Supporting information

Electroreductive Cross-Coupling between Aldehydes and Ketones or Imines via Cathodically Generated Dianions

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General

All manipulations were conducted in an IKA® ElectraSyn 2.0 cell under a nitrogen atmosphere. Unless otherwise noted, all chemicals and solvents obtained from commercial suppliers (*Alfa*, *Acros*, *Aldrich*, *TCI*, *J&K Chemical*, *Energy Chemical*) were used without further purification. Anhydrous DMF, DMA, CH_2Cl_2, CH_3CN, and DMSO were purchased from J&K Chemical or Energy Chemical and used as received. These solvents were dried and degassed by commercial suppliers.

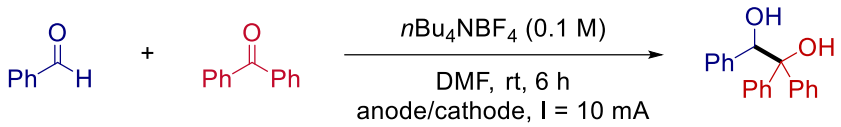
Flash column chromatography was carried out on silica gel (200-300 mesh). Thin layer chromatography (TLC) was performed using silica gel 60 F_{254} plates.

^1H NMR spectra were recorded on a *Bruker AV-300* or *AV-400* spectrometer at room temperature. Chemical shifts (in ppm) were referenced to tetramethylsilane (δ = 0 ppm) in $CDCl_3$ as an internal standard. ^{13}C NMR spectra were obtained by the same NMR spectrometer and were calibrated with $CDCl_3$ (δ = 77.00 ppm). ^{19}F NMR spectra were obtained by the same NMR spectrometer and using $CFCl_3$ as external standard. ^{19}F NMR spectra were decoupled with hydrogens. Data for ^1H NMR are reported as follows: chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, brs = broad singlet), coupling constant (Hz) and integration. Data for ^{13}C NMR are reported in terms of chemical shift and multiplicity where appropriate. High-Resolution Mass Spectrometry (HRMS) were performed on an *Aglient 6530 Q-TOF* for HRMS. Cyclic voltammetry (CV) experiments were performed using a Shanghai Chenhua CHI620E workstation. The yields were determined on a *METTLER TOLEDO ME 104* balance (accuracy: 0.1 mg).

Optimization of reaction conditions

Optimization studies for the electroreductive cross-coupling of aldehydes with ketones

Table S1. Evaluation of electrode materials[a]

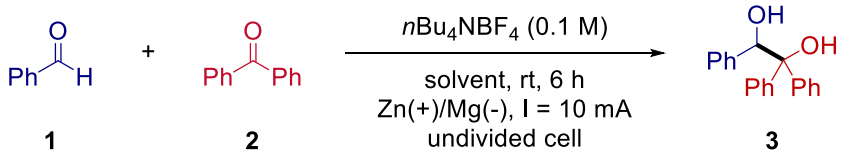


1 **2** **3**

entry	anode	cathode	yield[b] (%)
1	Zn	Zn	70
2	Zn	Sn	62
3	Zn	reticulated vitreous carbon (RVC)	44
4	Zn	graphite	69
5	Zn	nickel foam	66
6	Zn	Mg	75
7	Mg	Mg	58
8	graphite	Mg	46
9	graphite	graphite	55

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.80 mmol) and **2** (0.60 mmol) in DMF (6.0 mL) with 0.1 M *n*Bu$_4$NBF$_4$ (0.60 mmol) as supporting electrolyte were electrolyzed under a constant current of 10 mA at room temperature under N_2 for 6 h. [b]Isolated yields.

Table S2. Evaluation of solvents[a]

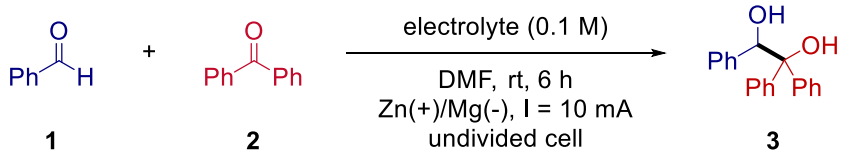


1 **2** **3**

entry	solvent	yield[b] (%)
1	DMF	75
2	DMA	72
3	CH_2Cl_2	31
4	CH_3CN	54
5	DMSO	72

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.80 mmol) and **2** (0.60 mmol) in solvent (6.0 mL) with 0.1 M *n*Bu$_4$NBF$_4$ (0.60 mmol) as supporting electrolyte were electrolyzed under a constant current of 10 mA at room temperature under N_2 for 6 h. [b]Isolated yields.

Table S3. Evaluation of electrolytes[a]

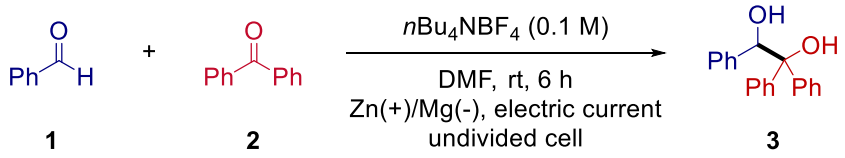


1 **2** **3**

entry	electrolyte	yield[b] (%)
1	*n*Bu$_4$NBF$_4$ (0.1 M)	75
2	*n*Bu$_4$NClO$_4$ (0.1 M)	65
3	*n*Bu$_4$NPF$_6$ (0.1 M)	66
4	*n*Bu$_4$NBr (0.1 M)	71
5	LiBr (0.1 M)	57

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.80 mmol) and **2** (0.60 mmol) in DMF (6.0 mL) with 0.1 M electrolyte (0.60 mmol) were electrolyzed under a constant current of 10 mA at room temperature under N_2 for 6 h. [b]Isolated yields.

Table S4. Evaluation of electric currents[a]

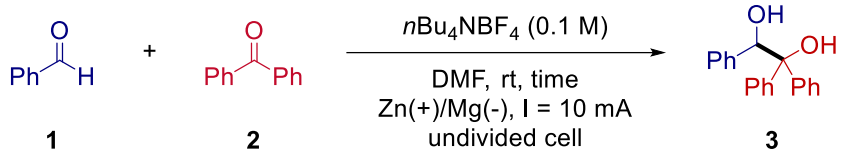


1 **2** **3**

entry	electric current (mA)	yield[b] (%)
1	5	28
2	10	75
3	15	72
4	0	0

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.80 mmol) and **2** (0.60 mmol) in DMF (6.0 mL) with 0.1 M *n*Bu$_4$NBF$_4$ (0.60 mmol) as supporting electrolyte were electrolyzed under a constant current at room temperature under N_2 for 6 h. [b]Isolated yields.

Table S5. Evaluation of other reaction parameters [a]

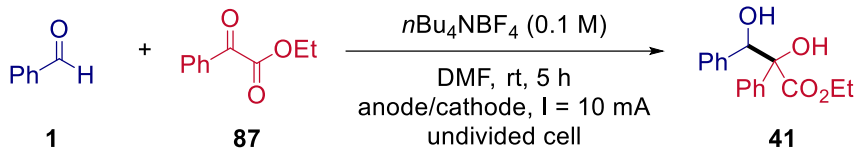


1 **2** **3**

entry	**1** (equiv)	time	yield[b] (%)
1	3.0	6 h	75
2	2.0	6 h	73
3	3.0	4 h	53
4	3.0	8 h	73
5[c]	3.0	8 h	50

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.80 mmol) and **2** (0.60 mmol) in DMF (6.0 mL) with 0.1 M *n*Bu$_4$NBF$_4$ (0.60 mmol) as supporting electrolyte were electrolyzed under a constant current of 10 mA at room temperature under N_2. [b]Isolated yields. [c]The reaction was electrolyzed under air.

Table S6. Optimization studies for the electroreductive cross-coupling of aldehydes with a-ketoesters[a]

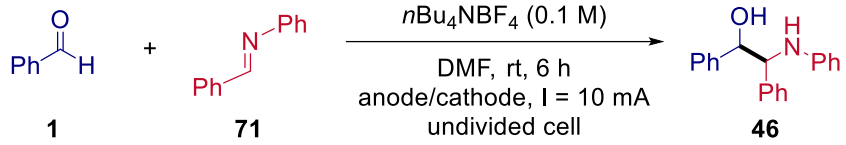


1 **87** **41**

entry	anode	cathode	yield[b] (%)
1	Zn	Mg	59
2	Zn	stainless steel	48
3	Zn	reticulated vitreous carbon (RVC)	75
4	Zn	graphite	55
5	Zn	nickel foam	53
6	Zn	Zn	81
7	Mg	Zn	38
8	graphite	Zn	64
9	RVC	Zn	57

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.20 mmol) and **87** (0.60 mmol) in DMF (6.0 mL) with 0.1 M nBu$_4$NBF$_4$ (0.60 mmol) as supporting electrolyte were electrolyzed under a constant current of 10 mA at room temperature under N_2 for 5 h. [b]Isolated yields.

Table S7. Optimization studies for the electroreductive cross-coupling of aldehydes with imines[a]

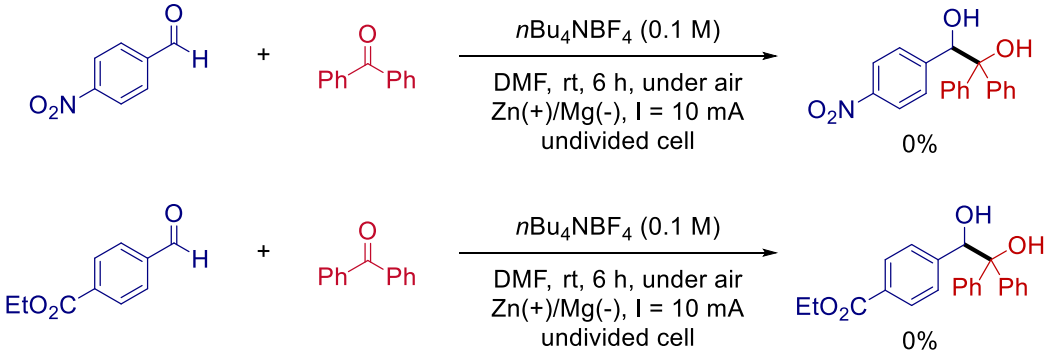


1 **71** **46**

entry	anode	cathode	**1** (equiv)	**71** (equiv)	yield[b] (%)
1	Zn	Mg	3.0	1.0	70
2	Zn	Zn	3.0	1.0	51
3	Zn	graphite	3.0	1.0	52
4	Zn	nickel foam	3.0	1.0	36
5	Mg	Mg	3.0	1.0	23
6	graphite	Mg	3.0	1.0	28
7	Zn	Mg	1.0	3.0	12

[a]Reaction conditions: in an ElectraSyn 2.0 cell, **1** (1.80 mmol) and **71** (0.60 mmol) in DMF (6.0 mL) with 0.1 M *n*Bu$_4$NBF$_4$ (0.60 mmol) as supporting electrolyte were electrolyzed under a constant current of 10 mA at room temperature under N_2 for 6 h. [b]Isolated yields.

Investigating aromatic aldehydes bearing strong electron-absorbing groups under the current electrolytic conditions



General procedure for electroreductive cross-coupling of aldehydes with ketones (GP 1):

Ketone (0.60 mmol, 1.0 equiv), aldehyde (1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (0.60 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash column chromatography on silica gel to afford the desired product.

General procedure for electroreductive cross-coupling of aldehydes with a-ketoesters (GP 2):

α-Ketoester (0.60 mmol, 1.0 equiv), aldehyde (1.20 mmol, 2.0 equiv), and *n*Bu_4NBF_4 (0.60 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 5 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash column chromatography on silica gel to afford the desired product.

General procedure for electroreductive cross-coupling of aldehydes with imines (GP 3):

Imine (0.60 mmol, 1.0 equiv), aldehyde (1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (0.60 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped

with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash column chromatography on silica gel to afford the desired product.

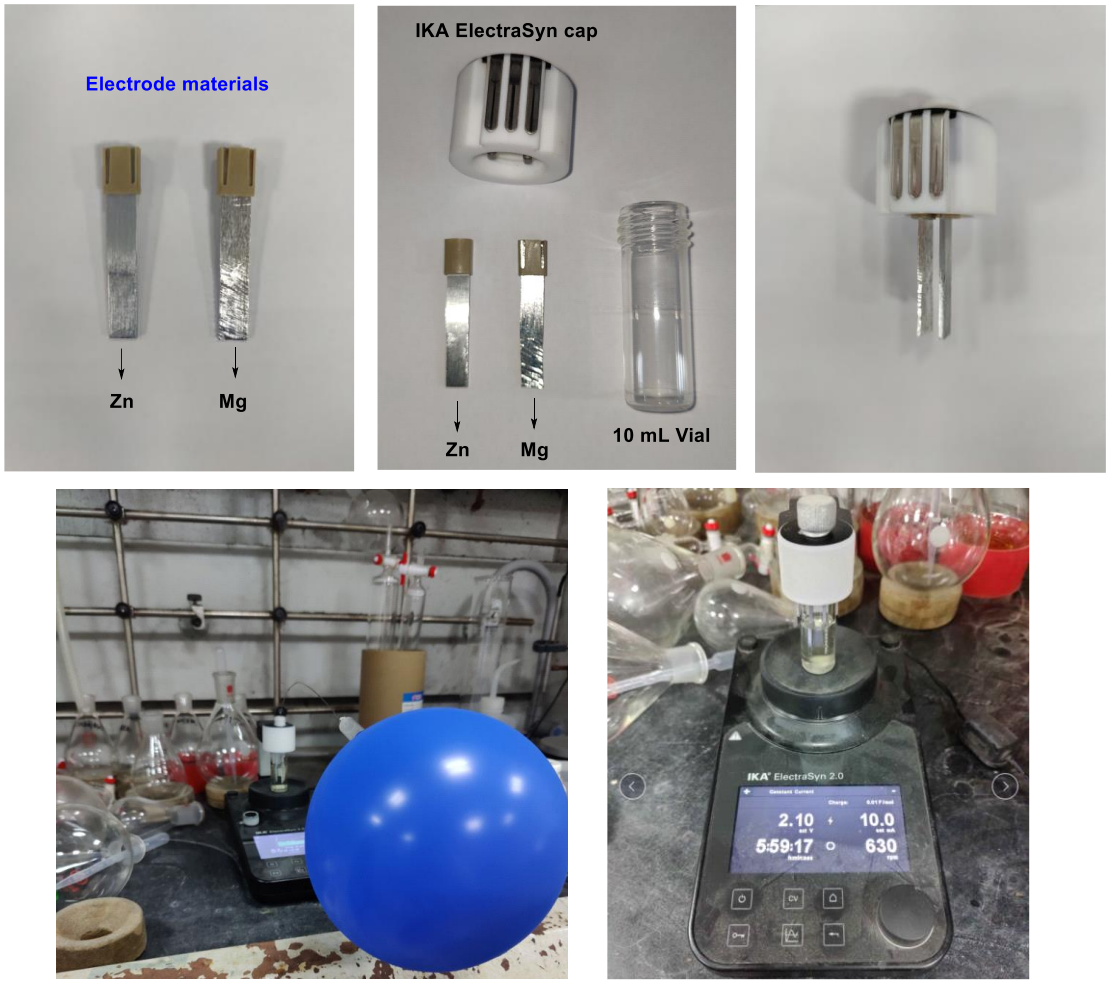
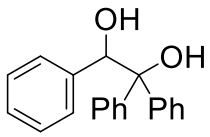


Figure S1 Experimental setup (IKA ElectraSyn 2.0)

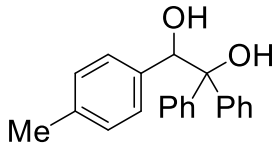
Physical data of the compounds

1,1,2-Triphenylethane-1,2-diol (3)[1]



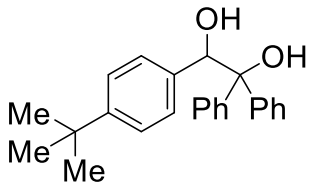
According to **GP1** with benzophenone (109.3 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **3** as white solid (130.6 mg, 75%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70-7.65 (m, 2H), 7.42-7.36 (m, 2H), 7.32-7.26 (m, 1H), 7.20-7.06 (m, 8H), 7.05-7.01 (m, 2H), 5.60 (d, *J* = 2.8 Hz, 1H), 3.15 (s, 1H), 2.46 (d, *J* = 3.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.1, 143.3, 138.8, 128.4, 128.1, 127.7, 127.6, 127.4, 127.3, 127.0, 126.7, 126.2, 80.7, 77.9; **HRMS** (ESI) calculated for $C_{20}H_{18}O_2Na$ $[M+Na]^+$ m/z 313.1199, found 313.1200.

1,1-Diphenyl-2-(*p*-tolyl)ethane-1,2-diol (4)[1]



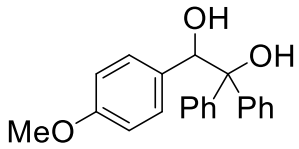
According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 4-methylbenzaldehyde (213 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **4** as white solid (150.8 mg, 83%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71-7.66 (m, 2H), 7.42-7.36 (m, 2H), 7.32-7.26 (m, 1H), 7.17-7.08 (m, 5H), 6.96-6.93 (m, 4H), 5.60 (s, 1H), 3.12 (s, 1H), 2.37 (s, 1H), 2.26 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.1, 143.5, 137.4, 135.8, 128.4, 128.2, 127.9, 127.6, 127.3, 126.9, 126.7, 126.2, 80.7, 77.8, 21.1; **HRMS** (ESI) calculated for $C_{21}H_{20}O_2Na$ $[M+Na]^+$ m/z 327.1356, found 327.1352.

2-(4-(*tert*-Butyl)phenyl)-1,1-diphenylethane-1,2-diol (5)[1]



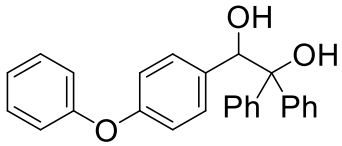
According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), 4-(*tert*-butyl)benzaldehyde (301 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **5** as white solid (155.2 mg, 75%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.71-7.64 (m, 2H), 7.43-7.34 (m, 2H), 7.32-7.25 (m, 1H), 7.19-7.05 (m, 7H), 7.00-6.94 (m, 2H), 5.58 (s, 1H), 3.14 (brs, 1H), 2.40 (brs, 1H), 1.25 (s, 9H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.6, 145.1, 143.5, 135.7, 128.3, 127.7, 127.5, 127.2, 127.0, 126.6, 126.2, 124.4, 80.7, 77.8, 34.4, 31.2; **HRMS** (ESI) calculated for $C_{24}H_{26}O_2Na$ $[M+Na]^+$ m/z 369.1825, found 369.1809.

2-(4-Methoxyphenyl)-1,1-diphenylethane-1,2-diol (6)[2]



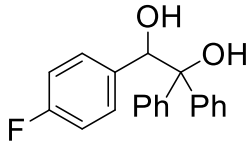
According to **GP1** with benzophenone (109.4 mg, 0.60 mmol, 1.0 equiv), 4-methoxybenzaldehyde (219 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (198.0 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **6** as white solid (153.1 mg, 80%). **^{1}H NMR** (300 MHz, DMSO-d_6) δ 7.61 (d, J = 7.5 Hz, 2H), 7.34-7.26 (m, 4H), 7.21-7.10 (m, 3H), 7.06-7.03 (m, 3H), 6.64 (d, J = 8.4 Hz, 2H), 5.56 (d, J = 4.2 Hz, 1H), 5.48 (d, J = 4.5 Hz, 1H), 5.48 (s, 1H), 3.65 (s, 3H); **^{13}C NMR** (75 MHz, DMSO-d_6) δ 157.9, 146.9, 146.2, 134.1, 129.8, 127.5, 127.2, 126.5, 126.0, 125.9, 111.9, 79.8, 76.2, 54.8; **HRMS** (ESI) calculated for $C_{21}H_{20}O_3Na$ $[M+Na]^+$ m/z 343.1305, found 343.1291.

2-(4-Phenoxyphenyl)-1,1-diphenylethane-1,2-diol (7)



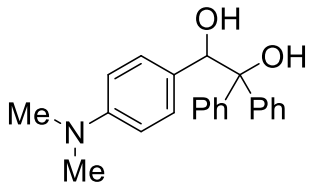
According to **GP1** with benzophenone (109.7 mg, 0.60 mmol, 1.0 equiv), 4-phenoxybenzaldehyde (315 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **7** as white solid (139.2 mg, 61%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72-7.63 (m, 2H), 7.40-7.37 (m, 2H), 7.31-7.27 (m, 3H), 7.12-7.05 (m, 6H), 7.03-6.96 (m, 2H), 6.95-6.89 (m, 2H), 6.82-6.74 (m, 2H), 5.57 (s, 1H), 3.17 (brs, 1H), 2.51 (brs, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.2, 156.5, 145.0, 143.3, 133.8, 129.6, 129.4, 128.4, 127.6, 127.4, 127.0, 126.7, 126.2, 123.1, 118.6, 118.0, 80.7, 77.5; **HRMS** (ESI) calculated for $C_{26}H_{22}O_3Na$ $[M+Na]^+$ m/z 405.1461, found 405.1464.

2-(4-Fluorophenyl)-1,1-diphenylethane-1,2-diol (8)[2]



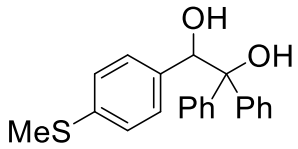
According to **GP1** with benzophenone (109.4 mg, 0.60 mmol, 1.0 equiv), 4-fluorobenzaldehyde (193 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **8** as white solid (116.1 mg, 63%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69-7.63 (m, 2H), 7.41-7.37 (m, 2H), 7.32-7.28 (m, 1H), 7.14-7.06 (m, 5H), 7.01-6.98 (m, 2H), 6.83-6.78 (m, 2H), 5.58 (s, 1H), 3.13 (brs, 1H), 2.49 (brs, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.2 (d, $J_{C\text{-}F}$ = 244.5 Hz), 144.9, 143.1, 134.5 (d, $J_{C\text{-}F}$ = 3.1 Hz), 129.7 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.5, 127.7, 127.5, 126.9, 126.8, 126.1, 114.2 (d, $J_{C\text{-}F}$ = 21.2 Hz), 80.7, 77.3; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114.66; **HRMS** (ESI) calculated for $C_{20}H_{17}FO_2Na$ $[M+Na]^+$ m/z 331.1105, found 331.1080.

2-(4-(Dimethylamino)phenyl)-1,1-diphenylethane-1,2-diol (9)



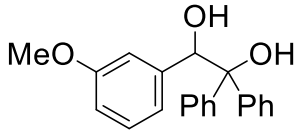
According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 4-(dimethylamino)benzaldehyde (268.5 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **9** as white solid (128.2 mg, 64%). **^{1}H NMR** (300 MHz, DMSO-d_6) δ 7.63-7.55 (m, 2H), 7.33-7.24 (m, 4H), 7.18-7.08 (m, 3H), 7.04-7.00 (m, 1H), 6.98-6.90 (m, 2H), 6.48-6.39 (m, 2H), 5.47 (brs, 1H), 5.34 (s, 1H), 5.28 (brs, 1H), 3.37 (s, 6H); **^{13}C NMR** (75 MHz, DMSO-d_6) δ 149.1, 147.2, 146.3, 129.7, 129.4, 127.3, 127.2, 126.5, 125.9, 125.7, 100.8, 79.9, 76.4, 40.2; **HRMS** (ESI) calculated for $C_{22}H_{23}NO_2Na$ $[M+Na]^+$ m/z 356.1621, found 356.1618.

2-(4-(Methylthio)phenyl)-1,1-diphenylethane-1,2-diol (10)[2]



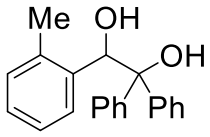
According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 4-(methylthio)benzaldehyde (239 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **10** as white solid (109.8 mg, 54%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.69-7.67 (m, 2H), 7.42-7.37 (m, 2H), 7.33-7.27 (m, 1H), 7.16-7.08 (m, 5H), 7.03-6.95 (m, 4H), 5.59 (s, 1H), 3.10 (s, 1H), 2.42 (s, 4H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.0, 143.2, 137.8, 135.6, 128.51, 128.48, 127.7, 127.4, 126.9, 126.8, 126.1, 125.4, 80.7, 77.6, 15.6; **HRMS** (ESI) calculated for $C_{21}H_{20}O_2SNa$ $[M+Na]^+$ m/z 359.1076, found 359.1073.

2-(3-Methoxyphenyl)-1,1-diphenylethane-1,2-diol (11)[2]



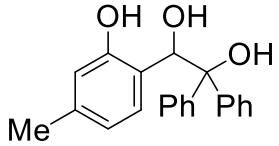
According to **GP1** with benzophenone (109.8 mg, 0.60 mmol, 1.0 equiv), 3-methoxybenzaldehyde (219 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **11** as white solid (112.6 mg, 59%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.70-7.67 (m, 2H), 7.42-7.37 (m, 2H), 7.32-7.29 (m, 1H), 7.19-7.04 (m, 6H), 6.74-6.66 (m, 2H), 6.55-6.49 (m, 1H), 5.60 (s, 1H), 3.59 (s, 3H), 3.10 (brs, 1H), 2.47 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 158.7, 149.9, 143.4, 140.3, 128.4, 127.7, 127.4, 127.0, 126.8, 126.2, 120.3, 113.8, 113.2, 80.7, 77.9, 55.1; **HRMS** (ESI) calculated for $C_{21}H_{20}O_3Na$ $[M+Na]^+$ m/z 343.1305, found 343.1305.

1,1-Diphenyl-2-(*o*-tolyl)ethane-1,2-diol (12)[2]



According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), 2-methylbenzaldehyde (208 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **12** as white solid (142.3 mg, 78%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.70-7.67 (m, 2H), 7.65-7.62 (m, 1H), 7.45-7.40 (m, 2H), 7.36-7.31 (m, 1H), 7.21-7.16 (m, 1H), 7.15-7.09 (m, 1H), 7.06-6.98 (m, 3H), 6.95-6.91 (m, 2H), 6.87 (d, J = 7.3 Hz, 1H), 5.81 (s, 1H), 3.37 (brs, 1H), 2.32 (brs, 1H), 1.70 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.3, 142.8, 137.2, 136.7, 129.6, 128.4, 128.2, 127.7, 127.43, 127.36, 127.19, 127.17, 126.9, 125.5, 80.9, 72.6, 19.1; **HRMS** (ESI) calculated for $C_{21}H_{20}O_2Na$ $[M+Na]^+$ m/z 327.1356, found 327.1339.

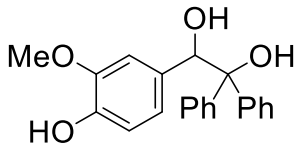
2-(2-Hydroxy-4-methylphenyl)-1,1-diphenylethane-1,2-diol (13)



According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 2-hydroxy-4-methylbenzaldehyde (245.2 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 5:1) to afford the desired

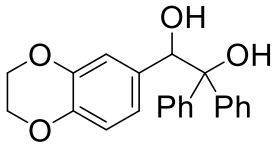
product **13** as white solid (107.6 mg, 56%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.50 (s, 1H), 7.59-7.57 (m, 2H), 7.38-7.34 (m, 2H), 7.31-7.27 (m, 1H), 7.24-7.21 (m, 2H), 7.16-7.07 (m, 3H), 6.57 (s, 1H), 6.45 (d, *J* = 7.6 Hz, 1H), 6.36 (dd, *J* = 7.6, 0.8 Hz, 1H), 5.64 (s, 1H), 3.86 (s, 1H), 3.01 (brs, 1H), 2.16 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.9, 143.8, 143.0, 139.5, 130.8, 128.5, 127.9, 127.7, 127.0, 126.9, 125.8, 120.8, 120.3, 118.2, 82.1, 79.3, 20.9; **HRMS** (ESI) calculated for $C_{21}H_{20}O_3Na$ $[M+Na]^+$ m/z 343.1305, found 343.1314.

2-(4-Hydroxy-3-methoxyphenyl)-1,1-diphenylethane-1,2-diol (14)



According to **GP1** with benzophenone (109.3 mg, 0.60 mmol, 1.0 equiv), 4-hydroxy-3-methoxybenzaldehyde (274.0 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3:1) to afford the desired product **14** as white solid (119.3 mg, 59%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.72-7.63 (m, 2H), 7.42-7.37 (m, 2H), 7.32-7.25 (m, 1H), 7.19-7.07 (m, 5H), 6.73-6.64 (m, 2H), 6.39 (s, 1H), 5.55 (s, 1H), 5.23 (brs, 1H), 3.58 (s, 3H), 3.08 (brs, 1H), 2.46 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.5, 145.1, 144.8, 143.6, 130.5, 128.4, 127.7, 127.3, 127.0, 126.7, 126.3, 120.9, 113.4, 110.7, 80.7, 77.8, 55.7; **HRMS** (ESI) calculated for $C_{21}H_{20}O_4Na$ $[M+Na]^+$ m/z 359.1254, found 359.1254.

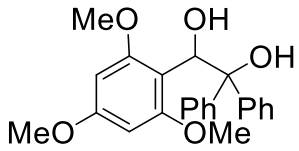
2-(2,3-Dihydrobenzo[*b*][1,4]dioxin-6-yl)-1,1-diphenylethane-1,2-diol (15)



According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 2,3-dihydrobenzo[*b*][1,4]dioxine-6-carbaldehyde (295.6 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **15** as white solid (153.8 mg, 74%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.71-7.62 (m, 2H), 7.40-7.35 (m, 2H), 7.32-7.26 (m, 1H), 7.19-7.07 (m, 5H), 6.70 (d, *J* = 1.8 Hz, 1H), 6.59 (d, *J* = 8.4 Hz, 1H), 6.45 (dd, *J* = 8.4, 1.8 Hz, 1H), 5.51 (s, 1H), 4.22-4.12 (m, 4H), 3.12 (brs, 1H), 2.41 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$)

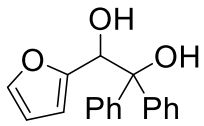
δ 145.1, 143.5, 143.0, 142.7, 132.1, 128.4, 127.6, 127.3, 126.9, 126.7, 126.1, 121.3, 117.1, 116.1, 80.6, 77.4, 64.3, 64.2; **HRMS** (ESI) calculated for $C_{22}H_{20}O_4Na$ $[M+Na]^+$ m/z 371.1254, found 371.1254.

1,1-Diphenyl-2-(2,4,6-trimethoxyphenyl)ethane-1,2-diol (16)



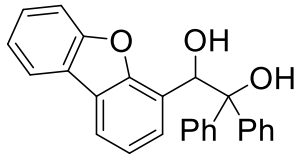
According to **GP1** with benzophenone (109.4 mg, 0.60 mmol, 1.0 equiv), 2,4,6-trimethoxybenzaldehyde (353.5 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 5:1) to afford the desired product **16** as white solid (144.3 mg, 63%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.52-7.48 (m, 2H), 7.42-7.37 (m, 2H), 7.33-7.28 (m, 2H), 7.25-7.08 (m, 4H), 6.11 (d, J = 9.6 Hz, 1H), 5.99 (s, 2H), 4.28 (d, J = 10.2 Hz, 1H), 4.09 (brs, 1H), 3.75 (s, 3H), 3.50 (s, 6H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 160.5, 158.6, 146.2, 144.7, 127.6, 127.4, 127.1, 127.0, 126.6, 126.4, 108.0, 90.8, 81.6, 72.0, 55.5, 55.2; **HRMS** (ESI) calculated for $C_{23}H_{24}O_5Na$ $[M+Na]^+$ m/z 403.1516, found 403.1535.

2-(Furan-2-yl)-1,1-diphenylethane-1,2-diol (17)[2]



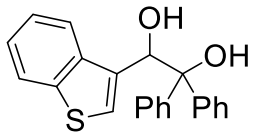
According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), furan-2-carbaldehyde (150 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **17** as white solid (100.2 mg, 60%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.67-7.58 (m, 2H), 7.38-7.10 (m, 9H), 6.21-6.19 (m, 1H), 6.12 (d, J = 3.2 Hz, 1H), 5.64 (s, 1H), 3.42 (s, 1H), 2.70 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 152.7, 144.2, 143.5, 141.9, 128.4, 127.9, 127.3, 126.9, 126.4, 125.6, 110.2, 109.1, 80.4, 72.3; **HRMS** (ESI) calculated for $C_{18}H_{16}O_3Na$ $[M+Na]^+$ m/z 303.0992, found 303.0984.

2-(Dibenzo[*b*,*d*]furan-4-yl)-1,1-diphenylethane-1,2-diol (18)



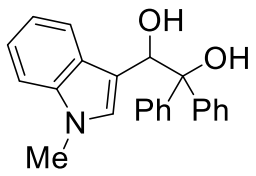
According to **GP1** with benzophenone (109.8 mg, 0.60 mmol, 1.0 equiv), dibenzo[*b*,*d*]furan-4-carbaldehyde (353.2 mg, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **18** as white solid (91.8 mg, 40%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.5 Hz, 1H), 7.77-7.70 (m, 3H), 7.42-7.38 (m, 5H), 7.32-7.23 (m, 2H), 7.20-7.16 (m, 3H), 6.97-6.83 (m, 3H), 6.26 (s, 1H), 3.52 (s, 1H), 2.86 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 155.7, 153.9, 144.9, 143.0, 128.4, 127.4, 127.2, 127.1, 127.0, 126.8, 126.7, 126.2, 123.8, 123.56, 123.53, 122.6, 122.3, 120.5, 120.0, 111.3, 81.1, 73.4; **HRMS** (ESI) calculated for $C_{26}H_{20}O_3Na$ $[M+Na]^+$ m/z 403.1305, found 403.1320.

2-(Benzo[*b*]thiophen-3-yl)-1,1-diphenylethane-1,2-diol (19)



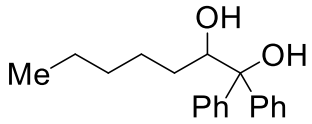
According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), benzo[*b*]thiophene-3-carbaldehyde (292.2 mg, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (198.1 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **19** as white solid (134.2 mg, 65%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76-7.69 (m, 3H), 7.60-7.53 (m, 1H), 7.43-7.39 (m, 3H), 7.33-7.29 (m, 1H), 7.24-7.18 (m, 4H), 7.04-6.97 (m, 3H), 6.04 (s, 1H), 3.41 (s, 1H), 2.42 (brs, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.1, 143.2, 139.5, 138.5, 133.9, 128.5, 127.7, 127.5, 126.85, 126.82, 126.3, 126.1, 124.0, 123.8, 122.4, 121.8, 80.7, 72.5; **HRMS** (ESI) calculated for $C_{22}H_{18}O_2SNa$ $[M+Na]^+$ m/z 369.0920, found 369.0920.

2-(1-Methyl-1*H*-indol-3-yl)-1,1-diphenylethane-1,2-diol (20)[3]



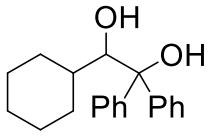
According to **GP1** with benzophenone (109.4 mg, 0.60 mmol, 1.0 equiv), 1-methyl-1*H*-indole-3-carbaldehyde (286.6 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 5:1) to afford the desired product **20** as white solid (135.8 mg, 66%). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.76-7.71 (m, 2H), 7.68 (d, J = 7.9 Hz, 1H), 7.47-7.42 (m, 2H), 7.30 (d, J = 7.7 Hz, 2H), 7.26 (d, J = 8.2 Hz, 1H), 7.20-7.16 (m, 1H), 7.10-7.03 (m, 4H), 6.97-6.91 (m, 2H), 5.98 (d, J = 5.4 Hz, 1H), 5.52 (s, 1H), 5.07 (d, J = 5.5 Hz, 1H), 3.62 (s, 3H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 147.9, 146.5, 135.7, 129.0, 127.9, 127.4, 127.3, 127.0, 126.0, 125.9, 125.6, 120.5, 119.9, 118.2, 114.8, 109.1, 80.3, 70.3, 32.3; **HRMS** (ESI) calculated for $C_{23}H_{21}NO_2Na$ $[M+Na]^+$ m/z 366.1465, found 366.1471.

1,1-Diphenylheptane-1,2-diol (21)



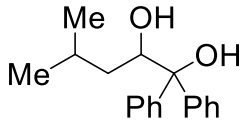
According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), hexanal (221 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 9:1) to afford the desired product **21** as white solid (152.8 mg, 90%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.64-7.56 (m, 2H), 7.45-7.41 (m, 2H), 7.37-7.16 (m, 6H), 4.57 (d, J = 9.6 Hz, 1H), 2.99 (s, 1H), 1.80 (brs, 1H), 1.56-1.43 (m, 2H), 1.36-1.18 (m, 6H), 0.85 (t, J = 6.6 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.8, 143.8, 128.6, 128.2, 127.2, 126.7, 126.1, 125.5, 80.1, 75.7, 31.7, 30.2, 26.2, 22.6, 14.0; **HRMS** (ESI) calculated for $C_{19}H_{24}O_2Na$ $[M+Na]^+$ m/z 307.1669, found 307.1660.

2-Cyclohexyl-1,1-diphenylethane-1,2-diol (22)[1]



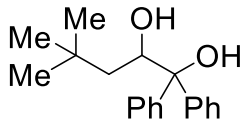
According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), cyclohexanecarbaldehyde (218 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10:1) to afford the desired product **22** as white solid (170.2 mg, 96%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.64-7.55 (m, 2H), 7.47-7.39 (m, 2H), 7.35-7.16 (m, 6H), 4.40 (d, *J* = 1.8 Hz, 1H), 3.07 (brs, 1H), 1.99 (d, *J* = 11.7 Hz, 1H), 1.85 (brs, 1H), 1.67-1.53 (m, 3H), 1.45-1.22 (m, 3H), 1.19-0.97 (m, 4H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.9, 144.1, 128.5, 128.1, 127.0, 126.6, 126.2, 125.5, 81.0, 78.7, 38.6, 32.3, 26.7, 26.6, 26.2, 26.1; **HRMS** (ESI) calculated for $C_{20}H_{24}O_2Na$ $[M+Na]^+$ m/z 319.1669, found 319.1641.

4-Methyl-1,1-diphenylpentane-1,2-diol (23)[1]



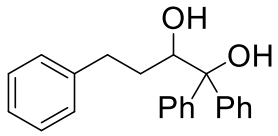
According to **GP1** with benzophenone (109.8 mg, 0.60 mmol, 1.0 equiv), 3-methylbutanal (193 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 9:1) to afford the desired product **23** as white solid (150.6 mg, 93%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63-7.57 (m, 2H), 7.46-7.40 (m, 2H), 7.36-7.16 (m, 6H), 4.67 (d, *J* = 10.4 Hz, 1H), 3.01 (brs, 1H), 1.83-1.73 (m, 2H), 1.50 (ddd, *J* = 14.2, 10.3, 3.9 Hz, 1H), 1.06 (ddd, *J* = 14.2, 10.2, 1.6 Hz, 1H), 0.89 (d, *J* = 6.8 Hz, 3H), 0.88 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.8, 143.8, 128.6, 128.2, 127.1, 126.7, 126.1, 125.5, 80.2, 73.7, 39.1, 24.8, 23.8, 21.4; **HRMS** (ESI) calculated for $C_{18}H_{22}O_2Na$ $[M+Na]^+$ m/z 293.1512, found 293.1510.

4,4-Dimethyl-1,1-diphenylpentane-1,2-diol (24)



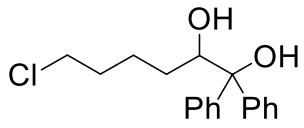
According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 3,3-dimethylbutanal (226 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 9:1) to afford the desired product **24** as white solid (159.8 mg, 94%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.67-7.57 (m, 2H), 7.50-7.42 (m, 2H), 7.37-7.15 (m, 6H), 4.71 (dd, *J* = 6.3, 3.4 Hz, 1H), 3.04 (brs, 1H), 1.72 (brs, 1H), 1.41-1.33 (m, 2H), 0.89 (s, 9H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.7, 128.6, 128.1, 127.1, 126.7, 126.0, 125.6, 81.0, 73.5, 43.7, 30.0; **HRMS** (ESI) calculated for $C_{19}H_{24}O_2Na$ $[M+Na]^+$ m/z 307.1669, found 307.1656.

1,1,4-Triphenylbutane-1,2-diol (25)



According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 3-phenylpropanal (237 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.5 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **25** as white solid (133.2 mg, 70%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.58-7.50 (m, 2H), 7.36-7.17 (m, 11H), 7.15-7.08 (m, 2H), 4.56 (d, *J* = 9.6 Hz, 1H), 3.00 (brs, 1H), 2.92-2.83 (m, 1H), 2.68-2.58 (m, 1H), 1.89-1.76 (m, 1H), 1.73-1.62 (m, 1H), 1.56 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.6, 143.5, 141.8, 128.7, 128.5, 128.3, 128.2, 127.2, 126.8, 126.0, 125.8, 125.5, 80.1, 74.8, 32.4, 31.7; **HRMS** (ESI) calculated for $C_{22}H_{22}O_2Na$ $[M+Na]^+$ m/z 341.1512, found 341.1495.

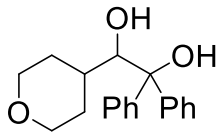
6-Chloro-1,1-diphenylhexane-1,2-diol (26)



According to **GP1** with benzophenone (109.3 mg, 0.60 mmol, 1.0 equiv),

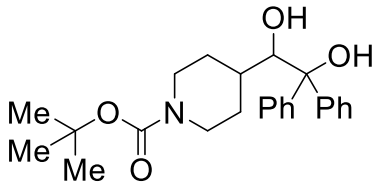
5-chloropentanal (216 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **26** as white solid (121.3 mg, 66%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.61-7.53 (m, 2H), 7.45-7.38 (m, 2H), 7.36-7.15 (m, 6H), 4.53 (d, *J* = 8.4 Hz, 1H), 3.45 (t, *J* = 6.4 Hz, 2H), 3.09 (brs, 1H), 1.96 (brs, 1H), 1.76-1.62 (m, 2H), 1.52-1.40 (m, 2H), 1.35-1.26 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.5, 143.6, 128.6, 128.2, 127.2, 126.8, 126.0, 125.4, 80.1, 75.6, 44.9, 32.4, 29.4, 23.9; **HRMS** (ESI) calculated for $C_{18}H_{21}ClO_2Na$ $[M+Na]^+$ m/z 327.1122, found 327.1124.

1,1-Diphenyl-2-(tetrahydro-2*H*-pyran-4-yl)ethane-1,2-diol (27)



According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), tetrahydro-2*H*-pyran-4-carbaldehyde (187 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3:1) to afford the desired product **27** as white solid (114.6 mg, 64%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.62-7.55 (m, 2H), 7.46-7.40 (m, 2H), 7.36-7.18 (m, 6H), 4.43-4.38 (m, 1H), 3.88-3.80 (m, 2H), 3.21-3.12 (m, 2H), 3.12 (s, 1H), 2.05 (d, *J* = 4.7 Hz, 1H), 1.88-1.78 (m, 1H), 1.71-1.55 (m, 3H), 1.15-1.07 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.5, 143.8, 128.6, 128.2, 127.2, 126.8, 126.0, 125.4, 80.8, 77.8, 67.9, 67.8, 36.0, 31.4, 27.3; **HRMS** (ESI) calculated for $C_{19}H_{22}NaO_3$ $[M+Na]^+$ m/z 321.1461, found 321.1461.

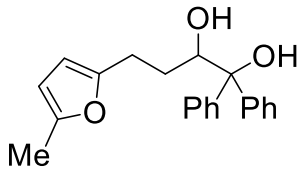
***tert*-Butyl 4-(1,2-dihydroxy-2,2-diphenylethyl)piperidine-1-carboxylate (28)**



According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), *tert*-butyl 4-formylpiperidine-1-carboxylate (383.9 mg, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 5:1) to afford the desired product **28** as white solid (154.8 mg, 65%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.62-7.56

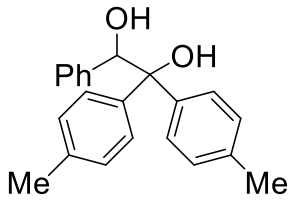
(m, 2H), 7.46-7.41 (m, 2H), 7.36-7.18 (m, 6H), 4.44 (d, *J* = 4.4 Hz, 1H), 4.01 (brs, 2H), 3.13 (s, 1H), 2.60-2.30 (m, 2H), 2.07 (d, *J* = 5.0 Hz, 1H), 1.96 (d, *J* = 12.7 Hz, 1H), 1.57-1.36 (m, 3H), 1.42 (s, 9H), 1.29-1.21 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.7, 146.5, 143.8, 128.6, 128.2, 127.2, 126.8, 126.0, 125.3, 80.9, 79.2, 77.7, 37.1, 30.8, 28.4, 26.2; **HRMS** (ESI) calculated for $C_{24}H_{31}NNaO_4$ $[M+Na]^+$ m/z 420.2145, found 420.2145.

4-(5-Methylfuran-2-yl)-1,1-diphenylbutane-1,2-diol (29)



According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), 3-(5-methylfuran-2-yl)propanal (240 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **29** as white solid (120.6 mg, 62%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59-7.53 (m, 2H), 7.40-7.16 (m, 8H), 5.86-5.79 (m, 2H), 4.60 (d, *J* = 9.9 Hz, 1H), 3.02 (s, 1H), 2.81-2.74 (m, 1H), 2.69-2.61 (m, 1H), 2.24 (s, 3H), 1.93 (d, *J* = 3.2 Hz, 1H), 1.83-1.65 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.7, 150.4, 145.6, 143.5, 128.6, 128.2, 127.2, 126.8, 126.0, 125.5, 105.82, 105.79, 80.1, 74.8, 28.8, 24.7, 13.5; **HRMS** (ESI) calculated for $C_{21}H_{22}NaO_3$ $[M+Na]^+$ m/z 345.1461, found 345.1461.

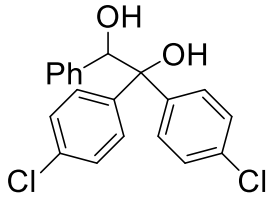
2-Phenyl-1,1-di-p-tolylethane-1,2-diol (30)[4]



According to **GP1** with di-*p*-tolylmethanone (126.5 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **30** as white solid (87.6 mg, 46%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.59-7.52 (m, 2H), 7.21-7.11 (m, 5H), 7.08-7.04 (m, 2H), 7.03-6.97 (m, 2H), 6.94-6.87 (m, 2H), 5.57 (s, 1H), 3.03 (s, 1H), 2.43 (brs, 1H), 2.35 (s, 3H), 2.21 (s, 3H); **^{13}C NMR** (75 MHz,

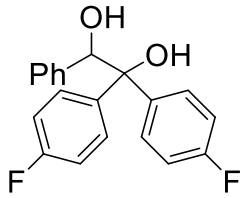
$CDCl_3$) δ 142.2, 140.6, 138.9, 136.9, 136.2, 129.1, 128.3, 128.1, 127.6, 127.4, 126.7, 126.0, 80.6, 78.0, 21.0, 20.9; **HRMS** (ESI) calculated for $C_{22}H_{22}O_2Na$ $[M+Na]^+$ m/z 341.1512, found 341.1521.

1,1-Bis(4-chlorophenyl)-2-phenylethane-1,2-diol (31)



According to **GP1** with bis(4-chlorophenyl)methanone (150.9 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and $n$$Bu_4NBF_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **31** as white solid (175.3 mg, 81%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.55-7.49 (m, 2H), 7.37-7.30 (m, 2H), 7.20-7.11 (m, 3H), 7.07-7.03 (m, 2H), 7.00-6.94 (m, 4H), 5.43 (s, 1H), 3.30 (brs, 1H), 2.51 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.4, 141.6, 138.4, 133.4, 132.7, 128.6, 128.5, 128.1, 128.0, 127.8, 127.7, 127.6, 80.0, 77.7; **HRMS** (ESI) calculated for $C_{20}H_{16}Cl_2O_2Na$ $[M+Na]^+$ m/z 381.0420, found 381.0419.

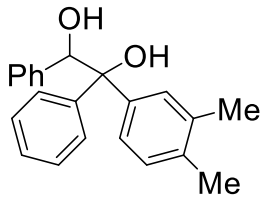
1,1-Bis(4-fluorophenyl)-2-phenylethane-1,2-diol (32)[4]



According to **GP1** with bis(4-fluorophenyl)methanone (131.2 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and $n$$Bu_4NBF_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **32** as white solid (124.7 mg, 64%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61-7.56 (m, 2H), 7.21-7.12 (m, 3H), 7.09-6.98 (m, 6H), 6.80-6.74 (m, 2H), 5.48 (s, 1H), 3.25 (s, 1H), 2.49 (brs, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.0 (d, $J_{C\text{-}F}$ = 245.2 Hz), 161.6 (d, $J_{C\text{-}F}$ = 244.5 Hz), 140.8 (d, $J_{C\text{-}F}$ = 3.3 Hz), 139.1 (d, $J_{C\text{-}F}$ = 3.0 Hz), 138.6, 129.0 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.1 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.0, 127.6, 115.1 (d, $J_{C\text{-}F}$ = 21.1 Hz), 114.4 (d, $J_{C\text{-}F}$ = 21.2 Hz), 80.0, 78.0; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -115.01 – -115.09 (m), -115.77 – -115.84 (m); **HRMS** (ESI) calculated for $C_{20}H_{16}F_2O_2Na$ $[M+Na]^+$ m/z

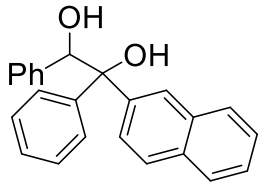
349.1011, found 349.1010.

1-(3,4-Dimethylphenyl)-1,2-diphenylethane-1,2-diol (33)



According to **GP1** with (3,4-dimethylphenyl)(phenyl)methanone (126.2 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **33** as white solid (106.4 mg, 56%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.68-7.63 (m, 1H), 7.44-7.35 (m, 2H), 7.30-7.26 (m, 0.5H), 7.18-7.02 (m, 8H), 6.89-6.86 (m, 1.5H), 5.58 (s, 0.5H), 5.57 (s, 0.5H), 3.10 (brs, 0.5H), 3.02 (brs, 0.5H), 4.44 (brs, 1H), 2.27 (s, 1.5H), 2.26 (s, 1.5H), 2.13 (s, 1.5H), 2.08 (s, 1.5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 145.0, 143.4, 142.5, 140.8, 138.9, 138.7, 136.8, 135.8, 135.7, 135.0, 130.0, 128.9, 128.3, 128.2, 128.13, 128.06, 127.58, 127.56, 127.5, 127.41, 127.38, 127.36, 127.2, 126.9, 126.6, 126.0, 124.0, 123.6, 80.63, 80.62, 78.0, 77.9, 20.1, 19.8, 19.4, 19.3; **HRMS** (ESI) calculated for $C_{22}H_{22}O_2Na$ $[M+Na]^+$ m/z 341.1512, found 341.1527.

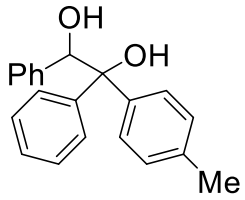
1-(Naphthalen-2-yl)-1,2-diphenylethane-1,2-diol (34)[2]



According to **GP1** with naphthalen-2-yl(phenyl)methanone (139.6, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **34** as white solid (168.7 mg, 83%). The diastereomeric ratio is 1.3:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 8.19 (s, 0.56H), 7.90-7.81 (m, 2H), 7.71-7.04 (m, 14.56H), 5.713 (s, 0.56H), 5.705 (s, 0.44H), 3.30 (s, 0.56H), 3.27 (s, 0.44H), 2.56 (d, J = 3.2 Hz, 0.56H), 2.53 (d, J = 3.3 Hz, 0.44H); **^{13}C NMR** (100 MHz,

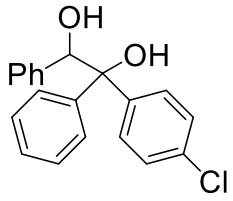
$CDCl_3$) δ two isomers: 144.9, 143.2, 142.4, 140.8, 138.8, 138.7, 133.0, 132.7, 132.5, 132.1, 128.5, 128.4, 128.23, 128.22, 128.1, 128.0, 127.72, 127.71, 127.6, 127.50, 127.46, 127.4, 127.3, 127.2, 127.0, 126.7, 126.3, 126.20, 126.18, 125.8, 125.3, 125.0, 124.5, 80.9, 77.9, 77.7; **HRMS** (ESI) calculated for $C_{24}H_{20}O_2Na$ $[M+Na]^+$ m/z 363.1356, found 363.1371.

1,2-Diphenyl-1-(p-tolyl)ethane-1,2-diol (35)[2]



According to **GP1** with phenyl(*p*-tolyl)methanone (117.9 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and $n$$Bu_4NBF_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **35** as white solid (103.2 mg, 57%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.67-7.61 (m, 1H), 7.57-7.51 (m, 1H), 7.39-7.35 (m, 1H), 7.29-7.26 (m, 0.5H), 7.22-6.98 (m, 9.5H), 6.94-6.87 (m, 1H), 5.55 (s, 0.5H), 5.54 (s, 0.5H), 3.14 (brs, 0.5H), 3.10 (brs, 0.5H), 2.52 (brs, 0.5H), 2.50 (brs, 0.5H), 2.35 (s, 1.5H), 2.21 (s, 1.5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 145.1, 143.4, 142.1, 140.4, 138.9, 138.8, 137.0, 136.2, 129.1, 128.35, 128.31, 128.1, 128.0, 127.6, 127.5, 127.40, 127.36, 127.2, 126.9, 126.8, 126.6, 126.1, 126.0, 80.64, 80.62, 77.92, 77.91, 21.0, 20.9; **HRMS** (ESI) calculated for $C_{21}H_{20}O_2Na$ $[M+H]^+$ m/z 327.1356, found 327.1369.

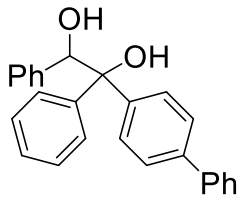
1-(4-Chlorophenyl)-1,2-diphenylethane-1,2-diol (36)



According to **GP1** with (4-chlorophenyl)(phenyl)methanone (130.5 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and $n$$Bu_4NBF_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **36** as white solid (138.3 mg, 71%). The diastereomeric ratio is 1.4:1 determined by ^{1}H

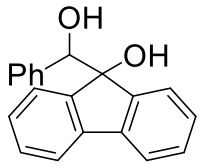
NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.67-7.59 (m, 1H), 7.58-7.53 (m, 1H), 7.41-7.37 (m, 1H), 7.34-7.30 (m, 1.42H), 7.19-6.98 (m, 9.58H), 5.51 (d, *J* = 2.9 Hz, 0.42H), 5.50 (d, *J* = 3.1 Hz, 0.58H), 3.27 (brs, 0.42H), 3.19 (brs, 0.58H), 2.51 (brs, 0.42H), 2.50 (brs, 0.58H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 144.8, 143.7, 143.0, 141.9, 138.7, 138.5, 133.1, 132.5, 128.7, 128.5, 128.3, 128.1, 128.0, 127.9, 127.8, 127.69, 127.66, 127.57, 127.55, 127.52, 126.9, 126.1, 80.40, 80.36, 77.8, 77.7; **HRMS** (ESI) calculated for $C_{20}H_{17}ClO_2Na$ $[M+Na]^+$ m/z 347.0809, found 347.0809.

1-([1,1'-Biphenyl]-4-yl)-1,2-diphenylethane-1,2-diol (37)[2]



According to **GP1** with [1,1'-biphenyl]-4-yl(phenyl)methanone (155.4 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **37** as white solid (193.8 mg, 88%). The diastereomeric ratio is 1.3:1 determined by ^{1}H NMR. **^{1}H NMR** (300 MHz, $CDCl_3$) δ two isomers: 7.77-7.71 (m, 2H), 7.64-7.59 (m, 2H), 7.51-7.26 (m, 6H), 7.29-7.26 (m, 9H), 5.65 (s, 0.56H), 5.64 (s, 0.44H), 3.20 (brs, 0.56H), 3.18 (brs, 0.44H), 2.48 (d, *J* = 3.1 Hz, 0.56H), 2.45 (d, *J* = 3.1 Hz, 0.54H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ two isomers: 144.9, 144.1, 143.2, 142.4, 140.6, 140.1, 139.4, 138.8, 138.7, 128.8, 128.7, 128.5, 128.09, 128.06, 127.75, 127.66, 127.50, 127.49, 127.4, 127.3, 127.2, 127.13, 127.07, 126.96, 126.92, 126.8, 126.6, 126.3, 126.1, 80.69, 80.66, 78.01, 77.97; **HRMS** (ESI) calculated for $C_{26}H_{22}O_2Na$ $[M+Na]^+$ m/z 389.1512, found 389.1510.

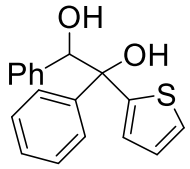
9-(Hydroxy(phenyl)methyl)-9H-fluoren-9-ol (38)[5]



According to **GP1** with 9*H*-fluoren-9-one (108.6 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column

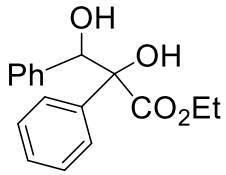
chromatography (petroleum ether/EtOAc = 5:1) to afford the desired product **38** as white solid (141.9 mg, 82%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.66-7.63 (m, 1H), 7.58-7.54 (m, 1H), 7.39-7.35 (m, 2H), 7.20-7.21 (m, 4H), 7.02-6.96 (m, 1H), 6.92-6.87 (m, 2H), 6.75-6.72 (m, 2H), 5.24 (s, 1H), 3.26 (brs, 1H), 2.96 (brs, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.8, 144.7, 140.2, 140.1, 137.5, 129.3, 129.1, 127.4, 127.31, 127.30, 127.2, 126.8, 125.7, 124.2, 119.8, 119.6, 84.8, 79.4; **HRMS** (ESI) calculated for $C_{20}H_{16}O_2Na$ $[M+Na]^+$ m/z 311.1043, found 311.1053.

1,2-Diphenyl-1-(thiophen-2-yl)ethane-1,2-diol (39)



According to **GP1** with phenyl(thiophen-2-yl)methanone (113.6 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **39** as white solid (103.8 mg, 58%). The diastereomeric ratio is 1.3:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.71-7.63 (m, 1H), 7.41-7.37 (m, 1H), 7.34-7.28 (m, 1H), 7.25-7.11 (m, 7H), 7.03-7.01 (m, 1H), 6.81 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.71 (dd, *J* = 3.6, 1.2 Hz, 1H), 5.45 (s, 0.38H), 5.45 (s, 0.50H), 3.54 (s, 0.50H), 3.23 (s, 0.38H), 2.68 (s, 0.50H), 2.48 (s, 0.38H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 150.4, 148.3, 143.5, 142.0, 138.2, 138.0, 128.3, 128.0, 127.94, 127.88, 127.78, 127.76, 127.6, 127.4, 127.1, 126.7, 126.37, 126.35, 125.9, 125.6, 125.0, 124.9, 124.6, 80.2, 79.9, 79.8, 79.7; **HRMS** (ESI) calculated for $C_{18}H_{16}O_2SNa$ $[M+Na]^+$ m/z 319.0763, found 319.0777.

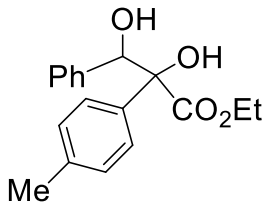
Ethyl 2,3-dihydroxy-2,3-diphenylpropanoate (40)



According to **GP2** with ethyl 2-oxo-2-phenylacetate (96 μL, 0.60 mmol, 1.0 equiv), benzaldehyde (122 μL, 1.20 mmol, 2.0 equiv), and *n*Bu_4NBF_4 (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column

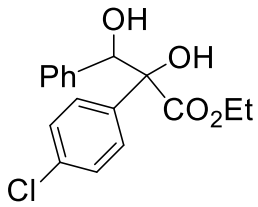
chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **40** as white solid (138.5 mg, 81%). The diastereomeric ratio is 1.2:1 determined by ^{1}H NMR. **^{1}H NMR** (300 MHz, $CDCl_3$) δ two isomers: 7.84-7.76 (m, 1H), 7.52-7.31 (m, 4.5H), 7.23-7.09 (m, 4.5H), 5.40 (brs, 1H), 4.37 (q, *J* = 7.1 Hz, 1H), 4.07 (qd, *J* = 7.2, 1.3 Hz, 0.45H), 4.04 (brs, 1H), 3.74 (brs, 0.45H), 2.94 (brs, 0.55H), 2.70 (brs, 0.45H), 1.37 (t, *J* = 7.1 Hz, 1.65H), 1.19 (t, *J* = 7.2 Hz, 1.35H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ two isomers: 174.0, 172.5, 138.4, 138.3, 138.1, 137.9, 128.4, 128.3, 127.99, 127.98, 127.89, 127.81, 127.80, 127.7, 127.5, 126.5, 125.8, 81.7, 81.0, 78.2, 77.9, 63.1, 62.7, 14.1, 13.9; **HRMS** (ESI) calculated for $C_{17}H_{18}O_4Na$ $[M+Na]^+$ m/z 309.1097, found 309.1097.

Ethyl 2,3-dihydroxy-3-phenyl-2-(p-tolyl)propanoate (41)



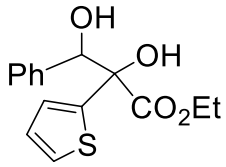
According to **GP2** with ethyl 2-oxo-2-(*p*-tolyl)acetate (105 μL, 0.60 mmol, 1.0 equiv), benzaldehyde (122 μL, 1.20 mmol, 2.0 equiv), and *n*Bu_4NBF_4 (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **41** as white solid (153.8 mg, 85%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.71-7.66 (m, 1H), 7.43-7.11 (m, 7H), 7.06-7.00 (m, 1H), 5.39 (d, *J* = 4.5 Hz, 0.5H), 5.37 (d, *J* = 2.7 Hz, 0.5H), 4.35 (qd, *J* = 7.1, 1.4 Hz, 1H), 4.06 (qd, *J* = 7.1, 1.4 Hz, 1H), 3.99 (brs, 0.5H), 3.69 (brs, 0.5H), 2.91 (d, *J* = 8.4 Hz, 0.5H), 2.65 (d, *J* = 6.7 Hz, 0.5H), 2.37 (brs, 1.5H), 2.27 (brs, 1.5H), 1.36 (t, *J* = 7.1 Hz, 1.5H), 1.19 (t, *J* = 7.1 Hz, 1.5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 174.2, 172.7, 138.5, 138.3, 138.0, 137.6, 135.4, 135.0, 129.1, 128.7, 128.3, 128.0, 127.9, 127.8, 127.6, 127.5, 126.4, 125.7, 81.7, 80.9, 78.1, 77.8, 63.0, 62.6, 21.1, 21.0, 14.1, 13.9; **HRMS** (ESI) calculated for $C_{18}H_{20}NaO_4$ $[M+Na]^+$ m/z 323.1254, found 323.1254.

Ethyl 2-(4-chlorophenyl)-2,3-dihydroxy-3-phenylpropanoate (42)



According to **GP2** with ethyl 2-(4-chlorophenyl)-2-oxoacetate (127.8 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (122 μL, 1.20 mmol, 2.0 equiv), and *n*Bu_4NBF_4 (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **42** as white solid (117.3 mg, 61%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.76-7.70 (m, 1H), 7.46-7.32 (m, 5H), 7.21-7.09 (m, 3H), 5.37-5.32 (m, 1H), 4.44-4.33 (m, 1H), 4.14-4.03 (m, 1H), 4.07 (brs, 0.5H), 3.75 (brs, 0.5H), 2.83 (d, *J* = 8.1 Hz, 0.5H), 2.68 (d, *J* = 7.2 Hz, 0.5H), 1.38 (t, *J* = 7.1 Hz, 1.5H), 1.19 (t, *J* = 7.2 Hz, 1.5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 173.7, 172.2, 138.2, 137.8, 137.0, 136.5, 134.3, 133.9, 128.5, 128.4, 128.13, 128.06, 128.0, 127.9, 127.8, 127.7, 127.3, 81.4, 80.7, 78.2, 77.9, 63.3, 62.9, 14.1, 13.8; **HRMS** (ESI) calculated for $C_{17}H_{17}ClNaO_4$ $[M+Na]^+$ m/z 343.0708, found 343.0708.

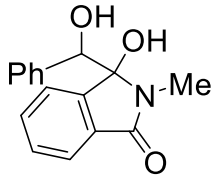
Ethyl 2,3-dihydroxy-3-phenyl-2-(thiophen-2-yl)propanoate (43)



According to **GP2** with ethyl 2-oxo-2-(thiophen-2-yl)acetate (89 μL, 0.60 mmol, 1.0 equiv), benzaldehyde (122 μL, 1.20 mmol, 2.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **43** as white solid (115.8 mg, 66%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (300 MHz, $CDCl_3$) δ two isomers: 7.40-7.28 (m, 4H), 7.21-7.16 (m, 2H), 7.12 (dd, *J* = 5.1, 1.1 Hz, 0.5H), 7.08-7.03 (m, 1H), 6.89 (dd, *J* = 5.0, 3.7 Hz, 0.5H), 5.24 (s, 0.5H), 5.22 (s, 0.5H), 4.41 (q, *J* = 7.1 Hz, 1H), 4.28 (brs, 0.5H), 4.18-4.06 (m, 1H), 4.04 (brs, 0.5H), 2.80 (brs, 1H), 1.41 (t, *J* = 7.1, 1.5H), 1.22 (t, *J* = 7.1, 1.5H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ two isomers: 173.0, 171.6, 142.9, 142.4, 137.9, 137.8,

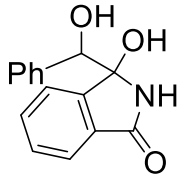
128.5, 128.0, 127.7, 127.61, 127.60, 127.2, 126.8, 125.9, 125.5, 125.4, 124.8, 80.6, 80.4, 79.2, 79.1, 63.4, 63.0, 14.0, 13.8; **HRMS** (ESI) calculated for $C_{15}H_{16}NaO_4S$ $[M+H]^+$ m/z 315.0662, found 315.0662.

3-Hydroxy-3-(hydroxy(phenyl)methyl)-2-methylisoindolin-1-one (44)[6]



According to **GP1** with 2-methylisoindoline-1,3-dione (96.9 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 1:2) to afford the desired product **44** as white solid (48.8 mg, 30%). The diastereomeric ratio is 1.7:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, DMSO-d_6) δ two isomers: 7.90-7.84 (m, 0.37H), 7.58 (td, *J* = 7.4, 1.4 Hz, 0.37H), 7.55-7.50 (m, 0.63H), 7.45-7.32 (m, 2.16H), 7.27-7.22 (m, 3.15H), 7.07-7.00 (m, 1.08H), 6.93-6.91 (m, 0.72H), 6.71 (s, 0.37H), 6.61-6.59 (m, 1.24H), 5.80 (d, *J* = 4.0 Hz, 0.37H), 5.67 (d, *J* = 4.7 Hz, 0.63H), 5.05 (d, *J* = 4.0 Hz, 0.37H), 5.03 (d, *J* = 4.7 Hz, 0.63H), 3.02 (s, 1.89H), 2.89 (s, 1.11H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ two isomers: 166.4, 166.2, 145.7, 145.0, 140.5, 139.7, 132.9, 132.1, 131.0, 130.3, 128.91, 128.85, 127.9, 127.2, 127.1, 126.97, 126.96, 126.89, 124.6, 123.7, 121.6, 121.5, 91.4, 91.0, 75.3, 73.4, 24.6, 24.2; **HRMS** (ESI) calculated for $C_{16}H_{16}NO_3$ $[M+H]^+$ m/z 270.1125, found 270.1149.

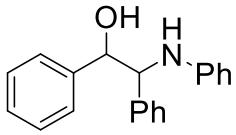
3-Hydroxy-3-(hydroxy(phenyl)methyl)isoindolin-1-one (45)[7]



According to **GP1** with isoindoline-1,3-dione (88.6 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (EtOAc) to afford the desired product **45** as white solid (86.5 mg, 56%). The diastereomeric ratio is 1.5:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, DMSO-d_6) δ two isomers: 8.65 (brs, 0.4H), 8.62 (brs, 0.6H), 7.74-7.68 (m, 0.6H),

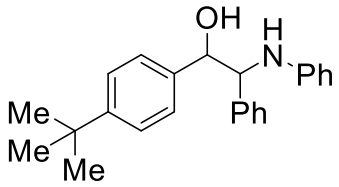
7.57-7.53 (m, 0.6H), 7.47-7.43 (m, 0.4H), 7.40-7.33 (m, 2.2H), 7.21-7.07 (m, 5.2H), 6.50 (brs, 0.6H), 6.43 (brs, 0.4H), 5.68 (d, *J* = 3.9 Hz, 0.6H), 5.62 (d, *J* = 4.2 Hz, 0.4H), 4.99 (d, *J* = 3.9 Hz, 0.4H), 4.93 (d, *J* = 3.6 Hz, 0.6H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ two isomers: 168.2, 167.8, 147.00, 146.98, 140.4, 140.1, 132.7, 132.5, 131.2, 131.0, 128.8, 128.7, 128.0, 127.8, 126.94, 126.91, 126.8, 124.3, 123.6, 122.0, 121.8, 89.4, 88.9, 76.7, 76.1; **HRMS** (ESI) calculated for $C_{15}H_{14}NO_3$ $[M+H]^+$ m/z 256.0968, found 256.0985.

1,2-Diphenyl-2-(phenylamino)ethan-1-ol (46)[2]



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.9 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10:1) to afford the desired product **46** as white solid (121.3 mg, 70%). The diastereomeric ratio is 2.3:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.27-7.20 (m, 7.5H), 7.14-7.03 (m, 4.5H), 6.66-6.62 (m, 1H), 6.53-6.49 (m, 2H), 5.04 (s, 0.7H), 4.86 (d, *J* = 5.8 Hz, 0.3H), 4.65 (d, *J* = 4.8 Hz, 0.7H), 4.52 (d, *J* = 5.8 Hz, 0.3H), 5.04 (brs, 1H), 2.53 (brs, 0.3H), 2.31 (brs, 0.7H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 147.2, 146.7, 140.5, 140.2, 140.0, 138.4, 129.1, 129.0, 128.5, 128.25, 128.21, 128.20, 128.0, 127.9, 127.6, 127.5, 127.2, 126.52, 126.49, 117.9, 114.1, 113.9, 78.0, 77.1, 64.7, 63.7; **HRMS** (ESI) calculated for $C_{20}H_{20}NO$ $[M+H]^+$ m/z 290.1539, found 290.1538.

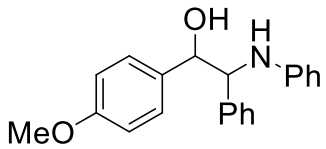
1-(4-(*tert*-Butyl)phenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (47)[2]



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.8 mg, 0.60 mmol, 1.0 equiv), 4-(*tert*-butyl)benzaldehyde (301 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **47** as colorless oil (105.8 mg, 51%). The diastereomeric ratio is 1.3:1

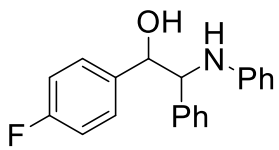
determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.29-7.14 (m, 8H), 7.04-6.99 (m, 3H), 6.62-6.59 (m, 1H), 6.49-6.45 (m, 2H), 4.91 (d, *J* = 5.0 Hz, 0.44H), 4.79 (d, *J* = 5.2 Hz, 0.56H), 4.59 (d, *J* = 5.1 Hz, 0.44H), 4.55 (brs, 1H), 4.50 (d, *J* = 5.3 Hz, 0.56H), 2.54 (brs, 0.56H), 2.35 (brs, 0.44H), 1.29 (s, 4H), 1.28 (s, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 150.9, 15.07, 147.2, 146.8, 140.5, 138.9, 137.6, 136.9, 129.0, 128.9, 128.4, 128.2, 127.8, 127.4, 127.3, 127.2, 126.3, 126.2, 125.09, 125.07, 117.7, 117.5, 113.90, 113.87, 77.6, 77.1, 64.1, 63.6, 34.5, 34.4, 31.3; **HRMS** (ESI) calculated for $C_{24}H_{28}NO$ $[M+H]^+$ m/z 346.2165, found 346.2179.

1-(4-Methoxyphenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (48)



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.7 mg, 0.60 mmol, 1.0 equiv), 4-methoxybenzaldehyde (219 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **48** as colorless oil (126.8 mg, 66%). The diastereomeric ratio is 1.5:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.24-7.12 (m, 6H), 7.07-6.98 (m, 3H), 6.81-6.76 (m, 2H), 6.66-6.61 (m, 1H), 6.53-6.47 (m, 2H), 4.95 (d, *J* = 4.8 Hz, 0.4H), 4.77 (d, *J* = 6.1 Hz, 0.6H), 4.59 (d, *J* = 5.0 Hz, 0.4H), 4.53 (brs, 1H), 4.46 (d, *J* = 6.2 Hz, 0.6H), 3.77 (s, 1.2H), 3.75 (s, 1.8H), 2.58 (brs, 0.6H), 2.32 (brs, 0.4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 159.2, 159.1, 147.3, 146.8, 140.2, 138.7, 132.6, 131.9, 129.02, 128.99, 128.4, 128.2, 127.9, 127.8, 127.7, 127.5, 127.4, 127.3, 117.81, 117.78, 114.1, 113.9, 113.6, 113.5, 77.6, 76.8, 64.7, 63.6, 55.19, 55.17; **HRMS** (ESI) calculated for $C_{21}H_{22}NO_2$ $[M+H]^+$ m/z 320.1645, found 320.1645.

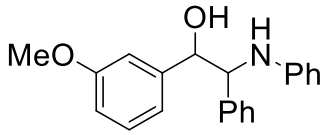
1-(4-Fluorophenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (49)



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (109.1 mg, 0.60 mmol, 1.0 equiv), 4-fluorobenzaldehyde (193 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel

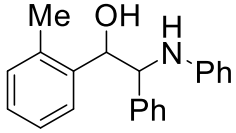
column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **49** as colorless oil (96.7 mg, 52%). The diastereomeric ratio is 1.2:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.25-7.14 (m, 5H), 7.11-6.99 (m, 4H), 6.95-6.91 (m, 2H), 6.67-6.63 (m, 1H), 6.54-6.50 (m, 2H), 5.02 (d, *J* = 4.4 Hz, 0.46H), 4.79 (d, *J* = 6.2 Hz, 0.54H), 4.61 (d, *J* = 6.2 Hz, 0.46H), 4.51 (brs, 1H), 4.44 (d, *J* = 6.2 Hz, 0.54H), 2.72 (brs, 0.54H), 2.40 (brs, 0.46H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 162.33 (d, $J_{C\text{-}F}$ = 244.8 Hz), 162.26 (d, $J_{C\text{-}F}$ = 244.6 Hz), 147.1, 146.6, 139.9, 138.1, 136.3 (d, $J_{C\text{-}F}$ = 3.1 Hz), 135.7 (d, $J_{C\text{-}F}$ = 3.1 Hz), 129.10, 129.07, 128.6, 128.29, 128.26, 128.2, 128.1 (d, $J_{C\text{-}F}$ = 8.4 Hz), 127.8, 127.6 (d, $J_{C\text{-}F}$ = 9.2 Hz), 127.2, 118.1, 118.0, 115.0 (d, $J_{C\text{-}F}$ = 21.3 Hz), 114.1 (d, $J_{C\text{-}F}$ = 27.6 Hz), 77.4, 76.4, 64.9, 63.6; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114.20, -114.34; **HRMS** (ESI) calculated for $C_{20}H_{19}FNO$ $[M+H]^+$ m/z 308.1445, found 308.1447.

1-(3-Methoxyphenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (50)[2]



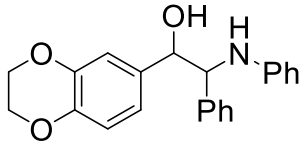
According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.9 mg, 0.60 mmol, 1.0 equiv), 3-methoxybenzaldehyde (219 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **50** as colorless oil (122.6 mg, 64%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.24-7.11 (m, 6H), 7.06-7.02 (m, 2H), 6.81-6.49 (m, 6H), 4.99 (d, *J* = 4.5 Hz, 0.5H), 4.78 (d, *J* = 5.8 Hz, 0.5H), 4.63 (d, *J* = 4.7 Hz, 0.5H), 4.55 (brs, 1H), 4.48 (d, *J* = 5.9 Hz, 0.5H), 3.68 (s, 1.5H), 3.62 (s, 1.5H), 2.73 (brs, 0.5H), 2.53 (brs, 0.5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 159.4, 159.3, 147.2, 146.7, 142.2, 141.6, 140.2, 138.5, 129.14, 129.11, 129.02, 128.96, 128.4, 128.1, 127.8, 127.45, 127.39, 127.2, 118.8, 118.7, 117.80, 117.78, 114.0, 113.9, 113.8, 113.4, 112.0, 111.7, 77.8, 76.9, 64.6, 63.5, 55.1, 55.0; **HRMS** (ESI) calculated for $C_{21}H_{22}NO_2$ $[M+H]^+$ m/z 320.1645, found 320.1648.

2-Phenyl-2-(phenylamino)-1-(o-tolyl)ethan-1-ol (51)[2]



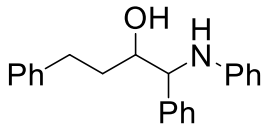
According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.8 mg, 0.60 mmol, 1.0 equiv), 2-methylbenzaldehyde (208 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **51** as colorless oil (125.6 mg, 69%). The diastereomeric ratio is 1.2:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.68-7.59 (m, 0.55H), 7.30-6.93 (m, 10.45H), 6.66-6.62 (m, 1H), 6.60-6.43 (m, 2H), 5.29 (d, *J* = 4.2 Hz, 0.45H), 5.07 (d, *J* = 6.2 Hz, 0.55H), 4.76 (brs, 1H), 4.62 (d, *J* = 4.8 Hz, 0.45H), 4.51 (d, *J* = 6.2 Hz, 0.55H), 2.41 (brs, 0.45H), 2.27 (s, 1.35H), 2.17 (brs, 0.55H), 1.98 (s, 1.65H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 147.4, 146.7, 140.2, 139.0, 138.4, 138.2, 135.5, 135.1, 130.4, 130.2, 129.1, 129.0, 128.5, 128.14, 128.09, 127.73, 127.70, 127.6, 127.5, 127.1, 126.32, 126.28, 126.15, 125.9, 117.80, 117.78, 114.1, 113.9, 74.1, 73.6, 63.8, 62.2, 19.2, 19.0; **HRMS** (ESI) calculated for $C_{21}H_{22}NO$ $[M+H]^+$ m/z 304.1696, found 304.1699.

1-(2,3-Dihydrobenzo[*b*][1,4]dioxin-6-yl)-2-phenyl-2-(phenylamino)ethan-1-ol (52)



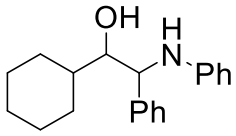
According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.8 mg, 0.60 mmol, 1.0 equiv), 2,3-dihydrobenzo[*b*][1,4]dioxine-6-carbaldehyde (295.9 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 6:1) to afford the desired product **52** as colorless oil (95.8 mg, 46%). The diastereomeric ratio is 1.5:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.28-7.17 (m, 5H), 7.06-7.02 (m, 2H), 6.83 (d, *J* = 1.9 Hz, 0.6H), 6.76-6.73 (m, 1H), 6.68 (dd, *J* = 8.9, 1.9 Hz, 1H), 6.64-6.61 (m, 1H), 6.54 (dd, *J* = 8.3, 2.0 Hz, 0.4H), 6.52-6.47 (m, 2H), 4.85 (d, *J* = 5.1 Hz, 0.4H), 4.74 (d, *J* = 5.6 Hz, 0.6H), 4.56 (d, *J* = 5.3 Hz, 0.4H), 4.52 (brs, 1H), 4.46 (d, *J* = 5.6 Hz, 0.6H), 4.20 (s, 1.6H), 4.19 (s, 2.4H), 2.51 (brs, 0.6H), 2.27 (brs, 0.4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 147.2, 146.8, 143.34, 143.25, 143.1, 140.3, 138.9, 134.0, 133.3, 129.00, 128.98, 128.5, 128.3, 127.8, 127.6, 127.4, 127.2, 119.6, 119.5, 117.8, 117.7, 116.93, 116.89, 115.5, 115.4, 114.0, 113.9, 77.4, 76.8, 64.5, 64.3, 64.2, 63.6; **HRMS** (ESI) calculated for $C_{22}H_{22}NO_3$ $[M+H]^+$ m/z 348.1594, found 348.1603.

1,4-Diphenyl-1-(phenylamino)butan-2-ol (53)



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.9 mg, 0.60 mmol, 1.0 equiv), 3-phenylpropanal (237 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **53** as colorless oil (99.7 mg, 52%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.32-7.23 (m, 7.5H), 7.19-7.04 (m, 5H), 6.66-6.51 (m, 2.5H), 4.58 (brs, 1H), 4.39 (d, *J* = 3.9 Hz, 0.5H), 4.32 (d, *J* = 4.5 Hz, 0.5H), 3.96 (d, *J* = 9.0 Hz, 0.5H), 3.83 (dt, *J* = 8.5, 4.3 Hz, 0.5H), 2.88-2.79 (m, 1H), 2.72-2.63 (m, 1H), 2.07-1.77 (m, 2H), 1.61-1.52 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 147.2, 146.9, 141.6, 141.5, 141.0, 138.8, 129.14, 129.10, 128.8, 128.6, 128.43, 128.39, 127.7, 127.6, 127.5, 126.9, 126.0, 125.9, 117.7, 117.6, 113.7, 113.6, 75.1, 73.8, 62.5, 62.3, 35.4, 35.3, 32.2, 32.1; **HRMS** (ESI) calculated for $C_{22}H_{24}NO$ $[M+H]^+$ m/z 318.1852, found 318.1854.

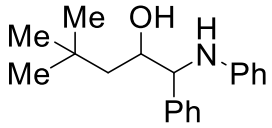
1-Cyclohexyl-2-phenyl-2-(phenylamino)ethan-1-ol (54)



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.7 mg, 0.60 mmol, 1.0 equiv), cyclohexanecarbaldehyde (218 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10:1) to afford the desired product **54** as colorless oil (101.3 mg, 57%). The diastereomeric ratio is 1.5:1 determined by ^{1}H NMR. **^{1}H NMR** (300 MHz, $CDCl_3$) δ two isomers: 7.44-7.25 (m, 5H), 7.15-7.09 (m, 2H), 6.69-6.64 (m, 1H), 6.60-6.55 (m, 2H), 4.73 (brs, 1H), 4.62 (d, *J* = 3.9 Hz, 0.4H), 4.57 (d, *J* = 3.4 Hz, 0.6H), 3.73-6.63 (m, 0.4H), 3.56 (dd, *J* = 7.3, 3.3 Hz, 0.6H), 2.10-1.61 (m, 7H), 1.28-1.06 (m, 5H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ two isomers: 147.1, 146.8, 141.6, 139.1, 129.11, 129.09, 128.8, 128.6, 128.0, 127.5, 127.2, 126.9, 117.3, 117.1, 113.31, 113.26, 80.5, 78.8, 58.6, 58.3, 40.0, 39.5, 30.0, 29.4, 29.0, 28.3, 26.4, 26.3, 26.0, 25.9, 25.8, 25.7; **HRMS** (ESI) calculated for

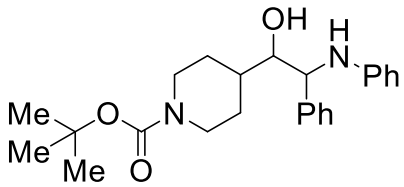
$C_{20}H_{26}NO$ $[M+H]^+$ m/z 296.2009, found 296.2009.

4,4-Dimethyl-1-phenyl-1-(phenylamino)pentan-2-ol (55)



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.8 mg, 0.60 mmol, 1.0 equiv), 3,3-dimethylbutanal (226 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 9:1) to afford the desired product **55** as colorless oil (109.8 mg, 65%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.34-7.21 (m, 5H), 7.09-7.05 (m, 2H), 6.65-6.61 (m, 1H), 6.56-6.51 (m, 2H), 4.58 (brs, 1H), 4.31 (d, *J* = 4.0 Hz, 0.5H), 4.22 (d, *J* = 4.1 Hz, 0.5H), 3.96 (d, *J* = 6.0 Hz, 0.5H), 3.96-3.93 (m, 0.5H), 1.62-1.46 (m, 2H), 1.11-1.00 (m, 1H), 0.93 (s, 4.5H), 0.89 (s, 4.5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 147.3, 147.0, 141.4, 139.1, 129.09, 129.07, 128.7, 128.5, 127.7, 127.41, 127.39, 126.9, 117.5, 117.4, 113.6, 113.5, 73.4, 72.1, 64.0, 63.5, 47.6, 47.2, 30.2, 30.1, 29.93, 29.90; **HRMS** (ESI) calculated for $C_{19}H_{26}NO$ $[M+H]^+$ m/z 284.2009, found 284.2015.

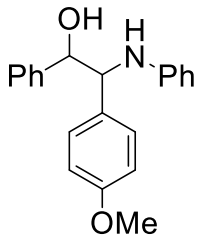
***tert*-Butyl 4-(1-hydroxy-2-phenyl-2-(phenylamino)ethyl)piperidine-1-carboxylate (56)**



According to **GP3** with (*E*)-*N*,1-diphenylmethanimine (108.8 mg, 0.60 mmol, 1.0 equiv), *tert*-butyl 4-formylpiperidine-1-carboxylate (384.5 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3:1) to afford the desired product **56** as white soild (171.1 mg, 72%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.39-7.24 (m, 5H), 7.10-7.06 (m, 2H), 6.66-6.62 (m, 1H), 6.55-6.51 (m, 2H), 4.64 (brs, 1H), 4.53-4.51 (m, 1H), 4.12 (brs, 2H), 3.69-3.65 (m, 0.5H), 3.53 (d, *J* = 7.3 Hz, 0.5H), 2.63 (brs, 2H), 2.01-1.96 (m, 1H), 1.81-1.73 (m, 2H), 1.67-1.61 (m, 1H), 1.450

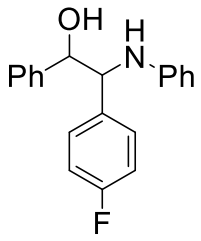
(s, 4.5H), 1.445 (s, 4.5H), 1.41-1.19 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 154.7, 146.8, 146.6, 141.1, 138.8, 129.2, 129.1, 128.9, 128.7, 127.9, 127.8, 127.4, 126.8, 117.6, 117.3, 113.3, 79.7, 79.4, 79.3, 77.8, 58.9, 58.0, 38.5, 38.2, 28.9, 28.414, 28.407, 27.5; **HRMS** (ESI) calculated for $C_{24}H_{32}N_2O_3Na$ $[M+H]^+$ m/z 419.2305, found 419.2346.

2-(4-Methoxyphenyl)-1-phenyl-2-(phenylamino)ethan-1-ol (57)[2]



According to **GP3** with (*E*)-1-(4-methoxyphenyl)-*N*-phenylmethanimine (127.4 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **57** as colorless oil (126.5 mg, 66%). The diastereomeric ratio is 1.3:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.25-7.21 (m, 4H), 7.08-7.00 (m, 5H), 6.79-6.71 (m, 2H), 6.65-6.61 (m, 1H), 6.52-6.48 (m, 2H), 4.98 (d, J = 3.4 Hz, 0.43H), 4.77 (d, J = 6.0 Hz, 0.57H), 4.58 (d, J = 4.1 Hz, 0.43H), 4.51 (brs, 1H), 4.44 (d, J = 5.9 Hz, 0.57H), 3.72 (s, 1.3H), 3.71 (s, 1.7H), 2.68 (brs, 0.57H), 2.38 (brs, 0.43H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 158.9, 158.8, 147.3, 146.8, 140.6, 140.1, 132.0, 130.2, 129.02, 128.97, 128.89, 128.3, 128.1, 127.85, 127.76, 126.6, 126.5, 117.82, 117.76, 114.2, 113.9, 113.8, 113.6, 78.0, 77.1, 64.1, 63.0, 55.1; **HRMS** (ESI) calculated for $C_{21}H_{22}NO_2$ $[M+H]^+$ m/z 320.1645, found 320.1645.

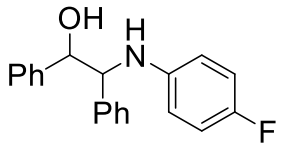
2-(4-Fluorophenyl)-1-phenyl-2-(phenylamino)ethan-1-ol (58)[2]



According to **GP3** with (*E*)-1-(4-fluorophenyl)-*N*-phenylmethanimine (119.9 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash

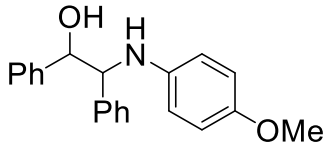
silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **58** as colorless oil (96.1 mg, 52%). The diastereomeric ratio is 1.1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.26-7.23 (m, 3H), 7.17-7.15 (m, 1H), 7.11-7.00 (m, 5H), 6.90-6.85 (m, 2H), 6.67-6.63 (m, 1H), 6.50-6.47 (m, 2H), 5.01 (d, *J* = 4.3 Hz, 0.48H), 4.71 (d, *J* = 6.5 Hz, 0.52H), 4.59 (d, *J* = 4.5 Hz, 0.48H), 4.54 (brs, 1H), 4.44 (d, *J* = 6.5 Hz, 0.52H), 2.68 (brs, 0.52H), 2.42 (brs, 0.48H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 162.1 (d, $J_{C\text{-}F}$ = 244.0 Hz), 162.0 (d, $J_{C\text{-}F}$ = 244.1 Hz), 147.1. 146.5, 140.3, 139.8, 135.7 (d, $J_{C\text{-}F}$ = 3.0 Hz), 133.9 (d, $J_{C\text{-}F}$ = 3.1 Hz), 129.4 (d, $J_{C\text{-}F}$ = 8.0 Hz), 129.1, 129.0, 128.8 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.23, 128.20, 128.0, 126.6, 126.4, 118.0, 117.9, 115.3 (d, $J_{C\text{-}F}$ = 21.4 Hz), 115.0 (d, $J_{C\text{-}F}$ = 21.3 Hz), 114.1, 113.8, 78.1, 76.9, 64.1, 62.8; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114.98, -114.98; **HRMS** (ESI) calculated for $C_{20}H_{19}FNO$ $[M+H]^+$ m/z 308.1445, found 308.1449.

2-((4-Fluorophenyl)amino)-1,2-diphenylethan-1-ol (59)



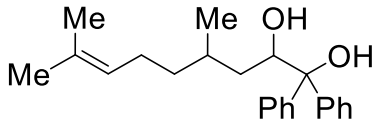
According to **GP3** with (*E*)-*N*-(4-fluorophenyl)-1-phenylmethanimine (119.8 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **59** as colorless oil (132.6 mg, 72%). The diastereomeric ratio is 1.1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.28-7.19 (m, 7H), 7.17-7.15 (m, 1H), 7.10-7.05 (m, 2H), 6.77-6.71 (m, 2H), 6.45-6.40 (m, 2H), 5.01 (d, *J* = 4.8 Hz, 0.47H), 4.80 (d, *J* = 6.2 Hz, 0.53H), 4.57 (d, *J* = 4.8 Hz, 0.47H), 4.41 (d, *J* = 6.2 Hz, 0.53H), 4.34 (brs, 1H), 2.66 (brs, 0.53H), 2.40 (brs, 0.47H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 156.1 (d, $J_{C\text{-}F}$ = 234.2 Hz), 156.0 (d, $J_{C\text{-}F}$ = 234.1 Hz), 143.6 (d, $J_{C\text{-}F}$ = 1.8 Hz), 143.1 (d, $J_{C\text{-}F}$ = 1.8 Hz), 140.5, 140.0, 139.9, 138.3, 128.6, 128.4, 128.3, 128.1, 128.0, 127.9, 127.7, 127.6, 127.4, 126.64, 126.57, 115.7 (d, $J_{C\text{-}F}$ = 5.9 Hz), 115.4 (d, $J_{C\text{-}F}$ = 5.9 Hz), 115.1 (d, $J_{C\text{-}F}$ = 7.4 Hz), 114.9 (d, $J_{C\text{-}F}$ = 7.4 Hz), 78.1, 77.2, 65.6, 64.3; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -127.331, -127.334; **HRMS** (ESI) calculated for $C_{20}H_{19}FNO$ $[M+H]^+$ m/z 308.1445, found 308.1446.

2-((4-Methoxyphenyl)amino)-1,2-diphenylethan-1-ol (60)[8]



According to **GP3** with (*E*)-*N*-(4-methoxyphenyl)-1-phenylmethanimine (126.9 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **60** as colorless oil (115.8 mg, 60%). The diastereomeric ratio is 2.3:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.25-7.05 (m, 10H), 6.68-6.60 (m, 2H), 6.54-6.43 (m, 2H), 5.00 (d, J = 4.5 Hz, 0.30H), 4.78 (d, J = 6.5 Hz, 0.70H), 4.58 (d, J = 4.7 Hz, 0.30H), 4.39 (d, J = 6.5 Hz, 0.70H), 4.30 (brs, 1H), 3.65 (s, 3H), 2.88 (brs, 0.70H), 2.5 (brs, 0.3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 152.6, 152.5, 141.3, 140.8, 140.6, 140.2, 140.1, 138.8, 128.5, 128.3, 128.21, 128.19, 128.0, 127.91, 127.86, 127.6, 127.5, 127.4, 126.7, 126.6, 115.8, 115.5, 114.71, 114.69, 78.1, 77.2, 66.2, 64.7, 55.69, 55.68; **HRMS** (ESI) calculated for $C_{21}H_{22}NO_2$ $[M+H]^+$ m/z 320.1645, found 320.1645.

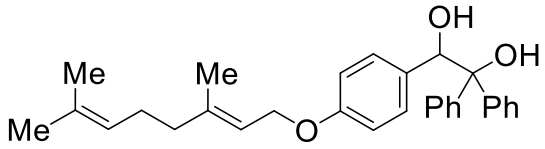
4,8-Dimethyl-1,1-diphenylnon-7-ene-1,2-diol (61)



According to **GP1** with benzophenone (109.7 mg, 0.60 mmol, 1.0 equiv), 3,7-dimethyloct-6-enal (324 μL, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 9:1) to afford the desired product **61** as white solid (189.7 mg, 93%). The diastereomeric ratio is 1:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.62-7.56 (m, 2H), 7.45-7.39 (m, 2H), 7.34-7.15 (m, 6H), 5.07 (q, J = 7.1 Hz, 1H), 4.67 (d, J = 9.6 Hz, 1H), 3.05 (brs, 1H), 1.97-1.81 (m, 3H), 1.68 (s, 1.5H), 1.65 (s, 1.5H), 1.60-1.53 (m, 1H), 1.58 (s, 1.5H), 1.57 (s, 1.5H), 1.49-1.28 (m, 1H), 1.26-0.97 (m, 3H), 0.88 (d, J = 6.6 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 145.9, 145.8, 143.8, 143.8, 131.2, 131.1, 128.6, 128.5, 128.1, 127.1, 126.68, 126.67, 126.10, 126.09, 125.52, 125.21, 124.8, 124.7, 80.25, 80.18, 73.8, 73.4, 38.0, 37.5, 37.2, 35.6, 29.6, 29.0, 25.71, 25.68, 25.5,

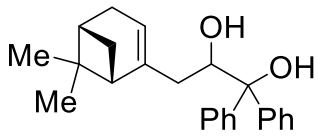
25.2, 20.6, 18.8, 17.7, 17.6; **HRMS** (ESI) calculated for $C_{23}H_{30}O_2Na$ $[M+Na]^+$ m/z 361.2138, found 361.2138.

(*E*)-2-(4-((3,7-Dimethylocta-2,6-dien-1-yl)oxy)phenyl)-1,1-diphenylethane-1,2-diol (62)



According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), (*E*)-4-((3,7-dimethylocta-2,6-dien-1-yl)oxy)benzaldehyde (465.5 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **62** as white solid (146.3 mg, 55%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.71-7.61 (m, 2H), 7.41-7.36 (m, 2H), 7.31-7.26 (m, 1H), 7.16-7.05 (m, 5H), 7.01-6.91 (m, 2H), 6.72-6.64 (m, 2H), 5.56 (d, *J* = 3.0 Hz, 1H), 5.44 (t, *J* = 6.1 Hz, 1H), 5.09 (t, *J* = 5.9 Hz, 1H), 4.45 (d, *J* = 6.5 Hz, 2H), 3.14 (s, 1H), 2.39 (d, *J* = 3.1 Hz, 1H), 2.13-2.03 (m, 4H), 1.70 (s, 3H), 1.68 (s, 3H), 1.60 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 158.3, 145.1, 143.5, 141.0, 131.8, 130.8, 129.1, 128.4, 127.6, 127.3, 126.9, 126.6, 126.1, 123.8, 119.4, 113.7, 80.7, 77.5, 64.7, 39.5, 26.3, 25.7, 17.7, 16.6; **HRMS** (ESI) calculated for $C_{30}H_{34}O_3Na$ $[M+Na]^+$ m/z 465.2400, found 465.2416.

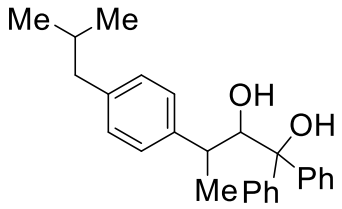
3-((1*S*,5*R*)-6,6-Dimethylbicyclo[3.1.1]hept-2-en-2-yl)-1,1-diphenylpropane-1,2-diol (63)



According to **GP1** with benzophenone (109.5 mg, 0.60 mmol, 1.0 equiv), 2-((1*S*,5*R*)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)acetaldehyde (295.8 mg, 1.80 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **63** as white solid (97.8 mg, 47%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.65-7.61 (m, 2H), 7.41-7.32 (m, 4H), 7.26-7.20 (m, 3H), 7.14-7.08 (m, 1H), 5.33 (dd, *J* = 9.1, 2.6 Hz, 1H), 5.23 (dt, *J* = 9.1, 2.1 Hz, 1H), 3.20 (s, 1H), 2.55-2.22 (m, 4H), 1.92-1.65 (m, 4H), 1.34-1.26 (m, 1H), 1.10 (s, 3H), 0.14 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.2, 145.8, 143.8, 128.3, 127.9, 127.1, 126.6,

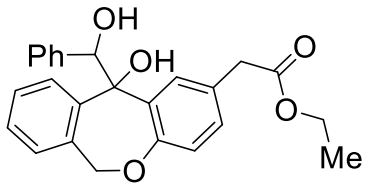
126.5, 125.8, 118.9, 79.7, 71.5, 52.6, 40.5, 40.4, 27.3, 25.8, 23.5, 21.5, 19.7; **HRMS** (ESI) calculated for $C_{24}H_{28}O_2Na$ $[M+Na]^+$ m/z 371.1982, found 371.1996.

3-(4-Isobutylphenyl)-1,1-diphenylbutane-1,2-diol (64)



According to **GP1** with benzophenone (109.6 mg, 0.60 mmol, 1.0 equiv), 2-(4-isobutylphenyl)propanal (342.7 mg, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.7 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **64** as colorless oil (101.3 mg, 45%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57-7.50 (m, 2H), 7.49-7.44 (m, 2H), 7.31-7.24 (m, 4H), 7.20-7.15 (m, 2H), 7.04-6.99 (m, 4H), 4.69 (dd, *J* = 4.0, 2.5 Hz, 1H), 3.07 (s, 1H), 2.86-2.81 (m, 1H), 2.42 (d, *J* = 7.2 Hz, 2H), 1.97 (d, *J* = 4.2 Hz, 1H), 1.88-1.78 (m, 1H), 1.30 (d, *J* = 7.1 Hz, 3H), 0.89 (d, *J* = 6.6 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.5, 144.1, 143.7, 139.6, 129.2, 128.3, 128.2, 127.2, 127.0, 126.7, 126.2, 125.6, 80.9, 78.5, 45.0, 39.6, 30.2, 22.4, 14.5; **HRMS** (ESI) calculated for $C_{26}H_{30}O_2Na$ $[M+Na]^+$ m/z 397.2138, found 397.2157.

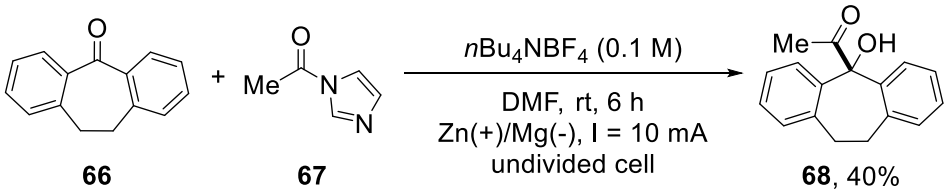
Ethyl 2-(11-hydroxy-11-(hydroxy(phenyl)methyl)-6,11-dihydrodibenzo[b,e]oxepin-2-yl) acetate (65)



According to **GP1** with ethyl 2-(11-oxo-6,11-dihydrodibenzo[*b*,*e*]oxepin-2-yl)acetate (178.5 mg, 0.60 mmol, 1.0 equiv), benzaldehyde (183 μL, 1.80 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.60 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 5:1) to afford the desired product **65** as white solid (128.6 mg, 53%). The diastereomeric ratio is 1.8:1 determined by ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.88 (dd, *J* = 7.7, 1.3 Hz, 0.36H), 7.66 (d, *J* = 2.1 Hz, 0.64H), 7.31-6.84 (m, 11H), 5.95-5.94 (m,

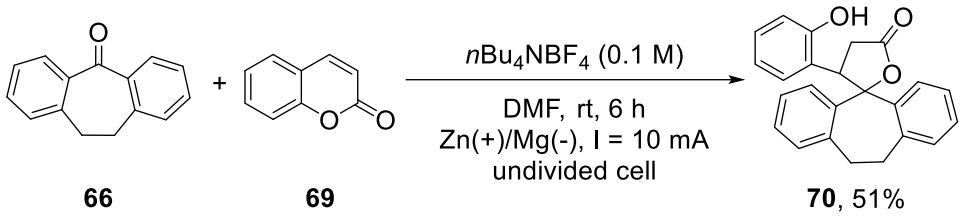
1H), 5.55 (d, *J* = 15.6 Hz, 0.36H), 5.46 (d, *J* = 15.6 Hz, 0.64H), 5.09 (d, *J* = 15.6 Hz, 0.36H), 5.04 (d, *J* = 15.6 Hz, 0.64H), 4.11 (q, *J* = 7.1 Hz, 1.28H), 3.99 (q, *J* = 7.1 Hz, 0.72H), 3.78 (s, 0.64H), 3.71 (s, 0.36H), 3.58 (s, 1.28H), 3.33 (d, *J* = 3.0 Hz, 0.72H), 2.69 (d, *J* = 3.5 Hz, 0.64H), 2.52 (d, *J* = 3.9 Hz, 0.36H), 1.24 (t, *J* = 7.2 Hz, 1.92H), 1.13 (t, *J* = 7.1 Hz, 1.08H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 171.8, 171.4, 154.7, 153.7, 140.5, 138.8, 138.6, 138.5, 137.60, 137.56, 135.0, 134.8, 130.4, 130.3, 130.2, 129.7, 128.4, 127.7, 127.5, 127.4, 127.3, 127.2, 127.12, 127.07, 127.0, 126.74, 127.73, 126.4, 126.1, 125.0, 121.8, 121.1, 79.7, 79.3, 78.2, 77.6, 73.9, 73.4, 60.9, 60.6, 40.8, 14.13, 14.05; **HRMS** (ESI) calculated for $C_{25}H_{24}O_5Na$ $[M+Na]^+$ m/z 427.1516, found 427.1537.

Investigation of other electrophiles:



10,11-Dihydro-5*H*-dibenzo[*a*,*d*][7]annulen-5-one **66** (108 μL, 0.6 mmol, 1.0 equiv), 1-(1*H*-imidazol-1-yl)ethan-1-one **67** (198.5 mg, 1.8 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20:1) to afford product **68** (59.9 mg, 40%).

1-(5-Hydroxy-10,11-dihydro-5*H*-dibenzo[*a*,*d*][7]annulen-5-yl)ethan-1-one: white solid; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.43-7.40 (m, 2H), 7.23-7.18 (m, 4H), 7.17-7.13 (m, 2H), 4.84 (s, 1H), 3.23-3.04 (m, 4H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 207.0, 140.9, 138.2, 130.4, 128.5, 128.1, 126.6, 84.8, 35.4, 24.9; **HRMS** (ESI) calculated for $C_{17}H_{16}O_2Na$ $[M+Na]^+$ m/z 275.1043, found 275.1071.

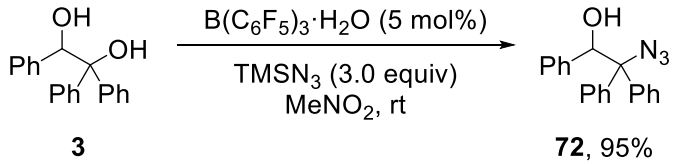


10,11-Dihydro-5*H*-dibenzo[*a*,*d*][7]annulen-5-one **66** (108 μL, 0.6 mmol, 1.0 equiv), 2*H*-chromen-2-one **69** (263.3 mg, 1.8 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3:1) to afford product **70** (109.2 mg, 51%).

3'-(2-Hydroxyphenyl)-3',4',10,11-tetrahydro-5'*H*-spiro[dibenzo[*a*,*d*][7]annulene-5,2'-furan]-5'-one:[9] white solid; **^{1}H NMR** (300 MHz, DMSO-d_6) δ 9.78 (brs, 1H), 7.68-7.54 (m, 1H), 7.47-7.38 (m, 1H), 7.29-7.20 (m, 3H), 7.05-6.98 (m, 2H), 6.93-6.85 (m, 3H), 6.73-6.62 (m, 1H), 6.52-6.47 (m, 1H), 4.77 (brs, 1H), 3.84-3.74 (m, 1H), 3.42-3.30 (m, 1H), 3.03-2.91 (m, 2H), 2.72-2.63 (m, 1H), 2.38 (d, J = 17.6 Hz, 1H); **^{13}C NMR** (75 MHz, DMSO-d_6) δ 176.2, 154.2, 141.5, 138.6, 137.0, 136.5, 132.0, 130.1, 128.2, 127.9, 127.5, 126.3, 126.1, 125.4, 125.0, 123.0, 118.7, 115.3, 91.5, 36.7, 31.6, 31.0; **HRMS** (ESI) calculated for $C_{24}H_{21}O_3$ $[M+H]^+$ m/z 357.1485, found 357.1505.

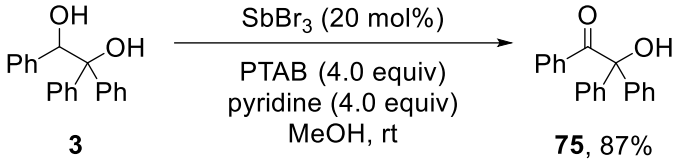
Derivatization of products:

2-Azido-1,2,2-triphenylethan-1-ol (72):[10]



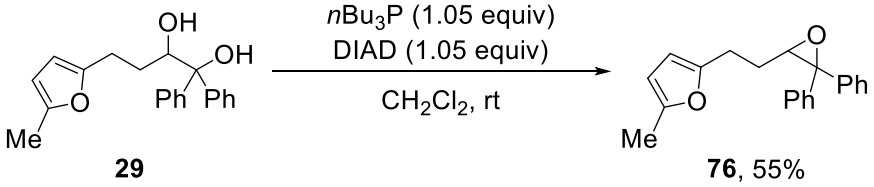
1,1,2-Triphenylethane-1,2-diol **3** (58.1 mg, 0.2 mmol, 1.0 equiv), $B(C_6F_5)_3$•H_2O (1.0 mg, 0.002 mmol, 0.01 equiv), and $TMSN_3$ (116 μL, 0.6 mmol, 3.0 equiv) were placed in a 10 mL Schlenk tube. Then $MeNO_2$ (1.0 mL) was added with a syringe, and the reaction mixure was stirred for 3.5 h at room temperature. After completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10:1) to afford product **72** as pale yellow solid (59.8 mg, 95%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51-7.48 (m, 2H), 7.41-7.33 (m, 3H), 7.22-7.11 (m, 6H), 7.07-7.05 (m, 2H), 6.96-6.94 (m, 2H), 5.68 (d, *J* = 3.0 Hz, 1H), 2.48 (d, *J* = 3.1 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.8, 140.1, 139.0, 128.7, 128.3, 127.93, 127.89, 127.84, 127.82, 127.5, 127.4, 79.0, 75.9; **HRMS** (ESI) calculated for $C_{20}H_{17}N_3ONa$ $[M+Na]^+$ m/z 338.1264, found 338.1292.

2-Hydroxy-1,2,2-triphenylethan-1-one (75):[11]



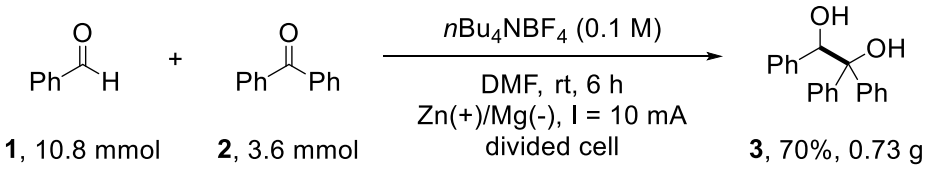
1,1,2-Triphenylethane-1,2-diol **3** (58.4 mg, 0.2 mmol, 1.0 equiv), $SbBr_3$ (14.6 mg, 0.04 mmol, 0.2 equiv), PTAB (300.9 mg, 0.8 mmol, 4.0 equiv), and pyridine (65 μL, 0.8 mmol, 4.0 equiv) were placed in a 10 mL Schlenk tube. Then MeOH (6.0 mL) was added with a syringe, and the reaction mixure was stirred for 48 h at room temperature. After completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford product **75** as white solid (50.5 mg, 87%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.73-7.70 (m, 2H), 7.46-7.39 (m, 5H), 7.36-7.24 (m, 8H), 5.01 (s, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.7, 141.8, 135.0, 132.9, 130.8, 128.3, 128.2, 128.1, 128.0, 85.0; **HRMS** (ESI) calculated for $C_{20}H_{16}O_2Na$ $[M+Na]^+$ m/z 311.1043, found 311.1079.

2-(2-(3,3-Diphenyloxiran-2-yl)ethyl)-5-methylfuran (76):

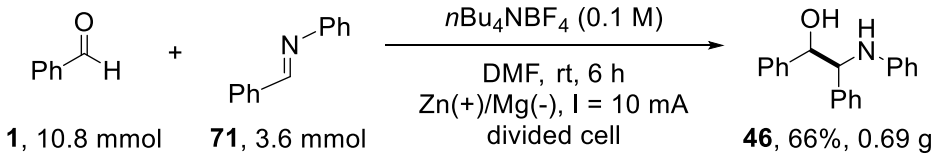


4-(5-Methylfuran-2-yl)-1,1-diphenylbutane-1,2-diol **29** (101.9 mg, 0.3 mmol, 1.0 equiv), *n*Bu_3P (80 μL, 0.315 mmol, 1.05 equiv), and DIAD (65 μL, 0.315 mmol, 1.05 equiv) were placed in a 10 mL Schlenk tube. Then CH_2Cl_2 (1.0 mL) was added with a syringe, and the reaction mixure was stirred for 6.5 h at room temperature. After completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 50:1) to afford product **76** as white solid (52.8 mg, 55%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36-7.25 (m, 10H), 5.86-5.78 (m, 2H), 3.47-3.44 (m, 1H), 2.79-2.67 (m, 2H), 2.21 (s, 3H), 1.82-1.73 (m, 1H), 1.66-1.57 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.8, 150.5, 141.0, 137.5, 128.2, 128.1, 127.9, 127.7, 127.5, 127.0, 106.0, 105.9, 66.4, 65.7, 28.2, 24.7, 13.5; **HRMS** (ESI) calculated for $C_{21}H_{21}O_2$ $[M+H]^+$ m/z 305.1536, found 305.1560.

Larger-scale synthesis:



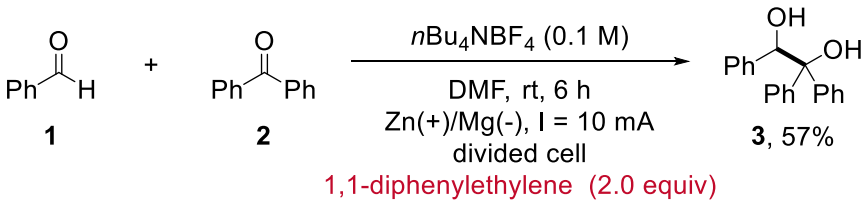
Electroreductive cross-coupling of **1** with **2** has been conducted on a preparative scale (3.6 mmol). Conditions for scale-up to **3** (each reaction): benzophenone **2** (109.8 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, the six reaction mixures were combined. Then H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the desired product **3** (0.73 g, 70%).



Electroreductive cross-coupling of **1** with **71** has been conducted on a preparative scale (3.6 mmol). Conditions for scale-up to **46** (each reaction): (*E*)-*N*,1-diphenylmethanimine **71** (109.5 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.6 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, the six reaction mixures were combined. Then H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10:1) to afford the desired product **46** (0.69 g, 66%, dr = 2.3:1).

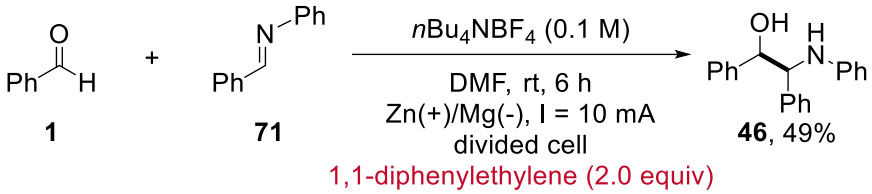
Mechanistic studies

1) Radical trapping experiments:



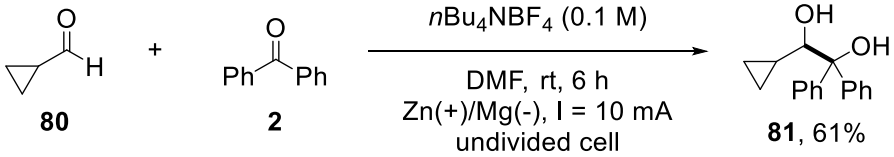
Benzophenone **2** (109.5 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), 1,1-diphenylethylene (212 μL, 1.2 mmol, 2.0 equiv), and nBu$_4$NBF$_4$ (197.9 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After

completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford product **3** (98.9 mg, 57%).



(*E*)-*N*,1-Diphenylmethanimine **71** (108.8 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), 1,1-diphenylethylene (212 μL, 1.2 mmol, 2.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford product **46** (85.3 mg, 49%).

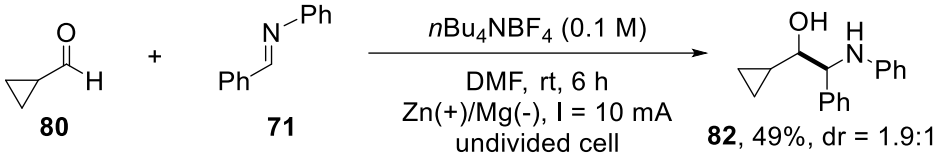
2) Radical clock experiments:



Benzophenone **2** (109.6 mg, 0.6 mmol, 1.0 equiv), cyclopropanecarbaldehyde **80** (135 μL, 1.8 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA

at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the corresponding product **81** (92.9 mg, 61%). In this reaction, the ring opening of cyclopropyl group was not observed.

2-Cyclopropyl-1,1-diphenylethane-1,2-diol: white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60-7.57 (m, 2H), 7.43-7.40 (m, 2H), 7.36-7.32 (m, 2H), 7.27-7.22 (m, 3H), 7.20-7.16 (m, 1H), 3.85 (dd, *J* = 8.2, 2.7 Hz, 1H), 3.24 (s, 1H), 1.98 (d, *J* = 2.9 Hz, 1H), 1.17-1.09 (m, 1H), 0.48-0.41 (m, 1H), 0.33-0.27 (m, 1H), 0.15-0.08 (m, 1H), -0.18--0.25 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.6, 143.8, 128.3, 127.7, 127.1, 126.7, 126.6, 126.1, 80.2, 79.7, 12.9, 4.7, 1.8; **HRMS** (ESI) calculated for $C_{17}H_{18}O_2Na$ $[M+Na]^+$ m/z 277.1199, found 277.1201.

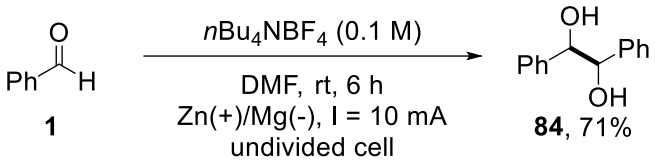


(*E*)-*N*,1-Diphenylmethanimine **71** (108.8 mg, 0.6 mmol, 1.0 equiv), cyclopropanecarbaldehyde **80** (135 μL, 1.8 mmol, 3.0 equiv), and *n*Bu_4NBF_4 (197.6 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford the corresponding product **82** (74.9 mg, 49%). The diastereomeric ratio is 1.9:1 determined by ^{1}H NMR. In this reaction, the ring opening of cyclopropyl group was not observed.

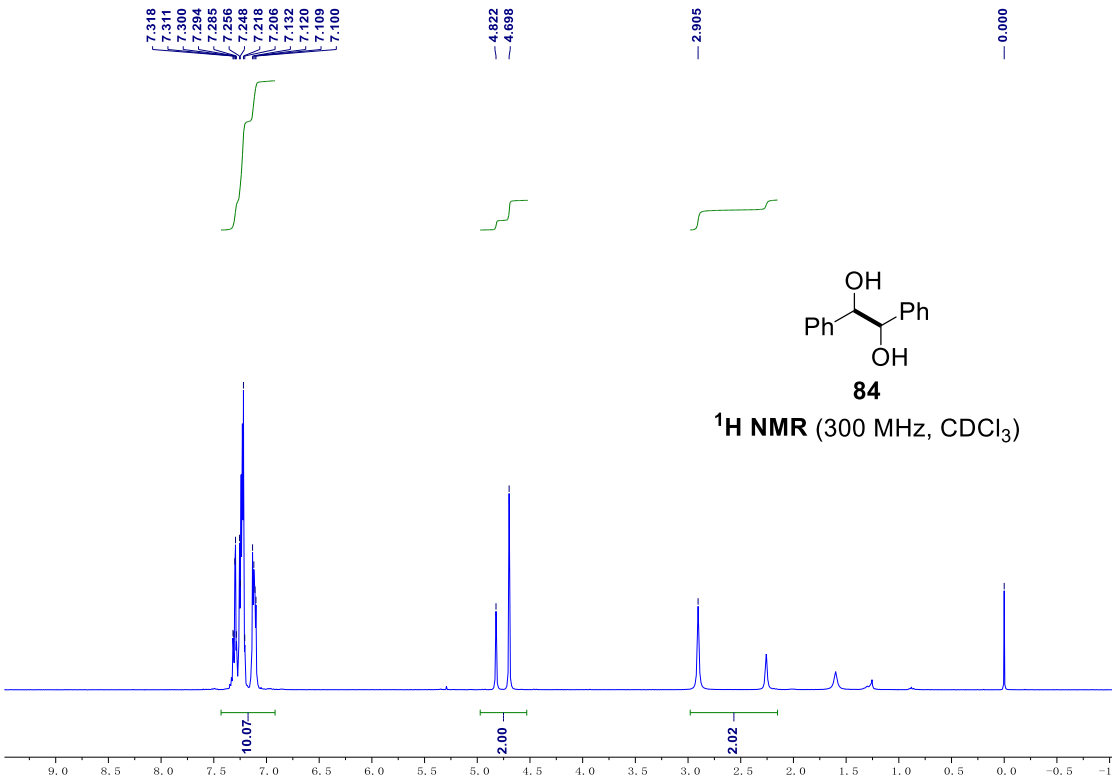
1-Cyclopropyl-2-phenyl-2-(phenylamino)ethan-1-ol: colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ two isomers: 7.42-7.20 (m, 5H), 7.10-7.04 (m, 2H), 6.65-6.52 (m,

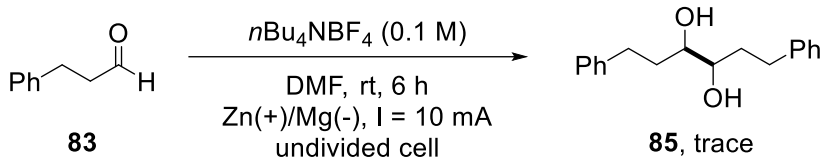
3H), 4.77 (brs, 1H), 4.51 (d, *J* = 3.6 Hz, 0.35H), 4.47 (d, *J* = 4.5 Hz, 0.65H), 3.23 (dd, *J* = 8.7, 3.6 Hz, 0.35H), 3.08 (dd, *J* = 8.5, 4.5 Hz, 0.65H), 2.02 (brs, 0.65H), 1.78 (brs, 0.35H), 1.17-1.09 (m, 0.65H), 0.71-0.62 (m, 0.35H), 0.57-0.39 (m, 2H), 0.37-0.31 (m, 1H), 0.25-0.20 (m, 0.35H), 0.12-0.06 (m, 0.65H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ two isomers: 147.3, 146.9, 141.2, 139.0, 129.1, 129.0, 128.5, 128.3, 127.9, 127.4, 127.3, 127.0, 117.4, 113.6, 113.5, 80.4, 79.0, 62.7, 61.9, 15.0, 14.3, 3.2, 3.1, 2.84, 2.76; **HRMS** (ESI) calculated for $C_{17}H_{20}NO$ $[M+H]^+$ m/z 254.1539, found 254.1564.

3) Control experiments:



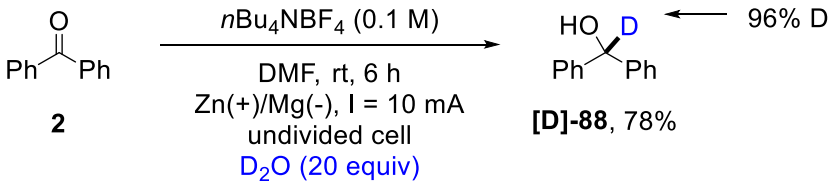
Benzaldehyde **1** (61 μL, 0.6 mmol, 1.0 equiv) and *n*Bu_4NBF_4 (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 4:1) to afford product **84** (45.9 mg, 71%).





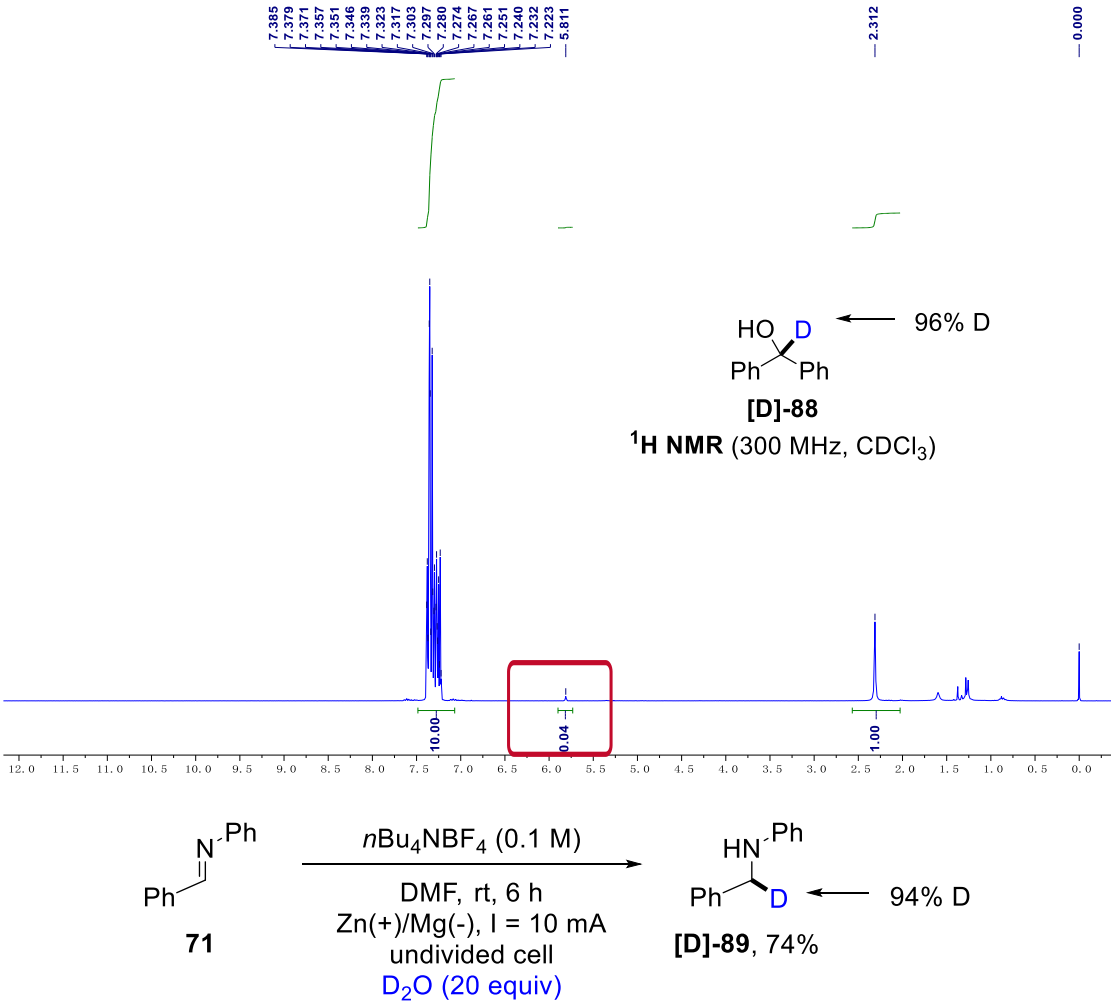
3-Phenylpropanal **83** (79 μL, 0.6 mmol, 1.0 equiv) and nBu$_4$NBF$_4$ (197.5 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. This reaction provided traces of the homopinacol coupling product **85**.

4) Deuteration labeling experiments:



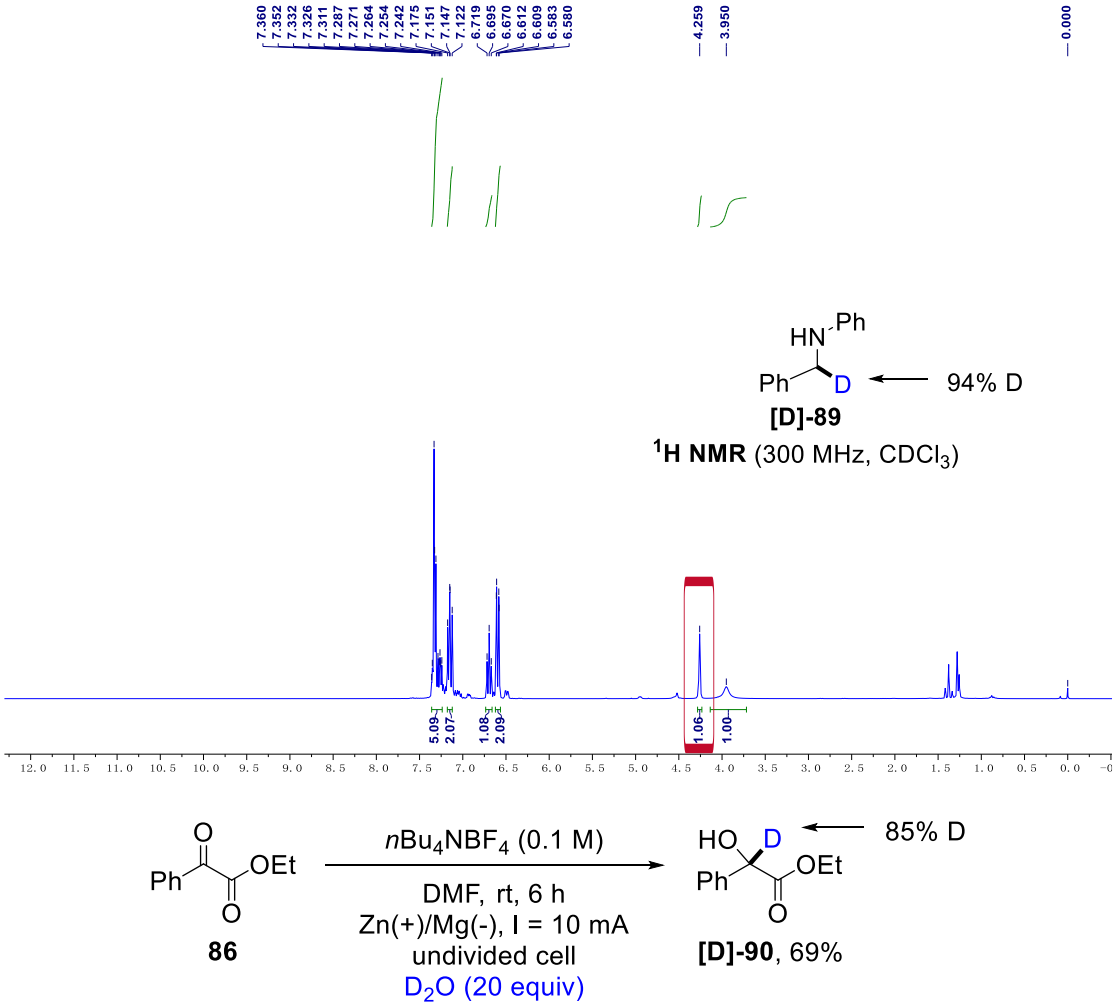
Benzophenone **2** (109.5 mg, 0.6 mmol, 1.0 equiv), D_2O (217 μL, 12 mmol, 20.0 equiv)

and nBu$_4$NBF$_4$ (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford product **[D]-88** (86.8 mg, 78%).



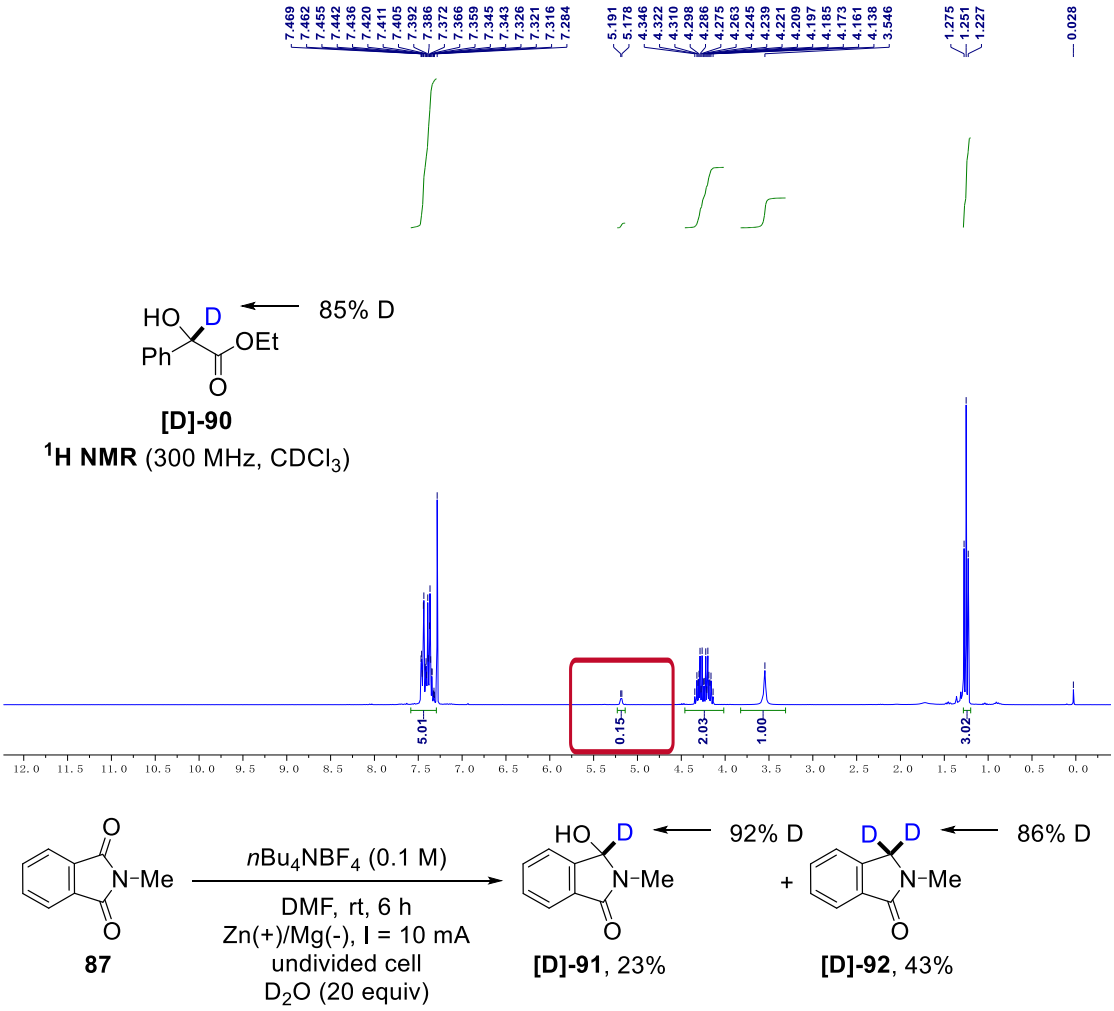
(*E*)-*N*,1-Diphenylmethanimine **71** (108.9 mg, 0.6 mmol, 1.0 equiv), D_2O (217 μL, 12 mmol, 20.0 equiv) and nBu$_4$NBF$_4$ (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with

anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20:1) to afford product **[D]-89** (81.6 mg, 74%).



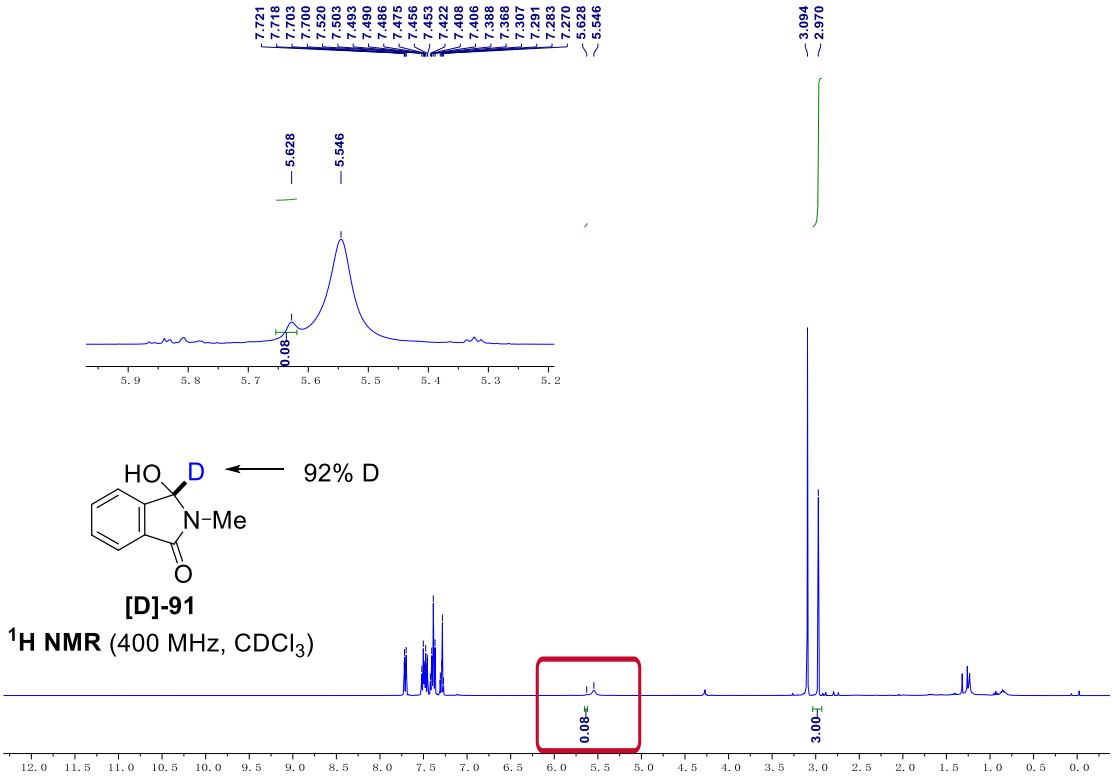
Ethyl 2-oxo-2-phenylacetate **86** (96 μL, 0.6 mmol, 1.0 equiv), D_2O (217 μL, 12 mmol, 20.0 equiv) and *n*Bu_4NBF_4 (197.9 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room

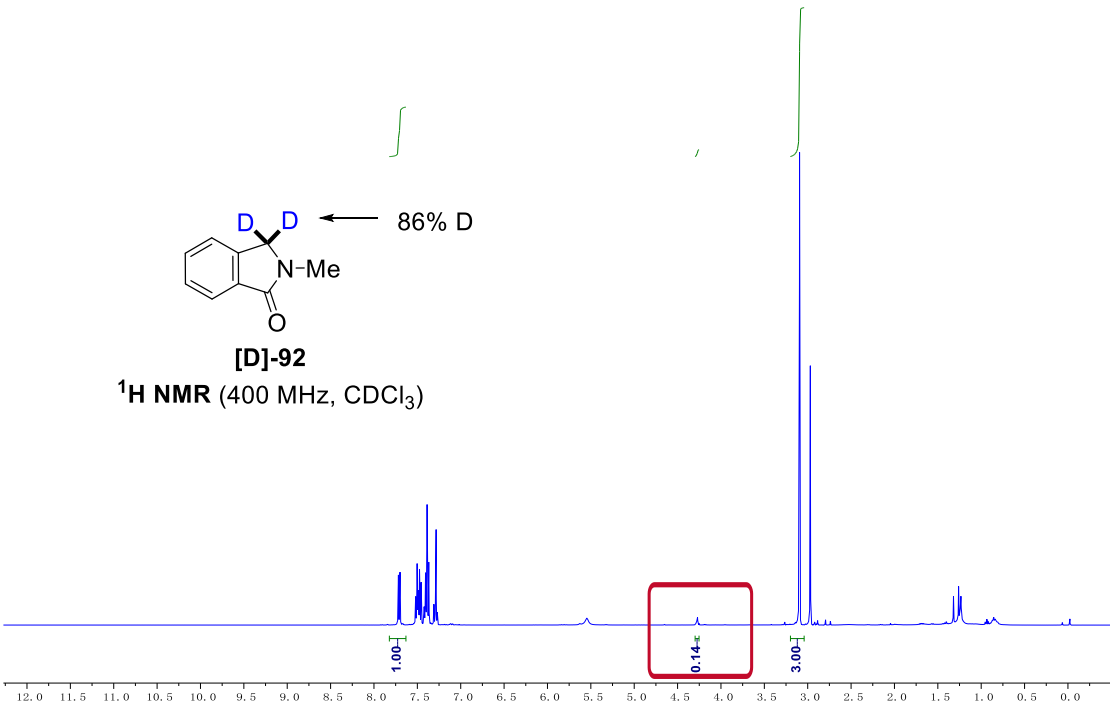
temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 7:1) to afford product **[D]-90** (74.9 mg, 69%).



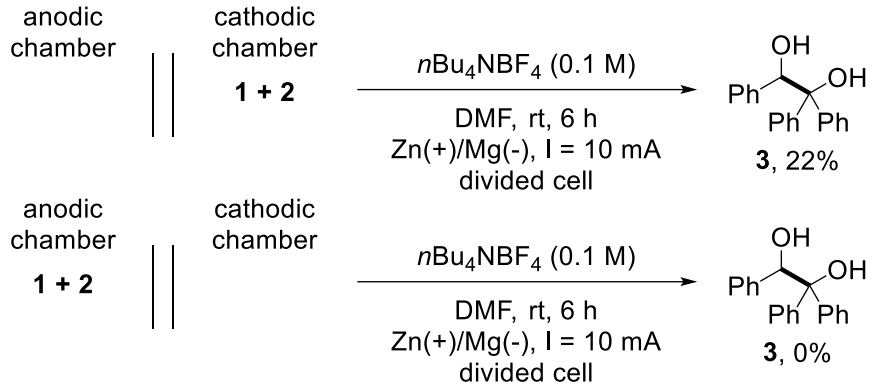
2-Methylisoindoline-1,3-dione **87** (96.7 mg, 0.6 mmol, 1.0 equiv), D_2O (217 μL, 12 mmol, 20.0 equiv) and nBu$_4$NBF$_4$ (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, H_2O (10.0 mL) was added, and the mixture was extracted by EtOAc (3x10.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The

crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 1:1) to afford product **[D]-91** (22.8 mg, 23%) and **[D]-92** (38.8 mg, 43%) as an inseparable mixture.

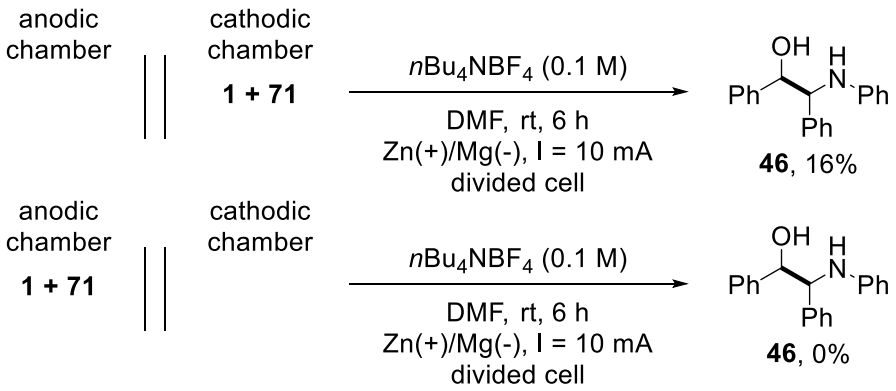




5) Divided-cell electrolysis experiments:



The divided-cell electrolysis was conducted in a H-type divided cell with one stirring bar at each chamber. The zinc plate was used as the anode (2.0 cm x 0.2 cm x 0.8 cm) and the magnesium plate was used as the cathode (2.0 cm x 0.2 cm x 0.8 cm), which were separated by a cation exchange membrane (CMI-7000 membrane). Benzophenone **2** (109.7 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), and *n*Bu$_4$NBF$_4$ (329.3 mg, 1.0 mmol, 0.1 M) were only added into the anodic chamber, while in another reaction the same substrates were only added into the cathodic chamber. Then anhydrous DMF (10.0 mL) was added with a syringe. After degassing with a stream of N_2 for 10 min, the two reactions were stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, the solution was treated by the standard procedure. The desired 1,2-diol product **3** was isolated in 22% yield (38.3 mg) from the reaction in which **1** and **2** were only added into the cathodic chamber. In contrast, no 1,2-diol product was observed from the reaction in which **1** and **2** were only added into the anodic chamber.



The divided-cell electrolysis was conducted in a H-type divided cell with one stirring

bar at each chamber. The zinc plate was used as the anode (2.0 cm x 0.2 cm x 0.8 cm) and the magnesium plate was used as the cathode (2.0 cm x 0.2 cm x 0.8 cm), which were separated by a cation exchange membrane (CMI-7000 membrane). (*E*)-*N*,1-diphenylmethanimine **71** (108.8 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), and *n*Bu$_4$NBF$_4$ (329.5 mg, 1.0 mmol, 0.1 M) were only added into the anodic chamber, while in another reaction the same substrates were only added into the cathodic chamber. Then anhydrous DMF (6.0 mL) was added with a syringe. After degassing with a stream of N_2 for 10 min, the two reactions were stirred and electrolyzed at a constant current of 10 mA at room temperature for 6 h. After completion of the reaction, the solution was treated by the standard procedure. The desired β-amino alcohol product **46** was isolated in 16% yield (27.8 mg) from the reaction in which **1** and **71** were only added into the cathodic chamber. In contrast, no β-amino alcohol product was observed from the reaction in which **1** and **71** were only added into the anodic chamber.

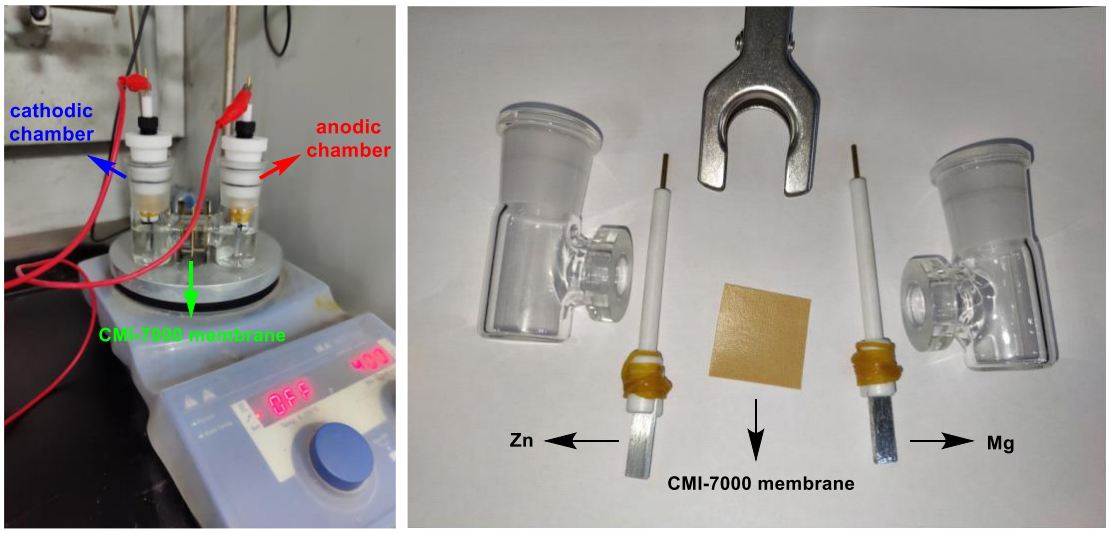
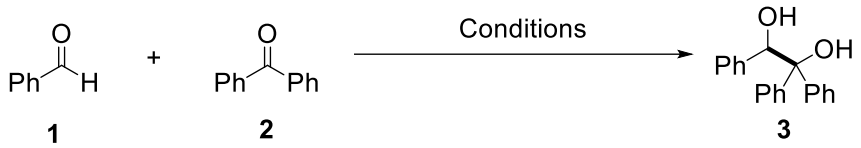


Figure S2 Reaction setup for divided cell electrolysi

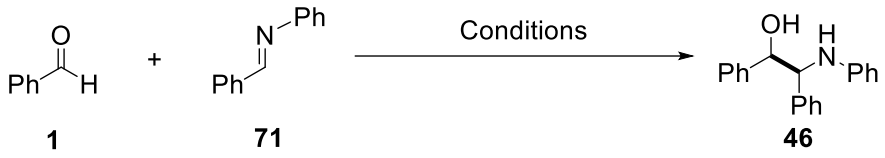
6) Zn^0 reduction control experiments:



entry	conditions	yield
1	No electrical input, **1**, **2**, Zn powder nBu$_4$NBF$_4$, DMF, 12 h, rt	0%
2	Zn(+)/Mg(-), nBu$_4$NBF$_4$, DMF, I = 10 mA, 3 h then, add **1** and **2**, 12 h, rt	0%

Entry 1: Benzophenone **2** (109.5 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), zinc power from a fresh opened bottle (117.9 mg, 1.8 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.7 mg, 0.6 mmol, 1.0 equiv) were placed in a 10 mL Schlenk tube equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. After degassing with a stream of N_2 for 10 min, and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no product **3** was observed.

Entry 2: nBu$_4$NBF$_4$ (197.6 mg, 0.6 mmol, 1.0 equiv) was placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 3 h. After the electrolysis, benzophenone **2** (109.9 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv) were added, and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no product **3** was observed.



1 **71** **46**

entry	conditions	yield
1	No electrical input, **1**, **71**, Zn powder nBu$_4$NBF$_4$, DMF, 12 h, rt	0%
2	Zn(+)/Mg(-), nBu$_4$NBF$_4$, DMF, I = 10 mA, 3 h then, add **1** and **71**, 12 h, rt	0%

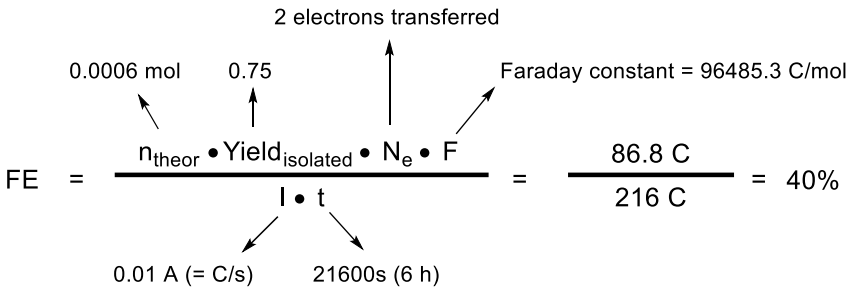
Entry 1: (*E*)-*N*,1-diphenylmethanimine **71** (108.9 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv), zinc power from a fresh opened bottle (117.9 mg, 1.8 mmol, 3.0 equiv), and nBu$_4$NBF$_4$ (197.8 mg, 0.6 mmol, 1.0 equiv) were placed in a 10 mL Schlenk tube equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. After degassing with a stream of N_2 for 10 min, and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no product **46** was observed.

Entry 2: nBu$_4$NBF$_4$ (197.9 mg, 0.6 mmol, 1.0 equiv) was placed in an ElectraSyn undivided cell (10.0 mL) equipped with a stiring bar. Then anhydrous DMF (6.0 mL) was added with a syringe. The ElectraSyn vial cap equipped with anode (Zn, 4.0 cm x 0.2 cm x 0.8 cm) and cathode (Mg, 4.0 cm x 0.2 cm x 0.8 cm) was inserted into the mixure. After degassing with a stream of N_2 for 10 min, the reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 3 h. After the electrolysis, (*E*)-*N*,1-diphenylmethanimine **71** (109.1 mg, 0.6 mmol, 1.0 equiv), benzaldehyde **1** (183 μL, 1.8 mmol, 3.0 equiv) were added, and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no product **3** was observed.

7) Determination of Faradaic efficiency:

The Faradaic efficiency (FE) was determined according to the methods reported in the literature.[12]

The Faradaic efficiency for product **3** (isolated yield = 75% on a 0.6 mmol scale) is calculated using the following equation:



8) Cyclic voltammetry analysis:

The cyclic voltammogram was carried out with a Shanghai Chenhua CHI620E workstation. Cyclic voltammetry was performed in a three-electrode cell at room temperature. The working electrode was a glassy carbon (GC) electrode, the counter electrode was a platinum wire, and the reference electrode was saturated calomel electrode (SCE). A DMF solution (10.0 mL) of sample including 0.02 M of each sample and 0.1 M of nBu$_4$NBF$_4$ was prepared as an electrochemical solution. The spectra were recorded with the scan rate of 100 mVs^{-1}.

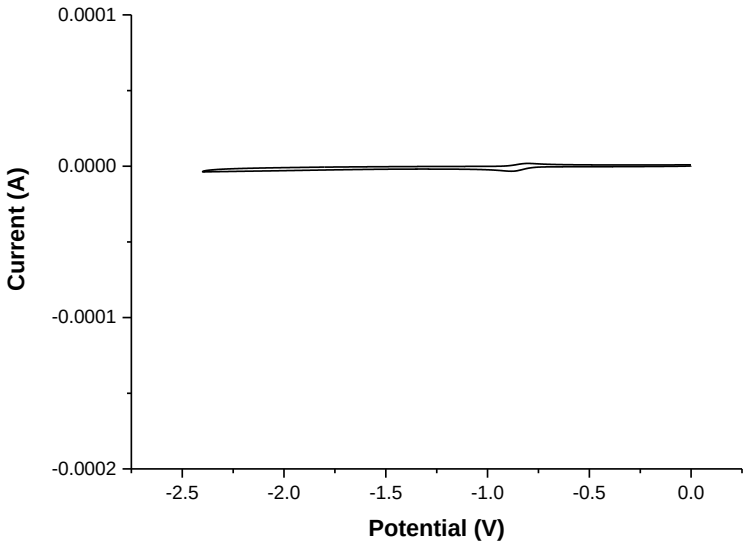


Figure S3 Cyclic voltammograms for blank experiment

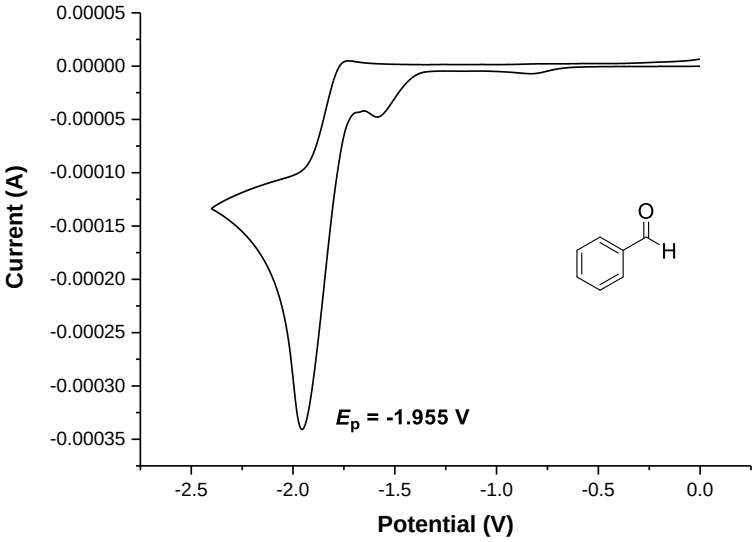


Figure S4 Cyclic voltammograms for benzaldehyde

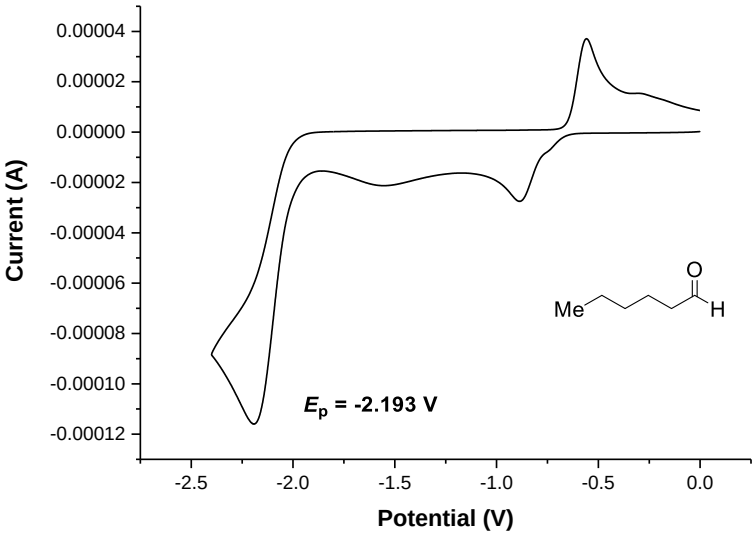


Figure S5 Cyclic voltammograms for hexanal

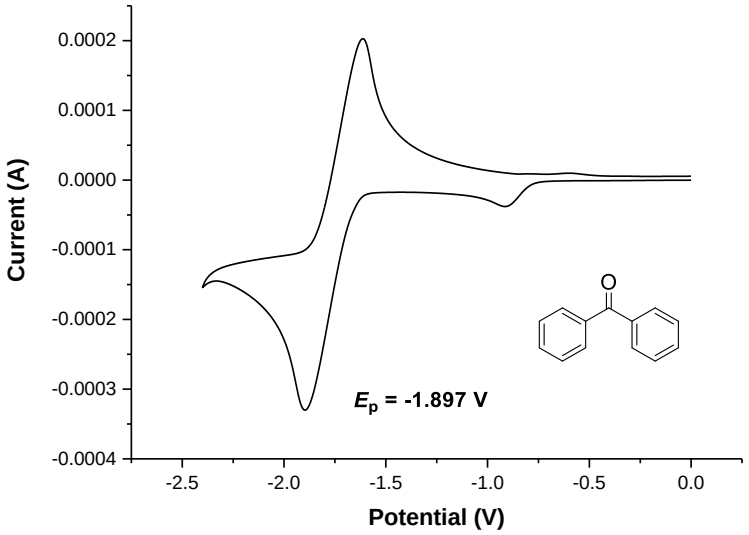


Figure S6 Cyclic voltammograms for benzophenone

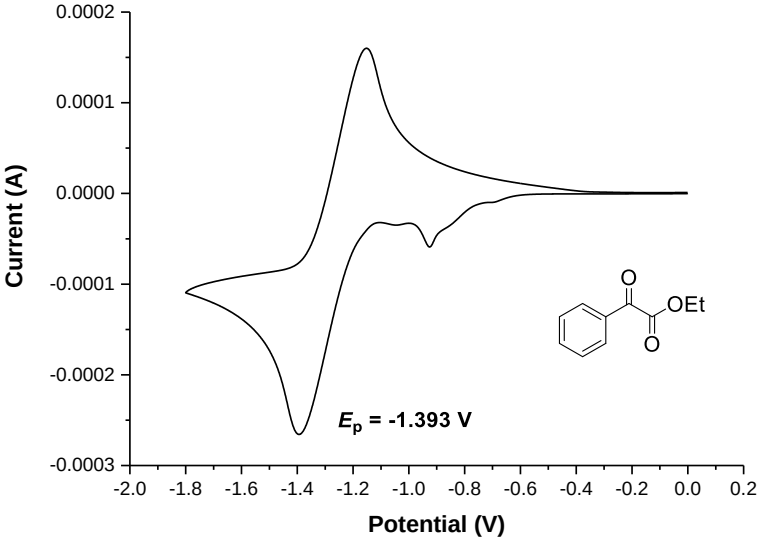


Figure S7 Cyclic voltammograms for ethyl 2-oxo-2-phenylacetate

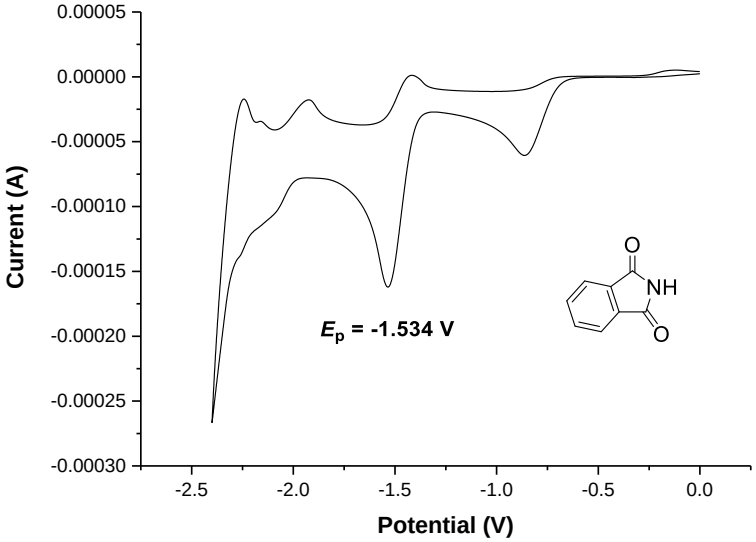


Figure S8 Cyclic voltammograms for isoindoline-1,3-dione

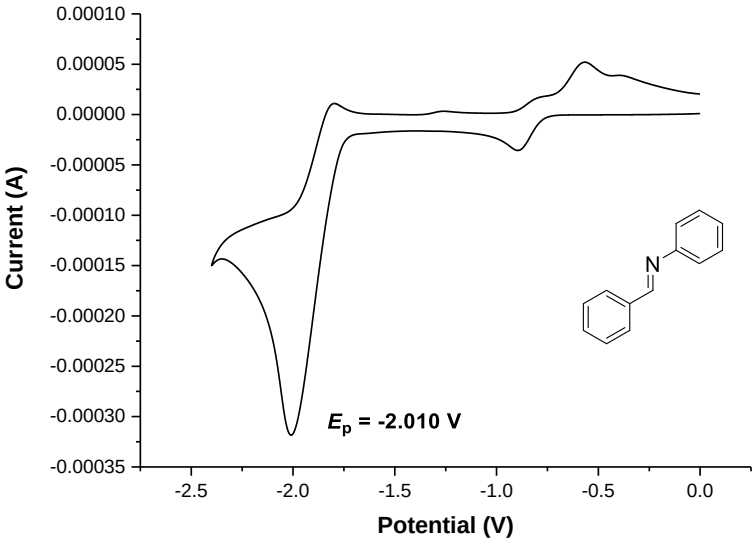


Figure S9 Cyclic voltammograms for (*E*)-*N*,1-diphenylmethanimine

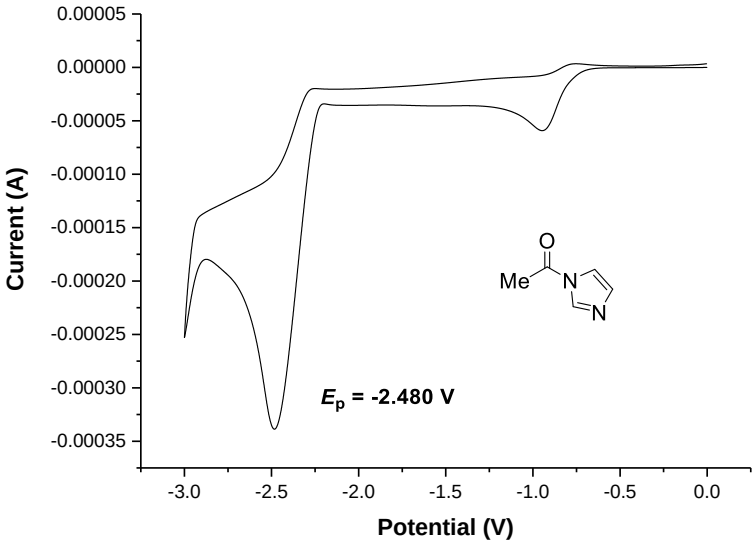


Figure S10 Cyclic voltammograms for 1-(1*H*-imidazol-1-yl)ethan-1-one

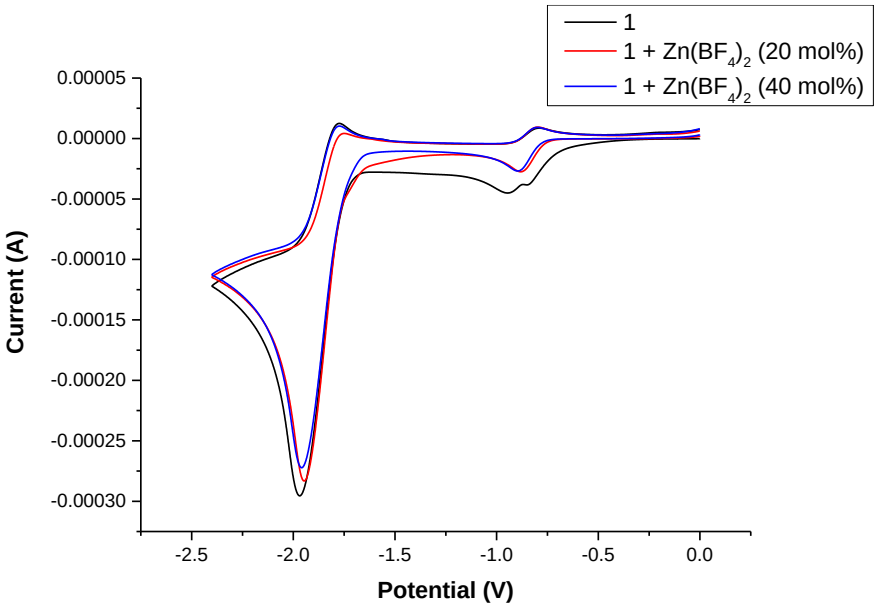


Figure S11 Cyclic voltammograms for benzaldehyde (**1**) + $Zn(BF_4)_2$

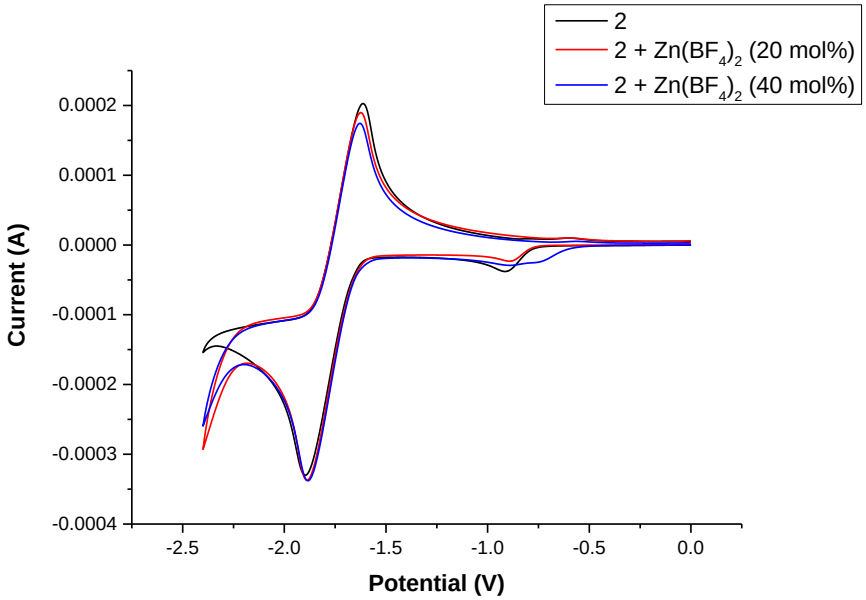


Figure S12 Cyclic voltammograms for benzophenone (**2**) + $Zn(BF_4)_2$

References:

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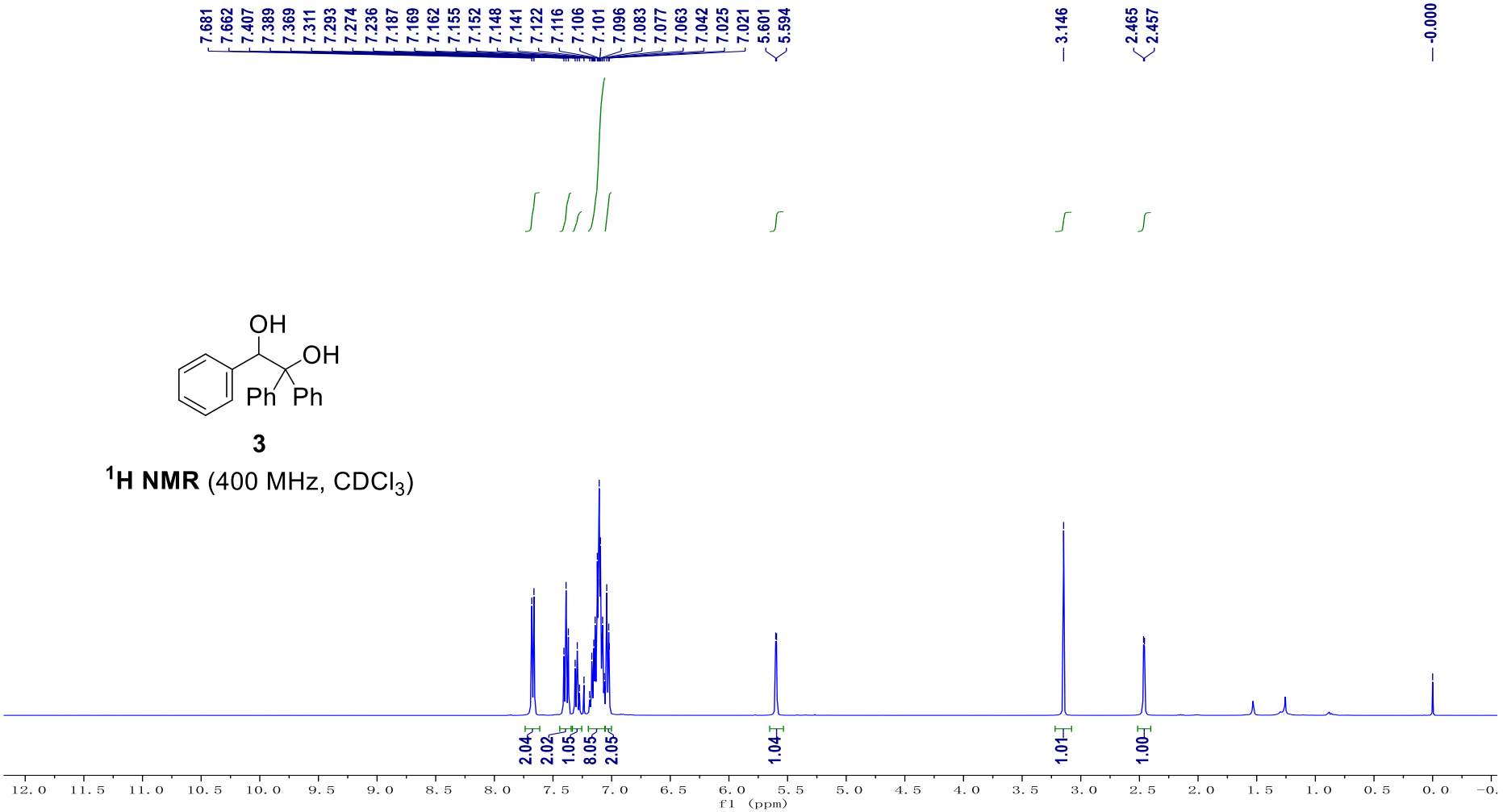
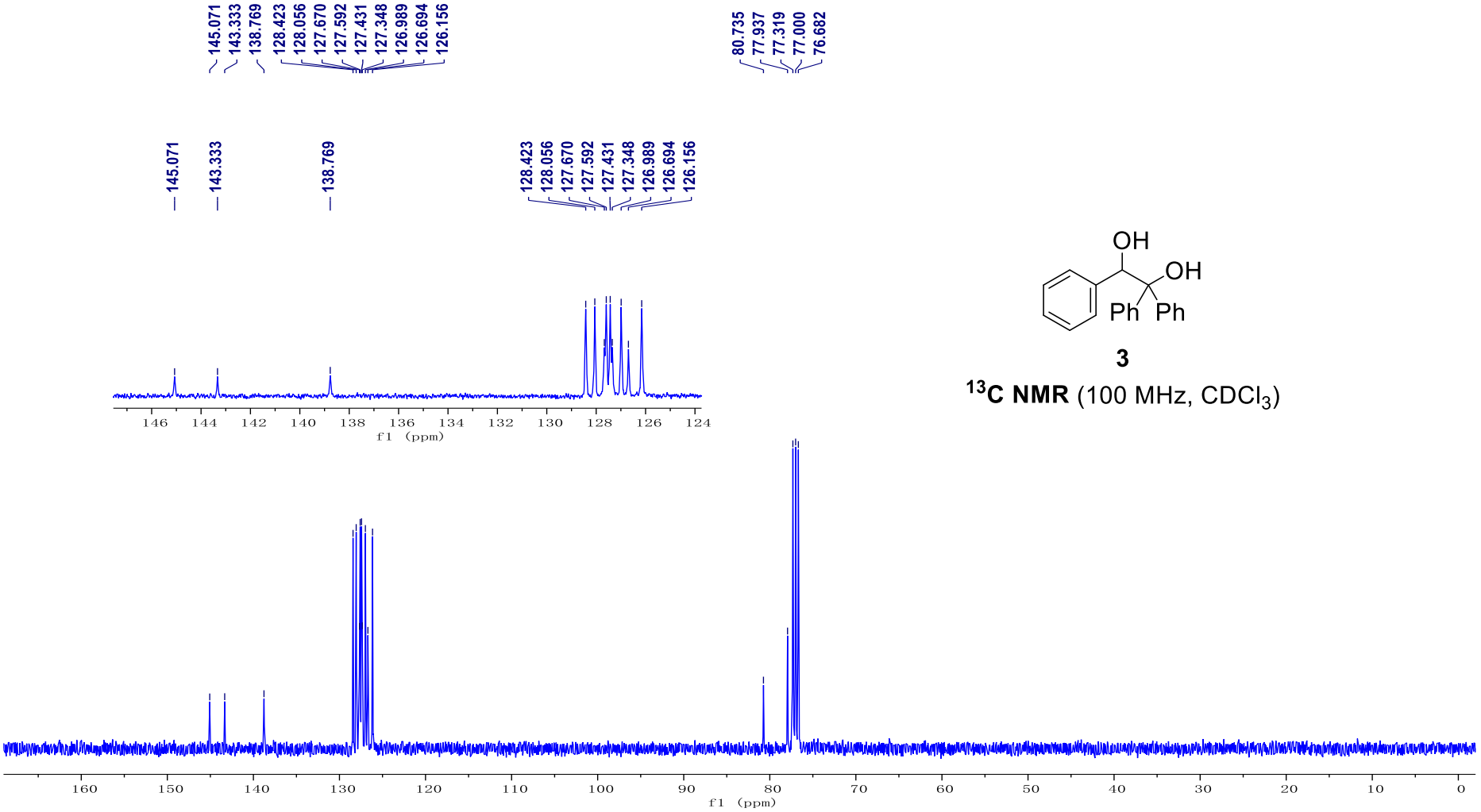
[12] Y. Qiu, L. Xin, D. J. Chadderdon, J. Qi, C. Liang, W. Li, *Green Chem.* **2014**, *16*, 1305.



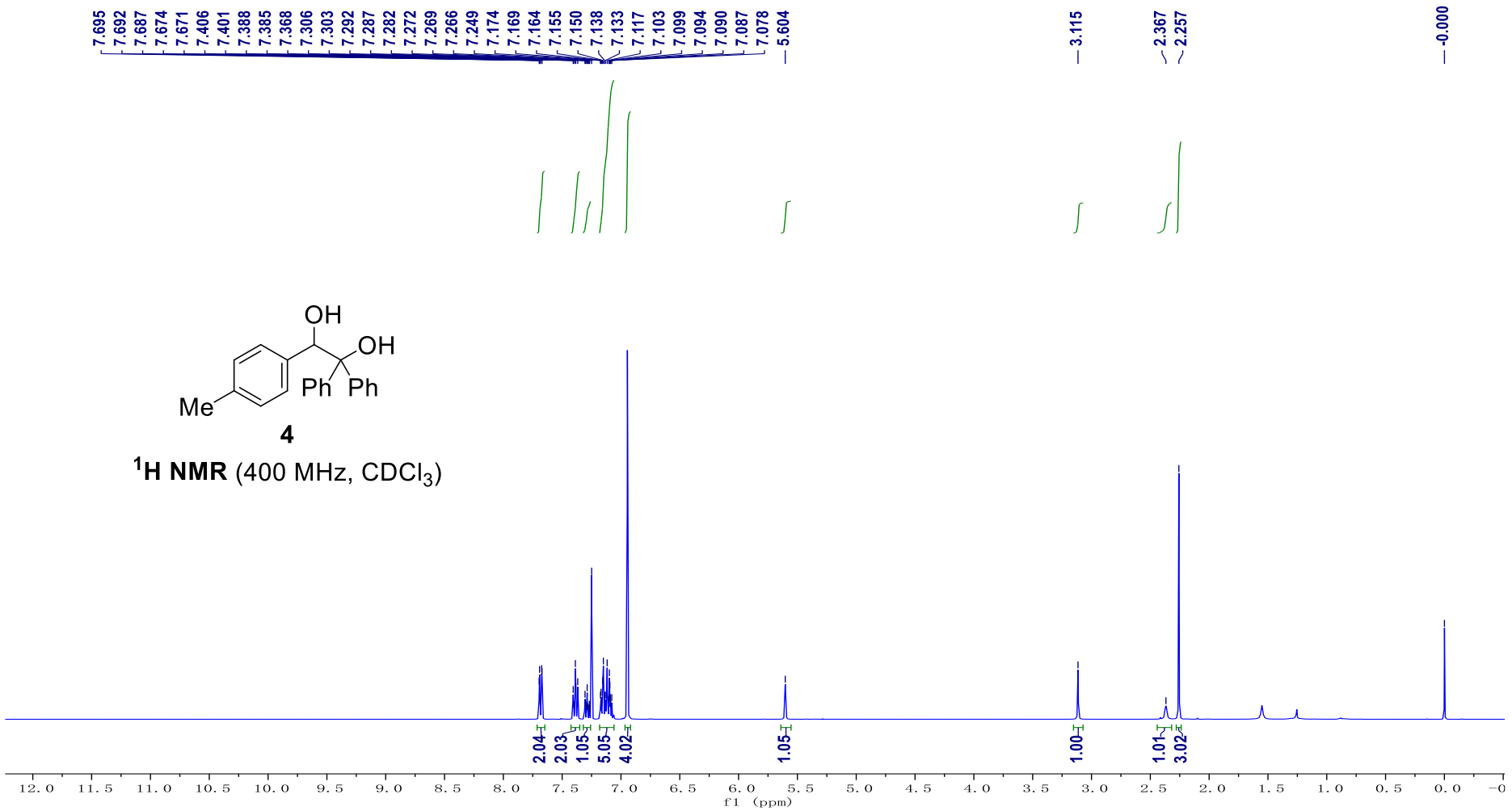
3

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

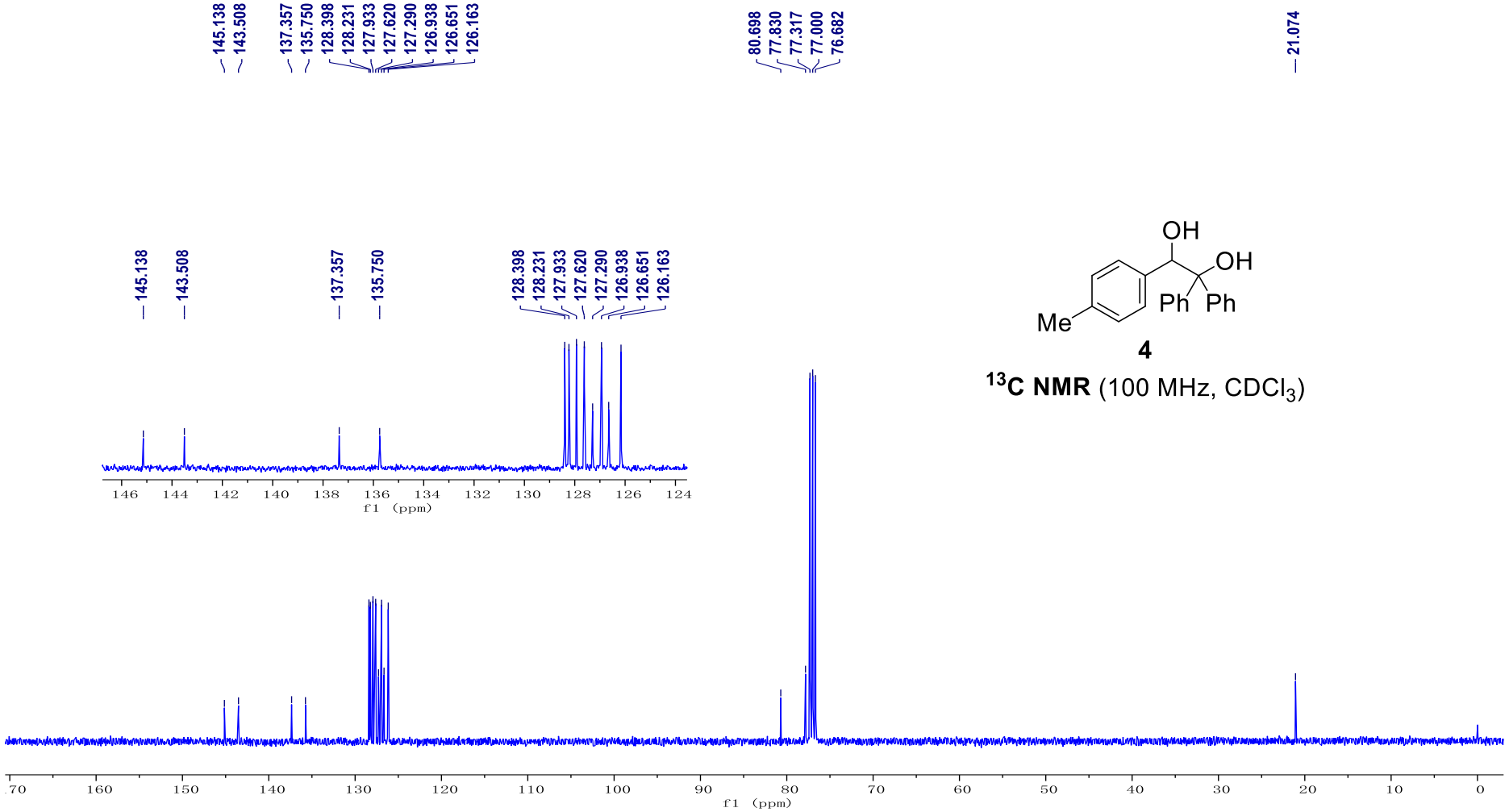


3

^{13}C NMR (100 MHz, $CDCl_3$)

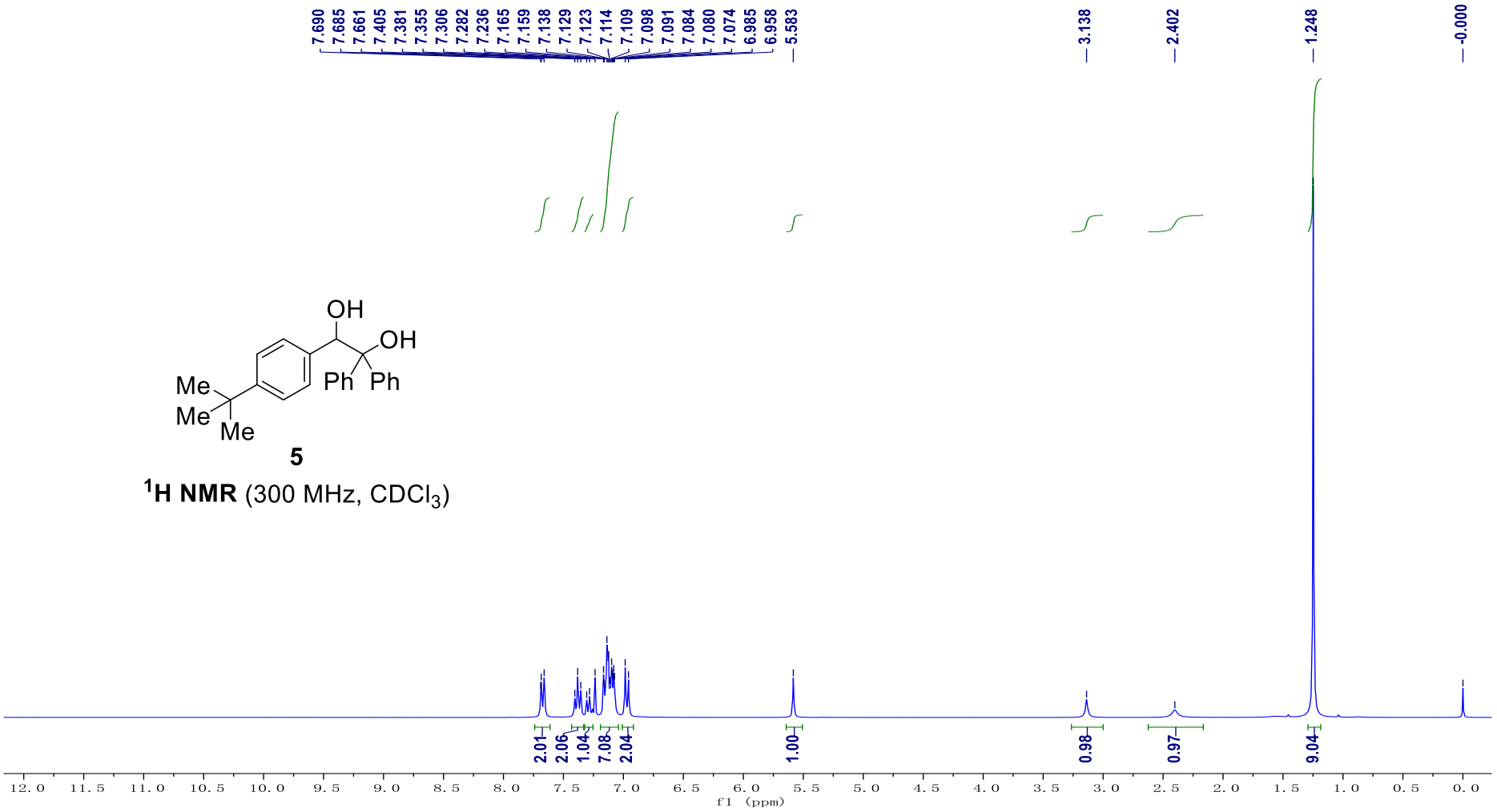


^{1}H NMR (400 MHz, $CDCl_3$)



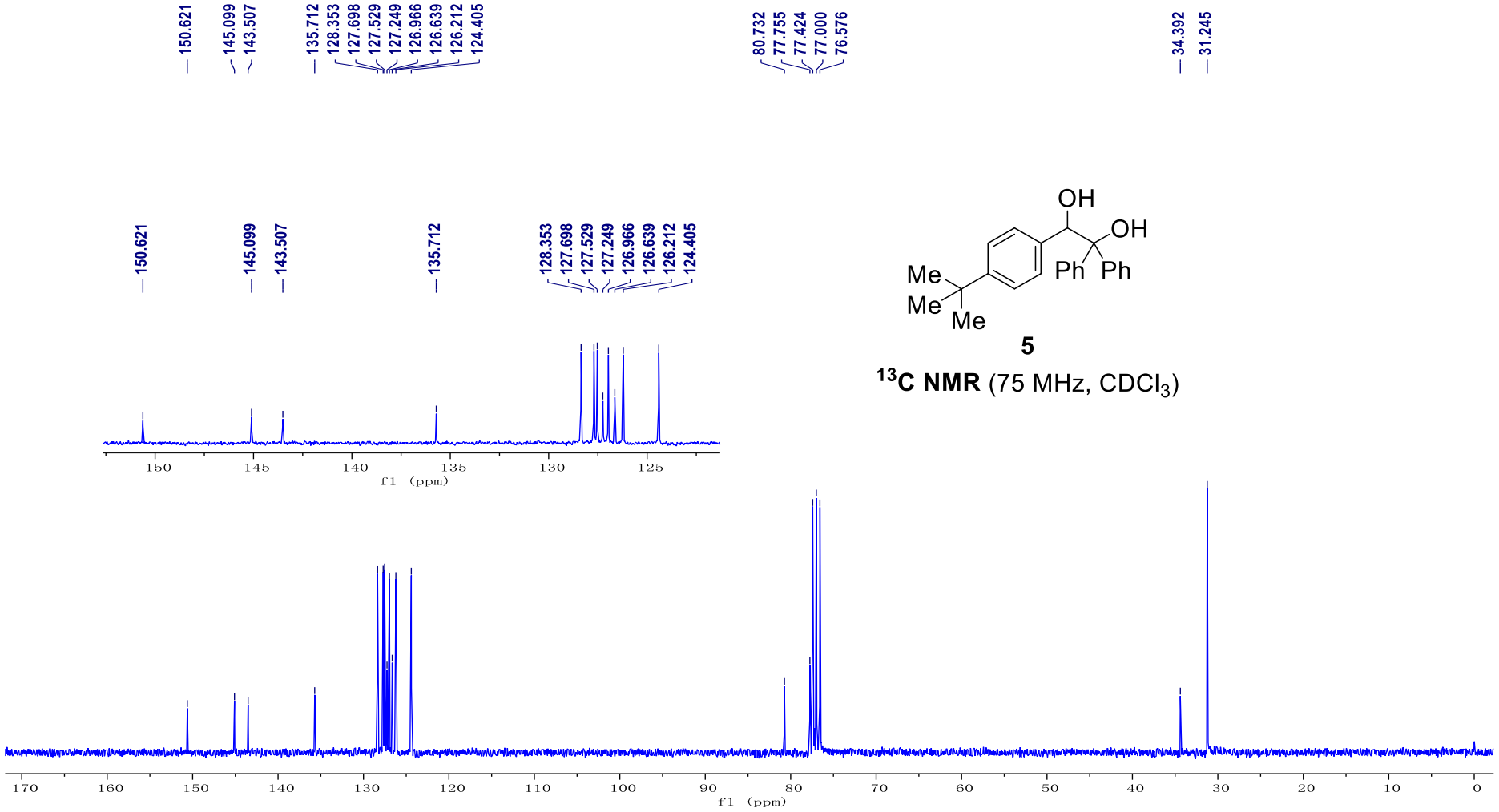
4

^{13}C NMR (100 MHz, $CDCl_3$)



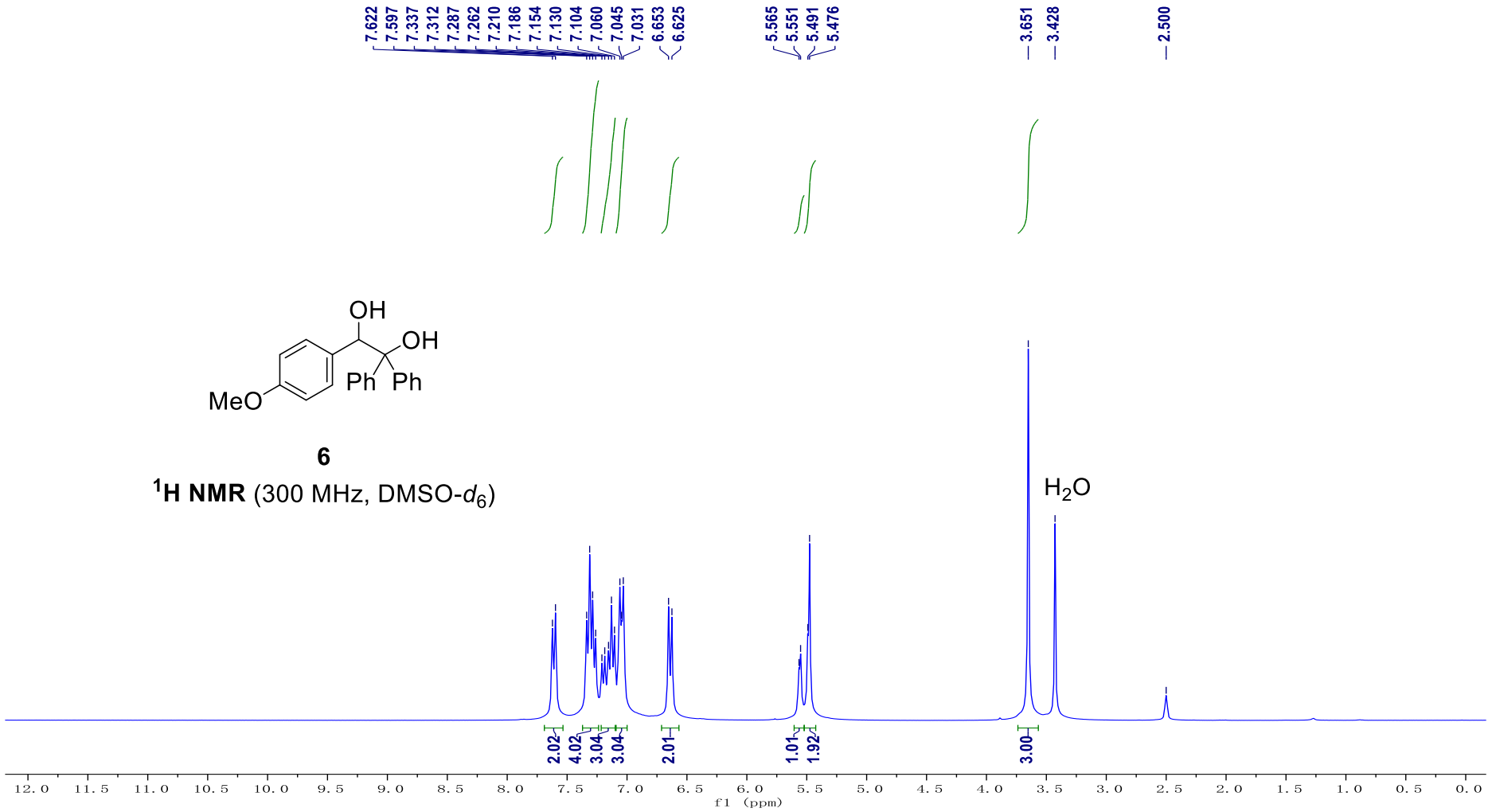
5

^{1}H NMR (300 MHz, $CDCl_3$)



5

^{13}C NMR (75 MHz, $CDCl_3$)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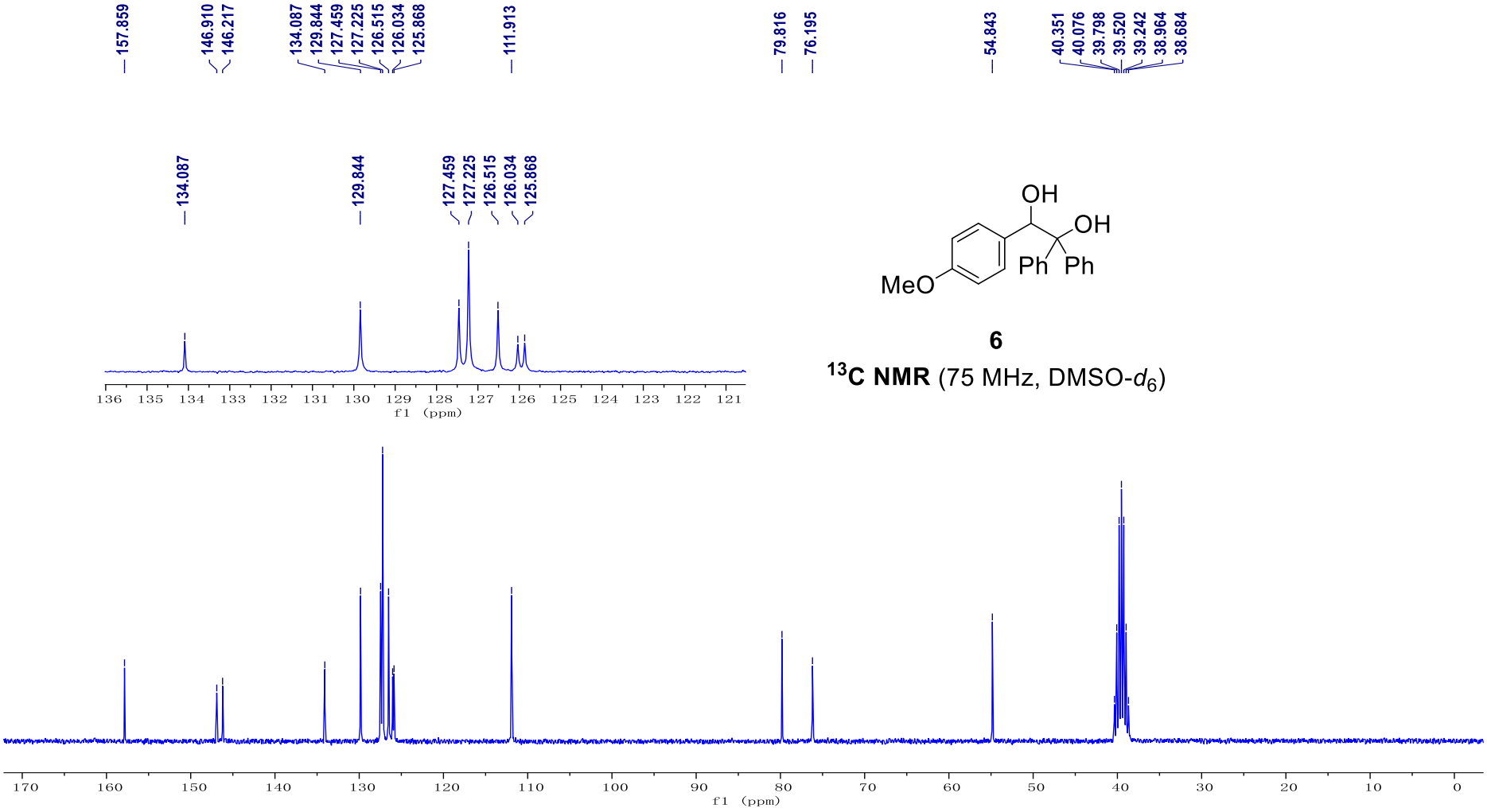
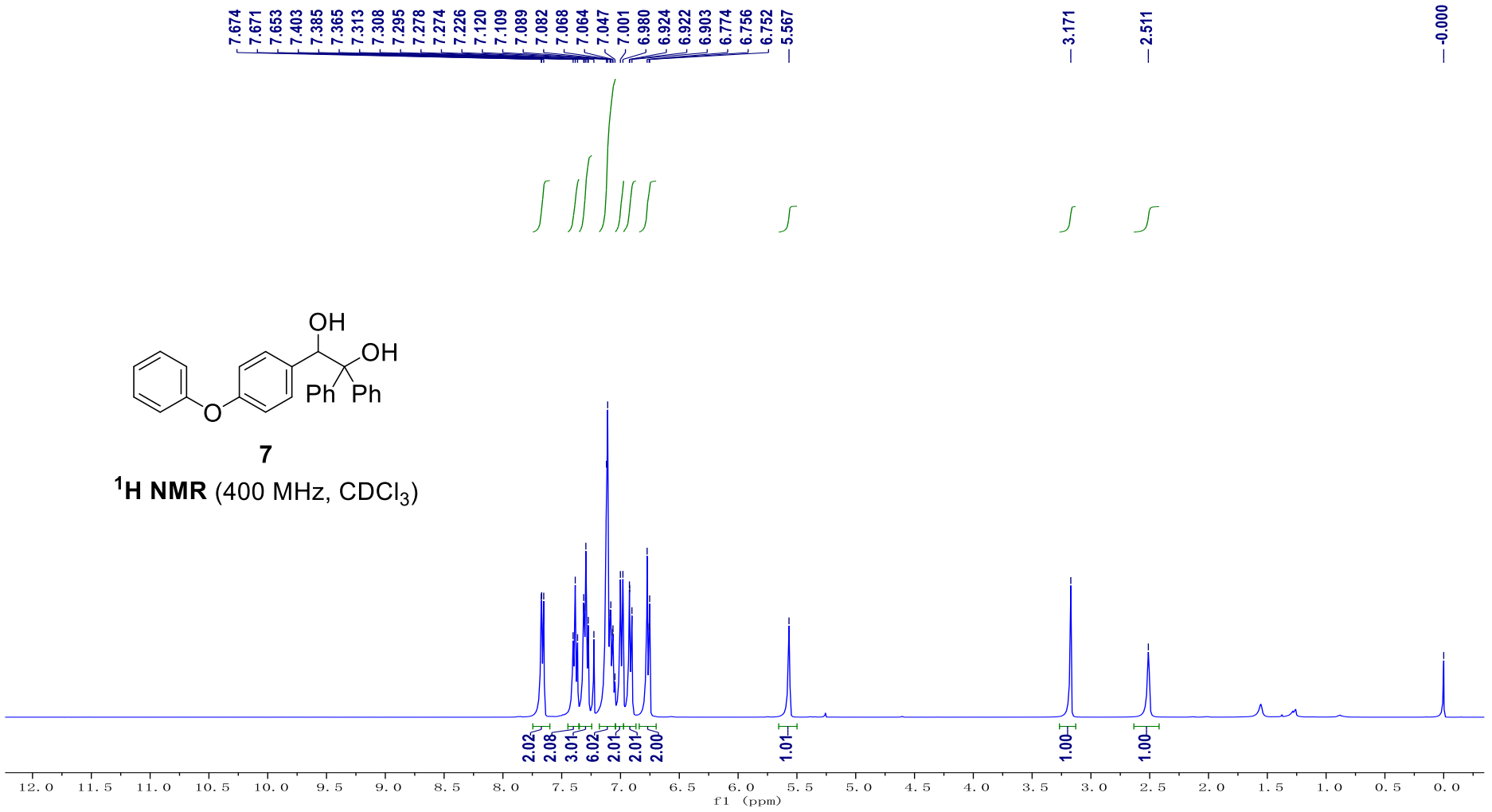
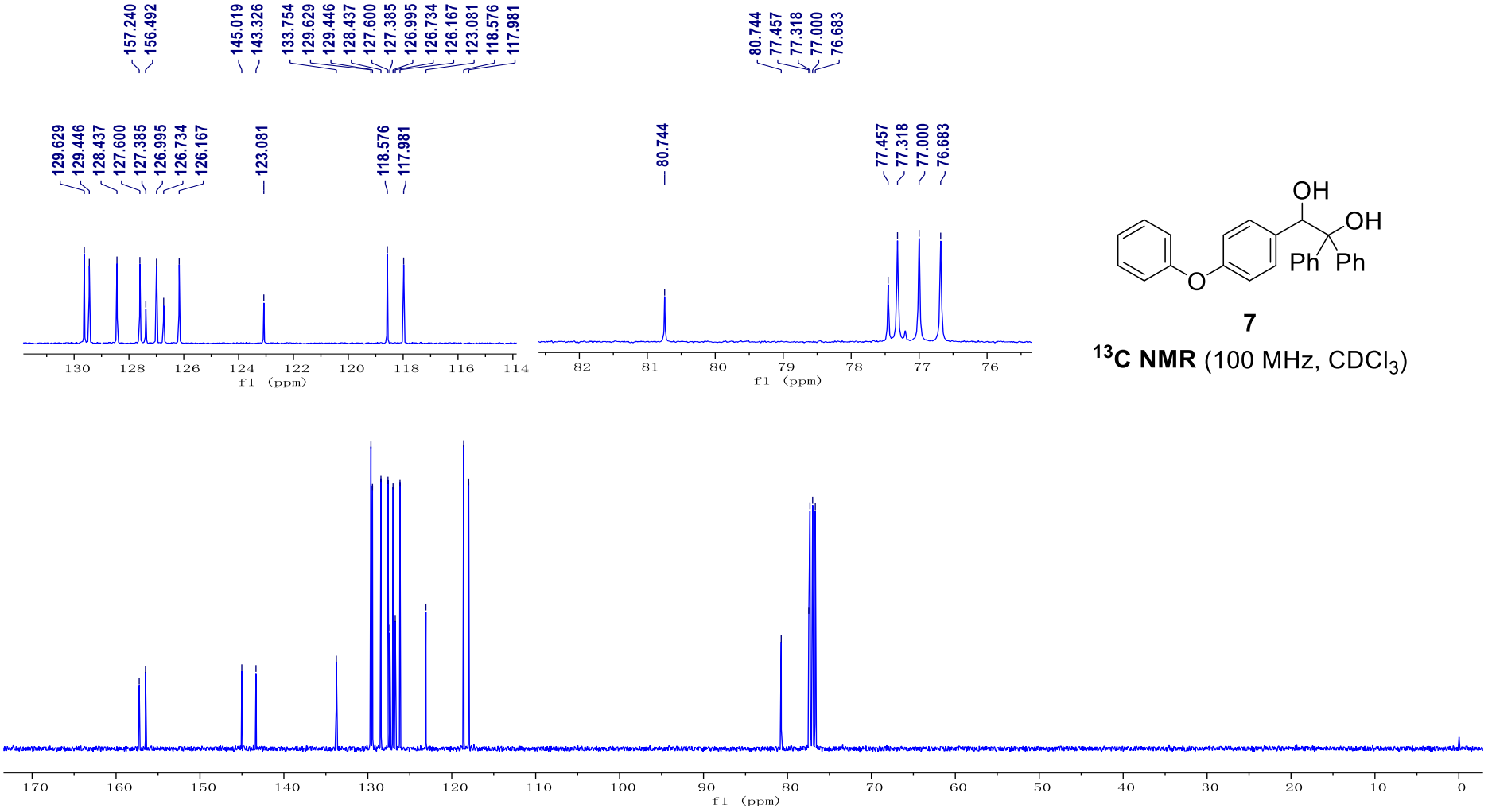
7.597
7.337
7.312
7.287
7.262
7.210
7.186
7.154
7.130
7.104
7.060
7.045
7.031
6.653
6.625
5.565
5.551
5.491
5.476
3.651
3.428
2.500
OH
OH
Ph
Ph
MeO
6
1H NMR (300 MHz, DMSO-d6)
H2O
2.02
4.02
3.04
3.04
2.01
1.01
1.92
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



6

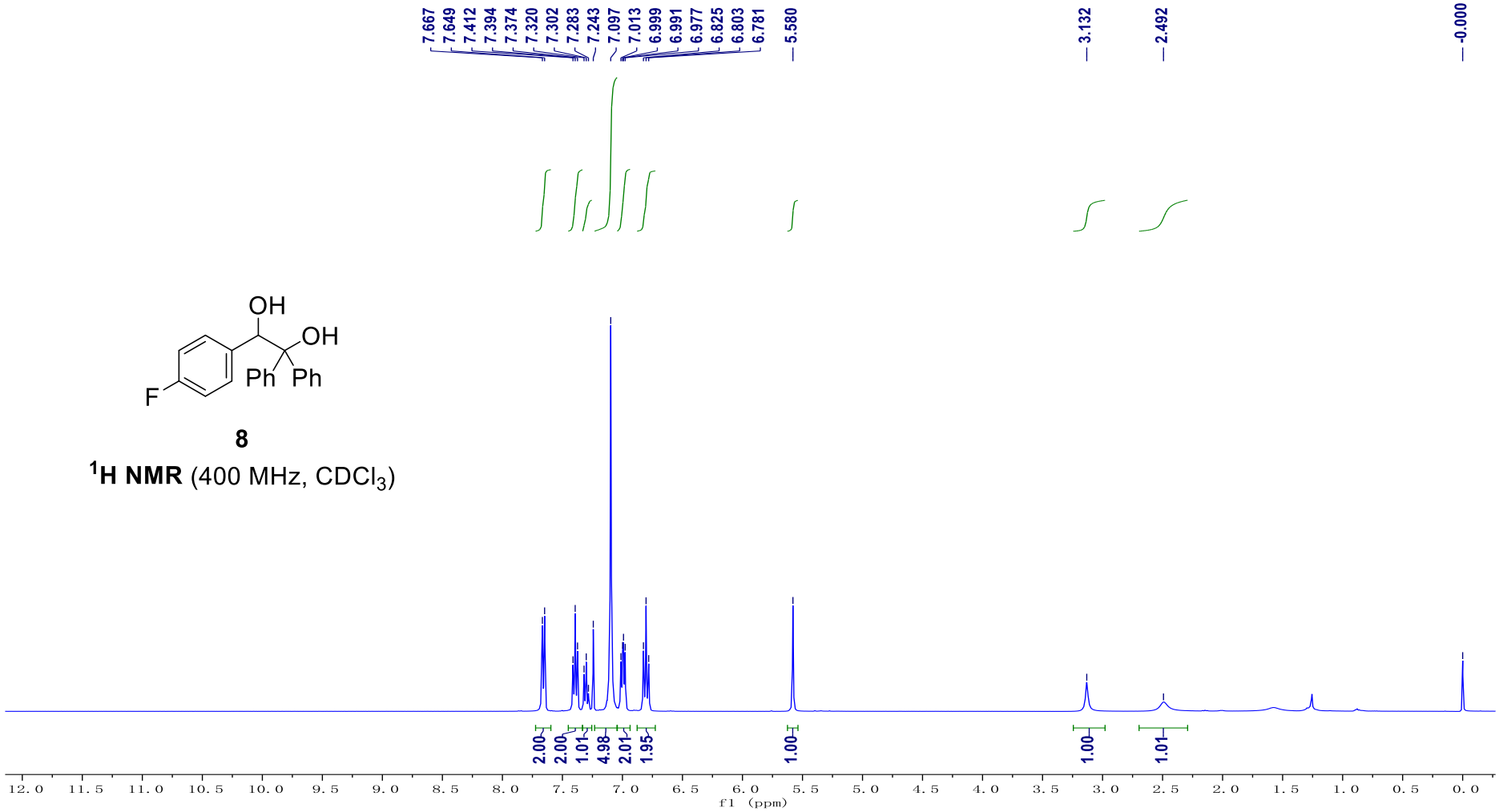
^{13}C NMR (75 MHz, DMSO-d_6)


7.674
7.671
7.653
7.403
7.385
7.365
7.313
7.308
7.295
7.278
7.274
7.226
7.120
7.109
7.089
7.082
7.068
7.064
7.047
7.001
6.980
6.924
6.922
6.903
6.774
6.756
6.752
5.567
3.171
2.511
-0.000
OH
OH
Ph
Ph
O
7
1H NMR (400 MHz, CDCl3)
2.02
2.08
3.01
6.02
2.01
2.01
2.00
1.01
1.00
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



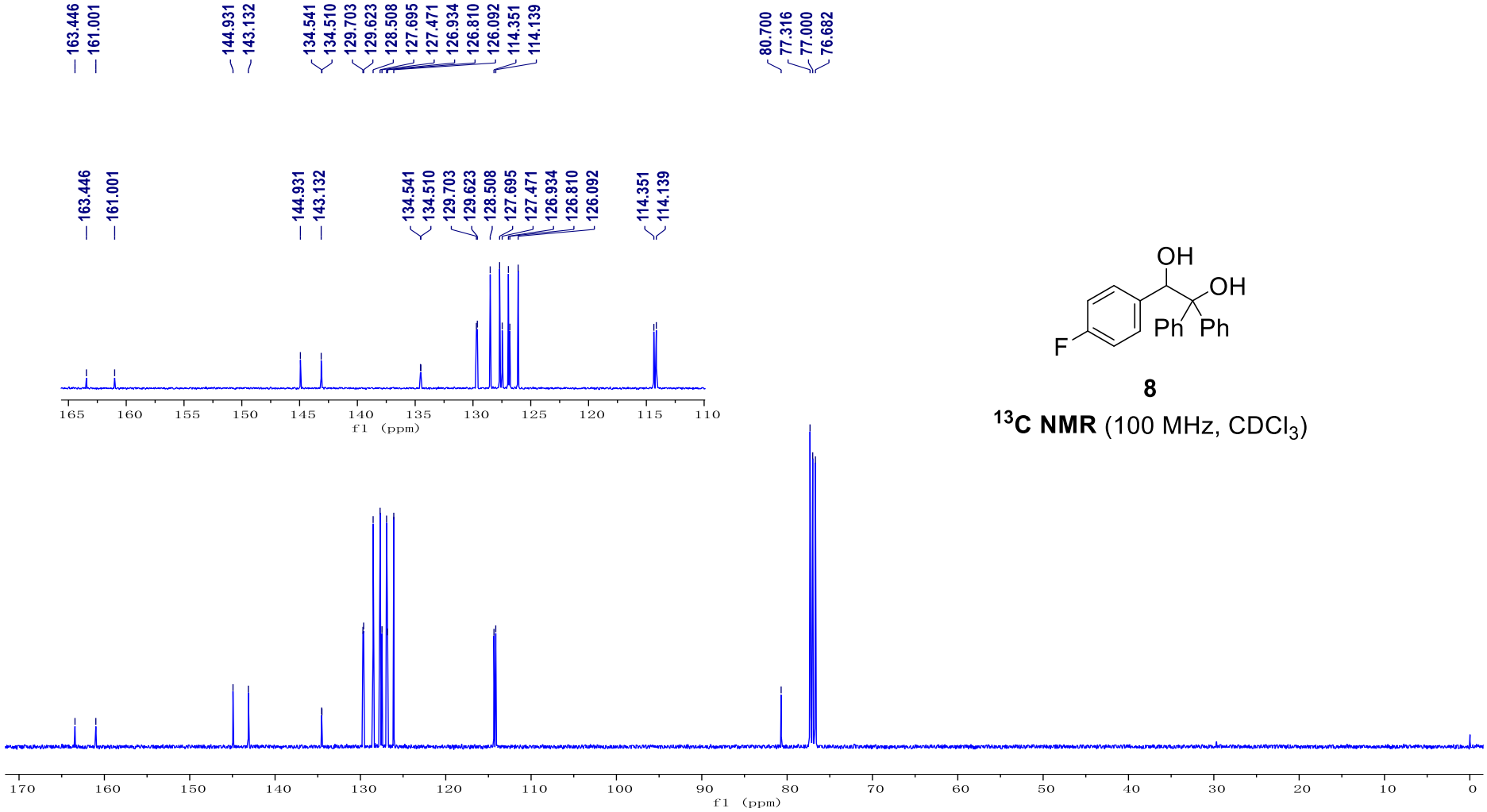
7

^{13}C NMR (100 MHz, $CDCl_3$)



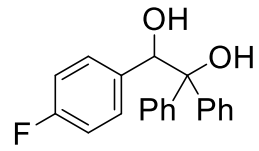
8

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



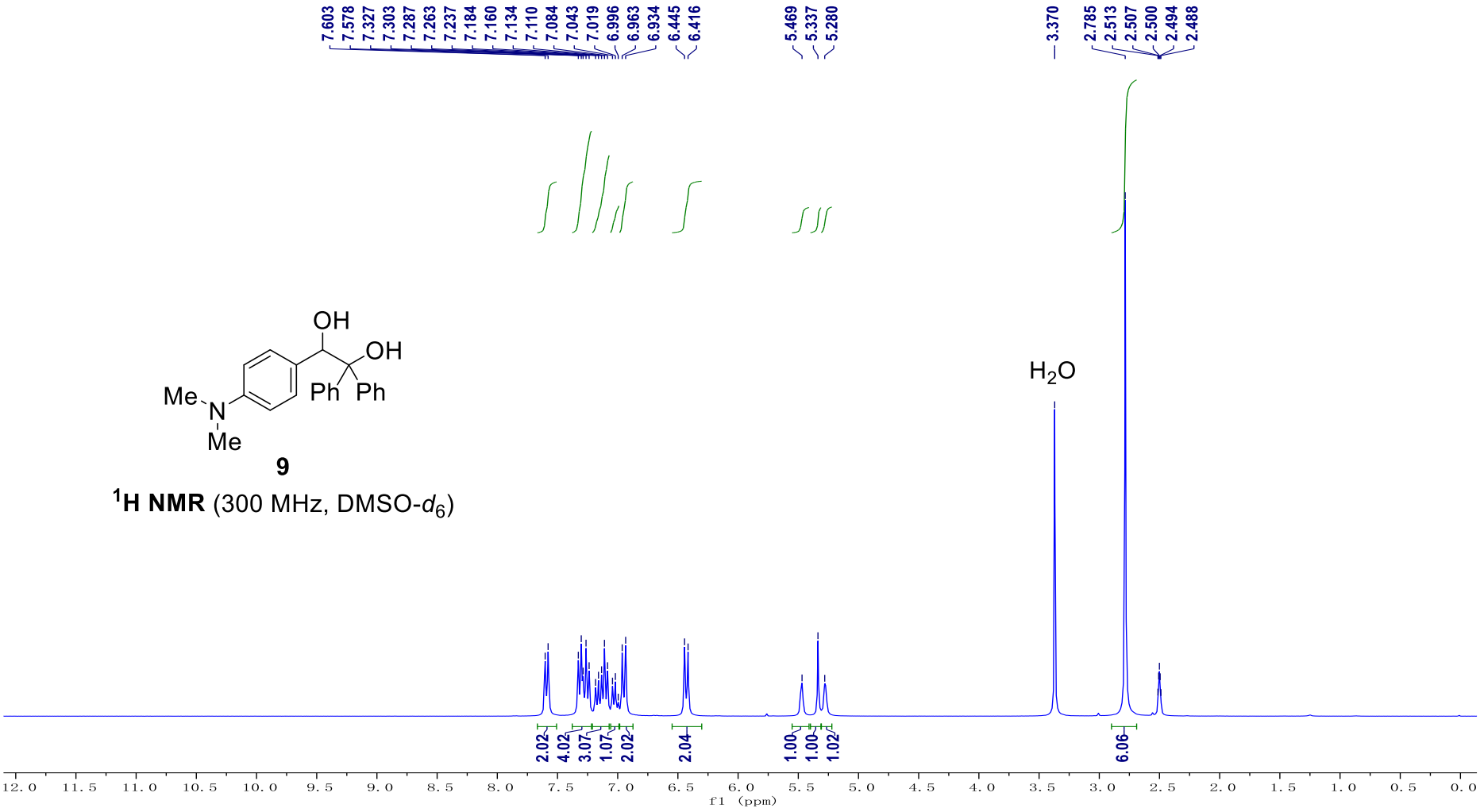
8

^{13}C NMR (100 MHz, $CDCl_3$)



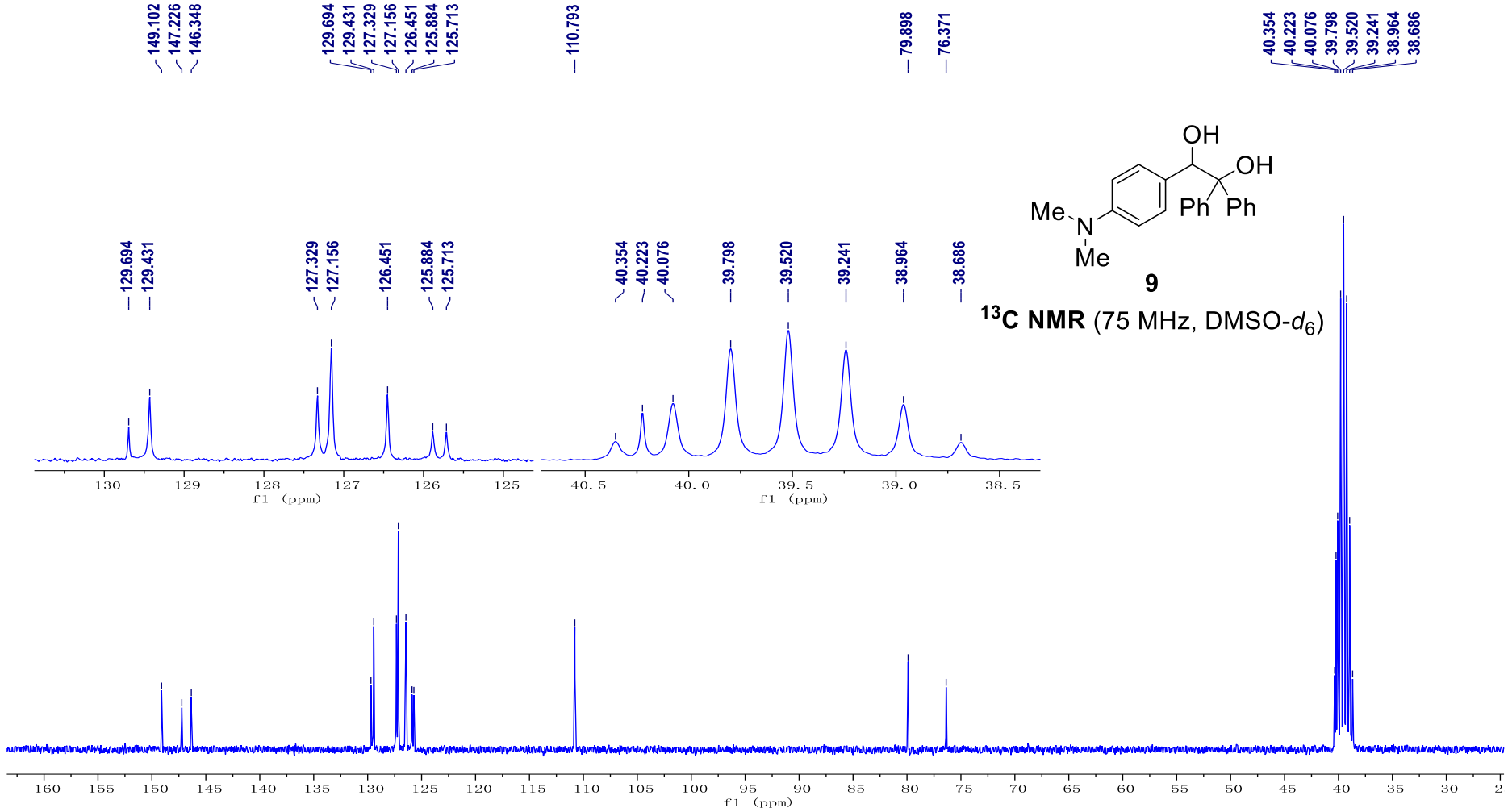
8

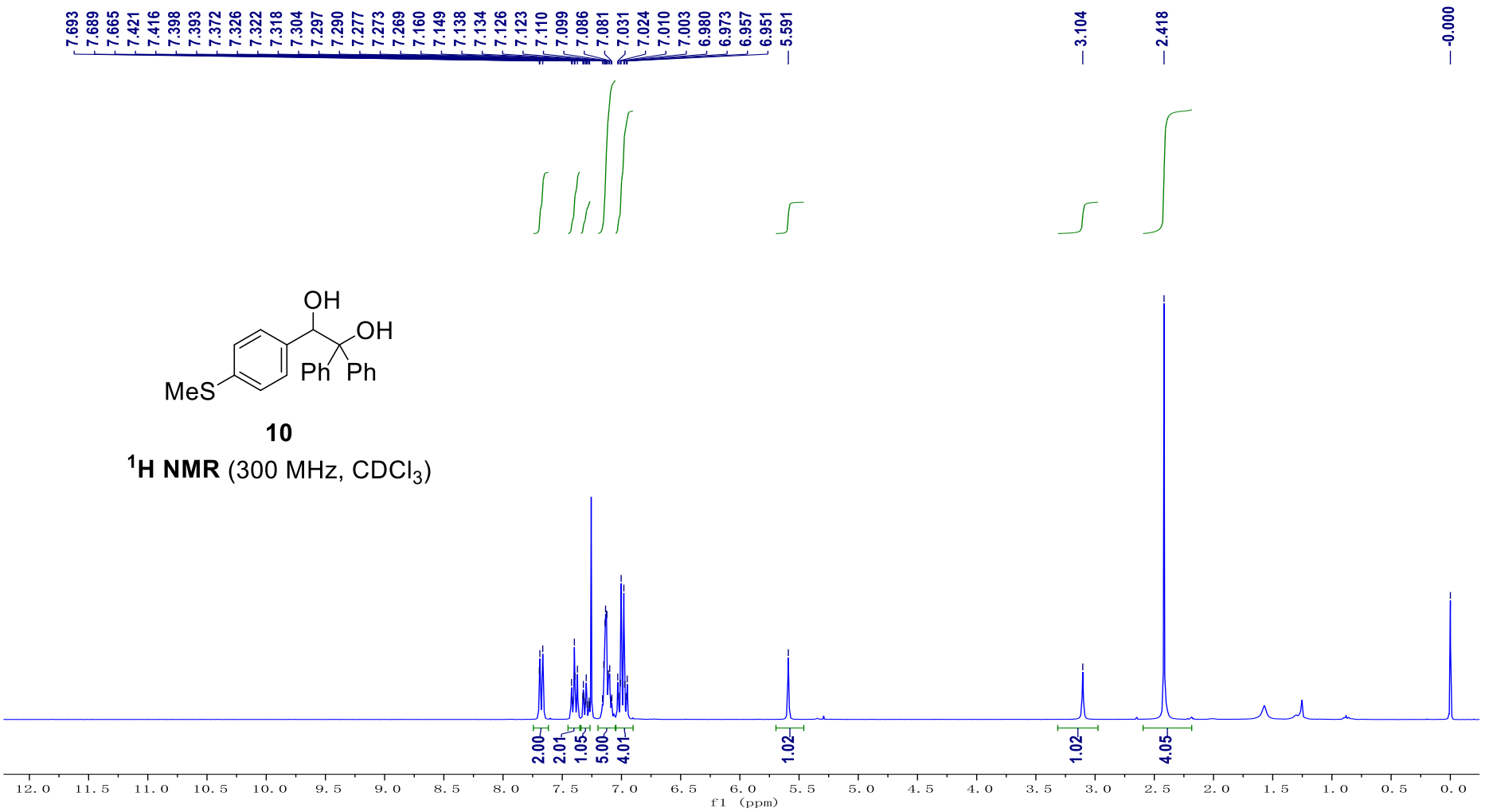
^{19}F NMR (376 MHz, $CDCl_3$)



9

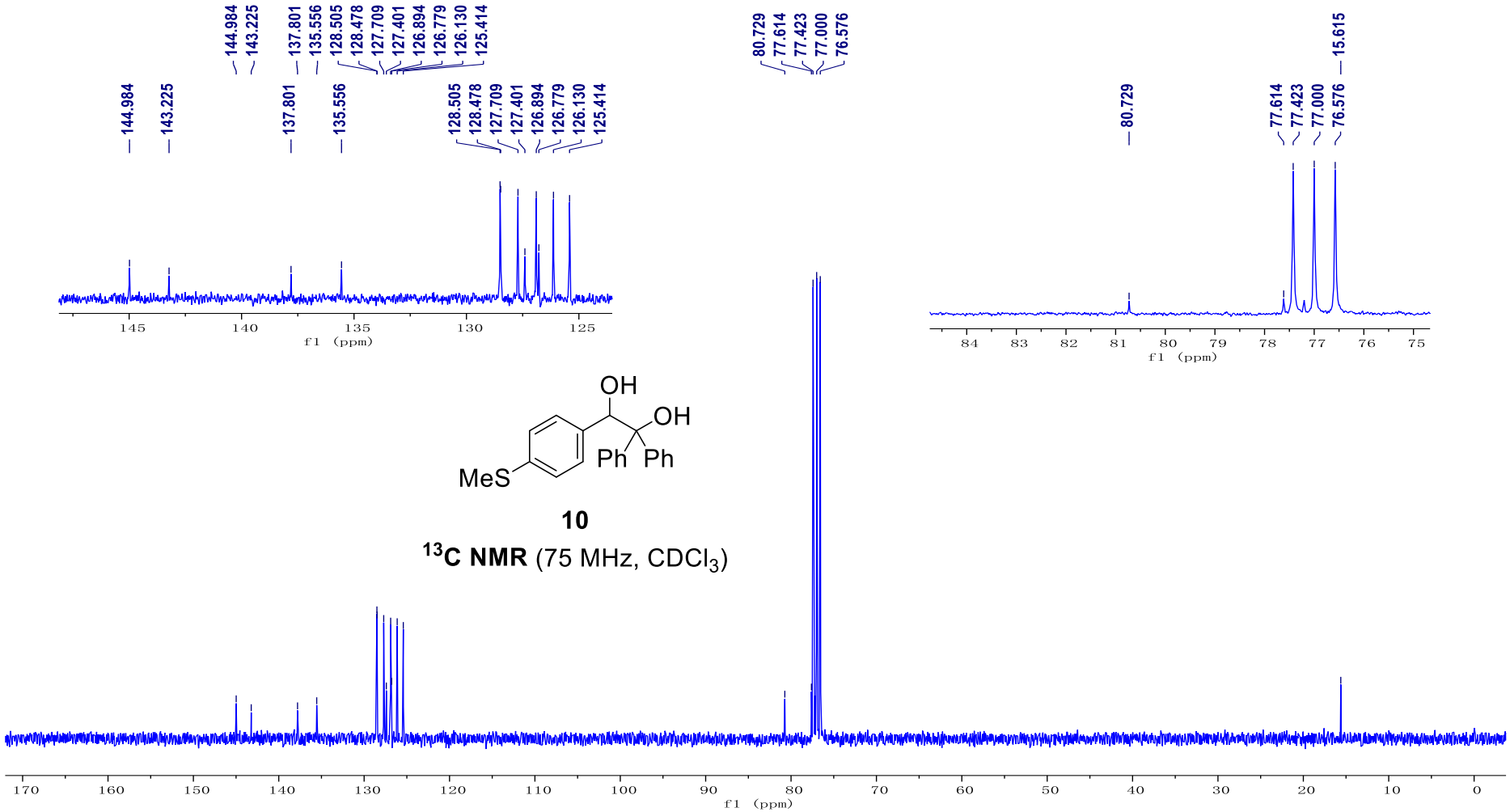
^{1}H NMR (300 MHz, DMSO-d_6)


149.102
147.226
146.348
129.694
129.431
127.329
127.156
126.451
125.884
125.713
110.793
79.898
76.371
40.354
40.223
40.076
39.798
39.520
39.241
38.964
38.686
OH
OH
Ph
Ph
Me
N
Me
9
13C NMR (75 MHz, DMSO-d6)
f1 (ppm)



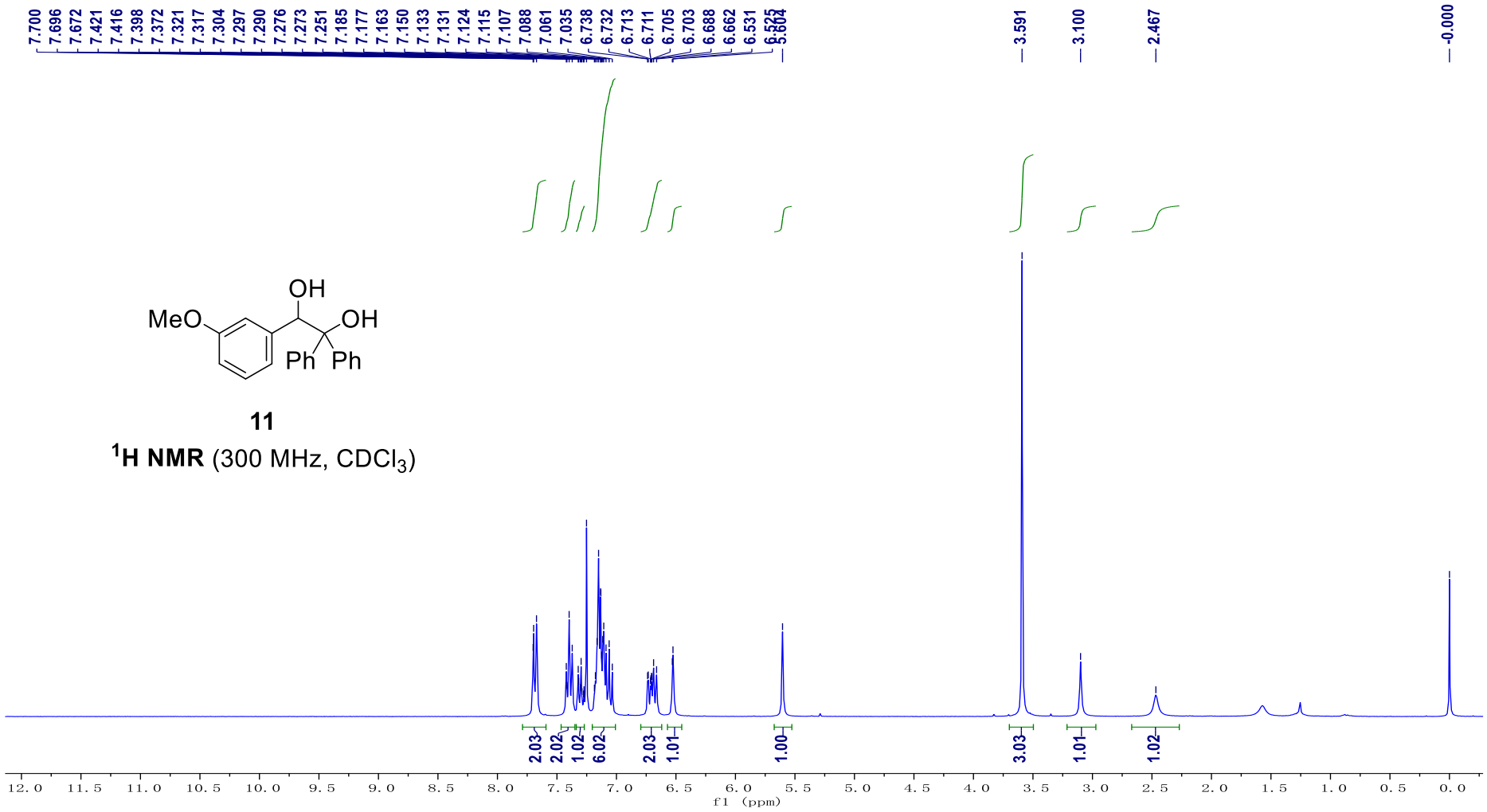
10

^{1}H NMR (300 MHz, $CDCl_3$)



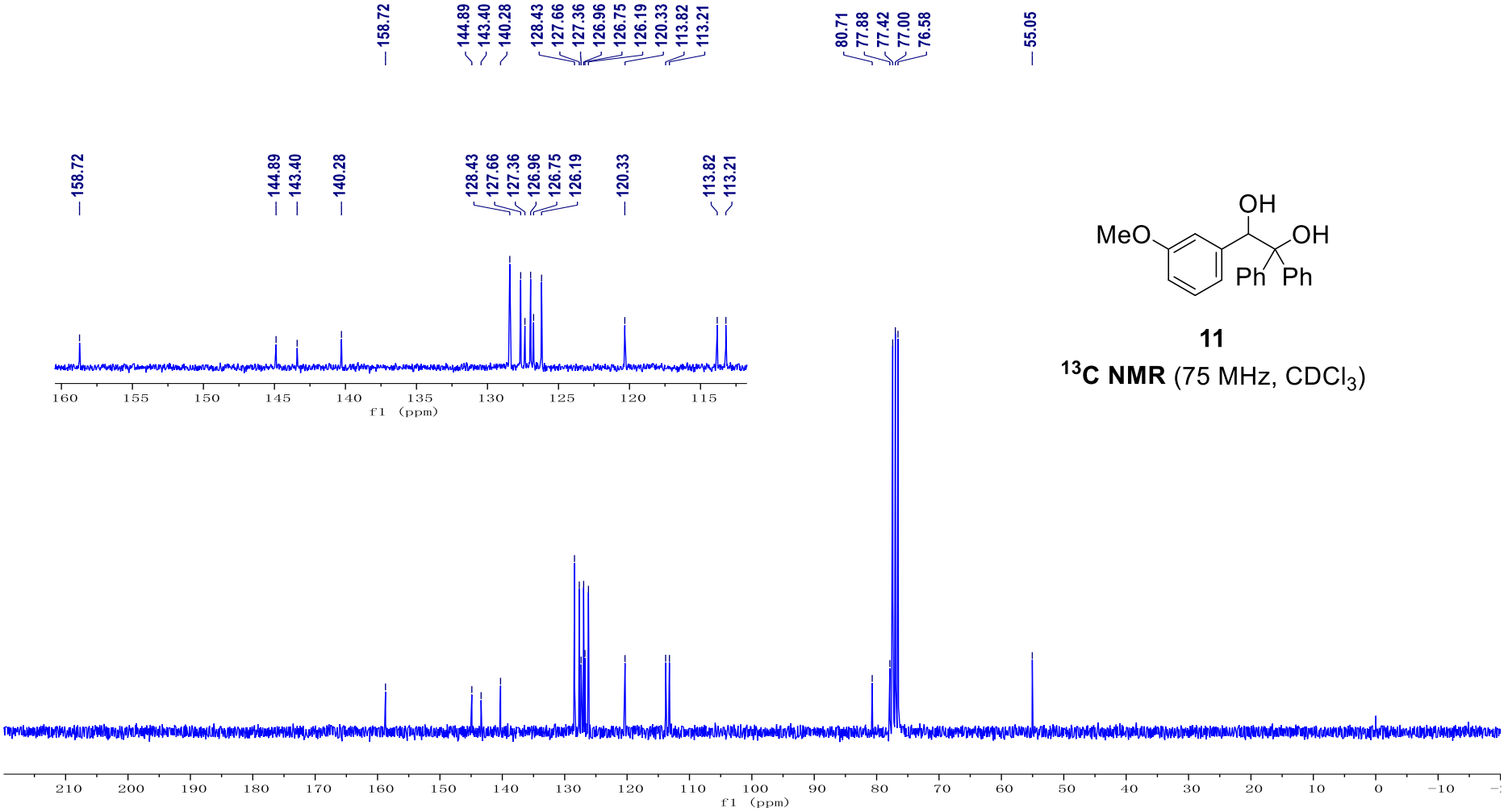
10

^{13}C NMR (75 MHz, $CDCl_3$)



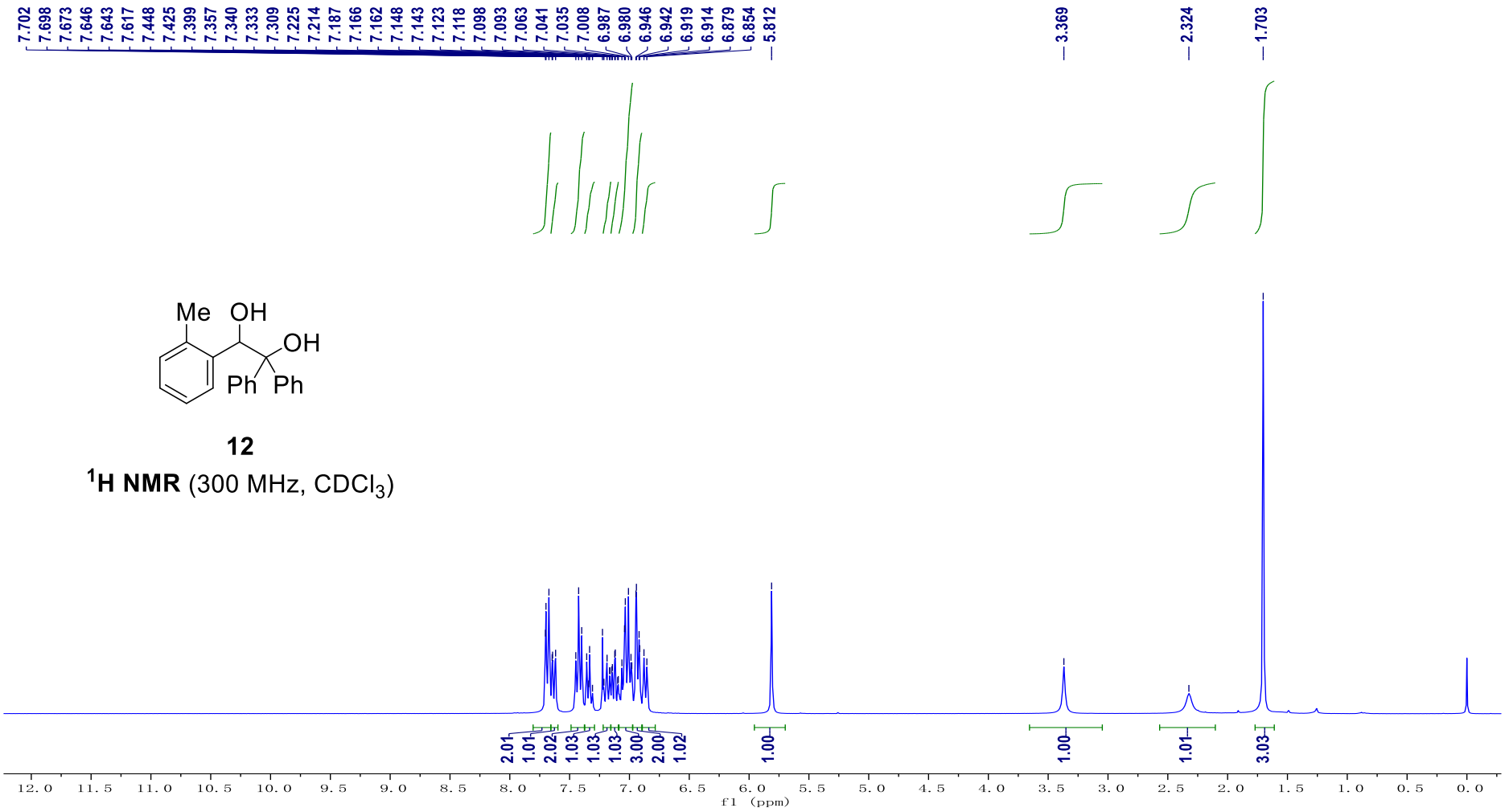
11

^{1}H NMR (300 MHz, $CDCl_3$)



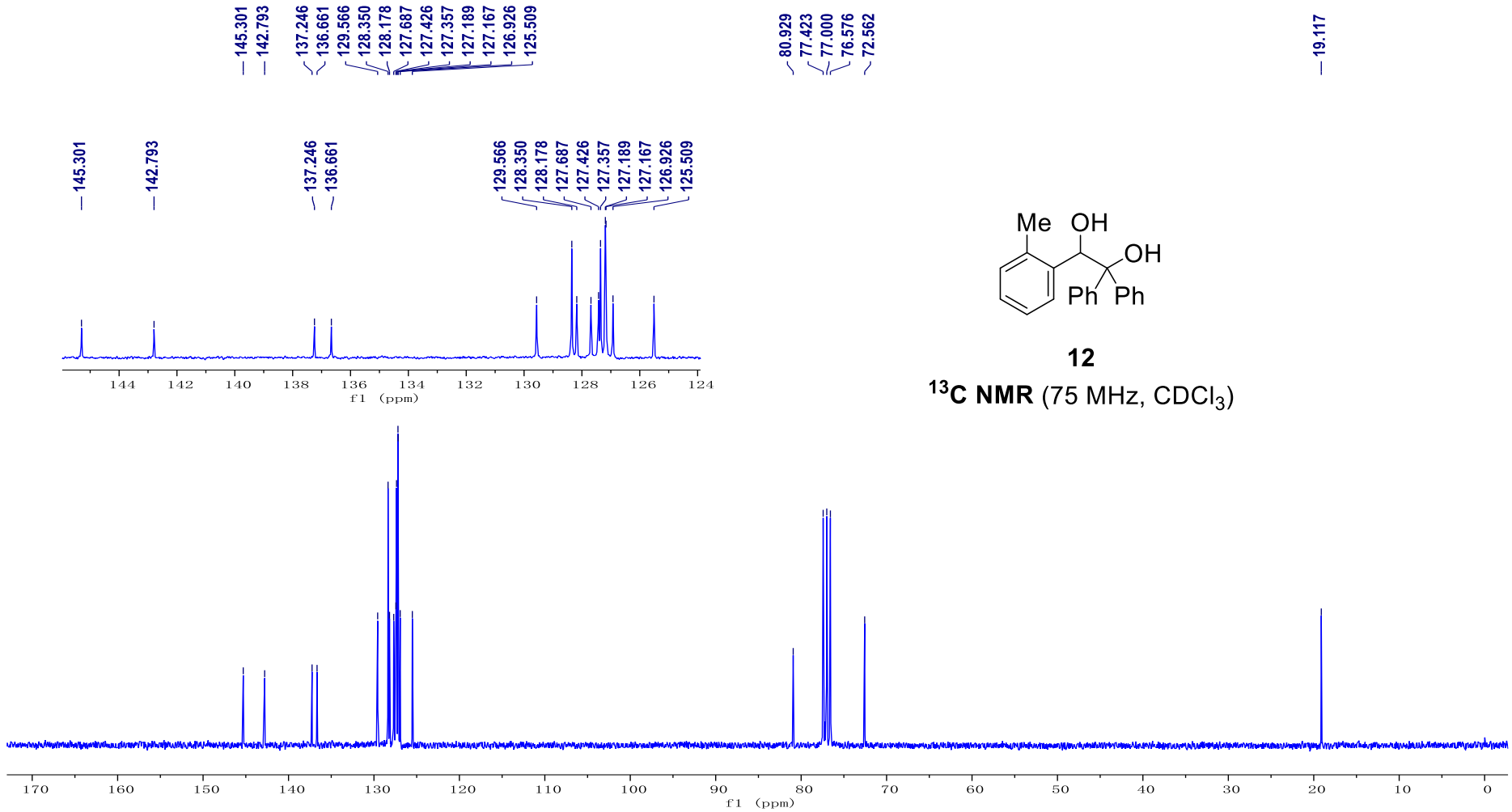
11

^{13}C NMR (75 MHz, $CDCl_3$)



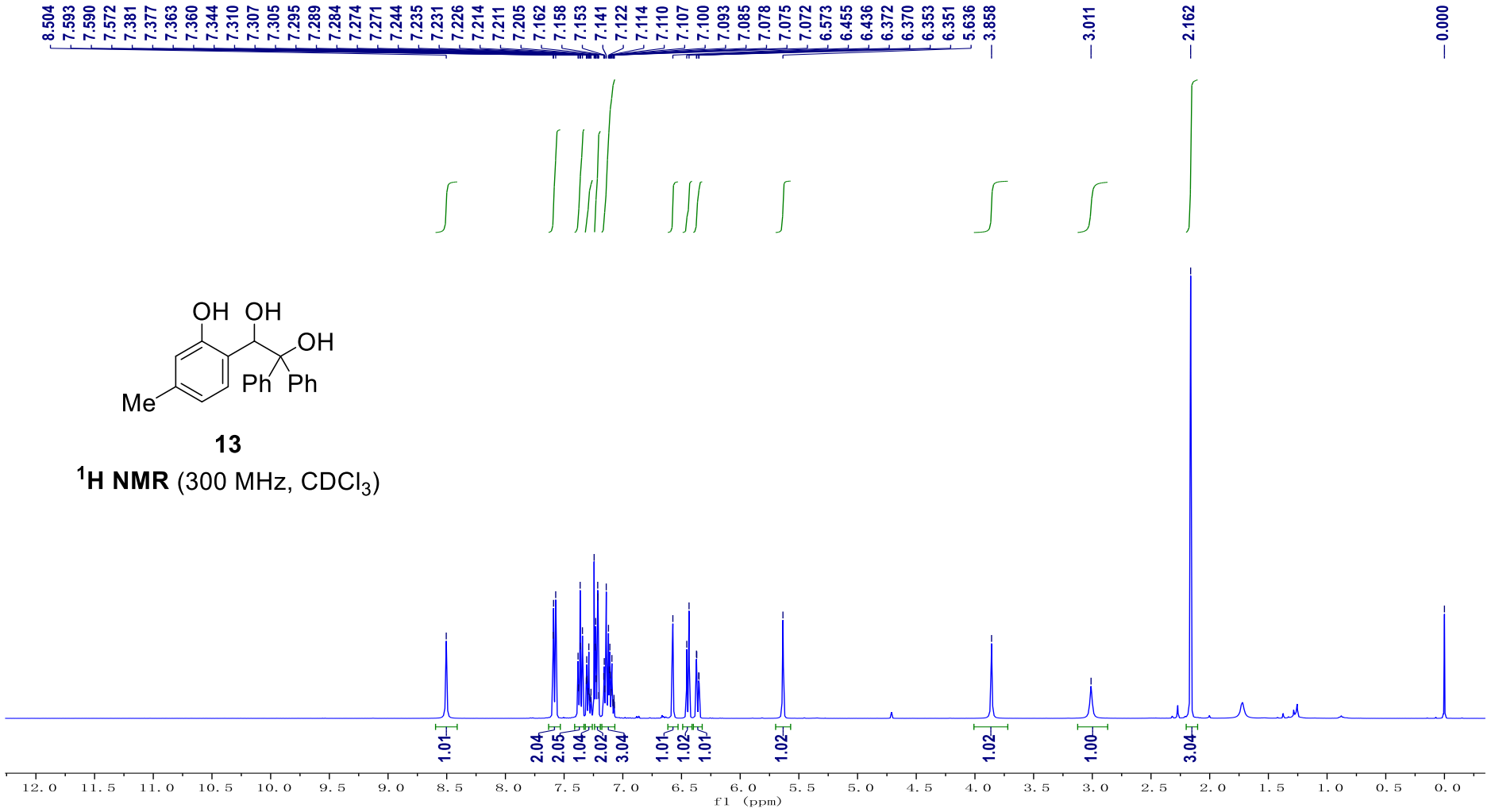
12

^{1}H NMR (300 MHz, $CDCl_3$)



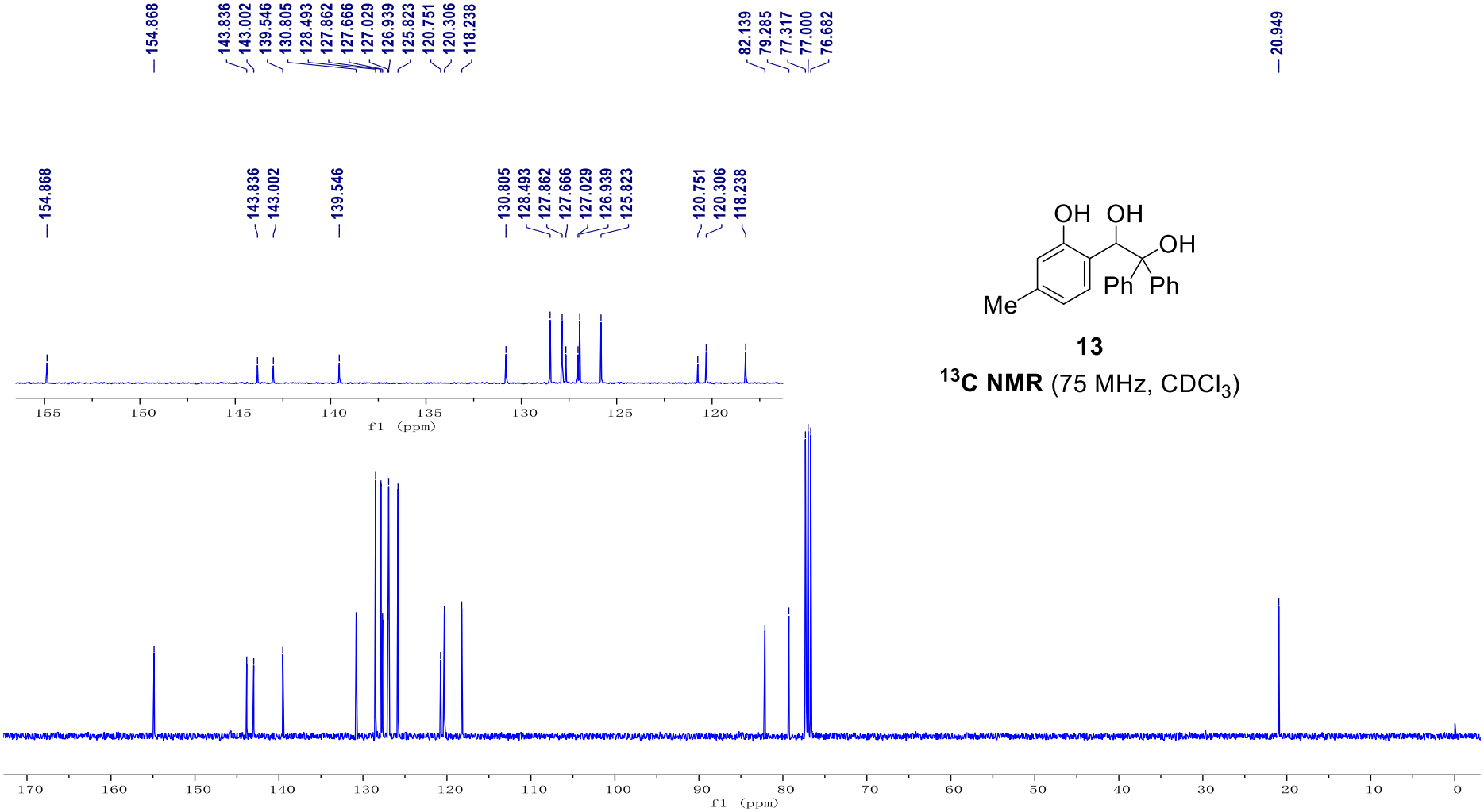
12

^{13}C NMR (75 MHz, $CDCl_3$)



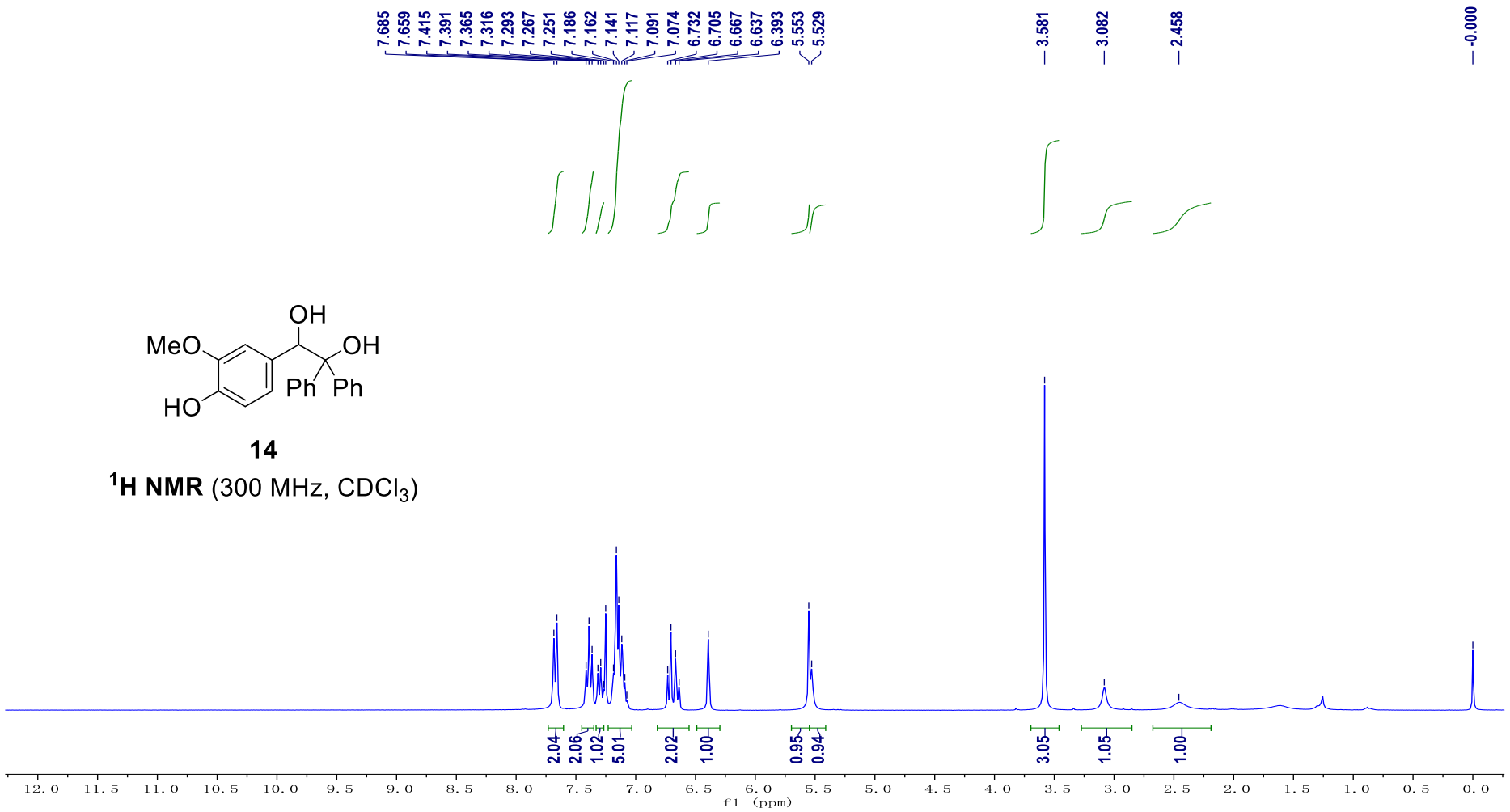
13

^{1}H NMR (300 MHz, $CDCl_3$)



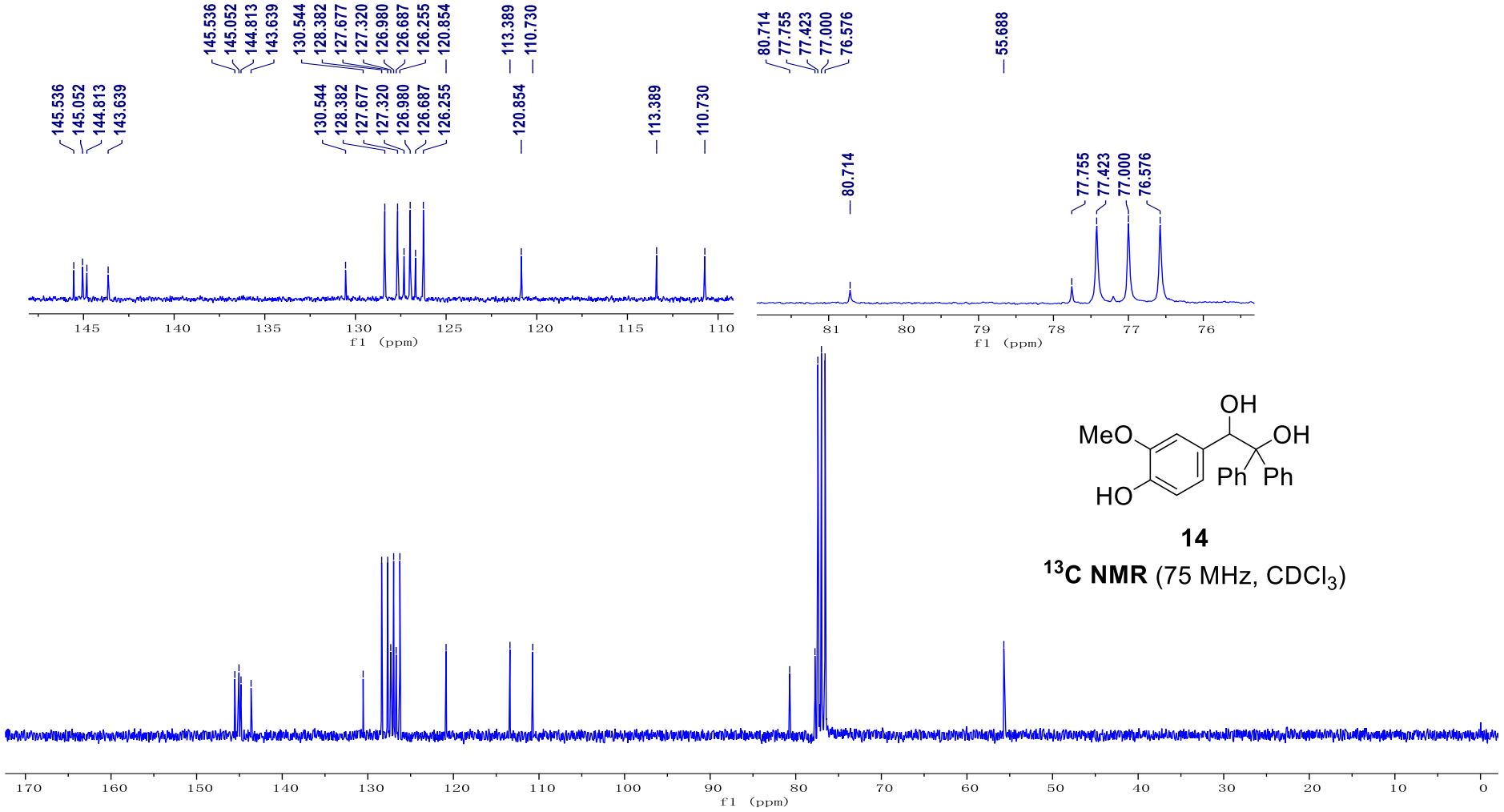
13

^{13}C NMR (75 MHz, $CDCl_3$)



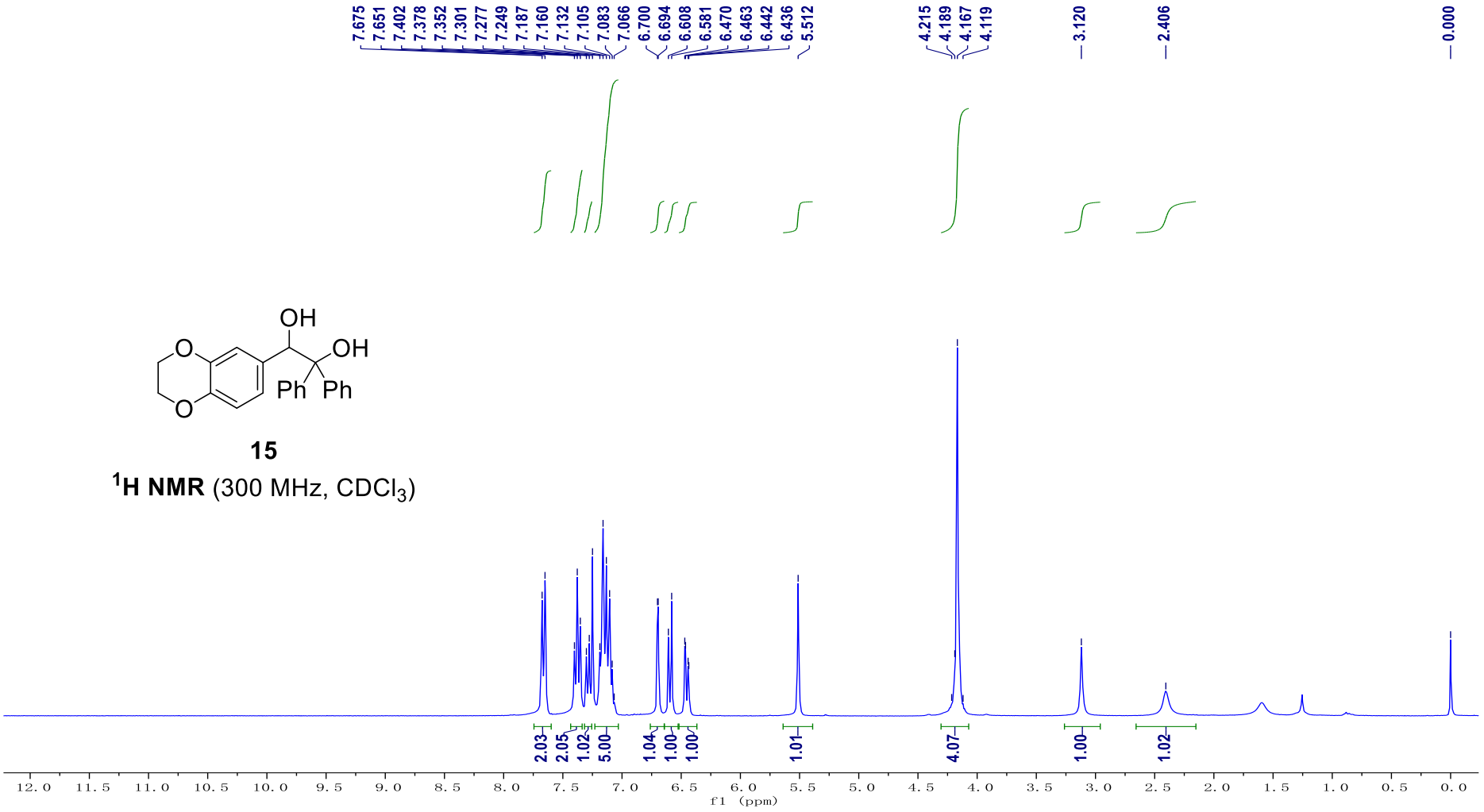
14

^{1}H NMR (300 MHz, $CDCl_3$)



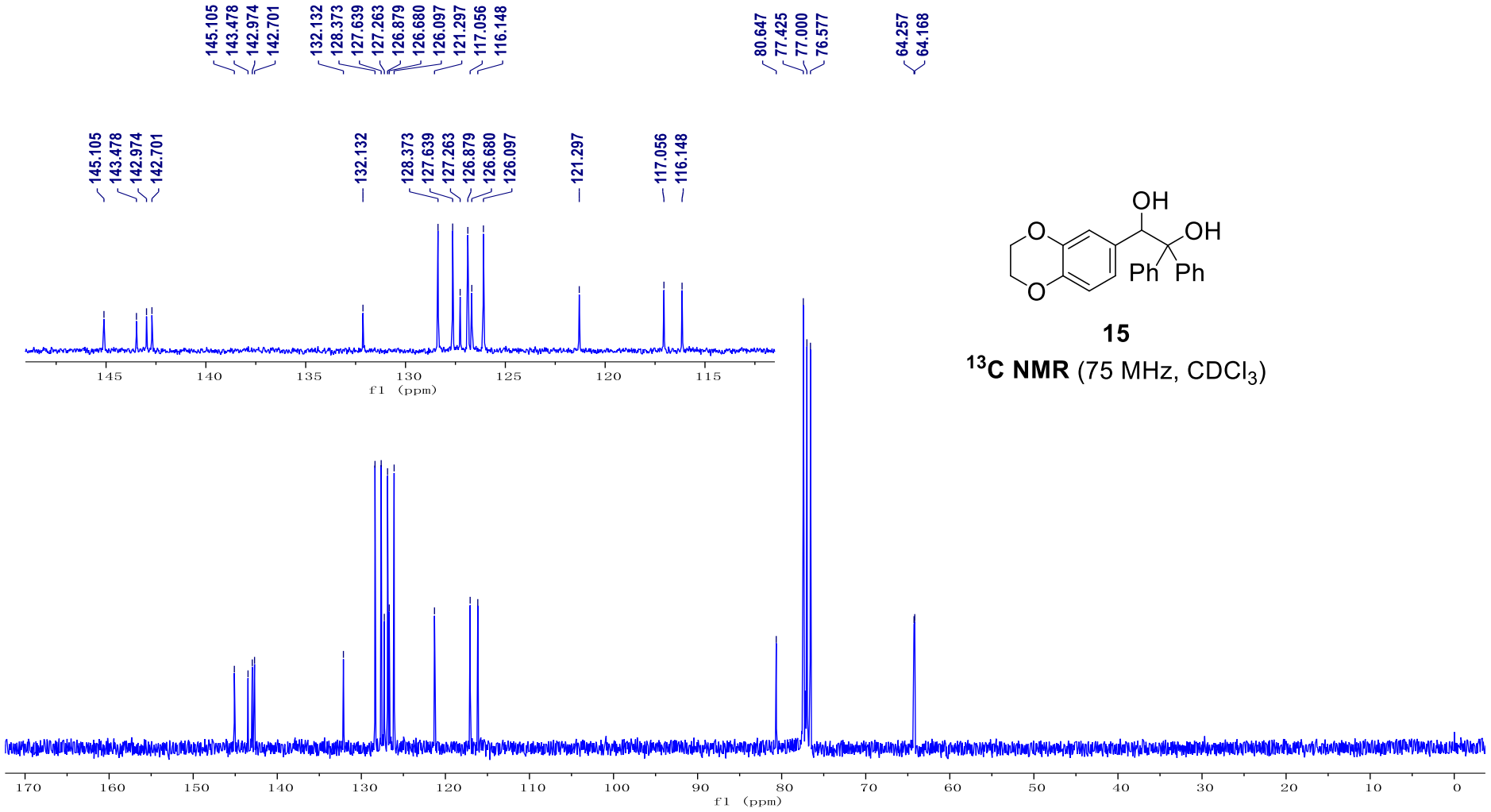
14

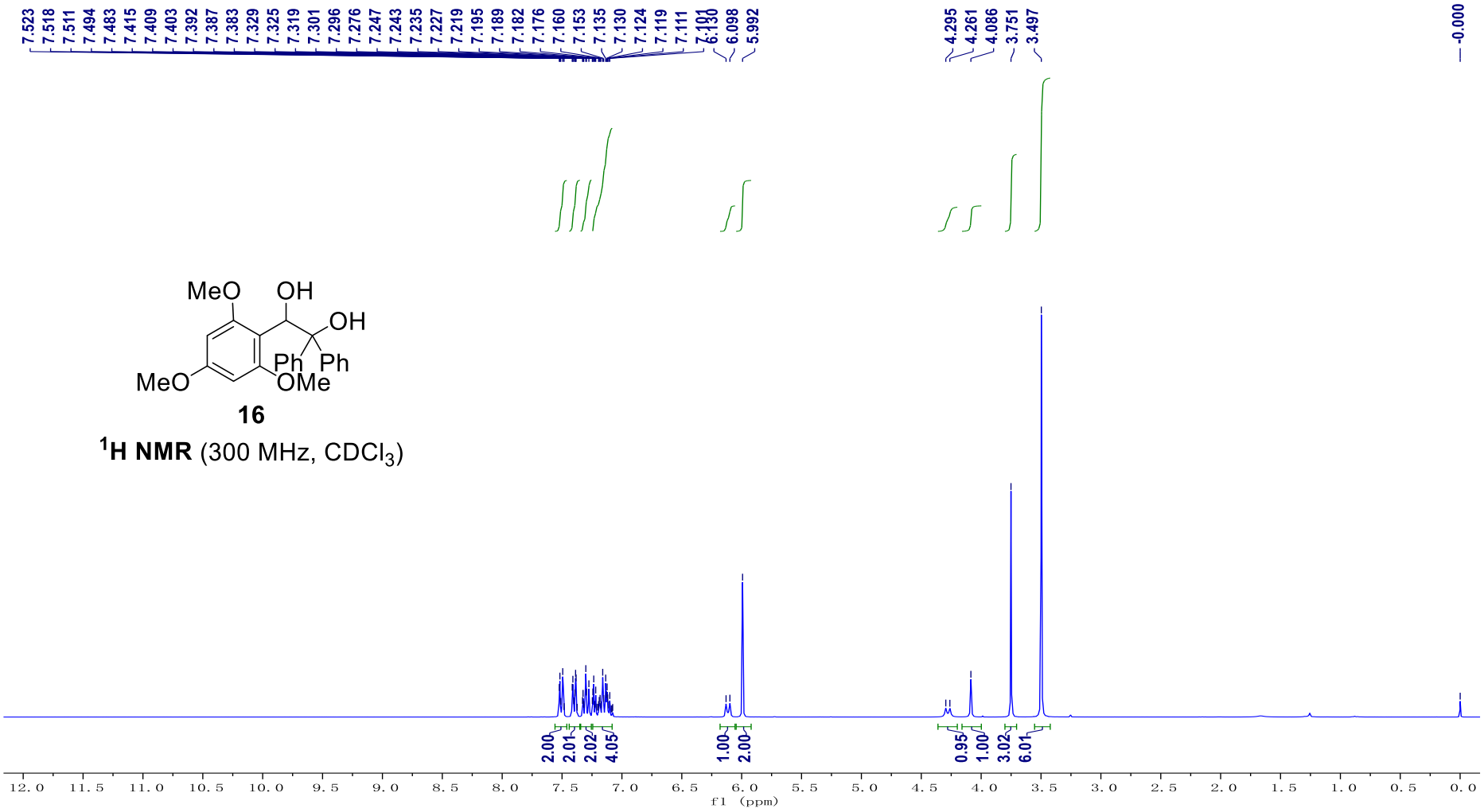
^{13}C NMR (75 MHz, $CDCl_3$)



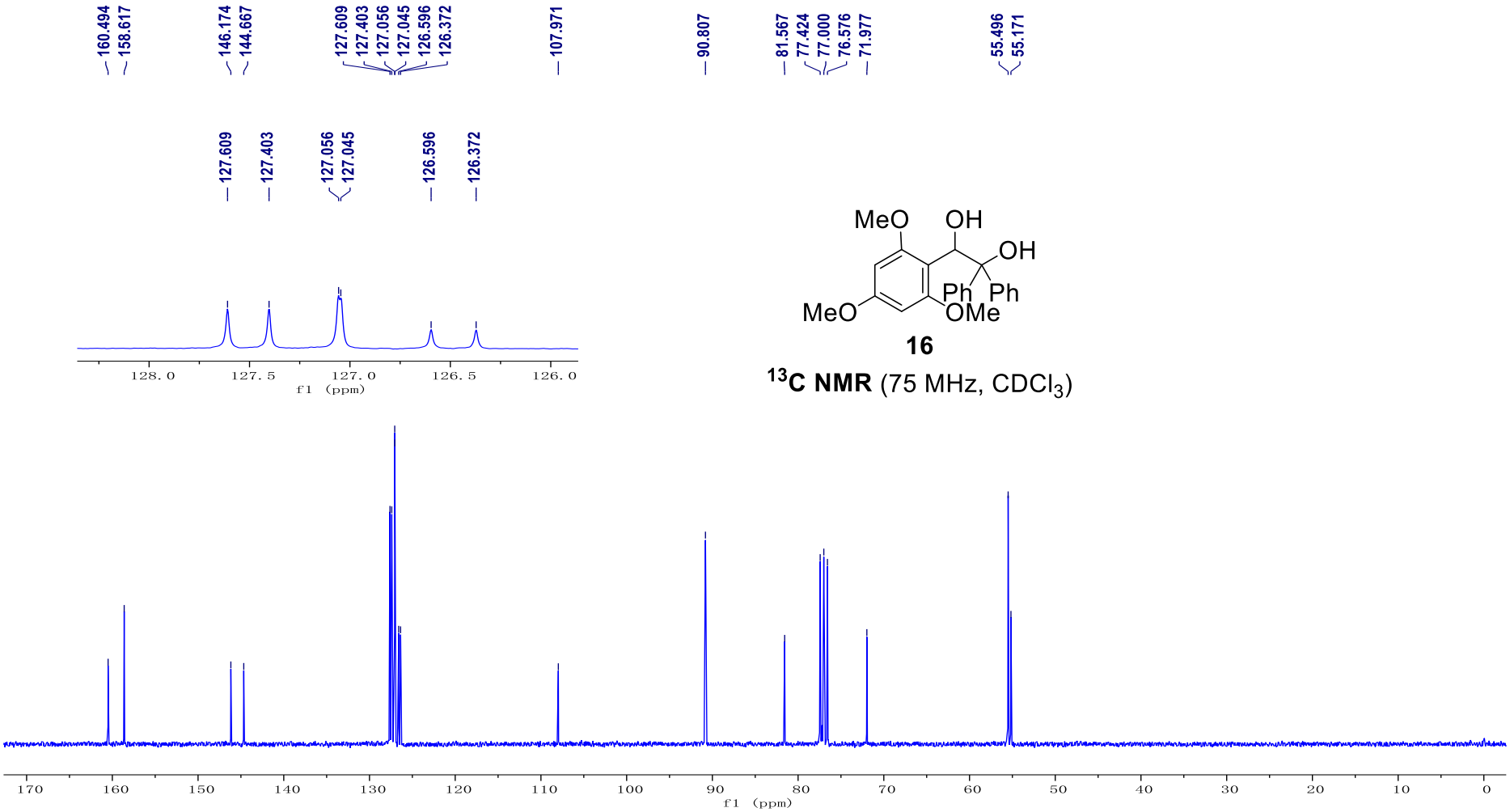
15

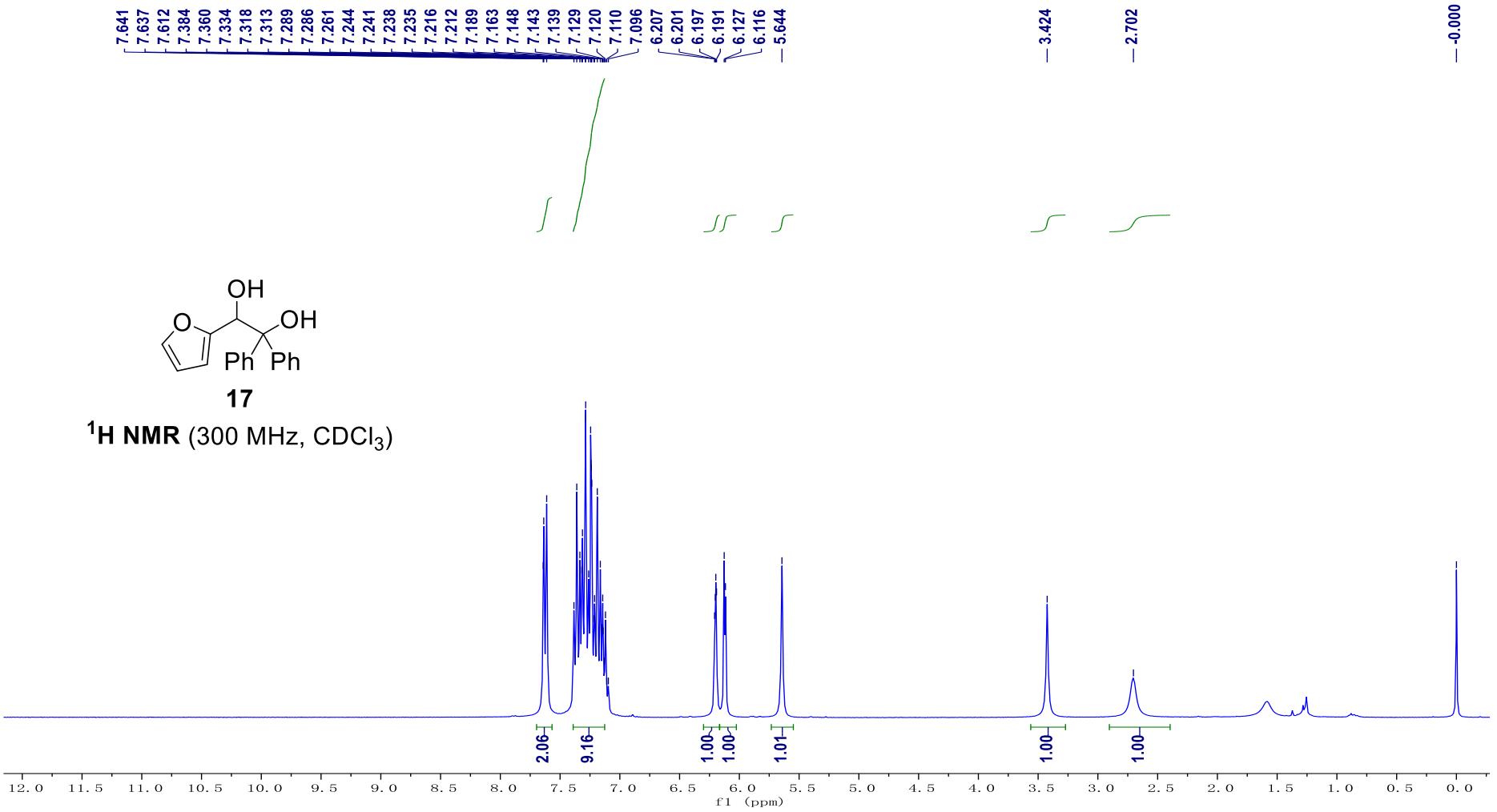
^{1}H NMR (300 MHz, $CDCl_3$)


145.105
143.478
142.974
142.701
132.132
128.373
127.639
127.263
126.879
126.680
126.097
121.297
117.056
116.148
80.647
77.425
77.000
76.577
64.257
64.168
OH
OH
O
O
Ph
Ph
15
13C NMR (75 MHz, CDCl3)
f1 (ppm)



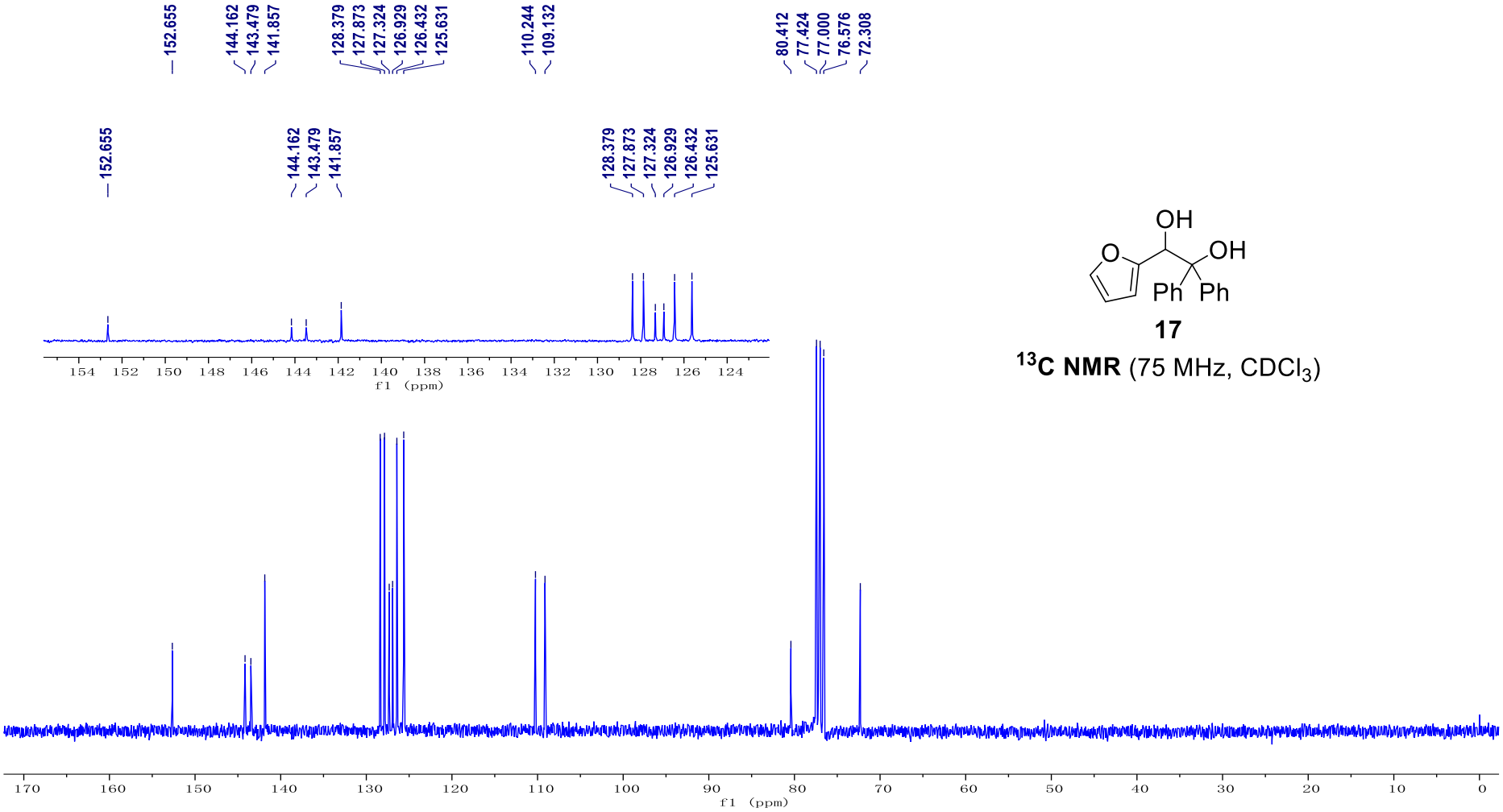
^{1}H NMR (300 MHz, $CDCl_3$)

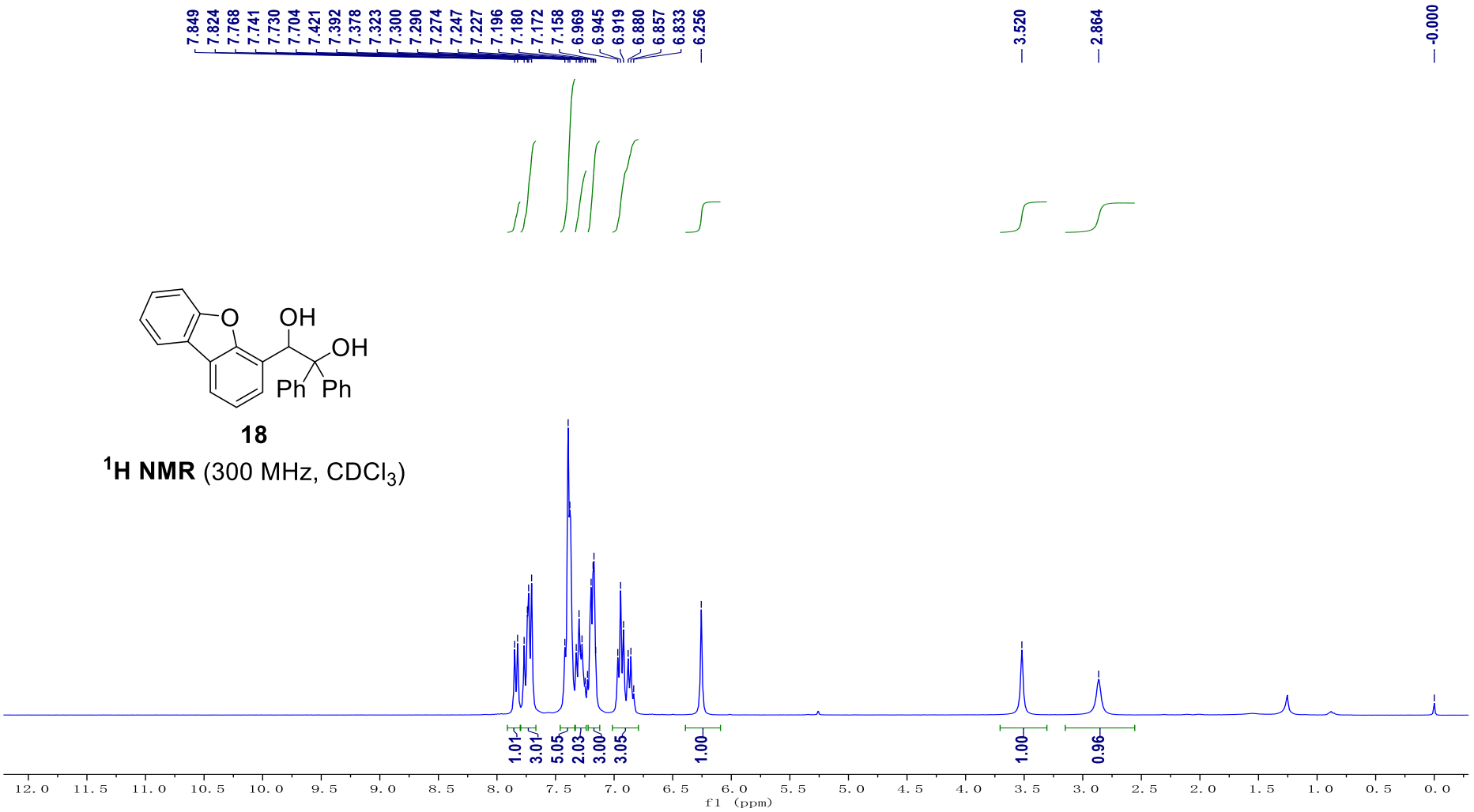

160.494
158.617
146.174
144.667
127.609
127.403
127.056
127.045
126.596
126.372
107.971
90.807
81.567
77.424
77.000
76.576
71.977
55.496
55.171
128.0
127.5
127.0
126.5
126.0
f1 (ppm)
MeO
OH
OH
Ph
Ph
MeO
OMe
16
13C NMR (75 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



17

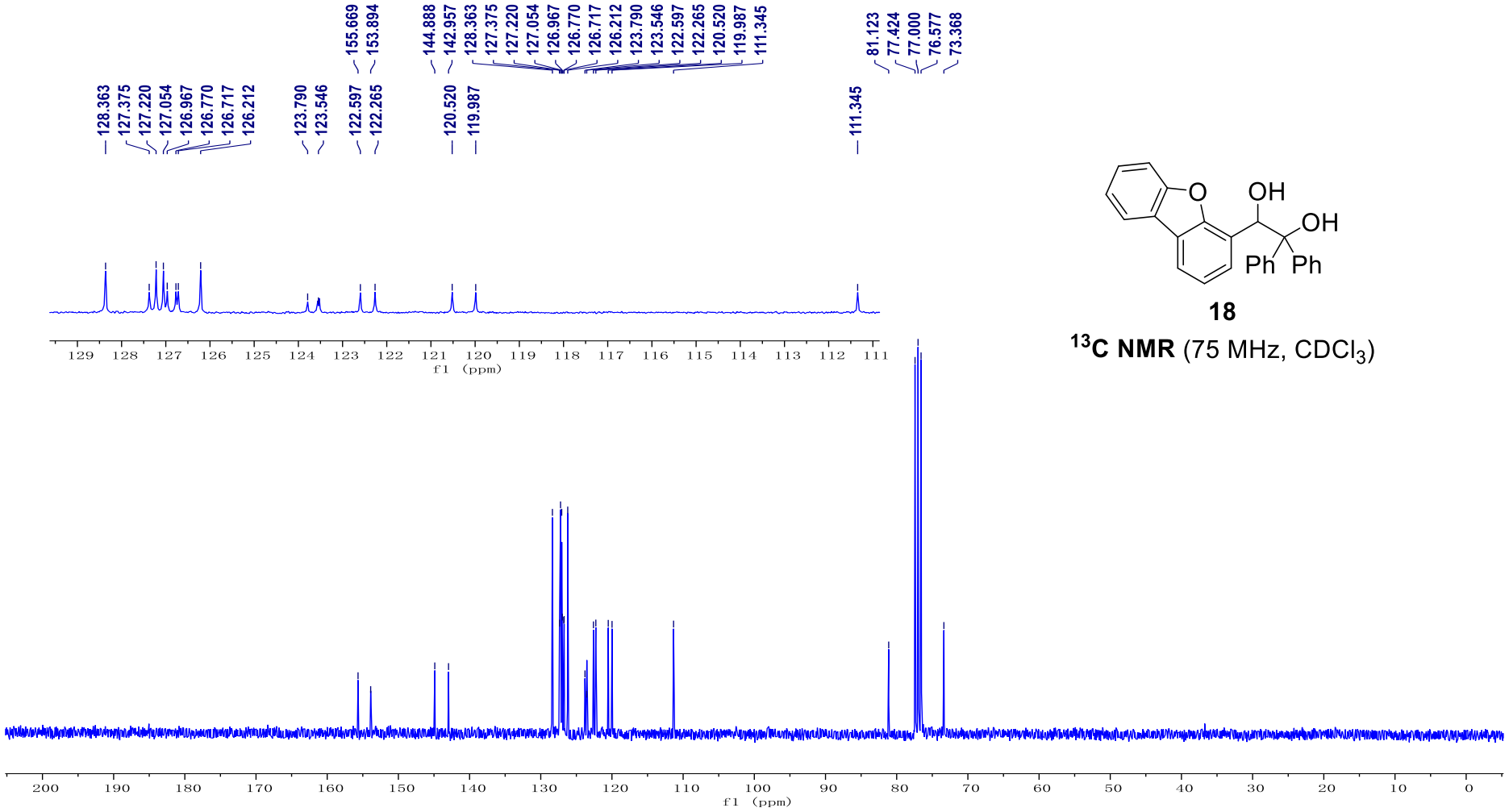
^{1}H NMR (300 MHz, $CDCl_3$)


152.655
144.162
143.479
141.857
128.379
127.873
127.324
126.929
126.432
125.631
110.244
109.132
80.412
77.424
77.000
76.576
72.308
f1 (ppm)
OH
O
OH
Ph
Ph
17
13C NMR (75 MHz, CDCl3)



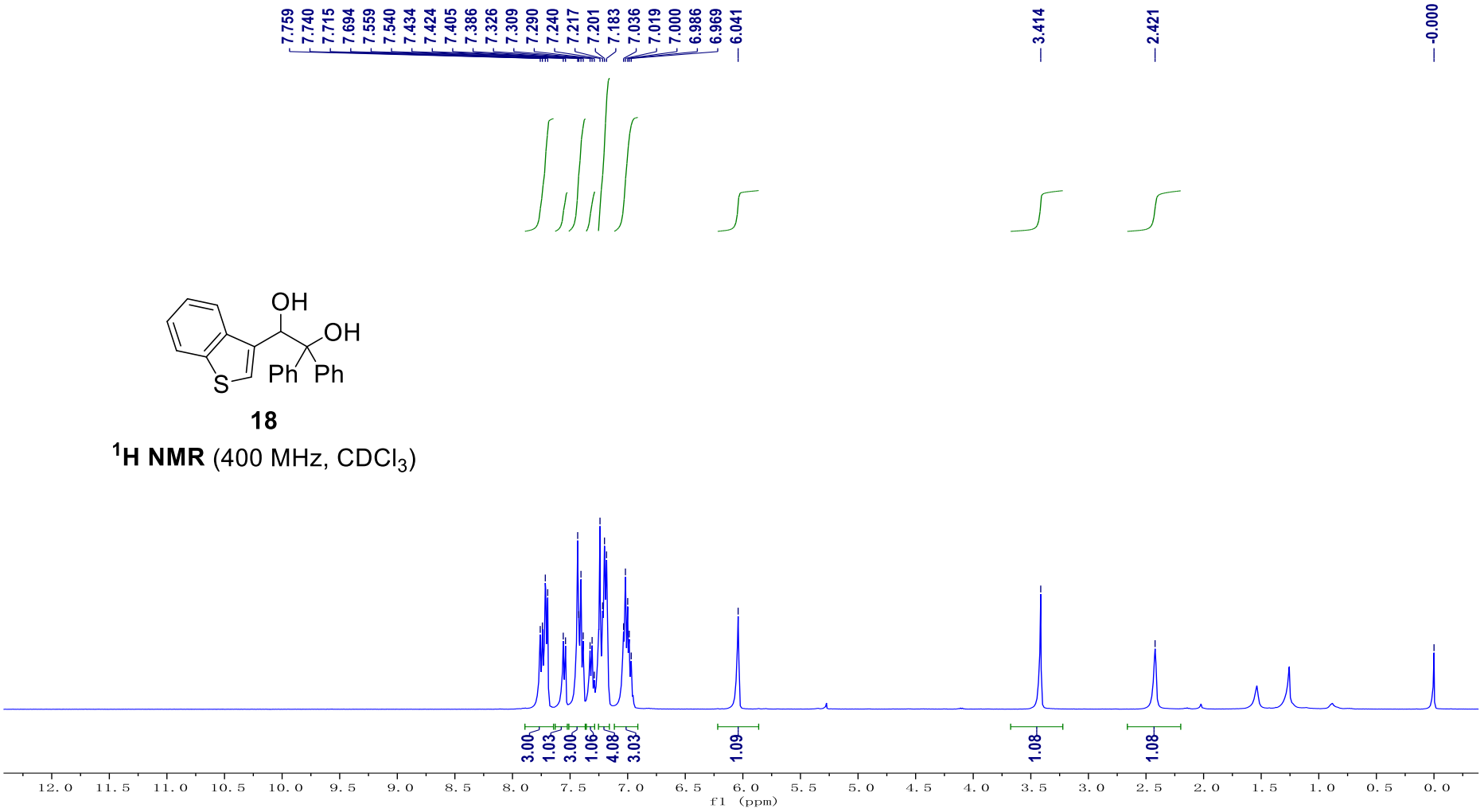
18

^{1}H NMR (300 MHz, $CDCl_3$)



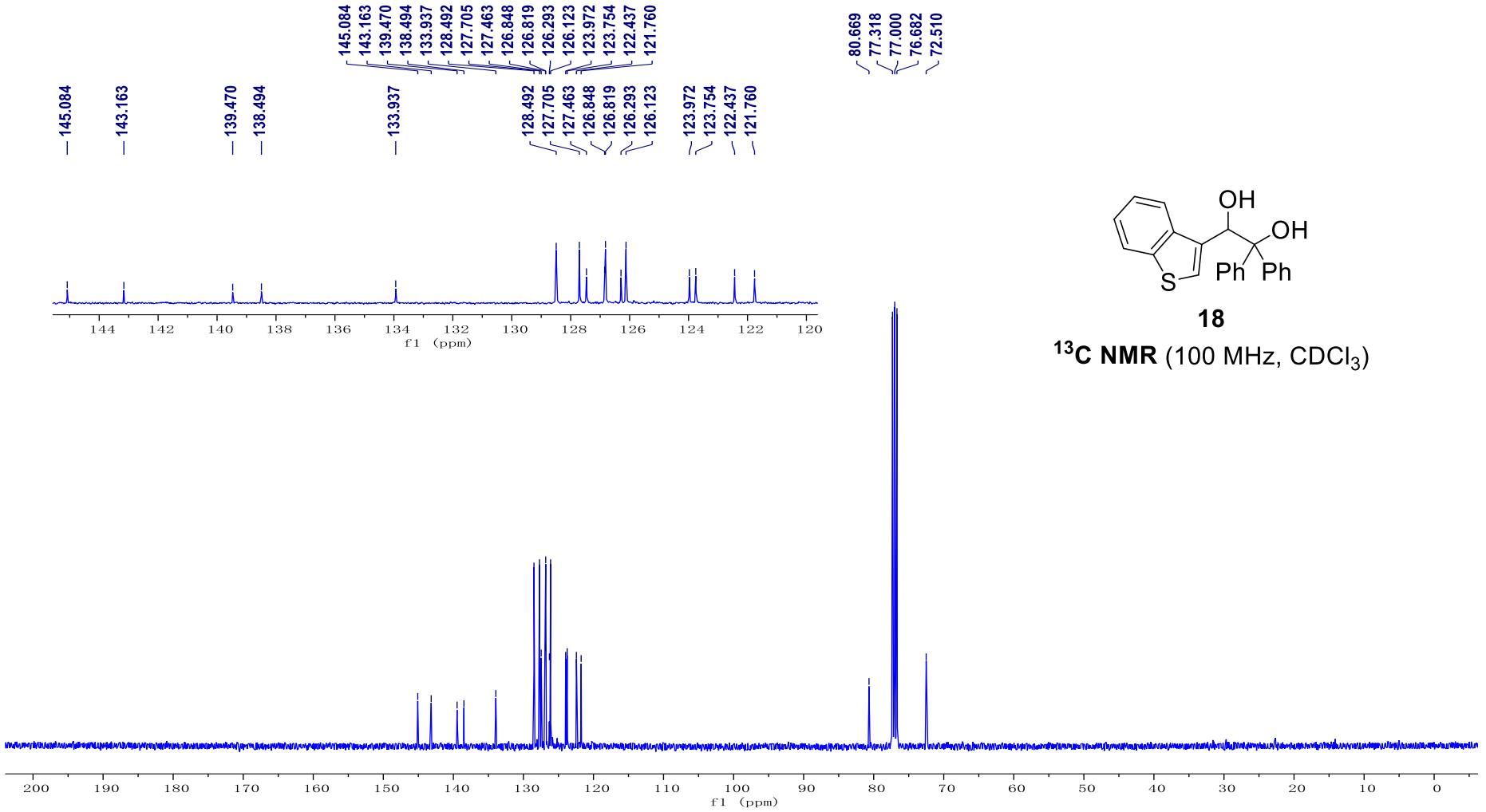
18

^{13}C NMR (75 MHz, $CDCl_3$)



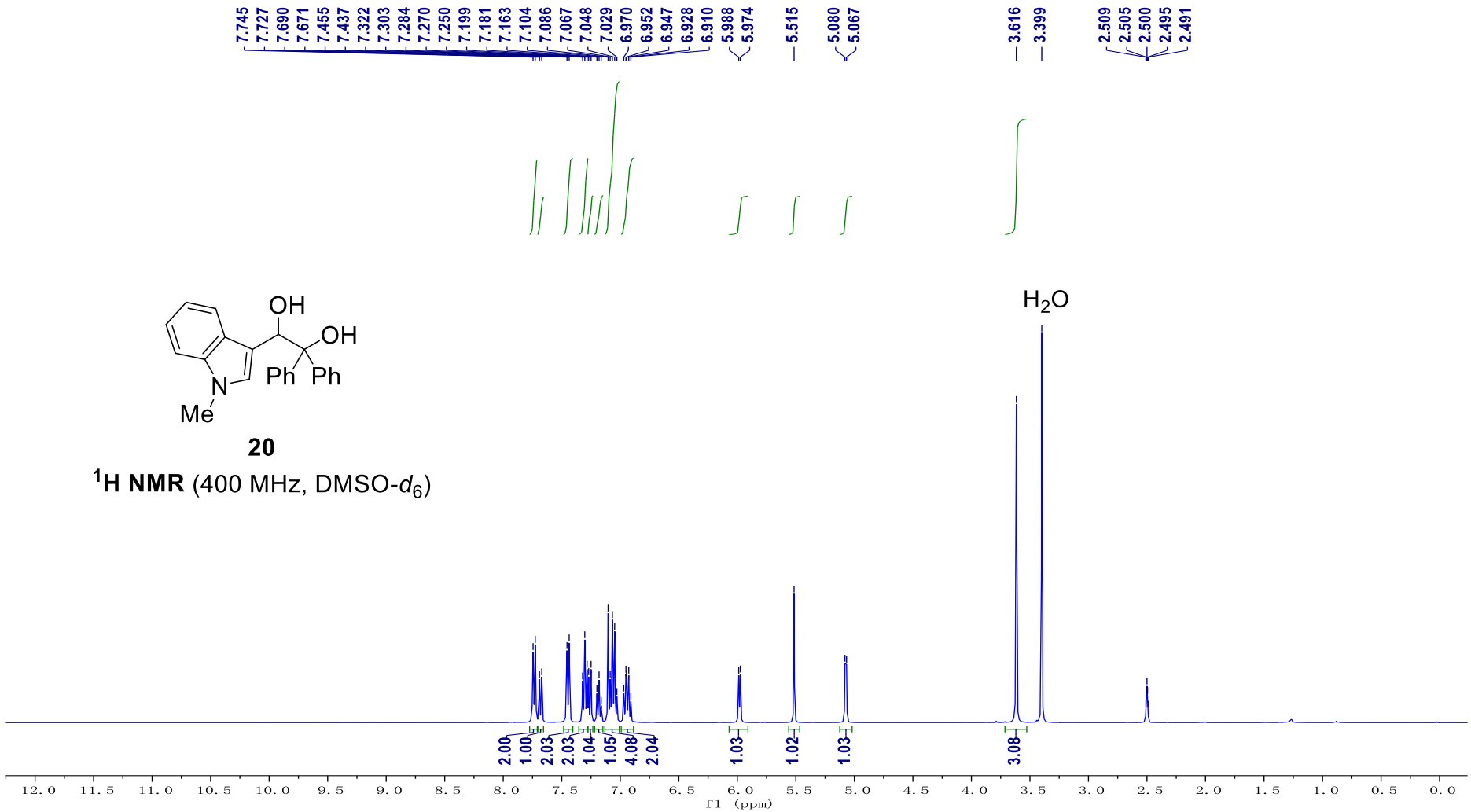
18

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



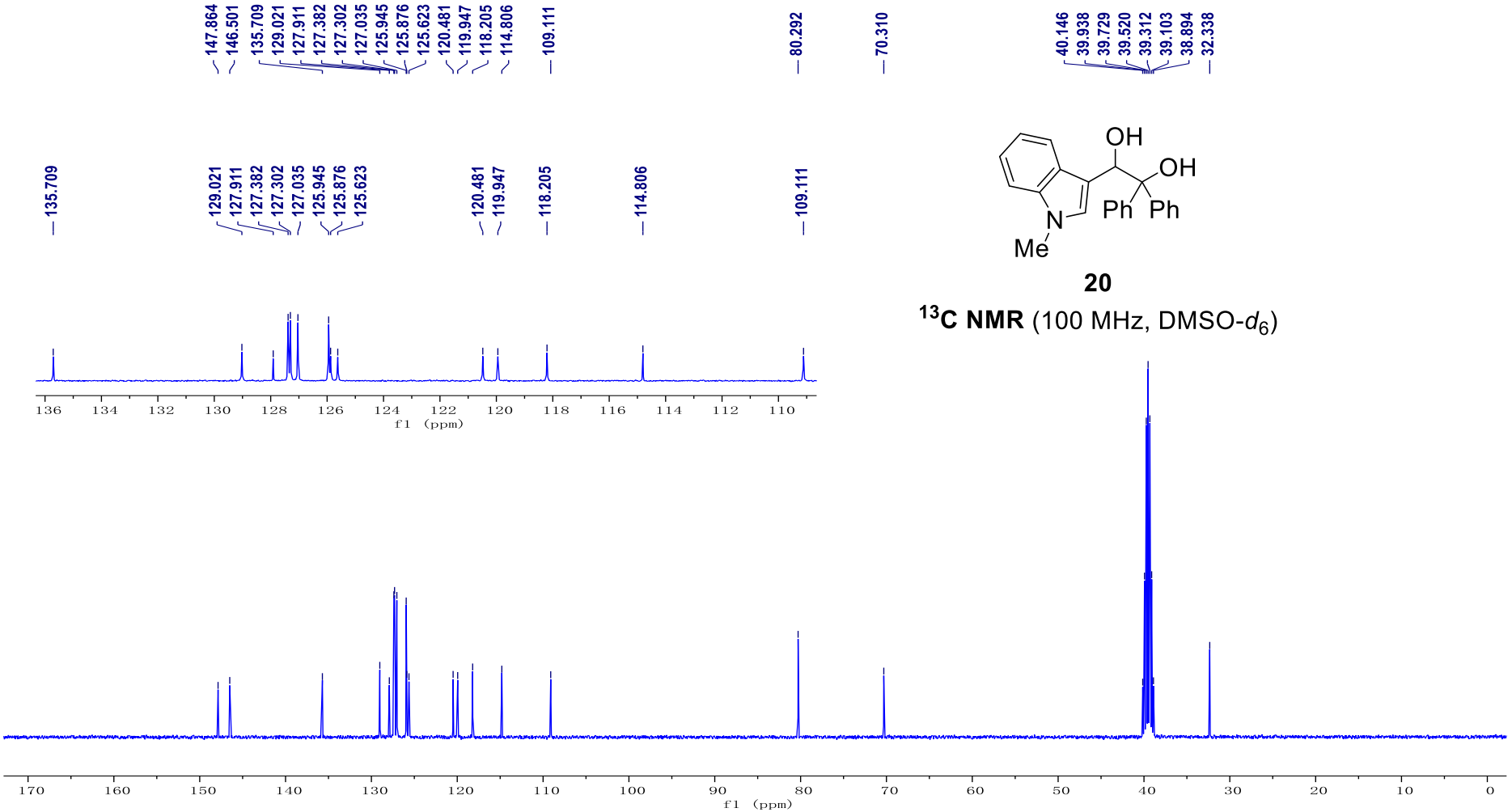
18

^{13}C NMR (100 MHz, $CDCl_3$)



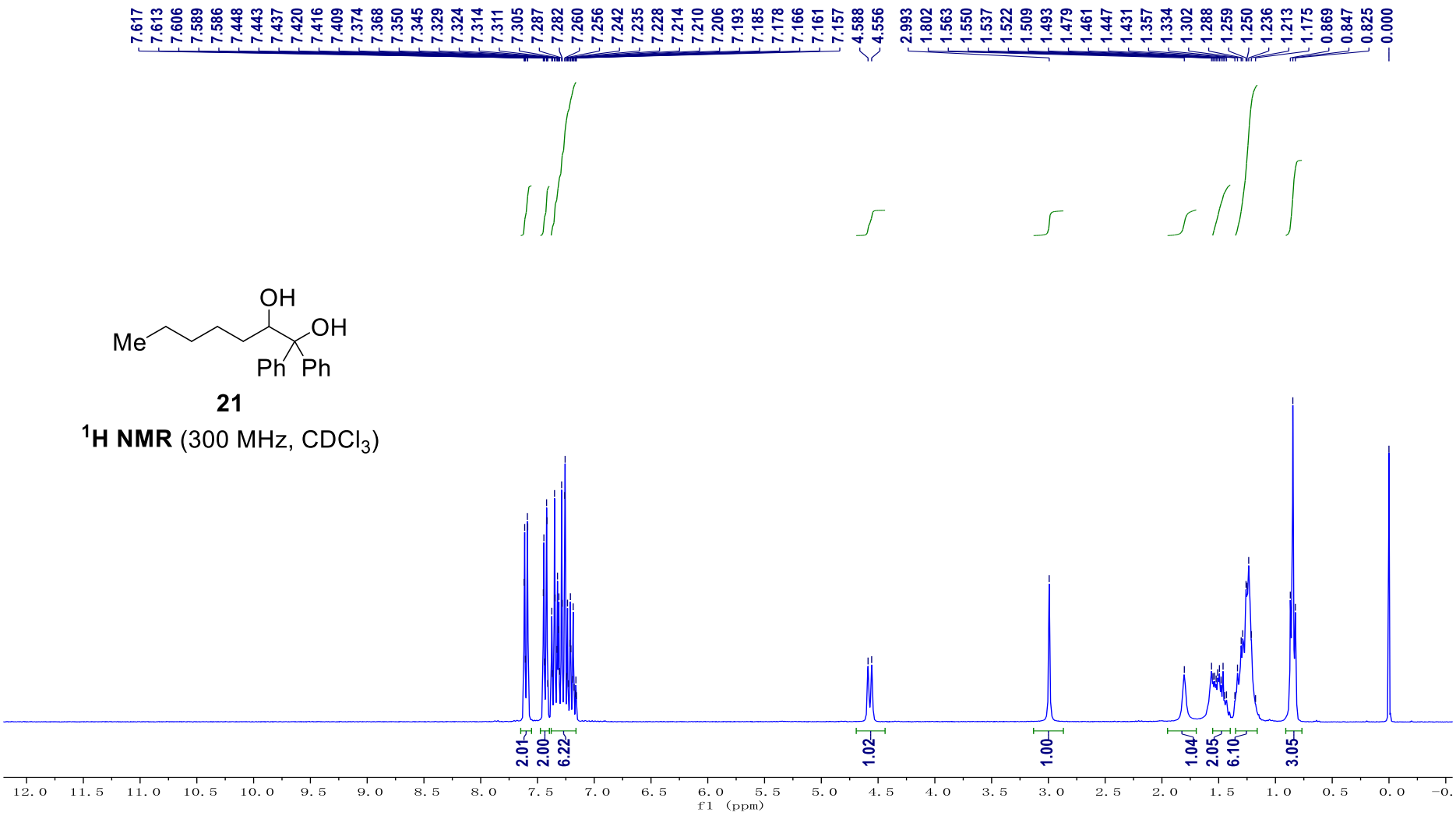
20

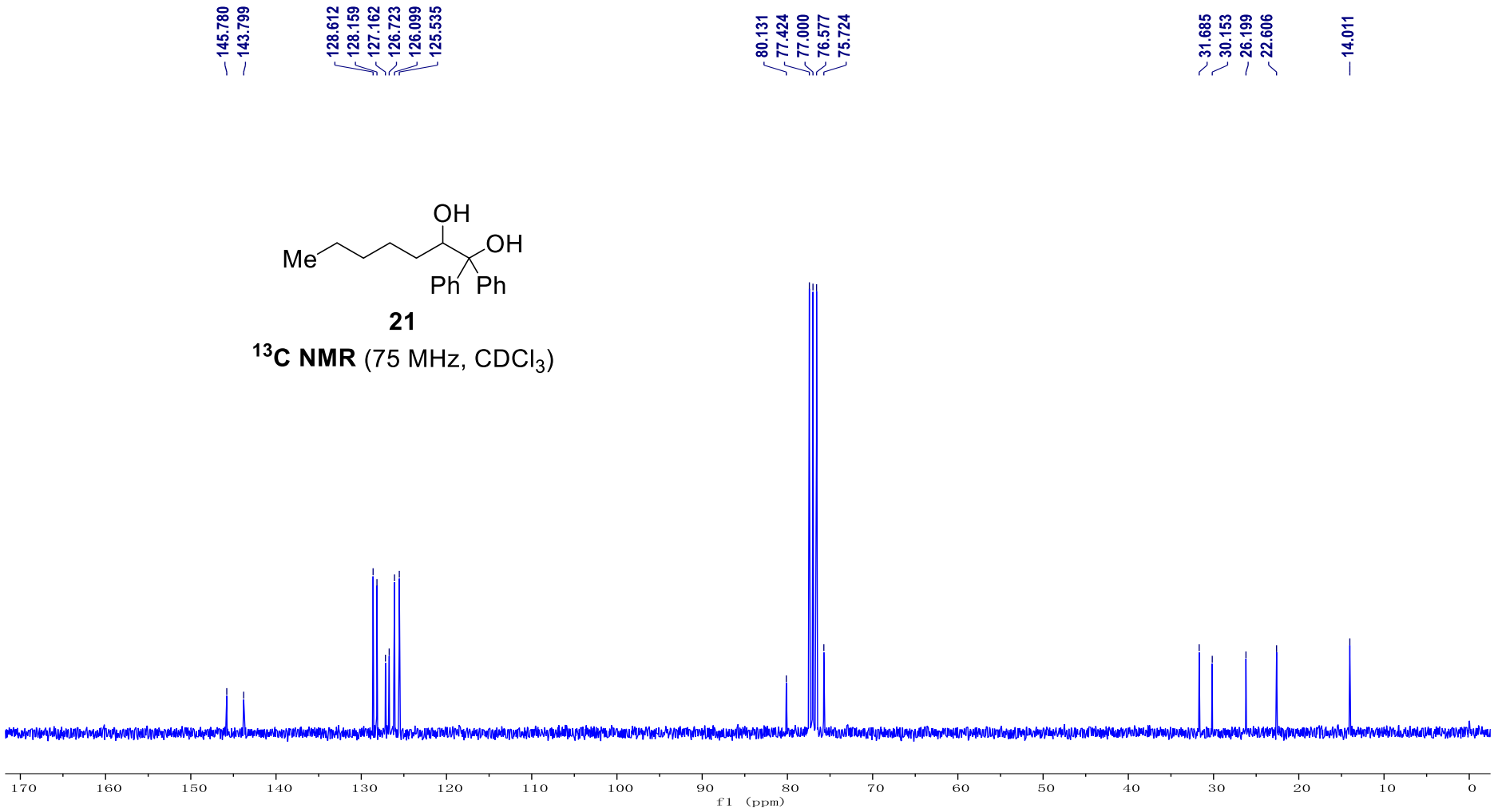
^{1}H NMR (400 MHz, DMSO-d_6)



20

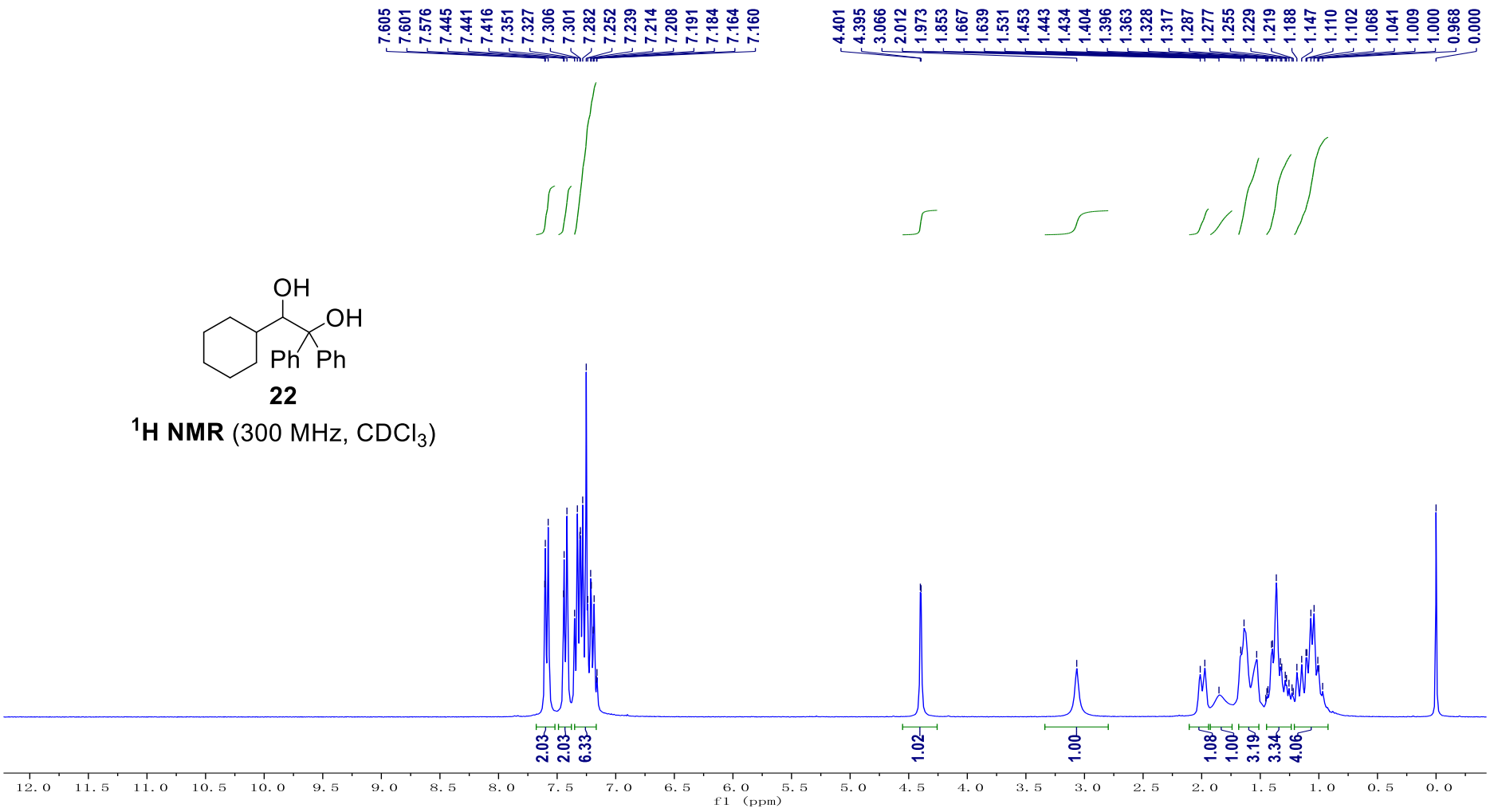
^{13}C NMR (100 MHz, DMSO-d_6)

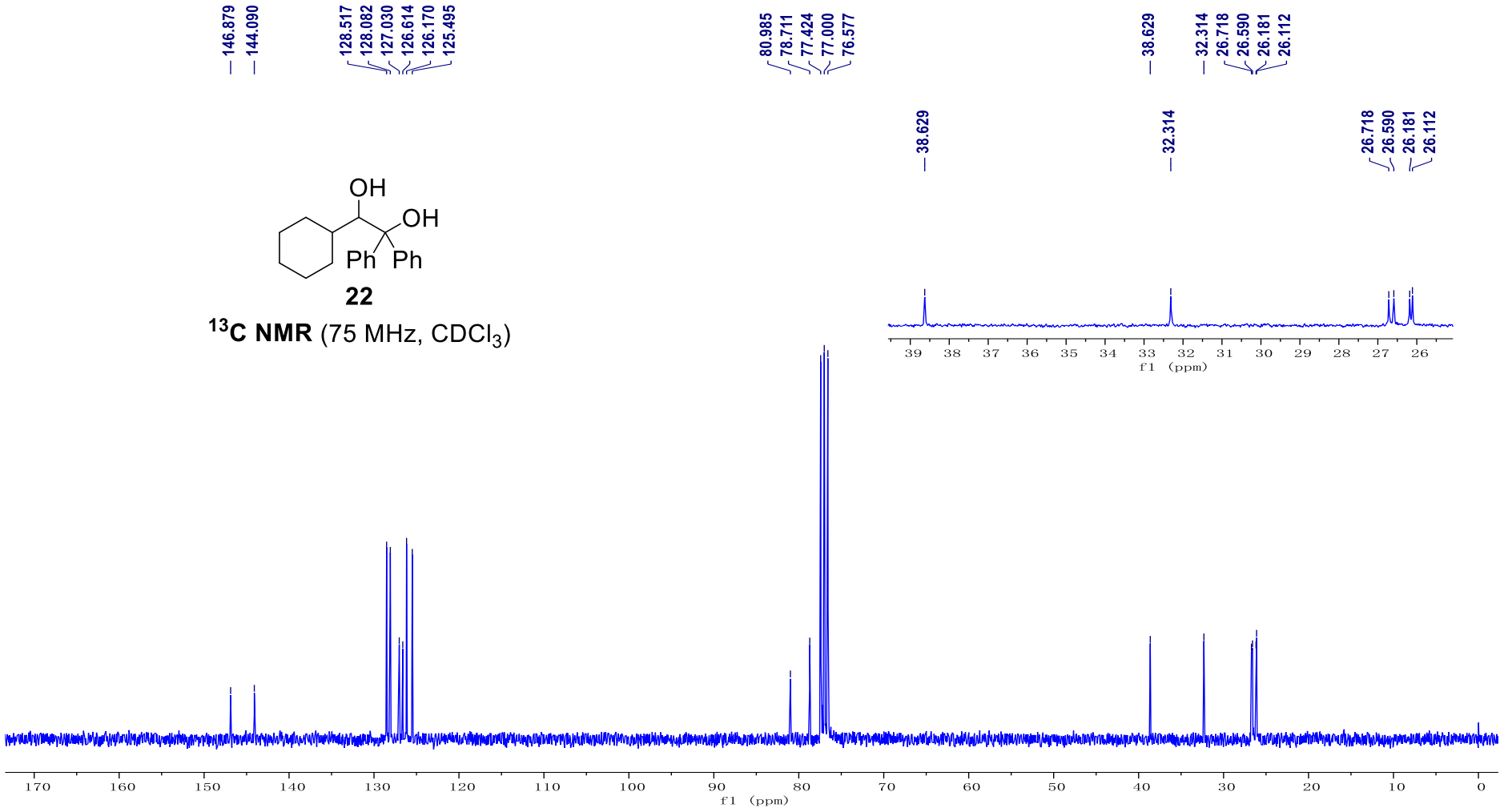

7.617
7.613
7.606
7.589
7.586
7.448
7.443
7.437
7.420
7.416
7.409
7.374
7.368
7.350
7.345
7.329
7.324
7.314
7.311
7.305
7.287
7.282
7.260
7.256
7.242
7.235
7.228
7.214
7.210
7.206
7.193
7.185
7.178
7.166
7.161
7.157
4.588
4.556
2.993
1.802
1.563
1.550
1.537
1.522
1.509
1.493
1.479
1.461
1.447
1.431
1.357
1.334
1.302
1.288
1.259
1.250
1.236
1.213
1.175
0.869
0.847
0.825
0.000
OH
OH
Me
Ph
Ph
21
1H NMR (300 MHz, CDCl3)
2.01
2.00
6.22
1.02
1.00
1.04
2.05
6.10
3.05
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

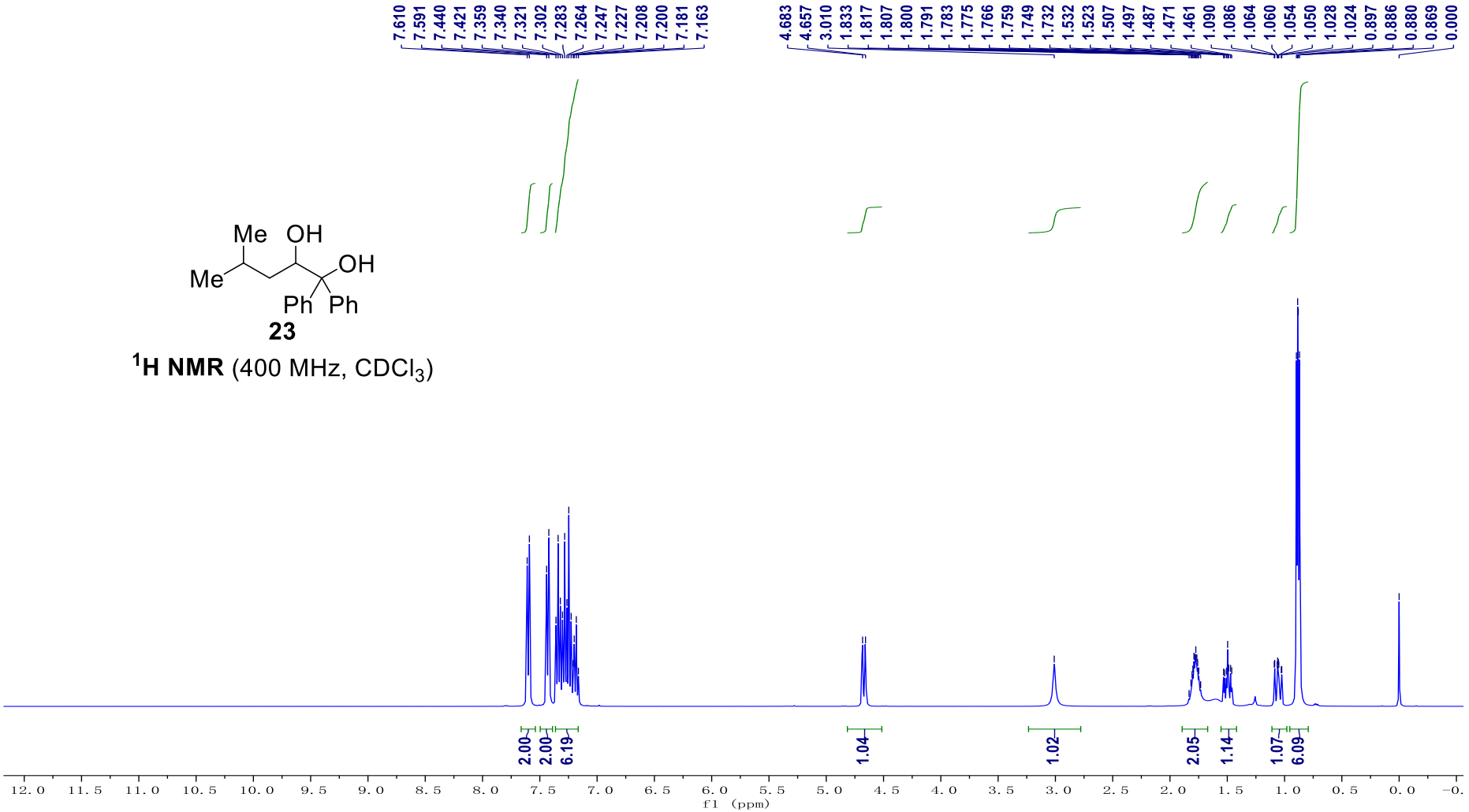


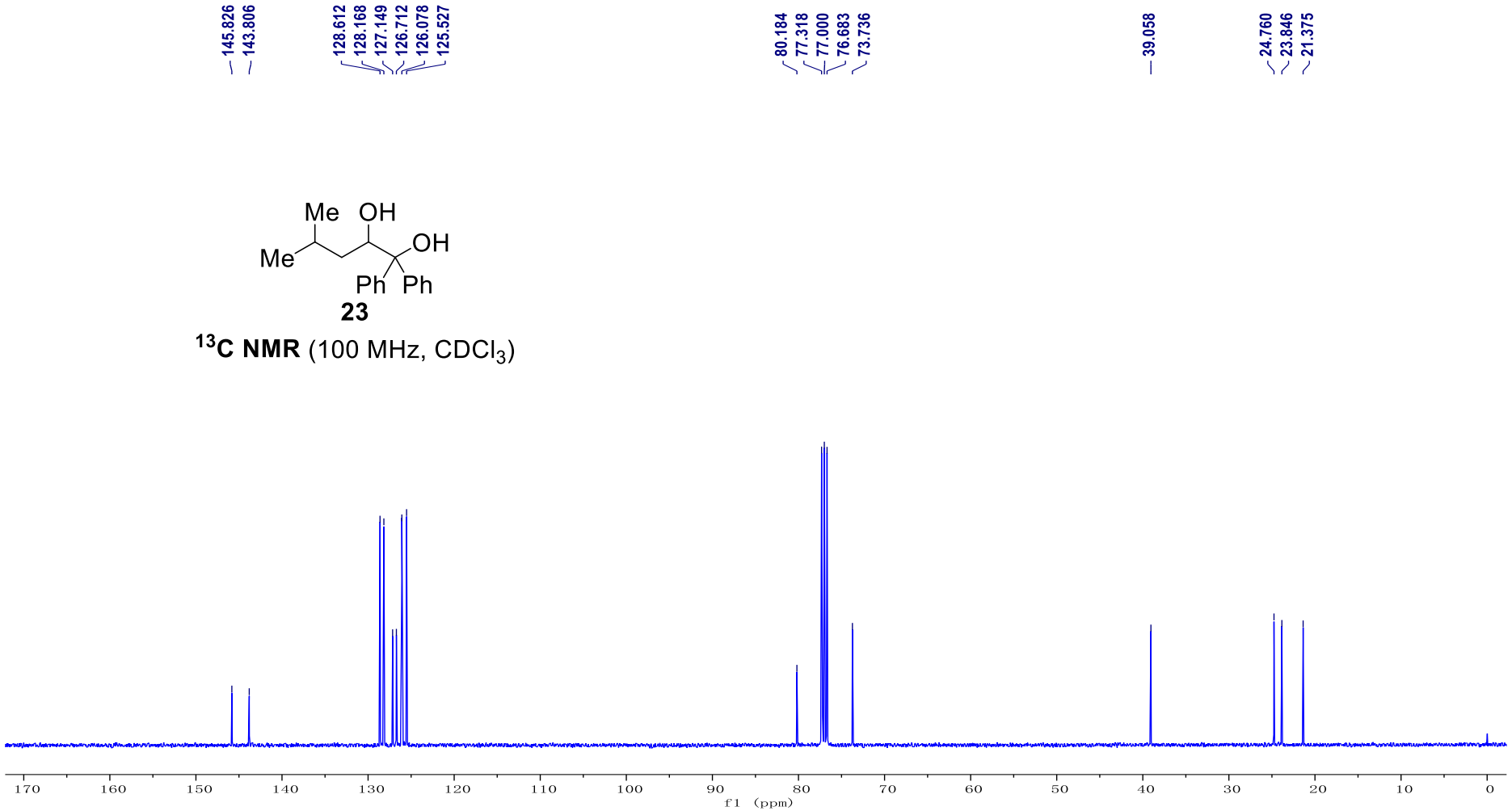
21

^{13}C NMR (75 MHz, $CDCl_3$)

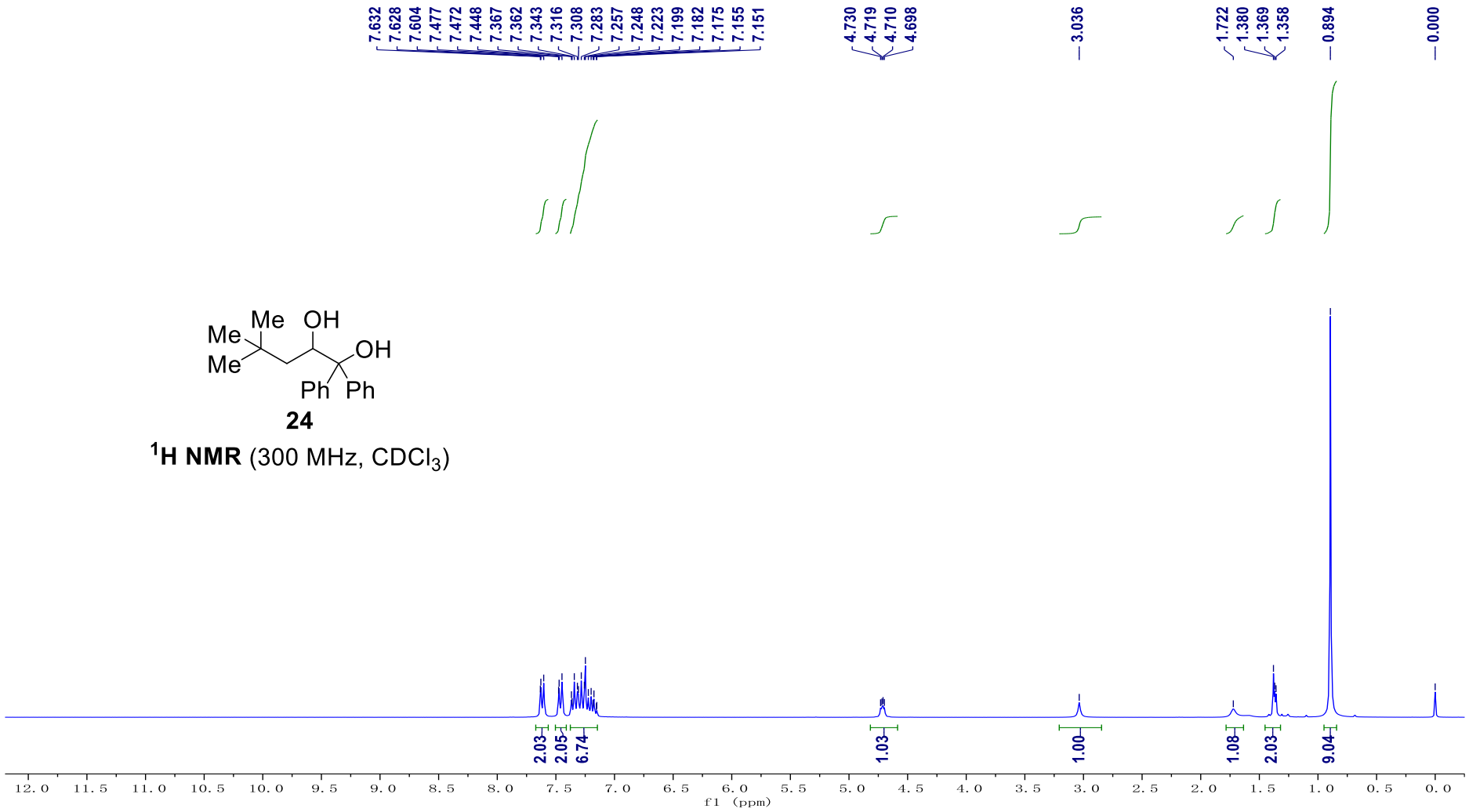

7.605
7.601
7.576
7.445
7.441
7.416
7.351
7.327
7.306
7.301
7.282
7.252
7.239
7.214
7.208
7.191
7.184
7.164
7.160
4.401
4.395
3.066
2.012
1.973
1.853
1.667
1.639
1.531
1.453
1.443
1.434
1.404
1.396
1.363
1.328
1.317
1.287
1.277
1.255
1.229
1.219
1.188
1.147
1.110
1.102
1.068
1.041
1.009
1.000
0.968
0.000
OH
OH
Ph
Ph
22
1H NMR (300 MHz, CDCl3)
2.03
2.03
6.33
1.02
1.00
1.08
1.00
3.19
3.34
4.06
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


146.879
144.090
128.517
128.082
127.030
126.614
126.170
125.495
80.985
78.711
77.424
77.000
76.577
38.629
32.314
26.718
26.590
26.181
26.112
OH
OH
Ph
Ph
22
13C NMR (75 MHz, CDCl3)
f1 (ppm)


Me
OH
OH
Me
Ph
Ph
23
1H NMR (400 MHz, CDCl3)
7.610
7.591
7.440
7.421
7.359
7.340
7.321
7.302
7.283
7.264
7.247
7.227
7.208
7.200
7.181
7.163
4.683
4.657
3.010
1.833
1.817
1.807
1.800
1.791
1.783
1.775
1.766
1.759
1.749
1.732
1.532
1.523
1.507
1.497
1.487
1.471
1.461
1.090
1.086
1.064
1.060
1.054
1.050
1.028
1.024
0.897
0.886
0.880
0.869
0.000
2.00
2.00
6.19
1.04
1.02
2.05
1.14
1.07
6.09
f1 (ppm)

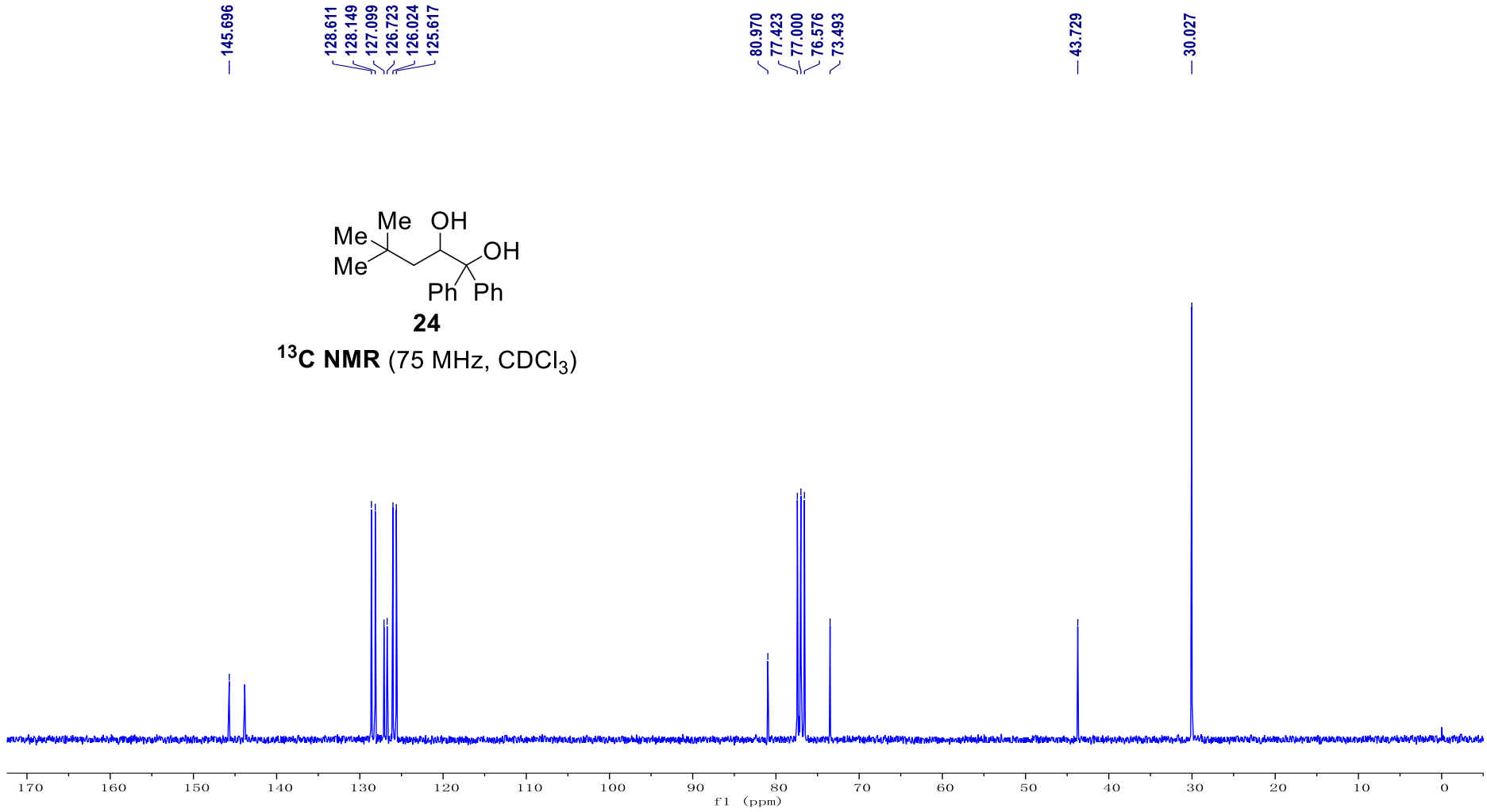


^{13}C NMR (100 MHz, $CDCl_3$)

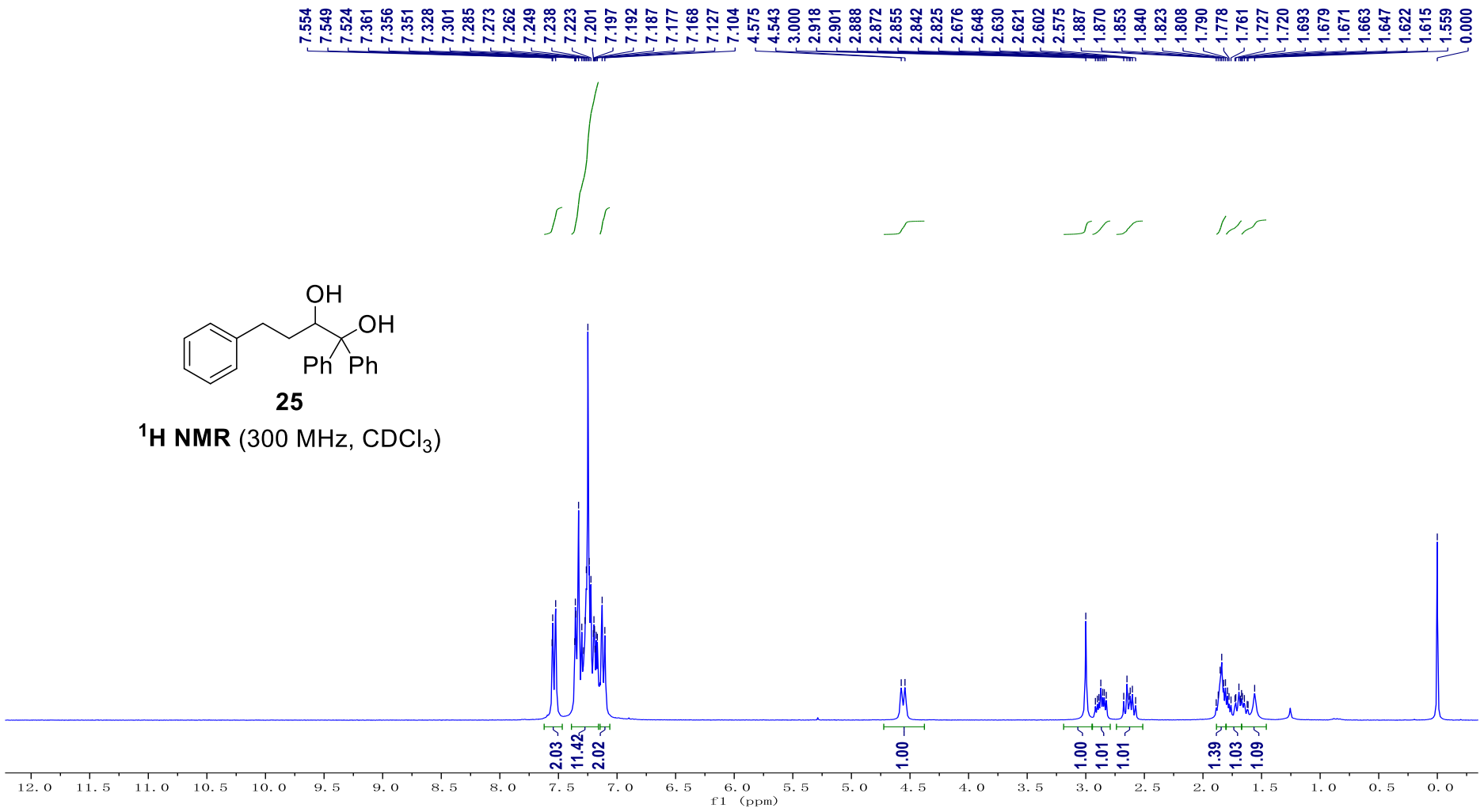


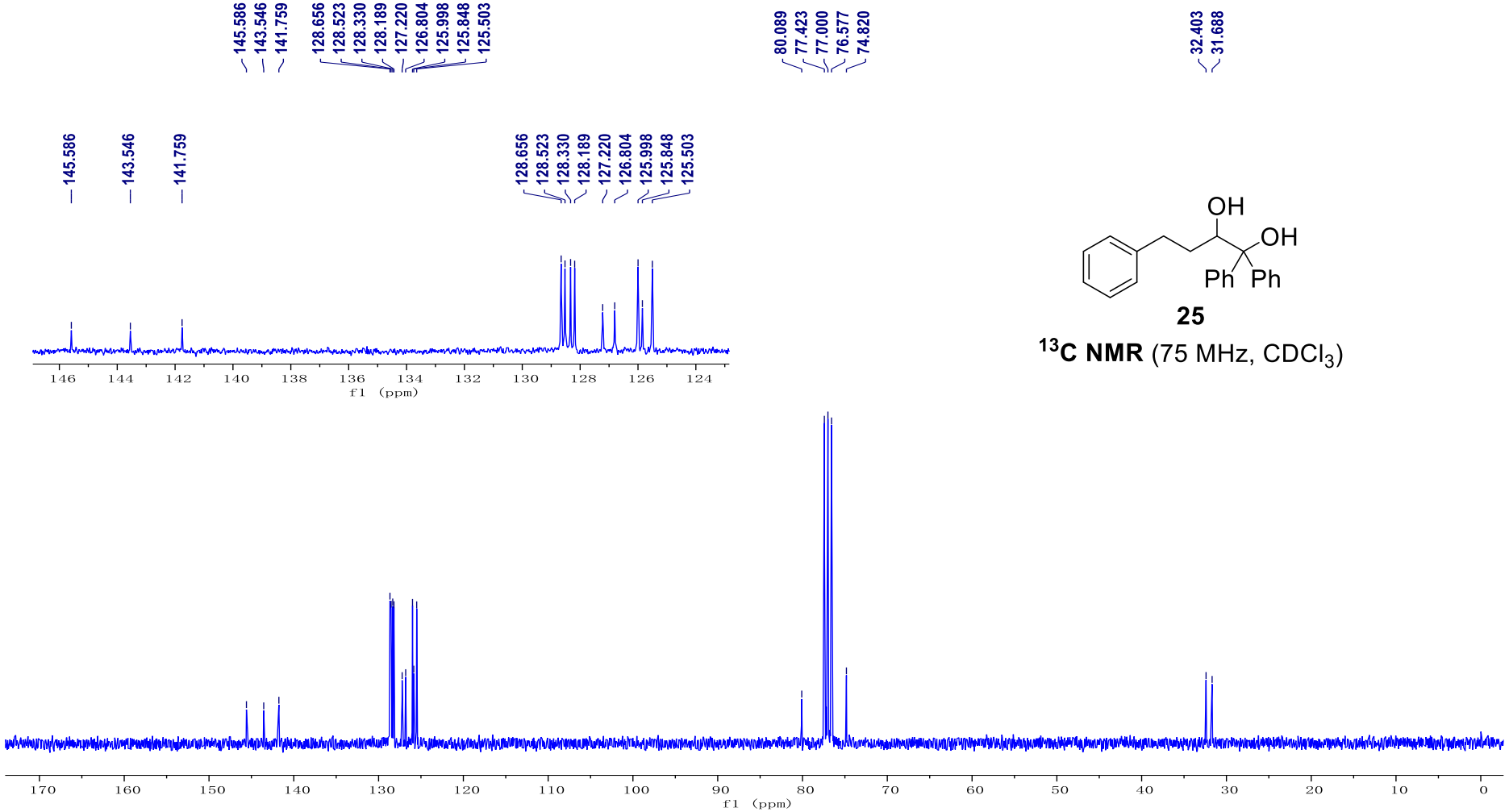
24

^{1}H NMR (300 MHz, $CDCl_3$)



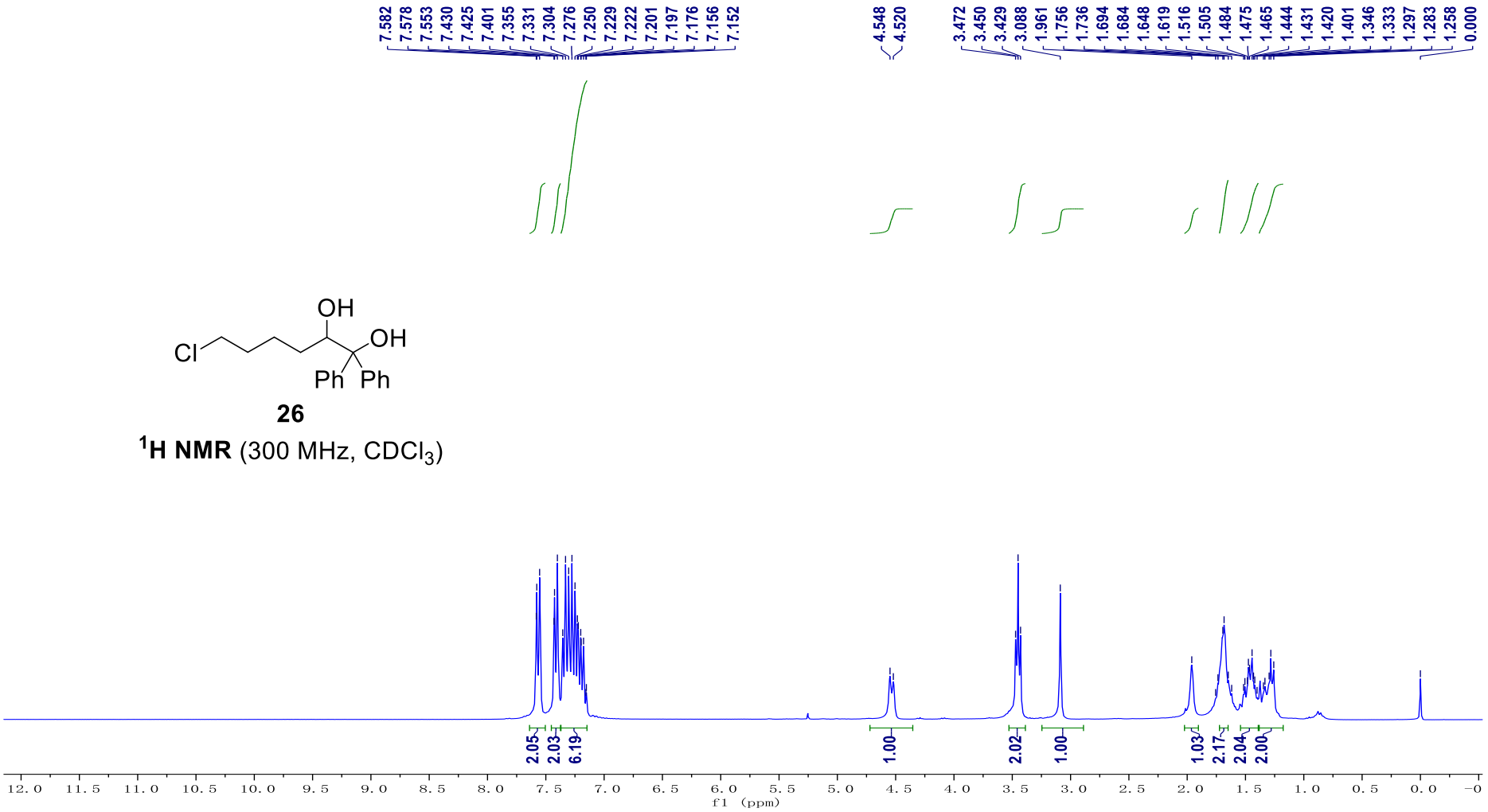
^{13}C NMR (75 MHz, $CDCl_3$)


7.554
7.549
7.524
7.361
7.356
7.351
7.328
7.301
7.285
7.273
7.262
7.249
7.238
7.223
7.201
7.197
7.192
7.187
7.177
7.168
7.127
7.104
4.575
4.543
3.000
2.918
2.901
2.888
2.872
2.855
2.842
2.825
2.676
2.648
2.630
2.621
2.602
2.575
1.887
1.870
1.853
1.840
1.823
1.808
1.790
1.778
1.761
1.727
1.720
1.693
1.679
1.671
1.663
1.647
1.622
1.615
1.559
0.000
OH
OH
Ph
Ph
25
1H NMR (300 MHz, CDCl3)
2.03
11.42
2.02
1.00
1.00
1.01
1.01
1.39
1.03
1.09
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



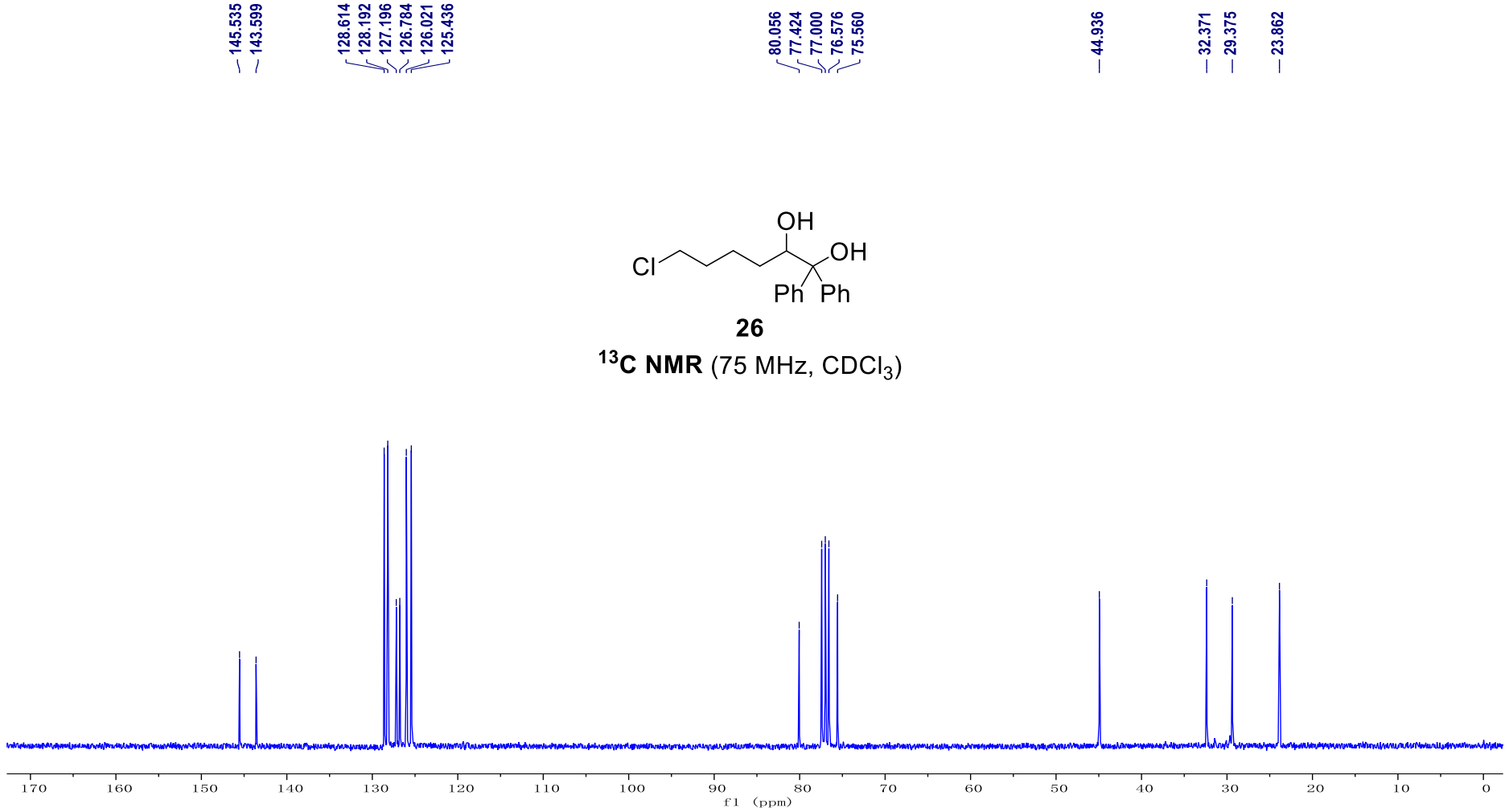
25

^{13}C NMR (75 MHz, $CDCl_3$)



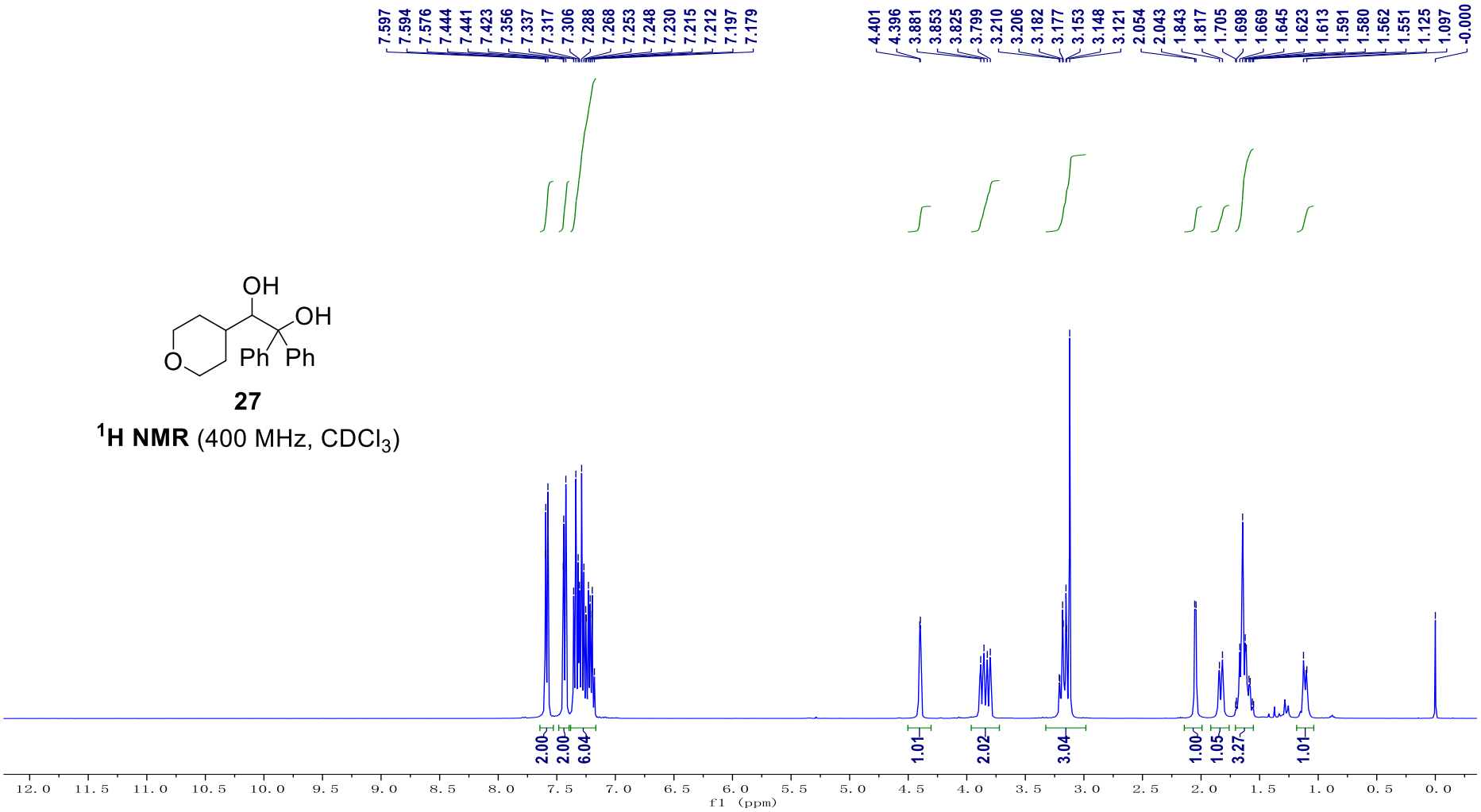
26

^{1}H NMR (300 MHz, $CDCl_3$)



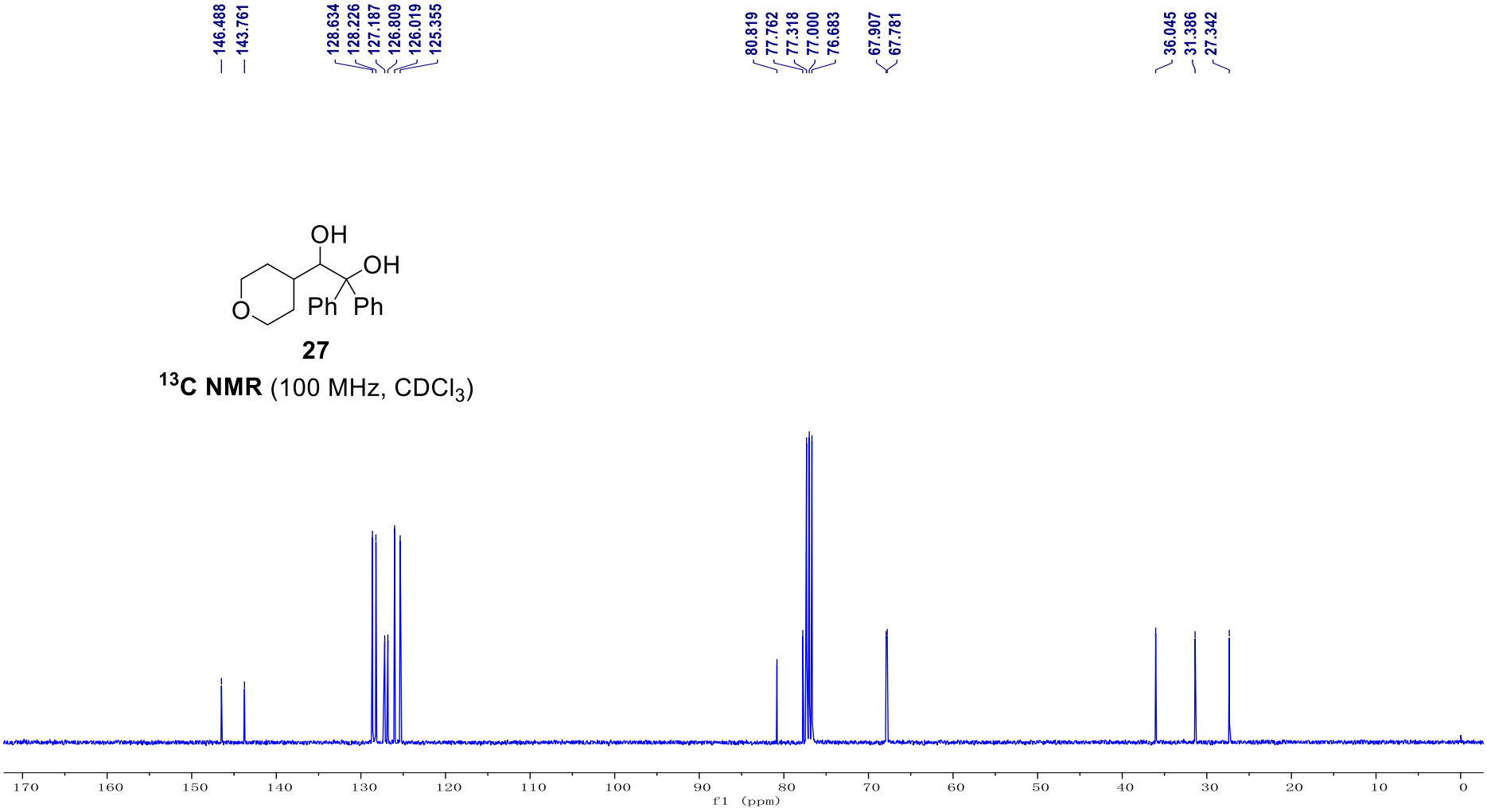
26

^{13}C NMR (75 MHz, $CDCl_3$)



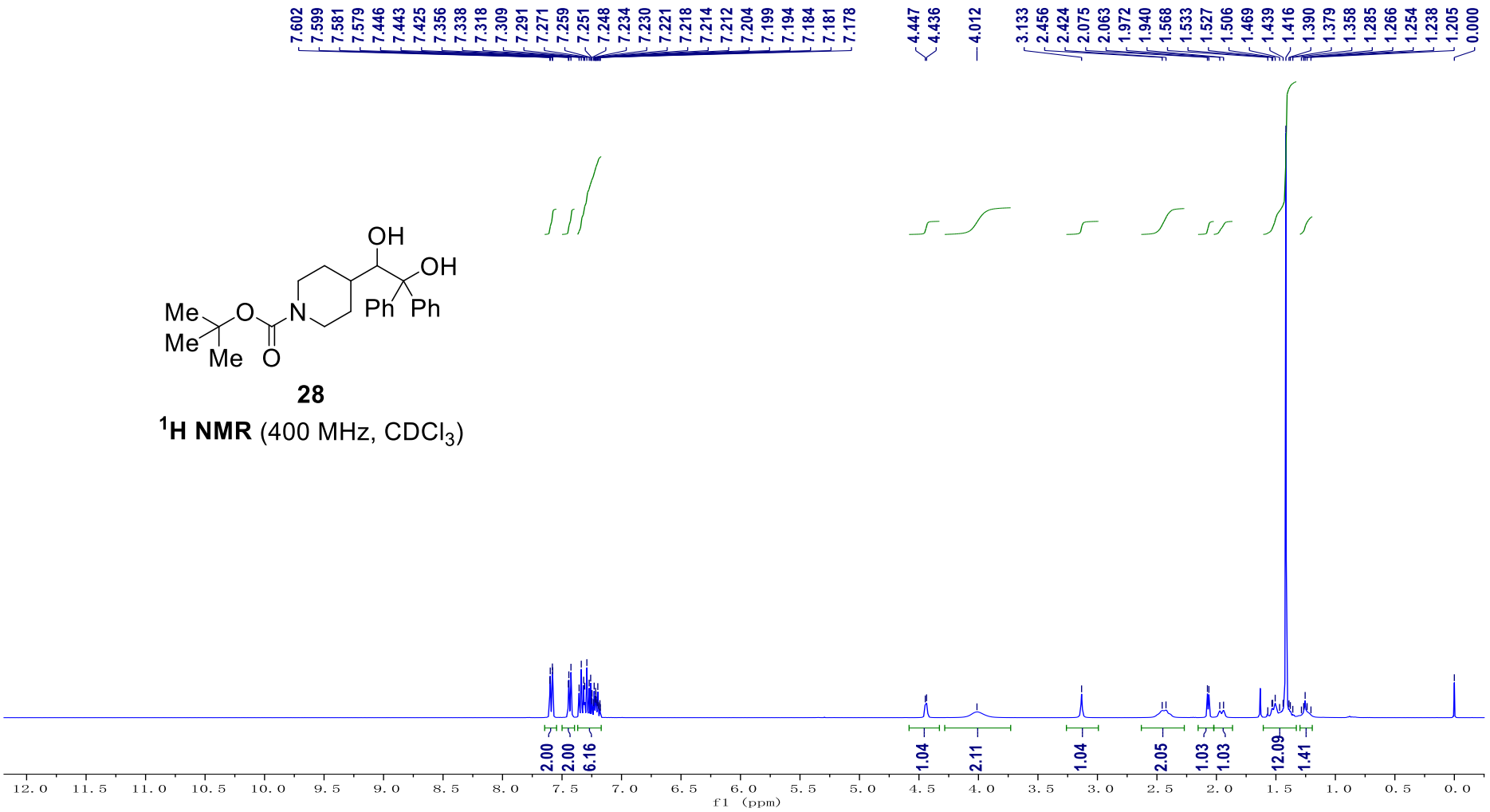
27

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



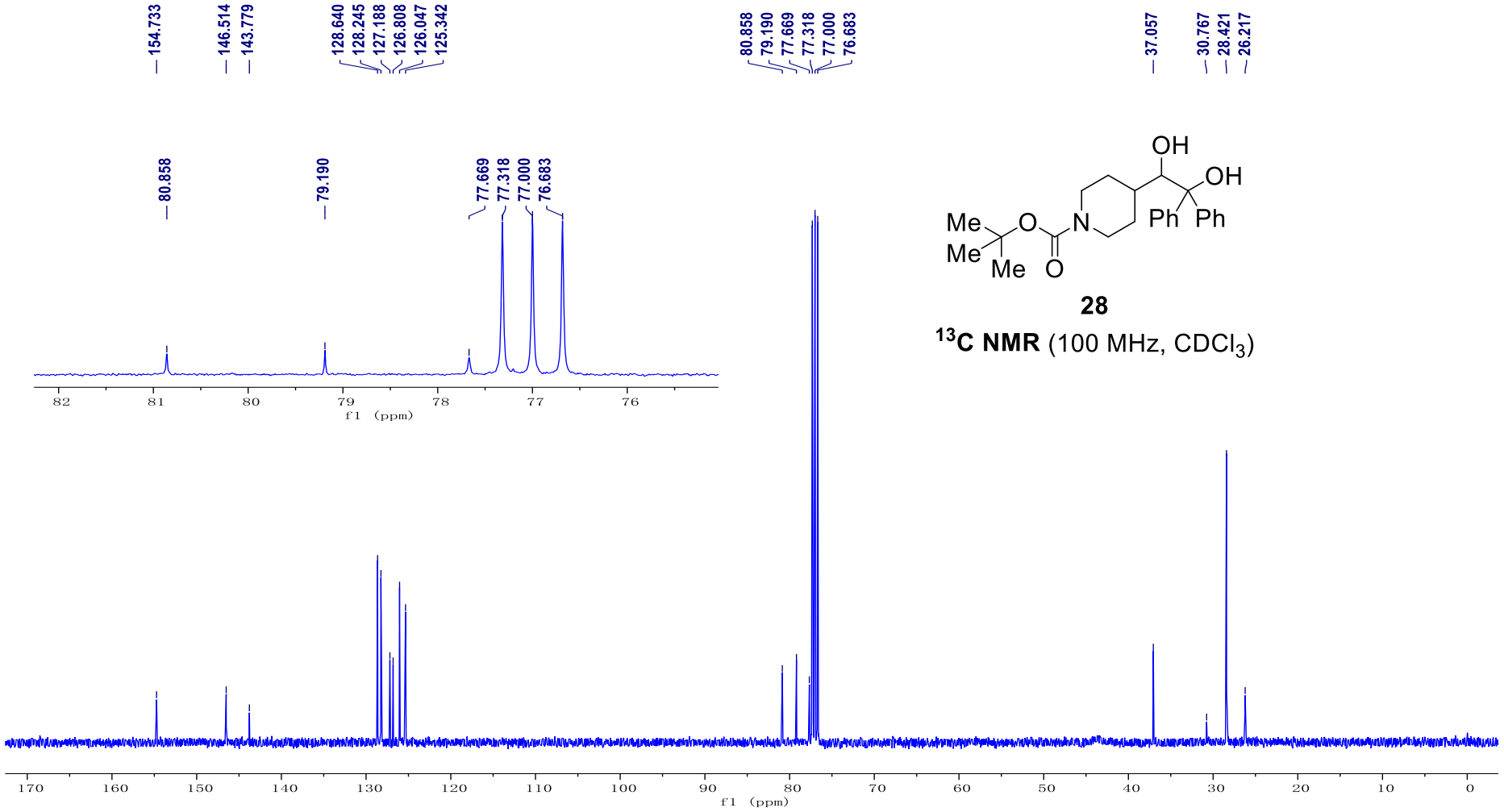
27

^{13}C NMR (100 MHz, $CDCl_3$)



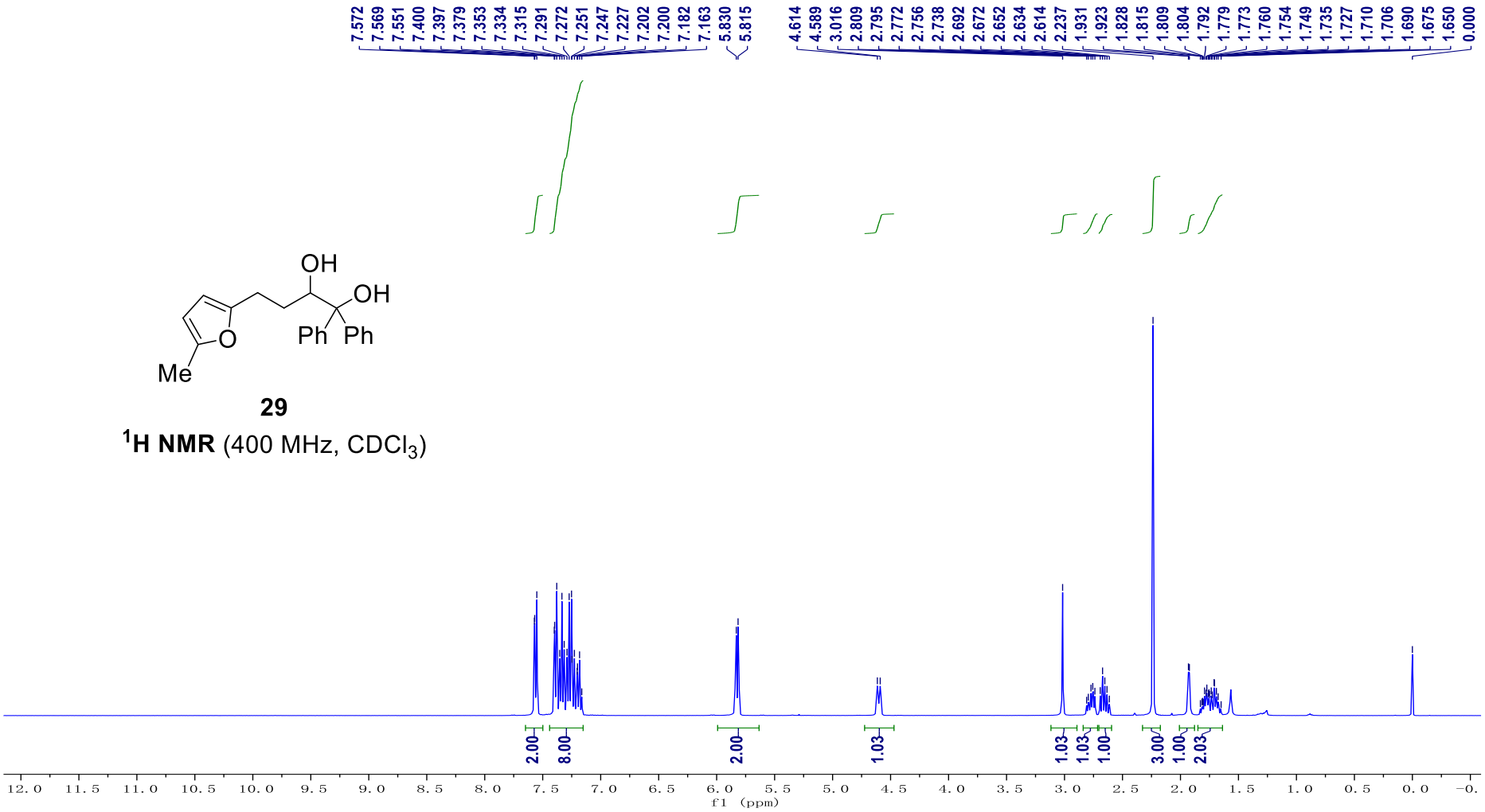
28

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



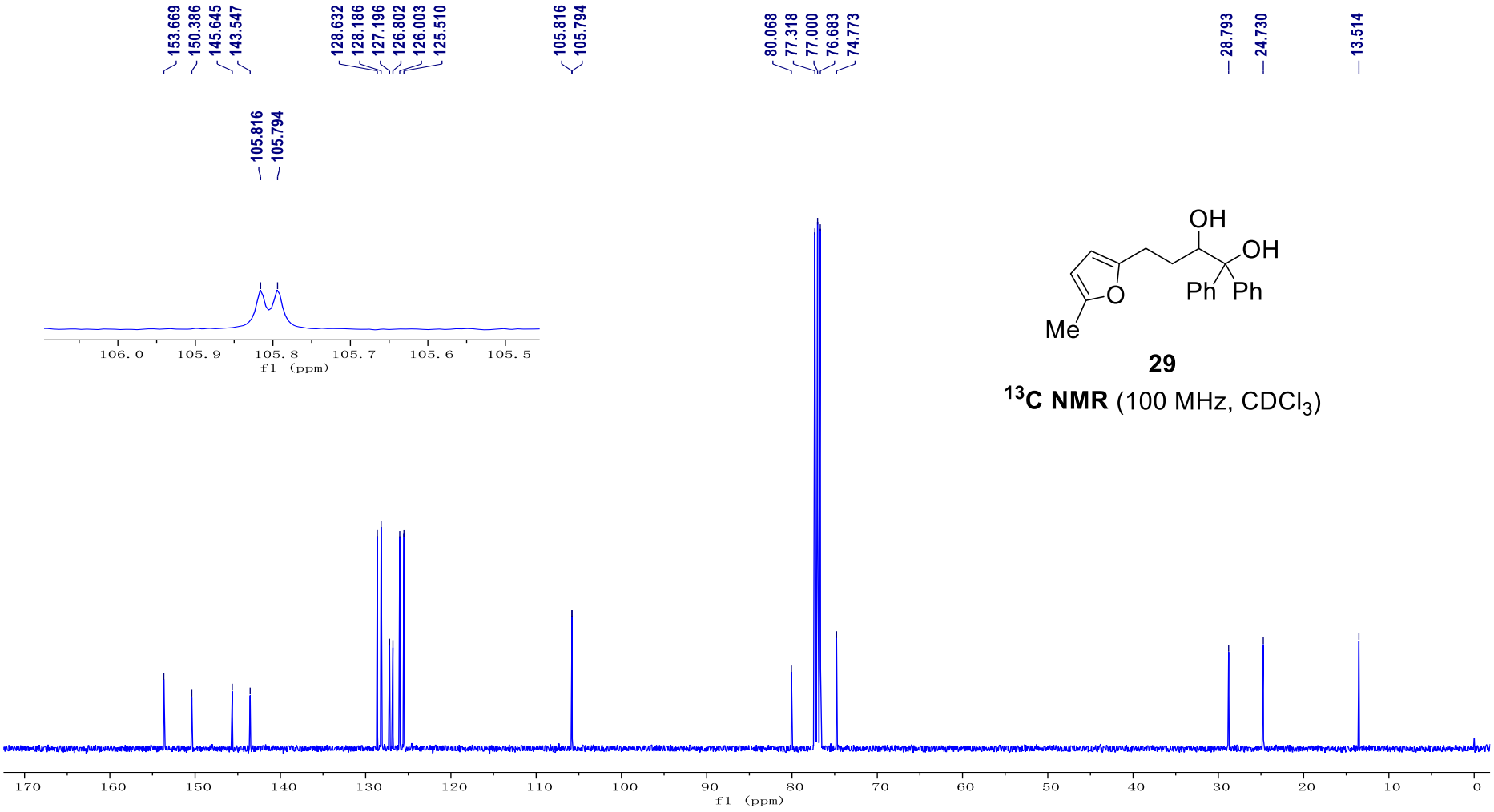
28

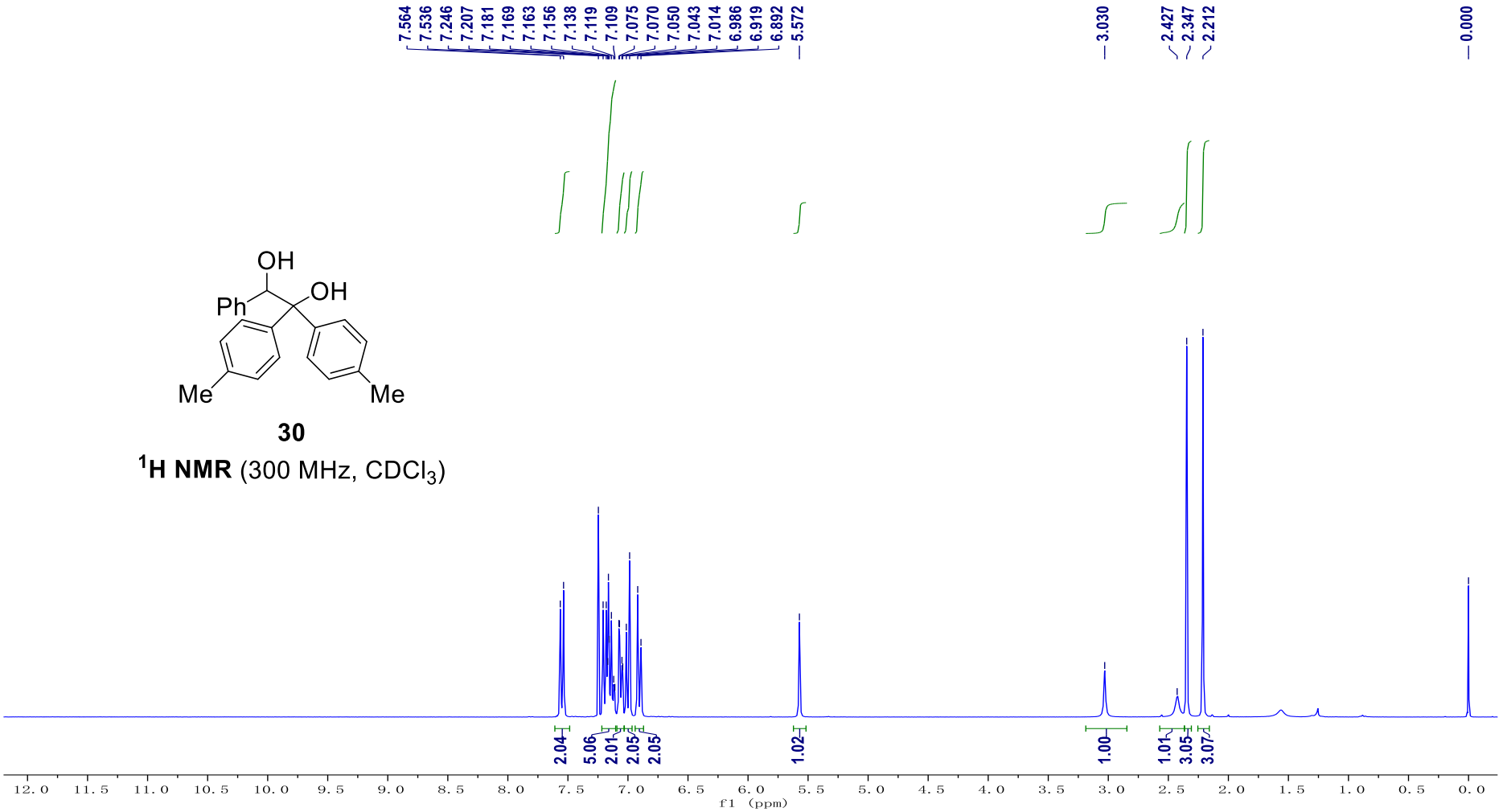
^{13}C NMR (100 MHz, $CDCl_3$)



29

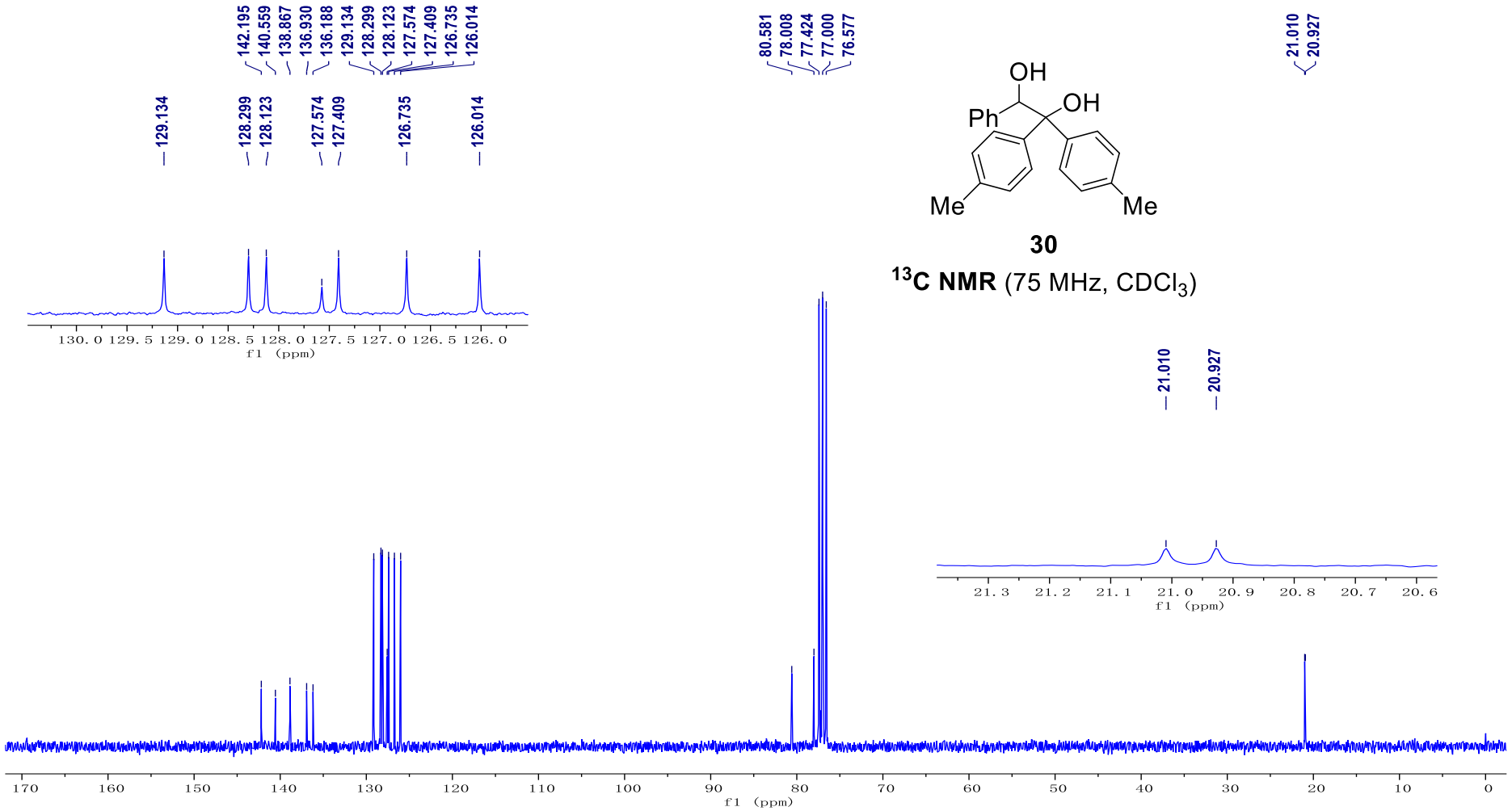
^{1}H NMR (400 MHz, $CDCl_3$)

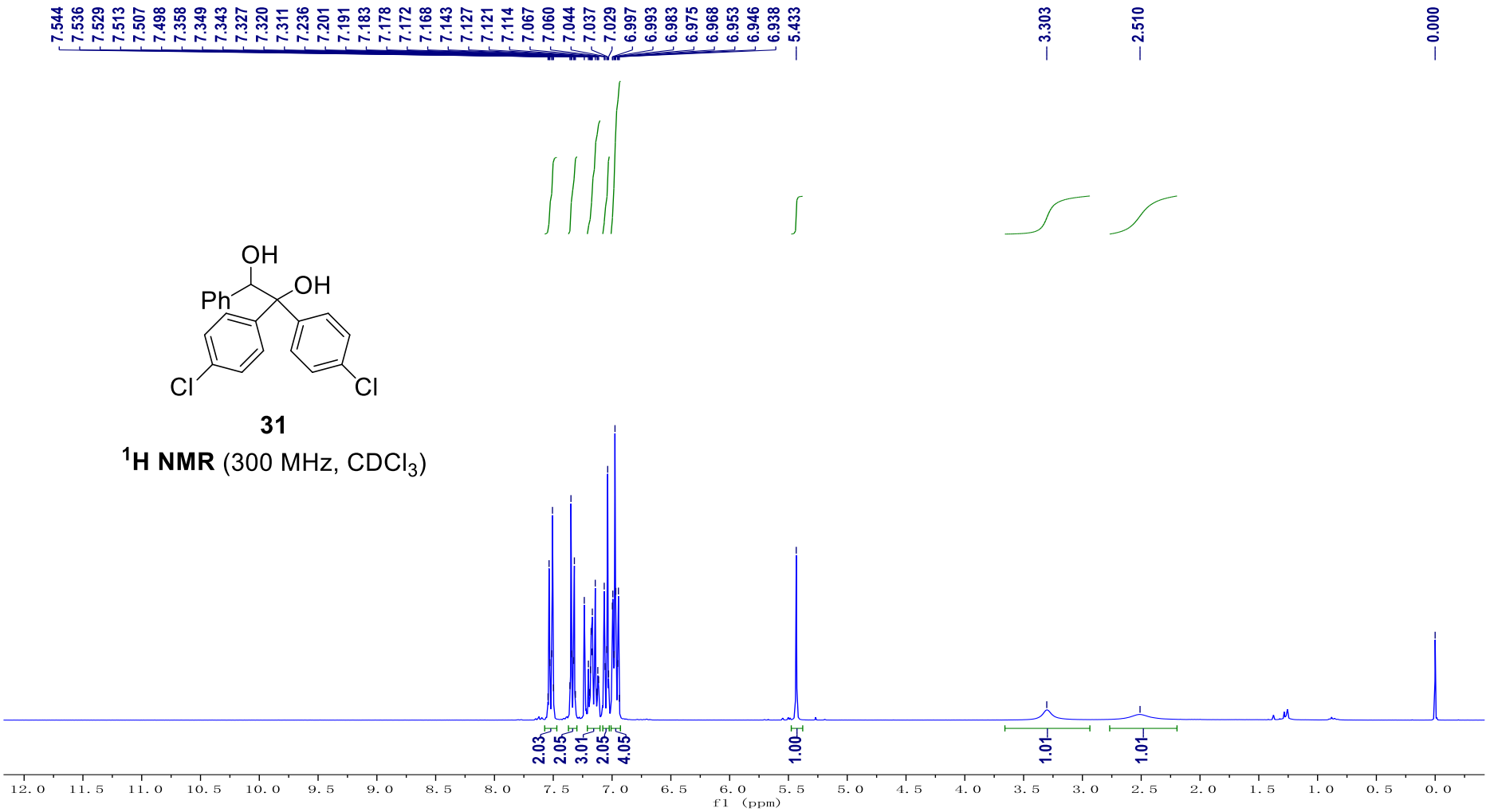

153.669
150.386
145.645
143.547
128.632
128.186
127.196
126.802
126.003
125.510
105.816
105.794
80.068
77.318
77.000
76.683
74.773
28.793
24.730
13.514
105.816
105.794
106.0
105.9
105.8
105.7
105.6
105.5
f1 (ppm)
OH
OH
Ph
Ph
O
Me
29
13C NMR (100 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

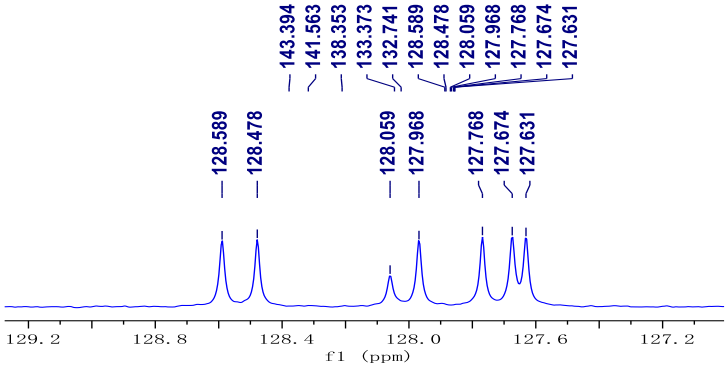


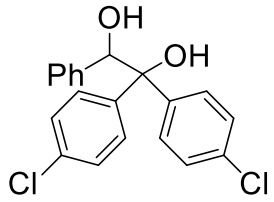
30

^{1}H NMR (300 MHz, $CDCl_3$)


142.195
140.559
138.867
136.930
136.188
129.134
128.299
128.123
127.574
127.409
126.735
126.014
80.581
78.008
77.424
77.000
76.577
21.010
20.927
OH
Ph
OH
Me
Me
30
13C NMR (75 MHz, CDCl3)
f1 (ppm)

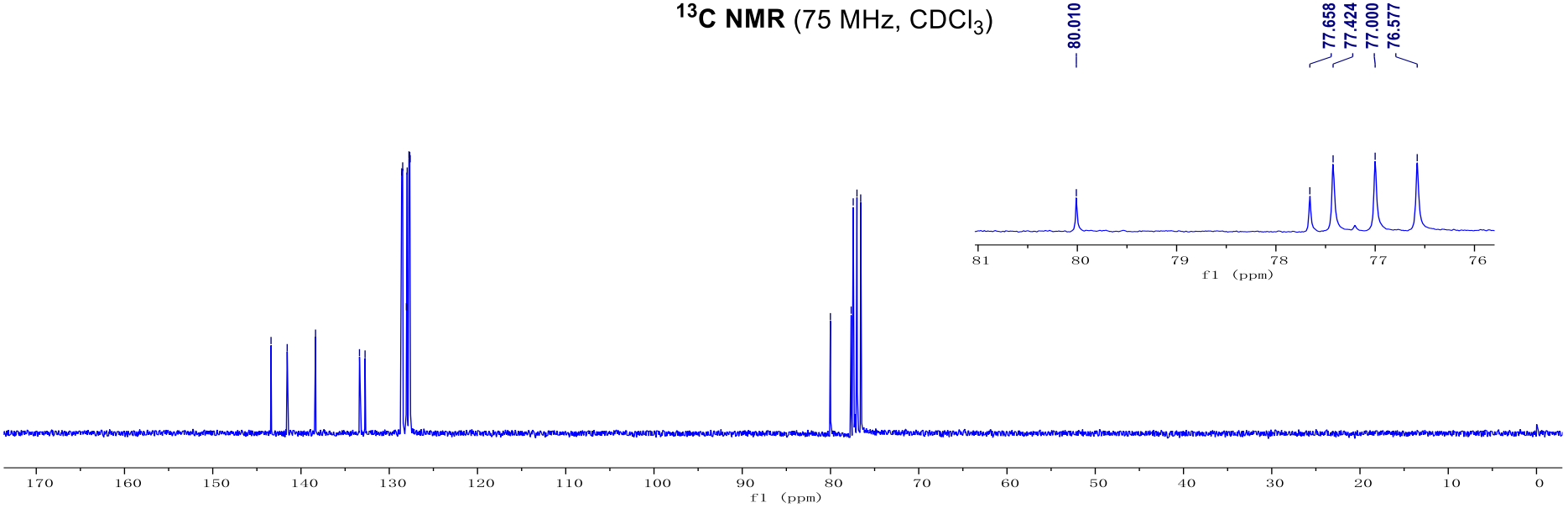

7.544
7.536
7.529
7.513
7.507
7.498
7.358
7.349
7.343
7.327
7.320
7.311
7.236
7.201
7.191
7.183
7.178
7.172
7.168
7.143
7.127
7.121
7.114
7.067
7.060
7.044
7.037
7.029
6.997
6.993
6.983
6.975
6.968
6.953
6.946
6.938
5.433
3.303
2.510
0.000
OH
OH
Ph
Cl
Cl
31
1H NMR (300 MHz, CDCl3)
2.03
2.05
3.01
2.05
4.05
1.00
1.01
1.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

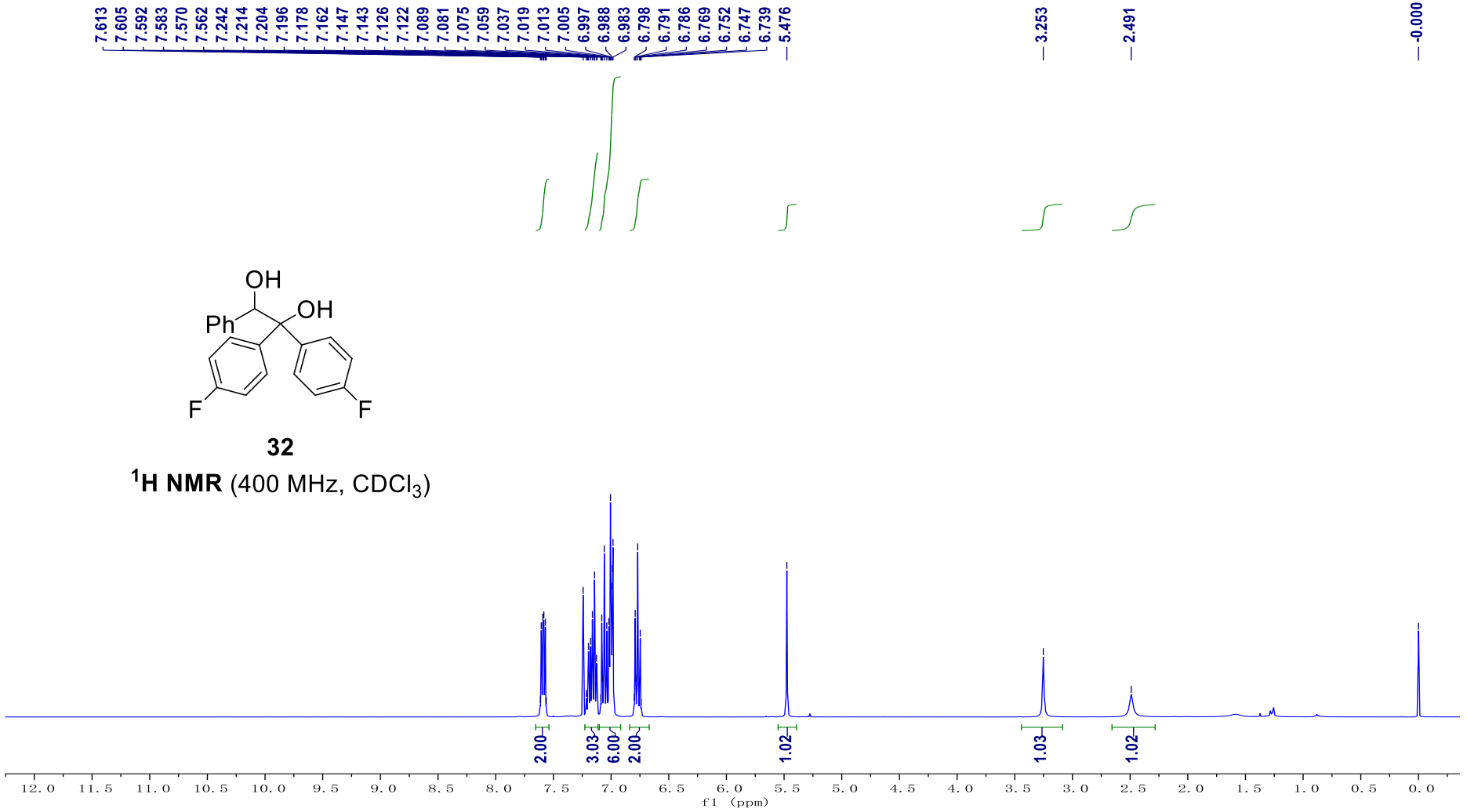




31

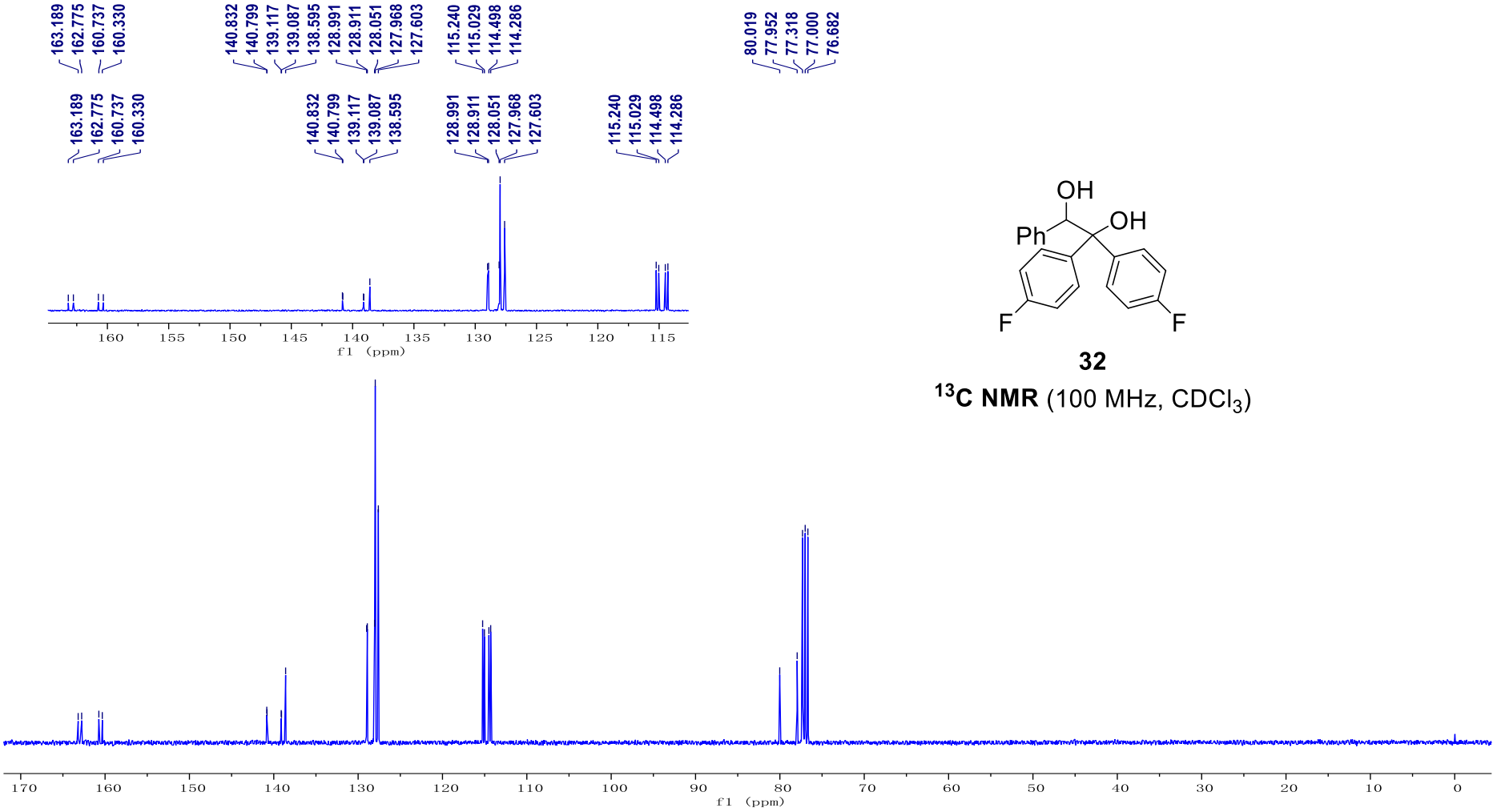
^{13}C NMR (75 MHz, $CDCl_3$)





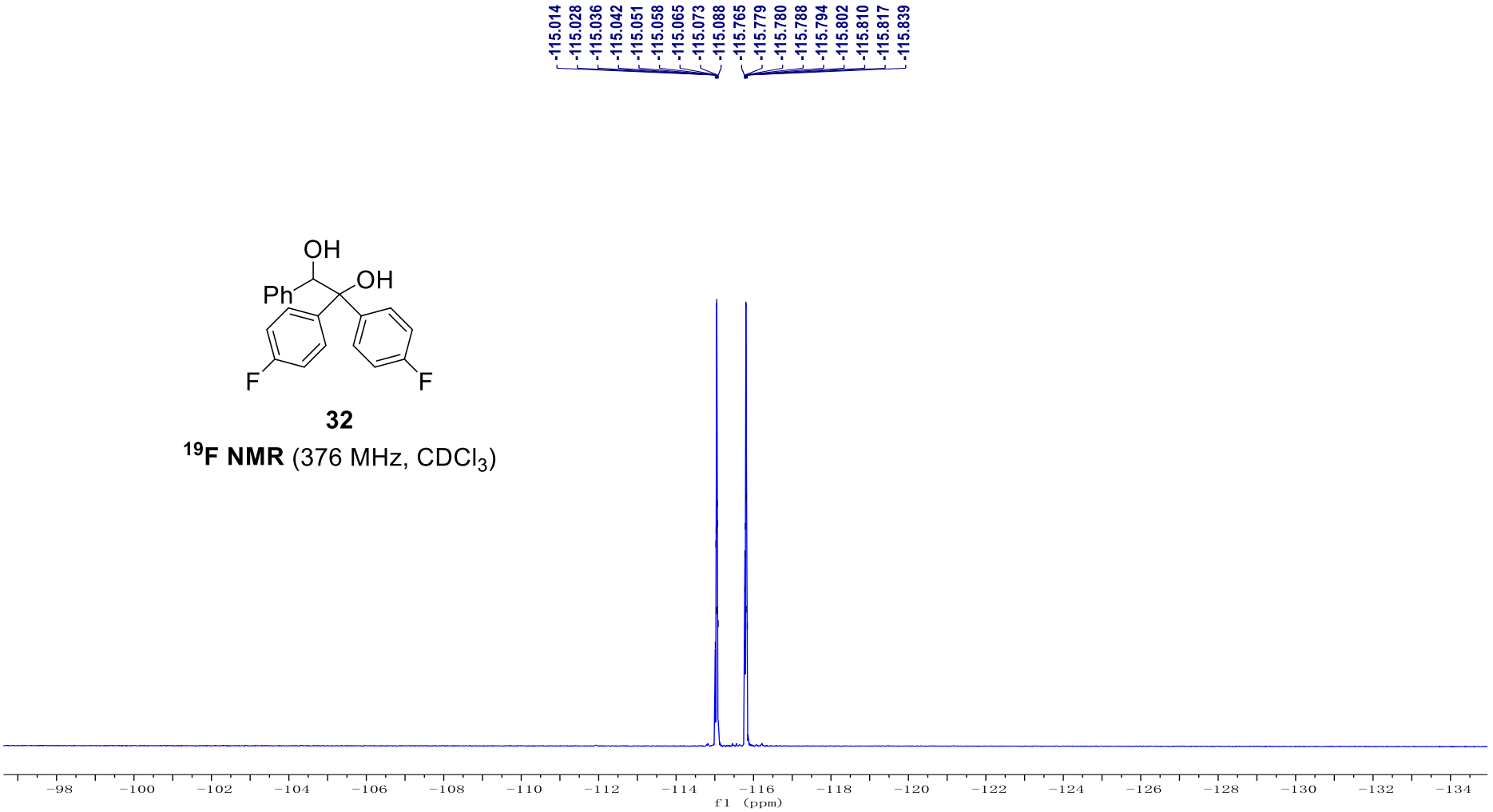
32

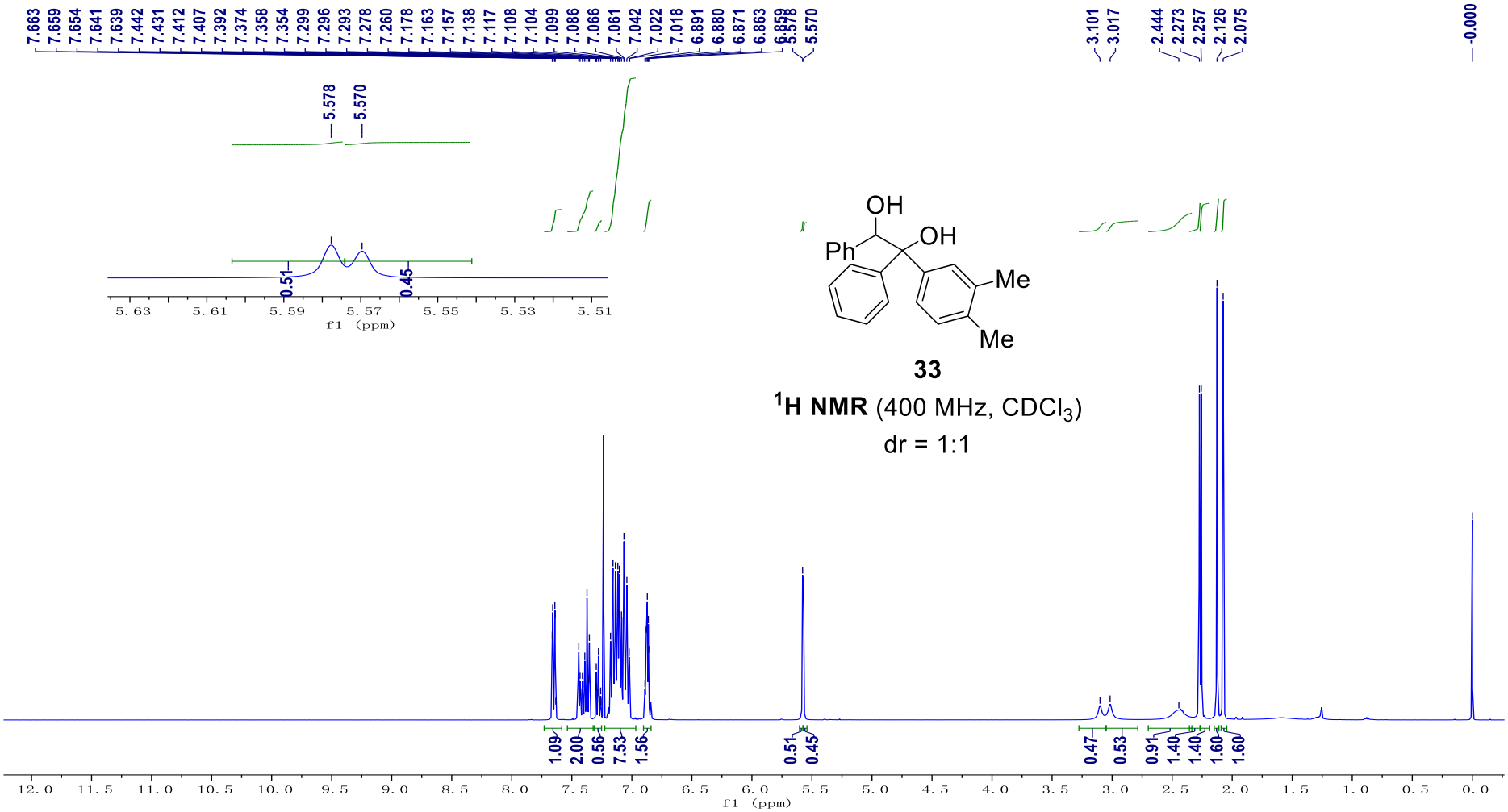
^{1}H NMR (400 MHz, $CDCl_3$)

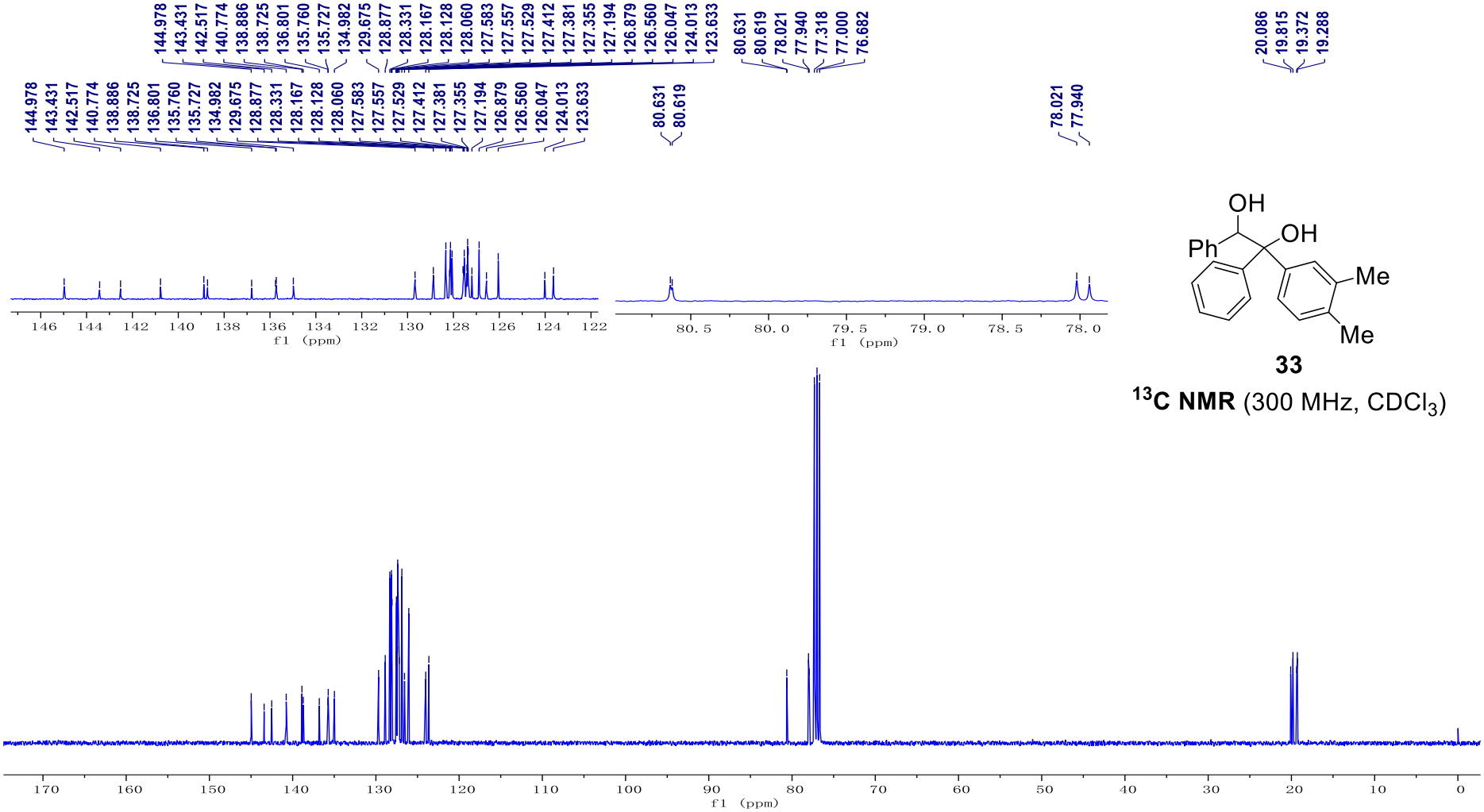


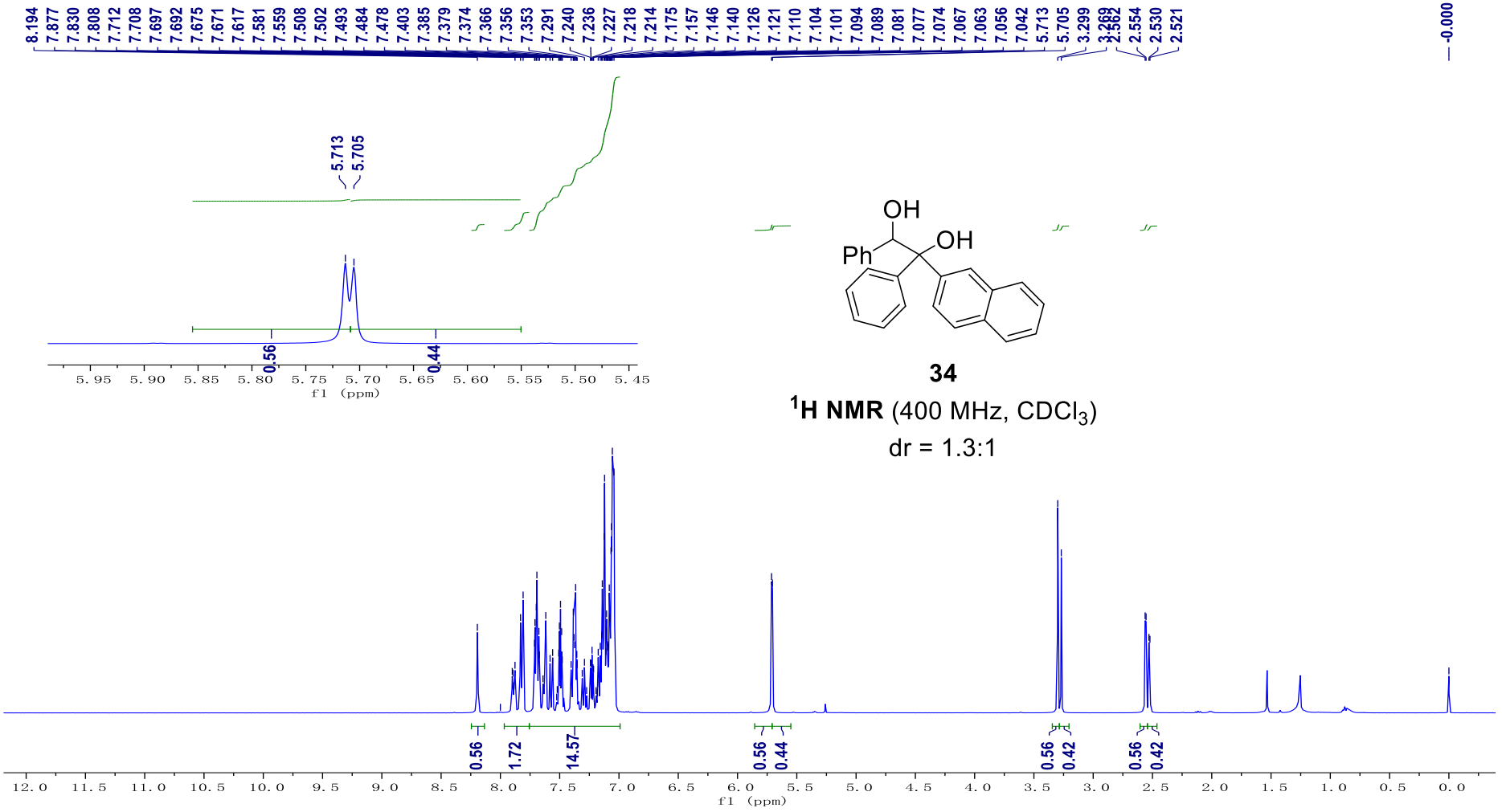
32

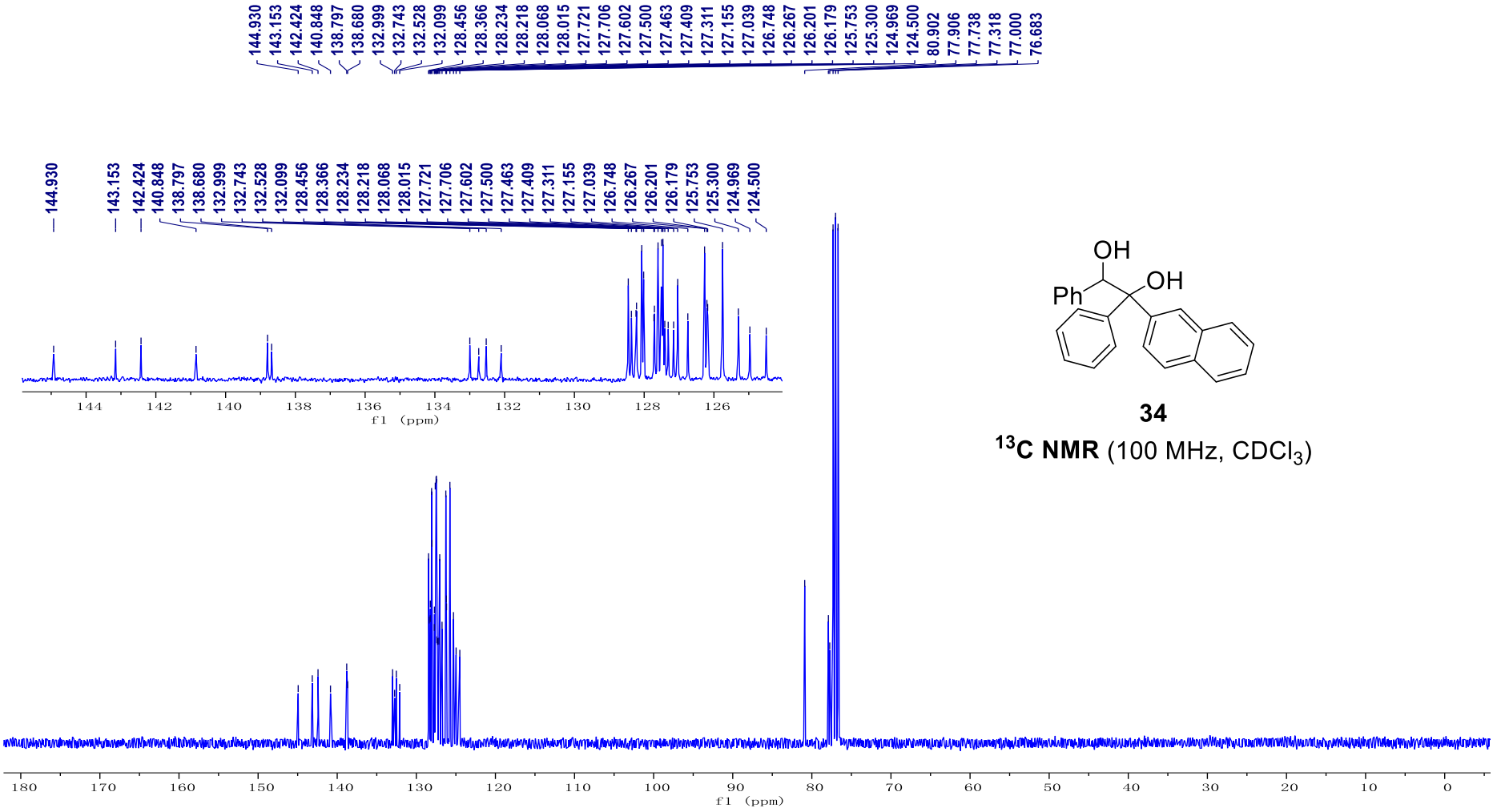
^{13}C NMR (100 MHz, $CDCl_3$)


-115.014
-115.028
-115.036
-115.042
-115.051
-115.058
-115.065
-115.073
-115.088
-115.765
-115.779
-115.780
-115.788
-115.794
-115.802
-115.810
-115.817
-115.839
OH
Ph
OH
F
F
32
19F NMR (376 MHz, CDCl3)
−98
−100
−102
−104
−106
−108
−110
−112
−114
−116
−118
−120
−122
−124
−126
−128
−130
−132
−134
f1 (ppm)


7.663
7.659
7.654
7.641
7.639
7.442
7.431
7.412
7.407
7.392
7.374
7.358
7.354
7.299
7.296
7.293
7.278
7.260
7.178
7.163
7.157
7.138
7.117
7.108
7.104
7.099
7.086
7.066
7.061
7.042
7.022
7.018
6.891
6.880
6.871
6.863
6.859
5.578
5.570
3.101
3.017
2.444
2.273
2.257
2.126
2.075
-0.000
5.578
5.570
0.51
0.45
5.63
5.61
5.59
5.57
5.55
5.53
5.51
f1 (ppm)
OH
OH
Ph
Me
Me
33
1H NMR (400 MHz, CDCl3)
dr = 1:1
1.09
2.00
0.56
7.53
1.56
0.51
0.45
0.47
0.53
0.91
1.40
1.40
1.60
1.60
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

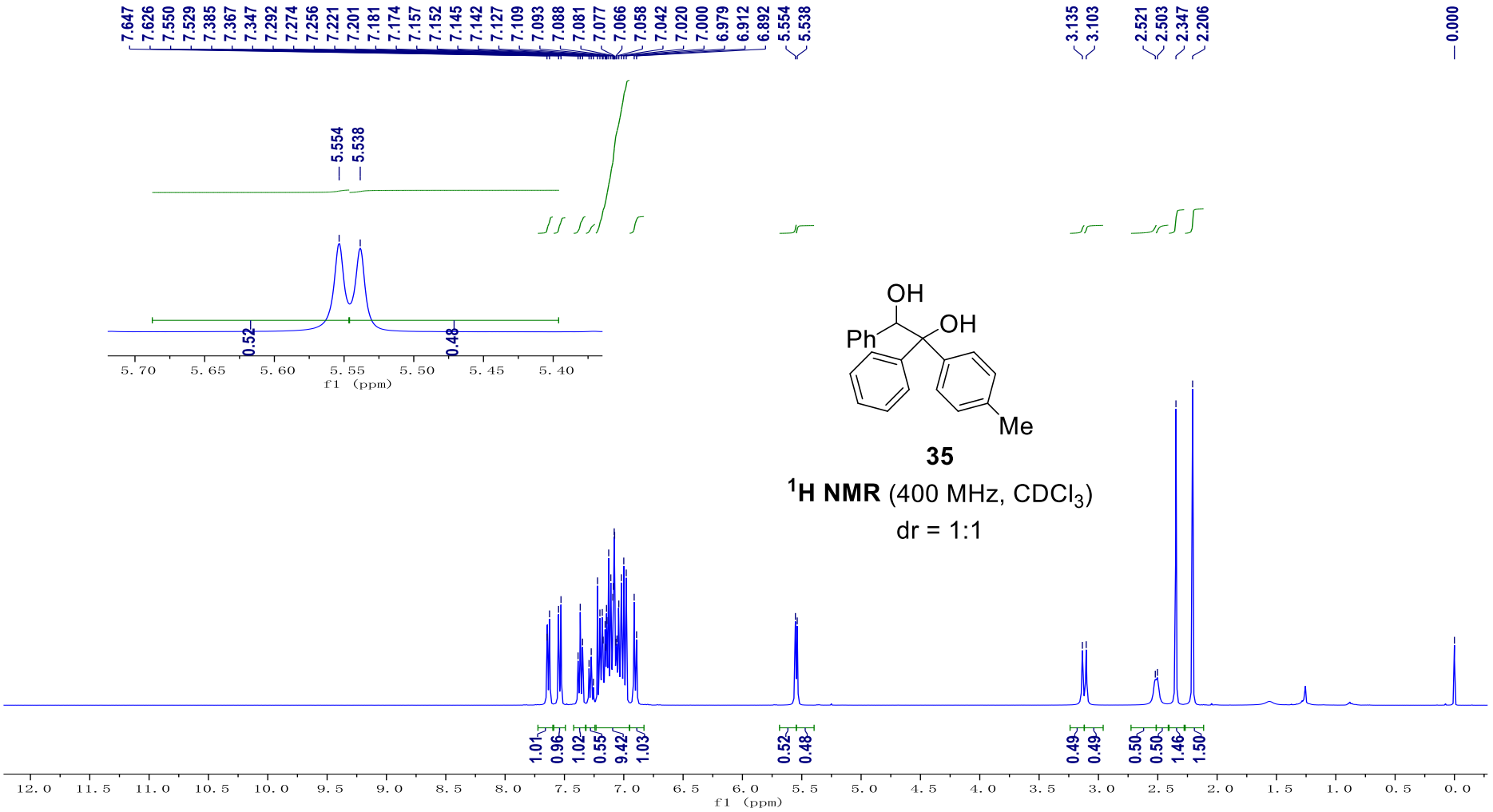

144.978
143.431
142.517
140.774
138.886
138.725
136.801
135.760
135.727
134.982
129.675
128.877
128.331
128.167
128.128
128.060
127.583
127.557
127.529
127.412
127.381
127.355
127.194
126.879
126.560
126.047
124.013
123.633
80.631
80.619
78.021
77.940
77.318
77.000
76.682
20.086
19.815
19.372
19.288
f1 (ppm)
OH
OH
Ph
Me
Me
33
13C NMR (300 MHz, CDCl3)

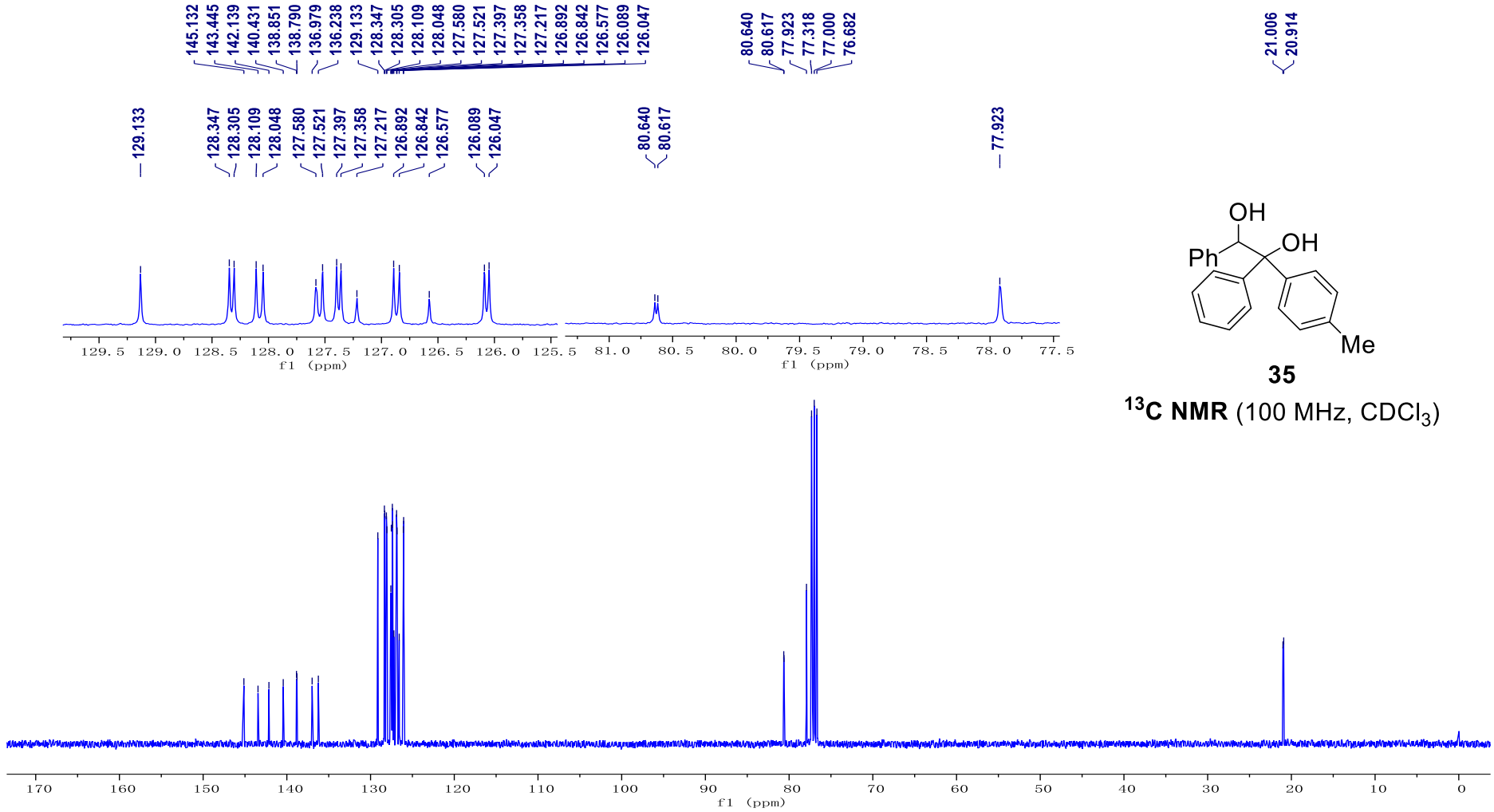

8.194 7.877 7.830 7.808 7.712 7.708 7.697 7.692 7.675 7.671 7.617 7.581 7.559 7.508 7.502 7.493 7.484 7.478 7.403 7.385 7.379 7.374 7.366 7.356 7.353 7.291 7.240 7.236 7.227 7.218 7.214 7.175 7.157 7.146 7.140 7.126 7.121 7.110 7.104 7.101 7.094 7.089 7.081 7.077 7.074 7.067 7.063 7.056 7.042 5.713 5.705 3.299 3.269 2.562 2.554 2.530 2.521 -0.000
5.713
5.705
0.56
0.44
5.95 5.90 5.85 5.80 5.75 5.70 5.65 5.60 5.55 5.50 5.45
f1 (ppm)
OH
OH
Ph
34
1H NMR (400 MHz, CDCl3)
dr = 1.3:1
0.56
1.72
14.57
0.56
0.44
0.56
0.42
0.56
0.42
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



34

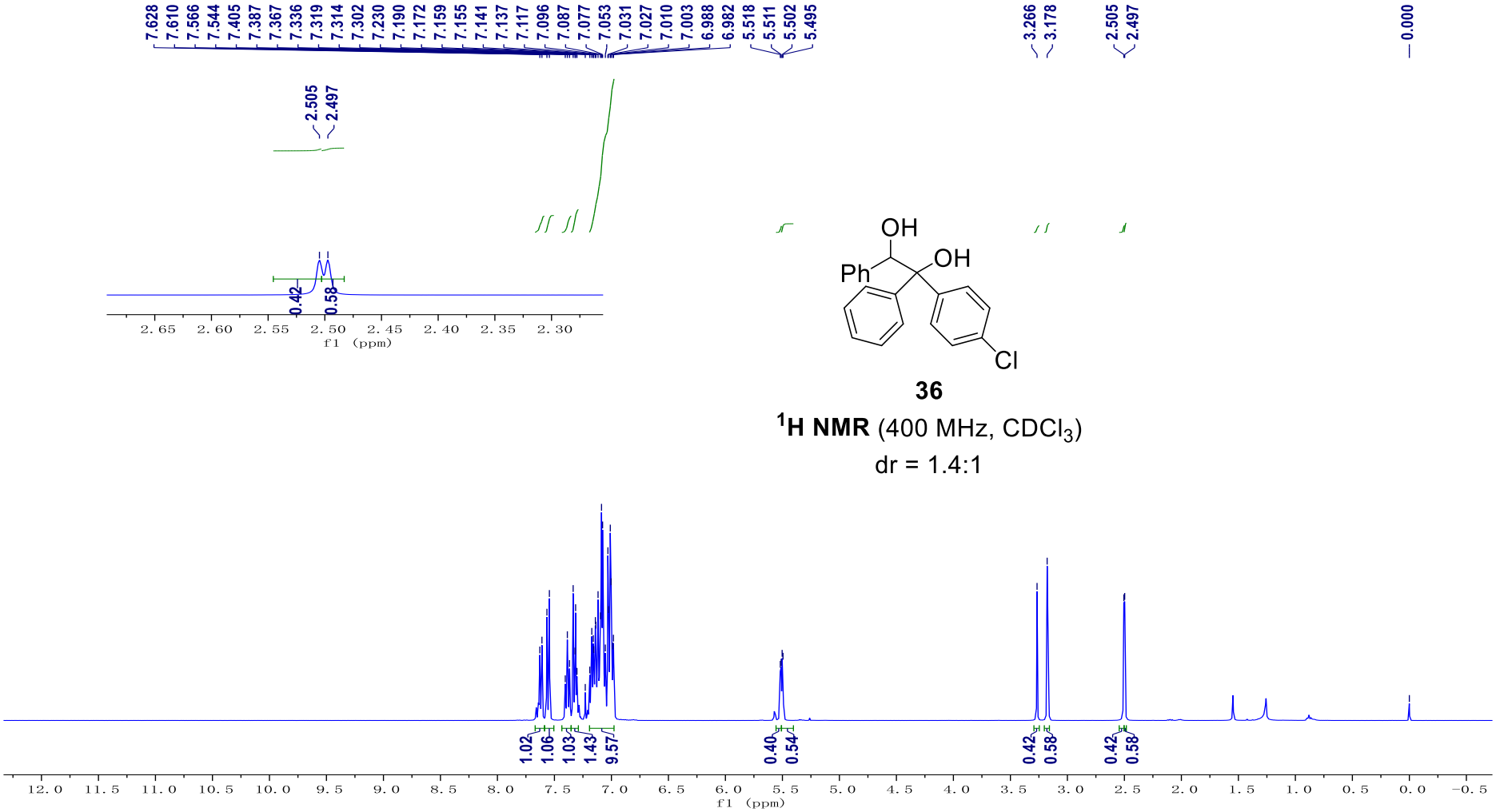
^{13}C NMR (100 MHz, $CDCl_3$)


7.647
7.626
7.550
7.529
7.385
7.367
7.347
7.292
7.274
7.256
7.221
7.201
7.181
7.174
7.157
7.152
7.145
7.142
7.127
7.109
7.093
7.088
7.081
7.077
7.066
7.058
7.042
7.020
7.000
6.979
6.912
6.892
5.554
5.538
3.135
3.103
2.521
2.503
2.347
2.206
0.000
5.554
5.538
0.52
0.48
5.70 5.65 5.60 5.55 5.50 5.45 5.40
f1 (ppm)
OH
OH
Ph
Me
35
1H NMR (400 MHz, CDCl3)
dr = 1:1
1.01
0.96
1.02
0.55
9.42
1.03
0.52
0.48
0.49
0.49
0.50
0.50
1.46
1.50
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



35

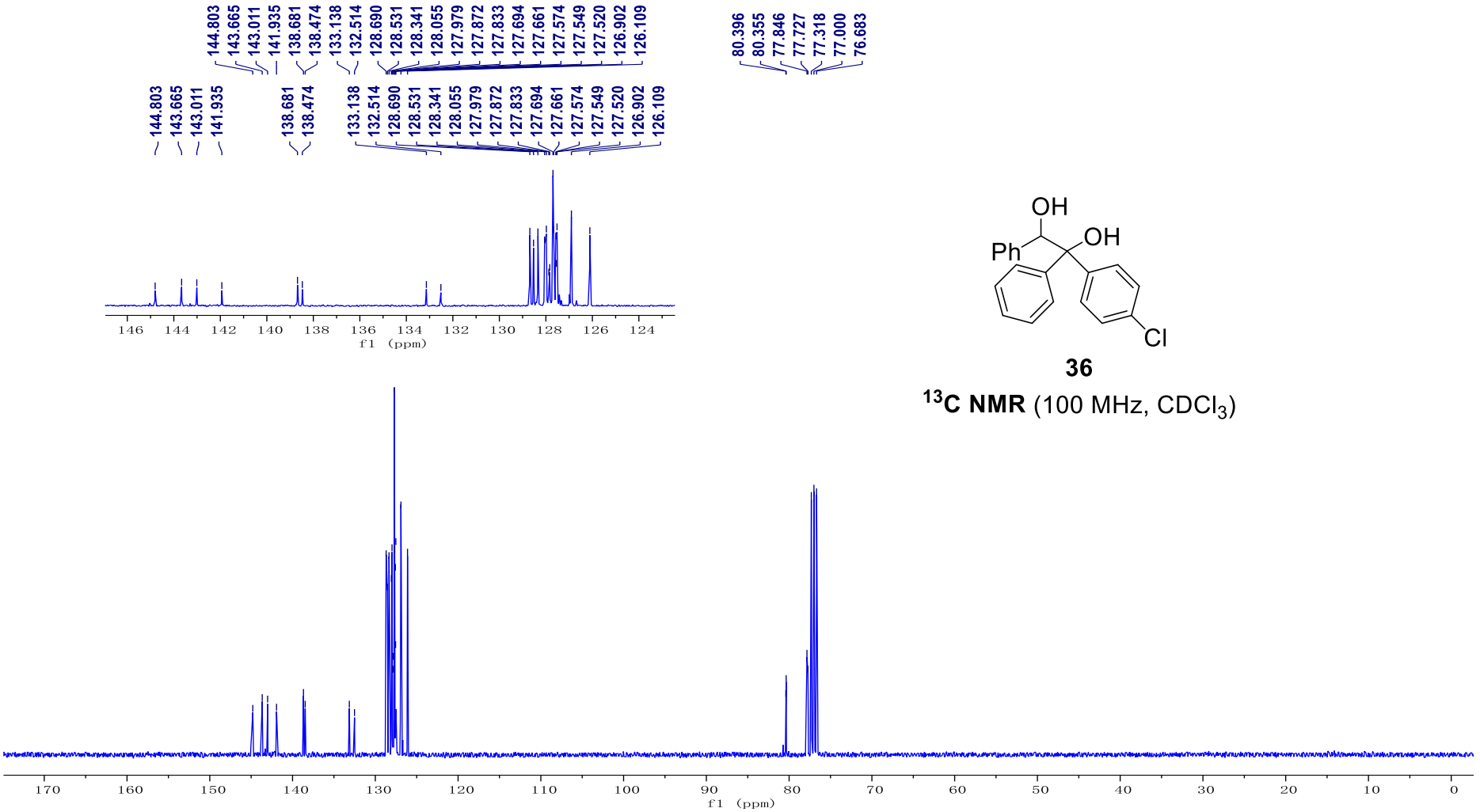
^{13}C NMR (100 MHz, $CDCl_3$)



36

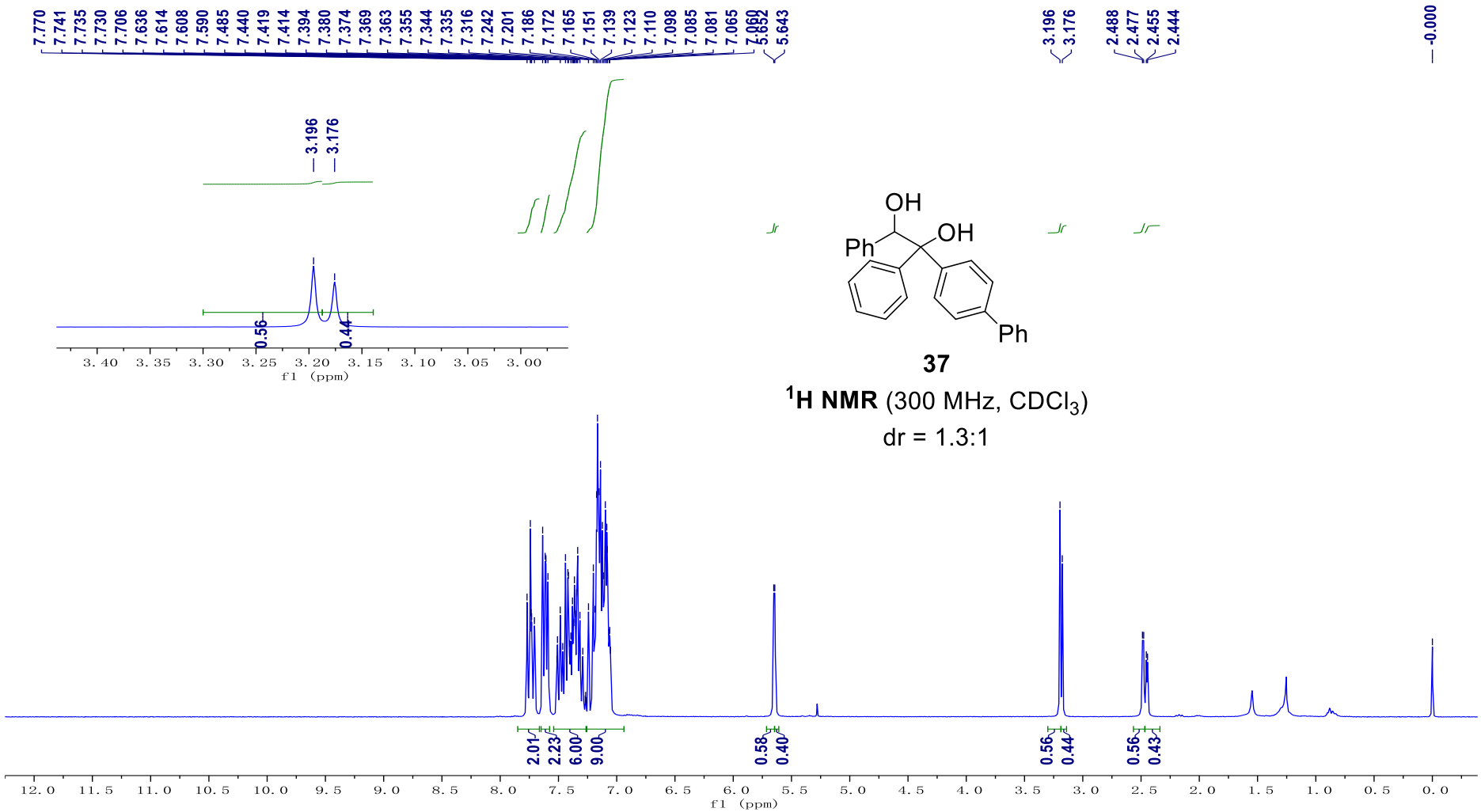
^{1}H NMR (400 MHz, $CDCl_3$)

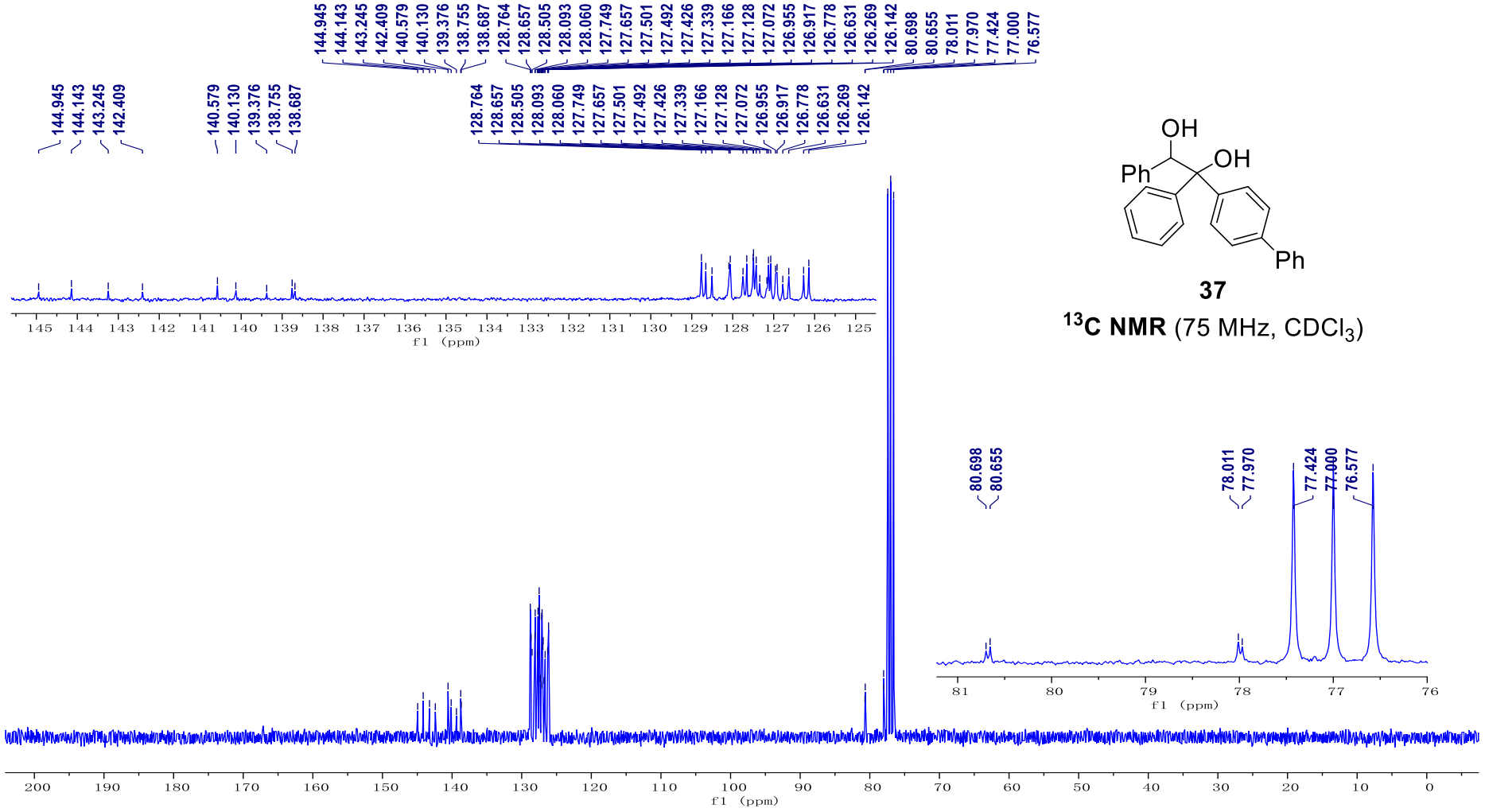
dr = 1.4:1



36

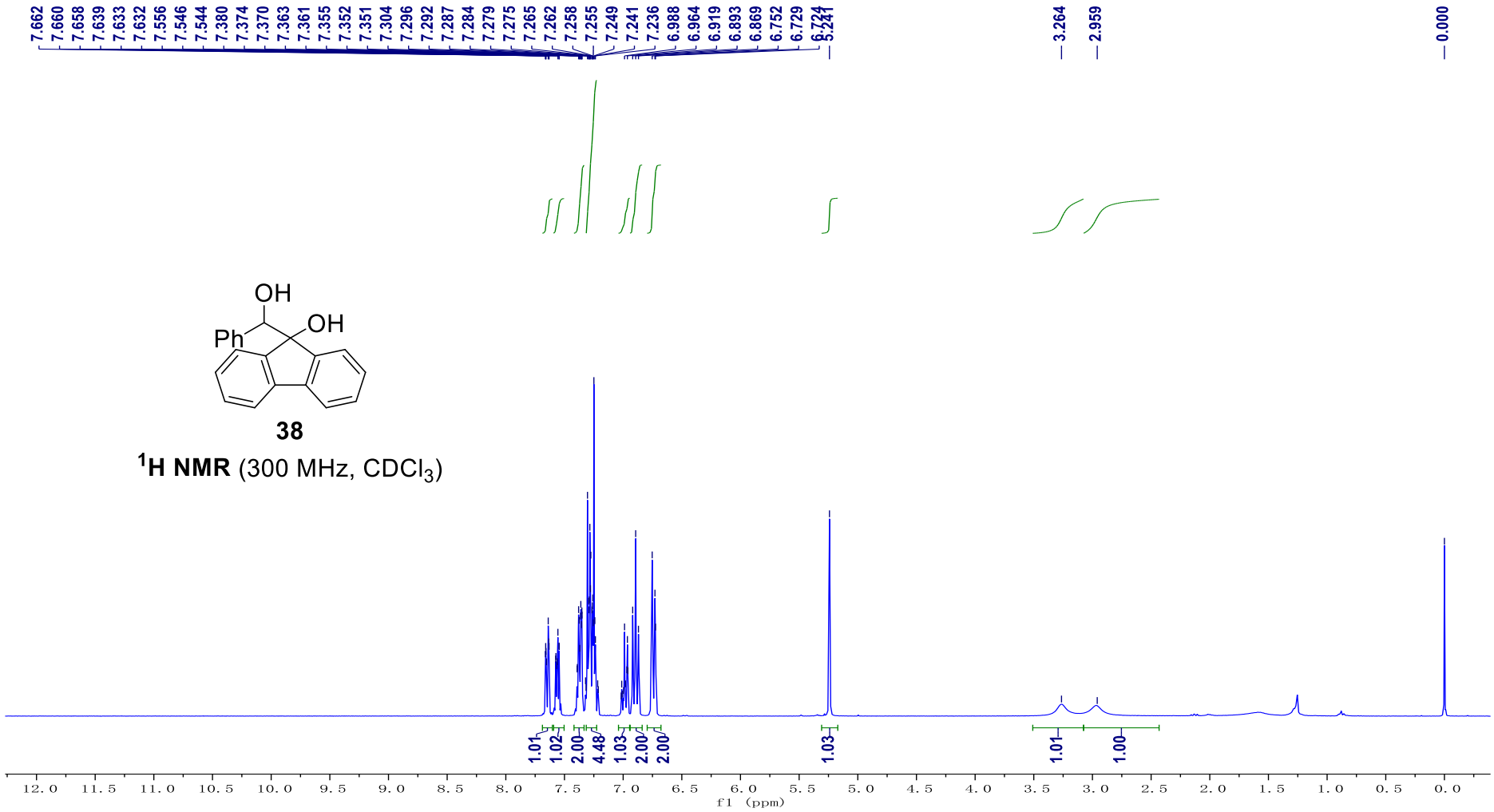
^{13}C NMR (100 MHz, $CDCl_3$)

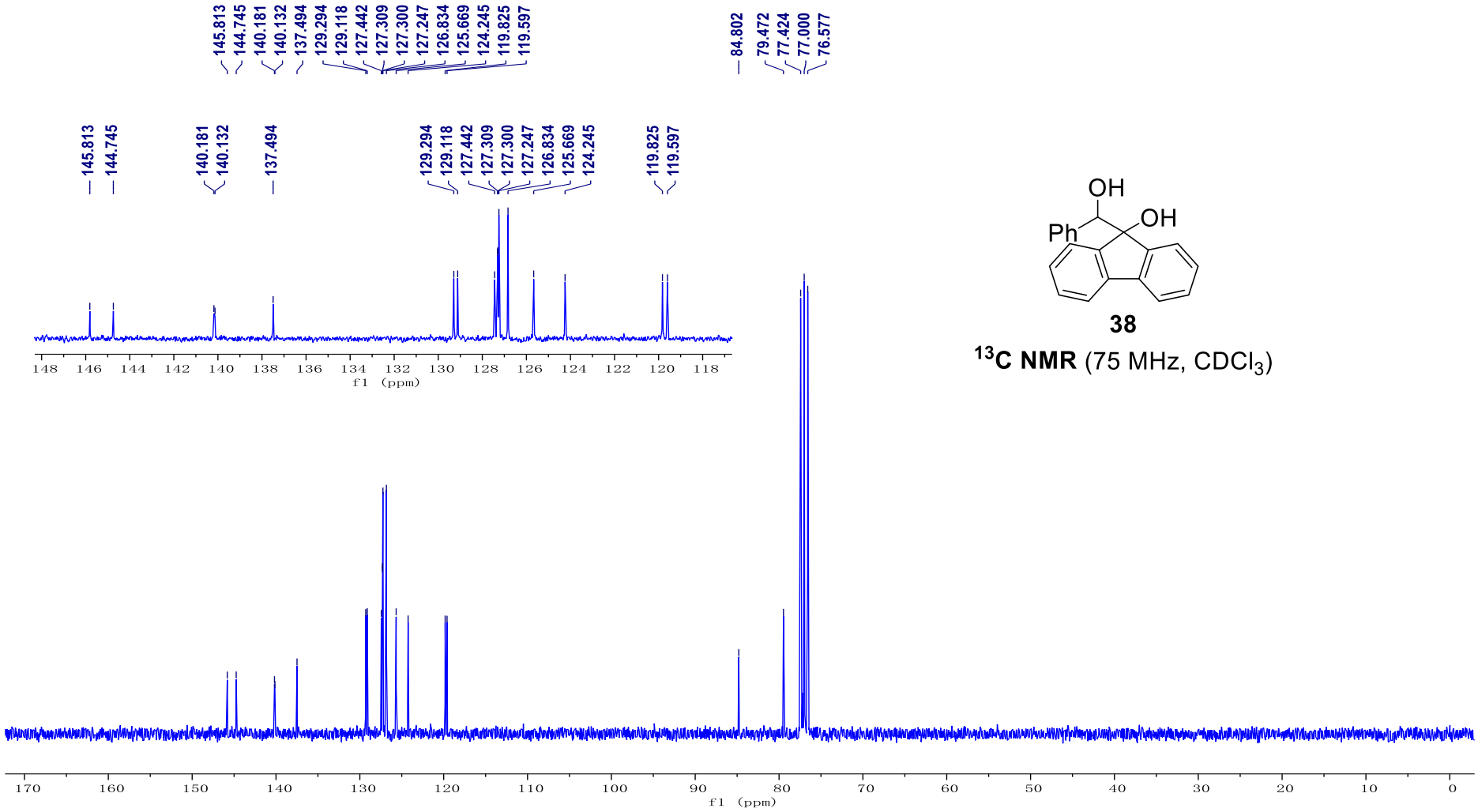

7.770
7.741
7.735
7.730
7.706
7.636
7.614
7.608
7.590
7.485
7.440
7.419
7.414
7.394
7.380
7.374
7.369
7.363
7.355
7.344
7.335
7.316
7.242
7.201
7.186
7.172
7.165
7.151
7.139
7.123
7.110
7.098
7.085
7.081
7.065
5.652
5.643
3.196
3.176
2.488
2.477
2.455
2.444
-0.000
OH
OH
Ph
Ph
37
1H NMR (300 MHz, CDCl3)
dr = 1.3:1
0.56
0.44
f1 (ppm)
2.01
2.23
6.00
9.00
0.58
0.40
0.56
0.44
0.56
0.43
f1 (ppm)



37

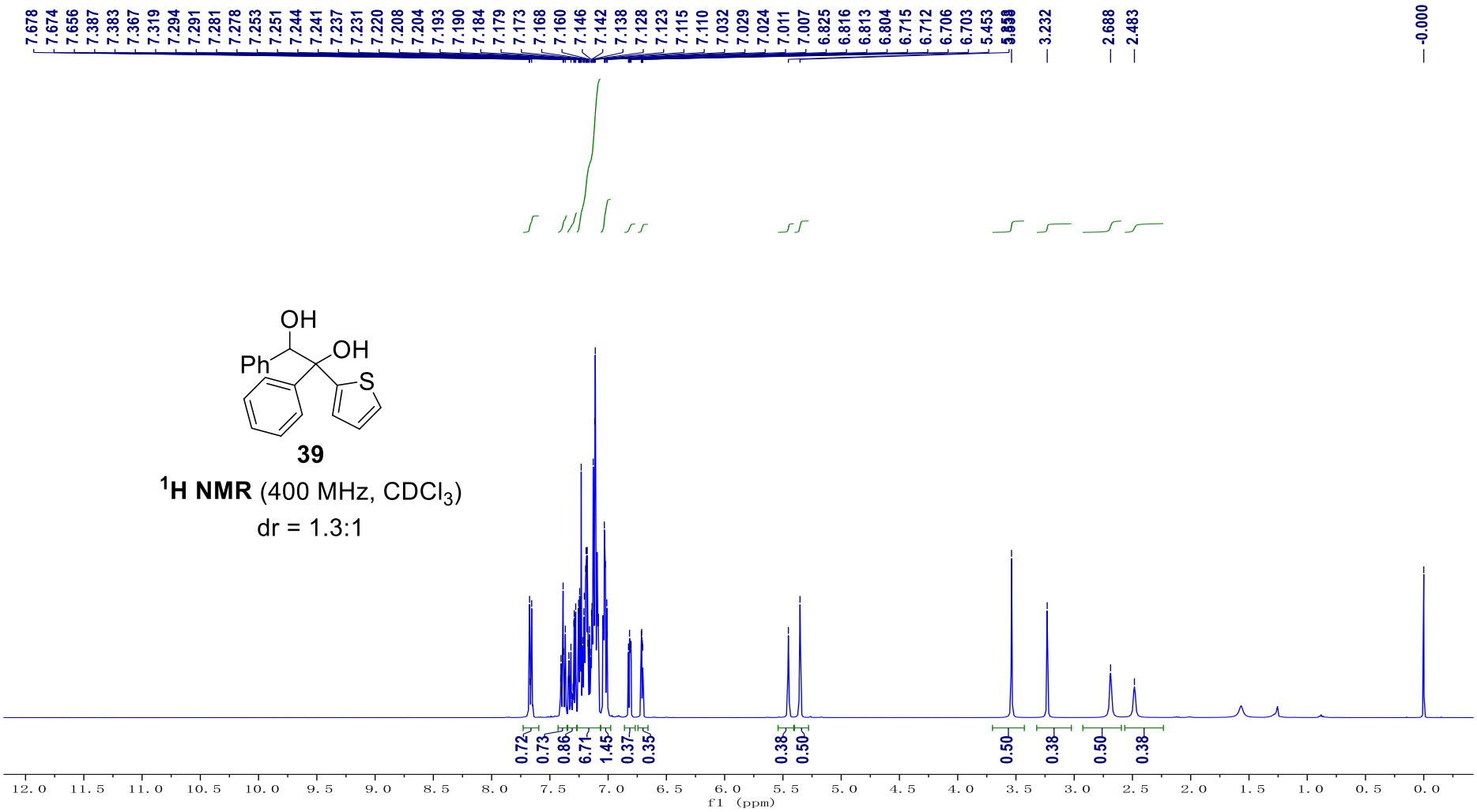
^{13}C NMR (75 MHz, $CDCl_3$)

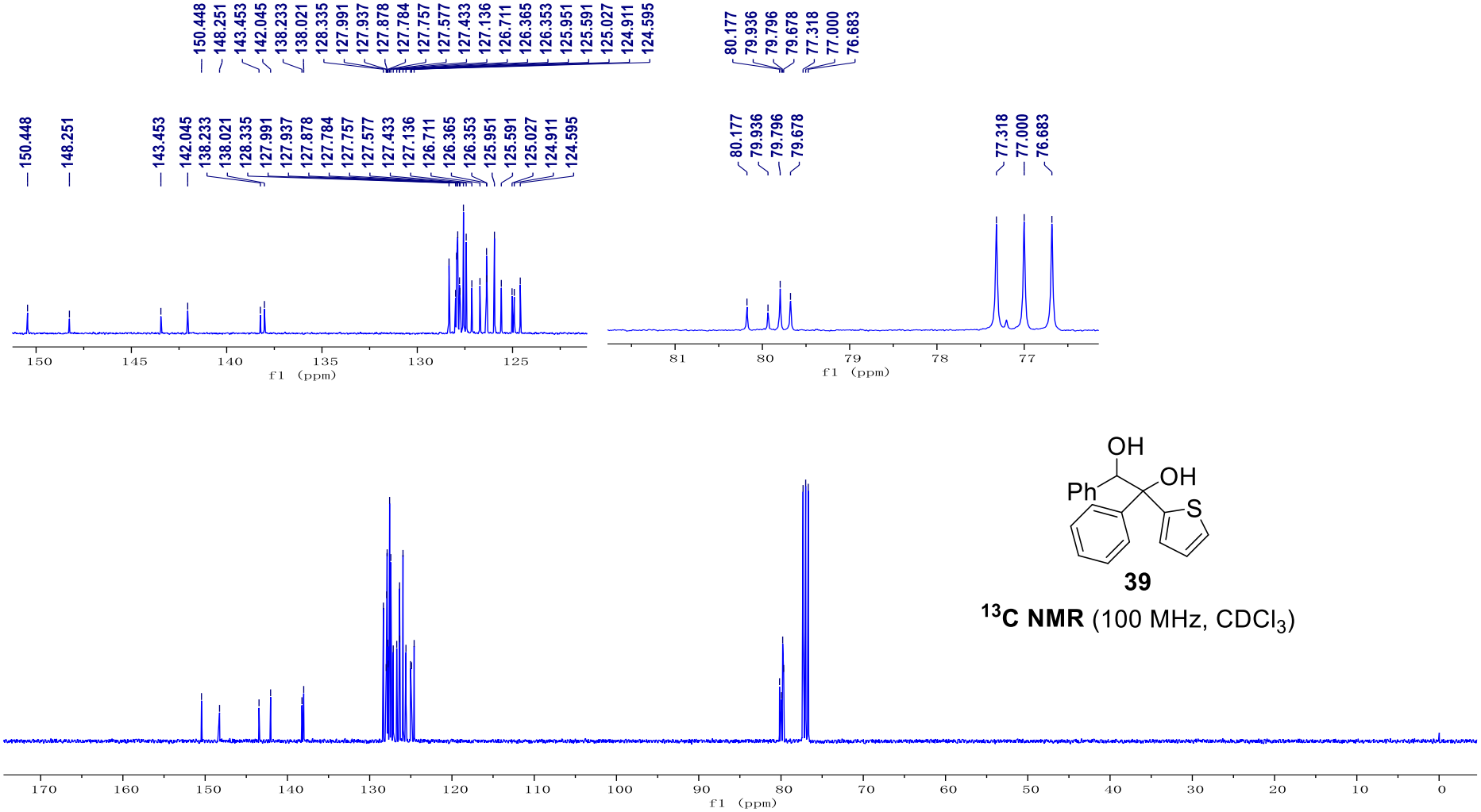

7.662
7.660
7.658
7.639
7.633
7.632
7.556
7.546
7.544
7.380
7.374
7.370
7.363
7.361
7.355
7.352
7.351
7.304
7.296
7.292
7.287
7.284
7.279
7.275
7.265
7.262
7.258
7.255
7.249
7.241
7.236
6.988
6.964
6.919
6.893
6.869
6.752
6.729
5.241
3.264
2.959
0.000
OH
OH
Ph
38
1H NMR (300 MHz, CDCl3)
1.01
1.02
2.00
4.48
1.03
2.00
2.00
1.03
1.01
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

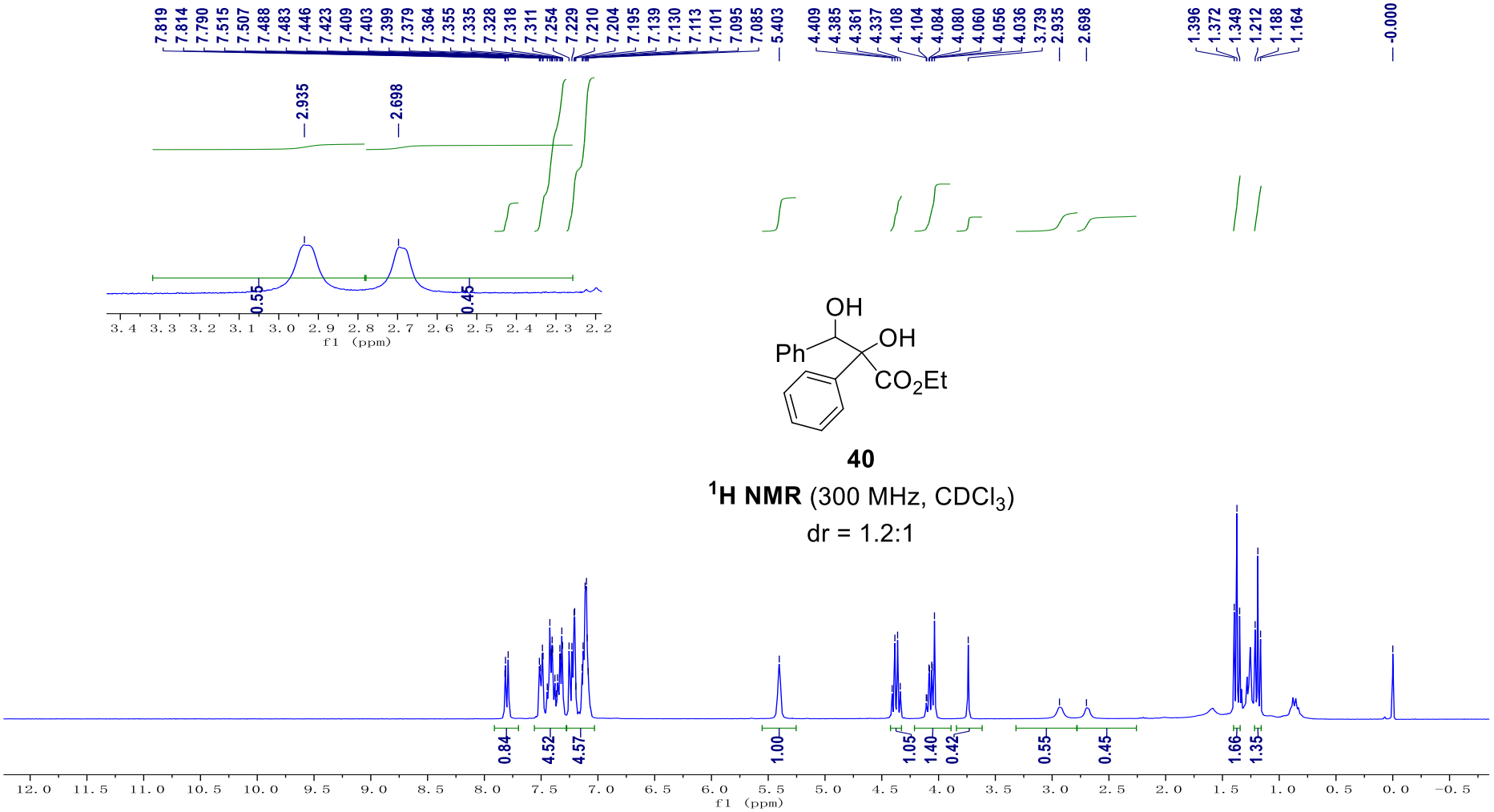


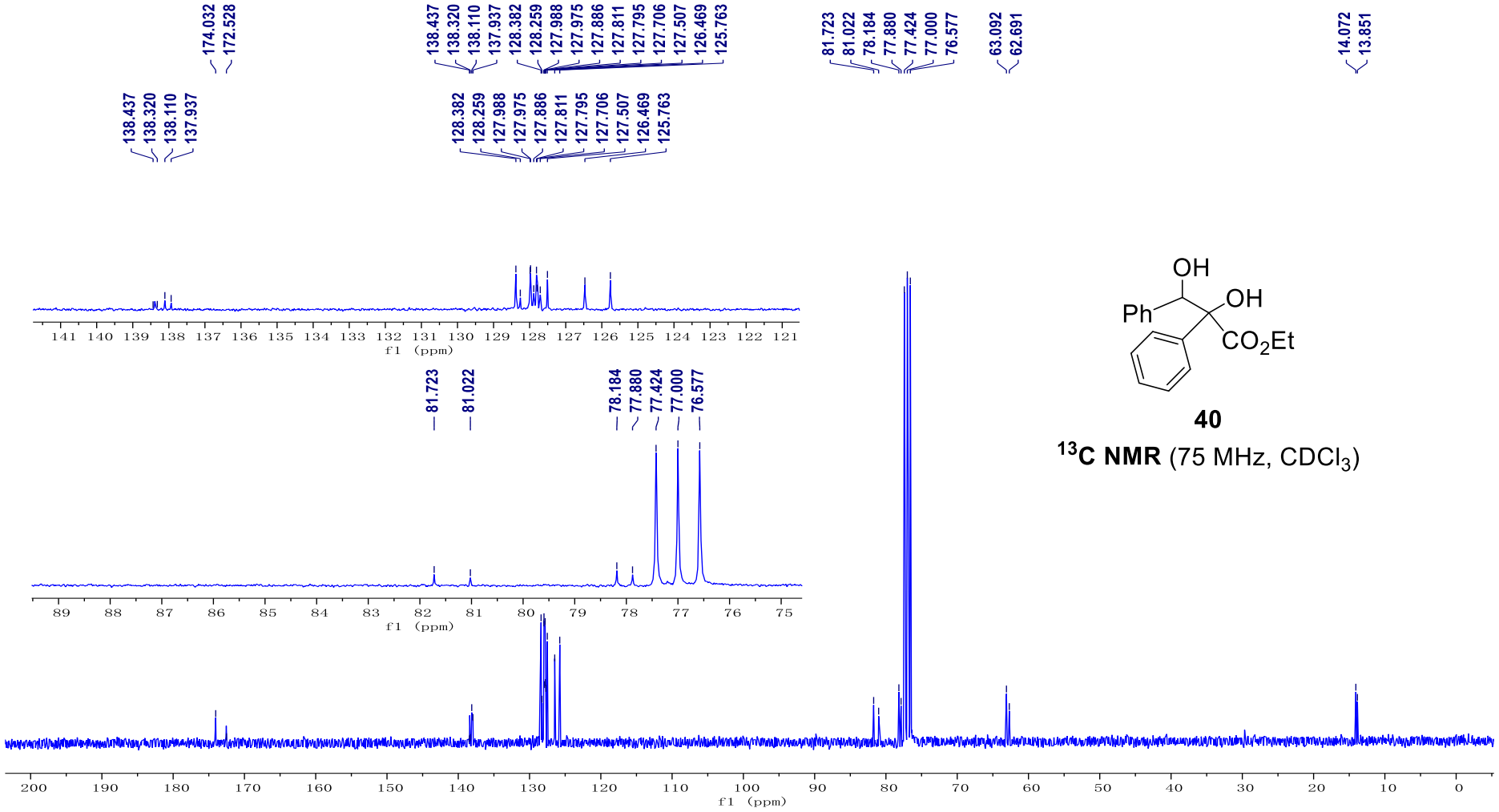
38

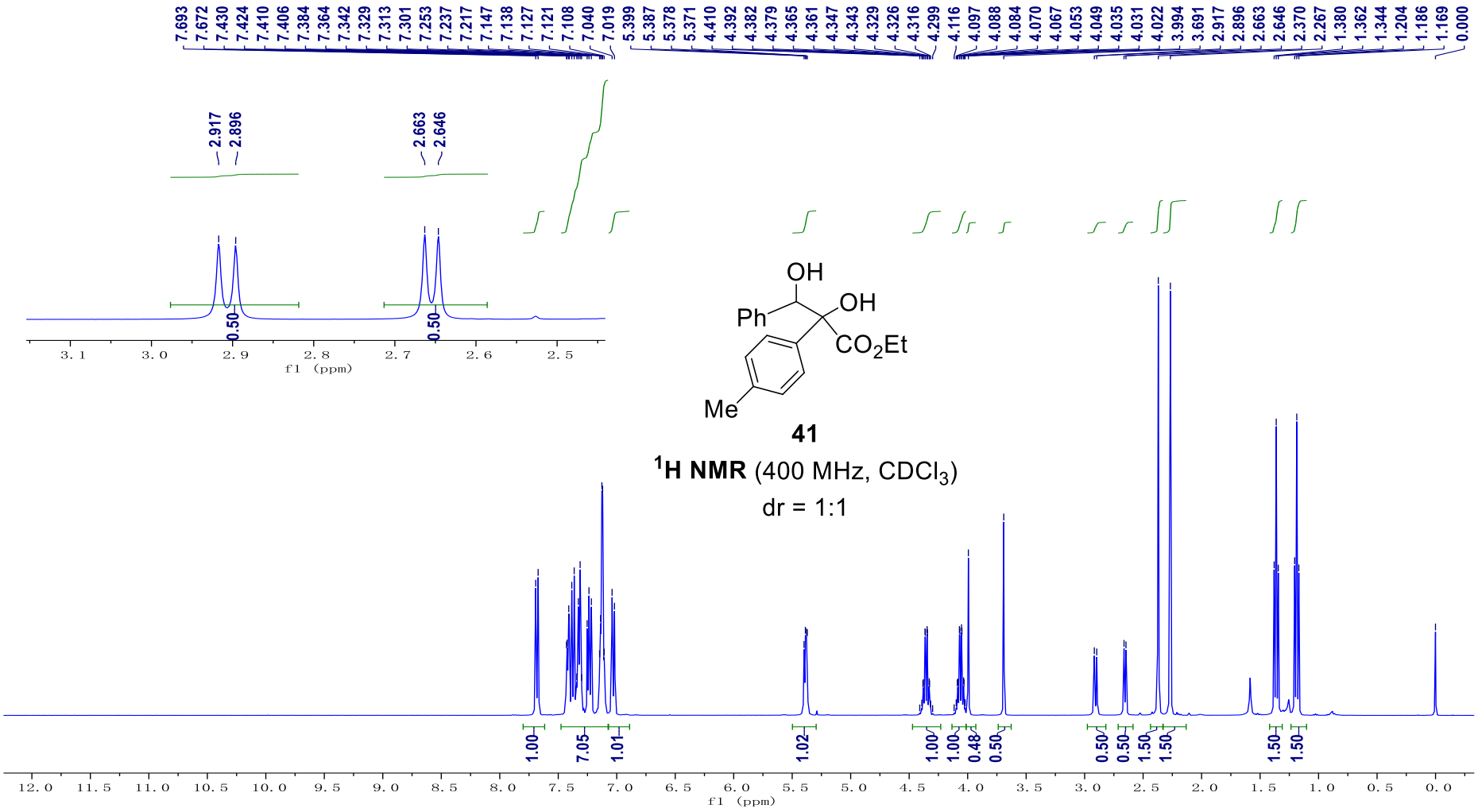
^{13}C NMR (75 MHz, $CDCl_3$)

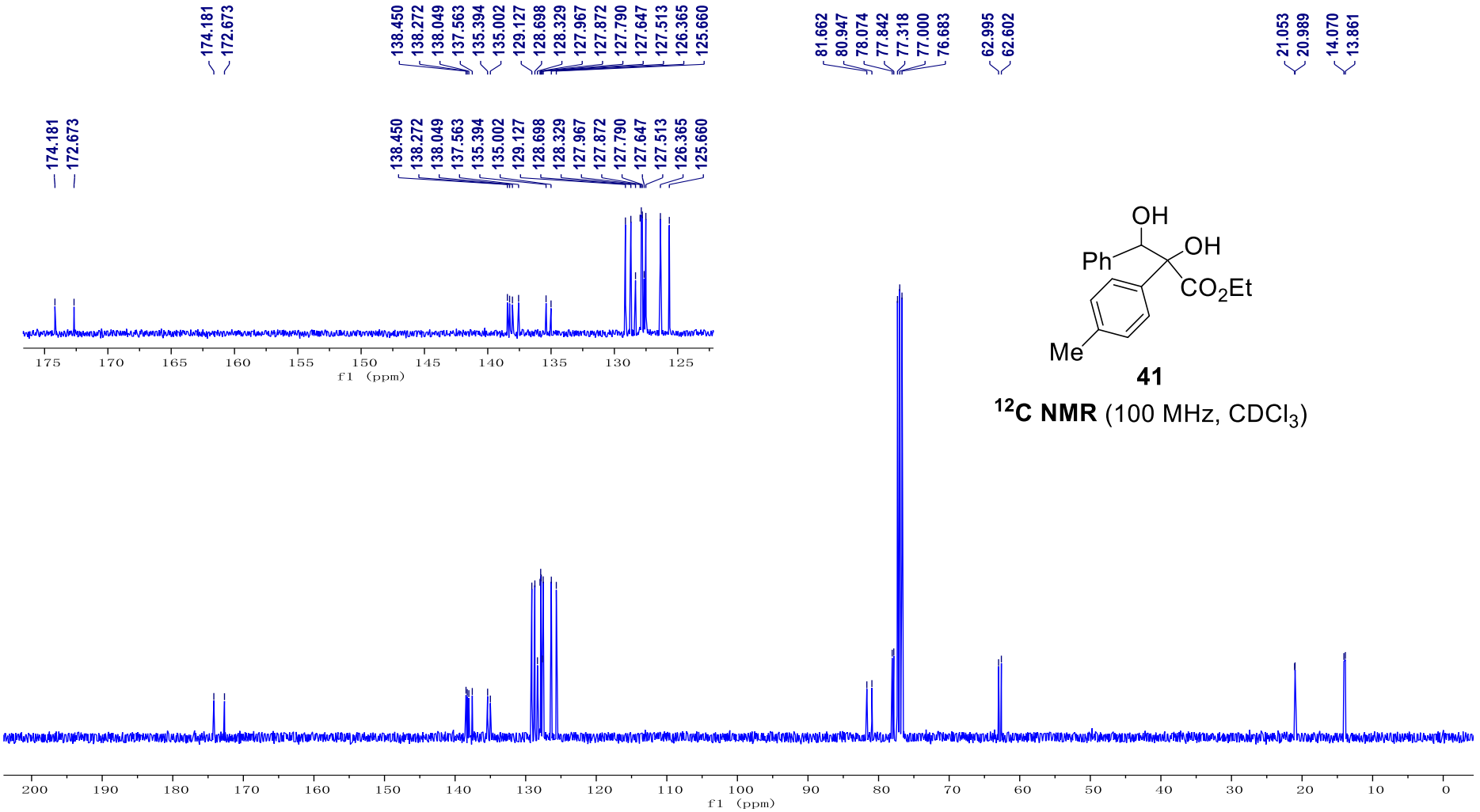

7.678
7.674
7.656
7.387
7.383
7.367
7.319
7.294
7.291
7.281
7.278
7.253
7.251
7.244
7.241
7.237
7.231
7.220
7.208
7.204
7.193
7.190
7.184
7.179
7.173
7.168
7.160
7.146
7.142
7.138
7.128
7.123
7.115
7.110
7.032
7.029
7.024
7.011
7.007
6.825
6.816
6.813
6.804
6.715
6.712
6.706
6.703
5.453
3.232
2.688
2.483
-0.000
OH
OH
Ph
S
39
1H NMR (400 MHz, CDCl3)
dr = 1.3:1
0.72
0.73
0.86
6.71
1.45
0.37
0.35
0.38
0.50
0.50
0.38
0.50
0.38
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

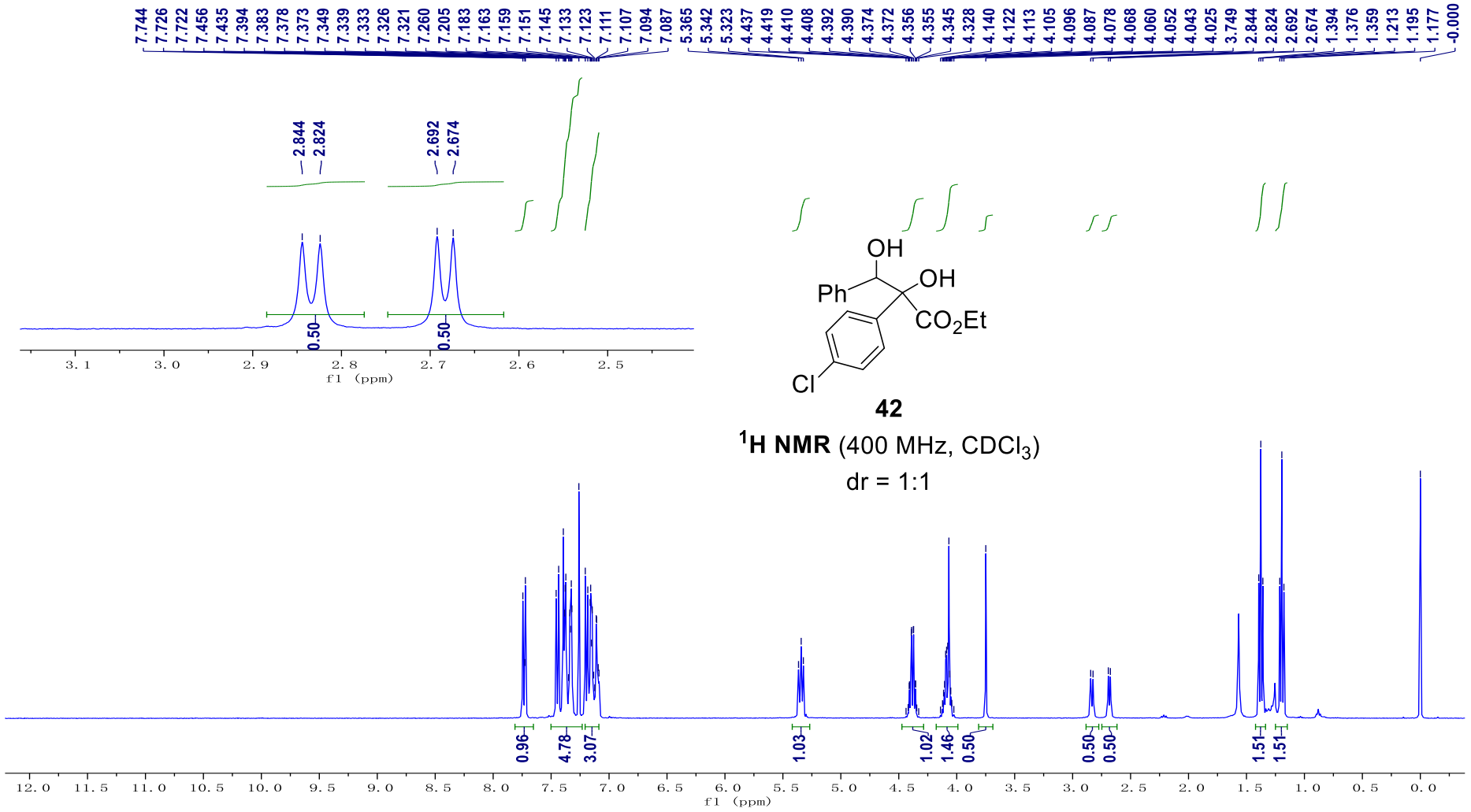

150.448
148.251
143.453
142.045
138.233
138.021
128.335
127.991
127.937
127.878
127.784
127.757
127.577
127.433
127.136
126.711
126.365
126.353
125.951
125.591
125.027
124.911
124.595
80.177
79.936
79.796
79.678
77.318
77.000
76.683
f1 (ppm)
OH
OH
Ph
S
39
13C NMR (100 MHz, CDCl3)

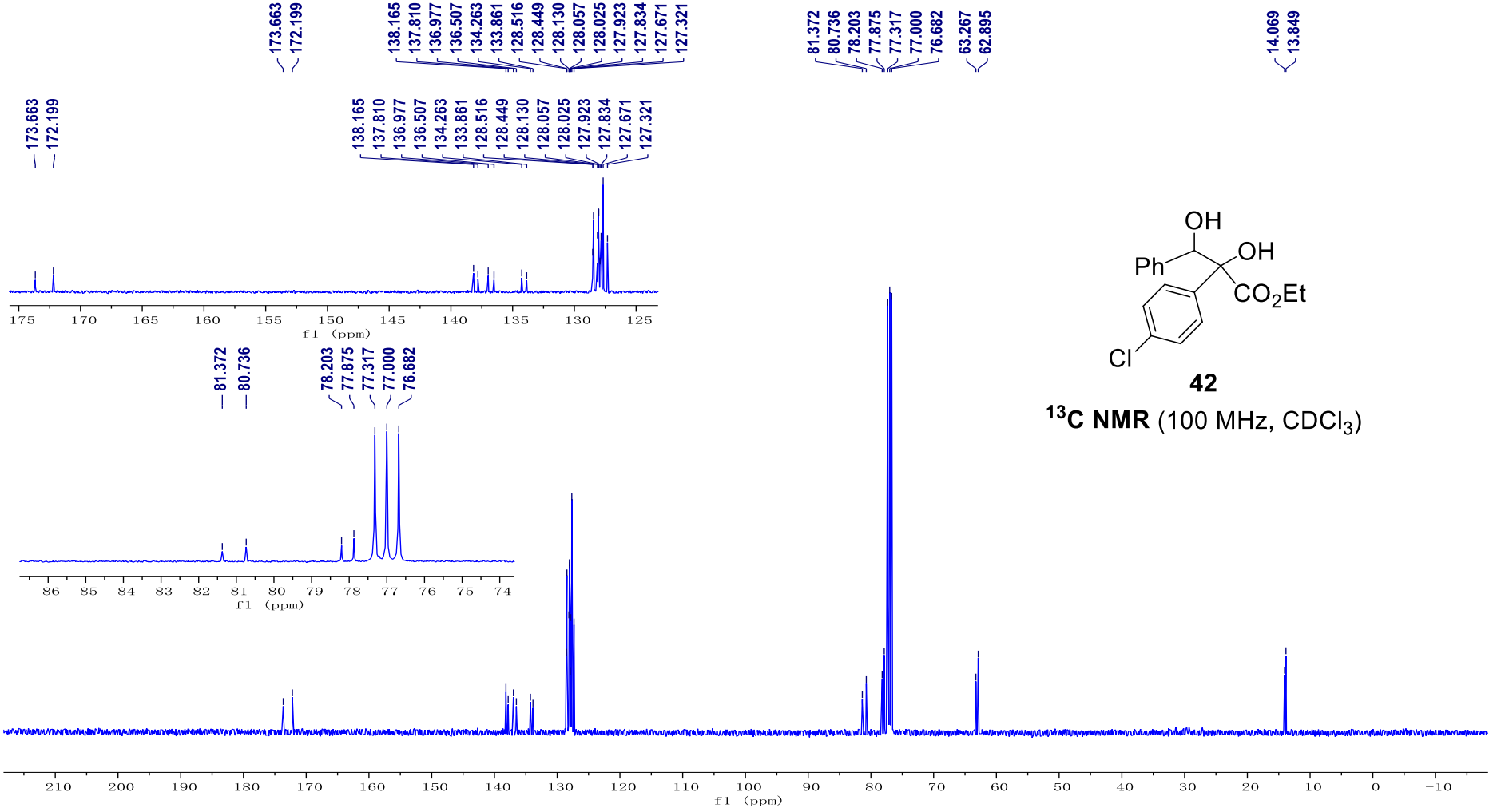

OH
Ph
OH
CO2Et
40
1H NMR (300 MHz, CDCl3)
dr = 1.2:1
f1 (ppm)


174.032
172.528
138.437
138.320
138.110
137.937
128.382
128.259
127.988
127.975
127.886
127.811
127.795
127.706
127.507
126.469
125.763
81.723
81.022
78.184
77.880
77.424
77.000
76.577
63.092
62.691
14.072
13.851
f1 (ppm)
OH
Ph
OH
CO2Et
40
13C NMR (75 MHz, CDCl3)


7.693 7.672 7.430 7.424 7.410 7.406 7.384 7.364 7.342 7.329 7.313 7.301 7.253 7.237 7.217 7.147 7.138 7.127 7.121 7.108 7.040 7.019
5.399 5.387 5.378 5.371 4.410 4.392 4.382 4.379 4.365 4.361 4.347 4.343 4.329 4.326 4.316 4.299
4.116 4.097 4.088 4.084 4.070 4.067 4.053 4.049 4.035 4.031 4.022 3.994 3.691 2.917 2.896 2.663 2.646 2.370 2.267 1.380 1.362 1.344 1.204 1.186 1.169 0.000
2.917 2.896
2.663 2.646
0.50 0.50
3.1 3.0 2.9 2.8 2.7 2.6 2.5
f1 (ppm)
OH
Ph
OH
CO2Et
Me
41
1H NMR (400 MHz, CDCl3)
dr = 1:1
1.00 7.05 1.01 1.02 1.00 1.00 0.48 0.50 0.50 0.50 1.50 1.50 1.50 1.50
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

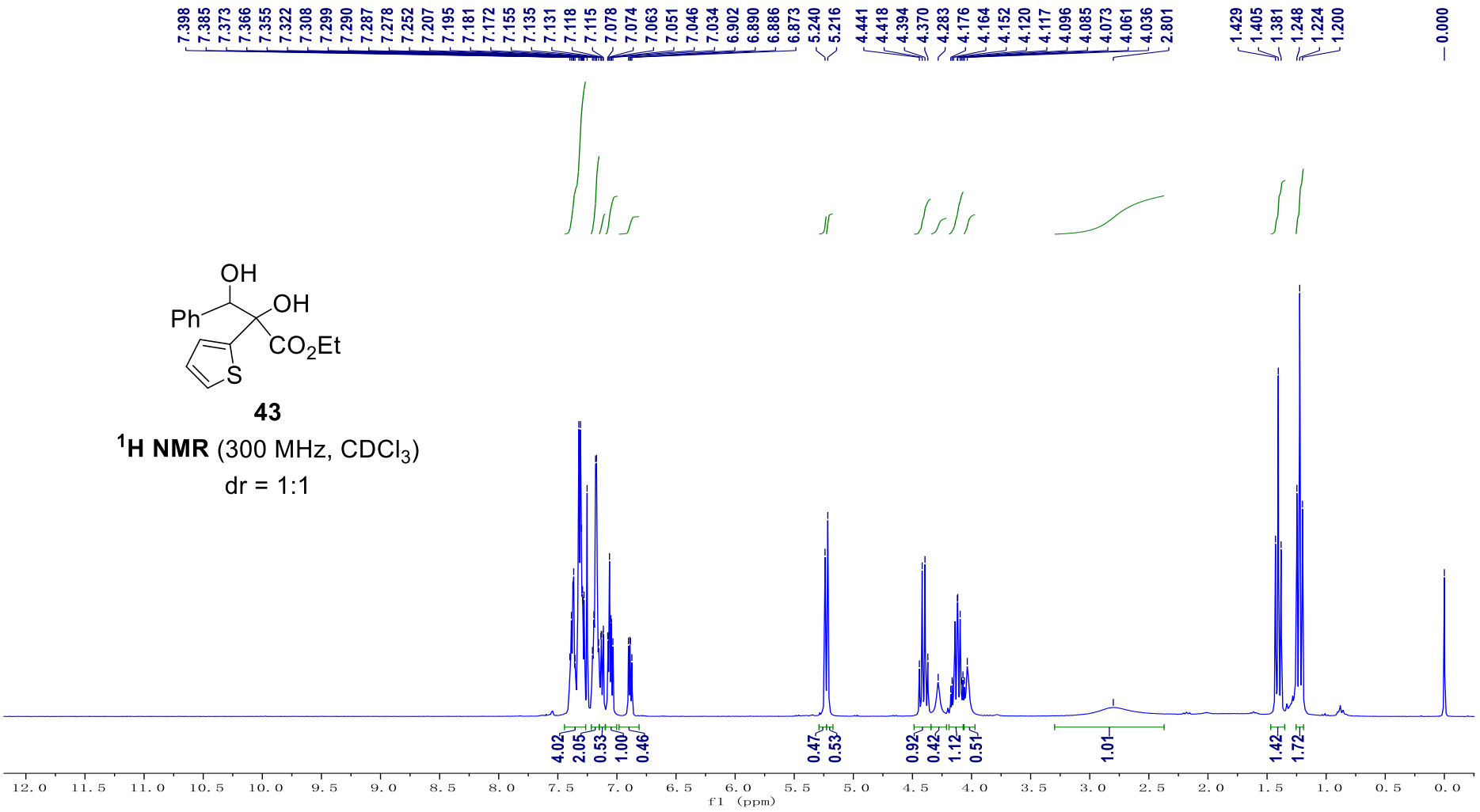

174.181
172.673
138.450
138.272
138.049
137.563
135.394
135.002
129.127
128.698
128.329
127.967
127.872
127.790
127.647
127.513
126.365
125.660
81.662
80.947
78.074
77.842
77.318
77.000
76.683
62.995
62.602
21.053
20.989
14.070
13.861
f1 (ppm)
OH
Ph
OH
CO2Et
Me
41
12C NMR (100 MHz, CDCl3)

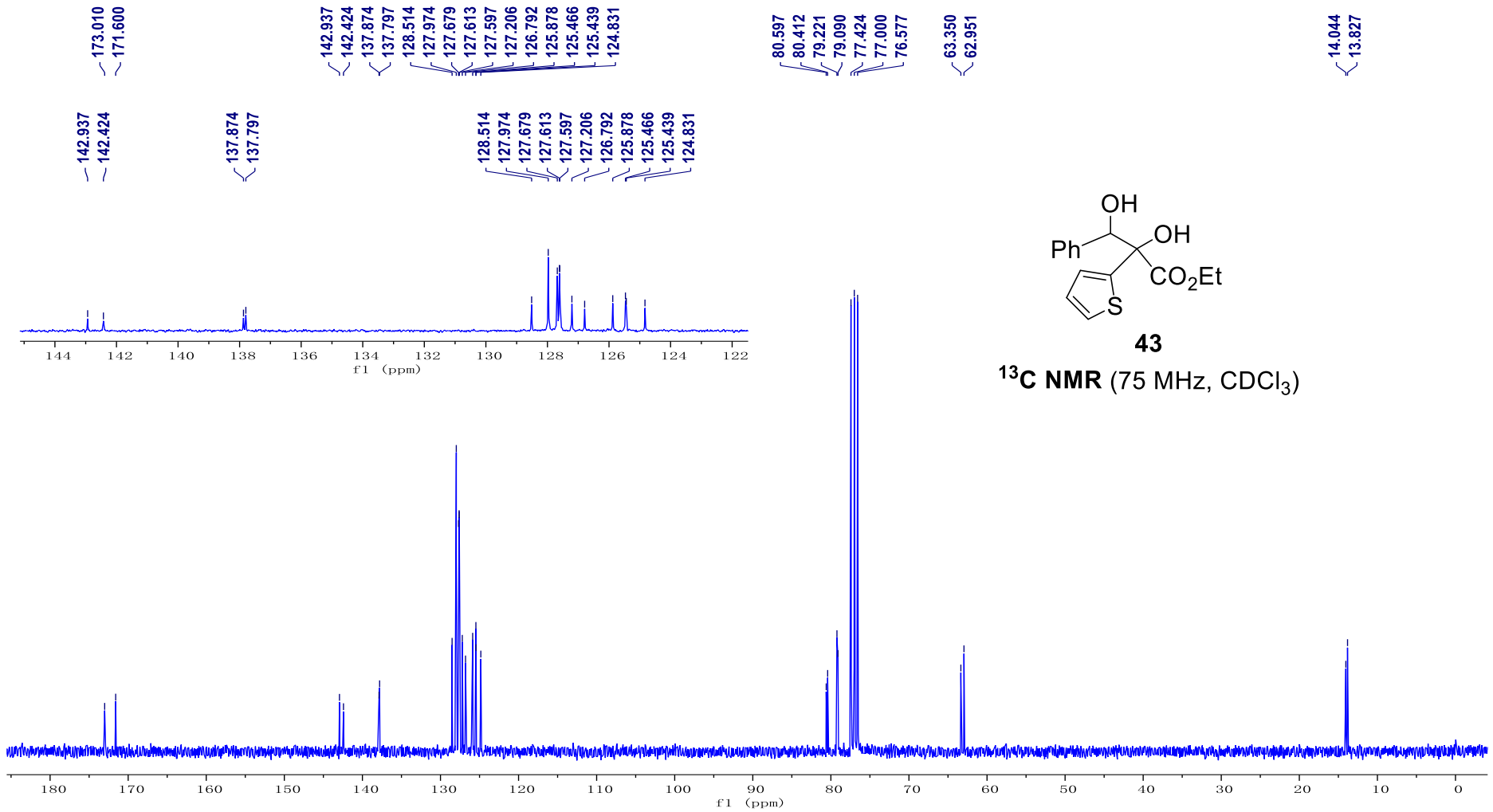

7.744 7.726 7.722 7.456 7.435 7.394 7.383 7.378 7.373 7.349 7.339 7.333 7.326 7.321 7.260 7.205 7.183 7.163 7.159 7.151 7.145 7.133 7.123 7.111 7.107 7.094 7.087 5.365 5.342 5.323 4.437 4.419 4.410 4.408 4.392 4.390 4.374 4.372 4.356 4.355 4.345 4.328 4.140 4.122 4.113 4.105 4.096 4.087 4.078 4.068 4.060 4.052 4.043 4.025 3.749 2.844 2.824 2.692 2.674 1.394 1.376 1.359 1.213 1.195 1.177 -0.000
2.844 2.824 2.692 2.674
0.50 0.50
3.1 3.0 2.9 2.8 2.7 2.6 2.5
f1 (ppm)
OH
Ph
OH
CO2Et
Cl
42
1H NMR (400 MHz, CDCl3)
dr = 1:1
0.96 4.78 3.07 1.03 1.02 1.46 0.50 0.50 0.50 1.51 1.51
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

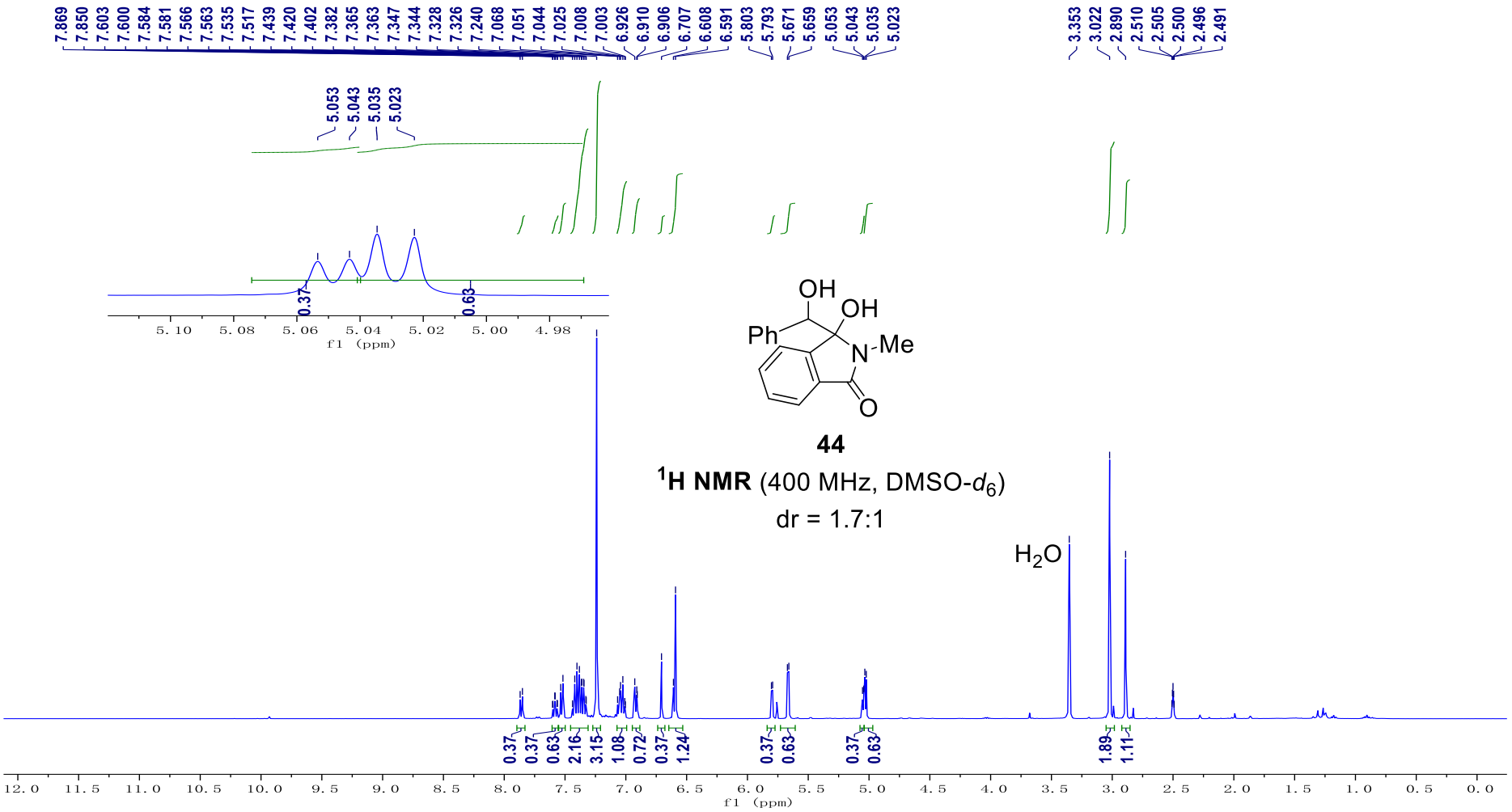


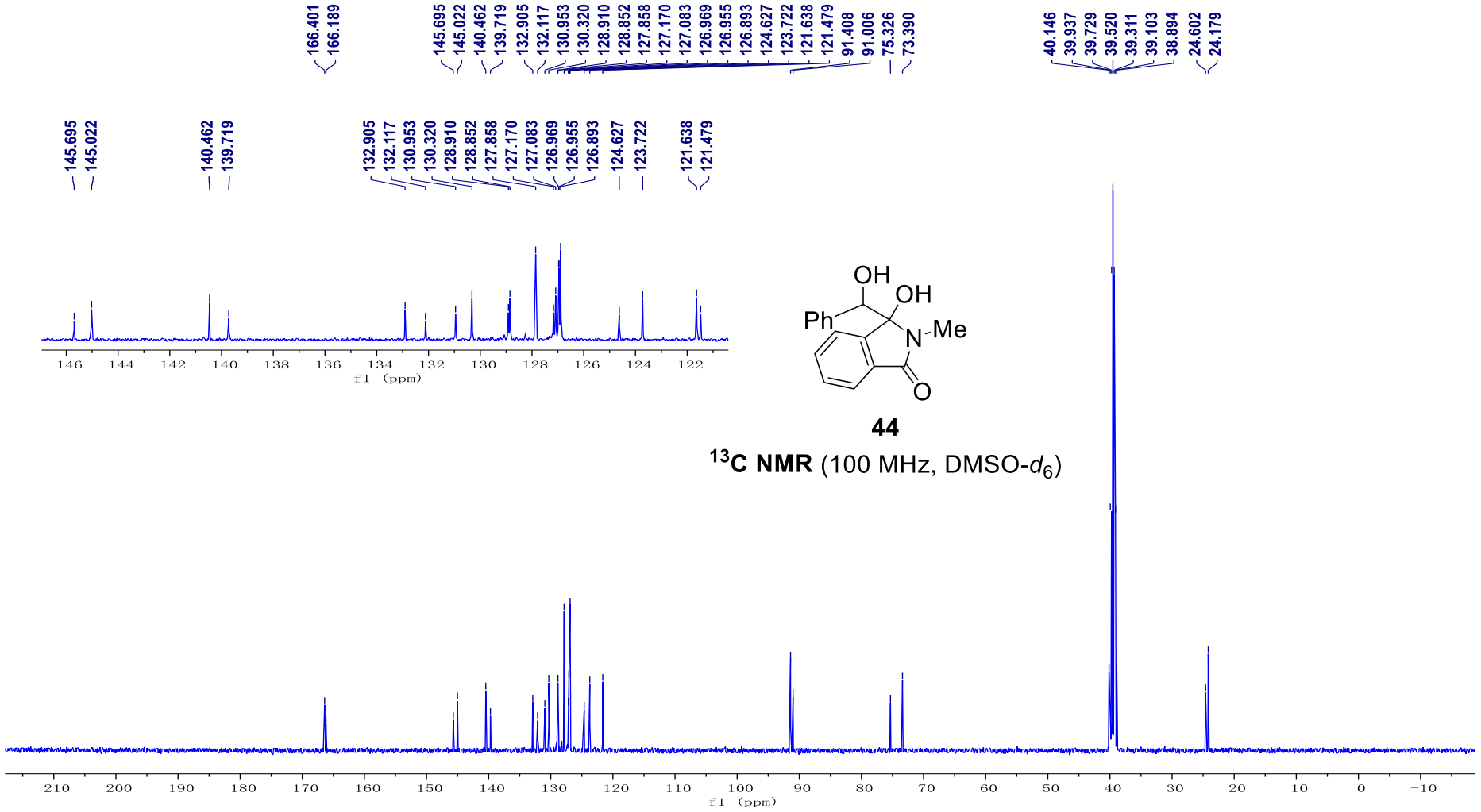
42

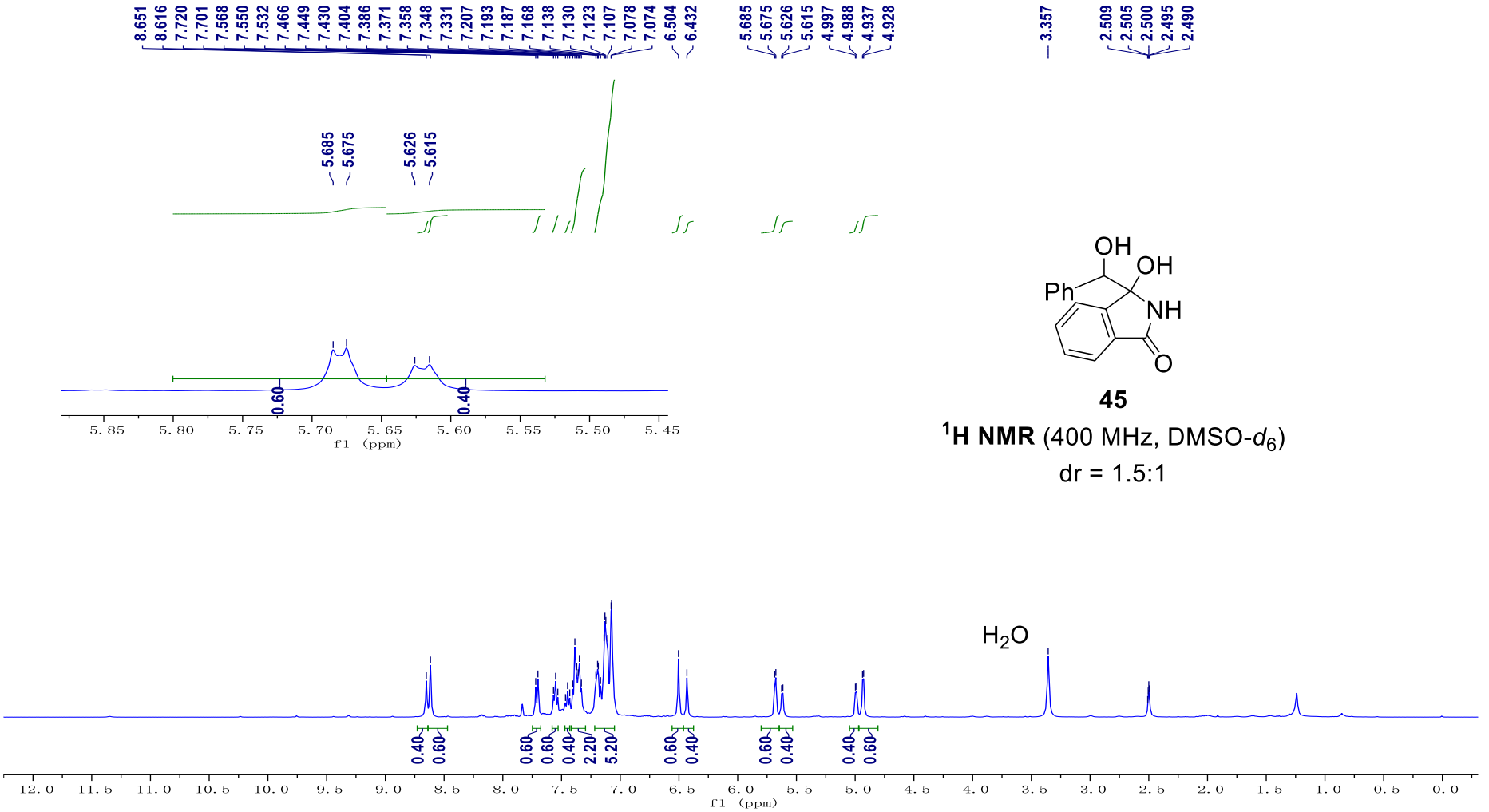
^{13}C NMR (100 MHz, $CDCl_3$)

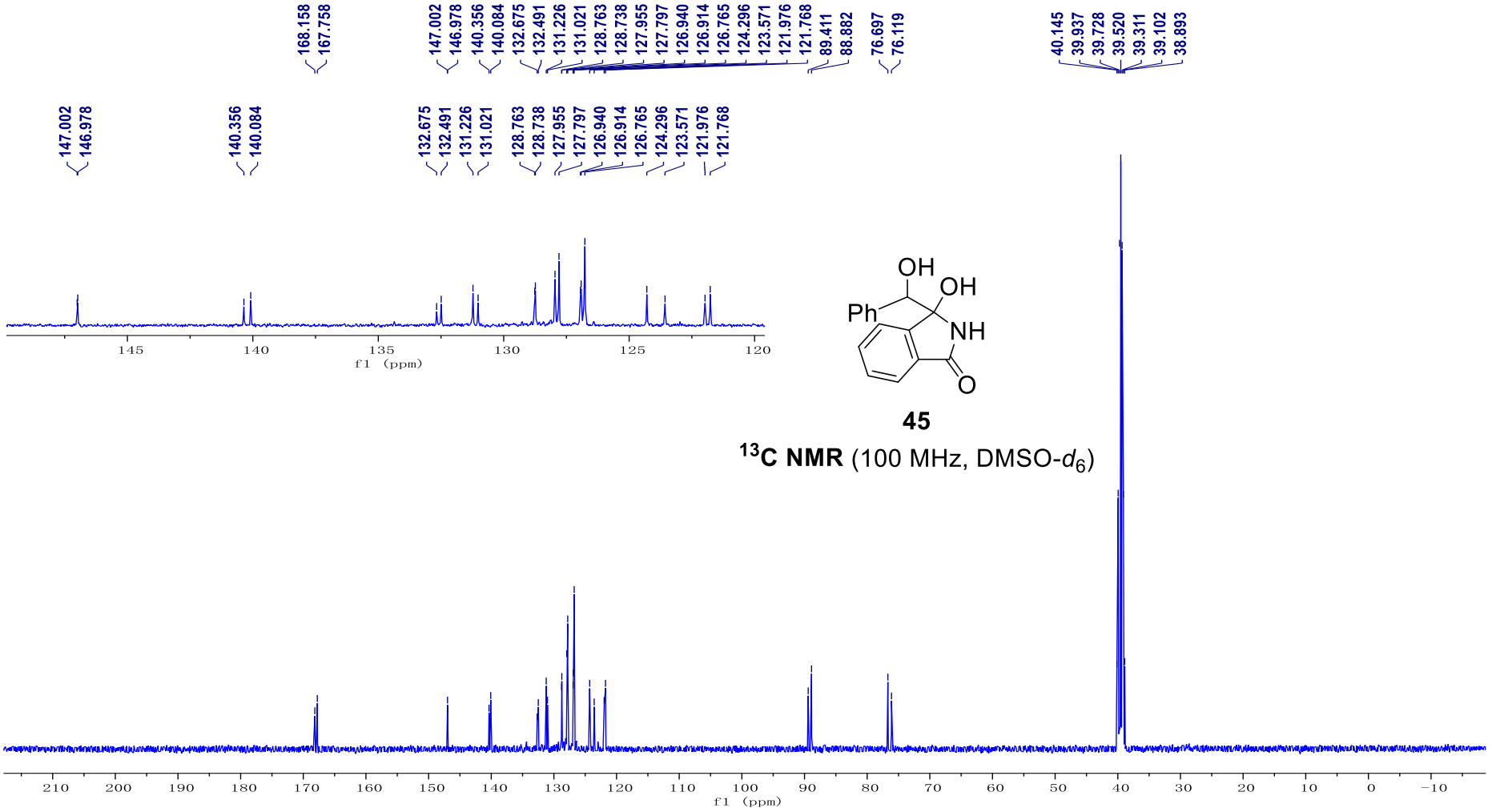

7.398
7.385
7.373
7.366
7.355
7.322
7.308
7.299
7.290
7.287
7.278
7.252
7.207
7.195
7.181
7.172
7.155
7.135
7.131
7.118
7.115
7.078
7.074
7.063
7.051
7.046
7.034
6.902
6.890
6.886
6.873
5.240
5.216
4.441
4.418
4.394
4.370
4.283
4.176
4.164
4.152
4.120
4.117
4.096
4.085
4.073
4.061
4.036
2.801
1.429
1.405
1.381
1.248
1.224
1.200
0.000
OH
Ph
OH
CO2Et
S
43
1H NMR (300 MHz, CDCl3)
dr = 1:1
4.02
2.05
0.53
1.00
0.46
0.47
0.53
0.92
0.42
1.12
0.51
1.01
1.42
1.72
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


173.010
171.600
142.937
142.424
137.874
137.797
128.514
127.974
127.679
127.613
127.597
127.206
126.792
125.878
125.466
125.439
124.831
80.597
80.412
79.221
79.090
77.424
77.000
76.577
63.350
62.951
14.044
13.827
OH
OH
Ph
CO2Et
S
43
13C NMR (75 MHz, CDCl3)
f1 (ppm)


7.869
7.850
7.603
7.600
7.584
7.581
7.566
7.563
7.535
7.517
7.439
7.420
7.402
7.382
7.365
7.363
7.347
7.344
7.328
7.326
7.240
7.068
7.051
7.044
7.025
7.008
7.003
6.926
6.910
6.906
6.707
6.608
6.591
5.803
5.793
5.671
5.659
5.053
5.043
5.035
5.023
3.353
3.022
2.890
2.510
2.505
2.500
2.496
2.491
0.37
0.63
5.10 5.08 5.06 5.04 5.02 5.00 4.98
f1 (ppm)
OH
OH
Ph
N-Me
O
44
1H NMR (400 MHz, DMSO-d6)
dr = 1.7:1
H2O
0.37
0.37
0.63
2.16
3.15
1.08
0.72
0.37
1.24
0.37
0.63
0.37
0.63
1.89
1.11
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

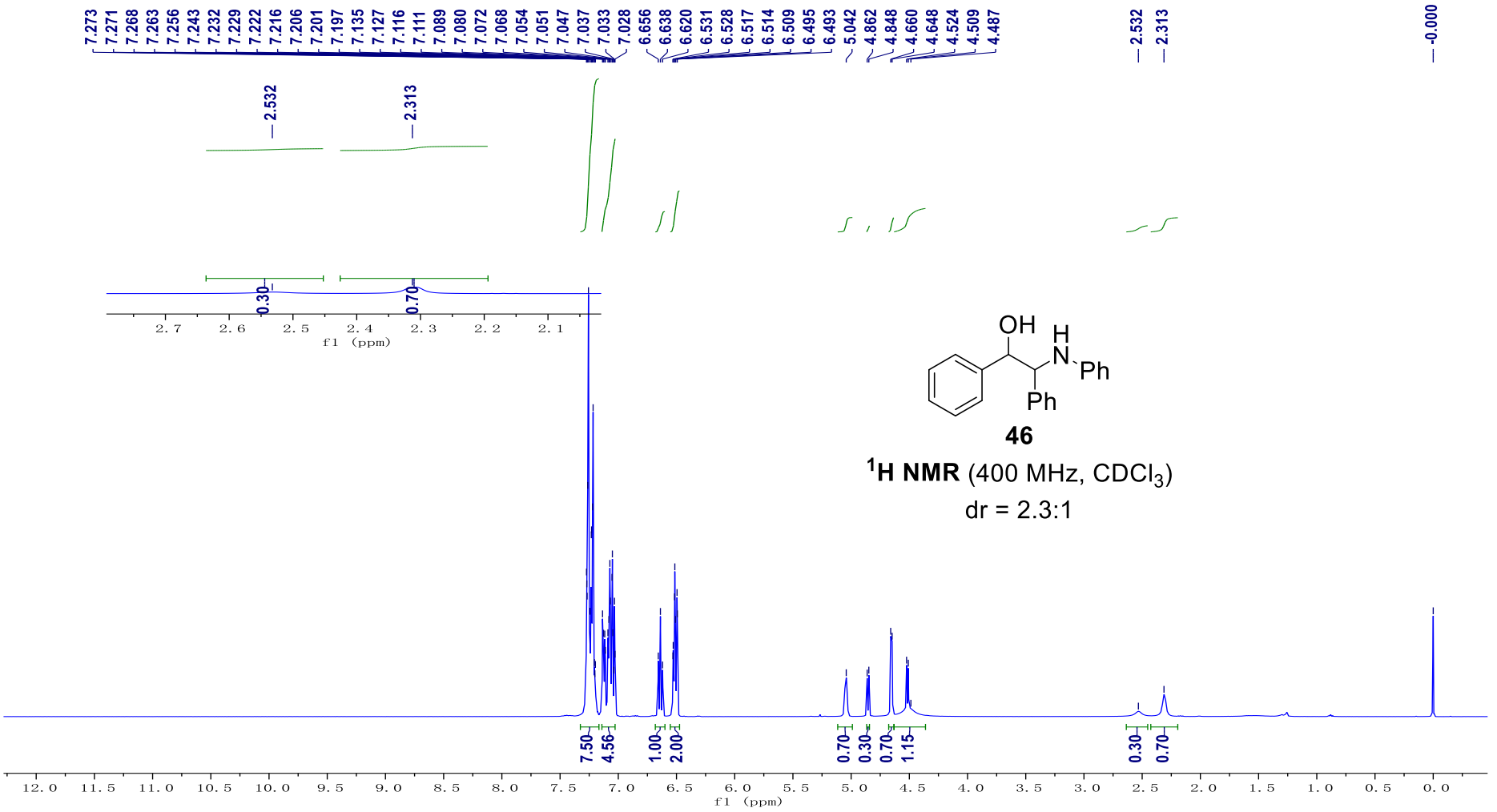

166.401
166.189
145.695
145.022
140.462
139.719
132.905
132.117
130.953
130.320
128.910
128.852
127.858
127.170
127.083
126.969
126.955
126.893
124.627
123.722
121.638
121.479
91.408
91.006
75.326
73.390
40.146
39.937
39.729
39.520
39.311
39.103
38.894
24.602
24.179
f1 (ppm)
OH
OH
Ph
N
Me
O
44
13C NMR (100 MHz, DMSO-d6)


8.651
8.616
7.720
7.701
7.568
7.550
7.532
7.466
7.449
7.430
7.404
7.386
7.371
7.358
7.348
7.331
7.207
7.193
7.187
7.168
7.138
7.130
7.123
7.107
7.078
7.074
6.504
6.432
5.685
5.675
5.626
5.615
4.997
4.988
4.937
4.928
3.357
2.509
2.505
2.500
2.495
2.490
0.60
0.40
f1 (ppm)
OH
OH
Ph
NH
O
45
1H NMR (400 MHz, DMSO-d6)
dr = 1.5:1
H2O
0.40
0.60
0.60
0.60
0.40
2.20
5.20
0.60
0.40
0.60
0.40
0.40
0.60
f1 (ppm)



45

^{13}C NMR (100 MHz, DMSO-d_6)

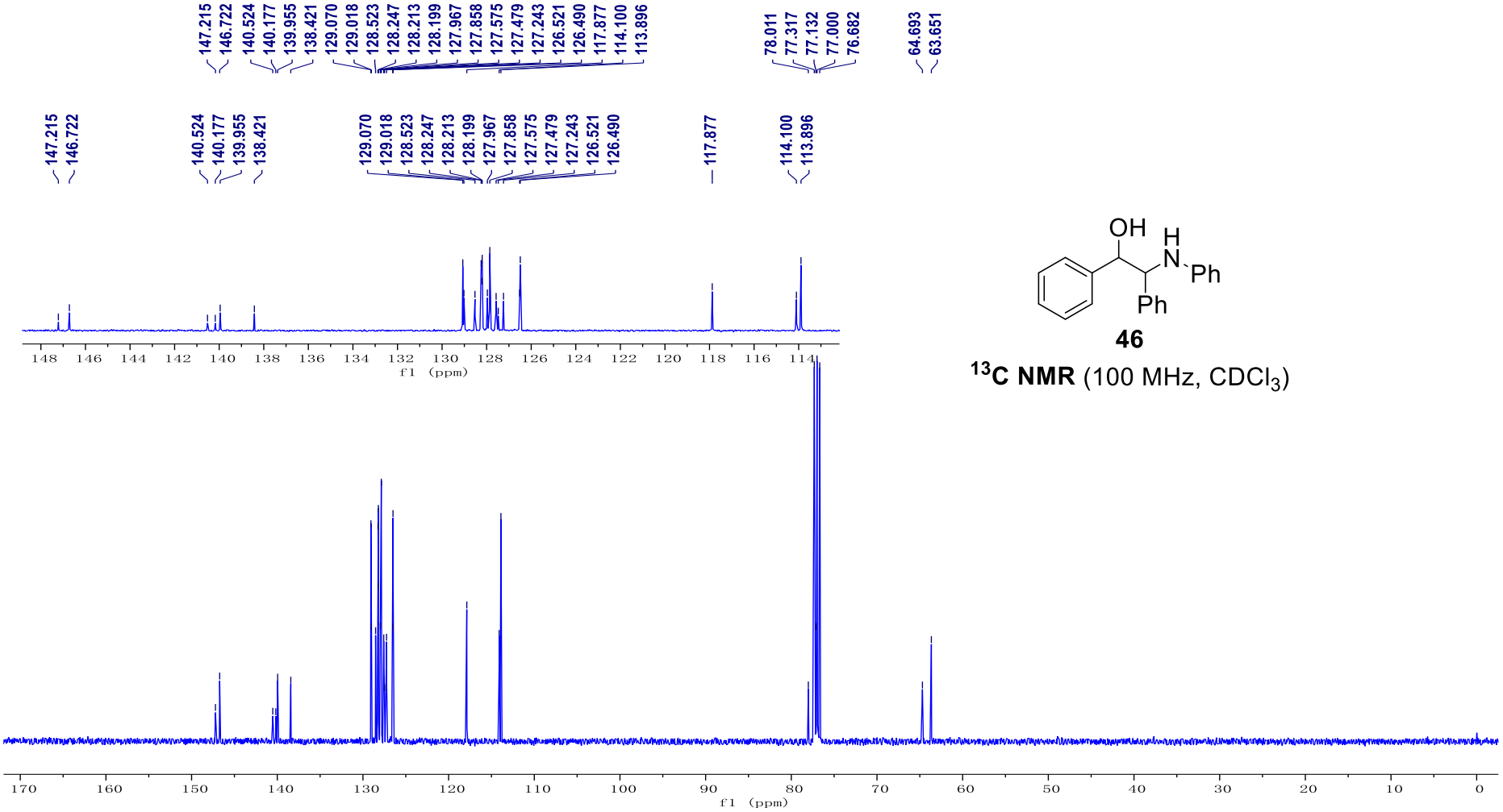


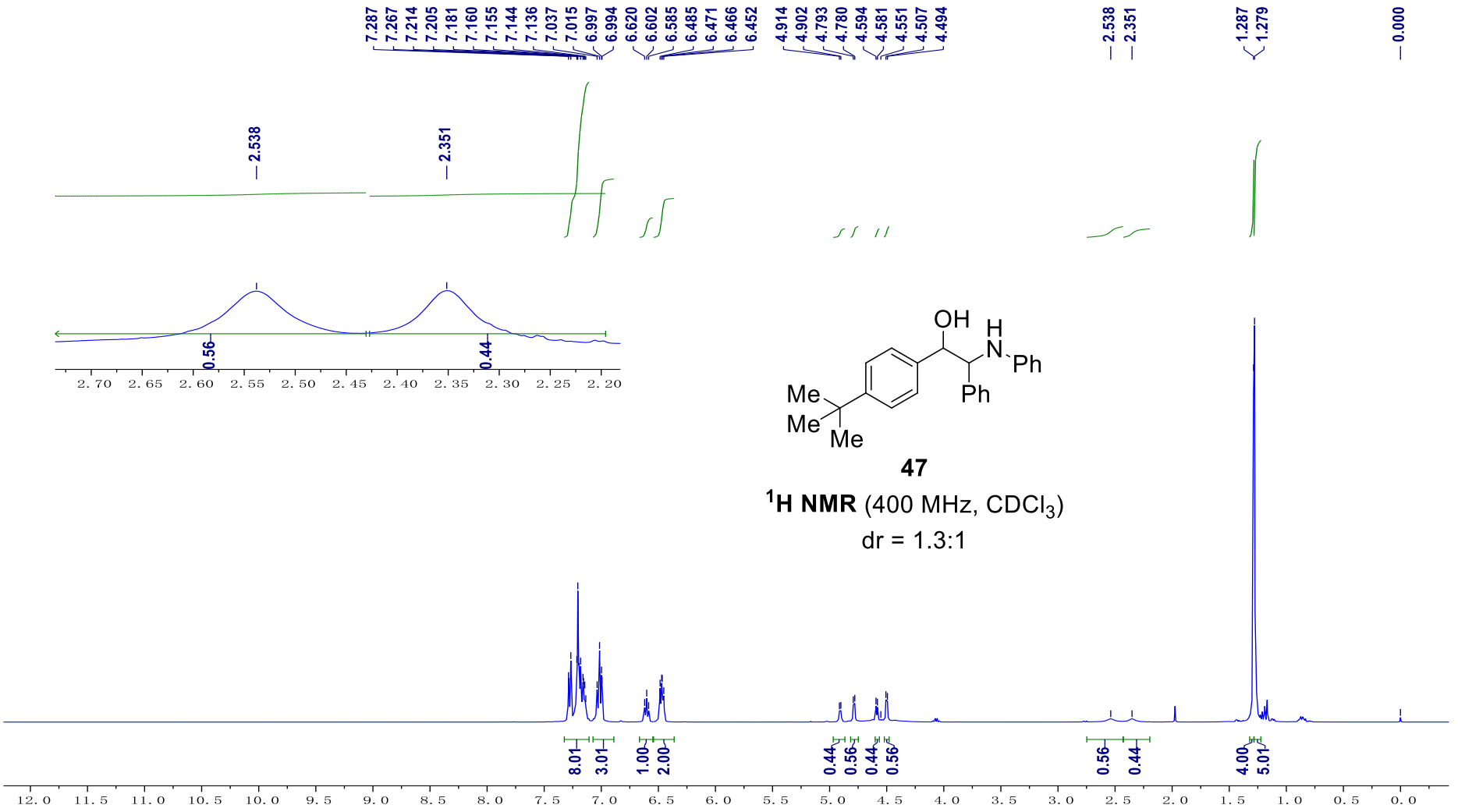
OH
H
N
Ph
Ph

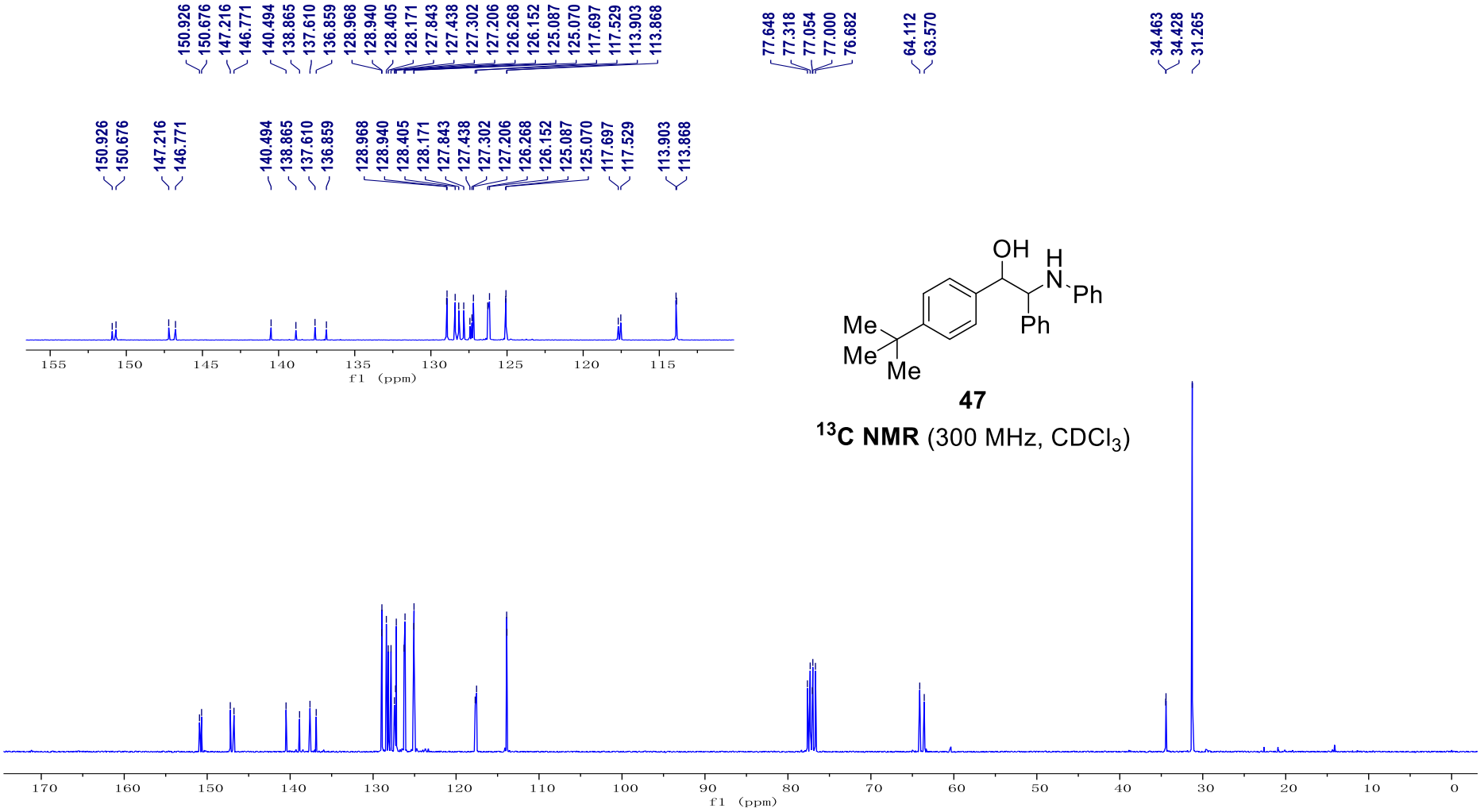
46

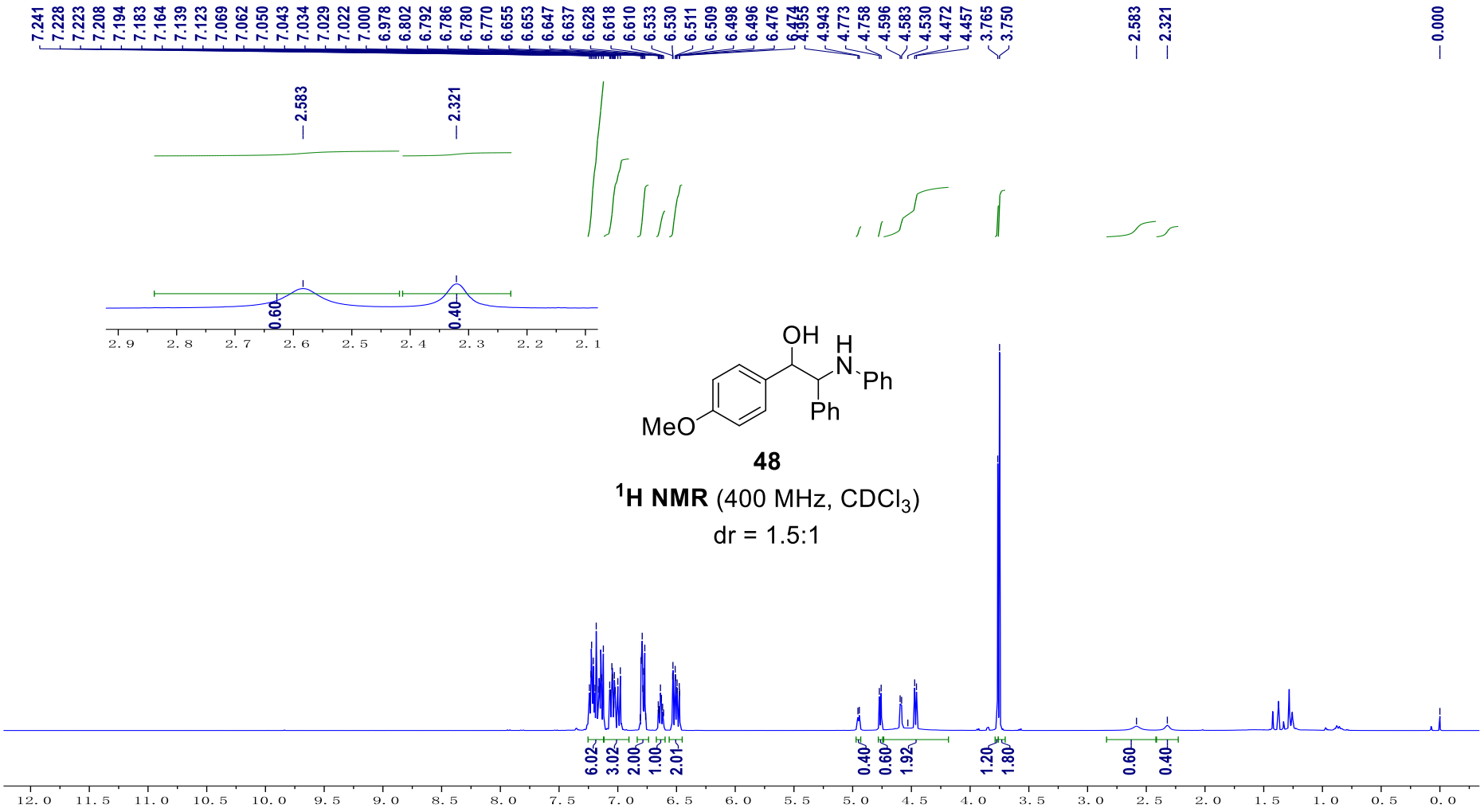
^{1}H NMR (400 MHz, $CDCl_3$)

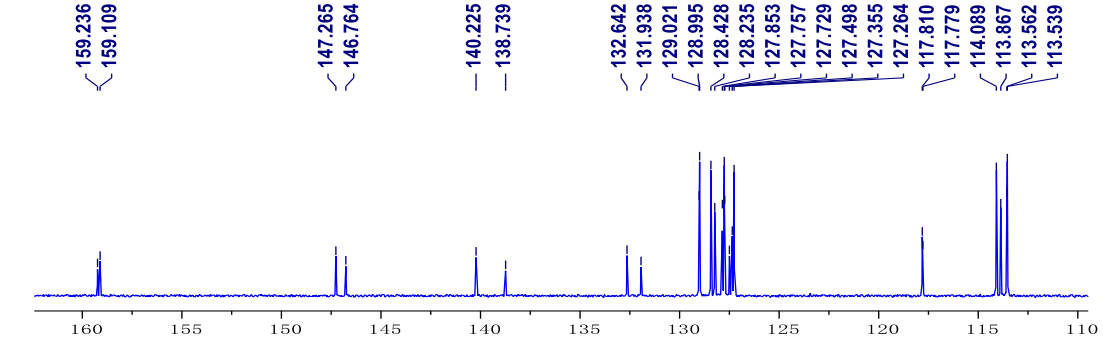
dr = 2.3:1

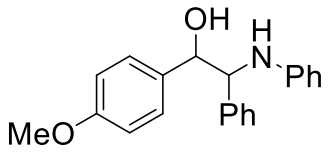

147.215
146.722
140.524
140.177
139.955
138.421
129.070
129.018
128.523
128.247
128.213
128.199
127.967
127.858
127.575
127.479
127.243
126.521
126.490
117.877
114.100
113.896
78.011
77.317
77.132
77.000
76.682
64.693
63.651
f1 (ppm)
OH
H
N
Ph
Ph
46
13C NMR (100 MHz, CDCl3)


7.287
7.267
7.214
7.205
7.181
7.160
7.155
7.144
7.136
7.037
7.015
6.997
6.994
6.620
6.602
6.585
6.485
6.471
6.466
6.452
4.914
4.902
4.793
4.780
4.594
4.581
4.551
4.507
4.494
2.538
2.351
1.287
1.279
0.000
0.56
0.44
OH
H
N
Ph
Ph
Me
Me
Me
47
1H NMR (400 MHz, CDCl3)
dr = 1.3:1
8.01
3.01
1.00
2.00
0.44
0.56
0.44
0.56
0.56
0.44
4.00
5.01


150.926
150.676
147.216
146.771
140.494
138.865
137.610
136.859
128.968
128.940
128.405
128.171
127.843
127.438
127.302
127.206
126.268
126.152
125.087
125.070
117.697
117.529
113.903
113.868
77.648
77.318
77.054
77.000
76.682
64.112
63.570
34.463
34.428
31.265
f1 (ppm)
OH
H
N
Ph
Ph
Me
Me
Me
47
13C NMR (300 MHz, CDCl3)

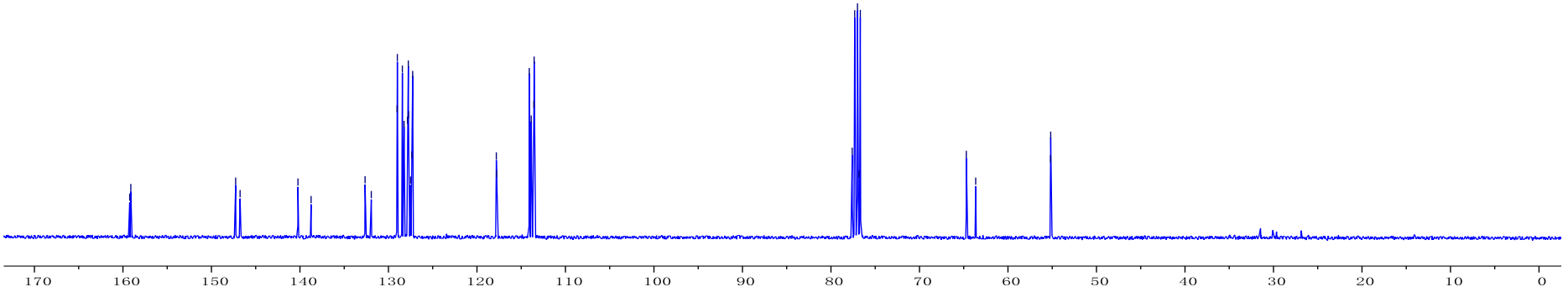

7.241
7.228
7.223
7.208
7.194
7.183
7.164
7.139
7.123
7.069
7.062
7.050
7.043
7.034
7.029
7.022
7.000
6.978
6.802
6.792
6.786
6.780
6.770
6.655
6.653
6.647
6.637
6.628
6.618
6.610
6.533
6.530
6.511
6.509
6.498
6.496
6.476
4.943
4.773
4.758
4.596
4.583
4.530
4.472
4.457
3.765
3.750
2.583
2.321
0.000
0.60
0.40
OH
H
N
Ph
Ph
MeO
48
1H NMR (400 MHz, CDCl3)
dr = 1.5:1
6.02
3.02
2.00
1.00
2.01
0.40
0.60
1.92
1.20
1.80
0.60
0.40

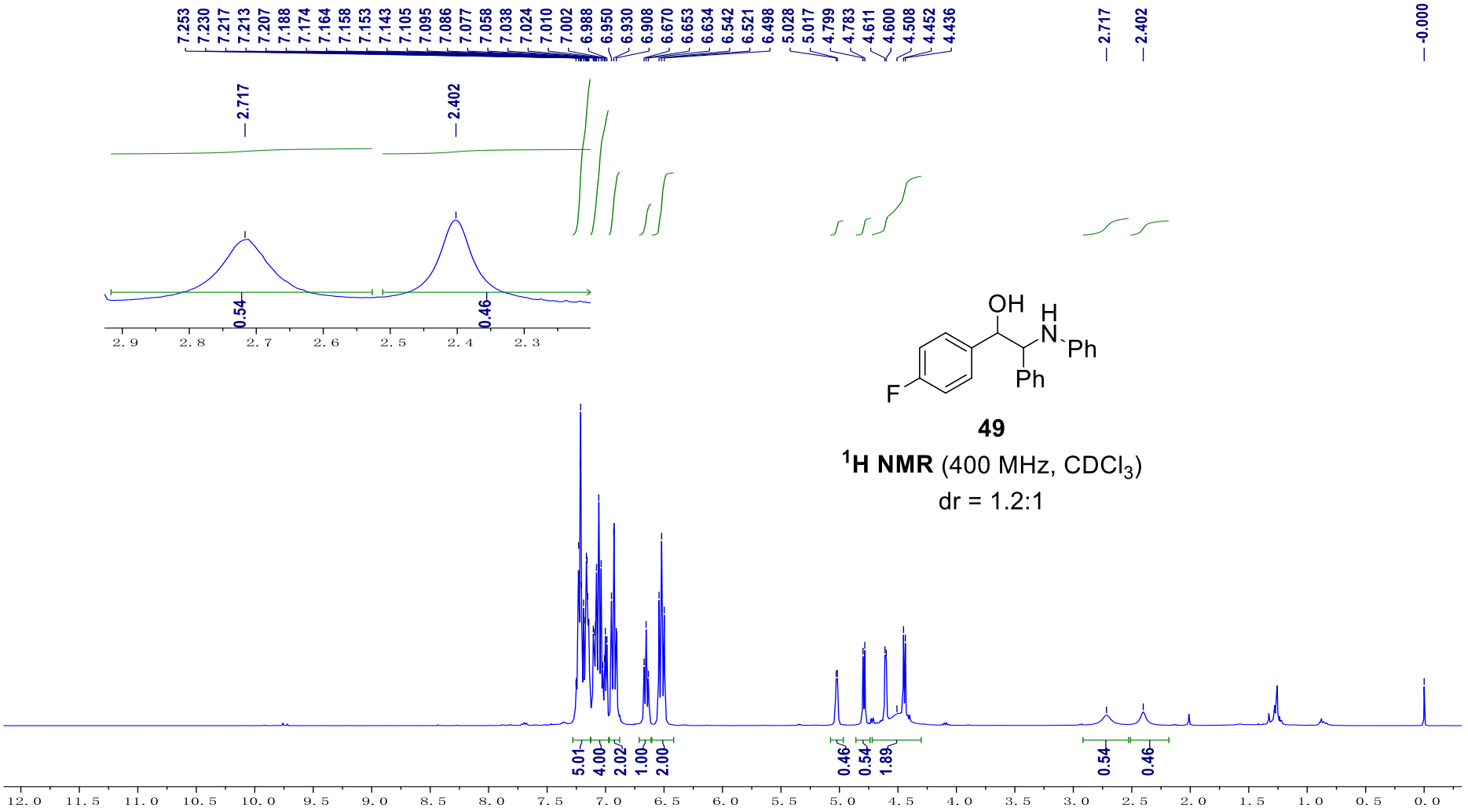


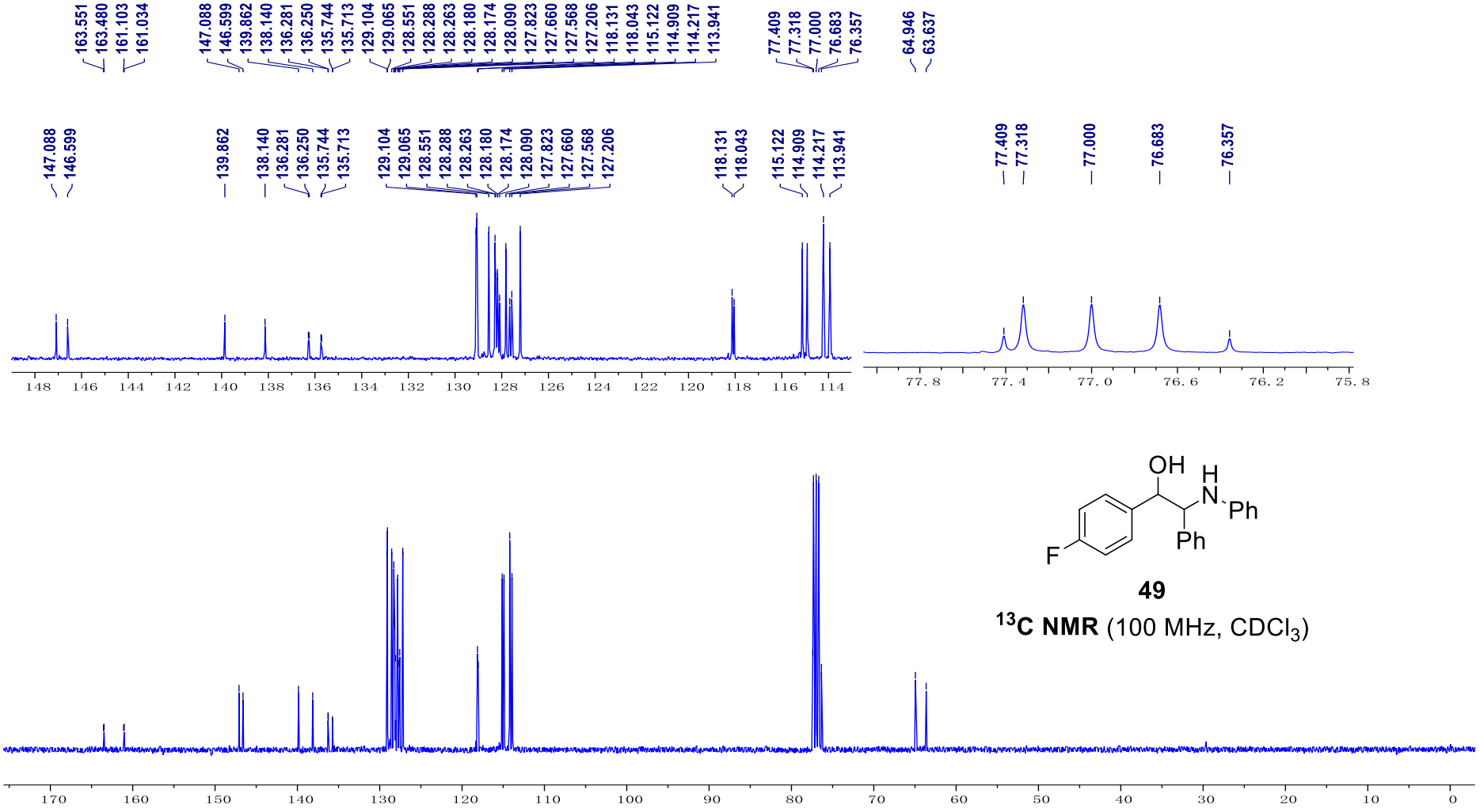


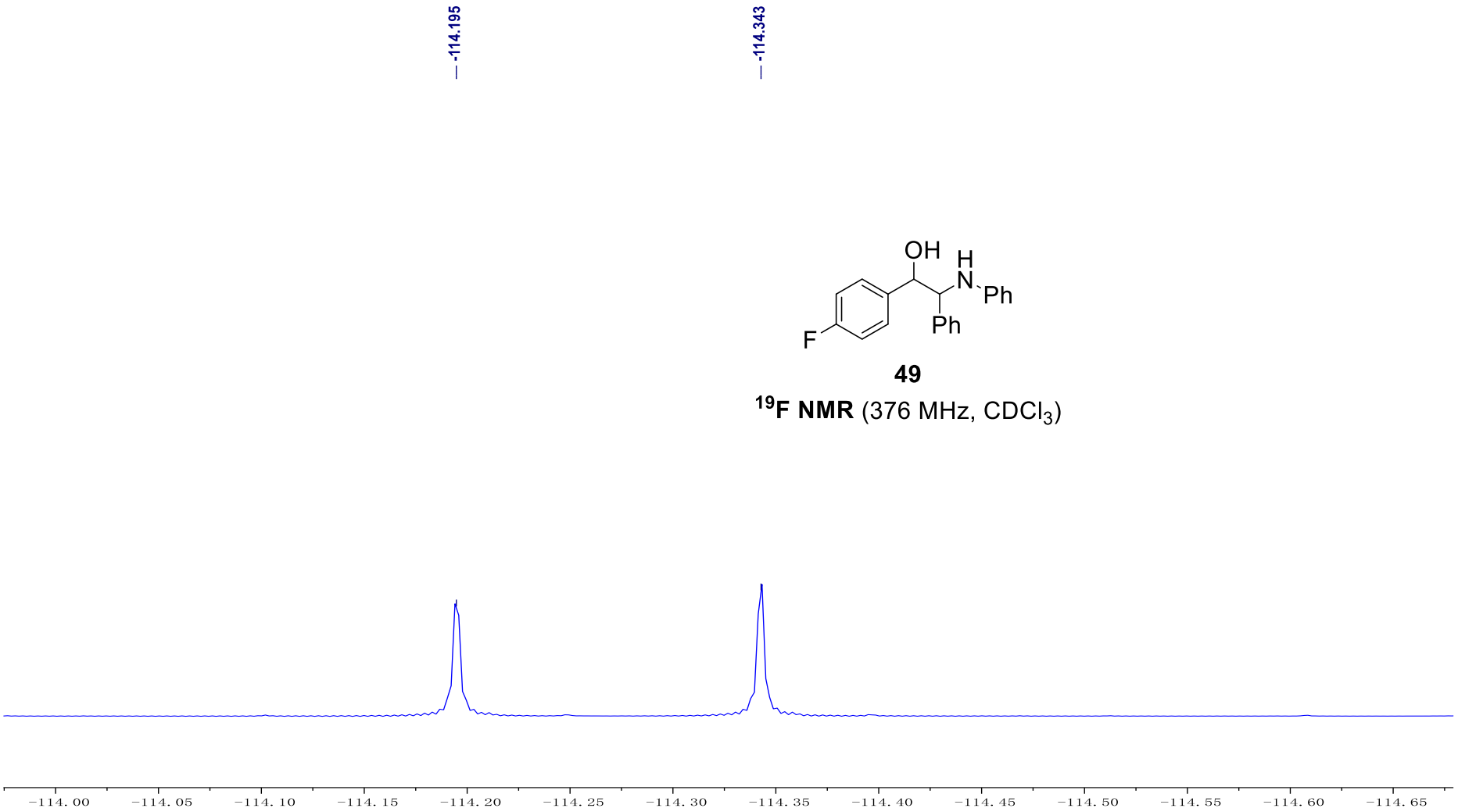
48

^{13}C NMR (100 MHz, $CDCl_3$)



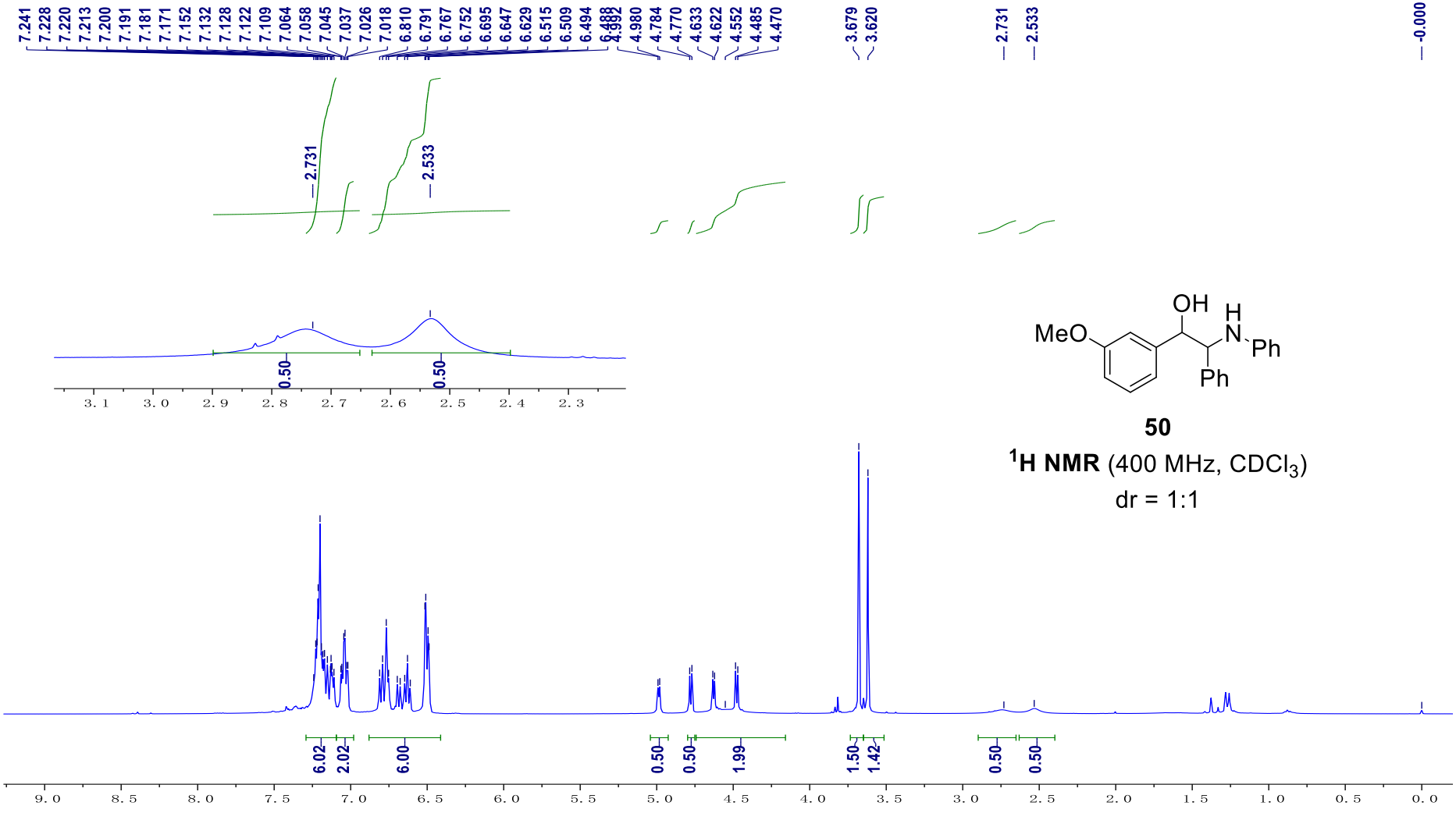

7.253
7.230
7.217
7.213
7.207
7.188
7.174
7.164
7.158
7.153
7.143
7.105
7.095
7.086
7.077
7.058
7.038
7.024
7.010
7.002
6.988
6.950
6.930
6.908
6.670
6.653
6.634
6.542
6.521
6.498
5.028
5.017
4.799
4.783
4.611
4.600
4.508
4.452
4.436
2.717
2.402
-0.000
0.54
0.46
OH
H
N
Ph
Ph
F
49
1H NMR (400 MHz, CDCl3)
dr = 1.2:1
5.01
4.00
2.02
1.00
2.00
0.46
0.54
1.89
0.54
0.46

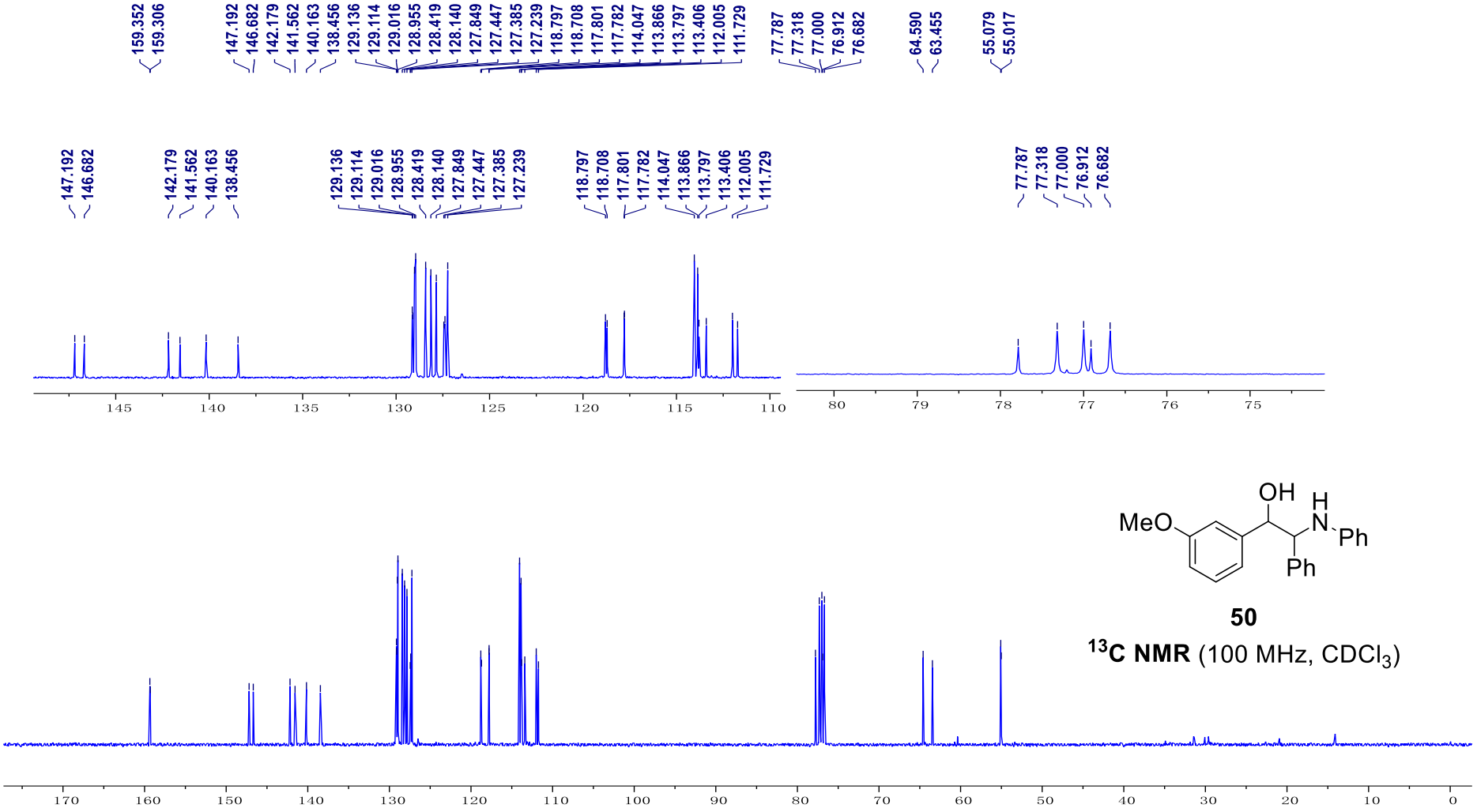

163.551
163.480
161.103
161.034
147.088
146.599
139.862
138.140
136.281
136.250
135.744
135.713
129.104
129.065
128.551
128.288
128.263
128.180
128.174
128.090
127.823
127.660
127.568
127.206
118.131
118.043
115.122
114.909
114.217
113.941
77.409
77.318
77.000
76.683
76.357
64.946
63.637
OH
H
N
Ph
Ph
F
49
13C NMR (100 MHz, CDCl3)

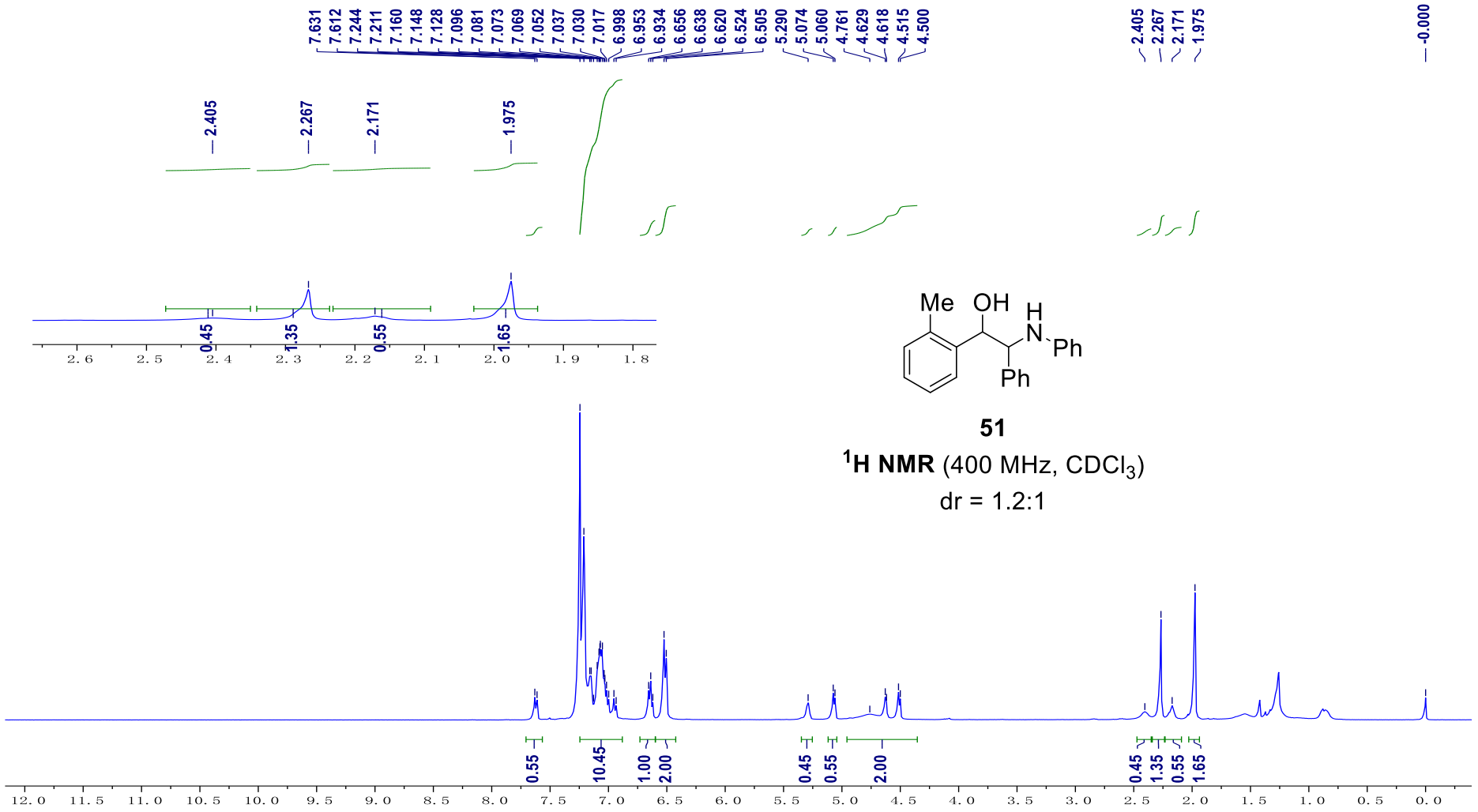


49

^{19}F NMR (376 MHz, $CDCl_3$)


7.241
7.228
7.220
7.213
7.200
7.191
7.181
7.171
7.152
7.132
7.128
7.122
7.109
7.064
7.058
7.045
7.037
7.026
7.018
6.810
6.791
6.767
6.752
6.695
6.647
6.629
6.515
6.509
6.494
4.980
4.784
4.770
4.633
4.622
4.552
4.485
4.470
3.679
3.620
2.731
2.533
-0.000
2.731
2.533
0.50
0.50
3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3
OH
H
N
MeO
Ph
Ph
50
1H NMR (400 MHz, CDCl3)
dr = 1:1
6.02
2.02
6.00
0.50
0.50
1.99
1.50
1.42
0.50
0.50
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

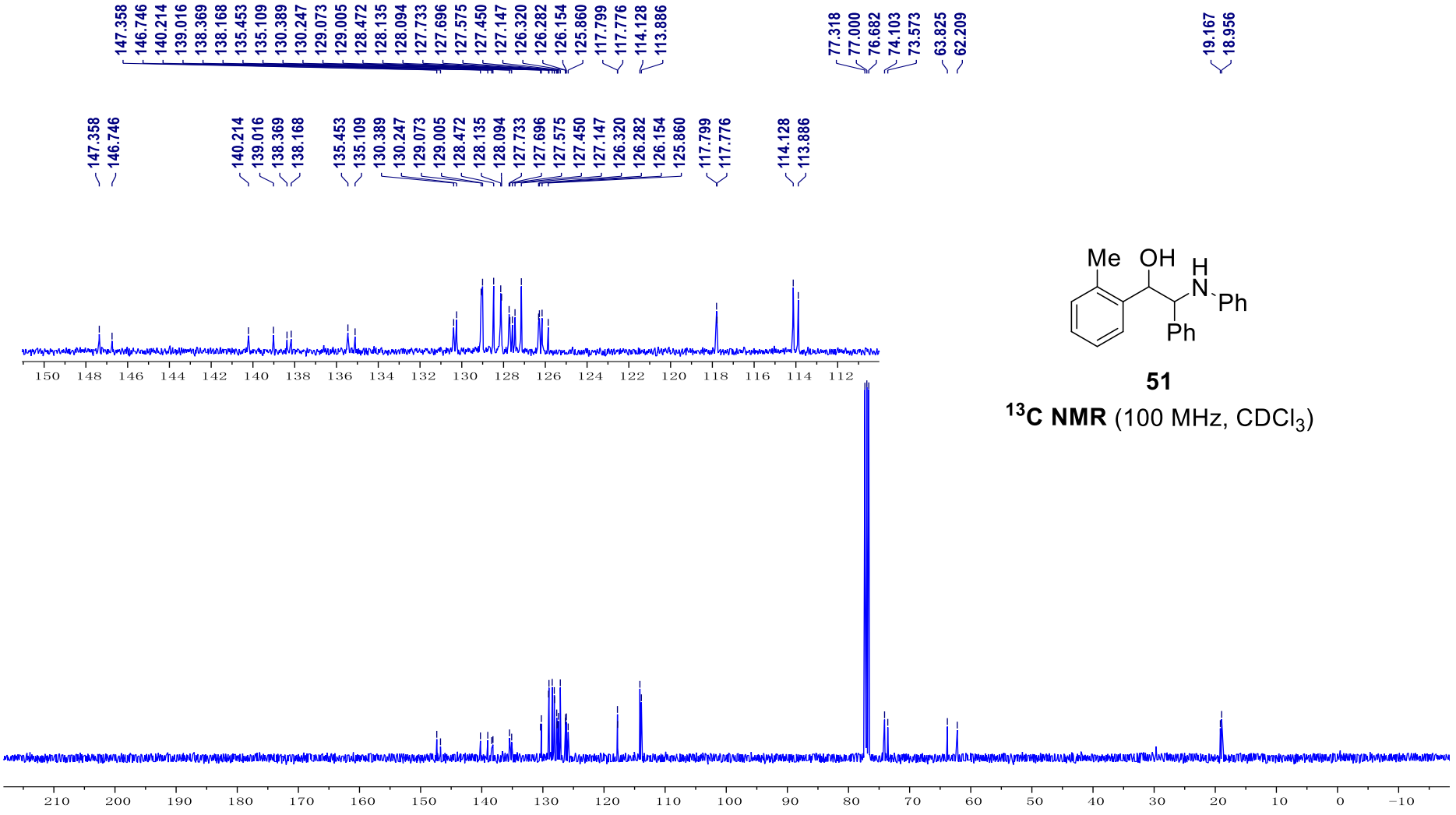

159.352
159.306
147.192
146.682
142.179
141.562
140.163
138.456
129.136
129.114
129.016
128.955
128.419
128.140
127.849
127.447
127.385
127.239
118.797
118.708
117.801
117.782
114.047
113.866
113.797
113.406
112.005
111.729
77.787
77.318
77.000
76.912
76.682
64.590
63.455
55.079
55.017
MeO
OH
H
N
Ph
Ph
50
13C NMR (100 MHz, CDCl3)



51

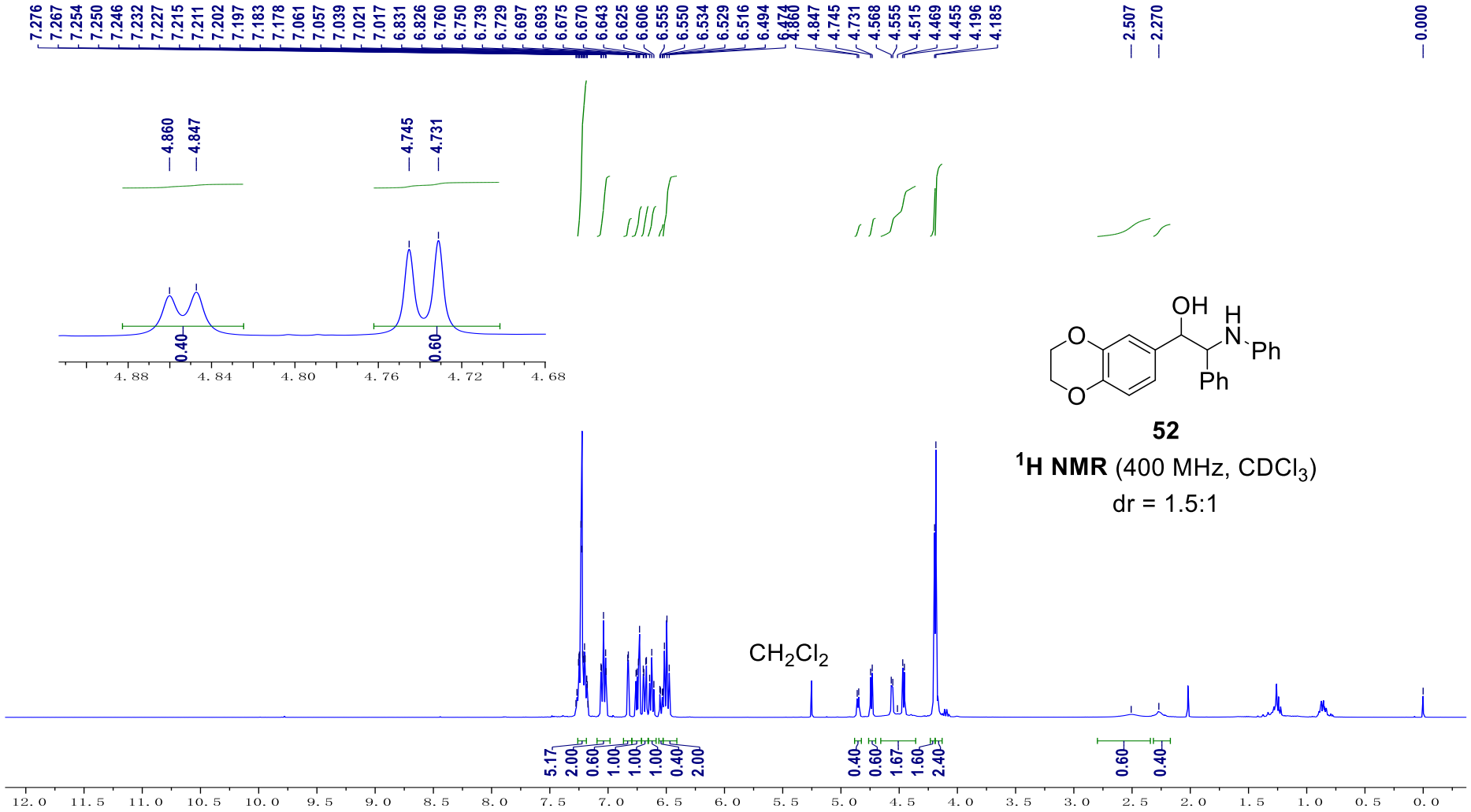
^{1}H NMR (400 MHz, $CDCl_3$)

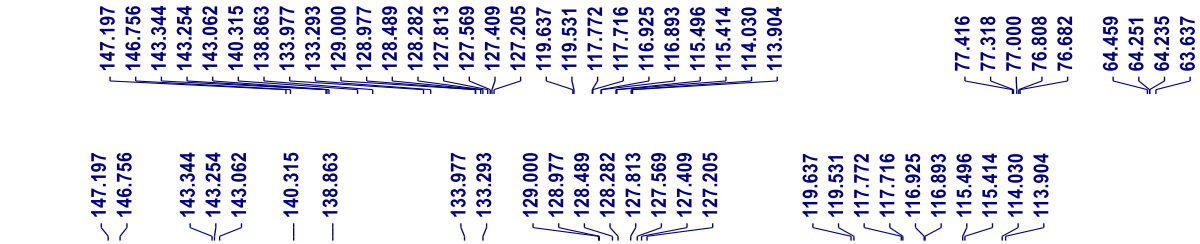
dr = 1.2:1

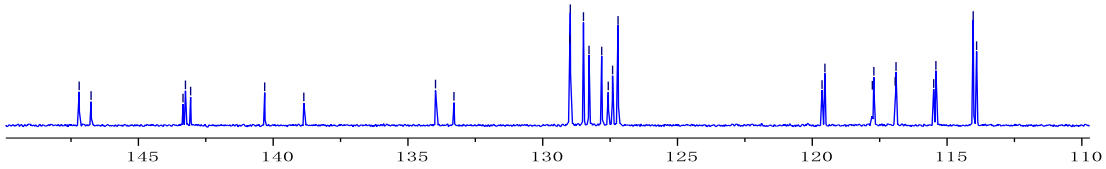


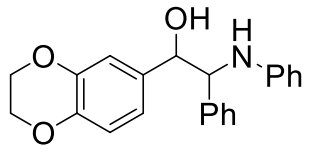
51

^{13}C NMR (100 MHz, $CDCl_3$)


7.276
7.267
7.254
7.250
7.246
7.232
7.227
7.215
7.211
7.202
7.197
7.183
7.178
7.061
7.057
7.039
7.021
7.017
6.831
6.826
6.760
6.750
6.739
6.729
6.697
6.693
6.675
6.670
6.643
6.625
6.606
6.555
6.550
6.534
6.529
6.516
6.494
6.474
6.460
4.847
4.745
4.731
4.568
4.555
4.515
4.469
4.455
4.196
4.185
2.507
2.270
0.000
4.860
4.847
4.745
4.731
0.40
0.60
4.88
4.84
4.80
4.76
4.72
4.68
OH
H
N
Ph
Ph
O
O
52
1H NMR (400 MHz, CDCl3)
dr = 1.5:1
CH2Cl2
5.17
2.00
0.60
1.00
1.00
1.00
0.40
2.00
0.40
0.60
1.67
1.60
2.40
0.60
0.40
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

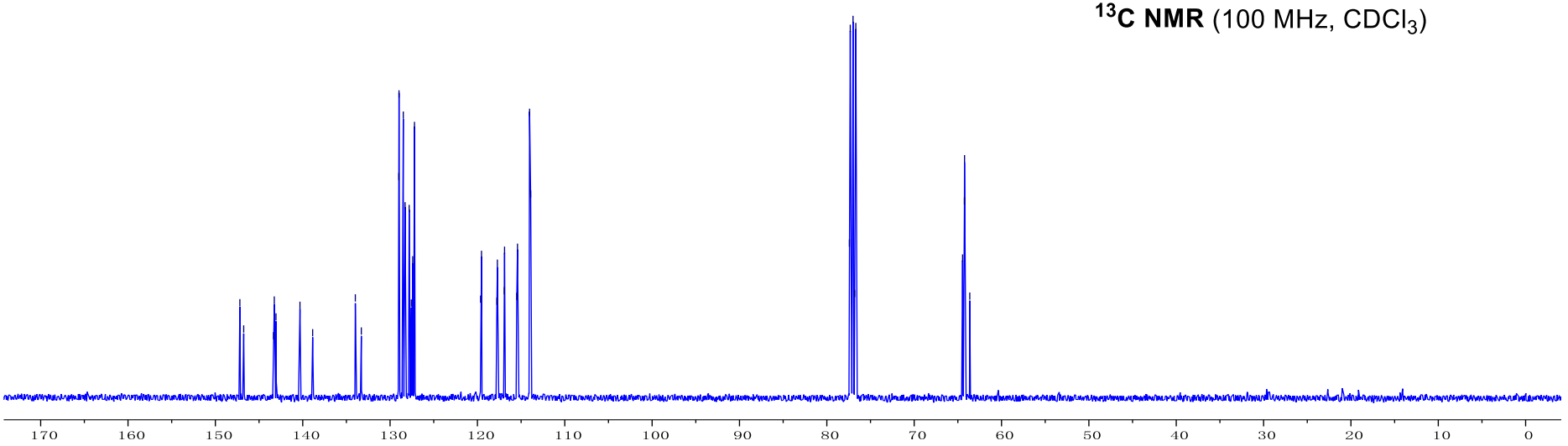


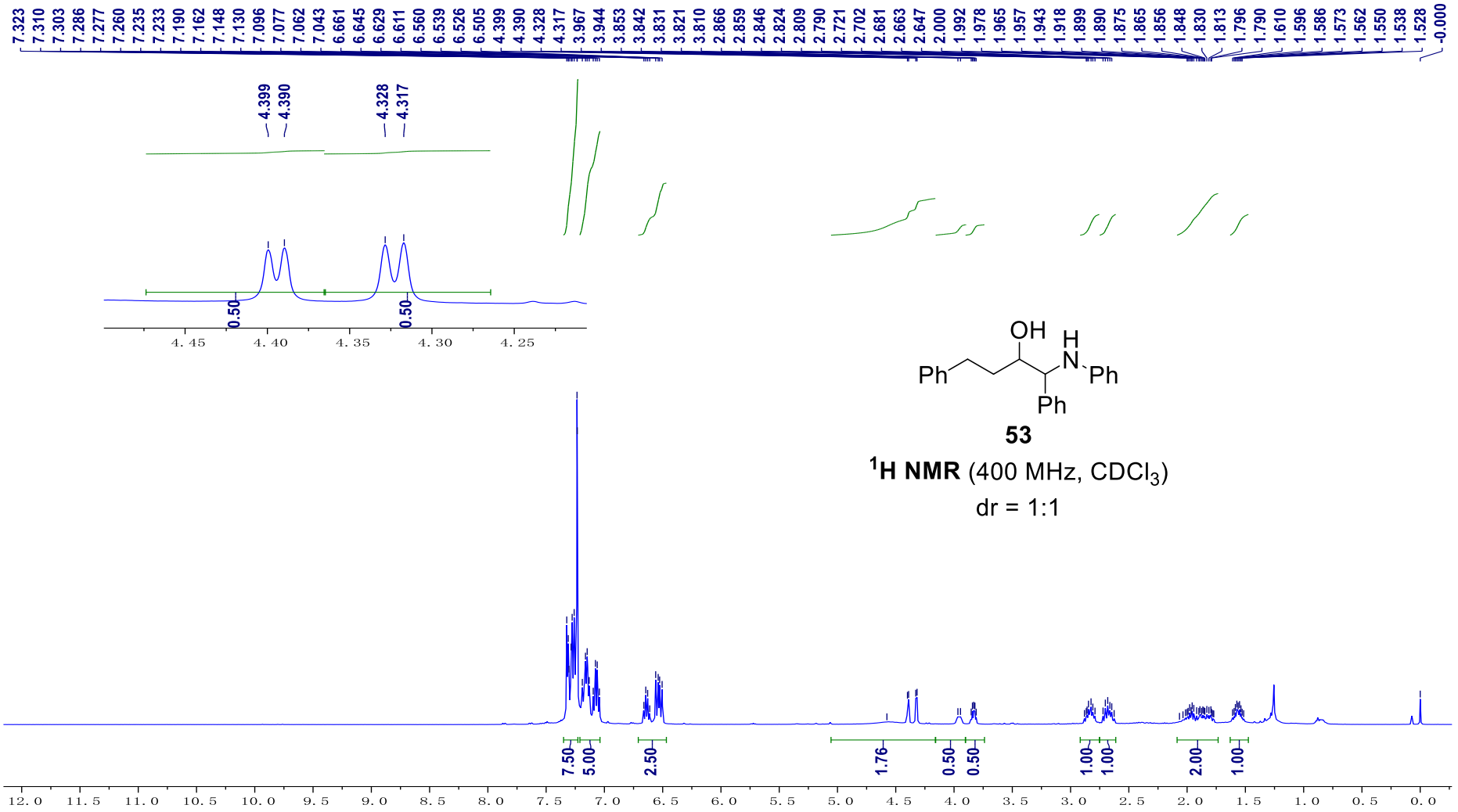


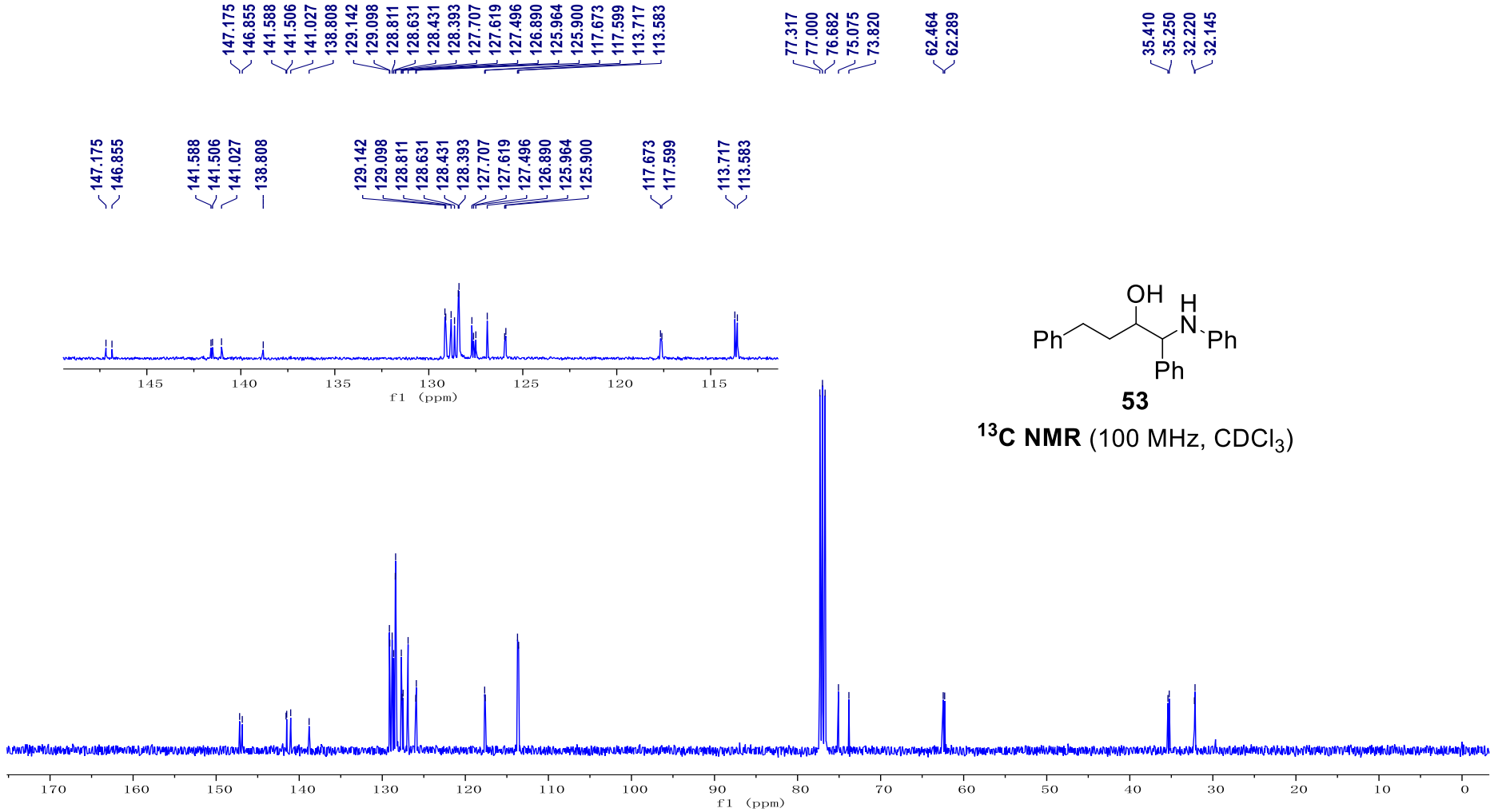


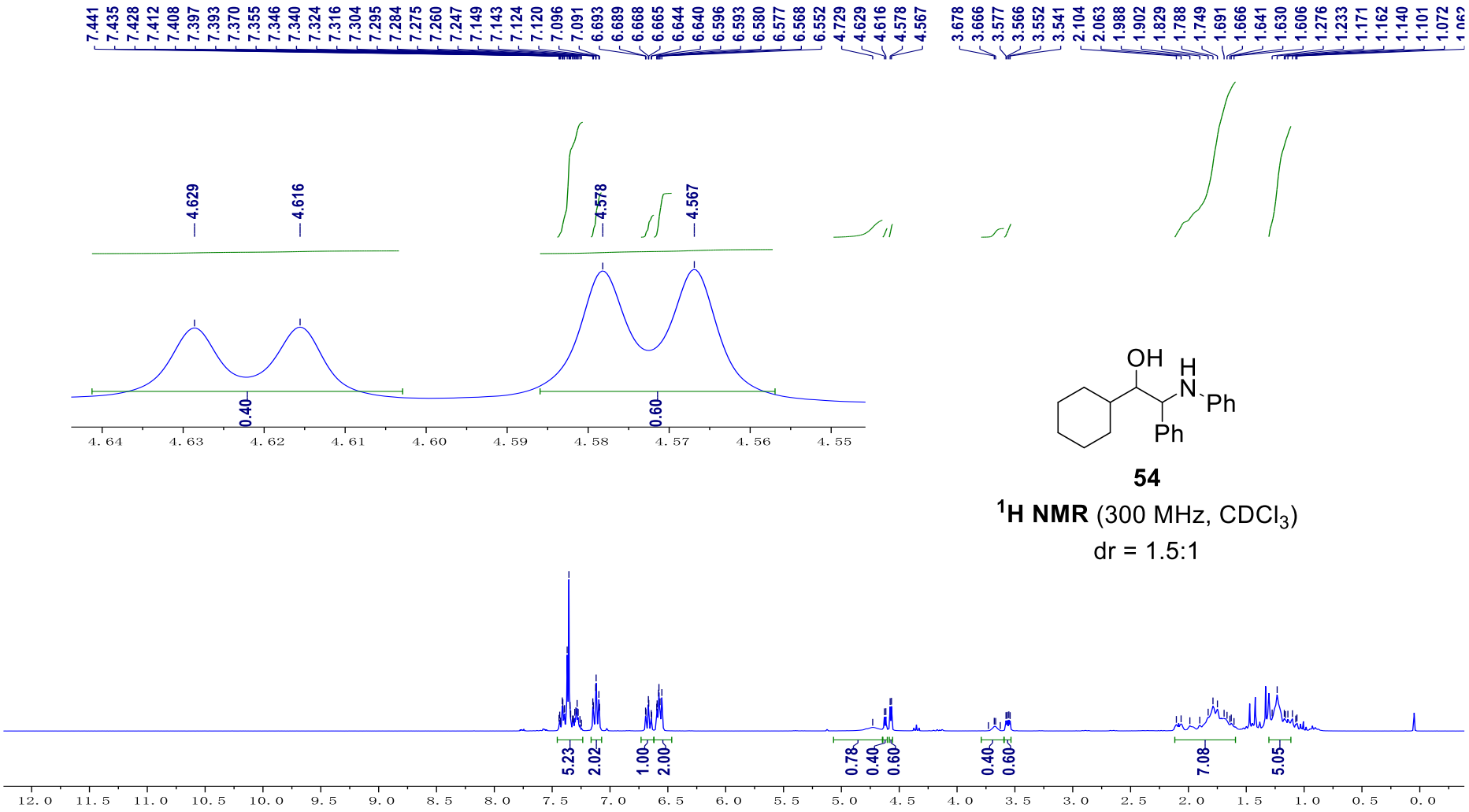
52

^{13}C NMR (100 MHz, $CDCl_3$)




7.323 7.310 7.303 7.286 7.277 7.260 7.235 7.233 7.190 7.162 7.148 7.130 7.096 7.077 7.062 7.043 6.661 6.645 6.629 6.611 6.560 6.539 6.526 6.505 4.399 4.390 4.328 4.317 3.967 3.944 3.853 3.842 3.831 3.821 3.810 2.866 2.859 2.846 2.824 2.809 2.790 2.721 2.702 2.681 2.663 2.647 2.000 1.992 1.978 1.965 1.957 1.943 1.918 1.899 1.890 1.875 1.865 1.856 1.848 1.830 1.813 1.796 1.790 1.610 1.596 1.586 1.573 1.562 1.550 1.538 1.528 -0.000
4.399
4.390
4.328
4.317
0.50
0.50
4.45 4.40 4.35 4.30 4.25
OH
H
N
Ph
Ph
Ph
53
1H NMR (400 MHz, CDCl3)
dr = 1:1
7.50
5.00
2.50
1.76
0.50
0.50
1.00
1.00
2.00
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

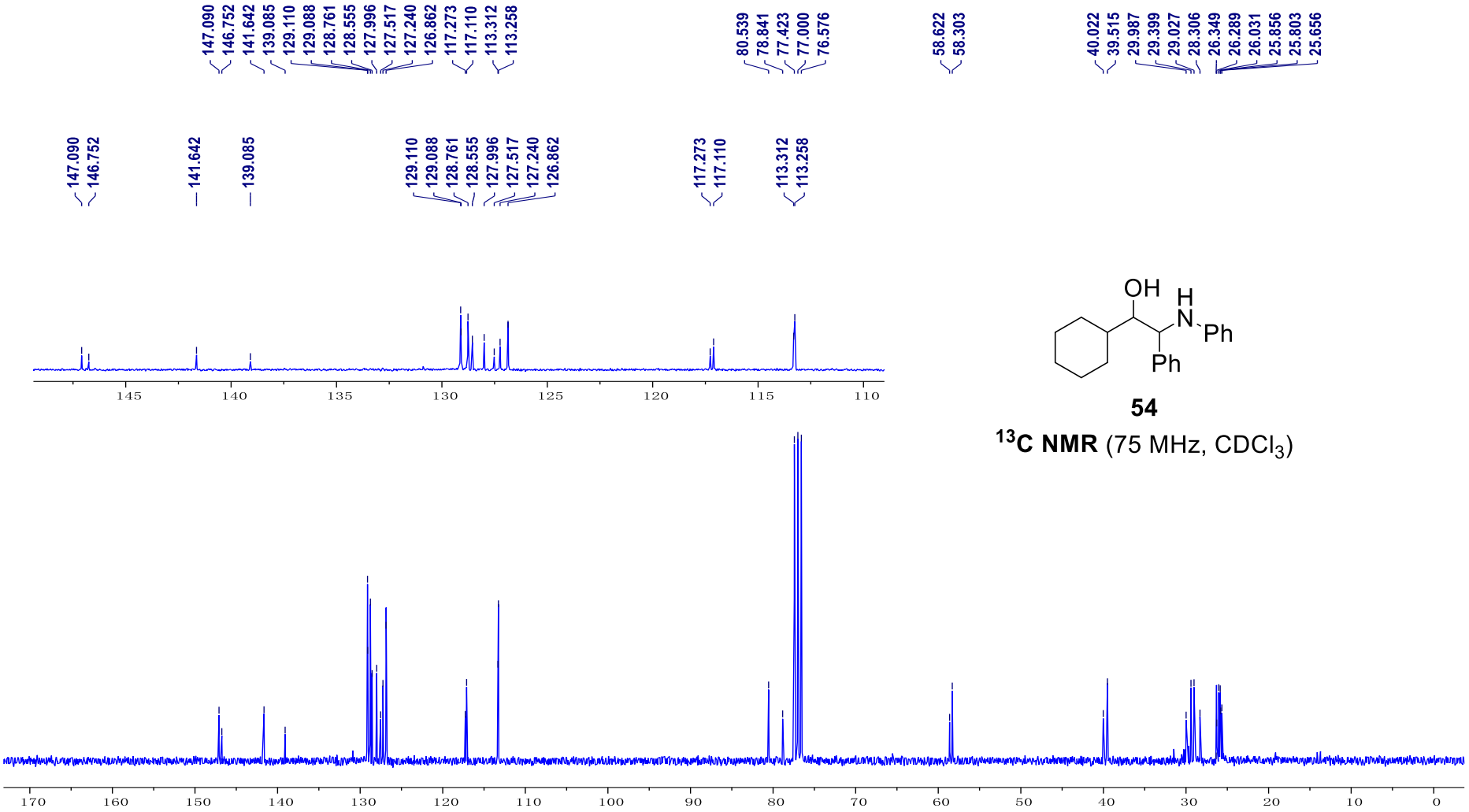

147.175
146.855
141.588
141.506
141.027
138.808
129.142
129.098
128.811
128.631
128.431
128.393
127.707
127.619
127.496
126.890
125.964
125.900
117.673
117.599
113.717
113.583
77.317
77.000
76.682
75.075
73.820
62.464
62.289
35.410
35.250
32.220
32.145
f1 (ppm)
OH
H
N
Ph
Ph
Ph
53
13C NMR (100 MHz, CDCl3)

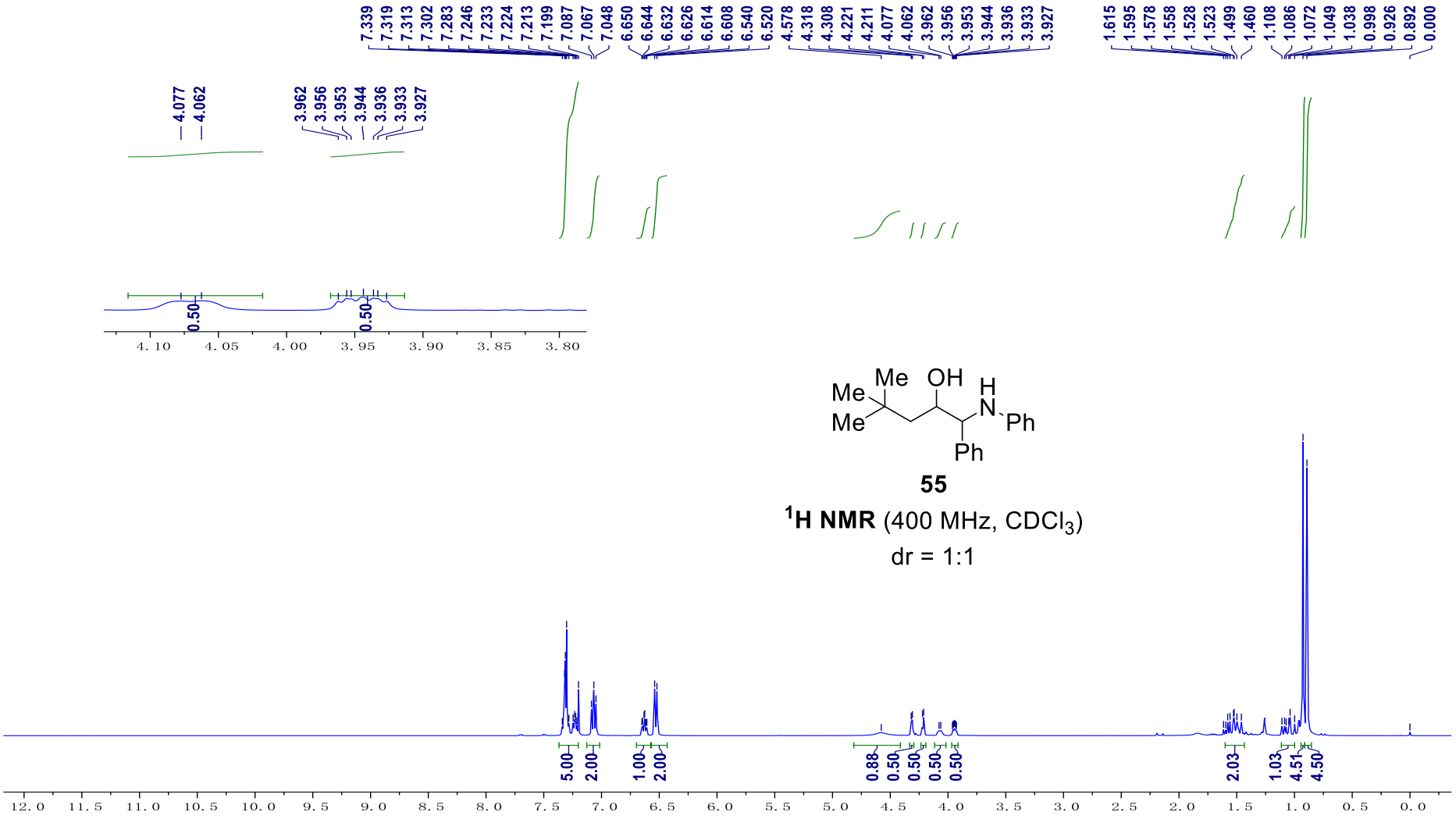


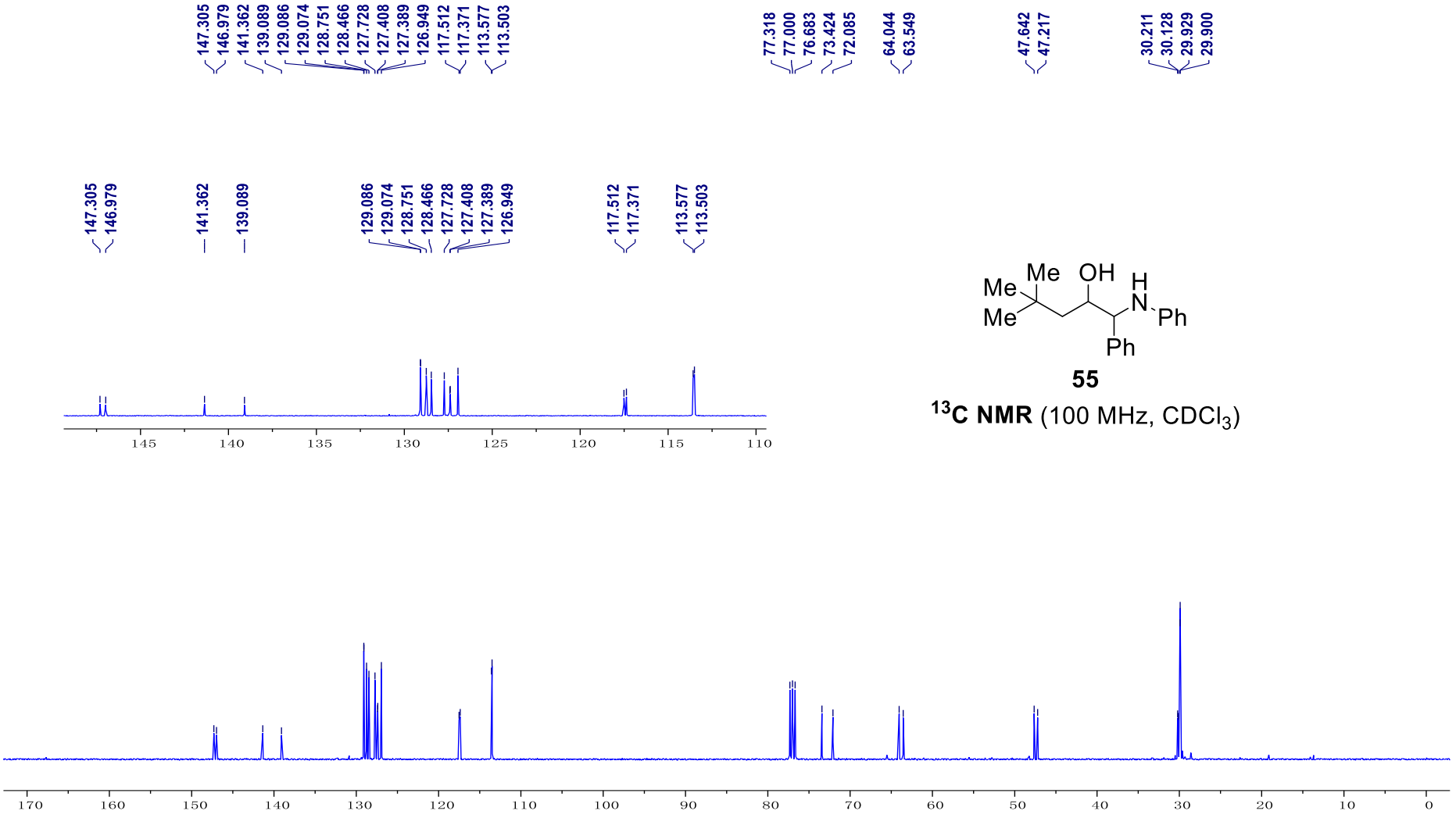
54

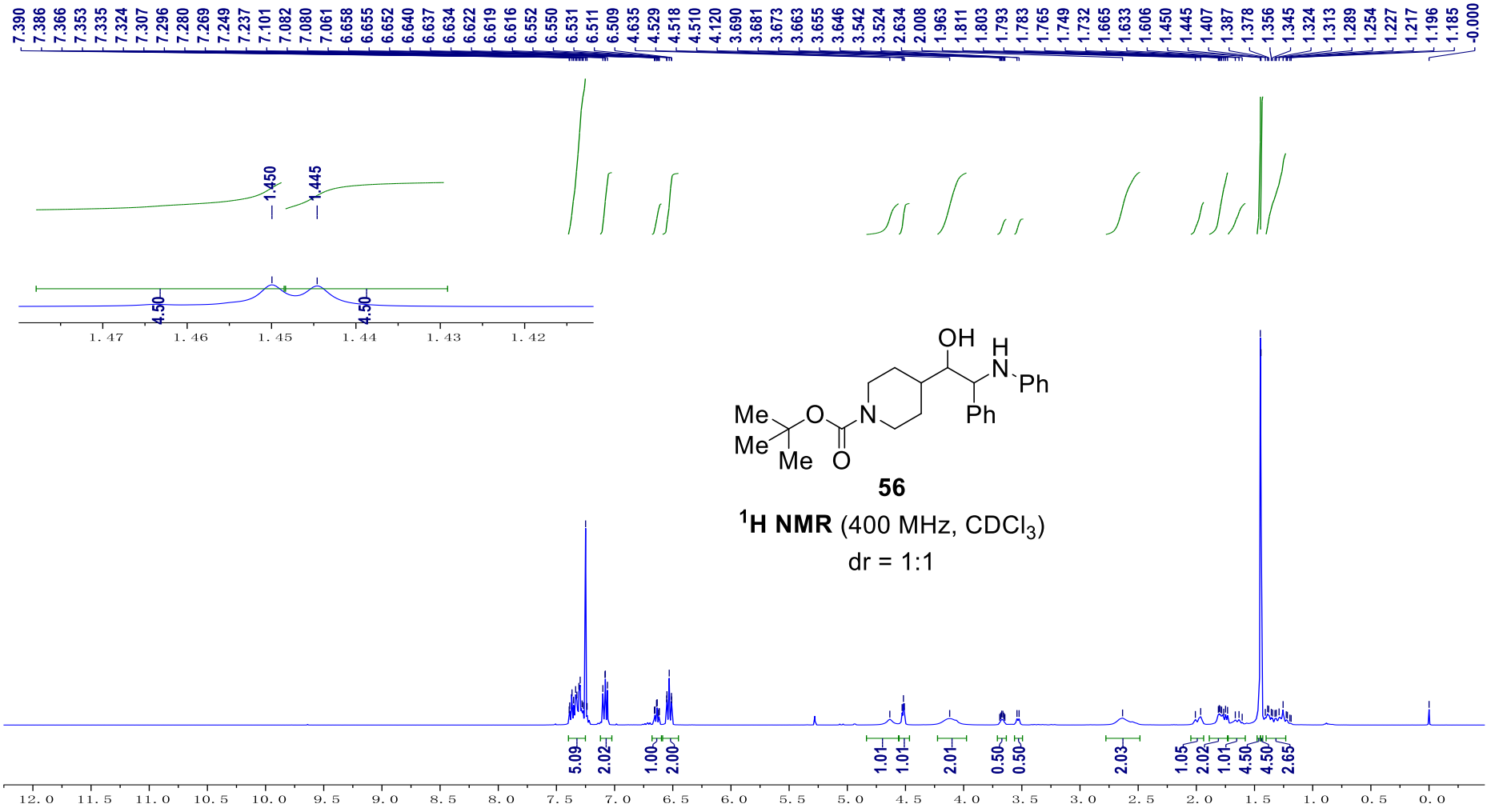
^{1}H NMR (300 MHz, $CDCl_3$)

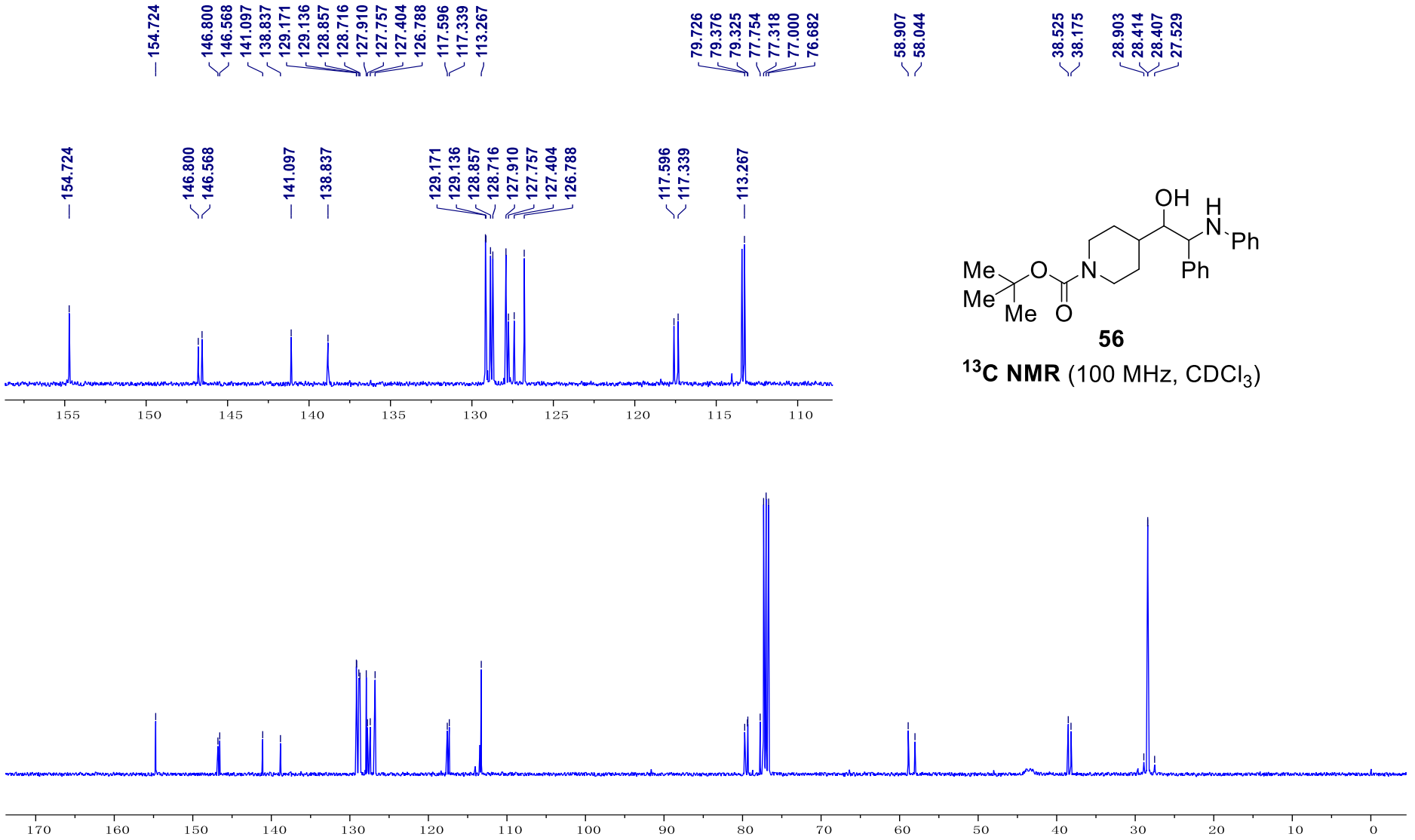
dr = 1.5:1

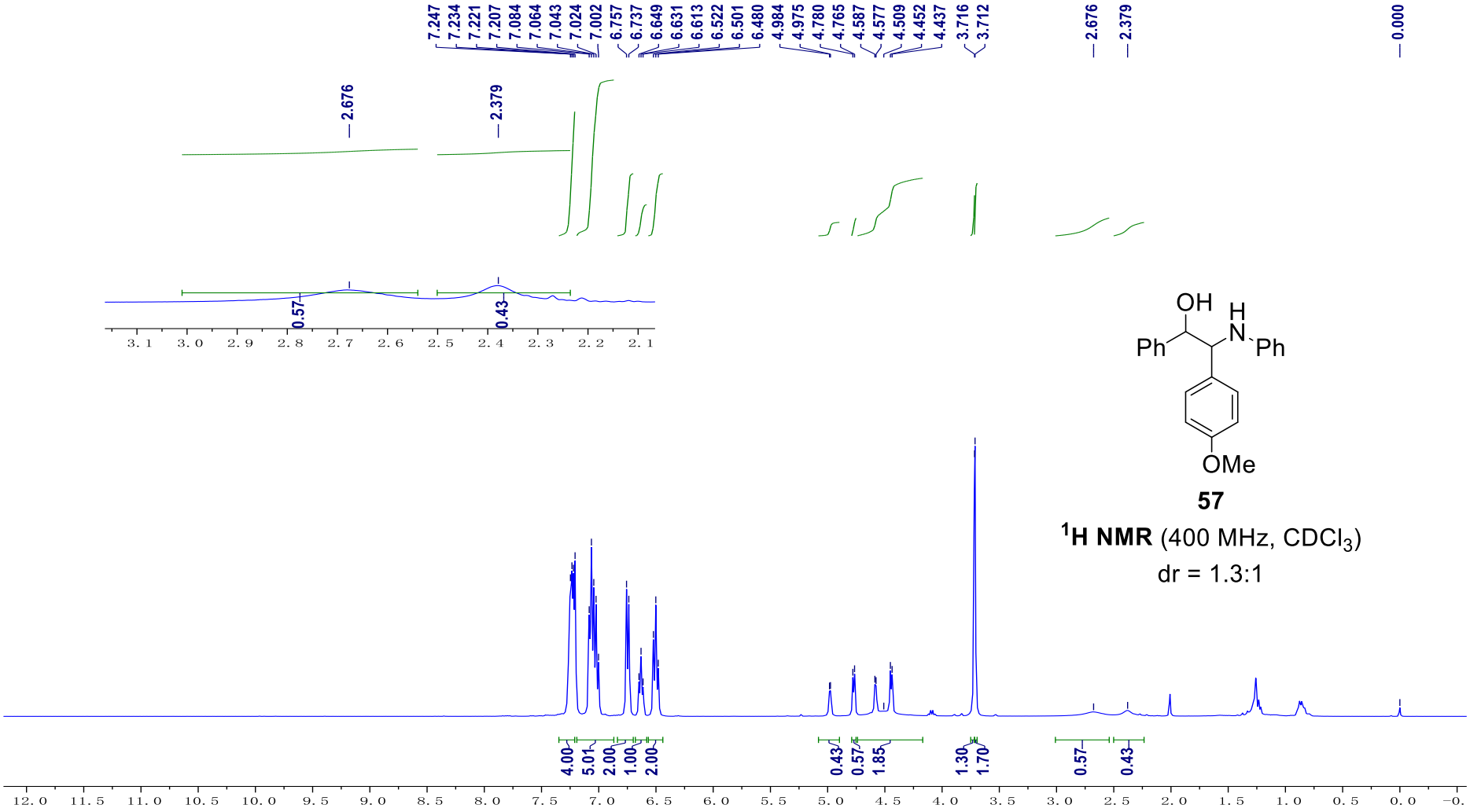

147.090
146.752
141.642
139.085
129.110
129.088
128.761
128.555
127.996
127.517
127.240
126.862
117.273
117.110
113.312
113.258
80.539
78.841
77.423
77.000
76.576
58.622
58.303
40.022
39.515
29.987
29.399
29.027
28.306
26.349
26.289
26.031
25.856
25.803
25.656
OH
H
N
Ph
Ph
54
13C NMR (75 MHz, CDCl3)

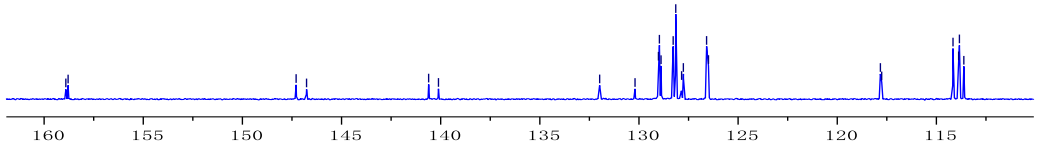

7.339
7.319
7.313
7.302
7.283
7.246
7.233
7.224
7.213
7.199
7.087
7.067
7.048
6.650
6.644
6.632
6.626
6.614
6.608
6.540
6.520
4.578
4.318
4.308
4.221
4.211
4.077
4.062
3.962
3.956
3.953
3.944
3.936
3.933
3.927
1.615
1.595
1.578
1.558
1.528
1.523
1.499
1.460
1.108
1.086
1.072
1.049
1.038
0.998
0.926
0.892
0.000
0.50
0.50
4.10
4.05
4.00
3.95
3.90
3.85
3.80
Me
OH
H
N
Ph
Me
Me
Ph
55
1H NMR (400 MHz, CDCl3)
dr = 1:1
5.00
2.00
1.00
2.00
0.88
0.50
0.50
0.50
0.50
2.03
1.03
4.51
4.50
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

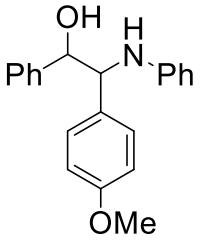

147.305
146.979
141.362
139.089
129.086
129.074
128.751
128.466
127.728
127.408
127.389
126.949
117.512
117.371
113.577
113.503
77.318
77.000
76.683
73.424
72.085
64.044
63.549
47.642
47.217
30.211
30.128
29.929
29.900
Me
OH
H
N
Ph
Me
Me
Ph
55
13C NMR (100 MHz, CDCl3)


7.390 7.386 7.366 7.353 7.335 7.324 7.307 7.296 7.280 7.269 7.249 7.237 7.101 7.082 7.080 7.061 6.658 6.655 6.652 6.640 6.637 6.634 6.622 6.619 6.616 6.552 6.550 6.531 6.511 6.509 4.635 4.529 4.518 4.510 4.120 3.690 3.681 3.673 3.663 3.655 3.646 3.542 3.524 2.634 2.008 1.963 1.811 1.803 1.793 1.783 1.765 1.749 1.732 1.665 1.633 1.606 1.450 1.445 1.407 1.387 1.378 1.356 1.345 1.324 1.313 1.289 1.254 1.227 1.217 1.196 1.185 -0.000
1.450
1.445
4.50
4.50
1.47 1.46 1.45 1.44 1.43 1.42
OH
H
N
Ph
Me
O
N
Ph
Me
Me
O
56
1H NMR (400 MHz, CDCl3)
dr = 1:1
5.09 2.02 1.00 2.00 1.01 1.01 2.01 0.50 0.50 2.03 1.05 2.02 1.01 4.50 4.50 2.65
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


154.724
146.800
146.568
141.097
138.837
129.171
129.136
128.857
128.716
127.910
127.757
127.404
126.788
117.596
117.339
113.267
79.726
79.376
79.325
77.754
77.318
77.000
76.682
58.907
58.044
38.525
38.175
28.903
28.414
28.407
27.529
OH
H
N
Ph
Ph
Me
O
N
Me
Me
O
56
13C NMR (100 MHz, CDCl3)

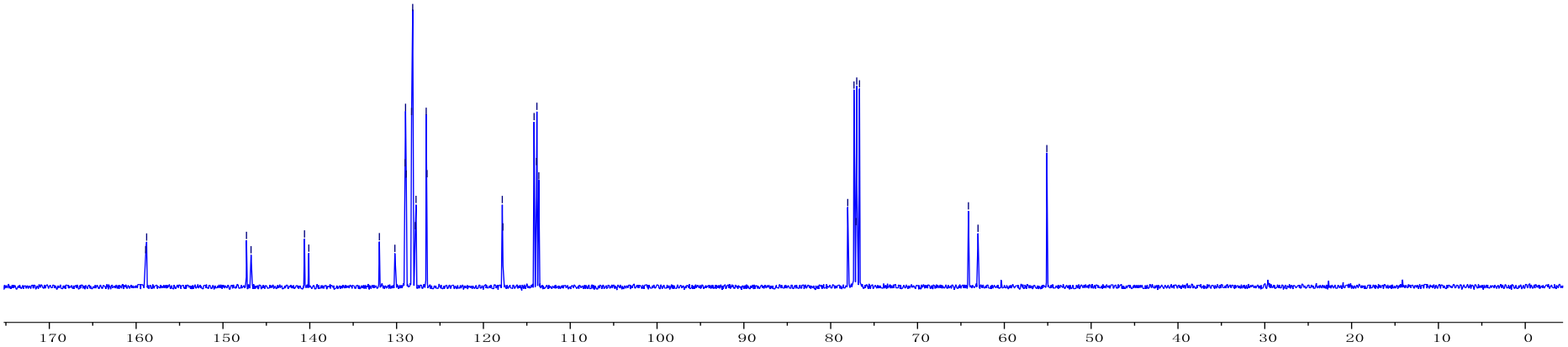

7.247
7.234
7.221
7.207
7.084
7.064
7.043
7.024
7.002
6.757
6.737
6.649
6.631
6.613
6.522
6.501
6.480
4.984
4.975
4.780
4.765
4.587
4.577
4.509
4.452
4.437
3.716
3.712
2.676
2.379
0.000
0.57
0.43
OH
H
N
Ph
Ph
OMe
57
1H NMR (400 MHz, CDCl3)
dr = 1.3:1
4.00
5.01
2.00
1.00
2.00
0.43
0.57
1.85
1.30
1.70
0.57
0.43

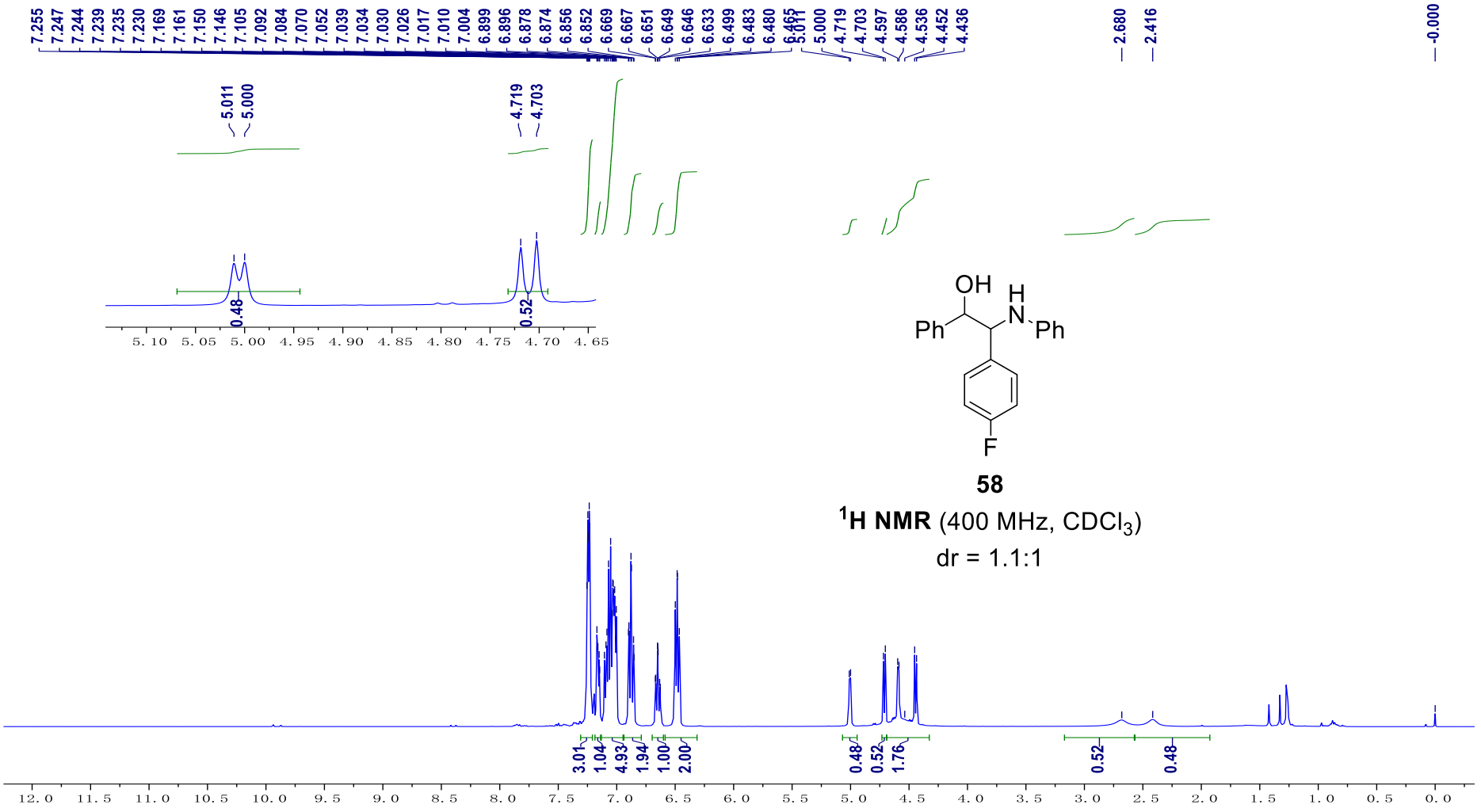


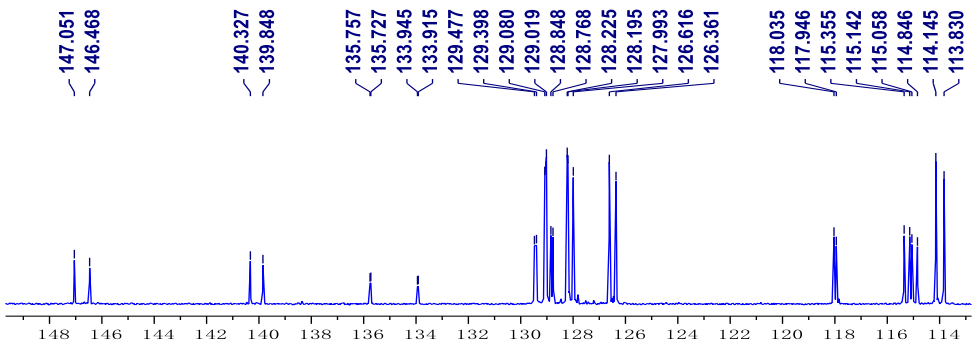


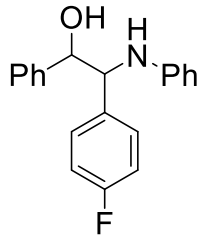
57

^{13}C NMR (100 MHz, $CDCl_3$)



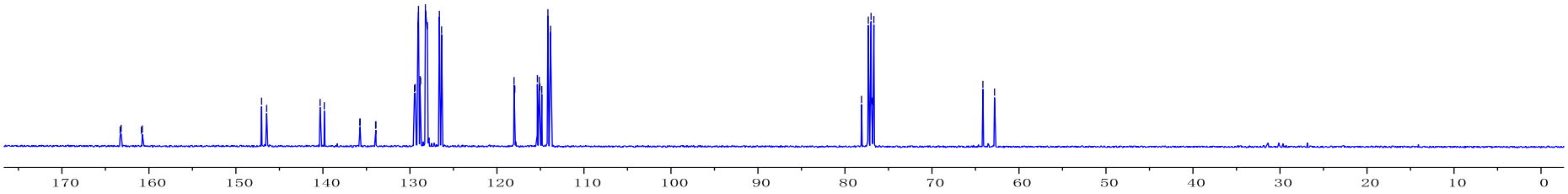

7.255
7.247
7.244
7.239
7.235
7.230
7.169
7.161
7.150
7.146
7.105
7.092
7.084
7.070
7.052
7.039
7.034
7.030
7.026
7.017
7.010
7.004
6.899
6.896
6.878
6.874
6.856
6.852
6.669
6.667
6.651
6.649
6.646
6.633
6.499
6.483
6.480
5.011
5.000
4.719
4.703
4.597
4.586
4.536
4.452
4.436
2.680
2.416
-0.000
0.48
0.52
OH
H
N
Ph
Ph
F
58
1H NMR (400 MHz, CDCl3)
dr = 1.1:1
3.01
1.04
4.93
1.94
1.00
2.00
0.48
0.52
1.76
0.52
0.48

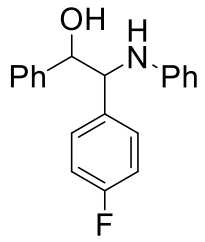




58

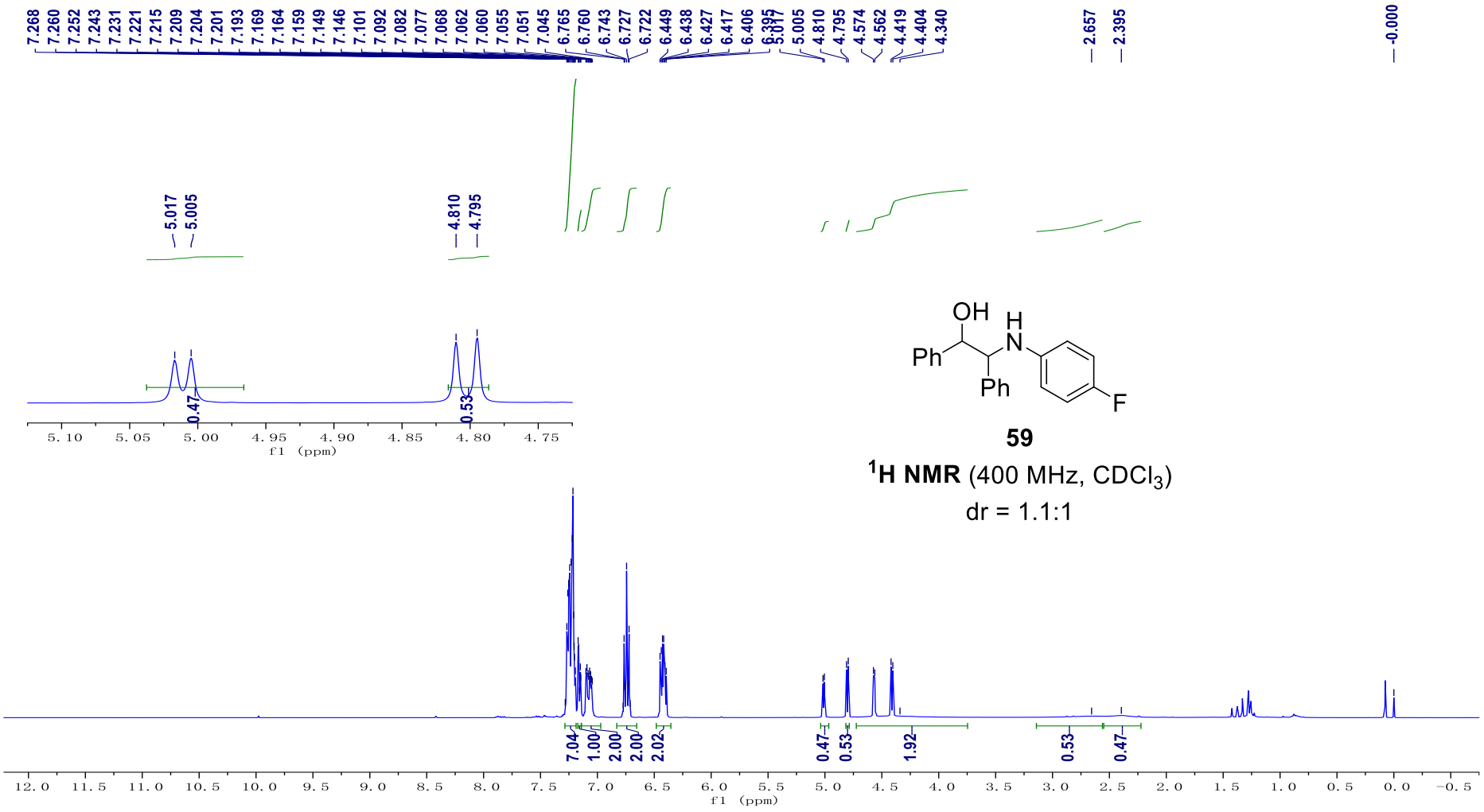
^{13}C NMR (100 MHz, $CDCl_3$)

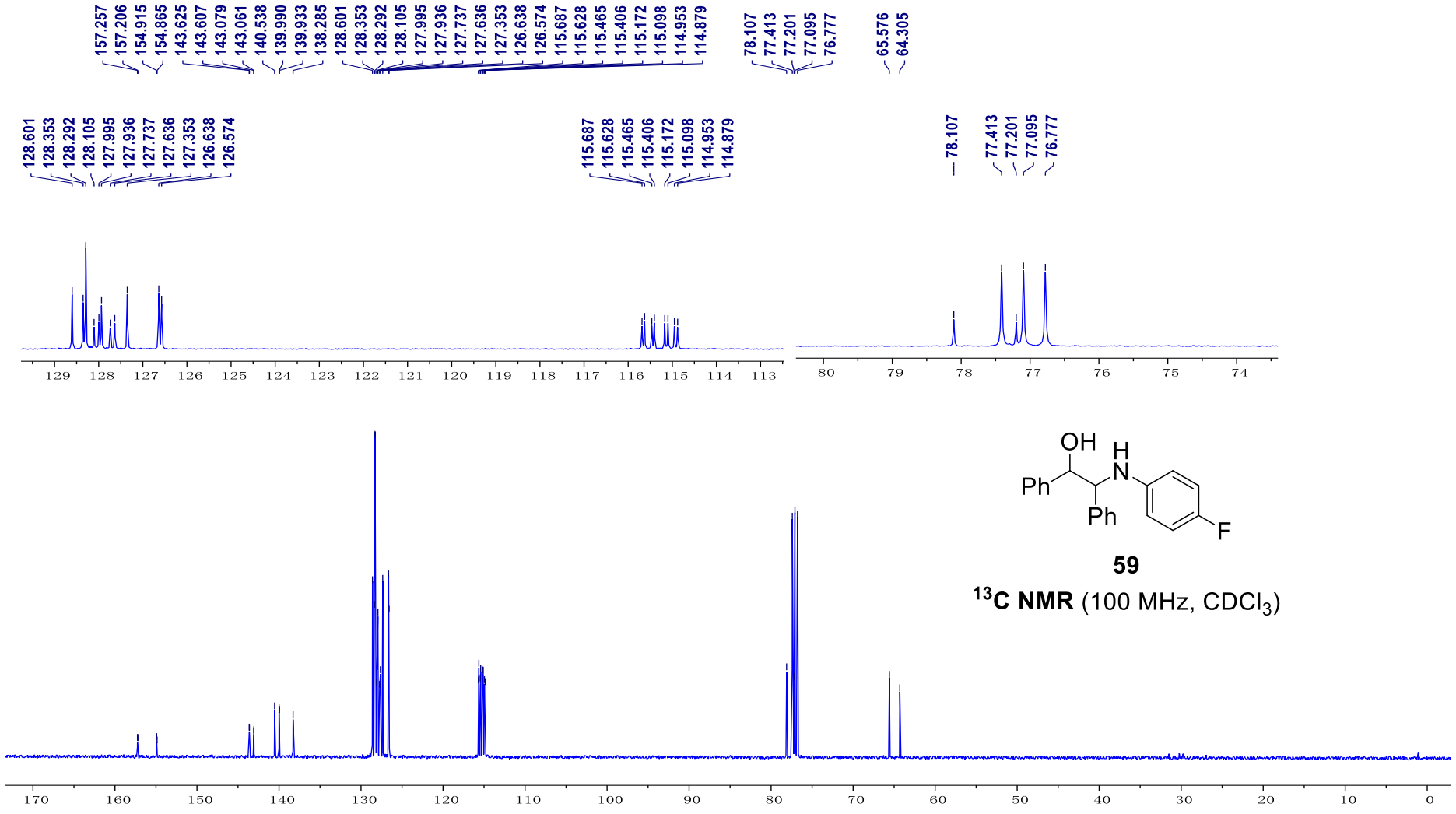


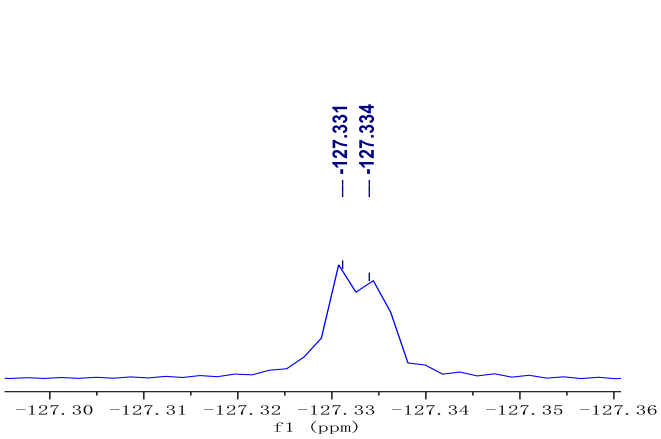


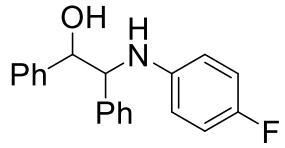
58

^{19}F NMR (376 MHz, $CDCl_3$)


7.268
7.260
7.252
7.243
7.231
7.221
7.215
7.209
7.204
7.201
7.193
7.169
7.164
7.159
7.149
7.146
7.101
7.092
7.082
7.077
7.068
7.062
7.060
7.055
7.051
7.045
6.765
6.760
6.743
6.727
6.722
6.449
6.438
6.427
6.417
6.406
6.395
5.017
5.005
4.810
4.795
4.574
4.562
4.419
4.404
4.340
2.657
2.395
-0.000
OH
H
N
Ph
Ph
F
59
1H NMR (400 MHz, CDCl3)
dr = 1.1:1
0.47
0.53
f1 (ppm)
7.04
1.00
2.00
2.00
2.02
0.47
0.53
1.92
0.53
0.47
f1 (ppm)

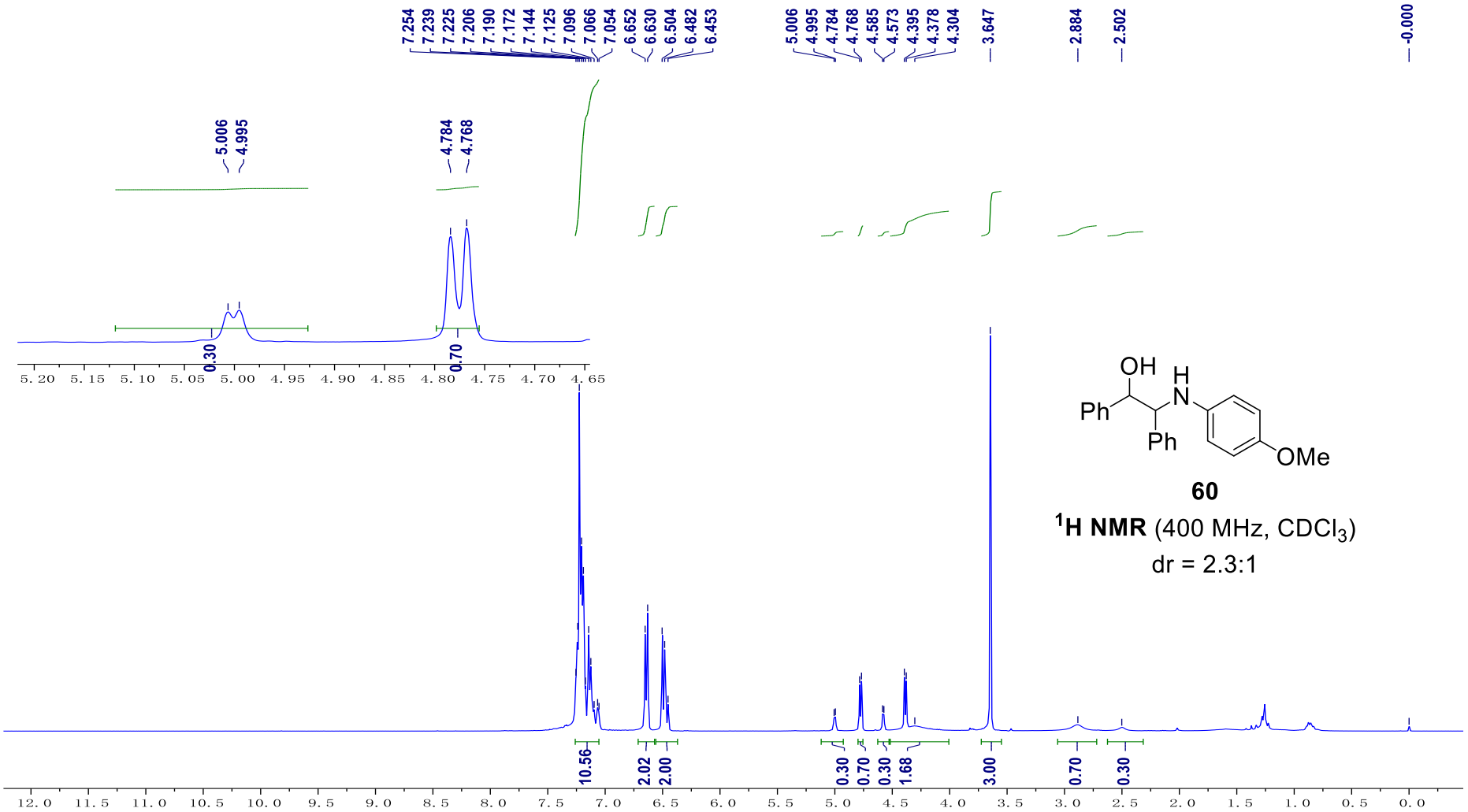

157.257
157.206
154.915
154.865
143.625
143.607
143.079
143.061
140.538
139.990
139.933
138.285
128.601
128.353
128.292
128.105
127.995
127.936
127.737
127.636
127.353
126.638
126.574
115.687
115.628
115.465
115.406
115.172
115.098
114.953
114.879
78.107
77.413
77.201
77.095
76.777
65.576
64.305
OH
H
N
Ph
Ph
F
59
13C NMR (100 MHz, CDCl3)

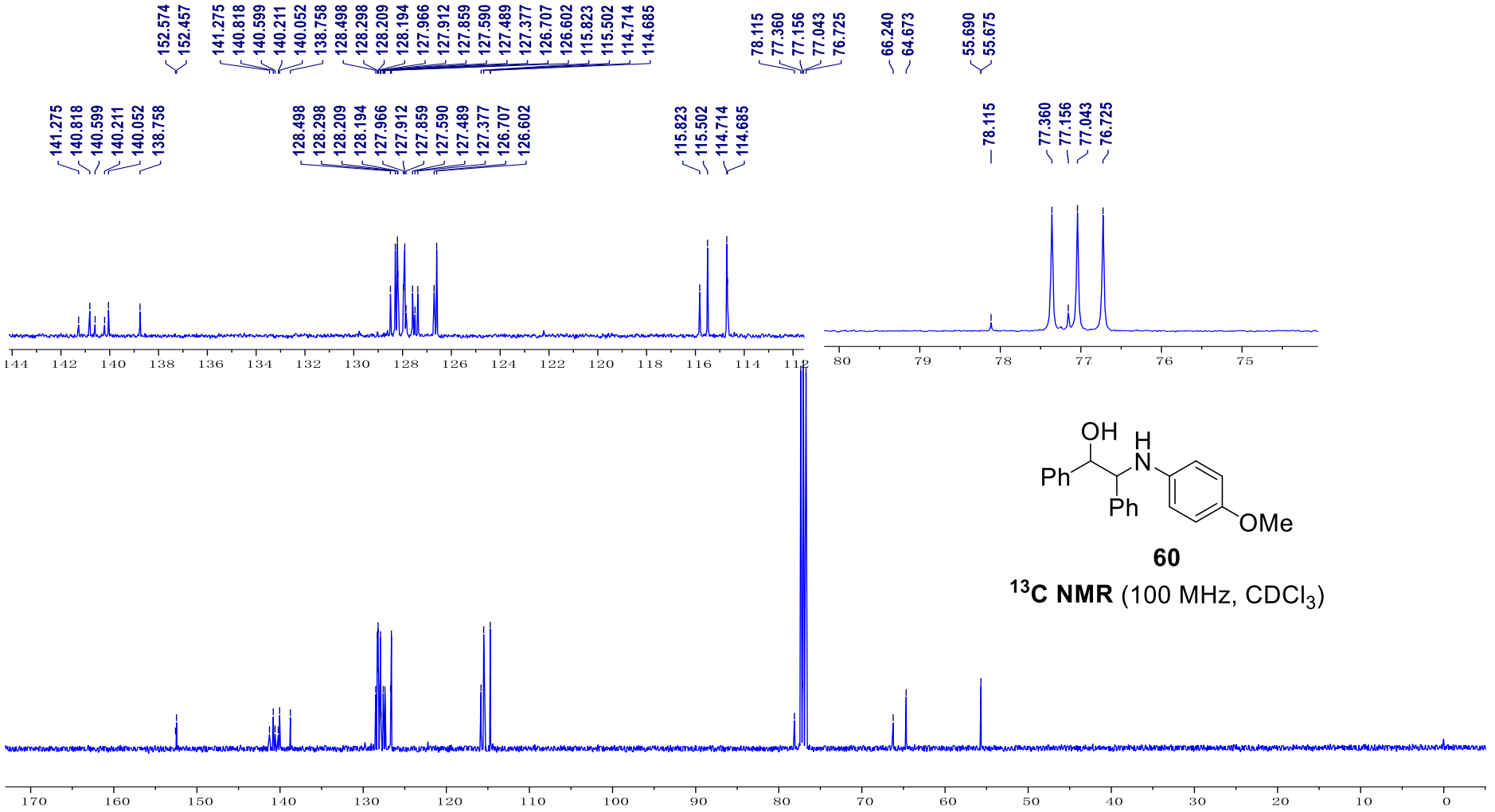


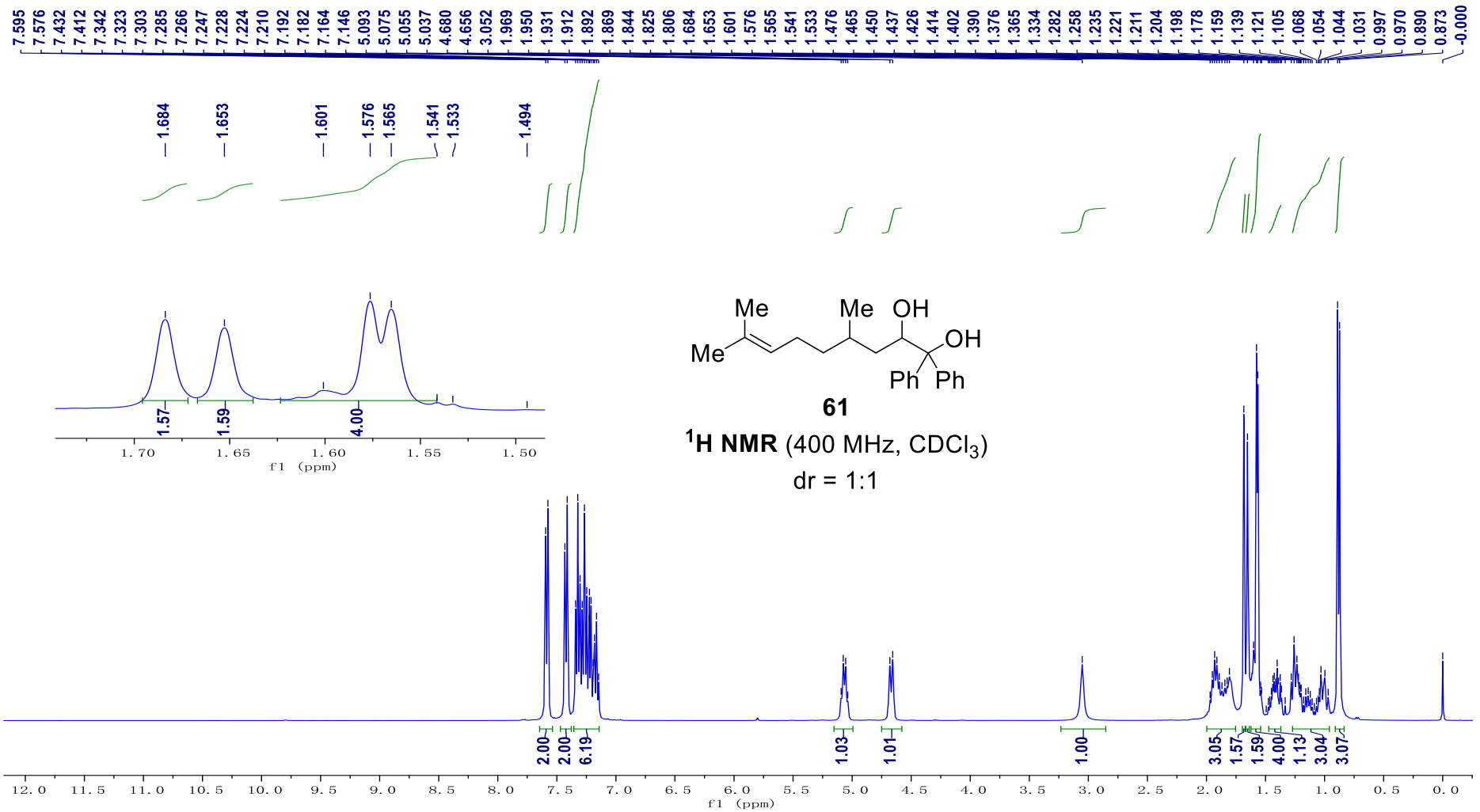


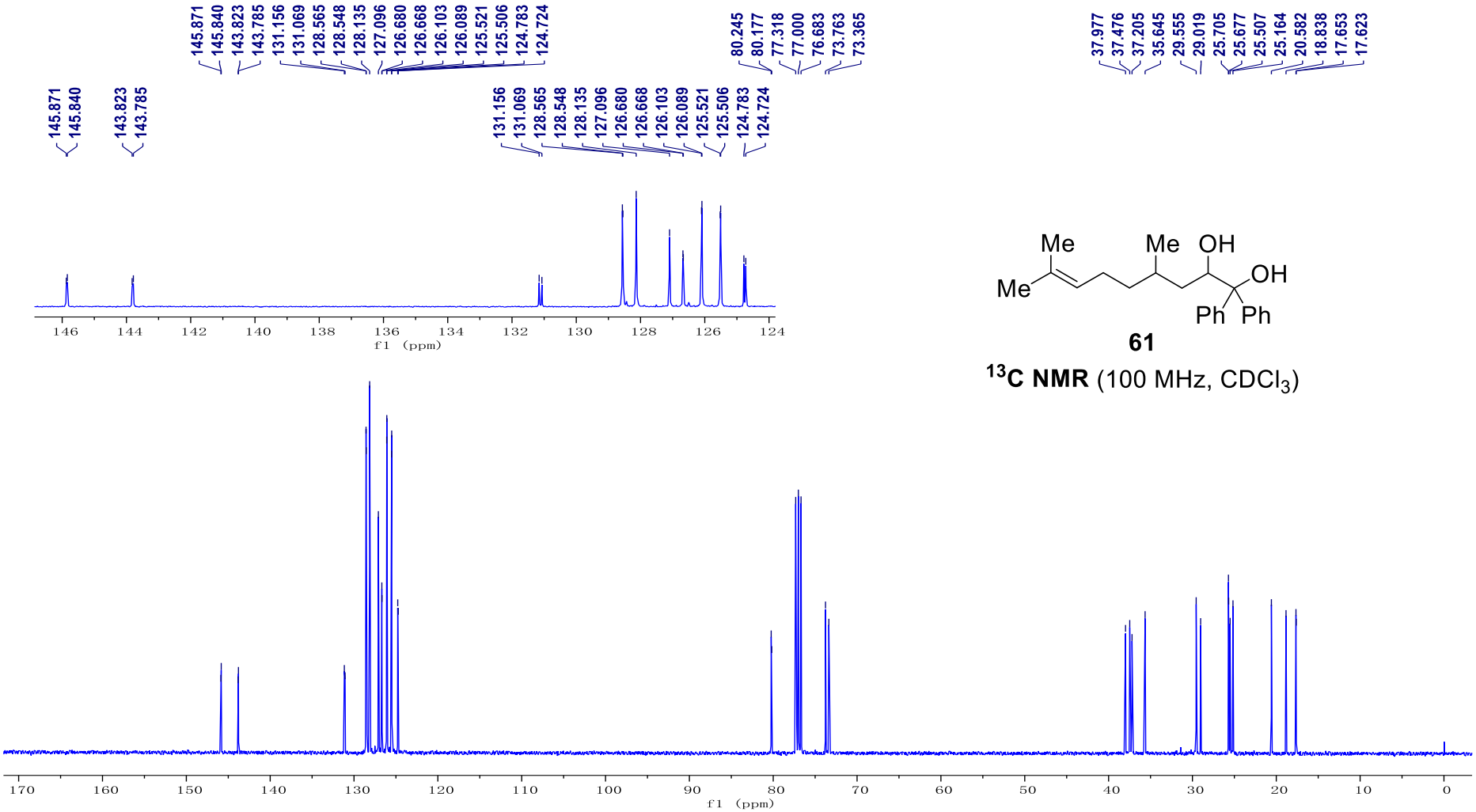
59

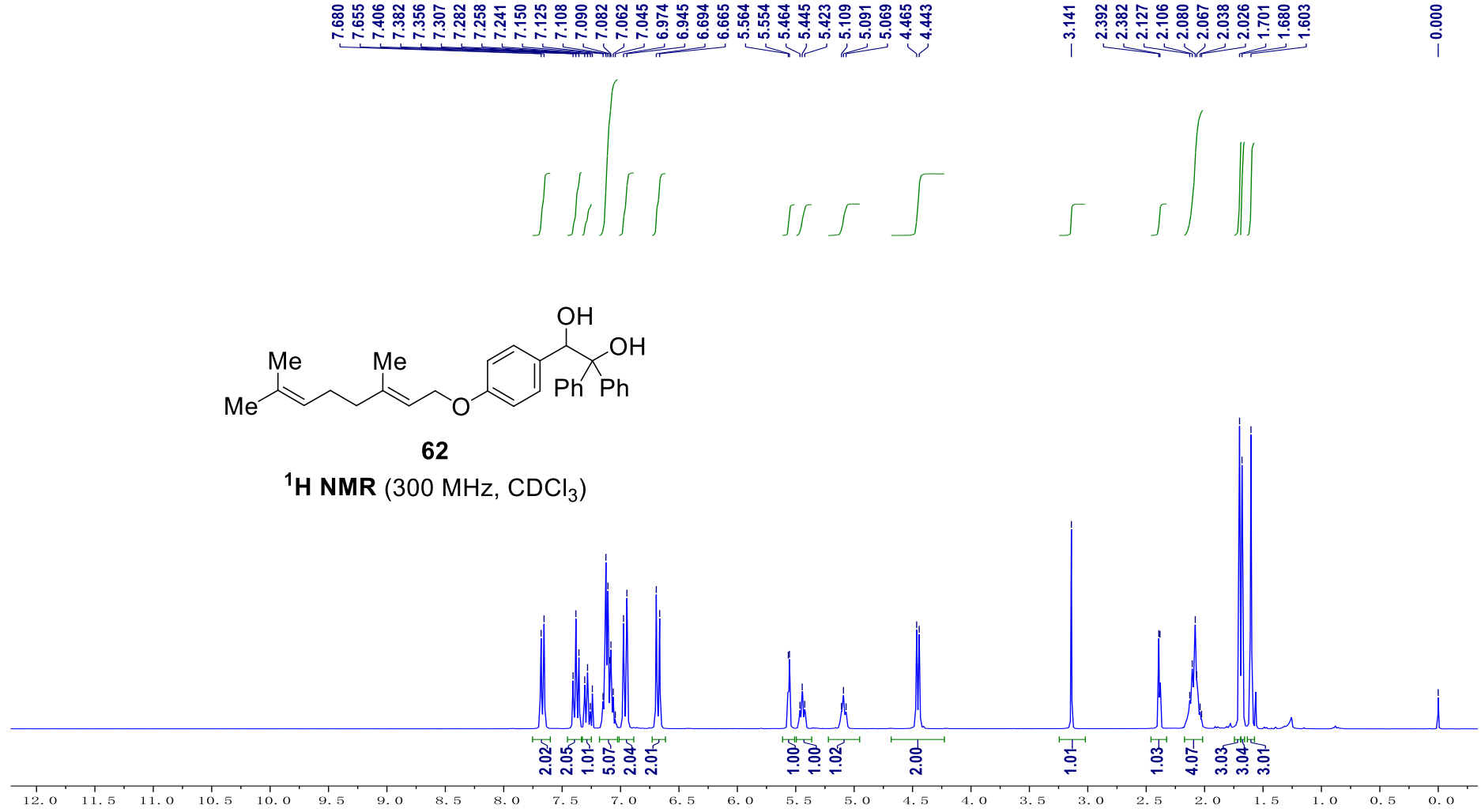
^{19}F NMR (376 MHz, $CDCl_3$)

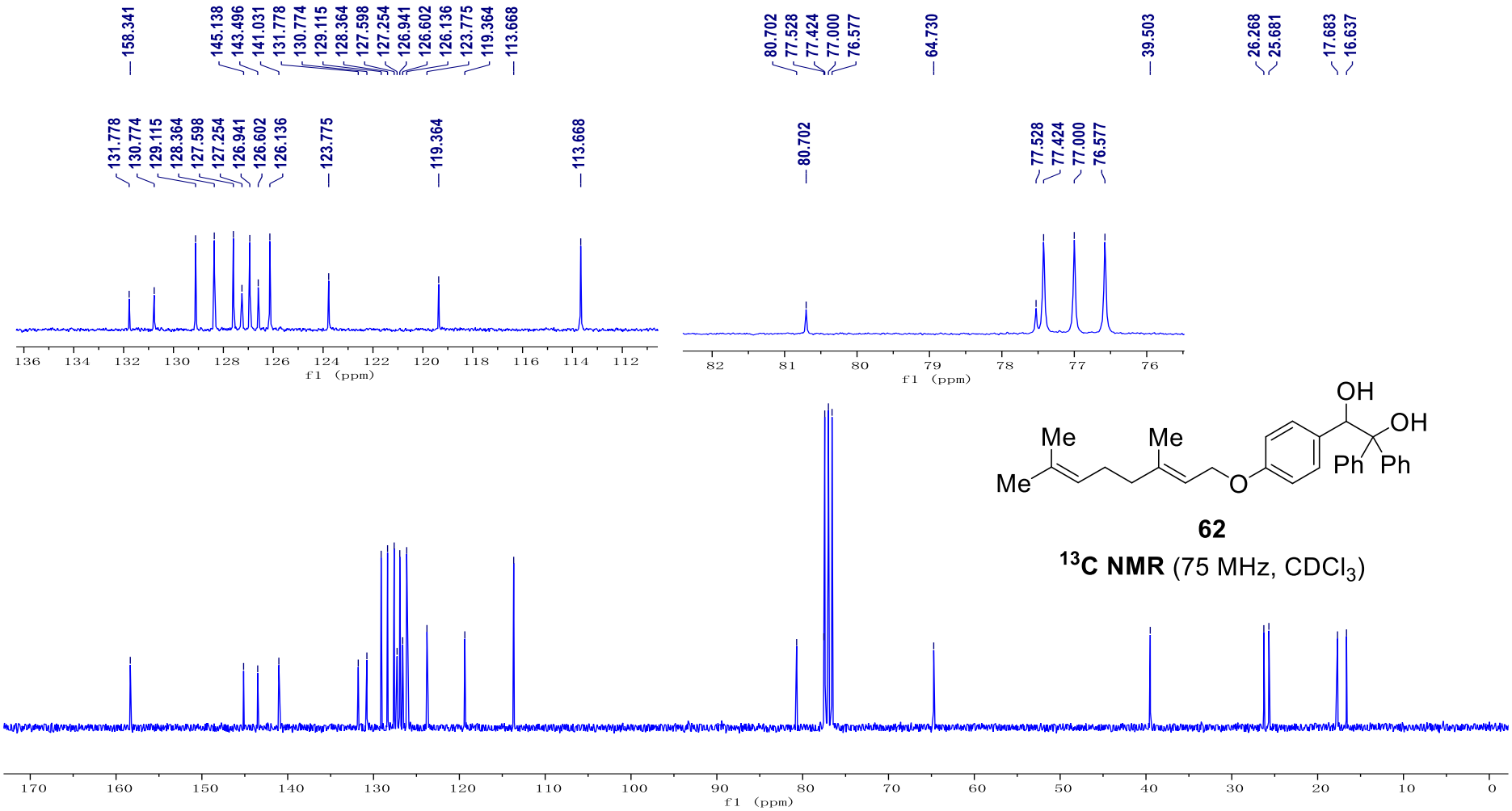

7.254
7.239
7.225
7.206
7.190
7.172
7.144
7.125
7.096
7.066
7.054
6.652
6.630
6.504
6.482
6.453
5.006
4.995
4.784
4.768
4.585
4.573
4.395
4.378
4.304
3.647
2.884
2.502
-0.000
0.30
0.70
10.56
2.02
2.00
0.30
0.70
0.30
1.68
3.00
0.70
0.30
OH
H
N
Ph
Ph
OMe
60
1H NMR (400 MHz, CDCl3)
dr = 2.3:1

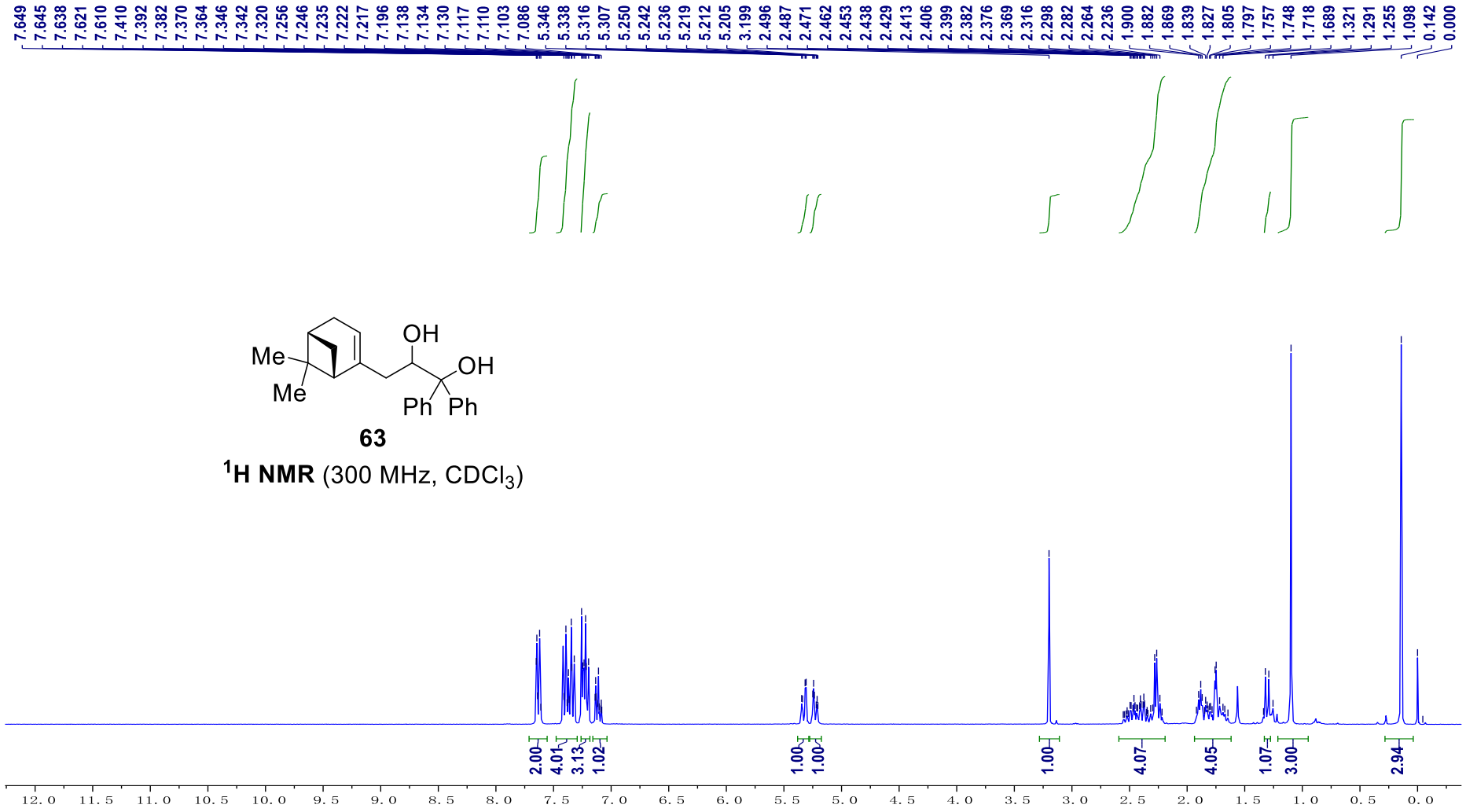

152.574
152.457
141.275
140.818
140.599
140.211
140.052
138.758
128.498
128.298
128.209
128.194
127.966
127.912
127.859
127.590
127.489
127.377
126.707
126.602
115.823
115.502
114.714
114.685
78.115
77.360
77.156
77.043
76.725
66.240
64.673
55.690
55.675
OH
H
N
Ph
Ph
OMe
60
13C NMR (100 MHz, CDCl3)


7.595 7.576 7.432 7.412 7.342 7.323 7.303 7.285 7.266 7.247 7.228 7.224 7.210 7.192 7.182 7.164 7.146 5.093 5.075 5.055 5.037 4.680 4.656 3.052 1.969 1.950 1.931 1.912 1.892 1.869 1.844 1.825 1.806 1.684 1.653 1.601 1.576 1.565 1.541 1.533 1.476 1.465 1.450 1.437 1.426 1.414 1.402 1.390 1.376 1.365 1.334 1.282 1.258 1.235 1.221 1.211 1.204 1.198 1.178 1.159 1.139 1.121 1.105 1.068 1.054 1.044 1.031 0.997 0.970 0.890 0.873 -0.000
1.684 1.653 1.601 1.576 1.565 1.541 1.533 1.494
1.57 1.59 4.00
1.70 1.65 1.60 1.55 1.50
f1 (ppm)
Me Me OH OH Me Ph Ph
61
1H NMR (400 MHz, CDCl3)
dr = 1:1
2.00 2.00 6.19 1.03 1.01 1.00 3.05 1.57 1.59 4.00 1.13 3.04 3.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


145.871
145.840
143.823
143.785
131.156
131.069
128.565
128.548
128.135
127.096
126.680
126.668
126.103
126.089
125.521
125.506
124.783
124.724
80.245
80.177
77.318
77.000
76.683
73.763
73.365
37.977
37.476
37.205
35.645
29.555
29.019
25.705
25.677
25.507
25.164
20.582
18.838
17.653
17.623
f1 (ppm)
Me
Me
OH
OH
Ph
Ph
61
13C NMR (100 MHz, CDCl3)

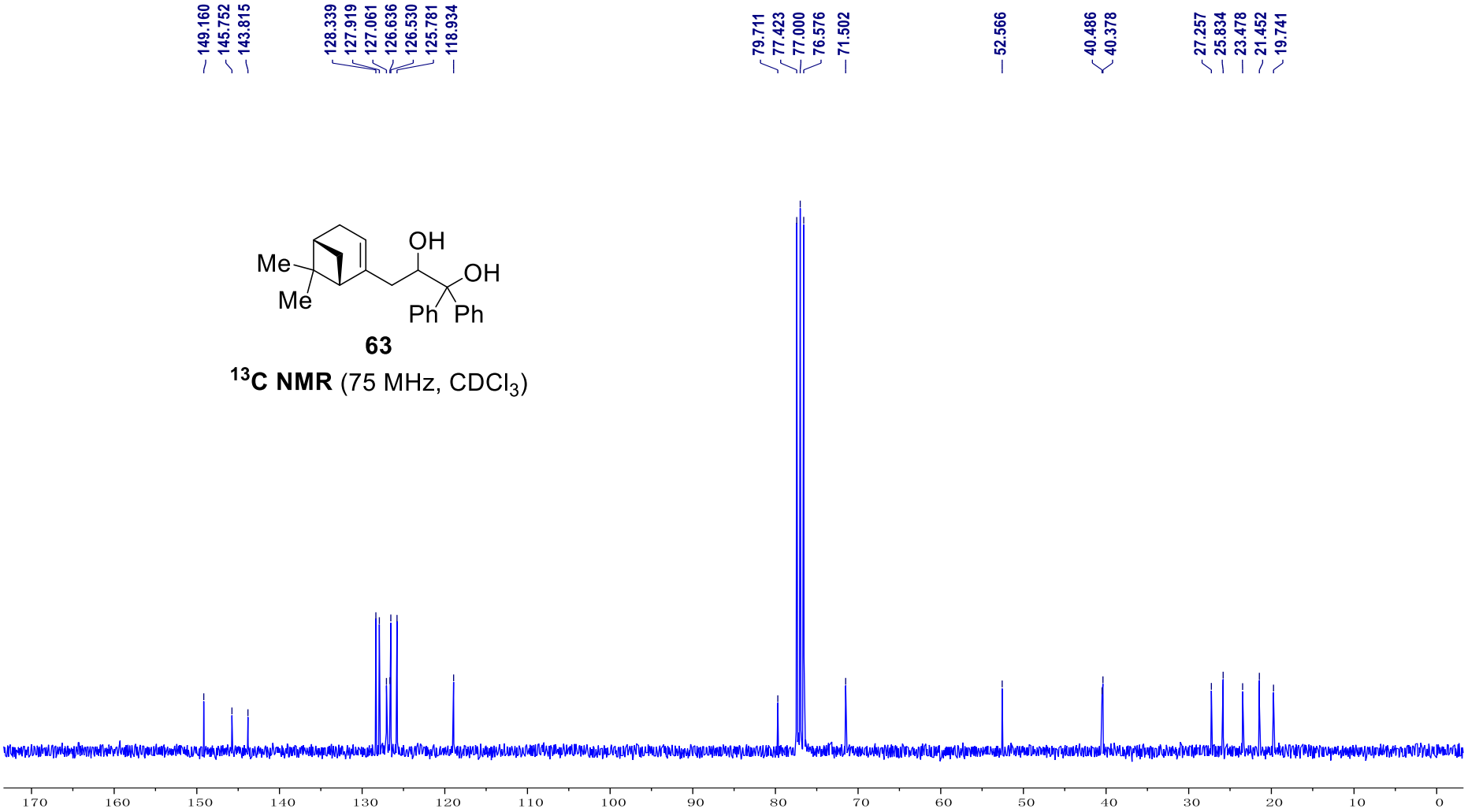

7.680
7.655
7.406
7.382
7.356
7.307
7.282
7.258
7.241
7.150
7.125
7.108
7.090
7.082
7.062
7.045
6.974
6.945
6.694
6.665
5.564
5.554
5.464
5.445
5.423
5.109
5.091
5.069
4.465
4.443
3.141
2.392
2.382
2.127
2.106
2.080
2.067
2.038
2.026
1.701
1.680
1.603
0.000
OH
OH
Me
Me
Me
Ph
Ph
O
62
1H NMR (300 MHz, CDCl3)
2.02
2.05
1.01
5.07
2.04
2.01
1.00
1.00
1.02
2.00
1.01
1.03
4.07
3.03
3.04
3.01
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

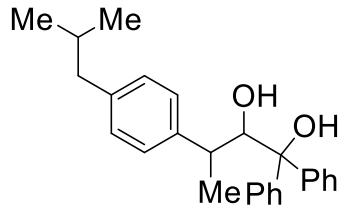

158.341
145.138
143.496
141.031
131.778
130.774
129.115
128.364
127.598
127.254
126.941
126.602
126.136
123.775
119.364
113.668
80.702
77.528
77.424
77.000
76.577
64.730
39.503
26.268
25.681
17.683
16.637
f1 (ppm)
OH
OH
Me
Me
Me
Ph
Ph
O
62
13C NMR (75 MHz, CDCl3)



63

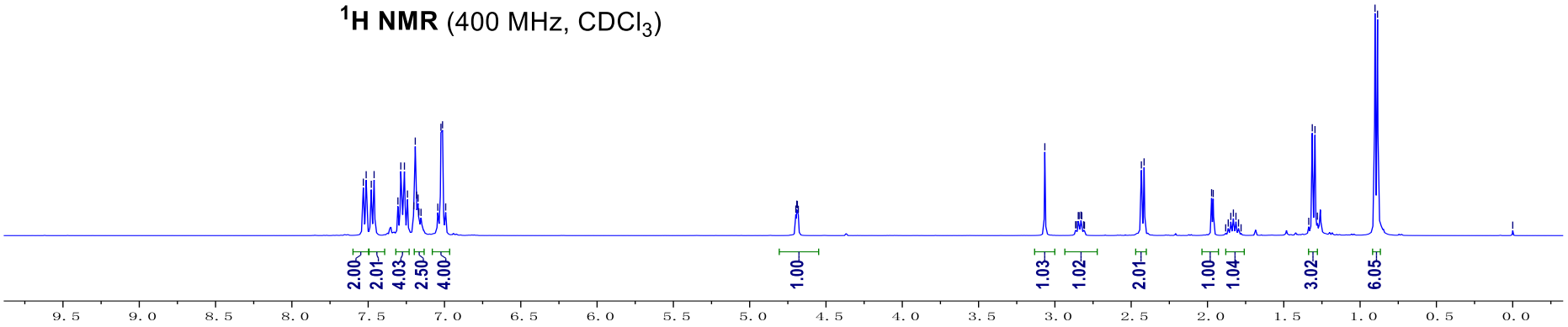
^{1}H NMR (300 MHz, $CDCl_3$)

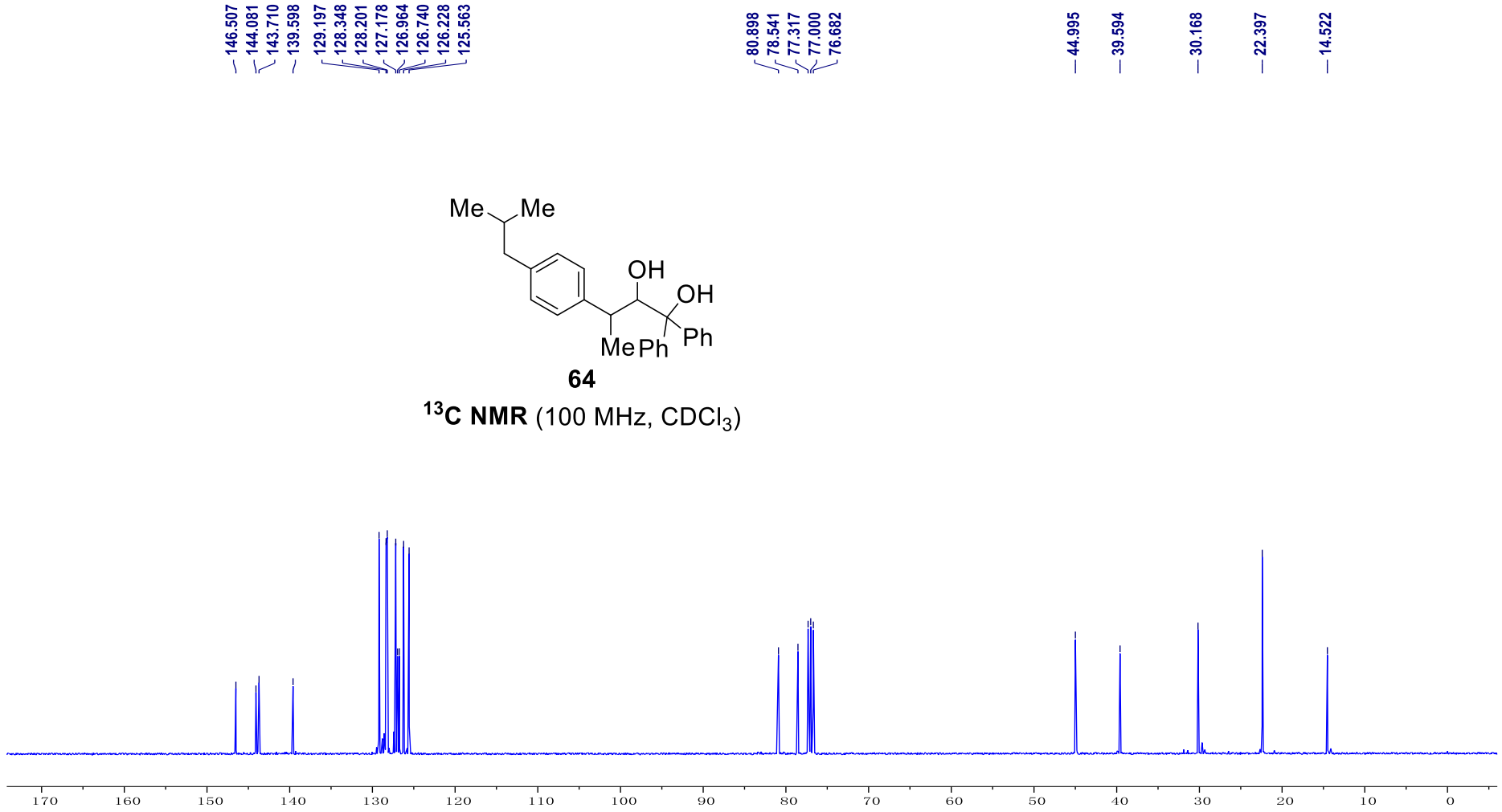

149.160
145.752
143.815
128.339
127.919
127.061
126.636
126.530
125.781
118.934
79.711
77.423
77.000
76.576
71.502
52.566
40.486
40.378
27.257
25.834
23.478
21.452
19.741
OH
Me
OH
Me
Ph
Ph
63
13C NMR (75 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



64

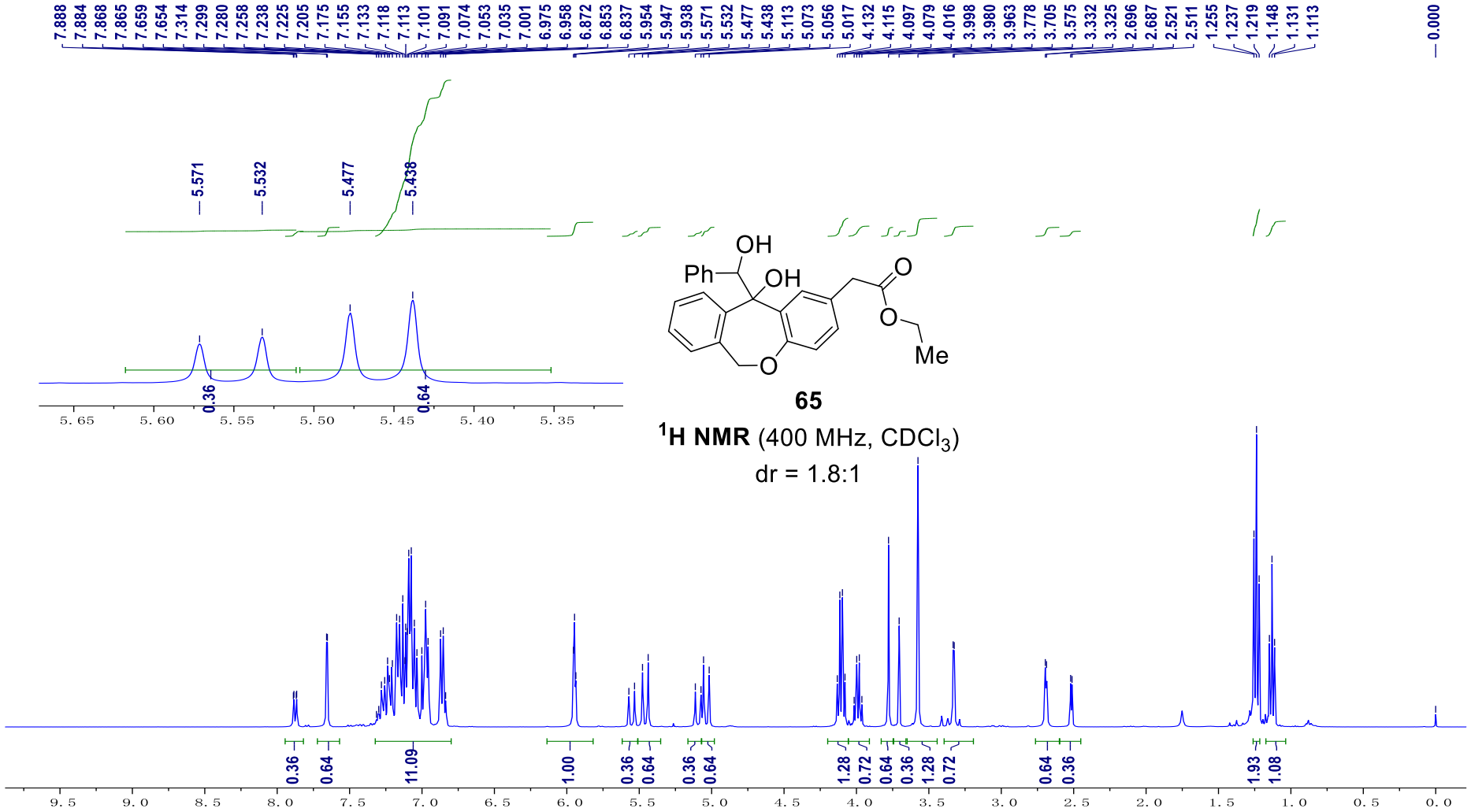
^{1}H NMR (400 MHz, $CDCl_3$)

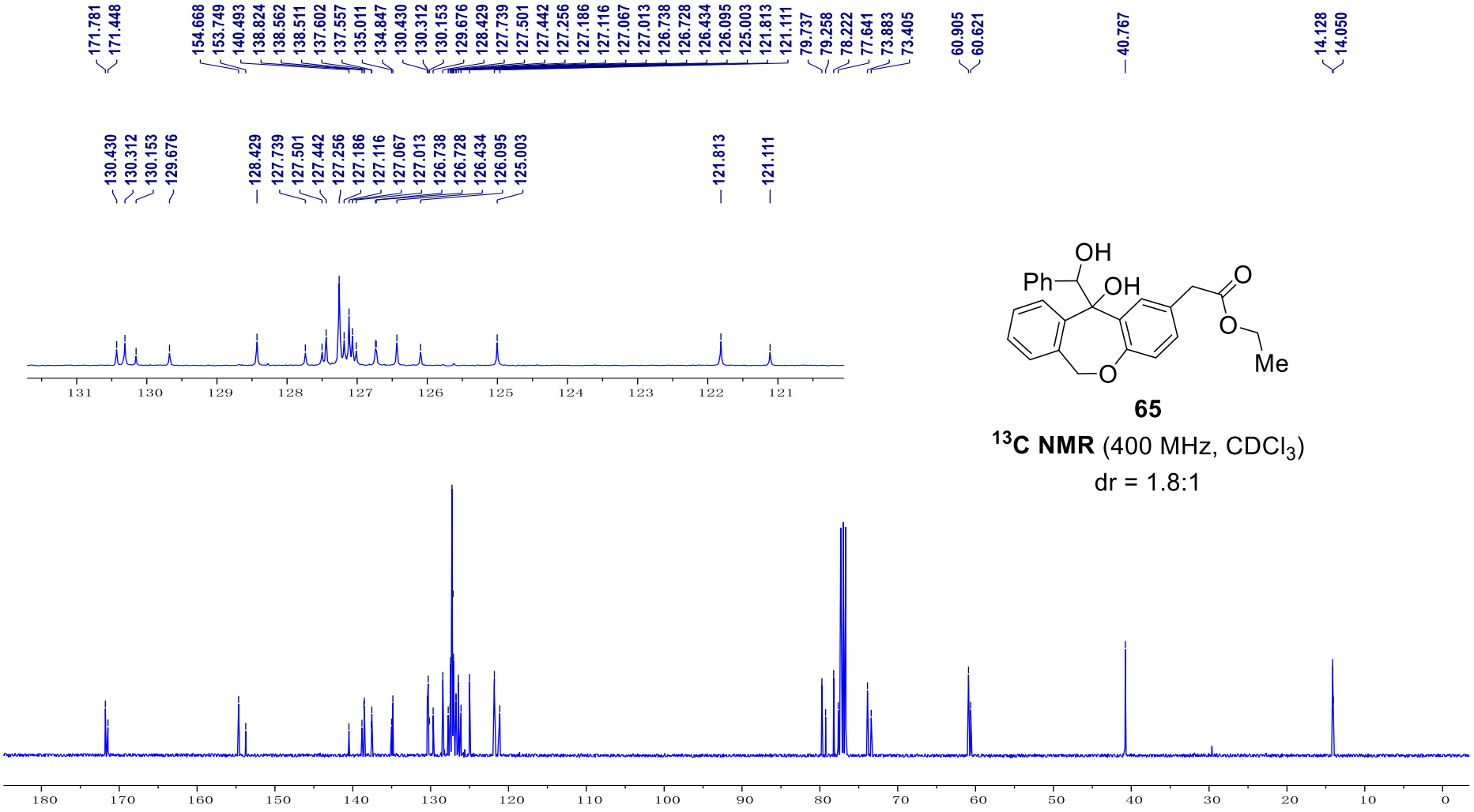


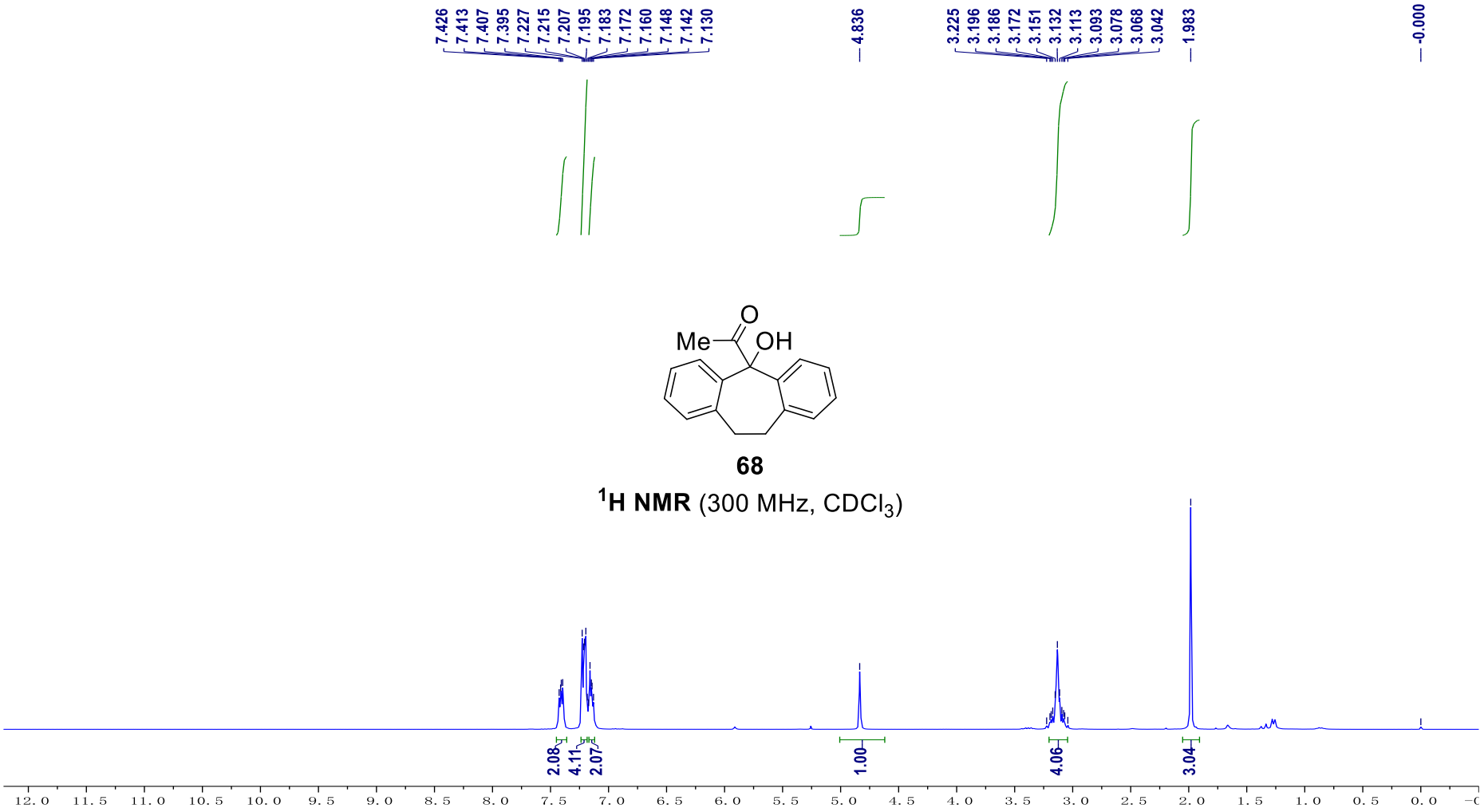


64

^{13}C NMR (100 MHz, $CDCl_3$)

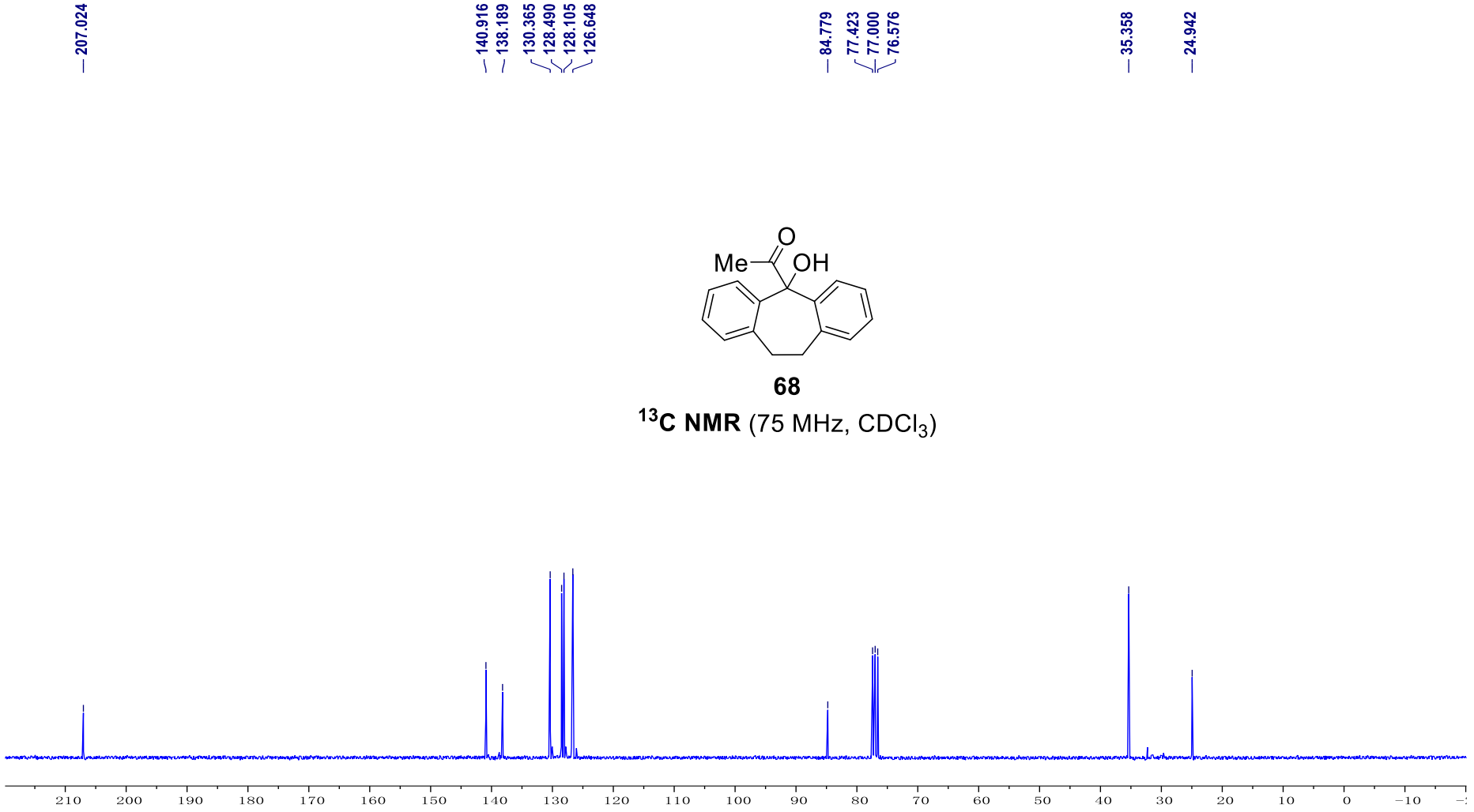

7.888 7.884 7.868 7.865 7.659 7.654 7.314 7.299 7.280 7.258 7.238 7.225 7.205 7.175 7.155 7.133 7.118 7.113 7.101 7.091 7.074 7.053 7.035 7.001 6.975 6.958 6.872 6.853 6.837 5.954 5.947 5.938 5.571 5.532 5.477 5.438 5.113 5.073 5.056 5.017 4.132 4.115 4.097 4.079 4.016 3.998 3.980 3.963 3.778 3.705 3.575 3.332 3.325 2.696 2.687 2.521 2.511 1.255 1.237 1.219 1.148 1.131 1.113 0.000
5.571 5.532 5.477 5.438
0.36 0.64
OH Ph OH O O Me O
65
1H NMR (400 MHz, CDCl3)
dr = 1.8:1
0.36 0.64 11.09 1.00 0.36 0.64 0.36 0.64 1.28 0.72 0.64 0.36 1.28 0.72 0.64 0.36 1.93 1.08


171.781
171.448
154.668
153.749
140.493
138.824
138.562
138.511
137.602
137.557
135.011
134.847
130.430
130.312
130.153
129.676
128.429
127.739
127.501
127.442
127.256
127.186
127.116
127.067
127.013
126.738
126.728
126.434
126.095
125.003
121.813
121.111
79.737
79.258
78.222
77.641
73.883
73.405
60.905
60.621
40.767
14.128
14.050
OH
Ph
OH
O
O
Me
O
65
13C NMR (400 MHz, CDCl3)
dr = 1.8:1



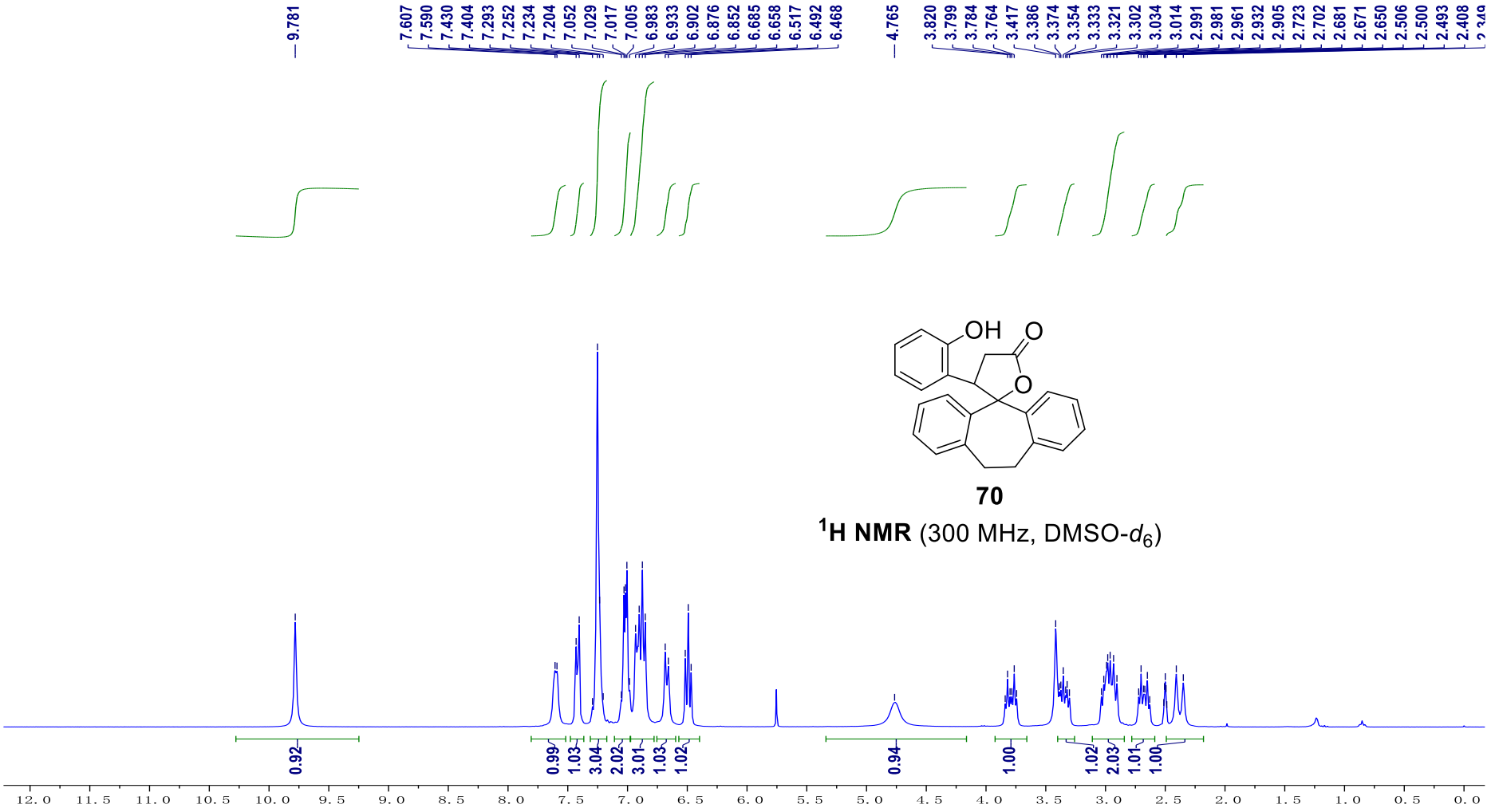
68

^{1}H NMR (300 MHz, $CDCl_3$)



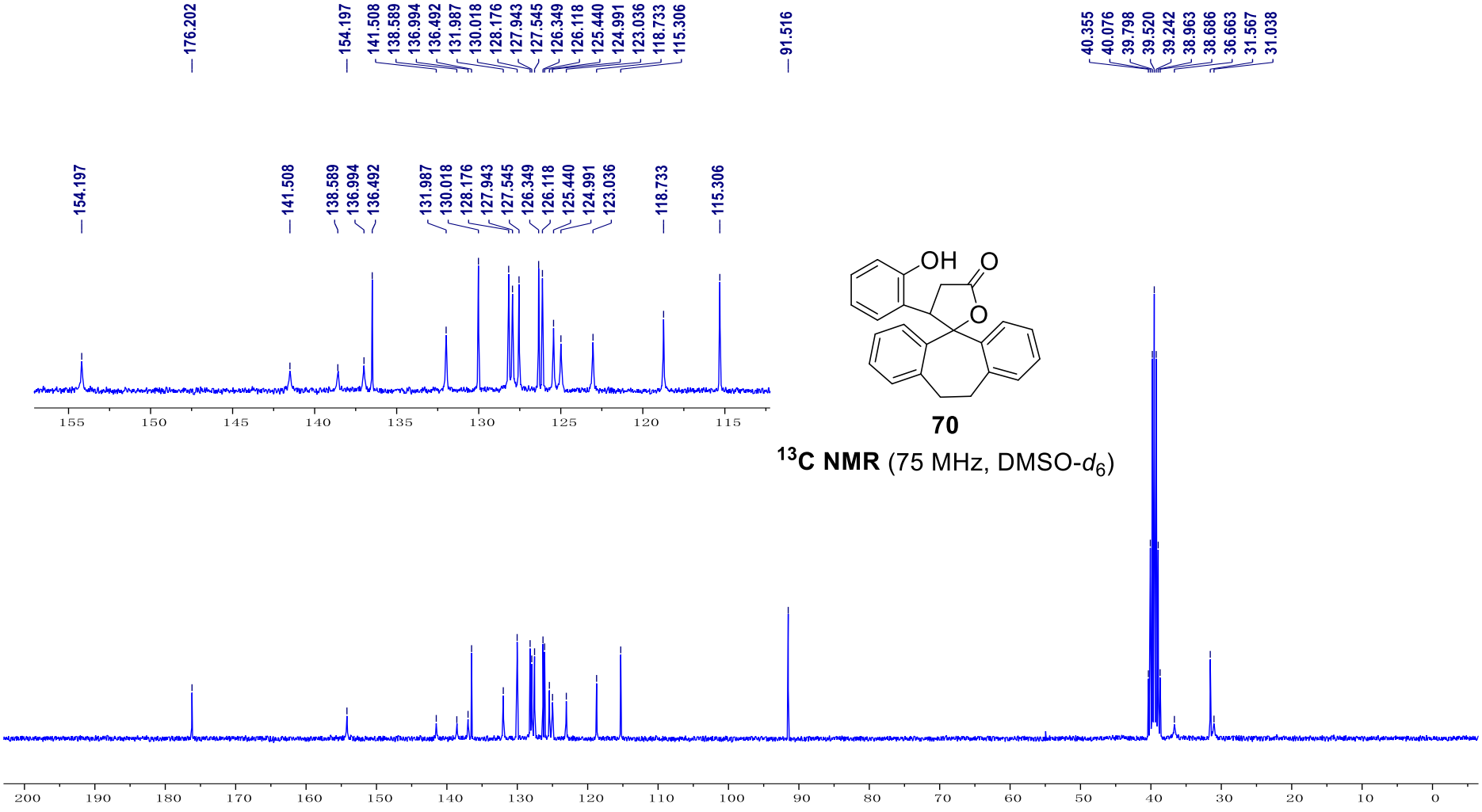
68

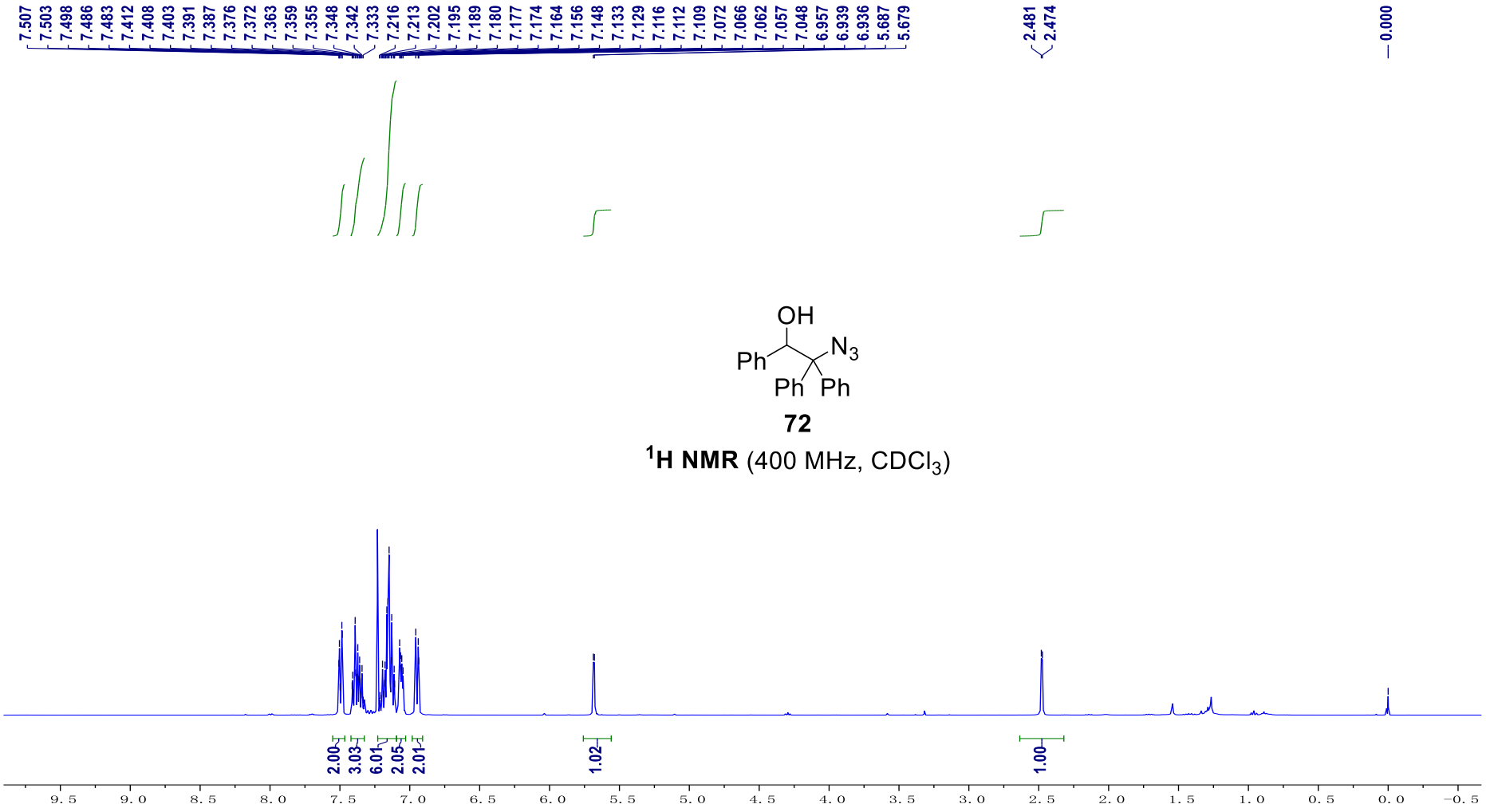
^{13}C NMR (75 MHz, $CDCl_3$)



70

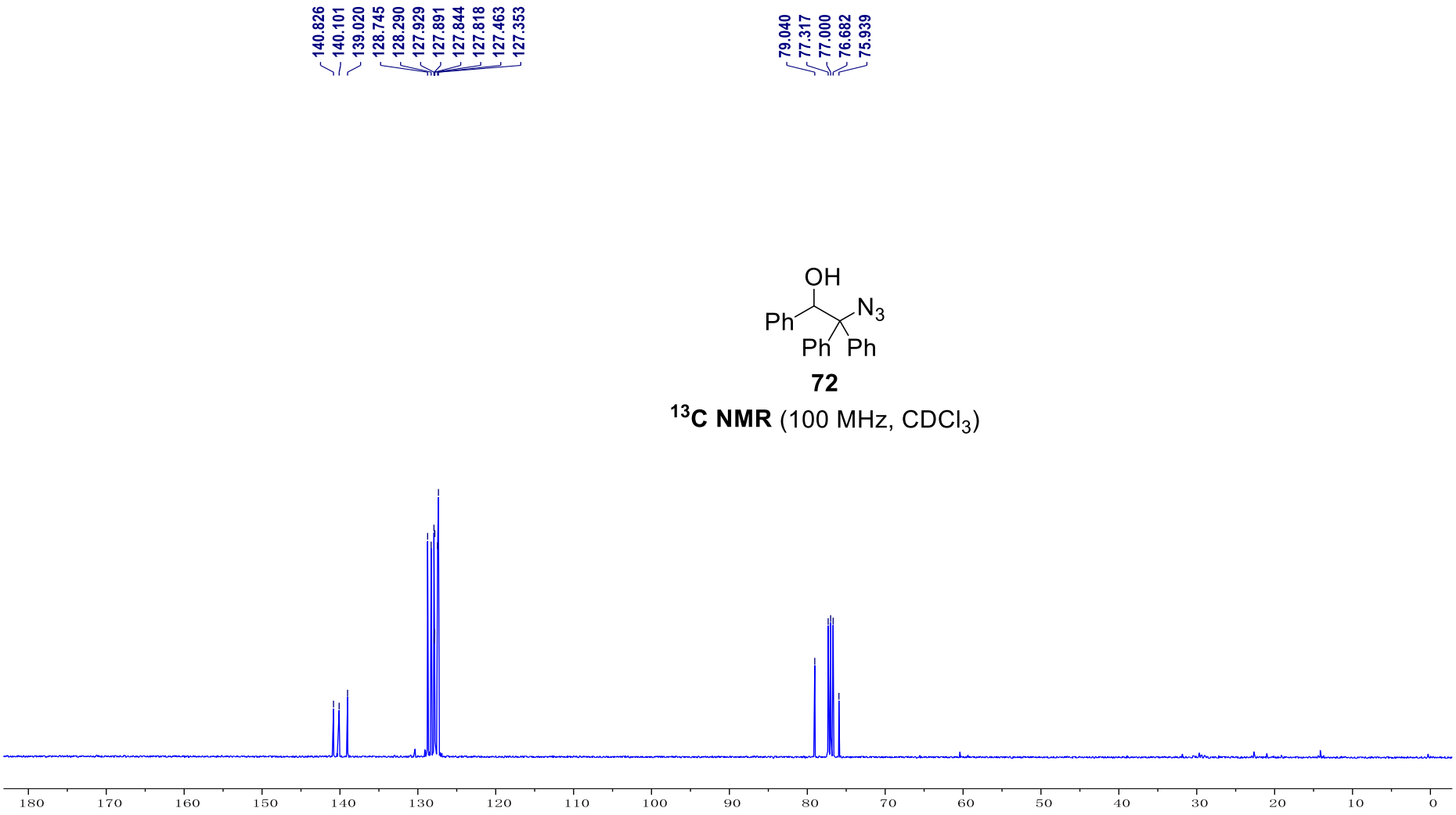
^{1}H NMR (300 MHz, DMSO-d_6)


176.202
154.197
141.508
138.589
136.994
136.492
131.987
130.018
128.176
127.943
127.545
126.349
126.118
125.440
124.991
123.036
118.733
115.306
91.516
40.355
40.076
39.798
39.520
39.242
38.963
38.686
36.663
31.567
31.038
OH
O
O
70
13C NMR (75 MHz, DMSO-d6)



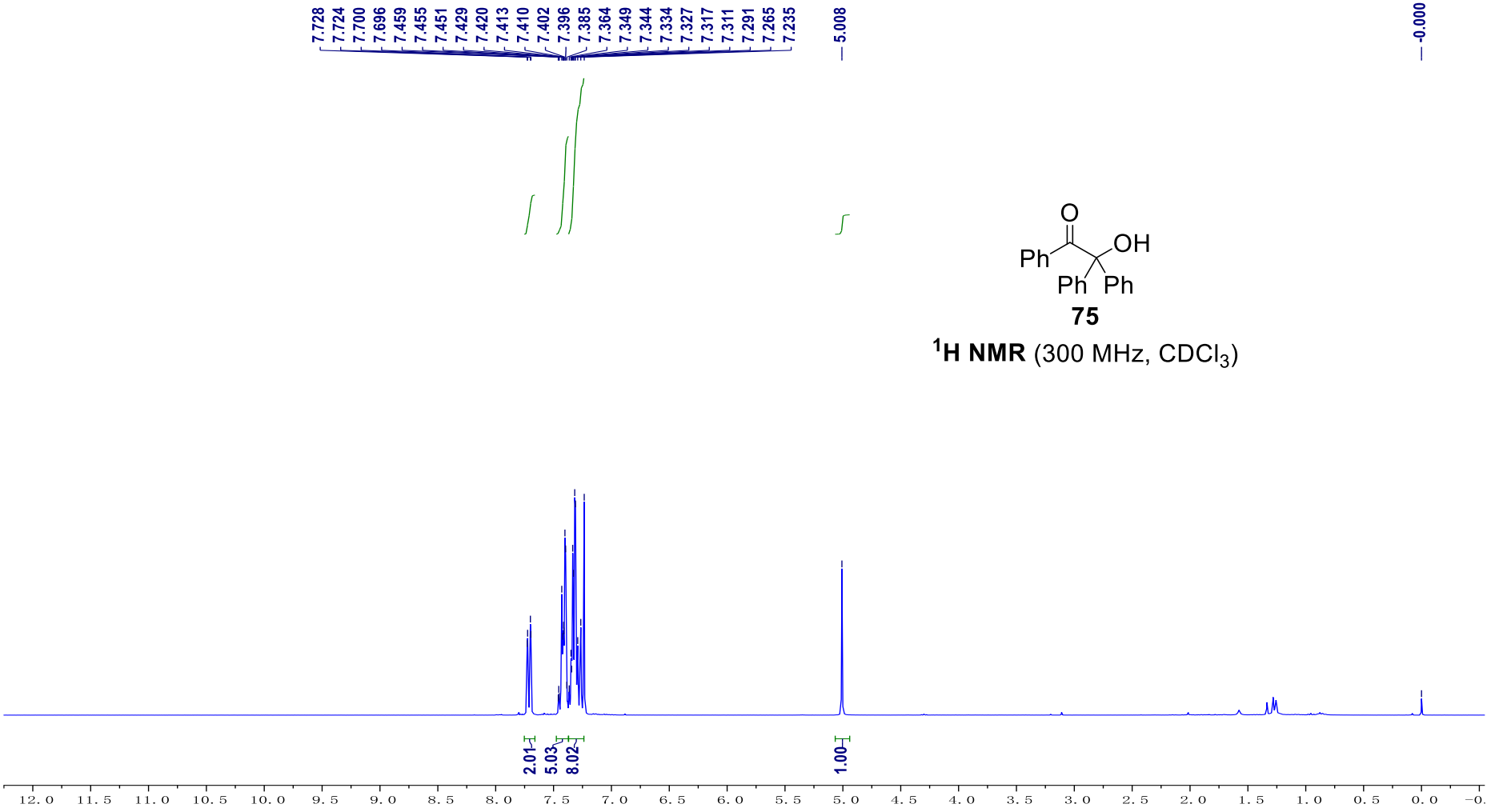
72

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



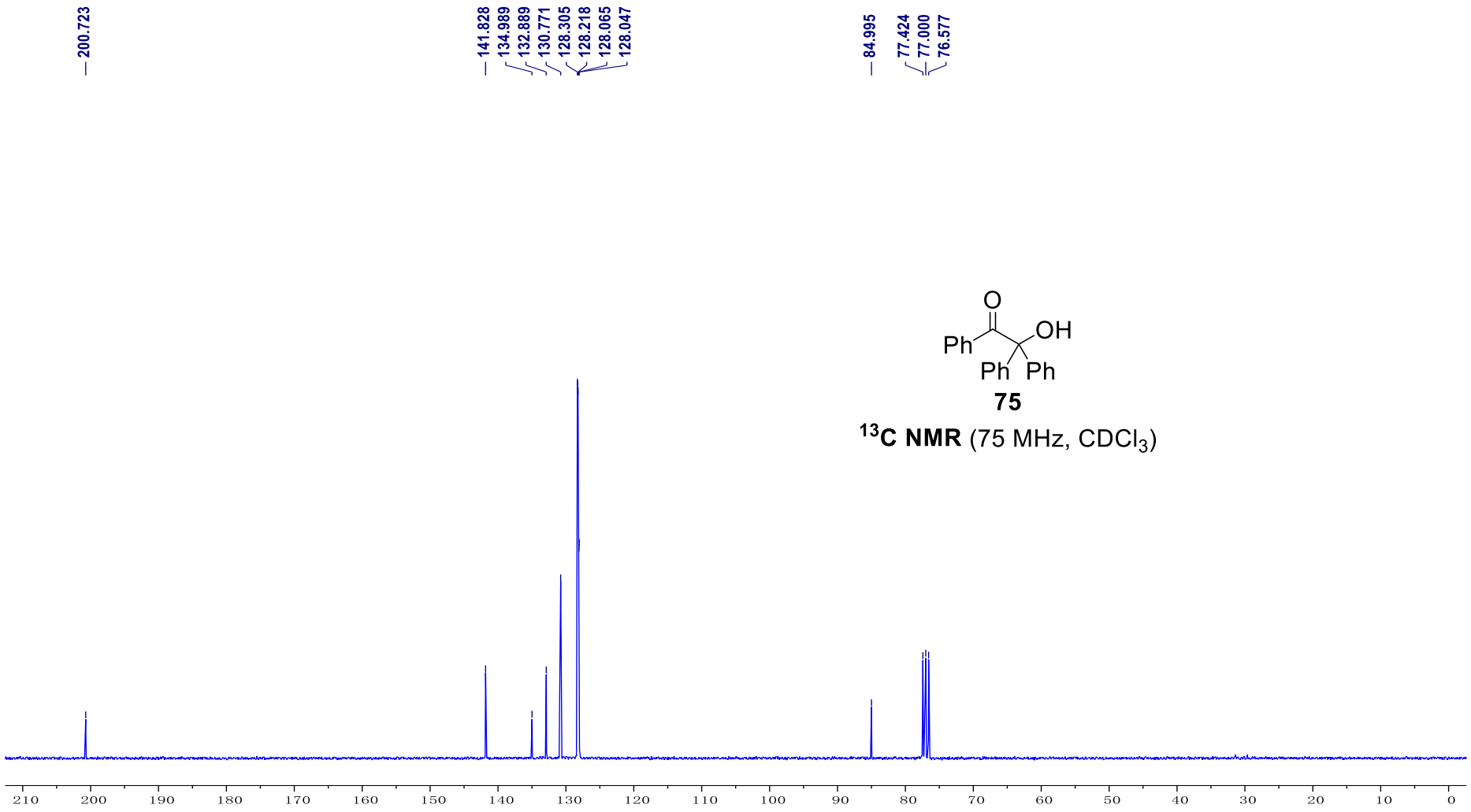
72

^{13}C NMR (100 MHz, $CDCl_3$)



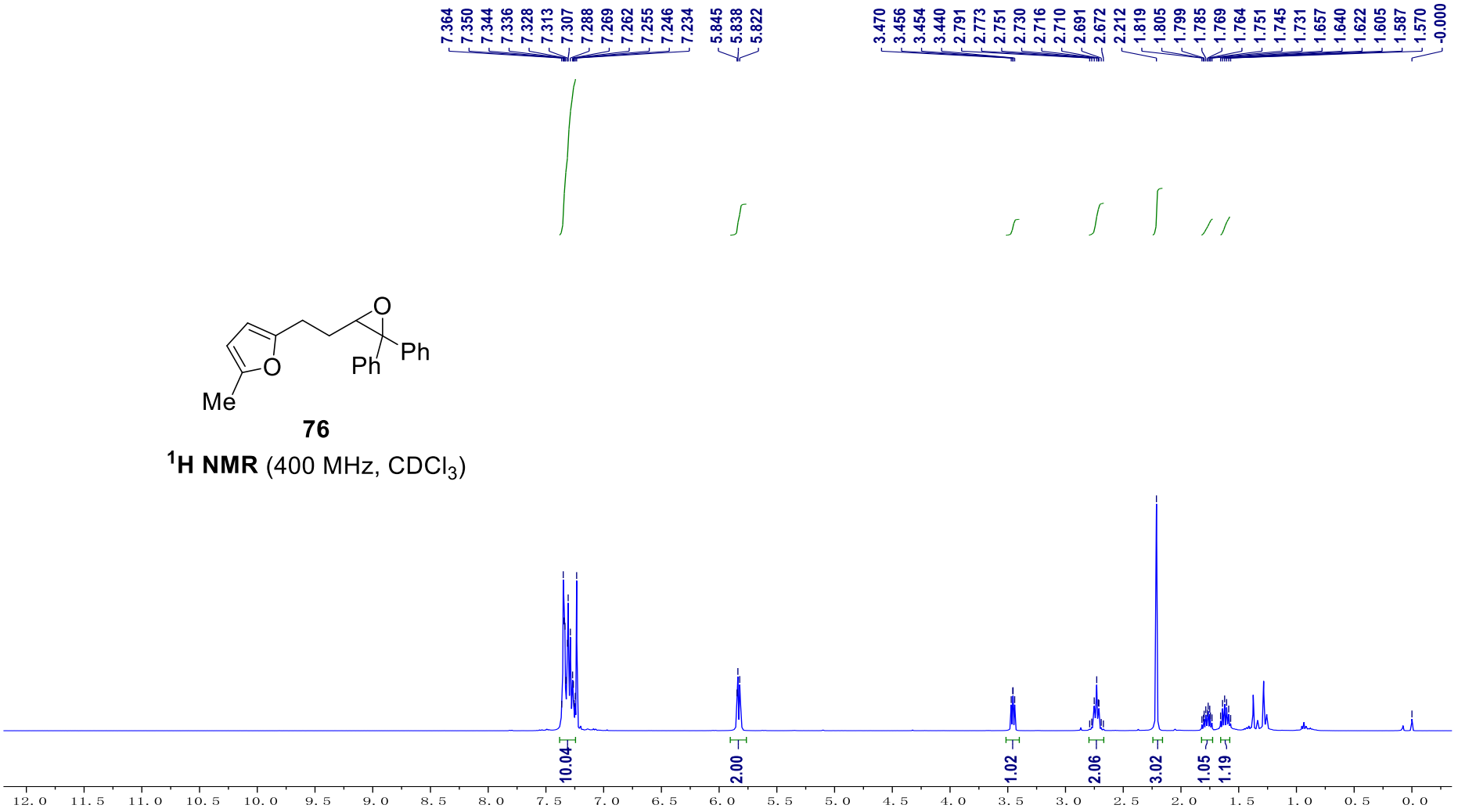
75

^{1}H NMR (300 MHz, $CDCl_3$)



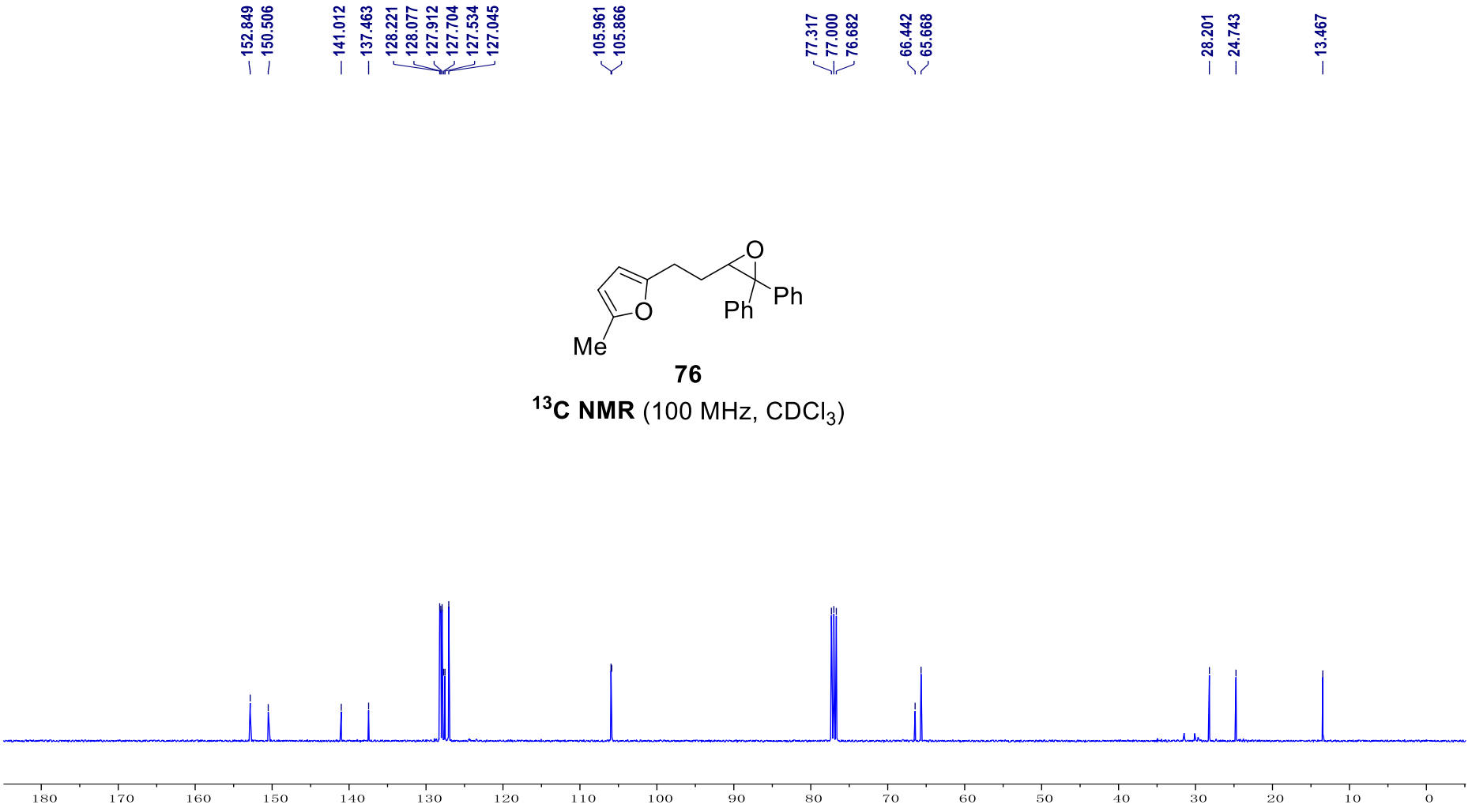
75

^{13}C NMR (75 MHz, $CDCl_3$)



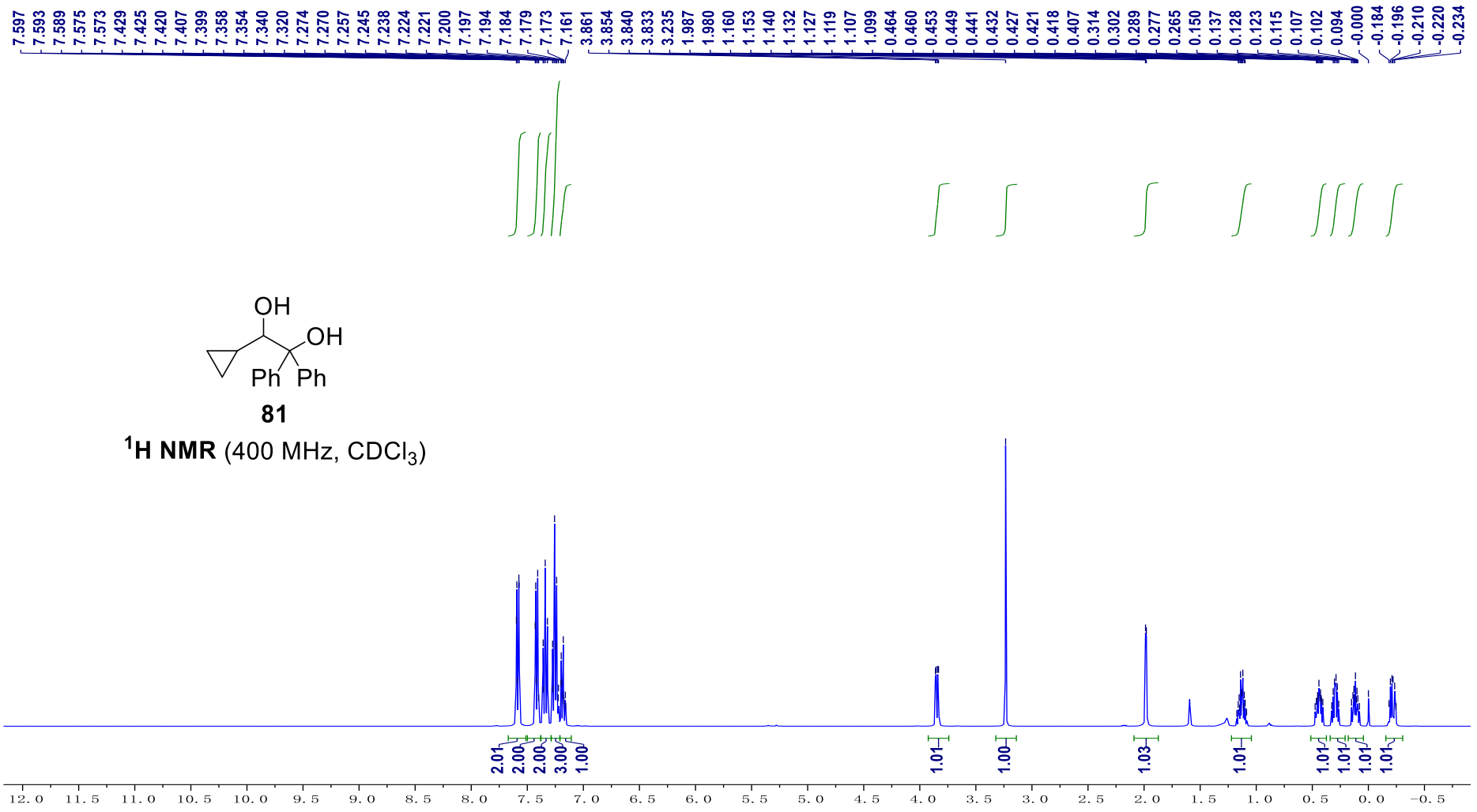
76

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



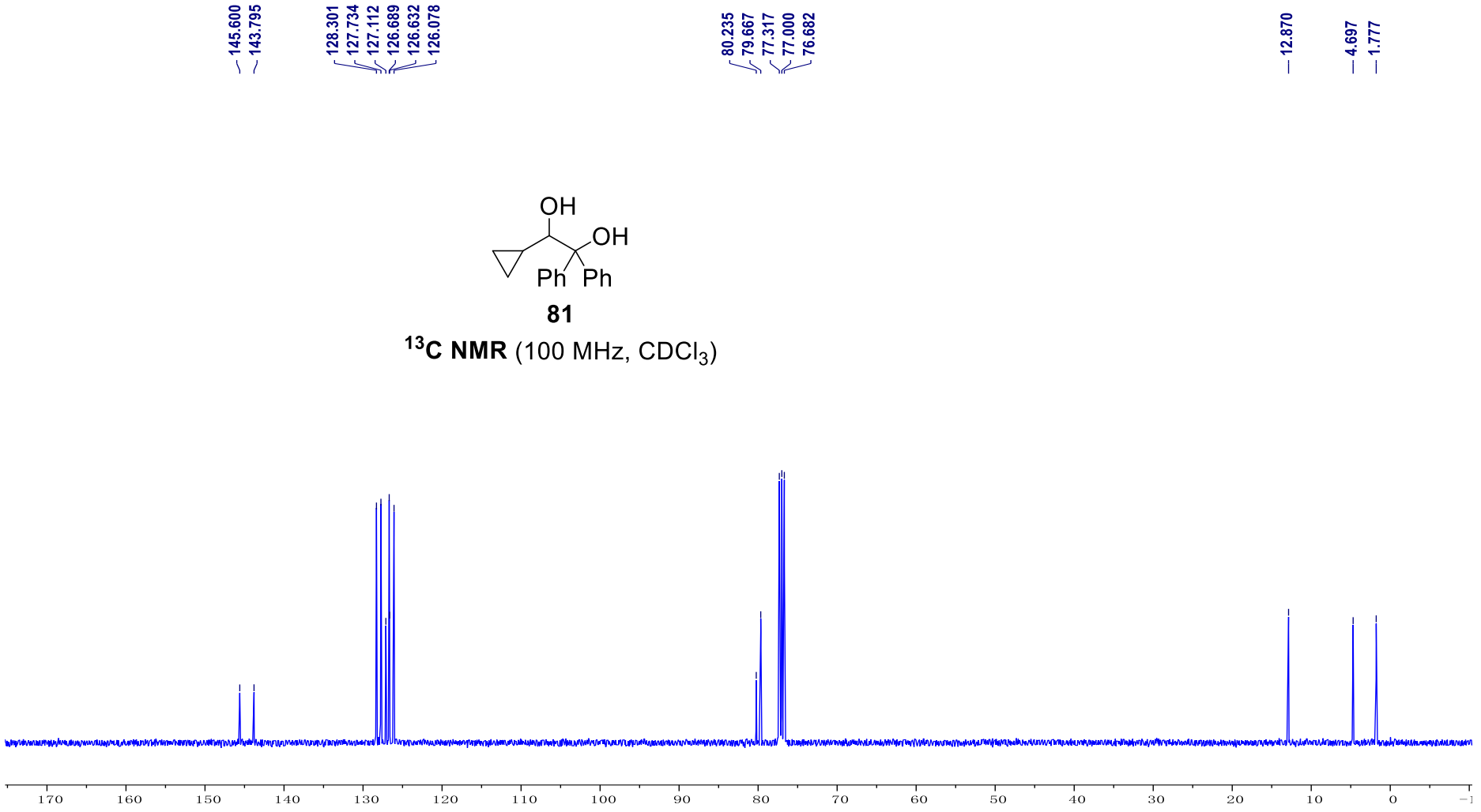
76

^{13}C NMR (100 MHz, $CDCl_3$)



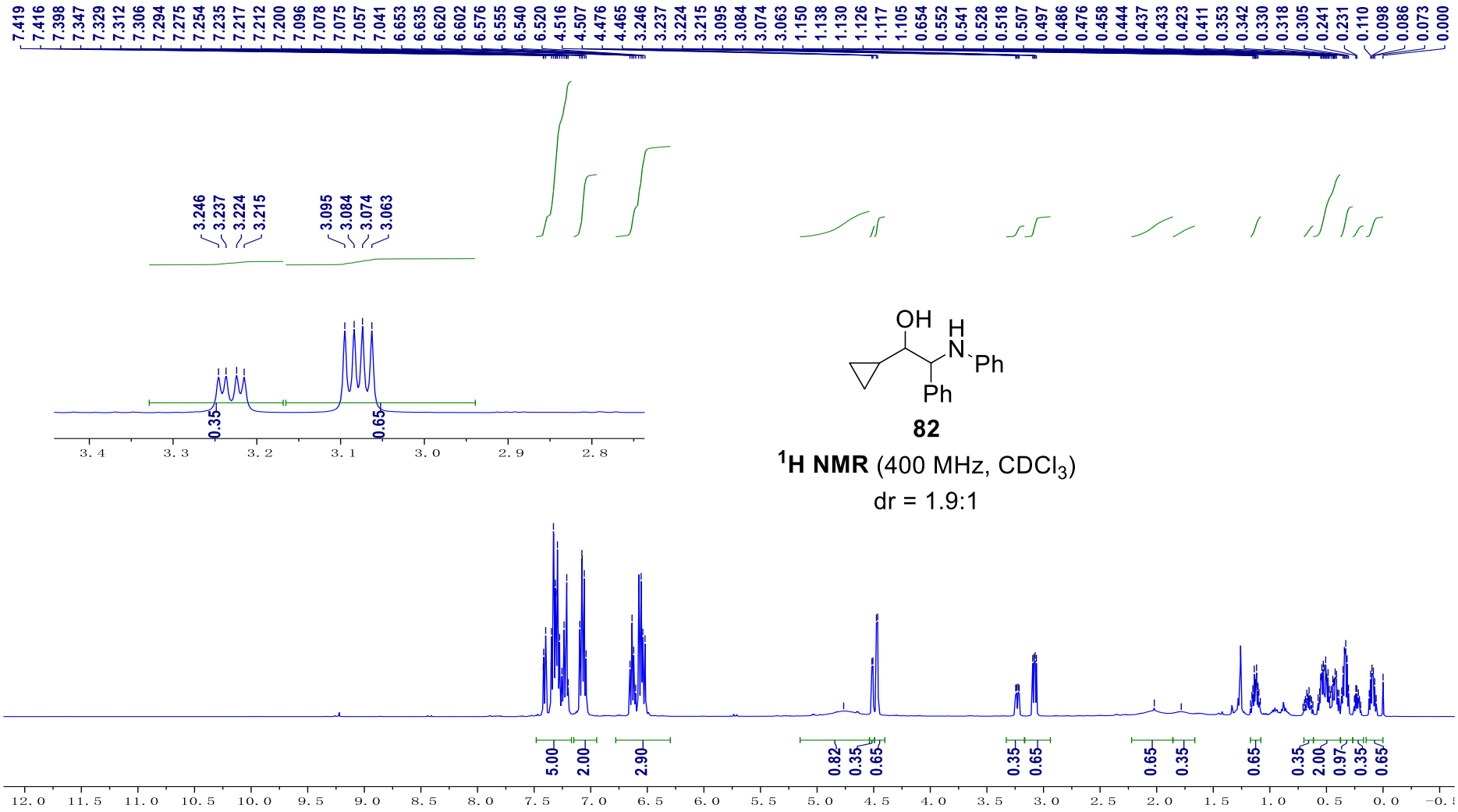
81

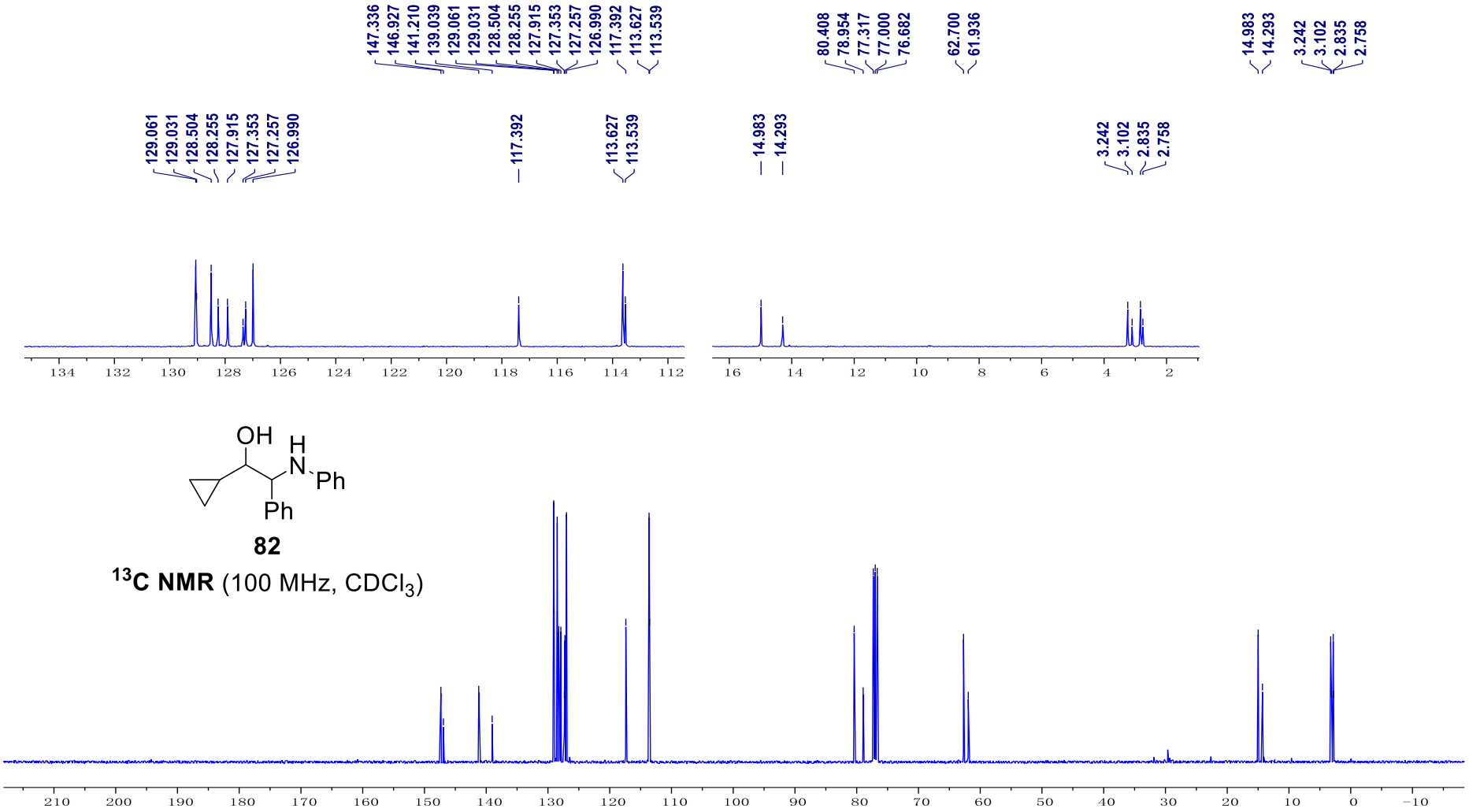
^{1}H NMR (400 MHz, $CDCl_3$)



81

^{13}C NMR (100 MHz, $CDCl_3$)


7.419 7.416 7.398 7.347 7.329 7.312 7.306 7.294 7.275 7.254 7.235 7.217 7.212 7.200 7.096 7.078 7.075 7.057 7.041 6.653 6.635 6.620 6.602 6.576 6.555 6.540 6.520 4.516 4.507 4.476 4.465 3.246 3.237 3.224 3.215 3.095 3.084 3.074 3.063 1.150 1.138 1.130 1.126 1.117 1.105 0.654 0.552 0.541 0.528 0.518 0.507 0.497 0.486 0.476 0.458 0.444 0.437 0.433 0.423 0.411 0.353 0.342 0.330 0.318 0.305 0.241 0.231 0.110 0.098 0.086 0.073 0.000
3.246 3.237 3.224 3.215
3.095 3.084 3.074 3.063
0.35
0.65
3.4 3.3 3.2 3.1 3.0 2.9 2.8
OH
H
N
Ph
Ph
82
1H NMR (400 MHz, CDCl3)
dr = 1.9:1
5.00 2.00 2.90 0.82 0.35 0.65 0.35 0.65 0.65 0.35 0.65 0.35 2.00 0.97 0.35 0.65
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


147.336
146.927
141.210
139.039
129.061
129.031
128.504
128.255
127.915
127.353
127.257
126.990
117.392
113.627
113.539
80.408
78.954
77.317
77.000
76.682
62.700
61.936
14.983
14.293
3.242
3.102
2.835
2.758
OH
H
N
Ph
Ph
82
13C NMR (100 MHz, CDCl3)