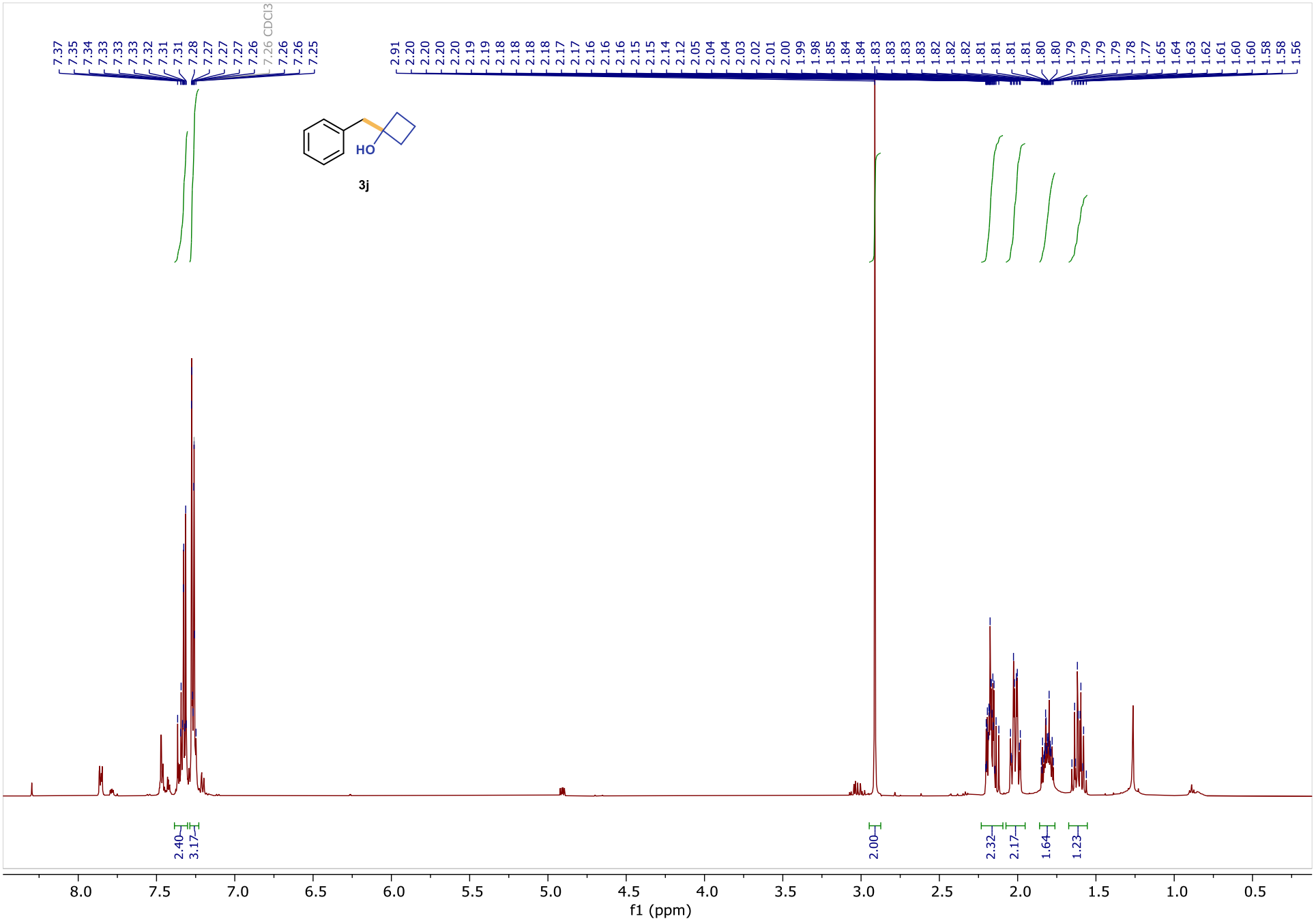
^{1}H NMR of 3i, $CDCl_3$, 500 MHz



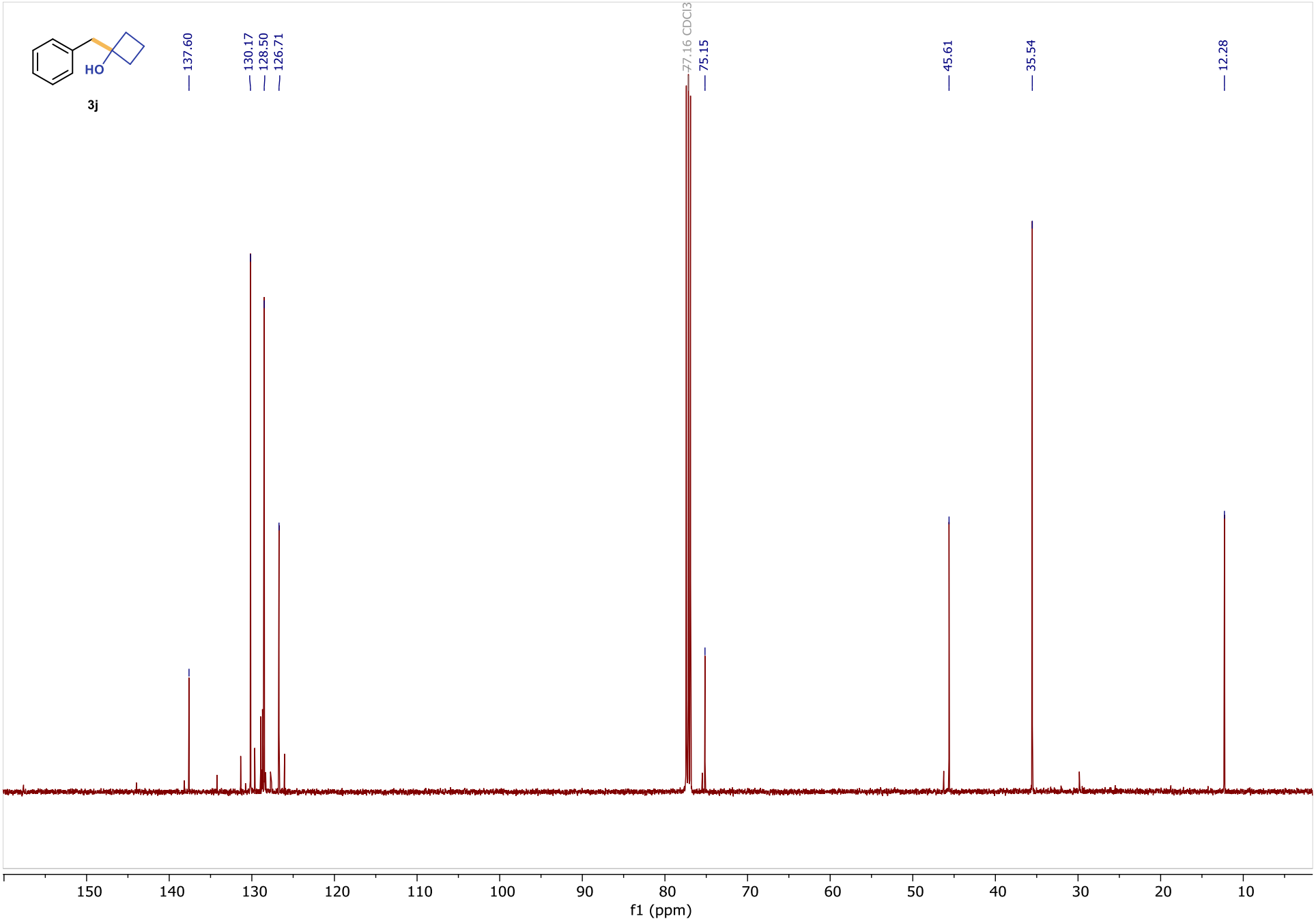
^{13}C NMR of 3i, $CDCl_3$, 126 MHz



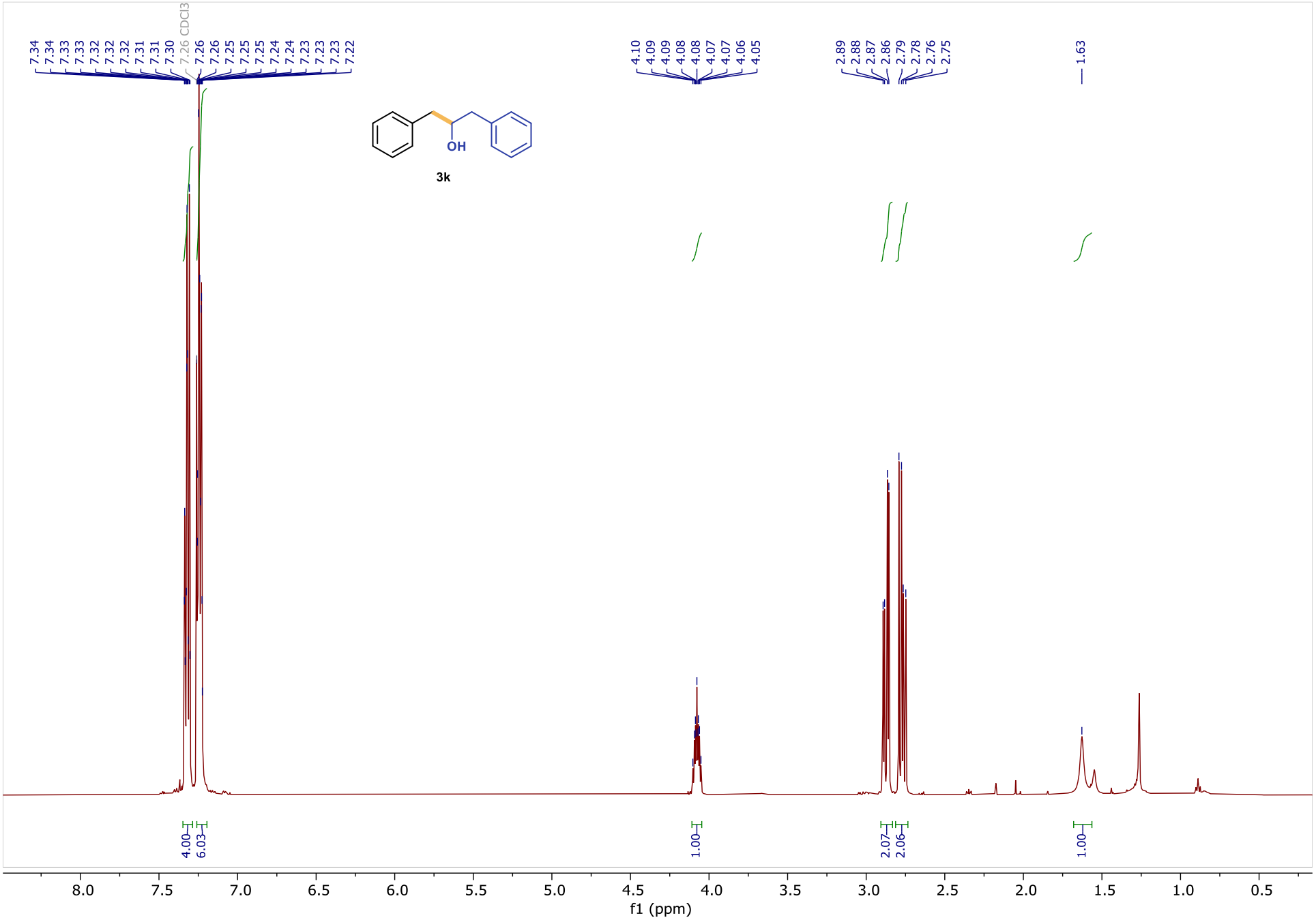
^{1}H NMR of 3j, $CDCl_3$, 500 MHz



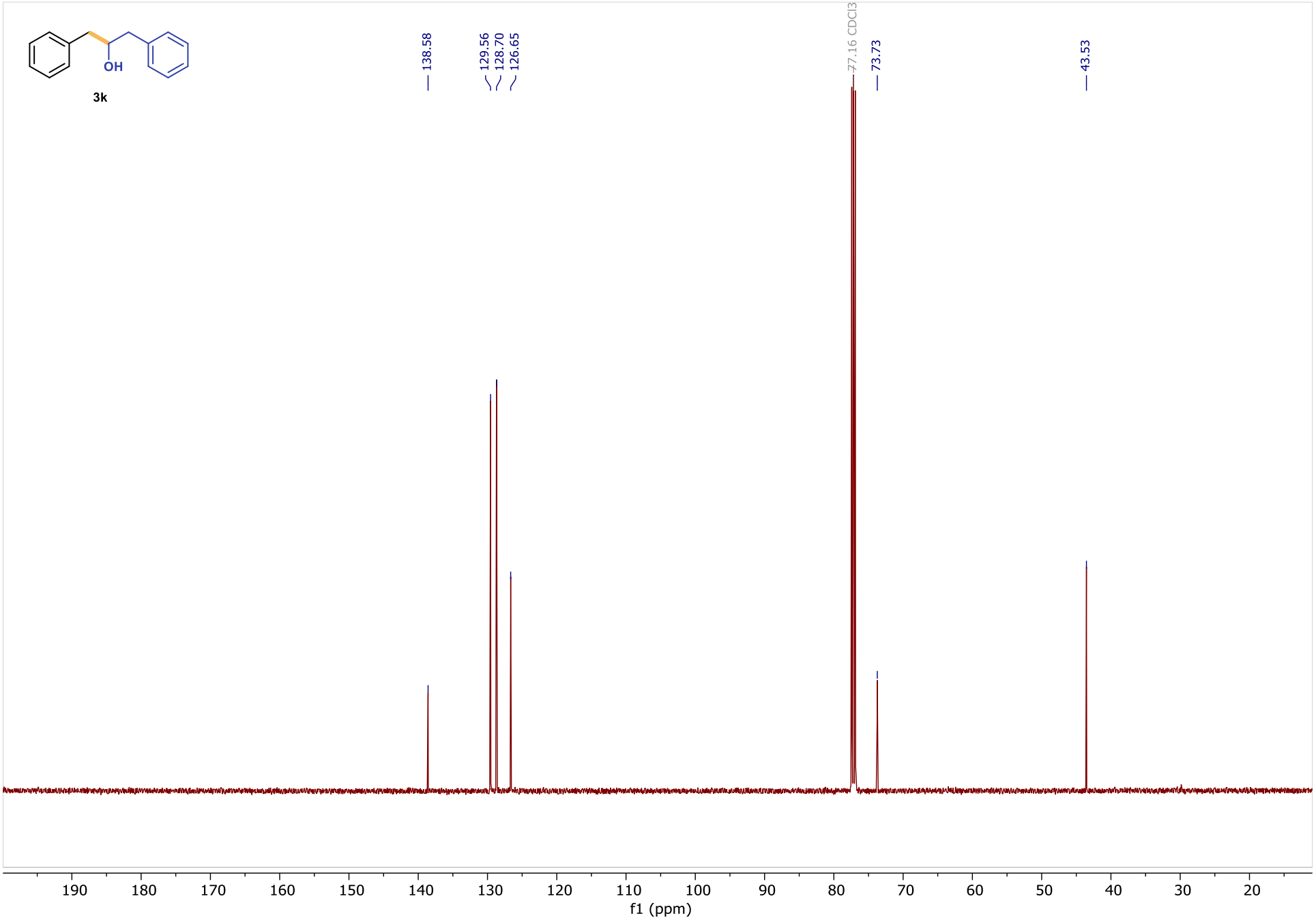
^{13}C NMR of 3j, $CDCl_3$, 126 MHz



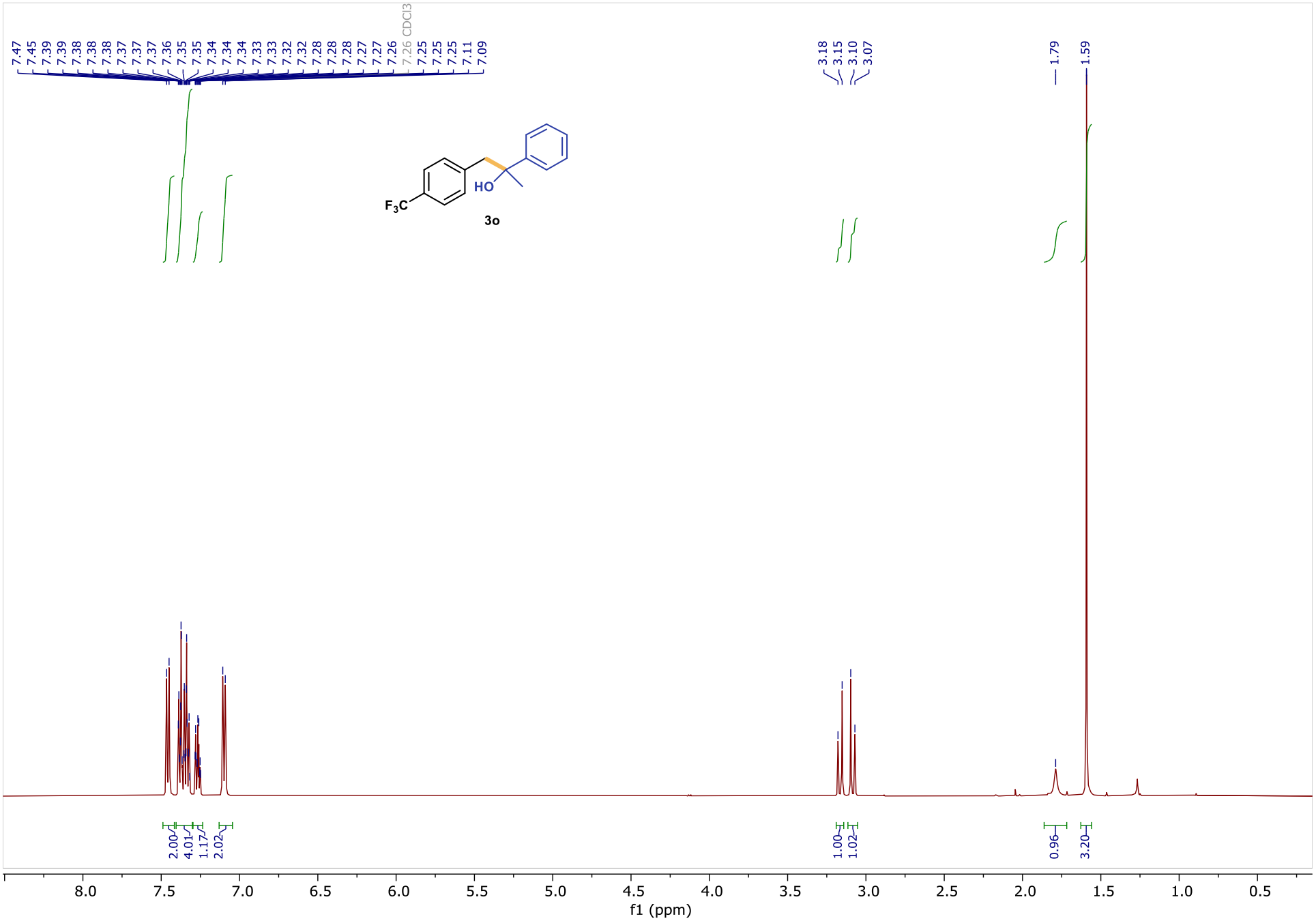
^{1}H NMR of 3k, $CDCl_3$, 500 MHz



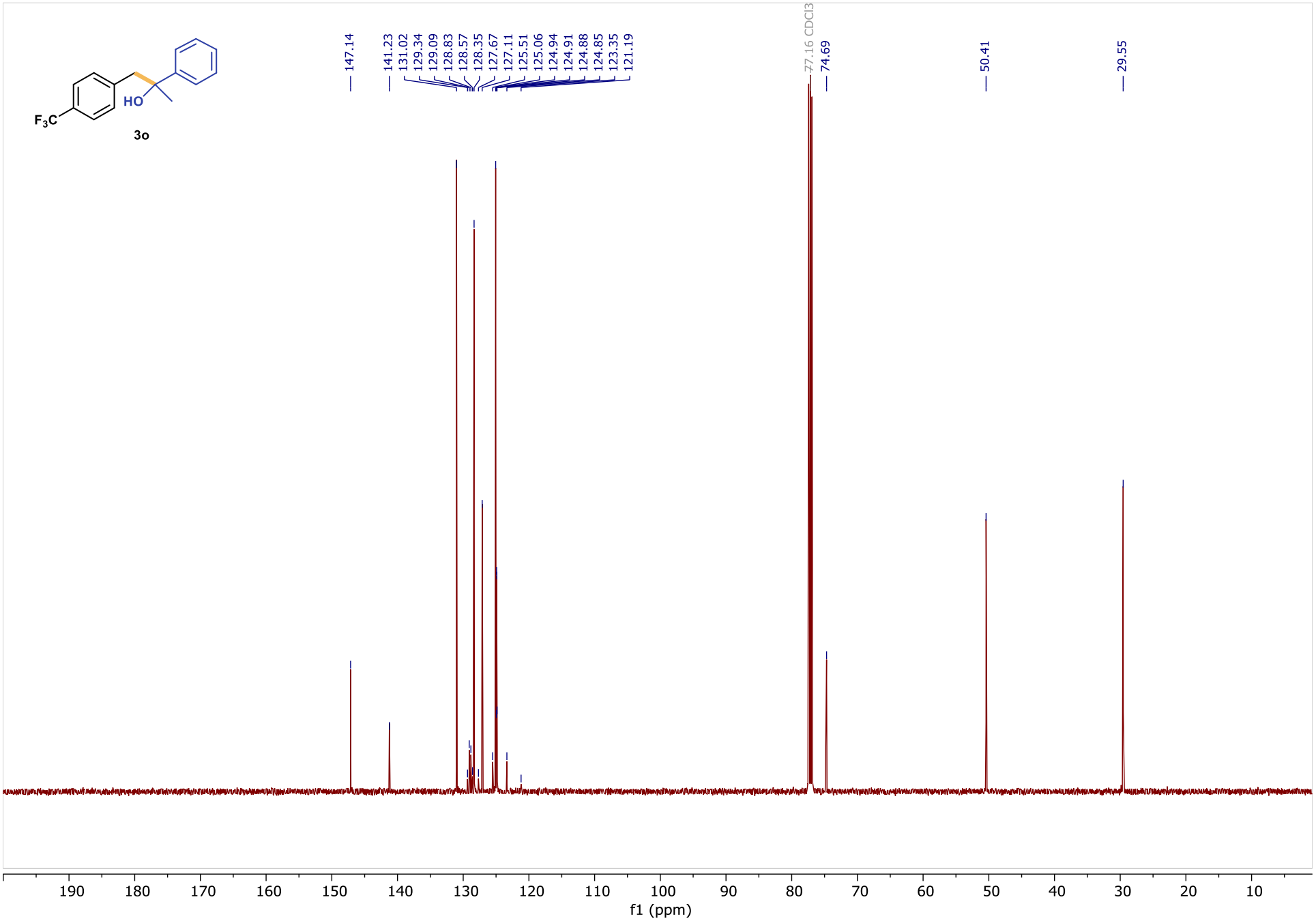
^{13}C NMR of 3k, $CDCl_3$, 126 MHz



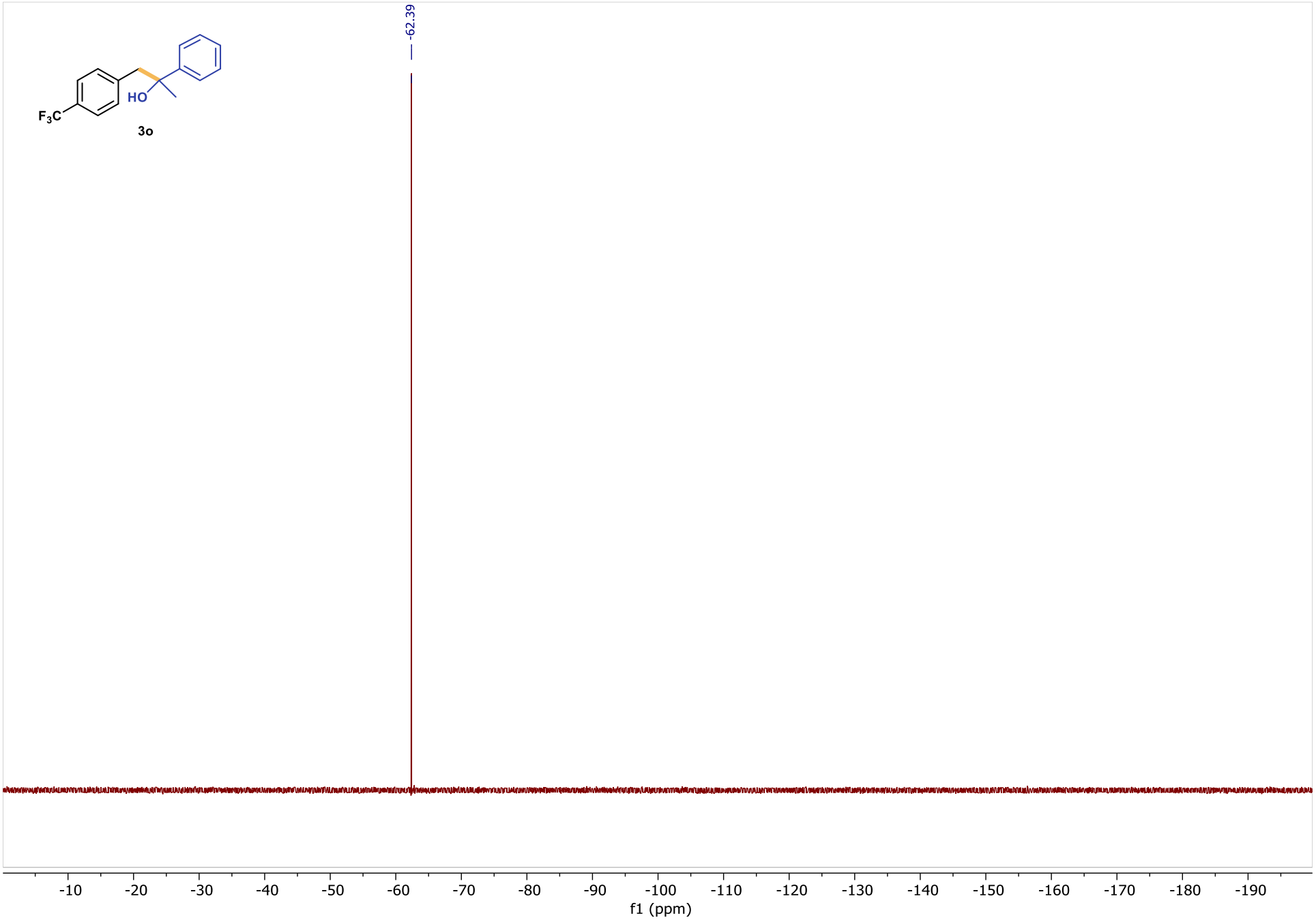
^{1}H NMR of 3o, $CDCl_3$, 500 MHz



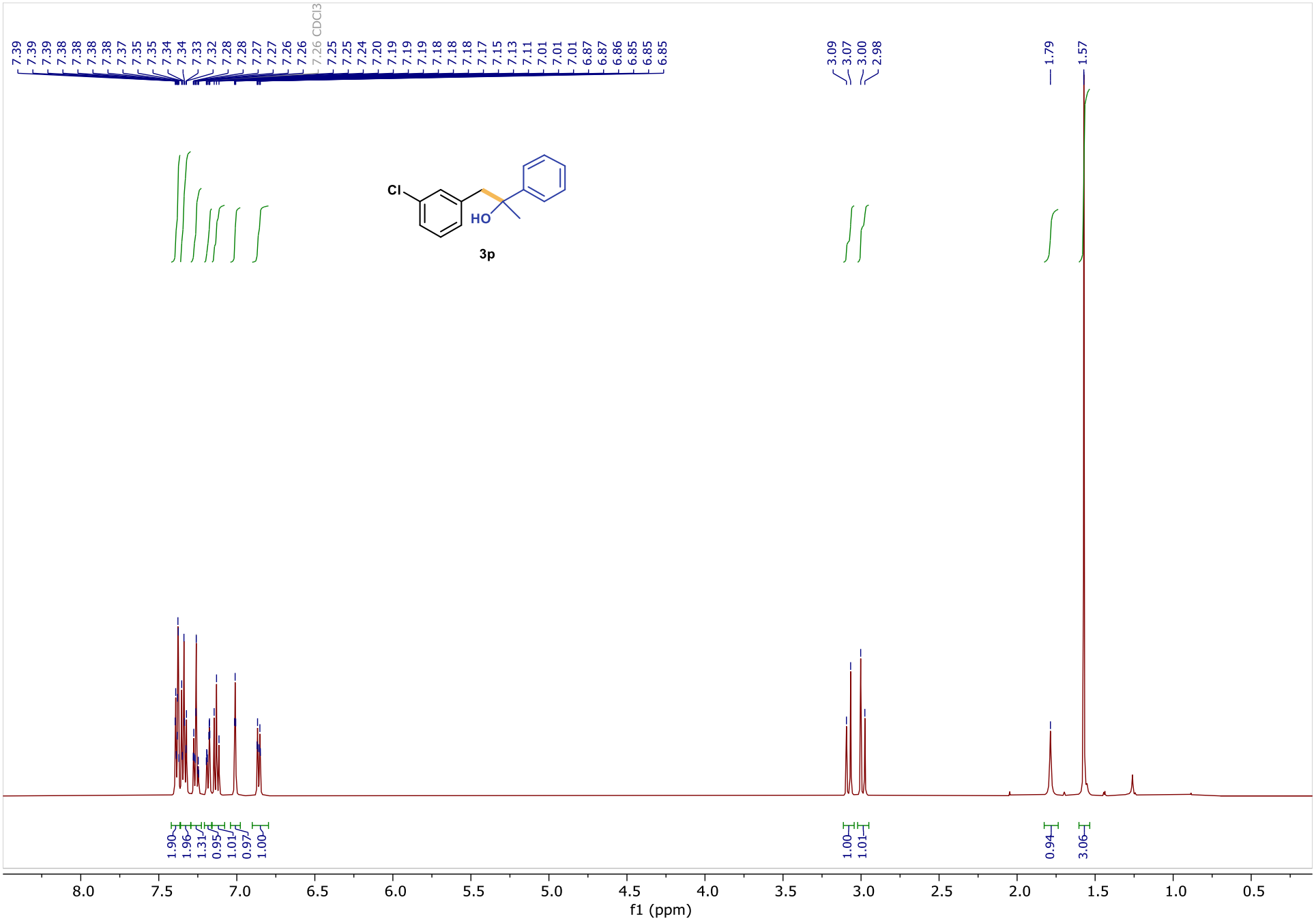
^{13}C NMR of 3o, $CDCl_3$, 126 MHz



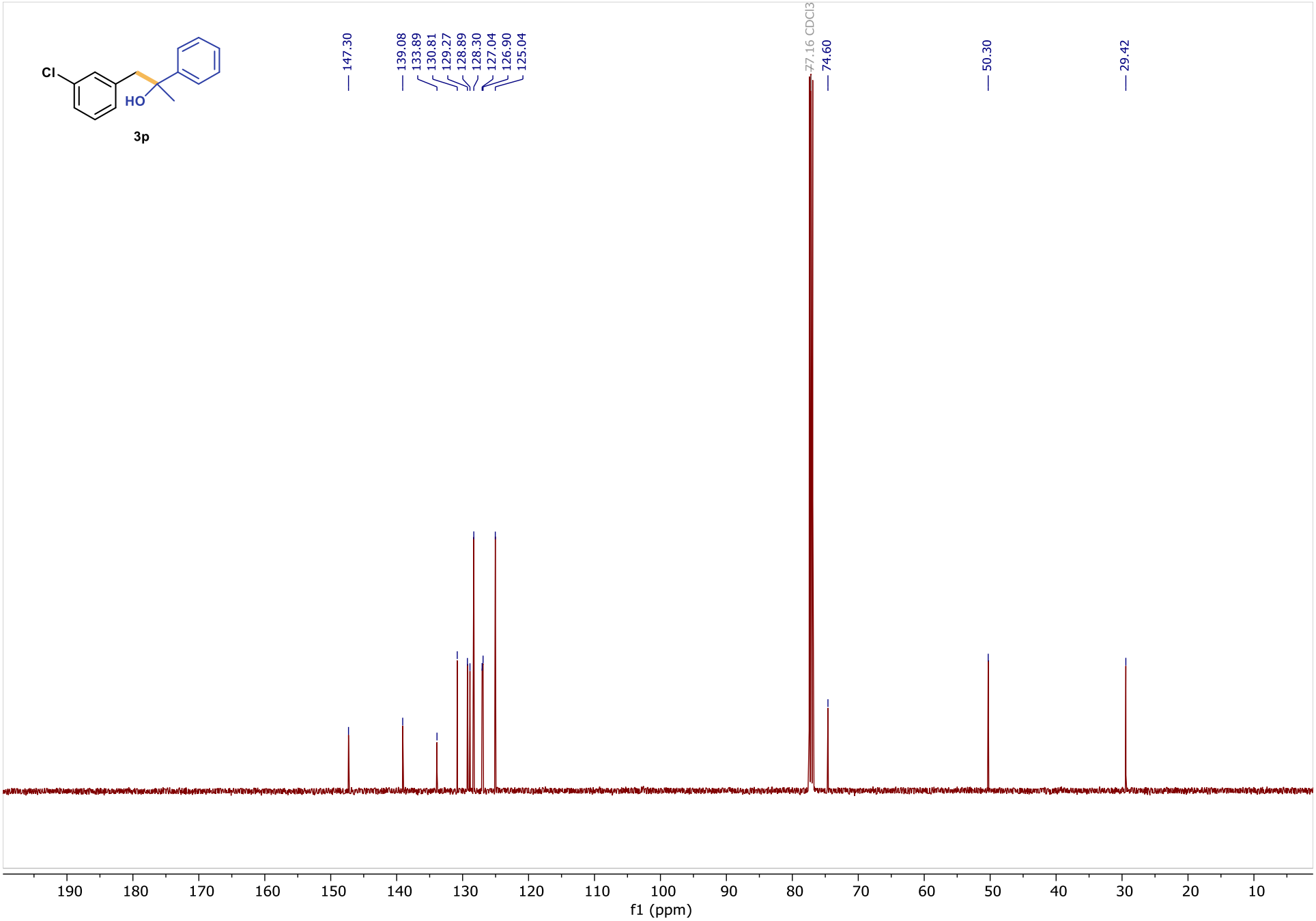
^{19}F NMR of 3o, $CDCl_3$, 471 MHz



^{1}H NMR of 3p, $CDCl_3$, 500 MHz



^{13}C NMR of 3p, $CDCl_3$, 126 MHz



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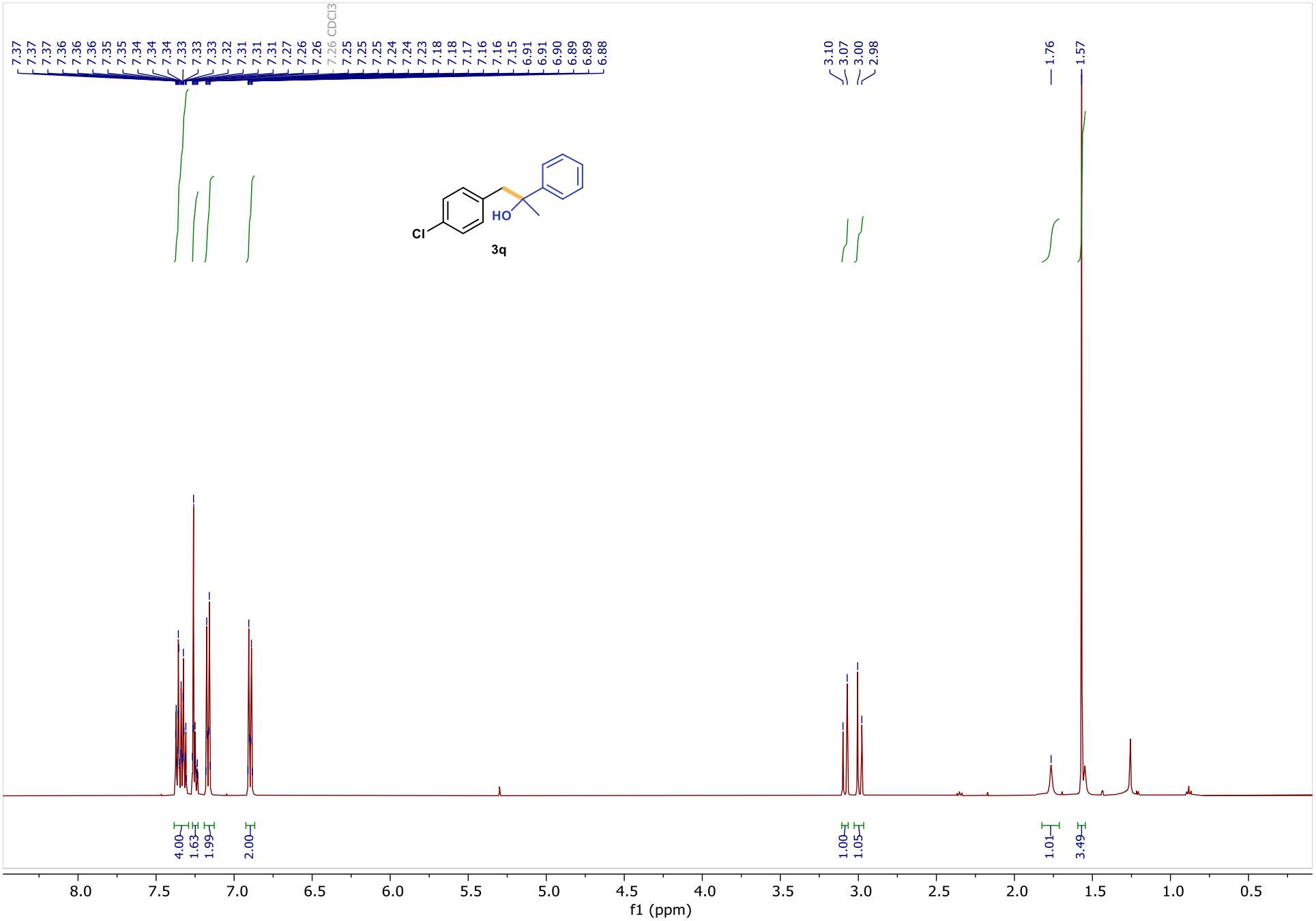
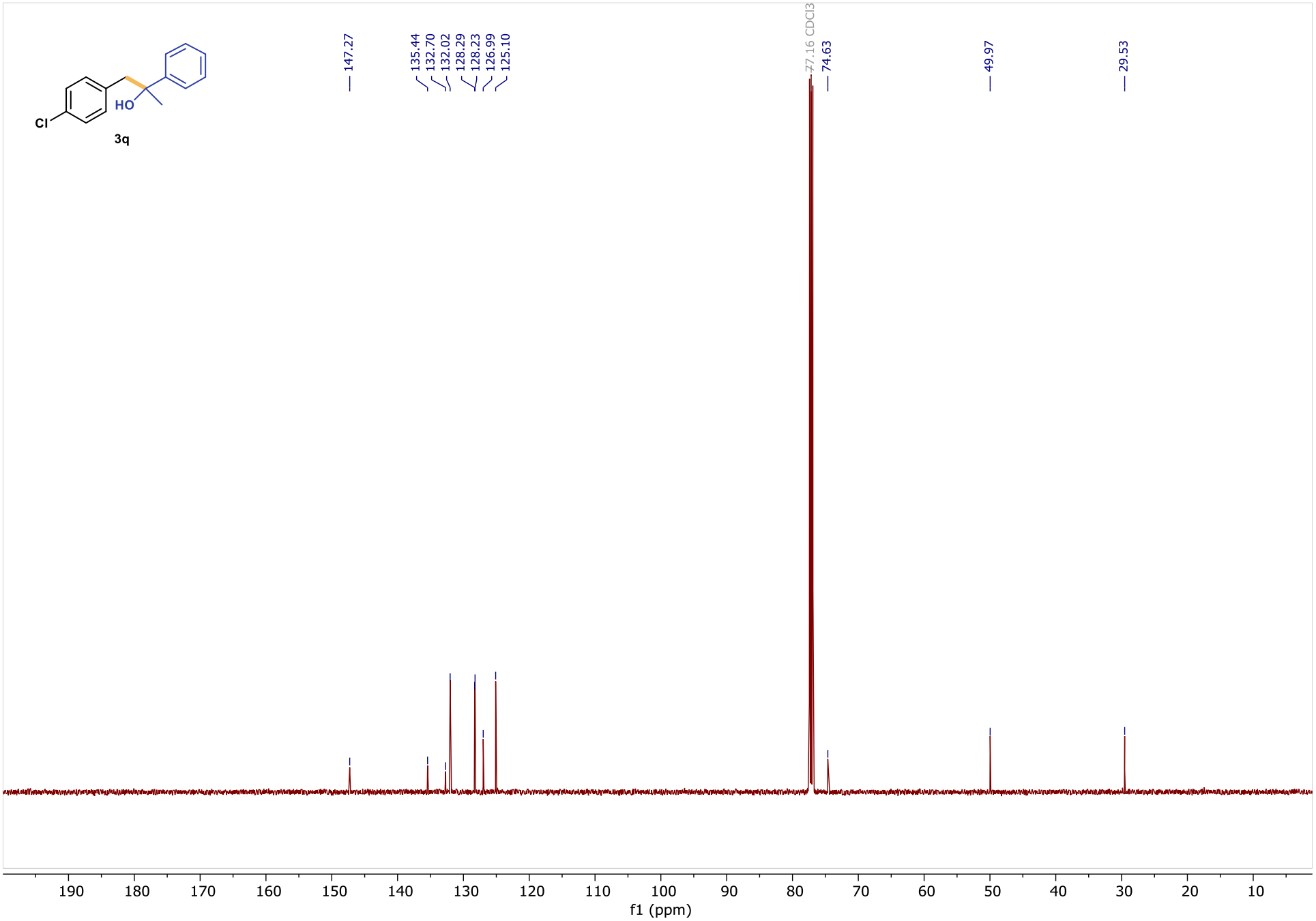
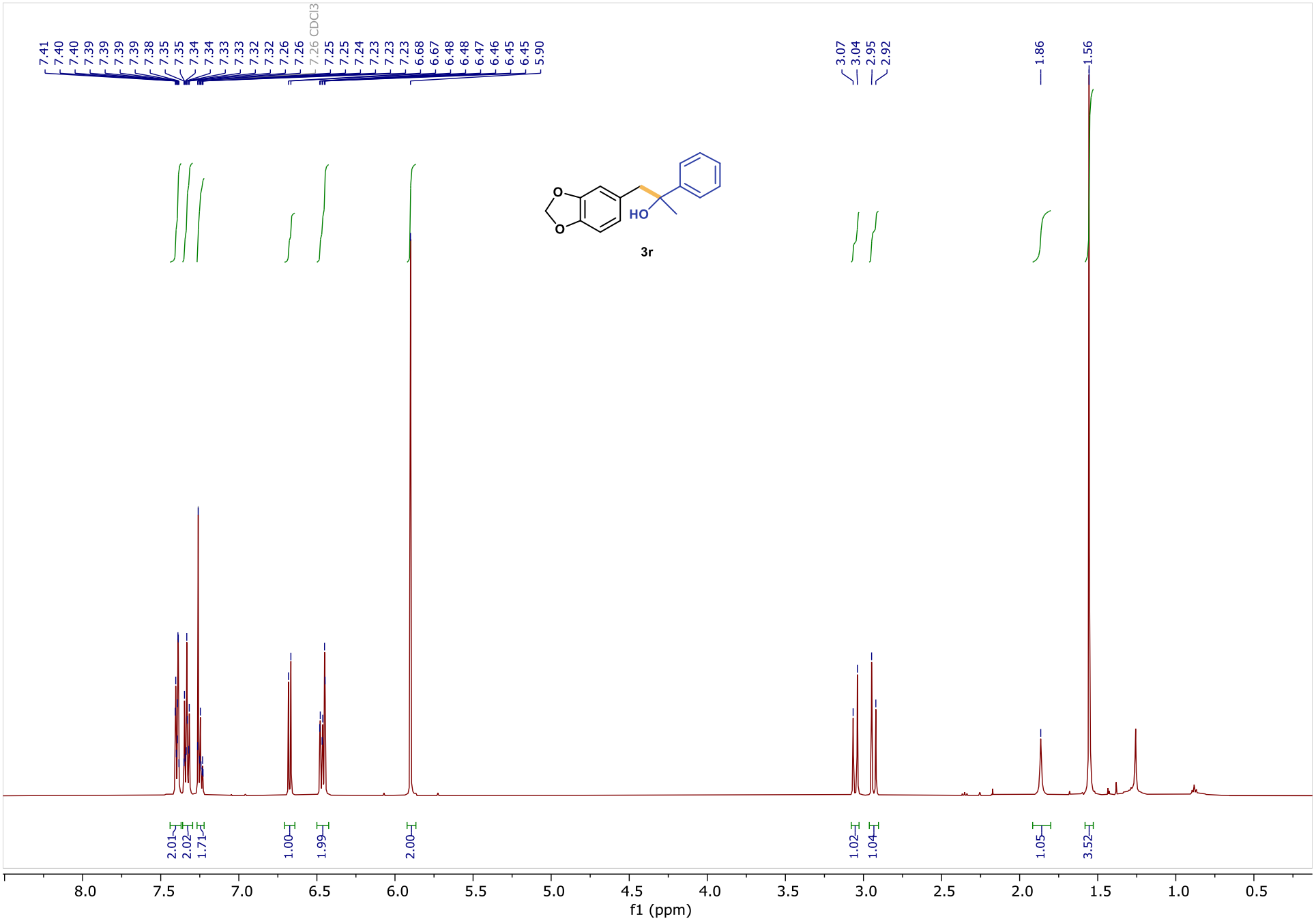


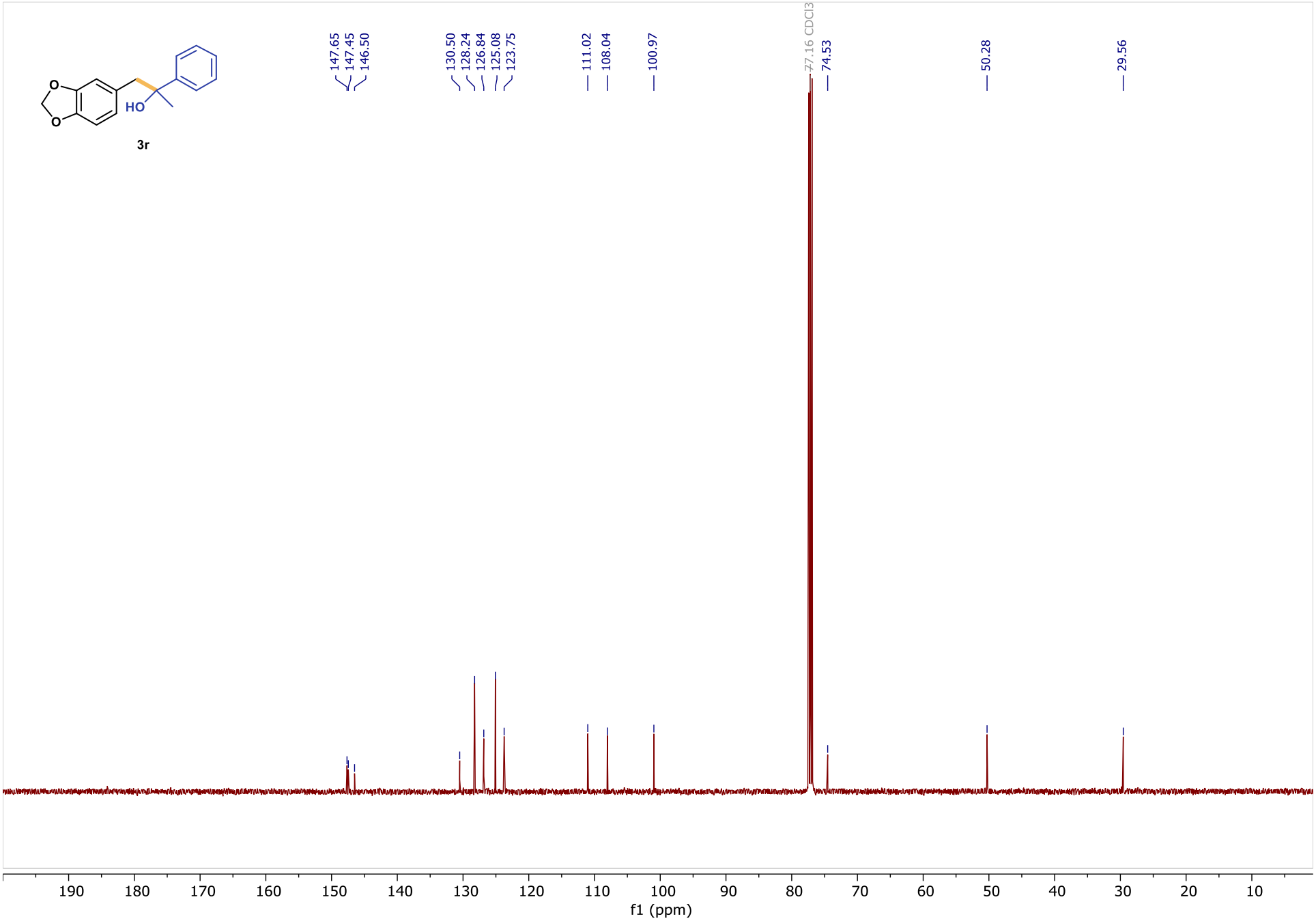
^{13}C NMR of 3q, $CDCl_3$, 126 MHz



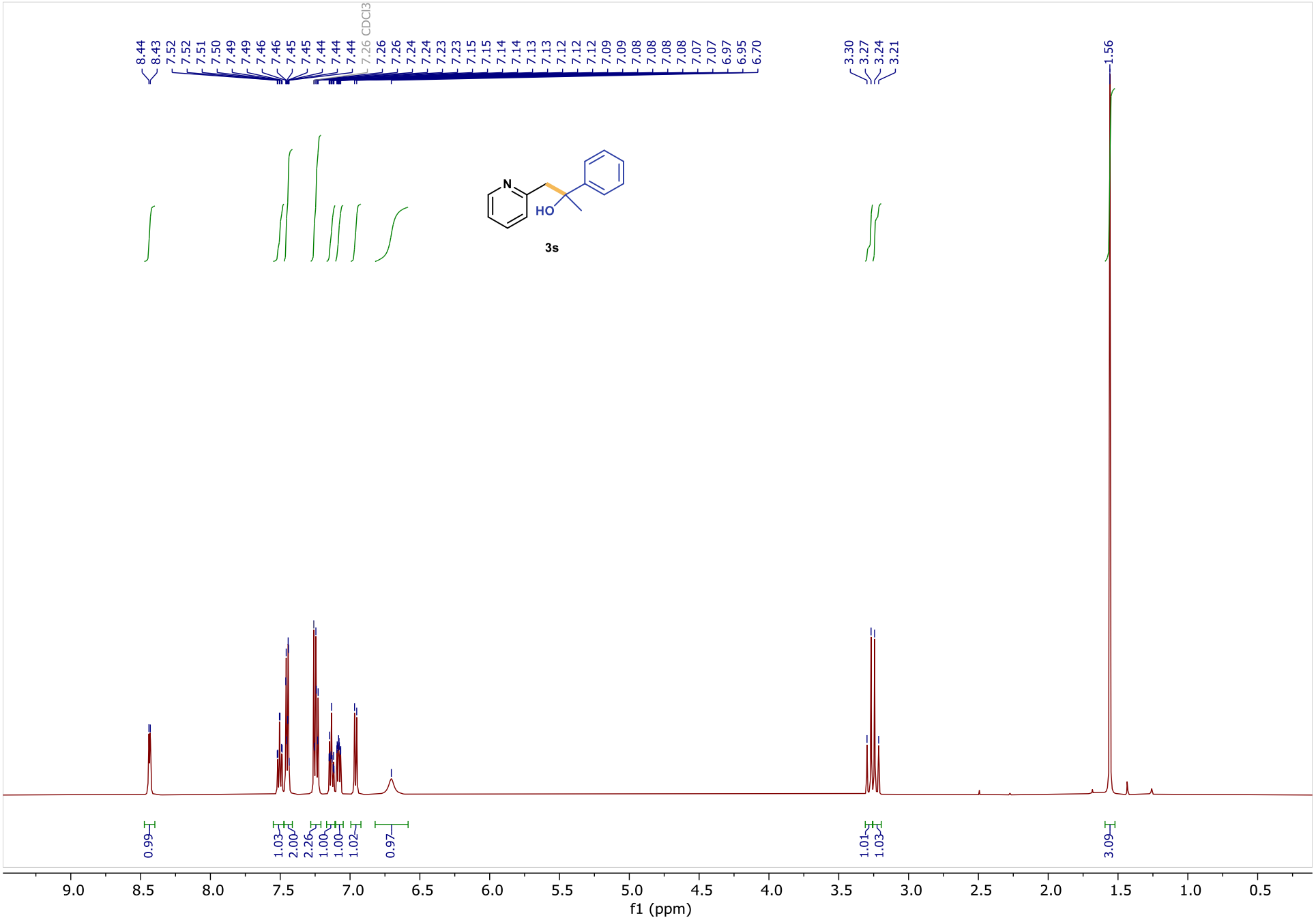
^{1}H NMR of 3r, $CDCl_3$, 500 MHz



^{13}C NMR of 3r, $CDCl_3$, 126 MHz



^{1}H NMR of 3s, $CDCl_3$, 500 MHz



^{13}C NMR of 3s, $CDCl_3$, 126 MHz