Supporting Information

Electrochemically Enabled, Nickel-Catalyzed Dehydroxylative Cross-Coupling of Alcohols with Aryl Halides

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General Information

NMR spectra were recorded on Varian 400 MHz instruments at ambient temperature with $CDCl_3$ as the solvent unless otherwise stated. Chemical shifts are reported in parts per million relative to $CDCl_3$ (^1H, δ 7.26 for $CDCl_3$; ^{13}C, δ 77.16 for $CDCl_3$ unless otherwise stated). Data for ^1H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad), coupling constants (Hz), and integration. High resolution mass spectra (HRMS) were recorded at NIBS Metabolomics Center using an Thermo Scientific Q Exactive HF-X Hybrid Quadrupole-Orbitrap MS. Optical rotations were recorded on an AUTOPOL III digital polarimeter at 589 nm and are recorded as $[\alpha]_D^{20}$ (concentration in grams/100 mL solvent). Chiral HPLC analysis was performed on an Agilent 1220 series. Analytical thin layer chromatography was performed using 0.25 mm silica gel 60-F plates. Column chromatography was performed using 300-400 mesh silica gel. Yields refer to chromatographically and spectroscopically pure materials, unless otherwise stated. All reactions were carried out under an inert argon atmosphere with dry solvents under anhydrous conditions unless otherwise stated. All glassware was dried in a drying oven before using. Anhydrous NMP (*N*-methyl-2-pyrrolidone), DMF (*N*, *N*-dimethylformamide), and DMA (*N*, *N*-dimethylacetamide) were purchased from ACROS or Sigma-Aldrich®. The Peking University X-ray Diffraction Laboratory collected and analyzed all X-ray diffraction data.

General Procedure for Electrochemical Dehydroxylative Arylation (General Procedure A)

Part I. Preparation of $NiBr_2$•2dtbbpy solution (0.02 M based on Ni).

A screw-capped culture tube charged with a magnetic stir bar was moved into a glove box. $NiBr_2$ (21.9 mg, 0.1 mmol), **L1** (53.9 mg, 0.2 mmol), and anhydrous NMP (5 mL) were added. The resulting mixture was stirred at 100 °C until a clear green solution was afforded (usually 2 h). (*Note 1*).

Part II. Electrochemical dehydroxylative cross-coupling. (*Note 2*)

A reaction tube charged with a magnetic stir bar was moved into a glove box. LiBr (17.4 mg, 0.2 mmol, 1.0 equiv), PPh_3 (7.0 or 3.0 equiv), DIPEA (39.7 μL, 0.24 mmol, 1.2 equiv), aryl bromide (0.6 mmol, 3.0 equiv), alcohol (0.2 mmol, 1.0 equiv), and anhydrous NMP (2 mL) were added. The resulting mixture was stirred until all reactants were completely dissolved (*ca.* 5 minutes). The $NiBr_2$•2dtbbpy solution (1 mL) was added before the electrodes were installed (*Note 3*), and the reaction mixture was stirred under a constant current of 4 mA for 14 hours.

The electrodes were rinsed with EtOAc (7 mL). The crude reaction mixture was further diluted with EtOAc (30 mL) (*Note 4*). The resulting mixture was washed with water (40×2 mL) and brine (40 mL) sequentially whereby the aqueous layers were back-extracted with EtOAc (1×40 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. Purification by column chromatography followed by further purification by preparative TLC or preparative HPLC afforded the desired product.

Note 1: Freshly prepared $NiBr_2$•2dtbbpy solution provides the best yield of coupling product.

Note 2: This dehydroxylative arylation is recommended to be operated in a glove box, since the reaction is sensitive to the moisture. Otherwise, the PPh_3 could be oxidized on the anode with H_2O.

Note 3: Low density graphite anode was used.

Note 4: If the product is not sensitive to H_2O_2, a few drops of aq. H_2O_2 could be added after the completion of the reaction to help the removal of the excess PPh_3.

Graphical Supporting Information

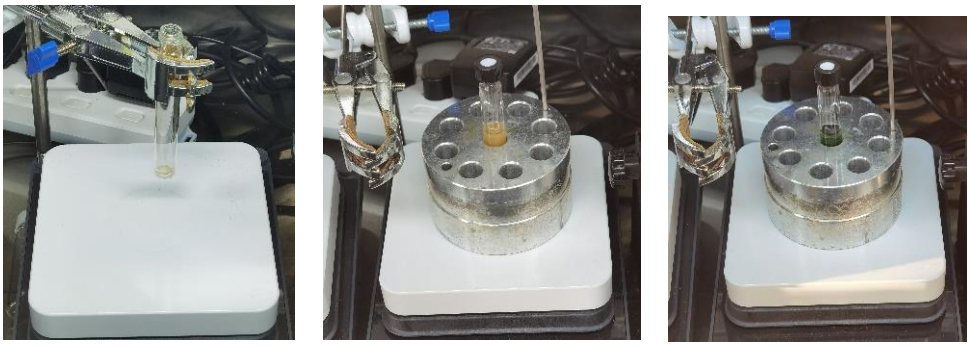


Figure S1. Preparation of $NiBr_2$•2dtbbpy solution: (1) A screw-capped culture tube was charged with $NiBr_2$ and dtbbpy; (2) The suspension of $NiBr_2$ and dtbbpy in NMP was heated to 100 °C; (3) After 2 h stirring, the solution was cooled to room temperature.

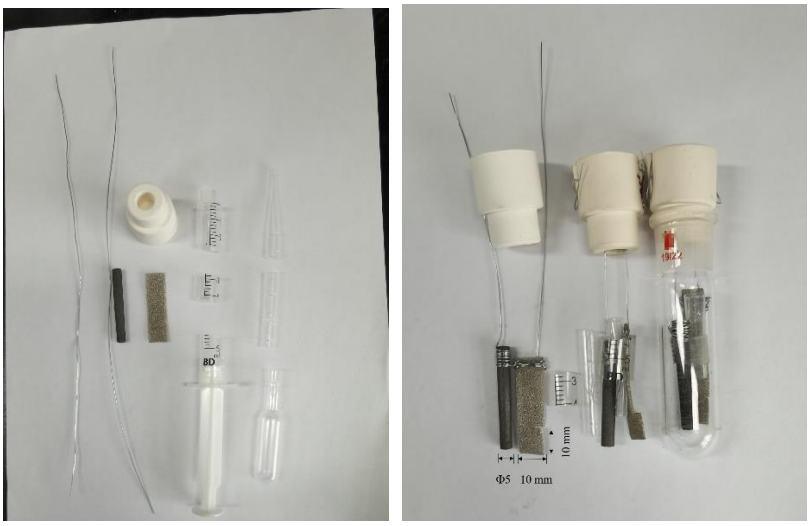


Figure S2. Preparation of the electrode: (1) Materials for making the electrodes (from left to right): iron wire, graphite rod, nickel foam, rubber plug, plastic syringe, and plastic pipes; (2) The assembled electrodes.

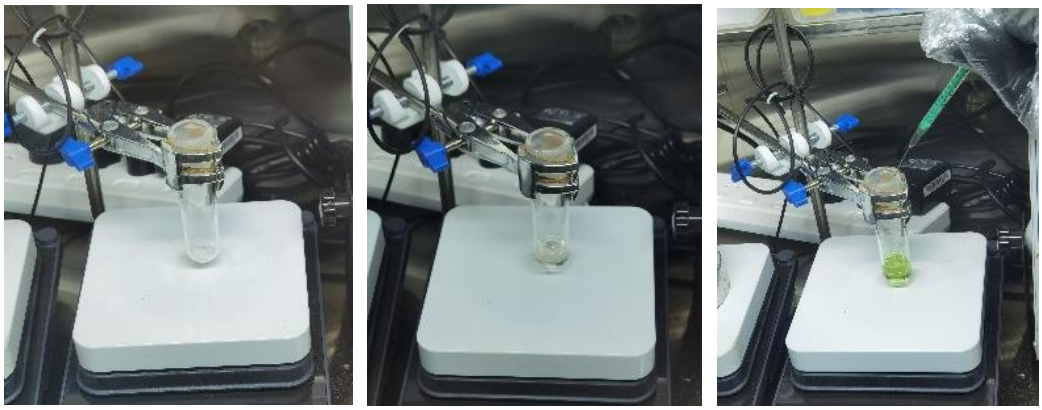


Figure S3. From left to right: (1) A reaction tube was charged with LiBr, PPh_3, DIPEA, PhBr, and alcohol; (2) NMP was added, and the mixture was stirred until all reactants were completely dissolved; (3) The solution of $NiBr_2$•2dtbbpy was added.

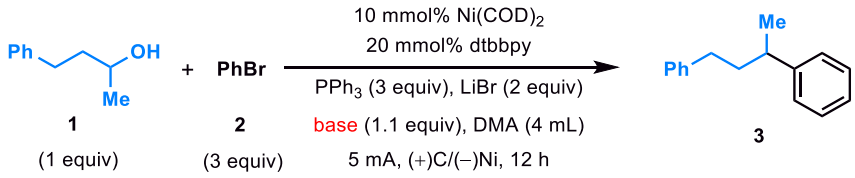
Figure S4. From left to right: (1) Starting the reaction on an electrochemical equipment with a constant current of 4 mA (10 mm electrodes were immerged into the reaction solution); (2) After 2 min stirring, the reaction mixture turned black; (3) After 10 h stirring.

Figure S5. From left to right: (1) The reaction mixture; (2) Diluted with EtOAc (adding the aq. H_2O_2 was optional); (3) Extraction.; (4) TLC: **Lane a**: the reaction mixture; **Lane b**: the reaction mixture after treatment with H_2O_2.

Optimization Details

All reactions were conducted at 0.2 mmol sacle.

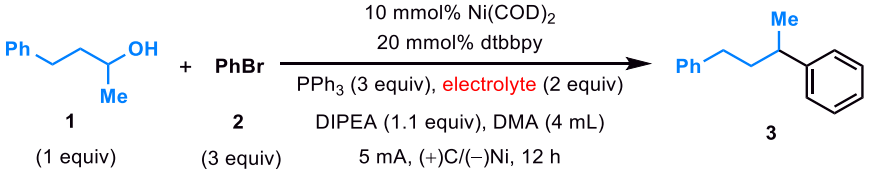
Table S1. Screening of the bases.



Entry	Base	Yield (%)[a]
1	w/o base	44
2	Li_2CO_3	45
3	Na_2CO_3	34
4	K_2CO_3	23
5	Cs_2CO_3	27
6	$NaHCO_3$	12
7	K_3PO_4	0
8	K_2HPO_4	0
9	DBU	40
10	2,6-lutidine	42
11	2,6-di-*tert*-butyl pyridine	46
12	2,4,6-collidine	45
13	DMAP	50
14	*N, N'*-tetramethylguanidine	51
15	1,4-diaza [2.2.2] bicyclooctane	46
16	TEA	52
17	**DIPEA**	**58**

[a]Yields were determined by GC-MS with dodecane as the internal standard.

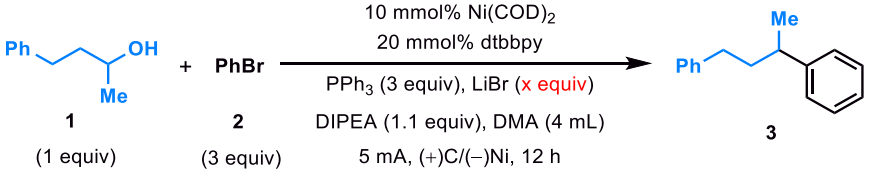
Table S2. Screening of the electrolytes.



Entry	Electrolyte	Yield (%)[a]
1	Et_4NBr	51
2	LiCl	0
3	**LiBr**	**55**
4	LiI	55
5	$LiClO_4$	50
6	$LiBF_4$	36
7	$LiPF_6$	49

[a]Yields were determined by GC-MS with dodecane as the internal standard.

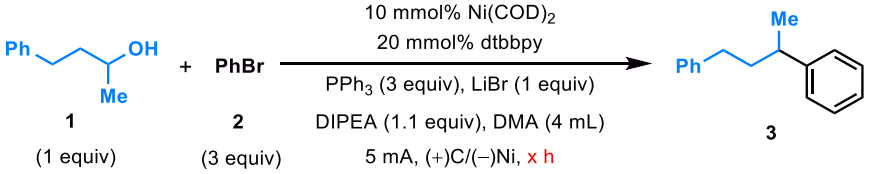
Table S3. Optimization of the equivalent of electrolytes.



Entry	Equivalent of Electrolyte	Yield (%)[a]
1	0.5	59
2	**1.0**	**64**
3	2.0	60
4	3.0	63

[a]Yields were determined by GC-MS with dodecane as the internal standard.

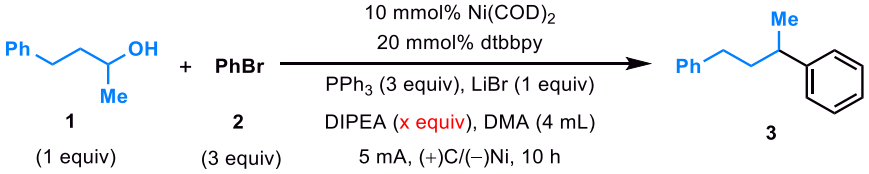
Table S4. Optimization of the reaction time.



Entry	Time (h)	Yield (%)[a]
1	2.5 (≈2 F/mmol)	12
2	4.0 (≈4 F/mmol)	35
3	6.5 (≈6 F/mmol)	51
4	**10.5 (≈10 F/mmol)**	**60**
5	12.0 (≈11 F/mmol)	57
6	21.5 (≈20 F/mmol)	59

[a]Yields were determined by GC-MS with dodecane as the internal standard.

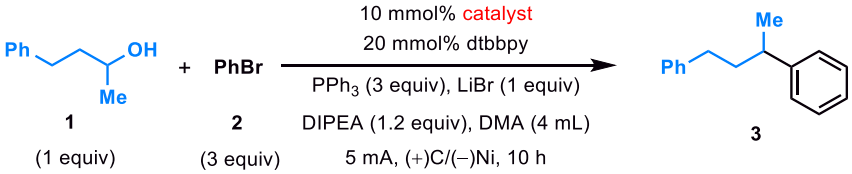
Table S5. Optimization of the equivalent of bases.



Entry	Equivalent of Base	Yield (%)[a]
1	0.0	38
2	1.0	56
3	**1.2**	**60**
4	2.0	62

[a]Yields were determined by GC-MS with dodecane as the internal standard.

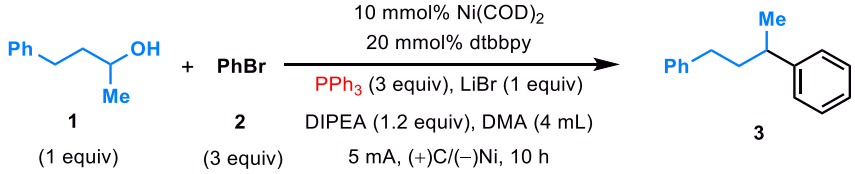
Table S6. Screening of the catalysts.



Entry	Catalyst	Yield (%)[a]
1	$Ni(COD)_2$	57
2	$Ni(acac)_2$	49
3	$Co(acac)_2$	0
4	$Cu(acac)_2$	0
5	$Fe(acac)_3$	0
6	$NiCl_2$	50
7	**$NiBr_2$**	**58**
8	$Ni(NO_3)_2$	38
9	$Ni(OAc)_2$	35
10	$Ni(ClO_4)_2$	47

[a]Yields were determined by GC-MS with dodecane as the internal standard.

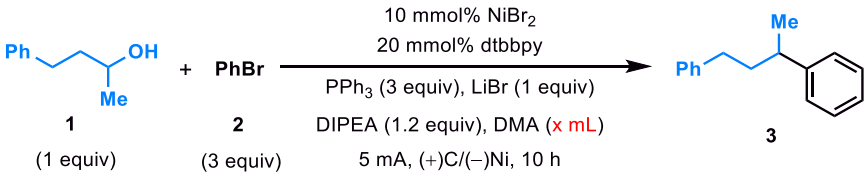
Table S7. Screening of PPh_3 analogs.



Entry	PPh_3 Analogs	Yield (%)[a]
1	**PPh_3**	**58**
2	$P(OPh)_3$	0
3	$P(OEt)_3$	0
4	nBu$_3$P	0
5	**tris(*p*-tolyl)phosphine**	**62**
6	tris(4-methoxyphenyl)phosphine (**4**)	44
7	tris(4-fluorophenyl)phosphine (**5**)	48

[a]Yields were determined by GC-MS with dodecane as the internal standard.

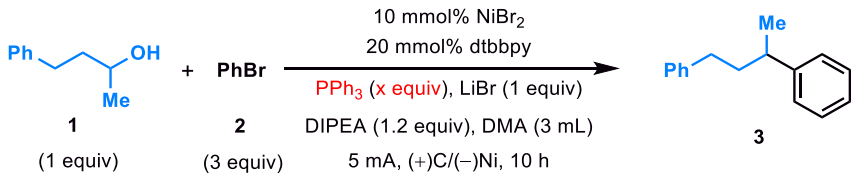
Table S8. Optimization of the reaction concentration.



Entry	Volume of Solvent (concentration of 1)	Yield (%)[a]
1	**3.0 mL (67 mM)**	**64**
2	3.5 mL (57 mM)	61
3	4.0 mL (50 mM)	60
4	4.5 mL (44 mM)	59
5	5.0 mL (40 mM)	58

[a]Yields were determined by GC-MS with dodecane as the internal standard.

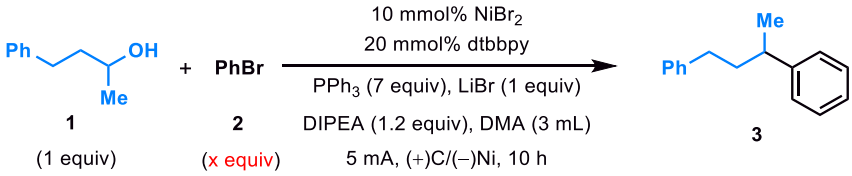
Table S9. Optimization of the equivalent of PPh_3.



Entry	Equivalent of PPh_3	Yield 1(%)[a, b]	Yield 2(%)[a, c]
1	2.0	-	48
2	3.0	63	57
3	4.0	76	67
4	5.0	80	67
5	6.0	81	72
6	**7.0**	**84**	81
7	8.0	82	76
8	9.0	76	-

[a]Yields were determined by GC-MS with dodecane as the internal standard;
[b]PPh_3 was used;
[c]tris(*p*-tolyl)phosphine was used.

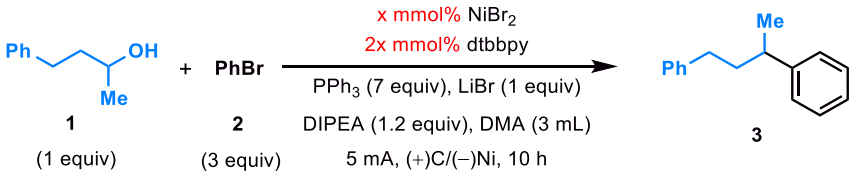
Table S10. Optimization of the equivalent of PhBr.



Entry	Equivalent of PhBr	Yield (%)[a]
1	1.0	75
2	2.0	81
3	**3.0**	**82**
4	4.0	81
5	5.0	82

[a]Yields were determined by GC-MS with dodecane as the internal standard.

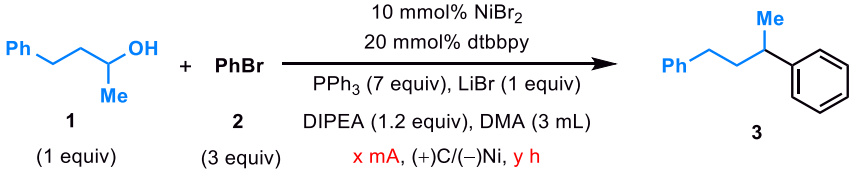
Table S11. Optimization of the equivalent of $NiBr_2$•2dtbbpy.



Entry	Equivalent of $NiBr_2$•2dtbbpy	Yield (%)[a]
1	3%	38
2	5%	67
3	7%	79
4	**10%**	**81**
5	15%	76

[a]Yields were determined by GC-MS with dodecane as the internal standard.

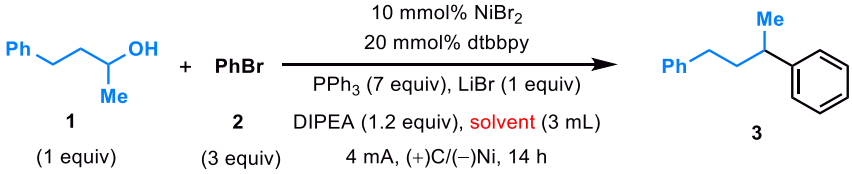
Table S12. Optimization of the current.



Entry	Current (mA)	Yield (%)[a]
1	3(18.5 h)	75
2	**4 (14.0 h)**	**89**
3	5 (10.5 h)	81
4	6 (9.0 h)	68
5	7 (8.0 h)	74
6	8 (7.0 h)	79
7	9 (6.5 h)	71
8	10 (5.5 h)	36

[a]Yields were determined by GC-MS with dodecane as the internal standard.

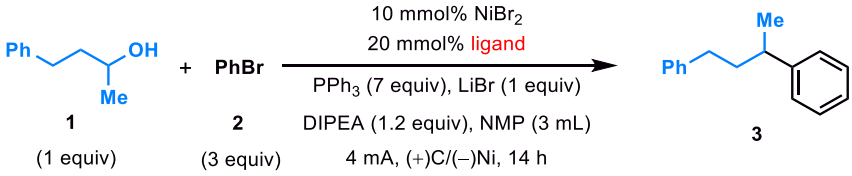
Table S13. Screening of the solvents.



Entry	Solvent	Yield (%)[a]
1	DMA	89
2	DMF	36
3	MeCN	33
4	DMSO	0
5	THF	0
6	**NMP**	**92**

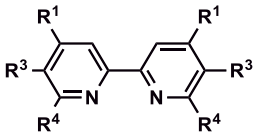
[a]Yields were determined by GC-MS with dodecane as the internal standard.

Table S14. Screening of the ligands.

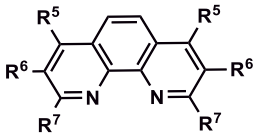


Entry	Ligand	Yield (%)[a]	Entry	Ligand	Yield (%)[a]
1	**L1**	**92**	8	**L8**	58
2	**L2**	80	9	**L9**	0
3	**L3**	88	10	**L10**	0
4	**L4**	86	11	**L11**	44
5	**L5**	76	12	**L12**	0
6	**L6**	0	13	**L13**	0
7	**L7**	66	14	**L14**	5

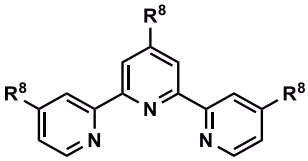
[a]Yields were determined by GC-MS with dodecane as the internal standard.



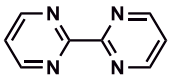
L1: R^1= *t*Bu, R^3= H, R^4= H
L2: R^1= H, R^3= H, R^4= H
L3: R^1= OMe, R^3= H, R^4= H
L4: R^1= Me, R^3= H, R^4= H
L5: R^1= H, R^3= Me, R^4= H
L6: R^1= H, R^3= H, R^4= Me



L7: R^5= H, R^6= H, R^7= H
L8: R^5= Ph, R^6= H, R^7= H
L9: R^5= H, R^6= H, R^7= Me
L10: R^5= Ph, R^6= H, R^7= Me
L11: R^5= Me, R^6= Me, R^7= H

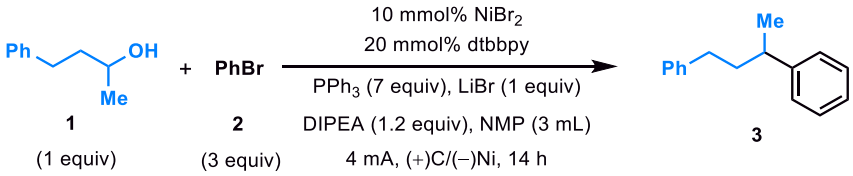


L12: R^8= H; **L13**: R^8= *t*Bu



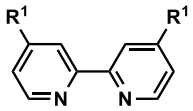
L14

Table S15. Other conditions.

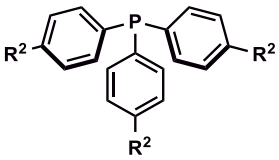


Entry	condition	Yield (%)[a]
1	above	92
2	no electricity	0
3	w/o $NiBr_2$	0
4	w/o **L1**	0
5	w/o PPh_3	0
6	RVC instead of graphite	14
7	2 mA, 28 h	94
8	6 mA, 9 h	84
9	$LiClO_4$ instead of LiBr	86
10	Et_4NBr instead of LiBr	84
11	$Ni(COD)_2$ instead of $NiBr_2$	85
12	**L2** instead of **L1**	80
13	**L3** instead of **L1**	88
14	10 mmol% **L1**	80
15	w/o DIPEA	47
16	PPh_3 (3 equiv)	70
17	**4** instead of PPh_3	78
18	**5** instead of PPh_3	57
19	DMA instead of NMP	89
20	PhBr (2 equiv)	89
21	PhOTf instead of **2**	92
22	PhCl instead of **2**	36
23	PhI instead of **2**	63

[a]Yields were determined by GC-MS with dodecane as the internal standard.

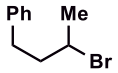


L1: R^1= *t*Bu
L2: R^1= H
L3: R^1= OMe



4: R^2= OMe
5: R^2= F

The major byproduct in the electrochemical dehydroxylative arylation of alcohol 1



S1

Physical state: colorless oil;

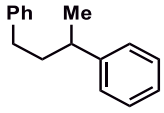
TLC: R_f = 0.57 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.24–7.17 (m, 3H), 4.12–4.04 (m, 1H), 2.90–2.83 (m, 1H), 2.79–2.71 (m, 1H), 2.19–2.00 (m, 2H), 1.73 (d, *J* = 6.7 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 141.0, 128.6, 128.6, 126.2, 51.0, 42.8, 34.1, 26.7 ppm;

HRMS (APCI): Calcd for $C_{10}H_{17}BrN$ $[M+NH_4]^+$: 230.0539; found 230.0537.

Experimental Procedure and Characterization Data



3

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) afforded crude **3**. Further purification by preparative TLC (silica gel, Petroleum ether) afforded **3** (37.8 mg, 90%).

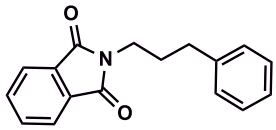
Physical state: colorless oil;

TLC: R_f = 0.57 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.26 (m, 4H), 7.25–7.11 (m, 6H), 2.77–2.68 (m, 1H), 2.57–2.45 (m, 2H), 2.01–1.82 (m, 2H), 1.28 (d, *J* = 7.0 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 147.4, 142.7, 128.5, 128.5, 128.4, 127.2, 126.1, 125.8, 40.1, 39.6, 34.1, 22.7 ppm;

HRMS (ESI): Calcd for $C_{16}H_{19}$ $[M+H]^+$: 211.1481; found 211.1483.



6

On 0.2 mmol scale, **General Procedure A** was followed with 3-phthalimido-1-propanol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **6**. Further purification by preparative TLC (silica gel, 1:4, EtOAc:Petroleum ether) afforded **6** (30.2 mg, 57%).

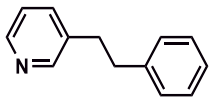
Physical state: white amorphous solid;

TLC: R_f = 0.50 (silica gel, 1:10, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84–7.80 (m, 2H), 7.73–7.67 (m, 2H), 7.28–7.22 (m, 2H), 7.22–7.18 (m, 2H), 7.17–7.11 (m, 1H), 3.75 (t, *J* = 7.2, 2H), 2.69 (t, *J* = 8.1, 2H), 2.07–1.99 (m, 2H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 168.6, 141.2, 134.0, 132.3, 128.5, 128.4, 126.1, 123.3, 38.0, 33.3, 30.0 ppm;

HRMS (ESI): Calcd for $C_{17}H_{16}NO_2$ $[M+H]^+$: 266.1176; found 266.1175.

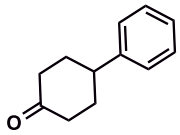


7

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 3-pyridinylethanol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 2:3 EtOAc: Petroleum Ether) afforded crude **7**. Further purification by preparative TLC (silica gel, 2:3, EtOAc:Petroleum ether) afforded **7** (26.4 mg, 72%).

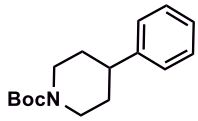
Physical state: white amorphous solid;

TLC: R_f = 0.51 (silica gel, 2:3, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46–8.41 (m, 2H), 7.43 (dt, *J* = 7.8, 2.0 Hz, 1H), 7.31–7.25 (m, 2H), 7.23–7.17 (m, 2H), 7.17–7.12 (m, 2H), 2.92 (s, 4H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 149.7, 147.2, 140.8, 137.1, 136.4, 128. 6, 126.3, 123.4, 37.5, 35.0 ppm;
HRMS (ESI): Calcd for $C_{13}H_{14}N$ $[M+H]^+$: 184.1121; found 184.1121.



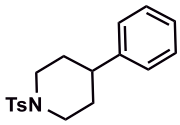
8

On 0.2 mmol scale, **General Procedure A** was followed with 4-hydroxycyclohexanone, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:1 CH_2Cl_2:Petroleum Ether) afforded **8** (21.3 mg, 61%).
Physical state: white amorphous solid;
TLC: R_f = 0.50 (silica gel, CH_2Cl_2, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.29 (m, 2H), 7.28–7.20 (m, 3H), 3.03 (tt, *J* = 12.2, 3.7 Hz, 1H), 2.58–2.44 (m, 4H), 2.32–2.19 (m, 2H), 2.01–1.89 (m, 2H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 211.4, 144.9, 128.7, 126.8, 126.7, 42.9, 41.5, 34.1 ppm;
HRMS (ESI): Calcd for $C_{12}H_{15}O$ $[M+H]^+$: 175.1117; found 175.1117.



9

On 0.2 mmol scale, **General Procedure A** was followed with *N*-Boc-4-piperidinol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **9**. Further purification by preparative TLC (silica gel, 1:9, EtOAc:Petroleum ether) afforded **9** (29.8 mg, 57%).
Physical state: colorless oil;
TLC: R_f = 0.46 (silica gel, 1:9, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.28 (m, 2H), 7.24–7.18 (m, 3H), 4.25 (d, *J* = 13.3 Hz, 2H), 2.80 (dt, *J* = 13.0, 2.6 Hz, 2H), 2.64 (tt, *J* = 12.2, 3.6 Hz, 1H), 1.86–1.78 (m, 2H), 1.69–1.56 (m, 2H), 1.48 (s, 9H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 155.0, 145.9, 128.6, 126.9, 126.5, 79.6, 44.5, 42.9, 33.3, 28.6 ppm;
HRMS (ESI): Calcd for $C_{16}H_{23}NNaO_2$ $[M+ Na]^+$: 284.1621; found 284.1619.



10

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 1-(4-methylphenyl)sulfonylpiperidin-4-ol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:9 EtOAc:Petroleum Ether) afforded

crude **10**, which was further treated by *m*-CPBA to removal of the alkene byproduct. Further purification by preparative TLC (silica gel, 1:4, EtOAc:Petroleum ether) afforded **10** (25.9 mg, 41%).

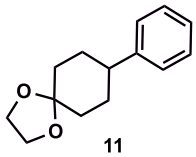
Physical state: white amorphous solid;

TLC: R_f = 0.50 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70–7.67 (m, 2H), 7.37–7.33 (m, 2H), 7.32–7.27 (m, 2H), 7.24–7.18 (m, 1H), 7.16–7.12 (m, 2H), 3.97–3.91 (m, 2H), 2.45 (s, 3H), 2.44–2.39 (m, 1H), 2.39–2.30 (m, 2H), 1.93–1.78 (m, 4H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.0, 143.6, 133.2, 129.8, 128.7, 127.9, 126.8, 126.7, 47.0, 42.0, 32.7, 21.7 ppm;

HRMS (ESI): Calcd for $C_{18}H_{22}NO_2S$ $[M+H]^+$: 316.1366; found 316.1367.



11

On 0.2 mmol scale, **General Procedure A** was followed with 4,4-ethylene-dioxycyclohexan-1-ol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:10 EtOAc:Petroleum Ether) afforded crude **11**. Further purification by preparative TLC (silica gel, 1:10, EtOAc:Petroleum ether) afforded **11** (19.6 mg, 45%).

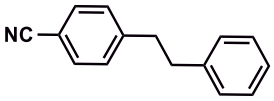
Physical state: white amorphous solid;

TLC: R_f = 0.45 (silica gel, 1:10, EtOAc:Petroleum Ether, Phosphomolybdic Acid);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35–7.26 (m, 2H), 7.26–7.16 (m, 3H), 3.99 (s, 4H), 2.61–2.49 (m, 1H), 1.91–1.84 (m, 4H), 1.83–1.74 (m, 2H), 1.74–1.65 (m, 2H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.7, 128.5, 127.0, 126.2, 108.6, 64.5, 64.4, 43.5, 35.3, 31.7 ppm;

HRMS (ESI): Calcd for $C_{14}H_{19}O_2$ $[M+H]^+$: 219.1380; found 219.1381.



12

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 2-(4-cyanophenyl)ethanol, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 0:100 to 3:17 EtOAc: Petroleum Ether) afforded crude **12**. Further purification by preparative TLC (silica gel, 1:9, EtOAc:Petroleum ether) afforded **12** (24.9 mg, 60%).

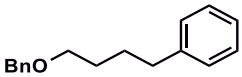
Physical state: white amorphous solid;

TLC: R_f = 0.68 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57–7.53 (m, 2H), 7.30–7.27 (m, 1H), 7.26–7.24 (m, 2H), 7.23–7.18 (m, 2H), 7.14–7.11 (m, 2H), 3.03–2.89 (m, 4H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 147.3, 140.7, 132.3, 129.5, 128.6, 128.5, 126.4, 119.2, 109.9, 38.0, 37.3 ppm;

HRMS (ESI): Calcd for $C_{15}H_{14}N$ $[M+H]^+$: 208.1121; found 208.1119.



13

On 0.2 mmol scale, **General Procedure A** was followed with 4-benzyloxy-butan-1-ol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:19 EtOAc:Petroleum Ether) afforded crude **13**. Further purification by preparative HPLC (83% to 98% CH_3CN/water) afforded **13** (20.2 mg, 42%).

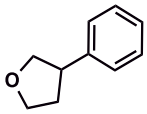
Physical state: colorless oil;

TLC: R_f = 0.47 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40–7.32 (m, 4H), 7.31–7.25 (m, 3H), 7.20–7.15 (m, 3H), 4.50 (s, 2H), 3.49 (t, *J* = 6.2 Hz, 2H), 2.63 (t, *J* = 7.3 Hz, 2H), 1.76–1.63 (m, 4H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.6, 138.8, 128.6, 128.5, 128.4, 127.8, 127.7, 125.8, 73.1, 70.4, 35.9, 29.6, 28.2 ppm;

HRMS (ESI): Calcd for $C_{17}H_{24}NO$ $[M+NH_4]^+$: 258.1852; found 258.1853.



14

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 3-hydroxytetrahydrofuran, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, hexane) afforded crude **14**. Further purification by preparative TLC (silica gel, 3:97, EtOAc:Petroleum ether) afforded **14** (19.6 mg, 66%).

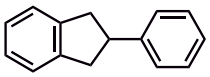
Physical state: yellow oil;

TLC: R_f = 0.50 (silica gel, 1:9, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35–7.29 (m, 2H), 7.28–7.21 (m, 3H), 4.16 (dd, *J* = 8.4, 7.5 Hz, 1H), 4.08 (dt, *J* = 8.3, 4.5 Hz, 1H), 3.93 (dt, *J* = 8.3, 7.3 Hz, 1H), 3.74 (dd, *J* = 8.4, 7.6 Hz, 1H), 3.46–3.36 (m, 1H), 2.42–2.31 (m, 1H), 2.07–1.97 (m, 1H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.8, 128.7, 127.4, 126.6, 74.8, 68.7, 45.1, 34.8 ppm;

HRMS (ESI): Calcd for $C_{10}H_{11}O$ $[M–H]^-$: 147.0815; found 147.0820.



15

On 0.2 mmol scale, **General Procedure A** was followed with indan-2-ol, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) afforded crude **15**. Further purification by preparative TLC (silica gel, Petroleum Ether) afforded **15** (28.8 mg, 74%).

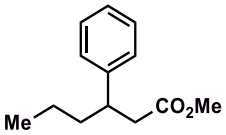
Physical state: colorless oil;

TLC: R_f = 0.49 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 4.4 Hz, 4H), 7.28–7.22 (m, 3H), 7.22–7.16 (m, 2H), 3.75–3.65 (m, 1H), 3.36 (dd, *J* = 15.3, 8.2 Hz, 2H), 3.10 (dd, *J* = 15.5, 9.0 Hz, 2H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.6, 143.1, 128.6, 127.2, 126.6, 126.3, 124.5, 45.7, 41.1 ppm;

HRMS (ESI): Calcd for $C_{15}H_{14}Na$ $[M+Na]^+$: 217.0988; found 217.0979.



16

On 0.2 mmol scale, **General Procedure A** was followed with methyl 3-hydroxy-hexanoate, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 3:47 EtOAc:Petroleum Ether) afforded **16** (30.1 mg, 73%).

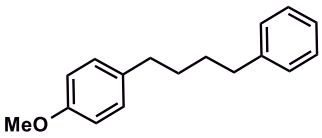
Physical state: yellow oil;

TLC: R_f = 0.53 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.23–7.15 (m, 3H), 3.58 (s, 3H), 3.15–3.07 (m, 1H), 2.68–2.54 (m, 2H), 1.66–1.55 (m, 2H), 1.24–1.12 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 173.1, 144.3, 128.5, 127.6, 126.5, 51.6, 42.1, 41.8, 38.5, 20.6, 14.1 ppm;

HRMS (ESI): Calcd for $C_{13}H_{19}O_2$ $[M+H]^+$: 207.1380; found 207.1380.



17

On 0.2 mmol scale, **General Procedure A** was followed with 4-(4-methoxyphenyl) butan-1-ol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:99 EtOAc:hexane) afforded crude **17**. Further purification by preparative TLC (silica gel, hexane) afforded **17** (25.0 mg, 52%).

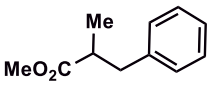
Physical state: white amorphous solid;

TLC: R_f = 0.40 (silica gel, 1:99, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.24 (m, 2H), 7.20–7.15 (m, 3H), 7.10–7.06 (m, 2H), 6.87–6.79 (m, 2H), 3.79 (s, 3H), 2.67–2.55 (m, 4H), 1.70–1.58 (m, 4H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 157.8, 142.8, 134.8, 129.4, 128.6, 128.4, 125.8, 113.8, 55.4, 36.0, 35.0, 31.5, 31.2 ppm;

HRMS (ESI): Calcd for $C_{17}H_{21}O$ $[M+H]^+$: 241.1587; found 241.1584.



18

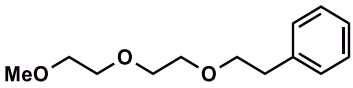
On 0.2 mmol scale, **General Procedure A** was followed with 3-hydroxy-2-methyl propionate, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **18**. Further purification by preparative TLC (silica gel, 1:19, EtOAc:Petroleum ether) afforded **18** (14.2 mg, 40%).

Physical state: colorless oil;

TLC: R_f = 0.58 (silica gel, 1:9, EtOAc:Petroleum Ether, UV);

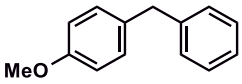
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31–7.25 (m, 2H), 7.23–7.19 (m, 1H), 7.19–7.13 (m, 2H), 3.64 (s, 3H), 3.03 (dd, *J* = 13.1, 6.5 Hz, 1H), 2.80–2.62 (m, 2H), 1.16 (d, *J* = 6.8 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 176.7, 139.5, 129.1, 128.5, 126.5, 51.7, 41.6, 39.8, 16.9 ppm;
HRMS (ESI): Calcd for $C_{11}H_{15}O_2$ $[M+H]^+$: 179.1067; found 179.1066.



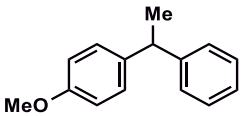
19

On 0.2 mmol scale, **General Procedure A** was followed with triethylene glucol monomethyl ether, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 1:5, EtOAc:Petroleum Ether) afforded **19** (32.9 mg, 73%).
Physical state: yellow oil;
TLC: R_f = 0.57 (silica gel, 2:3, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33–7.24 (m, 2H), 7.24–7.16 (m, 3H), 3.69 (t, *J* = 7.3 Hz, 2H), 3.67–3.61 (m, 6H), 3.56–3.53 (m, 2H), 3.38 (s, 3H), 2.91 (t, *J* = 7.3 Hz, 2H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 139.0, 129.0, 128.4, 126.3, 72.4, 72.1, 70.7, 70.7, 70.4, 59.1, 36.4 ppm;
HRMS (ESI): Calcd for $C_{13}H_{21}O_3$ $[M+H]^+$: 225.1485; found 225.1485.



20

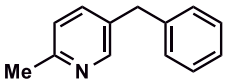
On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-methoxybenzyl alcohol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:33 EtOAc: Petroleum Ether) afforded **20** (37.3 mg, 94%).
Physical state: yellow oil;
TLC: R_f = 0.34 (silica gel, 1:33, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.28 (m, 2H), 7.25–7.19 (m, 3H), 7.16–7.12 (m, 2H), 6.89–6.84 (m, 2H), 3.96 (s, 2H), 3.81 (s, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 158.1, 141.7, 133.4, 130.0, 128.9, 128.6, 126.1, 114.0, 55.4, 41.2 ppm;
HRMS (ESI): Calcd for $C_{14}H_{15}O$ $[M+H]^+$: 199.1117; found 199.1115.



21

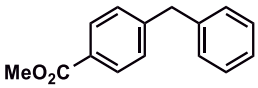
On 0.2 mmol scale, **General Procedure A** was followed with 1-(4-methoxyphenyl)-ethanol, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 1:99 CH_2Cl_2:Petroleum Ether) afforded crude **21**. Further purification by preparative TLC (silica gel, 1:19, EtOAc:Petroleum ether) afforded **21** (26.7 mg, 63%).
Physical state: yellow oil;
TLC: R_f = 0.60 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.26 (m, 2H), 7.24–7.18 (m, 3H), 7.17–7.13 (m, 2H),

6.86–6.82 (m, 2H), 4.12 (q, J = 7.2 Hz, 1H), 3.79 (s, 3H), 1.63 (d, J = 7.2 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 158.0, 146.9, 138.7, 128.6, 128.5, 127.7, 126.1, 113.8, 55.4, 44.1, 22.2 ppm;
HRMS (ESI): Calcd for $C_{15}H_{17}O$ $[M+H]^+$: 213.1274; found 213.1275.



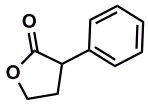
22

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 6-methyl-3-pyridinemethanol, bromobenzene, and triphenyl-phosphine (3.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:3 EtOAc:Petroleum Ether) afforded **22** (34.8 mg, 95%).
Physical state: white amorphous solid;
TLC: R_f = 0.42 (silica gel, 2:3, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 (d, J = 2.4 Hz, 1H), 7.40 (dd, J = 7.9, 2.4 Hz, 1H), 7.32–7.27 (m, 2H), 7.24–7.14 (m, 3H), 7.09 (d, J = 8.0 Hz, 1H), 3.95 (s, 2H), 2.55 (s, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 156.2, 149.2, 140.2, 137.0, 133.5, 128.9, 128.7, 126.5, 123.2, 38.7, 24.0 ppm;
HRMS (ESI): Calcd for $C_{13}H_{14}N$ $[M+H]^+$: 184.1121; found 184.1120.



23

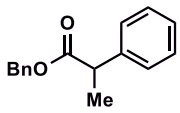
On 0.2 mmol scale, **General Procedure A** was followed with 4-(methoxycarbonyl) benzyl alcohol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:19 EtOAc:Petroleum Ether) afforded crude **23**. Further purification by preparative TLC (silica gel, 1:49, EtOAc:Petroleum ether) afforded **23** (38.9 mg, 86%).
Physical state: colorless oil;
TLC: R_f = 0.74 (silica gel, 1:24, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98–7.94 (m, 2H), 7.32–7.26 (m, 3H), 7.25–7.21 (m, 2H), 7.21–7.14 (m, 2H), 4.03 (s, 2H), 3.90 (s, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 167.2, 146.7, 140.3, 130.0, 129.1, 128.7, 128.2, 126.5, 52.1, 42.0 ppm;
HRMS (ESI): Calcd for $C_{15}H_{15}O_2$ $[M+H]^+$: 227.1067; found 227.1065.



24

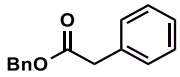
On 0.2 mmol scale, **General Procedure A** was followed with 3-hydroxyoxolan-2-one, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded **24** (30.8 mg, 95%).
Physical state: yellow oil;
TLC: R_f = 0.19 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41–7.35 (m, 2H), 7.34–7.27 (m, 3H), 4.52–4.45 (m, 1H), 4.36 (dt, J = 9.2, 6.7 Hz, 1H), 3.82 (dd, J = 10.2, 8.9 Hz, 1H), 2.77–2.68 (m, 1H), 2.51–2.40 (m, 1H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 177.5, 136.8, 129.1, 128.0, 127.8, 66.6, 45.6, 31.7 ppm;
HRMS (ESI): Calcd for $C_{10}H_{11}O_2$ $[M+H]^+$: 163.0754; found 163.0754.



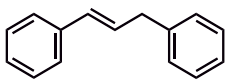
25

On 0.2 mmol scale, **General Procedure A** was followed with benzyl lactate, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 1:20 EtOAc: Petroleum Ether) afforded crude **25**. Further purification by preparative TLC (silica gel, 1:20, EtOAc:Petroleum ether) afforded **25** (37.9 mg, 79%).
Physical state: colorless oil;
TLC: R_f = 0.49 (silica gel, 1:30, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35–7.22 (m, 10H), 5.11 (q, J = 12.5 Hz, 2H), 3.78 (q, J = 7.2 Hz, 1H), 1.52 (d, J = 7.2 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 174.4, 140.5, 136.1, 128.7, 128.6, 128.2, 128.0, 127.6, 127.3, 66.5, 45.6, 18.6 ppm;
HRMS (ESI): Calcd for $C_{16}H_{17}O_2$ $[M+H]^+$: 241.1223; found 241.1223.



26

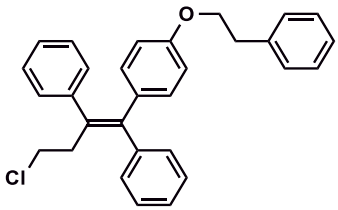
On 0.2 mmol scale, **General Procedure A** was followed with benzyl glycolate, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 1:19 EtOAc: Petroleum Ether) afforded crude **26**. Further purification by preparative TLC (silica gel, 1:19, EtOAc:Petroleum ether) afforded **26** (38.5 mg, 85%).
Physical state: colorless oil;
TLC: R_f = 0.40 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40–7.26 (m, 10H), 5.15 (s, 2H), 3.69 (s, 2H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 171.5, 136.0, 134.0, 129.4, 128.7, 128.7, 128.3, 128.3, 127.3, 66.7, 41.5 ppm;
HRMS (ESI): Calcd for $C_{15}H_{15}O_2$ $[M+H]^+$: 227.1067; found 227.1067.



27

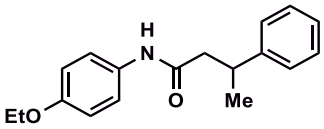
On 0.2 mmol scale, **General Procedure A** was followed with cinnamyl alcohol, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, hexane) afforded crude **27**. Further purification by preparative TLC (silica gel, hexane) afforded **27** (27.6 mg, 71%).
Physical state: colorless oil;
TLC: R_f = 0.40 (silica gel, *n*-hexane, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42–7.21 (m, 10H), 6.52–6.46 (m, 1H), 6.43–6.34 (m, 1H), 3.58 (dd, J = 6.6, 1.2 Hz, 2H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 140.3, 137.6, 131.2, 129.4, 128.8, 128.6, 127.2, 126.3, 126.3, 39.5 ppm;
HRMS (ESI): Calcd for $C_{15}H_{15}$ $[M+H]^+$: 195.1168; found 195.1168.



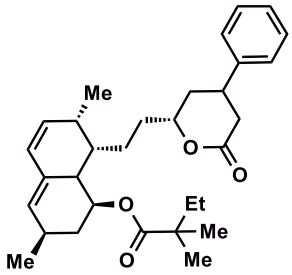
28

On 0.2 mmol scale, **General Procedure A** was followed with ospemifene, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **28**. Further purification by preparative TLC (silica gel, 1:19, EtOAc:Petroleum ether) afforded **28** (50.0 mg, 57%).
Physical state: white amorphous solid;
TLC: R_f = 0.45 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41–7.35 (m, 2H), 7.32–7.27 (m, 5H), 7.25–7.18 (m, 5H), 7.17–7.11 (m, 3H), 6.84–6.72 (m, 2H), 6.64–6.51 (m, 2H), 4.04 (t, J = 7.1 Hz, 2H), 3.42 (t, J = 7.5 Hz, 2H), 3.02 (t, J = 7.1 Hz, 2H), 2.93 (t, J = 7.5 Hz, 2H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 157.1, 143.0, 141.9, 141.1, 138.3, 135.3, 134.9, 131.8, 129.7, 129.5, 129.1, 128.6, 128.5, 128.4, 127.1, 126.7, 126.7, 113.6, 68.5, 43.0, 38.7, 35.9 ppm;
HRMS (ESI): Calcd for $C_{30}H_{28}ClO$ $[M+H]^+$: 439.1823; found 439.1820.



29

On 0.2 mmol scale, **General Procedure A** was followed with bucetin, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:4, EtOAc:Petroleum Ether) afforded crude **29**. Further purification by preparative TLC (silica gel, 3:7, EtOAc:Petroleum ether) afforded **29** (41.9 mg, 74%).
Physical state: white amorphous solid;
TLC: R_f = 0.55 (silica gel, 2:3, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.28 (m, 2H), 7.28–7.17 (m, 5H), 6.87 (*br*, s, 1H), 6.81–6.76 (m, 2H), 3.97 (q, J = 7.0 Hz, 2H), 3.41–3.31 (m, 1H), 2.64–2.51 (m, 2H), 1.38 (t, J = 6.8 Hz, 3H), 1.37 (d, J = 6.8 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 169.9, 155.9, 145.9, 130.7, 128.9, 127.0, 126.7, 122.1, 114.8, 63.8, 46.8, 37.3, 21.8, 15.0 ppm;
HRMS (ESI): Calcd for $C_{18}H_{22}NO_2$ $[M+H]^+$: 284.1645; found 284.1645.



30

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with simvastatin, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 1:4 EtOAc:Petroleum Ether) afforded **30** as two inseparable diastereomers (d.r. = 1.5:1, 70.8 mg, 74%).

Physical state: white amorphous solid;

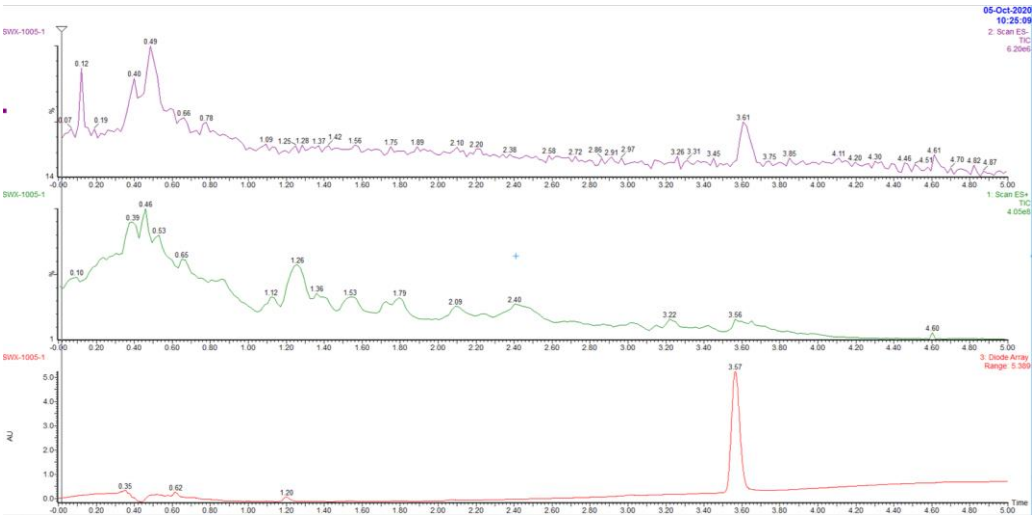
TLC: R_f = 0.37 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

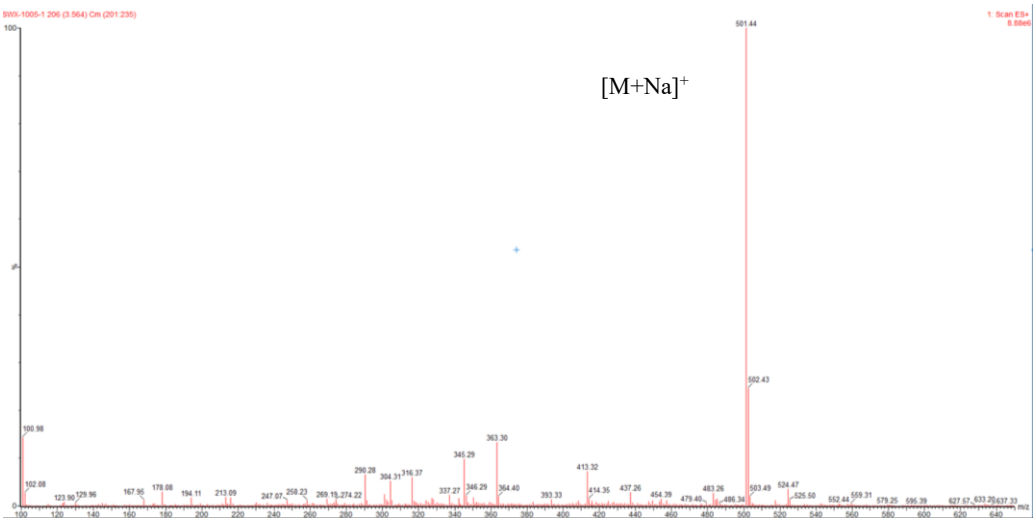
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.32 (m, 2H), 7.31–7.23 (m, 1H), 7.22–7.17 (m, 2H), 5.99 (dd, *J* = 9.7, 3.3 Hz, 1H), 5.81–5.74 (m, 1H), 5.52–5.48 (m, 1H), 5.37–5.31 (m, 1H), 4.38–4.27 (m, 1H), 3.41–3.27 (m, 1H), 3.23–3.12 (m, 0.5H), 2.92 (ddd, *J* = 17.7, 6.0, 1.9 Hz, 0.5H), 2.85–2.66 (m, 2H), 2.53 (dd, *J* = 17.8, 11.3 Hz, 1H), 2.46–2.39 (m, 1H), 2.39–2.32 (m, 1H), 2.30–2.20 (m, 1H), 2.20–2.12 (m, 1H), 2.09–1.99 (m, 2H), 1.98–1.91 (m, 2H), 1.93–1.82 (m, 1H), 1.73–1.62 (m, 1H), 1.58–1.30 (m, 2H), 1.08 (dt, *J* = 7.4, 5.0 Hz, 9H), 0.89 (t, *J* = 7.6 Hz, 3H), 0.78 (dt, *J* = 16.5, 7.5 Hz, 3H) ppm;

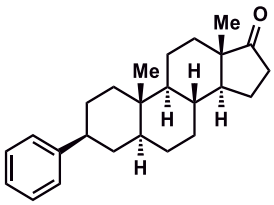
^{13}C NMR (101 MHz, $CDCl_3$): δ 177.8, 177.8, 171.8, 170.8, 143.3, 143.1, 133.0, 133.0, 131.7, 131.7, 129.9, 129.1, 129.1, 128.6, 127.3, 127.2, 126.7, 126.5, 81.2, 78.3, 68.1, 68.1, 43.1, 37.7, 37.7, 37.7, 36.9, 36.7, 36.3, 35.5, 34.9, 33.9, 33.2, 33.2, 33.1, 33.0, 33.0, 30.8, 27.4, 24.9, 24.8, 24.8, 24.8, 24.6, 23.2, 14.1, 9.4, 9.4 ppm;

HRMS (ESI): Calcd for $C_{31}H_{43}O_4$ $[M+H]^+$: 479.3156; found 479.3160.

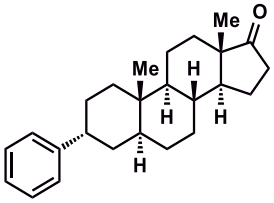
*The purity of **30** was further determined by UPLC-MS.*




[M+Na]+
501.44
502.43
100.98
152.08
363.30
345.29
413.32
290.28
304.31
316.37
337.27
346.29
364.40
393.33
414.35
437.26
454.39
479.40
483.26
486.34
503.49
524.47
525.50
552.44
559.31
579.25
596.39
627.57
633.20
637.33
m/z



31a



31b

On 0.2 mmol scale, **General Procedure A** was followed with epiandrosterone, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **31**. Further purification by preparative TLC (silica gel, 1:9, EtOAc:Petroleum ether) afforded **31a** and **31b** (d.r. = 1:1, 34.4 mg, 49%).

For characterization, these two diastereomers were further separated by preparative HPLC (100% CH_3CN).

31a:

Physical state: white solid;

Melting point: 167.5–167.9 °C;

TLC: R_f = 0.23 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.24–7.15 (m, 3H), 2.61–2.51 (m, 1H), 2.45 (ddd, *J* = 19.1, 8.9, 1.0 Hz, 1H), 2.08 (dt, *J* = 19.1, 9.0 Hz, 1H), 1.99–1.90 (m, 1H), 1.86–1.78 (m, 3H), 1.78–1.67 (m, 2H), 1.67–1.60 (m, 1H), 1.59–1.56 (m, 1H), 1.55–1.45 (m, 3H), 1.38–1.25 (m, 6H), 1.11 (dt, *J* = 13.0, 4.3 Hz, 1H), 1.08–0.99 (m, 1H), 0.91 (s, 3H), 0.88 (s, 3H), 0.84–0.74 (m, 1H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 221.7, 147.6, 128.4, 126.9, 126.0, 54.9, 51.7, 48.0, 47.2, 44.9, 39.0, 36.7, 36.1, 36.0, 35.3, 31.8, 31.1, 29.9, 28.7, 21.9, 20.5, 14.0, 12.6 ppm;

HRMS (ESI): Calcd for $C_{25}H_{35}O$ $[M+H]^+$: 351.2682; found 351.2675.

31b:

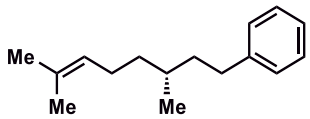
Physical state: white amorphous solid;

TLC: R_f = 0.23 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40–7.34 (m, 2H), 7.34–7.29 (m, 2H), 7.20–7.15 (m, 1H), 3.09 (t, *J* = 6.0 Hz, 1H), 2.41 (ddd, *J* = 19.1, 8.9, 1.1 Hz, 1H), 2.14–2.07 (m, 1H), 2.06–1.94 (m, 2H), 1.94–1.81 (m, 2H), 1.80–1.70 (m, 3H), 1.67–1.51 (m, 3H), 1.50–1.40 (m, 1H), 1.35–1.13 (m, 7H), 0.97–0.83 (m, 1H), 0.91 (d, *J* = 0.6 Hz, 3H), 0.85 (d, *J* = 0.5 Hz, 3H), 0.70–0.62 (m, 1H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 221.7, 145.5, 128.2, 127.7, 125.3, 54.8, 51.6, 48.0, 40.9, 36.5, 36.0, 35.1, 34.5, 33.3, 31.7, 30.8, 28.7, 25.1, 21.9, 20.1, 14.0, 12.2 ppm;

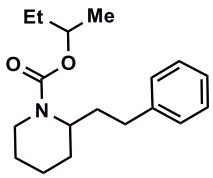
HRMS (ESI): Calcd for $C_{25}H_{35}O$ $[M+H]^+$: 351.2682; found 351.2678.



32

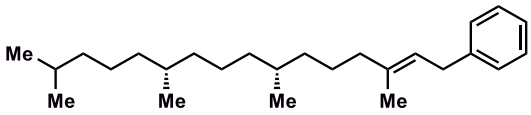
On 0.2 mmol scale, **General Procedure A** was followed with citronellol, bromo-benzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, pentane) afforded crude **32**. Further purification by preparative TLC (silica gel, Petroleum ether) afforded **32** (25.5 mg, 59%).

Physical state: colorless oil;
TLC: R_f = 0.62 (silica gel, Petroleum ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.25 (m, 2H), 7.20–7.12 (m, 3H), 5.13–5.07 (m, 1H), 2.70–2.51 (m, 2H), 2.07–1.89 (m, 2H), 1.68 (d, *J* = 1.3 Hz, 3H), 1.67–1.62 (m, 1H), 1.60 (d, *J* = 1.2 Hz, 3H), 1.52–1.33 (m, 3H), 1.25–1.14 (m, 1H), 0.94 (d, *J* = 6.3 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 143.3, 131.3, 128.5, 128.4, 125.7, 125.1, 39.1, 37.1, 33.6, 32.3, 25.9, 25.6, 19.7, 17.8 ppm;
HRMS (ESI): Calcd for $C_{16}H_{25}$ $[M+H]^+$: 217.1951; found 217.1956.



33

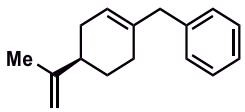
On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with icaridin, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 1:19 EtOAc:Petroleum Ether) afforded crude **33**. Further purification by preparative TLC (silica gel, 1:9, EtOAc:Petroleum ether) afforded **33** (48.6 mg, 84%).
Note: The starting material icaridin was used as a mixture of diastereomers.
Physical state: yellow oil;
TLC: R_f = 0.50 (silica gel, 1:9, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.25 (m, 2H), 7.22–7.16 (m, 3H), 4.82–4.72 (m, 1H), 4.35 (*br*, s, 1H), 4.06 (d, *J* = 13.5 Hz, 1H), 2.84 (dt, *J* = 13.2, 2.4 Hz, 1H), 2.66–2.48 (m, 2H), 2.09–1.96 (m, 1H), 1.78–1.68 (m, 1H), 1.68–1.48 (m, 7H), 1.47–1.37 (m, 1H), 1.21 (dd, *J* = 6.2, 0.7 Hz, 3H), 0.91 (dt, *J* = 7.4, 5.0 Hz, 3H) ppm;
^{13}C NMR (400 MHz, $CDCl_3$): δ 155.9, 142.3, 128.5, 128.5, 125.9, 73.0, 72.9, 50.6, 50.5, 39.1, 39.0, 33.0, 32.9, 32.0, 29.3, 29.3, 28.6, 25.7, 20.0, 20.0, 19.2, 9.9, 9.9 ppm;
HRMS (ESI): Calcd for $C_{18}H_{28}NO_2$ $[M+H]^+$: 290.2115; found 290.2115.



34

On 0.2 mmol scale, **General Procedure A** was followed with phytol, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded **34** (53.5 mg, 75%).
Physical state: colorless oil;
TLC: R_f = 0.71 (silica gel, Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.27 (m, 2H), 7.23–7.17 (m, 3H), 5.39–5.32 (m, 1H), 3.39 (d, *J* = 7.3 Hz, 2H), 2.09–1.97 (m, 2H), 1.73 (t, *J* = 1.5 Hz, 3H), 1.60–1.50 (m, 1H), 1.50–1.37 (m, 4H), 1.36–1.24 (m, 8H), 1.19–1.14 (m, 2H), 1.11–1.03 (m, 4H), 0.89 (d, *J* = 6.8 Hz, 6H), 0.88 (d, *J* = 1.9 Hz, 3H), 0.87 (d, *J* = 1.9 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 142.0, 136.8, 128.5, 125.8, 122.9, 40.2, 39.5, 37.6, 37.6, 37.5, 36.8, 34.4, 33.0, 32.8, 28.1, 25.5, 25.0, 24.6, 22.9, 22.8, 19.9, 16.2 ppm;

HRMS (ESI): Calcd for $C_{26}H_{45}$ $[M+H]^+$: 357.3516; found 357.3527.



35

On 0.2 mmol scale, **General Procedure A** was followed with perillol, bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **35**. Further purification by preparative TLC (silica gel, Petroleum ether) afforded **35** (23.4 mg, 55%).

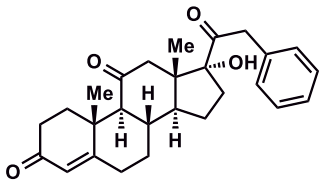
Physical state: colorless oil;

TLC: R_f = 0.67 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.25 (m, 2H), 7.23–7.14 (m, 3H), 5.53–5.45 (m, 1H), 4.75–4.67 (m, 2H), 3.27 (s, 2H), 2.20–2.07 (m, 2H), 2.03–1.92 (m, 3H), 1.82–1.76 (m, 1H), 1.73 (t, J = 1.2 Hz, 3H), 1.51–1.40 (m, 1H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 150.3, 140.5, 137.1, 129.0, 128.3, 126.0, 122.6, 108.6, 44.4, 41.3, 31.0, 28.7, 28.0, 20.9 ppm;

HRMS (ESI): Calcd for $C_{16}H_{21}$ $[M+H]^+$: 213.1638; found 213.1638.



36

On 0.2 mmol scale, **General Procedure A** was followed with cortisone, bromobenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 1:2, EtOAc:Petroleum ether) afforded crude **36**. Further purification by preparative TLC (silica gel, 1:1, EtOAc:Petroleum ether) afforded **36** (51.3 mg, 61%).

Physical state: white solid;

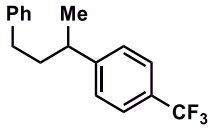
Melting point: 210.4–213.5 °C;

TLC: R_f = 0.39 (silica gel, 1:1, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35–7.29 (m, 2H), 7.28–7.22 (m, 1H), 7.19–7.13 (m, 2H), 5.72 (s, 1H), 4.02 (d, J = 16.0 Hz, 1H), 3.72 (d, J = 16.0 Hz, 1H), 2.91–2.72 (m, 3H), 2.52–2.35 (m, 3H), 2.33–2.25 (m, 2H), 2.12 (d, J = 12.4 Hz, 1H), 2.02–1.89 (m, 4H), 1.73–1.58 (m, 2H), 1.52–1.42 (m, 1H), 1.41 (s, 3H), 1.35–1.22 (m, 1H), 0.75 (s, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 209.7, 209.6, 200.0, 168.9, 133.8, 129.7, 128.7, 127.2, 124.7, 89.4, 62.7, 51.7, 50.4, 49.7, 46.3, 38.3, 36.6, 34.8, 34.5 33.8, 32.4, 32.4, 23.6, 17.3, 16.4 ppm;

HRMS (ESI): Calcd for $C_{27}H_{33}O_4$ $[M+H]^+$: 421.2373; found 421.2374.



37

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 1-bromo-4-

trifluoromethylbenzene, and triphenylphosphine (10.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) afforded crude **37**. Further purification by preparative TLC (silica gel, pentane) afforded **37** (33.4 mg, 60%).

Physical state: colorless oil;

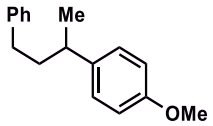
TLC: R_f = 0.80 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59–7.54 (m, 2H), 7.34–7.29 (m, 2H), 7.28–7.24 (m, 2H), 7.21–7.15 (m, 1H), 7.14–7.09 (m, 2H), 2.85–2.74 (m, 1H), 2.58–2.45 (m, 2H), 1.97–1.90 (m, 2H), 1.29 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 151.5, 142.2, 128.5, 128.5, 127.6, 126.0, 125.5 (q, *J* = 4.0 Hz), 124.5 (q, *J* = 273 Hz), 39.8, 39.5, 33.9, 22.4 ppm;

^{19}F NMR (376 MHz, $CDCl_3$): δ 62.28 ppm;

HRMS (APCI): Calcd for $C_{17}H_{21}F_3N$ $[M+NH_4]^+$: 296.1621; found 296.1635.



38

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 1-bromo-4-methoxy-benzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) afforded crude **38**. Further purification by preparative TLC (silica gel, Petroleum ether) afforded **38** (29.2 mg, 61%).

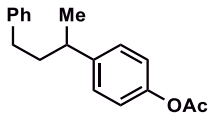
Physical state: colorless oil;

TLC: R_f = 0.39 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.25 (m, 2H), 7.22–7.18 (m, 1H), 7.18–7.10 (m, 4H), 6.93–6.83 (m, 2H), 3.82 (s, 3H), 2.76–2.64 (m, 1H), 2.58–2.47 (m, 2H), 1.97–1.84 (m, 2H), 1.27 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 157.9, 142.7, 139.5, 128.5, 128.4, 128.0, 125.7, 113.9, 55.4, 40.3, 38.8, 34.1, 22.9 ppm;

HRMS (ESI): Calcd for $C_{17}H_{21}O$ $[M+H]^+$: 241.1587; found 241.1586.



39

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 4-bromophenyl acetate, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **39**. Further purification by preparative TLC (silica gel, 1:9, EtOAc: Petroleum ether) afforded **39** (22.0 mg, 41%).

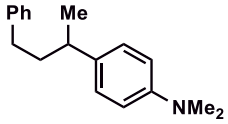
Physical state: colorless oil;

TLC: R_f = 0.37 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29–7.24 (m, 2H), 7.22–7.16 (m, 3H), 7.16–7.11 (m, 2H), 7.05–7.01 (m, 2H), 2.78–2.68 (m, 1H), 2.58–2.46 (m, 2H), 2.30 (s, 3H), 1.97–1.85 (m, 2H), 1.27 (d, *J* = 6.9 Hz, 3H) ppm;

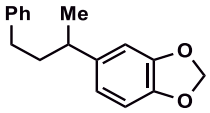
^{13}C NMR (101 MHz, $CDCl_3$): δ 169.8, 148.9, 144.9, 142.5, 128.5, 128.4, 128.1, 125.8, 121.5, 40.1,

39.1, 34.0, 22.6, 21.3 ppm;
HRMS (ESI): Calcd for $C_{18}H_{21}O_2$ $[M+H]^+$: 269.1536; found 269.1535.



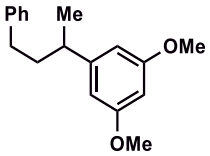
40

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-phenylbutan-2-ol, 4-bromo-*N,N*-dimethylaniline, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, hexane) afforded crude **40**. Further purification by preparative TLC (silica gel, 1:9, EtOAc: Petroleum ether) afforded **40** (31.0 mg, 62%).
Physical state: yellow oil;
TLC: R_f = 0.42 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.23 (m, 2H), 7.20–7.07 (m, 5H), 6.77 (*br*, s, 2H), 2.94 (s, 6H), 2.70–2.60 (m, 1H), 2.52 (t, *J* = 8.0 Hz, 2H), 1.95–1.81 (m, 2H), 1.24 (d, *J* = 6.9 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 148.7, 142.9, 128.5, 128.3, 127.8, 125.7, 113.5, 41.3, 40.3, 38.6, 34.1, 22.9 ppm;
HRMS (ESI): Calcd for $C_{18}H_{24}N$ $[M+H]^+$: 254.1903; found 254.1903.



41

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-phenylbutan-2-ol, 1,2-(methylenedioxy)-4-bromobenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 1:19 EtOAc:Petroleum Ether) afforded crude **41**. Further purification by preparative TLC (silica gel, 100:1:1, Petroleum ether: EtOAc:CH_2Cl_2) afforded **41** (32.0 mg, 63%).
Physical state: yellow oil;
TLC: R_f = 0.60 (silica gel, 1:19, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.24 (m, 2H), 7.21–7.13 (m, 3H), 6.78 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 1.7 Hz, 1H), 6.69–6.65 (m, 1H), 5.95 (s, 2H), 2.72–2.62 (m, 1H), 2.57–2.48 (m, 2H), 1.93–1.84 (m, 2H), 1.25 (d, *J* = 6.9 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 147.8, 145.7, 142.6, 141.4, 128.5, 128.4, 125.8, 120.1, 108.2, 107.4, 100.9, 40.3, 39.4, 34.0, 22.9 ppm;
HRMS (APCI): Calcd for $C_{17}H_{19}O_2$ $[M+H]^+$: 255.1380; found 255.1388.



42

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 1-bromo-3,5-dimethoxybenzene, and triphenylphosphine (7.0 equiv). Purification by column chromatography

(silica gel, 1:19 EtOAc:Petroleum Ether) afforded crude **42**. Further purification by preparative TLC (silica gel, 1:19, EtOAc:Petroleum ether) afforded **42** (31.0 mg, 57%).

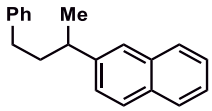
Physical state: colorless oil;

TLC: R_f = 0.55 (silica gel, 1:24, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31–7.23 (m, 2H), 7.20–7.12 (m, 3H), 6.38 (d, *J* = 2.3 Hz, 2H), 6.33 (t, *J* = 2.3 Hz, 1H), 3.80 (s, 6H), 2.71–2.61 (m, 1H), 2.53 (t, *J* = 8.0 Hz, 2H), 1.98–1.81 (m, 2H), 1.26 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 160.9, 150.1, 142.7, 128.5, 128.4, 125.8, 105.4, 97.7, 55.4, 40.0, 39.9, 34.1, 22.6 ppm;

HRMS (ESI): Calcd for $C_{18}H_{23}O_2$ $[M+H]^+$: 271.1693; found 271.1693.



43

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 2-bromo-naphthalene, and triphenylphosphine (10.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **43**. Further purification by preparative TLC (silica gel, pentane) afforded **43** (23.5 mg, 45%).

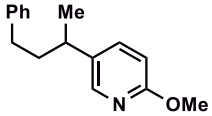
Physical state: colorless oil;

TLC: R_f = 0.32 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85–7.78 (m, 3H), 7.63 (d, *J* = 1.7 Hz, 1H), 7.49–7.40 (m, 2H), 7.38 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.29–7.23 (m, 2H), 7.20–7.10 (m, 3H), 2.96–2.84 (m, 1H), 2.61–2.48 (m, 2H), 2.18–1.86 (m, 2H), 1.36 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 144.8, 142.7, 133.8, 132.4, 128.5, 128.4, 128.2, 127.8, 127.7, 126.0, 125.9, 125.8, 125.5, 125.3, 40.0, 39.8, 34.1, 22.7 ppm;

HRMS (ESI): Calcd for $C_{20}H_{21}$ $[M+H]^+$: 261.1638; found 261.1639.



44

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-phenylbutan-2-ol, 2-methoxy-5-bromopyridine, and triphenylphosphine (10.0 equiv). Purification by column chromatography (silica gel, 100:3:3, Petroleum ether:EtOAc:CH_2Cl_2) afforded crude **44**. Further purification by preparative TLC (silica gel, 100:5:5, Petroleum ether:EtOAc:CH_2Cl_2) afforded **44** (31.4 mg, 65%).

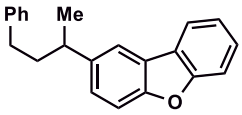
Physical state: colorless oil;

TLC: R_f = 0.30 (silica gel, 3:97, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (dd, *J* = 2.5, 0.6 Hz, 1H), 7.43 (dd, *J* = 8.5, 2.5 Hz, 1H), 7.29–7.23 (m, 2H), 7.20–7.14 (m, 1H), 7.14–7.09 (m, 2H), 6.72 (dd, *J* = 8.6, 0.6 Hz, 1H), 3.93 (s, 3H), 2.74–2.63 (m, 1H), 2.50 (t, *J* = 8.0 Hz, 2H), 1.96–1.80 (m, 2H), 1.25 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 162.9, 145.5, 142.2, 137.3, 135.0, 128.5, 125.9, 110.8, 53.4, 39.9, 36.2, 33.9, 22.6 ppm;

HRMS (ESI): Calcd for $C_{16}H_{20}NO$ $[M+H]^+$: 242.1539; found 242.1540.



45

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 2-bromo-dibenzofuran, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) afforded crude **45**. Further purification by preparative TLC (silica gel, Petroleum ether) afforded **45** (24.0 mg, 40%).

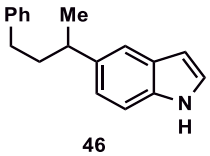
Physical state: colorless oil;

TLC: R_f = 0.30 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00–7.92 (m, 1H), 7.79–7.75 (m, 1H), 7.55 (dt, *J* = 8.2, 0.8 Hz, 1H), 7.50 (dd, *J* = 8.4, 0.6 Hz, 1H), 7.46–7.41 (m, 1H), 7.36–7.22 (m, 4H), 7.19–7.11 (m, 3H), 2.95–2.83 (m, 1H), 2.62–2.47 (m, 2H), 2.08–1.93 (m, 2H), 1.36 (d, *J* = 7.0 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 156.6, 155.0, 142.6, 142.0, 128.5, 128.4, 127.1, 126.5, 125.8, 124.5, 124.4, 122.7, 120.7, 118.8, 111.8, 111.5, 40.6, 39.7, 34.1, 23.2 ppm;

HRMS (ESI): Calcd for $C_{22}H_{21}O$ $[M+H]^+$: 301.1587; found 301.1585.



46

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-phenylbutan-2-ol, 5-bromo-1H-indole, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 1:9 EtOAc:Petroleum Ether) afforded crude **46**. Further purification by preparative TLC (silica gel, 1:9, EtOAc:toluene) afforded **46** (21.4 mg, 43%).

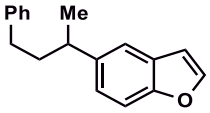
Physical state: colorless oil;

TLC: R_f = 0.58 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 (*br*, s, 1H), 7.52–7.45 (m, 1H), 7.35 (d, *J* = 8.3 Hz, 1H), 7.29–7.23 (m, 2H), 7.22–7.12 (m, 4H), 7.08 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.54–6.51 (m, 1H), 2.88–2.77 (m, 1H), 2.60–2.48 (m, 2H), 2.06–1.88 (m, 2H), 1.33 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 143.1, 138.9, 134.6, 128.6, 128.4, 128.1, 125.7 124.4, 121.7, 118.7, 111.0, 102.5, 40.7, 39.8, 34.2, 23.4 ppm;

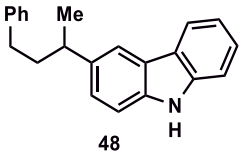
HRMS (ESI): Calcd for $C_{18}H_{20}N$ $[M+H]^+$: 250.1590; found 250.1592.



47

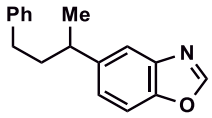
On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 5-bromo-benzofuran, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, CH_2Cl_2) afforded crude **47**. Further purification by preparative TLC (silica gel, Petroleum ether) afforded **47** (33.0 mg, 66%).

Physical state: colorless oil;
TLC: R_f = 0.35 (silica gel, Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, *J* = 2.2 Hz, 1H), 7.45 (dt, *J* = 8.5, 0.8 Hz, 1H), 7.43 (dd, *J* = 1.5, 1.0 Hz, 1H), 7.29–7.24 (m, 2H), 7.20–7.16 (m, 1H), 7.16–7.12 (m, 3H), 6.74 (dd, *J* = 2.2, 1.0 Hz, 1H), 2.89–2.78 (m, 1H), 2.59–2.45 (m, 2H), 2.02–1.91 (m, 2H), 1.32 (d, *J* = 6.9 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 153.8, 145.2, 142.7, 142.0, 128.5, 128.4, 127.6, 125.8, 123.7, 119.3, 111.3, 106.7, 40.6, 39.6, 34.1, 23.3 ppm;
HRMS (ESI): Calcd for $C_{18}H_{19}O$ $[M+H]^+$: 251.1430; found 251.1430.



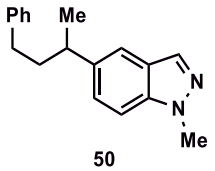
48

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 3-bromo-9H-carbazole, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 1:19, EtOAc:Petroleum ether) afforded **48** (37.1 mg, 62%).
Physical state: white solid;
Melting point: 129.0–131.01°C;
TLC: R_f = 0.62 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11–8.06 (m, 1H), 7.96 (*br*, s, 1H), 7.91 (d, *J* = 1.6 Hz, 1H), 7.45–7.40 (m, 2H), 7.39–7.36 (m, 1H), 7.30-7.25 (m, 3H), 7.25-7.21 (m, 1H), 7.20–7.13 (m, 3H), 2.98–2.85 (m, 1H), 2.64–2.50 (m, 2H), 2.11–1.95 (m, 2H), 1.39 (d, *J* = 7.0 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 142.9, 140.0, 138.7, 138.2, 128.6, 128.4, 125.8, 125.7, 125.4, 123.6, 123.5, 120.4, 119.4, 118.5, 110.7, 110.6, 40.7, 39.8, 34.2, 23.4 ppm;
HRMS (ESI): Calcd for $C_{22}H_{22}N$ $[M+H]^+$: 300.1747; found 300.1747.



49

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 5-bromo-1,3-benzoxazole, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 1:20 EtOAc:Petroleum Ether) afforded crude **49**. Further purification by preparative TLC (silica gel, 1:15, EtOAc:Petroleum ether) followed by preparative HPLC (85% to 90% CH_3CN/water) afforded **49** (33.7 mg, 67%).
Physical state: yellow oil;
TLC: R_f = 0.34 (silica gel, 1:10, EtOAc:Petroleum Ether, UV);
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (s, 1H), 7.63 (d, *J* = 1.7 Hz, 1H), 7.52 (dd, *J* = 8.4, 0.6 Hz, 1H), 7.29–7.22 (m, 3H), 7.19–7.14 (m, 1H), 7.14–7.10 (m, 2H), 2.92–2.81 (m, 1H), 2.58–2.45 (m, 2H), 2.02–1.92 (m, 2H), 1.33 (d, *J* = 6.9 Hz, 3H) ppm;
^{13}C NMR (101 MHz, $CDCl_3$): δ 152.9, 148.6, 144.3, 142.4, 140.4, 128.5, 128.4, 125.8, 125.1, 118.7, 110.7, 40.4, 39.6, 34.0, 23.1 ppm;
HRMS (ESI): Calcd for $C_{17}H_{18}NO$ $[M+H]^+$: 252.1383; found 252.1383.



50

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-phenylbutan-2-ol, 5-bromo-1-methyl-benzoimidazole, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:4 EtOAc:Petroleum Ether) afforded crude **50**. Further purification by preparative TLC (silica gel, 1:4, EtOAc:Petroleum ether) followed by preparative HPLC (85% to 95% CH_3CN/water) afforded **50** (31.2 mg, 59%).

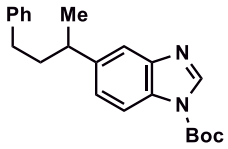
Physical state: white amorphous solid;

TLC: R_f = 0.33 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 0.9 Hz, 1H), 7.54–7.52 (m, 1H), 7.36 (dt, *J* = 8.7, 0.9 Hz, 1H), 7.30–7.23 (m, 3H), 7.19–7.14 (m, 1H), 7.14 –7.10 (m, 2H), 4.07 (s, 3H), 2.92–2.78 (m, 1H), 2.60–2.45 (m, 2H), 2.04–1.91 (m, 2H), 1.33 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.6, 139.7, 139.1, 132.4, 128.5, 128.4, 126.4, 125.8, 124.4, 118.6, 109.1, 40.4, 39.5, 35.7, 34.1, 23.1 ppm;

HRMS (ESI): Calcd for $C_{18}H_{21}N_2$ $[M+H]^+$: 265.1699; found 265.1700.



51

On 0.2 mmol scale, without treatment by hydrogen peroxide, **General Procedure A** was followed with 4-phenylbutan-2-ol, *tert*-butyl 5-bromo-benzimidazole-1-carboxylate, and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:9 EtOAc:Petroleum Ether) afforded crude **51**. Further purification by preparative TLC (silica gel, 1:4, EtOAc:Petroleum ether) followed by preparative HPLC (90% to 95% CH_3CN/water) afforded **51** (34.3 mg, 49%).

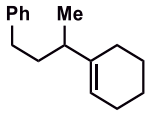
Physical state: colorless oil;

TLC: R_f = 0.46 (silica gel, 1:4, EtOAc:Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 7.91 (d, *J* = 8.4 Hz, 1H), 7.64 (d, *J* = 1.5 Hz, 1H), 7.28–7.23 (m, 3H), 7.19–7.14 (m, 1H), 7.14–7.10 (m, 2H), 2.92–2.81 (m, 1H), 2.56–2.45 (m, 2H), 2.02–1.92 (m, 2H), 1.70 (s, 9H), 1.33 (d, *J* = 7.0 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 148.2, 144.4, 144.0, 142.6, 142.2, 129.8, 128.5, 128.4, 125.8, 124.9, 118.7, 114.3, 85.7, 40.4, 39.6, 34.0, 28.2, 23.1 ppm;

HRMS (ESI): Calcd for $C_{22}H_{27}N_2O_2$ $[M+H]^+$: 351.2067; found 351.2062.



52

On 0.2 mmol scale, **General Procedure A** was followed with 4-phenylbutan-2-ol, 1-bromocyclohex-1-ene, and triphenylphosphine (7.0 equiv). Purification by column chromatography

(silica gel, 1:19 CH_2Cl_2:Petroleum Ether) afforded crude **52**. Further purification by preparative TLC (silica gel, pentane) afforded **52** (23.6 mg, 55%).

Physical state: colorless oil;

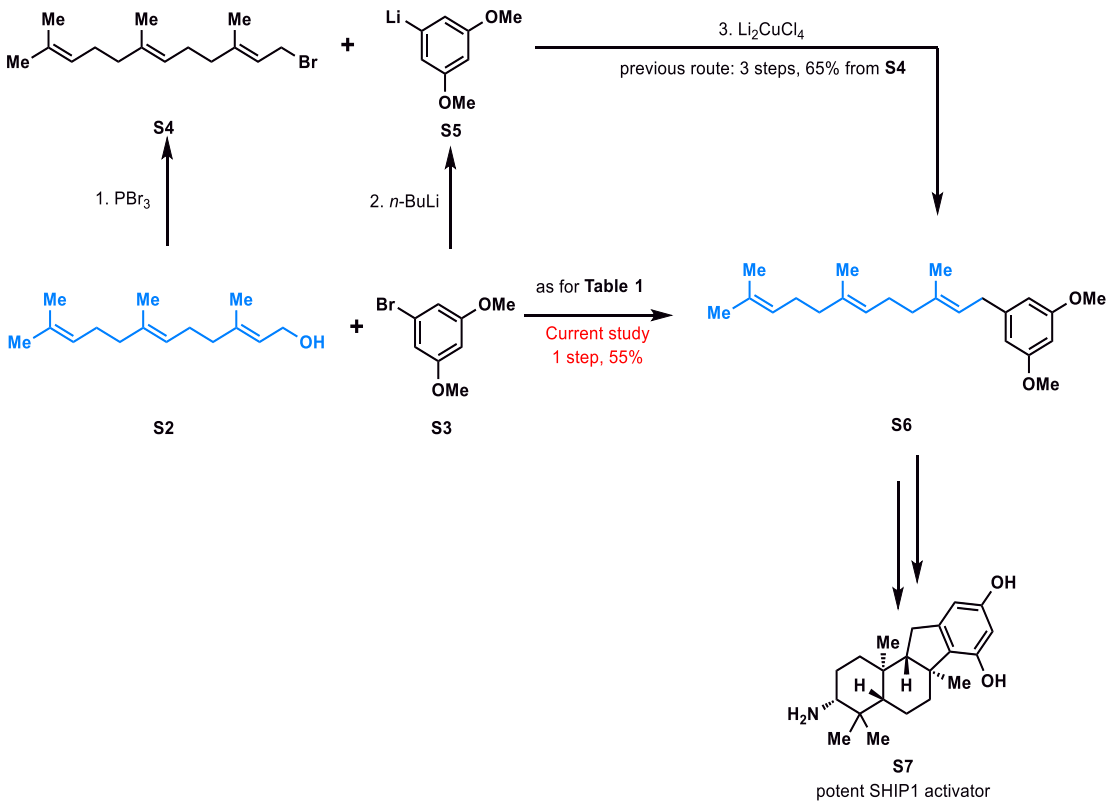
TLC: R_f = 0.70 (silica gel, Petroleum Ether, UV);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29-7.23 (m, 2H), 7.19−7.12 (m, 3H), 5.45−5.40 (m, 1H), 2.58−2.46 (m, 2H), 2.11–2.03 (m, 1H), 2.03−1.98 (m, 2H), 1.95−1.83 (m, 2H), 1.74−1.65 (m, 1H), 1.65−1.50 (m, 5H), 1.01 (d, *J* = 6.9 Hz, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 143.3, 141.6, 128.5, 128.4, 125.6, 120.9, 41.2, 37.0, 34.1, 25.4, 24. 9, 23.3, 23.1, 19.9 ppm;

HRMS (ESI): Calcd for $C_{16}H_{23}$ $[M+H]^+$: 215.1794; found 215.1796.

An Application of the Electrochemical Dehydroxylative Arylation[1]



On 0.2 mmol scale, **General Procedure A** was followed with (*E*,*E*)-farnesol (**S2**), 1-bromo-3,5-dimethoxybenzene, and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, 0:100 to 1:9 EtOAc:Petroleum Ether) afforded crude **S6**. Further purification by preparative HPLC (80% to 91% CH_3CN/water) afforded **S6** (37.7 mg, 55%).

Physical state: colorless oil;

TLC: R_f = 0.42 (silica gel, 3:47 EtOAc:Petroleum Ether, UV);

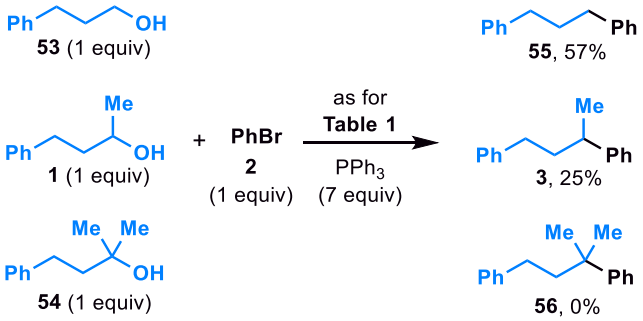
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.35 (d, *J* = 2.3 Hz, 2H), 6.30 (t, *J* = 2.3 Hz, 1H), 5.36–5.30 (m, 1H), 5.16–5.06 (m, 2H), 3.77 (s, 6H), 3.30 (d, *J* = 7.3 Hz, 2H), 2.16–2.01 (m, 6H), 2.01–1.94 (m, 2H), 1.71 (d, *J* = 0.8 Hz, 3H), 1.68 (d, *J* = 1.3 Hz, 3H), 1.60 (s, 6H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$): δ 160.9, 144.4, 136.7, 135.3, 131.4, 124.5, 124.2, 122.8, 106.6, 97.8, 55.4, 39.9, 34.6, 26.9, 26.8, 25.9, 17.8, 16.4, 16.2 ppm;

HRMS (ESI): Calcd for $C_{23}H_{35}O_2$ $[M+H]^+$: 343.2632; found 343.2631.

Mechanistic Studies

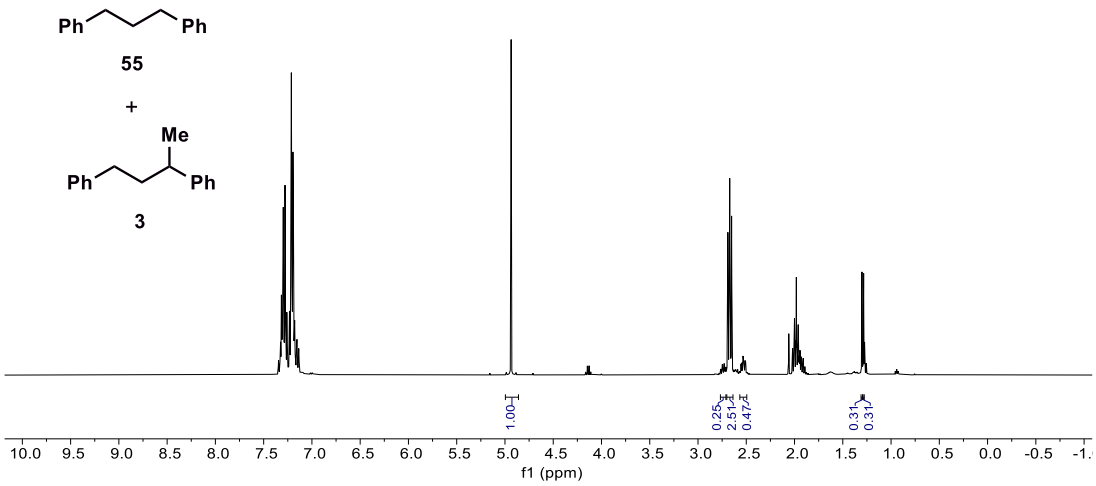
1. Relative reactivity study

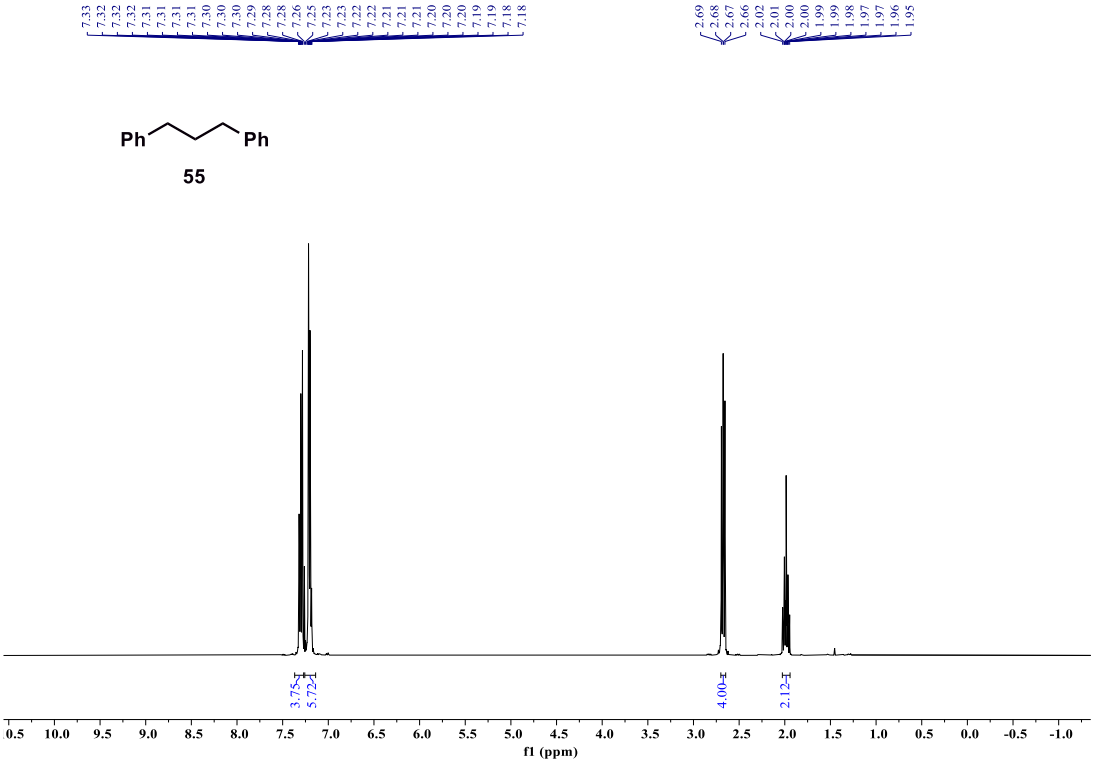


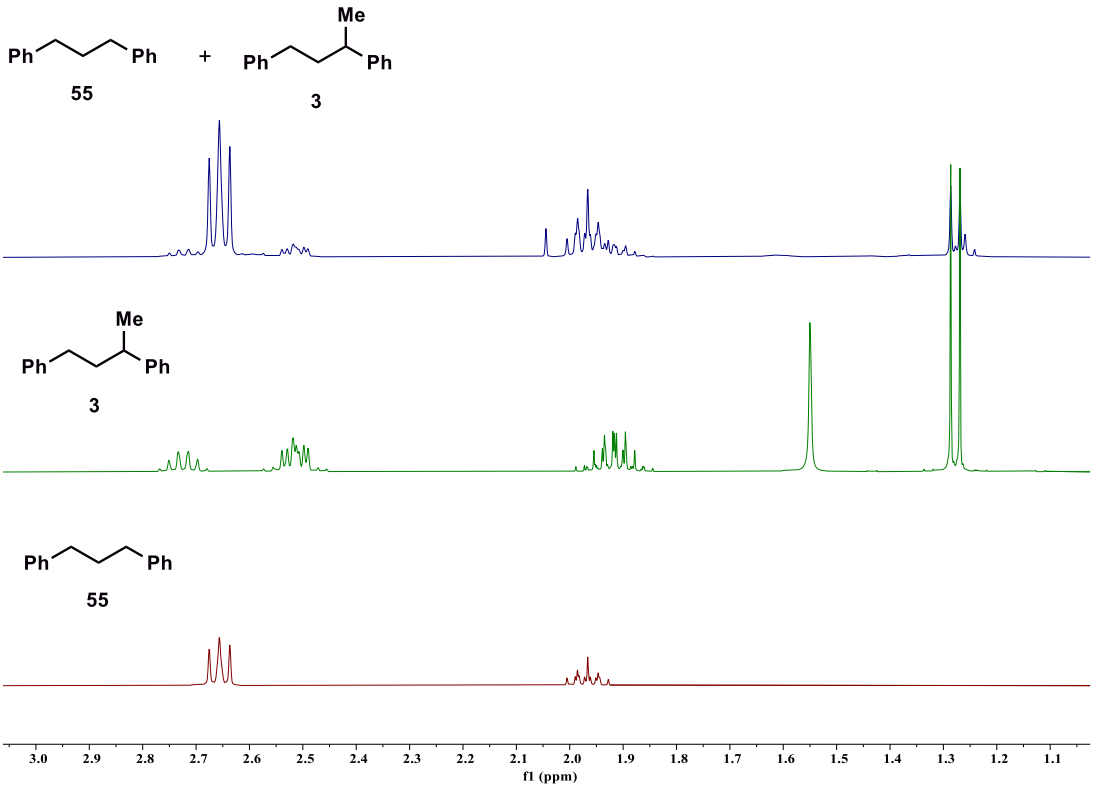
On 0.2 mmol scale, **General Procedure A** was followed with **53** (1.0 equiv), **1** (1.0 equiv), **54** (1.0 equiv), bromobenzene (1.0 equiv), and triphenylphosphine (7.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) followed by preparative TLC (silica gel, 1:1, CH_2Cl_2: Petroleum ether) afforded a mixture of **55** and **3**.

The yields of **55** and **3** was determined by ^{1}H NMR with CH_2Br_2 (0.1 mmol) as internal standard.

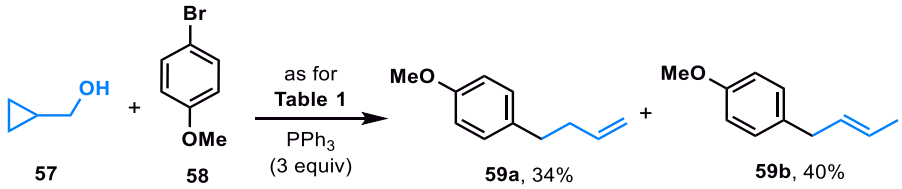
The standard sample of **55** was purchased from Alfa Aesar.




Ph
Ph
55
3.75
5.72
4.00
2.12
f1 (ppm)


Ph
Ph
55
+
Me
Ph
Ph
3
Me
Ph
Ph
3
Ph
Ph
55
f1 (ppm)

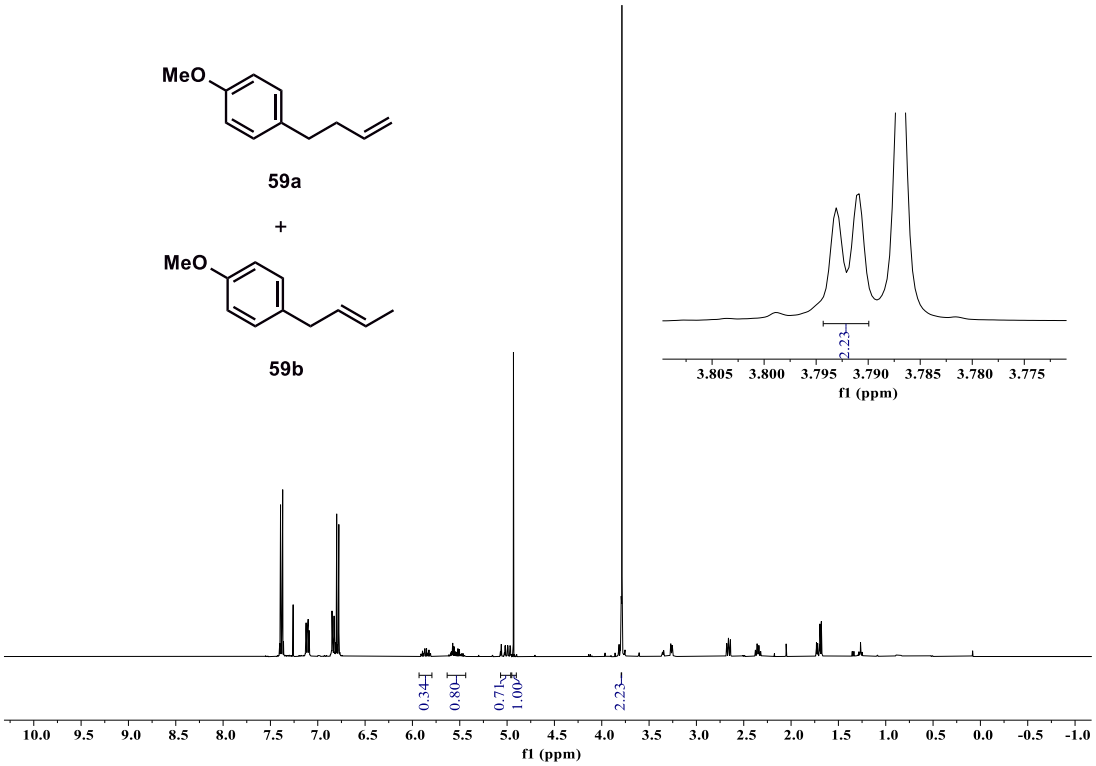
2. Radical ring opening

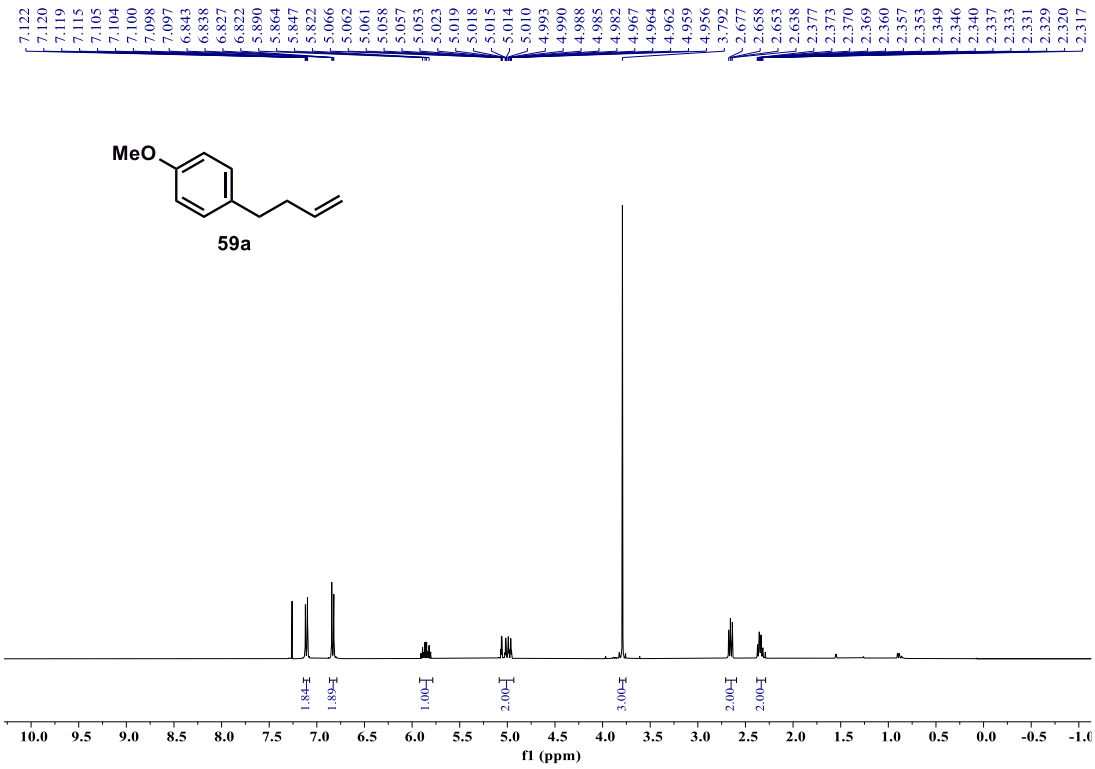


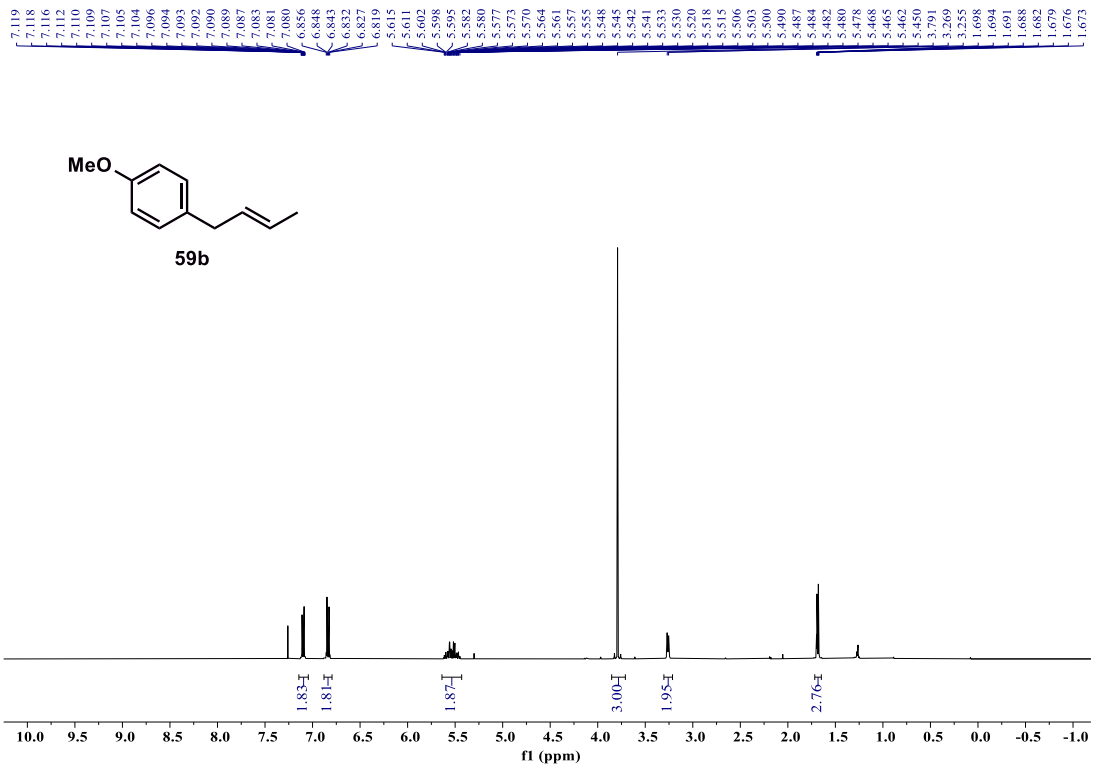
On 0.2 mmol scale, **General Procedure A** was followed with **57**, 4-bromoanisole (**58**), and triphenylphosphine (3.0 equiv). Purification by column chromatography (silica gel, Petroleum Ether) afforded a mixture of **59a** and **59b**.

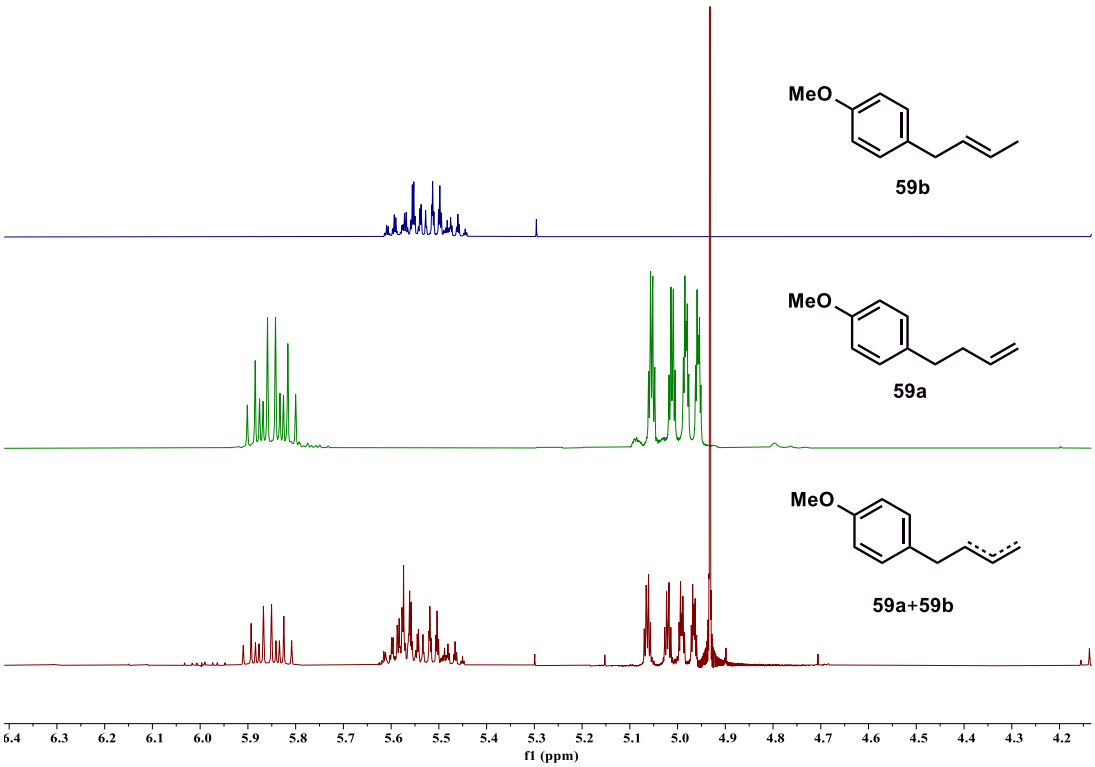
The yield of **59a** and **59b** was determined by ^{1}H NMR with CH_2Br_2 (0.1 mmol) as internal standard.

The standard sample of **59a** was prepared according to Konig's procedure;[2] the standard sample of **59b** was prepared according to Gong's procedure.[3]



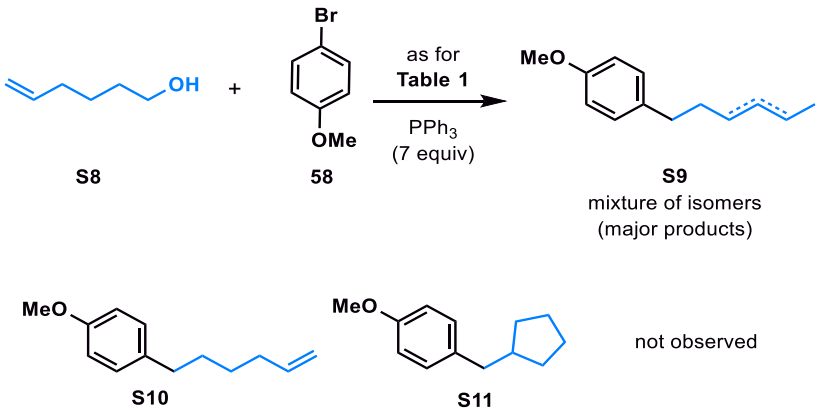

MeO
59a
f1 (ppm)


MeO
59b
f1 (ppm)

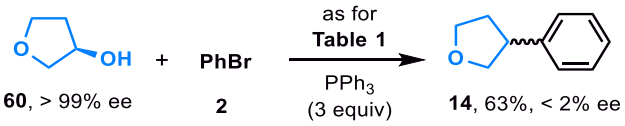


3. 5-*exo*-trig type cyclization

It was found that the monosubstituted alkenes could easily undergo migration before the 5-*exo*-trig cyclization. The desired cyclized product was not observed in this reaction conditions.



4. Enantiopurity erosion



The *ee* value of **60** was determined by its benzoate ester.

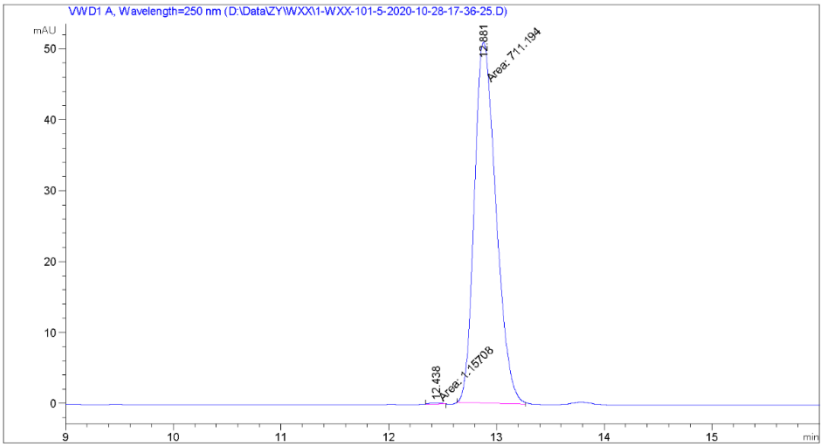
*Method for determining the ee of the **R-60** benzoate and **Rac-60** benzoate:*

Column: Chiralpak® IE

Dimensions: 4.6 ×250 mm

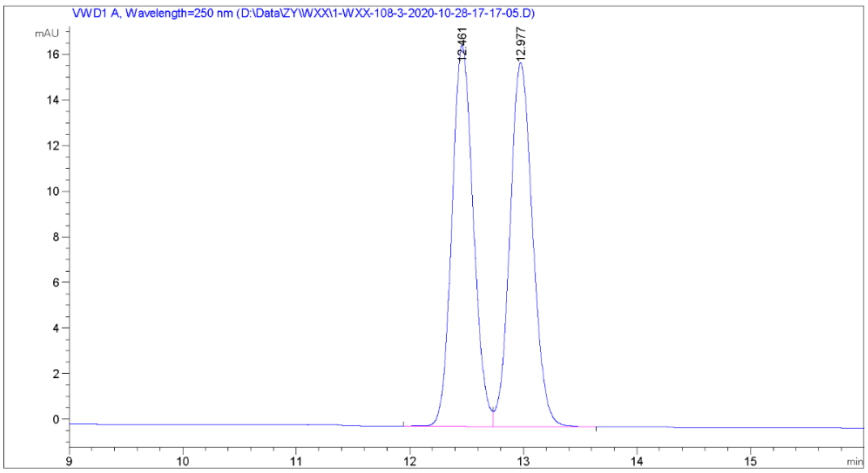
Eluent: *n*-hexane:IPA = 99:1

Flow rate: 1.0 mL/min



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.438	MM	0.1308	1.15708	1.47455e-1	0.1624
2	12.881	MM	0.2328	711.19403	50.92102	99.8376
Totals :				712.35111	51.06848	



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.461	BV	0.2025	218.47711	16.71409	49.6626
2	12.977	VB	0.2147	221.44586	15.97765	50.3374
Totals :				439.92297	32.69174	

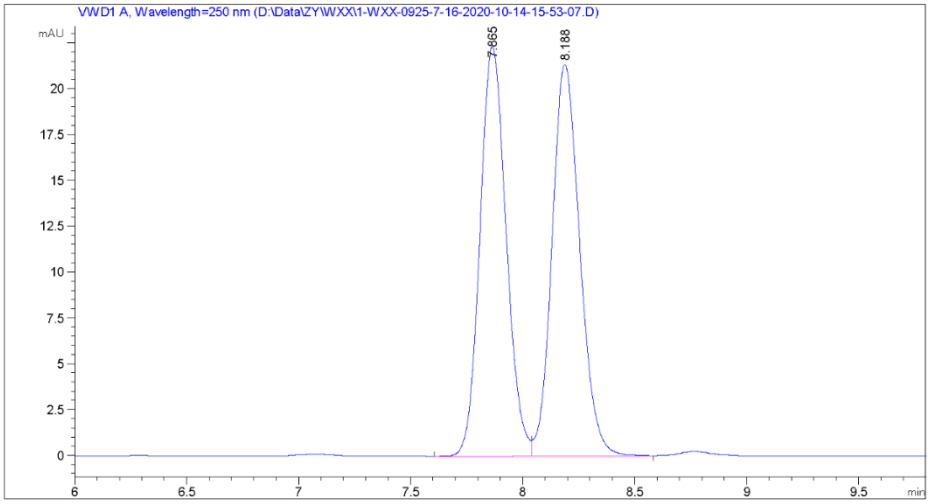
Method for determining the ee value of the coupling products from ***R-60*** *and* ***Rac-60****:*

Column: Chiralpak® IE

Dimensions: 4.6 ×250 mm

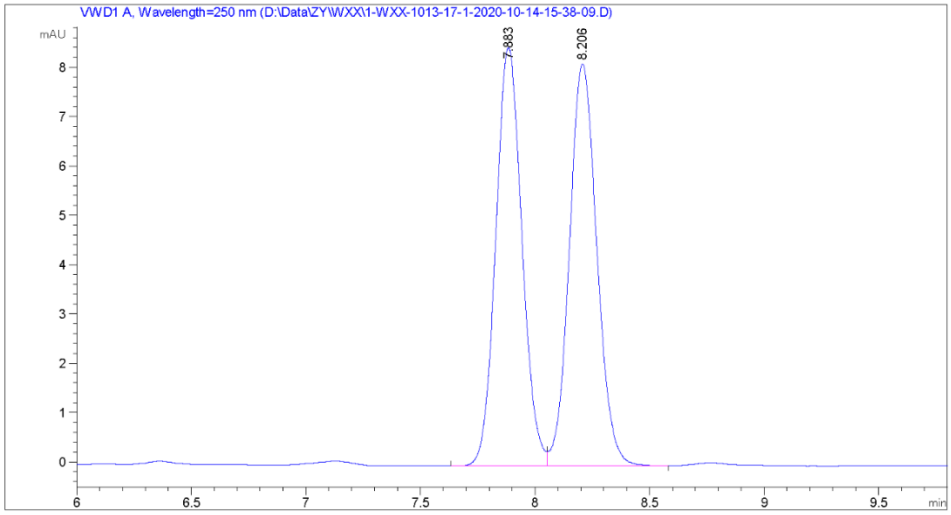
Eluent: *n*-hexane:IPA = 99.5:0.5

Flow rate: 1.0 mL/min



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.865	BV	0.1267	181.28423	22.34322	49.6946
2	8.188	VB	0.1333	183.51257	21.35150	50.3054
Totals :				364.79680	43.69472	



Signal 1: VWD1 A, Wavelength=250 nm

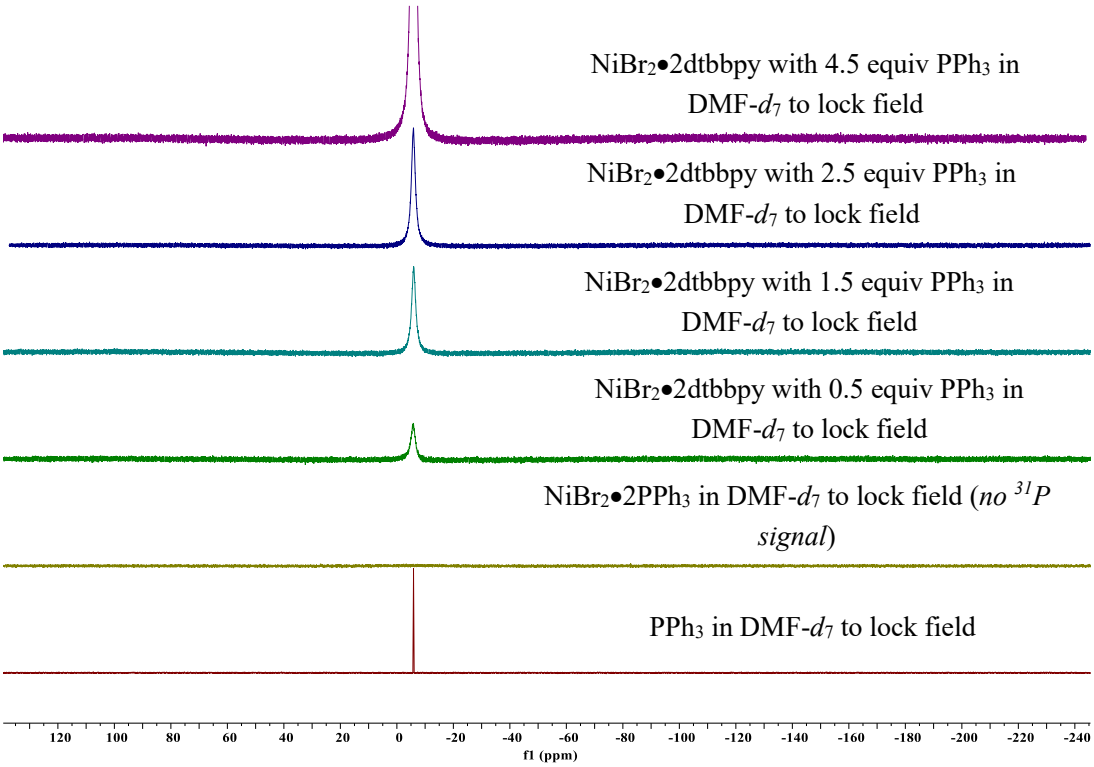
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.883	BV	0.1224	67.17370	8.47946	49.7633
2	8.206	VB	0.1290	67.81274	8.15273	50.2367
Totals :				134.98644	16.63220	

5. Ligand exchange experiments

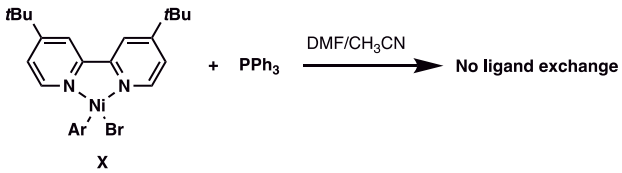
A screw-capped culture tube charged with a magnetic stir bar was moved into a glove box. $NiBr_2$ (21.9 mg, 0.1 mmol), dtbbpy (**L1**, 53.9 mg, 0.2 mmol), and DMF-d_7 (1 mL) were added. The resulting mixture was stirred at 100 °C for 2 hours and then cooled to room temperature.

PPh_3 was added, and the mixture was stirred for additional 2 hours at room temperature.

The reaction mixture was detected by ^{31}P NMR. ^{31}P NMR (162 MHz, DMF-d_7) δ –5.87 (PPh_3). It was shown that the addition of PPh_3 didn't result in the ligand exchange.



Additionally, according to the study from the group of Xie,[4] the ligand exchange between Ni(II) aryl complex and PPh_3 was deemed unlikely to occur.



Moreover, given that: 1) this coupling reaction did not proceed in the absence of dtbbpy (**L1**, Table 1, entry 3); 2) not all of the bipyridyl ligands worked fine (e.g. **L6** in Table S14), the possibility that ligand exchange occurred in the reaction conditions could be further ruled out.

Cyclic Voltammetry Data

All cyclic voltammetry studies were performed in a glovebox with the IKA ElectraSyn 2.0.
Measurements were performed in 0.1 M $LiClO_4$ in anhydrous NMP.
Reference electrode: Ag/AgCl (Ag wire in aqueous 3 M KCl);
Working electrode: glassy carbon disc electrode;
Counter electrode: platinum plate electrode
Scan rate = 100 mV/s.

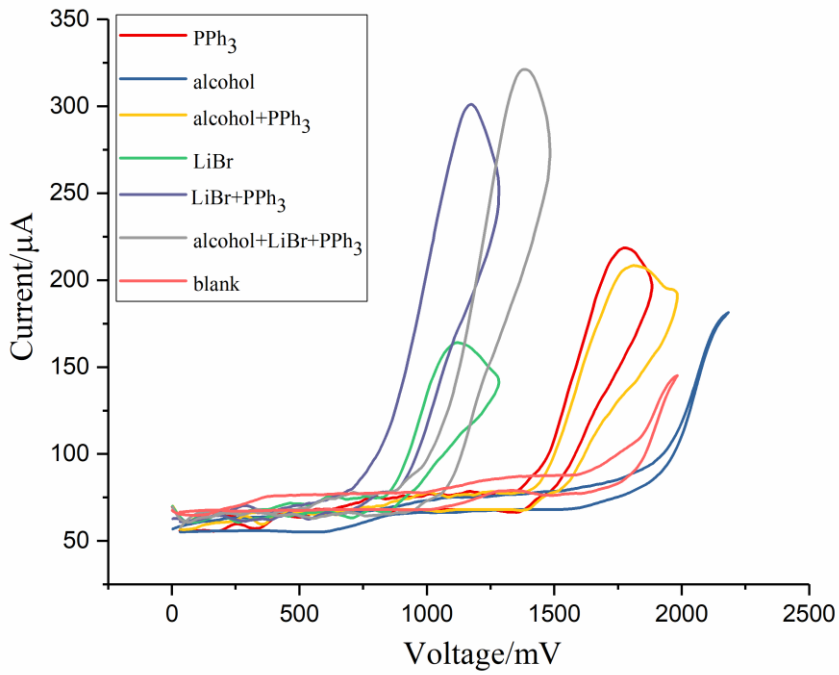


Figure S6. Cyclic voltammetry studies

References

[1] Meimetis, L. G.; Nodwell, M.; Yang, L.; Wang, X.; Wu, J.; Harwig, C.; Stenton, G. R.; Mackenzie, L. F.; MacRury, T.; Patrick, B. O.; Ming Lum, A.; Ong, C. J.; Krystal, G.; Mui, A. L. F.; Andersen, R. J., Synthesis of SHIP1-Activating Analogs of the Sponge Meroterpenoid Pelorol. *Eur. J. Org. Chem.* **2012**, *2012*, 5195–5207.

[2] Meng, Q. Y.; Schirmer, T. E.; Katou, K.; König, B., Controllable Isomerization of Alkenes by Dual Visible-Light-Cobalt Catalysis. *Angew. Chem. Int. Ed.* **2019**, *58*, 5723–5728.

[3] Cui, X.; Wang, S.; Zhang, Y.; Deng, W.; Qian, Q.; Gong, H., Nickel-catalyzed reductive allylation of aryl bromides with allylic acetates. *Org. Biomol. Chem.* **2013**, *11*, 3094–3097.

[4] Ruzi, R.; Liu, K.; Zhu, C.; Xie, J., Upgrading ketone synthesis direct from carboxylic acids and organohalides. *Nat. Commun.* **2020**, *11*, 3312.

Single Crystal X-ray Diffraction Data

X-ray crystallographic data for 31a

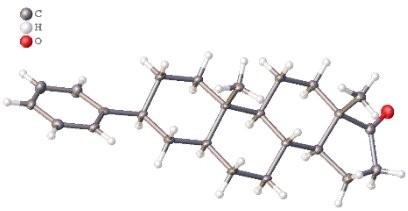


Table S16. Crystal data and structure refinement for **31a**.

Identification code	CCDC 2049258
Empirical formula	$C_{25}H_{34}O$
Formula weight	350.52
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	10.4271(2)
b/Å	6.24210(10)
c/Å	15.6044(4)
α/°	90
β/°	105.739(2)
γ/°	90
Volume/Å^3	977.56(4)
Z	2
ρ_{calc}g/cm^3	1.191
μ/mm^{-1}	0.527
F(000)	384.0
Crystal size/mm^3	0.34 × 0.05 × 0.03
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.81 to 147.454
Index ranges	$-11 \leq h \leq 12$, $-7 \leq k \leq 7$, $-19 \leq l \leq 19$
Reflections collected	17733
Independent reflections	3857 [R_{int} = 0.1191, R_{sigma} = 0.0665]
Data/restraints/parameters	3857/1/237
Goodness-of-fit on F^2	1.095
Final R indexes [I>=2σ (I)]	R_1 = 0.0481, wR_2 = 0.1258
Final R indexes [all data]	R_1 = 0.0541, wR_2 = 0.1323
Largest diff. peak/hole / e Å^{-3}	0.19/-0.29

Flack parameter	0.0(3)

Table S17. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **31**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O25	2772.4(19)	1168(4)	-105.0(13)	28.0(5)
C5	3044(2)	5960(4)	2407.0(15)	13.6(5)
C18	2472(2)	6238(4)	6633.3(16)	15.6(5)
C13	2484(2)	4280(4)	2942.7(16)	13.8(5)
C23	2571(2)	8340(4)	6932.1(16)	16.2(5)
C9	3080(2)	2730(4)	368.5(16)	18.4(6)
C4	4165(2)	7280(4)	3018.5(16)	16.0(5)
C21	2217(2)	7254(5)	8336.1(16)	18.6(5)
C22	2447(2)	8849(5)	7781.2(16)	19.1(5)
C7	4181(2)	5992(5)	1071.4(16)	18.8(6)
C6	3512(2)	4786(4)	1689.0(16)	15.7(5)
C1	2027(2)	5303(4)	3720.9(16)	13.8(5)
C15	1407(2)	4386(4)	5166.4(16)	17.8(5)
C10	2382(2)	3526(4)	1050.8(16)	15.9(5)
C2	3240(2)	6526(4)	4314.4(16)	14.7(5)
C16	1636(2)	3540(4)	4297.9(16)	16.6(5)
C12	1405(2)	2870(4)	2328.4(16)	17.0(5)
C11	1858(2)	1820(4)	1569.7(16)	18.4(6)
C19	2250(3)	4661(5)	7208.6(17)	22.1(6)
C3	3732(2)	8264(4)	3789.4(16)	17.2(5)
C14	2637(2)	5567(4)	5732.7(16)	15.3(5)
C17	3009(2)	7374(4)	5184.2(17)	16.7(5)
C24	825(2)	6800(5)	3362.9(17)	19.2(6)
C26	1232(2)	4952(5)	510.8(17)	20.3(6)
C20	2114(3)	5156(5)	8046.9(18)	23.9(6)
C8	4221(2)	4272(5)	375.9(17)	21.1(6)

X-ray crystallographic data for 36

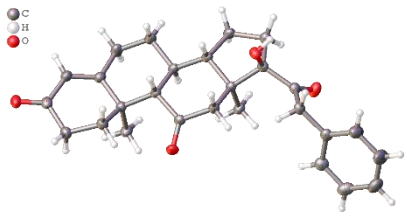


Table S18. Crystal data and structure refinement for **36**.

Identification code	CCDC 2049257
Empirical formula	$C_{27}H_{32}O_4$
Formula weight	420.553
Temperature/K	100.00(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	8.0345(1)
b/Å	12.2693(1)
c/Å	22.3318(2)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2201.42(4)
Z	4
ρ_{calc}g/cm^3	1.269
μ/mm^{-1}	0.666
F(000)	906.8
Crystal size/mm^3	0.17 × 0.17 × 0.12
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.92 to 150.3
Index ranges	$-7 \leq h \leq 10$, $-14 \leq k \leq 15$, $-28 \leq l \leq 27$
Reflections collected	25049
Independent reflections	4454 [R_{int} = 0.0383, R_{sigma} = 0.0214]
Data/restraints/parameters	4454/0/283
Goodness-of-fit on F^2	1.046
Final R indexes [I>=2σ (I)]	R_1 = 0.0327, wR_2 = 0.0835
Final R indexes [all data]	R_1 = 0.0337, wR_2 = 0.0844
Largest diff. peak/hole / e Å^{-3}	0.15/-0.19
Flack parameter	-0.13(13)

Table S19. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **36**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O23	5235.7(12)	4310.5(8)	4484.7(4)	24.7(2)
O21	2770.7(12)	7953.5(8)	3739.8(5)	29.3(2)
O22	6419.2(14)	12292.1(8)	4826.9(5)	32.2(2)
O19	4838.9(13)	3230.7(9)	3046.9(4)	32.4(2)
C15	3951.8(16)	7444.2(11)	3933.5(6)	20.9(3)
C4	6920.5(15)	6133.5(11)	3995.4(5)	20.3(3)
C18	4402.0(17)	3677.3(10)	3502.5(6)	23.1(3)
C10	6162.7(19)	11383.8(11)	4613.2(6)	26.0(3)
C1	5571.4(16)	4427.3(11)	3860.6(5)	21.5(3)
C24	5262.1(17)	5832.0(11)	3031.6(6)	24.4(3)
C8	7350.1(16)	9655.7(11)	4240.3(6)	23.4(3)
C17	5328.1(15)	5657.4(10)	3712.4(5)	19.5(2)
C13	5665.9(16)	9232.4(11)	4029.0(5)	20.0(2)
C3	8297.2(16)	5361.7(12)	3779.9(6)	25.2(3)
C9	7519.4(18)	10620.9(12)	4517.2(6)	28.3(3)
C5	7129.0(15)	7348.8(11)	3886.1(6)	20.3(3)
C12	4288.7(17)	9790.2(11)	4398.1(6)	25.1(3)
C14	5585.5(16)	7976.6(10)	4128.7(5)	19.0(2)
C26	1446.4(16)	3005.5(11)	3328.0(6)	25.0(3)
C31	866.9(18)	3565.1(13)	2829.0(6)	29.2(3)
C20	2651.1(16)	3531.4(11)	3755.4(6)	24.3(3)
C6	8685.6(16)	7786.6(11)	4196.1(7)	27.9(3)
C16	3825.4(16)	6216.0(11)	4005.6(6)	21.6(3)
C2	7432.7(16)	4235.2(12)	3720.6(6)	25.5(3)
C30	-279.6(18)	3106.0(15)	2443.6(6)	34.6(3)
C25	5499.4(18)	9517.4(11)	3357.8(5)	26.1(3)
C7	8880.7(17)	9007.1(12)	4089.1(7)	31.9(3)
C11	4462.2(18)	11030.9(11)	4416.3(6)	27.0(3)
C29	-862(2)	2067.8(16)	2548.0(8)	44.1(4)
C27	837(2)	1966.8(14)	3432.7(9)	43.0(4)
C28	-310(3)	1500.9(15)	3042.4(10)	55.3(5)

X-ray crystallographic data for 48

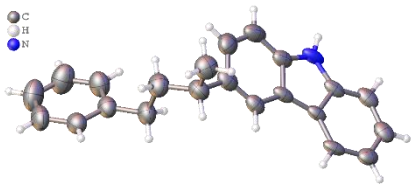


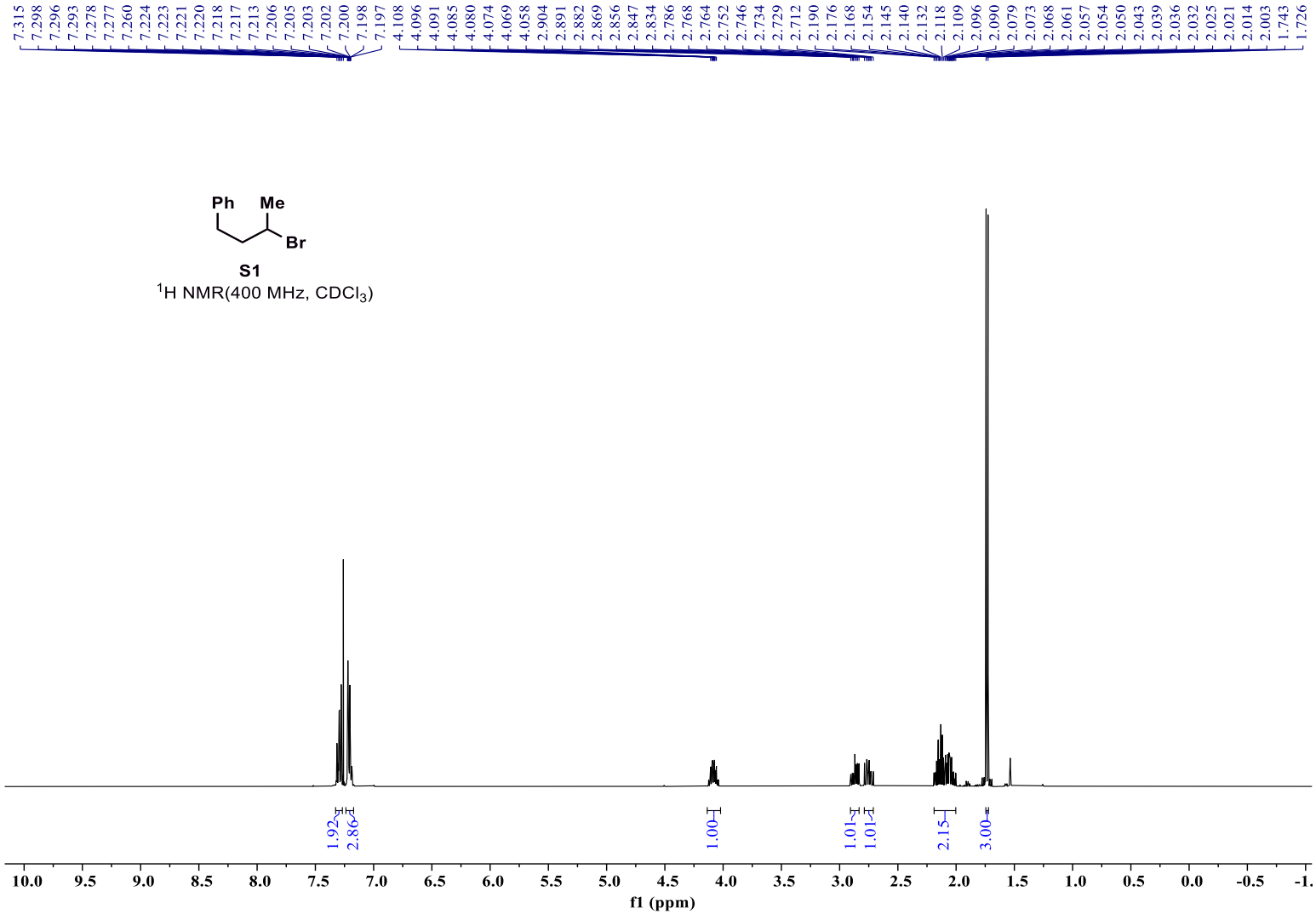
Table S20. Crystal data and structure refinement for **48**.

Identification code	CCDC 2049256
Empirical formula	$C_{22}H_{21}N$
Formula weight	299.40
Temperature/K	100.00(10)
Crystal system	orthorhombic
Space group	$Pca2_1$
a/Å	11.6809(3)
b/Å	19.0963(6)
c/Å	7.3771(2)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1645.55(8)
Z	4
ρ_{calc}g/cm^3	1.208
μ/mm^{-1}	0.526
F(000)	640.0
Crystal size/mm^3	0.41 × 0.32 × 0.02
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.874 to 151.154
Index ranges	$-14 \leq h \leq 14$, $-23 \leq k \leq 23$, $-8 \leq l \leq 7$
Reflections collected	22045
Independent reflections	2752 [R_{int} = 0.0623, R_{sigma} = 0.0243]
Data/restraints/parameters	2752/1/209
Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0604, wR_2 = 0.1608
Final R indexes [all data]	R_1 = 0.0644, wR_2 = 0.1666
Largest diff. peak/hole / e Å^{-3}	0.37/-0.20
Flack parameter	-0.2(5)

Table S21. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **49**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

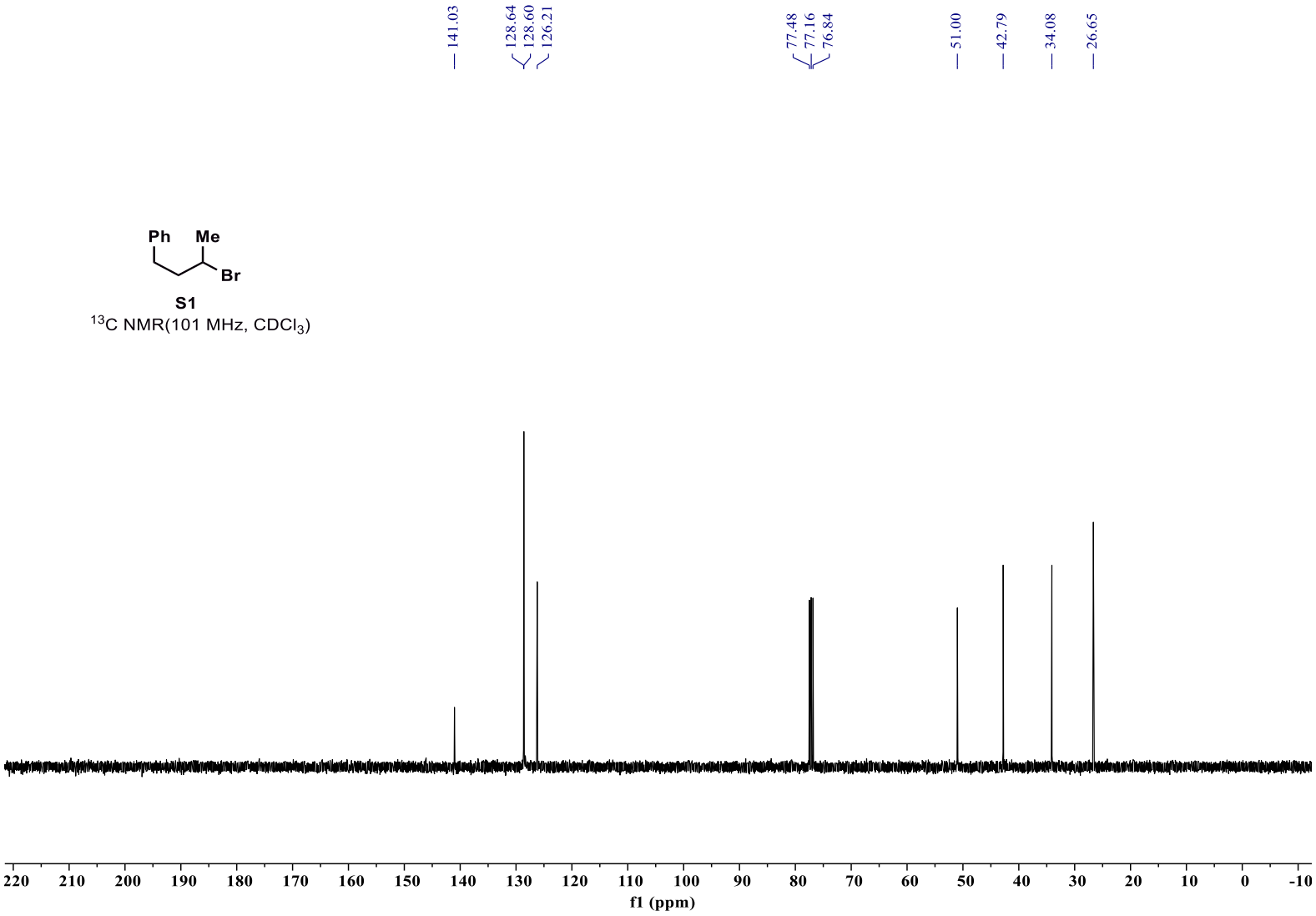
Atom	***x***	***y***	***z***	**U(eq)**
N9	2516(3)	4440.7(17)	8897(4)	47.8(8)
C2	4101(3)	4839.2(19)	7469(5)	37.9(8)
C1	3054(3)	5040(2)	8297(5)	41.0(8)
C3	4164(3)	4093(2)	7588(5)	41.2(8)
C13	4832(3)	5364(2)	6814(5)	44.6(9)
C8	3178(3)	3862(2)	8522(5)	45.8(9)
C4	4995(3)	3601(2)	7062(6)	46.2(9)
C12	4505(4)	6056(2)	6976(5)	51.2(10)
C18	4563(4)	1340(2)	2993(6)	56.2(11)
C10	2731(3)	5729(2)	8439(5)	50.9(10)
C7	3036(4)	3163(3)	8944(6)	60.9(12)
C11	3463(4)	6230(2)	7765(6)	54.6(10)
C5	4875(3)	2917(2)	7440(6)	56.8(11)
C6	3907(4)	2700(2)	8420(6)	63.9(12)
C23	5274(4)	903(2)	2005(7)	60.1(11)
C22	4860(5)	333(2)	1056(8)	72.3(14)
C14	5838(4)	2396(2)	6986(7)	61.4(11)
C16	5012(4)	1962(2)	3993(7)	62.5(12)
C15	5418(5)	1774(2)	5903(7)	71.1(14)
C17	6437(4)	2159(3)	8679(8)	72.8(14)
C19	3420(4)	1169(3)	3032(8)	71.4(14)
C21	3699(6)	186(3)	1105(10)	92.0(19)
C20	3002(5)	604(3)	2066(11)	91.9(19)

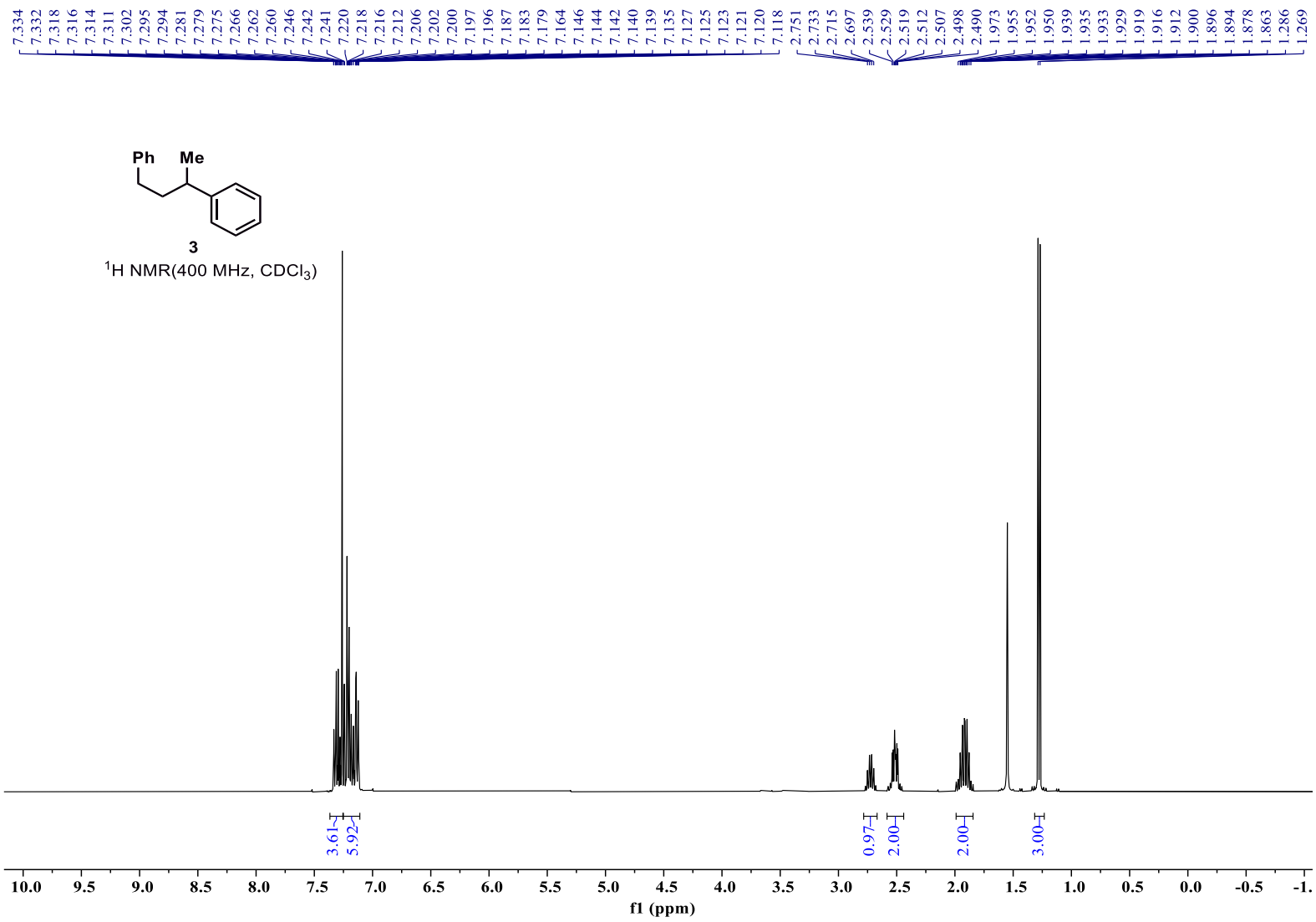
NMR Spectra


7.315 7.298 7.296 7.293 7.278 7.277 7.260 7.224 7.223 7.221 7.220 7.218 7.217 7.213 7.206 7.205 7.203 7.202 7.200 7.198 7.197
4.108 4.096 4.091 4.085 4.080 4.074 4.069 4.058 2.904 2.891 2.882 2.869 2.856 2.847 2.834 2.786 2.768 2.764 2.752 2.746 2.734 2.729 2.712 2.190 2.176 2.168 2.154 2.145 2.140 2.132 2.118 2.109 2.096 2.090 2.079 2.073 2.068 2.061 2.057 2.054 2.050 2.043 2.039 2.036 2.032 2.025 2.021 2.014 2.003 1.743 1.726
Ph
Me
Br
S1
1H NMR(400 MHz, CDCl3)
1.92
2.86
1.00
1.01
1.01
2.15
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

S1

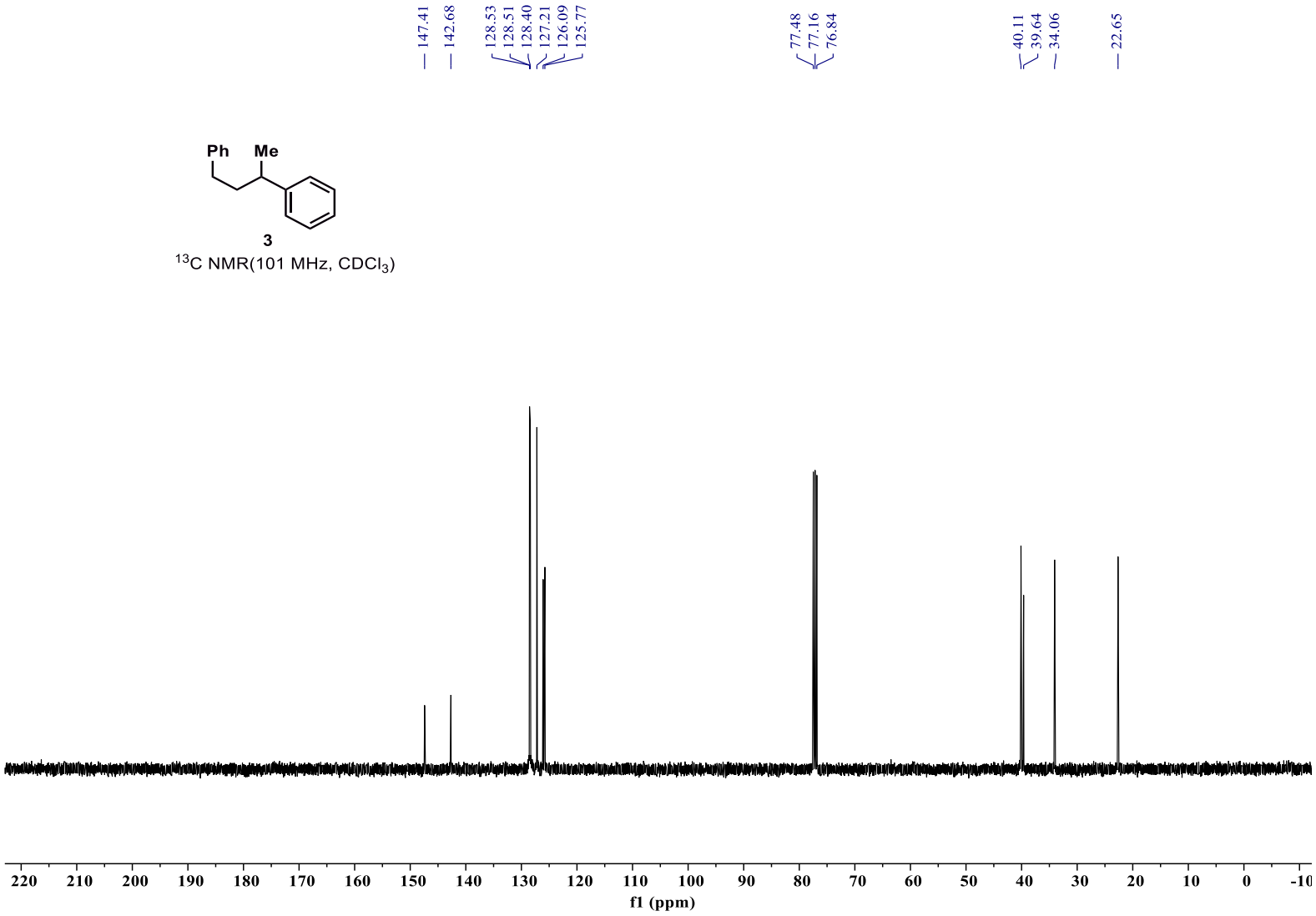
^{13}C NMR(101 MHz, $CDCl_3$)




7.334 7.332 7.318 7.316 7.314 7.311 7.302 7.295 7.294 7.281 7.279 7.275 7.266 7.262 7.260 7.246 7.242 7.241 7.220 7.218 7.216 7.212 7.206 7.202 7.200 7.197 7.196 7.187 7.183 7.179 7.164 7.146 7.144 7.142 7.140 7.139 7.135 7.127 7.125 7.123 7.121 7.120 7.118
2.751 2.733 2.715 2.697 2.539 2.529 2.519 2.512 2.507 2.498 2.490 1.973 1.955 1.952 1.950 1.939 1.935 1.933 1.929 1.919 1.916 1.912 1.900 1.896 1.894 1.878 1.863 1.286 1.269
Ph
Me
3
1H NMR(400 MHz, CDCl3)
3.61
5.92
0.97
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

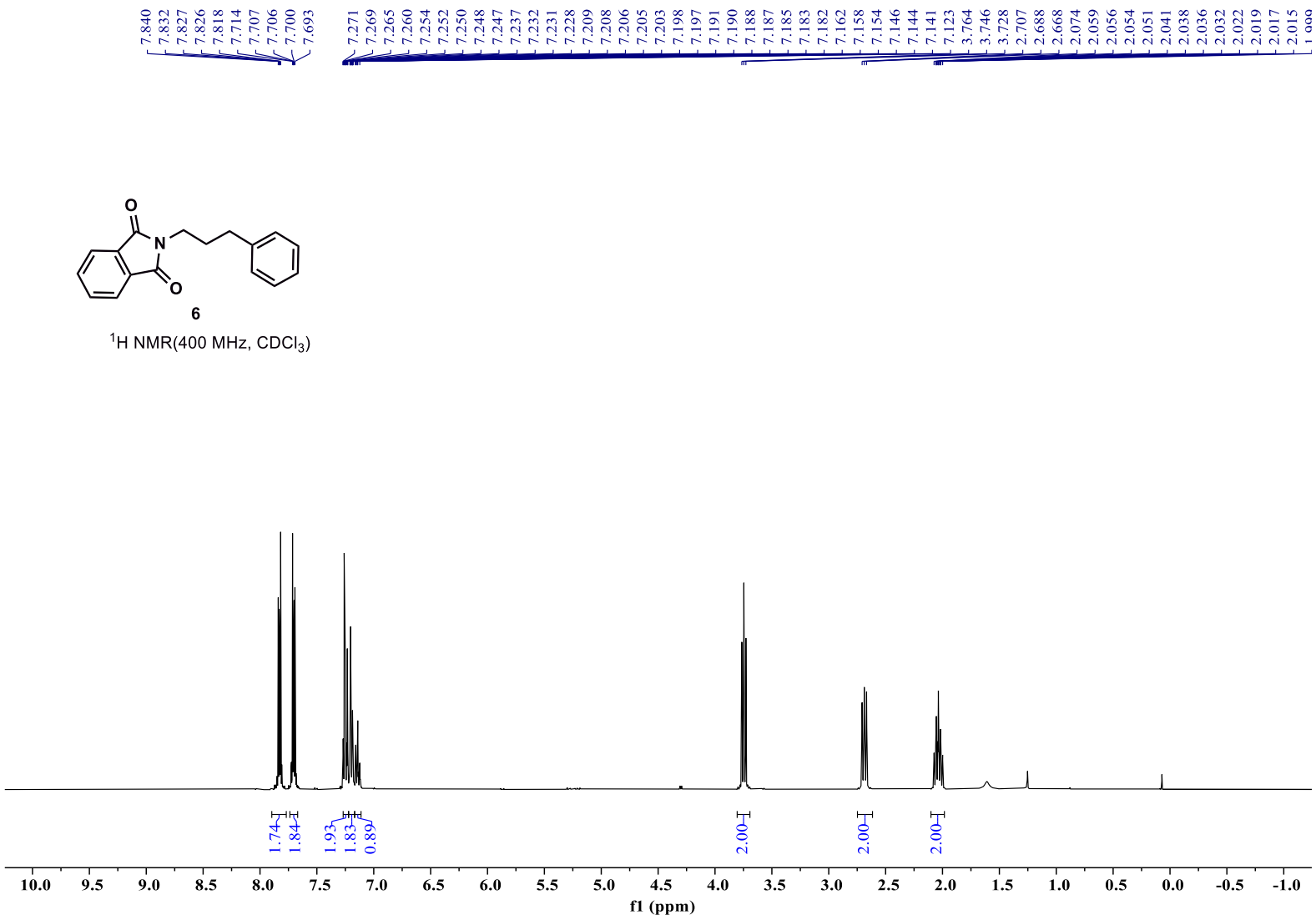
3

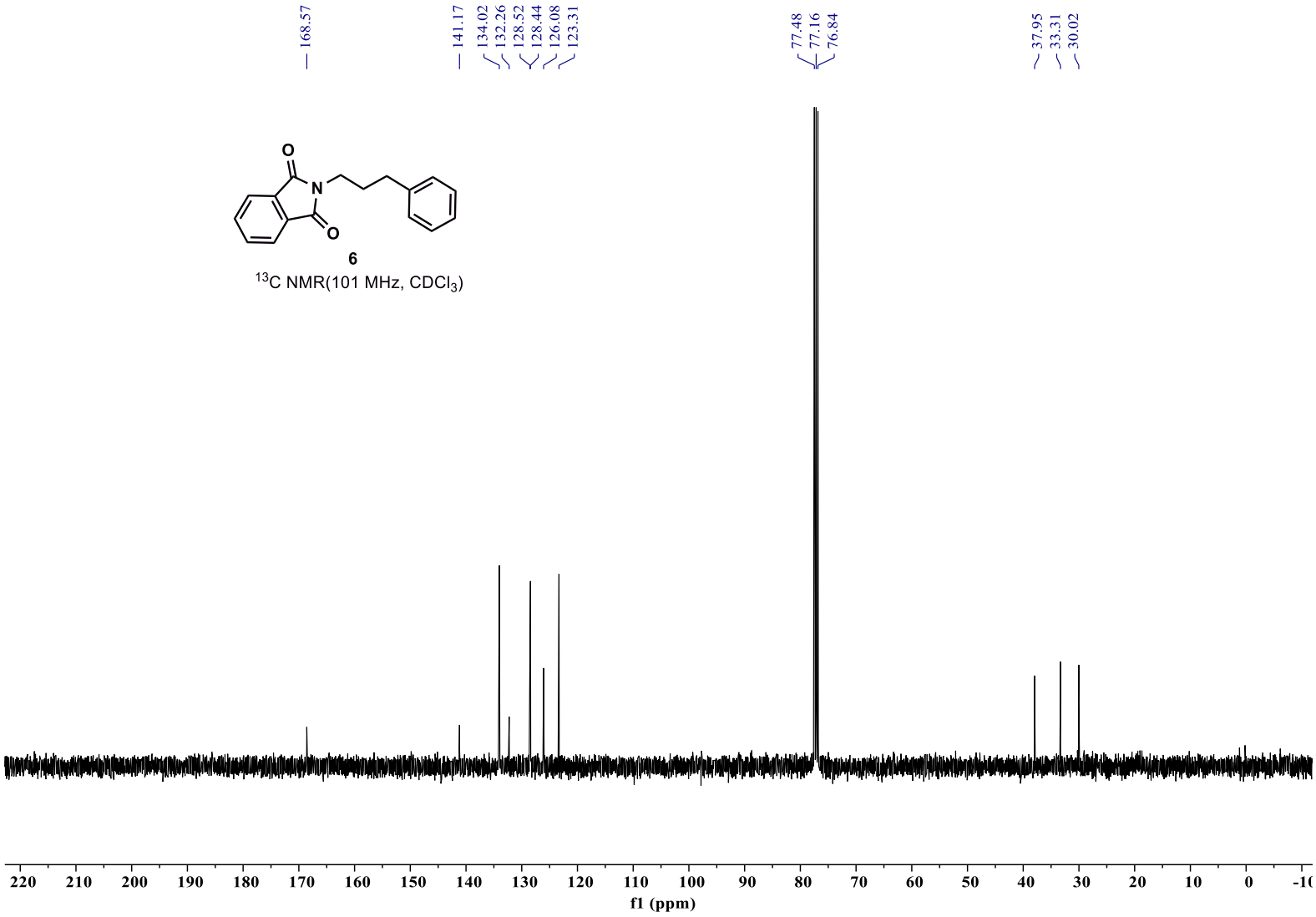
^{13}C NMR(101 MHz, $CDCl_3$)

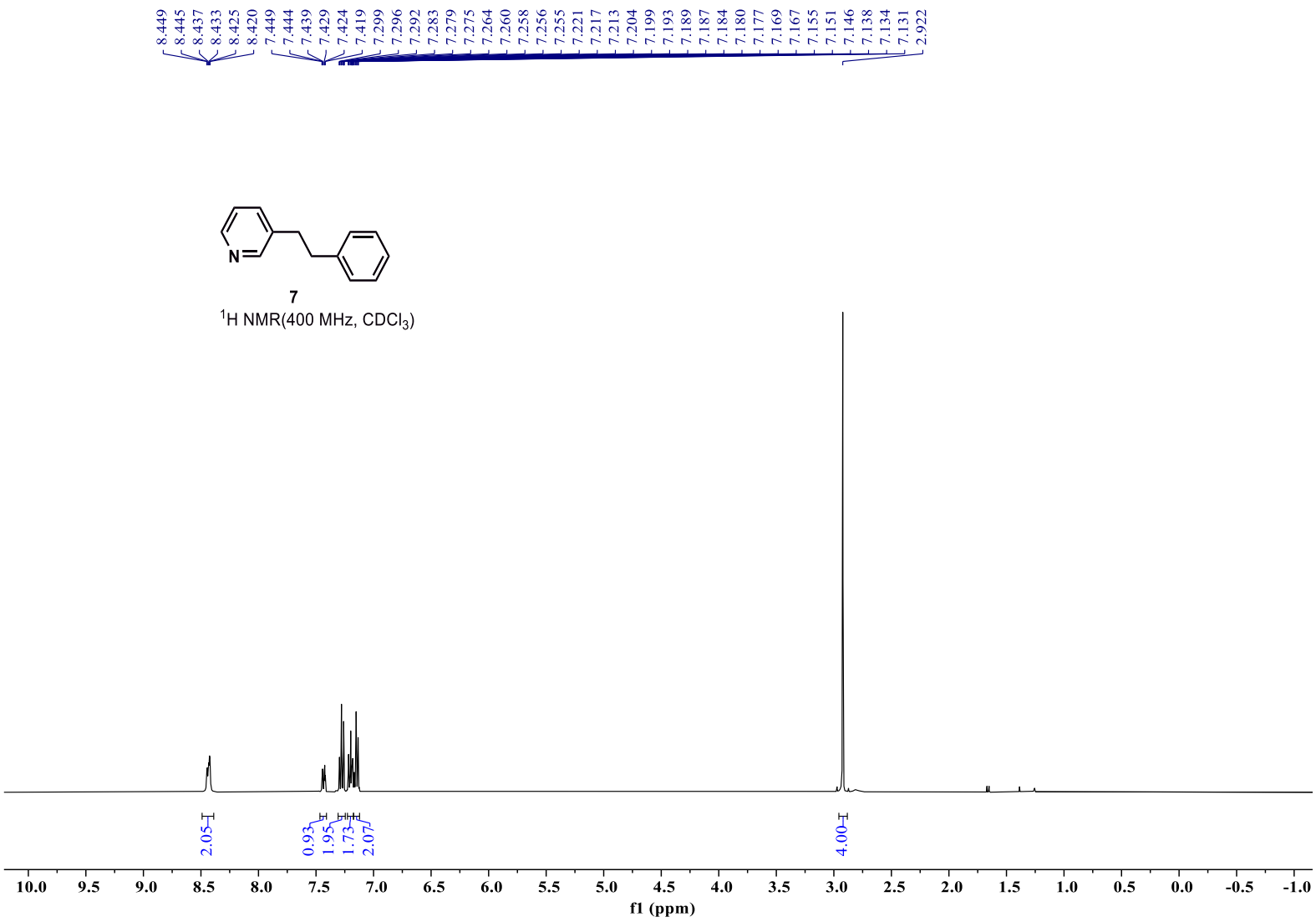


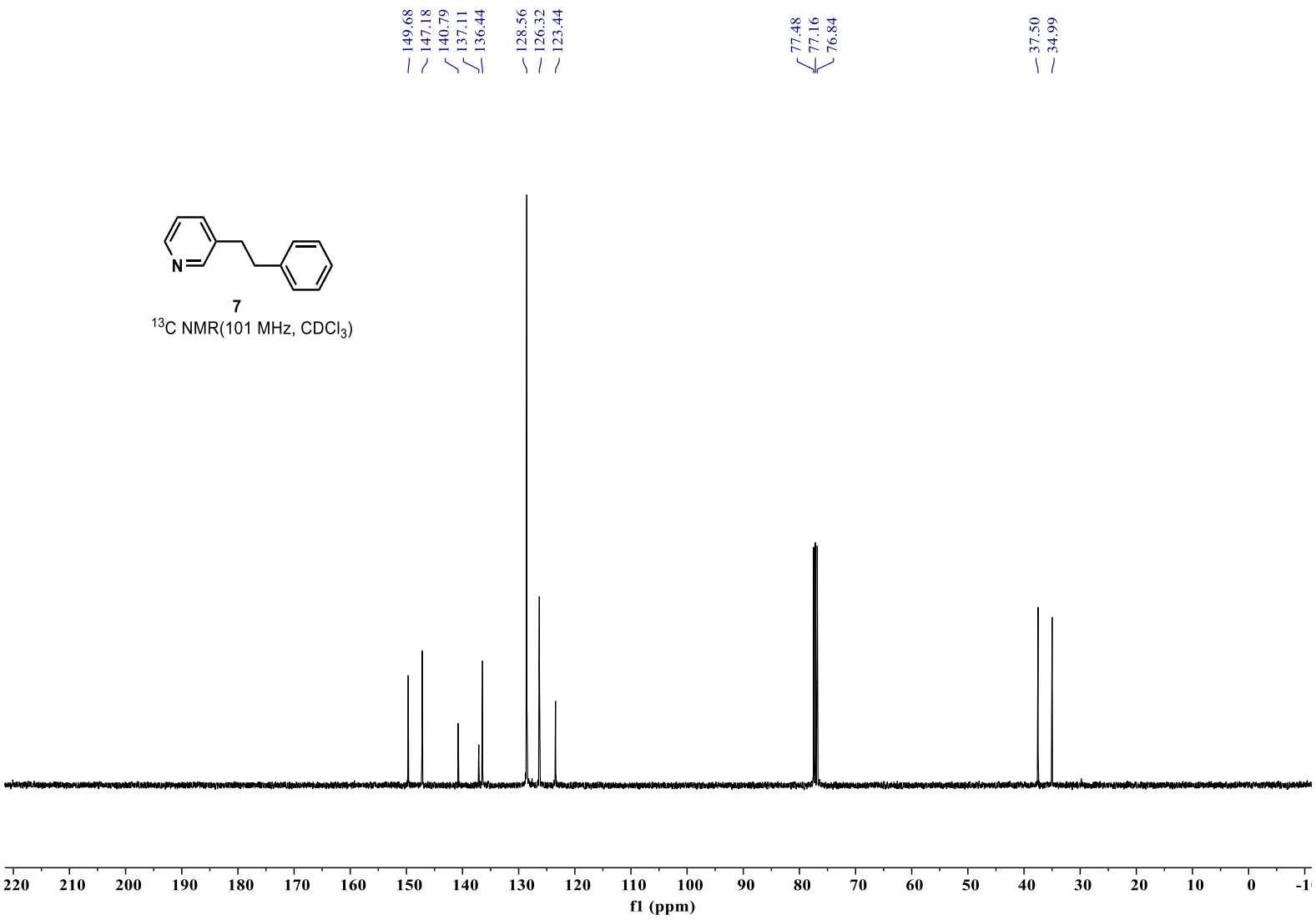
6

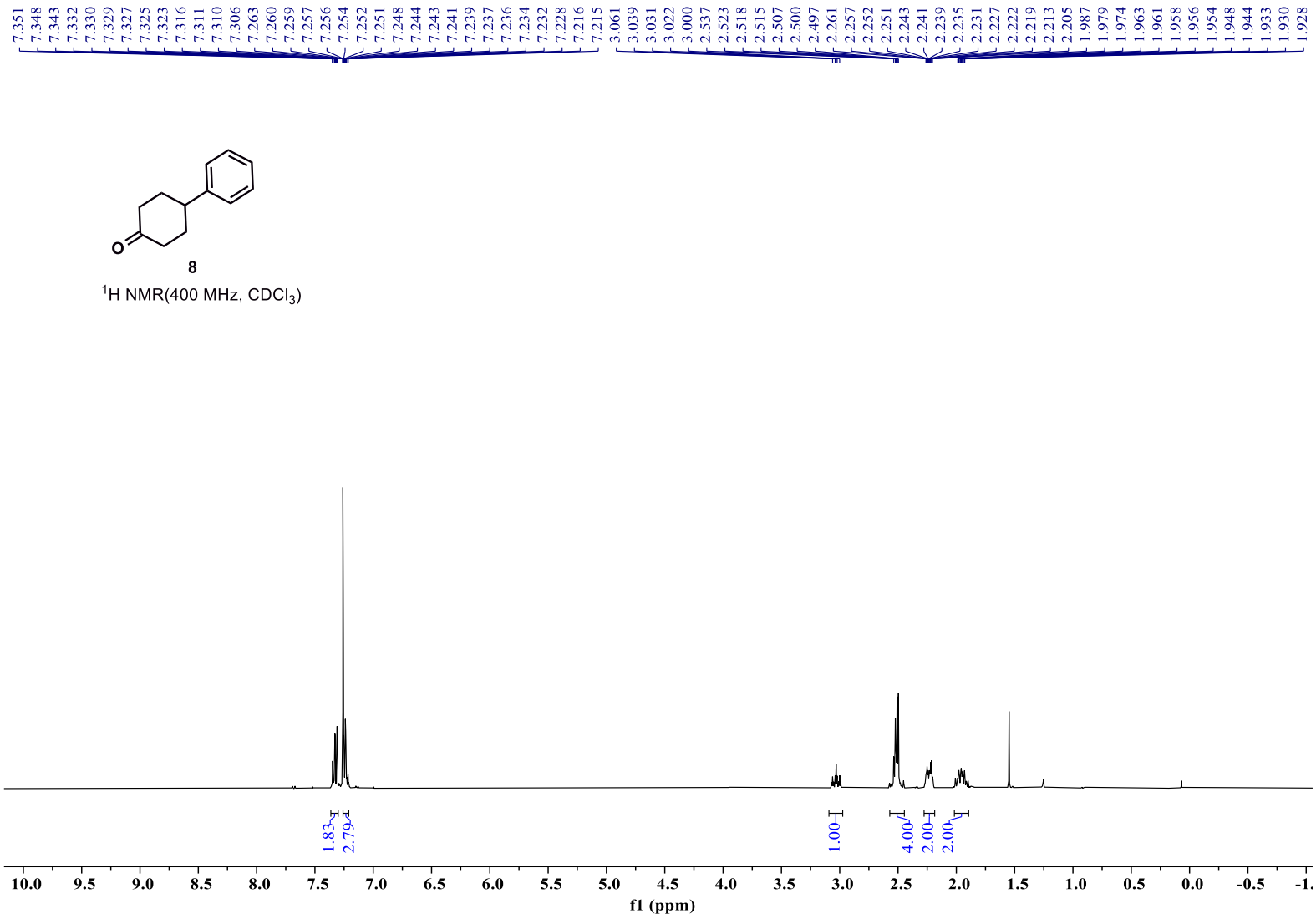
^{1}H NMR(400 MHz, $CDCl_3$)

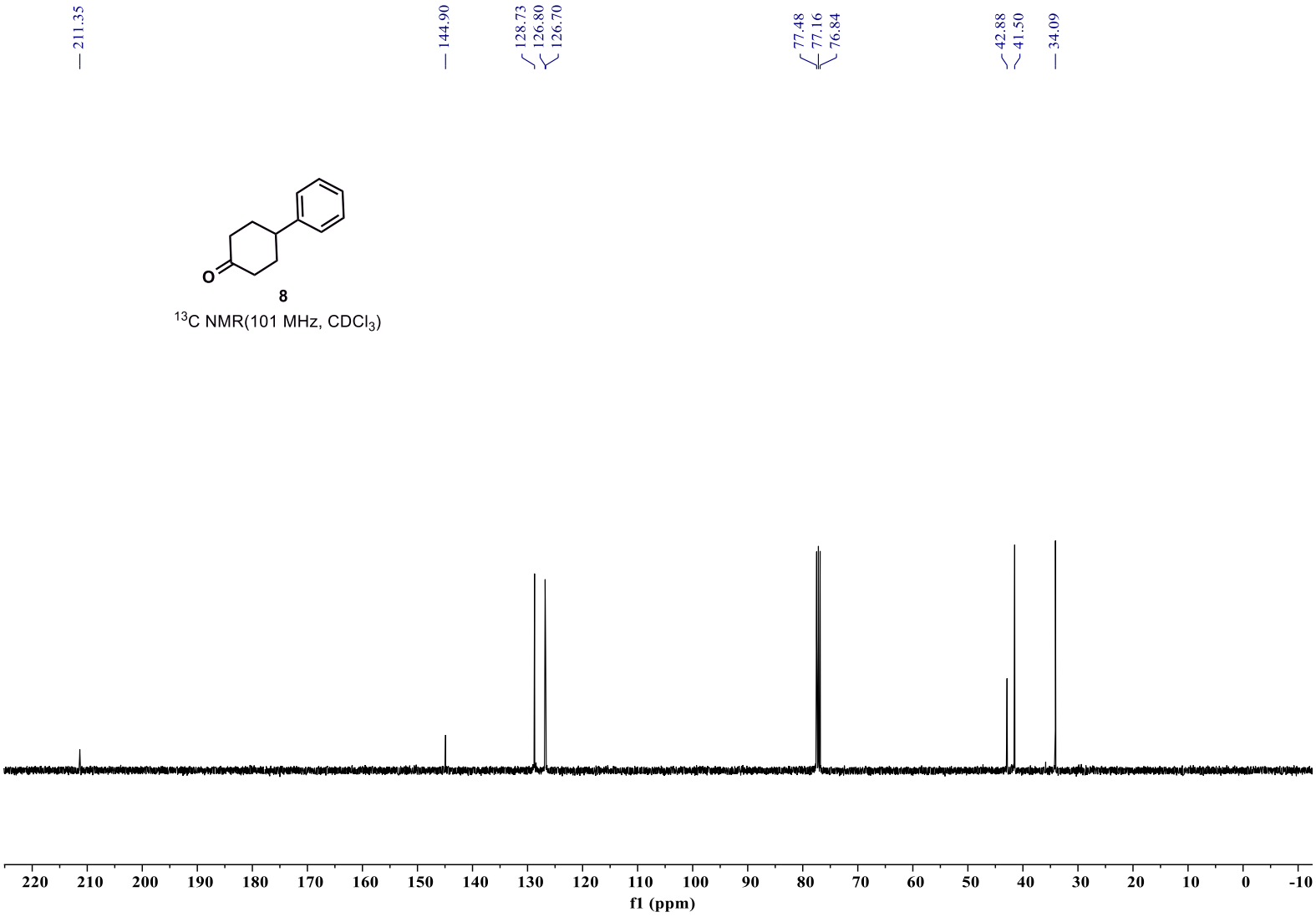



168.57
141.17
134.02
132.26
128.52
128.44
126.08
123.31
77.48
77.16
76.84
37.95
33.31
30.02
O
N
O
6
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.449
8.445
8.437
8.433
8.425
8.420
7.449
7.444
7.439
7.429
7.424
7.419
7.299
7.296
7.292
7.283
7.279
7.275
7.264
7.260
7.258
7.256
7.255
7.221
7.217
7.213
7.204
7.199
7.193
7.189
7.187
7.184
7.180
7.177
7.169
7.167
7.155
7.151
7.146
7.138
7.134
7.131
2.922
N
7
1H NMR(400 MHz, CDCl3)
2.05
0.93
1.95
1.73
2.07
4.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

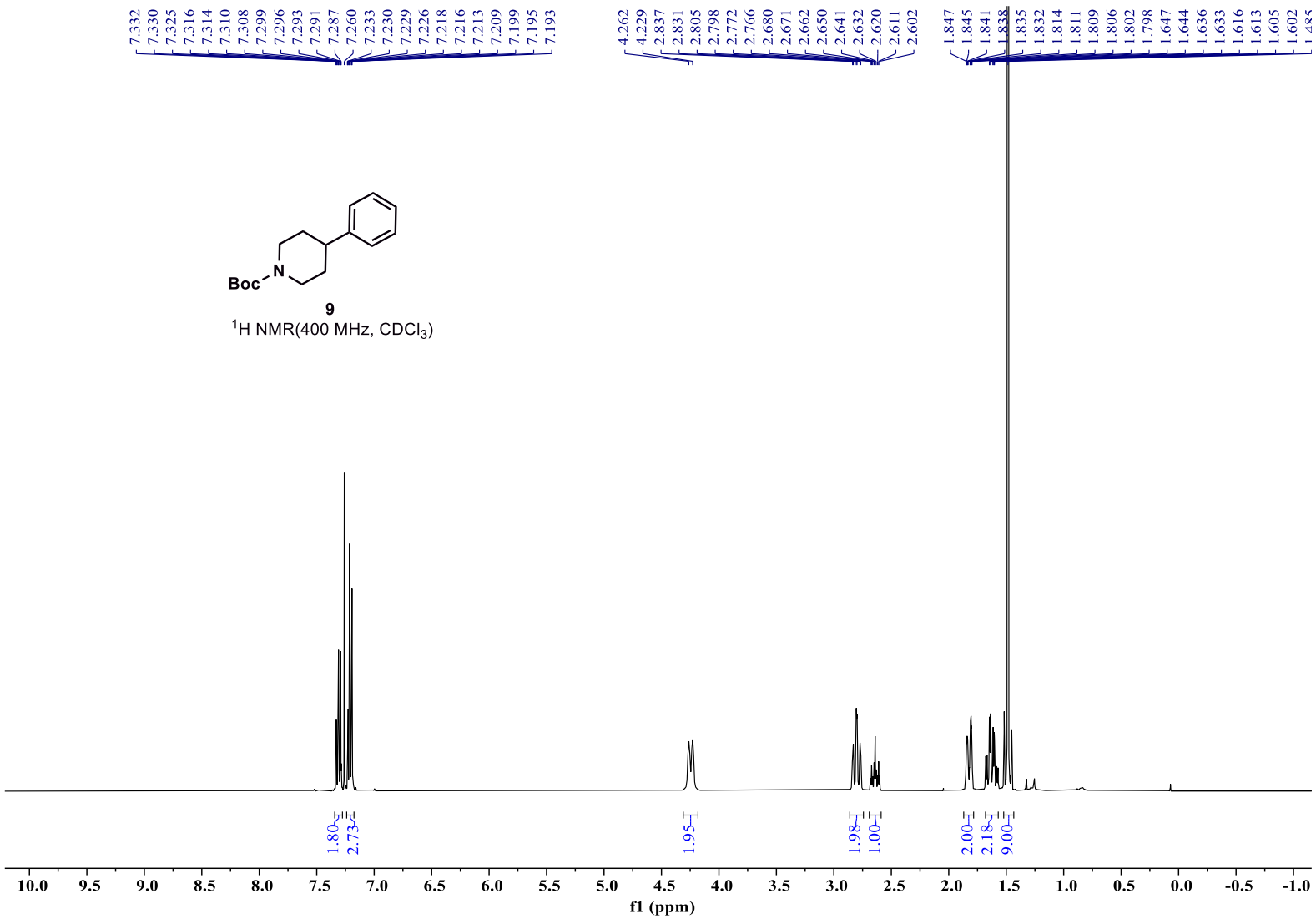

149.68
147.18
140.79
137.11
136.44
128.56
126.32
123.44
77.48
77.16
76.84
37.50
34.99
N
7
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.351 7.348 7.343 7.332 7.330 7.329 7.327 7.325 7.323 7.316 7.311 7.310 7.306 7.263 7.260 7.259 7.257 7.256 7.254 7.252 7.251 7.248 7.244 7.243 7.241 7.239 7.237 7.236 7.234 7.232 7.228 7.216 7.215
3.061 3.039 3.031 3.022 3.000 2.537 2.523 2.518 2.515 2.507 2.500 2.497 2.261 2.257 2.252 2.251 2.243 2.241 2.239 2.235 2.231 2.227 2.222 2.219 2.213 2.205 1.987 1.979 1.974 1.963 1.961 1.958 1.956 1.954 1.948 1.944 1.933 1.930 1.928
O
8
1H NMR(400 MHz, CDCl3)
1.83
2.79
1.00
4.00
2.00
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)



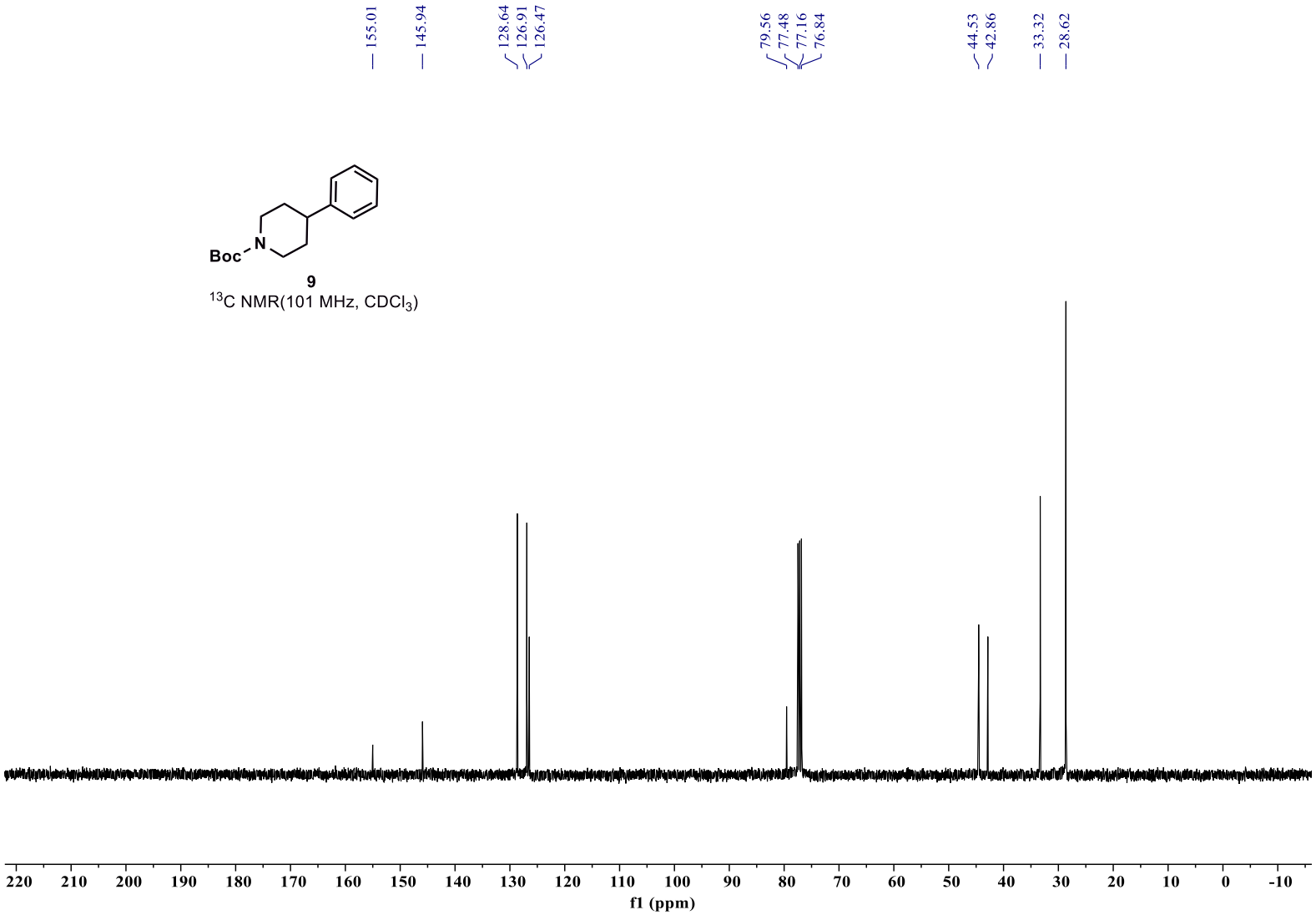
8

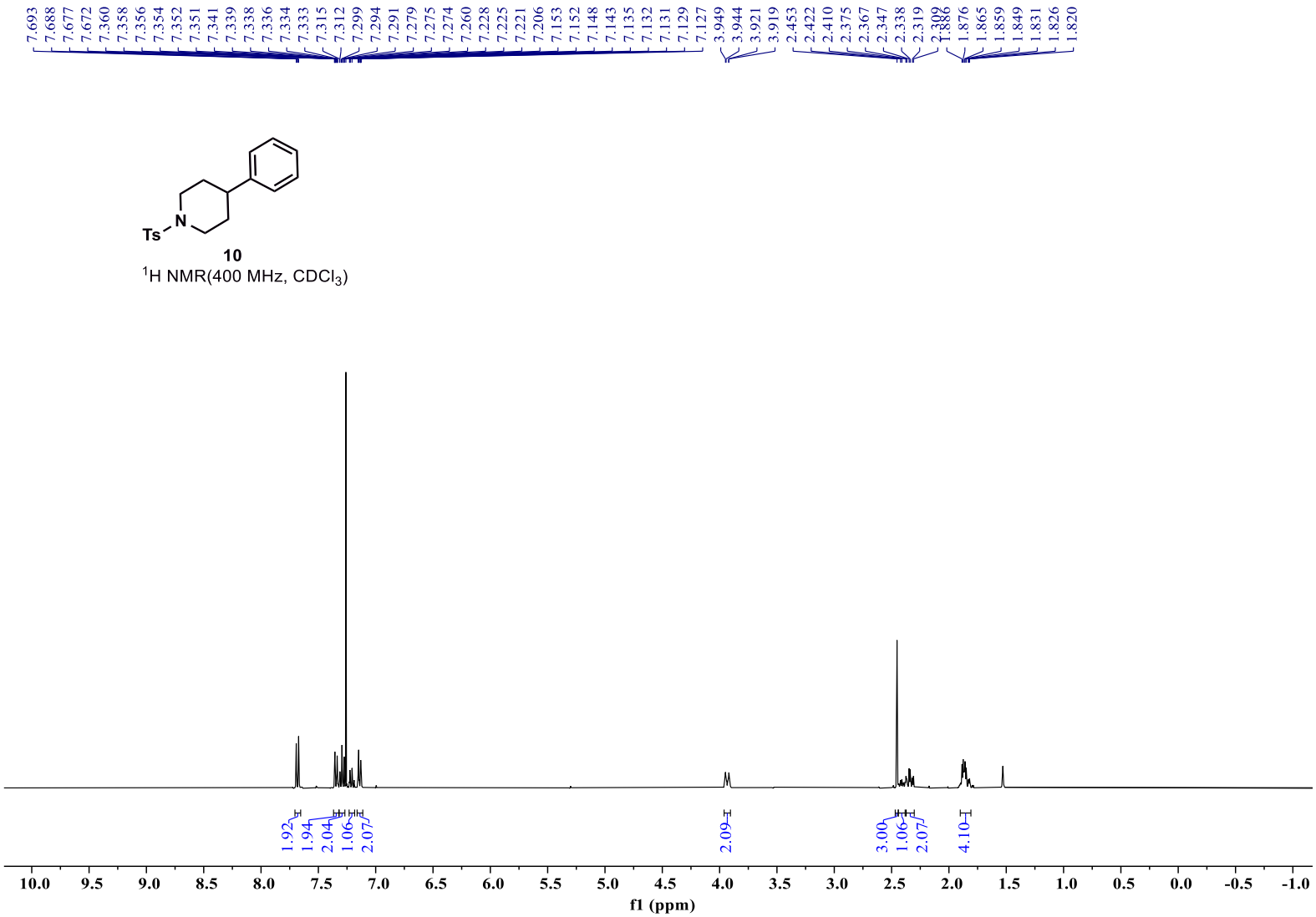
^{13}C NMR(101 MHz, $CDCl_3$)


7.332
7.330
7.325
7.316
7.314
7.310
7.308
7.299
7.296
7.293
7.291
7.287
7.260
7.233
7.230
7.229
7.226
7.218
7.216
7.213
7.209
7.199
7.195
7.193
4.262
4.229
2.837
2.831
2.805
2.798
2.772
2.766
2.680
2.671
2.662
2.650
2.641
2.632
2.620
2.611
2.602
1.847
1.845
1.841
1.838
1.835
1.832
1.814
1.811
1.809
1.806
1.802
1.798
1.647
1.644
1.636
1.633
1.616
1.613
1.605
1.602
1.485
Boc
N
9
1H NMR(400 MHz, CDCl3)
1.80
2.73
1.95
1.98
1.00
2.00
2.18
9.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

9

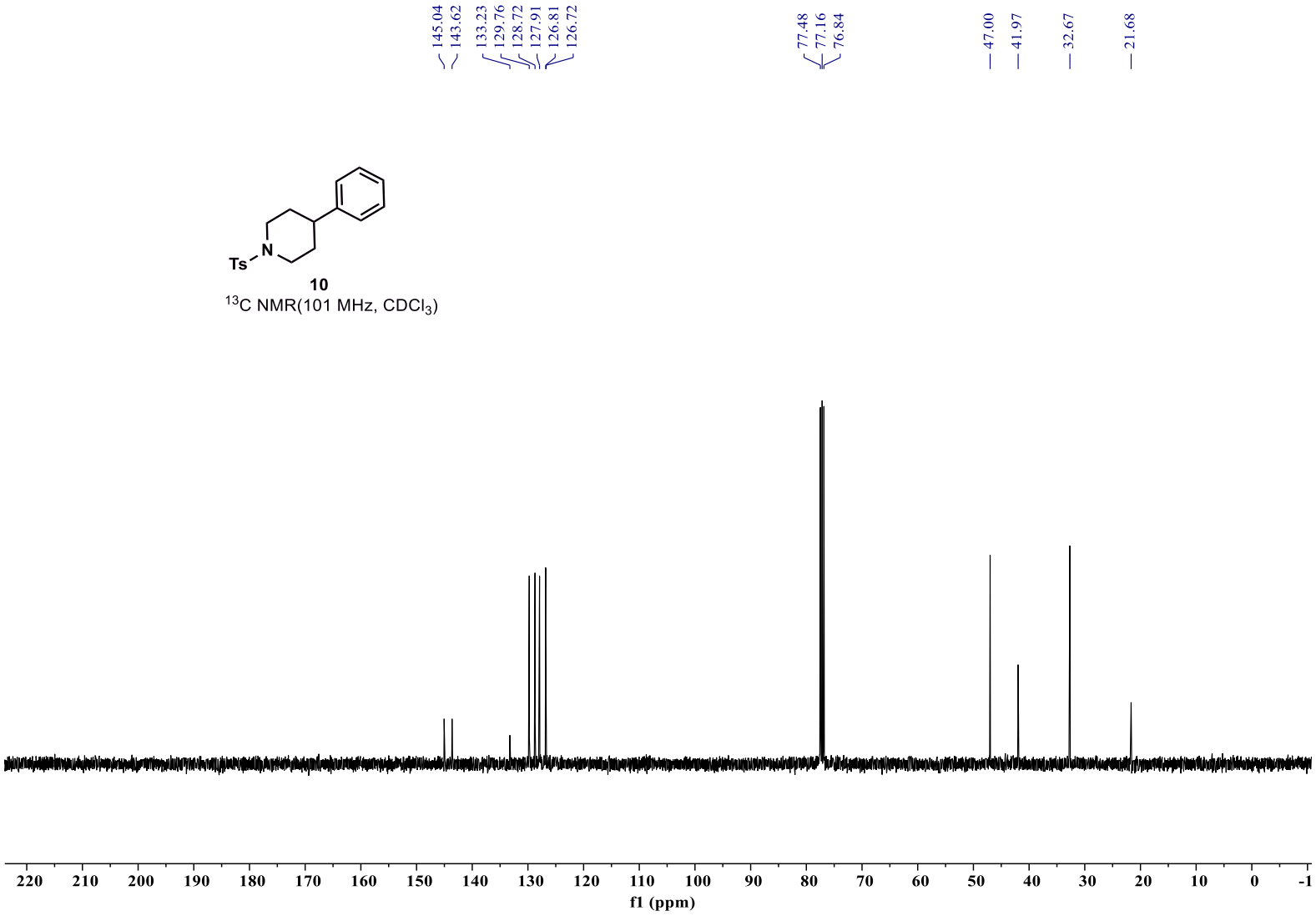
^{13}C NMR(101 MHz, $CDCl_3$)

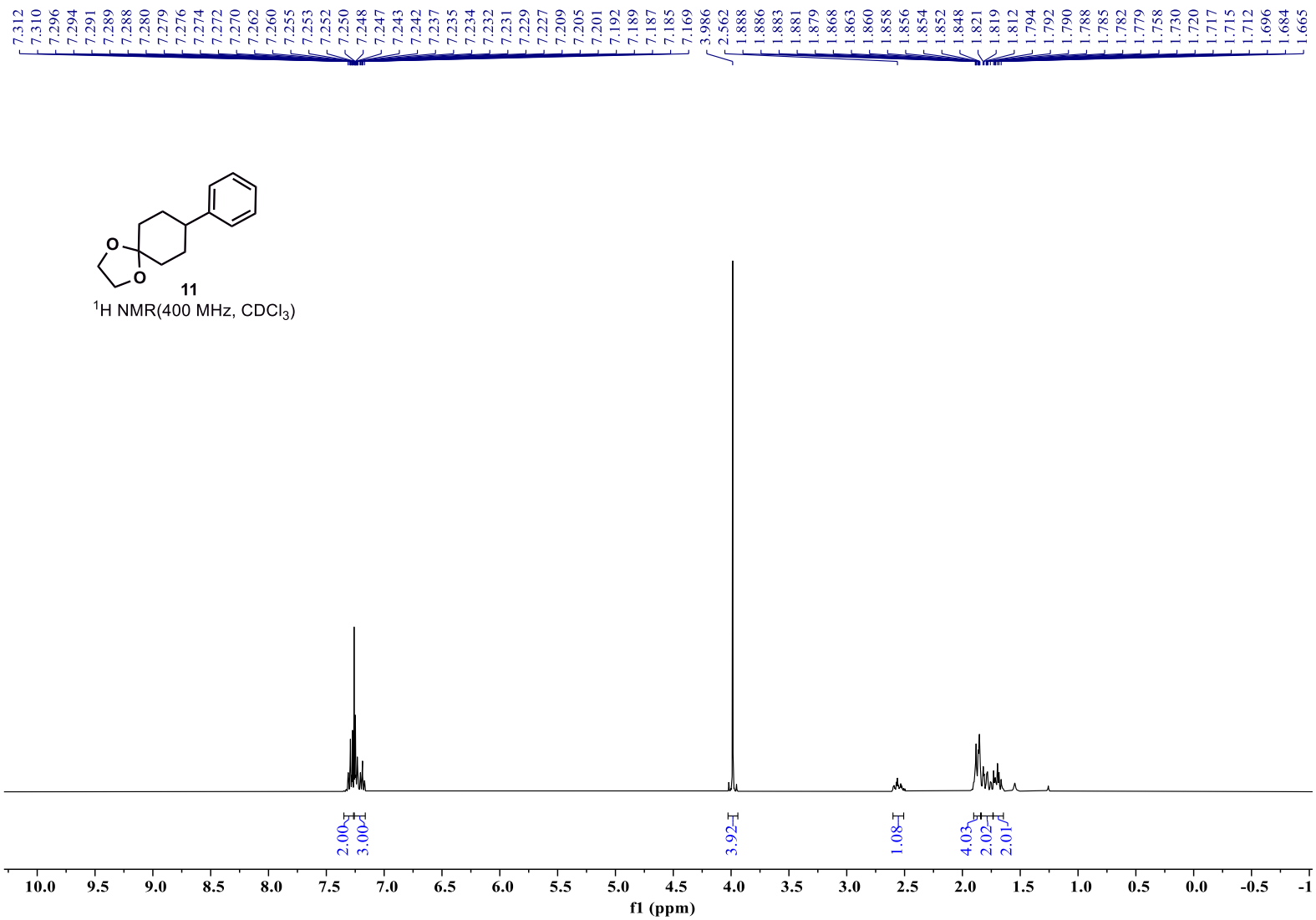


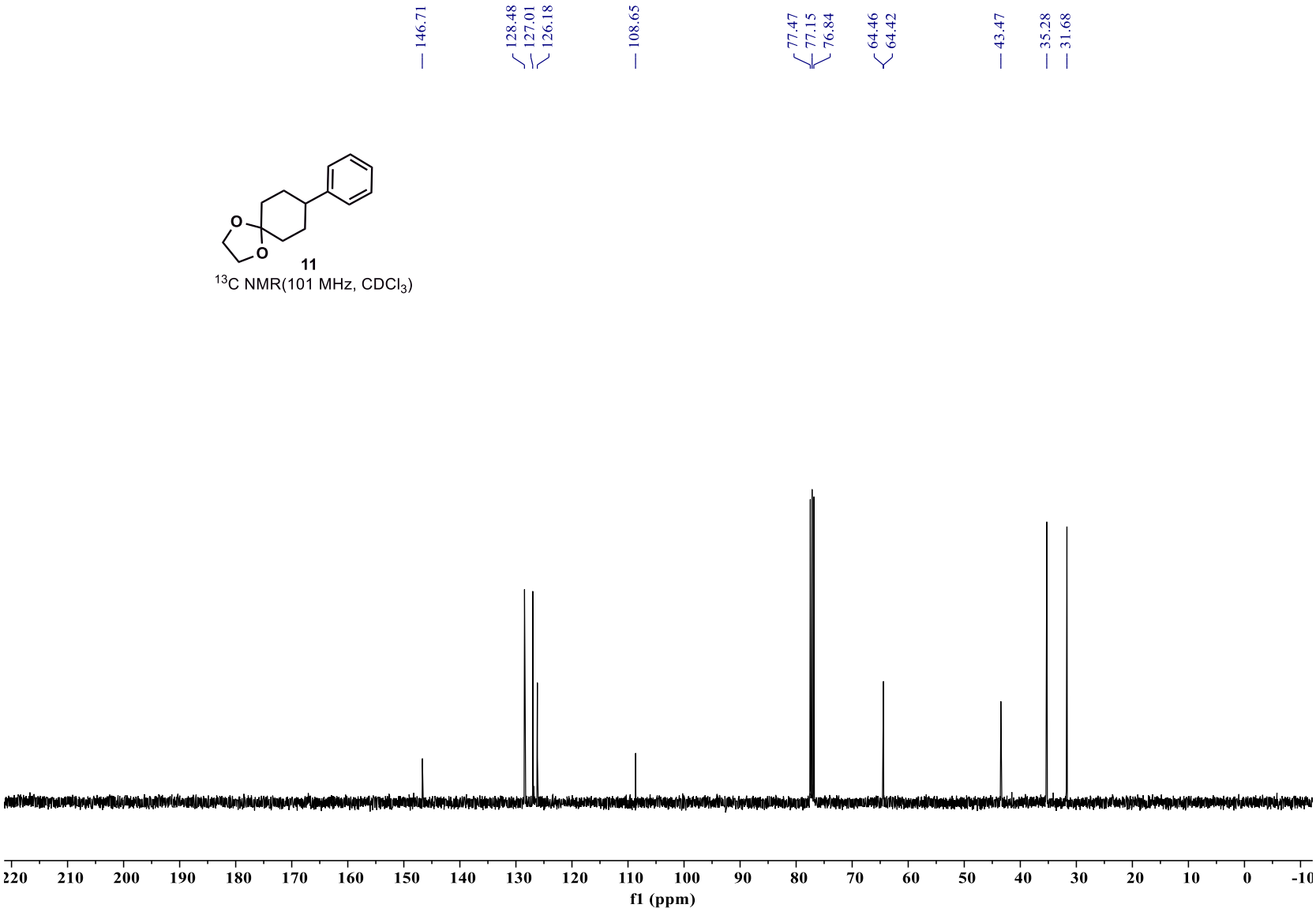

7.693
7.688
7.677
7.672
7.360
7.358
7.356
7.354
7.352
7.351
7.341
7.339
7.338
7.336
7.334
7.333
7.315
7.312
7.299
7.294
7.291
7.279
7.275
7.274
7.260
7.228
7.225
7.221
7.206
7.153
7.152
7.148
7.143
7.135
7.132
7.131
7.129
7.127
3.949
3.944
3.921
3.919
2.453
2.422
2.410
2.375
2.367
2.347
2.338
2.319
2.309
1.886
1.876
1.865
1.859
1.849
1.831
1.826
1.820
Ts
N
10
1H NMR(400 MHz, CDCl3)
1.92
1.94
2.04
1.06
2.07
2.09
3.00
1.06
2.07
4.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

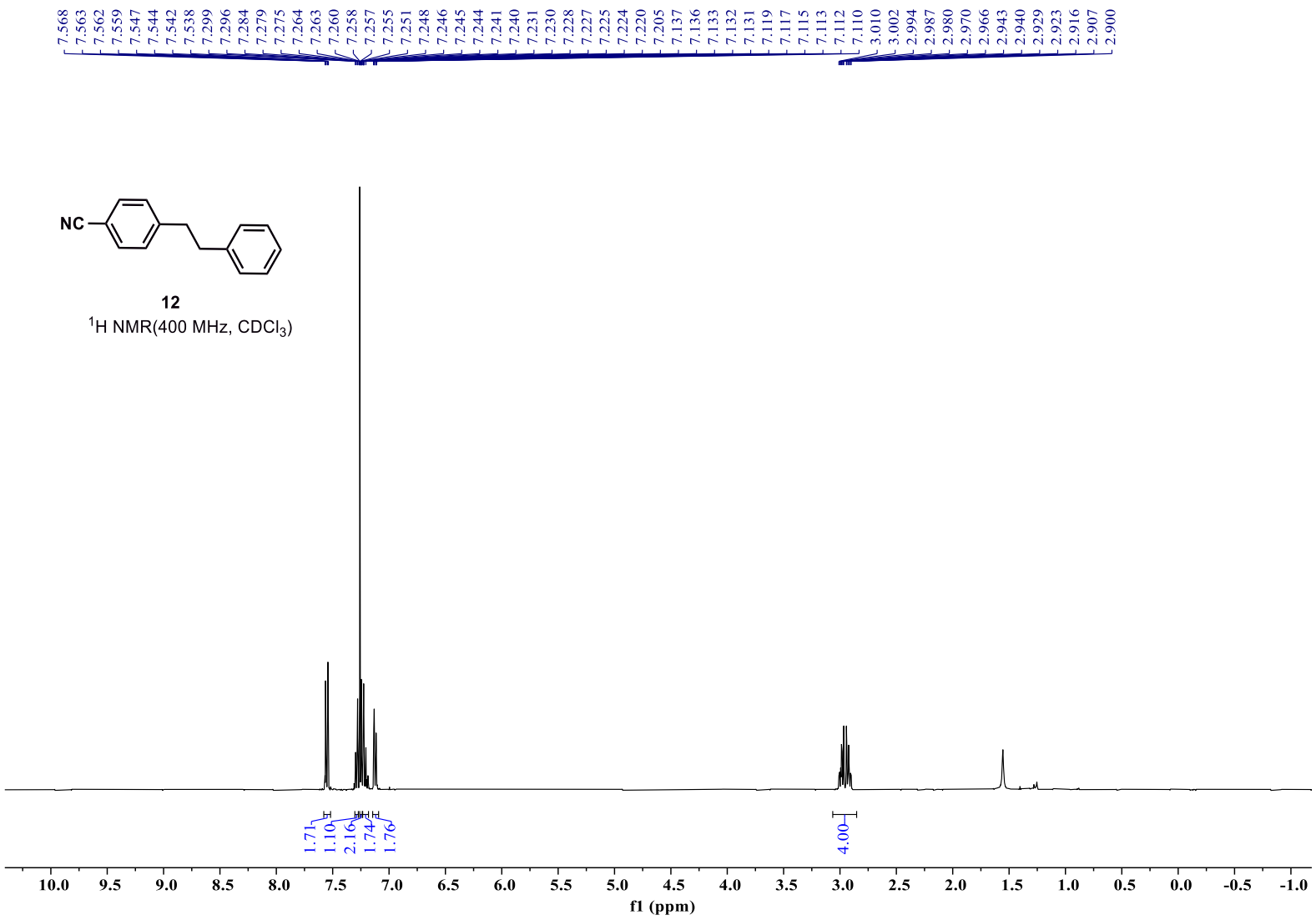
10

^{13}C NMR(101 MHz, $CDCl_3$)



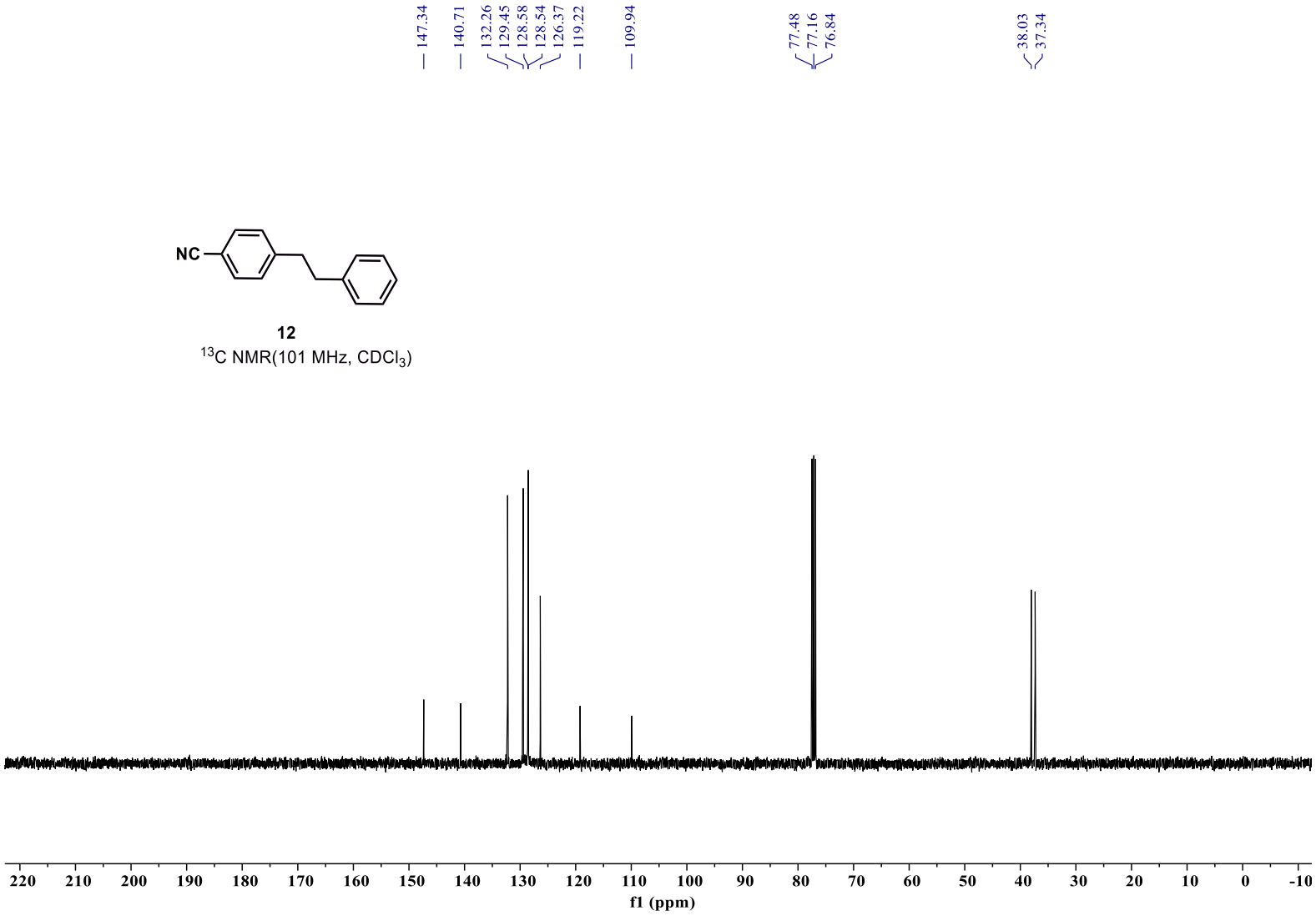

7.312
7.310
7.296
7.294
7.291
7.289
7.288
7.280
7.279
7.276
7.274
7.272
7.270
7.262
7.260
7.255
7.253
7.252
7.250
7.248
7.247
7.243
7.242
7.237
7.235
7.234
7.232
7.231
7.229
7.227
7.209
7.205
7.201
7.192
7.189
7.187
7.185
7.169
3.986
2.562
1.888
1.886
1.883
1.881
1.879
1.868
1.863
1.860
1.858
1.856
1.854
1.852
1.848
1.821
1.819
1.812
1.794
1.792
1.790
1.788
1.785
1.782
1.779
1.758
1.730
1.720
1.717
1.715
1.712
1.696
1.684
1.665
O
O
11
1H NMR(400 MHz, CDCl3)
2.00
3.00
3.92
1.08
4.03
2.02
2.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

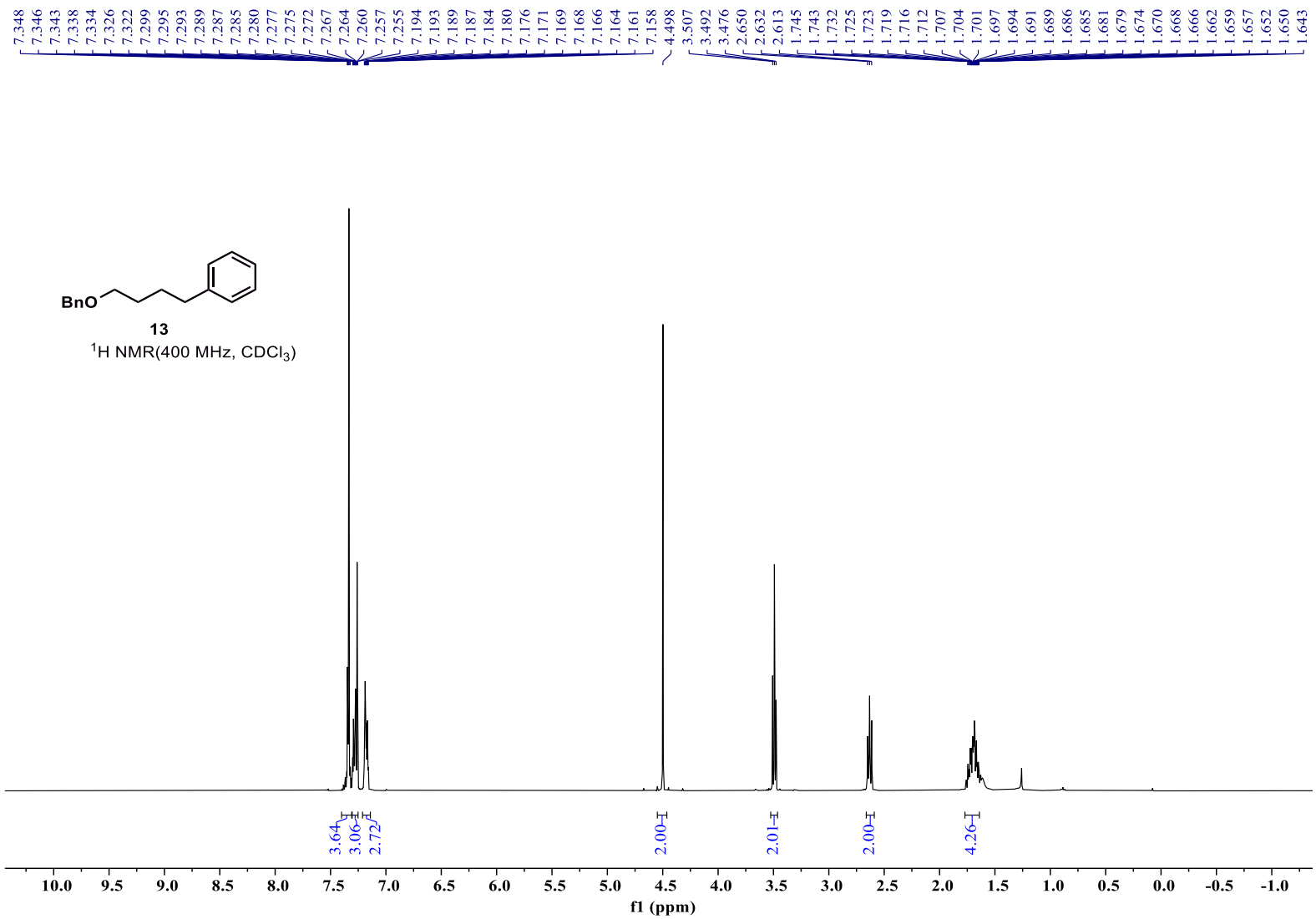

146.71
128.48
127.01
126.18
108.65
77.47
77.15
76.84
64.46
64.42
43.47
35.28
31.68
O
O
11
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

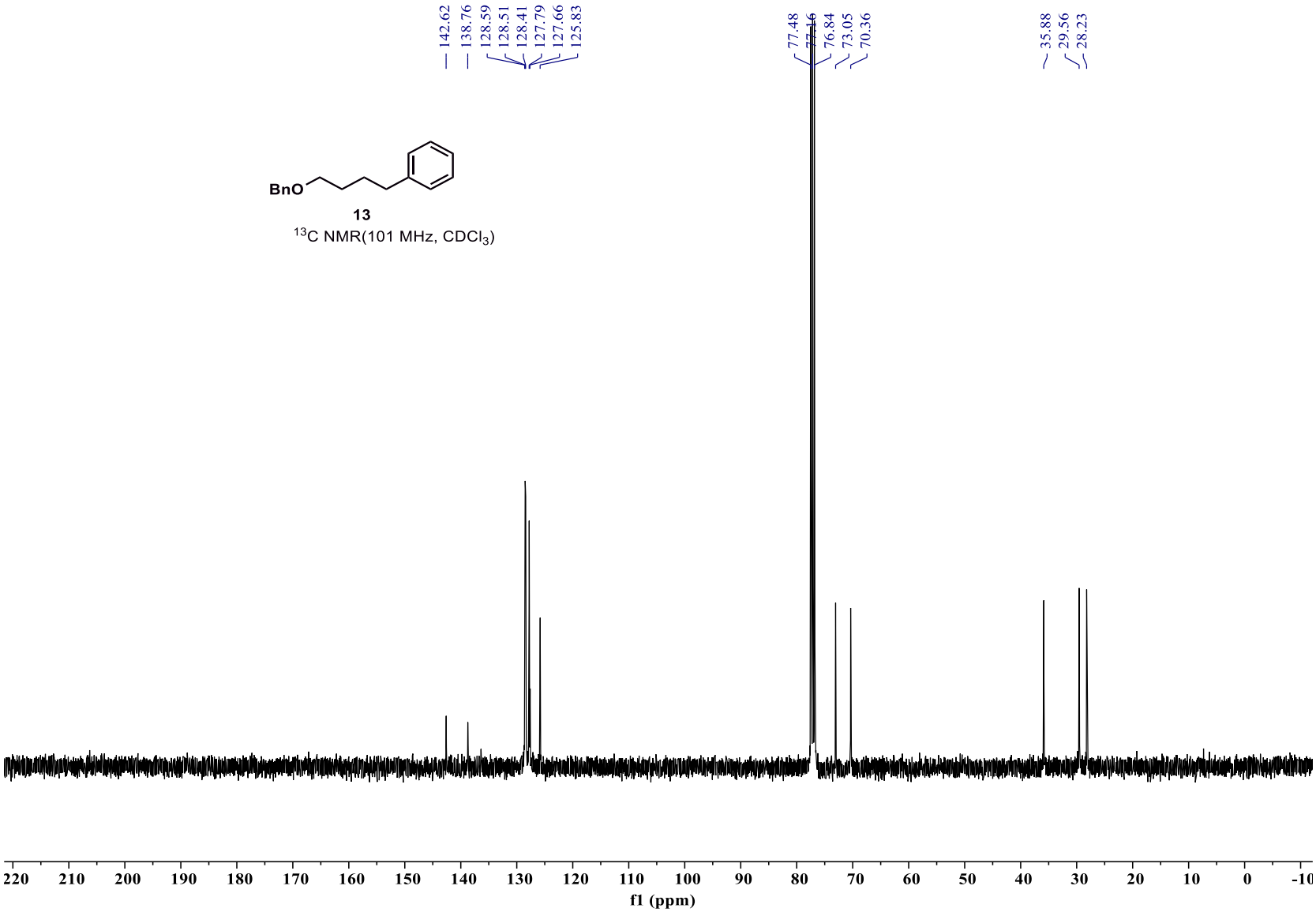

7.568
7.563
7.562
7.559
7.547
7.544
7.542
7.538
7.299
7.296
7.284
7.279
7.275
7.264
7.263
7.260
7.258
7.257
7.255
7.251
7.248
7.246
7.245
7.244
7.241
7.240
7.231
7.230
7.228
7.227
7.225
7.224
7.220
7.205
7.137
7.136
7.133
7.132
7.131
7.119
7.117
7.115
7.113
7.112
7.110
3.010
3.002
2.994
2.987
2.980
2.970
2.966
2.943
2.940
2.929
2.923
2.916
2.907
2.900
NC
12
1H NMR(400 MHz, CDCl3)
1.71
1.10
2.16
1.74
1.76
4.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

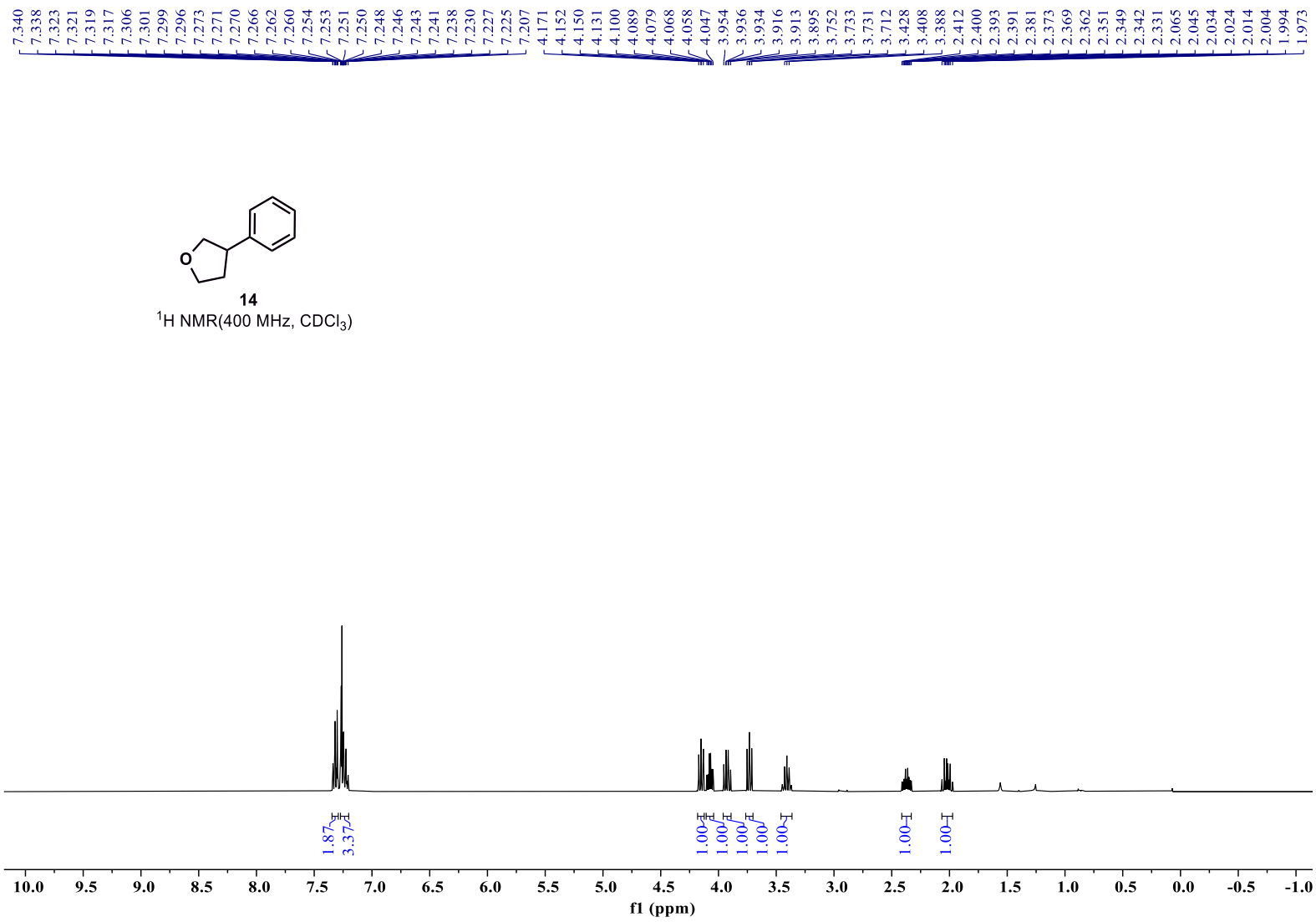
12

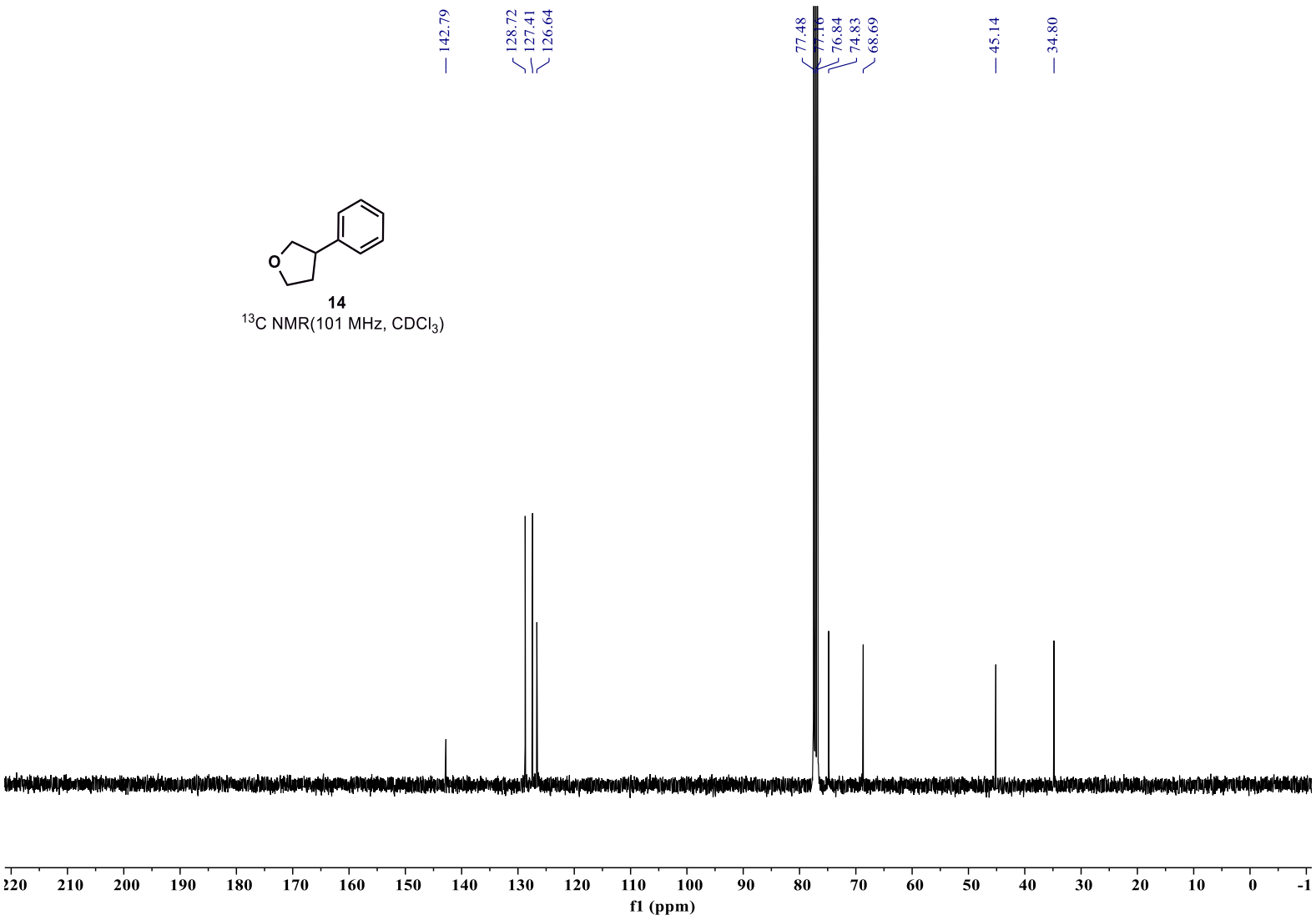
^{13}C NMR(101 MHz, $CDCl_3$)




7.348
7.346
7.343
7.338
7.334
7.326
7.322
7.299
7.295
7.293
7.289
7.287
7.285
7.280
7.277
7.275
7.272
7.267
7.264
7.260
7.257
7.255
7.194
7.193
7.189
7.187
7.184
7.180
7.176
7.171
7.169
7.168
7.166
7.164
7.161
7.158
4.498
3.507
3.492
3.476
2.650
2.632
2.613
1.745
1.743
1.732
1.725
1.723
1.719
1.716
1.712
1.707
1.704
1.701
1.697
1.694
1.691
1.689
1.686
1.685
1.681
1.679
1.674
1.670
1.668
1.666
1.662
1.659
1.657
1.652
1.650
1.643
BnO
13
1H NMR(400 MHz, CDCl3)
3.64
3.06
2.72
2.00
2.01
2.00
4.26
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

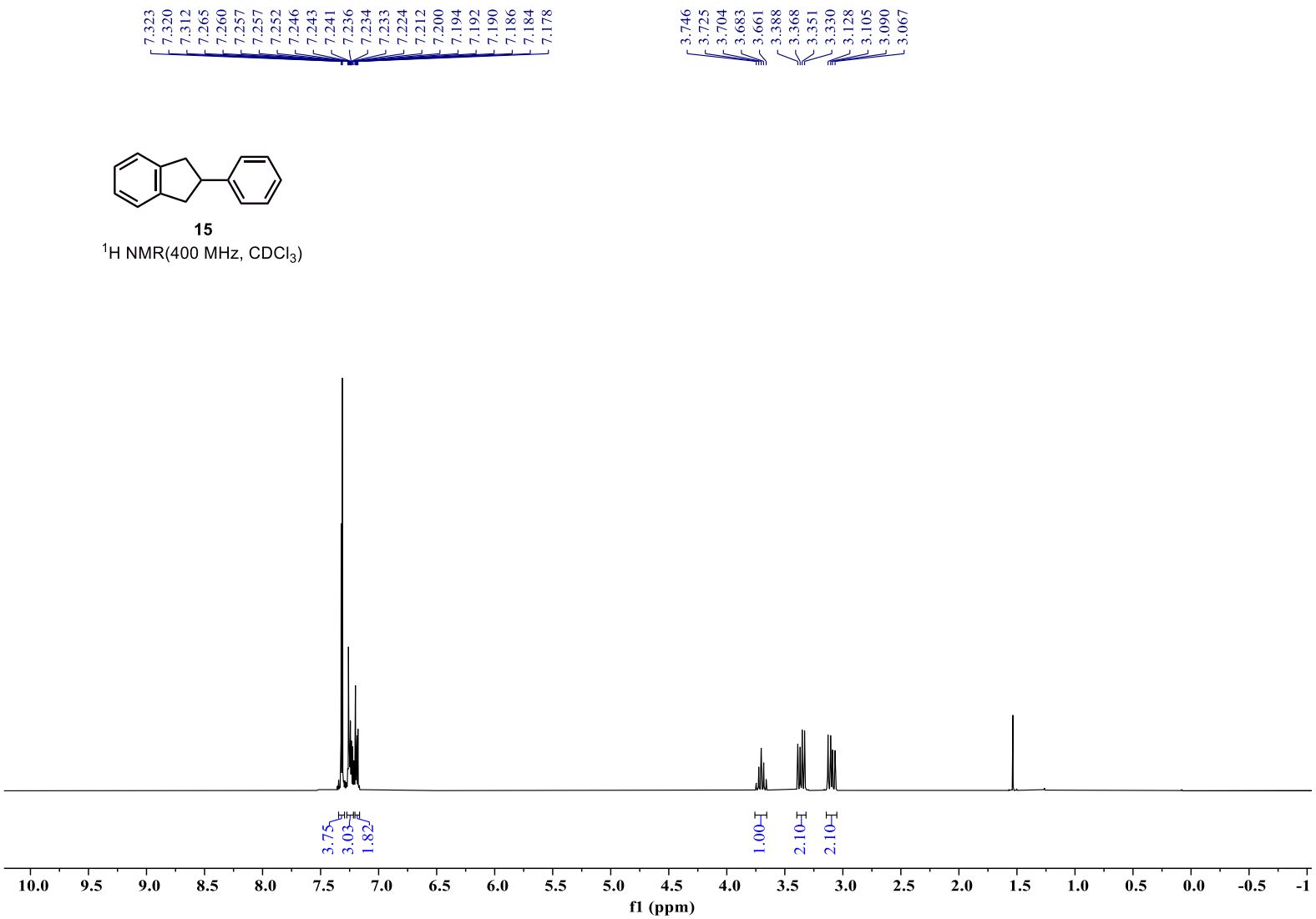

142.62
138.76
128.59
128.51
128.41
127.79
127.66
125.83
77.48
77.16
76.84
73.05
70.36
35.88
29.56
28.23
BnO
13
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

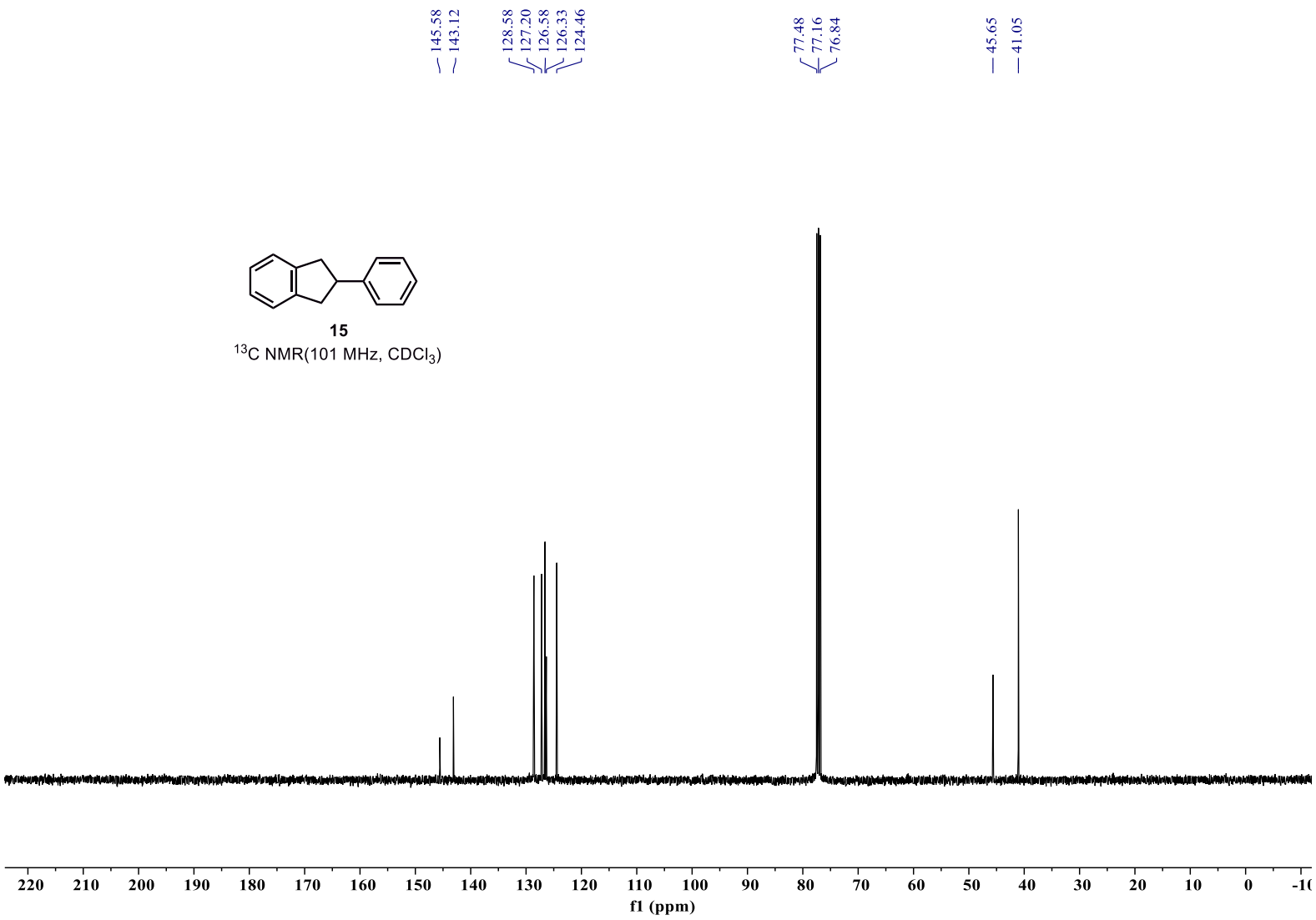

7.340 7.338 7.323 7.321 7.319 7.317 7.306 7.301 7.299 7.296 7.273 7.271 7.270 7.266 7.262 7.260 7.254 7.253 7.251 7.250 7.248 7.246 7.243 7.241 7.238 7.230 7.227 7.225 7.207
4.171 4.152 4.150 4.131 4.100 4.089 4.079 4.068 4.058 4.047 3.954 3.936 3.934 3.916 3.913 3.895 3.752 3.733 3.731 3.712 3.428 3.408 3.388 2.412 2.400 2.393 2.391 2.381 2.373 2.369 2.362 2.351 2.349 2.342 2.331 2.065 2.045 2.034 2.024 2.014 2.004 1.994 1.973
O
14
1H NMR(400 MHz, CDCl3)
1.87
3.37
1.00
1.00
1.00
1.00
1.00
1.00
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

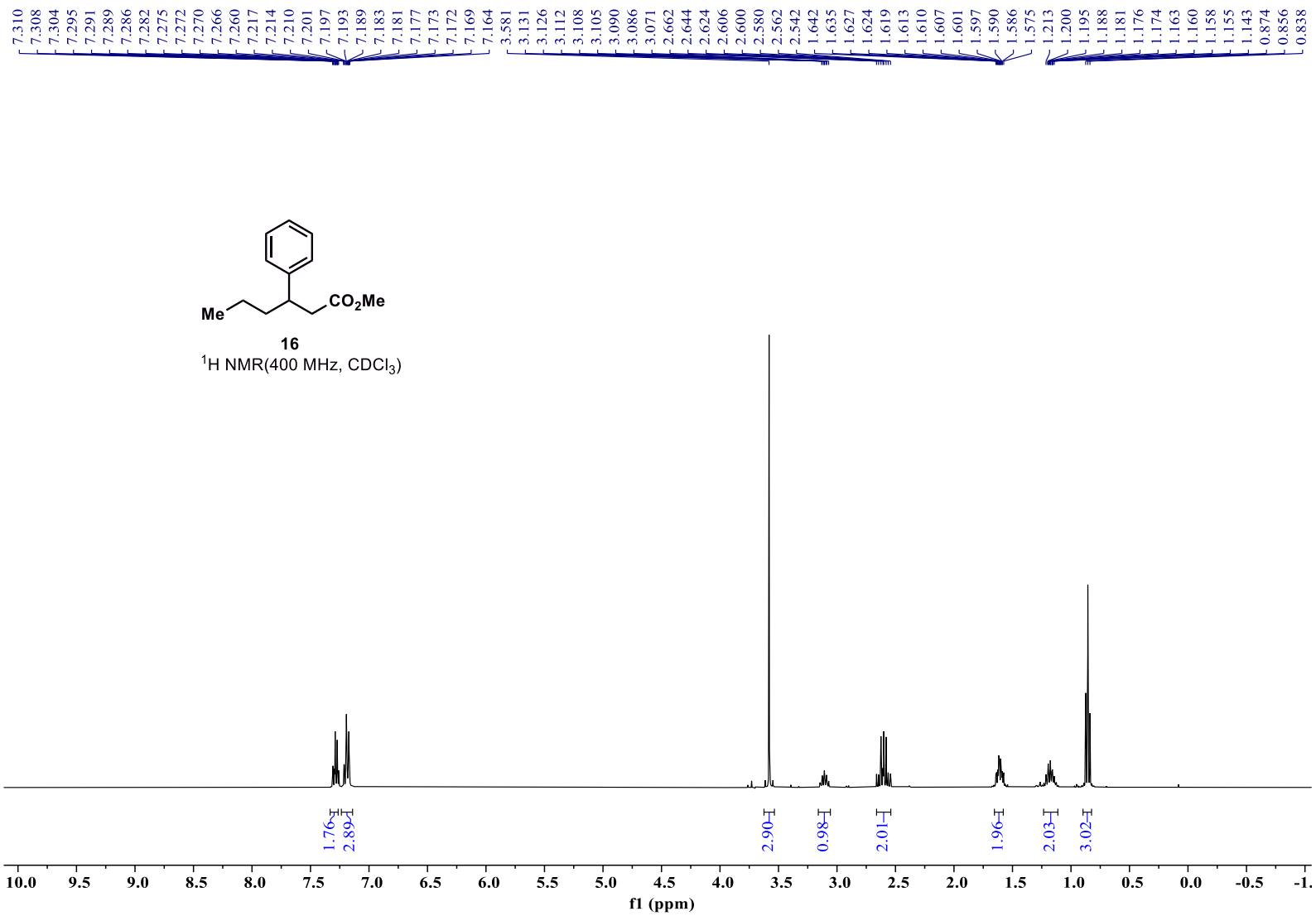


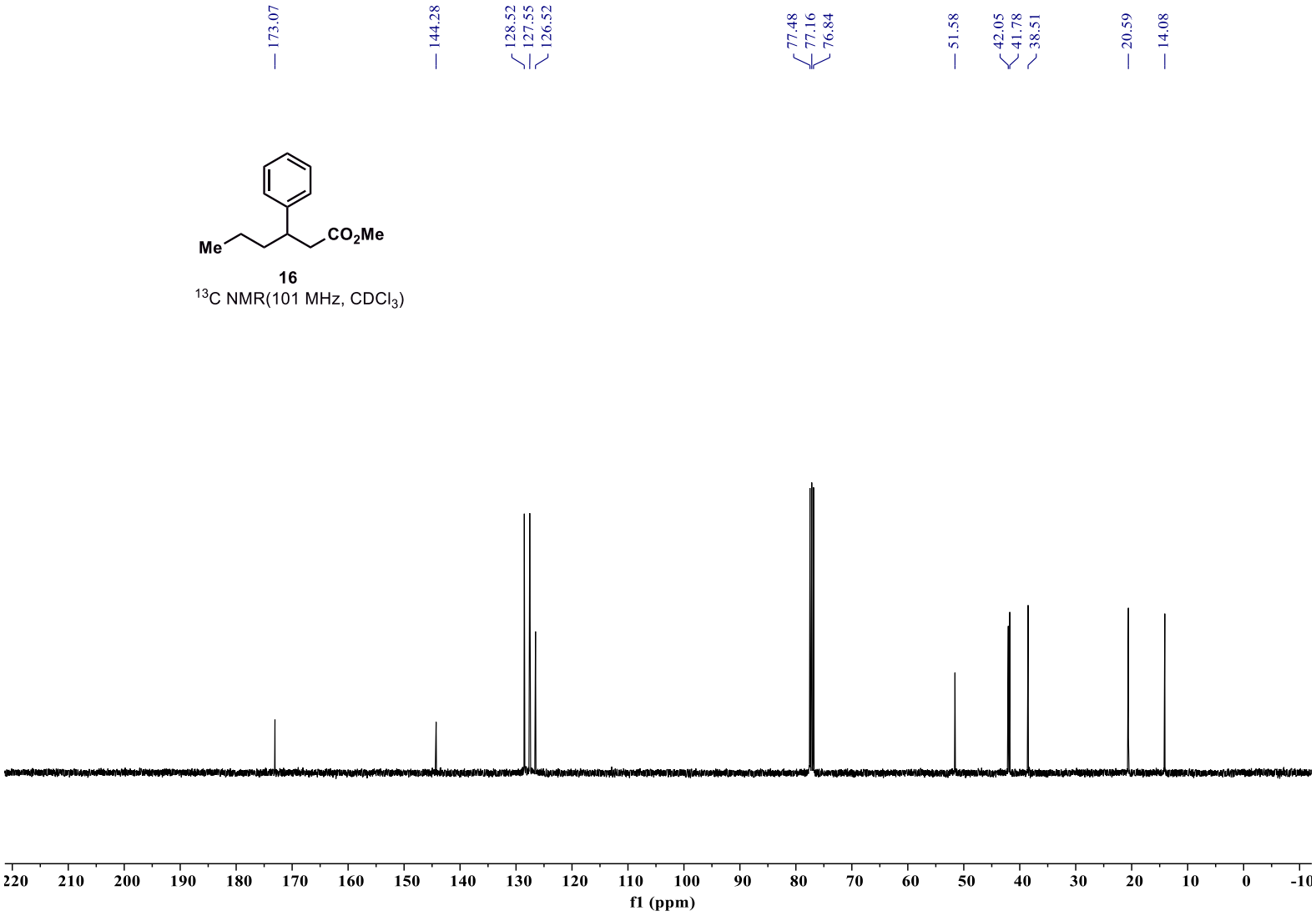
14

^{13}C NMR(101 MHz, $CDCl_3$)


7.323
7.320
7.312
7.265
7.260
7.257
7.257
7.252
7.246
7.243
7.241
7.236
7.234
7.233
7.224
7.212
7.200
7.194
7.192
7.190
7.186
7.184
7.178
3.746
3.725
3.704
3.683
3.661
3.388
3.368
3.351
3.330
3.128
3.105
3.090
3.067
15
1H NMR(400 MHz, CDCl3)
3.75
3.03
1.82
1.00
2.10
2.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

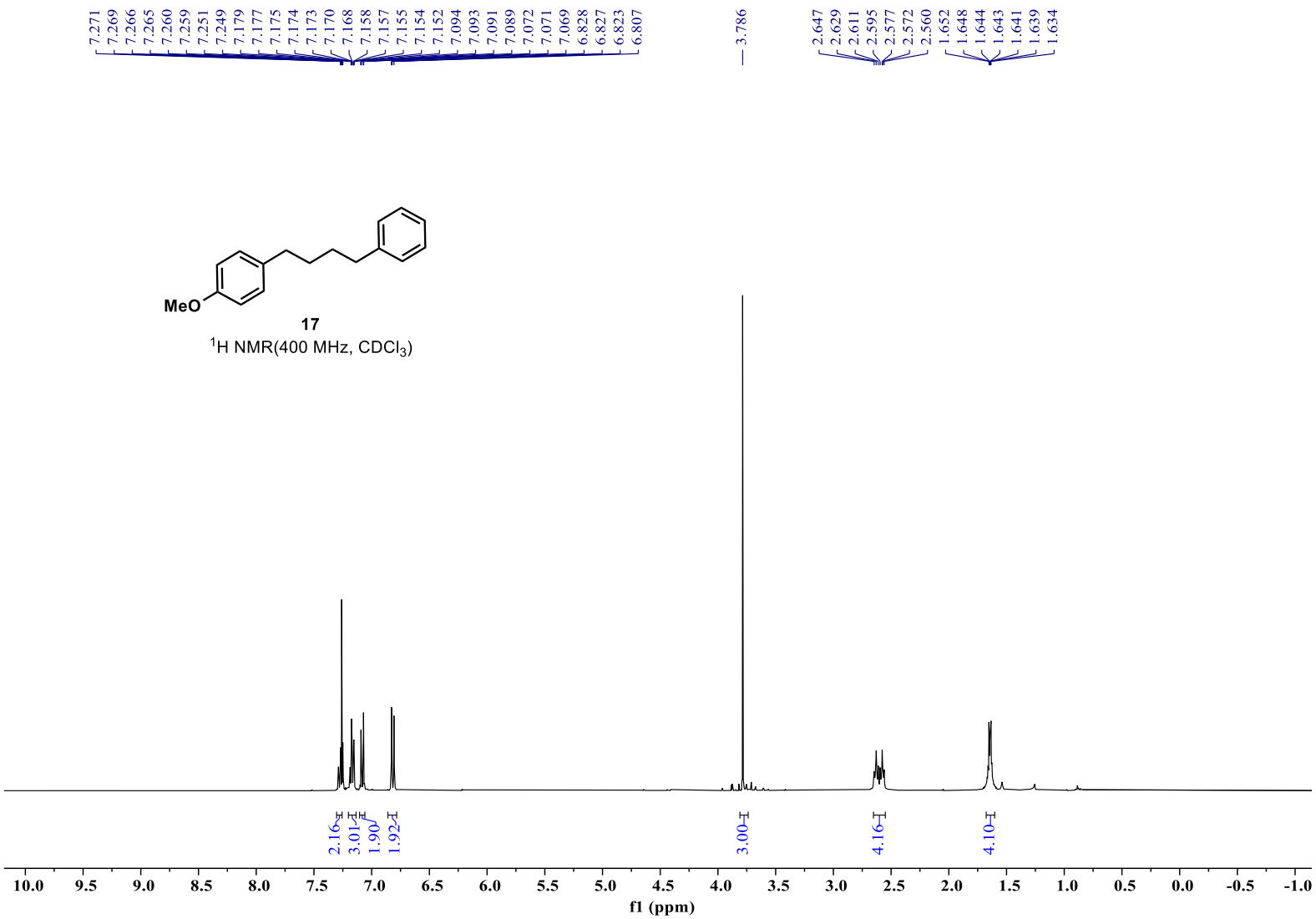

145.58
143.12
128.58
127.20
126.58
126.33
124.46
77.48
77.16
76.84
45.65
41.05
15
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.310 7.308 7.304 7.295 7.291 7.289 7.286 7.282 7.275 7.272 7.270 7.266 7.260 7.217 7.214 7.210 7.201 7.197 7.193 7.189 7.183 7.181 7.177 7.173 7.172 7.169 7.164
3.581 3.131 3.126 3.112 3.108 3.105 3.090 3.086 3.071 2.662 2.644 2.624 2.606 2.600 2.580 2.562 2.542 1.642 1.635 1.627 1.624 1.619 1.613 1.610 1.607 1.601 1.597 1.590 1.586 1.575
1.213 1.200 1.195 1.188 1.181 1.176 1.174 1.163 1.160 1.158 1.155 1.143 0.874 0.856 0.838
Me
CO2Me
16
1H NMR(400 MHz, CDCl3)
1.76
2.89
2.90
0.98
2.01
1.96
2.03
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)



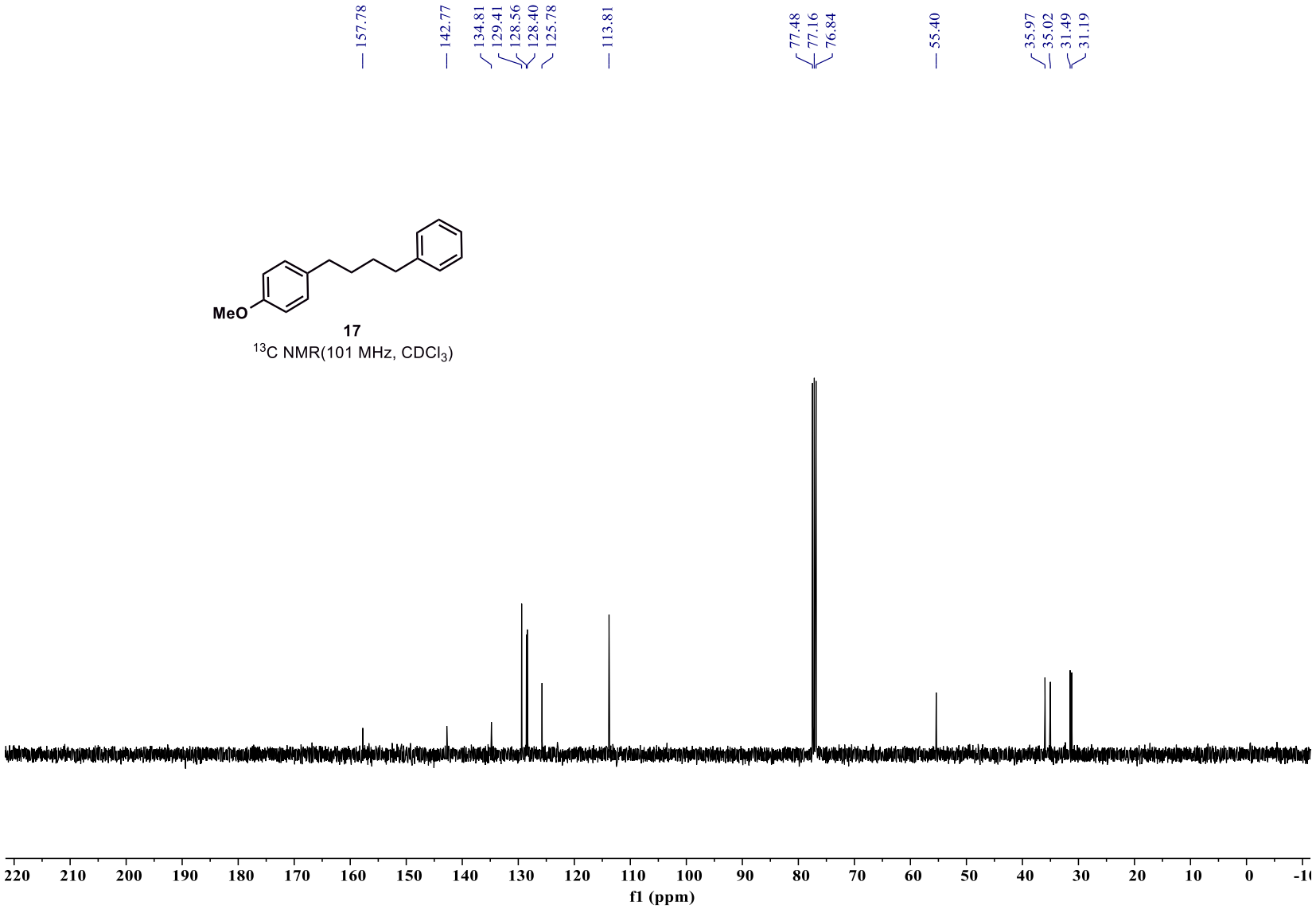
16

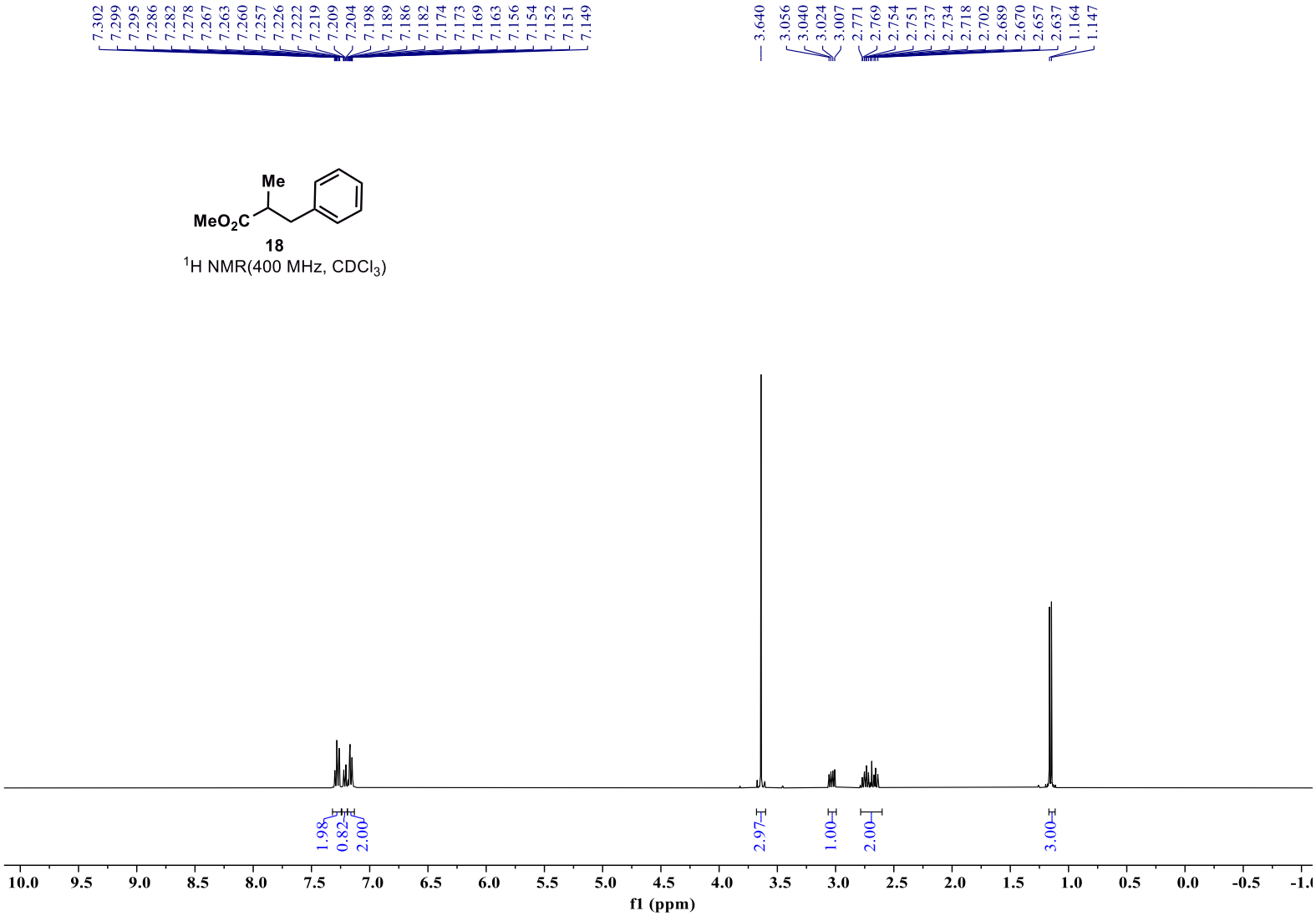
^{13}C NMR(101 MHz, $CDCl_3$)

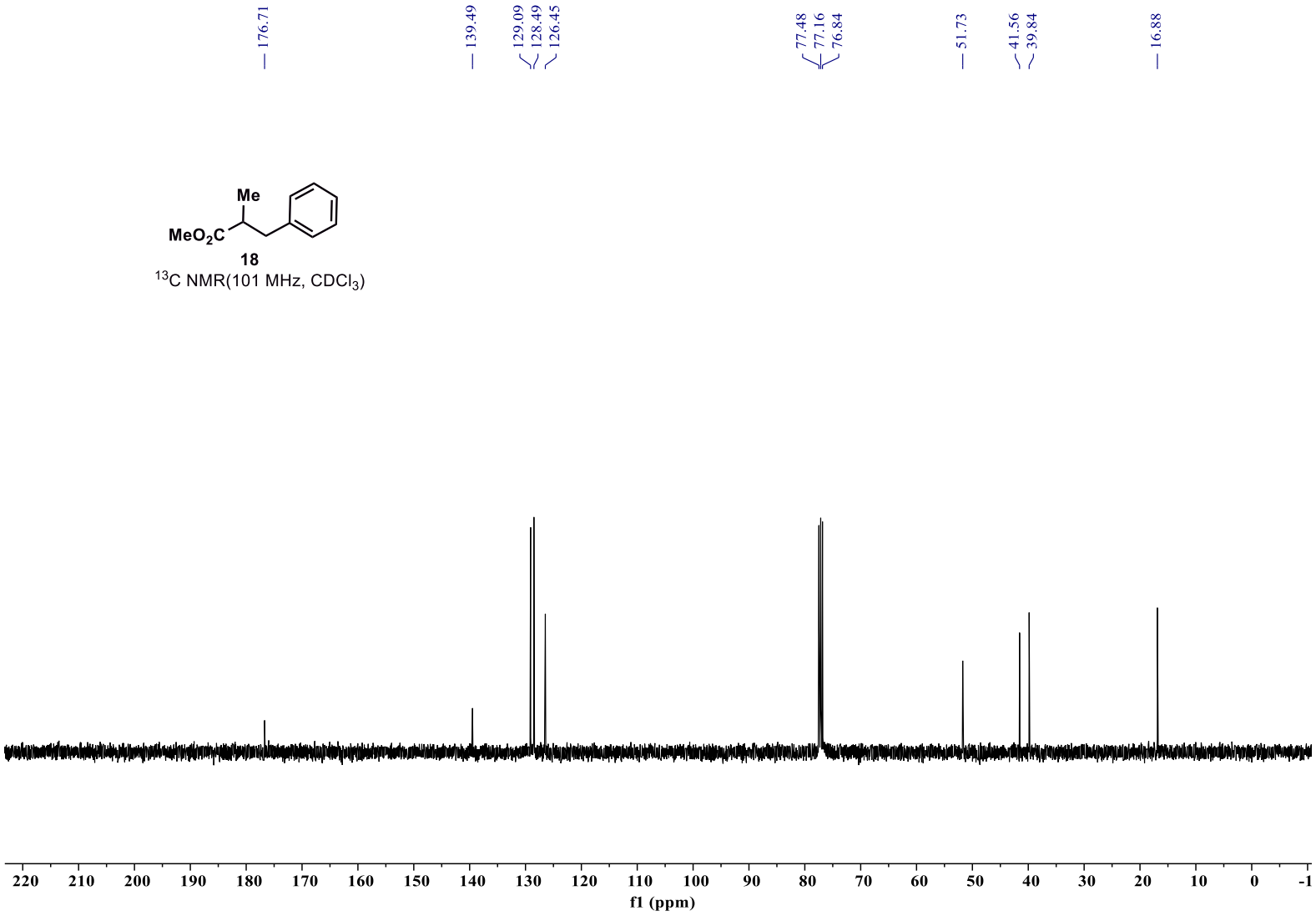

7.271
7.269
7.266
7.265
7.260
7.259
7.251
7.249
7.179
7.177
7.175
7.174
7.173
7.170
7.168
7.158
7.157
7.155
7.154
7.152
7.094
7.093
7.091
7.089
7.072
7.071
7.069
6.828
6.827
6.823
6.807
3.786
2.647
2.629
2.611
2.595
2.577
2.572
2.560
1.652
1.648
1.644
1.643
1.641
1.639
1.634
MeO
17
1H NMR(400 MHz, CDCl3)
2.16
3.01
1.90
1.92
3.00
4.16
4.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

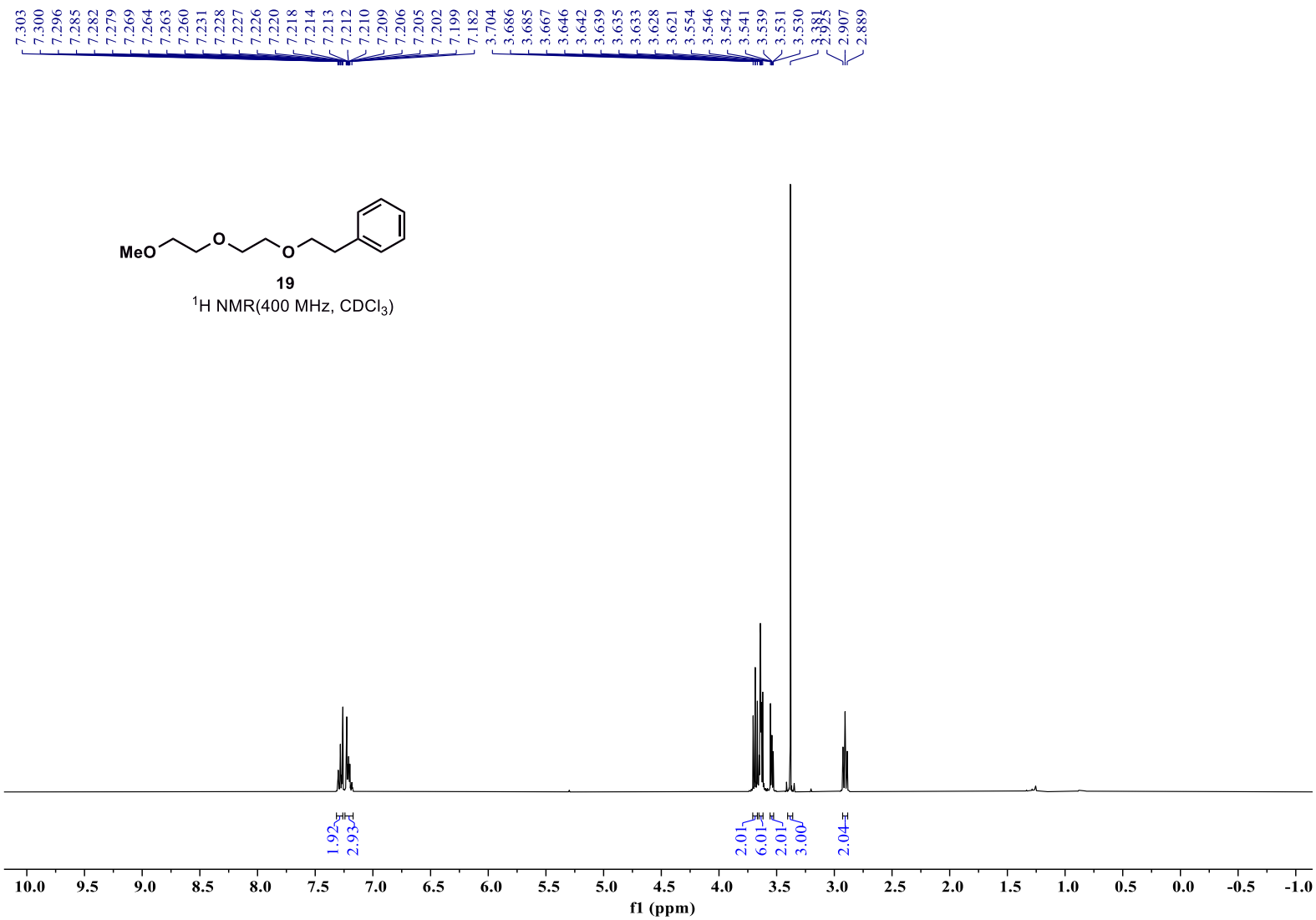
17

^{13}C NMR(101 MHz, $CDCl_3$)



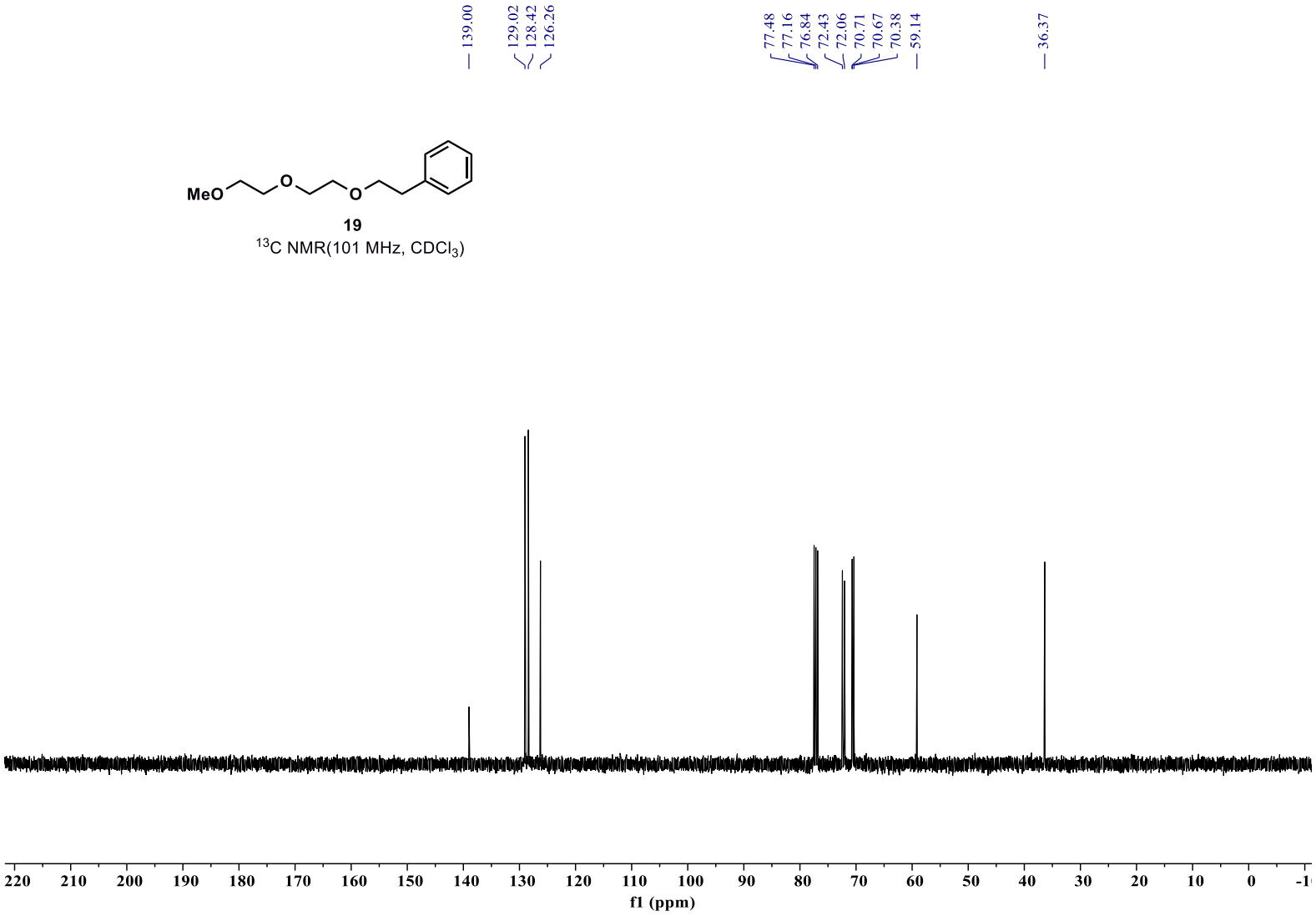

7.302
7.299
7.295
7.286
7.282
7.278
7.267
7.263
7.260
7.257
7.226
7.222
7.219
7.209
7.204
7.198
7.189
7.186
7.182
7.174
7.173
7.169
7.163
7.156
7.154
7.152
7.151
7.149
3.640
3.056
3.040
3.024
3.007
2.771
2.769
2.754
2.751
2.737
2.734
2.718
2.702
2.689
2.670
2.657
2.637
1.164
1.147
Me
MeO2C
18
1H NMR(400 MHz, CDCl3)
1.98
0.82
2.00
2.97
1.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

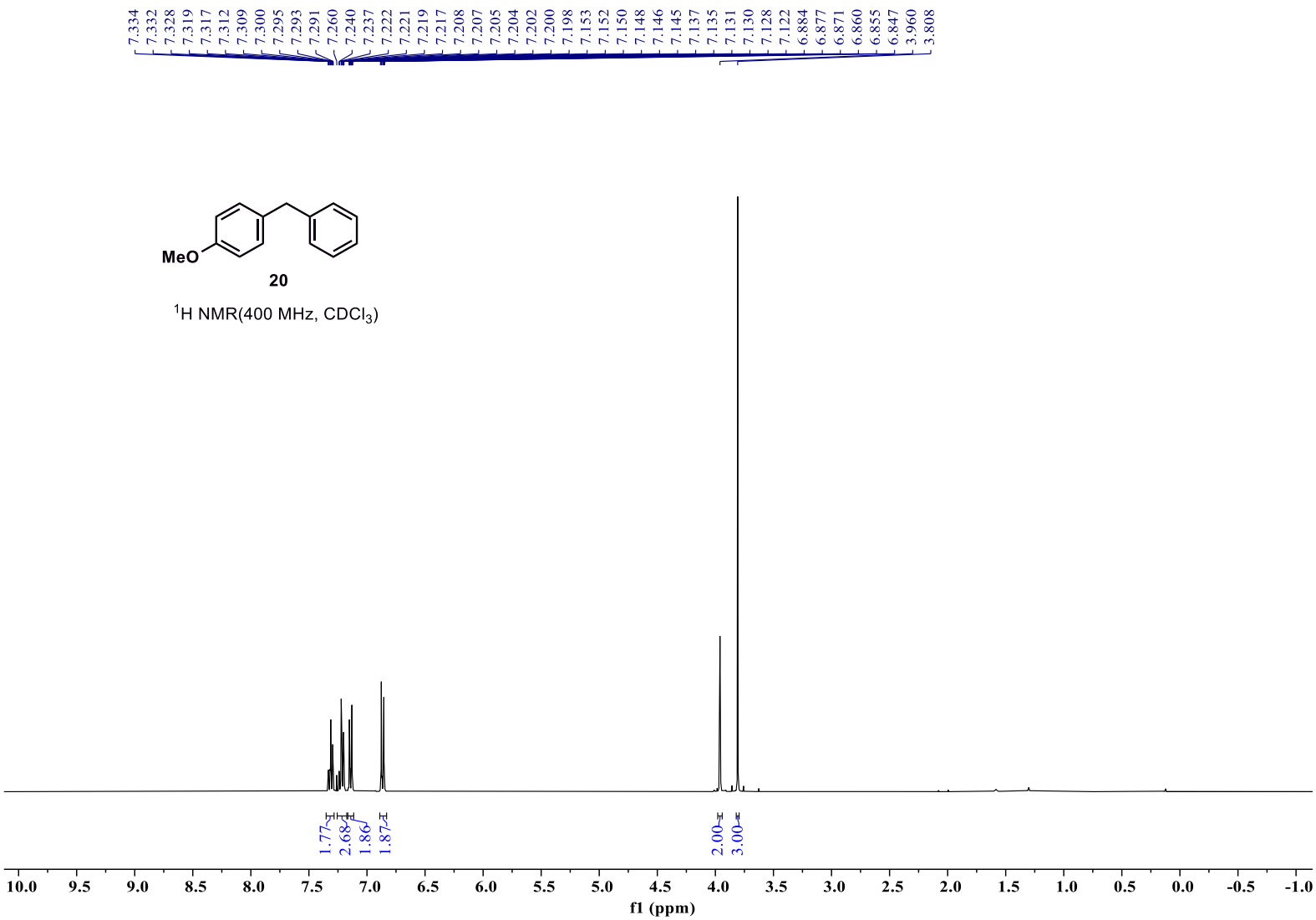

176.71
139.49
129.09
128.49
126.45
77.48
77.16
76.84
51.73
41.56
39.84
16.88
Me
MeO2C
18
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


7.303
7.300
7.296
7.285
7.282
7.279
7.269
7.264
7.263
7.260
7.231
7.228
7.227
7.226
7.220
7.218
7.214
7.213
7.212
7.210
7.209
7.206
7.205
7.202
7.199
7.182
3.704
3.686
3.685
3.667
3.646
3.642
3.639
3.635
3.633
3.628
3.621
3.554
3.546
3.542
3.541
3.539
3.531
3.530
3.381
2.925
2.907
2.889
MeO
O
O
19
1H NMR(400 MHz, CDCl3)
1.92
2.93
2.01
6.01
2.01
3.00
2.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

19

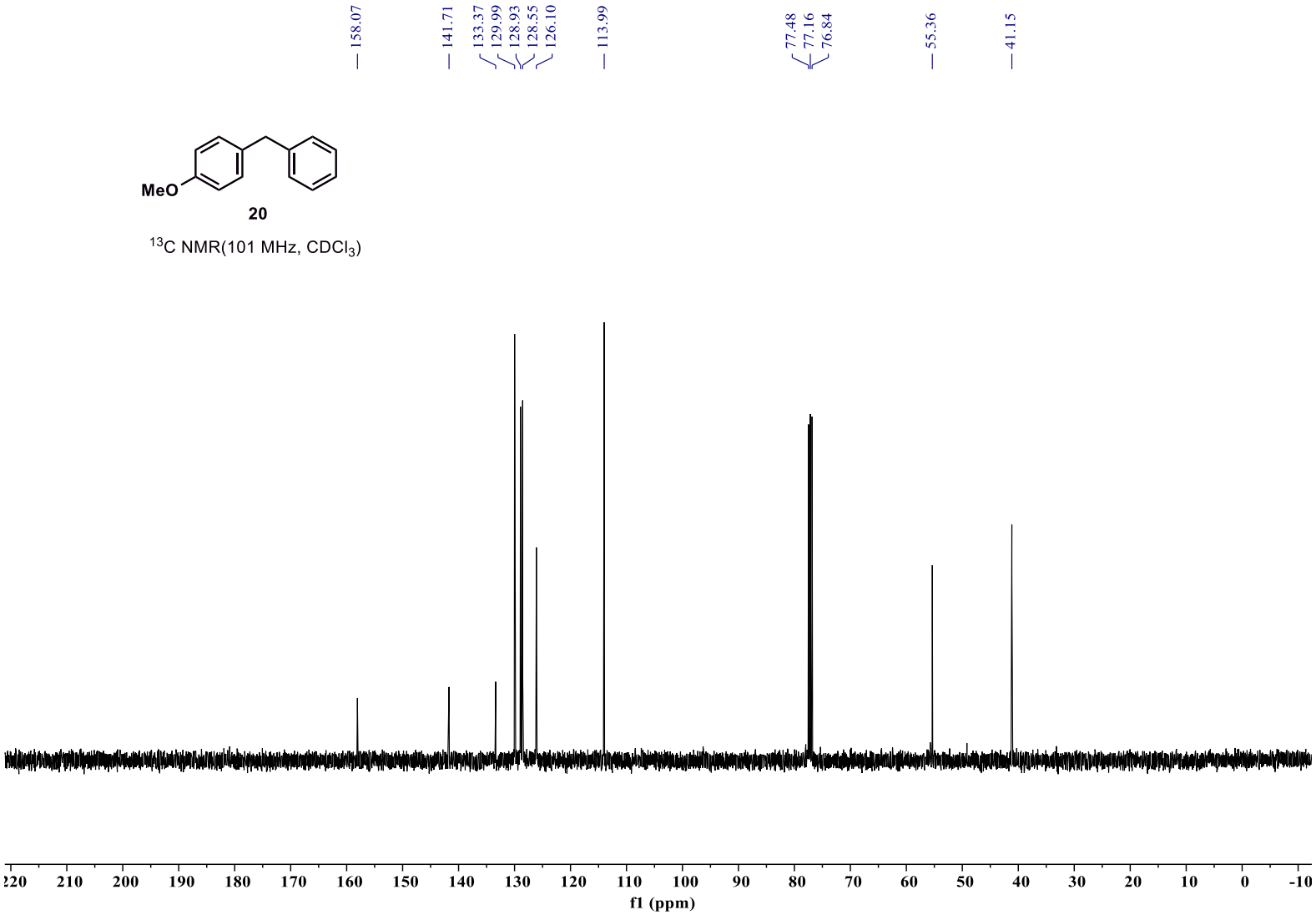
^{13}C NMR(101 MHz, $CDCl_3$)

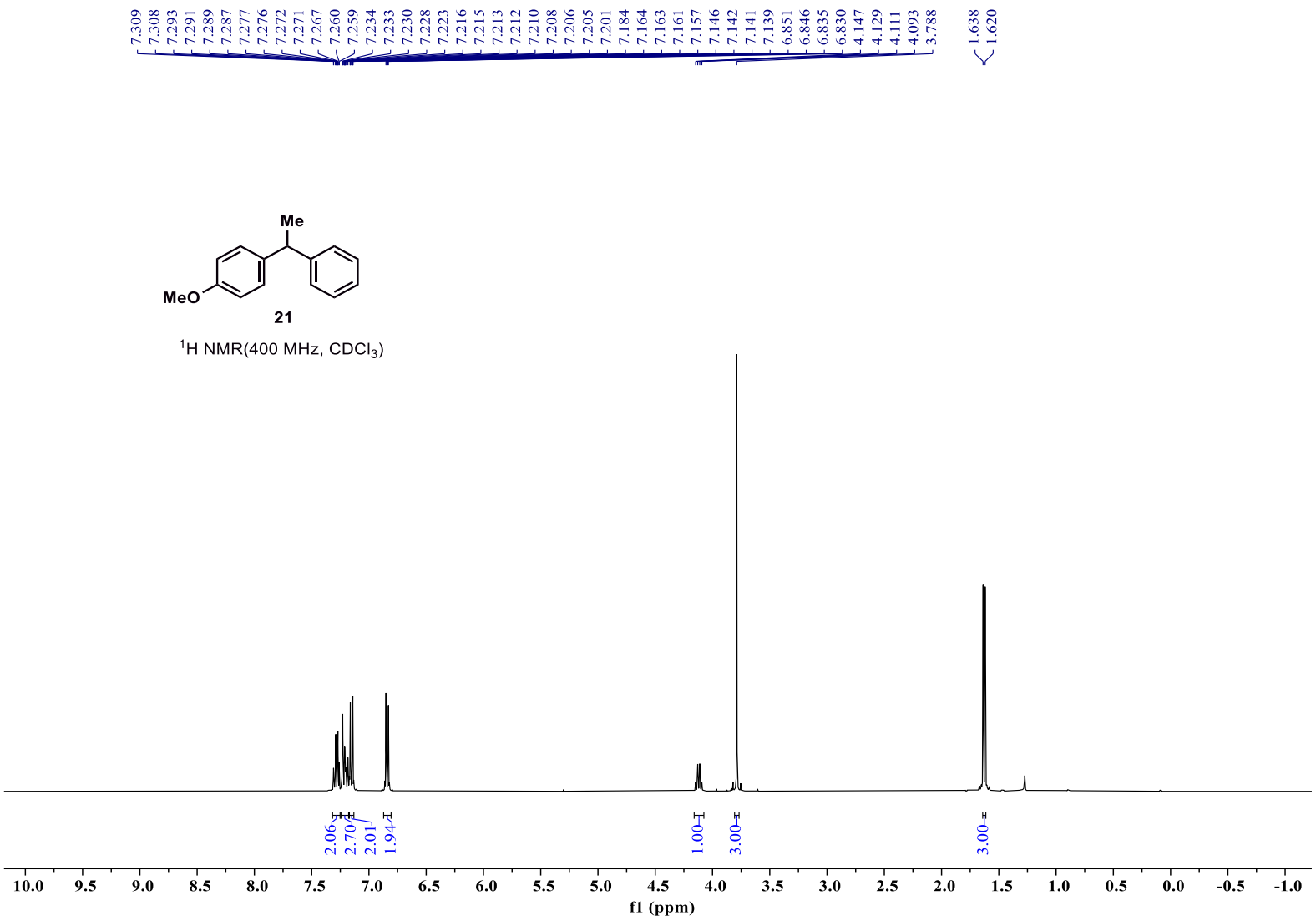


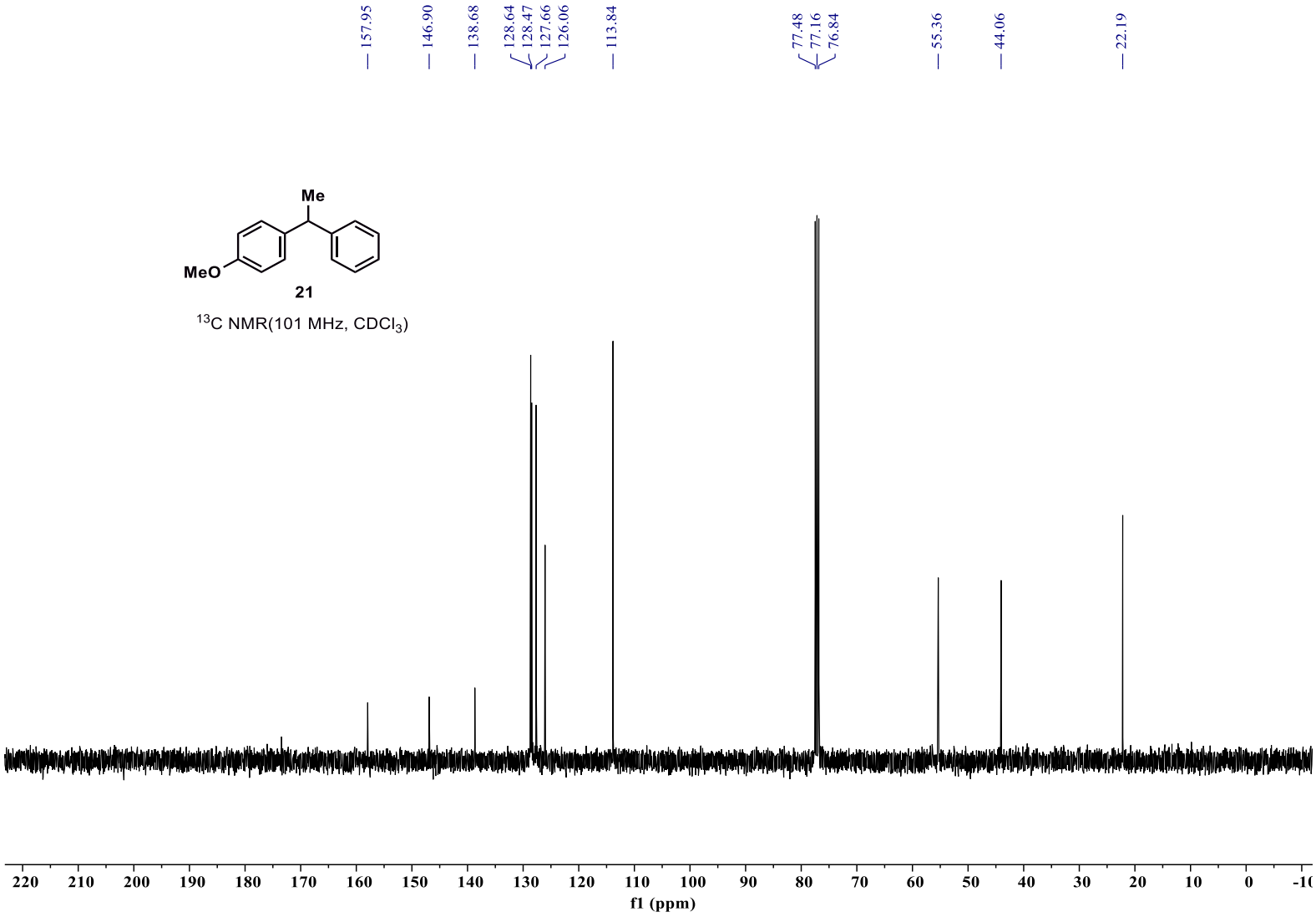

7.334
7.332
7.328
7.319
7.317
7.312
7.309
7.300
7.295
7.293
7.291
7.260
7.240
7.237
7.222
7.221
7.219
7.217
7.208
7.207
7.205
7.204
7.202
7.200
7.198
7.153
7.152
7.150
7.148
7.146
7.145
7.137
7.135
7.131
7.130
7.128
7.122
6.884
6.877
6.871
6.860
6.855
6.847
3.960
3.808
MeO
20
1H NMR(400 MHz, CDCl3)
1.77
2.68
1.86
1.87
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

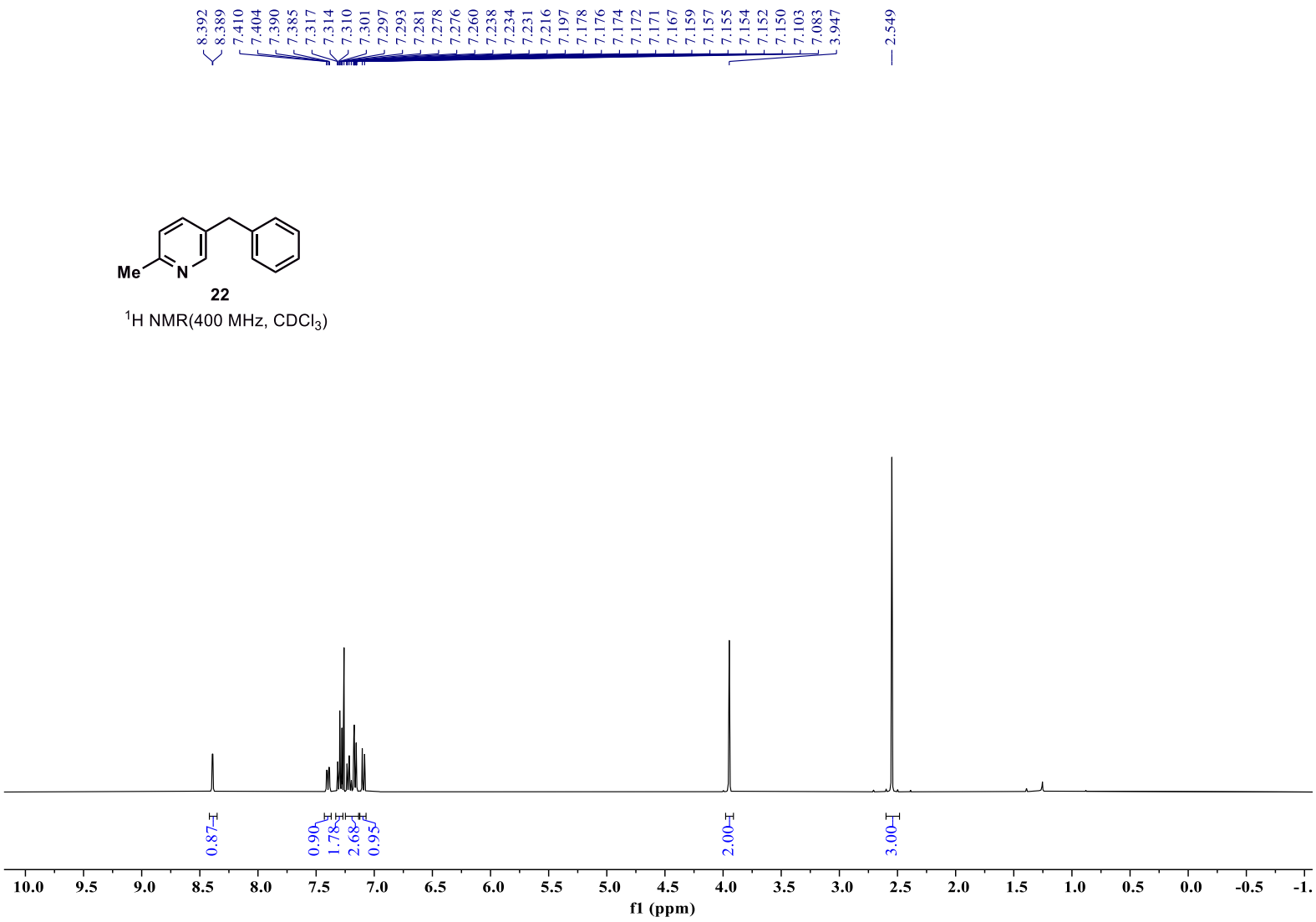
20

^{13}C NMR(101 MHz, $CDCl_3$)




7.309
7.308
7.293
7.291
7.289
7.287
7.277
7.276
7.272
7.271
7.267
7.260
7.259
7.234
7.233
7.230
7.228
7.223
7.216
7.215
7.213
7.212
7.210
7.208
7.206
7.205
7.201
7.184
7.164
7.163
7.161
7.157
7.146
7.142
7.141
7.139
6.851
6.846
6.835
6.830
4.147
4.129
4.111
4.093
3.788
1.638
1.620
Me
MeO
21
1H NMR(400 MHz, CDCl3)
2.06
2.70
2.01
1.94
1.00
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

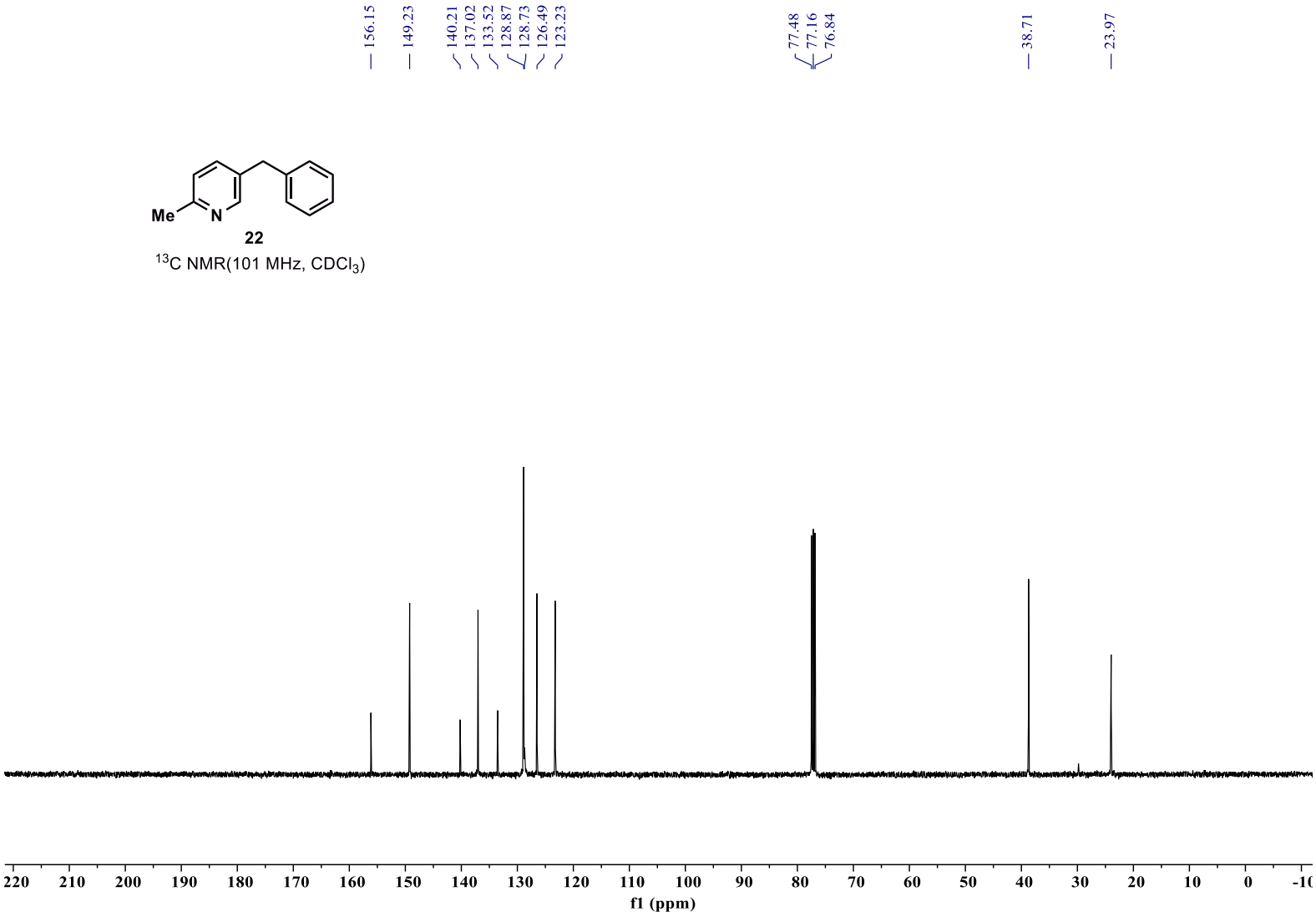

157.95
146.90
138.68
128.64
128.47
127.66
126.06
113.84
77.48
77.16
76.84
55.36
44.06
22.19
Me
MeO
21
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

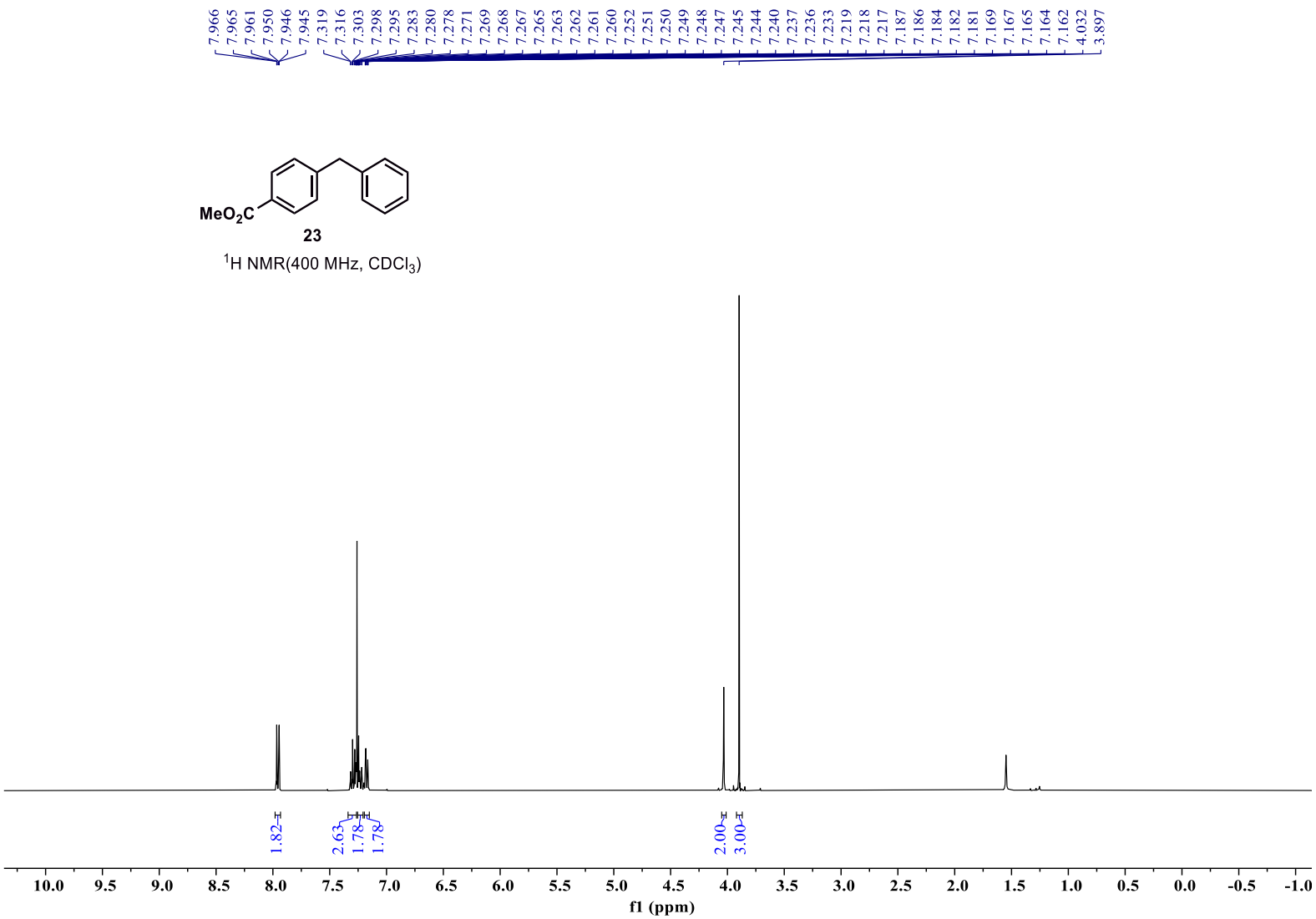


^{1}H NMR(400 MHz, $CDCl_3$)

22

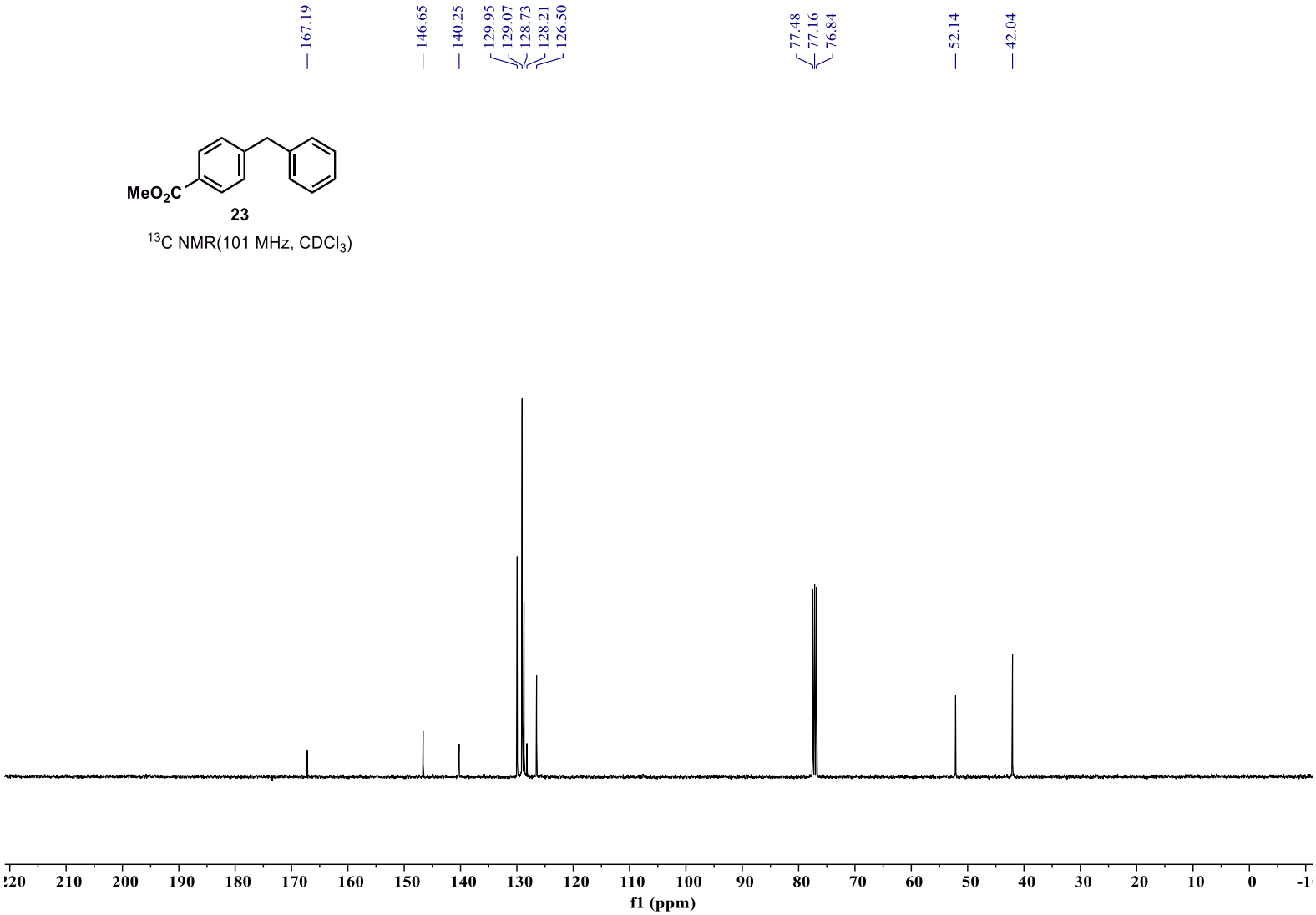
^{13}C NMR(101 MHz, $CDCl_3$)

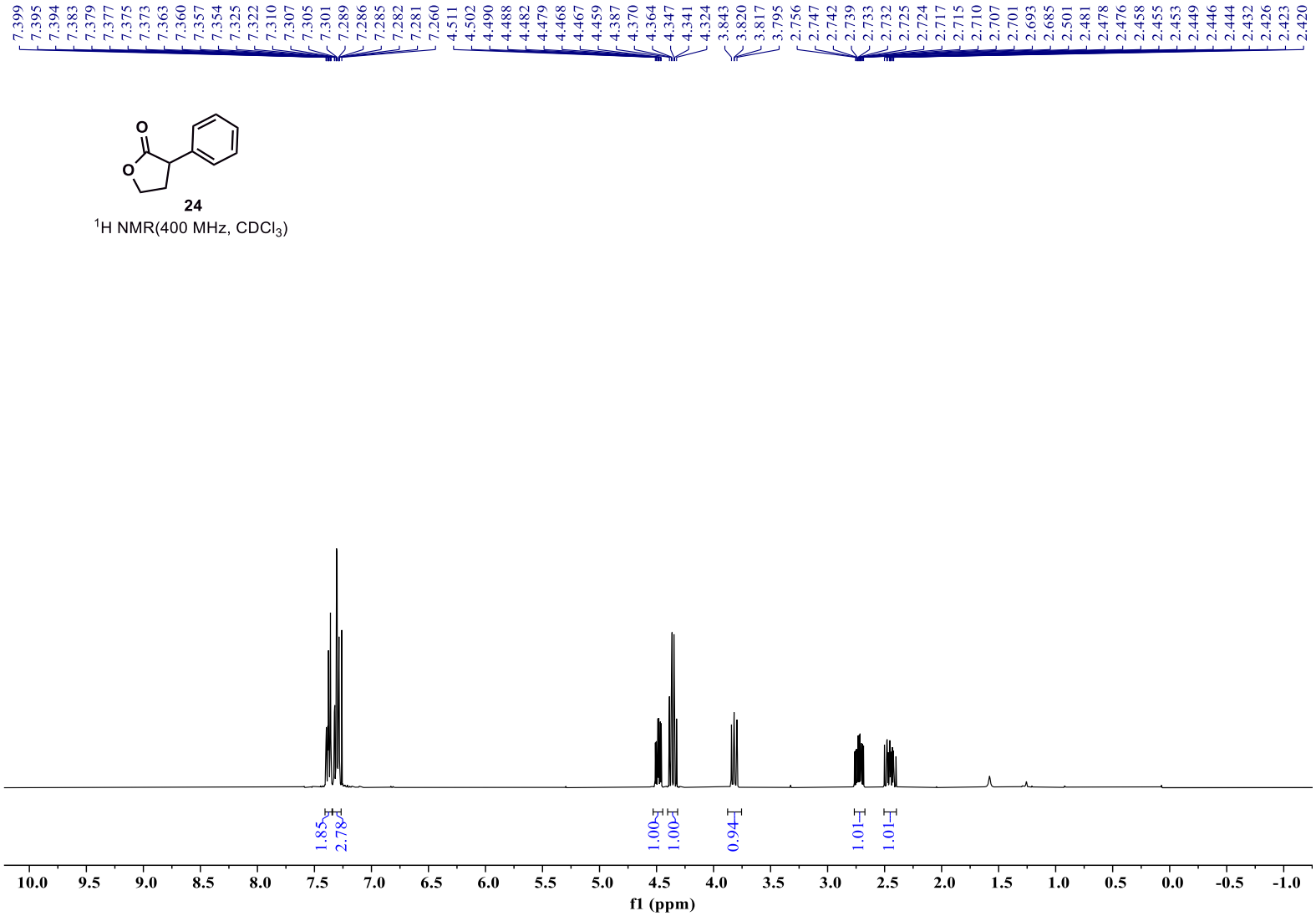


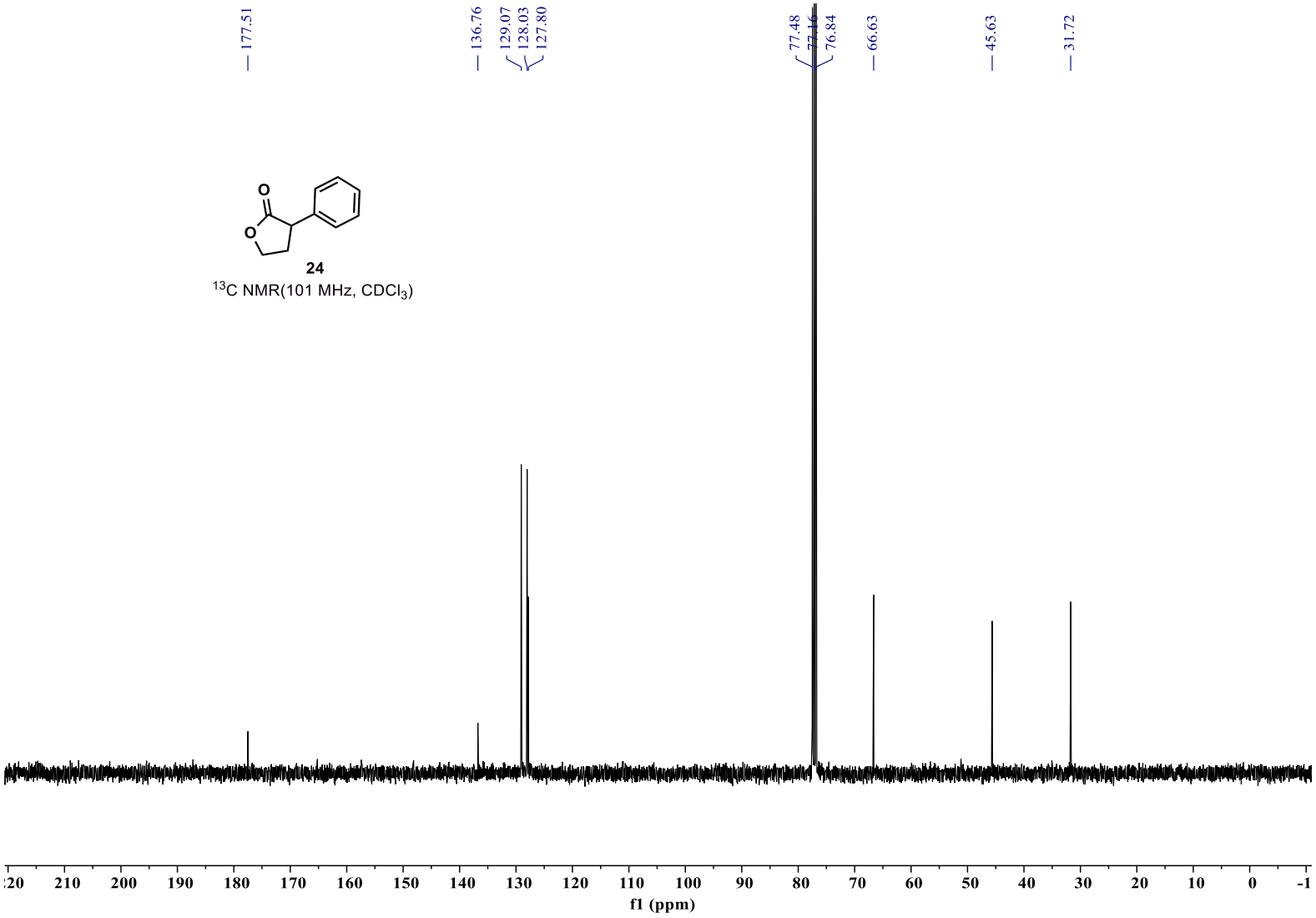

7.966
7.965
7.961
7.950
7.946
7.945
7.319
7.316
7.303
7.298
7.295
7.283
7.280
7.278
7.271
7.269
7.268
7.267
7.265
7.263
7.262
7.261
7.260
7.252
7.251
7.250
7.249
7.248
7.247
7.245
7.244
7.240
7.237
7.236
7.233
7.219
7.218
7.217
7.187
7.186
7.184
7.182
7.181
7.169
7.167
7.165
7.164
7.162
4.032
3.897
MeO2C
23
1H NMR(400 MHz, CDCl3)
1.82
2.63
1.78
1.78
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

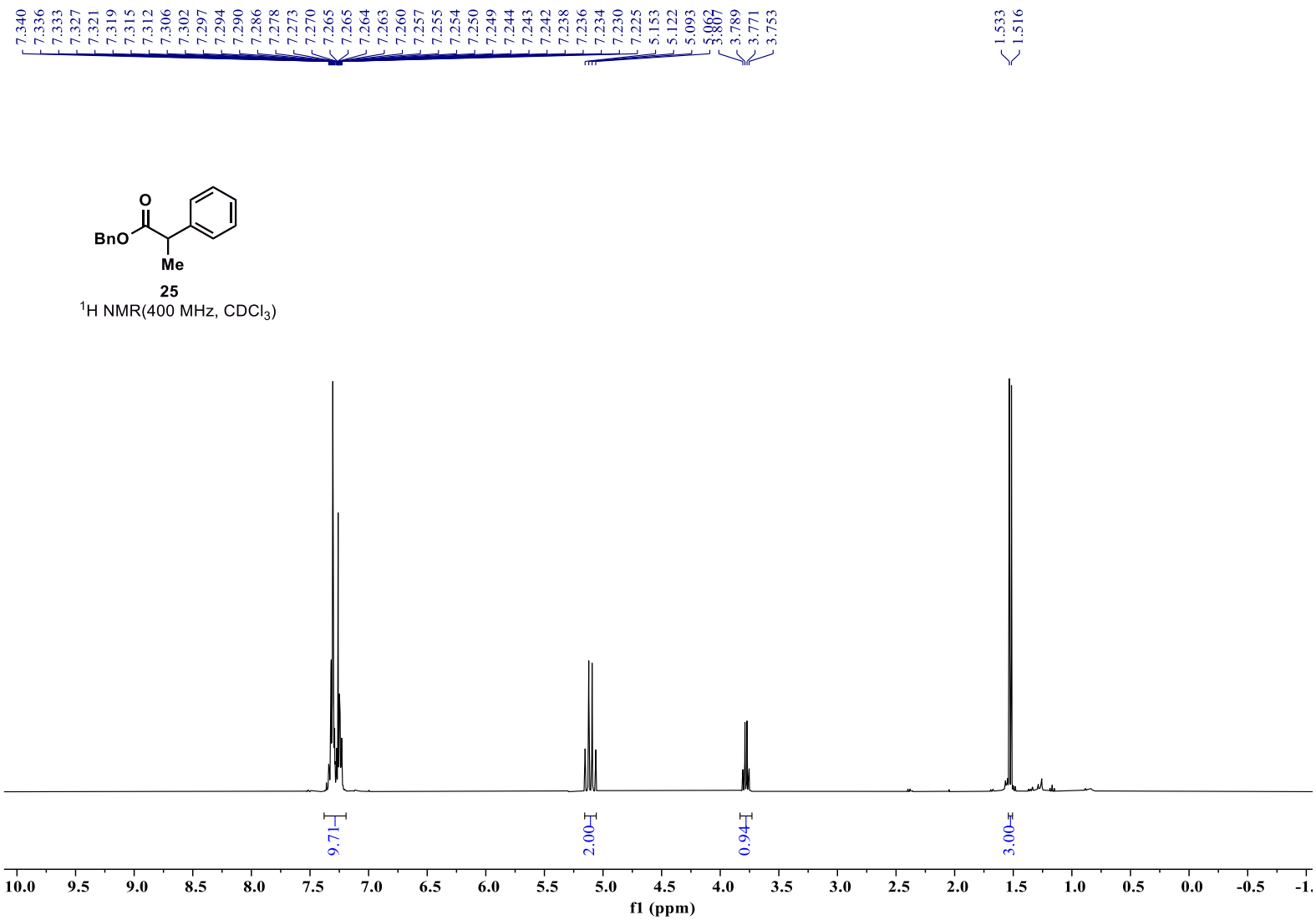
23

^{13}C NMR(101 MHz, $CDCl_3$)



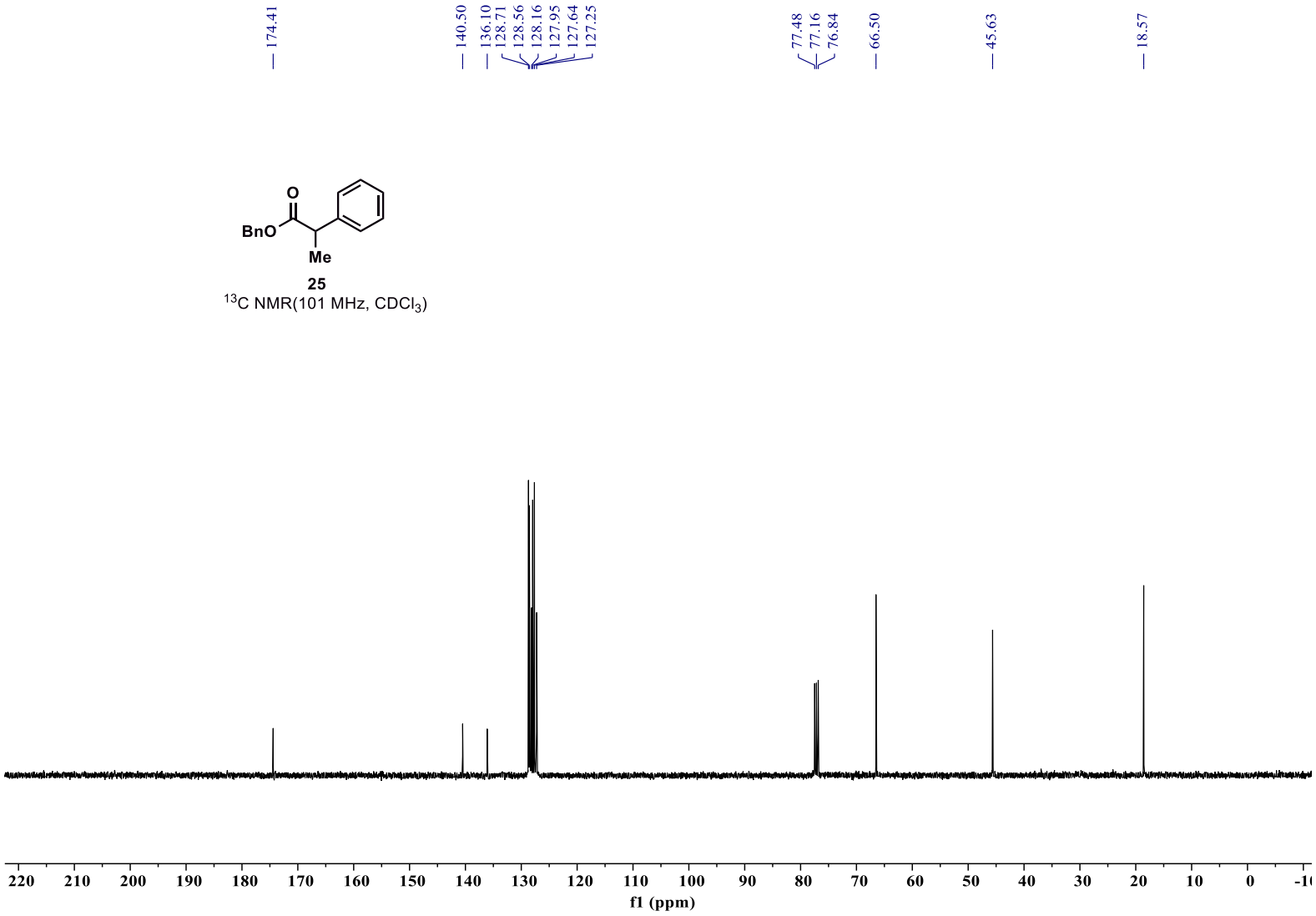

7.399
7.395
7.394
7.383
7.379
7.377
7.375
7.373
7.363
7.360
7.357
7.354
7.325
7.322
7.310
7.307
7.305
7.301
7.289
7.286
7.285
7.282
7.281
7.260
4.511
4.502
4.490
4.488
4.482
4.479
4.468
4.467
4.459
4.387
4.370
4.364
4.347
4.341
4.324
3.843
3.820
3.817
3.795
2.756
2.747
2.742
2.739
2.733
2.732
2.725
2.724
2.717
2.715
2.710
2.707
2.701
2.693
2.685
2.501
2.481
2.478
2.476
2.458
2.455
2.453
2.449
2.446
2.444
2.432
2.426
2.423
2.420
O
O
24
1H NMR(400 MHz, CDCl3)
1.85
2.78
1.00
1.00
0.94
1.01
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

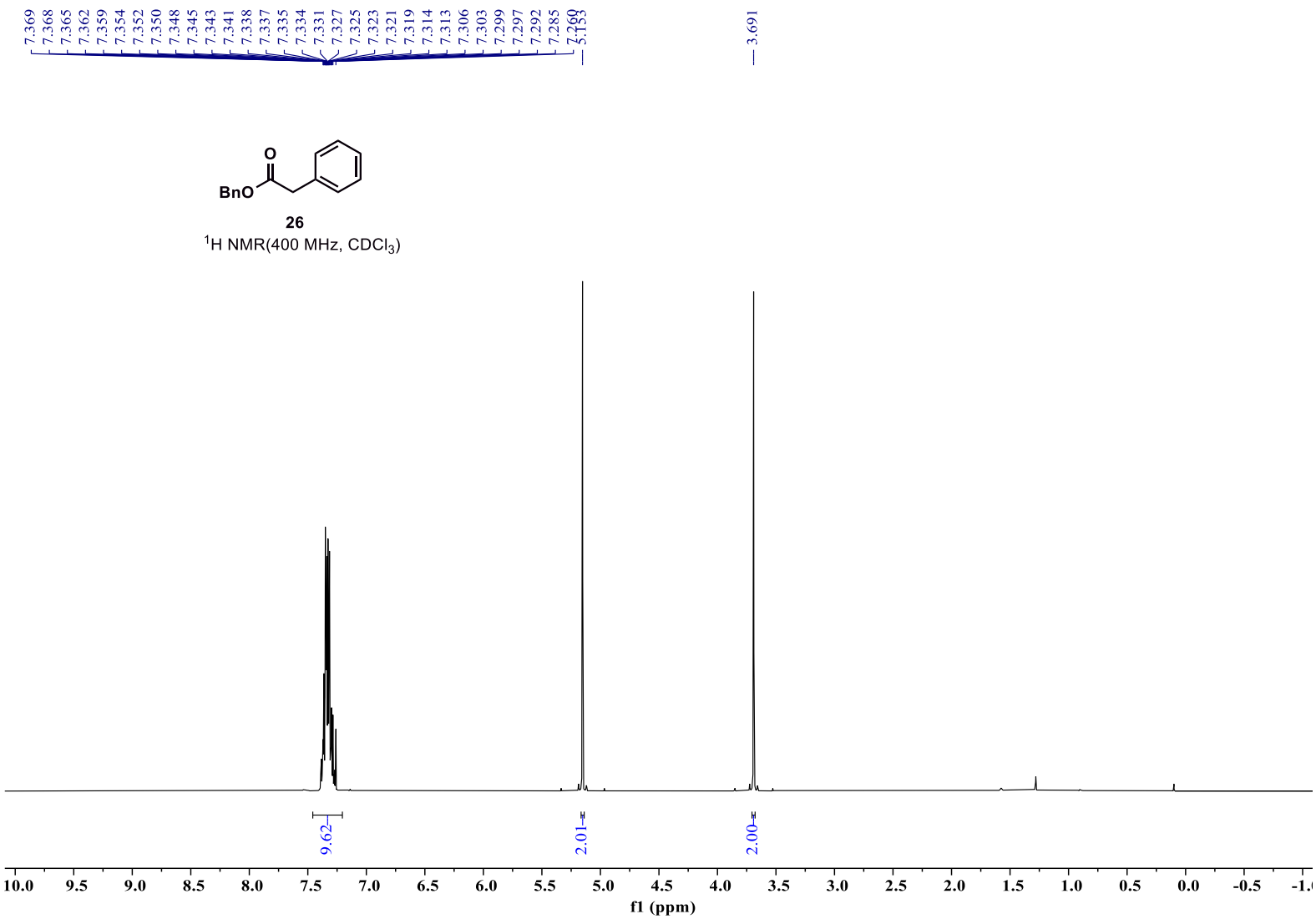

177.51
136.76
129.07
128.03
127.80
77.48
77.16
76.84
66.63
45.63
31.72
O
O
24
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



25

^{1}H NMR(400 MHz, $CDCl_3$)


174.41
140.50
136.10
128.71
128.56
128.16
127.95
127.64
127.25
77.48
77.16
76.84
66.50
45.63
18.57
BnO
O
Me
25
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

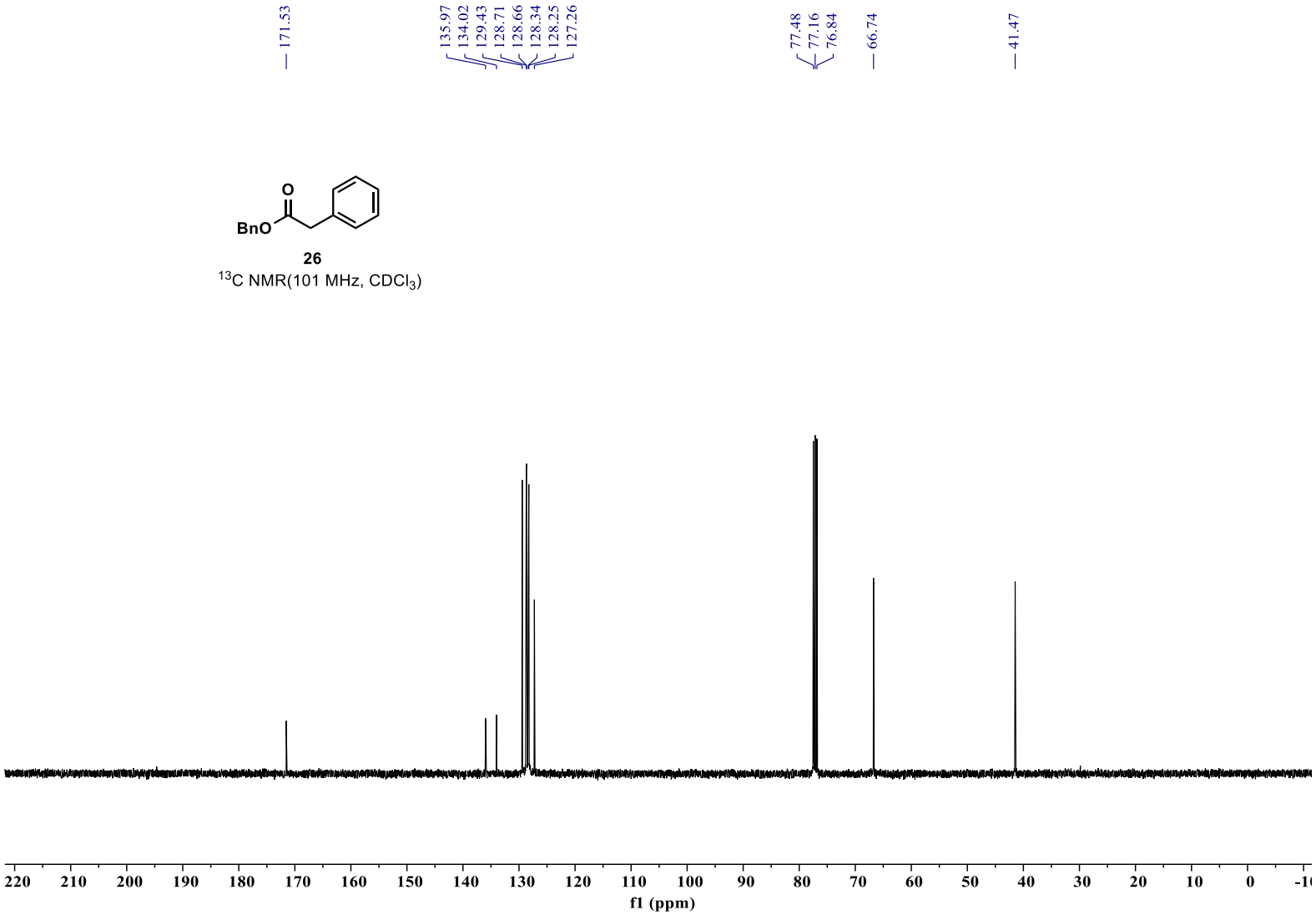


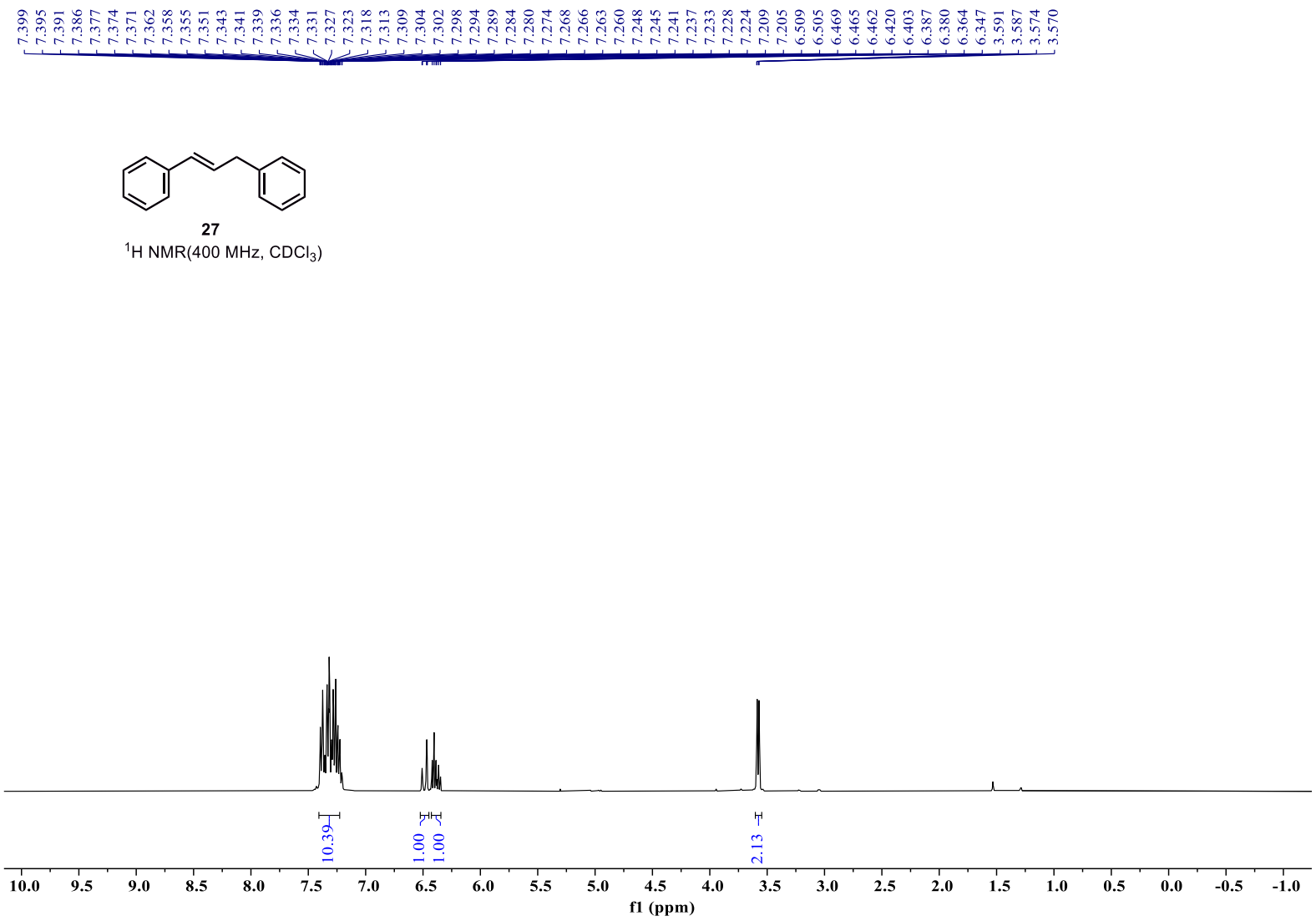
26

^{1}H NMR(400 MHz, $CDCl_3$)

26

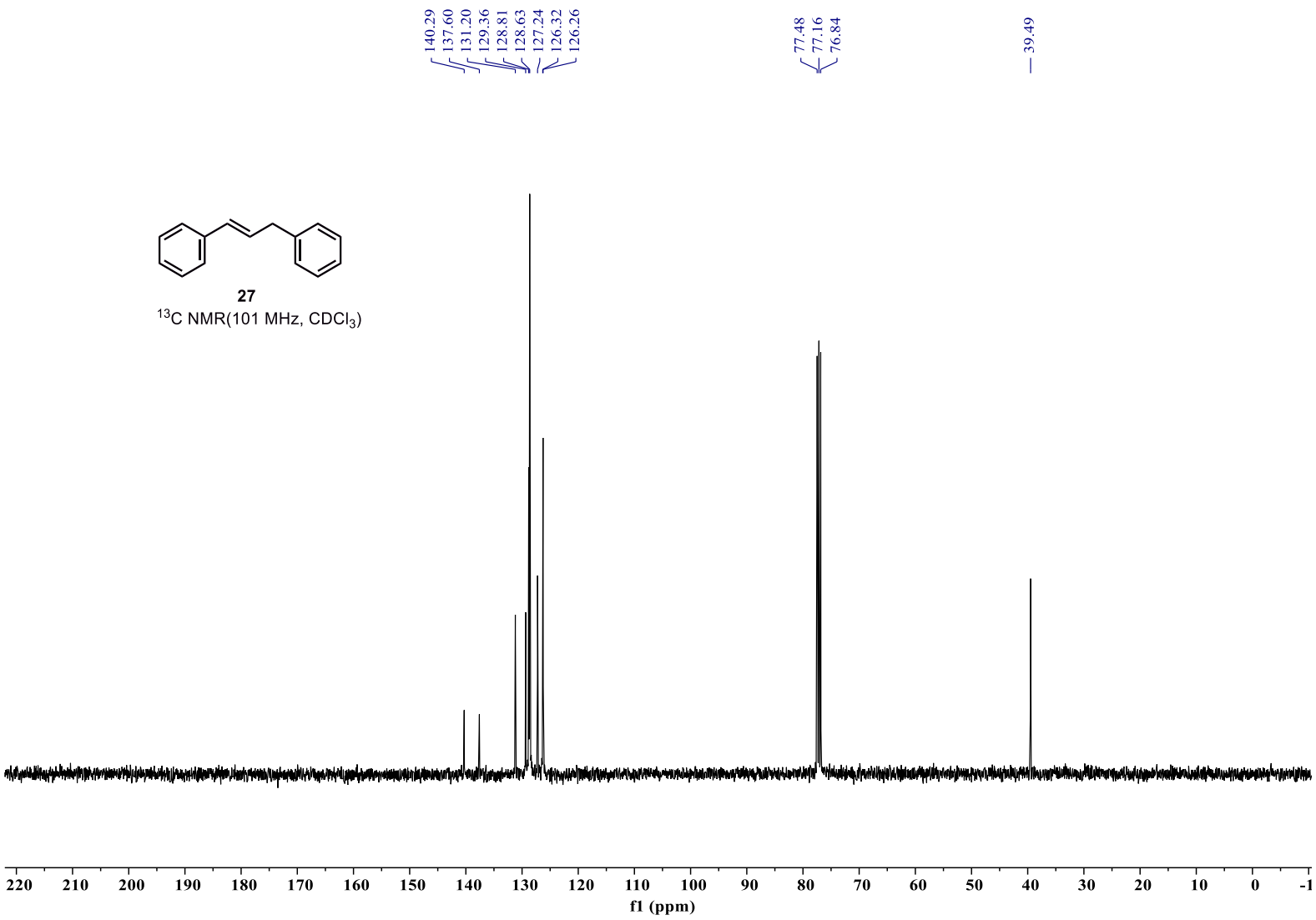
^{13}C NMR(101 MHz, $CDCl_3$)

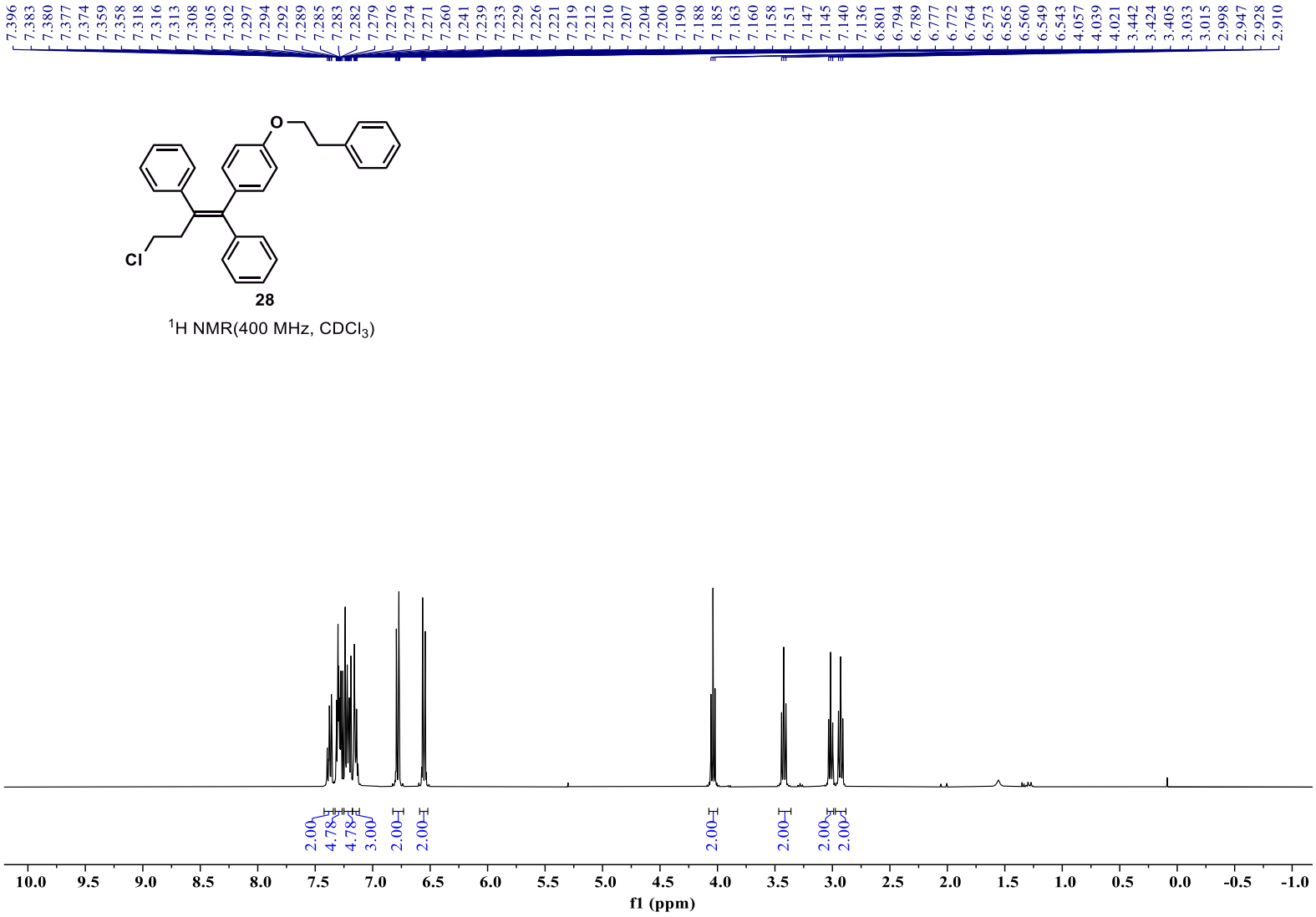




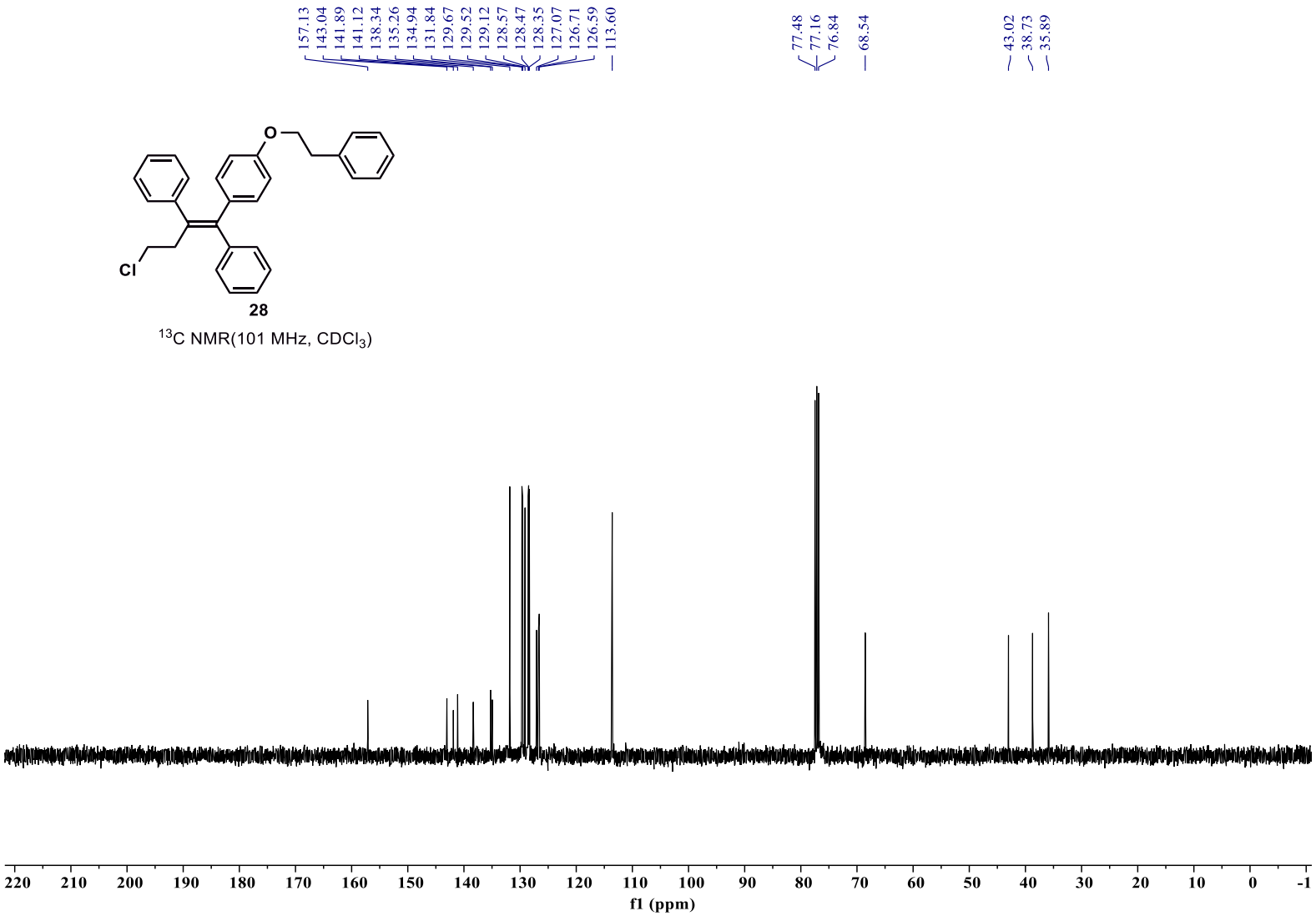
27

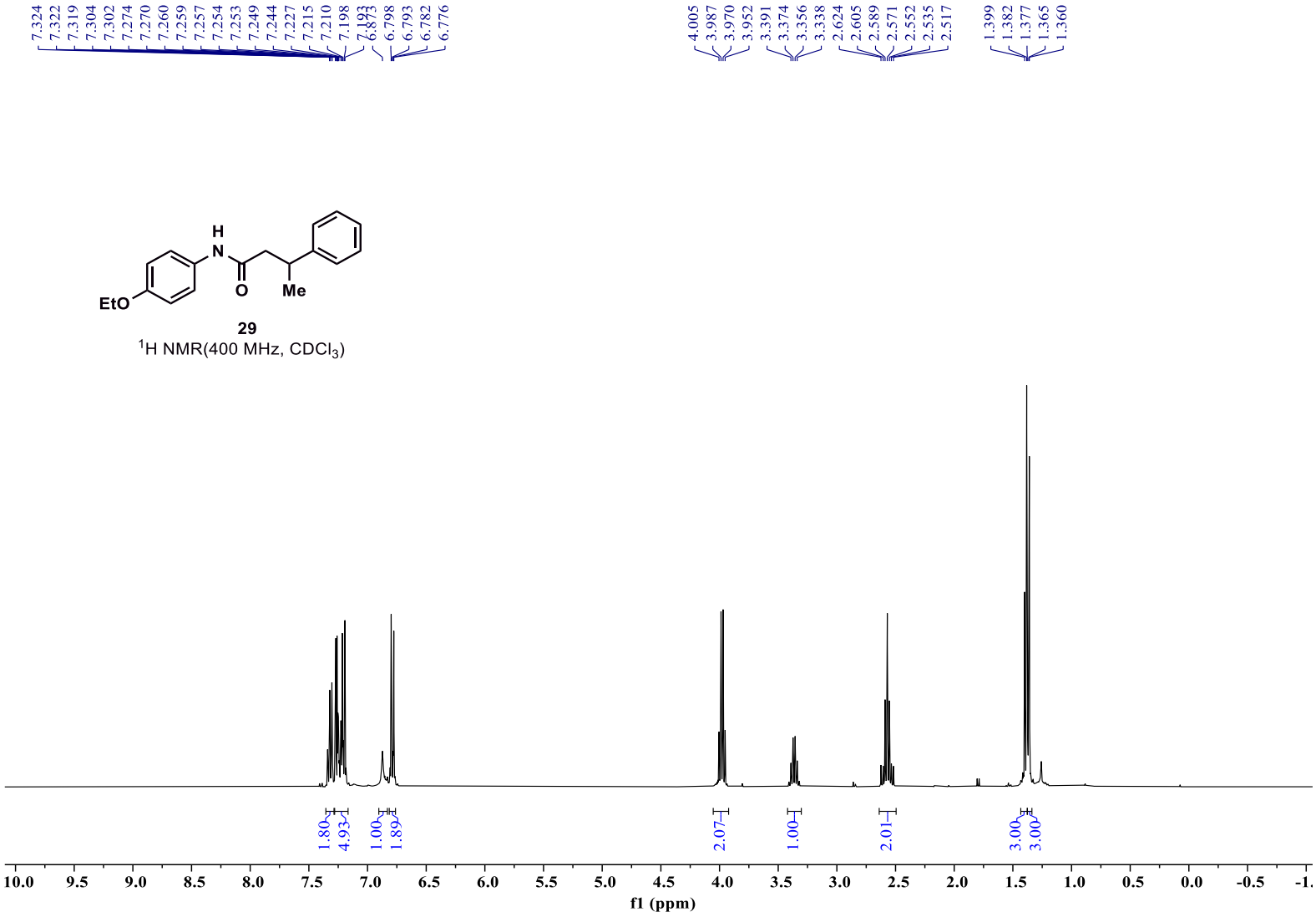
^{1}H NMR(400 MHz, $CDCl_3$)


140.29
137.60
131.20
129.36
128.81
128.63
127.24
126.32
126.26
77.48
77.16
76.84
39.49
27
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



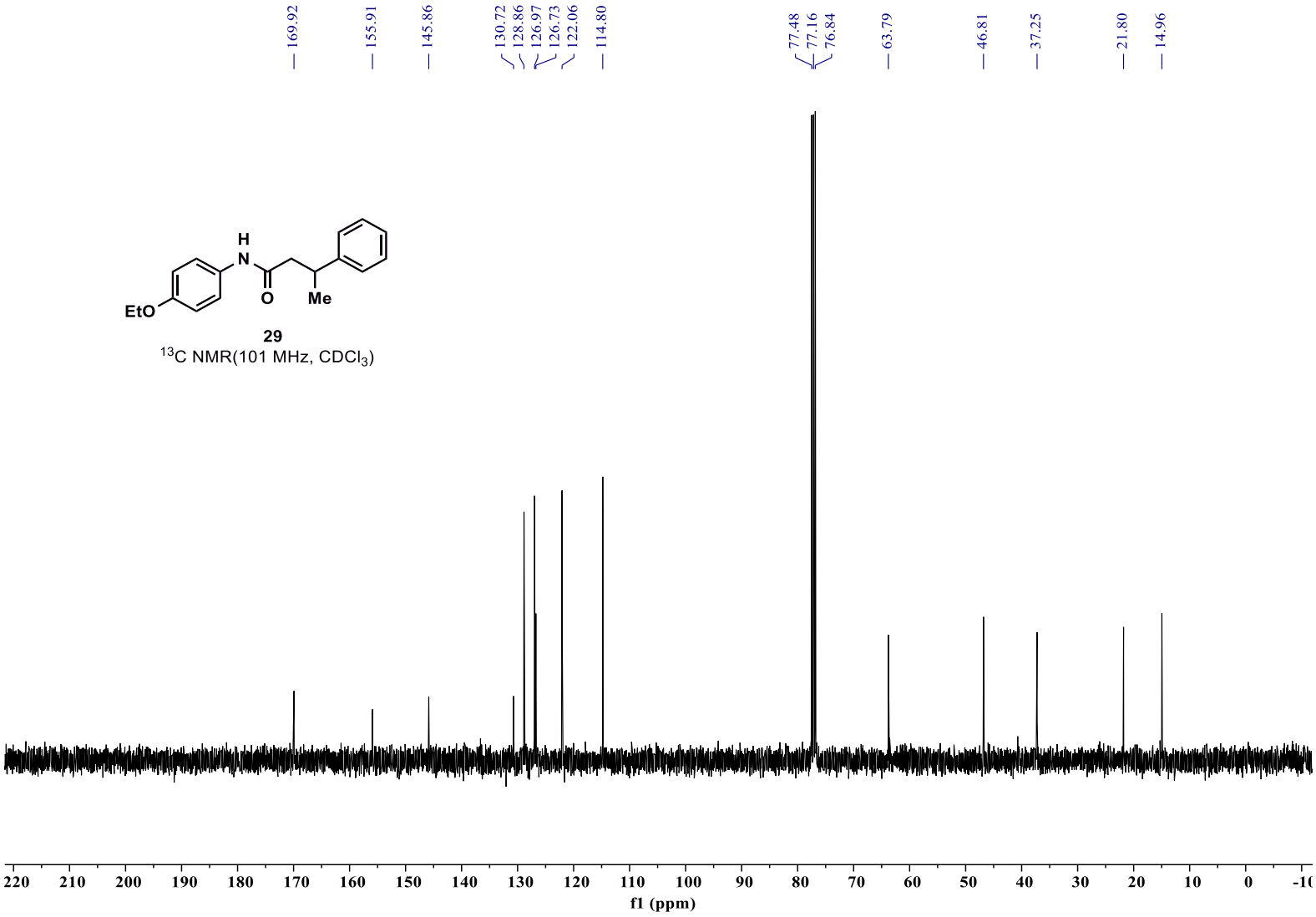
^{1}H NMR(400 MHz, $CDCl_3$)


157.13
143.04
141.89
141.12
138.34
135.26
134.94
131.84
129.67
129.52
129.12
128.57
128.47
128.35
127.07
126.71
126.59
113.60
77.48
77.16
76.84
68.54
43.02
38.73
35.89
O
Cl
28
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



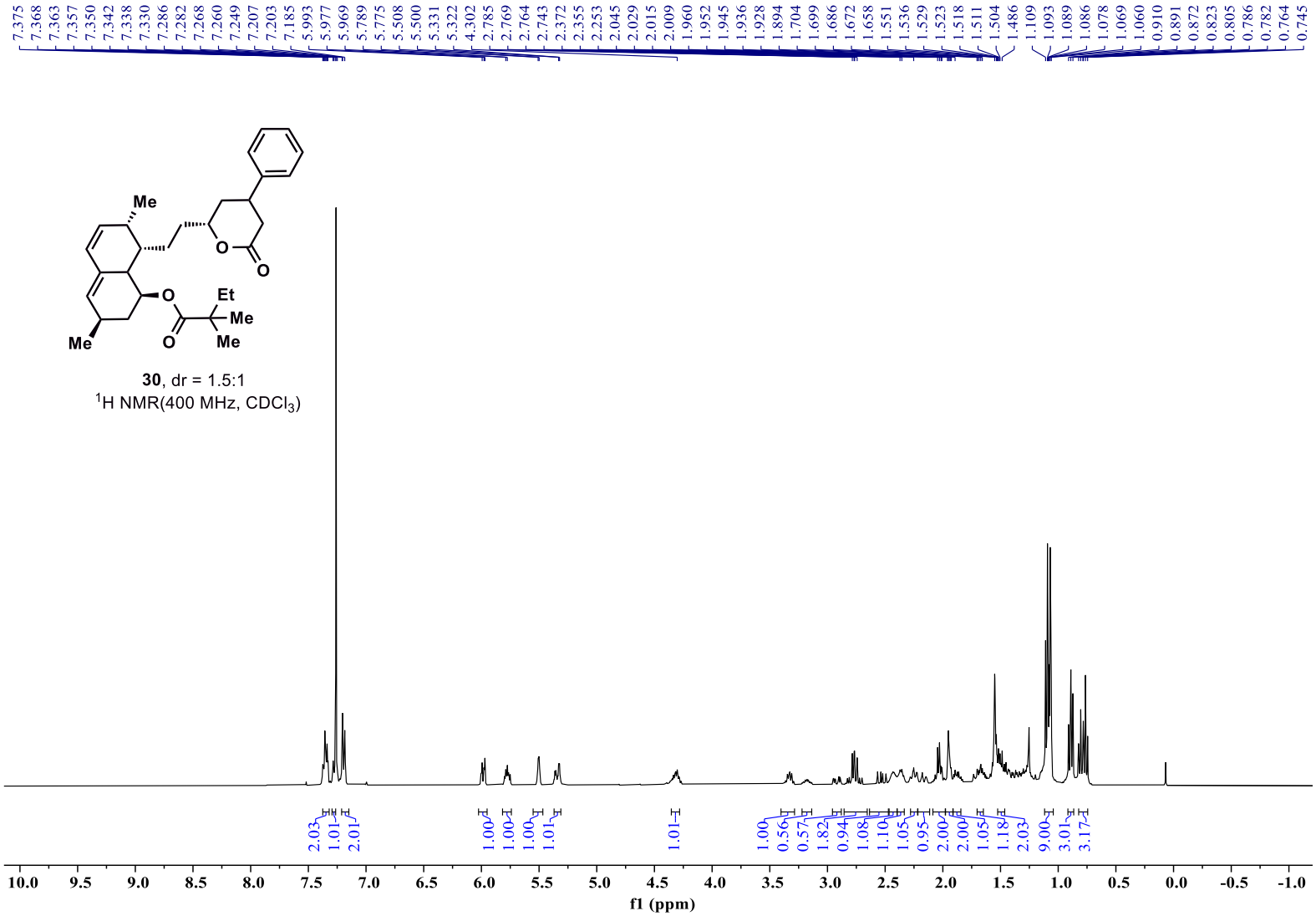
29

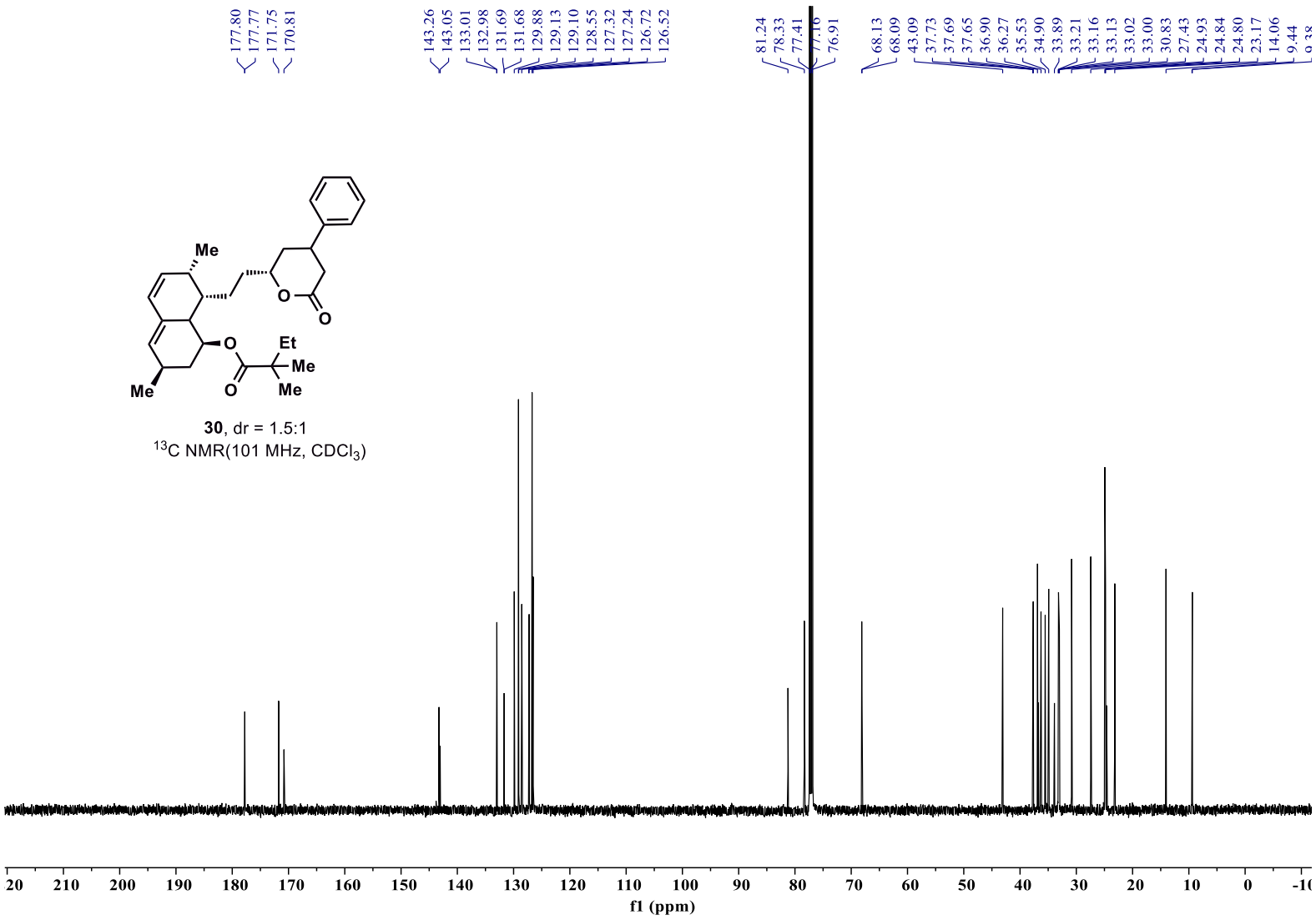
^{1}H NMR(400 MHz, $CDCl_3$)



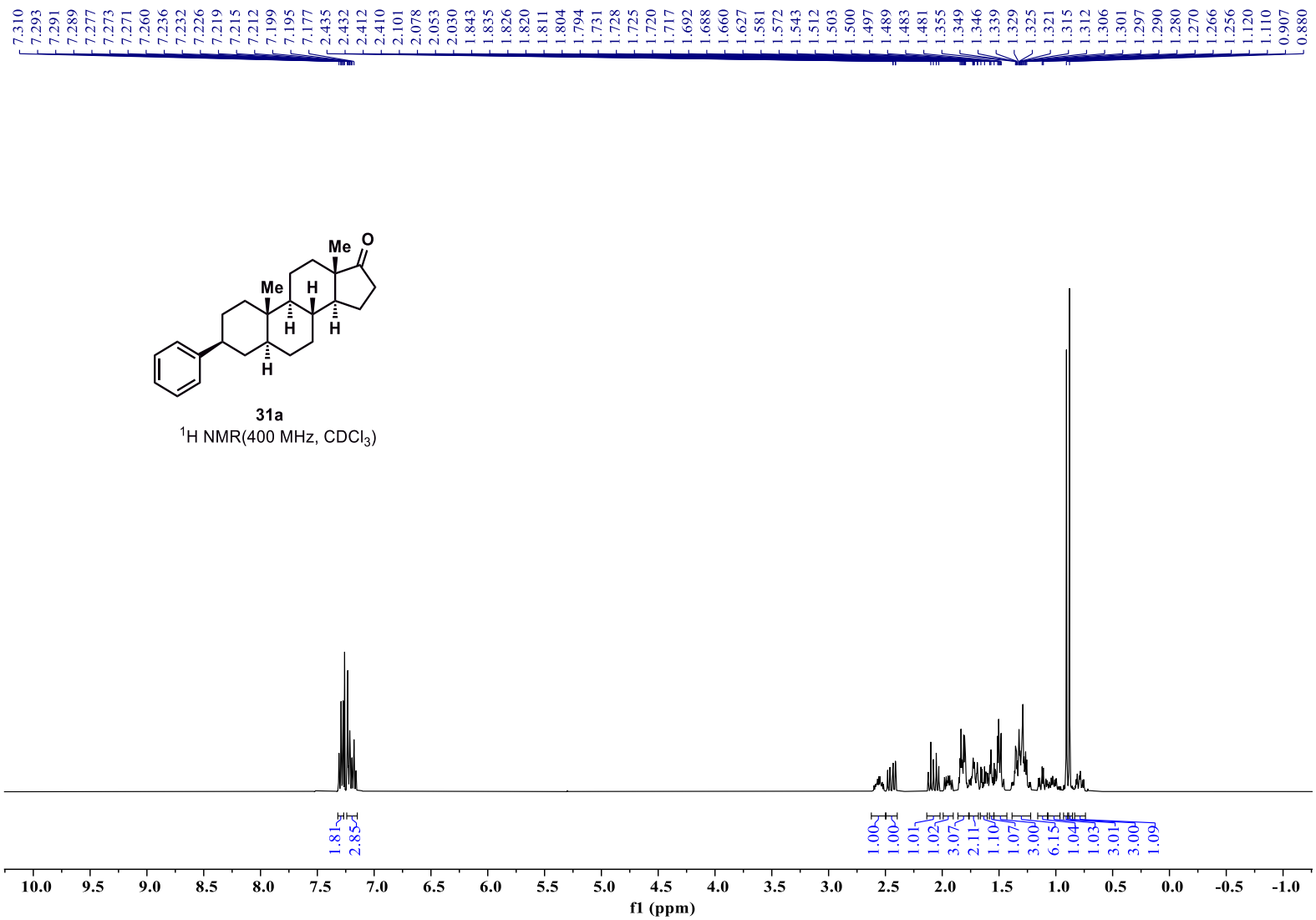
29

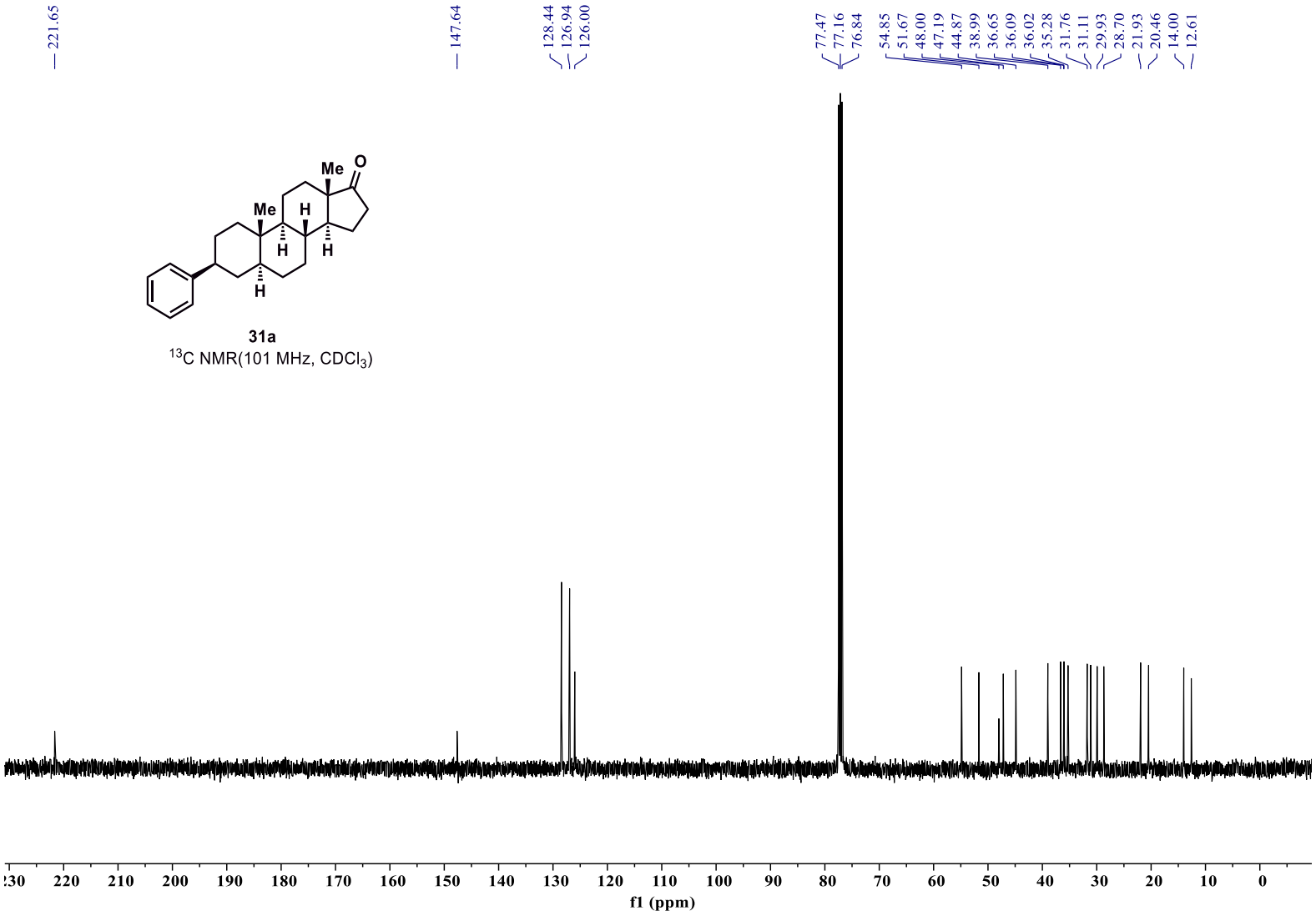
^{13}C NMR(101 MHz, $CDCl_3$)

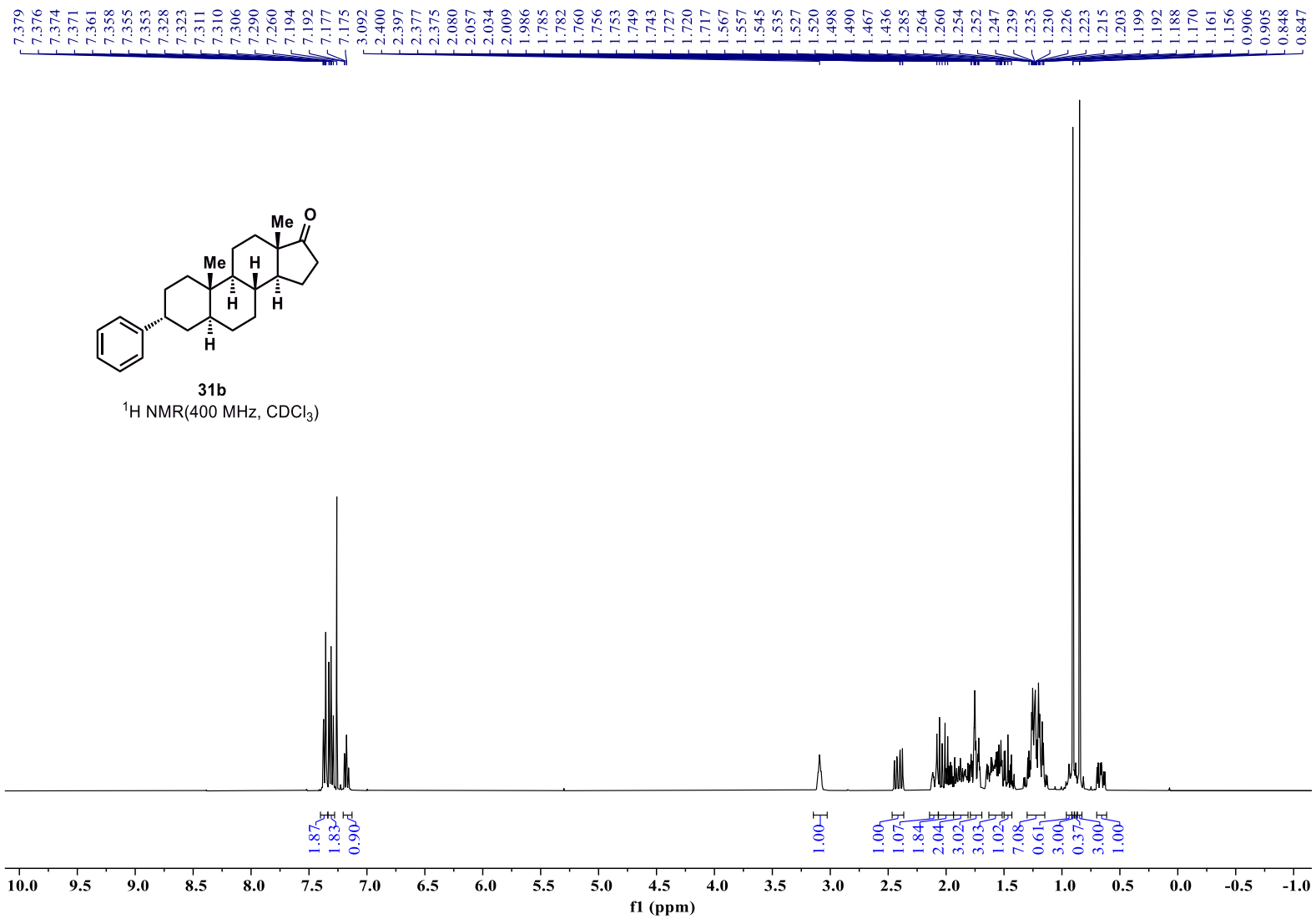

7.375 7.368 7.363 7.357 7.350 7.342 7.338 7.330 7.286 7.282 7.268 7.260 7.249 7.207 7.203 7.185 5.993 5.977 5.969 5.789 5.775 5.508 5.500 5.331 5.322 4.302 2.785 2.769 2.764 2.743 2.372 2.355 2.253 2.045 2.029 2.015 2.009 1.960 1.952 1.945 1.936 1.928 1.894 1.704 1.699 1.686 1.672 1.658 1.551 1.536 1.529 1.523 1.518 1.511 1.504 1.486 1.109 1.093 1.089 1.086 1.078 1.069 1.060 0.910 0.891 0.872 0.823 0.805 0.786 0.782 0.764 0.745
Me
O
O
O
Et
Me
Me
O
Me
30, dr = 1.5:1
1H NMR(400 MHz, CDCl3)
2.03 1.01 2.01 1.00 1.00 1.00 1.01 1.01 1.00 0.56 0.57 1.82 0.94 1.08 1.10 1.05 0.95 2.00 2.00 1.05 1.18 2.03 9.00 3.01 3.17
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

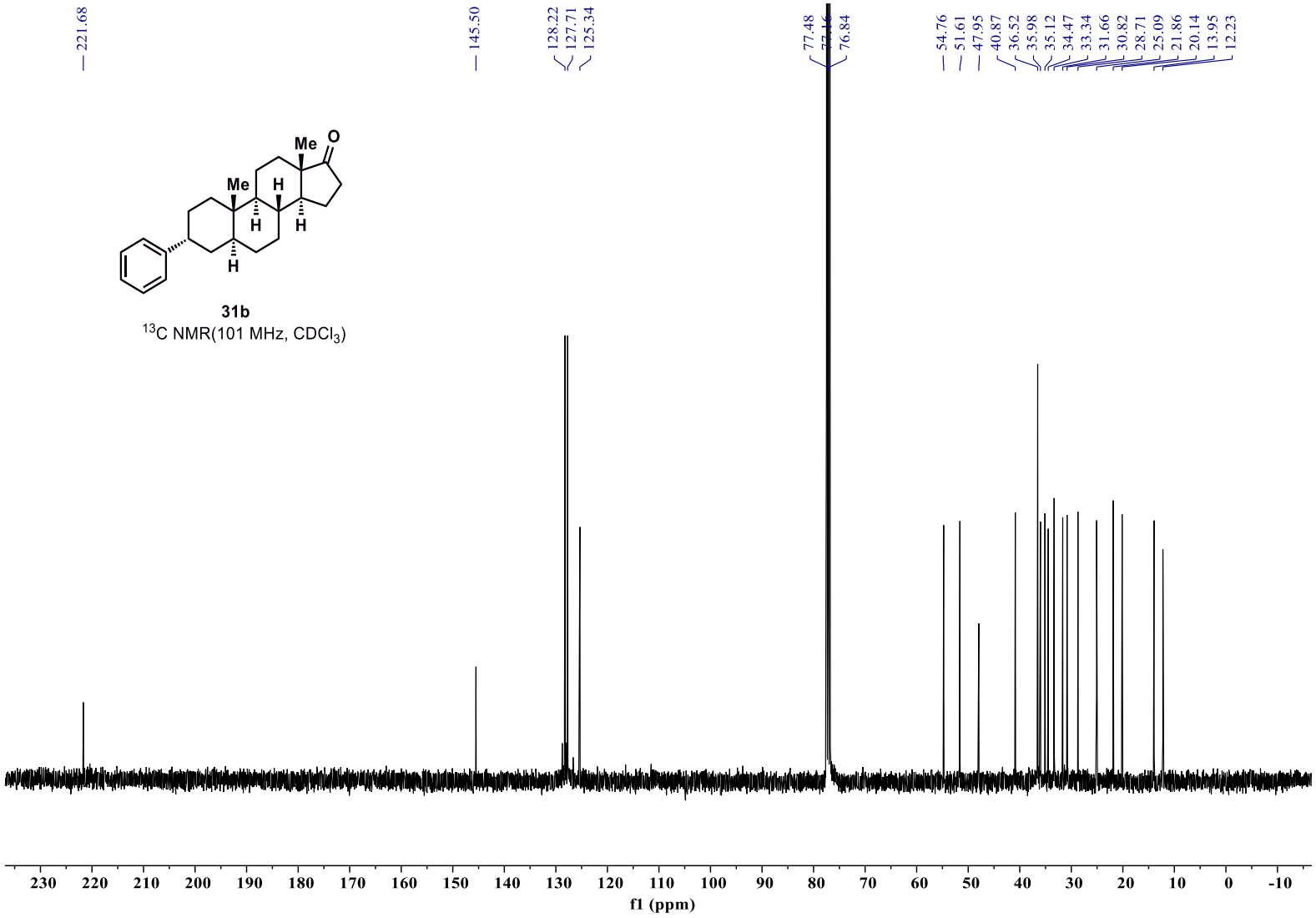


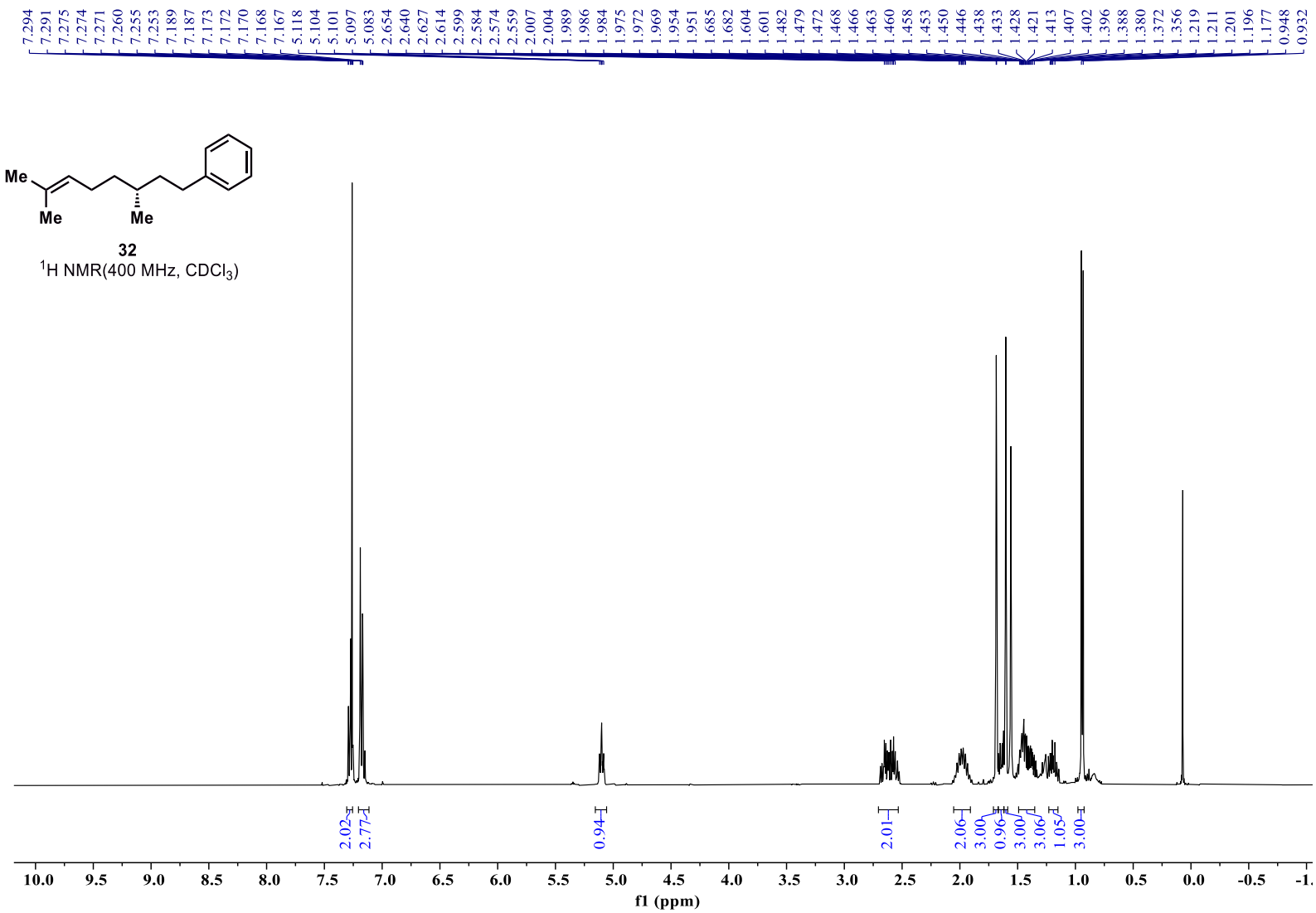
30, dr = 1.5:1
^{13}C NMR(101 MHz, $CDCl_3$)


31a
^{1}H NMR(400 MHz, $CDCl_3$)
f1 (ppm)


221.65
147.64
128.44
126.94
126.00
77.47
77.16
76.84
54.85
51.67
48.00
47.19
44.87
38.99
36.65
36.09
36.02
35.28
31.76
31.11
29.93
28.70
21.93
20.46
14.00
12.61
Me
O
Me
H
H
H
H
31a
13C NMR(101 MHz, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

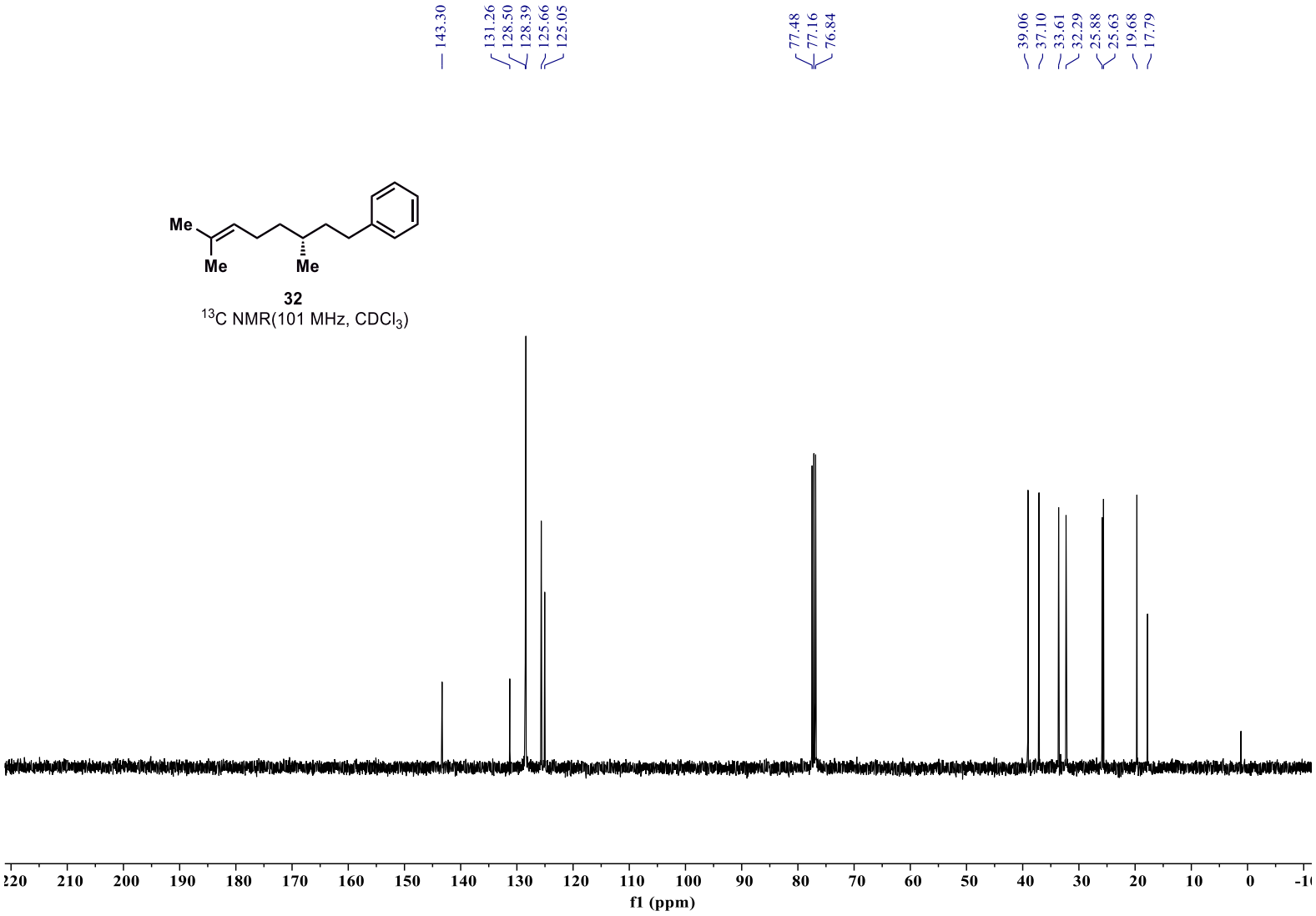

31b
^{1}H NMR(400 MHz, $CDCl_3$)
Me
O
Me
H
H
H
H
f1 (ppm)

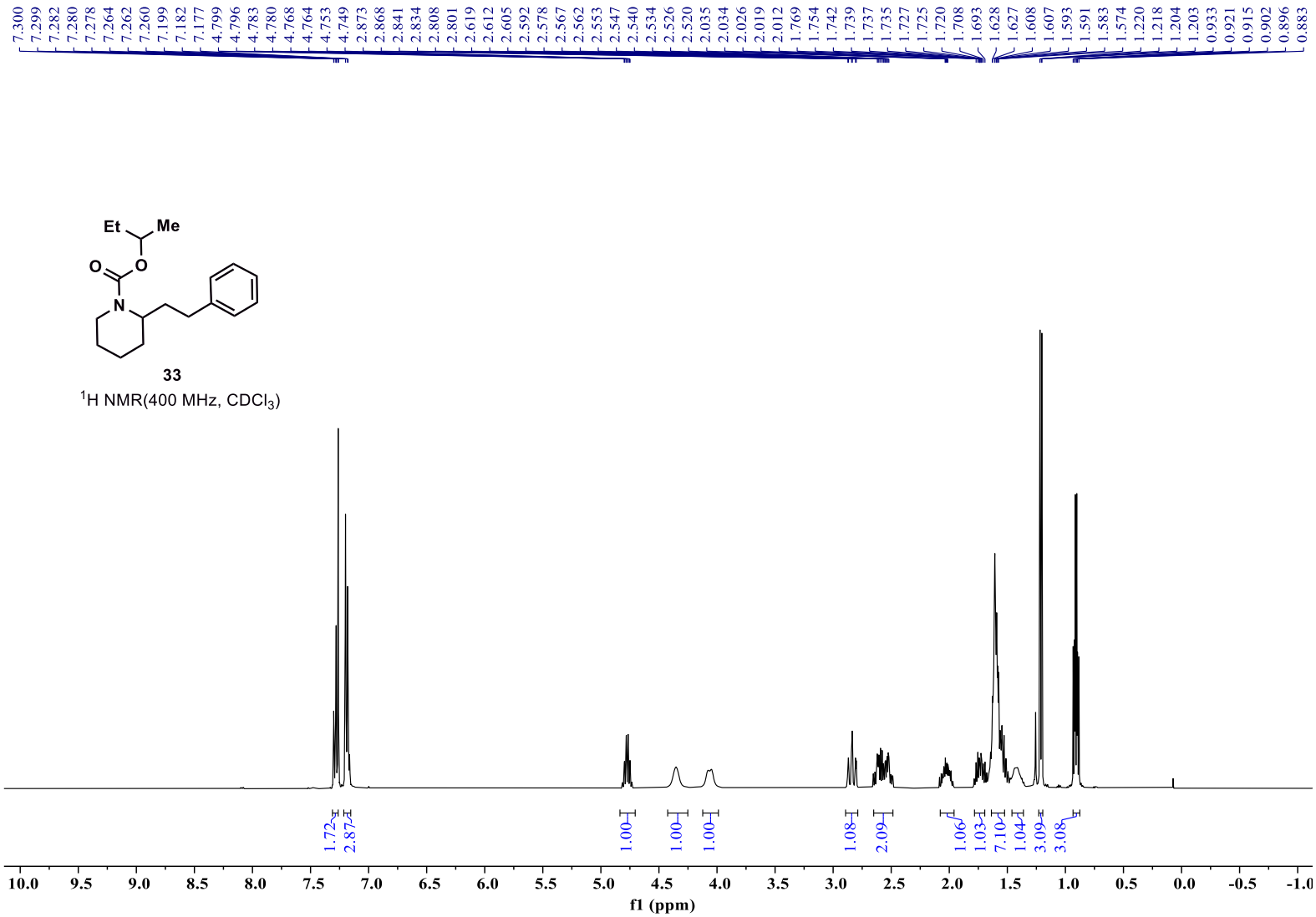

221.68
145.50
128.22
127.71
125.34
77.48
77.16
76.84
54.76
51.61
47.95
40.87
36.52
35.98
35.12
34.47
33.34
31.66
30.82
28.71
25.09
21.86
20.14
13.95
12.23
Me
O
Me
H
H
H
H
31b
13C NMR(101 MHz, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

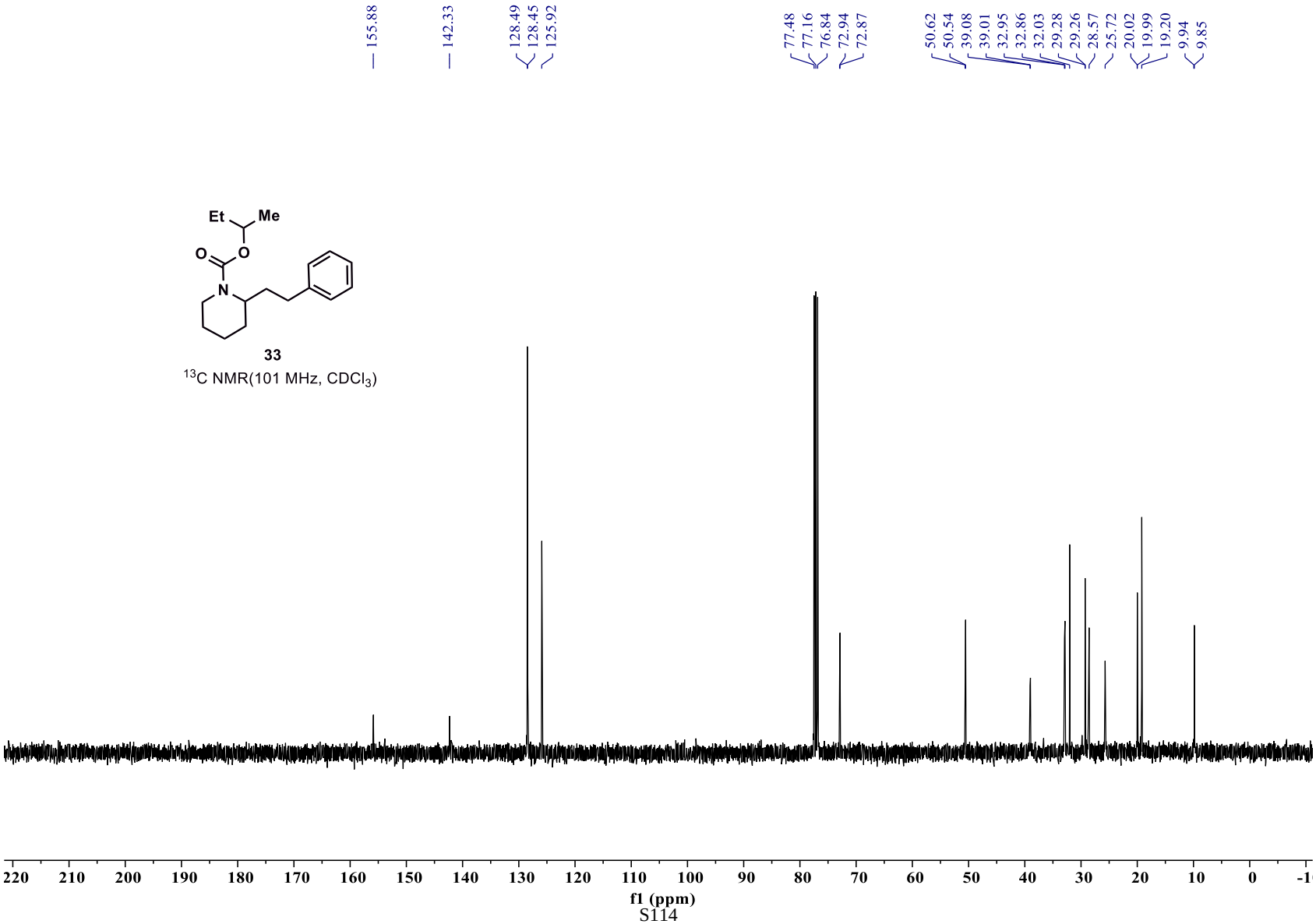

Me
Me
Me
32
1H NMR(400 MHz, CDCl3)
f1 (ppm)

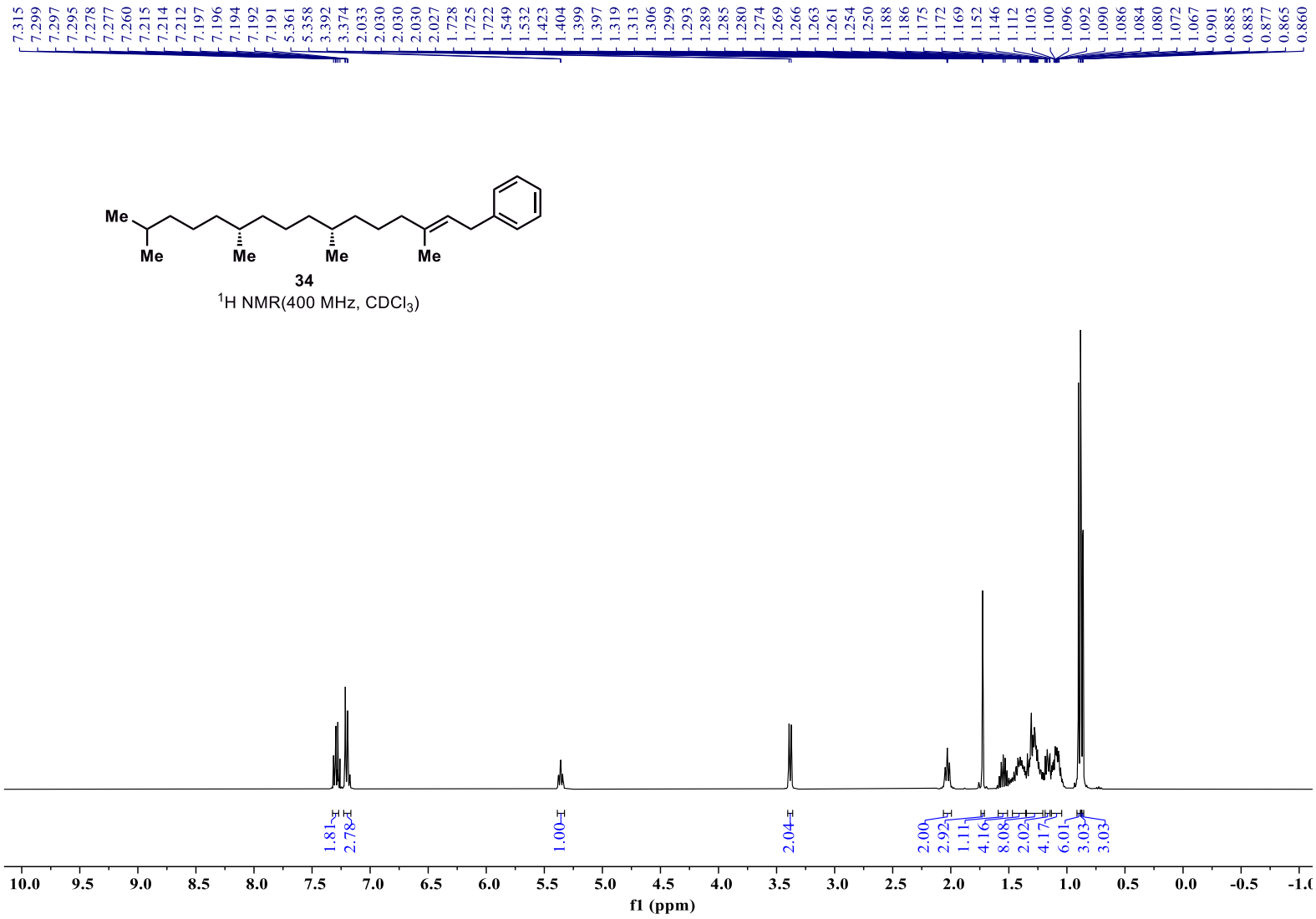
32

^{13}C NMR(101 MHz, $CDCl_3$)



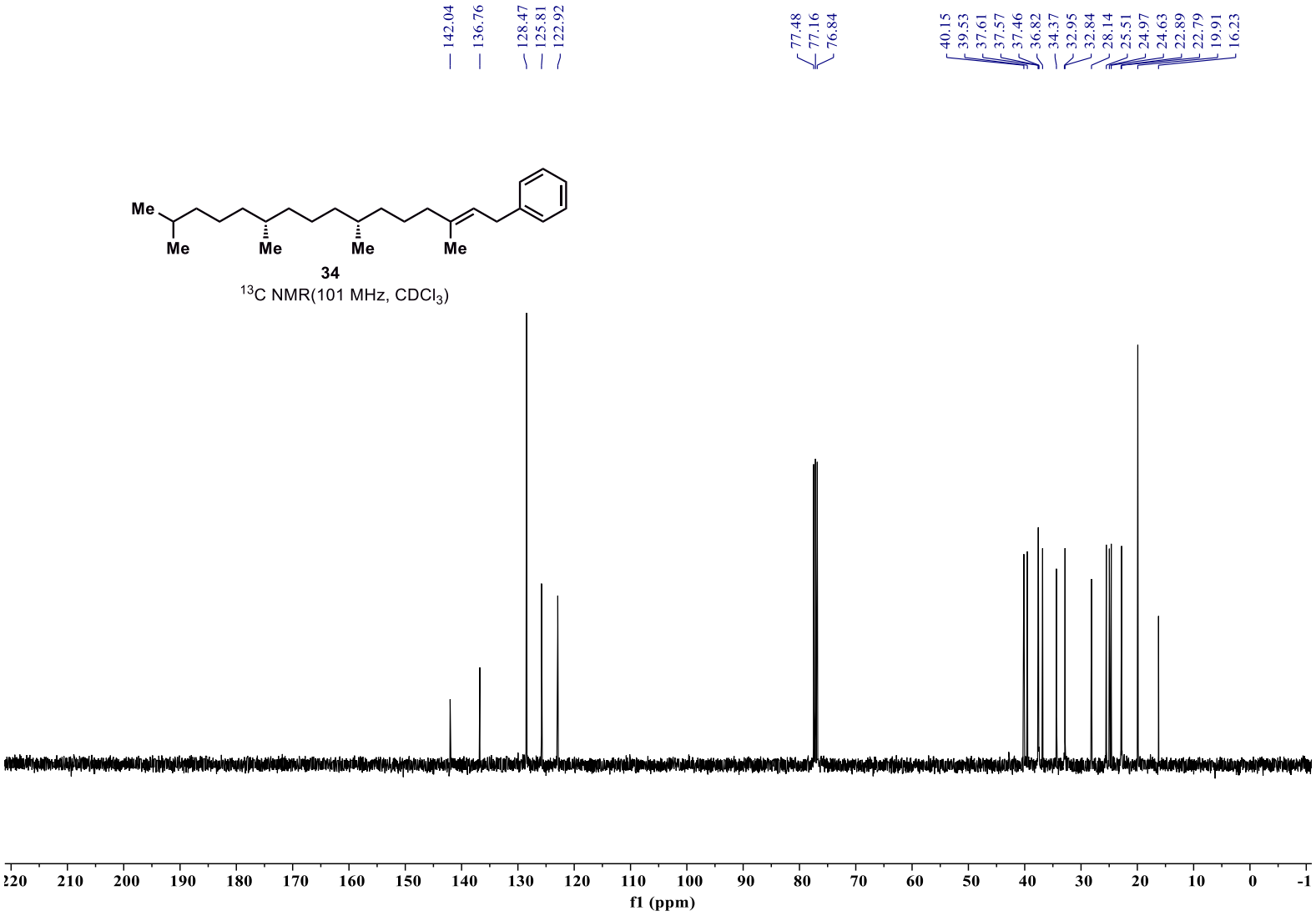

7.300 7.299 7.282 7.280 7.278 7.264 7.262 7.260 7.199 7.182 7.177 4.799 4.796 4.783 4.780 4.768 4.764 4.753 4.749 2.873 2.868 2.841 2.834 2.808 2.801 2.619 2.612 2.605 2.592 2.578 2.567 2.562 2.553 2.547 2.540 2.534 2.526 2.520 2.035 2.034 2.026 2.019 2.012 1.769 1.754 1.742 1.739 1.737 1.735 1.727 1.725 1.720 1.708 1.693 1.628 1.627 1.608 1.607 1.593 1.591 1.583 1.574 1.220 1.218 1.204 1.203 0.933 0.921 0.915 0.902 0.896 0.883
Et
Me
O
O
N
33
1H NMR(400 MHz, CDCl3)
1.72
2.87
1.00
1.00
1.00
1.08
2.09
1.06
1.03
7.10
1.04
3.09
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

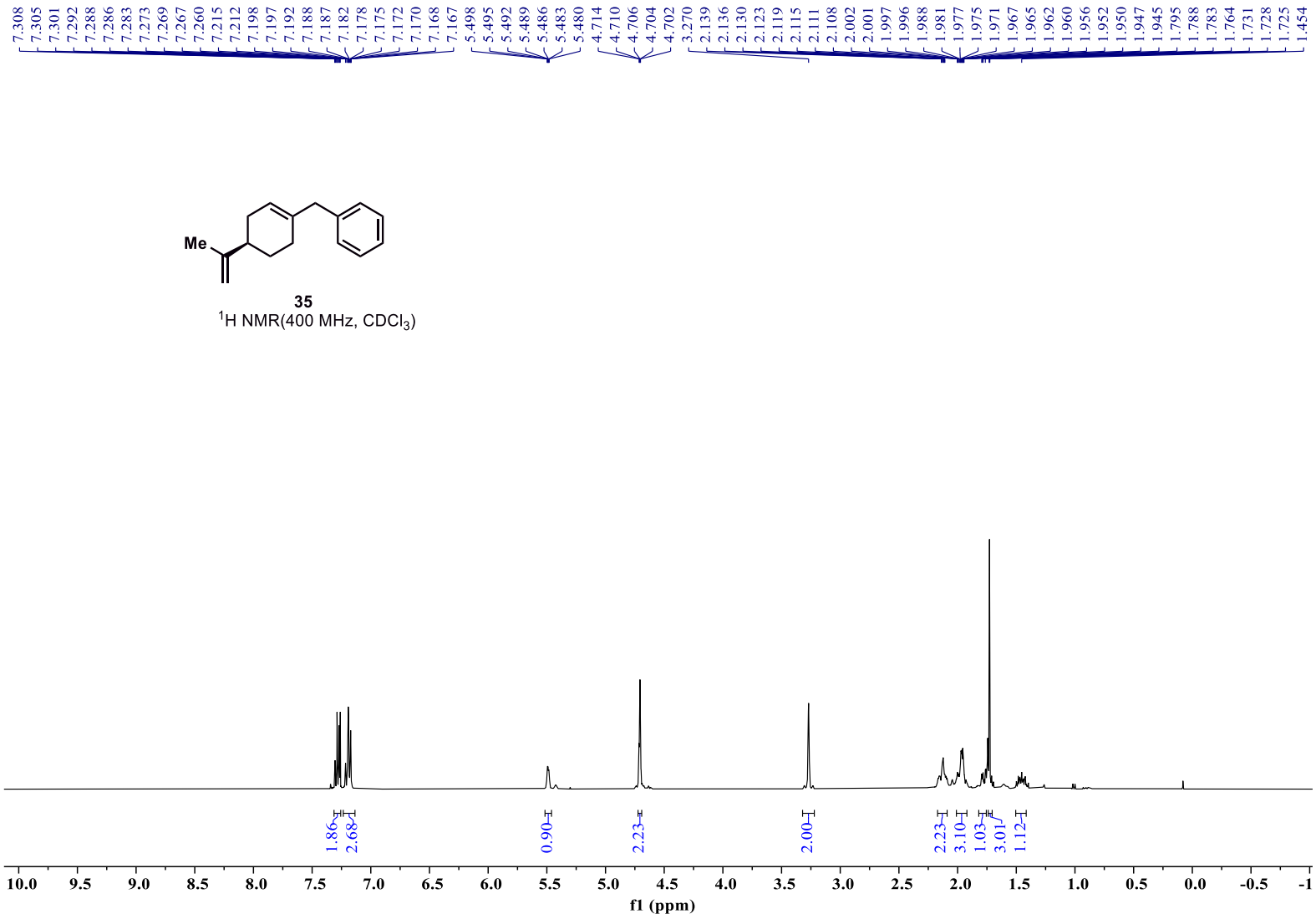

155.88
142.33
128.49
128.45
125.92
77.48
77.16
76.84
72.94
72.87
50.62
50.54
39.08
39.01
32.95
32.86
32.03
29.28
29.26
28.57
25.72
20.02
19.99
19.20
9.94
9.85
Et
Me
O
O
N
33
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.315 7.299 7.297 7.295 7.278 7.277 7.260 7.215 7.214 7.212 7.197 7.196 7.194 7.192 7.191 5.361 5.358 3.392 3.374 2.033 2.030 2.030 2.030 2.027 1.728 1.725 1.722 1.549 1.532 1.423 1.404 1.399 1.397 1.319 1.313 1.306 1.299 1.293 1.289 1.285 1.280 1.274 1.269 1.266 1.263 1.261 1.254 1.250 1.188 1.186 1.175 1.172 1.169 1.152 1.146 1.112 1.103 1.100 1.096 1.092 1.090 1.086 1.084 1.080 1.072 1.067 0.901 0.885 0.883 0.877 0.865 0.860
Me
Me
Me
Me
Me
34
1H NMR(400 MHz, CDCl3)
1.81
2.78
1.00
2.04
2.00
2.92
1.11
4.16
8.08
2.02
4.17
6.01
3.03
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

34

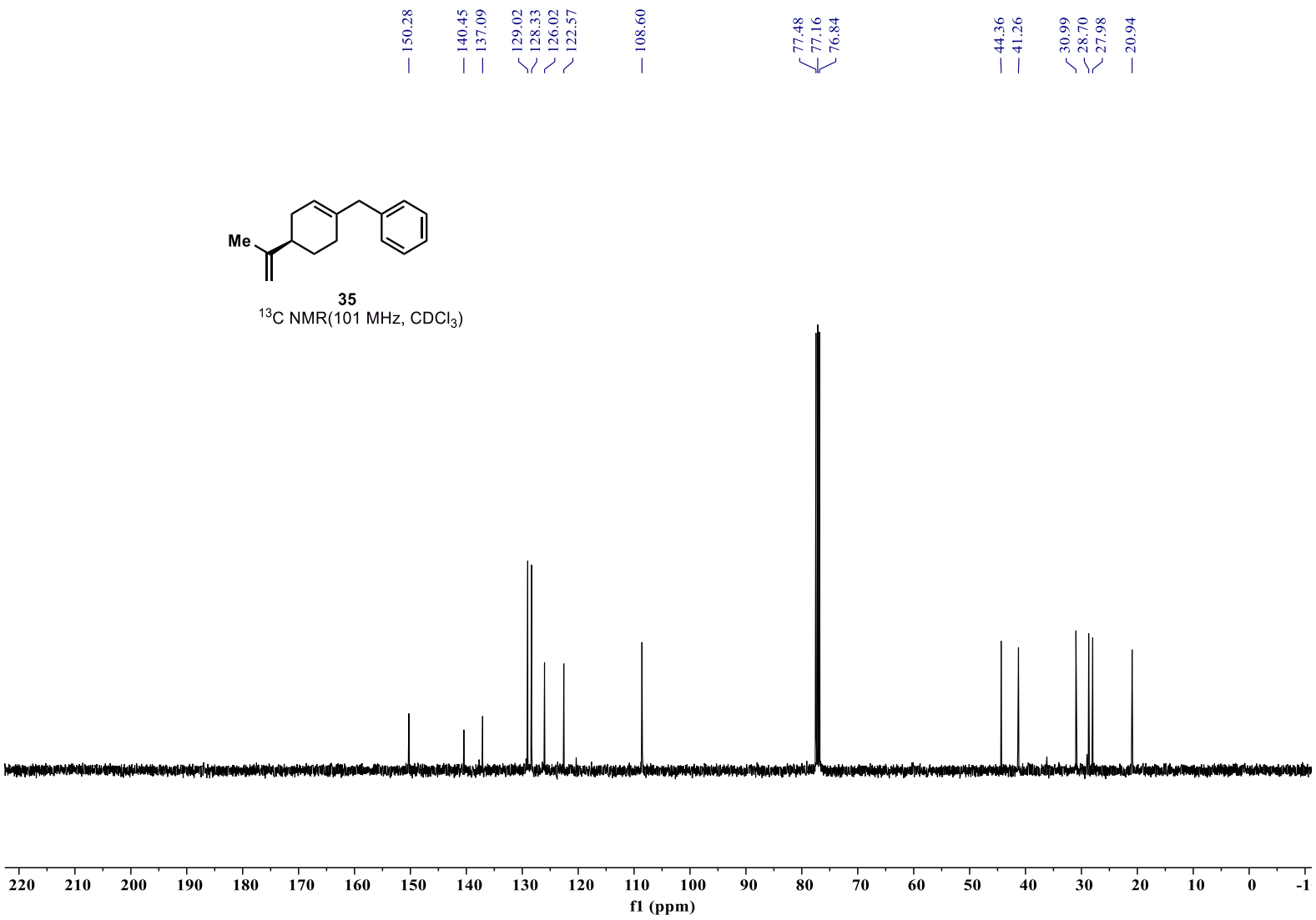
^{13}C NMR(101 MHz, $CDCl_3$)

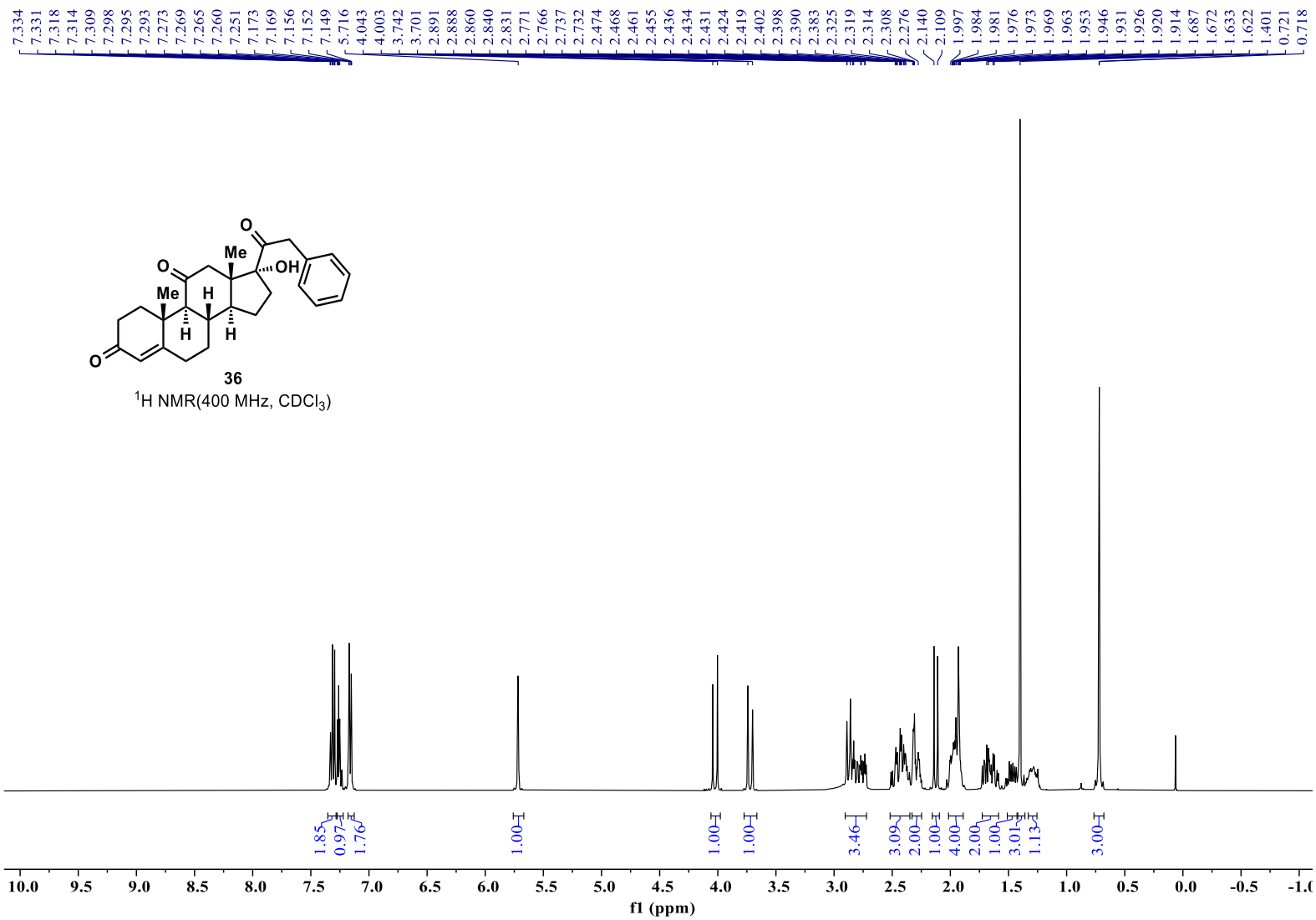


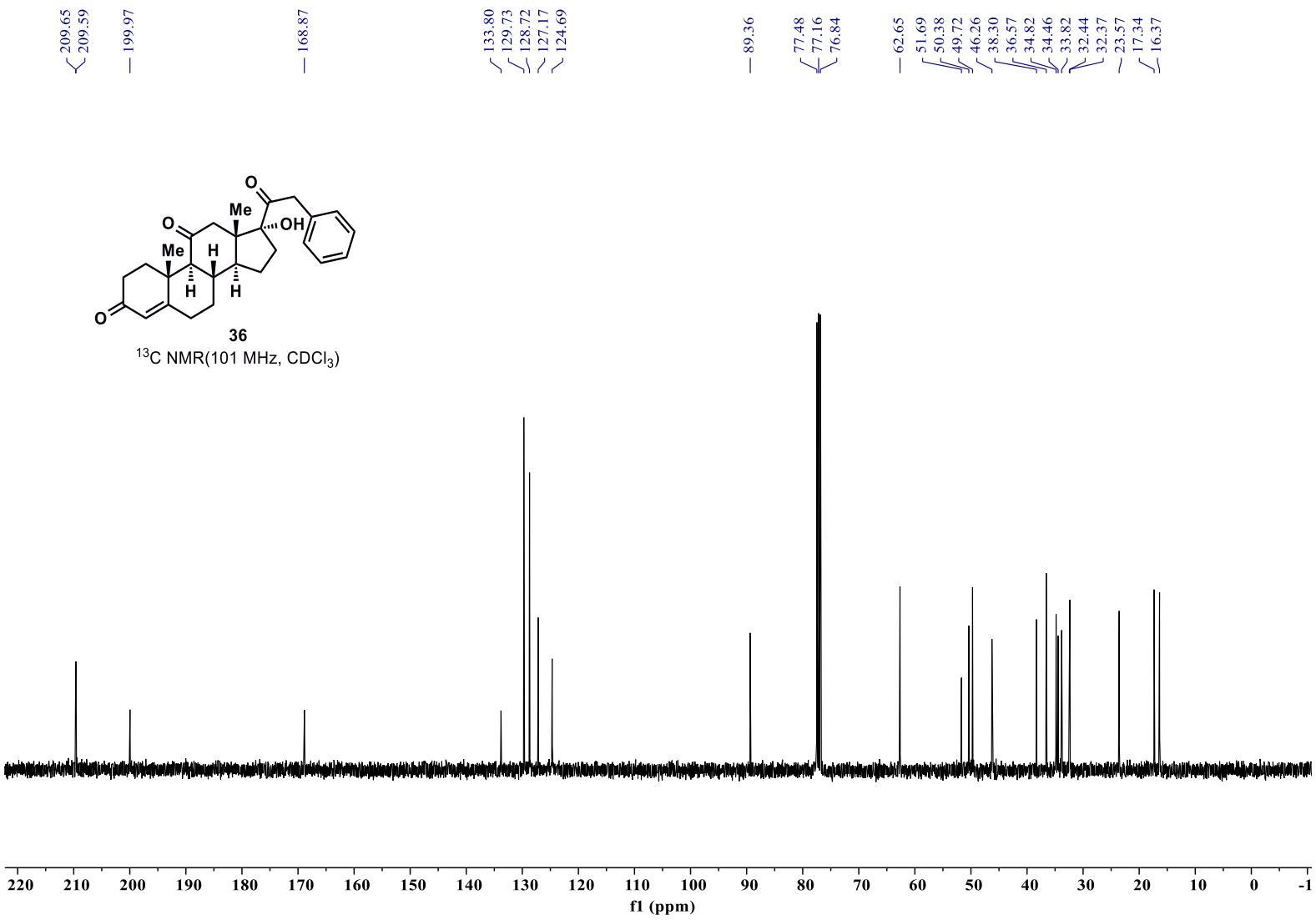


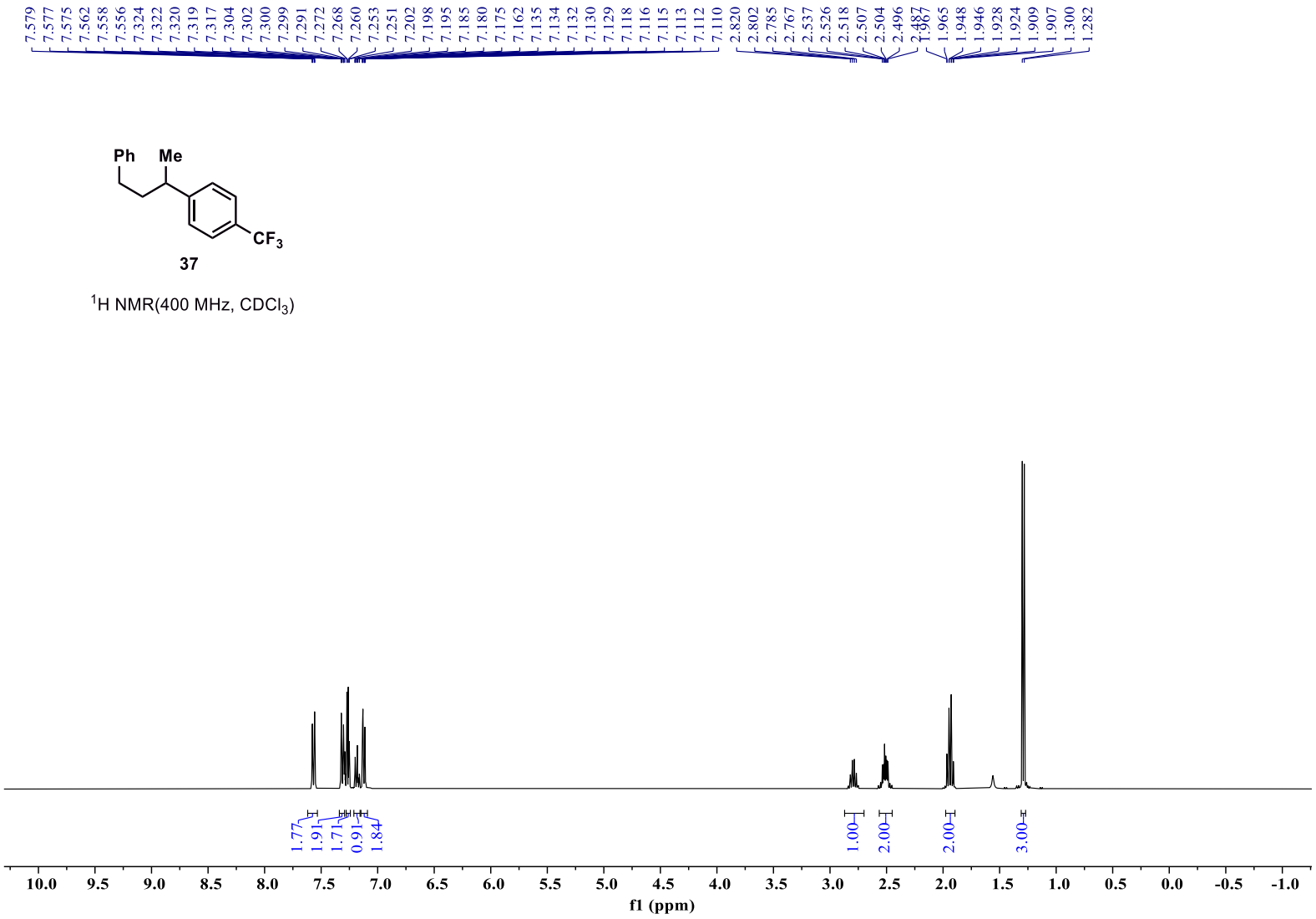
35

^{1}H NMR(400 MHz, $CDCl_3$)


150.28
140.45
137.09
129.02
128.33
126.02
122.57
108.60
77.48
77.16
76.84
44.36
41.26
30.99
28.70
27.98
20.94
Me
35
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


7.334 7.331 7.318 7.314 7.309 7.298 7.295 7.293 7.273 7.269 7.265 7.260 7.251 7.173 7.169 7.156 7.152 7.149 5.716 4.043 4.003 3.742 3.701 2.891 2.888 2.860 2.840 2.831 2.771 2.766 2.737 2.732 2.474 2.468 2.461 2.455 2.436 2.434 2.431 2.424 2.419 2.402 2.398 2.390 2.383 2.325 2.319 2.314 2.308 2.276 2.140 2.109 1.997 1.984 1.981 1.976 1.973 1.969 1.963 1.953 1.946 1.931 1.926 1.920 1.914 1.687 1.672 1.633 1.622 1.401 0.721 0.718
O
Me
OH
O
Me
H
H
H
H
O
36
1H NMR(400 MHz, CDCl3)
1.85
0.97
1.76
1.00
1.00
1.00
3.46
3.09
2.00
1.00
4.00
2.00
1.00
3.01
1.13
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

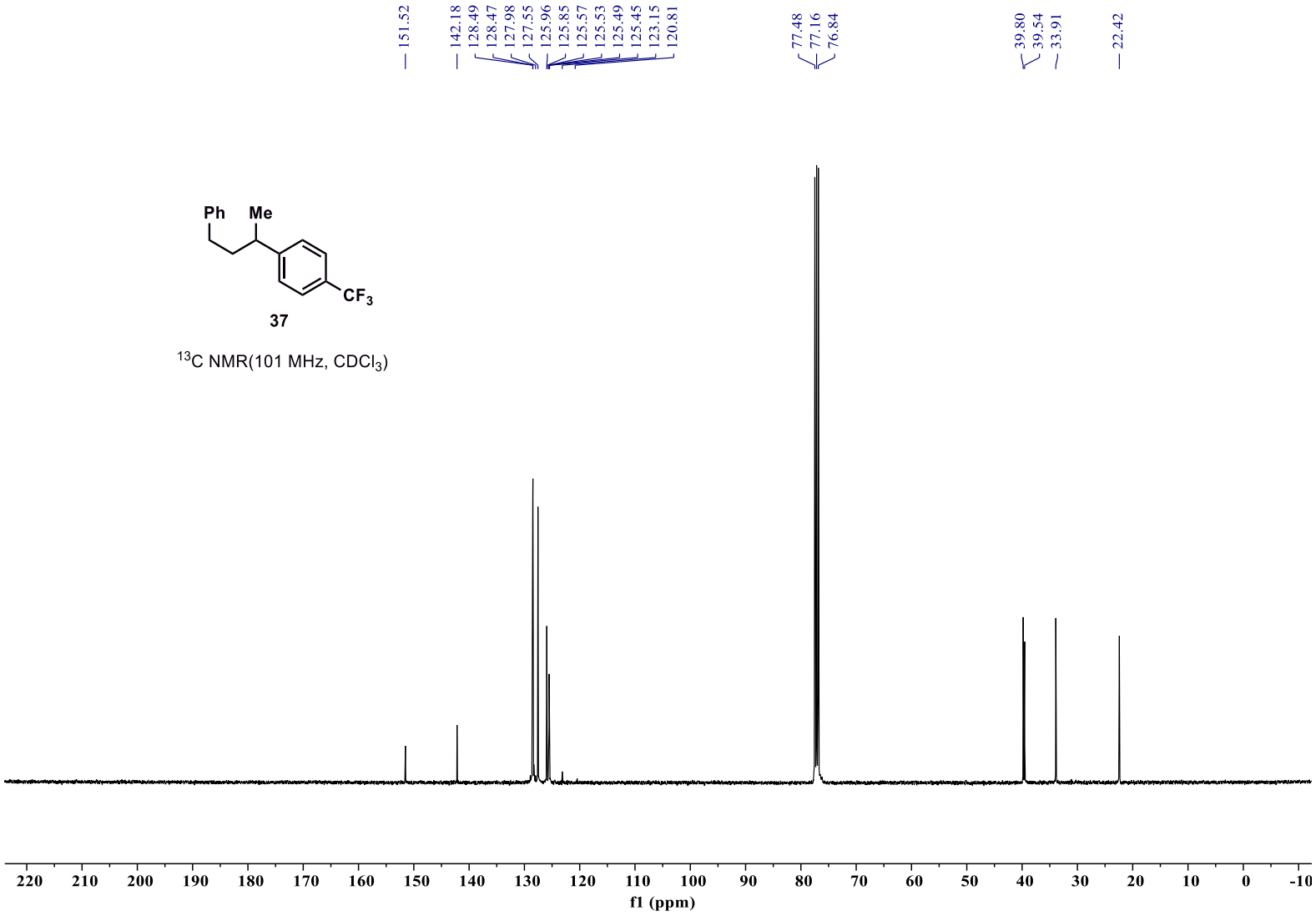

209.65
209.59
199.97
168.87
133.80
129.73
128.72
127.17
124.69
89.36
77.48
77.16
76.84
62.65
51.69
50.38
49.72
46.26
38.30
36.57
34.82
34.46
33.82
32.44
32.37
23.57
17.34
16.37
O
Me
OH
O
Me
H
H
H
O
36
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



^{1}H NMR(400 MHz, $CDCl_3$)

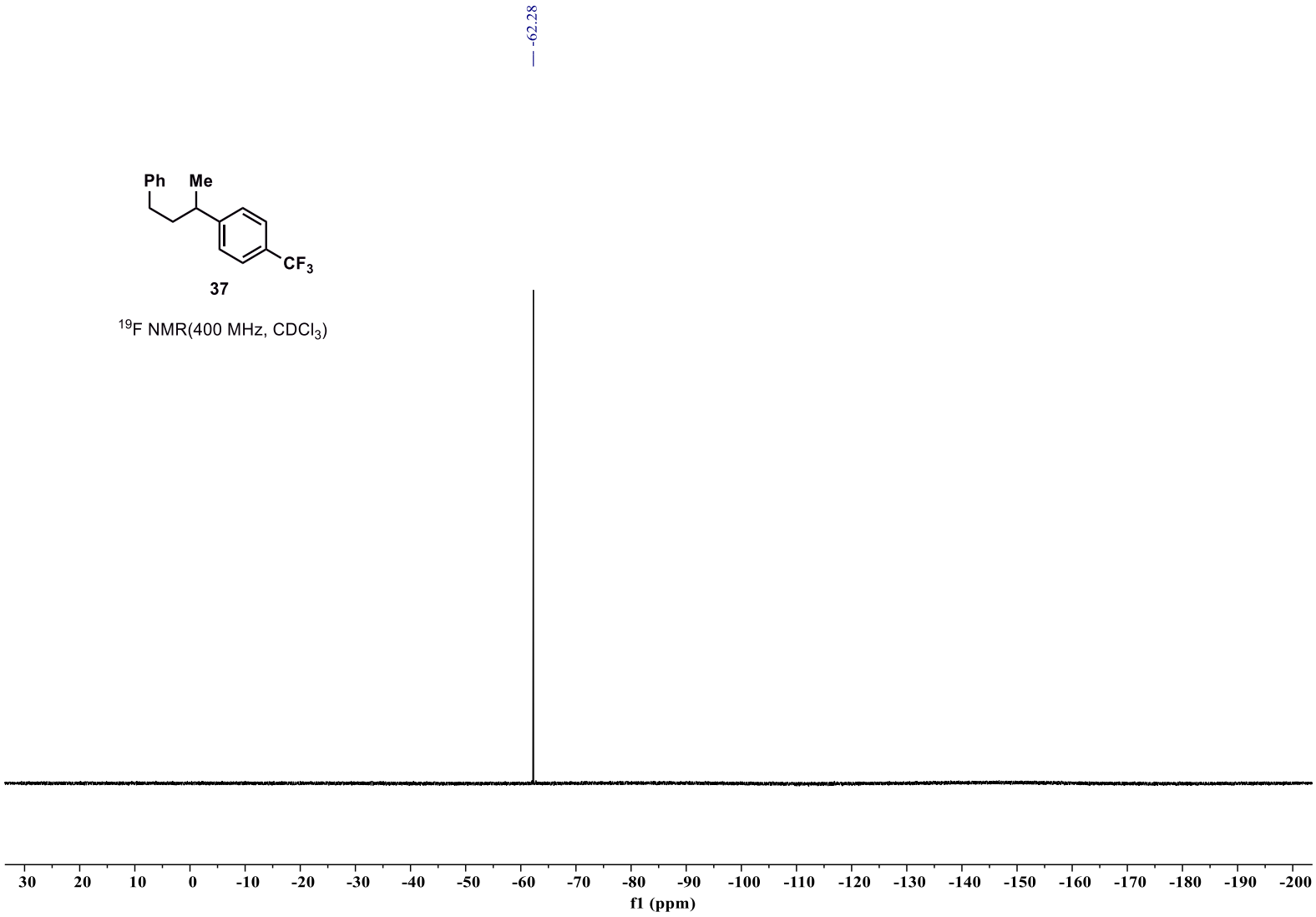
37

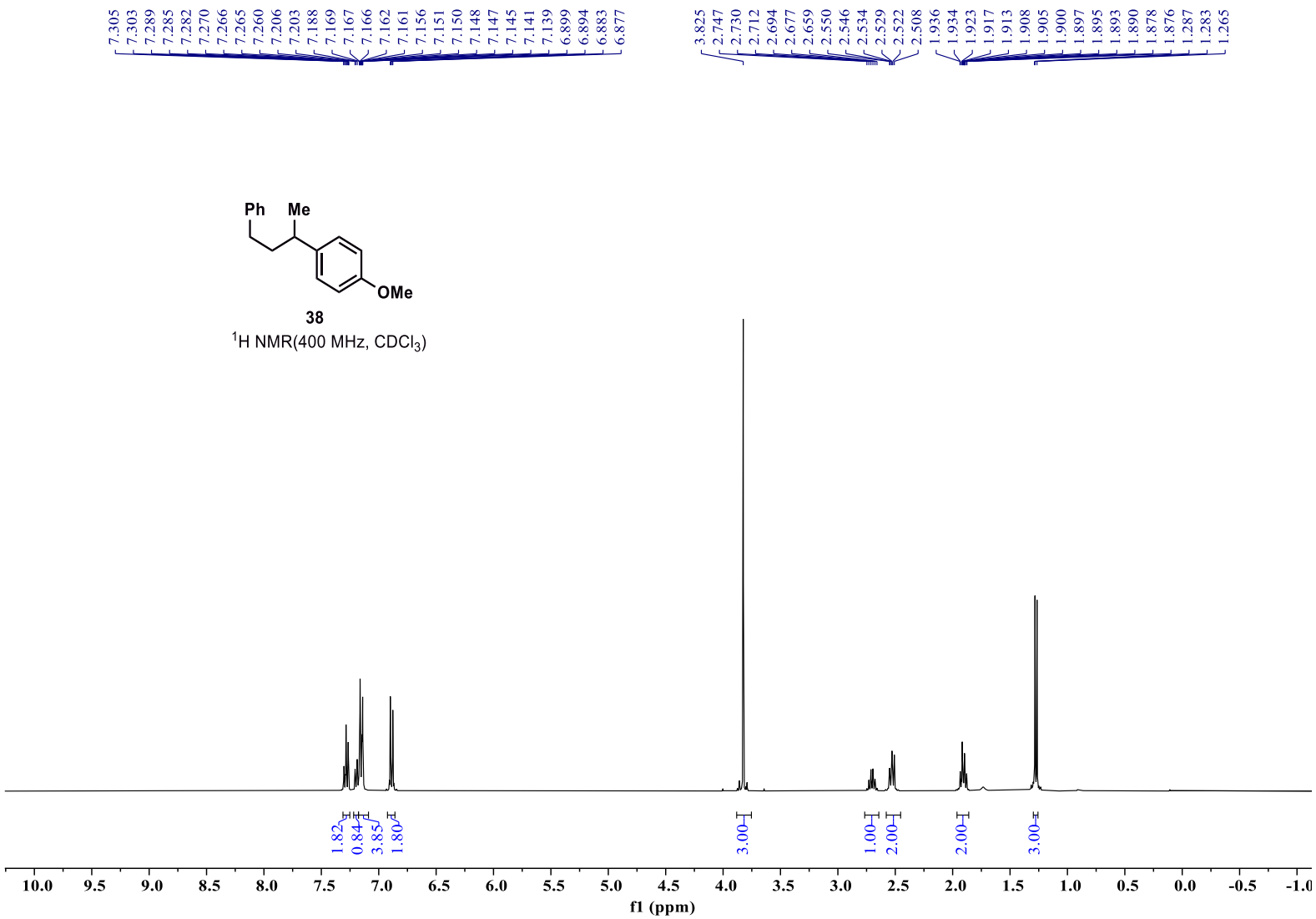
^{13}C NMR(101 MHz, $CDCl_3$)



37

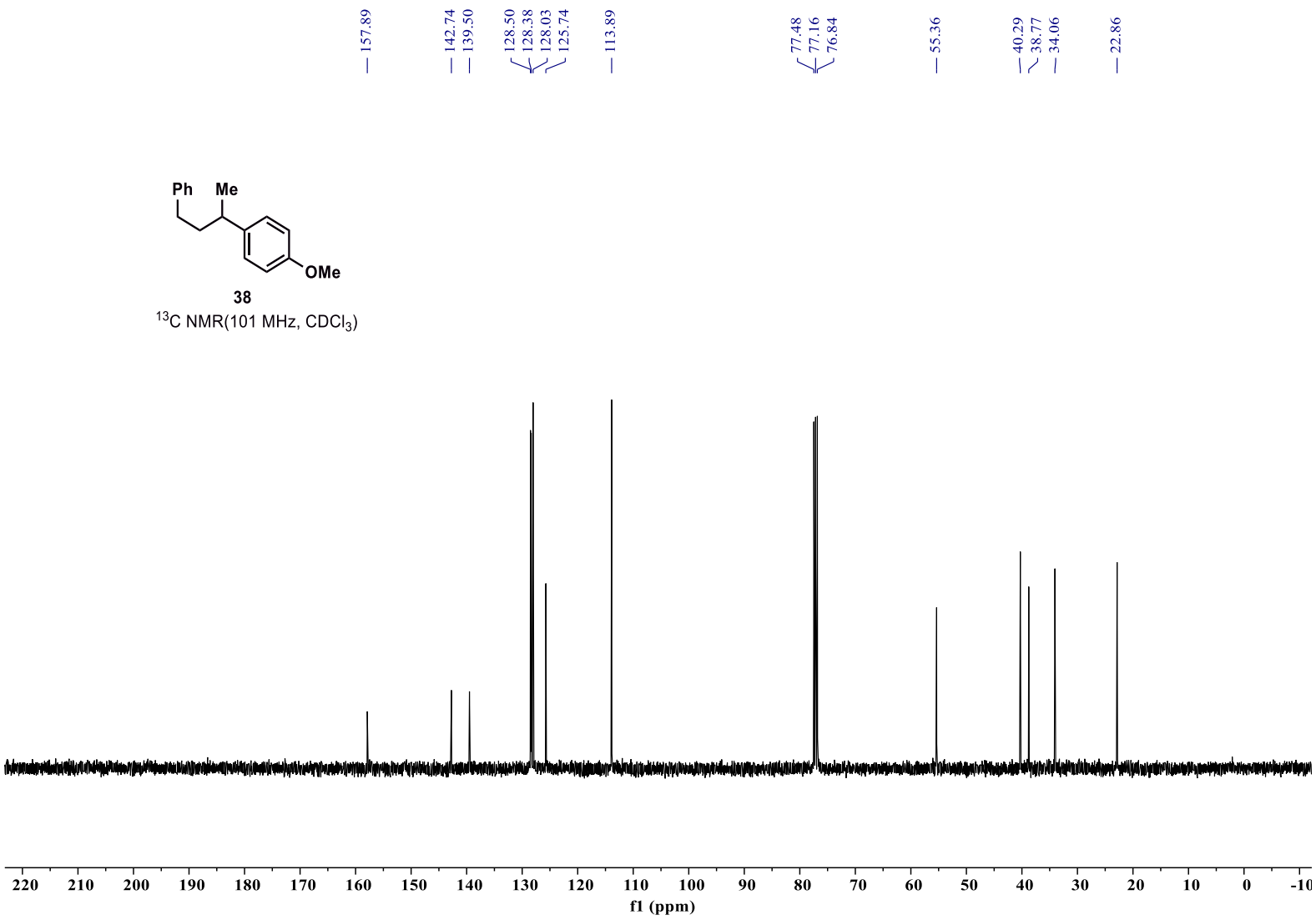
^{19}F NMR(400 MHz, $CDCl_3$)

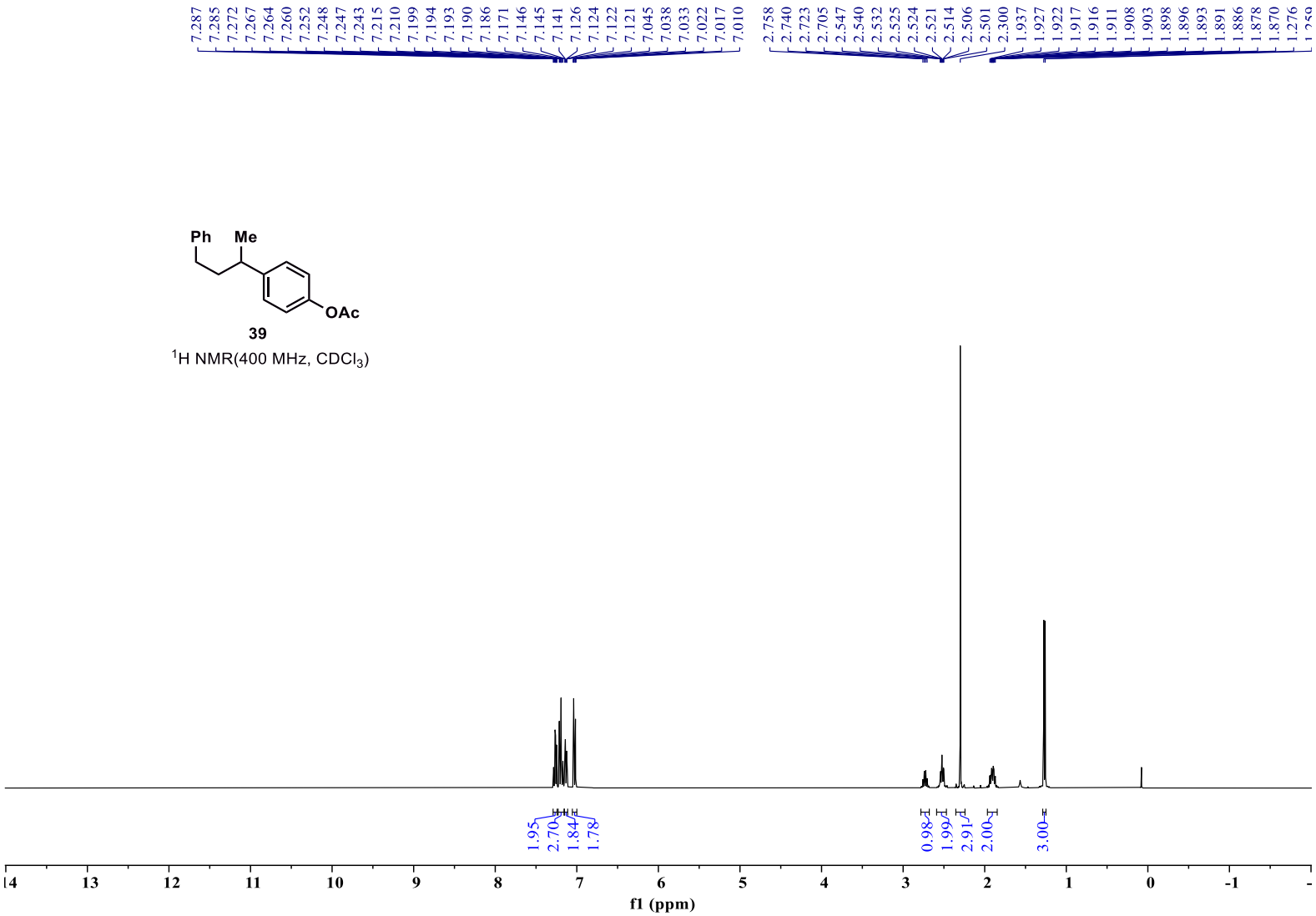


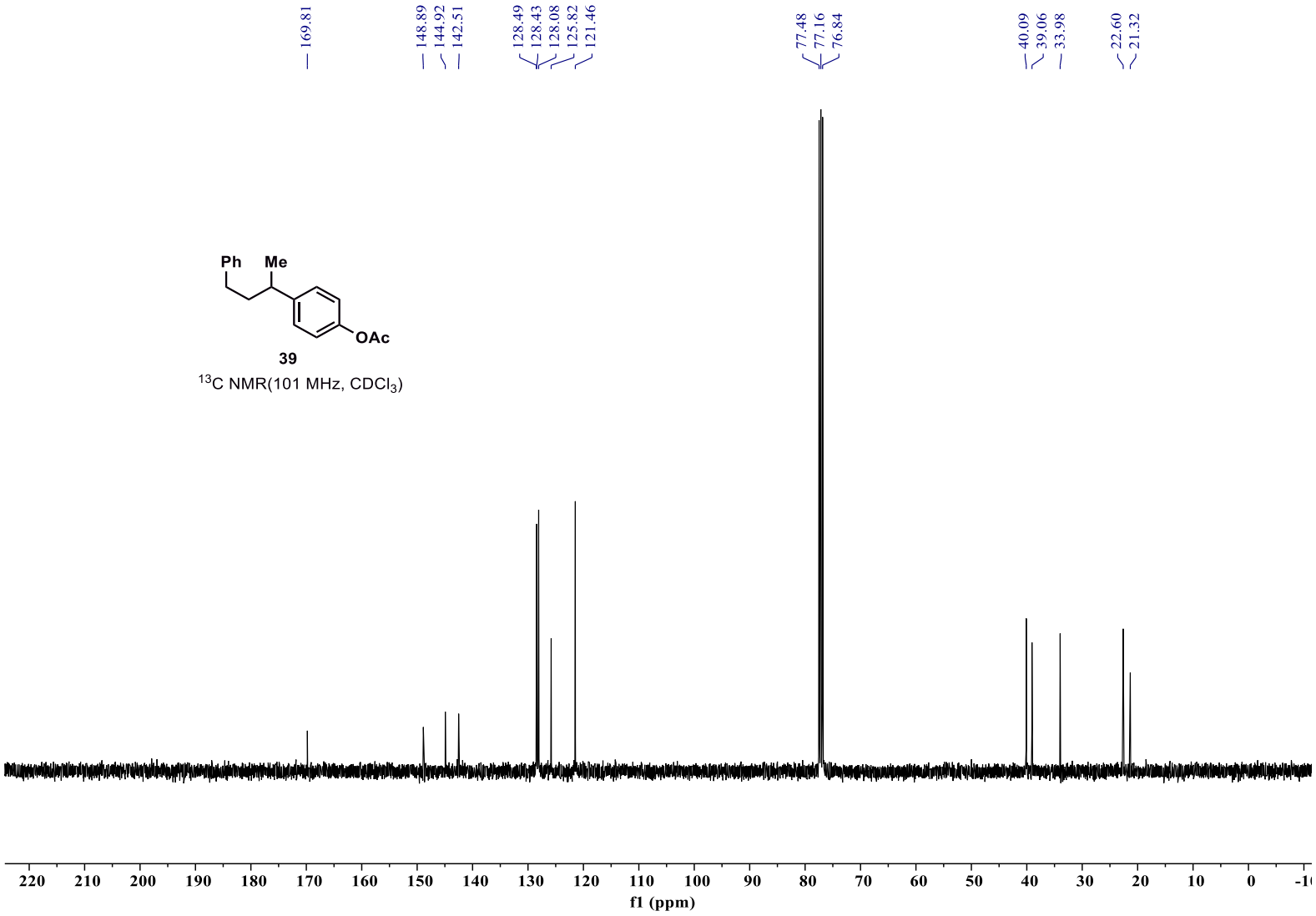

7.305
7.303
7.289
7.285
7.282
7.270
7.266
7.265
7.260
7.206
7.203
7.188
7.169
7.167
7.166
7.162
7.161
7.156
7.151
7.150
7.148
7.147
7.145
7.141
7.139
6.899
6.894
6.883
6.877
3.825
2.747
2.730
2.712
2.694
2.677
2.659
2.550
2.546
2.534
2.529
2.522
2.508
1.936
1.934
1.923
1.917
1.913
1.908
1.905
1.900
1.897
1.895
1.893
1.890
1.878
1.876
1.287
1.283
1.265
Ph
Me
OMe
38
1H NMR(400 MHz, CDCl3)
1.82
0.84
3.85
1.80
3.00
1.00
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

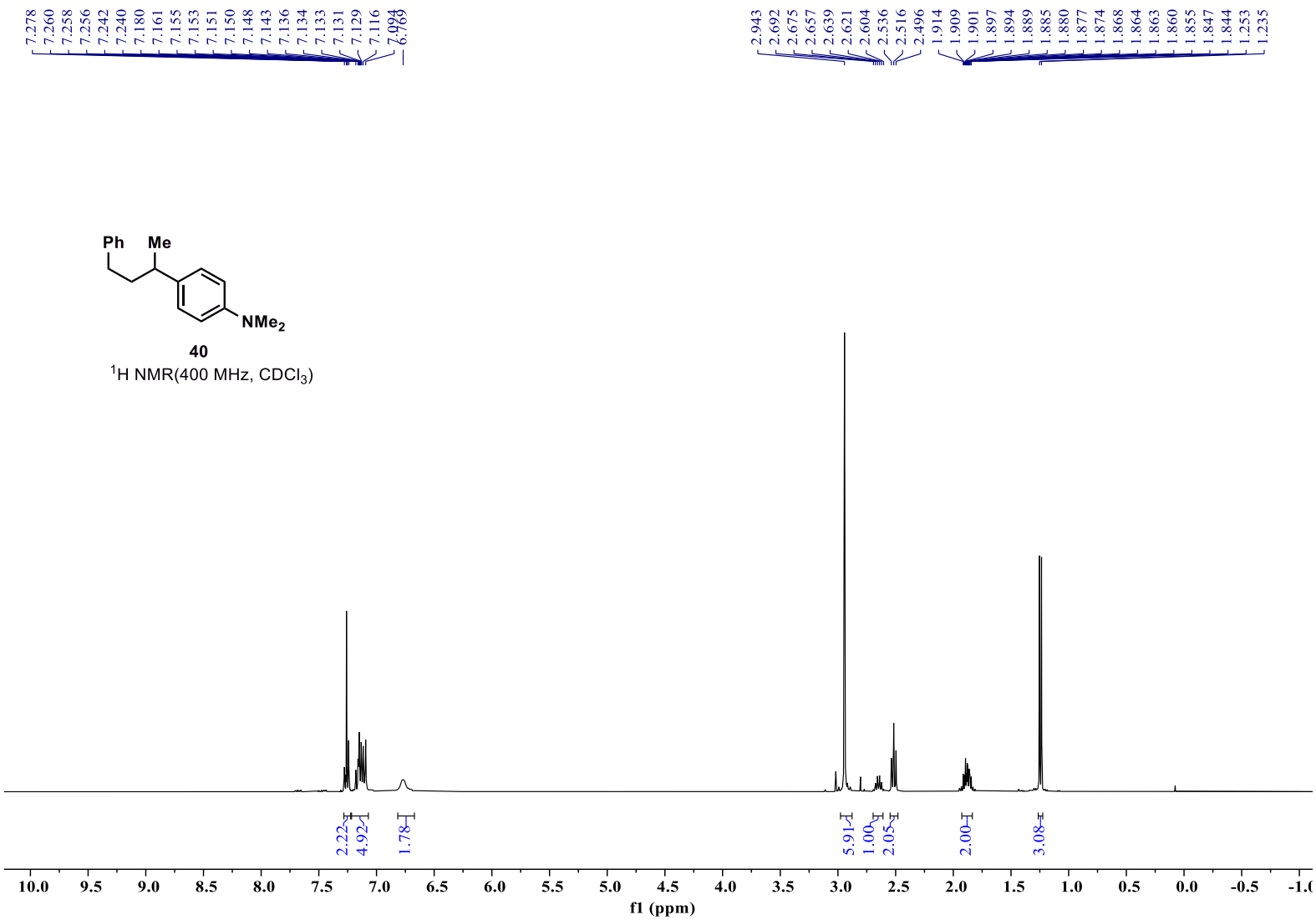
38

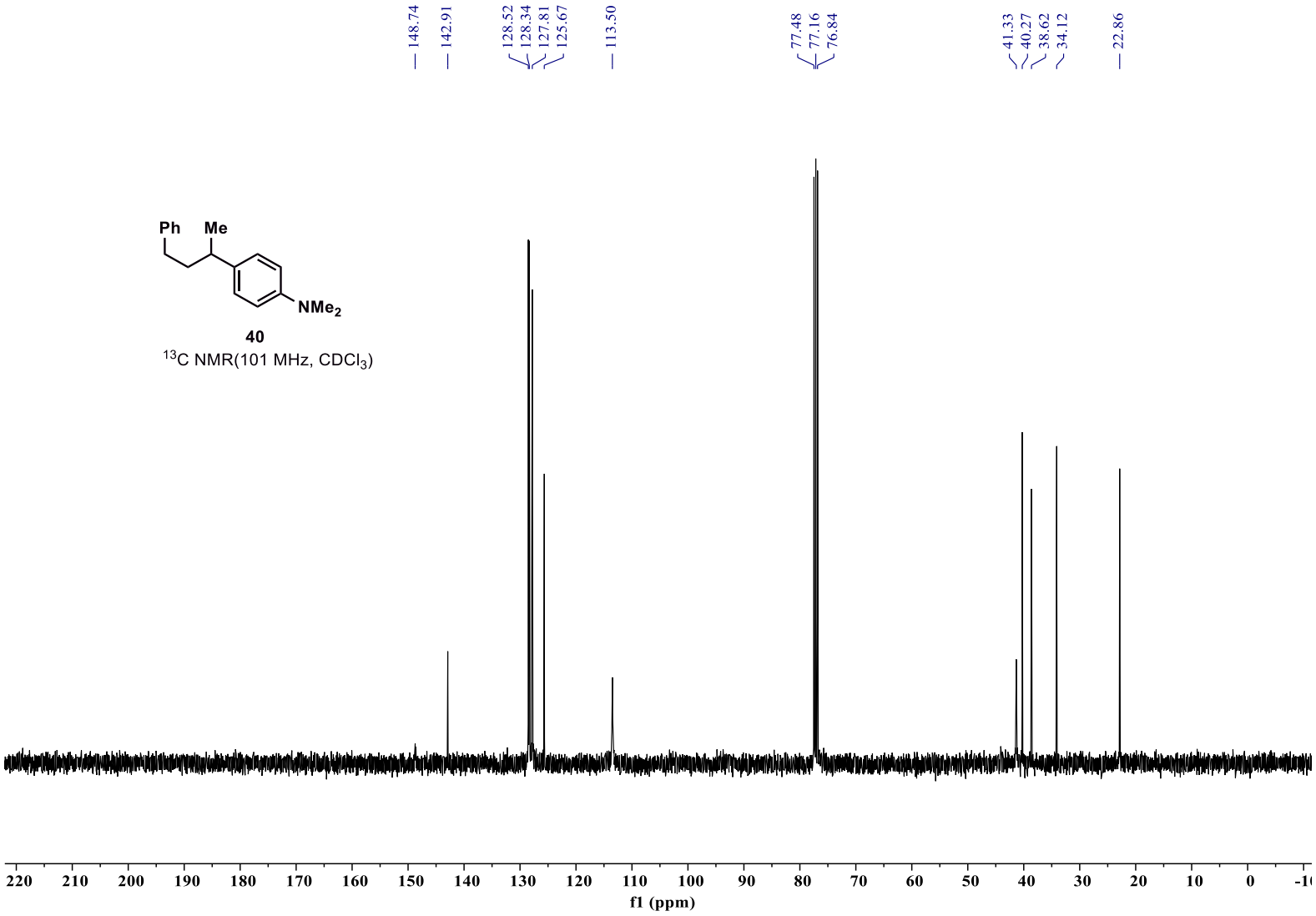
^{13}C NMR(101 MHz, $CDCl_3$)

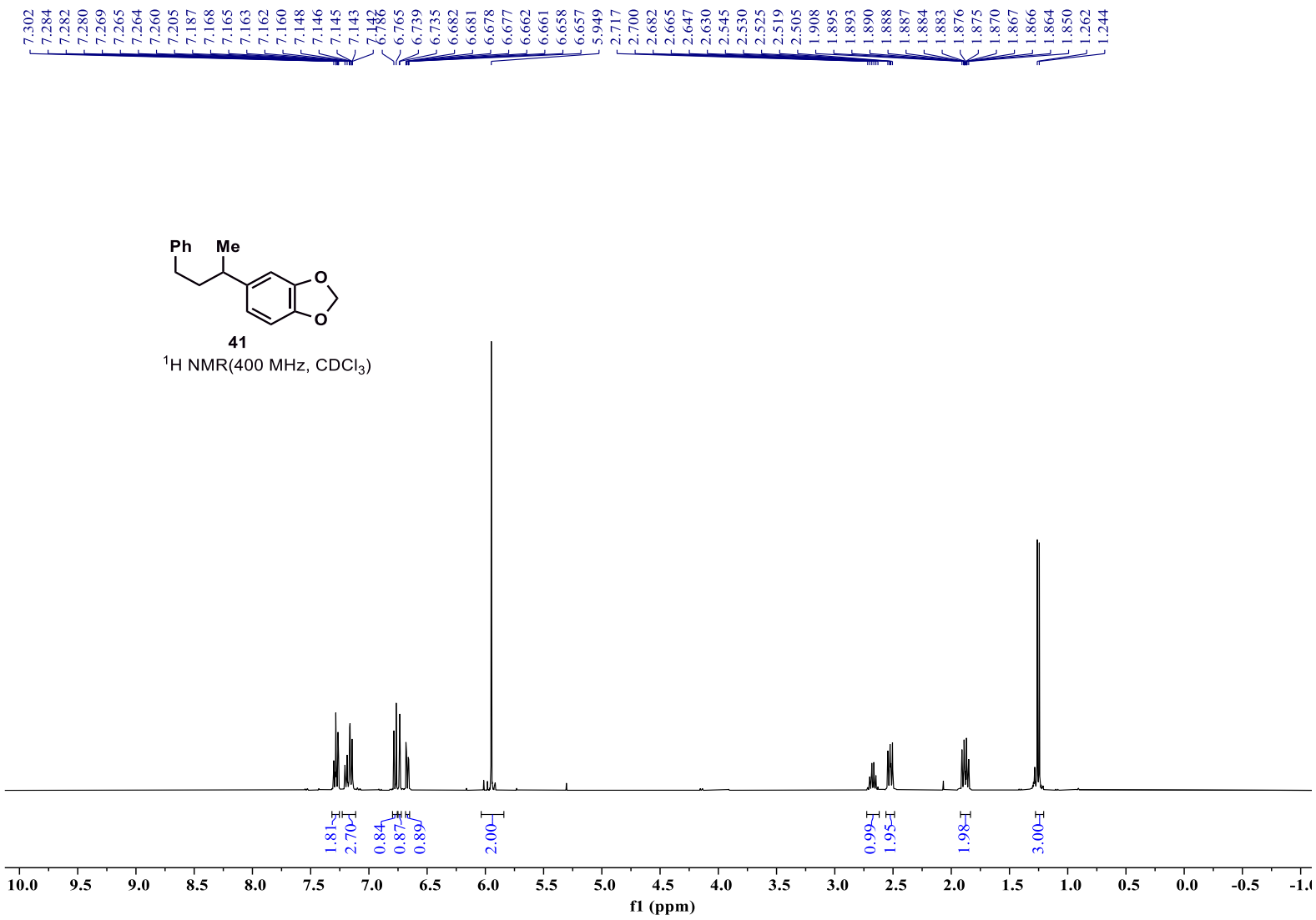


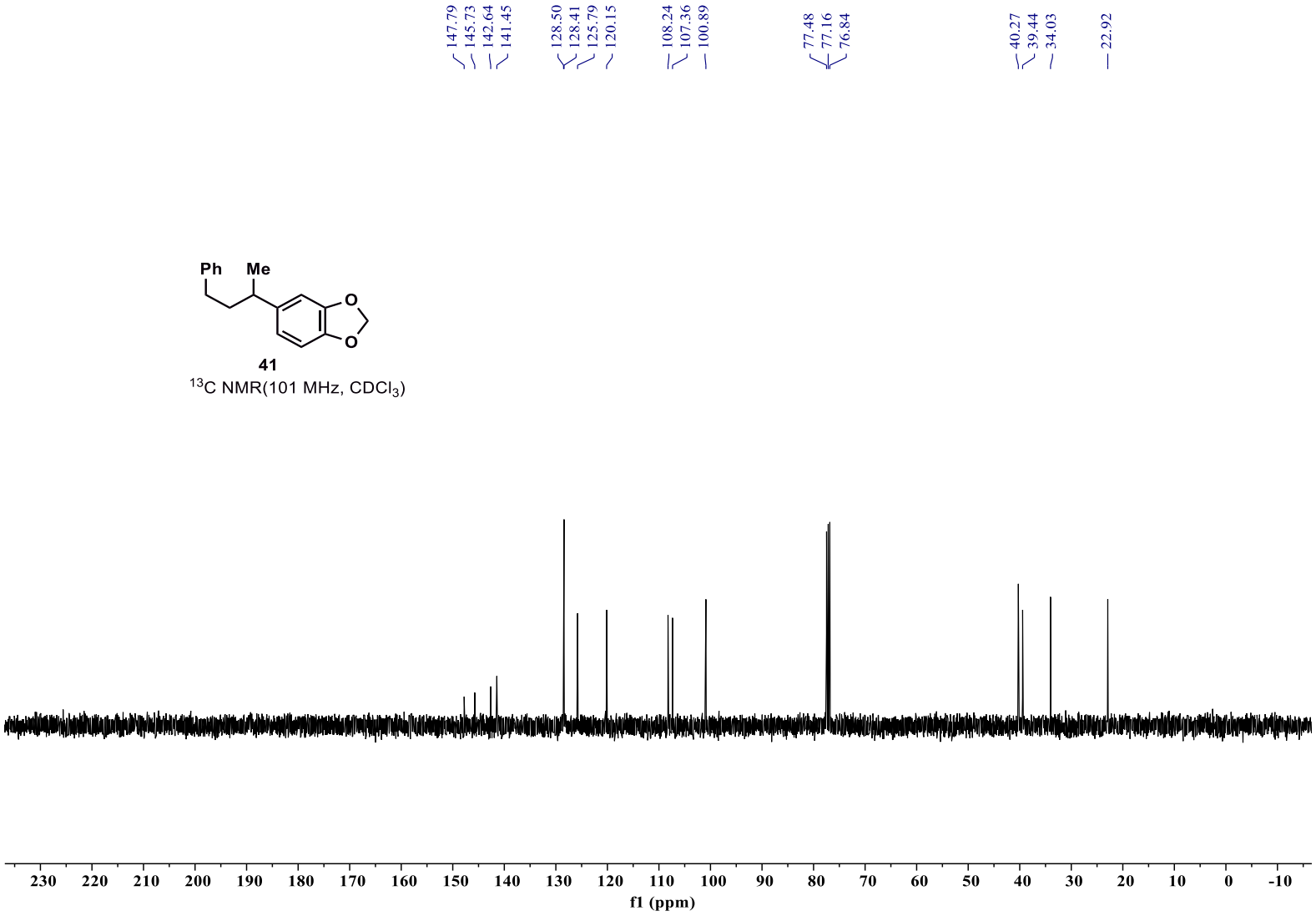

7.287
7.285
7.272
7.267
7.264
7.260
7.252
7.248
7.247
7.243
7.215
7.210
7.199
7.194
7.193
7.190
7.186
7.171
7.146
7.145
7.141
7.126
7.124
7.122
7.121
7.045
7.038
7.033
7.022
7.017
7.010
2.758
2.740
2.723
2.705
2.547
2.540
2.532
2.525
2.524
2.521
2.514
2.506
2.501
2.300
1.937
1.927
1.922
1.917
1.916
1.911
1.908
1.903
1.898
1.896
1.893
1.891
1.886
1.878
1.870
1.276
Ph
Me
OAc
39
1H NMR(400 MHz, CDCl3)
1.95
2.70
1.84
1.78
0.98
1.99
2.91
2.00
3.00
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

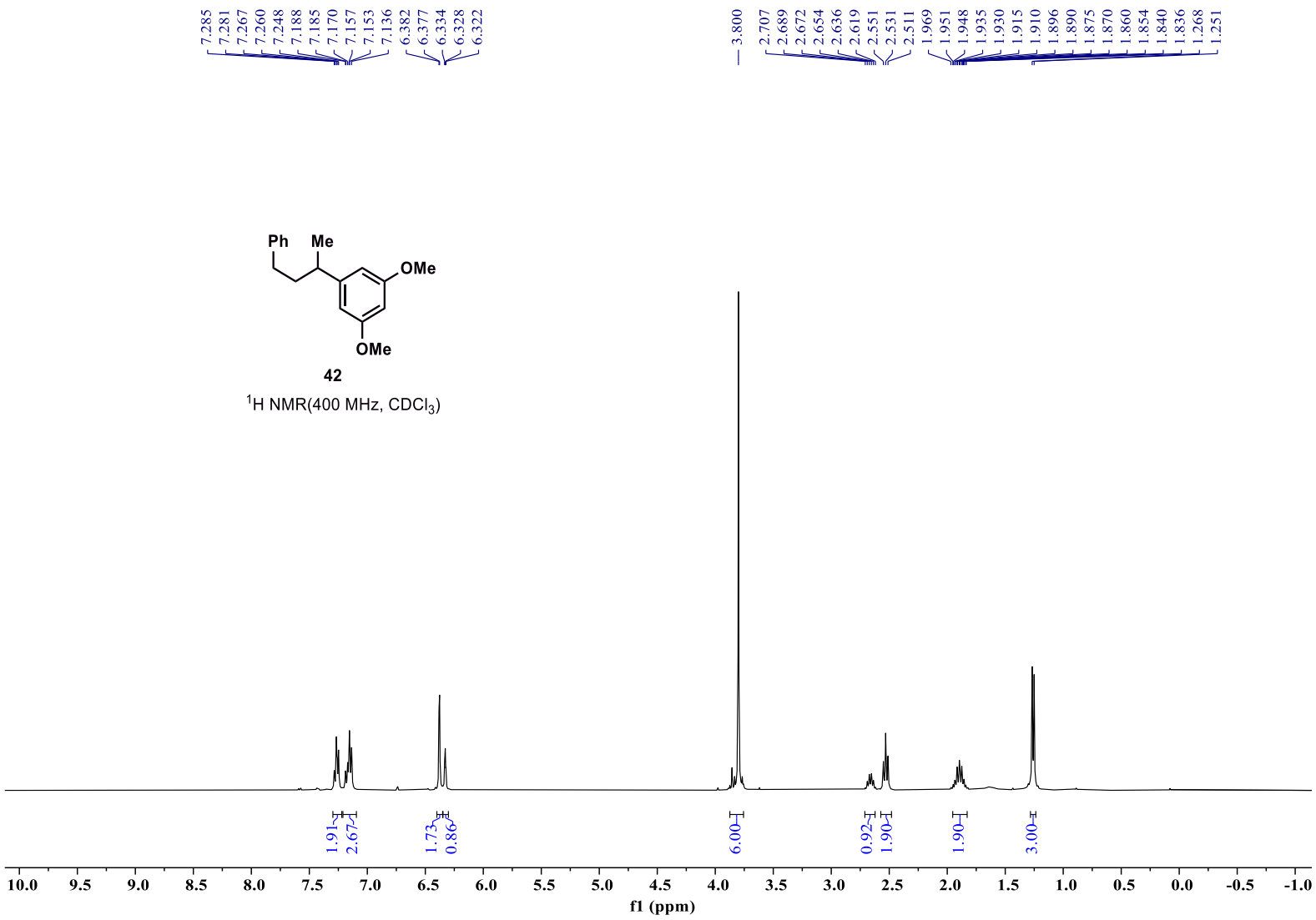

169.81
148.89
144.92
142.51
128.49
128.43
128.08
125.82
121.46
77.48
77.16
76.84
40.09
39.06
33.98
22.60
21.32
Ph
Me
OAc
39
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

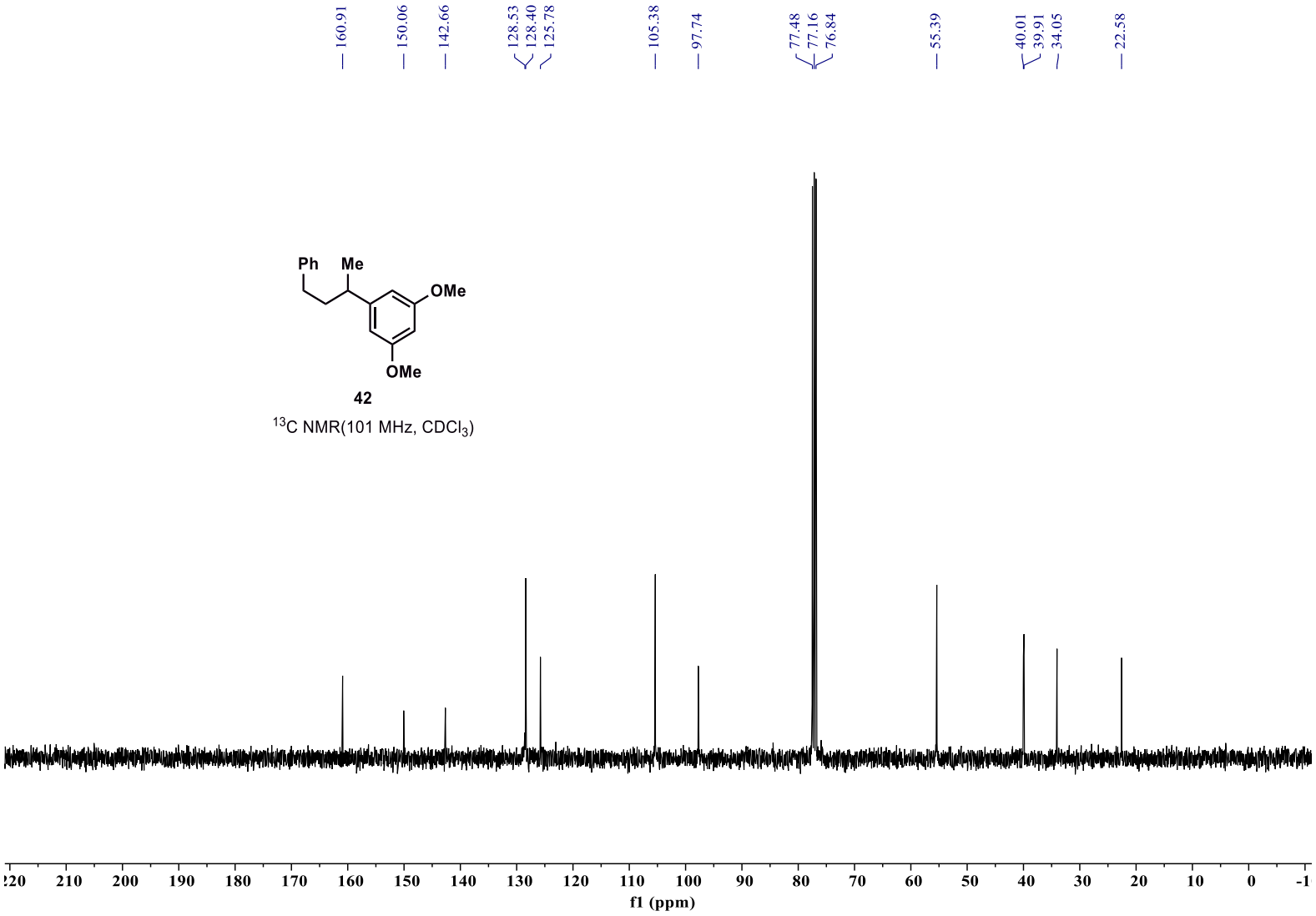

7.278
7.260
7.258
7.256
7.242
7.240
7.180
7.161
7.155
7.153
7.151
7.150
7.148
7.143
7.136
7.134
7.133
7.131
7.129
7.116
7.094
6.763
2.943
2.692
2.675
2.657
2.639
2.621
2.604
2.536
2.516
2.496
1.914
1.909
1.901
1.897
1.894
1.889
1.885
1.880
1.877
1.874
1.868
1.864
1.863
1.860
1.855
1.847
1.844
1.253
1.235
Ph
Me
NMe2
40
1H NMR(400 MHz, CDCl3)
2.22
4.92
1.78
5.91
1.00
2.05
2.00
3.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

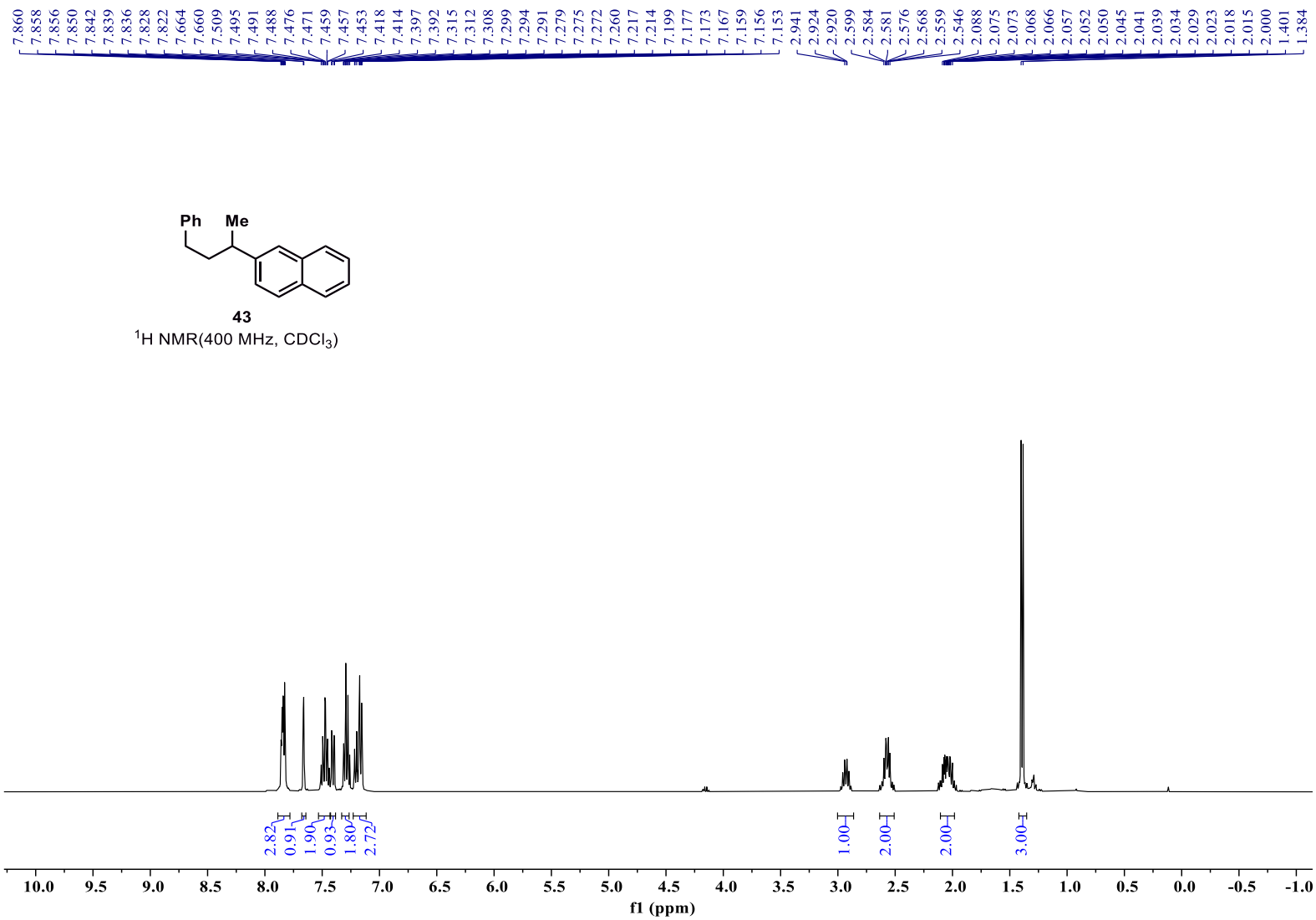

148.74
142.91
128.52
128.34
127.81
125.67
113.50
77.48
77.16
76.84
41.33
40.27
38.62
34.12
22.86
Ph
Me
NMe2
40
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

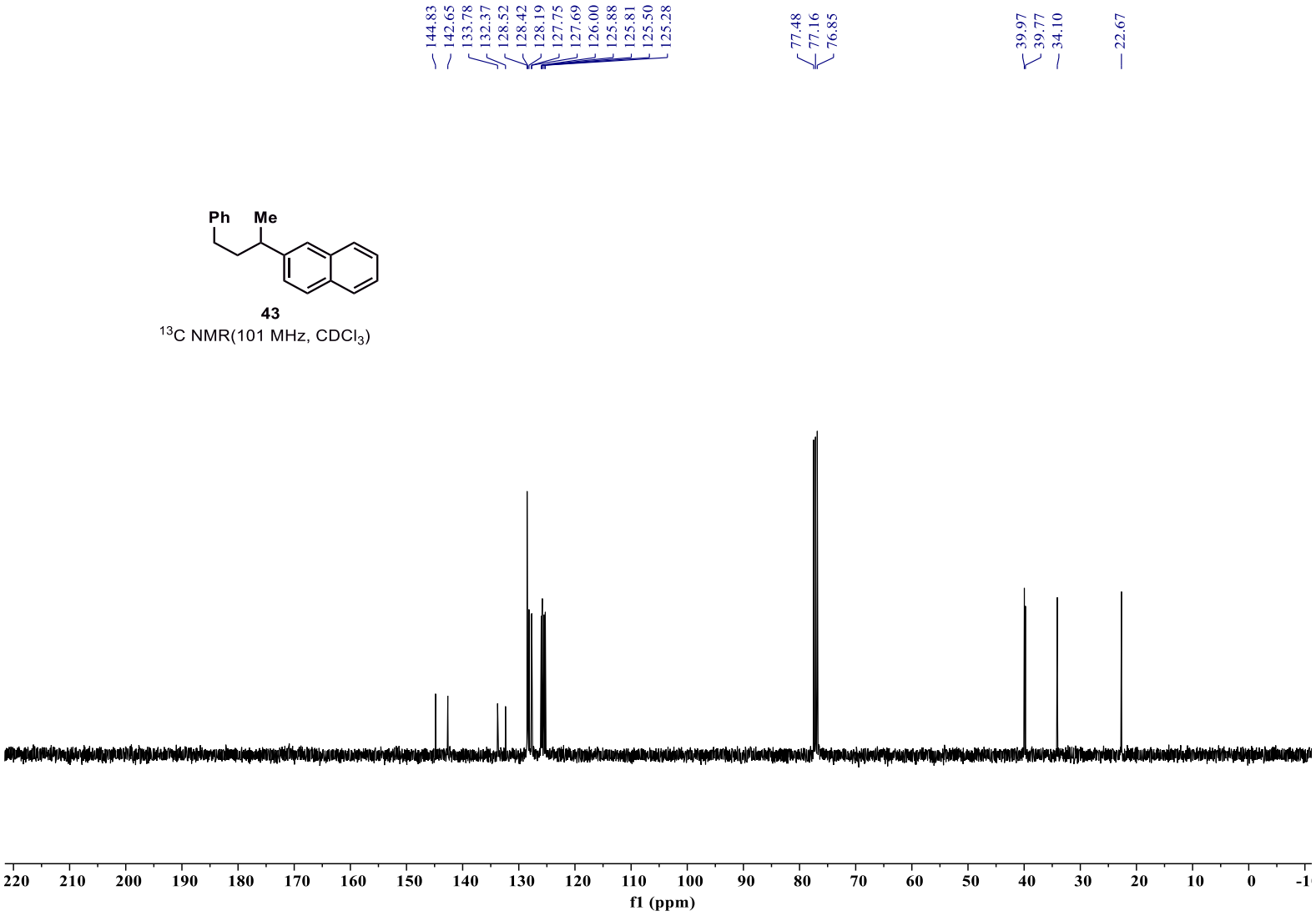

7.302
7.284
7.282
7.280
7.269
7.265
7.264
7.260
7.205
7.187
7.168
7.165
7.163
7.162
7.160
7.148
7.146
7.145
7.143
7.142
7.136
6.765
6.739
6.735
6.682
6.681
6.678
6.677
6.662
6.661
6.658
6.657
5.949
2.717
2.700
2.682
2.665
2.647
2.630
2.545
2.530
2.525
2.519
2.505
1.908
1.895
1.893
1.890
1.888
1.887
1.884
1.883
1.876
1.875
1.870
1.867
1.866
1.864
1.850
1.262
1.244
Ph
Me
O
O
41
1H NMR(400 MHz, CDCl3)
1.81
2.70
0.84
0.87
0.89
2.00
0.99
1.95
1.98
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


147.79
145.73
142.64
141.45
128.50
128.41
125.79
120.15
108.24
107.36
100.89
77.48
77.16
76.84
40.27
39.44
34.03
22.92
Ph
Me
O
O
41
13C NMR(101 MHz, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.285
7.281
7.267
7.260
7.248
7.188
7.185
7.170
7.157
7.153
7.136
6.382
6.377
6.334
6.328
6.322
3.800
2.707
2.689
2.672
2.654
2.636
2.619
2.551
2.531
2.511
1.969
1.951
1.948
1.935
1.930
1.915
1.910
1.896
1.890
1.875
1.870
1.860
1.854
1.840
1.836
1.268
1.251
Ph
Me
OMe
OMe
42
1H NMR(400 MHz, CDCl3)
1.91
2.67
1.73
0.86
6.00
0.92
1.90
1.90
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


160.91
150.06
142.66
128.53
128.40
125.78
105.38
97.74
77.48
77.16
76.84
55.39
40.01
39.91
34.05
22.58
Ph
Me
OMe
OMe
42
13C NMR(101 MHz, CDCl3)
f1 (ppm)


7.860 7.858 7.856 7.850 7.842 7.839 7.836 7.828 7.822 7.664 7.660 7.509 7.495 7.491 7.488 7.476 7.471 7.459 7.457 7.453 7.418 7.414 7.397 7.392 7.315 7.312 7.308 7.299 7.294 7.291 7.279 7.275 7.272 7.260 7.217 7.214 7.199 7.177 7.173 7.167 7.159 7.156 7.153
2.941 2.924 2.920 2.599 2.584 2.581 2.576 2.568 2.559 2.546 2.088 2.075 2.073 2.068 2.066 2.057 2.052 2.050 2.045 2.041 2.039 2.034 2.029 2.023 2.018 2.015 2.000 1.401 1.384
Ph
Me
43
1H NMR(400 MHz, CDCl3)
2.82
0.91
1.90
0.93
1.80
2.72
1.00
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



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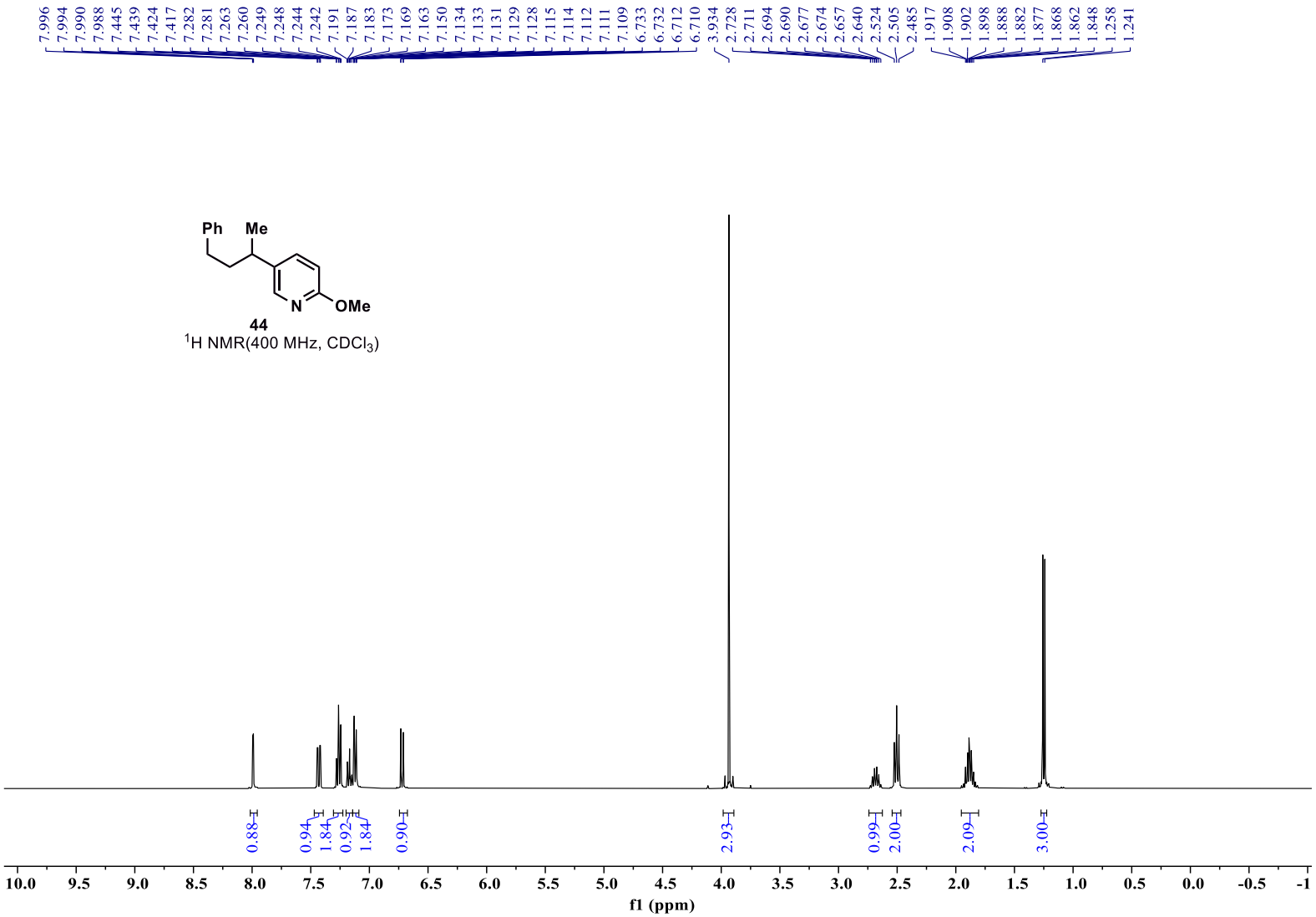
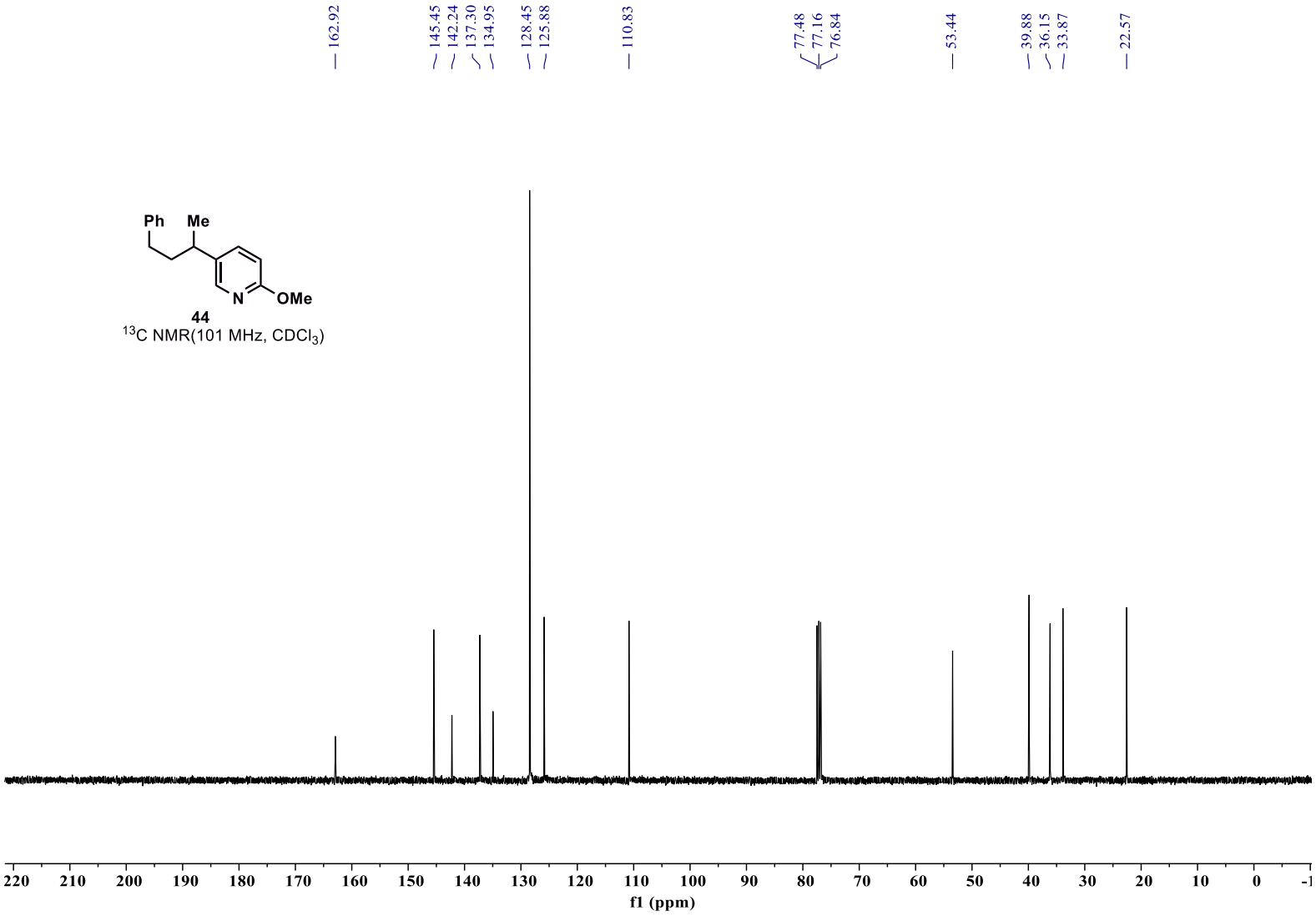
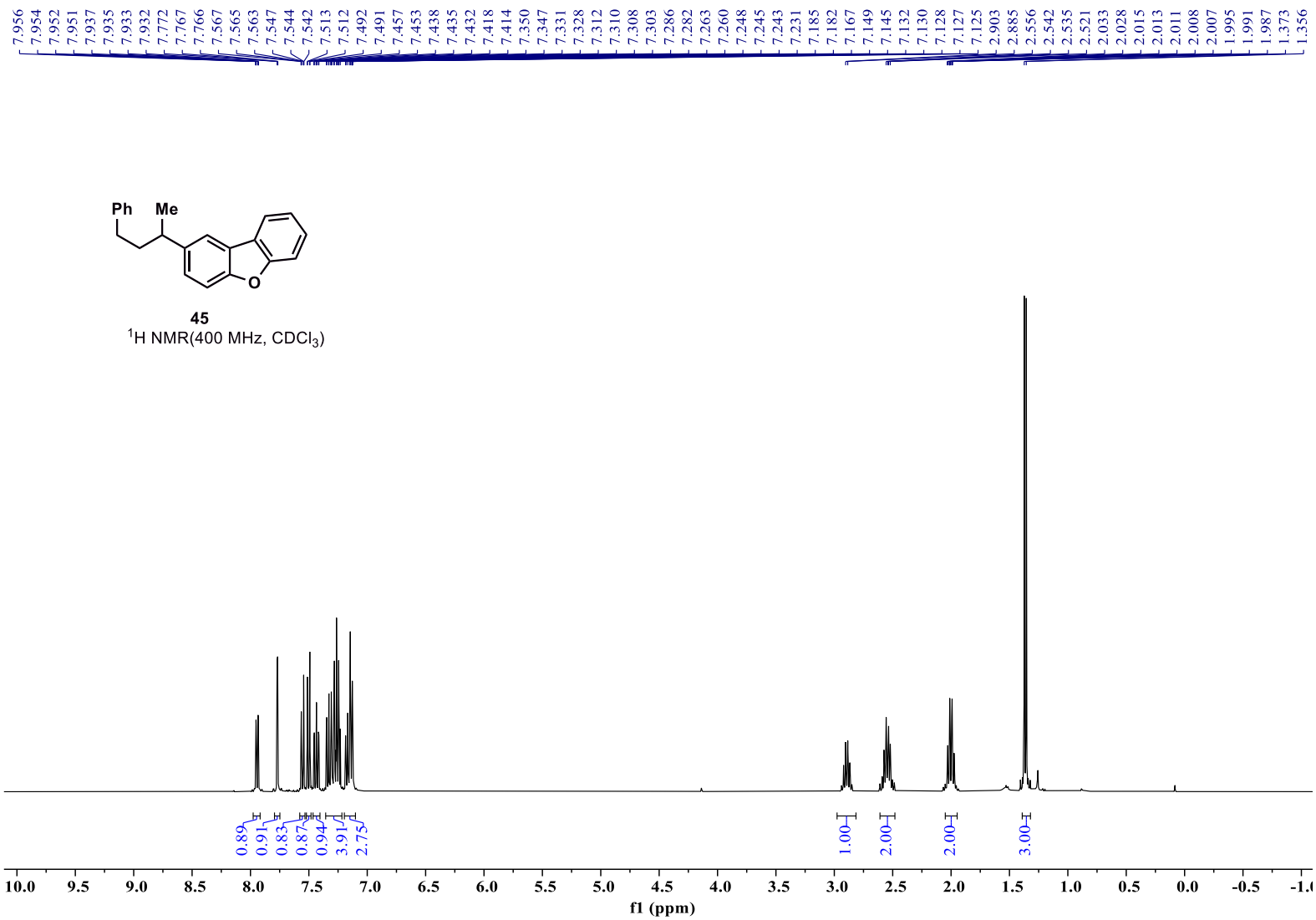
^{13}C NMR(101 MHz, $CDCl_3$)

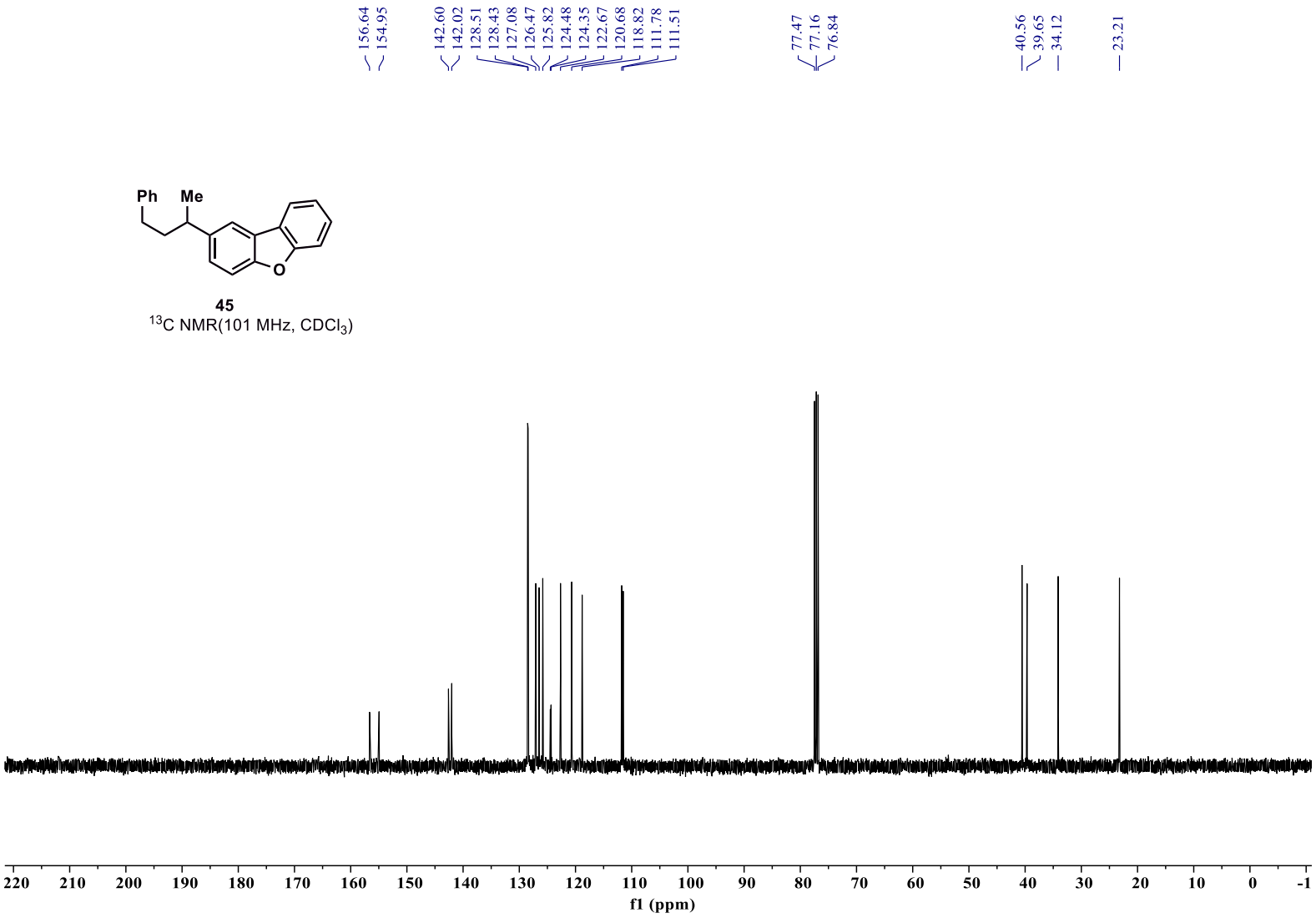

7.996
7.994
7.990
7.988
7.445
7.439
7.424
7.417
7.282
7.281
7.263
7.260
7.249
7.248
7.244
7.242
7.191
7.187
7.183
7.173
7.169
7.163
7.150
7.134
7.133
7.131
7.129
7.128
7.115
7.114
7.112
7.111
7.109
6.733
6.732
6.712
6.710
3.934
2.728
2.711
2.694
2.690
2.677
2.674
2.657
2.640
2.524
2.505
2.485
1.917
1.908
1.902
1.898
1.888
1.882
1.877
1.868
1.862
1.848
1.258
1.241
Ph
Me
N
OMe
44
1H NMR(400 MHz, CDCl3)
0.88
0.94
1.84
0.92
1.84
0.90
2.93
0.99
2.00
2.09
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

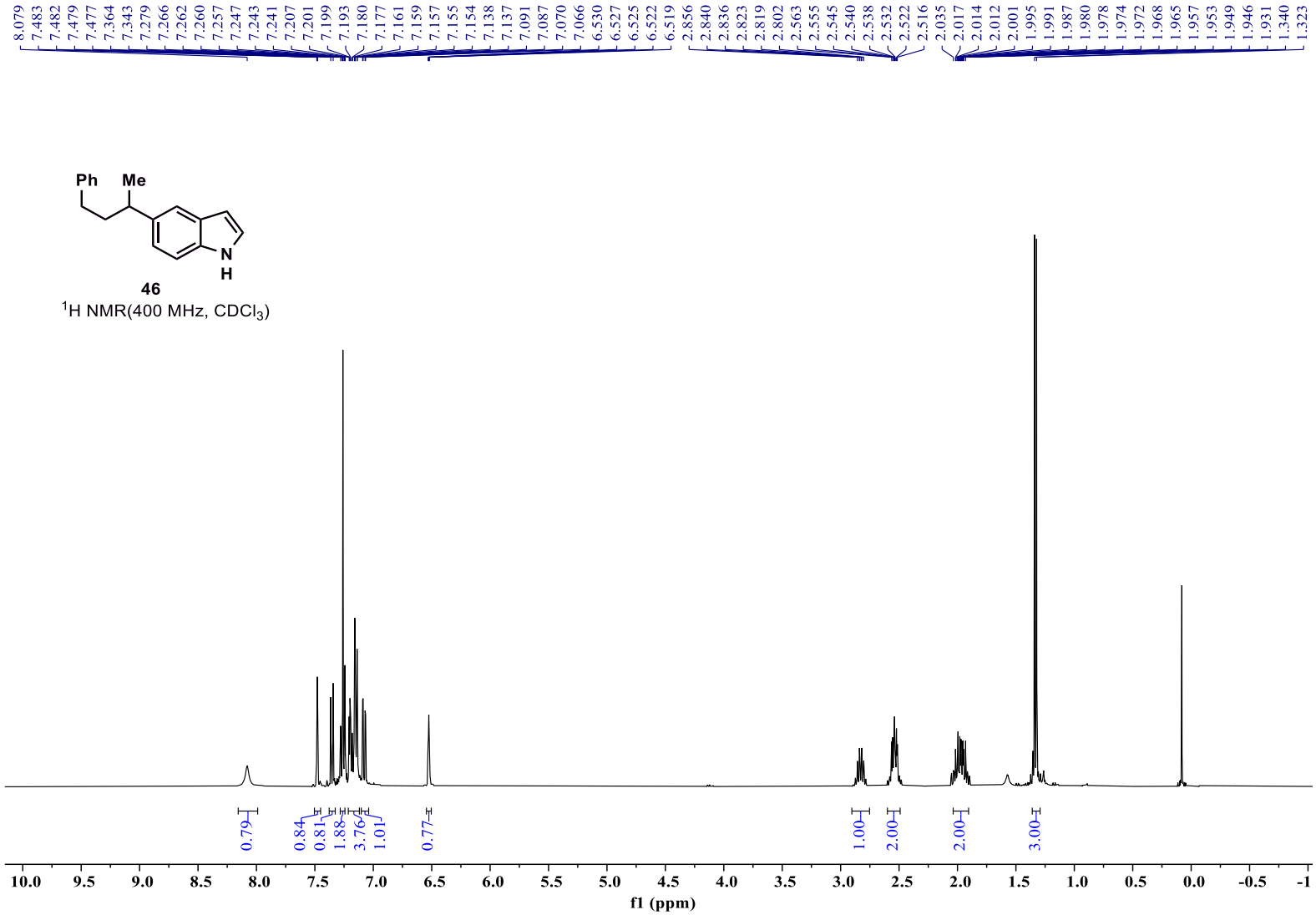

162.92
145.45
142.24
137.30
134.95
128.45
125.88
110.83
77.48
77.16
76.84
53.44
39.88
36.15
33.87
22.57
Ph
Me
N
OMe
44
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

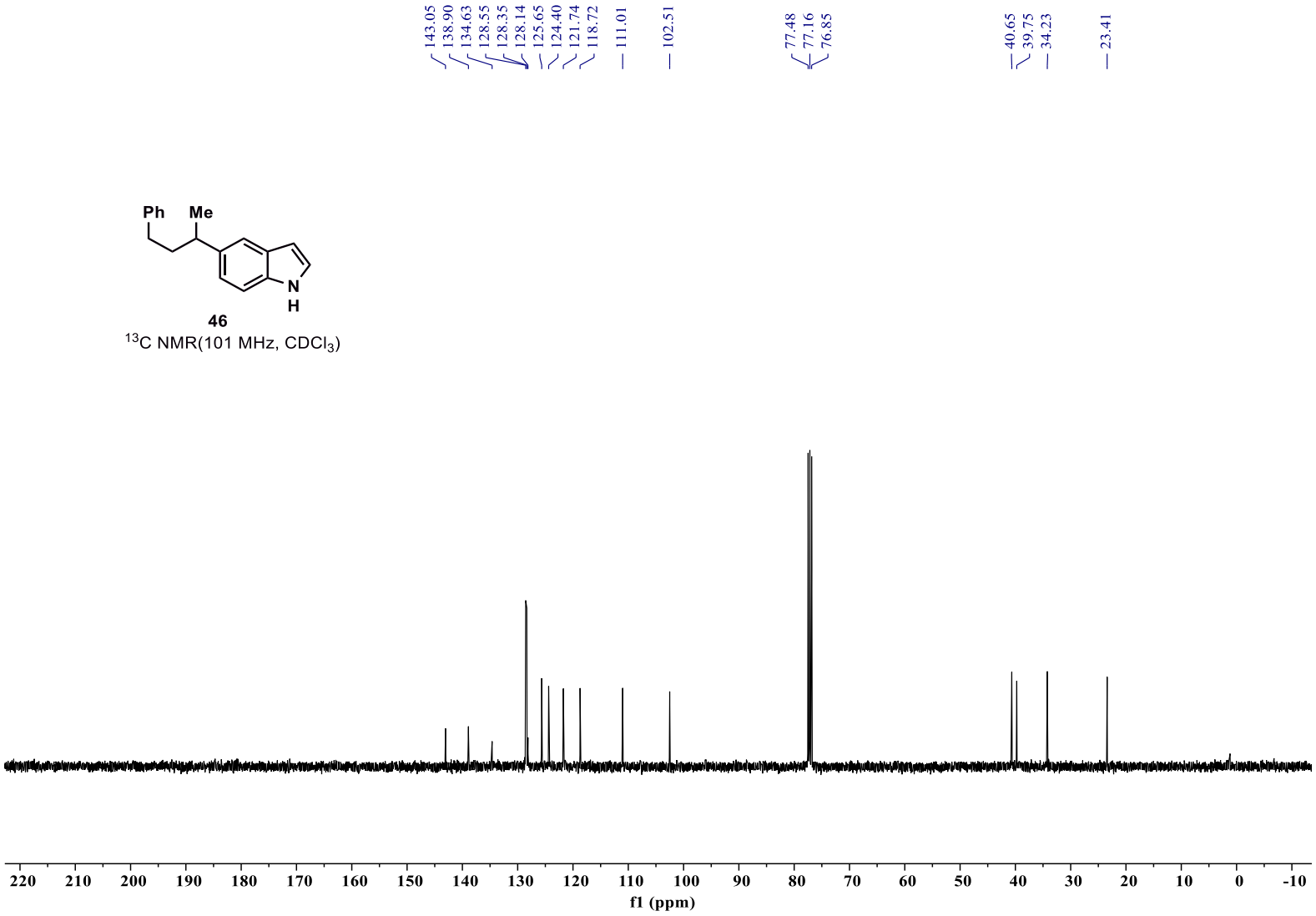

7.956
7.954
7.952
7.951
7.937
7.935
7.933
7.932
7.772
7.767
7.766
7.567
7.565
7.563
7.547
7.544
7.542
7.513
7.512
7.492
7.491
7.457
7.453
7.438
7.435
7.432
7.418
7.414
7.350
7.347
7.331
7.328
7.312
7.310
7.308
7.303
7.286
7.282
7.263
7.260
7.248
7.245
7.243
7.231
7.185
7.182
7.167
7.149
7.145
7.132
7.130
7.128
7.127
7.125
2.903
2.885
2.556
2.542
2.535
2.521
2.033
2.028
2.015
2.013
2.011
2.008
2.007
1.995
1.991
1.987
1.373
1.356
Ph
Me
O
45
1H NMR(400 MHz, CDCl3)
0.89
0.91
0.83
0.87
0.94
3.91
2.75
1.00
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

45

^{13}C NMR(101 MHz, $CDCl_3$)

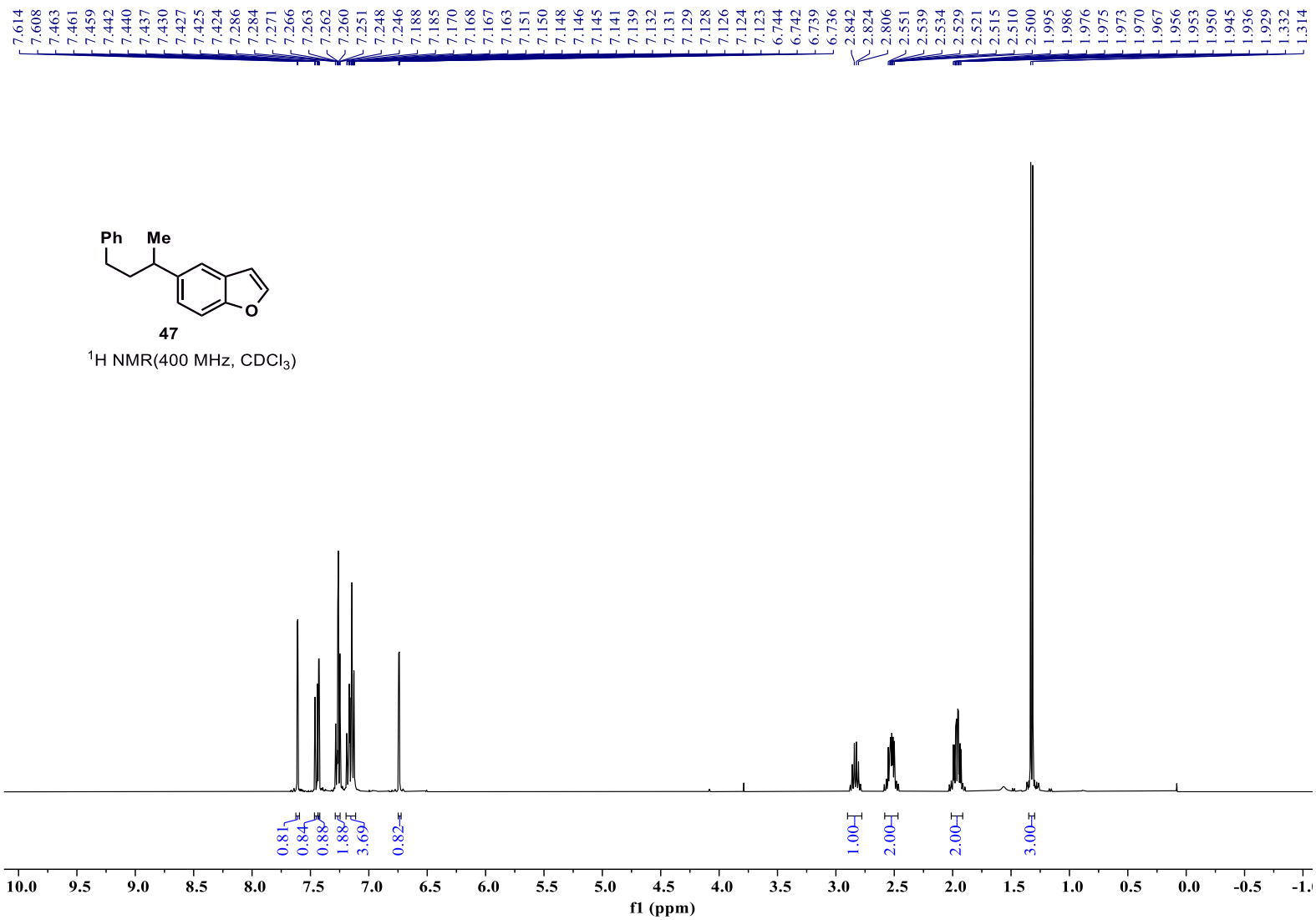


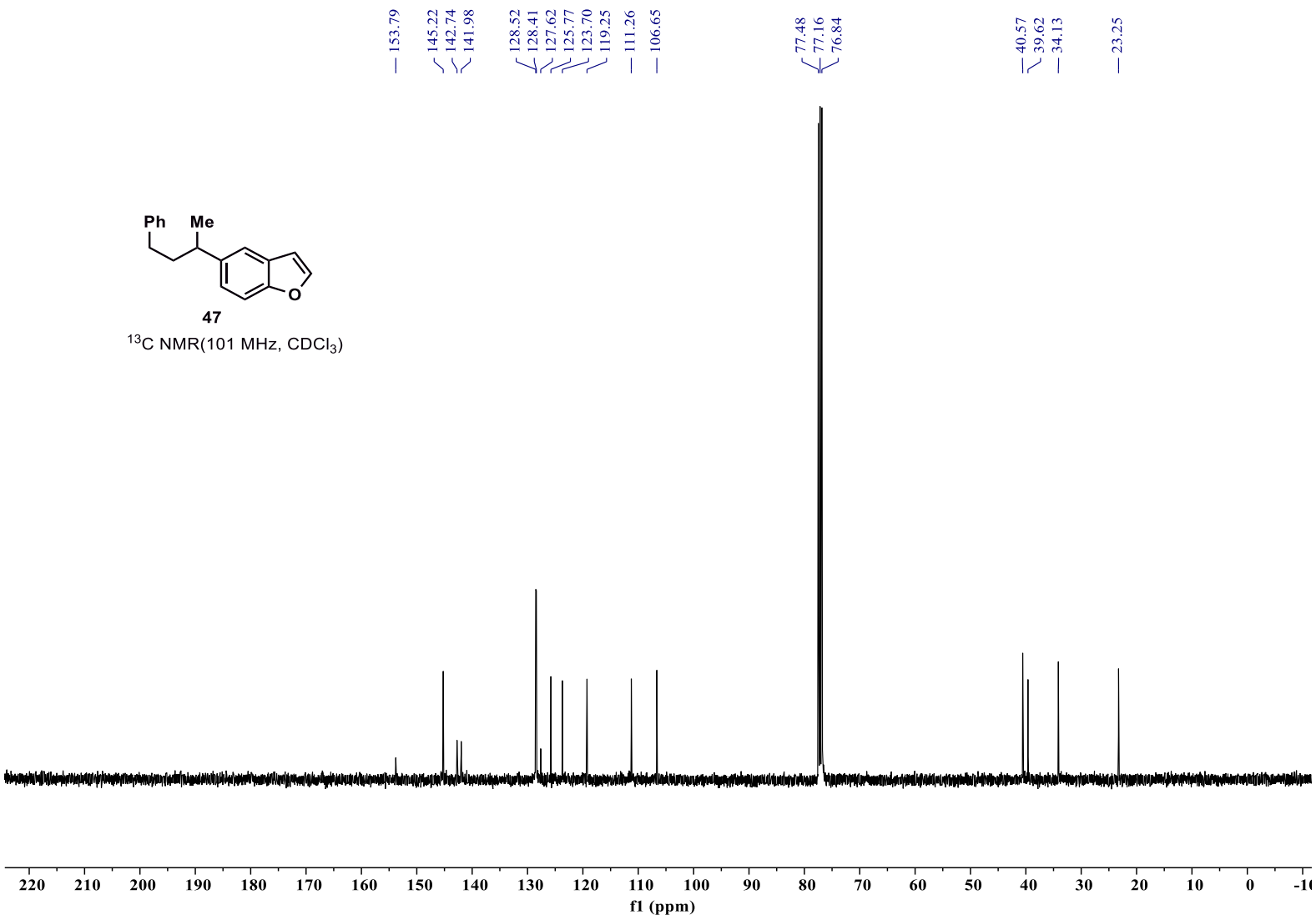

8.079
7.483
7.482
7.479
7.477
7.364
7.343
7.279
7.266
7.262
7.260
7.257
7.247
7.243
7.241
7.207
7.201
7.199
7.193
7.180
7.177
7.161
7.159
7.157
7.155
7.154
7.138
7.137
7.091
7.087
7.070
7.066
6.530
6.527
6.525
6.522
6.519
2.856
2.840
2.836
2.823
2.819
2.802
2.563
2.555
2.545
2.540
2.538
2.532
2.522
2.516
2.035
2.017
2.014
2.012
2.001
1.995
1.991
1.987
1.980
1.978
1.974
1.972
1.968
1.965
1.957
1.953
1.949
1.946
1.931
1.340
1.323
Ph
Me
N
H
46
1H NMR(400 MHz, CDCl3)
0.79
0.84
0.81
1.88
3.76
1.01
0.77
1.00
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

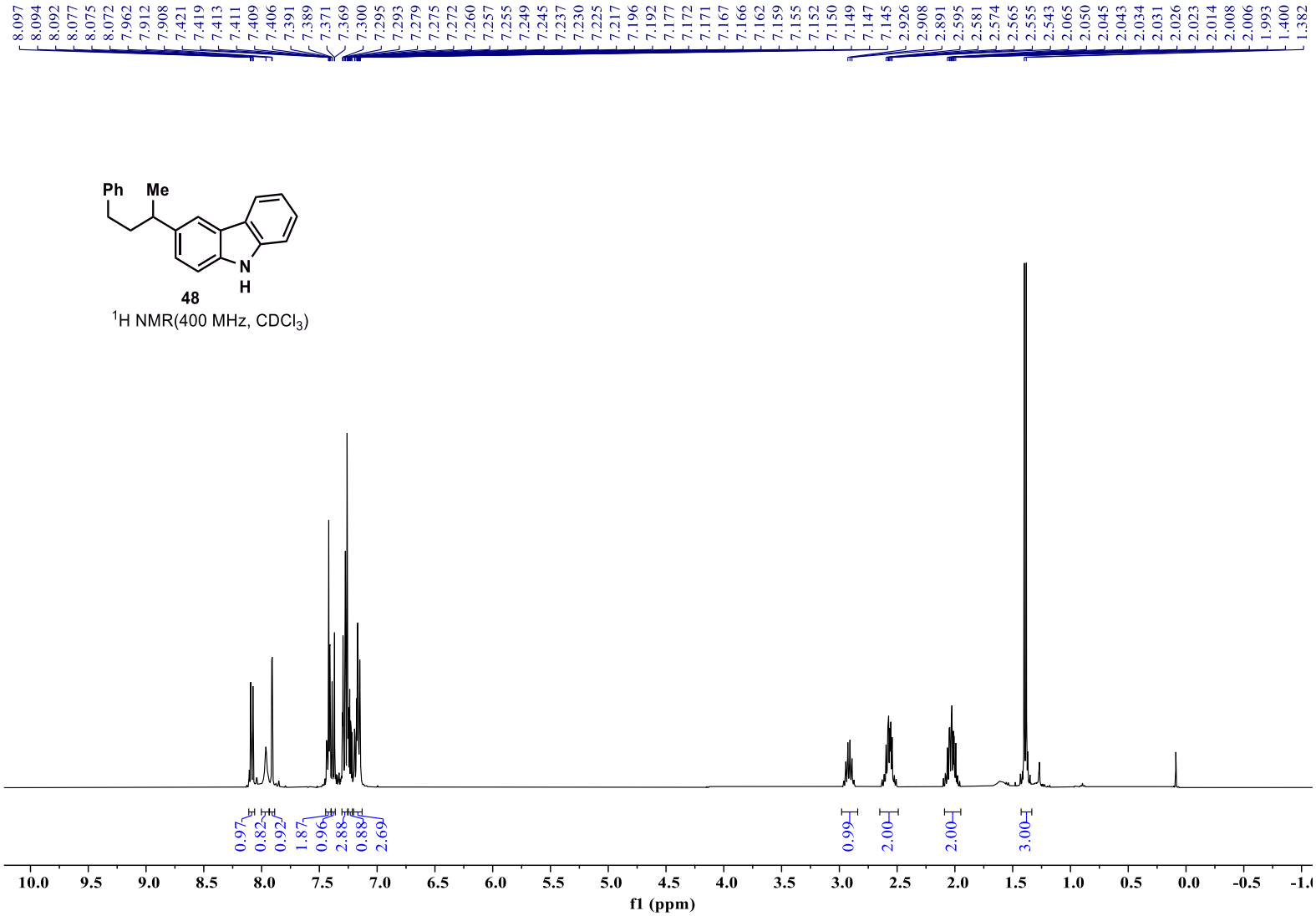


46

^{13}C NMR(101 MHz, $CDCl_3$)

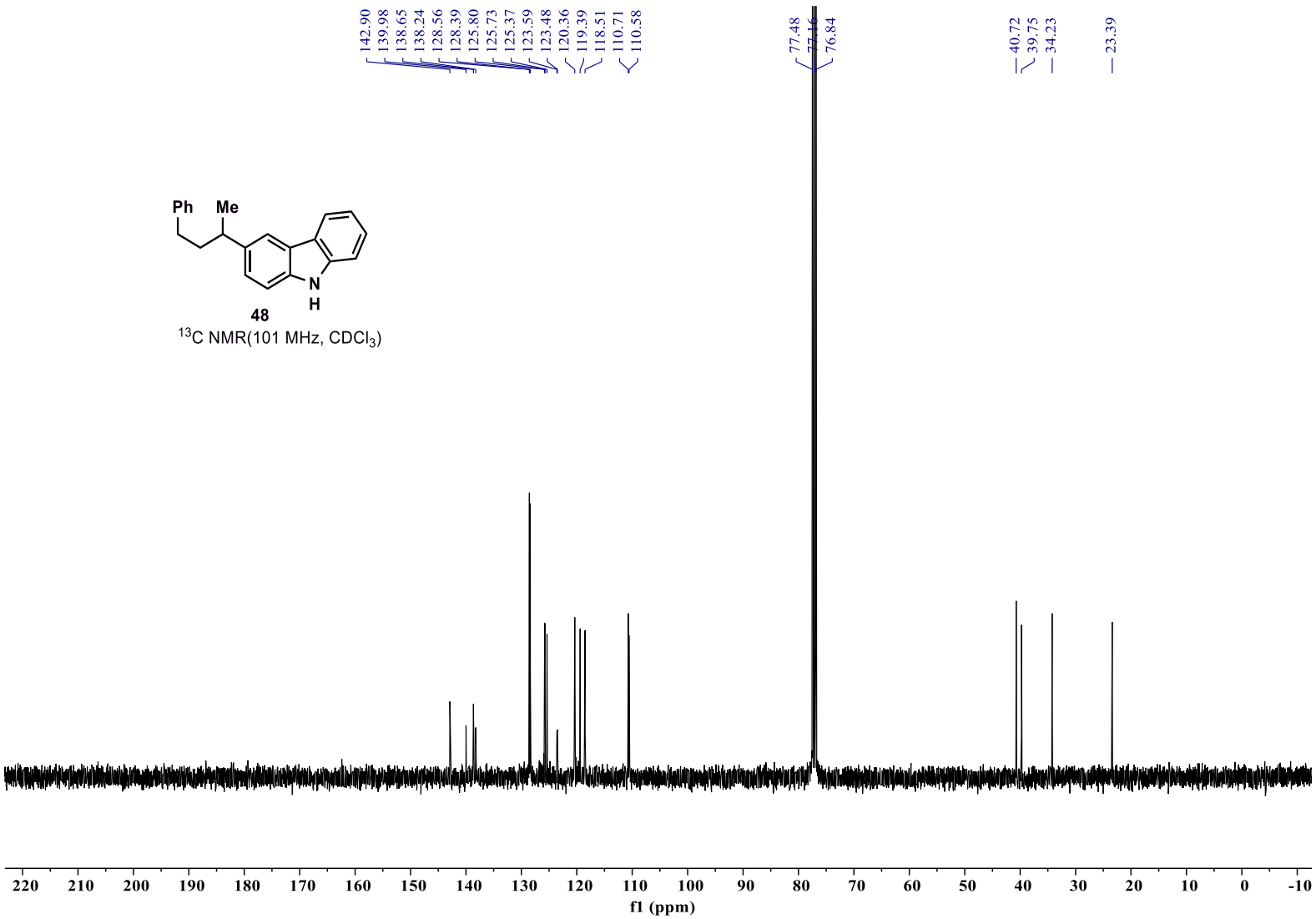

7.614 7.608 7.463 7.461 7.459 7.442 7.440 7.437 7.430 7.427 7.425 7.424 7.286 7.284 7.271 7.266 7.263 7.262 7.260 7.251 7.248 7.246 7.188 7.185 7.170 7.168 7.167 7.163 7.151 7.150 7.148 7.146 7.145 7.141 7.139 7.132 7.131 7.129 7.128 7.126 7.124 7.123 6.744 6.742 6.739 6.736 2.842 2.824 2.806 2.551 2.539 2.534 2.529 2.521 2.515 2.510 2.500 1.995 1.986 1.976 1.975 1.973 1.970 1.967 1.956 1.953 1.950 1.945 1.936 1.929 1.332 1.314
Ph
Me
O
47
^{1}H NMR(400 MHz, $CDCl_3$)
0.81
0.84
0.88
1.88
3.69
0.82
1.00
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

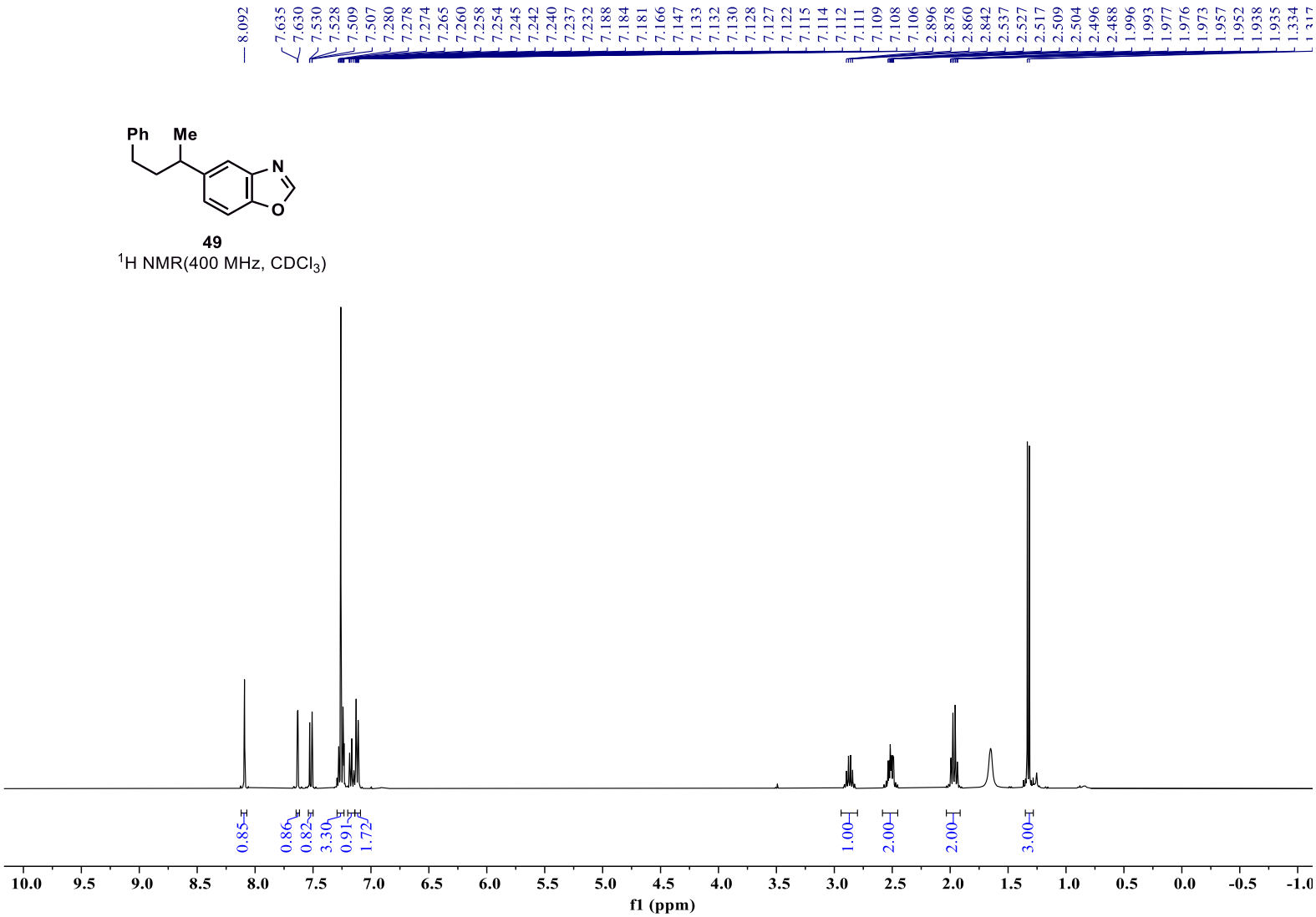

153.79
145.22
142.74
141.98
128.52
128.41
127.62
125.77
123.70
119.25
111.26
106.65
77.48
77.16
76.84
40.57
39.62
34.13
23.25
Ph
Me
O
47
13C NMR(101 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

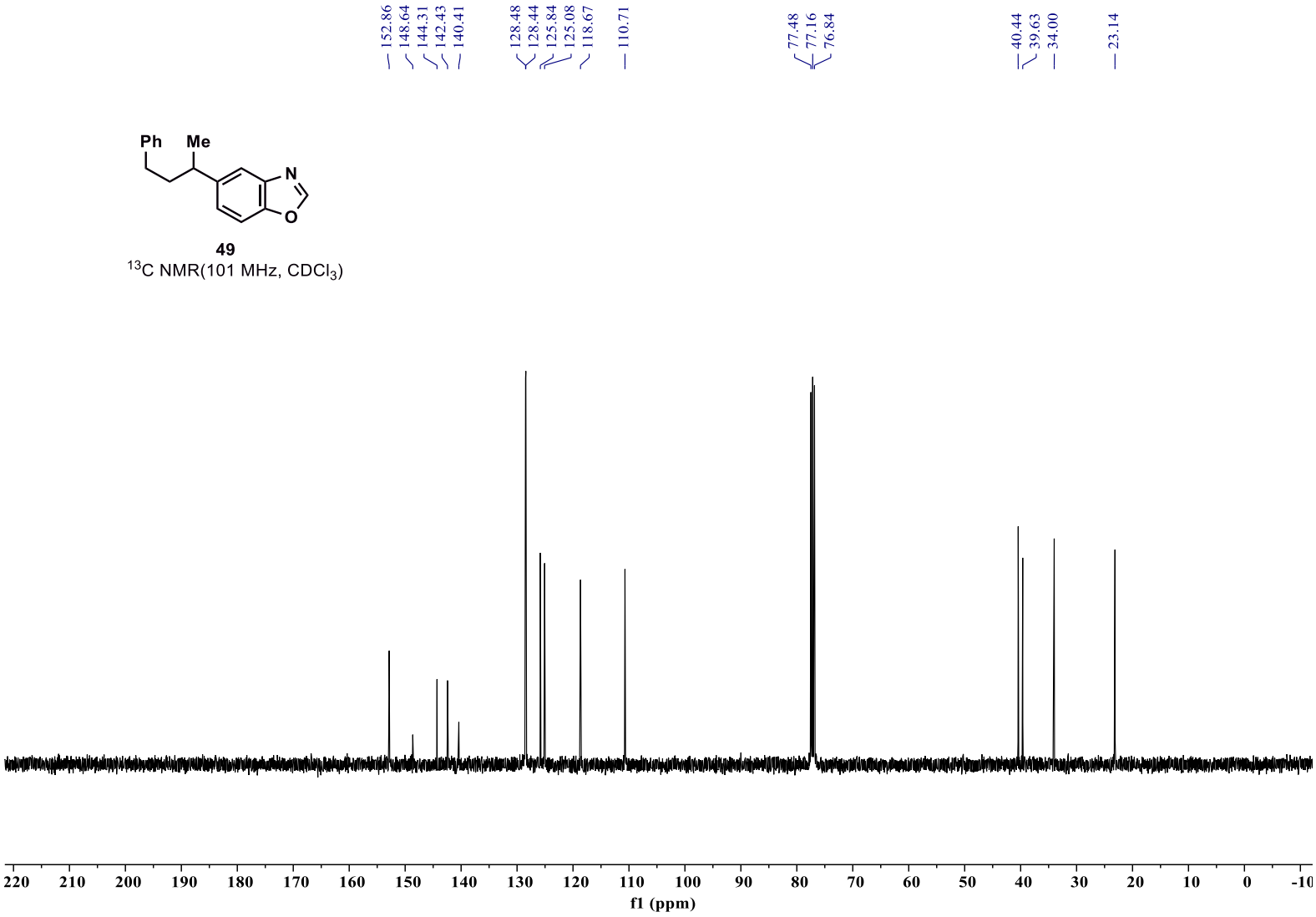

8.097 8.094 8.092 8.077 8.075 8.072 7.962 7.912 7.908 7.421 7.419 7.413 7.411 7.409 7.406 7.391 7.389 7.371 7.369 7.300 7.295 7.293 7.279 7.275 7.272 7.260 7.257 7.255 7.249 7.245 7.237 7.230 7.225 7.217 7.196 7.192 7.177 7.172 7.171 7.167 7.166 7.162 7.159 7.155 7.152 7.150 7.149 7.147 7.145 2.926 2.908 2.891 2.595 2.581 2.574 2.565 2.555 2.543 2.065 2.050 2.045 2.043 2.034 2.031 2.026 2.023 2.014 2.008 2.006 1.993 1.400 1.382
Ph
Me
N
H
48
^{1}H NMR(400 MHz, $CDCl_3$)
0.97
0.82
0.92
1.87
0.96
2.88
0.88
2.69
0.99
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

48

^{13}C NMR(101 MHz, $CDCl_3$)

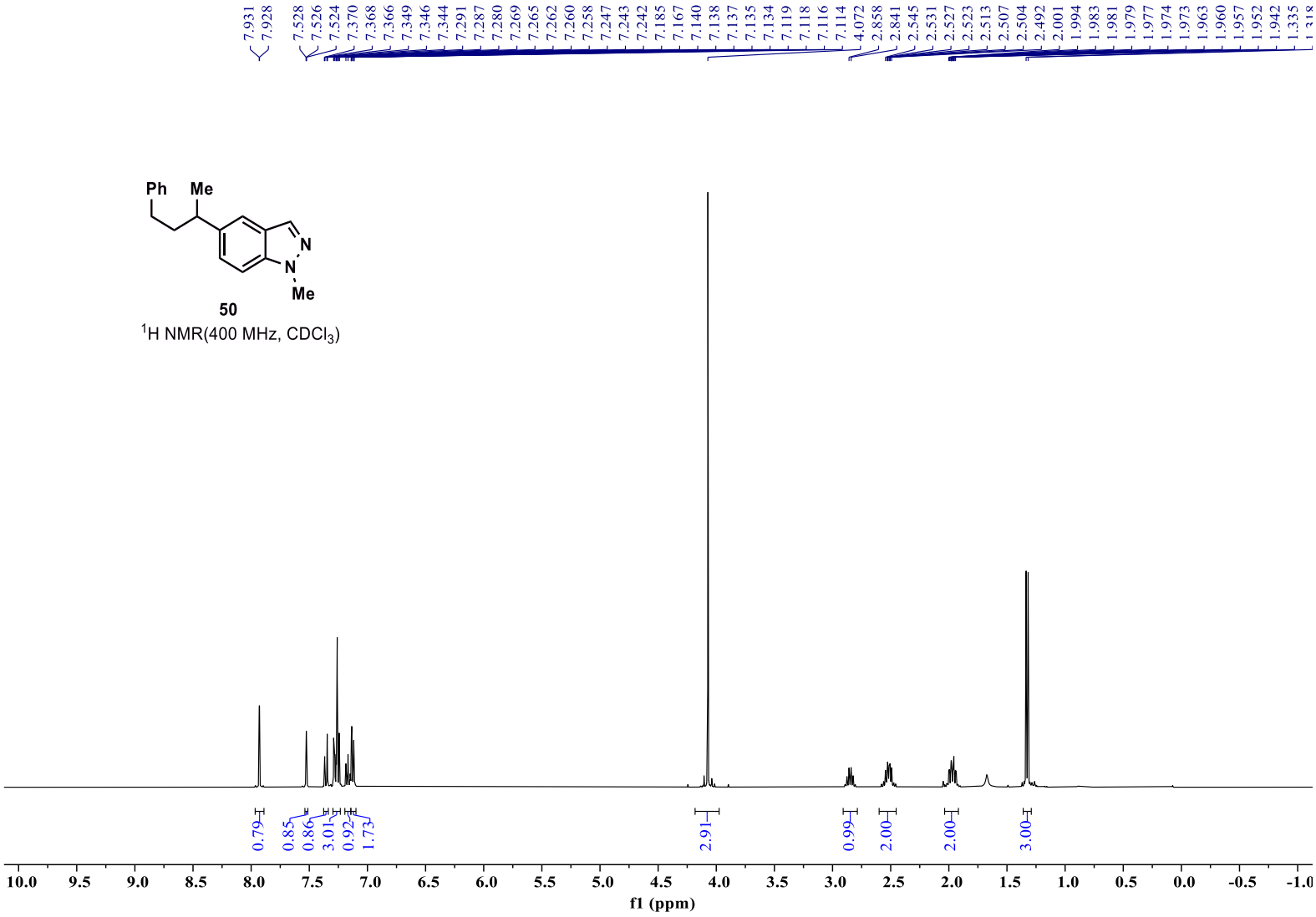



8.092
7.635
7.630
7.530
7.528
7.509
7.507
7.280
7.278
7.274
7.265
7.260
7.258
7.254
7.245
7.242
7.240
7.237
7.232
7.188
7.184
7.181
7.166
7.147
7.133
7.132
7.130
7.128
7.127
7.122
7.115
7.114
7.112
7.111
7.109
7.108
7.106
2.896
2.878
2.860
2.842
2.537
2.527
2.517
2.509
2.504
2.496
2.488
1.996
1.993
1.977
1.976
1.973
1.957
1.952
1.938
1.935
1.334
Ph
Me
N
O
49
1H NMR(400 MHz, CDCl3)
0.85
0.86
0.82
3.30
0.91
1.72
1.00
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



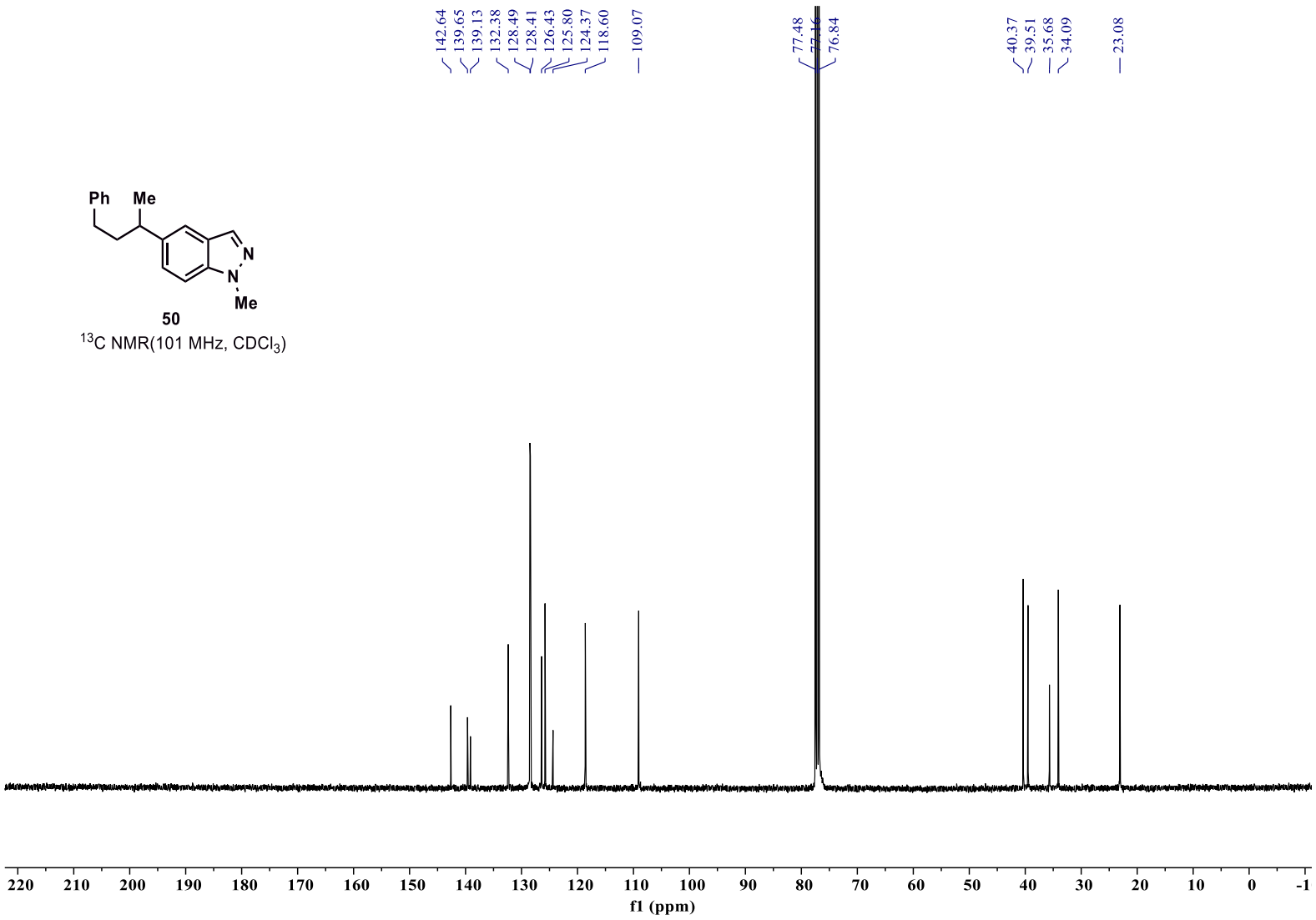
49

^{13}C NMR(101 MHz, $CDCl_3$)


7.931
7.928
7.528
7.526
7.524
7.370
7.368
7.366
7.349
7.346
7.344
7.291
7.287
7.280
7.269
7.265
7.262
7.260
7.258
7.247
7.243
7.242
7.185
7.167
7.140
7.138
7.137
7.135
7.134
7.119
7.118
7.116
7.114
4.072
2.858
2.841
2.545
2.531
2.527
2.523
2.513
2.507
2.504
2.492
2.001
1.994
1.983
1.981
1.979
1.977
1.974
1.973
1.963
1.960
1.957
1.952
1.942
1.335
Ph
Me
N
N
Me
50
1H NMR(400 MHz, CDCl3)
0.79
0.85
0.86
3.01
0.92
1.73
2.91
0.99
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

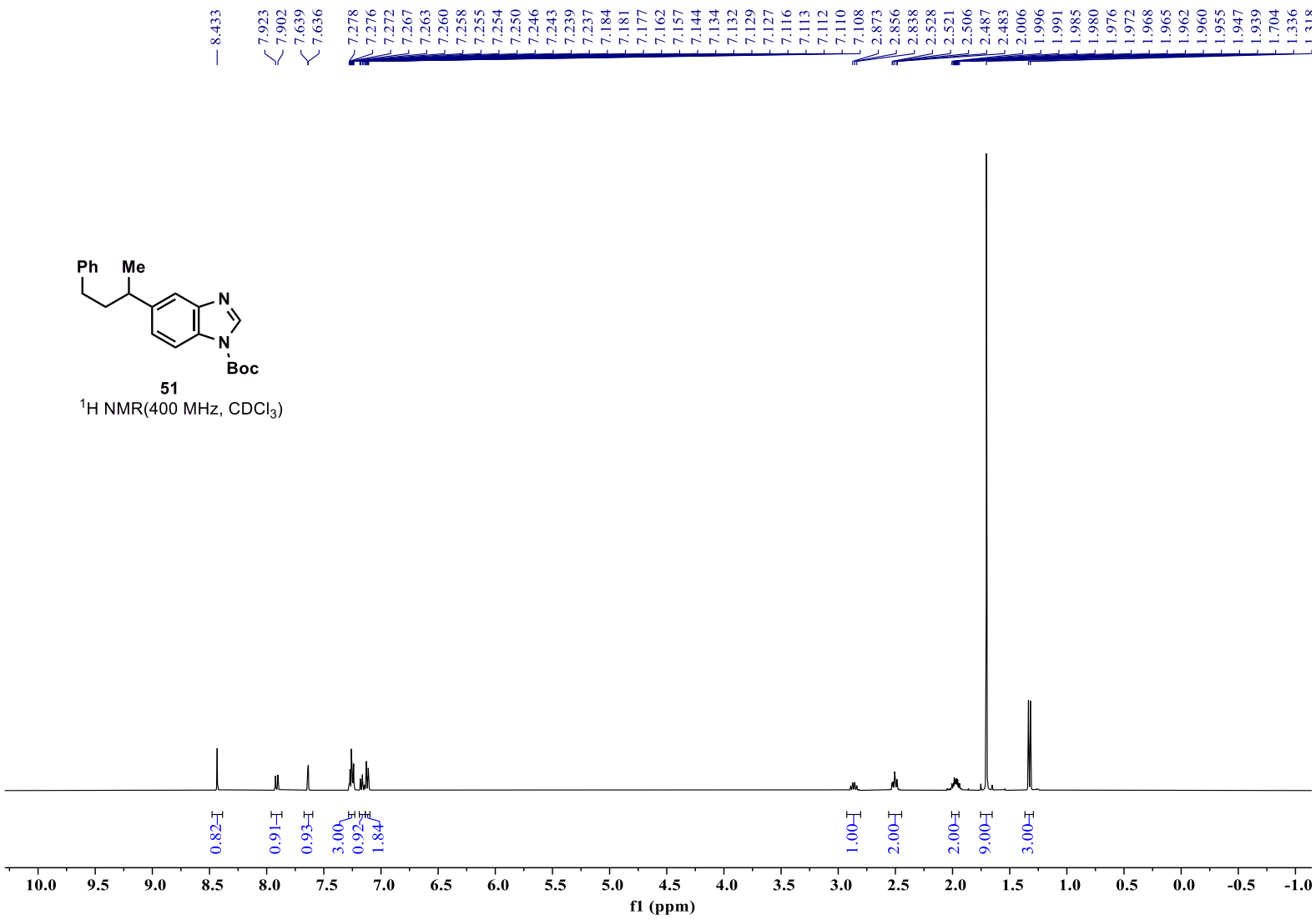
50

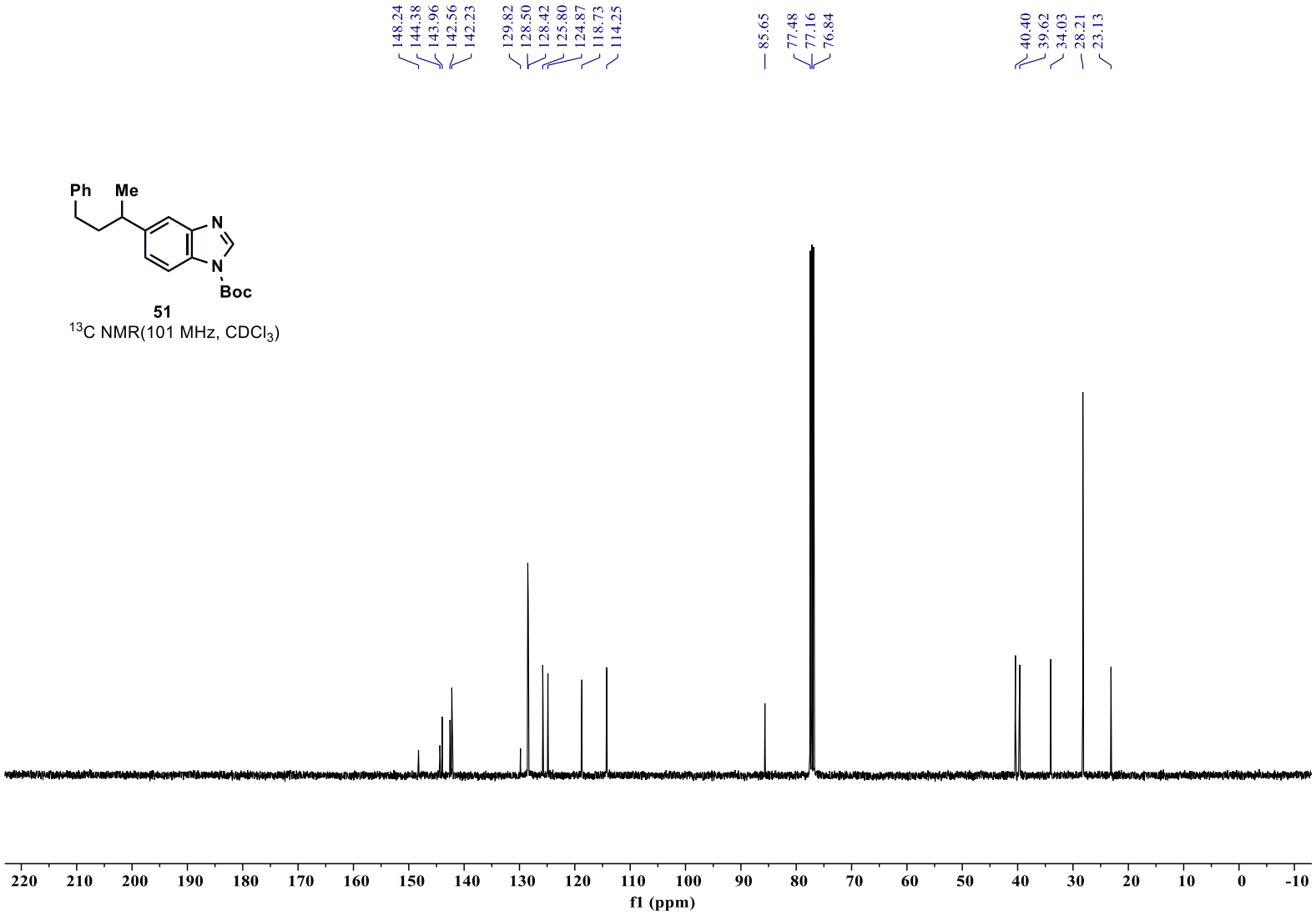
^{13}C NMR(101 MHz, $CDCl_3$)



51

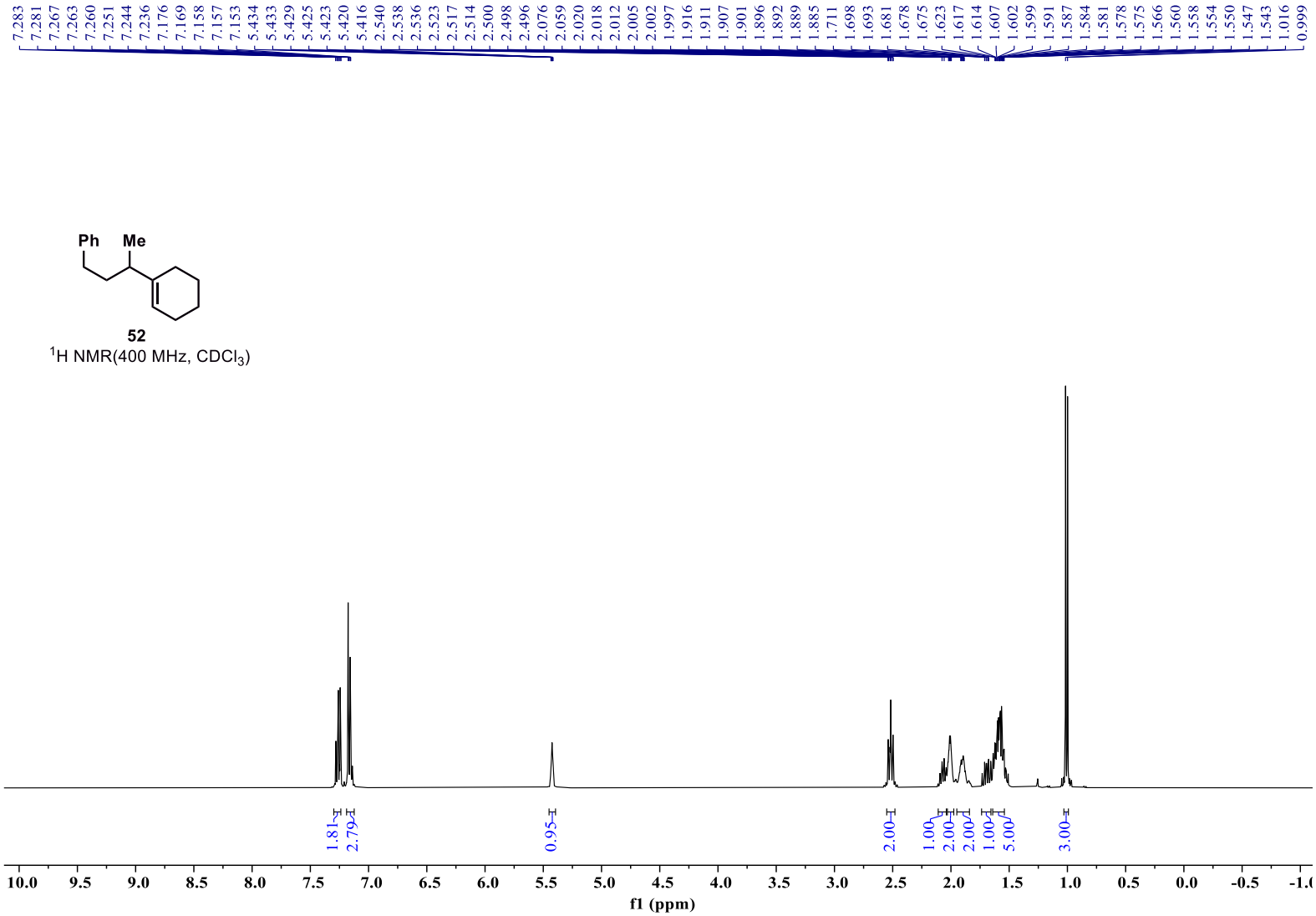
^{1}H NMR(400 MHz, $CDCl_3$)





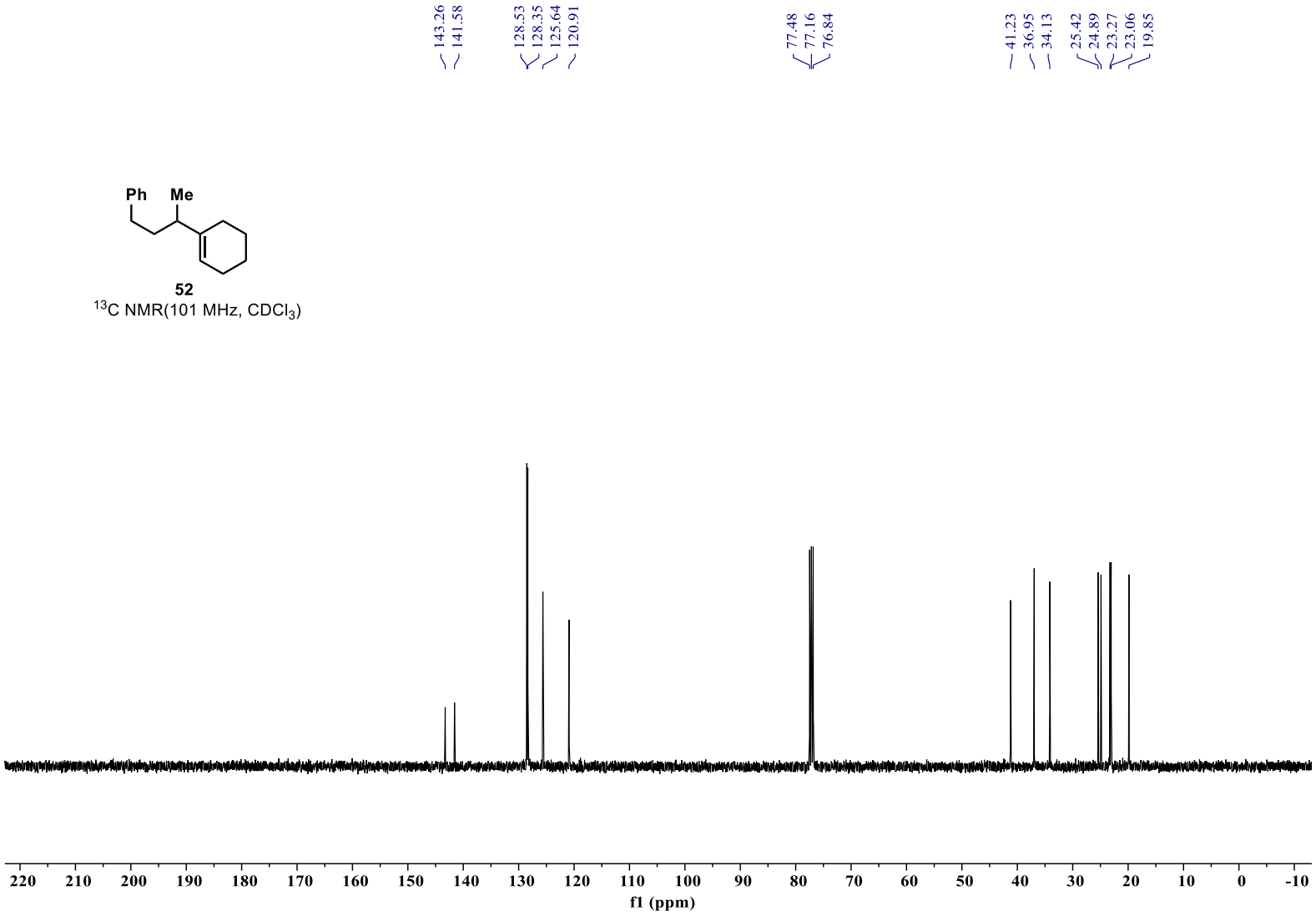
51

^{13}C NMR(101 MHz, $CDCl_3$)


7.283
7.281
7.267
7.263
7.260
7.251
7.244
7.236
7.176
7.169
7.158
7.157
7.153
5.434
5.433
5.429
5.425
5.423
5.420
5.416
2.540
2.538
2.536
2.523
2.517
2.514
2.500
2.498
2.496
2.076
2.059
2.020
2.018
2.012
2.005
2.002
1.997
1.916
1.911
1.907
1.901
1.896
1.892
1.889
1.885
1.711
1.698
1.693
1.681
1.678
1.675
1.623
1.617
1.614
1.607
1.602
1.599
1.591
1.587
1.584
1.581
1.578
1.575
1.566
1.560
1.558
1.554
1.550
1.547
1.543
1.016
0.999
Ph
Me
52
1H NMR(400 MHz, CDCl3)
1.81
2.79
0.95
2.00
1.00
2.00
2.00
1.00
5.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

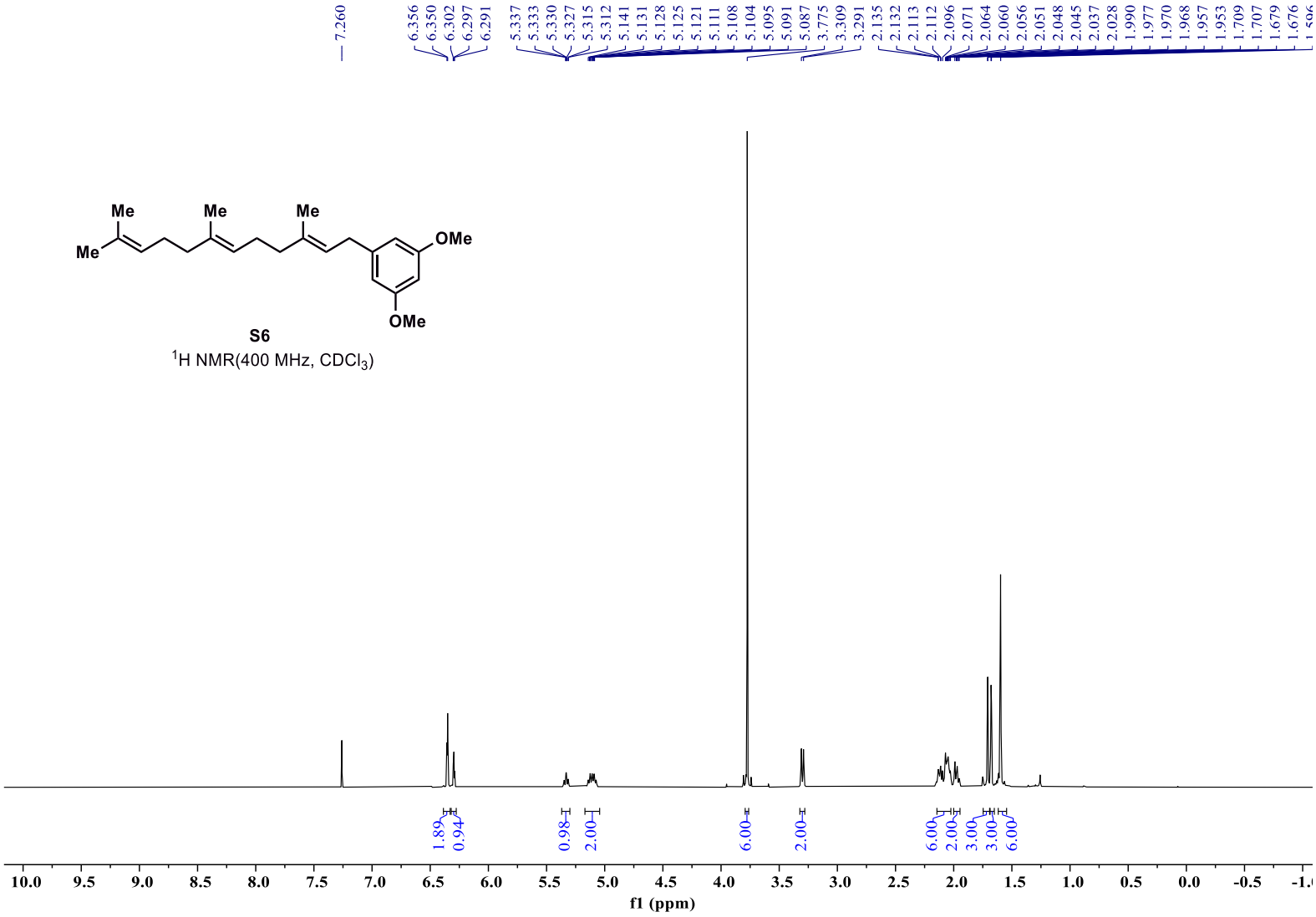
52

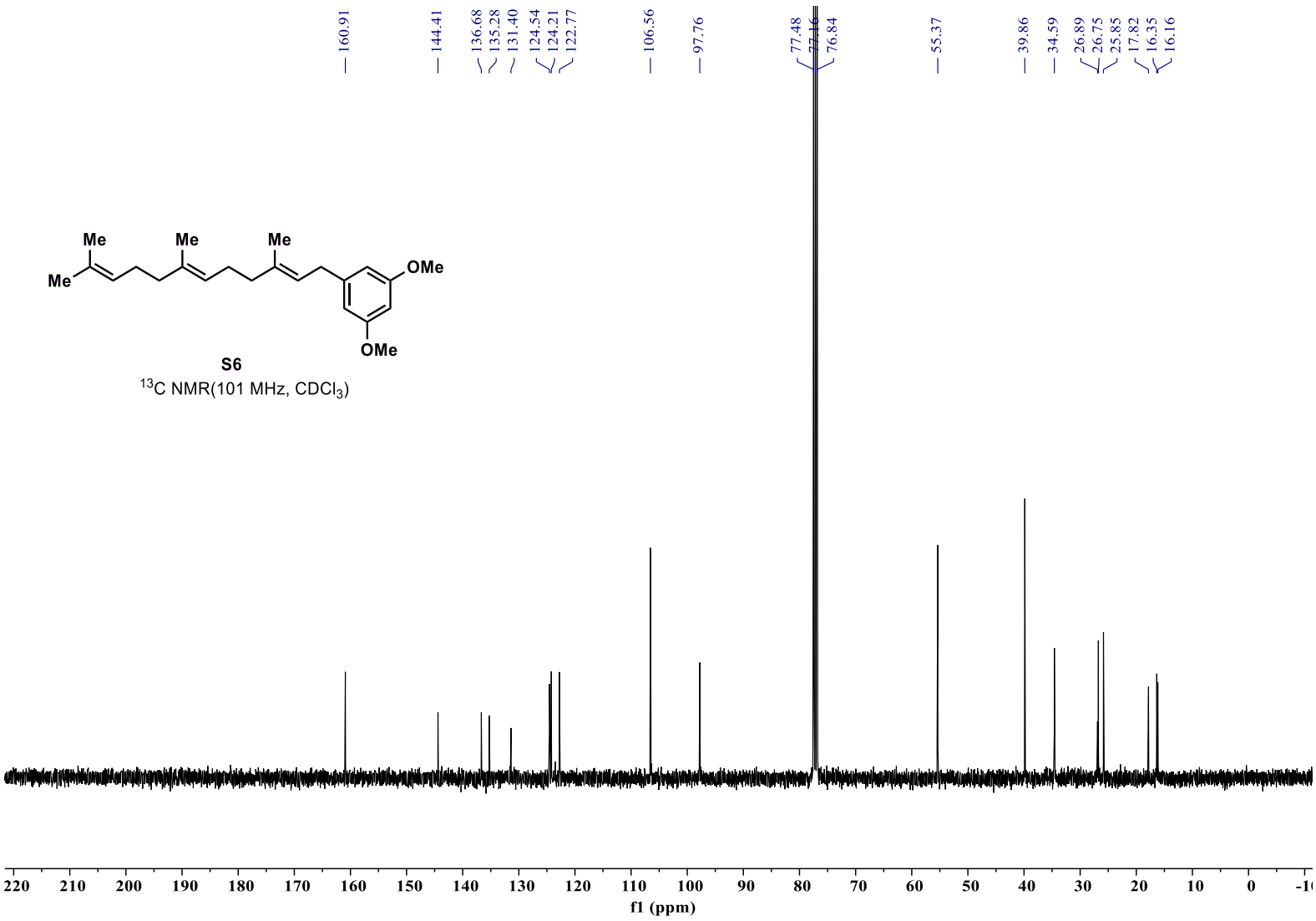
^{13}C NMR(101 MHz, $CDCl_3$)



S6

^{1}H NMR(400 MHz, $CDCl_3$)





S6

^{13}C NMR(101 MHz, $CDCl_3$)