Copper-Catalyzed Modular Amino Oxygenation of Alkenes: Access to Diverse 1,2-Amino Oxygen-Containing Skeletons

Brett N. Hemric, Andy W. Chen, and Qiu Wang*

Department of Chemistry, Duke University, Durham, NC 27708

Email: qiu.wang@duke.edu

SUPPORTING INFORMATION

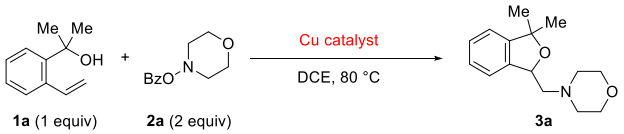
Table of Contents

1.1 Condition Optimization

General Example Optimization Screening Conditions

In a 1 Dram vial, 2-(2-vinylphenyl)propan-2-ol **1a** (32.4 mg, 0.2 mmol, 1.0 equiv), 4-benzoyloxymorpholine **2a** (82.9 mg, 2.0 equiv), copper (II) trifluoromethanesulfonate (14.4 mg, 0.2 equiv), and pyridine *p*-toluenesulfonate (50.3 mg, 1.0 equiv) were combined and 1,2-dichloroethane (1.0 mL) was added. The vial was capped and stirred with Teflon-coated stir bar at 60 °C. The consumption of **2a** was monitored by TLC (25% EtOAc–hexanes). The reaction mixture was filtered through activated, neutral (Brockman Grade I, 58–60Å mesh powder) Al_2O_3 and concentrated *in vacuo* to yield the crude product. To determine yields by quantitative ^{1}H NMR spectroscopy, dibromomethane (7.0 µL, 0.2 mmol) was added by 10-µL microsyringe to the crude reaction mixture in $CDCl_3$ (0.5 mL). The resulting solution was analyzed by ^{1}H NMR with a 90° pulse angle and 2 second relaxation time with 12–16 scans. The resulting spectra were analyzed in MestReNova, with the dibromomethane peak set relative to 2.0. All yields were scaled relative to a normalization curve created with known amounts of the isolated product.

Table S1. Initial Copper Catalyst Loading Screen on Model System.[a]



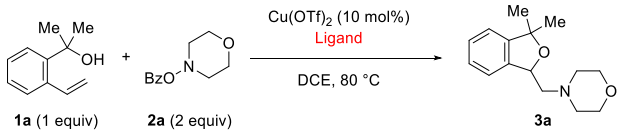
1a (1 equiv) **2a** (2 equiv) **3a**

Entry	Cu catalyst loading (mol%)	**3a**[b] (%)	
		$Cu(OTf)_2$ as catalyst	$[Cu(OTf)]_2$·PhMe as catalyst
1	1	23	22
2	5	27	24
3	10	30	24

4	15	30	24
5	20	30	33
6	30	40	31
7	50	36	33
8	100	47	41

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), Cu catalyst in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^{1}H NMR spectroscopy with dibromomethane as a quantitative standard.

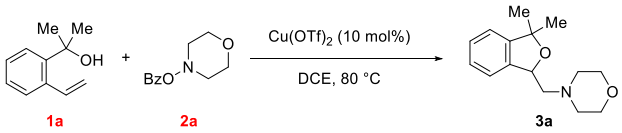
Table S2. Ligand Screen on Model System.[a]



Entry	Ligand	Ligand loading (mol%)	**3a**[b] (%)
1	Pyridine	20	46
2	2,2'-Bipyridine	10	26
3	1,10-Penanthroline	10	29
4	DMEDA	10	26
5	PPh_3	20	23
6	XPhos	10	27
7	BINAP	10	27
8	Xantphos	10	28
9	dppe	10	40
10	dppp	10	26
11	dppb	10	24

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (10 mol%), Ligand, in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^{1}H NMR spectroscopy with dibromomethane as a quantitative standard.

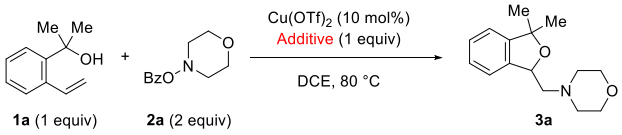
Table S3. Equivalents Screen on Model System.[a]



Entry	**1** (equiv)	**2** (equiv)	**3a**[b] (%)
1	3	1	43
2	2	1	35
3	1	1	30
4	1	2	36
5	1	3	36

[a]Reaction Conditions: **1a**, **2a**, $Cu(OTf)_2$ (0.02 mmol, 0.1 equiv), in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^{1}H NMR spectroscopy with dibromomethane as a quantitative standard.

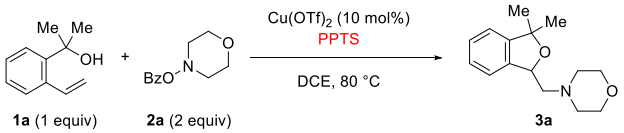
Table S4. Additive Screen on Model System.[a]



Entry	Additive	**3a**[b] (%)
1	2,6-Lutidine	35
2	DBU	0
3	DIPEA	0
4	DABCO	0
5	DMAP	0
6	K_2CO_3	37
7	Cs_2CO_3	29
8	MsOH	0
9	HCO_2H	43
10	BzOH	41
11	PPTS	66
12	NaH_2PO_4	38
13	HFIP	34

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (10 mol%), Additive (1.0 equiv) in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^1H NMR spectroscopy with dibromomethane as a quantitative standard.

Table S5. Pyridinium *p*-toluenesulfonate (PPTS) Equivalents Screen on Model System.[a]

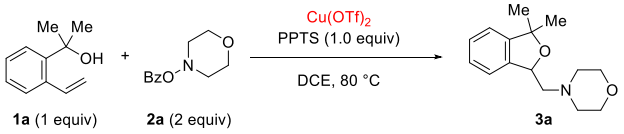


1a (1 equiv) **2a** (2 equiv) **3a**

Entry	PPTS (equiv)	**3a**[b] (%)
1	0.1	44
2	0.5	63
3	1	74
4	1.5	68
5	2	68

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (10 mol%), PPTS in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^1H NMR spectroscopy with dibromomethane as a quantitative standard.

Table S6. Catalyst Loading Screen on Model System.[a]

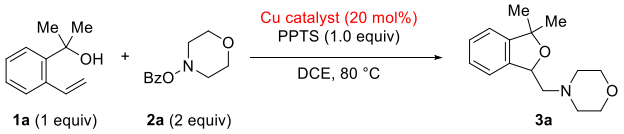


1a (1 equiv) **2a** (2 equiv) **3a**

Entry	$Cu(OTf)_2$ (mol%)	**3a**[b] (%)
1	0	0
2	5	66
3	10	71
4	15	72
5	20	78
6	30	80
7	50	92
8	100	94

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$, PPTS (1.0 equiv) in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^1H NMR spectroscopy with dibromomethane as a quantitative standard.

Table S7. Copper Catalyst Screen on Model System.[a]

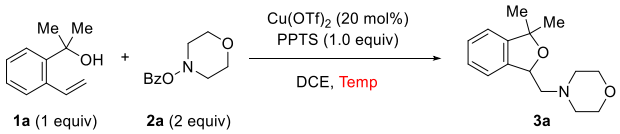


1a (1 equiv) **2a** (2 equiv) **3a**

Entry	Cu catalyst	**3a**[b] (%)
1	$Cu(OTf)_2$	82
2	$Cu(OAc)_2$	78
3	$Cu(eh)_2$	72
4	$Cu(acac)_2$	65
5	$[Cu(OTf)_2 \cdot PhMe$	82
6	CuOAc	75
7	$Cu(MeCN)_4 \cdot BF_4$	75
8	CuI	54

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), Cu catalyst (20 mol%), PPTS (1.0 equiv) in DCE (1.0 mL) at 80 °C. [b]Yield determined by ^{1}H NMR spectroscopy with dibromomethane as a quantitative standard.

Table S8. Temperature Screen on Model System.[a]

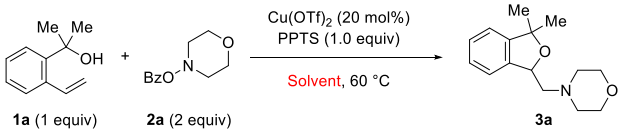


1a (1 equiv) **2a** (2 equiv) **3a**

Entry	Temp (°C)	**3a**[b] (%)
1	100	78
2	80	74
3	60	78
4	40	68
5	20	63

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (20 mol%), PPTS (1.0 equiv) in DCE (1.0 mL) at temp. [b]Yield determined by ^{1}H NMR spectroscopy with dibromomethane as a quantitative standard.

Table S9. Solvent Screen on Model System.[a]



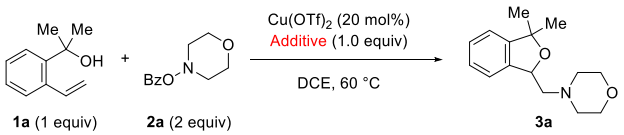
1a (1 equiv) **2a** (2 equiv) **3a**

Entry	Solvent	**3a**[b] (%)
1	DCE	74
2	$CHCl_3$	68
3	THF	66
4	DME	68
5	$PhCF_3$	72
6	PhMe	53

7	PhH	73
8	EtOH	64
9	MeCN	57
10	DMF	38

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (20 mol%), PPTS (1.0 equiv) in solvent (1.0 mL) at 60 °C. [b]Yield determined by ^1H NMR spectroscopy with dibromomethane as a quantitative standard.

Table S10. Pyridinium Scource Screen on Model System.[a]



1a (1 equiv) **2a** (2 equiv) **3a**

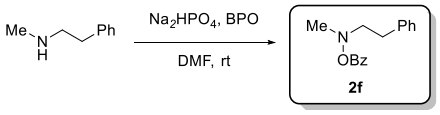
Entry	Additive	**3a**[b] (%)
1	Pyridinium TsOH (PPTS)	72
2	Pyridinium TFA	66
3	Pyridinium TfOH	66
4	4-Isonicotinic acid	36
5	Pyridine	43

[a]Reaction Conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (20 mol%), Additive (1.0 equiv) in DCE (1.0 mL) at 60 °C. [b]Yield determined by ^1H NMR spectroscopy with dibromomethane as a quantitative standard.

1.2 Synthetic Schemes

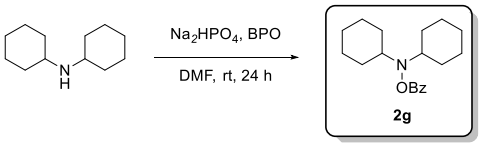
1.2.1 Synthesis of *O*-Benzoylhydroxylamines

_O_-Benzoyl-_N_-methyl-_N_-phenethylhydroxylamine (2f)



2f

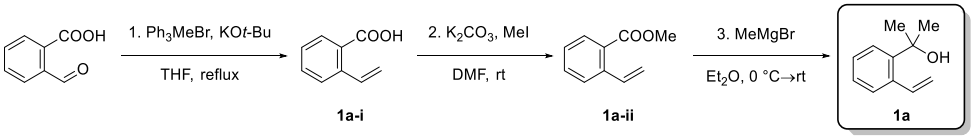
_O_-Benzoyl-_N_,_N_-dicyclohexylhydroxylamine (2g)



2g

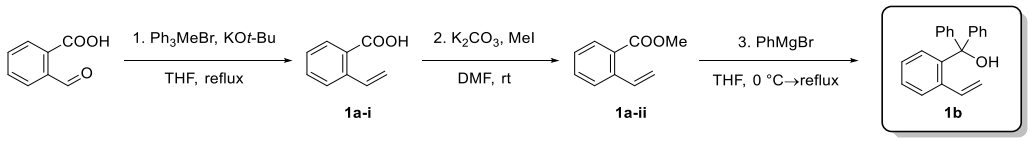
1.2.2 Synthesis of Alcohol Substrates

2-(2-Vinylphenyl)propan-2-ol (1a).

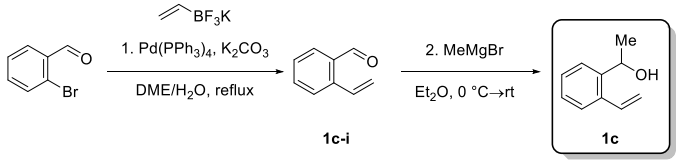


1a-i **1a-ii** **1a**

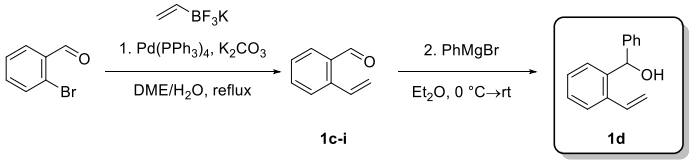
Diphenyl(2-vinylphenyl)methanol (1b).



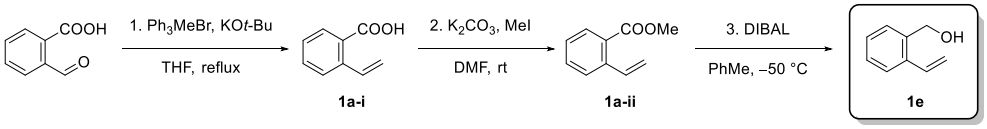
1-(2-Vinylphenyl)ethan-1-ol (1c).



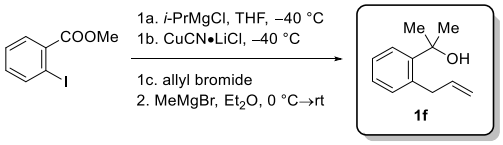
Phenyl(2-vinylphenyl)methanol (1d).



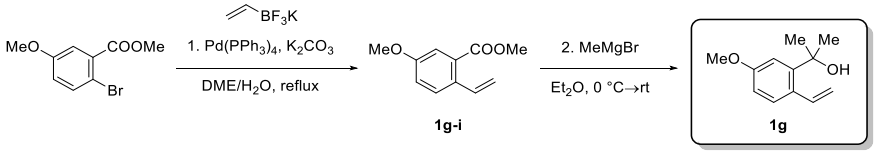
(2-Vinylphenyl)methanol (1e).



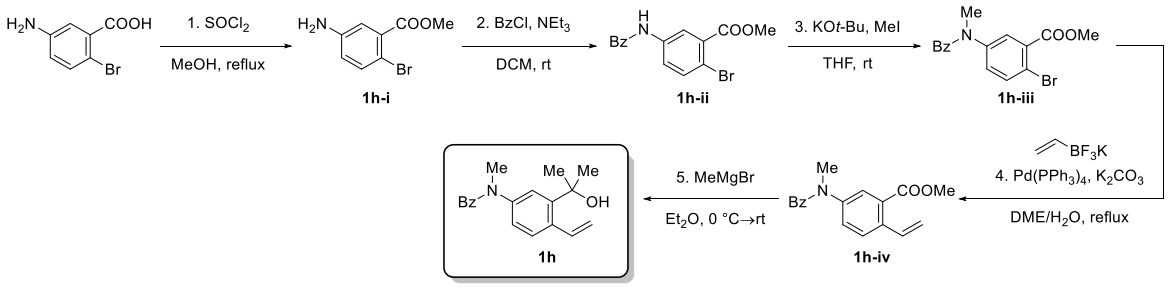
2-(2-Allylphenyl)propan-2-ol (1f).



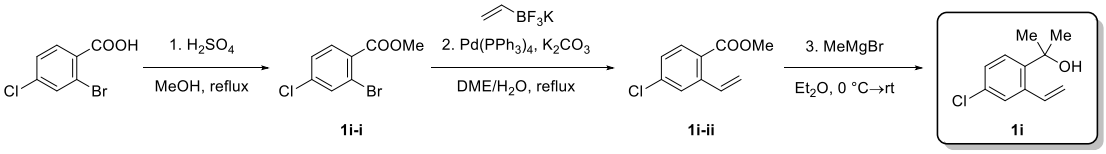
2-(5-Methoxy-2-vinylphenyl)propan-2-ol (1g).



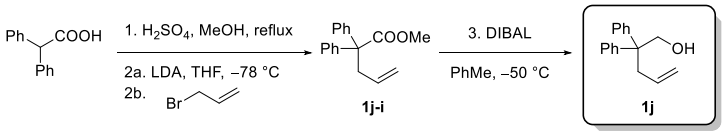
***N*-(3-(2-Hydroxypropan-2-yl)-4-vinylphenyl)-N-methylbenzamide (1h).**



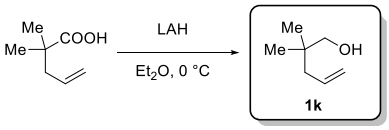
2-(4-Chloro-2-vinylphenyl)propan-2-ol (1i).



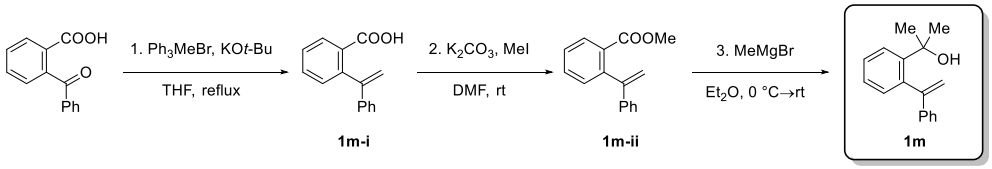
2,2-Diphenylpent-4-en-1-ol (1j).



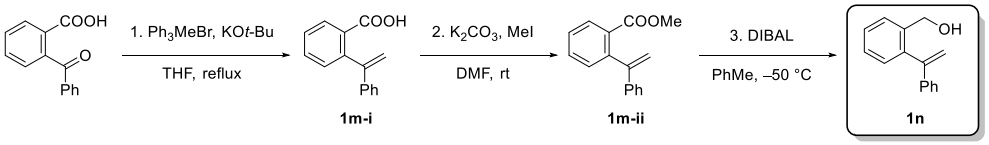
2,2-Dimethylpent-4-en-1-ol (1k).



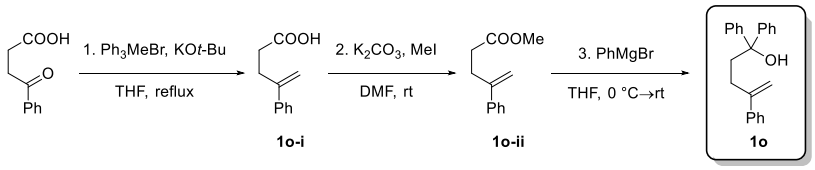
2-(2-(1-Phenylvinyl)phenyl)propan-2-ol (1m).



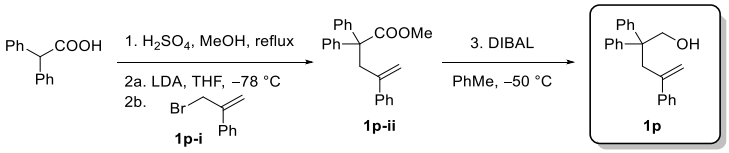
(2-(1-Phenylvinyl)phenyl)methanol (1n).



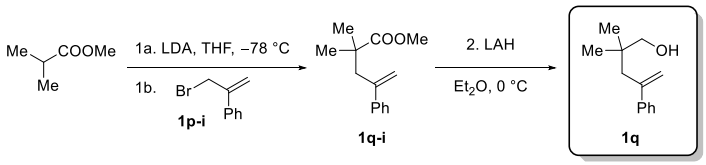
1,1,4-Triphenylpent-4-en-1-ol (1o).



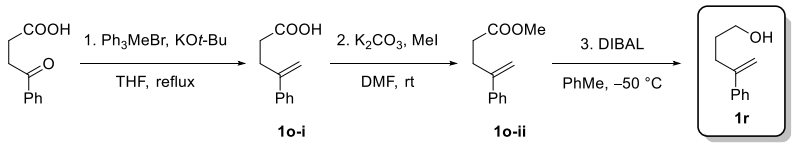
2,2,4-Triphenylpent-4-en-1-ol (1p).



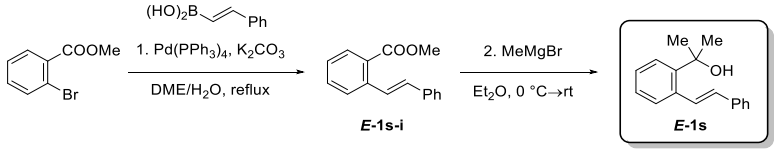
2,2-Dimethyl-4-phenylpent-4-en-1-ol (1q).



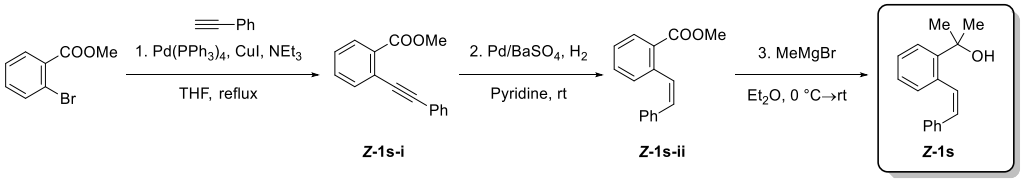
4-Phenylpent-4-en-1-ol (1r).



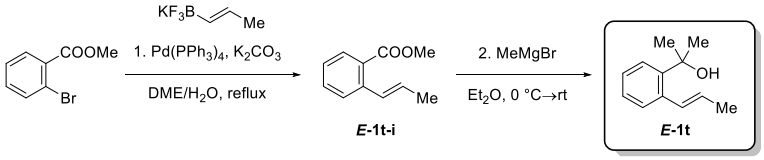
(*E*)-2-(2-Styrylphenyl)propan-2-ol (*E*-1s).



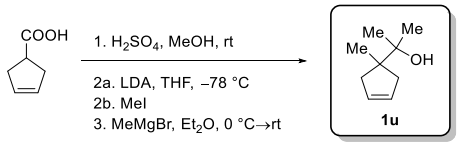
(*Z*)-2-(2-Styrylphenyl)propan-2-ol (*Z*-1s).



(*E*)-2-(2-(Prop-1-en-1-yl)phenyl)propan-2-ol (*E*-1t).

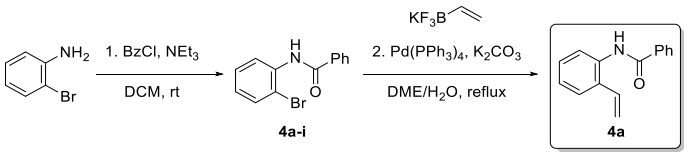


2-(1-Methylcyclopent-3-en-1-yl)propan-2-ol (1u).

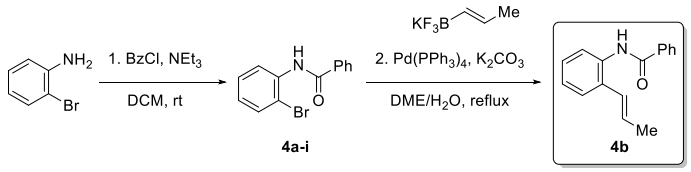


1.2.3 Synthesis of Additional Oxygen Source Substrates

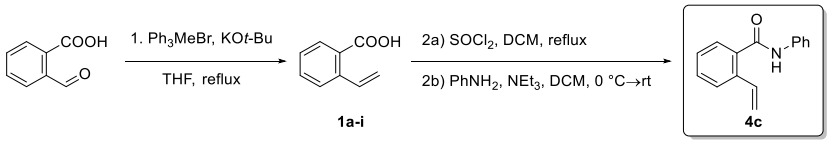
***N*-(2-Vinylphenyl)benzamide (4a).**



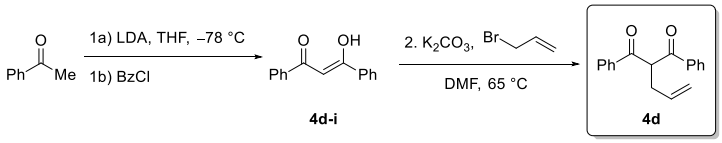
***N*-(2-Vinylphenyl)benzamide (4b).**



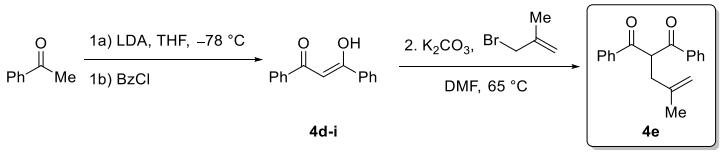
***N*-Phenyl-2-vinylbenzamide (4c).**



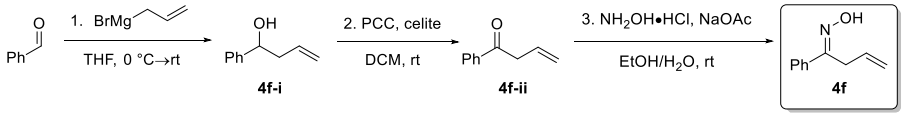
2-Allyl-1,3-diphenylpropane-1,3-dione (4d).



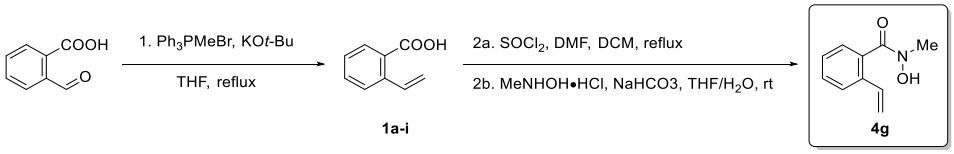
2-(2-Methylallyl)-1,3-diphenylpropane-1,3-dione (4e).



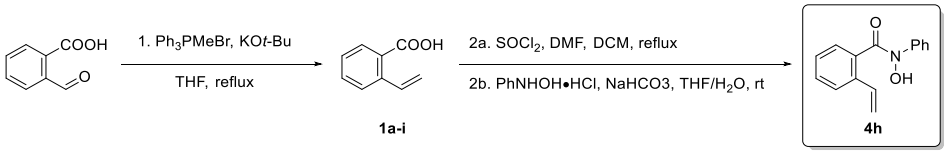
(*E*)-1-phenylbut-3-en-1-one oxime (4f).



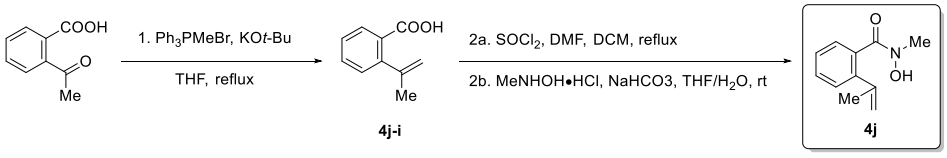
***N*-hydroxy-*N*-methyl-2-vinylbenzamide (4g).**



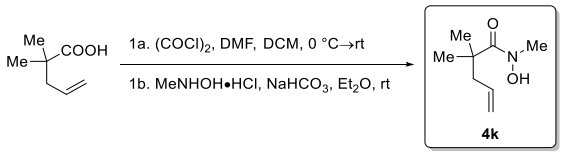
***N*-hydroxy-*N*-phenyl-2-vinylbenzamide (4h).**



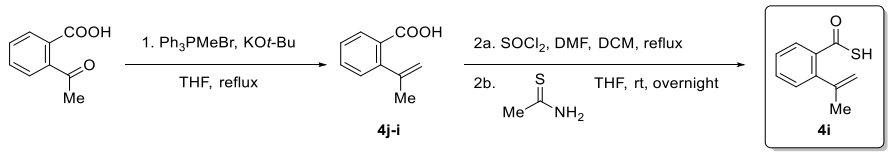
***N*-hydroxy-*N*-methyl-2-(prop-1-en-2-yl)benzamide (4j).**



***N*-hydroxy-*N*,2,2-trimethylpent-4-enamide (4k).**

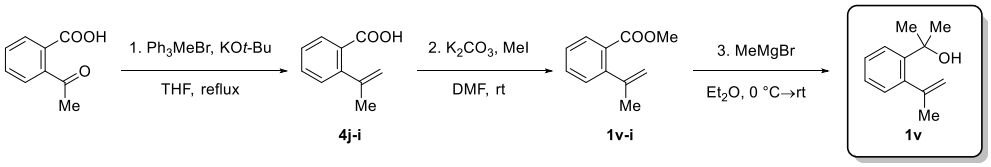


2-(Prop-1-en-2-yl)benzothioic *S*-acid (4i).

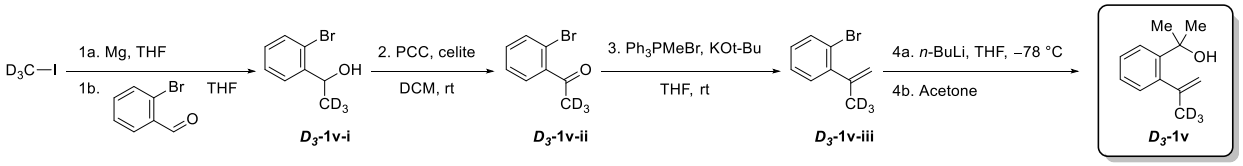


1.2.4 Synthesis of substrates for mechanistic studies

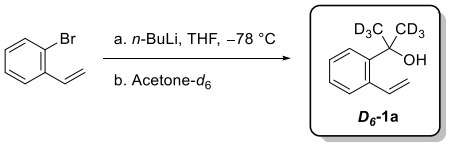
2-(2-(Prop-1-en-2-yl)phenyl)propan-2-ol (1v).



2-(2-(Prop-1-en-2-yl-3,3,3-d_3)phenyl)propan-2-ol (D_3-1v).

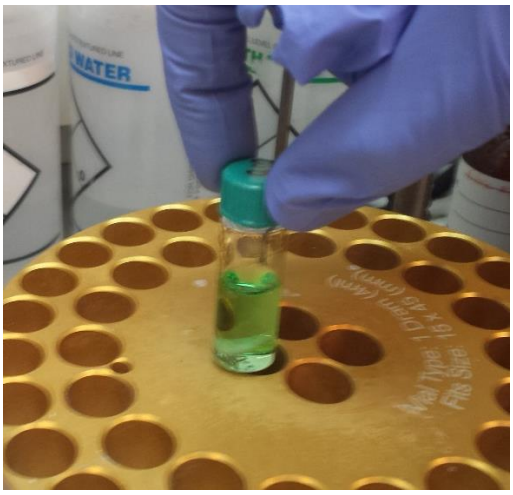


2-(2-Vinylphenyl)propan-1,1,1,3,3,3-d_6-2-ol (D_6-1a).



2-(2-Vinylphenyl)propan-1,1,1,3,3,3-d_6-2-ol (D_6-1a): 2-Bromostyrene (732.2 mg, 4.0 mmol, 1.0 equiv) was added to a reaction vessel and vacuum purged three times, backfilling with N_2. THF (12.0 mL) was added. To the solution at –78 °C, was added slowly *n*-BuLi (2.5 M in hexanes, 1.9 mL, 1.2 equiv) over 9 min. The resulting yellow solution was stirred at –78 °C for 40 min followed by slow addition of acetone-d_6 (0.44 mL, 1.5 equiv) over 2 min. The resulting solution was stirred at –78 °C for 5 min and then the yellow color became clear. The solution was then warmed to room temperature over 1 h. The reaction was quenched with a saturated aqueous solution of NH_4Cl (15 mL). The aqueous layer was extracted with EtOAc (25 mL x 3). The combined organic layers were washed with brine (30 mL), dried with Na_2SO_4, and concentrated *in vacuo* to yield the crude alcohol. Purification by flash column chromatography (5% EtOAc–hexanes to 10% EtOAc–hexanes) afforded alcohol ***D*$_6$-1a** as a white solid (547.4 mg, 3.3 mmol, 81%). $\mathbf{R}_f$ = 0.35 (10% EtOAc–hexanes); **^{1}H NMR** ($CDCl_3$, 400 MHz): δ 7.64 (dd, *J* = 17.4, 10.9 Hz, 1H), 7.51–7.40 (m, 2H), 7.29–7.21 (m, 2H), 5.52 (dd, *J* = 17.4, 1.5 Hz, 1H), 5.28 (dd, *J* = 10.9, 1.5 Hz, 1H), 1.92 (s, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz): δ 144.7, 138.1, 137.2, 128.5, 127.4, 127.3, 124.9, 115.2, 73.3, 30.3 (multiplet); **FTIR** (thin film): cm^{-1} 3364 (broad), 2230, 1623, 1477, 1131, 1045, 1107, 911, 769, 757; **HRGCMS-ESI** (m/z) Calcd for ($C_{11}H_8D_6O$) ([M]$^+$): 168.14158; found: 168.14147.

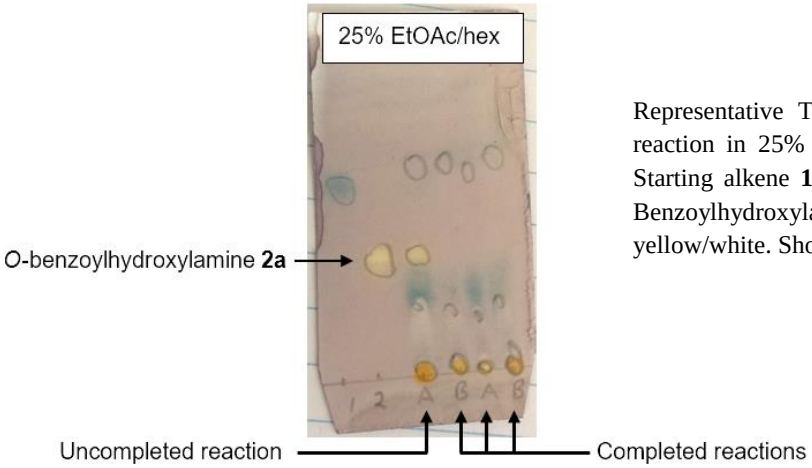
1.3 Amino Oxygenation Representative Pictures



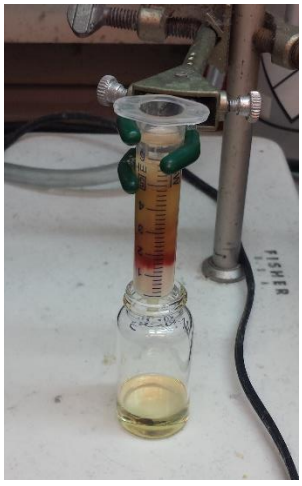
The reaction generally (but not always) became red/brown as reaction progressed.

For reactions with all solid components, add all solids to vial, then add DCE. Add vial to a pre-heated hotplate.

For reactions with a liquid component, add all solids to vial, then add DCE, followed by the liquid component. Add vial to a pre-heated hotplate.



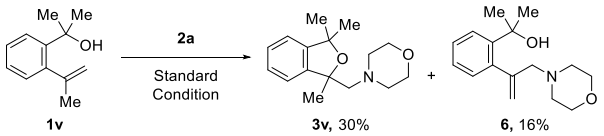
Representative TLC for the amino oxygenation reaction in 25% EtOAc/hex using vanillin stain. Starting alkene **1/4** will vary in color and R_f. *O*-Benzoylhydroxylamines (**2**) generally stain yellow/white. Shown is **2a**.



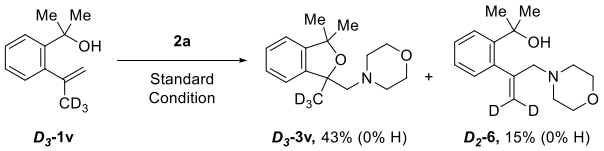
Representative filtration for the amino oxygenation reaction using Al_2O_3 (Brockman grade I, 58-60Å). For best results, dampen the Al_2O_3 plug (made using 5 mL syringe with cotton on top and bottom of the Al_2O_3) with 100% EtOAc, then add the crude reaction mixture. Allow the crude to gravity filter with 100% EtOAc for ~5 mL, then 100% EtOAc can be flushed through with air/N_2 to a total filtrate volume of ~15 mL. The filtrate is generally a yellow/orange solution, while with Al_2O_3 retains the red/brown color with a blue/green band (copper) at the top (intensity of this blue/green band varies by substrate).

1.4 Mechanism studies and characterization data

1.4.1 β-Hydride Elimination Study

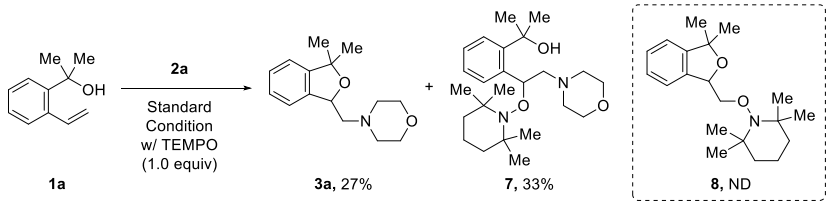


4-((1,3,3-Trimethyl-1,3-dihydroisobenzofuran-1-yl)methyl)morpholine (3v) and 2-(2-(3-Morpholinoprop-1-en-2-yl)phenyl)propan-2-ol (6). Run using standard conditions. Isolated by flash column chromatography (addition of 0.2 mL TFA to crude reaction mixture, 50% EtOAc–hexanes, then 50% EtOAc–hexanes with 5% NEt_3), followed by flash column chromatography (100% hexanes to 50% EtOAc–hexanes).

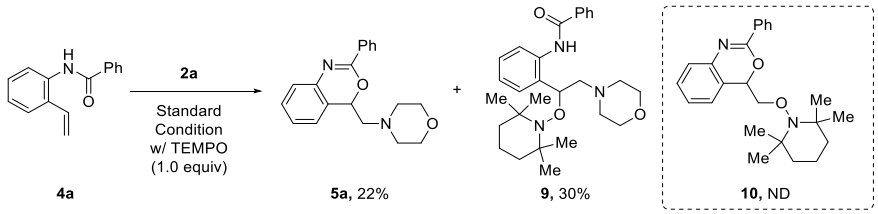


4-((3,3-Dimethyl-1-(methyl-d_3)-1,3-dihydroisobenzofuran-1-yl)methyl)morpholine (D_3-3v) and 2-(2-(3-Morpholinoprop-1-en-2-yl-1,1-d_2)phenyl)propan-2-ol (D_2-6). Run using standard conditions. Isolated by flash column chromatography (addition of 0.2 mL TFA to crude reaction mixture, 50% EtOAc–hexanes, then 50% EtOAc–hexanes with 5% NEt_3), followed by flash column chromatography (100% hexanes to 50% EtOAc–hexanes)

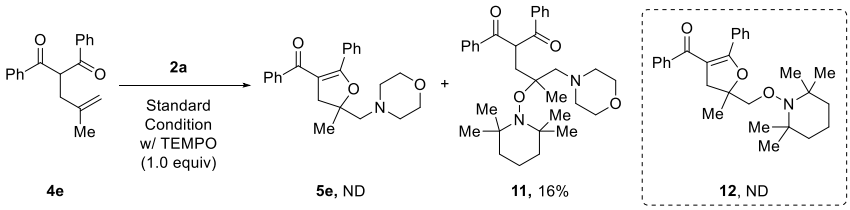
1.4.2 Radical Trapping Study



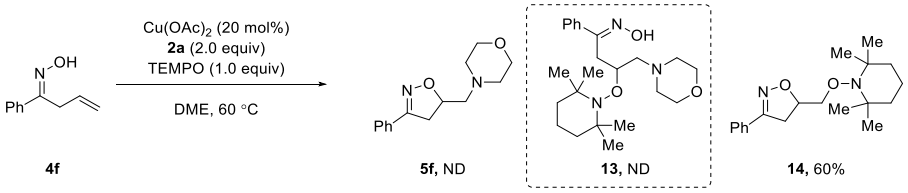
2-(2-(2-Morpholino-1-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)phenyl)propan-2-ol (7). Synthesized using standard conditions with addition of TEMPO (1.0 equiv). Isolated by flash column chromatography (100% hexanes to 70% EtOAc–hexanes) as a colorless oil (53.2 mg, 33%).



***N*-(2-(2-Morpholino-1-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)phenyl)benzamide (9).** Synthesized by standard conditions with addition of TEMPO (1.0 equiv). Isolated by flash column chromatography (100% hexanes to 50% EtOAc–hexanes) as a white solid (55.9 mg, 30%).

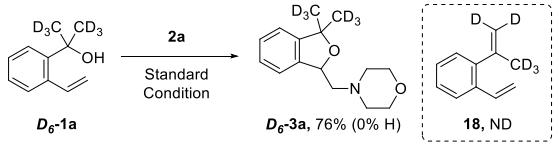


2-(2-Methyl-3-morpholino-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propyl)-1,3-diphenylpropane-1,3-dione (11). Synthesized by standard conditions with addition of TEMPO (1.0 equiv). Isolated by flash column chromatography (100% hexanes to 75% EtOAc–hexanes) as a colorless oil (33.1 mg, 16%).



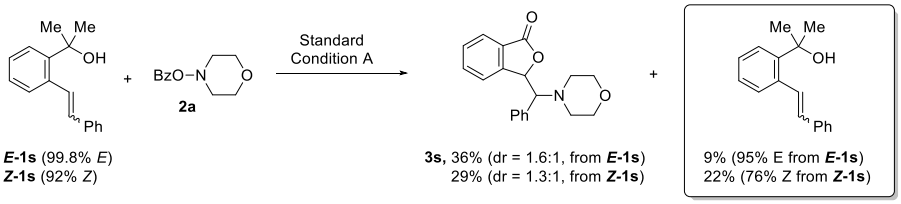
3-Phenyl-5-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)-4,5-dihydroisoxazole (14). Synthesized by standard condition with the following modification: with copper(II) acetate instead of copper(II) trifluoromethanesulfonate, 1,2-dimethoxyethane instead of 1,2-dichloroethane, and with addition of TEMPO (1.0 equiv). Isolated by flash column chromatography (100% hexanes to 70% EtOAc–hexanes) as a white solid (38.2 mg, 60%).

1.4.3 Study Eliminating Dehydration as an Intermediate in the Productive Reaction Pathway



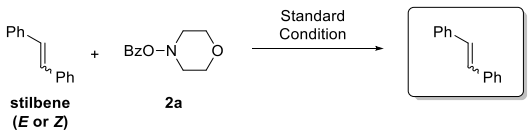
4-((3,3-Bis(methyl-*d*$_3$)-1,3-dihydroisobenzofuran-1-yl)methyl)morpholine (*D*$_6$-3a). Synthesized using standard conditions. Isolated by flash column chromatography (addition of 0.2 mL TFA to crude reaction mixture, 50% EtOAc–hexanes, then 50% EtOAc–hexanes with 5% NEt_3) as a colorless oil (77.2 mg, 76%). $\mathbf{R}_f$ = 0.40 (5% MeOH–CH_2Cl_2); **^{1}H NMR** ($CDCl_3$, 400 MHz): δ 7.28–7.19 (m, 3H), 7.09–7.05 (m, 1H), 5.32 (dd, *J* = 7.5, 4.0 Hz, 1H), 3.74 (t, *J* = 4.6 Hz, 4H), 2.70 (dd, *J* = 13.1, 4.0 Hz, 1H), 2.60 (dd, *J* = 13.1, 7.5 Hz, 1H), 2.73–2.50 (m, 4H); **^{13}C NMR** ($CDCl_3$, 125 MHz): δ 147.2, 140.1, 127.7, 127.1, 121.5, 120.4, 85.0, 79.2, 66.8, 65.9, 54.4, 29.1 (multiplet), 28.0 (multiplet); **FTIR** (thin film): cm^{-1} 2852, 2807, 1455, 1116, 1036, 1009, 865, 750; **HRLCMS-ESI** (m/z) Calcd for ($C_{15}H_{16}D_6NO_2$) ($[M+H]^+$): 254.2022; found: 254.2025.

1.4.4 Study on the Reversibility of Amine Addition to the Alkene



In the study of the alkene scope, the geometric purity of starting alkene ***E*-1s** was 99.8% *E*, while ***Z*-1s** was 92% *Z*. When the alkene was recovered, it was noted that the reaction of ***E*-1s** only gave 95% *E* in the recovered alkene, while ***Z*-1s** only gave 76% *Z* in the recovered alkene. This intriguing result led us to further explore the idea of alkene reversibility.

Table S11. Loss of Stilbene Geometric Purity in Reaction Conditions.[a]



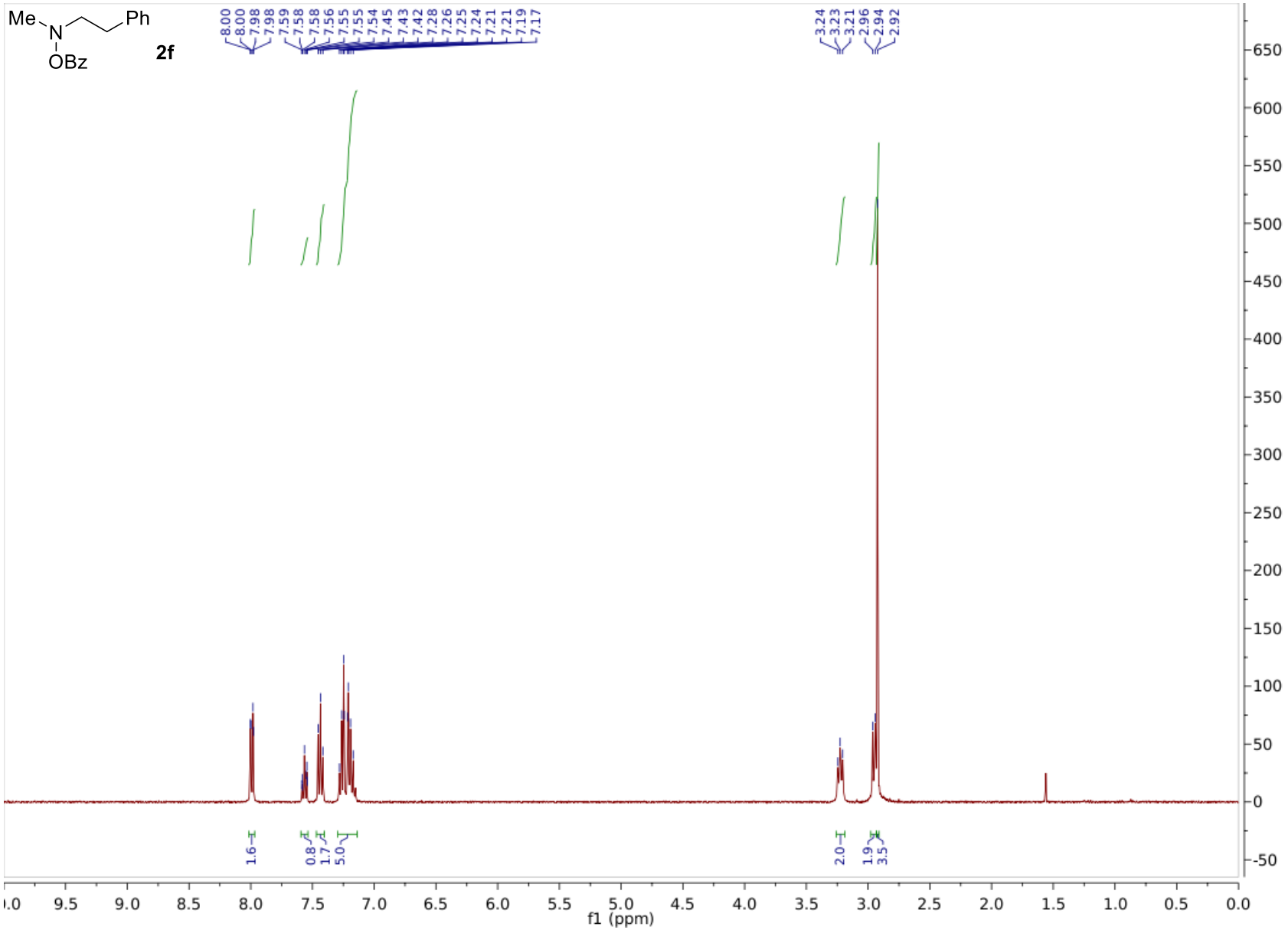
Starting Alkene	**2a** (equiv)	Recovered Alkene[bc]	
E-stilbene (**99% *E***)	2	10%	**83% *E***

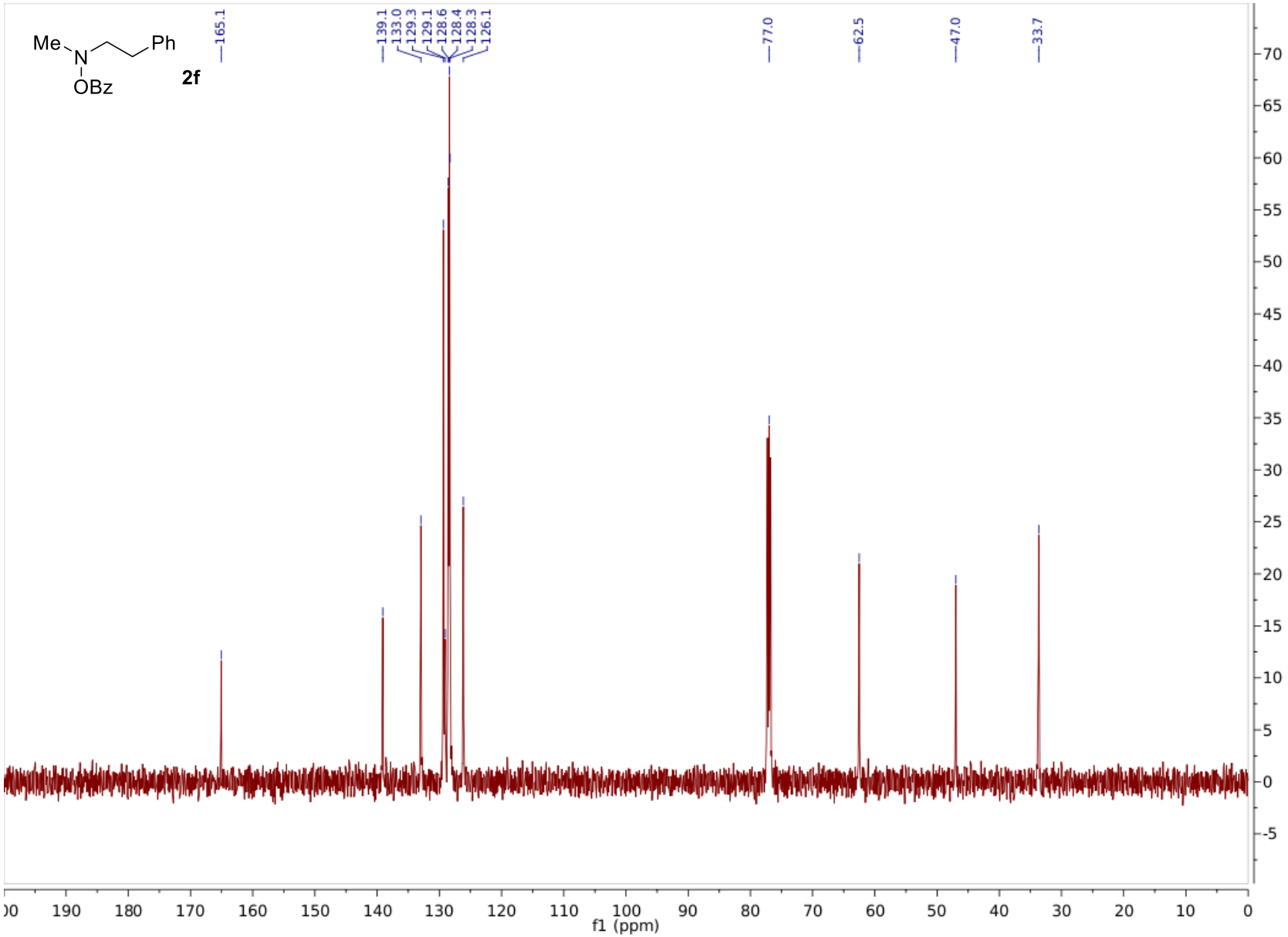
Z-stilbene (**99% *Z***)	2	28 %	**84% *Z***
E-stilbene (**99% *E***)	0	100%	**99% *E***
Z-stilbene (**99% *Z***)	0	99%	**99% *Z***

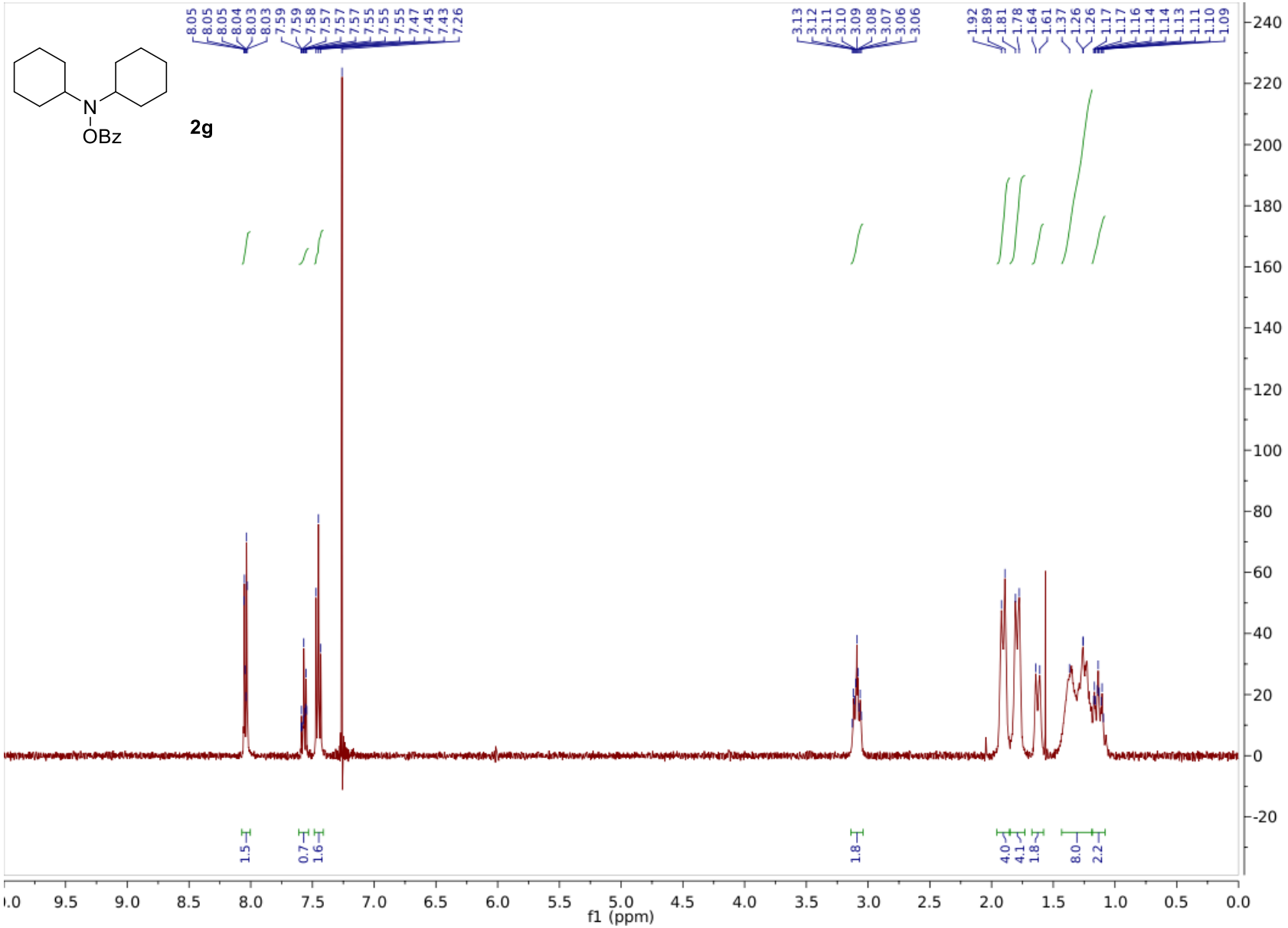
Standard conditions: stilbene (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), $Cu(OTf)_2$ (0.2 equiv), PPTS (1.0 equiv) in DCE (1.0 mL) at 60 °C for 1 h. [b]Isolation yield. [c]Geometric isomer ratios determined by GCMS.

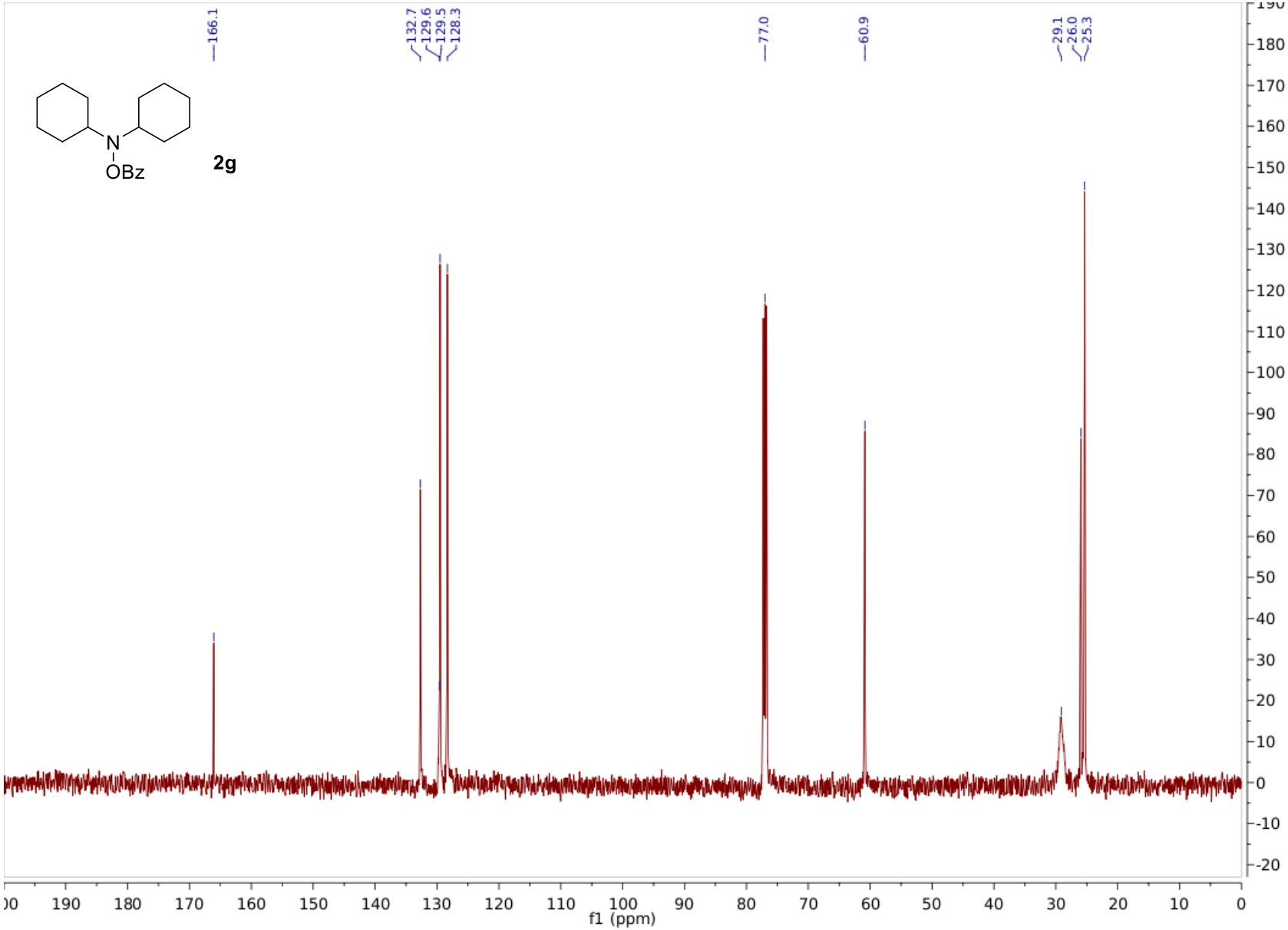
To test this idea, we subjected stilbene substrates to the reaction condition, since they had been shown to be poor substrates for the intermolecular amino oxygenation in the past. We started with isomers of 99% geometric purity for each. When subjected to the reaction conditions with the *O*-benzoylhydroxylamine, the recovered alkene showed only 83% *E* when starting with the *E*-stilbene, and 84% Z when starting with the *Z*-stilbene. To exclude the possibility that the copper catalyst and/or heat was producing the observed isomerization, a control was run with the *O*-benzoylhydroxylamine removed from the reaction, but all other conditions held the same. In this case, 99%+ of each alkene was recovered with retention of the 99% geometric purities.

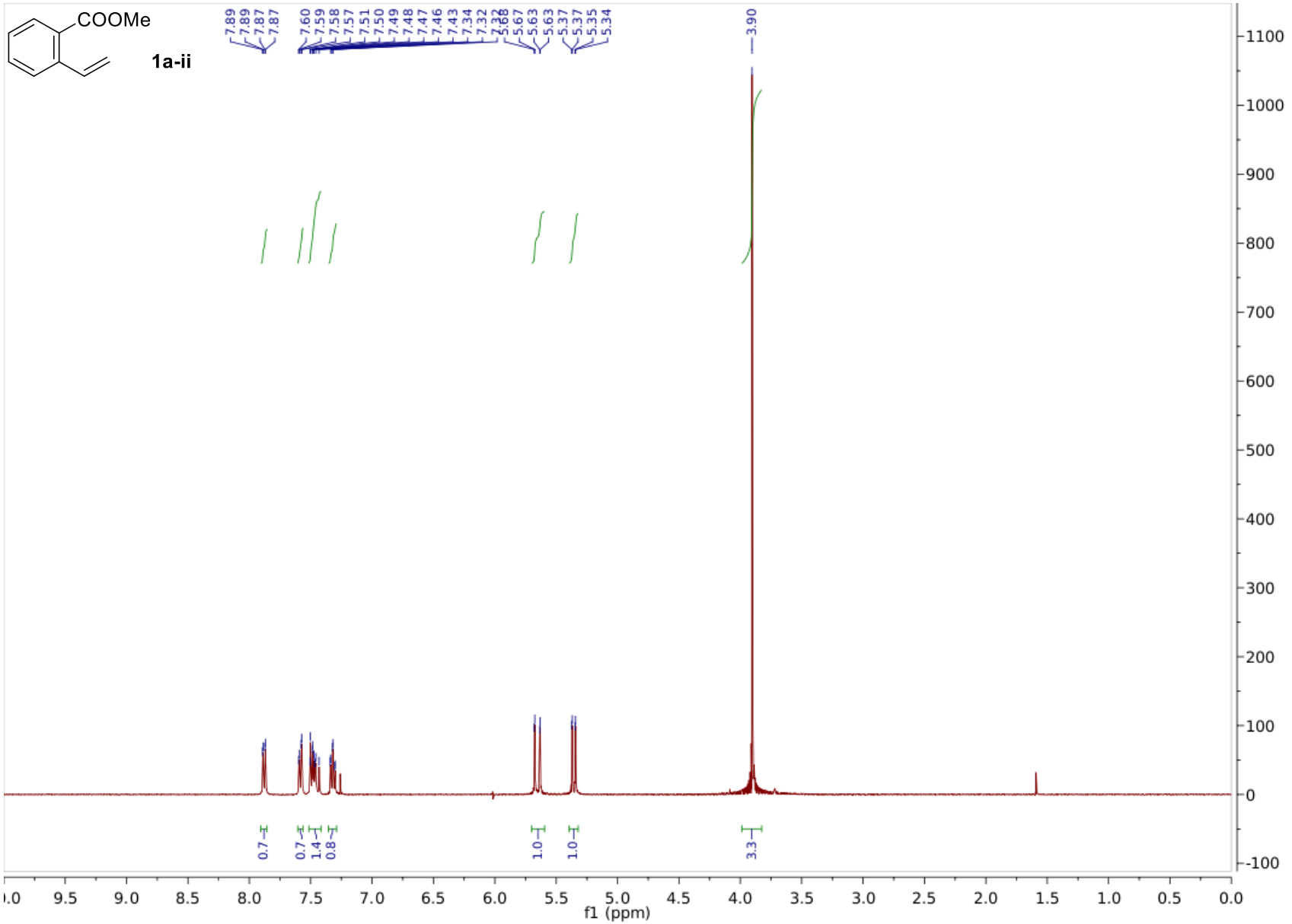
1.5 ^{1}H and ^{13}C Spectra

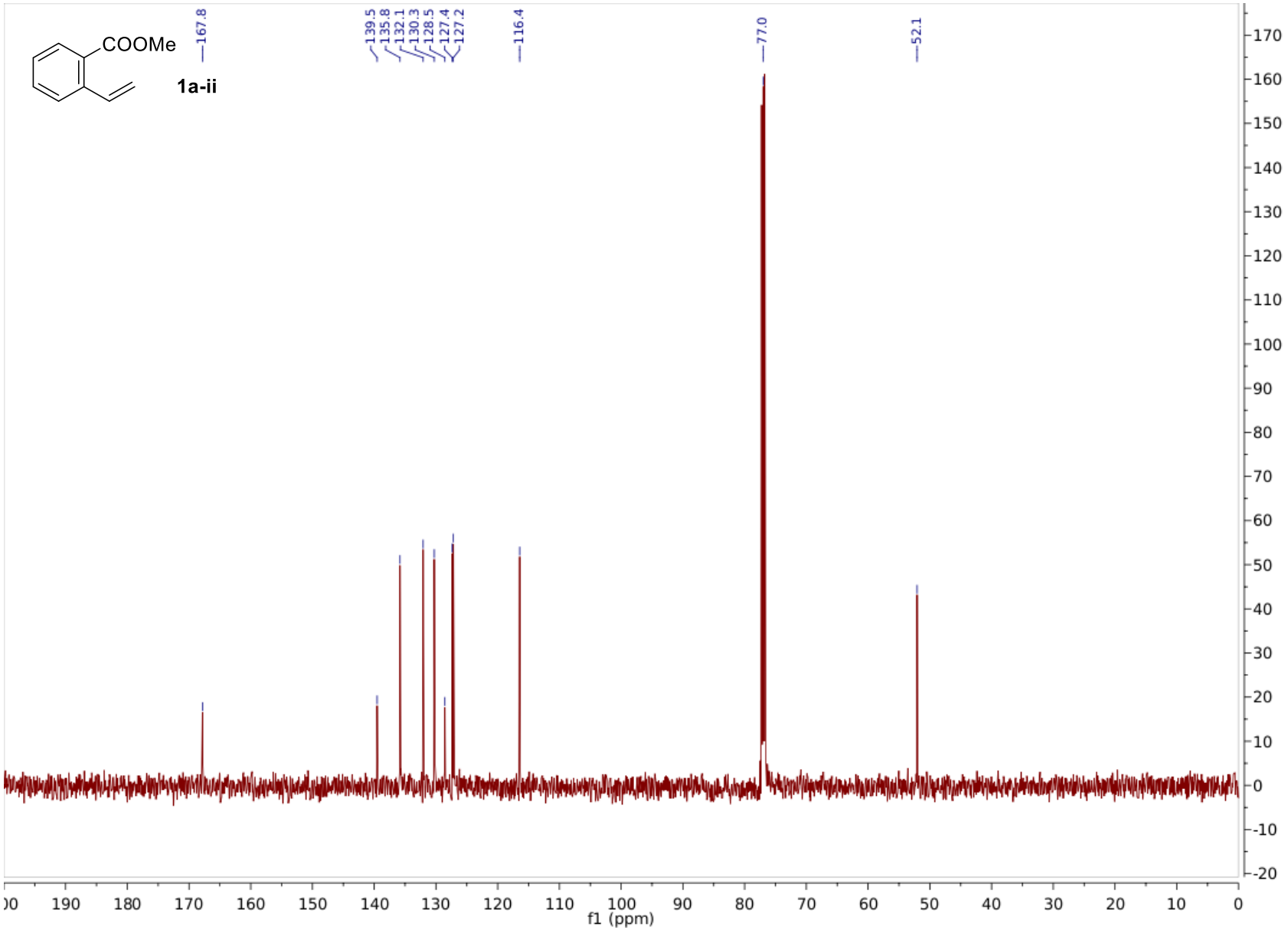


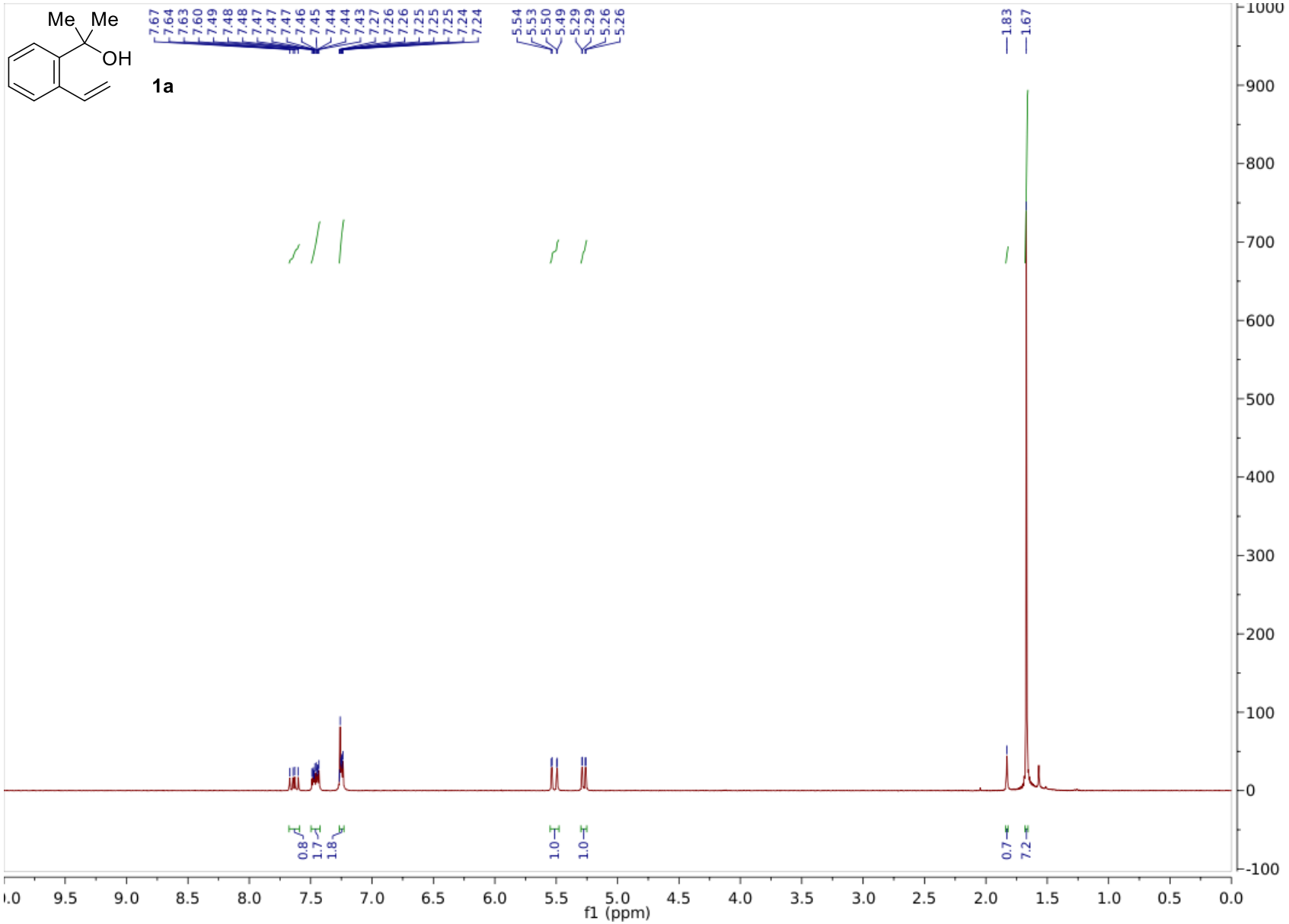

Me
N
OBz
Ph
2f
165.1
139.1
133.0
129.3
129.1
128.6
128.4
128.3
126.1
77.0
62.5
47.0
33.7
f1 (ppm)

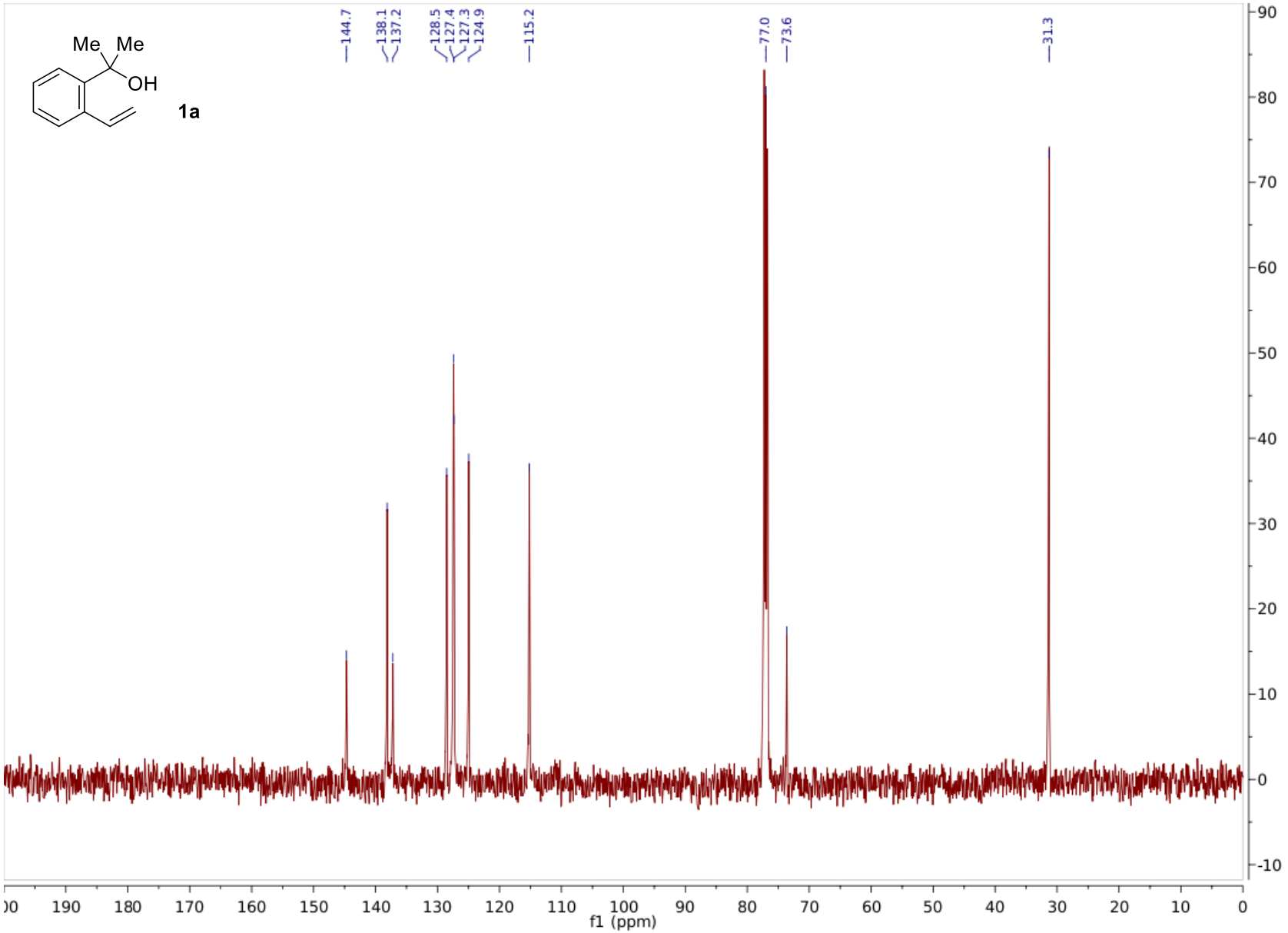

N
OBz
2g
8.05
8.05
8.05
8.04
8.03
8.03
7.59
7.59
7.58
7.57
7.57
7.57
7.55
7.55
7.55
7.47
7.45
7.43
7.26
3.13
3.12
3.11
3.10
3.09
3.08
3.07
3.06
3.06
1.92
1.89
1.81
1.78
1.64
1.61
1.37
1.26
1.26
1.17
1.17
1.16
1.14
1.14
1.13
1.11
1.10
1.09
1.5
0.7
1.6
1.8
4.0
4.1
1.8
8.0
2.2
f1 (ppm)

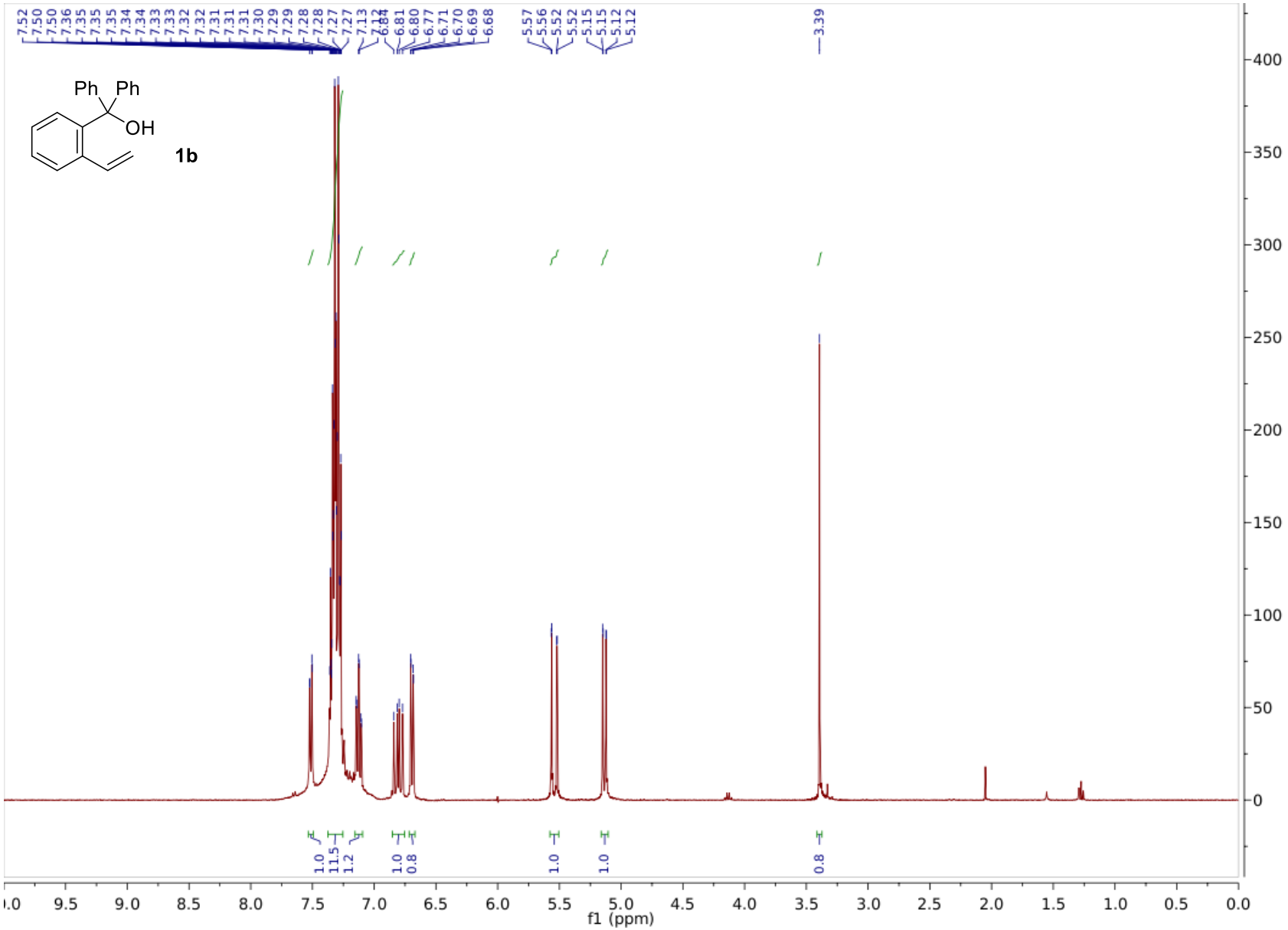

166.1
132.7
129.6
129.5
128.3
77.0
60.9
29.1
26.0
25.3
N
OBz
2g
f1 (ppm)

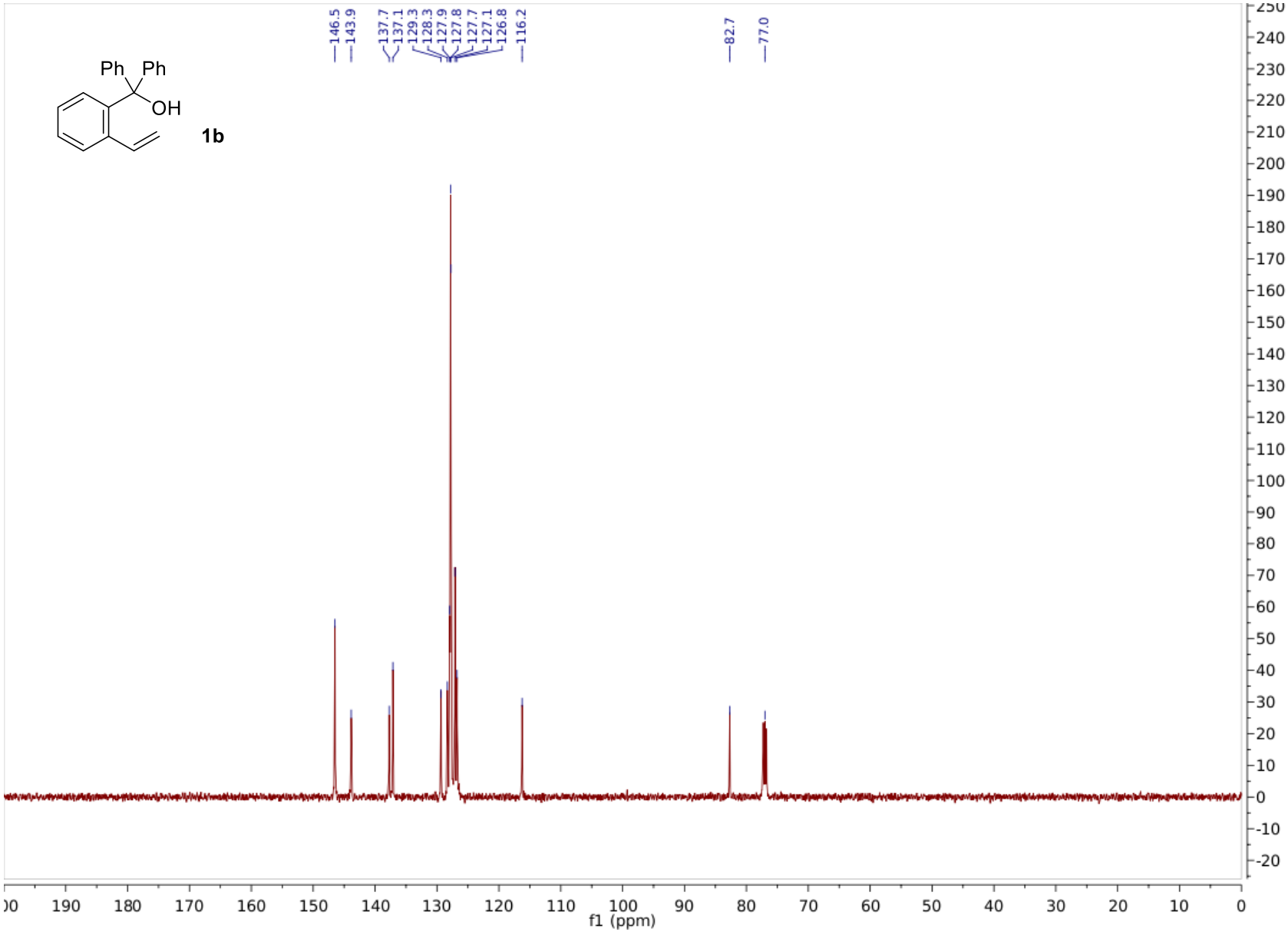

COOMe
1a-ii
7.89
7.89
7.87
7.87
7.60
7.59
7.58
7.57
7.51
7.50
7.49
7.48
7.47
7.46
7.43
7.34
7.32
5.67
5.63
5.63
5.37
5.37
5.35
5.34
3.90
0.7
0.7
1.4
0.8
1.0
1.0
3.3
f1 (ppm)

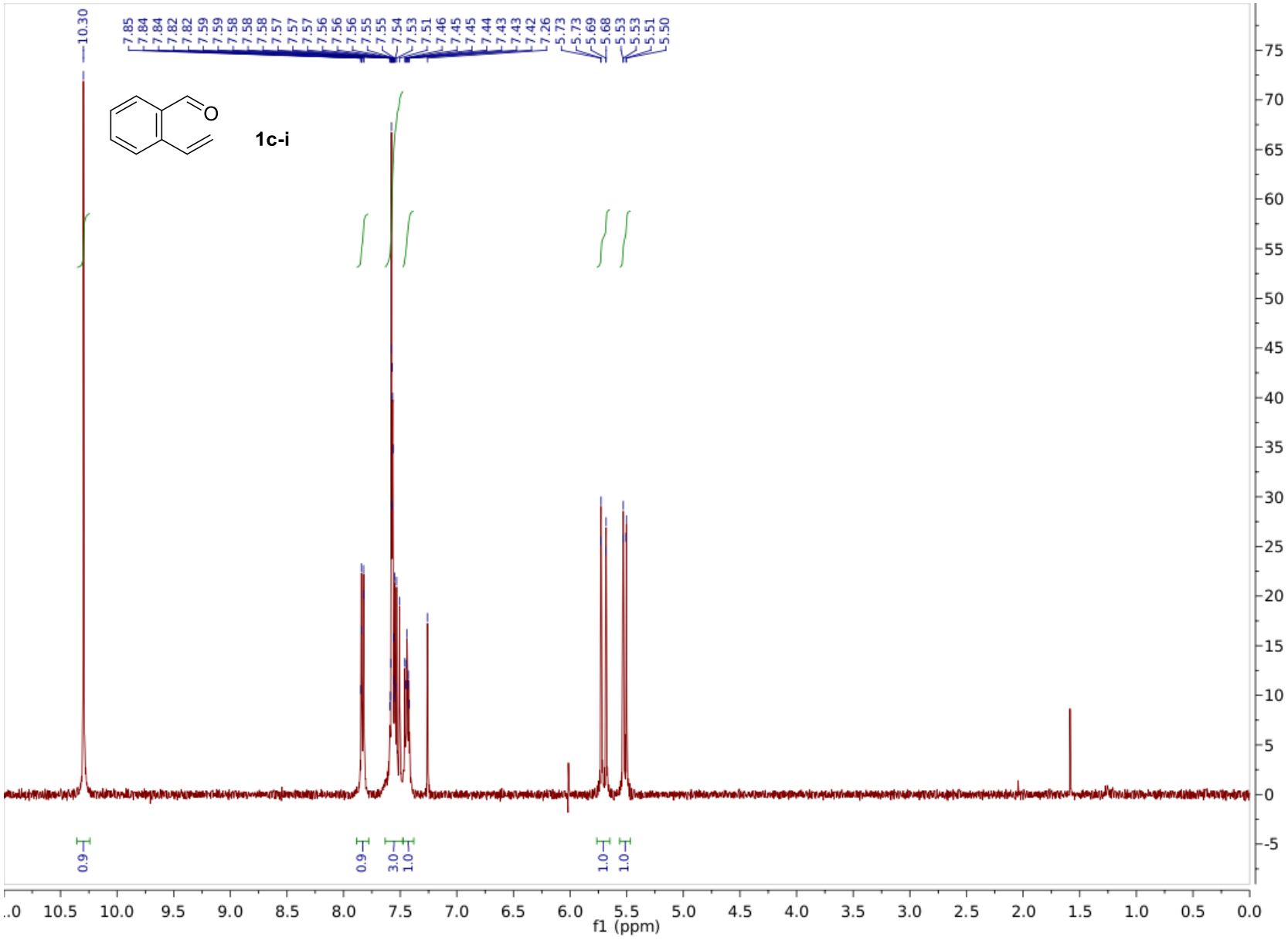

COOMe
1a-ii
167.8
139.5
135.8
132.1
130.3
128.5
127.4
127.2
116.4
77.0
52.1
f1 (ppm)

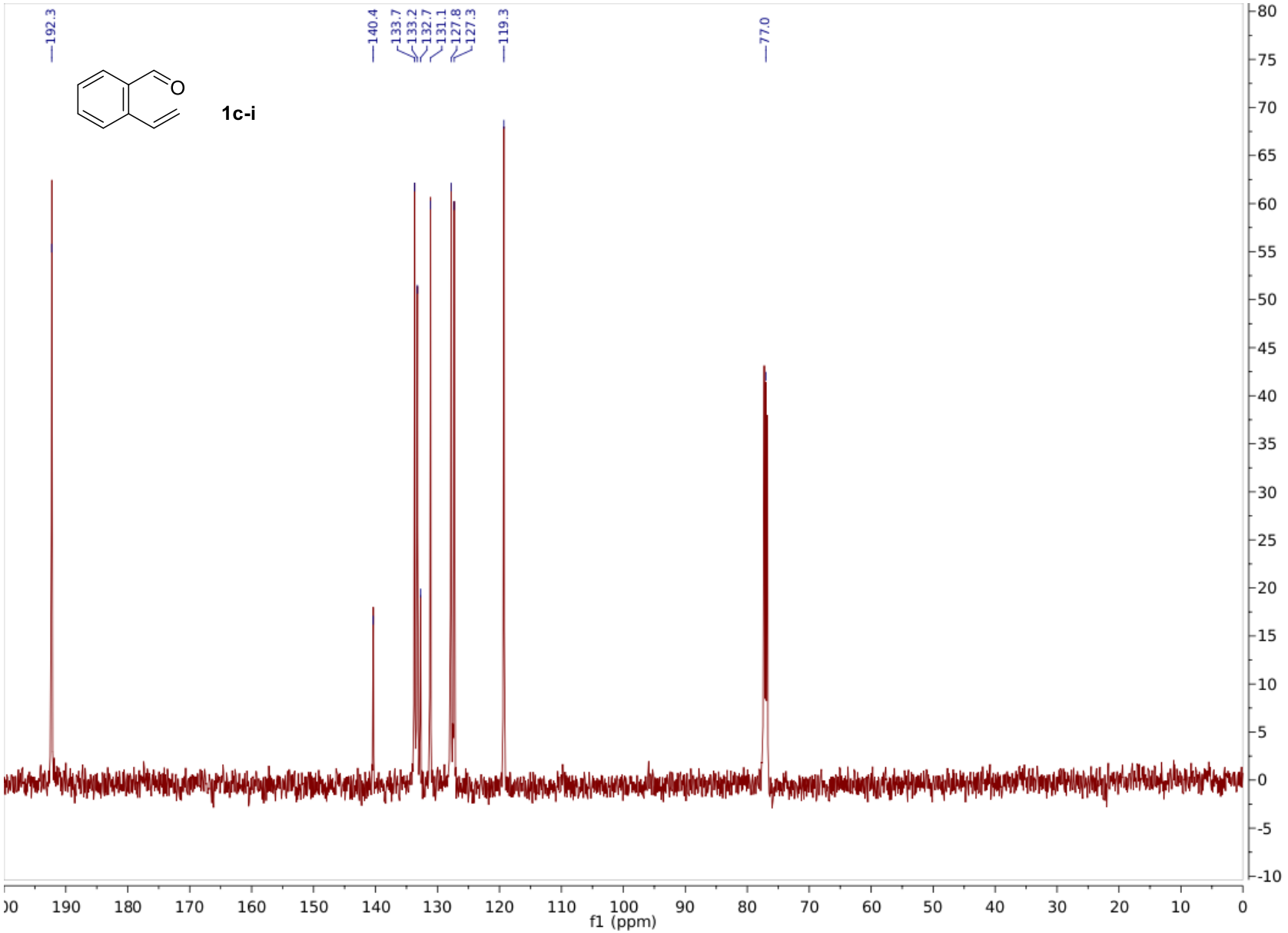

Me
Me
OH
1a
7.67
7.64
7.63
7.60
7.49
7.48
7.48
7.47
7.47
7.47
7.46
7.45
7.44
7.44
7.43
7.27
7.26
7.26
7.25
7.25
7.25
7.24
7.24
5.54
5.53
5.50
5.49
5.29
5.29
5.26
5.26
1.83
1.67
0.8
1.7
1.8
1.0
1.0
0.7
7.2
f1 (ppm)

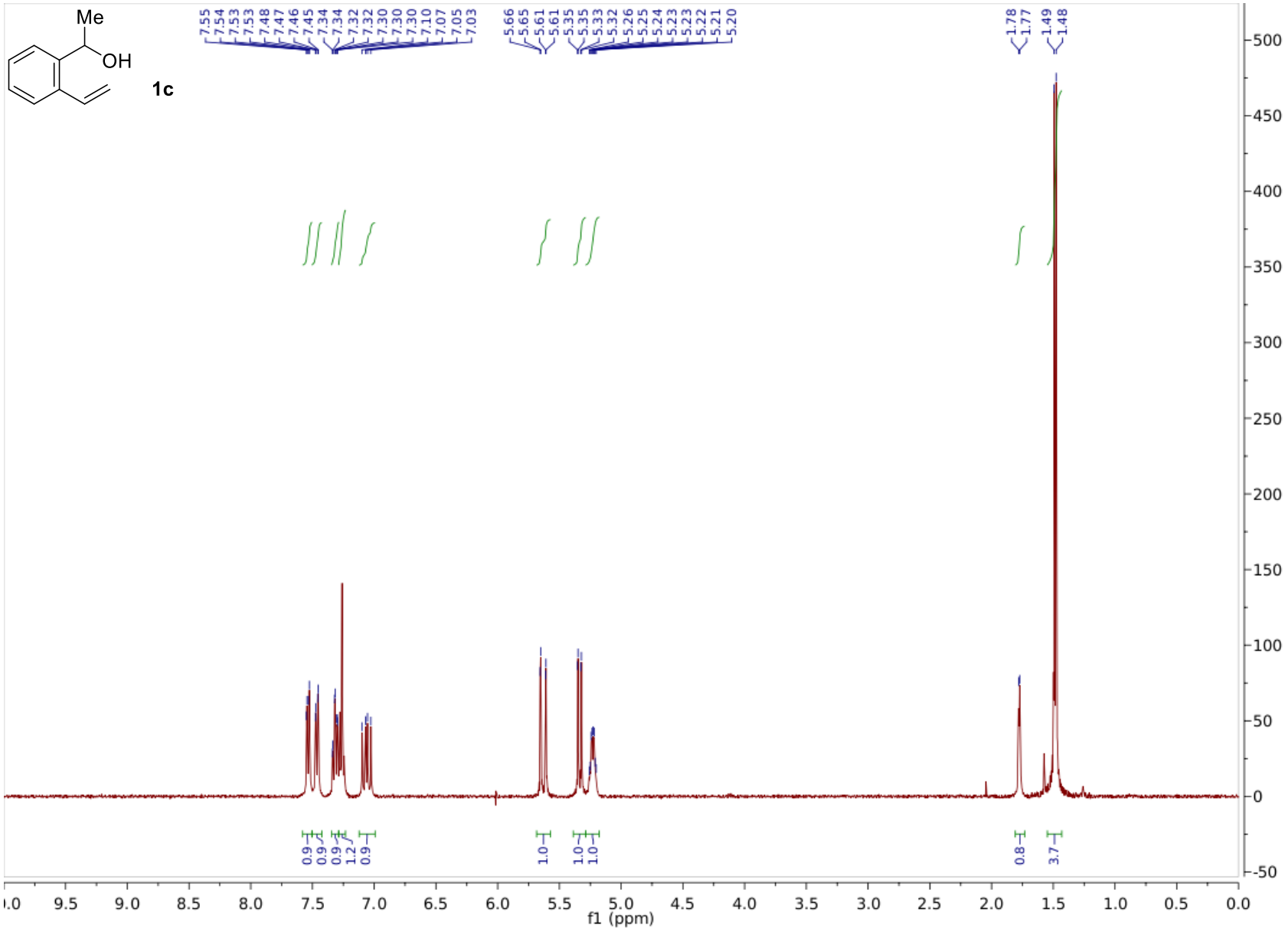

Me
Me
OH
1a
144.7
138.1
137.2
128.5
127.4
127.3
124.9
115.2
77.0
73.6
31.3
f1 (ppm)

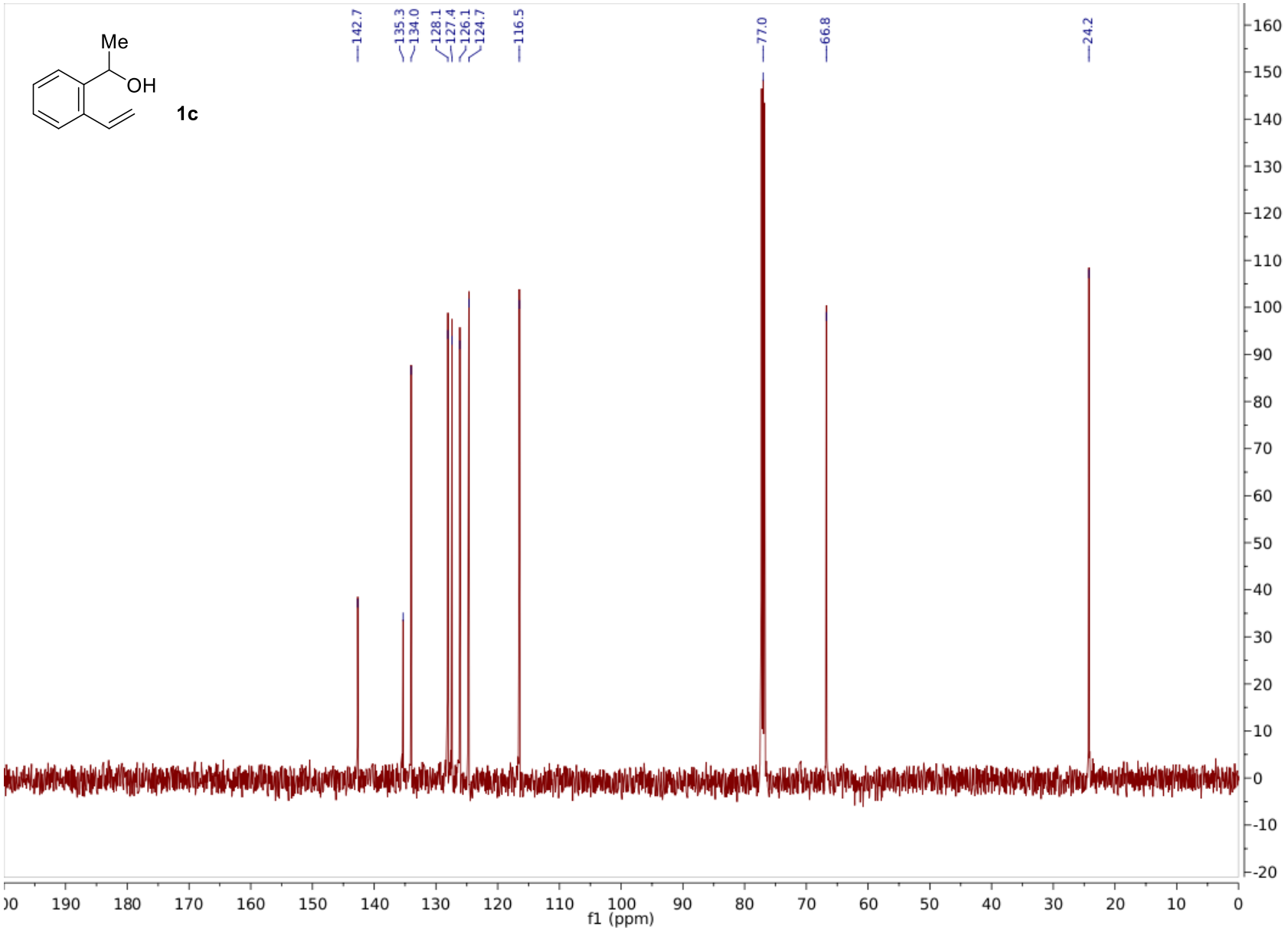

Ph
Ph
OH
1b
7.52
7.50
7.50
7.36
7.35
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.13
6.84
6.81
6.80
6.77
6.71
6.70
6.69
6.68
5.57
5.56
5.52
5.52
5.15
5.15
5.12
5.12
3.39
1.0
11.5
1.2
1.0
0.8
1.0
1.0
0.8
f1 (ppm)

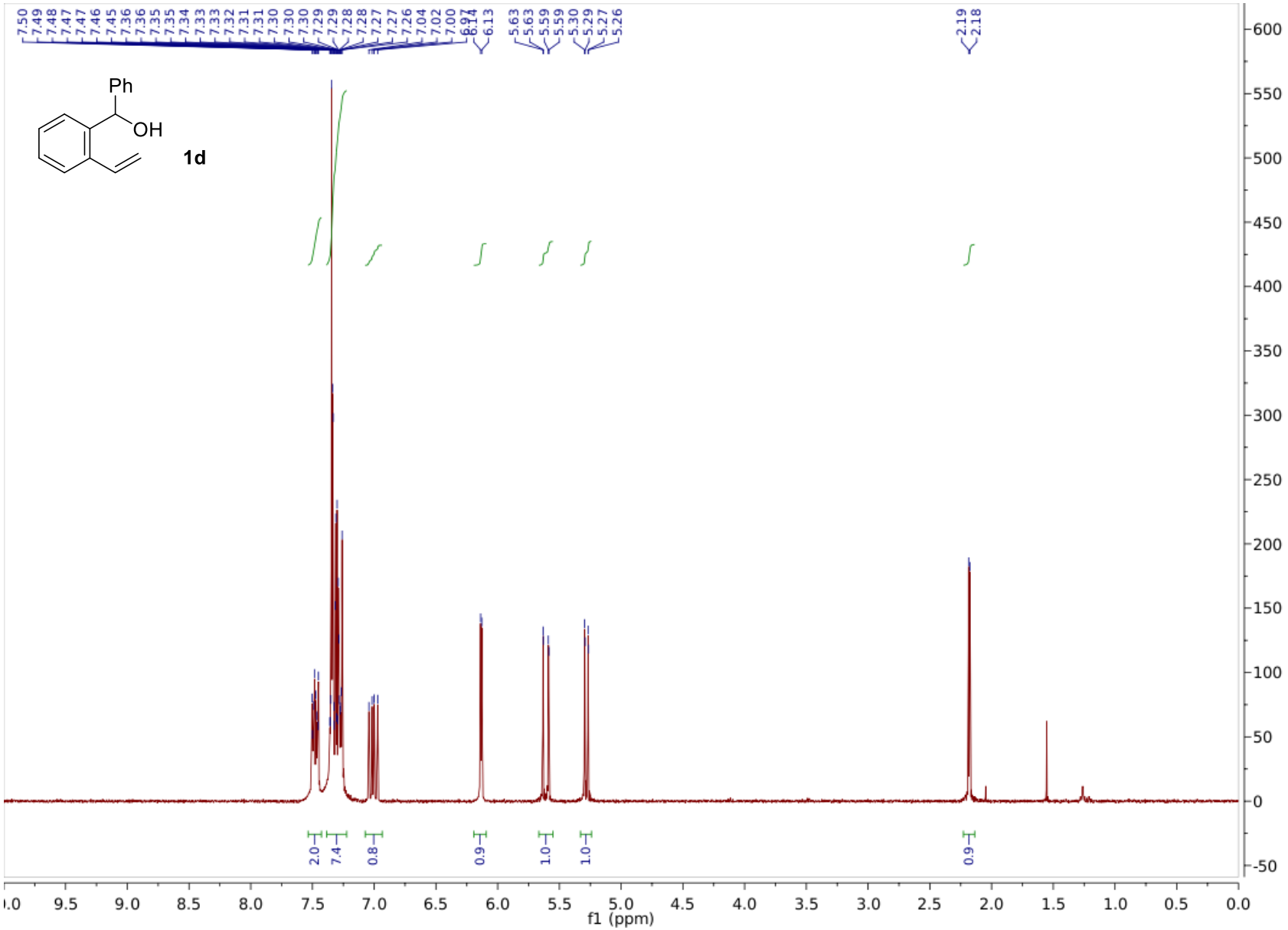

Ph
Ph
OH
1b
146.5
143.9
137.7
137.1
129.3
128.3
127.9
127.8
127.7
127.1
126.8
116.2
82.7
77.0
f1 (ppm)

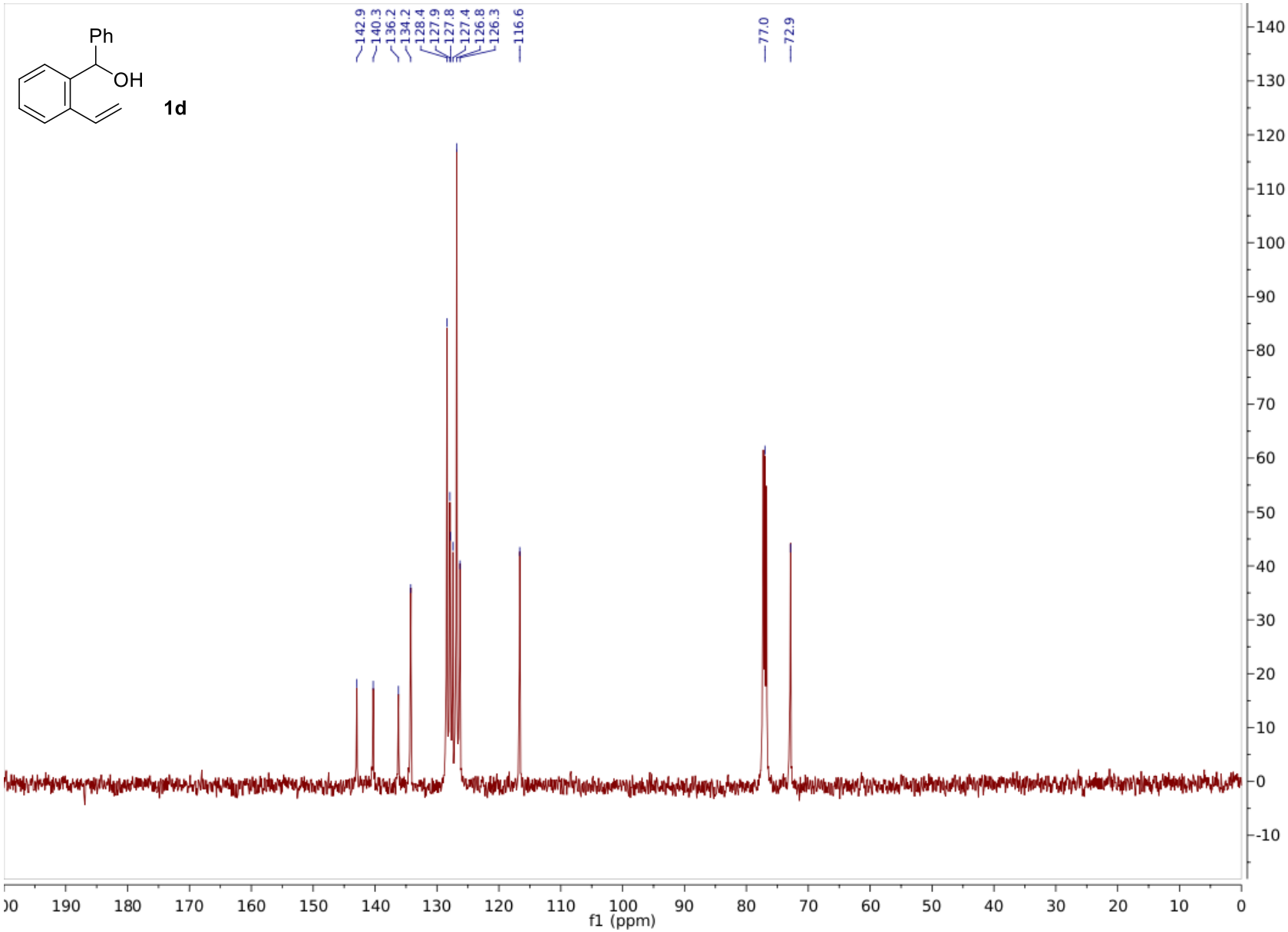

10.30
7.85
7.84
7.84
7.82
7.82
7.59
7.59
7.58
7.58
7.58
7.57
7.57
7.57
7.56
7.56
7.56
7.55
7.55
7.54
7.53
7.51
7.46
7.45
7.45
7.44
7.43
7.43
7.42
7.26
5.73
5.69
5.68
5.53
5.53
5.51
5.50
O
1c-i
0.9
0.9
3.0
1.0
1.0
1.0
f1 (ppm)

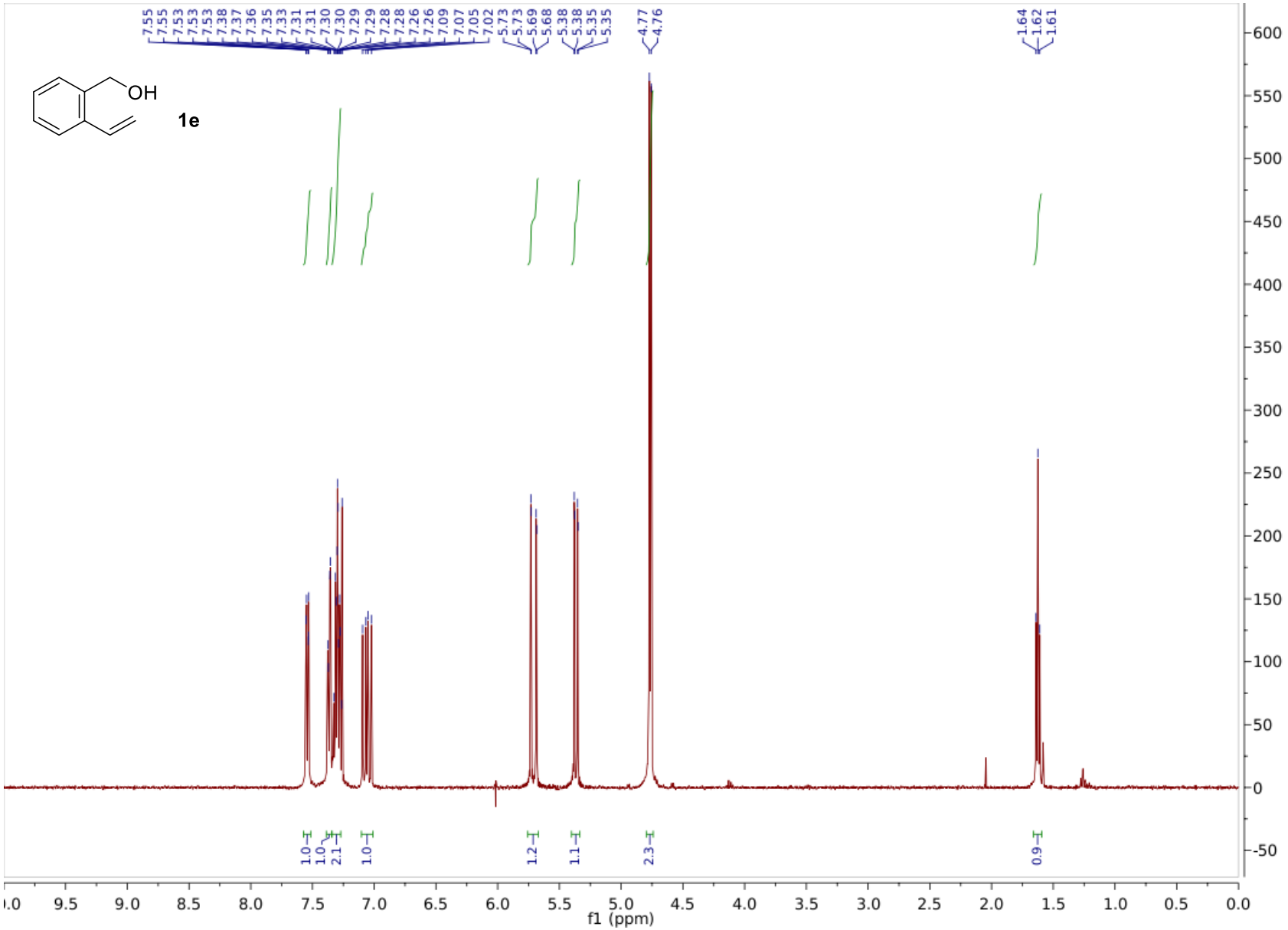

192.3
140.4
133.7
133.2
132.7
131.1
127.8
127.3
119.3
77.0
O
1c-i
f1 (ppm)

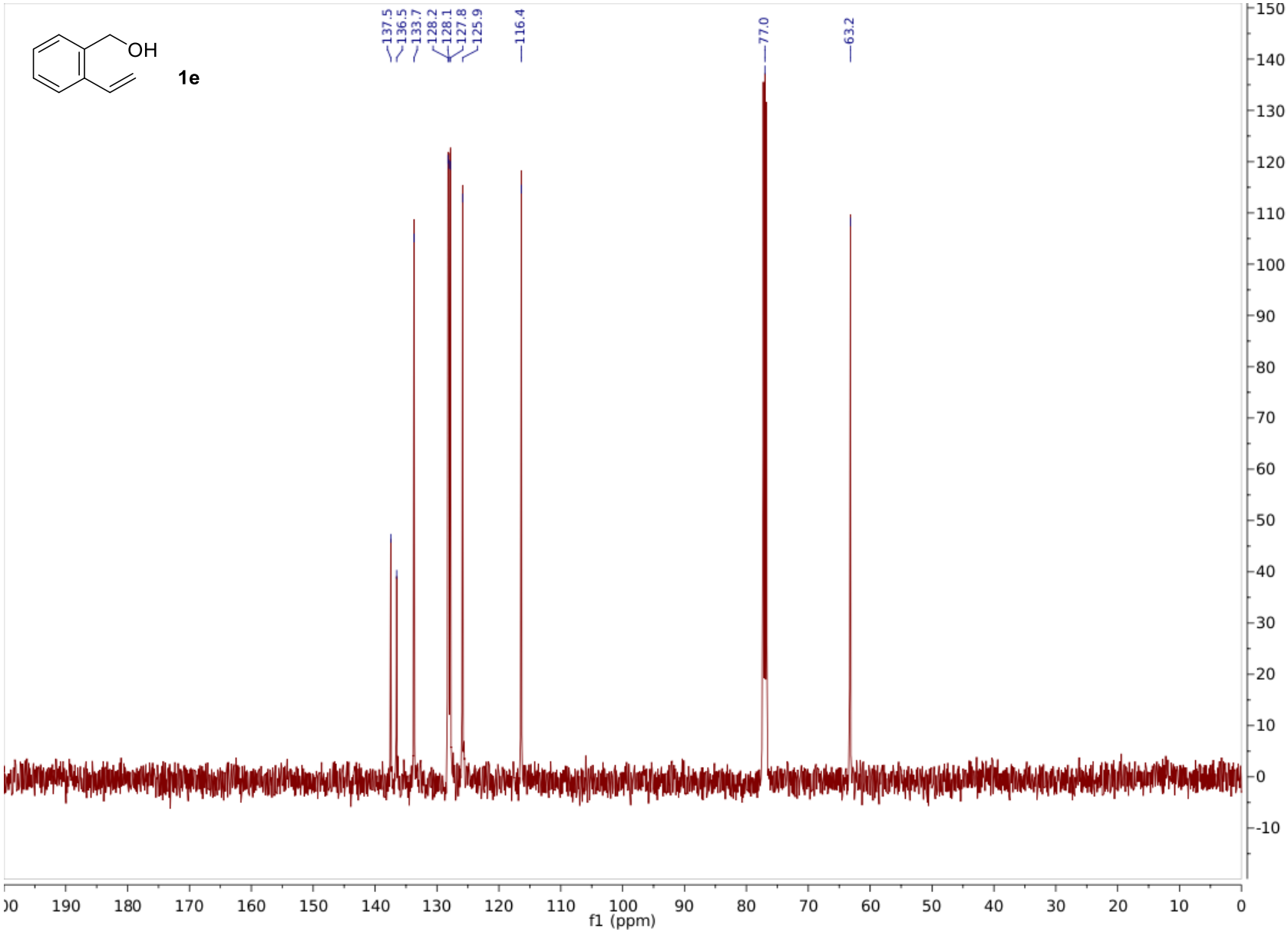

Me
OH
1c
7.55
7.54
7.53
7.53
7.48
7.47
7.46
7.45
7.34
7.34
7.32
7.32
7.30
7.30
7.30
7.10
7.07
7.05
7.03
5.66
5.65
5.61
5.61
5.35
5.35
5.33
5.32
5.26
5.25
5.24
5.23
5.23
5.22
5.21
5.20
1.78
1.77
1.49
1.48
0.9
0.9
0.9
1.2
0.9
1.0
1.0
1.0
0.8
3.7
f1 (ppm)

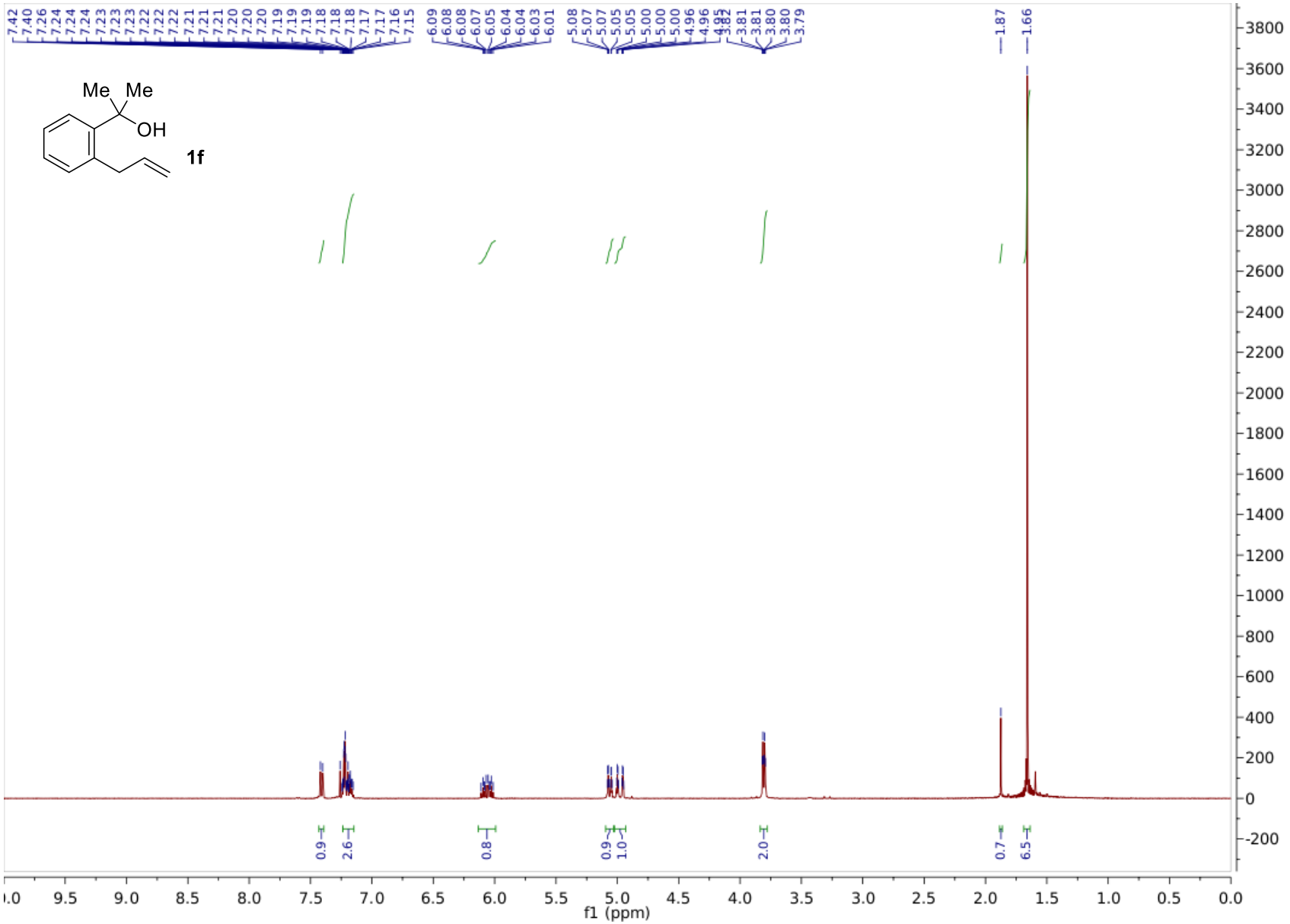

Me
OH
1c
142.7
135.3
134.0
128.1
127.4
126.1
124.7
116.5
77.0
66.8
24.2
f1 (ppm)

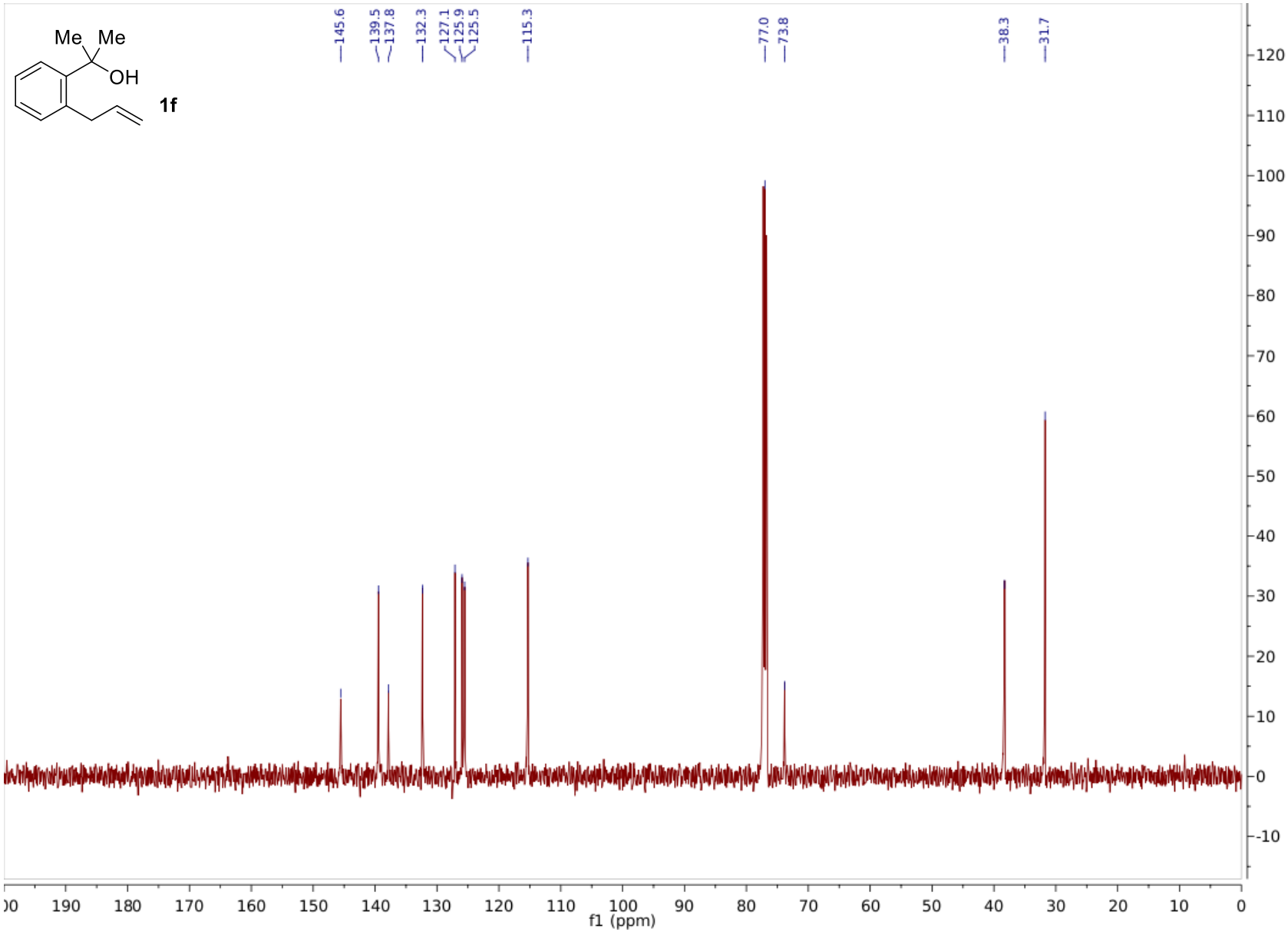

Ph
OH
1d
f1 (ppm)

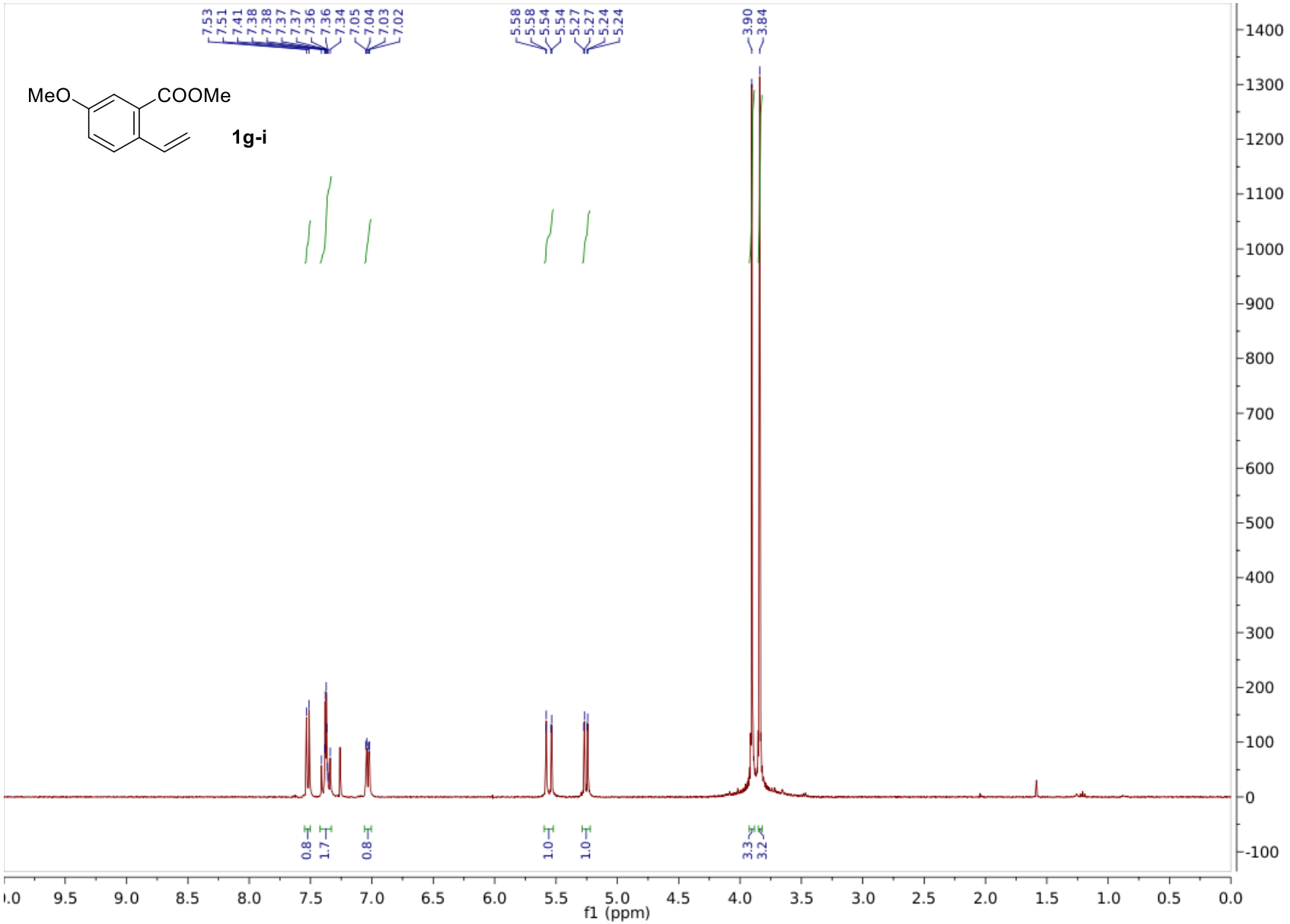

Ph
OH
1d
142.9
140.3
136.2
134.2
128.4
127.9
127.8
127.4
126.8
126.3
116.6
77.0
72.9
f1 (ppm)

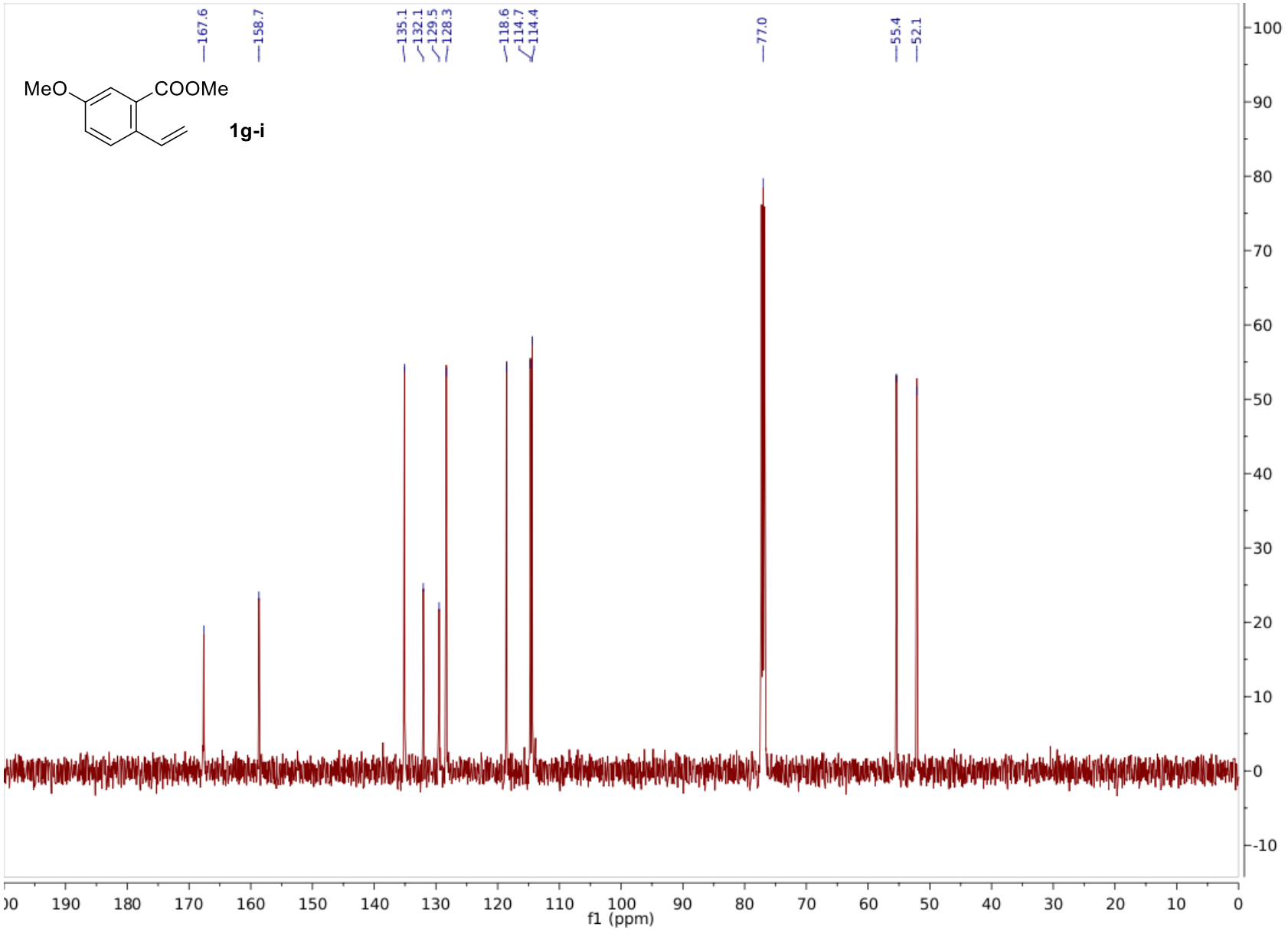

OH
1e
7.55
7.55
7.53
7.53
7.53
7.38
7.37
7.36
7.35
7.33
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.26
7.26
7.09
7.07
7.05
7.02
5.73
5.73
5.69
5.68
5.38
5.38
5.35
5.35
4.77
4.76
1.64
1.62
1.61
1.0
1.0
2.1
1.0
1.2
1.1
2.3
0.9
f1 (ppm)

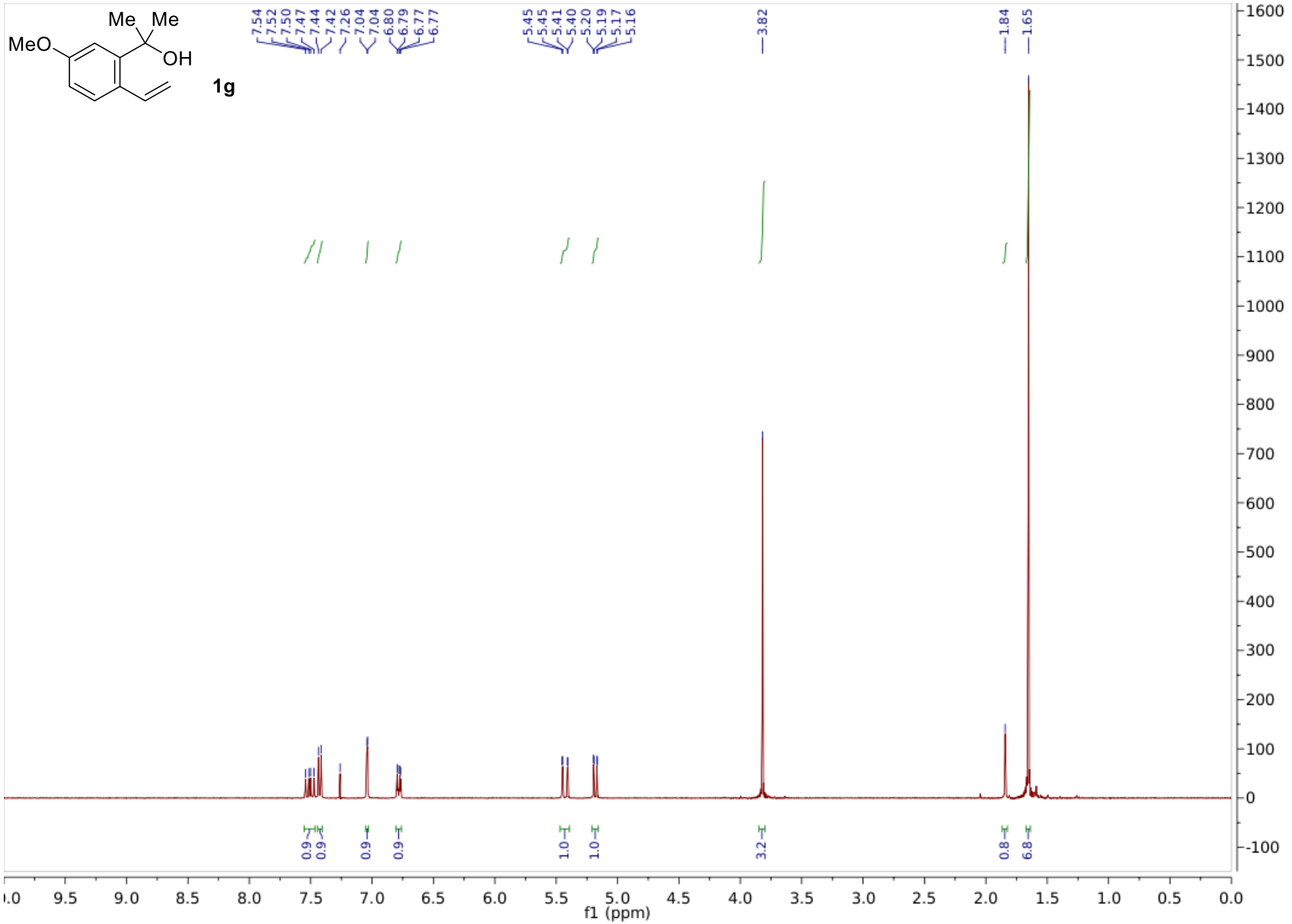

OH
1e
137.5
136.5
133.7
128.2
128.1
127.8
125.9
116.4
77.0
63.2
f1 (ppm)

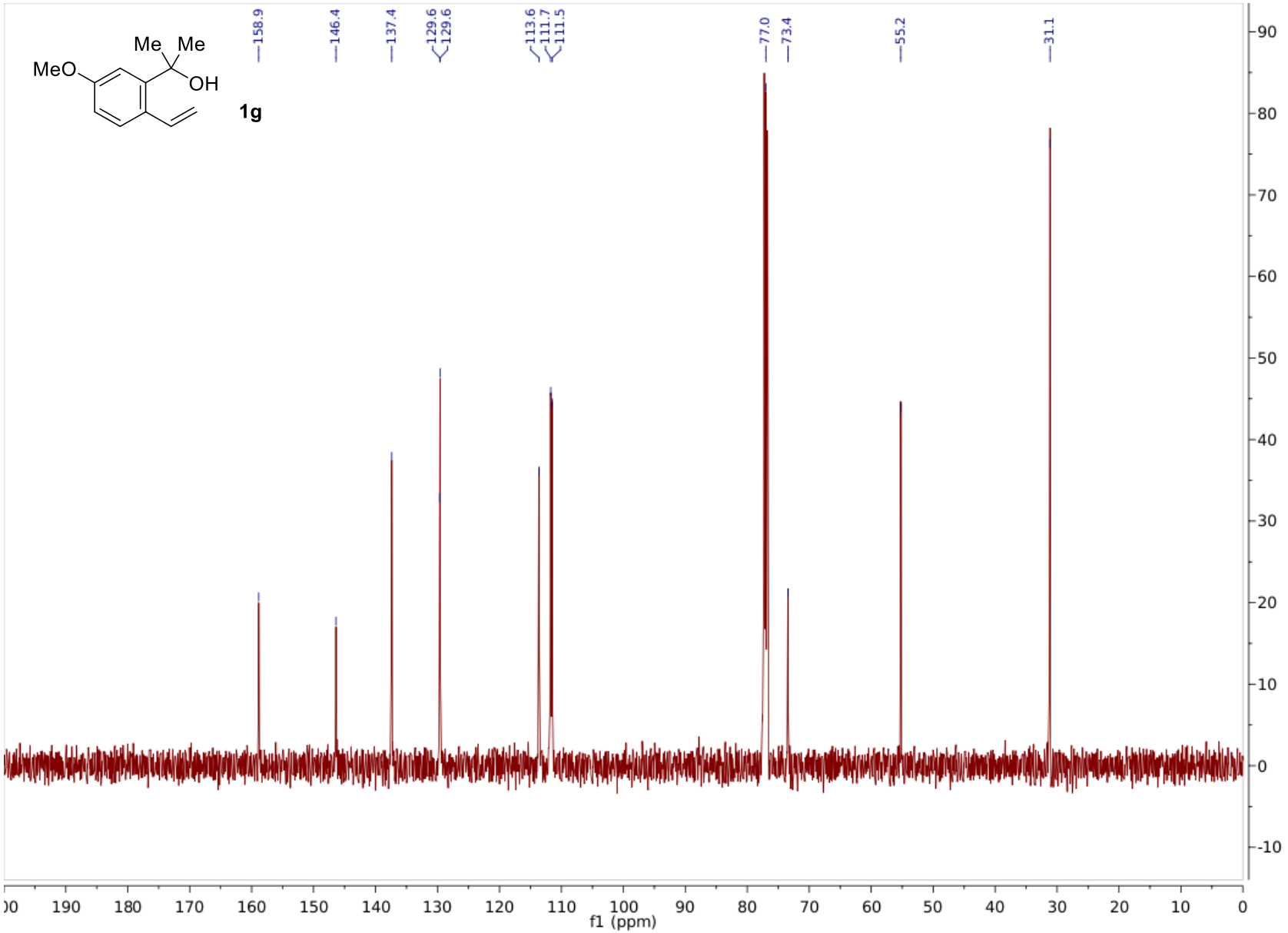

Me Me
OH
1f
f1 (ppm)

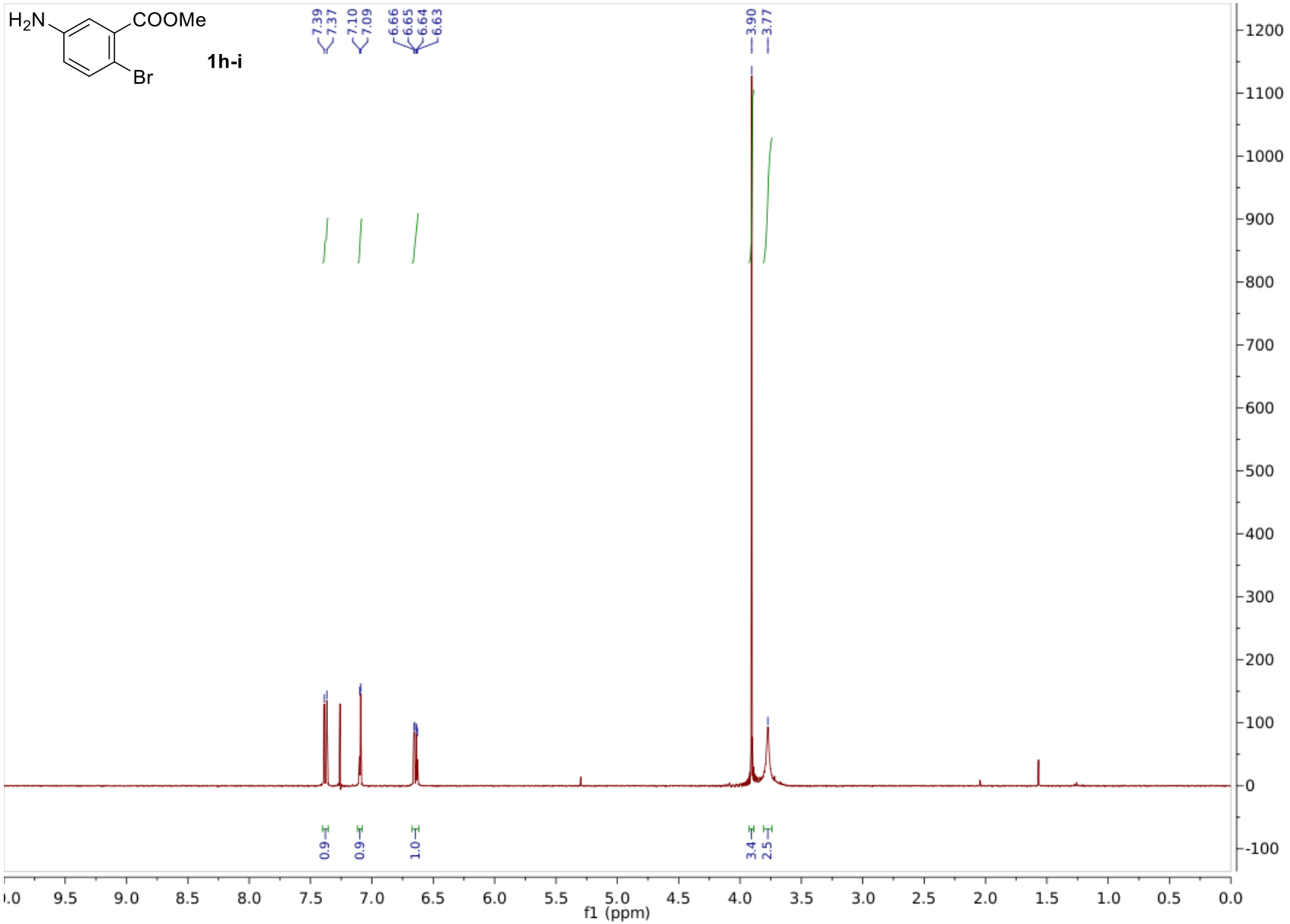

Me
Me
OH
1f
145.6
139.5
137.8
132.3
127.1
125.9
125.5
115.3
77.0
73.8
38.3
31.7
f1 (ppm)

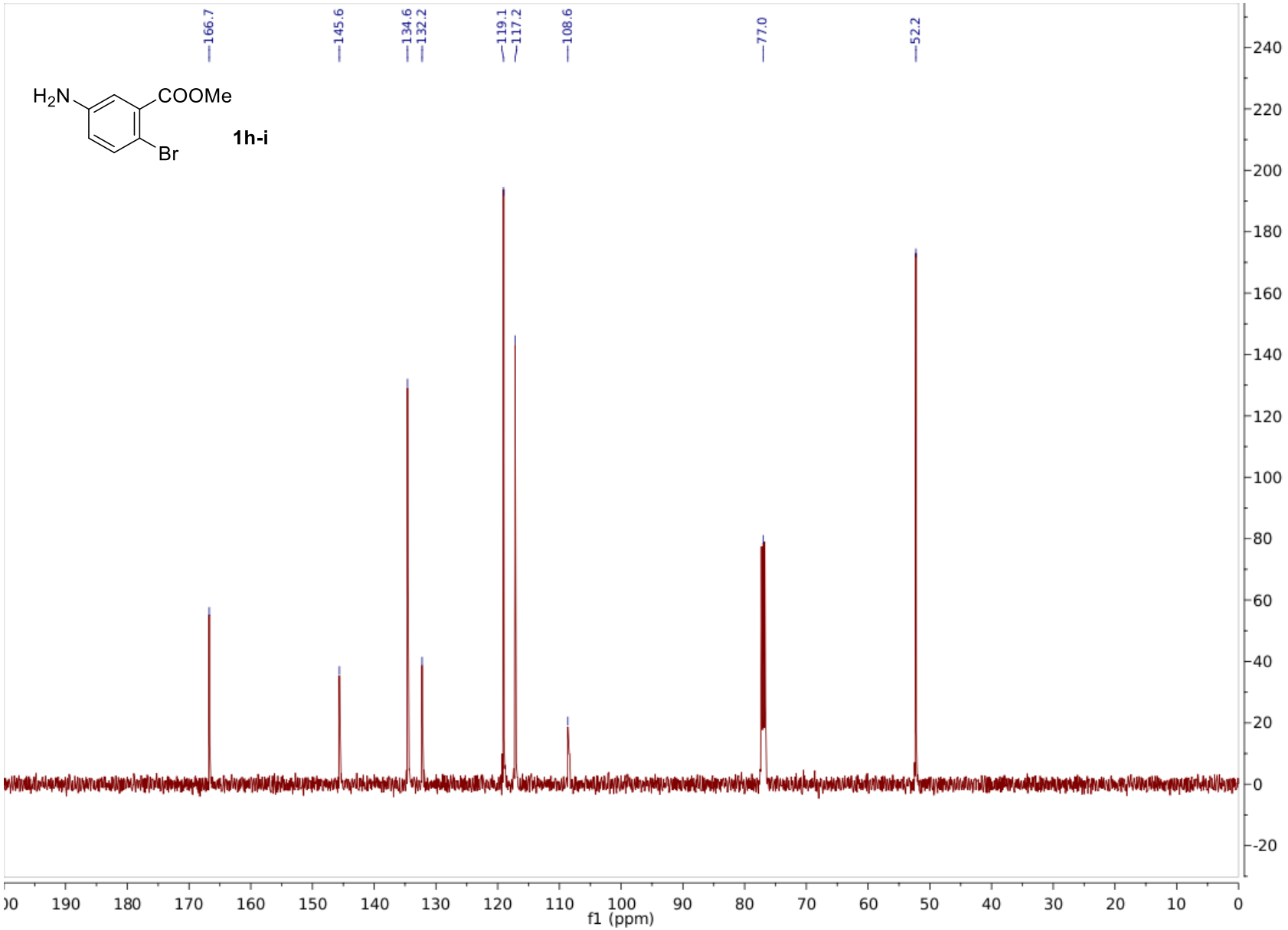

MeO
COOMe
1g-i
7.53
7.51
7.41
7.38
7.38
7.37
7.37
7.36
7.36
7.34
7.05
7.04
7.03
7.02
5.58
5.58
5.54
5.54
5.27
5.27
5.24
5.24
3.90
3.84
0.8
1.7
0.8
1.0
1.0
3.3
3.2
f1 (ppm)

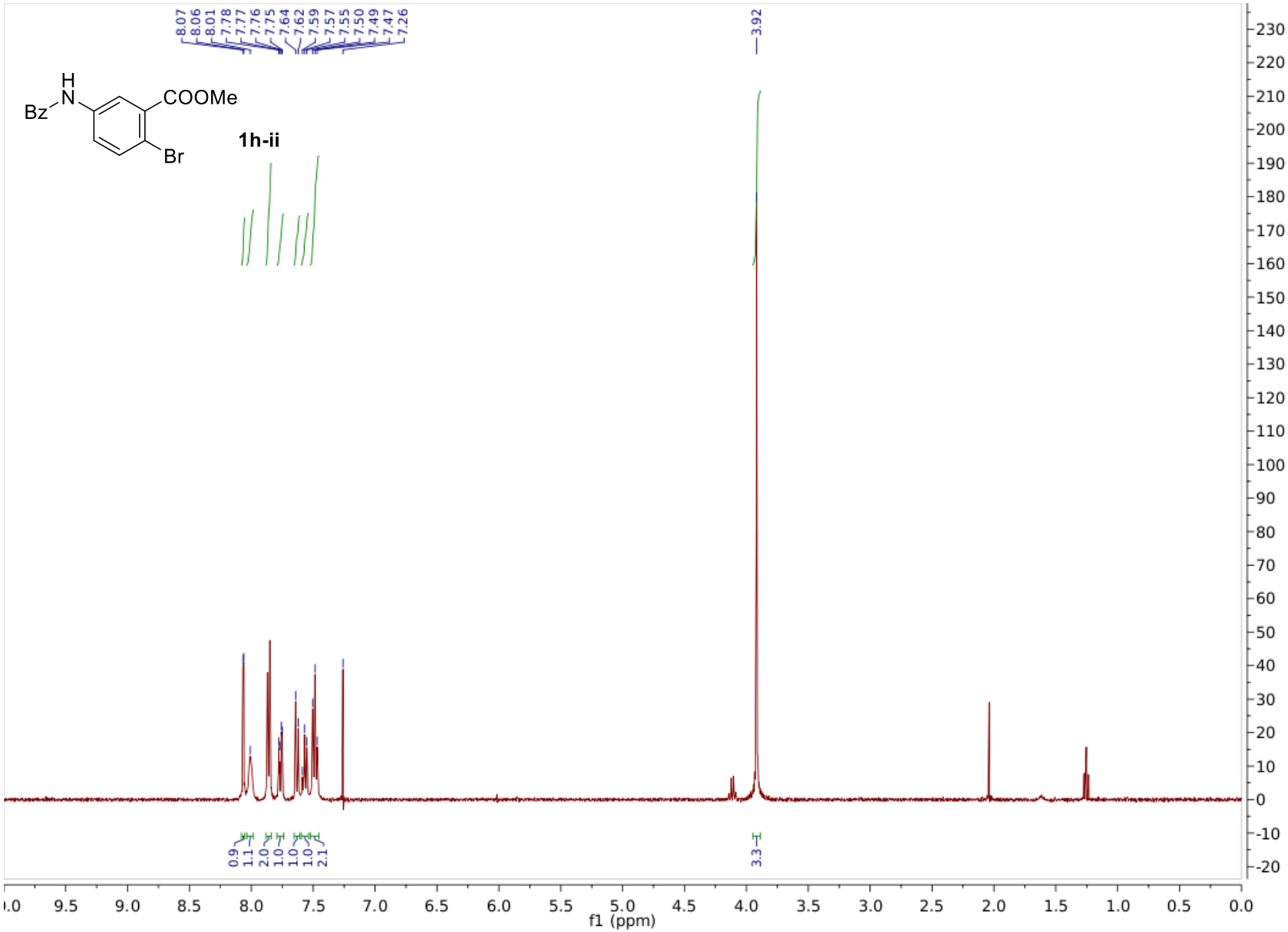

167.6
158.7
135.1
132.1
129.5
128.3
118.6
114.7
114.4
77.0
55.4
52.1
MeO
COOMe
1g-i
f1 (ppm)

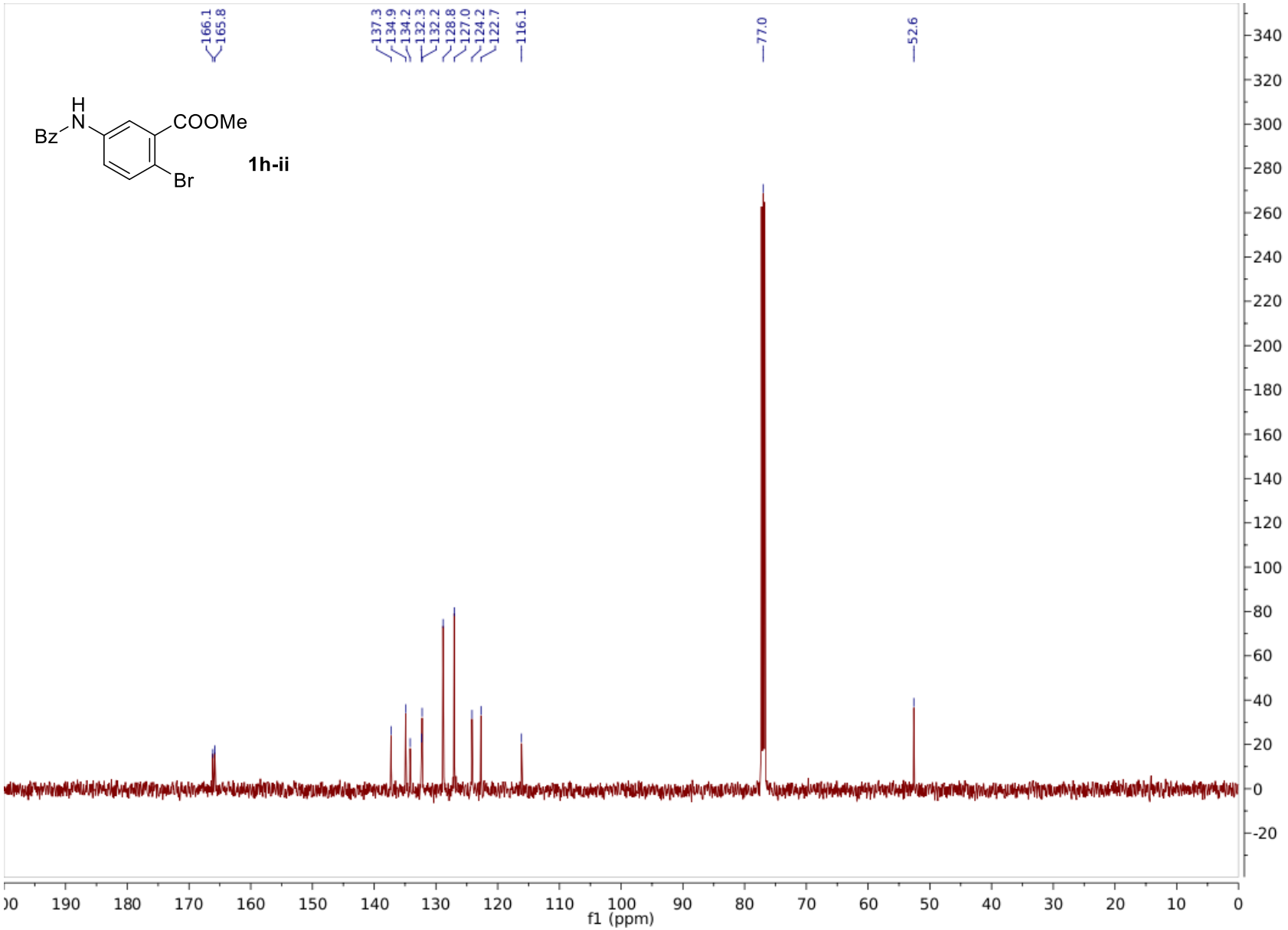

Me
Me
MeO
OH
1g
7.54
7.52
7.50
7.47
7.44
7.42
7.26
7.04
7.04
6.80
6.79
6.77
6.77
5.45
5.45
5.41
5.40
5.20
5.19
5.17
5.16
3.82
1.84
1.65
0.9
0.9
0.9
0.9
1.0
1.0
3.2
0.8
6.8
f1 (ppm)

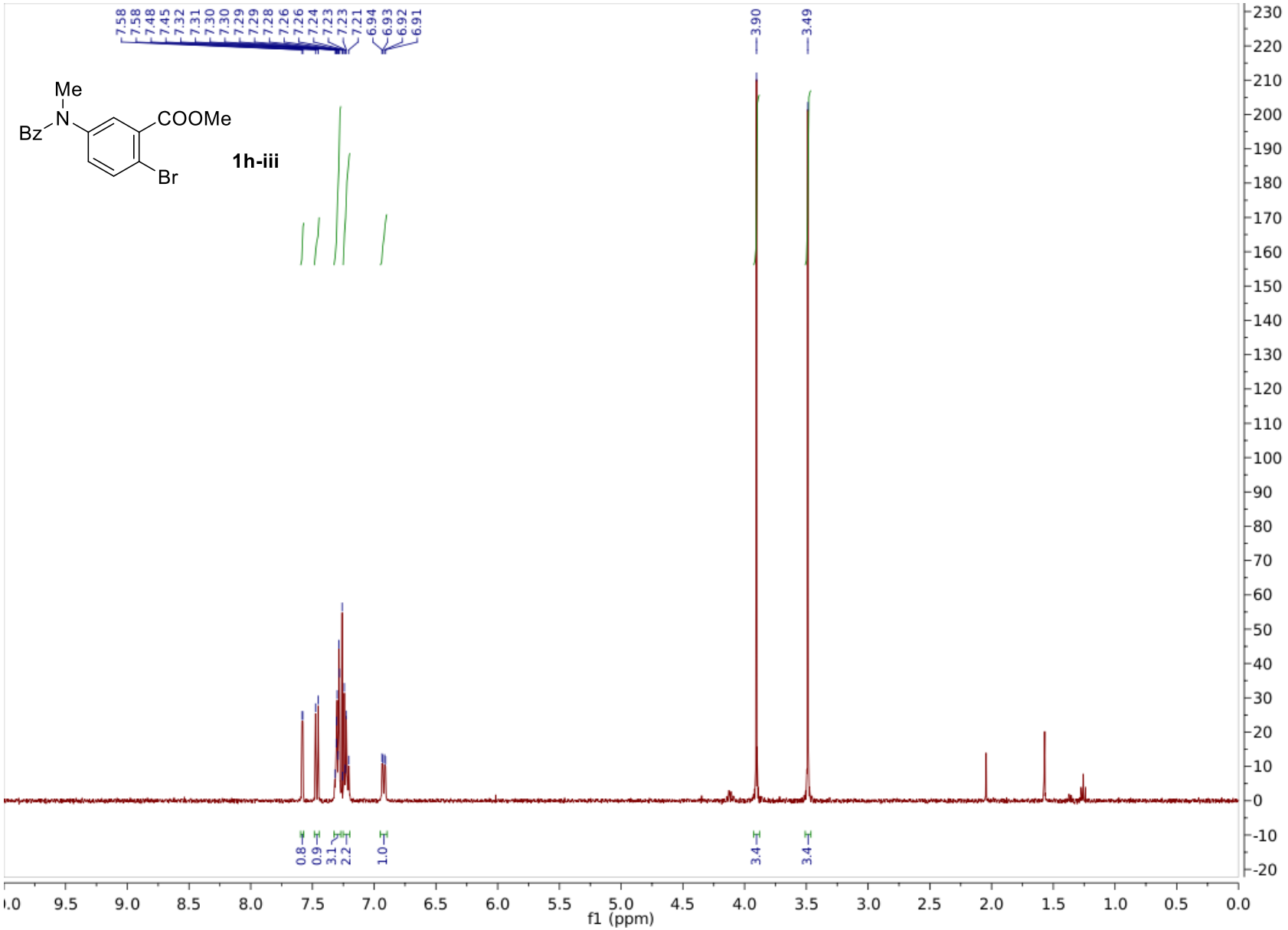

Me
Me
MeO
OH
1g
158.9
146.4
137.4
129.6
129.6
113.6
111.7
111.5
77.0
73.4
55.2
31.1
f1 (ppm)

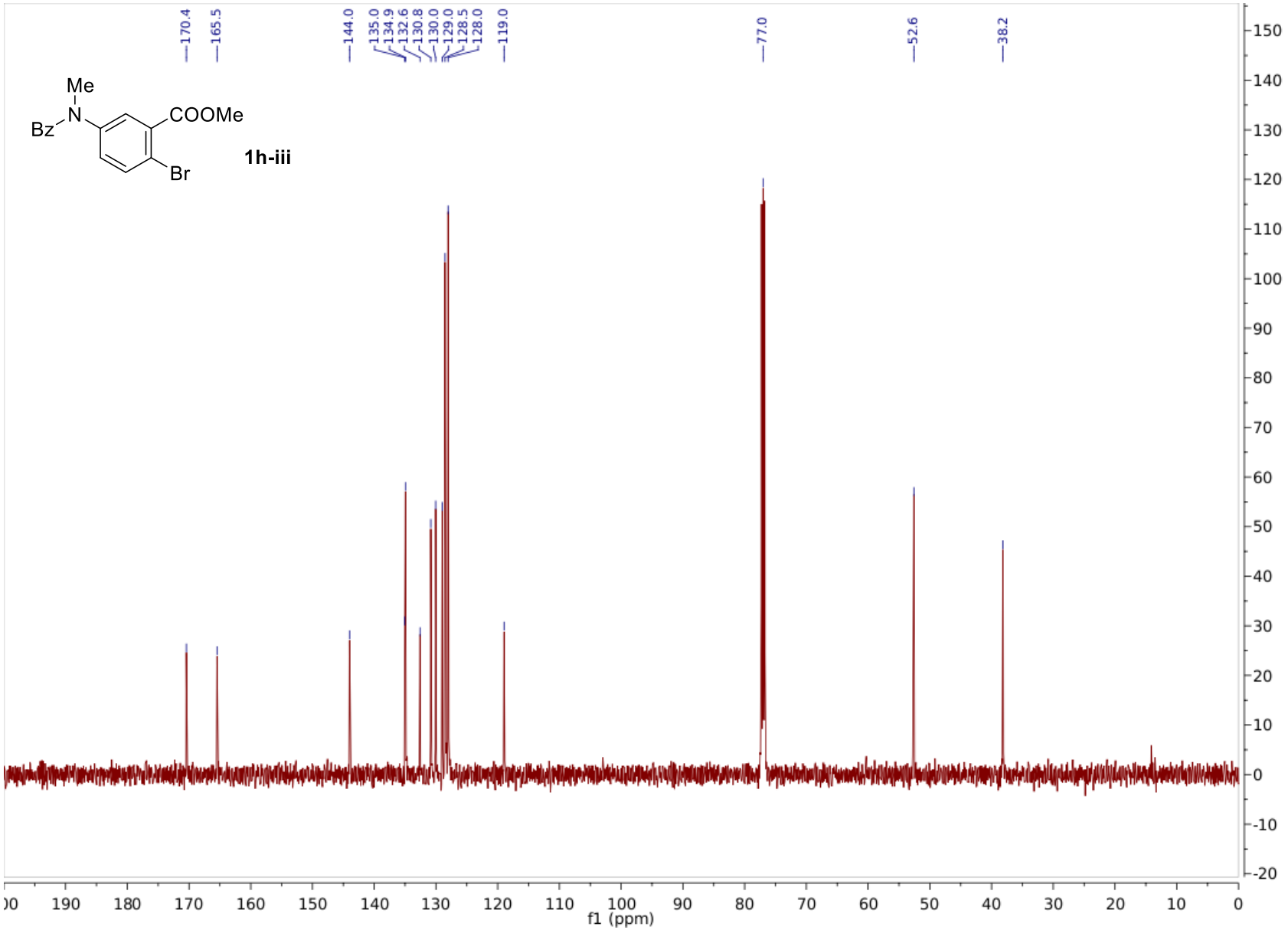

H2N
COOMe
Br
1h-i
7.39
7.37
7.10
7.09
6.66
6.65
6.64
6.63
3.90
3.77
0.9
0.9
1.0
3.4
2.5
f1 (ppm)

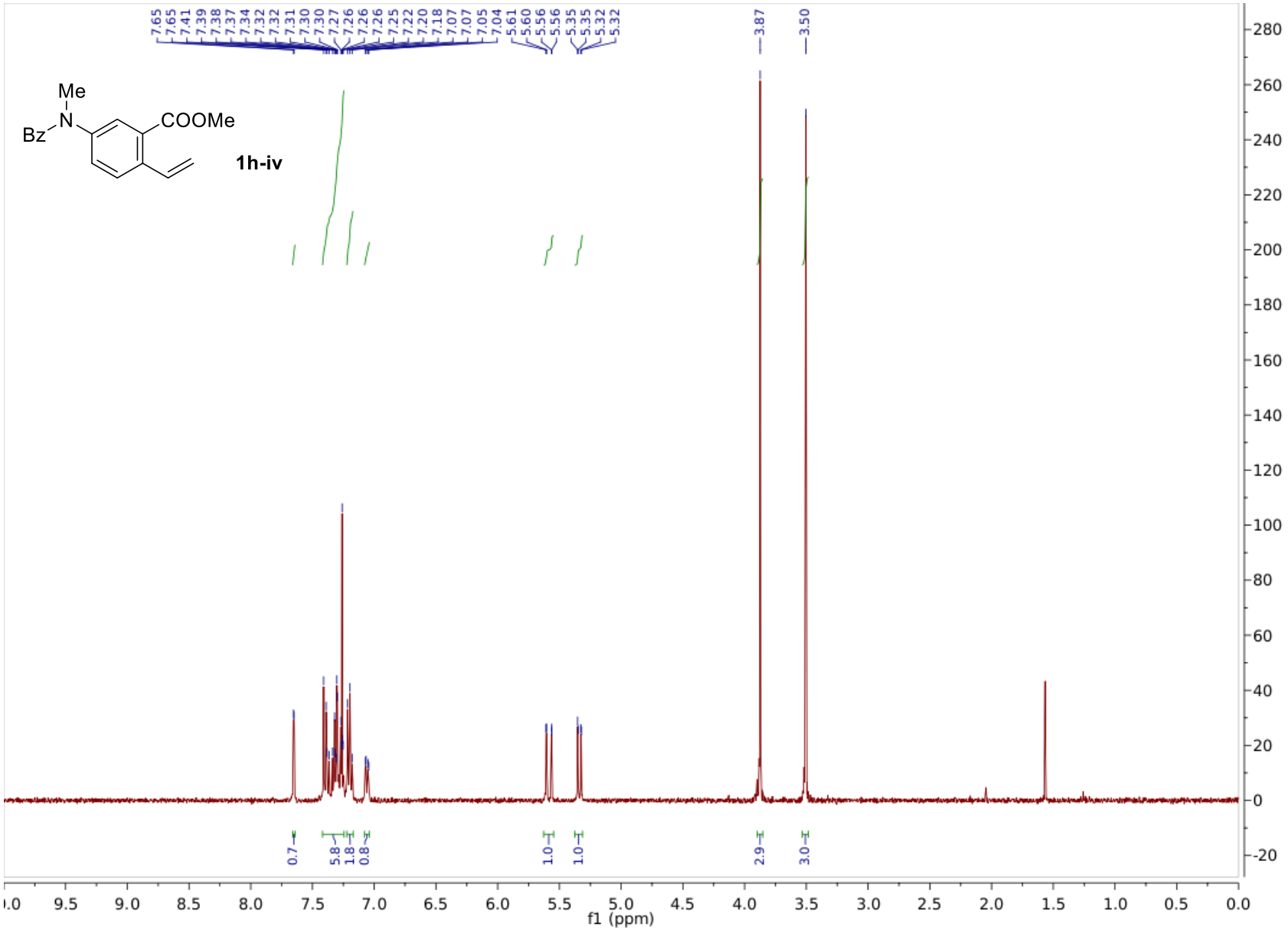

166.7
145.6
134.6
132.2
119.1
117.2
108.6
77.0
52.2
H2N
COOMe
Br
1h-i
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

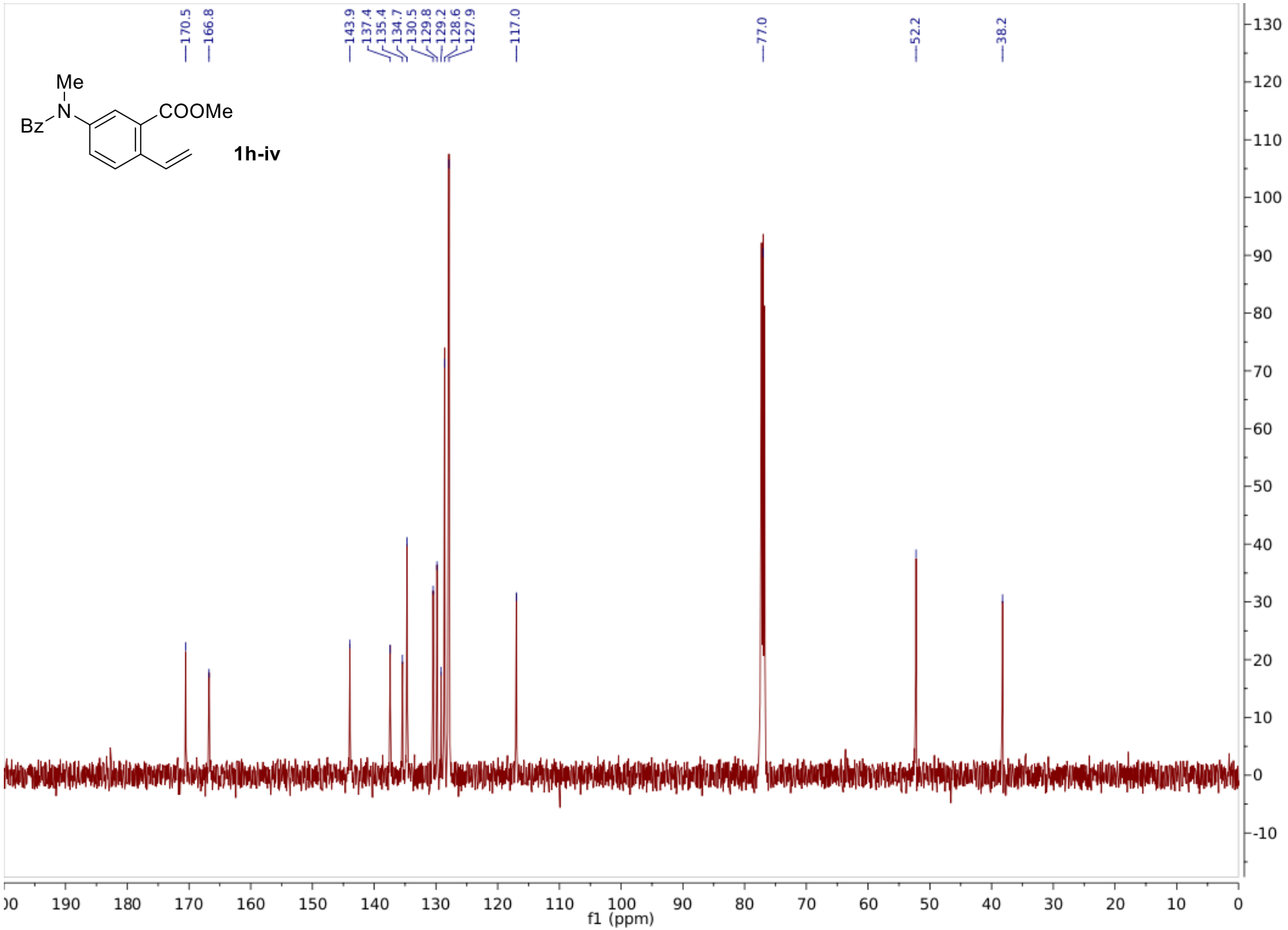

8.07
8.06
8.01
7.78
7.77
7.76
7.75
7.64
7.62
7.59
7.57
7.55
7.50
7.49
7.47
7.26
3.92
H
N
Bz
COOMe
Br
1h-ii
0.9
1.1
2.0
1.0
1.0
1.0
2.1
3.3
f1 (ppm)

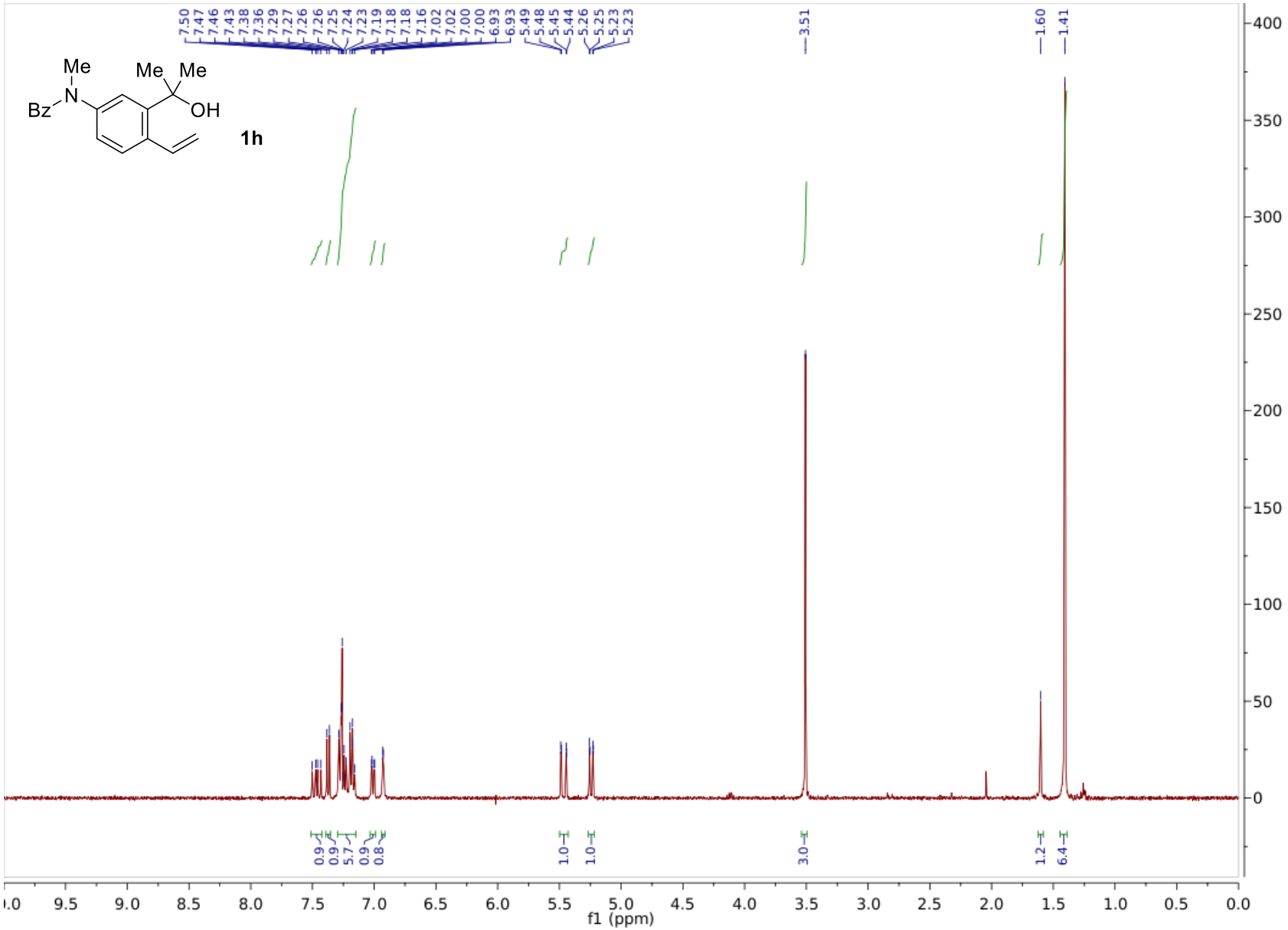

166.1
165.8
137.3
134.9
134.2
132.3
132.2
128.8
127.0
124.2
122.7
116.1
77.0
52.6
H
N
Bz
COOMe
Br
1h-ii
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

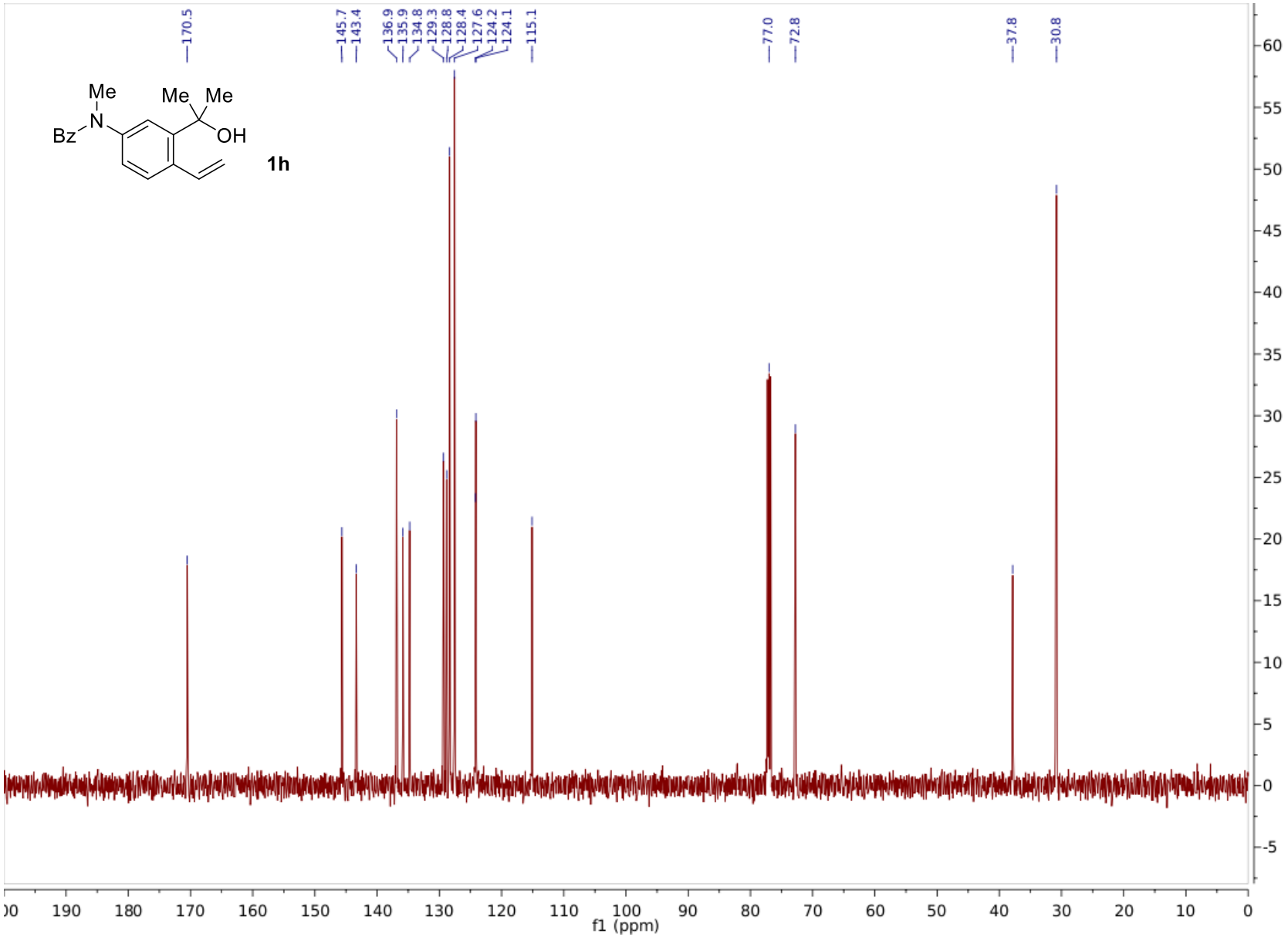

7.58
7.58
7.48
7.45
7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.26
7.26
7.24
7.23
7.23
7.21
6.94
6.93
6.92
6.91
3.90
3.49
Me
N
Bz
COOMe
Br
1h-iii
0.8
0.9
3.1
2.2
1.0
3.4
3.4
f1 (ppm)

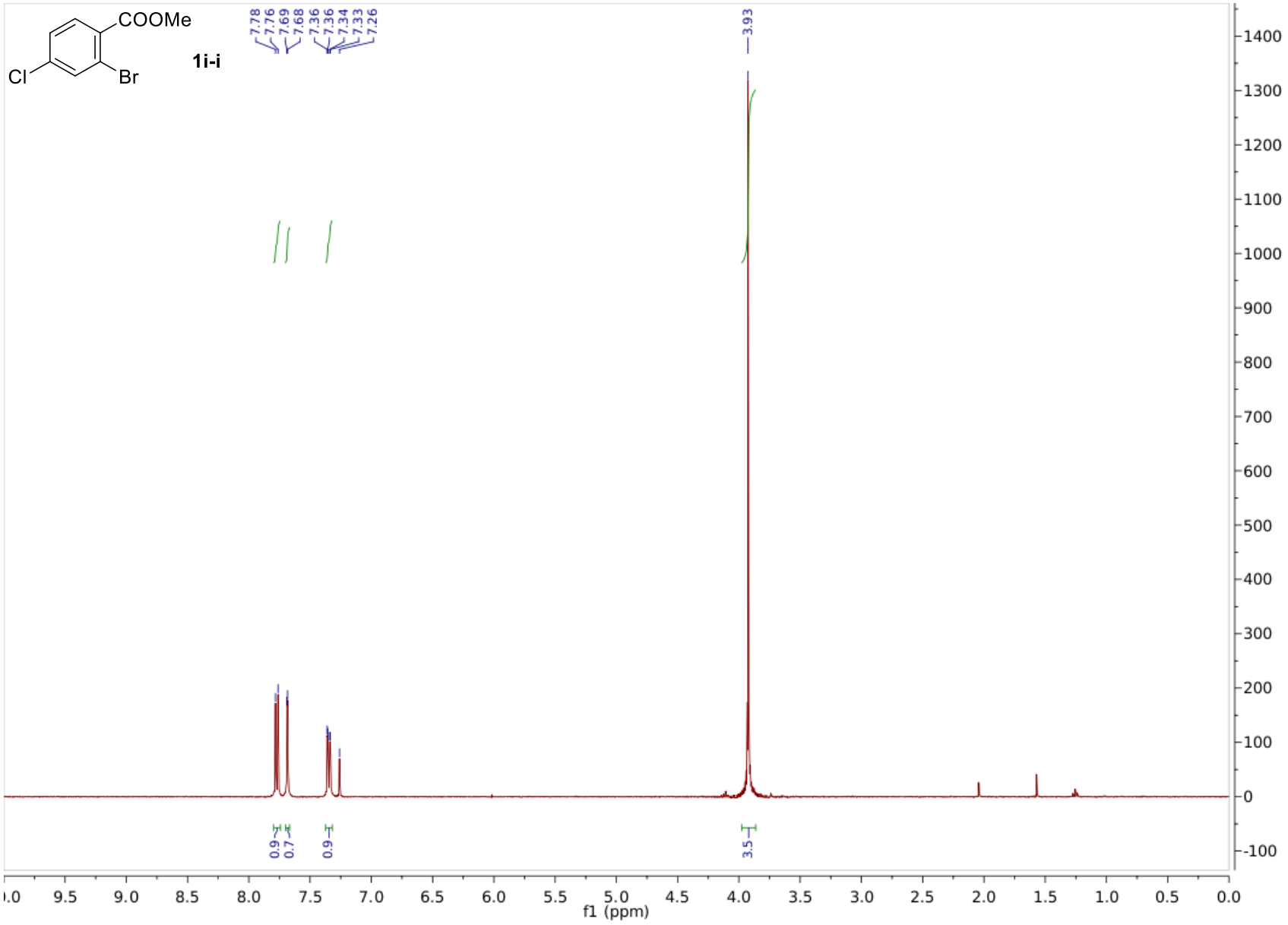

170.4
165.5
144.0
135.0
134.9
132.6
130.8
130.0
129.0
128.5
128.0
119.0
77.0
52.6
38.2
Me
N
Bz
COOMe
Br
1h-iii
f1 (ppm)

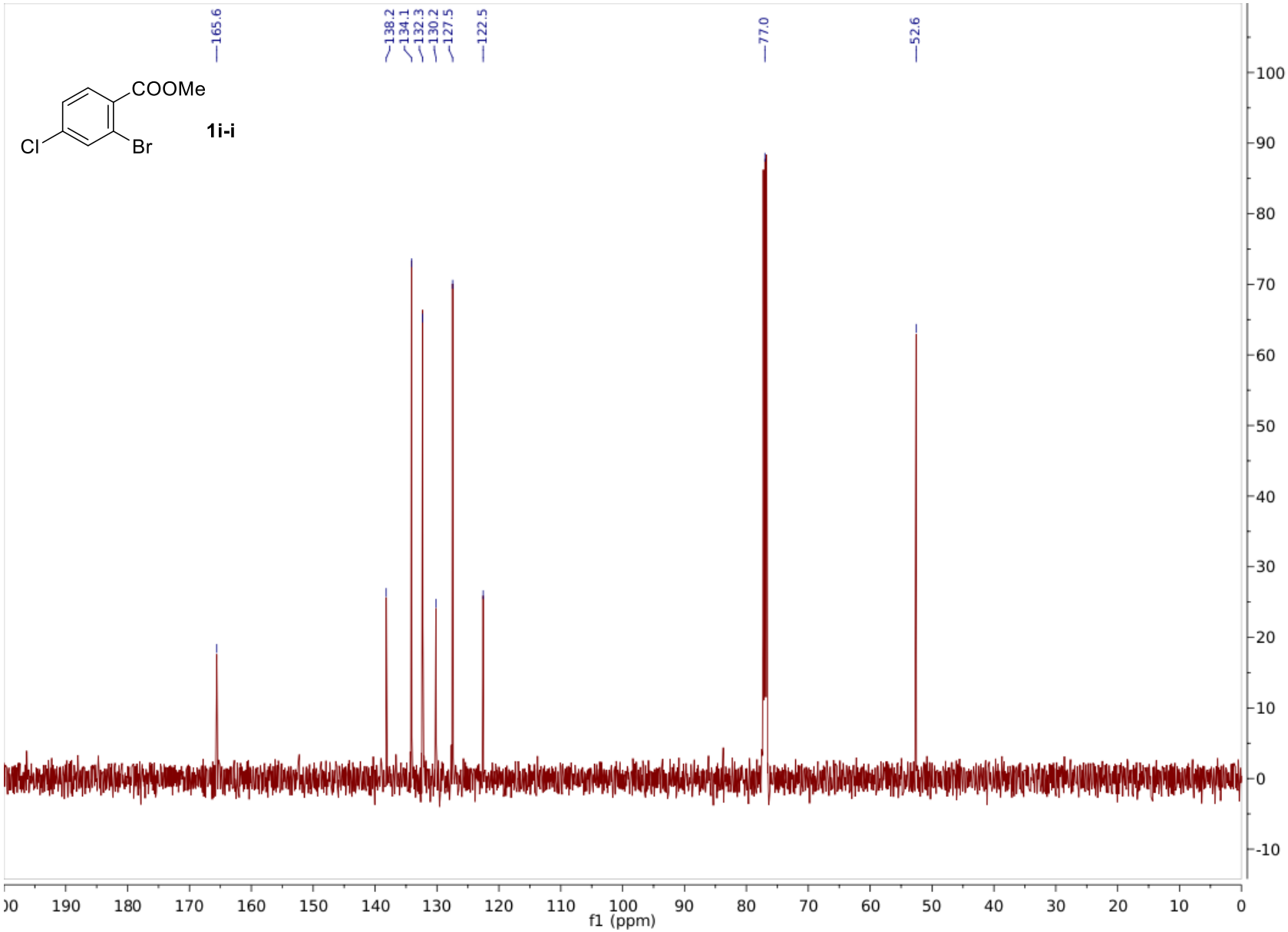

Me
N
Bz
COOMe
1h-iv
7.65
7.65
7.41
7.39
7.38
7.37
7.34
7.32
7.32
7.31
7.30
7.30
7.27
7.26
7.26
7.25
7.22
7.20
7.18
7.07
7.07
7.05
7.04
5.61
5.60
5.56
5.56
5.35
5.35
5.32
5.32
3.87
3.50
0.7
5.8
1.8
0.8
1.0
1.0
2.9
3.0
f1 (ppm)

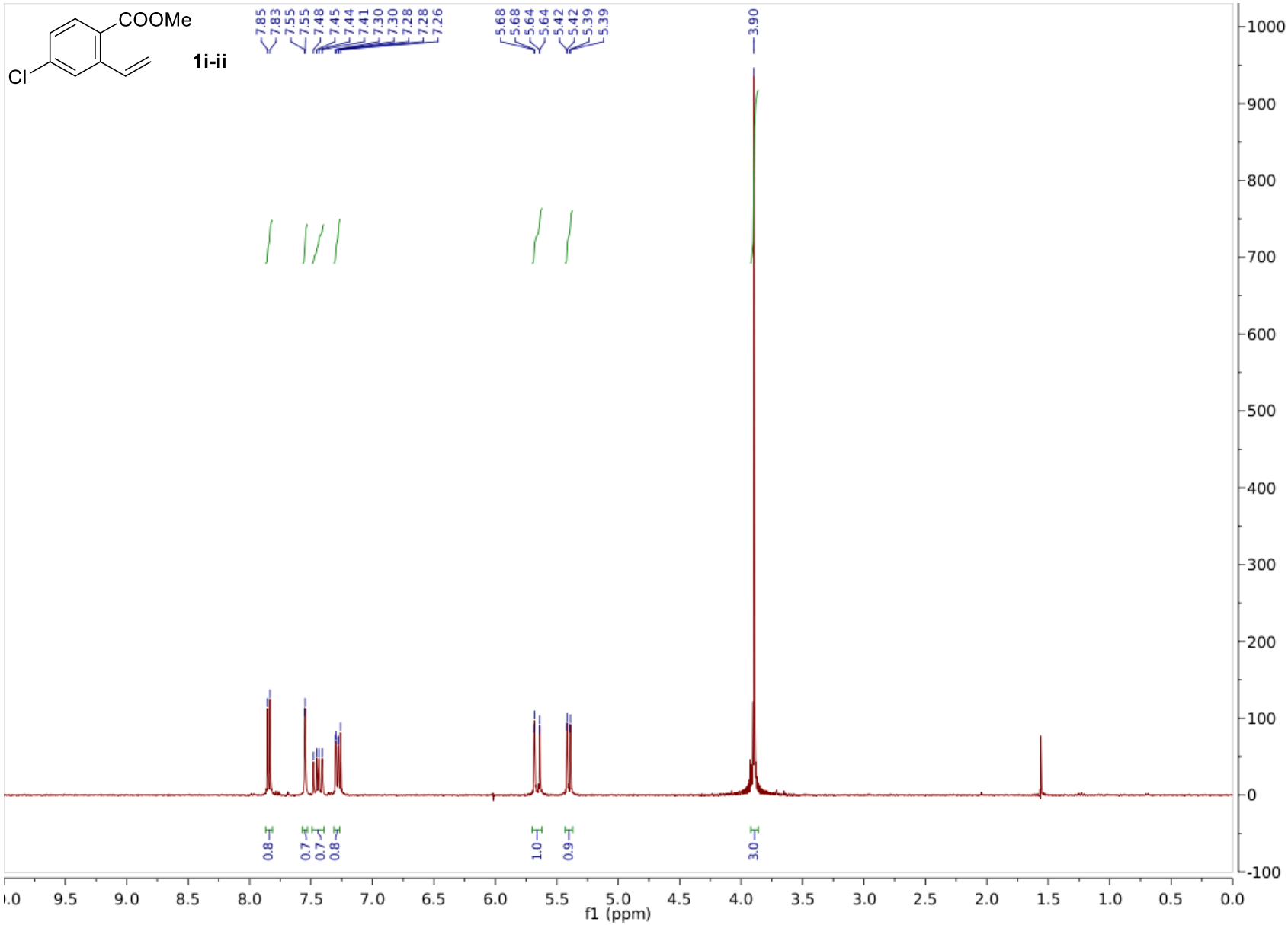

170.5
166.8
143.9
137.4
135.4
134.7
130.5
129.8
129.2
128.6
127.9
117.0
77.0
52.2
38.2
Me
N
Bz
COOMe
1h-iv
f1 (ppm)

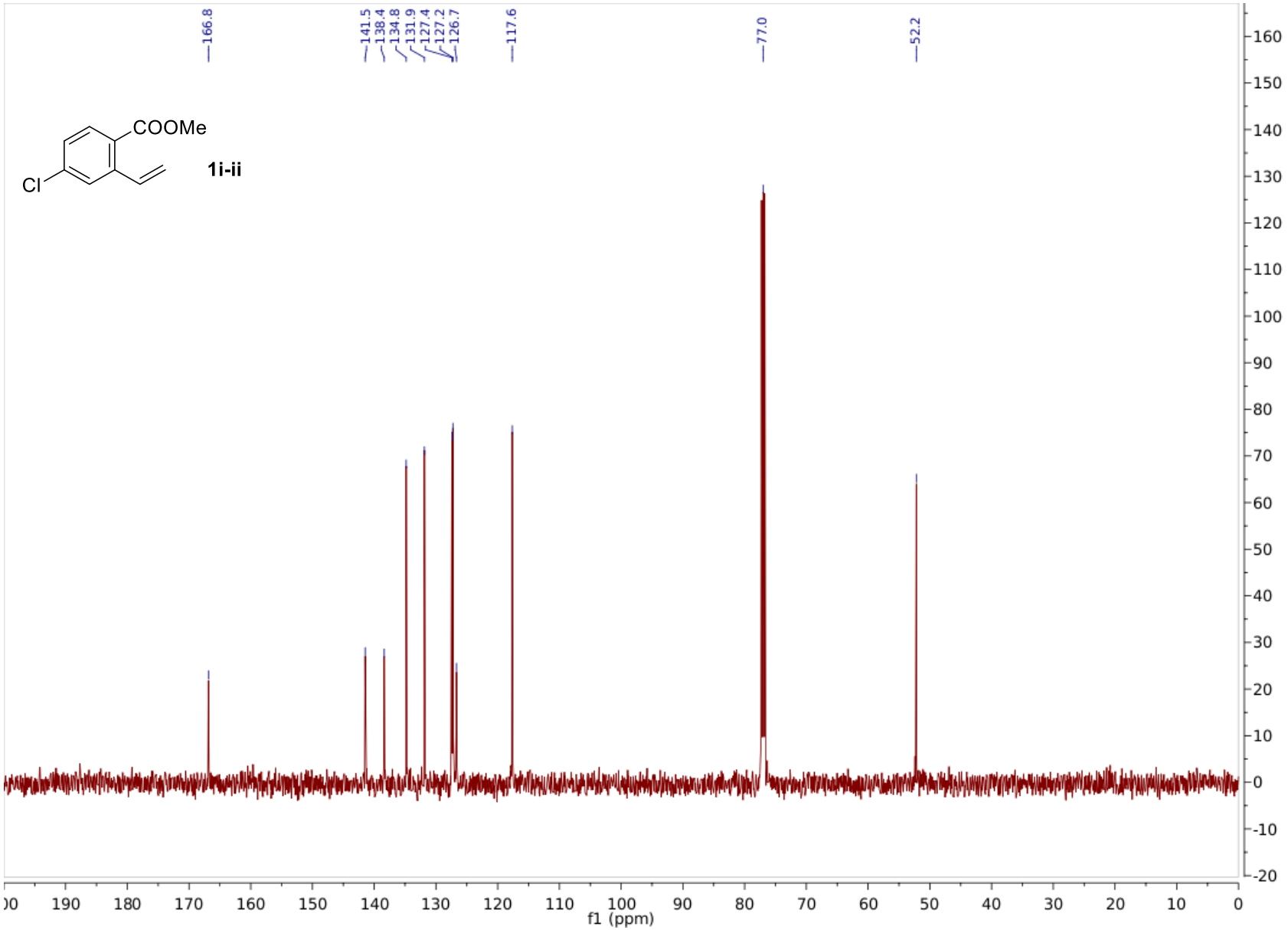

Me
N
Bz
Me
Me
OH
1h
7.50
7.47
7.46
7.43
7.38
7.36
7.29
7.27
7.26
7.26
7.25
7.24
7.23
7.19
7.18
7.18
7.16
7.02
7.02
7.00
7.00
6.93
6.93
5.49
5.48
5.45
5.44
5.26
5.25
5.23
5.23
3.51
1.60
1.41
0.9
0.9
5.7
0.9
0.8
1.0
1.0
3.0
1.2
6.4
f1 (ppm)

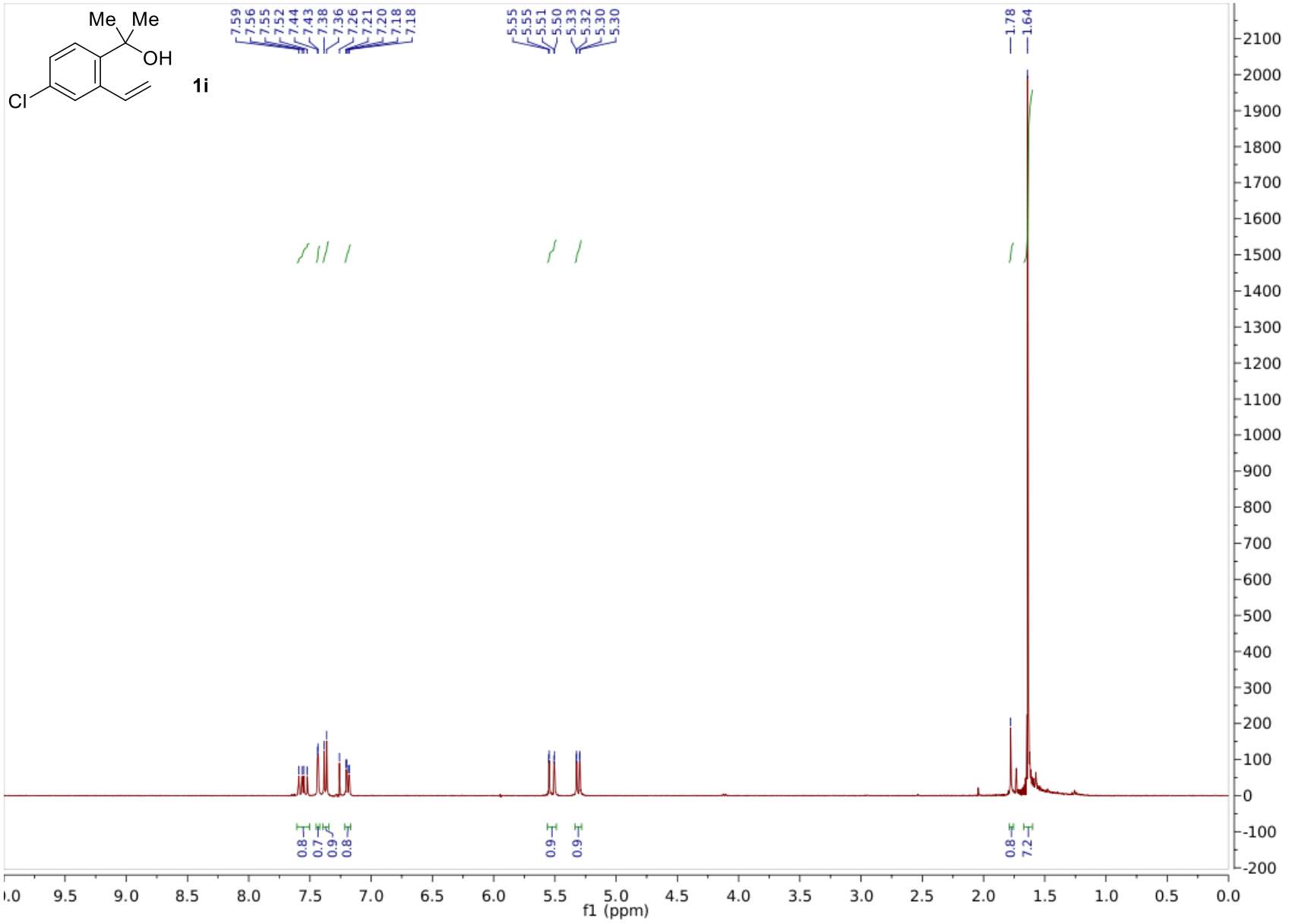

170.5
145.7
143.4
136.9
135.9
134.8
129.3
128.8
128.4
127.6
124.2
124.1
115.1
77.0
72.8
37.8
30.8
Me
N
Bz
Me
Me
OH
1h
f1 (ppm)

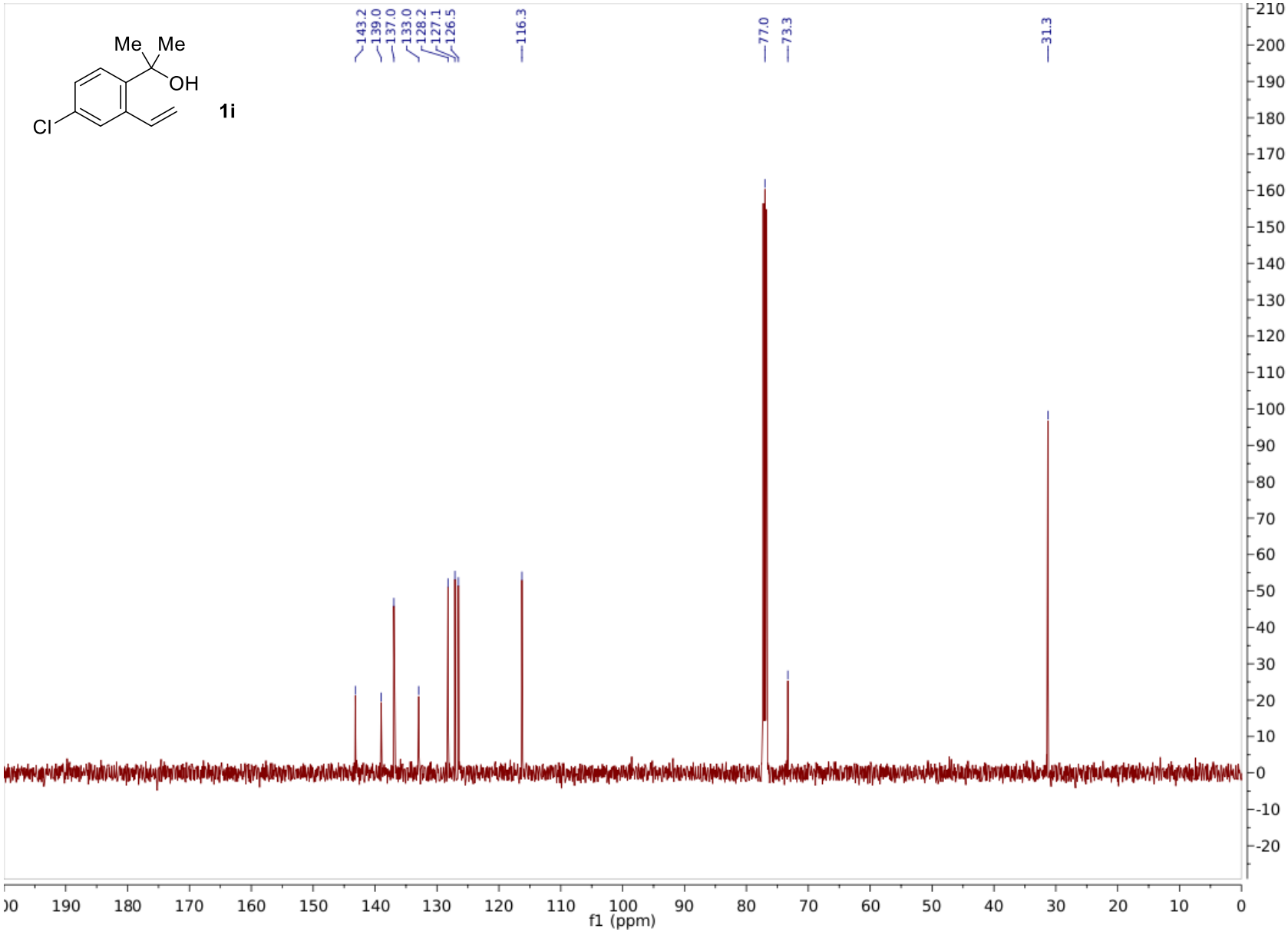

COOMe
Cl
Br
1i-i
7.78
7.76
7.69
7.68
7.36
7.36
7.34
7.33
7.26
3.93
0.9
0.7
0.9
3.5
f1 (ppm)

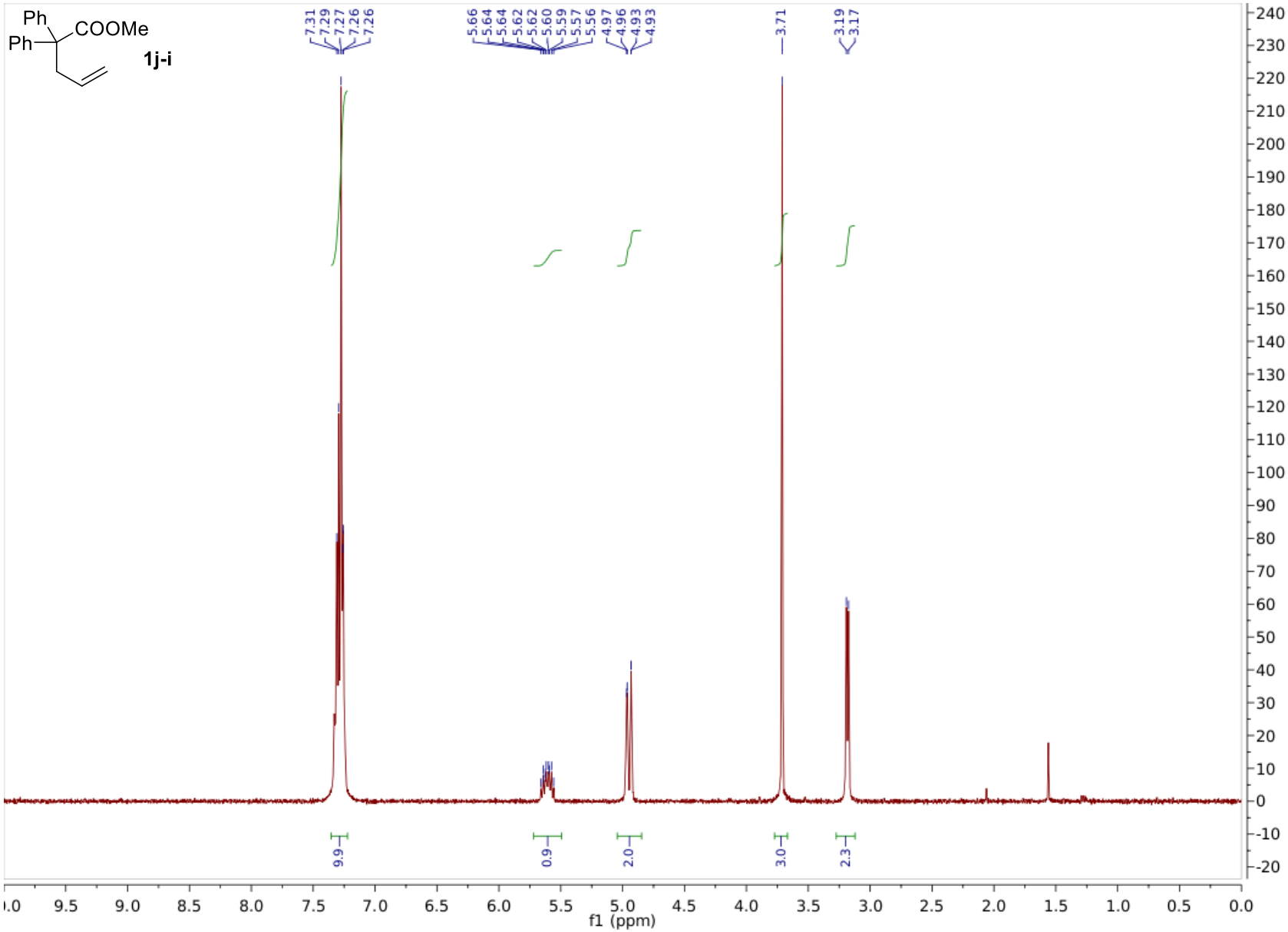

165.6
138.2
134.1
132.3
130.2
127.5
122.5
77.0
52.6
COOMe
Cl
Br
1i-i
f1 (ppm)

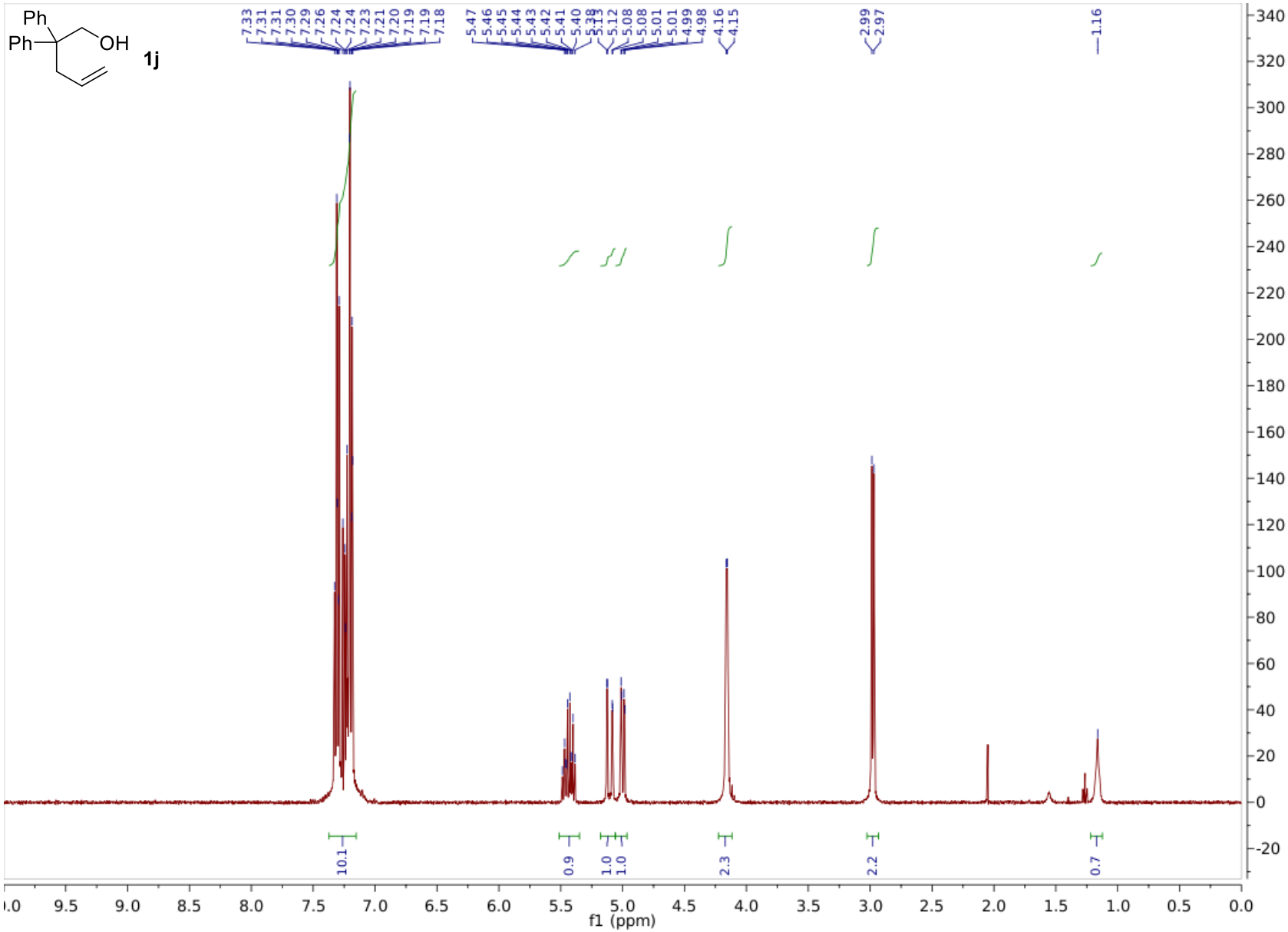

COOMe
Cl
1i-ii
7.85
7.83
7.55
7.55
7.48
7.45
7.44
7.41
7.30
7.28
7.28
7.26
5.68
5.68
5.64
5.64
5.42
5.42
5.39
5.39
3.90
0.8
0.7
0.7
0.8
1.0
0.9
3.0
f1 (ppm)

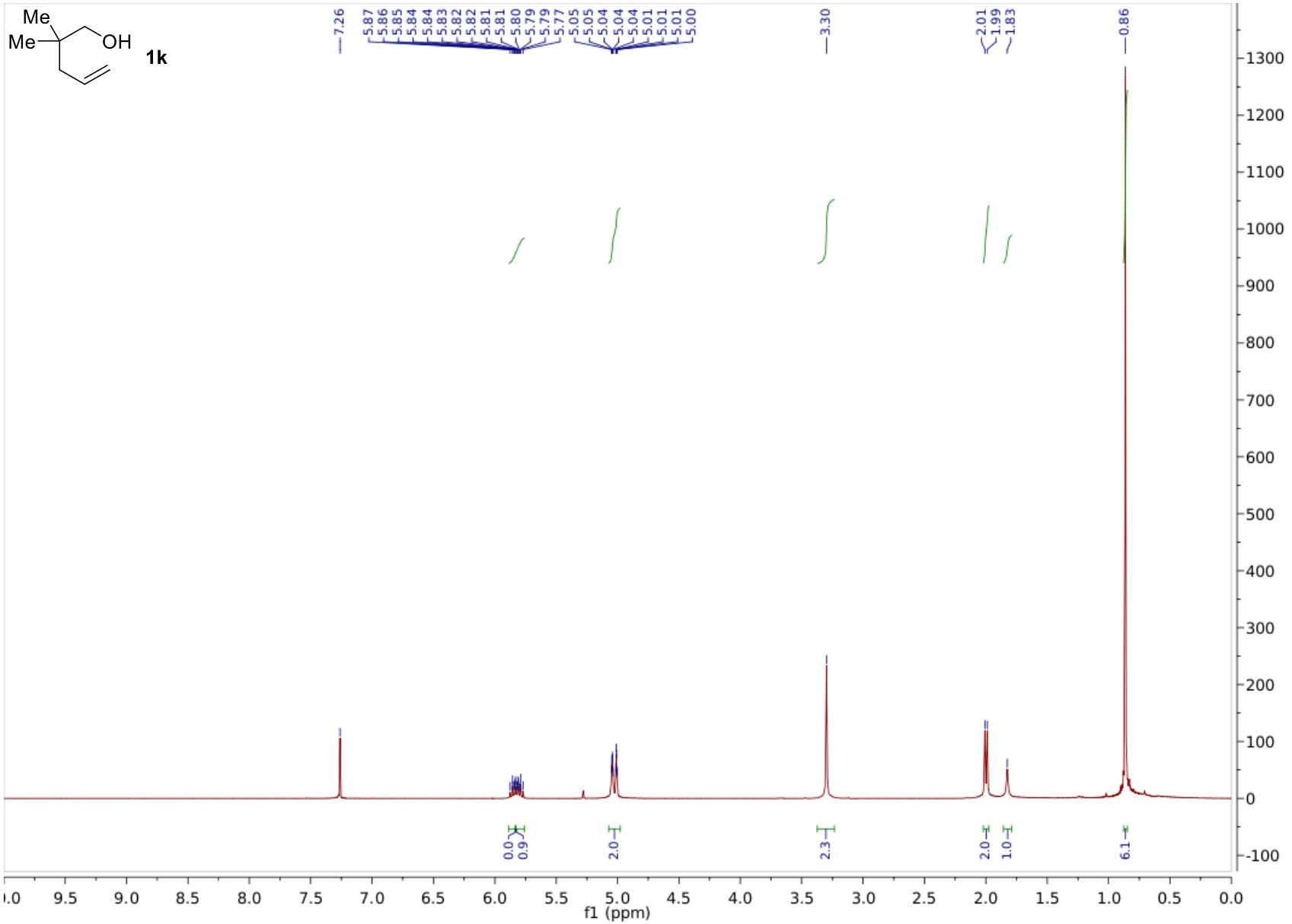

166.8
141.5
138.4
134.8
131.9
127.4
127.2
126.7
117.6
77.0
52.2
COOMe
Cl
1i-ii
f1 (ppm)

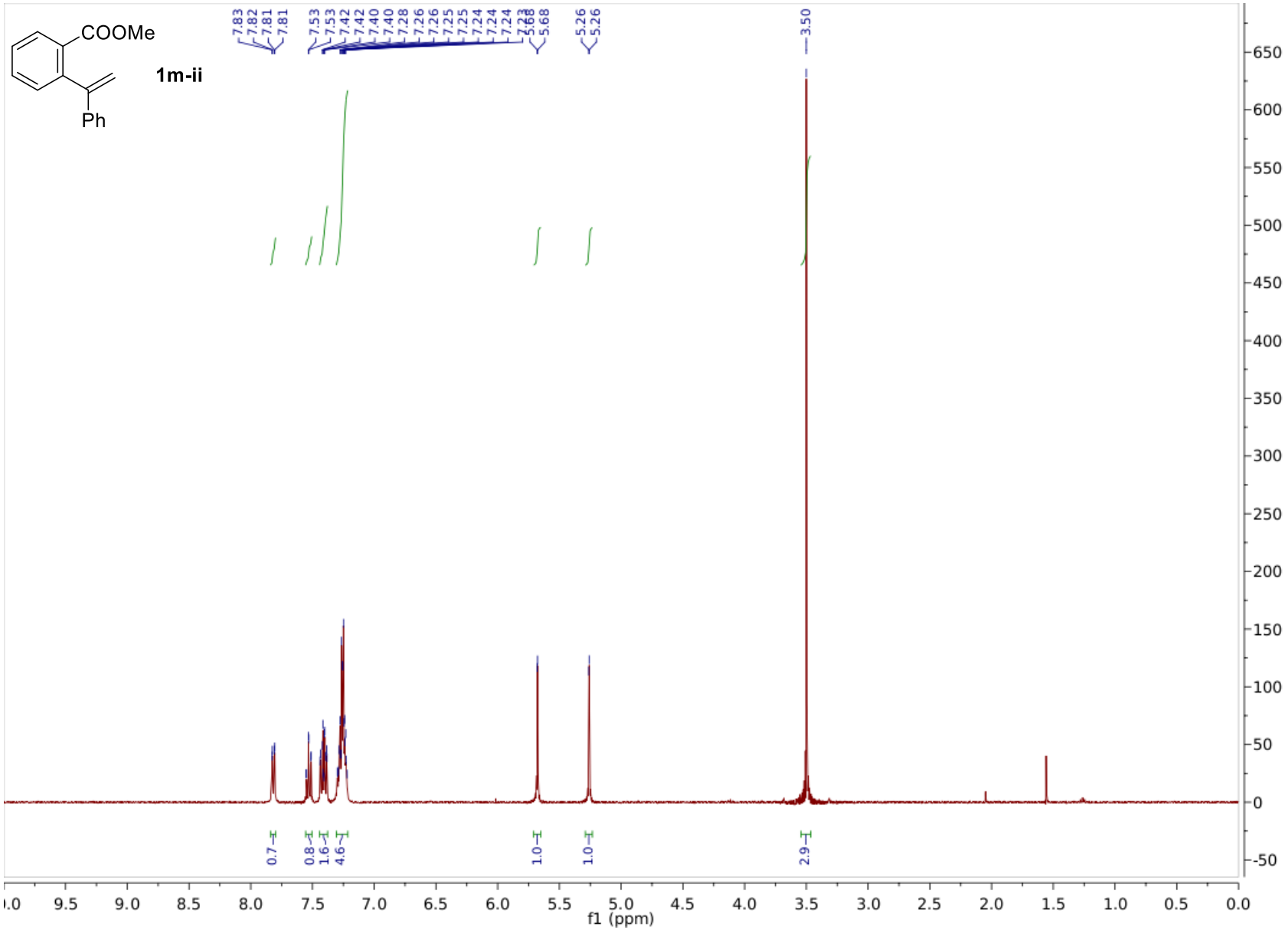

Me Me
OH
Cl
1i
7.59
7.56
7.55
7.52
7.44
7.43
7.38
7.36
7.26
7.21
7.20
7.18
7.18
5.55
5.55
5.51
5.50
5.33
5.32
5.30
5.30
1.78
1.64
0.8
0.7
0.9
0.8
0.9
0.9
0.8
7.2
f1 (ppm)

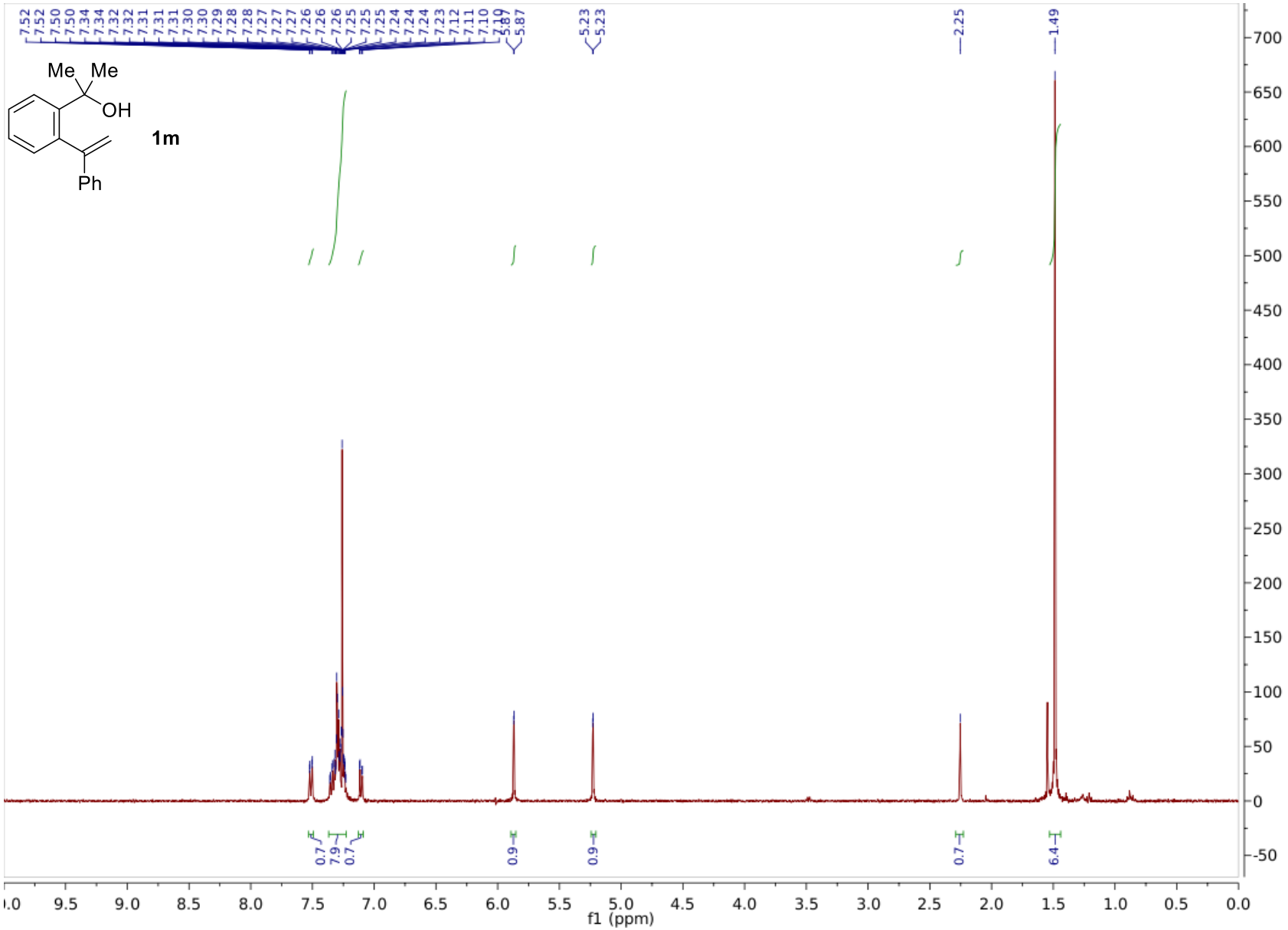

Me
Me
OH
Cl
1i
143.2
139.0
137.0
133.0
128.2
127.1
126.5
116.3
77.0
73.3
31.3
f1 (ppm)

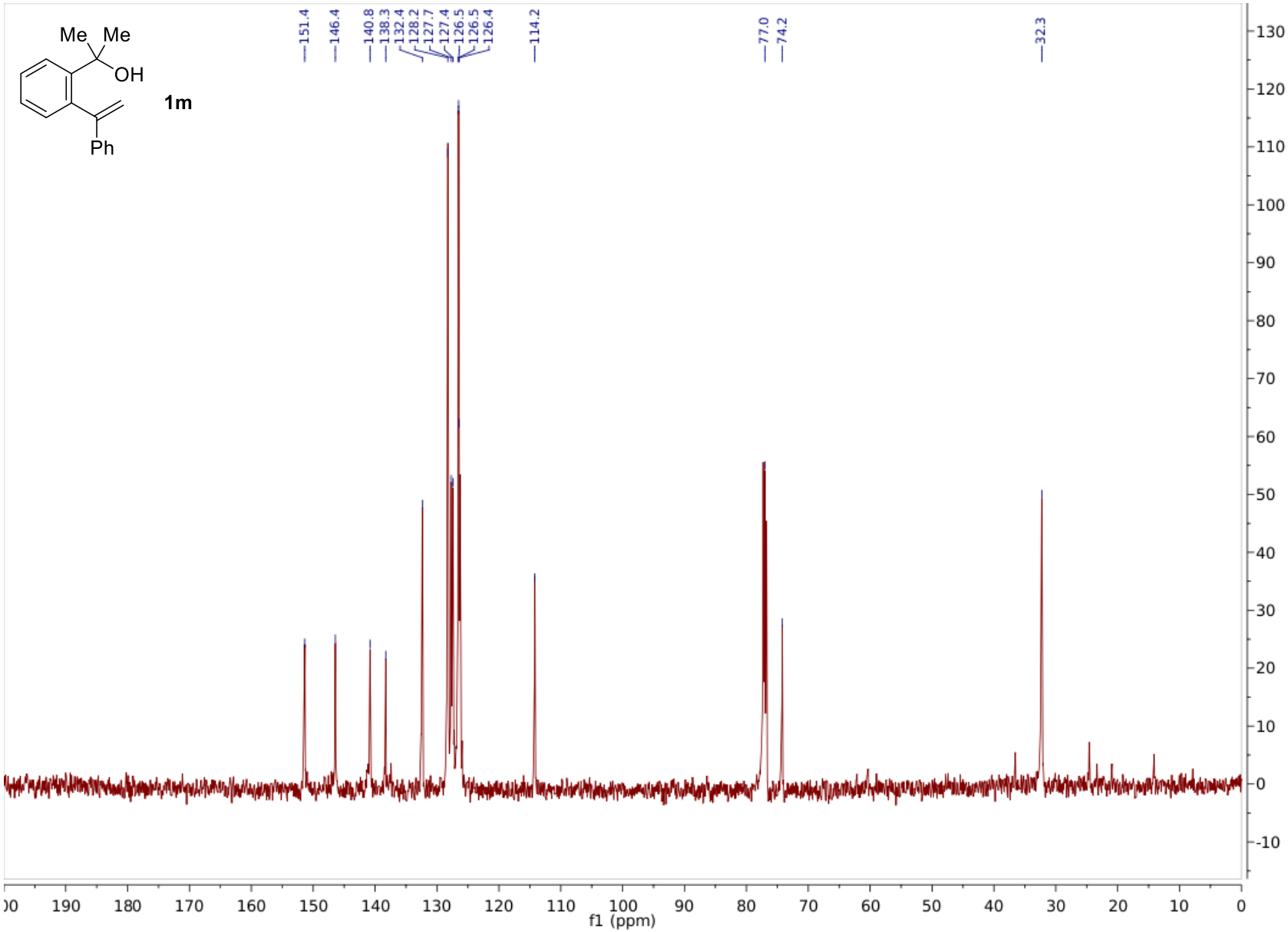

Ph
Ph
COOMe
1j-i
7.31
7.29
7.27
7.26
7.26
5.66
5.64
5.64
5.62
5.62
5.60
5.59
5.57
5.56
4.97
4.96
4.93
4.93
3.71
3.19
3.17
9.9
0.9
2.0
3.0
2.3
f1 (ppm)

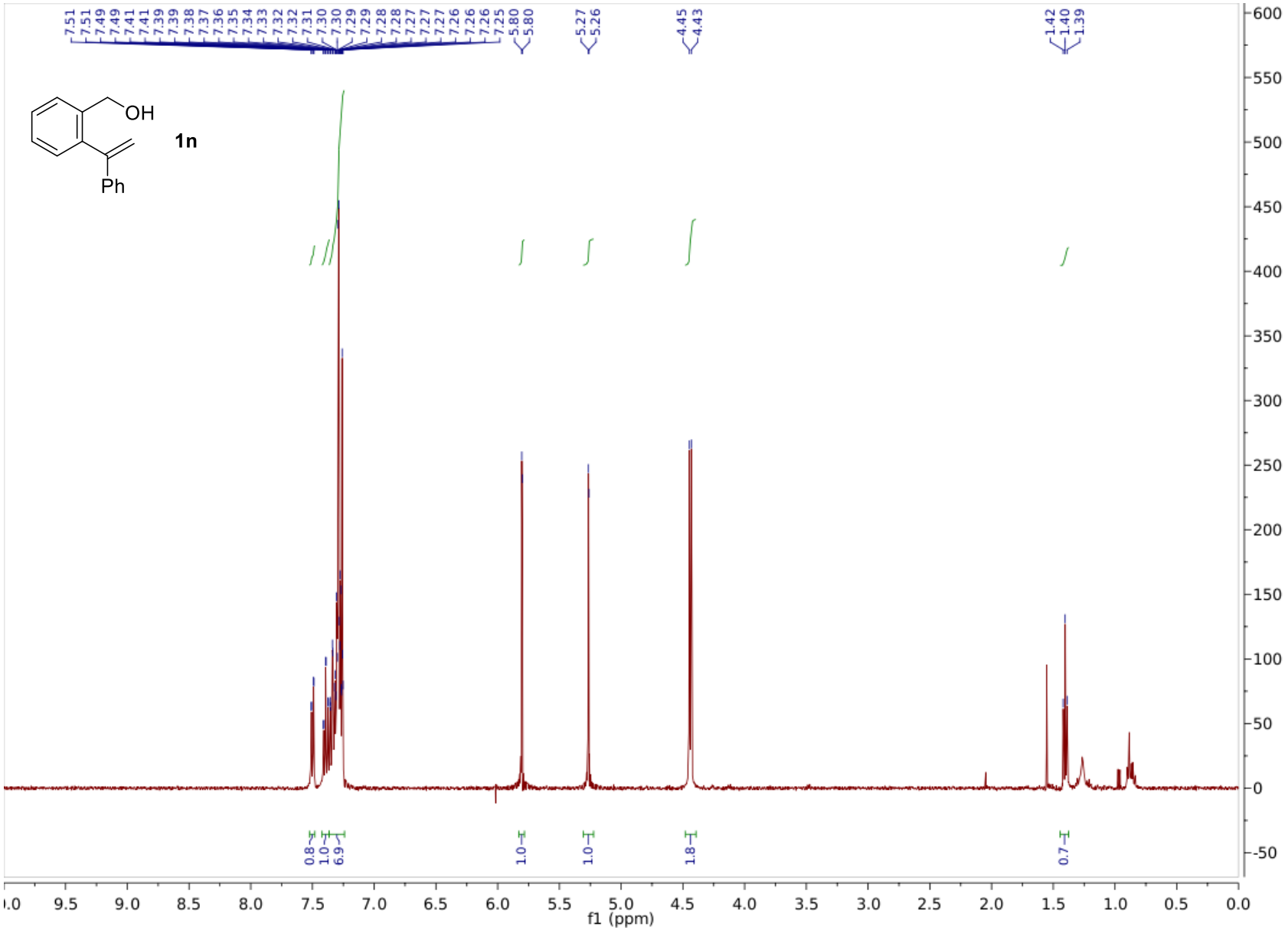

Ph
Ph
OH
1j
7.33
7.31
7.31
7.30
7.29
7.26
7.24
7.24
7.23
7.21
7.20
7.19
7.19
7.18
5.47
5.46
5.45
5.44
5.43
5.42
5.41
5.40
5.38
5.13
5.12
5.08
5.08
5.01
5.01
4.99
4.98
4.16
4.15
2.99
2.97
1.16
10.1
0.9
1.0
1.0
2.3
2.2
0.7
f1 (ppm)

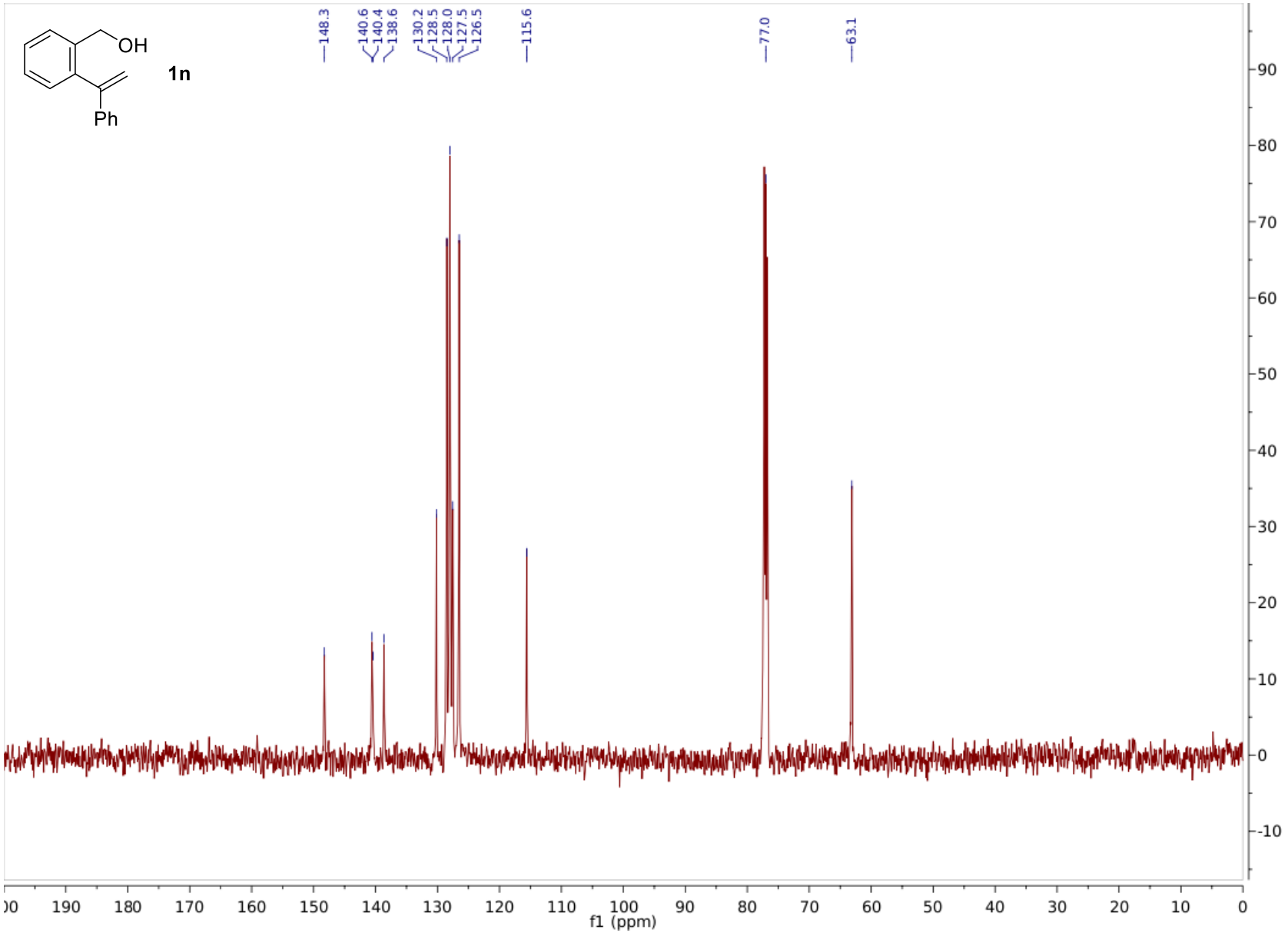

Me
Me
OH
1k
7.26
5.87
5.86
5.85
5.84
5.84
5.83
5.82
5.82
5.81
5.81
5.80
5.79
5.79
5.77
5.05
5.05
5.04
5.04
5.04
5.01
5.01
5.01
5.00
3.30
2.01
1.99
1.83
0.86
0.0
0.9
2.0
2.3
2.0
1.0
6.1
f1 (ppm)

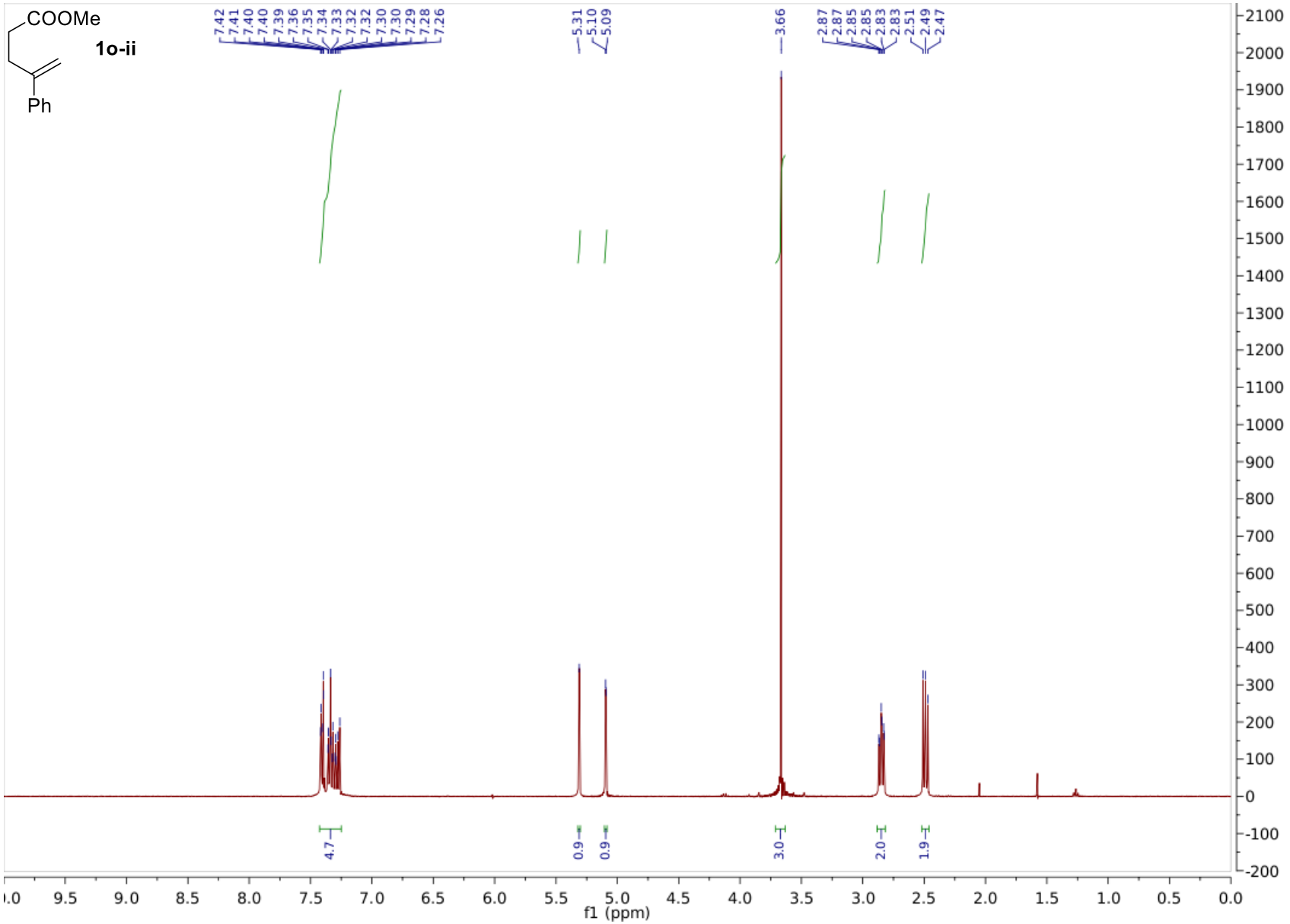

COOMe
Ph
1m-ii
7.83
7.82
7.81
7.81
7.53
7.53
7.42
7.42
7.40
7.40
7.28
7.26
7.26
7.25
7.25
7.24
7.24
7.24
7.23
5.68
5.68
5.26
5.26
3.50
0.7
0.8
1.6
4.6
1.0
1.0
2.9
f1 (ppm)

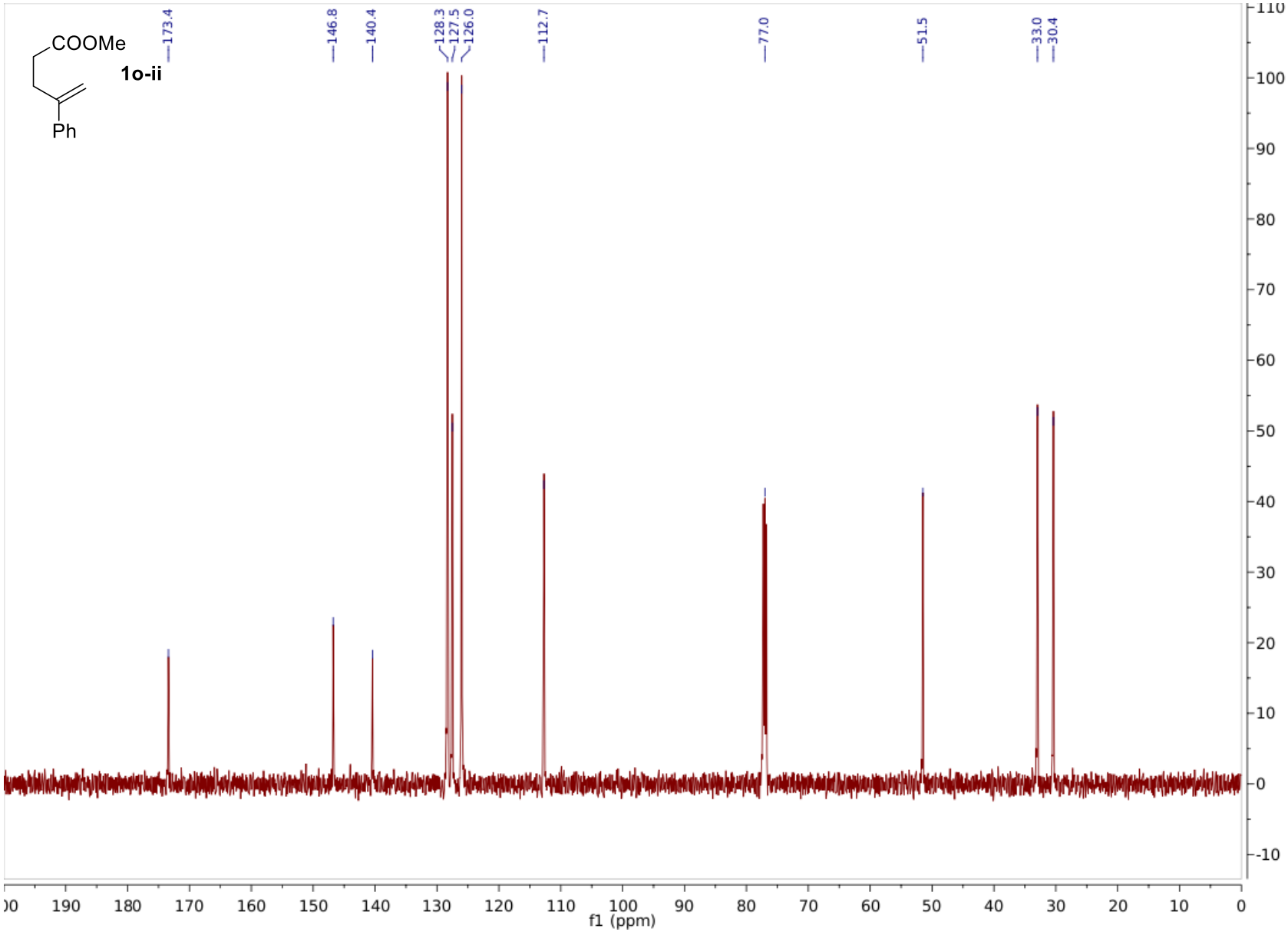

Me
Me
OH
Ph
1m
f1 (ppm)

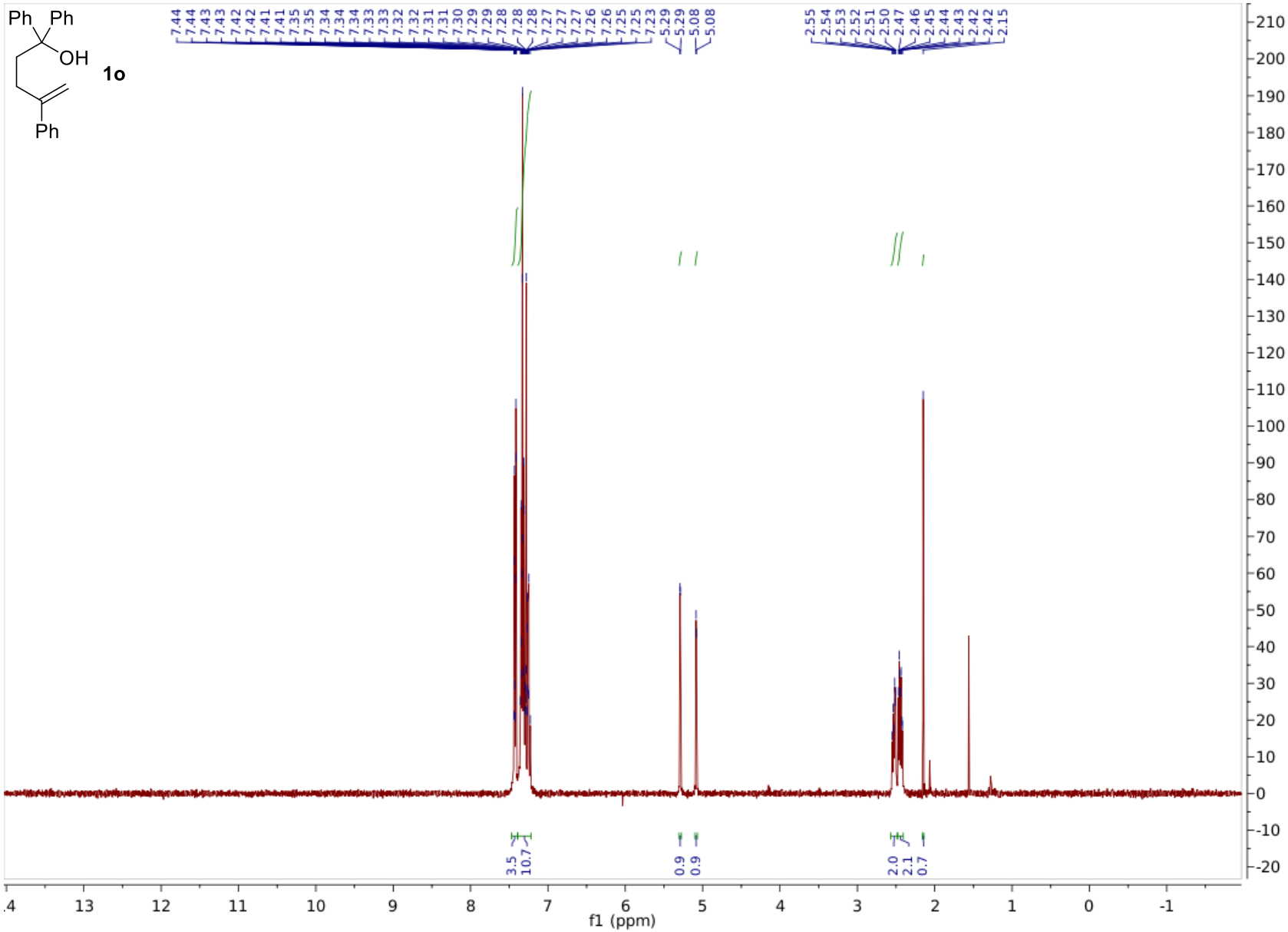

Me
Me
OH
Ph
1m
151.4
146.4
140.8
138.3
132.4
128.2
127.7
127.4
126.5
126.5
126.4
114.2
77.0
74.2
32.3
f1 (ppm)

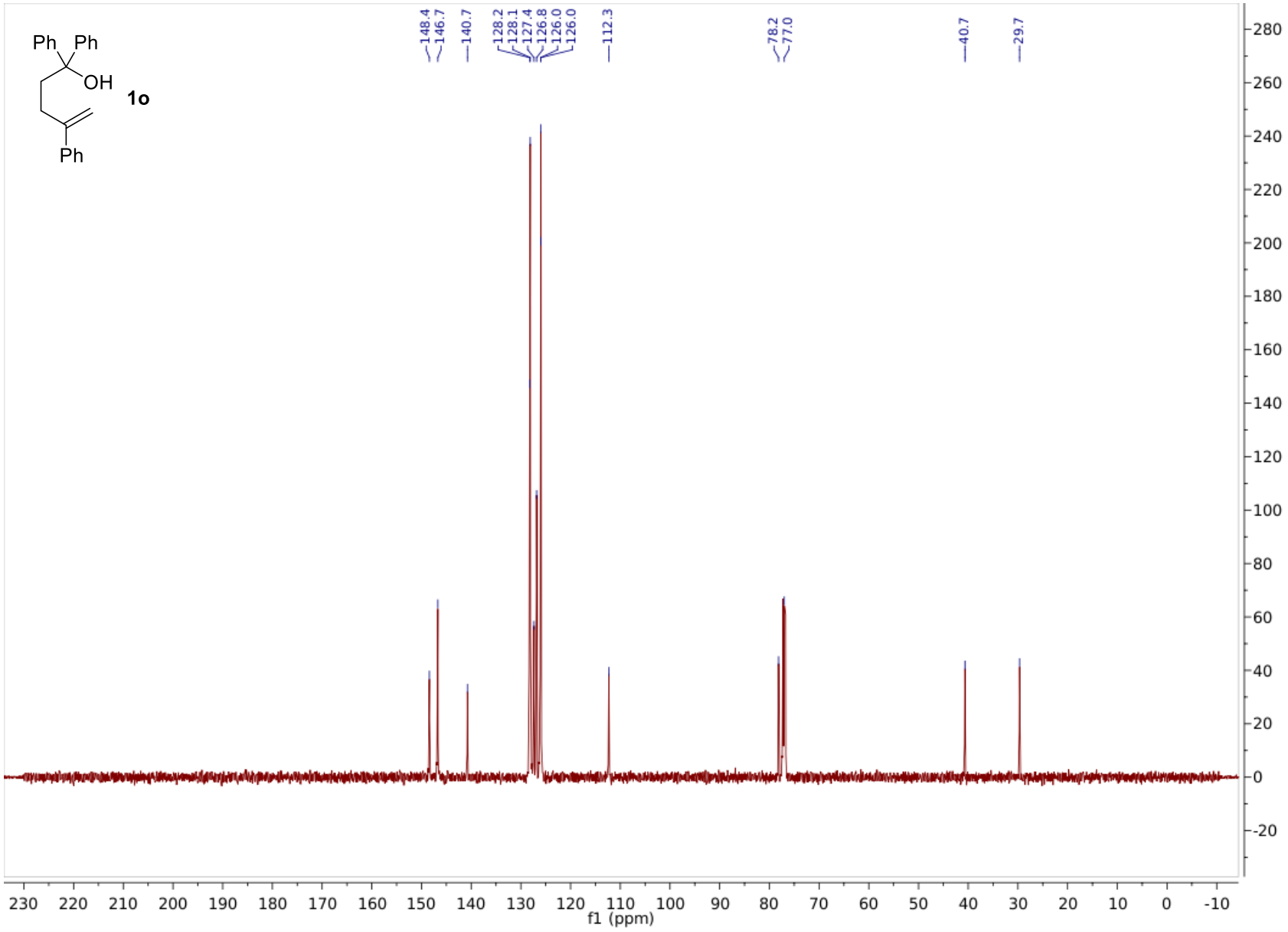

OH
Ph
1n
f1 (ppm)

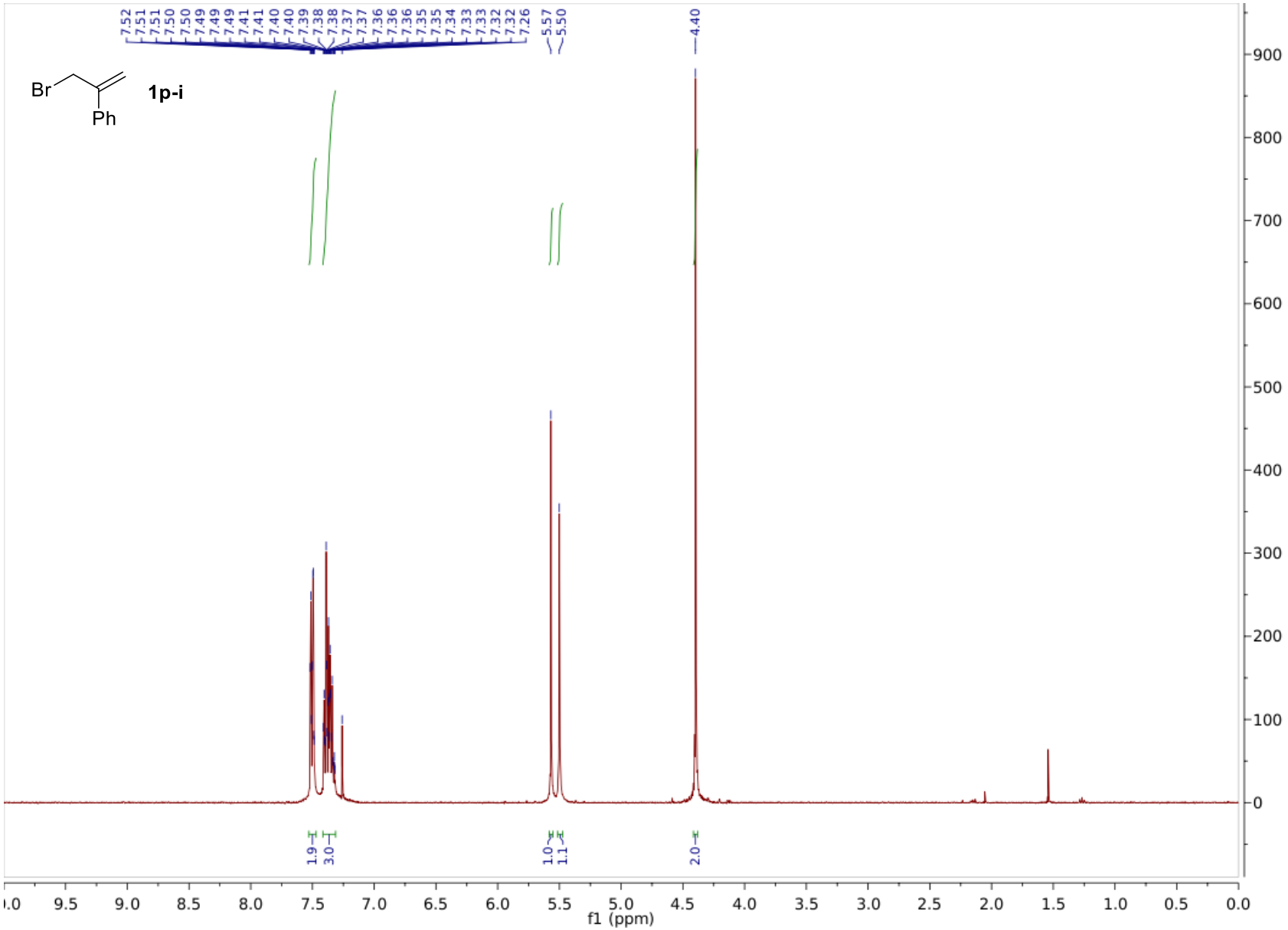

OH
Ph
1n
148.3
140.6
140.4
138.6
130.2
128.5
128.0
127.5
126.5
115.6
77.0
63.1
f1 (ppm)

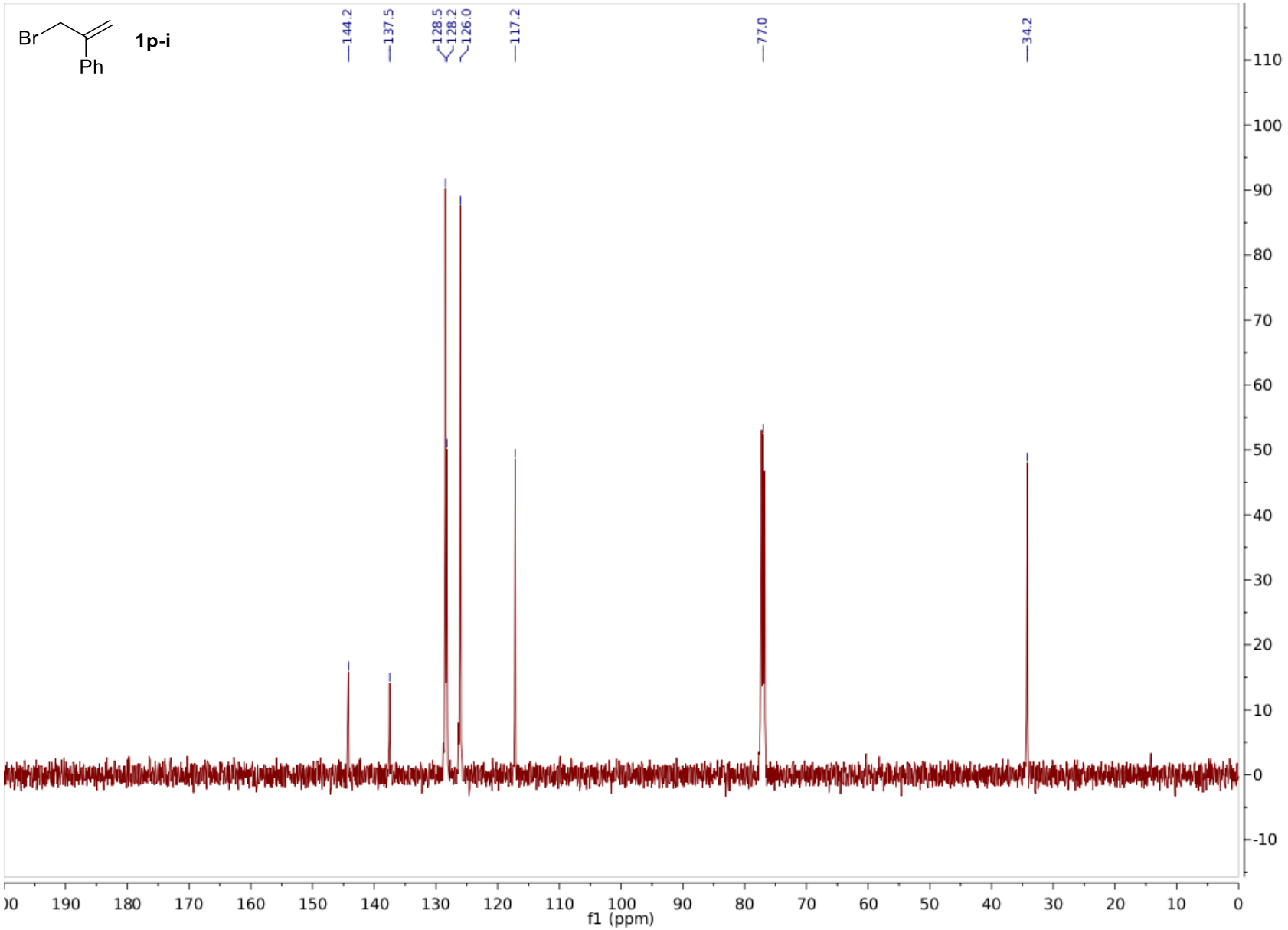

COOMe
1o-ii
Ph
7.42
7.41
7.40
7.40
7.39
7.36
7.35
7.34
7.33
7.32
7.32
7.30
7.30
7.29
7.28
7.26
5.31
5.10
5.09
3.66
2.87
2.87
2.85
2.85
2.83
2.83
2.51
2.49
2.47
4.7
0.9
0.9
3.0
2.0
1.9
f1 (ppm)

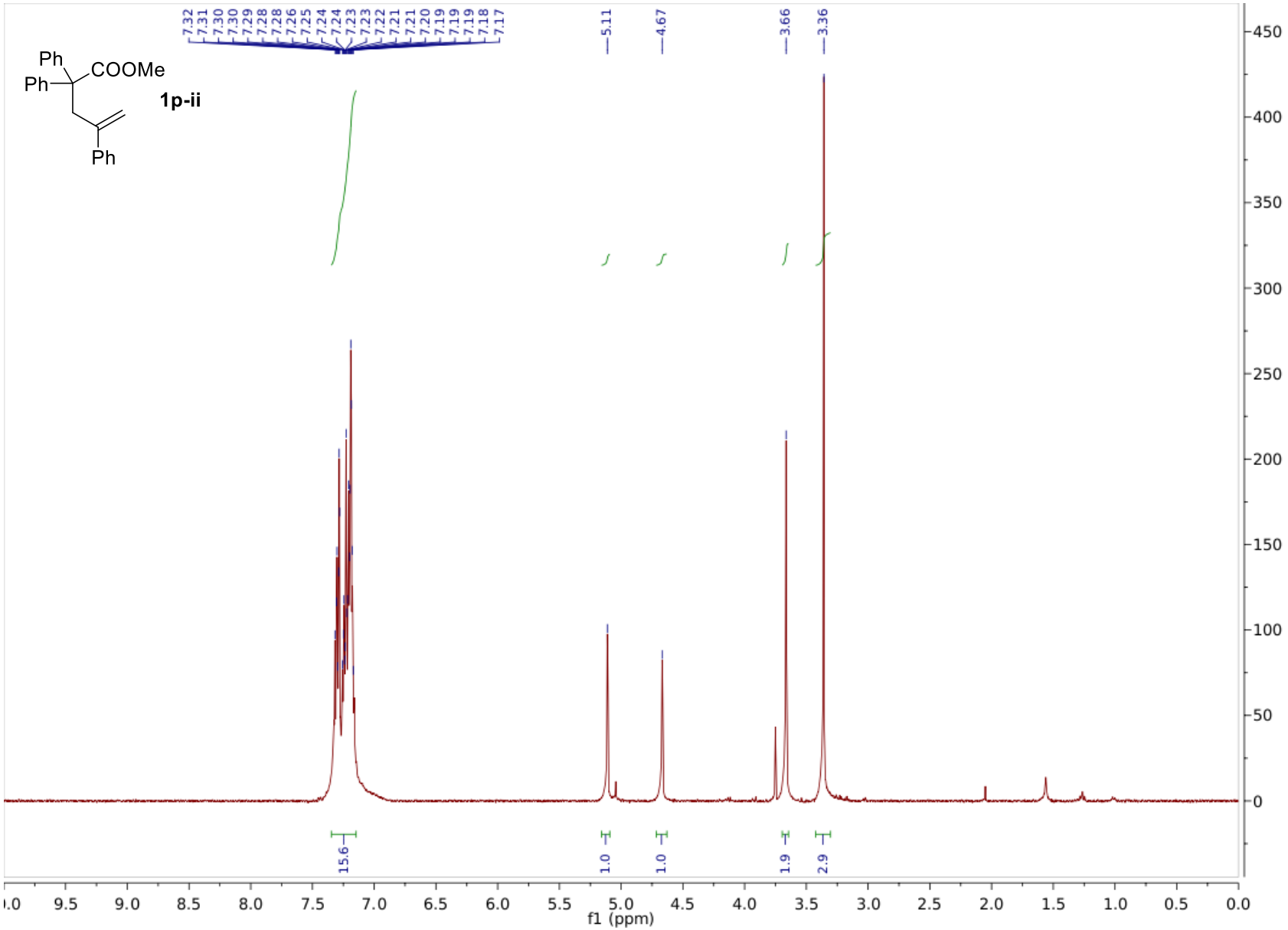

COOMe
1o-ii
Ph
173.4
146.8
140.4
128.3
127.5
126.0
112.7
77.0
51.5
33.0
30.4
f1 (ppm)

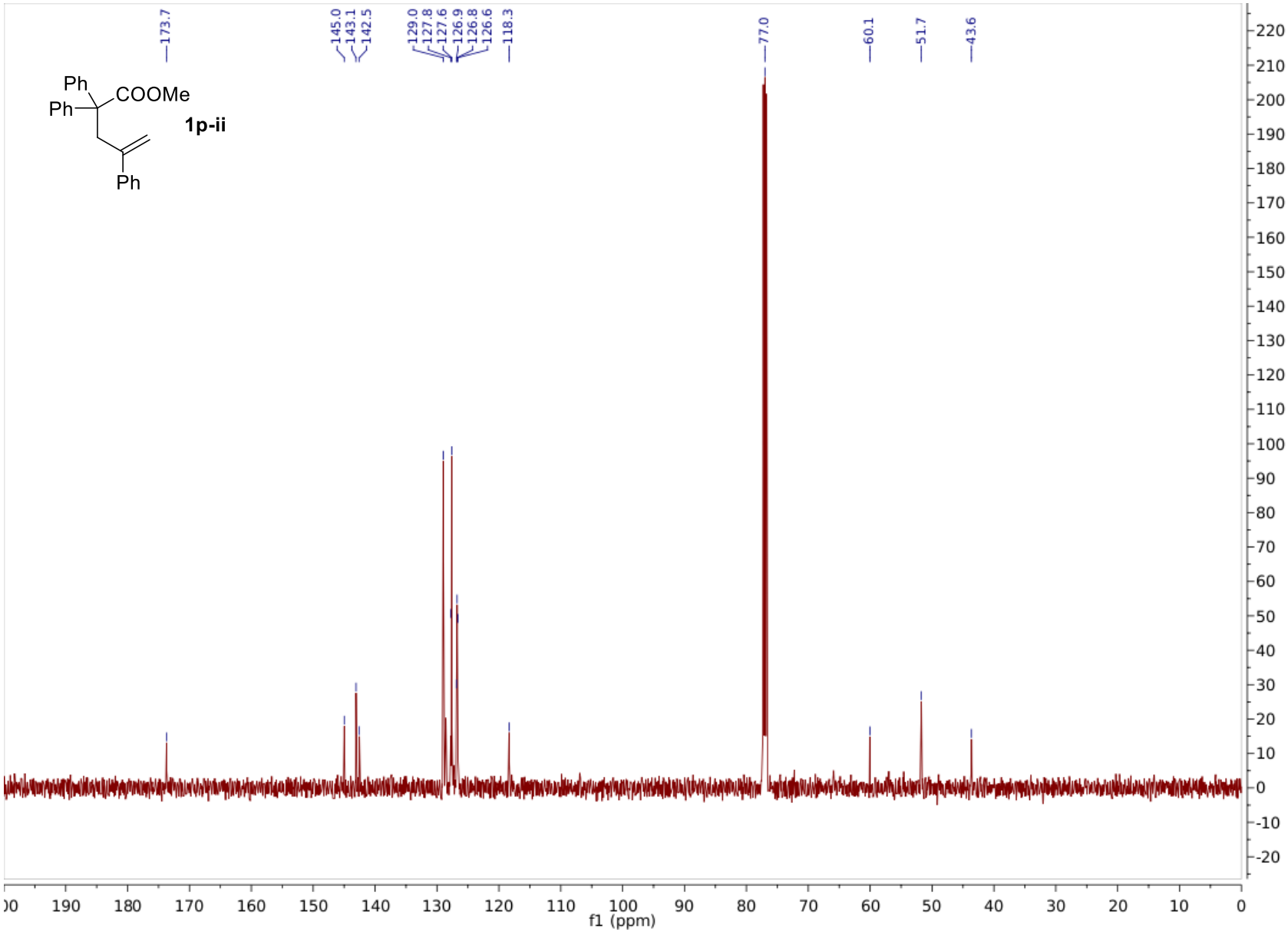

Ph
Ph
OH
Ph
1o
f1 (ppm)

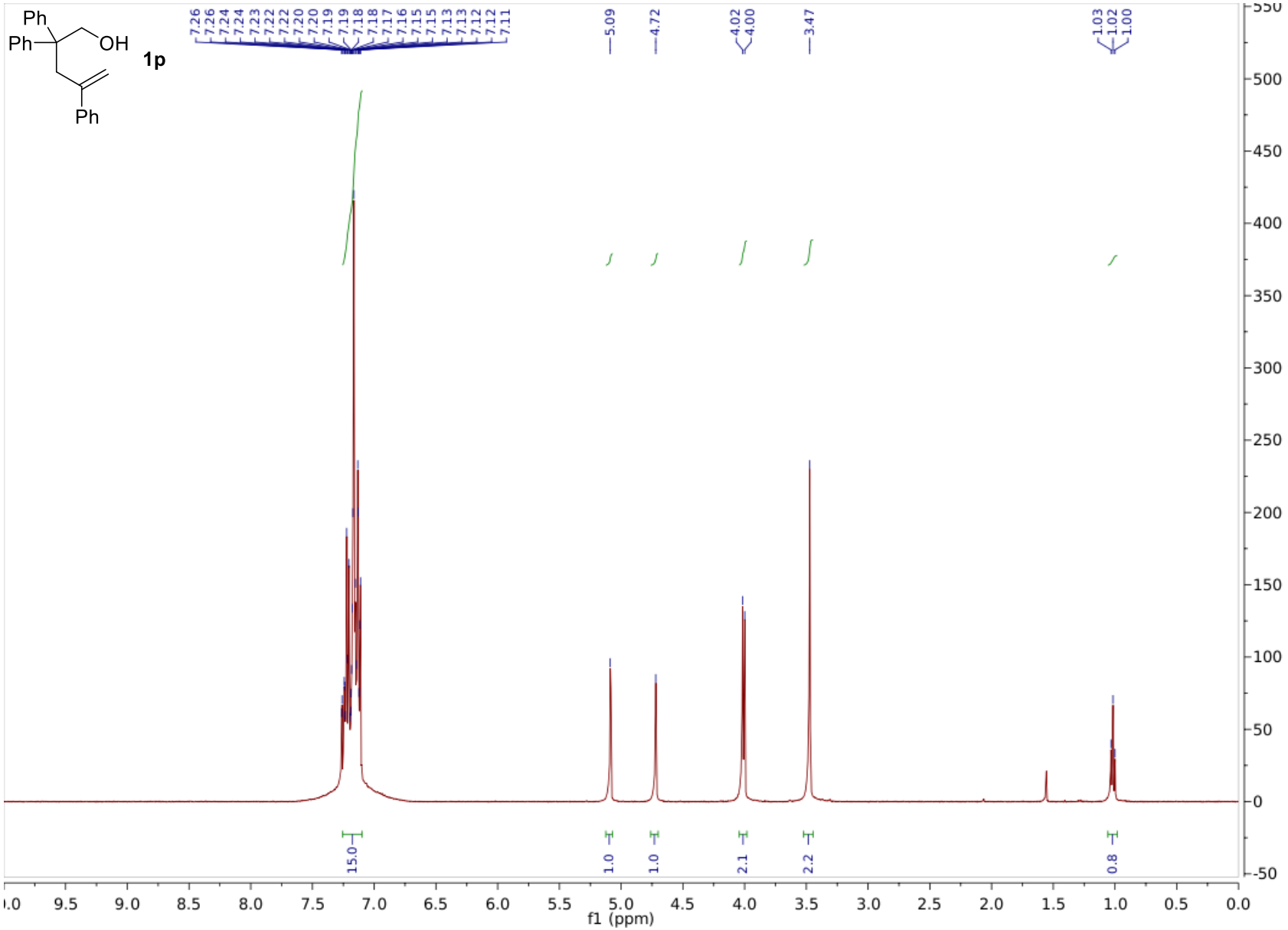

Ph
Ph
OH
Ph
1o
148.4
146.7
140.7
128.2
128.1
127.4
126.8
126.0
126.0
112.3
78.2
77.0
40.7
29.7
f1 (ppm)

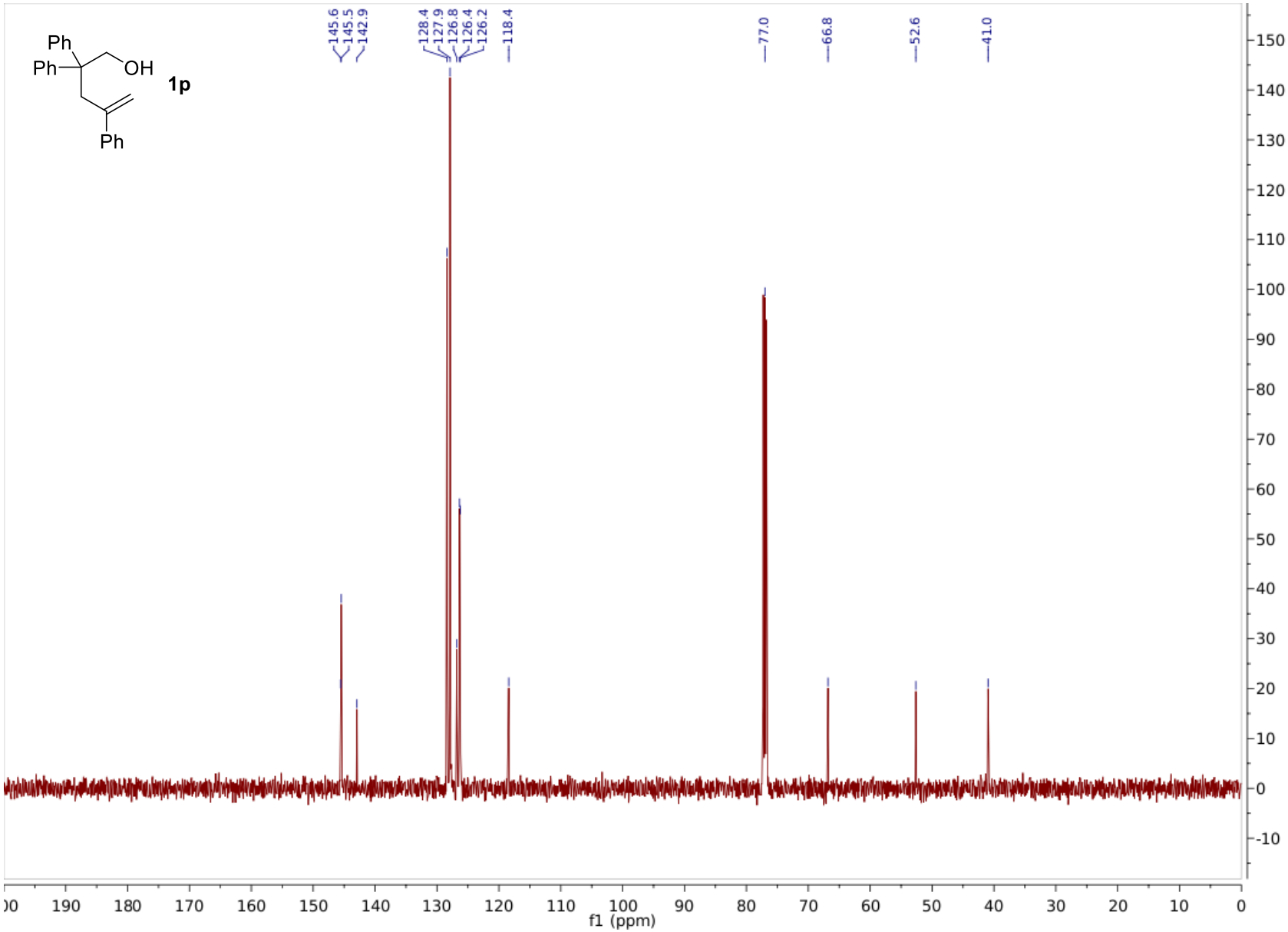

7.52
7.51
7.51
7.50
7.50
7.49
7.49
7.49
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.37
7.37
7.36
7.36
7.36
7.35
7.35
7.34
7.33
7.33
7.32
7.32
7.26
5.57
5.50
4.40
Br
Ph
1p-i
1.9
3.0
1.0
1.1
2.0
f1 (ppm)

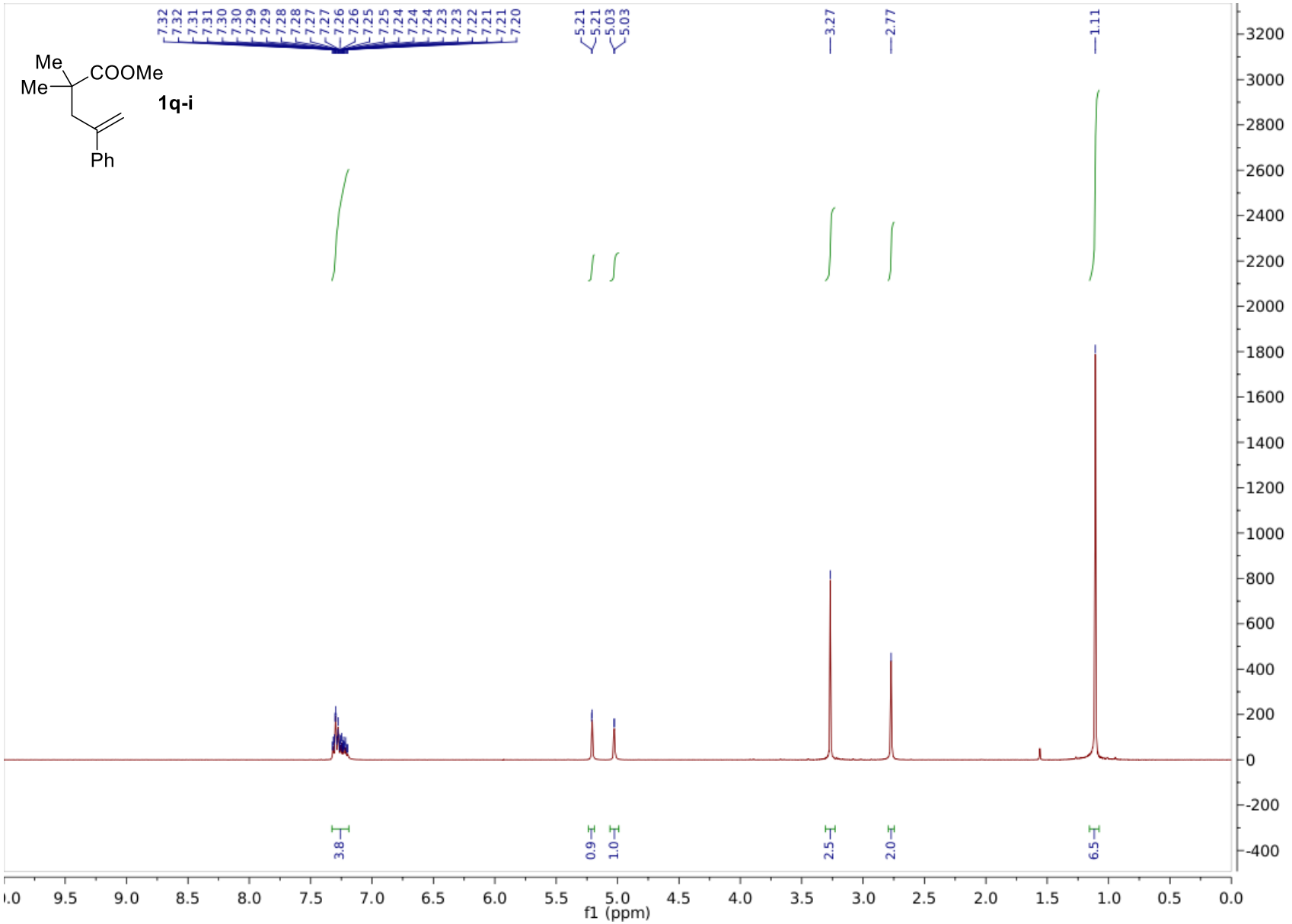

Br
Ph
1p-i
144.2
137.5
128.5
128.2
126.0
117.2
77.0
34.2
f1 (ppm)

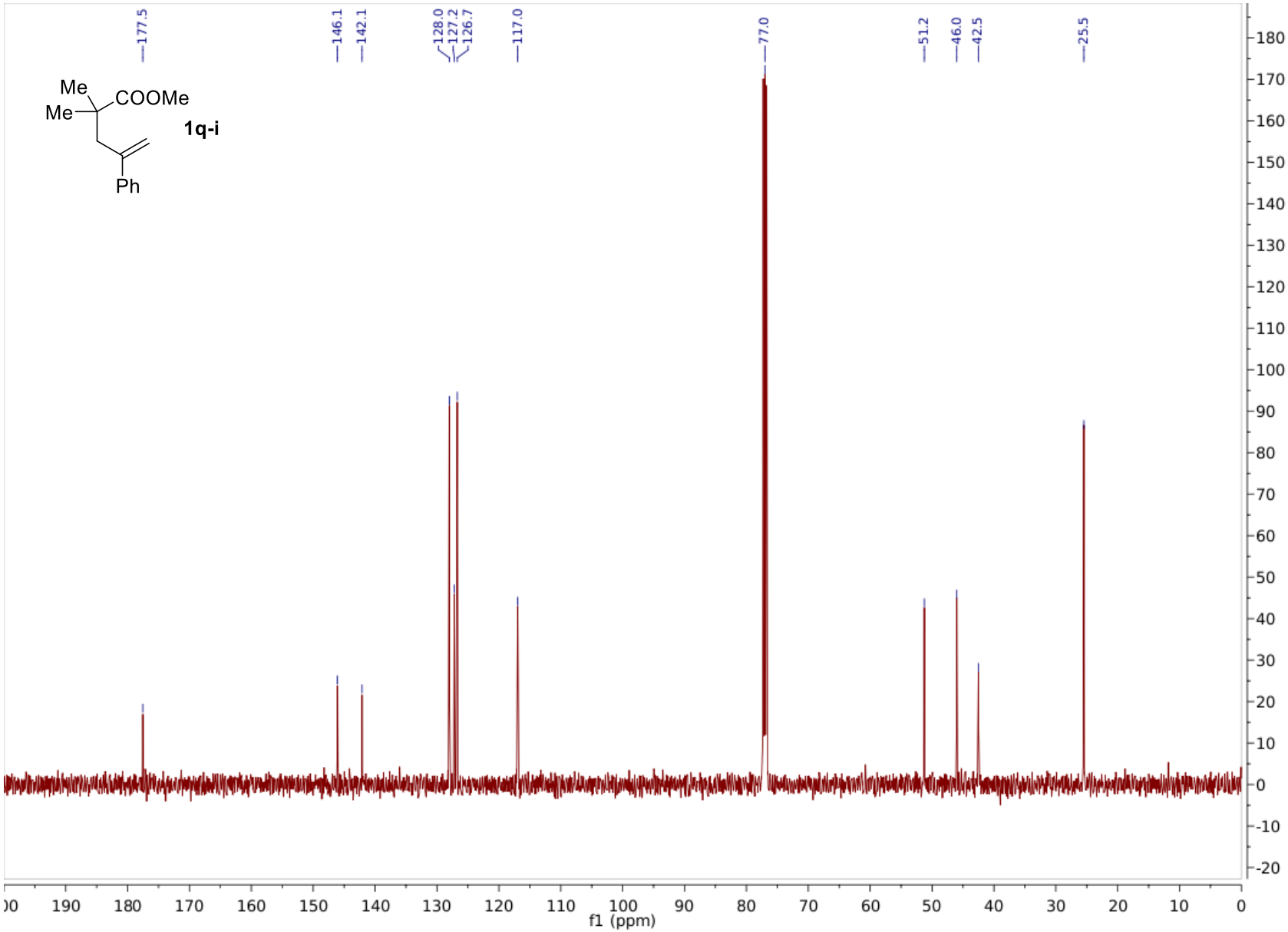

Ph
Ph
COOMe
Ph
1p-ii
7.32
7.31
7.30
7.30
7.29
7.28
7.28
7.26
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.21
7.20
7.19
7.19
7.19
7.18
7.17
5.11
4.67
3.66
3.36
15.6
1.0
1.0
1.9
2.9
f1 (ppm)

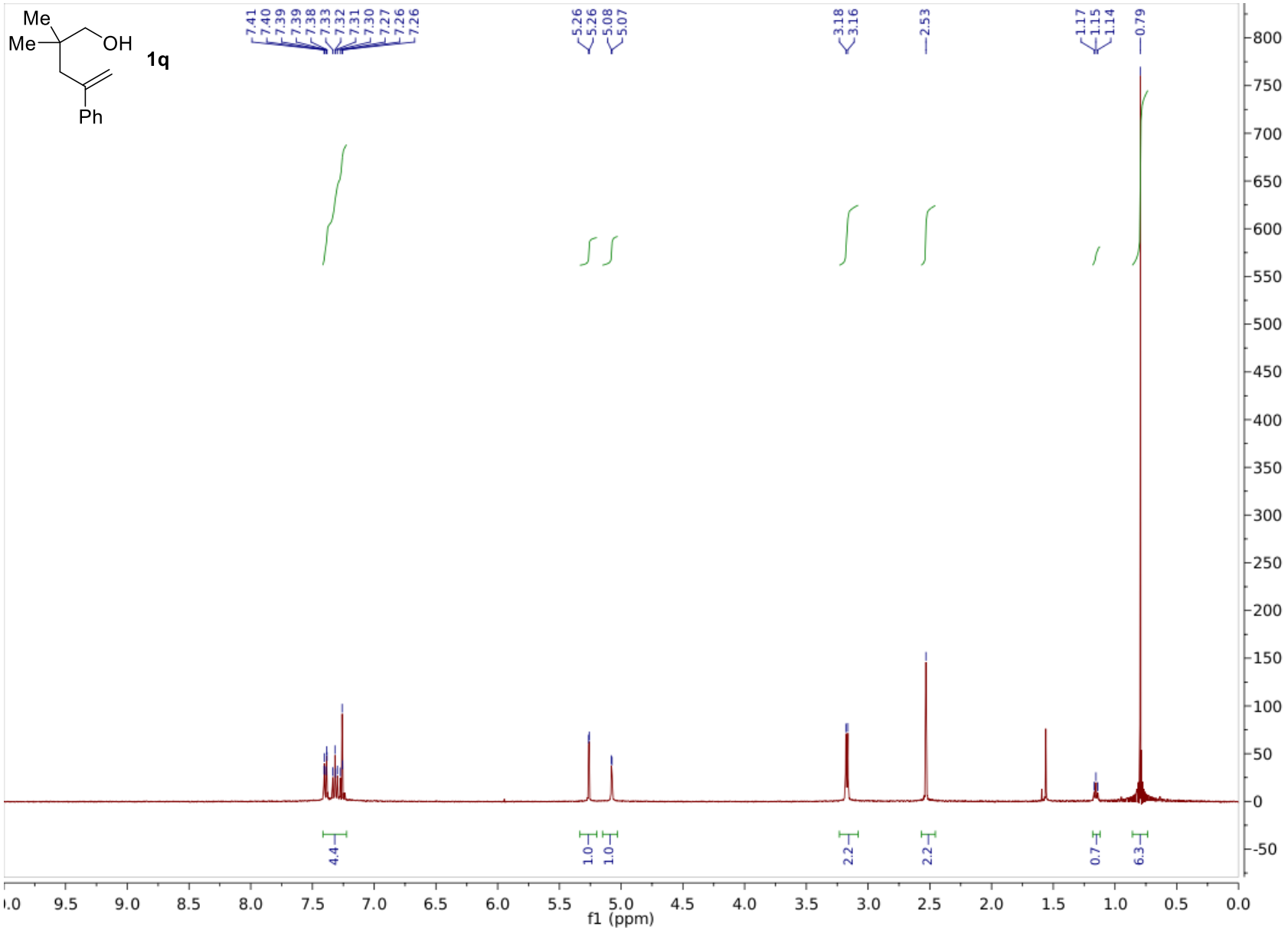

Ph
Ph
COOMe
Ph
1p-ii
173.7
145.0
143.1
142.5
129.0
127.8
127.6
126.9
126.8
126.6
118.3
77.0
60.1
51.7
43.6
f1 (ppm)

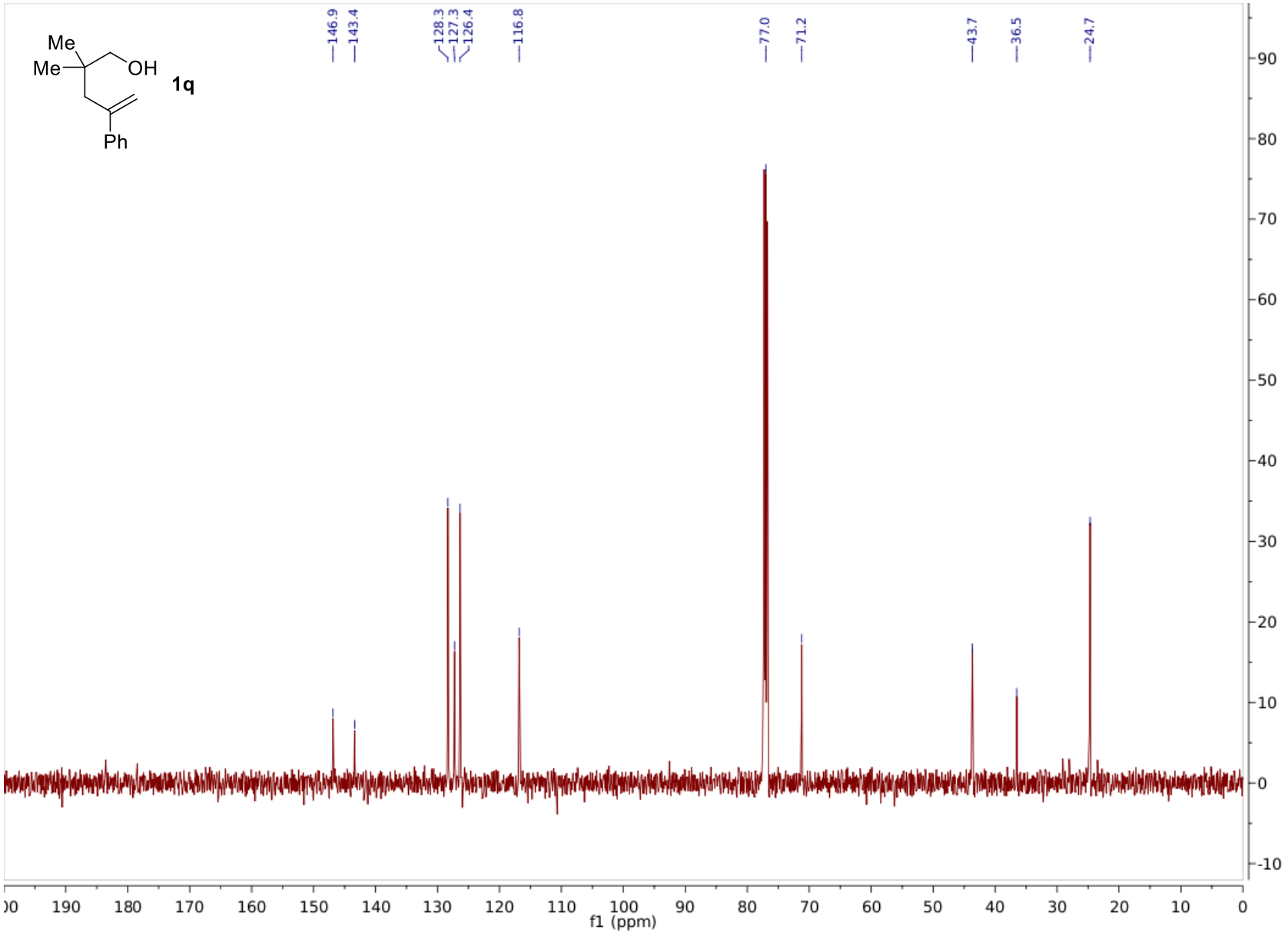

Ph
Ph
OH
Ph
1p
7.26
7.26
7.24
7.24
7.23
7.22
7.22
7.20
7.20
7.19
7.19
7.18
7.18
7.17
7.16
7.15
7.15
7.13
7.13
7.12
7.12
7.11
5.09
4.72
4.02
4.00
3.47
1.03
1.02
1.00
15.0
1.0
1.0
2.1
2.2
0.8
f1 (ppm)

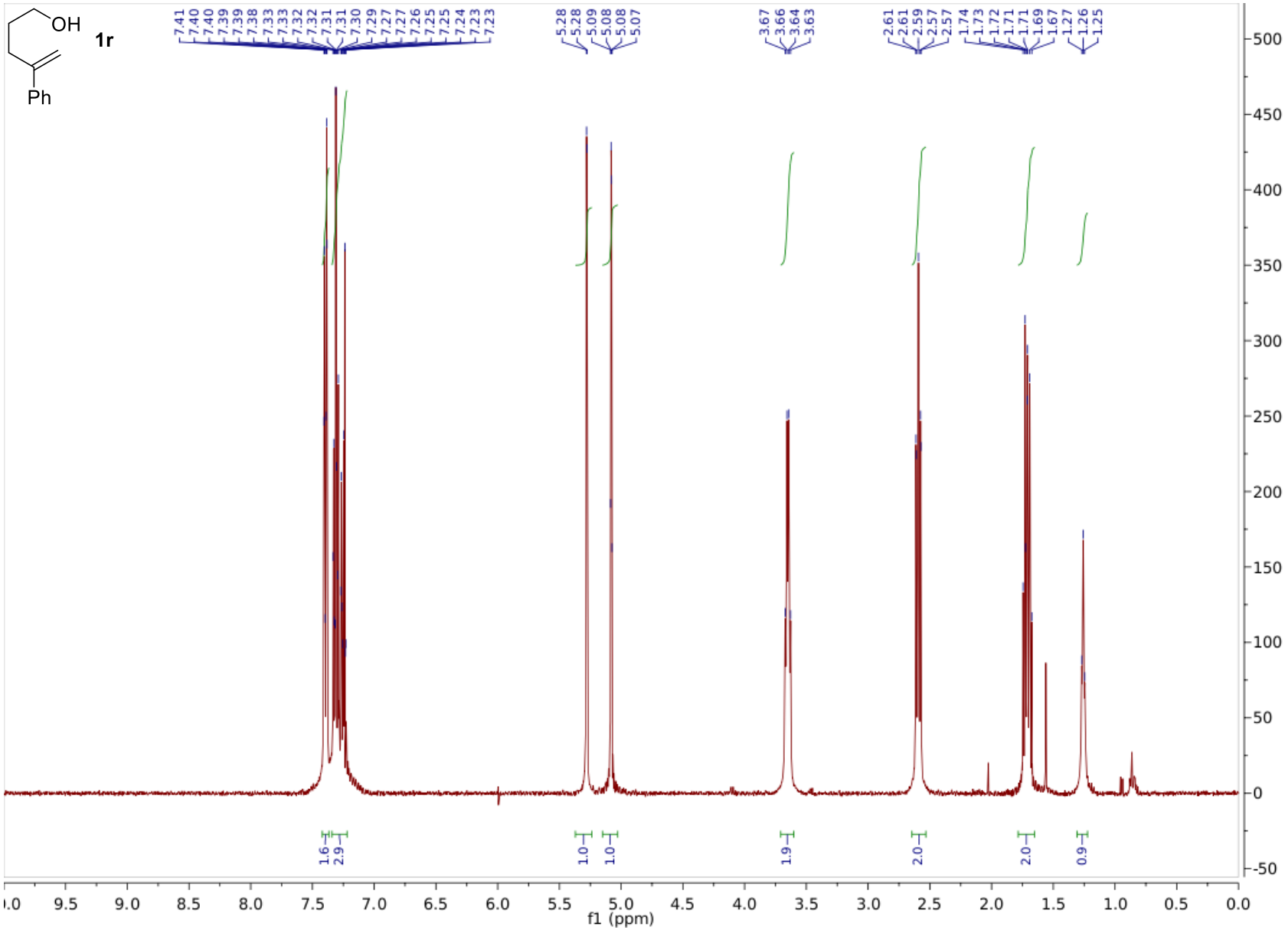

Ph
Ph
OH
Ph
1p
145.6
145.5
142.9
128.4
127.9
126.8
126.4
126.2
118.4
77.0
66.8
52.6
41.0
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

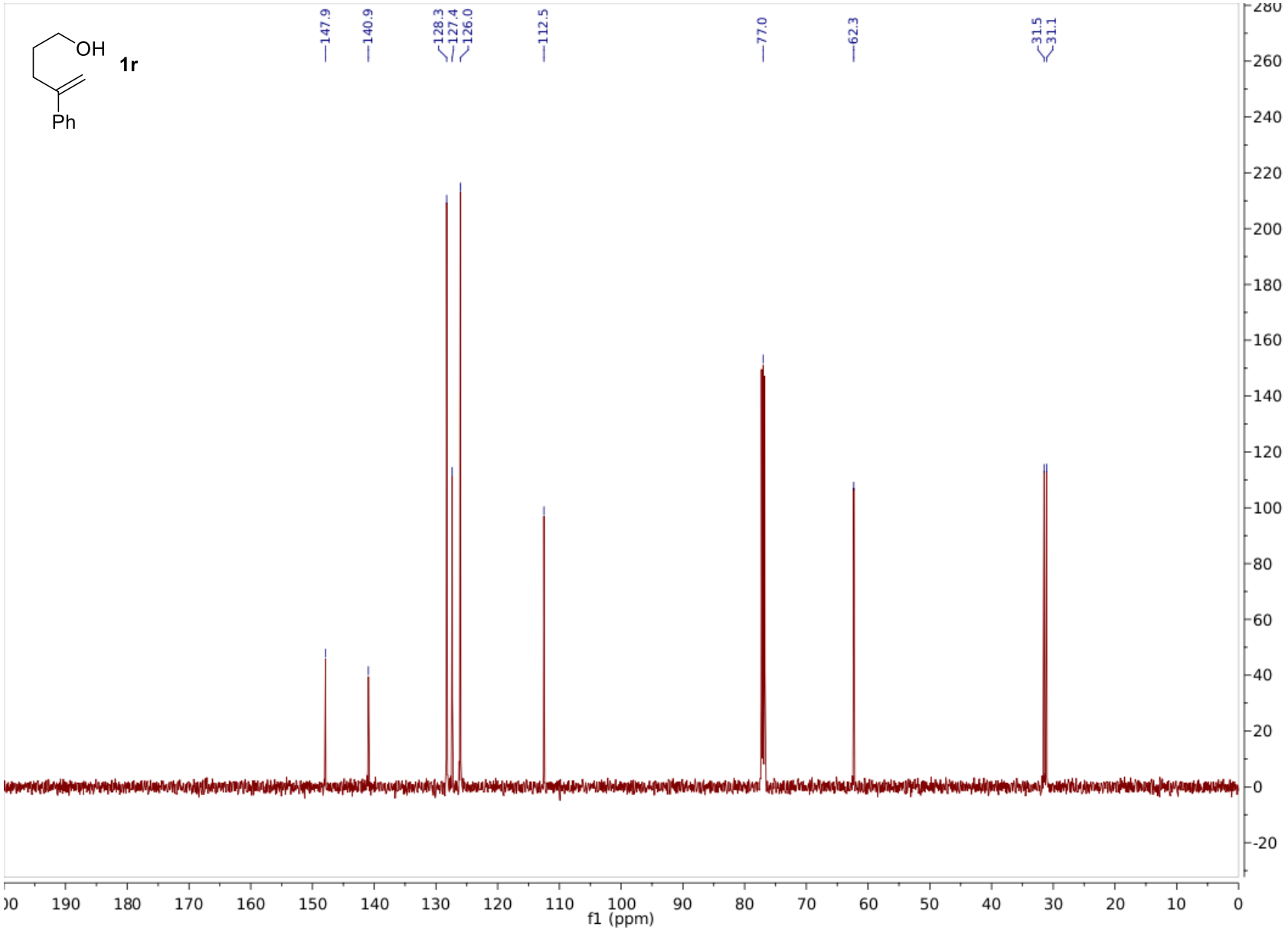

Me
COOMe
Me
Ph
1q-i
7.32
7.32
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.24
7.23
7.23
7.22
7.21
7.21
7.20
5.21
5.21
5.03
5.03
3.27
2.77
1.11
3.8
0.9
1.0
2.5
2.0
6.5
f1 (ppm)

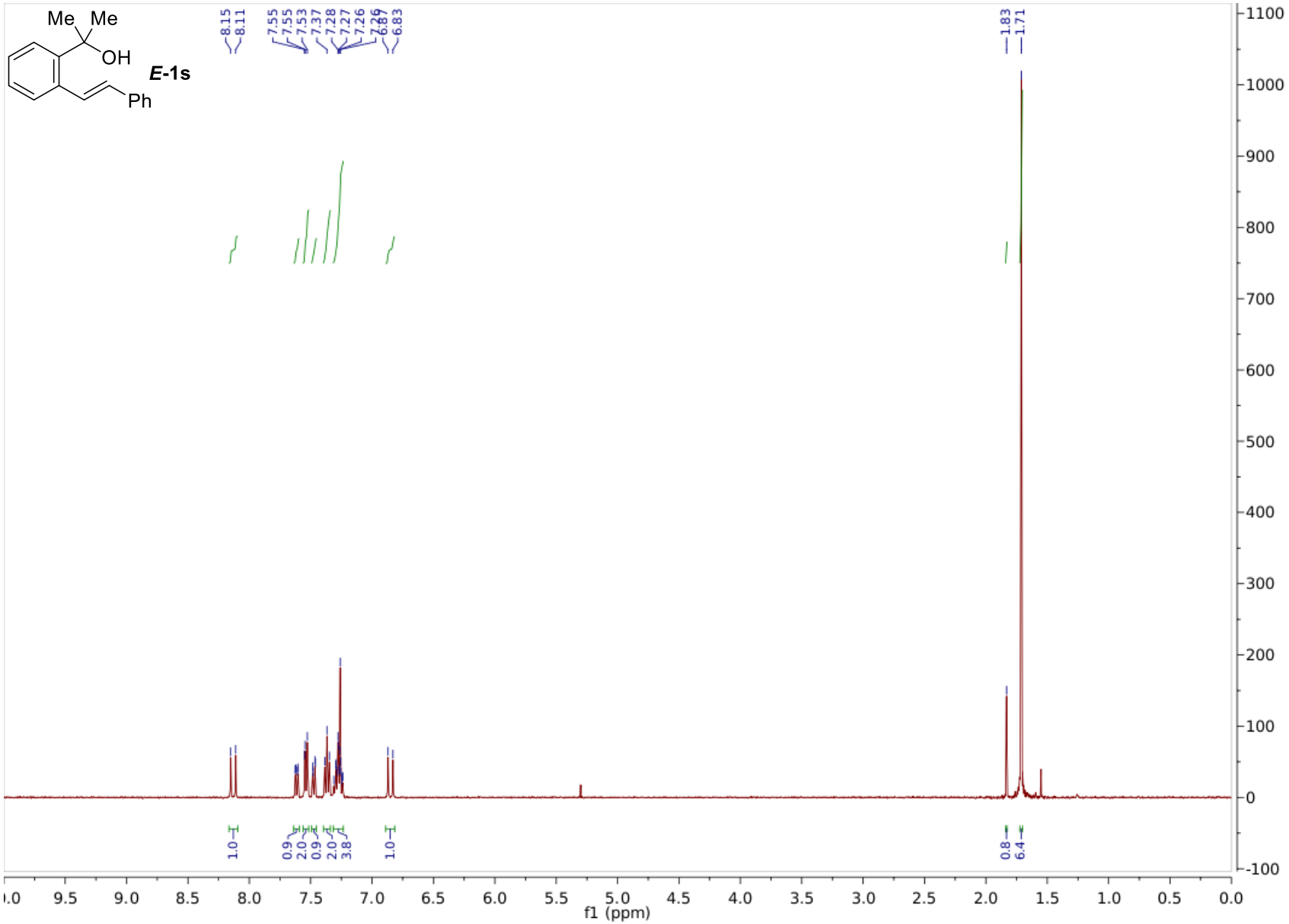

177.5
146.1
142.1
128.0
127.2
126.7
117.0
77.0
51.2
46.0
42.5
25.5
Me
Me
COOMe
Ph
1q-i
f1 (ppm)

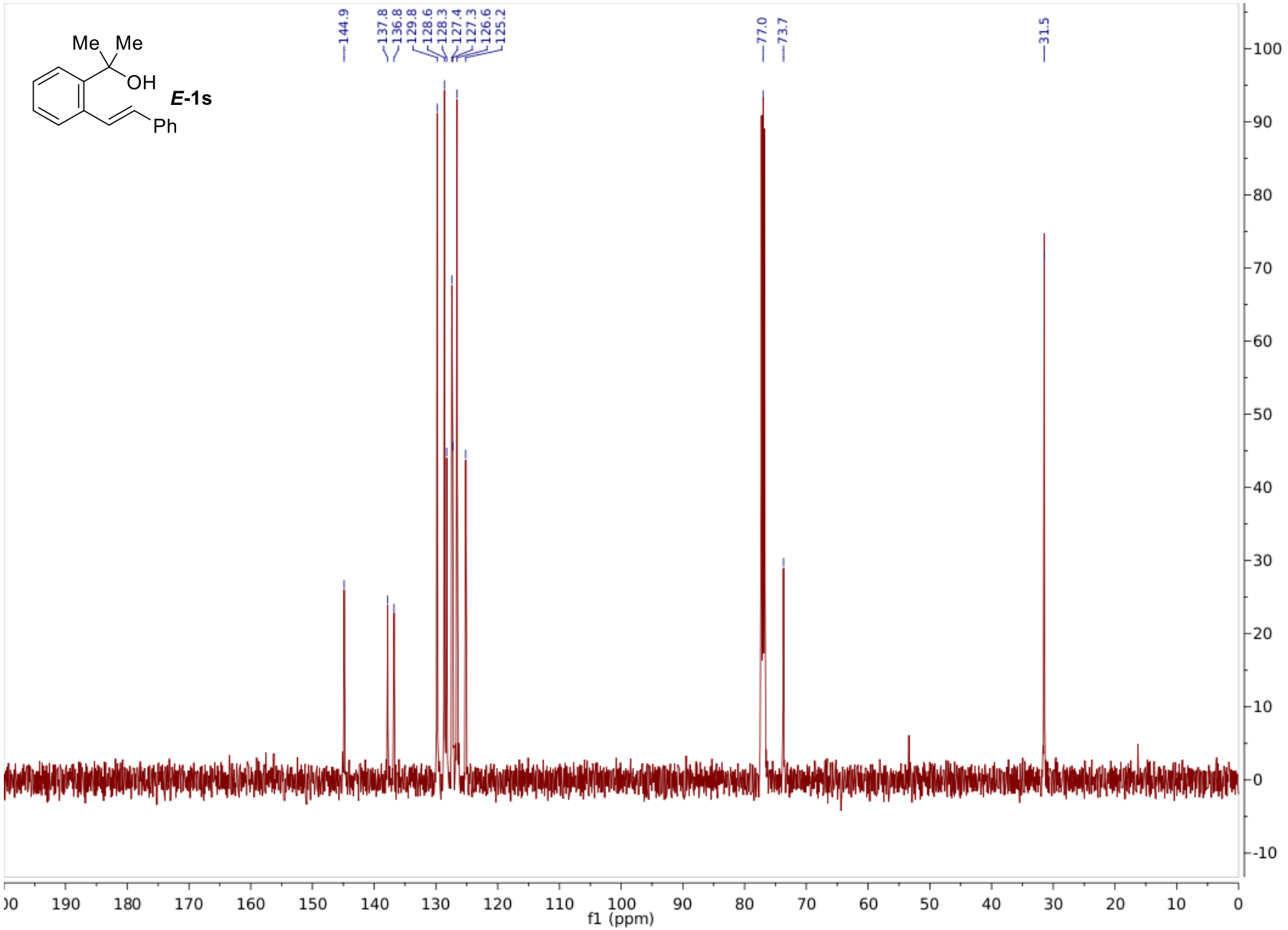

Me
Me
OH
Ph
1q
7.41
7.40
7.39
7.39
7.38
7.33
7.32
7.31
7.30
7.27
7.26
7.26
5.26
5.26
5.08
5.07
3.18
3.16
2.53
1.17
1.15
1.14
0.79
4.4
1.0
1.0
2.2
2.2
0.7
6.3
f1 (ppm)

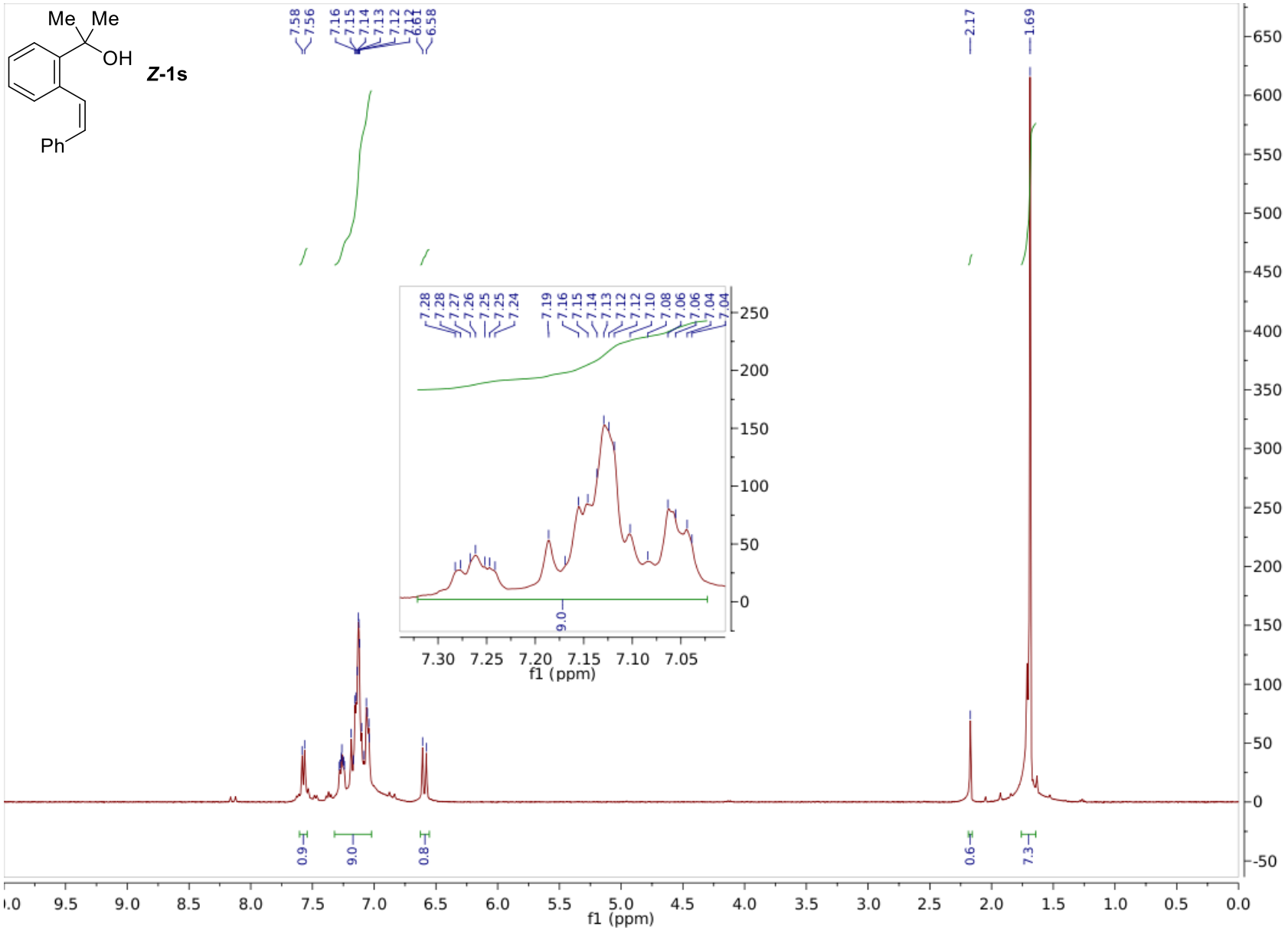

Me
Me
OH
Ph
1q
146.9
143.4
128.3
127.3
126.4
116.8
77.0
71.2
43.7
36.5
24.7
f1 (ppm)

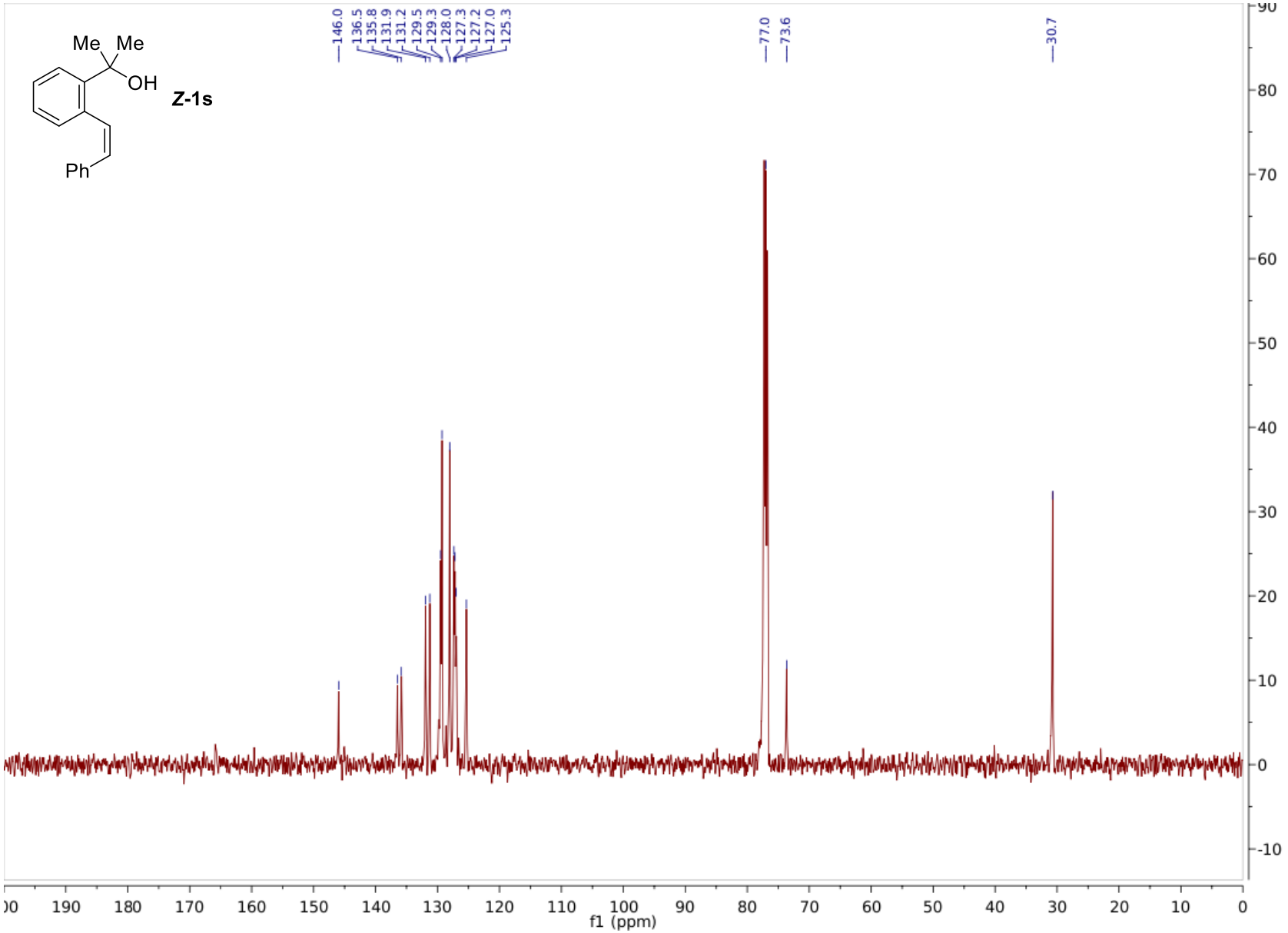

OH
Ph
1r
7.41 7.40 7.40 7.39 7.39 7.38 7.33 7.33 7.32 7.32 7.31 7.31 7.30 7.29 7.27 7.27 7.26 7.25 7.25 7.24 7.23 7.23
5.28 5.28 5.09 5.08 5.08 5.07
3.67 3.66 3.64 3.63
2.61 2.61 2.59 2.57 2.57
1.74 1.73 1.72 1.71 1.71 1.69 1.67
1.27 1.26 1.25
1.6 2.9 1.0 1.0 1.9 2.0 2.0 0.9
f1 (ppm)

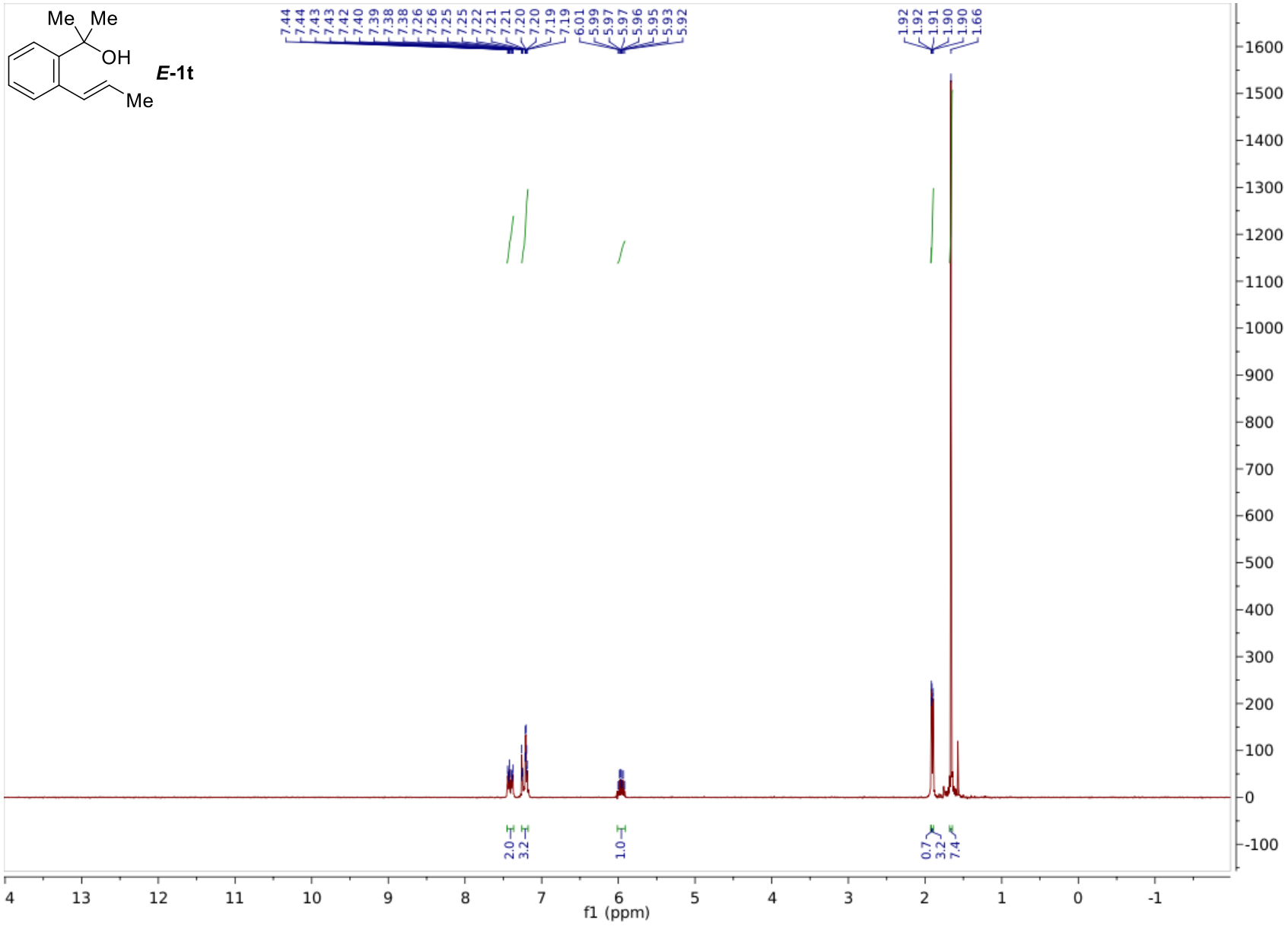

OH
1r
Ph
147.9
140.9
128.3
127.4
126.0
112.5
77.0
62.3
31.5
31.1
f1 (ppm)

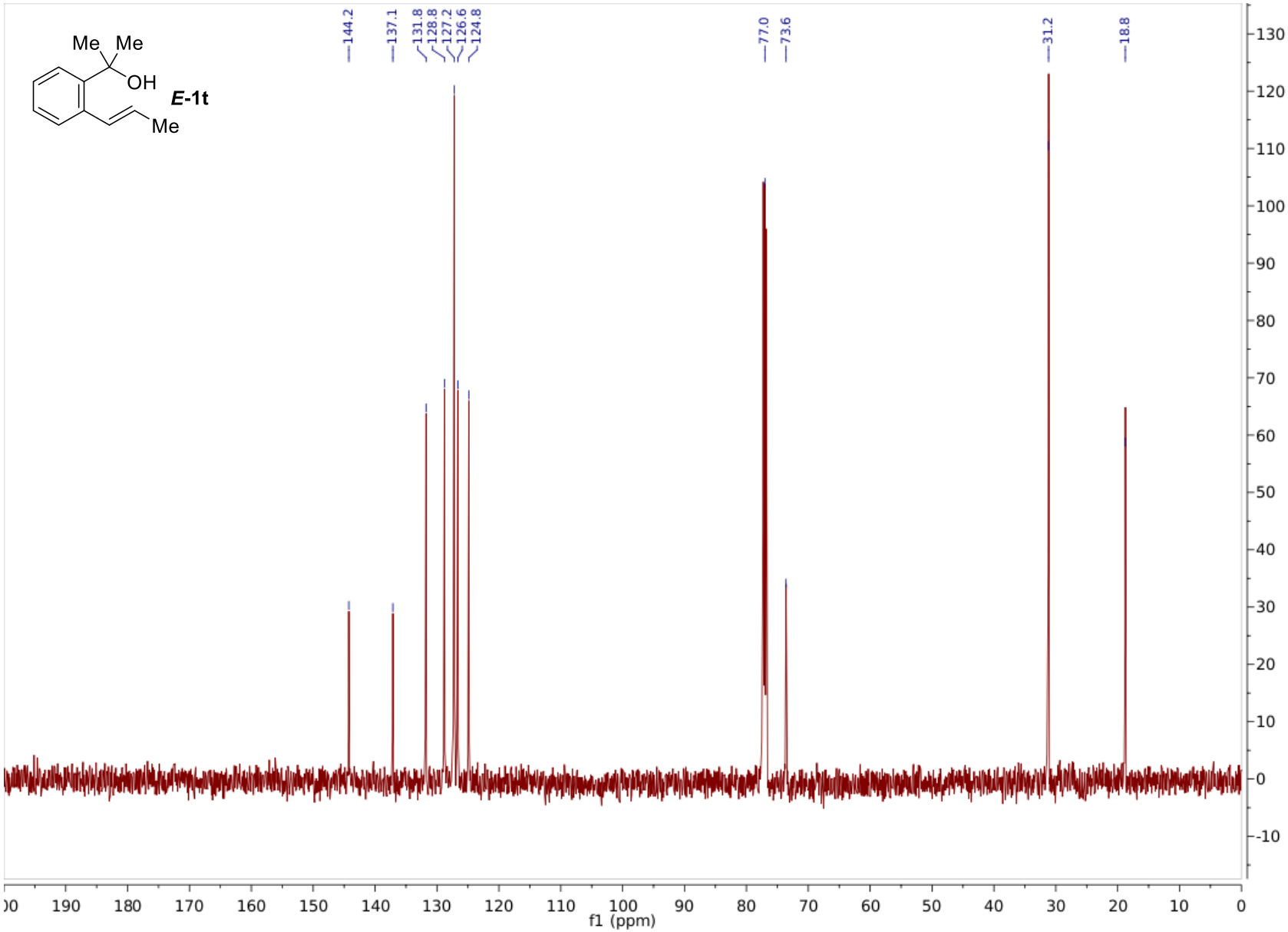

Me Me
OH
Ph
E-1s
8.15
8.11
7.55
7.55
7.53
7.37
7.28
7.27
7.26
6.87
6.83
1.83
1.71
1.0
0.9
2.0
0.9
2.0
3.8
1.0
0.8
6.4
f1 (ppm)

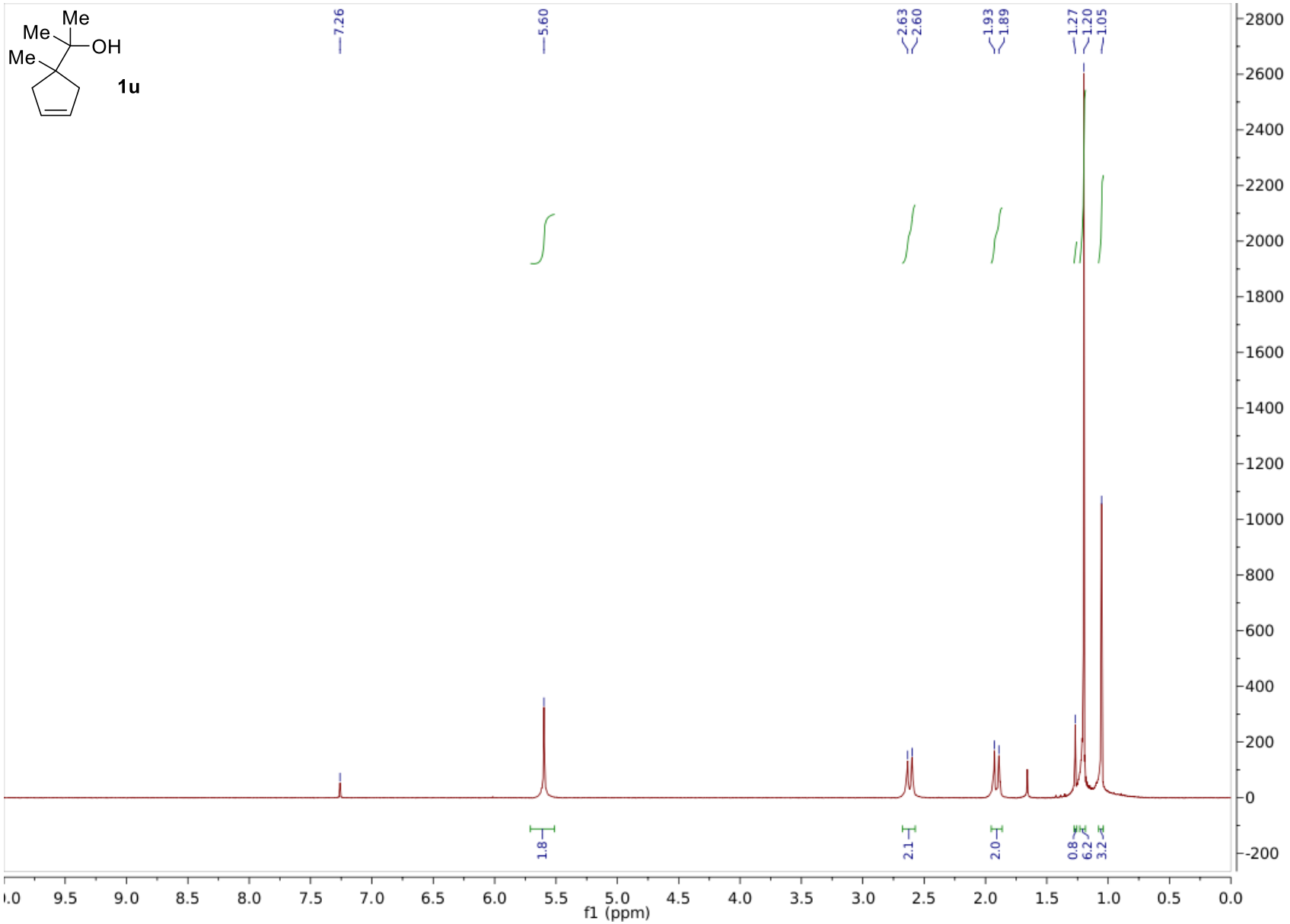

Me
Me
OH
Ph
E-1s
144.9
137.8
136.8
129.8
128.6
128.3
127.4
127.3
126.6
125.2
77.0
73.7
31.5
f1 (ppm)

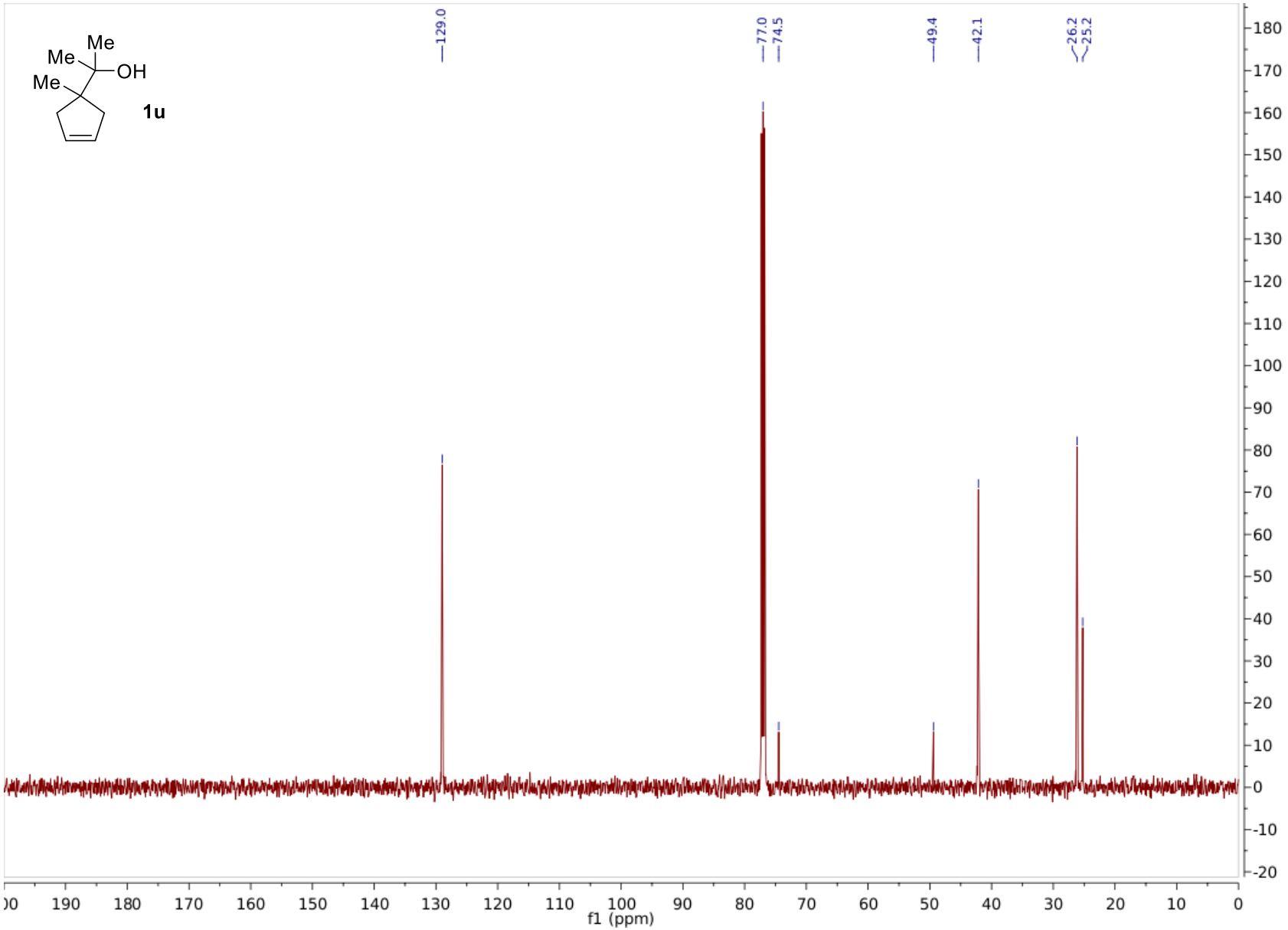

Me
Me
OH
Ph
Z-1s
7.58
7.56
7.16
7.15
7.14
7.13
7.12
6.61
6.58
2.17
1.69
7.28
7.27
7.26
7.25
7.24
7.19
7.16
7.15
7.14
7.13
7.12
7.10
7.08
7.06
7.04
9.0
7.30 7.25 7.20 7.15 7.10 7.05
f1 (ppm)
0.9
9.0
0.8
0.6
7.3
f1 (ppm)

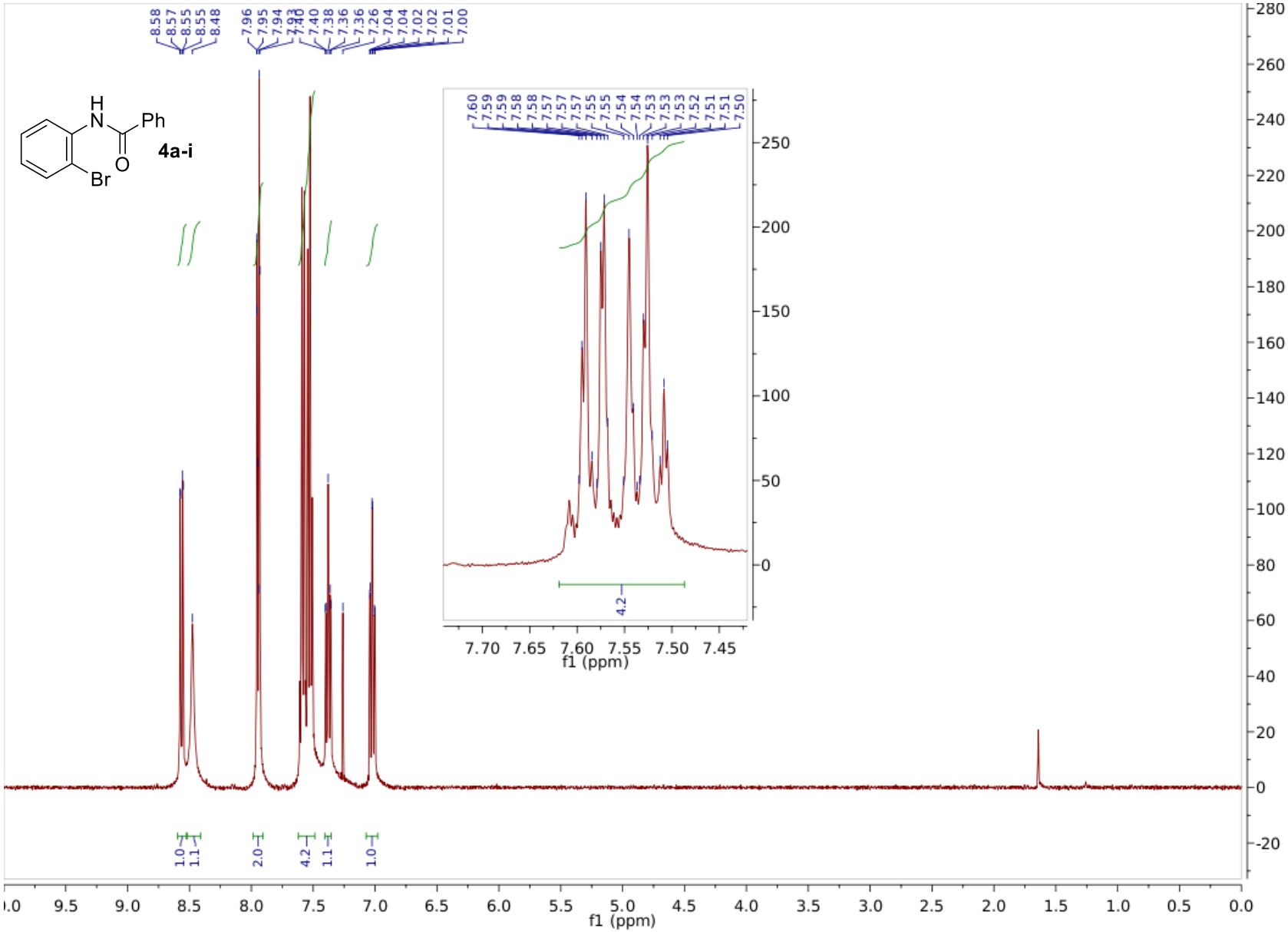

Me
Me
OH
Ph
Z-1s
146.0
136.5
135.8
131.9
131.2
129.5
129.3
128.0
127.3
127.2
127.0
125.3
77.0
73.6
30.7
f1 (ppm)

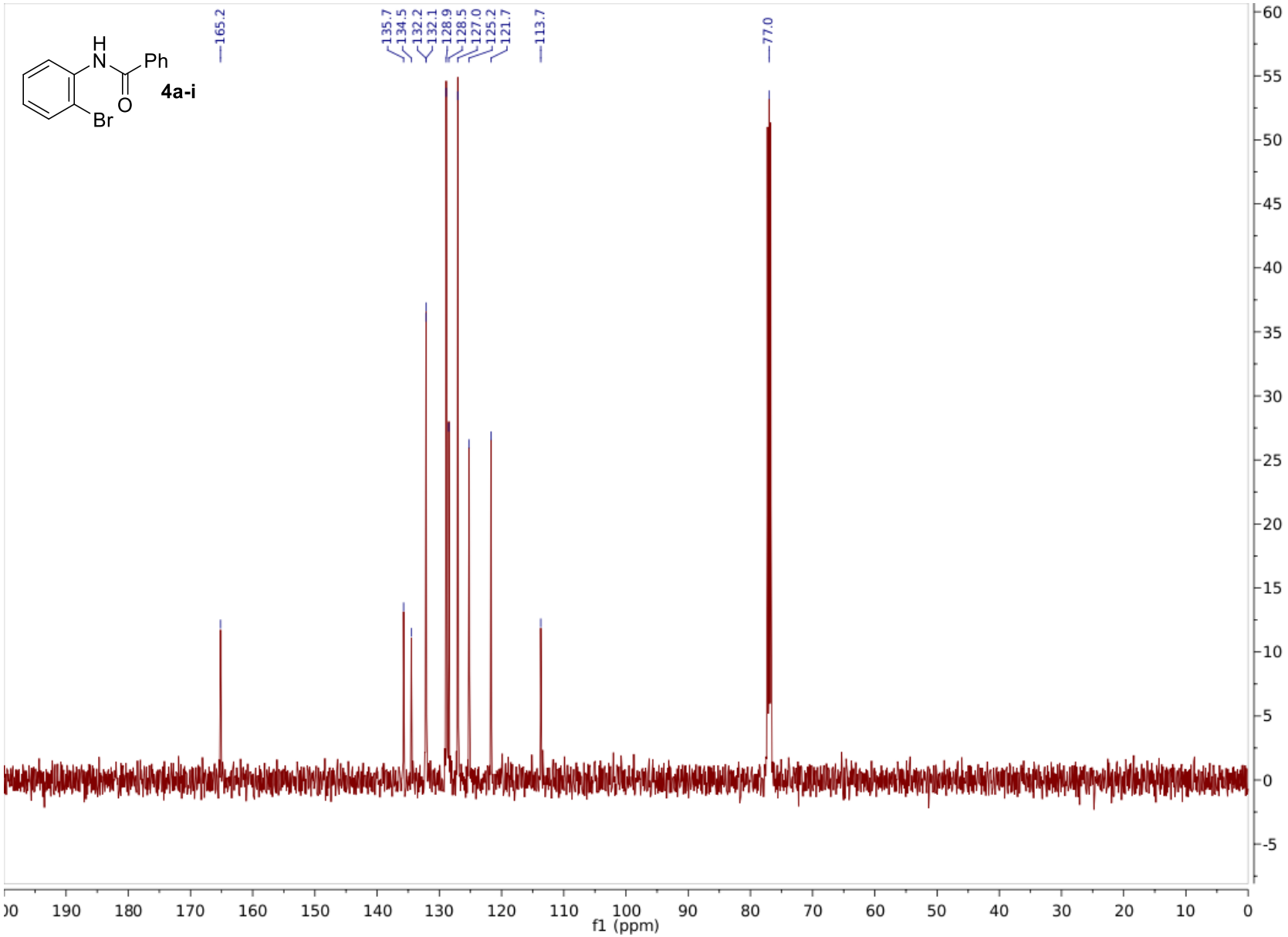

Me Me
OH
Me
E-1t
7.44
7.44
7.43
7.43
7.42
7.40
7.39
7.38
7.38
7.26
7.26
7.25
7.25
7.22
7.21
7.21
7.20
7.20
7.19
7.19
6.01
5.99
5.97
5.97
5.96
5.95
5.93
5.92
1.92
1.92
1.91
1.90
1.90
1.66
2.0
3.2
1.0
0.7
3.2
7.4
f1 (ppm)

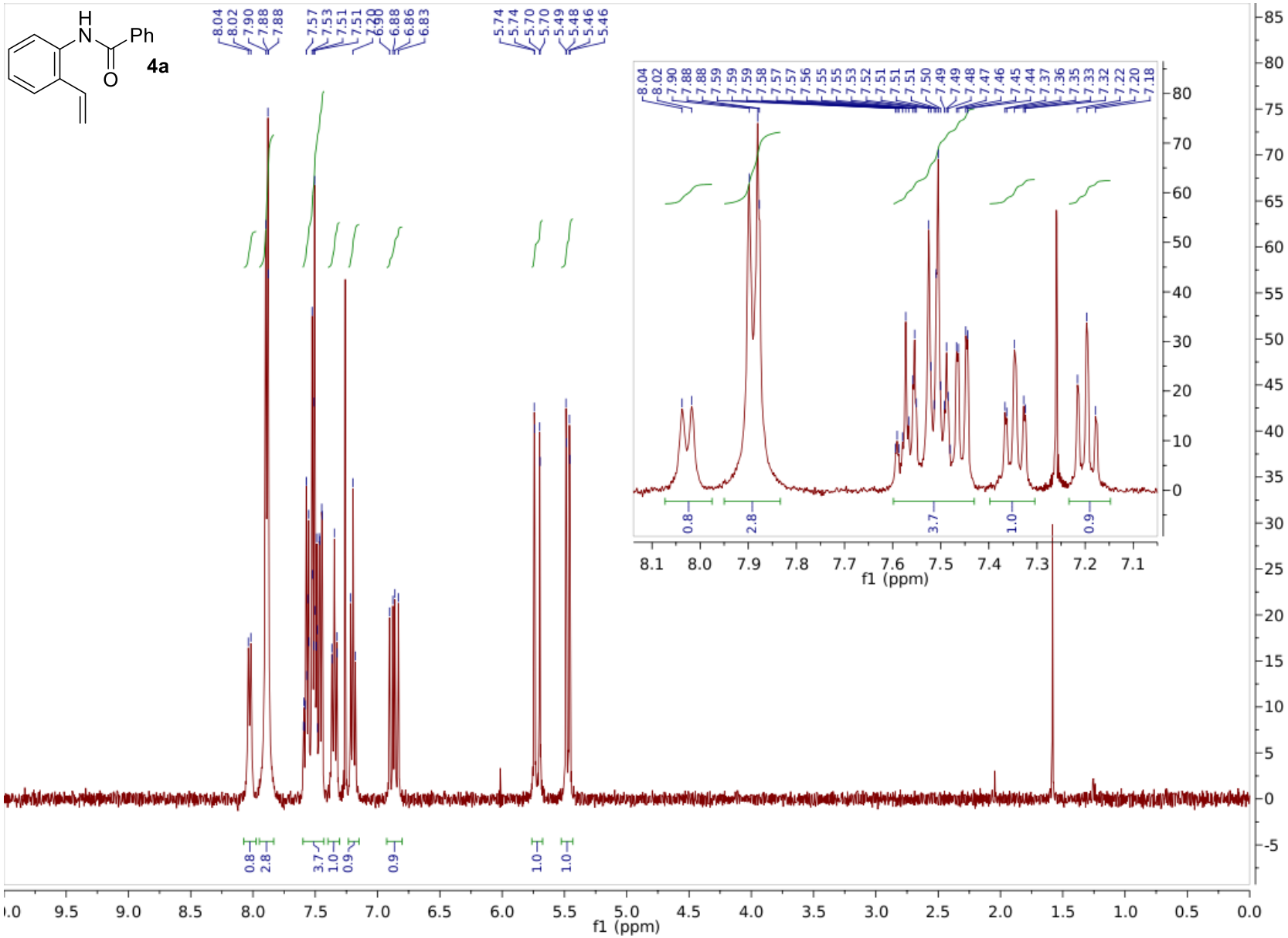

Me Me
OH
Me
E-1t
144.2
137.1
131.8
128.8
127.2
126.6
124.8
77.0
73.6
31.2
18.8
f1 (ppm)

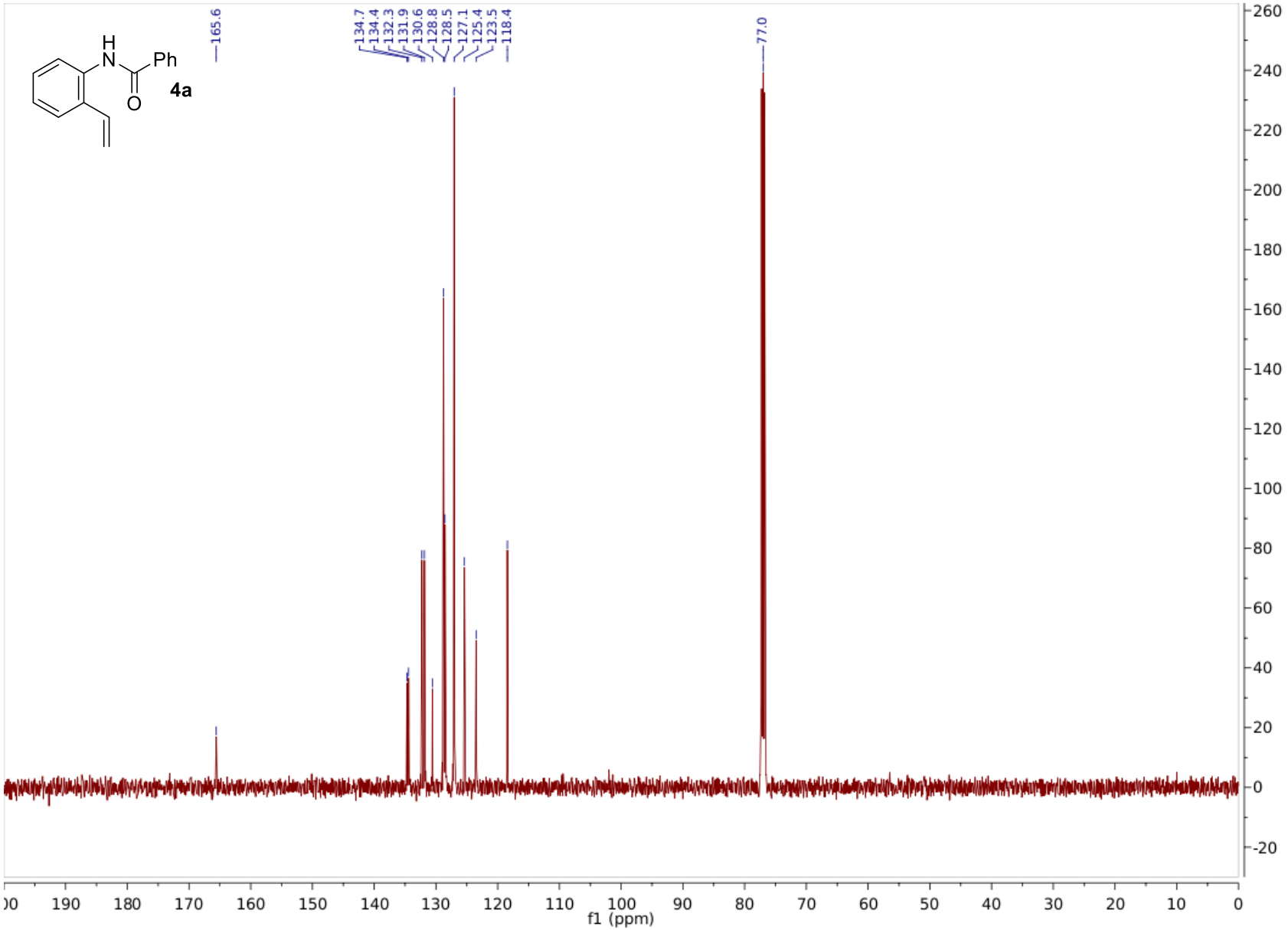

Me
Me
Me
OH
1u
7.26
5.60
2.63
2.60
1.93
1.89
1.27
1.20
1.05
1.8
2.1
2.0
0.8
6.2
3.2
f1 (ppm)

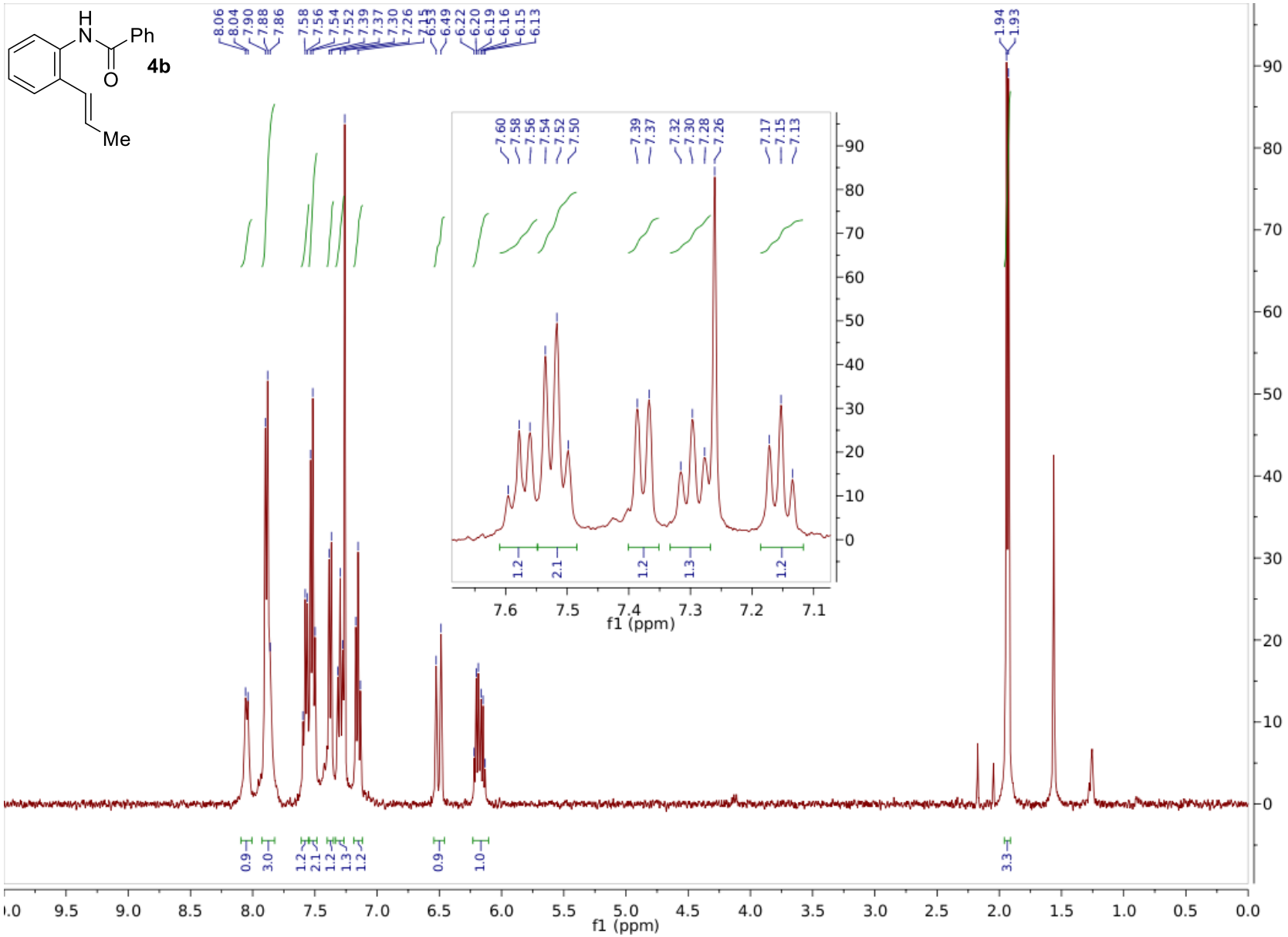

Me
Me
Me
OH
1u
129.0
77.0
74.5
49.4
42.1
26.2
25.2
f1 (ppm)

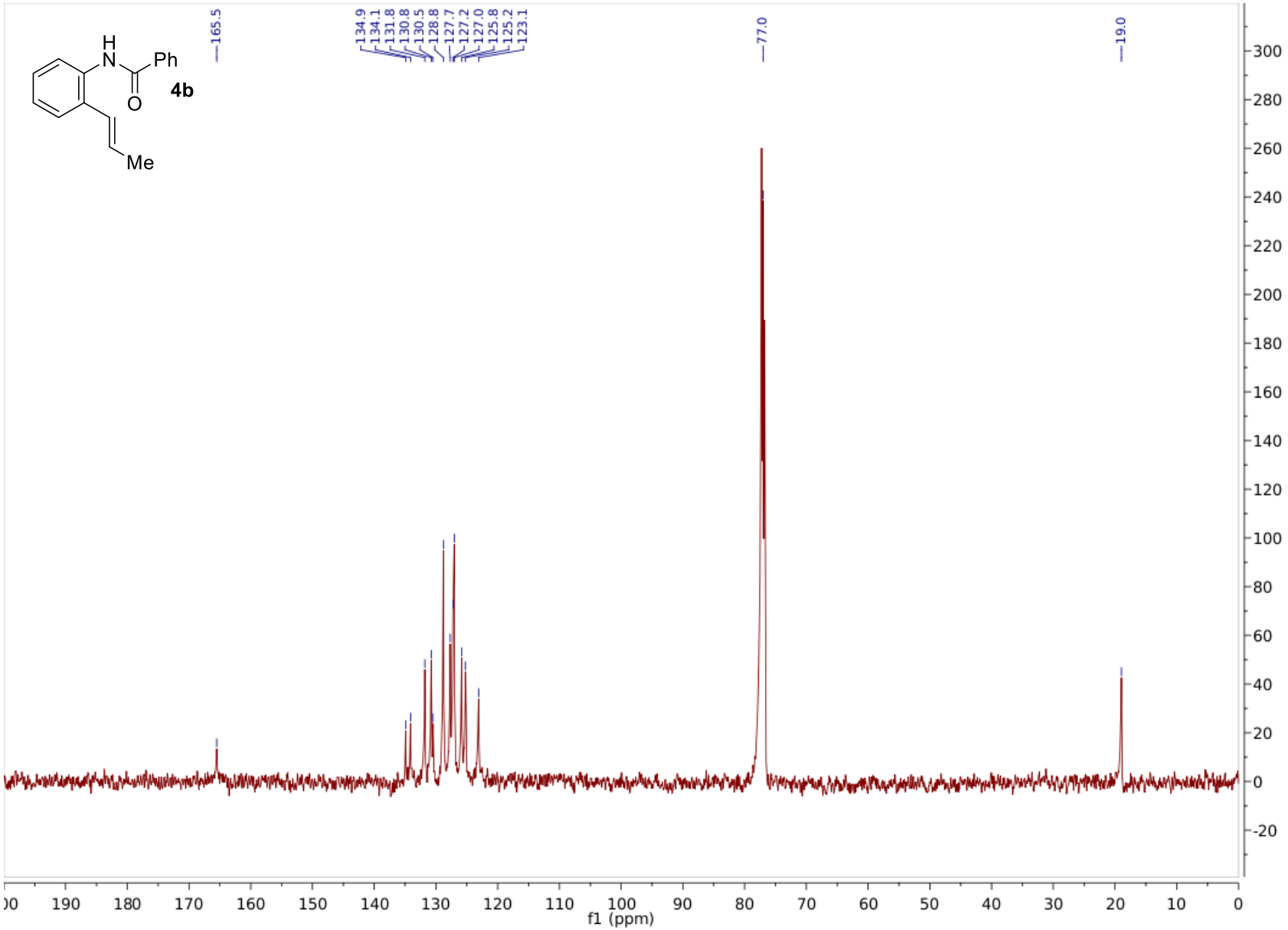

4a-i
f1 (ppm)

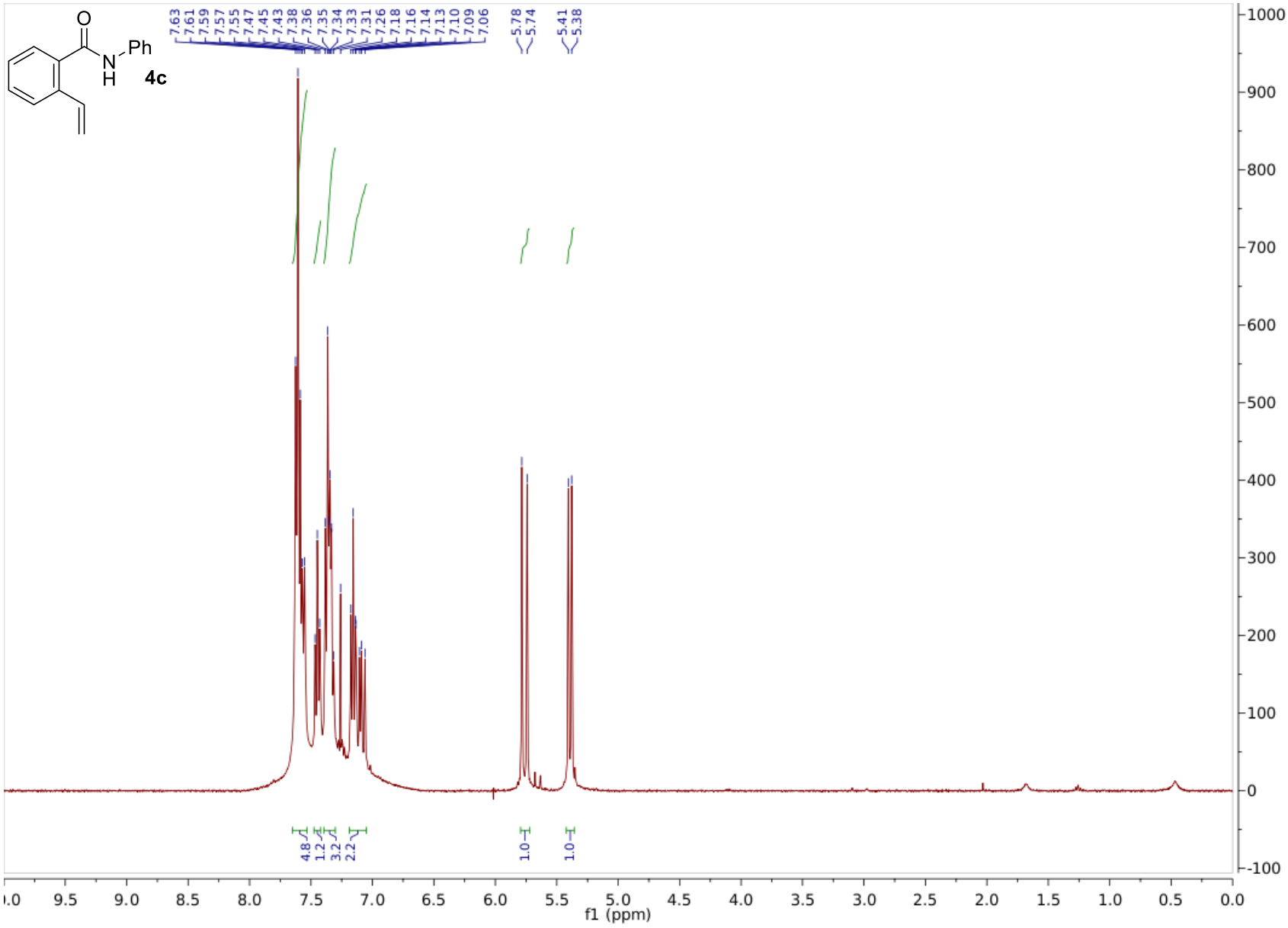

H
N
Ph
O
Br
4a-i
165.2
135.7
134.5
132.2
132.1
128.9
128.5
127.0
125.2
121.7
113.7
77.0
f1 (ppm)

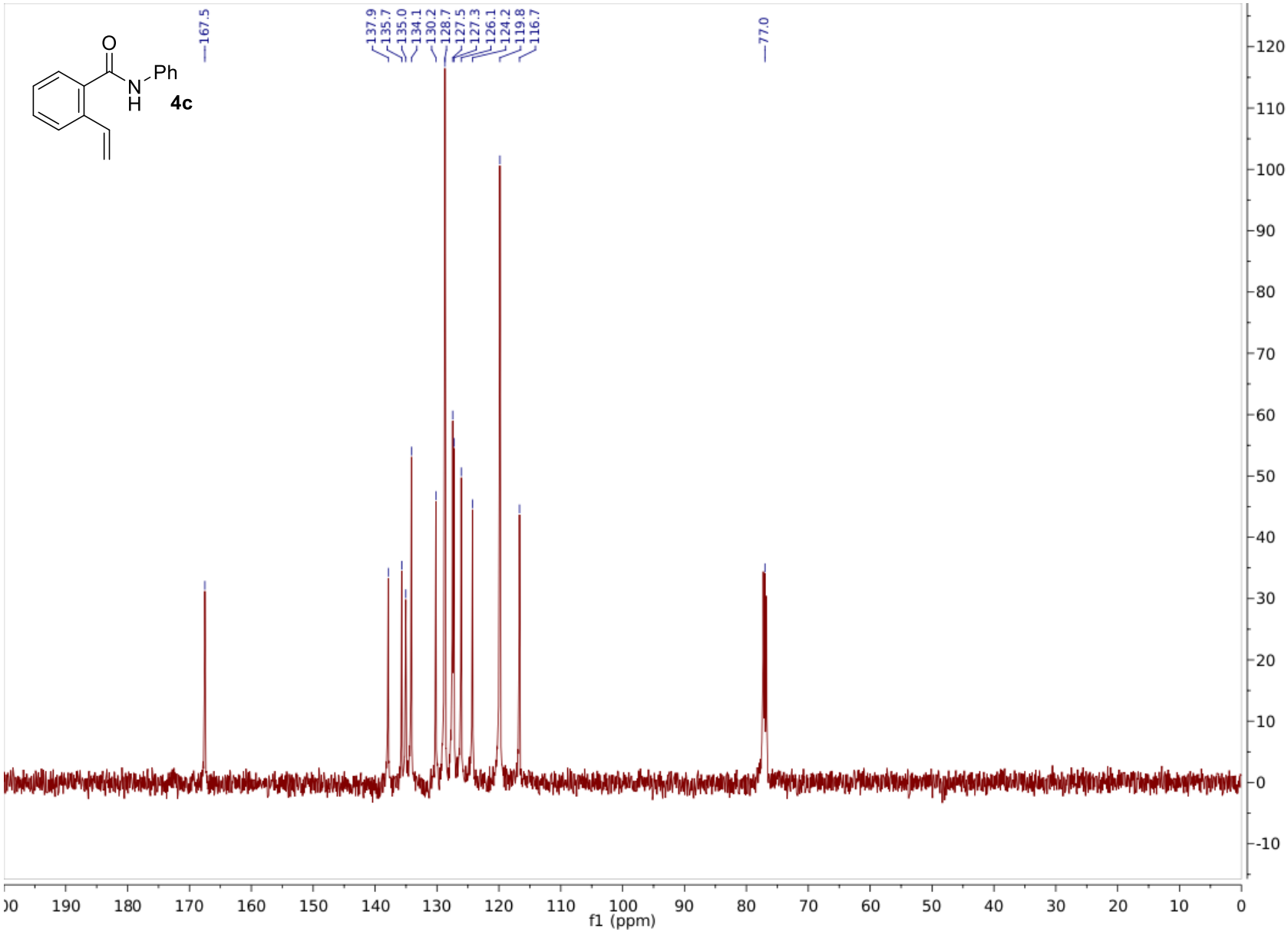

4a
f1 (ppm)

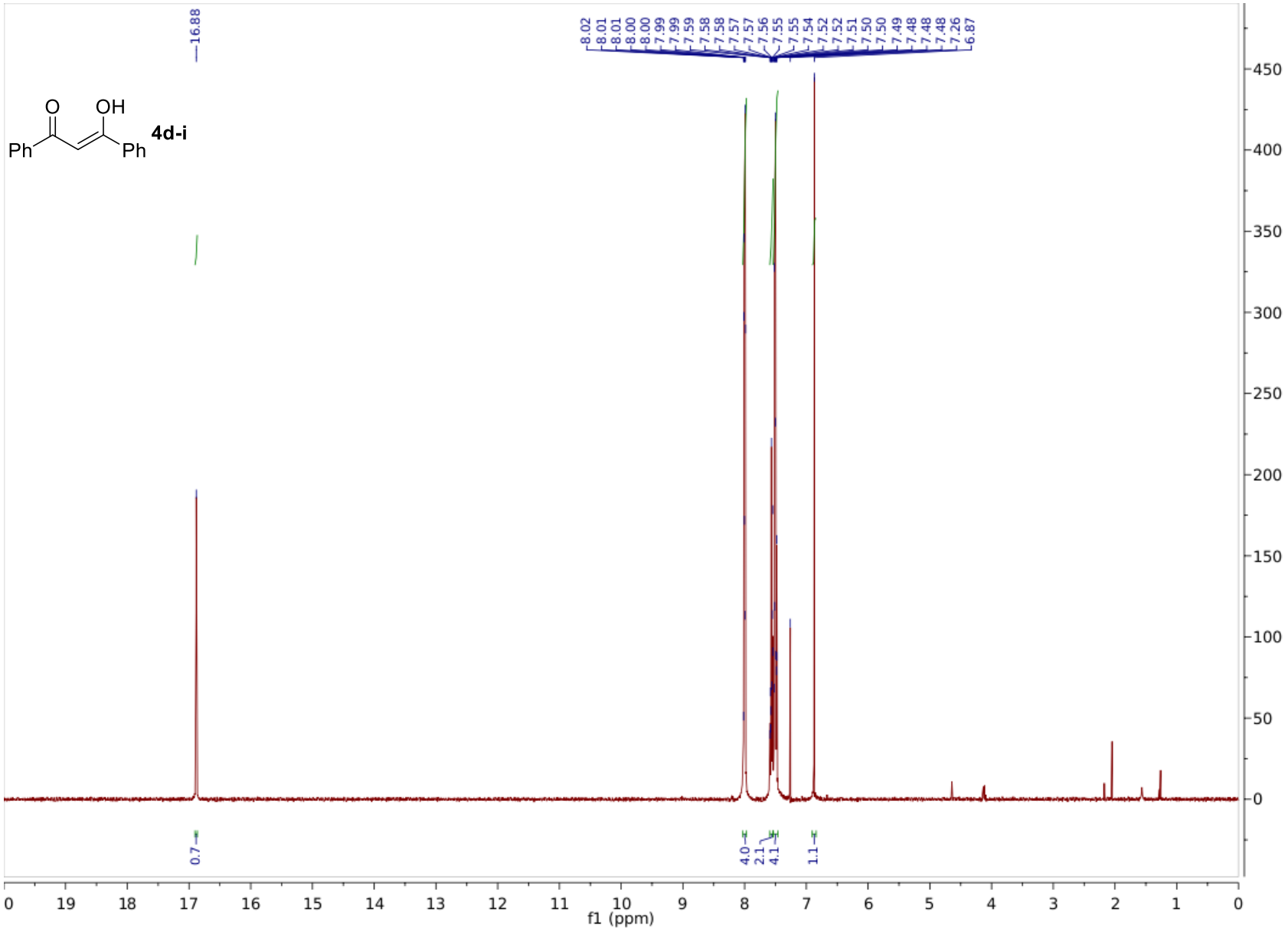

H
N
Ph
O
4a
165.6
134.7
134.4
132.3
131.9
130.6
128.8
128.5
127.1
125.4
123.5
118.4
77.0
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

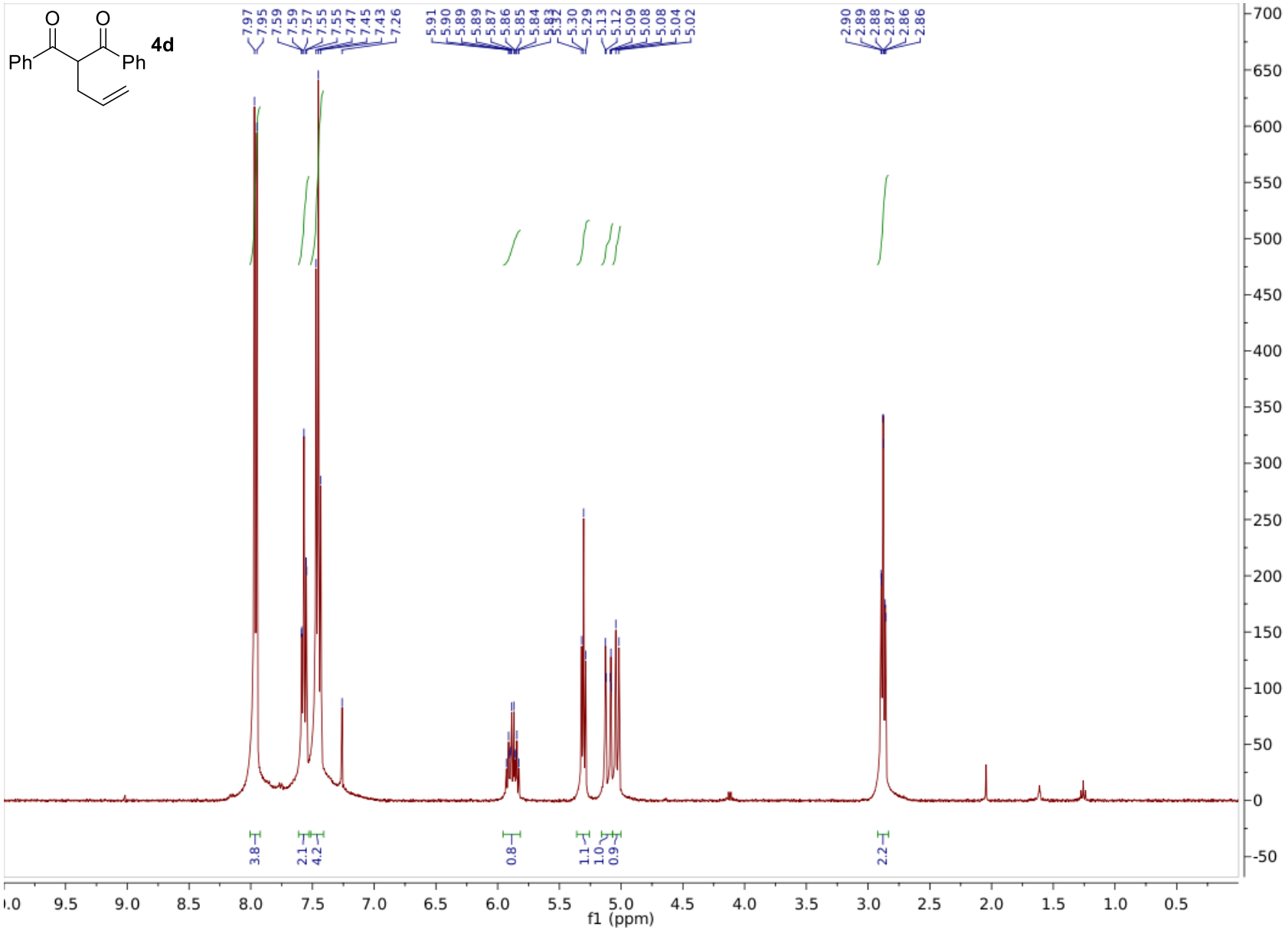

4b
f1 (ppm)

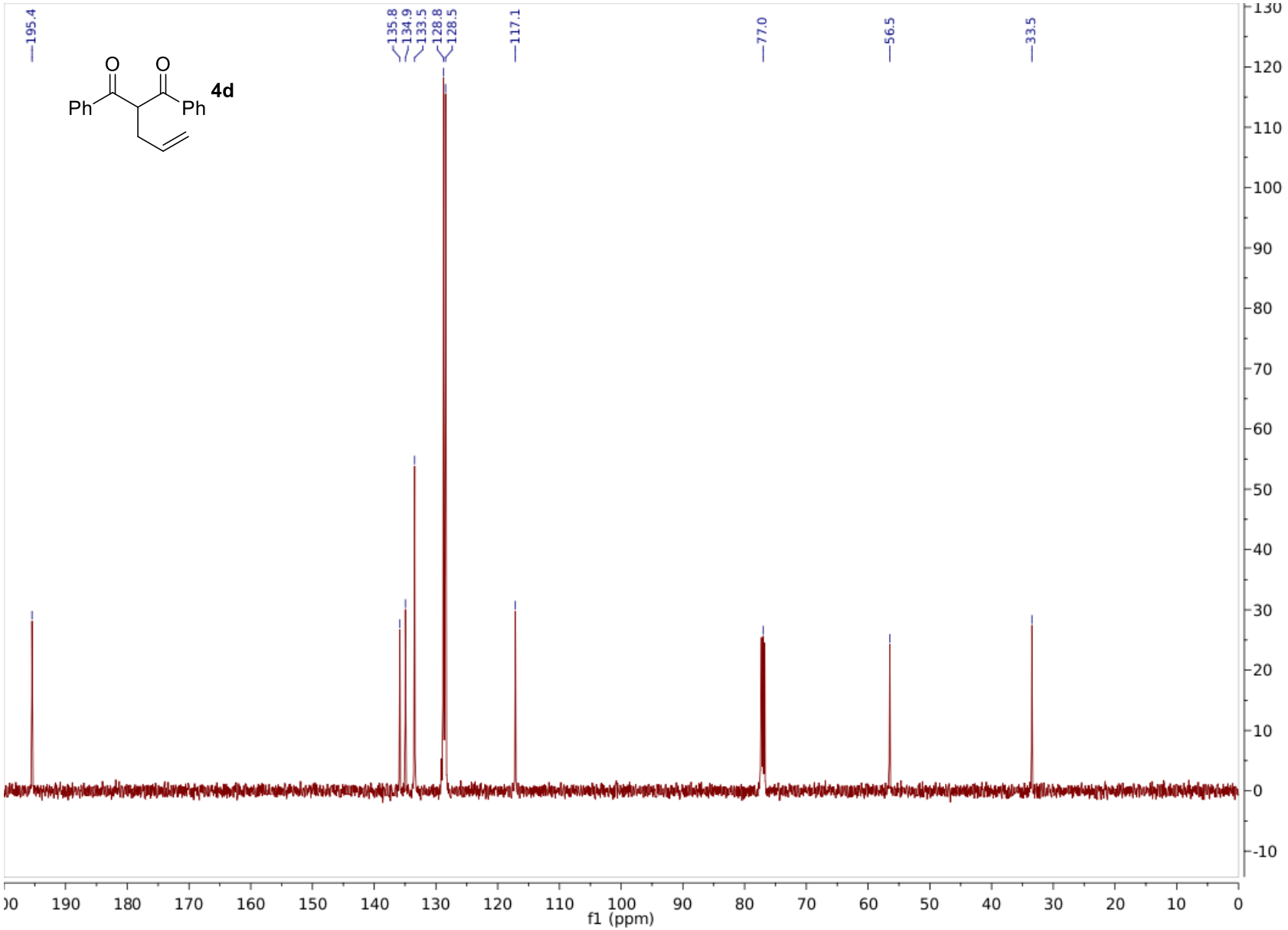

H
N
Ph
O
Me
4b
165.5
134.9
134.1
131.8
130.8
130.5
128.8
127.7
127.2
127.0
125.8
125.2
123.1
77.0
19.0
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

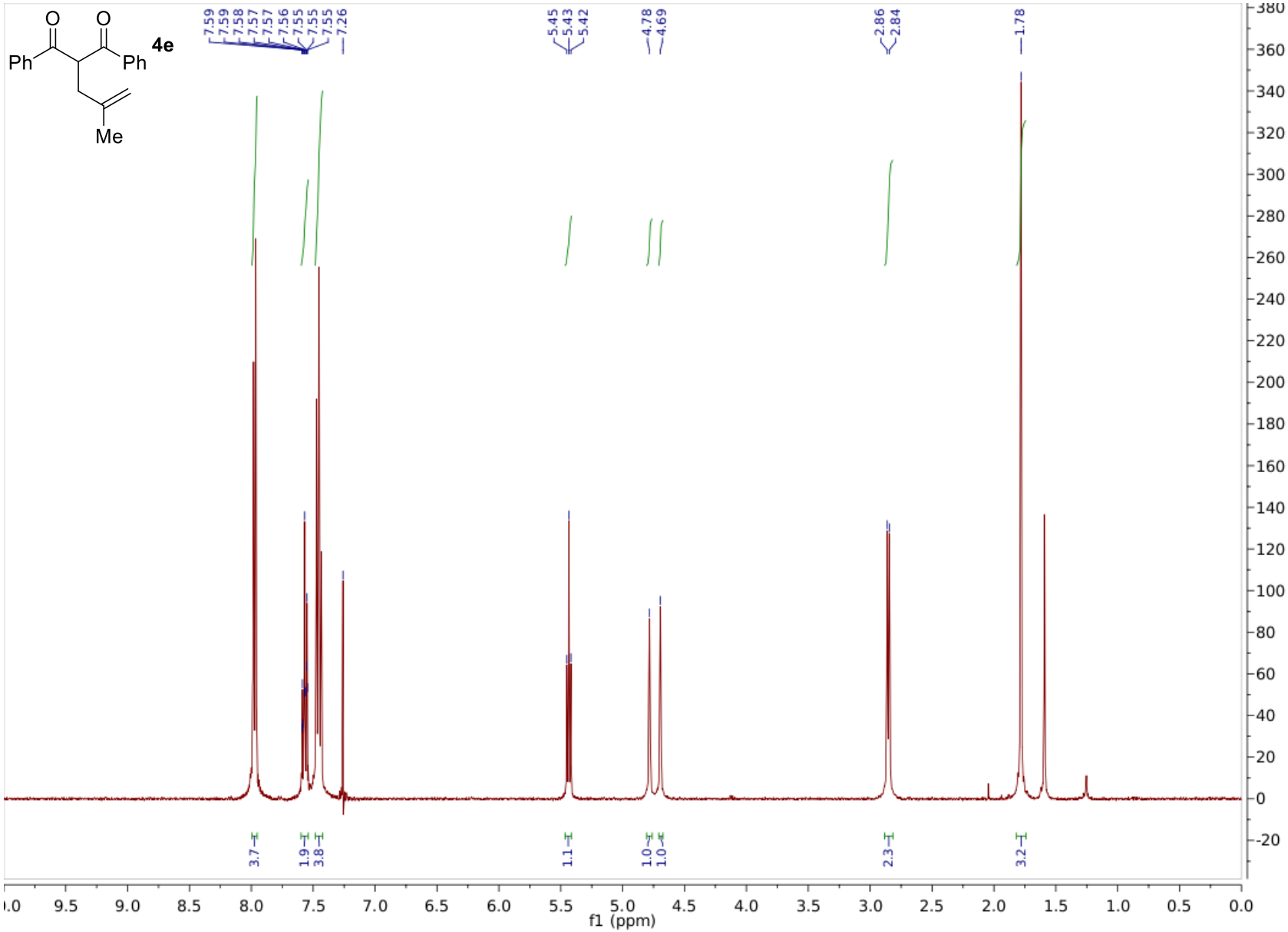

O
N
H
Ph
4c
7.63
7.61
7.59
7.57
7.55
7.47
7.45
7.43
7.38
7.36
7.35
7.34
7.33
7.31
7.26
7.18
7.16
7.14
7.13
7.10
7.09
7.06
5.78
5.74
5.41
5.38
4.8
1.2
3.2
2.2
1.0
1.0
f1 (ppm)

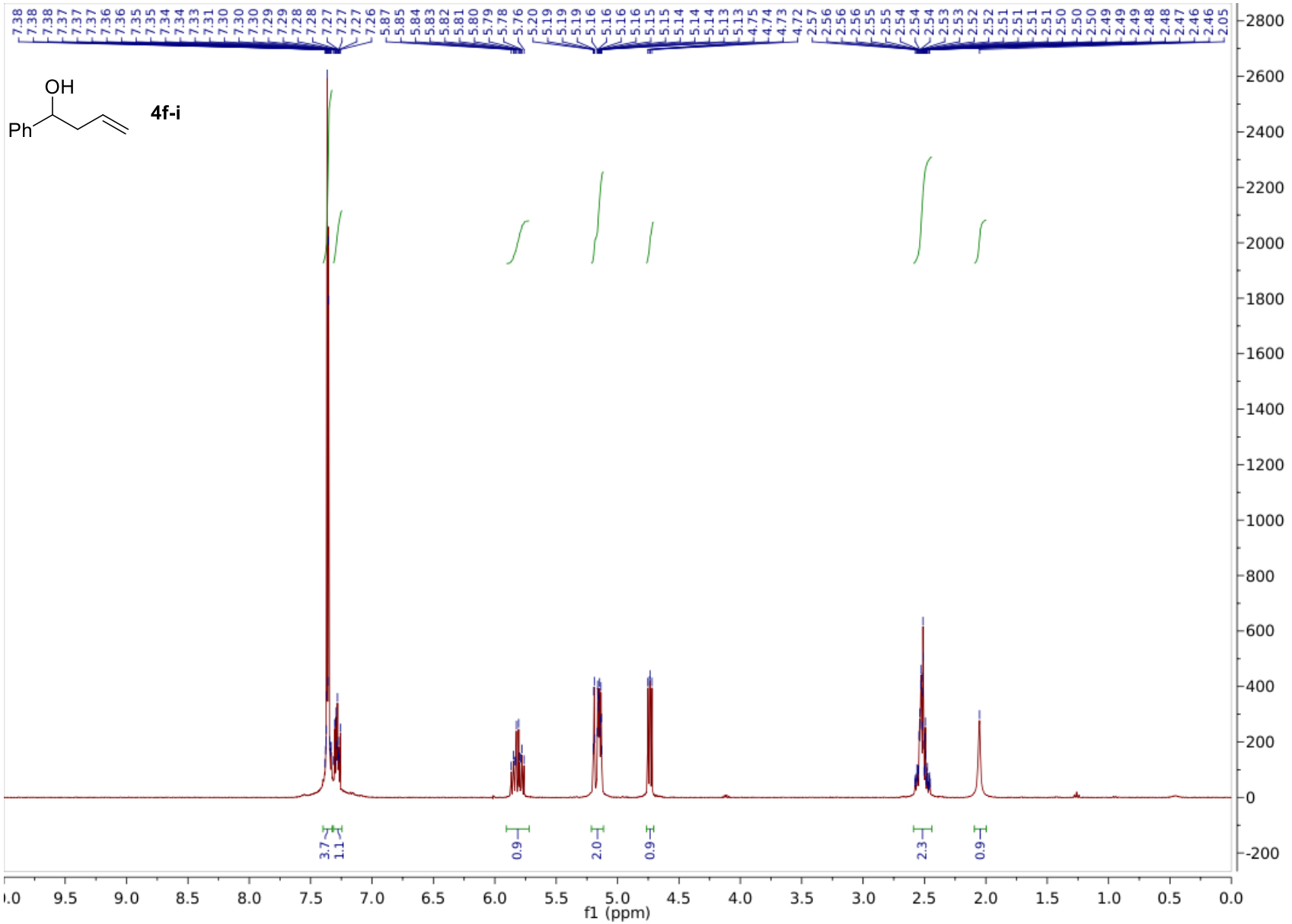

O
N
H
Ph
4c
167.5
137.9
135.7
135.0
134.1
130.2
128.7
127.5
127.3
126.1
124.2
119.8
116.7
77.0
f1 (ppm)

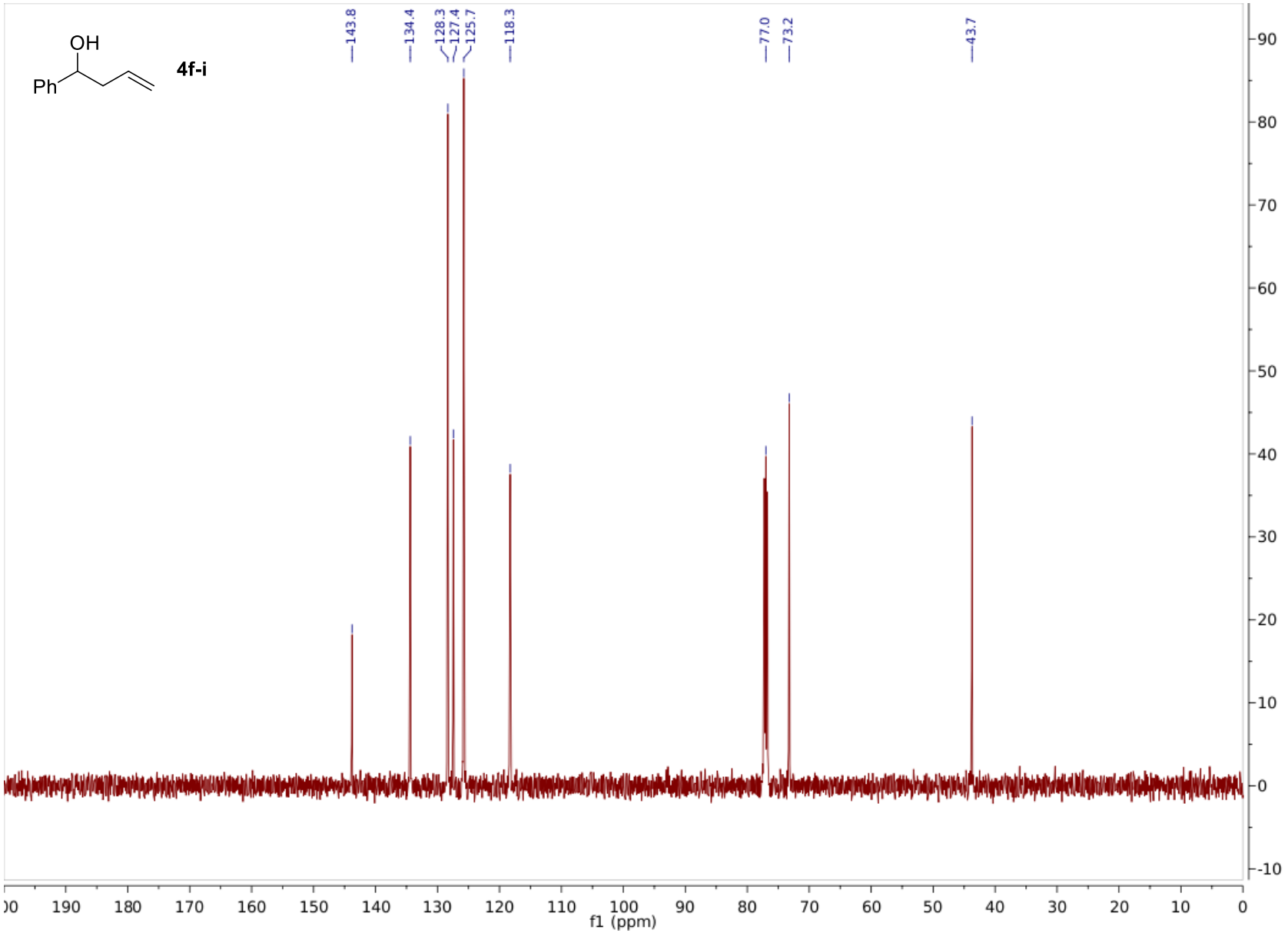

16.88
8.02
8.01
8.01
8.00
8.00
7.99
7.99
7.59
7.58
7.58
7.57
7.57
7.56
7.55
7.55
7.54
7.52
7.52
7.51
7.50
7.50
7.49
7.48
7.48
7.48
7.26
6.87
O
OH
Ph
Ph
4d-i
0.7
4.0
2.1
4.1
1.1
f1 (ppm)

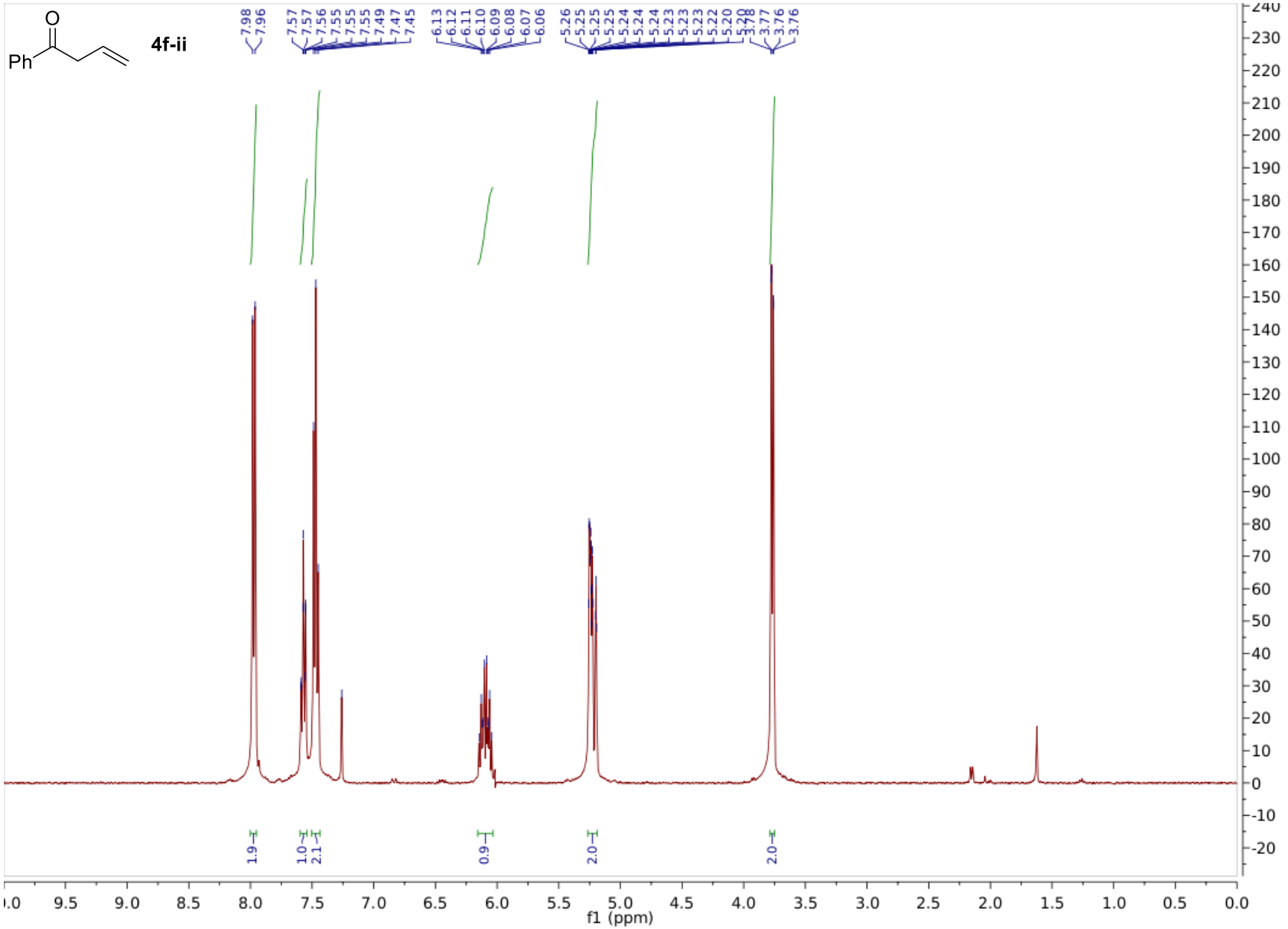

4d
Ph
Ph
7.97
7.95
7.59
7.59
7.57
7.55
7.55
7.47
7.45
7.43
7.26
5.91
5.90
5.89
5.89
5.87
5.86
5.85
5.84
5.30
5.29
5.13
5.12
5.09
5.08
5.08
5.04
5.02
2.90
2.89
2.88
2.87
2.86
2.86
3.8
2.1
4.2
0.8
1.1
1.0
0.9
2.2
f1 (ppm)

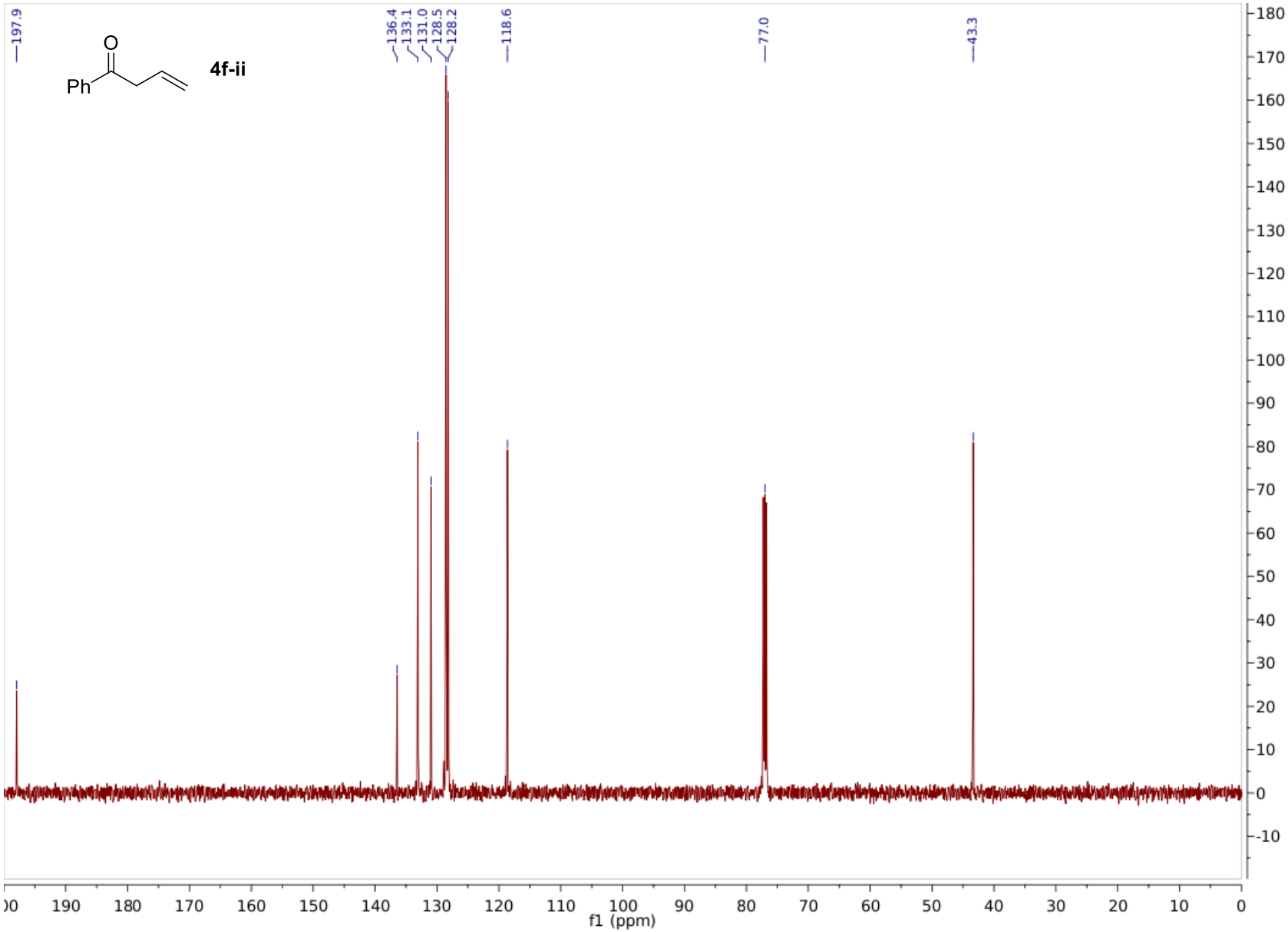

195.4
135.8
134.9
133.5
128.8
128.5
117.1
77.0
56.5
33.5
O
O
Ph
Ph
4d
f1 (ppm)

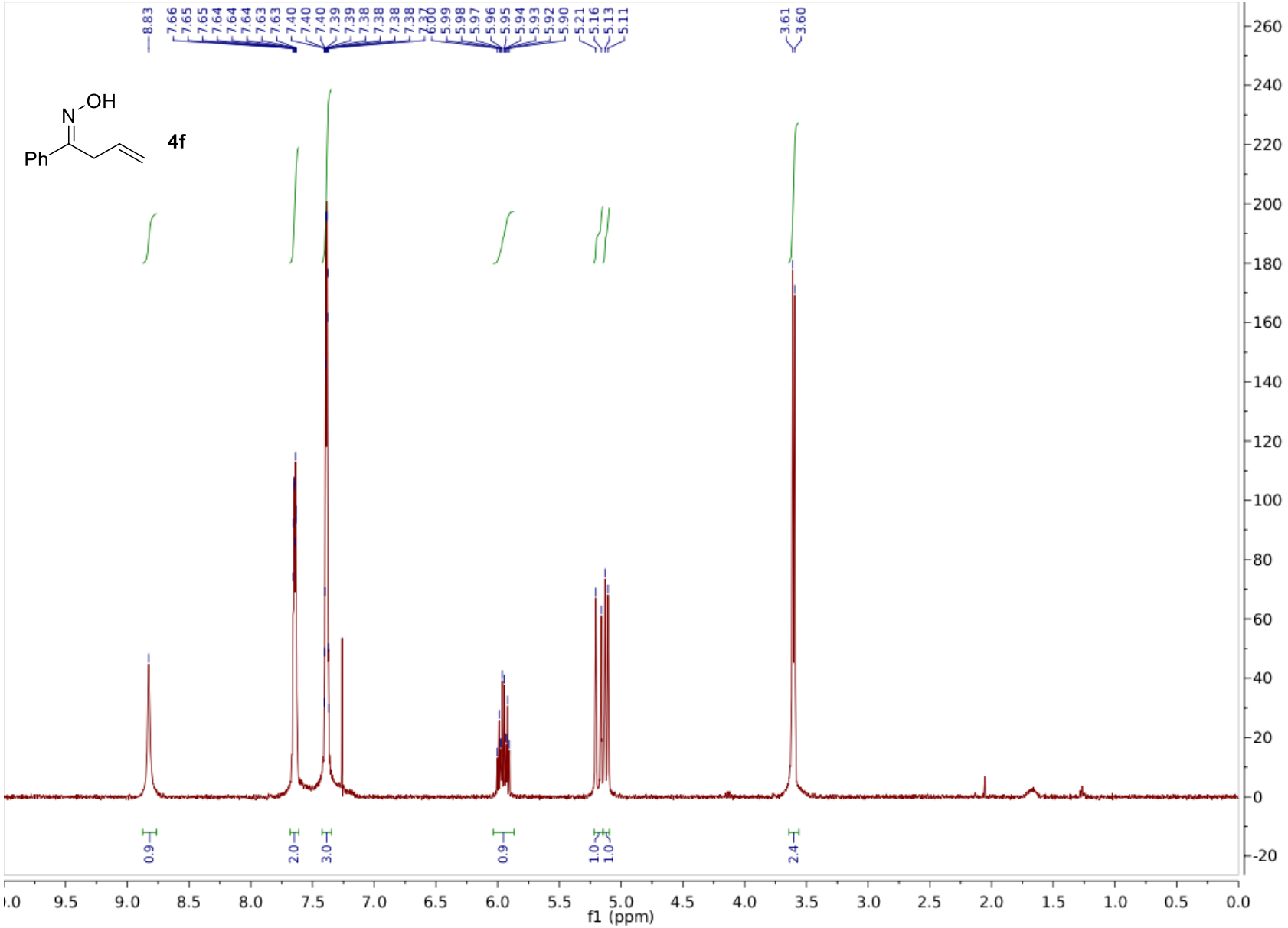

4e
Ph
Ph
Me
7.59
7.59
7.58
7.57
7.57
7.56
7.55
7.55
7.55
7.26
5.45
5.43
5.42
4.78
4.69
2.86
2.84
1.78
3.7
1.9
3.8
1.1
1.0
1.0
2.3
3.2
f1 (ppm)

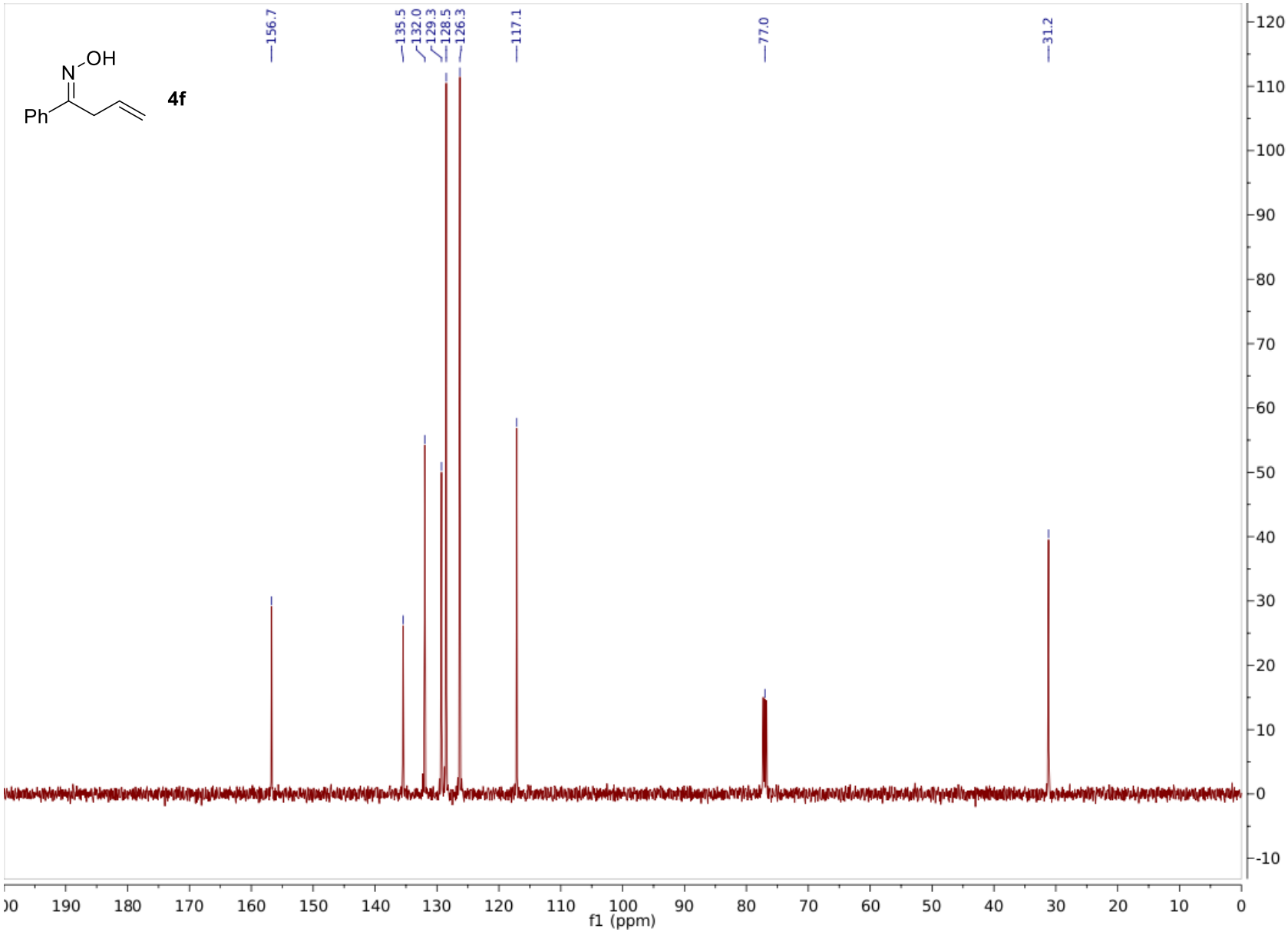

OH
Ph
4f-i
f1 (ppm)

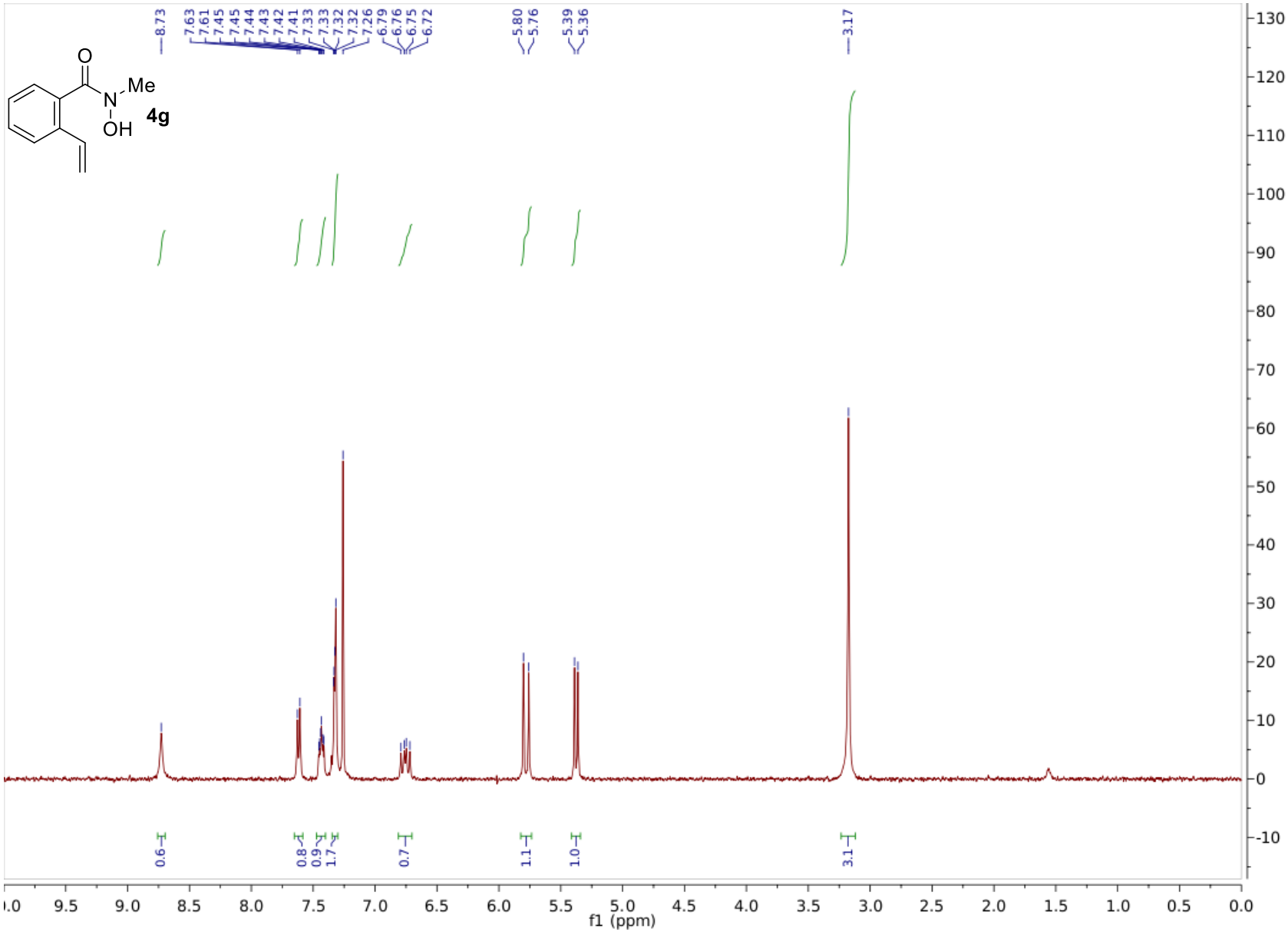

OH
Ph
4f-i
143.8
134.4
128.3
127.4
125.7
118.3
77.0
73.2
43.7
f1 (ppm)

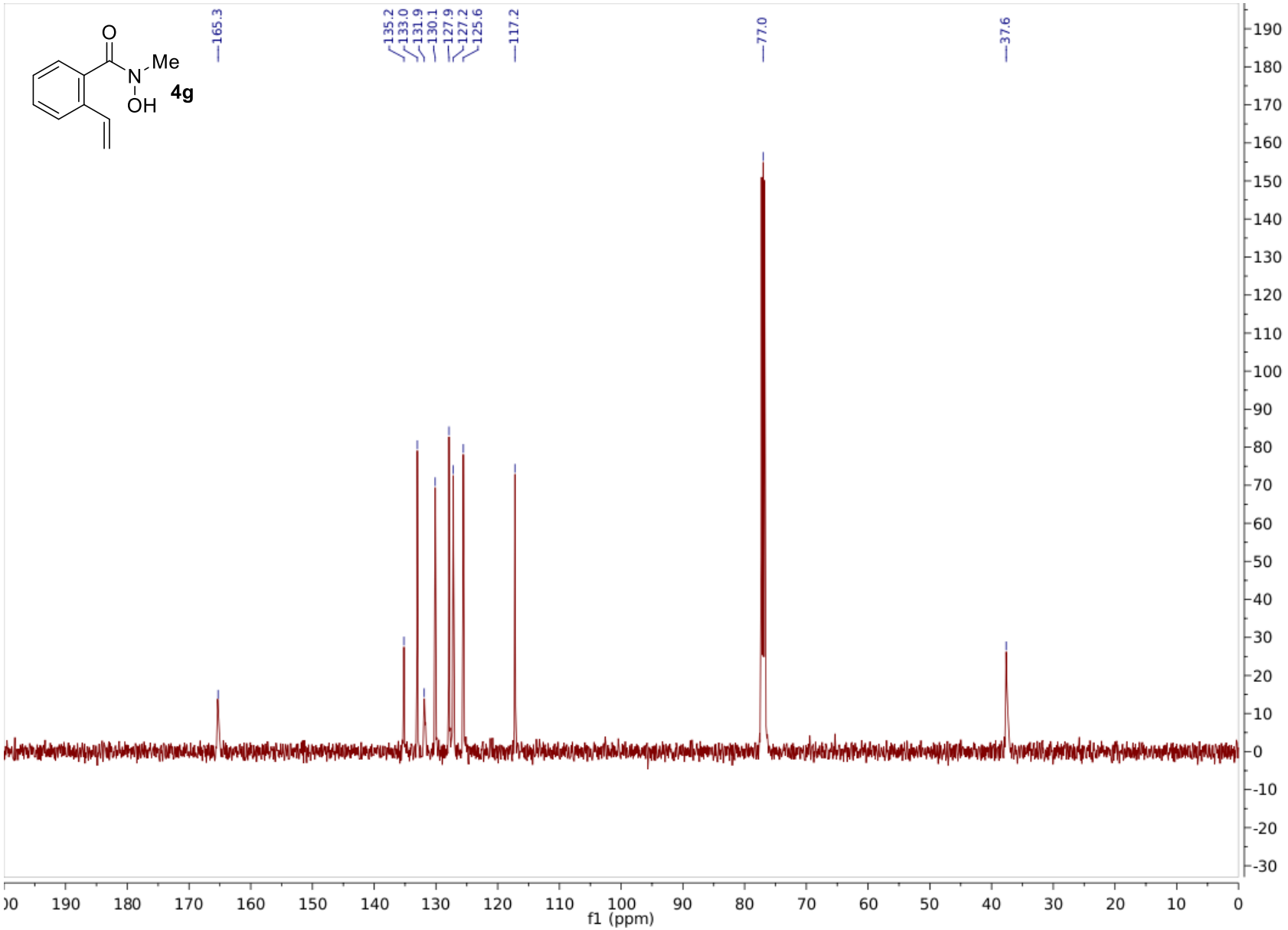

4f-ii
Ph
O
7.98
7.96
7.57
7.57
7.56
7.55
7.55
7.49
7.47
7.45
6.13
6.12
6.11
6.10
6.09
6.08
6.07
6.06
5.26
5.25
5.25
5.25
5.24
5.24
5.24
5.23
5.23
5.23
5.22
5.20
3.78
3.77
3.76
3.76
1.9
1.0
2.1
0.9
2.0
2.0
f1 (ppm)

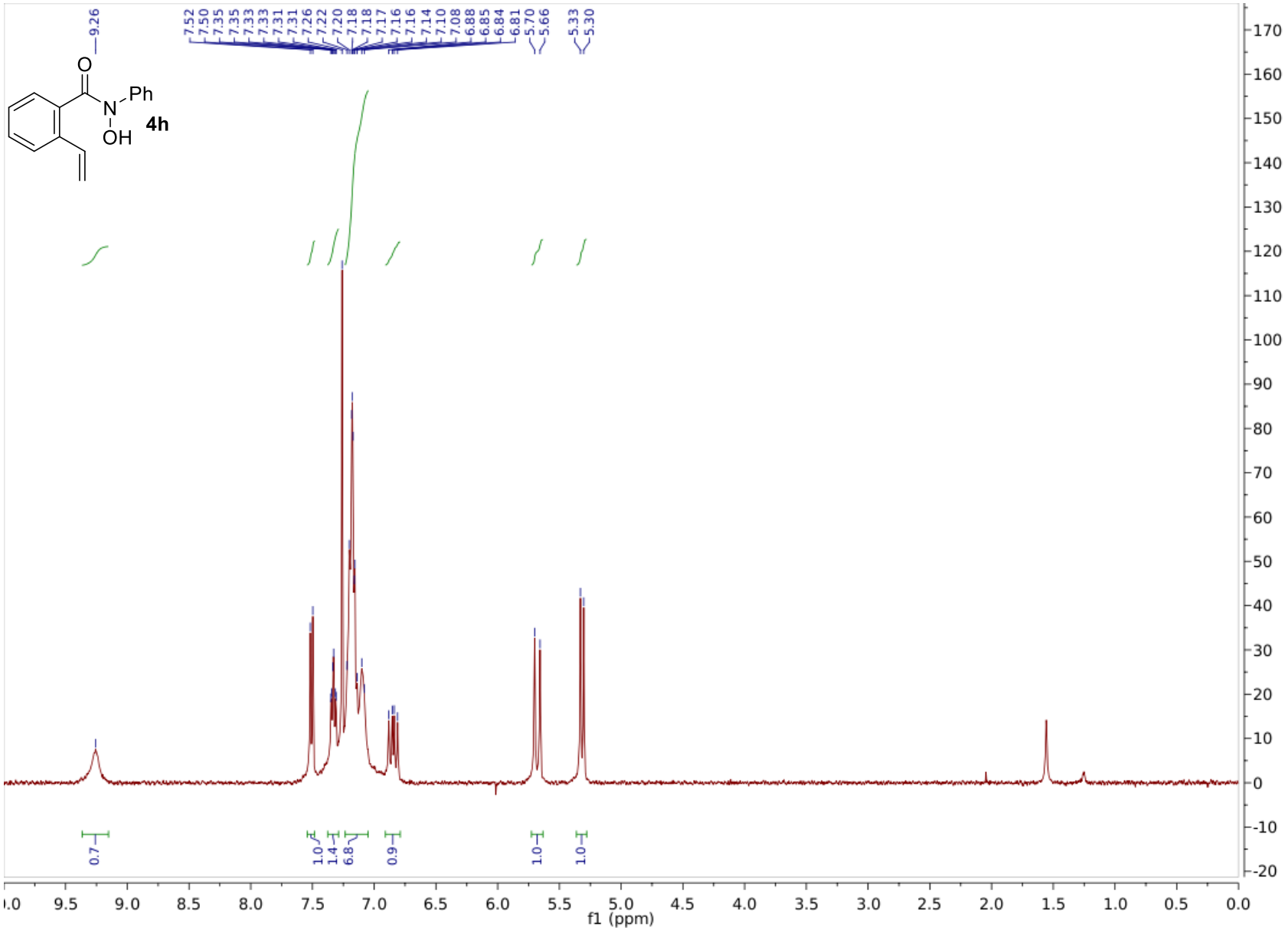

197.9
136.4
133.1
131.0
128.5
128.2
118.6
77.0
43.3
O
Ph
4f-ii
f1 (ppm)

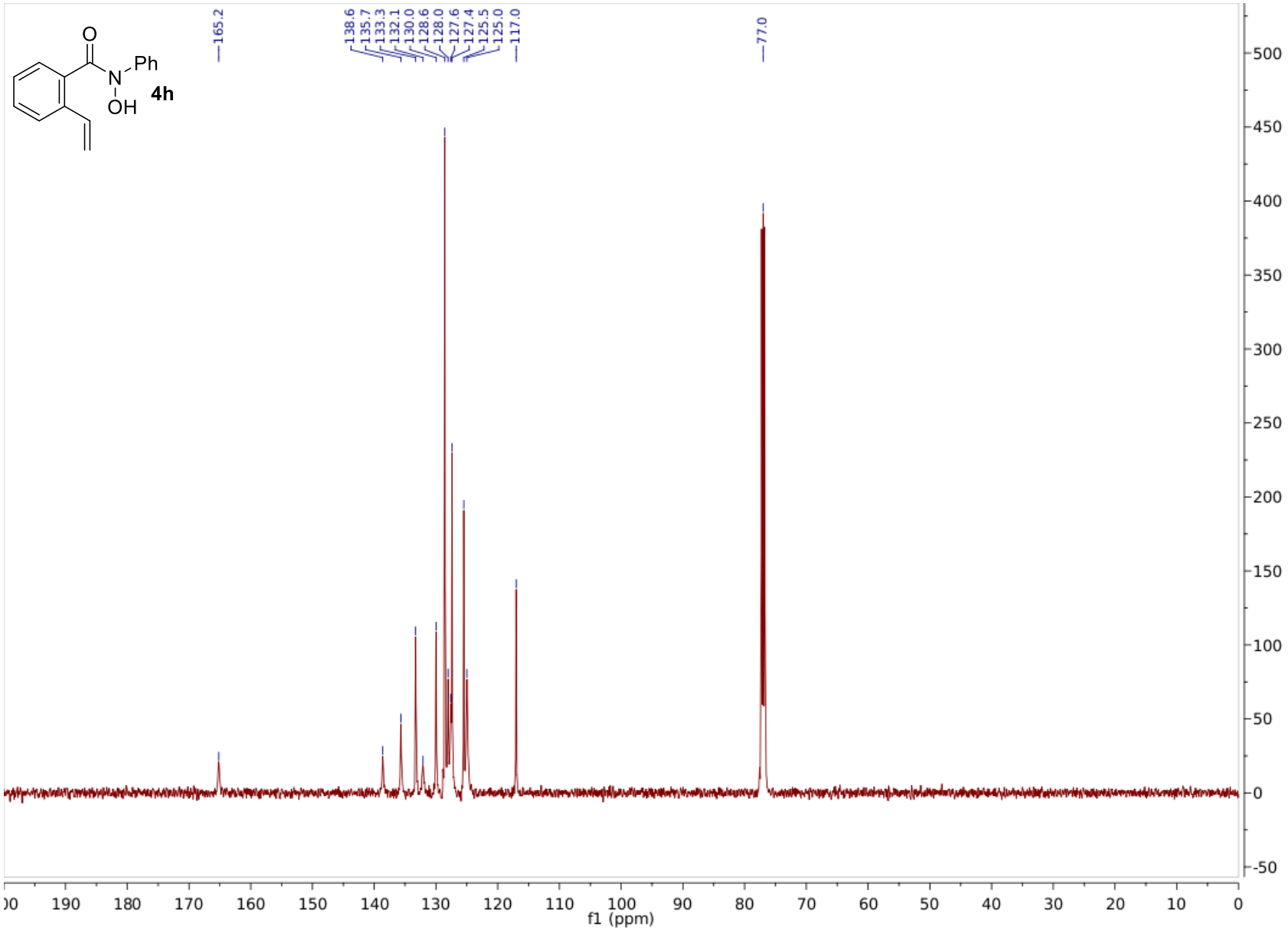

8.83
7.66
7.65
7.65
7.64
7.64
7.64
7.63
7.63
7.40
7.40
7.40
7.39
7.39
7.38
7.38
7.38
7.37
6.00
5.99
5.98
5.97
5.96
5.95
5.94
5.93
5.92
5.90
5.21
5.16
5.13
5.11
3.61
3.60
OH
N
Ph
4f
0.9
2.0
3.0
0.9
1.0
1.0
2.4
f1 (ppm)

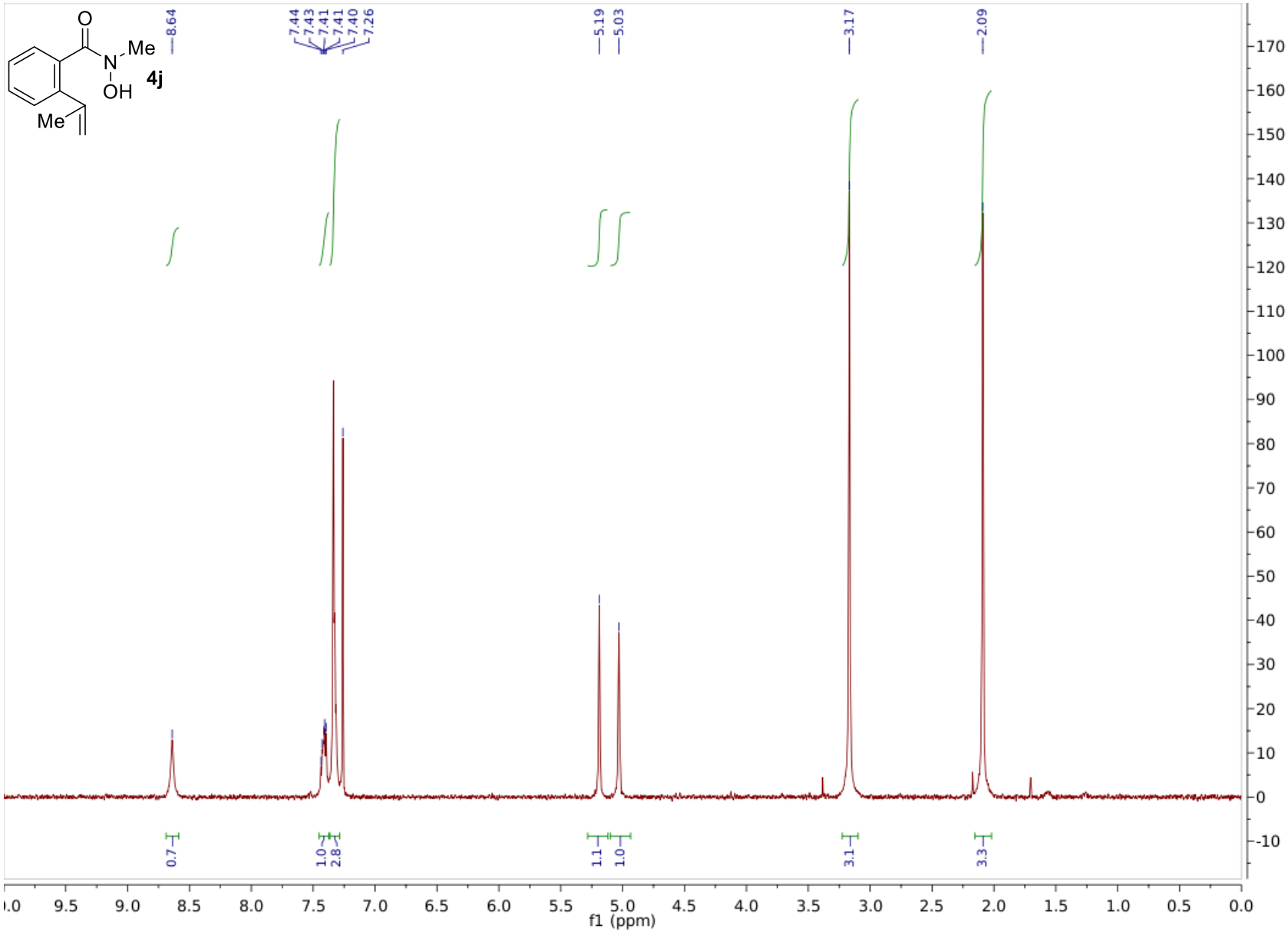

OH
N
Ph
4f
156.7
135.5
132.0
129.3
128.5
126.3
117.1
77.0
31.2
f1 (ppm)

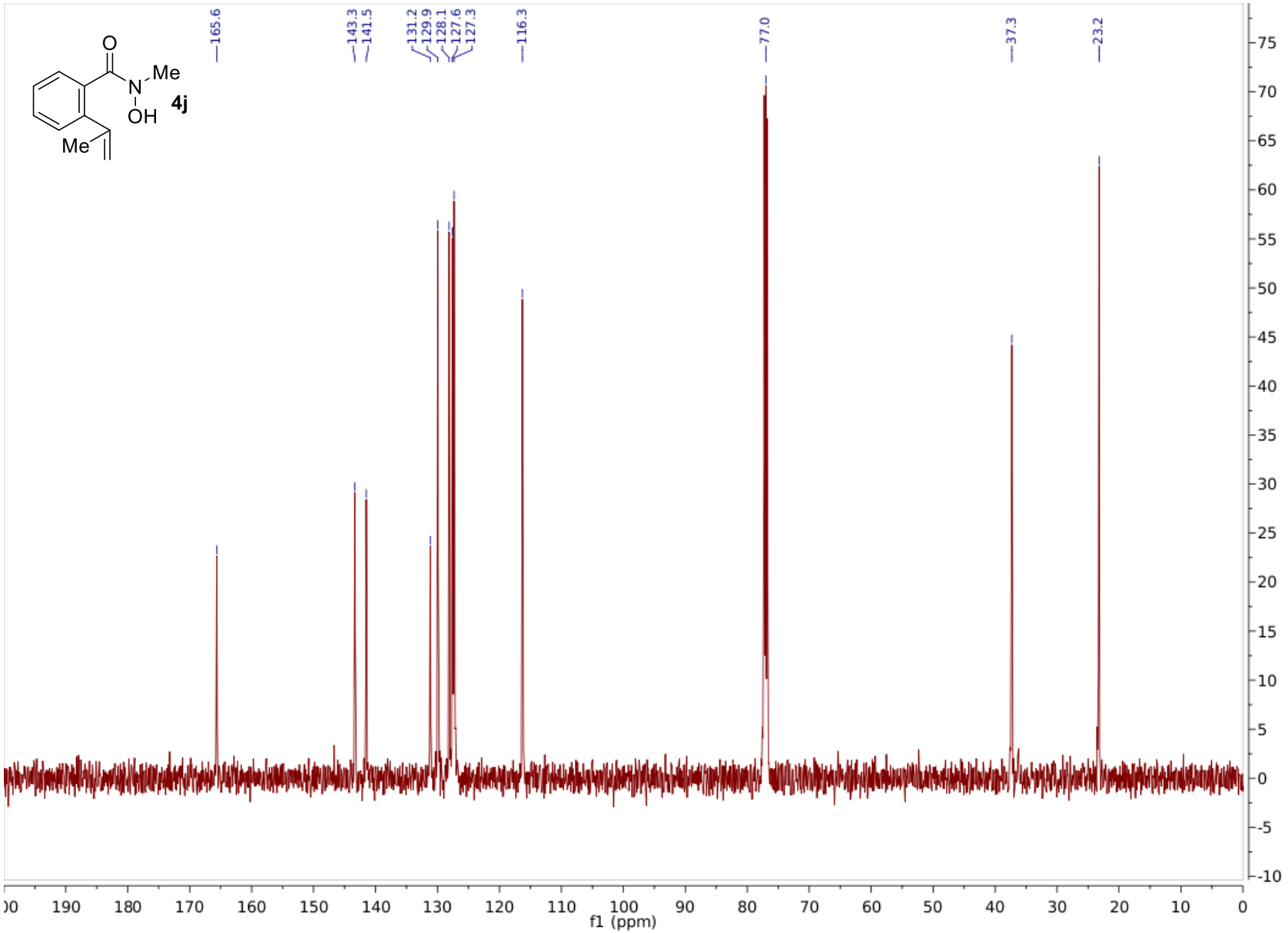

8.73
7.63
7.61
7.45
7.45
7.44
7.43
7.42
7.41
7.33
7.33
7.32
7.32
7.26
6.79
6.76
6.75
6.72
5.80
5.76
5.39
5.36
3.17
O
N
Me
OH
4g
0.6
0.8
0.9
1.7
0.7
1.1
1.0
3.1
f1 (ppm)

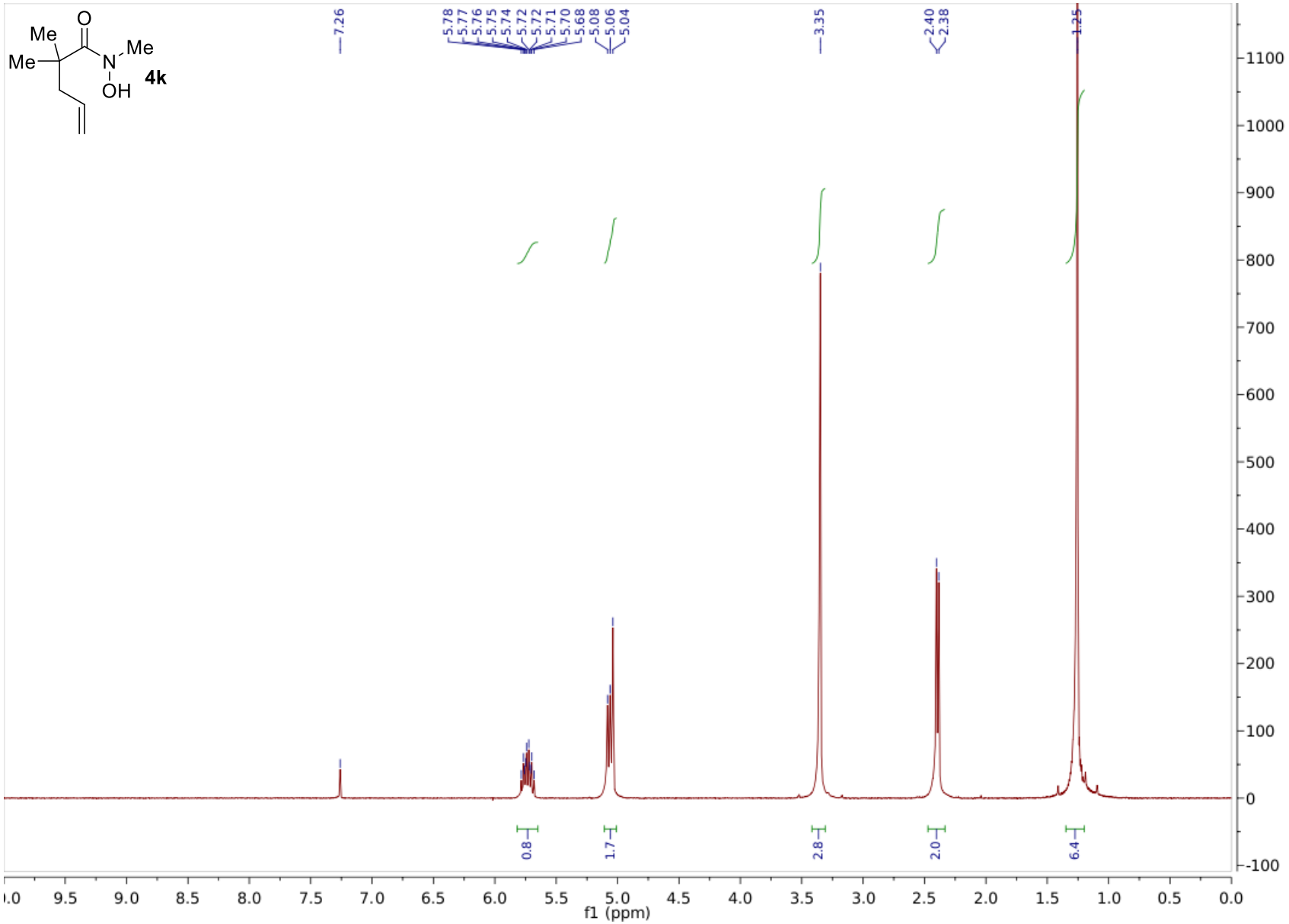

O
N
Me
OH
4g
165.3
135.2
133.0
131.9
130.1
127.9
127.2
125.6
117.2
77.0
37.6
f1 (ppm)

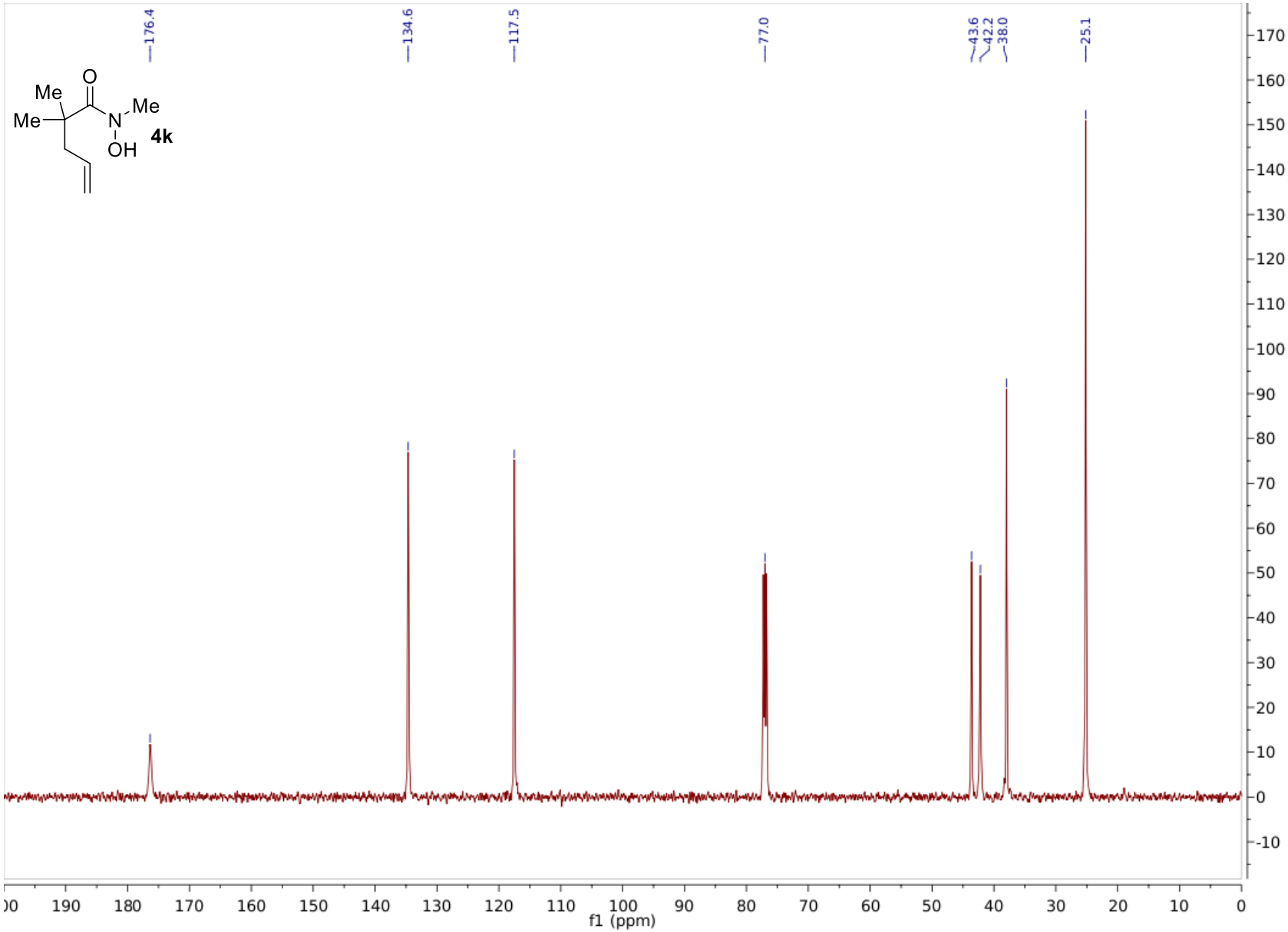

9.26
7.52
7.50
7.35
7.35
7.33
7.33
7.31
7.31
7.26
7.22
7.20
7.18
7.18
7.17
7.16
7.16
7.14
7.10
7.08
6.88
6.85
6.84
6.81
5.70
5.66
5.33
5.30
O
N
Ph
OH
4h
0.7
1.0
1.4
6.8
0.9
1.0
1.0
f1 (ppm)

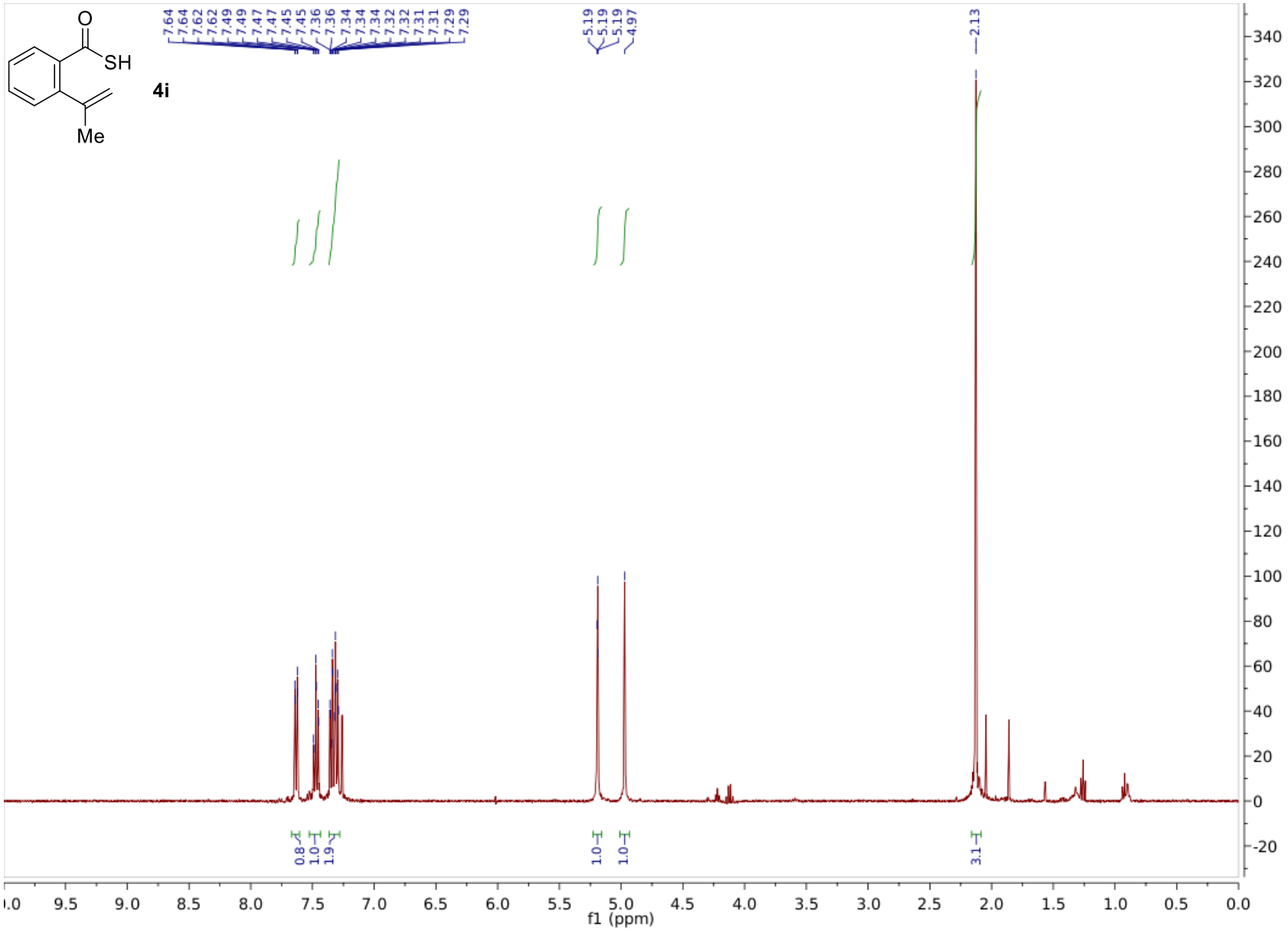

O
Ph
N
OH
4h
165.2
138.6
135.7
133.3
132.1
130.0
128.6
128.0
127.6
127.4
125.5
125.0
117.0
77.0
500
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

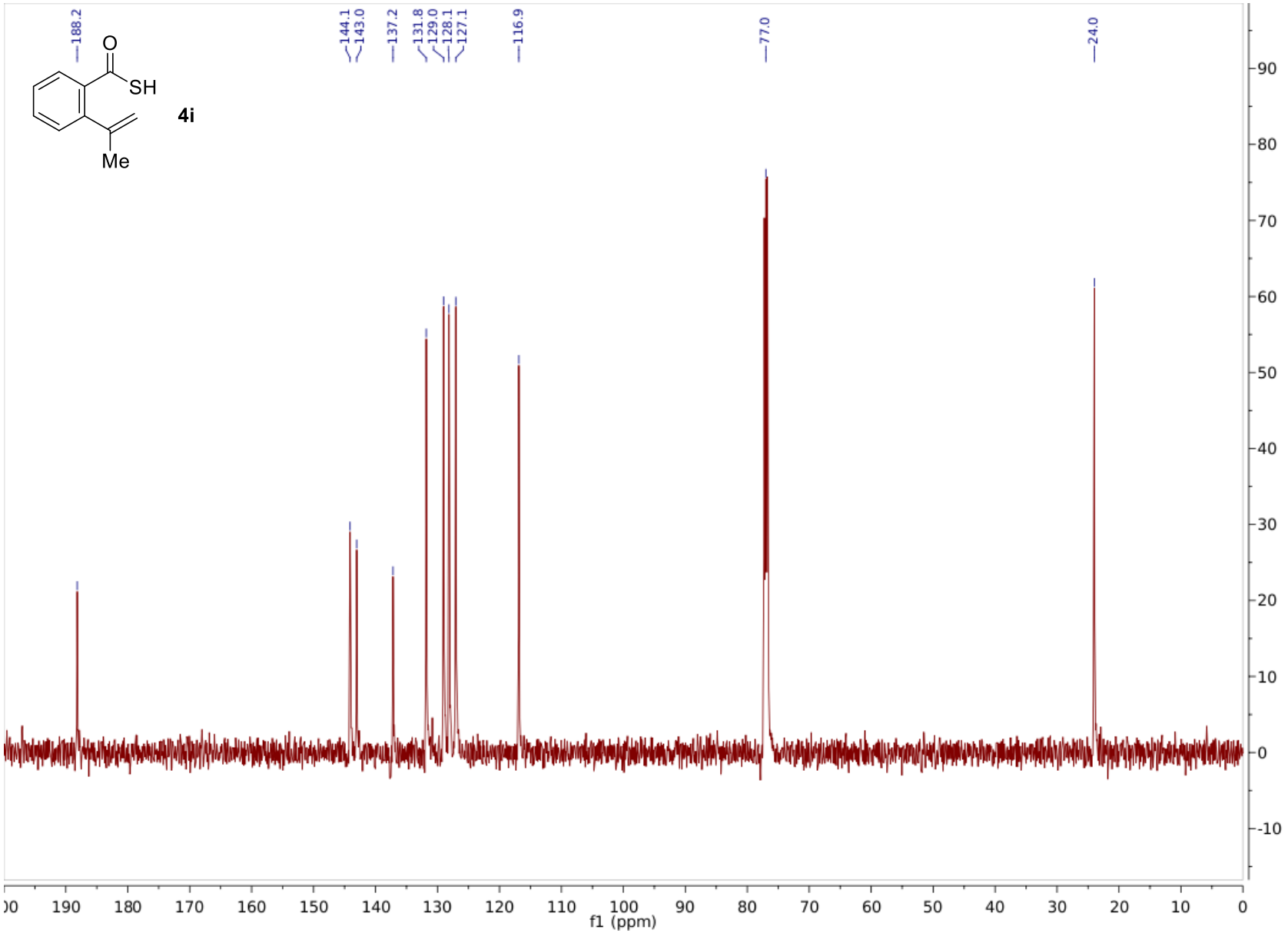

O
N
Me
OH
Me
4j
8.64
7.44
7.43
7.41
7.41
7.40
7.26
5.19
5.03
3.17
2.09
0.7
1.0
2.8
1.1
1.0
3.1
3.3
f1 (ppm)

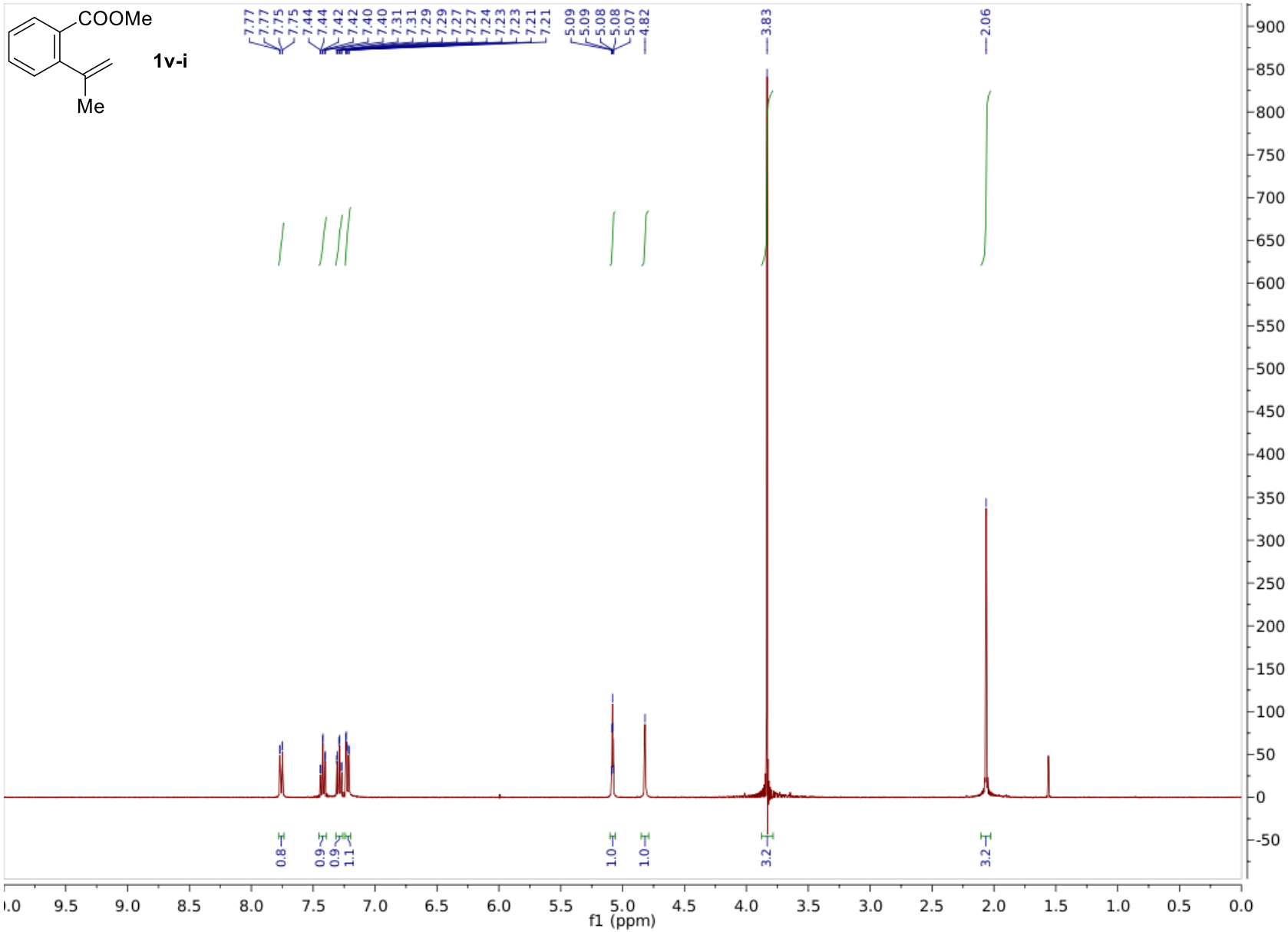

O
N
Me
OH
Me
4j
165.6
143.3
141.5
131.2
129.9
128.1
127.6
127.3
116.3
77.0
37.3
23.2
f1 (ppm)

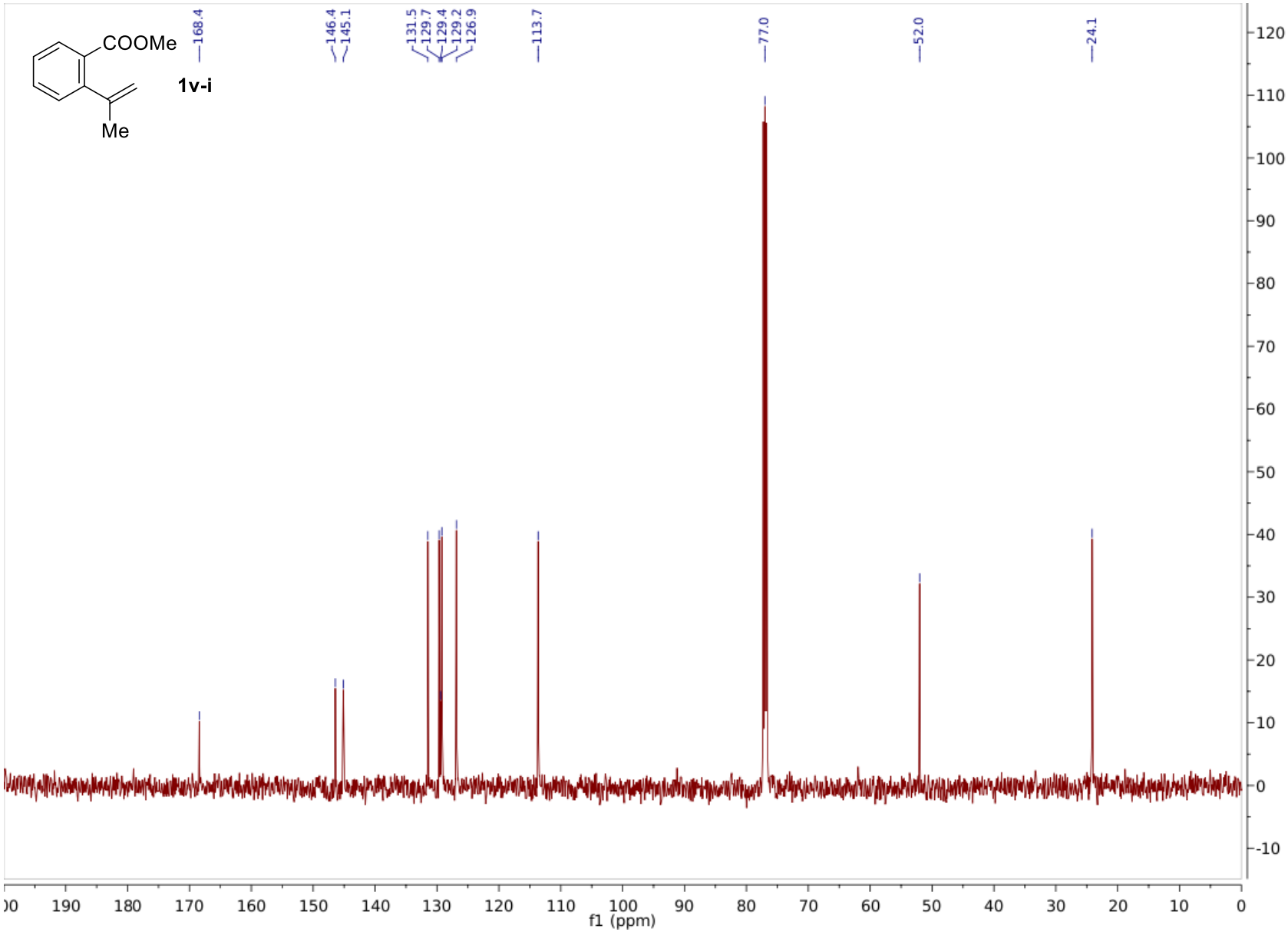

Me
Me
O
N
Me
OH
4k
7.26
5.78
5.77
5.76
5.75
5.74
5.72
5.72
5.71
5.70
5.68
5.08
5.06
5.04
3.35
2.40
2.38
1.25
0.8
1.7
2.8
2.0
6.4
f1 (ppm)

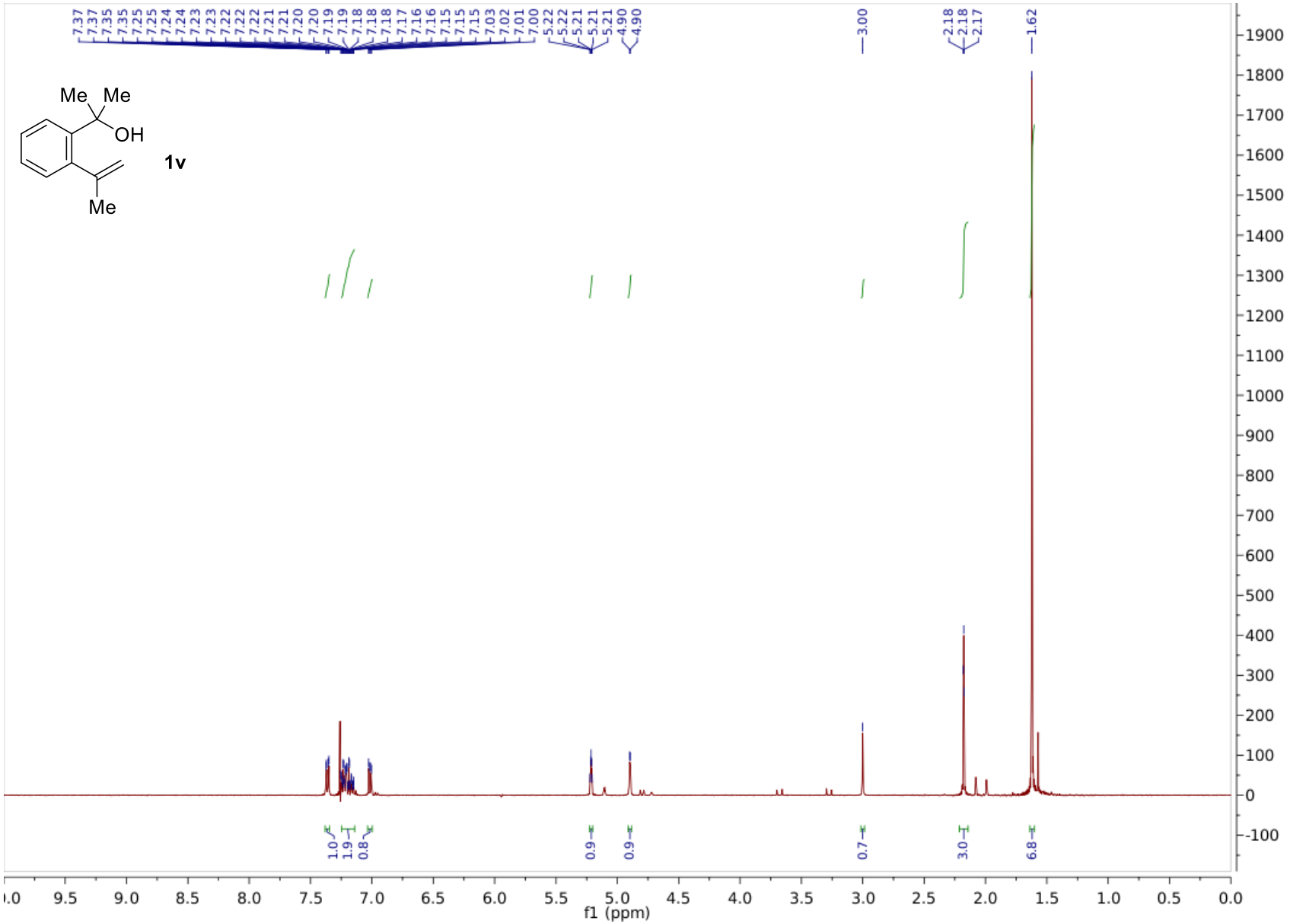

176.4
134.6
117.5
77.0
43.6
42.2
38.0
25.1
Me
Me
O
N
Me
OH
4k
f1 (ppm)

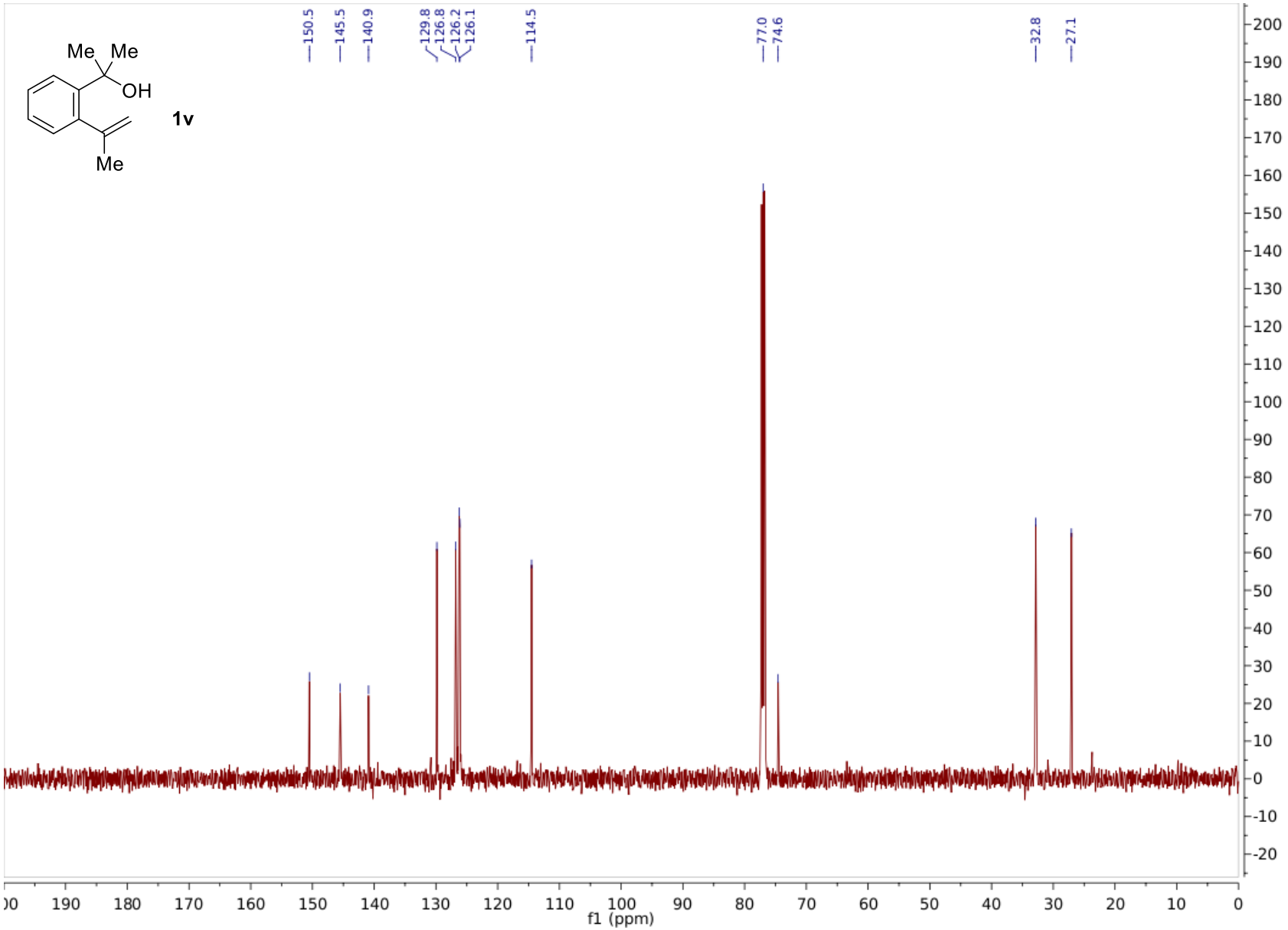

O
SH
Me
4i
7.64
7.64
7.62
7.62
7.49
7.49
7.47
7.47
7.45
7.45
7.36
7.34
7.34
7.34
7.32
7.32
7.31
7.31
7.29
7.29
5.19
5.19
5.19
4.97
2.13
0.8
1.0
1.9
1.0
1.0
3.1
f1 (ppm)

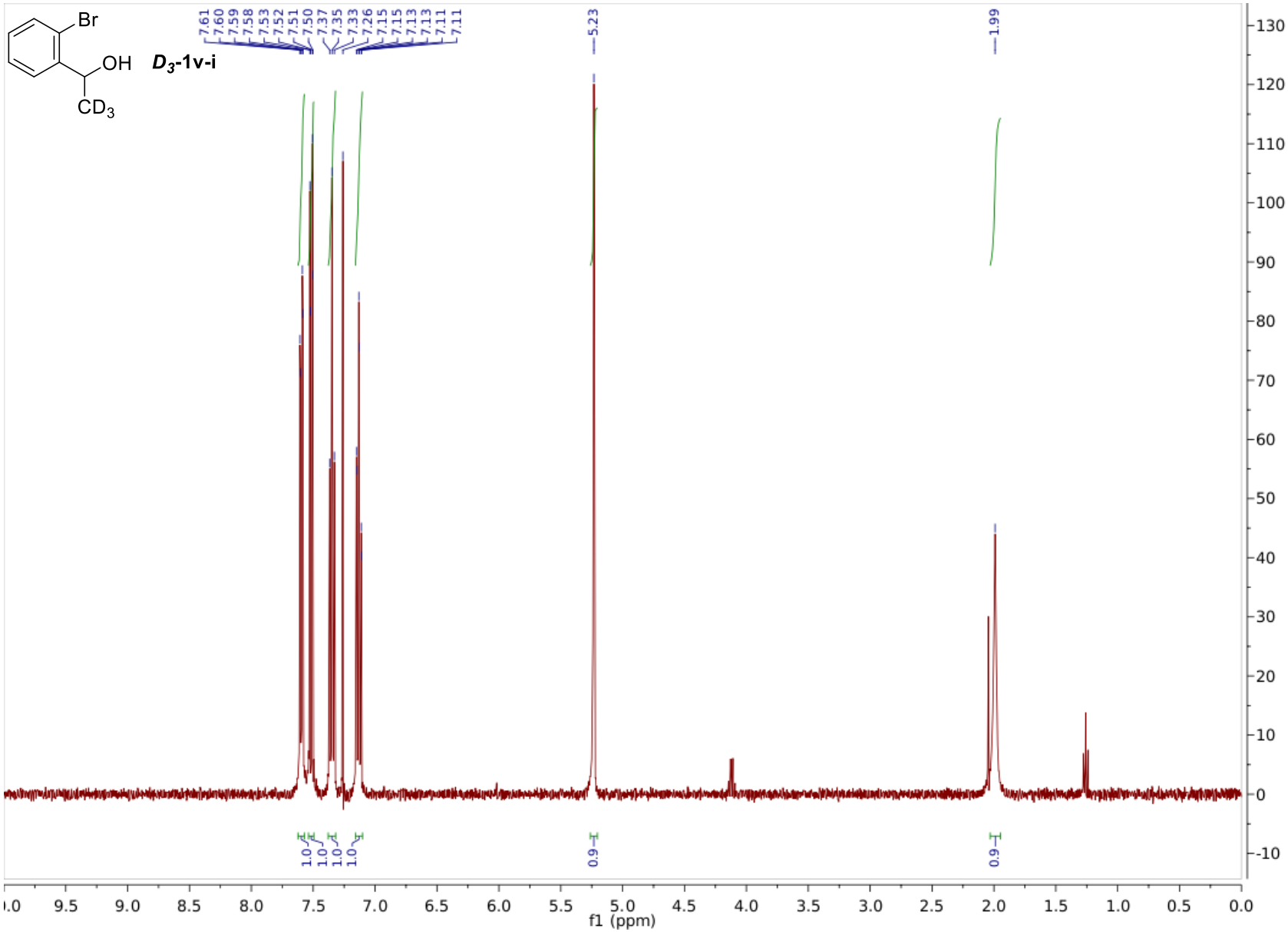

188.2
144.1
143.0
137.2
131.8
129.0
128.1
127.1
116.9
77.0
24.0
O
SH
Me
4i
f1 (ppm)

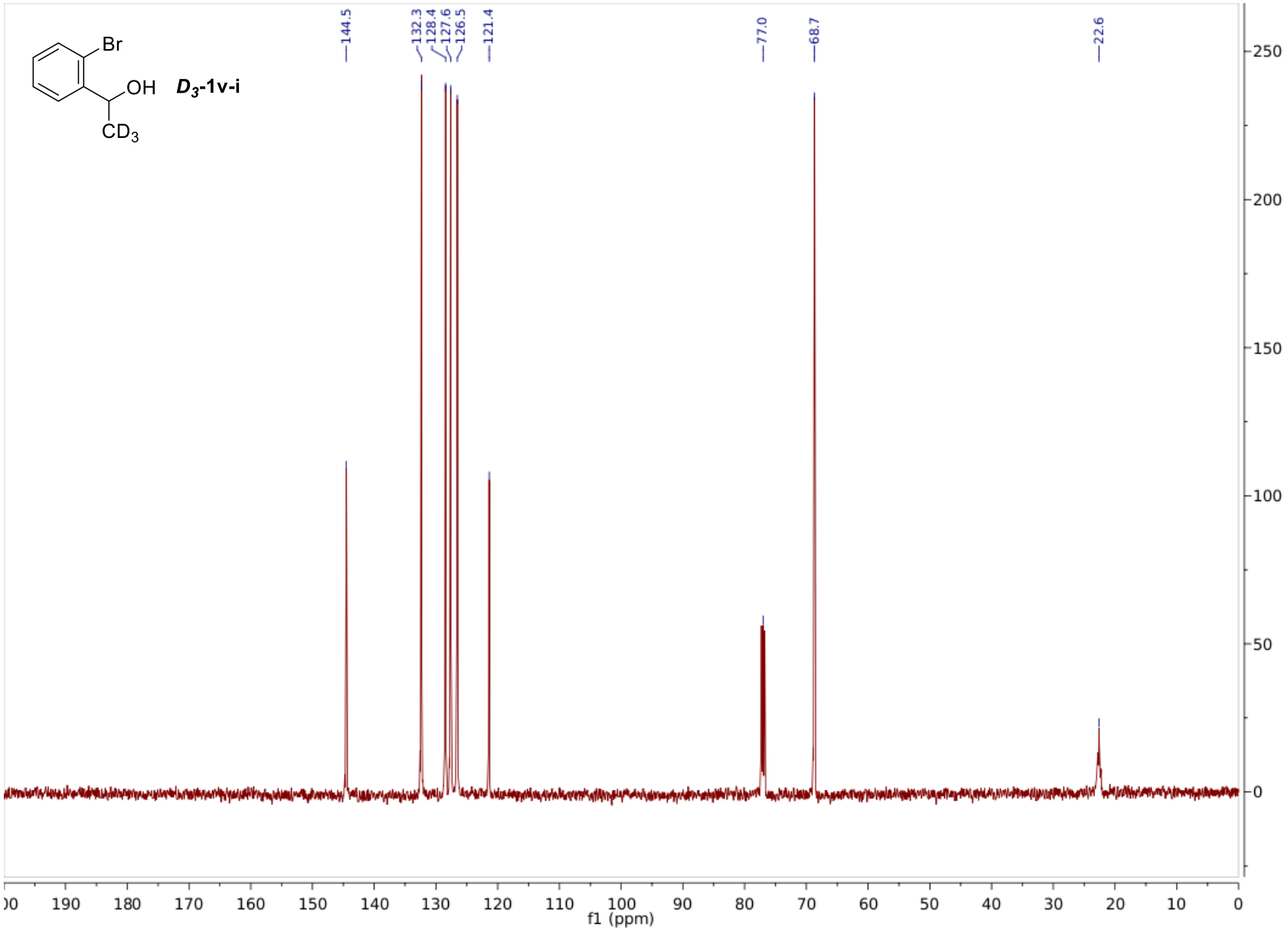

COOMe
Me
1v-i
7.77
7.77
7.75
7.75
7.44
7.44
7.42
7.42
7.40
7.40
7.31
7.31
7.29
7.29
7.27
7.27
7.24
7.23
7.23
7.21
7.21
5.09
5.09
5.08
5.08
5.07
4.82
3.83
2.06
0.8
0.9
0.9
1.1
1.0
1.0
3.2
3.2
f1 (ppm)

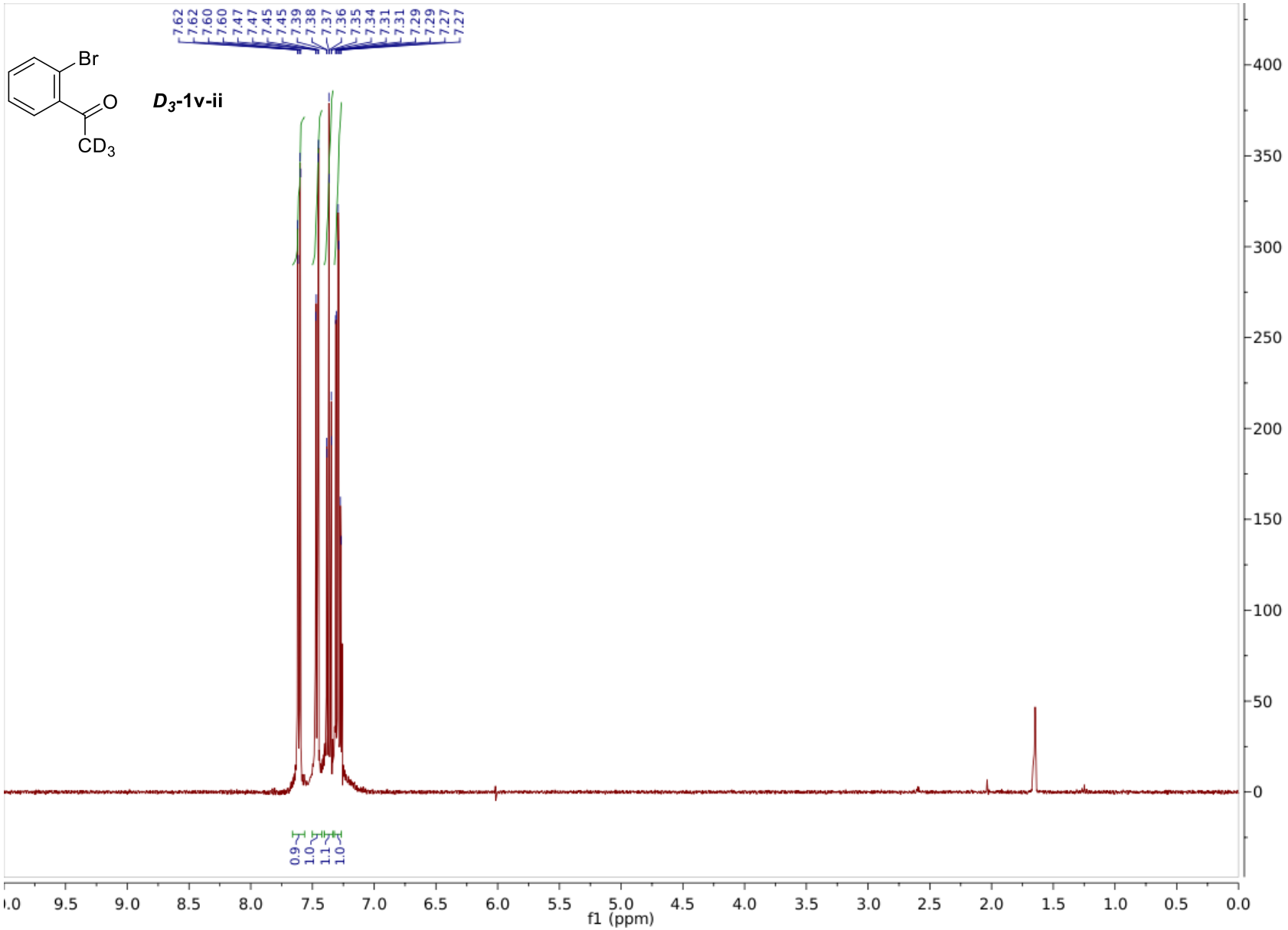

COOMe
Me
1v-i
168.4
146.4
145.1
131.5
129.7
129.4
129.2
126.9
113.7
77.0
52.0
24.1
f1 (ppm)

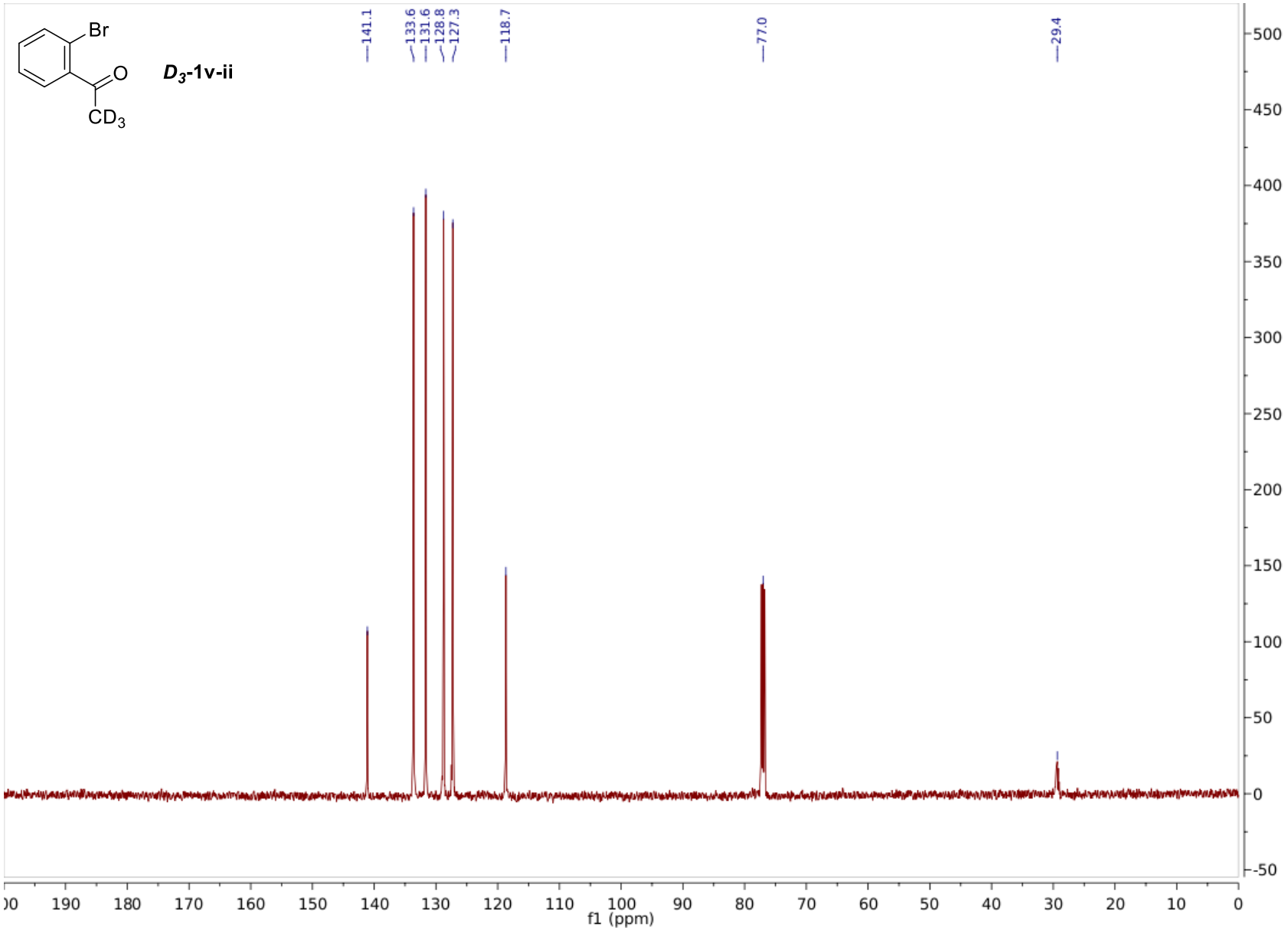

Me Me
OH
Me
1v
7.37 7.37 7.35 7.35 7.25 7.25 7.24 7.24 7.23 7.23 7.22 7.22 7.22 7.21 7.21 7.20 7.20 7.19 7.19 7.18 7.18 7.18 7.17 7.16 7.16 7.15 7.15 7.15 7.03 7.02 7.01 7.00
5.22 5.22 5.21 5.21 5.21 4.90 4.90
3.00
2.18 2.18 2.17
1.62
1.0 1.9 0.8 0.9 0.9 0.7 3.0 6.8
f1 (ppm)

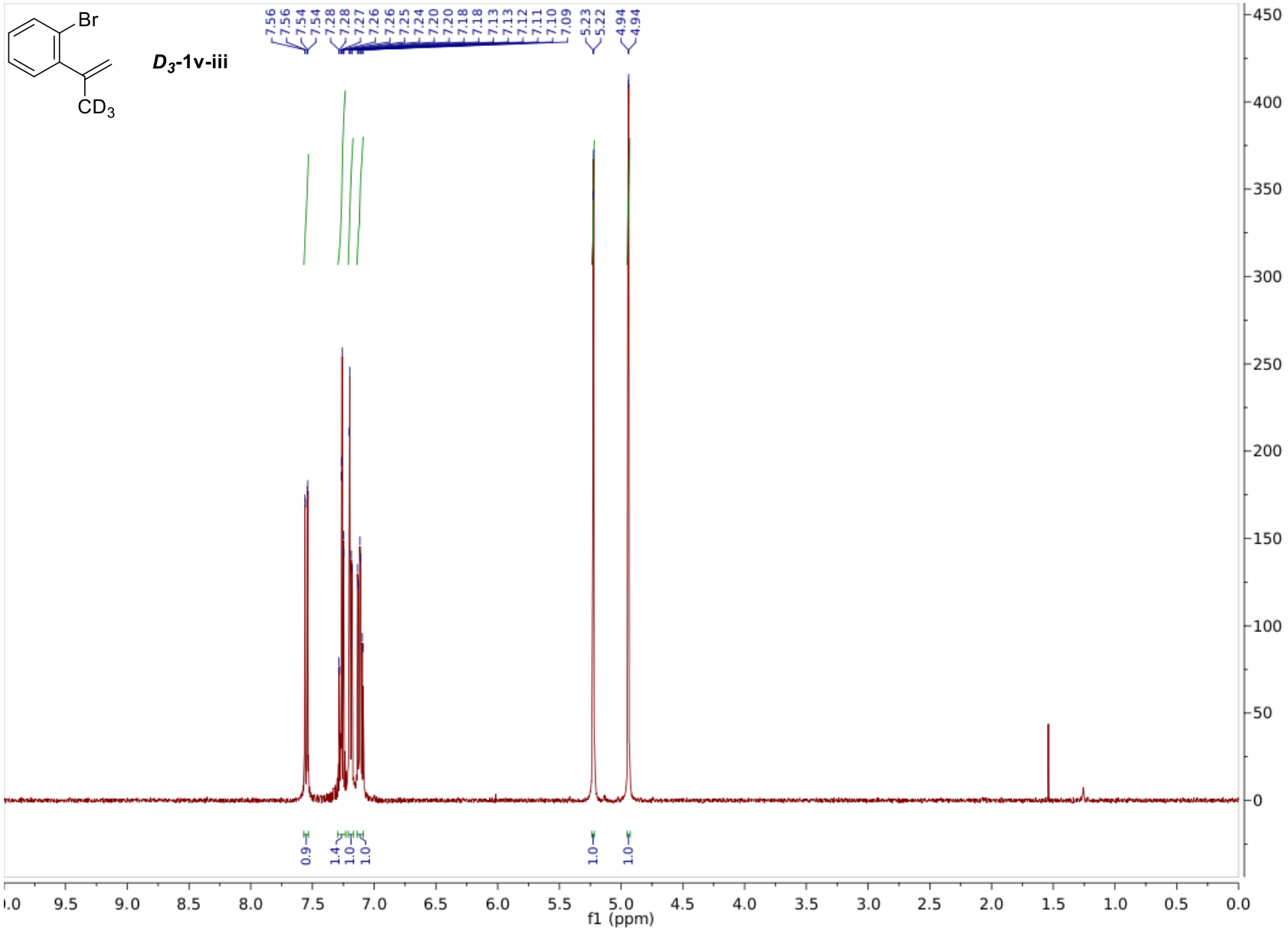

Me
Me
OH
Me
1v
150.5
145.5
140.9
129.8
126.8
126.2
126.1
114.5
77.0
74.6
32.8
27.1
f1 (ppm)

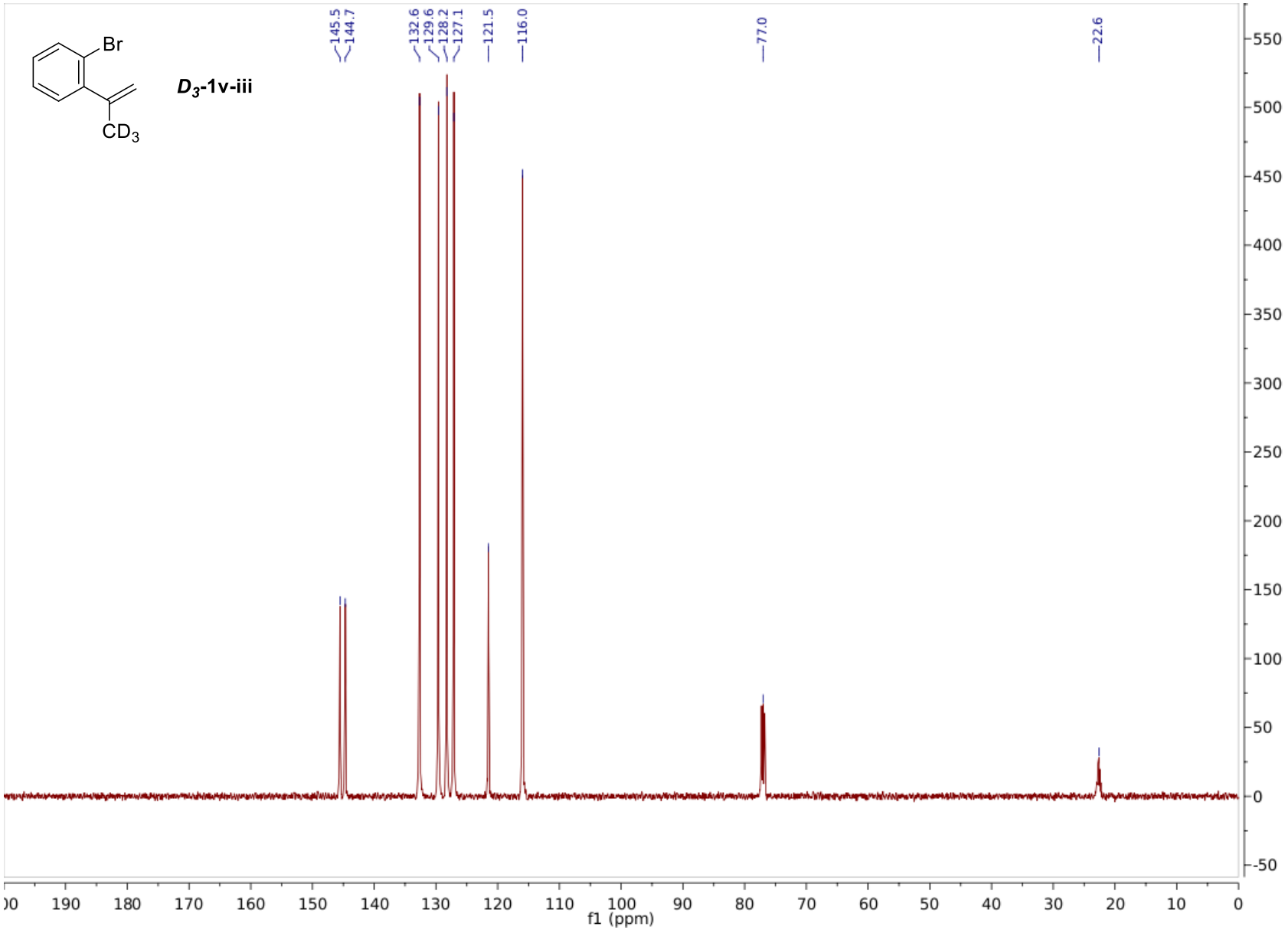

Br
OH
CD3
D3-1v-i
7.61
7.60
7.59
7.58
7.53
7.52
7.51
7.50
7.37
7.35
7.33
7.26
7.15
7.15
7.13
7.13
7.11
7.11
5.23
1.99
1.0
1.0
1.0
1.0
0.9
0.9
f1 (ppm)

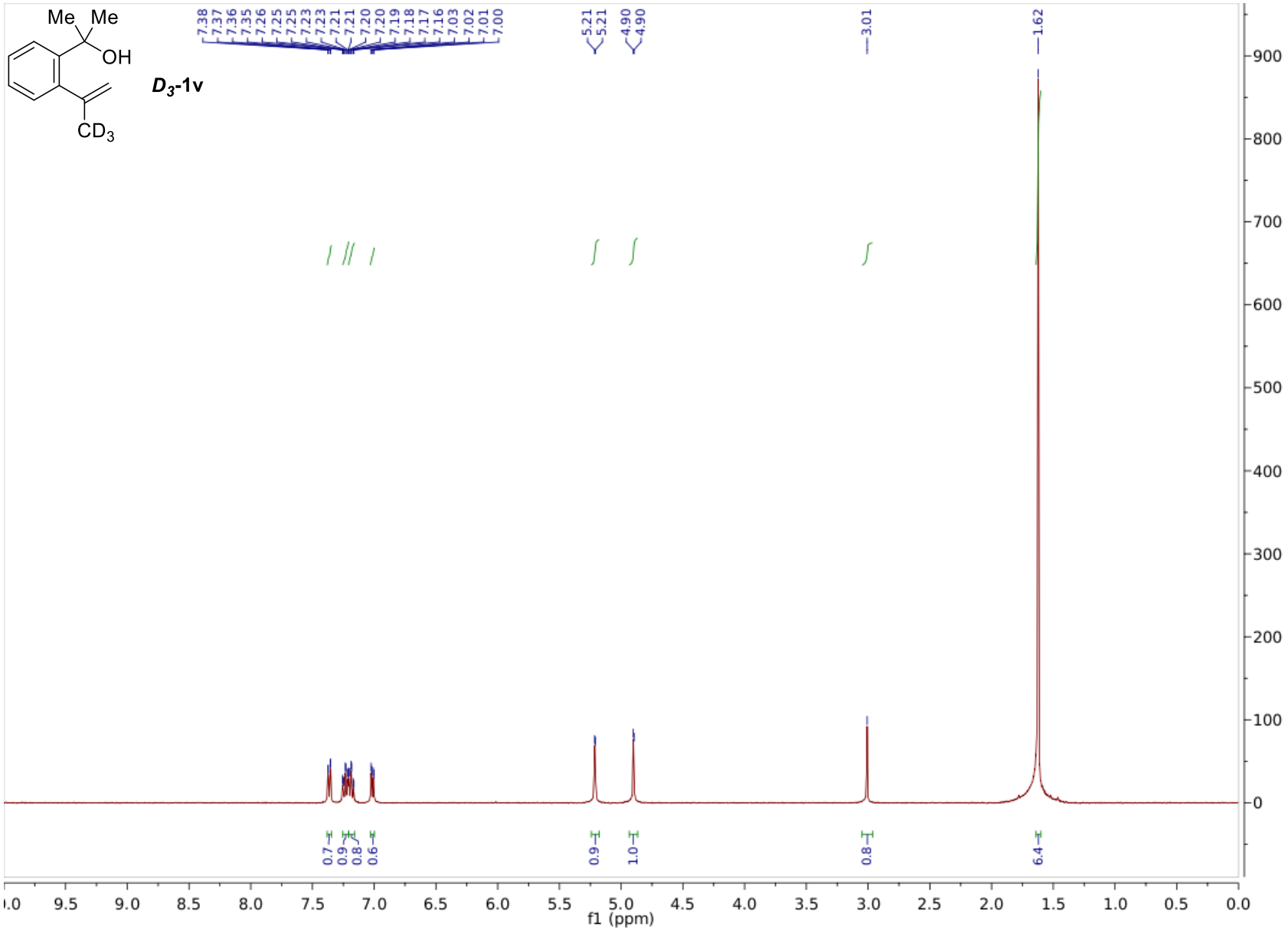

Br
OH
CD3
D3-1v-i
144.5
132.3
128.4
127.6
126.5
121.4
77.0
68.7
22.6
250
200
150
100
50
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

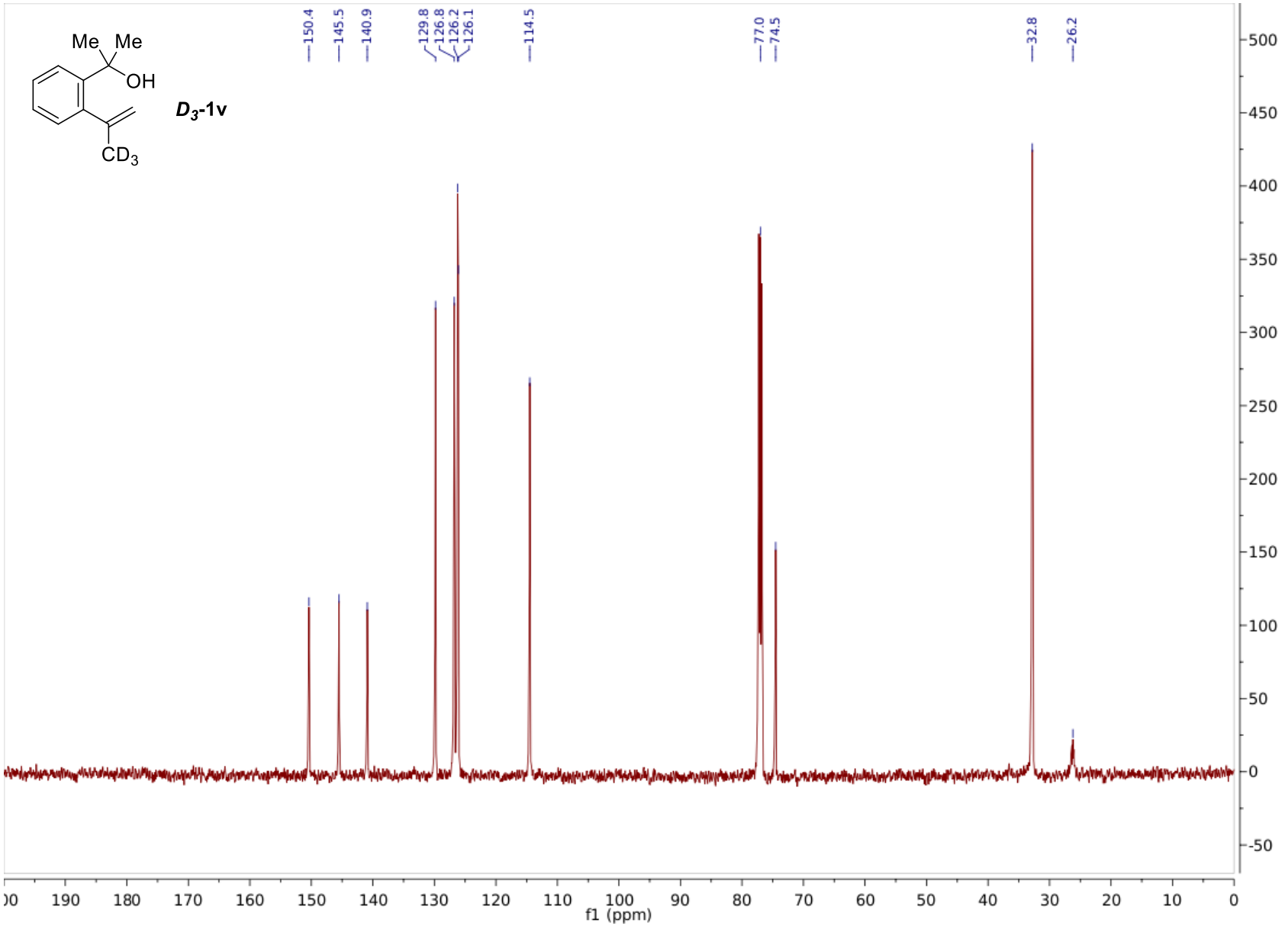

Br
O
CD3
D3-1v-ii
7.62
7.62
7.60
7.60
7.47
7.47
7.45
7.45
7.39
7.38
7.37
7.36
7.35
7.34
7.31
7.31
7.29
7.29
7.27
7.27
0.9
1.0
1.1
1.0
400
350
300
250
200
150
100
50
0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

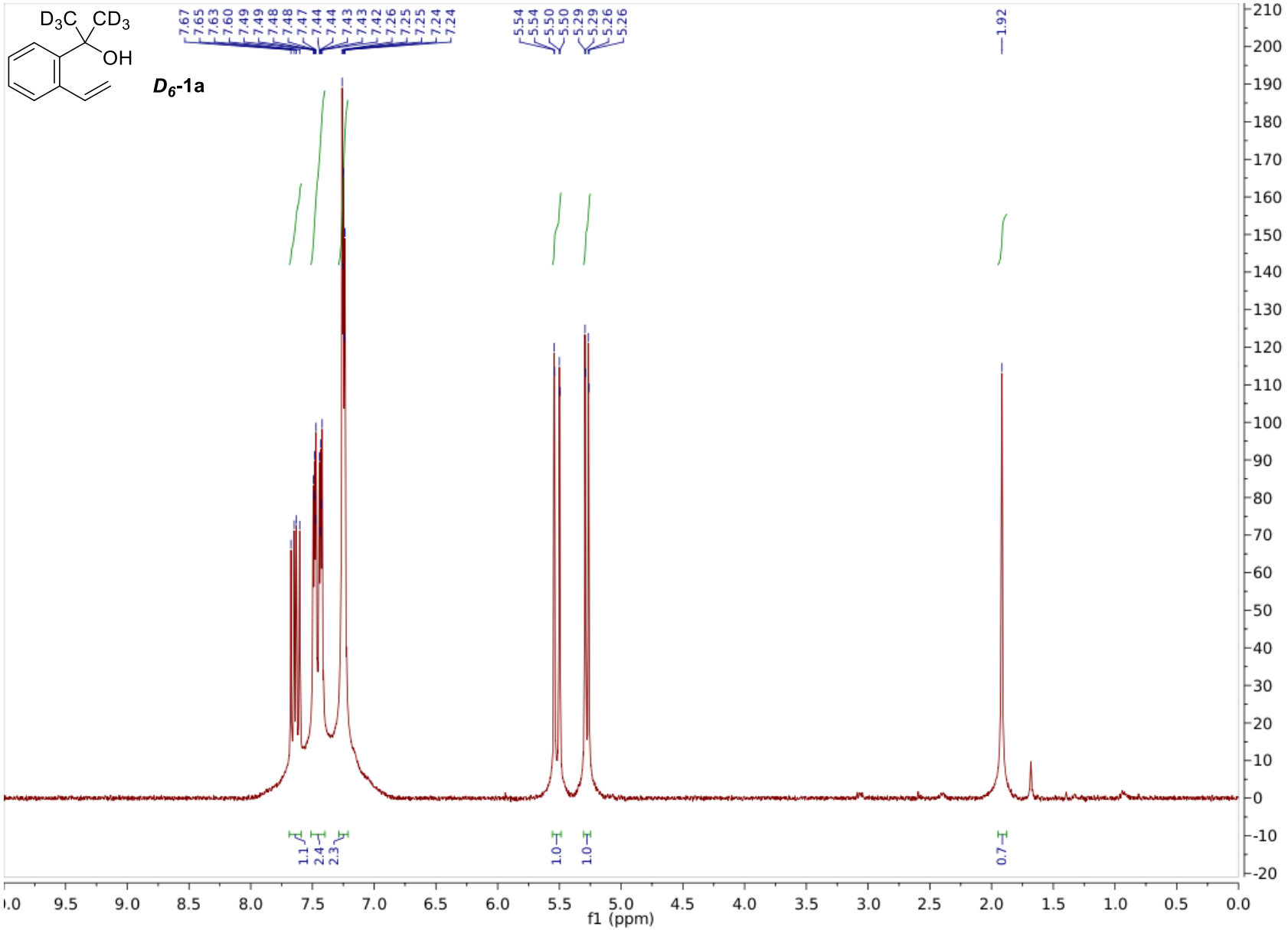

Br
O
CD3
D3-1v-ii
141.1
133.6
131.6
128.8
127.3
118.7
77.0
29.4
500
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

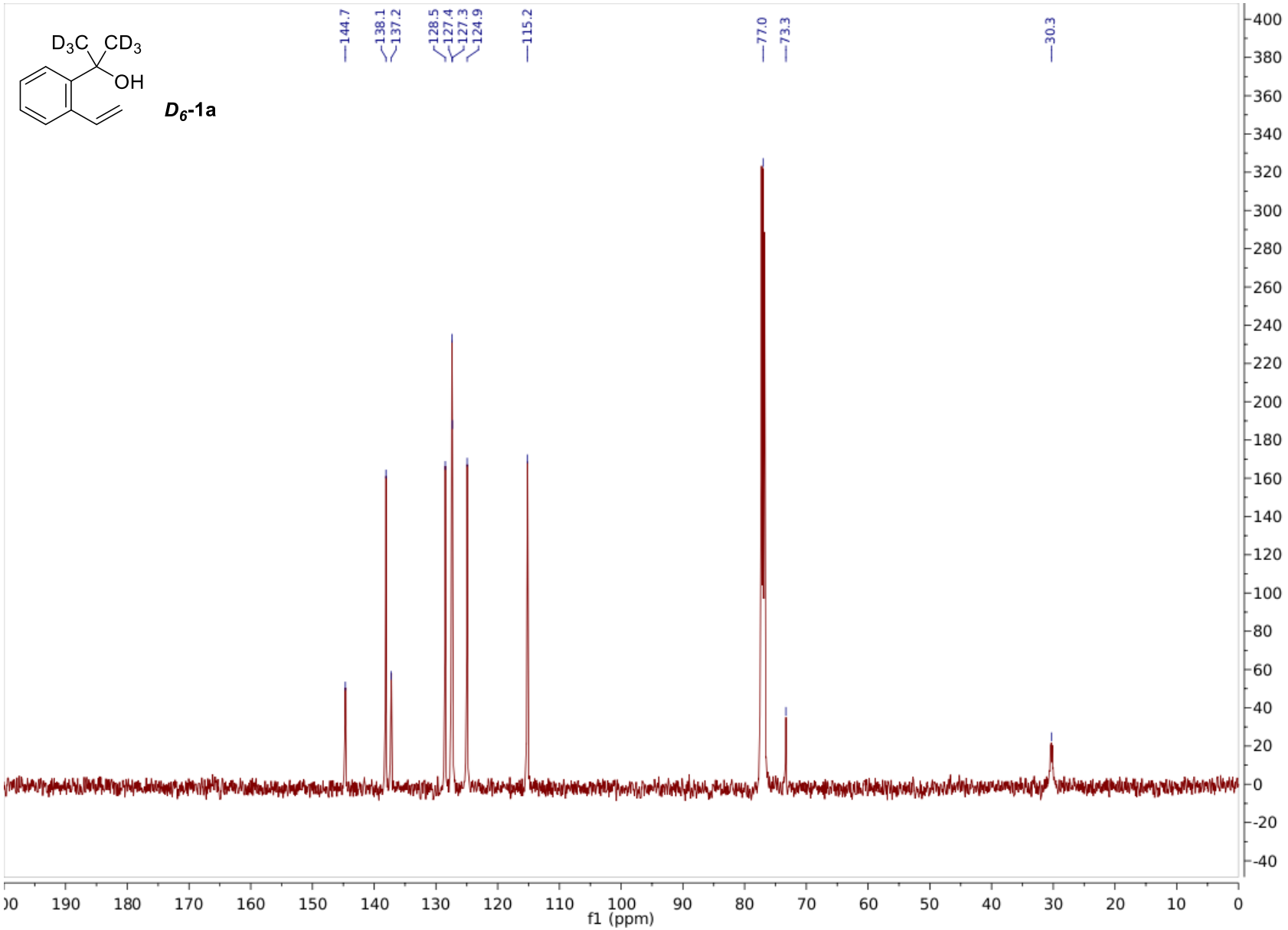

Br
CD3
D3-1v-iii
7.56
7.56
7.54
7.54
7.28
7.27
7.26
7.25
7.24
7.20
7.20
7.18
7.13
7.13
7.12
7.11
7.10
7.09
5.23
5.22
4.94
4.94
0.9
1.4
1.0
1.0
1.0
1.0
450
400
350
300
250
200
150
100
50
0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

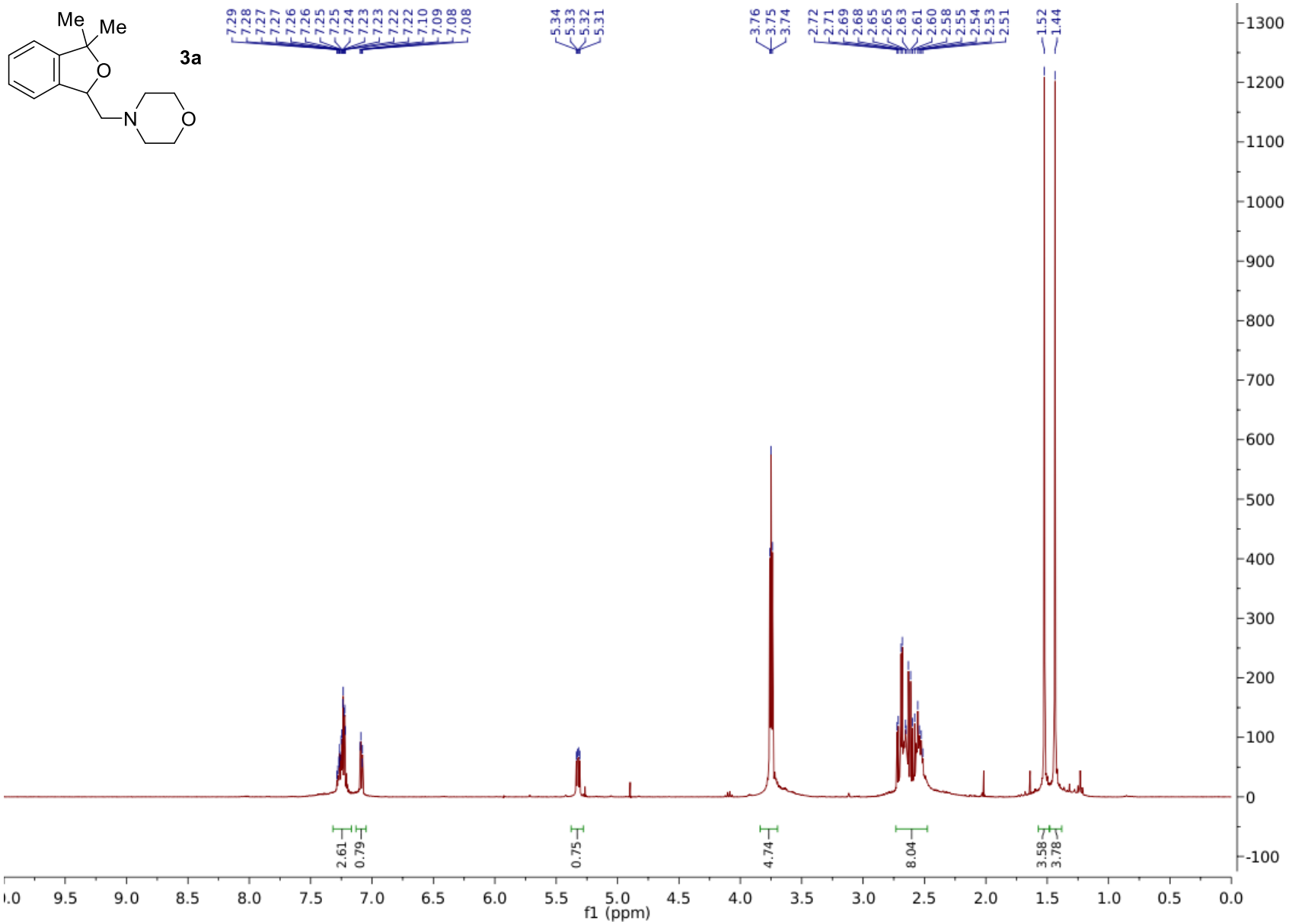

Br
CD3
D3-1v-iii
145.5
144.7
132.6
129.6
128.2
127.1
121.5
116.0
77.0
22.6
550
500
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

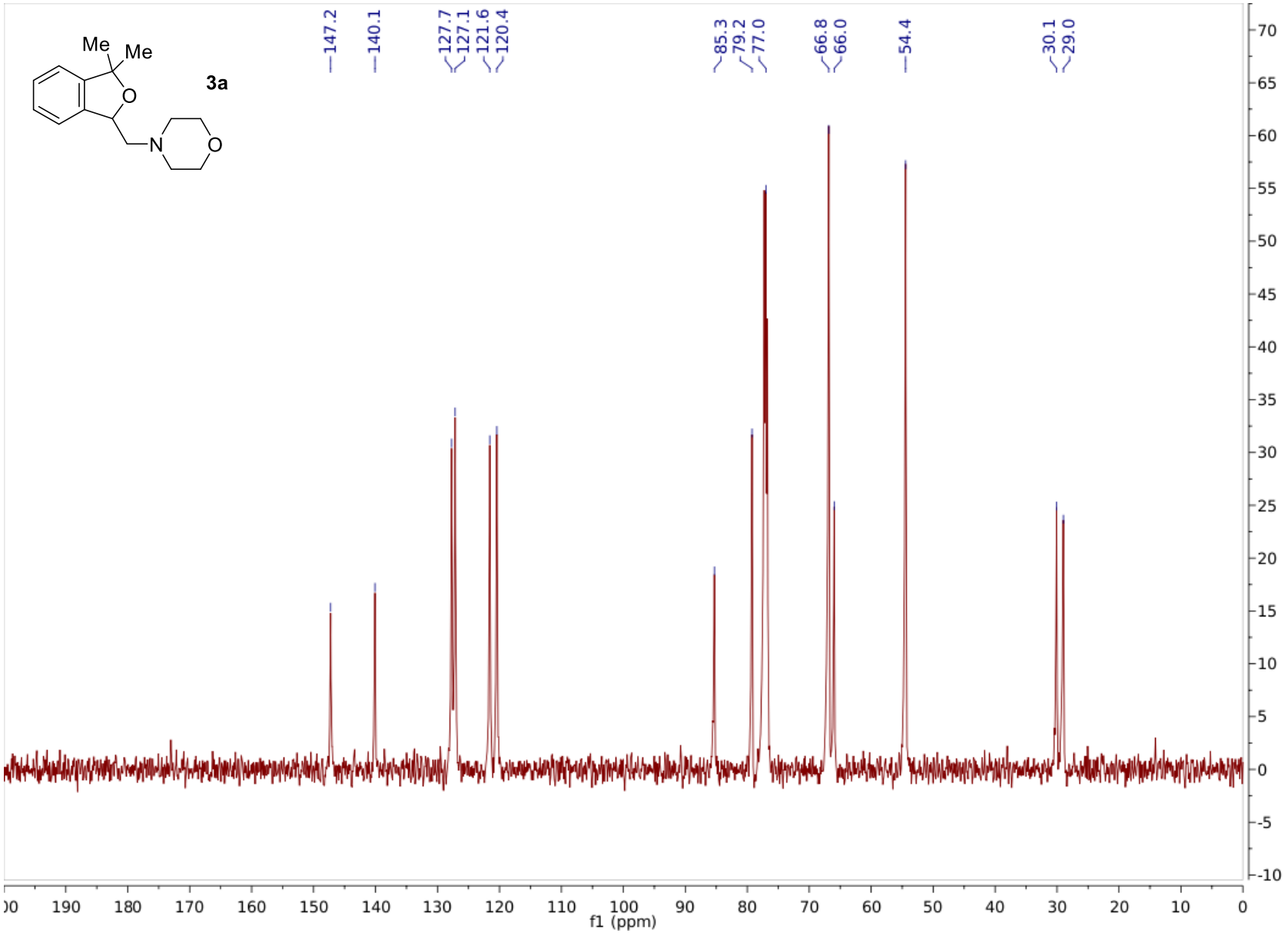

Me Me
OH
CD3
D3-1v
7.38
7.37
7.36
7.35
7.26
7.25
7.25
7.23
7.23
7.21
7.21
7.20
7.20
7.19
7.18
7.17
7.16
7.03
7.02
7.01
7.00
5.21
5.21
4.90
4.90
3.01
1.62
0.7
0.9
0.8
0.6
0.9
1.0
0.8
6.4
f1 (ppm)

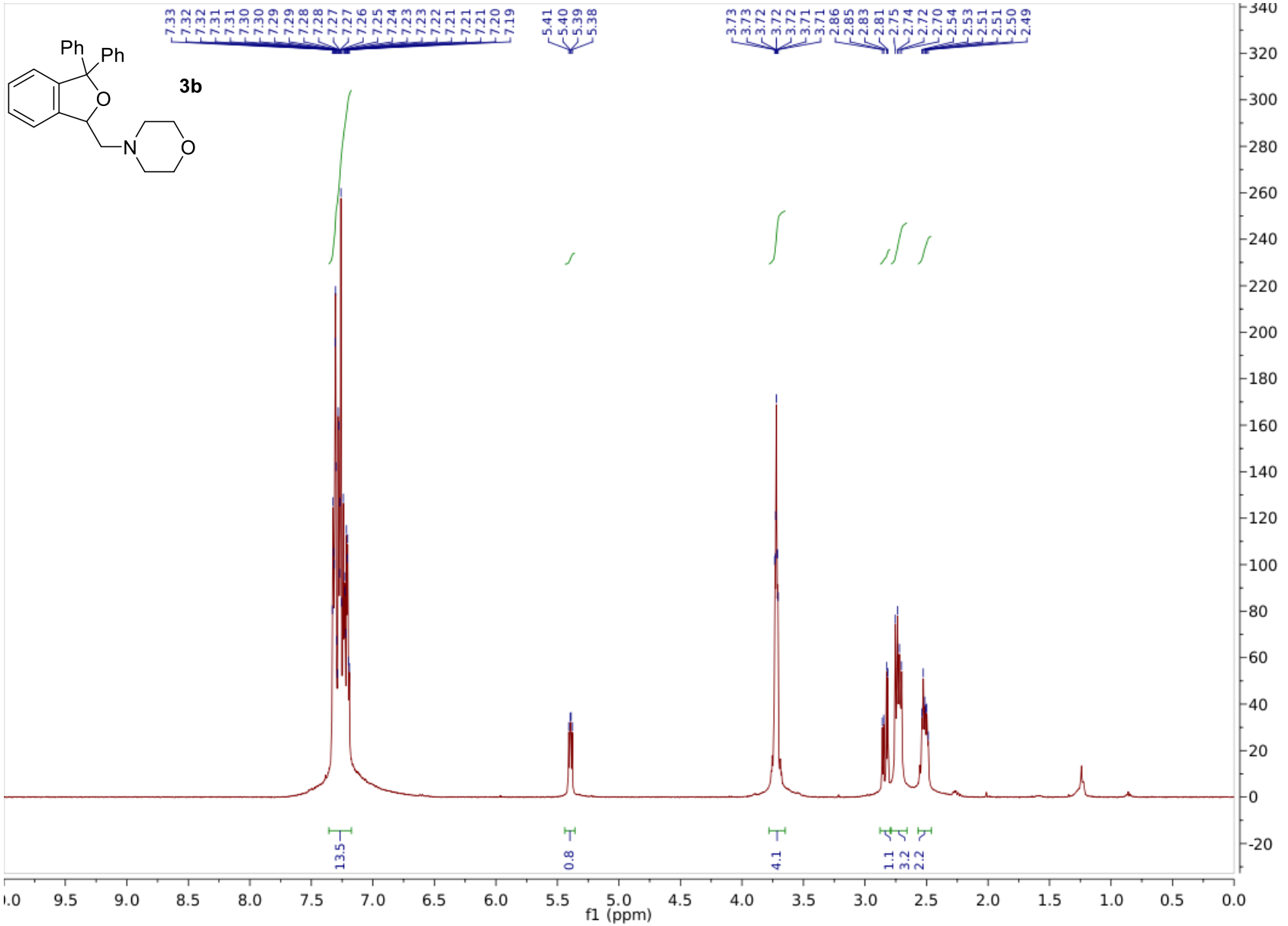

Me Me
OH
CD3
D3-1v
150.4
145.5
140.9
129.8
126.8
126.2
126.1
114.5
77.0
74.5
32.8
26.2
f1 (ppm)

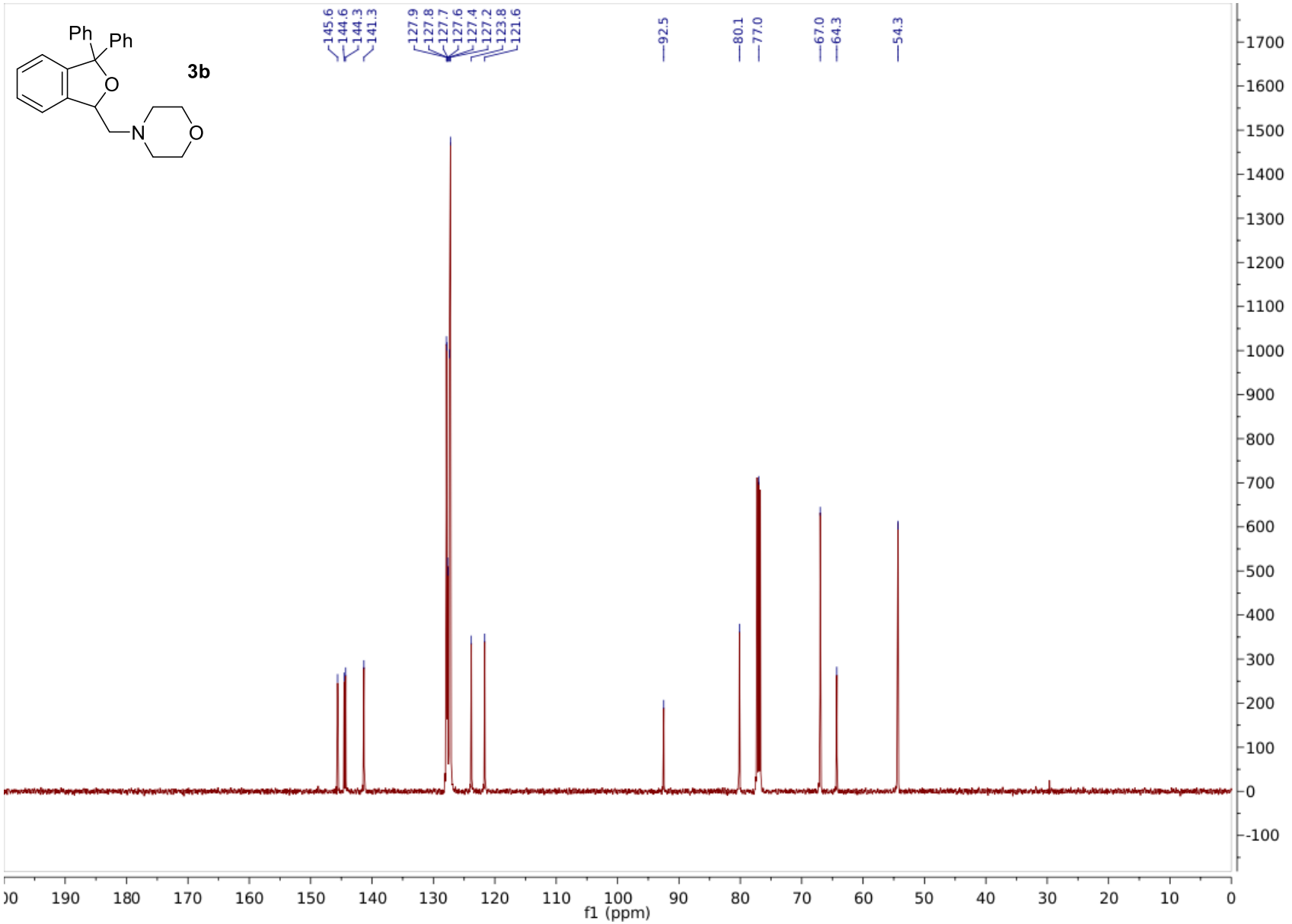

D3C CD3
OH
D6-1a
7.67
7.65
7.63
7.60
7.49
7.49
7.48
7.48
7.47
7.44
7.44
7.43
7.43
7.42
7.26
7.25
7.25
7.24
7.24
5.54
5.54
5.50
5.50
5.29
5.29
5.26
5.26
1.92
1.1
2.4
2.3
1.0
1.0
0.7
f1 (ppm)

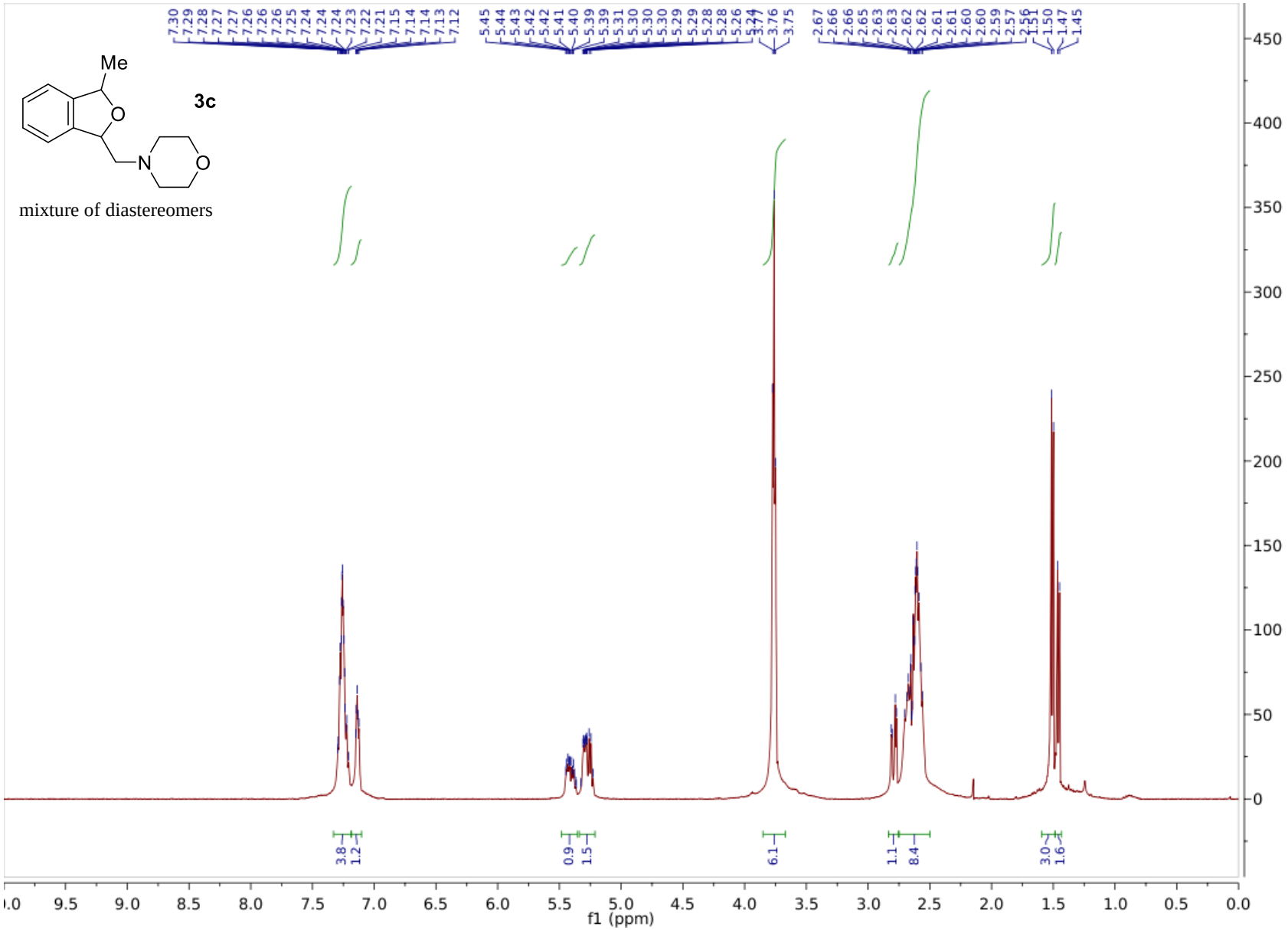

D3C CD3
OH
D6-1a
144.7
138.1
137.2
128.5
127.4
127.3
124.9
115.2
77.0
73.3
30.3
f1 (ppm)

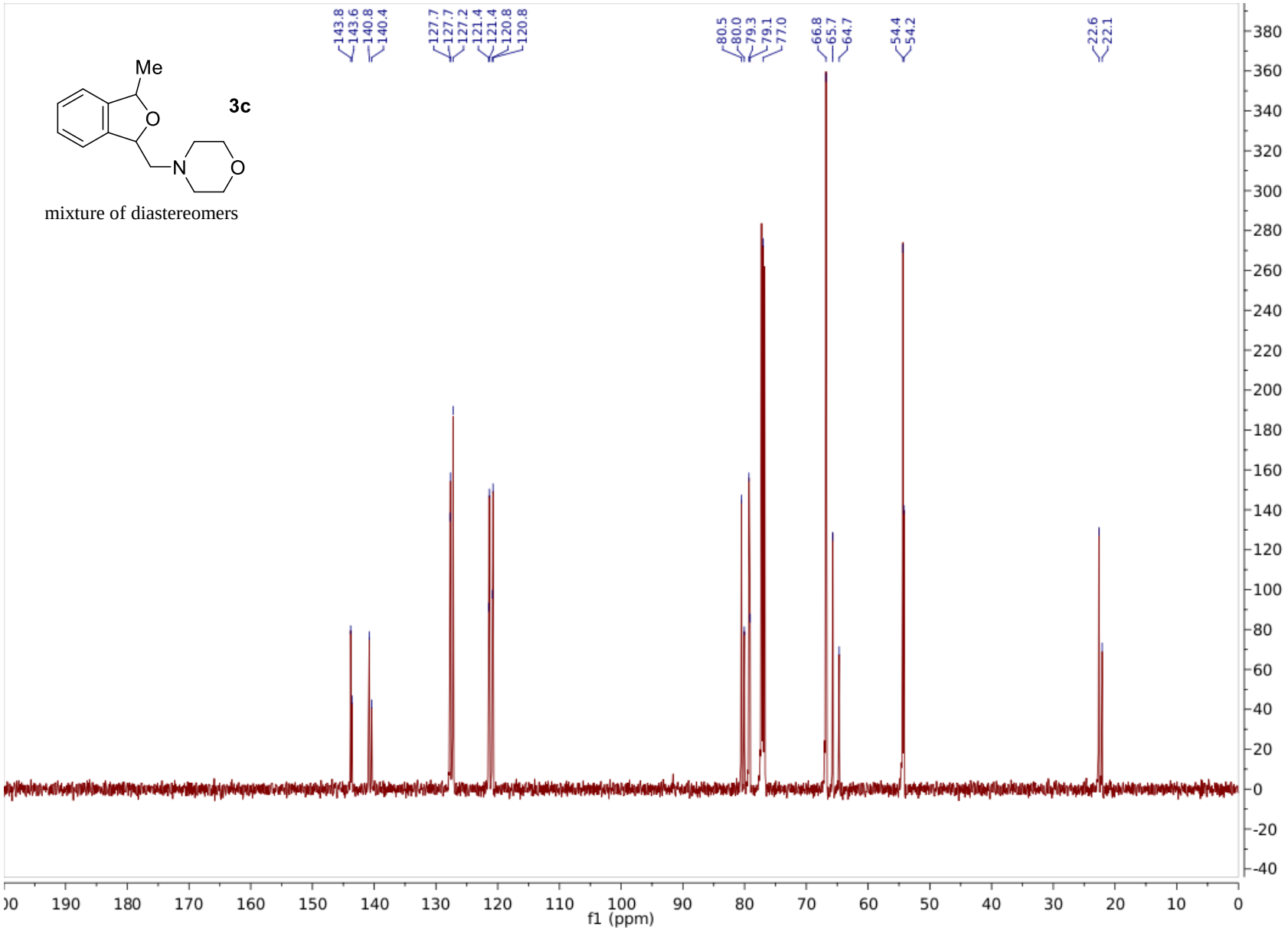

Me
Me
O
N
O
3a
7.29
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.23
7.23
7.22
7.22
7.10
7.09
7.08
7.08
5.34
5.33
5.32
5.31
3.76
3.75
3.74
2.72
2.71
2.69
2.68
2.65
2.65
2.63
2.61
2.60
2.58
2.55
2.54
2.53
2.51
1.52
1.44
2.61
0.79
0.75
4.74
8.04
3.58
3.78
f1 (ppm)

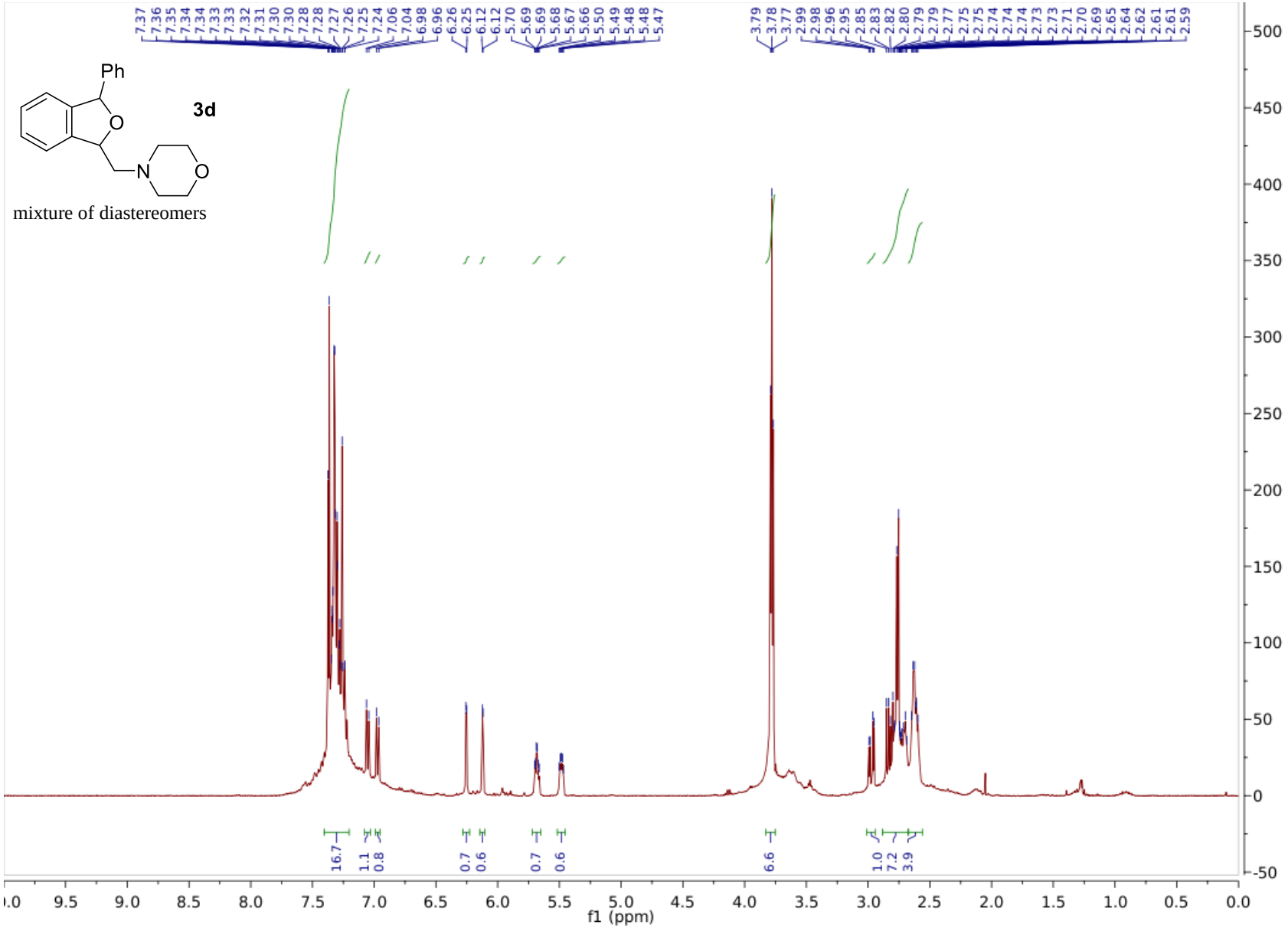

Me
Me
O
N
O
3a
147.2
140.1
127.7
127.1
121.6
120.4
85.3
79.2
77.0
66.8
66.0
54.4
30.1
29.0
f1 (ppm)

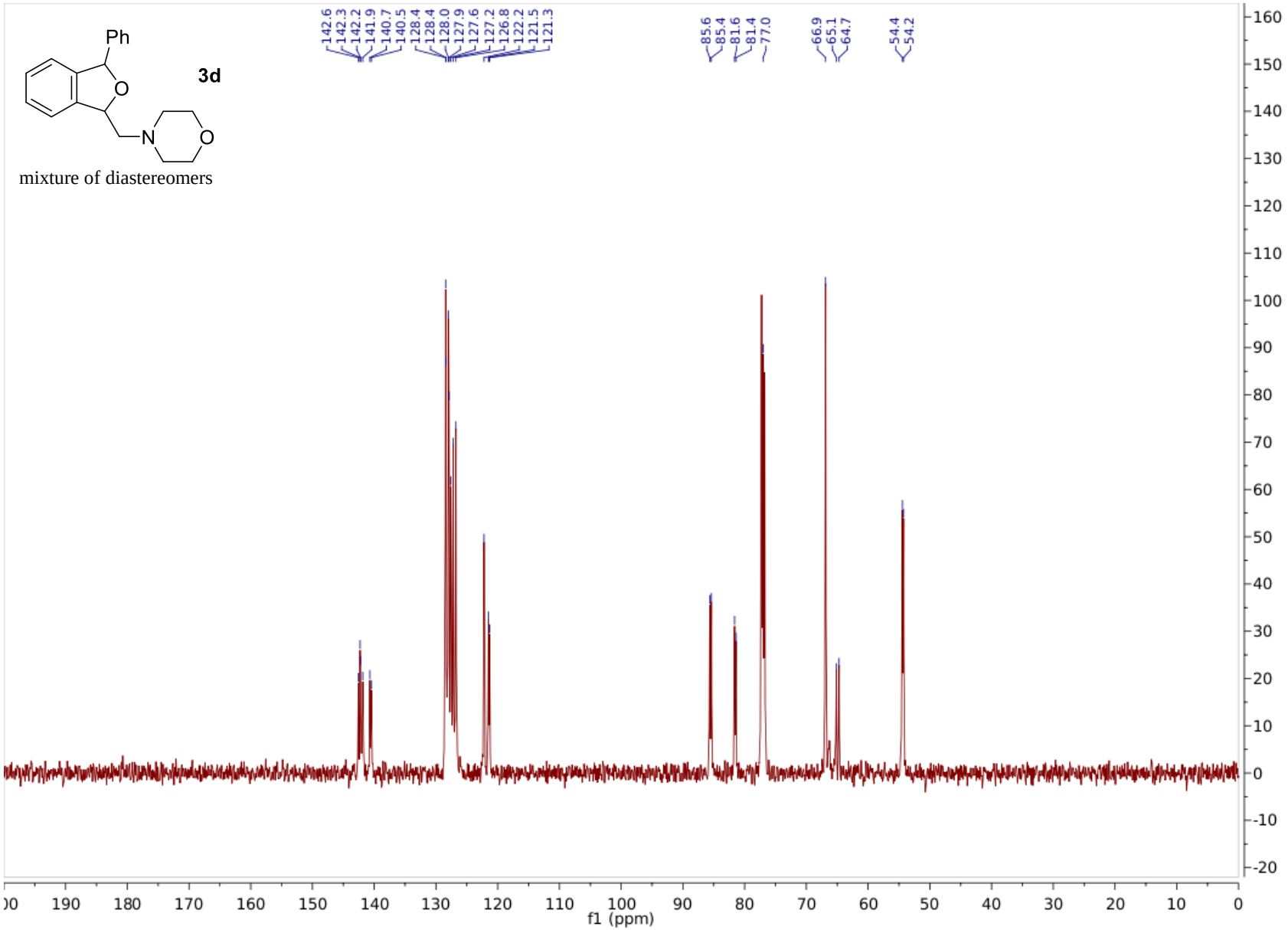

Ph
Ph
O
N
O
3b
7.33 7.32 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.24 7.23 7.23 7.22 7.21 7.21 7.21 7.20 7.19
5.41 5.40 5.39 5.38
3.73 3.73 3.72 3.72 3.72 3.71 3.71
2.86 2.85 2.83 2.81 2.75 2.74 2.72 2.70 2.54 2.53 2.51 2.51 2.50 2.49
13.5
0.8
4.1
1.1
3.2
2.2
f1 (ppm)

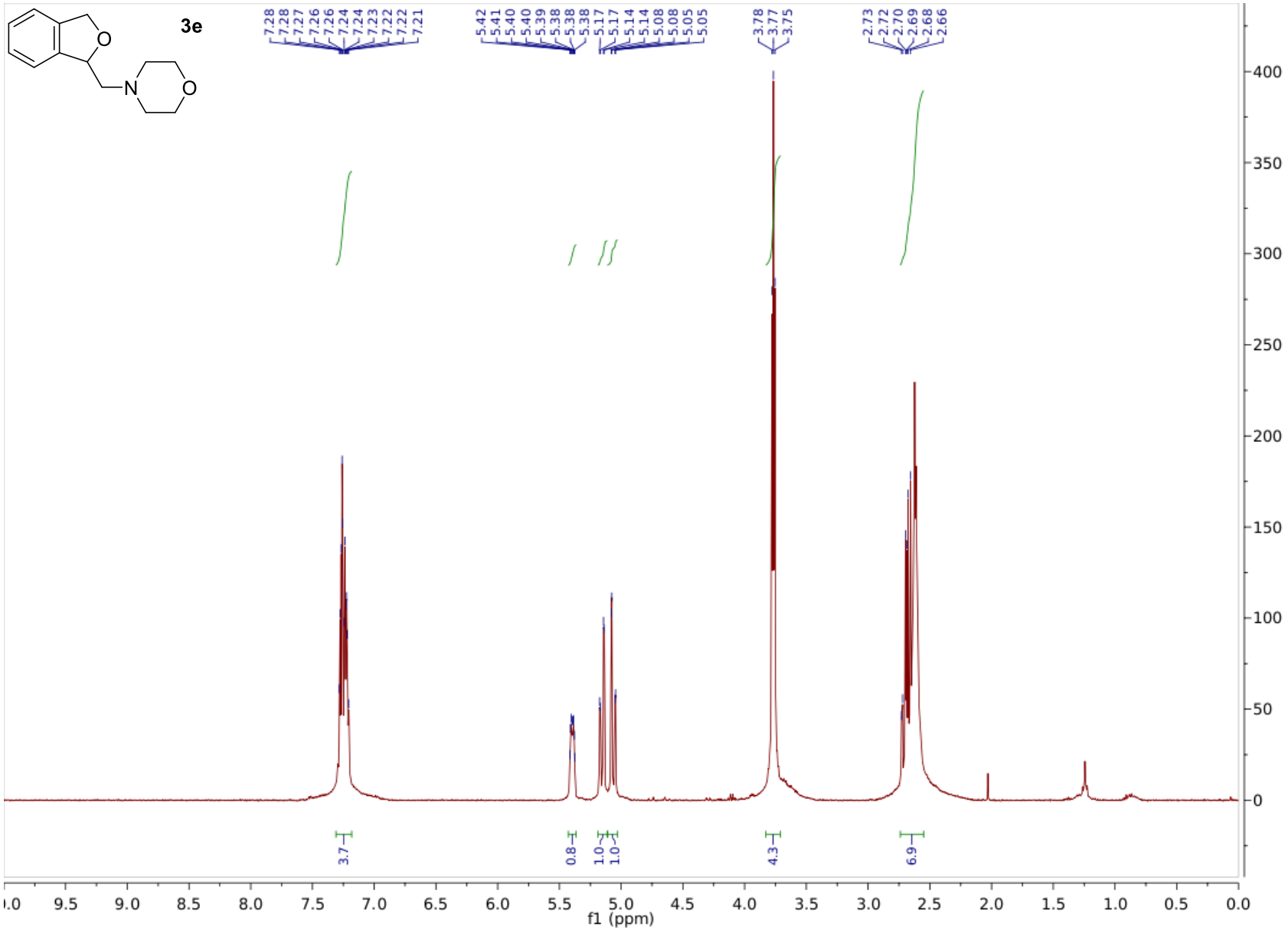

Ph
Ph
O
N
O
3b
145.6
144.6
144.3
141.3
127.9
127.8
127.7
127.6
127.4
127.2
123.8
121.6
92.5
80.1
77.0
67.0
64.3
54.3
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

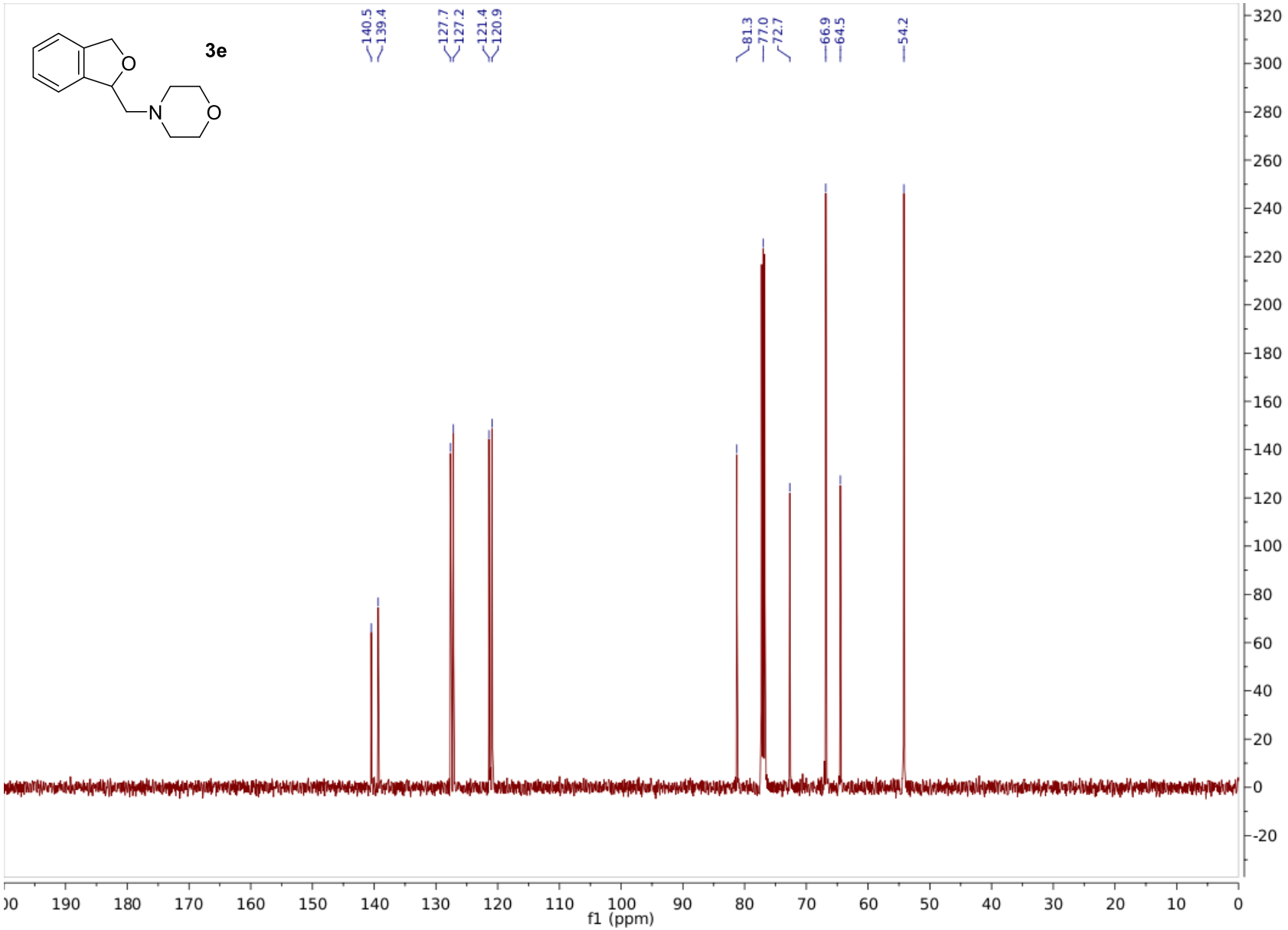

Me
O
N
O
3c
mixture of diastereomers
7.30 7.29 7.28 7.27 7.27 7.26 7.26 7.26 7.25 7.24 7.24 7.24 7.23 7.22 7.21 7.15 7.14 7.14 7.13 7.12
5.45 5.44 5.43 5.42 5.42 5.41 5.40 5.39 5.39 5.31 5.30 5.30 5.30 5.29 5.29 5.28 5.28 5.26 5.24 3.77 3.76 3.75
2.67 2.66 2.66 2.65 2.63 2.63 2.62 2.62 2.61 2.61 2.60 2.60 2.59 2.57 2.56 1.51 1.50 1.47 1.45
3.8 1.2 0.9 1.5 6.1 1.1 8.4 3.0 1.6
450 400 350 300 250 200 150 100 50 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)

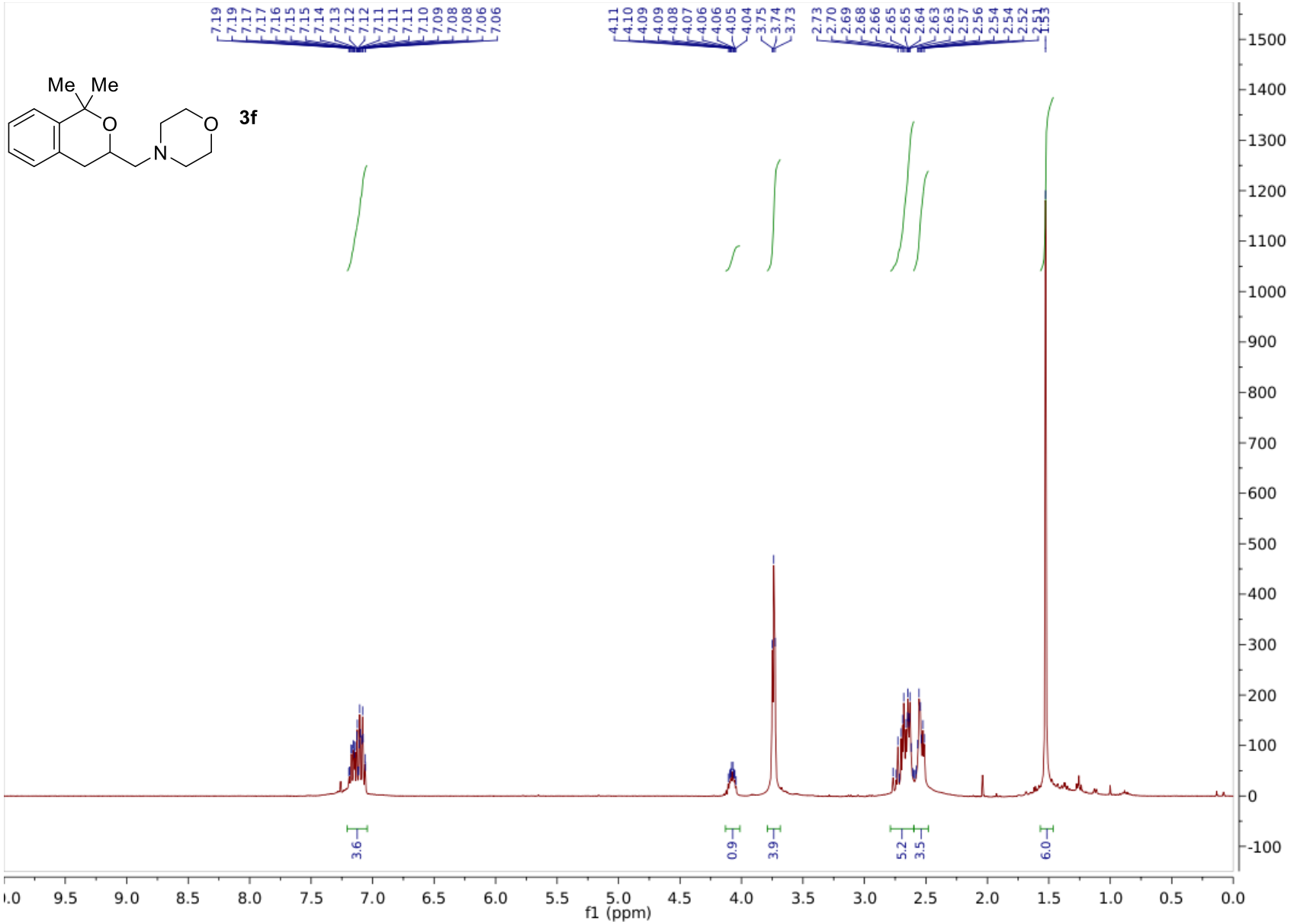

Me
O
N
O
3c
mixture of diastereomers
143.8
143.6
140.8
140.4
127.7
127.7
127.2
121.4
121.4
120.8
120.8
80.5
80.0
79.3
79.1
77.0
66.8
65.7
64.7
54.4
54.2
22.6
22.1
f1 (ppm)

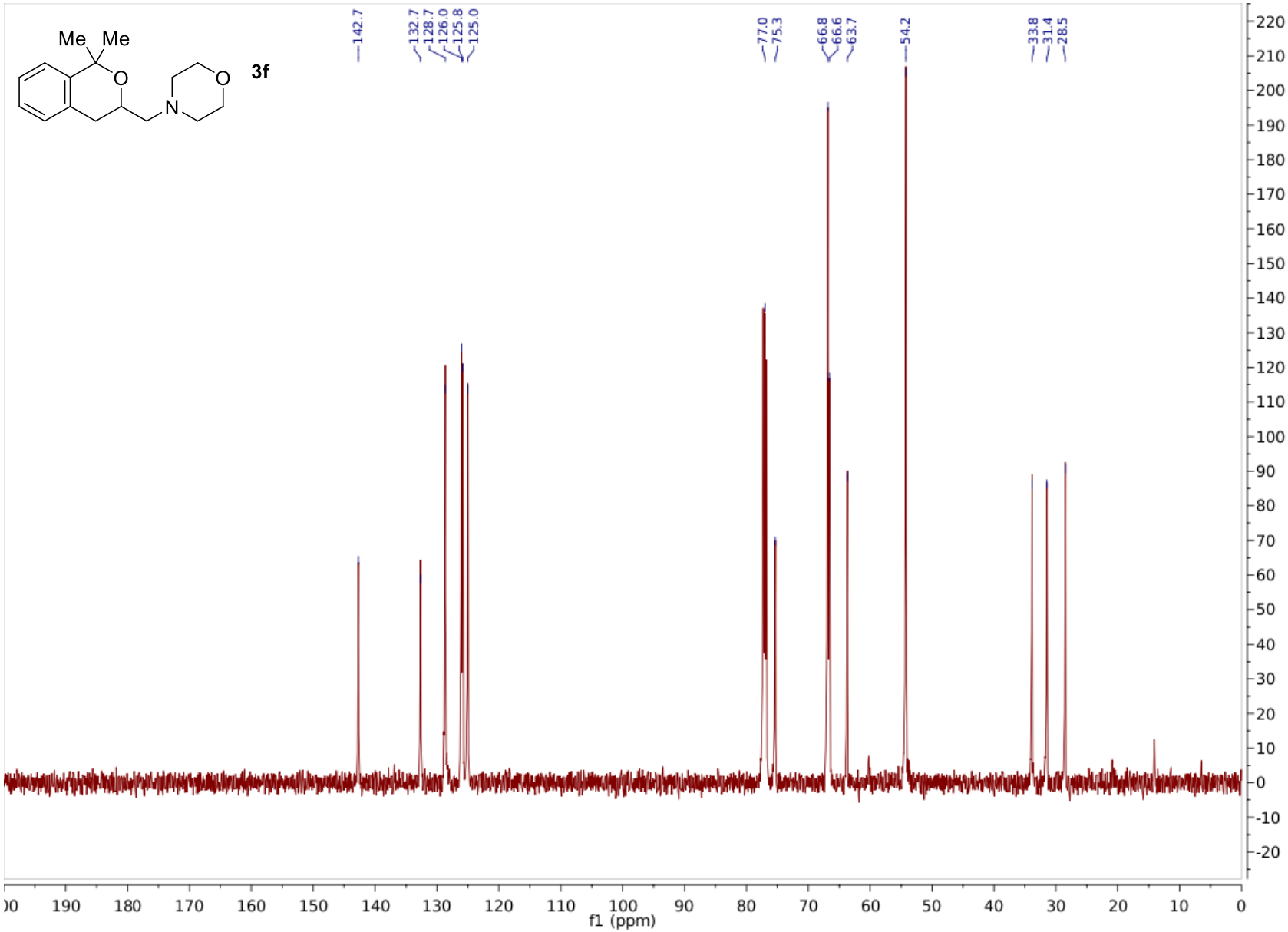

Ph
O
N
O
3d
mixture of diastereomers
f1 (ppm)

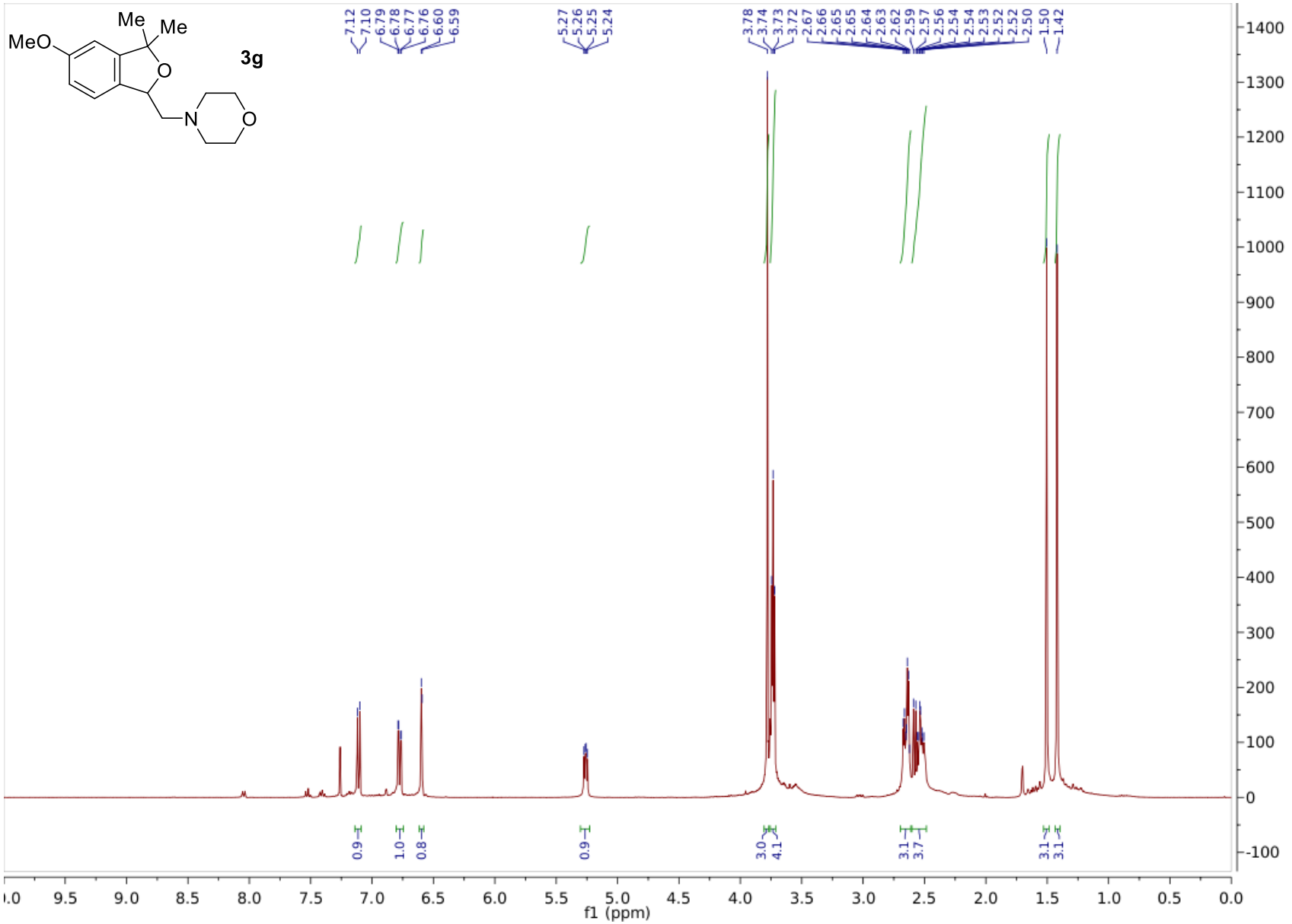

Ph
O
N
O
3d
mixture of diastereomers
142.6
142.3
142.2
141.9
140.7
140.5
128.4
128.4
128.0
127.9
127.6
127.2
126.8
122.2
121.5
121.3
85.6
85.4
81.6
81.4
77.0
66.9
65.1
64.7
54.4
54.2
f1 (ppm)

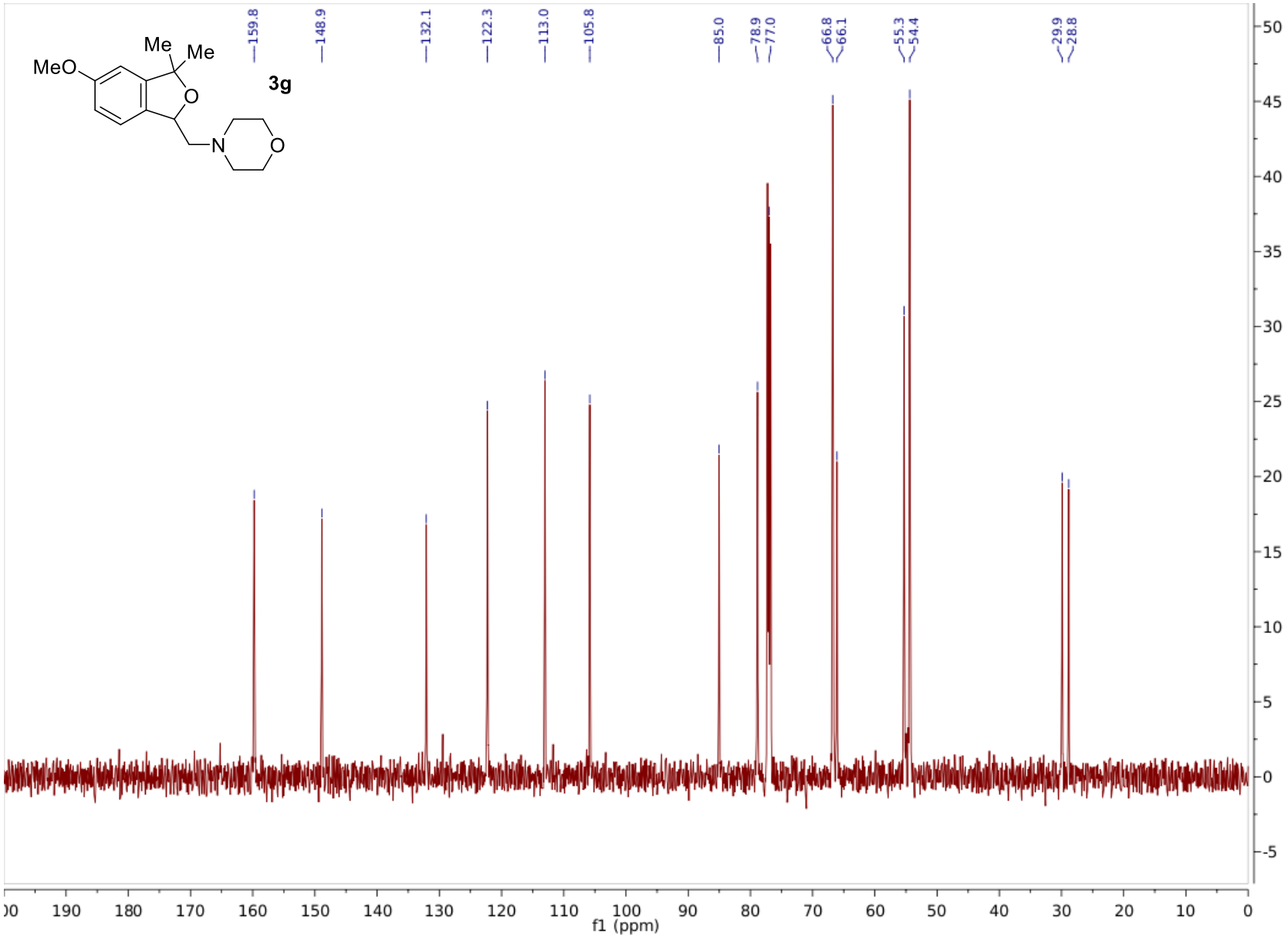

3e
7.28
7.28
7.27
7.26
7.26
7.24
7.24
7.23
7.22
7.22
7.21
5.42
5.41
5.40
5.40
5.39
5.38
5.38
5.38
5.17
5.17
5.14
5.14
5.08
5.08
5.05
5.05
3.78
3.77
3.75
2.73
2.72
2.70
2.69
2.68
2.66
3.7
0.8
1.0
1.0
4.3
6.9
f1 (ppm)

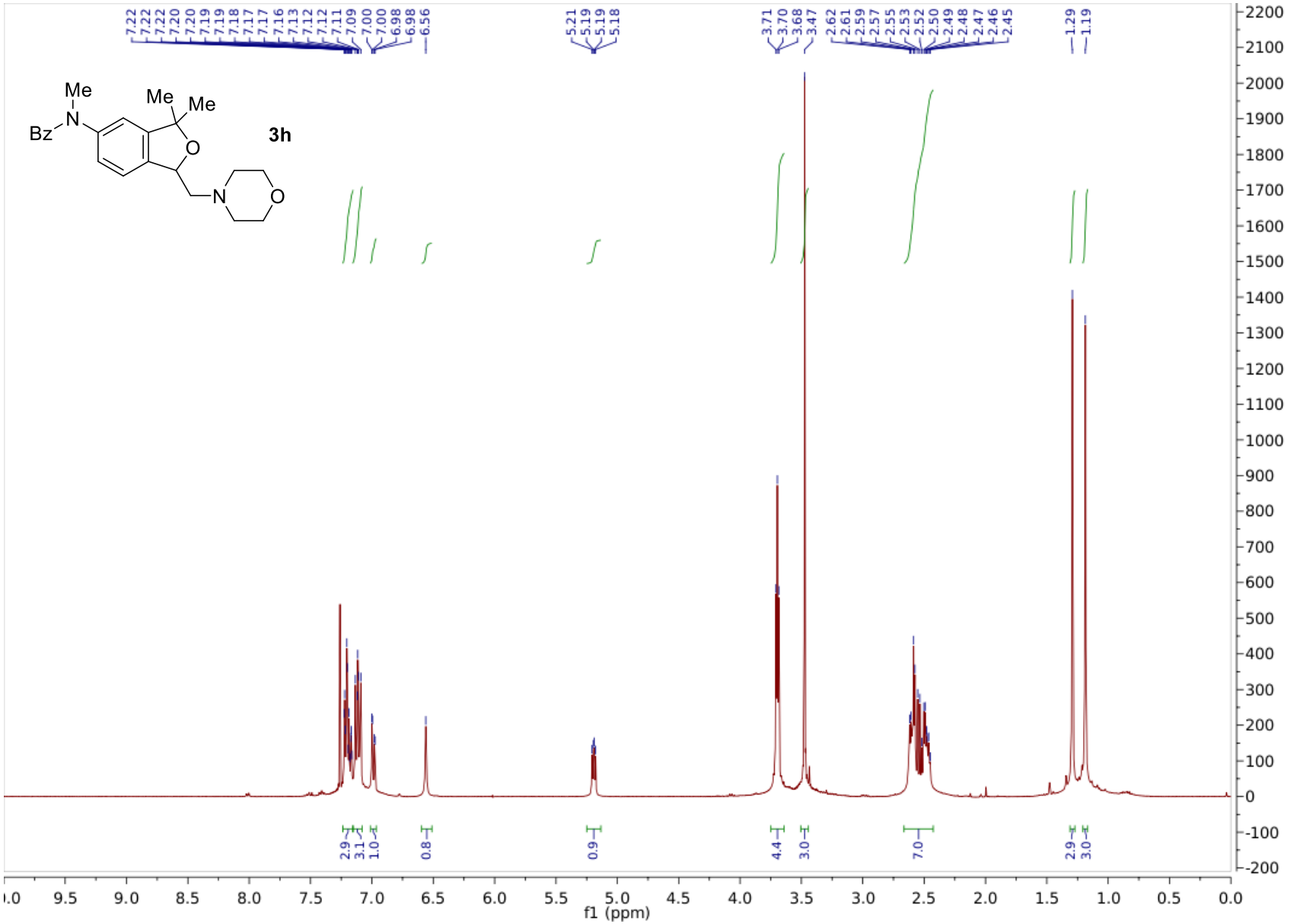

3e
140.5
139.4
127.7
127.2
121.4
120.9
81.3
77.0
72.7
66.9
64.5
54.2
f1 (ppm)

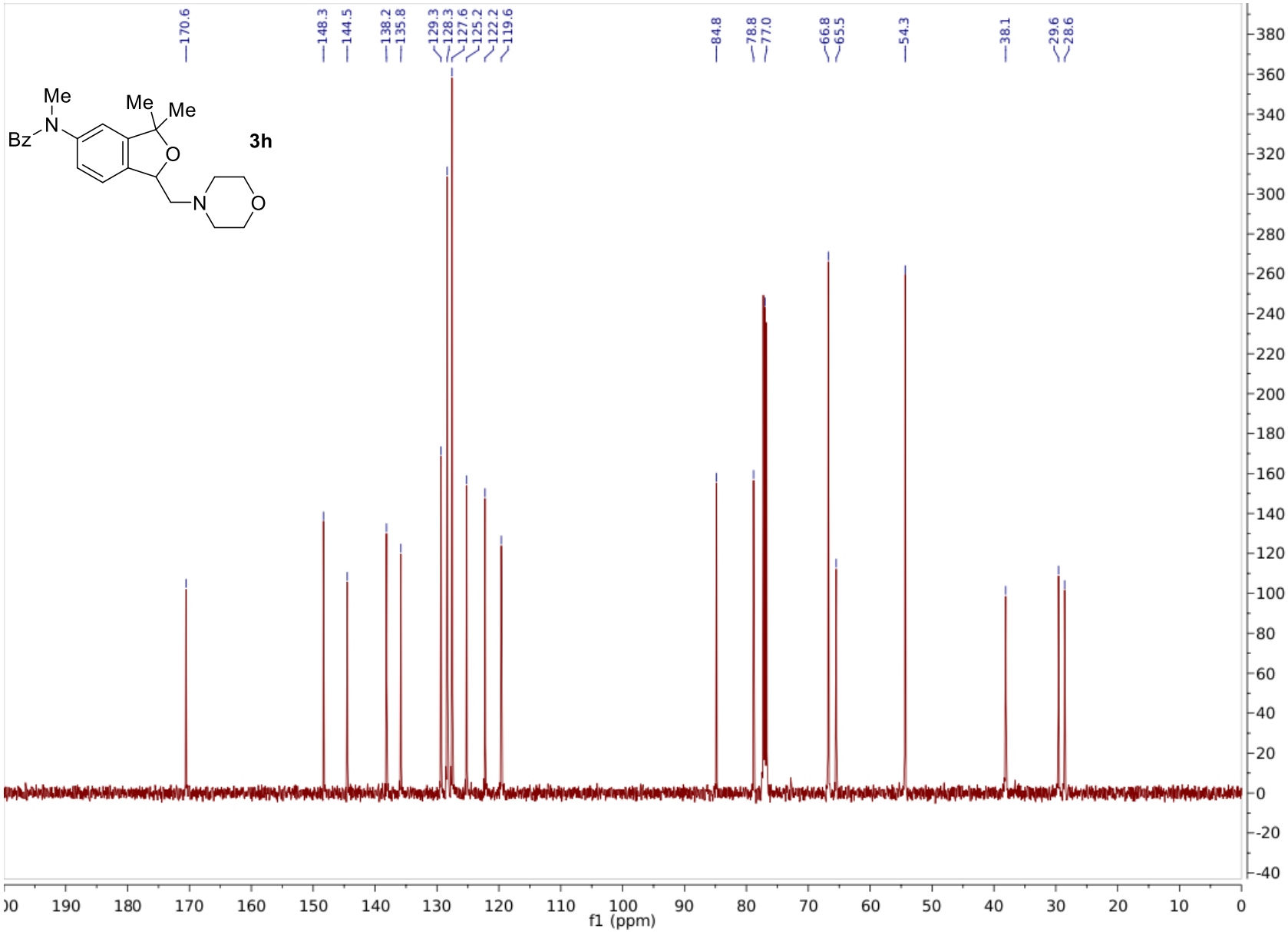

Me Me
O
O
N
3f
7.19 7.19 7.17 7.17 7.16 7.15 7.15 7.14 7.13 7.12 7.12 7.11 7.11 7.11 7.10 7.09 7.08 7.08 7.06 7.06
4.11 4.10 4.09 4.09 4.08 4.07 4.06 4.06 4.05 4.04 3.75 3.74 3.73
2.73 2.70 2.69 2.68 2.66 2.65 2.65 2.64 2.63 2.63 2.57 2.56 2.54 2.54 2.52 2.51 1.53
3.6
0.9
3.9
5.2
3.5
6.0
f1 (ppm)

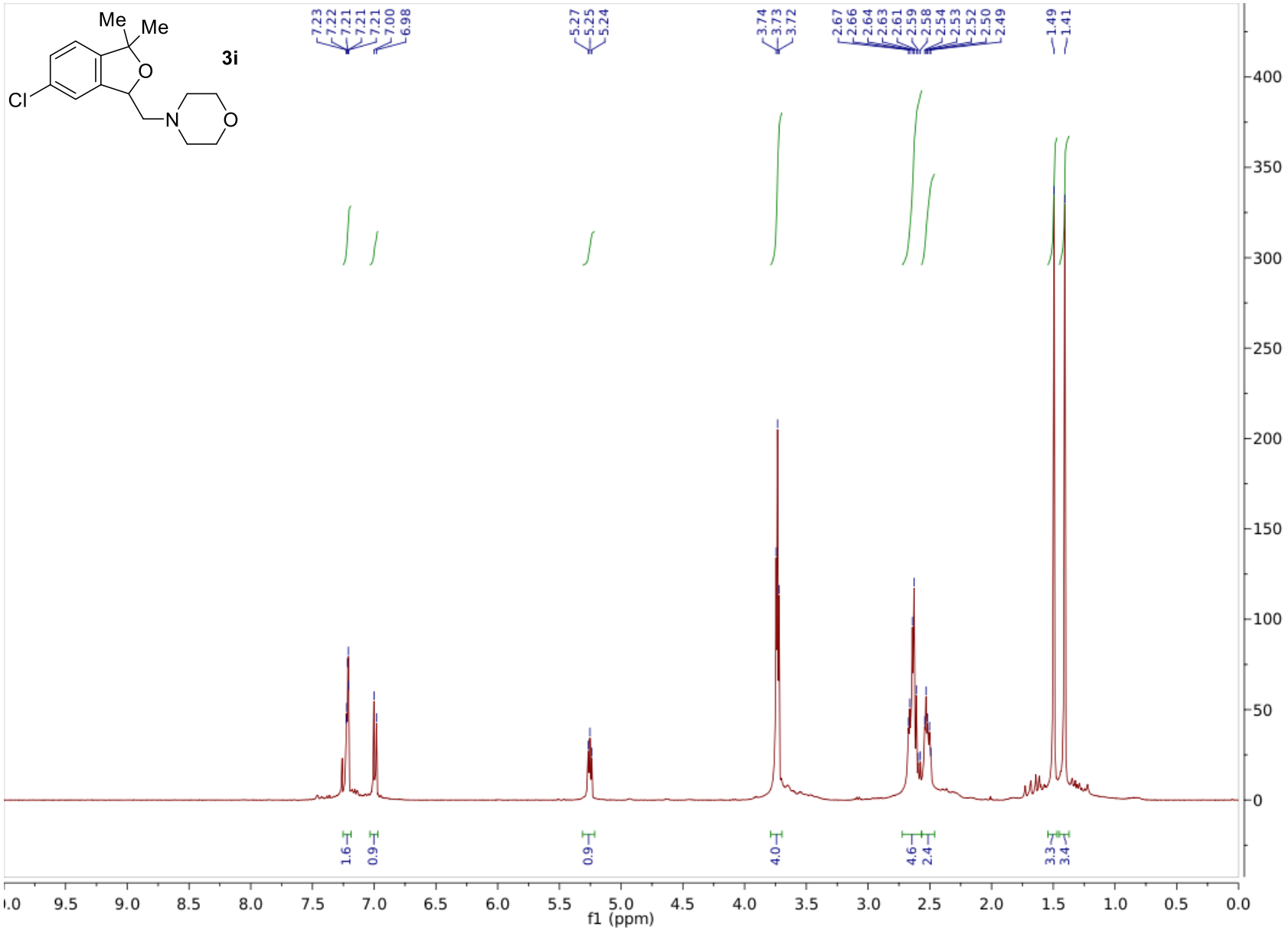

Me
Me
O
O
N
3f
142.7
132.7
128.7
126.0
125.8
125.0
77.0
75.3
66.8
66.6
63.7
54.2
33.8
31.4
28.5
f1 (ppm)

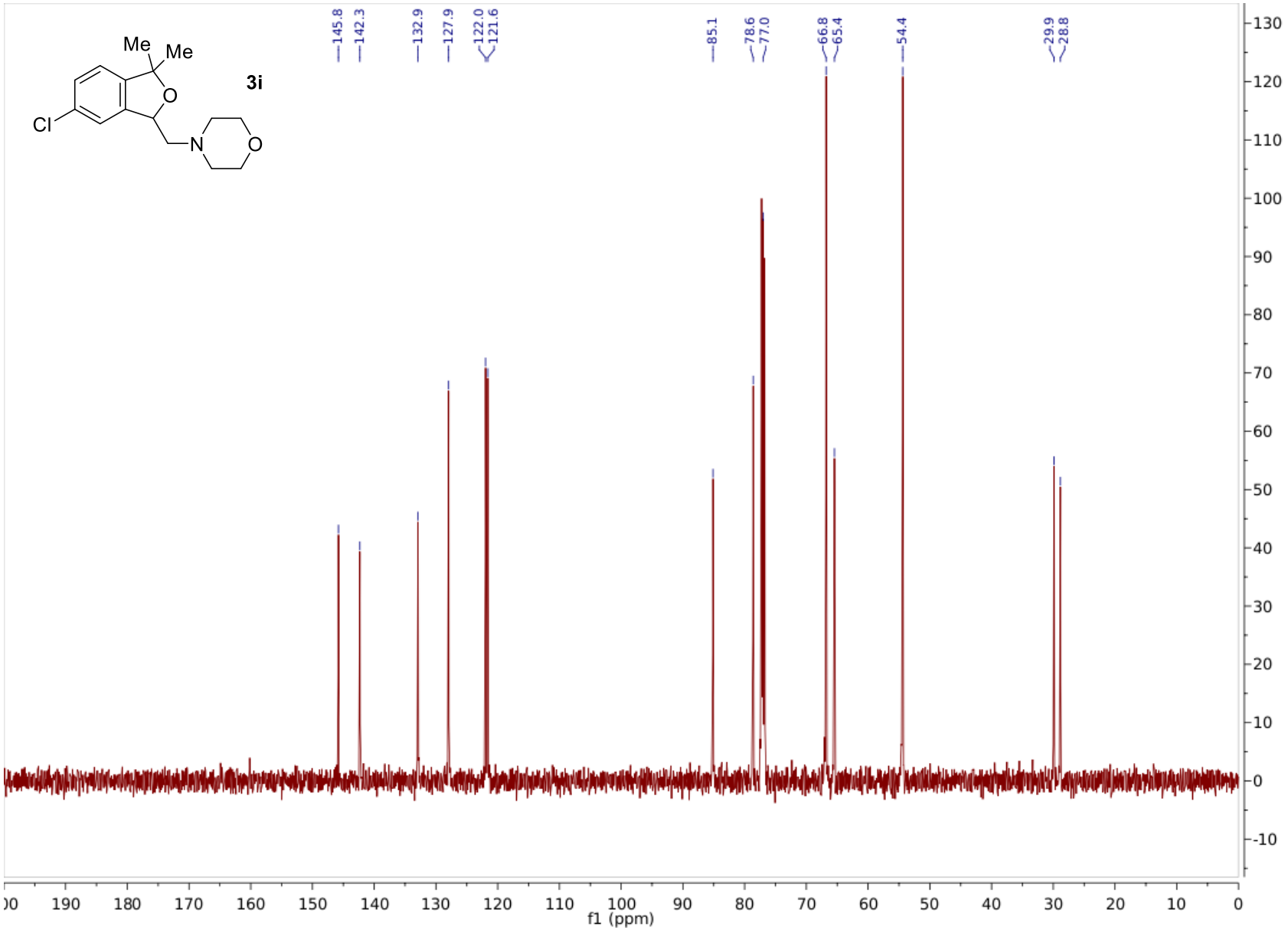

Me
Me
MeO
O
3g
N
O
7.12
7.10
6.79
6.78
6.77
6.76
6.60
6.59
5.27
5.26
5.25
5.24
3.78
3.74
3.73
3.72
2.67
2.66
2.65
2.65
2.64
2.63
2.62
2.59
2.57
2.56
2.54
2.54
2.53
2.52
2.52
2.50
1.50
1.42
0.9
1.0
0.8
0.9
3.0
4.1
3.1
3.7
3.1
3.1
f1 (ppm)

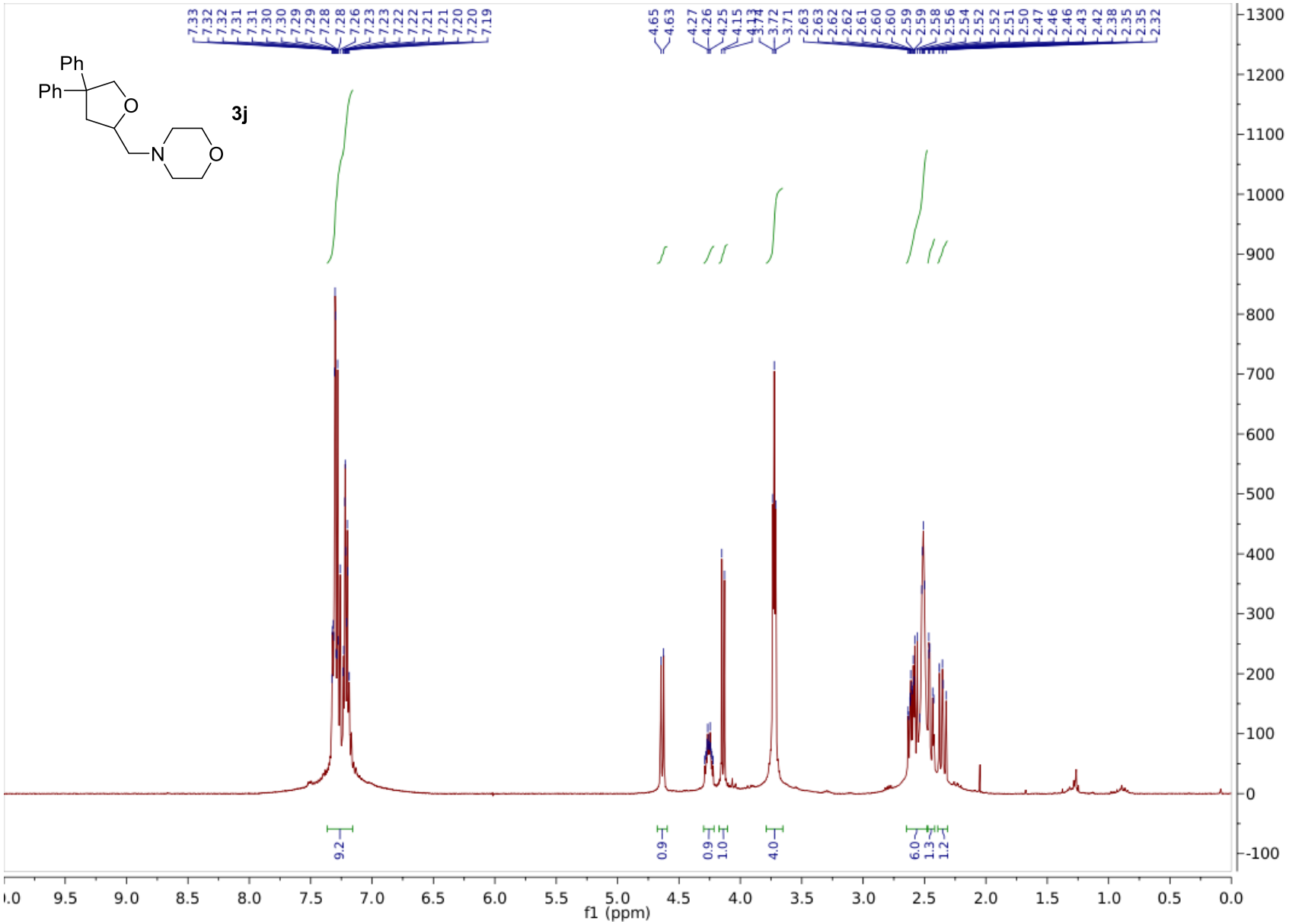

MeO
Me
Me
O
N
O
3g
159.8
148.9
132.1
122.3
113.0
105.8
85.0
78.9
77.0
66.8
66.1
55.3
54.4
29.9
28.8
f1 (ppm)

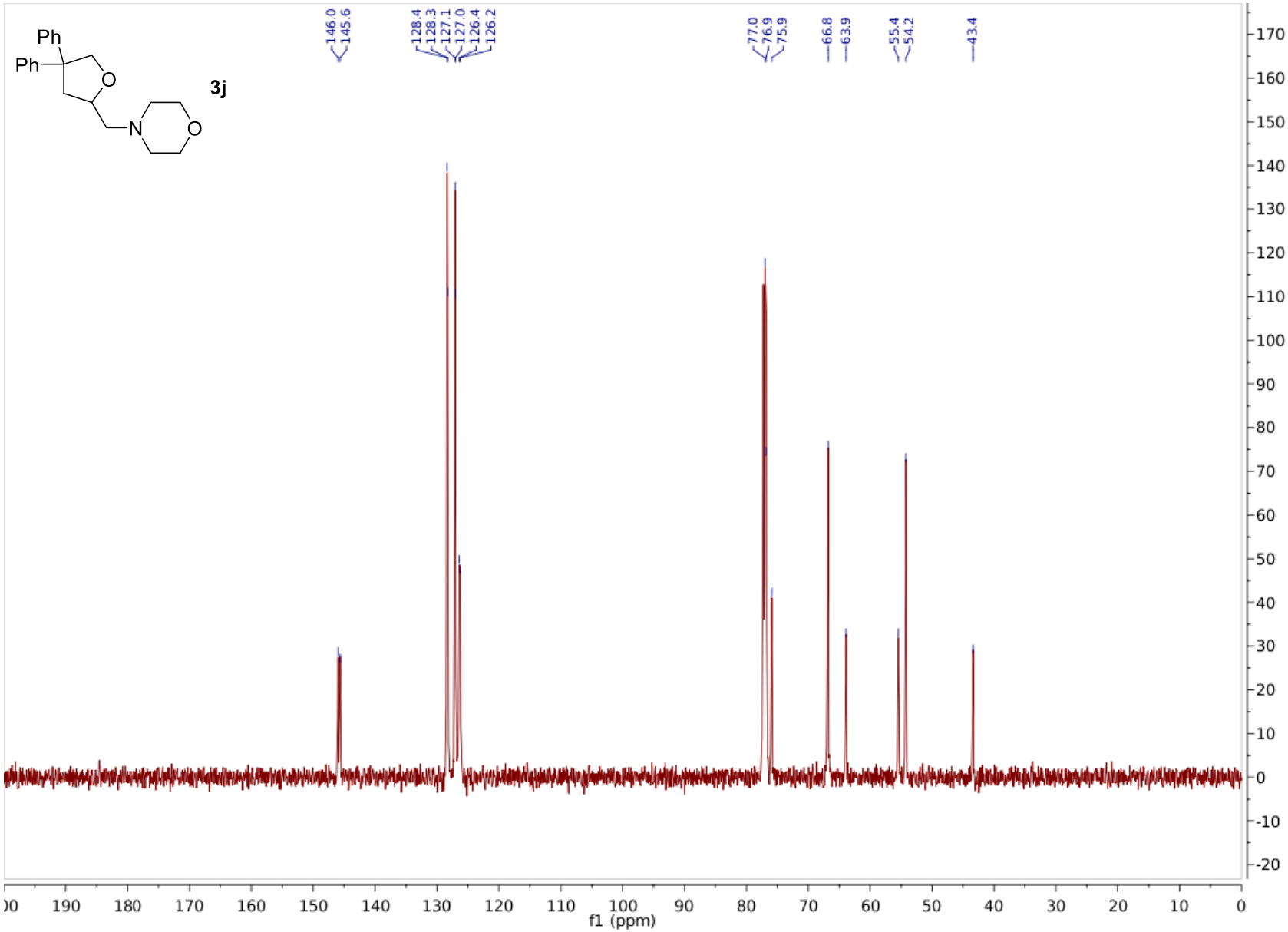

Me
Me
Me
Bz
N
O
N
O
3h
7.22
7.22
7.22
7.20
7.20
7.19
7.19
7.18
7.17
7.17
7.16
7.13
7.12
7.12
7.11
7.09
7.00
7.00
6.98
6.98
6.56
5.21
5.19
5.19
5.18
3.71
3.70
3.68
3.47
2.62
2.61
2.59
2.57
2.55
2.53
2.52
2.50
2.49
2.48
2.47
2.46
2.45
1.29
1.19
2.9
3.1
1.0
0.8
0.9
4.4
3.0
7.0
2.9
3.0
f1 (ppm)

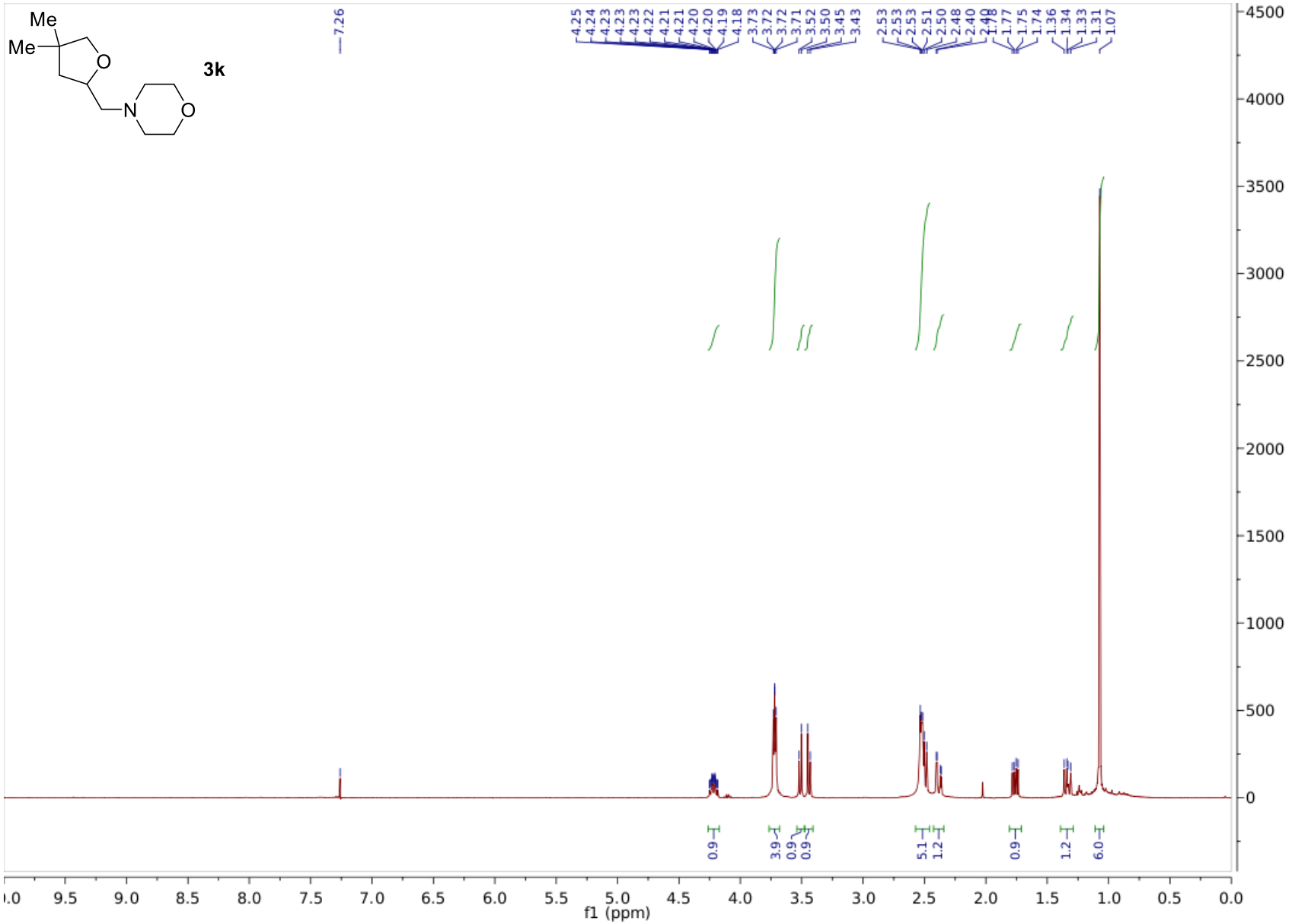

170.6
148.3
144.5
138.2
135.8
129.3
128.3
127.6
125.2
122.2
119.6
84.8
78.8
77.0
66.8
65.5
54.3
38.1
29.6
28.6
Me
N
Bz
Me
Me
O
N
O
3h
f1 (ppm)

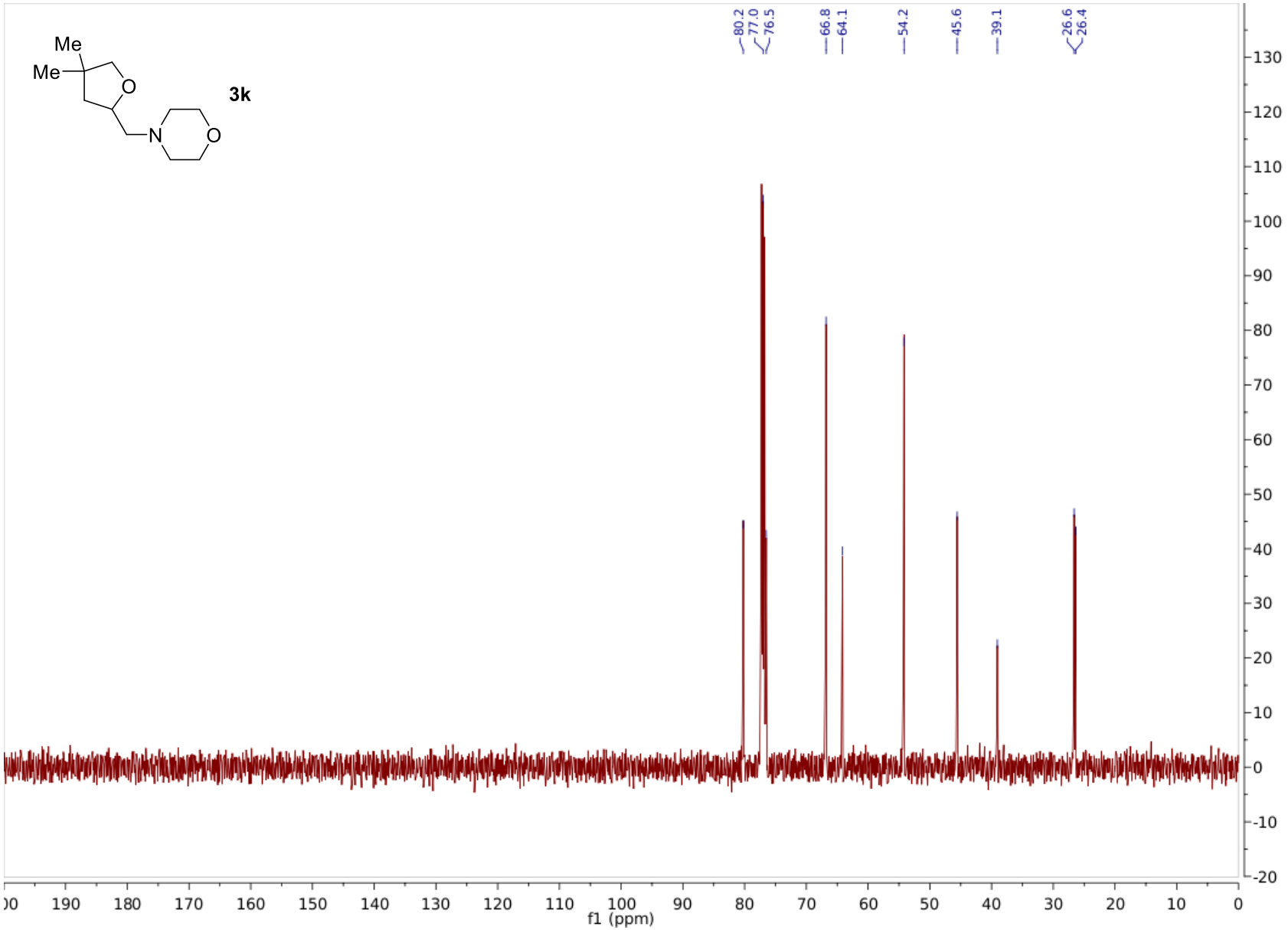

Me
Me
O
Cl
N
O
3i
7.23
7.22
7.21
7.21
7.21
7.00
6.98
5.27
5.25
5.24
3.74
3.73
3.72
2.67
2.66
2.64
2.63
2.61
2.59
2.58
2.54
2.53
2.52
2.50
2.49
1.49
1.41
1.6
0.9
0.9
4.0
4.6
2.4
3.3
3.4
f1 (ppm)

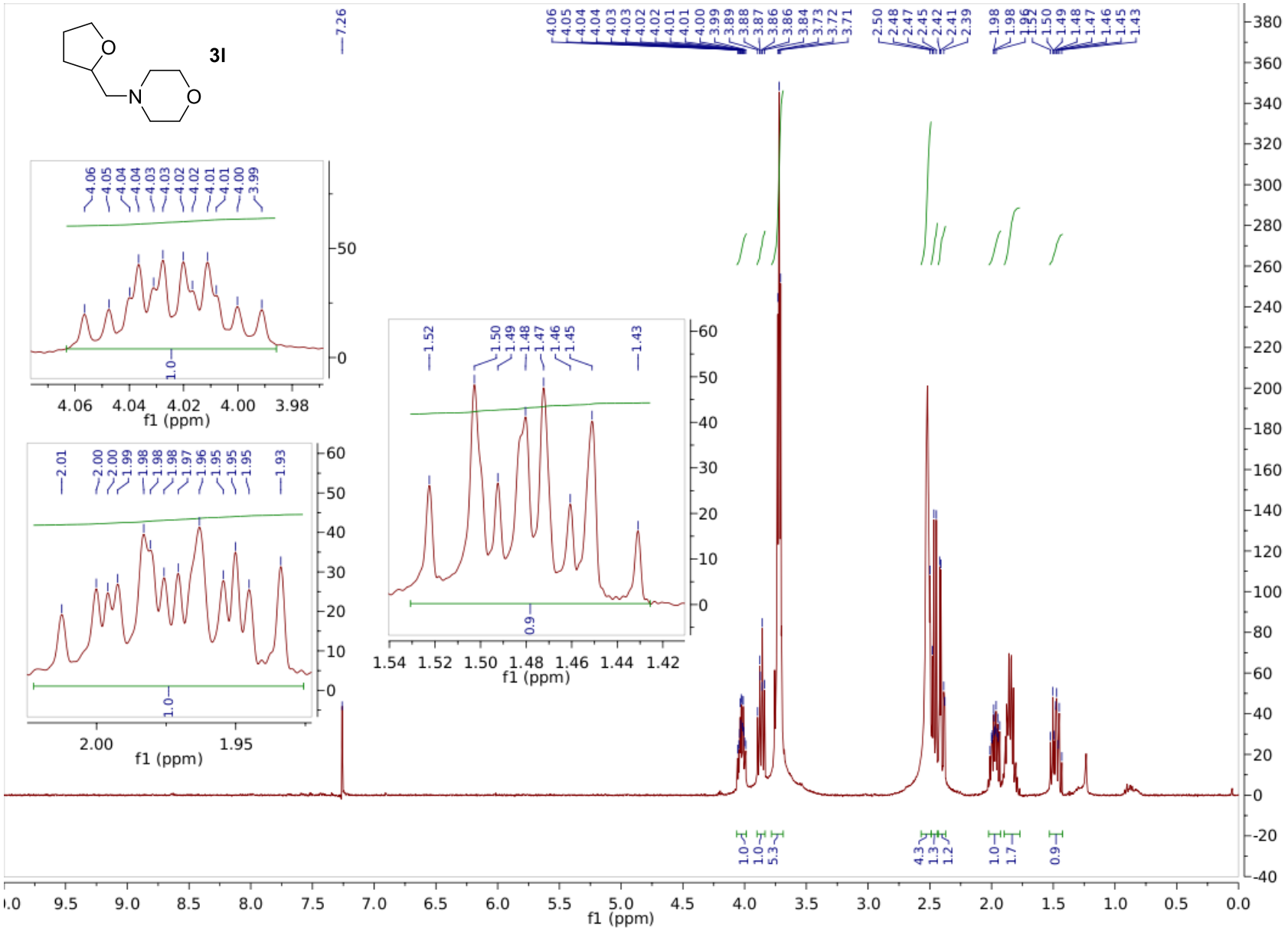

3i
Me
Me
O
Cl
N
O
145.8
142.3
132.9
127.9
122.0
121.6
85.1
78.6
77.0
66.8
65.4
54.4
29.9
28.8
f1 (ppm)

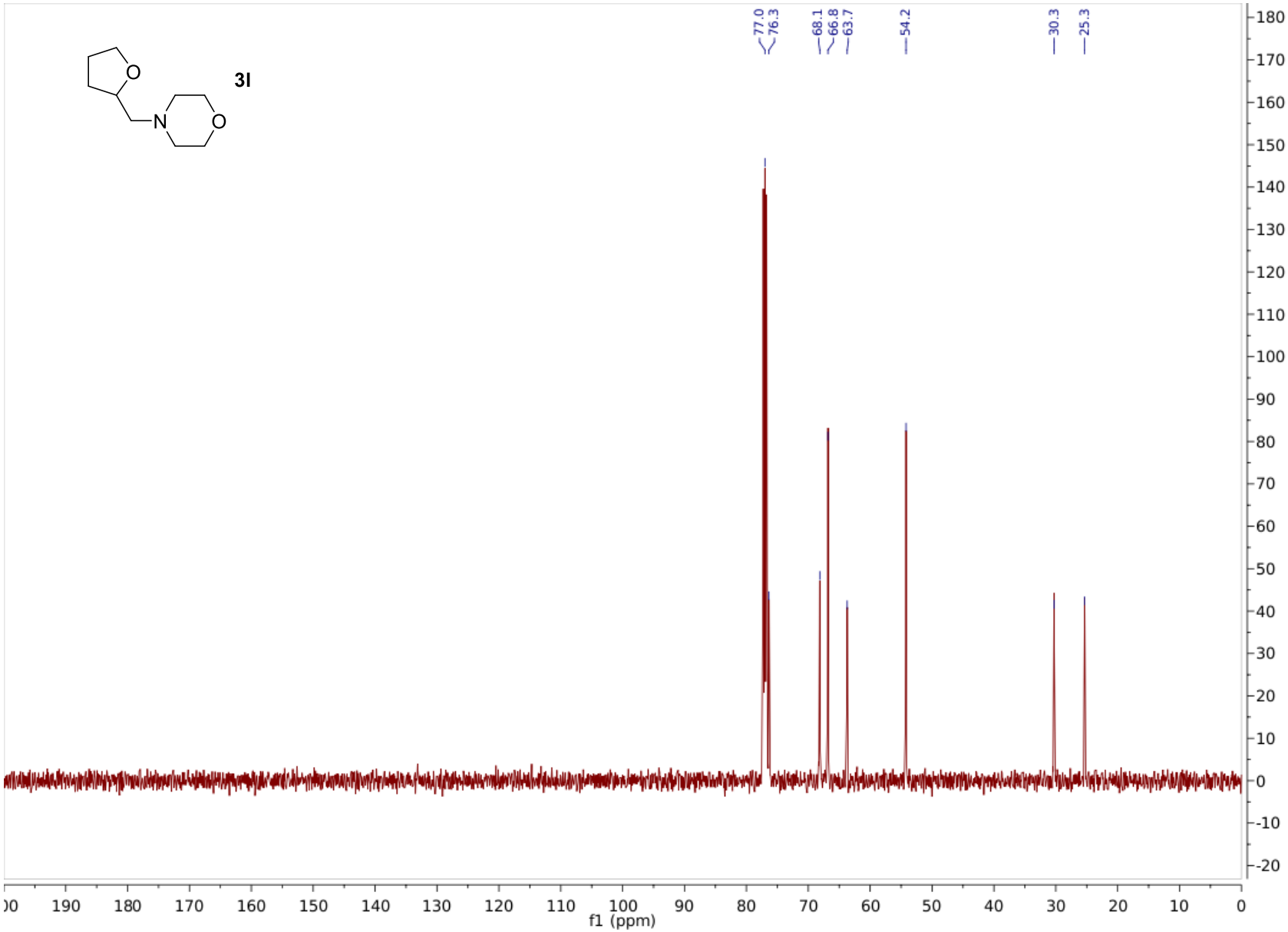

Ph
Ph
O
N
O
3j
f1 (ppm)

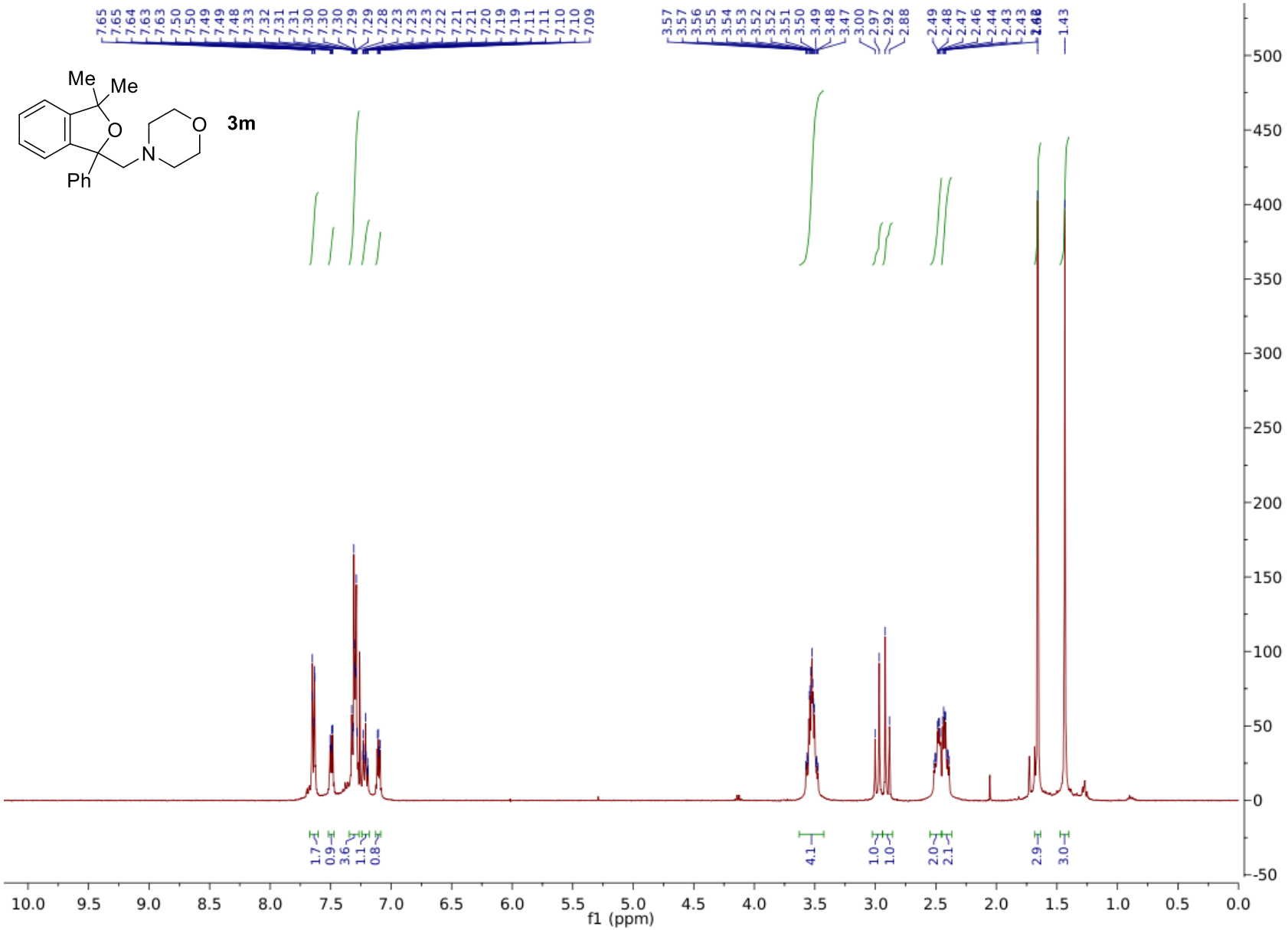

Ph
Ph
O
N
O
3j
146.0
145.6
128.4
128.3
127.1
127.0
126.4
126.2
77.0
76.9
75.9
66.8
63.9
55.4
54.2
43.4
f1 (ppm)

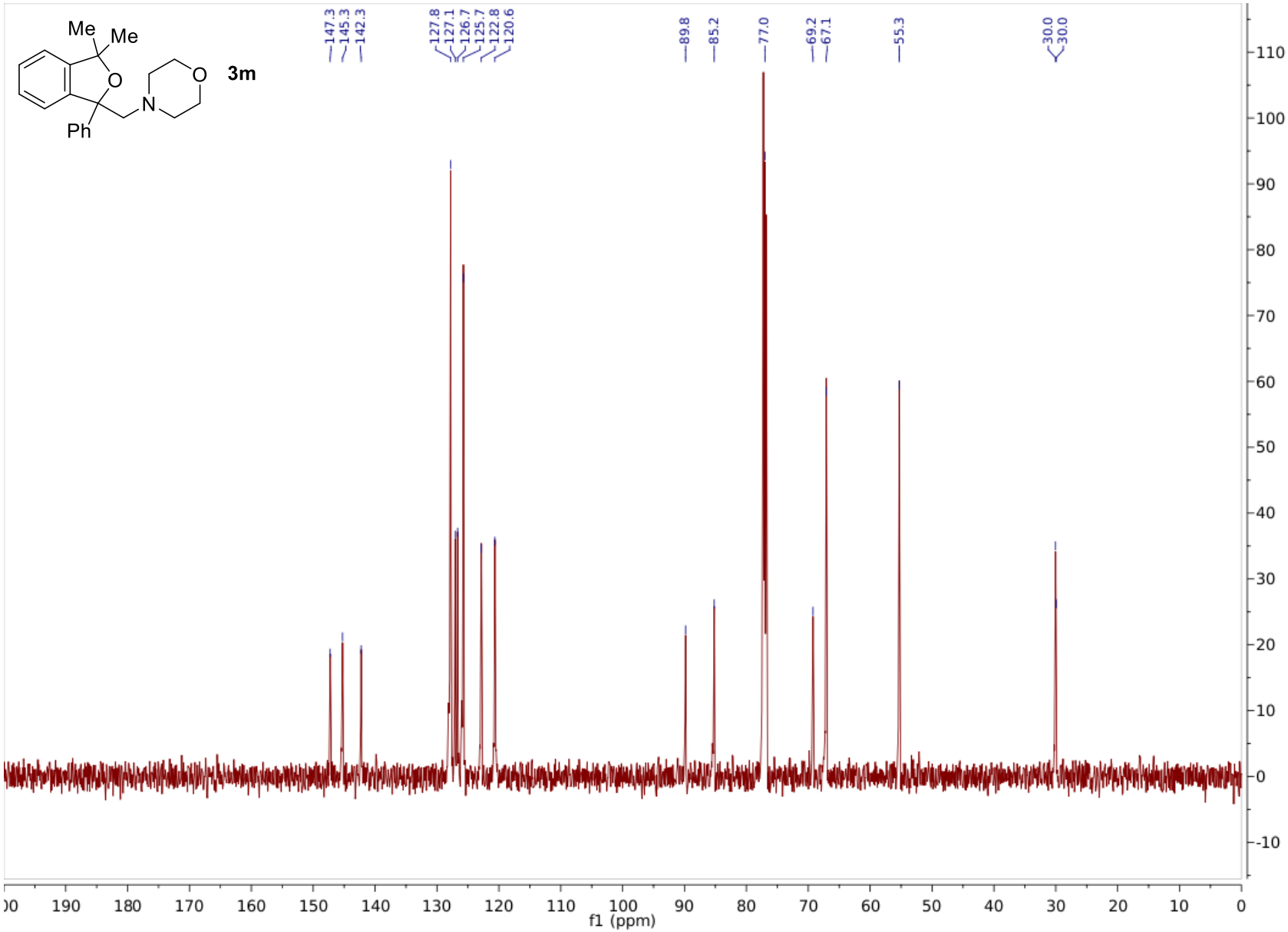

Me
Me
O
N
O
3k
7.26
4.25
4.24
4.23
4.23
4.23
4.22
4.21
4.21
4.20
4.20
4.19
4.18
3.73
3.72
3.72
3.71
3.52
3.50
3.45
3.43
2.53
2.53
2.53
2.51
2.50
2.48
2.40
2.40
1.78
1.77
1.75
1.74
1.36
1.34
1.33
1.31
1.07
0.9
3.9
0.9
0.9
5.1
1.2
0.9
1.2
6.0
4500
4000
3500
3000
2500
2000
1500
1000
500
0
9.5
9.0
8.5
8.0
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)

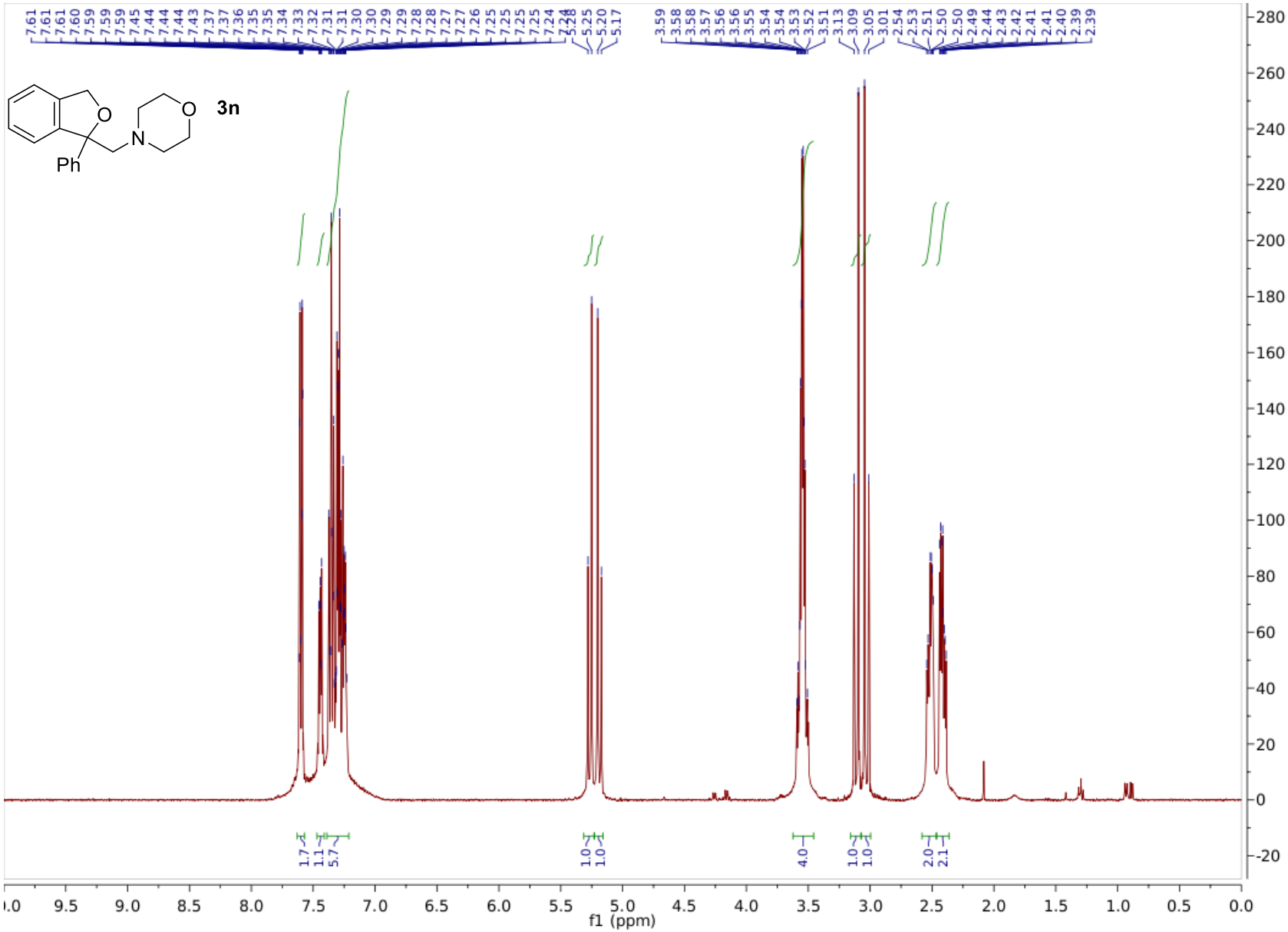

Me
Me
O
N
O
3k
80.2
77.0
76.5
66.8
64.1
54.2
45.6
39.1
26.6
26.4
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

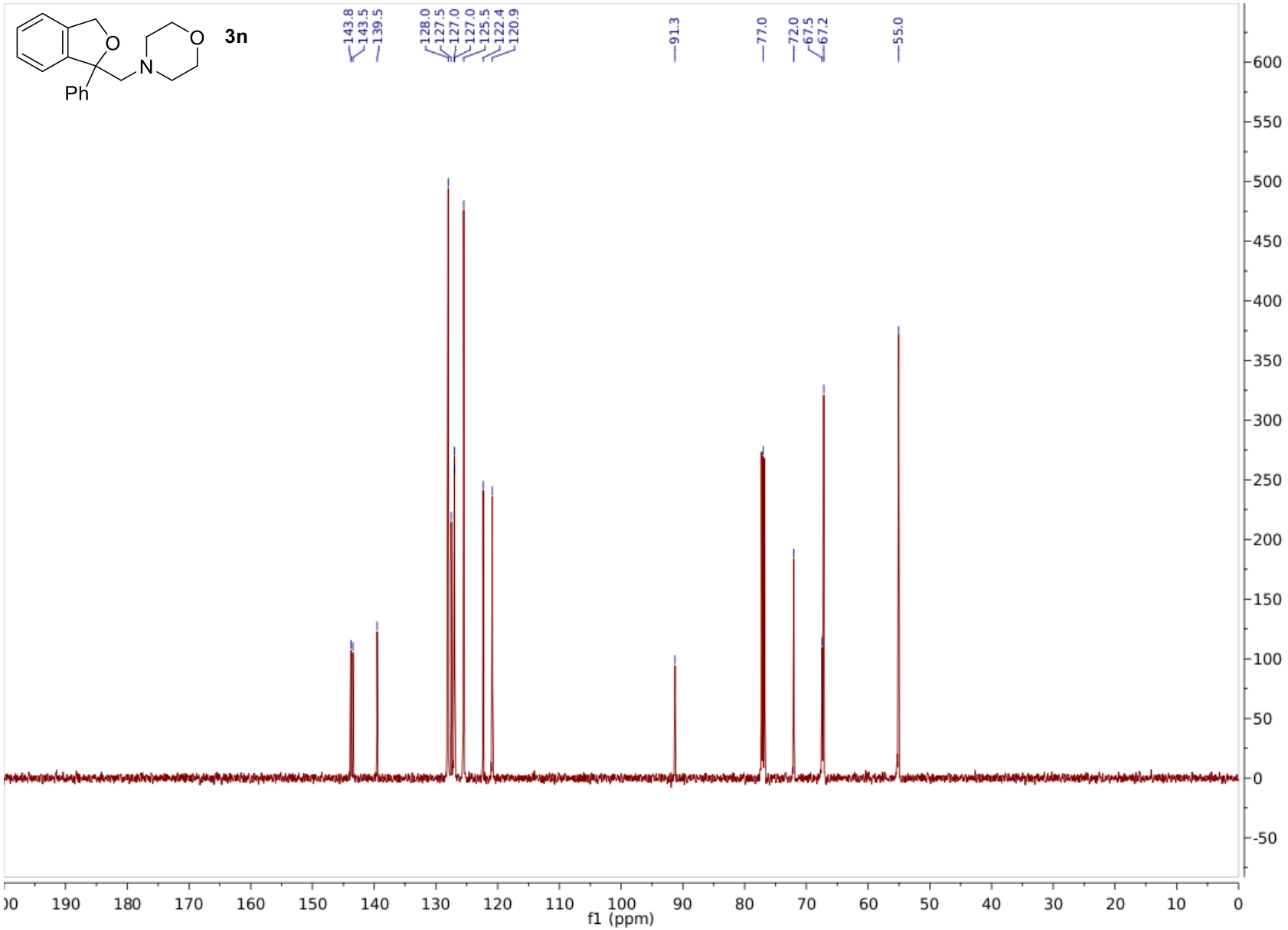

3l
f1 (ppm)

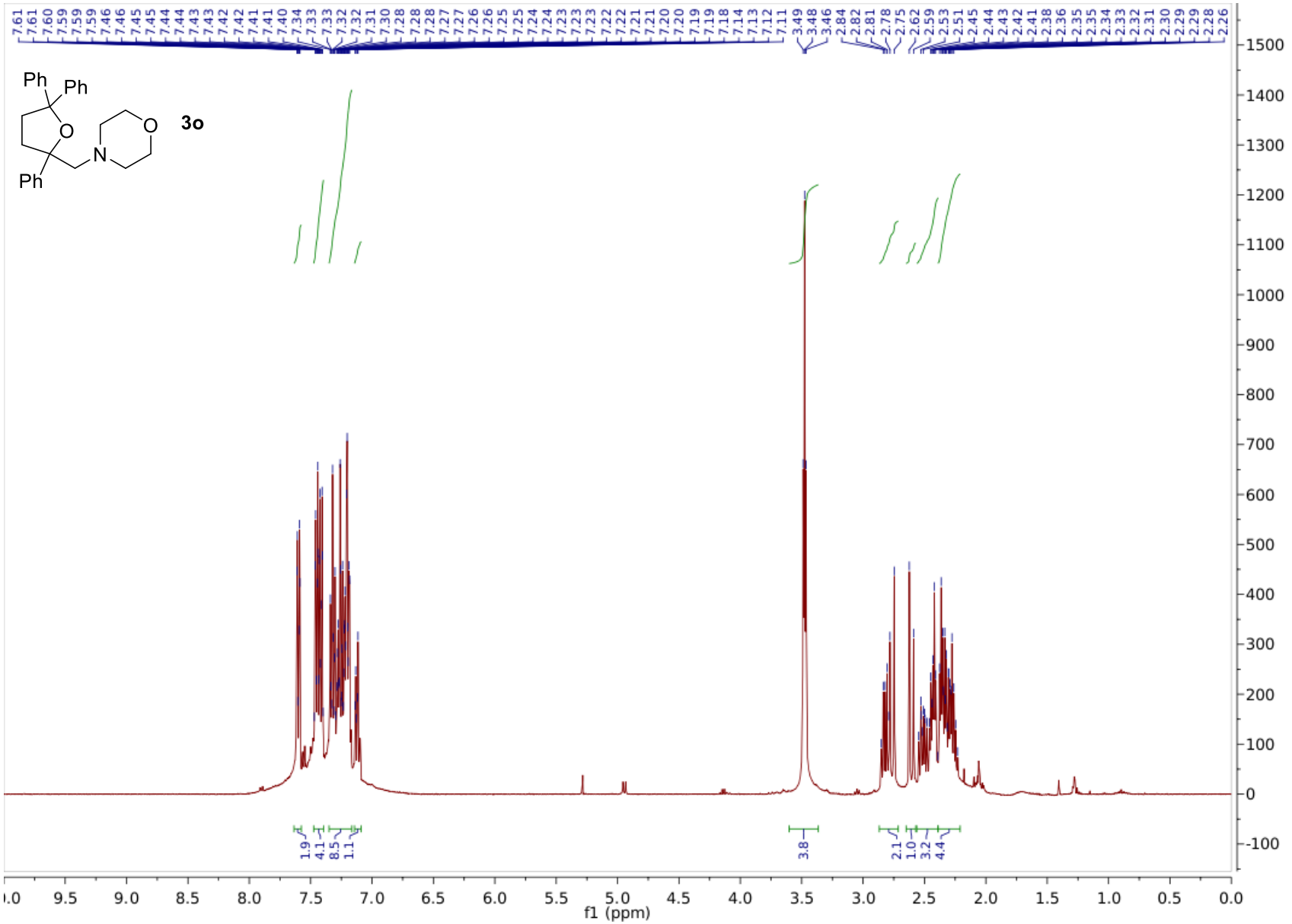

3l
77.0
76.3
68.1
66.8
63.7
54.2
30.3
25.3
f1 (ppm)

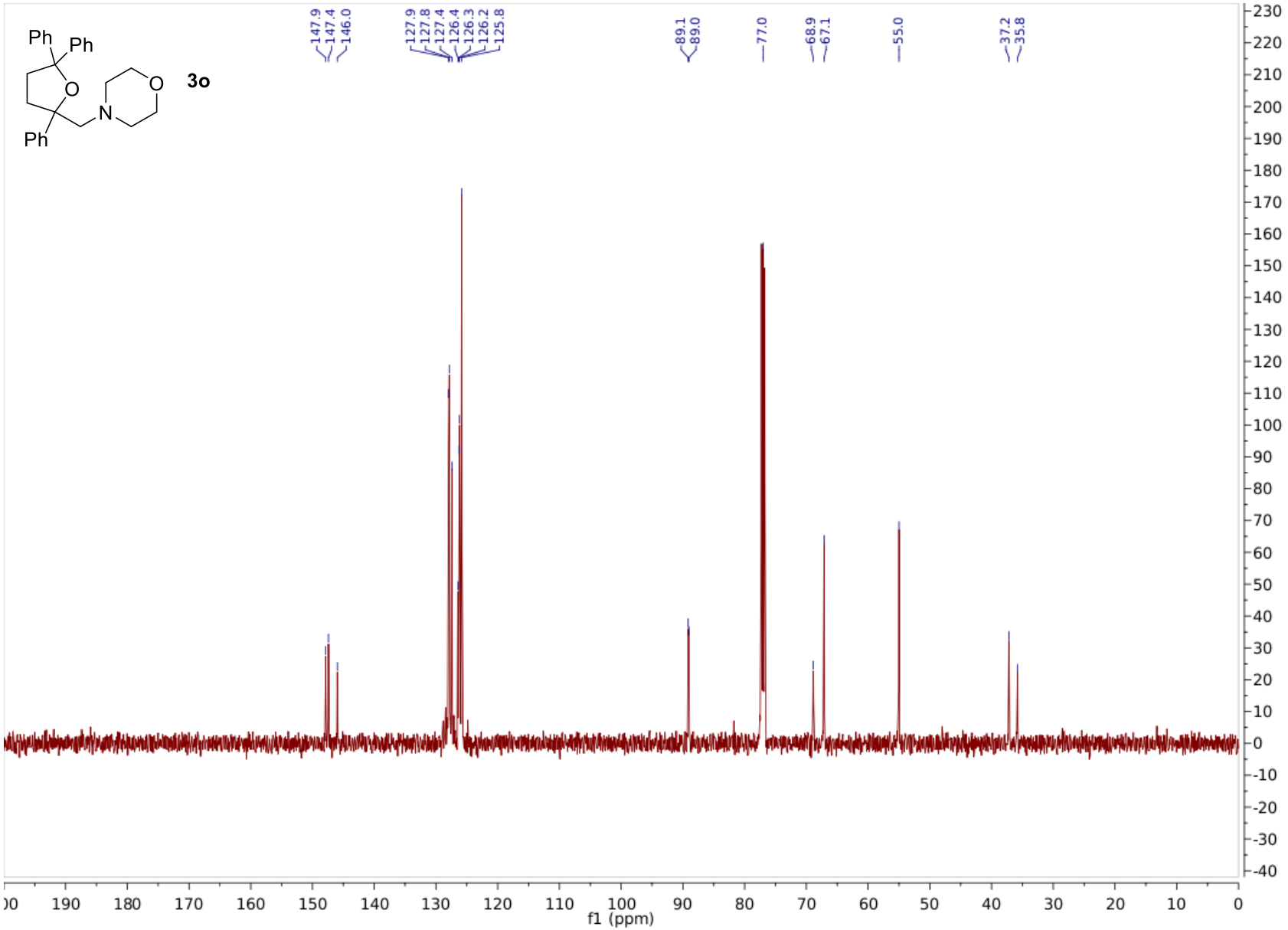

Me
Me
O
O
N
Ph
3m
f1 (ppm)

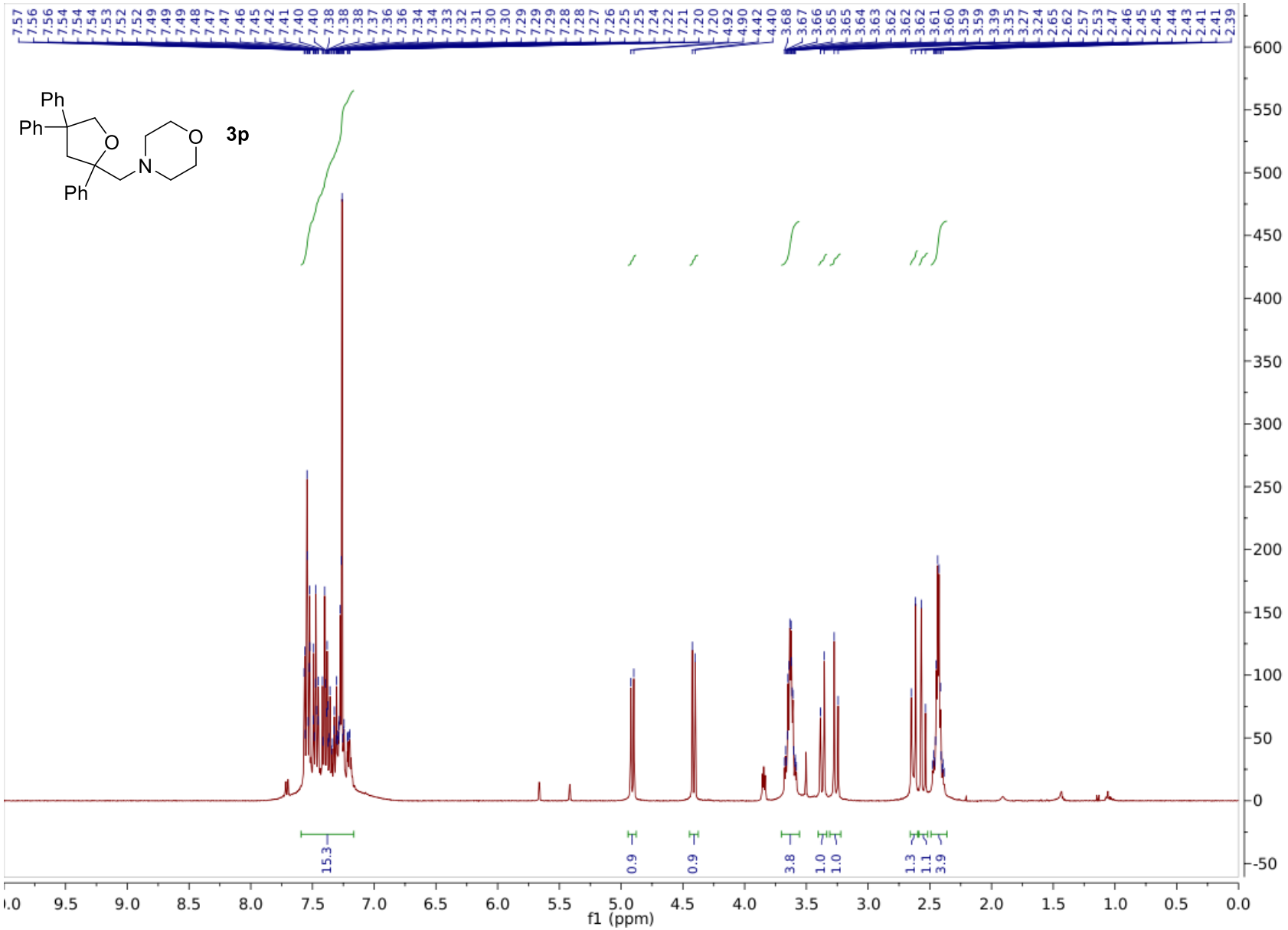

Me
Me
O
O
N
Ph
3m
147.3
145.3
142.3
127.8
127.1
126.7
125.7
122.8
120.6
89.8
85.2
77.0
69.2
67.1
55.3
30.0
30.0
110
100
90
80
70
60
50
40
30
20
10
0
-10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

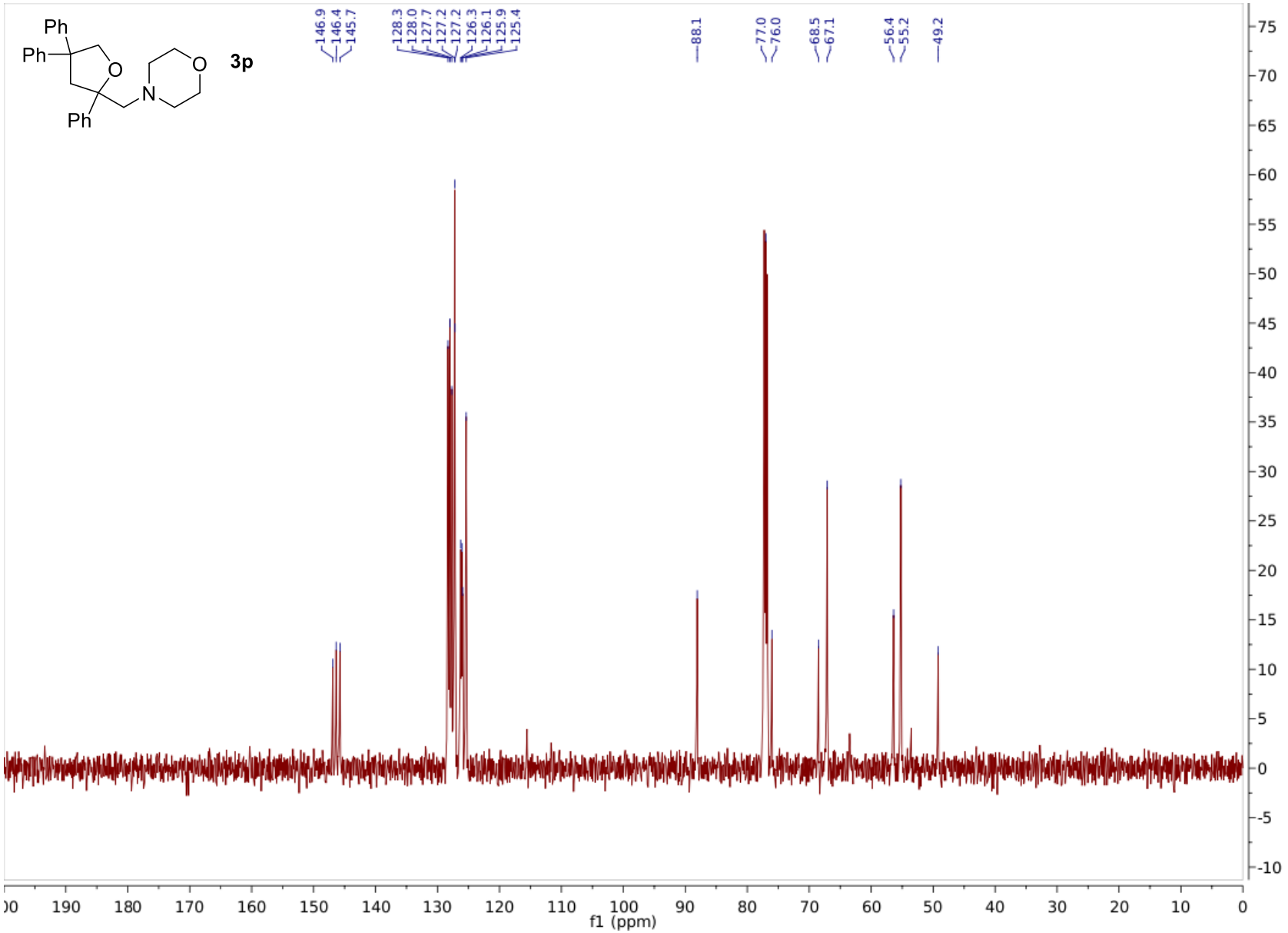

3n
Ph
O
O
N
f1 (ppm)

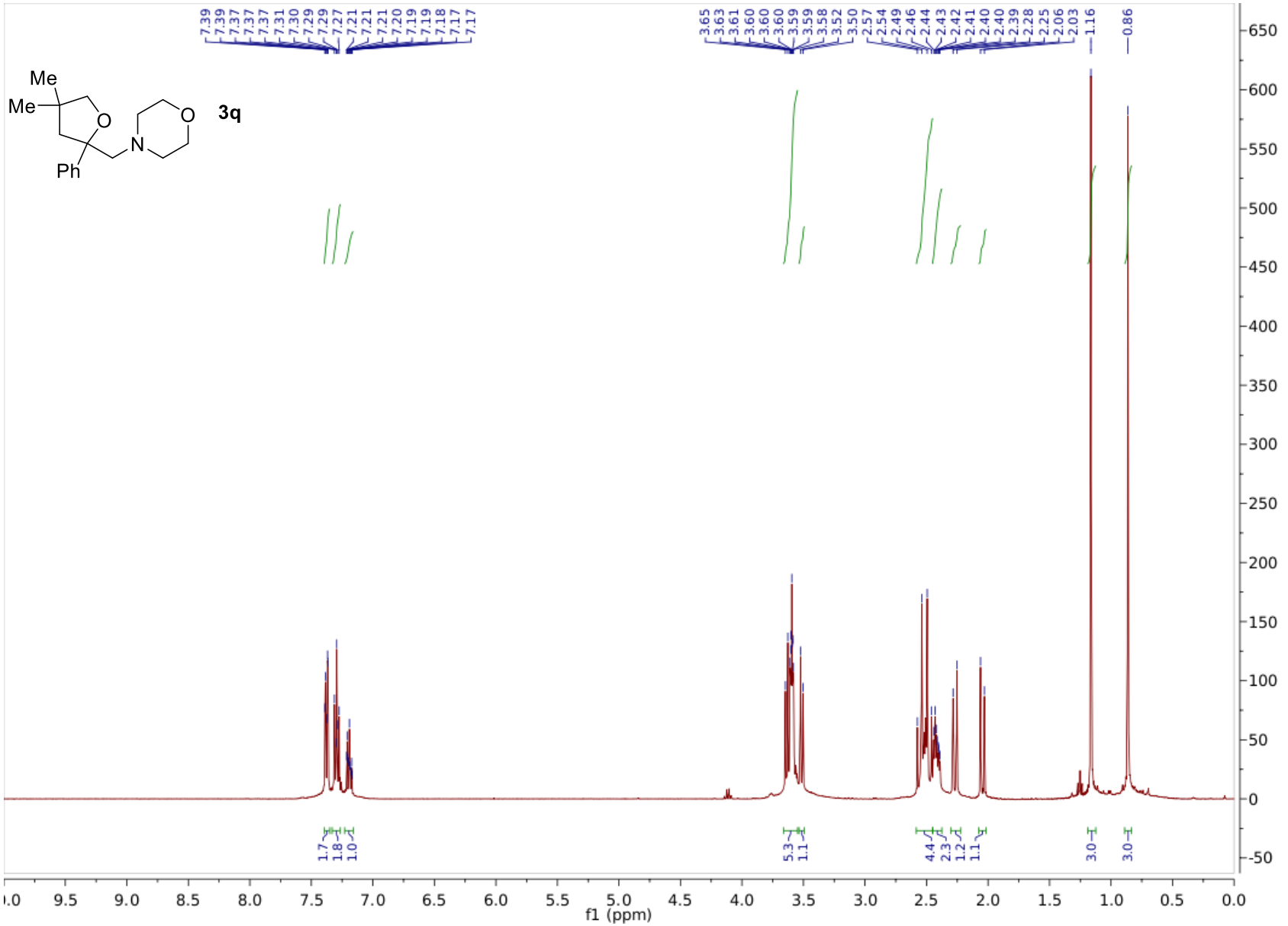

3n
143.8
143.5
139.5
128.0
127.5
127.0
127.0
125.5
122.4
120.9
91.3
77.0
72.0
67.5
67.2
55.0
f1 (ppm)

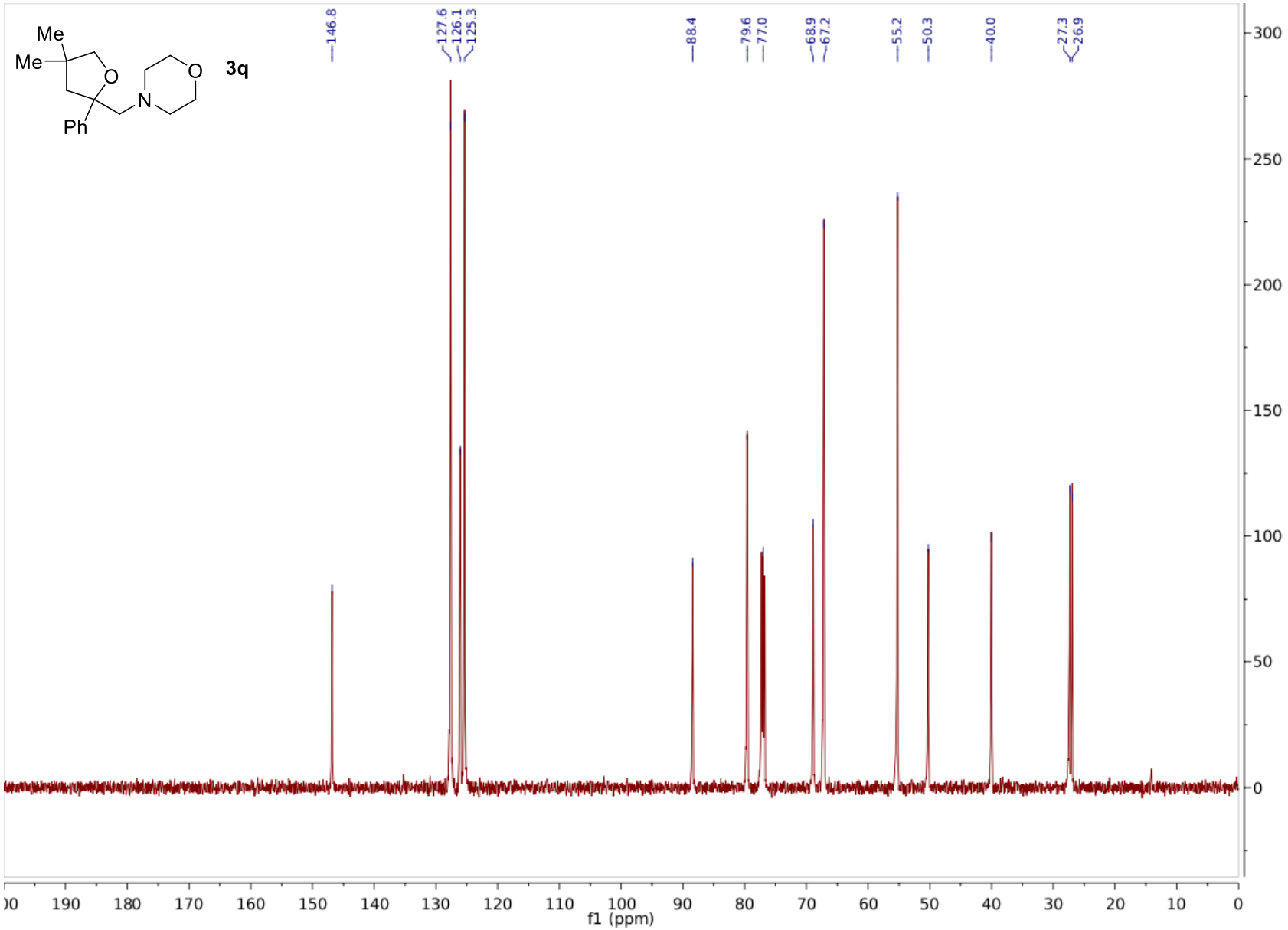

Ph
Ph
O
O
N
Ph
3o
f1 (ppm)

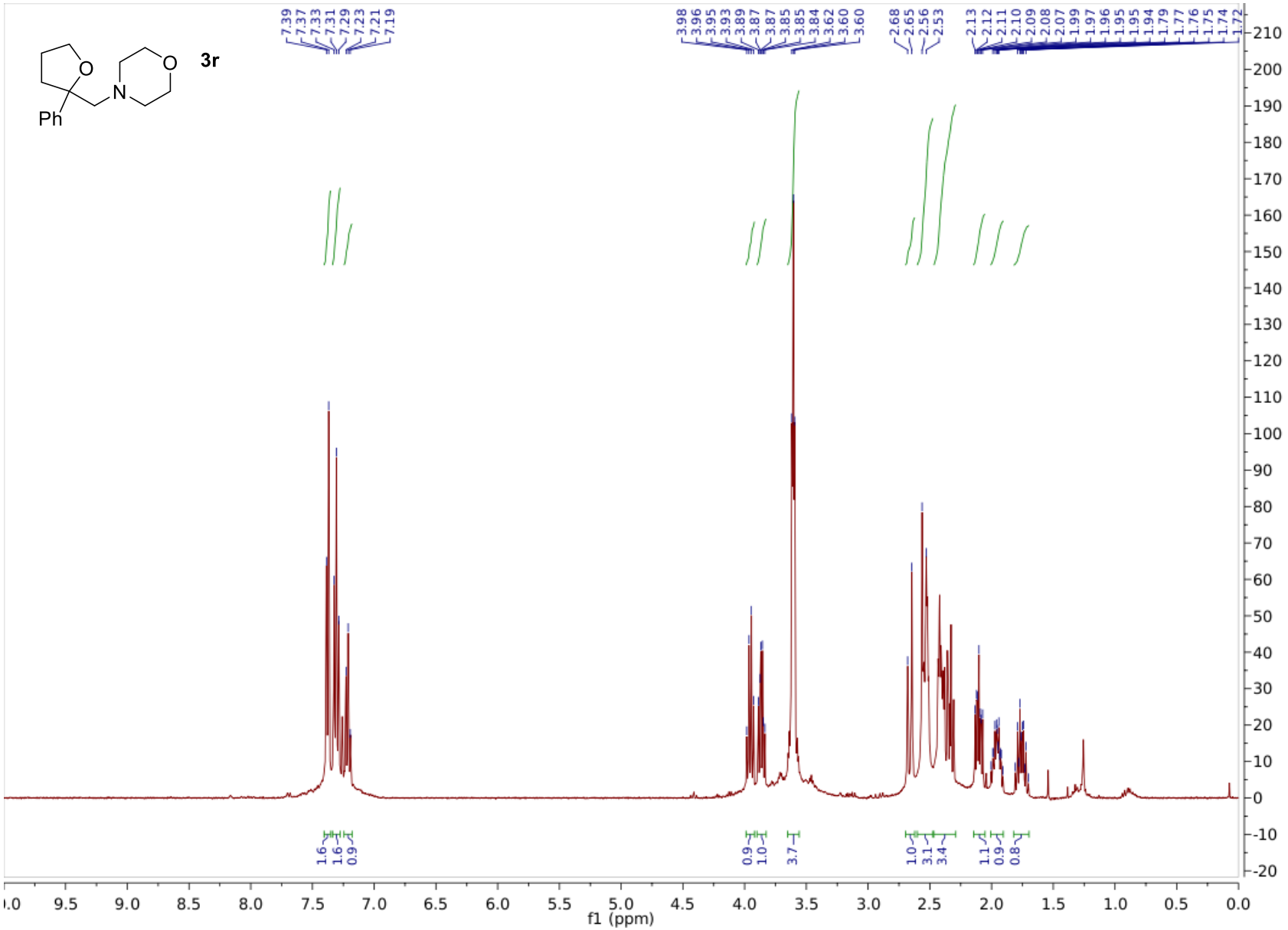

Ph
Ph
O
O
N
Ph
3o
147.9
147.4
146.0
127.9
127.8
127.4
126.4
126.3
126.2
125.8
89.1
89.0
77.0
68.9
67.1
55.0
37.2
35.8
f1 (ppm)

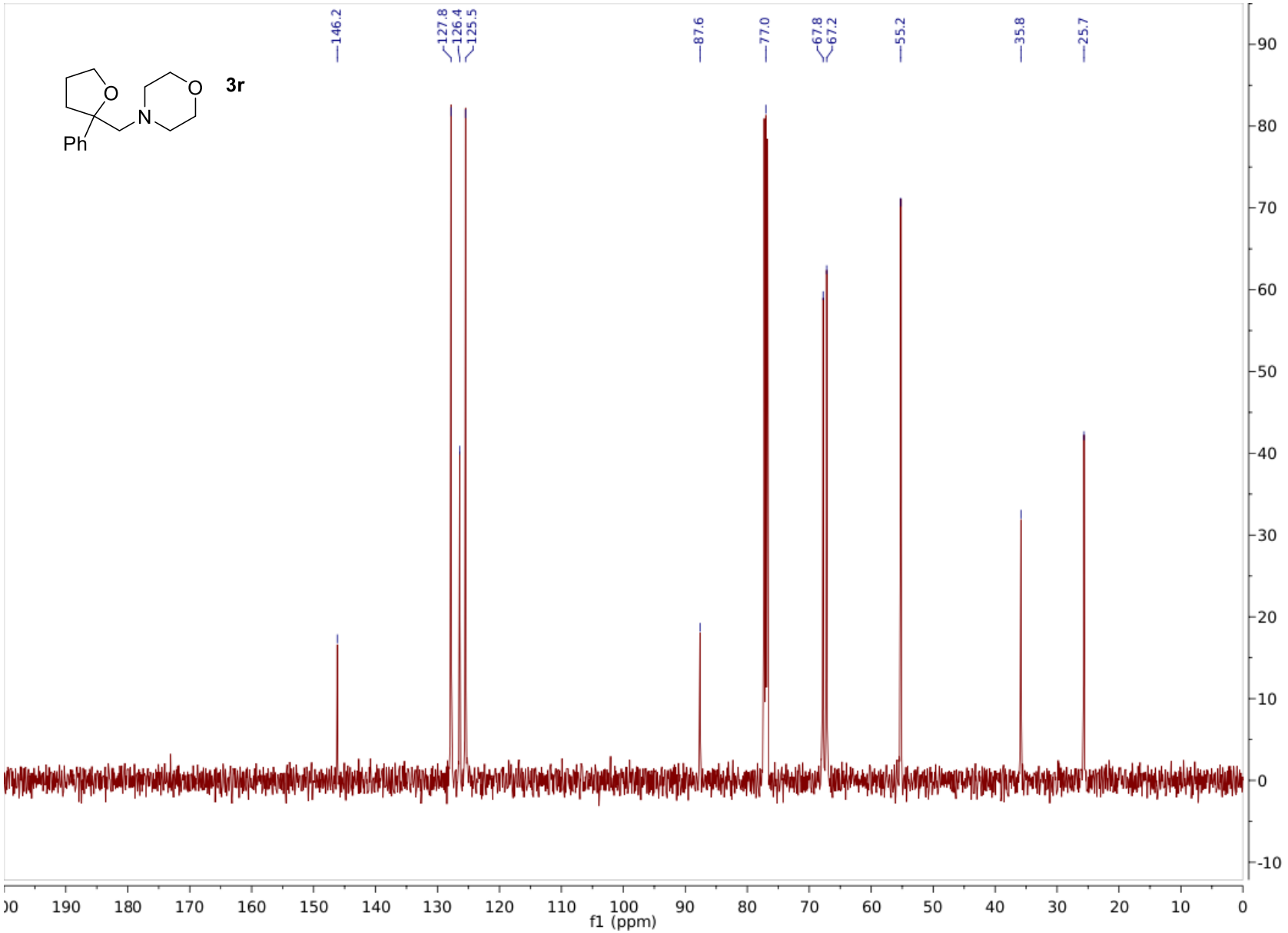

Ph
Ph
O
O
N
Ph
3p
f1 (ppm)

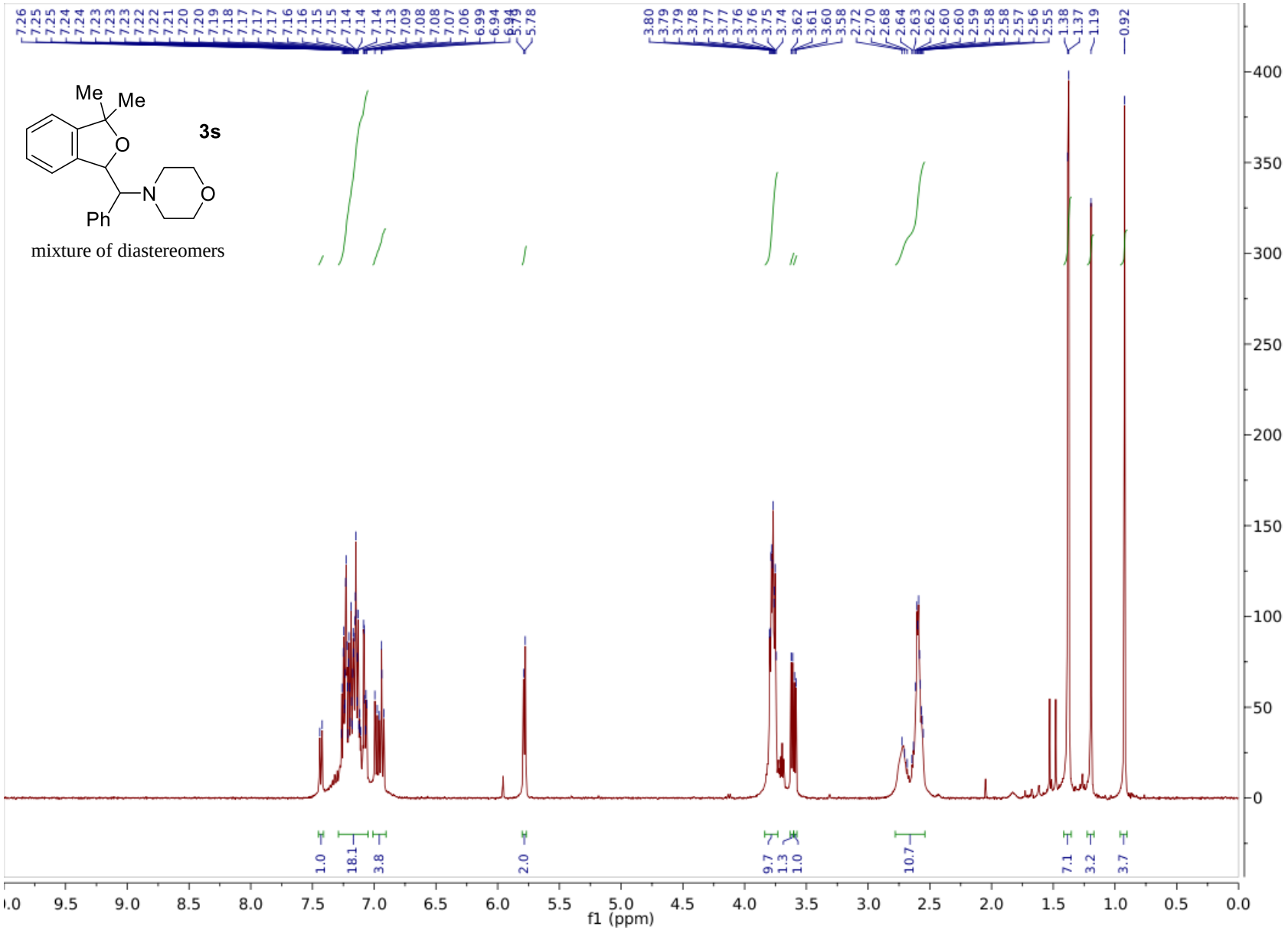

Ph
Ph
O
O
N
Ph
3p
146.9
146.4
145.7
128.3
128.0
127.7
127.2
127.2
126.3
126.1
125.9
125.4
88.1
77.0
76.0
68.5
67.1
56.4
55.2
49.2
f1 (ppm)

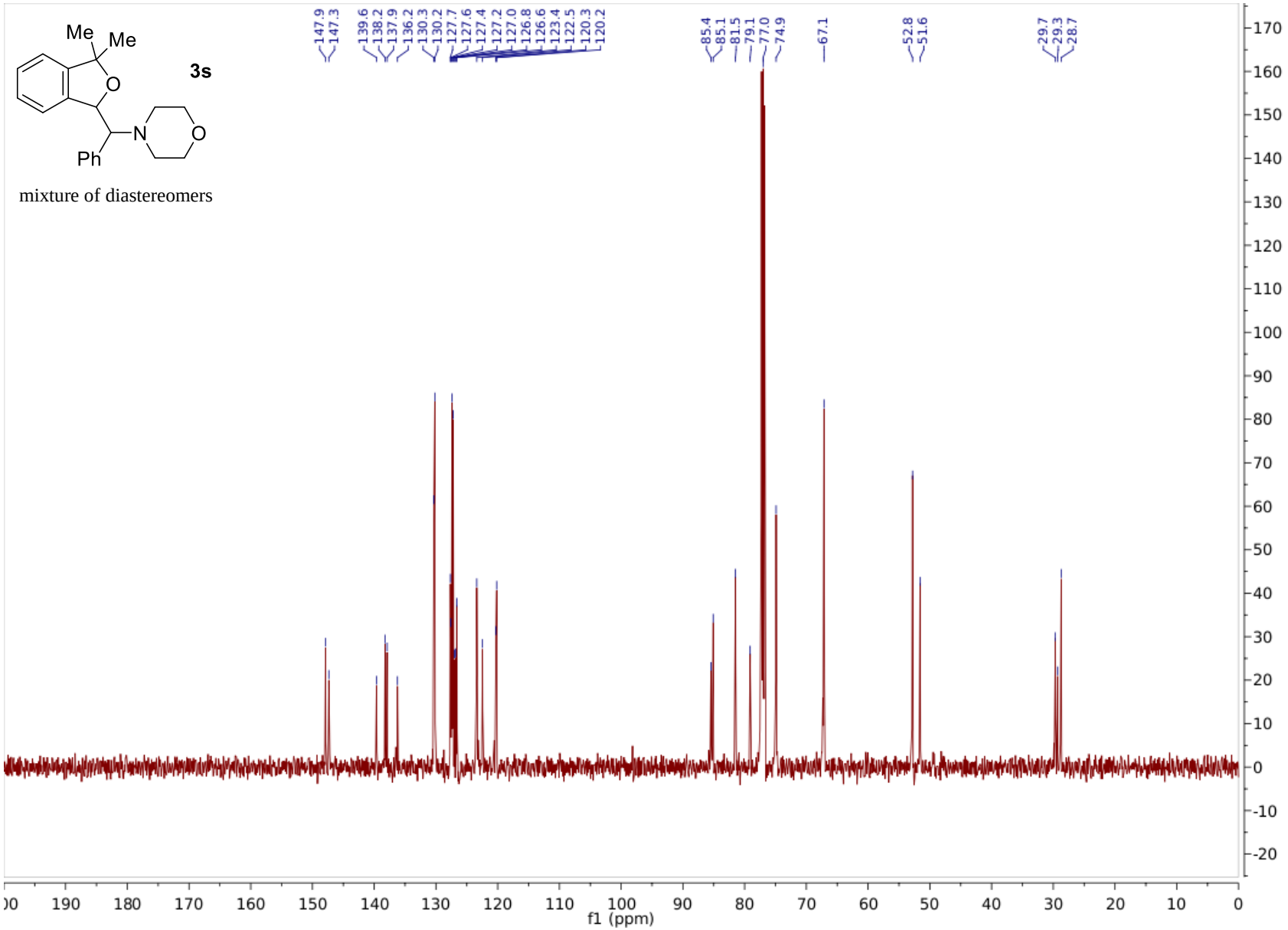

Me
Me
O
O
N
Ph
3q
f1 (ppm)

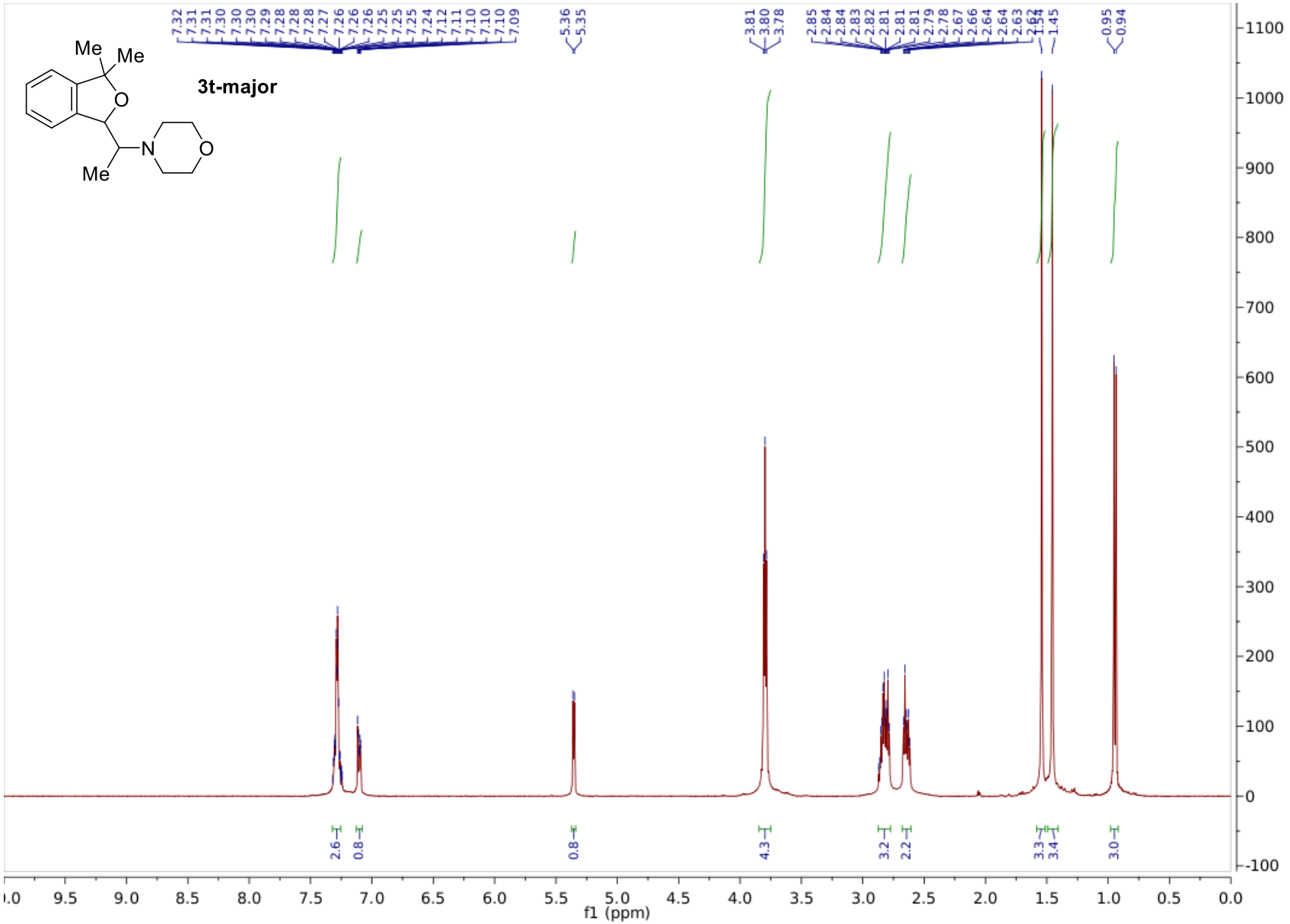

Me
Me
O
O
N
Ph
3q
146.8
127.6
126.1
125.3
88.4
79.6
77.0
68.9
67.2
55.2
50.3
40.0
27.3
26.9
300
250
200
150
100
50
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

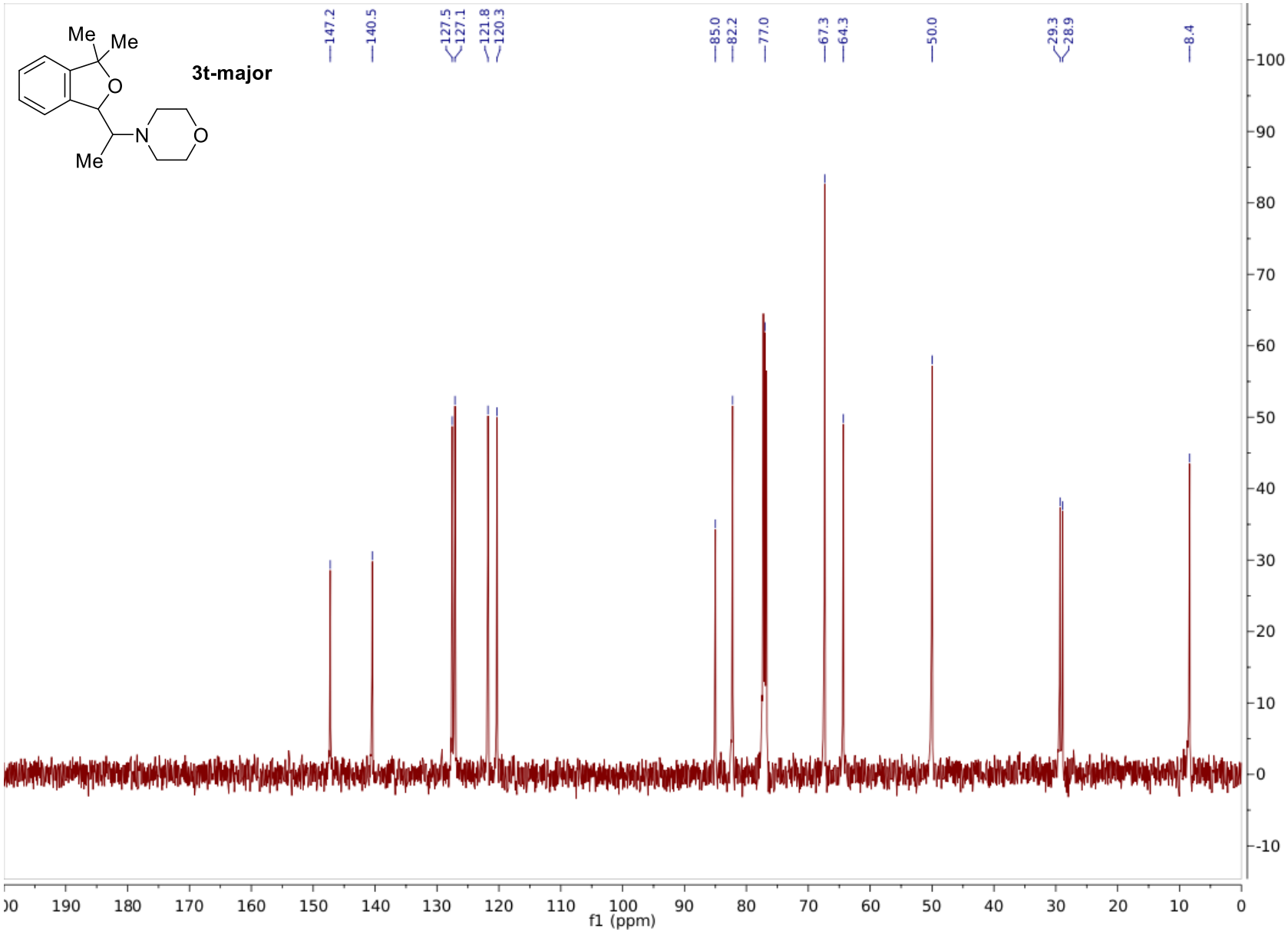

3r
Ph
O
O
N
7.39
7.37
7.33
7.31
7.29
7.23
7.21
7.19
3.98
3.96
3.95
3.93
3.89
3.87
3.87
3.85
3.85
3.84
3.62
3.60
3.60
2.68
2.65
2.56
2.53
2.13
2.12
2.11
2.10
2.09
2.08
2.07
1.99
1.97
1.96
1.95
1.95
1.94
1.79
1.77
1.76
1.75
1.74
1.72
1.6
1.6
0.9
0.9
1.0
3.7
1.0
3.1
3.4
1.1
0.9
0.8
f1 (ppm)

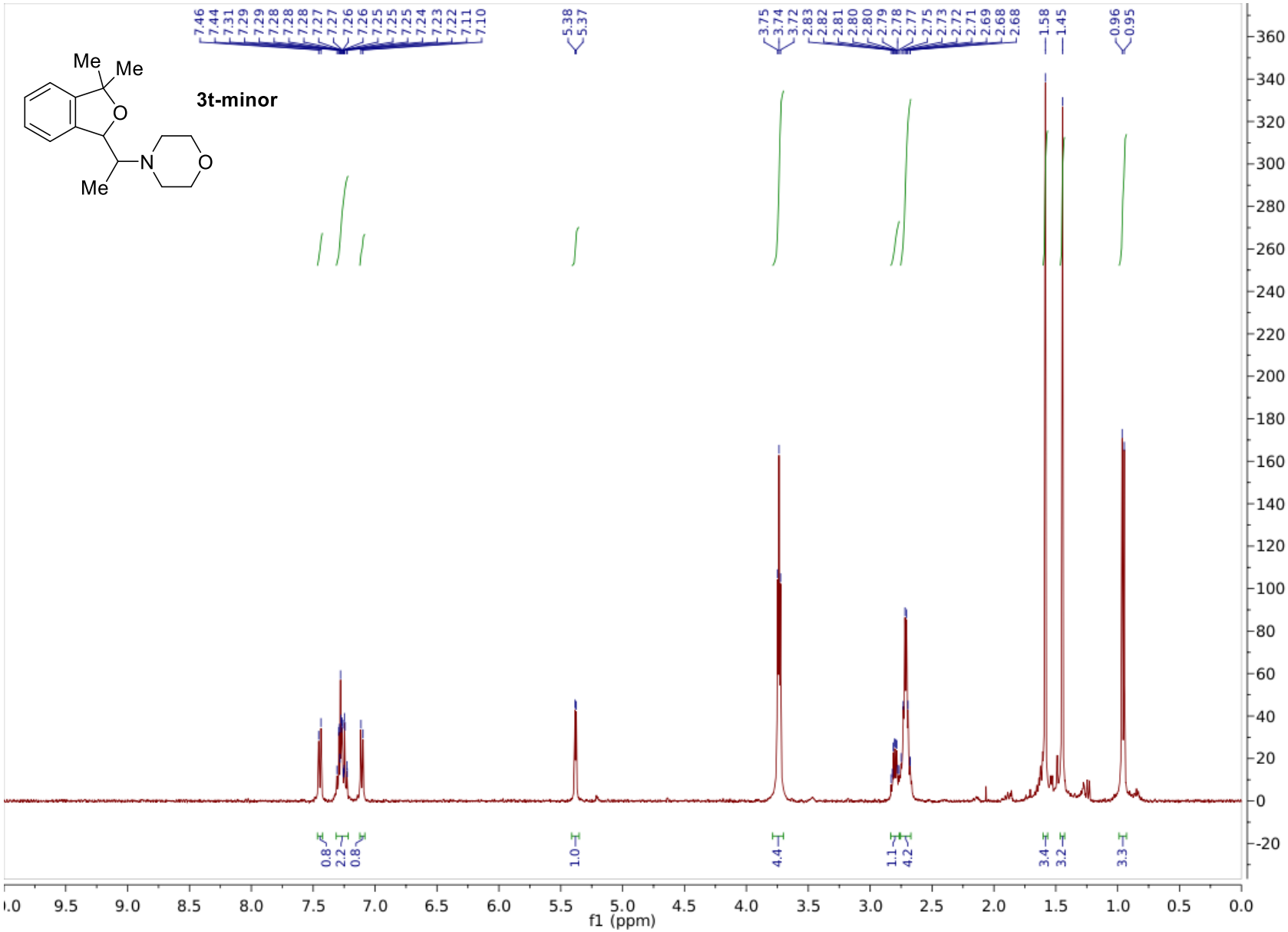

3r
Ph
O
O
N
146.2
127.8
126.4
125.5
87.6
77.0
67.8
67.2
55.2
35.8
25.7
f1 (ppm)

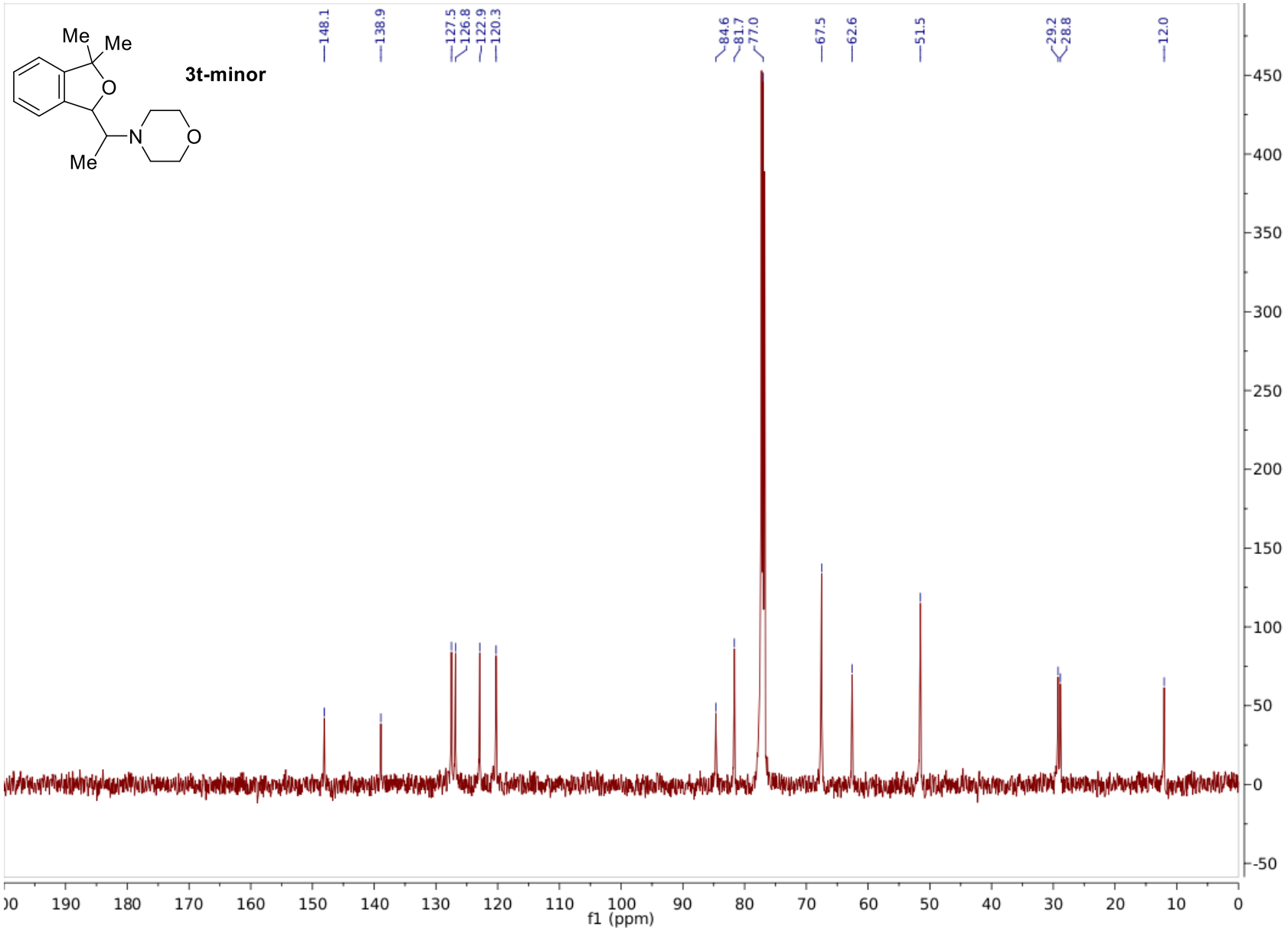

Me
Me
O
N
O
Ph
3s
mixture of diastereomers
f1 (ppm)

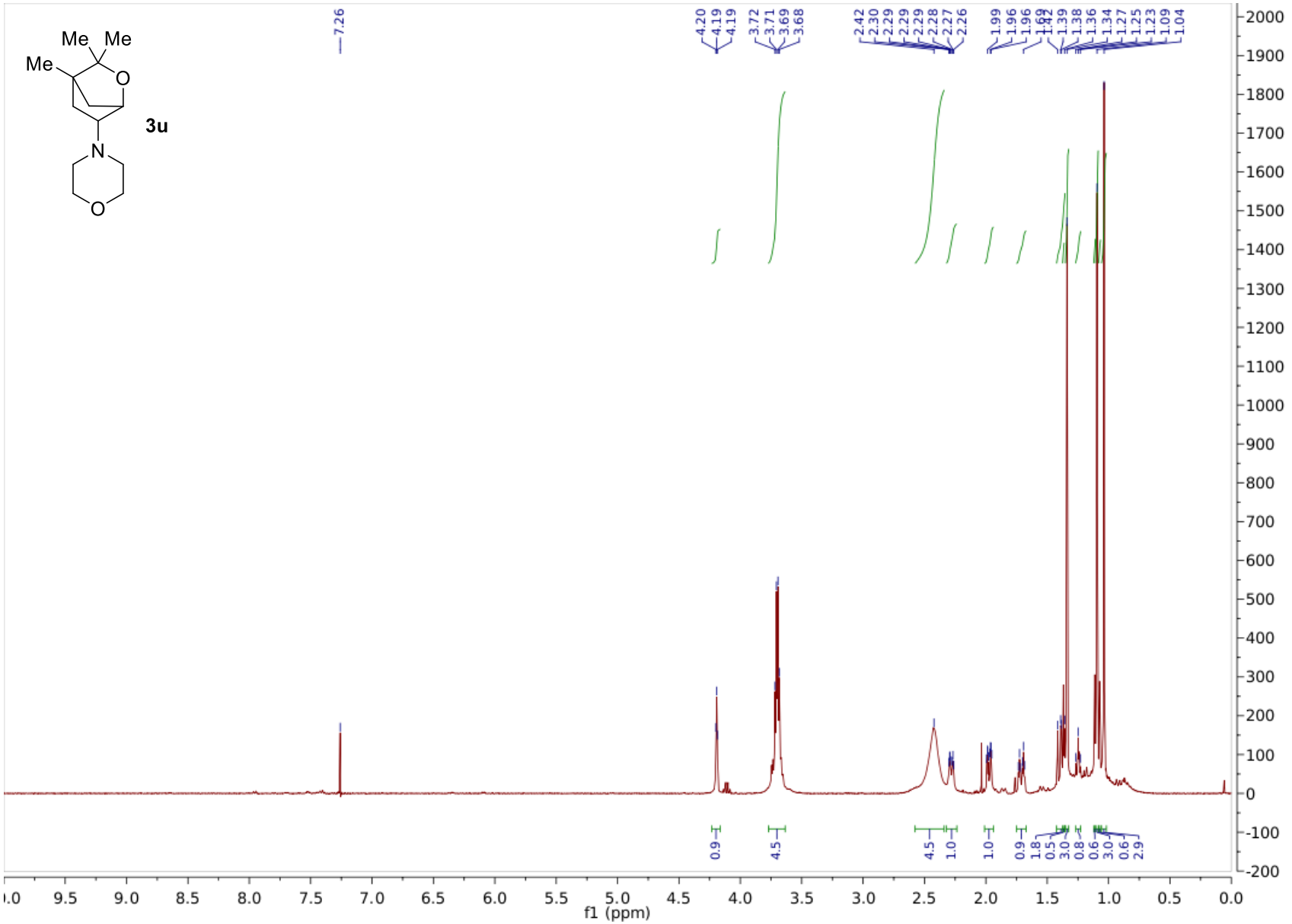

Me
Me
O
N
O
Ph
3s
mixture of diastereomers
147.9
147.3
139.6
138.2
137.9
136.2
130.3
130.2
127.7
127.6
127.4
127.2
127.0
126.8
126.6
123.4
122.5
120.3
120.2
85.4
85.1
81.5
79.1
77.0
74.9
67.1
52.8
51.6
29.7
29.3
28.7
f1 (ppm)

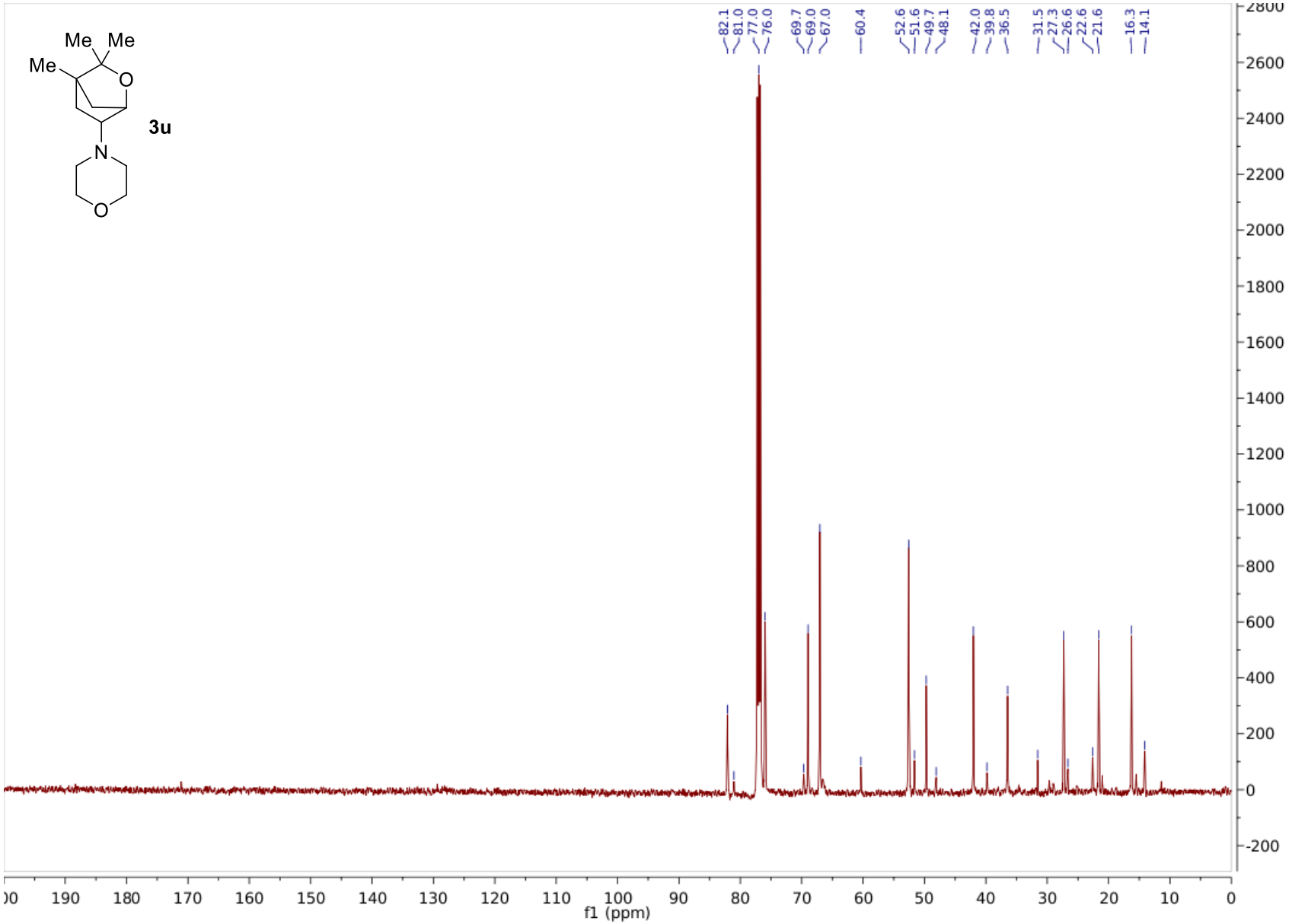

3t-major
Me
Me
O
N
O
Me
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.28
7.28
7.28
7.27
7.26
7.26
7.26
7.25
7.25
7.25
7.24
7.12
7.11
7.10
7.10
7.10
7.09
5.36
5.35
3.81
3.80
3.78
2.85
2.84
2.84
2.83
2.82
2.81
2.81
2.81
2.79
2.78
2.67
2.66
2.64
2.64
2.63
1.54
1.45
0.95
0.94
2.6
0.8
0.8
4.3
3.2
2.2
3.3
3.4
3.0
f1 (ppm)

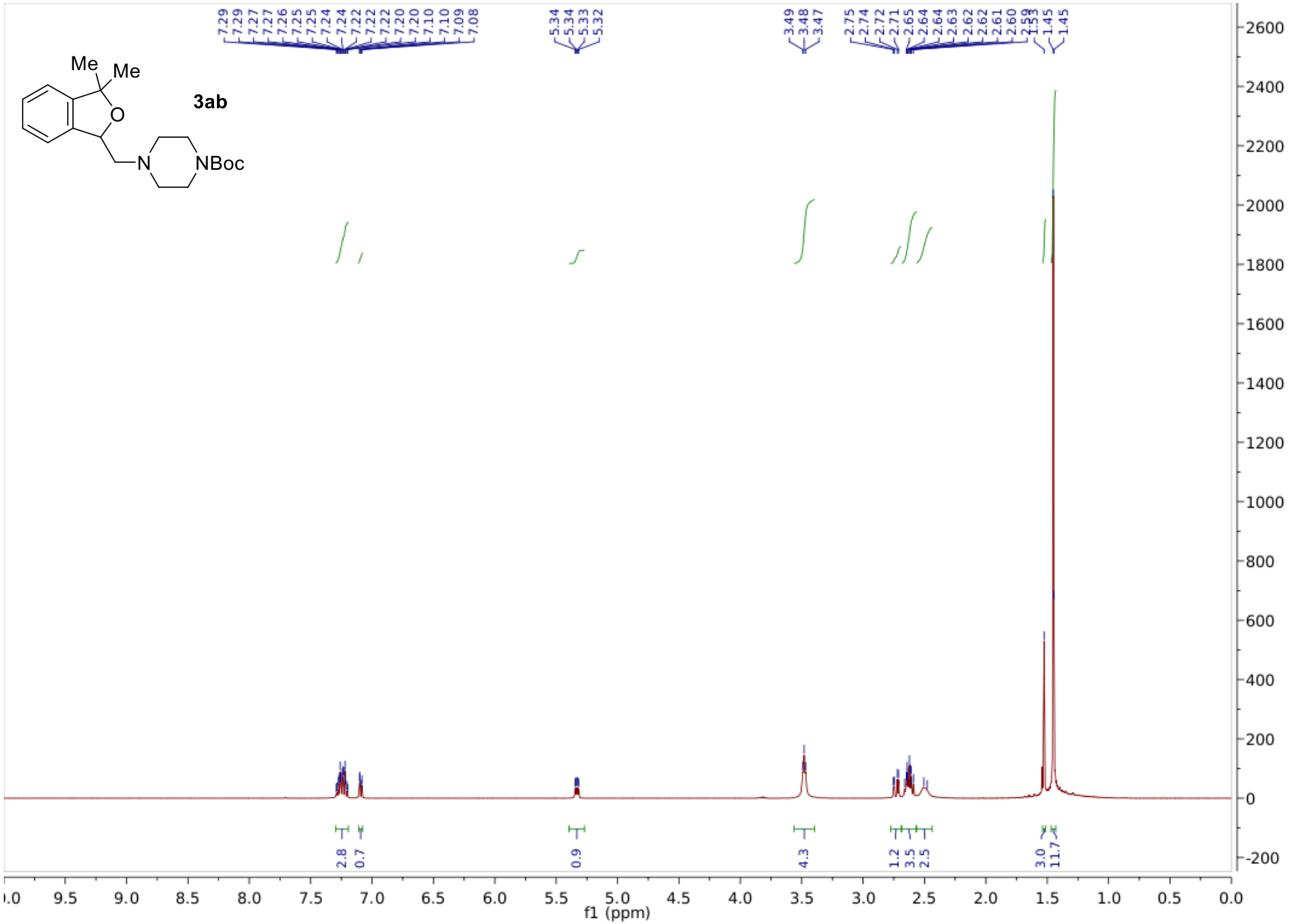

Me
Me
O
N
O
Me
3t-major
147.2
140.5
127.5
127.1
121.8
120.3
85.0
82.2
77.0
67.3
64.3
50.0
29.3
28.9
8.4
100
90
80
70
60
50
40
30
20
10
0
-10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

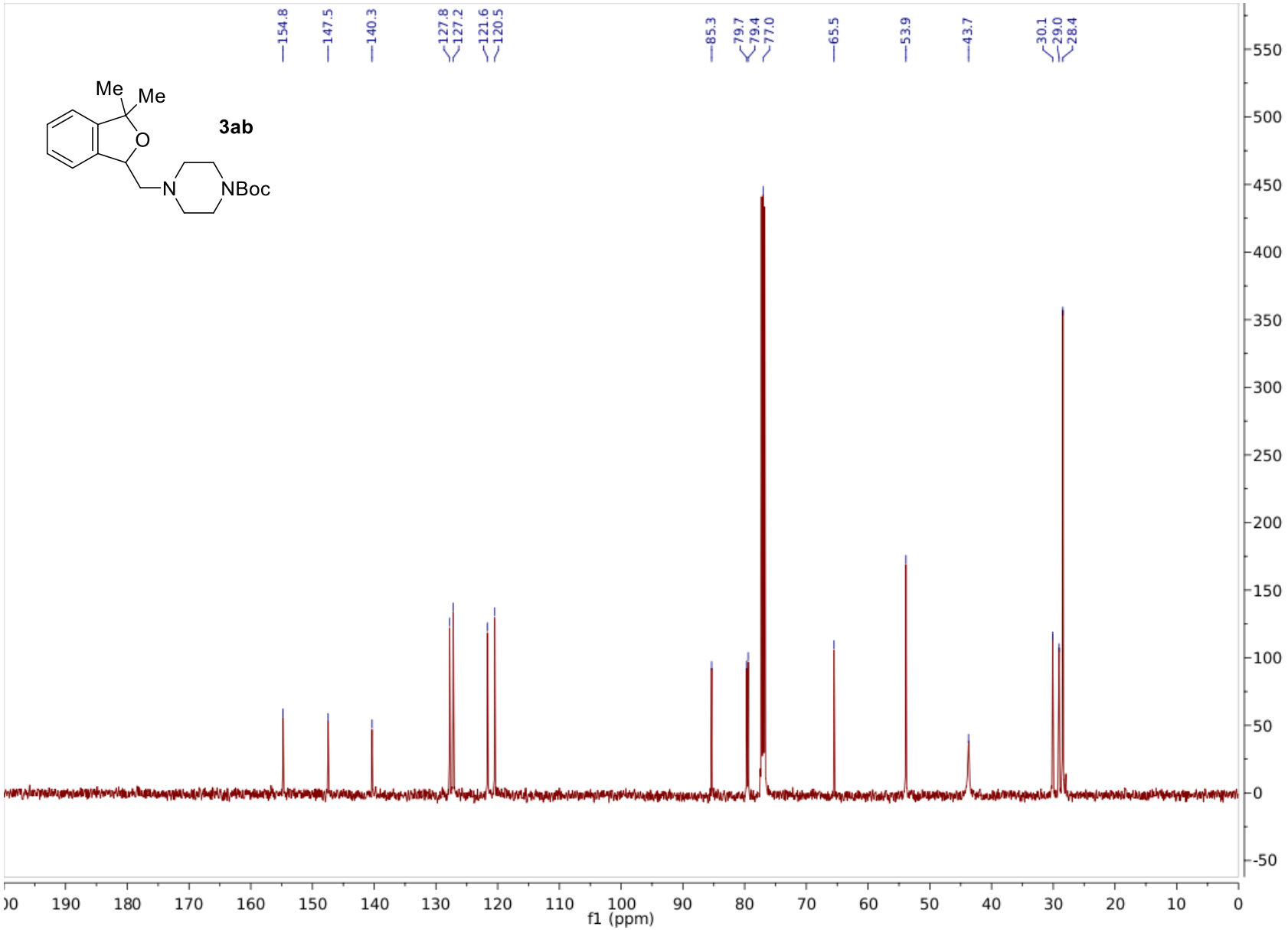

Me
Me
O
N
O
Me
3t-minor
7.46
7.44
7.31
7.29
7.29
7.28
7.28
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.25
7.24
7.23
7.22
7.11
7.10
5.38
5.37
3.75
3.74
3.72
2.83
2.82
2.81
2.80
2.80
2.79
2.78
2.77
2.75
2.73
2.72
2.71
2.69
2.68
2.68
1.58
1.45
0.96
0.95
0.8
2.2
0.8
1.0
4.4
1.1
4.2
3.4
3.2
3.3
f1 (ppm)

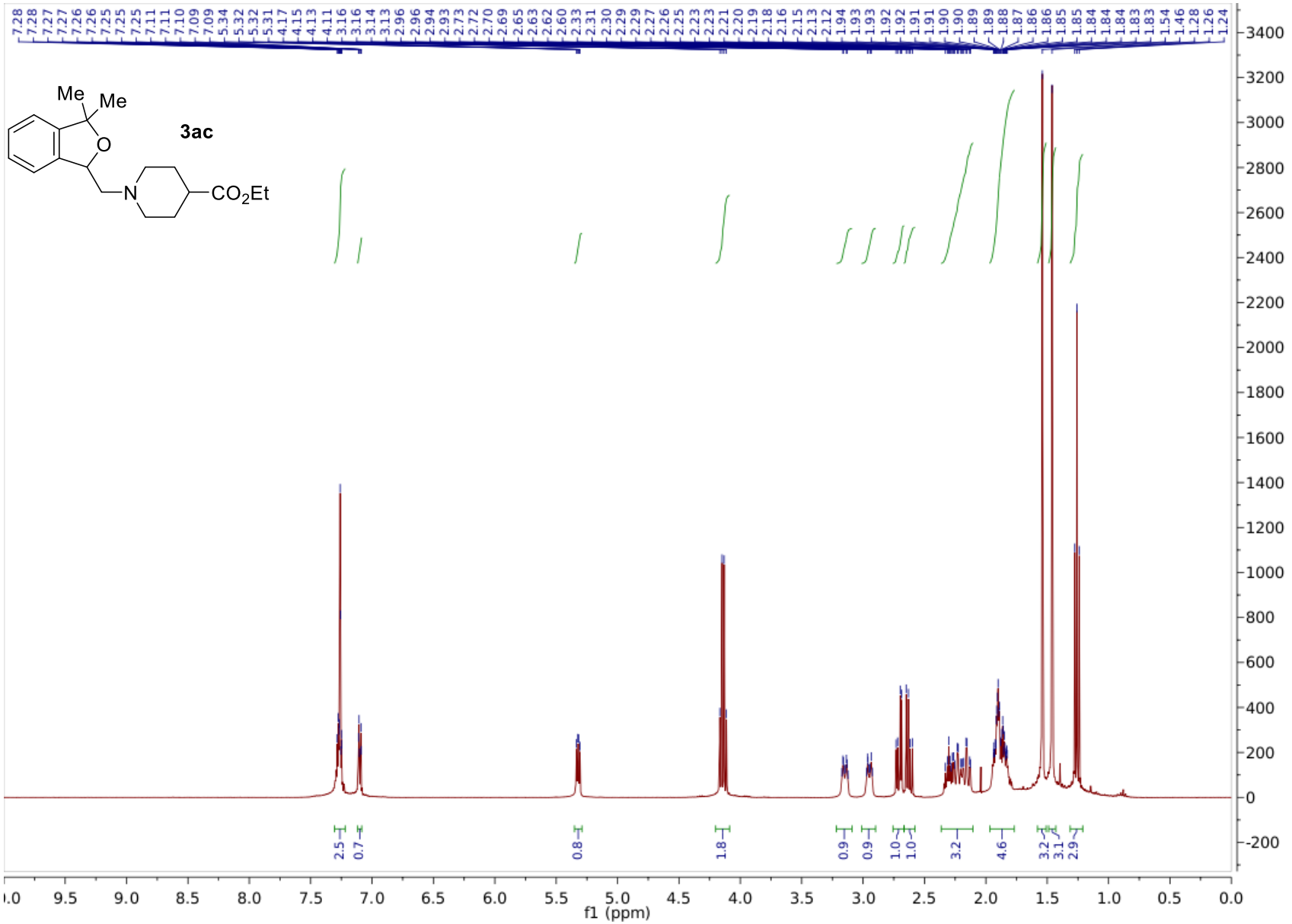

Me
Me
O
N
O
Me
3t-minor
148.1
138.9
127.5
126.8
122.9
120.3
84.6
81.7
77.0
67.5
62.6
51.5
29.2
28.8
12.0
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

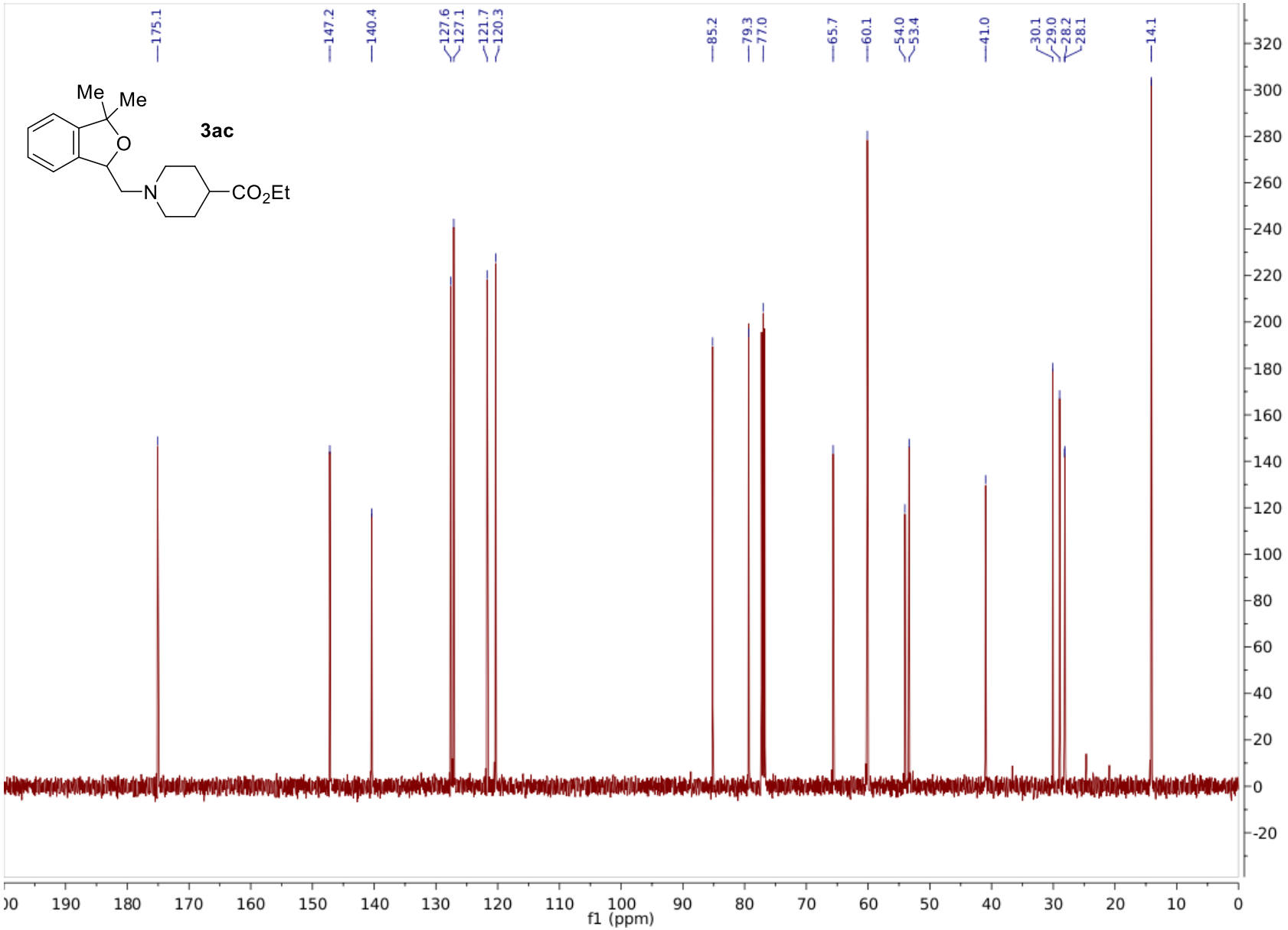

Me
Me
Me
O
N
O
3u
7.26
4.20
4.19
4.19
3.72
3.71
3.69
3.68
2.42
2.30
2.29
2.29
2.29
2.28
2.27
2.26
1.99
1.96
1.96
1.69
1.42
1.39
1.38
1.36
1.34
1.27
1.25
1.23
1.09
1.04
0.9
4.5
4.5
1.0
1.0
0.9
1.8
0.5
3.0
0.8
0.6
3.0
0.6
2.9
f1 (ppm)

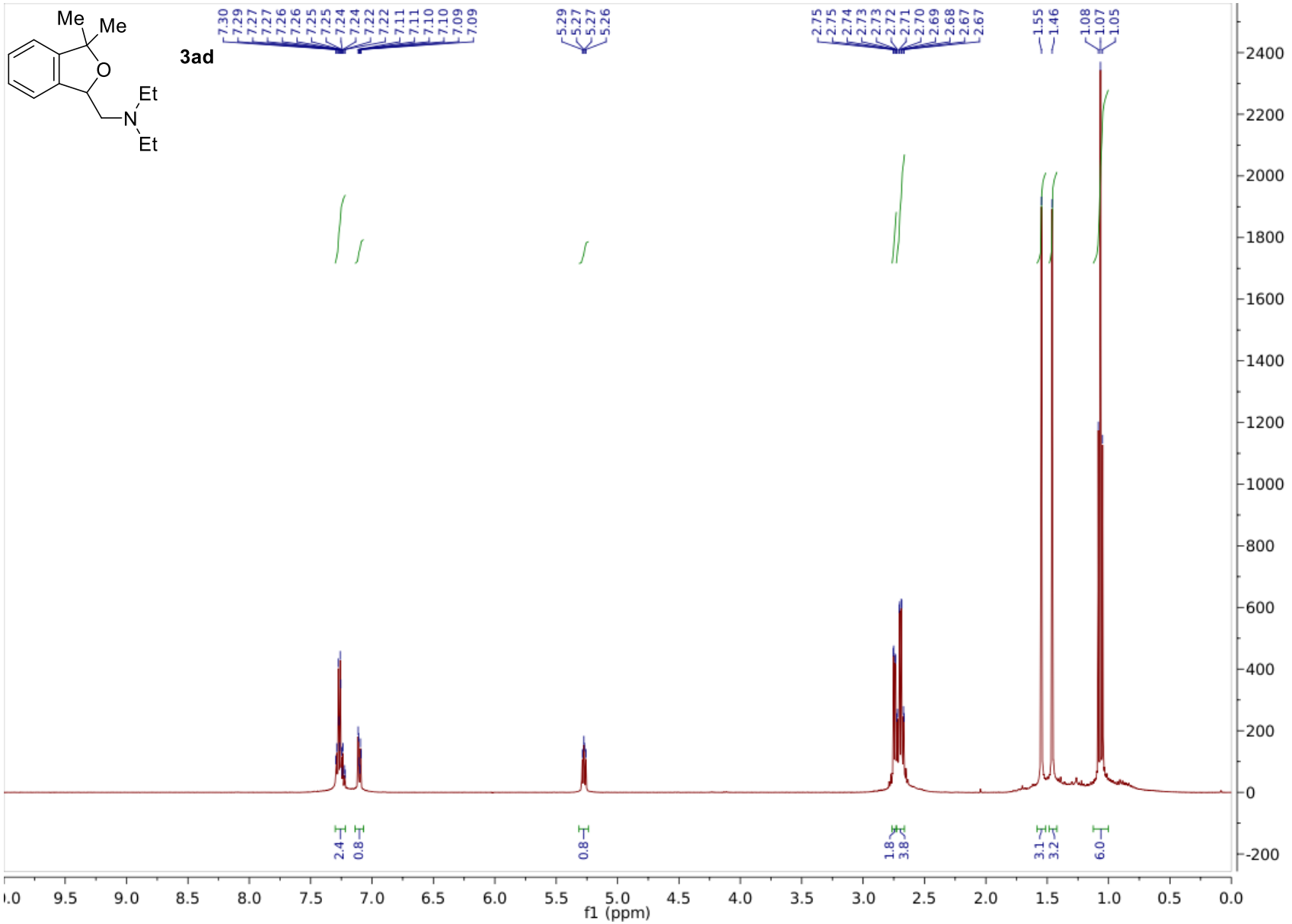

Me
Me
Me
O
N
O
3u
82.1
81.0
77.0
76.0
69.7
69.0
67.0
60.4
52.6
51.6
49.7
48.1
42.0
39.8
36.5
31.5
27.3
26.6
22.6
21.6
16.3
14.1
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

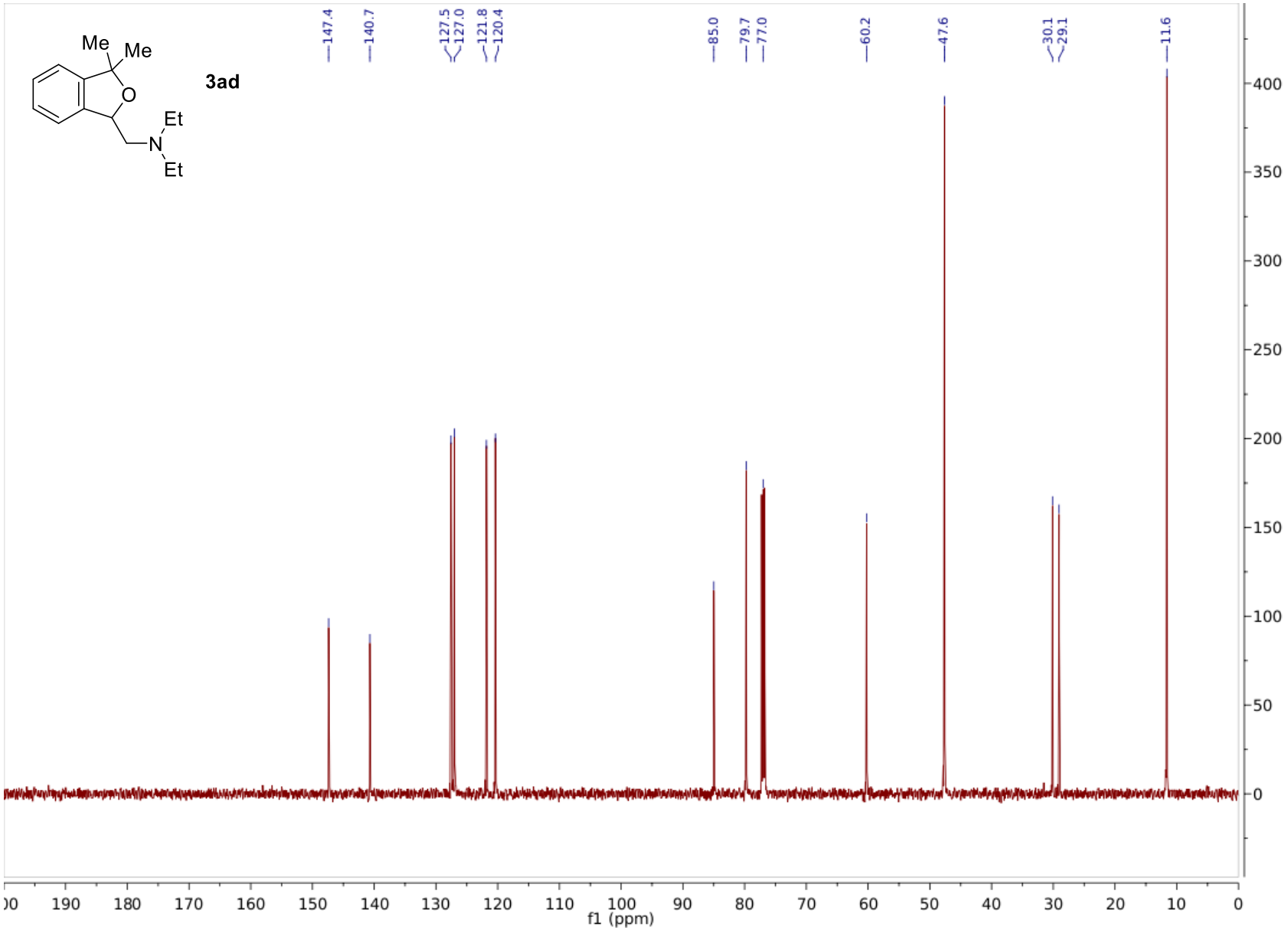

Me
Me
O
N
NBoc
3ab
7.29
7.29
7.27
7.27
7.26
7.25
7.25
7.24
7.24
7.22
7.22
7.22
7.20
7.20
7.10
7.10
7.09
7.08
5.34
5.34
5.33
5.32
3.49
3.48
3.47
2.75
2.74
2.72
2.71
2.65
2.64
2.64
2.63
2.62
2.62
2.61
2.60
2.59
1.53
1.45
1.45
2.8
0.7
0.9
4.3
1.2
3.5
2.5
3.0
11.7
f1 (ppm)

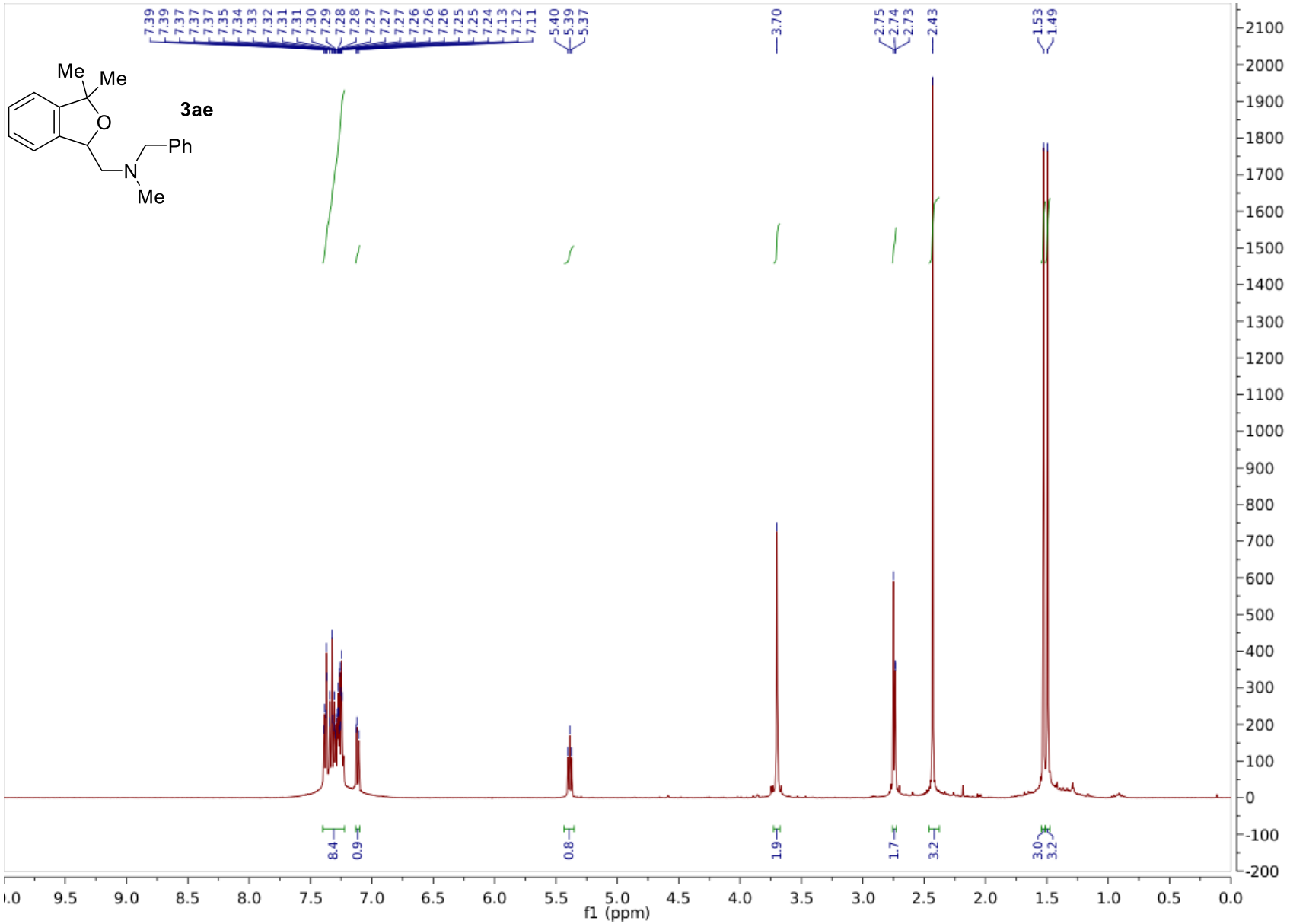

154.8
147.5
140.3
127.8
127.2
121.6
120.5
85.3
79.7
79.4
77.0
65.5
53.9
43.7
30.1
29.0
28.4
Me
Me
O
N
NBoc
3ab
f1 (ppm)

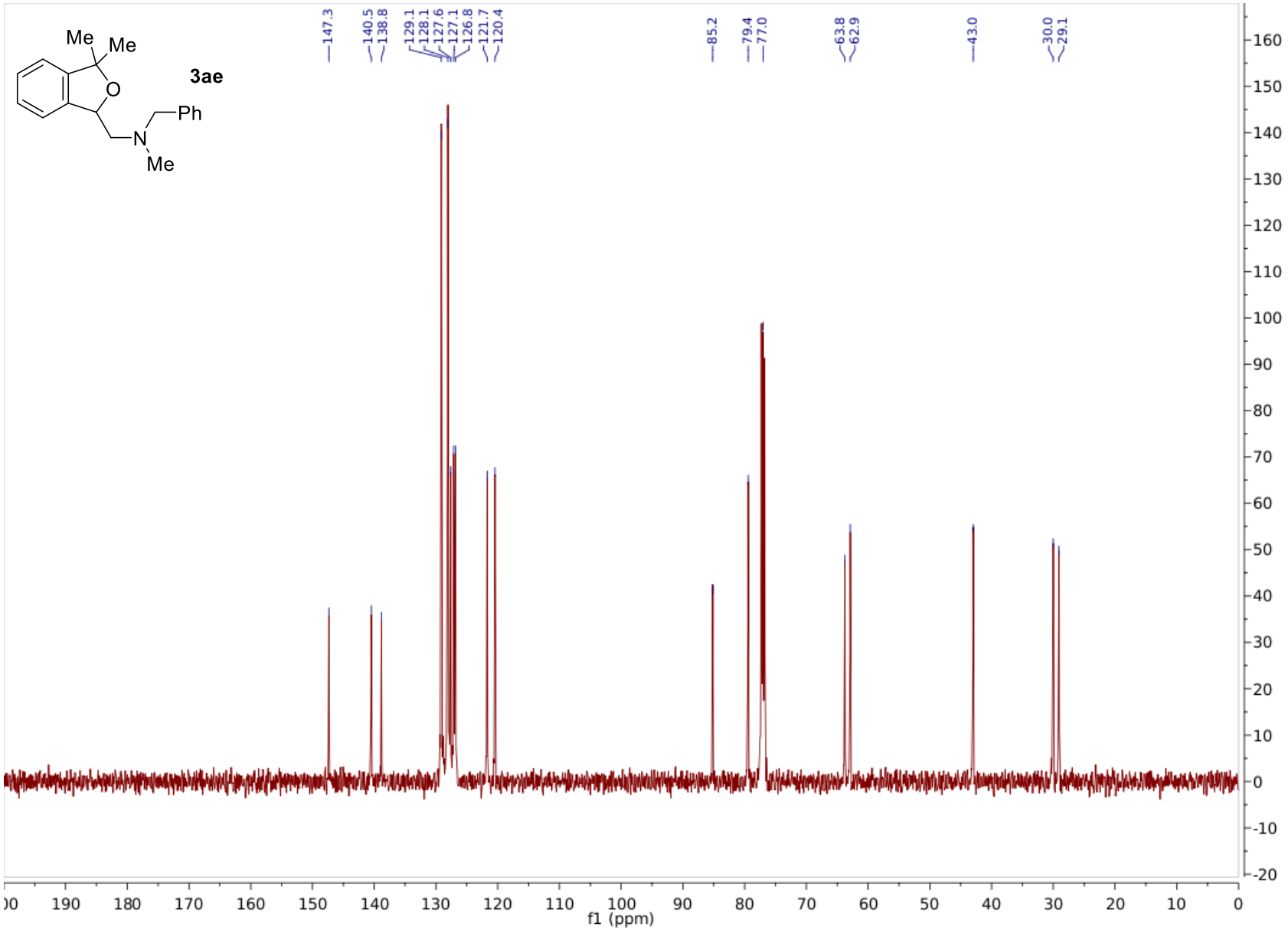

Me
Me
O
N
CO2Et
3ac
f1 (ppm)

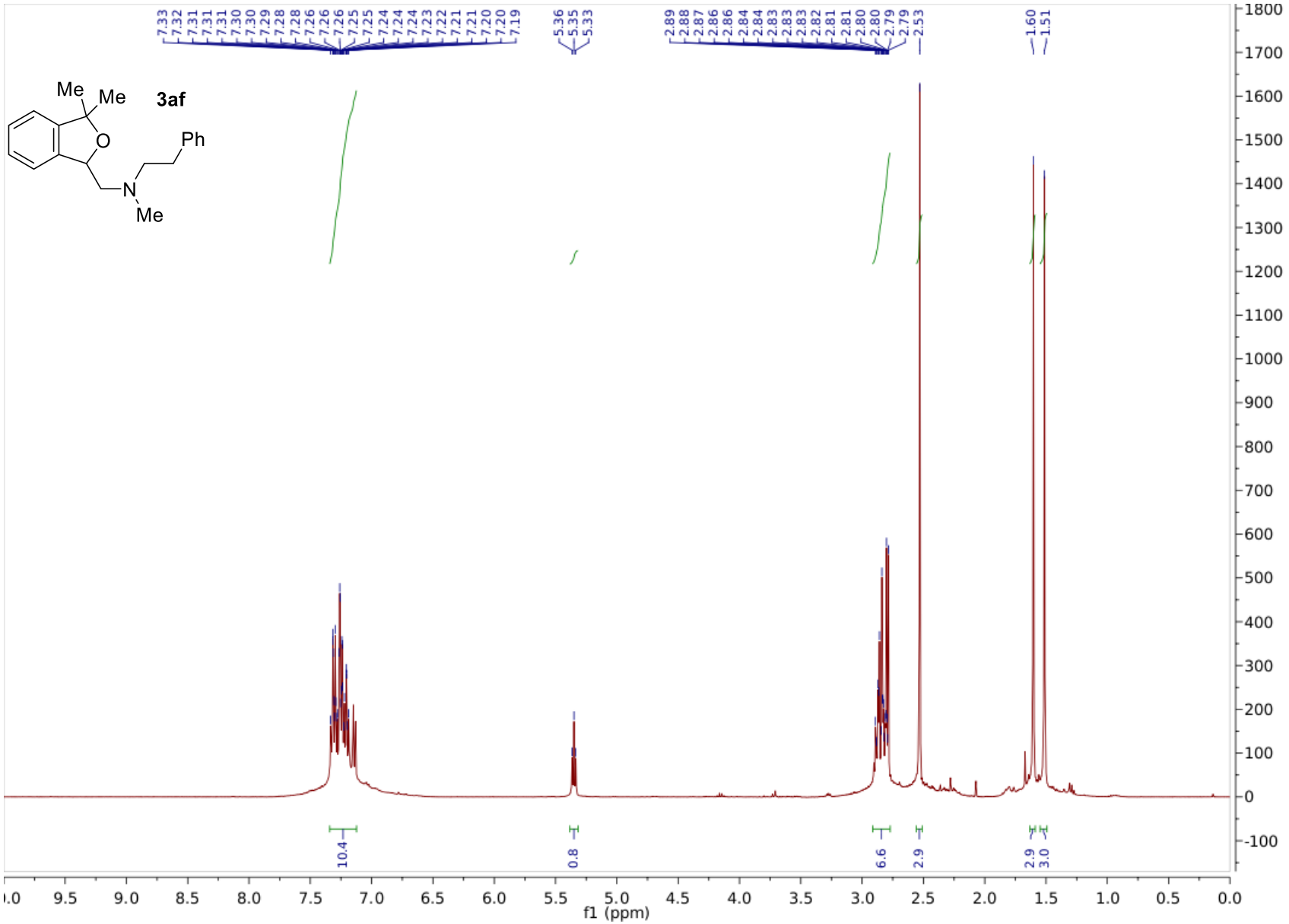

175.1
147.2
140.4
127.6
127.1
121.7
120.3
85.2
79.3
77.0
65.7
60.1
54.0
53.4
41.0
30.1
29.0
28.2
28.1
14.1
Me
Me
O
N
CO2Et
3ac
f1 (ppm)

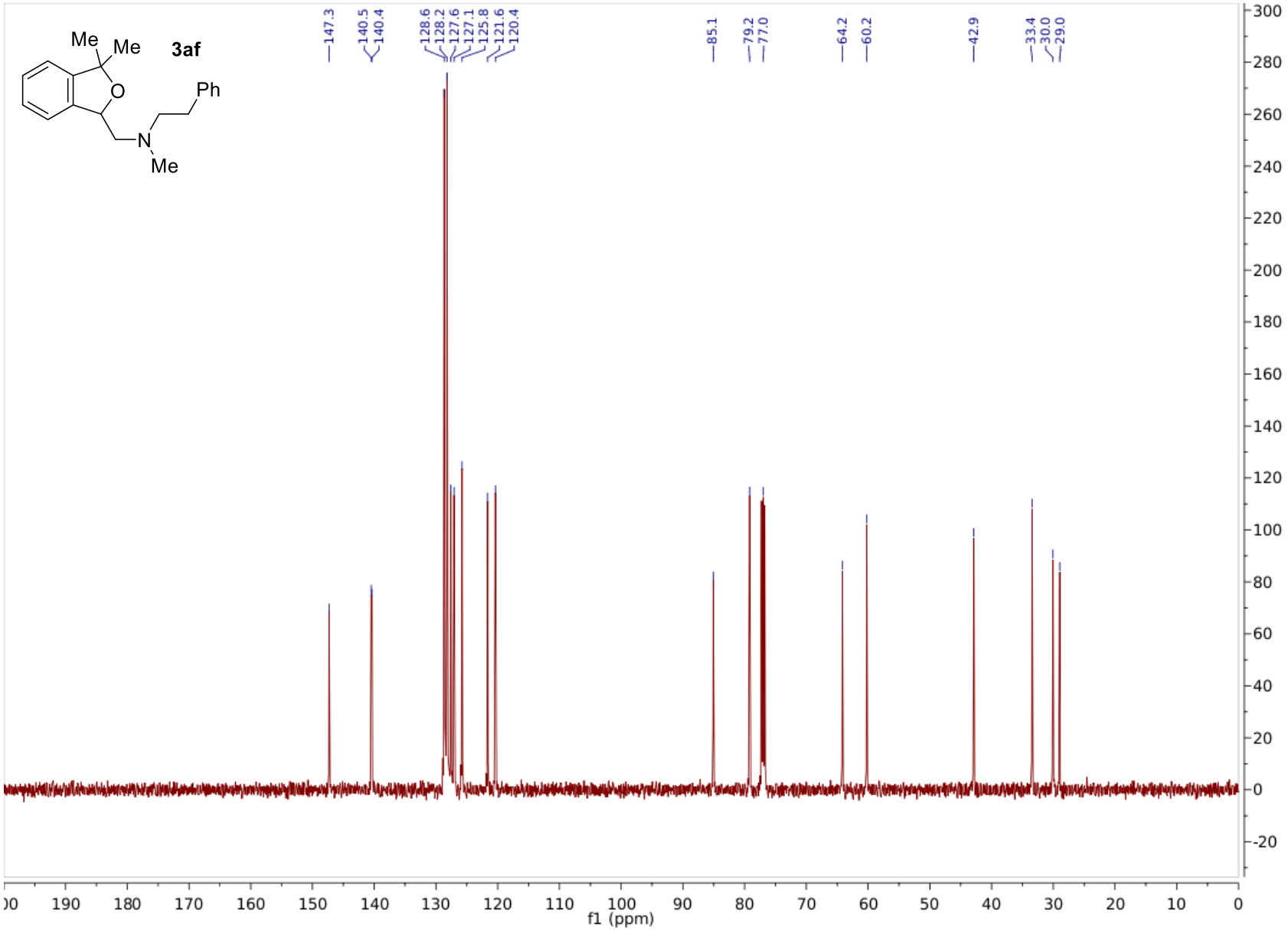

Me
Me
O
Et
N
Et
3ad
7.30
7.29
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.22
7.22
7.11
7.11
7.10
7.10
7.09
7.09
5.29
5.27
5.27
5.26
2.75
2.75
2.74
2.73
2.73
2.72
2.71
2.70
2.69
2.68
2.67
2.67
1.55
1.46
1.08
1.07
1.05
2.4
0.8
0.8
1.8
3.8
3.1
3.2
6.0
f1 (ppm)

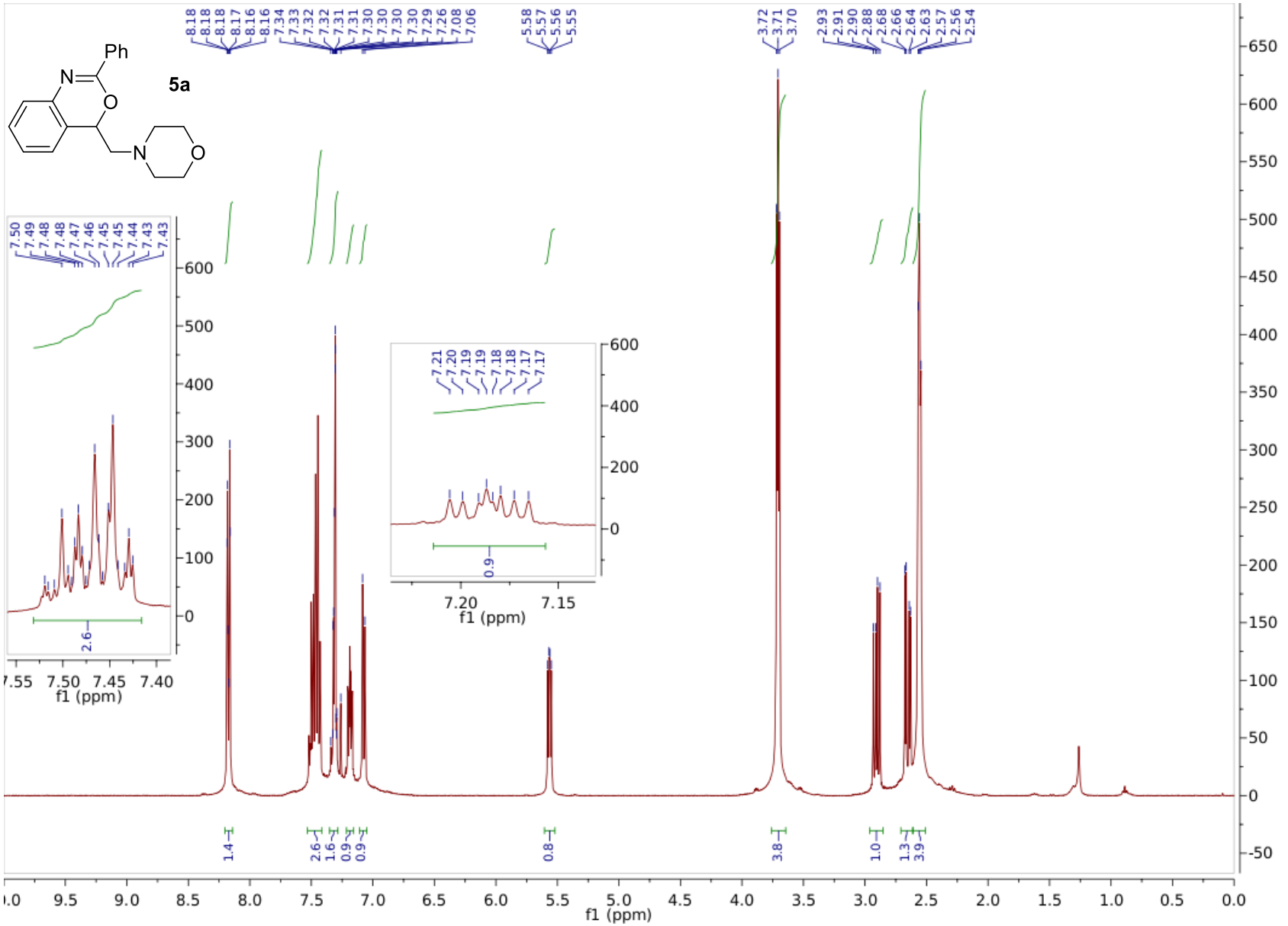

Me
Me
O
Et
N
Et
3ad
147.4
140.7
127.5
127.0
121.8
120.4
85.0
79.7
77.0
60.2
47.6
30.1
29.1
11.6
400
350
300
250
200
150
100
50
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

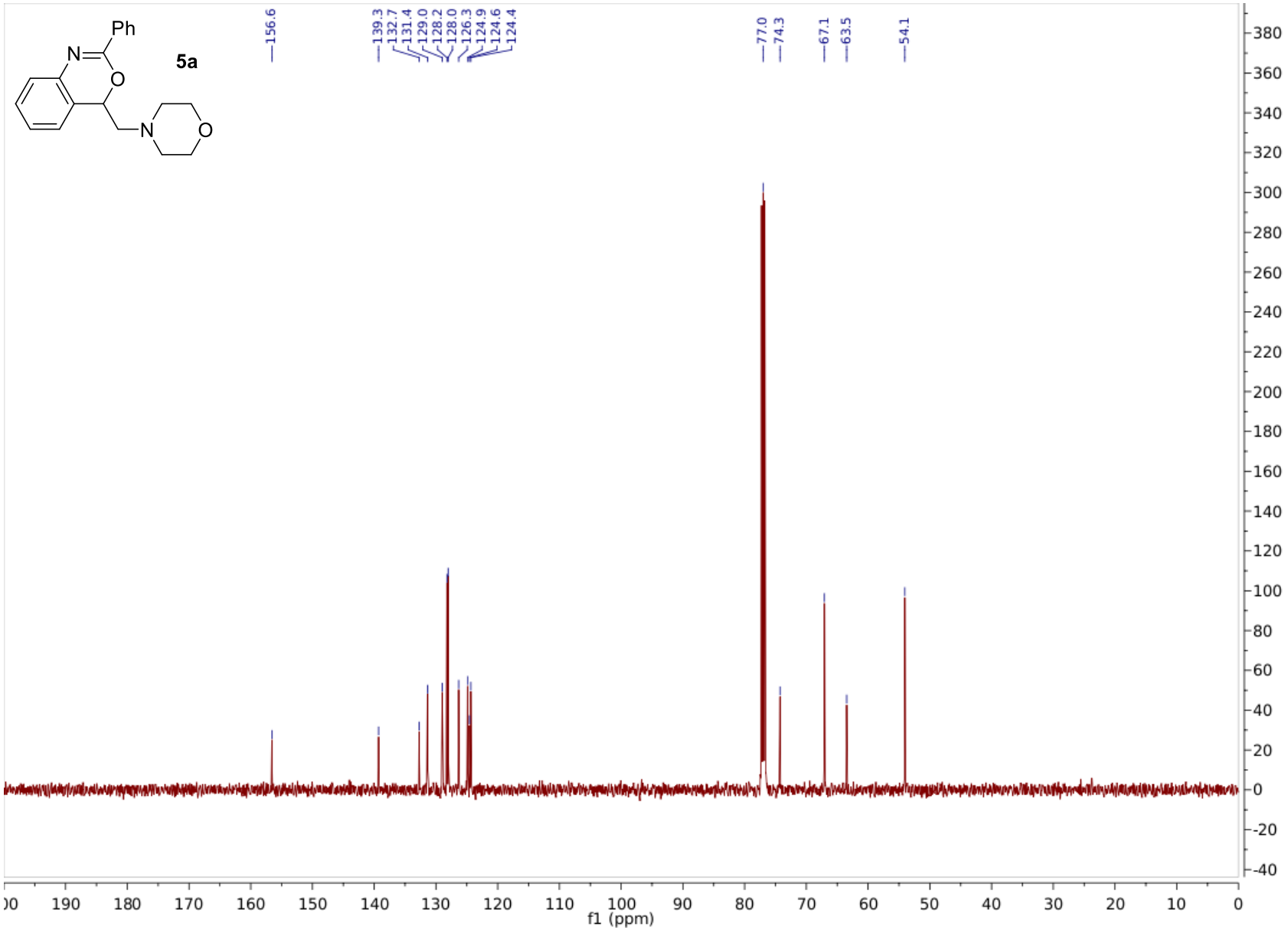

Me
Me
O
Ph
N
Me
3ae
7.39
7.39
7.37
7.37
7.37
7.35
7.34
7.33
7.32
7.31
7.31
7.30
7.29
7.28
7.28
7.27
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.13
7.12
7.11
5.40
5.39
5.37
3.70
2.75
2.74
2.73
2.43
1.53
1.49
8.4
0.9
0.8
1.9
1.7
3.2
3.0
3.2
f1 (ppm)

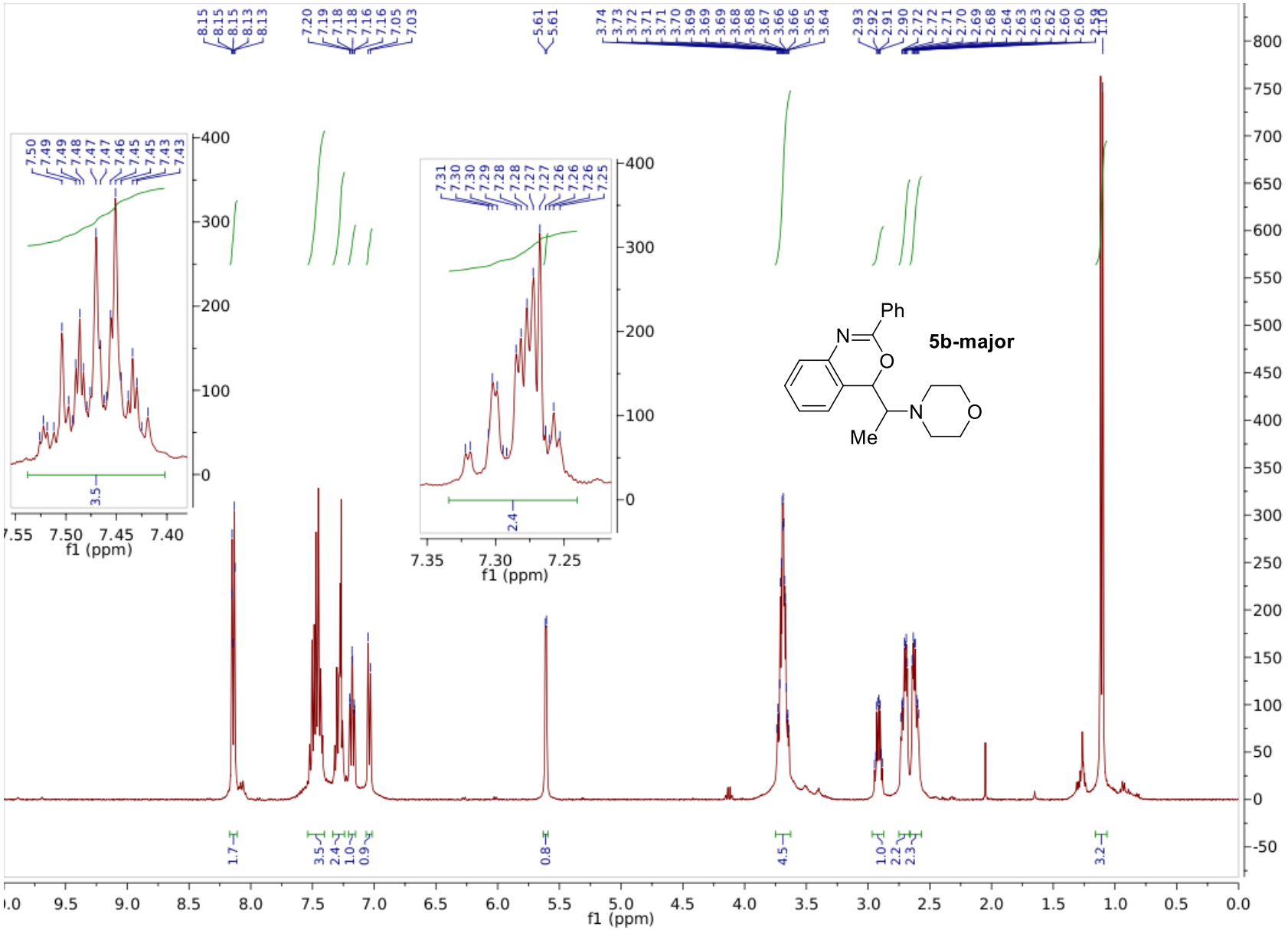

Me
Me
O
Ph
N
Me
3ae
147.3
140.5
138.8
129.1
128.1
127.6
127.1
126.8
121.7
120.4
85.2
79.4
77.0
63.8
62.9
43.0
30.0
29.1
f1 (ppm)

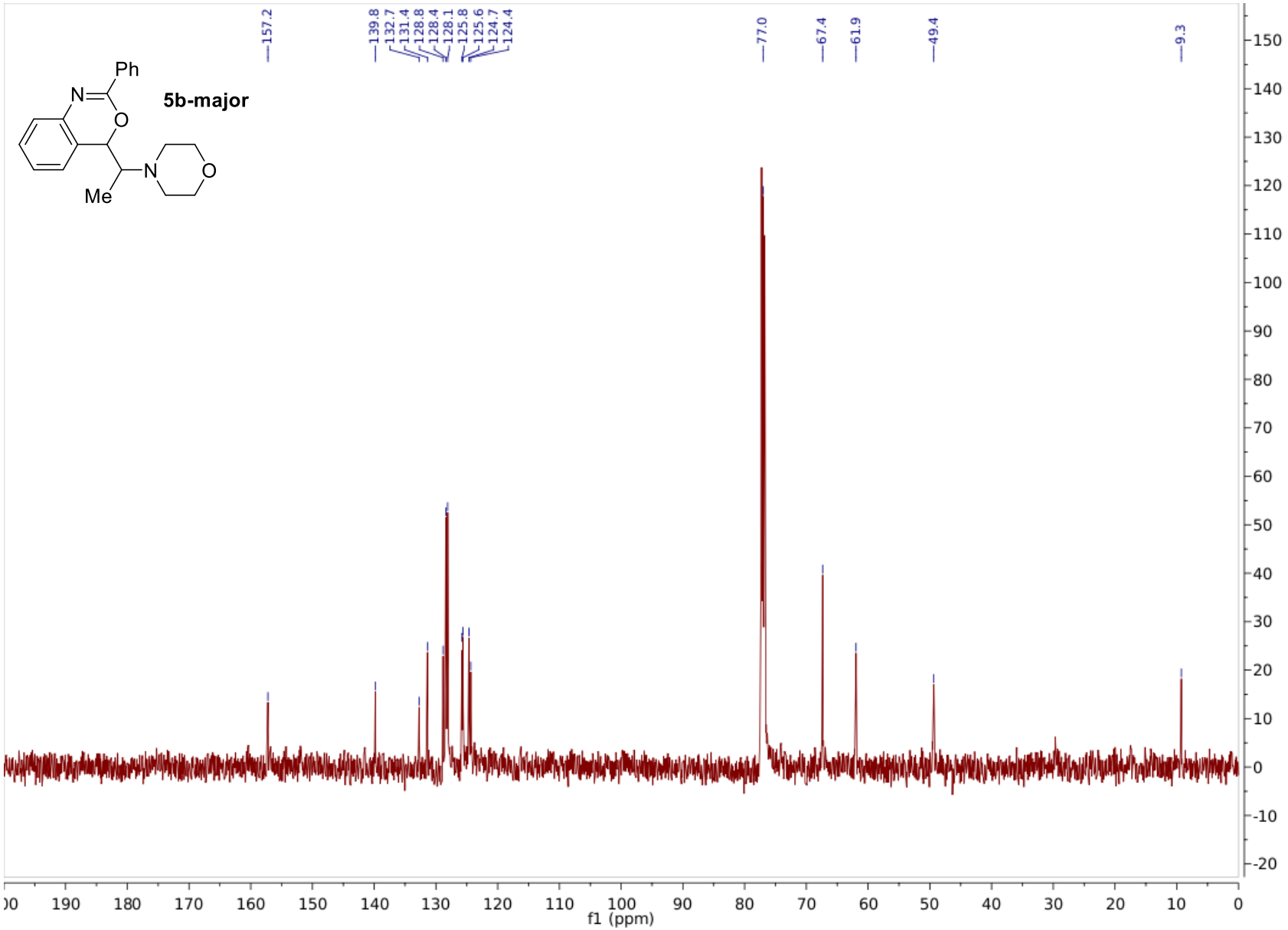

Me
Me
O
Ph
N
Me
3af
7.33 7.32 7.31 7.31 7.30 7.30 7.29 7.28 7.28 7.26 7.26 7.26 7.25 7.25 7.24 7.24 7.24 7.23 7.22 7.21 7.21 7.20 7.20 7.19
5.36 5.35 5.33
2.89 2.88 2.87 2.86 2.86 2.84 2.84 2.83 2.83 2.83 2.82 2.81 2.81 2.80 2.80 2.79 2.79 2.53
1.60 1.51
10.4
0.8
6.6
2.9
2.9
3.0
f1 (ppm)

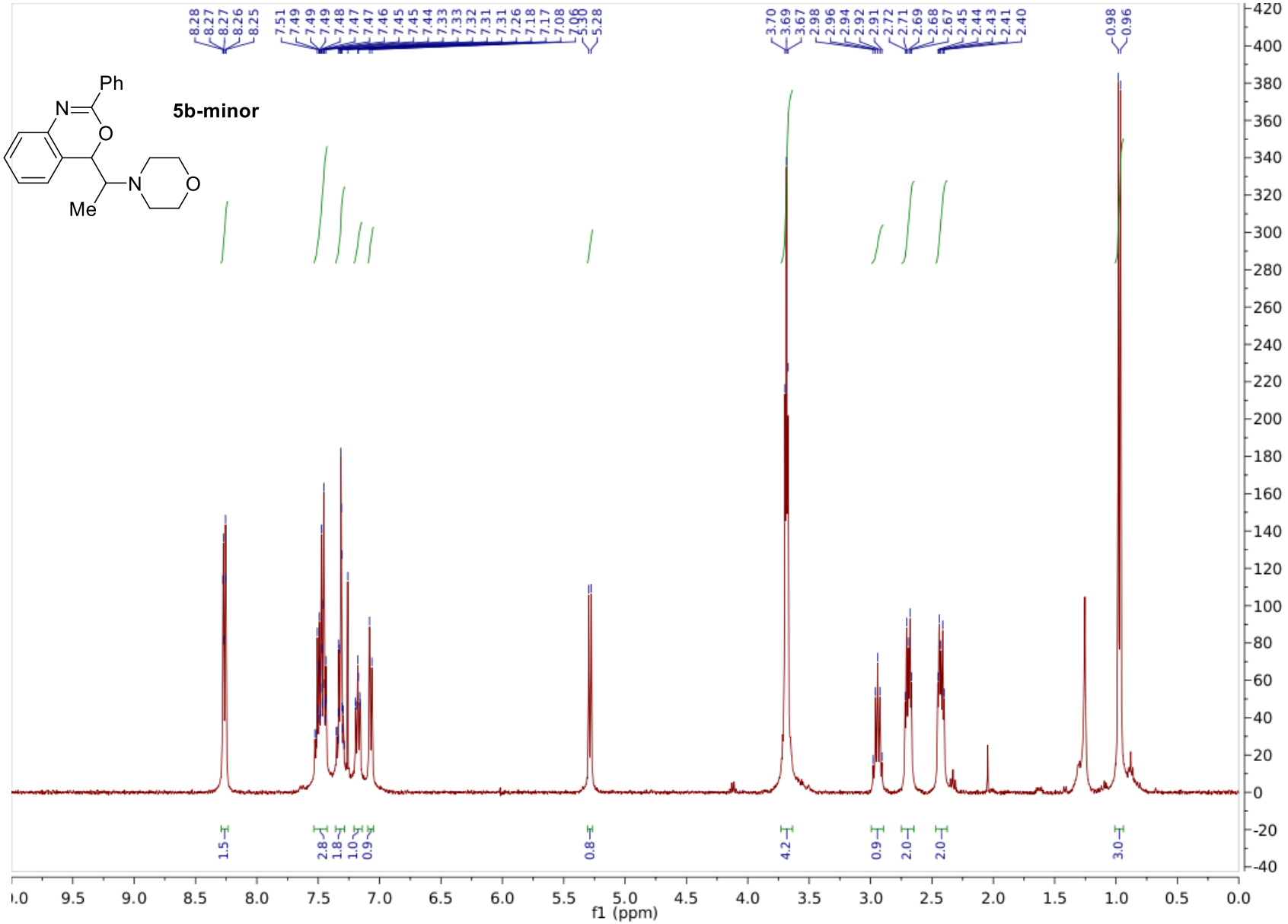

3af
Me
Me
O
Ph
N
Me
147.3
140.5
140.4
128.6
128.2
127.6
127.1
125.8
121.6
120.4
85.1
79.2
77.0
64.2
60.2
42.9
33.4
30.0
29.0
f1 (ppm)

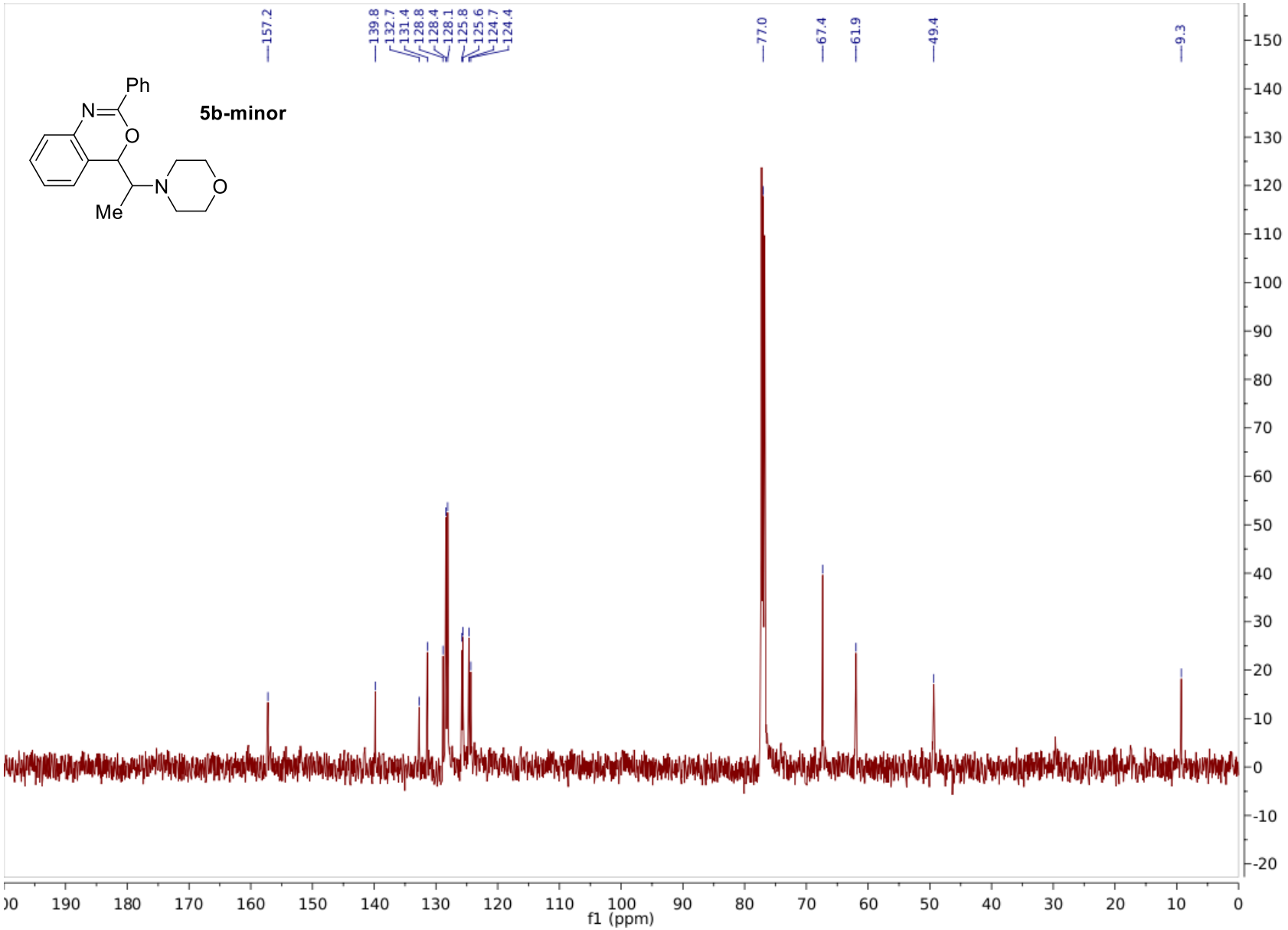

Ph
N
O
5a
N
O
f1 (ppm)

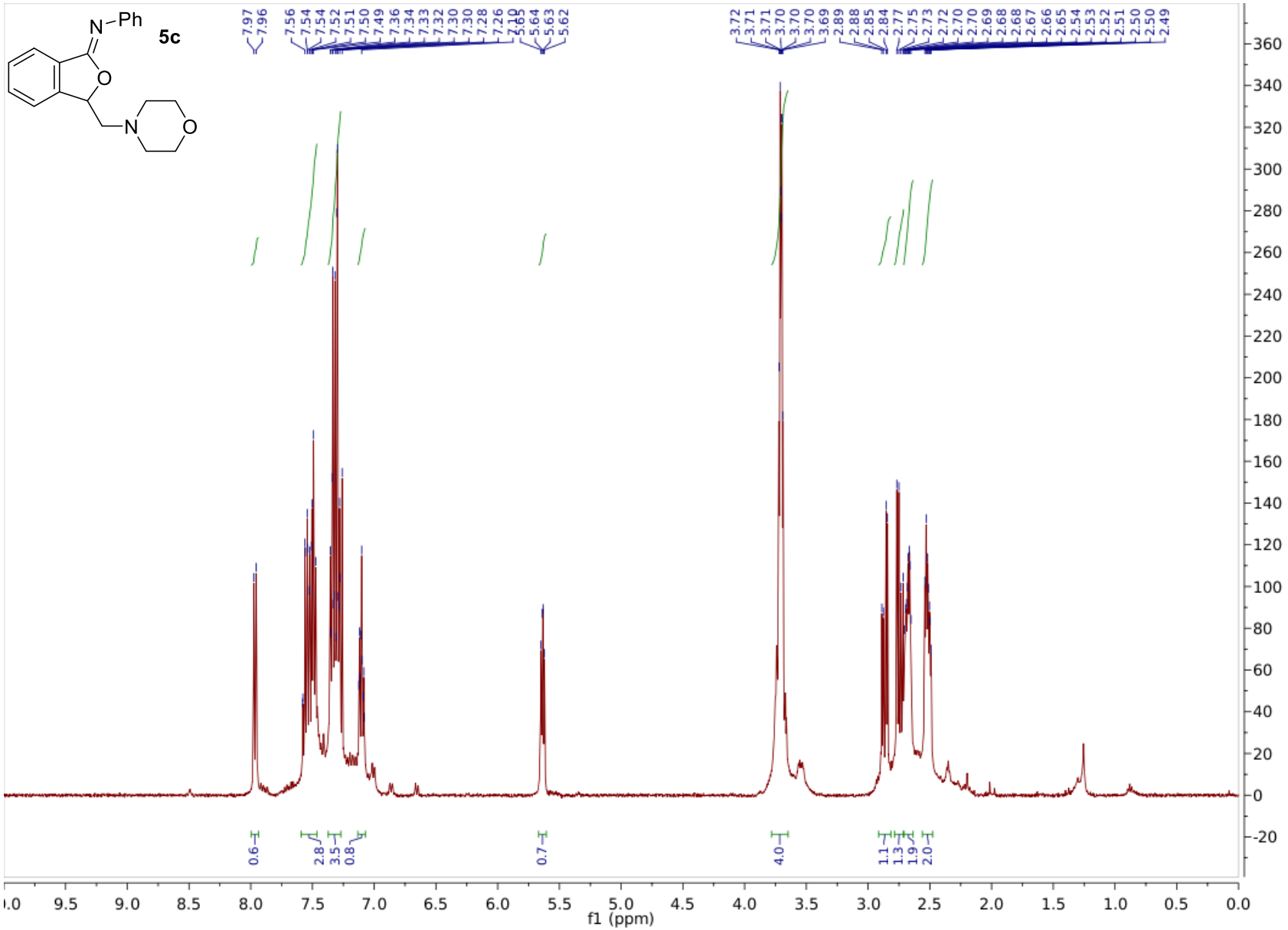

Ph
N
O
5a
N
O
156.6
139.3
132.7
131.4
129.0
128.2
128.0
126.3
124.9
124.6
124.4
77.0
74.3
67.1
63.5
54.1
380
360
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

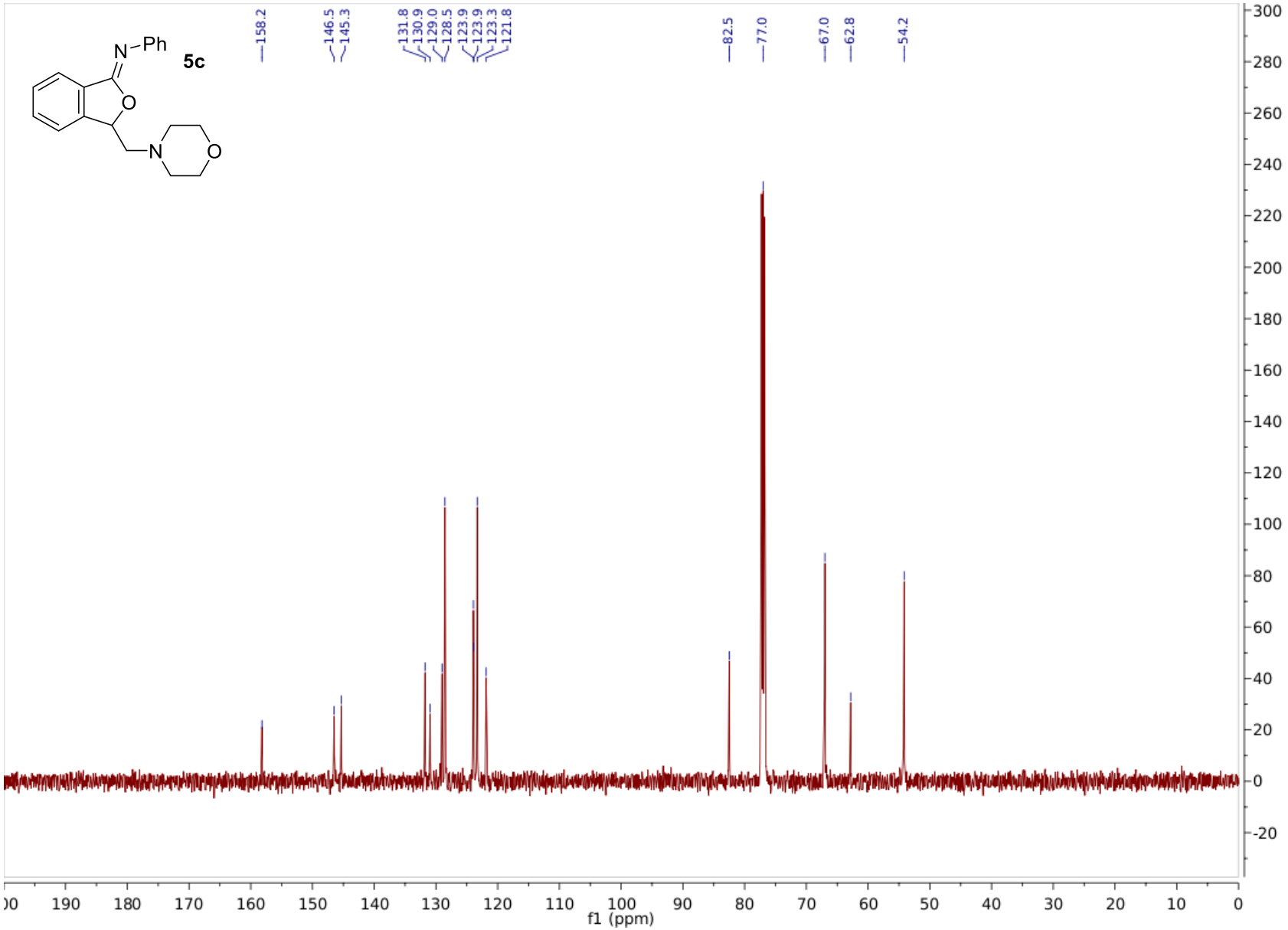

5b-major
Ph
N
O
N
O
Me
f1 (ppm)

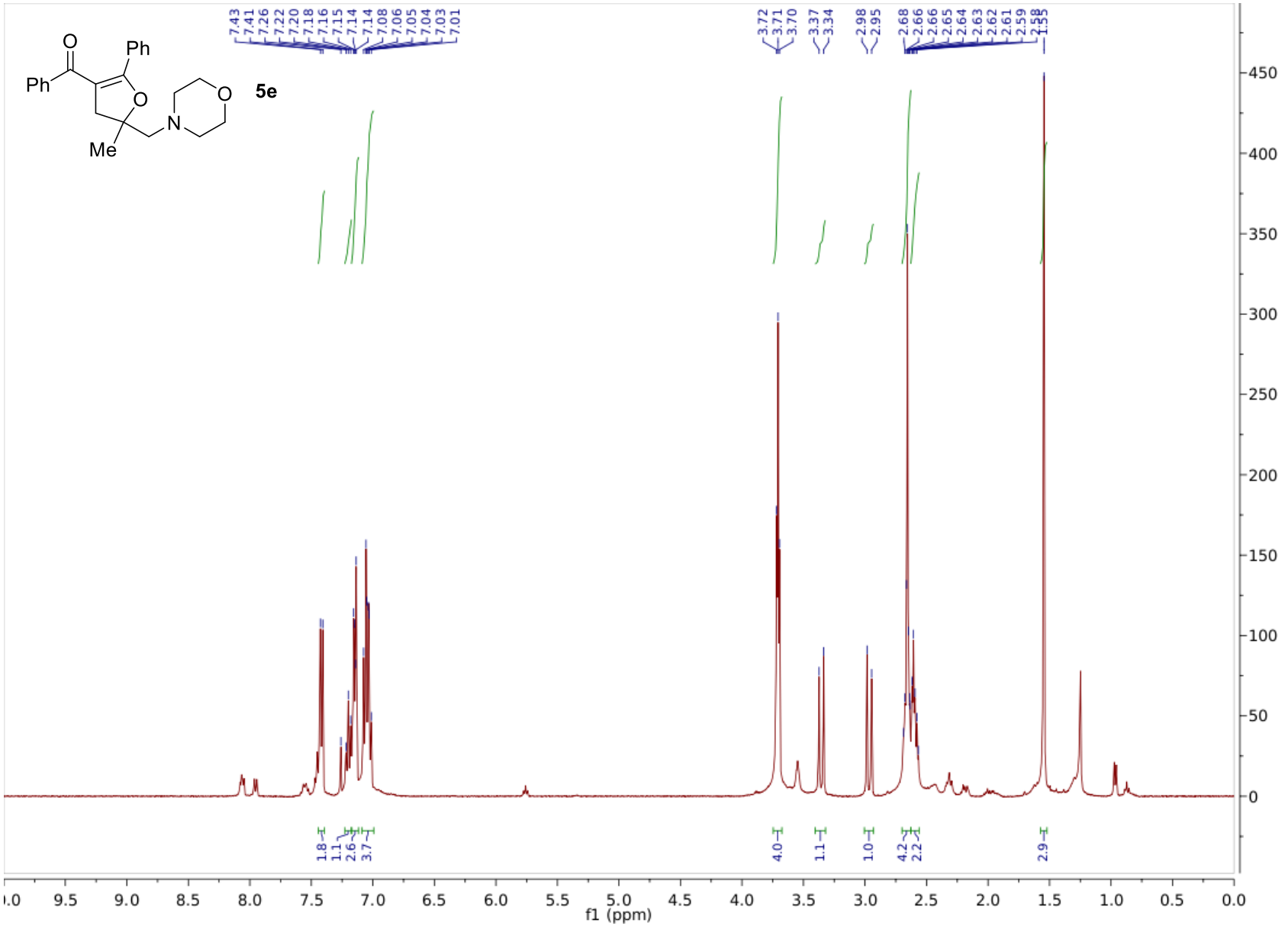

157.2
139.8
132.7
131.4
128.8
128.4
128.1
125.8
125.6
124.7
124.4
77.0
67.4
61.9
49.4
9.3
Ph
N
O
5b-major
N
O
Me
f1 (ppm)

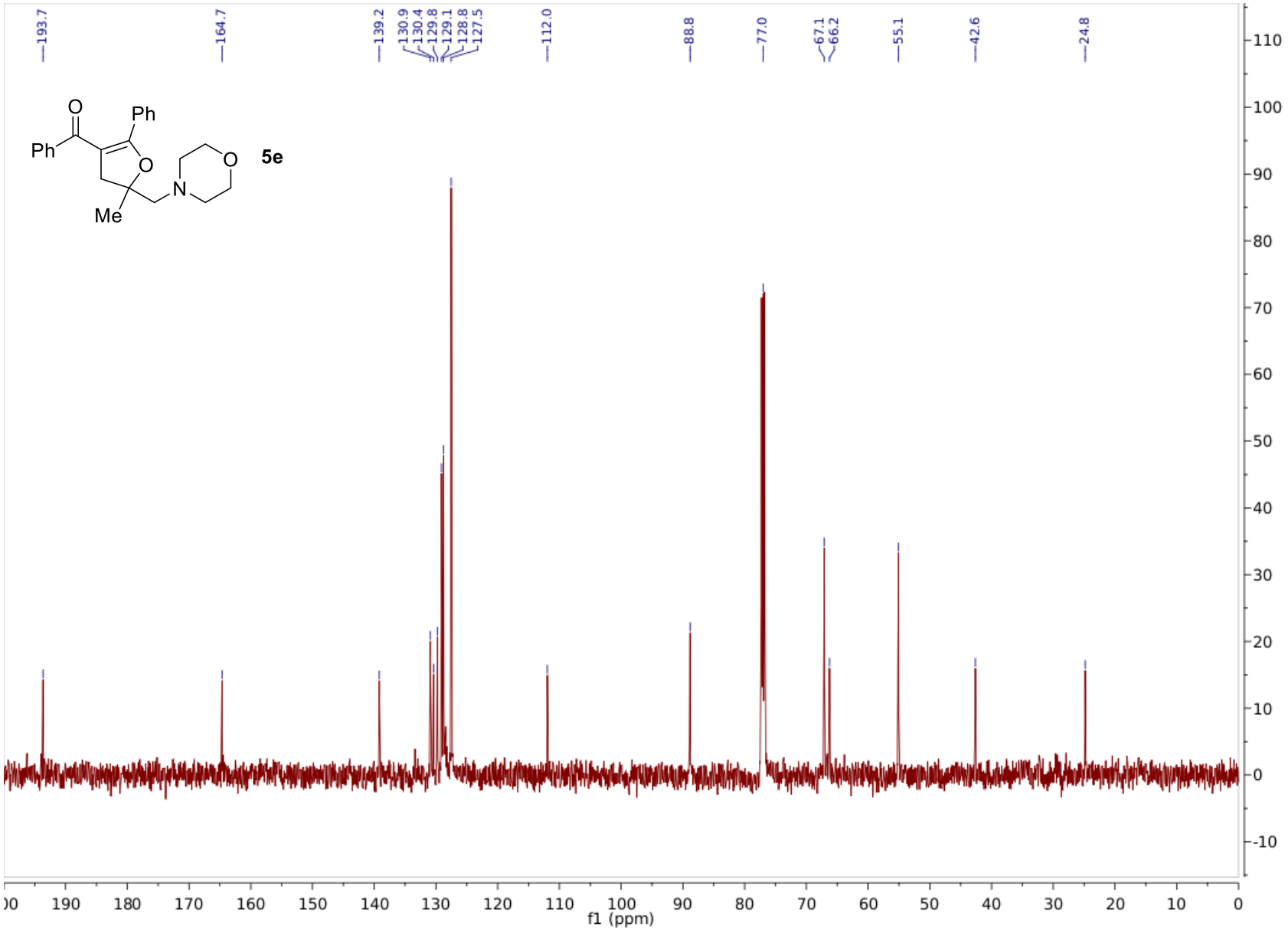

5b-minor
Ph
N
O
N
O
Me
8.28
8.27
8.27
8.26
8.25
7.51
7.49
7.49
7.49
7.48
7.47
7.47
7.46
7.45
7.45
7.44
7.33
7.33
7.32
7.31
7.31
7.26
7.18
7.17
7.08
7.06
5.30
5.28
3.70
3.69
3.67
2.98
2.96
2.94
2.92
2.91
2.72
2.71
2.69
2.68
2.67
2.45
2.44
2.43
2.41
2.40
0.98
0.96
1.5
2.8
1.8
1.0
0.9
0.8
4.2
0.9
2.0
2.0
3.0
f1 (ppm)

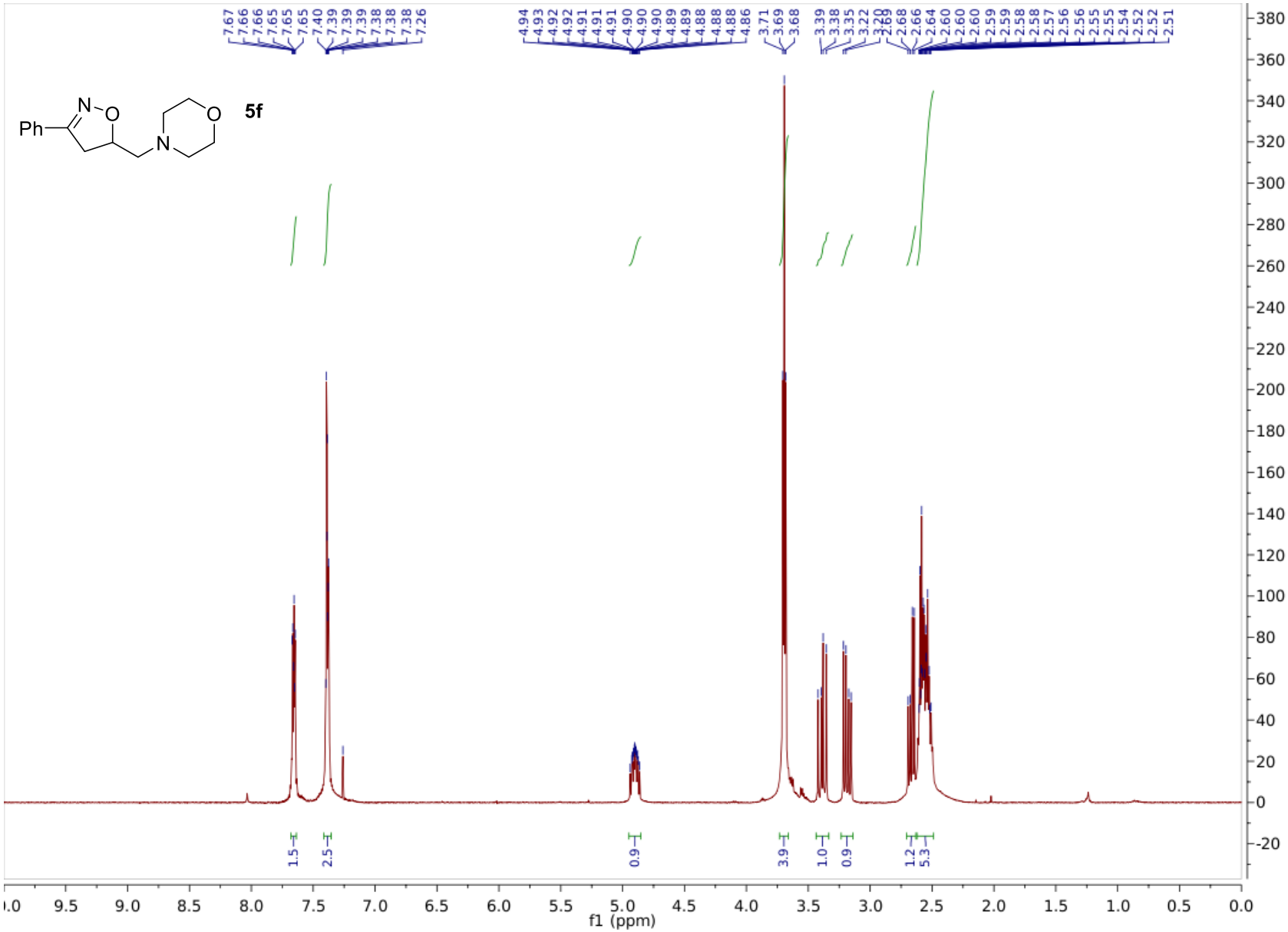

157.2
139.8
132.7
131.4
128.8
128.4
128.1
125.8
125.6
124.7
124.4
77.0
67.4
61.9
49.4
9.3
Ph
N
O
N
O
Me
5b-minor
f1 (ppm)

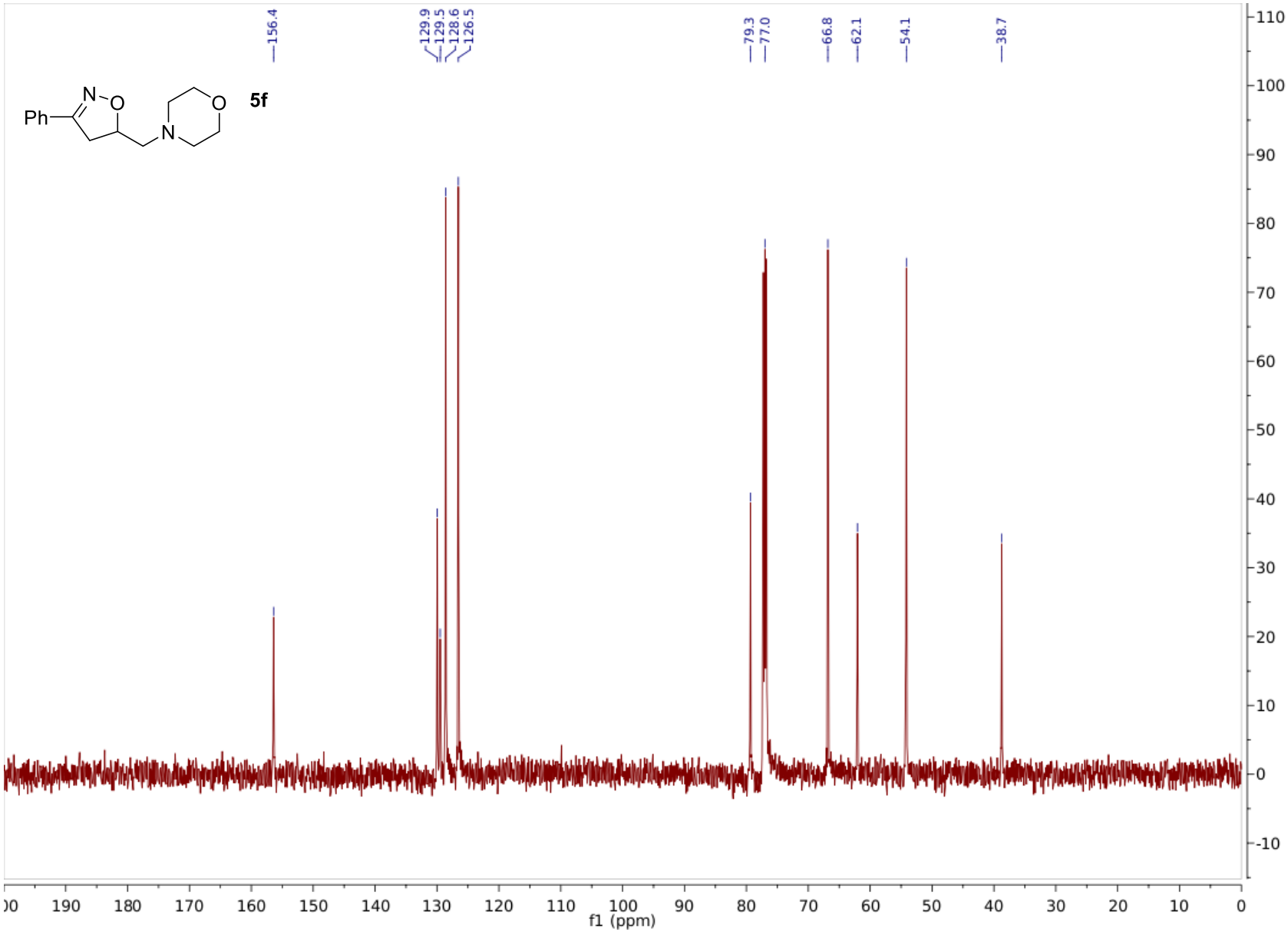

5c
N–Ph
O
N
O
7.97
7.96
7.56
7.54
7.54
7.52
7.51
7.50
7.49
7.36
7.34
7.33
7.32
7.30
7.30
7.28
7.26
5.64
5.63
5.62
3.72
3.71
3.71
3.70
3.70
3.70
3.69
2.89
2.88
2.85
2.84
2.77
2.75
2.73
2.72
2.70
2.70
2.69
2.68
2.68
2.67
2.66
2.65
2.54
2.53
2.52
2.51
2.50
2.50
2.49
0.6
2.8
3.5
0.8
0.7
4.0
1.1
1.3
1.9
2.0
f1 (ppm)

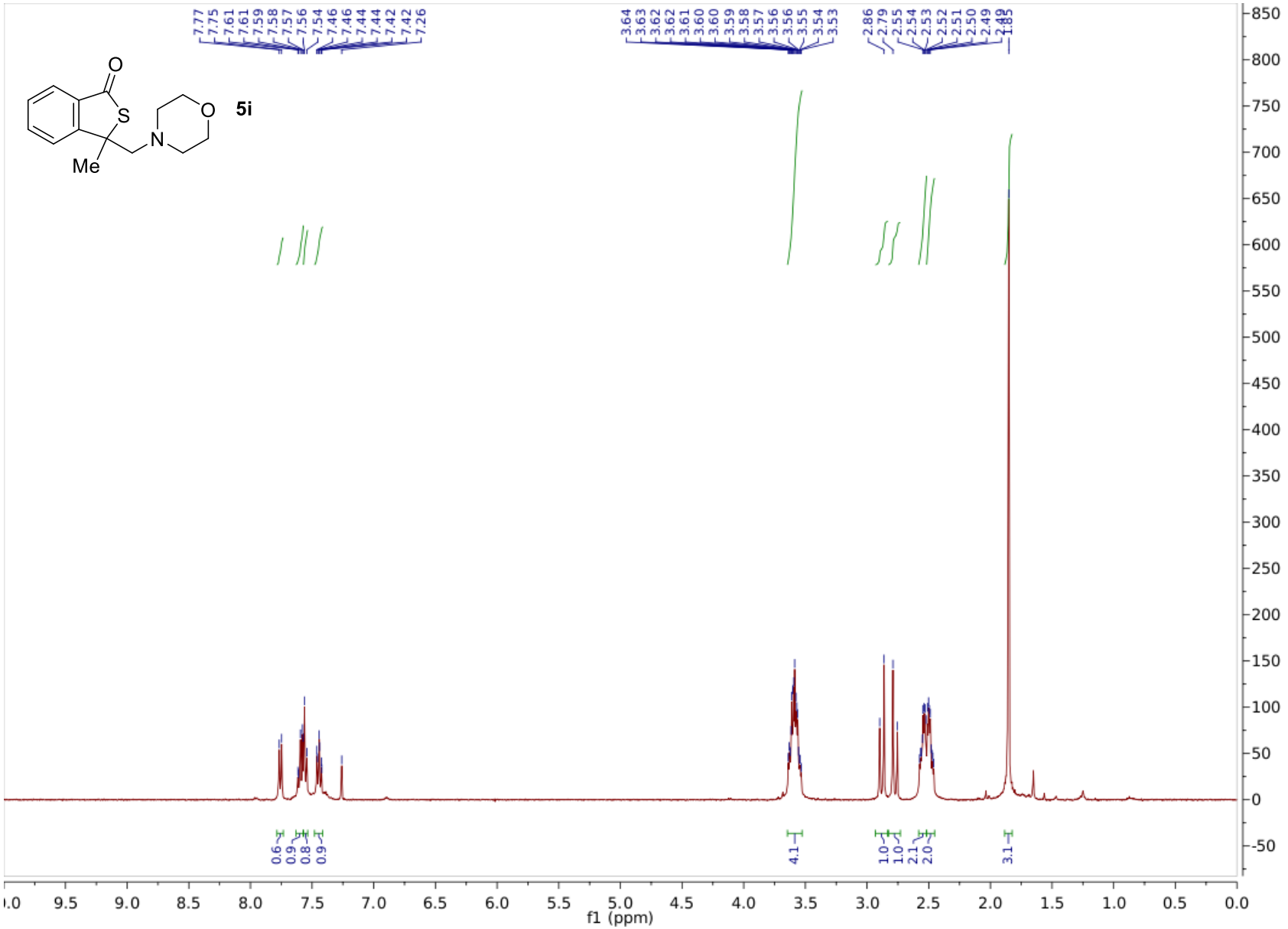

N–Ph
5c
O
N
O
158.2
146.5
145.3
131.8
130.9
129.0
128.5
123.9
123.9
123.3
121.8
82.5
77.0
67.0
62.8
54.2
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

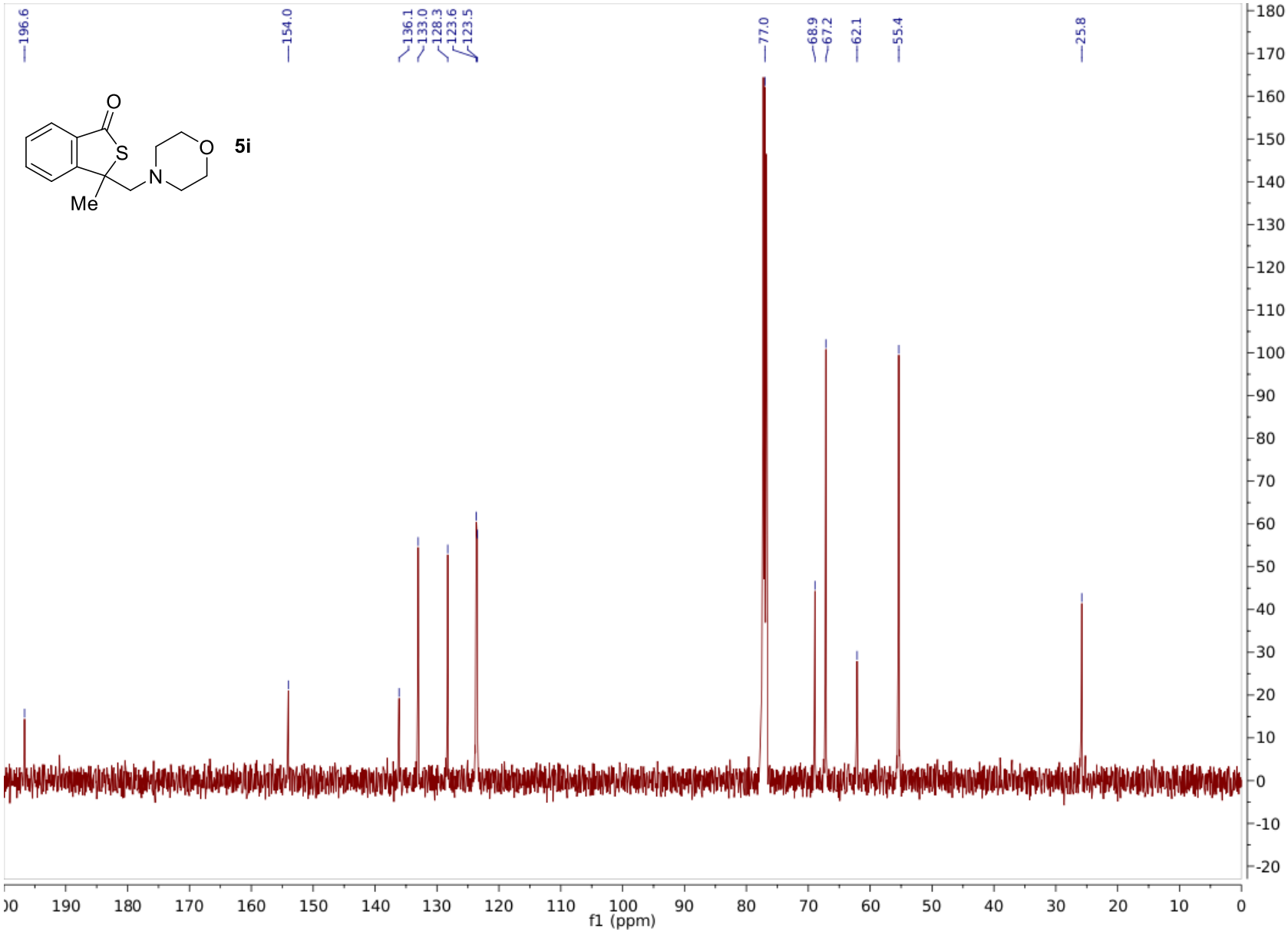

5e
f1 (ppm)

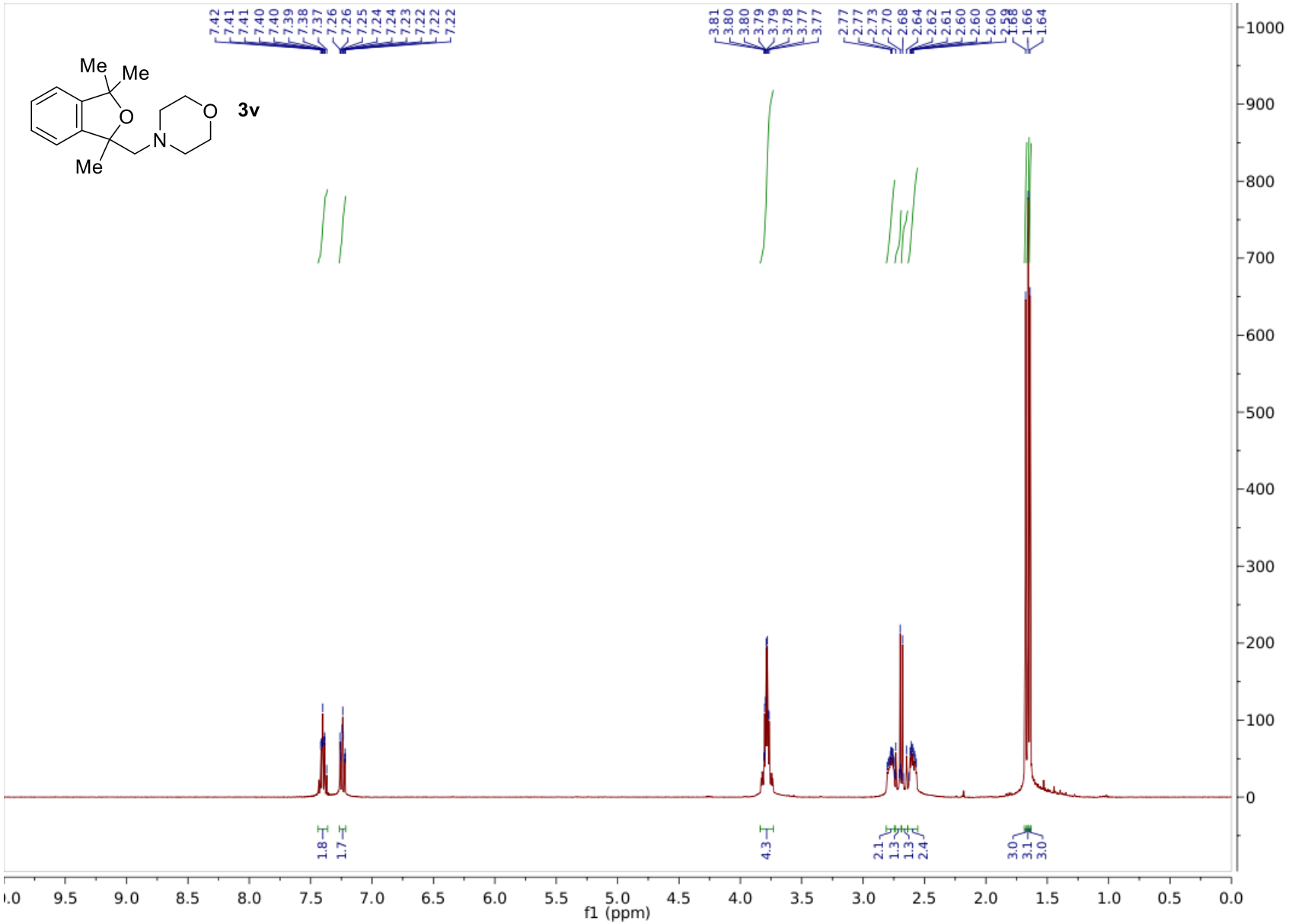

193.7
164.7
139.2
130.9
130.4
129.8
129.1
128.8
127.5
112.0
88.8
77.0
67.1
66.2
55.1
42.6
24.8
O
Ph
Ph
O
O
N
Me
5e
f1 (ppm)

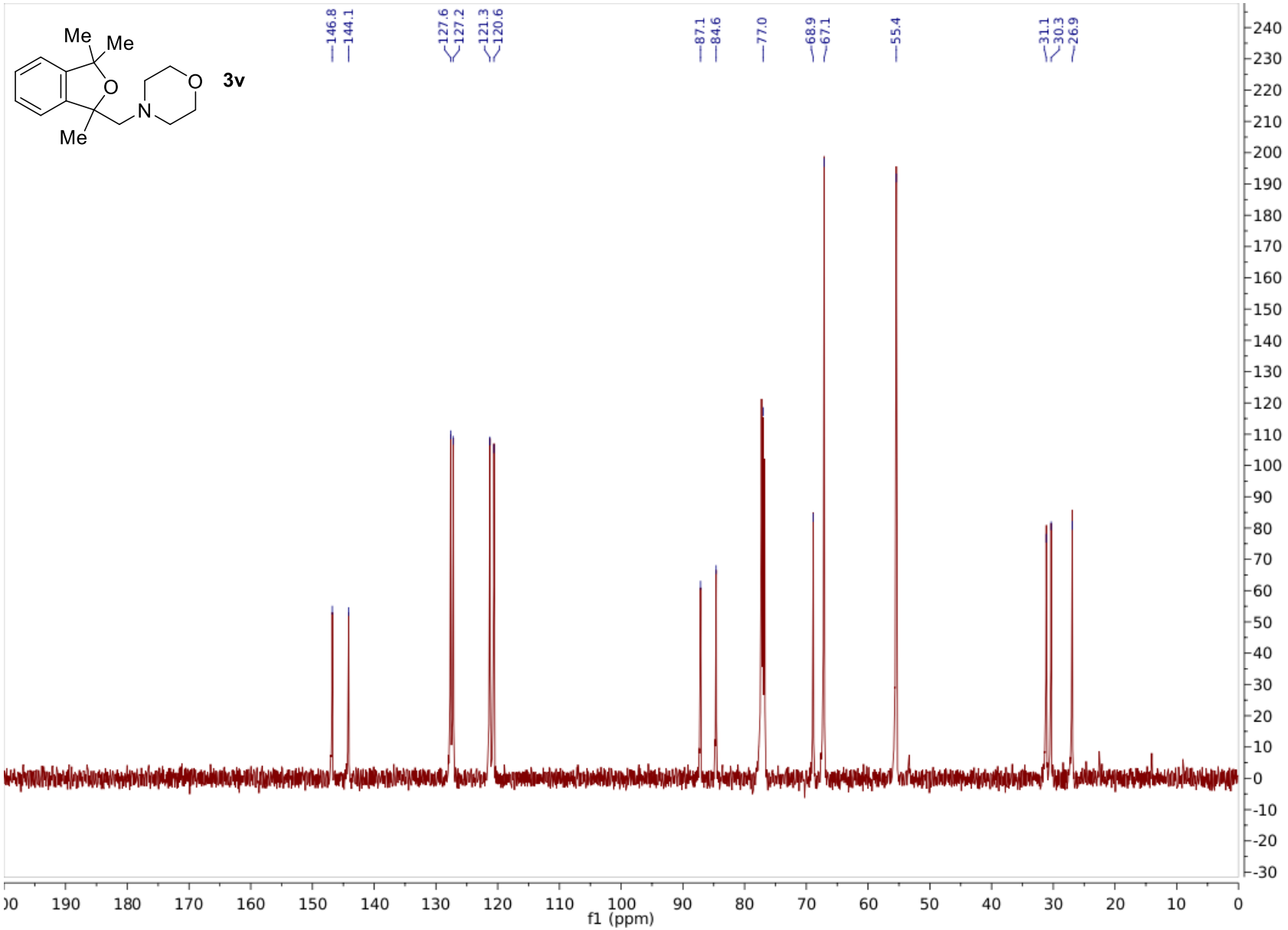

5f
Ph
N
O
O
N
7.67 7.66 7.66 7.65 7.65 7.65 7.40 7.39 7.39 7.39 7.38 7.38 7.38 7.26
4.94 4.93 4.92 4.92 4.91 4.91 4.90 4.90 4.90 4.89 4.89 4.88 4.88 4.88 4.86
3.71 3.69 3.68
3.39 3.38 3.35 3.22 3.20 3.19 2.68 2.66 2.64 2.60 2.60 2.60 2.59 2.59 2.58 2.58 2.57 2.56 2.56 2.55 2.54 2.52 2.52 2.51
1.5 2.5 0.9 3.9 1.0 0.9 1.2 5.3
f1 (ppm)

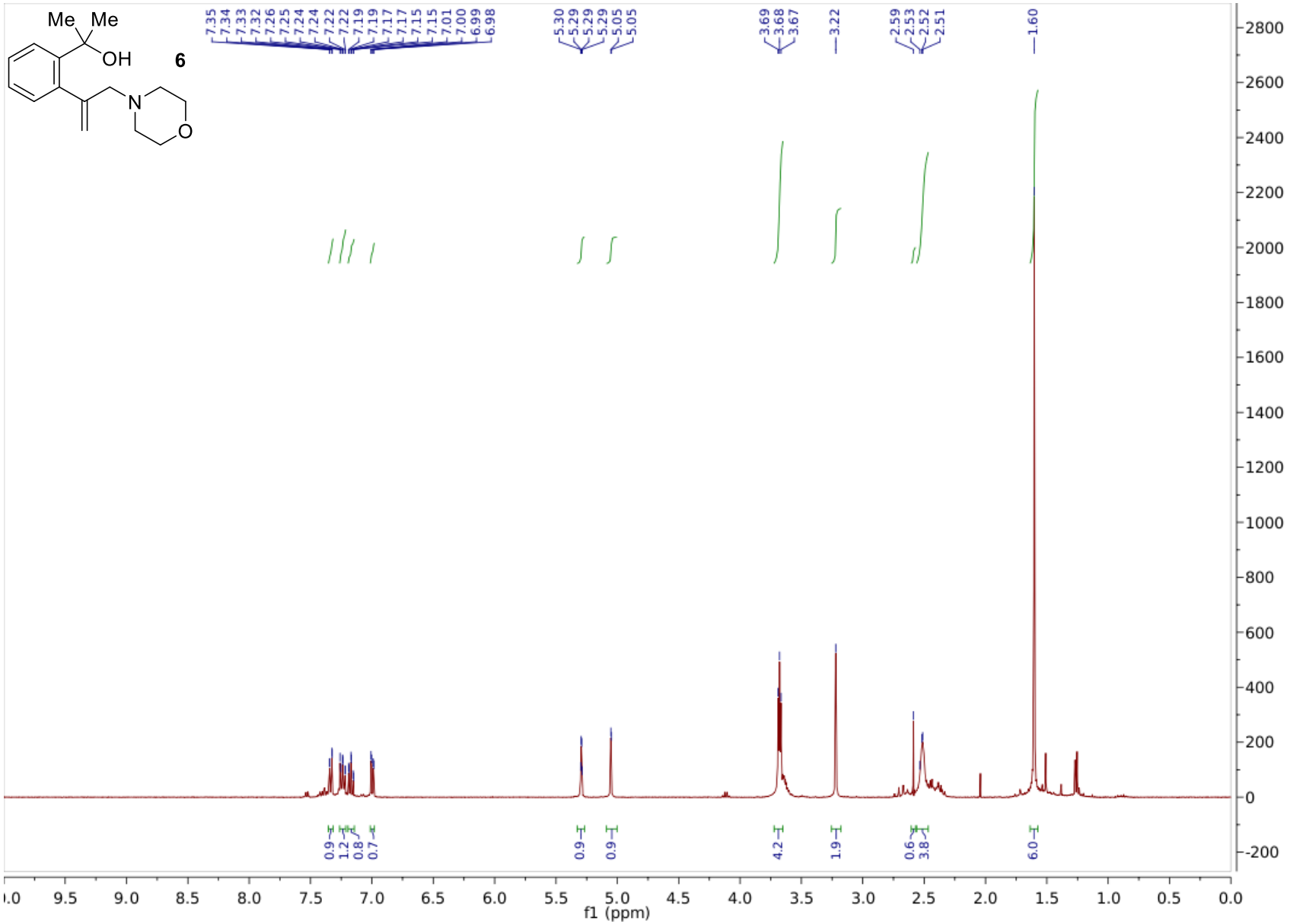

156.4
129.9
129.5
128.6
126.5
79.3
77.0
66.8
62.1
54.1
38.7
Ph
N
O
O
N
5f
110
100
90
80
70
60
50
40
30
20
10
0
-10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

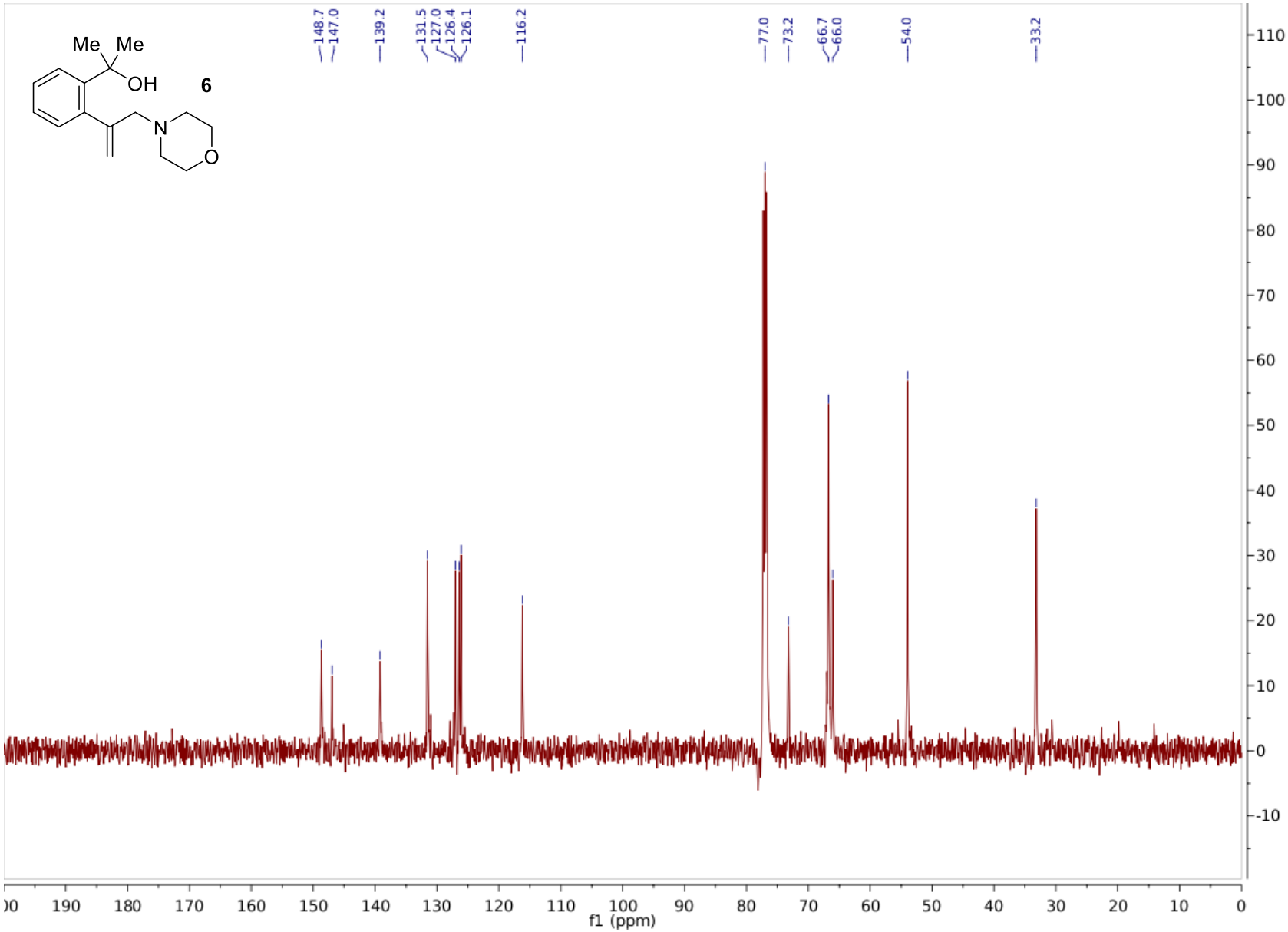

5i
O
S
O
N
Me
7.77
7.75
7.61
7.61
7.59
7.58
7.57
7.56
7.54
7.46
7.46
7.44
7.44
7.42
7.42
7.26
3.64
3.63
3.62
3.62
3.61
3.60
3.60
3.59
3.58
3.57
3.56
3.56
3.55
3.54
3.53
2.86
2.79
2.55
2.54
2.53
2.52
2.51
2.50
2.49
2.49
1.85
0.6
0.9
0.8
0.9
4.1
1.0
1.0
2.1
2.0
3.1
f1 (ppm)

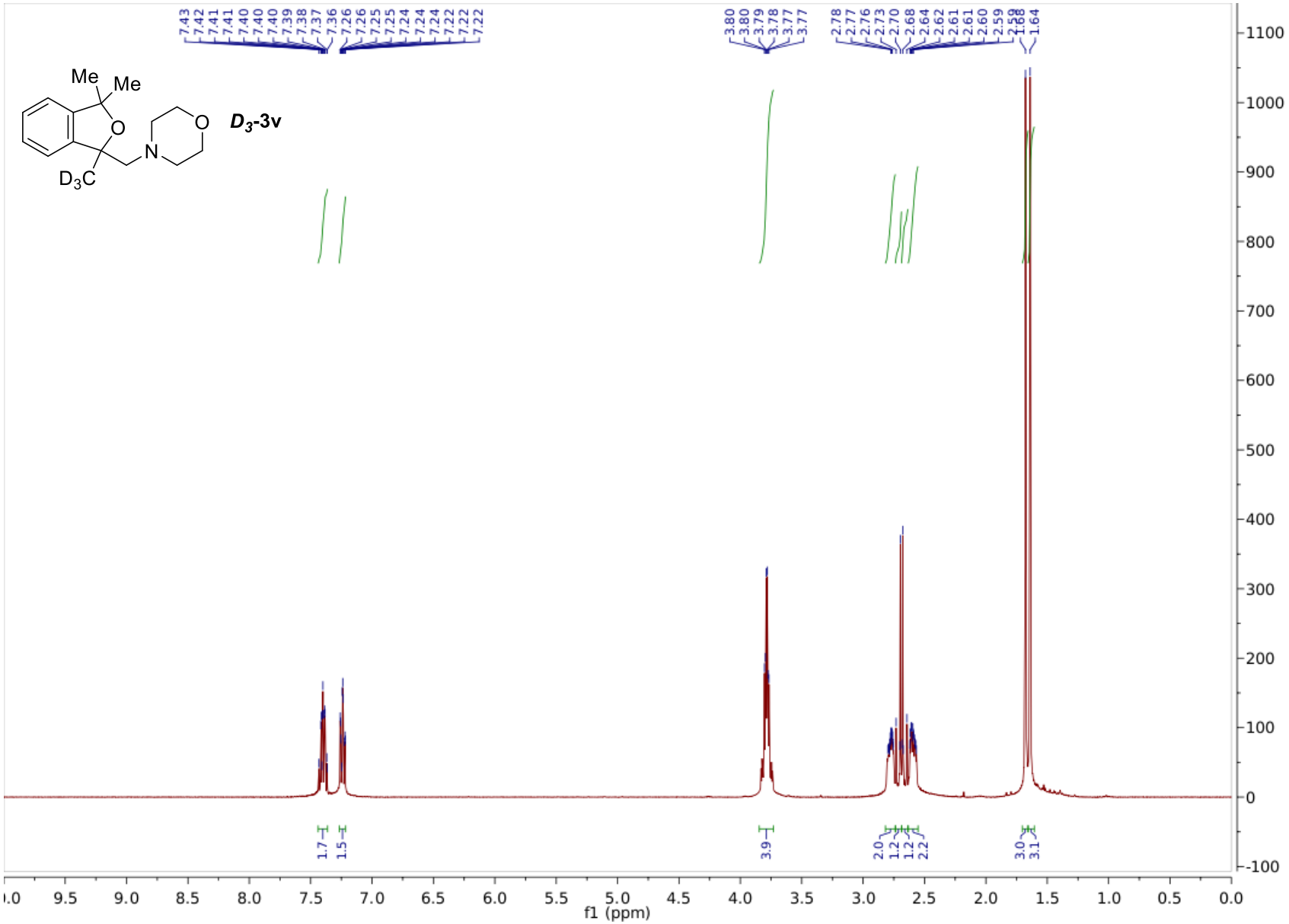

196.6
154.0
136.1
133.0
128.3
123.6
123.5
77.0
68.9
67.2
62.1
55.4
25.8
O
S
N
O
Me
5i
f1 (ppm)

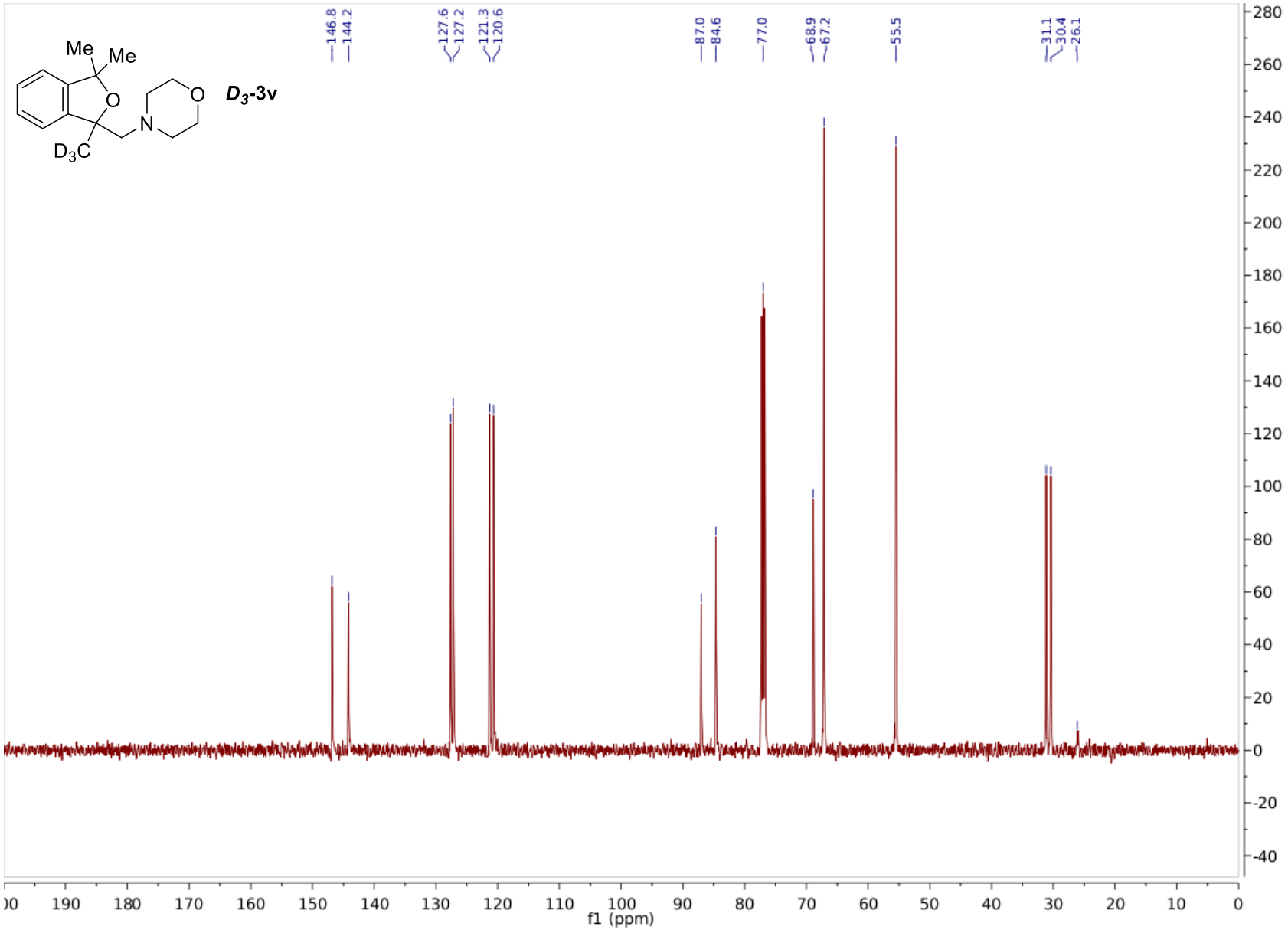

Me
Me
O
O
N
Me
3v
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.37
7.26
7.26
7.25
7.24
7.24
7.23
7.22
7.22
7.22
3.81
3.80
3.80
3.79
3.79
3.78
3.77
3.77
2.77
2.77
2.73
2.70
2.68
2.64
2.62
2.61
2.60
2.60
2.60
2.59
1.66
1.64
1000
900
800
700
600
500
400
300
200
100
0
1.8
1.7
4.3
2.1
1.3
1.3
2.4
3.0
3.1
3.0
f1 (ppm)

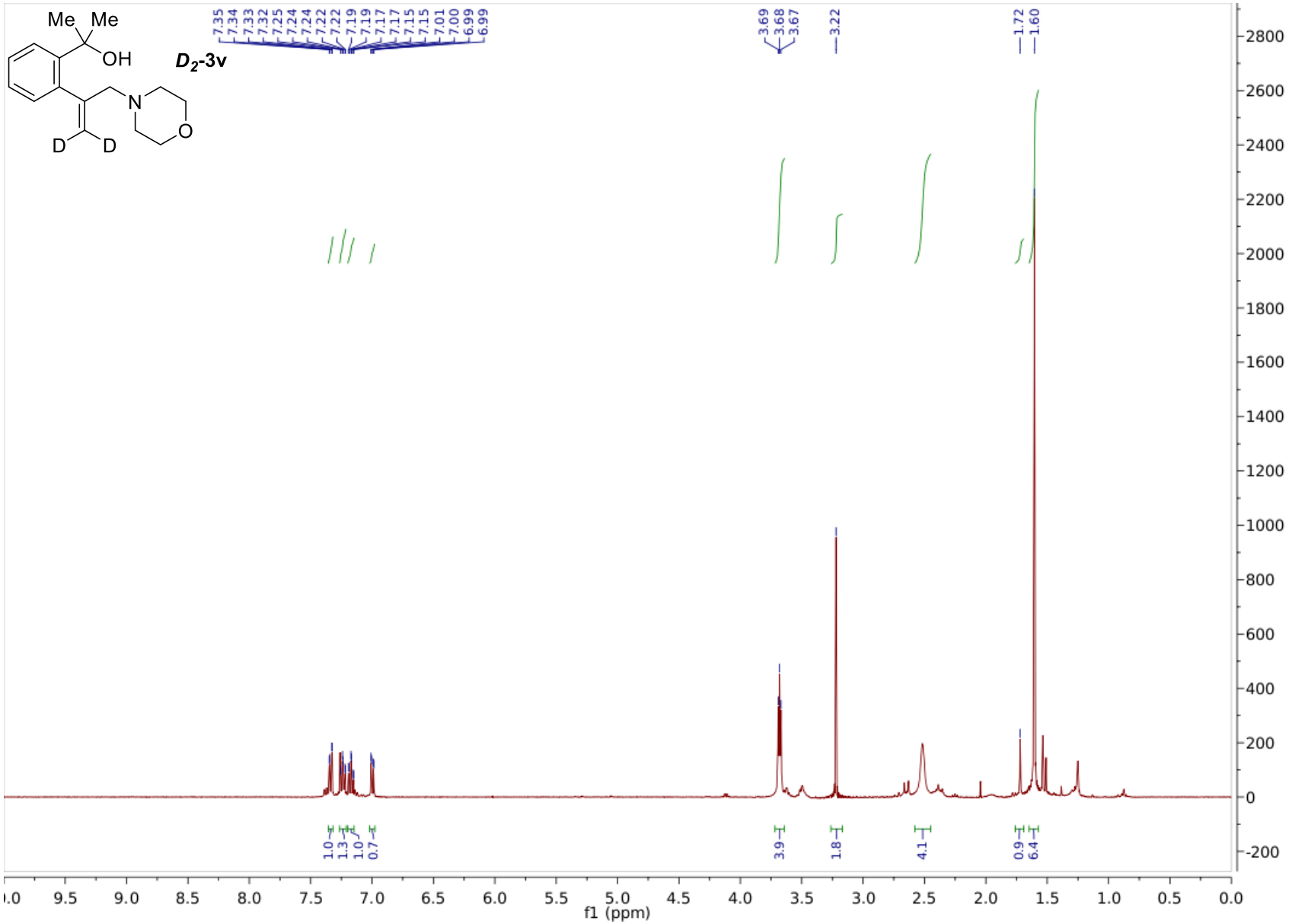

Me
Me
O
O
N
Me
3v
146.8
144.1
127.6
127.2
121.3
120.6
87.1
84.6
77.0
68.9
67.1
55.4
31.1
30.3
26.9
f1 (ppm)

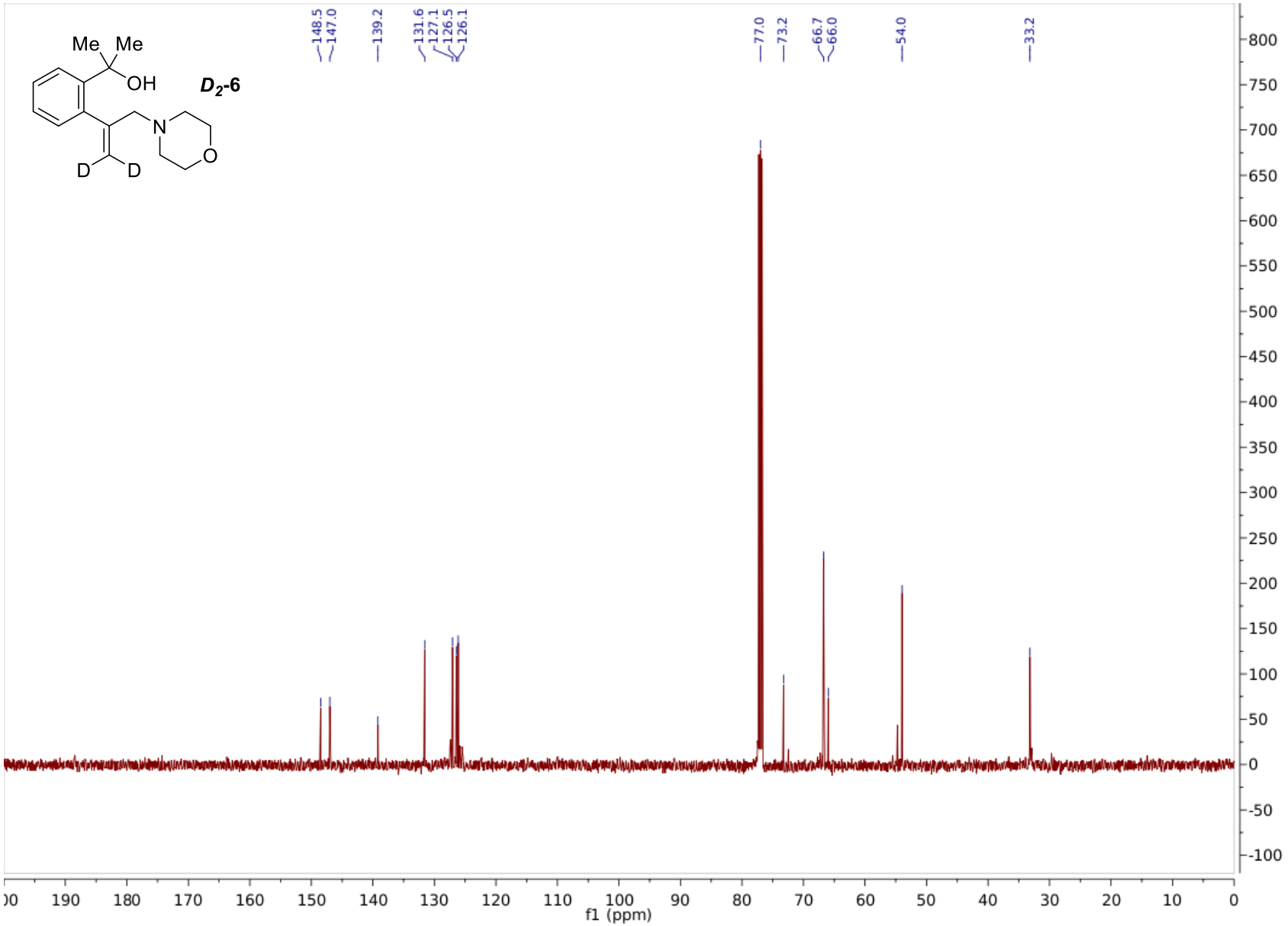

Me Me
OH
6
N
O
7.35
7.34
7.33
7.32
7.26
7.25
7.24
7.24
7.22
7.22
7.19
7.19
7.17
7.17
7.15
7.15
7.01
7.00
6.99
6.98
5.30
5.29
5.29
5.29
5.05
5.05
3.69
3.68
3.67
3.22
2.59
2.53
2.52
2.51
1.60
0.9
1.2
0.8
0.7
0.9
0.9
4.2
1.9
0.6
3.8
6.0
f1 (ppm)

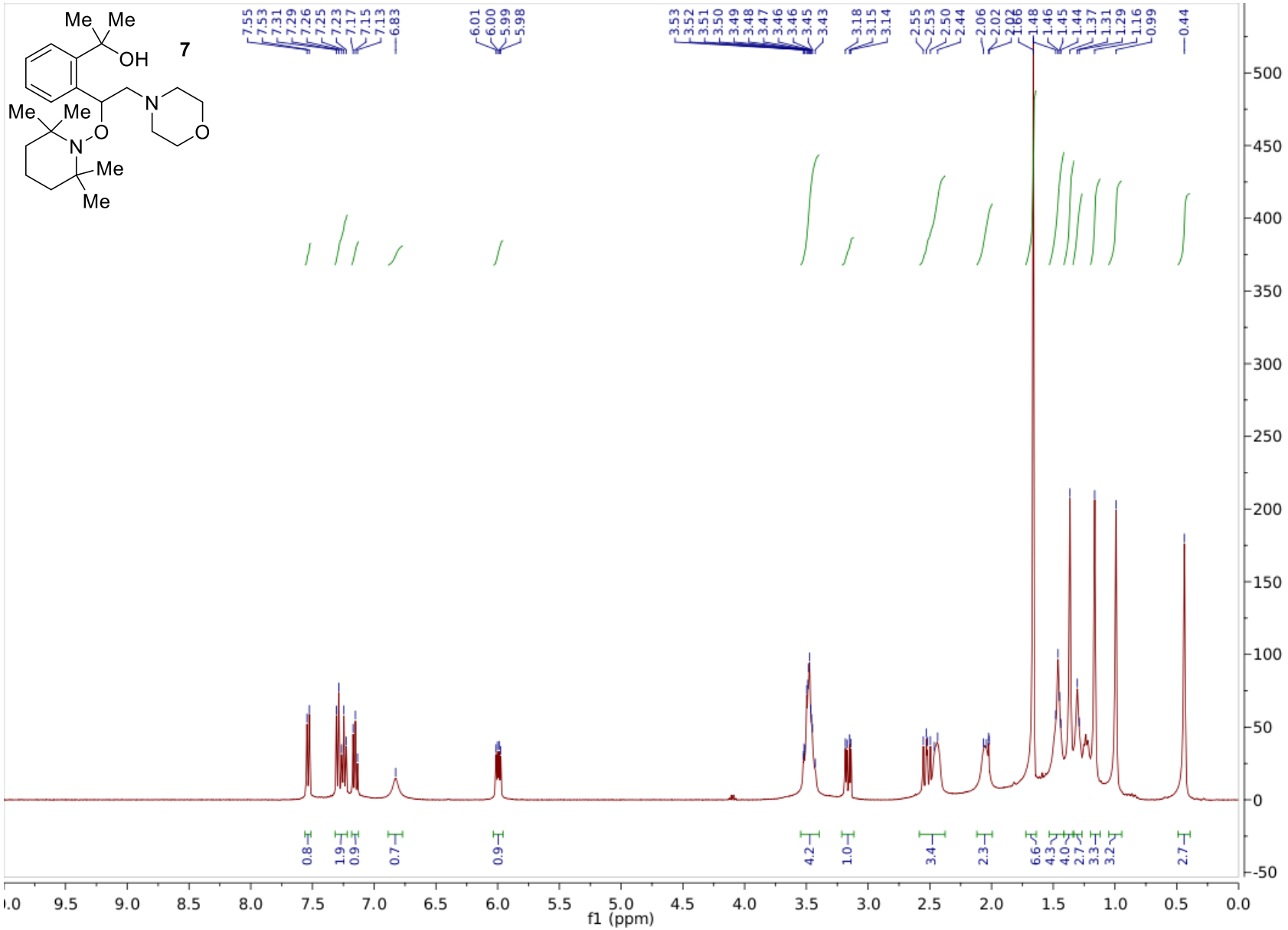

Me Me
OH
6
N
O
148.7
147.0
139.2
131.5
127.0
126.4
126.1
116.2
77.0
73.2
66.7
66.0
54.0
33.2
f1 (ppm)

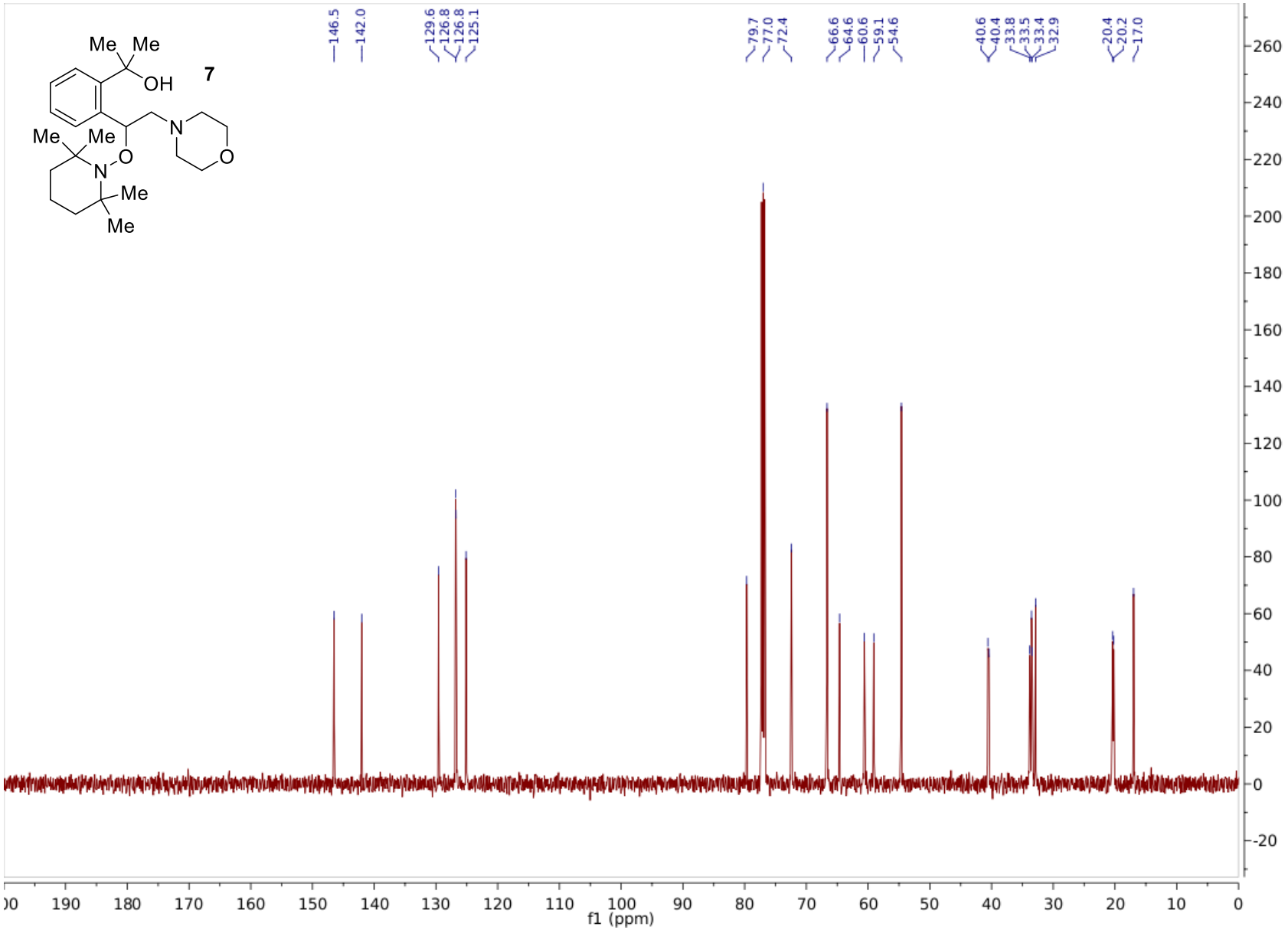

Me
Me
O
O
N
D3C
D3-3v
7.43
7.42
7.41
7.41
7.40
7.40
7.40
7.39
7.38
7.37
7.36
7.26
7.26
7.25
7.25
7.24
7.24
7.24
7.22
7.22
7.22
3.80
3.80
3.79
3.78
3.77
3.77
2.78
2.77
2.76
2.73
2.70
2.68
2.64
2.62
2.61
2.61
2.60
2.59
1.68
1.64
1.7
1.5
3.9
2.0
1.2
1.2
2.2
3.0
3.1
f1 (ppm)

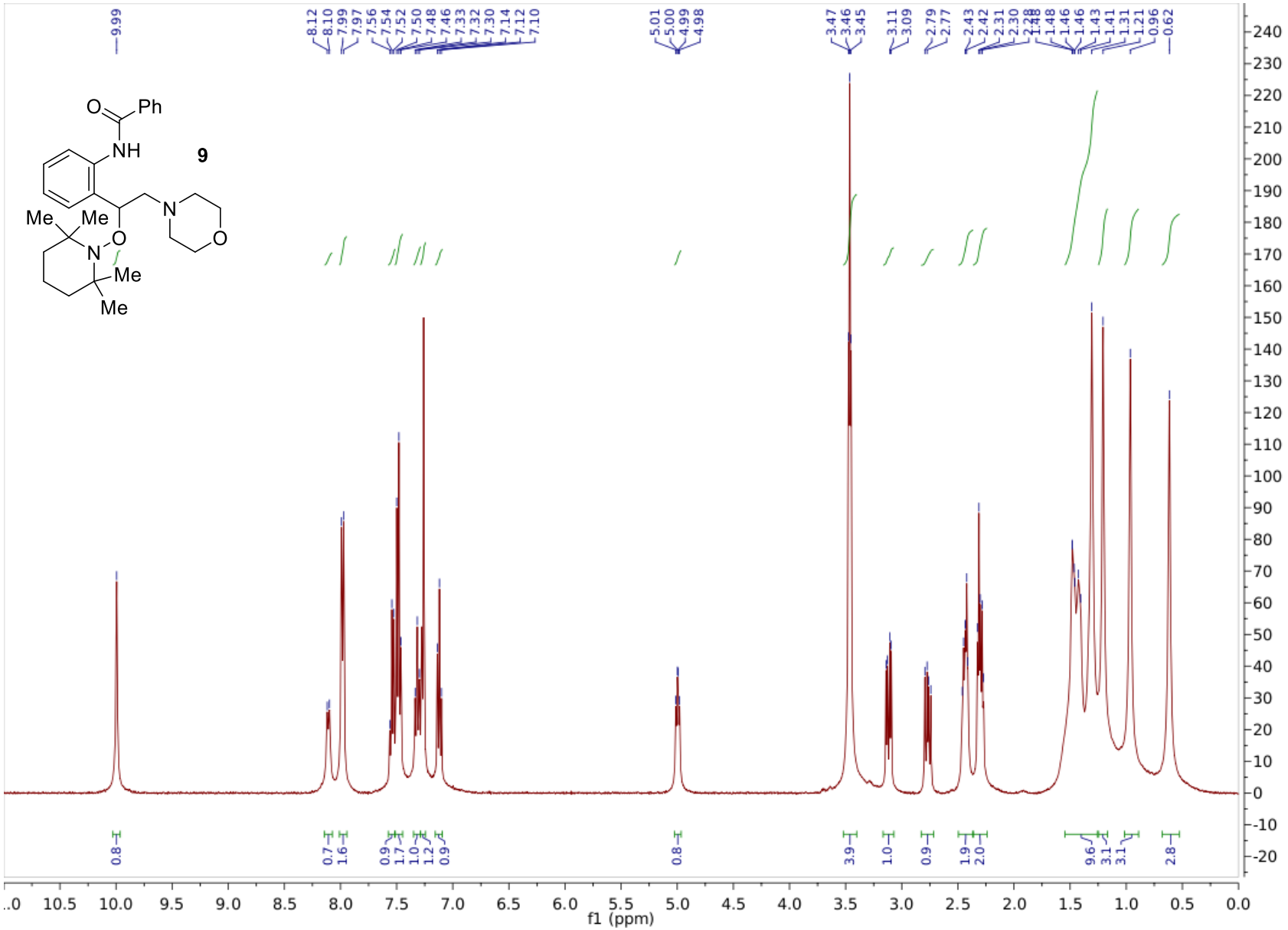

Me
Me
O
O
N
D3C
D3-3v
146.8
144.2
127.6
127.2
121.3
120.6
87.0
84.6
77.0
68.9
67.2
55.5
31.1
30.4
26.1
f1 (ppm)

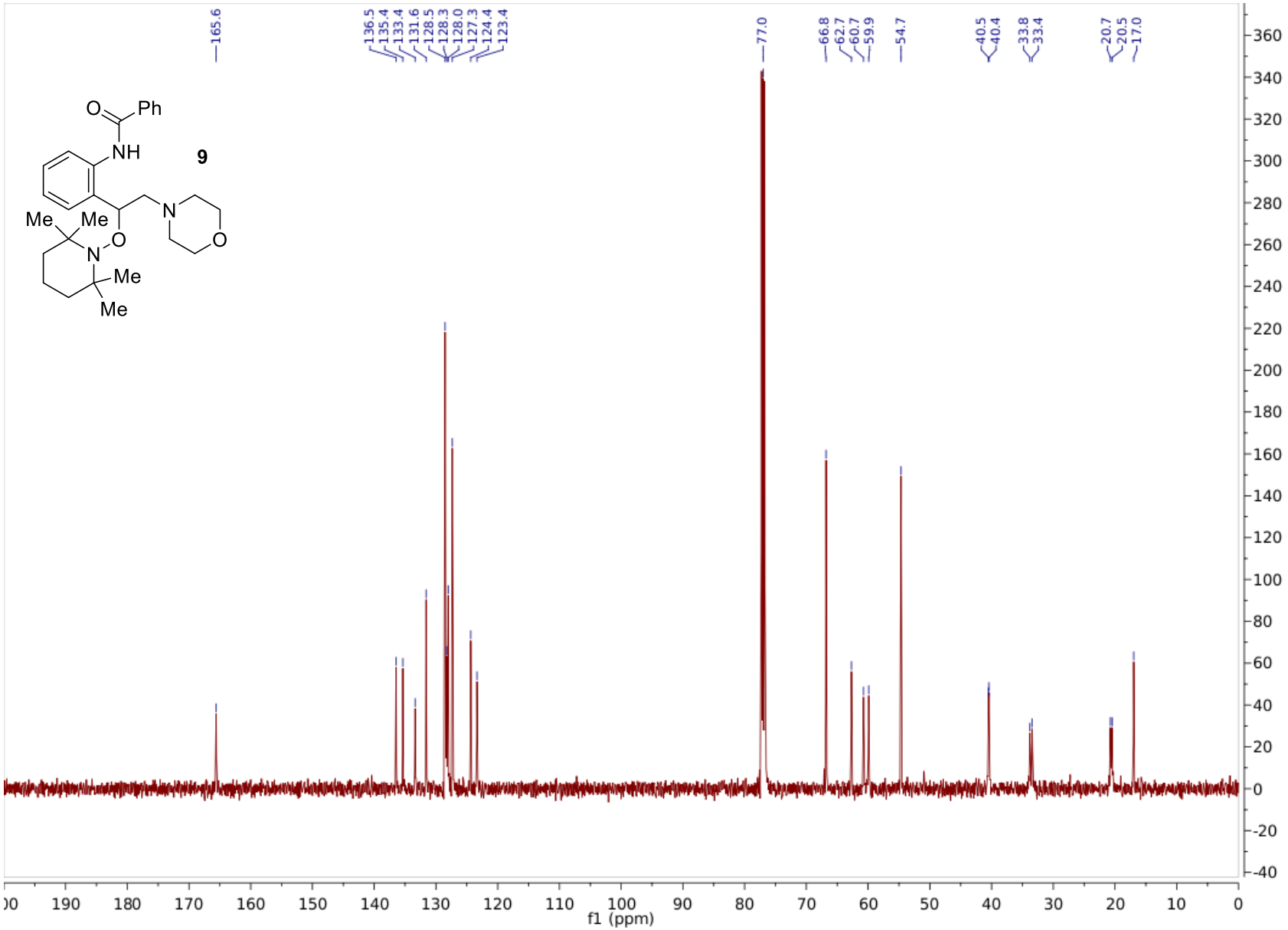

Me
Me
OH
D
D
N
O
D_2-3v
7.35
7.34
7.33
7.32
7.25
7.24
7.24
7.22
7.22
7.19
7.19
7.17
7.17
7.15
7.15
7.01
7.00
6.99
6.99
3.69
3.68
3.67
3.22
1.72
1.60
1.0
1.3
1.0
0.7
3.9
1.8
4.1
0.9
6.4
f1 (ppm)

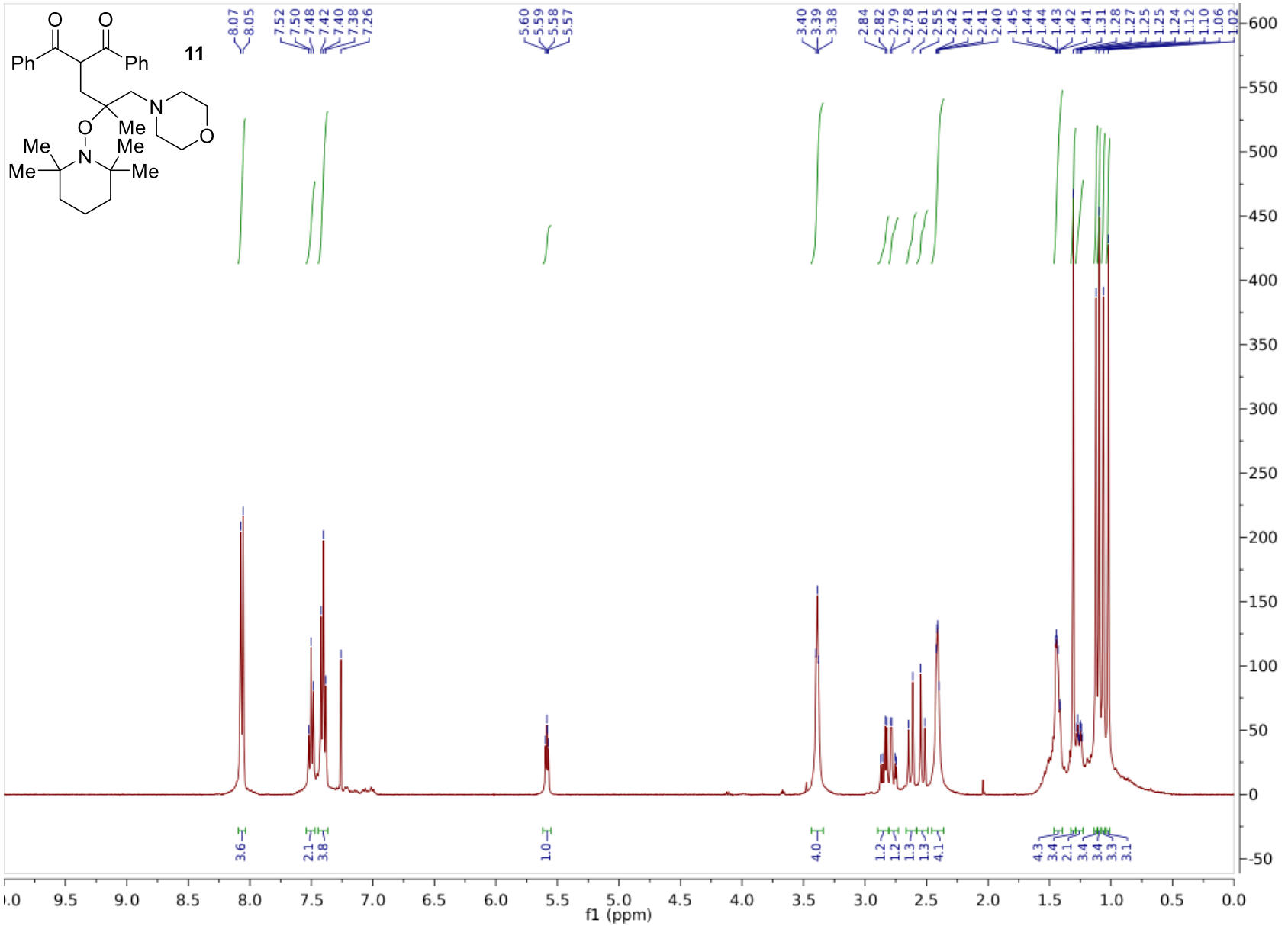

Me Me
OH
N
O
D D
D_2-6
148.5
147.0
139.2
131.6
127.1
126.5
126.1
77.0
73.2
66.7
66.0
54.0
33.2
f1 (ppm)

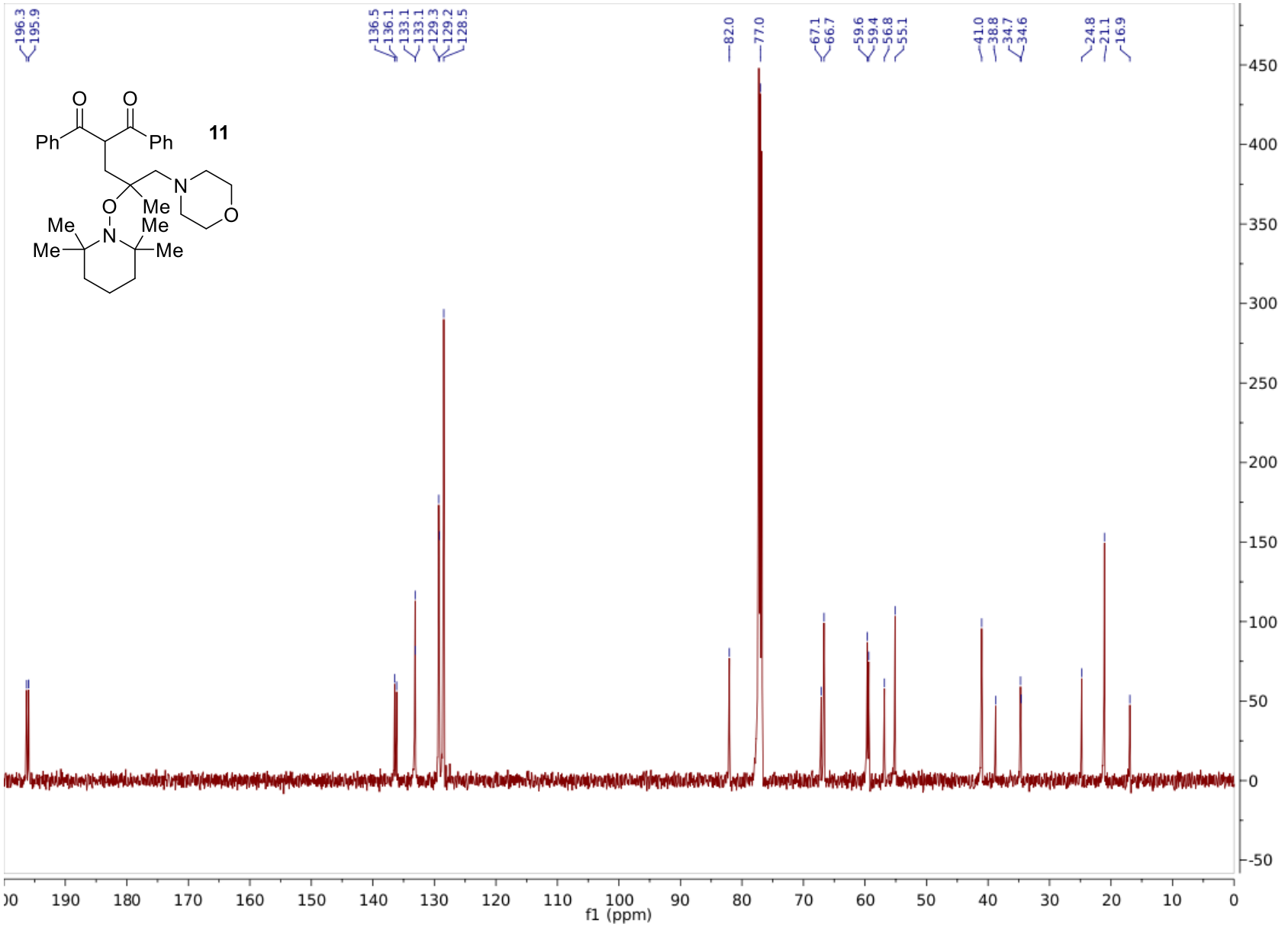

Me
Me
OH
7
Me
Me
N
O
N
O
Me
Me
7.55
7.53
7.31
7.29
7.26
7.25
7.23
7.17
7.15
7.13
6.83
6.01
6.00
5.99
5.98
3.53
3.52
3.51
3.50
3.49
3.48
3.47
3.46
3.46
3.45
3.43
3.18
3.15
3.14
2.55
2.53
2.50
2.44
2.06
2.02
2.02
1.66
1.48
1.46
1.45
1.44
1.37
1.31
1.29
1.16
0.99
0.44
0.8
1.9
0.9
0.7
0.9
4.2
1.0
3.4
2.3
6.6
4.3
4.0
2.7
3.3
3.2
2.7
f1 (ppm)

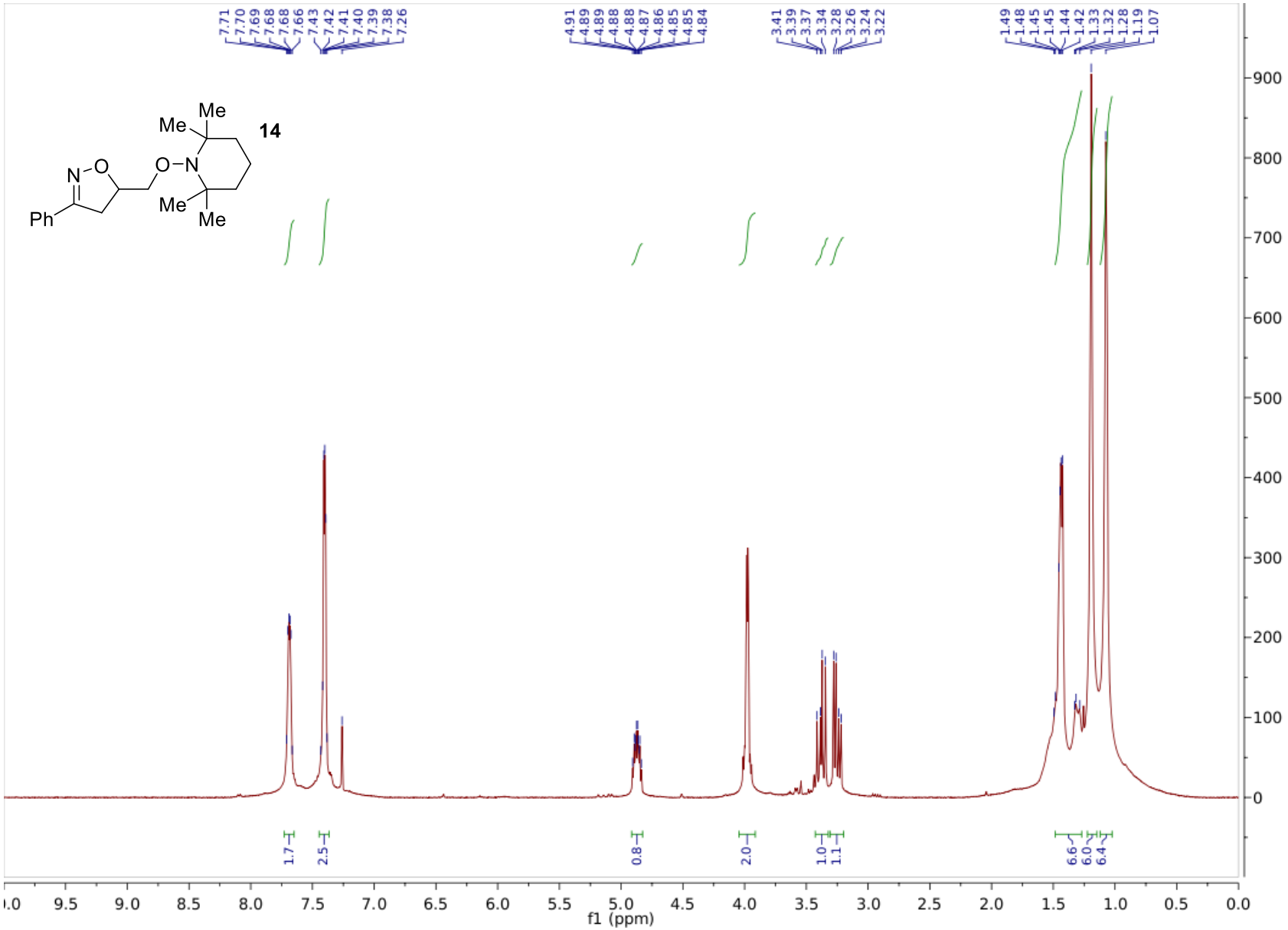

Me Me
OH
7
Me Me
N
O
N
O
Me
Me
146.5
142.0
129.6
126.8
126.8
125.1
79.7
77.0
72.4
66.6
64.6
60.6
59.1
54.6
40.6
40.4
33.8
33.5
33.4
32.9
20.4
20.2
17.0
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

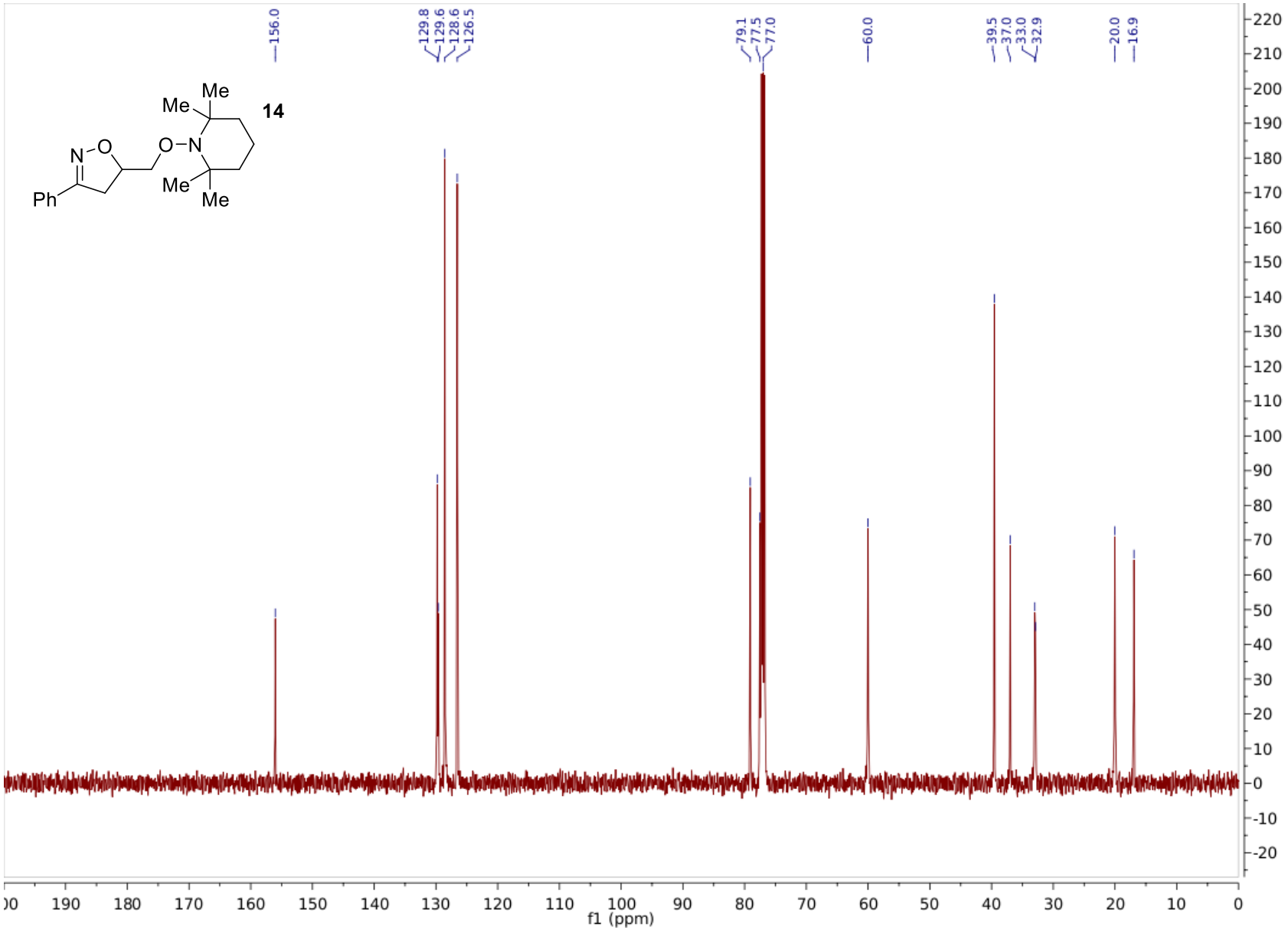

9.99
8.12
8.10
7.99
7.97
7.56
7.54
7.52
7.50
7.48
7.46
7.33
7.32
7.30
7.14
7.12
7.10
5.01
5.00
4.99
4.98
3.47
3.46
3.45
3.11
3.09
2.79
2.77
2.43
2.42
2.31
2.30
1.48
1.46
1.46
1.43
1.41
1.31
1.21
0.96
0.62
O
Ph
NH
9
Me
Me
N
O
N
O
Me
Me
f1 (ppm)

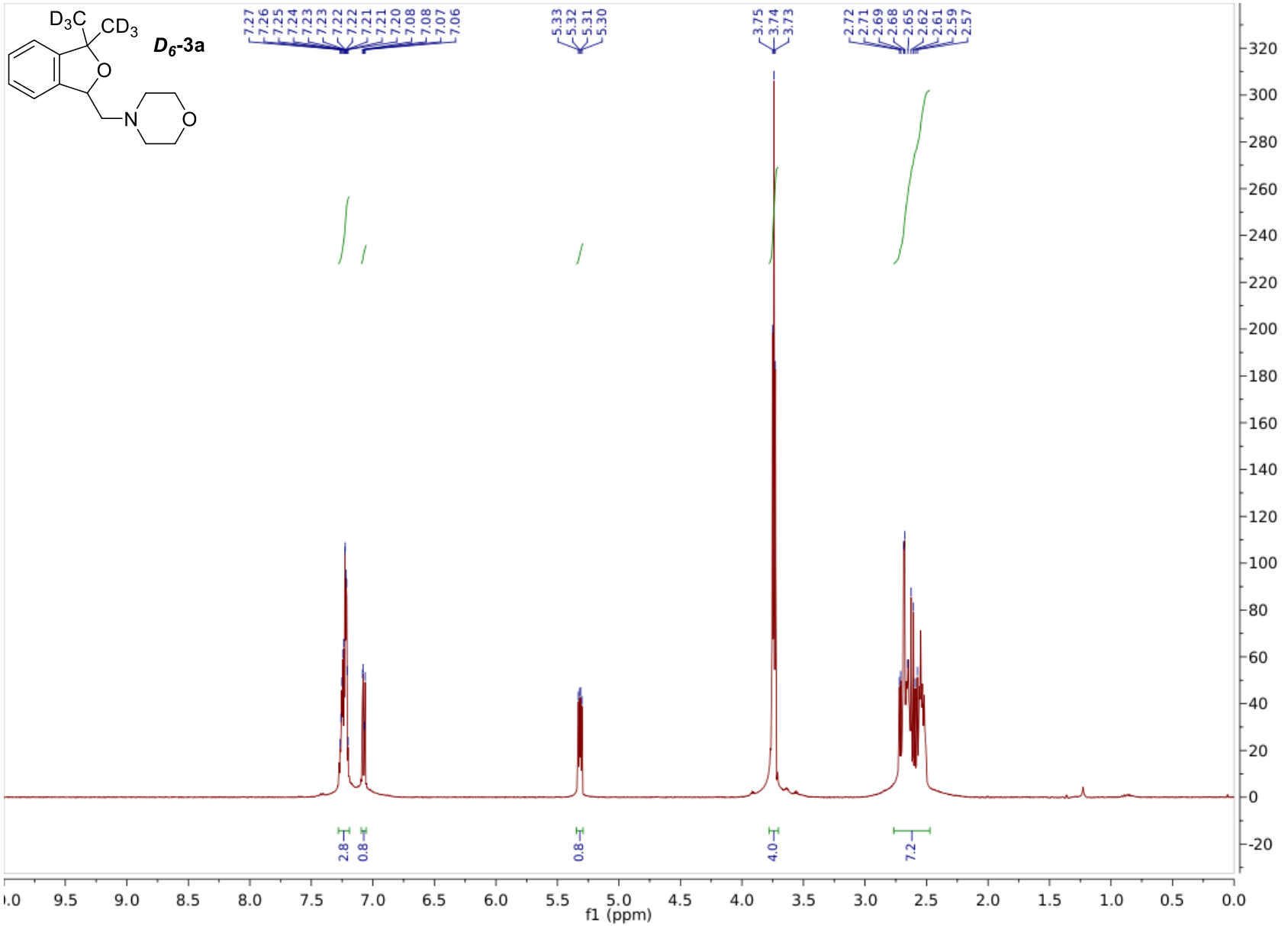

165.6
136.5
135.4
133.4
131.6
128.5
128.3
128.0
127.3
124.4
123.4
77.0
66.8
62.7
60.7
59.9
54.7
40.5
40.4
33.8
33.4
20.7
20.5
17.0
O
Ph
NH
9
Me
Me
O
N
N
O
Me
Me
f1 (ppm)

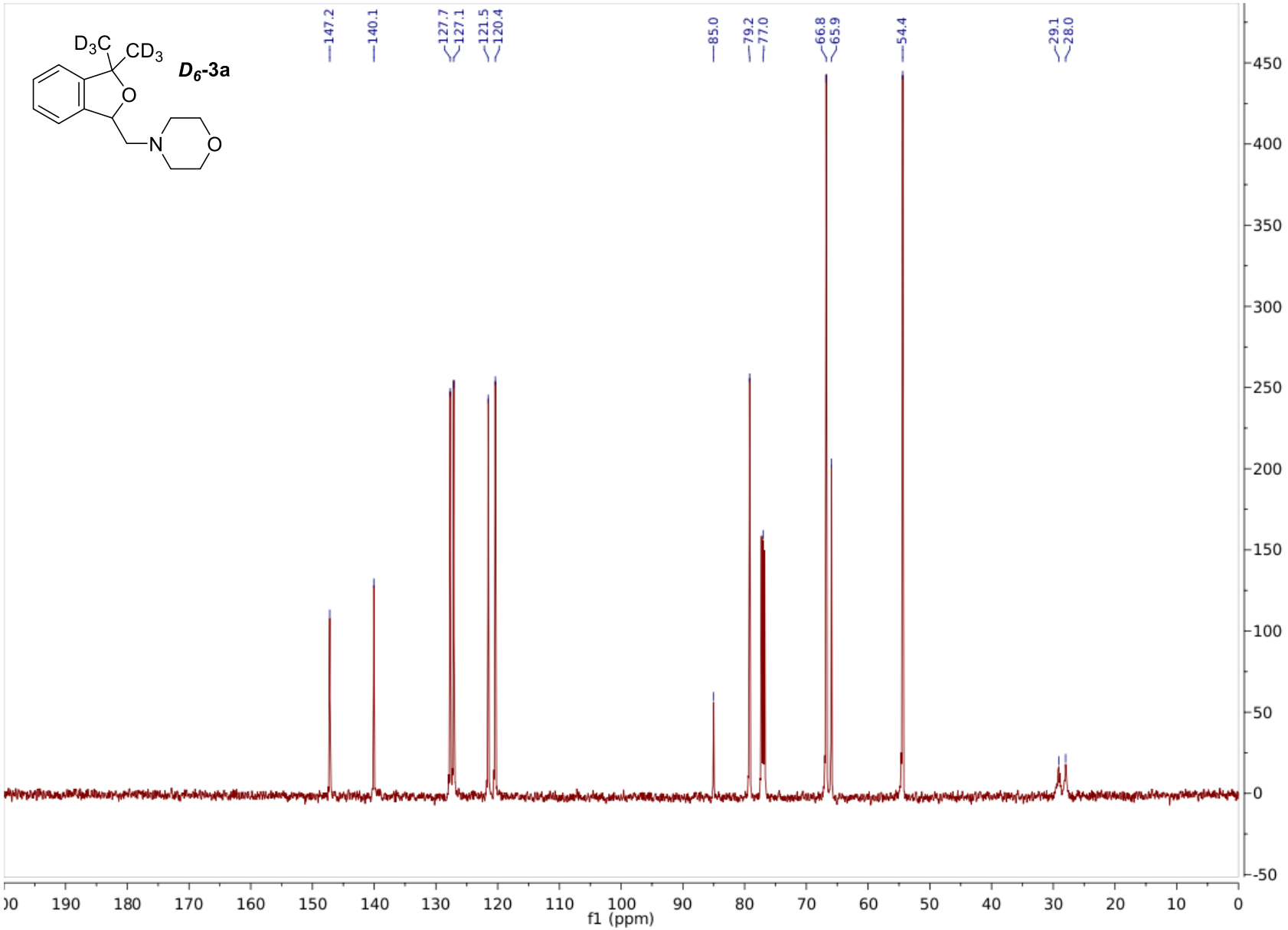

11
Ph
Ph
O
O
N
O
Me
Me
Me
Me
Me
8.07
8.05
7.52
7.50
7.48
7.42
7.40
7.38
7.26
5.60
5.59
5.58
5.57
3.40
3.39
3.38
2.84
2.82
2.79
2.78
2.61
2.55
2.42
2.41
2.41
2.40
1.45
1.44
1.44
1.43
1.42
1.41
1.31
1.28
1.27
1.25
1.25
1.24
1.12
1.10
1.06
1.02
3.6
2.1
3.8
1.0
4.0
1.2
1.2
1.3
1.3
4.1
4.3
3.4
2.1
3.4
3.3
3.1
f1 (ppm)

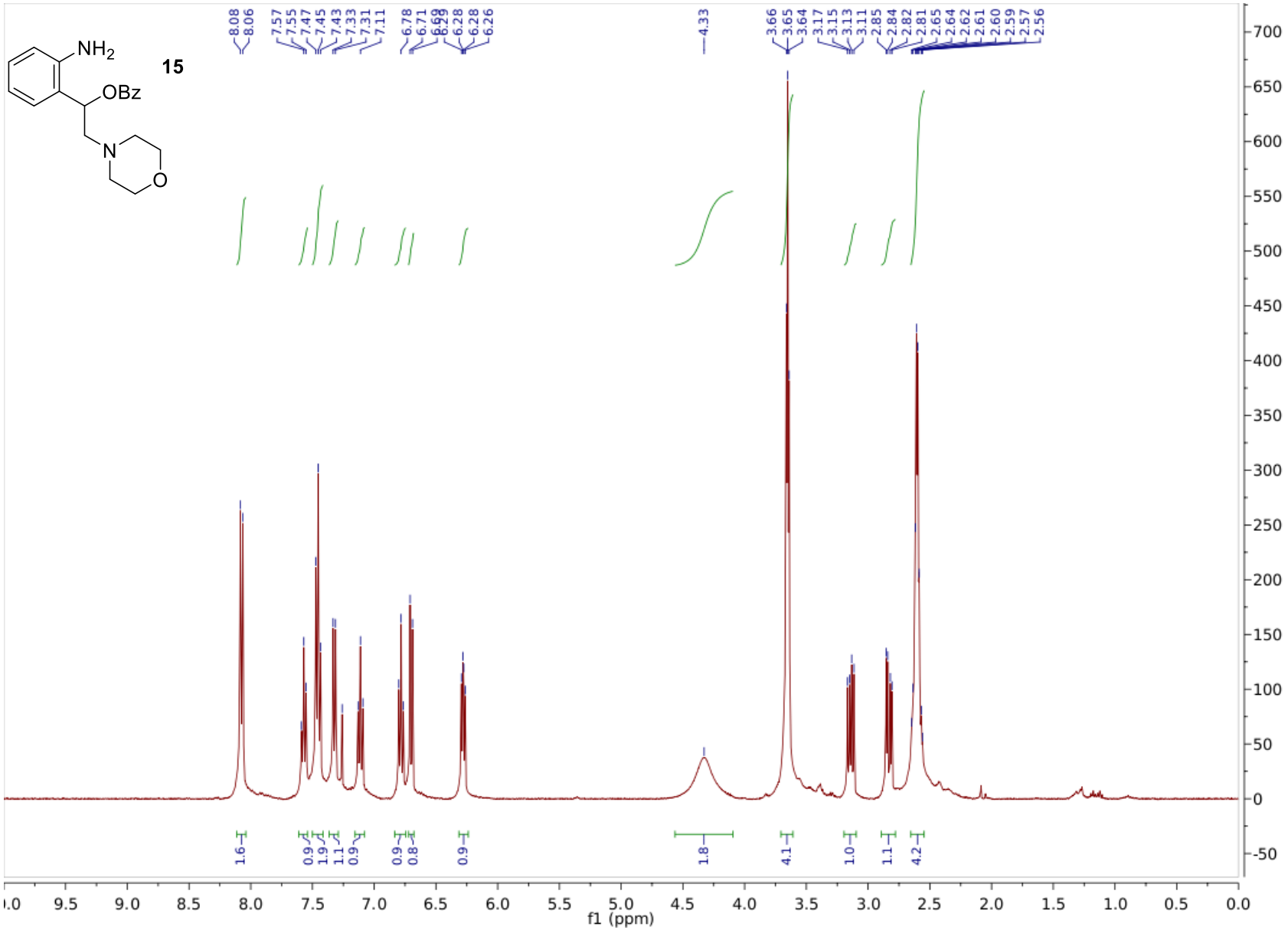

196.3
195.9
136.5
136.1
133.1
133.1
129.3
129.2
128.5
82.0
77.0
67.1
66.7
59.6
59.4
56.8
55.1
41.0
38.8
34.7
34.6
24.8
21.1
16.9
11
Ph
Ph
O
O
N
O
Me
Me
Me
Me
Me
N
O
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

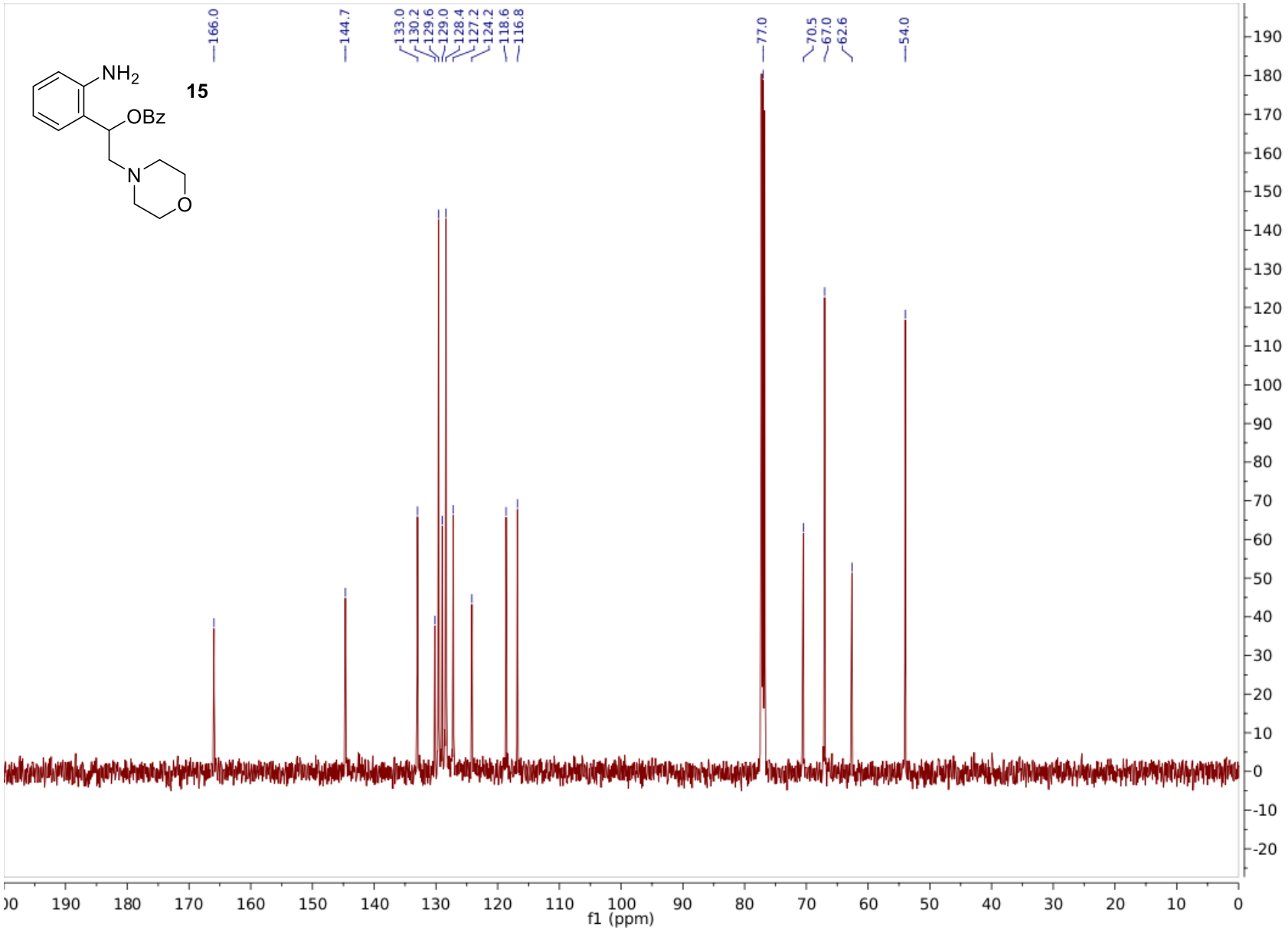

14
Me
Me
Me
Me
N
O
O
N
Ph
7.71
7.70
7.69
7.68
7.68
7.66
7.43
7.42
7.41
7.40
7.39
7.38
7.26
4.91
4.89
4.89
4.88
4.88
4.87
4.86
4.85
4.85
4.84
3.41
3.39
3.37
3.34
3.28
3.26
3.24
3.22
1.49
1.48
1.45
1.45
1.44
1.42
1.33
1.32
1.28
1.19
1.07
1.7
2.5
0.8
2.0
1.0
1.1
6.6
6.0
6.4
f1 (ppm)

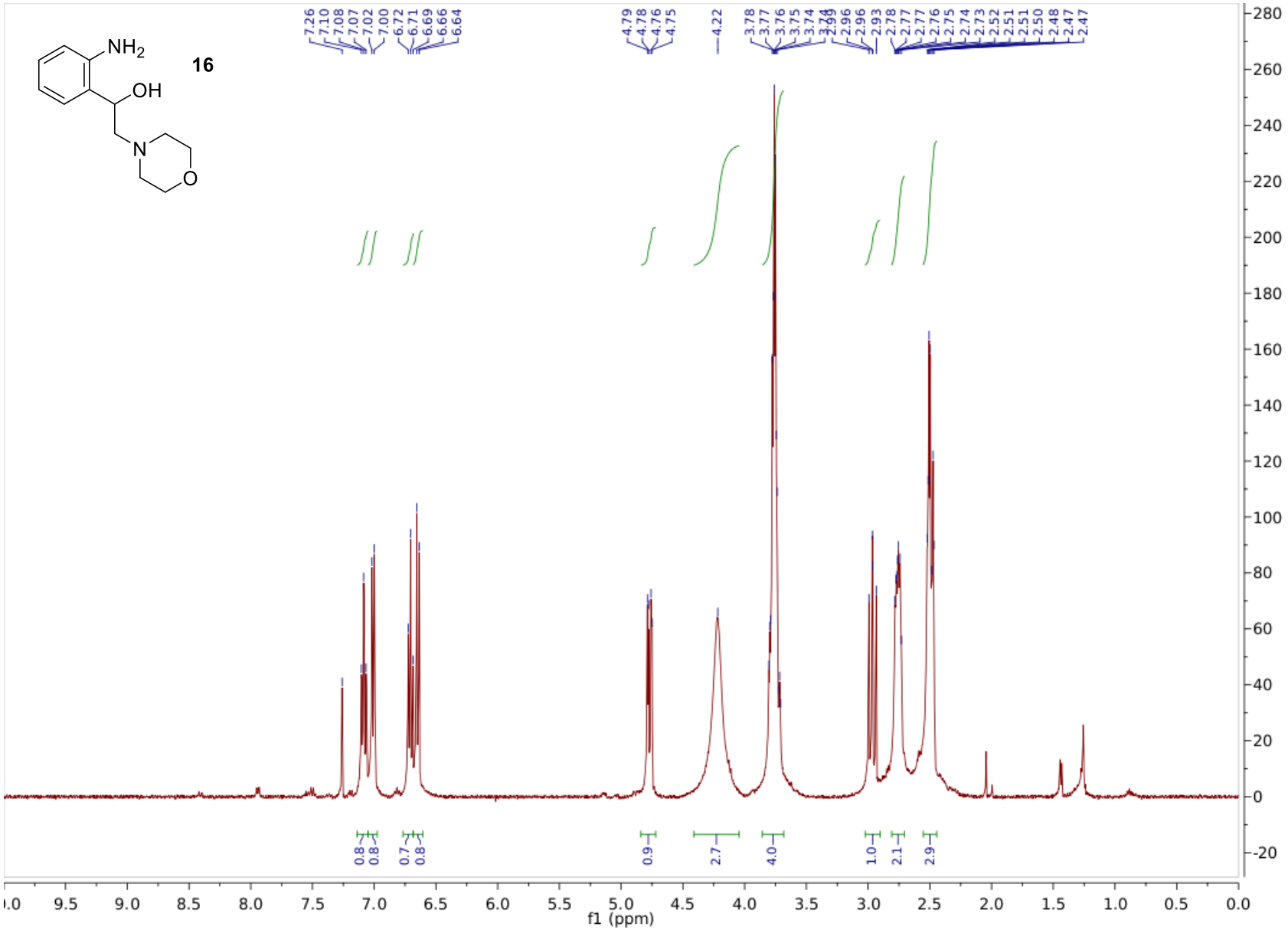

156.0
129.8
129.6
128.6
126.5
79.1
77.5
77.0
60.0
39.5
37.0
33.0
32.9
20.0
16.9
14
Me
Me
Me
Me
N
O
O
N
Ph
f1 (ppm)

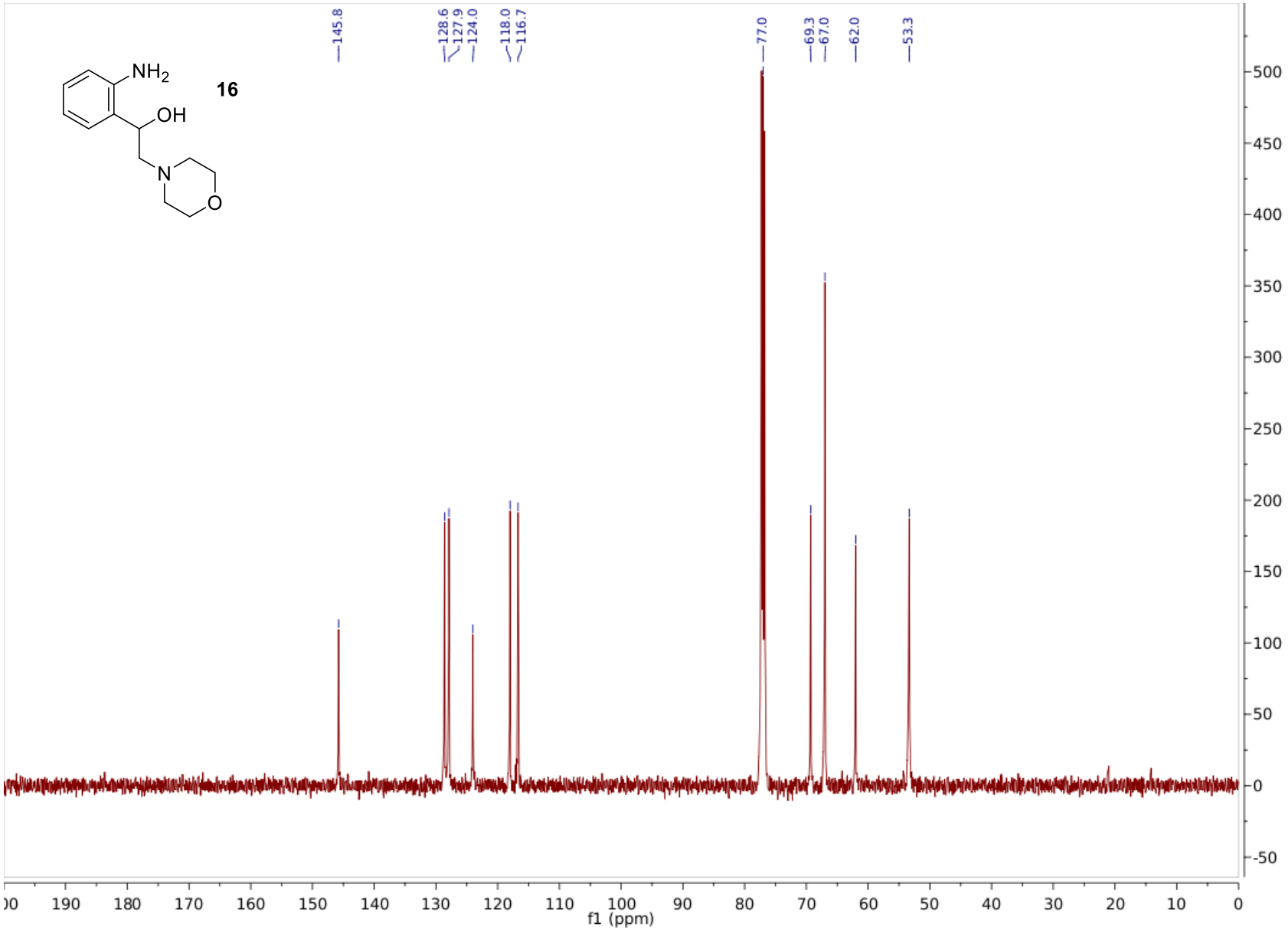

D3C
CD3
O
N
O
D6-3a
7.27
7.26
7.25
7.24
7.23
7.23
7.22
7.22
7.21
7.21
7.20
7.08
7.08
7.07
7.06
5.33
5.32
5.31
5.30
3.75
3.74
3.73
2.72
2.71
2.69
2.68
2.65
2.62
2.61
2.59
2.57
2.8
0.8
0.8
4.0
7.2
f1 (ppm)


D3C
CD3
O
N
O
D6-3a
147.2
140.1
127.7
127.1
121.5
120.4
85.0
79.2
77.0
66.8
65.9
54.4
29.1
28.0
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


NH2
OBz
N
O
15
8.08
8.06
7.57
7.55
7.47
7.45
7.43
7.33
7.31
7.11
6.78
6.71
6.28
6.26
4.33
3.66
3.65
3.64
3.17
3.15
3.13
3.11
2.85
2.84
2.82
2.81
2.65
2.64
2.62
2.61
2.60
2.59
2.57
2.56
1.6
0.9
1.9
1.1
0.9
0.9
0.8
0.9
1.8
4.1
1.0
1.1
4.2
f1 (ppm)


166.0
144.7
133.0
130.2
129.6
129.0
128.4
127.2
124.2
118.6
116.8
77.0
70.5
67.0
62.6
54.0
NH2
OBz
N
O
15
f1 (ppm)


NH2
OH
N
O
16
f1 (ppm)


16
NH2
OH
N
O
145.8
128.6
127.9
124.0
118.0
116.7
77.0
69.3
67.0
62.0
53.3
f1 (ppm)