Organomediator Enabled Electrochemically Regioselective 1,2-Arylalkylation of Alkenes with Aryl and Alkyl Halides

Qian Su, Yulin Peng, Sheng Li, Jing Lei, Linhai Jing, and Pan Han*

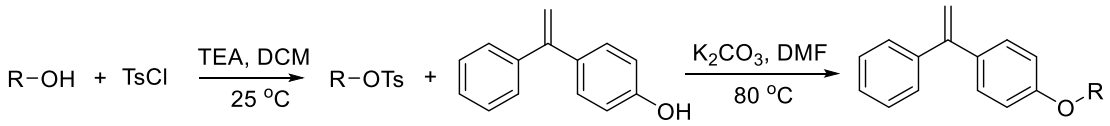
Precise Synthesis and Function Development Key Laboratory of Sichuan Province, College of Chemistry and Chemical Engineering, China West Normal University, Nanchong 637002, China; Email: hanpan_093@163.com;

Supporting Information

1 General information

All the electrochemical reactions were performed in an oven-dried undivided electrochemical cell unless otherwise noted. THF was distilled from sodium/benzophenone. Anhydrous DMF, DMA, NMP and DMSO were purchased from Adamas-beta and were further dried over molecular sieves before use. All commercial reagents were purchased from Acros Organics, Sigma-Aldrich, Alfa Aesar, Adamas-beta, Bidepharm, and Energy Chemical of the highest purity grade. ^{1}H NMR and ^{13}C NMR spectra were recorded respectively at 400 MHz and 100 MHz on a Bruker AVANCE 400 and chemical shifts are reported in δ (ppm) referenced to the TMS signal for ^{1}H NMR (0.00 ppm) and residual undeuterated solvent signal for ^{13}C NMR (77.16 ppm). HRMS was conducted on a Thermo Scientific LTQ Orbitrap XL apparatus using an electrospray (ESI) or MALDI ionization source. The following abbreviations were used to designate chemical shift mutiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. For reactions requiring heating, an oil bath was employed. All the electrochemical reactions were performed with an HY3005B DC (31 V) power supply purchased from Zhejiang Huayi Electronic Industry Co., Ltd. Carbon rod and graphite felt (GF) was purchased from Beijing Jinglong special carbon technology Co., Ltd. Platinum electrode, Al electrode, Fe electrode and Zn electrode are all purchased from Shanghai yueci Electronic Technology Co., Ltd. Magnesium plate (The purity is 99.95%) was purchased from Xintong Weiye Metal Material Sales Co., Ltd.

2 The general procedure for substrates s-2m', s-2n', s-2o'.

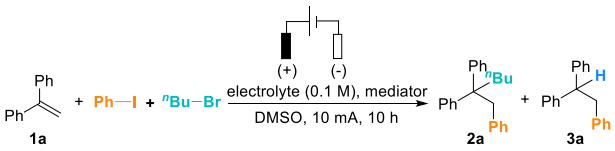


s-2m', s-2n', and **s-2o'** was prepared according to the method of reported references.[1] Tosyl chloride (6 mmol, 1.2 eq) and triethylamine (10 mmol, 2.0 eq) were added to a solution of the corresponding alcohols (5 mmol) in DCM (20 mL). The mixture was allowed to stirred overnight at 25 °C. The mixture was diluted with H_2O (30 mL), extracted with DCM (3 x 10 mL) and the combined organics were washed with H_2O (10 mL), brine (10 mL) and dried over with anhydrous Na_2SO_4. The volatile was removed under reduced pressure and the crude residue was purified by column chromatography to afford the benzenesulphonate.

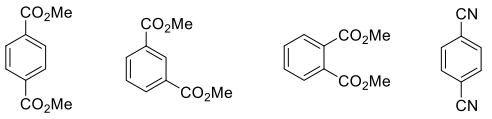
4-(1-Phenylvinyl)phenol (6 mmol, 1.2 eq) and K_2CO_3 (6 mmol, 1.2 eq) were added to a solution of benzenesulphonate (5 mmol, 1.0 eq) in DMF (20 mL). After stirring overnight at 80 °C, the mixture was diluted with H_2O (30 mL), extracted with ethyl acetate (3 x 10 mL) and the combined organics were washed with H_2O (10 mL), brine (10 mL) and dried over with anhydrous Na_2SO_4. The volatile was removed under reduced pressure and the crude residue was purified by column chromatography to afford the desired products.

3 Optimization of the reaction conditions.

Table S1. Optimization of the reaction conditions.[a]



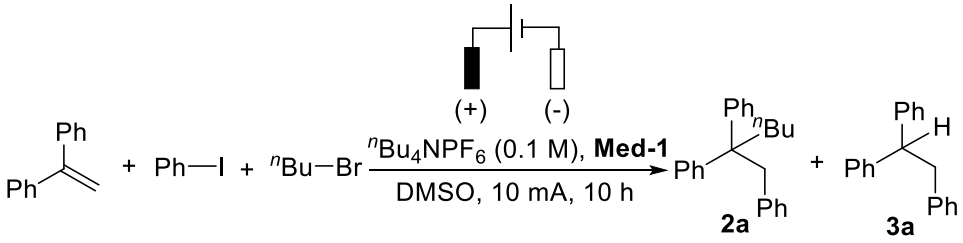
Entry	Mediator	Electrodes	Electrolyte	Yield(**2a**/**3a**)/%[b]
1	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	48/42
2	**Med-2**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	38/33
3	**Med-3**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	30/34
4	**Med-4**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	45/40
5	**Med-1**	Mg(+)/C(-)	$^{n}Bu_4NPF_6$	33/26
6	**Med-1**	Mg(+)/Ni(-)	$^{n}Bu_4NPF_6$	13/38
7	**Med-1**	Zn(+)/GF(-)	$^{n}Bu_4NPF_6$	0/45
8	**Med-1**	Fe(+)/GF(-)	$^{n}Bu_4NPF_6$	0/15
9	**Med-1**	Mg(+)/GF(-)	TBAI	46/38
10	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NBF_4$	37/43
11	**Med-1**	Mg(+)/GF(-)	Me_4NBF_4	32/66
12	**Med-1**	Mg(+)/GF(-)	$LiBF_4$	38/46
13[c]	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	0/40
14[d]	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	0/10
15[e]	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	0/10
16[f]	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	55/27
17[g]	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	71/18
18	**/**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	0
19[h]	**Med-1**	Mg(+)/GF(-)	$^{n}Bu_4NPF_6$	0



[a]Reaction conditions: undivided cell, 1,1-diphenylethylene (0.2 mmol), iodobenzene (0.4 mmol), 1-bromobutane (0.4 mmol), mediator (0.2 mmol), $^{n}Bu_4NPF_6$ (0.1 M), DMSO (3 mL), 10 mA. [b]Yield determined by ^1H NMR analysis using CH_2Br_2 as the internal standard. [c]MeCN instead of DMSO. [d]DMAc instead of DMSO. [e]DMF instead of DMSO. [f]0.6 mmol $^{n}BuBr$ instead of 0.4 mmol $^{n}BuBr$. [g]0.8 mmol $^{n}BuBr$ instead of 0.4 mmol $^{n}BuBr$. [h]No electricity.

4 Detailed Reaction Condition Optimization

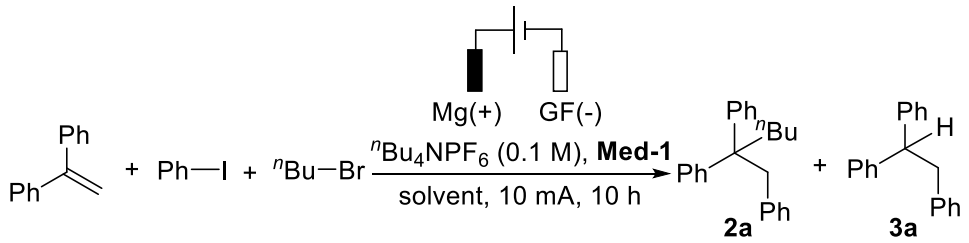
Table S2: Optimization of the electrodes.



Entry	Electrodes	Yield(**4a**/**5a**)/%
1	Mg(+)/GF(-)	48/42
2	Mg(+)/C(-)	33/26
3	Mg(+)/Ni(-)	13/38
4	Mg(+)/Pt(-)	7/23
5	Mg(+)/Ni foam(-)	10/15
6	Mg(+)/Al foam(-)	10/15
7	Mg(+)/SS	10/40
8	Zn(+)/GF(-)	0/45
9	Fe(+)/GF(-)	0/15
10	Al(+)/GF(-)	8/17

[a]Reaction conditions: undivided cell, 1,1-diphenylethylene (0.2 mmol), iodobenzene (0.4 mmol), n-butyl bromide (0.4 mmol), dimethyl terephthalate (0.2 mmol), $^{n}Bu_4NPF_6$ (0.1 M), DMSO (3 mL), 10 mA. [b]Yield determined by ^{1}H NMR analysis using CH_2Br_2 as the internal standard.

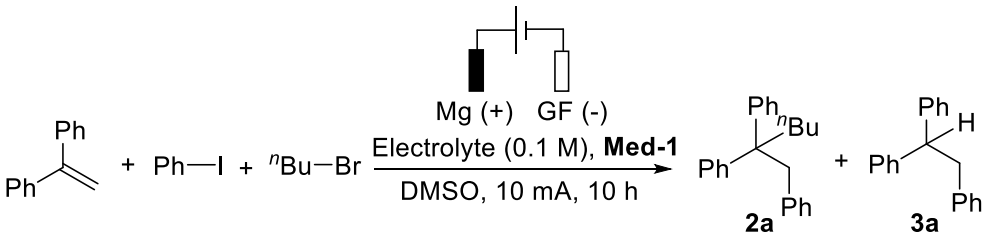
Table S3: Optimization of the solvent.



Entry	Solvent	Yield(**4a**/**5a**)/%
1	DMSO	48/42
2	MeCN	0/40
3	DMAc	0/10
4	DMF	0/10
5	NMP	0/12
6	DMPU	0/10
7	Sulfolane	0/8

[a]Reaction conditions: undivided cell, 1,1-diphenylethylene (0.2 mmol), iodobenzene (0.4 mmol), n-butyl bromide (0.4 mmol), dimethyl terephthalate (0.2 mmol), $^{n}Bu_4NPF_6$ (0.1 M), Solvent (3 mL), 10 mA. [b]Yield determined by ^{1}H NMR analysis using CH_2Br_2 as the internal standard.

Table S4: Optimization of the Electrolyte.

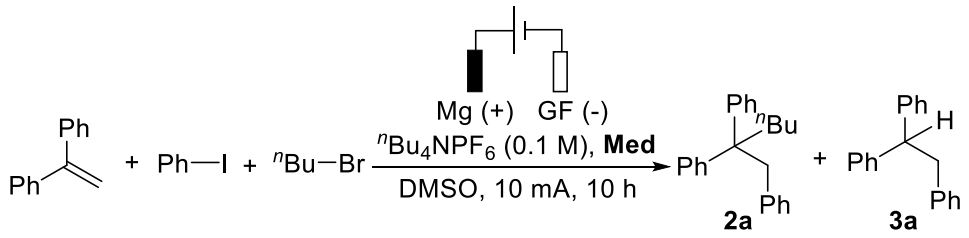


Entry	Electrolyte	Yield(**4a**/**5a**)/%
1	$^{n}Bu_4NPF_6$	48/42
2	TBAI	46/38

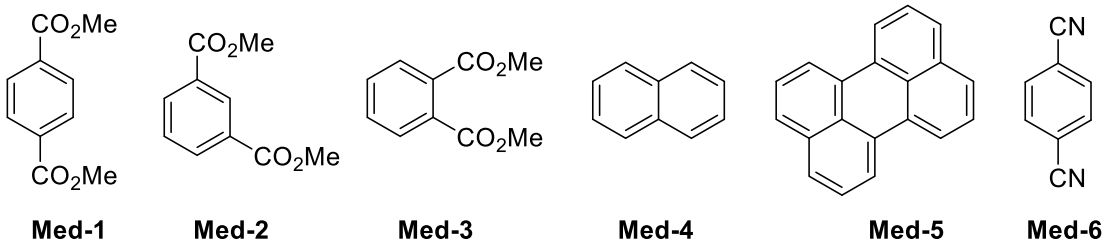
3	$^{n}Bu_4NBF_4$	37/43
4	Me_4NBF_4	32/66
5	$LiBF_4$	38/46
6	$^{n}Bu_4NClO_4$	24/38
7	TBAB	36/26

[a]Reaction conditions: undivided cell, 1,1-diphenylethylene (0.2 mmol), iodobenzene (0.4 mmol), n-butyl bromide (0.4 mmol), dimethyl terephthalate (0.2 mmol), electrolyte (0.1 M), DMSO (3 mL), 10 mA. [b]Yield determined by ^{1}H NMR analysis using CH_2Br_2 as the internal standard.

Table S5: Optimization of the mediator

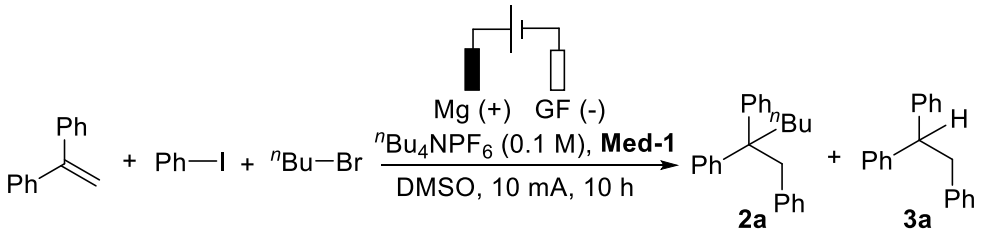


Entry	Mediator	Yield(**4a**/**5a**)/%
1	**Med-1**	48/42
2	**Med-2**	38/33
3	**Med-3**	30/34
4	**Med-4**	0/0
5	**Med-5**	35/23
6	**Med-6**	45/40



[a]Reaction conditions: undivided cell, 1,1-diphenylethylene (0.2 mmol), iodobenzene (0.4 mmol), n-butyl bromide (0.4 mmol), mediator (0.2 mmol), $^{n}Bu_4NPF_6$ (0.1 M), DMSO (3 mL), 10 mA. [b]Yield determined by ^{1}H NMR analysis using CH_2Br_2 as the internal standard.

Table S6: Optimization of equivalent of n-butyl bromide

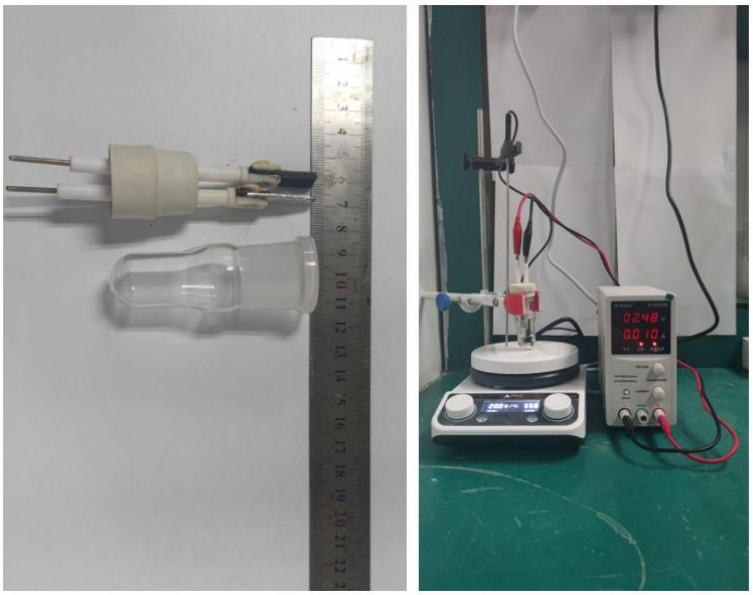


Entry	The equivalent of n-butyl bromide	Yield(**4a**/**5a**)/%
1	2.0 eq	48/42

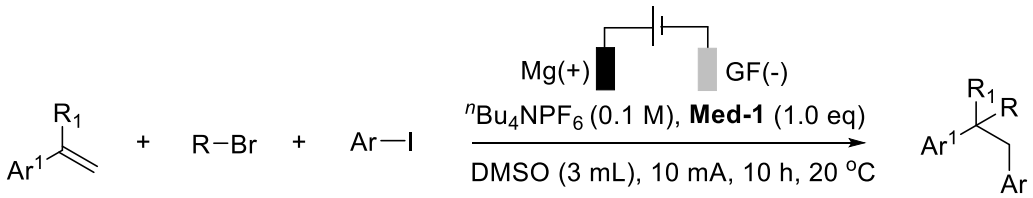
2	3.0 eq	55/27
3	4.0 eq	71/18

[a]Reaction conditions: undivided cell, 1,1-diphenylethylene (0.2 mmol), iodobenzene (0.4 mmol), n-butyl bromide, dimethyl terephthalate (0.2 mmol), $^{n}Bu_4NPF_6$ (0.1 M), DMSO (3 mL), 10 mA. [b]Yield determined by ^1H NMR analysis using CH_2Br_2 as the internal standard.

5 The setups of the electrochemical 1,2-arylalkylation of alkenes.

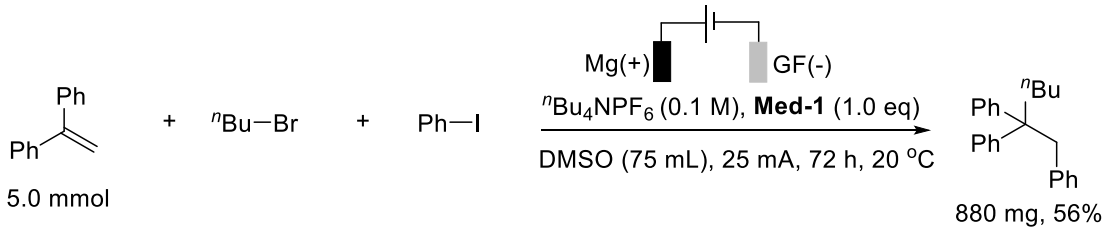


6 General procedure for the electrochemical 1,2-arylalkylation of alkenes.



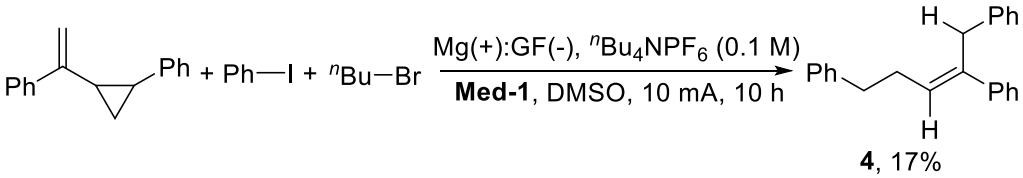
The electrolysis was conducted in an undivided cell equipped with an Mg anode (15 mm x 10 mm x 2 mm) and a graphite felt cathode (15 mm x 10 mm x 3 mm) with a distance of about 0.8 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 10 mm. To a 25 mL oven-dried undivided electrochemical cell were added $^{n}Bu_4NPF_6$ (116 mg, 0.1 M), dimethyl terephthalate (0.2 mmol, 1.0 eq), DMSO (3 mL), aryl iodides (0.4 mmol, 2.0 eq), alkyl bromide (0.8 mmol, 4.0 eq), alkenes (0.2 mmol, 1.0 eq) in the glove box. Then, seal the reaction cell and remove it from the glove box. The reaction mixture was electrolyzed under a constant current of 10 mA at 20 °C until the complete consumption of the starting materials (monitored by TLC). The mixture was diluted with H_2O, extracted with ethyl acetate (3 x 10 mL) and the combined organics were washed with H_2O (10 mL), 1 M HCl (10 mL), brine (10 mL) and dried over with anhydrous Na_2SO_4. The volatile was removed under reduced pressure and the crude residue was purified by preparative thin-layer chromatography to afford the desired products (PE/EA as eluent).

7 The procedure for the scale-up experiment.



The electrolysis was conducted in an undivided cell equipped with an Mg anode (50 mm x 30 mm x 2 mm) and a graphite felt cathode (50 mm x 30 mm x 3 mm) with a distance of about 2 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 35 mm. To a 150 mL oven-dried undivided electrochemical cell were added $^{n}Bu_4NPF_6$ (4.84 g, 12.5 mmol), dimethyl terephthalate (971 mg, 5.0 mmol), DMSO (75 mL), iodobenzene (2.04 g, 10.0 mmol), 1-bromobutane (2.7 g, 20.0 mmol), ethene-1,1-diyldibenzene (0.88 mL, 5.0 mmol) in the glove box. Then, seal the reaction cell and remove it from the glove box. The reaction mixture was electrolyzed under a constant current of 25 mA at 20 °C until the complete consumption of the starting materials (monitored by TLC). The mixture was diluted with H_2O, extracted with ethyl acetate (3 x 100 mL) and the combined organics were washed with H_2O (30 mL), 1 M HCl (30 mL), brine (30 mL) and dried over with anhydrous Na_2SO_4. The volatile was removed under reduced pressure and the crude residue was purified by column chromatography to afford the desired product (880 mg, 56% yield).

8 Radical clock experiment.



The electrolysis was conducted in an undivided cell equipped with an Mg anode (15 mm x 10 mm x 2 mm) and a graphite felt cathode (15 mm x 10 mm x 3 mm) with a distance of about 0.8 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 10 mm. To a 25 mL oven-dried undivided electrochemical cell were added $^{n}Bu_4NPF_6$ (116 mg, 0.1 M), dimethyl terephthalate (0.2 mmol, 1.0 eq), DMSO (3 mL), iodobenzene (0.4 mmol, 2.0 eq), alkyl bromide (0.8 mmol, 4.0 eq), (1-(2-phenylcyclopropyl)vinyl)benzene (0.2 mmol, 1.0 eq) in the glove box. Then, seal the reaction cell and remove it from the glove box. The reaction mixture was electrolyzed under a constant current of 10 mA at 20 °C until the complete consumption of the starting materials (monitored by TLC). The mixture was diluted with H_2O, extracted with ethyl acetate (3 x 10 mL) and the combined organics were washed with H_2O (10 mL), 1 M HCl (10 mL), brine (10 mL) and dried over with anhydrous Na_2SO_4. The volatile was removed under reduced pressure and the crude residue was purified by preparative thin-layer chromatography to afford the desired products (PE/EA as eluent). **pent-2-ene-1,2,5-triyltribenzene** Isolated yield: Colorless oil, (eluent: petroleum ether). 17% (10 mg), ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 3H), 7.26 – 7.25 (m, 1H), 7.23 – 7.08 (m, 11H), 6.00 (t, J = 7.2 Hz, 1H), 3.82 (s, 2H), 2.77 (t, J = 7.6 Hz, 2H), 2.57 (dd, J = 15.2, 8 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9, 141.8, 139.7, 138.1, 129.9,

128.5, 128.4, 128.2, 128.2, 126.7, 126.3, 125.9, 125.8, 35.9, 35.8, 31.0 ppm.

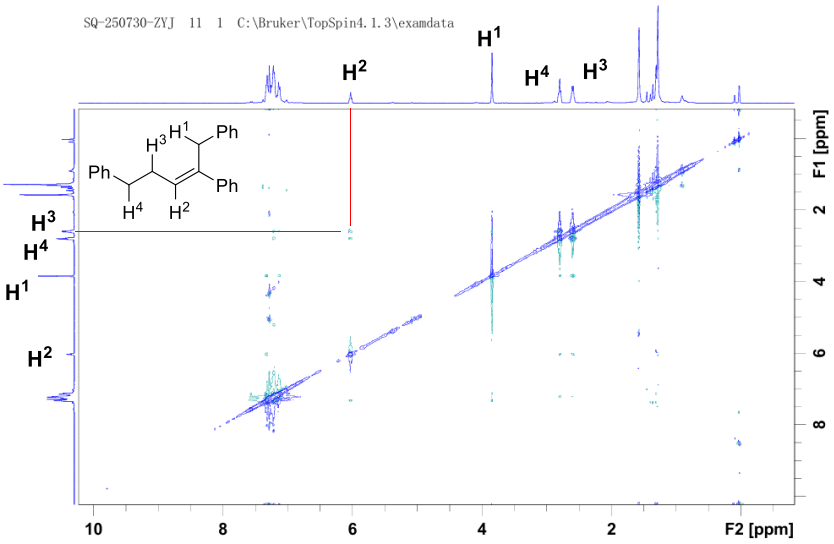
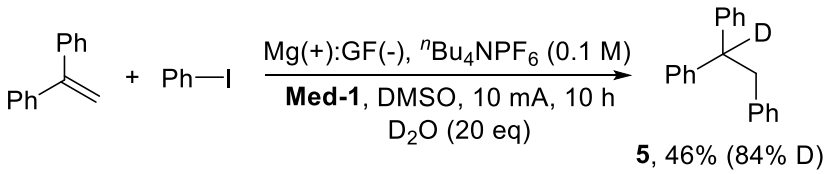


Figure S1 NOSEY spectra of compound **3**

9 General procedure for the deuteration experiment.

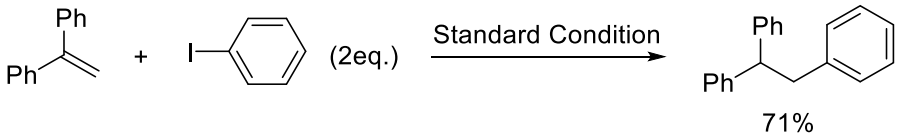


The electrolysis was conducted in an undivided cell equipped with an Mg anode (15 mm x 10 mm x 2 mm) and a graphite felt cathode (15 mm x 10 mm x 3 mm) with a distance of about 0.8 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 10 mm. To a 25 mL oven-dried undivided electrochemical cell were added $^{n}Bu_4NPF_6$ (116 mg, 0.1 M), dimethyl terephthalate (0.2 mmol, 1.0 eq), DMSO (3 mL), iodobenzene (0.4 mmol, 2.0 eq), D_2O (4.0 mmol, 20.0 eq), alkenes (0.2 mmol, 1.0 eq) in the glove box. Then, seal the reaction cell and remove it from the glove box. The reaction mixture was electrolyzed under a constant current of 10 mA at 20 °C until the complete consumption of the starting materials (monitored by TLC). The mixture was diluted with H_2O, extracted with ethyl acetate (3 x 10 mL) and the combined organics were washed with H_2O (10 mL), 1 M HCl (10 mL), brine (10 mL) and dried over with anhydrous Na_2SO_4. The volatile was removed under reduced pressure and the crude residue was purified by preparative thin-layer chromatography to afford the desired products (PE/EA as eluent). **(ethane-1,1,2-triyl-1-*d*)tribenzene** Isolated yield: Colorless oil, (eluent: petroleum ether). 46% (24 mg), ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 8H), 7.18 – 7.09 (m, 5H), 7.00 (d, J = 6.8 Hz, 2H), 4.23 (t, J = 7.6 Hz, 1H), 3.36 (d, J = 6.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 140.4, 129.2, 128.5, 128.2, 128.1, 126.3, 126.0, 53.2, 52.8 (t, J = 19.1 Hz), 42.1 ppm.

10 Investigating the reaction of the alkene with the same halide.

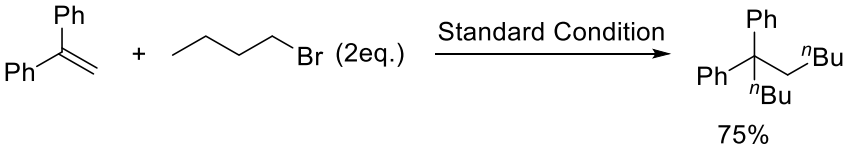
10.1 Investigating the reaction of alkene with aryl halide (Using the following reaction as a representative example).

This reaction was conducted according to the general procedure for the electrochemical 1,2-arylalkylation of alkenes.

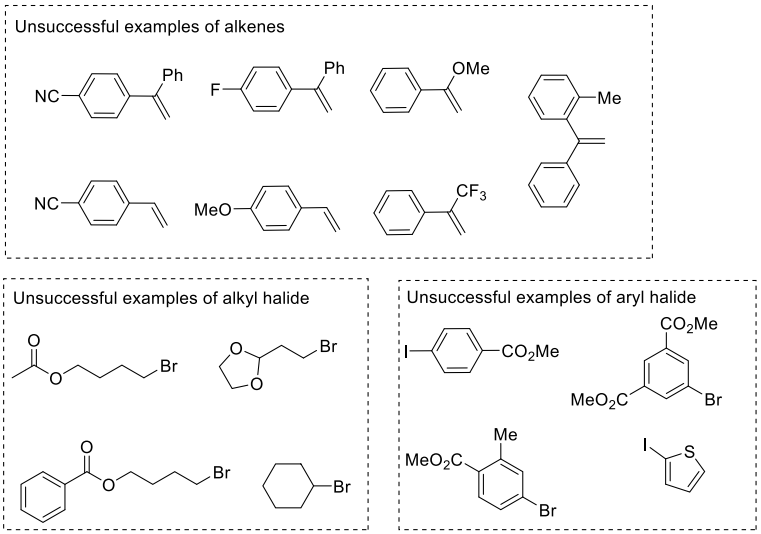


10.2 Investigating the reaction of alkene with aryl halide (Using the following reaction as a representative example).

This reaction was conducted according to the general procedure for the electrochemical 1,2-arylalkylation of alkenes. The similar electrochemical dialkylation of alkenes has been documented in literature.[2]



11 Unsuccessful examples.



12 Cyclic voltammetry experiments.

CV plotting convention is IUPAC. Electrochemical studies were carried out with a CHI600D electrochemical workstation. All cyclic voltammograms were measured at 25 ºC using an Ag/Ag^+ reference electrode, a platinum (Pt) wire counter electrode and a glassy carbon working electrode (3 mm-diameter, disc-electrode). The measurements were performed at a scan rate of 100 mV s^{-1} in DMF/^{n}Bu$_4$NPF$_6$ (0.05 M), scanning from 0 to -3.5 V in the cathodic direction. All data are measured in Ar atmosphere.

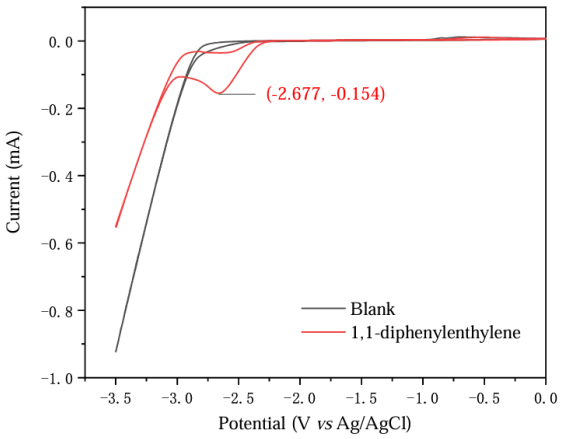


Figure S2 Cyclic voltammograms of 1,1-diphenylethylene (40 mM) in DMSO/^{n}Bu$_4$NPF$_6$ (0.1 M) under argon atmosphere.

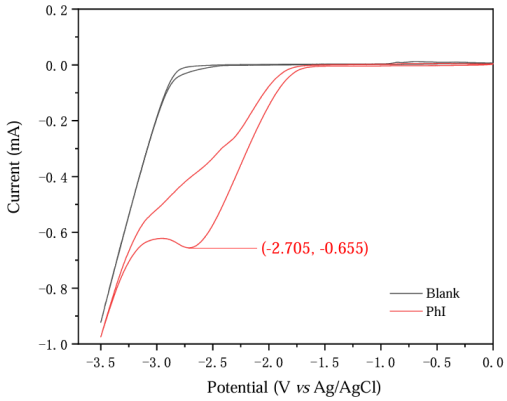


Figure S3 Cyclic voltammograms of iodobenzene (80 mM) in DMSO/^{n}Bu$_4$NPF$_6$ (0.1 M) under argon atmosphere.

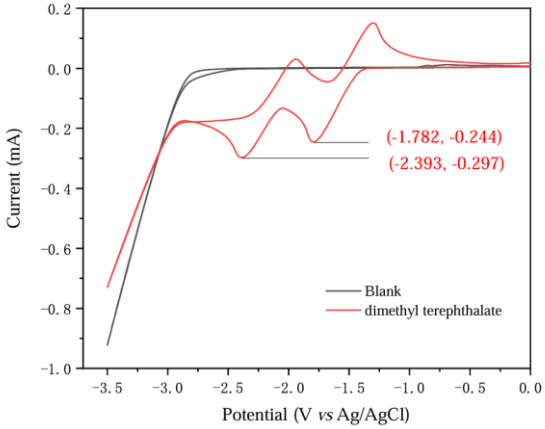


Figure S4 Cyclic voltammograms of dimethyl terephthalate (40 mM) in DMSO/^{n}Bu$_4$NPF$_6$ (0.1 M) under argon atmosphere.

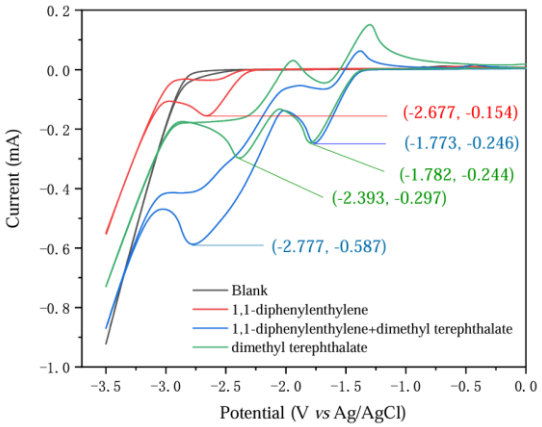


Figure S5 Cyclic voltammograms of dimethyl terephthalate (40 mM) and 1,1-diphenylethylene (40 mM) in DMSO/$^{n}Bu_4NPF_6$ (0.1 M) under argon atmosphere.

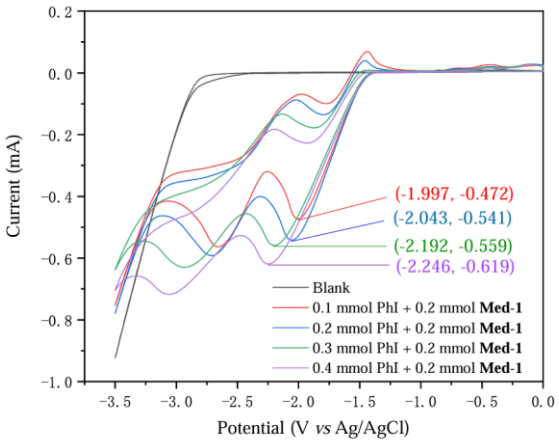


Figure S6 The cyclic voltammetry of dimethyl terephthalate (40 mM) with varying amounts of PhI in DMSO/$^{n}Bu_4NPF_6$ (0.1 M) under argon atmosphere. Dimethyl terephthalate (40 mM) + PhI (20 mM) under argon atmosphere; Dimethyl terephthalate (40 mM) + PhI (40 mM) under argon atmosphere; Dimethyl terephthalate (40 mM) + PhI (60 mM) under argon atmosphere; Dimethyl terephthalate (40 mM) + PhI (80 mM) under argon atmosphere;

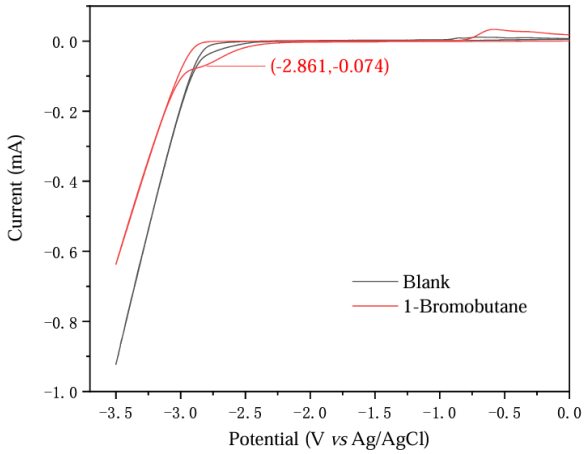


Figure S7 Cyclic voltammograms of 1-bromobutane (5 mM) in DMSO/$^{n}Bu_4NPF_6$ (0.1 M) under argon atmosphere.

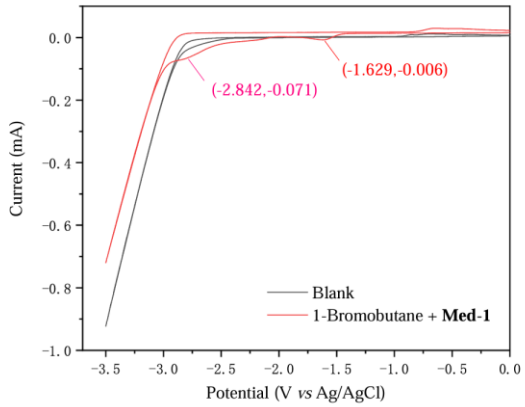


Figure S8 Cyclic voltammograms of 1-bromobutane (5 mM) + dimethyl terephthalate (1.25 mM) in DMSO/$^{n}Bu_4NPF_6$ (0.1 M) under argon atmosphere.

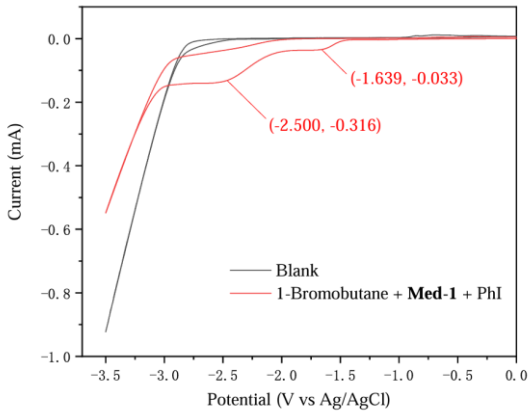
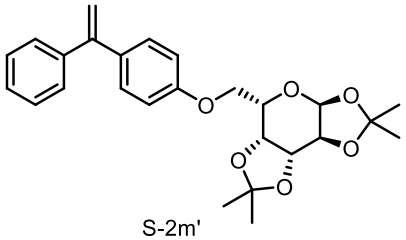


Figure S9 Cyclic voltammograms of 1-bromobutane (5 mM) + dimethyl terephthalate (1.25 mM) + iodobenzene in DMSO/$^{n}Bu_4NPF_6$ (0.1 M) under argon atmosphere.

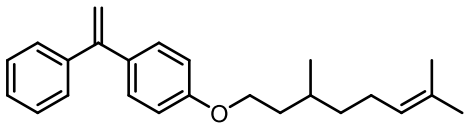
13 Characterization data.



S-2m'

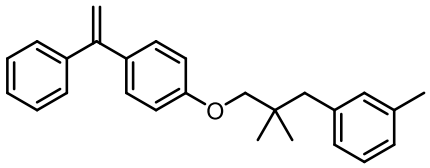
(3a*S*,5*S*,5a*R*,8a*R*,8b*S*)-2,2,7,7-tetramethyl-5-((4-(1-phenylvinyl)phenoxy)methyl)tetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran (S-2m'). Isolated yield: 31% (701 mg) (eluent: petroleum ether/EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 5H), 7.27 – 7.24 (m, 2H), 6.93 – 6.88 (m, 2H), 5.57 (d, J = 5.2 Hz, 1H), 5.38 (d, J = 1.2 Hz, 1H), 5.35 (d, J = 1.2 Hz, 1H), 4.65 (dd, J = 8.0, 2.4 Hz, 1H), 4.39 – 4.33 (m, 2H), 4.23 – 4.11 (m, 3H), 1.52 (s, 3H), 1.47 (s, 3H), 1.35 (d, J = 6.0 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 149.7, 142.0, 134.4, 129.5, 128.4, 128.3,

127.8, 114.6, 113.1, 109.6, 108.9, 96.5, 71.1, 70.8, 66.7, 66.3, 26.2, 26.2, 25.1, 24.6 ppm. HPMS (ESI) calcd for $C_{26}H_{31}O_6^+$ $[M+H]^+$ 439.2115; found 439.2117.



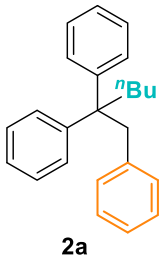
S-2n'

1-((3,7-dimethyloct-6-en-1-yl)oxy)-4-(1-phenylvinyl)benzene (S-2n'). Isolated yield: 52% (870 mg) (eluent: petroleum ether/EtOAc = 100:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 5H), 7.26 – 7.22 (m, 2H), 6.84 (d, J = 8.8 Hz, 2H), 5.38 (d, J = 0.8 Hz, 1H), 5.33 (d, J = 0.8 Hz, 1H), 5.14 – 5.07 (m, 1H), 4.04 – 3.95 (m, 2H), 2.06 – 1.94 (m, 2H), 1.90 – 1.78 (m, 1H), 1.71 – 1.66 (m, 4H), 1.62 – 1.54 (m, 4H), 1.45 – 1.33 (m, 1H), 1.27 – 1.18 (m, 1H), 0.95 (d, J = 6.4 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 149.6, 141.9, 133.8, 129.4, 128.4, 128.2, 128.1, 127.6, 124.7, 114.1, 112.8, 66.3, 37.2, 36.2, 29.6, 25.7, 25.5, 19.6, 17.7 ppm. HPMS (ESI) calcd for $C_{24}H_{31}O^+$ $[M+H]^+$ 335.2369; found 335.2377.



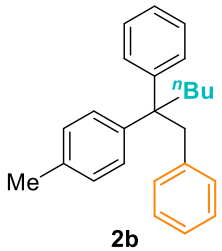
S-2o'

1-(2,2-dimethyl-3-(4-(1-phenylvinyl)phenoxy)propyl)-3-methylbenzene (S-2o'). Isolated yield: 68% (1212 mg) (eluent: petroleum ether/EtOAc = 100:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.32 (m, 5H), 7.29 (d, J = 8.4 Hz, 2H), 7.17 – 7.12 (m, 1H), 7.04 – 7.00 (m, 1H), 6.96 – 6.93 (m, 2H), 6.89 (d, J = 8.8 Hz, 2H), 5.42 (d, J = 0.8 Hz, 1H), 5.37 (s, 1H), 3.57 (s, 2H), 2.70 (s, 2H), 2.29 (s, 3H), 1.04 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 149.8, 142.1, 138.6, 137.4, 133.9, 131.6, 129.5, 128.5, 128.3, 127.9, 127.8, 127.7, 126.8, 114.3, 112.9, 75.2, 44.9, 35.7, 24.9, 21.5 ppm. HPMS (ESI) calcd for $C_{26}H_{29}O^+$ $[M+H]^+$ 357.2213; found 357.2204.



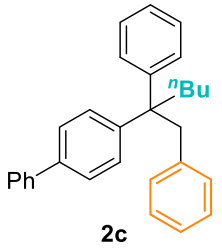
2a

hexane-1,2,2-triyltribenzene (2a).[3] Isolated yield: 71% (45 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.22 (m, 4H), 7.20 – 7.15 (m, 2H), 7.14 – 7.08 (m, 5H), 7.07 – 7.02 (m, 2H), 6.51 (d, J = 6.8 Hz, 2H), 3.40 (s, 2H), 1.95 – 1.90 (m, 2H), 1.30 – 1.23 (m, 2H), 1.13 – 1.05 (m, 2H), 0.84 (t, J = 7.2 Hz, 3H). ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.8, 138.2, 130.6, 128.5, 127.8, 127.4, 126.0, 125.8, 50.7, 43.9, 36.2, 26.7, 23.4, 14.3 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{24}H_{27}^+$ 315.2107; found 315.2113.



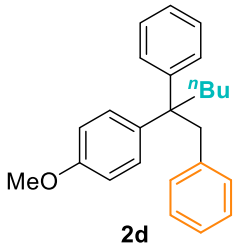
2b

(*p*-tolyl)hexane-1,2-diyl)dibenzene (2b). Isolated yield: 70% (46 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.18 (m, 2H), 7.17 – 7.12 (m, 1H), 7.11 – 7.06 (m, 3H), 7.05 – 6.97 (m, 6H), 6.51 (d, J = 6.8 Hz, 2H), 3.37 (d, J = 2.4 Hz, 2H), 2.31 (s, 3H), 1.93 – 1.85 (m, 2H), 1.28 – 1.20 (m, 2H), 1.12 – 1.03 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 145.8, 138.3, 135.1, 130.7, 128.5, 128.5, 128.3, 127.7, 127.4, 126.0, 125.7, 50.3, 43.9, 36.3, 27.1, 26.7, 23.4, 22.8, 21.1, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+H]^+$ Calcd for $C_{25}H_{29}^+$ 329.2264; found 329.2263.



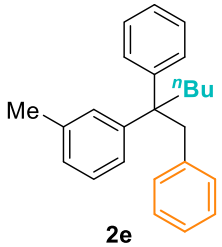
2c

(1,2-diphenylhexan-2-yl)-1,1'-biphenyl (2c). Isolated yield: 51% (46 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.58 (m, 2H), 7.50 – 7.40 (m, 4H), 7.35 – 7.29 (m, 1H), 7.27 – 7.23 (m, 2H), 7.20 – 7.13 (m, 5H), 7.11 – 7.01 (m, 3H), 6.54 (d, J = 6.8 Hz, 2H), 3.42 (s, 2H), 1.99 – 1.89 (m, 2H), 1.31 – 1.22 (m, 2H), 1.16 – 1.06 (m, 2H), 0.85 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 148.0, 140.9, 138.3, 138.1, 130.7, 128.9, 128.8, 128.5, 127.8, 127.4, 127.2, 127.0, 126.4, 126.1, 125.8, 50.5, 43.9, 36.3, 26.7, 23.4, 14.4 ppm. HRMS (ESI) *m*/*z*: $[M+Na]^+$ Calcd for $C_{30}H_{30}Na^+$ 413.2240; found 413.2232.



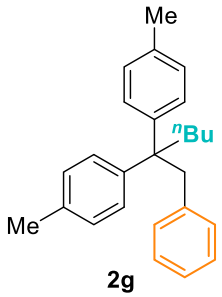
2d

(2-(4-methoxyphenyl)hexane-1,2-diyl)dibenzene(2d). Isolated yield: 72% (50 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 2H), 7.19 – 7.14 (m, 1H), 7.12 – 6.99 (m, 7H), 6.80 – 6.74 (m, 2H), 6.55 – 6.49 (m, 2H), 3.79 (s, 3H), 3.38 (d, J = 12.8 Hz, 1H), 3.34 (d, J = 12.8 Hz, 1H), 1.91 – 1.83 (m, 2H), 1.28 – 1.21 (m, 2H), 1.14 – 1.02 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.5, 149.0, 140.9, 138.3, 130.7, 129.4, 128.4, 127.8, 127.4, 126.0, 125.7, 113.1, 55.3, 50.0, 44.0, 36.4, 26.8, 23.3, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+H]^+$ Calcd for $C_{25}H_{29}O^+$ 345.2213; found 345.2207.



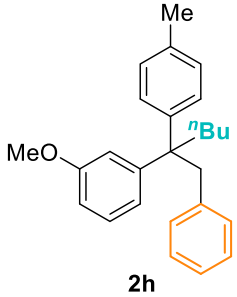
2e

(2-(*m*-tolyl)hexane-1,2-diyl)dibenzene (2e). Isolated yield: 58% (38 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.20 (m, 2H), 7.18 – 7.13 (m, 1H), 7.13 – 7.06 (m, 4H), 7.05 – 6.96 (m, 3H), 6.91 (d, J = 8.8 Hz, 2H), 6.49 (d, J = 8.4 Hz, 2H), 3.37 (d, J = 4.0 Hz, 2H), 2.27 (s, 3H), 1.93 – 1.86 (m, 2H), 1.31 – 1.20 (m, 2H), 1.13 – 1.02 (m, 2H), 0.84 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 148.7, 138.3, 137.1, 130.6, 129.1, 128.5, 127.7, 127.6, 127.3, 126.5, 126.0, 125.7, 125.6, 50.5, 43.9, 36.2, 26.7, 23.4, 21.8, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+H]^+$ Calcd for $C_{25}H_{29}^+$ 329.2264; found 329.2271.



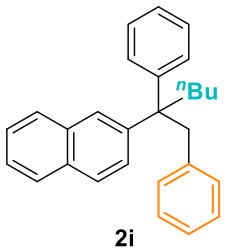
2g

4,4'-(1-phenylhexane-2,2-diyl)bis(methylbenzene) (2g). Isolated yield: 56% (37 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 – 7.09 (m, 1H), 7.08 – 7.03 (m, 6H), 7.02 – 6.98 (m, 4H), 6.55 – 6.51 (m, 2H), 3.37 (s, 2H), 2.33 (s, 6H), 1.90 – 1.84 (m, 2H), 1.30 – 1.22 (m, 2H), 1.13 – 1.04 (m, 2H), 0.85 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.9, 138.4, 135.0, 130.7, 128.5, 128.3, 127.3, 125.9, 50.0, 44.0, 36.3, 26.7, 23.3, 21.1, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+Na]^+$ Calcd for $C_{30}H_{30}Na^+$ 365.2240; found 365.2231.



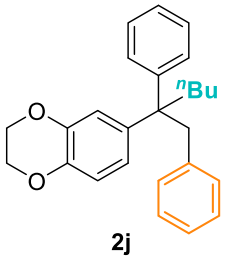
2h

1-methoxy-3-(1-phenyl-2-(p-tolyl)hexan-2-yl)benzene (2h). Isolated yield: 74% (55 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.03 (m, 6H), 7.02 – 6.98 (m, 2H), 6.75 – 6.69 (m, 3H), 6.55 (d, J = 6.8 Hz, 2H), 3.74 (s, 3H), 3.37 (s, 2H), 2.33 (s, 3H), 1.92 – 1.85 (m, 2H), 1.31 – 1.21 (m, 2H), 1.15 – 1.05 (m, 2H), 0.85 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 150.8, 145.5, 138.3, 135.1, 130.6, 128.6, 128.5, 128.2, 127.4, 126.0, 121.2, 115.0, 110.2, 55.3, 50.3, 43.8, 36.2, 26.7, 23.4, 21.1, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+H]^+$ Calcd for $C_{26}H_{31}O^+$ 359.2369; found 359.2376.



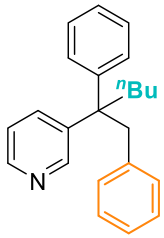
2i

2-(1,2-diphenylhexan-2-yl)naphthalene(2i). Isolated yield: 55% (40 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.66 (m, 2H), 7.62 (d, J = 1.2 Hz, 1H), 7.58 (d, J = 8.8 Hz, 1H), 7.41 – 7.32 (m, 2H), 7.15 – 7.07 (m, 3H), 7.05 – 6.96 (m, 4H), 6.95 – 6.90 (m, 2H), 6.49 – 6.37 (m, 2H), 3.48 (d, J = 12.8 Hz, 1H), 3.39 (d, J = 12.8 Hz, 1H), 2.01 – 1.88 (m, 2H), 1.30 –1.16 (m, 2H), 1.12 – 0.95 (m, 2H), 0.75 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.4, 146.1, 138.1, 133.1, 131.9, 130.6, 128.6, 128.2, 128.0, 127.9, 127.5, 127.4, 126.1, 126.0, 125.9, 125.8, 125.6, 50.8, 43.5, 35.9, 26.8, 23.4, 14.3 ppm. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{28}H_{29}^+$ 365.2264; found 365.2274.



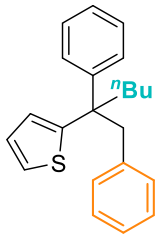
2j

6-(1,2-diphenylhexan-2-yl)-2,3-dihydrobenzo[*b*][1,4]dioxine (2j). Isolated yield: 54% (40 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.20 m, 2H), 7.19 – 7.13 (m, 1H), 7.13 – 7.02 (m, 5H), 6.74 (d, J = 8.4 Hz, 1H), 6.66 (d, J = 2.4 Hz, 1H), 6.57 (dd, J = 8.4, 2.4 Hz, 1H), 6.53 (d, J = 6.8 Hz, 2H), 4.28 – 4.21 (m, 4H), 3.37 – 3.30 (m, 2H), 1.91 – 1.79 (m, 2H), 1.29 – 1.21 (m, 2H), 1.14 – 1.05 (m, 2H), 0.85 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 142.7, 142.4, 141.4, 138.2, 130.6, 128.4, 127.7, 127.4, 126.0, 125.7, 121.6, 117.3, 116.4, 64.5, 50.0, 43.9, 36.3, 26.7, 23.3, 14.3 ppm. HRMS (ESI) m/z: $[M+K]^+$ Calcd for $C_{26}H_{28}KO_2^+$ 411.1721; found 411.1722.



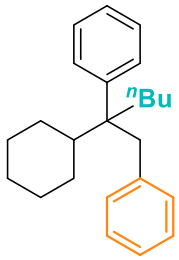
2k

2-(1,2-diphenylhexan-2-yl)pyridine (2k). Isolated yield: 58% (37 mg) (eluent: petroleum ether/EtOAc = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (dd, J = 4.8, 0.8 Hz, 1H), 7.57 – 7.49 (m, 1H), 7.26 – 7.15 (m, 3H), 7.13 – 7.01 (m, 7H), 6.60 – 6.49 (m, 2H), 3.70 (d, J = 13.2 Hz, 1H), 3.47 (d, J = 13.2 Hz, 1H), 2.05 – 1.96 (m, 2H), 1.31 – 1.23 (s, 2H), 1.20 – 1.11 (m, 1H), 1.05 – 0.95 (m, 1H), 0.83 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 148.2, 147.7, 138.5, 135.8, 130.5, 128.3, 127.9, 127.5, 126.0, 126.0, 123.7, 121.0, 53.4, 43.2, 35.9, 26.7, 23.4, 14.3 ppm. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{26}N^+$ 316.2060; found 316.2063.



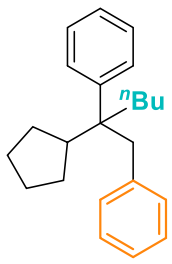
2l

2-(1,2-diphenylhexan-2-yl)thiophene (2l). Isolated yield: 48% (29 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 –7.23 (m, 2H), 7.22 – 7.16 (m, 4H), 7.15 –7.04 (m, 3H), 6.91 (dd, J = 5.2, 3.6 Hz, 1H), 6.76 (dd, J = 3.2, 0.8 Hz, 1H), 6.62 – 6.56 (m, 2H), 3.44 (d, J = 12.8 Hz, 1H), 3.40 (d, J = 12.8 Hz, 1H), 2.00 – 1.86 (m, 2H), 1.30 – 1.23 (m, 2H), 1.22 – 1.11 (m, 2H), 0.85 (t, J = 6.8 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.7, 147.7, 137.6, 130.5, 127.9, 127.8, 127.5, 126.3, 126.3, 126.0, 124.8, 123.8, 49.5, 45.6, 37.8, 26.7, 23.2, 14.3 ppm. HRMS (ESI) calcd for $C_{22}H_{24}NaS^+$ $[M+Na]^+$ 343.1491; found 343.1498.



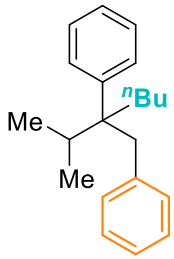
2m

(2-cyclohexylhexane-1,2-diyl)dibenzene (2m). Isolated yield: 62% (40 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 4H), 7.13 – 7.23 (m, 4H), 6.99 – 6.92 (m, 2H), 3.20 (d, J = 13.6 Hz, 1H), 3.12 (d, J = 14.0 Hz, 1H), 1.96 – 1.81 (m, 2H), 1.79 – 1.67 (m, 3H), 1.61 – 1.52 (m, 3H), 1.36 – 1.09 (m, 6H), 1.02 – 0.91 (m, 1H), 0.86 (t, J = 6.8 Hz, 3H), 0.83 – 0.64 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.8, 139.7, 130.6, 128.3, 127.8, 127.5, 125.9, 125.5, 47.7, 45.1, 40.7, 32.8, 28.8, 27.9, 27.4, 27.3, 26.8, 23.7, 14.3 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{24}H_{32}Na^+$ 343.2396; found 343.2404.



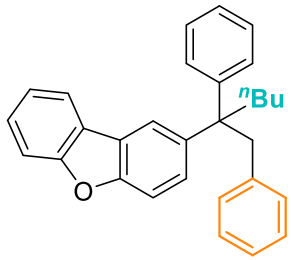
2n

(2-cyclopentylhexane-1,2-diyl)dibenzene (2n). Isolated yield: 50% (31 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 4H), 7.25 – 7.15 (m, 4H), 7.08 – 7.01 (m, 2H), 3.20 (d, J = 14.0 Hz, 1H), 3.12 (d, J = 14.0 Hz, 1H), 2.17 – 2.07 (m, 1H), 1.90 – 1.75 (m, 2H), 1.70 – 1.61 (M, 1H), 1.57 – 1.52 (m, 1H), 1.51 – 1.33 (m, 5H), 1.32 – 1.08 (m, 6H), 0.88 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.3, 139.5, 130.7, 128.8, 127.8, 127.5, 126.0, 125.6, 47.7, 47.0, 42.6, 34.6, 27.8, 27.2, 26.6, 24.9, 24.8, 23.6, 14.3 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{23}H_{30}Na^+$ 329.2240; found 329.2242.



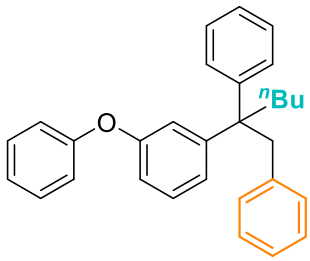
2o

(2-isopropylhexane-1,2-diyl)dibenzene (2o). Isolated yield: 41% (23 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 4H), 7.24 – 7.14 (m, 4H), 7.01 – 6.96 (m, 2H), 3.18 (d, J = 14.0 Hz, 1H), 3.13 (d, J = 14.0 Hz, 1H), 2.05 – 2.195 (m, 1H), 1.81 – 1.72 (m, 1H), 1.66 – 1.57 (m, 1H), 1.37 – 1.21 (m, 4H), 0.87 (t, J = 6.8 Hz, 3H), 0.83 (dd, J = 6.8, 2.4 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 139.6, 130.6, 128.5, 127.8, 127.5, 125.9, 125.5, 47.7, 41.2, 34.1, 33.2, 26.8, 23.7, 18.8, 17.9, 14.3 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{21}H_{28}Na^+$ 303.2083; found 303.2081.



2p

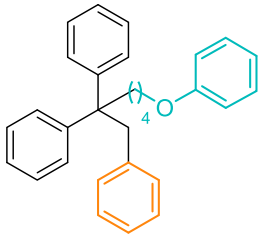
3-(1,2-diphenylhexan-2-yl)dibenzo[*b,d*]furan (2p). Isolated yield: 67% (54 mg) (eluent: petroleum ether/EtOAc = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 7.6 Hz, 1H), 7.86 (dd, J = 7.6, 0.8 Hz, 1H), 7.37 – 7.31 (m, 3H), 7.30 – 7.25 (m, 2H), 7.22 – 7.04 (m, 6H), 7.03 – 6.98 (m, 2H), 6.48 (d, J = 7.2 Hz, 2H), 3.91 (d, J = 12.8 Hz, 1H), 3.53 (d, J = 12.8 Hz, 1H), 2.37 –2.26 (m, 1H), 2.09 – 1.98 (m, 1H), 1.29 – 1.22 (m, 2H), 1.20 – 1.04 (m, 2H), 0.82 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 154.4, 147.5, 138.2, 132.8, 130.6, 127.7, 127.7, 127.4, 126.8, 126.1, 126.0, 125.7, 124.5, 124.2, 122.5, 122.4, 120.5, 119.0, 111.8, 49.3, 41.9, 35.1, 26.7, 23.3, 14.3 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{30}H_{29}O^+$ 405.2213; found 405.2223.



2q

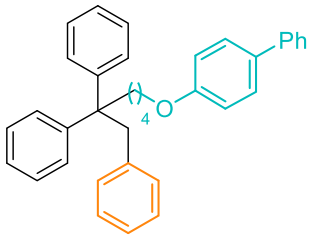
(2-(3-phenoxyphenyl)hexane-1,2-diyl)dibenzene (2q). Isolated yield: 48% (39 mg). (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.22 (m, 4H), 7.21 – 7.00 (m, 8H), 6.91 – 6.78 (m, 5H), 6.55 (d, J = 6.8 Hz, 2H), 3.40 (d, J = 12.8 Hz, 1H), 3.34 (d, J = 12.8 Hz, 1H), 1.94 – 1.83 (m, 2H), 1.30 –1.20 (m, 2H), 1.19 – 1.01 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 156.1, 151.2, 148.4, 138.0, 130.6, 129.7, 129.1, 128.4, 127.9, 127.5, 126.1, 125.9, 124.3, 122.7, 119.9, 118.0, 116.8, 50.7, 43.8, 36.2, 26.7, 23.3, 14.3 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{30}H_{30}NaO^+$ 429.2189; found 429.2199.



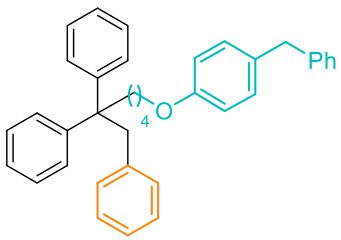
2t

(6-phenoxyhexane-1,2,2-triyl)tribenzene (2t). Isolated yield: 70% (57 mg) (eluent: petroleum ether/EtOAc = 80:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.21 (m, 6H), 7.19 – 7.14 (m, 2H), 7.13 – 7.03 (m, 5H), 6.97 – 6.90 (m, 3H), 6.88 – 6.82 (m, 2H), 6.49 (d, J = 7.2 Hz, 2H), 3.88 (t, J = 6.4 Hz, 2H), 3.40 (s, 2H), 2.01 – 1.93 (m, 2H), 1.76 – 1.68 (m, 2H), 1.34 – 1.25 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 148.6, 138.0, 130.6, 129.5, 128.4, 127.9, 127.5, 126.1, 125.9, 120.6, 114.6, 67.3, 50.7, 43.8, 36.0, 29.7, 20.9 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{30}H_{30}NaO^+$ 429.2189; found 429.2199.



2u

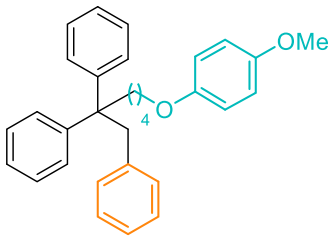
4-((5,5,6-triphenylhexyl)oxy)-1,1'-biphenyl (2u). Isolated yield: 49% (48 mg) (eluent: petroleum ether/EtOAc = 80:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 4H), 7.44 – 7.37 (m, 2H), 7.32 – 7.27 (m, 1H), 7.26 – 7.22 (m, 4H), 7.20 – 7.15 (m, 2H), 7.14 – 7.09 (m, 4H), 7.08 – 7.03 (m, 1H), 6.99 – 6.90 (m, 4H), 6.50 (d, J = 7.2 Hz, 2H), 3.92 (t, J = 6.4 Hz, 2H), 3.41 (s, 2H), 2.03 – 1.93 (m, 2H), 1.79 – 1.70 (m, 2H), 1.36 – 1.26 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 148.5, 141.0, 138.0, 133.7, 130.6, 128.9, 128.5, 128.2, 127.9, 127.5, 126.8, 126.7, 126.1, 125.90, 114.9, 67.6, 50.7, 43.8, 36.1, 29.7, 20.9 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{35}O^+$ 483.2682; found 483.2680.



2v

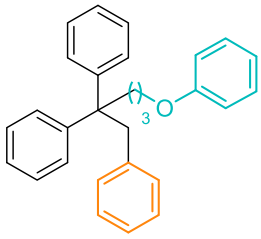
(4-benzylphenoxy)hexane-1,2,2-triyl)tribenzene (2v). Isolated yield: 42% (42 mg) (eluent: petroleum ether/EtOAc = 80:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.27 (m, 1H), 7.25 – 7.15 (m, 9H), 7.12 – 7.02 (m, 7H), 6.94 (t, J = 7.6 Hz, 2H), 6.81 – 6.75 (m, 2H), 6.49 (d, J = 7.2 Hz, 2H), 3.93 (s, 2H), 3.86 (t, J = 6.4 Hz, 2H), 3.40 (s, 2H), 2.01 – 1.93 (m, 2H), 1.76 – 1.66 (m, 2H), 1.31 – 1.26 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.8, 153.3, 148.6, 138.0, 130.6, 128.5, 127.9,

127.5, 126.1, 125.9, 115.5, 114.7, 68.1, 55.9, 50.7, 43.8, 36.1, 29.8, 20.9 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{30}H_{30}NaO^+$ 519.2658; found 519.2649.



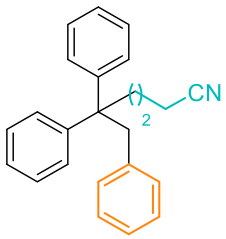
2w

(4-methoxyphenoxy)hexane-1,2,2-triyl)tribenzene (2w). Isolated yield: 67% (59 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 4H), 7.20 – 7.15 (m, 2H), 7.13 – 7.04 (m, 5H), 6.96 (t, J = 7.2 Hz, 2H), 6.83 – 6.77 (m, 4H), 6.99 – 6.93 (m, 2H), 3.83 (t, J = 6.4 Hz, 2H), 3.76 (s, 3H), 3.40 (s, 2H), 2.01 – 1.93 (m, 2H), 1.73 – 1.65 (m, 2H), 1.32 – 1.24 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.8, 153.3, 148.6, 138.0, 130.6, 128.5, 127.9, 127.5, 126.1, 125.9, 115.5, 114.7, 68.1, 55.9, 50.7, 43.9, 36.1, 29.8, 20.9 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{31}H_{33}O_2^+$ 437.2475; found 437.2480.



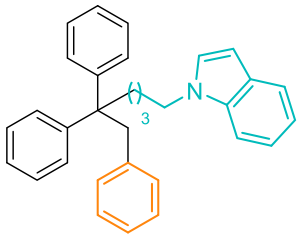
2x

(5-phenoxypentane-1,2,2-triyl)tribenzene (2x). Isolated yield: 59% (46 mg) (eluent: petroleum ether/EtOAc = 80:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.22 (m, 7H), 7.20 – 7.16 (m, 2H), 7.14 – 7.10 (m, 4H), 7.07 – 7.01 (m, 2H), 6.93 – 6.89 (m, 1H), 6.83 (d, J = 8.0 Hz, 2H), 6.55 (d, J = 6.8 Hz, 2H), 3.86 (t, J = 6.4 Hz, 2H), 3.42 (s, 2H), 2.14 – 2.08 (m, 2H), 1.65 – 1.57 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 148.3, 137.8, 130.7, 129.6, 128.5, 127.9, 127.5, 126.2, 126.0, 120.6, 114.5, 68.1, 50.5, 43.9, 32.9, 24.8 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{29}H_{28}NaO^+$ 415.2032; found 415.2024.



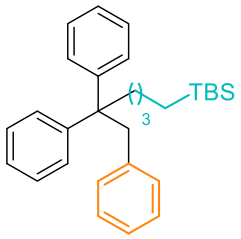
2y

5,5,6-triphenylhexanenitrile (2y). Isolated yield: 34% (23 mg) (eluent: petroleum ether/EtOAc = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 4H), 7.23 – 7.18 (m, 2H), 7.13 – 7.03 (m, 7H), 6.48 (d, J = 7.2 Hz, 2H), 3.40 (s, 2H), 2.22 (t, J = 7.2 Hz, 2H), 2.08 – 2.02 (m, 2H), 1.53 – 1.44 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 137.3, 130.5, 128.3, 128.1, 127.7, 126.4, 126.3, 119.7, 50.5, 44.1, 35.7, 21.1, 17.6 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{24}H_{24}N^+$ 326.1903; found 326.1905.



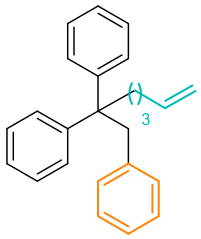
2z

1-(5,5,6-triphenylhexyl)-1*H*-indole (2z). Isolated yield: 41% (35 mg) (eluent: petroleum ether/EtOAc = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 8.0 Hz, 1H), 7.26 – 7.15 (m, 8H), 7.12 – 7.02 (m, 6H), 6.97 (d, J = 3.2 Hz, 1H), 6.95 – 6.88 (m, 2H), 6.45 (d, J = 2.8 Hz, 1H), 6.29 (d, J = 7.2 Hz, 2H), 4.00 (t, J = 6.8 Hz, 2H), 3.31 (s, 2H), 1.93 – 1.86 (m, 2H), 1.80 – 1.70 (m, 2H), 1.21 – 1.09 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.3, 137.8, 136.0, 130.5, 128.8, 128.4, 127.9, 127.8, 127.5, 126.1, 126.0, 121.4, 121.1, 119.3, 109.5, 101.0, 50.7, 46.4, 43.9, 36.2, 30.6, 22.0 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{32}H_{31}NNa^+$ 452.2349; found 452.2339.



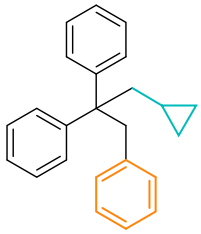
2a'

***tert*-butyldimethyl(5,5,6-triphenylhexyl)silane (2a')**. Isolated yield: 48% (41 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 4H), 7.20 – 7.15 (m, 2H), 7.12 – 7.07 (m, 5H), 7.06 – 7.01 (m, 2H), 6.51 (d, J = 7.2 Hz, 2H), 3.54 (t, J = 6.4 Hz, 2H), 3.40 (s, 2H), 1.99 – 1.88 (m, 2H), 1.52 – 1.41 (m, 2H), 1.21 – 1.09 (m, 2H), 0.86 (s, 9H), 0.00 (s, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 138.1, 130.6, 128.5, 127.8, 127.4, 126.0, 125.8, 63.1, 50.8, 43.8, 36.3, 33.5, 26.1, 20.9, 18.4, -5.1 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{30}H_{42}Si^+$ 429.2972; found 429.2965.



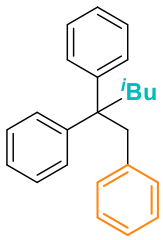
2b'

hept-6-ene-1,2,2-triyltribenzene (2b'). Isolated yield: 52% (34 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.22 (m, 4H), 7.20 – 7.15 (m, 2H), 7.13 – 7.08 (m, 5H), 7.07 – 7.01 (m, 2H), 6.50 (d, J = 6.8 Hz, 2H), 5.78 – 5.67 (m, 1H), 4.99 – 4.87 (m, 2H), 3.40 (s, 2H), 2.02 – 1.90 (m, 4H), 1.26 – 1.17 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 138.9, 138.1, 130.6, 128.5, 127.8, 127.4, 126.1, 125.8, 114.7, 50.7, 43.9, 36.0, 34.3, 24.0 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{25}H_{26}Na^+$ 349.1927; found 349.1932.



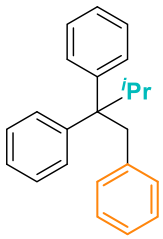
2c'

(3-cyclopropylpropane-1,2,2-triyl)tribenzene (2c'). Isolated yield: 41% (26 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.17 (m, 6H), 7.16 – 7.11 (m, 4H), 7.10 – 7.00 (m, 3H), 6.58 (d, J = 6.8 Hz, 2H), 3.52 (s, 2H), 1.92 (d, J = 6.4 Hz, 2H), 0.73 – 0.60 (m, 1H), 0.28 – 0.14 (m, 2H), -0.24 – -0.33 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 138.2, 130.9, 128.8, 127.7, 127.4, 126.0, 125.8, 51.7, 44.6, 41.9, 6.8, 5.2 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{24}H_{24}Na^+$ 335.1770; found 335.1781.



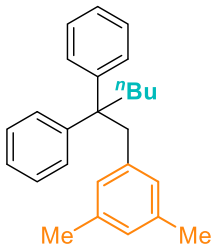
2d'

(4-methylpentane-1,2,2-triyl)tribenzene (2d'). Isolated yield: 44% (28 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.19 (m, 4H), 7.19 – 7.11 (m, 6H), 7.10 – 6.99 (m, 3H), 6.50 (d, J = 7.2 Hz, 2H), 3.45 (s, 2H), 1.86 (d, J = 5.2 Hz, 2H), 1.79 – 1.69 (m, 1H), 0.55 (d, J = 6.8 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 138.2, 131.0, 128.9, 127.7, 127.3, 126.0, 125.9, 51.1, 45.5, 45.1, 24.8, 24.5. ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{24}H_{26}Na^+$ 337.1927; found 337.1930.



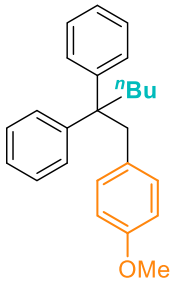
2e'

(3-methylbutane-1,2,2-triyl)tribenzene (2e'). Isolated yield: 32% (19.5 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 - 7.19 (m, 6H), 7.13 - 7.05 (m, 5H), 7.03 - 9.97 (m, 2H), 6.47 (d, J = 7.6 Hz, 2H), 3.39 (s, 2H), 2.01 - 2.53 (m, 1H), 0.90 (d, J = 6.8 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 138.3, 130.9, 130.6, 127.2, 127.0, 125.9, 125.9, 56.2, 45.5, 28.9, 18.8 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{23}H_{25}^+$ 301.1951; found 301.1959.



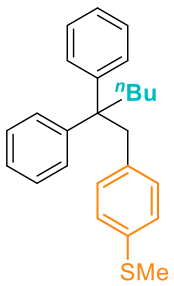
2f'

(1-(3,5-dimethylphenyl)hexane-2,2-diyl)dibenzene (2f'). Isolated yield: 56% (26 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 4H), 7.19 – 7.13 (m, 2H), 7.13 – 7.07 (m, 4H), 6.73 (s, 1H), 6.06 (s, 2H), 3.30 (s, 2H), 2.09 (s, 6H), 1.94 – 1.83 (m, 2H), 1.30 – 1.20 (m, 2H), 1.11 – 1.01 (m, 2H), 0.84 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.0, 137.9, 136.5, 128.6, 128.6, 127.7, 127.5, 125.7, 50.7, 43.7, 36.1, 26.6, 23.3, 21.3, 14.2 ppm. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{26}H_{30}Na^+$ 365.2240; found 365.2236.



2g'

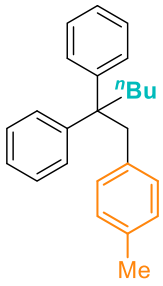
(1-(4-methoxyphenyl)hexane-2,2-diyl)dibenzene (2g'). Isolated yield: 71% (57 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 4H), 7.19 – 7.14 (m, 2H), 7.13 – 7.08 (m, 4H), 6.59 (d, J = 8.8 Hz, 2H), 6.40 (d, J = 8.4 Hz, 2H), 3.72 (s, 3H), 3.33 (s, 2H), 1.93 – 1.87 (m, 2H), 1.29 – 1.21 (m, 2H), 1.11 – 1.01 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 148.9, 131.5, 130.1, 128.5, 127.8, 125.7, 112.8, 55.2, 50.7, 43.0, 36.2, 26.7, 23.4, 14.3 ppm. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{29}O^+$ 345.2213; found 345.2210.



2h'

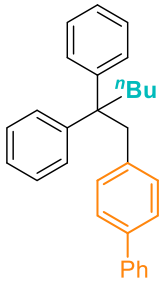
(4-(2,2-diphenylhexyl)phenyl)(methyl)sulfane (2h'). Isolated yield: 57% (41 mg) (eluent: petroleum ether/EtOAc = 200:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.14 (m, 8H), 7.14 – 7.08 (m, 4H), 6.46 (d, J = 8.4 Hz, 2H), 3.38 (s, 2H), 2.41 (s, 3H), 1.94 – 1.87 (m, 2H), 1.29 – 1.23 (m, 2H), 1.11 – 1.02 (m, 2H), 0.84 (t, J = 7.6 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 137.3,

134.2, 131.3, 128.4, 127.9, 127.0, 125.9, 50.7, 43.5, 36.2, 26.7, 23.3, 23.0, 14.3 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{25}H_{29}S^+$ 361.1984; found 361.1980.



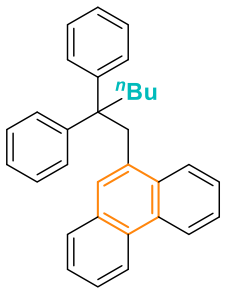
2i'

(1-(*p*-tolyl)hexane-2,2-diyl)dibenzene (2i'). Isolated yield: 66% (44 mg) (eluent: petroleum ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 4H), 7.20 – 7.15 (m, 2H), 7.14 – 7.10 (m, 4H), 6.86 (d, J = 8.0 Hz, 2H), 6.39 (d, J = 7.6 Hz, 2H), 3.37 (s, 2H), 2.26 (s, 3H), 1.96 – 1.89 (m, 2H), 1.31 – 1.21 (m, 2H), 1.14 – 1.04 (m, 2H), 0.85 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 135.4, 135.0, 130.5, 128.5, 128.13, 127.8, 125.7, 50.6, 43.4, 36.2, 26.7, 23.4, 21.1, 14.3 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{25}H_{29}^+$ 329.2264; found 329.2270.



2j'

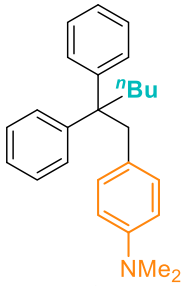
4-(2,2-diphenylhexyl)-1,1'-biphenyl (2j'). Isolated yield: 35% (28 mg) (eluent: petroleum ether/EtOAc = 150:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.51 (m, 2H), 7.42 – 7.37 (m, 2H), 7.30 – 7.22 (m, 7H), 7.20 – 7.12 (m, 6H), 6.56 (d, J = 8.4 Hz, 2H), 3.43 (s, 2H), 2.00 – 1.90 (m, 2H), 1.31 – 1.23 (m, 2H), 1.16 – 1.05 (m, 2H), 0.85 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.8, 141.0, 138.7, 137.4, 131.0, 128.8, 128.5, 127.8, 127.1, 127.0, 126.0, 125.8, 50.7, 43.6, 36.3, 26.8, 23.4, 14.3 ppm. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{30}H_{31}^+$ 391.2420; found 391.2431.



2k'

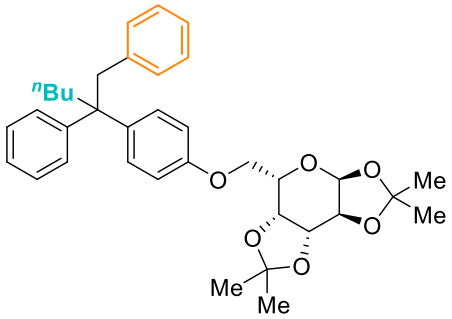
9-(2,2-diphenylhexyl)phenanthrene (2k'). Isolated yield: 44% (35 mg) (eluent: petroleum ether/EtOAc = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 – 8.60 (m, 2H), 7.66 (d, J = 8.4 Hz, 1H), 7.62 – 7.56 (m, 1H), 7.54 – 7.46 (m, 3H), 7.31 – 7.27 (m, 1H), 7.21 – 7.13 (m, 10H), 6.91 (s, 1H),

3.91 (s, 2H), 2.07 – 1.98 (m, 2H), 1.23 – 1.10 (m, 4H), 0.82 (t, J =6.8 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 132.8, 132.6, 131.23, 130.1, 129.7, 129.7, 128.9, 128.3, 127.8, 126.5, 126.2, 125.9, 125.9, 125.5, 124.7, 122.8, 122.4, 51.6, 39.5, 37.3, 27.4, 23.3, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+Na]^+$ Calcd for $C_{32}H_{30}Na^+$ 437.2240; found 437.2251.



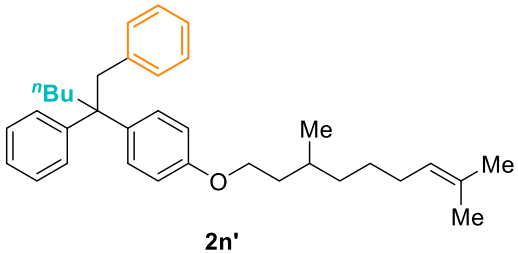
2l'

4-(2,2-diphenylhexyl)-*N*,*N*-dimethylaniline (2l'). Isolated yield: 47% (34 mg) (eluent: petroleum ether/EtOAc = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 4H), 7.20 – 7.15 (m, 2H), 7.15 – 7.10 (m, 4H), 6.46 (d, J = 8.4 Hz, 2H), 6.36 (d, J = 8.8 Hz, 2H), 3.31 (s, 2H), 2.86 (s, 6H), 1.97 – 1.89 (m, 2H), 1.29 – 1.21 (m, 2H), 1.12 – 1.02 (m, 2H), 0.84 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.2, 131.2, 128.6, 127.7, 125.6, 112.0, 50.7, 42.9, 40.8, 36.3, 26.7, 23.4, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+Na]^+$ Calcd for $C_{26}H_{31}NNa^+$ 380.2349; found 380.2349.



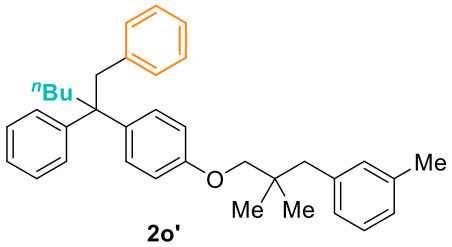
2m'

(3a*S*,5*S*,5a*R*,8a*R*,8b*S*)-5-((4-(1,2-diphenylhexan-2-yl)phenoxy)methyl)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran (2m'). Isolated yield: 54% (62 mg) (eluent: petroleum ether/EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 2H), 7.20 – 7.14 (t, J = 7.1 Hz, 1H), 7.13 – 6.98 (m, 7H), 6.83 (dd, J = 8.8, 1.6 Hz, 2H), 6.52 (d, J = 6.8 Hz, 2H), 5.59 (d, J = 5.2 Hz, 1H), 4.67 (dd, J = 8.0, 2.4 Hz, 1H), 4.43 – 4.34 (m, 2H), 4.23 – 4.09 (m, 3H), 3.41 – 3.32 (m, 2H), 1.92 – 1.83 (m, 2H), 1.50 (d, J = 12.4 Hz, 6H), 1.37 (d, J = 8.8 Hz, 6H), 1.29 – 1.22 (m, 2H), 1.15 – 1.02 (m, 2H), 0.84 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.5, 149.0, 141.2, 138.3, 130.7, 129.3, 128.4, 128.4, 127.7, 127.4, 126.0, 125.7, 114.0, 114.0, 109.6, 108.9, 96.5, 71.1, 70.8, 66.5, 66.3, 66.3, 50.0, 44.0, 36.4, 26.7, 26.2, 26.1, 25.1, 24.6, 23.3, 14.3 ppm. HRMS (ESI) *m*/*z*: $[M+Na]^+$ Calcd for $C_{36}H_{44}NaO_6^+$ 595.3030; found 595.3030.



2n'

(2-(4-((3,8-dimethylnon-7-en-1-yl)oxy)phenyl)hexane-1,2-diyl)dibenzene (2n'). Isolated yield: 52% (50 mg) (eluent: petroleum ether/EtOAc = 100:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 2H), 7.18 – 6.98 (m, 8H), 6.77 (d, J = 8.4 Hz, 2H), 6.52 (d, J = 6.8 Hz, 2H), 5.12 (t, J = 6.8 Hz, 1H), 4.04 – 3.91 (m, 2H), 3.41 – 3.32 (m, 2H), 2.11 – 1.95 (m, 2H), 1.90 – 1.58 (m, 2H), 1.76 – 1.54 (m, 10H), 1.45 – 1.35 (m, 1H), 1.27 – 1.23 (m, 2H), 1.15 – 1.03 (m, 2H), 0.96 (d, J = 6.4 Hz, 3H), 0.84 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 149.0, 140.7, 138.4, 131.4, 130.7, 129.4, 128.5, 127.8, 127.4, 126.0, 125.7, 124.9, 113.7, 66.4, 50.1, 44.1, 37.3, 36.5, 36.4, 29.7, 26.8, 25.9, 25.6, 23.4, 19.8, 17.8, 14.3 ppm. HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{34}H_{44}NaO^+$ 491.3284; found 491.3282.



2o'

(2-(4-(2,2-dimethyl-3-(*m*-tolyl)propoxy)phenyl)hexane-1,2-diyl)dibenzene (2o'). Isolated yield: 55% (54 mg) (eluent: petroleum ether/EtOAc = 100:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (d, J = 7.6 Hz, 2H), 7.21 – 7.00 (m, 10H), 6.97 – 6.92 (m, 2H), 6.80 (d, J = 8.8 Hz, 2H), 6.58 – 6.51 (m, 2H), 3.54 (s, 2H), 3.38 (s, 2H), 2.69 (s, 2H), 2.28 (s, 3H), 1.95 – 1.85 (m, 2H), 1.33 – 1.22 (m, 2H), 1.18 – 1.07 (m, 2H), 1.03 (s, 6H), 0.86 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 149.1, 140.7, 138.7, 138.4, 137.4, 131.6, 130.7, 129.4, 128.5, 127.7, 127.4, 126.8, 126.0, 125.7, 113.8, 75.2, 50.1, 44.9, 44.1, 36.5, 35.7, 26.8, 24.9, 23.4, 21.5, 14.3 ppm. HRMS (ESI) *m/z*: $[M+K]^+$ Calcd for $C_{36}H_{42}KO^+$ 529.2867; found 529.2878.

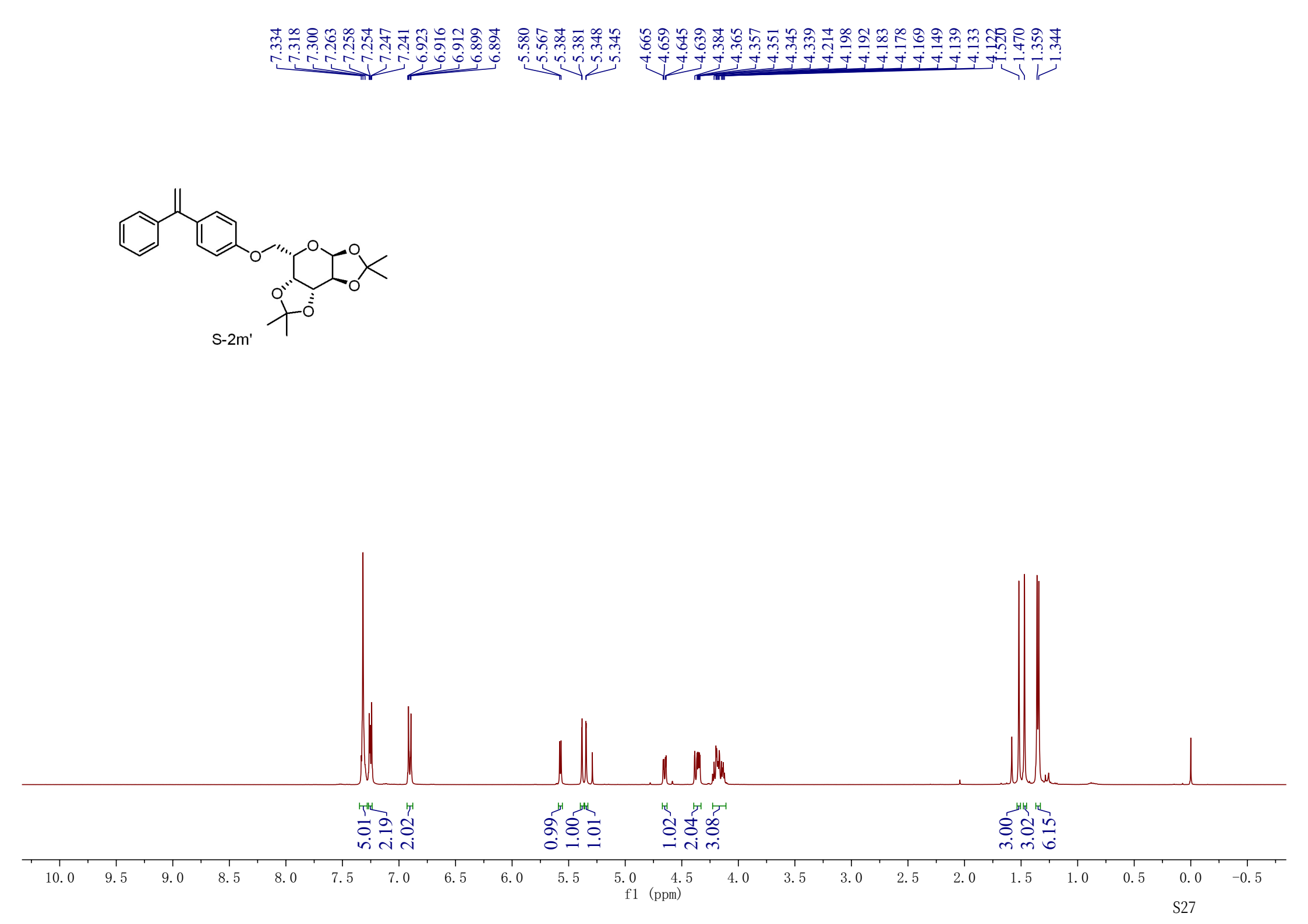
14 References.

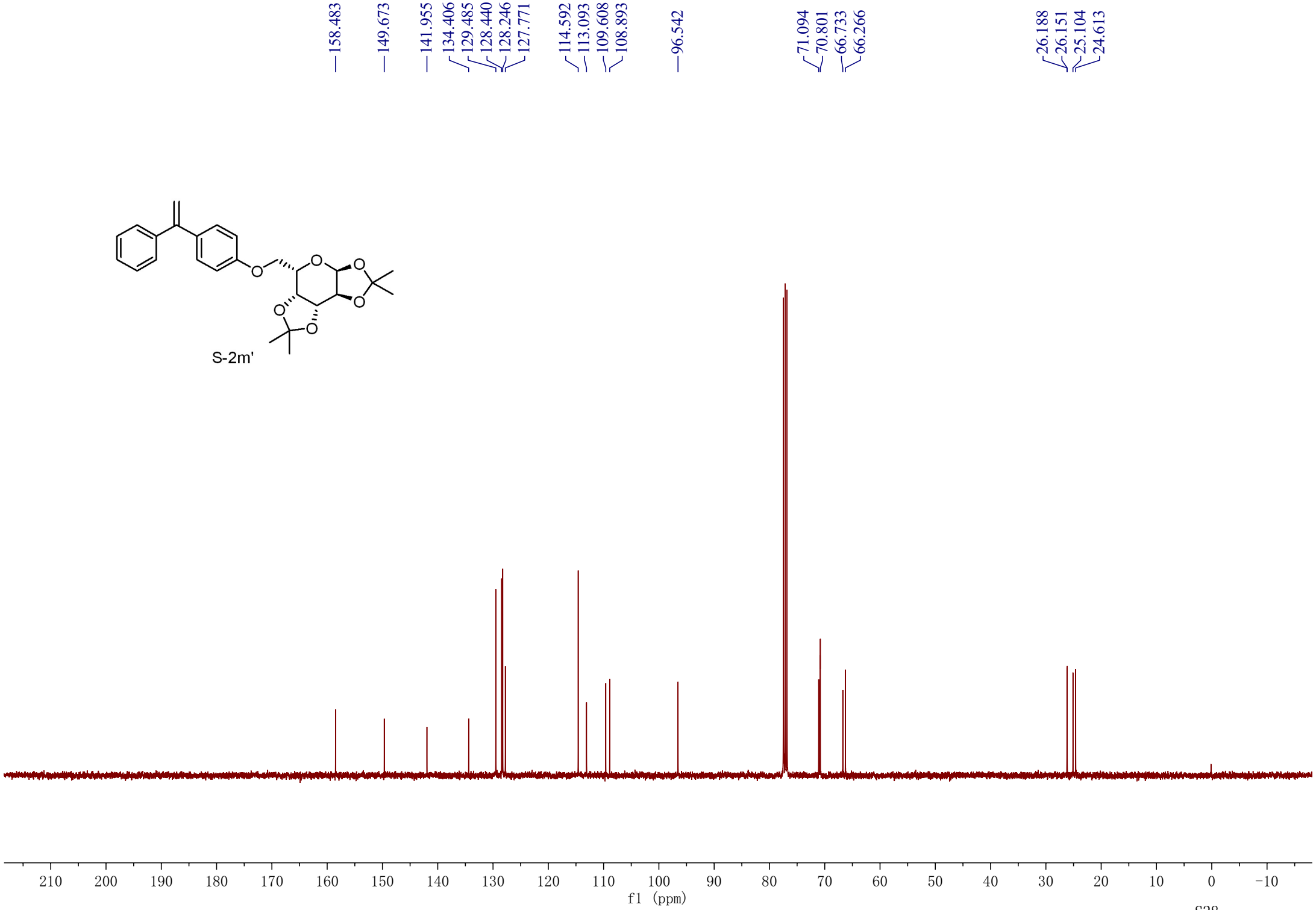
(1) (a) Hao, J.; Cheng, Y.; Ranatunga, R. J. K. U.; Senevirathne, S.; Biewer, M. C.; Nielsen, S. O.; Wang, Q.; Stefan, M. C., A Combined Experimental and Computational Study of the Substituent Effect on Micellar Behavior of γ-Substituted Thermoresponsive Amphiphilic Poly(ε-caprolactone)s. *Macromolecules* **2013,** *46*, 4829-4838; (b) Chen, W.; Dong, J.; Plate, L.; Mortenson, D. E.; Brighty, G. J.; Li, S.; Liu, Y.; Galmozzi, A.; Lee, P. S.; Hulce, J. J.; Cravatt, B. F.; Saez, E.; Powers, E. T.; Wilson, I. A.; Sharpless, K. B.; Kelly, J. W., Arylfluorosulfates Inactivate Intracellular Lipid Binding Protein(s) through Chemoselective SuFEx Reaction with a Binding Site Tyr Residue. *J. Am. Chem. Soc.* **2016,** *138*, 7353-7364.

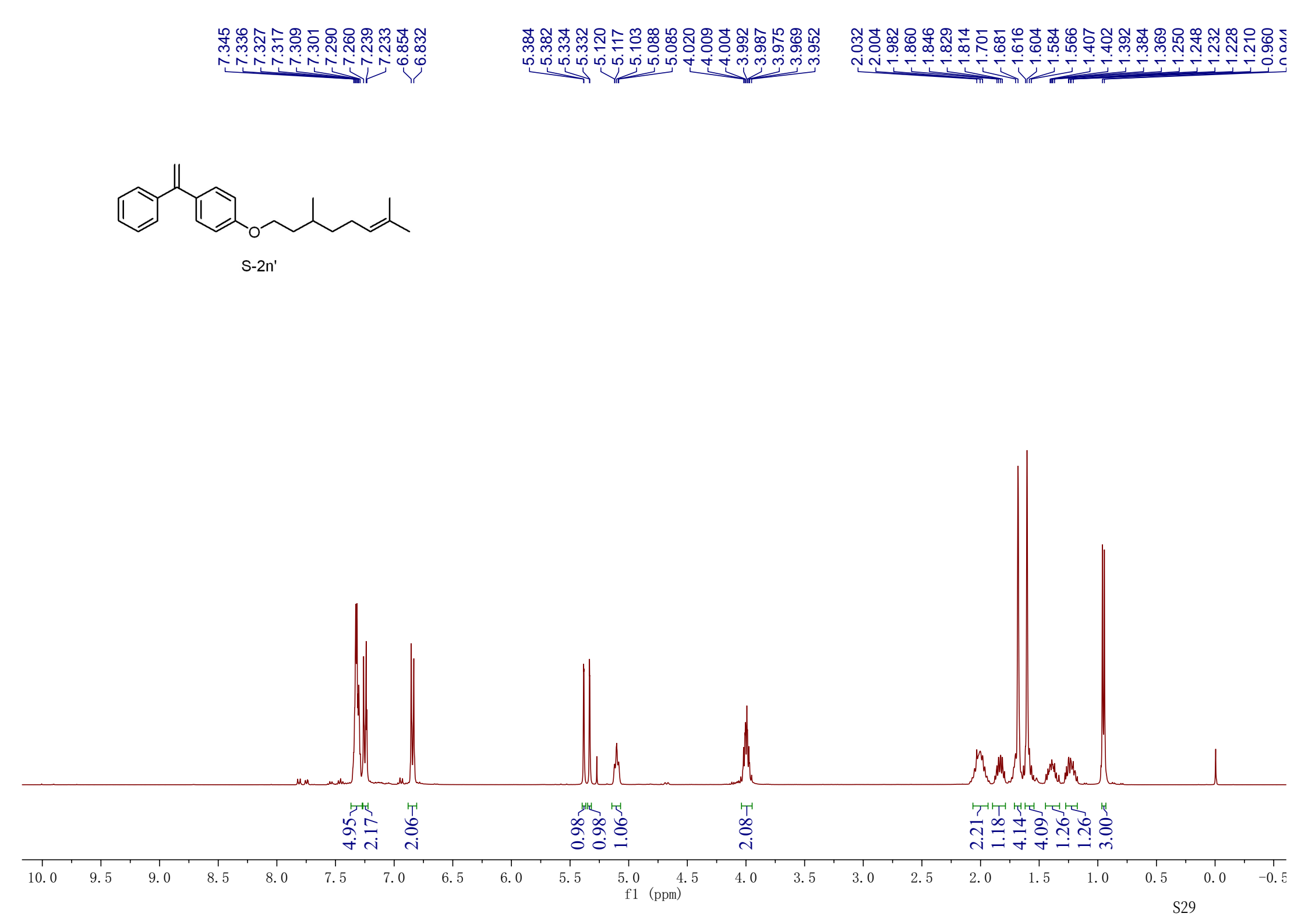
(2) Satoh, S. h.; Taguchi, T. r.; Itoh, M.; Tokuda, M., Electrochemical Vicinal Addition of Two Alkyl Groups to Phenyl Substituted Olefins and Diethyl Fumarate. *Bull. Chem. Soc. Jpn.* **1979,** *52*, 951-952.

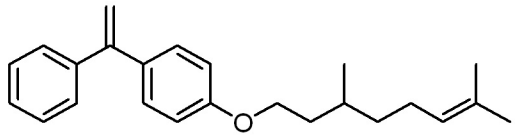
(3) Murphy, W. S.; Hauser, C. R., Alkylations of Benzhydryl-Type Alkanes and Alkenes by Means of Alkali Amides and Metals in Liquid Ammonia to Form (C6H5)2CRR'1. *J. Org. Chem.* **1966,** *31*, 1781-1784.

15 NMR Spectra of Coupled Products.


7.334
7.318
7.300
7.263
7.258
7.254
7.247
7.241
6.923
6.916
6.912
6.899
6.894
5.580
5.567
5.384
5.381
5.348
5.345
4.665
4.659
4.645
4.639
4.384
4.365
4.357
4.351
4.345
4.339
4.214
4.198
4.192
4.183
4.178
4.169
4.149
4.139
4.133
4.122
1.520
1.470
1.359
1.344
S-2m'
5.01
2.19
2.02
0.99
1.00
1.01
1.02
2.04
3.08
3.00
3.02
6.15
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

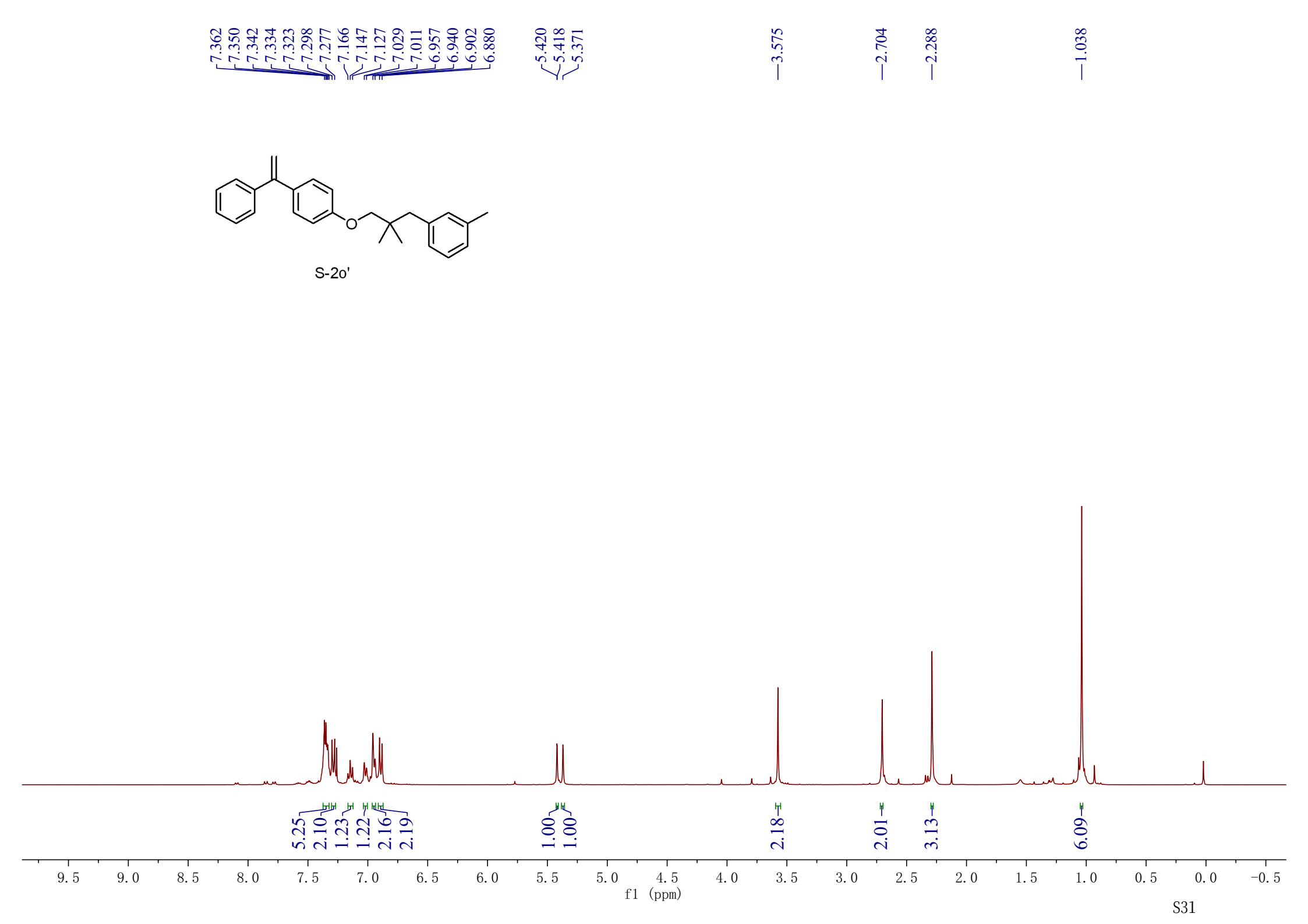

158.483
149.673
141.955
134.406
129.485
128.440
128.246
127.771
114.592
113.093
109.608
108.893
96.542
71.094
70.801
66.733
66.266
26.188
26.151
25.104
24.613
S-2m'
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

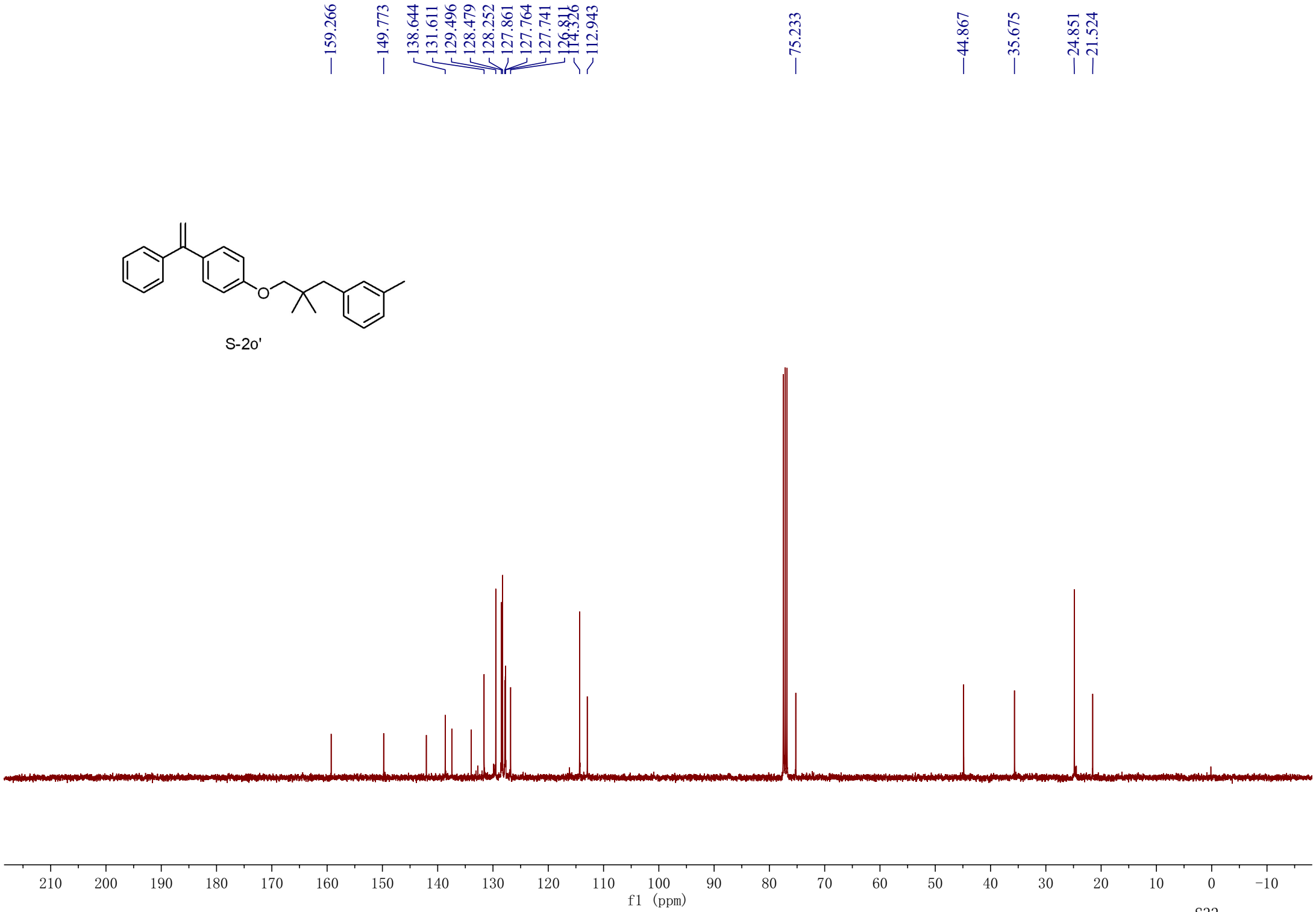

7.345
7.336
7.327
7.317
7.309
7.301
7.290
7.260
7.239
7.233
6.854
6.832
5.384
5.382
5.334
5.332
5.120
5.117
5.103
5.088
5.085
4.020
4.009
4.004
3.992
3.987
3.975
3.969
3.952
2.032
2.004
1.982
1.860
1.846
1.829
1.814
1.701
1.681
1.616
1.604
1.584
1.566
1.407
1.402
1.392
1.384
1.369
1.250
1.248
1.232
1.228
1.210
0.960
O
S-2n'
4.95
2.17
2.06
0.98
0.98
1.06
2.08
2.21
1.18
4.14
4.09
1.26
1.26
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

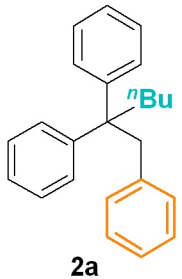


S-2n'

158.947
149.611
141.907
133.787
129.362
128.352
128.186
128.119
127.629
124.713
114.125
112.818
66.325
37.171
36.189
29.575
25.743
25.494
19.590
17.695
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

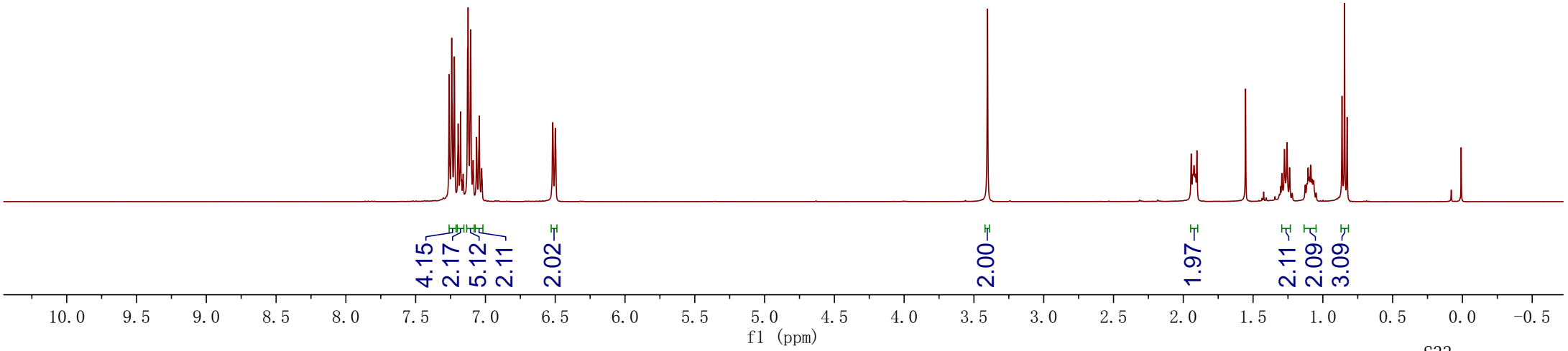

7.362
7.350
7.342
7.334
7.323
7.298
7.277
7.166
7.147
7.127
7.029
7.011
6.957
6.940
6.902
6.880
5.420
5.418
5.371
3.575
2.704
2.288
1.038
S-2o'
5.25
2.10
1.23
1.22
2.16
2.19
1.00
1.00
2.18
2.01
3.13
6.09
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

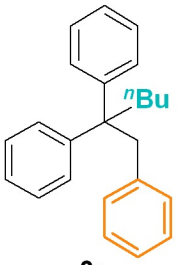

159.266
149.773
138.644
131.611
129.496
128.479
128.252
127.861
127.764
127.741
126.811
114.326
112.943
75.233
44.867
35.675
24.851
21.524
O
S-2o'
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



2a

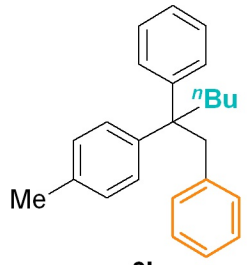
^{1}H NMR (400 MHZ, $CDCl_3$)





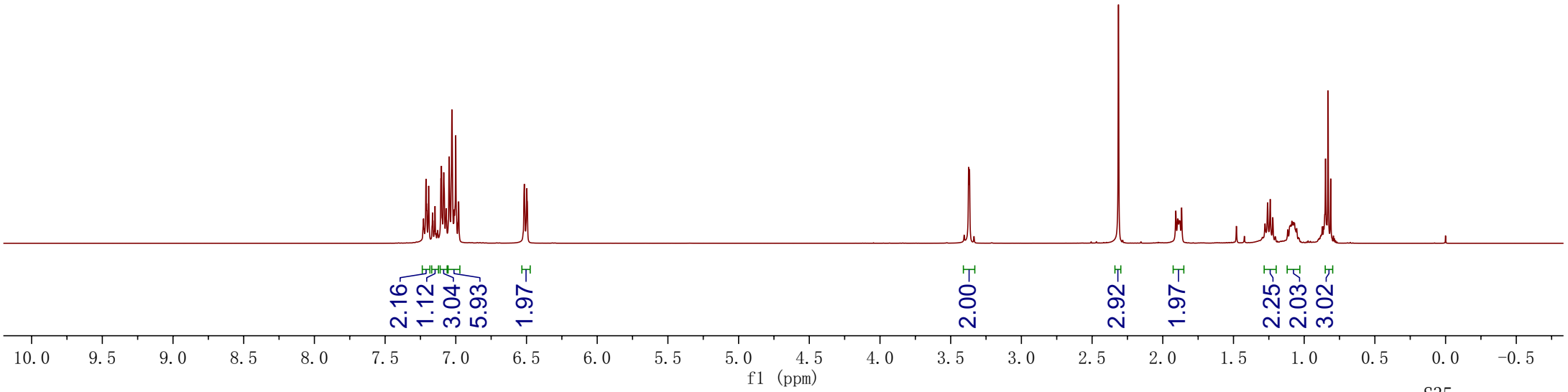
2a

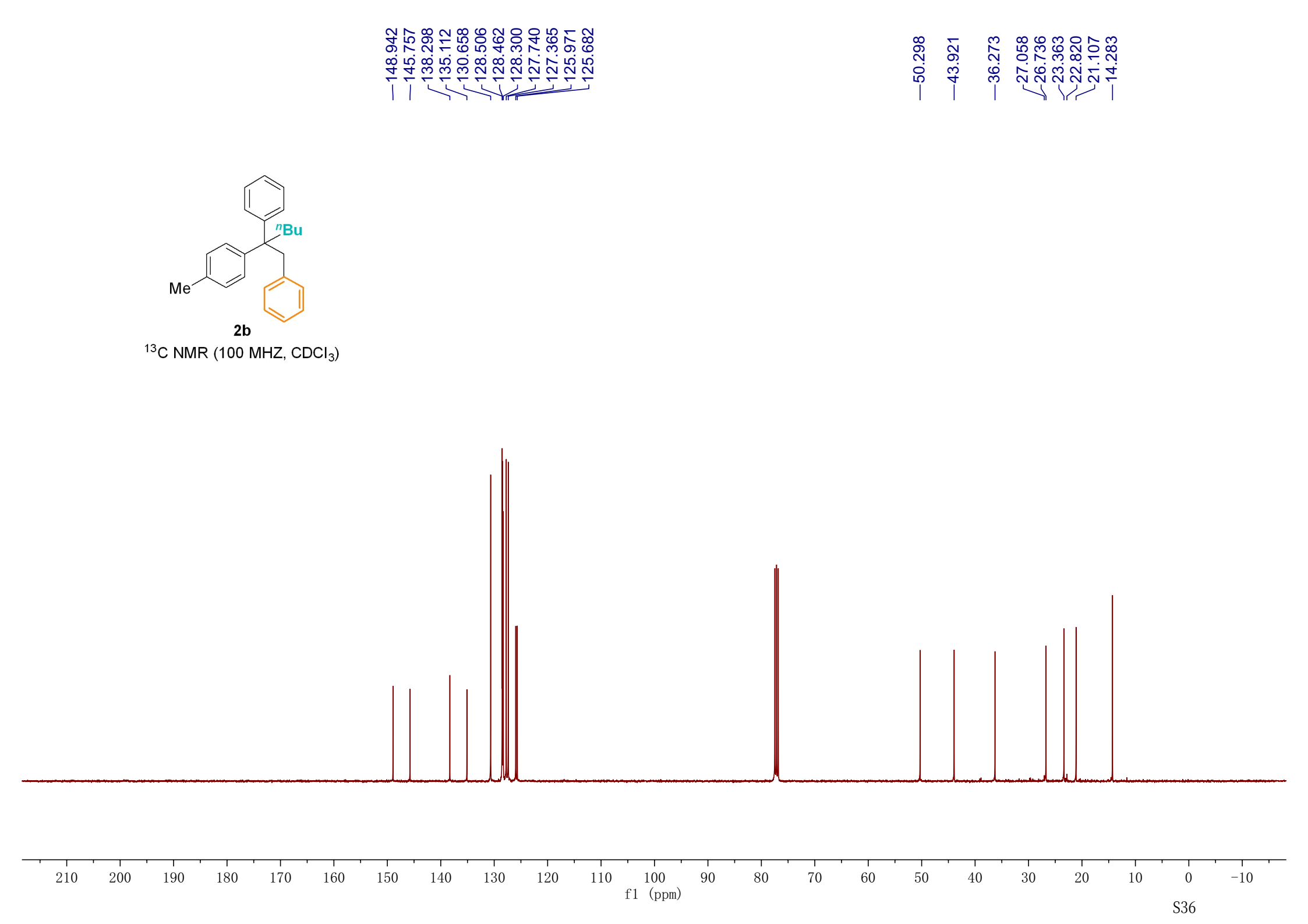
^{13}C NMR (100 MHZ, $CDCl_3$)



2b

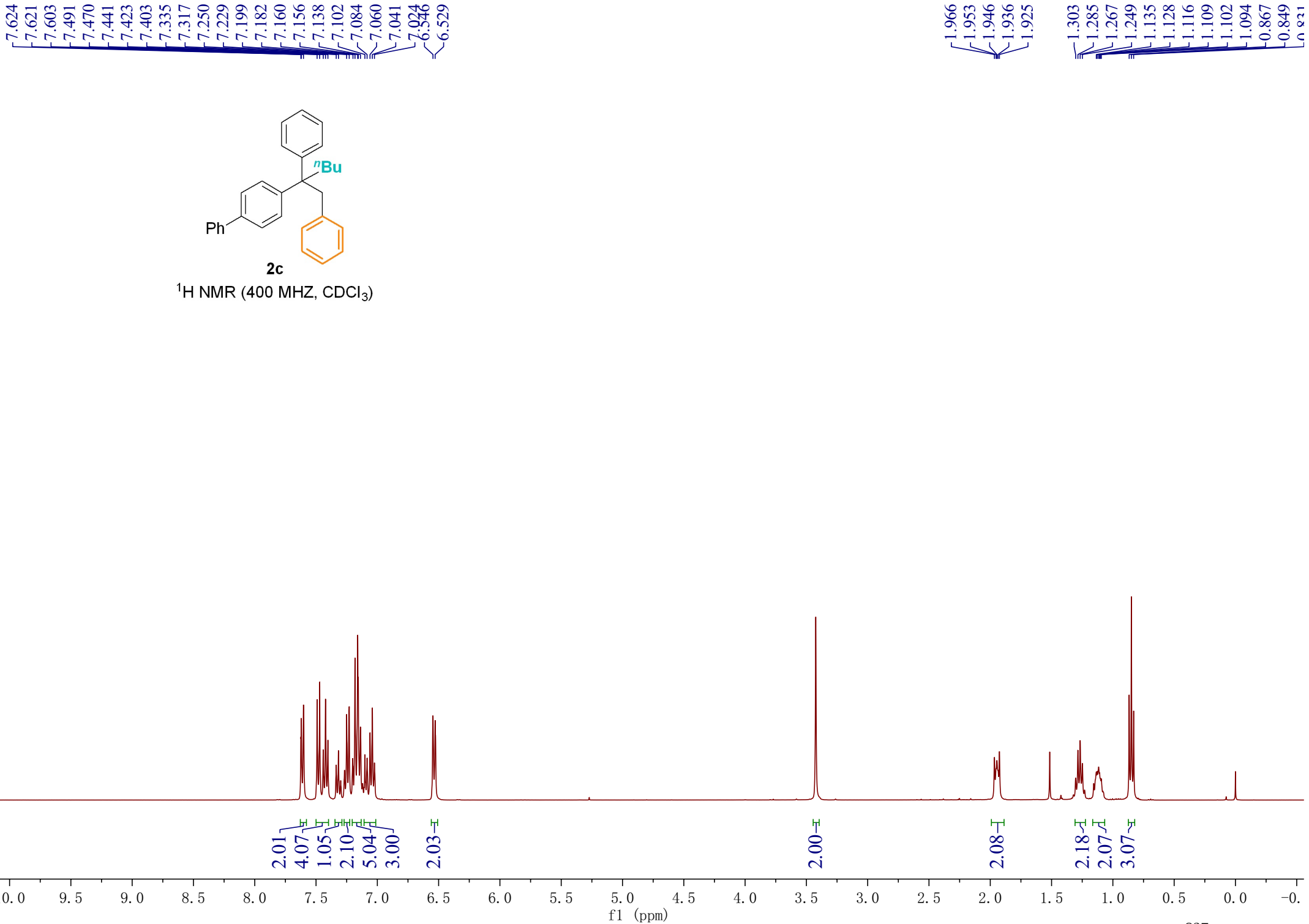
^{1}H NMR (400 MHZ, $CDCl_3$)





2b

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

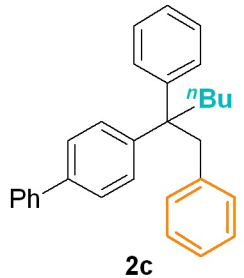


2c

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

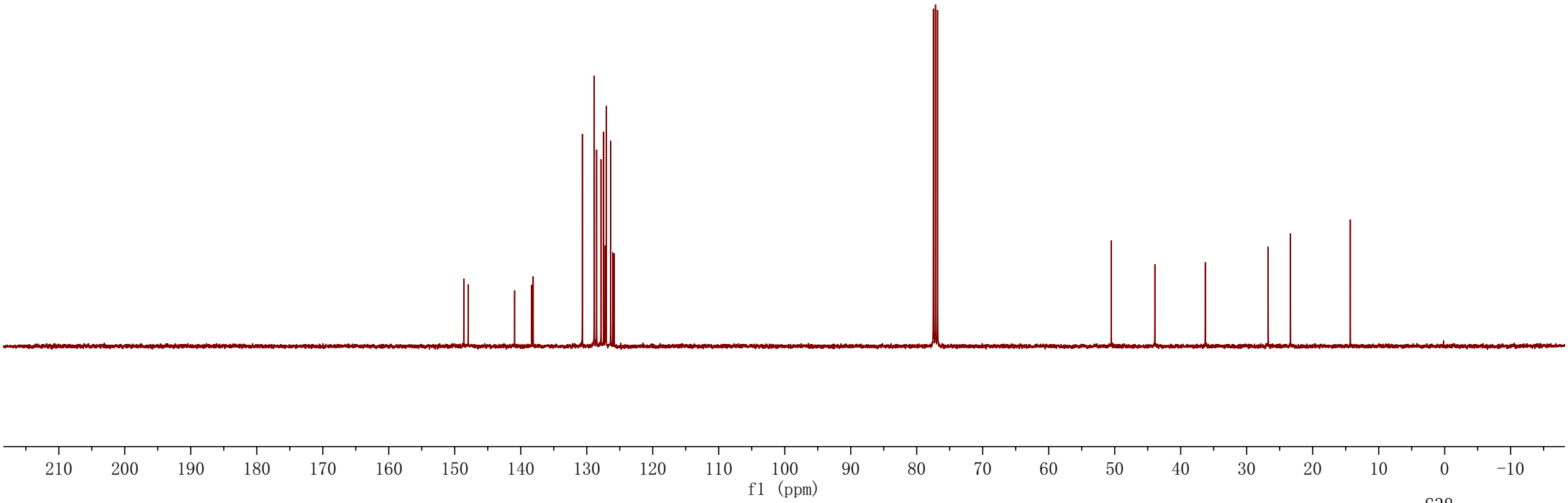
148.635
147.969
140.931
138.336
138.115
130.668
128.874
128.855
128.510
127.842
127.433
127.194
127.049
126.372
126.063
125.849

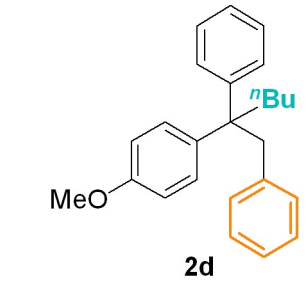
50.524
43.911
36.277
26.743
23.365
14.401



2c

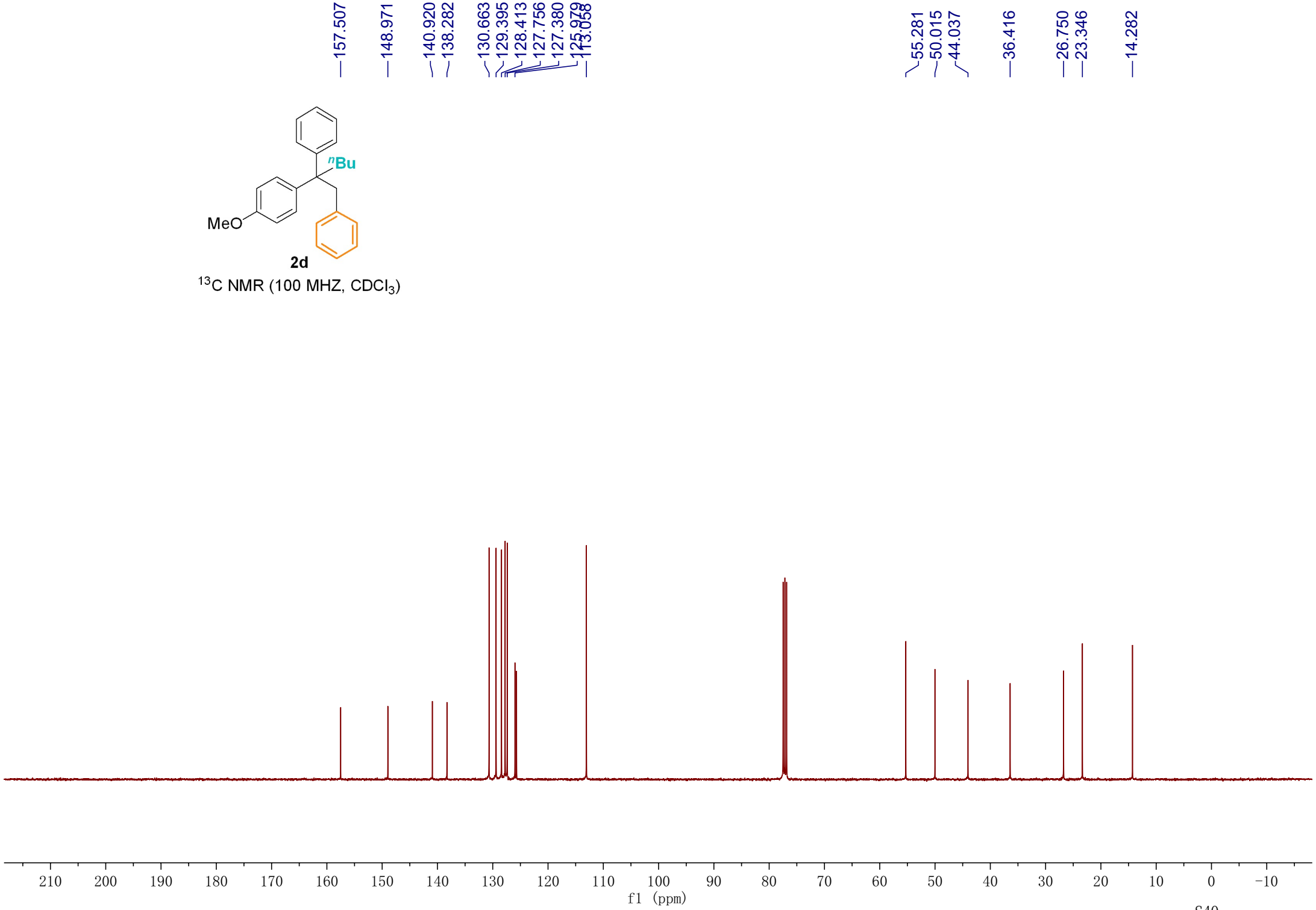
^{13}C NMR (100 MHZ, $CDCl_3$)

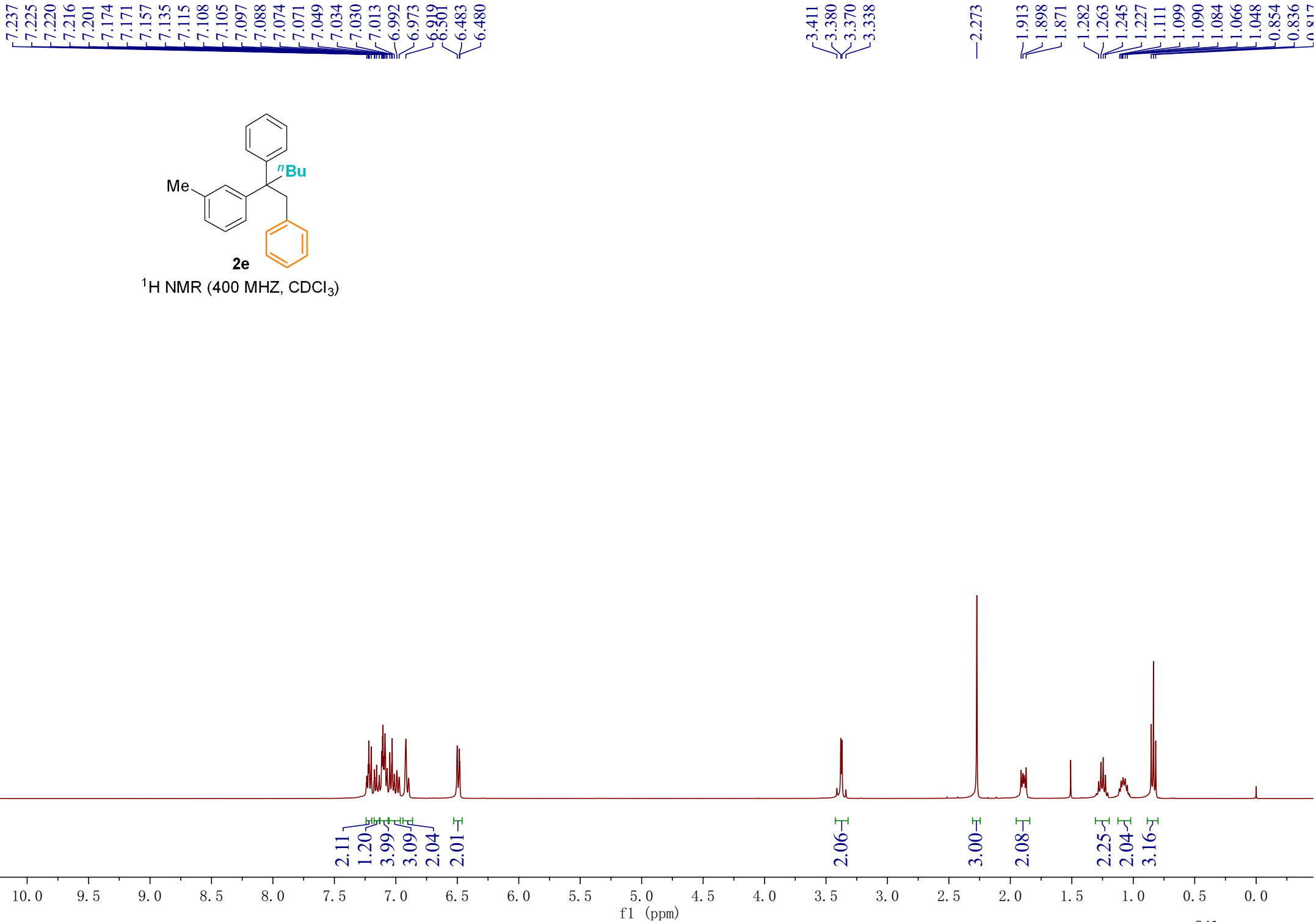


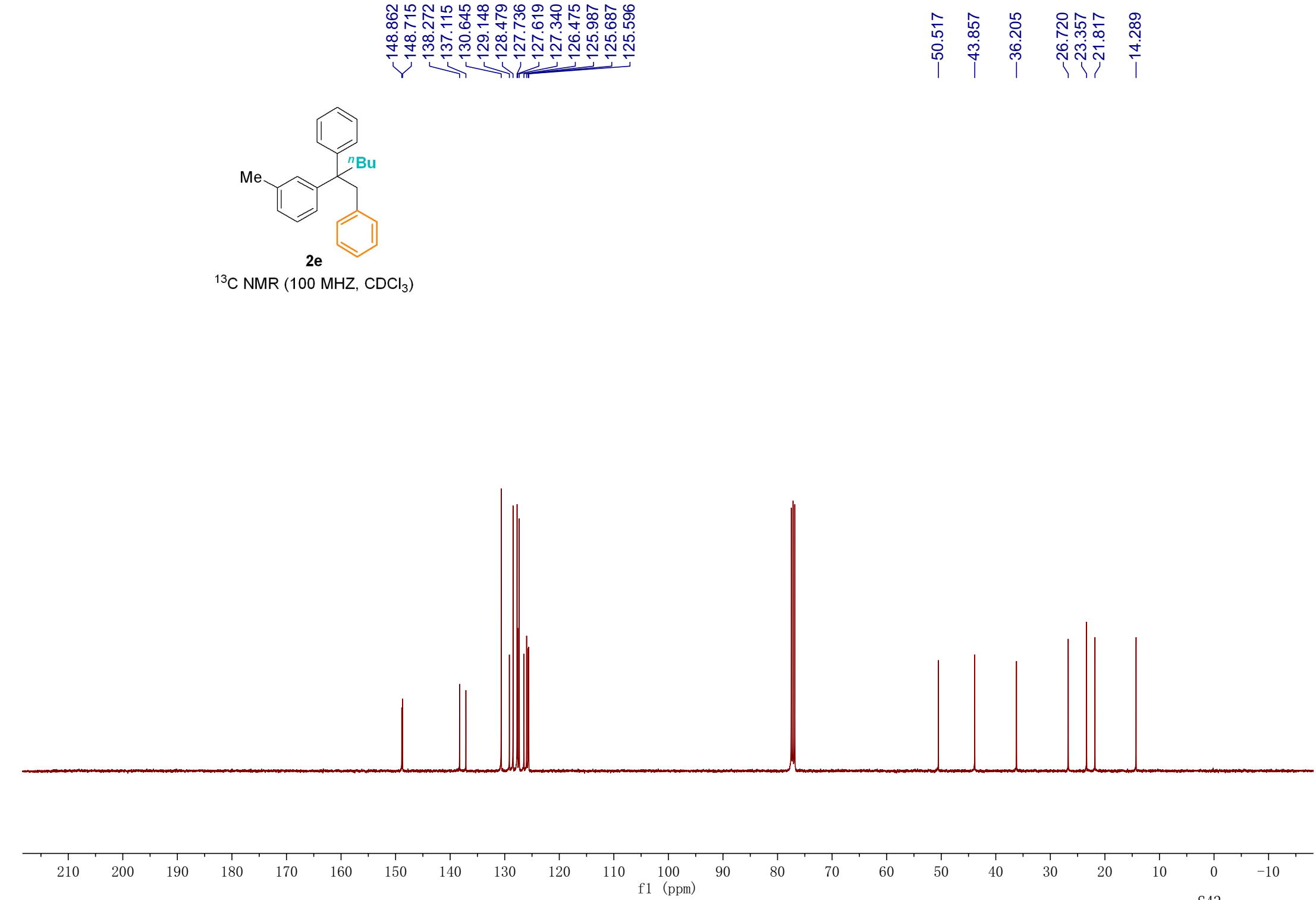


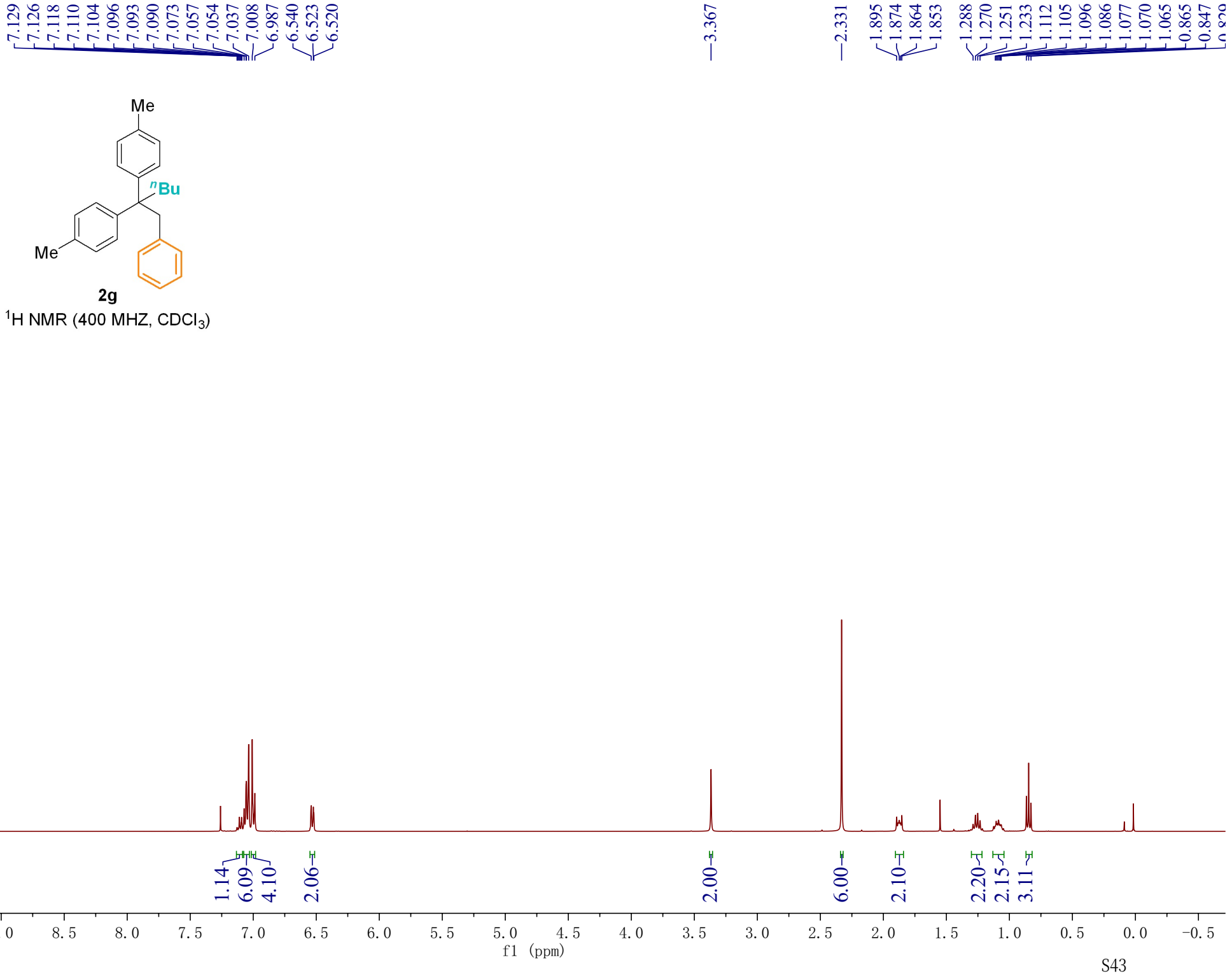
2d

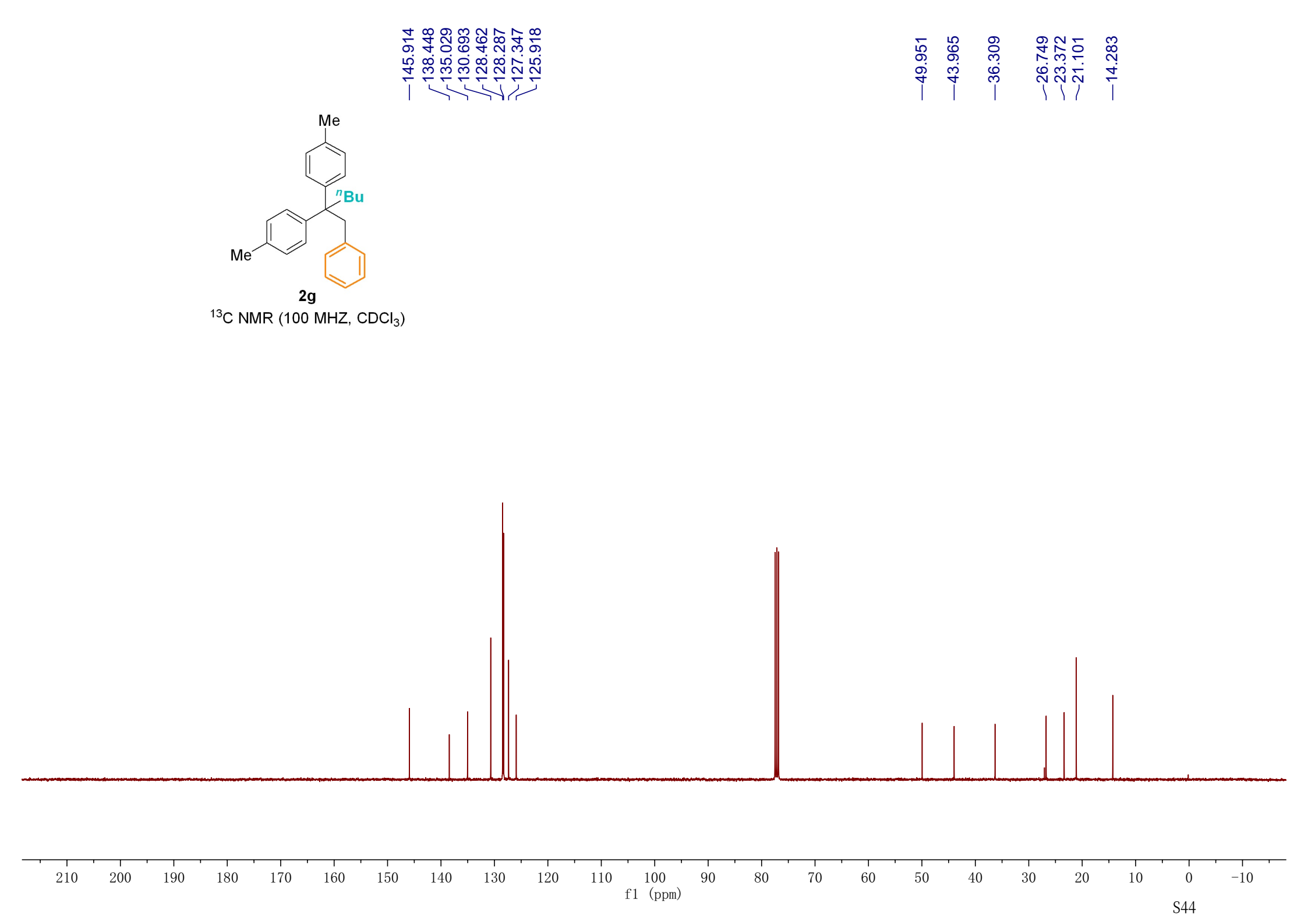
^{1}H NMR (400 MHZ, $CDCl_3$)

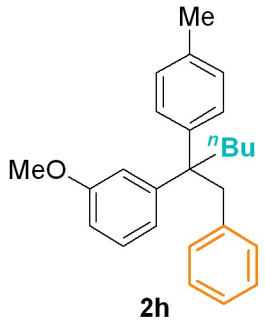

157.507
148.971
140.920
138.282
130.663
129.395
128.413
127.756
127.380
125.979
113.058
55.281
50.015
44.037
36.416
26.750
23.346
14.282
nBu
MeO
2d
13C NMR (100 MHZ, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.237
7.225
7.220
7.216
7.201
7.174
7.171
7.157
7.135
7.115
7.108
7.105
7.097
7.088
7.074
7.071
7.049
7.034
7.030
7.013
6.992
6.973
6.919
6.501
6.483
6.480
3.411
3.380
3.370
3.338
2.273
1.913
1.898
1.871
1.282
1.263
1.245
1.227
1.111
1.099
1.090
1.084
1.066
1.048
0.854
0.836
Me
nBu
2e
1H NMR (400 MHZ, CDCl3)
2.11
1.20
3.99
3.09
2.04
2.01
2.06
3.00
2.08
2.25
2.04
3.16
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)


148.862
148.715
138.272
137.115
130.645
129.148
128.479
127.736
127.619
127.340
126.475
125.987
125.687
125.596
50.517
43.857
36.205
26.720
23.357
21.817
14.289
Me
nBu
2e
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

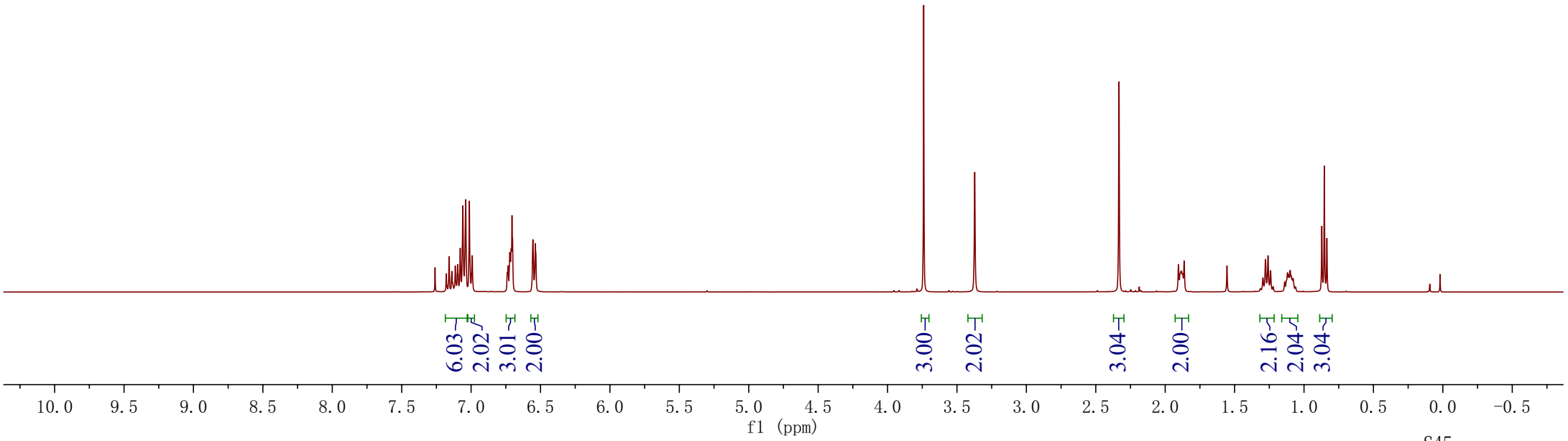

7.129
7.126
7.118
7.110
7.104
7.096
7.093
7.090
7.073
7.057
7.054
7.037
7.008
6.987
6.540
6.523
6.520
3.367
2.331
1.895
1.874
1.864
1.853
1.288
1.270
1.251
1.233
1.112
1.105
1.096
1.086
1.077
1.070
1.065
0.865
0.847
Me
nBu
Me
2g
1H NMR (400 MHZ, CDCl3)
1.14
6.09
4.10
2.06
2.00
6.00
2.10
2.20
2.15
3.11
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

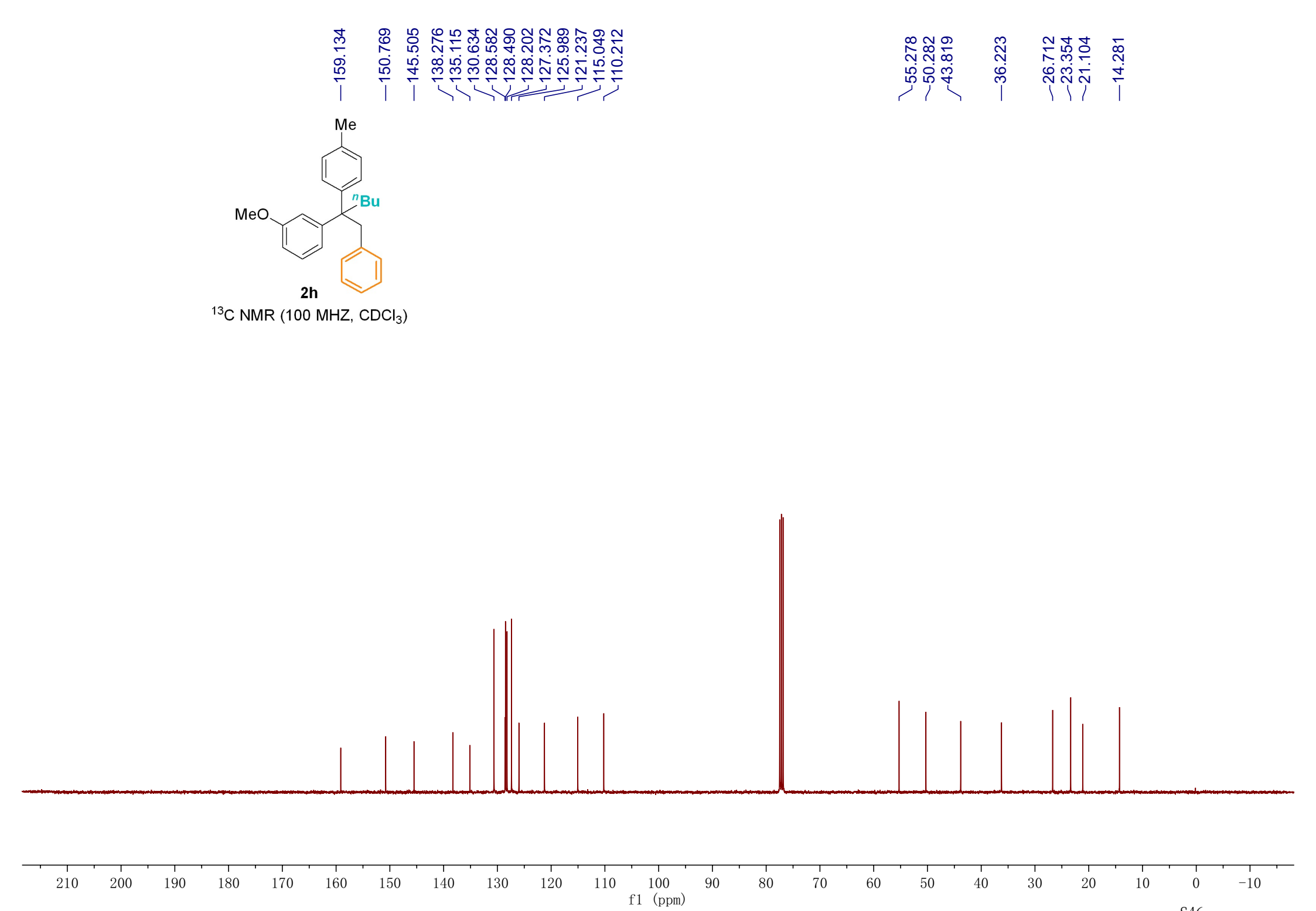

145.914
138.448
135.029
130.693
128.462
128.287
127.347
125.918
49.951
43.965
36.309
26.749
23.372
21.101
14.283
Me
nBu
Me
2g
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

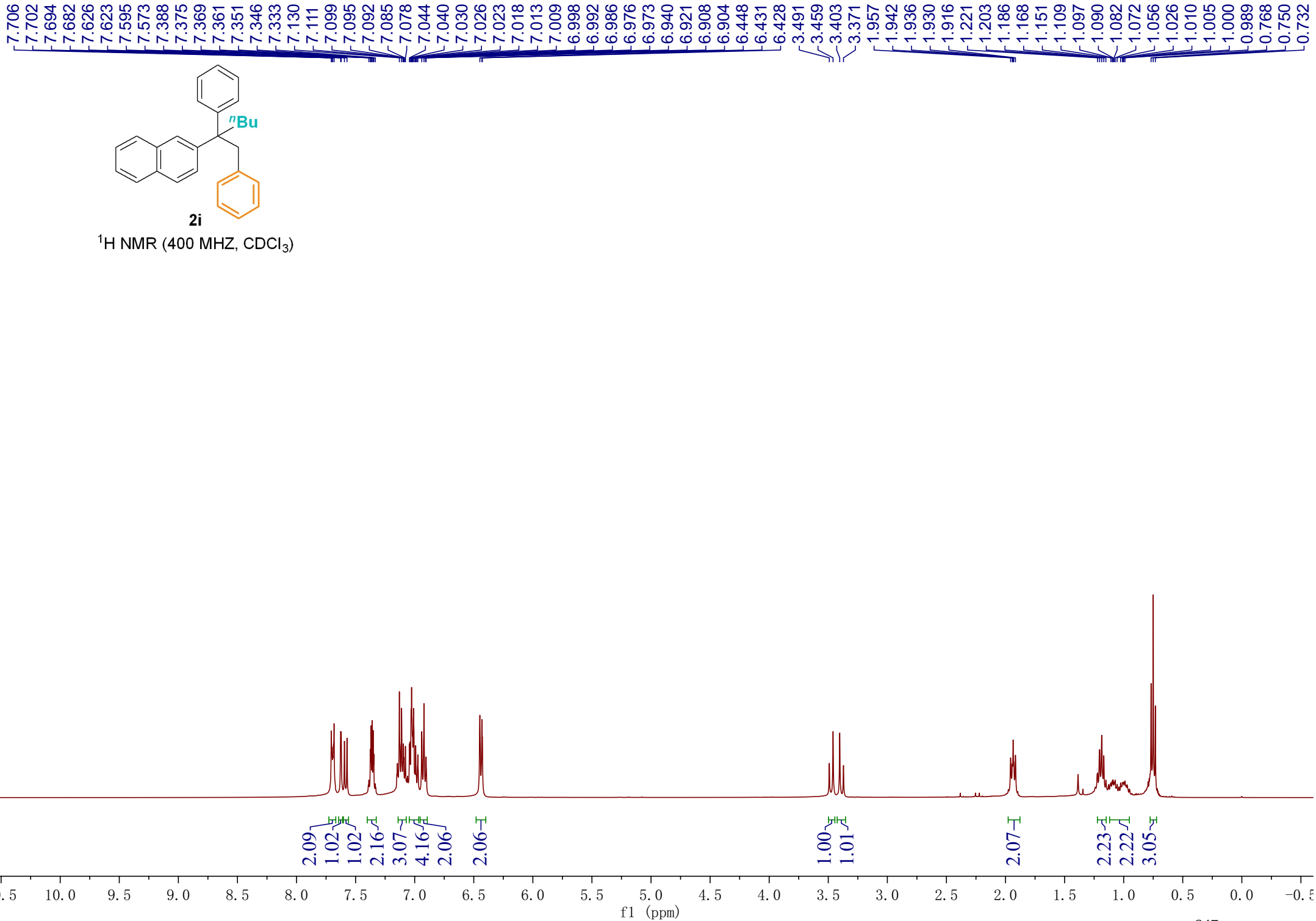


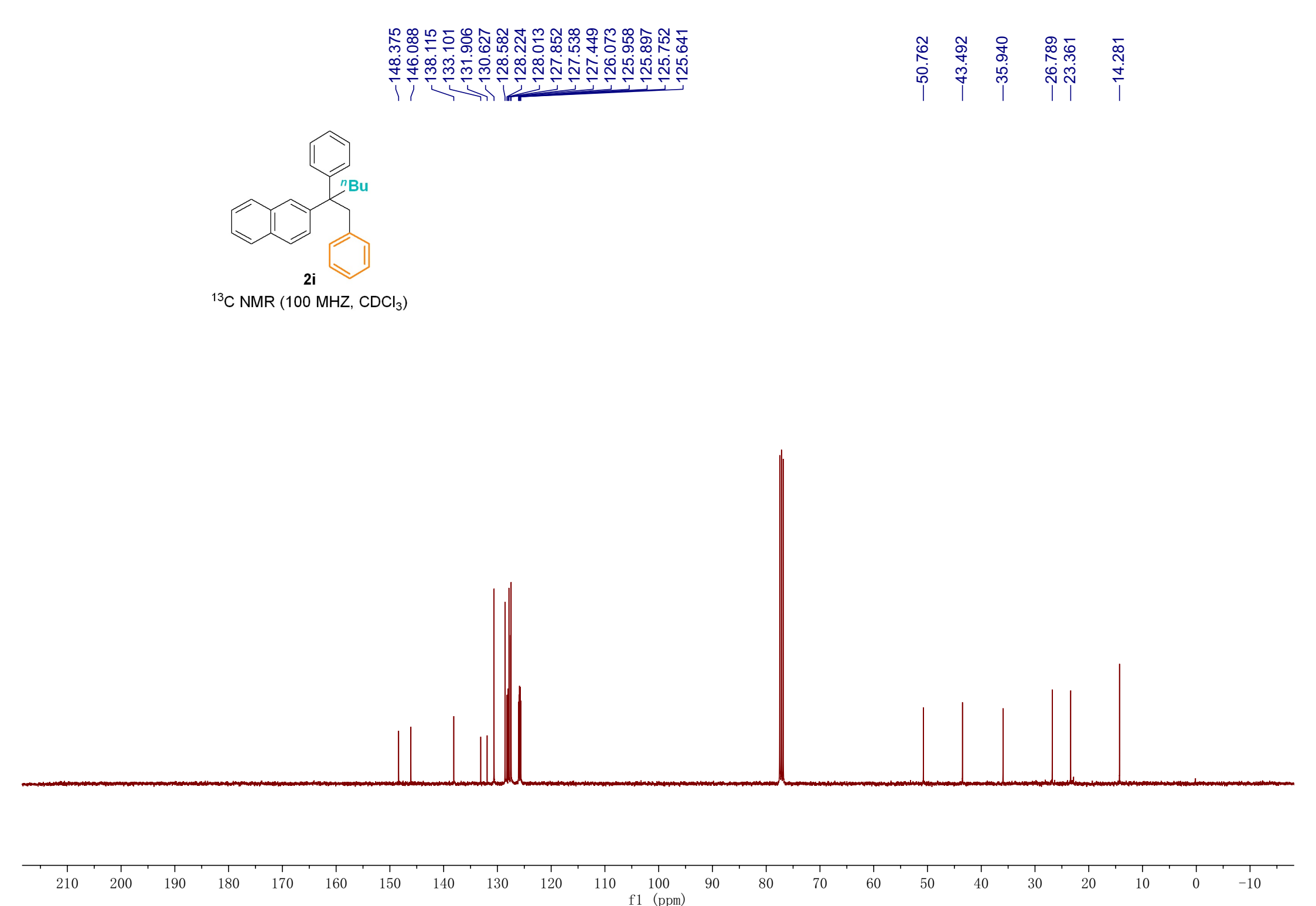
2h

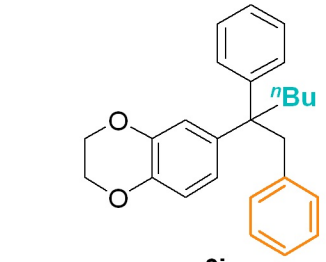
^{1}H NMR (400 MHZ, $CDCl_3$)




159.134
150.769
145.505
138.276
135.115
130.634
128.582
128.490
128.202
127.372
125.989
121.237
115.049
110.212
55.278
50.282
43.819
36.223
26.712
23.354
21.104
14.281
Me
MeO
nBu
2h
13C NMR (100 MHZ, CDCl3)
f1 (ppm)

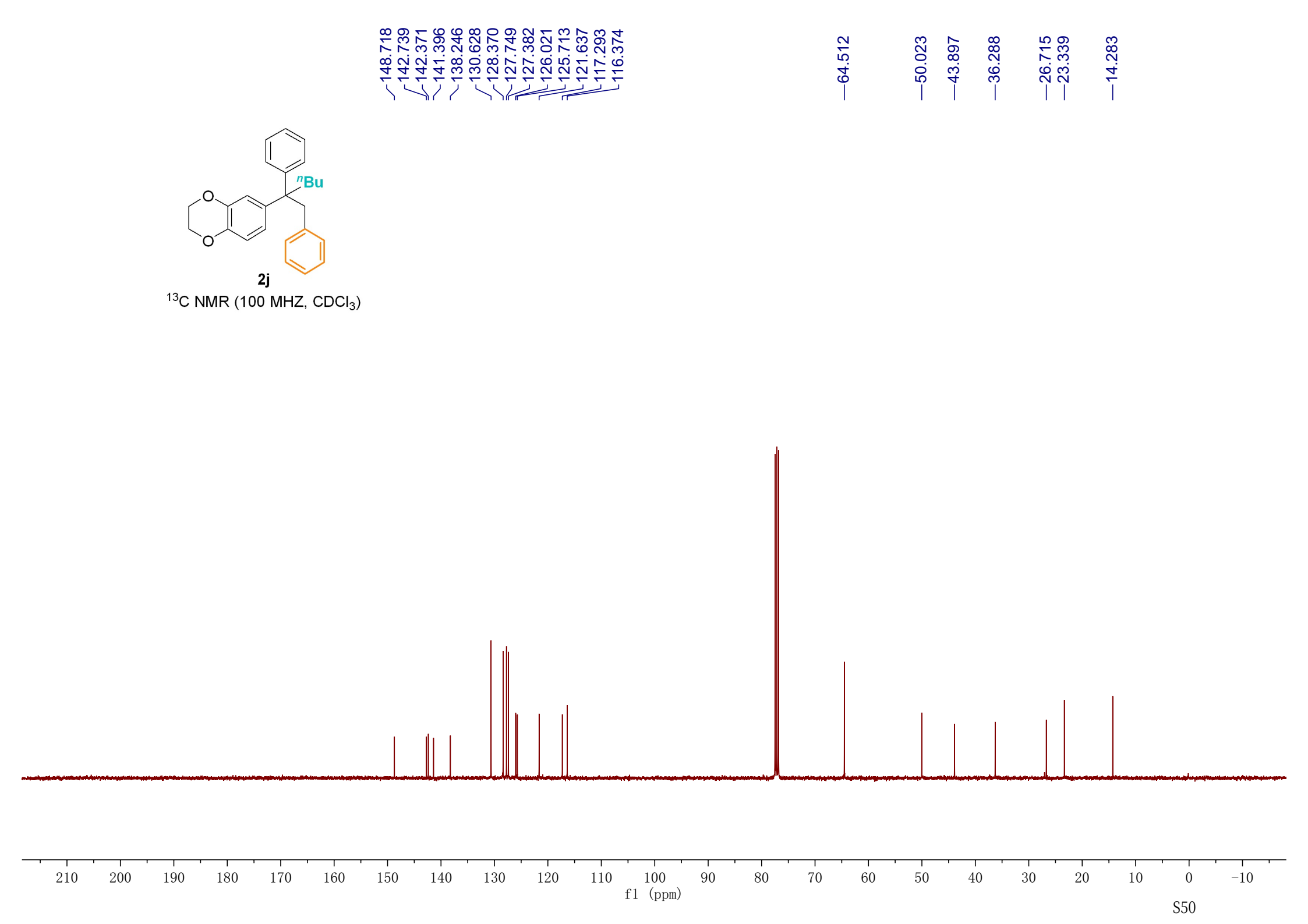

7.706
7.702
7.694
7.682
7.626
7.623
7.595
7.573
7.388
7.375
7.369
7.361
7.351
7.346
7.333
7.130
7.111
7.099
7.095
7.092
7.085
7.078
7.044
7.040
7.030
7.026
7.023
7.018
7.013
7.009
6.998
6.992
6.986
6.976
6.973
6.940
6.921
6.908
6.904
6.448
6.431
6.428
3.491
3.459
3.403
3.371
1.957
1.942
1.936
1.930
1.916
1.221
1.203
1.186
1.168
1.151
1.109
1.097
1.090
1.082
1.072
1.056
1.026
1.010
1.005
1.000
0.989
0.768
0.750
0.732
nBu
2i
1H NMR (400 MHZ, CDCl3)
2.09
1.02
1.02
2.16
3.07
4.16
2.06
2.06
1.00
1.01
2.07
2.23
2.22
3.05
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)

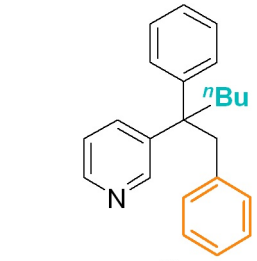

148.375
146.088
138.115
133.101
131.906
130.627
128.582
128.224
128.013
127.852
127.538
127.449
126.073
125.958
125.897
125.752
125.641
50.762
43.492
35.940
26.789
23.361
14.281
nBu
2i
13C NMR (100 MHZ, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



2j

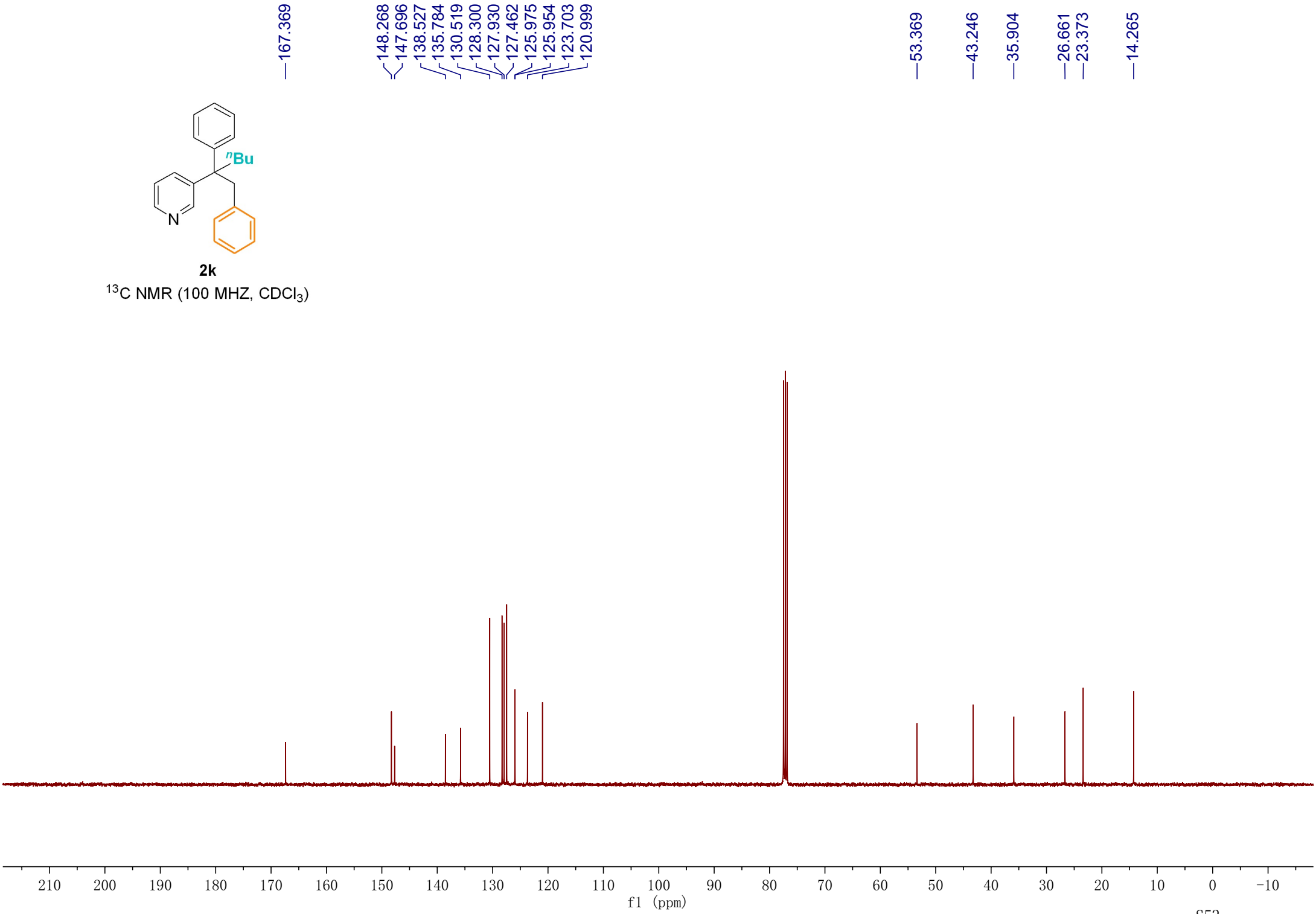
^{1}H NMR (400 MHZ, $CDCl_3$)


148.718
142.739
142.371
141.396
138.246
130.628
128.370
127.749
127.382
126.021
125.713
121.637
117.293
116.374
64.512
50.023
43.897
36.288
26.715
23.339
14.283
nBu
O
O
2j
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



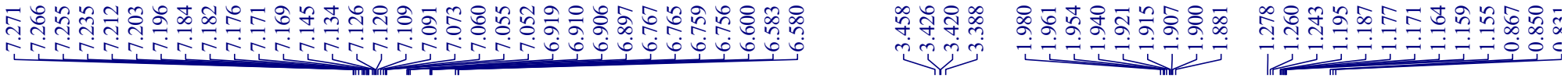
2k

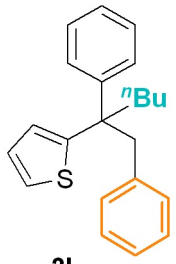
^{1}H NMR (400 MHZ, $CDCl_3$)



2k

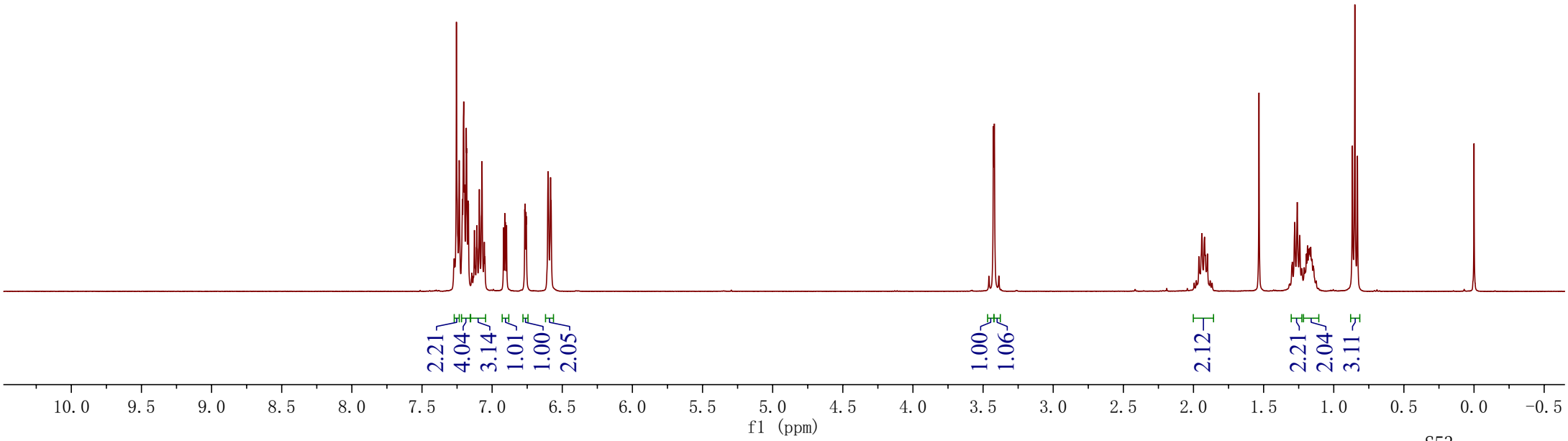
^{13}C NMR (100 MHZ, $CDCl_3$)

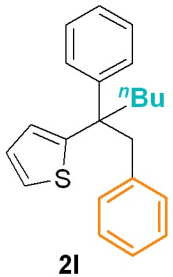




2l

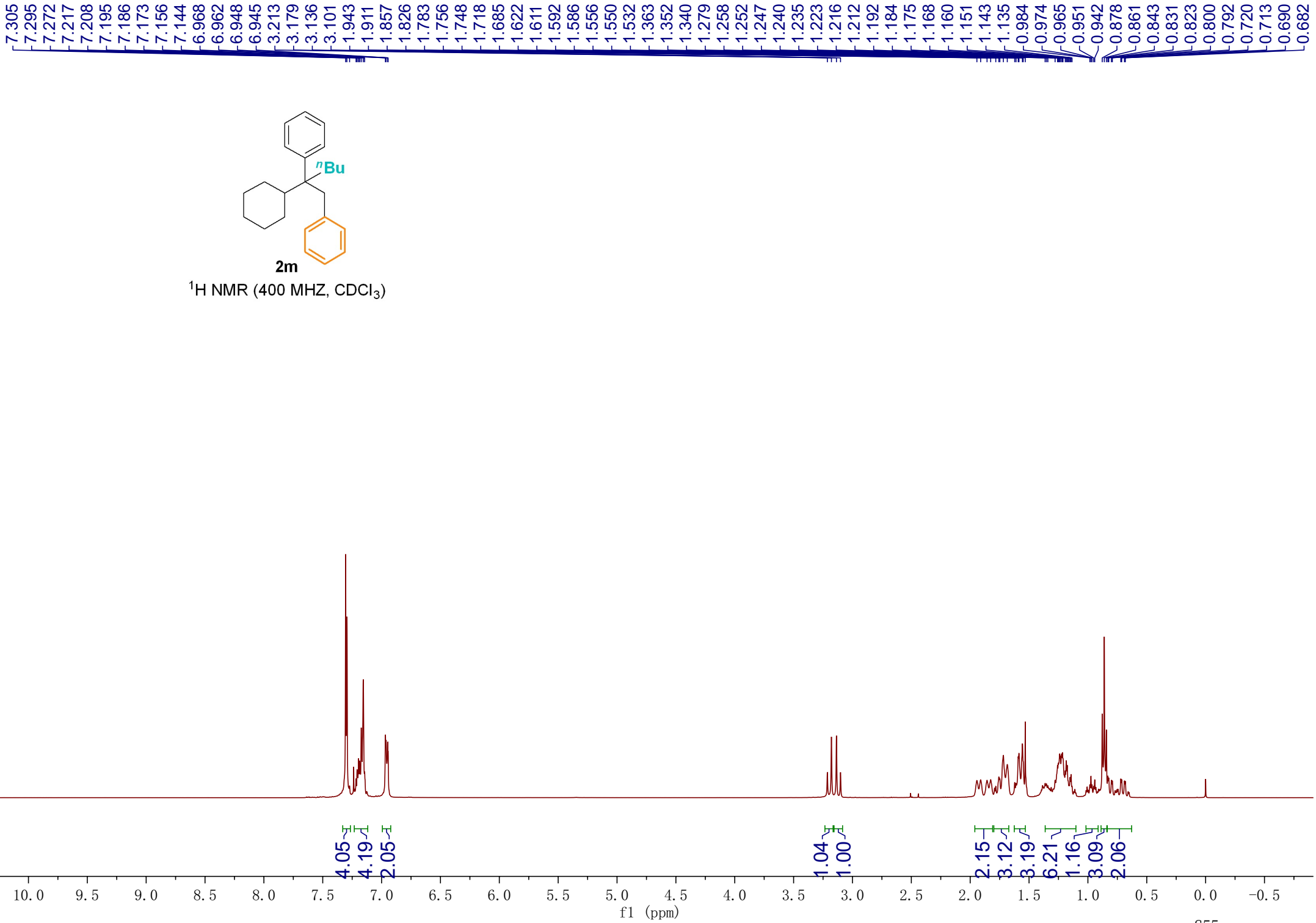
^{1}H NMR (400 MHZ, $CDCl_3$)

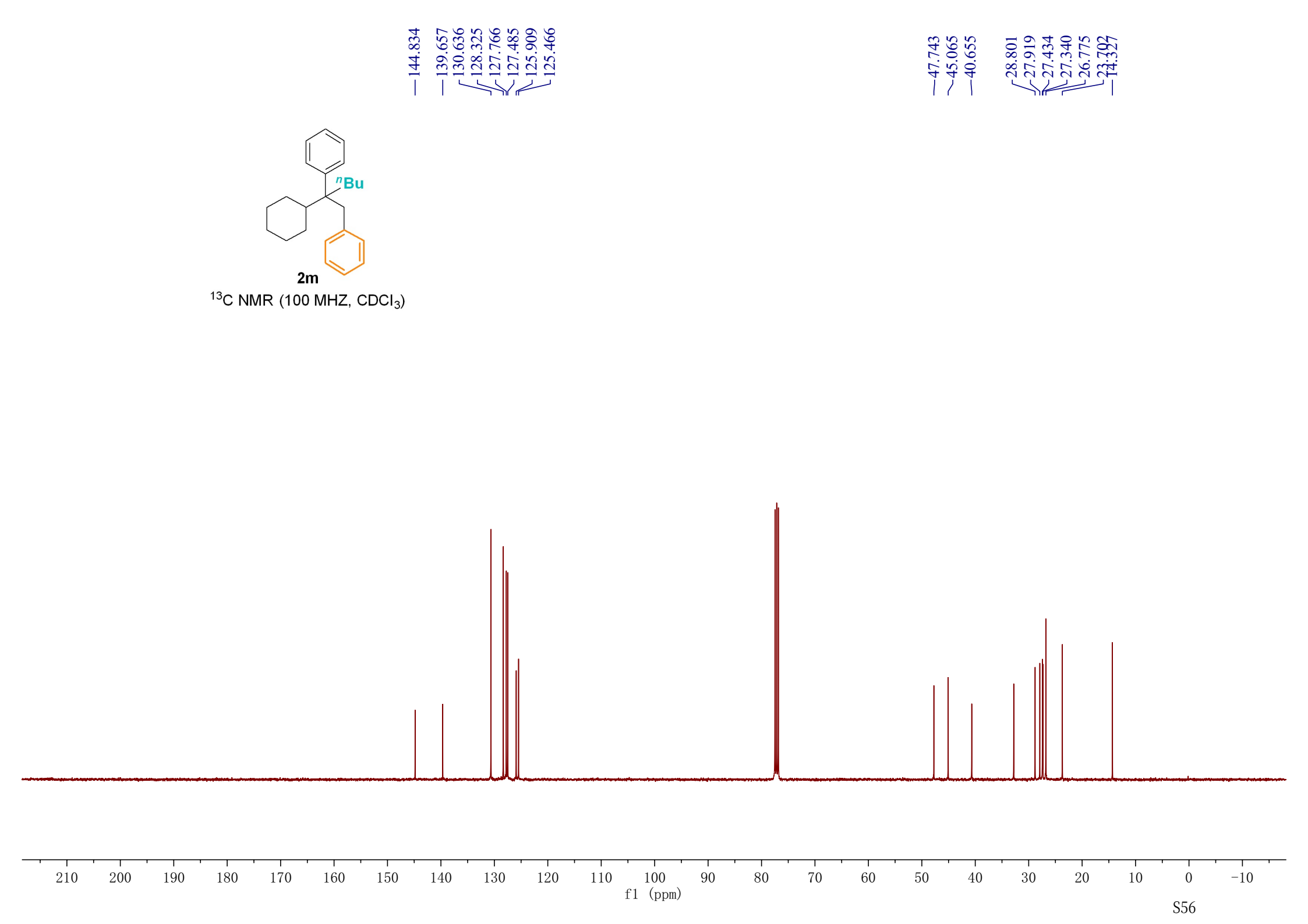


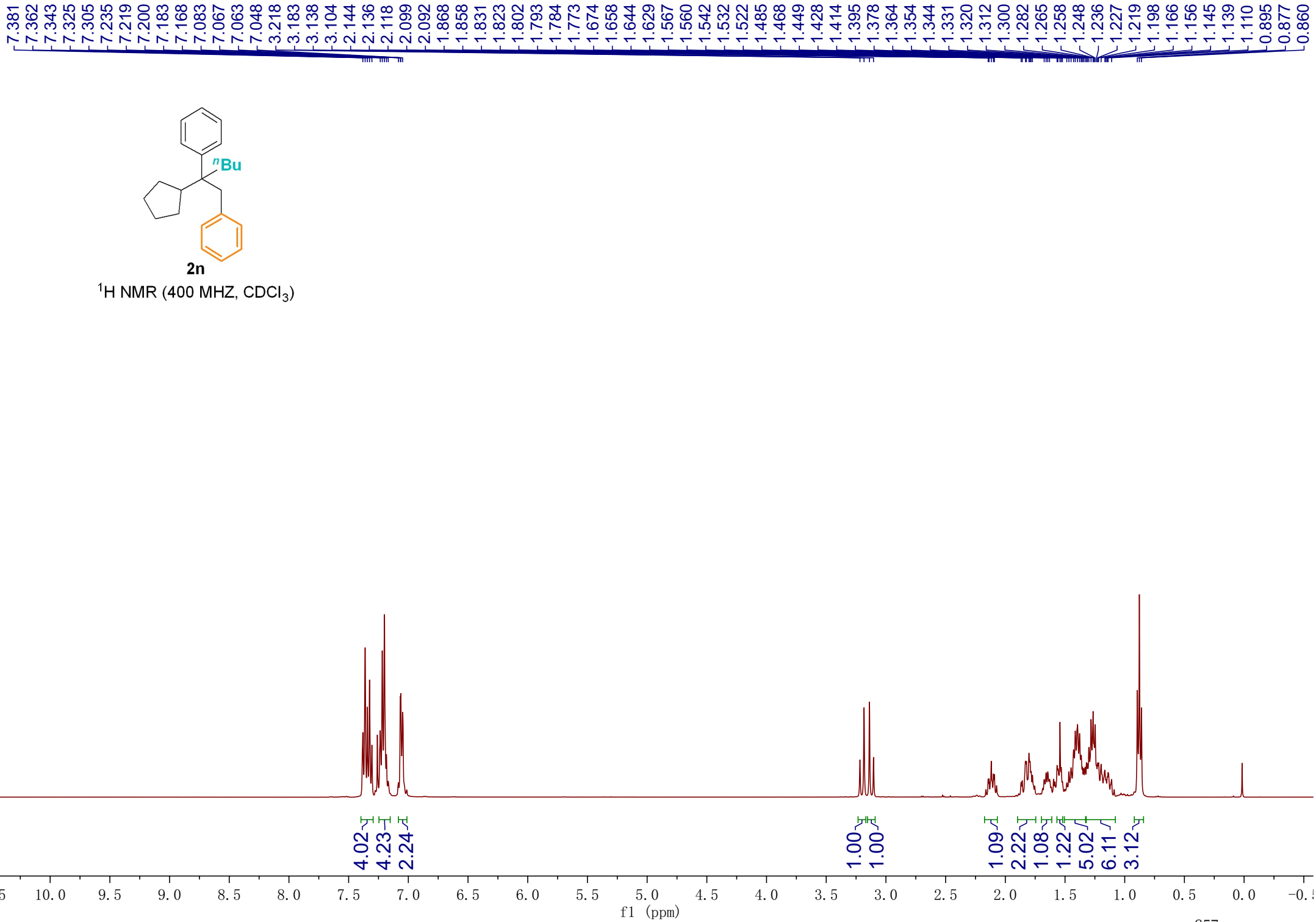


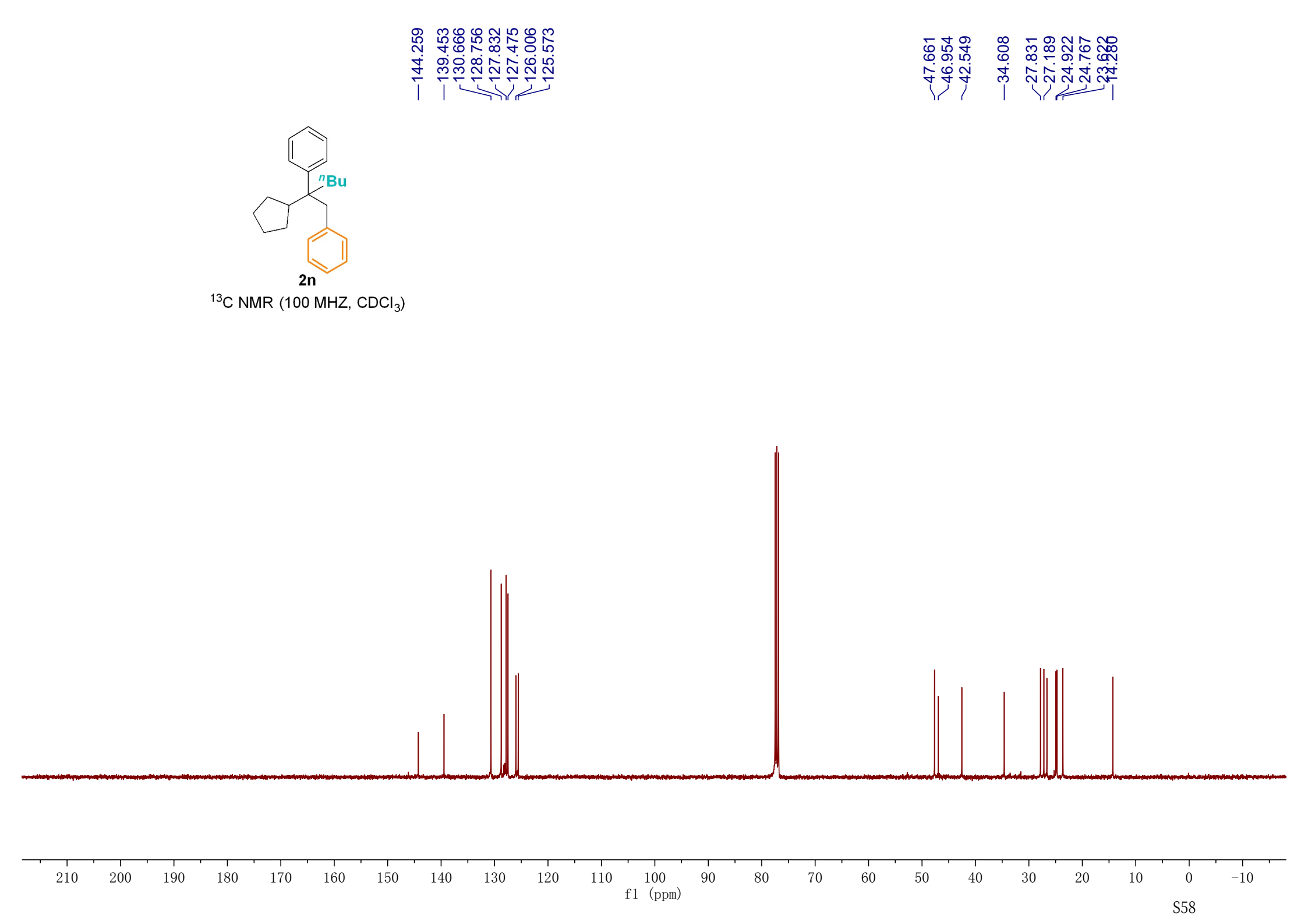
2l

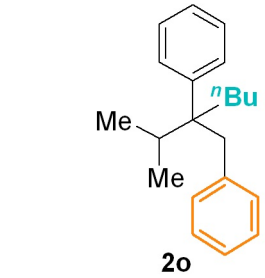
^{13}C NMR (100 MHZ, $CDCl_3$)


7.305
7.295
7.272
7.217
7.208
7.195
7.186
7.173
7.156
7.144
6.968
6.962
6.948
6.945
3.213
3.179
3.136
3.101
1.943
1.911
1.857
1.826
1.783
1.756
1.748
1.718
1.685
1.622
1.611
1.592
1.586
1.556
1.550
1.532
1.363
1.352
1.340
1.279
1.258
1.252
1.247
1.240
1.235
1.223
1.216
1.212
1.192
1.184
1.175
1.168
1.160
1.151
1.143
1.135
0.984
0.974
0.965
0.951
0.942
0.878
0.861
0.843
0.831
0.823
0.800
0.792
0.720
0.713
0.690
0.682
nBu
2m
1H NMR (400 MHZ, CDCl3)
4.05
4.19
2.05
1.04
1.00
2.15
3.12
3.19
6.21
1.16
3.09
2.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


144.834
139.657
130.636
128.325
127.766
127.485
125.909
125.466
47.743
45.065
40.655
28.801
27.919
27.434
27.340
26.775
23.792
14.327
nBu
2m
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

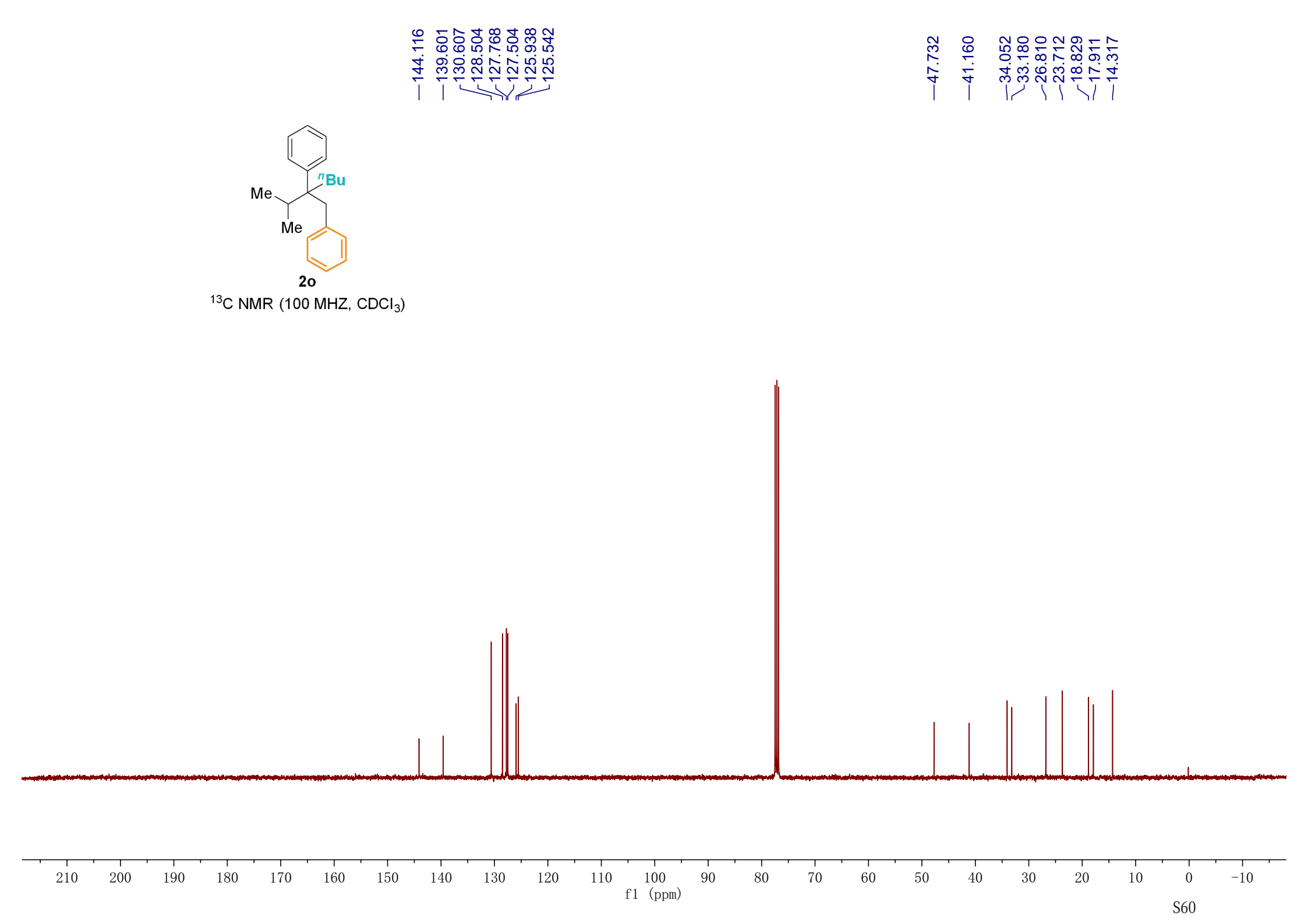

7.381
7.362
7.343
7.325
7.305
7.235
7.219
7.200
7.183
7.168
7.083
7.067
7.063
7.048
3.218
3.183
3.138
3.104
2.144
2.136
2.118
2.099
2.092
1.868
1.858
1.831
1.823
1.802
1.793
1.784
1.773
1.674
1.658
1.644
1.629
1.567
1.560
1.542
1.532
1.522
1.485
1.468
1.449
1.428
1.414
1.395
1.378
1.364
1.354
1.344
1.331
1.320
1.312
1.300
1.282
1.265
1.258
1.248
1.236
1.227
1.219
1.198
1.166
1.156
1.145
1.139
1.110
0.895
0.877
0.860
nBu
2n
1H NMR (400 MHZ, CDCl3)
4.02
4.23
2.24
1.00
1.00
1.09
2.22
1.08
1.22
5.02
6.11
3.12
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)

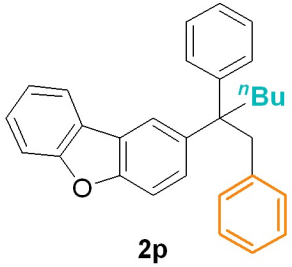

144.259
139.453
130.666
128.756
127.832
127.475
126.006
125.573
47.661
46.954
42.549
34.608
27.831
27.189
24.922
24.767
23.622
14.280
nBu
2n
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



2o

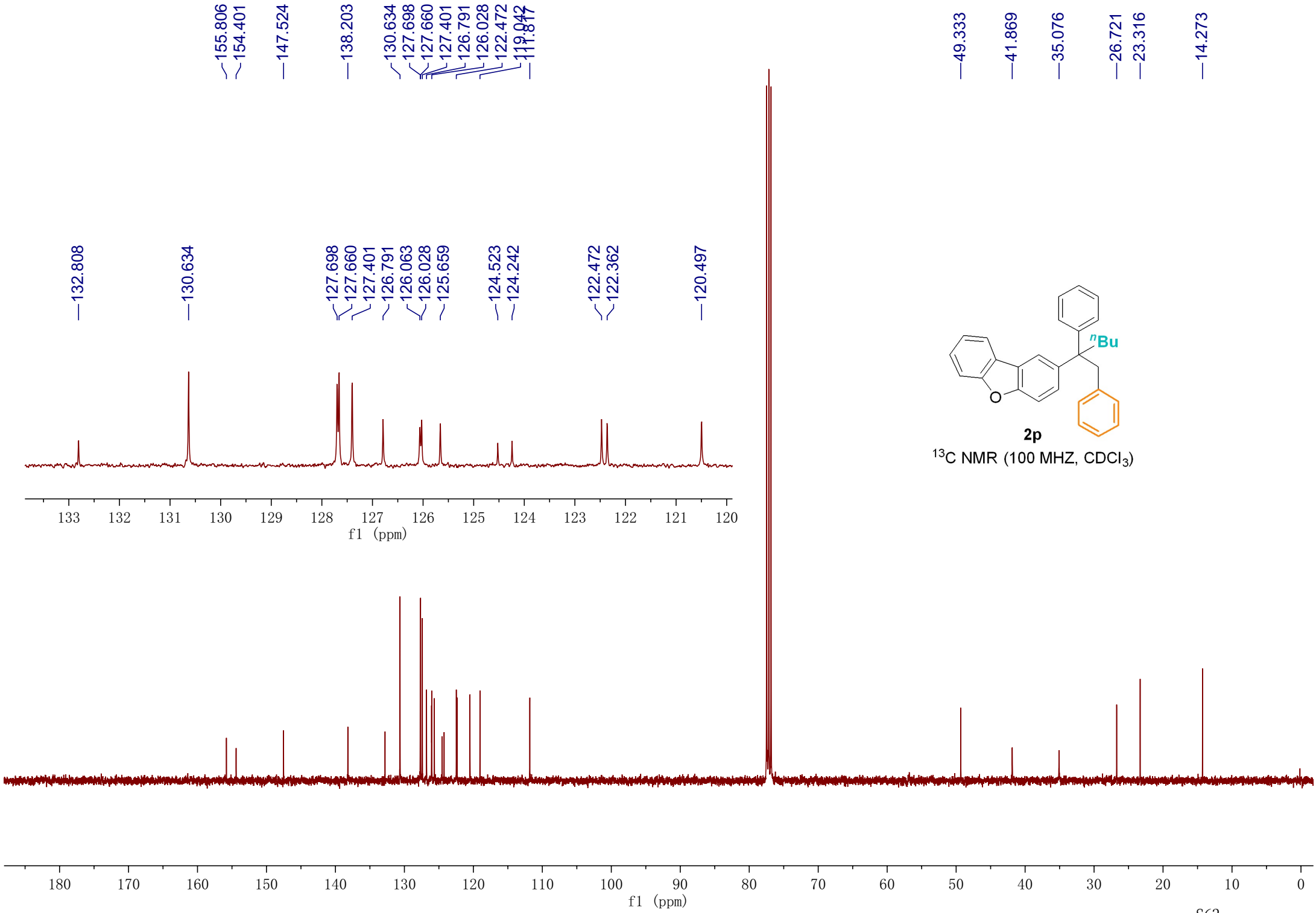
^{1}H NMR (400 MHZ, $CDCl_3$)

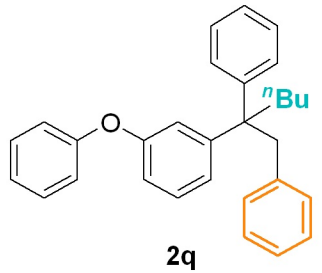

144.116
139.601
130.607
128.504
127.768
127.504
125.938
125.542
47.732
41.160
34.052
33.180
26.810
23.712
18.829
17.911
14.317
nBu
Me
Me
2o
13C NMR (100 MHZ, CDCl3)
f1 (ppm)



2p

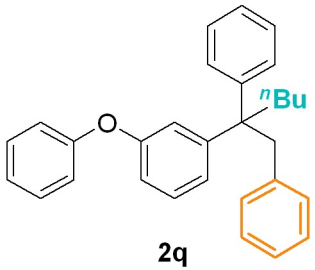
^{1}H NMR (400 MHZ, $CDCl_3$)


155.806
154.401
147.524
138.203
130.634
127.698
127.660
127.401
126.791
126.028
122.472
119.042
111.817
49.333
41.869
35.076
26.721
23.316
14.273
132.808
130.634
127.698
127.660
127.401
126.791
126.063
126.028
125.659
124.523
124.242
122.472
122.362
120.497
nBu
O
2p
13C NMR (100 MHZ, CDCl3)
f1 (ppm)
f1 (ppm)



2q

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



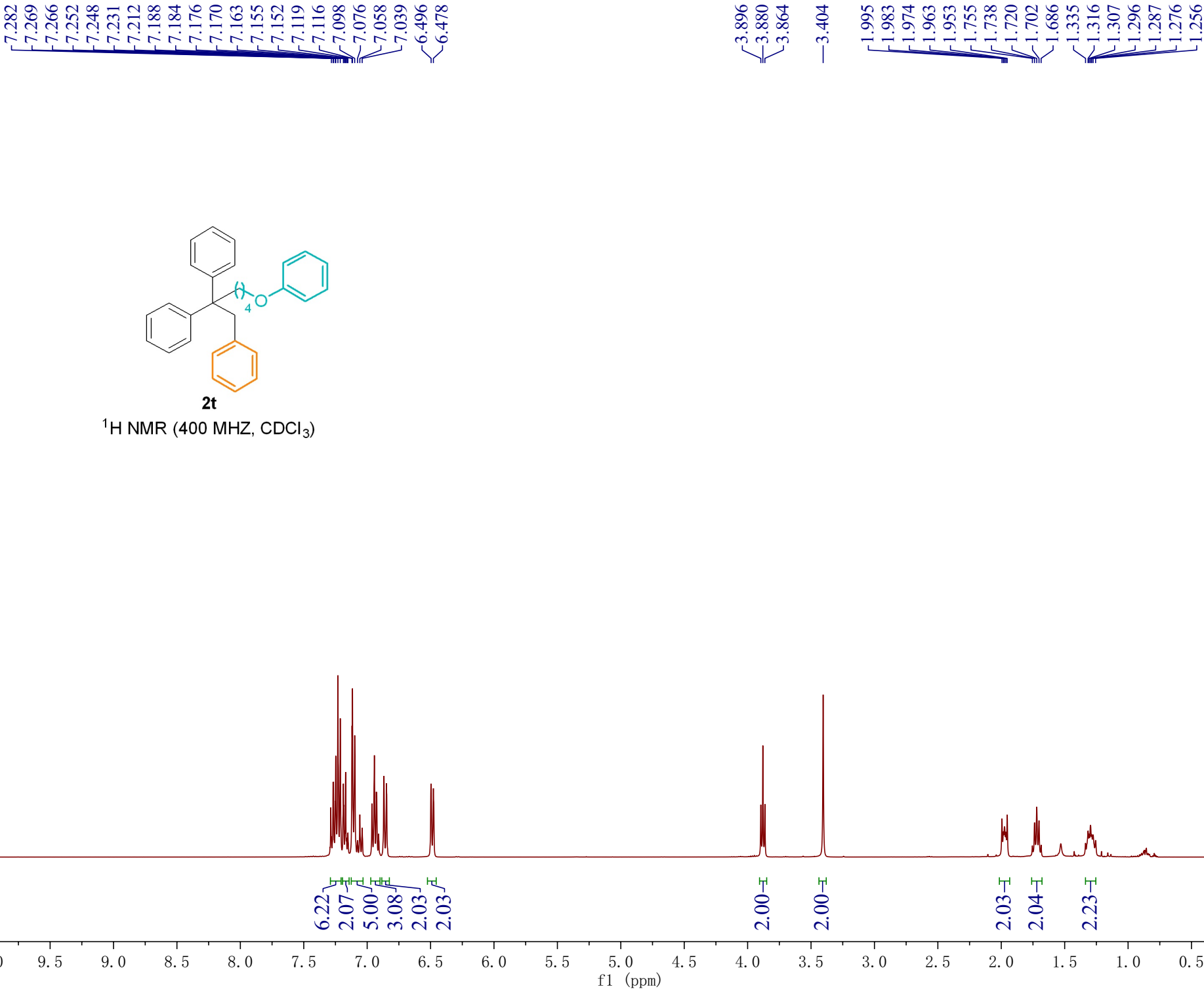
2q

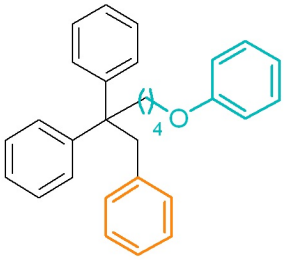
^{13}C NMR (100 MHZ, $CDCl_3$)

157.889
156.138
151.164
148.364
138.018
130.635
129.702
129.047
128.354
127.870
127.502
126.112
125.903
124.307
122.713
119.912
118.005
116.800
50.709
43.760
36.204
26.736
23.302
14.280

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

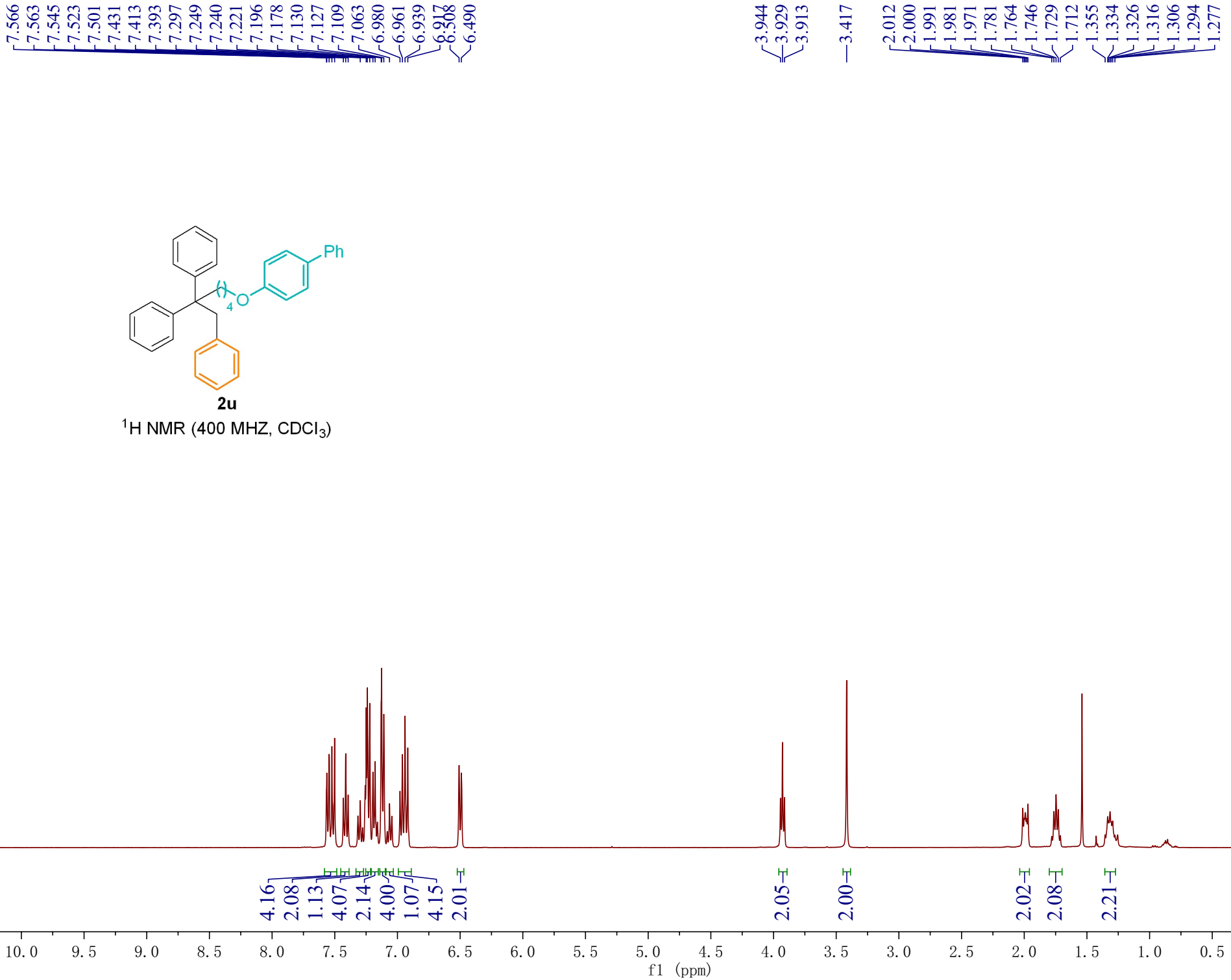
f1 (ppm)

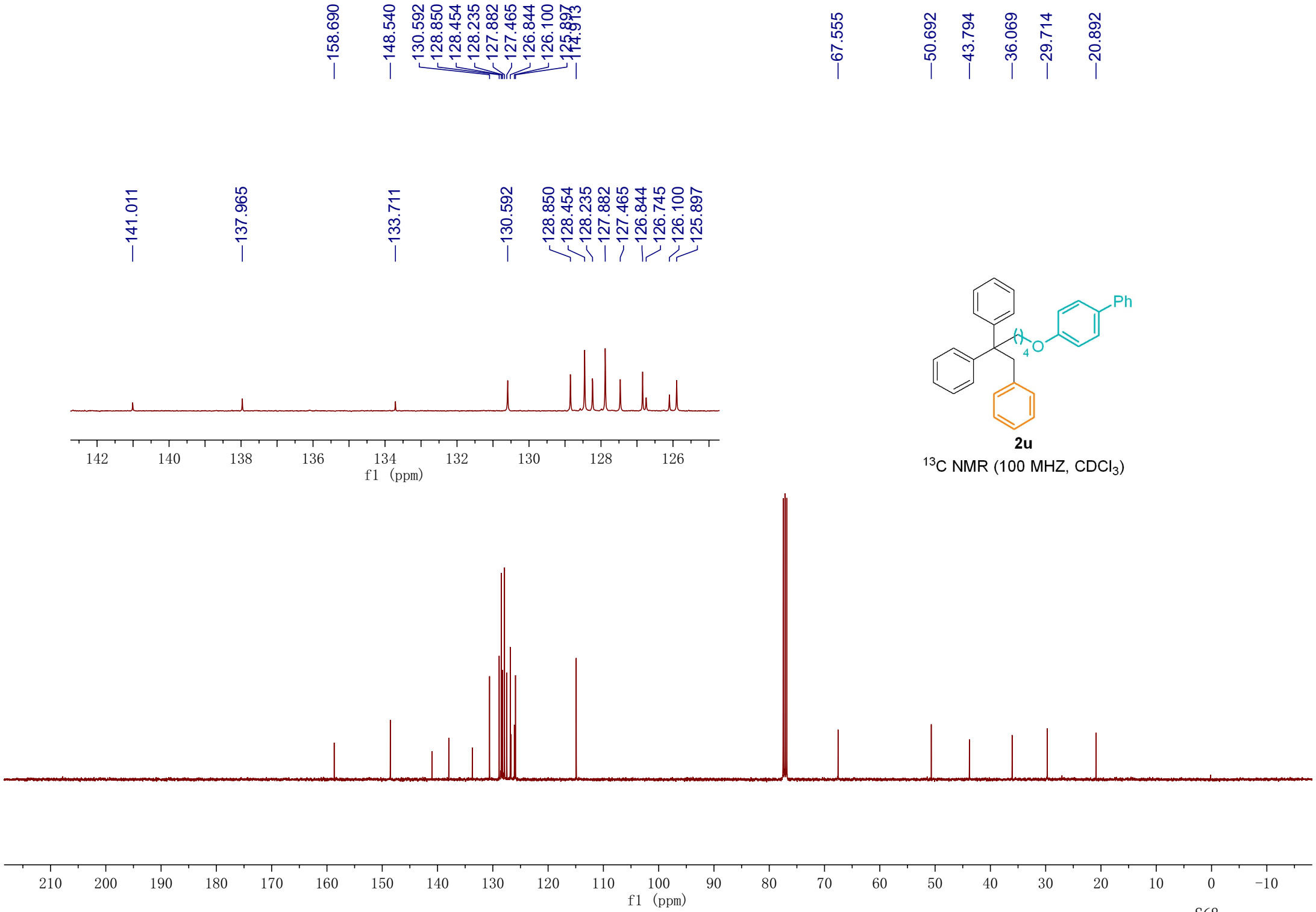

7.282
7.269
7.266
7.252
7.248
7.231
7.212
7.188
7.184
7.176
7.170
7.163
7.155
7.152
7.119
7.116
7.098
7.076
7.058
7.039
6.496
6.478
3.896
3.880
3.864
3.404
1.995
1.983
1.974
1.963
1.953
1.755
1.738
1.720
1.702
1.686
1.335
1.316
1.307
1.296
1.287
1.276
1.256
4
O
2t
1H NMR (400 MHZ, CDCl3)
6.22
2.07
5.00
3.08
2.03
2.03
2.00
2.00
2.03
2.04
2.23
f1 (ppm)

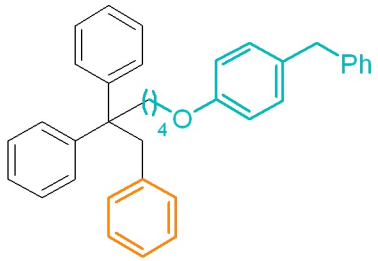


2t

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

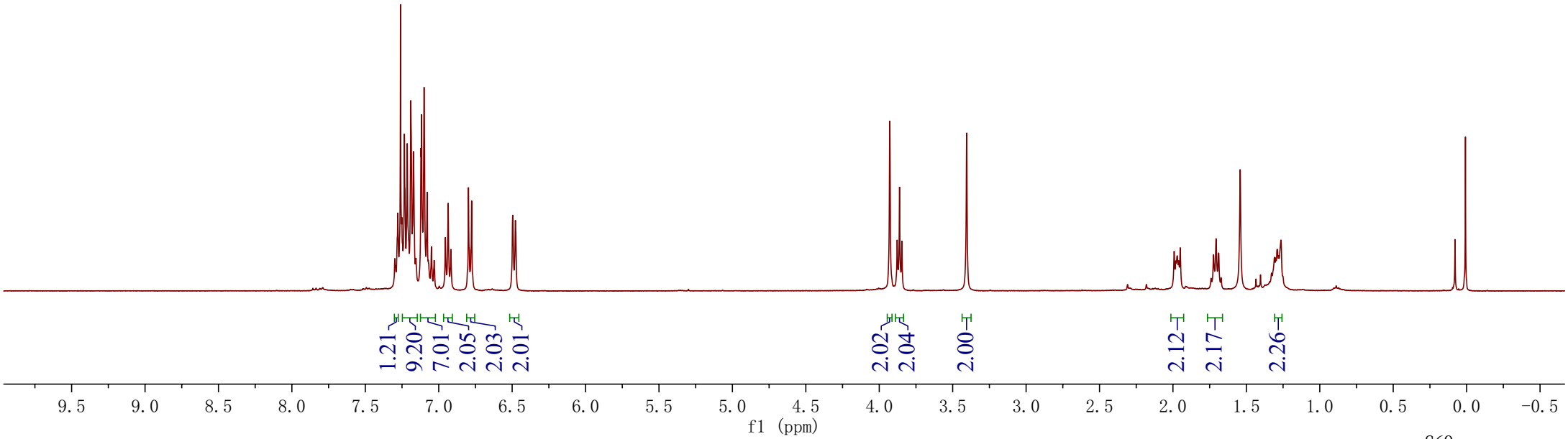

7.566
7.563
7.545
7.523
7.501
7.431
7.413
7.393
7.297
7.249
7.240
7.221
7.196
7.178
7.130
7.127
7.109
7.063
6.980
6.961
6.939
6.917
6.508
6.490
3.944
3.929
3.913
3.417
2.012
2.000
1.991
1.981
1.971
1.781
1.764
1.746
1.729
1.712
1.355
1.334
1.326
1.316
1.306
1.294
1.277
Ph
O
4
2u
1H NMR (400 MHZ, CDCl3)
4.16
2.08
1.13
4.07
2.14
4.00
1.07
4.15
2.01
2.05
2.00
2.02
2.08
2.21
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

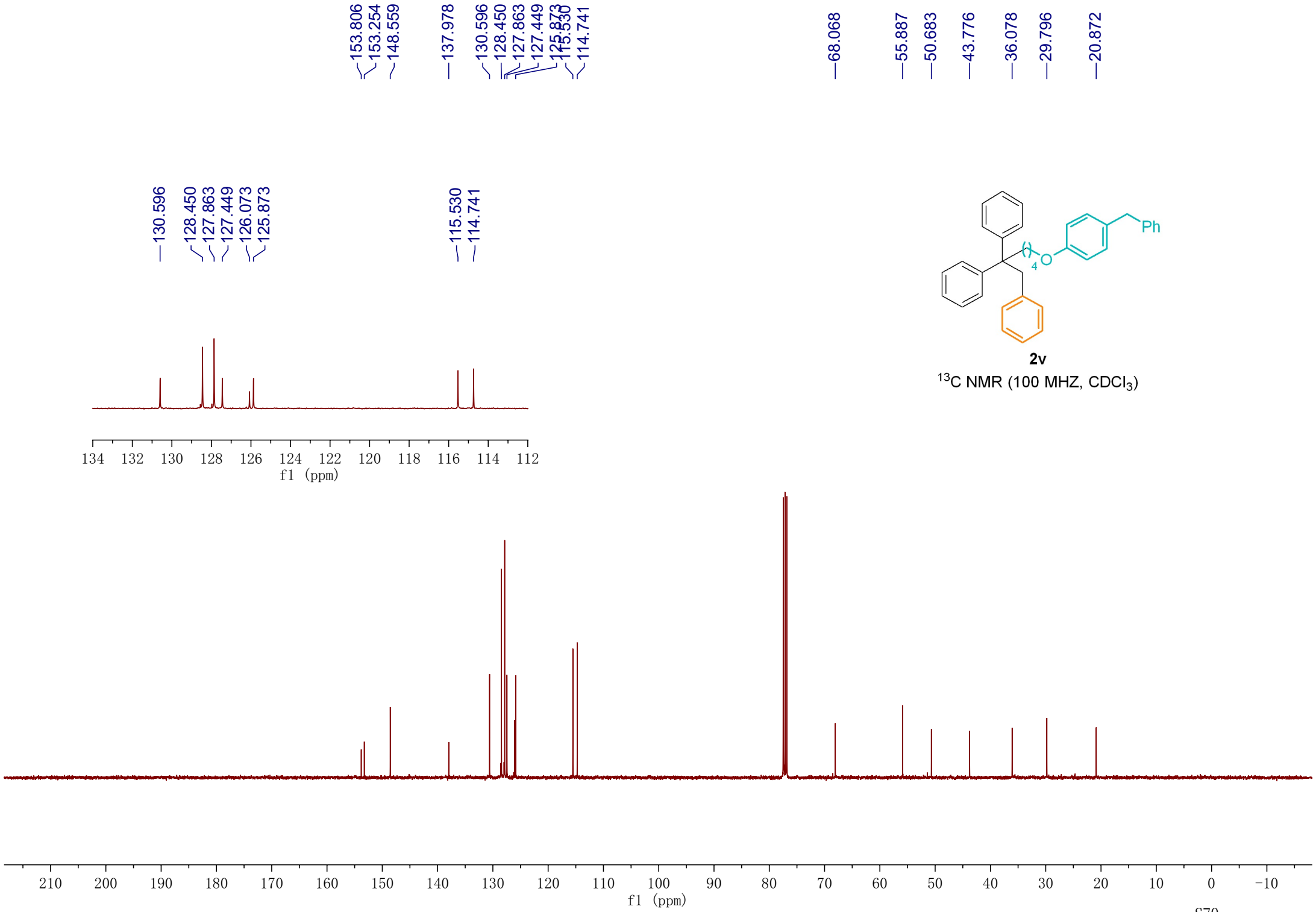

158.690
148.540
130.592
128.850
128.454
128.235
127.882
127.465
126.844
126.100
125.897
114.913
67.555
50.692
43.794
36.069
29.714
20.892
141.011
137.965
133.711
130.592
128.850
128.454
128.235
127.882
127.465
126.844
126.745
126.100
125.897
142
140
138
136
134
132
130
128
126
f1 (ppm)
Ph
O
4
2u
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



2v

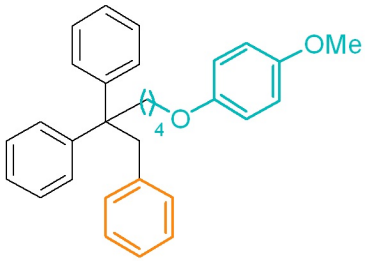
^{1}H NMR (400 MHZ, $CDCl_3$)





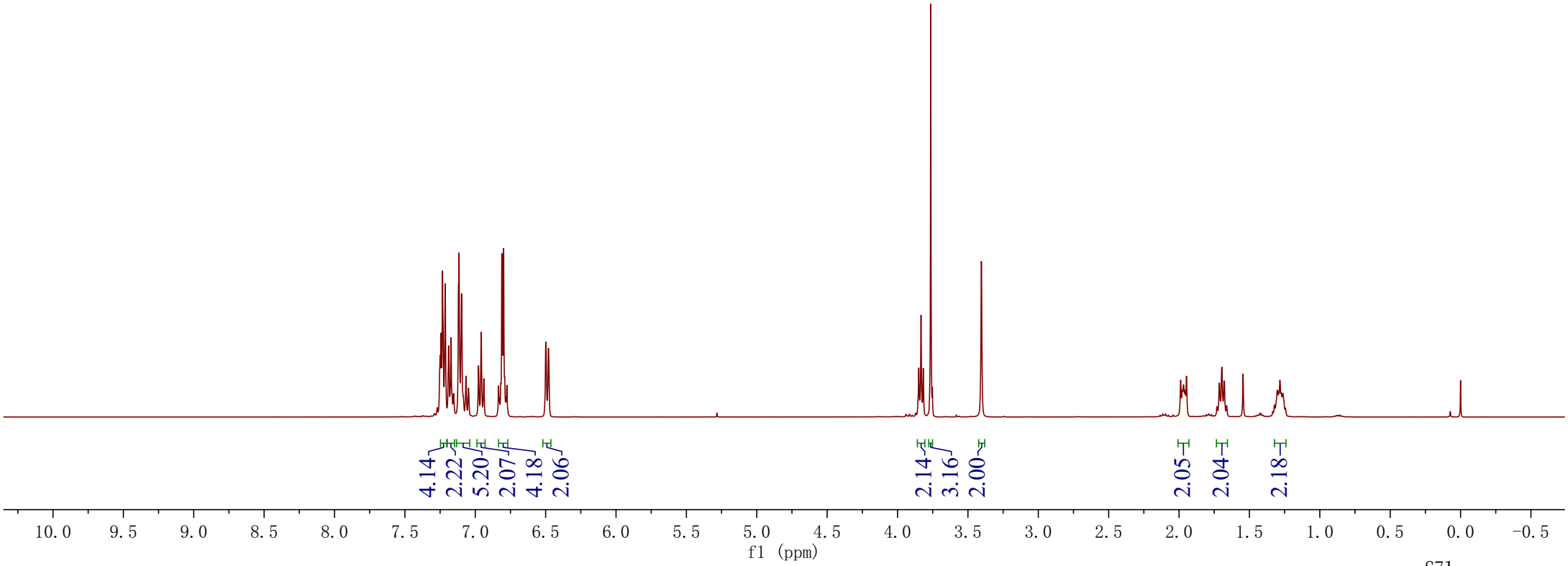
2v

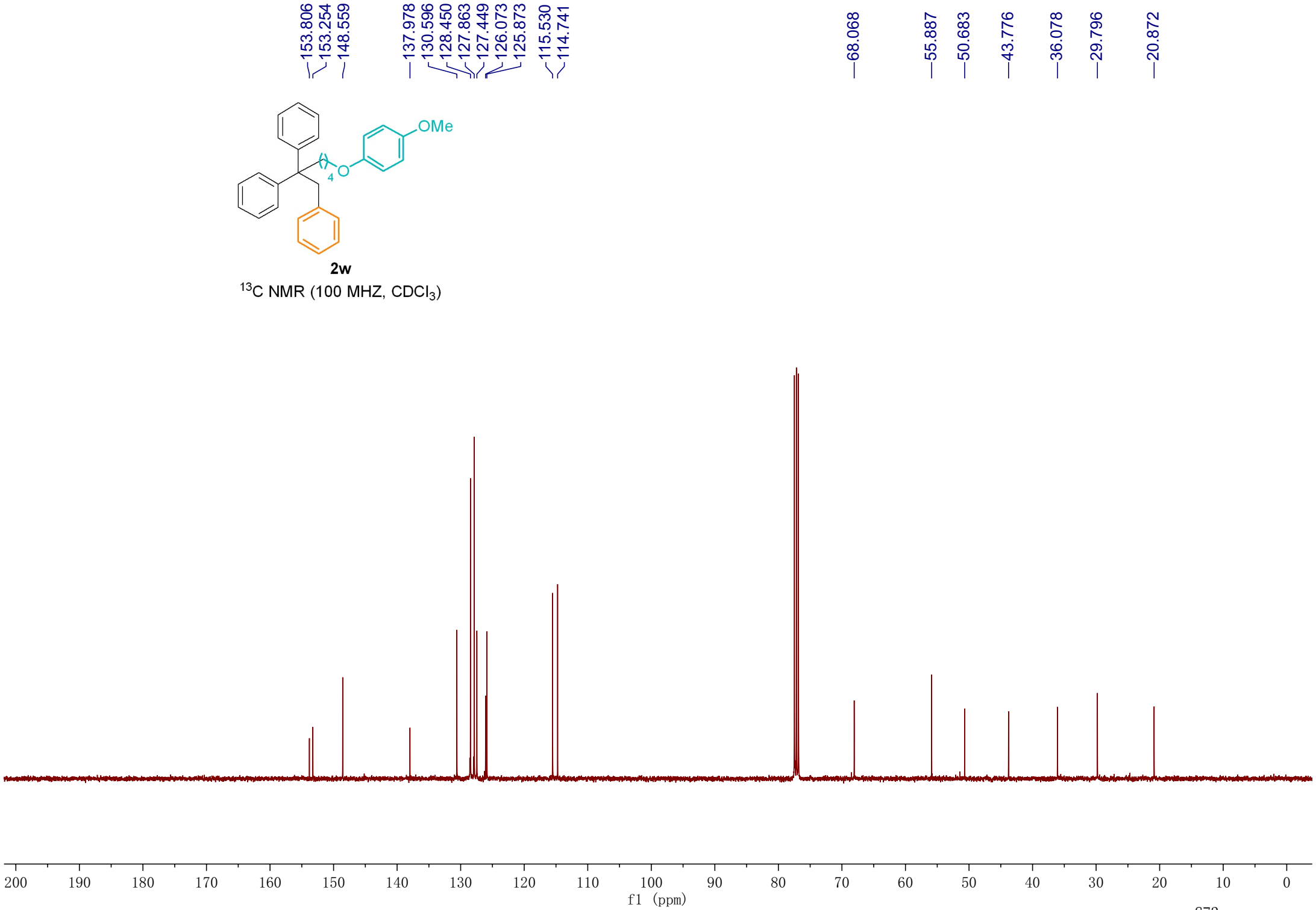
^{13}C NMR (100 MHZ, $CDCl_3$)

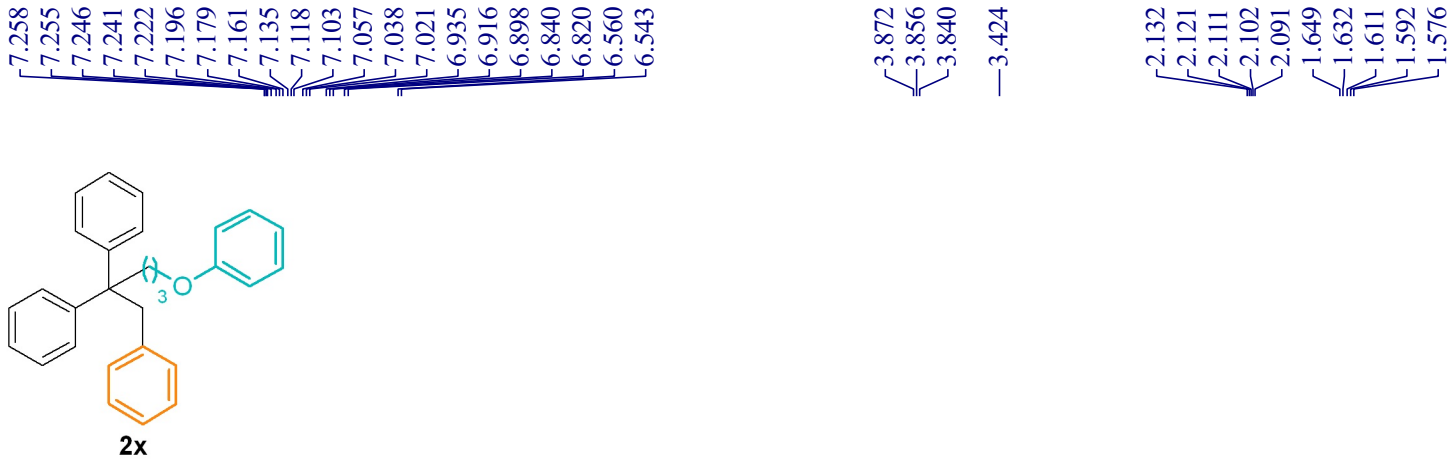


2w

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

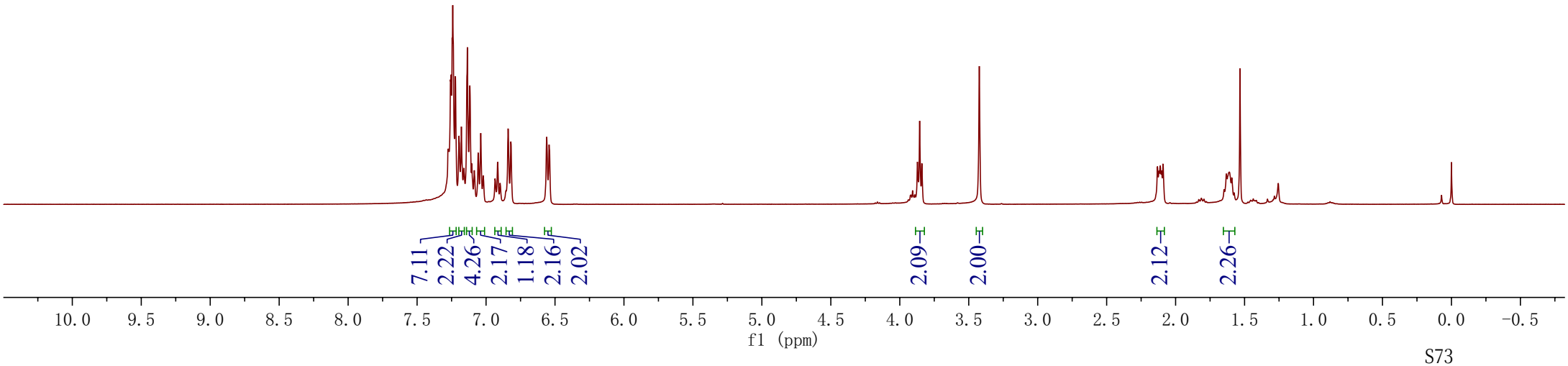


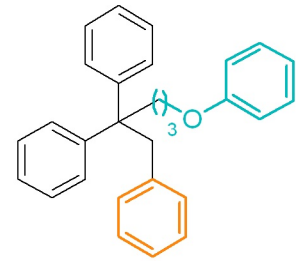

153.806
153.254
148.559
137.978
130.596
128.450
127.863
127.449
126.073
125.873
115.530
114.741
68.068
55.887
50.683
43.776
36.078
29.796
20.872
OMe
4
O
2w
13C NMR (100 MHZ, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



2x

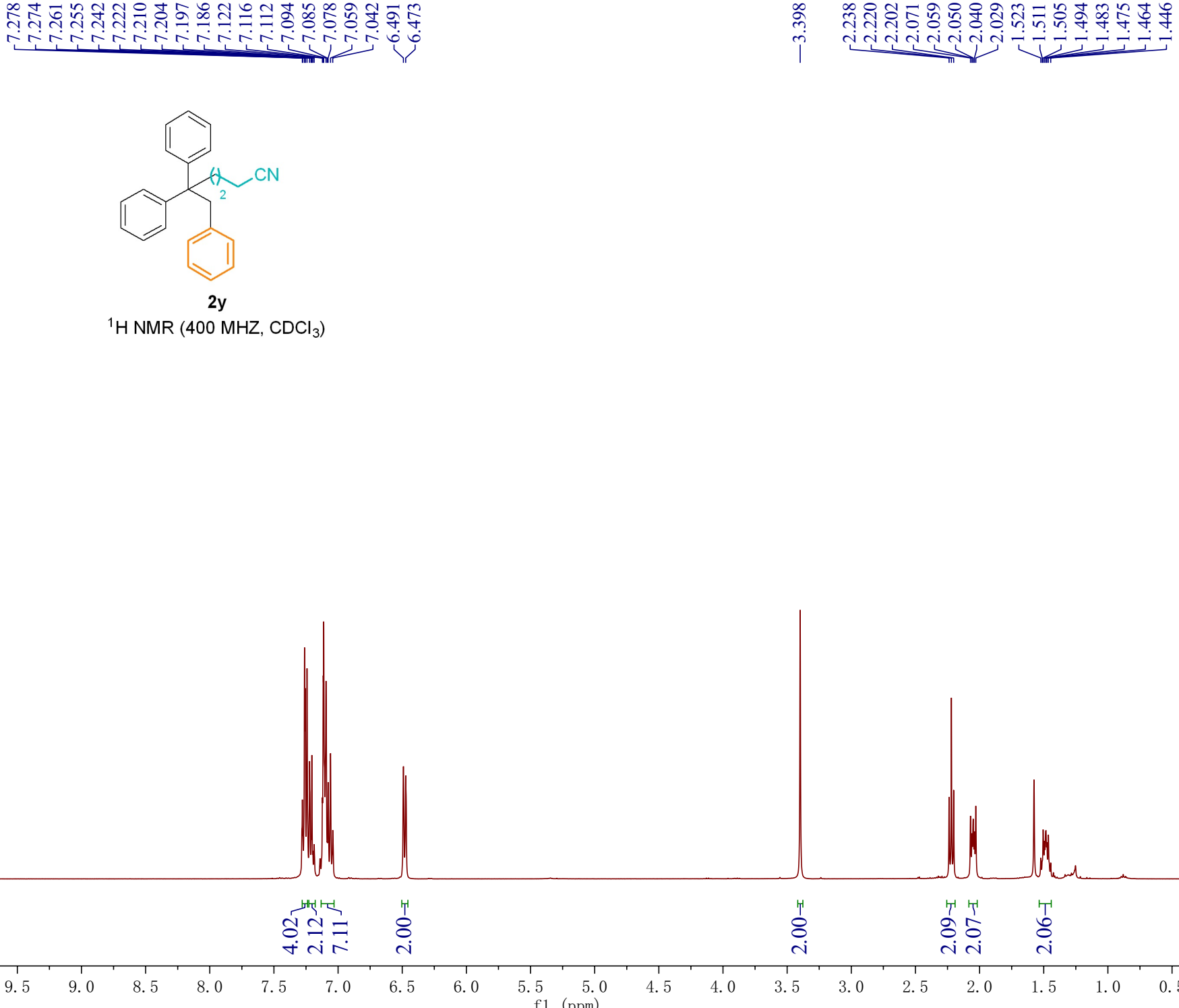
^{1}H NMR (400 MHZ, $CDCl_3$)





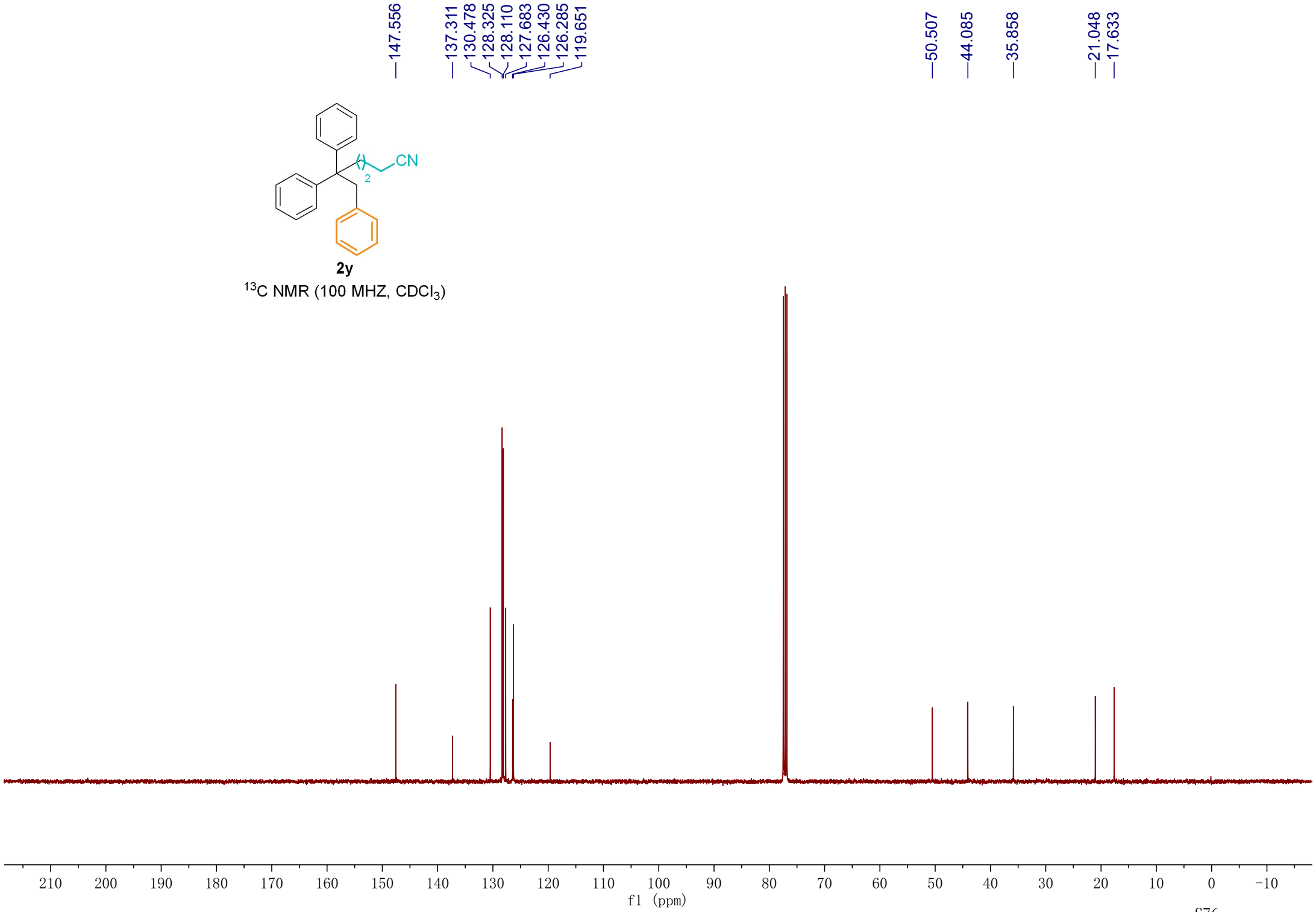
2x

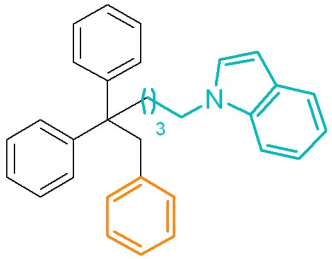
^{13}C NMR (100 MHZ, $CDCl_3$)



2y

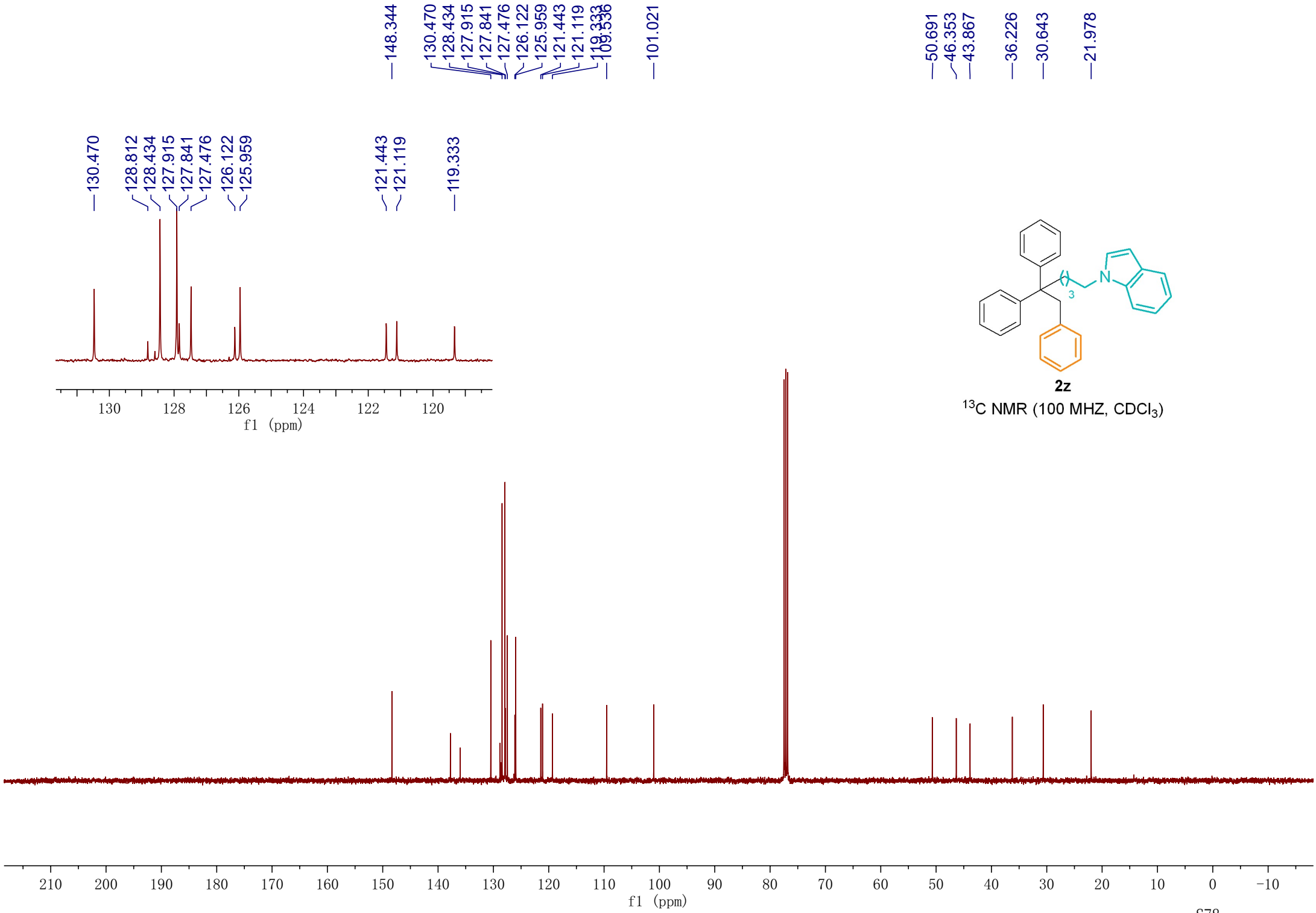
^{1}H NMR (400 MHZ, $CDCl_3$)

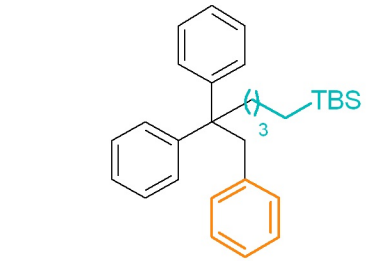

147.556
137.311
130.478
128.325
128.110
127.683
126.430
126.285
119.651
50.507
44.085
35.858
21.048
17.633
CN
2
2y
13C NMR (100 MHZ, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



2z

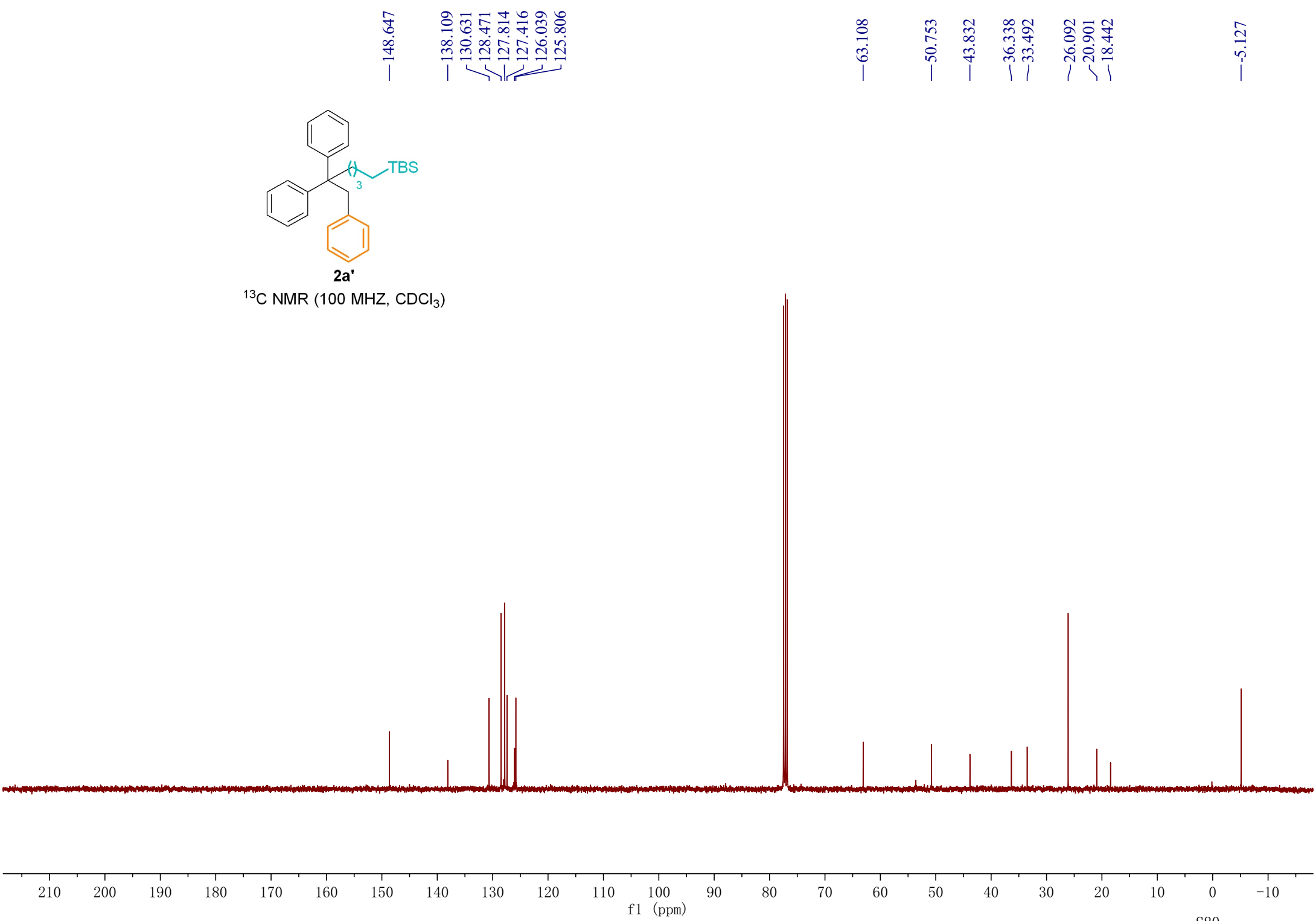
^{1}H NMR (400 MHZ, $CDCl_3$)

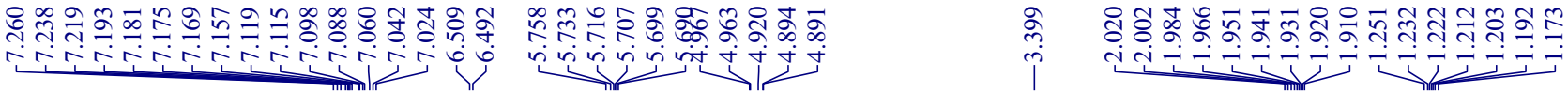

148.344
130.470
128.434
127.915
127.841
127.476
126.122
125.959
121.443
121.119
119.333
109.536
101.021
50.691
46.353
43.867
36.226
30.643
21.978
130.470
128.812
128.434
127.915
127.841
127.476
126.122
125.959
121.443
121.119
119.333
130
128
126
124
122
120
f1 (ppm)
N
3
2z
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

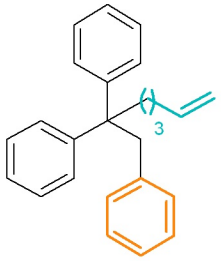


2a'

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

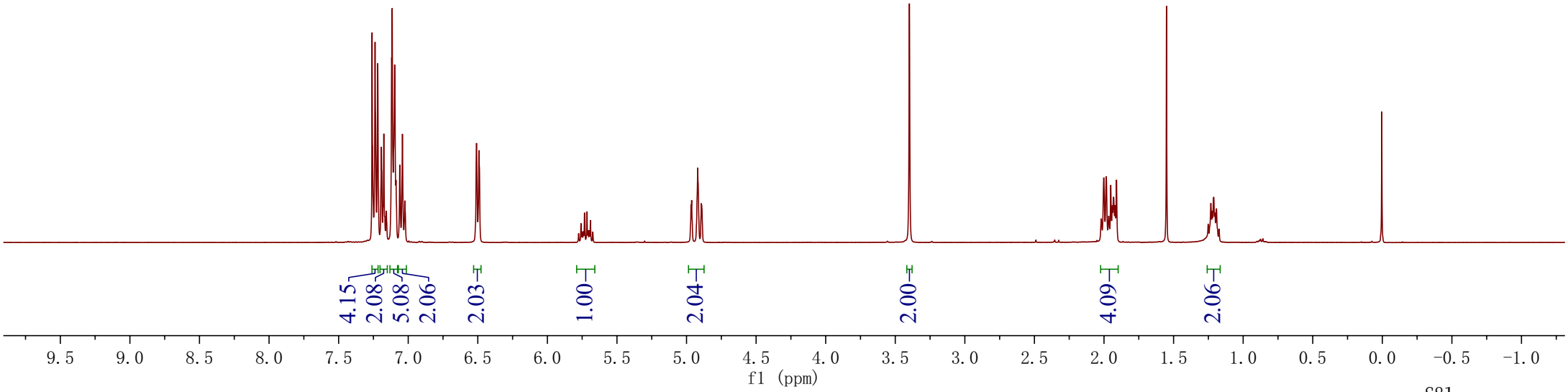

148.647
138.109
130.631
128.471
127.814
127.416
126.039
125.806
63.108
50.753
43.832
36.338
33.492
26.092
20.901
18.442
-5.127
TBS
3
2a'
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

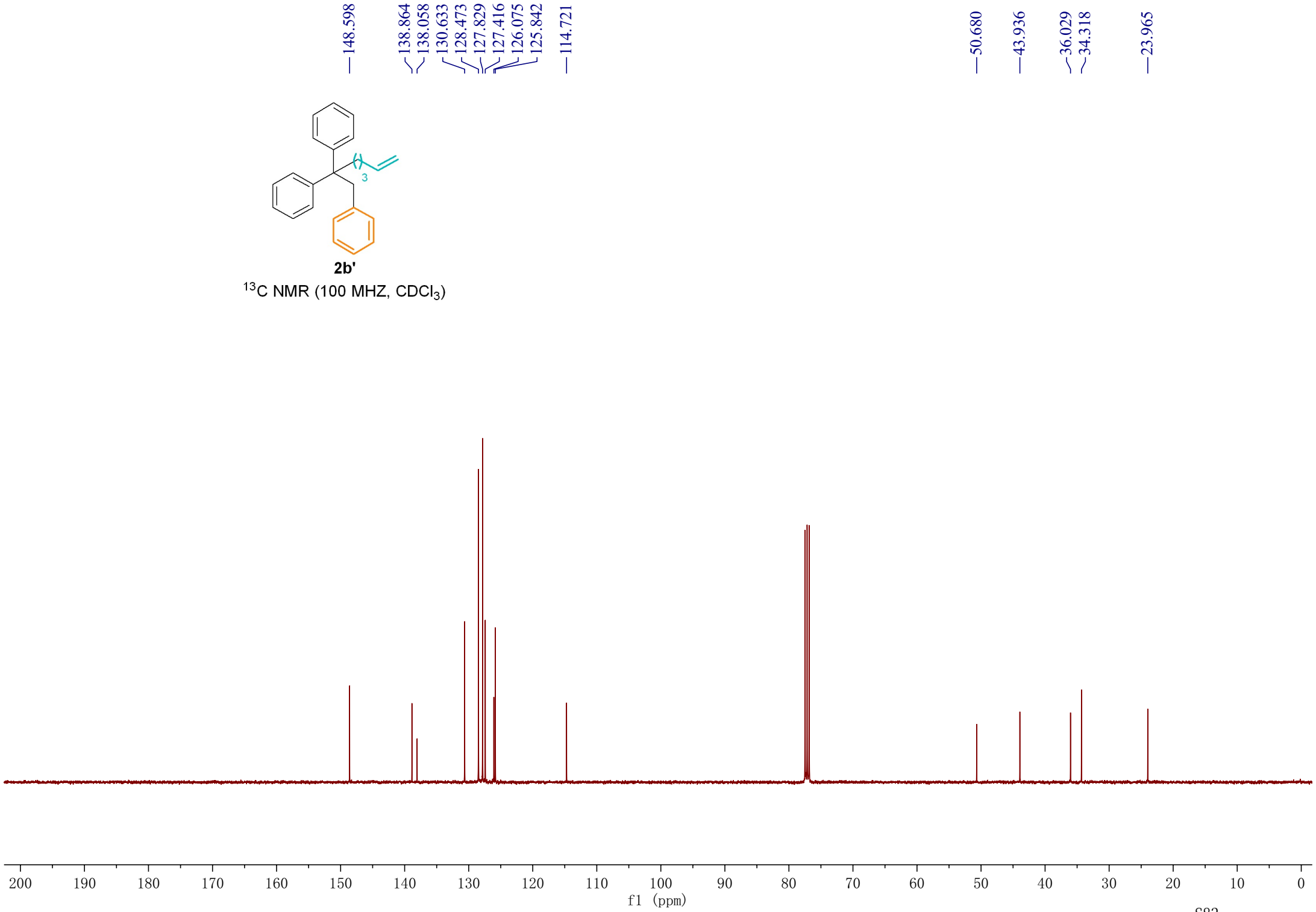


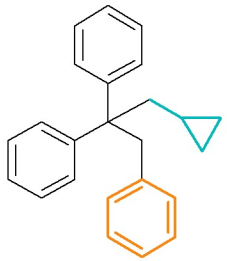


2b'

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

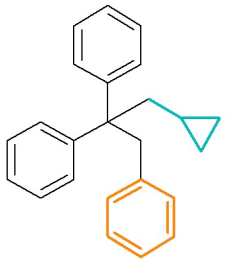



148.598
138.864
138.058
130.633
128.473
127.829
127.416
126.075
125.842
114.721
50.680
43.936
36.029
34.318
23.965
3
2b'
13C NMR (100 MHZ, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



2c'

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

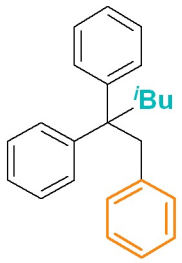


2c'

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

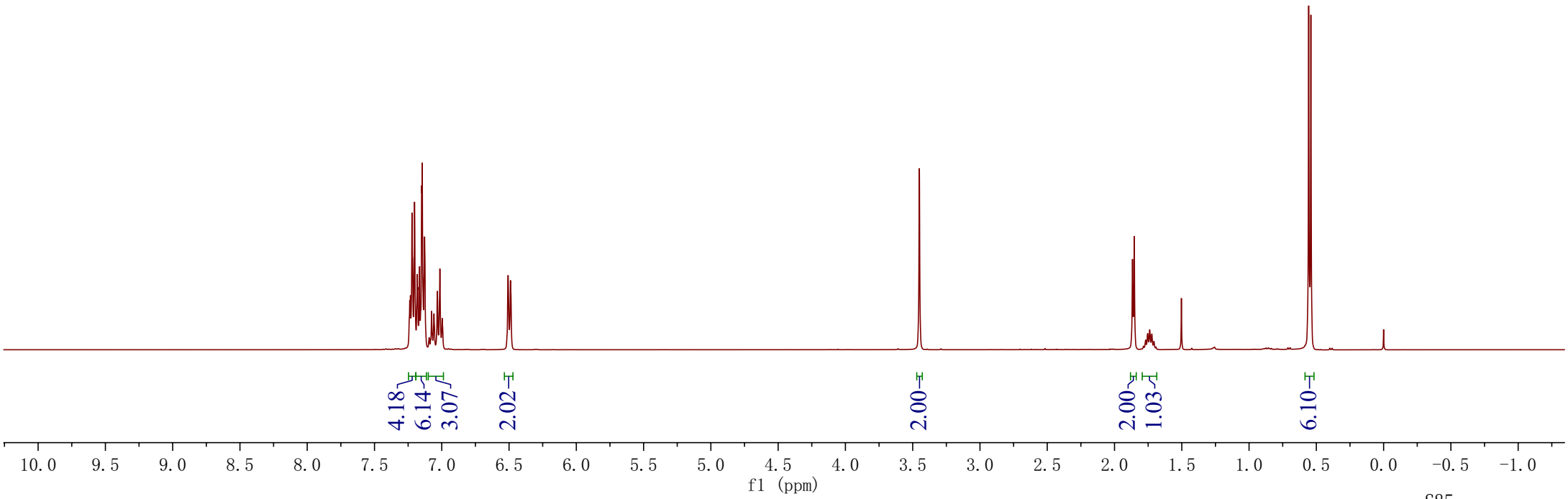
148.686
138.209
130.850
128.817
127.652
127.394
126.001
125.812
51.679
44.612
41.918
6.801
5.172

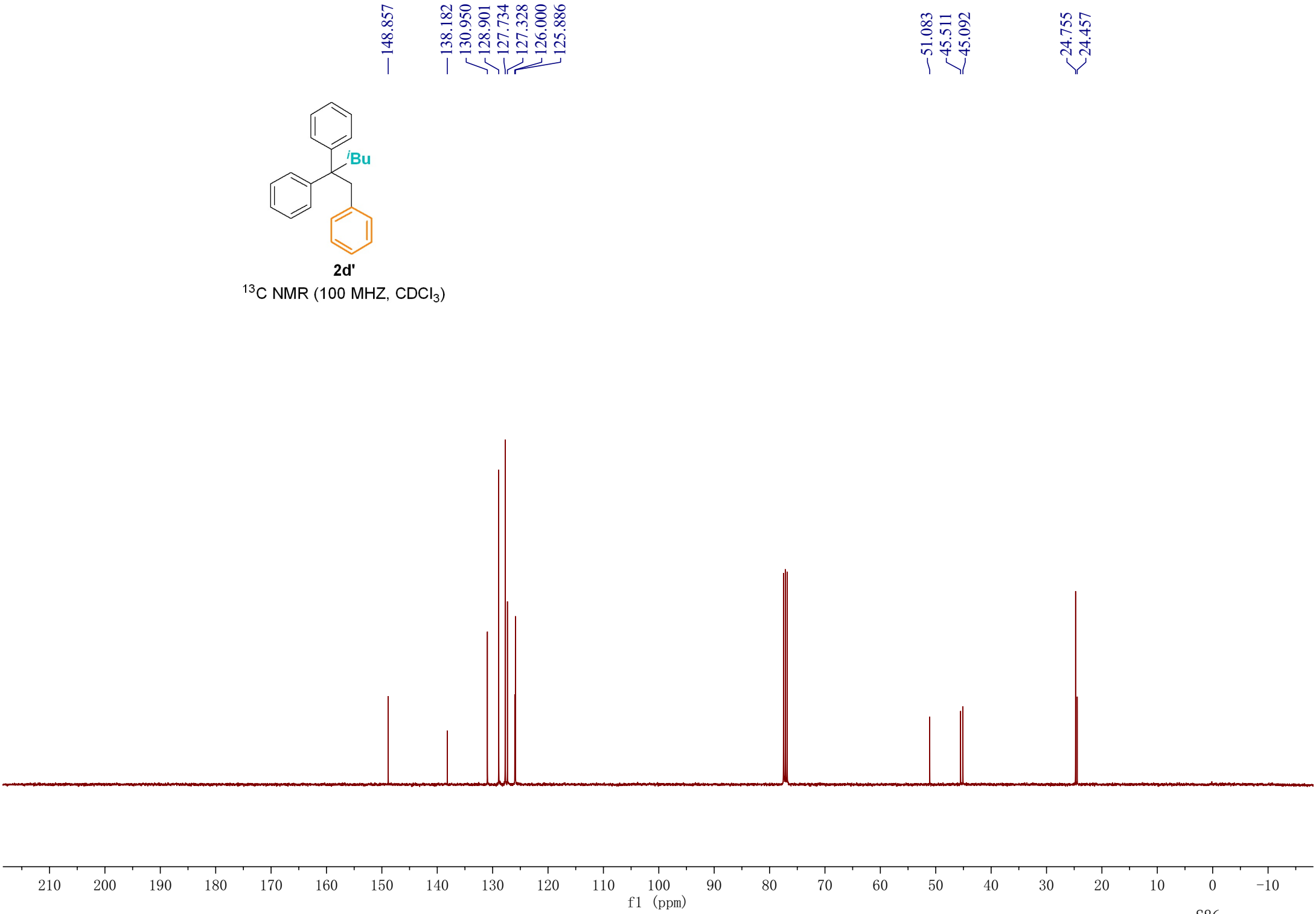
f1 (ppm)

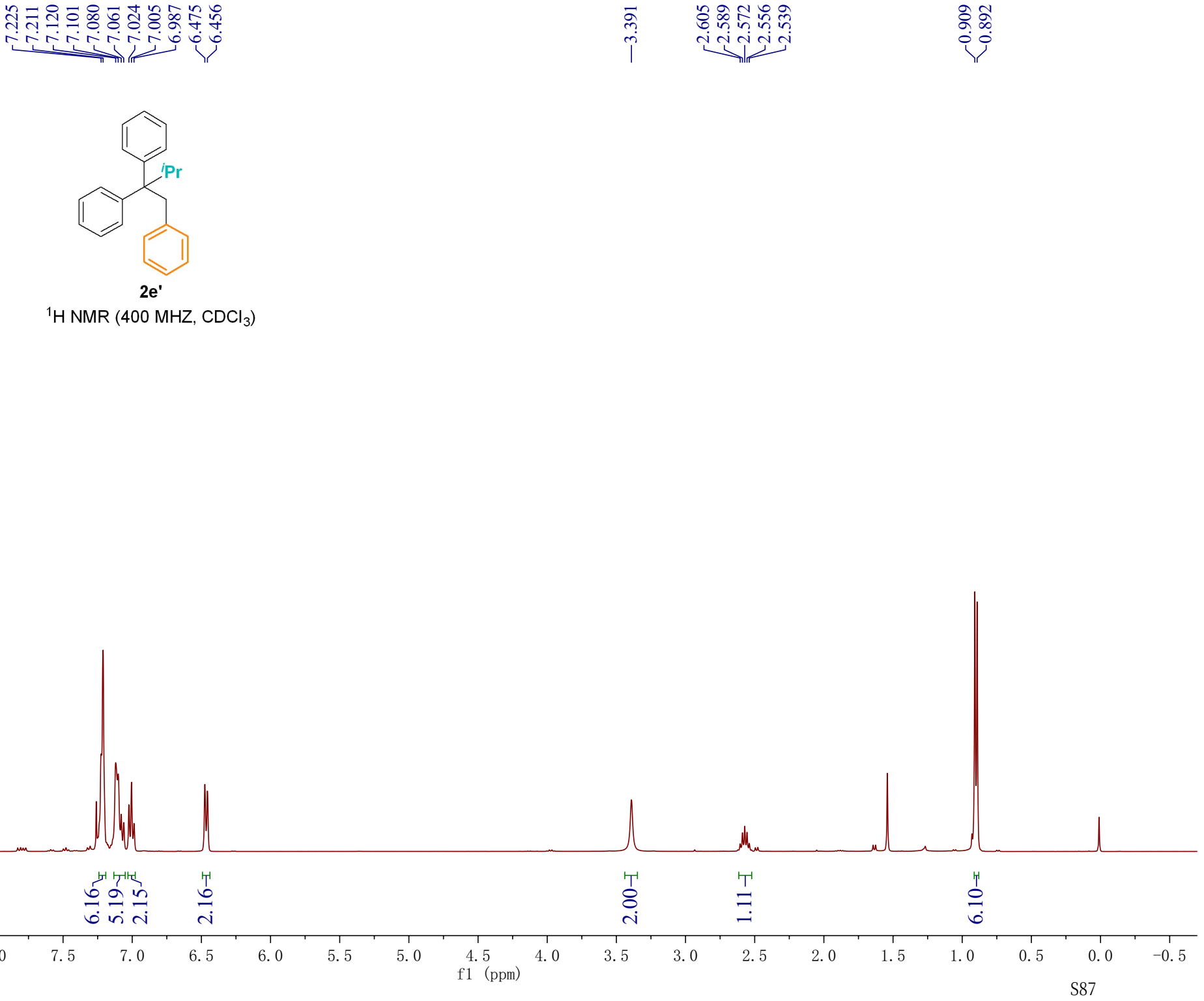


2d'

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

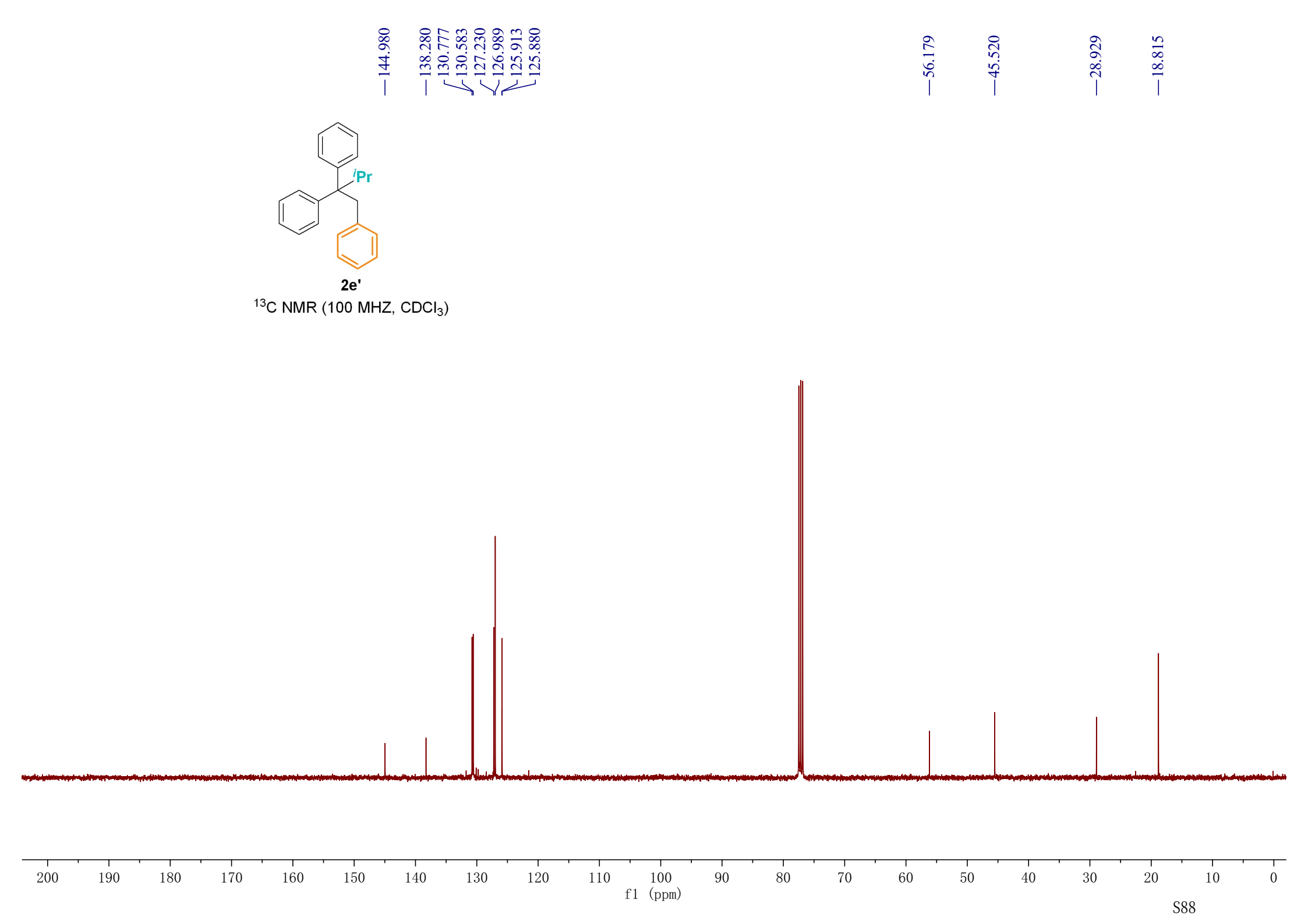


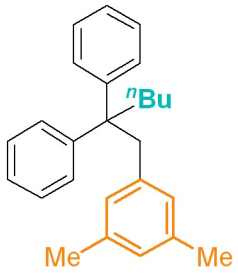

148.857
138.182
130.950
128.901
127.734
127.328
126.000
125.886
51.083
45.511
45.092
24.755
24.457
iBu
2d'
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



2e'

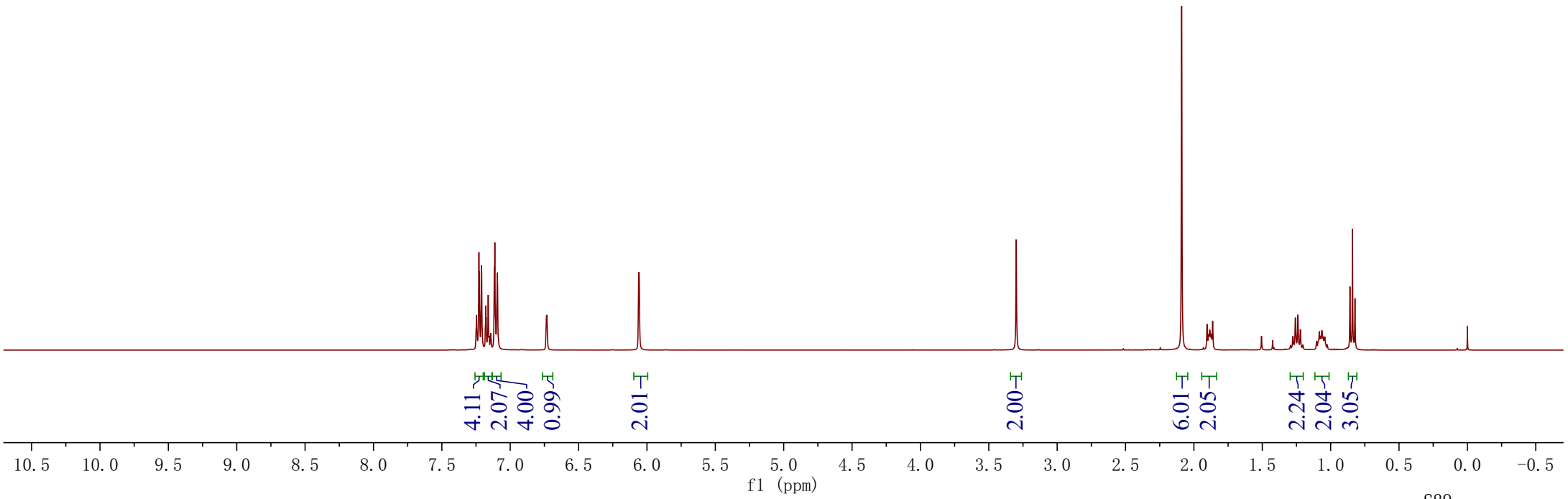
^{1}H NMR (400 MHZ, $CDCl_3$)

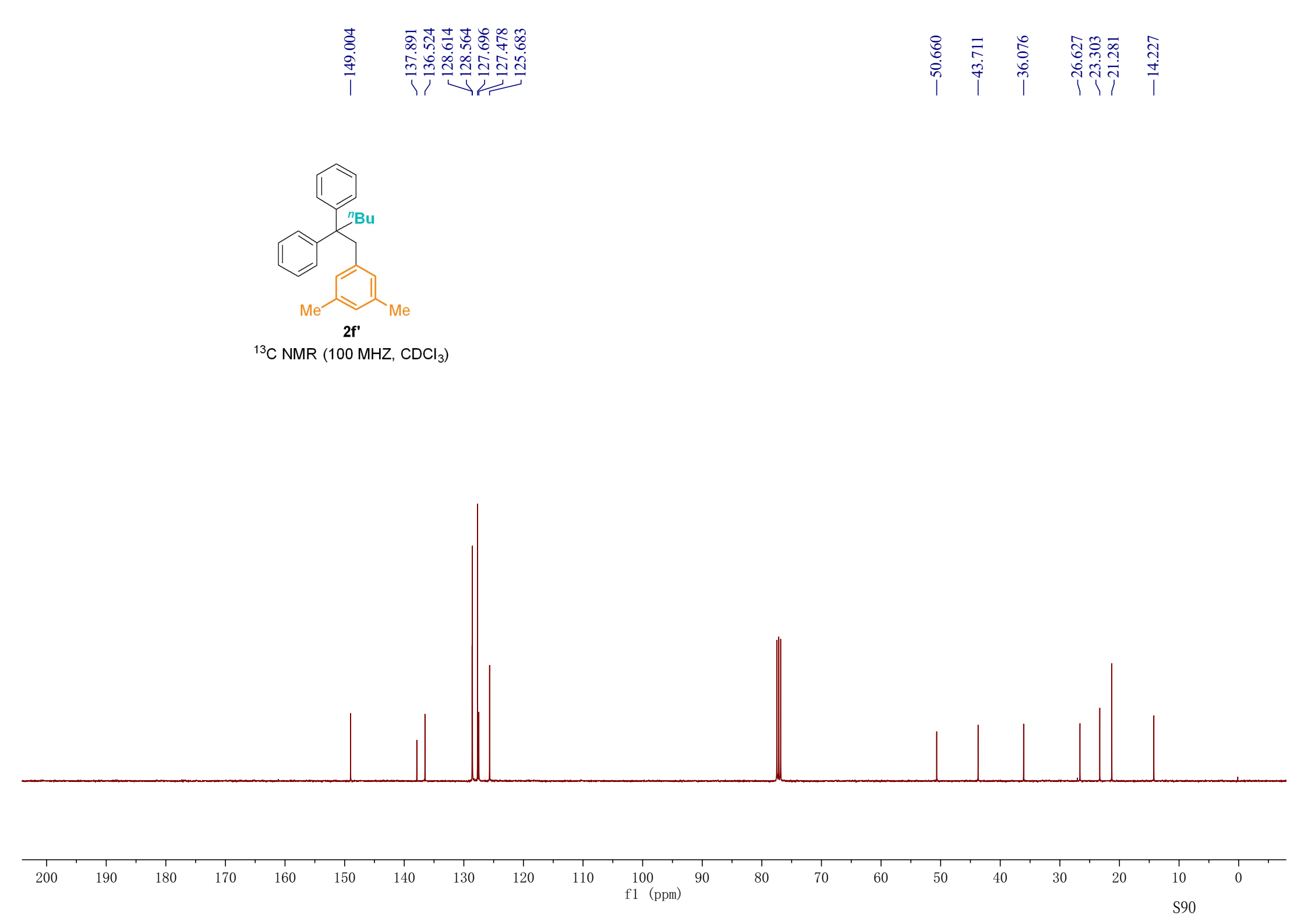

144.980
138.280
130.777
130.583
127.230
126.989
125.913
125.880
56.179
45.520
28.929
18.815
iPr
2e'
13C NMR (100 MHZ, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



2f'

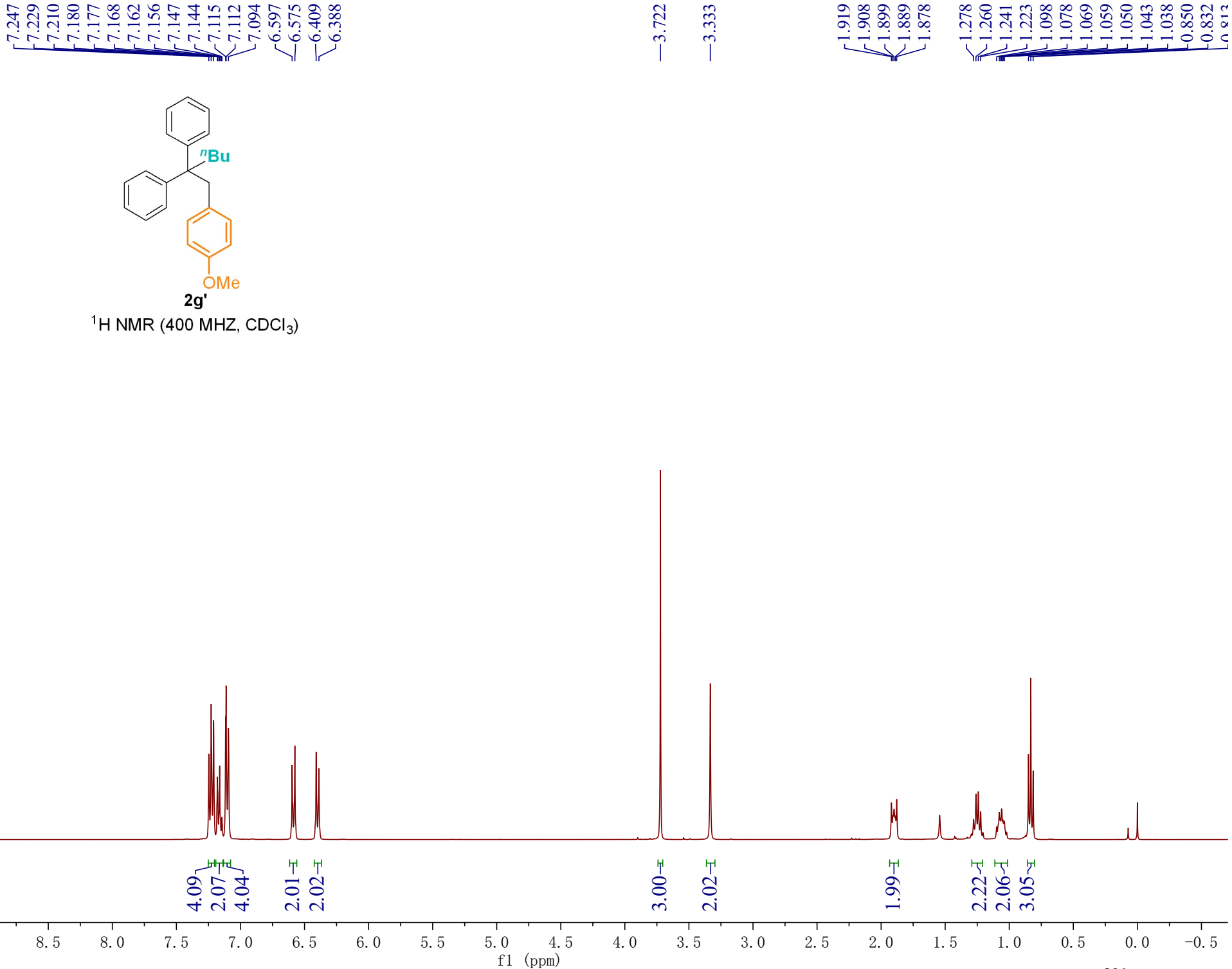
^{1}H NMR (400 MHZ, $CDCl_3$)

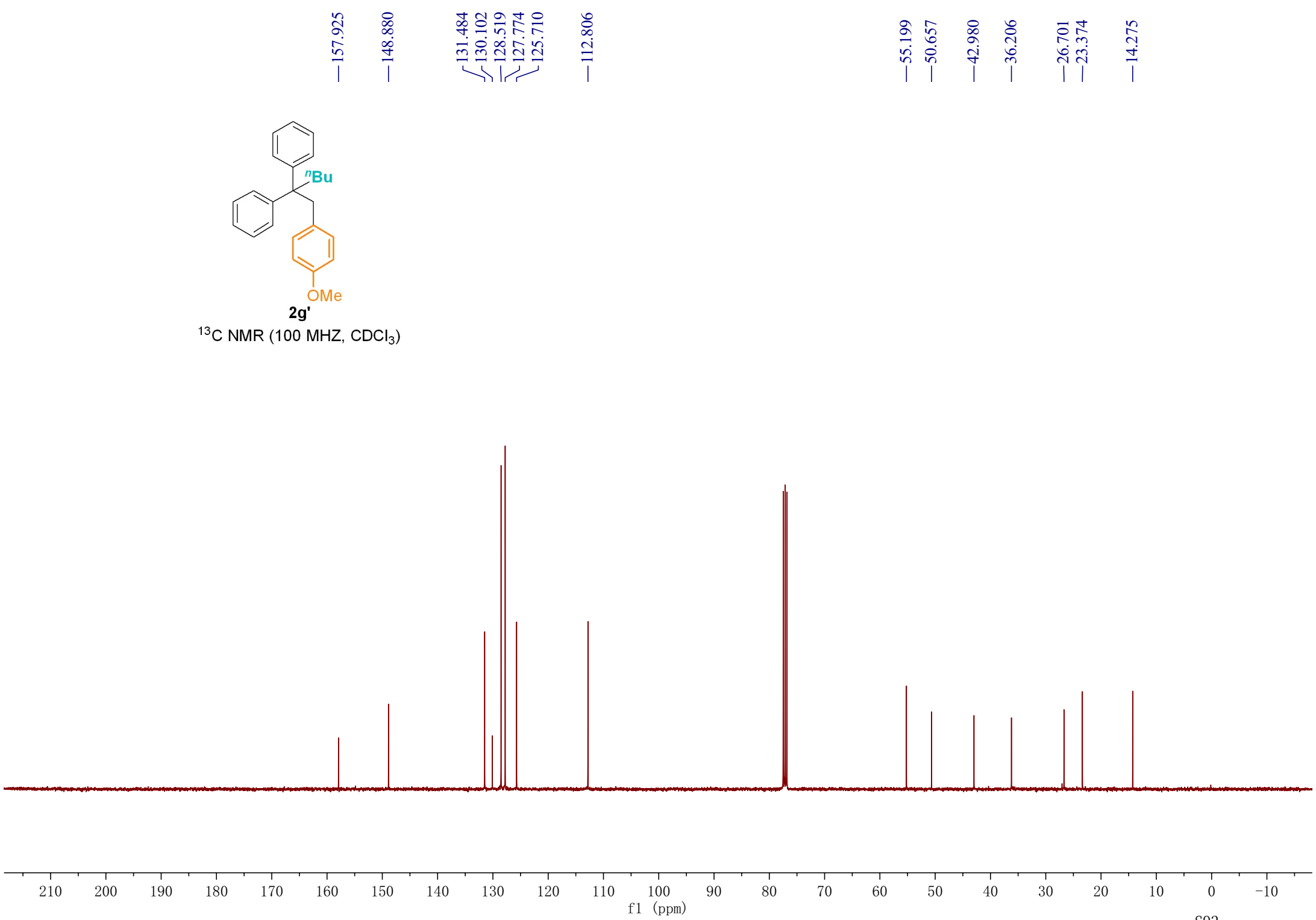


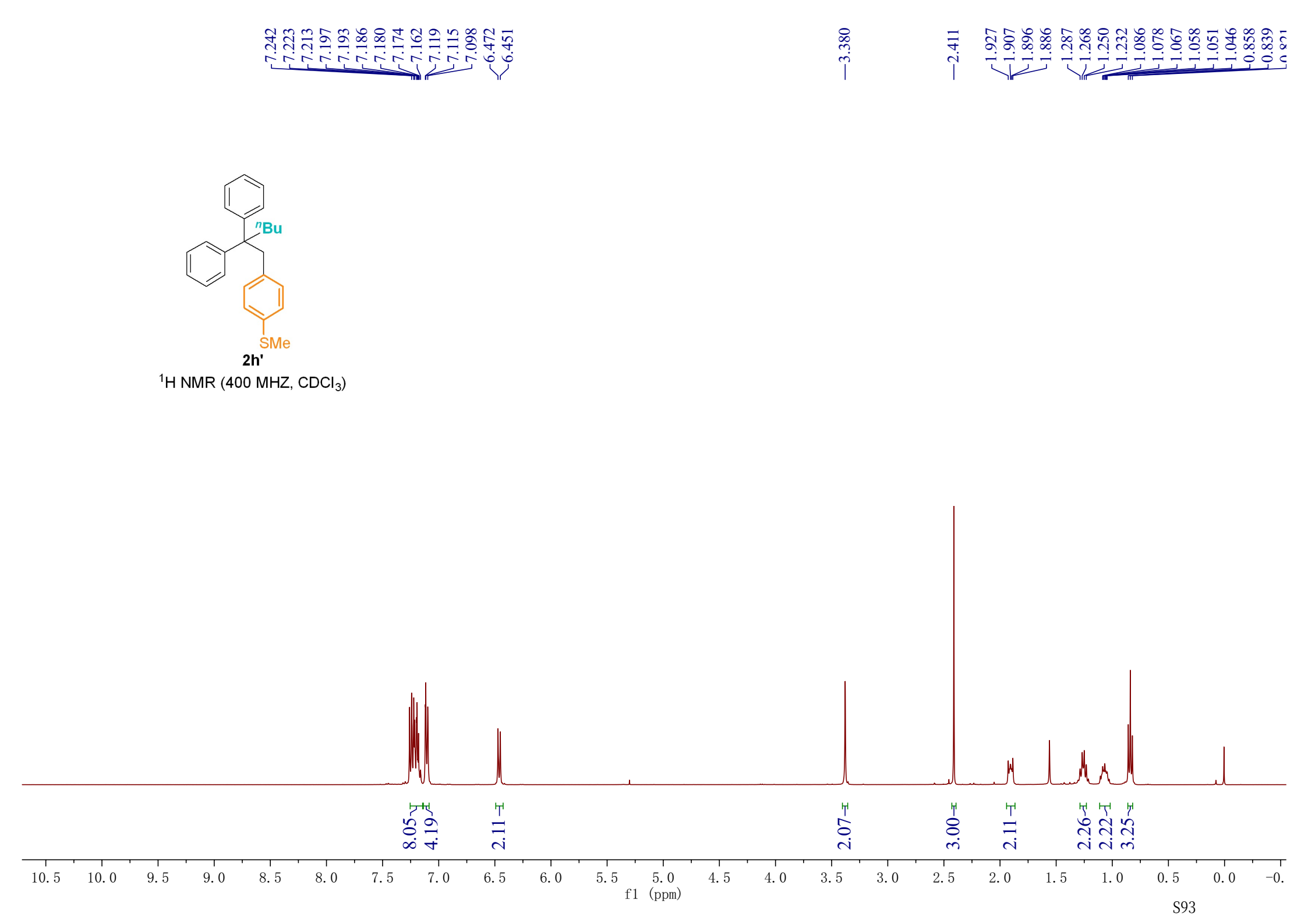


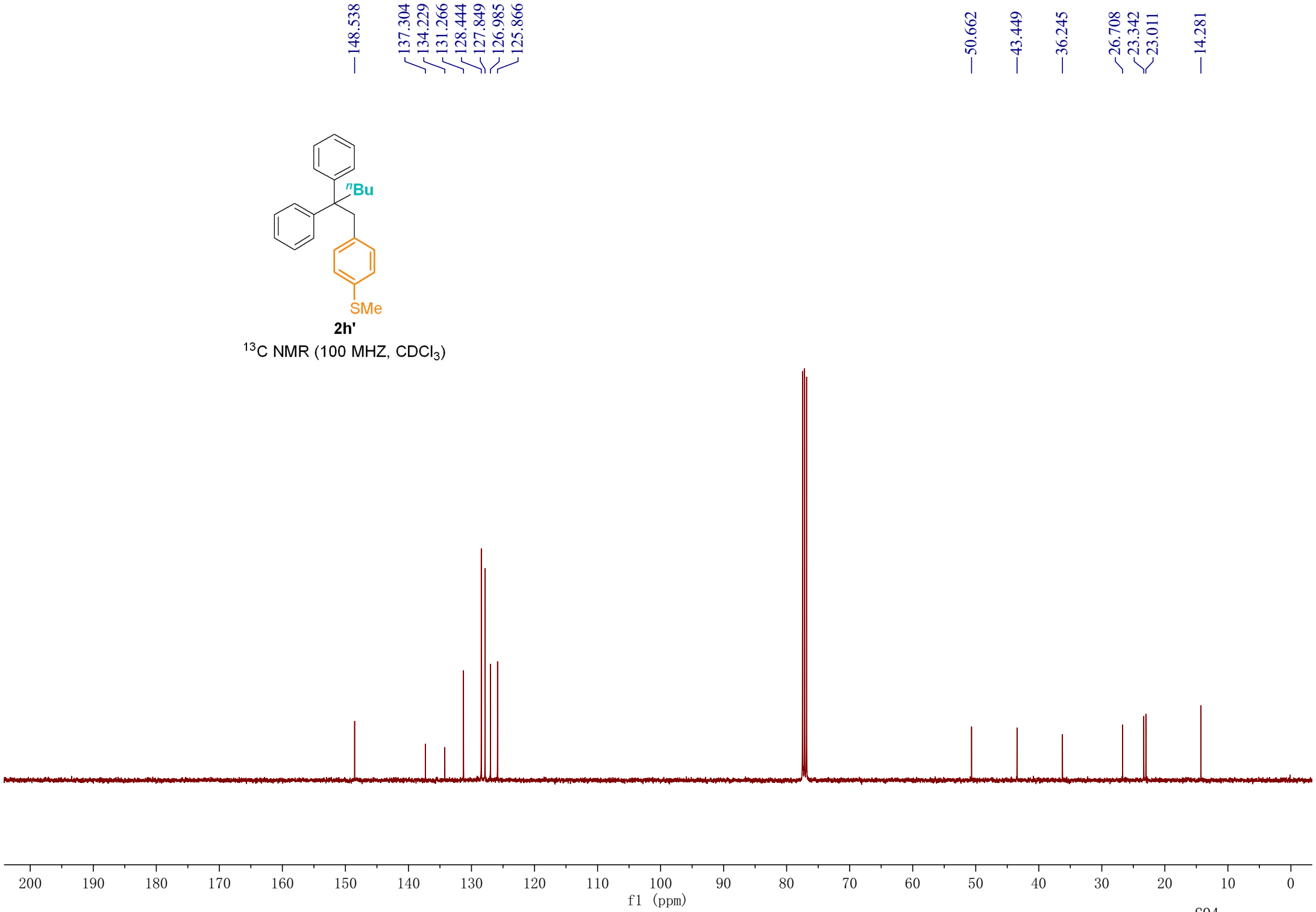
2f'

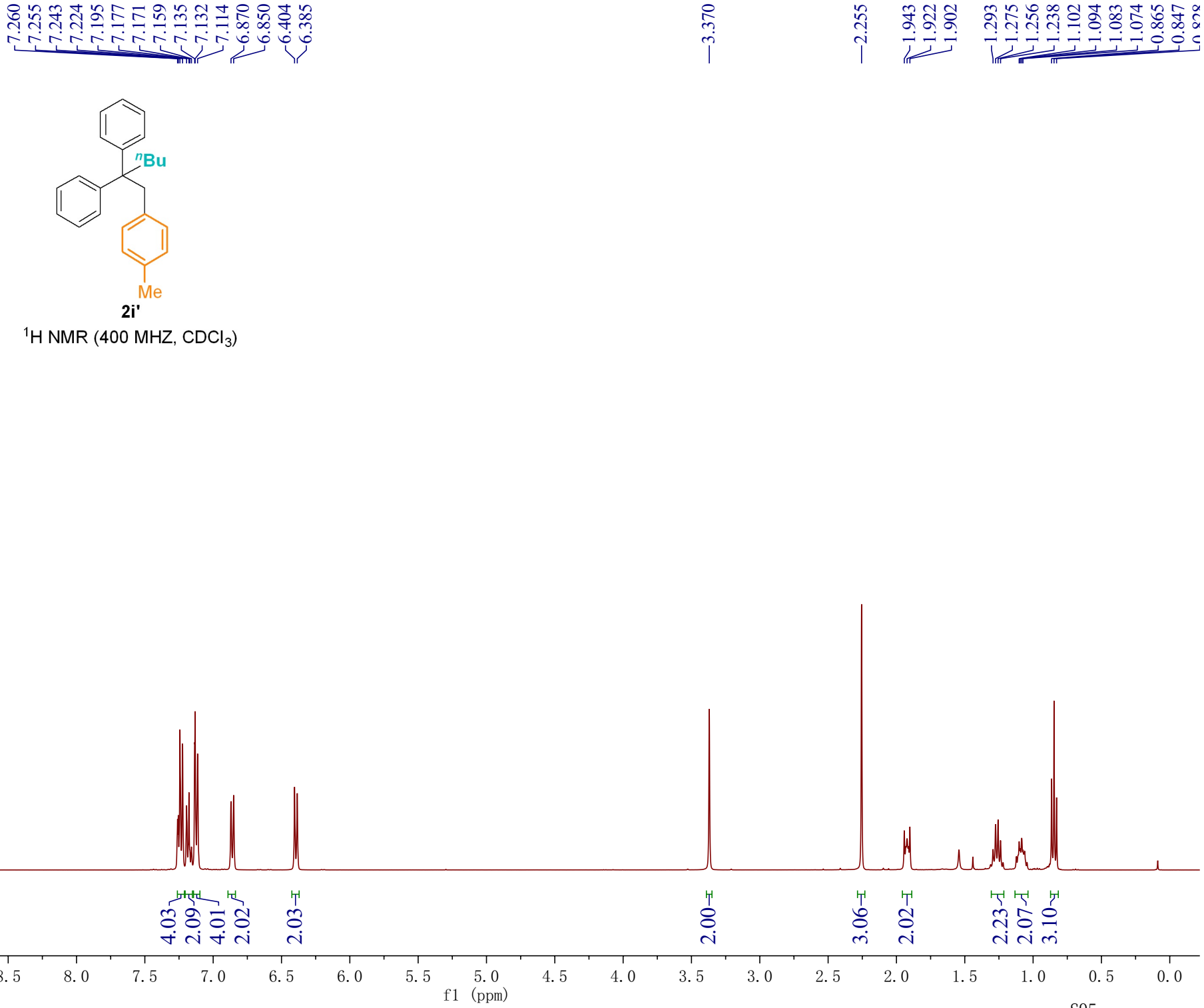
^{13}C NMR (100 MHZ, $CDCl_3$)


7.247
7.229
7.210
7.180
7.177
7.168
7.162
7.156
7.147
7.144
7.115
7.112
7.094
6.597
6.575
6.409
6.388
3.722
3.333
1.919
1.908
1.899
1.889
1.878
1.278
1.260
1.241
1.223
1.098
1.078
1.069
1.059
1.050
1.043
1.038
0.850
0.832
nBu
OMe
2g'
1H NMR (400 MHZ, CDCl3)
4.09
2.07
4.04
2.01
2.02
3.00
2.02
1.99
2.22
2.06
3.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


157.925
148.880
131.484
130.102
128.519
127.774
125.710
112.806
55.199
50.657
42.980
36.206
26.701
23.374
14.275
nBu
OMe
2g'
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

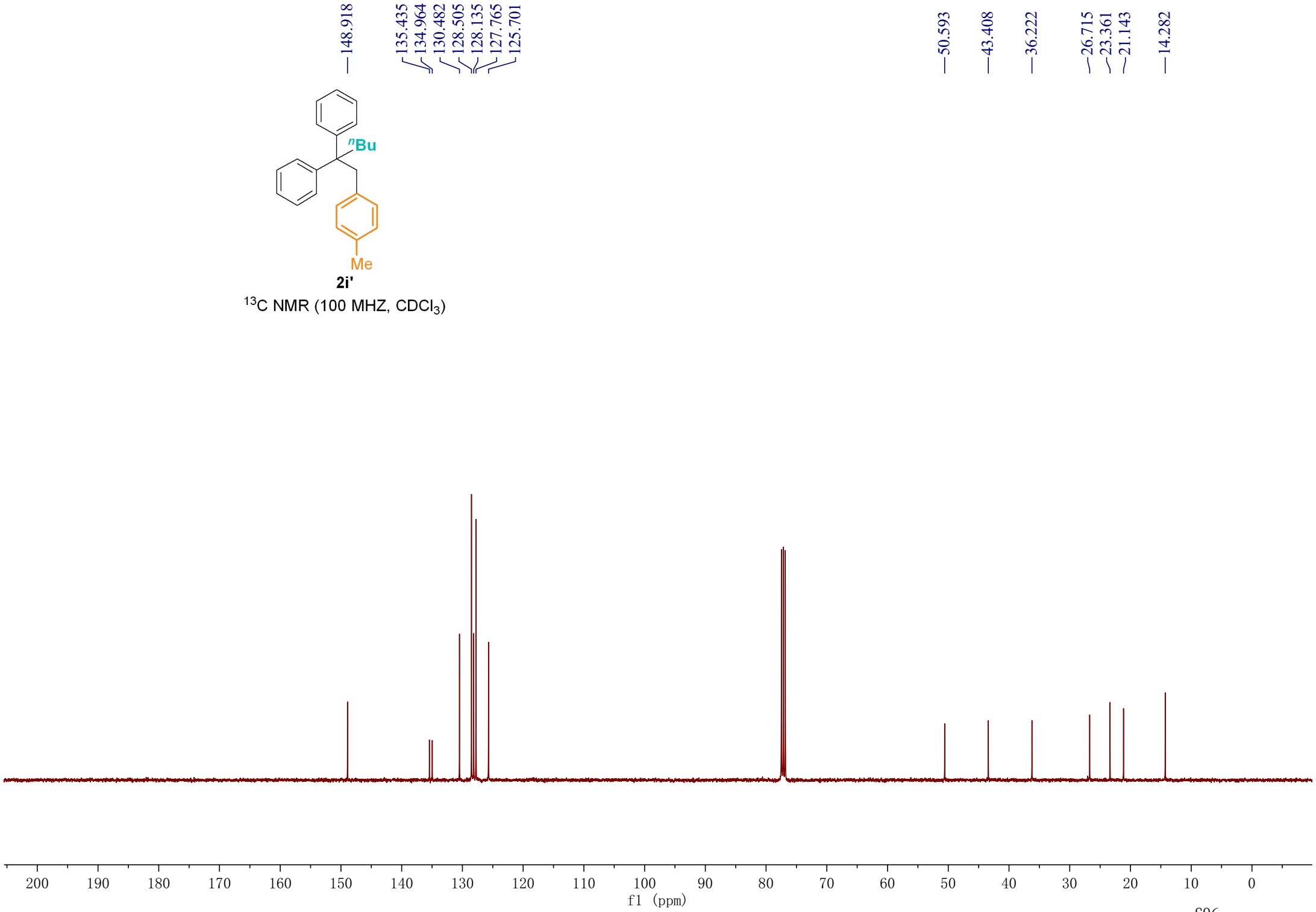

7.242
7.223
7.213
7.197
7.193
7.186
7.180
7.174
7.162
7.119
7.115
7.098
6.472
6.451
3.380
2.411
1.927
1.907
1.896
1.886
1.287
1.268
1.250
1.232
1.086
1.078
1.067
1.058
1.051
1.046
0.858
0.839
nBu
SMe
2h'
1H NMR (400 MHZ, CDCl3)
8.05
4.19
2.11
2.07
3.00
2.11
2.26
2.22
3.25
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

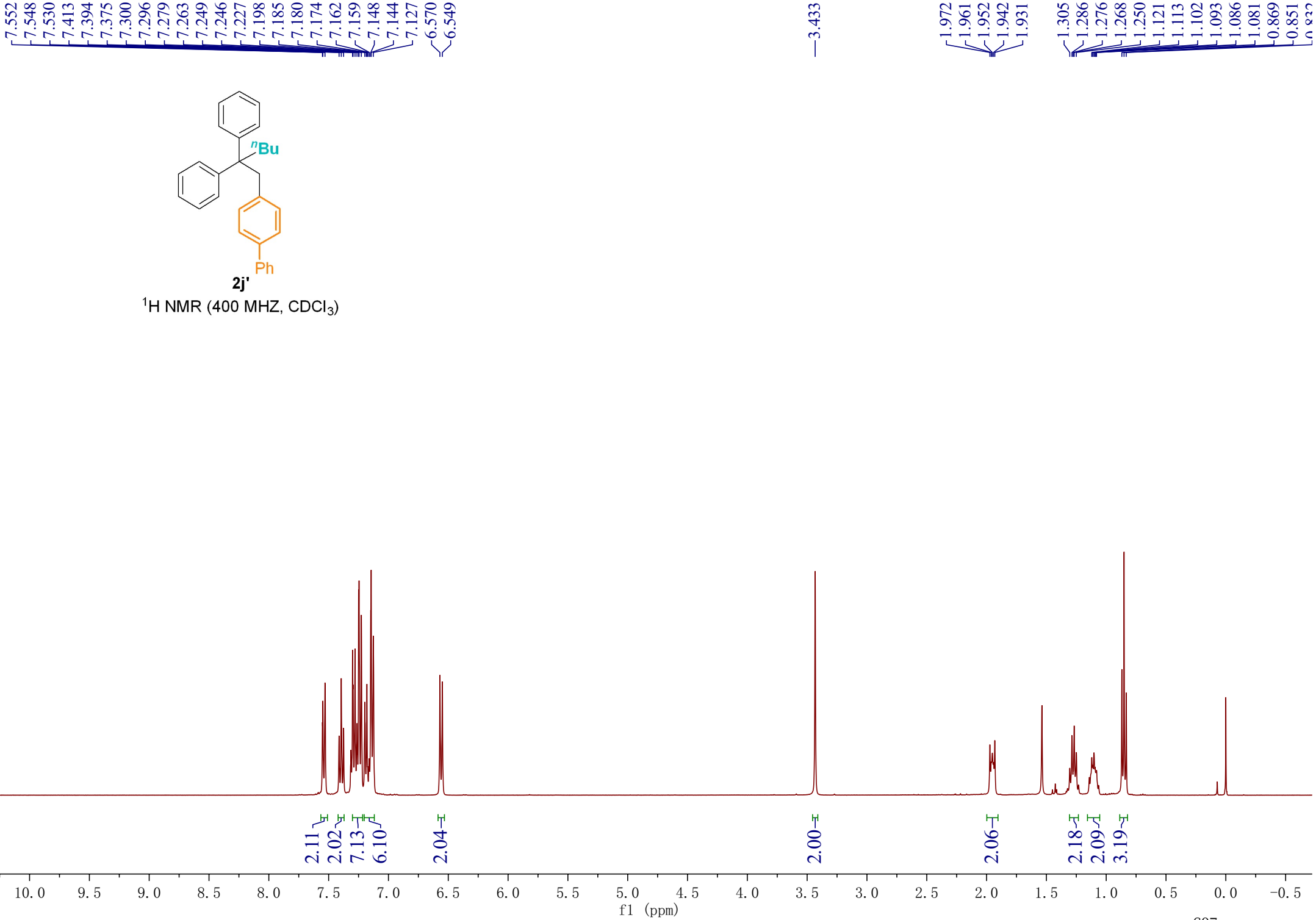

148.538
137.304
134.229
131.266
128.444
127.849
126.985
125.866
50.662
43.449
36.245
26.708
23.342
23.011
14.281
nBu
SMe
2h'
13C NMR (100 MHZ, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



2i'

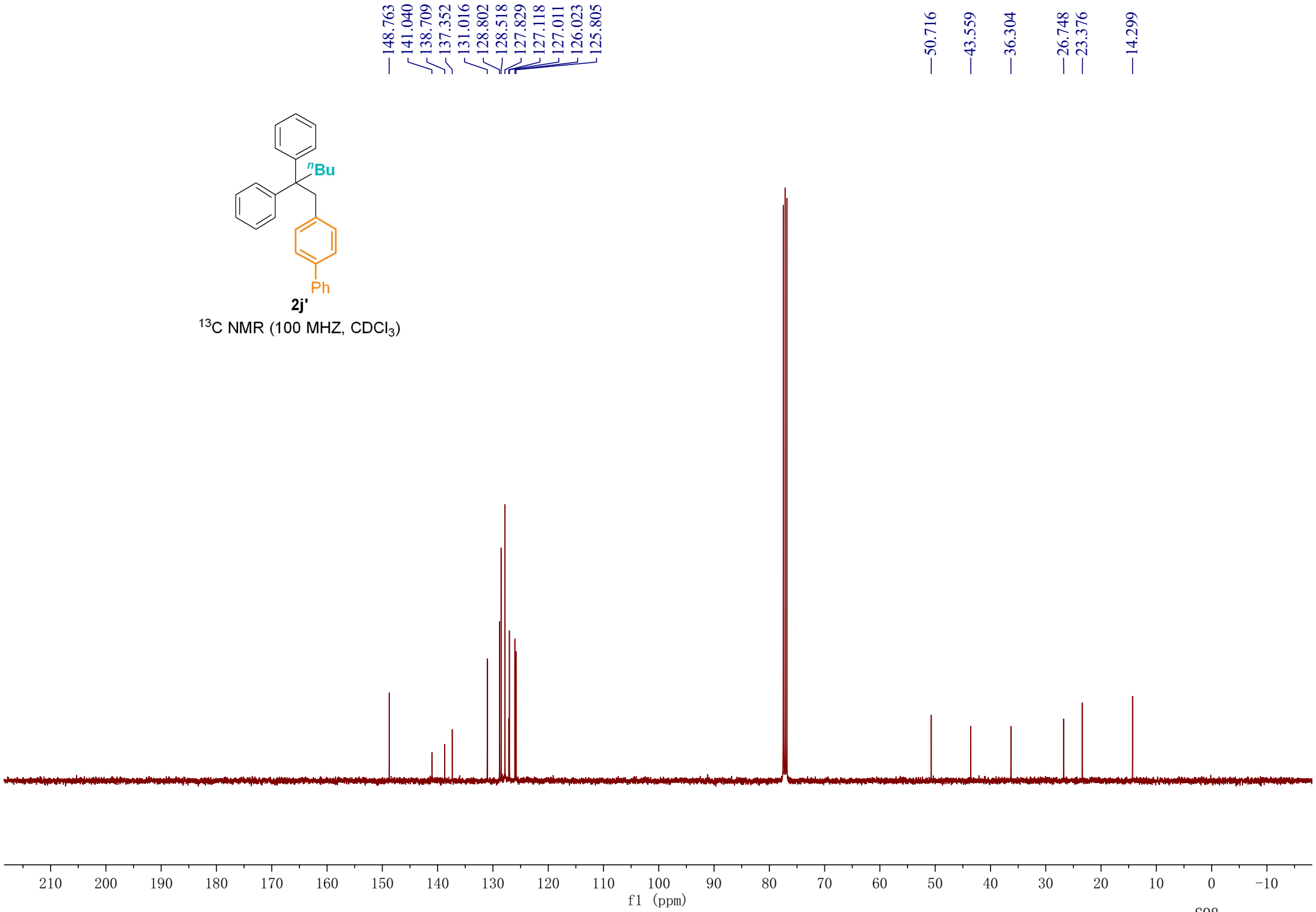
^{1}H NMR (400 MHZ, $CDCl_3$)

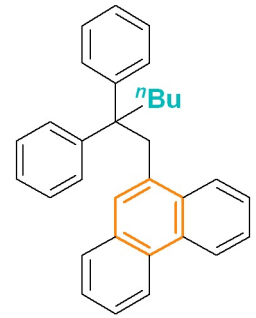

148.918
135.435
134.964
130.482
128.505
128.135
127.765
125.701
50.593
43.408
36.222
26.715
23.361
21.143
14.282
nBu
Me
2i'
13C NMR (100 MHZ, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



2j'

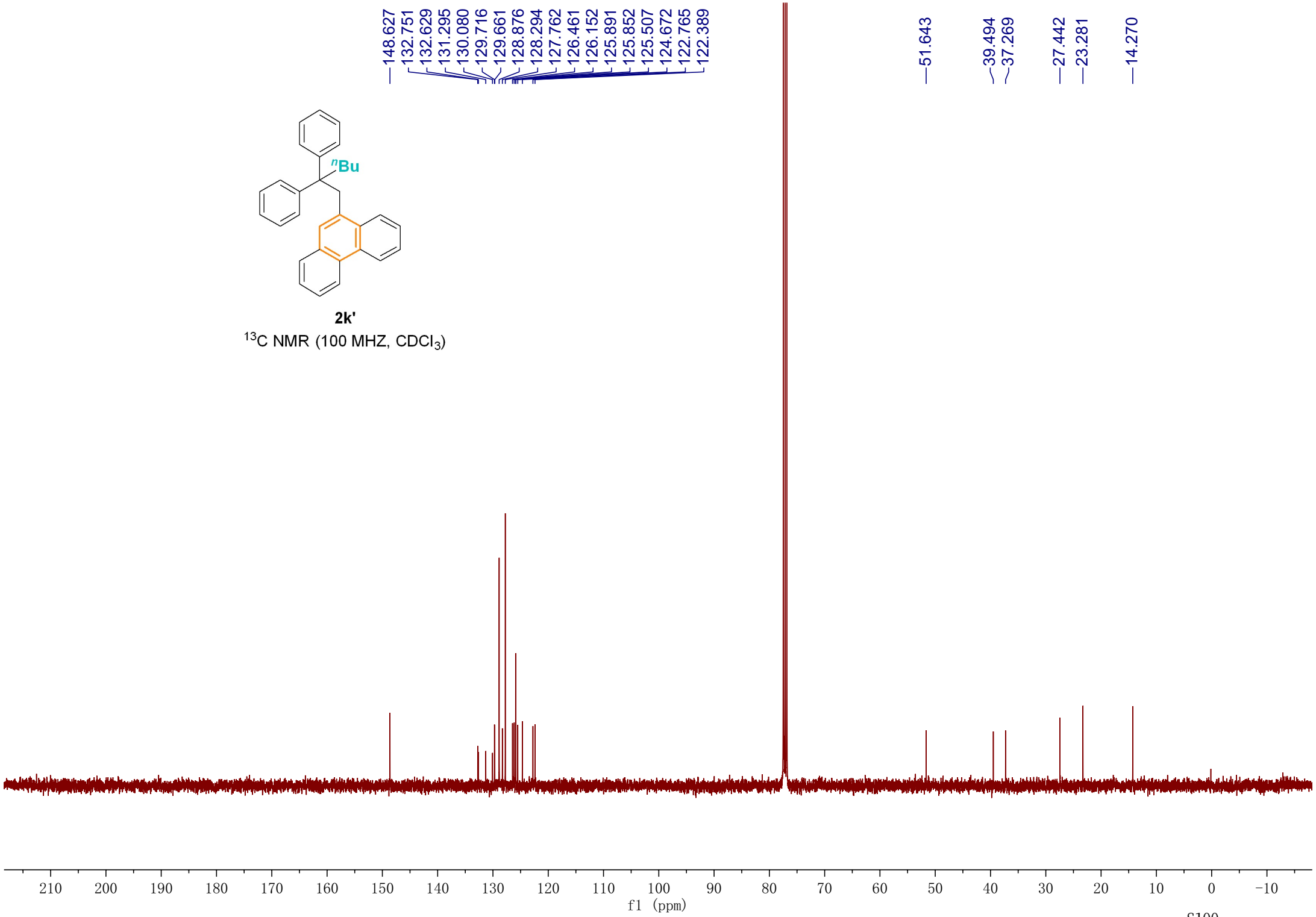
^{1}H NMR (400 MHZ, $CDCl_3$)

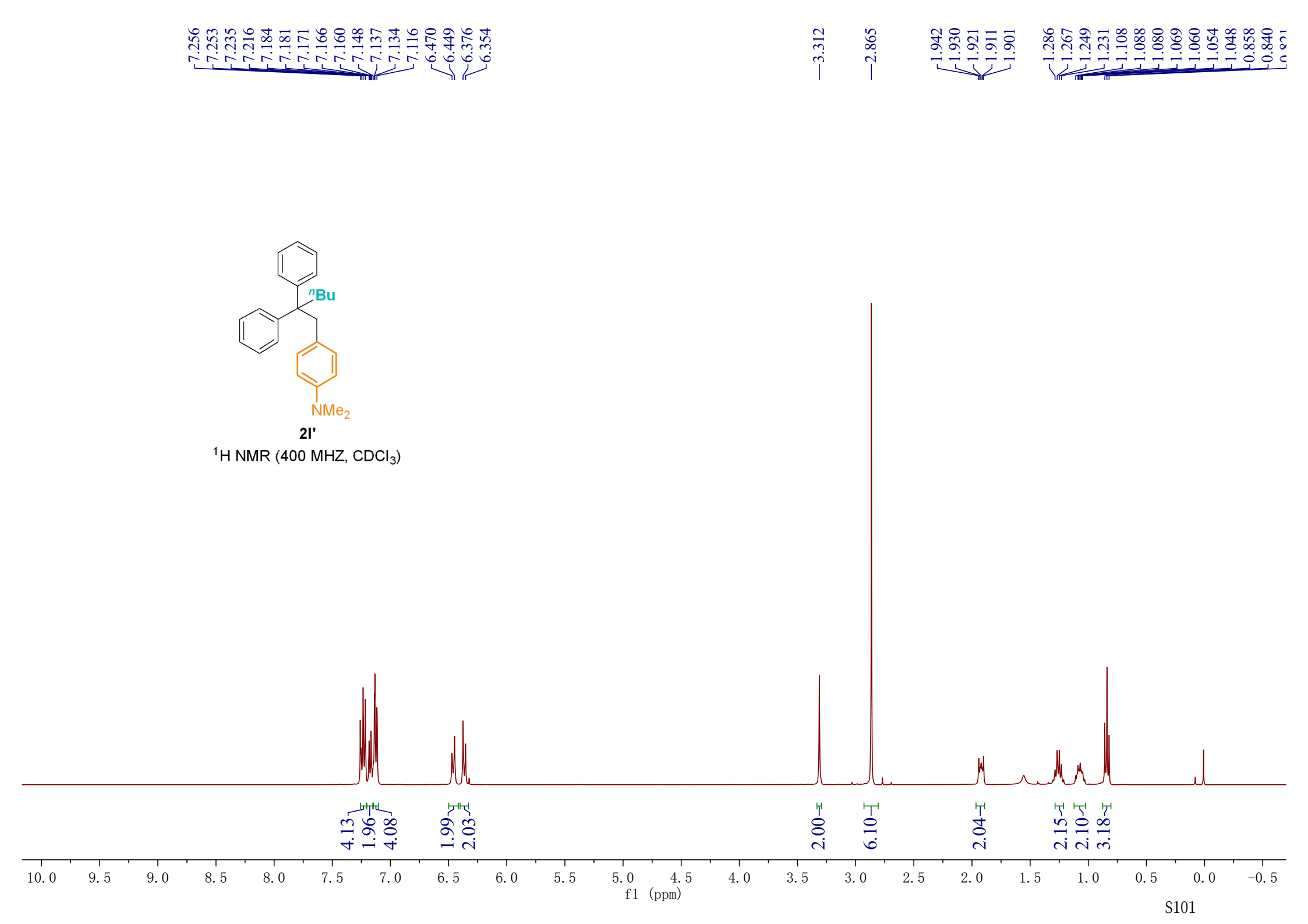

148.763
141.040
138.709
137.352
131.016
128.802
128.518
127.829
127.118
127.011
126.023
125.805
50.716
43.559
36.304
26.748
23.376
14.299
nBu
Ph
2j'
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

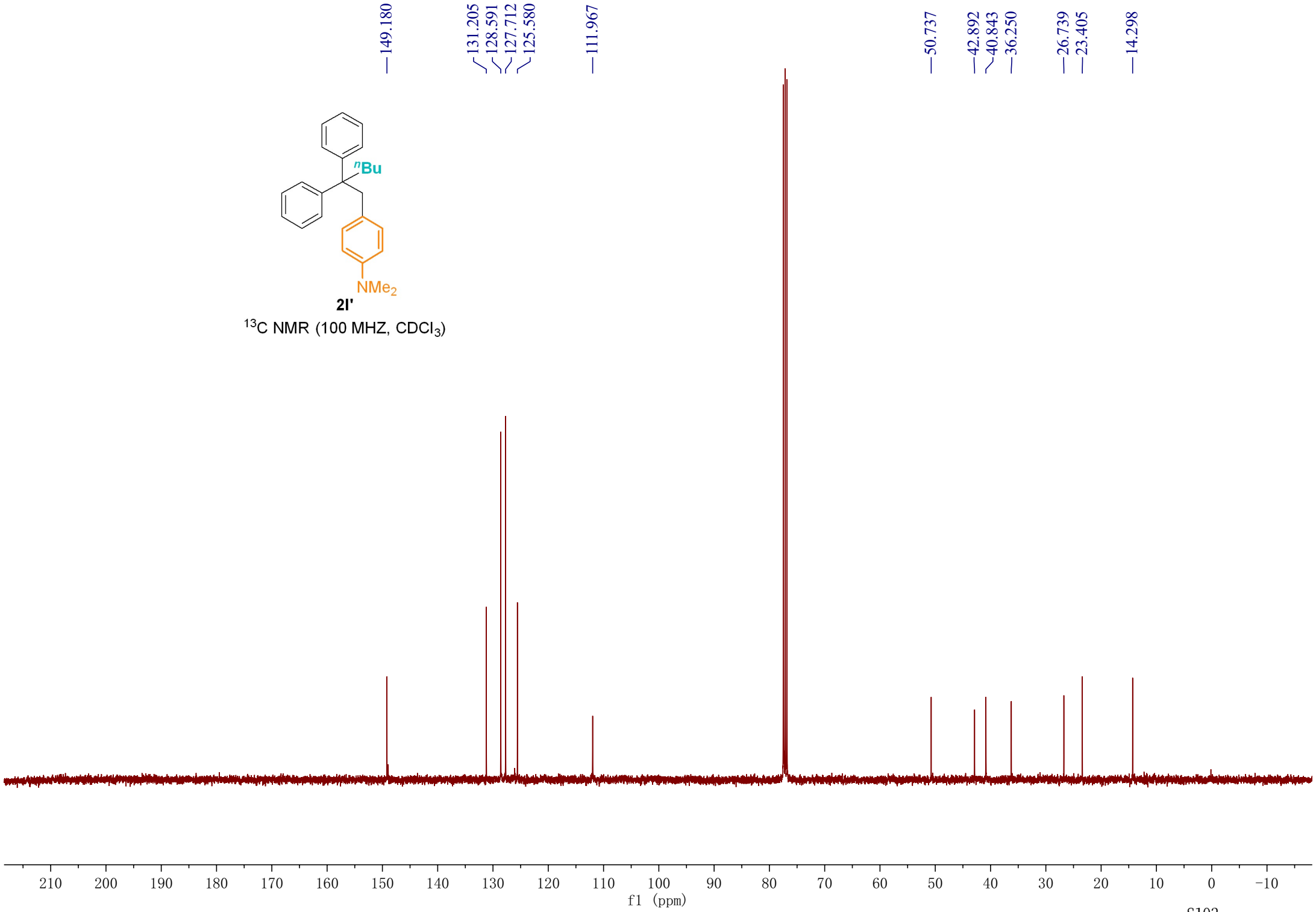


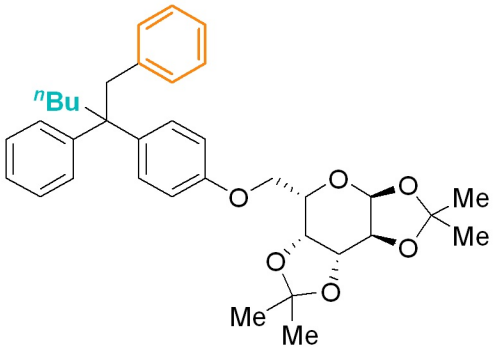
2k'

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


148.627
132.751
132.629
131.295
130.080
129.716
129.661
128.876
128.294
127.762
126.461
126.152
125.891
125.852
125.507
124.672
122.765
122.389
51.643
39.494
37.269
27.442
23.281
14.270
nBu
2k'
13C NMR (100 MHZ, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

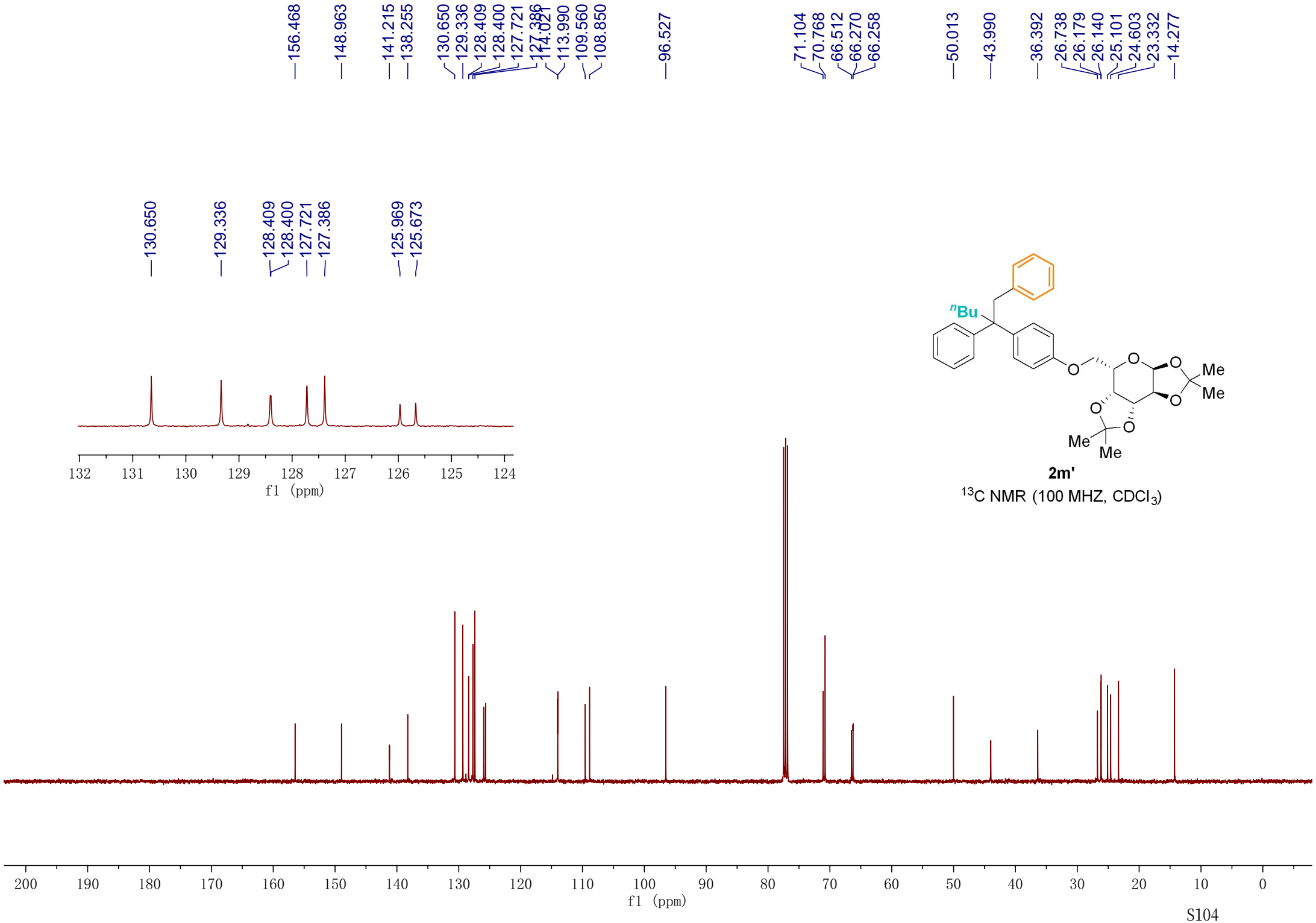

7.256
7.253
7.235
7.216
7.184
7.181
7.171
7.166
7.160
7.148
7.137
7.134
7.116
6.470
6.449
6.376
6.354
3.312
2.865
1.942
1.930
1.921
1.911
1.901
1.286
1.267
1.249
1.231
1.108
1.088
1.080
1.069
1.060
1.054
1.048
0.858
0.840
nBu
NMe2
2l'
1H NMR (400 MHZ, CDCl3)
4.13
1.96
4.08
1.99
2.03
2.00
6.10
2.04
2.15
2.10
3.18
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

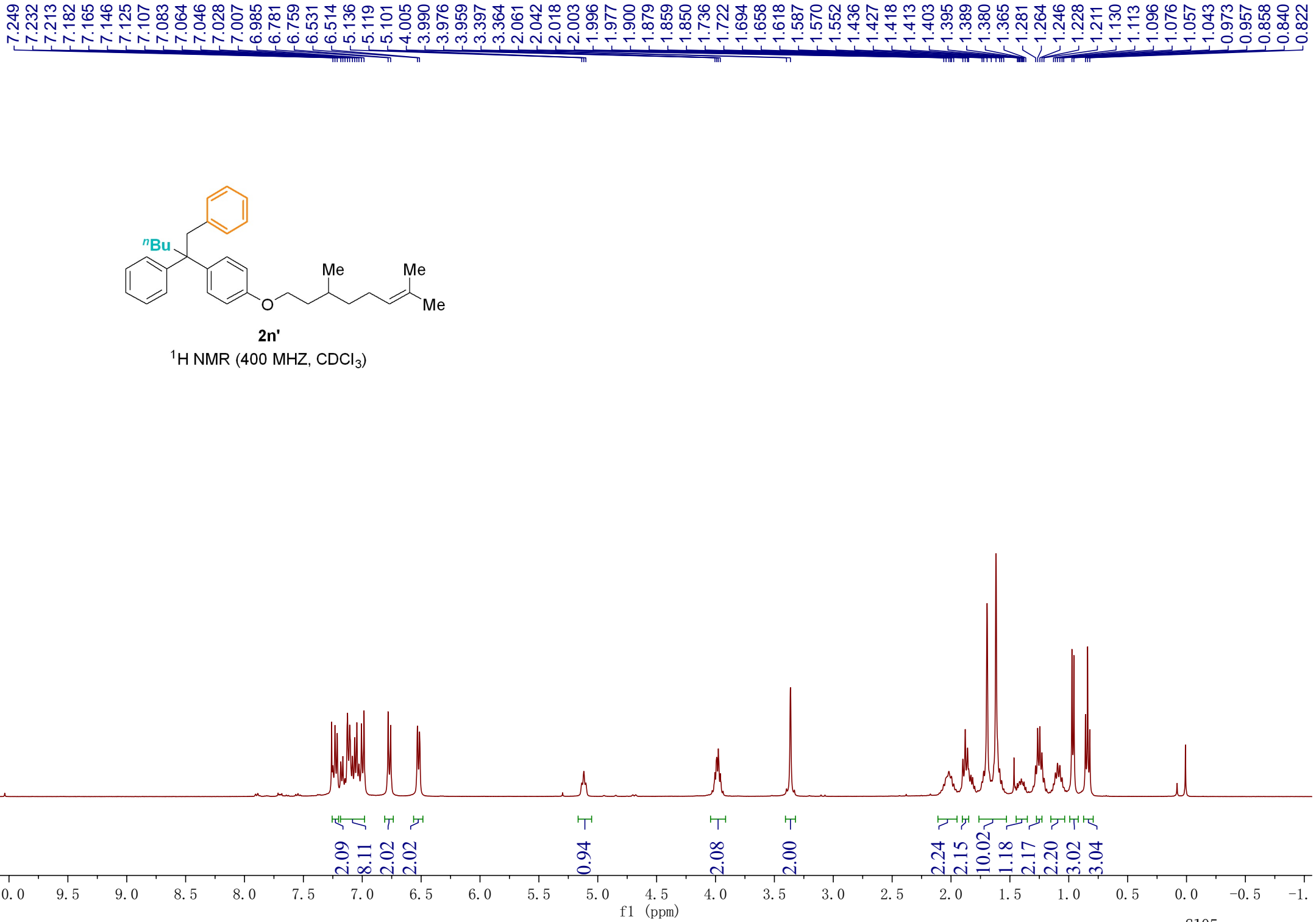

149.180
131.205
128.591
127.712
125.580
111.967
50.737
42.892
40.843
36.250
26.739
23.405
14.298
nBu
NMe2
2l'
13C NMR (100 MHZ, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



2m'

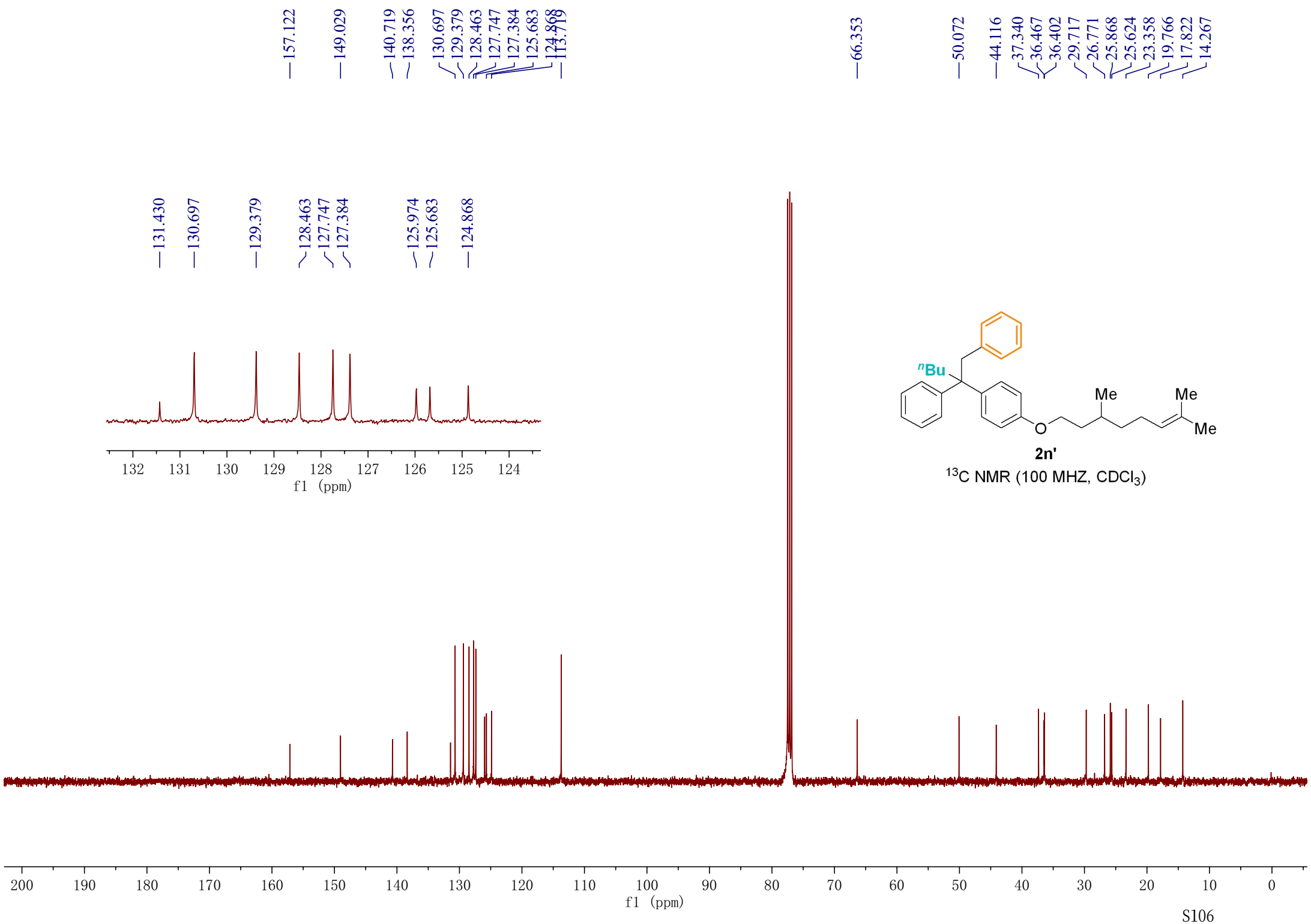
^{1}H NMR (400 MHZ, $CDCl_3$)

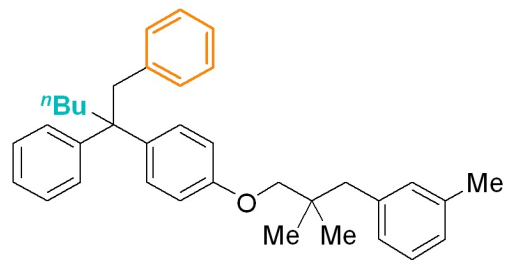

156.468
148.963
141.215
138.255
130.650
129.336
128.409
128.400
127.721
127.386
114.021
113.990
109.560
108.850
96.527
71.104
70.768
66.512
66.270
66.258
50.013
43.990
36.392
26.738
26.179
26.140
25.101
24.603
23.332
14.277
130.650
129.336
128.409
128.400
127.721
127.386
125.969
125.673
132 131 130 129 128 127 126 125 124
f1 (ppm)
nBu
O
O
O
O
O
O
Me
Me
Me
Me
2m'
13C NMR (100 MHZ, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



2n'

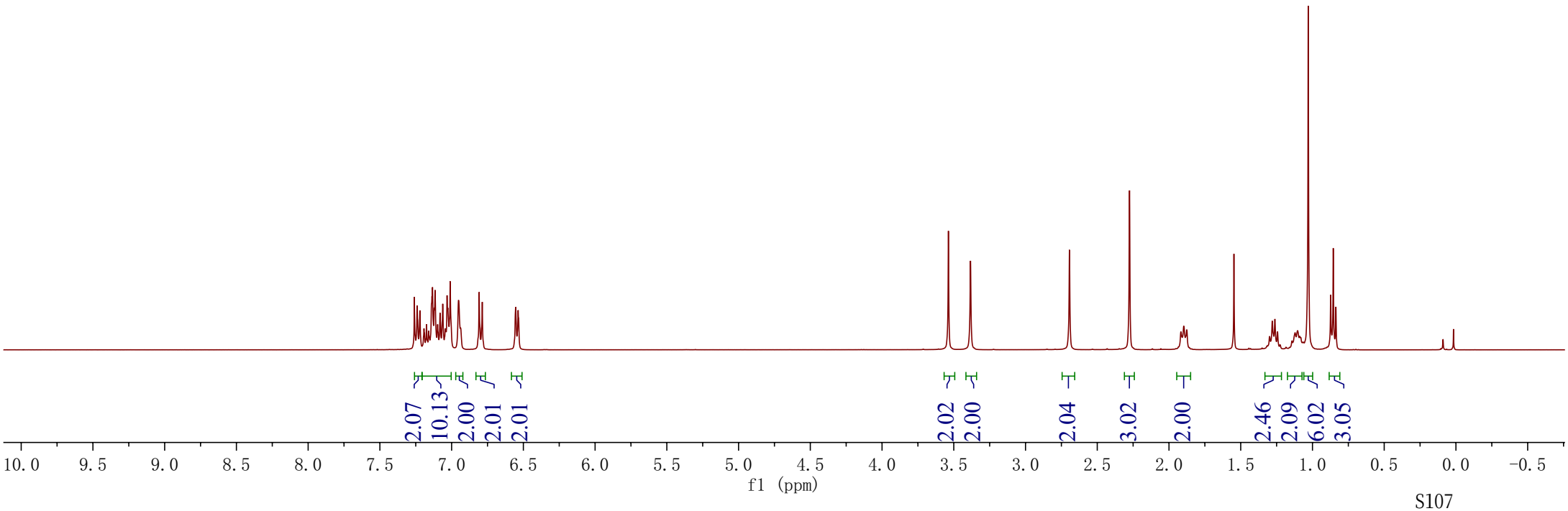
^{1}H NMR (400 MHZ, $CDCl_3$)

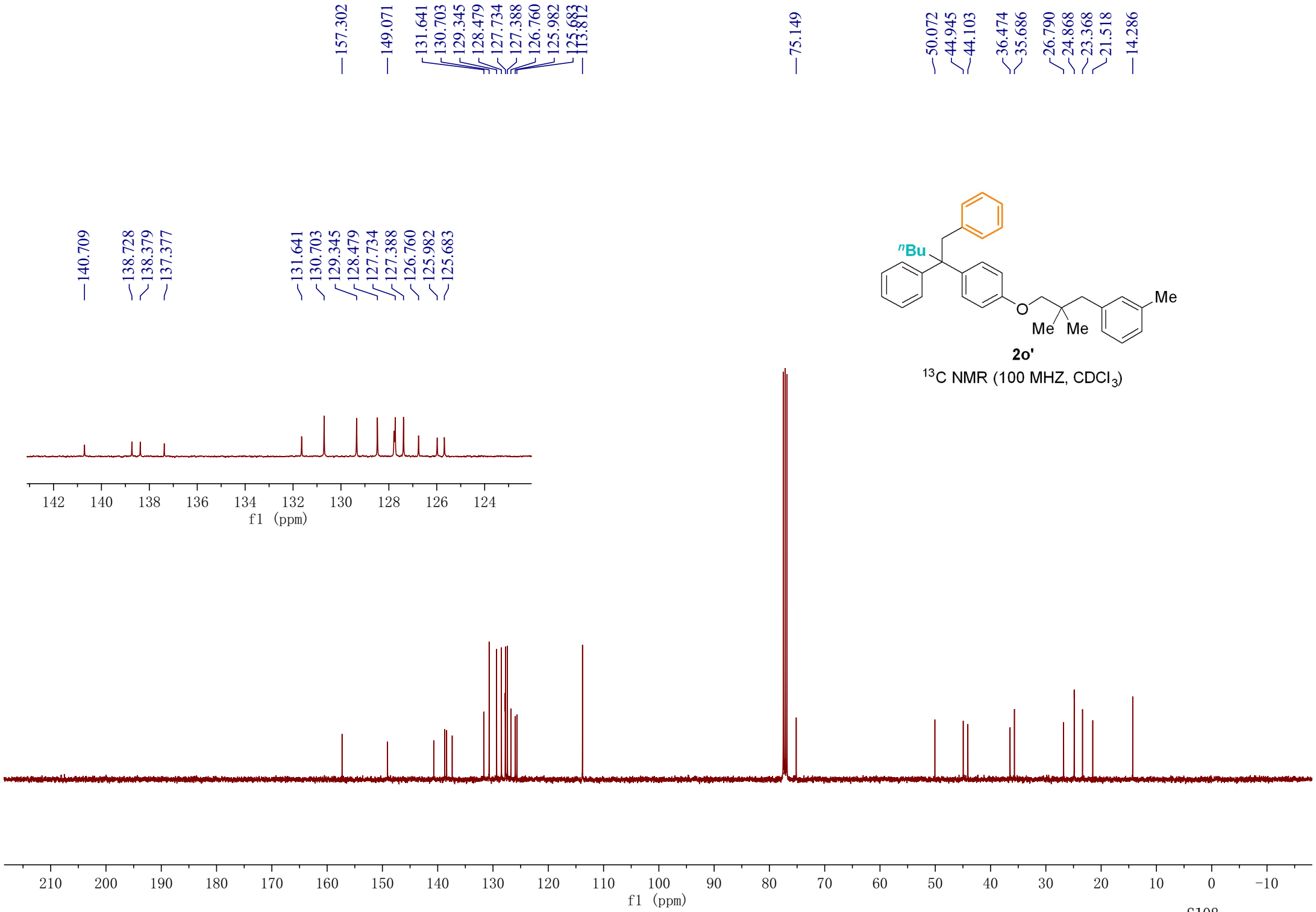

157.122
149.029
140.719
138.356
130.697
129.379
128.463
127.747
127.384
125.683
124.868
113.719
66.353
50.072
44.116
37.340
36.467
36.402
29.717
26.771
25.868
25.624
23.358
19.766
17.822
14.267
131.430
130.697
129.379
128.463
127.747
127.384
125.974
125.683
124.868
132 131 130 129 128 127 126 125 124
f1 (ppm)
nBu
Me
Me
Me
O
2n'
13C NMR (100 MHZ, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

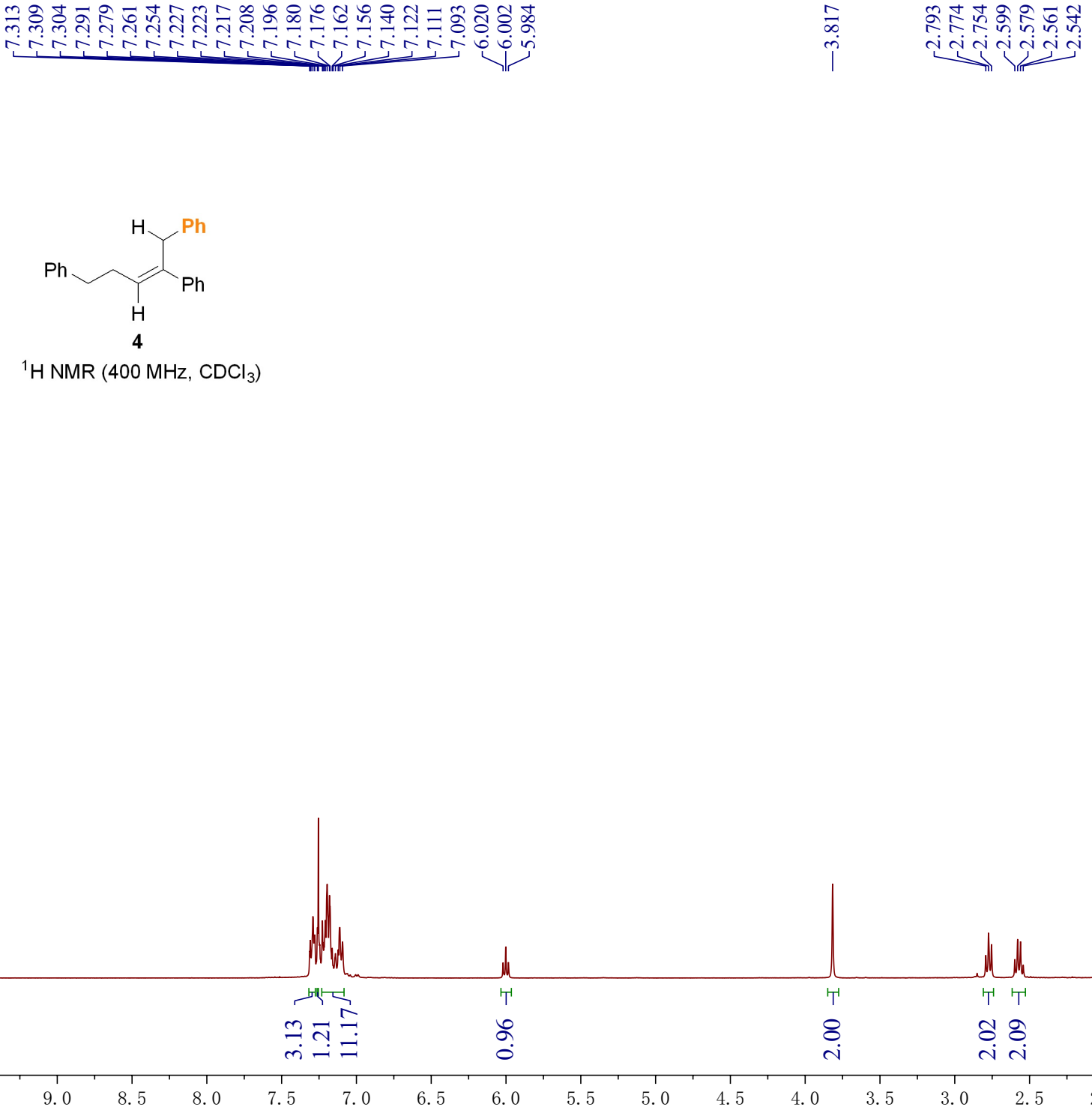


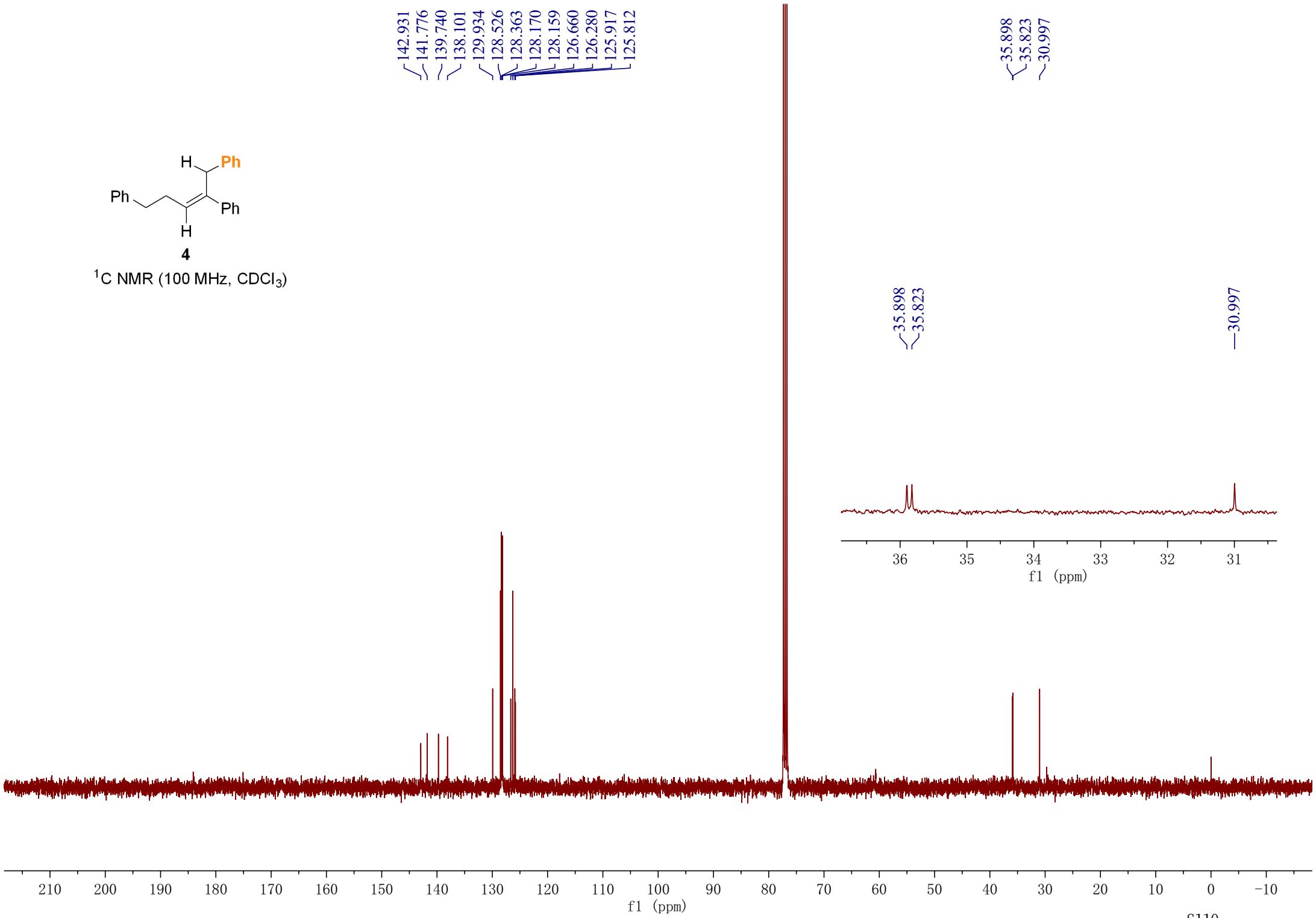
2o'

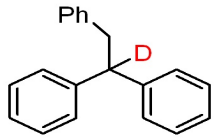
^{1}H NMR (400 MHZ, $CDCl_3$)




157.302
149.071
131.641
130.703
129.345
128.479
127.734
127.388
126.760
125.982
125.683
113.812
75.149
50.072
44.945
44.103
36.474
35.686
26.790
24.868
23.368
21.518
14.286
140.709
138.728
138.379
137.377
131.641
130.703
129.345
128.479
127.734
127.388
126.760
125.982
125.683
nBu
Me
O
Me
Me
2o'
13C NMR (100 MHZ, CDCl3)
f1 (ppm)
f1 (ppm)

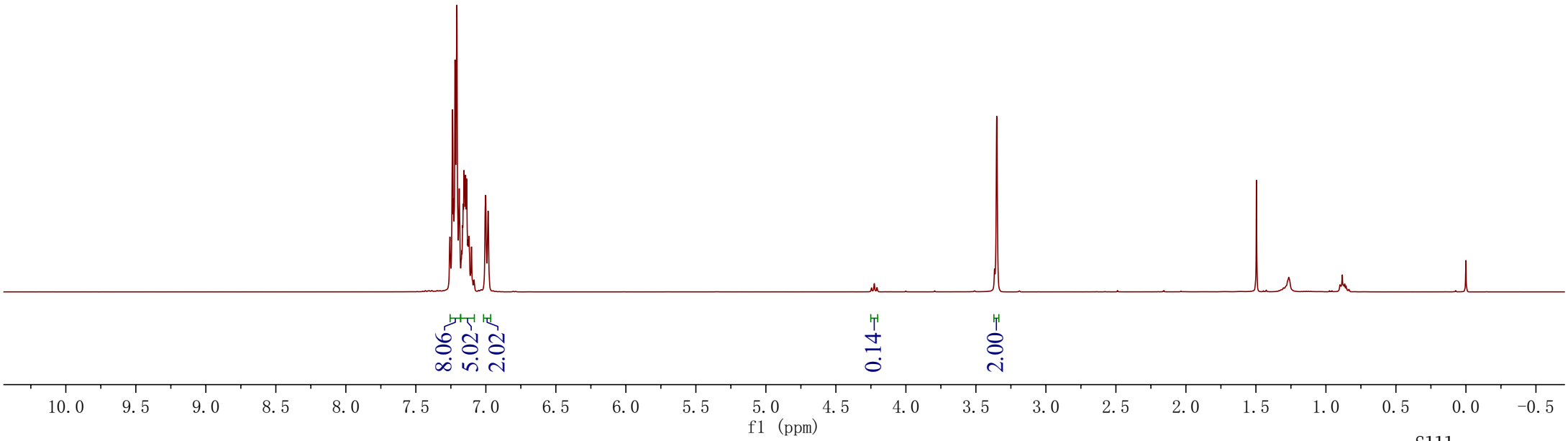

7.313
7.309
7.304
7.291
7.279
7.261
7.254
7.227
7.223
7.217
7.208
7.196
7.180
7.176
7.162
7.156
7.140
7.122
7.111
7.093
6.020
6.002
5.984
3.817
2.793
2.774
2.754
2.599
2.579
2.561
2.542
H
Ph
Ph
Ph
H
4
1H NMR (400 MHz, CDCl3)
3.13
1.21
11.17
0.96
2.00
2.02
2.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

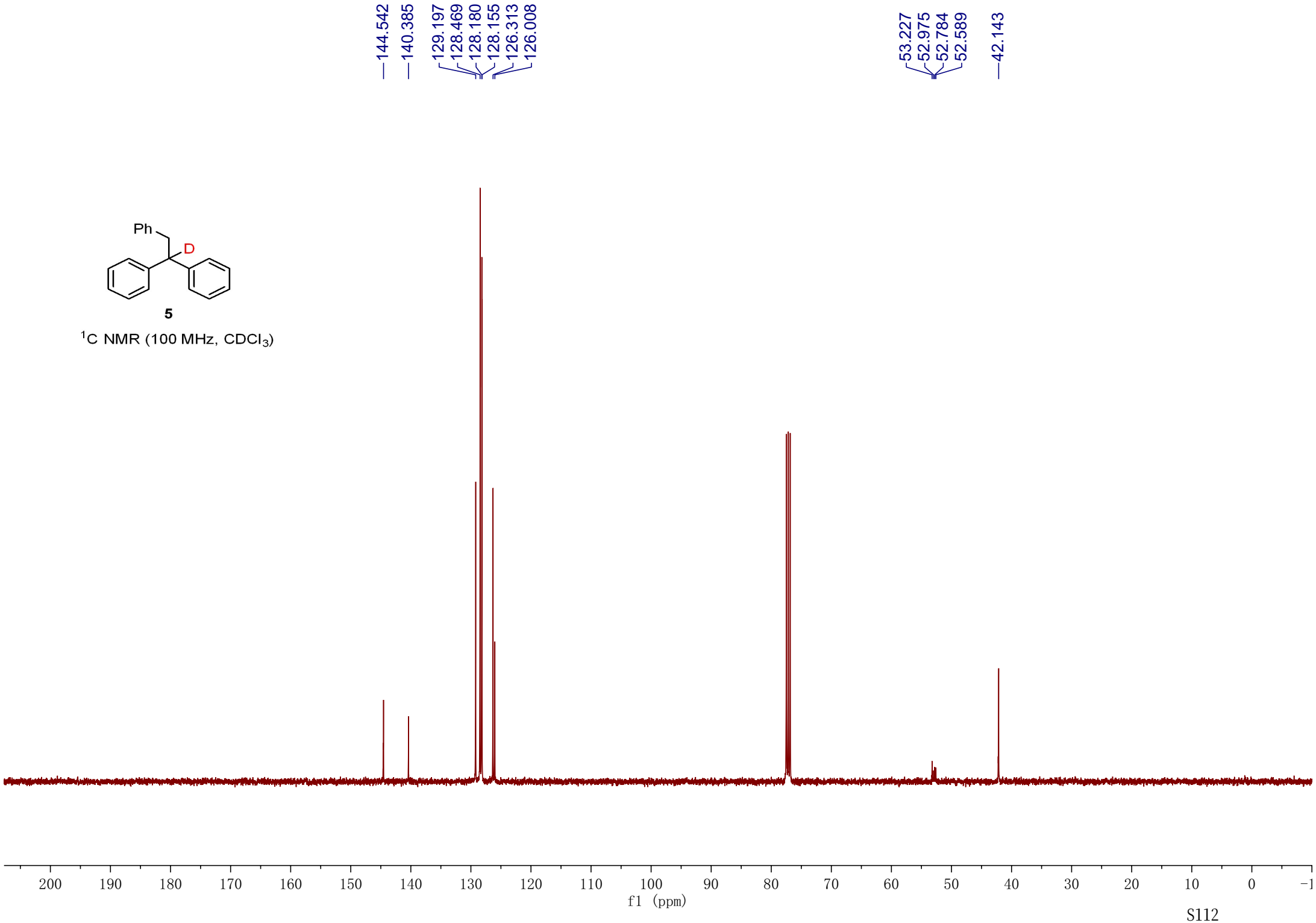

142.931
141.776
139.740
138.101
129.934
128.526
128.363
128.170
128.159
126.660
126.280
125.917
125.812
35.898
35.823
30.997
H
Ph
Ph
Ph
H
4
^{1}C NMR (100 MHz, $CDCl_3$)
35.898
35.823
30.997
36
35
34
33
32
31
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)



5

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




144.542
140.385
129.197
128.469
128.180
128.155
126.313
126.008
53.227
52.975
52.784
52.589
42.143
Ph
D
5
^{1}C NMR (100 MHz, $CDCl_3$)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)