

Supporting Information

Iodine-promoted transfer of dihydrogen from ketones to alkenes, triphenylmethyl and diphenylmethyl derivatives

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DOI: 10.1002/anie.2021XXXXX

Supporting Information

Table of Contents

1. General Information.....	3
2. Preparation of Substrates	3
2.1. Preparation of 5b-e , 5m-n , 5u	3
2.2. Preparation of 1f-l , 1o	4
2.3. Preparation of 1r	4
2.4. Preparation of 5o , 5l , 5q	4
2.5. Preparation of 5k , 5m	4
2.6. Preparation of 5b-e , 5i	5
3. Optimization of the Reaction Conditions	5
4. General Procedure of Hydrogenation	6
4.1. General Procedure for the Hydrogenation of Alkenes and Alkyne.....	6
4.2. General Procedure for Hydrogenation of of Alcohol, Ether, Ester, Amine and <i>et al.</i>	6
5. Mechanism Studies	7
5.1. Compound 8 NMR and MS Characterization	7
5.2. General Procedure for Compound 9 Derivatives Synthesis	8
5.3. General Procedure for Compound 9-1 Derivatives Synthesis.....	10
5.4. General Procedure for Deuterium-Labeling Ketones 2k Synthesis	11
6. Product Characterization.....	11
7. References.....	17
8. Product NMR Spectra	18

1. General Information

All reaction reagents or intermediates were carried out in dried glassware under atmosphere using 20 mL of Schlenk tube. *n*-Hexane was purchase from shanghai titan scientific. Otherwise noted, other commercially available reagents were purchased from Energy Chemical, Sigma-Aldrich or Bidepharm in the highest purity grade and used without further purification. Thin layer chromatography (TLC) was performed on Energy Chemical silica gel GF-254 plates and visualized by fluorescence quenching under UV light (254/366 nm) or iodine quenching under silica gel and iodine mixture. Column chromatography was performed on Qingdao Ocean Chemical Co., Ltd. silica gel (300-400 mesh). ¹H and ¹³C NMR spectra were recorded on a Bruker AC-400 FT or Bruker AC-300 FT spectrometer using tetramethylsilane as an internal reference. NMR multiplicities are abbreviated as follows: s = singlet, d = doublet, m = multiplet. Chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz, respectively. High-resolution mass spectra (HRMS) measurement was performed on Agilent Q-TOF 6520 mass spectrometer with electron ionization (EI) and electrospray ion source (ESI) as the ion source.

Abbreviations: TH = transfer hydrogenation, NIS = *N*-iodosuccinimide, TBAI = tetrabutylammonium iodide, TfOH = trifluoromethanesulfonic acid, DCM = dichloromethane, EA = ethyl acetate, THF = tetrahydrofuran, MeCN = acetonitril, DIDMH= 1,3-diiodo-5,5-dimethylimidazolidine-2,4-dione.

2. Preparation of Substrates

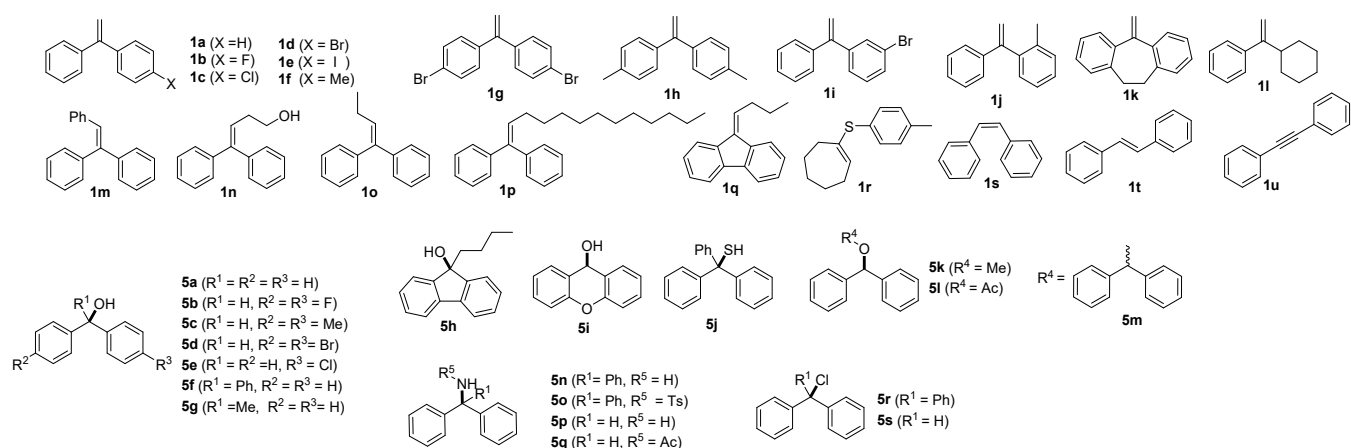
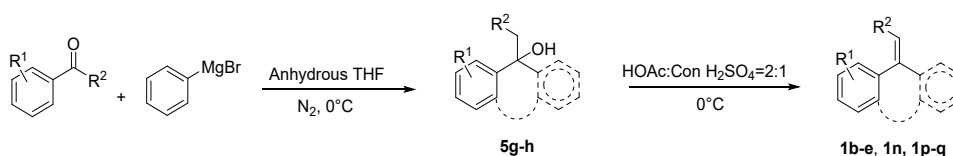


Figure S1. Substrate structures.

2.1. Preparation of 5b-e, 5m-n, 5u

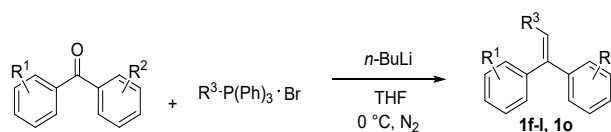


To a solution of substituent aromatic ketones (10.0 mmol) in dry tetrahydrofuran (20 mL) under a nitrogen atmosphere at 0 °C was added dropwise a solution of phenyl magnesium bromide in tetrahydrofuran (2.0 M, 7.5 mL, 15.0 mmol). The mixture was stirred at 0 °C for 10 min and then for another 2 h at room temperature. The mixture was added a saturated aqueous NH₄Cl solution (10 mL) carefully, then extracted with ethyl acetate (3 × 20 mL). The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated to give the crudes, which were purified by silica gel column chromatography, eluting with petroleum ether, to give desired products **5g-h**.

The crude product was dissolved in acetic acid (4 mL) at 0 °C, and Con. H₂SO₄ (2.0 mL) was added dropwise. The mixture was allowed to warm to room temperature and stirred for 20 min. The reaction mixture was poured out into about 50 mL ice water, and then extracted with ethyl acetate (3 × 15 mL). The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtered,

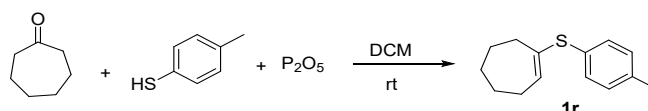
and concentrated to give the crudes, which were purified by silica gel column chromatography, eluting with petroleum ether, to give desired products **1b-e**, **1n**, **1p-q**.

2.2. Preparation of **1f-l**, **1o**



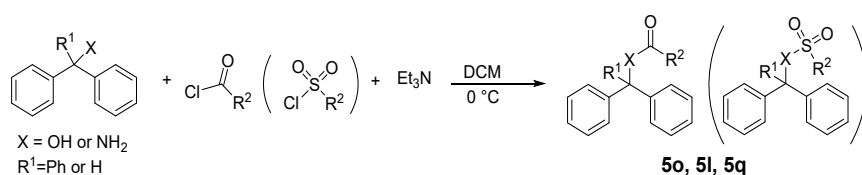
To a solution of $R^3(\text{Ph})_3\text{Br}$ (5.13 g, 15.0 mmol) in dry tetrahydrofuran (20 mL) under a nitrogen atmosphere at 0 °C was added dropwise a solution of *n*-BuLi in tetrahydrofuran (2.5 M, 6.0 mL, 15.0 mmol). The mixture was allowed to warm to room temperature and stirred for 0.5 h. Ketones (12.0 mmol) were dissolved in anhydrous and added into the above reaction mixture with a syringe, the results mixture was stirred with another 2 h. After the reaction was completed, the mixture was added a saturated aqueous NH_4Cl solution (10 mL) carefully, then extracted with ethyl acetate (3×20 mL). The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated to give the crudes, which were purified by silica gel column chromatography, eluting with petroleum ether, to give desired products **1f-l**, **1o**.

2.3. Preparation of **1r**



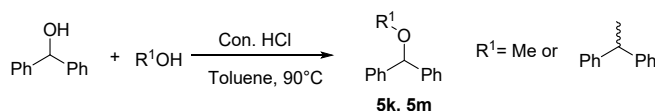
Cycloheptanone (1.12 g, 10.0 mmol) and P_2O_5 (2.84 g, 20.0 mmol) were dissolved in 30 mL anhydrous DCM (30 mL), and *p*-toluenethiol (1.37 g, 11.0 mmol) was added dropwise. Then, the mixture was stirred for 5 h at room temperature. The mixture was quenched with 2N NaOH (30 mL) solution, then extracted with ethyl acetate (3×20 mL). The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated to give the crude, which was purified by silica gel column chromatography, eluting with petroleum ether, to give the desired product **1r**.

2.4. Preparation of **5o**, **5l**, **5q**



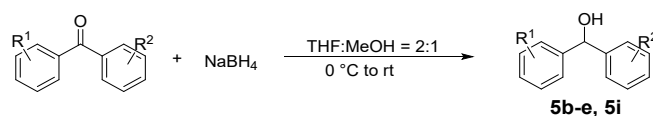
Substituents benzyl alcohol (10.0 mmol) and Et_3N (2.77 mL, 20.0 mmol) were dissolved in anhydrous dichloromethane (30 mL) at 0 °C and acyl chloride/ sulfonyl chloride (15.0 mmol) was added dropwise. The mixture was allowed to warm to room temperature and stirred for 3 h. Then, the mixture was extracted with dichloromethane (3×20 mL). The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated to give the crude, which was purified by silica gel column chromatography, eluting with ethyl acetate/petroleum ether (1: 5 to 1: 3 v/v), to give desired products **5o**, **5l**, **5q**.

2.5. Preparation of **5k**, **5m**



To a solution of benzhydrol (1.84 g, 10.0 mmol) in toluene (20 mL) was added methanol/ethanol (30.0 mmol) and 3 drops Con. HCl. The mixture was heated at 100 °C for 12 h, cooled to room temperature, and concentrated under reduced pressure to give the crudes, which as purified by silica gel column chromatography, eluting with ethyl acetate/petroleum ether (1: 20 to 1: 10 v/v), to give desired products **5k**, **5m**.

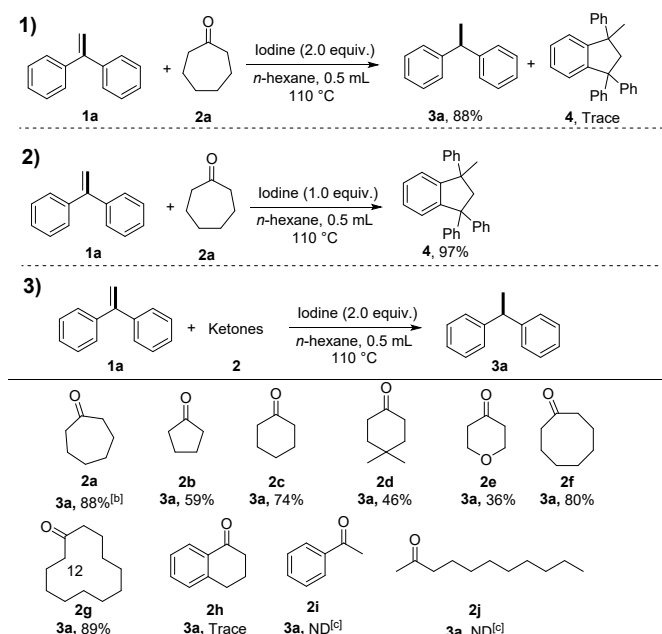
2.6. Preparation of **5b-e**, **5i**



Ketones (10.0 mmol) were dissolved in MeOH (30 mL) at 0 °C, and NaBH₄ (757 mg, 20.0 mmol) was added slowly. The mixture was allowed to warm to room temperature and stirred for 2 h. The mixture was quenched with 10 mL saturated aqueous NH₄Cl solution, then extracted with ethyl acetate (3 × 20 mL). The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated to give the crude, which was purified by silica gel column chromatography, eluting with ethyl acetate/petroleum ether (1: 10 to 1: 4 v/v), to give desired product **5b-e**, **5i**.

3. Optimization of the Reaction Conditions

The template reaction of iodine-promoted transfer of dihydrogen from ketones to alkenes were performed. (Scheme S1, eq 1) Various of ketones (**2a-j**) were tested for the hydrogenation reaction under the template reaction conditions. (Scheme S1, eq 2)



[a] Reaction conditions: **1a** (0.3 mmol), ketones (0.75 mmol), iodine (0.6 mmol) and *n*-hexane (0.5 mL) under the atmosphere for 4 h at 110 °C. [b] The yields are isolated yield. [c] ND = not detected.

Scheme S1. The effect of ketones on hydrogenation reactions.

For the procedure, see below. The results are summarized in the following Table.

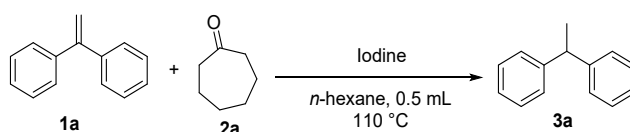
Table S1. optimization of the reaction condition^[a]

Entry ^[a]	Change from standard conditions	Yield of 3a [%] ^[b]
1	None	88
2	DCM instead of <i>n</i> -hexane	23

3	Toluene instead of <i>n</i> -hexane	80
4	CHCl ₃ instead of <i>n</i> -hexane	18
5	MeCN instead of <i>n</i> -hexane	65
6	Lower the temperature to 90 °C	77
7	DIDMH instead of iodine	26
8	NIS instead of iodine	29
9	HI instead of iodine	37
10	TBAI instead of iodine	NR
11	TfOH instead of iodine	62(48 ^[c])

[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.75 mmol), and Lewis acid (0.6 mmol) in *n*-hexane (0.5 mL), at 110 °C (oil bath), for 4 h. [b] Isolate yield. [c] 0.15 mmol TfOH, the isolated yield is 48%.

Table S2. Optimization of the reaction conditions.

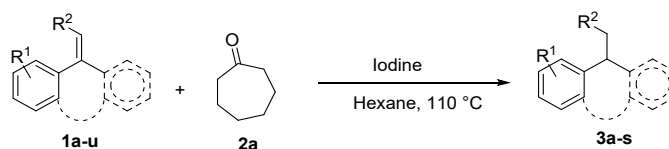


Entry	cycloheptanone (equiv)	Iodine (equiv)	Solvent (0.5 mL)	T [°C]	t [h]	Yield [%]
1	1	2	<i>n</i> -hexane	110	8	28
2	1.5	2	<i>n</i> -hexane	110	8	37
3	2	2	<i>n</i> -hexane	110	8	68
4	2.5	2	<i>n</i> -hexane	110	8	88
5	3	2	<i>n</i> -hexane	110	8	84
6	2.5	0.5	<i>n</i> -hexane	110	8	Trace
7	2.5	1	<i>n</i> -hexane	110	8	trace
8	2.5	1.5	<i>n</i> -hexane	110	8	38
9	2.5	3	<i>n</i> -hexane	110	8	79
10	2.5	2	DCM	110	8	23
11	2.5	2	Toluene	110	8	80
12	2.5	3	EA	110	8	NR
13	2.5	2	Dioxane	110	8	NR
14	2.5	2	THF	110	8	NR
15	2.5	2	CHCl ₃	110	8	18
16	2.5	2	MeCN	110	8	65
17	2.5	2	<i>n</i> -hexane	90	8	77
18	2.5	2	<i>n</i> -hexane	70	8	trace
19	2.5	2	<i>n</i> -hexane	110	2	56
20	2.5	2	<i>n</i> -hexane	110	4	89

According to the results, the amount of iodine and ketones is critical factors influencing the efficiency of the reaction. The amount of cycloheptanone needs more than 2.0 equiv., otherwise the yield of 1,1-diphenylethane will reduced sharply. The amount of iodine is also very important to the yield. The solvent of this hydrogenation reaction was compatible with *n*-hexane, toluene and MeCN. The temperature of the reaction is also important, while the temperature is under 70 °C, the reaction will not take place.

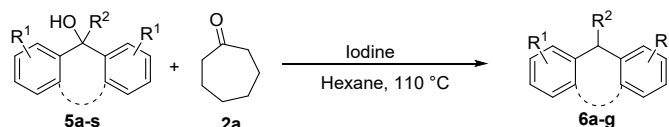
4. General Procedure of Hydrogenation

4.1. General Procedure for the Hydrogenation of Alkenes and Alkyne



To a solution of alkene derivatives (0.3 mmol) in *n*-hexane (0.5 mL) was added cycloheptanone (88.5 μ L, 0.75 mmol) and iodine (152.3 mg, 0.6 mmol). The mixture was heated at 110 $^{\circ}$ C (oil bath) under air in a sealed tube for 4 h, cooled to room temperature, and purified by silica gel column chromatography, eluting with ethyl acetate/petroleum ether (0: 10 to 1: 10 v/v), to give the desired products.

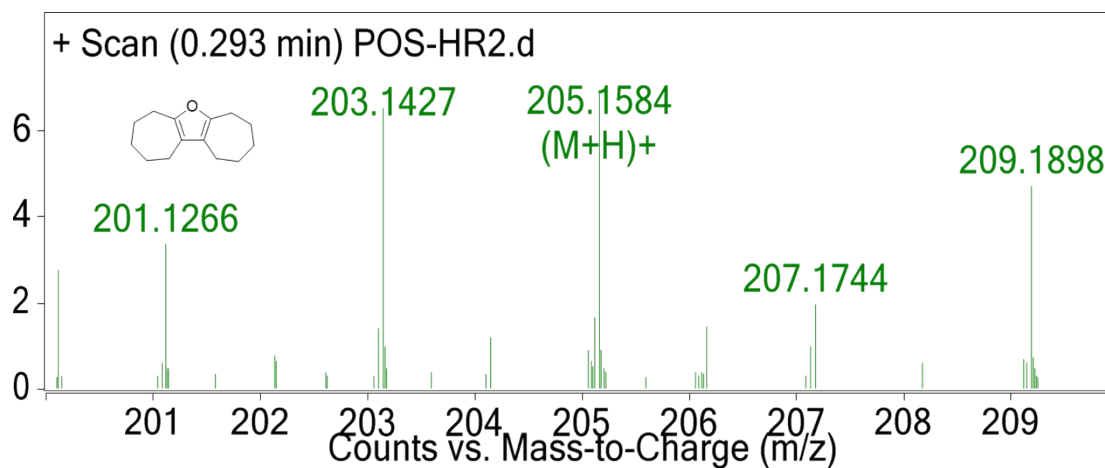
4.2. General Procedure for Hydrogenation of alcohol, ether, ester, amine *et al.*



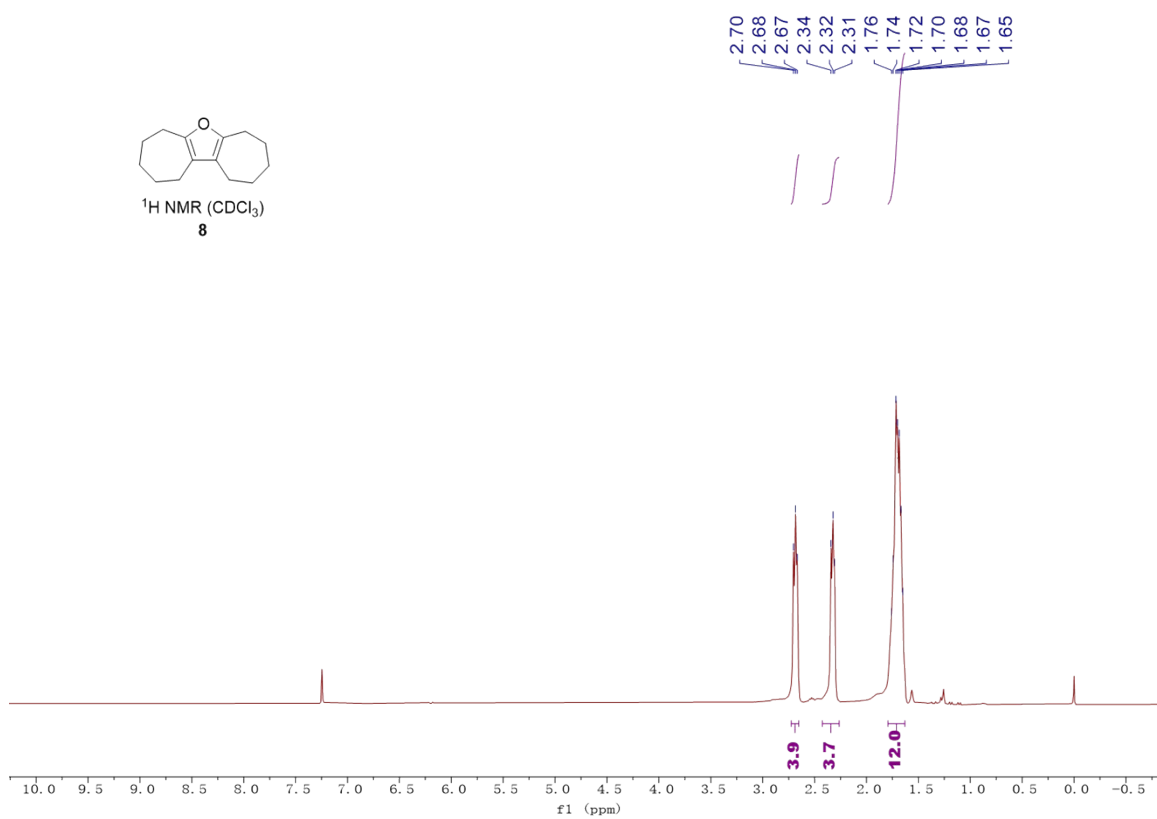
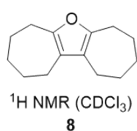
To a solution of benzhydryl derivatives (0.3 mmol) in *n*-hexane (0.5 mL) was added cycloheptanone (88.5 μ L, 0.75 mmol) and iodine (152.3 mg, 0.6 mmol). The mixture was heated at 110 $^{\circ}$ C (oil bath) under air in a sealed tube for 4 h, cooled to room temperature, and purified by silica gel column chromatography, eluting with ethyl acetate/petroleum ether (0: 10 to 1: 10 v/v), to give the desired products.

5. Mechanism Studies

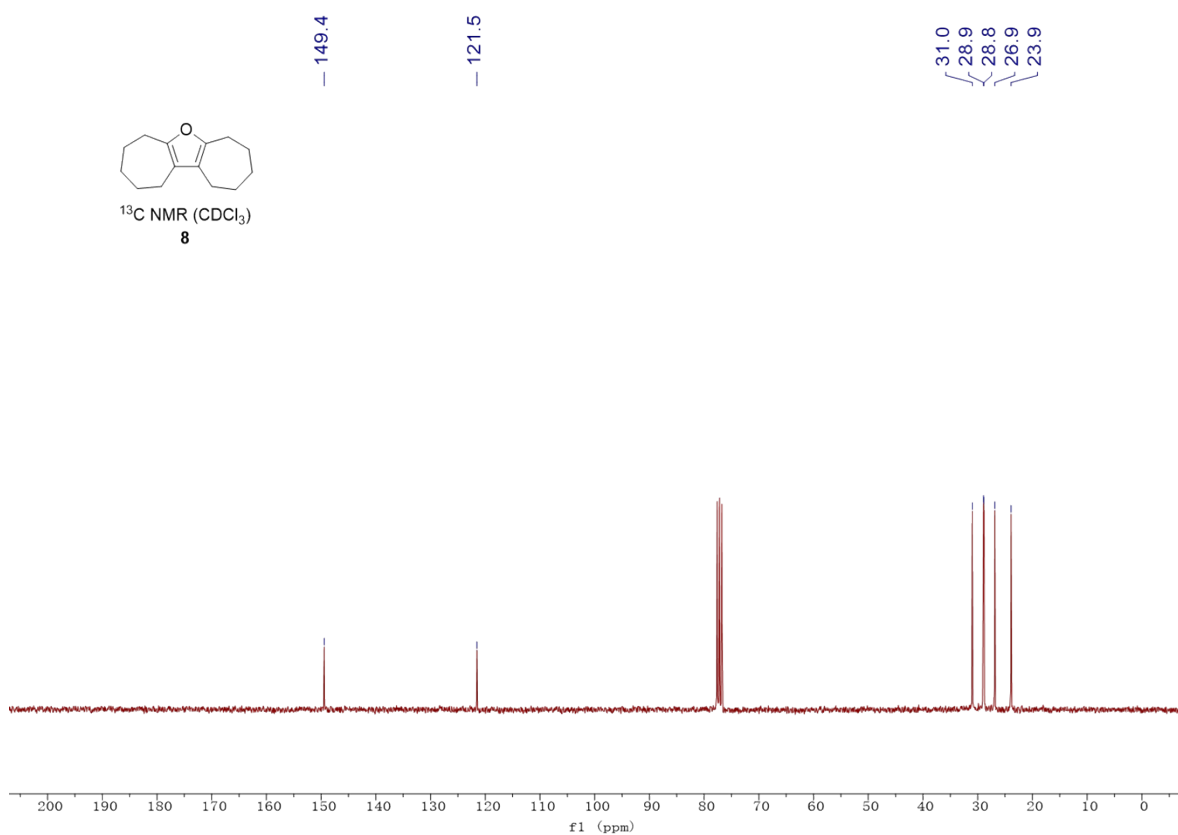
5.1. Compound 8 NMR and MS Characterization



Compound 8 HRMS (ESI) calcd for $C_{14}H_{21}O$ ($M+H^+$) 205.1587, found 205.1584.

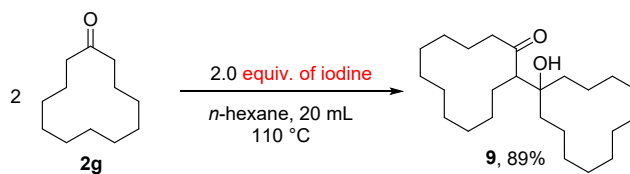


¹H NMR (300 MHz, CDCl₃) δ 2.73 – 2.65 (m, 4H), 2.43 – 2.26 (m, 4H), 1.79 – 1.63 (m, 12H).

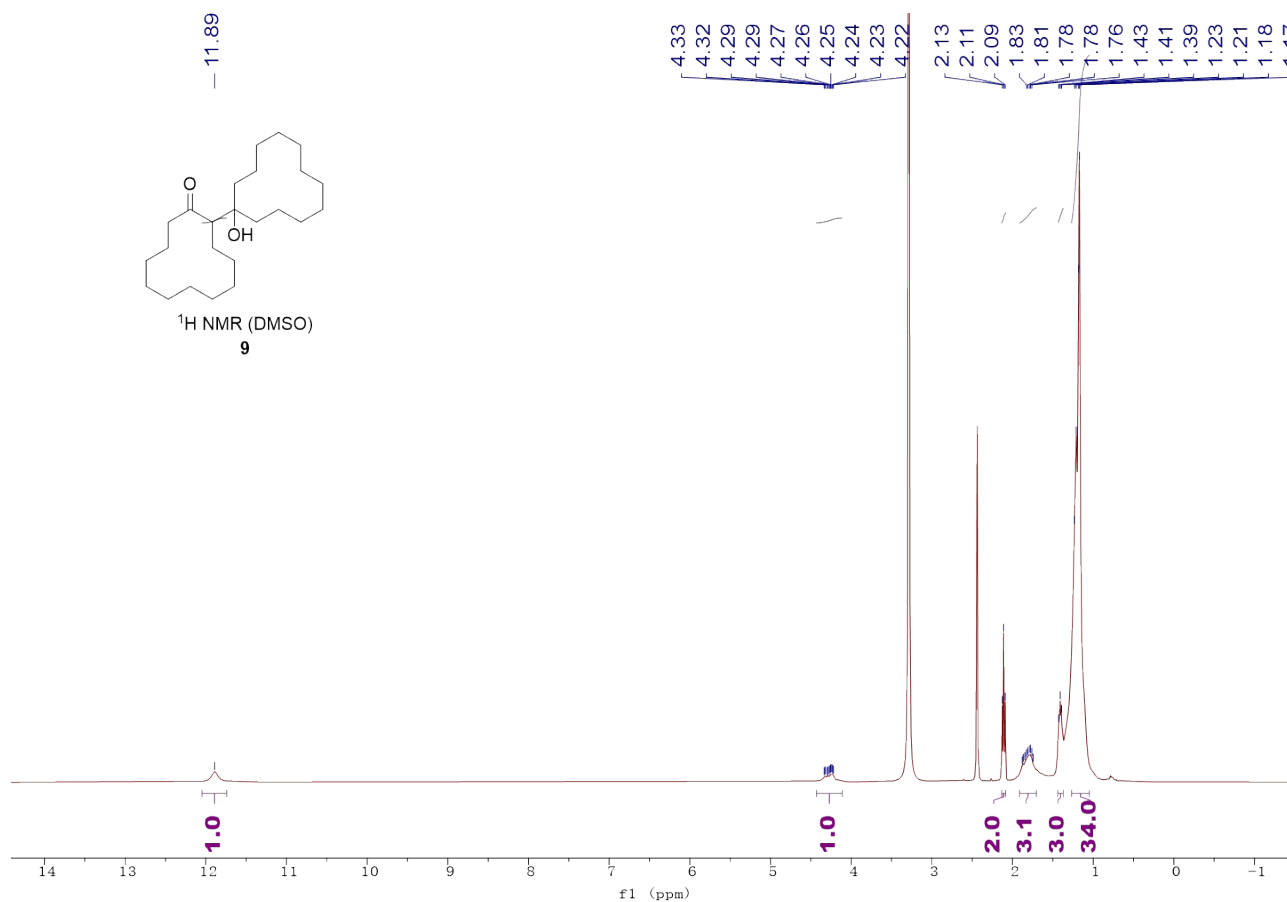


¹³C NMR (75 MHz, CDCl₃) δ 149.42, 121.51, 30.99, 28.93, 28.84, 26.88, 23.90.

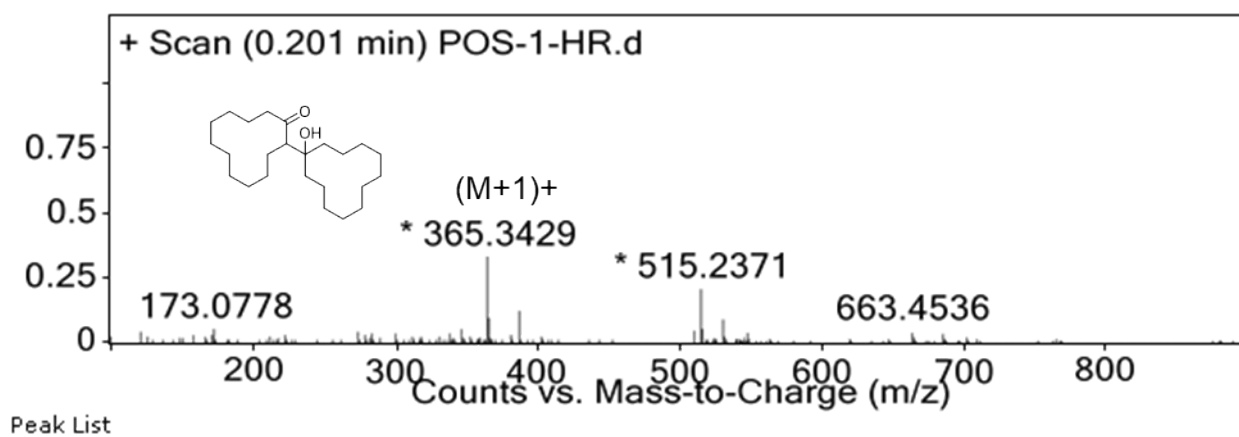
5.2. General Procedure for Compound **9** Derivatives Synthesis and Reaction of β -Hydroxyketones



To a solution of cyclododecanone (3.65 g, 20 mmol) in *n*-hexane (20 mL) was added iodine (10.15 g, 40 mmol). The mixture was heated at 90 °C (oil bath) for 2 h, cooled to room temperature, and concentrated under reduced pressure to give the crudes, which was purified by silica gel column chromatography, eluting with ethyl acetate/petroleum ether (1: 10), to give desired product **9**.

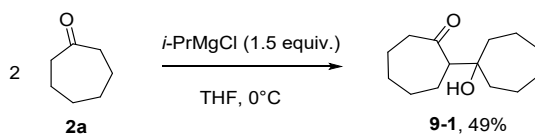


¹H NMR (400 MHz, DMSO) δ 11.89 (s, 1H), 4.43 – 4.11 (m, 1H), 2.11 (t, $J = 7.3$ Hz, 2H), 1.91 – 1.70 (m, 3H), 1.44 – 1.37 (m, 3H), 1.27 – 1.05 (m, 34H).

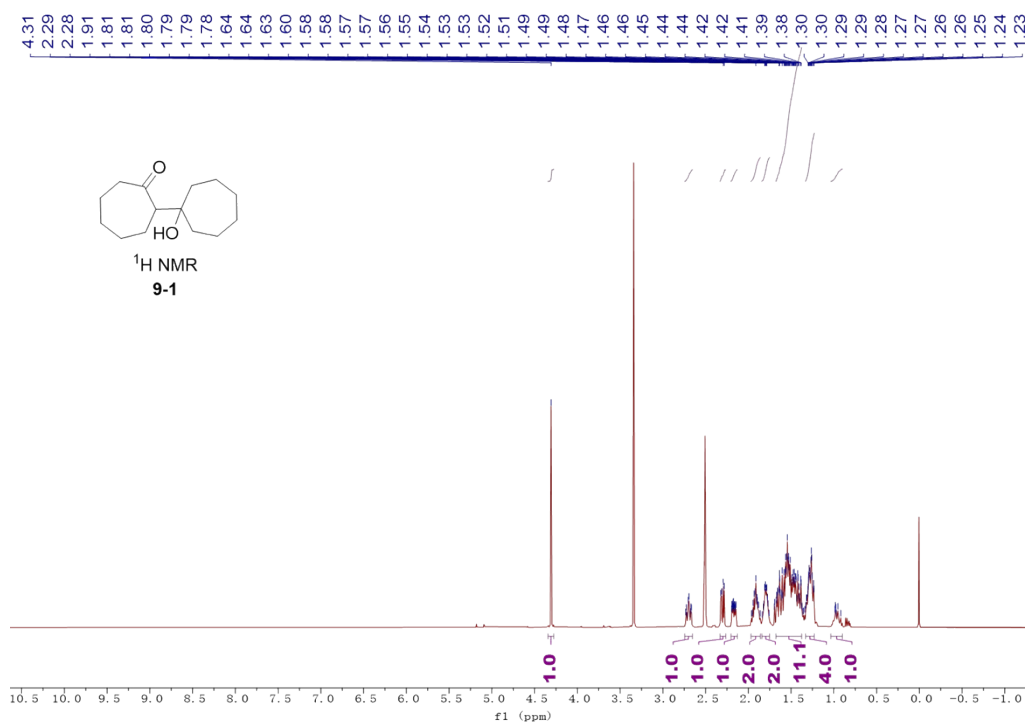


Compound **9** HRMS (ESI) calcd for C₂₄H₄₅O₂ (M+H⁺) 365.3431, found 365.3429.

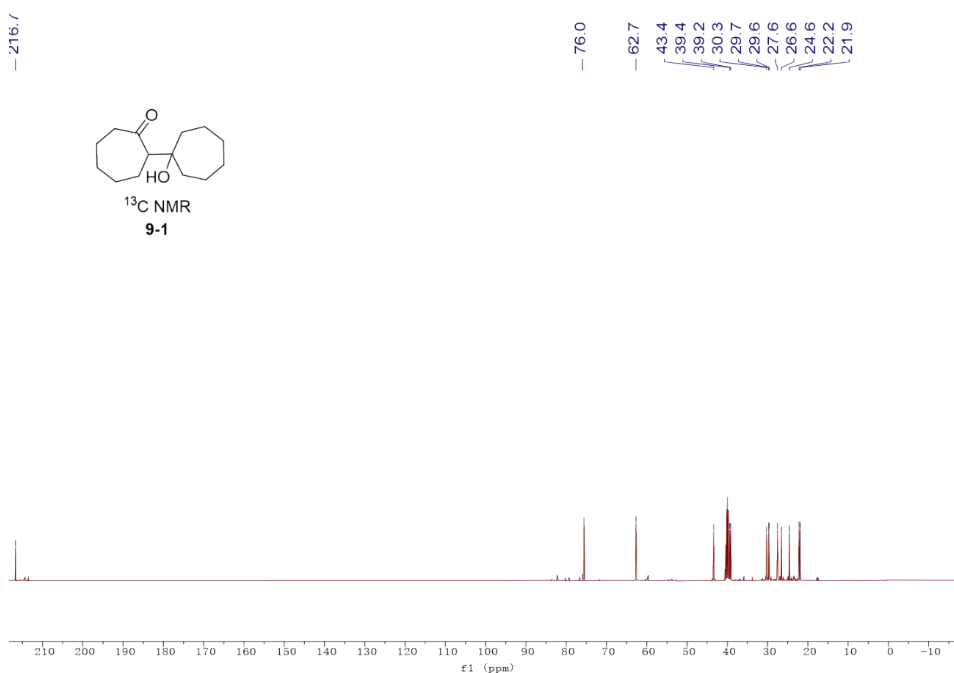
5.3. General Procedure for Compound **9-1** Derivatives Synthesis



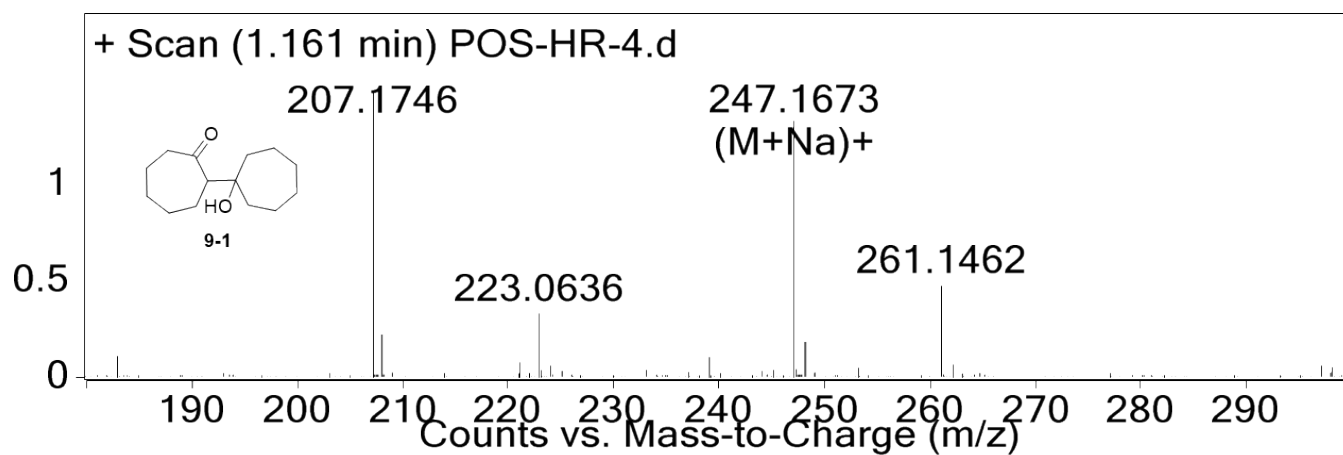
To a solution of cycloheptanone (1.12 g, 10 mmol) in THF, and the mixture was stirred at 0 °C for 10 min. Then, *i*-PrMgCl (7.5 mL, 2.0 M in THF) was added, the mixture was stirred another 4h at 0 °C. After the reaction was completed, quenched with water and extracted with EA, the organic extracts were dried over Na₂SO₄. The crude product was purified by silica gel chromatography using a mixture of ethyl acetate and petroleum ether as eluent to get the desired products **9-1**.



¹H NMR (400 MHz, DMSO) δ 4.31 (s, 1H), 2.70 (td, J = 12.0, 2.9 Hz, 1H), 2.30 (dd, J = 11.7, 4.6 Hz, 1H), 2.20 - 2.13 (m, 1H), 1.97 - 1.86 (m, 2H), 1.84 - 1.75 (m, 2H), 1.68 - 1.38 (m, 11H), 1.33 - 1.23 (m, 4H), 1.04 - 0.90 (m, 1H).

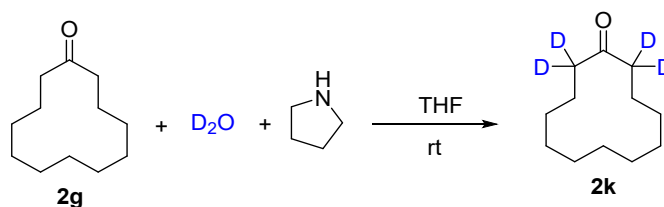


¹³C NMR (101 MHz, DMSO) δ 216.7, 75.9, 62.7, 43.4, 39.4, 39.2, 30.3, 29.7, 29.6, 27.6, 26.6, 24.6, 22.2, 21.9.

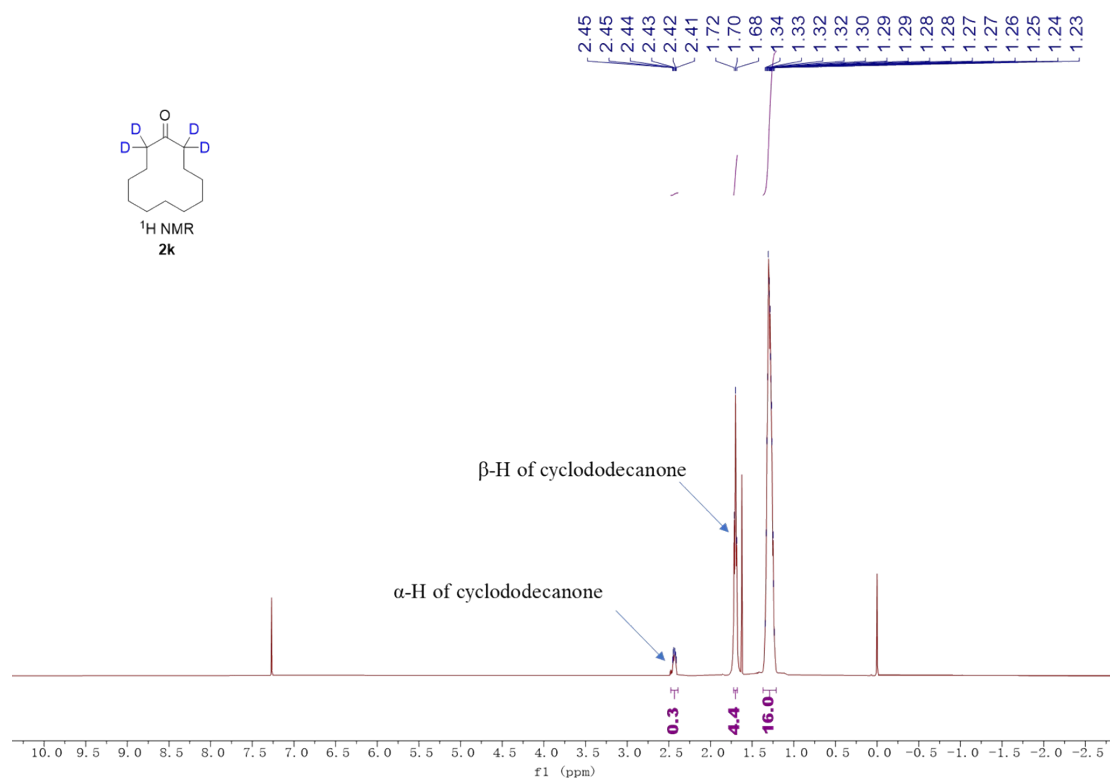


Compound **9-1** HRMS (ESI) calcd for $C_{14}H_{20}O_2$ (M+Na⁺) 247.1669, found 247.1673.

5.4. General Procedure for Deuterium-Labeling Ketones **2k** Synthesis

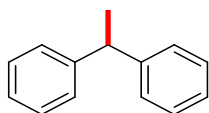


To a round bottom flask, cyclododecone (**2g**) (20 mmol, 3.65g) was dissolved in 10 mL anhydrous THF, D_2O (8.01g, 400 mmol) and tetrahydropyrrole (142.2 mg, 2.0 mmol) was added. The reaction mixture was stirred at room temperature for 48 h. After the reaction was completed, the mixture was acidic with 2 N HCl and extracted with DCM, the organic extracts were dried over Na_2SO_4 . The crude product was purified by silica gel chromatography using a mixture of ethyl acetate and petroleum ether as eluent to get the desired products.



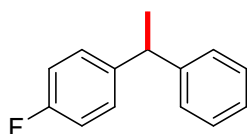
¹H NMR (400 MHz, CDCl₃) δ 2.48 – 2.39 (m, 0H), 1.70 (t, J = 6.4 Hz, 4H), 1.37 – 1.21 (m, 16H).

6. Product Characterization



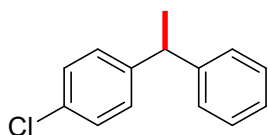
Ethane-1,1-diylidibenzene (**3a**)¹ was obtained as a colorless oil (48.1mg, 88%, from **1a**; 53.0 mg, 97%, from **5g**).

¹H NMR (300 MHz, CDCl₃) δ 7.45 – 7.09 (m, 10H), 4.18 (q, J = 7.2 Hz, 1H), 1.67 (d, J = 7.2 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 146.4, 128.4, 127.6, 126.1, 44.8, 21.9.



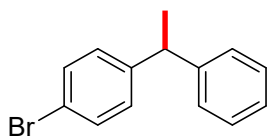
1-Fluoro-4-(1-phenylethyl) benzene (**3b**)² was obtained as a colorless oil (54.1 mg, 90% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.43 – 7.35 (m, 2H), 7.30 – 7.24 (m, 5H), 7.06 (t, J = 8.7 Hz, 2H), 4.23 (q, J = 7.3 Hz, 1H), 1.71 (d, J = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 162.5, 160.12, 146.2, 142.1, 142.1, 129.08, 129.0, 128.5, 127.6, 126.2, 115.2, 115.0, 44.1, 22.1.



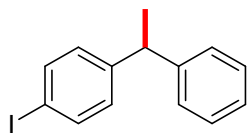
1-Chloro-4-(1-phenylethyl) benzene (**3c**)² was obtained as a colorless oil (40.3 mg, 62% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.37 – 7.29 (m, 2H), 7.29 – 7.27 (m, 2H), 7.27 – 7.18 (m, 3H), 7.22 – 7.14 (m, 2H), 4.16 (q, J = 7.2 Hz, 1H), 1.65 (d, J = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 145.9, 144.9, 131.8, 129.1, 128.6, 128.5, 127.6, 126.3, 44.2, 21.9.



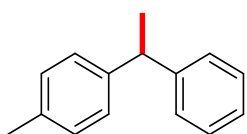
1-Bromo-4-(1-phenylethyl) benzene (**3d**)³ was obtained as a colorless oil (71.3 mg, 91% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.41 – 7.35 (m, 2H), 7.32 – 7.24 (m, 2H), 7.21 – 7.15 (m, 3H), 7.11 – 7.04 (m, 2H), 4.09 (q, *J* = 7.2 Hz, 1H), 1.60 (d, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 145.8, 145.4, 131.5, 129.5, 128.5, 127.6, 126.3, 119.9, 44.3, 21.8.



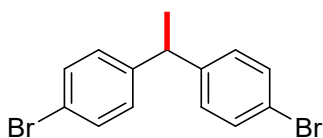
1-Iodo-4-(1-phenylethyl) benzene (**3e**)⁴ was obtained as a white solid at room temperature (60.1 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.65 (m, 2H), 7.40 – 7.31 (m, 2H), 7.29 – 7.24 (m, 3H), 7.06 – 7.02 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 1H), 1.68 (d, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 146.2, 145.8, 137.5, 129.9, 128.6, 127.7, 126.4, 91.4, 44.4, 21.8.



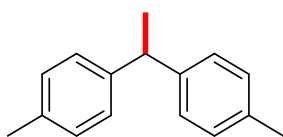
1-Methyl-4-(1-phenylethyl) benzene (**3f**)² was obtained as a colorless oil (46.5 mg, 79% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.31 (m, 2H), 7.30 – 7.26 (m, 2H), 7.26 – 7.20 (m, 1H), 7.20 – 7.14 (m, 4H), 4.18 (q, *J* = 7.2 Hz, 1H), 2.37 (s, 3H), 1.69 (d, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 146.7, 143.5, 135.6, 129.1, 128.4, 127.7, 127.6, 126.0, 44.5, 22.0, 21.1.



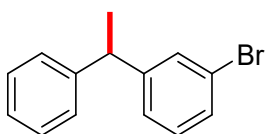
4,4'-(Ethane-1,1-diyl) bis(bromobenzene) (**3g**)⁵ was obtained as a white solid at room temperature (65.3 mg, 64% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.49 – 7.38 (m, 4H), 7.14 – 7.03 (m, 4H), 4.08 (q, *J* = 7.2 Hz, 1H), 1.61 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 144.8, 131.6, 129.4, 120.1, 43.7, 21.7.



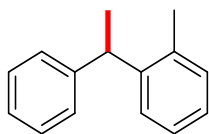
4,4'-(Ethane-1,1-diyl) bis(methylbenzene) (**3h**)⁵ was obtained as a colorless oil (34.7 mg, 55% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.20 – 7.14 (m, 8H), 4.16 (q, *J* = 7.2 Hz, 1H), 2.38 (s, 6H), 1.68 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 143.6, 135.3, 128.9, 127.3, 43.8, 21.9, 20.9.



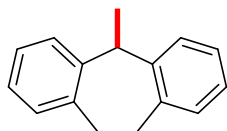
1-Bromo-3-(1-phenylethyl) benzene (**3i**)⁶ was obtained as a colorless oil (40.0 mg, 51% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.39 (d, *J* = 1.7 Hz, 1H), 7.36 – 7.31 (m, 2H), 7.25 – 7.20 (m, 3H), 7.18 – 7.14 (m, 2H), 4.13 (q, *J* = 7.3 Hz, 1H), 1.64 (d, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 148.8, 145.5, 130.7, 129., 129.2, 128.6, 127.6, 126.4, 126.4, 122.6, 44.6, 21.7.



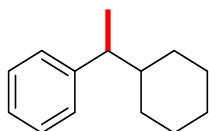
1-Methyl-2-(1-phenylethyl) benzene (**3j**)² was obtained as a colorless oil (53.0mg, 90% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.30 – 7.16 (m, 4H), 7.19 – 7.09 (m, 5H), 4.31 (q, *J* = 7.2 Hz, 1H), 2.23 (s, 3H), 1.60 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 146.3, 143.9, 136.2, 130.5, 128.4, 127.8, 126.8, 126.2, 126.1, 125.9, 41.1, 22.2, 19.8.



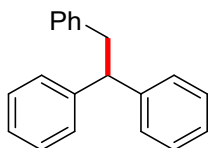
5-Methyl-10,11-dihydro-5H-dibenzo[*a,d*][7]annulene (**3k**)⁷ was obtained as a colorless oil (41.8 mg, 67% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.26 – 7.17 (m, 2H), 7.18 – 7.04 (m, 6H), 4.42 (q, *J* = 7.4 Hz, 1H), 3.20 (s, 4H), 1.71 (d, *J* = 7.4 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 143.3, 139.3, 130.1, 127.5, 126.4, 126.2, 77.3, 43.7, 33.3, 22.0.



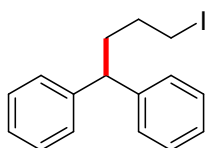
(1-Cyclohexylethyl) benzene (**3l**)² was obtained as a colorless oil (24.8 mg, 44% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.27 (m, 2H), 7.25 – 7.15 (m, 3H), 2.48 (p, *J* = 7.2 Hz, 1H), 1.99 – 1.87 (m, 1H), 1.83 – 1.73 (m, 1H), 1.72 – 1.60 (m, 2H), 1.50 – 1.40 (m, 2H), 1.36 – 1.22 (m, 1H), 1.27 (d, *J* = 7.1 Hz, 3H), 1.25 – 1.05 (m, 2H), 1.03 – 0.93 (m, 1H), 0.92 – 0.78 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 147.2, 128.0, 127.8, 125.7, 45.9, 44.2, 31.5, 30.7, 26.6, 26.6, 18.9.



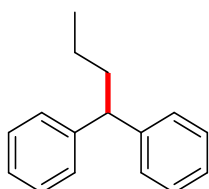
Ethane-1,1,2-triyltribenzene (**3m**)⁸ was obtained as a white solid at room temperature (24.8 mg, 32% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.30 – 7.28 (m, 3H), 7.27 – 7.21 (m, 6H), 7.21 – 7.12 (m, 4H), 7.07 – 6.99 (m, 2H), 4.27 (t, *J* = 7.8 Hz, 1H), 3.40 (d, *J* = 7.8 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 144.6, 140.4, 129.2, 128.4, 128.2, 126.5, 126.3, 126.0, 53.2, 42.2.



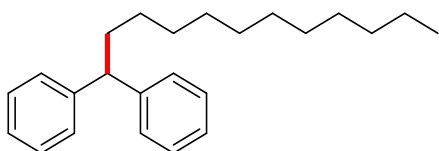
(4-Iodobutane-1,1-diyl) dibenzene (**3n**)⁹ was obtained as a colorless oil (64.6 mg, 64% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.32 – 7.14 (m, 10H), 3.91 (t, *J* = 7.9 Hz, 1H), 3.18 (t, *J* = 6.8 Hz, 2H), 2.25 – 2.09 (m, 2H), 1.86 – 1.69 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 144.5, 128.6, 127.8, 126.4, 50.5, 36.5, 31.8, 7.0.



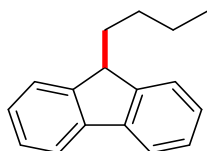
Butane-1,1-diyl dibenzene (**3o**)¹⁰ was obtained as a colorless oil (44.2 mg, 70% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.38 – 7.32 (m, 8H), 7.21 – 7.09 (m, 2H), 3.90 (t, *J* = 7.8 Hz, 1H), 2.14 – 1.93 (m, 2H), 1.38 – 1.18 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 145.4, 128.4, 128.0, 126.1, 51.2, 38.0, 21.2, 14.2.



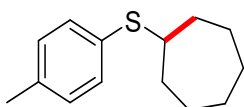
Dodecane-1,1-diyl dibenzene (**3p**)¹¹ was obtained as a colorless oil (78.4 mg, 81% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.30 – 7.19 (m, 8H), 7.19 – 7.11 (m, 2H), 3.87 (t, *J* = 7.8 Hz, 1H), 2.02 (q, *J* = 7.7 Hz, 2H), 1.34 – 1.18 (m, 20H), 0.87 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 145.4, 128.4, 127.9, 126.0, 51.4, 35.8, 31.9, 29.7, 29.7, 29.5, 29.4, 28.1, 22.7, 14.2.



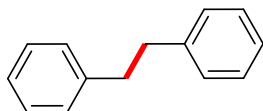
9-Butyl-9H-fluorene (**3q**)¹² was obtained as a colorless oil with (46.7 mg, 70%, from **1q**; 35.0 mg, 53%, from **5h**).

¹H NMR (400 MHz, CDCl₃) δ 7.85 – 7.78 (m, 2H), 7.57 (d, *J* = 7.4 Hz, 2H), 7.46 – 7.32 (m, 4H), 4.03 (t, *J* = 5.9 Hz, 1H), 2.12 – 2.02 (m, 2H), 1.36 – 1.32 (m, 2H), 1.28 – 1.19 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 147.7, 141.2, 126.9, 126.8, 124.4, 119.8, 47.5, 32.8, 27.8, 23.1, 14.0.



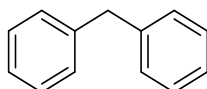
Cycloheptyl (*p*-tolyl) sulfane (**3r**)¹³ was obtained as a colorless oil (52.2 mg, 79% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.24 – 7.15 (m, 2H), 7.01 (d, *J* = 7.9 Hz, 2H), 3.17 (tt, *J* = 9.0, 4.1 Hz, 1H), 2.23 (s, 3H), 1.99 – 1.83 (m, 2H), 1.67 – 1.57 (m, 2H), 1.55 – 1.26 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 136.6, 132.0, 129.5, 48.6, 34.6, 28.2, 25.9, 21.0.



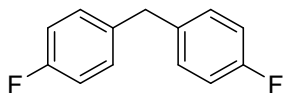
1,2-Diphenylethane (**3s**)¹ was obtained as a colorless oil (13.1 mg, 24% from **1s**; 4.9 mg, 9% from **1t**; 20.8 mg, 38% from **1u**).

¹H NMR (300 MHz, CDCl₃) δ 7.35 – 7.23 (m, 4H), 7.22 – 7.16 (m, 6H), 2.92 (s, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 141., 128.5, 128.3, 125.9, 37.9.



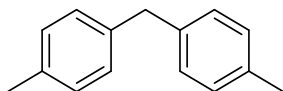
Diphenylmethane (**6a**)¹⁴ was obtained as a colorless oil (31.8 mg, 63%, from **5a**; 44.4 mg, 88%, from **5k**; 38.5 mg, 76%, from **5l**; 100.3 mg, 99%, from **5m**; 11.1 mg, 22%, from **5p**; 25.3 mg, 50%, from **5q**; 47.1 mg, 93%, from **5s**).

¹H NMR (300 MHz, CDCl₃) δ 7.32 – 7.24 (m, 4H), 7.22 – 7.17 (m, 6H), 3.98 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 141.2, 128.9, 128.5, 126.1, 42.0.



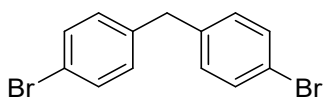
Bis(4-fluorophenyl) methane (**6b**)¹⁴ was obtained as a colorless oil (25.1 mg, 41% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.29 (d, *J* = 8.6 Hz, 3H), 7.24 – 7.14 (m, 6H), 3.97 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 141.3, 129.52, 128.7, 126.3, 42.2.



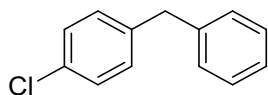
Di-*p*-tolylmethane (**6c**)¹⁴ was obtained as a colorless oil (31.2 mg, 53% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.15 (s, 8H), 3.98 (s, 2H), 2.38 (s, 6H); ¹³C NMR (101 MHz, CDCl₃) δ 138.5, 135.5, 129.2, 128.9, 41.2, 21.1.



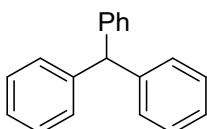
Bis(4-bromophenyl) methane (**6d**)¹⁵ was obtained as a colorless oil (49.9 mg, 51% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.39 – 7.30 (m, 4H), 7.31 – 7.21 (m, 4H), 4.05 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 141.2, 129.0, 128.6, 126.2, 42.0.



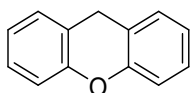
1-Benzyl-4-chlorobenzene (**6e**)¹⁵ was obtained as a colorless oil (18.8 mg, 31% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.29 (d, *J* = 8.6 Hz, 3H), 7.24 – 7.14 (m, 6H), 3.97 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 141.3, 129.2, 128.7, 126.3, 42.2.



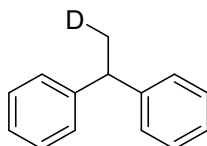
Triphenylmethane (**6f**)¹⁶ was obtained as a white solid at room temperature with (69.6 mg, 95%, from **5f**; 71.8 mg, 98%, from **5j**; 56.4 mg, 77%, from **5n**; 71.8 mg, 98%, from **5o**; 69.6 mg, 95%, from **5r**).

¹H NMR (300 MHz, CDCl₃) δ 7.33 – 7.14 (m, 9H), 7.16 – 7.07 (m, 6H), 5.55 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 143.7, 129.3, 128.1, 126.1, 56.7.



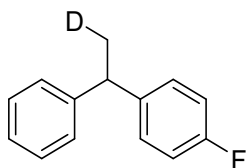
9H-xanthene (**6g**)¹⁷ was obtained as a colorless oil (24.0 mg, 44% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.27 – 7.16 (m, 4H), 7.11 – 7.01 (m, 4H), 4.08 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 128.9, 127.6, 123.0, 116.5, 27.9.



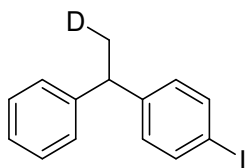
Deuterium ethane-1,1-diylidibenzene (**7a**)¹⁸ was obtained as a colorless oil (42.9 mg, 78% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.27 (t, *J* = 7.4 Hz, 4H), 7.25 – 7.18 (m, 4H), 7.17 – 7.15 (m, 2H), 4.15 (q, *J* = 6.7 Hz, 1H), 1.63 (t, *J* = 6.9 Hz, 2H).



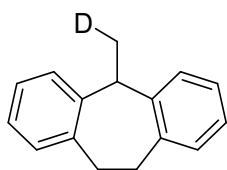
Deuterium 1-fluoro-4-(1-phenylethyl) benzene (**7b**) was obtained as a colorless oil (52.5 mg, 87% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.33 – 7.23 (m, 2H), 7.23 – 7.13 (m, 5H), 6.96 (t, *J* = 8.7 Hz, 2H), 4.12 (t, *J* = 6.1 Hz, 1H), 1.63 – 1.58 (m, 2H); IR (film): ν 2924, 2860, 1606, 1508, 1446 cm⁻¹; HRMS (EI) calcd for C₁₄H₁₂DF (M): 201.1064, found: 201.1075.



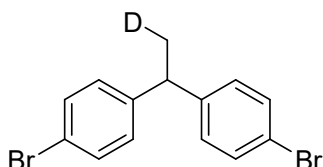
Deuterium 1-iodo-4-(1-phenylethyl) benzene (**7c**) was obtained as a colorless oil (45.4 mg, 49% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.58 (d, $J = 8.1$ Hz, 2H), 7.25 (s, 2H), 7.21 – 7.15 (m, 3H), 6.96 (d, $J = 8.0$ Hz, 2H), 4.19 – 4.02 (m, 1H), 1.65 – 1.55 (m, 2H); IR (film): ν 3057, 3020, 2958, 2937, 1481, 1452, 1398 cm^{-1} ; HRMS (EI) calcd for $\text{C}_{14}\text{H}_{12}\text{D}$ (M): 309.0125, found: 309.0121.



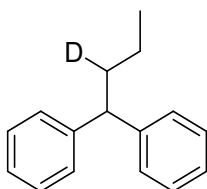
Deuterium 5-Methyl-10,11-dihydro-5H-dibenzo[a,d][7]annulene (**7d**) was obtained as a colorless oil (47.7 mg, 76% yield).

^1H NMR (300 MHz, CDCl_3) δ 7.21 – 7.13 (m, 2H), 7.12 – 6.99 (m, 6H), 4.44 – 4.33 (m, 1H), 3.15 (s, 4H), 1.67 – 1.63 (m, 2H); IR (film): ν 3053, 3014, 2926, 2248, 2220, 1489, 1460 cm^{-1} ; HRMS (EI) calcd for $\text{C}_{16}\text{H}_{15}\text{D}$ (M): 209.1315, found: 209.1325.



Deuterium 4,4'-(ethane-1,1-diyl) bis(bromobenzene) (**7e**) was obtained as a white solid at room temperature (59.3 mg, 58% yield).

^1H NMR (300 MHz, CDCl_3) δ 7.40 – 7.36 (m, 4H), 7.04 (d, $J = 8.4$ Hz, 4H), 4.04 (t, $J = 6.1$ Hz, 1H), 1.58 – 1.54 (m, 2H); IR (film): ν 2353, 1485 cm^{-1} ; HRMS (EI) calcd for $\text{C}_{14}\text{H}_{11}\text{DBr}_2$ (M): 338.9369, found: 338.9366.



Deuterium butane-1,1-diyl dibenzene (**7f**) was obtained as a colorless oil (48.2 mg, 76% yield).

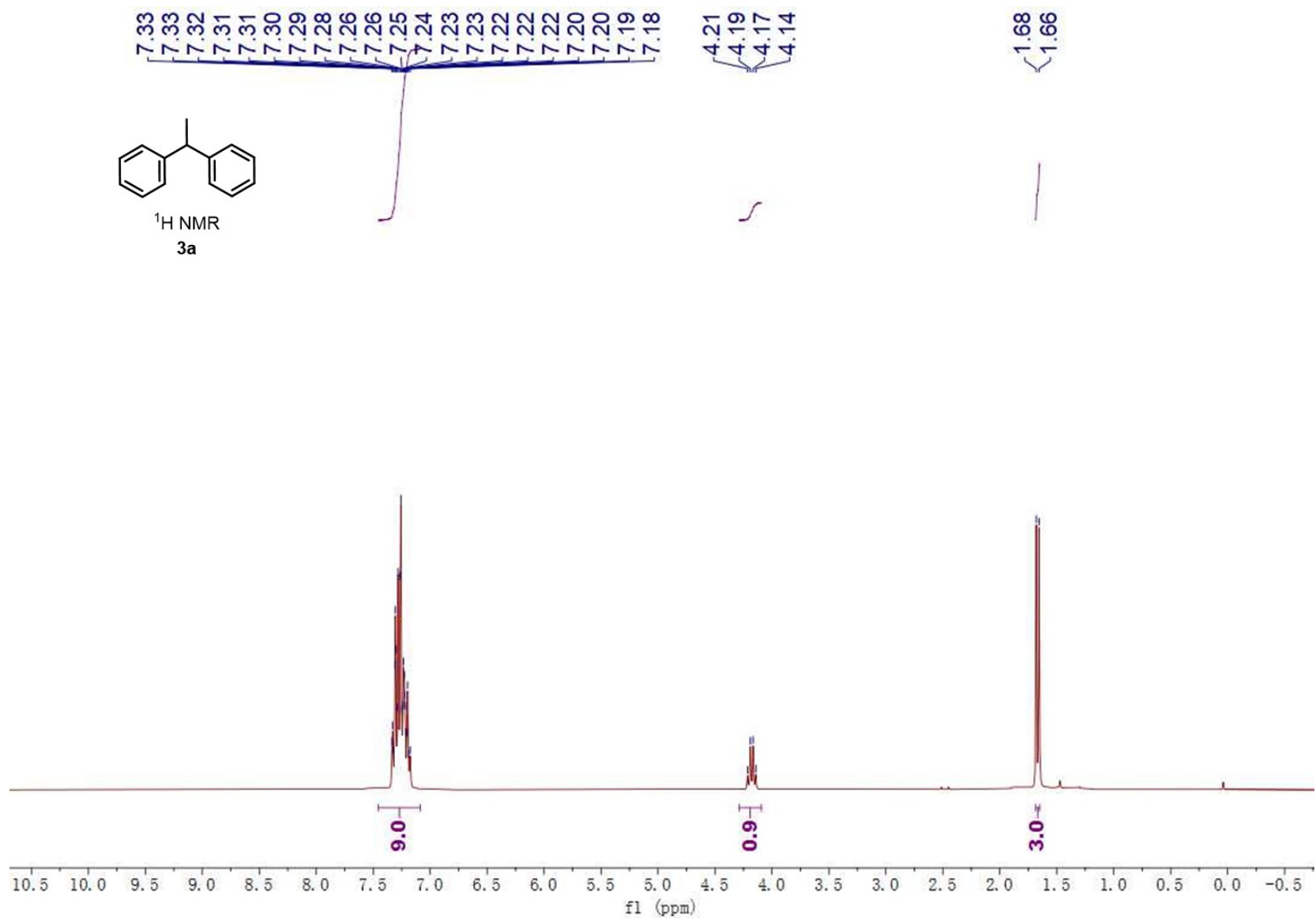
^1H NMR (300 MHz, CDCl_3) δ 7.26 – 7.22 (m, 5H), 3.89 (d, $J = 6.8$ Hz, 1H), 1.99 (d, $J = 7.6$ Hz, 1H), 1.25 (d, $J = 7.1$ Hz, 2H), 0.91 (t, $J = 7.3$ Hz, 3H); IR (film): ν 3062, 3028, 2954, 2926, 2872, 1600, 1490, 1448 cm^{-1} ; HRMS (EI) calcd for $\text{C}_{16}\text{H}_{17}\text{D}$ (M): 211.1471, found: 211.1470.

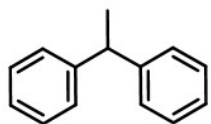
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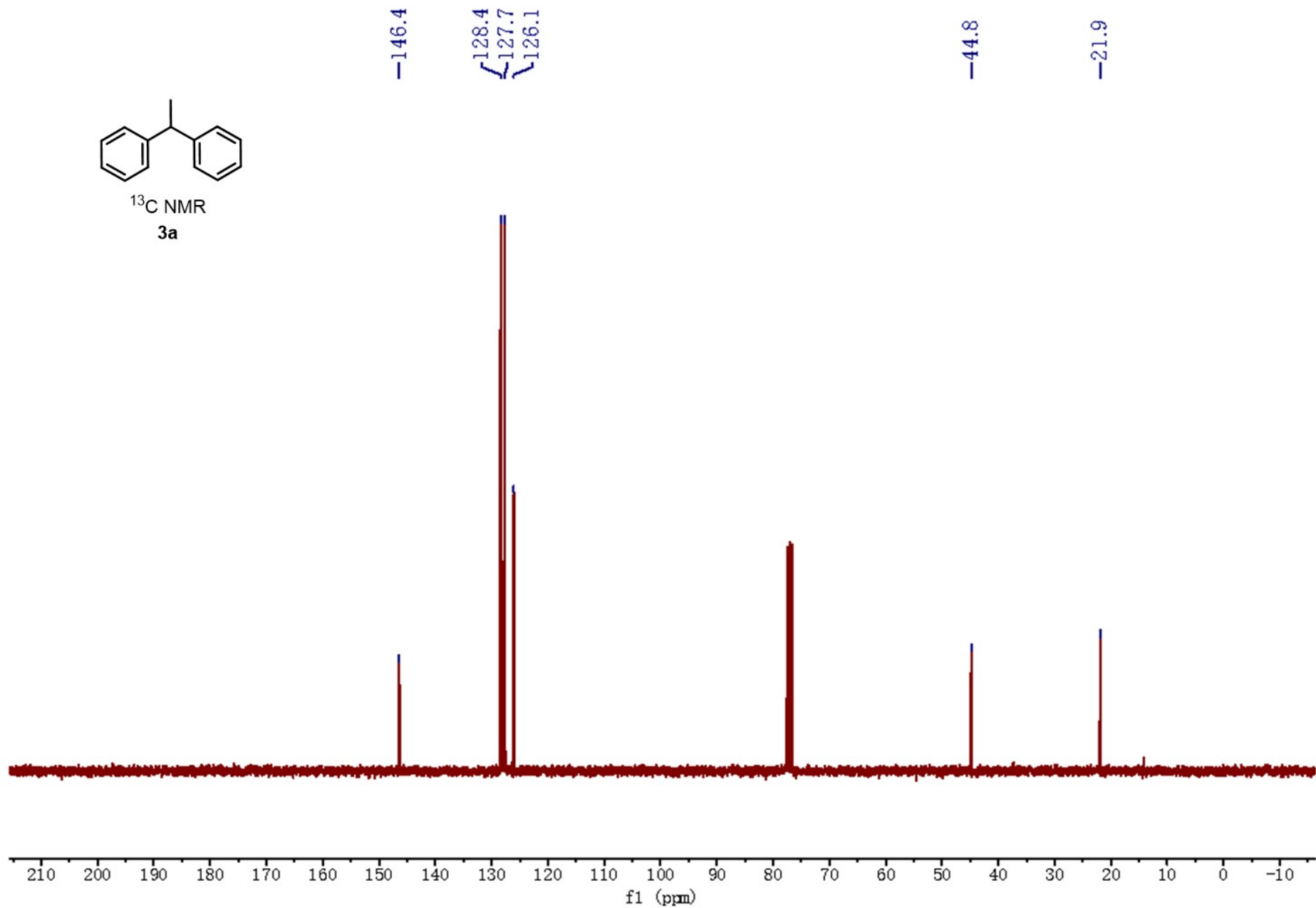
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8. Product NMR Spectra

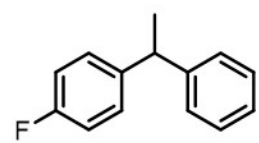




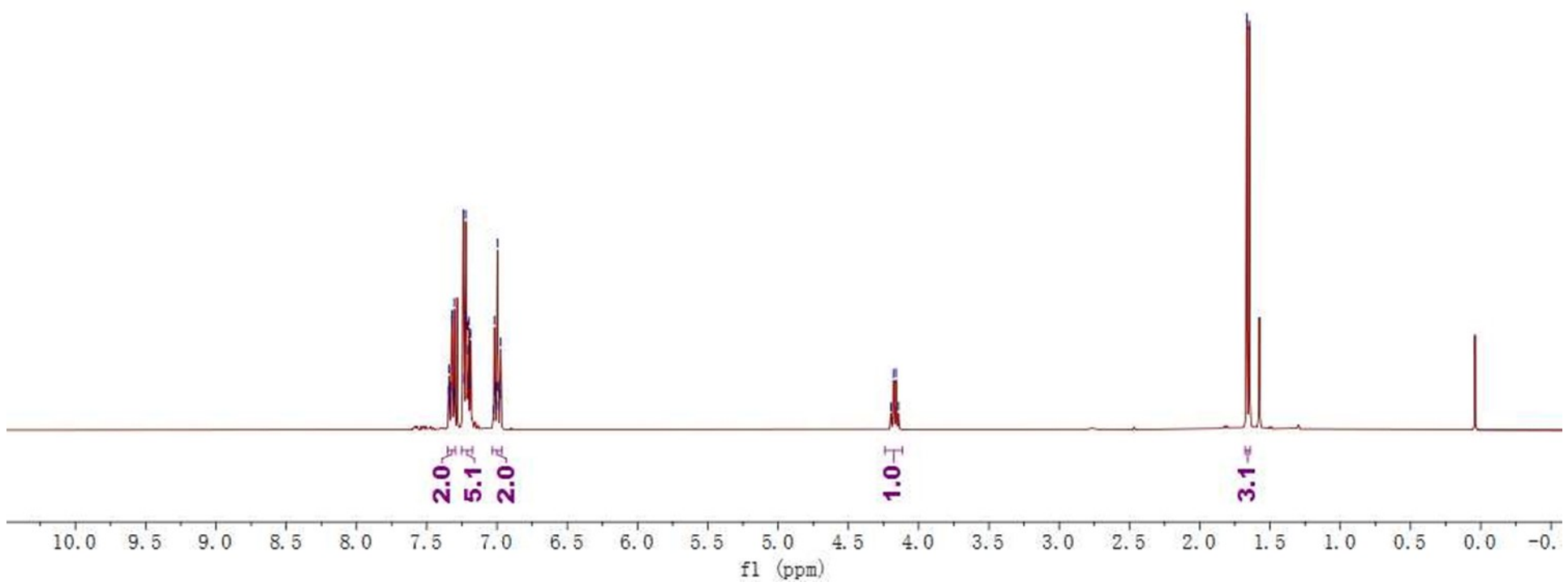
¹³C NMR
3a



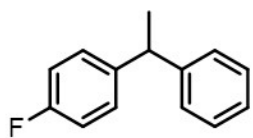
7.34
7.34
7.34
7.33
7.32
7.32
7.31
7.30
7.30
7.24
7.23
7.22
7.22
7.21
7.20
7.20
7.19
7.19
7.02
7.01
7.00
7.00
7.00
6.99
6.99
6.98
6.97
6.20
4.18
4.16
4.14



¹H NMR
3b

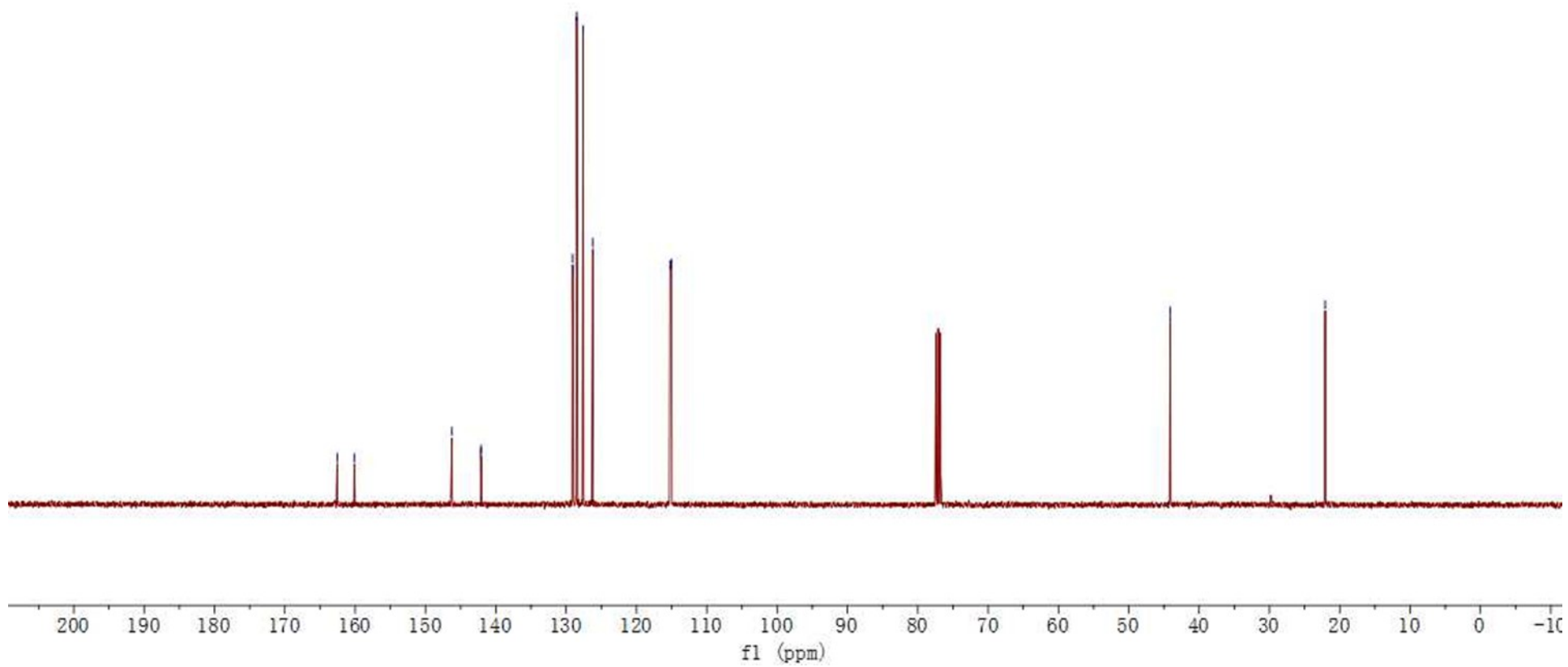


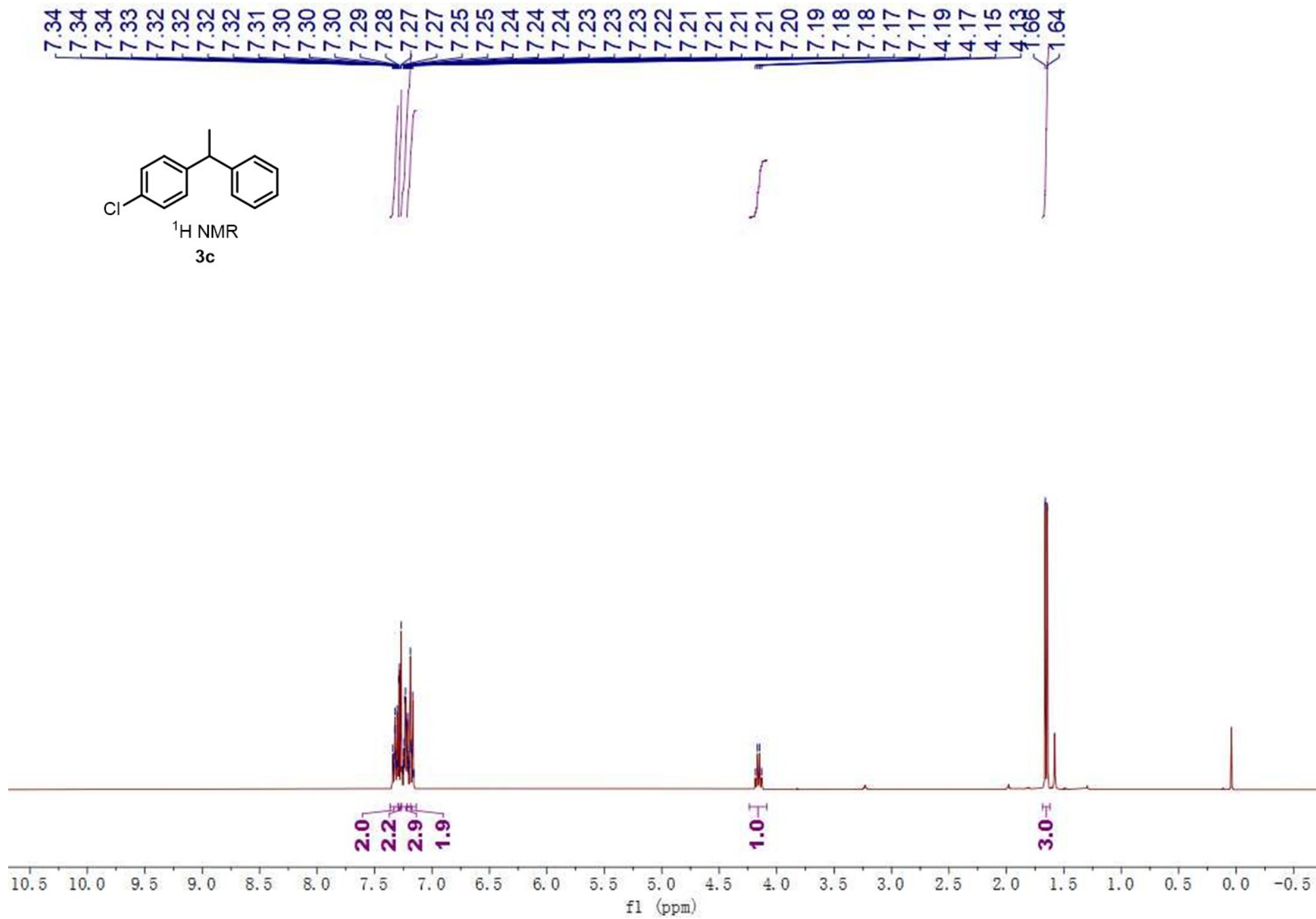
1.66
1.65

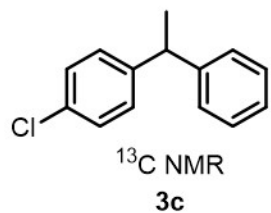


¹³C NMR
3b

~162.5
~160.1
146.2
142.1
142.1
129.1
129.0
128.5
127.6
126.2
115.2
115.0
-44.1
-22.1



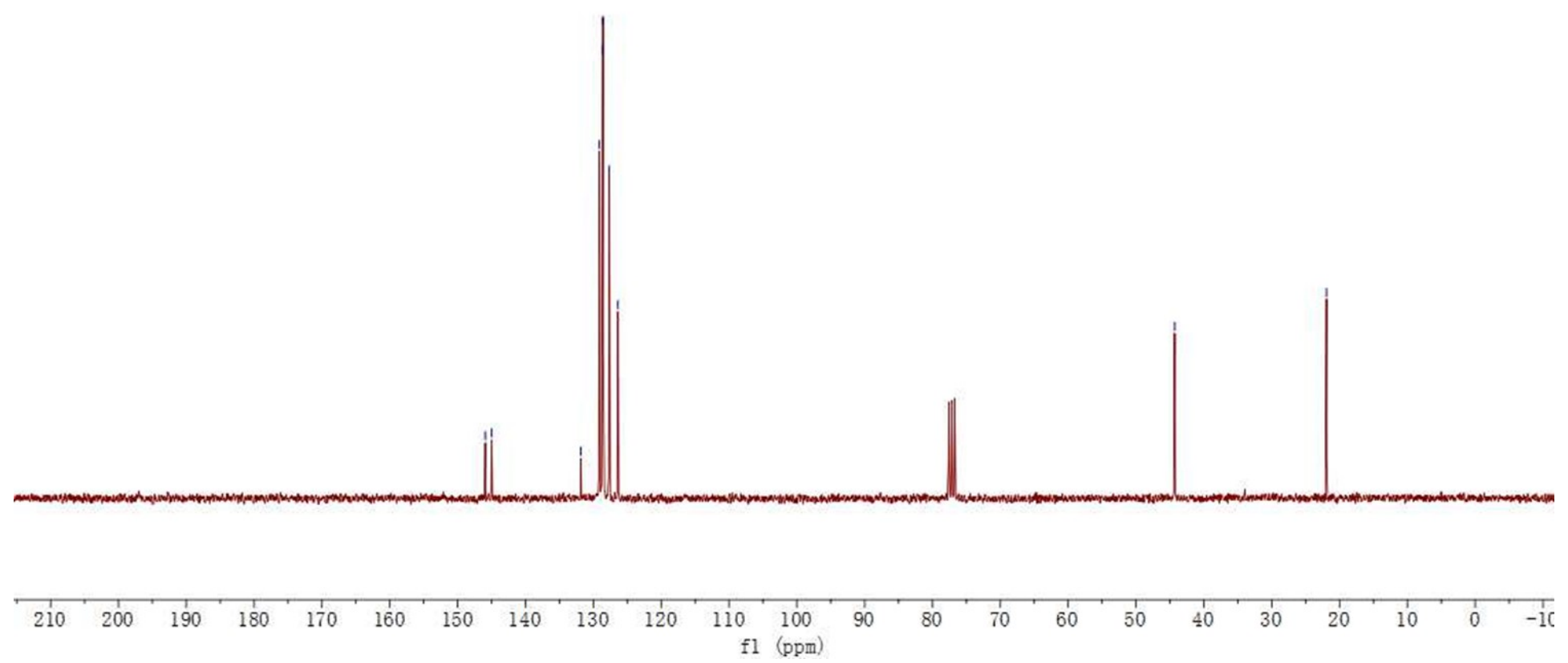


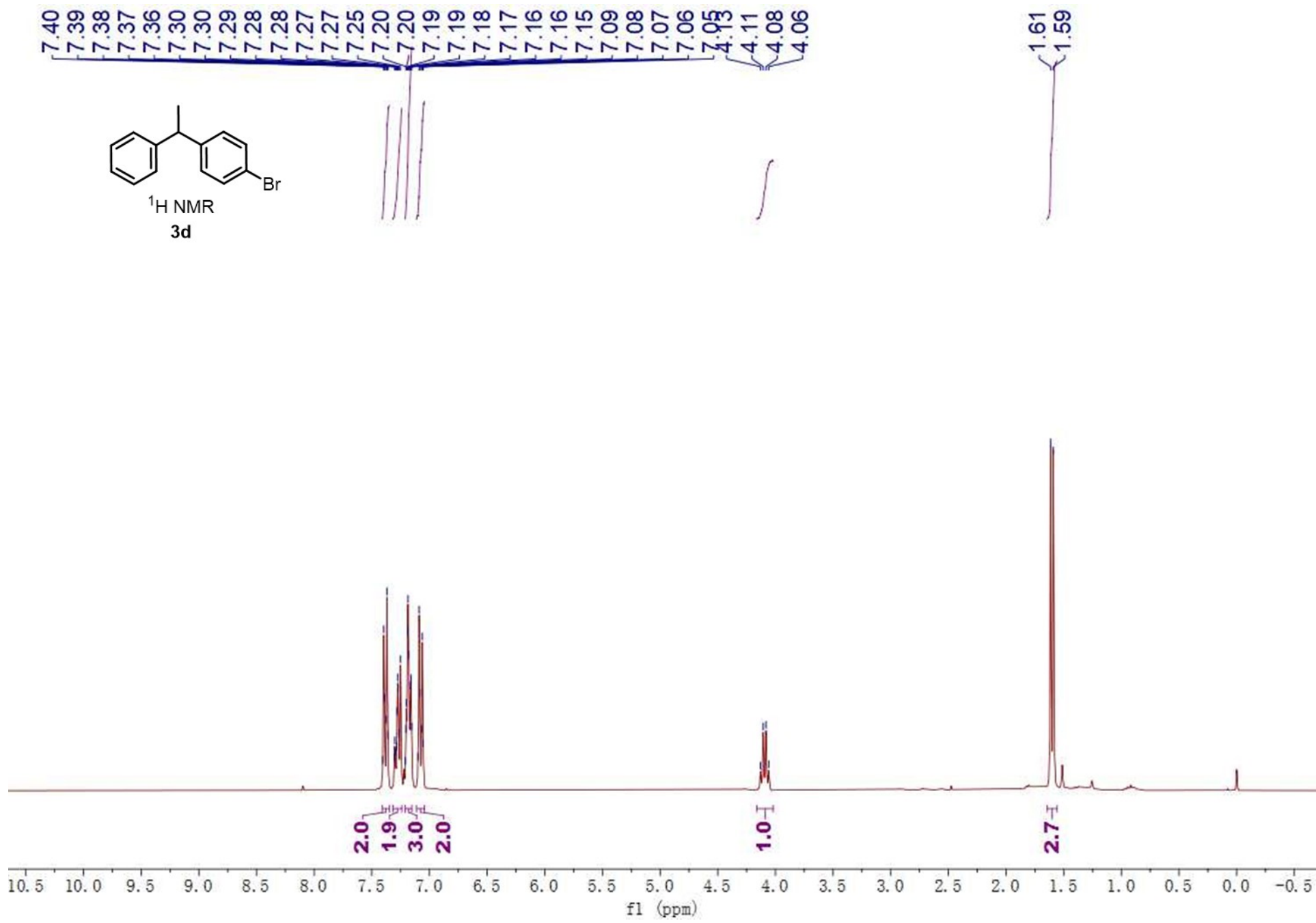


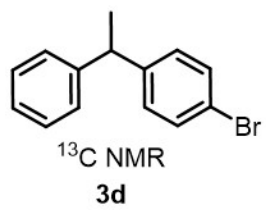
145.9
145.0
131.9
129.1
128.6
128.6
127.7
126.4

-44.3

-21.9



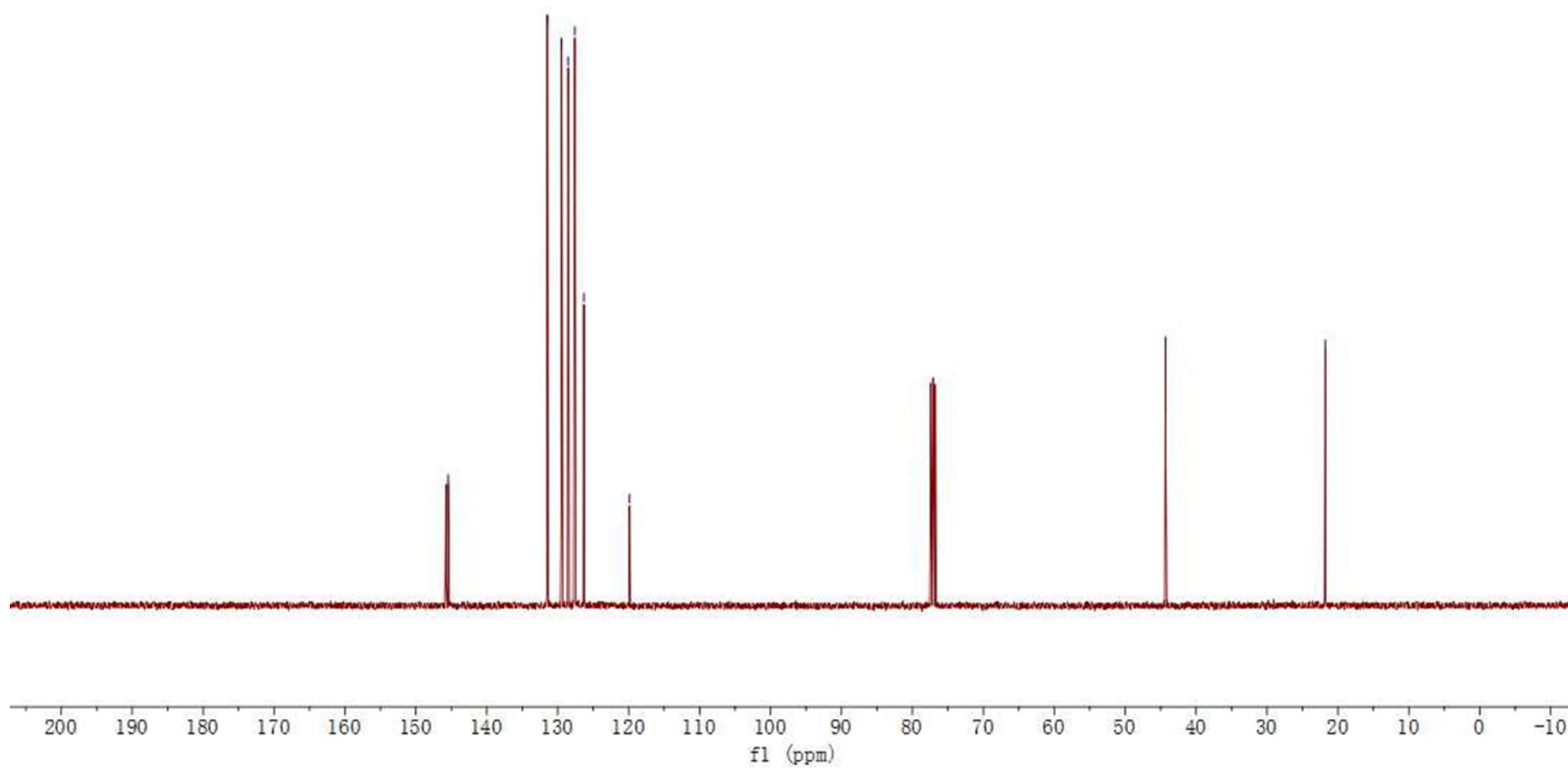


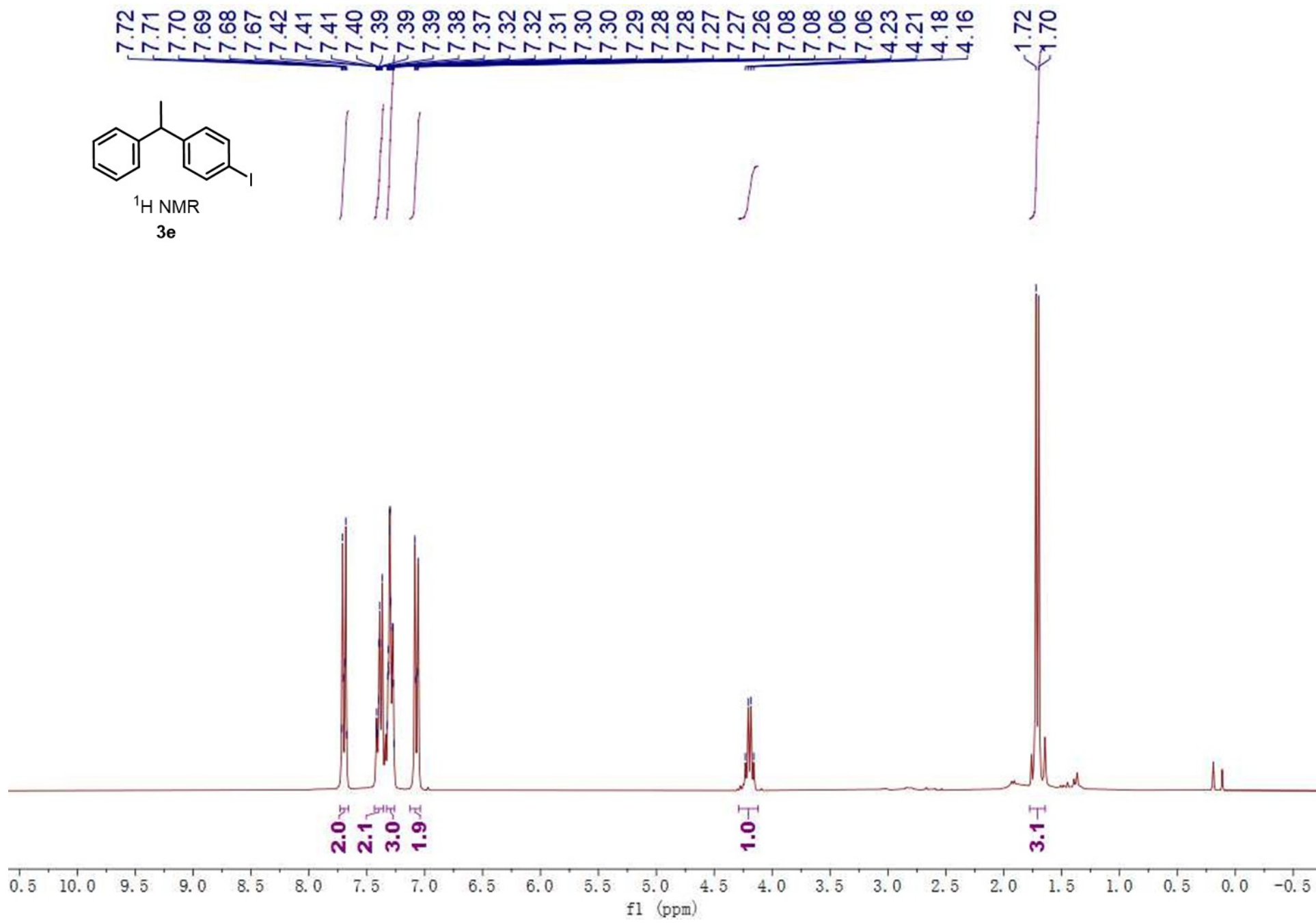


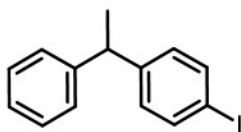
145.8
145.5
131.5
129.5
128.5
127.6
126.3
119.9

-44.3

-21.8







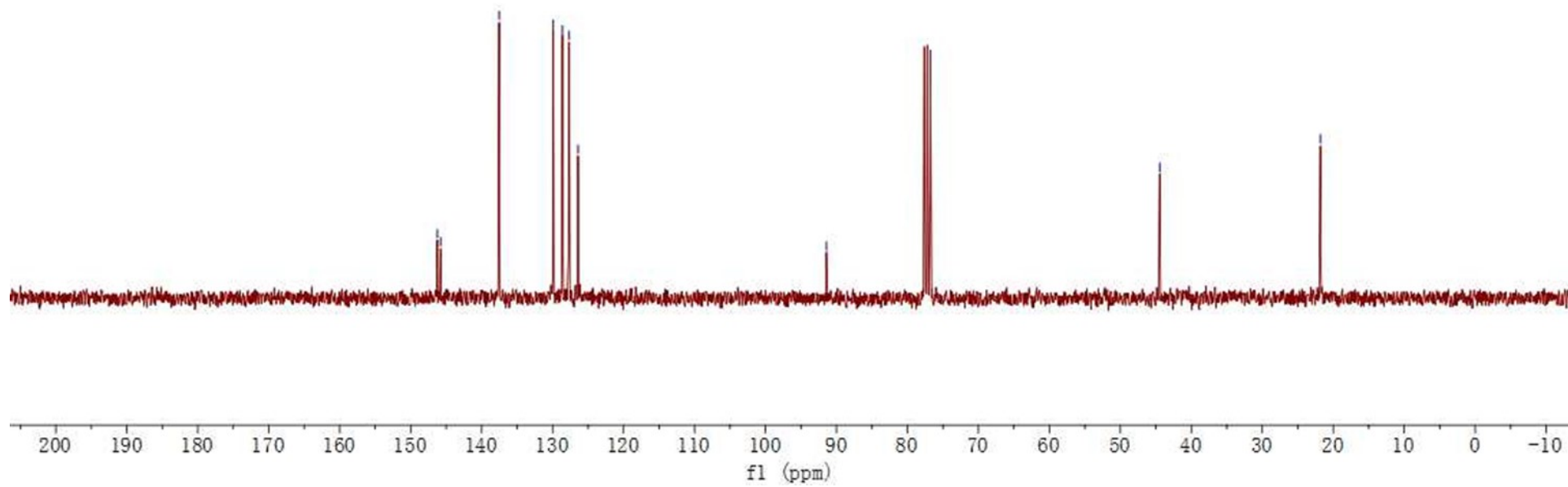
¹³C NMR
3e

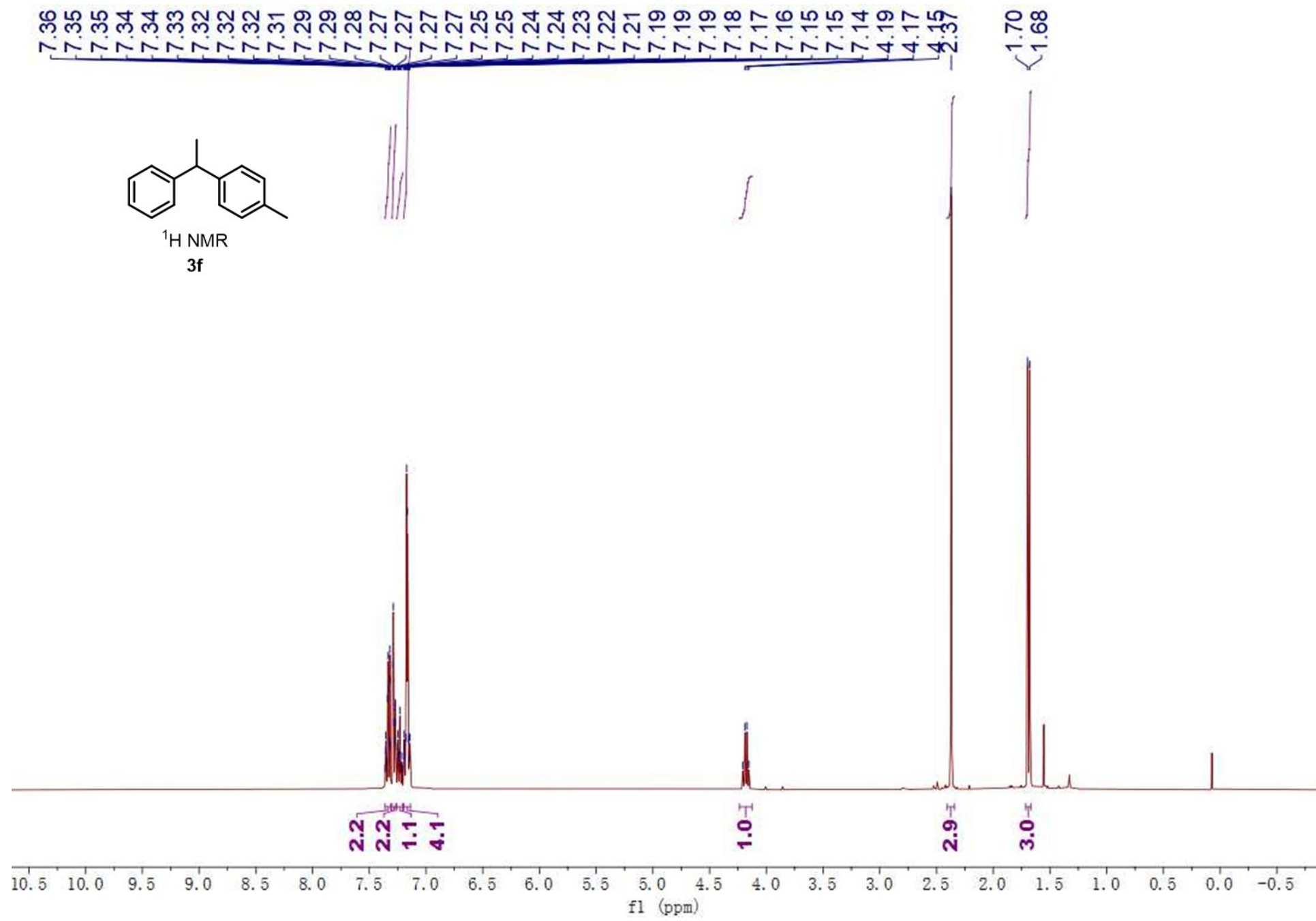
146.2
145.8
137.5
129.9
128.6
127.7
126.4

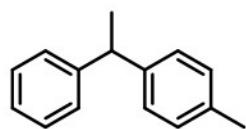
-91.4

-44.5

-21.8





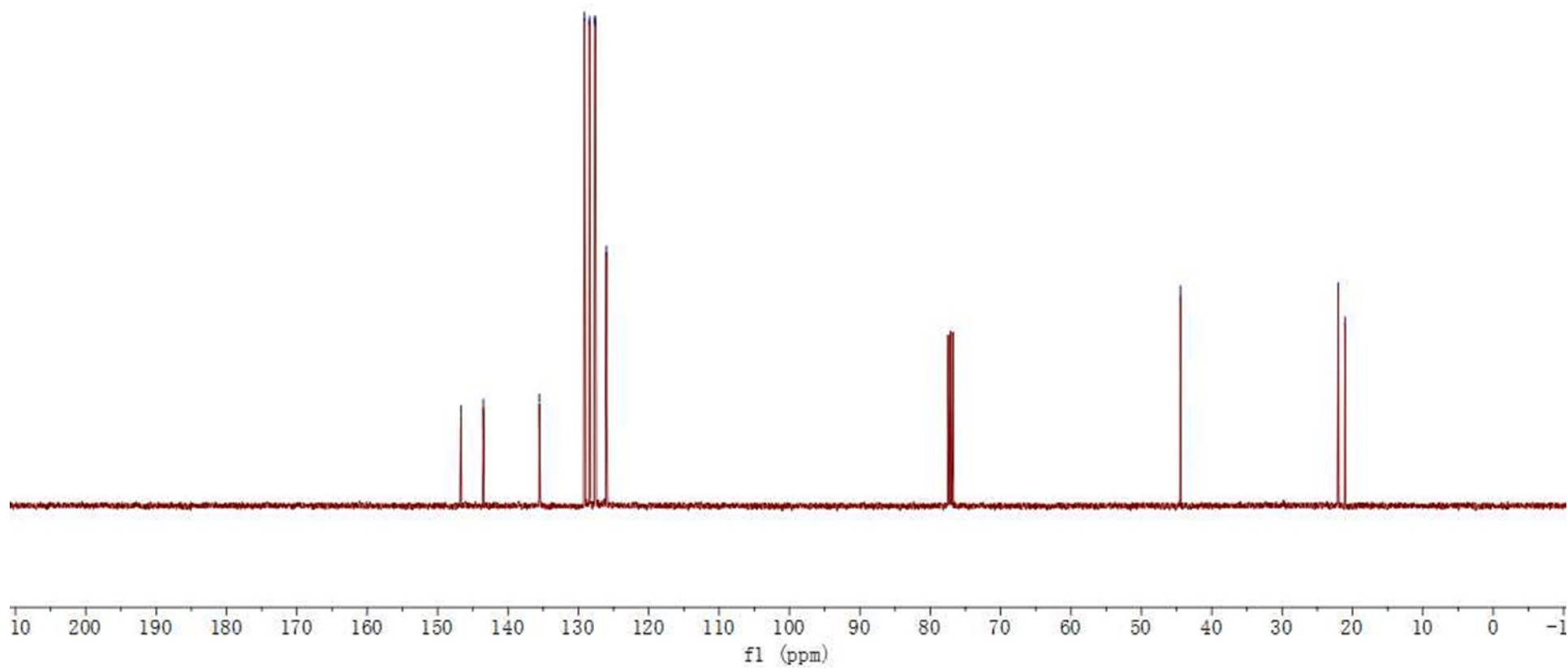


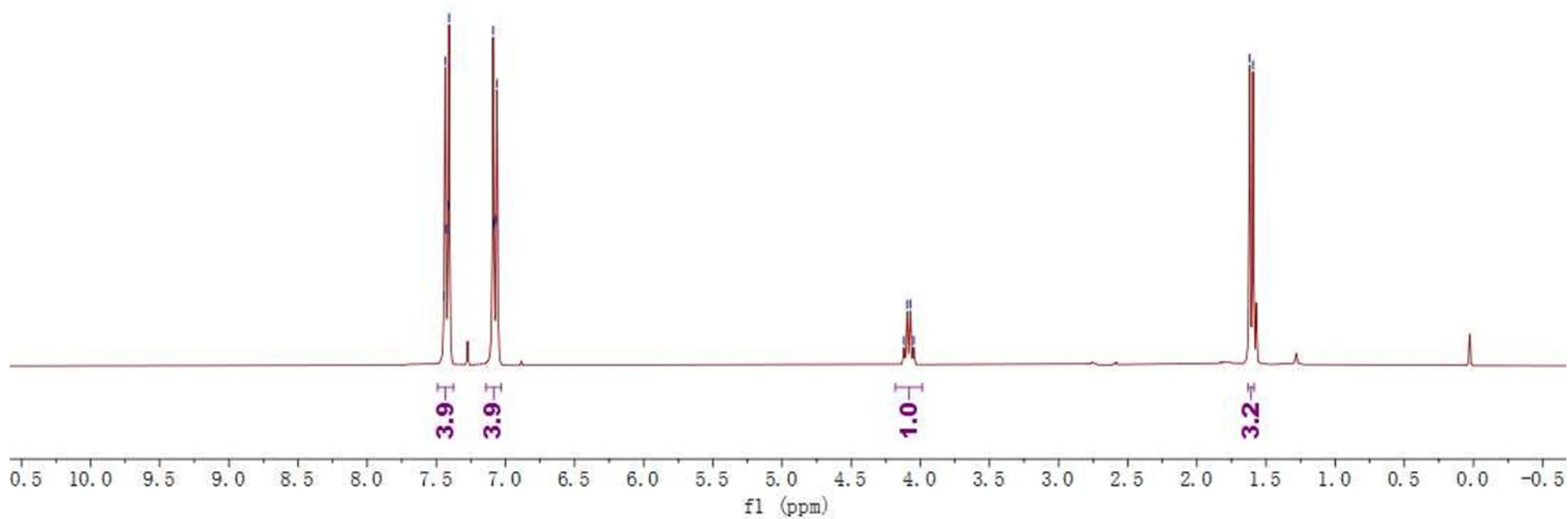
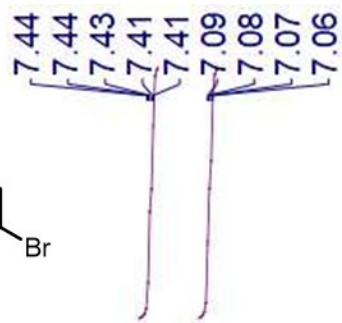
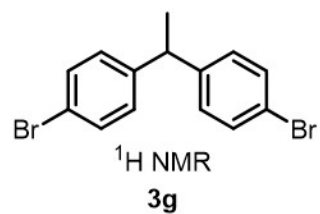
¹³C NMR
3f

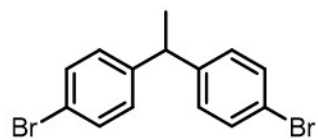
146.7
143.5
135.6
129.1
128.4
127.7
127.6
126.0

-44.5

22.0
21.1







¹³C NMR

3g

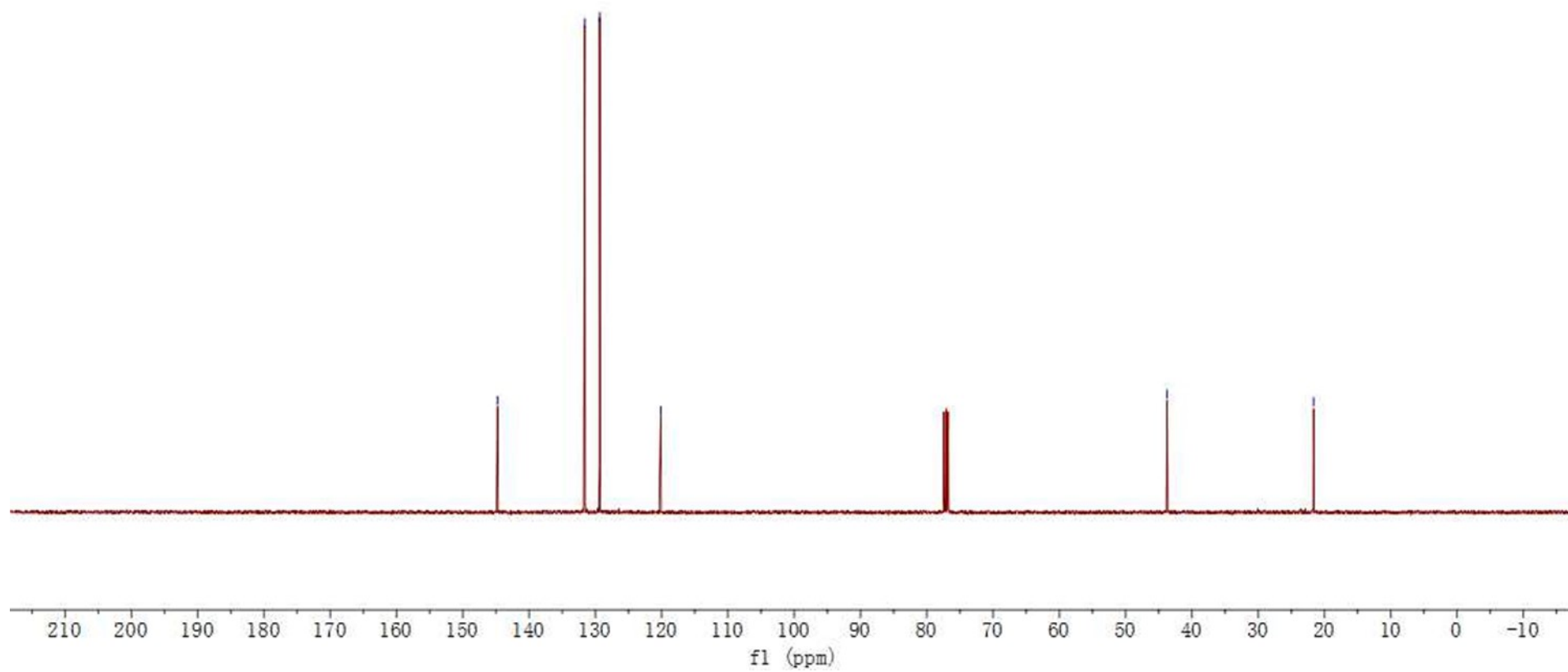
—144.8

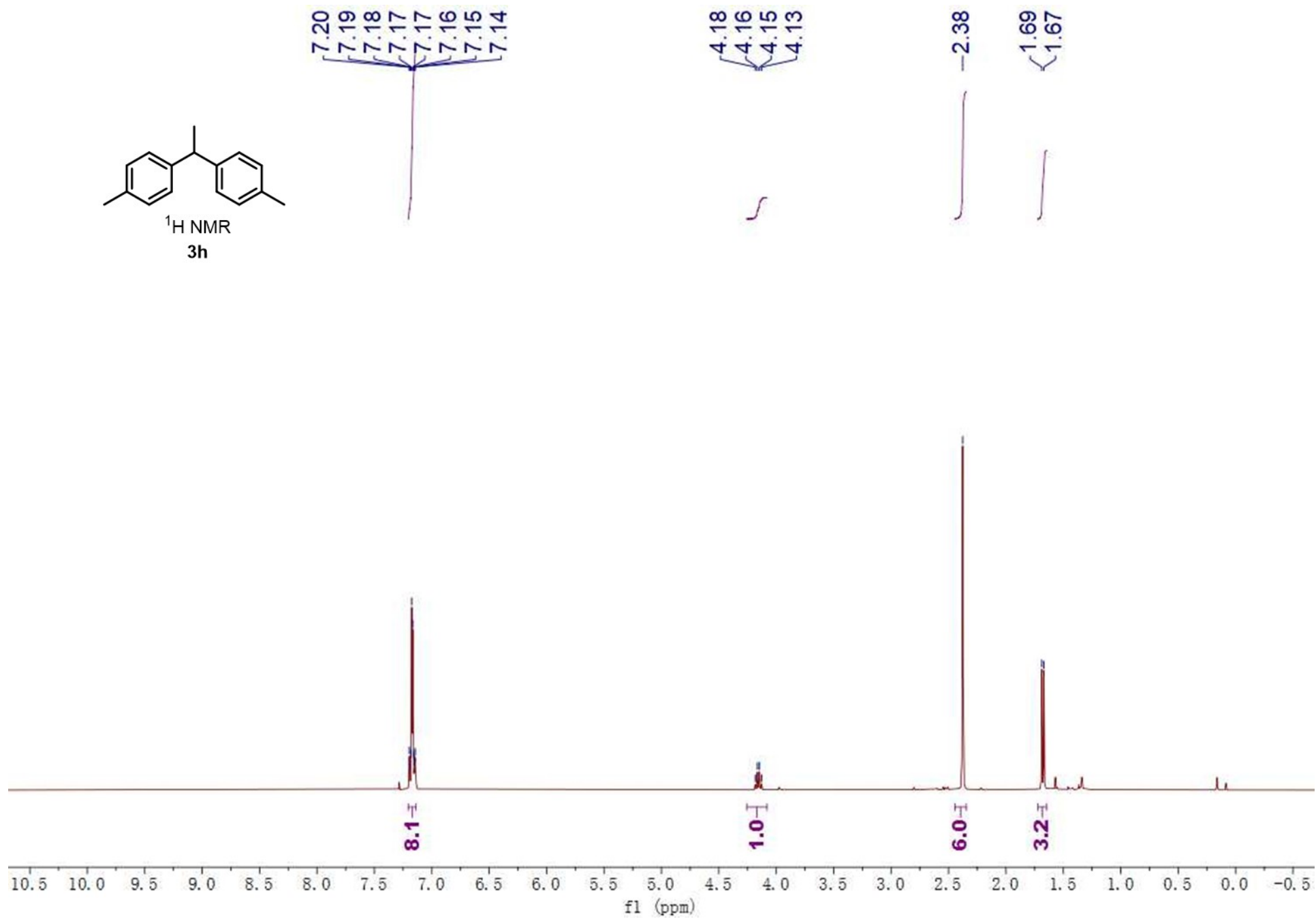
~131.6
~129.3

—120.1

—43.7

—21.7



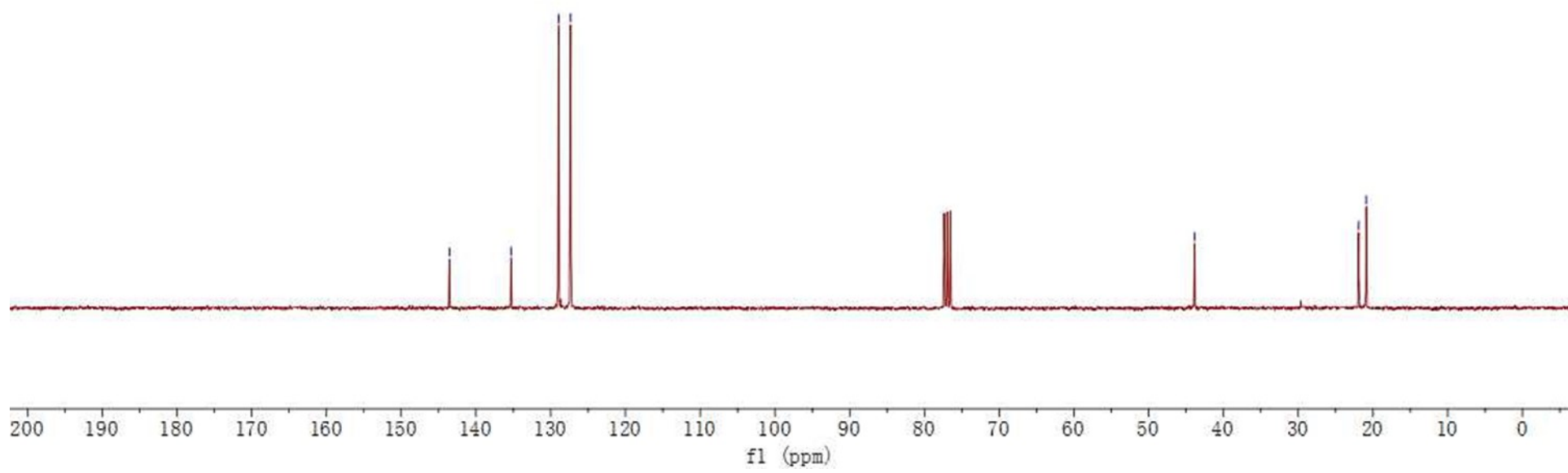


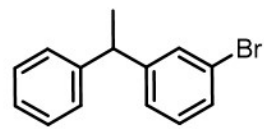


—143.5
—135.3
—128.9
—127.3

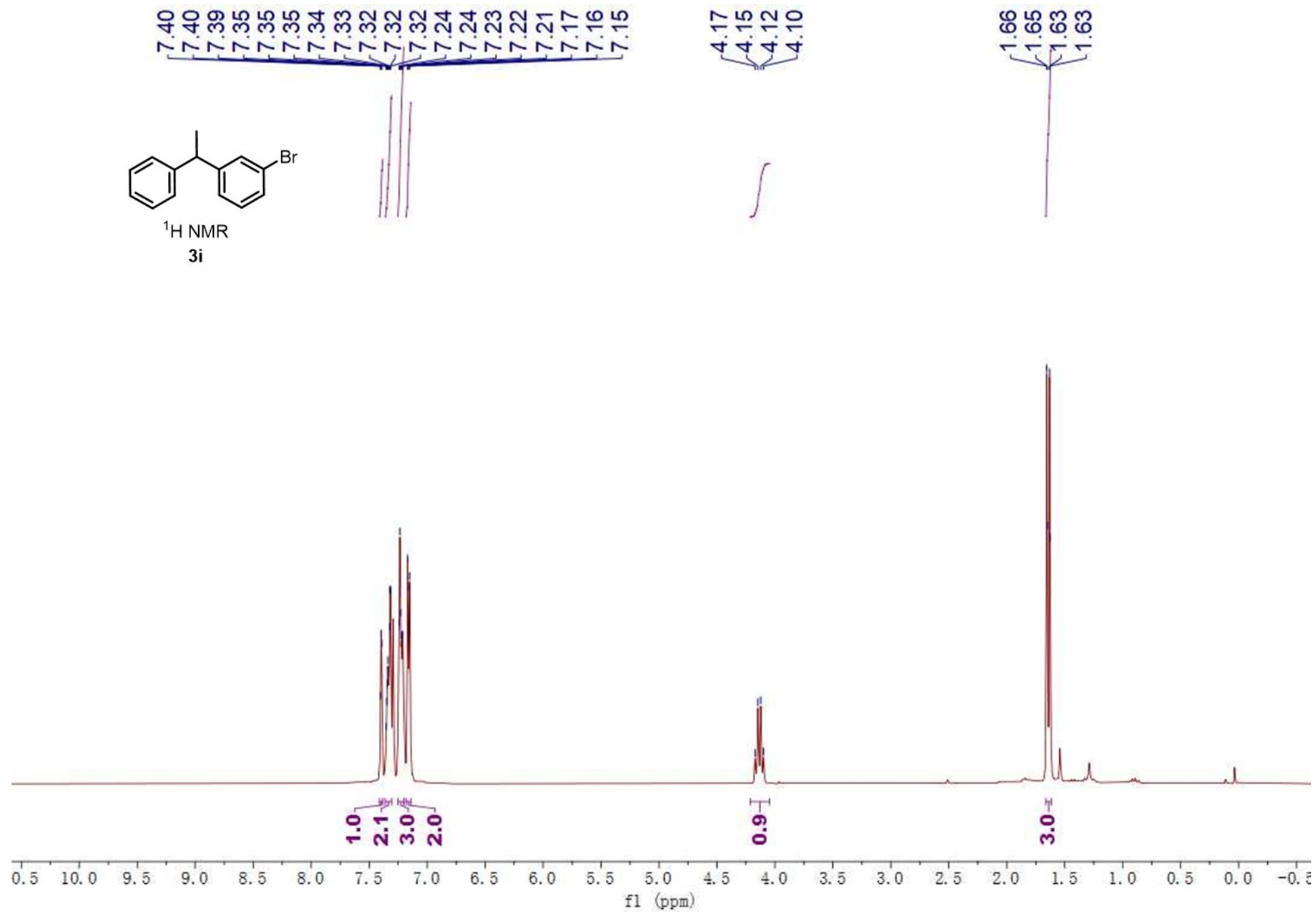
—43.8

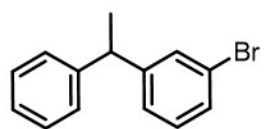
—21.9
—20.9





¹H NMR
3i



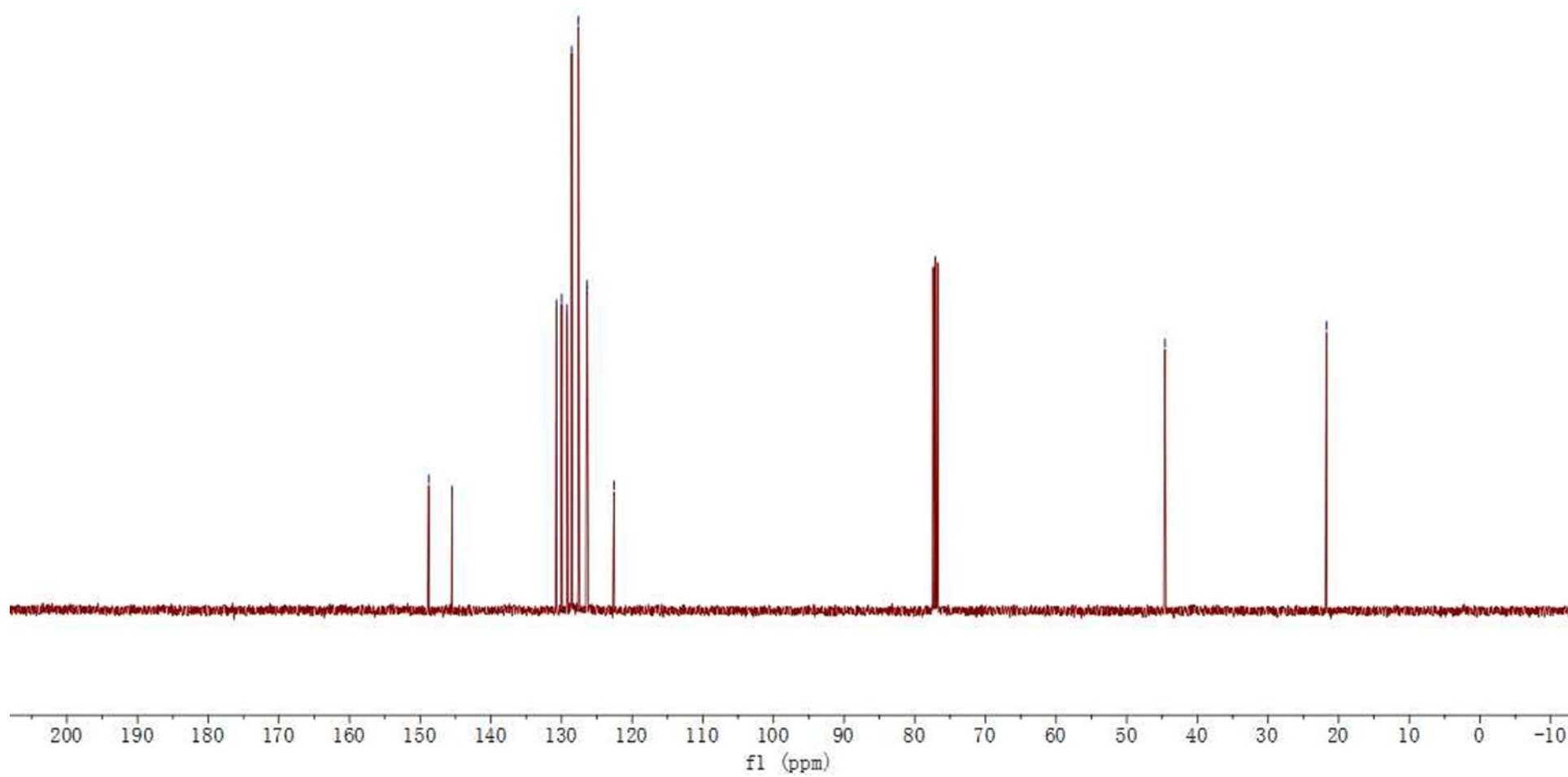


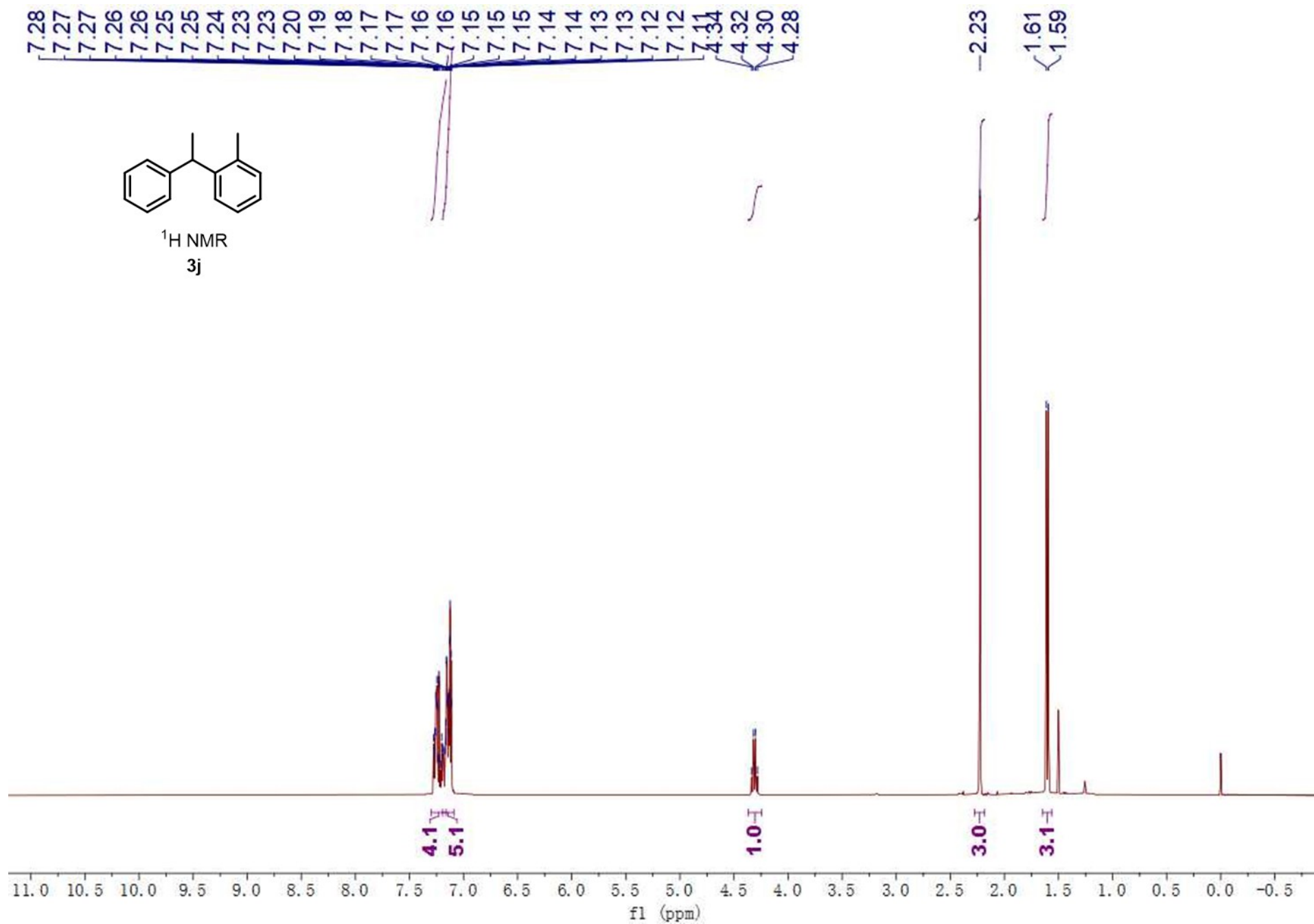
¹³C NMR
3i

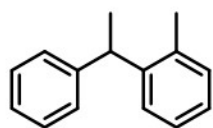
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145.5
130.7
130.0
129.2
128.6
127.6
126.4
126.4
122.6

-44.6

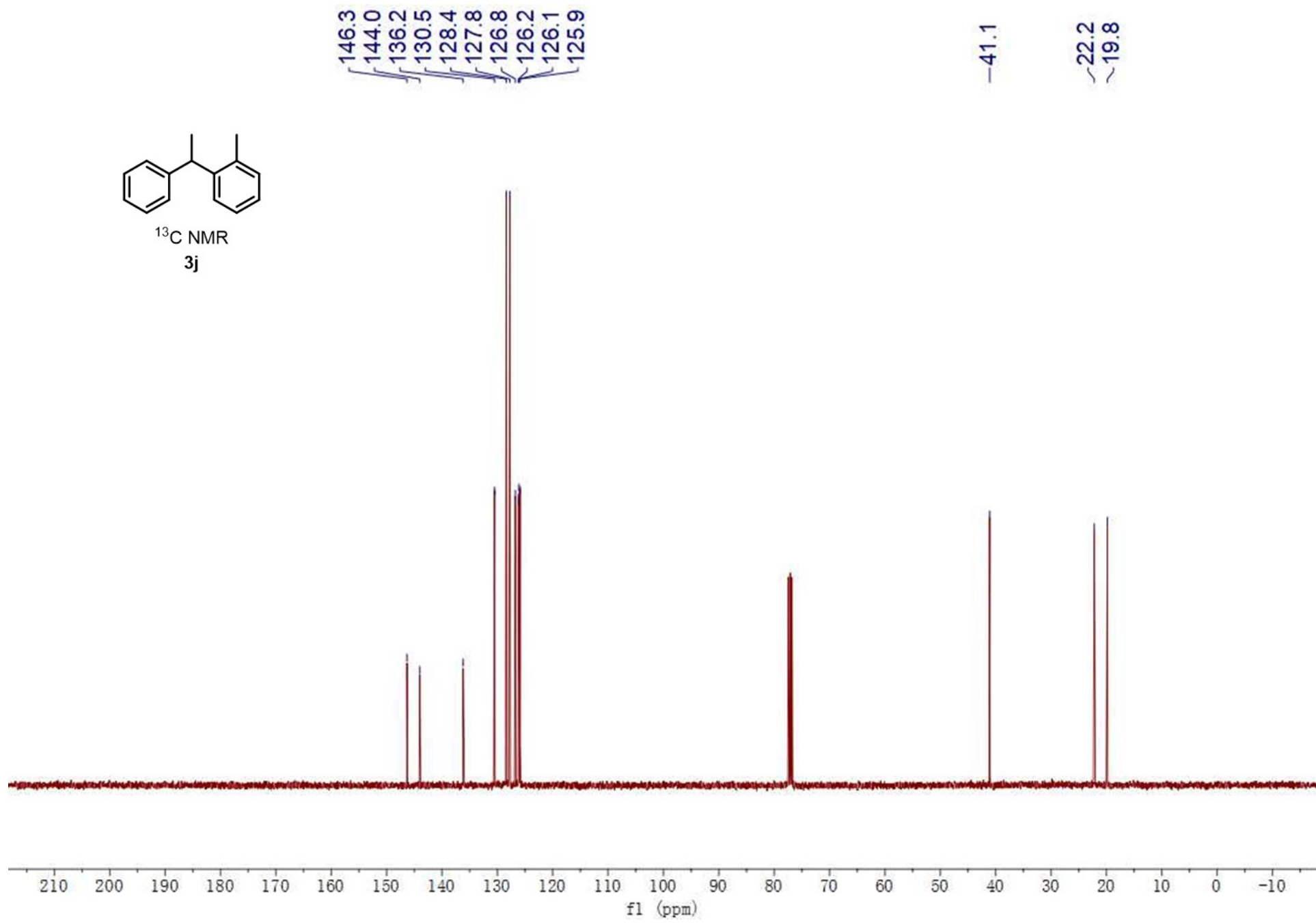
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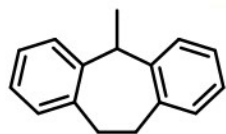






¹³C NMR
3j





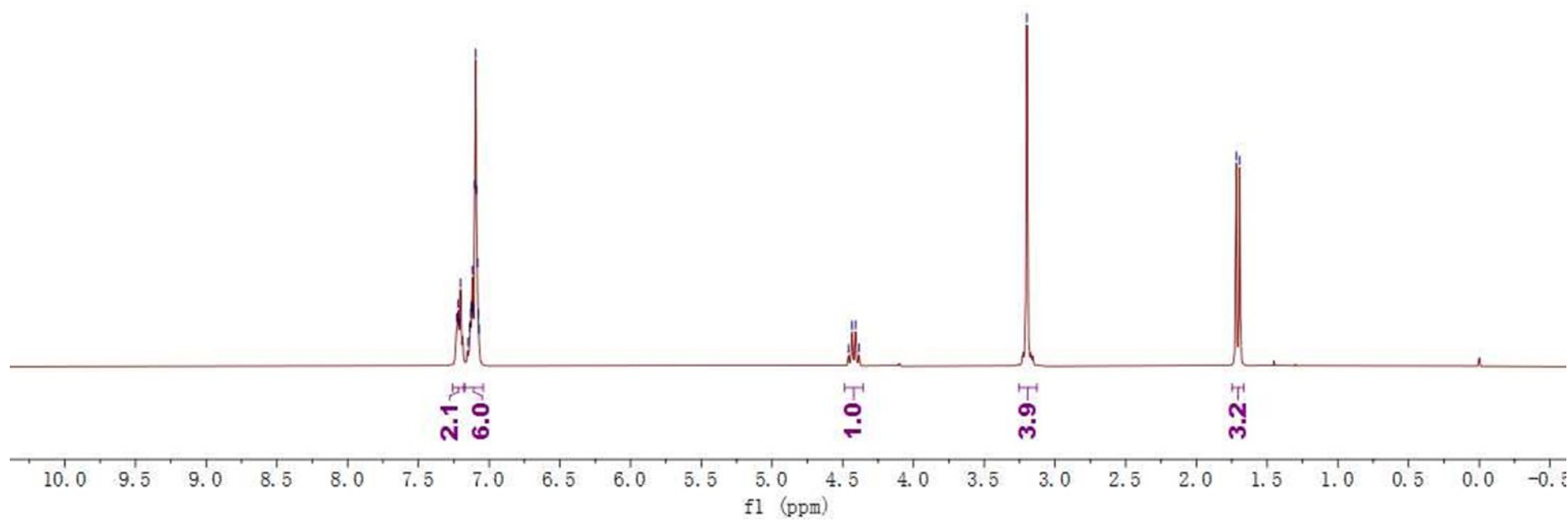
¹H NMR
3k

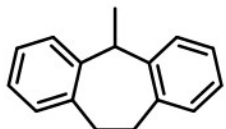
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7.22
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7.15
7.14
7.13
7.12
7.12
7.11
7.10
7.09
7.09
7.08
7.08
7.07

4.46
4.43
4.41
4.39

3.20

1.72
1.69

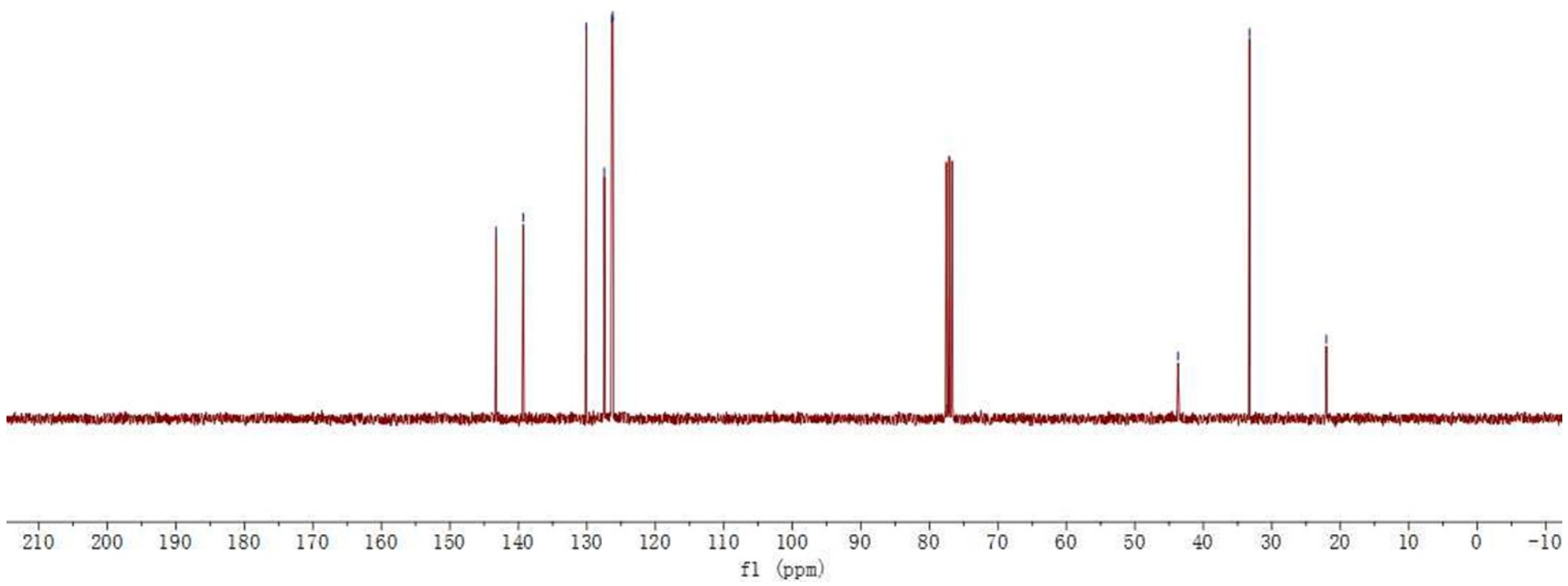


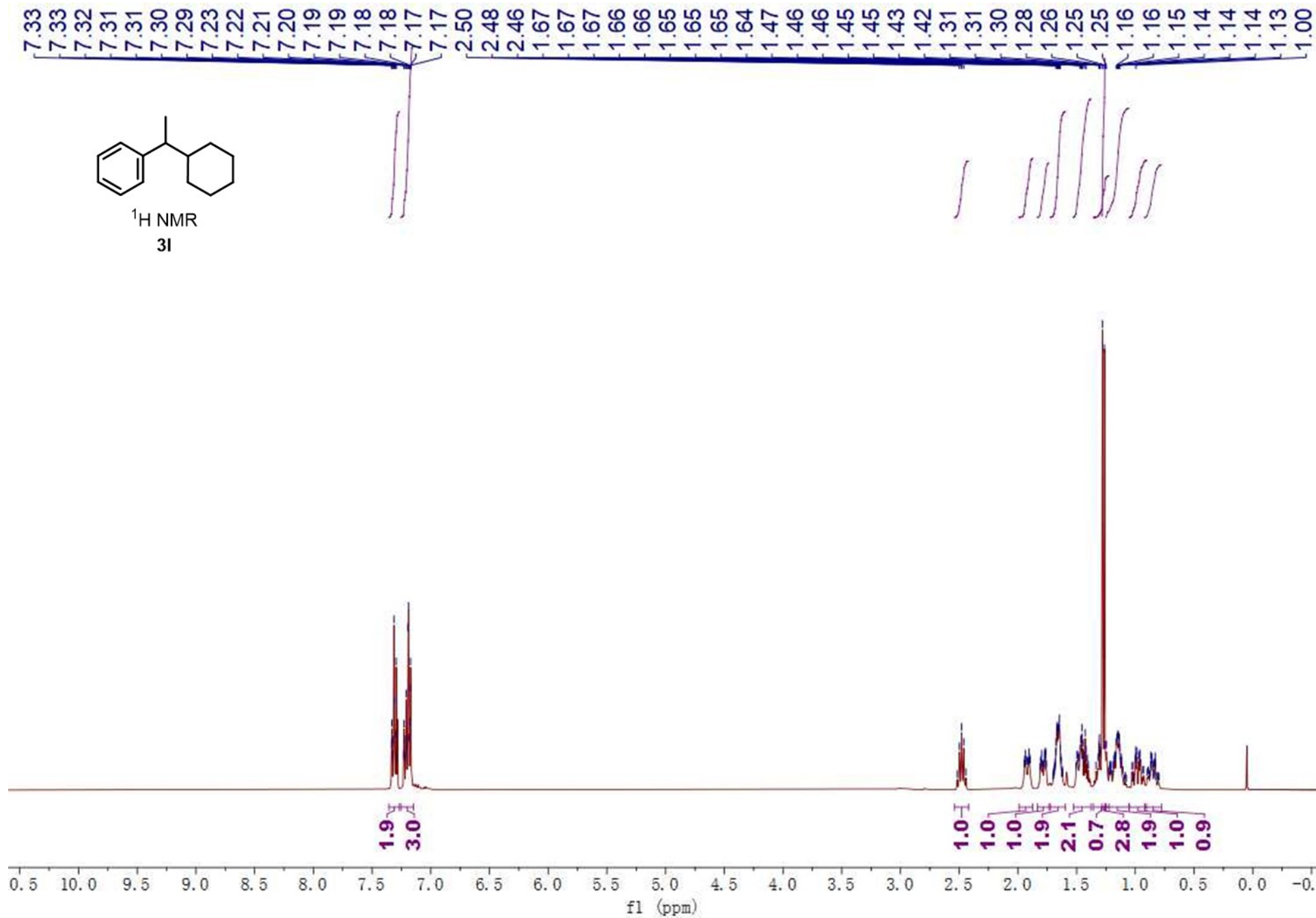


¹³C NMR
3k

143.3
139.3
130.1
127.5
126.4
126.2

43.7
33.3
22.1





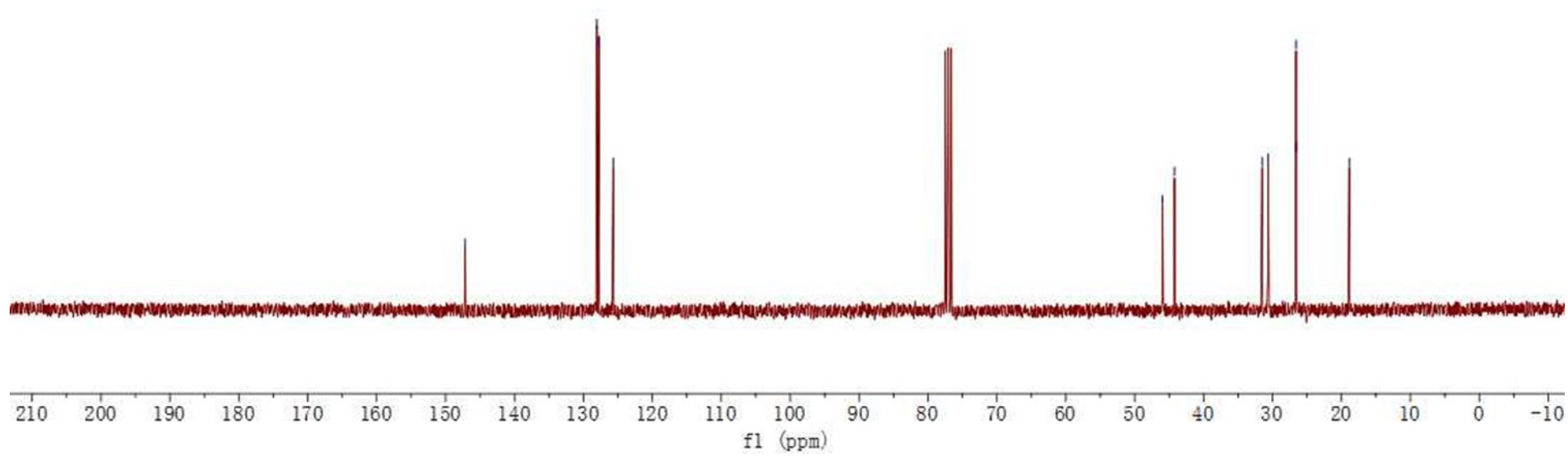


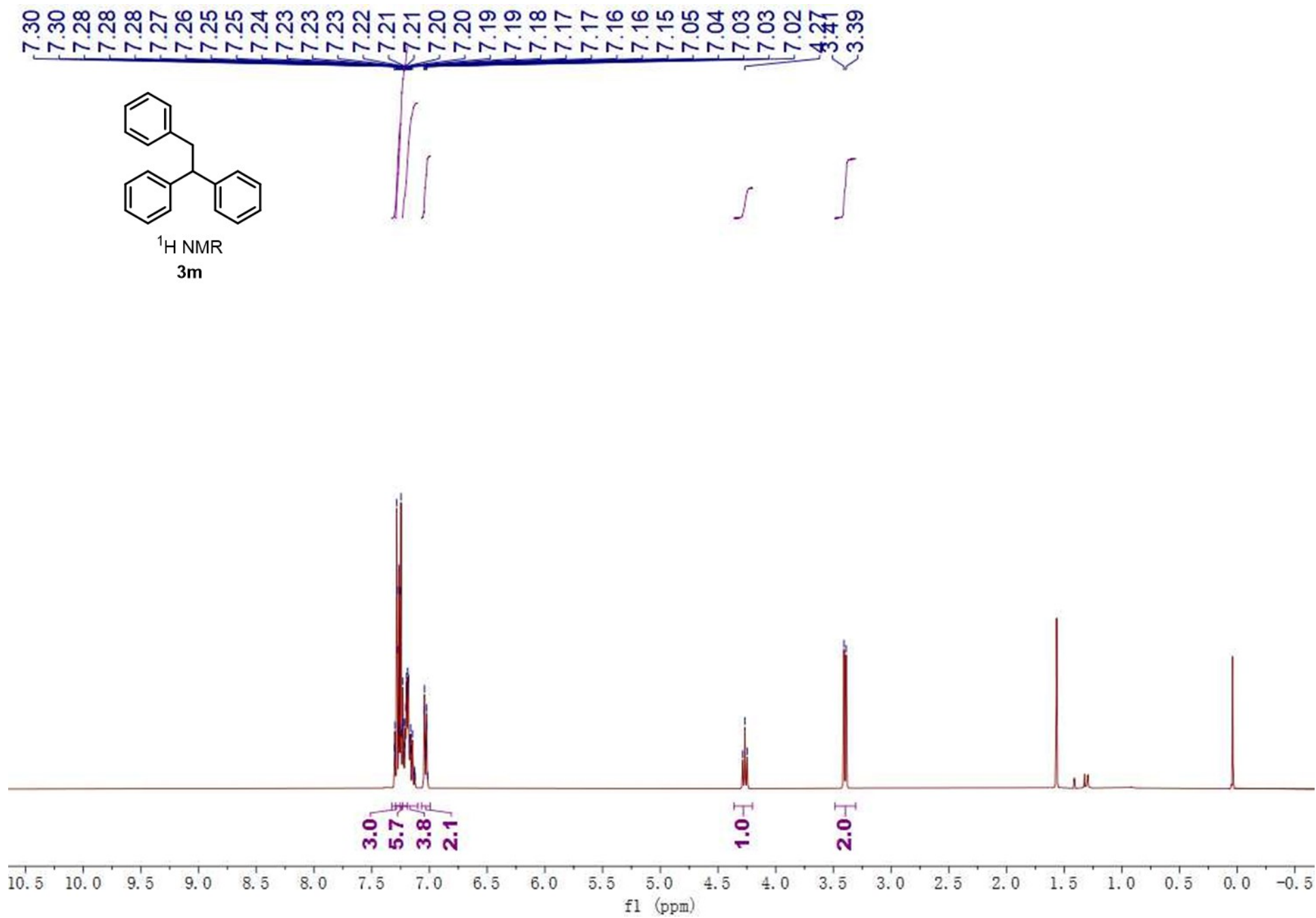
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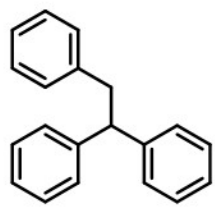
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127.7
125.7

46.0
44.2

31.5
30.7
26.6
26.6
18.9



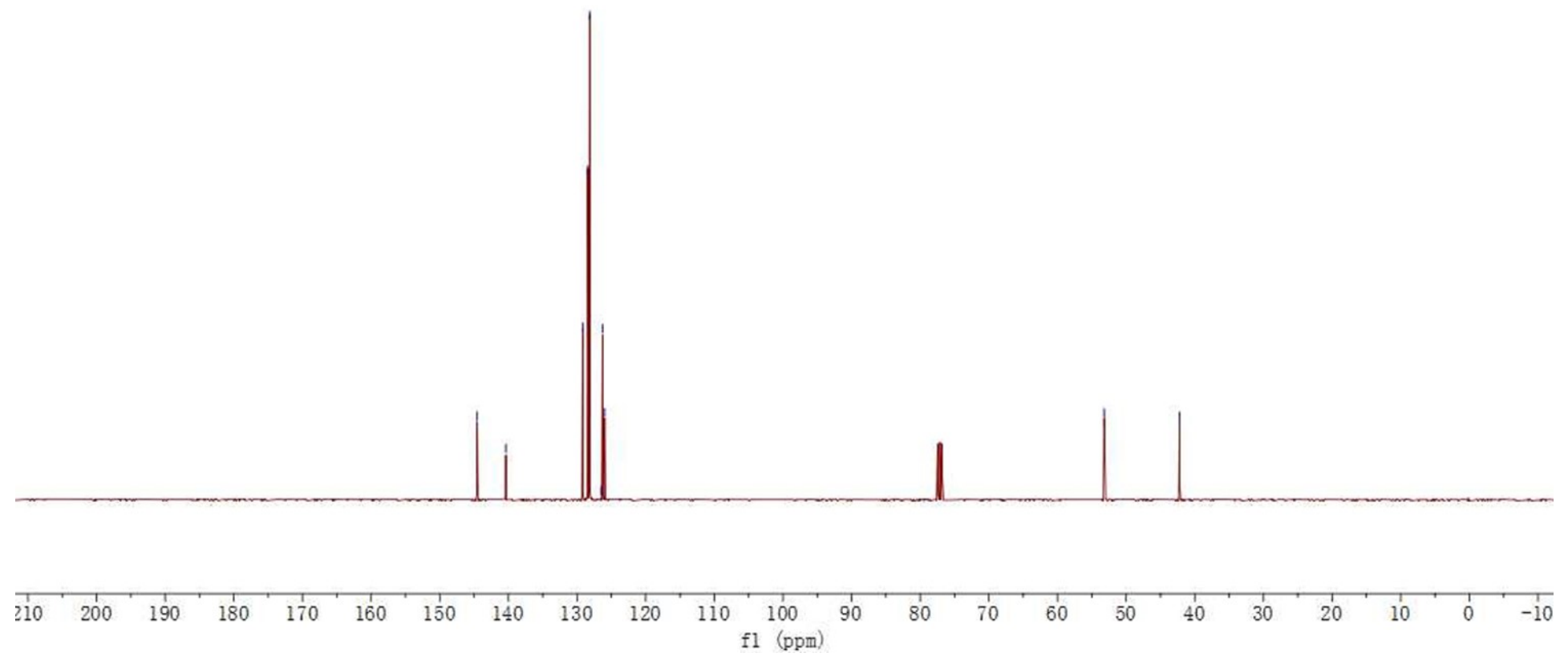




¹³C NMR
3m

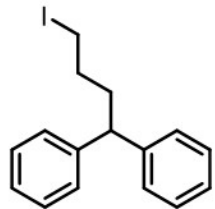
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140.4
129.2
128.4
128.2
126.5
126.3
126.0

53.2
42.2

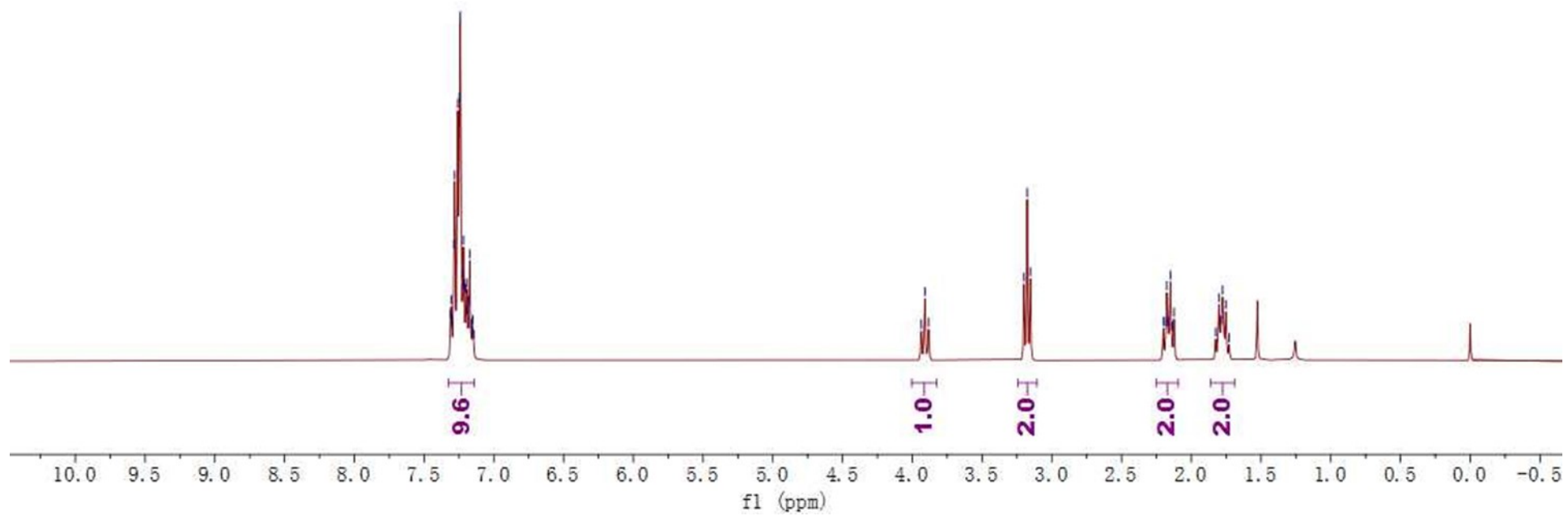


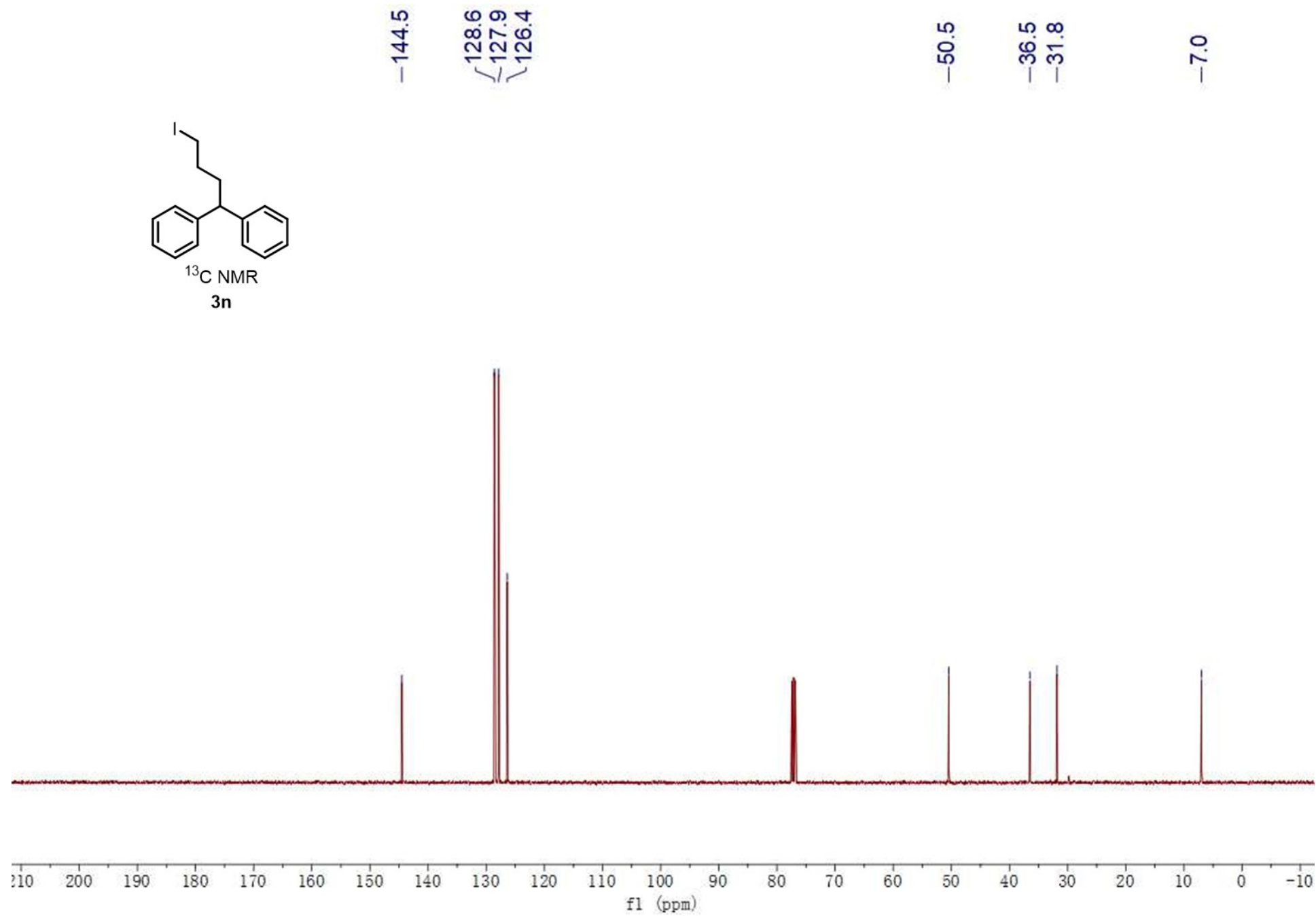
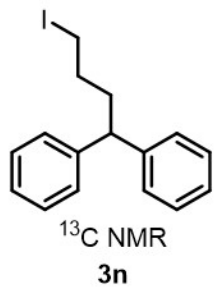
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7.29
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7.24
7.22
7.22
7.21
7.20
7.20
7.19
7.18
7.17
7.16
7.16
7.15
7.14

3.94
3.91
3.88
3.20
3.18
3.15
2.20
2.18
2.18
2.17
2.15
2.14
2.12
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1.80
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1.78
1.75
1.73

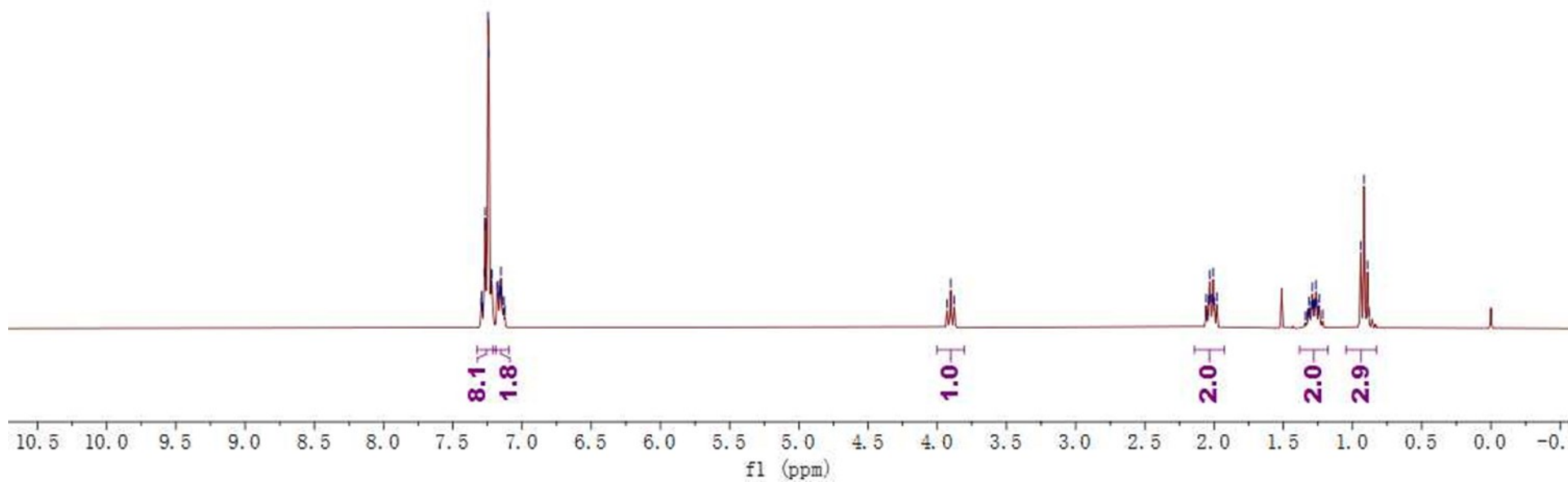
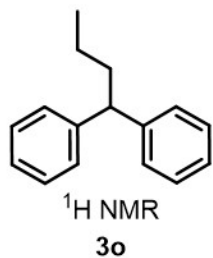


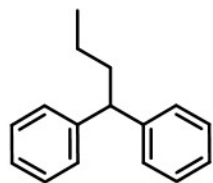
¹H NMR
3n





7.30
7.29
7.29
7.27
7.27
7.26
7.25
7.24
7.22
7.22
7.22
7.18
7.17
7.16
7.16
7.15
7.15
7.14
7.13
7.12
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3.90
3.88
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1.33
1.32
1.32
1.31
1.30
1.29
1.28
1.27
1.26
1.26
1.25
1.24
1.22
0.94
0.92
0.89
0.88





¹³C NMR

3o

-145.4

128.4

128.0

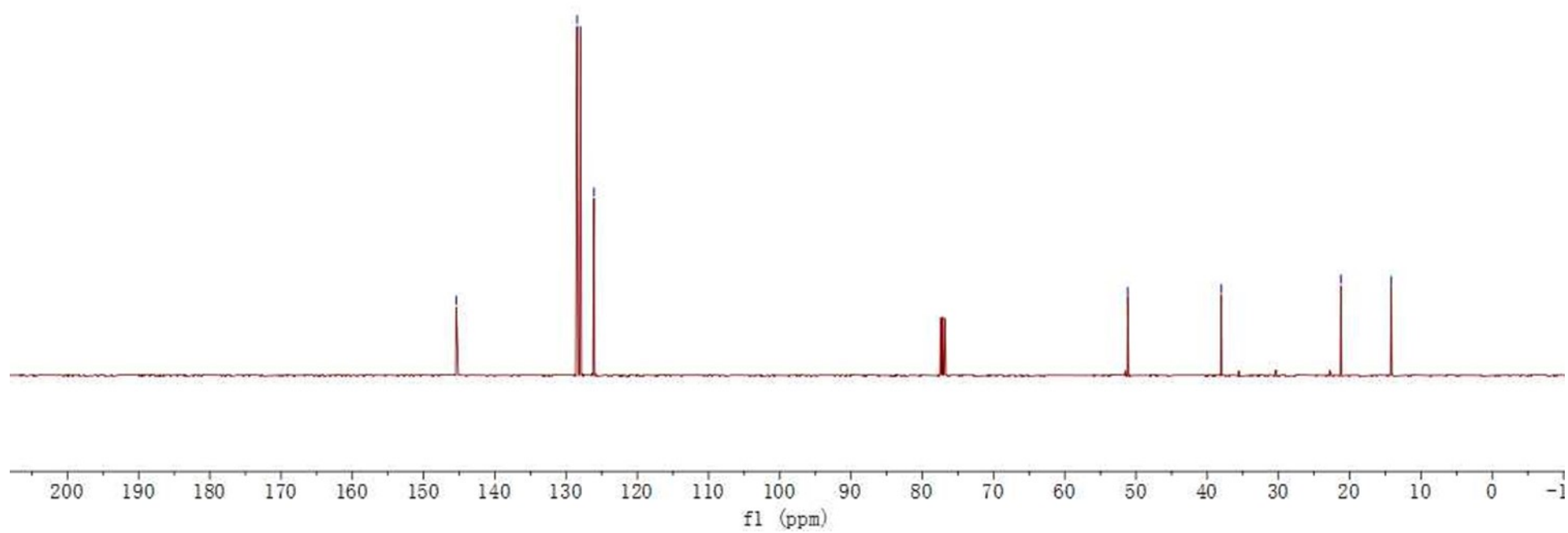
126.1

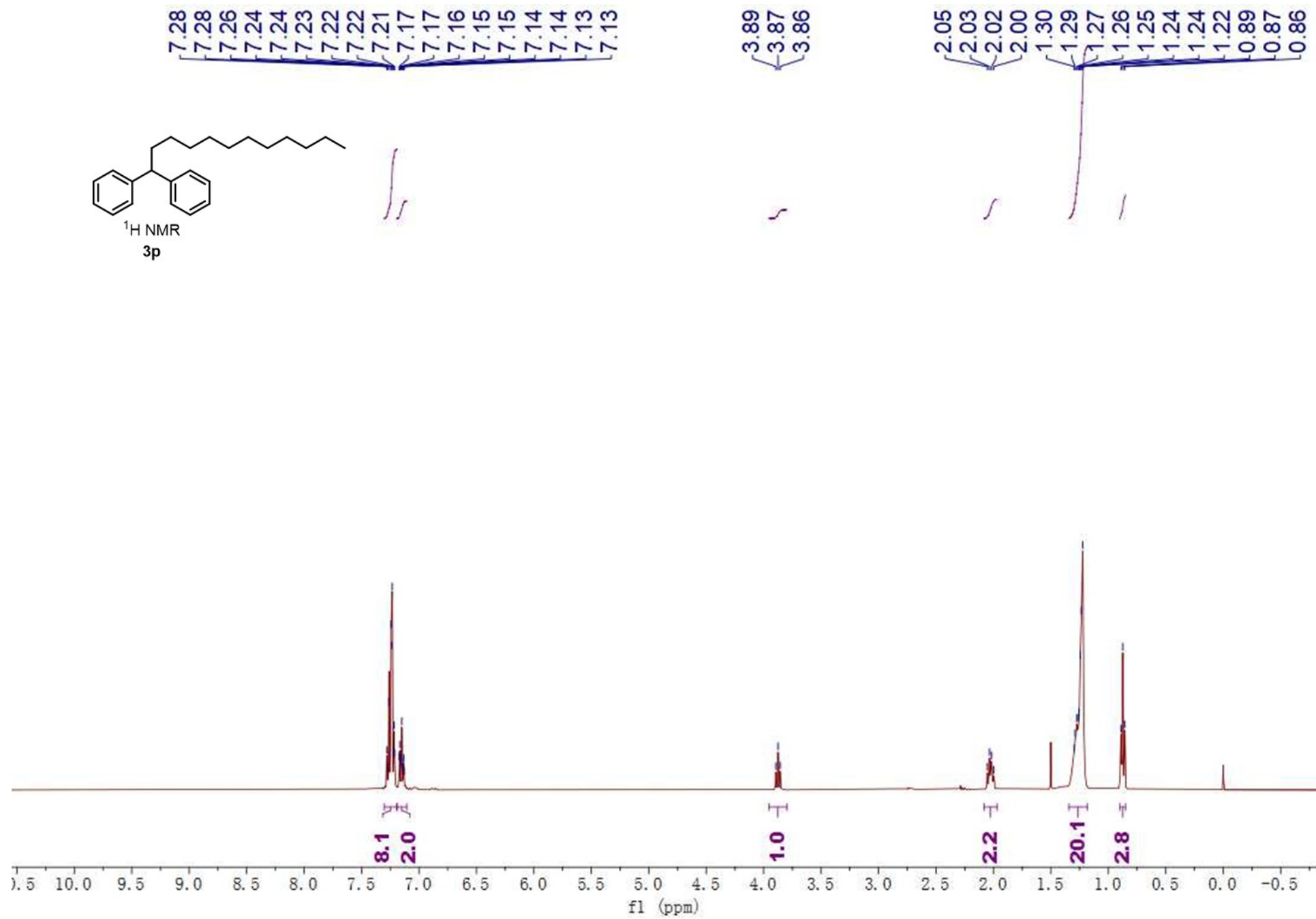
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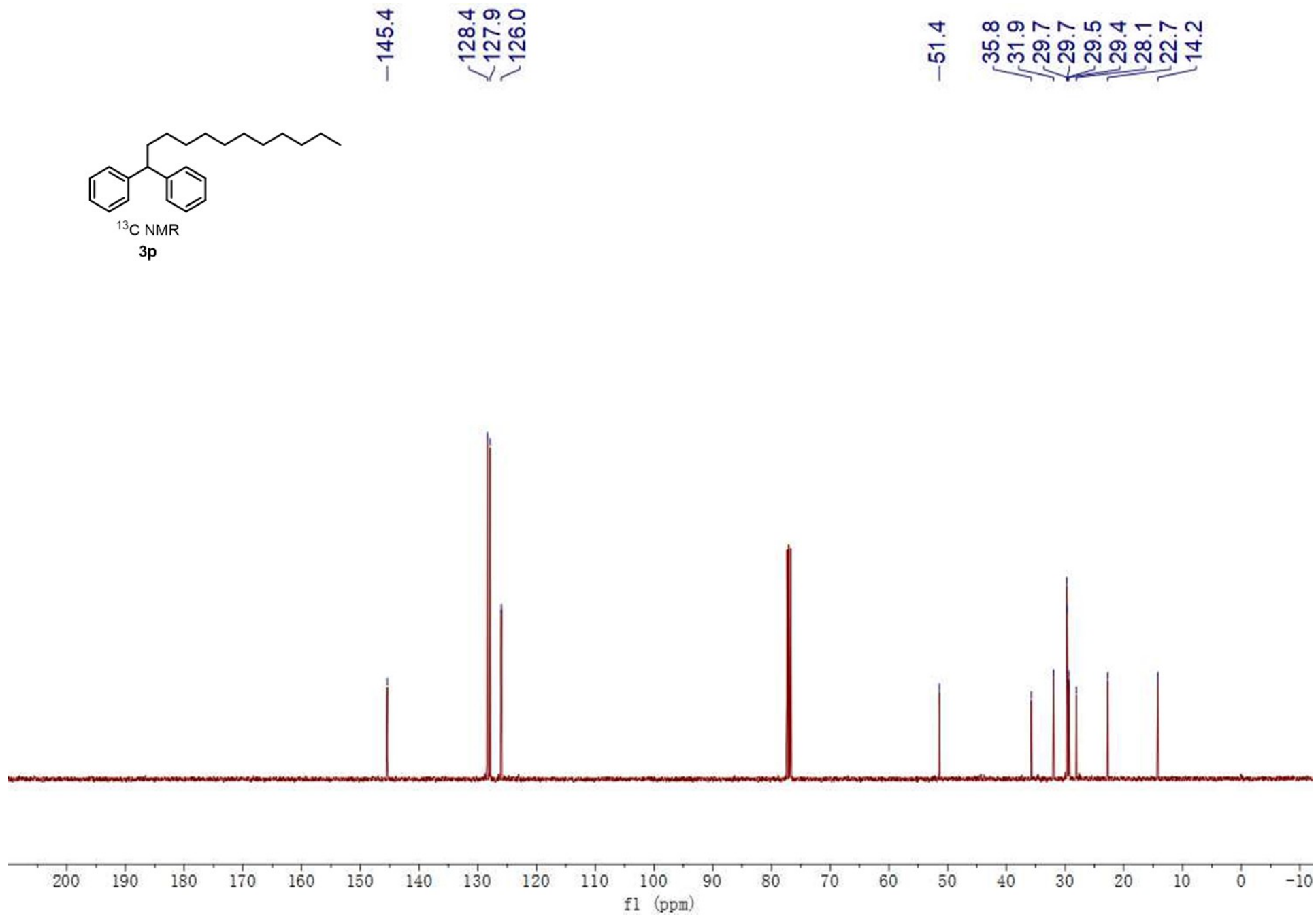
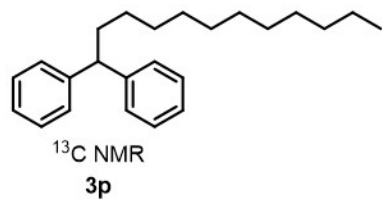
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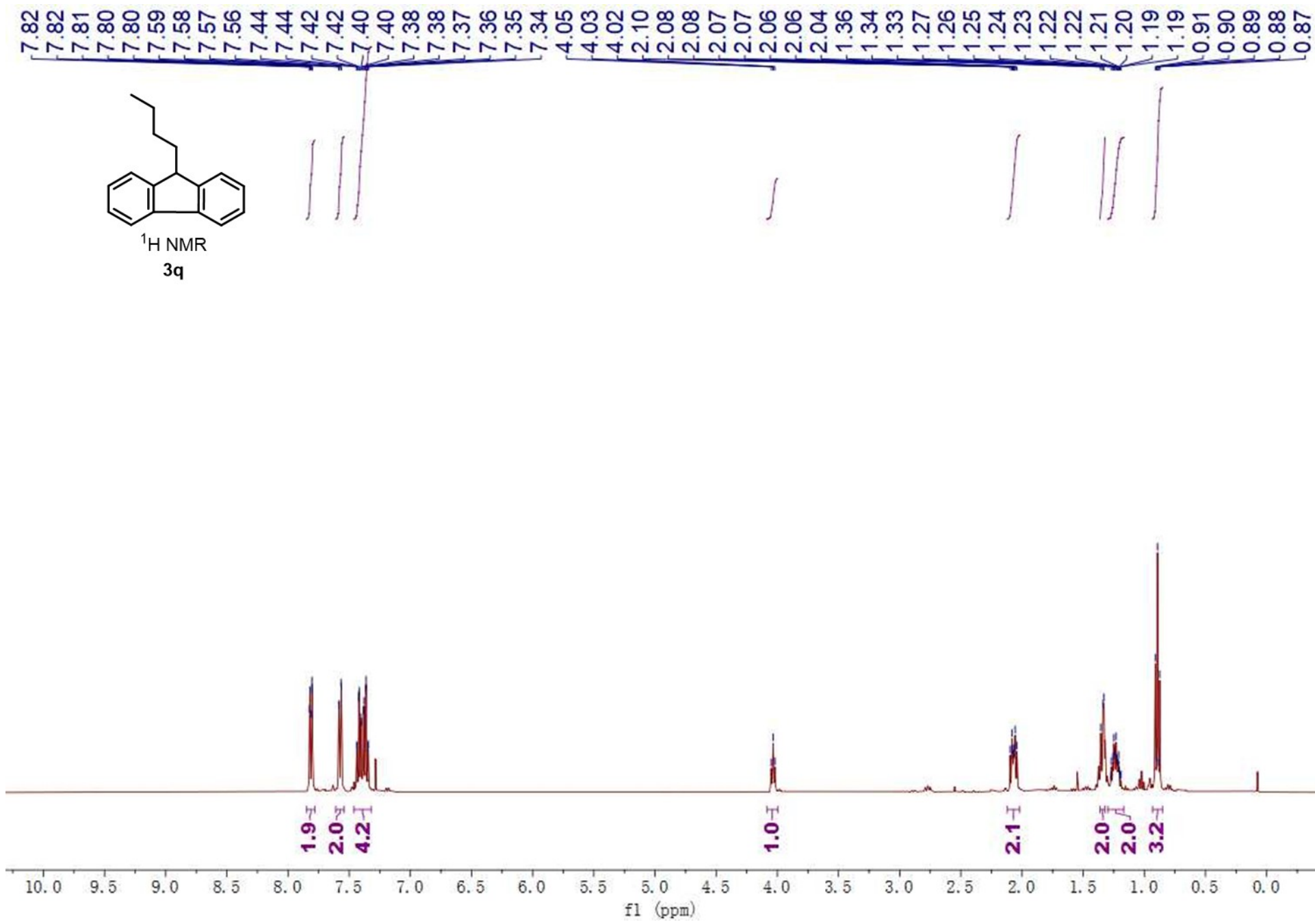
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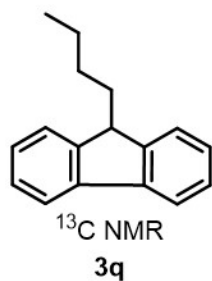
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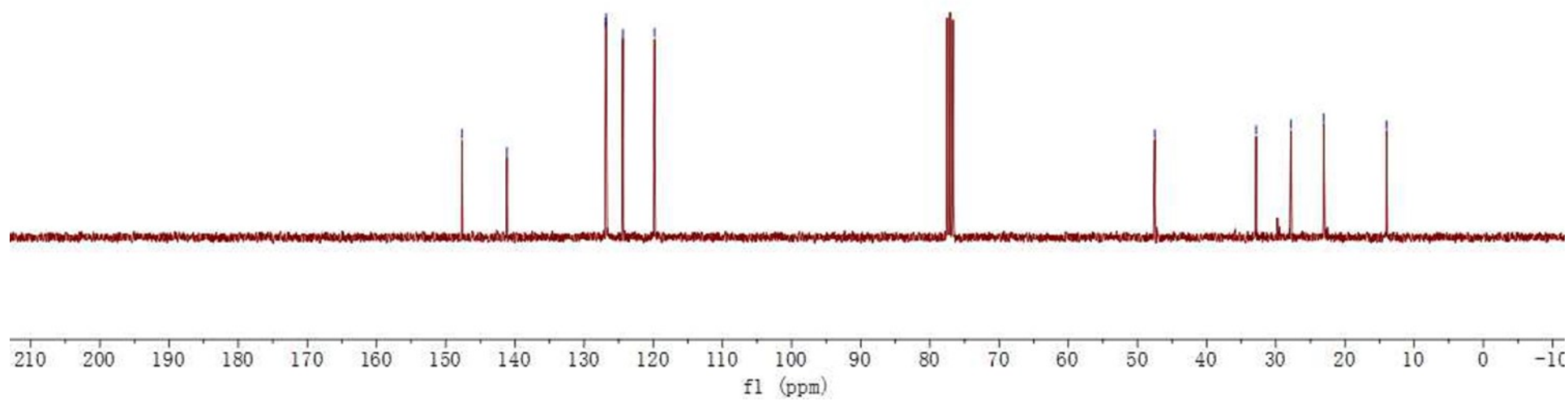




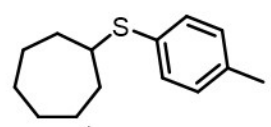


-147.7
-141.2
126.9
126.8
124.4
119.8

-47.5
-32.9
-27.8
-23.1
-14.0



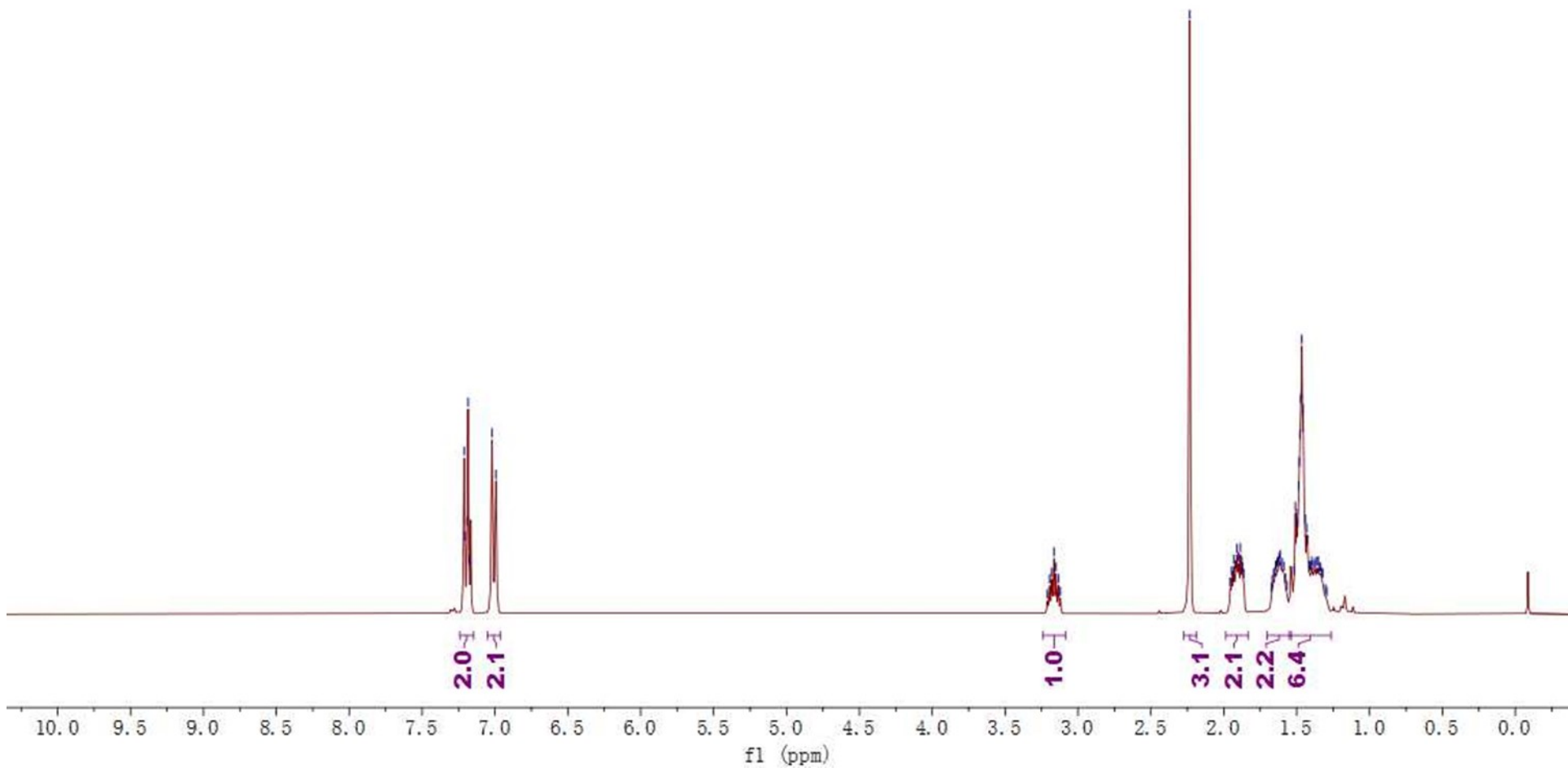
7.21
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1.88
1.87
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1.47
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1.45
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1.34
1.33

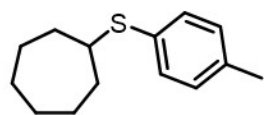


¹H NMR
3r

|||

~





^{13}C NMR
3r

~136.6
~132.0
~129.5

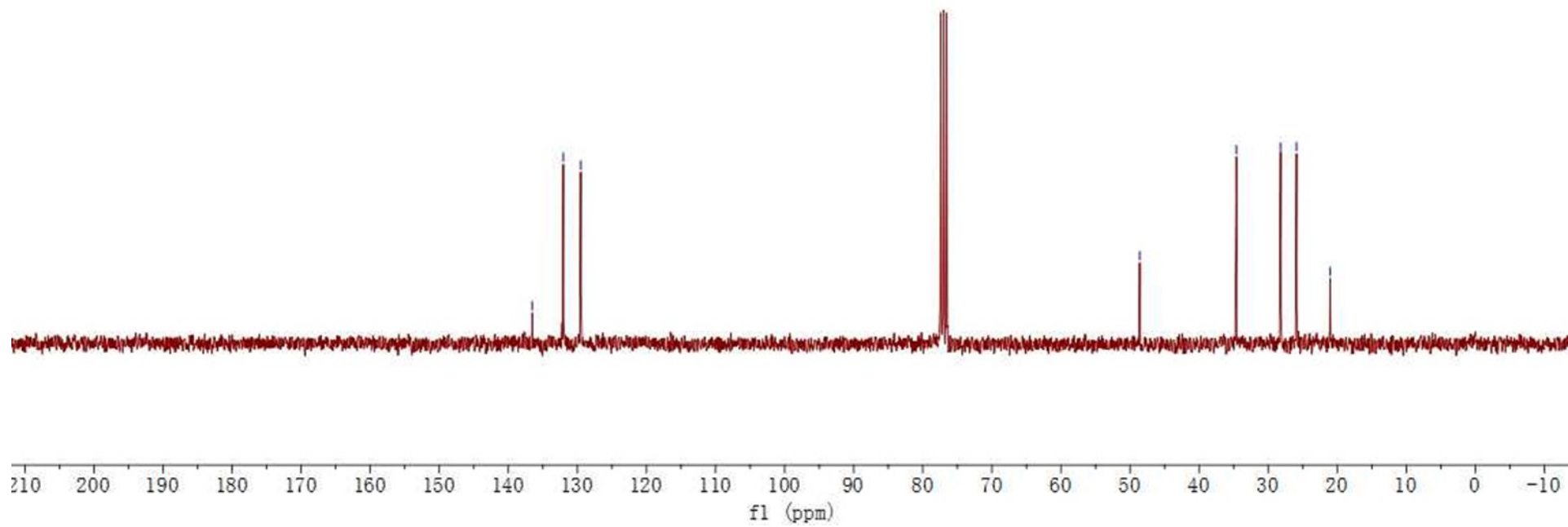
-48.6

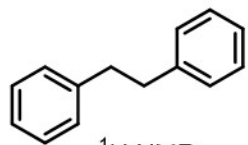
-34.6

~28.2

-25.9

-21.0

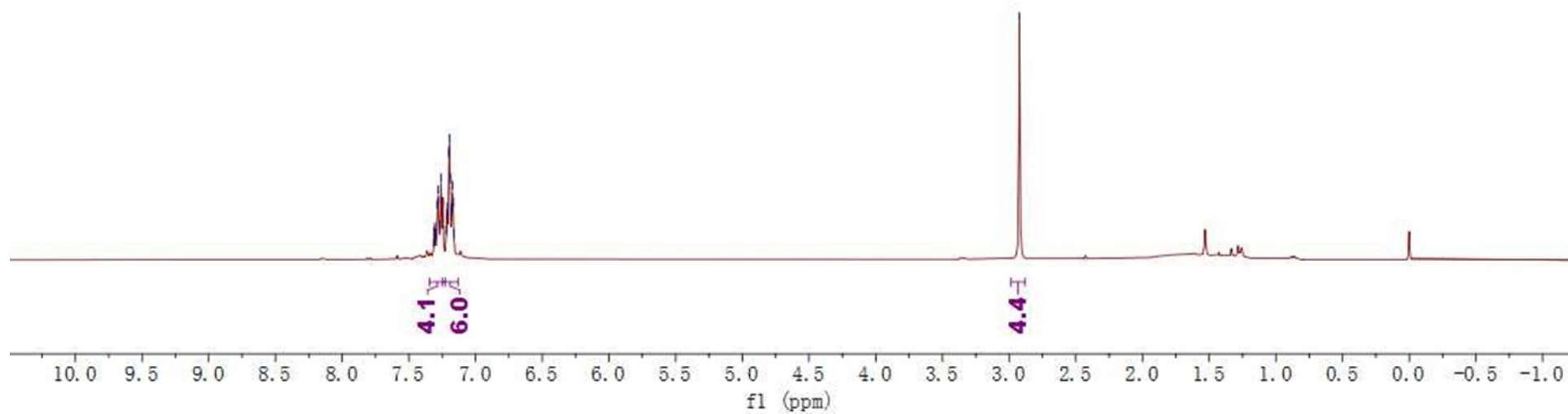


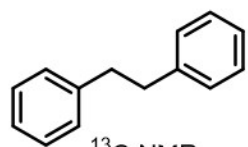


¹H NMR
3s

7.32
7.31
7.31
7.30
7.29
7.28
7.28
7.27
7.26
7.22
7.21
7.21
7.20
7.20
7.19
7.18
7.17
7.17
7.16

-2.92





¹³C NMR
3s

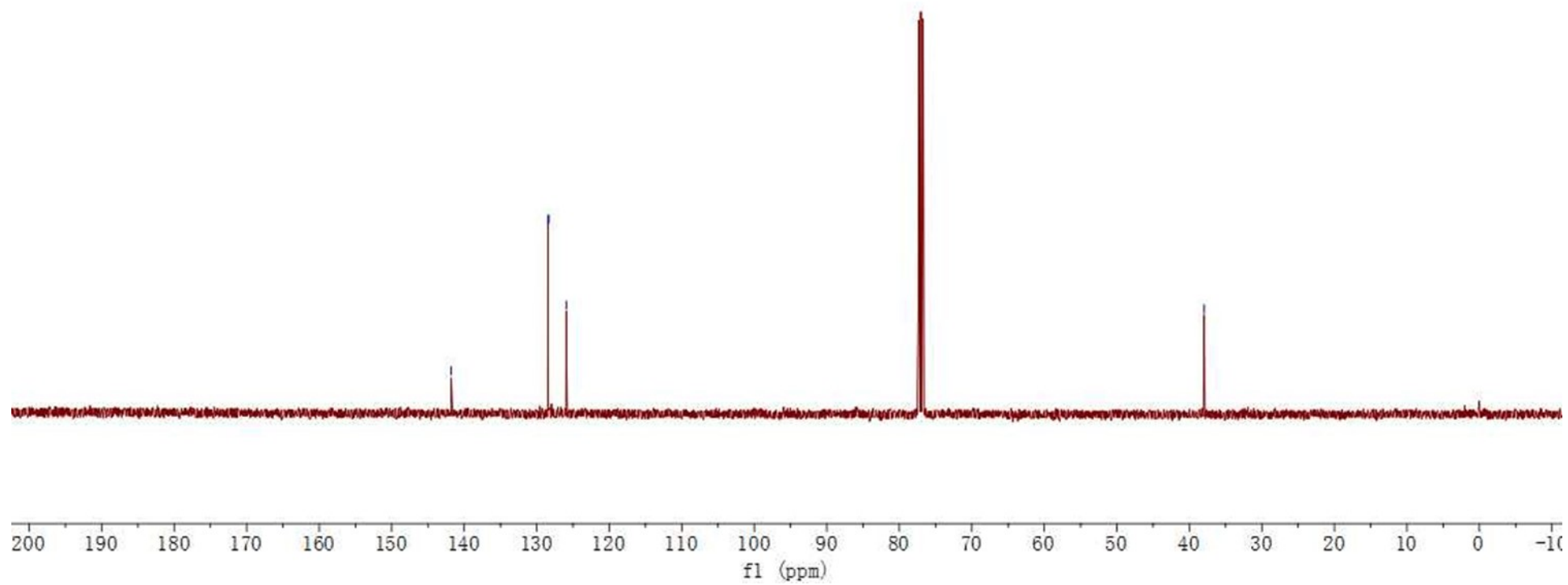
—141.8

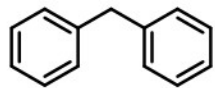
128.5

128.3

125.9

—38.0

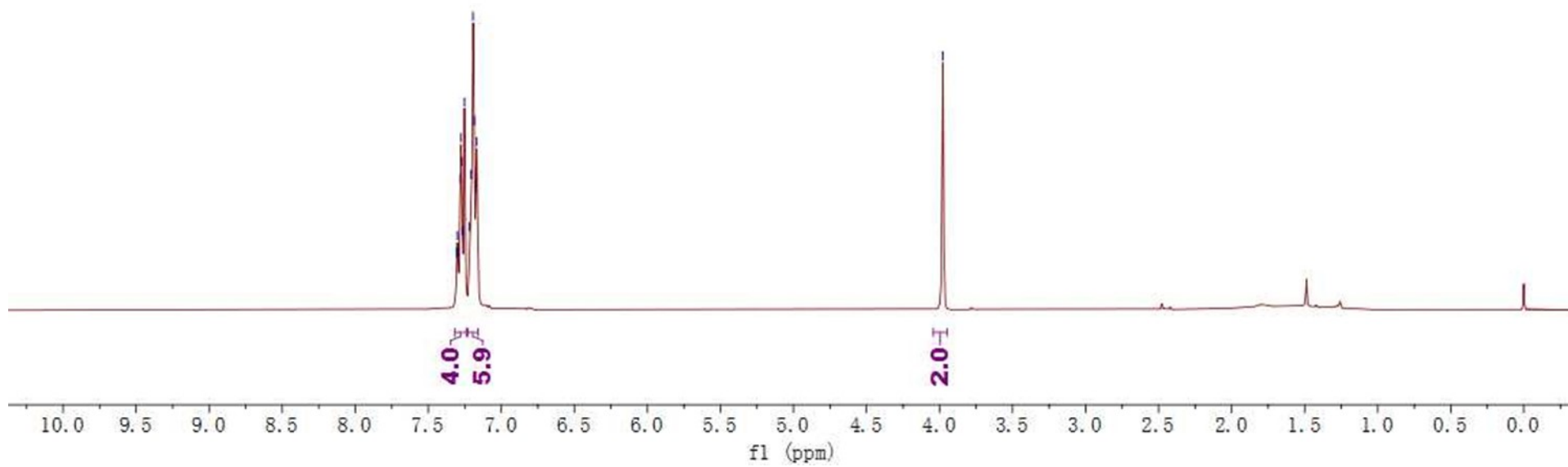


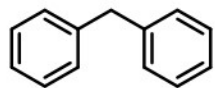


¹H NMR
6a

7.31
7.30
7.30
7.28
7.28
7.27
7.27
7.25
7.22
7.21
7.19
7.18
7.17
7.17

—3.98





¹³C NMR
6a

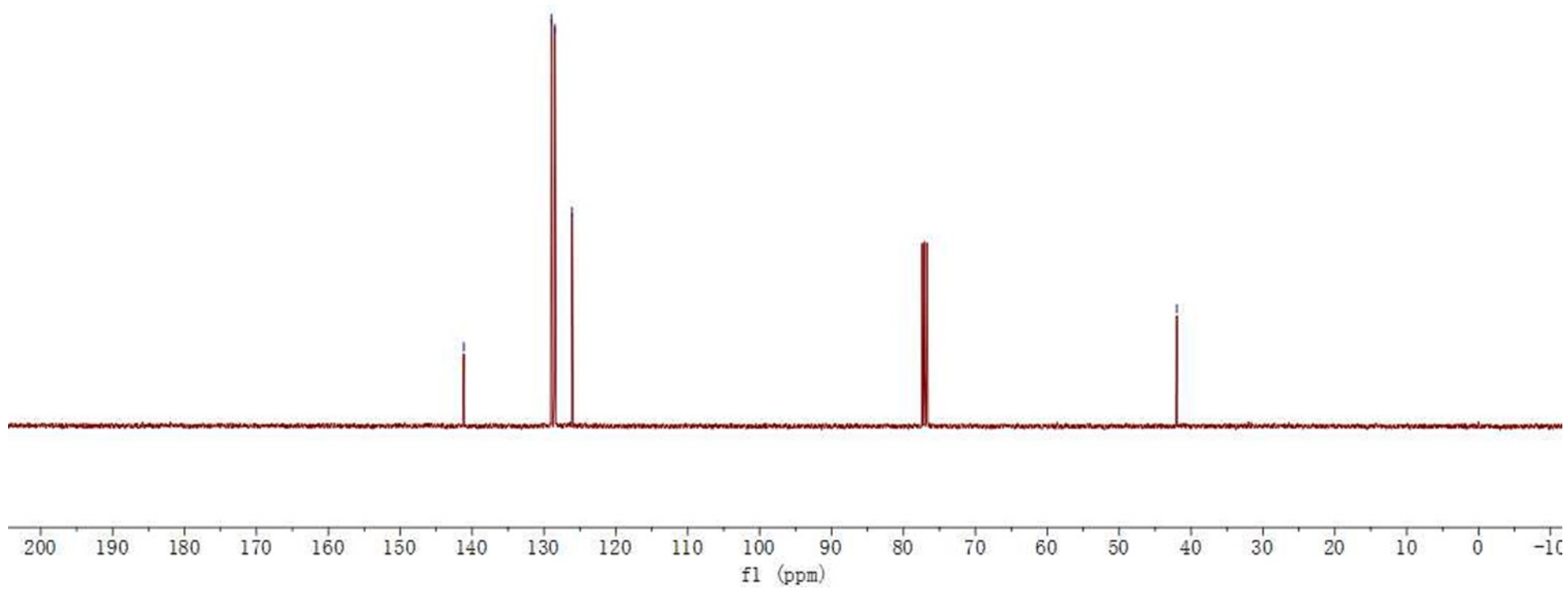
-141.2

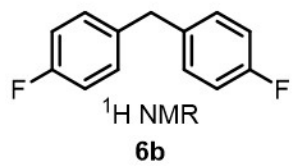
129.0

128.5

126.1

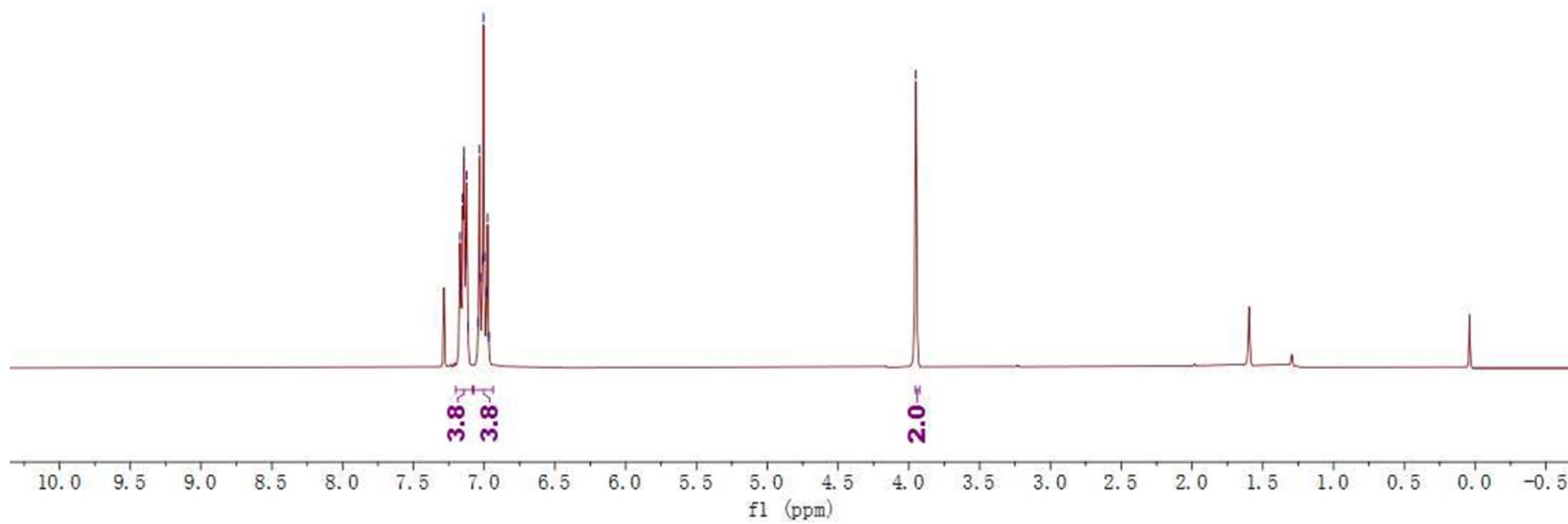
-42.0

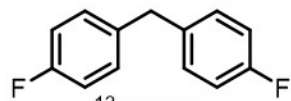




7.17
7.16
7.15
7.14
7.13
7.12
7.11
7.04
7.03
7.03
7.01
7.01
7.00
6.98
6.98
6.97

3.95





¹³C NMR

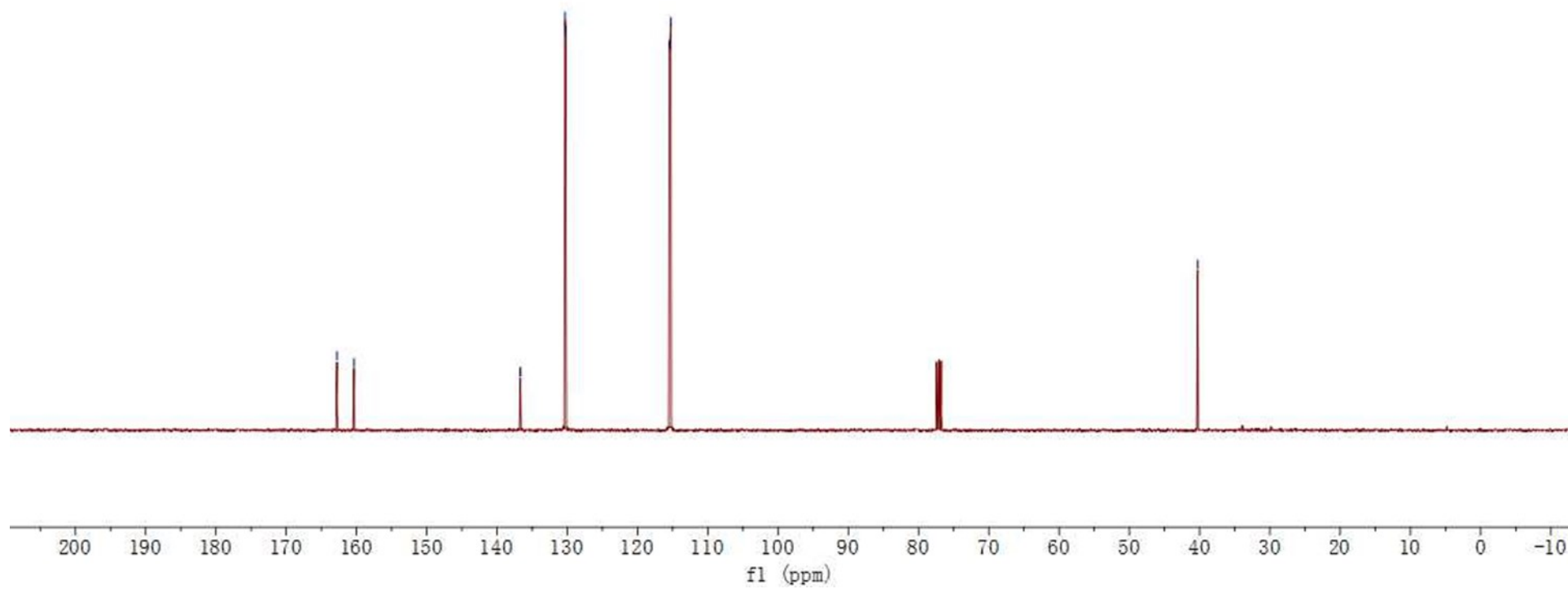
6b

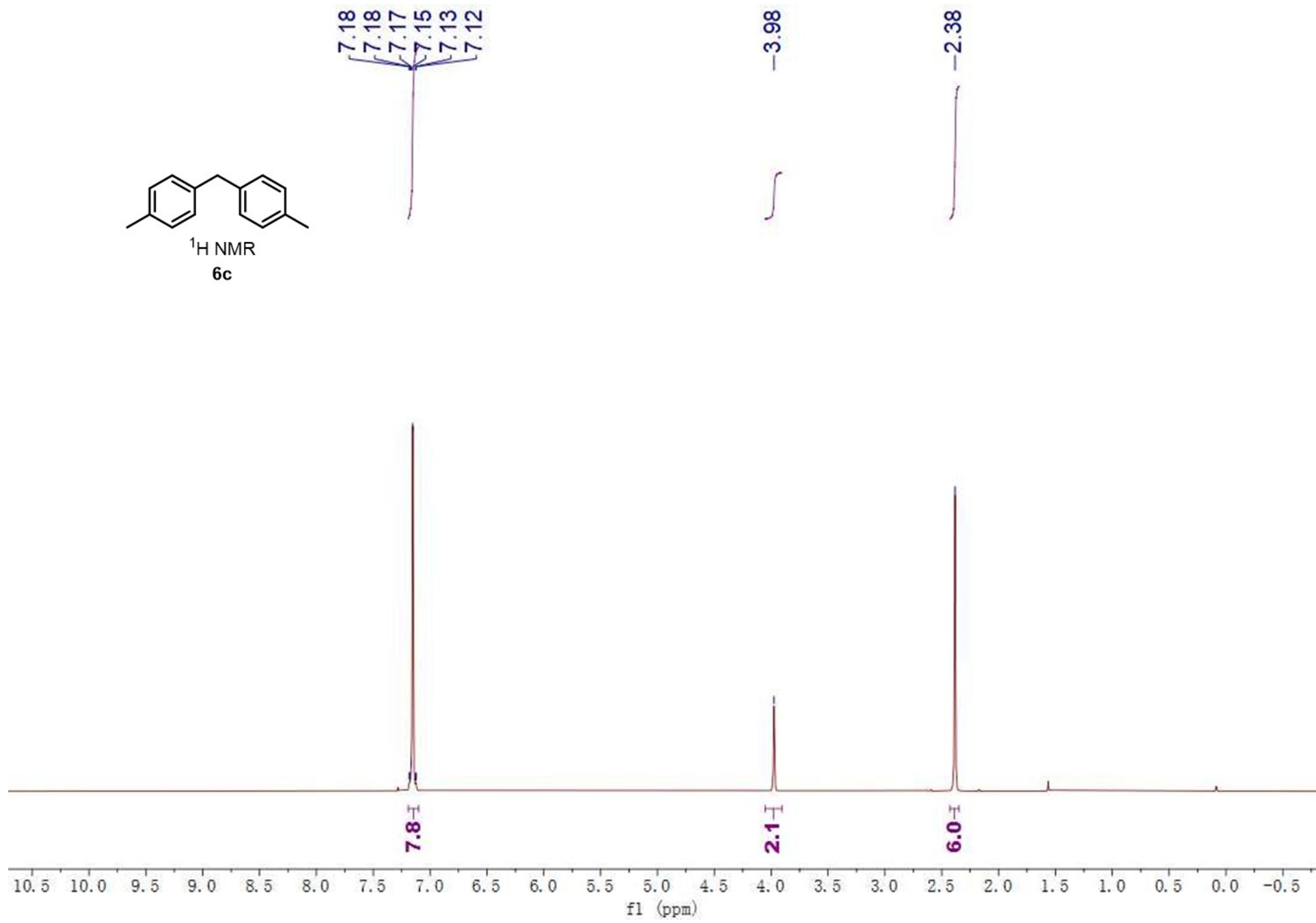
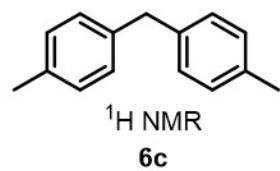
~162.8
~160.3

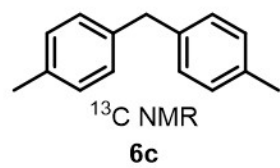
136.7
136.7
130.3
130.2

115.5
115.3

-40.3



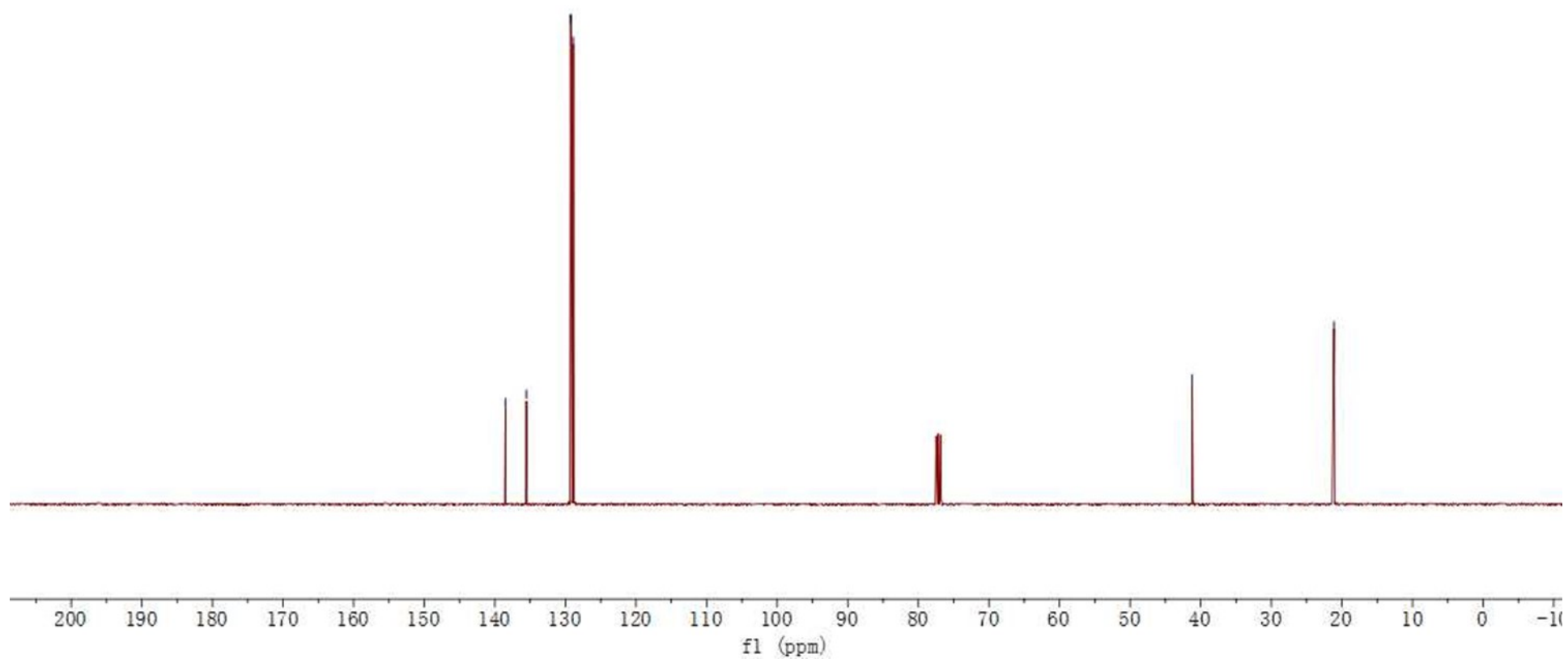


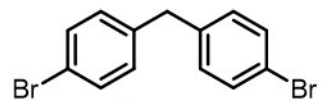


138.5
135.5
129.2
128.9

-41.2

-21.1



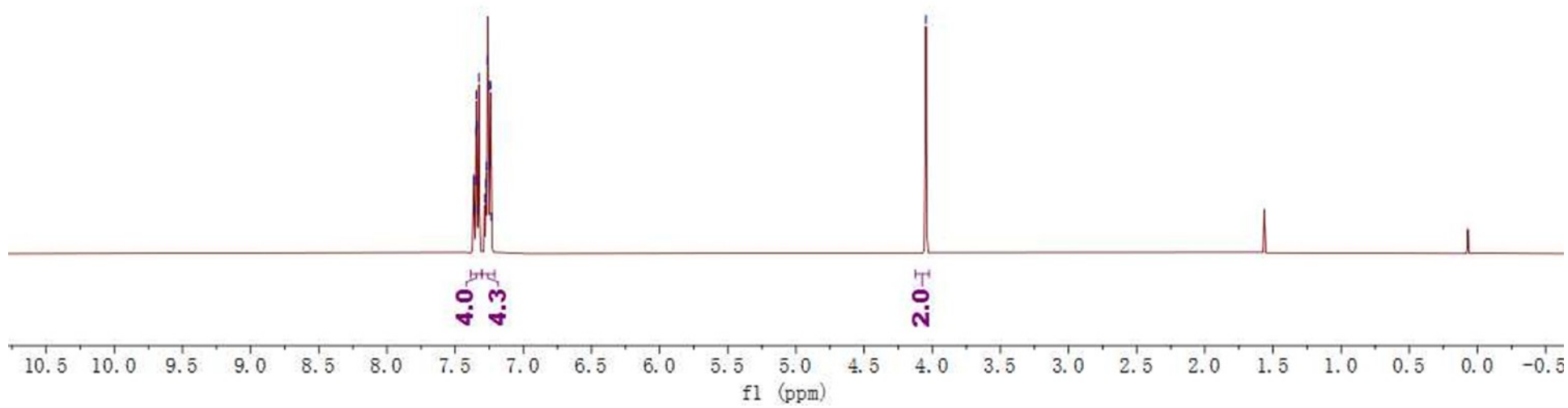


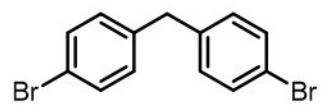
¹H NMR

6d

7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.28
7.27
7.27
7.26
7.25
7.25
7.24
7.23

-4.05





¹³C NMR

6d

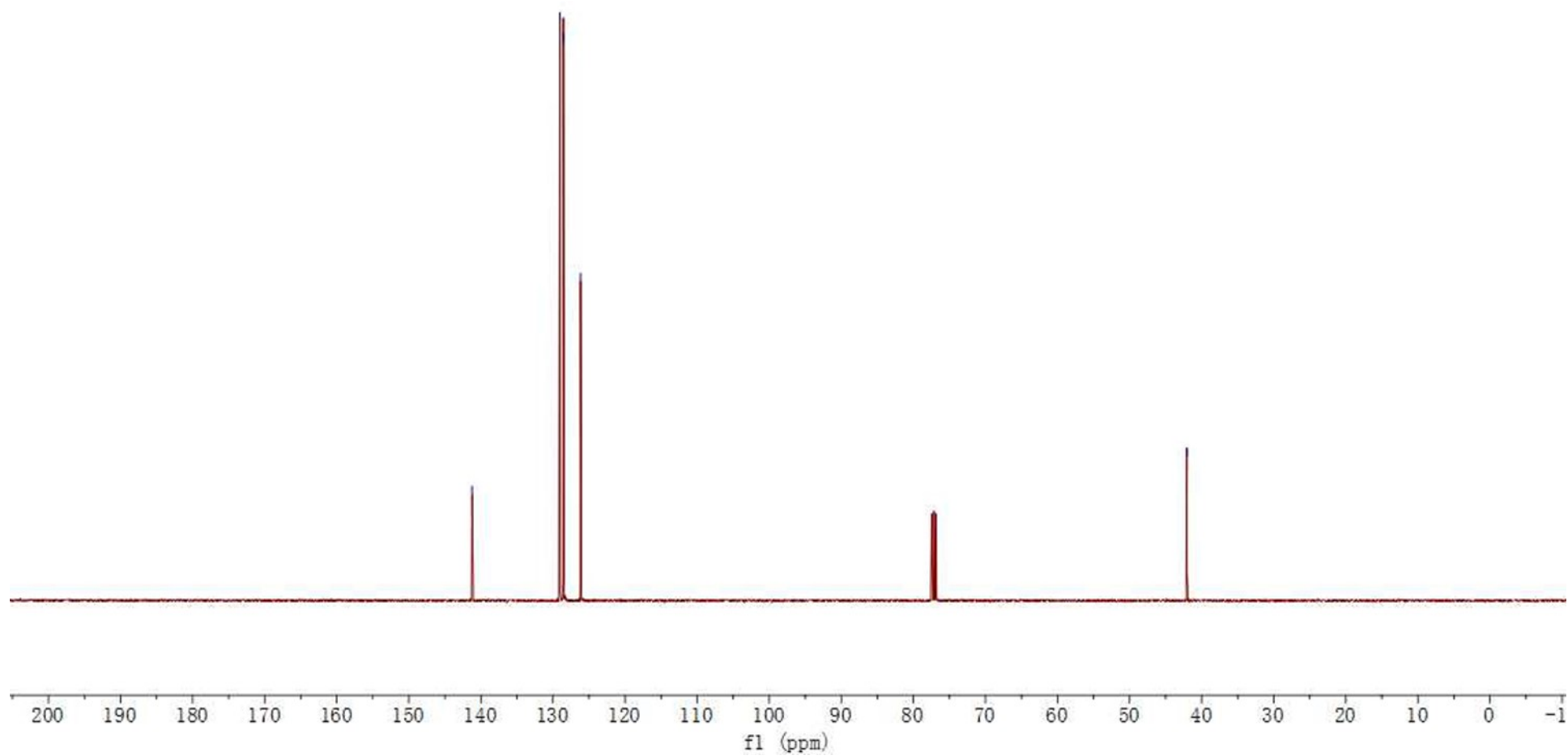
-141.2

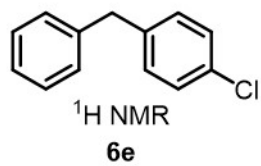
129.1

128.6

126.2

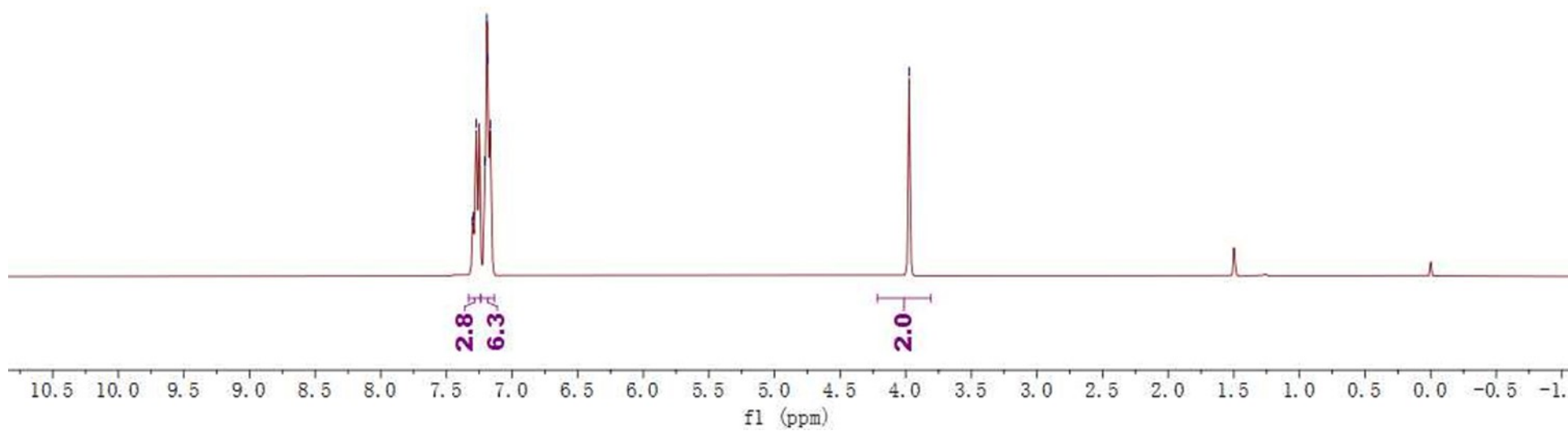
-42.1

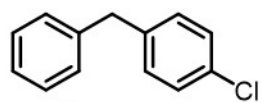




7.30
7.30
7.27
7.21
7.19
7.18
7.17

-3.97





¹³C NMR

6e

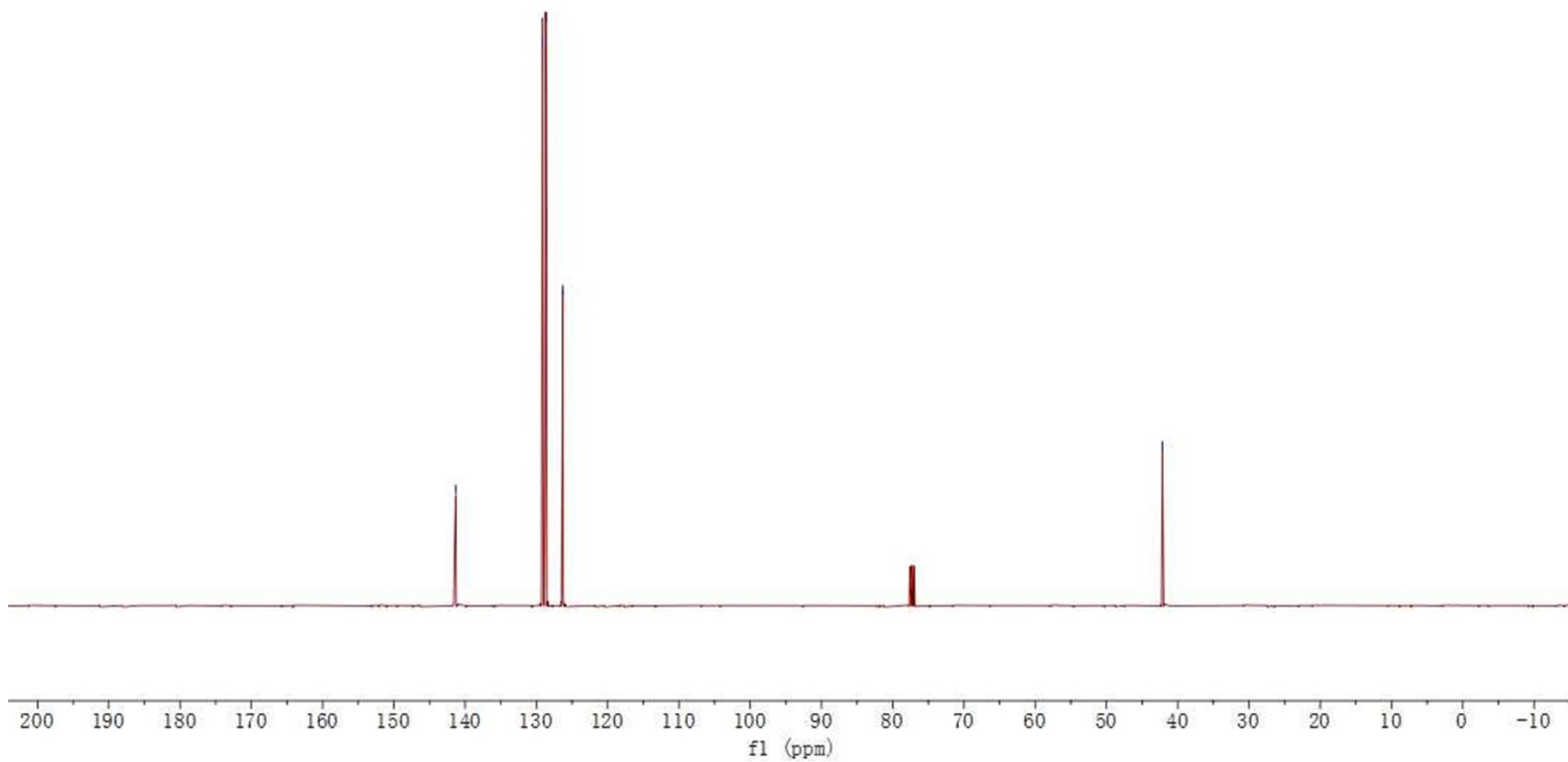
-141.3

129.2

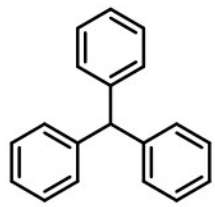
128.7

126.3

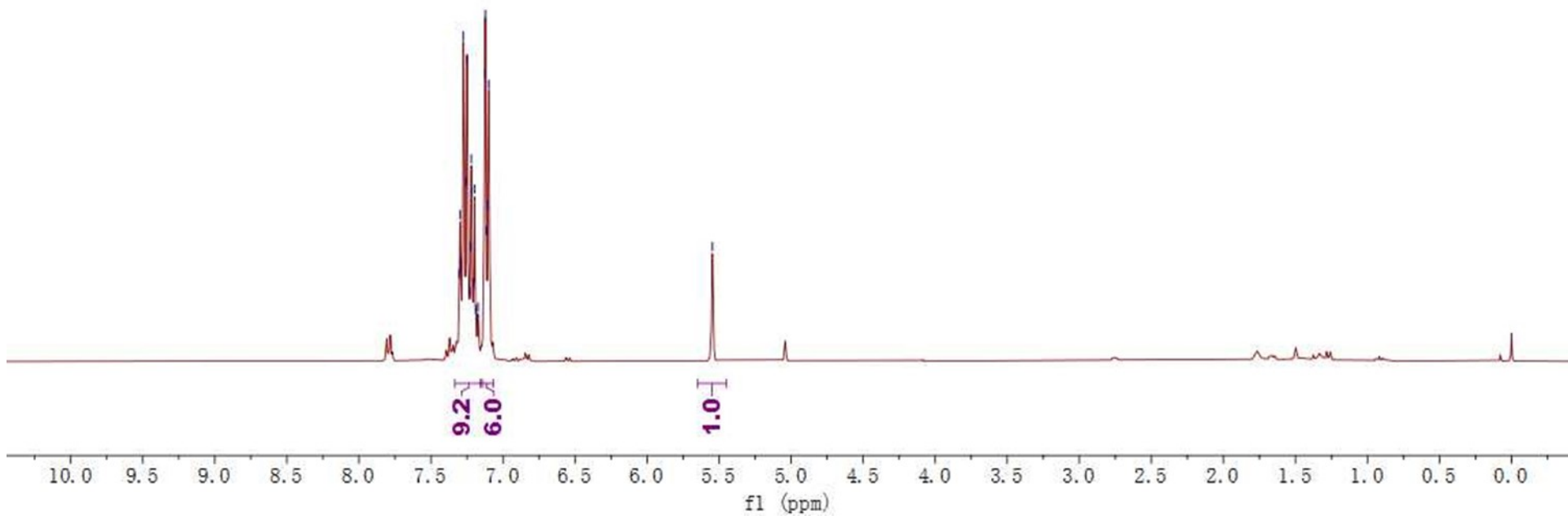
-42.2

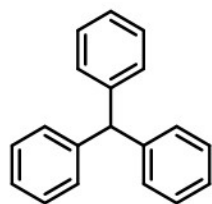


7.30
7.30
7.29
7.28
7.25
7.25
7.23
7.23
7.22
7.22
7.21
7.20
7.19
7.18
7.17
7.13
7.12
7.11
7.11
7.10
-5.55



¹H NMR
6f





¹³C NMR
6f

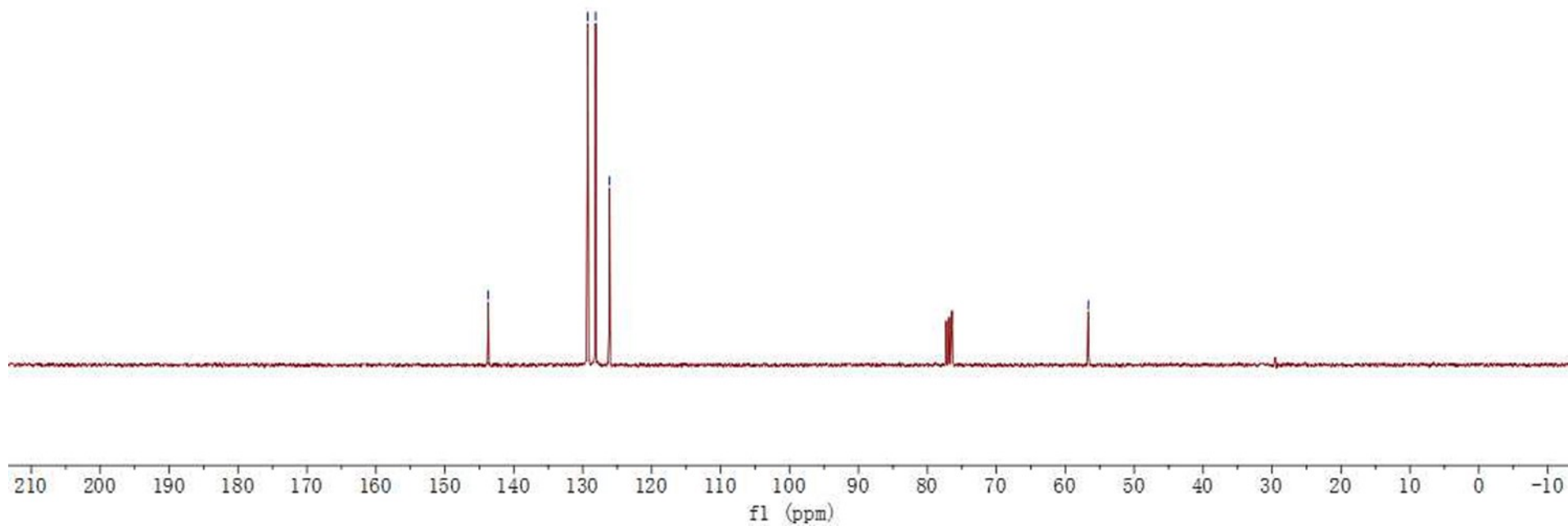
—143.7

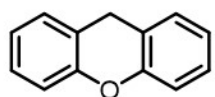
—129.3

—128.1

—126.1

—56.7

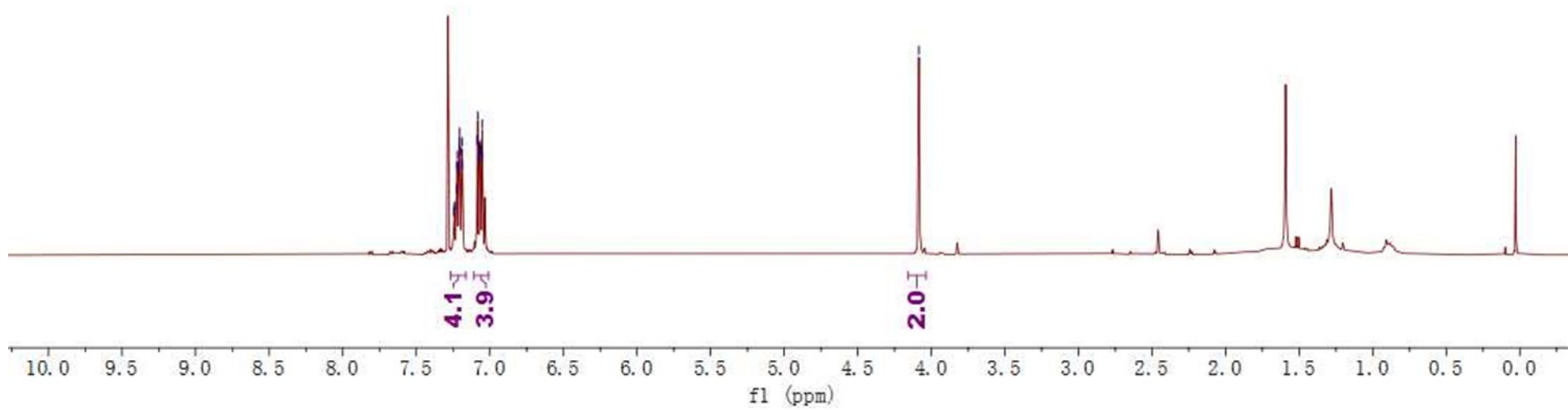


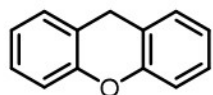


¹H NMR
6g

7.24
7.24
7.24
7.23
7.22
7.22
7.22
7.21
7.21
7.20
7.19
7.19
7.19
7.09
7.08
7.07
7.07
7.06
7.06
7.05
7.05

4.08



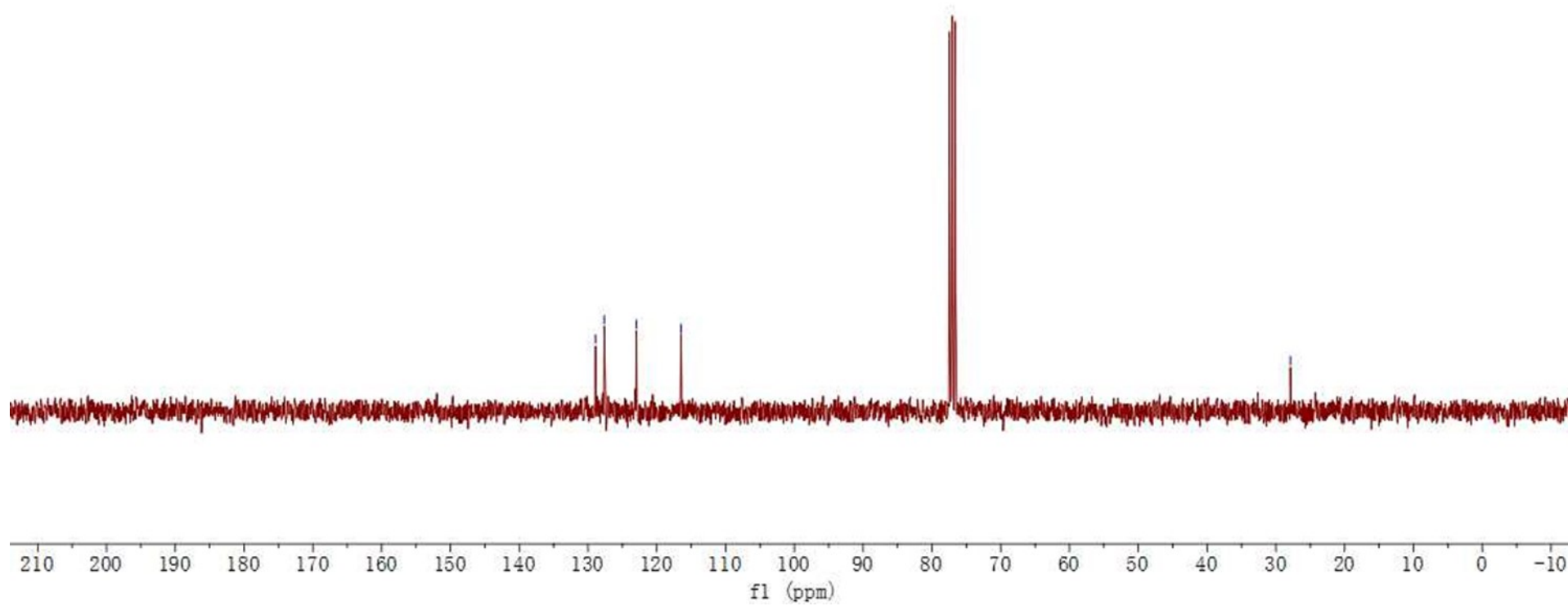


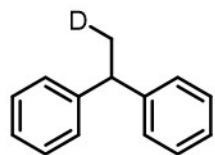
¹³C NMR

6g

128.9
127.6
123.0
116.5

-27.9



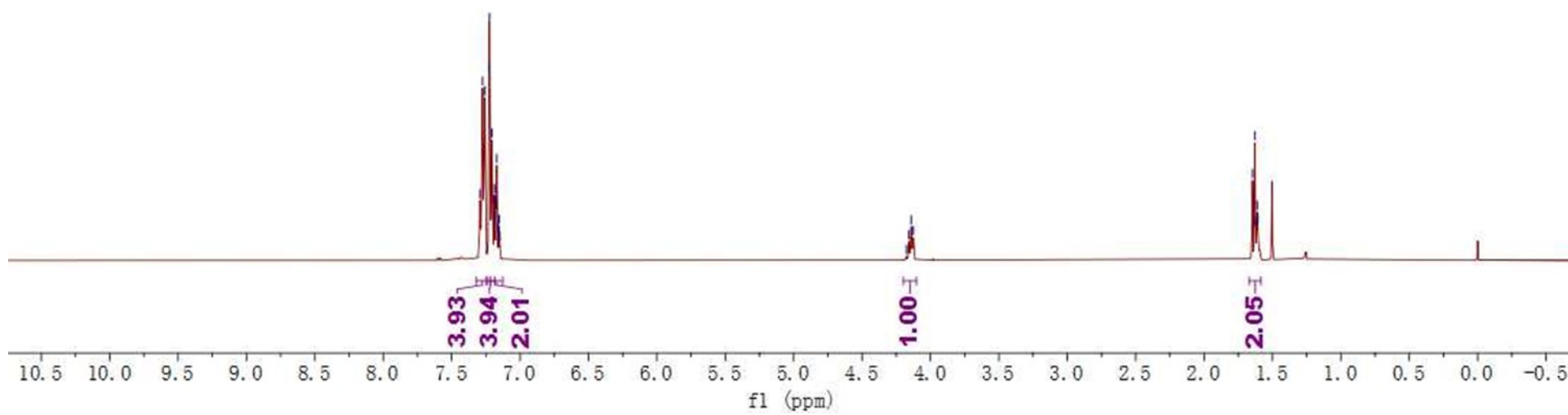


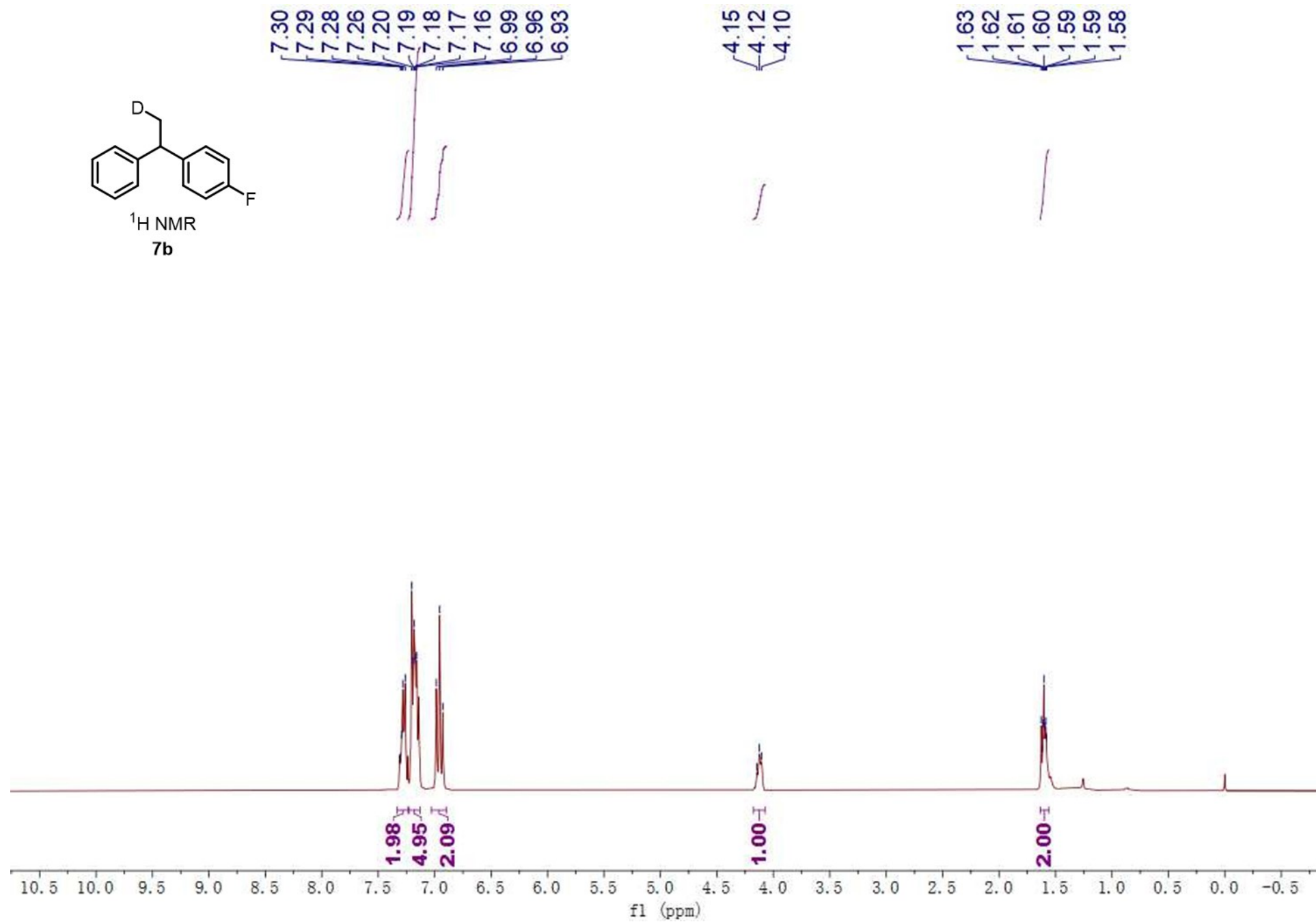
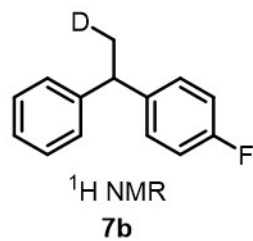
¹H NMR
7a

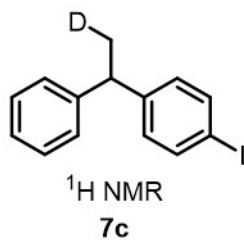
7.29
7.27
7.26
7.23
7.22
7.21
7.19
7.19
7.18
7.17
7.17
7.16
7.16
7.15
7.15

4.18
4.16
4.14
4.13

1.65
1.63
1.63
1.61
1.61



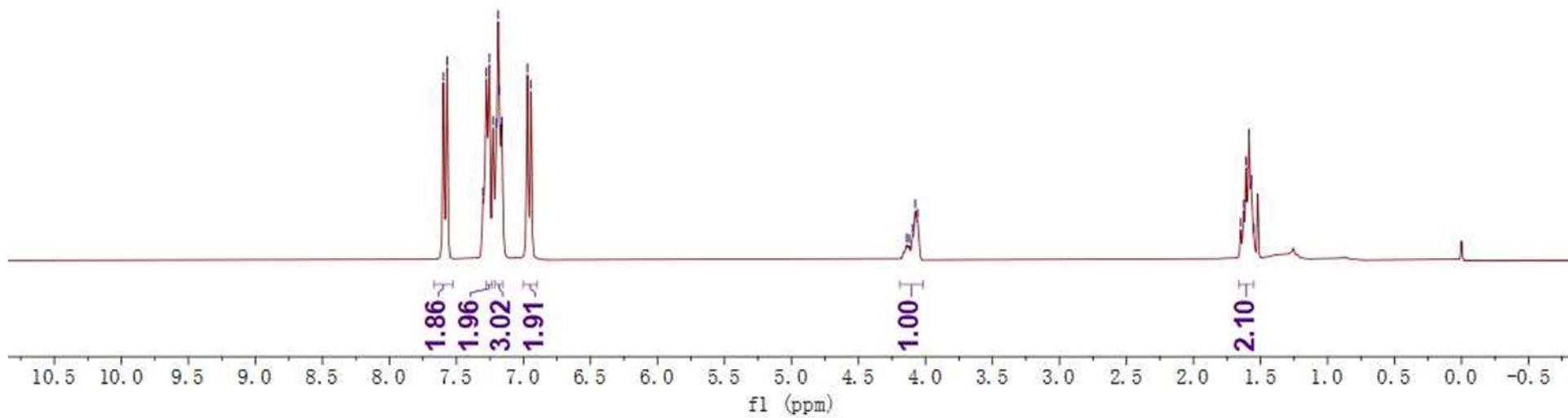


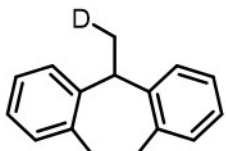


7.60
 7.57
 7.30
 7.28
 7.25
 7.23
 7.20
 7.19
 7.18
 7.17
 7.16
 6.97
 6.94

4.14
 4.12
 4.10
 4.08
 4.06

1.65
 1.62
 1.61
 1.59
 1.57
 1.55





¹H NMR
7d

7.18
7.17
7.16
7.15
7.08
7.08
7.07
7.06
7.05
7.05
7.05
7.04
7.04
7.03

4.37
4.36
4.35

3.15

1.67
1.65
1.65
1.64
1.63

