Supporting Information

A Tertiary Amine Synthesis via a Hydroxylamine Alkylation/Catalytic Reduction Sequence

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1. General Information

All reactions were performed under ambient air. Heating of reaction mixtures was accomplished via oil bath or microwave irradiation. Analytical thin layer chromatography (TLC) was performed on aluminum-backed plates, cut to size. Visualization was accomplished with UV light followed by staining with a ninhydrin solution and heating. When applicable, flash column chromatography was done using silica gel (40-63 μm).^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker AVANCE 300 MHz, 400 MHz or 600 MHz spectrometers at ambient temperature. Chemical shifts are reported in ppm using residual protio-solvent in deuterated solvent as the reference ($CDCl_3$ at 7.26 ppm for ^{1}H NMR or 77.16 ppm for ^{13}C NMR). ^{1}H NMR data was reported as: multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), integration and coupling constant(s) in Hz. NMR yields were calculated relative to 1,3,5-trimethoxybenzene (TMB) as an internal standard. Reactions relying on microwave heating were performed in the Anton Paar Monowave 400 Reactor, using the G10 vials sealed with a snap cap. Reactions performed in an oil bath used Kimble KIMAX reusable screw thread culture tubes as a conveniently sized vessel, unless otherwise stated.

2. Materials

Unless otherwise stated, all materials were purchased from commercial sources and used without further purifications. Hydroxylamines were prepared according to a previously reported procedure[1a] except for **1a** and **1d** which are commercially available and *N*-hydroxypyrrolidine which was prepared according to literature methods.[2] Benzyl iodide, 2-phenethyl iodide and 3-phenylpropyl iodide were all freshly prepared from the corresponding alkyl bromide prior to reaction according to literature methods.[3] Benzyl tosylate was freshly prepared from benzyl alcohol prior to reaction according to literature methods.[4]

3. Determination of reaction yields by ^{1}H NMR analysis

To determine reaction yields during optimization, we found that the use of the $DMSO_2$ peak was reliable and consistent. The stoichiometric reductant in the reaction is the DMSO solvent that reduces the osmium catalyst following the *N*-oxide reduction, resulting in a 1:1 ratio with the tertiary amine formed. Using 1,3,5-trimethoxybenzene (TMB) as standard, accurate ^{1}H NMR yields were determined relative to this consistent peak. See below for an example of this analysis and validation of this approach.

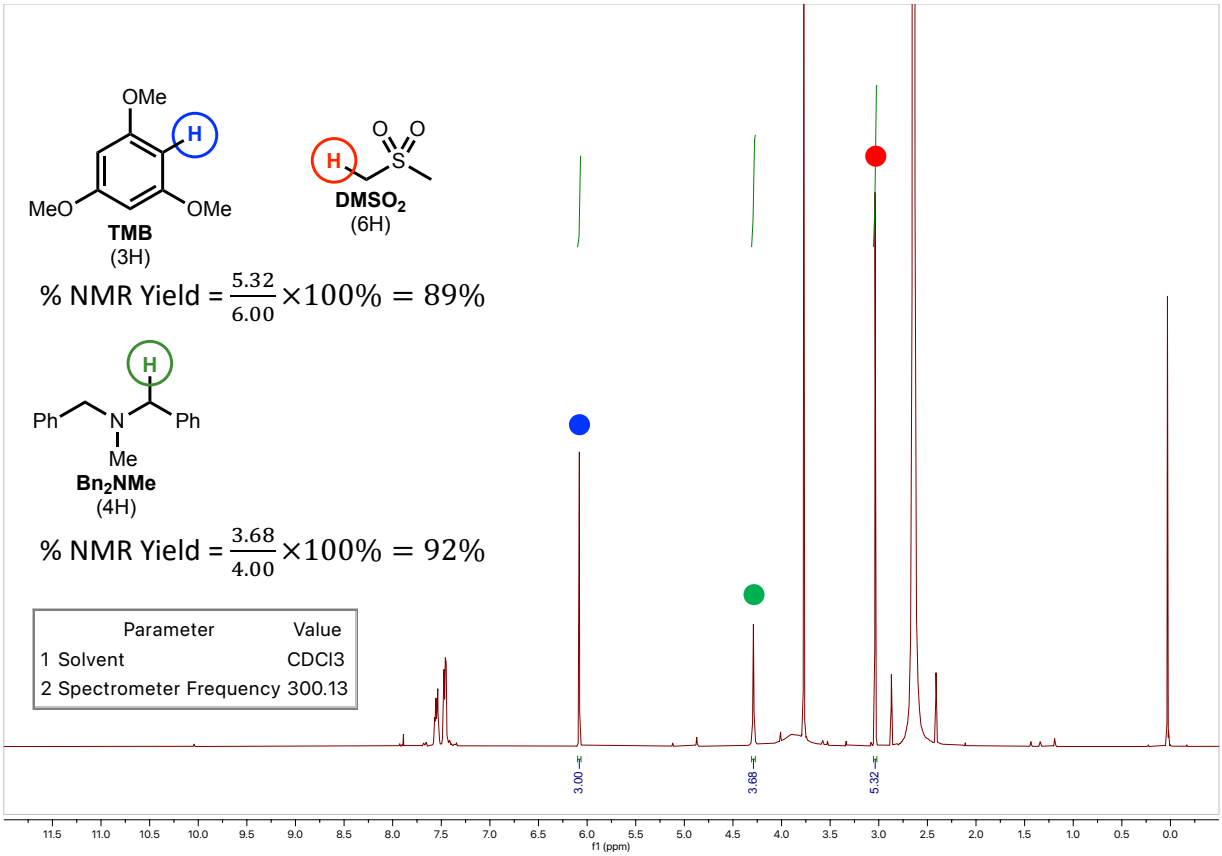
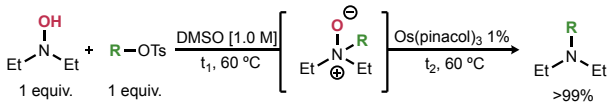


Figure S1. Key peaks for determination of the yield of tertiary amine product using ^{1}H NMR

4. Supplementary data using tosylate electrophiles

The use of tosylate electrophiles was also explored. However, the yields were significantly lower than with iodomethane, and therefore, this avenue was not pursued further. In this section, we report our early optimization using benzyl tosylate with diethylhydroxylamine (Table S1). The use of sodium iodide (10 mol%) as an additive gave the best yields.

Table S1. Optimization using benzyl tosylate



R	t_1 (h)	t_2 (h)	NaI equiv.	Heat Source	Yield %
Bn	2	1	0	Oil Bath	<5
Bn	5	3	0	Oil Bath	5
Bn	5	48	0	Oil Bath	38
Bn	2	2	0	Microwave	<5
Bn	5	1	0.1	Oil Bath	31
Bn	4	2	0.1	Oil Bath	23
Bn	2	4	0.1	Oil Bath	37
Bn	2	18	0.1	Oil Bath	43
Bn	**2**	**2**	**0.1**	**Microwave**	**53**
Bn	2	3	0.1	Microwave	24
Bn	2	4	0.5	Oil Bath	20
Bn	2	18	1.0	Oil Bath	25

(a) *N,N*-Diethylhydroxylamine (1 equiv.) was dissolved in DMSO (1.0 M), alkyl tosylate (1 equiv.) and NaI were added, then stirred at 60 °C for $\mathbf{t_1}$ hours. $Os(pin)_3$ DMSO solution (1 mol%) was added, then continued stirring at 60 °C for $\mathbf{t_2}$ hours. ^{1}H NMR yields using TMB as an internal standard.

5. General Procedures[1]

Hydroxylamine Synthesis, Procedure A: Oxidative Synthesis of Hydroxylamines

The amine (5 equiv.) was added to a G10 microwave vial charged with a stir bar, followed by trifluoroethanol (TFE; 0.5 M). To the solution, urea-hydrogen peroxide (UHP; 1 equiv.) was added and the mixture was stirred at 70 °C for 2 hours in a microwave reactor. Upon completion, the reaction mixture was concentrated using rotary evaporation, then diluted in EtOAc (0.15 M with

respect to UHP). The unreacted amine was removed from the mixture by adding an equimolar amount of oxalic acid, then stirred at room temperature for 30 minutes to precipitate the ammonium salt. The mixture was filtered and washed using EtOAc, then the filtrate was concentrated using rotary evaporation. The resulting crude hydroxylamine was dissolved in a minimal amount of eluent and isolated using a 3-inch silica plug with the same eluent, then concentrated using rotary evaporation to yield the hydroxylamine product.

Hydroxylamine Synthesis Procedure B: Oxidative Synthesis of Hydroxylamines

The amine (1 equiv.) was added to a round bottom flask charged with a stir bar, followed by acetone (0.067 M). To the solution, hexafluoroacetone trihydrate ($HFA \cdot 3H_2O$; 0.72 equiv.) and UHP (1 equiv.) were added, and the mixture was stirred at room temperature for 5 hours. The product was isolated using silica gel flash column chromatography then concentrated using rotary evaporation to yield the hydroxylamine product (unless noted otherwise).

Preparation of Osmium Catalyst: Synthesis of 5 mM / 10 mM $Os(pin)_3$

To a vial charged with a stir bar, potassium osmate dihydrate ($K_2OsO_2(OH)_4$; 7.4 mg, 0.020 mmol, 1 equiv.) was added, followed by hexafluoroacetone trihydrate ($HFA \cdot 3H_2O$; 28 μL, 0.20 mmol, 10 equiv.) to solubilize the osmium. Next, dimethyl sulfoxide (DMSO; 5 mM / 10 mM) was added, followed by pinacol (7.1 mg, 0.060 mmol, 3 equiv.). This mixture was stirred at 60 °C in a heated oil bath for a minimum of 1 hour, resulting in a gray solution. Due to the low solubility of the catalyst in DMSO, a black precipitate sometimes formed. When this was the case, the solution was shaken before measuring and adding the catalyst into the reaction mixture.

General Procedure A: Synthesis of Tertiary Amines Using Iodomethane

The hydroxylamine (1 equiv.) was added to a vial charged with a stir bar, followed by dimethyl sulfoxide (DMSO; 1.0 M). To the solution, the alkyl iodide (1 equiv.) was added, and the mixture was stirred at 60 °C in an oil bath. After 2 hours, the osmium catalyst solution (1 mol%) was added to the mixture, and stirring continued at 60 °C for 18 hours. Upon completion, the product was isolated using the **General Purification Procedure** (see below). The efficiency of the formation of volatile amines (**3b**, **3c**, **3d**, **3m** & **3p**) and (**3v**) in **Scheme 3** and in **Scheme 4** of the manuscript (**3aa-3aj**) were evaluated using ^{1}H NMR analysis, with reaction yields being determined by adding TMB as an internal standard to a solution in 1 mL of $CDCl_3$. The conversion to product was calculated as described in section 3.

General Procedure B: Synthesis of Tertiary Amines Using Epoxides

The hydroxylamine (1 equiv.) was added to a vial charged with a stir bar, followed by MeOH (4.5 M). To the solution, the epoxide (2 equiv.) was added, and the mixture was stirred at 60 °C in an oil bath. After 6 hours, the osmium catalyst solution (1 mol%) was added to the mixture, and stirring continued at 60 °C for 18 hours. Upon completion, the product was isolated using the **General Purification Procedure** (see below).

General Procedure C: Addition of Hydroxylamines to Michael Acceptors

The Michael acceptor (3 equiv.) was added to a vial charged with a stir bar, followed by the osmium catalyst solution (1 mol%) in DMSO, then the secondary hydroxylamine (1.0 equiv.) was added, the vial was sealed and heated to 60 ºC for 18 hours. The tertiary amine product was then

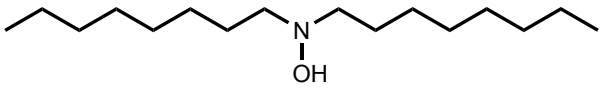
either purified as described for **3u**, **3w**-**3z** below, or in the case of **3v**, **3ae** and **3aj**, an NMR yield was obtained.

General Purification Procedure: Purification of Amines Made Using Methyl Iodide

To the reaction mixture, a few drops of saturated aqueous $Na_2S_2O_3$ was added until the color of iodine was consumed, then pH was adjusted to 10-11 using 1 M aqueous NaOH or KOH. The mixture was diluted with water (10 mL on a 0.2 mmol scale), then transferred to a separatory funnel. The aqueous solution was extracted 3 times using CH_2Cl_2 (10 mL each on a 0.2 mmol scale), then the aqueous phase was discarded. The organic phase was washed 5 times with brine (10 mL each on a 0.2 mmol scale), then dried over Na_2SO_4, filtered and concentrated using a rotary evaporator to yield the amine product.

6. Characterization Data

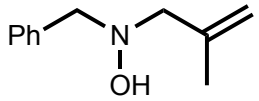
6.1 Hydroxylamines



***N,N*-Dioctylhydroxylamine (2e):** The title compound was synthesized according to **Hydroxylamine Synthesis Procedure B** using dioctylamine (0.242 g, 1.00 mmol, 1.00 equiv.), HFA·$3H_2O$ (0.100 mL, 0.717 mmol, 0.717 equiv.), UHP (0.094 g, 1.0 mmol, 1.0 equiv.) in acetone (15 mL, 0.067 M). The product was purified by cooling in a freezer for 1 hour to precipitate the hydroxylamine, then filtered and washed with acetone to remove remaining impurities. The solid was dissolved in CH_2Cl_2 and filtered to avoid urea contamination, then concentrated using a rotary evaporator to yield the title compound as a white solid (0.086 g, 33%). Characterization data was in good agreement with previously reported data.[5]

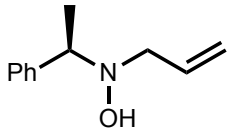
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 (br s, 1H), 2.68 – 2.60 (m, 4H), 1.63 – 1.51 (m, 4H), 1.35 – 1.20 (m, 20H), 0.91 – 0.83 (m, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 60.9, 32.0, 29.7, 29.4, 27.6, 27.3, 22.8, 14.2.



<u>*N*-Benzyl-*N*-(2-methylallyl)hydroxylamine</u> (2j): The title compound was synthesized according to a modified **Hydroxylamine Synthesis Procedure B** using *N*-benzyl-2-methylallylamine hydrochloride (0.198 g, 1.00 mmol, 1.00 equiv.), 1 M aqueous KOH (1 mL, 1 mmol, 1 equiv.), HFA·3H_2O (0.100 mL, 0.717 mmol, 0.717 equiv.), UHP (0.094 g, 1.0 mmol, 1.0 equiv.) in acetone (15 mL, 0.067 M). The product was purified using silica gel flash column chromatography (1:9 EtOAc: Pet. Ether) to yield the compound as a white solid (0.073 g, 41 %). Characterization data was in good agreement with previously reported data.[6]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.24 (m, 5H), 6.22 (br s, 1H), 4.95 (dq, J = 2.1, 1.1 Hz, 1H), 4.91 (ddq, J = 2.2, 1.4, 0.8 Hz, 1H), 3.72 (s, 2H), 3.21 (d, J = 1.2 Hz, 2H), 1.76 (t, J = 1.2 Hz, 3H).



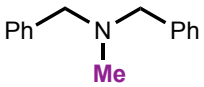
<u>(*R*)-*N*-Allyl-*N*-(1-phenylethyl)hydroxylamine</u> (2l): The title compound was synthesized according to the **Hydroxylamine Synthesis Procedure B** using (*R*)-*N*-allyl-(1-phenylethyl)amine (0.175 mL, 1.00 mmol, 1.00 equiv.), HFA·3H_2O (0.100 mL, 0.717 mmol, 0.717 equiv.), UHP (0.094 g, 1.0 mmol, 1.0 equiv.) in acetone (15 mL, 0.067 M). The product was purified using silica

gel flash column chromatography (15:85 EtOAc:Pet. Ether) to yield the compound as a colourless oil (0.087 g, 49 %). Characterization data is in good agreement with previously reported data.[7]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.24 (m, 5H), 6.33 (br s, 1H), 6.08 – 5.93 (m, 1H), 5.20 (t, *J* = 1.2 Hz, 1H), 5.17 – 5.13 (m, 1H), 3.86 (q, *J* = 6.7 Hz, 1H), 3.33 (ddt, *J* = 13.9, 6.1, 1.4 Hz, 1H), 3.22 (dd, *J* = 14.0, 6.9 Hz, 1H), 1.50 (d, *J* = 6.7 Hz, 3H).

6.2 Isolated Tertiary Amines

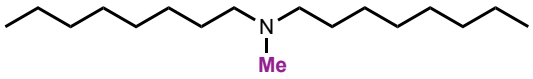
Amines Prepared using Methyl Iodide



***N*-Methyldibenzylamine (3a):** The title compound was synthesized according to a modified **General Procedure A** with a 2-hour reduction time using *N,N*-dibenzylhydroxylamine (0.107 g, 0.500 mmol, 1.00 equiv.), iodomethane (0.031 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.083 g, 79%). Characterization data is in good agreement with previously reported data.[8] Trace benzaldehyde is observed in ^{1}H NMR.[32]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.33 (m, 8H), 7.32 – 7.25 (m, 2H), 3.57 (s, 4H), 2.23 (s, 3H).

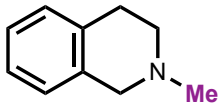
^{13}C NMR (101 MHz, $CDCl_3$) δ 139.4, 129.0, 128.3, 127.0, 62.0, 42.3.



***N*-Methyldioctylamine (3e):** The title compound was synthesized according to **General Procedure A** using *N,N*-dioctylhydroxylamine (0.097 g, 0.38 mmol, 1.0 equiv.), iodomethane (0.024 mL, 0.38 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.38 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a dark yellow oil (0.092 g, 96%). Characterization data is in good agreement with previously reported data.[9]

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.33 – 2.26 (m, 4H), 2.19 (s, 3H), 1.50 – 1.37 (m, 4H), 1.31 – 1.19 (m, 20H), 0.88 – 0.81 (m, 6H).

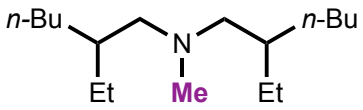
^{13}C NMR (101 MHz, $CDCl_3$) δ 57.9, 42.3, 32.0, 29.7, 29.4, 27.7, 27.3, 22.8, 14.2.



2-Methyl-1,2,3,4-tetrahydroisoquinoline (3f): The title compound was synthesized according to **General Procedure A** using 3,4-dihydroisoquinolin-2(1H)-ol (0.075 g, 0.50 mmol, 1.0 equiv.), iodomethane (0.031 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.069 mg, 92%). Characterization data is in good agreement with previously reported data.[8]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 – 7.08 (m, 3H), 7.01 (d, J = 6.8 Hz, 2H), 3.60 (s, 2H), 2.93 (t, J = 6.1 Hz, 2H), 2.70 (t, J = 6.0 Hz, 2H), 2.46 (s, 3H).

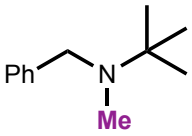
^{13}C NMR (151 MHz, $CDCl_3$) δ 134.7, 133.8, 128.8, 126.5, 126.3, 125.7, 58.0, 53.0, 46.1, 29.2.



***N*-Methyl-bis(2-ethylhexyl)amine (3g):** The title compound was synthesized according to **General Procedure A** using *N,N*-bis(2-ethylhexyl)hydroxylamine (0.257 g, 1.00 mmol, 1.00 equiv.), iodomethane (0.062 mL, 1.0 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (1.0 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.251 g, 98%).[10]

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.10 (s, 3H), 2.06 (d, J = 8.3 Hz, 4H), 1.37 – 1.20 (m, 18H), 0.94 – 0.79 (m, 12H).

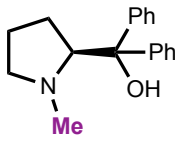
^{13}C NMR (101 MHz, $CDCl_3$) δ 63.4, 43.4, 43.4, 37.3, 31.5, 31.5, 29.0, 24.7, 24.6, 23.4, 14.3, 10.9, 10.8.



***N*-Methyl-*N*-benzyl-*tert*-butylamine (3h):** The title compound was synthesized according to **General Procedure A** using *N*-benzyl-*N*-(*tert*-butyl)hydroxylamine (0.054 g, 0.30 mmol, 1.0 equiv.), iodomethane (0.019 mL, 0.30 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.30 mL, 1.0 M). The product was purified according to the **General Purification Procedure**, then additionally purified using silica gel flash column chromatography (1:9 acetone:hexanes) to yield the title compound as a yellow oil (0.015 g, 27%).[11] Trace benzaldehyde is observed in ^{1}H NMR.[32]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 – 7.19 (m, 5H), 3.52 (s, 2H), 2.11 (s, 3H), 1.18 (s, 9H).

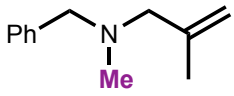
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.7, 128.8, 128.3, 126.6, 55.4, 34.9, 28.5, 26.4.



(*S*)-1,1-Diphenyl-*N*-methylprolinol (3i): The title compound was synthesized according to **General Procedure A** using (*S*)-2-(hydroxydiphenylmethyl)pyrrolidine-1-ol (0.108 g, 0.400 mmol, 1.00 equiv.), iodomethane (0.025 mL, 0.40 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.40 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a dark brown solid (0.094 g, 88%). Characterization data is in good agreement with previously reported data.[8]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 – 7.64 (m, 2H), 7.59 – 7.54 (m, 2H), 7.33 – 7.24 (m, 4H), 7.19 – 7.11 (m, 2H), 4.69 (br s, 1H), 3.66 (dd, *J* = 9.4, 4.1 Hz, 1H), 3.21 – 3.08 (m, 1H), 2.47 (td, *J* = 9.5, 6.6 Hz, 1H), 2.01 – 1.88 (m, 1H), 1.85 (s, 3H), 1.79 – 1.61 (m, 3H).

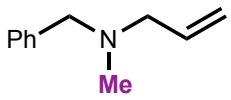
^{13}C NMR (101 MHz, $CDCl_3$) δ 148.2, 146.8, 128.4, 128.1, 126.3, 125.6, 125.5, 77.6, 72.2, 59.3, 43.1, 30.0, 24.1.



***N*-Benzyl-*N*-methyl-2-methylallylamine (3j):** The title compound was synthesized according to **General Procedure A** using *N*-benzyl-*N*-(2-methylallyl)hydroxylamine (0.071 g, 0.40 mmol, 1.0 equiv.), iodomethane (0.025 mL, 0.40 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.40 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.056 g, 80%). Characterization data is in good agreement with previously reported data.[12] Trace benzaldehyde is observed in ^{1}H NMR.[32]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.21 (m, 5H), 4.95 – 4.92 (m, 1H), 4.89 – 4.86 (m, 1H), 3.46 (s, 2H), 2.91 (s, 2H), 2.15 (s, 3H), 1.81 (s, 3H).

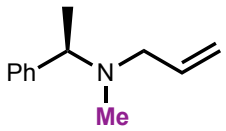
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.9, 139.7, 129.0, 128.3, 126.9, 112.7, 64.7, 61.9, 42.4, 20.9.



***N*-Allyl-*N*-methylbenzylamine (3k):** The title compound was synthesized according to **General Procedure A** using *N*-allyl-*N*-benzylhydroxylamine (0.098 g, 0.60 mmol, 1.0 equiv.), iodomethane (0.037 mL, 0.60 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.60 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a brown oil (0.055 g, 57%). Characterization data is in good agreement with previously reported data.[12] Trace benzaldehyde is observed in ^{1}H NMR.[32]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 5H), 5.93 (ddt, J = 16.8, 10.2, 6.5 Hz, 1H), 5.26 – 5.13 (m, 2H), 3.51 (s, 2H), 3.05 (d, J = 6.5 Hz, 2H), 2.20 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.0, 135.9, 129.3, 128.3, 127.1, 117.7, 61.8, 60.6, 42.2.

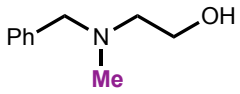


(*R*)-*N*-Allyl-*N*-methyl-(1-phenylethyl)amine (3l): The title compound was synthesized according to **General Procedure A** using (*R*)-*N*-allyl-*N*-(1-phenylethyl)hydroxylamine (0.073 g, 0.41 mmol, 1.0 equiv.), iodomethane (0.026 mL, 0.41 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.41 mL, 1.0 M). The product was purified according to the **General**

Purification Procedure to yield the title compound as a brown oil (0.039 g, 54%). Characterization data is in good agreement with previously reported data.[13]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 4H), 7.28 – 7.22 (m, 1H), 5.87 (ddt, *J* = 16.9, 10.1, 6.6 Hz, 1H), 5.19 – 5.10 (m, 2H), 3.60 (q, *J* = 6.8 Hz, 1H), 3.10 (dd, *J* = 13.8, 6.1 Hz, 1H), 2.91 (dd, *J* = 13.7, 6.9 Hz, 1H), 2.20 (s, 3H), 1.40 (d, *J* = 6.8 Hz, 3H).

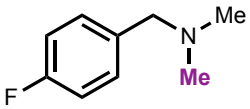
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.5, 135.9, 128.4, 127.9, 127.1, 117.6, 63.2, 57.8, 38.4, 19.0.



***N*-Benzyl-*N*-methylethanolamine (3n):** The title compound was synthesized according to **General Procedure A** using 2-(benzyl(hydroxy)amino)ethanol (0.067 g, 0.40 mmol, 1.0 equiv.), iodomethane (0.025 mL, 0.40 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.40 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.042 g, 63%). Characterization data is in good agreement with previously reported data.[8] Trace benzaldehyde is observed in ^{1}H NMR.[32]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 – 7.22 (m, 5H), 3.70 – 3.60 (m, 2H), 3.59 (s, 2H), 3.01 (br s, 1H), 2.65 – 2.60 (m, 2H), 2.25 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.5, 129.1, 128.5, 127.3, 62.4, 58.5, 58.5, 41.6.



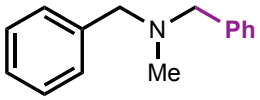
***N*,*N*-Dimethyl-4-fluorobenzylamine (3o):** The title compound was synthesized according to **General Procedure A** using *N*-(4-fluorobenzyl)-*N*-methylhydroxylamine (0.062 g, 0.40 mmol, 1.0 equiv.), iodomethane (0.025 mL, 0.40 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%)

in DMSO (0.4 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.044 g, 72%). Characterization data is in good agreement with previously reported data.[14]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.05 – 6.95 (m, 2H), 3.38 (s, 2H), 2.23 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.1 (d, $^1J_{C\text{-}F}$ = 245 Hz), 134.7 (d, $^4J_{C\text{-}F}$ = 3 Hz), 130.7 (d, $^3J_{C\text{-}F}$ = 8 Hz), 115.1 (d, $^2J_{C\text{-}F}$ = 21 Hz), 63.7, 45.3.

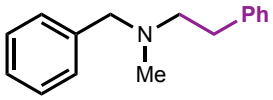
Compounds Prepared with Electrophiles other than Methyl Iodide



<u>*N*-Methyldibenzylamine</u> (3a): The title compound was synthesized according to a modified **General Procedure A** using *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.500 mmol, 1.00 equiv.), benzyl iodide (0.109 mL, 0.500 mmol, 1.00 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.102 g, 95%). Characterization data is in good agreement with previously reported data.[8] Trace benzaldehyde is observed in ^{1}H NMR.[32]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 6.86 (m, 10H), 3.55 (s, 4H), 2.20 (s, 3H).

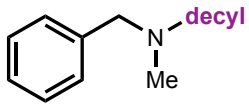
^{13}C NMR (101 MHz, $CDCl_3$) δ 139.3, 129.1, 128.4, 127.1, 62.0, 42.3.



***N*-Benzyl-*N*-methyl-2-phenylethan-1-amine (3q):** The title compound was synthesized according to a modified **General Procedure A** using *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.500 mmol, 1.00 equiv.), phenethyl iodide (0.072 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.045 g, 45%). Characterization data is in good agreement with previously reported data.[15]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.11 (m, 10H), 3.55 (s, 2H), 2.81 (dd, *J* = 9.6, 6.1 Hz, 2H), 2.68 – 2.60 (m, 2H), 2.26 (s, 3H).

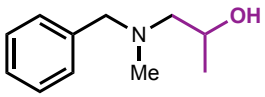
^{13}C NMR (101 MHz, $CDCl_3$) δ 129.2, 128.9, 128.5, 128.4, 127.2, 126.1, 62.3, 59.3, 42.3, 34.0.



***N*-Benzyl-*N*-methyldecan-1-amine (3r):** The title compound was synthesized according to a modified **General Procedure A** using *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.500 mmol, 1.00 equiv.), iododecane (0.11 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a brown oil (0.081 g, 62%). Characterization data is in good agreement with previously reported data.[16]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.20 (m, 5H), 3.50 (s, 2H), 2.37 (t, *J* = 7.6 Hz, 2H), 2.20 (s, 3H), 1.56 – 1.46 (m, 2H), 1.36 – 1.21 (m, 14H), 0.88 (t, *J* = 6.8 Hz, 3H).

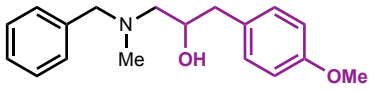
^{13}C NMR (101 MHz, $CDCl_3$) 133.6, 129.3, 128.3, 127.1, 62.4, 57.7, 42.3, 32.1, 29.8, 29.7, 29.5, 27.6, 27.4, 22.8, 14.3.



1-(*N*-benzyl-*N*-methylamino)propan-2-ol (3s): The title compound was synthesized according to **General Procedure B** using *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol, 1.0 equiv.), propylene oxide (0.070 mL, 1.0 mmol, 2.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in MeOH (0.11 mL, 4.5 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a yellow oil (0.066 g, 74%). Characterization data is in good agreement with previously reported data.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.23 (m, 5H), 3.88 (dqd, *J* = 9.6, 6.1, 3.4 Hz, 1H), 3.69 (d, *J* = 13.1 Hz, 1H), 3.48 (d, *J* = 13.1 Hz, 1H), 2.39 (dd, *J* = 12.3, 10.0 Hz, 1H), 2.33 (dd, *J* = 12.3, 3.4 Hz, 1H), 2.25 (s, 3H), 1.15 (d, *J* = 6.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.5, 129.1, 128.5, 127.3, 65.1, 63.1, 62.6, 42.0, 20.1.

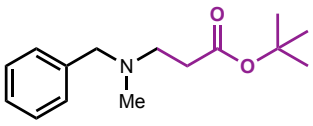


1-(*N*-benzyl-*N*-methylamino)-3-(4-methoxyphenyl)propan-2-ol (3t): The title compound was synthesized according to **General Procedure B** using *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol, 1.0 equiv.), estragole oxide (0.164 g, 1.00 mmol, 2.00 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in MeOH (0.11 mL, 4.5 M). The product was purified according to the **General Purification Procedure** to yield the title compound as a brown oil (0.107 g, 75%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.25 (m, 5H), 7.19 – 7.15 (m, 2H), 6.89 – 6.84 (m, 2H), 3.97 – 3.89 (m, 1H), 3.81 (s, 3H), 3.67 (d, *J* = 13.1 Hz, 1H), 3.46 (d, *J* = 13.1 Hz, 1H), 2.78 (dd, *J* = 13.8, 7.0 Hz, 1H), 2.64 (dd, *J* = 13.8, 5.6 Hz, 1H), 2.47 (dd, *J* = 12.3, 10.0 Hz, 1H), 2.38 (dd, *J* = 12.2, 3.4 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.2, 138.4, 130.5, 130.3, 129.1, 128.4, 113.9, 68.2, 62.9, 62.4, 55.3, 42.1, 40.5.

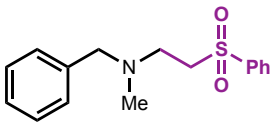
HRMS (EI) m/z: $[M]^+$ calcd for $C_{18}H_{23}NO_2^+$ 285.1729; found 285.1706.



***tert*-Butyl 3-(benzyl(methyl)amino)propanoate (3u):** The title compound was synthesized according to **General Procedure C** using *tert*-butyl acrylate (0.22 mL, 1.5 mmol, 3.0 equiv.), *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). The mixture was diluted with water (25 mL on a 0.5 mmol scale), then transferred to a separatory funnel. The aqueous solution was extracted 3 times using CH_2Cl_2 (25 mL each on a 0.5 mmol scale), then the aqueous phase was discarded. The organic phase was washed 5 times with brine (25 mL each on 0.5 mmol scale), then dried over Na_2SO_4, filtered, concentrated using a rotary evaporator and dried in vacuo to yield the title product as a brown oil (0.113g, 91%). Characterization data is in good agreement with previously reported data.[18]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.20 (m, 5H), 3.51 (s, 2H), 2.73 (t, J = 7.3 Hz, 2H), 2.45 (t, J = 7.3 Hz, 2H), 2.20 (s, 3H), 1.45 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 129.1, 128.4, 127.1, 80.4, 62.2, 53.2, 41.9, 34.1, 28.3.



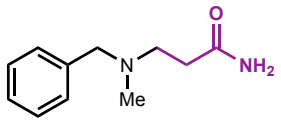
***N*-Benzyl-*N*-methyl-2-(phenylsulfonyl)ethan-1-amine (3w):** The title compound was synthesized according to **General Procedure C** using phenyl vinyl sulfone (0.23 g, 1.5 mmol,

3.0 equiv.), *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). To the reaction mixture, 10 mL of brine was added, and then the pH was adjusted to 12 using solid KOH. The mixture was transferred to a separatory funnel, and the aqueous phase was extracted 3 times with EtOAc (10 mL each on a 0.5 mmol scale), and then the aqueous phase was discarded. The product was extracted 3 times with 1 M HCl. The combined HCl phases were neutralized with solid KOH to a pH of 12 and were extracted 3 times with CH_2Cl_2. The organic phase was washed 5 times with brine (10 mL each on a 0.5 mmol scale), then dried over Na_2SO_4, filtered and concentrated using a rotary evaporator. Then this compound was additionally purified using silica gel flash column chromatography (10:0 to 0:10 pet ether: Et_2O) to yield the title compound as a yellow oil (0.096 g, 66%) yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.72 (m, 2H), 7.56 (tt, *J* = 7.5, 1.0 Hz, 1H), 7.45 (tt, *J* = 7.9, 1.6 Hz, 2H), 7.23 – 7.09 (m, 3H), 7.10 – 7.03 (m, 2H), 3.36 (s, 2H), 3.30 – 3.05 (m, 2H), 2.92 – 2.66 (m, 2H), 2.05 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.5, 137.9, 133.7, 129.3, 128.8, 128.3, 128.0, 127.3, 61.8, 53.8, 50.3, 41.8.

HRMS (EI) m/z: $[M]^+$ calcd for $C_{16}H_{19}N_2O^+$ 289.1137; found 289.1109.



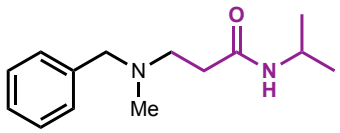
3-(Benzyl(methyl)amino)propanamide (3x): The title compound was synthesized according to **General Procedure C** using acrylamide (0.11 g, 1.5 mmol, 3.0 equiv.), *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). To the reaction mixture, 10 mL of brine was added, and then the pH was adjusted to 12 using

solid KOH. The mixture was transferred to a separatory funnel, and the aqueous phase was extracted 3 times with EtOAc (10 mL each on a 0.5 mmol scale), and then the aqueous phase was discarded. The product was extracted 3 times with 1 M HCl. The combined HCl phases were neutralized with solid KOH to a pH of 12 and were extracted 3 times with CH_2Cl_2. The organic phase was washed 5 times with brine (10 mL each on a 0.5 mmol scale), then dried over Na_2SO_4, filtered and concentrated using a rotary evaporator to yield the title compound as a white solid (0.080 g, 83%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.27 – 7.20 (m, 2H), 7.20 – 7.14 (m, 3H), 6.05 (s, 1H), 3.44 (s, 2H), 2.60 (t, *J* = 6.2 Hz, 2H), 2.34 (t, *J* = 6.2 Hz, 2H), 2.12 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 175.3, 137.9, 129.1, 128.4, 127.4, 62.1, 53.3, 41.2, 33.0.

HRMS (EI) m/z: $[M]^+$ calcd for $C_{11}H_{16}N_2O^+$ 192.1263; found 192.1251.

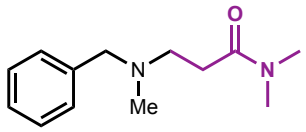


3-(Benzyl(methyl)amino)-*N*-isopropylpropanamide (3y): The title compound was synthesized according to **General Procedure C** using *N*-isopropylacrylamide (0.17 g, 1.5 mmol, 3.0 equiv.), *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). To the reaction mixture, 10 mL of brine was added, and then the pH was adjusted to 12 using solid KOH. The mixture was transferred to a separatory funnel and the aqueous phase was extracted 3 times with EtOAc (10 mL each on a 0.5 mmol scale), the organic layers were dried over Na_2SO_4, filtered and concentrated using a rotary evaporator. Then this compound was additionally purified using silica gel flash column chromatography (10:0 to 9:1 EtOAc: MeOH (with 3% NH_3OH) to yield the title compound as a yellow oil (0.088 g, 75%) yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.14 – 7.93 (s 1H), 7.38 – 7.30 (m, 2H), 7.30 – 7.19 (m, 3H), 4.22 – 3.95 (m, 1H), 3.51 (s, 2H), 2.66 (t, *J* = 6.1 Hz, 2H), 2.38 (t, *J* = 6.1 Hz, 2H), 2.20 (d, *J* = 1.1 Hz, 3H), 1.13 (dd, *J* = 6.7, 1.0 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 175.3, 137.9, 129.1, 128.4, 127.4, 62.1, 53.3, 41.2, 33.0.

HRMS (EI) m/z: $[M]^+$ calcd for $C_{14}H_{22}N_2O^+$ 234.1732; found 234.1724.



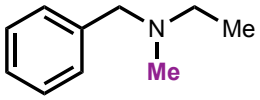
3-(Benzyl(methyl)amino)-*N*,*N*-dimethylpropanamide (3z): The title compound was synthesized according to **General Procedure C** using *N,N*-dimethylacrylamide (0.15 g, 1.5 mmol, 3.0 equiv.), *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). To the reaction mixture, 10 mL of brine (NaCl) was added, and then the pH was adjusted to 12 using solid KOH. The mixture was transferred to a separatory funnel, and the aqueous solution was extracted 3 times with EtOAc (10 mL each on a 0.5 mmol scale), the organic layers were dried over Na_2SO_4, filtered and concentrated using a rotary evaporator. Then this compound was additionally purified using silica gel flash column chromatography (10:0 to 7:3 EtOAc: MeOH (with 3% NH_3OH) to yield the title compound as a yellow oil (0.109 g, 99%) yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 4H), 7.18 – 7.11 (m, 1H), 3.47 (s, 2H), 2.87 (s, 3H), 2.83 (s, 3H), 2.69 (t, *J* = 8.1 Hz, 2H), 2.46 (t, *J* = 7.7 Hz, 2H), 2.17 (s, 3H).

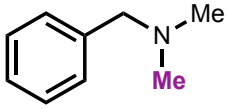
^{13}C NMR (151 MHz, $CDCl_3$) δ 171.7, 138.6, 129.1, 128.2, 127.1, 62.3, 53.0, 42.1, 37.2, 35.3, 31.6.

HRMS (EI) m/z: $[M]^+$ calcd for $C_{13}H_{20}N_2O^+$ 220.1576; found 220.1554.

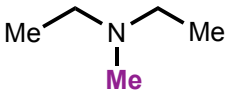
6.3 NMR Yields of Non-Isolated Amines



***N*-Ethyl-*N*-methylbenzylamine (3b):** The title compound was synthesized according to **General Procedure A** using *N*-ethyl-*N*-benzylhydroxylamine (0.015 g, 0.10 mmol, 1.0 equiv.), iodomethane (0.006 mL, 0.10 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.10 mL, 1.0 M). 95% NMR yield observed using 1.0 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[19]

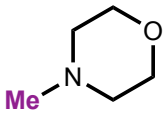


***N,N*-Dimethylbenzylamine (3c):** The title compound was synthesized according to **General Procedure A** using *N*-methyl-*N*-benzylhydroxylamine (0.067 g, 0.50 mmol, 1.0 equiv.), iodomethane (0.031 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 89% NMR yield observed using 0.2 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[8]

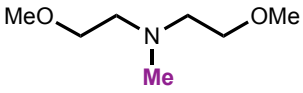


***N,N*-Diethylmethylamine (3d):** The title compound was synthesized according to **General Procedure A** using *N,N*-diethylhydroxylamine (0.010 mL, 0.10 mmol, 1.0 equiv.), iodomethane (0.006 mL, 0.10 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.10 mL, 1.0

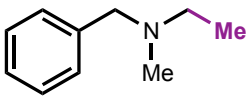
M). >99% NMR yield observed using 1.0 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[20]



4-Methyl-morpholine (3m): The title compound was synthesized according to **General Procedure A** using 4-hydroxymorpholine (0.021 g, 0.20 mmol, 1.0 equiv.), iodomethane (0.012 mL, 0.20 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.20 mL, 1.0 M). 66% NMR yield observed using 1.0 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[8]

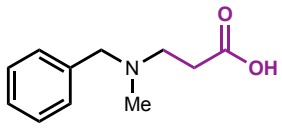


***N*,*N*-bis(2-Methoxyethyl)methylamine (3p):** The title compound was synthesized according to **General Procedure A** using *N*-hydroxy-2-methoxy-*N*-(2-methoxyethyl)ethanamine (0.015 g, 0.10 mmol, 1.0 equiv.), iodomethane (0.006 mL, 0.10 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.10 mL, 1.0 M). 45% NMR yield observed using 1.0 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[8]

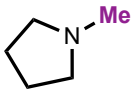


***N*-Ethyl-*N*-methylbenzylamine (3b):** The title compound was synthesized according to a modified **General Procedure A** using *N*-ethyl-*N*-benzylhydroxylamine (0.069 g, 0.50 mmol, 1.0

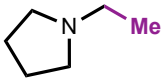
equiv.), ethyl iodide (0.040 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 89% NMR yield observed using 1.0 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[19]



3-(Benzyl(methyl)amino)propanoic acid (3v): The title compound was synthesized according to **General Procedure C** using acrylic acid (0.10 mL, 1.5 mmol, 3.0 equiv.), *N*-benzyl-*N*-methylhydroxylamine (0.069 g, 0.50 mmol), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 91% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[21]

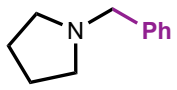


1-Methylpyrrolidine (3aa): The title compound was synthesized according to **General Procedure A** using pyrrolidin-1-ol (0.044 g, 0.500 mmol, 1.00 equiv.), iodomethane (0.031 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 85% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[22]

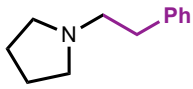


1-Ethylpyrrolidine (3ab): The title compound was synthesized according to a modified **General Procedure A** using pyrrolidine-1-ol (0.044 g, 0.500 mmol, 1.00 equiv.), ethyl iodide (0.040 mL,

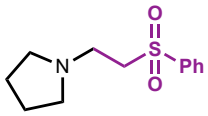
0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 93% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[23]



1-Benzylpyrrolidine (3ac): The title compound was synthesized according to a modified **General Procedure A** using pyrrolidine-1-ol (0.044 g, 0.500 mmol, 1.00 equiv.), benzyl iodide (0.109 g, 0.500 mmol, 1.00 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 62% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[22]

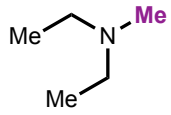


1-Phenethylpyrrolidine (3ad): The title compound was synthesized according to a modified **General Procedure A** using pyrrolidine-1-ol (0.044 g, 0.500 mmol, 1.00 equiv.), phenethyl iodide (0.072 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 8% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[24]

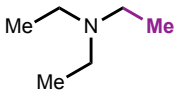


1-(2-(Phenylsulfonyl)ethyl)pyrrolidine (3ae): The title compound was synthesized according to **General Procedure C** using phenyl vinyl sulfone (0.23 g, 1.5 mmol, 3.0 equiv.), pyrrolidin-1-ol

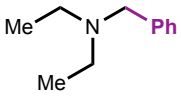
(0.044 g, 0.500 mmol, 1.00 equiv.), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 49% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[25]



<u>*N*,*N*-Diethylmethylamine</u> (3af): The title compound was synthesized according to **General Procedure A** using *N*,*N*-diethylhydroxylamine (0. 051 mL, 0.50 mmol, 1.0 equiv.), iodomethane (0.031 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 74% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data. [20]

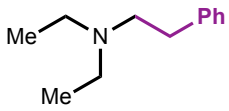


<u>Triethylamine</u> (3ag): The title compound was synthesized according to a modified **General Procedure A** using *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0 equiv.), ethyl iodide (0.049 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 23% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[26]

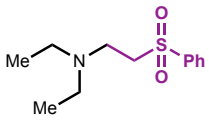


<u>*N*-Benzyl-*N*-ethylethanamine</u> (3ah): The title compound was synthesized according to a modified **General Procedure A** using *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0

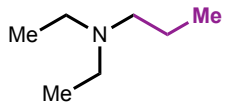
equiv.), benzyl iodide (0.109 g, 0.500 mmol, 1.00 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 53% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[27]



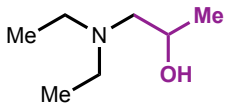
***N*,*N*-Diethyl-2-phenylethan-1-amine (3ai):** The title compound was synthesized according to a modified **General Procedure A** using *N*,*N*-diethylhydroxylamine (0.051 g, 0.50 mmol, 1.0 equiv.), phenethyl iodide (0.072 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 8% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[28]



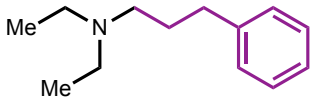
***N*,*N*-Diethyl-2-(phenylsulfonyl)ethan-1-amine (3aj):** The title compound was synthesized according to **General Procedure C** using phenyl vinyl sulfone (0.23 g, 1.5 mmol, 3.0 equiv.), *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 52% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[25]



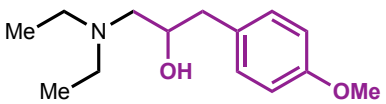
***N*,*N*-Diethylpropan-1-amine (3ak):** The title compound was synthesized according to a modified **General Procedure A** using *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0 equiv.), propyl iodide (0.049 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 5% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[29]



1-(Diethylamino)propan-2-ol (3al): The title compound was synthesized according to **General Procedure B** using *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0 equiv.), propylene oxide (0.070 mL, 1.0 mmol, 2.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 72% NMR yield observed using 1.0 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[30]



***N*,*N*-Diethyl-3-phenylpropan-1-amine (3am):** The title compound was synthesized according to a modified **General Procedure A** using *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0 equiv.), phenpropyl iodide (0.080 mL, 0.50 mmol, 1.0 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 8% NMR yield observed using 0.4 equivalent of TMB as an internal standard. Spectral data is in good agreement with previously reported data.[31]



1-(Diethylamino)-3-(4-methoxyphenyl)propan-2-ol (3an): The title compound was synthesized according to **General Procedure B** using *N*,*N*-diethylhydroxylamine (0.051 mL, 0.50 mmol, 1.0 equiv.), estragole oxide (0.164 g, 1.00 mmol, 2.00 equiv.), $Os(pin)_3$ DMSO solution (1 mol%) in DMSO (0.50 mL, 1.0 M). 65% NMR yield observed using 1.0 equivalent of TMB as an internal standard. The product was purified according to the **General Purification Procedure** to yield the title compound as a brown oil (0.055 g, 47%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 – 7.07 (m, 2H), 6.91 – 6.79 (m, 2H), 3.88 – 3.64 (m, 5H), 2.77 (dd, *J* = 13.8, 7.0 Hz, 1H), 2.70 – 2.53 (m, 3H), 2.53 – 2.39 (m, 3H), 2.32 (dd, *J* = 12.6, 10.1 Hz, 1H), 1.00 (t, *J* = 7.1 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.2, 130.7, 130.3, 113.9, 68.1, 59.2, 55.3, 47.3, 40.6, 12.0.

HRMS (EI) m/z: $[M]^+$ calcd for $C_{14}H_{23}NO_2^+$ 237.1729; found 237.1730.

7. References

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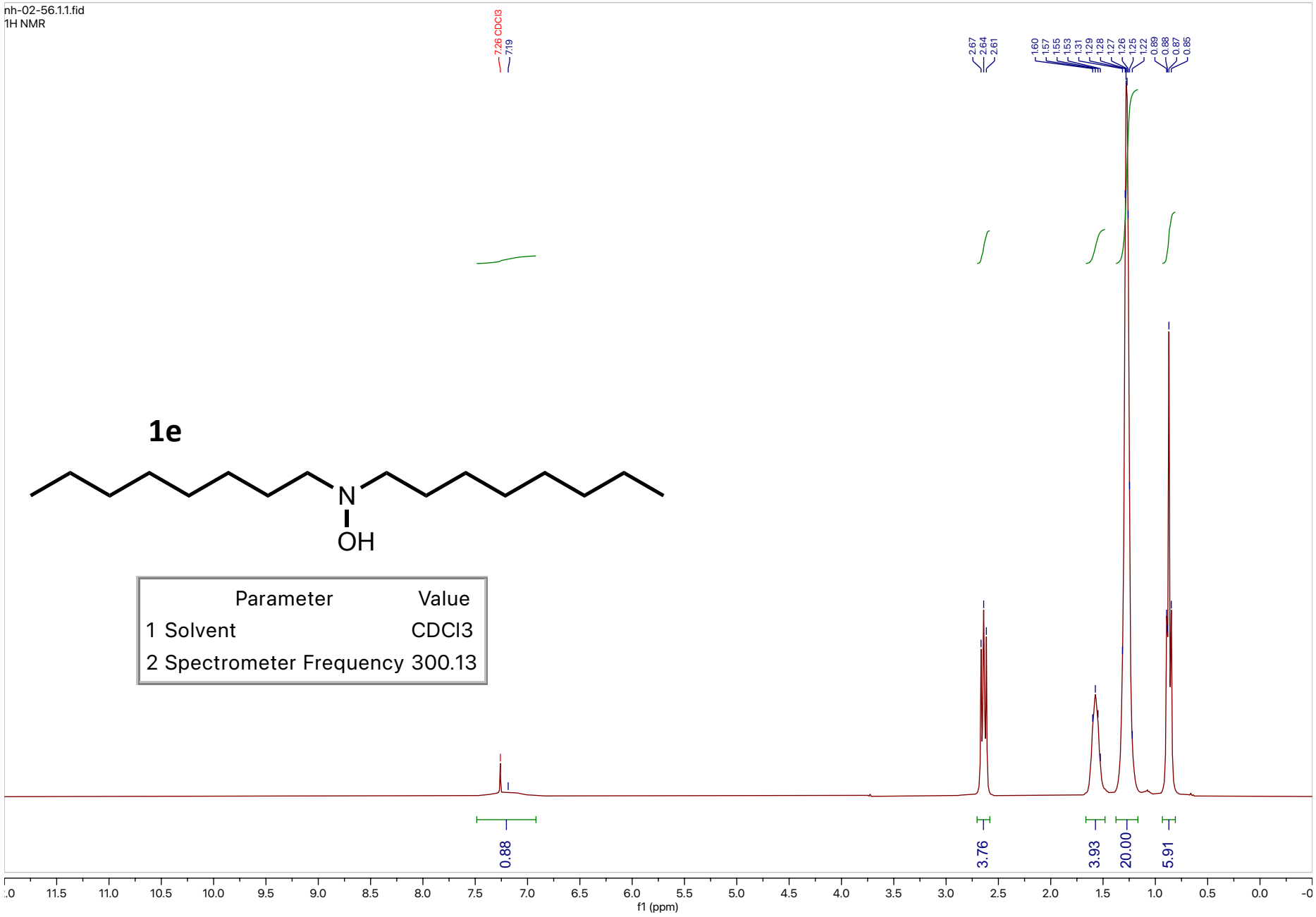
32. Trace aldehyde can be observed in the spectra of **3a**, **3h**, **3j**, **3k** and **3n**. We hypothesize this arises from the slow oxidation of the secondary hydroxylamine starting materials to the nitrone and subsequent hydrolysis under non-anhydrous conditions. For examples, see: (a) T. B. Nguyen, A. Martel, R. Dhal and G. Dujardin, *Synthesis* 2009, **18**, 3174-3176. DOI: 10.1055/s-0029-1216932. (b) F. Clemente, C. Matassini, A. Goti, A. Morrone, P. Paoli and F. Cardona, *ACS Med. Chem. Lett.* 2009, **10**, 621-626. DOI: 10.1021/acsmedchemlett.8b00602.

8. ^{1}H and ^{13}C NMR Spectra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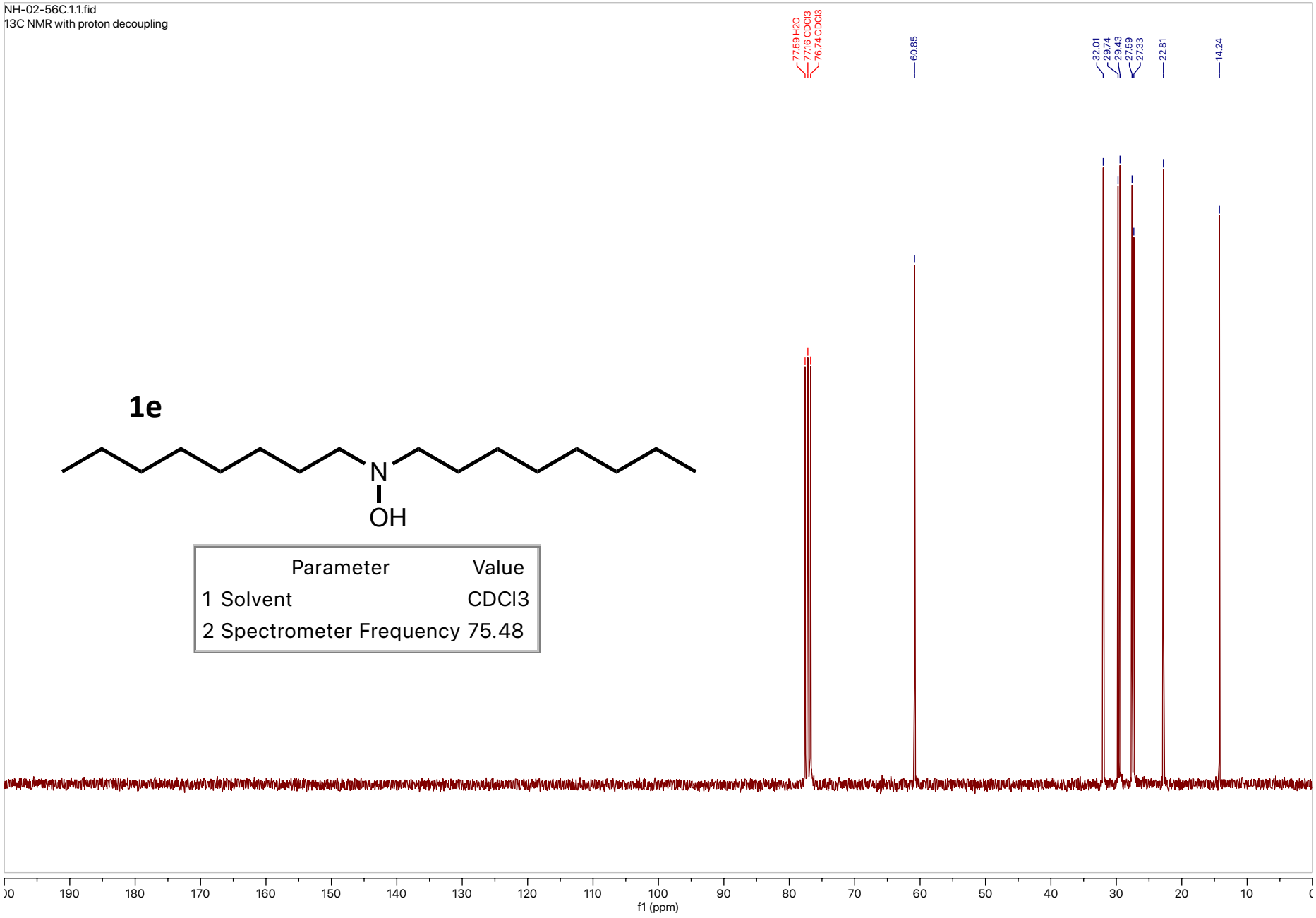
1H NMR
7.26 CDCl3
7.19
2.67
2.64
2.61
1.60
1.57
1.55
1.53
1.31
1.29
1.28
1.27
1.26
1.25
1.22
0.89
0.88
0.87
0.85
1e
N
OH
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.13
0.88
3.76
3.93
20.00
5.91
f1 (ppm)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76.74 CDCl3
60.85
32.01
29.74
29.43
27.59
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22.81
14.24
1e
N
OH
Parameter Value
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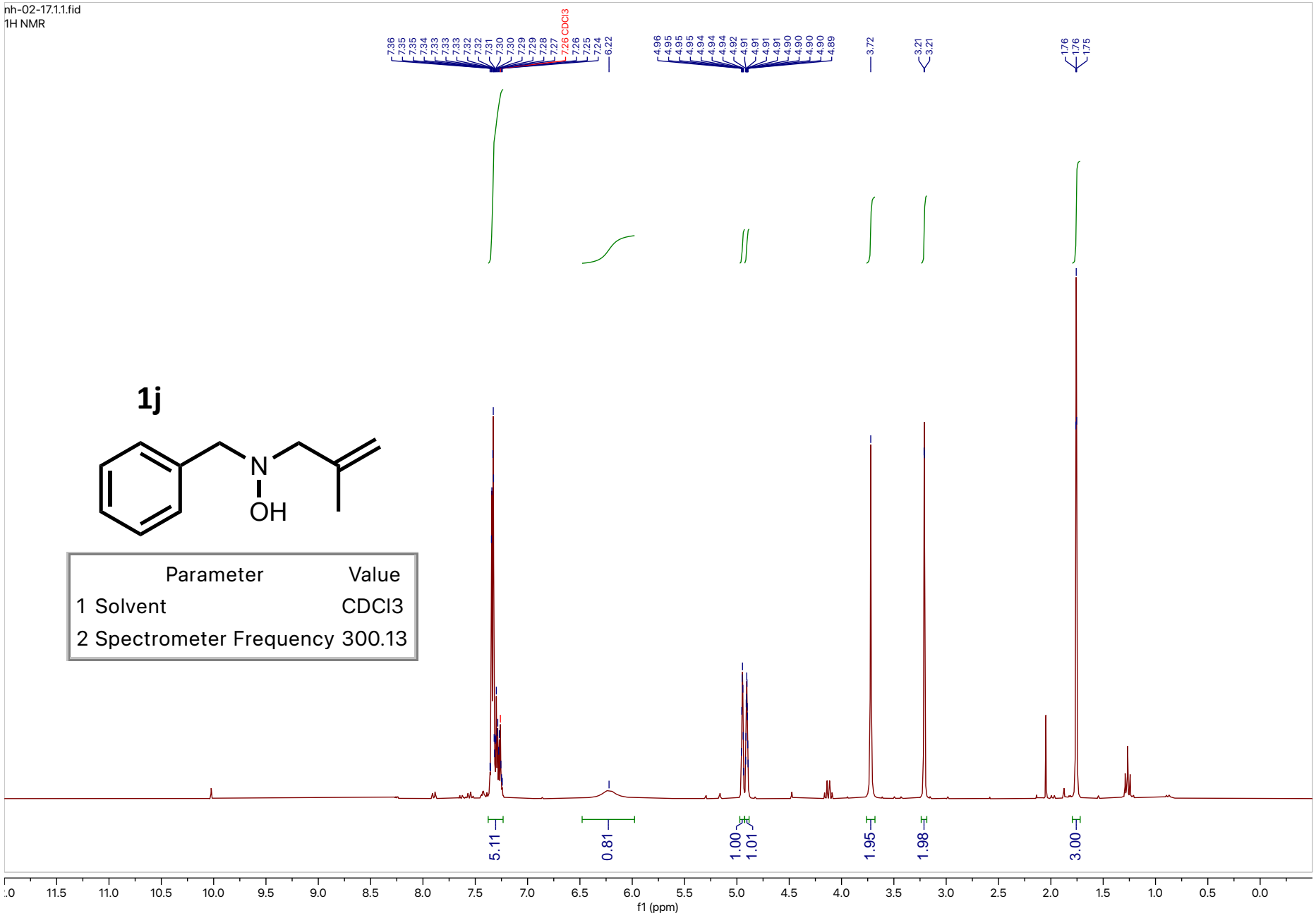
f1 (ppm)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1j
N
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f1 (ppm)

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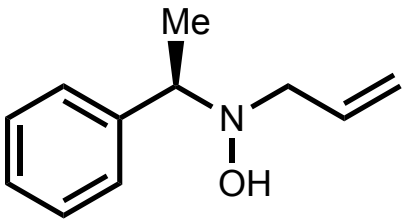
1H NMR

1l



Parameter	Value
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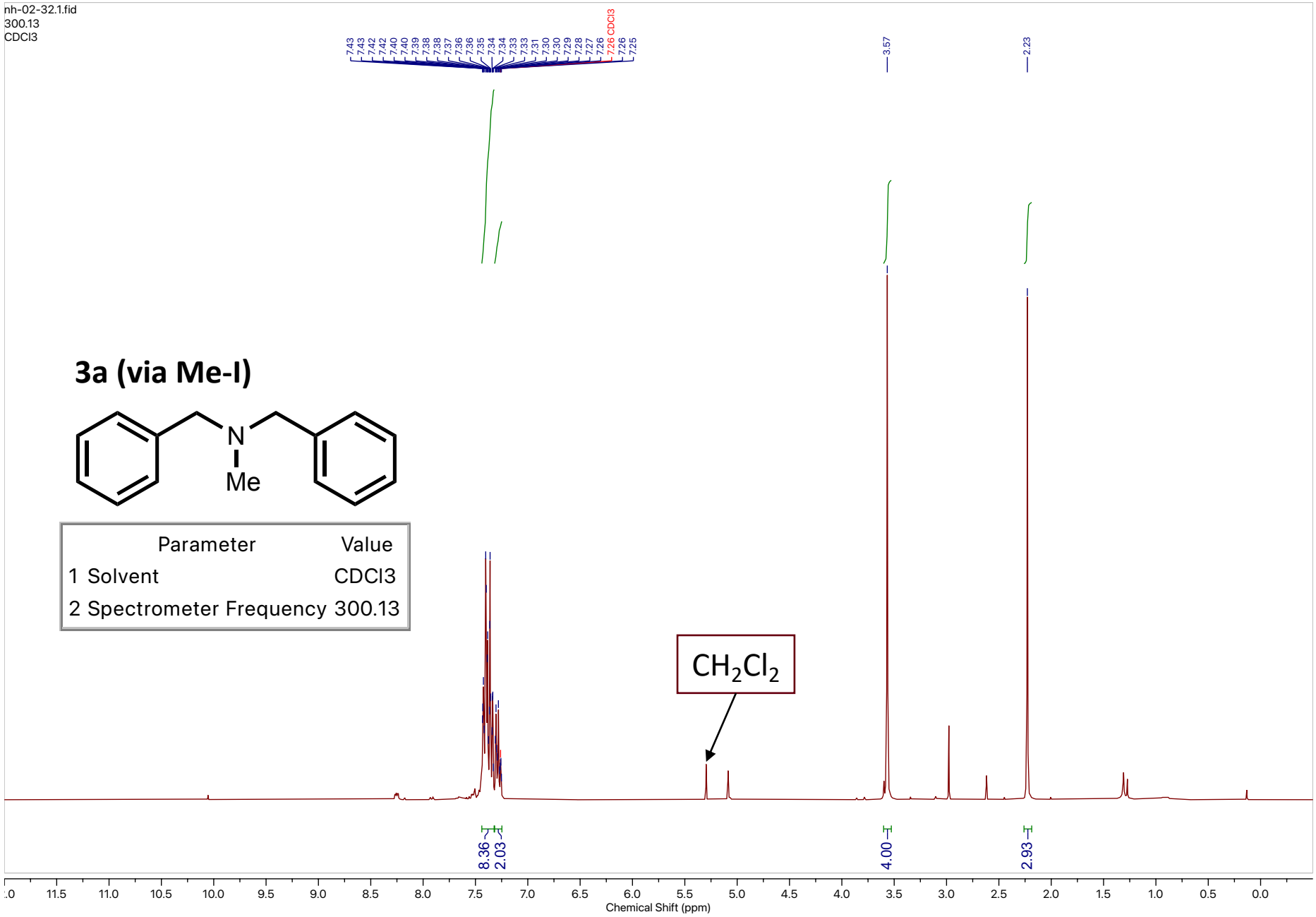
2 Spectrometer Frequency	300.33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300.13
CDCl3
7.43
7.43
7.42
7.42
7.40
7.40
7.39
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7.37
7.36
7.36
7.35
7.34
7.34
7.33
7.33
7.31
7.30
7.30
7.29
7.28
7.27
7.26
726 CDCl3
7.26
7.25
3.57
2.23
3a (via Me-I)
N
Me
Parameter Value
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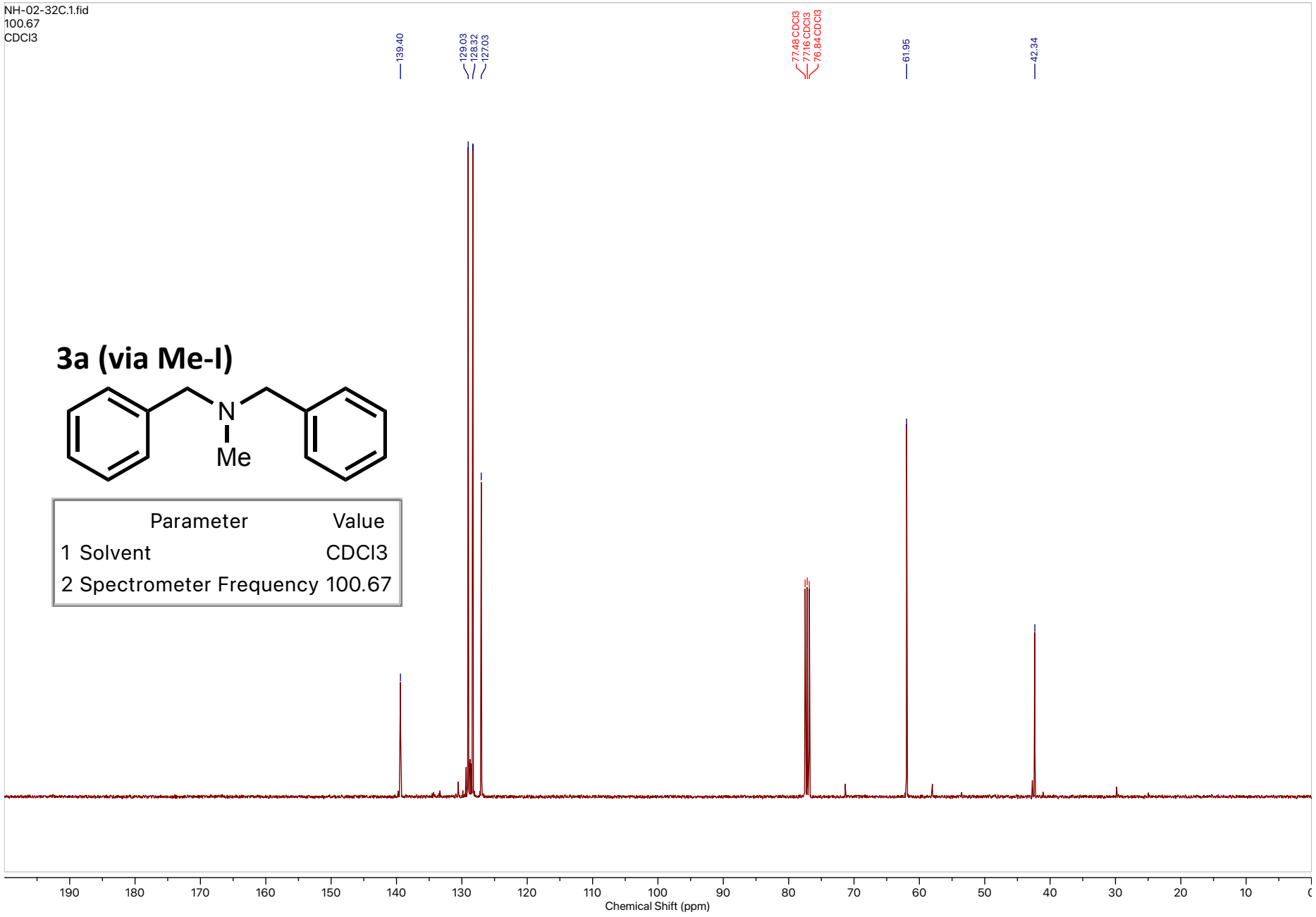
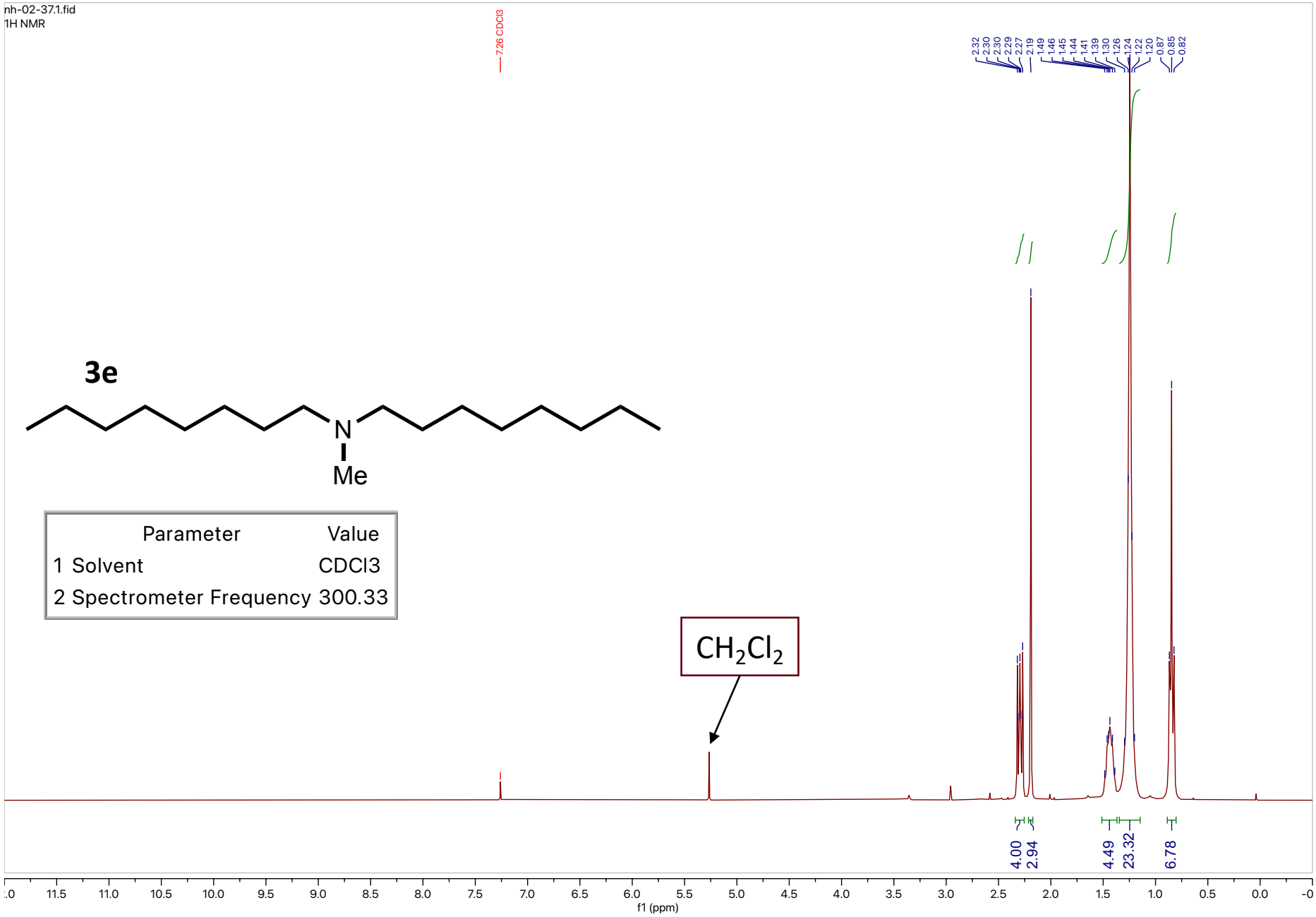
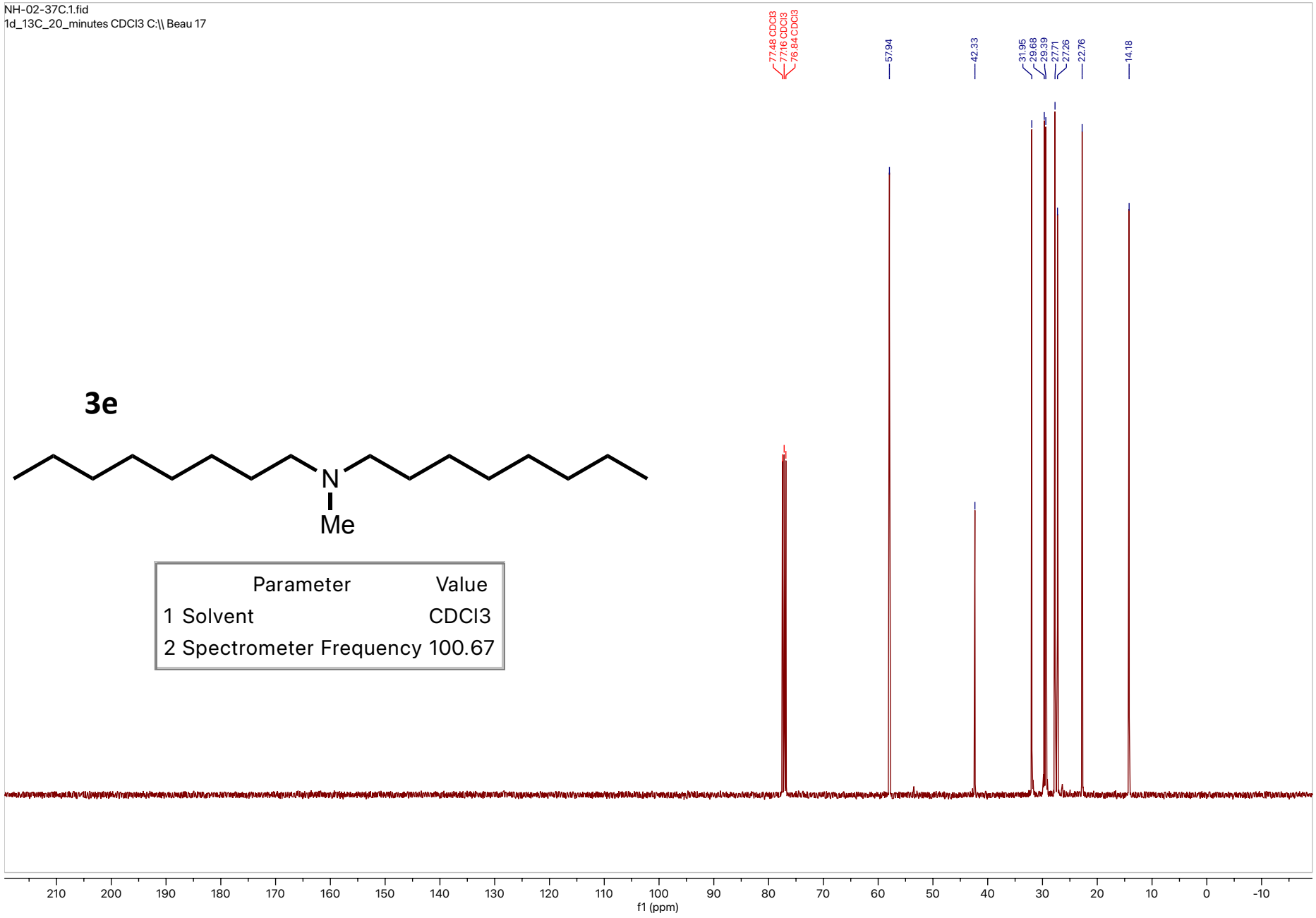
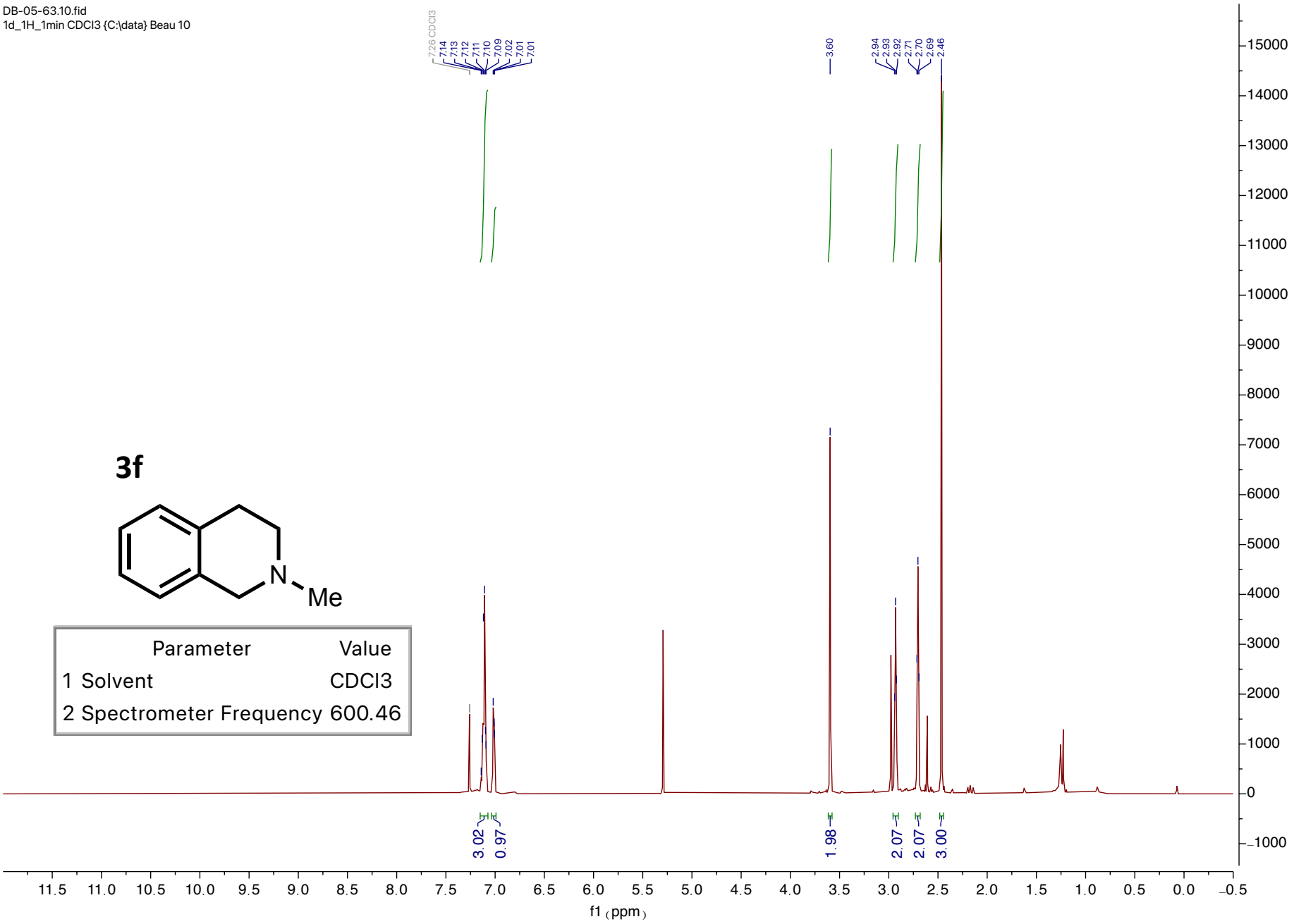
CH2Cl2
8.36
2.03
4.00
2.93
Chemical Shift (ppm)


NH-02-32C.1.fid
100.67
CDCl3
139.40
129.03
128.32
127.03
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
61.95
42.34
3a (via Me-I)
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
Chemical Shift (ppm)


nh-02-37.1.fid
1H NMR
7.26 CDCl3
2.32
2.30
2.30
2.29
2.27
2.19
1.49
1.46
1.45
1.44
1.41
1.39
1.30
1.26
1.24
1.22
1.20
0.87
0.85
0.82
3e
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.33
CH2Cl2
4.00
2.94
4.49
23.32
6.78
f1 (ppm)


NH-02-37C.1.fid
1d_13C_20_minutes CDCl3 C:\\ Beau 17
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
57.94
42.33
31.95
29.68
29.39
27.71
27.26
22.76
14.18
3e
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

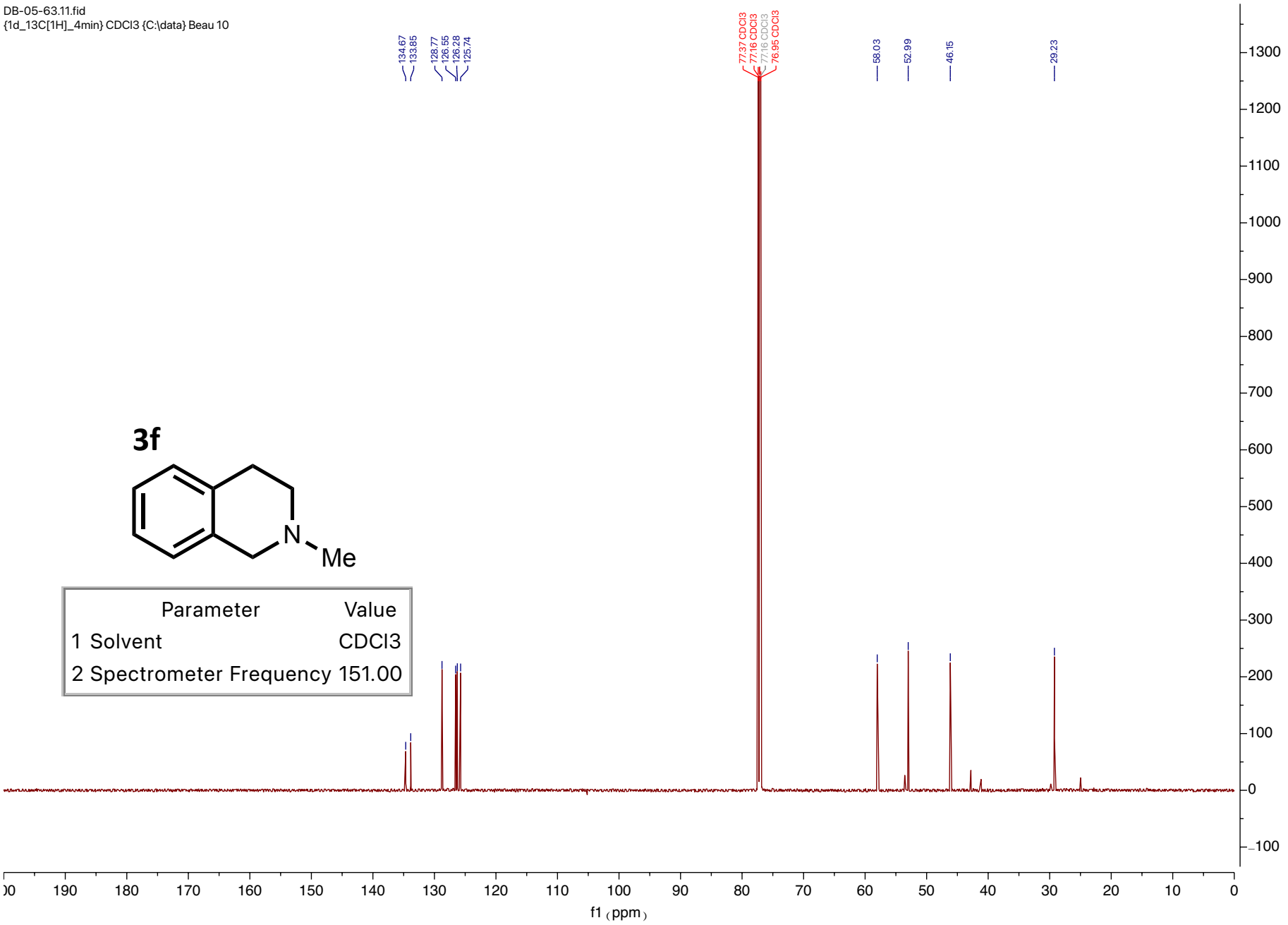
DB-05-63.10.fid
1d_1H_1min CDCl3 {C:\data} Beau 10

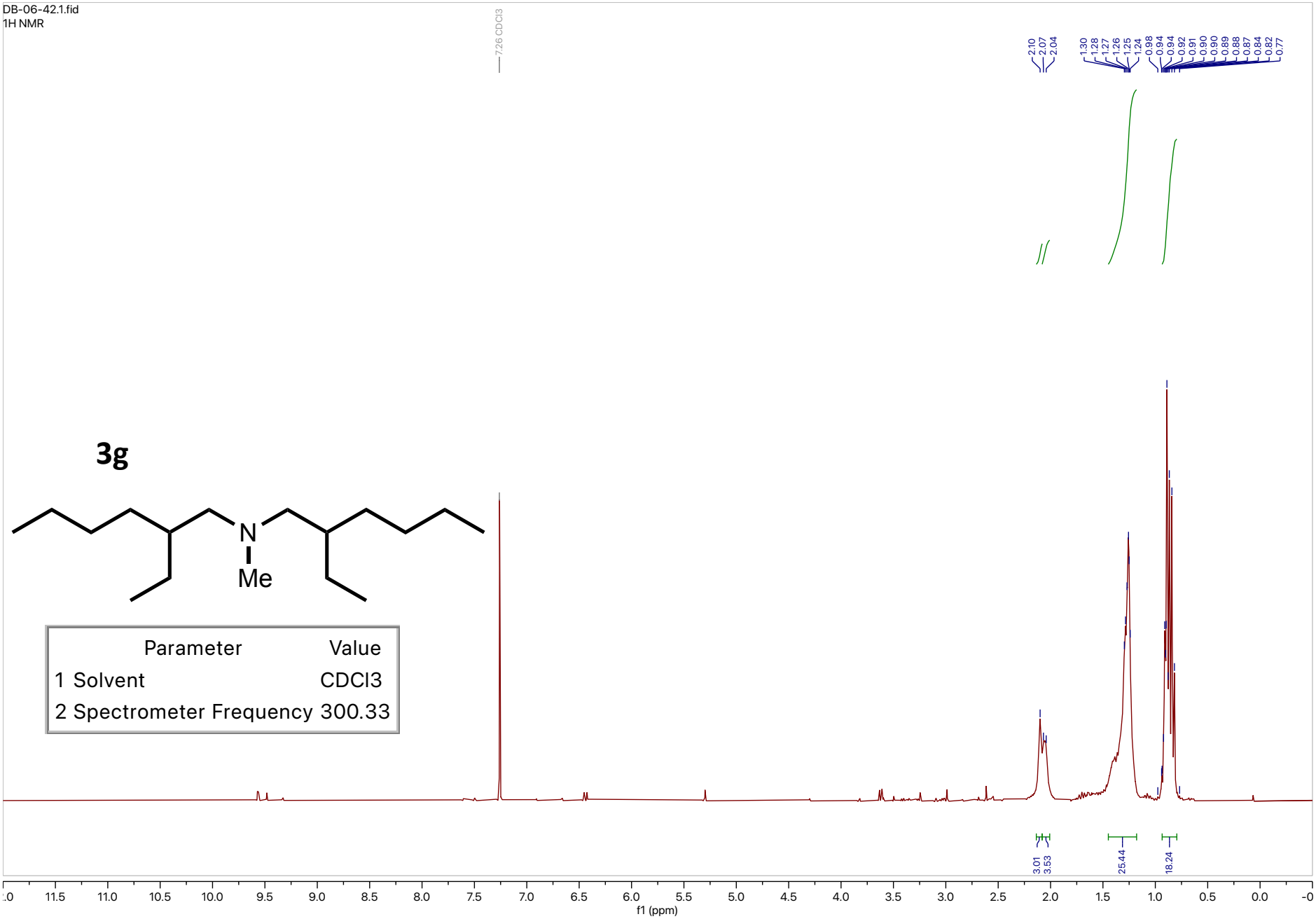


3f

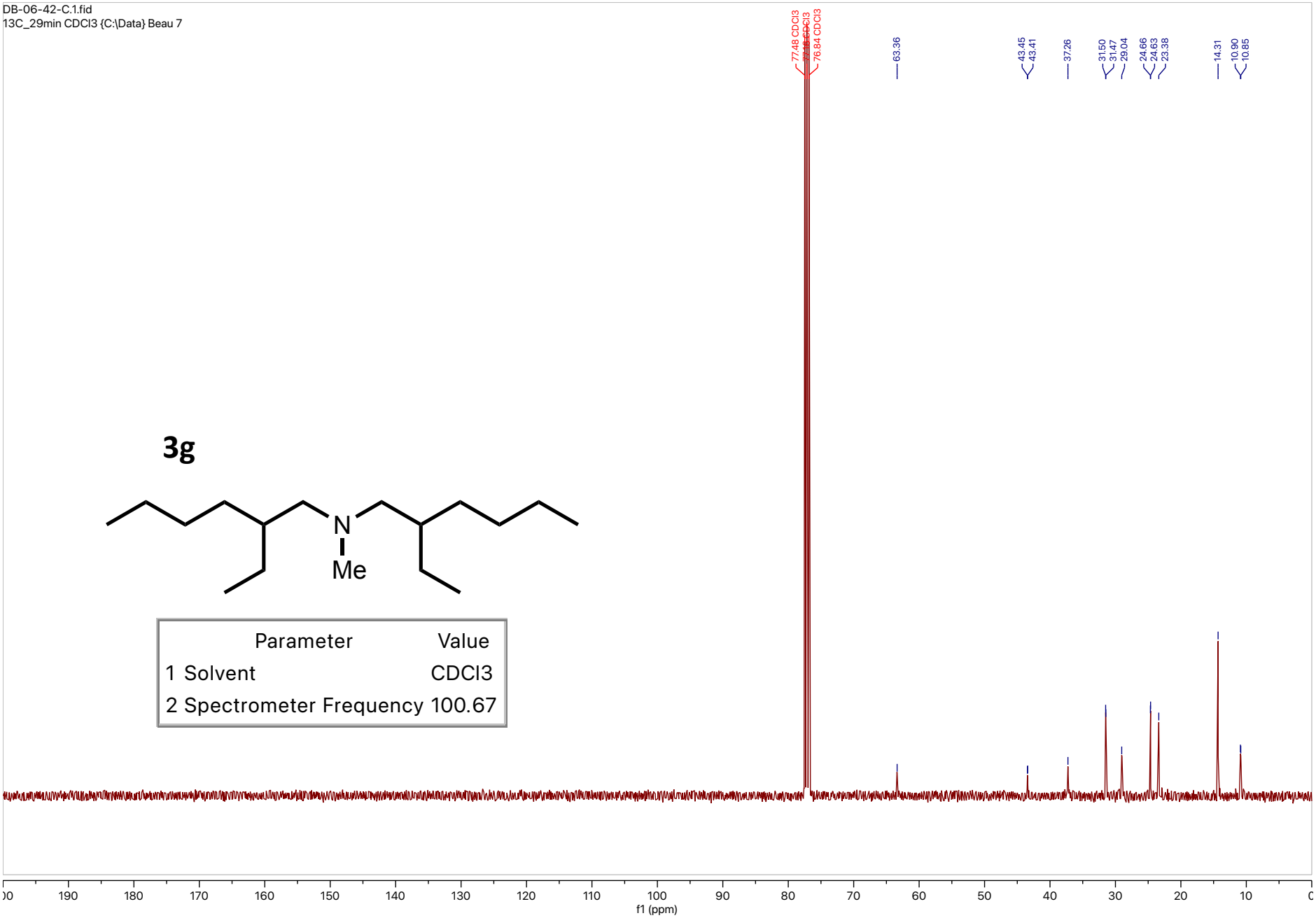
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	600.46

DB-05-63.11.fid
{1d_13C[1H]_4min} CDCl3 {C:\data} Beau 10



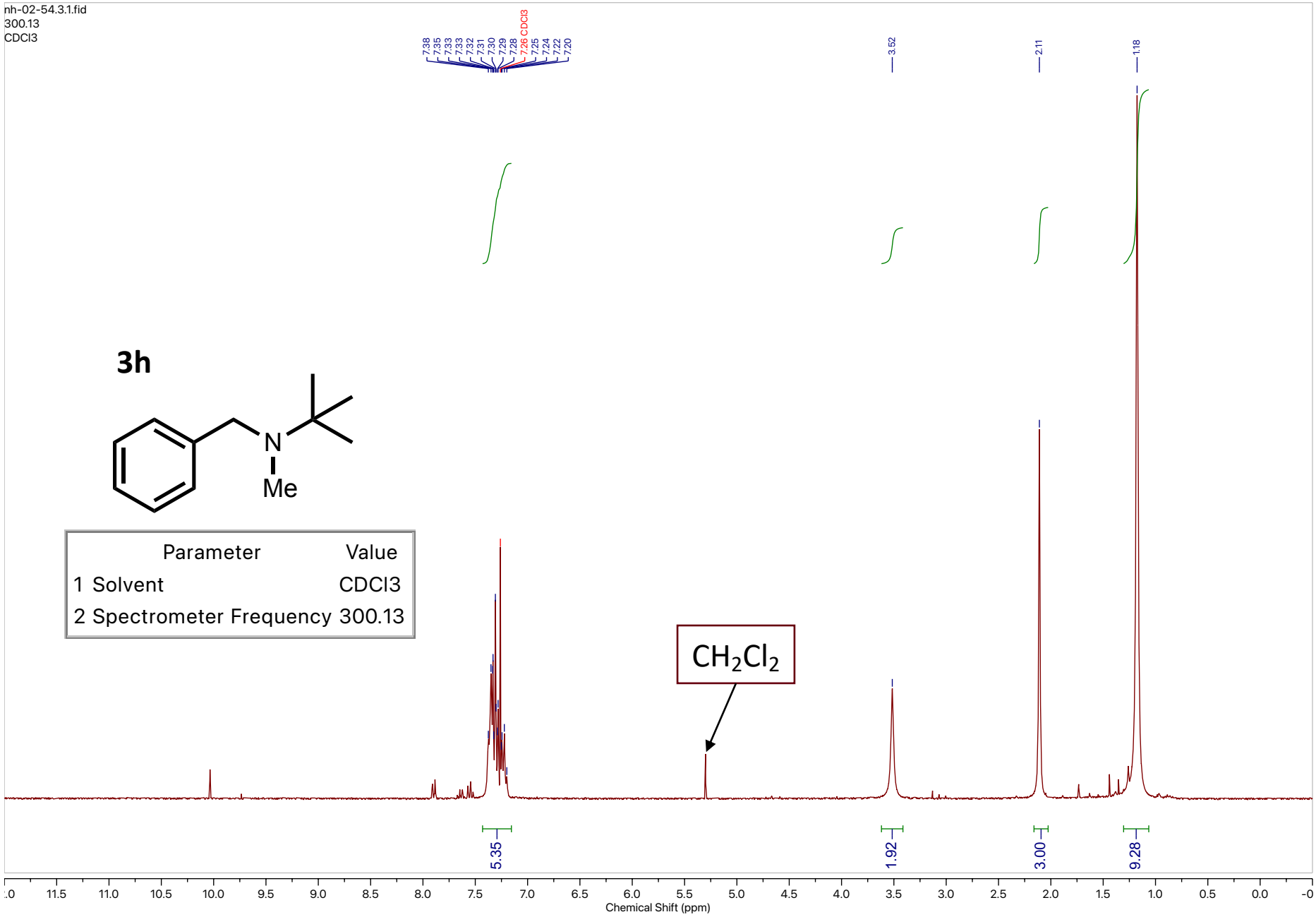

DB-06-42.1.fid
1H NMR
7.26 CDCl3
2.10
2.07
2.04
1.30
1.28
1.27
1.26
1.25
1.24
0.98
0.94
0.94
0.92
0.91
0.90
0.90
0.89
0.88
0.87
0.84
0.82
0.77
3g
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.33
3.01
3.53
25.44
18.24
f1 (ppm)

DB-06-42-C.1.fid
13C_29min CDCl3 {C:\Data} Beau 7

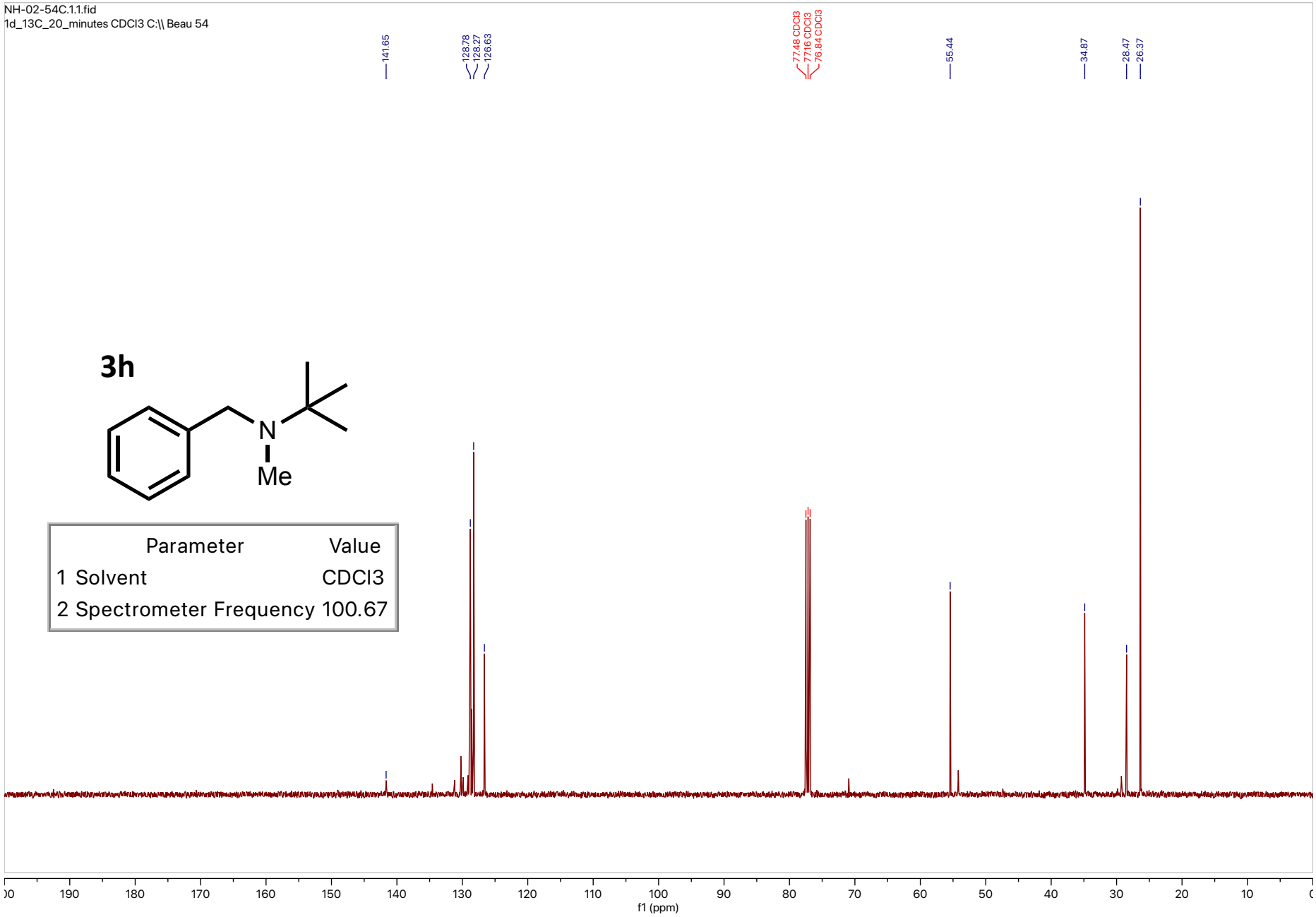


3g

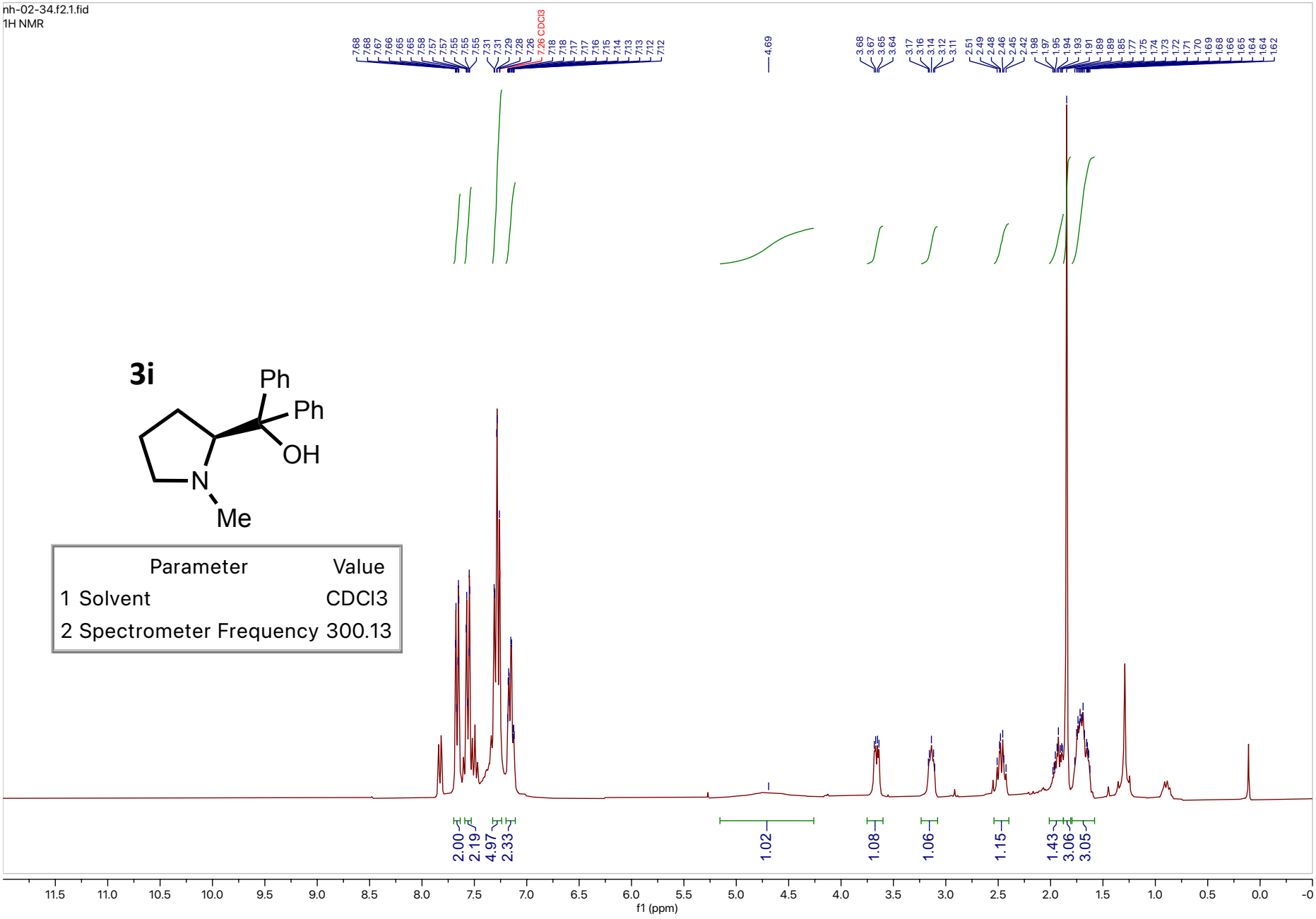
Parameter	Value
1 Solvent	CDCl3
2 Spectrometer Frequency	100.67


nh-02-54.3.1.fid
300.13
CDCl3
7.38
7.35
7.33
7.33
7.32
7.31
7.30
7.29
7.28
7.26 CDCl3
7.25
7.24
7.22
7.20
3.52
2.11
1.18
3h
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.13
CH2Cl2
5.35
1.92
3.00
9.28
Chemical Shift (ppm)

NH-02-54C.1.1.fid
1d_13C_20_minutes CDCl3 C:\\ Beau 54



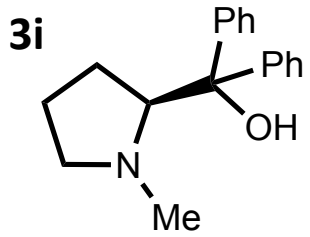
nh-02-34.f2.1.fid
1H NMR



NH-02-34C.F2.1.fid
1d_13C_20_minutes CDCl3 C:\\ Beau 10

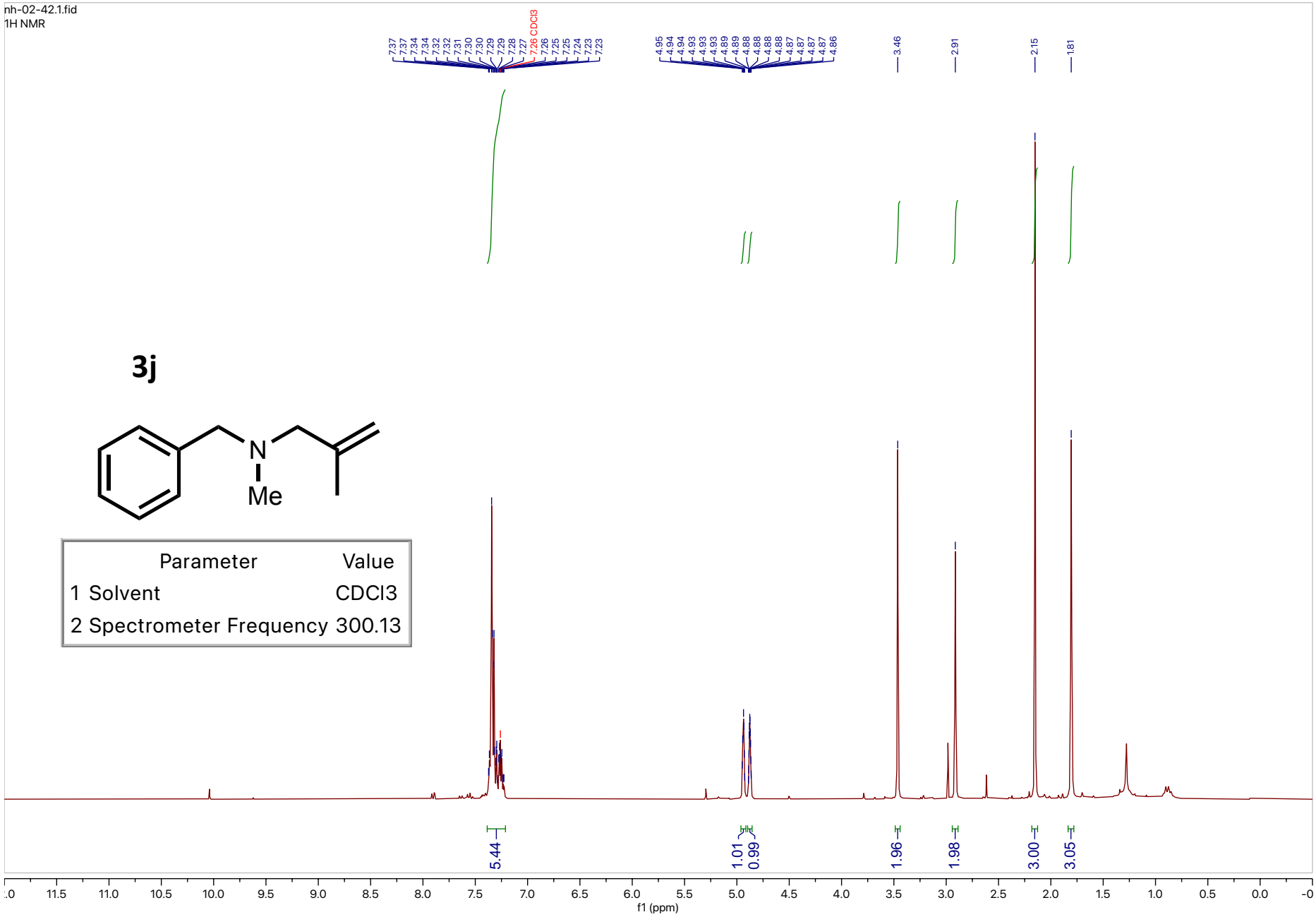
148.24
146.78
128.37
128.12
128.11
126.25
125.59
125.53
77.56
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
72.17
59.25
43.08
29.96
24.10

3i

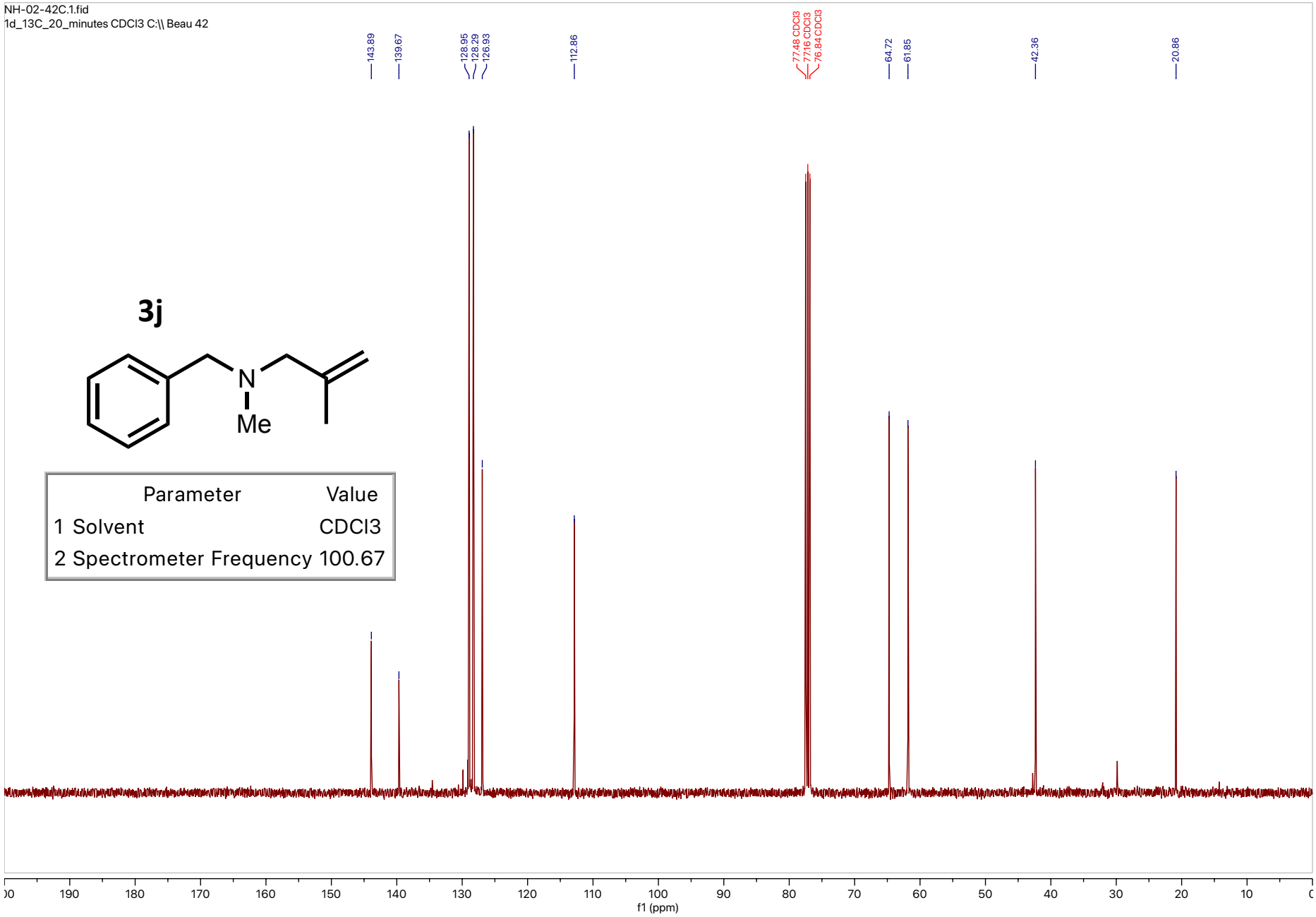


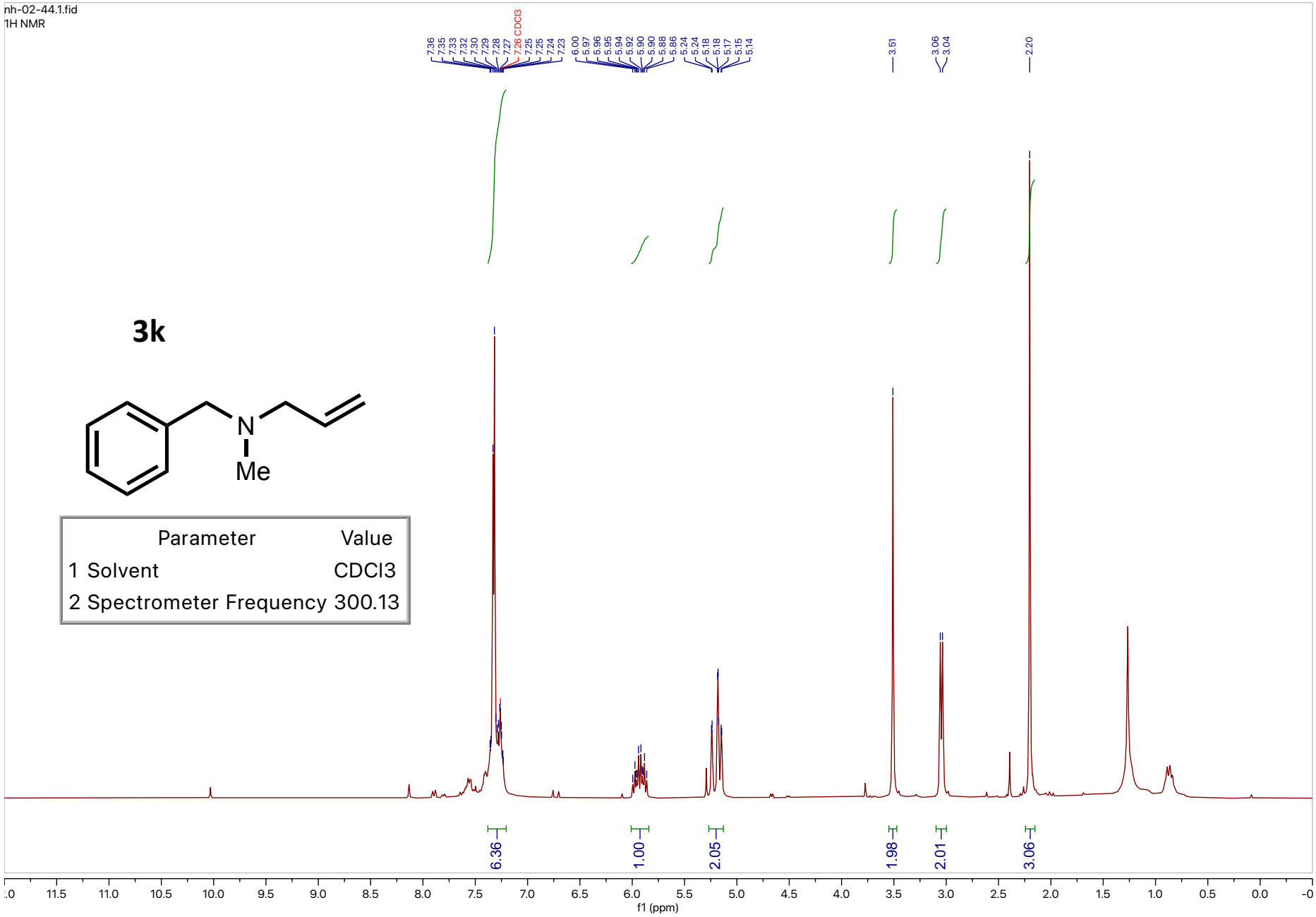
Parameter	Value
1 Solvent	CDCl3
2 Spectrometer Frequency	100.67

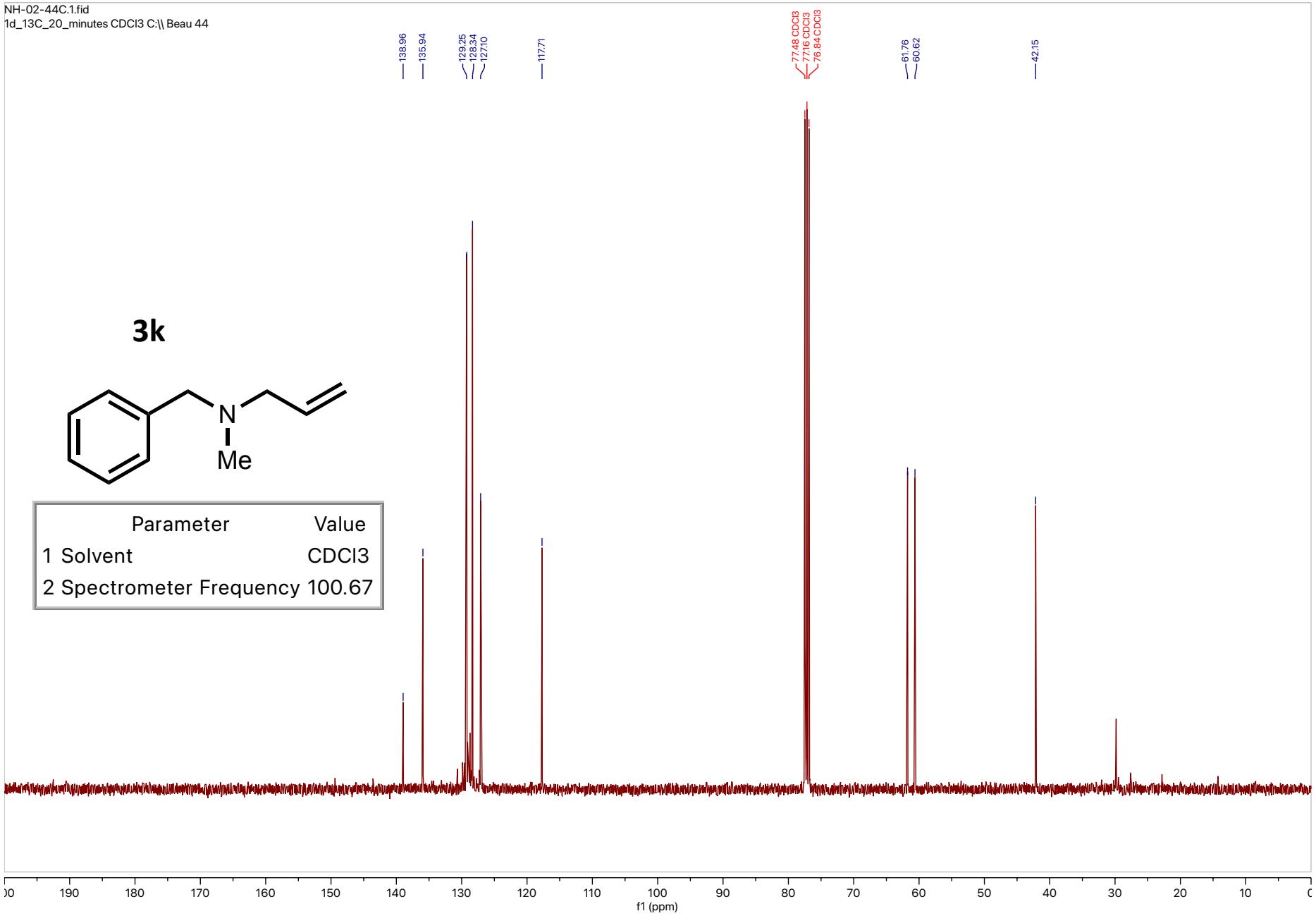
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

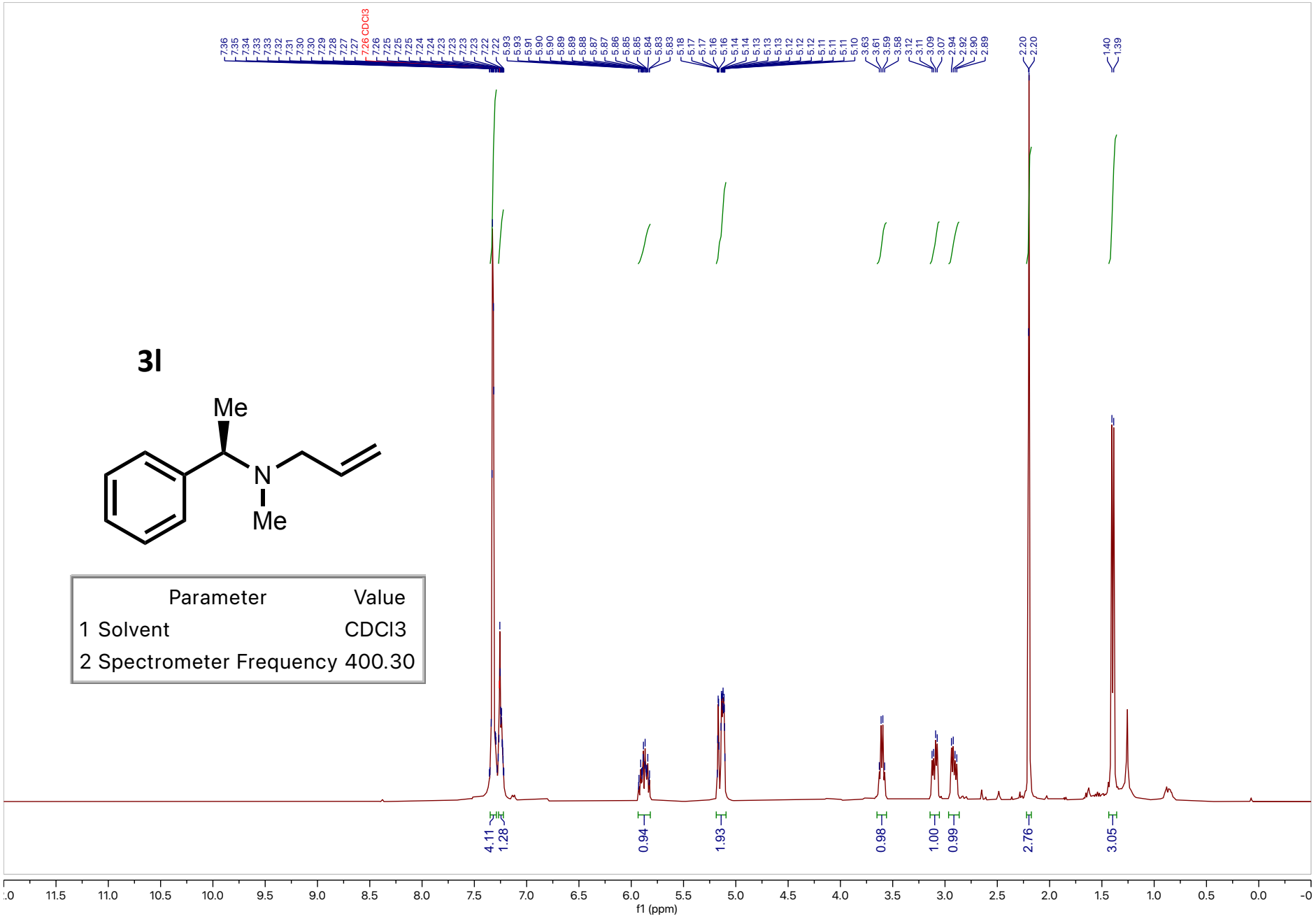

nh-02-42.1.fid
1H NMR
7.37 7.37 7.34 7.34 7.32 7.32 7.31 7.30 7.30 7.29 7.29 7.28 7.27 7.26 CDCl3 7.26 7.25 7.25 7.24 7.23 7.23
4.95 4.94 4.94 4.93 4.93 4.89 4.89 4.88 4.88 4.88 4.88 4.87 4.87 4.87 4.87 4.86
3.46
2.91
2.15
1.81
3j
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.13
5.44
1.01
0.99
1.96
1.98
3.00
3.05
f1 (ppm)

NH-02-42C.1.fid
1d_13C_20_minutes CDCl3 C:\\ Beau 42

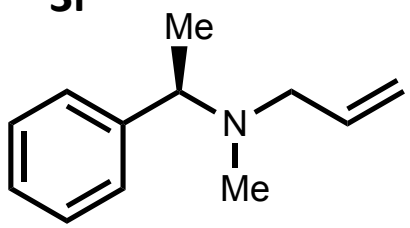



nh-02-44.1.fid
1H NMR
7.36
7.35
7.33
7.32
7.30
7.29
7.28
7.27
7.26 CDCl3
7.25
7.25
7.24
7.23
6.00
5.97
5.96
5.95
5.94
5.92
5.90
5.90
5.88
5.86
5.24
5.24
5.18
5.18
5.17
5.15
5.14
3.51
3.06
3.04
2.20
3k
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.13
6.36
1.00
2.05
1.98
2.01
3.06
f1 (ppm)

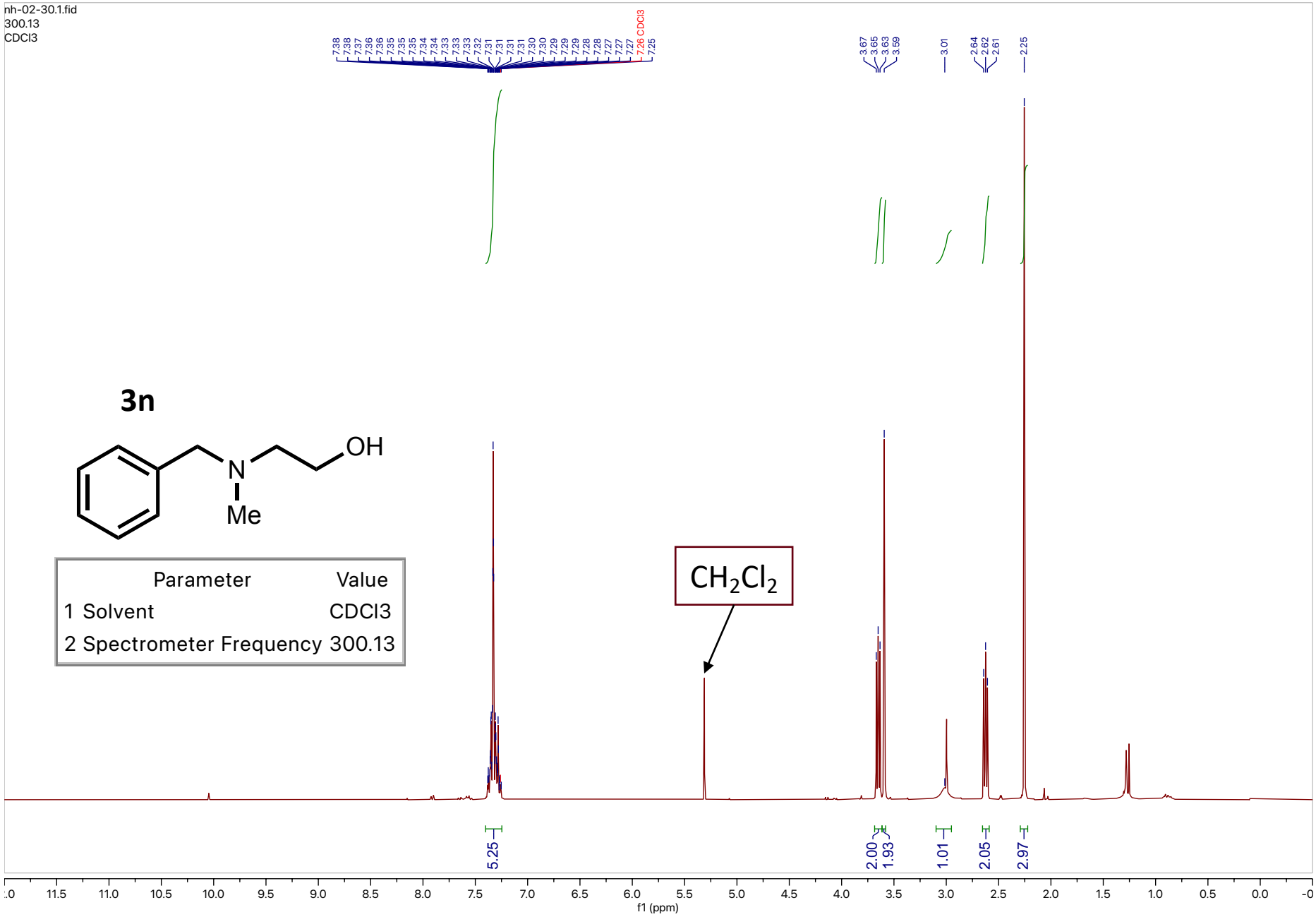

NH-02-44C.1.fid
1d_13C_20_minutes CDCl3 C:\\ Beau 44
138.96
135.94
129.25
128.34
127.10
117.71
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
61.76
60.62
42.15
3k
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)


3l
Me
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
7.26 CDCl3
4.11
1.28
0.94
1.93
0.98
1.00
0.99
2.76
3.05
f1 (ppm)

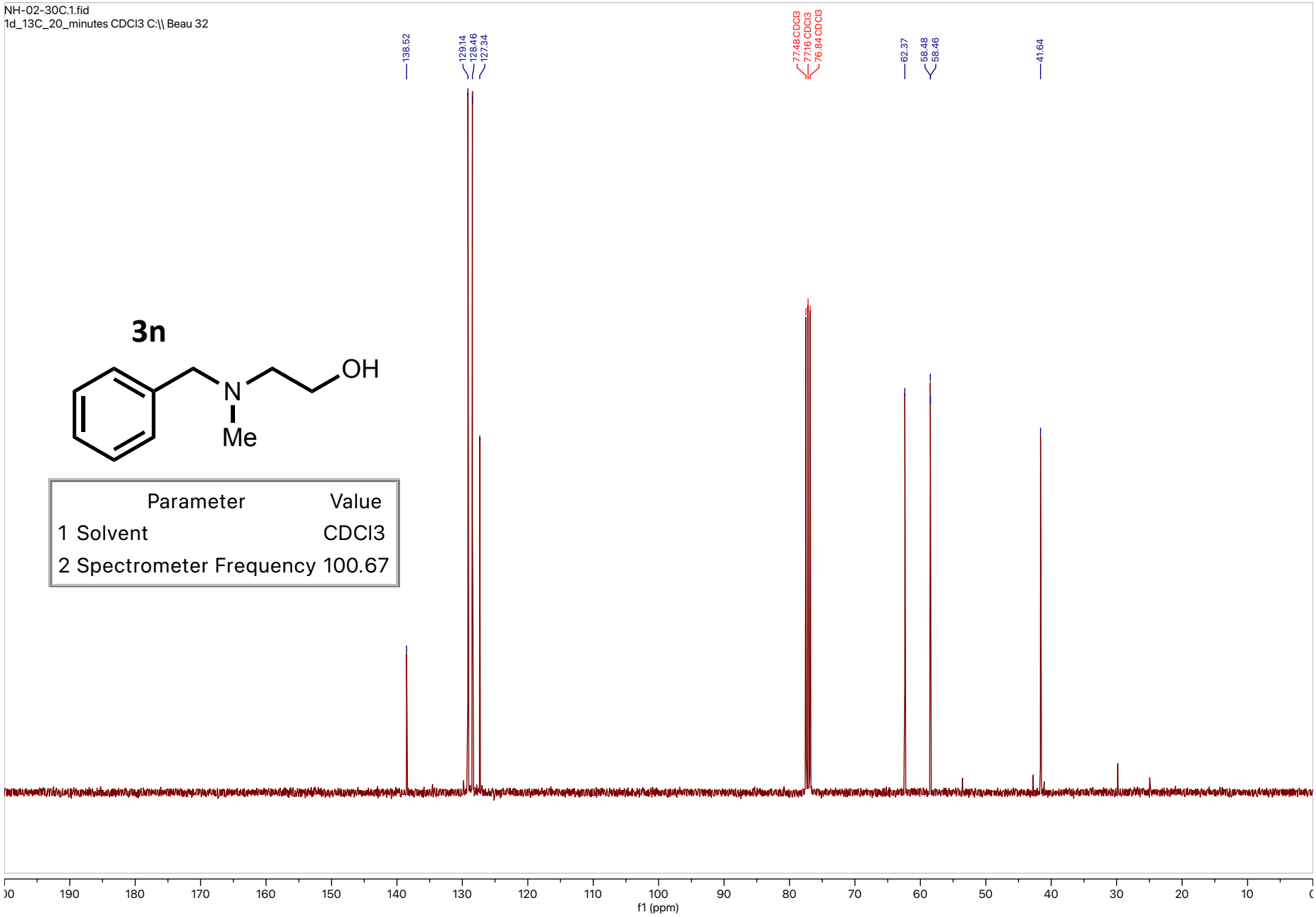
3l



Parameter	Value
1 Solvent	CDCl3
2 Spectrometer Frequency	100.67


nh-02-30.1.fid
300.13
CDCl3
7.38 7.38 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.33 7.32 7.31 7.31 7.31 7.31 7.30 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.27 7.27 7.26 CDCl3 7.25
3.67 3.65 3.63 3.59
3.01
2.64 2.62 2.61
2.25
3n
OH
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.13
CH2Cl2
5.25
2.00
1.93
1.01
2.05
2.97
f1 (ppm)

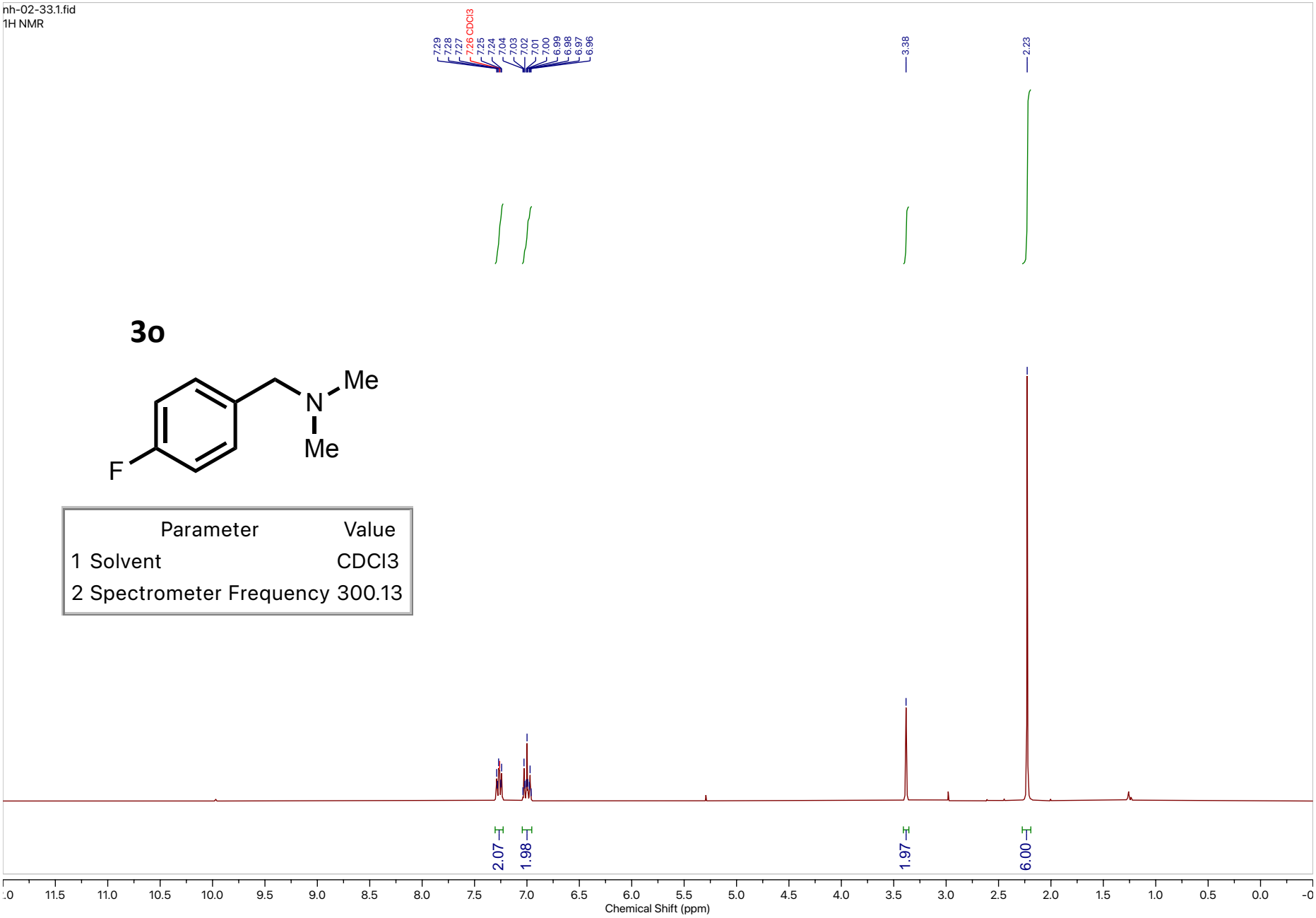
NH-02-30C.1.fid
1d_13C_20_minutes CDCl3 C:\\ Beau 32



3n

	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	100.67

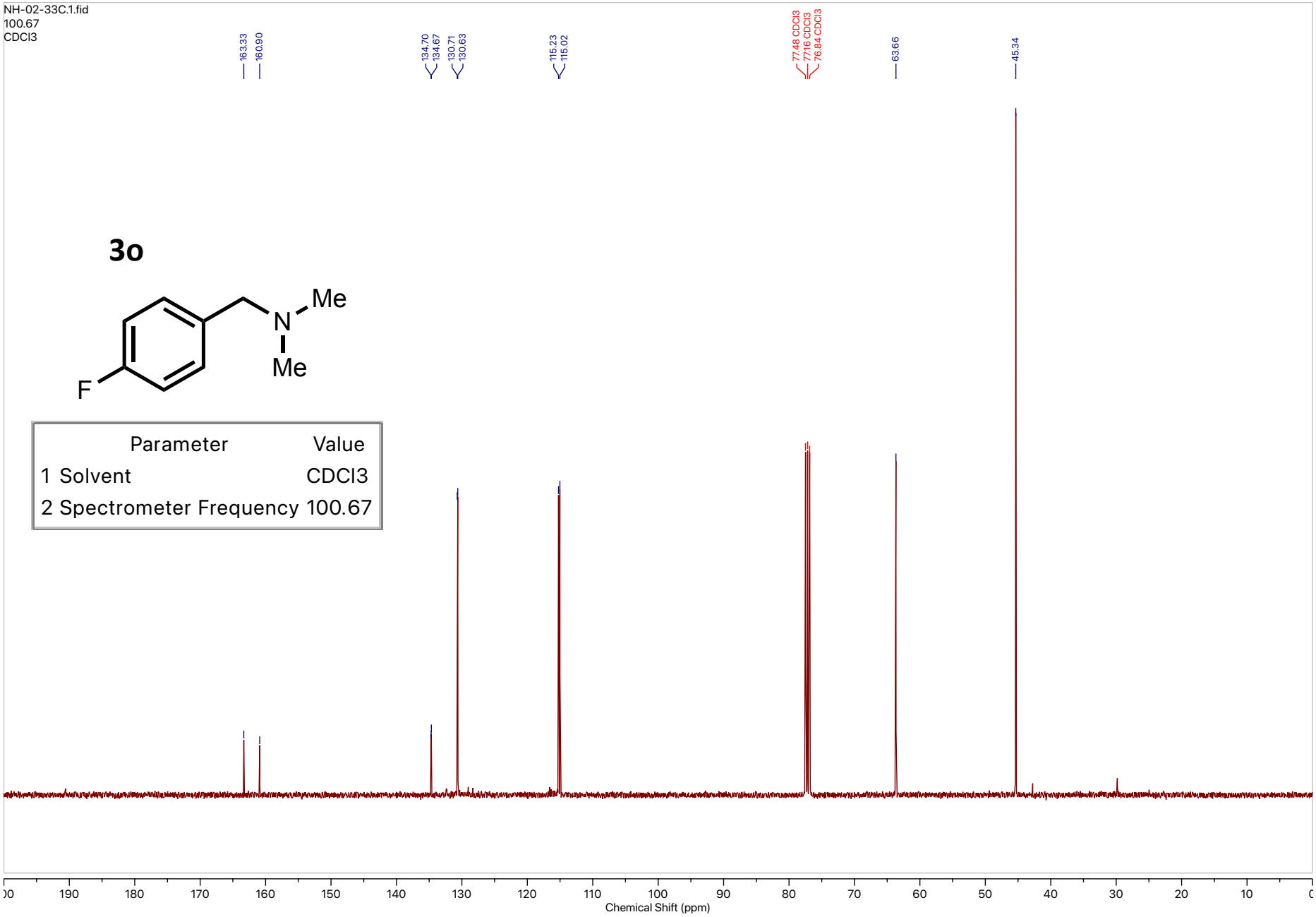
nh-02-33.1.fid
1H NMR

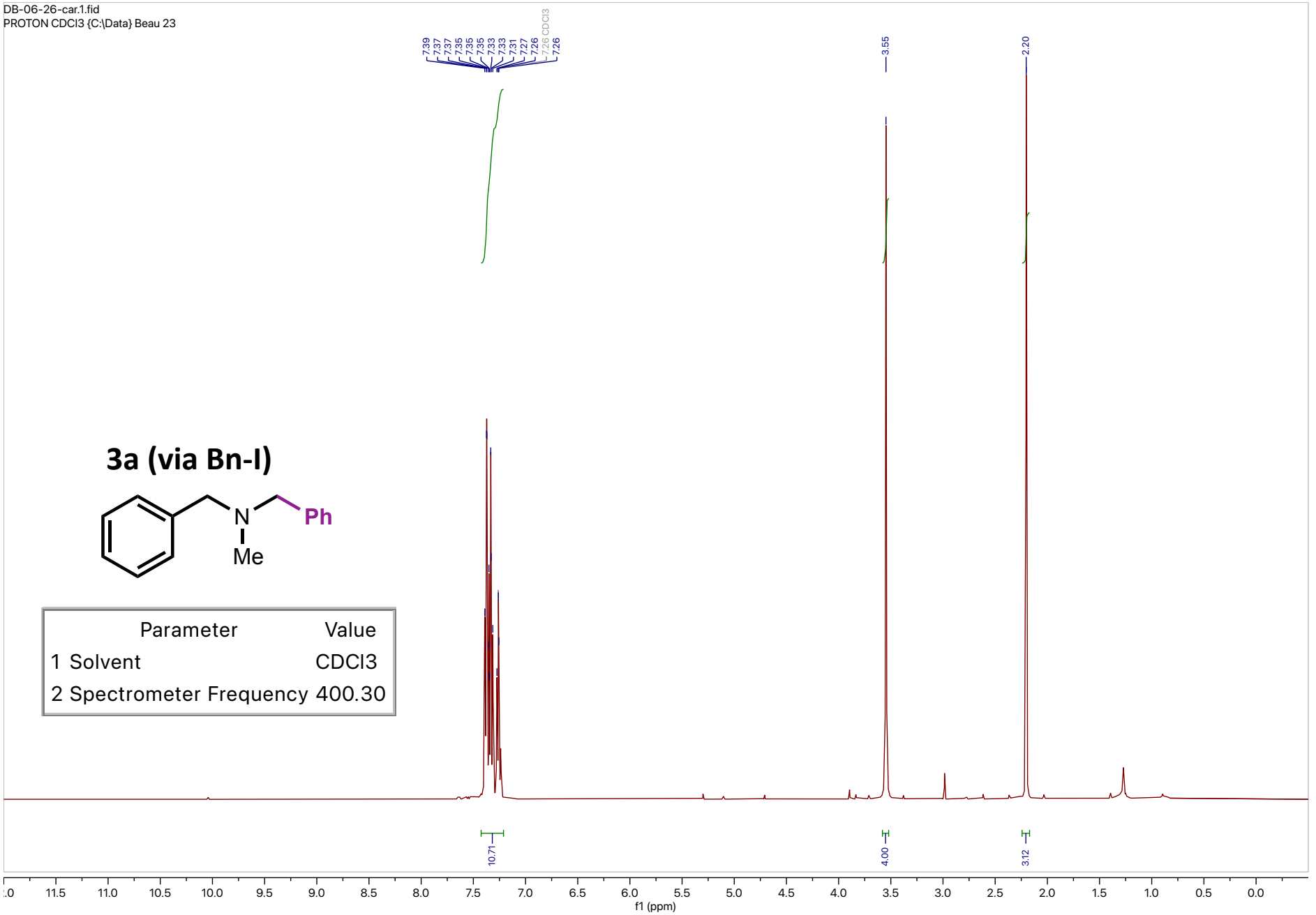


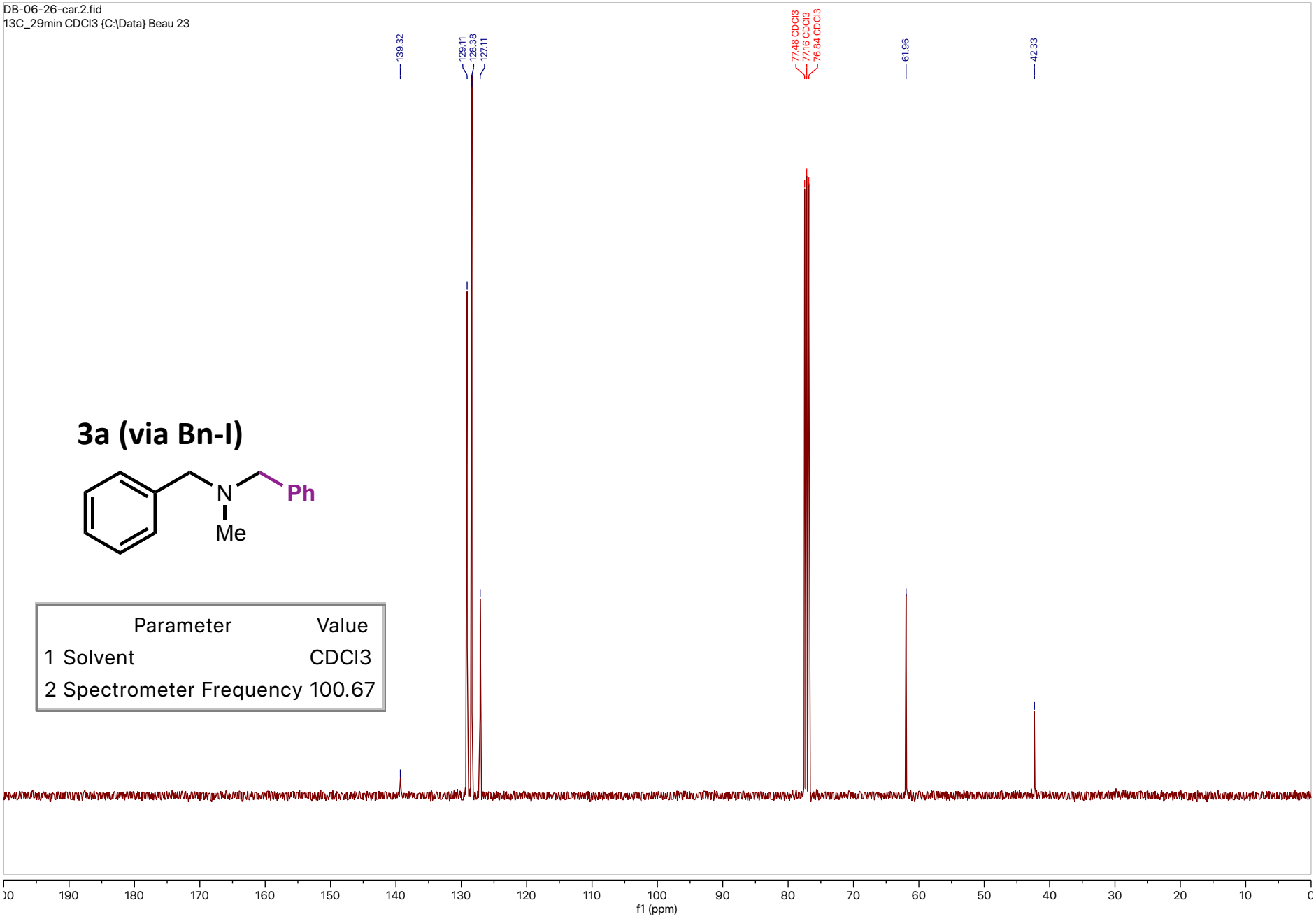
3o

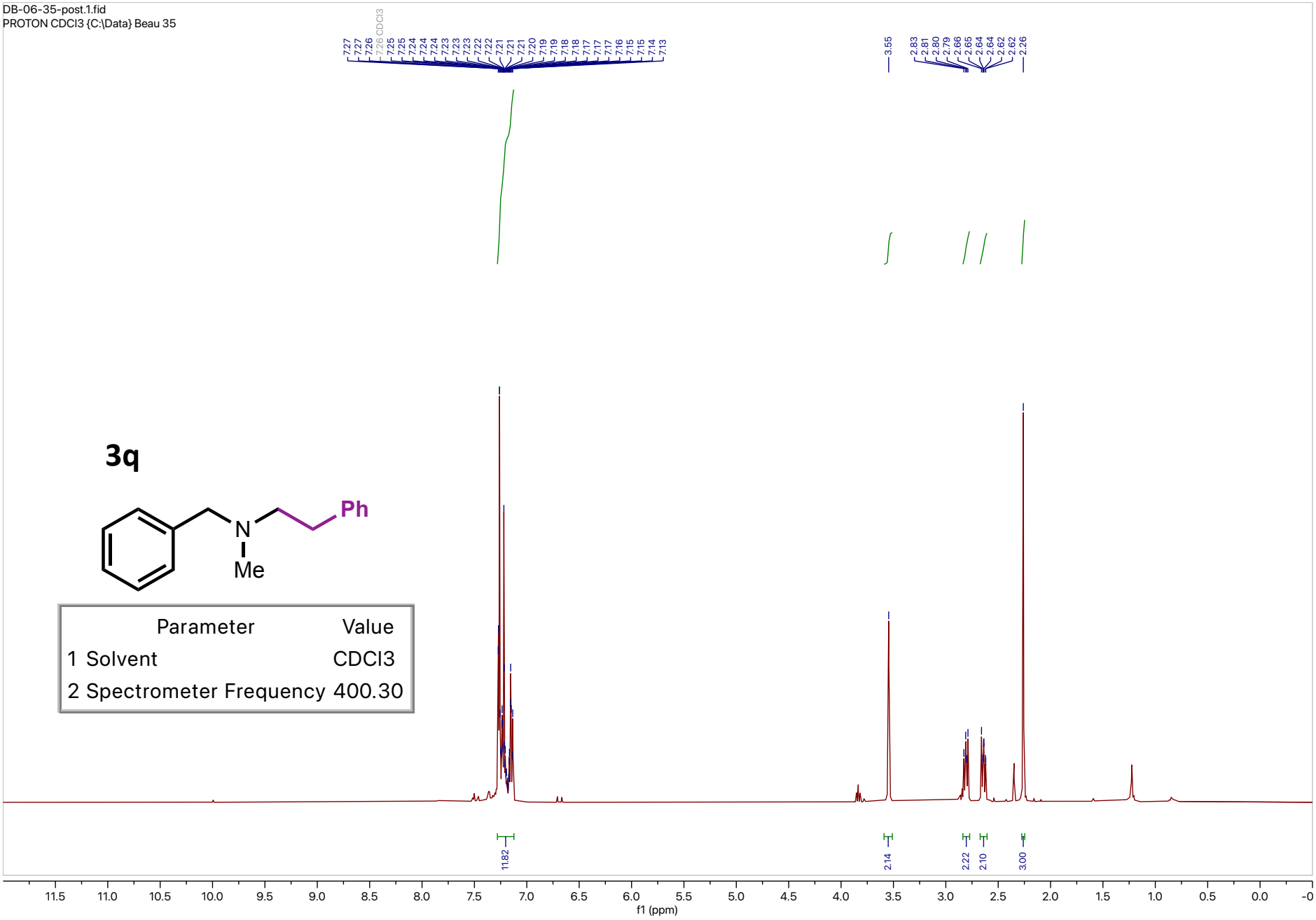
Parameter	Value
1 Solvent	CDCl3
2 Spectrometer Frequency	300.13

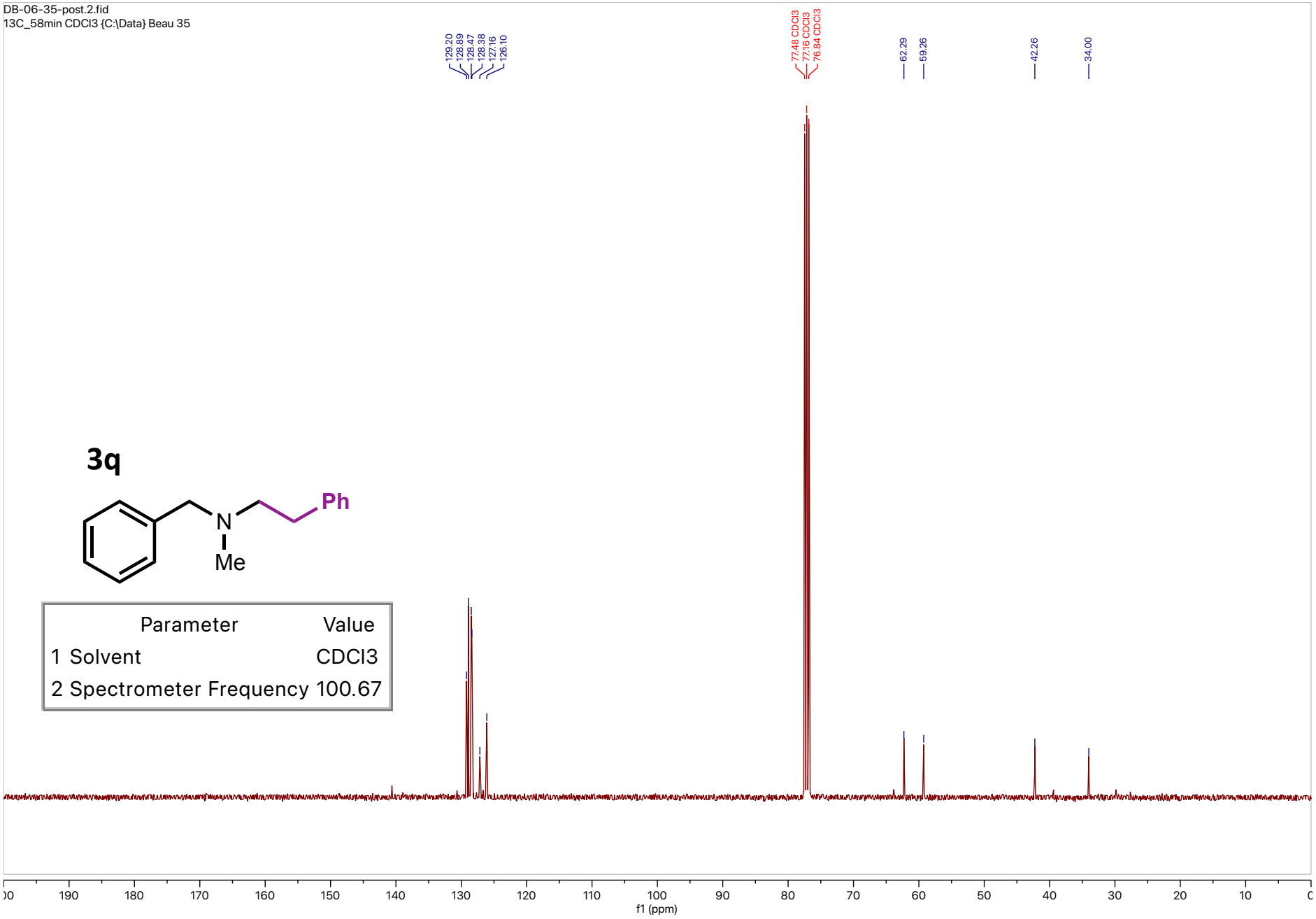
NH-02-33C.1.fid
100.67
CDCl3

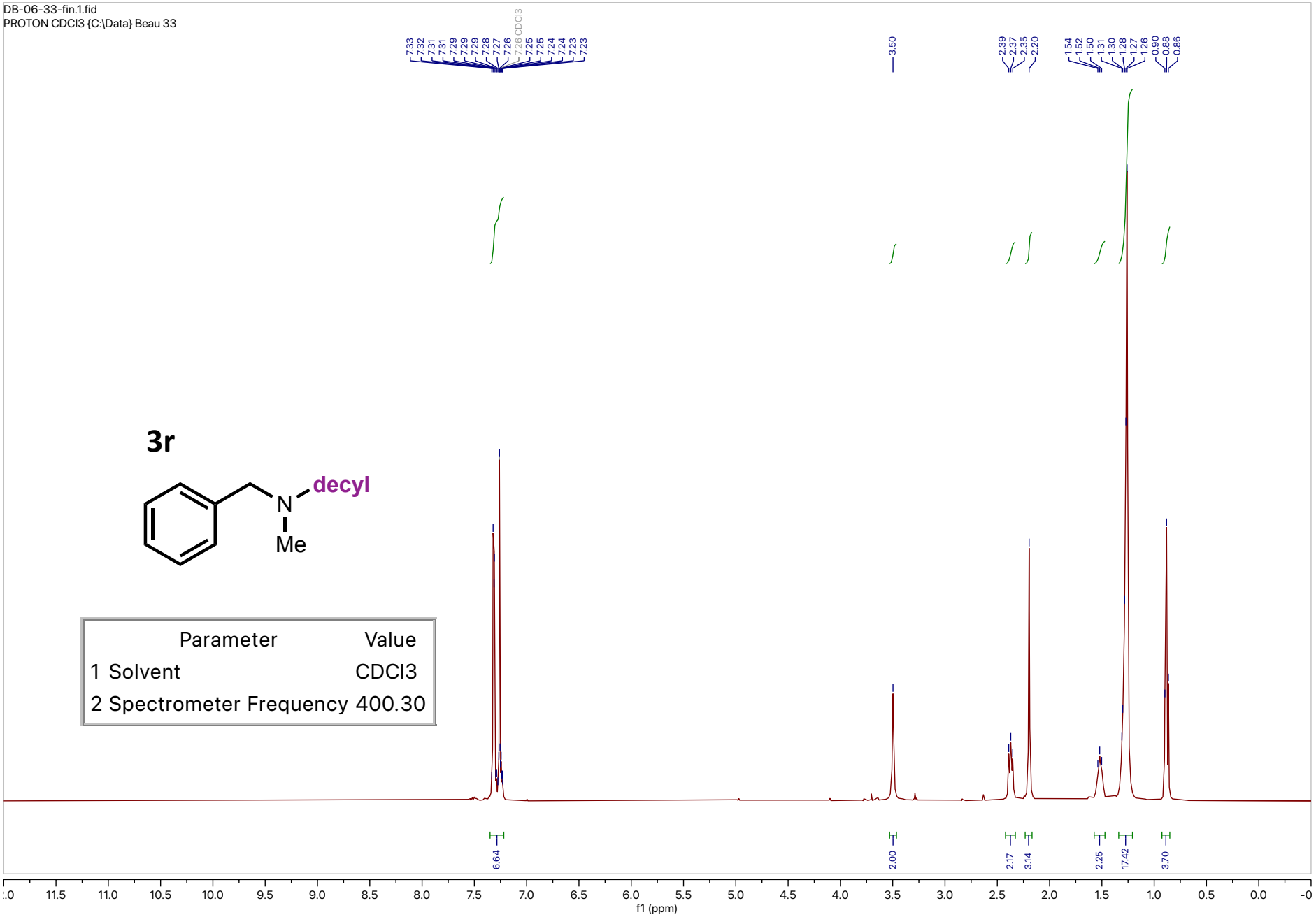


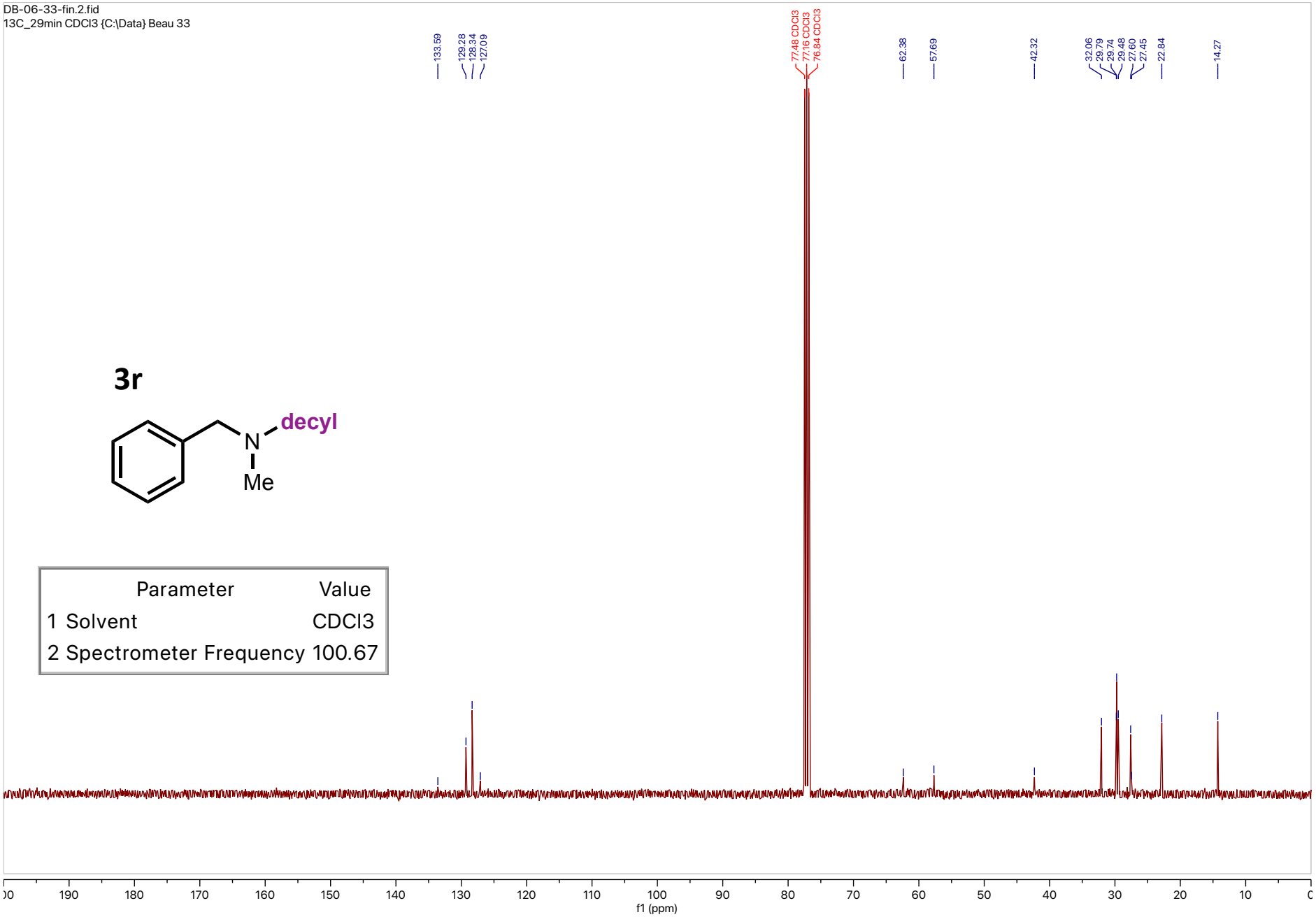

DB-06-26-car.1.fid
PROTON CDCl3 {C:\Data} Beau 23
7.39
7.37
7.37
7.35
7.35
7.33
7.33
7.31
7.27
7.26
7.26 CDCl3
7.26
3.55
2.20
3a (via Bn-I)
N
Ph
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
10.71
4.00
3.12
f1 (ppm)

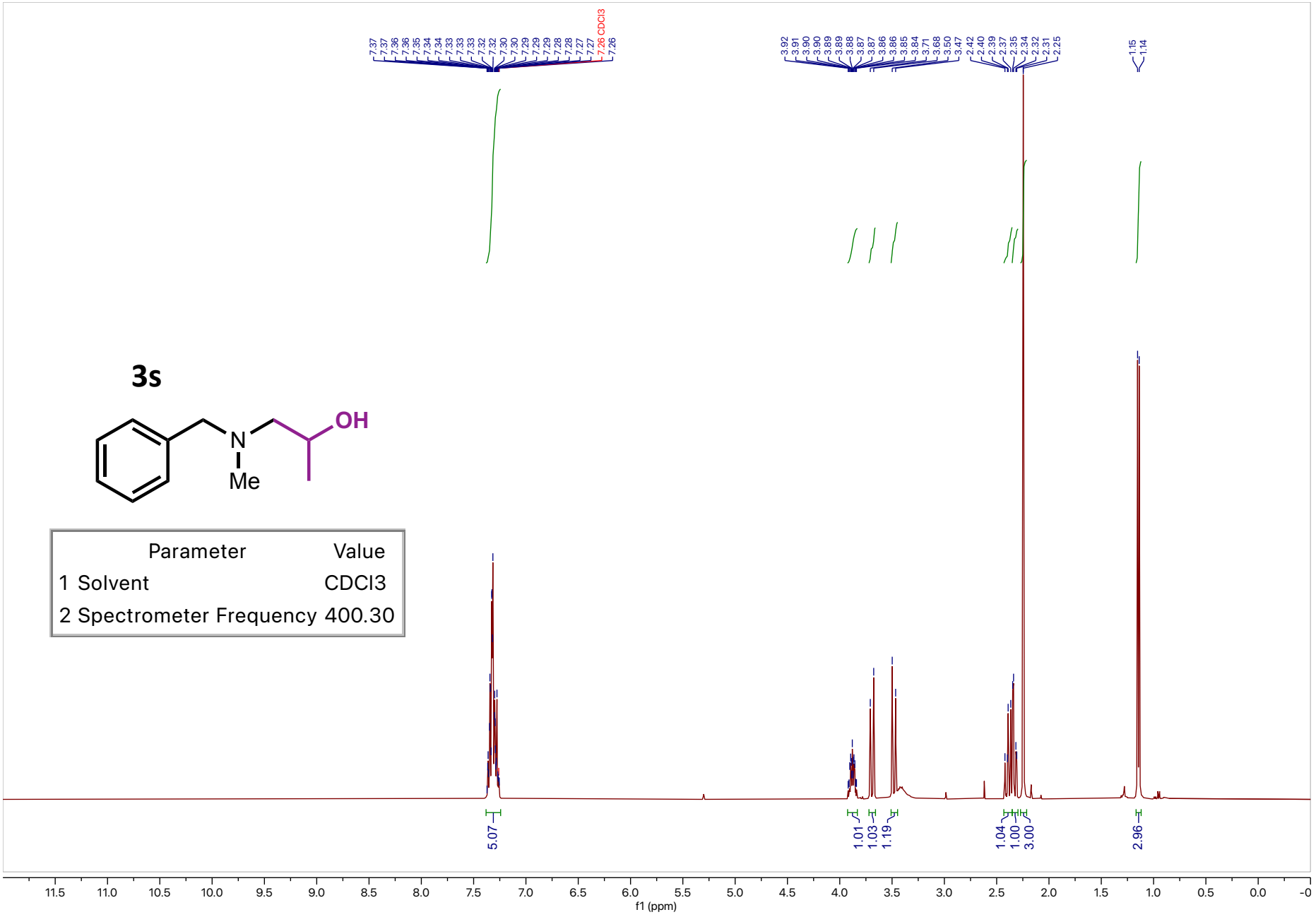

DB-06-26-car.2.fid
13C_29min CDCl3 {C:\Data} Beau 23
139.32
129.11
128.38
127.11
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
61.96
42.33
3a (via Bn-I)
N
Ph
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

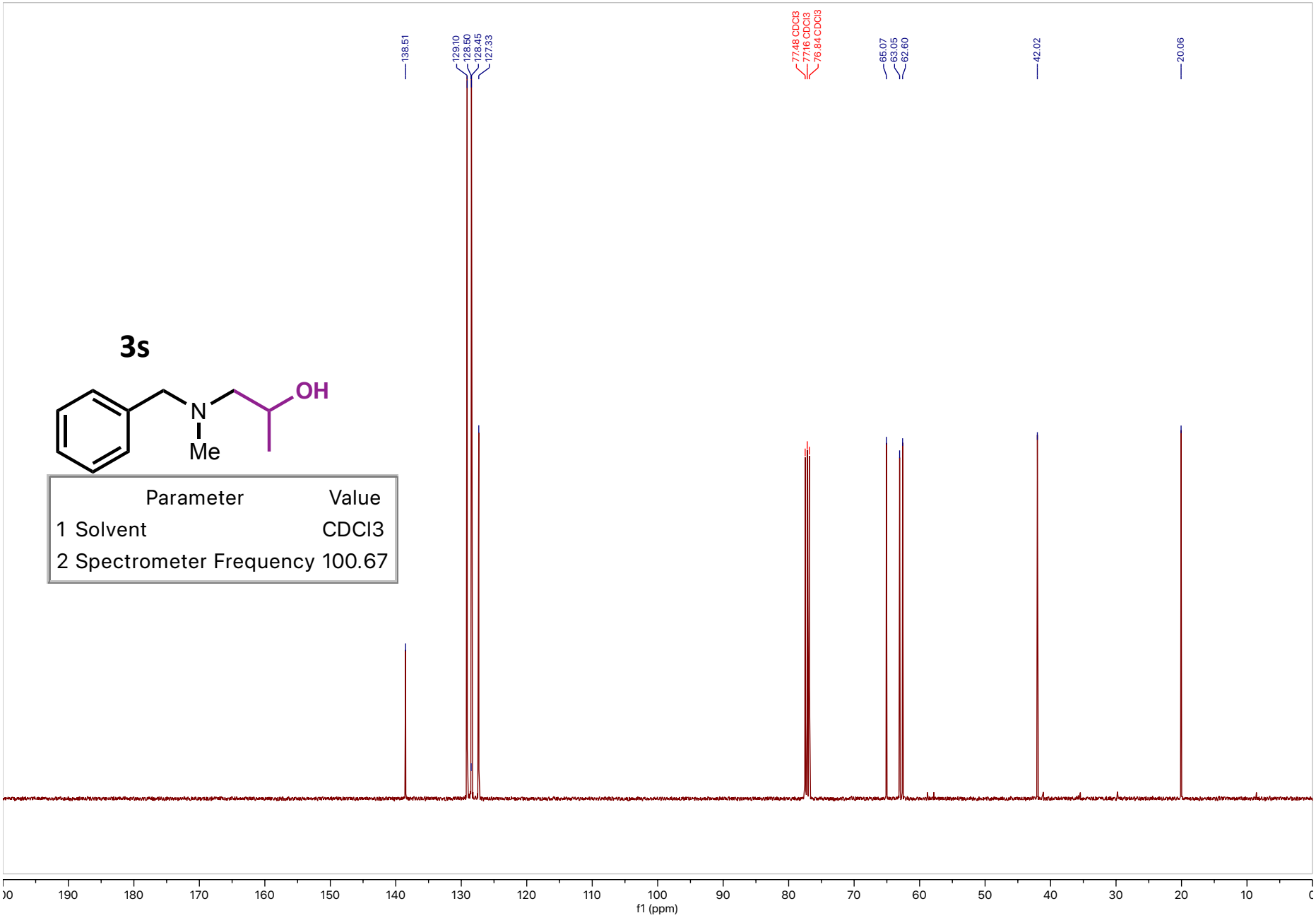

DB-06-35-post.1.fid
PROTON CDCl3 {C:\Data} Beau 35
3q
Ph
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
11.82
2.14
2.22
2.10
3.00
f1 (ppm)

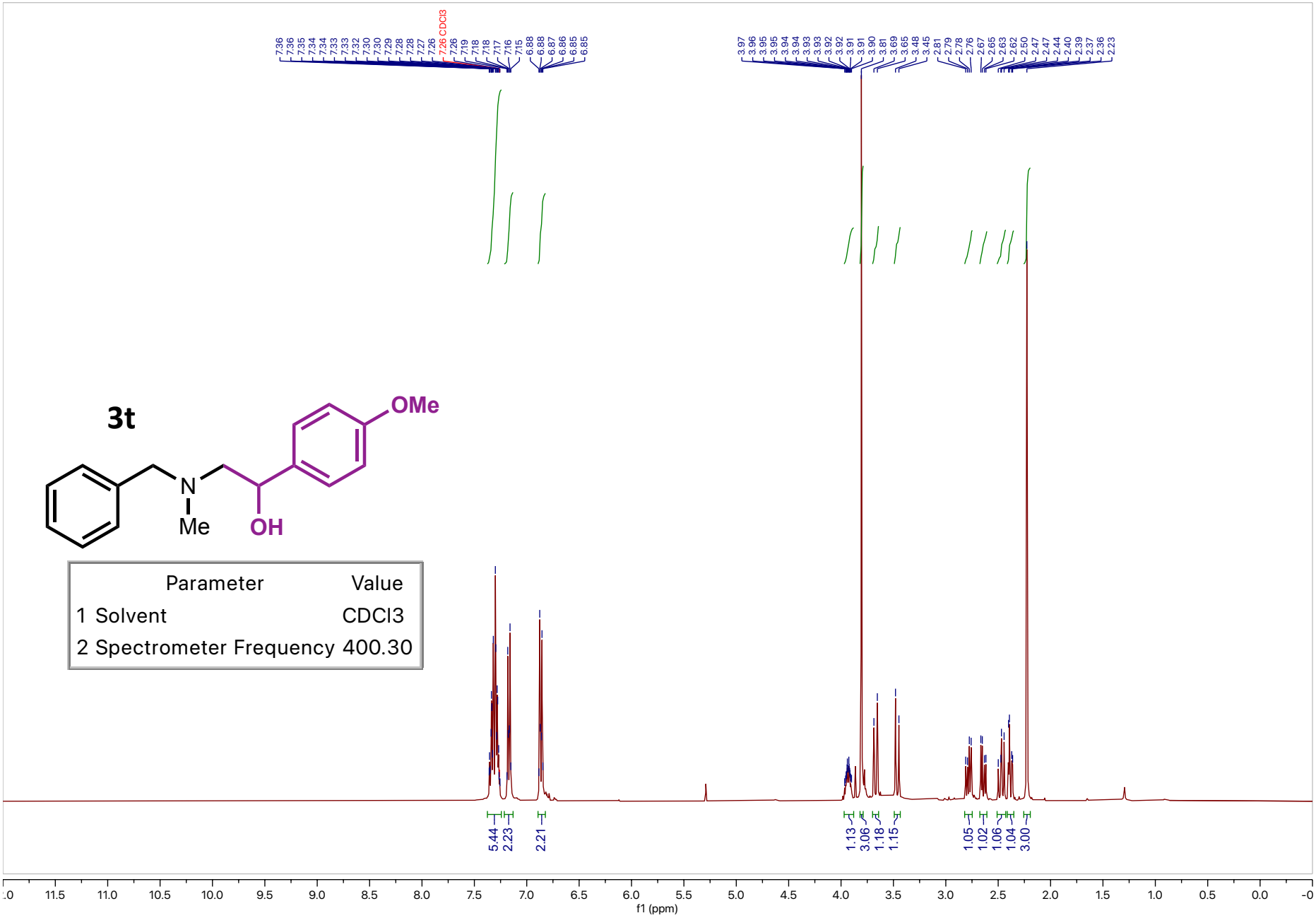

DB-06-35-post.2.fid
13C_58min CDCl3 {C:\Data} Beau 35
129.20
128.89
128.47
128.38
127.16
126.10
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
62.29
59.26
42.26
34.00
3q
Ph
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)

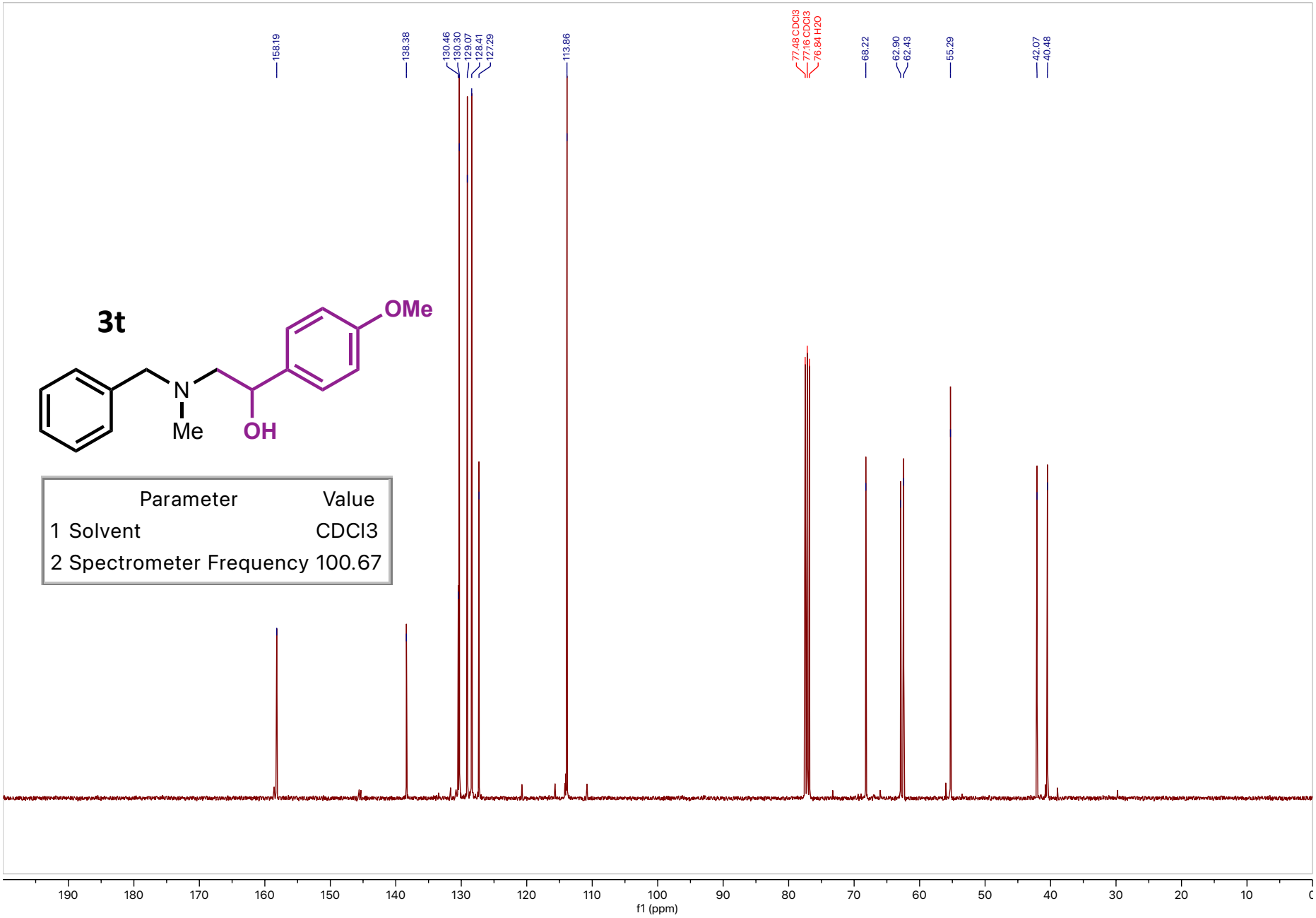

DB-06-33-fin.1.fid
PROTON CDCl3 {C:\Data} Beau 33
7.33
7.32
7.31
7.31
7.29
7.29
7.29
7.28
7.27
7.26
7.26 CDCl3
7.25
7.25
7.24
7.24
7.23
7.23
3.50
2.39
2.37
2.35
2.20
1.54
1.52
1.50
1.31
1.30
1.28
1.27
1.26
0.90
0.88
0.86
3r
decyl
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
6.64
2.00
2.17
3.14
2.25
17.42
3.70
f1 (ppm)

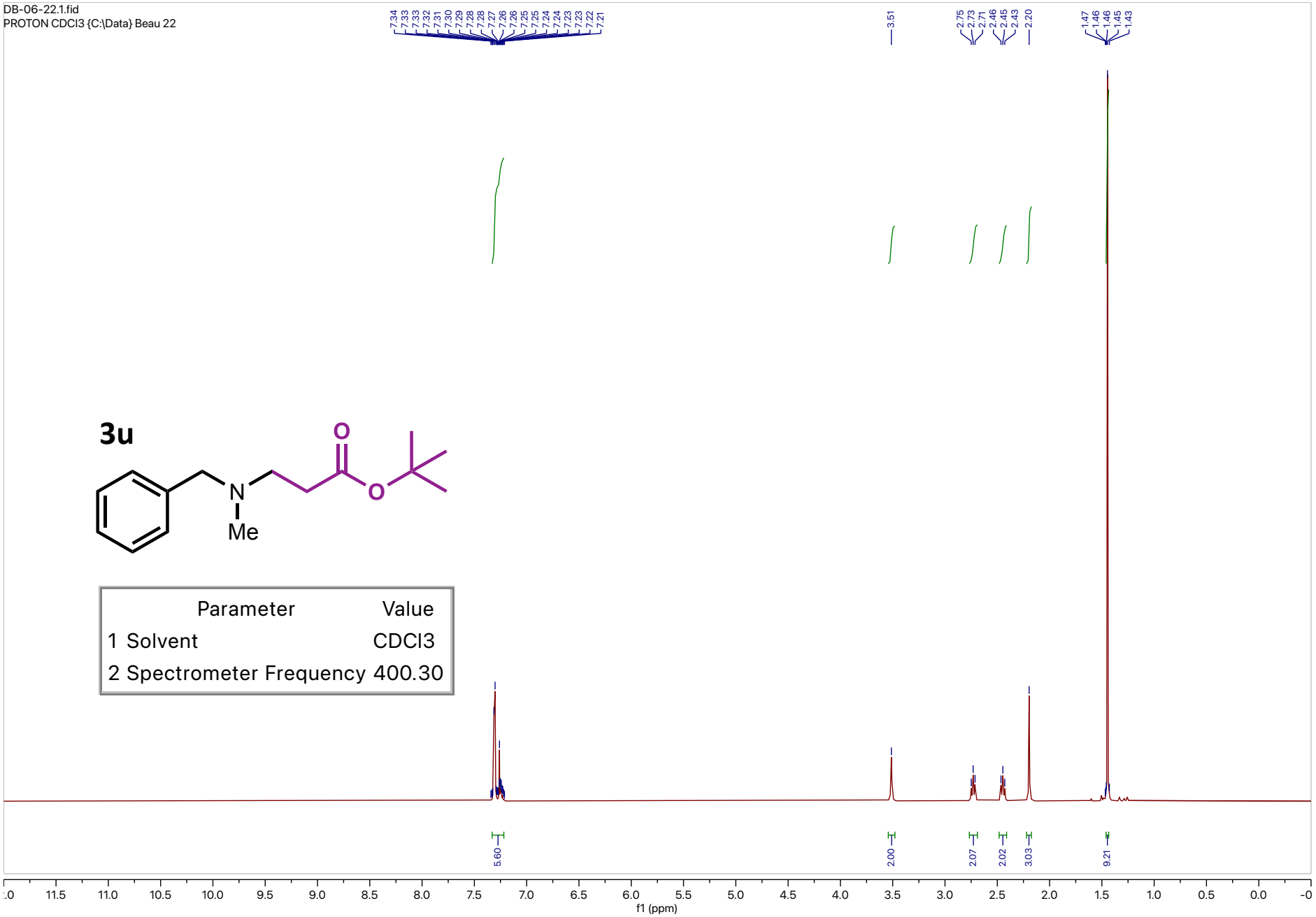

DB-06-33-fin.2.fid
13C_29min CDCl3 {C:\Data} Beau 33
133.59
129.28
128.34
127.09
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
62.38
57.69
42.32
32.06
29.79
29.74
29.48
27.60
27.45
22.84
14.27
3r
decyl
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)

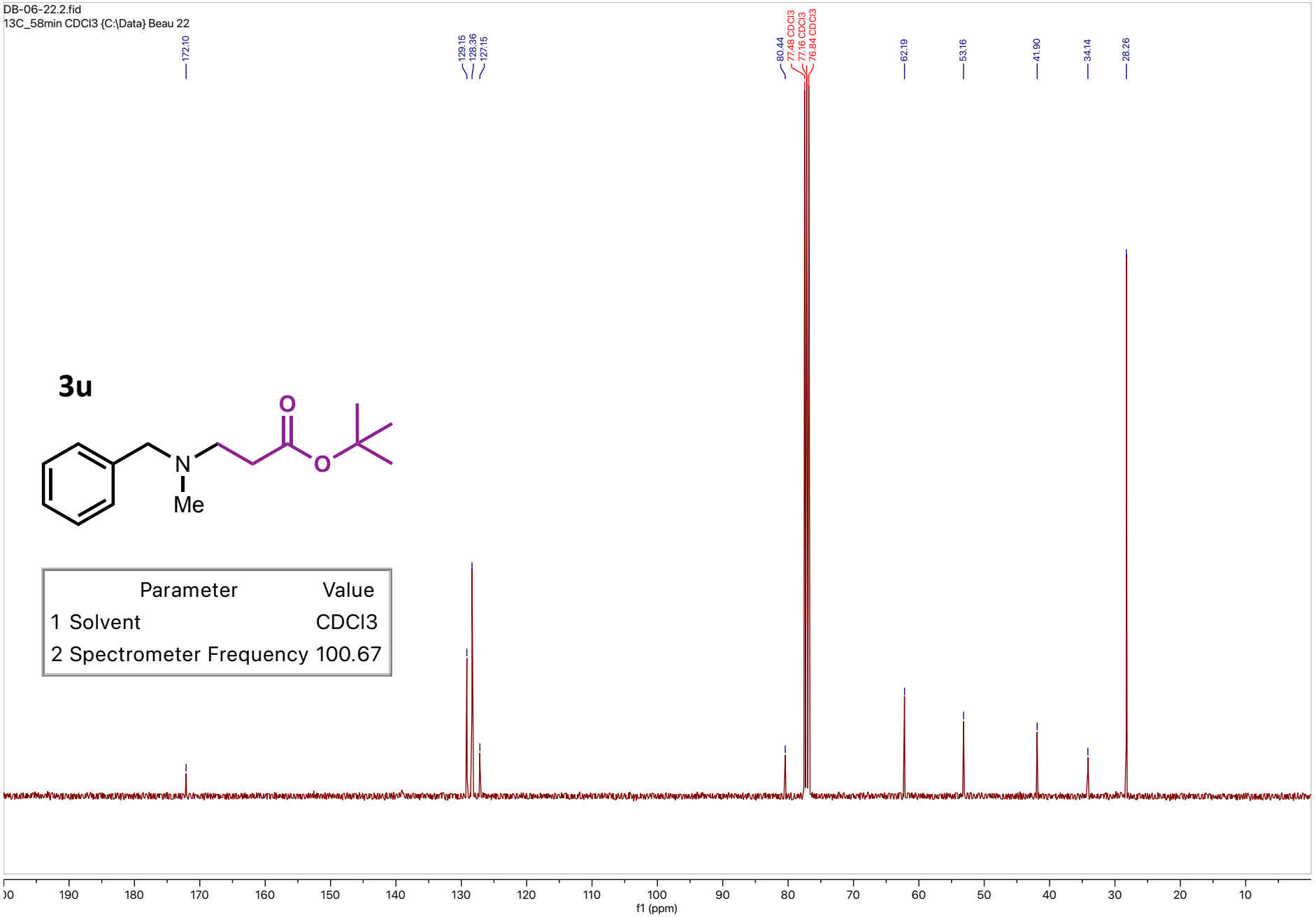

3s
OH
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
7.37 7.37 7.36 7.36 7.35 7.34 7.34 7.33 7.33 7.33 7.32 7.32 7.30 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.27 7.26 CDCl3 7.26
3.92 3.91 3.90 3.90 3.89 3.89 3.88 3.87 3.87 3.86 3.86 3.85 3.84 3.71 3.68 3.50 3.47 2.42 2.40 2.39 2.37 2.35 2.34 2.32 2.31 2.25
1.15 1.14
5.07
1.01 1.03 1.19
1.04 1.00 3.00
2.96
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

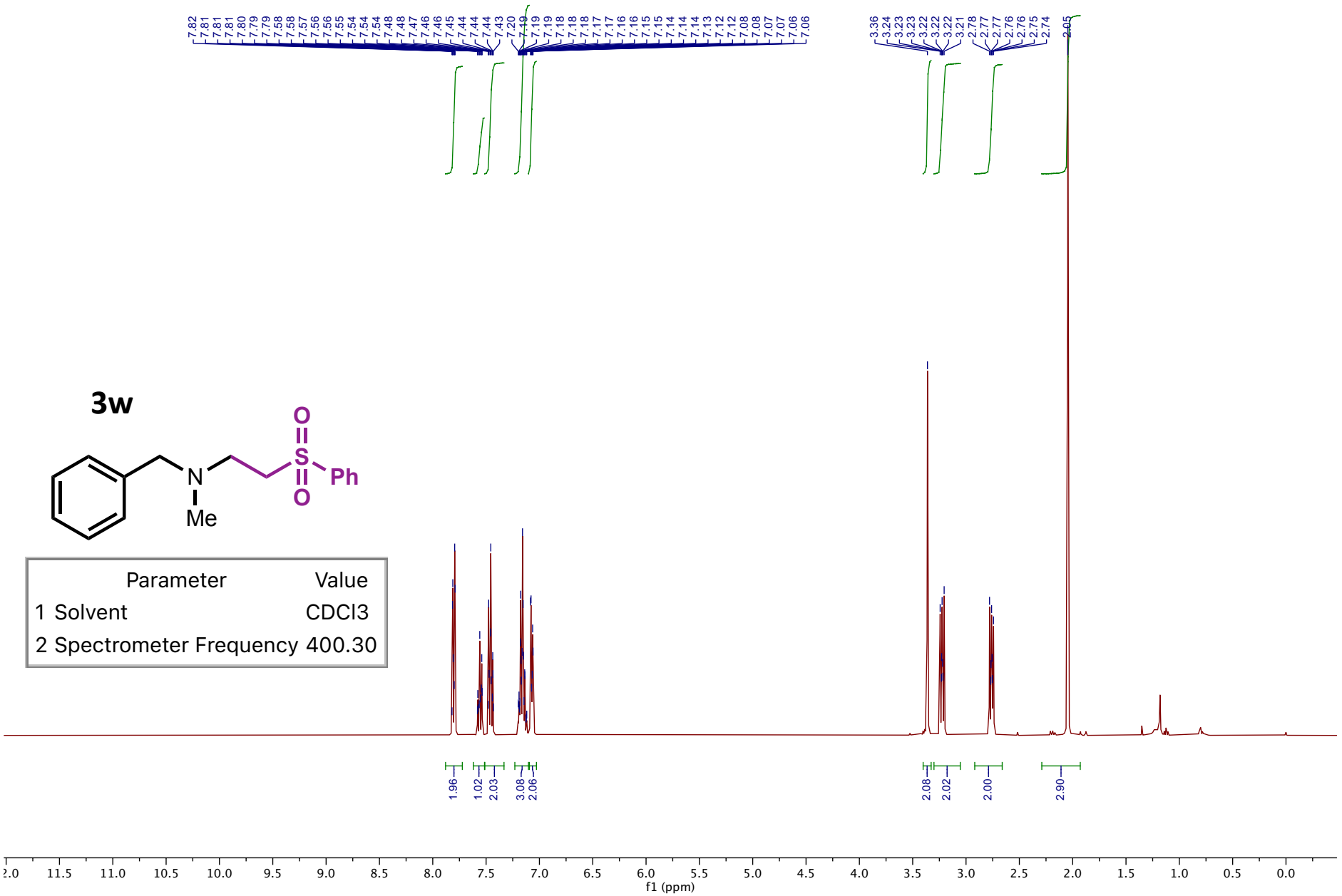

138.51
129.10
128.50
128.45
127.33
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
65.07
63.05
62.60
42.02
20.06
3s
OH
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)

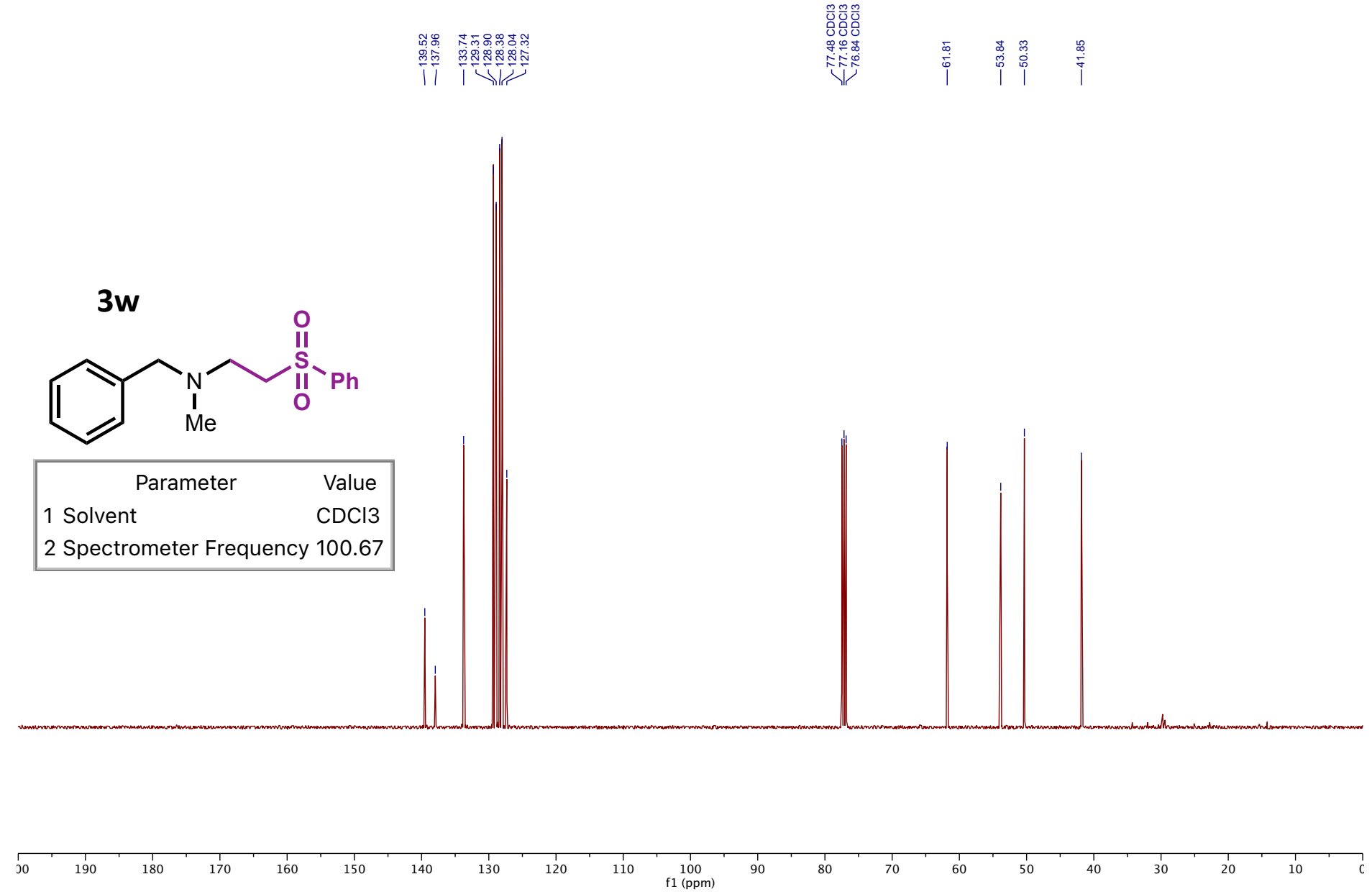

3t
OMe
N
Me
OH
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
f1 (ppm)

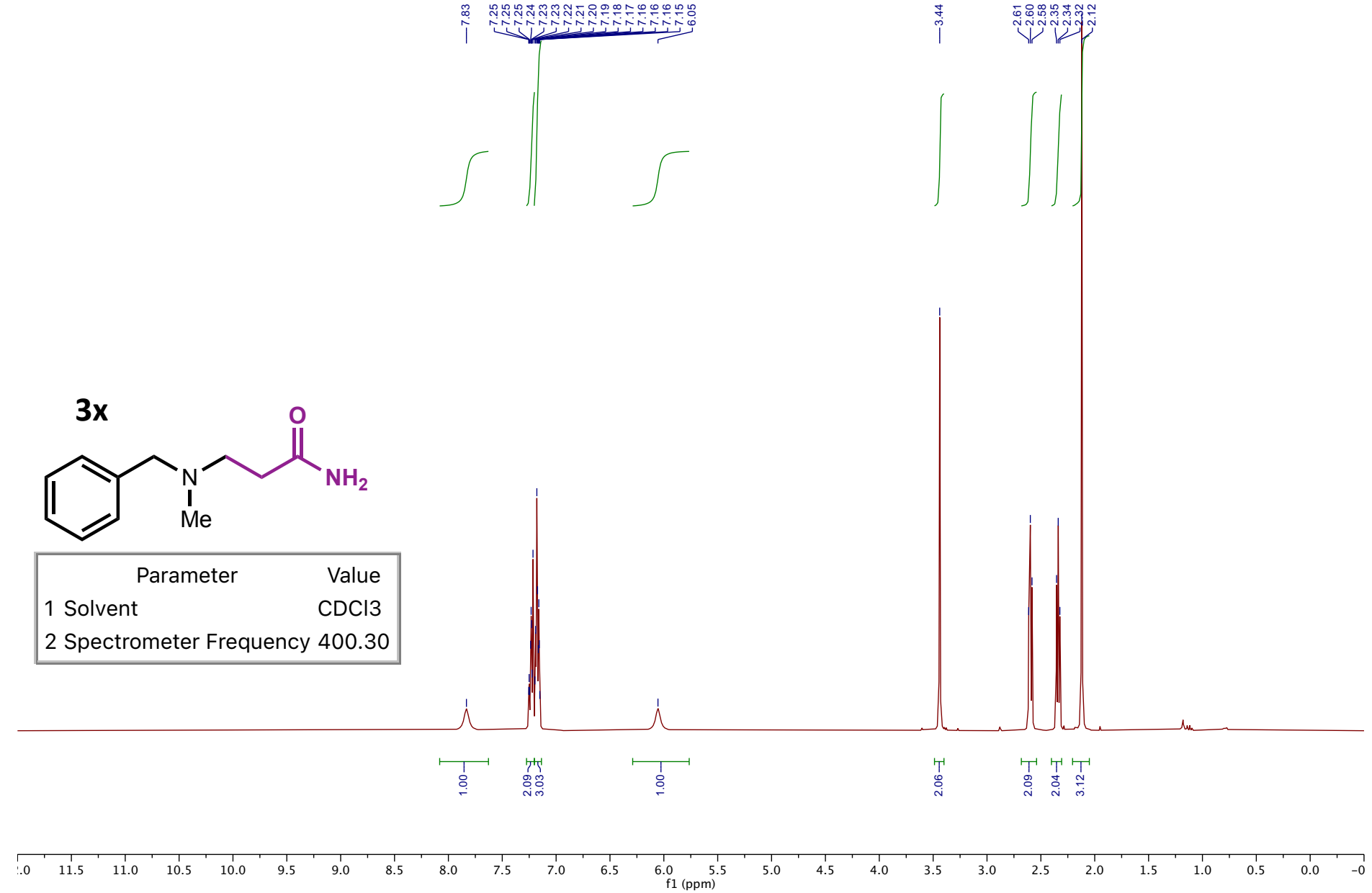

158.19
138.38
130.46
130.30
129.07
128.41
127.29
113.86
77.48 CDCl3
77.16 CDCl3
76.84 H2O
68.22
62.90
62.43
55.29
42.07
40.48
3t
OMe
N
Me
OH
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


DB-06-22.1.fid
PROTON CDCl3 {C:\Data} Beau 22
7.34 7.33 7.33 7.32 7.31 7.30 7.29 7.28 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.24 7.23 7.23 7.22 7.21
3.51
2.75 2.73 2.71 2.46 2.45 2.43 2.20
1.47 1.46 1.46 1.45 1.43
3u
O
N
Me
O
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
5.60
2.00
2.07
2.02
3.03
9.21
f1 (ppm)


DB-06-22.2.fid
13C_58min CDCl3 {C:\Data} Beau 22
172.10
129.15
128.36
127.15
80.44
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
62.19
53.16
41.90
34.14
28.26
3u
O
N
Me
O
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)

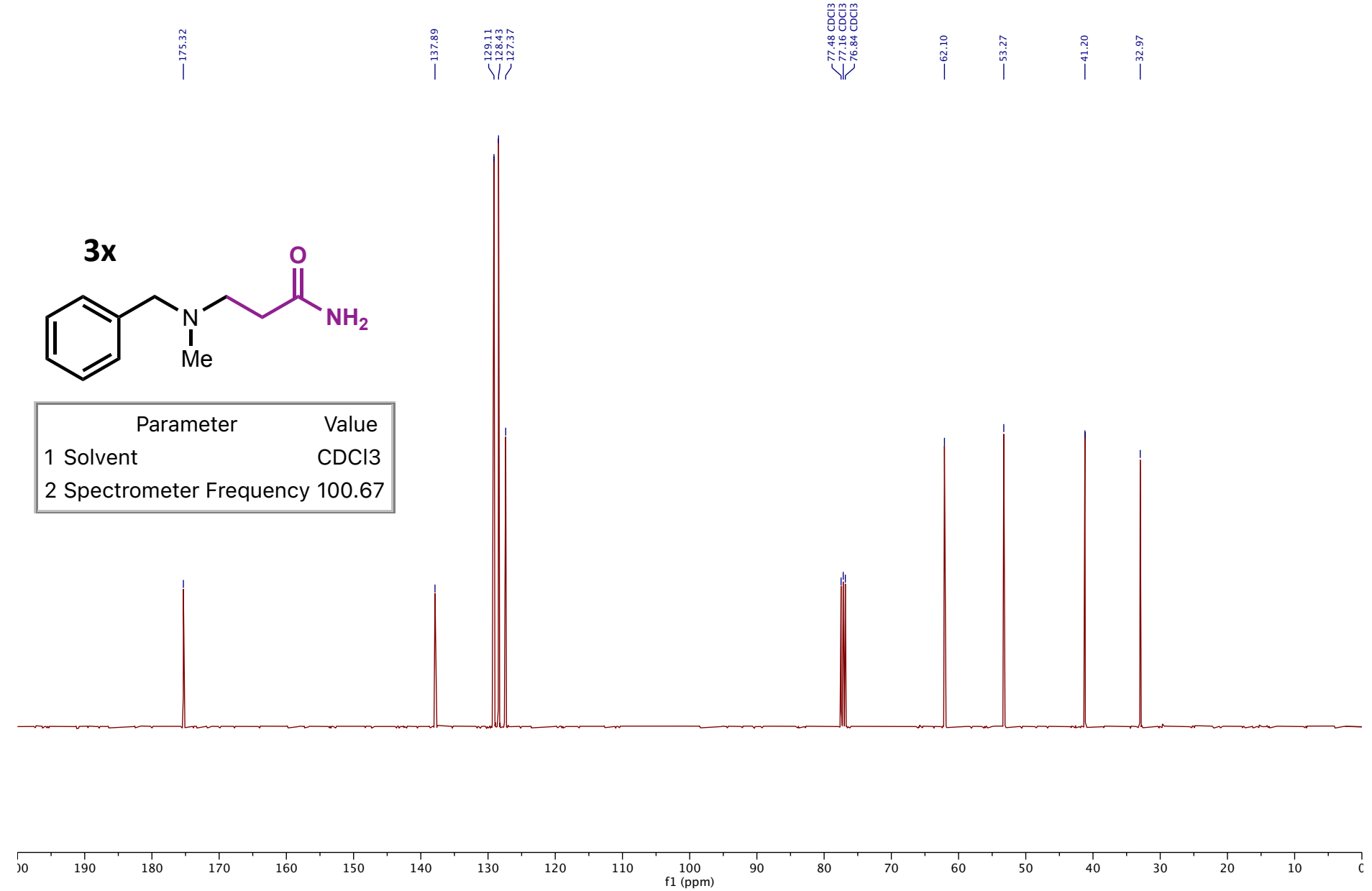

3w
O
S
O
Ph
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
1.96
1.02
2.03
3.08
2.06
2.08
2.02
2.00
2.90
f1 (ppm)


139.52
137.96
133.74
129.31
128.90
128.38
128.04
127.32
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
61.81
53.84
50.33
41.85
3w
O
S
O
Ph
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)

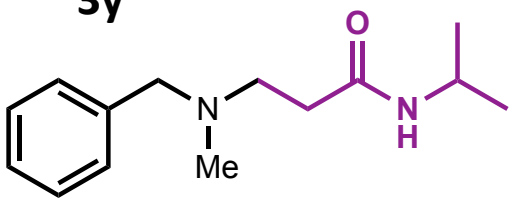


3x

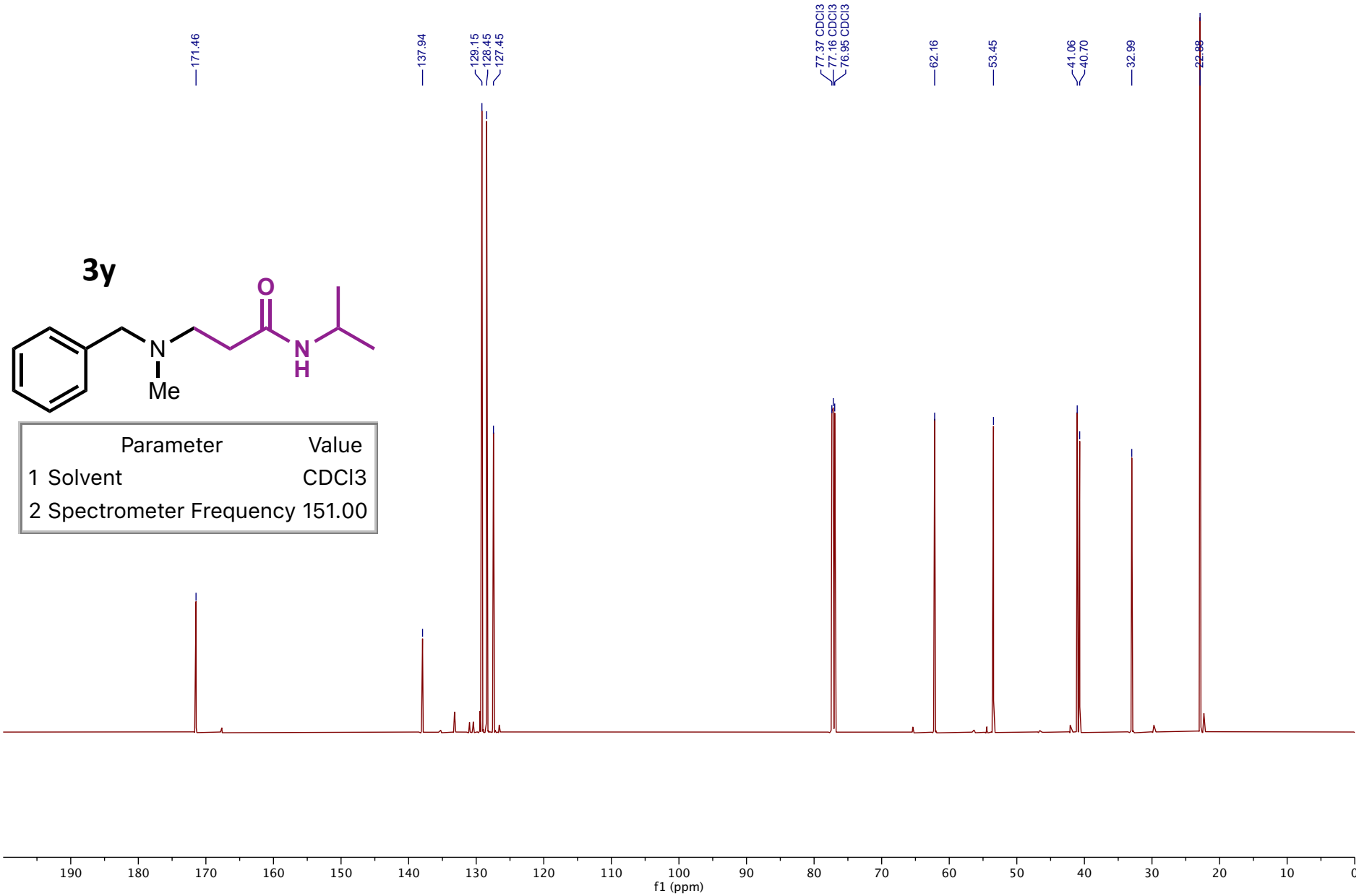
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	400.30

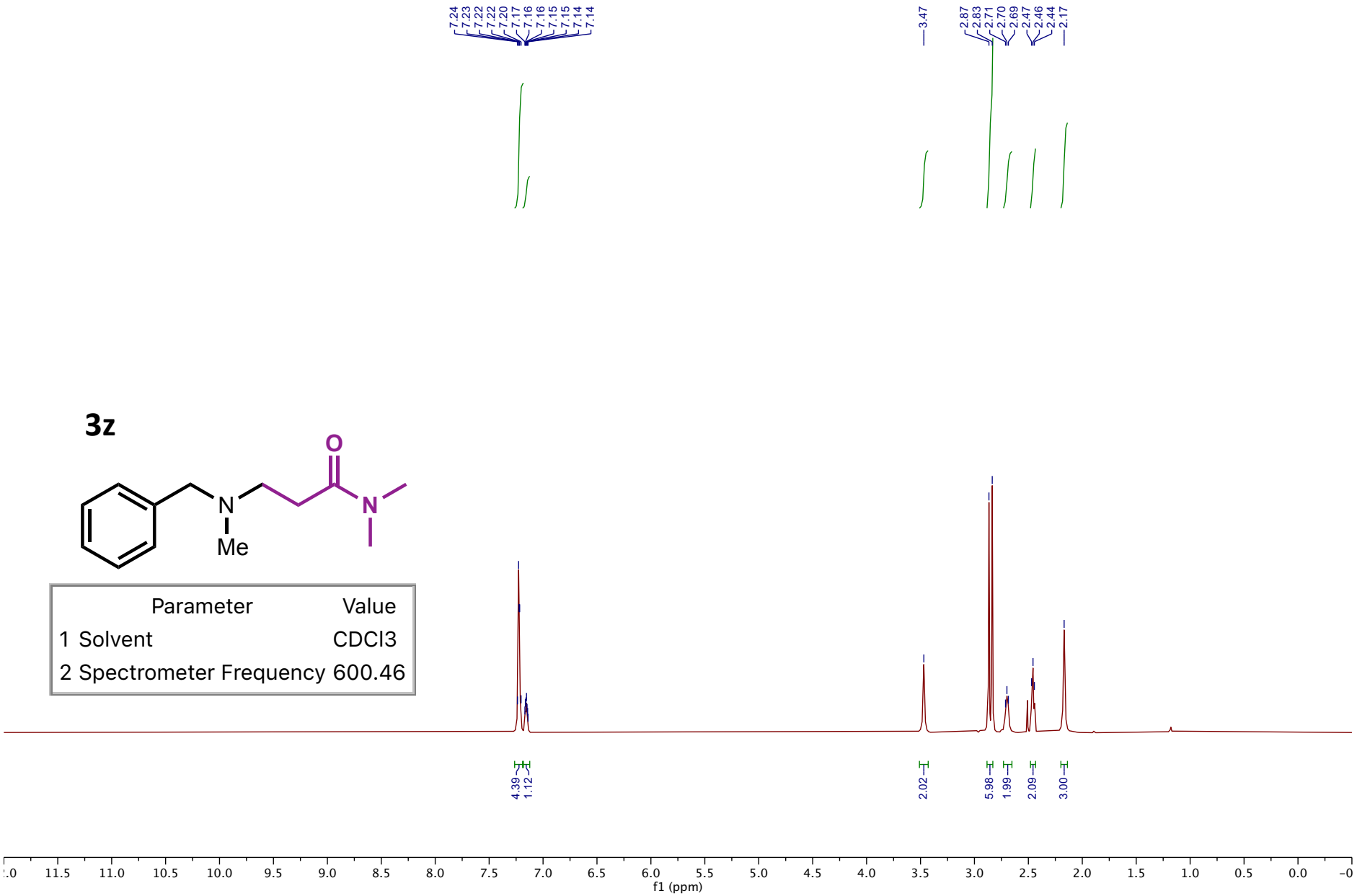

175.32
137.89
129.11
128.43
127.37
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
62.10
53.27
41.20
32.97
3x
O
NH2
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)

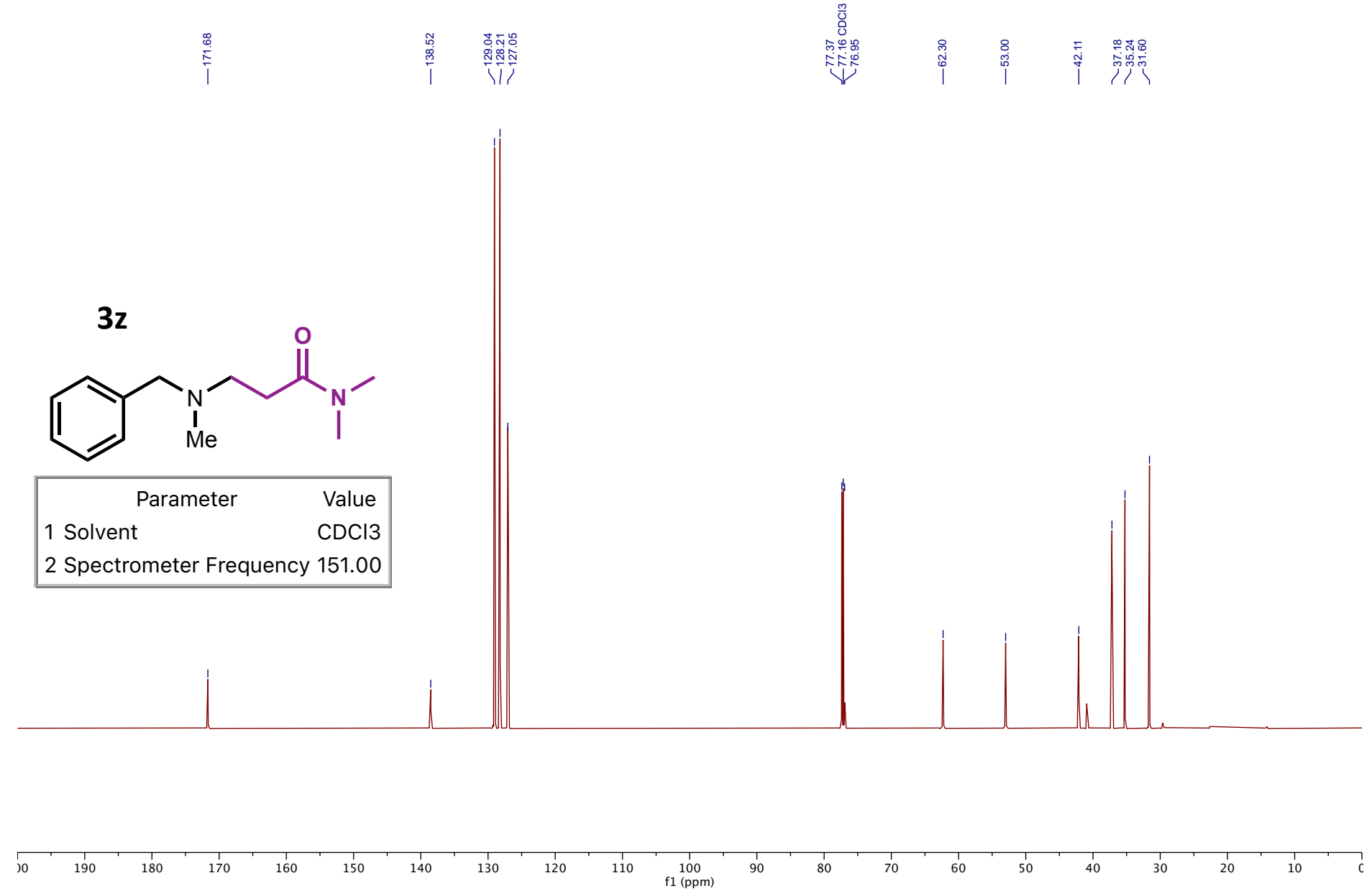
3y

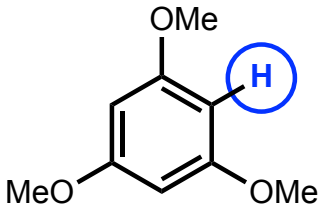


	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	600.46

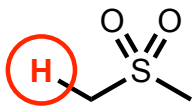

171.46
137.94
129.15
128.45
127.45
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
62.16
53.45
41.06
40.70
32.99
22.88
3y
O
N
Me
N
H
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 151.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.24
7.23
7.22
7.22
7.20
7.17
7.16
7.16
7.15
7.15
7.14
7.14
3.47
2.87
2.83
2.71
2.70
2.69
2.47
2.46
2.44
2.17
3z
O
N
Me
N
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 600.46
4.39
1.12
2.02
5.98
1.99
2.09
3.00
f1 (ppm)


171.68
138.52
129.04
128.21
127.05
77.37
77.16 CDCl3
76.95
62.30
53.00
42.11
37.18
35.24
31.60
3z
O
N
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 151.00
f1 (ppm)

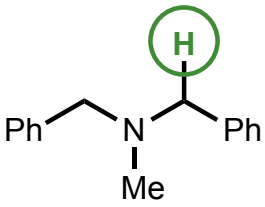


TMB
(3H)



$DMSO_2$
(6H)

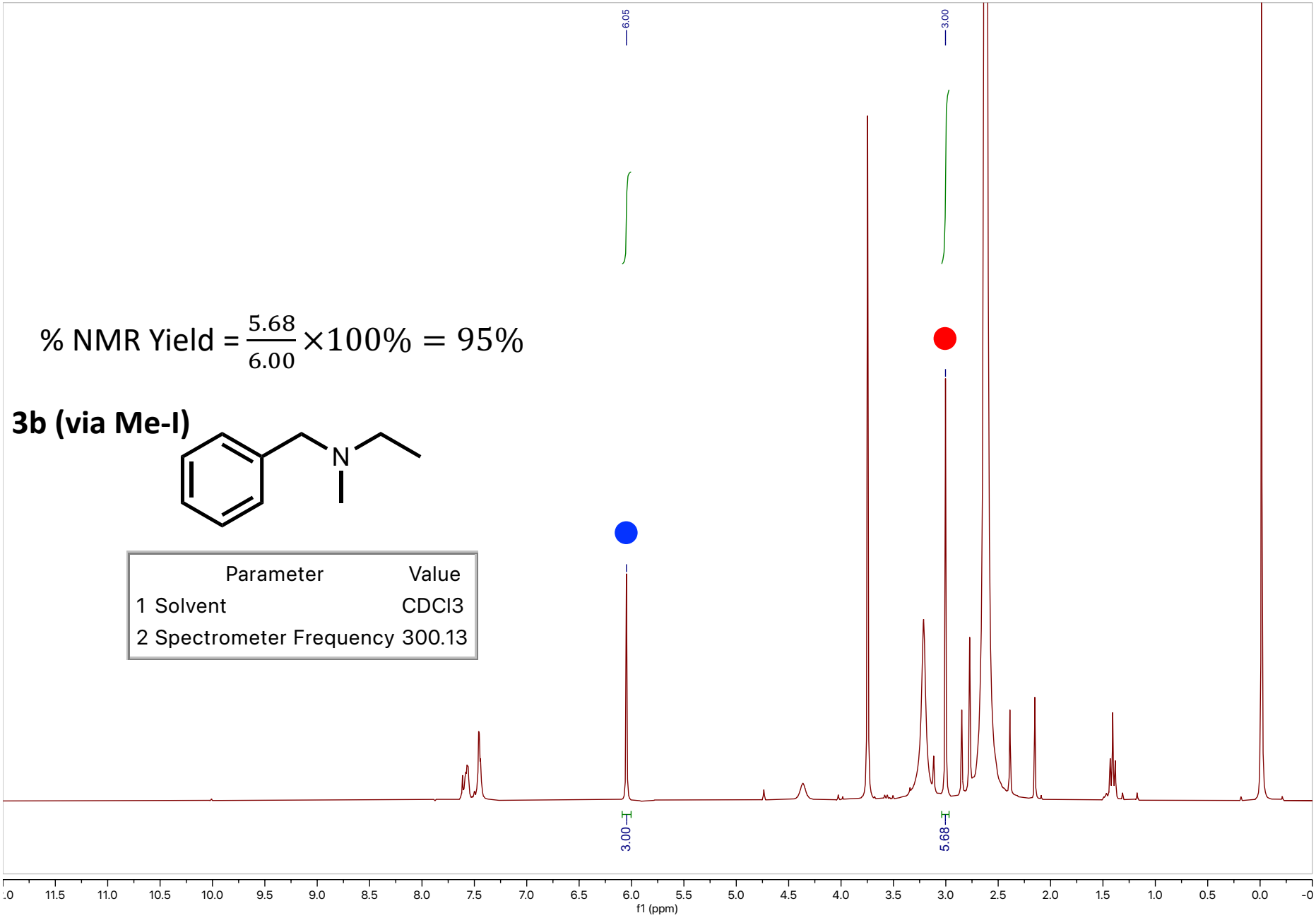
$$\% \text{ NMR Yield} = \frac{5.32}{6.00} \times 100\% = 89\%$$



Bn_2NMe
(4H)

$$\% \text{ NMR Yield} = \frac{3.68}{4.00} \times 100\% = 92\%$$

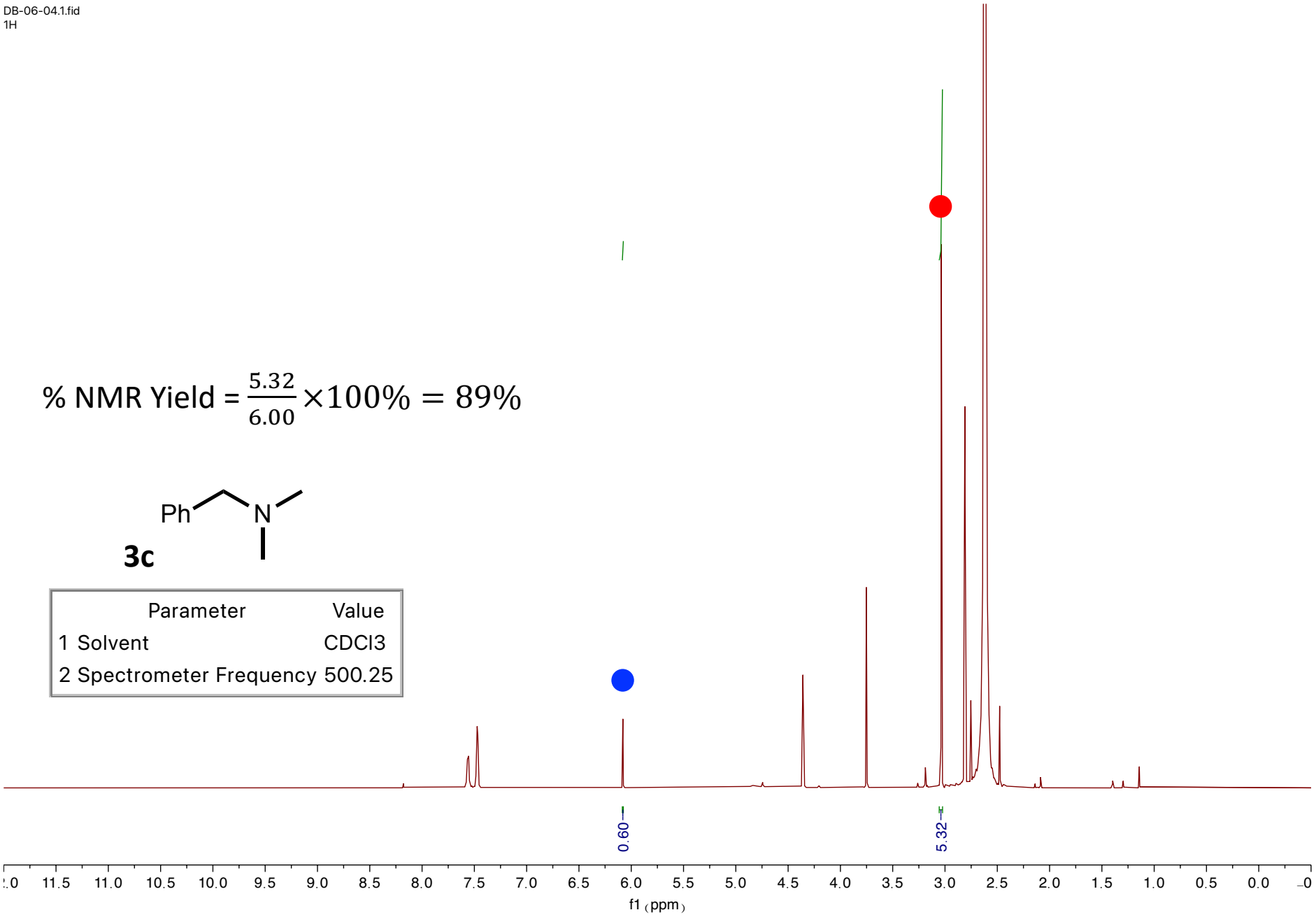
Parameter	Value
1 Solvent	CDCl3
2 Spectrometer Frequency	300.13

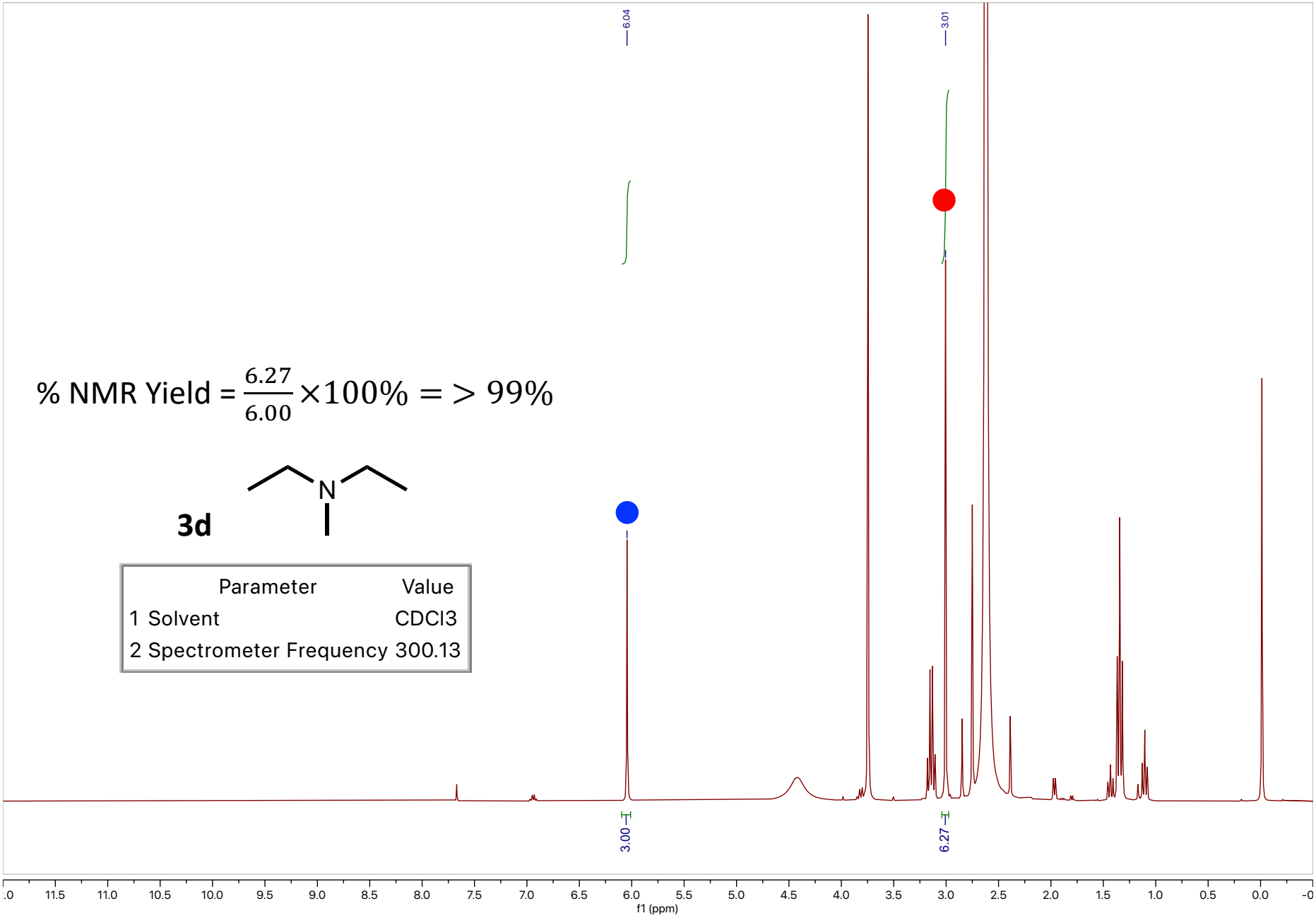


$$\% \text{ NMR Yield} = \frac{5.68}{6.00} \times 100\% = 95\%$$

3b (via Me-I)

	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	300.13

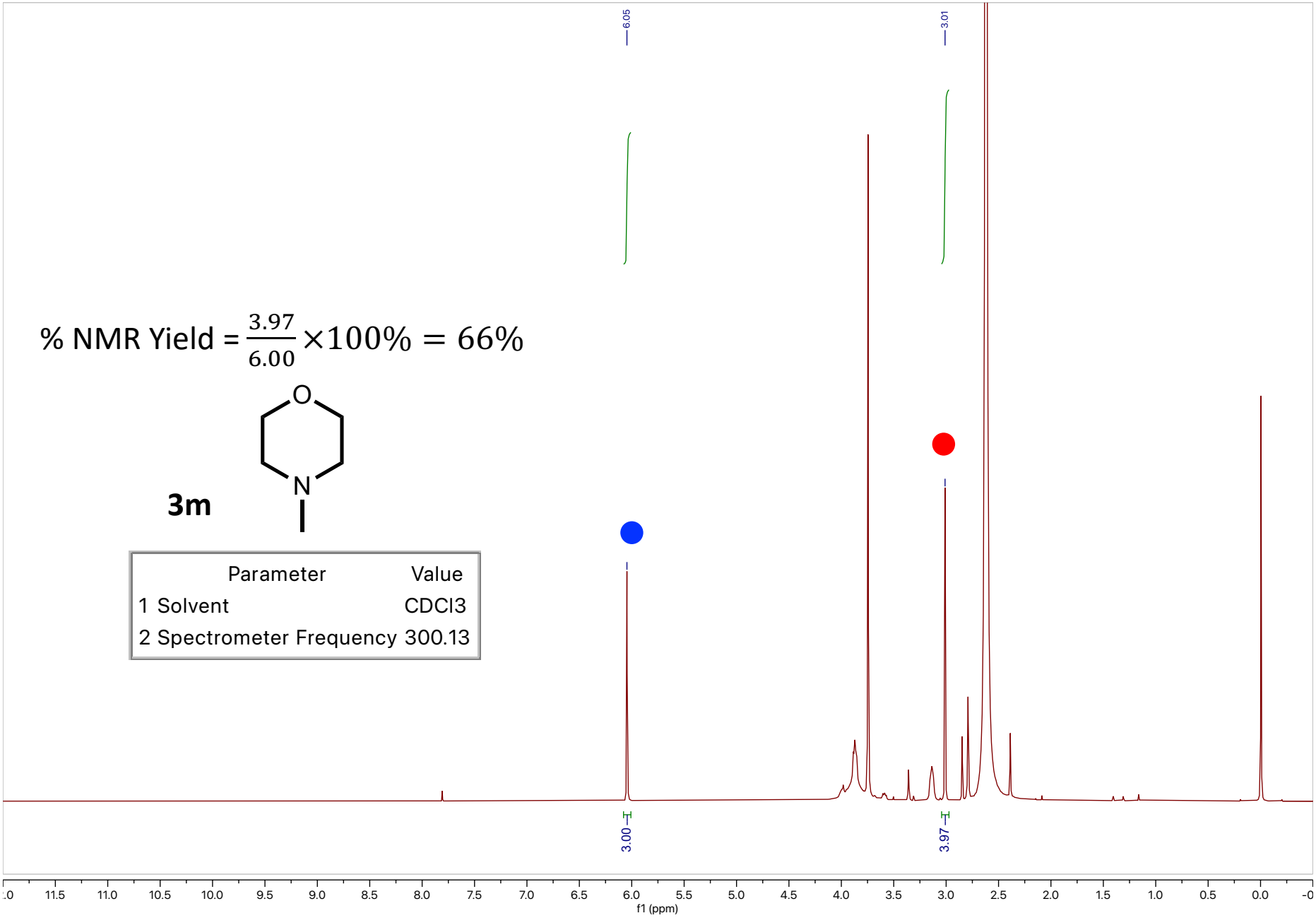

DB-06-04.1.fid
1H
% NMR Yield = $\frac{5.32}{6.00} \times 100\% = 89\%$
Ph
N
3c
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 500.25
0.60
5.32
f1 (ppm)

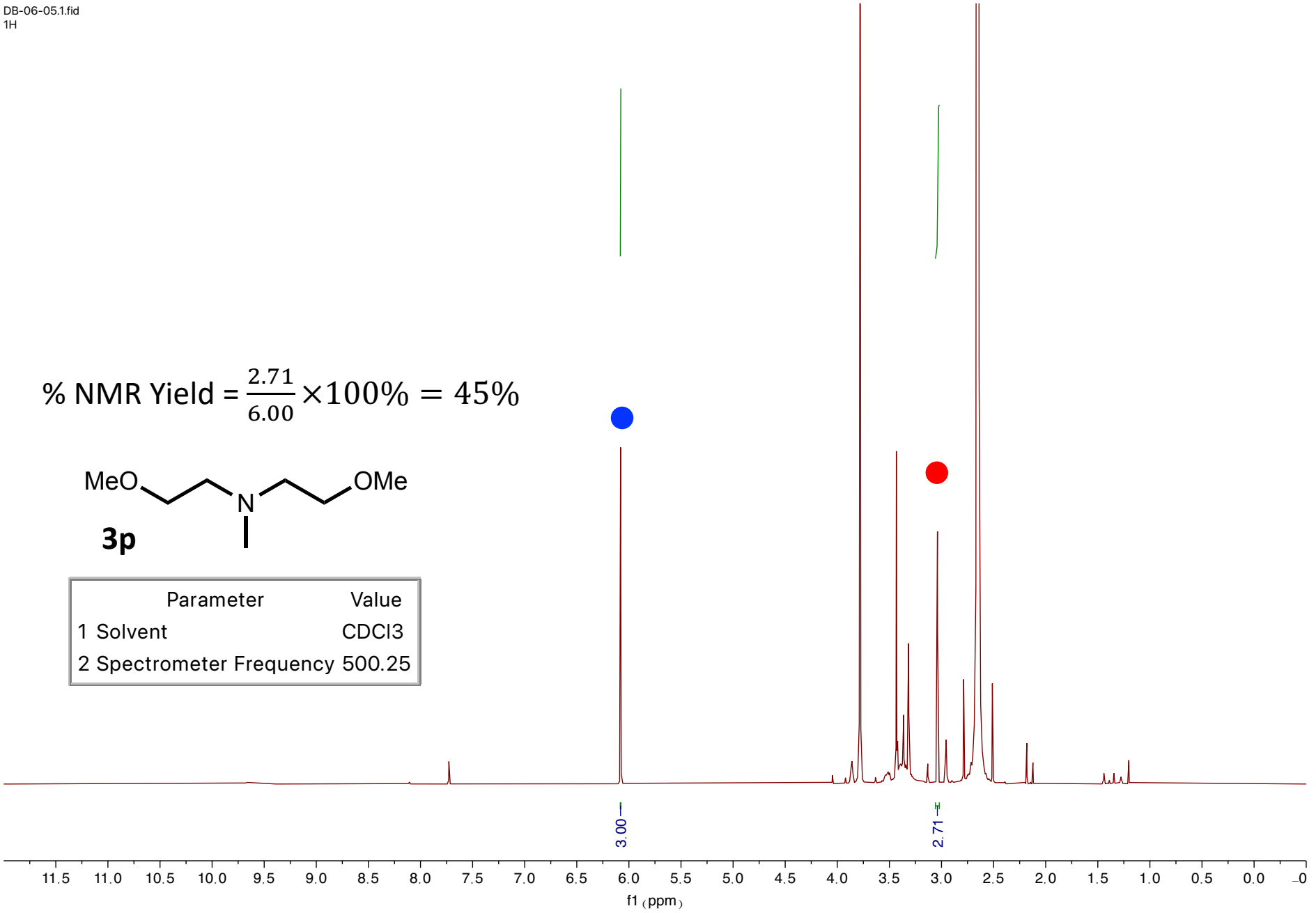


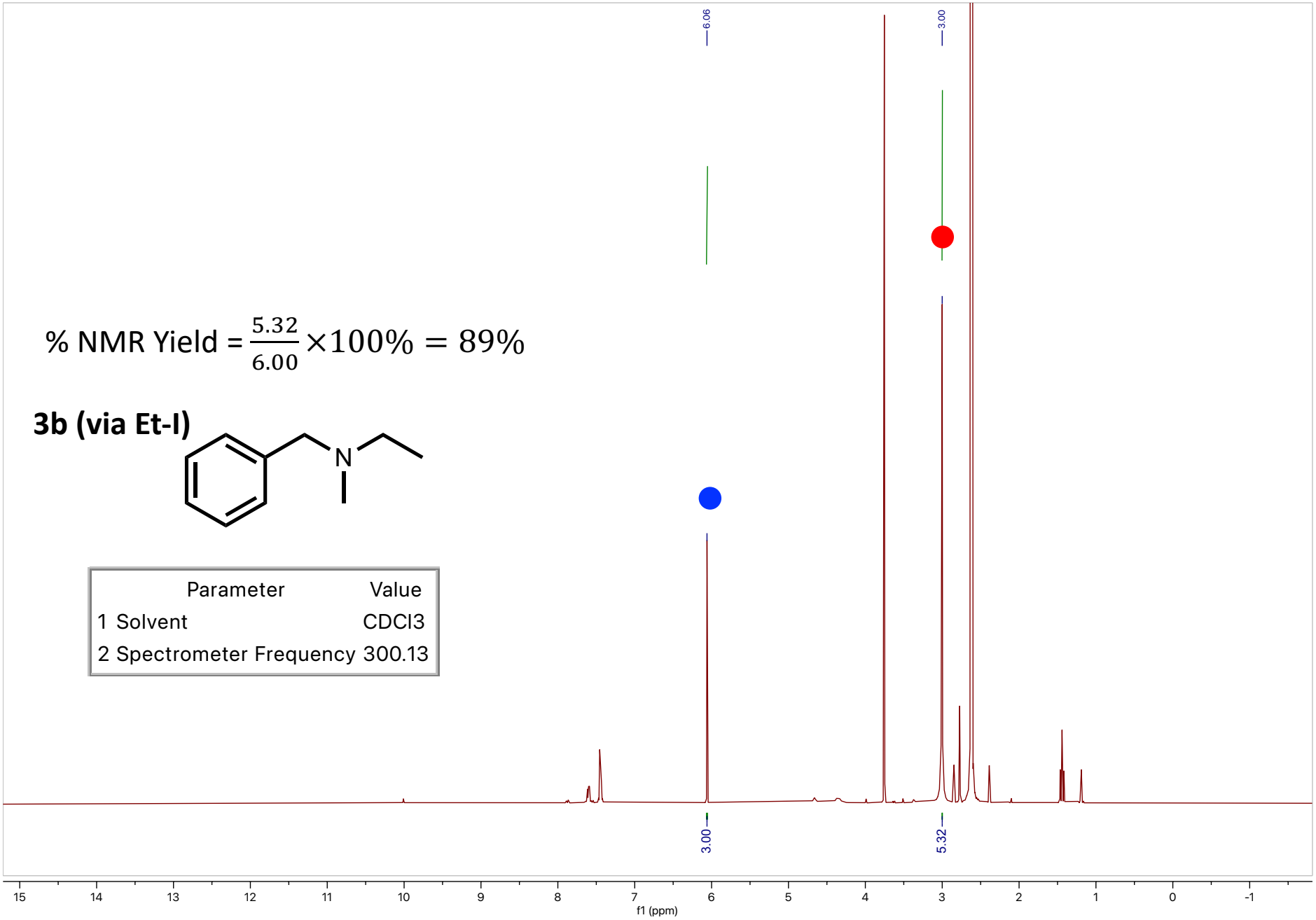
$$\% \text{ NMR Yield} = \frac{6.27}{6.00} \times 100\% = > 99\%$$

3d

	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	300.13


% NMR Yield = $\frac{3.97}{6.00} \times 100\% = 66\%$
3m
O
N
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 300.13
6.05
3.01
3.00
3.97
f1 (ppm)


DB-06-05.1.fid
1H
% NMR Yield = $\frac{2.71}{6.00} \times 100\% = 45\%$
MeO
N
OMe
3p
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 500.25
3.00
2.71
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



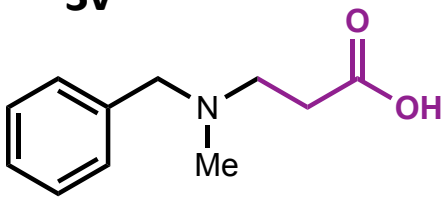
$$\% \text{ NMR Yield} = \frac{5.32}{6.00} \times 100\% = 89\%$$

3b (via Et-I)

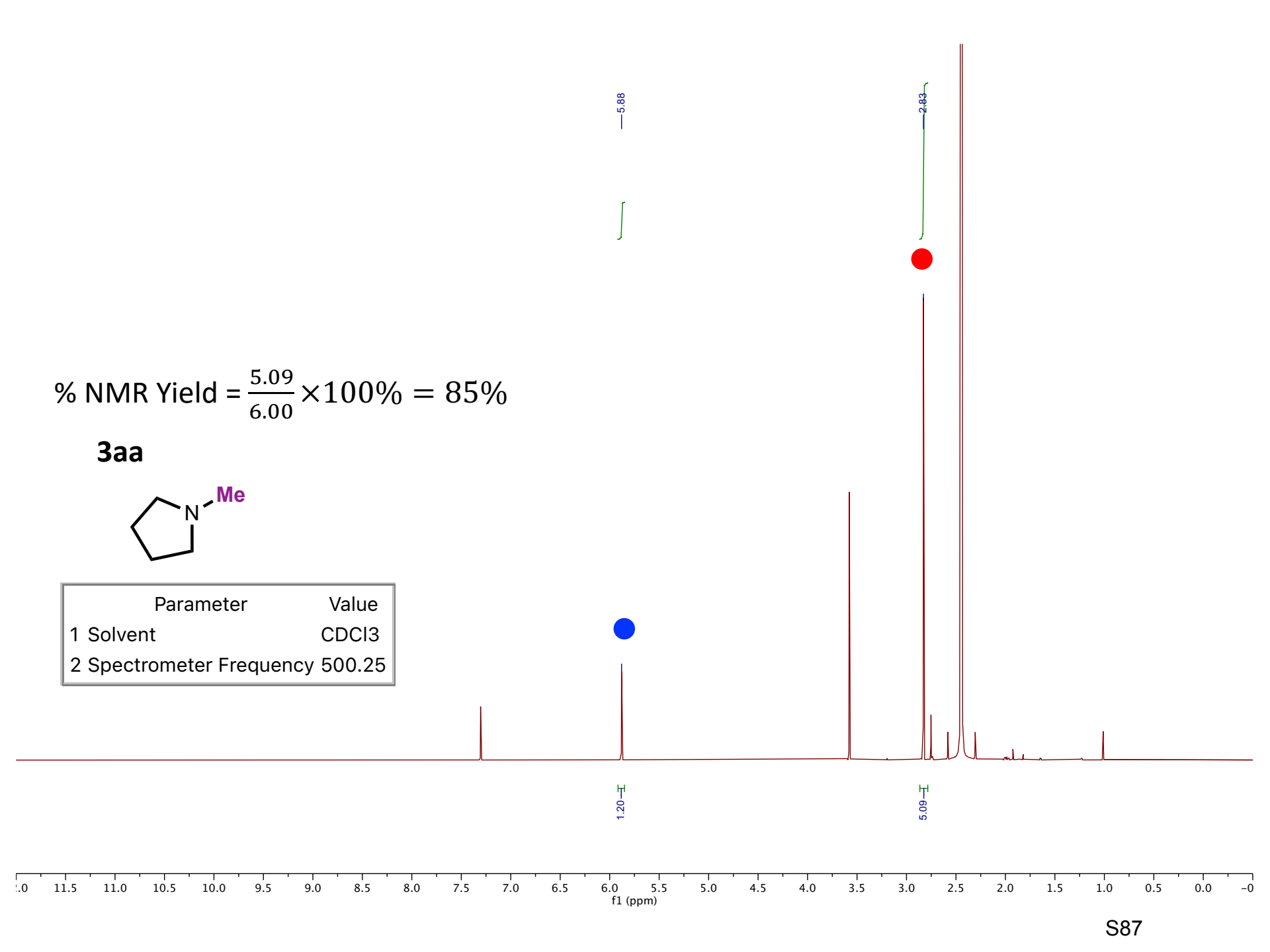
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	300.13

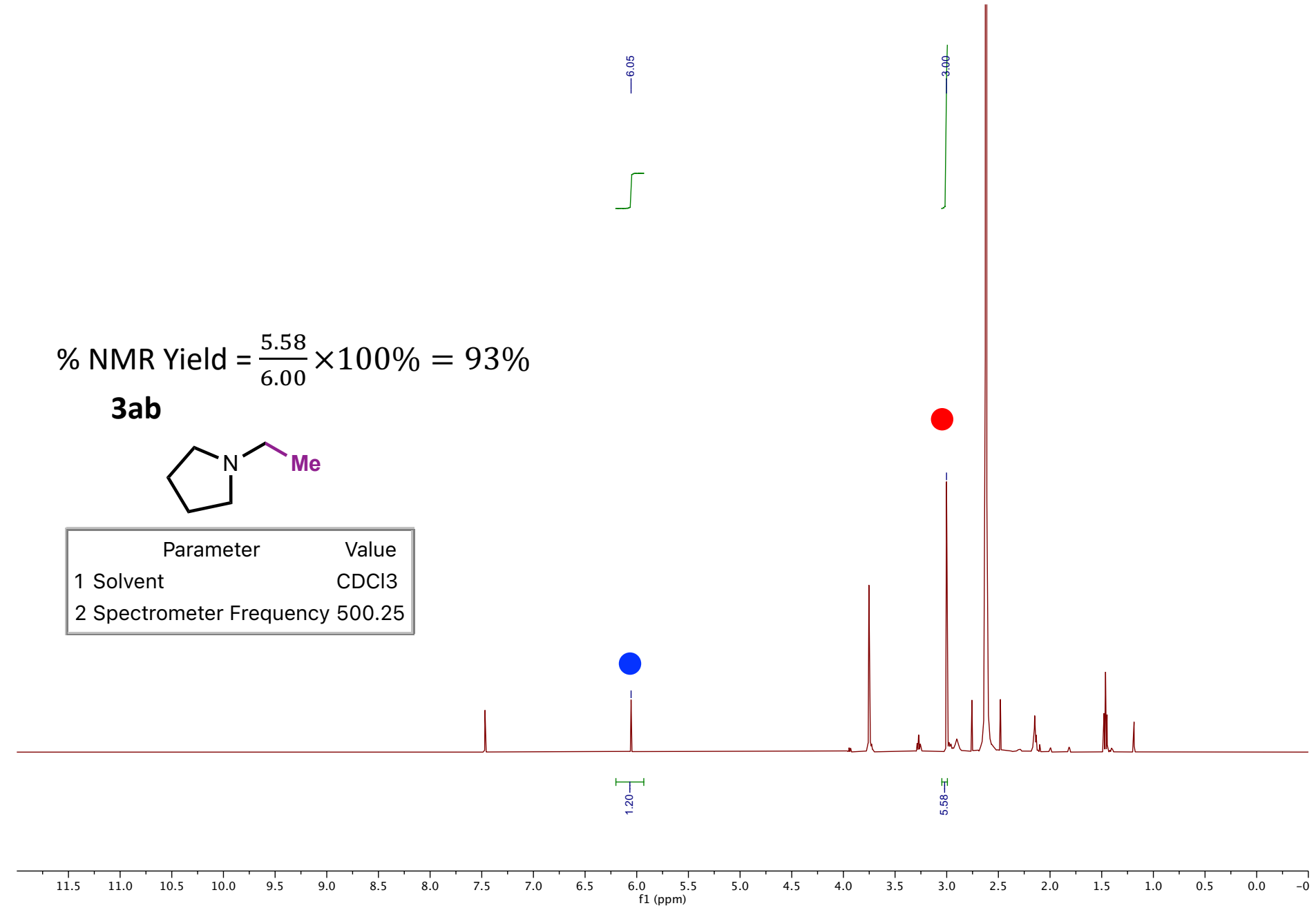
$$\% \text{ NMR Yield} = \frac{5.43}{6.00} \times 100\% = 91\%$$

3v



	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	600.46

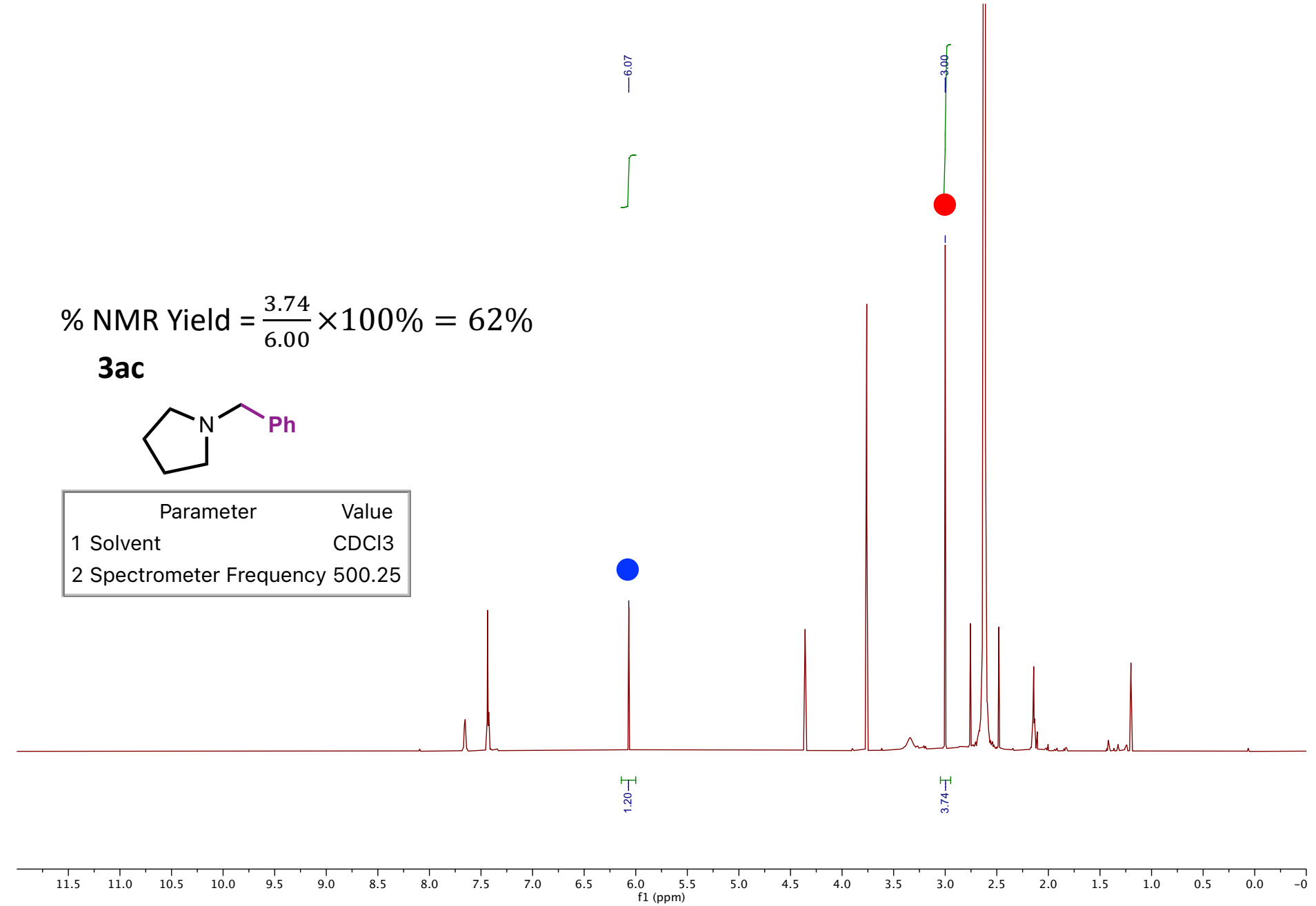

% NMR Yield = $\frac{5.09}{6.00} \times 100\% = 85\%$
3aa
Me
N
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 500.25
5.88
2.83
1.20
5.09
f1 (ppm)



$$\% \text{ NMR Yield} = \frac{5.58}{6.00} \times 100\% = 93\%$$

3ab

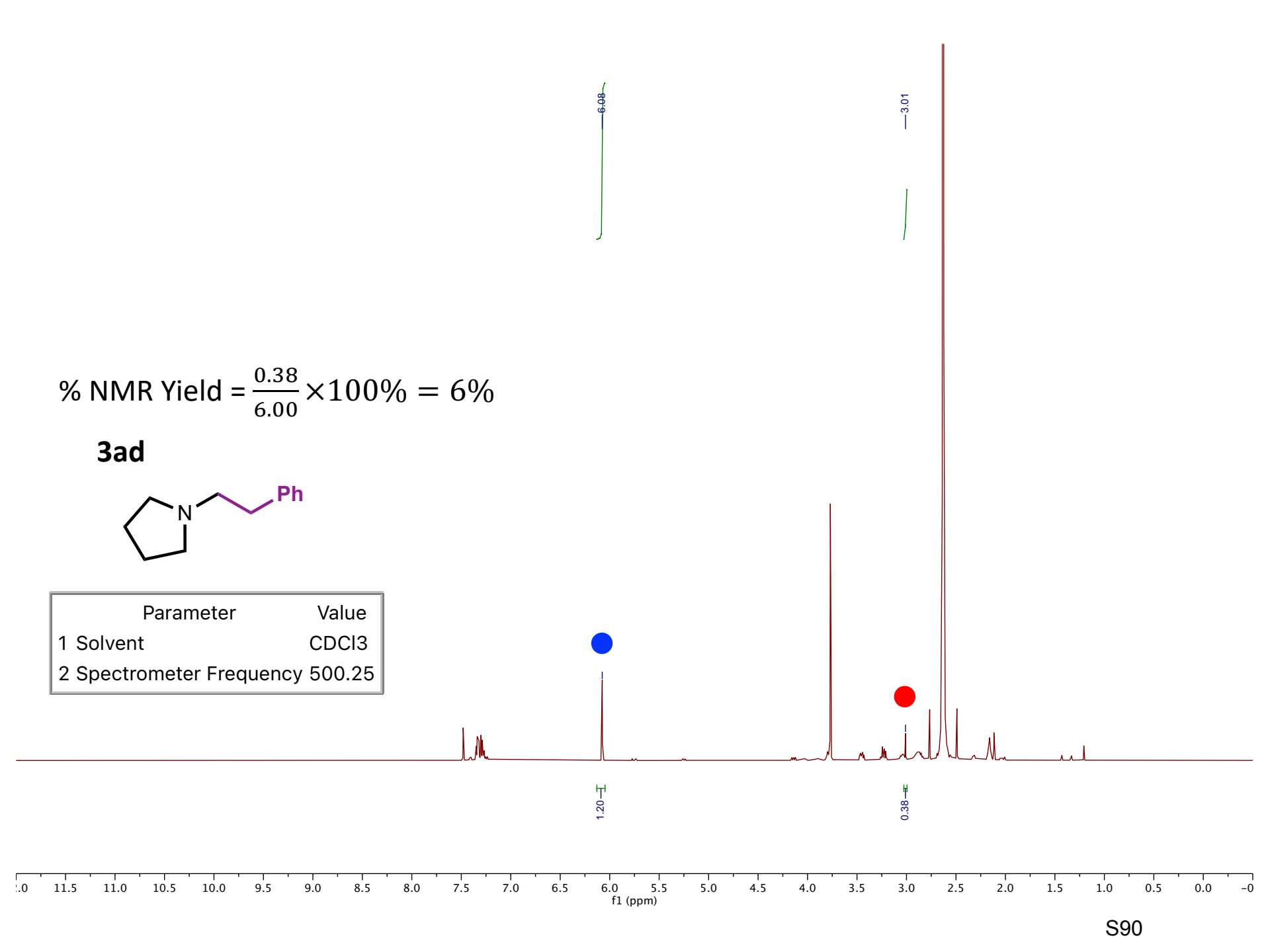
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	500.25



$$\% \text{ NMR Yield} = \frac{3.74}{6.00} \times 100\% = 62\%$$

3ac

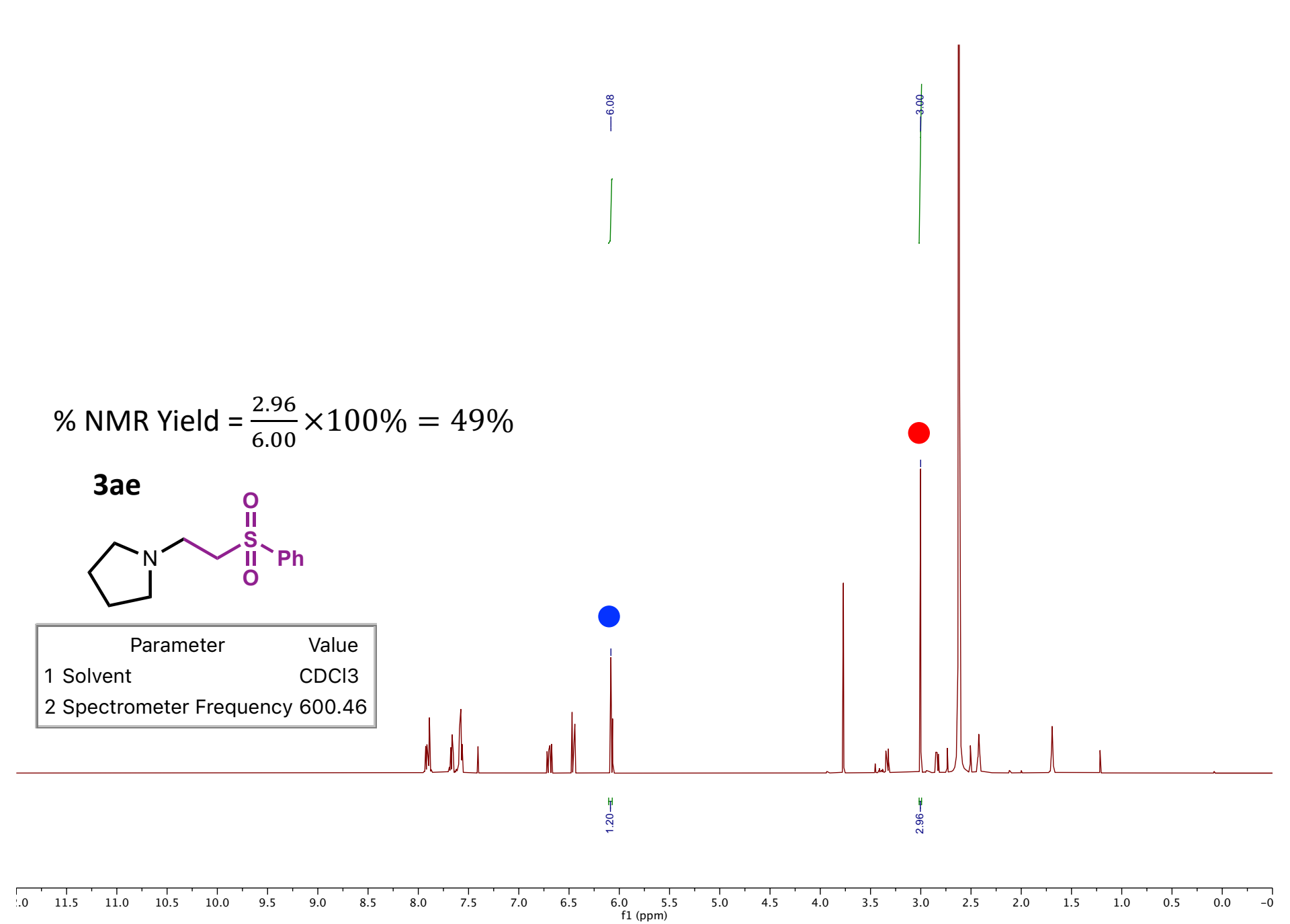
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	500.25



$$\% \text{ NMR Yield} = \frac{0.38}{6.00} \times 100\% = 6\%$$

3ad

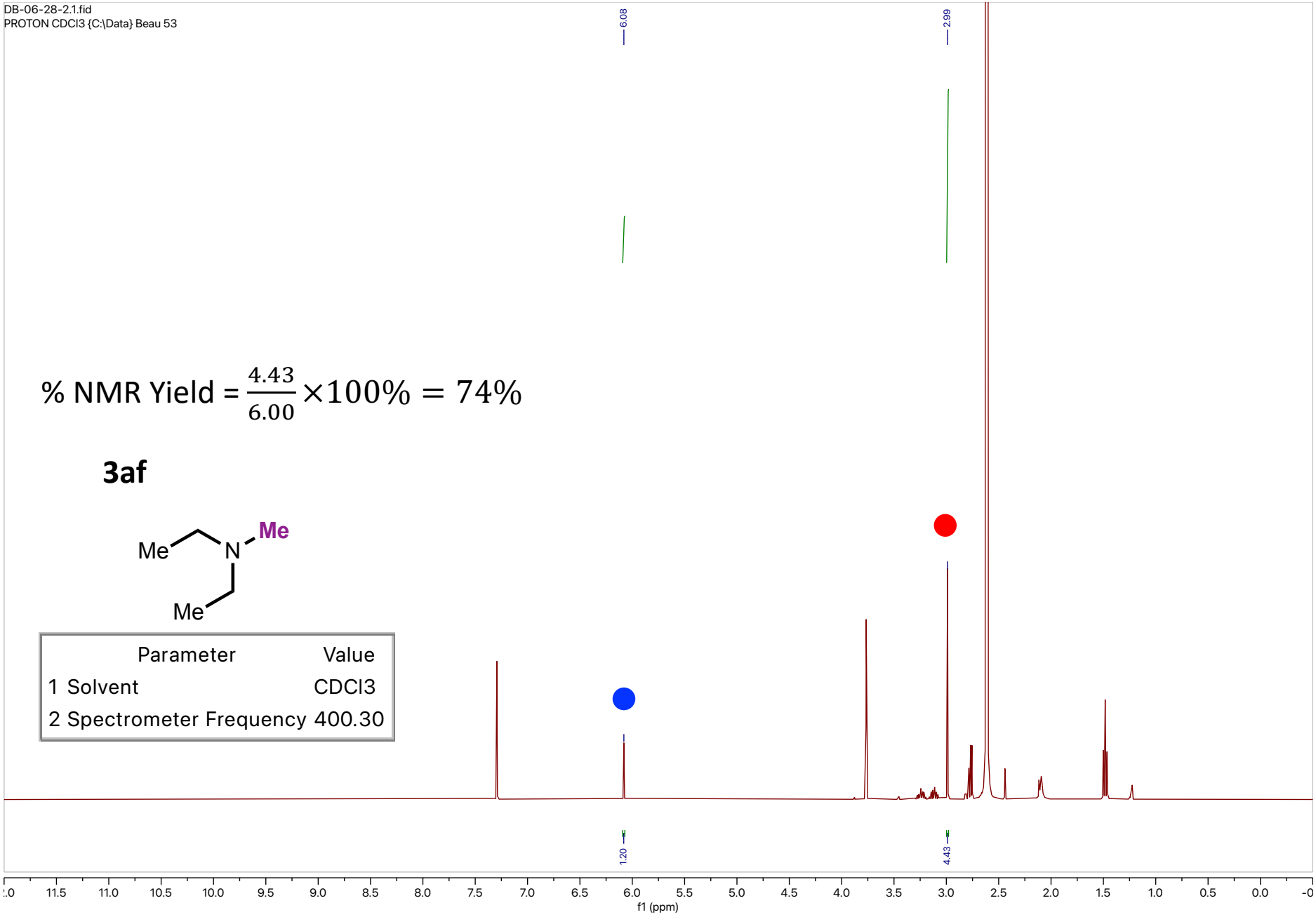
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	500.25



$$\% \text{ NMR Yield} = \frac{2.96}{6.00} \times 100\% = 49\%$$

3ae

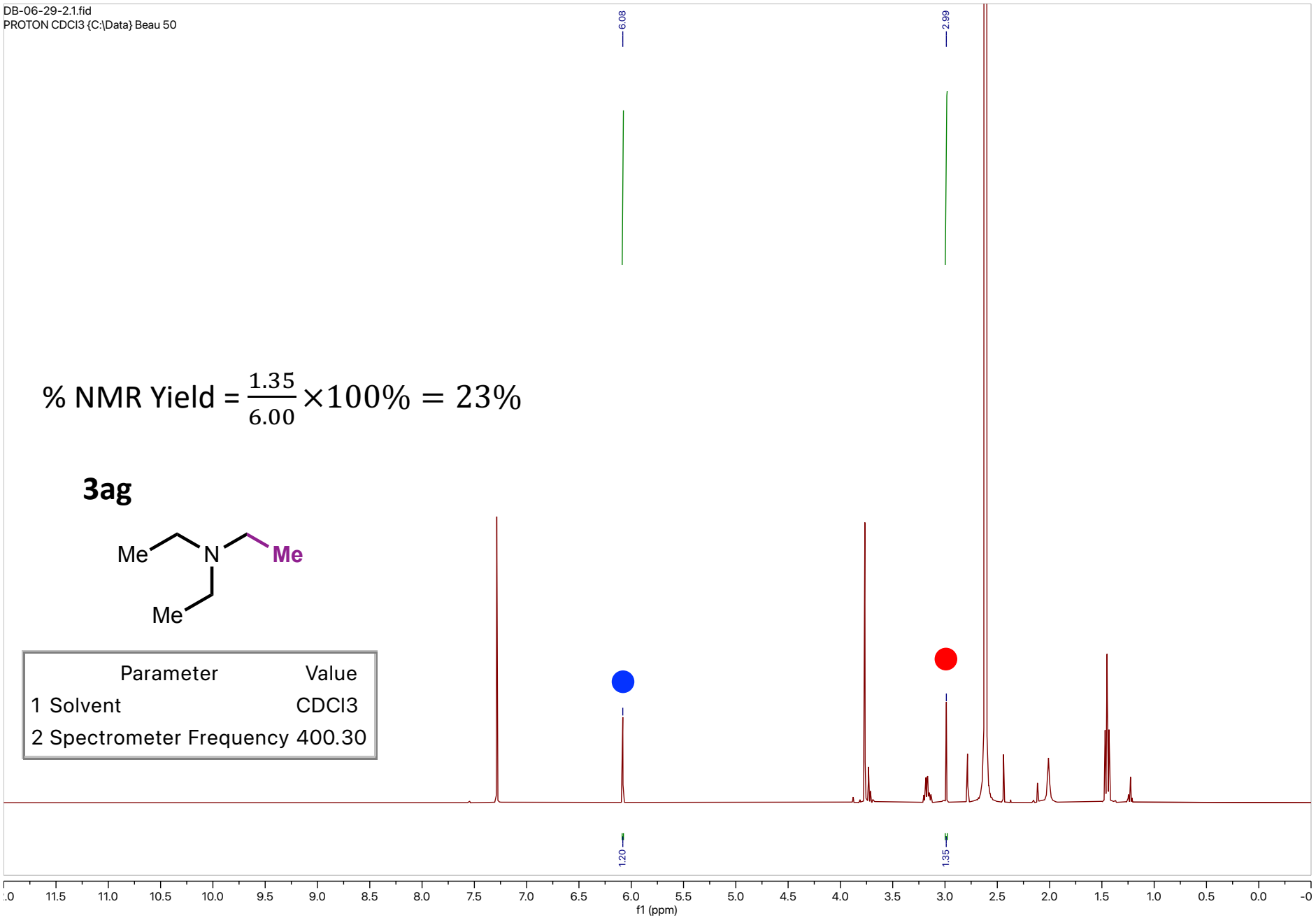
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	600.46

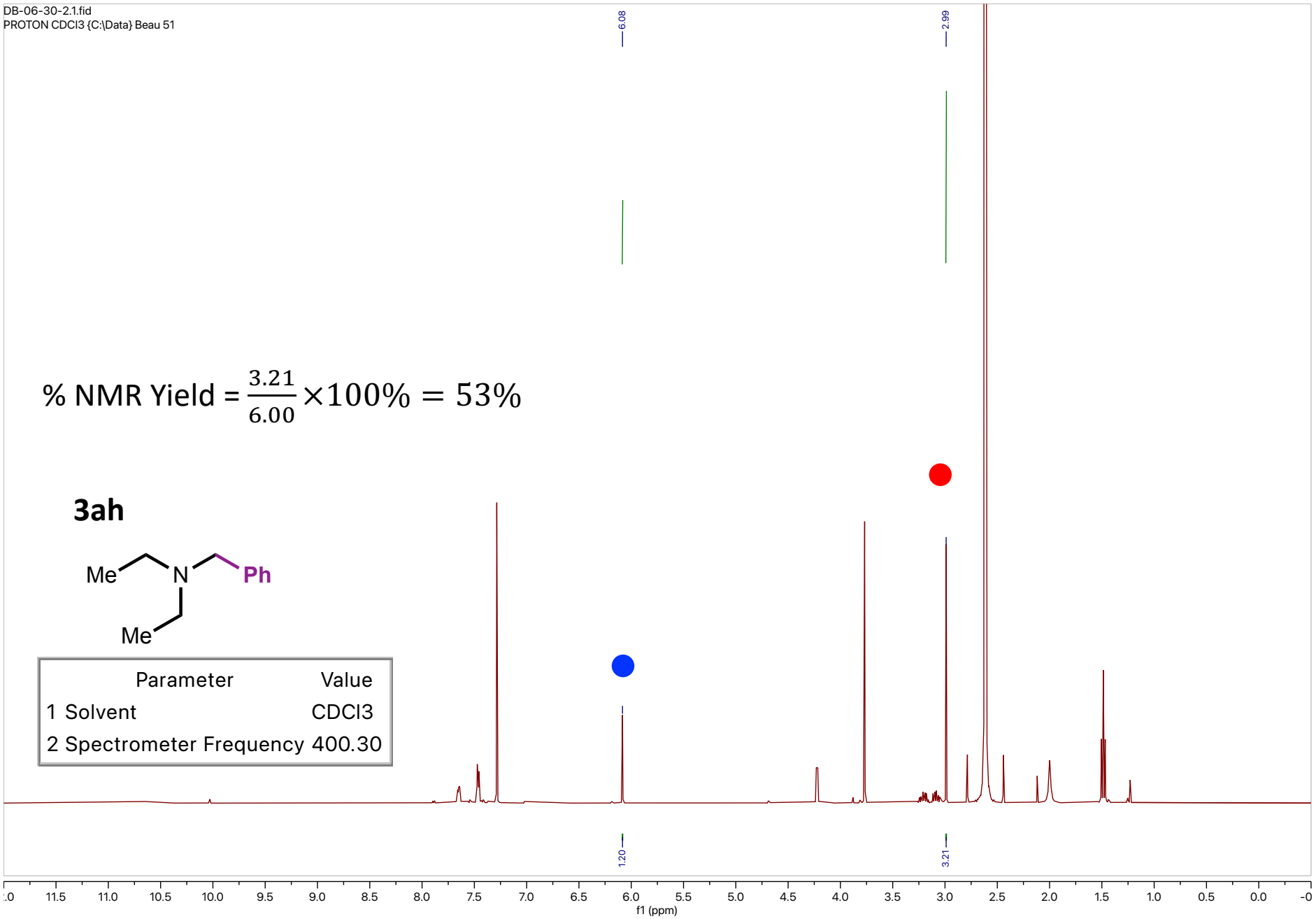


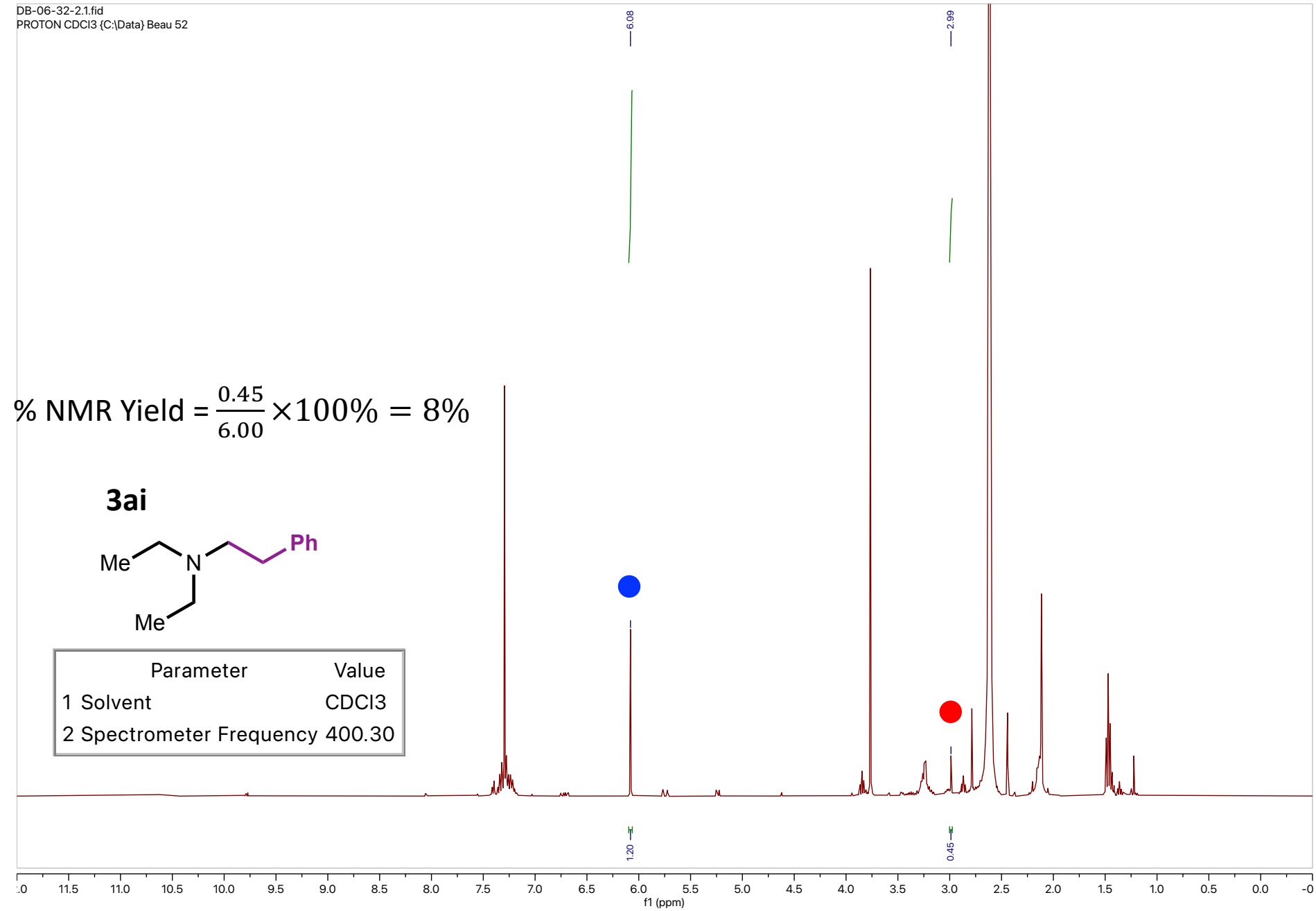
$$\% \text{ NMR Yield} = \frac{4.43}{6.00} \times 100\% = 74\%$$

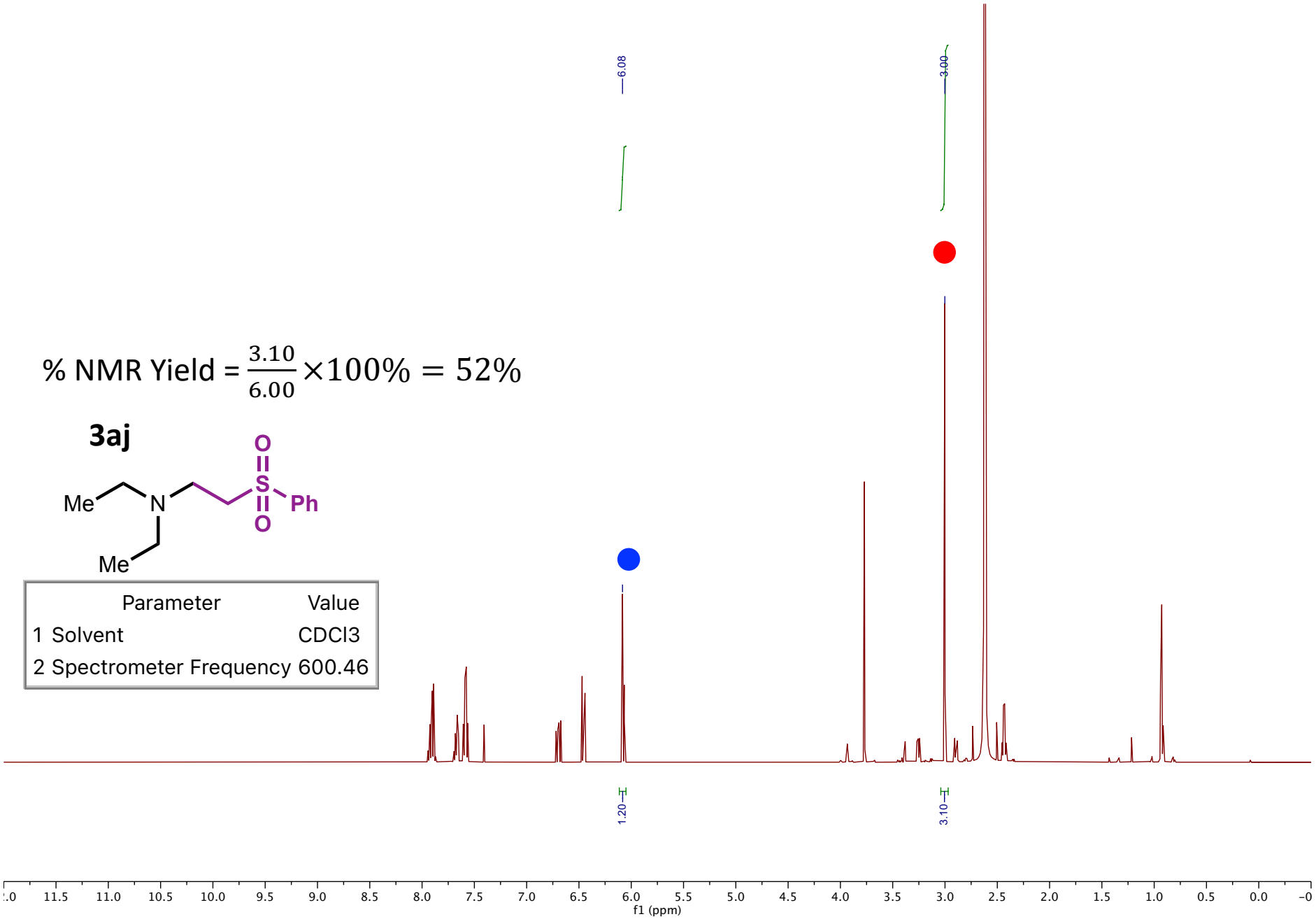
3af

	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	400.30


DB-06-29-2.1.fid
PROTON CDCl3 {C:\Data} Beau 50
6.08
2.99
% NMR Yield = $\frac{1.35}{6.00} \times 100\% = 23\%$
3ag
Me
N
Me
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
1.20
1.35
f1 (ppm)


DB-06-30-2.1.fid
PROTON CDCl3 {C:\Data} Beau 51
6.08
2.99
% NMR Yield = $\frac{3.21}{6.00} \times 100\% = 53\%$
3ah
Me
N
Ph
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
1.20
3.21
f1 (ppm)

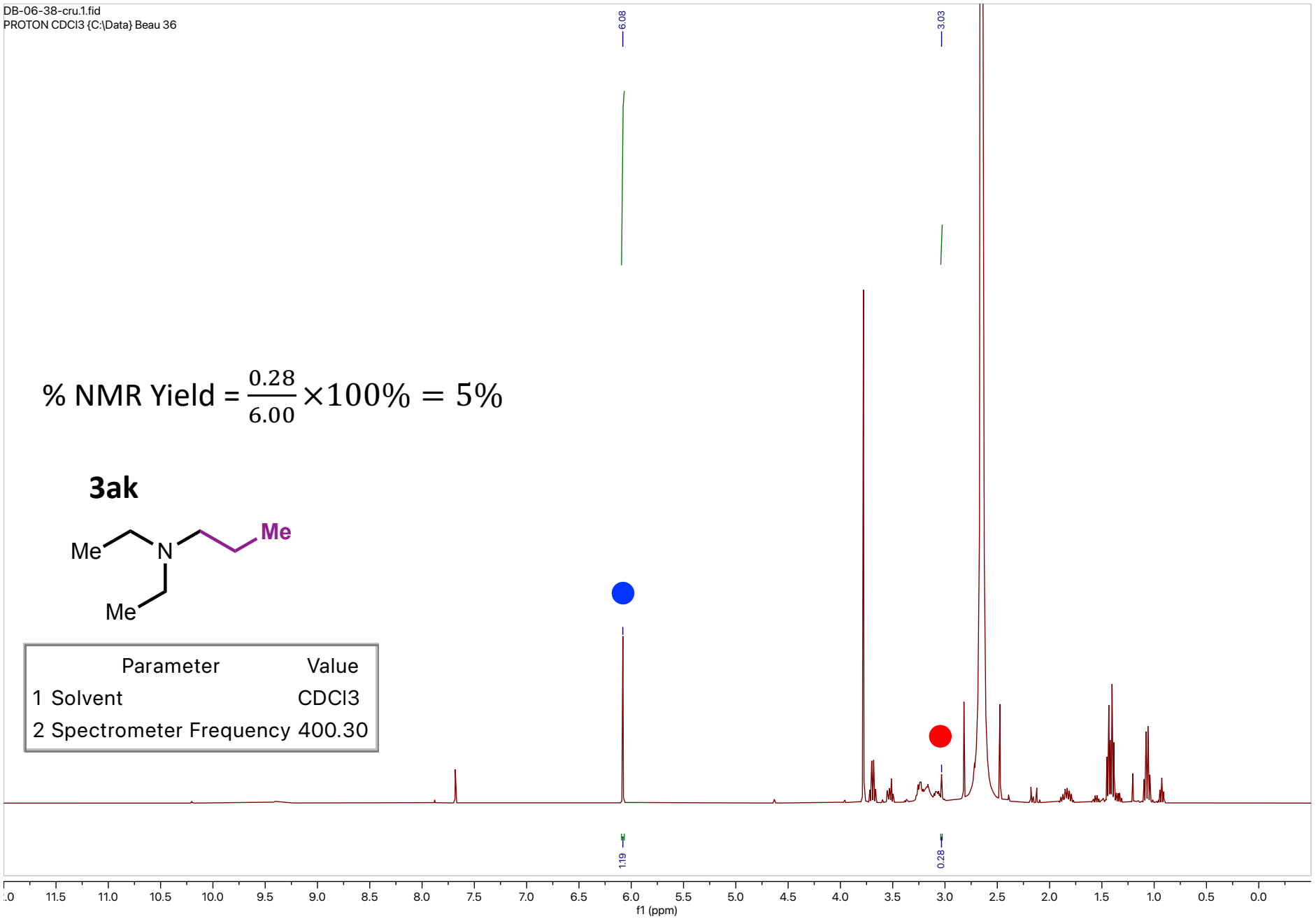

DB-06-32-2.1.fid
PROTON CDCl3 {C:\Data} Beau 52
6.08
2.99
% NMR Yield = $\frac{0.45}{6.00} \times 100\% = 8\%$
3ai
Me
N
Ph
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
1.20
0.45
f1 (ppm)



$$\% \text{ NMR Yield} = \frac{3.10}{6.00} \times 100\% = 52\%$$

3aj

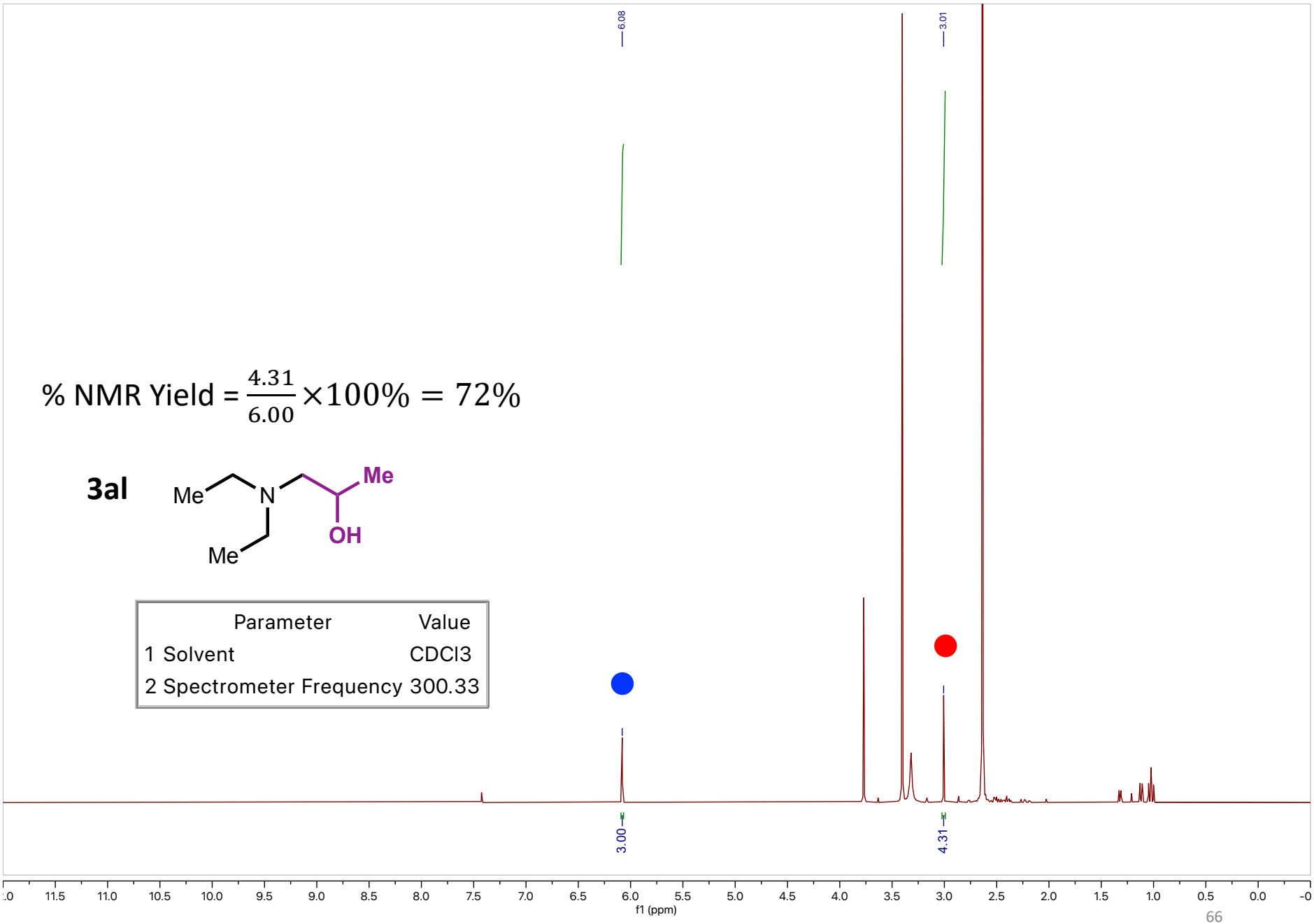
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	600.46



$$\% \text{ NMR Yield} = \frac{0.28}{6.00} \times 100\% = 5\%$$

3ak

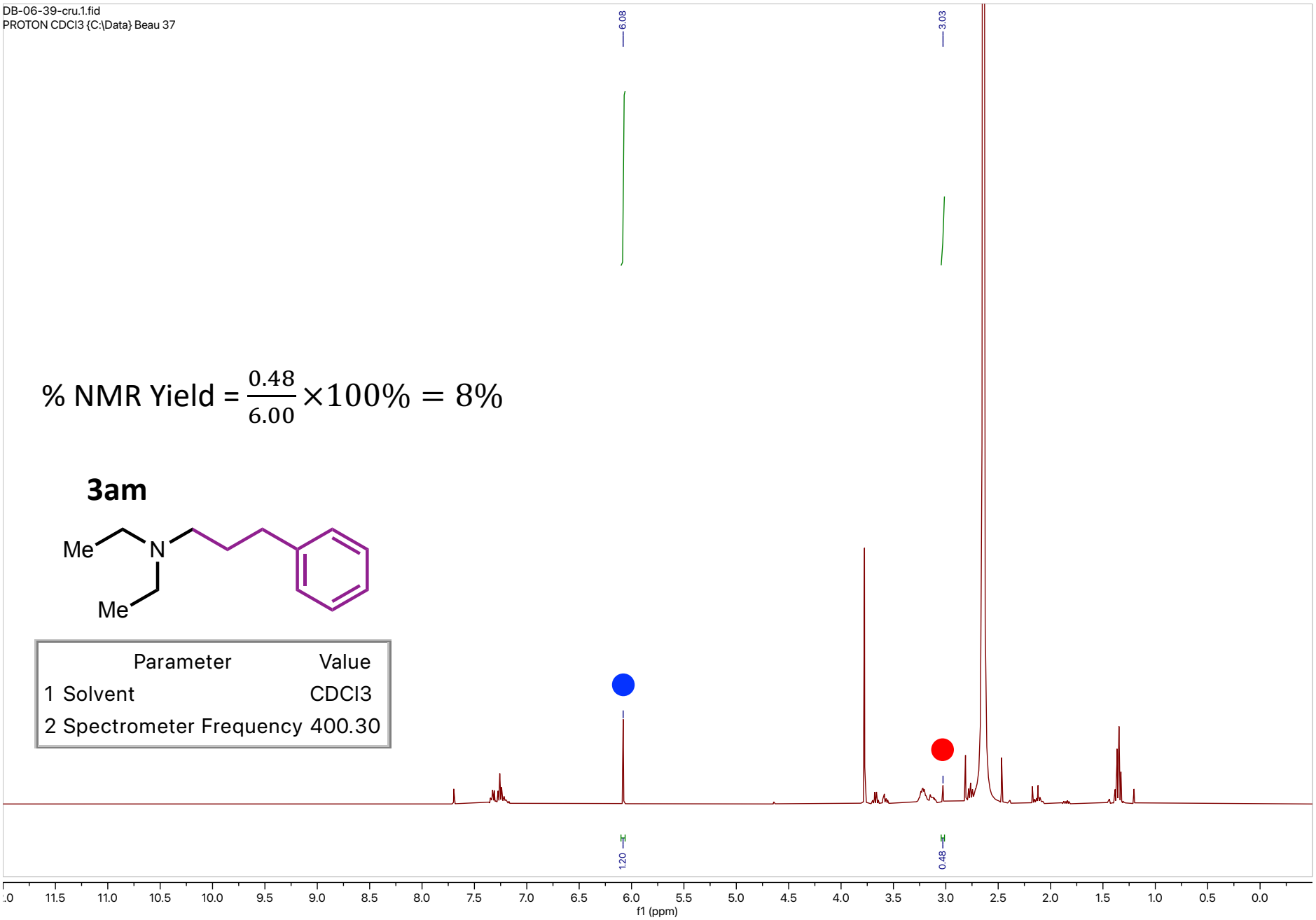
	Parameter	Value
1	Solvent	CDCl3
2	Spectrometer Frequency	400.30

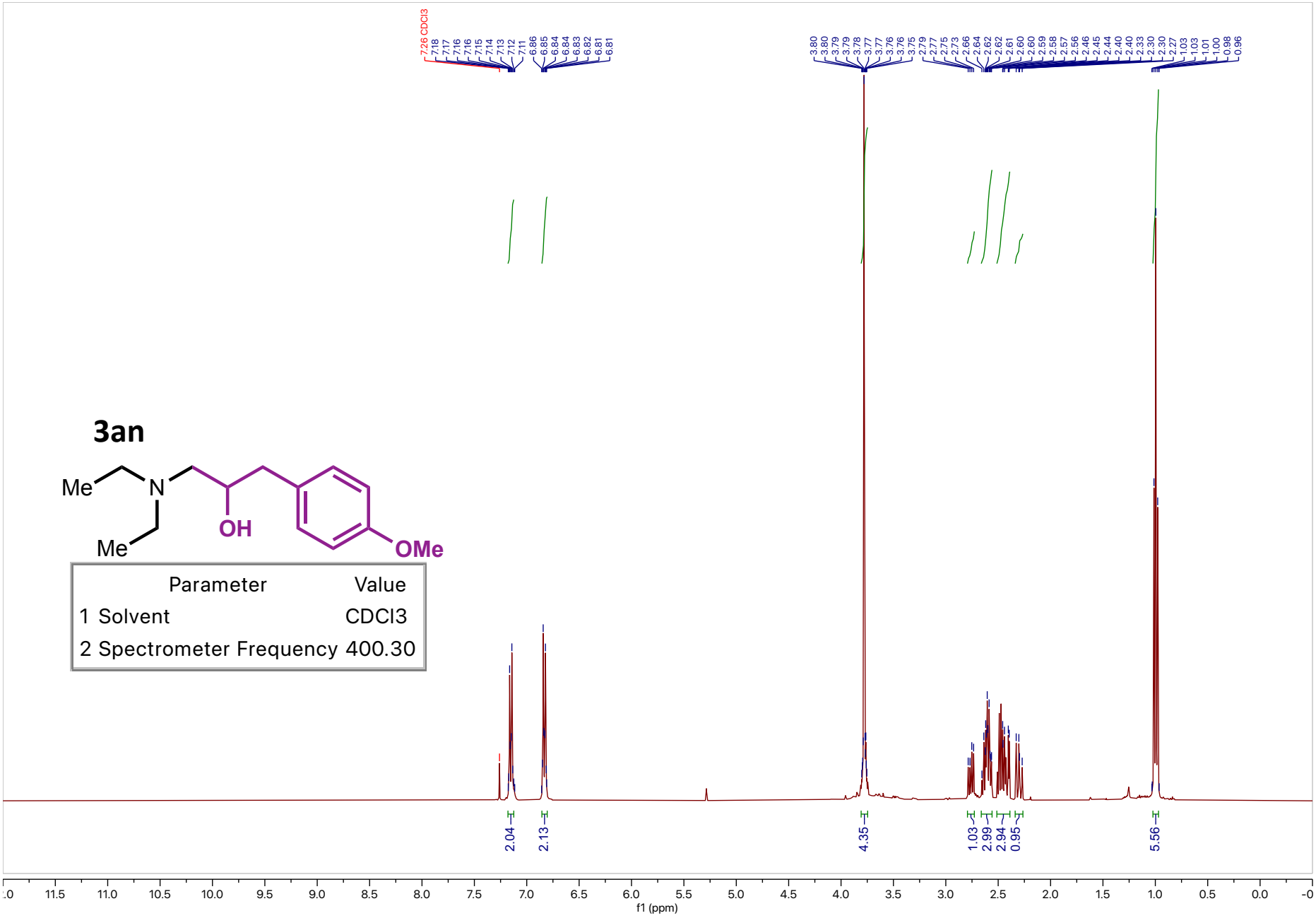


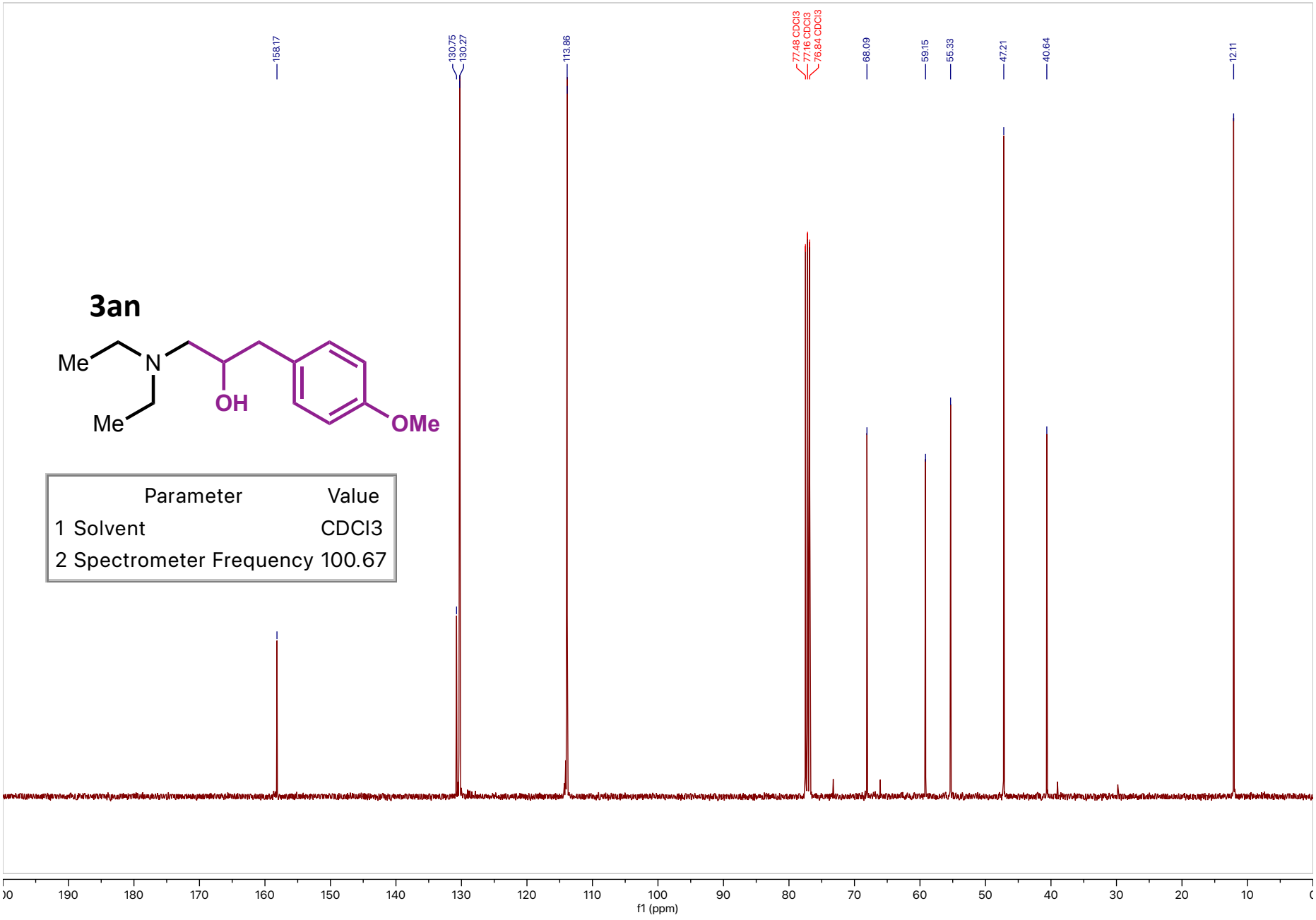
$$\% \text{ NMR Yield} = \frac{4.31}{6.00} \times 100\% = 72\%$$

3al

Parameter	Value
1 Solvent	CDCl3
2 Spectrometer Frequency	300.33


DB-06-39-cru.1.fid
PROTON CDCl3 {C:\Data} Beau 37
6.08
3.03
% NMR Yield = $\frac{0.48}{6.00} \times 100\% = 8\%$
3am
Me
N
Me
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
1.20
0.48
f1 (ppm)


3an
Me
N
Me
OH
OMe
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 400.30
f1 (ppm)


158.17
130.75
130.27
113.86
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
68.09
59.15
55.33
47.21
40.64
12.11
3an
Me
N
Me
OH
OMe
Parameter Value
1 Solvent CDCl3
2 Spectrometer Frequency 100.67
f1 (ppm)