

Tropylum-Promoted Oxidative Functionalization of Tetrahydroisoquinolines

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Table of Contents

Numbering of All THIQ Substrates Used in this Study.....	S2
Table S1. Optimization of the Iminium Formation Reaction with Tropylum Salt ^a	S3
¹ H NMR Studies of Iminium Formation Step for Substrates in Table 1	S4
Confirmation of 6aa-6ac Structures by HMBC NMR	S5
NMR Spectra	S99

Numbering of All THIQ Substrates Used in this Study

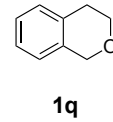
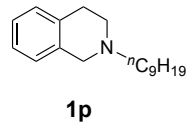
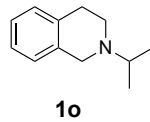
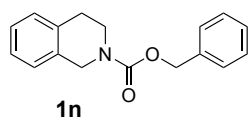
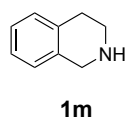
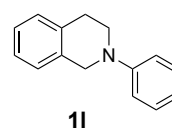
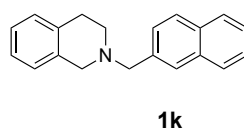
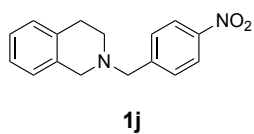
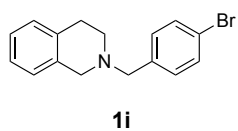
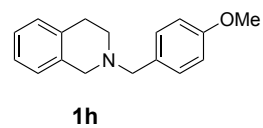
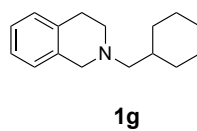
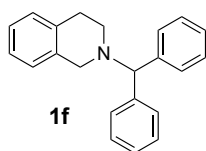
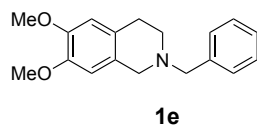
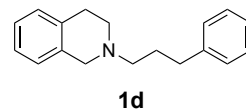
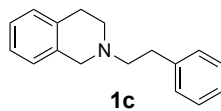
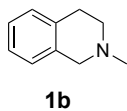
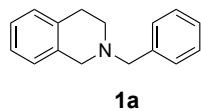
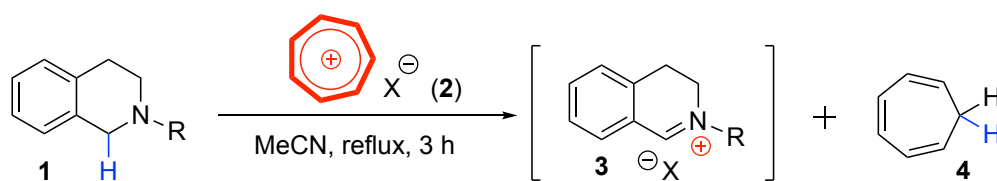


Table S1. Optimization of the Iminium Formation Reaction with Tropylium Salt^a

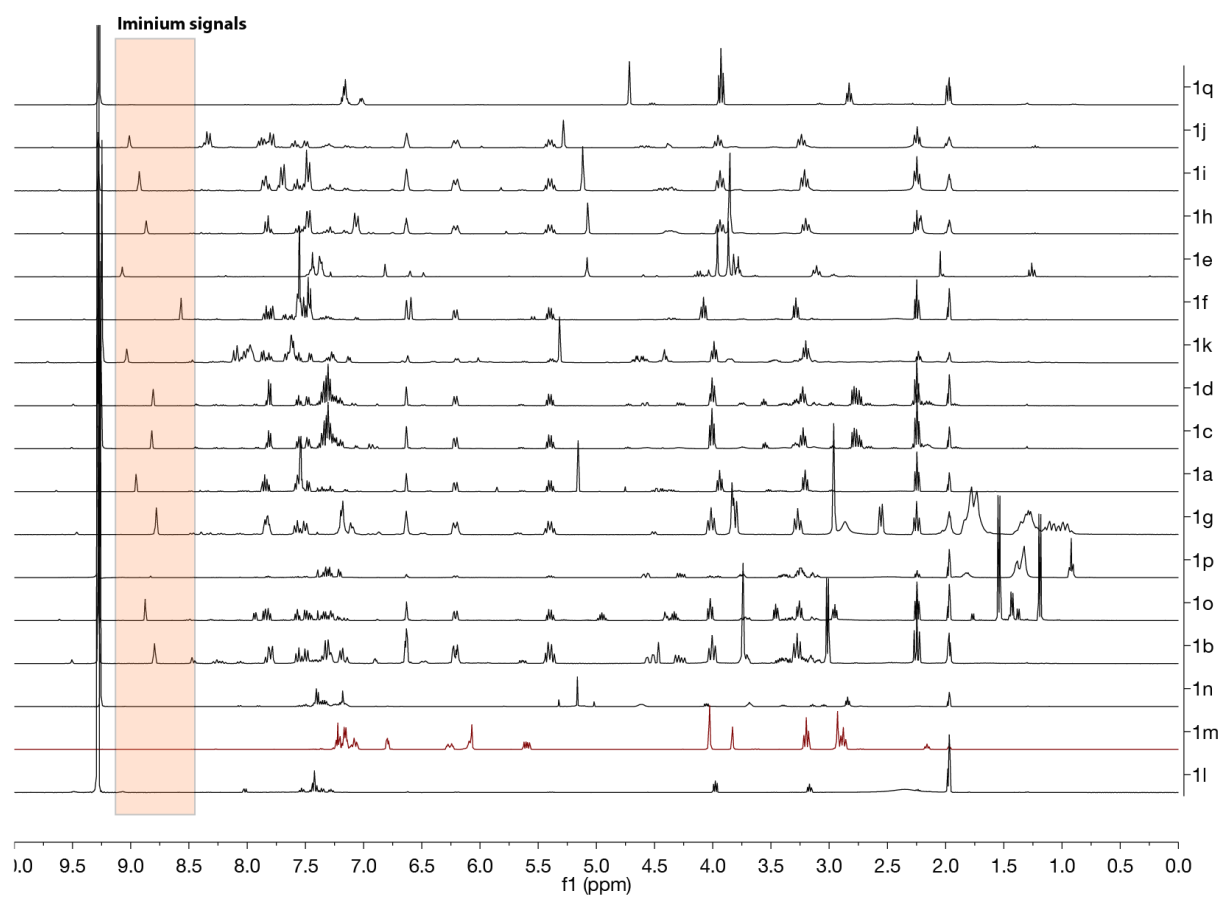


entry ^a	R	X	conversion ^b (%)
1	Ph (1l)	BF ₄	< 1
2	H (1m)	BF ₄	n.r.
3	Cbz (1n)	BF ₄	n.r.
4	Me (1b)	BF ₄	~90
5	ⁱ Pr (1o)	BF ₄	< 10
6	ⁿ C ₉ H ₁₉ (1p)	BF ₄	< 5
7	-CH ₂ Cy (1g)	BF ₄	100
8 ^c	-CH ₂ Ph (Bn, 1a)	BF ₄	100
9	-CH ₂ CH ₂ Ph (1c)	BF ₄	100
10	-CH ₂ CH ₂ CH ₂ Ph (1d)	BF ₄	100
11	-CH ₂ Naphthyl (1k)	BF ₄	100
12	-CHPh ₂ (1f)	BF ₄	100
13	Bn (1a)	Br	~45
14	Bn (1a)	BPh ₄	100
15 ^d	Bn (1a)	Ph ₃ C ⁺ BF ₄ ⁻	~70
16 ^d	Bn (1a)	NO ⁺ BF ₄ ⁻	~80
17 ^e	Bn on 6,7-(OMe) ₂ THIQ (1e)	BF ₄	100
18 ^f	Isochroman (1q)	BF ₄	n.r.

^a Reaction conditions: THIQ precursor (0.5 mmol) and tropylium salt (0.65 mmol) in acetonitrile at reflux for 3 h (optimized conditions for entry 8). ^b Conversion of **1** by ¹H NMR.

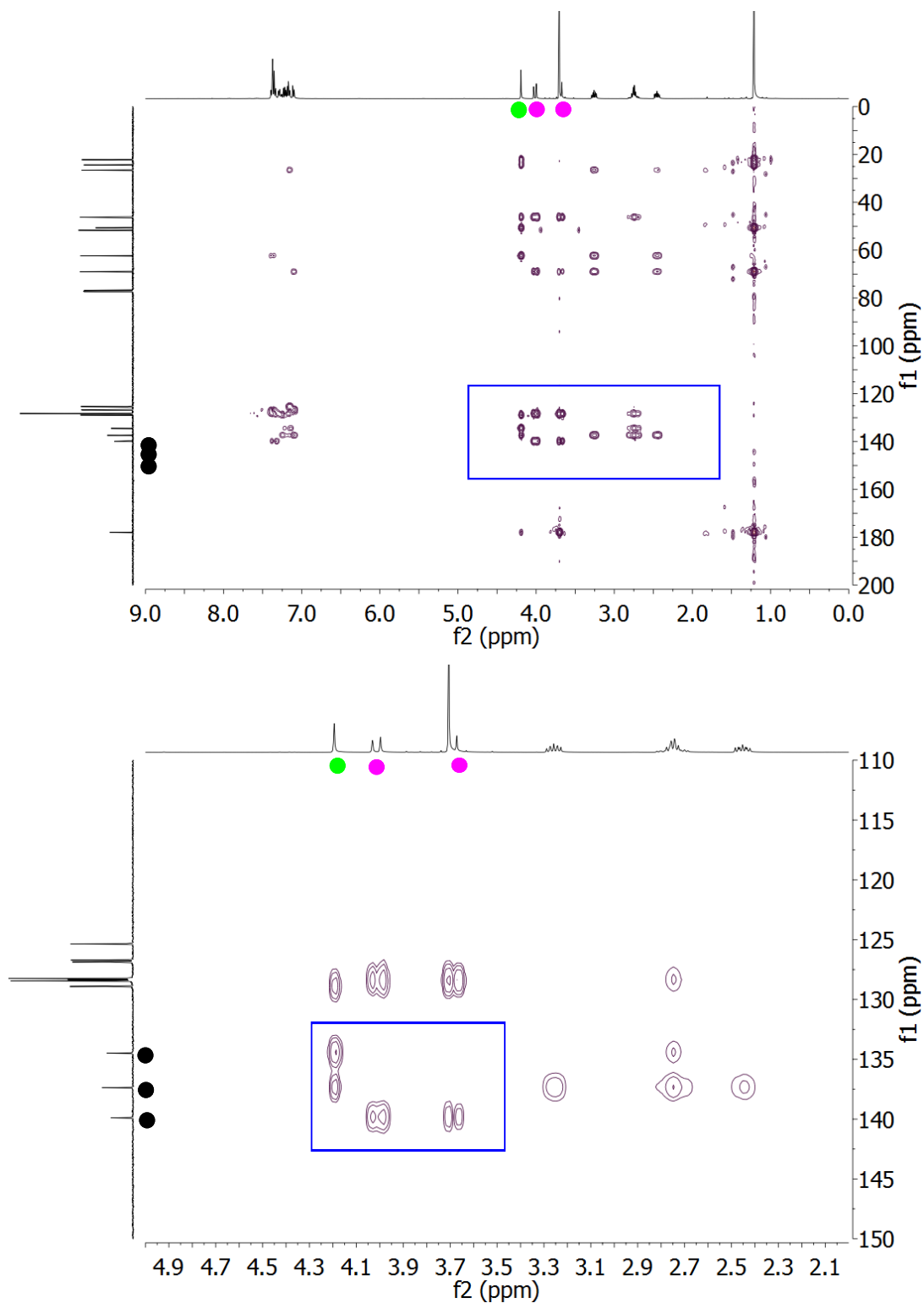
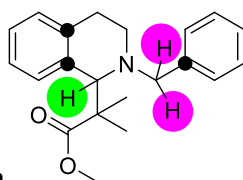
^c Solvent, temperature and reaction time were optimized for this substrate. ^d Tritylium or nitrosonium tetrafluoroborate were used as the oxidant instead of tropylium salt. ^e *N*-Benzyl-6,7-dimethoxy THIQ was used as substrate. ^f Isochroman was used as substrate.

¹H NMR Studies of Iminium Formation Step for Substrates in Table 1

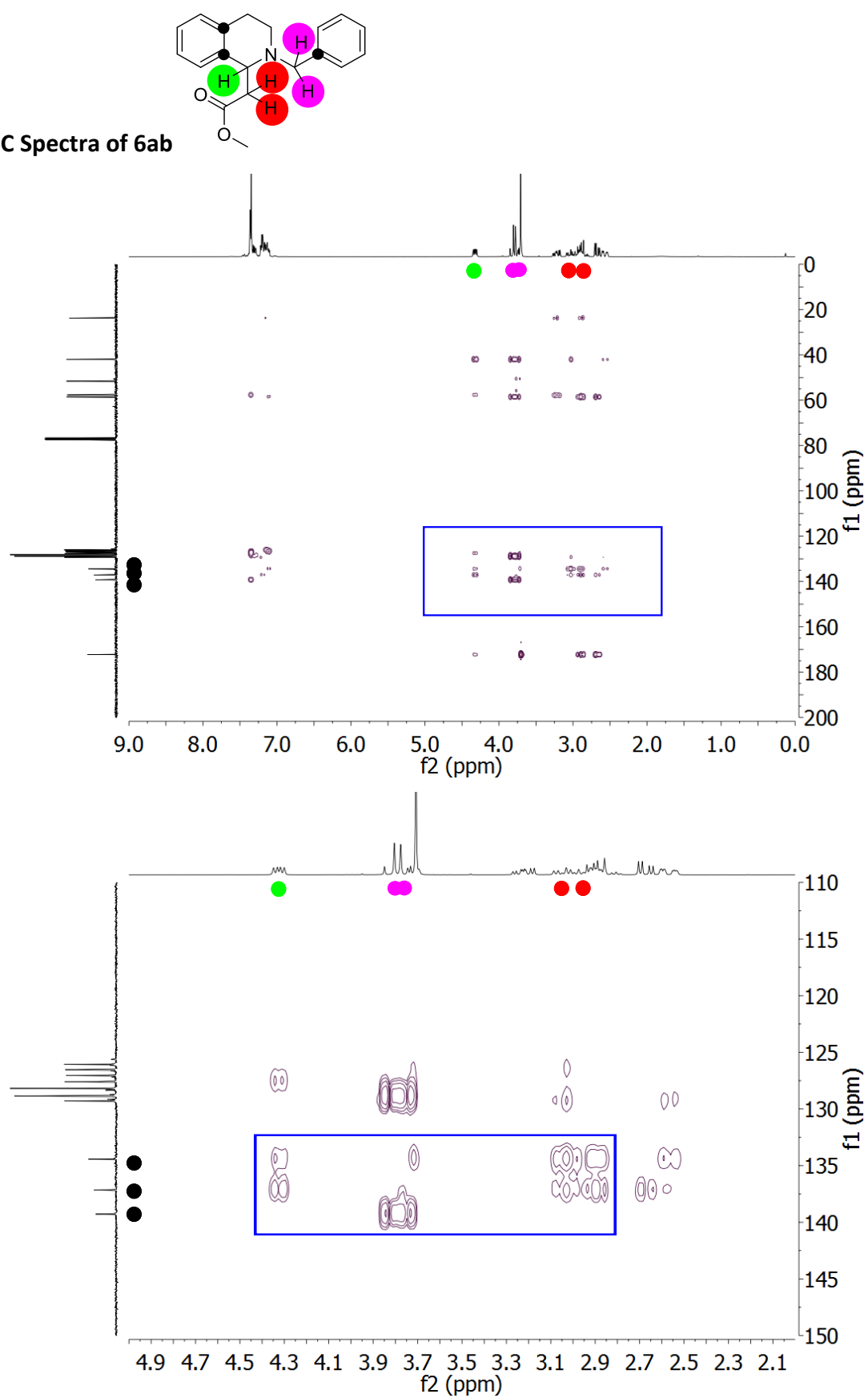


Confirmation of 6aa-6ac Structures by HMBC NMR

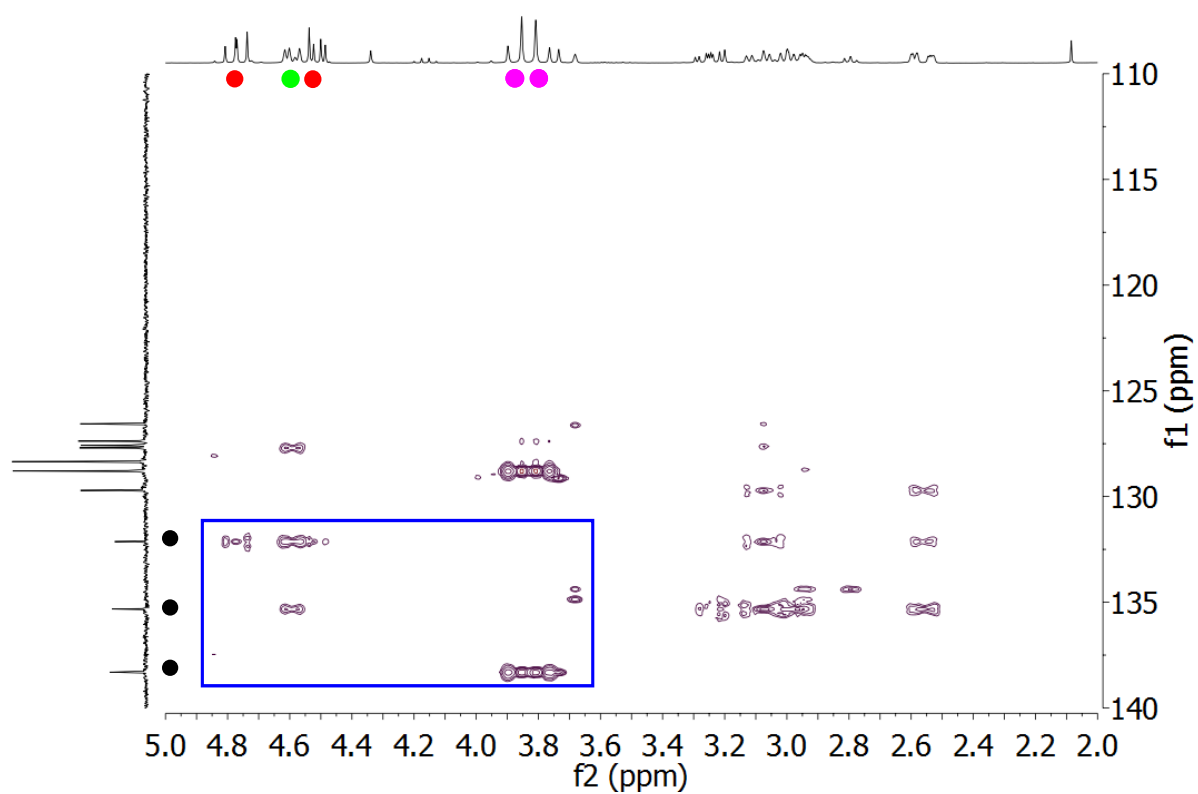
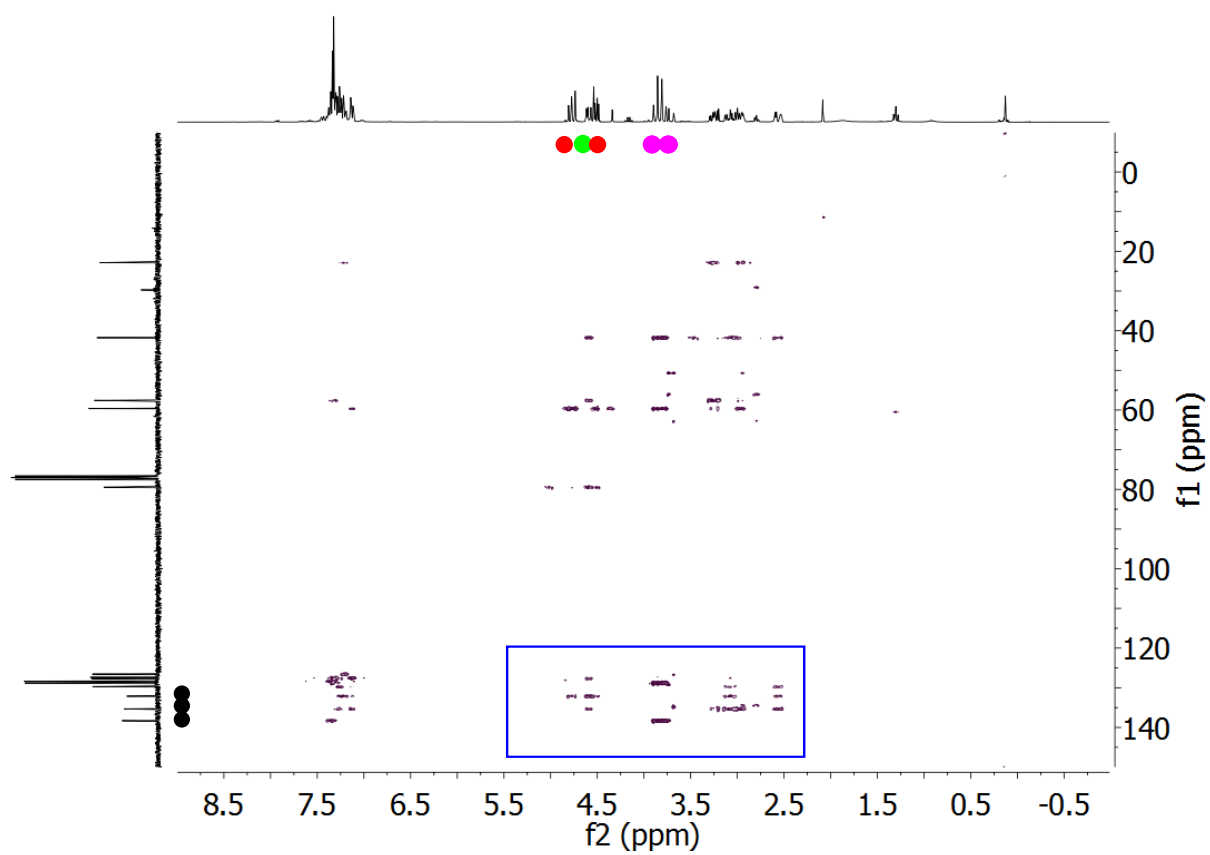
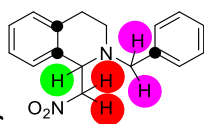
HMBC Spectra of 6aa



HMBC Spectra of 6ab

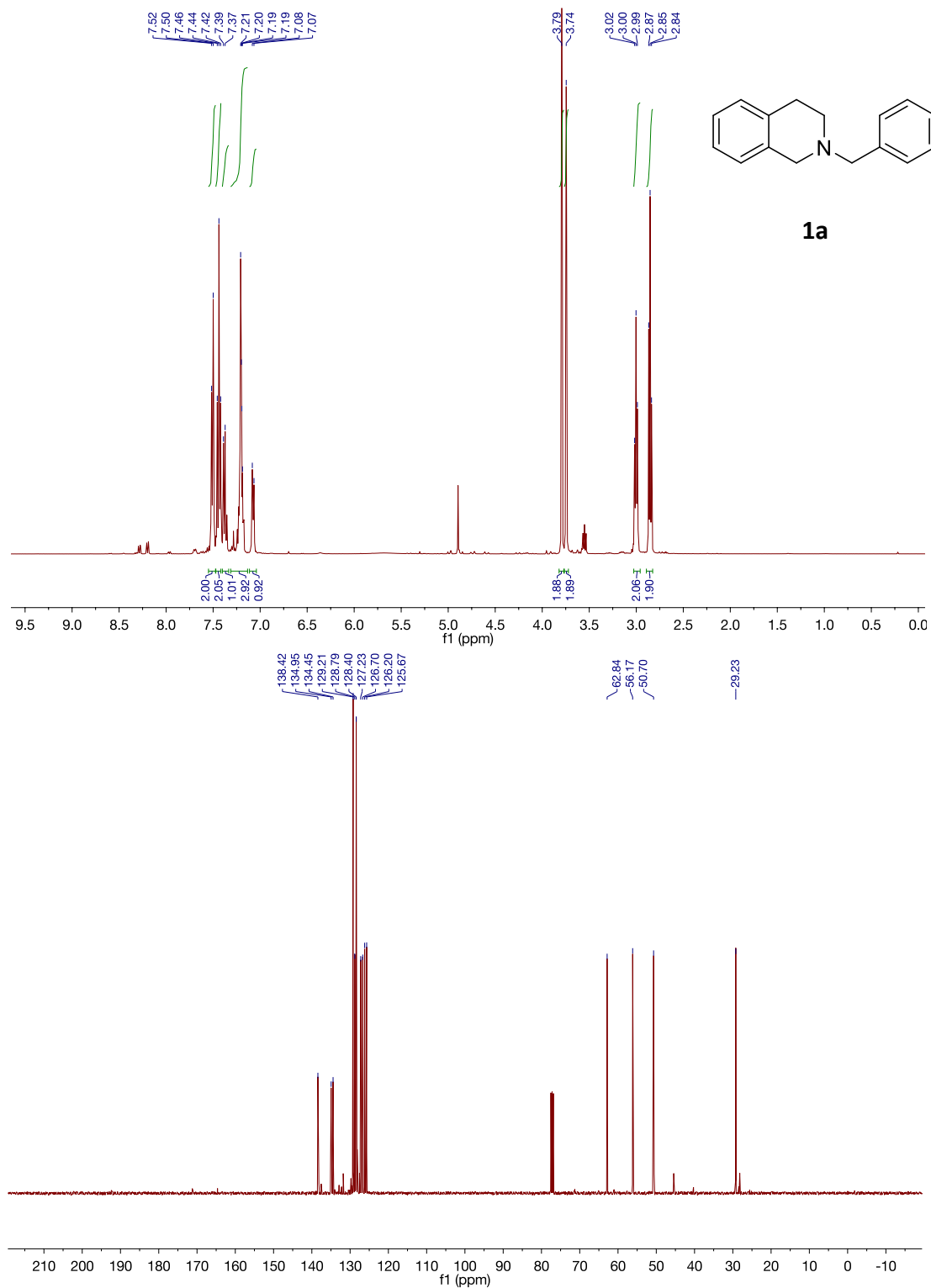


HMBC Spectra of 6ac

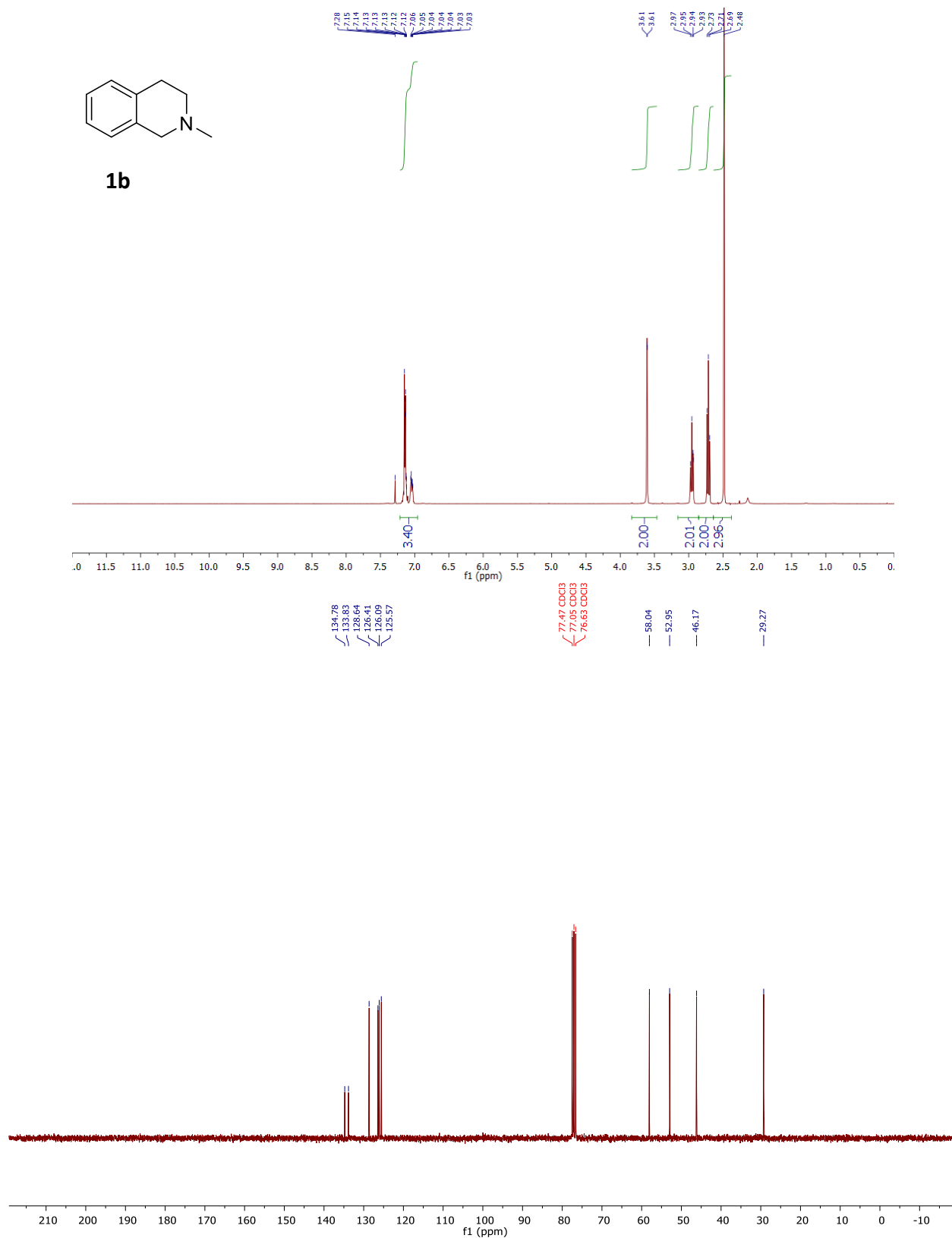


NMR Spectra

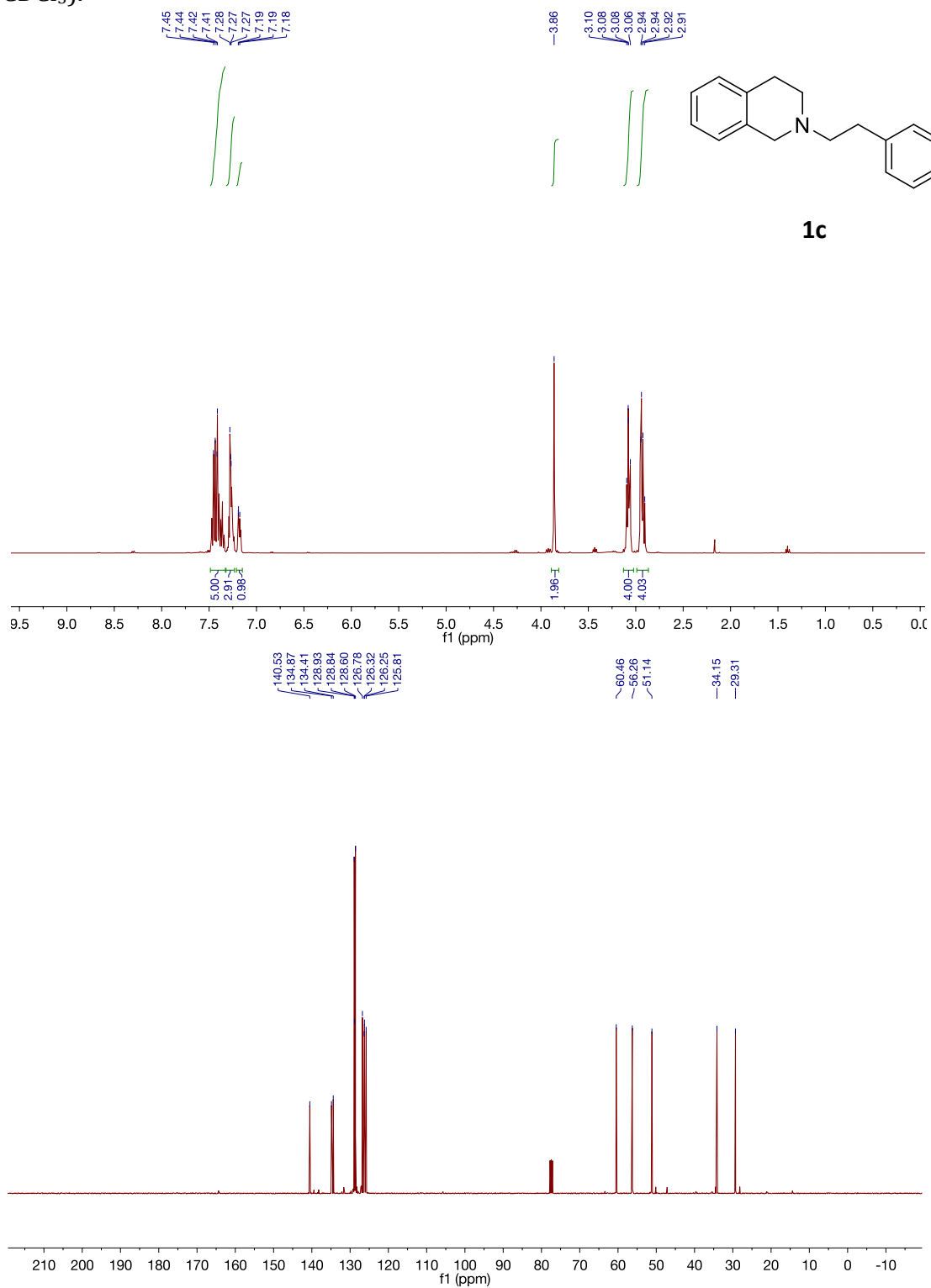
2-benzyl-1,2,3,4-tetrahydroisoquinoline (**1a**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



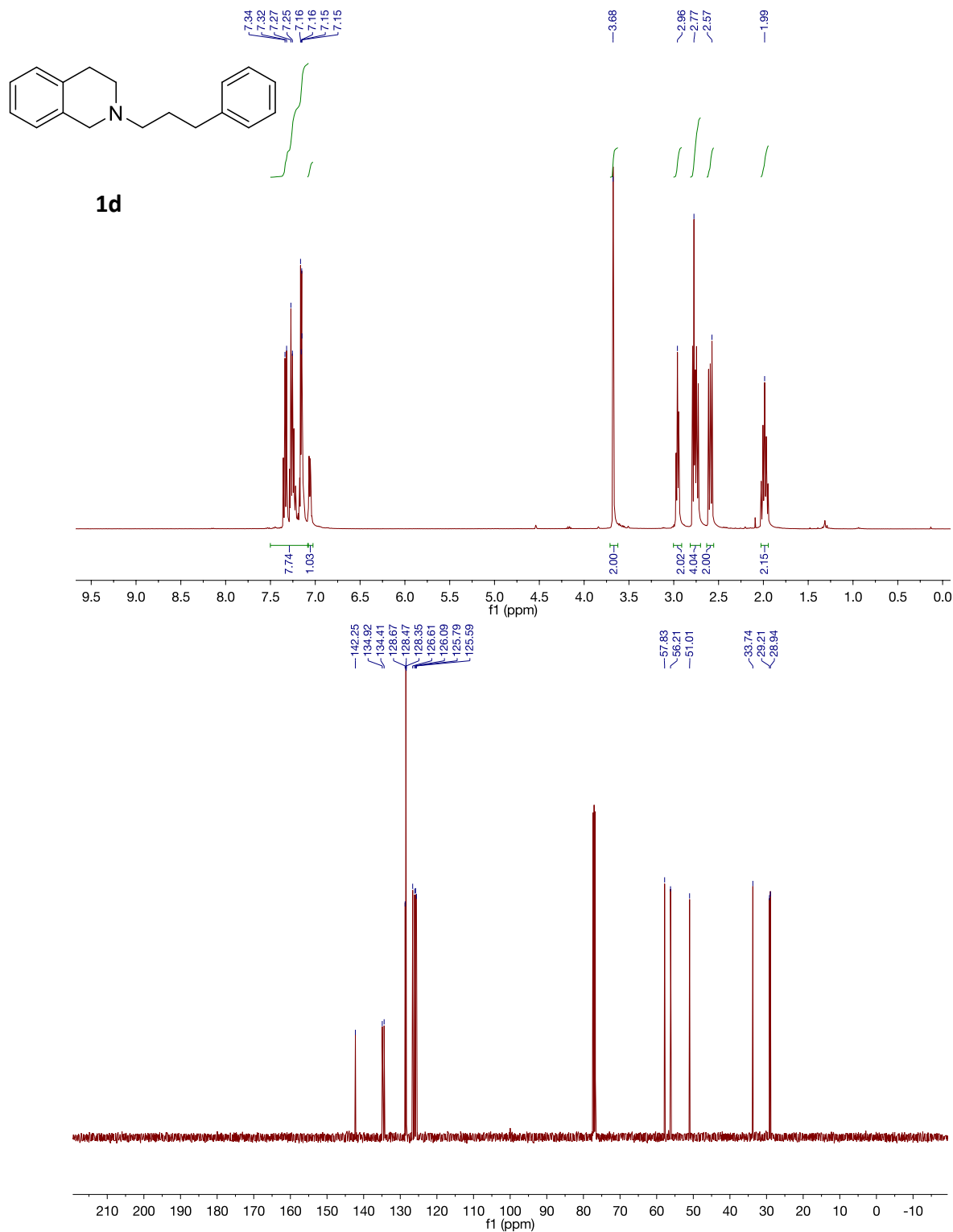
2-methyl-1,2,3,4-tetrahydroisoquinoline (**1b**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



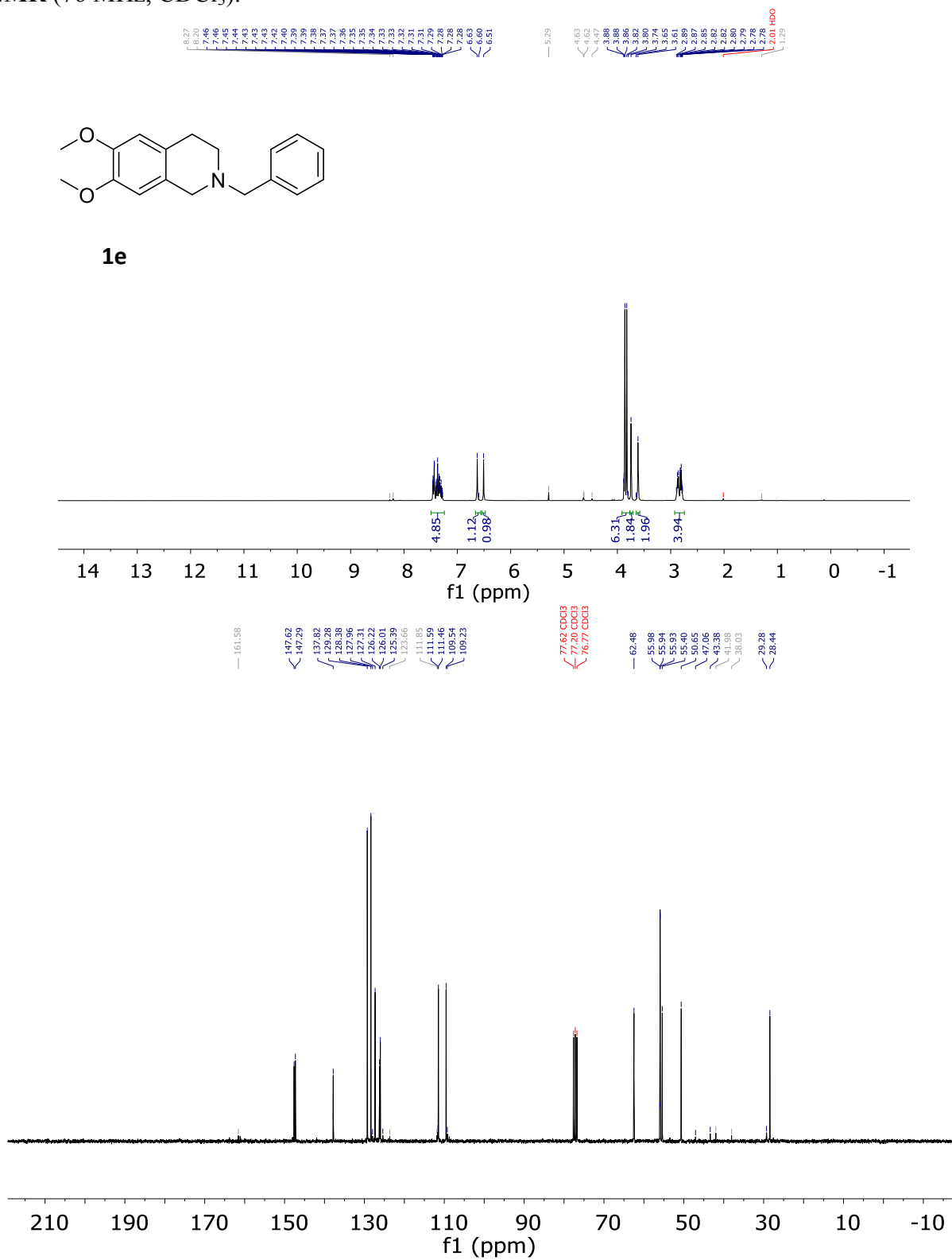
2-phenethyl-1,2,3,4-tetrahydroisoquinoline (**1c**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



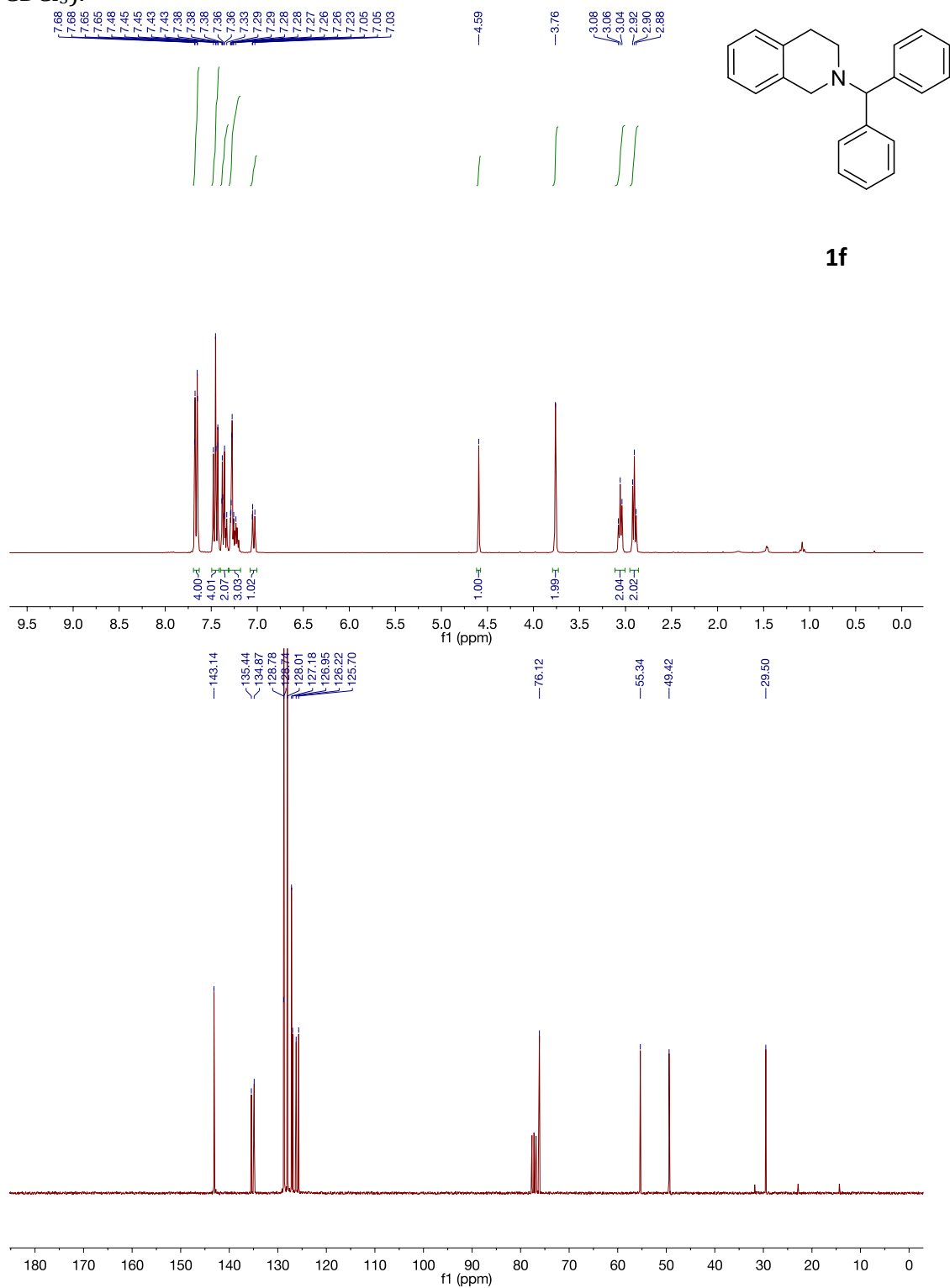
2-(3-phenylpropyl)-1,2,3,4-tetrahydroisoquinoline (**1d**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



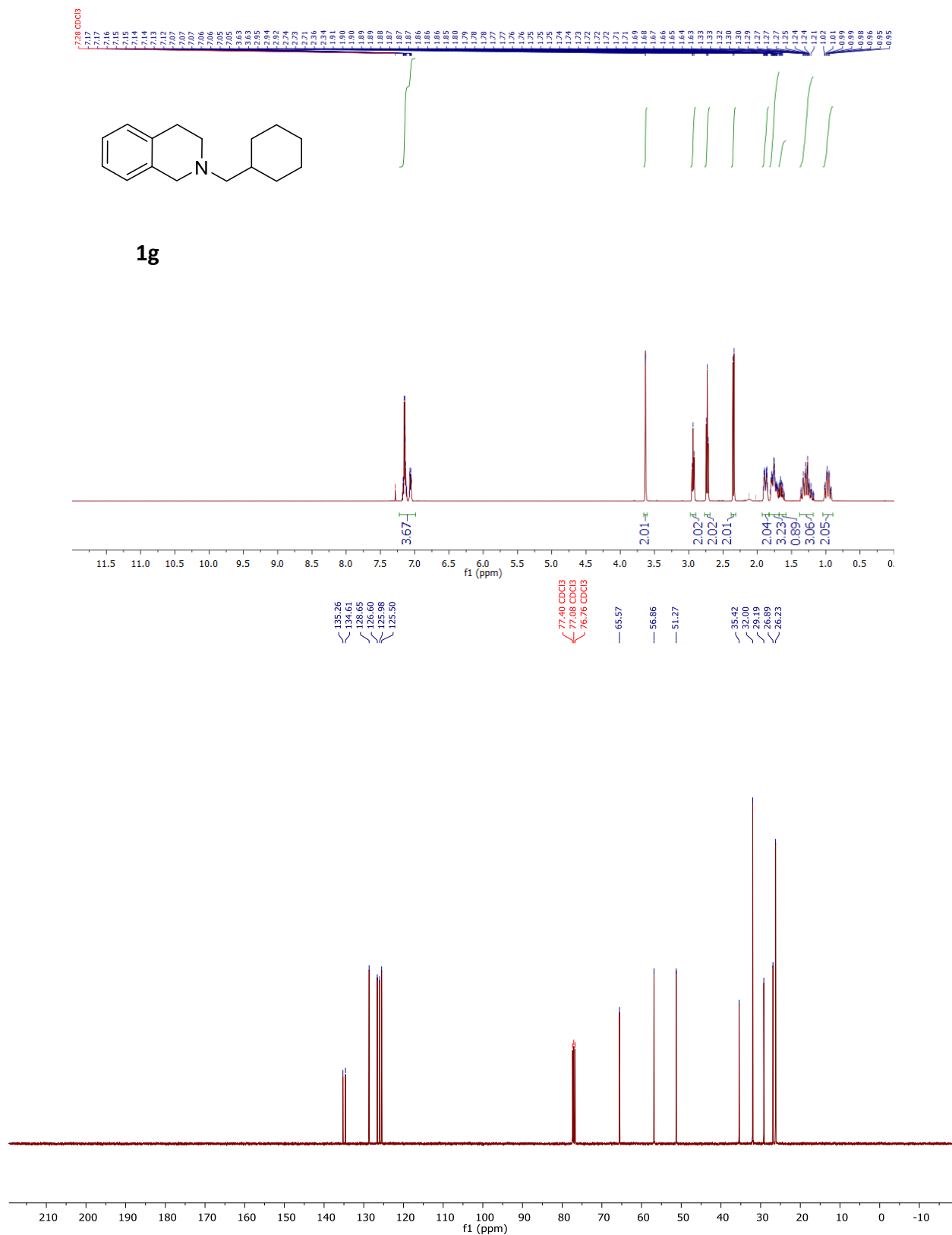
2-benzyl-2,6-dimethoxy-1,2,3,4-tetrahydroisoquinoline (**1e**); ^1H NMR (300 MHz, CDCl_3), ^{13}C NMR (76 MHz, CDCl_3).



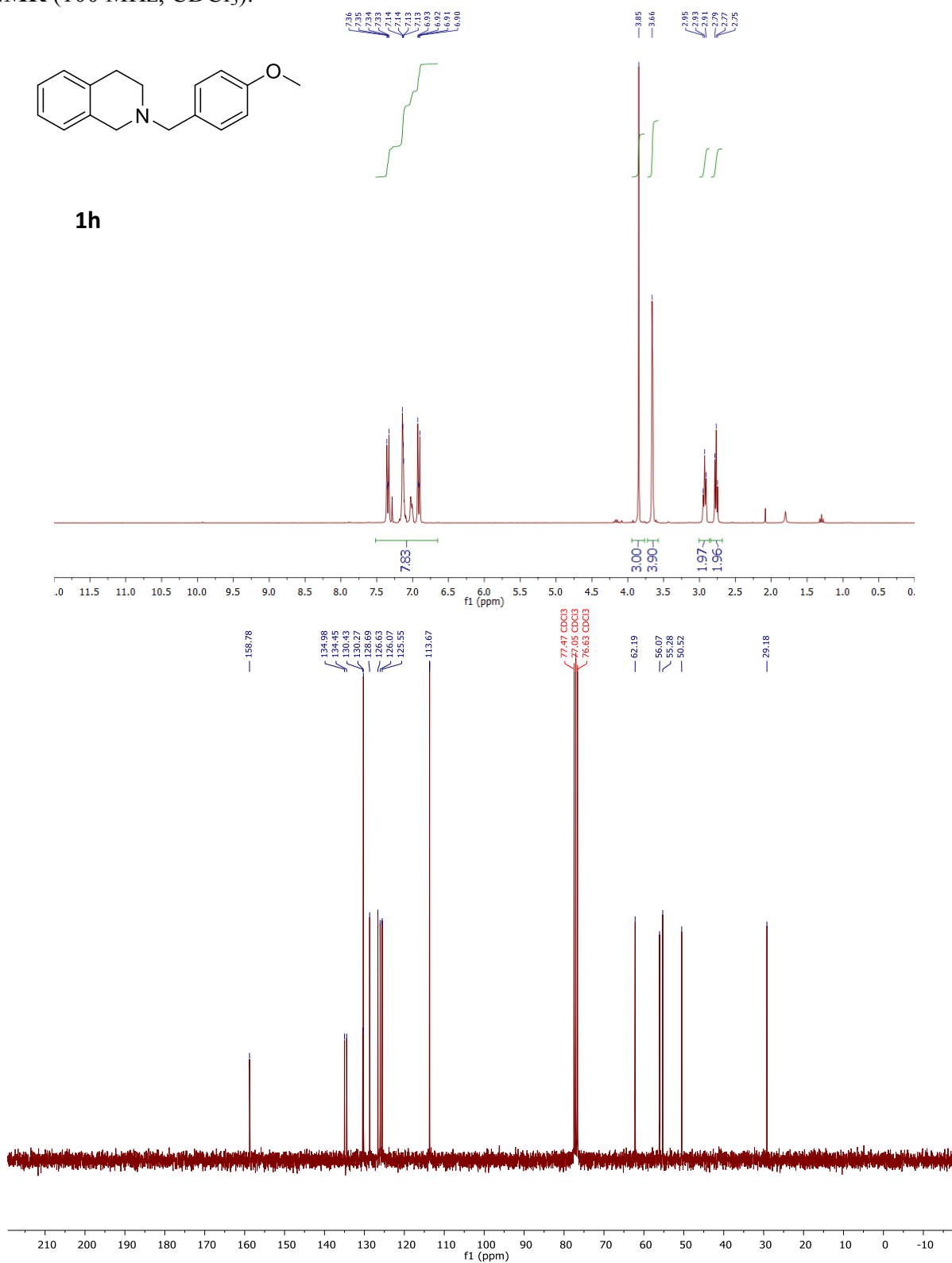
2-benzhydryl-1,2,3,4-tetrahydroisoquinoline (**1f**); ^1H NMR (300 MHz, CDCl_3), ^{13}C NMR (75 MHz, CDCl_3).



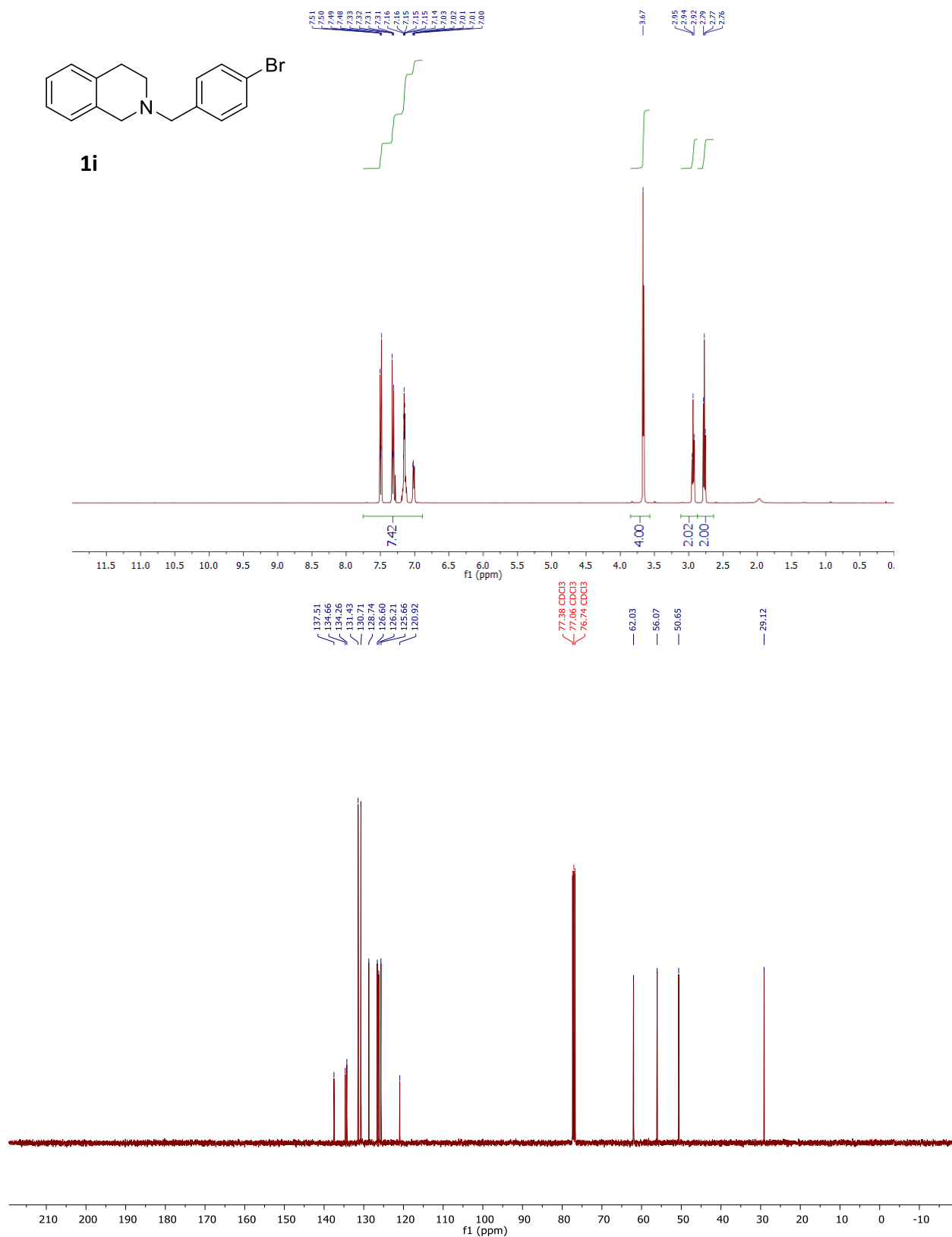
2-(cyclohexylmethyl)-1,2,3,4-tetrahydroisoquinoline (**1g**): ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



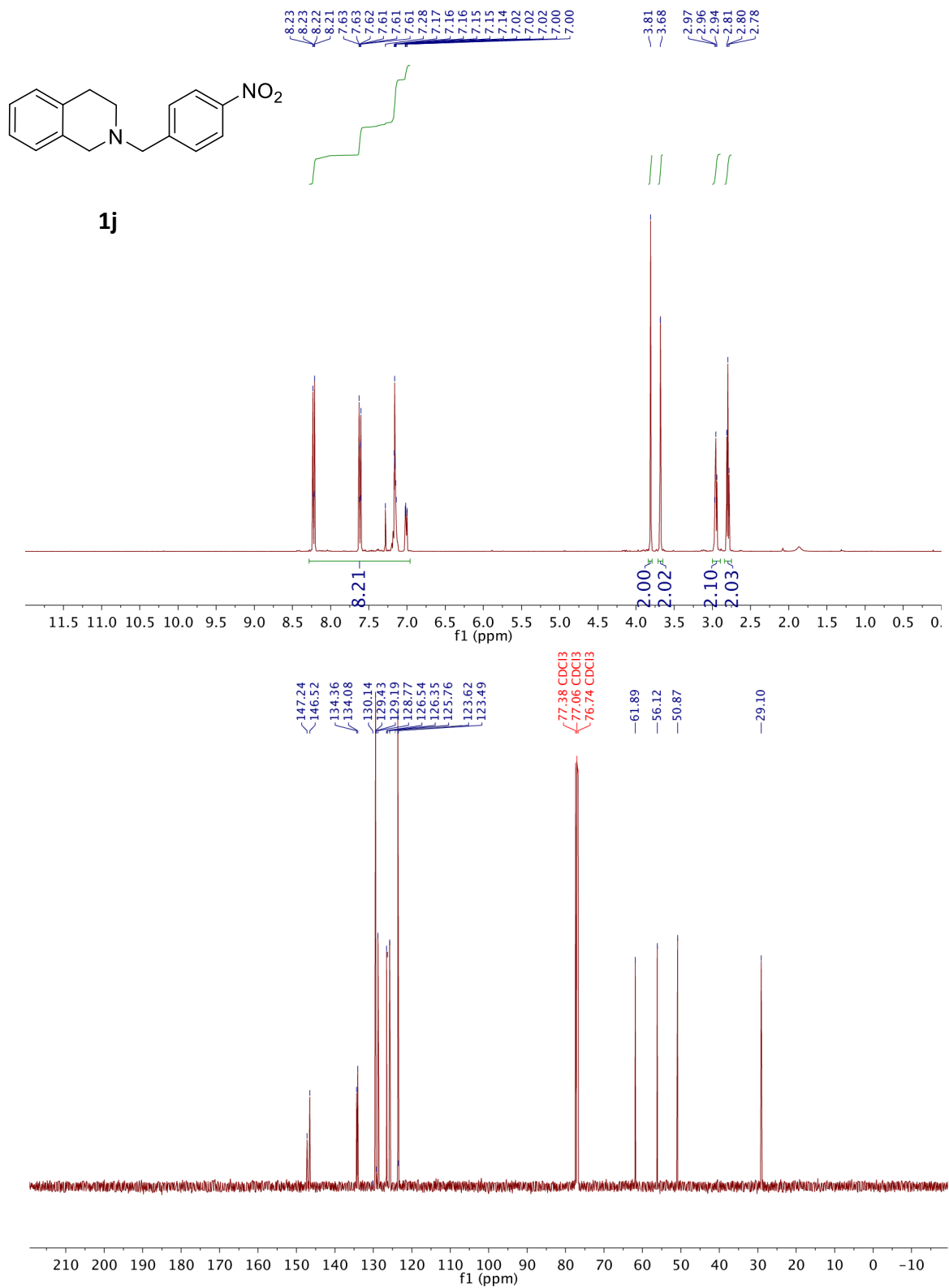
2-(4-methoxybenzyl)-1,2,3,4-tetrahydroisoquinoline (**1h**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



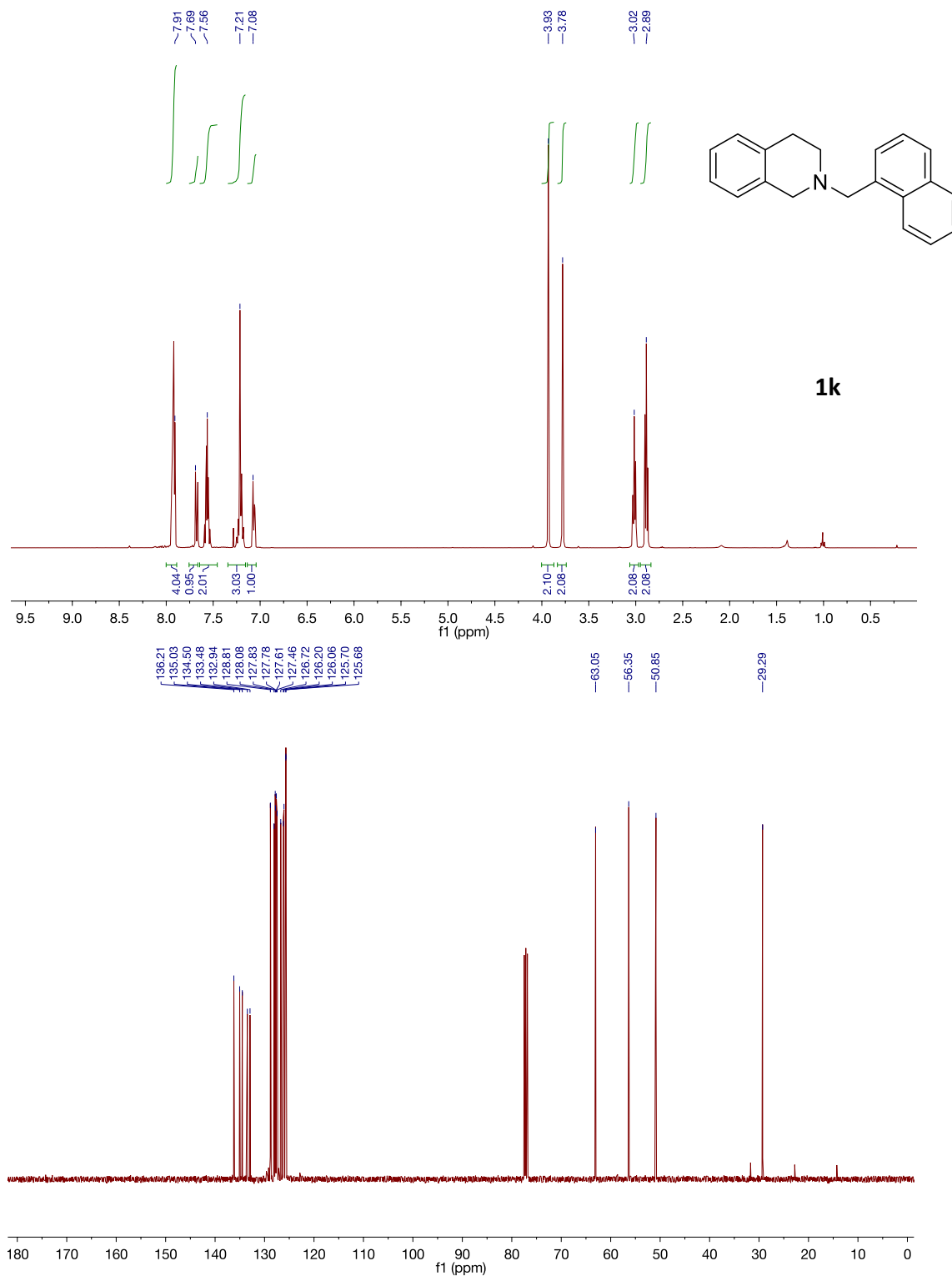
2-(4-bromobenzyl)-1,2,3,4-tetrahydroisoquinoline (**1i**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



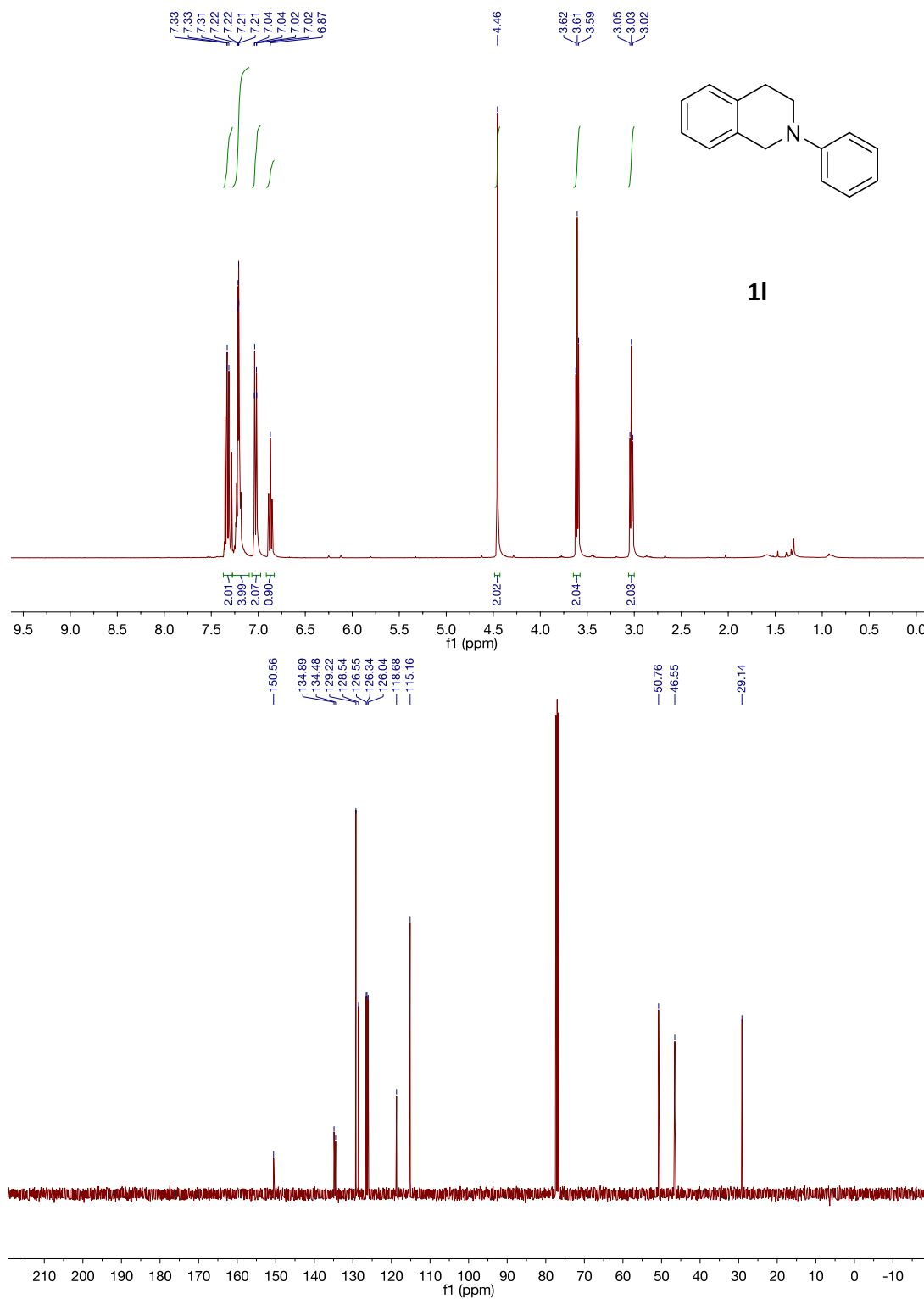
2-(4-nitrobenzyl)-1,2,3,4-tetrahydroisoquinoline (**1j**): ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



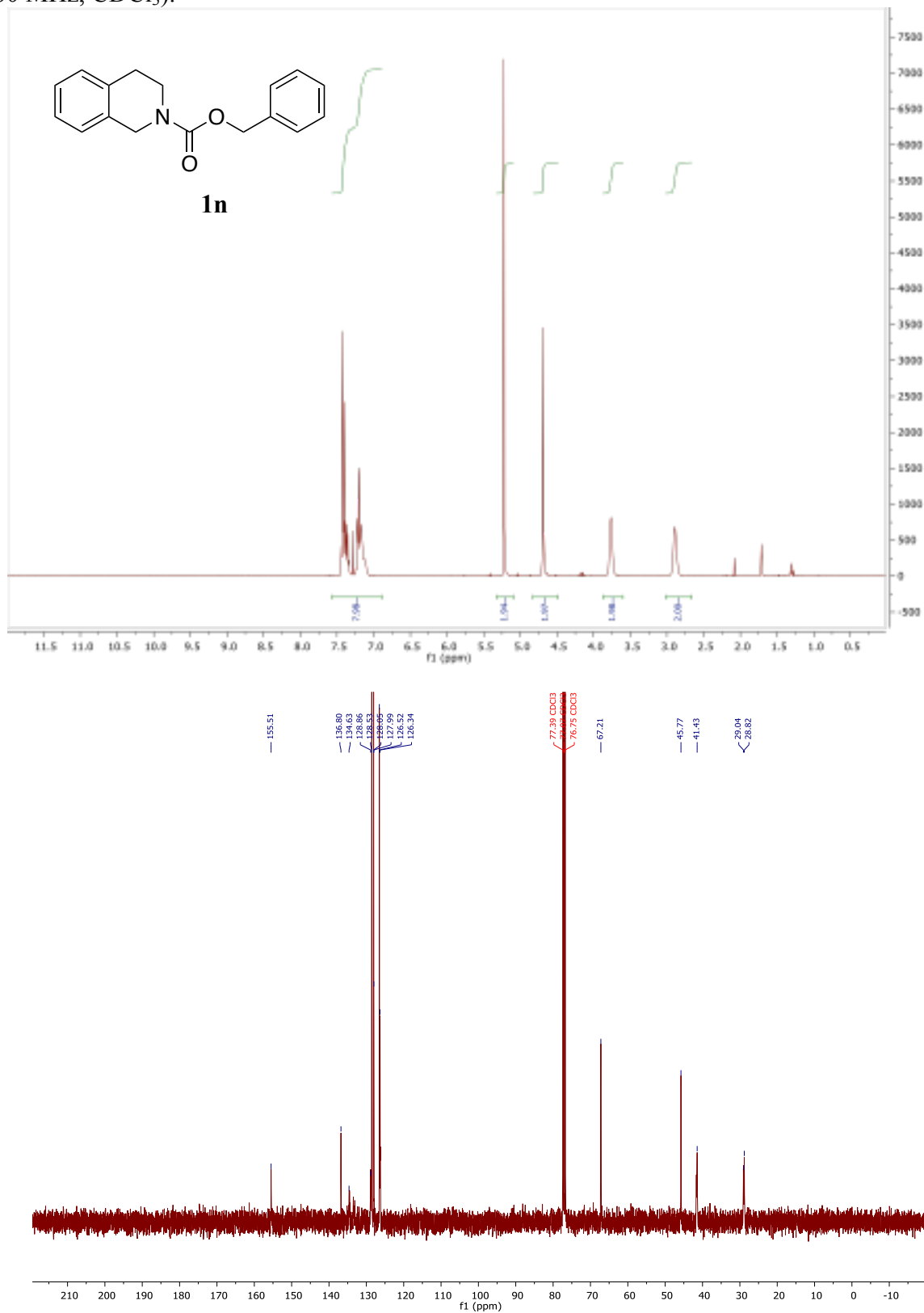
2-(naphthalen-2-ylmethyl)-1,2,3,4-tetrahydroisoquinoline (**1k**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



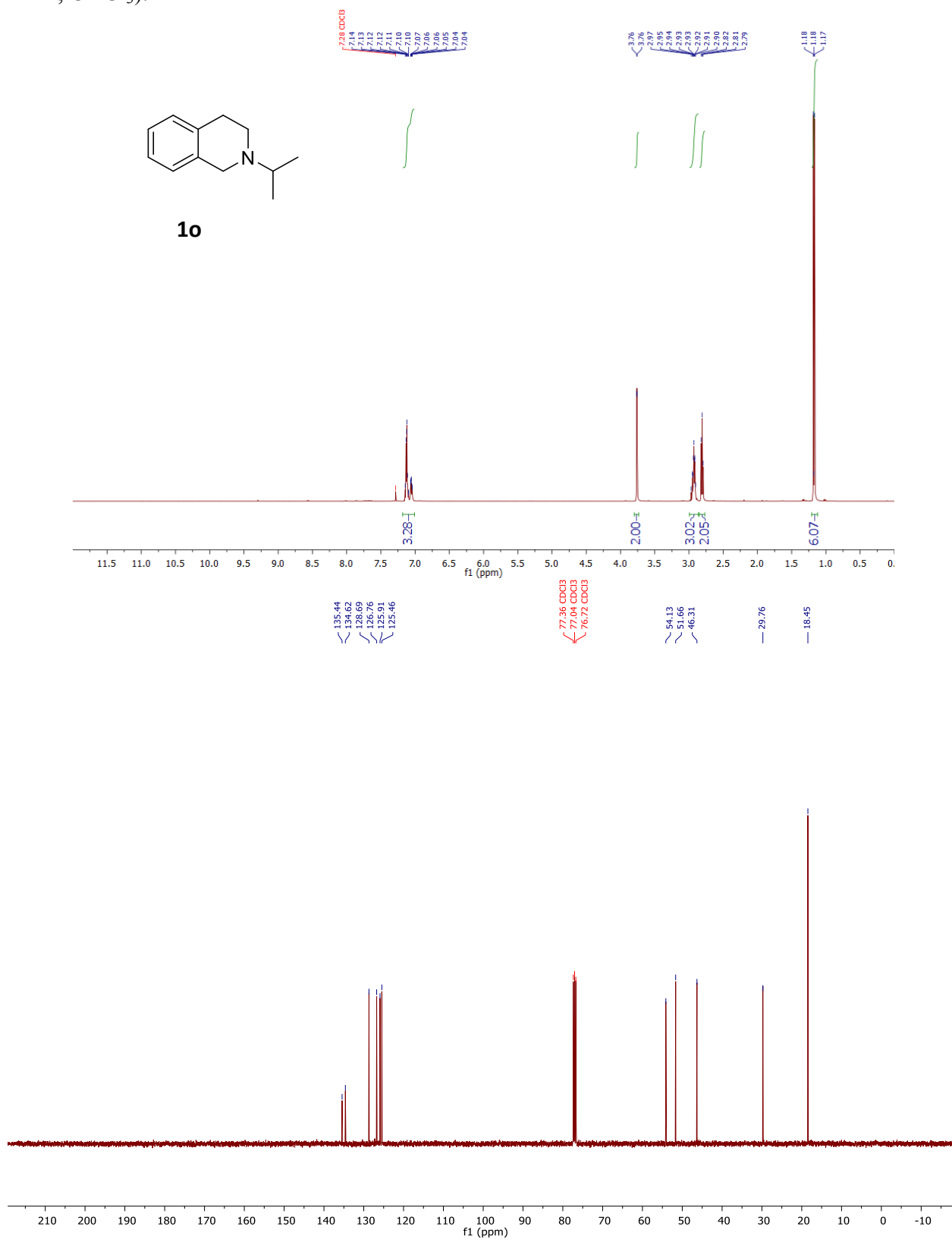
2-phenyl-1,2,3,4-tetrahydroisoquinoline (**1l**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



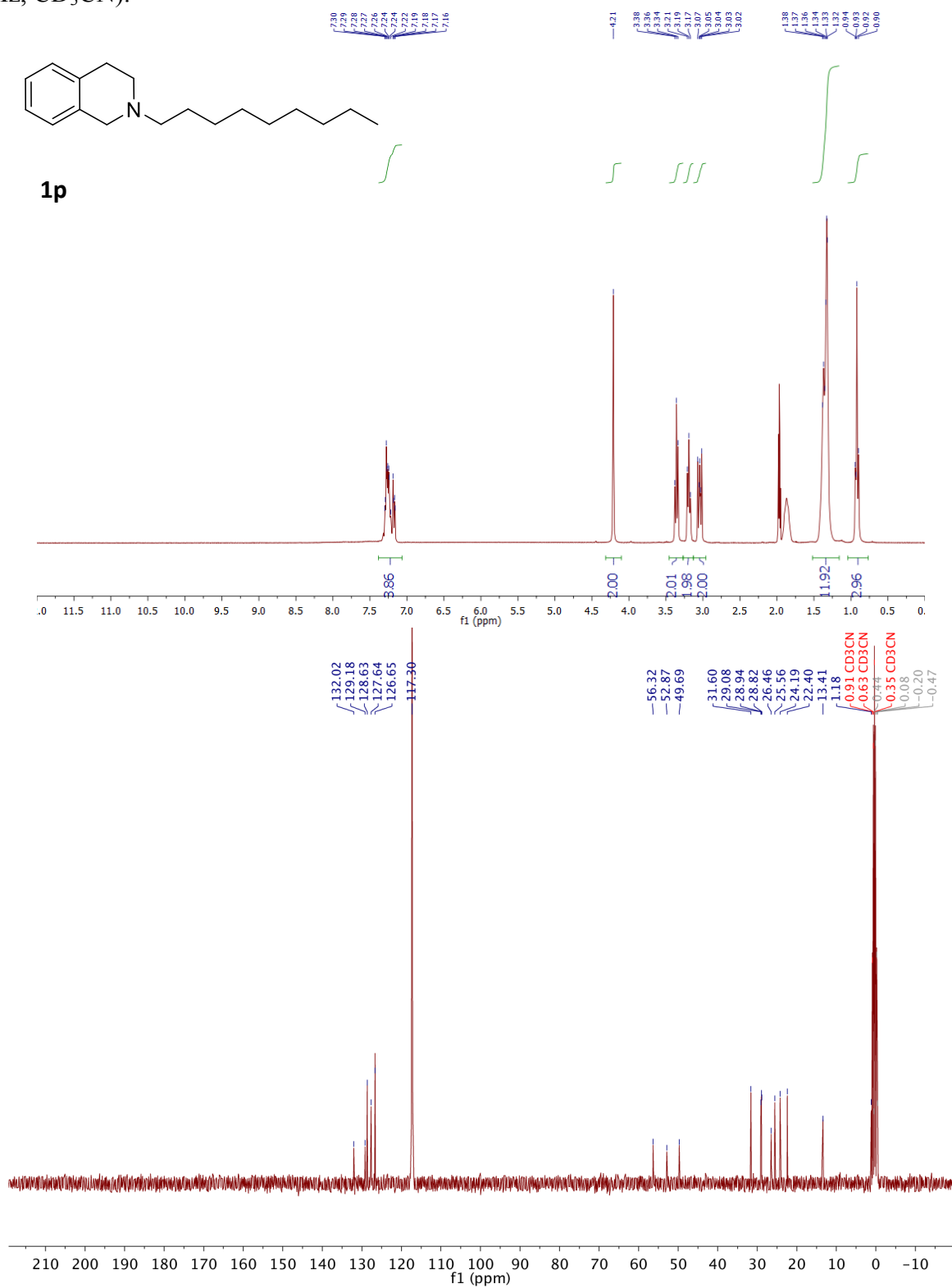
2-carboxybenzyl-1,2,3,4-tetrahydroisoquinoline (**1n**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



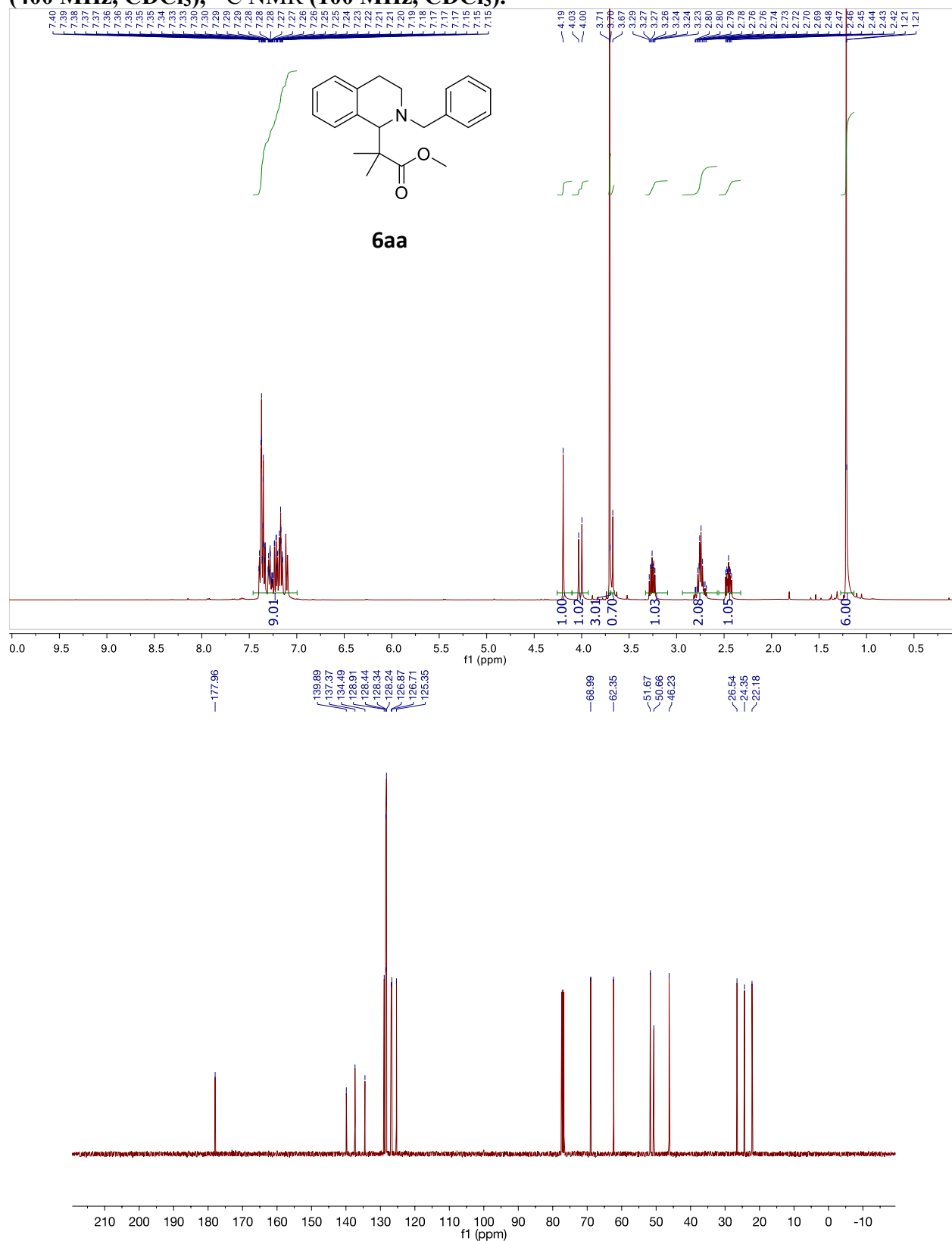
2-isopropyl-1,2,3,4-tetrahydroisoquinoline (**1o**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



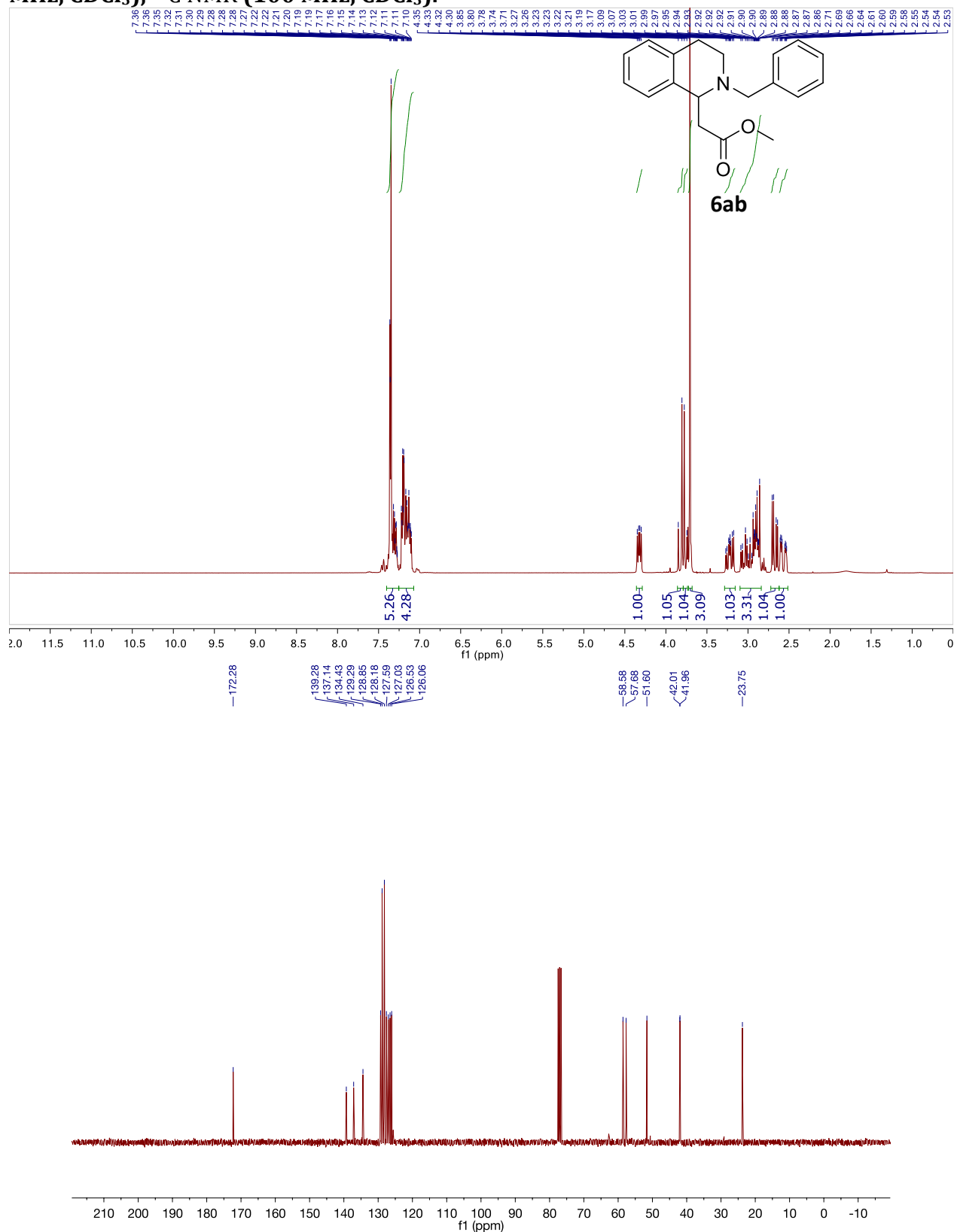
2-nonyl-1,2,3,4-tetrahydroisoquinoline (**1p**); ^1H NMR (400 MHz, CD_3CN), ^{13}C NMR (100 MHz, CD_3CN).



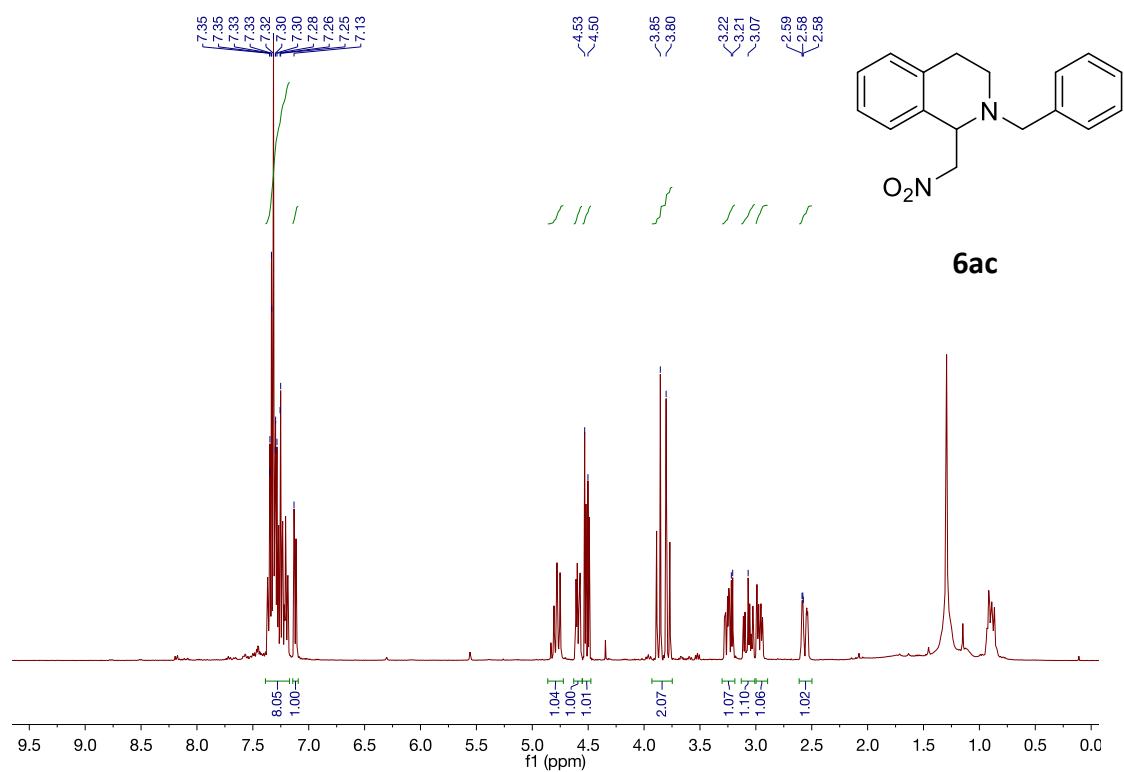
Methyl 2-(2-benzyl-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (6aa); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).

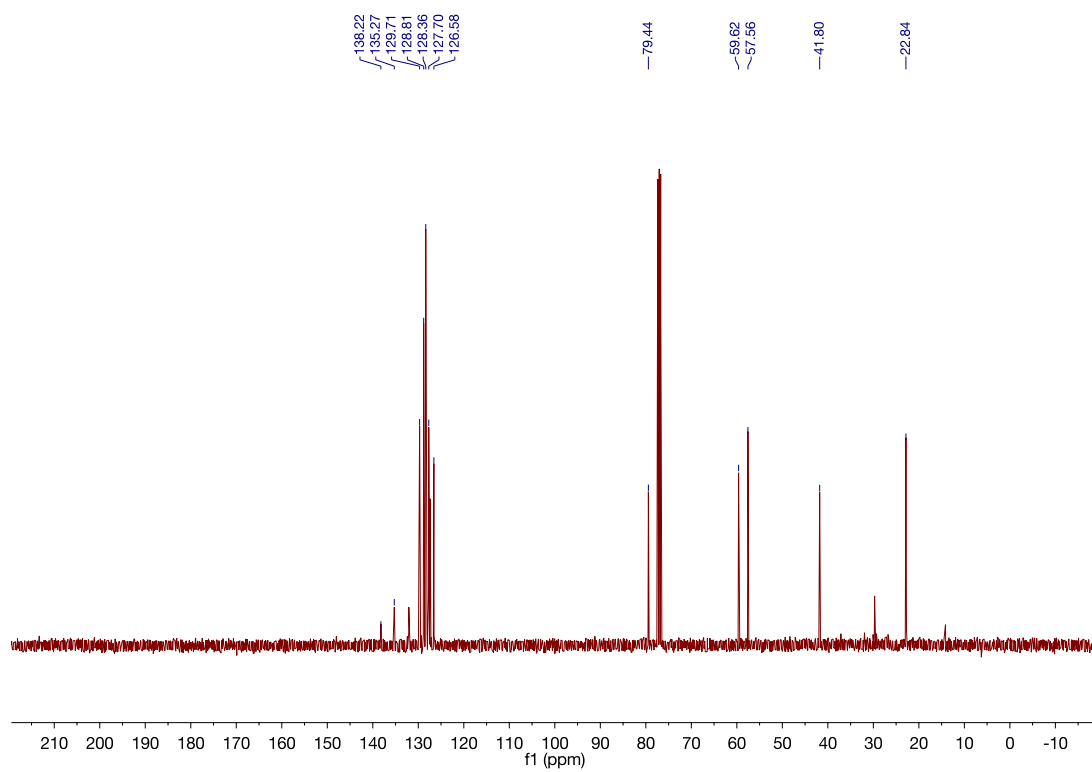


Methyl 2-(2-benzyl-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (6ab); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).

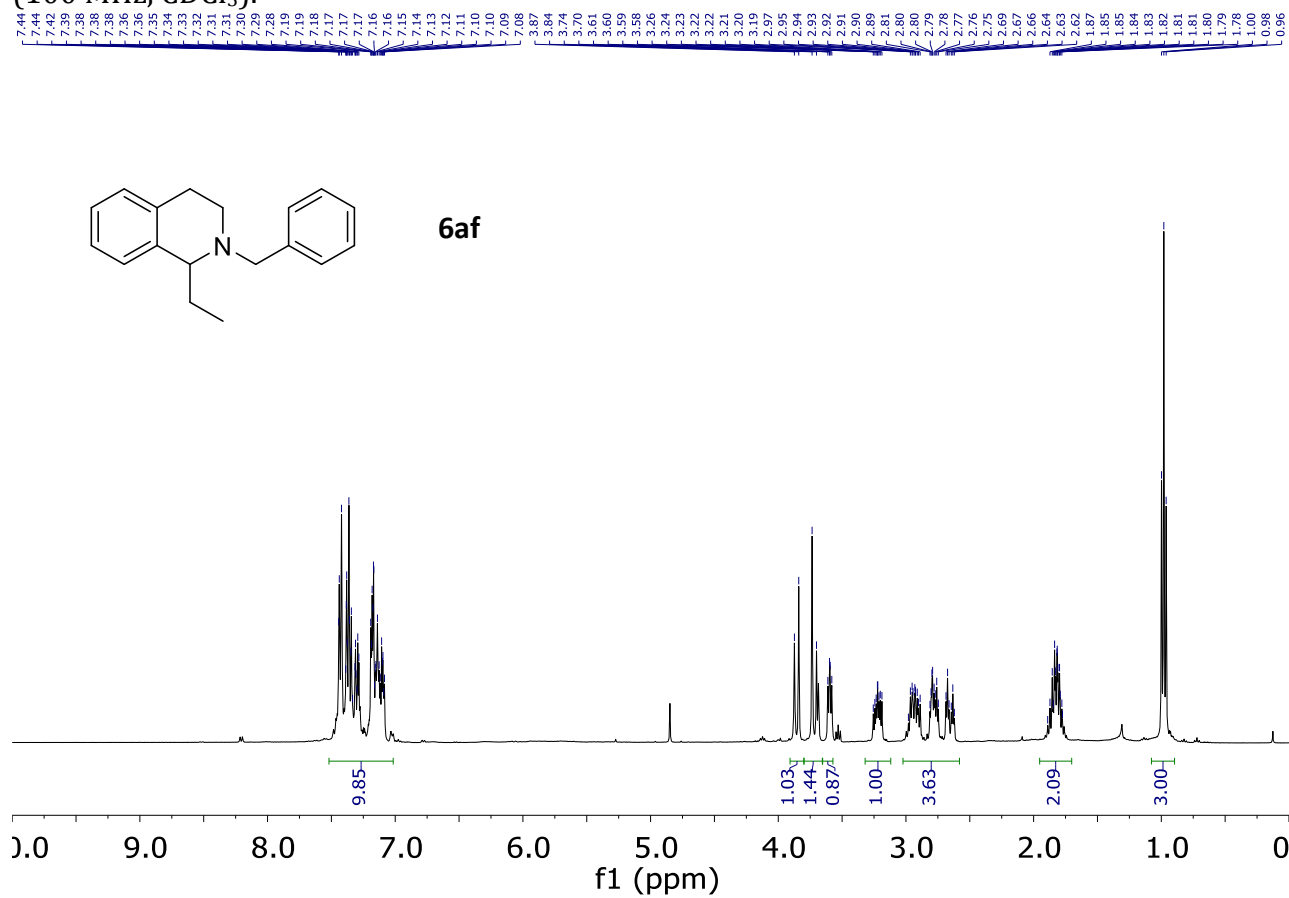


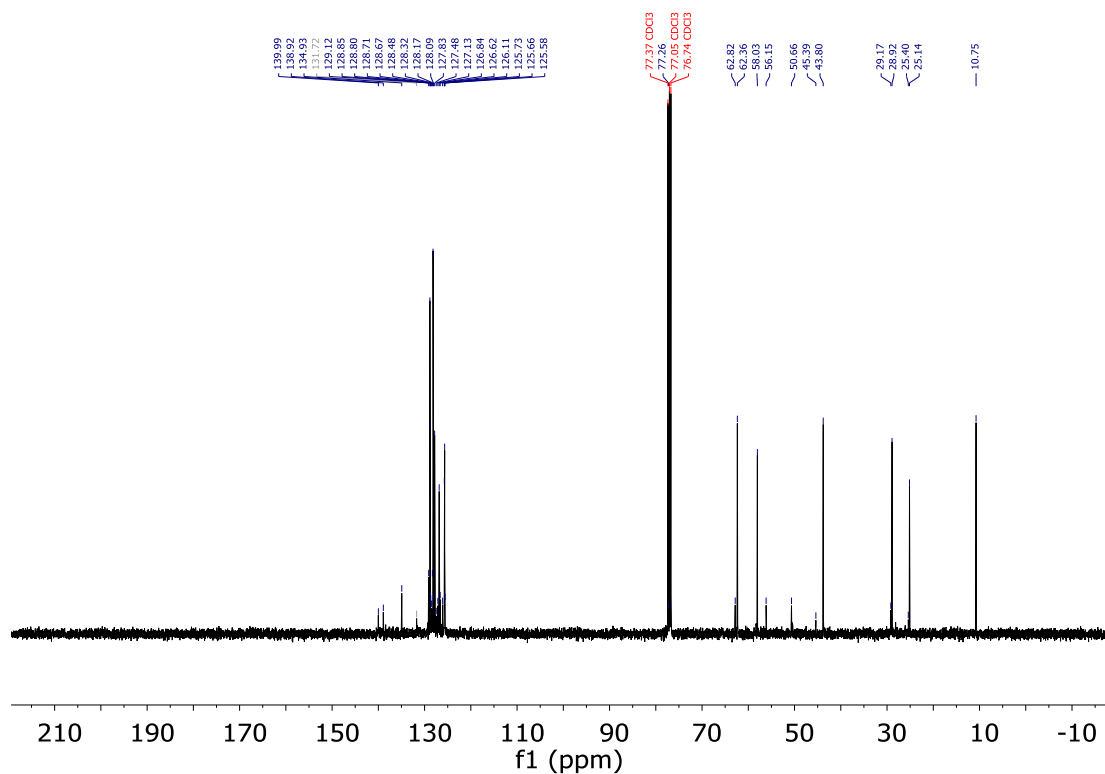
2-benzyl-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (**6ac**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3), HMBC (300 MHz, CDCl_3).



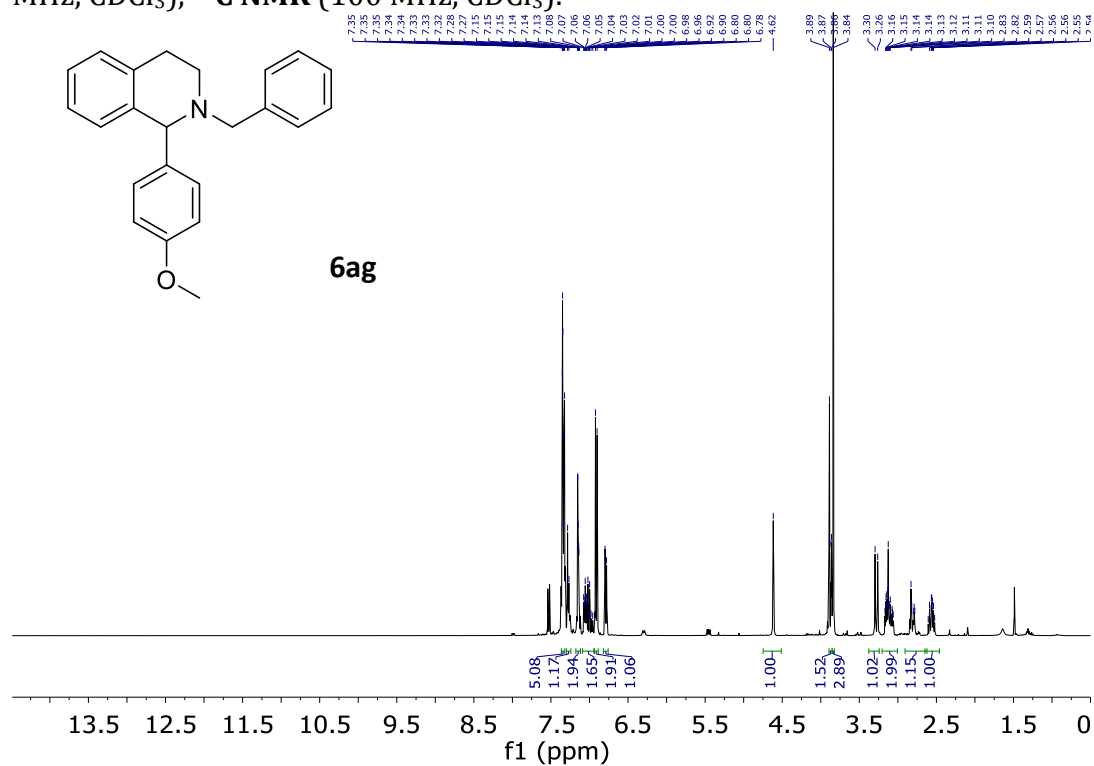


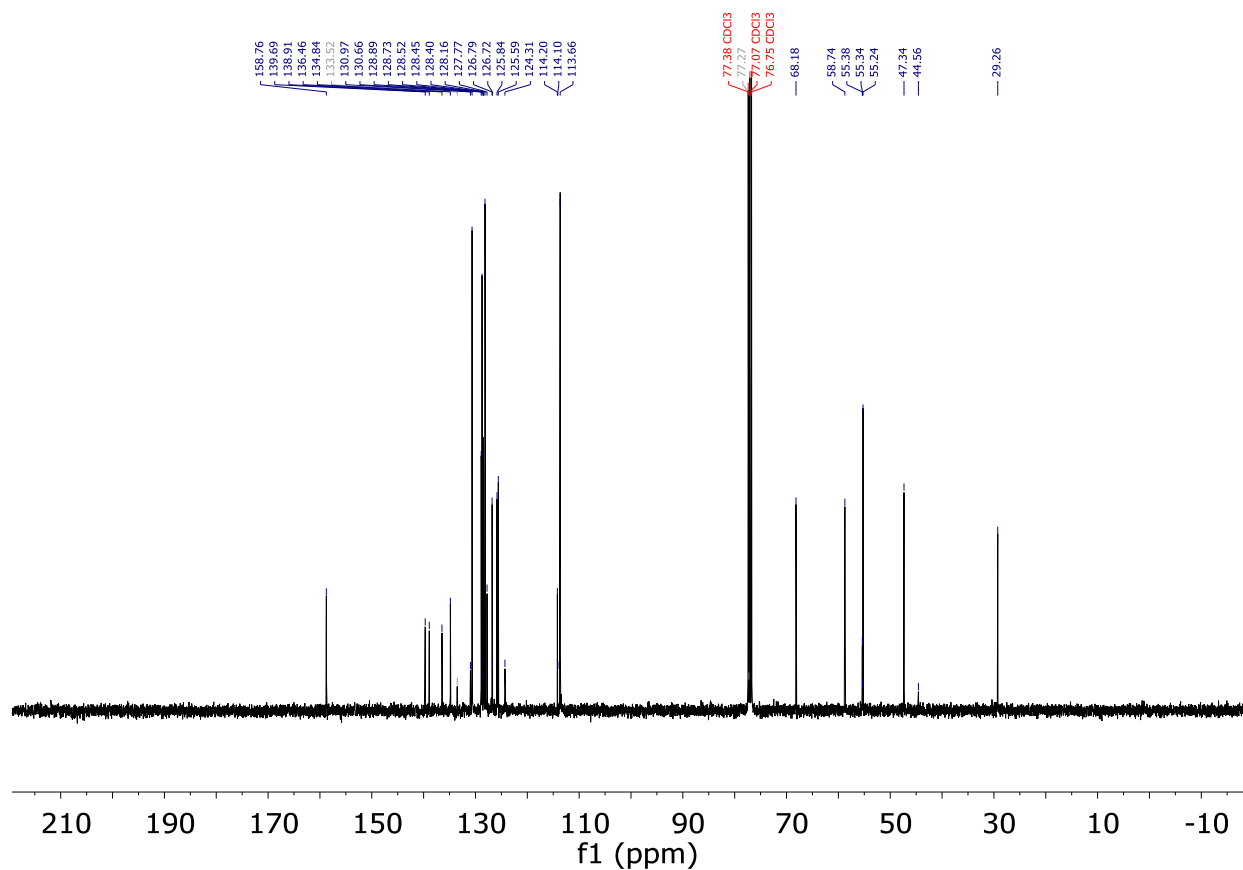
2-benzyl-1-ethyl-1,2,3,4-tetrahydroisoquinoline (**6af**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



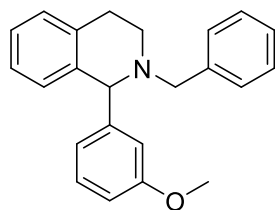


2-benzyl-1-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (6ag); ¹H NMR (400 MHz, CDCl₃), ¹³C NMR (100 MHz, CDCl₃).

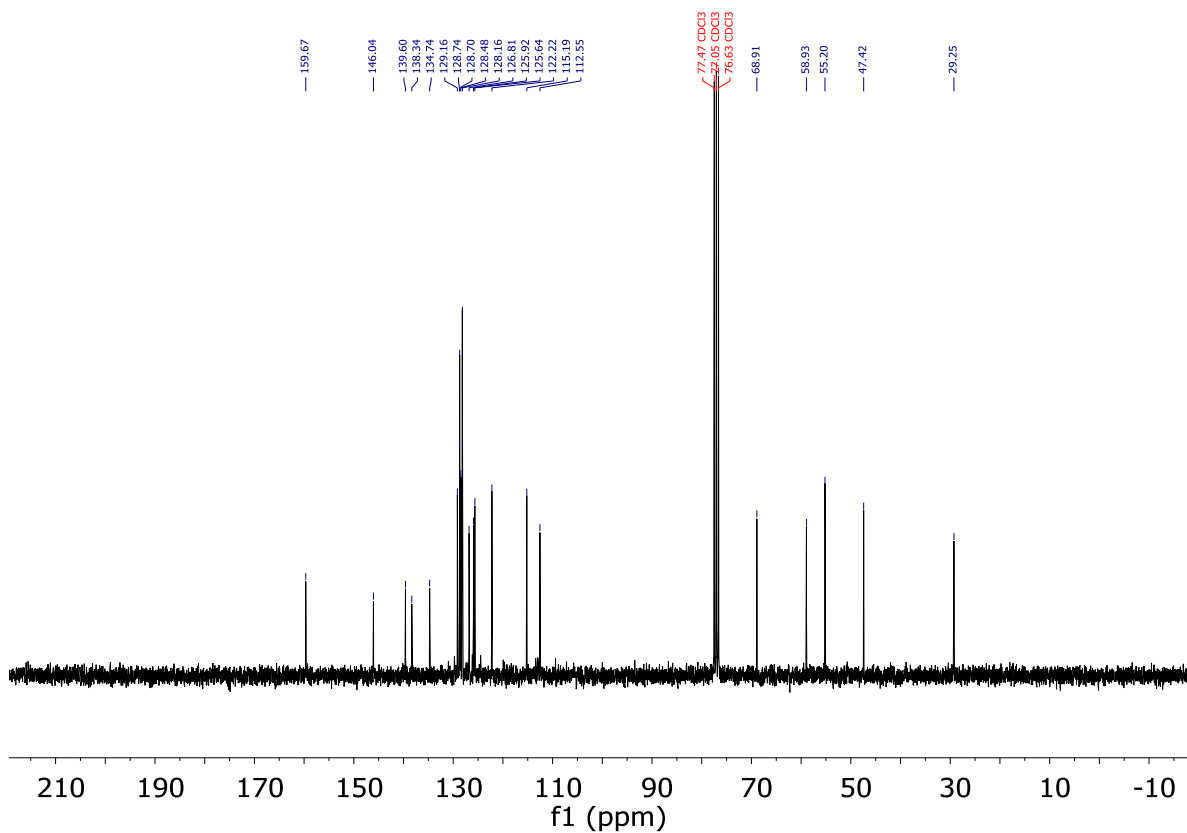
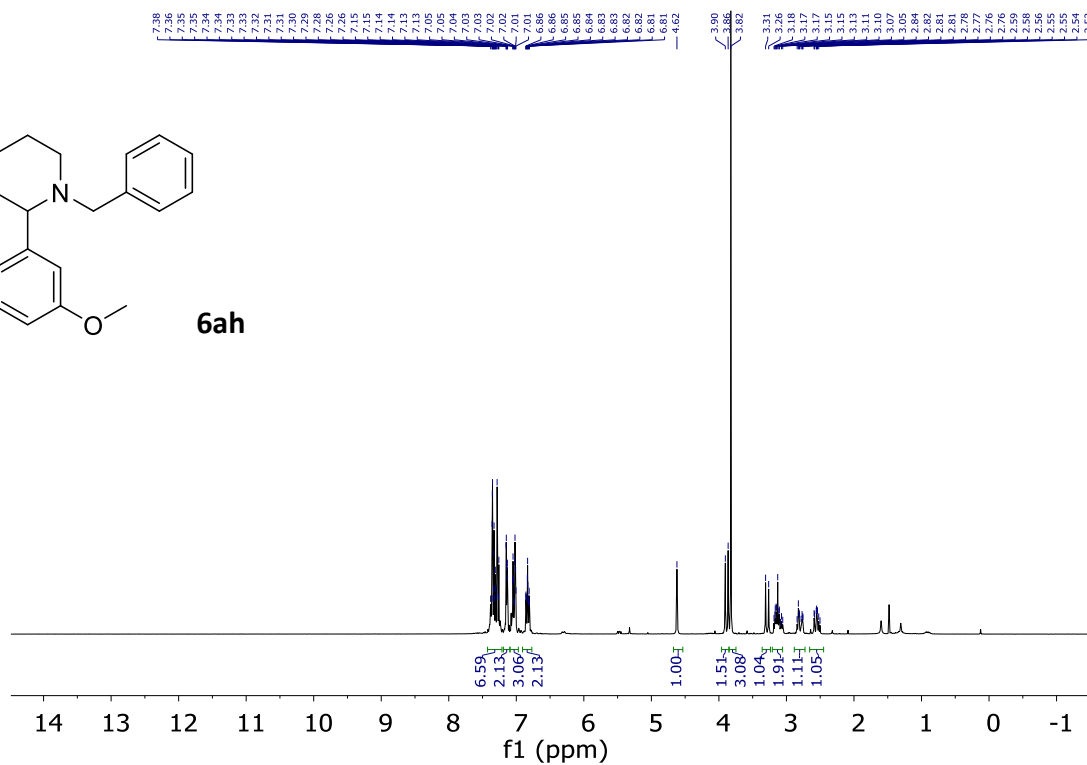




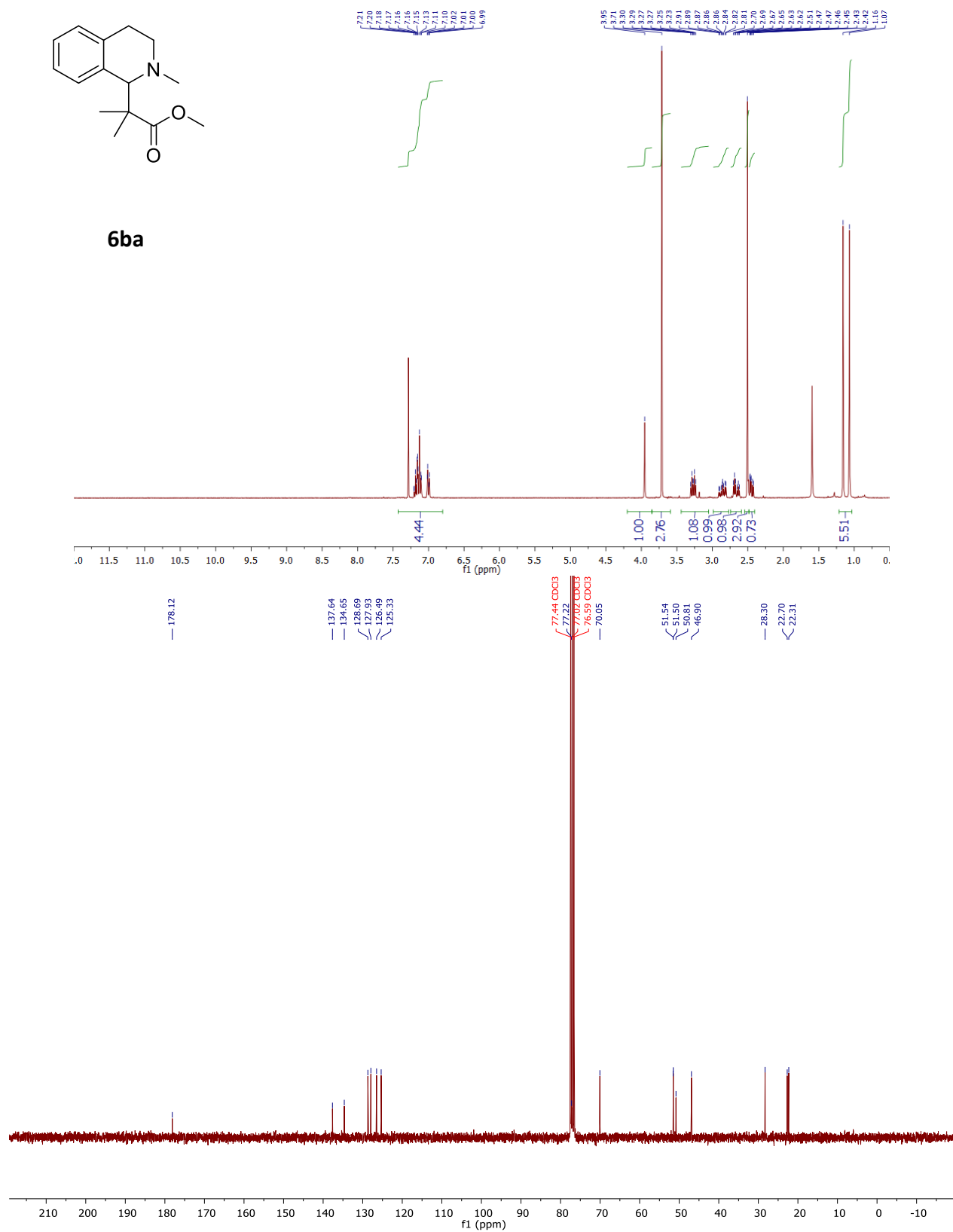
2-benzyl-1-(3-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (**6ah**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



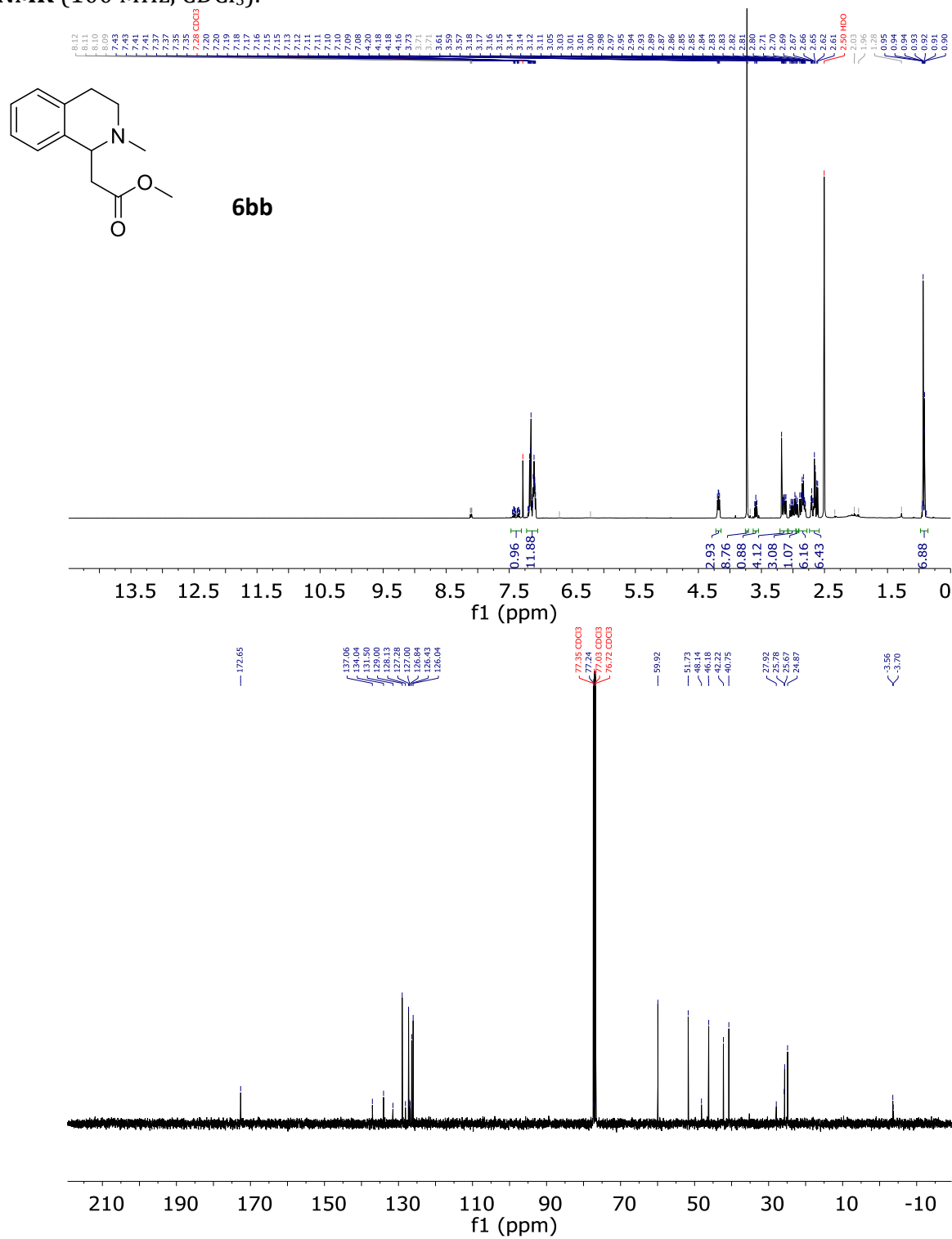
6ah



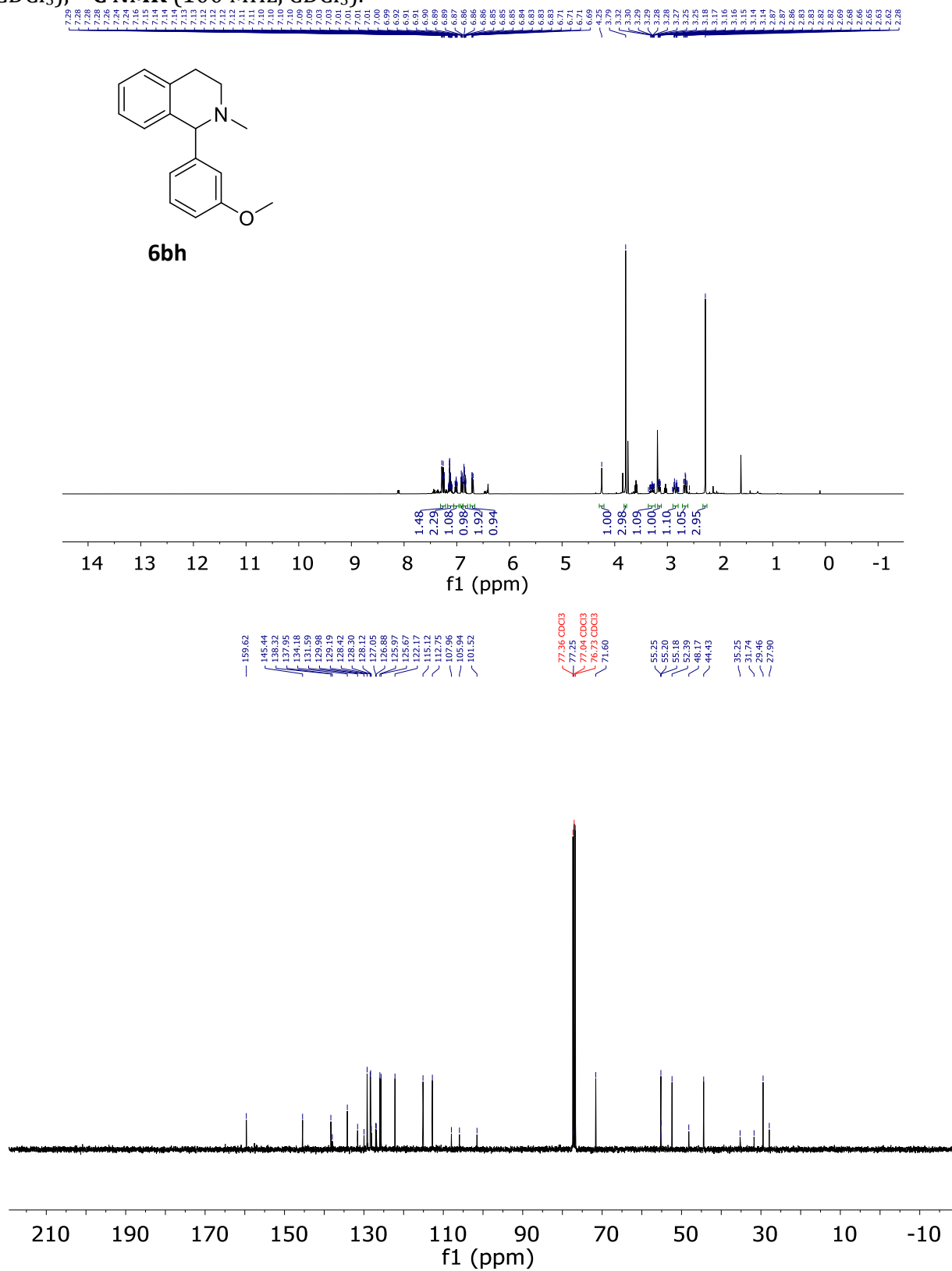
Methyl 2-(1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (**6ba**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



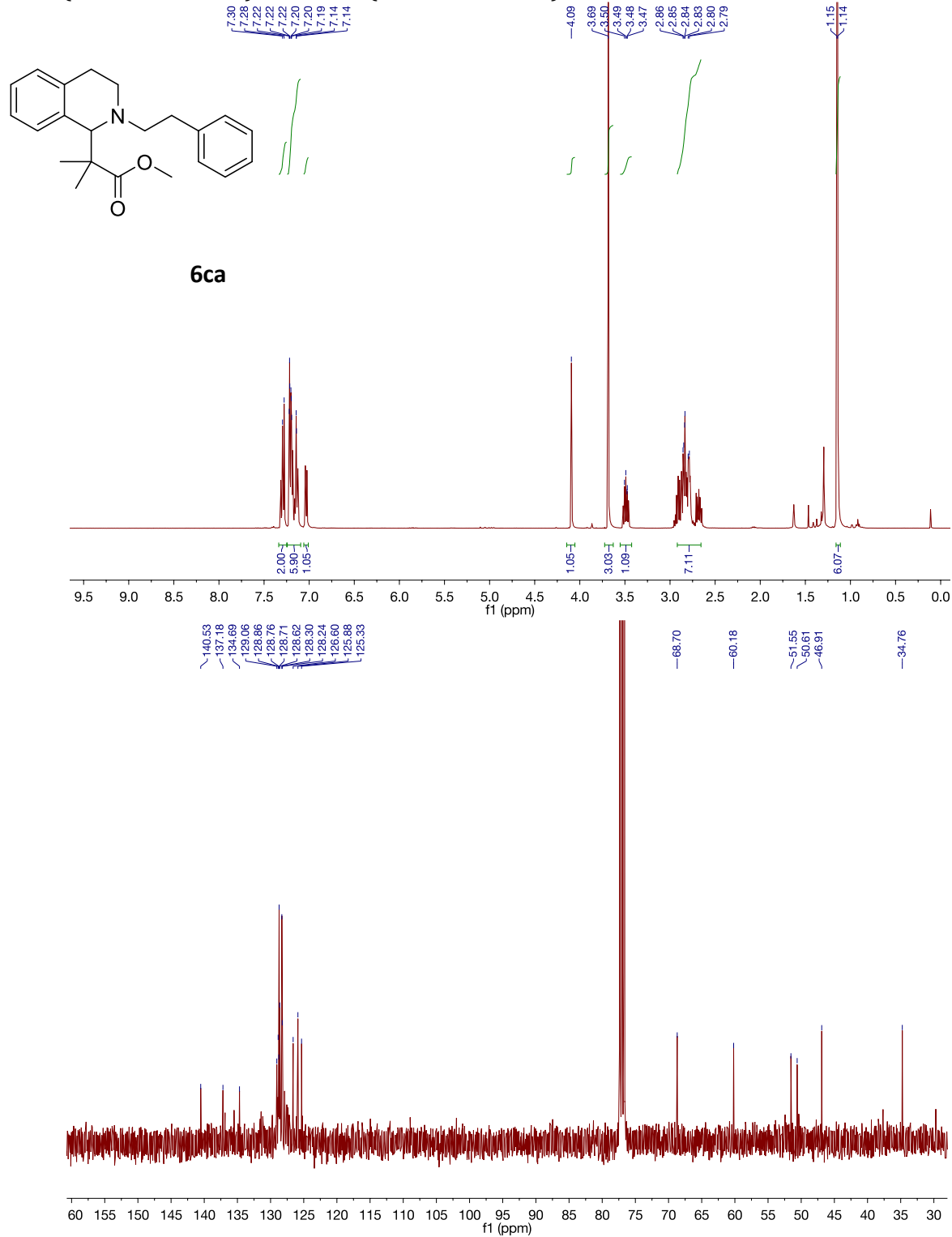
Methyl 2-(1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6bb**) ; ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



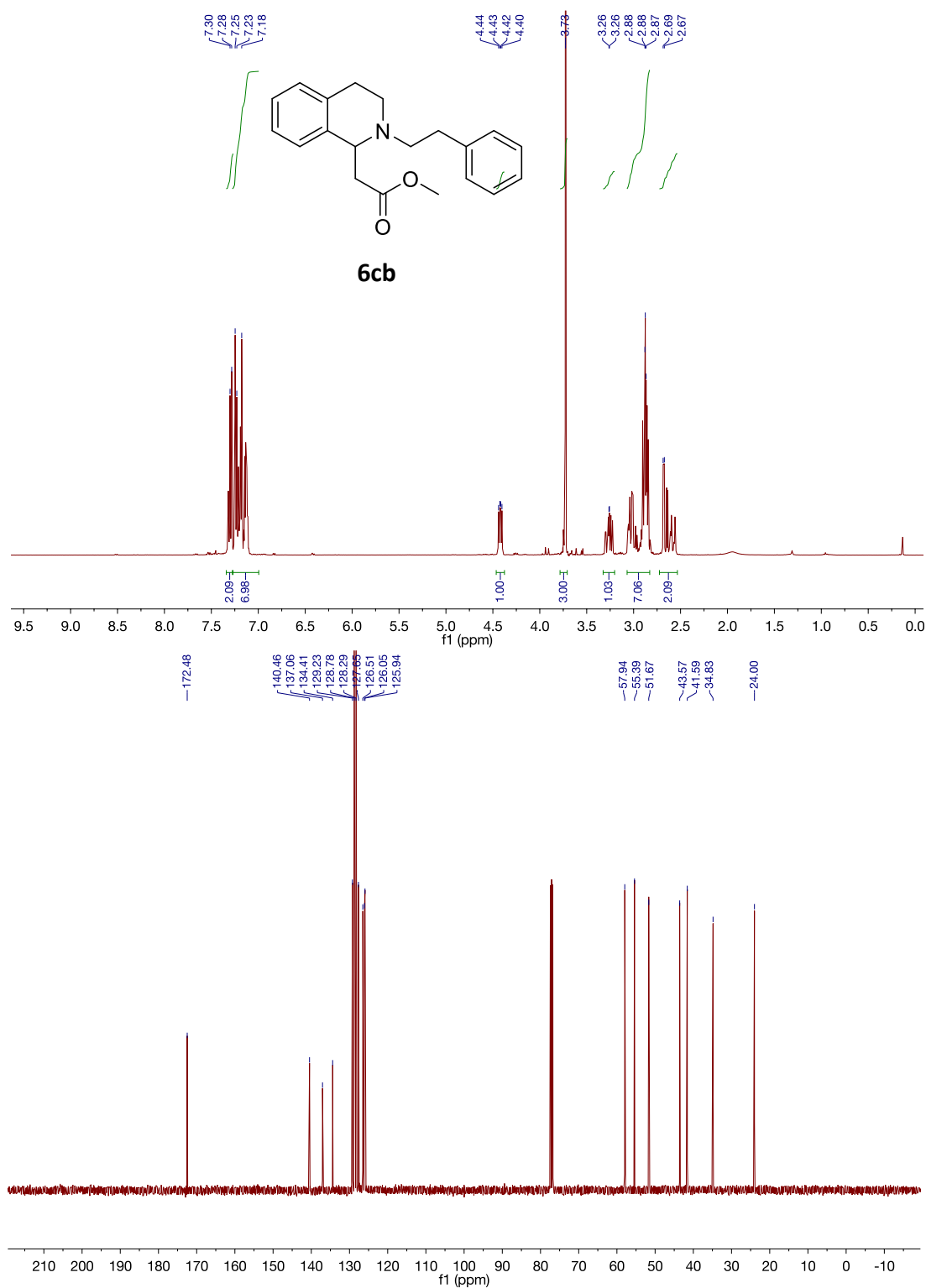
1-(3-methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroisoquinoline (**6bh**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



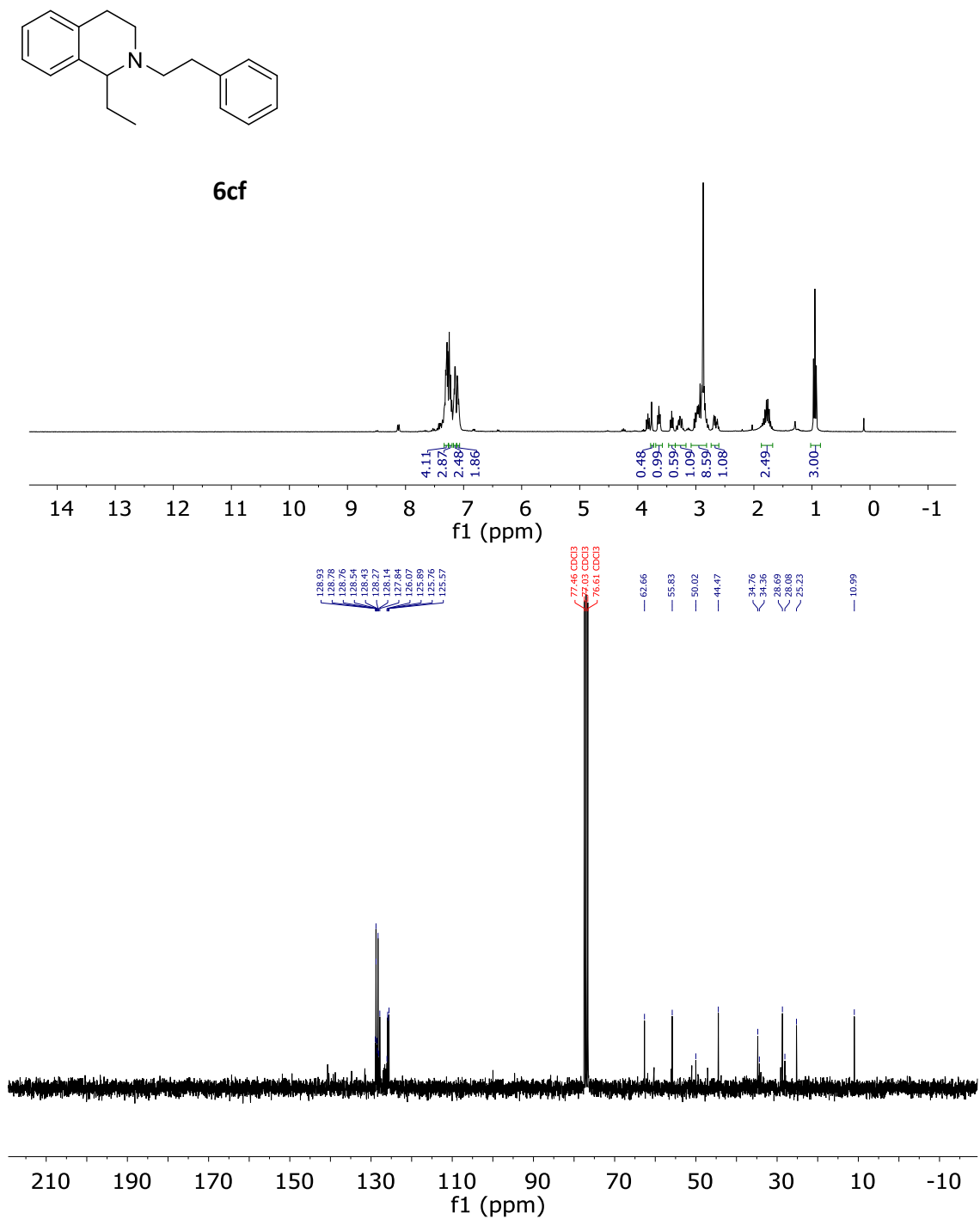
Methyl 2-methyl-2-(2-phenethyl-1,2,3,4-tetrahydroisoquinolin-1-yl)propanoate (**6ca**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



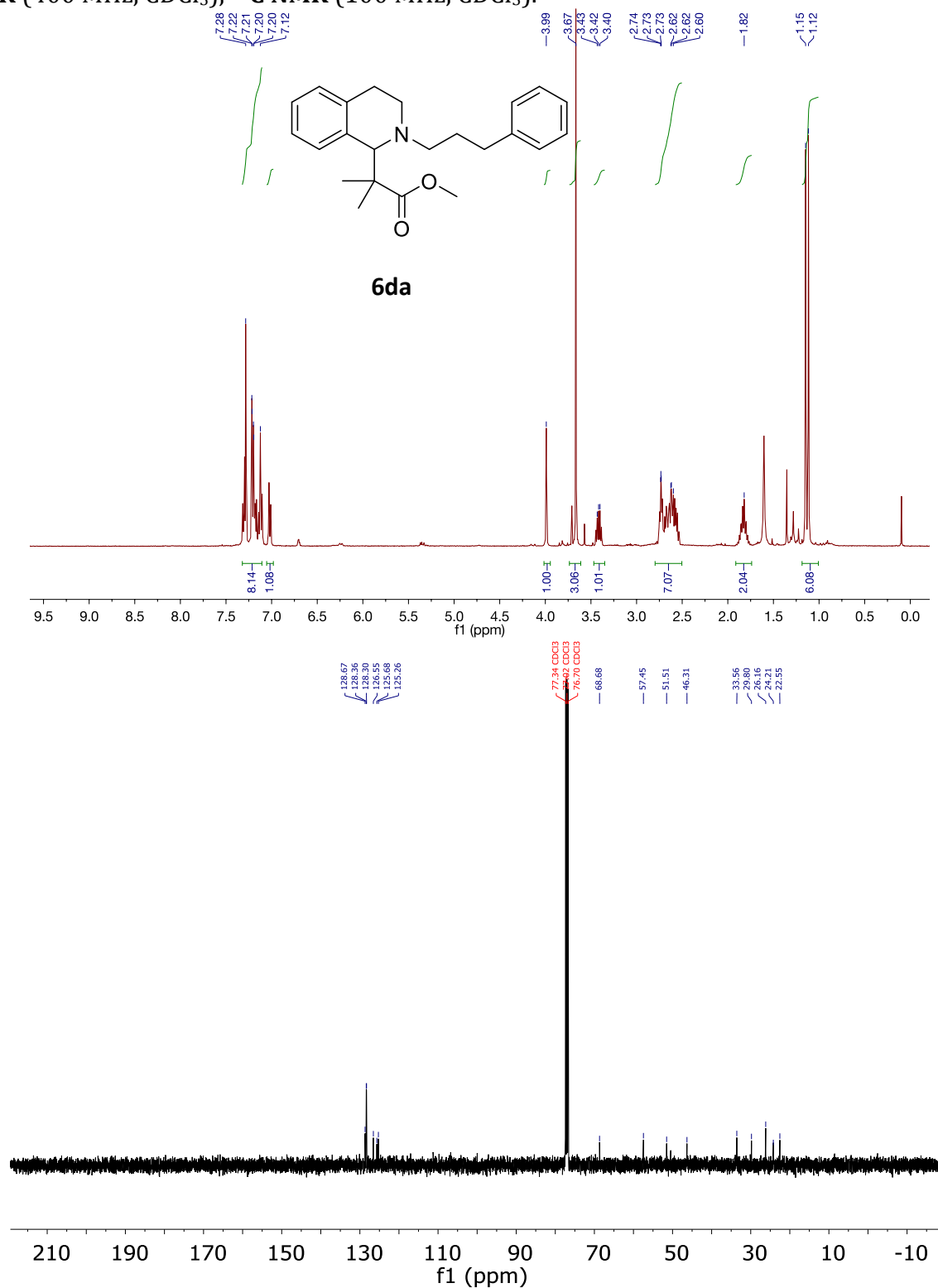
Methyl 2-(2-phenethyl-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6cb**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



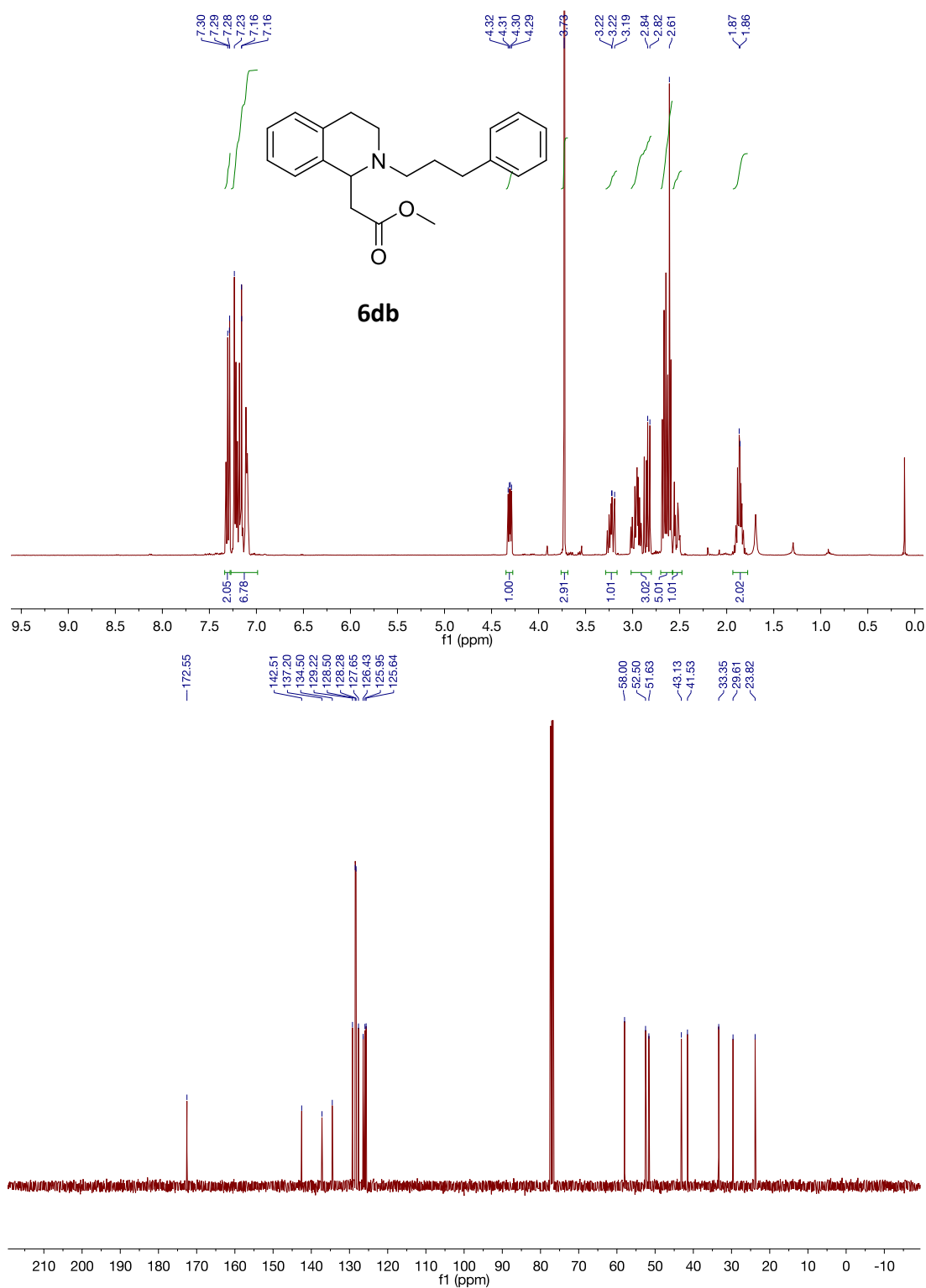
1-ethyl-2-phenylethyl-1,2,3,4-tetrahydroisoquinoline (**6cf**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



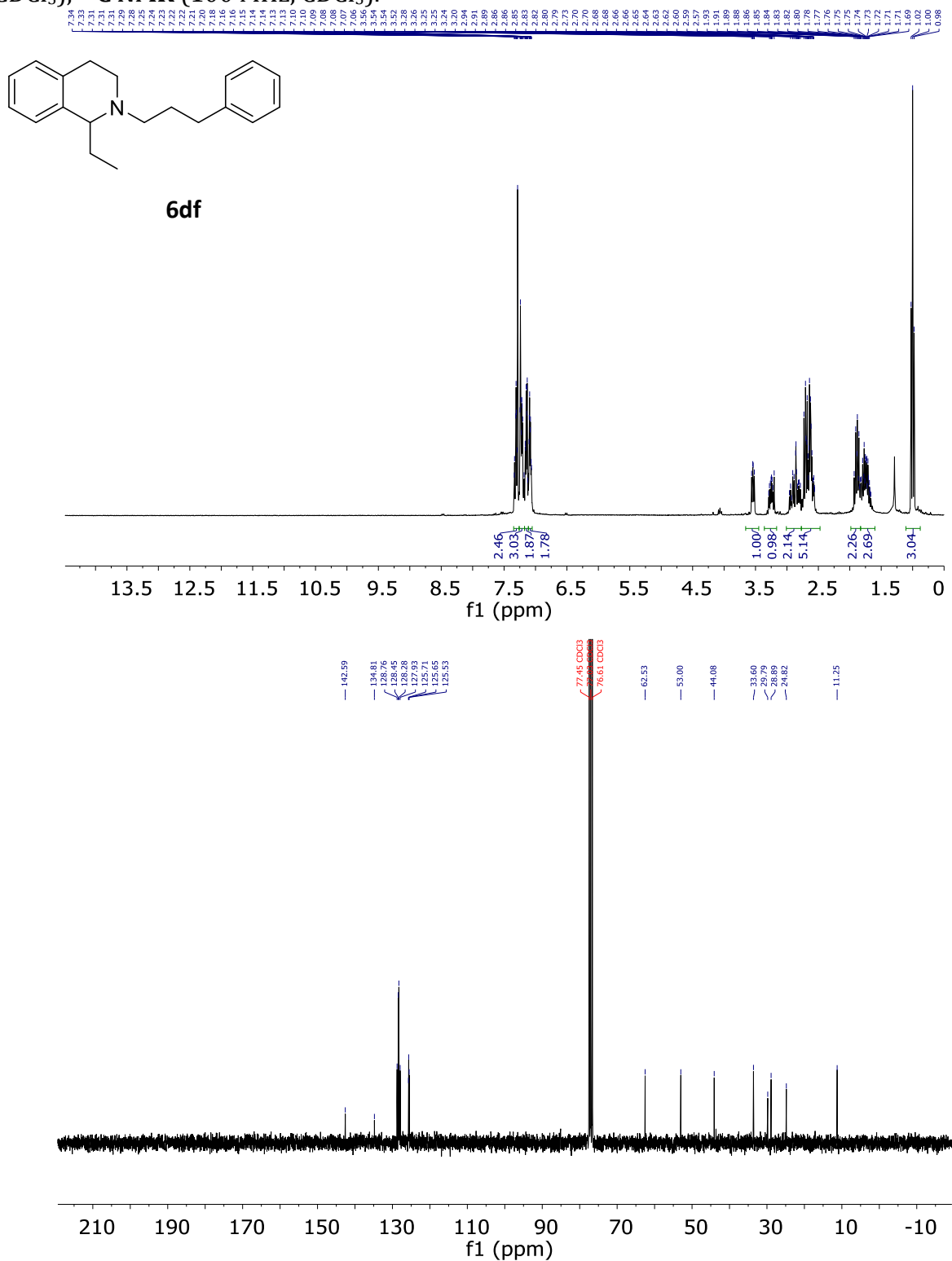
Methyl 2-methyl-2-(2-(3-phenylpropyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propanoate (**6da**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



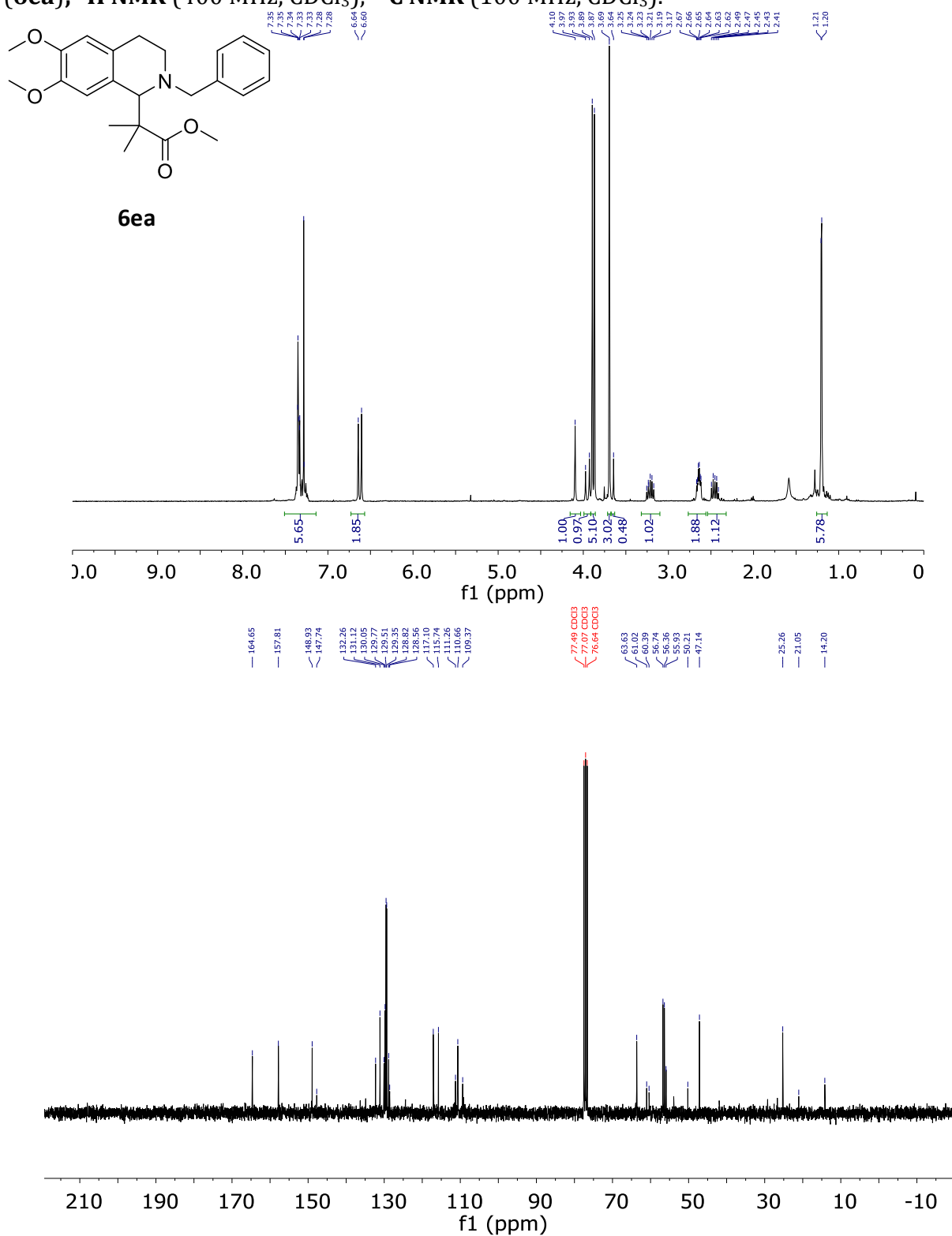
Methyl 2-(2-(3-phenylpropyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6db**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



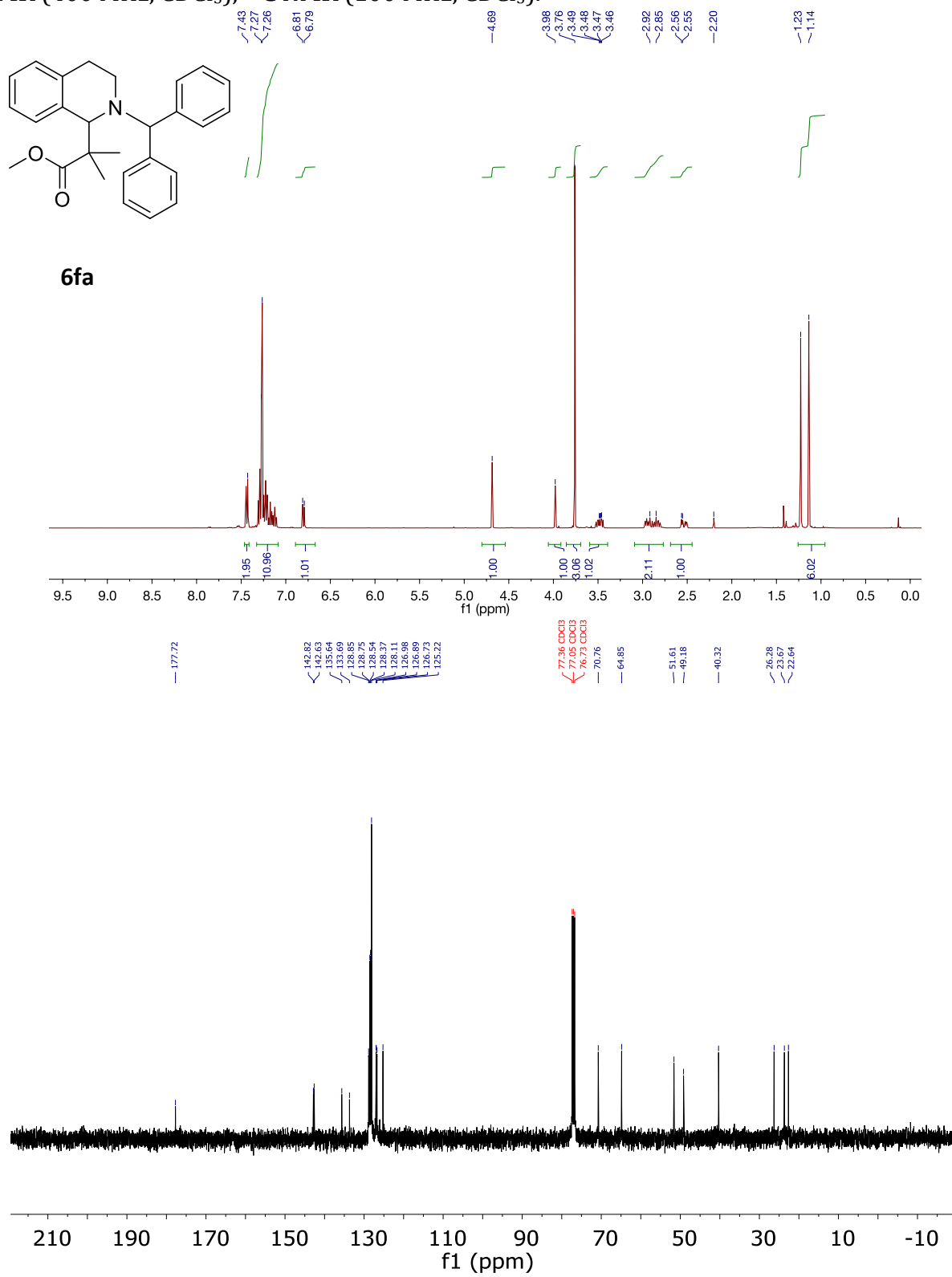
1-ethyl-2-(3-phenylpropyl)-1,2,3,4-tetrahydroisoquinoline (**6df**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



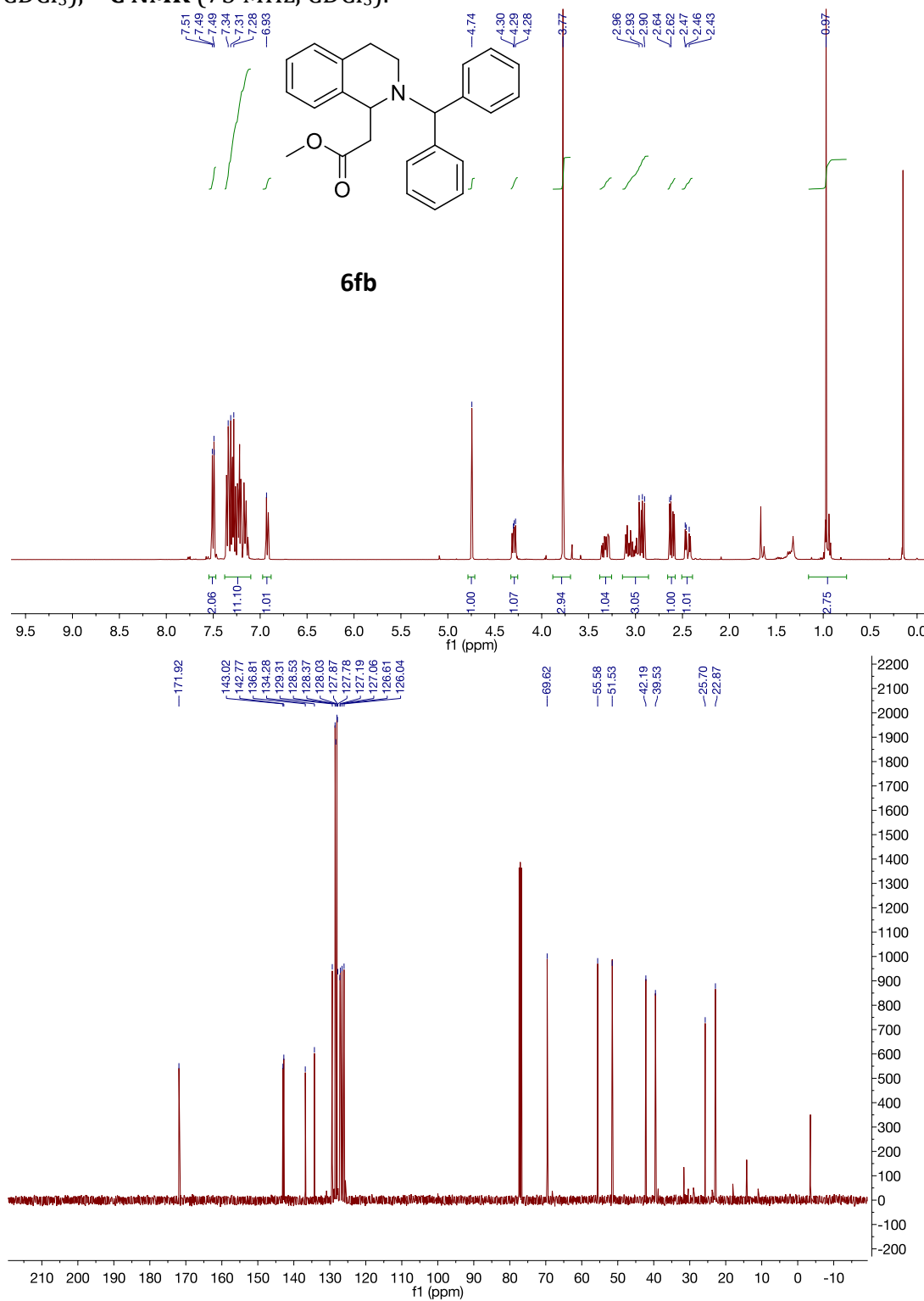
Methyl 2-(2benzyl-6,7-dimethoxy-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (6ea); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



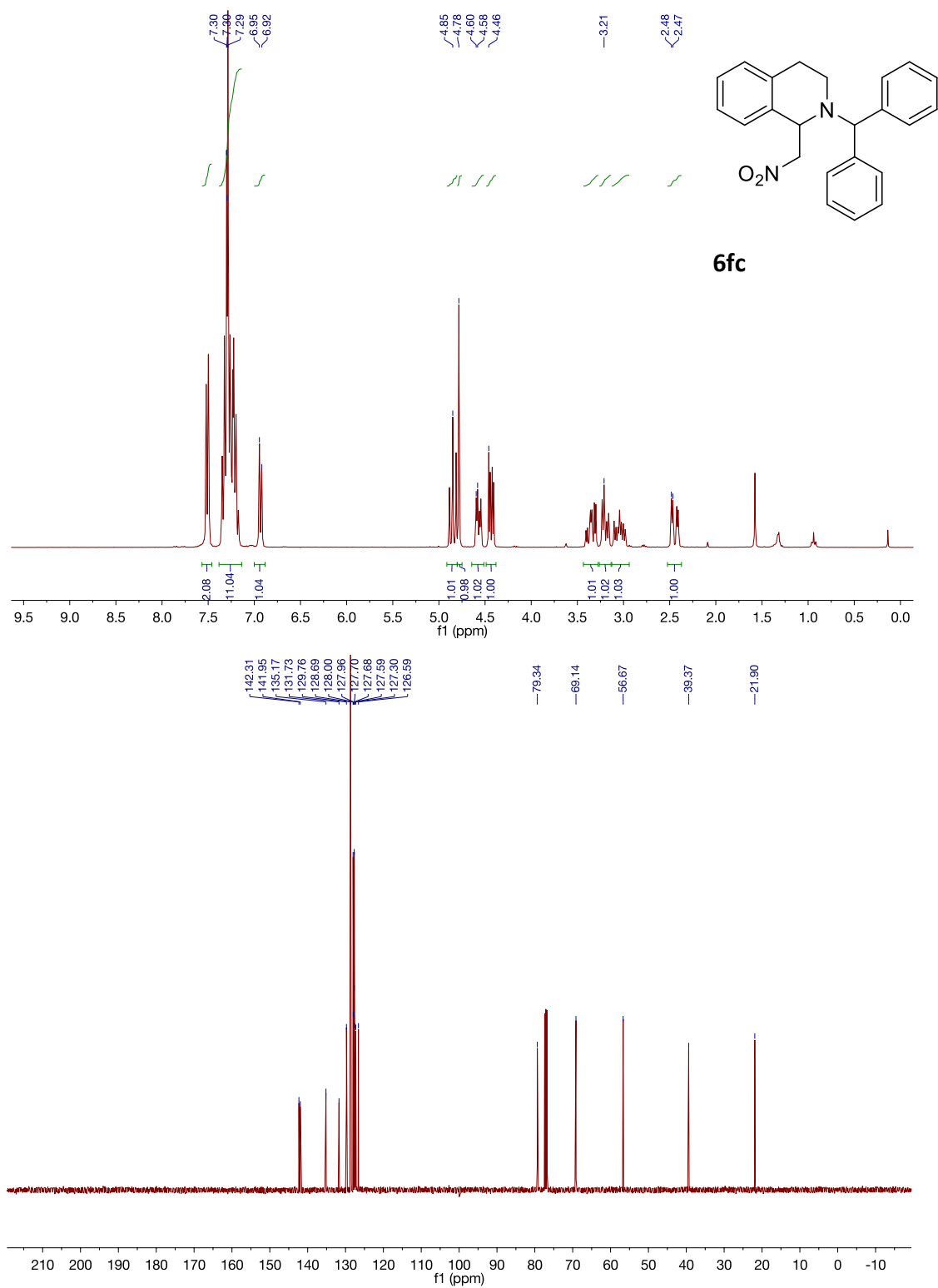
Methyl 2-(2-benzhydryl-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (**6fa**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



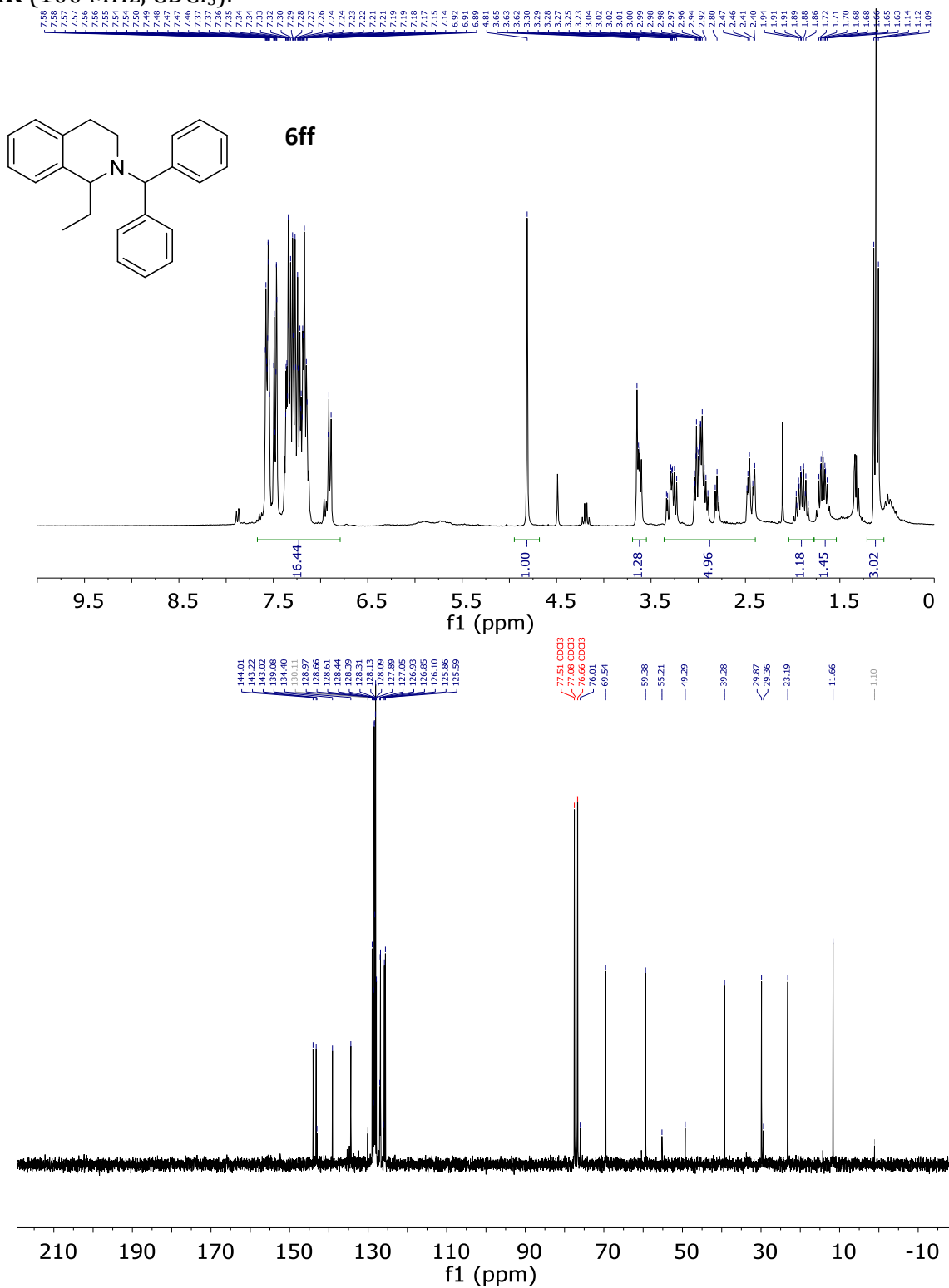
Methyl 2-(2-benzhydryl-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6fb**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (75 MHz, CDCl_3).



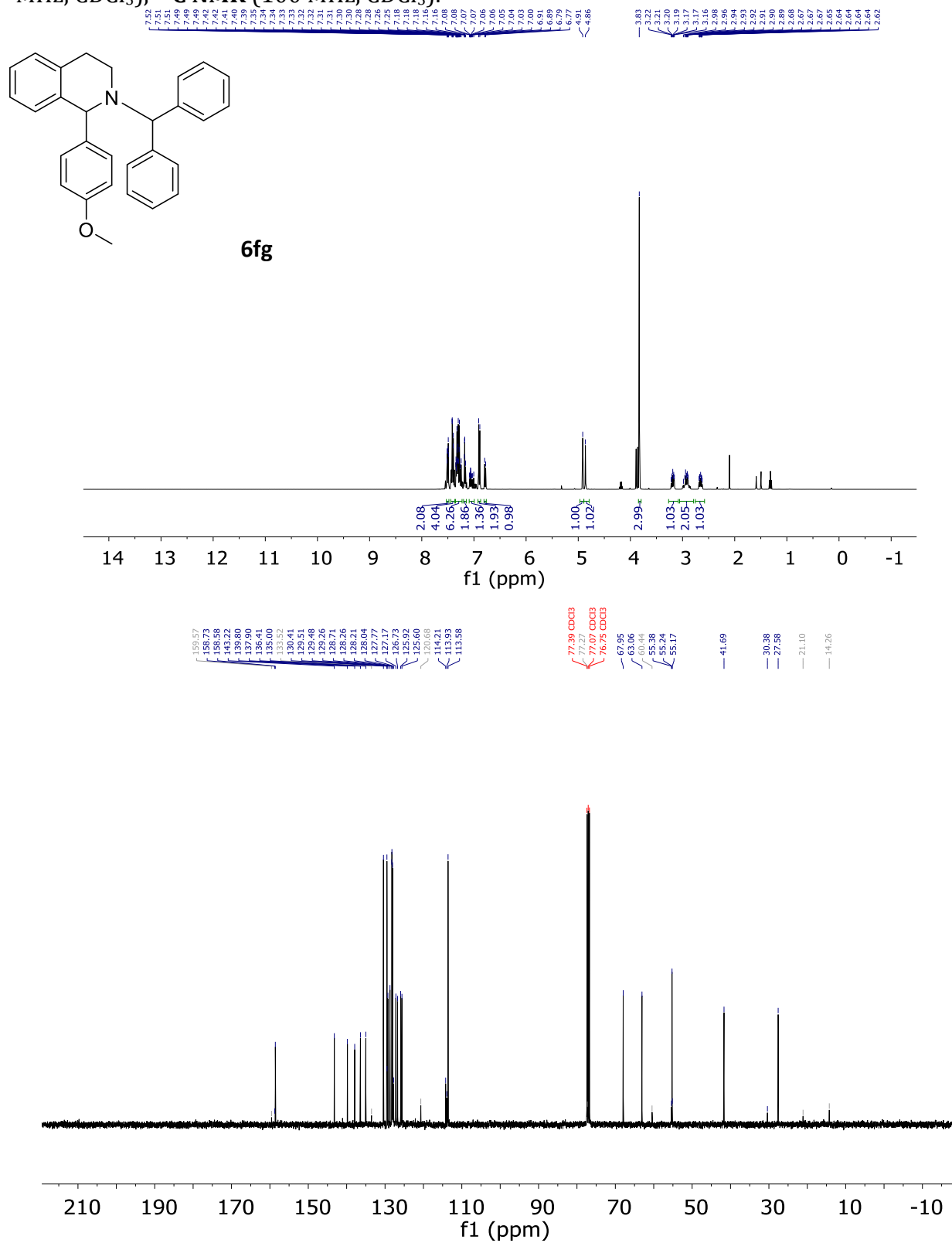
2-benzhydryl-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (**6fc**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



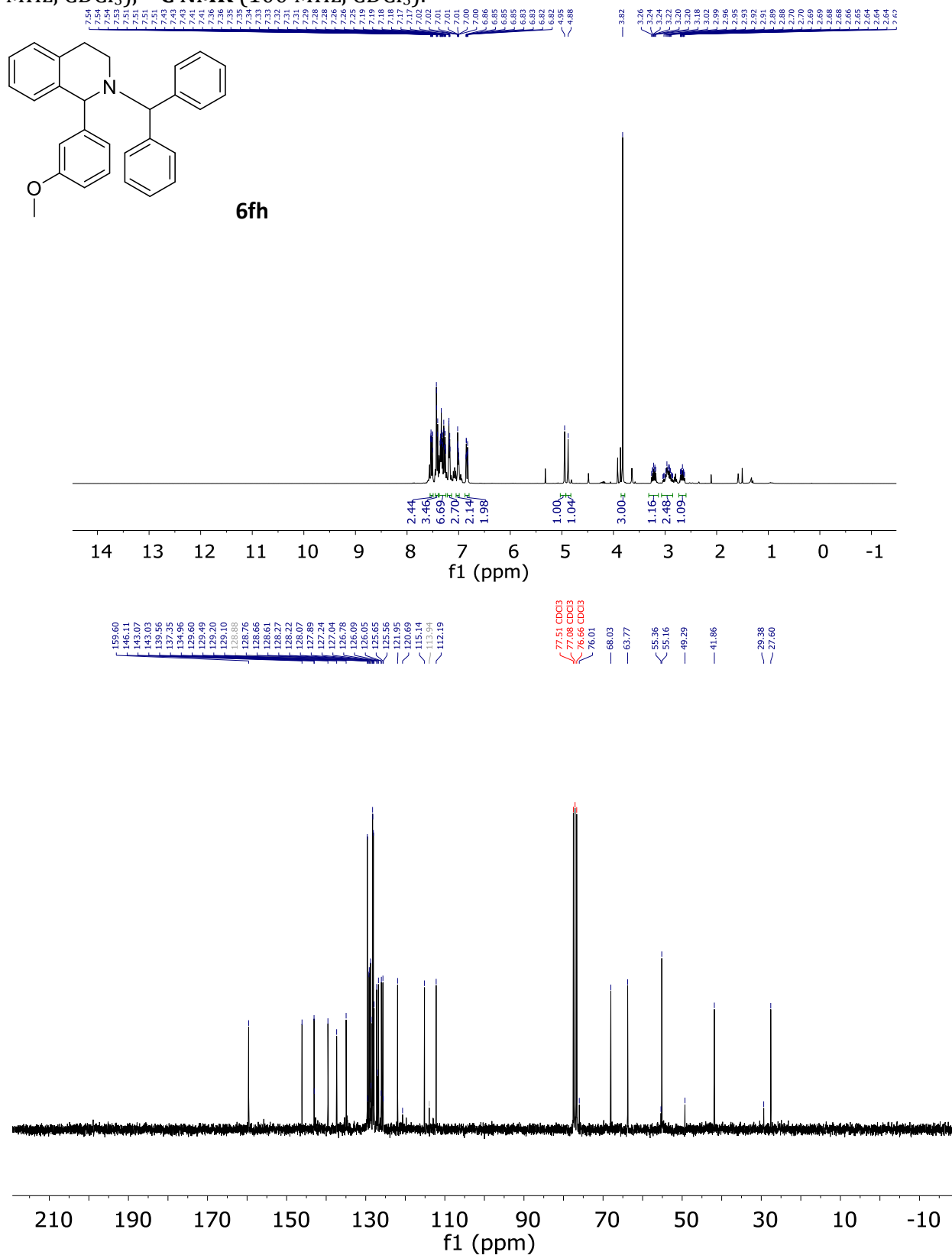
2-benzhydryl-1-ethyl-1,2,3,4-tetrahydroisoquinoline (**6ff**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



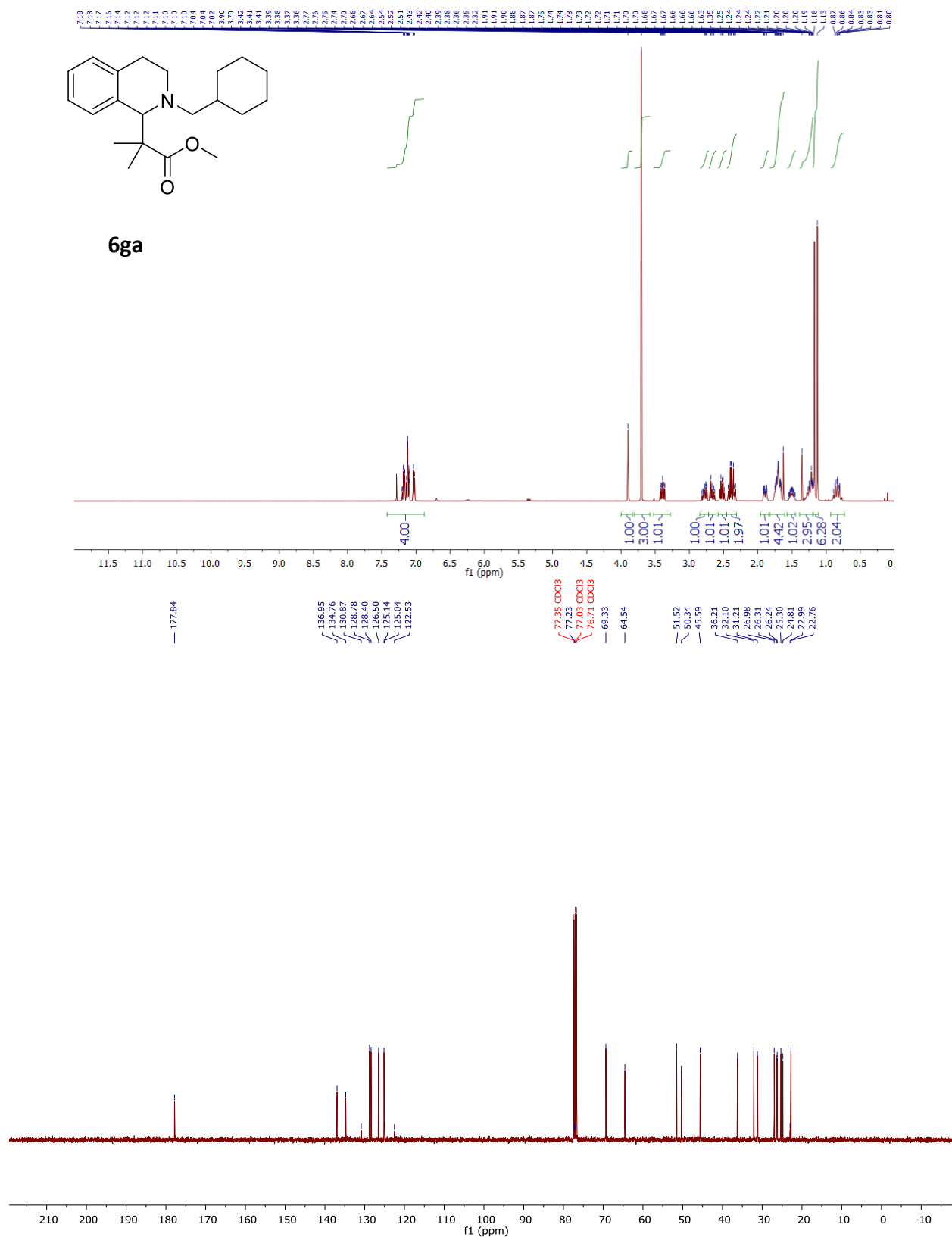
2-benzhydryl-1-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (**6fg**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



2-benzhydryl-1-(3-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (**6fh**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



Methyl 2-(2-(cyclohexylmethyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (**6ga**);
 ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



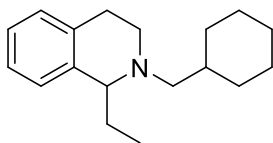
COC(=O)CC1Cc2ccccc2CN1Cc3ccccc3

6gb

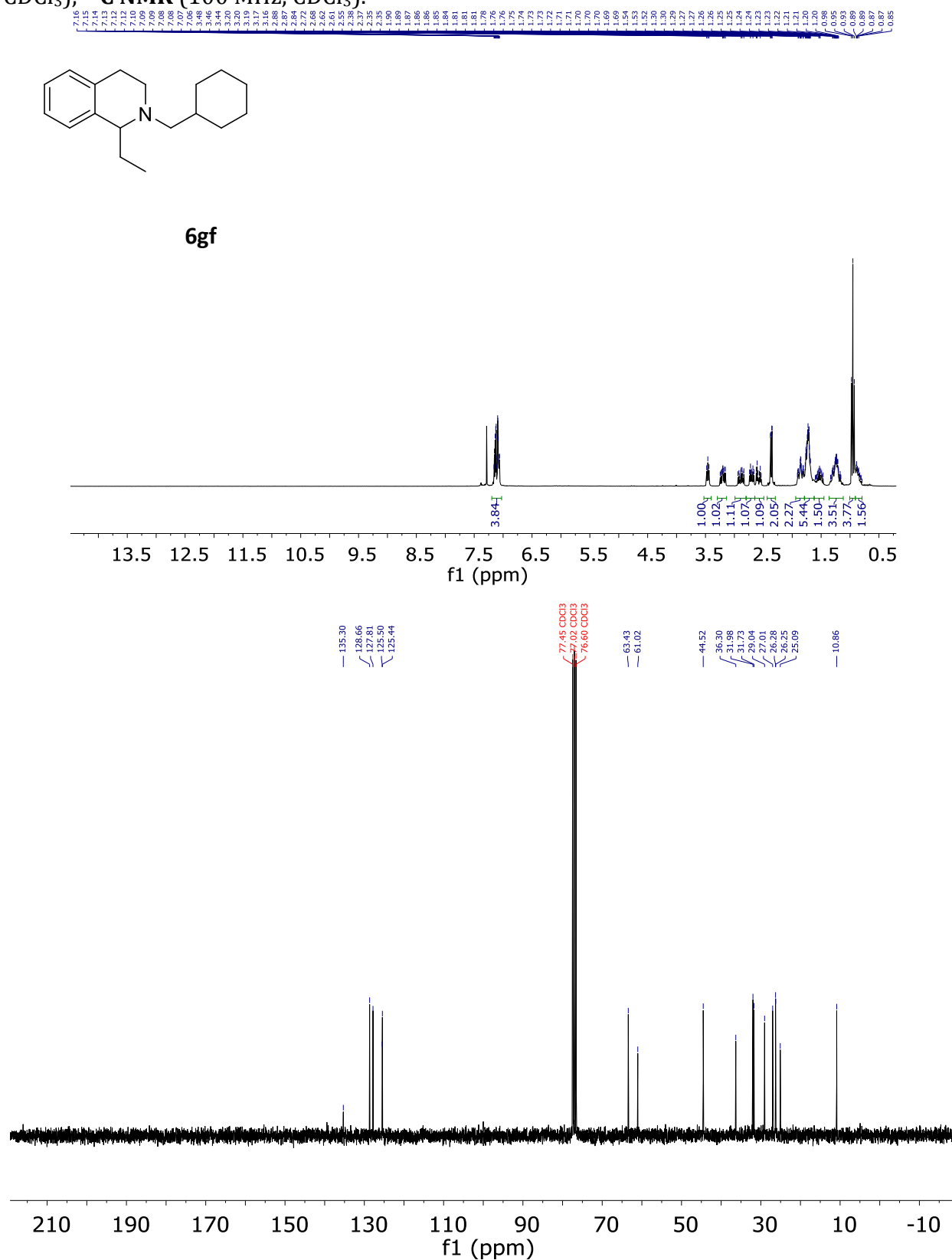
¹H NMR (CDCl₃)

Chemical Shift (ppm)	Integration
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4.38, 4.37, 4.36, 4.35, 4.34, 4.33, 4.32, 4.31, 4.30, 4.29, 4.28, 4.27, 4.26, 4.25, 4.24, 4.23, 4.22, 4.21, 4.20, 4.19, 4.18, 4.17, 4.16, 4.15, 4.14, 4.13, 4.12, 4.11, 4.10, 4.09, 4.08, 4.07, 4.06, 4.05, 4.04, 4.03, 4.02, 4.01, 4.00, 3.99, 3.98, 3.97, 3.96, 3.95, 3.94, 3.93, 3.92, 3.	

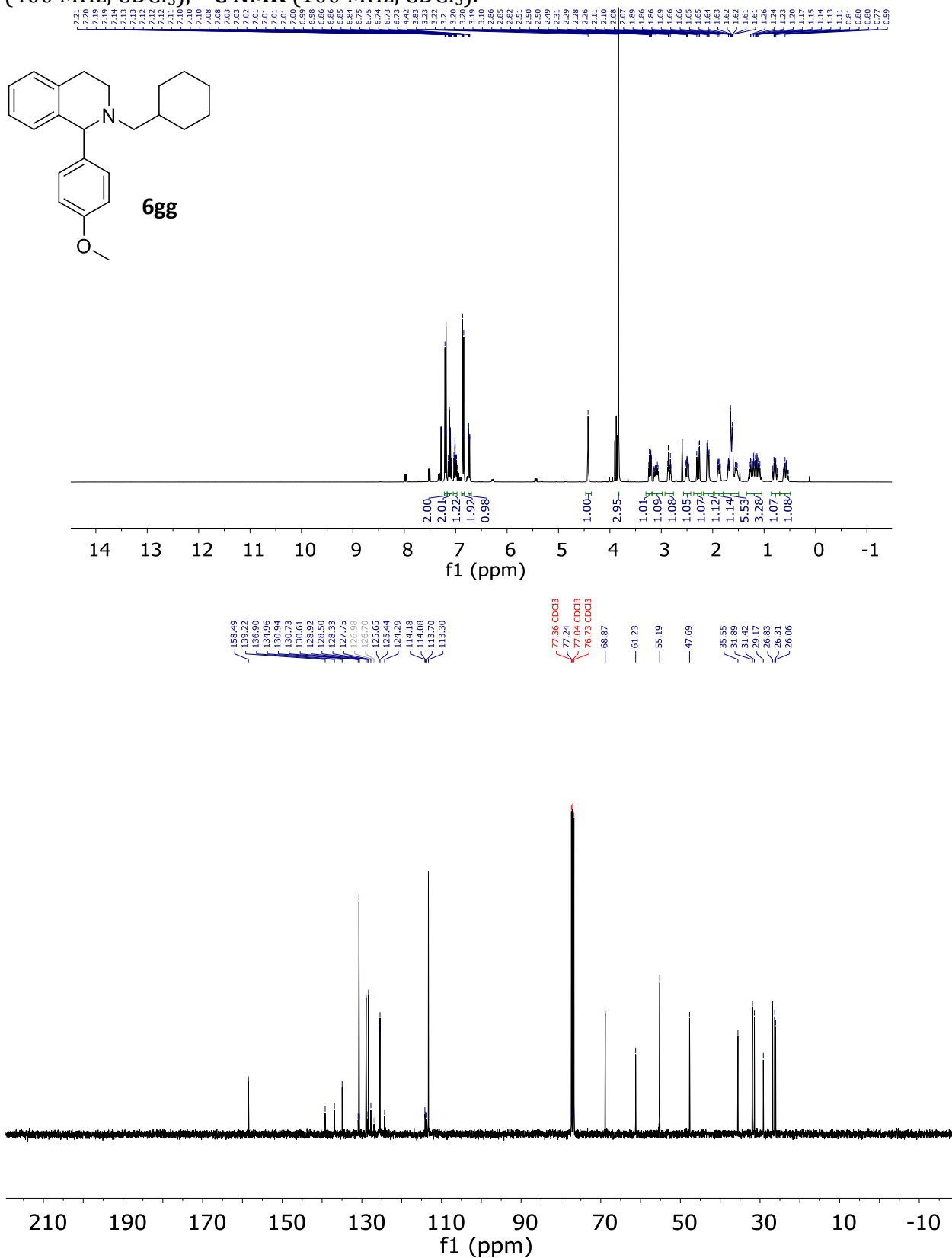
2-(cyclohexylmethyl)-1-ethyl-1,2,3,4-tetrahydroisoquinoline (**6gf**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



6gf



2-(cyclohexylmethyl)-1-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (**6gg**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



COc1ccc(cc1)C2=CC=CC=C2N(C2)CC3CCCCC3

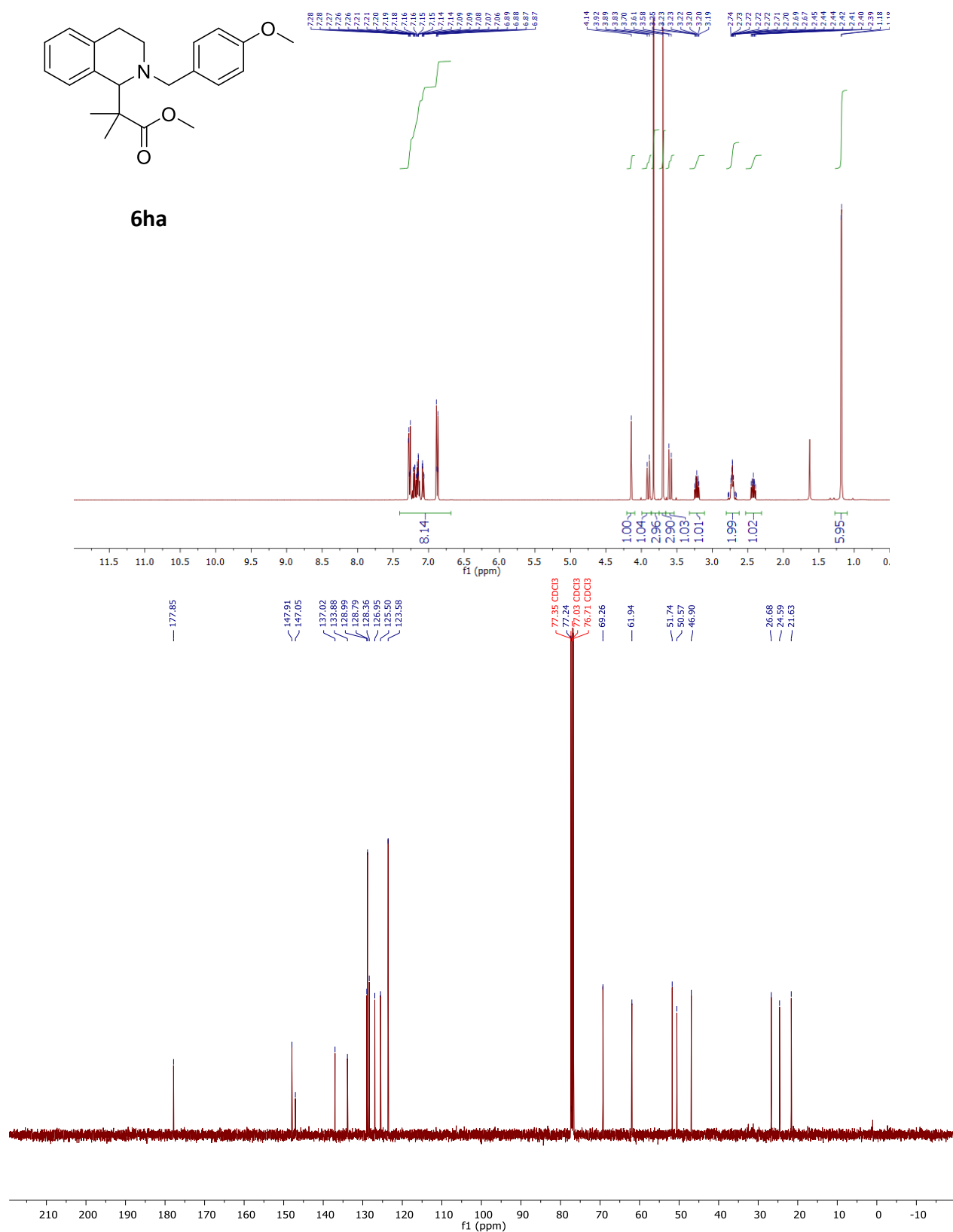
6gh

¹H NMR (400 MHz, CDCl₃)

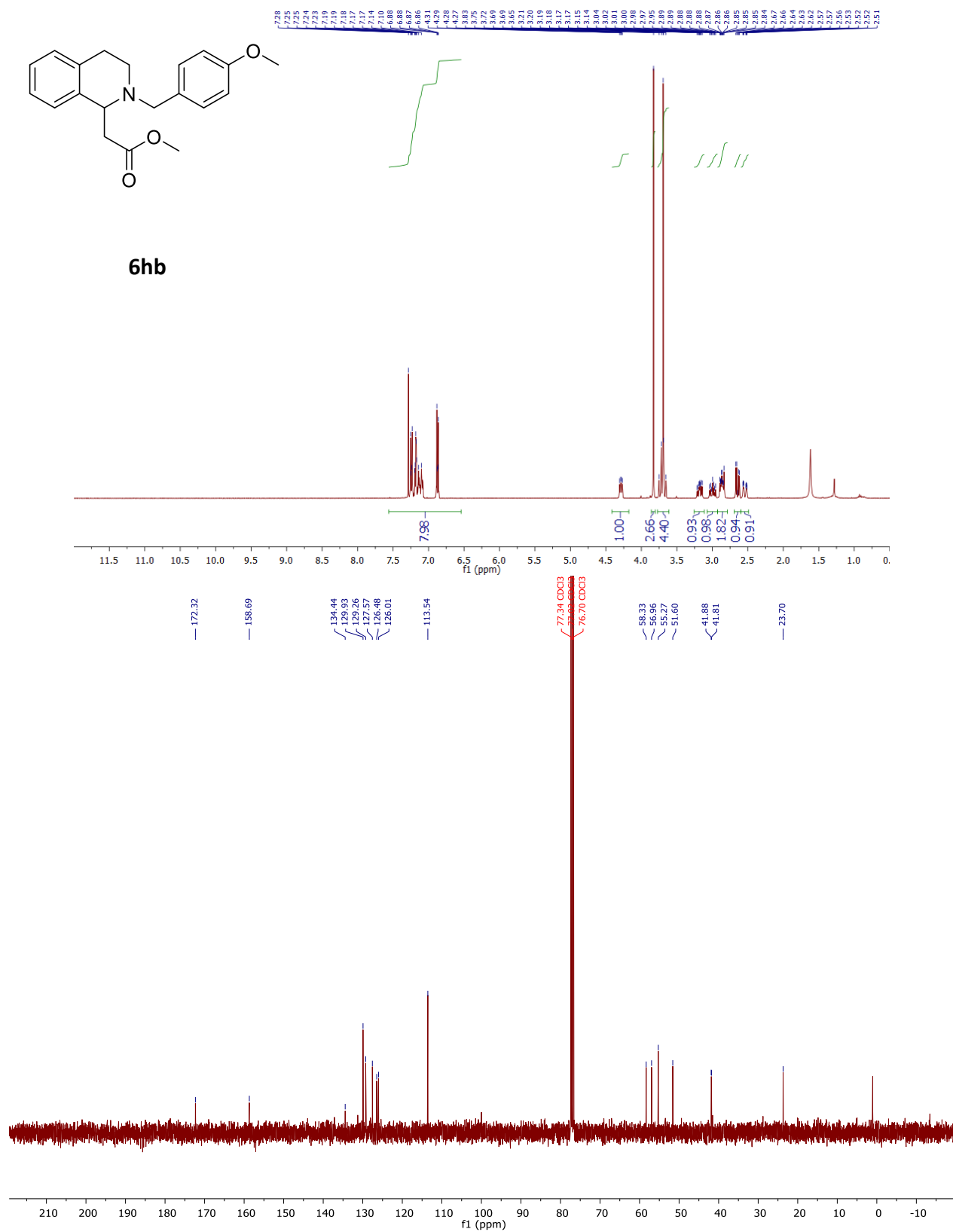
Chemical structure of 6gh: COc1ccc(cc1)C2=CC=CC=C2N(C2)CC3CCCCC3

¹³C NMR (100 MHz, CDCl₃)

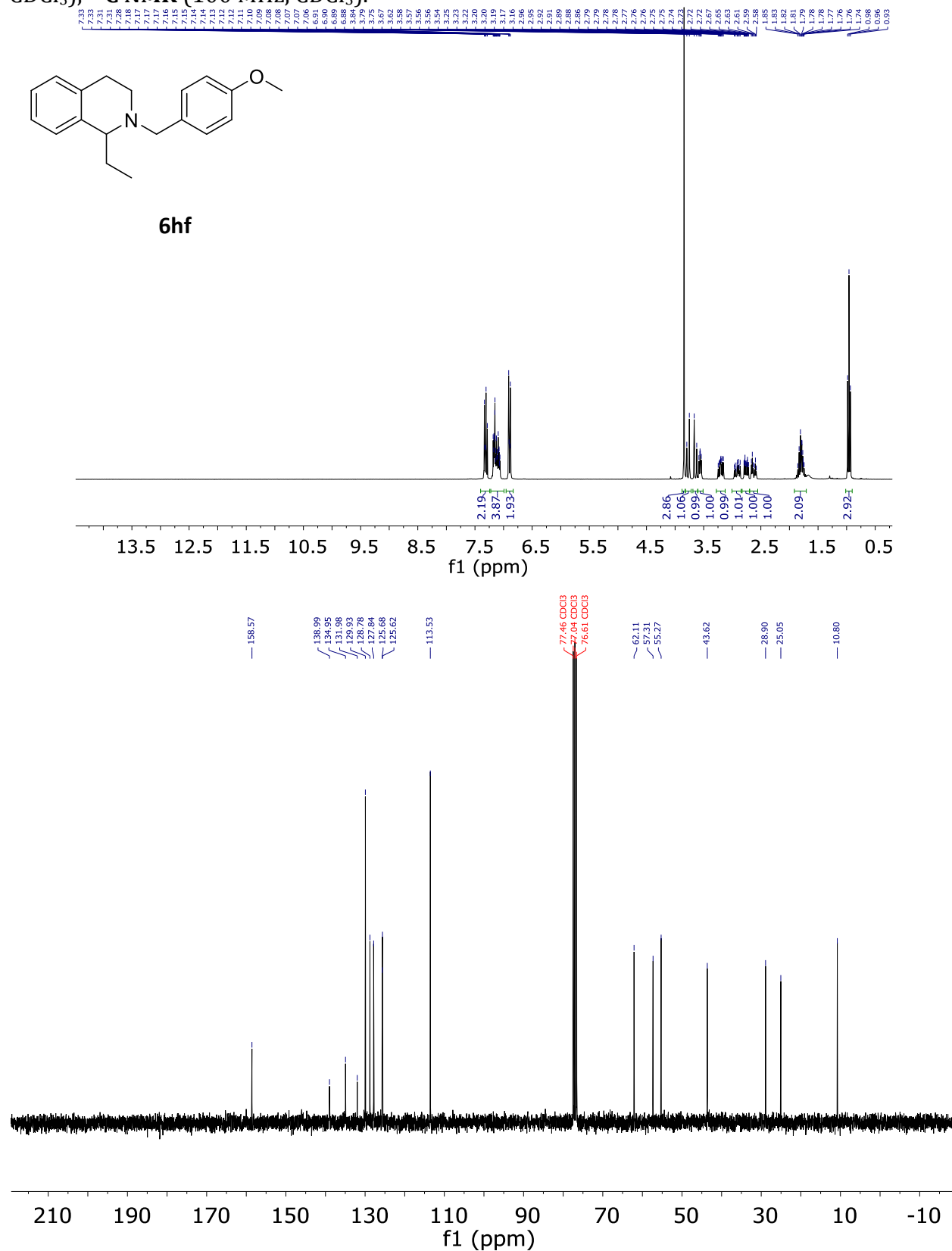
Methyl 2-(2-(4-methoxybenzyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (**6ha**);
 ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



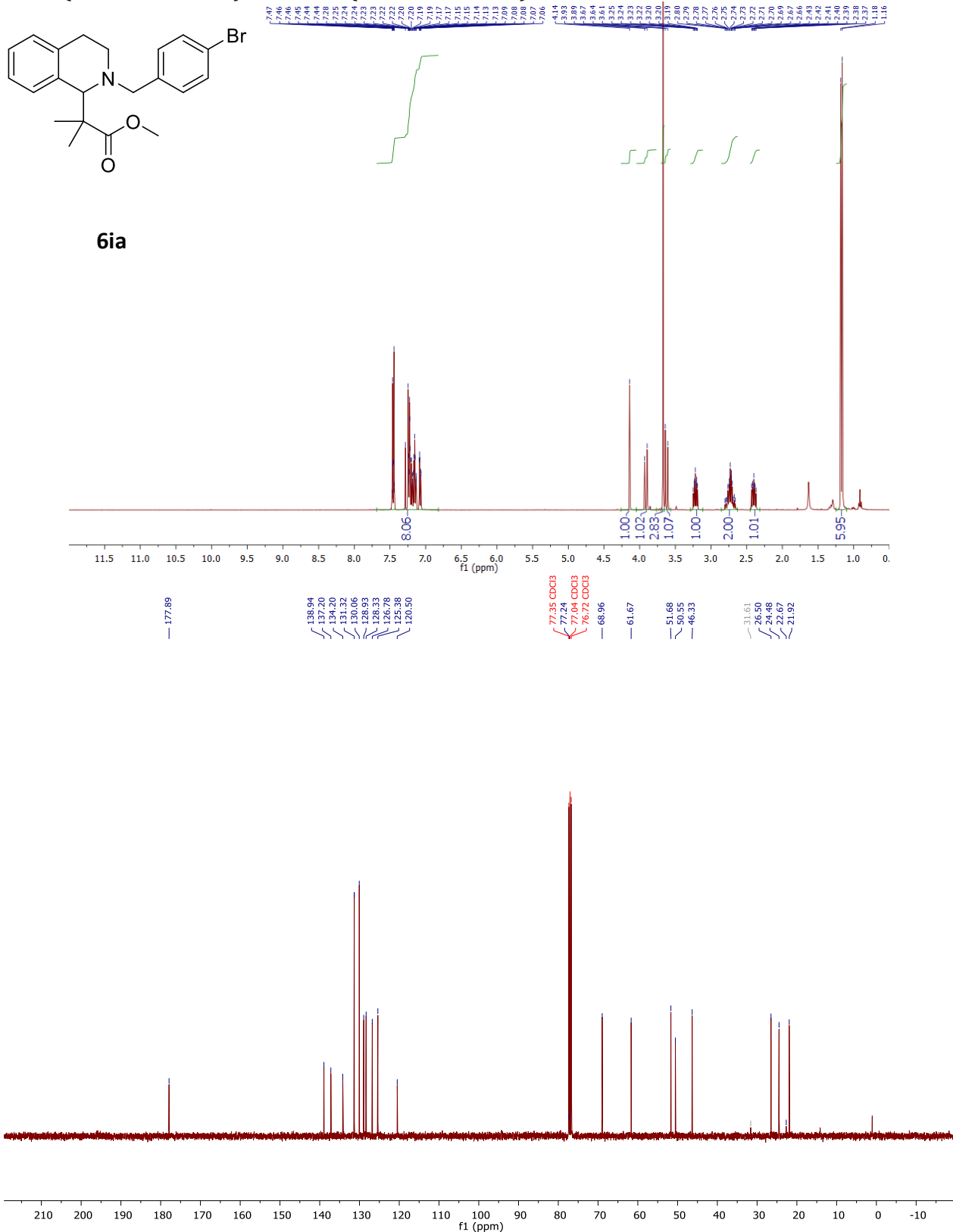
Methyl 2-(2-(4-methoxybenzyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6hb**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



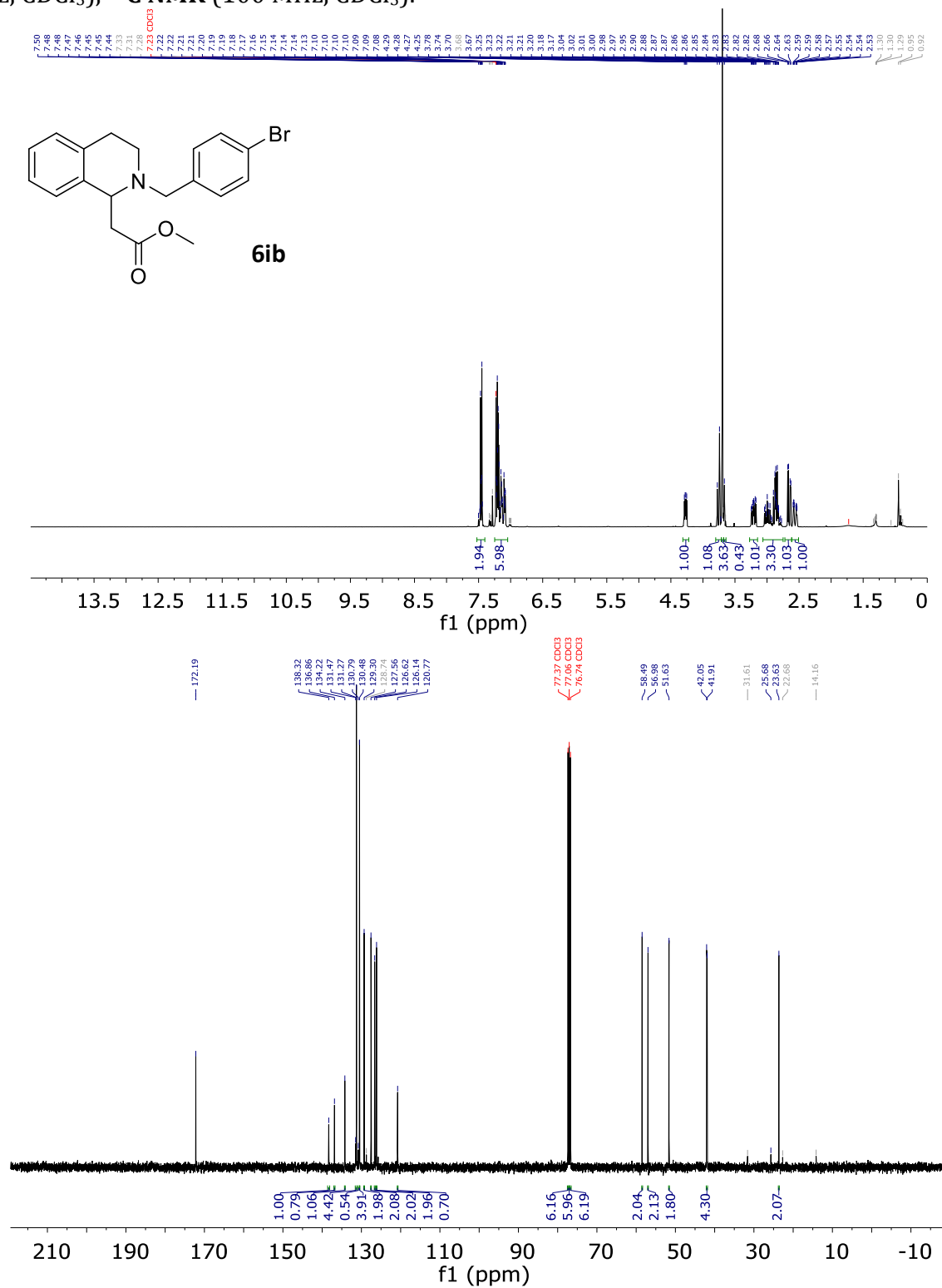
1-ethyl-2-(4-methoxybenzyl)-1,2,3,4-tetrahydroisoquinoline (**6hf**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



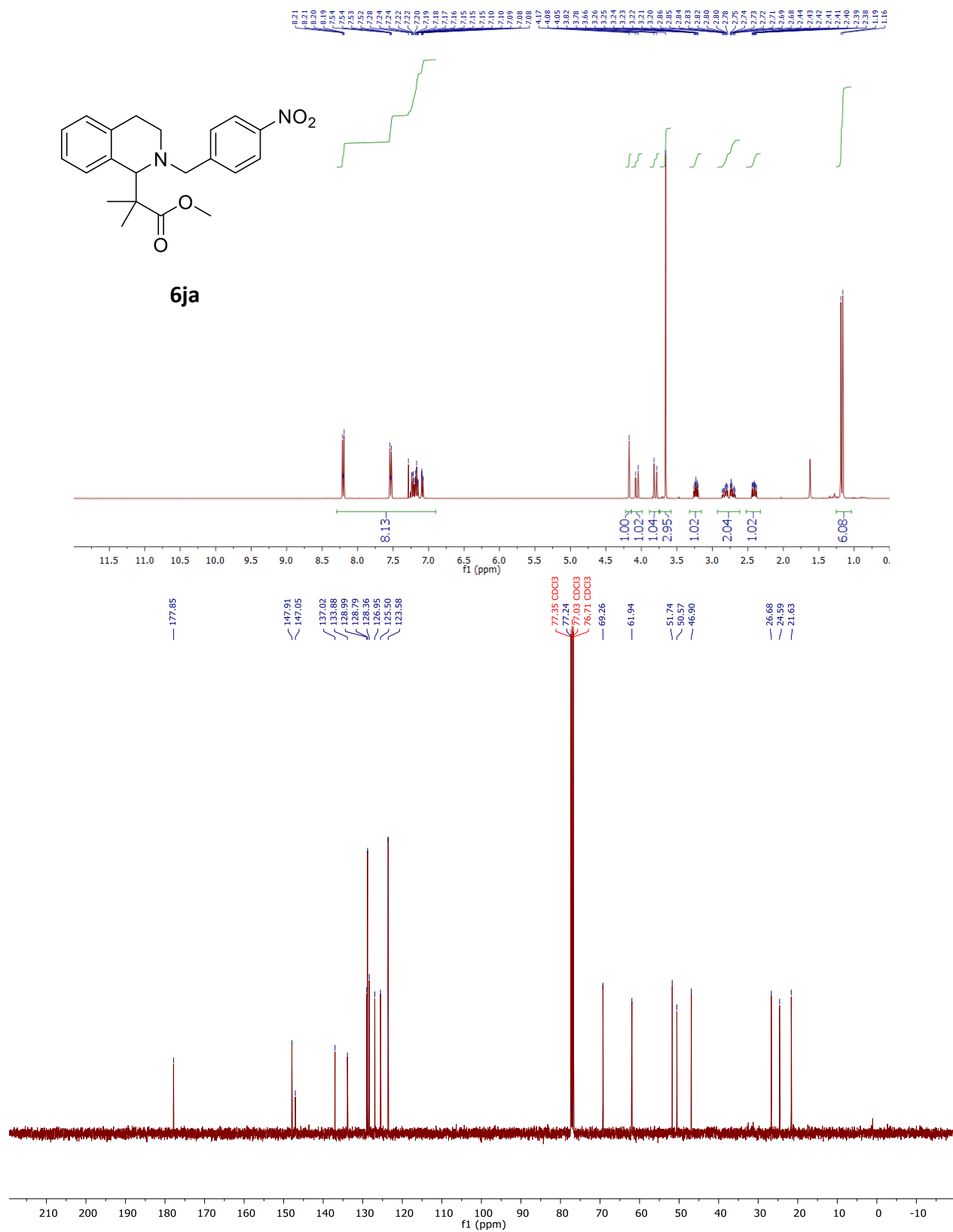
Methyl 2-(2-(4-bromobenzyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (**6ia**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



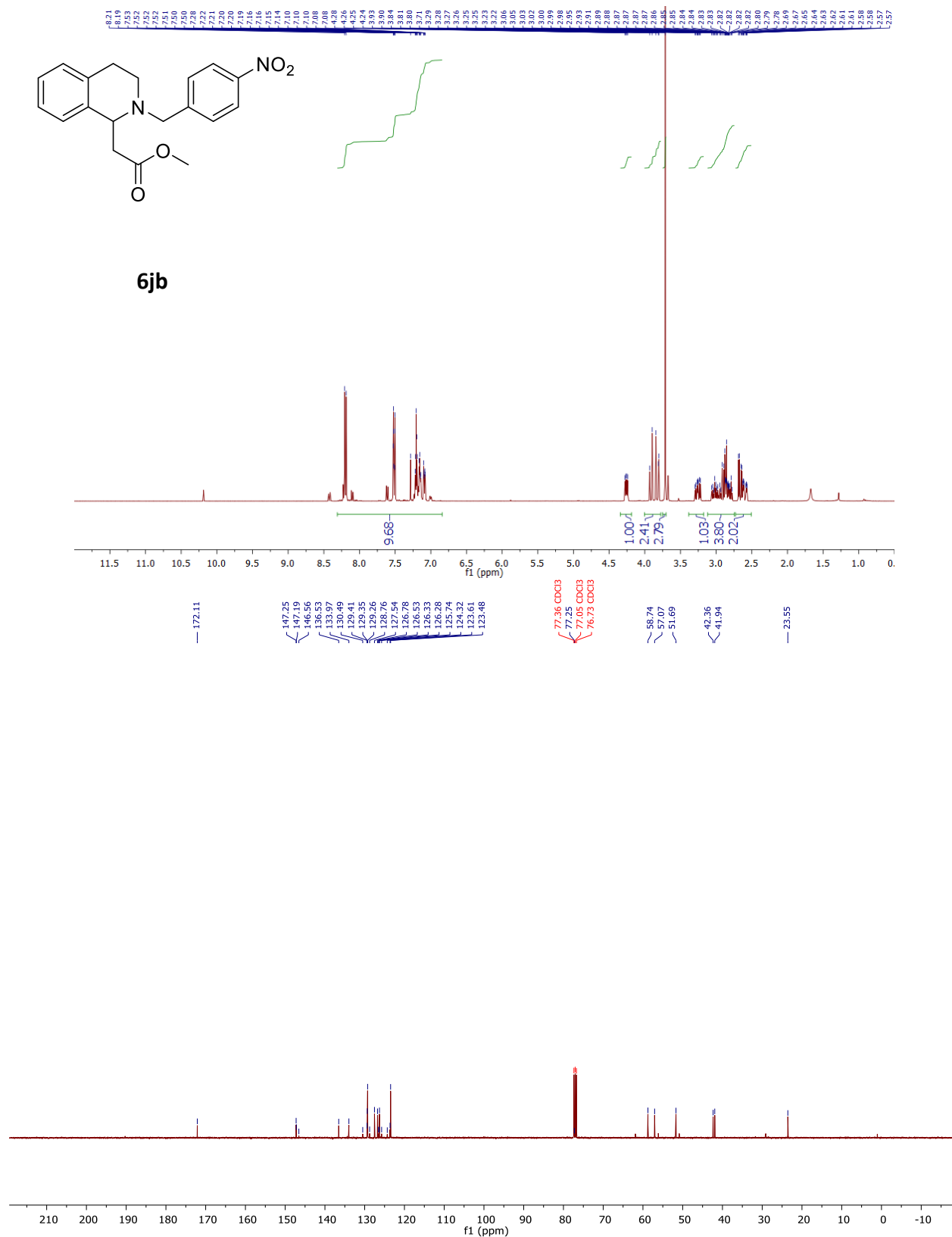
Methyl 2-(2-(4-bromobenzyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6ib**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



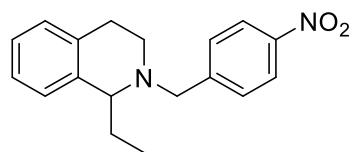
Methyl 2-(2-(4-nitrobenzyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)-2-methylpropanoate (**6ja**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



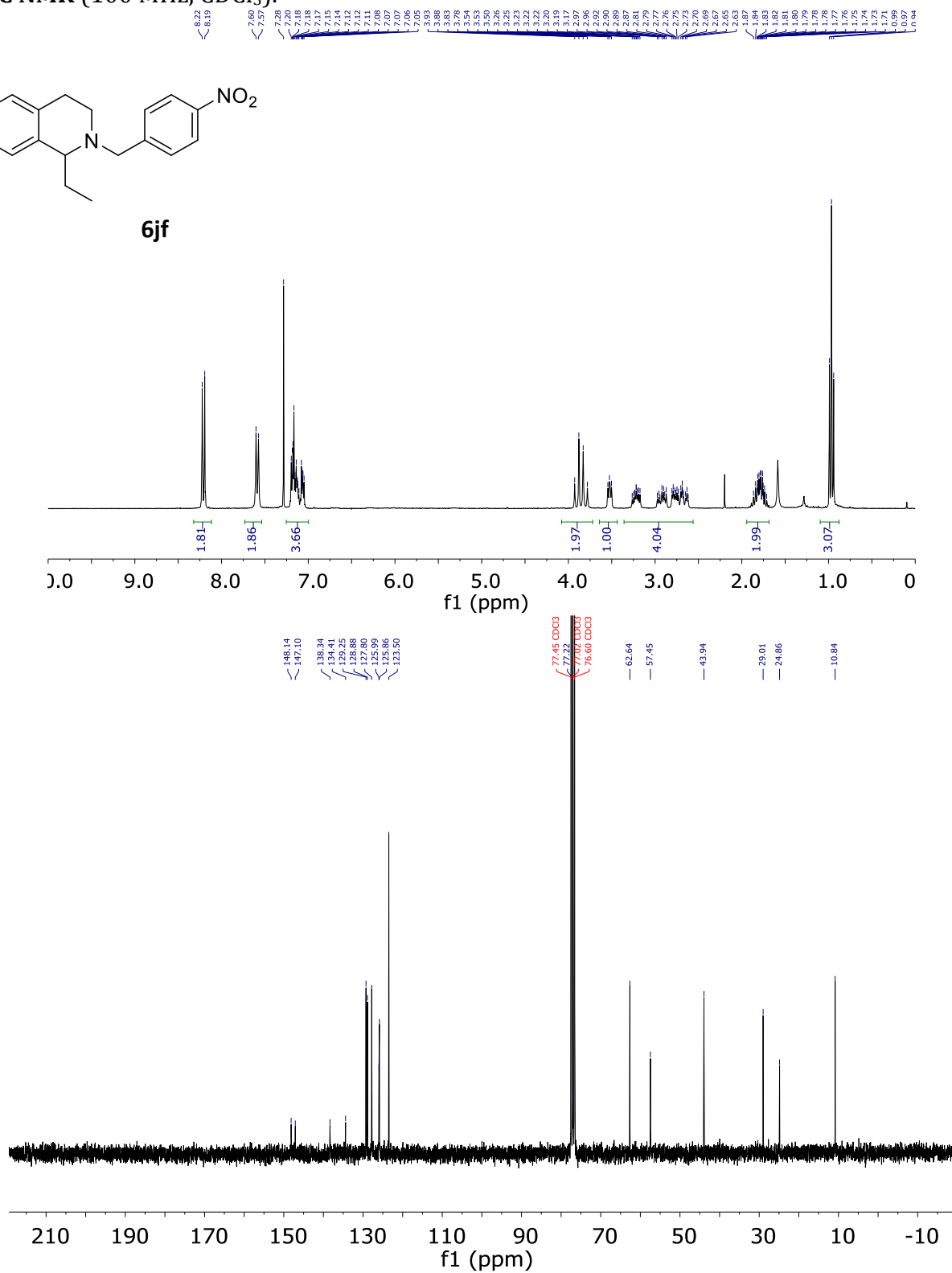
Methyl 2-(2-(4-nitrobenzyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6jb**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



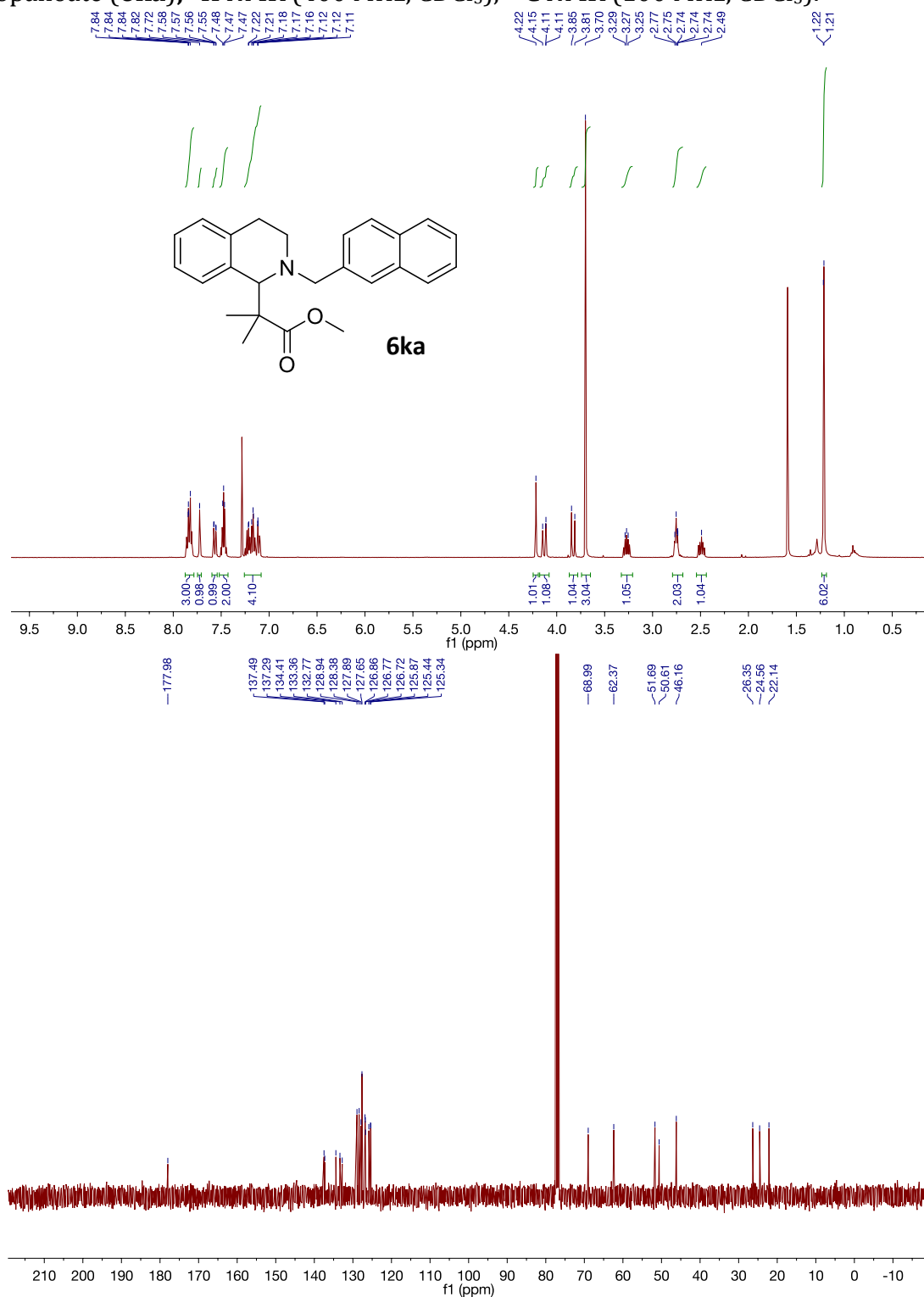
1-ethyl-2-(4-nitrobenzyl)-1,2,3,4-tetrahydroisoquinoline (**6jf**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



6jf



Methyl 2-methyl-2-(2-(naphthalen-2-ylmethyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propanoate (**6ka**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).



Methyl (2-(naphthalen-2-ylmethyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (**6kb**); ^1H NMR (400 MHz, CDCl_3), ^{13}C NMR (100 MHz, CDCl_3).

