Supplementary Information (SI) for Organic & Biomolecular Chemistry.

Supporting Information for:

Iron-Catalyzed Regioselective Reductive Cyclotrimerization of Alkynyl Bromides to 1,2,4-Substituted Arenes/D$_3$-Arenes

Xin Zhang,[a] Yuhang Luo,[a] Xinjie Zhao,[a] Tiebo Xiao,[a] Yubo Jiang,[a] Guiping Qin,*[a] and Yubo Wei*[b]

[a] Faculty of Science, Kunming University of Science and Technology, Kunming 650500, P. R. China
[b] School of Pharmaceutical Sciences and Yunnan Key Laboratory of Pharmacology for Natural Products, Kunming Medical University, Kunming, Yunnan, 650500, P. R. China.
* Corresponding author.
E-mail: qin-guiping@kust.edu.cn; weiyubu@kmmu.edu.cn

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1. General experimental details and materials

All non-aqueous reactions and manipulations were using standard Schlenk techniques. All solvents before use were dried and degassed by standard methods and stored under argon atmosphere. All reactions were monitored by TLC with silica gel-coated plates.

NMR spectra were recorded on Bruker Avance III 500 MHz and Bruker Avance III HD 600 MHz spectrometers. Chemical shifts were reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (*J*) were reported in Hertz (Hz) and referred to apparent peak multiplications. The term m, q, t, d, s referred to multiplet, quartet, triplet, doublet, singlet respectively. GC-MS spectra were recorded on a ThermoFisher Scientific ISQ 7000 Series GC-MS system. High resolution mass spectra were recorded on an Agilent Technologies 7250 GCQTOF using electrospray ionization (EI) techniques.

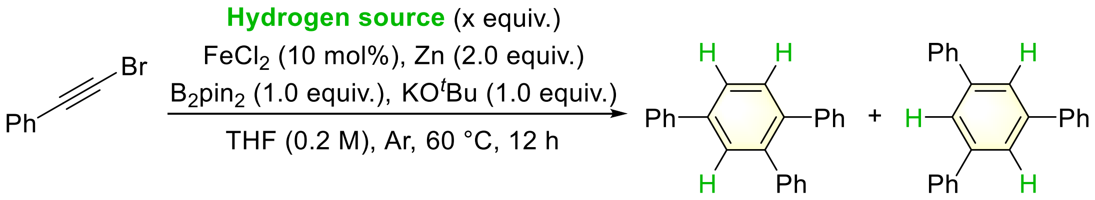
Unless extra specified, the used iron salts, alkynes, and other reagents were all purchased from *Energy*. Alkynyl bromides **1a**-**1al** were prepared according to the known literatures.[5]

2. Optimization of the reaction conditions

Optimization of the reaction conditions for 2a

B_2pin_2 (x equiv.), reductant (x equiv.), catalyst (x mol%), base (x equiv.), solvent (0.2 M), hydrogen source (x equiv.) and phenylethynyl bromide **1a** (0.6 mmol) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford the desired product **2a**.

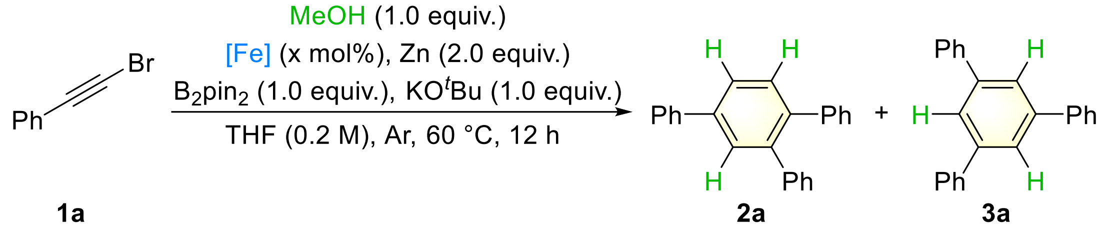
Table S1. Screening of hydrogen source [a]



Entry	Hydrogen soure (equiv.)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	None	<5	-
2	HO^{t}Bu (1.0)	14	99:1
3	H_2O (1.0)	46	99:1
4	$HSiEt_3$ (1.0)	58	97:3
5	HBPin (1.0) [c]	7	99:1
6	**MeOH (1.0)**	**84**	**99:1**
7	MeOH (10)	45	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), hydrogen source, B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu (67.3 mg, 1.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis. [c] HBpin instead of B_2pin_2.

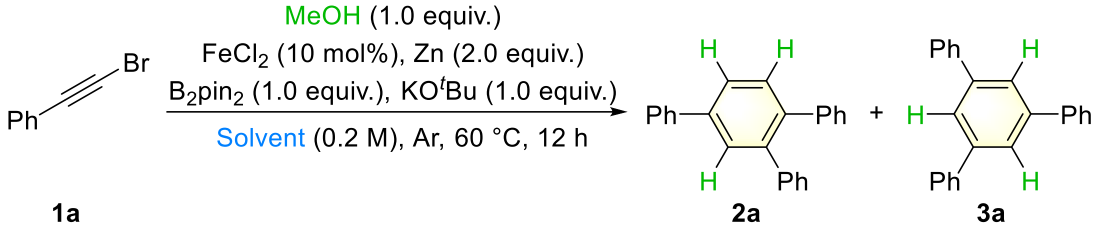
Table S2. Screening of [Fe] catalyst



Entry	[Fe] (mol%)	Yield of **2a**+**3a** (%)	Ratio of **2a**/ **3a** [b]
1	FeC_2O_4 (10)	24	99:1
2	$Fe(OAc)_2$ (10)	30	99:1
3	$Fe(acac)_2$ (10)	<5	-
4	$Fe(OTf)_2$ (10)	<5	-
5	$Fe(OTf)_3$ (10)	<5	-
6	$FeCl_3$ (10)	<5	-
7	**$FeCl_2$ (10)**	**84**	**99:1**
8	$FeCl_2$ (20)	71	99:1
9	$FeCl_2$ (15)	75	99:1
10	$FeCl_2$ (7.5)	71	99:1
11	$FeCl_2$ (5.0)	48	99:1
12	$FeCl_2$ (2.5)	<5	-
13	$FeCl_2$ (1.0)	<5	-
14	None	0	-
15[c]	Fe (10)	0	-

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu (67.3 mg, 1.0 equiv.), [Fe] catalyst, Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.[c] Zn (1.0 equiv.) and $ZnCl_2$ (1.0 equiv.).

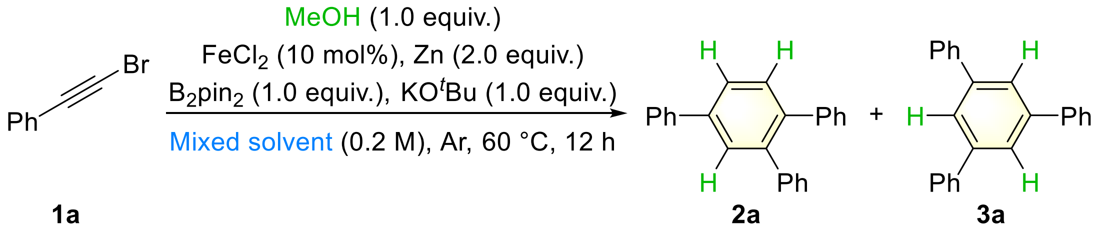
Table S3. Screening of solvent [a]



Entry	Solvent (0.2 M)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	Et_2O	<5	-
2	1,4-Dioxane	15	99:1
3	1,3-Dioxolane	7	99:1
4	**THF**	**84**	**99:1**
5	MeCN	79	93:7
6	Acetone	<5	-
7	EtOH	<5	-
8	MeOH	0	-

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^tBu (67.3 mg, 1.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and solvent (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

Table S4. Screening of mixed solvent [a]

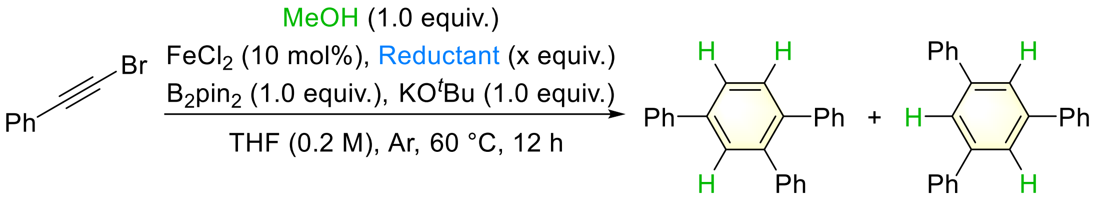


Entry	Mixed solvent (0.2 M)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	THF/MeCN = 1:1	72	92:8
2	THF/MeCN = 2:1	71	92:8
3	THF/MeCN = 3:2	77	94:6
4	THF/MeCN = 4:1	77	97:3
5	THF/MeCN = 9:1	68	98:2
6	THF/MeCN = 1:9	73	89:11

7	THF/MeCN = 1:4	85	87:13
8	THF/MeCN = 2:3	82	90:10
9	THF/MeCN = 1:2	81	87:13
10	**THF/MeCN = 1:0**	**84**	**99:1**
11	THF/MeCN = 0:1	79	93:7

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu (67.3 mg, 1.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and mixed solvents (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

Table S5. Screening of reductant [a]

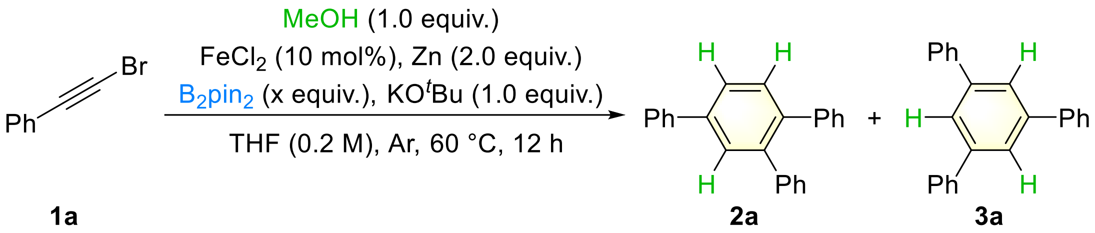


1a **2a** **3a**

Entry	Reductant (equiv.)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	In (2.0)	0	-
2	Mn (2.0)	0	-
3	Mg (2.0)	71	99:1
4	Cr (2.0)	0	-
5	Sn (2.0)	0	-
6	**Zn (2.0)**	**84**	**99:1**
7	Zn (1.5)	71	99:1
8	Zn (1.0)	61	99:1
9	Zn (0.75)	67	99:1
10	Zn (0.5)	19	99:1
11	Zn (0.25)	0	-
12	None	0	-

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu (67.3 mg, 1.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), reductant and THF (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

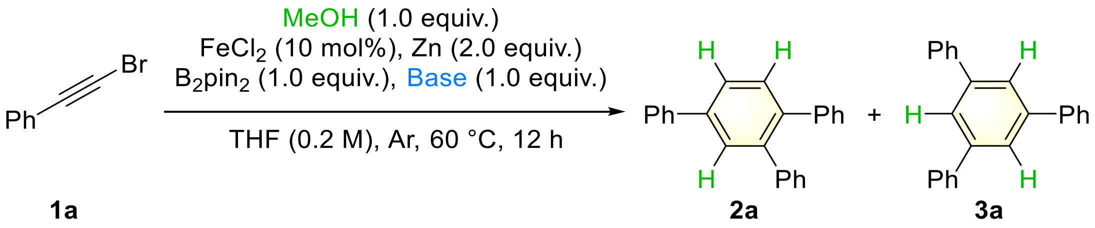
Table S6. Screening the loading of B_2pin_2 [a]



Entry	B_2pin_2 (equiv.)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	1.5	78	99:1
2	**1**	**84**	**99:1**
3	0.75	60	99:1
4	0.5	62	99:1
5	0.25	52	99:1
6	None	<5	-

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2, KO^{t}Bu (67.3 mg, 1.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv) and THF (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

Table S7. Screening of base [a]

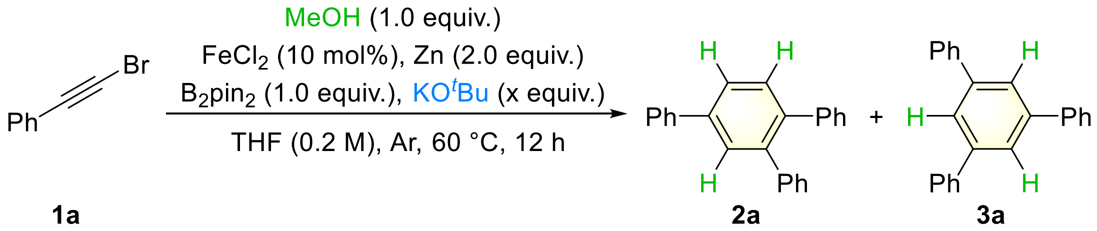


Entry	Base (1.0 equiv.)	Yield of **2a**, **3a** (%)	Ratio of **2a**/ **3a** [b]
1	KO^{t}Bu	84	99:1
2	NaO^{t}Bu	10	99:1
3	LiO^{t}Bu	<5	-
4	K_2CO_3	<5	-
5	Na_2CO_3	<5	-
6	DABCO	0	-
7	DBU	<5	-

8	Et_3N	<5	-
9	None	<5	-

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), base (1.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 °C for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

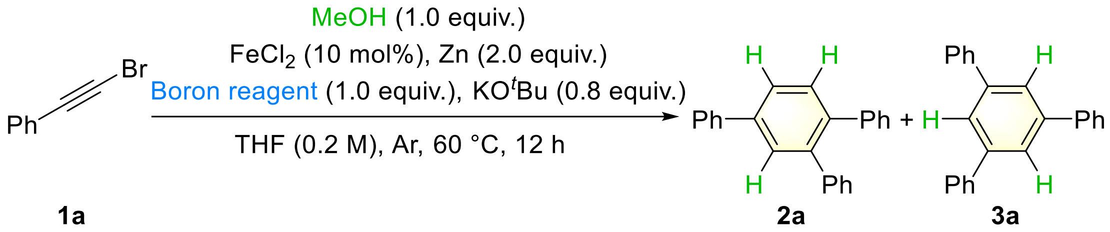
Table S8. Screening the loading of base [a]



Entry	KO^{t}Bu (equiv.)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	0.25	57	99:1
2	0.5	50	99:1
3	0.6	71	99:1
4	0.7	63	99:1
5	0.75	70	99:1
6	**0.8**	**84**	**99:1**
7	0.9	73	99:1
8	1.0	84	99:1
9	1.5	<5	99:1
10	2.0	<5	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu, $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 °C for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

Table S9. Screening of boron reagent [a]



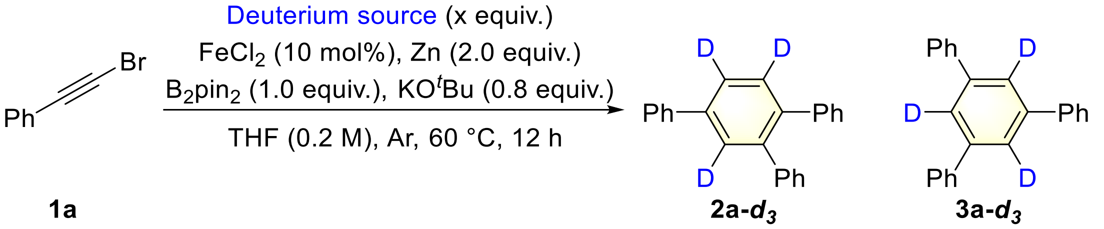
Entry	Boron reagent (1.0 equiv.)	Yield of **2a**+**3a** (%)	Ratio of **2a**/**3a** [b]
1	$B_2(OH)_4$	ND	-
2	$B_2(cat)_2$	22	99:1
3	B_2pin_2	84	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), MeOH (19.2 mg, 1.0 equiv.), boron reagent (1.0 equiv.), KO^{t}Bu (53.9 mg, 0.8 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 ºC for 12 h, isolated yields. [b] Ratio of **2a**/**3a** was determined by GC-MS analysis.

Optimization of the reaction conditions for 2a-*d*$_3$

B_2pin_2 (1.0 equiv.), Zn (2.0 equiv.), catalyst (x mol%), KO^tBu (x equiv.), additive (x mol%), solvent (x M), deuterium source (x equiv.) and phenylethynyl bromide **1a** (0.6 mmol) were added in sequence to a flame-dried 25 mL Schlenk tube in the glove box. The mixture was stirred at x ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford the desired product **2a-*d*$_3$**.

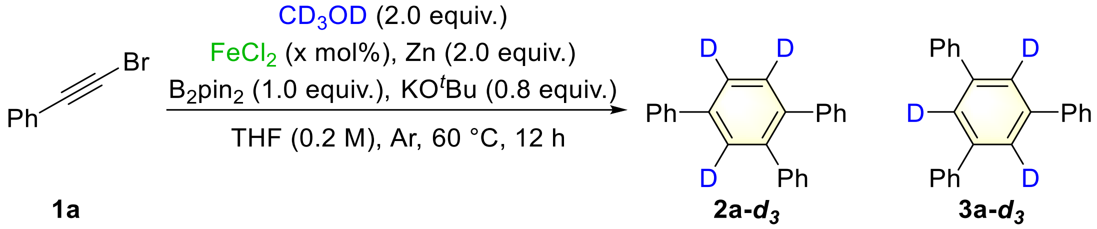
Table S10. Screening of deuterium source[a]



Entry	Deuterium (equiv.)	Yield of **2a-*d*$_3$**+**3a-*d*$_3$** (%)	Ratio of **2a-*d*$_3$**/**3a-*d*$_3$**[b]
1	D_2O (1.0)	15	99:1
2	CH_3OD (1.0)	18	99:1
3	CD_3OD (1.0)	20	99:1
4	**CD_3OD (2.0)**	**50**	**99:1**
5	CD_3OD (4.0)	34	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), deuterium source, B_2pin_2 (152.4 mg, 1.0 equiv.), KO^tBu (53.9 mg, 0.8 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 ºC for 12 h in the glove box, isolated yields. [b] Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** was determined by GC-MS analysis.

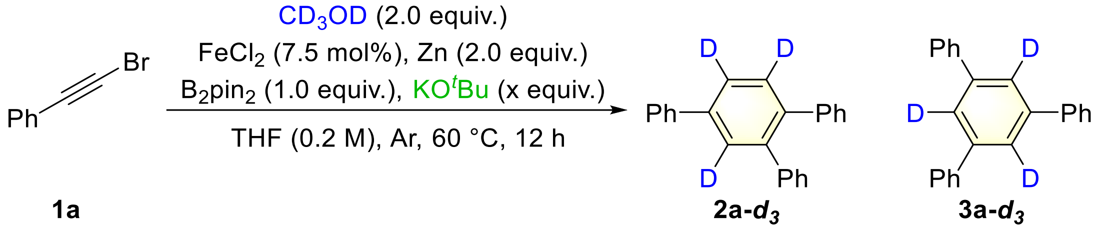
Table S11. Screening the loading of $FeCl_2$ [a]



Entry	$FeCl_2$ (mol%)	Yield of **2a-*d*$_3$**+**3a-*d*$_3$** (%)	Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** [b]
1	2.5	10	99:1
2	5	12	99:1
3	**7.5**	**68**	**99:1**
4	10	50	99:1
5	15	48	99:1
6	20	12	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), CD_3OD (2.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu (53.9 mg, 0.8 equiv.), $FeCl_2$, Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 ºC for 12 h in the glove box, isolated yields. [b] Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** was determined by GC-MS analysis.

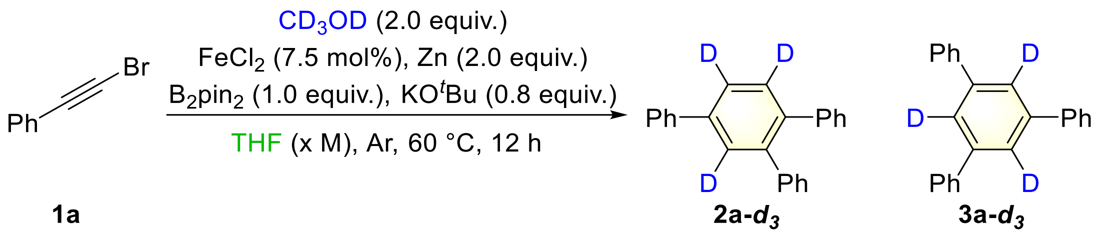
Table S12. Screening the loading of KO^{t}Bu [a]



Entry	KO^{t}Bu (equiv.)	Yield of **2a-*d*$_3$**+**3a-*d*$_3$** (%)	Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** [b]
1	0.25	<5	99:1
2	0.5	16	99:1
3	**0.8**	**68**	**99:1**
4	0.9	30	99:1
5	1.0	37	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), CD_3OD (2.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^{t}Bu, $FeCl_2$ (5.7 mg, 7.5 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL) under the argon at 60 ºC for 12 h in the glove box, isolated yields. [b] Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** was determined by GC-MS analysis.

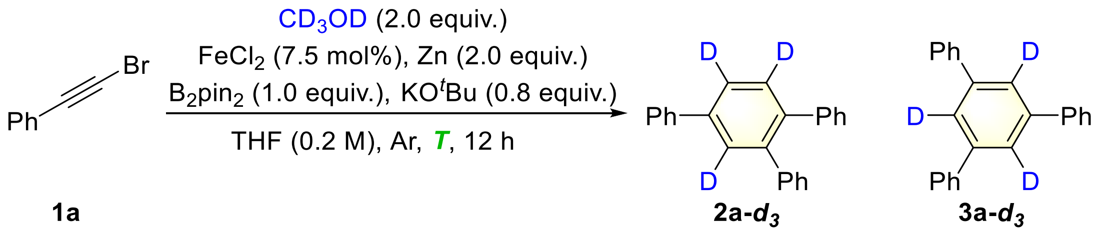
Table S13. Screening of concentration [a]



Entry	THF (M)	Yield of **2a-*d*$_3$**+**3a-*d*$_3$** (%)	Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** [b]
1	0.1	20	99:1
2	0.15	26	99:1
3	**0.2**	**68**	**99:1**
4	0.3	40	99:1
5	0.6	41	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), CD_3OD (2.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^tBu (53.9 mg, 0.8 equiv.), $FeCl_2$ (5.7 mg, 7.5 mol%), Zn (78.5 mg, 2.0 equiv.) and THF, under the argon at 60 ºC for 12 h in the glove box, isolated yields. [b] Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** was determined by GC-MS analysis.

Table S14. Screening of temperature [a]



Entry	*T* (ºC)	Yield of **2a-*d*$_3$**+**3a-*d*$_3$** (%)	Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** [b]
1	40	23	99:1
2	50	50	99:1
3	**60**	**68**	**99:1**
4	80	41	99:1
5	100	38	99:1

[a] Reaction condition: **1a** (108.6 mg, 0.6 mmol), CD_3OD (2.0 equiv.), B_2pin_2 (152.4 mg, 1.0 equiv.), KO^tBu (53.9 mg, 0.8 equiv.), $FeCl_2$ (5.7 mg, 7.5 mol%), Zn (78.5 mg, 2.0 equiv.) and THF (3.0 mL), under the argon at designed temperature for 12 h in the glove box, isolated yields. [b] Ratio of **2a-*d*$_3$**/**3a-*d*$_3$** was determined by GC-MS analysis.

3. General procedure

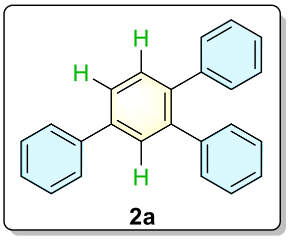
Condition A: B_2pin_2 (152.4 mg, 1.0 equiv.), Zn (78.5 mg, 2.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), KO^{t}Bu (53.9 mg, 0.8 equiv.), THF (3 mL, 0.2 M), MeOH (19.2 mg, 1.0 equiv.) and alkynyl bromides **1** (0.6 mmol) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the Argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether/ethyl acetate to afford the mixture of **2** + **3** as the products. The regioselectivity was determined by GC-MS or ^{1}H NMR.

Condition B: B_2pin_2 (152.4 mg, 1.0 equiv.), Zn (78.5 mg, 2.0 equiv.), $FeCl_2$ (5.7 mg, 7.5 mol%), KO^{t}Bu (53.9 mg, 0.8 equiv.), THF (3 mL, 0.2 M), CD_3OD (43.3 mg, 2.0 equiv.) and alkynyl bromides **1** (0.6 mmol) were added in sequence to a flame-dried 25 mL Schlenk tube in an argon-filled glove box. The mixture was stirred at 60 ºC for 12 hours. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether/ethyl acetate to afford the mixture of **2a-*d*$_3$** + **3a-*d*$_3$** as the products. The regioselectivity was determined by GC-MS.

Condition C: B_2pin_2 (152.4 mg, 1.0 equiv.), Zn (78.5 mg, 2.0 equiv.), $FeCl_2$ (5.7 mg, 7.5 mol%), KO^{t}Bu (53.9 mg, 0.8 equiv.), THF (3 mL, 0.2 M), CD_3OD (32.5 mg, 1.5 equiv.) and alkynyl bromides **1** (0.6 mmol) were added in sequence to a flame-dried 25 mL Schlenk tube in an argon-filled glove box. The mixture was stirred at 60 ºC for 12 hours. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether/ethyl acetate to afford the mixture of **2a-*d*$_3$** + **3a-*d*$_3$** as the products. The regioselectivity was determined by GC-MS.

4. Experimental characterization data for products

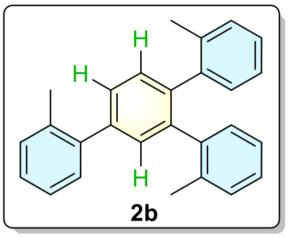
1,2,4-Triphenylbenzene (2a):



2a

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (51.3 mg, 84% yield). This compound is previously known.[1] Therefore the evaluation of ^{13}C NMR was omitted. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67-7.64 (m, 4H), 7.51-7.43 (m, 3H), 7.37-7.34 (m, 1H), 7.22-7.17 (m, 10H). Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.78 ppm) according to ^{1}H NMR: (0.03/3)/[1+(0.03/3)] = 1%.

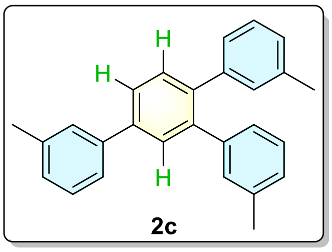
1,2,4-Tris(2-methylphenyl)benzene (2b):



2b

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (32.1 mg, 46% yield). This compound is previously known.[4] ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35-7.33 (m, 3H), 7.28-7.24 (m, 5H), 7.08-6.90 (m, 8H), 2.39 (s, 3H), 2.20 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 140.4, 139.3, 135.8, 135.5, 131.6, 131.5, 130.5, 130.1, 127.7, 127.4, 126.9, 126.0, 125.1, 124.9, 20.8, 20.2. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 69/7428 = 0.9%.

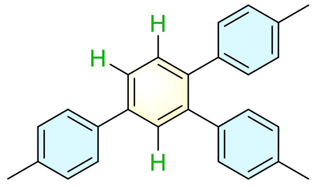
1,2,4-Tris(3-methylphenyl)benzene (2c):



2c

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (34.0 mg, 49% yield). This compound is previously known.[1] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64-7.60 (m, 2H), 7.48-7.46 (m, 3H), 7.35-7.32 (m, 1H), 7.18 (d, J = 7.5 Hz, 1H), 7.10-7.00 (m, 6H), 6.94-6.91 (m, 2H), 2.42 (s, 3H), 2.27 (s, 3H), 2.26 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 141.2, 141.1, 140.8, 140.4, 139.6, 138.5, 137.6, 137.5, 131.1, 130.7, 130.6, 129.5, 128.9, 128.3, 128.1, 127.8, 127.7, 127.4, 127.3, 127.2, 127.2, 126.1, 124.4, 21.7, 21.6. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 13/5428 = 0.2%.

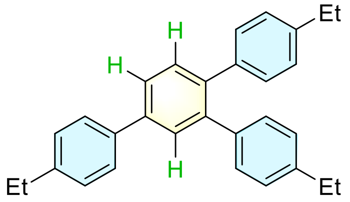
1,2,4-Tris(4-methylphenyl)benzene (2d):



2d

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (38.9 mg, 56% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.62-7.55 (m, 4H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.10-7.02 (m, 8H), 2.39 (s, 3H), 2.32 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 141.0, 140.2, 139.4, 139.0, 138.6, 138.0, 137.3, 136.3, 136.2, 131.2, 129.9, 129.9, 129.7, 129.4, 128.8, 128.8, 127.1, 125.9, 21.3, 21.2, 21.2. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.73 ppm) according to ^{1}H NMR: (0.02/3)/[1+(0.02/3)] = 0.7%.

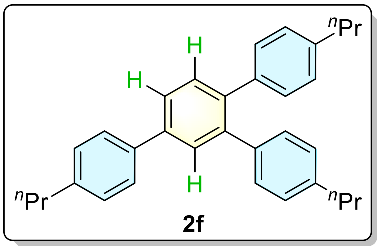
1,2,4-Tris(4-ethylphenyl)benzene (2e):



2e

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (46.1 mg, 59% yield). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.63-7.57 (m, 4H), 7.47 (dd, *J* = 8.0, 2.5 Hz, 1H), 7.28-7.26 (m, 2H), 7.12-7.04 (m, 8H), 2.71 (q, *J* = 7.5 Hz, 2H), 2.64-2.59 (m, 4H), 1.29-1.26 (m, 3H), 1.23-1.20 (m, 6H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 143.6, 142.5, 142.4, 140.9, 140.2, 139.3, 139.1, 138.7, 138.2, 131.3, 129.9, 129.9, 129.5, 128.5, 127.5, 127.5, 127.2, 125.9, 28.7, 28.6, 15.7, 15.6, 15.5. HRMS (EI): m/z = calcd. for $(C_{30}H_{30})^{\bullet+}$: 390.2342, found 390.2353. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 81/24223 = 0.3%.

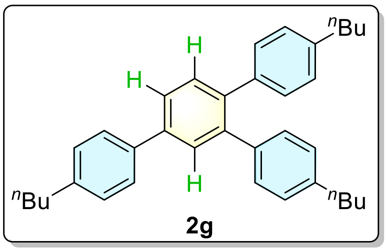
1,2,4-Tris(4-*n*-propylphenyl)benzene (2f):



2f

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (61.2 mg, 71% yield). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.64-7.57 (m, 4H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.10-7.00 (m, 8H), 2.63 (t, *J* = 7.5 Hz, 2H), 2.56-2.53 (m, 4H), 1.72-1.58 (m, 6H), 0.97 (t, *J* = 7.5 Hz, 3H), 0.91 (m, 6H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 142.0, 141.0, 140.9, 140.9, 140.1, 139.4, 139.1, 138.7, 138.2, 131.2, 129.9, 129.8, 129.3, 129.1, 128.1, 128.1, 127.1, 125.8, 37.9, 37.8, 24.7, 24.6, 14.1, 13.9, 13.9. HRMS (EI): m/z = calcd. for $(C_{33}H_{36})^{\bullet+}$: 432.2812, found 432.2826. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.74 ppm) according to ^{1}H NMR: (0.02/3)/[1+(0.02/3)] = 0.7%.

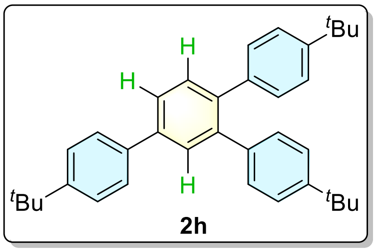
1,2,4-Tris(4-*n*-butylphenyl)benzene (2g):



2g

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (74.7 mg, 79% yield). This compound is previously known.[1] ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.64-7.56 (m, 4H), 7.46-7.44 (m 1H), 7.24 (d, *J* = 7.2 Hz, 2H), 7.14-7.00 (m, 8H), 2.65-2.55 (m, 6H), 1.64-1.55 (m, 6H), 1.41-1.30 (m, 6H), 0.95-0.90 (m, 9H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 142.3, 141.2, 141.1, 141.0, 140.1, 139.4, 139.1, 138.7, 138.2, 131.1, 129.9, 129.8, 129.4, 129.0, 128.1, 128.0, 127.0, 125.8, 35.5, 35.4, 33.8, 33.7, 22.6, 22.5, 22.5, 14.2. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.74 ppm) according to ^{1}H NMR: (0.01/3)/[1+(0.01/3)] = 0.3%.

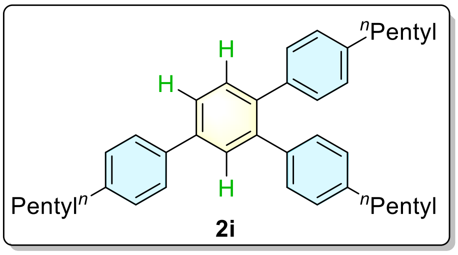
1,2,4-Tris(4-tert-butylphenyl)benzene (2h):



2h

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow solid (65.1 mg, 69% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (s, 1H), 7.61 (d, J = 8.5 Hz, 3H), 7.48-7.46 (m, 3H), 7.24-7.21 (m, 4H), 7.12-7.08 (m, 4H), 1.36 (s, 9H), 1.30 (s, 9H), 1.29 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 150.4, 149.4, 149.3, 140.9, 140.0, 139.3, 138.8, 138.4, 138.0, 131.2, 129.7, 129.6, 129.4, 126.9, 125.9, 124.8, 124.8, 34.7, 34.6, 34.6, 31.5, 31.5. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

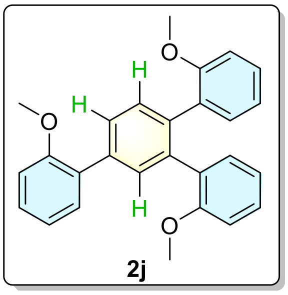
1,2,4-Tris(4-*n*-pentylphenyl)benzene (2i):



2i

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (48.5 mg, 47% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, J = 2.0 Hz, 1H), 7.60-7.56 (m, 3H), 7.47 (d, J = 7.5 Hz, 1H), 7.26 (d, J = 8.0 Hz, 2H), 7.10-7.06 (m, 4H), 7.03-7.00 (m, 4H), 2.64 (t, J =7.5 Hz, 2H), 2.58-2.54 (m, 4H), 1.69-1.56 (m, 6H), 1.36-1.27 (m, 12H), 0.92-0.87 (m, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.3, 141.2, 141.1, 141.0, 140.1, 139.3, 139.1, 138.7, 138.2, 131.1, 129.9, 129.8, 129.4, 129.0, 128.1, 128.0, 127.1, 125.8, 35.8, 35.7, 31.7, 31.7, 31.6, 31.4, 31.2, 22.7, 22.7, 14.3, 14.2. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.74 ppm) according to ^{1}H NMR: (0.02/3)/[1+(0.02/3)] = 0.7%.

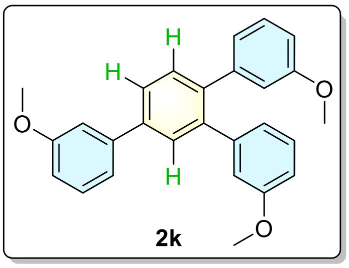
1,2,4-Tris(2-methoxyphenyl)benzene (2j):



2j

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (97:3) to give a yellow solid (55.9 mg, 70% yield). This compound is previously known.[3] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61-7.59 (m, 2H), 7.45-7.43 (m, 2H), 7.32-7.29 (m, 1H), 7.16-7.10 (m, 4H), 7.04-6.98 (m, 2H), 6.83-6.80 (m, 2H), 6.71 (t, *J* = 8.5 Hz, 2H), 3.83 (s, 3H), 3.49 (s, 3H), 3.46 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 156.7, 156.4, 137.2, 131.9, 131.6, 131.2, 131.0, 130.5, 130.3, 128.5, 128.4, 128.1, 120.9, 119.9, 111.1, 110.3, 55.6, 55.1, 55.0. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 6/12887 = 0.05%.

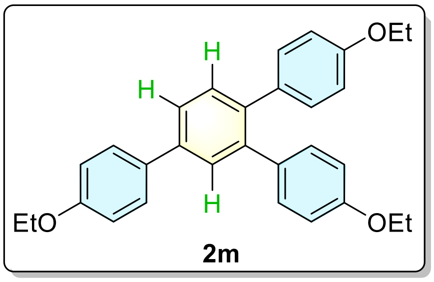
1,2,4-Tris(3-methoxyphenyl)benzene (2k):



2k

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (97:3) to give a yellow oil (58.1 mg, 73% yield). This compound is previously known.[3] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67-7.62 (m, 2H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.38-7.35 (m, 1H), 7.26-7.24 (m, 1H), 7.20-7.19 (m, 1H), 7.17-7.13 (m, 2H), 6.92-6.90 (m, 1H), 6.84-6.70 (m, 6H), 3.86 (s, 3H), 3.62 (s, 3H), 3.62 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.1, 159.2, 159.2, 142.9, 142.5, 142.1, 140.9, 140.4, 139.7, 131.0, 130.0, 129.3, 129.1, 129.1, 126.4, 122.4, 122.3, 119.8, 115.2, 115.2, 113.0, 112.9, 112.8, 55.4, 55.2, 55.2. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.77 ppm) according to ^{1}H NMR: $(0.02/3)/[1+(0.02/3)] = 0.7\%$.

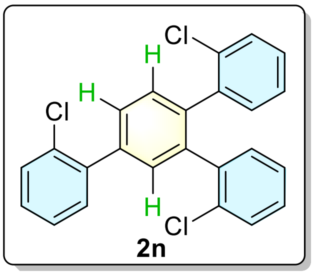
1,2,4-Tris(4-ethoxyphenyl)benzene (2m):



2m

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (97:3) to give a yellowish solid (59.5 mg, 68% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.53 (m, 4H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.11-7.06 (m, 4H), 6.98-6.95 (m, 2H), 6.78-6.74 (m, 4H), 4.09 (q, *J* = 7.0 Hz, 2H), 4.02-3.97 (m, 4H), 1.45-1.38 (m, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 158.7, 157.8, 157.7, 140.5, 139.7, 138.5, 134.1, 133.7, 133.1, 131.1, 131.0, 131.0, 129.0, 128.2, 125.4, 114.9, 114.0, 114.0, 63.6, 63.4, 63.4, 15.0. HRMS (EI): m/z = calcd. for $(C_{30}H_{30}O_3)^{\bullet+}$: 438.2189, found 438.2196. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

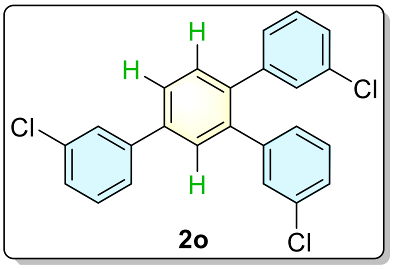
1,2,4-Tris(2-chlorophenyl)benzene (2n):



2n

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (61.4 mg, 75% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56-7.46 (m, 5H), 7.35-7.27 (m, 4H), 7.13-7.01 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.9, 139.6, 139.5, 138.5, 138.4, 137.9, 133.3, 133.2, 132.7, 132.5, 131.9, 131.7, 131.6, 130.7, 130.4, 130.2, 129.8, 129.2, 128.8, 128.6, 128.6, 127.0, 126.3, 125.9. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 14/11458 = 0.1%.

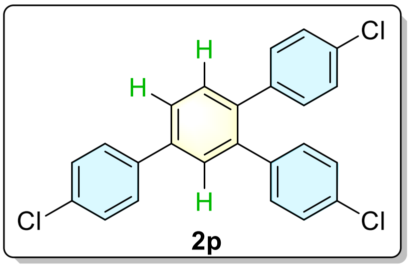
1,2,4-Tris(3-chlorophenyl)benzene (2o):



2o

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellowish solid (43.4 mg, 53% yield). This compound is previously known.[1] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64-7.58 (m, 3H), 7.53-7.46 (m, 2H), 7.39-7.33 (m, 2H), 7.25-7.20 (m, 4H), 7.16-7.12 (m, 2H), 6.98-6.94 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.8, 142.5, 142.2, 139.9, 139.7, 138.8, 135.0, 134.3, 134.2, 131.3, 130.3, 129.8, 129.8, 129.4, 129.4, 129.3, 128.3, 128.3, 127.9, 127.4, 127.3, 127.2, 126.8, 125.4. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.65 ppm) according to ^{1}H NMR: (0.02/3)/[1+(0.02/3)] = 0.7%.

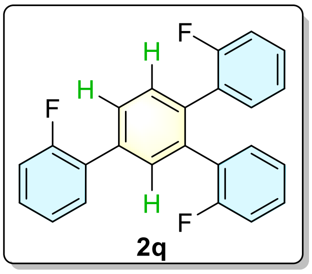
1,2,4-Tris(4-chlorophenyl)benzene (2p):



2p

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (51.8 mg, 63% yield). This compound is previously known.[1] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61-7.56 (m, 4H), 7.46-7.41 (m, 3H), 7.24-7.21 (m, 4H), 7.11-7.06 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 140.1, 139.8, 139.6, 139.3, 138.9, 138.8, 134.0, 133.3, 133.2, 131.3, 131.2, 131.2, 129.3, 129.2, 128.6, 128.5, 128.5, 126.5. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.68 ppm) according to ^{1}H NMR: (0.02/3)/[1+(0.02/3)] = 0.7%.

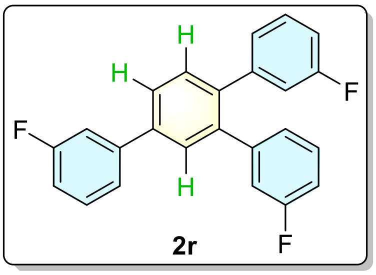
1,2,4-Tris(2-fluorophenyl)benzene (2q):



2q

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellowish solid (50.3 mg, 70% yield). This compound is previously known.[2] The analytical data were in agreement with the literature, therefore the evaluation of ^{13}C NMR was omitted. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67-7.64 (m, 2H), 7.54-7.51 (m, 2H), 7.35-7.30 (m, 1H), 7.22-7.13 (m, 6H), 7.02-6.99 (m, 2H), 6.96-6.91 (m, 2H). ^{19}F NMR (471 MHz, $CDCl_3$) δ -115.15 (2F), -117.59 (1F). Amount of the 1,3,5-regioisomer according to GC-MS analysis: 4/1971 = 0.2%.

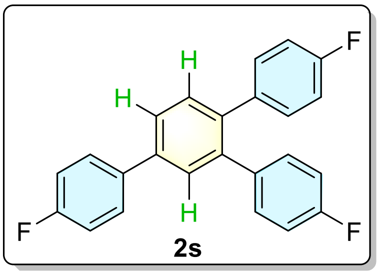
1,2,4-Tris(3-fluorophenyl)benzene (2r):



2r

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (25.1 mg, 35% yield). This compound is previously known.[1] The analytical data were in agreement with the literature, therefore the evaluation of ^{13}C NMR was omitted. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.65-7.61 (m, 2H), 7.50 (d, J = 7.8 Hz, 1H), 7.45-7.40 (m, 2H), 7.36 (d, J = 9.6 Hz, 1H), 7.23-7.18 (m, 2H), 7.09–7.06 (m, 1H), 6.95-6.88 (m, 6H). ^{19}F NMR (471 MHz, CDCl$_3$) δ -112.73 (1F), -113.27 (1F), -113.35 （1F）. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.75 ppm) according to ^{1}H NMR: (0.02/3)/[1+(0.02/3)] = 0.7%.

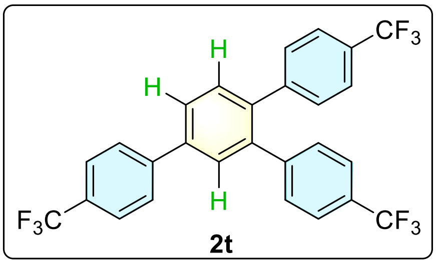
1,2,4-Tris(4-fluorophenyl)benzene (2s):



2s

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (40.5 mg, 56% yield). This compound is previously known.[1] The analytical data were in agreement with the literature, therefore the evaluation of ^{13}C NMR and ^{19}F NMR were omitted. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.61-7.55 (m, 4H), 7.45 (d, J = 7.5 Hz, 1H), 7.15-7.07 (m, 6H), 6.95-6.90 (m, 4H). Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.65 ppm) according to ^{1}H NMR: (0.01/3)/[1+(0.01/3)] = 0.3%.

1,2,4-Tris(4-trifluoromethylphenyl)benzene (2t):

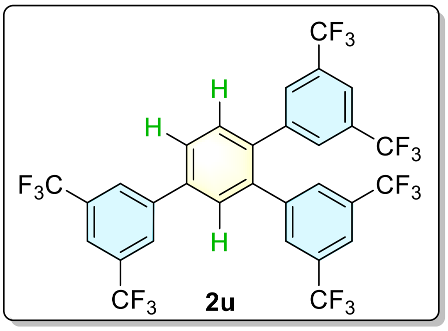


2t

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (33.2 mg, 33% yield). This compound is previously known.[2] The analytical data were in agreement with the literature, therefore the evaluation of ^{13}C

NMR was omitted. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78-7.72 (m, 5H), 7.66 (s, 1H), 7.56-7.52 (m, 5H), 7.31-7.27 (m, 4H). ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.48 (3F), -62.49 (6F). Amount of the 1,3,5-regioisomer according to GC-MS analysis: 26/8716 = 0.3%.

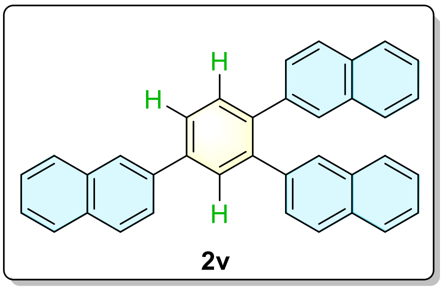
1,2,4-Tris(3,5-bis(trifluoromethyl)phenyl)benzene (2u):



2u

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (12.8 mg, 9% yield). This compound is previously known.[2] The analytical data were in agreement with the literature, therefore the evaluation of ^{13}C NMR was omitted. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (s, 2H), 7.95 (s, 1H), 7.84-7.79 (m, 3H), 7.74 (d, J = 2.0 Hz, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.58 (s, 2H), 7.55 (s, 2H). ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.84 (6F), -63.29 (6F), -63.33 (6F). No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

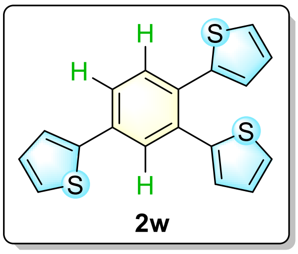
1,2,4-Tris(2-naphthyl)benzene (2v):



2v

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (66.0 mg, 72% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.96-7.85 (m, 8H), 7.79-7.70 (m, 5H), 7.58-7.48 (m, 4H), 7.46-7.41 (m, 4H), 7.23-7.19 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.2, 140.6, 139.7, 139.3, 138.9, 138.0, 133.8, 133.6, 133.5, 132.9, 132.3, 132.2, 131.9, 130.3, 128.7, 128.5, 128.5, 128.4, 128.2, 127.8, 127.8, 127.7, 127.5, 127.4, 126.8, 126.5, 126.2, 126.2, 126.1, 126.0, 126.0, 125.6. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

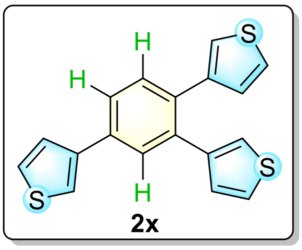
1,2,4-Tris(2-thienyl)benzene (2w):



2w

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow solid (41.4 mg, 64% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.72 (d, *J* = 2.0 Hz, 1H), 7.59 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.35 (dd, *J* = 3.5, 1.5 Hz, 1H), 7.28-7.23 (m, 3H), 7.08 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.98-6.93 (m, 3H), 6.88-6.87 (m, 1H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 143.3, 142.3, 142.3, 134.2, 134.0, 133.0, 131.5, 128.6, 128.3, 127.5, 127.1, 127.1, 126.3, 126.2, 125.4, 125.4, 123.7. Amount of the 1,3,5-regioisomer (characteristic NMR signal at δ = 7.88 ppm) according to ^{1}H NMR: (0.01/3)/[1+(0.01/3)] = 0.3%.

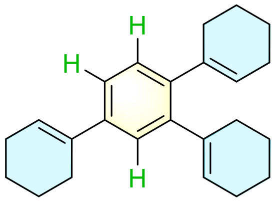
1,2,4-Tris(3-thienyl)benzene (2x):



2x

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (26.6 mg, 41% yield). This compound is previously known.[2] ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.67 (d, *J* = 2.0 Hz, 1H), 7.58 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.49-7.47 (m, 2H), 7.43-7.38 (m, 2H), 7.20-7.16 (m, 2H), 7.12-7.11 (m, 1H), 7.07-7.06 (m, 1H), 6.84-6.80 (m, 2H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 142.1, 141.7, 141.7, 135.9, 135.1, 134.3, 130.8, 129.1, 129.0, 128.4, 126.5, 126.4, 125.6, 125.0, 124.9, 123.1, 123.0, 120.7. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 86/28465 = 0.3%.

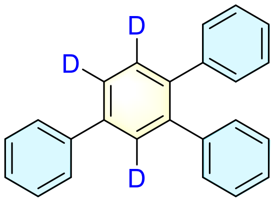
1,2,4-Tris(1-cyclohexenyl)benzene (2y):



2y

The title compound was prepared according to the general procedure **A** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (34.8 mg, 55% yield). This compound is previously known.[3] ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.20 (d, *J* = 7.8 Hz, 1H), 7.14 (s, 1H), 7.06 (d, *J* = 7.8 Hz, 1H), 6.12 (s, 1H), 5.67 (s, 2H), 2.42-2.39 (m, 2H), 2.22-2.18 (m, 6H), 2.15-2.12 (m, 4H), 1.78-1.74 (m, 2H), 1.71-1.63 (m, 10H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.6, 141.1, 140.8, 139.9, 139.2, 136.4, 128.6, 126.0, 125.9, 125.4, 124.4, 123.0, 29.7, 29.6, 27.5, 26.0, 25.9, 25.9, 23.4, 23.2, 22.4, 22.3, 22.3. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

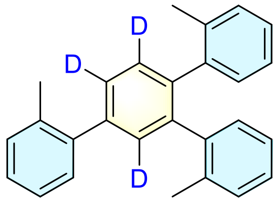
1,2,4-Triphenyl-3,5,6-trideuterobenzene (2a-d_3):



2a-d_3

The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (41.9 mg, 68% yield). This compound is previously known.[1] D-inc. determined by ^{1}H NMR: 3-D: 99%, 5-D: 97%, 6-D: 99%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.8 Hz, 1.99H), 7.66 (s, 0.03H), 7.45 (t, *J* = 7.8 z, 1.96H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.22-7.17 (m, 10H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 141.2, 141.0, 140.6, 140.3, 139.6, 130.0, 130.0, 129.0, 128.1, 128.1, 127.6, 127.3, 126.7, 126.7. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 29/6502 = 0.4%.

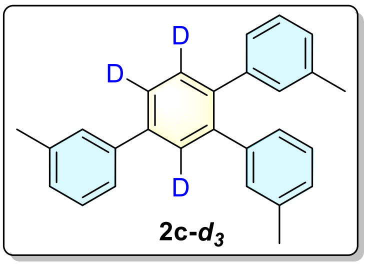
1,2,4-Tris(2-methylphenyl)-3,5,6-trideuterobenzene (2b-d_3):



2b-d_3

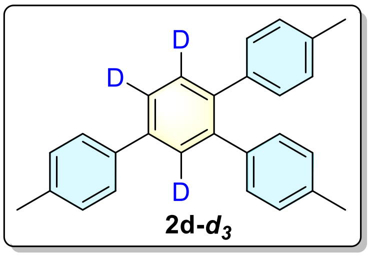
The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (30.4 mg, 43% yield). D-inc. determined by ^{1}H NMR: 3-D: 99%, 5-D: 99%, 6-D: 97%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35-7.32 (m, 1.03H), 7.30-7.23 (m, 3H), 7.09-6.90 (m, 8H), 2.38 (s, 3H), 2.19 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.7, 140.6, 140.4, 139.3, 135.9, 135.7, 135.6, 130.6, 130.1, 130.0, 127.4, 127.0, 126.0, 125.0, 20.8. HRMS (EI): m/z = calcd. for $(C_{27}H_{21}D_3)^{\bullet+}$: 351.2061, found 351.2072. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 30/1545 = 2%.

1,2,4-Tris(3-methylphenyl)-3,5,6-trideuterobenzene (2c-*d*₃):



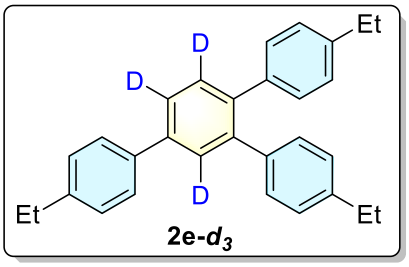
The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (51.2 mg, 73% yield). D-inc. determined by ^{1}H NMR: 3-D: 95%, 5-D: 95%, 6-D: 95%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.65 (s, 0.05H), 7.62 (s, 0.05H), 7.49 (d, J = 10.2 Hz, 2.05H), 7.35 (t, J = 7.2 Hz, 1H), 7.19 (d, J = 7.2 Hz, 1H), 7.10-7.01 (m, 6H), 6.94-6.91 (m, 2H), 2.43 (s, 3H), 2.28 (s, 3H), 2.27 (s, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 141.7, 141.4, 141.2, 140.9, 140.3, 139.7, 138.5, 137.6, 137.5, 130.8, 130.7, 128.9, 128.3, 128.1, 127.8, 127.8, 127.4, 127.3, 127.3, 127.2, 124.4, 21.7, 21.5. HRMS (EI): m/z = calcd. for $(C_{27}H_{21}D_3)^{\bullet+}$: 351.2061, found 351.2073. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

1,2,4-Tris(4-methylphenyl)-3,5,6-trideuterobenzene (2d-*d*₃):



The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellowish solid (36.4 mg, 52% yield). D-inc. determined by ^{1}H NMR: 3-D: 98%, 5-D: 96%, 6-D: 98%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.62-7.56 (m, 2.04H), 7.47 (s, 0.04H) 7.27-7.25 (m, 2H), 7.11-7.04 (m, 8H), 2.40 (s, 3H), 2.33 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 140.8, 140.0, 139.2, 138.8, 138.4, 137.8, 137.3, 136.3, 136.2, 129.9, 129.8, 129.7, 128.8, 128.8, 127.1, 21.3, 21.3. HRMS (EI): m/z = calcd. for $(C_{27}H_{21}D_3)^{\bullet+}$: 351.2061, found 351.2071. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 7/7265 = 0.1%.

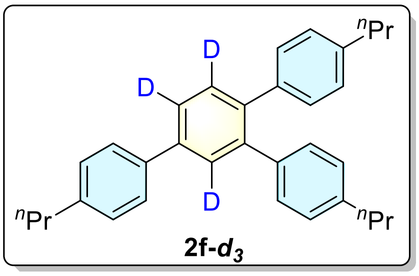
1,2,4-Tris(4-ethylphenyl)-3,5,6-trideuterobenzene (2e-*d*$_3$):



2e-*d*$_3$

The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (27.3 mg, 35% yield). D-inc. determined by ^{1}H NMR: 3-D: 97%, 5-D: 95%, 6-D: 95%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.64 (s, 0.05H), 7.60 (d, J = 7.8 Hz, 2.03H), 7.47 (s, 0.05H), 7.29 (d, J = 7.8 Hz, 2H), 7.13-7.05 (m, 8H), 2.72 (q, J = 7.2 Hz, 2H), 2.65-2.61 (m, 4H), 1.28 (t, J = 7.8 Hz, 3H), 1.24-1.21 (m, 6H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 143.6, 142.6, 142.5, 141.0, 140.1, 139.4, 139.2, 138.8, 138.3, 130.0, 129.9, 128.5, 127.5, 127.5, 127.2, 28.7, 28.6, 15.6, 15.5, 15.5. HRMS (EI): m/z = calcd. for $(C_{30}H_{27}D_3)^{\bullet+}$: 393.2530, found 393.2534. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

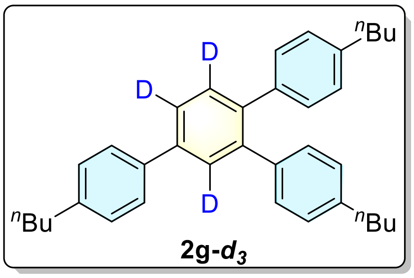
1,2,4-Tris(4-*n*-propylphenyl)-3,5,6-trideuterobenzene (2f-*d*$_3$):



2f-*d*$_3$

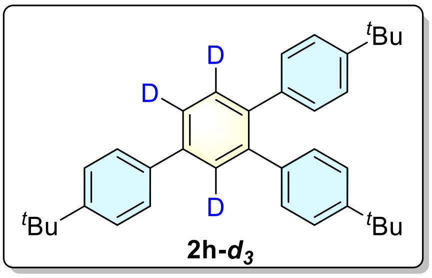
The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (55.6 mg, 64% yield). D-inc. determined by ^{1}H NMR: 3-D: 95%, 5-D: 94%, 6-D: 94%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.65 (s, 0.06H), 7.59 (d, J = 7.8 Hz, 2.05H), 7.48 (s, 0.06H), 7.26 (d, J = 7.8 Hz, 2H), 7.10-7.01 (m, 8H), 2.63 (t, J = 7.8 Hz, 2H), 2.56-2.53 (m, 4H), 1.71-1.59 (m, 6H), 0.98 (t, J = 7.2 Hz, 3H), 0.93-0.90 (m, 6H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 142.1, 141.1, 141.0, 140.9, 140.1, 139.4, 139.2, 138.8, 138.3, 129.9, 129.9, 129.1, 128.1, 128.1, 127.1, 37.9, 37.8, 24.6, 24.5, 14.0, 13.8. HRMS (EI): m/z = calcd. for $(C_{33}H_{33}D_3)^{\bullet+}$: 435.3000, found 435.3009. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

1,2,4-Tris(4-*n*-butylphenyl)-3,5,6-trideuterobenzene (2g-*d*$_3$)


2g-*d*$_3$

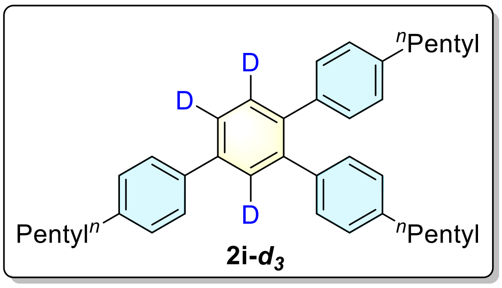
The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (57.8 mg, 60% yield). D-inc. determined by ^{1}H NMR: 3-D: 99%, 5-D: 96%, 6-D: 96%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.64 (s, 0.04H), 7.59 (d, *J* = 7.8 Hz, 2.01H), 7.48 (s, 0.04H), 7.27 (d, *J* = 7.8 Hz, 2H), 7.10-7.02 (m, 8H), 2.66 (t, *J* = 7.8 Hz, 2H), 2.59-2.56 (m, 4H), 1.67-1.57 (m, 6H), 1.42-1.31 (m, 6H), 0.96-0.91 (m, 9H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 142.3, 141.2, 141.2, 141.1, 140.1, 139.4, 139.2, 138.8, 138.2, 129.9, 129.9, 129.0, 128.1, 128.0, 127.1, 35.5, 35.4, 33.8, 33.6, 33.6, 22.6, 22.5, 22.5, 14.1. HRMS (EI): m/z = calcd. for $(C_{36}H_{39}D_3)^{\bullet+}$: 477.3469, found 477.3489. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 6/2847 = 0.2%.

1,2,4-Tris(4-tert-butylphenyl)-3,5,6-trideuterobenzene (2h-*d*$_3$):


2h-*d*$_3$

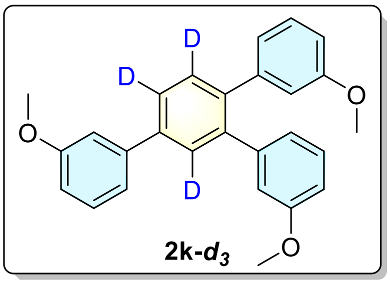
The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (23.1 mg, 24% yield). D-inc. determined by ^{1}H NMR: 3-D: 96%, 5-D: 95%, 6-D: 95%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.66 (s, 0.05H), 7.61 (d, *J* = 7.8 Hz, 2.04H), 7.48 (d, *J* = 8.4 Hz, 2.05H), 7.25-7.22 (m, 4H), 7.12-7.09 (m, 4H), 1.37 (s, 9H), 1.30 (s, 9H), 1.30 (s, 9H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 150.5, 149.6, 149.5, 141.0, 139.9, 139.4, 138.9, 138.5, 138.0, 129.7, 129.7, 126.9, 125.9, 124.8, 124.8, 34.7, 34.6, 31.6, 31.5. HRMS (EI): m/z = calcd. for $(C_{36}H_{39}D_3)^{\bullet+}$: 477.3469, found 477.3470. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 5/2655 = 0.2%.

1,2,4-Tris(4-*n*-pentylphenyl)-3,5,6-trideuterobenzene (2i-*d*$_3$):


2i-*d*$_3$

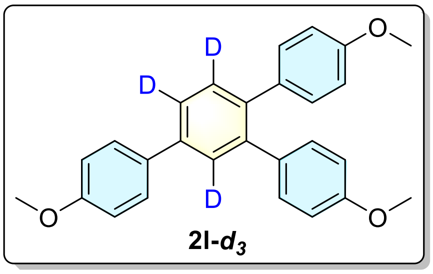
The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (33.5 mg, 32% yield). D-inc. determined by ^{1}H NMR: 3-D: 98%, 5-D: 95%, 6-D: 98%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.65-7.57 (m, 2.04H), 7.48 (s, 0.05H), 7.27 (d, J = 7.8 Hz, 2H), 7.10-7.02 (m, 8H), 2.65 (t, J = 7.8 Hz, 2H), 2.58-2.55 (m, 4H), 1.68-1.57 (m, 6H), 1.36-1.28 (m, 12H), 0.92-0.88 (m, 9H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 142.3, 141.3, 141.2, 141.1, 140.1, 139.4, 139.2, 138.8, 138.2, 129.9, 129.9, 129.0, 128.1, 128.0, 127.1, 35.8, 35.7, 31.8, 31.7, 31.6, 31.3, 31.1, 31.1, 22.7, 22.7, 14.2, 14.1. HRMS (EI): m/z = calcd. for $(C_{39}H_{45}D_3)^{\bullet +}$: 519.3939, found 519.3950. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

1,2,4-Tris(3-methoxyphenyl)-3,5,6-trideuterobenzene (2k-*d*$_3$):


2k-*d*$_3$

The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (97:3) to give a yellow oil (36.1 mg, 45% yield). D-inc. determined by ^{1}H NMR: 3-D: 96%, 5-D: 96%, 6-D: 96%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.68 (s, 0.04H), 7.64 (s, 0.04H), 7.52 (s, 0.04H), 7.38 (t, J = 7.8 Hz, 1H), 7.27 (d, J = 9.0 Hz, 1H), 7.20 (s, 1H), 7.18-7.15 (m, 2H), 6.93-6.91 (m, 1H), 6.83-6.71 (m, 6H), 3.87 (s, 3H), 3.64 (s, 3H), 3.63 (s, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 160.3, 159.5, 159.4, 143.1, 142.7, 142.3, 141.0, 140.4, 139.7, 130.0, 129.1, 129.1, 122.5, 122.5, 119.9, 115.5, 115.4, 113.2, 113.1, 113.1, 113.0, 55.5, 55.3, 55.3. HRMS (EI): m/z = calcd. for $(C_{27}H_{21}D_3O_3)^{\bullet +}$: 399.1908, found 399.1919. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

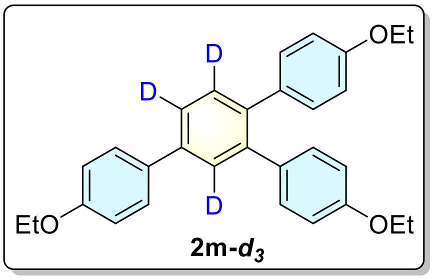
1,2,4-Tris(4-methoxyphenyl)-3,5,6-trideuterobenzene (2l-*d_3*):



2l-d_3

The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (97:3) to give a colorless oil (31.7 mg, 40% yield). D-inc. determined by ^{1}H NMR: 3-D: 96%, 5-D: 94%, 6-D: 96%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (d, J = 8.4 Hz, 2H), 7.56 (s, 0.08H), 7.44 (s, 0.06H), 7.13-7.09 (m, 4H), 6.99 (d, J = 8.4 Hz, 2H), 6.78 (t, J = 7.2 Hz, 4H), 3.85 (s, 3H), 3.79 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.5, 158.6, 158.5, 140.5, 139.6, 138.6, 134.4, 134.0, 133.4, 131.1, 131.0, 128.2, 114.5, 113.7, 113.6, 55.5, 55.4, 55.3. HRMS (EI): m/z = calcd. for $(C_{27}H_{21}D_3O_3)^{\bullet+}$: 399.1908, found 399.1915. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

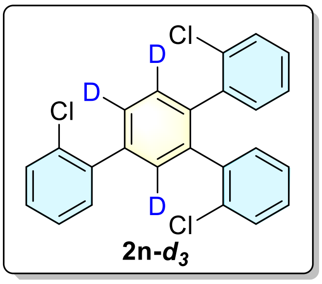
1,2,4-Tris(4-ethoxyphenyl)-3,5,6-trideuterobenzene (2m-*d_3*):



2m-d_3

The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (97:3) to give a yellowish solid (42.6 mg, 48% yield). D-inc. determined by ^{1}H NMR: 3-D: 90%, 5-D: 90%, 6-D: 90%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.59 (d, J = 8.4 Hz, 2H), 7.55 (s, 0.2H), 7.43 (s, 0.1H), 7.11-7.07 (m, 4H), 6.98 (d, J = 7.8 Hz, 2H), 6.78-6.75 (m, 4H), 4.09 (q, J = 6.6 Hz, 2H), 4.02-3.98 (m, 4H), 1.44 (t, J = 6.6 Hz, 3H), 1.41-1.39 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.8, 157.9, 157.9, 140.6, 139.6, 138.6, 134.3, 133.9, 133.3, 131.1, 131.0, 128.2, 115.1, 114.2, 114.2, 63.7, 63.6, 63.5, 15.0. HRMS (EI): m/z = calcd. for $(C_{30}H_{27}D_3O_3)^{\bullet+}$: 441.2378, found 441.2390. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

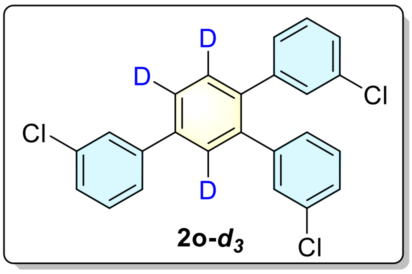
1,2,4-Tris(2-chlorophenyl)-3,5,6-trideuterobenzene (2n-*d₃*):



2n-*d*₃

The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (53.0 mg, 64% yield). D-inc. determined by ^{1}H NMR: 3-D: 98%, 5-D: 98%, 6-D: 98%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49-7.46 (m, 2.07H), 7.34-7.27 (m, 4H), 7.14-7.00 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 140.0, 139.7, 138.5, 138.4, 137.9, 133.4, 132.8, 131.7, 130.3, 129.3, 128.8, 128.6, 127.0, 126.2. HRMS (EI): m/z = calcd. for $(C_{24}H_{12}D_3Cl_3)^{\bullet+}$: 411.0422, found 411.0432. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 23/11744 = 0.2%.

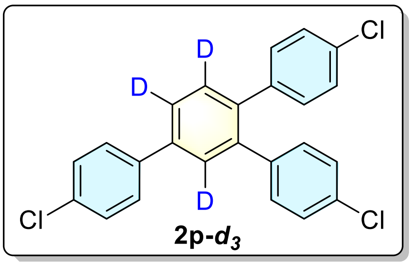
1,2,4-Tris(3-chlorophenyl)-3,5,6-trideuterobenzene (2o-*d₃*):



2o-*d*₃

The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow oil (52.6 mg, 64% yield). D-inc. determined by ^{1}H NMR: 3-D: 98%, 5-D: 96%, 6-D: 98%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.60 (s, 0.04H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.49 (s, 0.04H), 7.41-7.35 (m, 2H), 7.26-7.22 (m, 4H), 7.17-7.14 (m, 2H), 6.99-6.95 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.7, 142.4, 142.0, 139.8, 139.6, 138.7, 135.0, 134.2, 134.1, 130.3, 129.8, 129.7, 129.4, 129.4, 128.3, 128.3, 127.9, 127.4, 127.3, 127.2, 125.4. HRMS (EI): m/z = calcd. for $(C_{24}H_{12}D_3Cl_3)^{\bullet+}$: 411.0422, found 411.0431. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

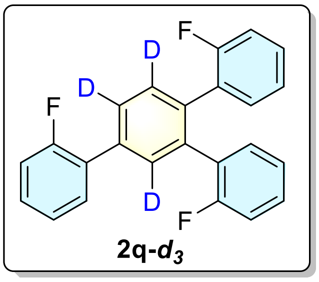
1,2,4-Tris(4-chlorophenyl)-3,5,6-trideuterobenzene (2p-*d₃*):



2p-*d*₃

The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (39.8 mg, 48% yield). D-inc. determined by ^{1}H NMR: 3-D: 98%, 5-D: 97%, 6-D: 98%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 (d, J = 8.4 Hz, 2.04H), 7.43 (d, J = 8.4 Hz, 2.03H), 7.24-7.21 (m, 4H), 7.10-7.06 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.8, 139.5, 139.5, 139.1, 138.7, 138.5, 133.9, 133.2, 133.1, 131.2, 131.2, 129.2, 128.5, 128.5, 128.4. HRMS (EI): m/z = calcd. for $(C_{24}H_{12}D_3Cl_3)^{\bullet+}$: 411.0422, found 411.0440. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

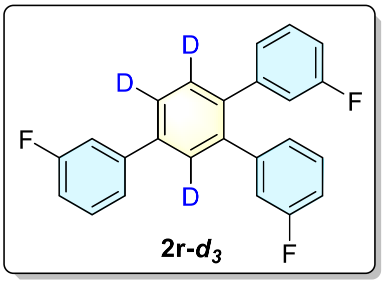
1,2,4-Tris(2-fluorophenyl)-3,5,6-trideuterobenzene (2q-*d₃*):



2q-*d*₃

The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (29.8 mg, 41% yield). D-inc. determined by ^{1}H NMR: 3-D: 96%, 5-D: 99%, 6-D: 96%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (s, 0.04H), 7.64 (s, 0.04H), 7.54 (t, J = 7.8 Hz, 1.01H), 7.36-7.32 (m, 1H), 7.25-7.14 (m, 6H), 7.03-7.01 (m, 2H), 6.97-6.92 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.8(d, J = 247.6 Hz), 160.5 (d, J = 246.1Hz), 160.4 (d, J = 246.1Hz), 135.8, 135.4, 135.0, 132.0 (d, J = 3.2 Hz), 131.9 (d, J = 3.2 Hz), 130.9 (d, J = 3.3 Hz), 129.4 (d, J = 8.3 Hz), 129.2 (d, J = 3.9 Hz), 129.2 (d, J = 3.8 Hz), 128.7, 128.6, 128.5, 128.4, 128.4, 128.3, 124.6 (d, J = 3.6 Hz), 123.7 (t, J = 3.3 Hz), 116.4, 116.3, 115.6 (d, J = 2.0 Hz), 115.4 (d, J = 2.6 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ -115.23 (2F), -117.66 (1F). HRMS (EI): m/z = calcd. for $(C_{24}H_{12}D_3F_3)^{\bullet+}$: 363.1309, found 363.1325. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 18/3869 = 0.5%.

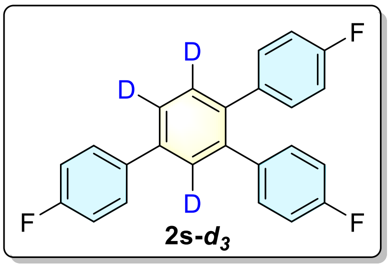
1,2,4-Tris(3-fluorophenyl)-3,5,6-trideuterobenzene (2r-*d₃*):



2r-*d*₃

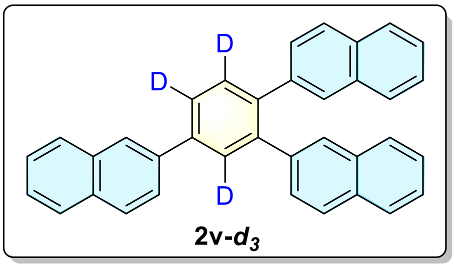
The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a colorless oil (24.0 mg, 33% yield). D-inc. determined by ^{1}H NMR: 3-D: 94%, 5-D: 94%, 6-D: 95%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (s, 0.05H), 7.62 (s, 0.06H), 7.50 (s, 0.06H), 7.46-7.41 (m, 2H), 7.37 (d, J = 10.8 Hz, 1H), 7.24-7.19 (m, 2H), 7.09-7.06 (m, 1H), 6.96-6.88 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.5 (d, J = 246.6 Hz), 163.7 (d, J = 245.7 Hz), 143.4 (d, J = 8.2 Hz), 143.0 (d, J = 7.9 Hz), 142.7 (d, J = 7.7 Hz), 140.1, 139.7, 139.0, 130.6 (d, J = 8.4 Hz), 129.7(t, J = 7.6 Hz), 125.8 (d, J = 2.8 Hz), 125.7 (d, J = 2.9 Hz), 122.9 (d, J = 3.0 Hz), 116.9 (d, J = 8.4 Hz), 116.8 (d, J = 8.4 Hz), 114.7, 114.6, 114.3, 114.2, 114.1 (d, J = 3.2 Hz), 114.0, 113.9. ^{19}F NMR (471 MHz, CDCl$_3$) δ -112.78 (1F), -113.32 (1F), -113.40 (1F). HRMS (EI): m/z = calcd. for $(C_{24}H_{12}D_3F_3)^{\bullet+}$: 363.1309, found 363.1320. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 36/1068 = 3%.

1,2,4-Tris(4-fluorophenyl)-3,5,6-trideuterobenzene (2s-*d$_3$*):



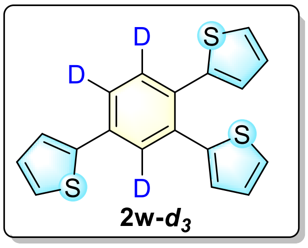
The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (32.8 mg, 45% yield). D-inc. determined by ^{1}H NMR: 3-D: 97%, 5-D: 96%, 6-D: 97%. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.62-7.56 (m, 2.06H), 7.46 (s, 0.04H), 7.16-7.09 (m, 6H), 6.96-6.92 (m, 4H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 163.6 (d, J = 246.7 Hz), 162.8 (d, J = 246.7 Hz), 162.8 (d, J = 246.3 Hz), 140.1, 139.6, 138.5, 137.2 (d, J = 3.5 Hz), 136.9 (d, J = 3.3 Hz), 136.5 (d, J = 3.3 Hz), 131.5 (d, J = 6.0 Hz), 131.5 (d, J = 5.9 Hz), 128.8 (d, J = 8.2 Hz), 116.0, 115.9, 115.3 (d, J = 4.8 Hz), 115.1 (d, J = 4.7 Hz). ^{19}F NMR (471 MHz, CDCl$_3$) δ -115.13 (1F), -115.60 (1F), -115.72 (1F). HRMS (EI): m/z = calcd. for $(C_{24}H_{12}D_3F_3)^{\bullet+}$: 363.1309, found 363.1322. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 29/6040 = 0.5%.

1,2,4-Tris(2-naphthyl)-3,5,6-trideuterobenzene (2v-*d$_3$*):



The title compound was prepared according to the general procedure **B** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (28.0 mg, 30% yield). D-inc. determined by ^{1}H NMR: 3-D: 94%, 5-D: 94%, 6-D: 94%. ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.17 (s, 1H), 7.96-7.91 (m, 3.06H), 7.89-7.85 (m, 3.06H), 7.78-7.73 (m, 4H), 7.70 (s, 0.06H), 7.56 (t, J = 8.0 Hz, 2H), 7.53-7.47 (m, 2H), 7.46-7.40 (m, 4H), 7.24-7.19 (m, 2H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 141.0, 140.3, 139.6, 139.2, 138.8, 137.8, 133.8, 133.5, 133.5, 132.8, 132.2, 132.2, 128.6, 128.5, 128.4, 128.4, 128.4, 128.3, 128.0, 127.7, 127.6, 127.6, 127.3, 127.3, 126.4, 126.0, 126.0, 126.0, 125.9, 125.8, 125.5. HRMS (EI): m/z = calcd. for $(C_{36}H_{21}D_3)^{\bullet+}$: 459.2061, found 459.2076. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

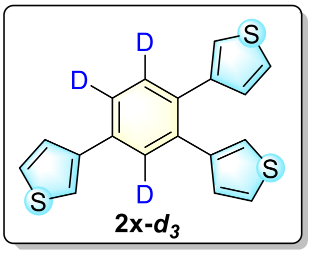
1,2,4-Tris(2-thienyl)-3,5,6-trideuterobenzene (2w-*d3*):



2w-*d*3

The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a yellow solid (37.1 mg, 57% yield). D-inc. determined by ^{1}H NMR: 3-D: 90%, 5-D: 90%, 6-D: 90%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (s, 0.10H), 7.60 (s, 0.10H), 7.52 (s, 0.10H), 7.37 (d, *J* = 3.6 Hz, 1H), 7.31 (d, *J* = 4.8 Hz, 2H), 7.27 (d, *J* = 4.8 Hz, 1H), 7.09 (t, *J* = 4.2 Hz, 1H), 6.99 (t, *J* = 4.2 Hz, 1H), 6.96-6.95 (m, 2H), 6.90 (d, *J* = 3.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.4, 142.4, 142.4, 134.3, 134.0, 133.1, 131.4, 128.7, 128.3, 127.5, 127.2, 127.1, 126.3, 126.2, 125.5, 123.8. HRMS (EI): m/z = calcd. for $(C_{18}H_9D_3S_3)^{\bullet+}$: 327.0284, found 327.0284. No significant signal was detected, amount of the 1,3,5-regioisomer <1% was assumed.

1,2,4-Tris(3-thienyl)-3,5,6-trideuterobenzene (2x-*d3*):



2x-*d*3

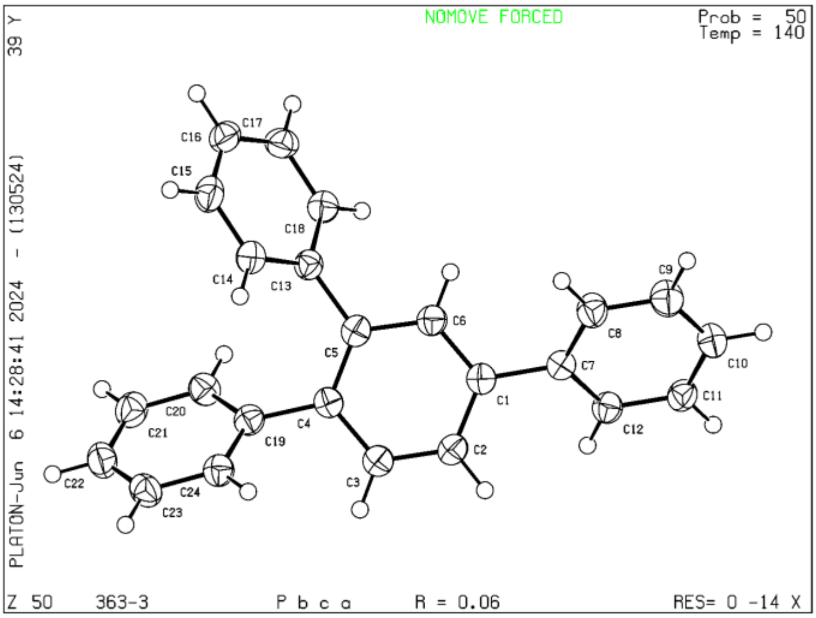
The title compound was prepared according to the general procedure **C** and purified by column chromatography on silica gel eluting with petroleum ether to give a white solid (30.5 mg, 47% yield). D-inc. determined by ^{1}H NMR: 3-D: 93%, 5-D: 96%, 6-D: 92%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (s, 0.08H), 7.59 (s, 0.07H), 7.51 (s, 1.04H), 7.44-7.41 (m, 2H), 7.22-7.19 (m, 2H), 7.14 (s, 1H), 7.09 (s, 1H), 6.84 (dd, *J* = 16.2, 4.8 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.2, 141.9, 141.8, 136.0, 135.1, 134.4, 129.1, 129.0, 126.5, 126.5, 125.0, 124.9, 123.2, 123.0, 120.7. HRMS (EI): m/z = calcd. for $(C_{18}H_9D_3S_3)^{\bullet+}$: 327.0284, found 327.0290. Amount of the 1,3,5-regioisomer according to GC-MS analysis: 13/2498 = 0.5%.

5. X-ray structure of 2a-*d*$_3$

The electron of hydrogen and deuterium is identical. Therefore, X-ray is unable to differentiate between hydrogen and deuterium.

Table S15 Crystal data and structure refinement for 2a-*d*$_3$.

Identification code	2a-*d*$_3$
Empirical formula	$C_{24}H_{18}$
Formula weight	306.38
Temperature/K	142(50)
Crystal system	orthorhombic
Space group	Pbca
a/Å	10.3109(6)
b/Å	17.7703(11)
c/Å	18.3580(9)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3363.7(3)
Z	8
ρ_{calc}g/cm^3	1.210
μ/mm^{-1}	0.516
F(000)	1296.0
Crystal size/mm^3	0.15 × 0.12 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.636 to 146.688
Index ranges	$-12 \leq h \leq 12$, $-21 \leq k \leq 19$, $-8 \leq l \leq 22$
Reflections collected	10988
Independent reflections	3185 [R_{int} = 0.0518, R_{sigma} = 0.0535]
Data/restraints/parameters	3185/0/218
Goodness-of-fit on F^2	1.127
Final R indexes [I>=2σ (I)]	R_1 = 0.0562, wR_2 = 0.1621
Final R indexes [all data]	R_1 = 0.0687, wR_2 = 0.1777
Largest diff. peak/hole / e Å^{-3}	0.22/-0.37



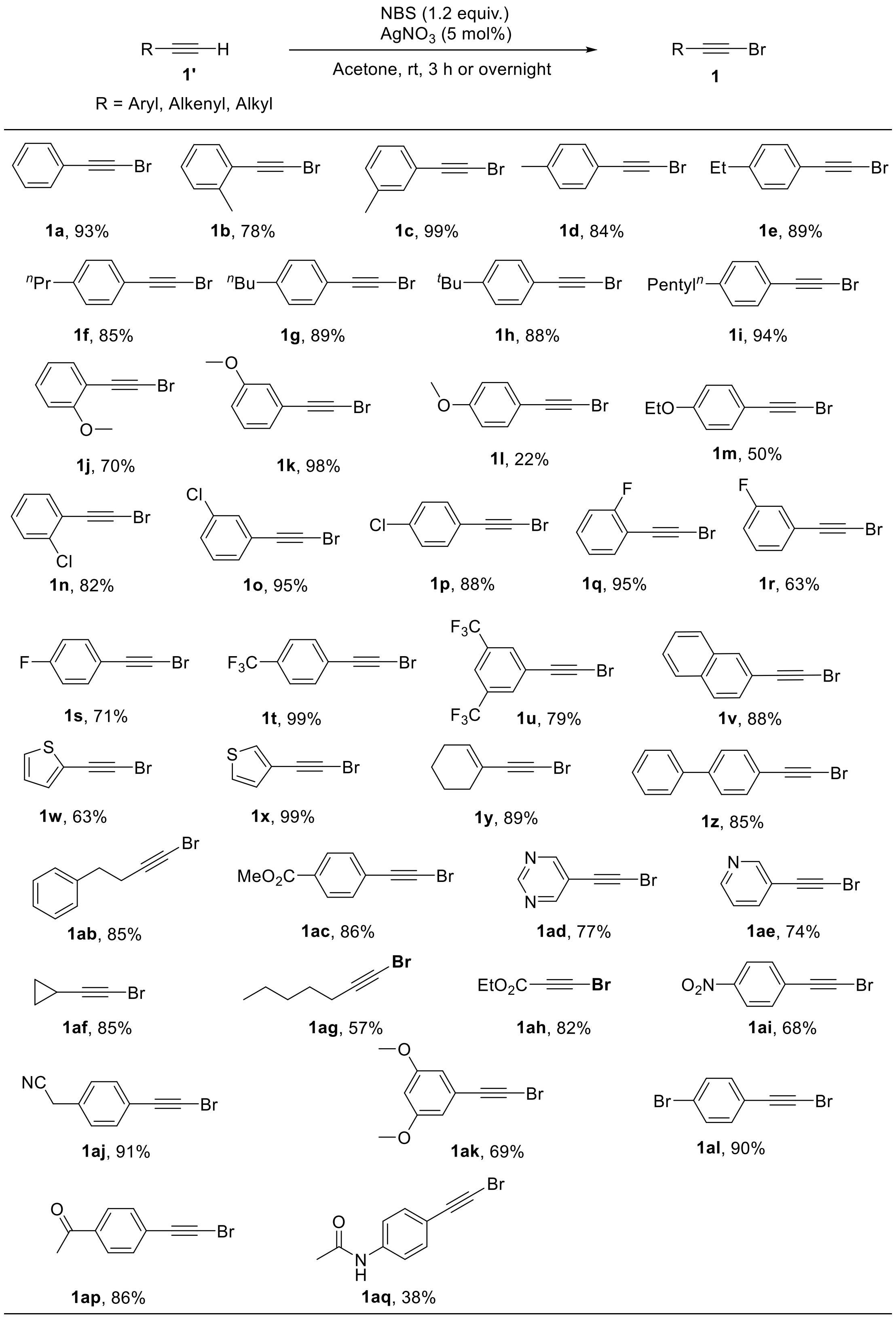
```
Bond precision:    C-C = 0.0030 A           Wavelength=1.54184

Cell:               a=10.3109(6)       b=17.7703(11)       c=18.3580(9)
                    alpha=90           beta=90             gamma=90
Temperature:        142 K

                  Calculated                    Reported
Volume            3363.7(3)                     3363.7(3)
Space group       P b c a                       P b c a
Hall group        -P 2ac 2ab                    -P 2ac 2ab
Moiety formula    C24 H18                       C24 H18
Sum formula       C24 H18                       C24 H18
Mr                306.38                        306.38
Dx,g cm-3         1.210                         1.210
Z                 8                             8
Mu (mm-1)         0.516                         0.516
F000              1296.0                        1296.0
F000'             1299.32
h,k,lmax          12,22,22                      12,21,22
Nref              3381                          3185
Tmin,Tmax         0.928,0.950                   0.217,1.000
Tmin'             0.926

Correction method= # Reported T Limits: Tmin=0.217 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.942           Theta(max)= 73.344

                                                      wR2(reflections)=
R(reflections)= 0.0562( 2560)                         0.1777( 3185)

S = 1.127                    Npar= 218
```

CCDC 2361191 which contains the crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK, which can be obtained free of charge via Fax: +44 (0)1223 336033; E-Mail: deposit@ccdc.cam.ac.uk, https://www.ccdc.cam.ac.uk/structures/.

Crystal structure determination of 2a-*d*$_3$

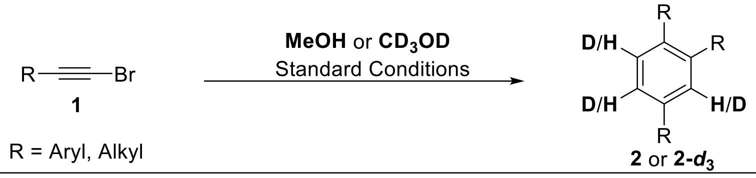
Crystal Data for $C_{24}H_{18}$ (M = 306.38 g/mol): orthorhombic, space group Pbca (no. 61), a = 10.3109 (6) Å, b = 17.7703 (11) Å, c = 18.3580 (9) Å, V = 3363.7 (3) Å^3, Z = 8, T = 142 (50) K, μ (Cu Kα) = 0.516 mm^{-1}, $Dcalc$ = 1.210 g/cm^3, 10988 reflections measured (9.636° ≤ 2Θ ≤ 146.688°), 3185 unique (R_{int} = 0.0518, R_{sigma} = 0.0535) which were used in all calculations. The final R_1 was 0.0562 (I > 2σ(I)) and wR_2 was 0.1777 (all data).

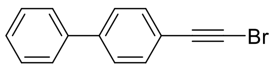
6. Preparation of substrates 1a-1al



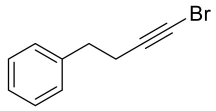
Add *N*-bromosuccinimide (1.28 g, 7.2 mmol) and $AgNO_3$ (51 mg, 0.3 mmol) to a solution of the alkyne derivative (6 mmol) in acetone (30 mL) at room temperature with magnetic stirring for 3 hours or overnight. After the completion of the reaction, filter the mixture. Purify the residue by column chromatography by using ethyl acetate and petroleum ether as eluents. All substrates mentioned are known and prepared according to the literatures.[5]

7. Unsuccessful substrates

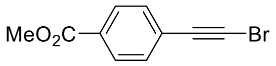




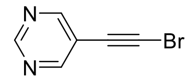
1z, ND



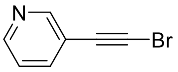
1ab, ND



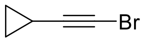
1ac, ND



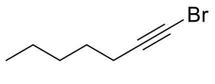
1ad, ND



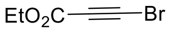
1ae, ND



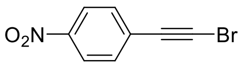
1af, ND



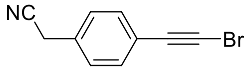
1ag, ND



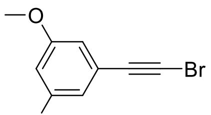
1ah, ND



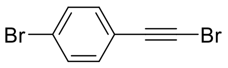
1ai, ND



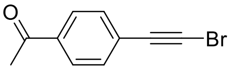
1aj, ND



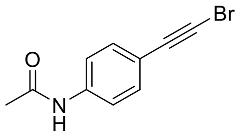
1ak, ND



1al, ND



1ap, ND

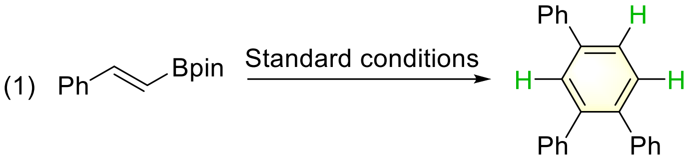


1aq, ND

All of the above compounds were performed under the standard reaction conditions respectively. The corresponding desired products were not detected by the TLC.

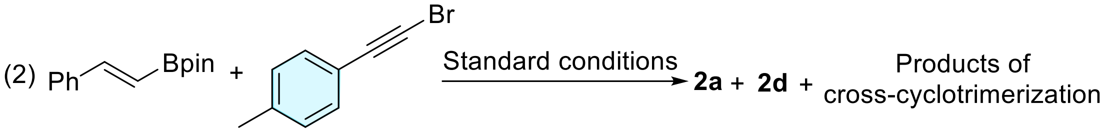
8. Mechanistic investigations and scale-up synthesis

A) Mechanistic investigations:



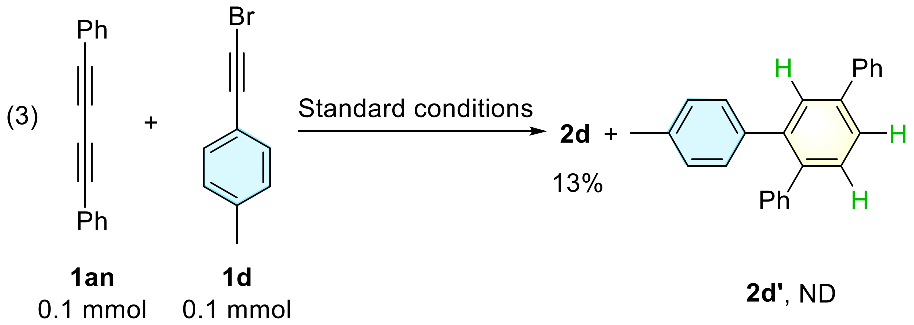
1am, 0.1 mmol **2a**, ND

(*E*)-4,4,5,5-Tetramethyl-2-styryl-1,3,2-dioxaborolane **1am** (23.0 mg, 0.1 mmol), B_2pin_2 (25.4 mg, 1.0 equiv.), Zn (13.1 mg, 2.0 equiv.), $FeCl_2$ (1.3 mg, 10 mol%), KO^tBu (9.0 mg, 0.8 equiv.), THF (0.5 mL, 0.2 M) and MeOH (3.2 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under argon atmosphere. Upon completion of the reaction, the desired product 1,2,4-triphenylbenzene **2a** was not detected.

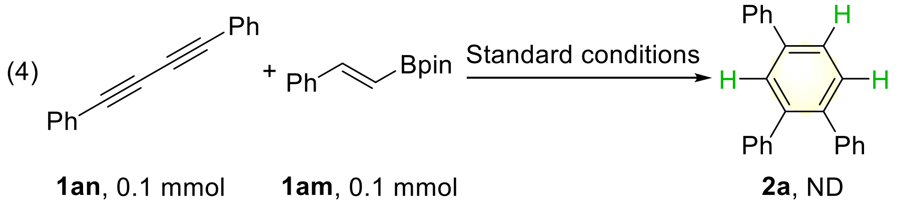


1am, 0.1 mmol **1d**, 0.2 mmol ND 44% ND

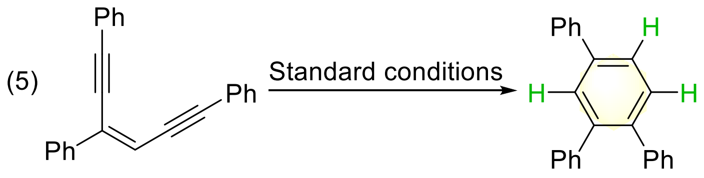
(*E*)-4,4,5,5-Tetramethyl-2-styryl-1,3,2-dioxaborolane **1am** (23.0 mg, 0.1 mmol), 1-(2-bromoethynyl)-4-methylbenzene **1d** (39.0 mg, 0.2 mmol), B_2pin_2 (50.8 mg, 1.0 equiv.), Zn (26.2 mg, 2.0 equiv.), $FeCl_2$ (2.5 mg, 10 mol%), KO^tBu (18.0 mg, 0.8 equiv.), THF (1.0 mL, 0.2 M) and MeOH (3.2 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford **2d** in 44% yield (15.2 mg). The **2a** and the cross-cyclotrimerization products were not detected. The results obtained from experiments (1) and (2) indicated that **1am** might not be the intermediate for this iron-catalyzed reductive cyclotrimerization reaction.



1,4-Diphenyl-1,3-butadiyne **1an** (20.2 mg, 0.1 mmol), 1-(2-bromoethynyl)-4-methylbenzene **1d** (19.5 mg, 0.1 mmol), B$_2$pin$_2$ (25.4 mg, 1.0 equiv.), Zn (13.1 mg, 2.0 equiv.), FeCl$_2$ (1.3 mg, 10 mol%), KO^{t}Bu (9.0 mg, 0.8 equiv.), THF (0.5 mL, 0.2 M) and MeOH (3.2 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford **2d** in 13% yield (4.6 mg). However, the conceived cross-cyclotrimerization product **2d'** was not detected.



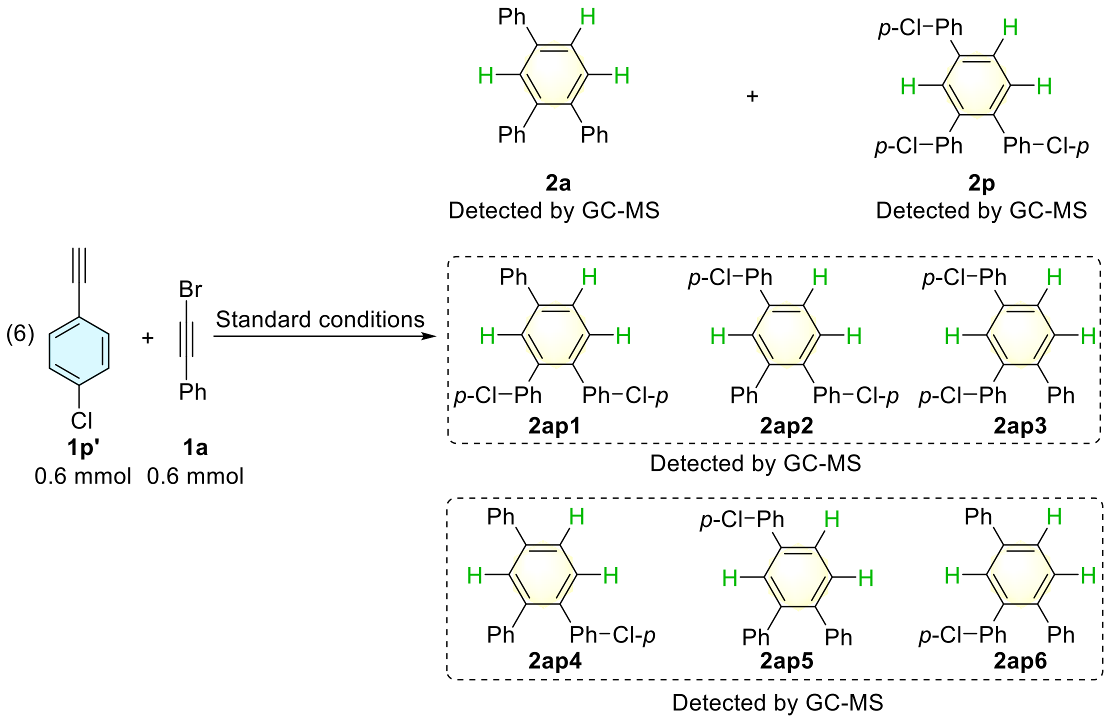
1,4-Diphenyl-1,3-butadiyne **1an** (20.2 mg, 0.1 mmol), (*E*)-4,4,5,5-tetramethyl-2-styryl-1,3,2-dioxaborolane **1am** (23.0 mg, 0.1 mmol), B$_2$pin$_2$ (25.4 mg, 1.0 equiv.), Zn (13.1 mg, 2.0 equiv.), FeCl$_2$ (1.3 mg, 10 mol%), KO^{t}Bu (9.0 mg, 0.8 equiv.), THF (0.5 mL, 0.2 M) and MeOH (3.2 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the **2a** was not detected. Experiments (3) and (4) showed that **1am** and **1an** might not be the intermediates in this iron-catalyzed reductive cyclotrimerization reaction.



1ao, 0.2 mmol

2a, ND

(*Z*)-Hexa-3-en-1,5-diyne-1,3,6-triyltribenzene [6] **1ao** (60.9 mg, 0.2 mmol), B$_2$pin$_2$ (50.8 mg, 1.0 equiv.), Zn (26.2 mg, 2.0 equiv.), FeCl$_2$ (2.5 mg, 10 mol%), KO^{t}Bu (18.0 mg, 0.8 equiv.), THF (1.0 mL, 0.2 M) and MeOH (6.4 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the **2a** was not detected. Experiment (5) showed that **1ao** might not be the intermediate in this iron-catalyzed reductive cyclotrimerization reaction.



4-Chlorophenylacetylene **1p'** (152.4 mg, 0.6 mmol), (bromoethynyl)benzene **1a** (108.6 mg, 0.6 mmol), B$_2$pin$_2$ (152.4 mg, 1.0 equiv.), Zn (78.5 mg, 2.0 equiv.), FeCl$_2$ (7.6 mg, 10 mol%), KO^{t}Bu (53.9 mg, 0.8 equiv.), THF (3 mL, 0.2 M) and MeOH (19.2 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Some of the reaction mixture was taken and analyzed by GC-MS, it showed that the **2a** (found RT:

14.95 min, m/z: 306, **Figure S1**) and **2p** (found RT: 22.04 min, m/z: 408, **Figure S2**) were formed under the standard reaction conditions. According to the GC-MS spectra of the reaction mixture of Experiment (6), the cross-cyclotrimerization products, one of **2ap1-2ap3** (found RT: 18.81 min, m/z: 374, **Figure S3**) and two of **2ap4-2ap6** (found RT: 16.40 min, m/z: 340; 16.59 min, m/z: 340; **Figure S4**), were also formed under the standard reaction conditions. Experiment (6) showed that terminal alkyne **1p'** might be an intermediate in this iron-catalyzed reductive cyclotrimerization reaction.

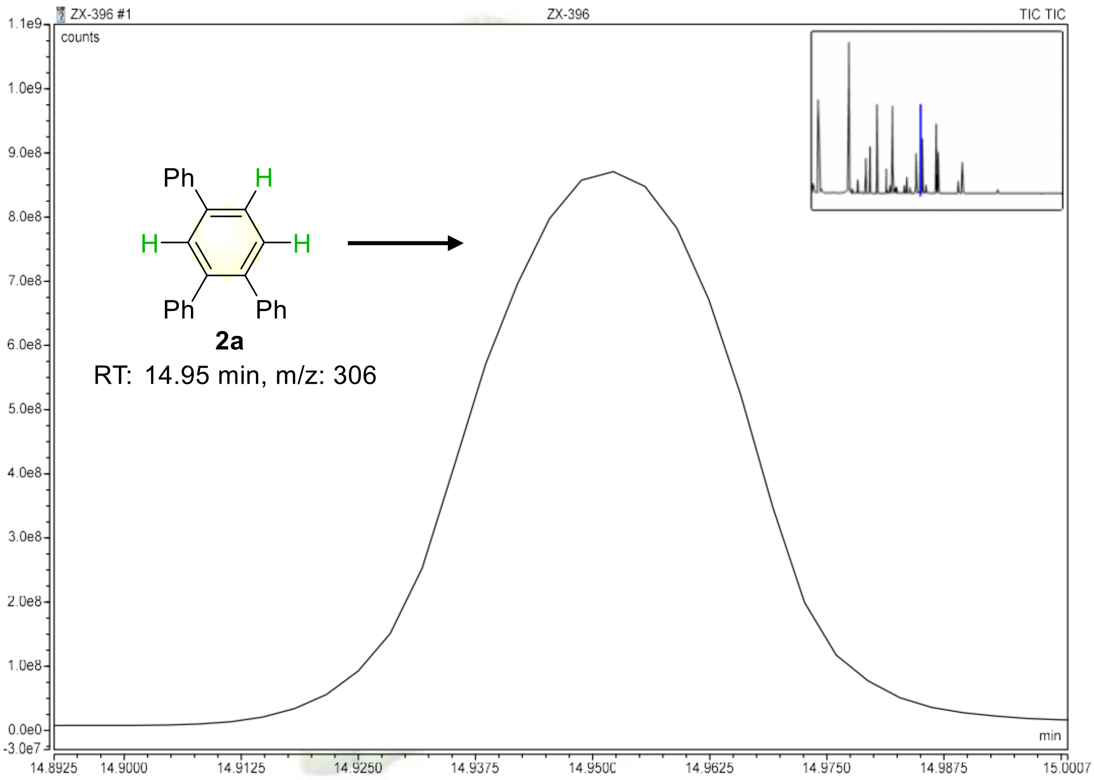


Figure S1. GC-MS spectra of the reaction mixture of experiment (6) (**2a** was found)

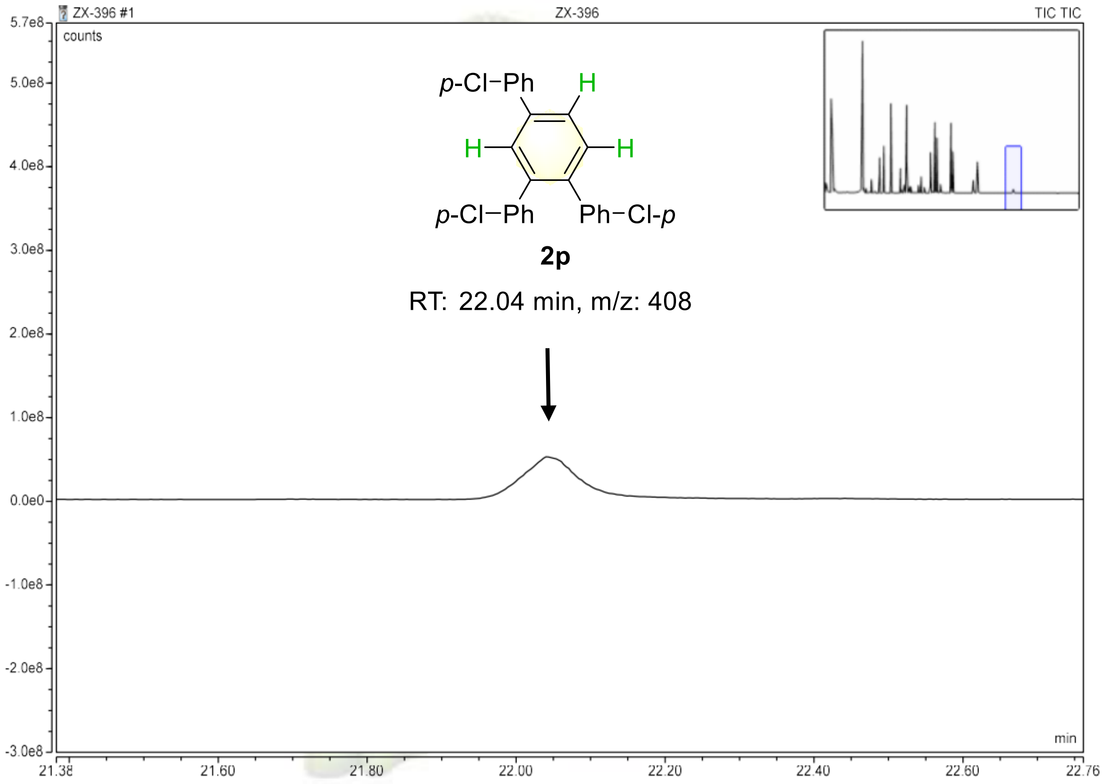


Figure S2. GC-MS spectra of the reaction mixture of experiment (6) (**2p** was found)

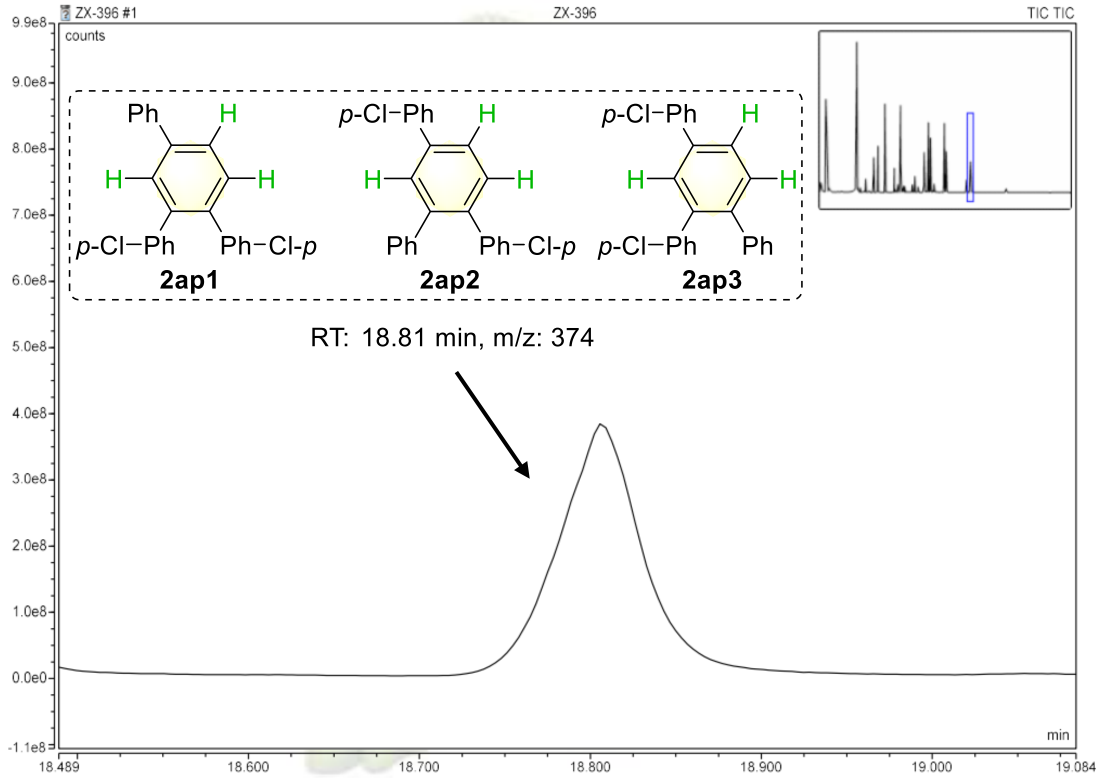


Figure S3. GC-MS spectra of the reaction mixture of experiment (6) (one of **2ap1-2ap3** was found)

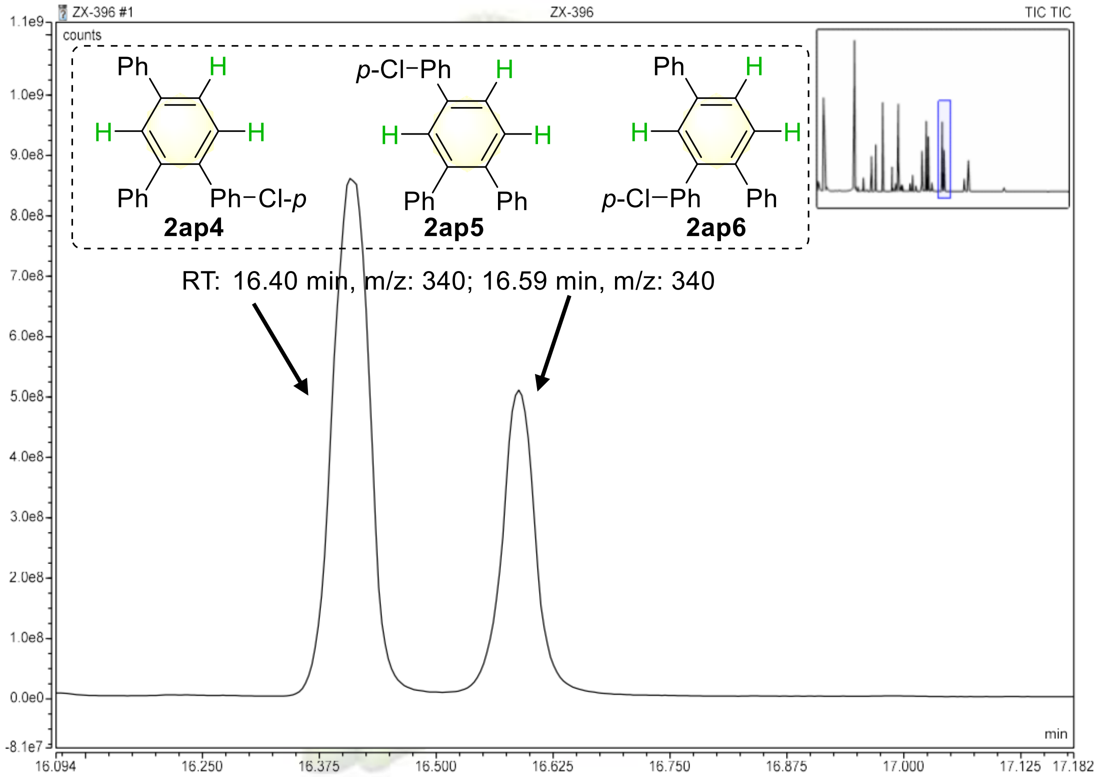
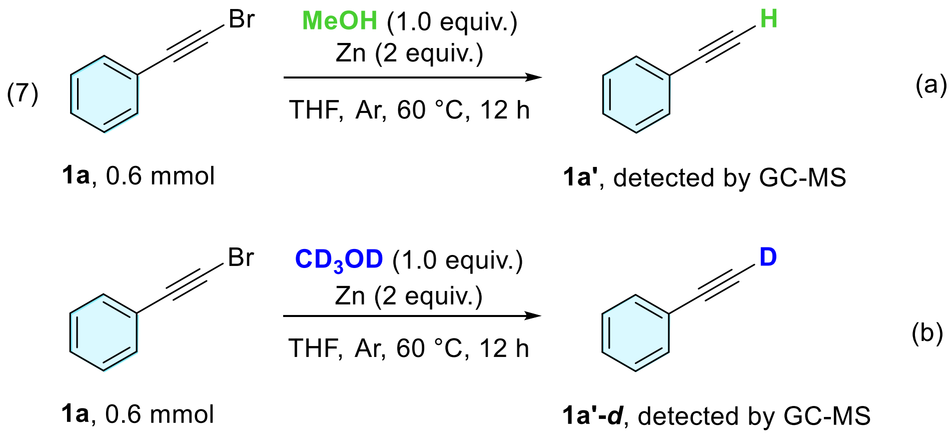


Figure S4. GC-MS spectra of the reaction mixture of experiment (6) (two of **2ap4**-**2ap6** were found)



(Bromoethynyl)benzene **1a** (108.6 mg, 0.6 mmol), Zn (78.5 mg, 2.0 equiv.), THF (3 mL, 0.2 M) and MeOH (19.2 mg, 1.0 equiv.) [or CD_3OD (21.6 mg, 1.0 equiv.)] were added in sequence to a flame-dried 25 mL Schlenk tube respectively. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Some of the reaction mixtures of experiments (7a and 7b) were taken and analyzed by GC-MS, phenylacetylene **1a'** (found RT: 2.56 min, m/z: 102, **Figure S5-S6**) or terminal deuterium-substituted phenylacetylene **1a'-*d*** (found RT: 6.06 min, m/z: 103, **Figure S7**-**S8**) were successfully formed. Experiment (7) indicated that (bromoethynyl)benzene can be reductive to phenylacetylene/terminal deuterium-substituted phenylacetylene in the presence of zinc powder and methanol or methanol-d_4.

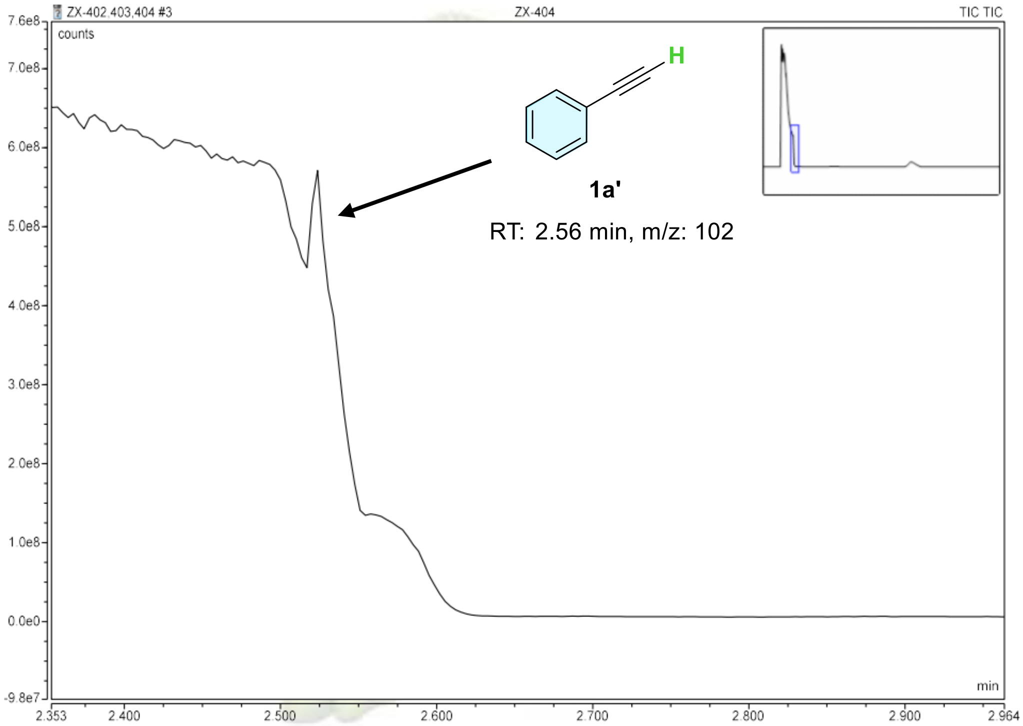


Figure S5. GC-MS chromatogram of the reaction mixture of experiment (7a) (**1a'** was found)

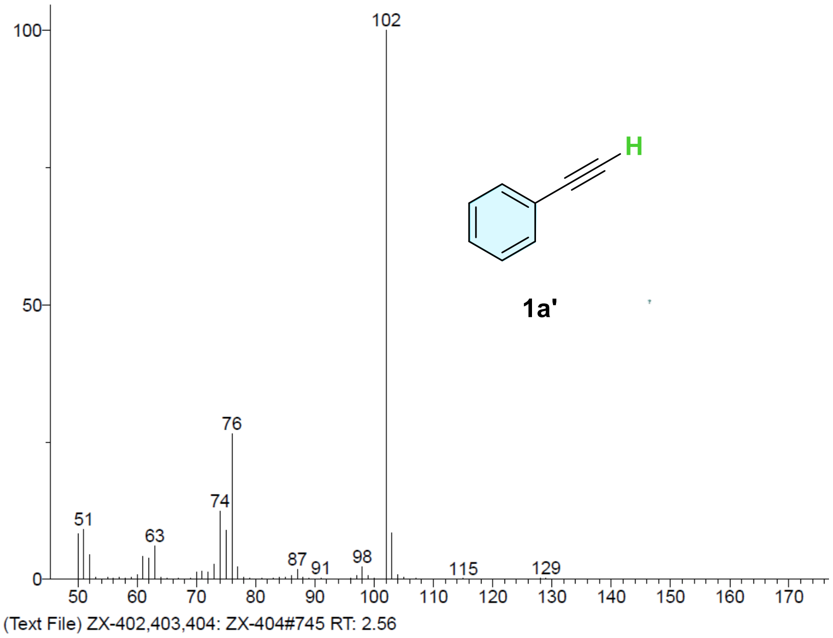


Figure S6. MS spectra of the reaction mixture of experiment (7a) (**1a'** was found)

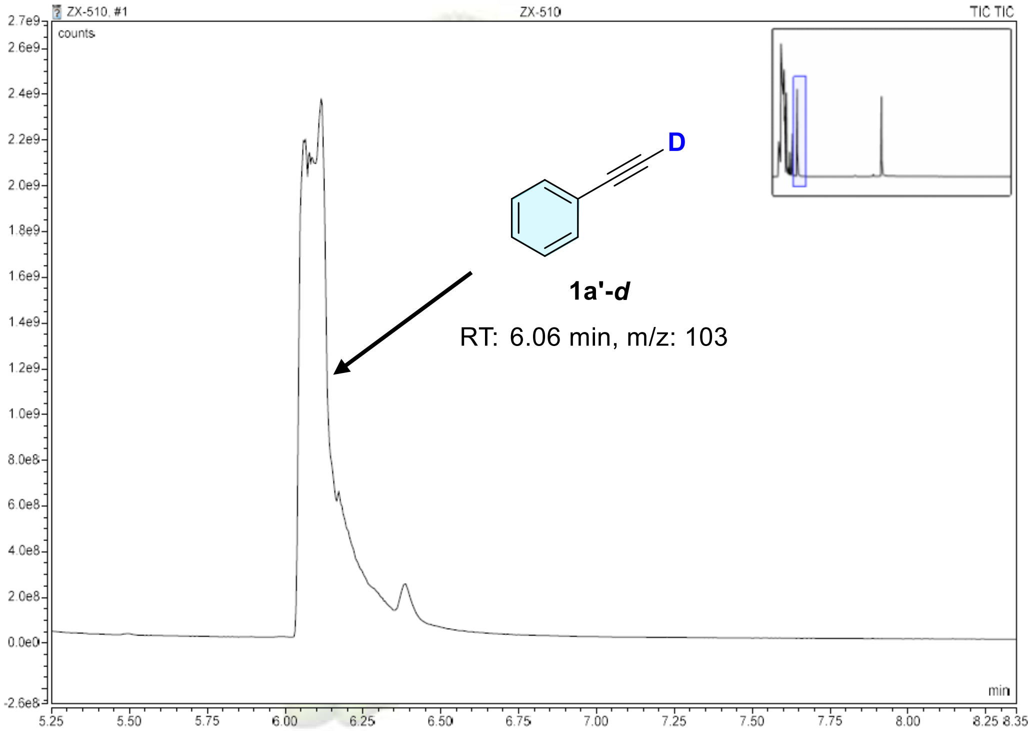


Figure S7. GC-MS chromatogram of the reaction mixture of experiment (7b) (**1a'** was found)

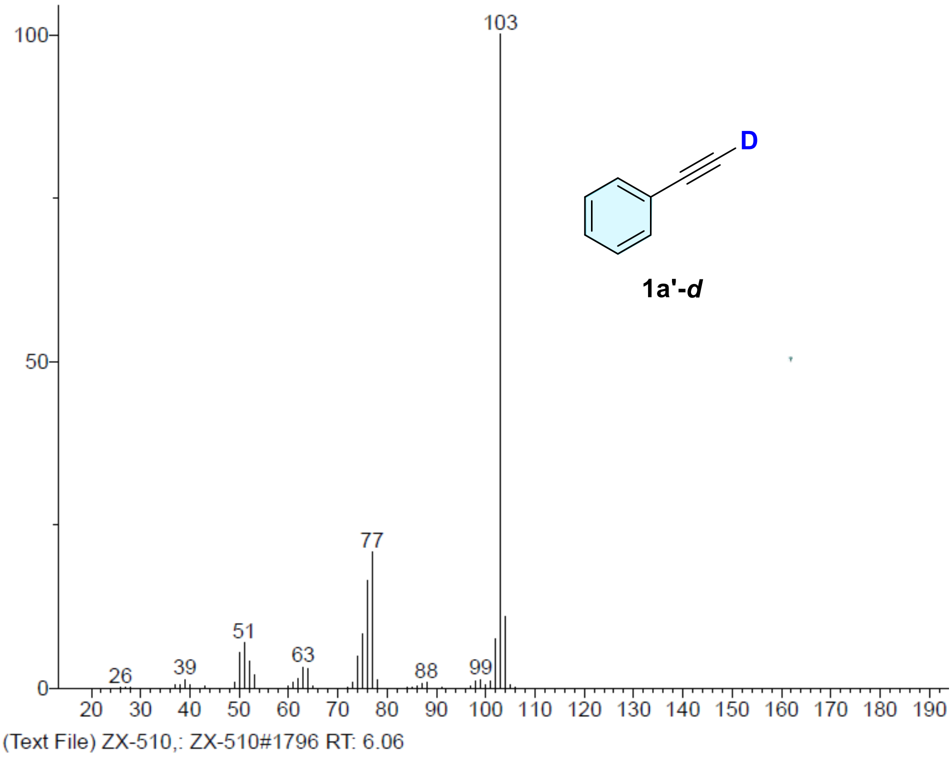
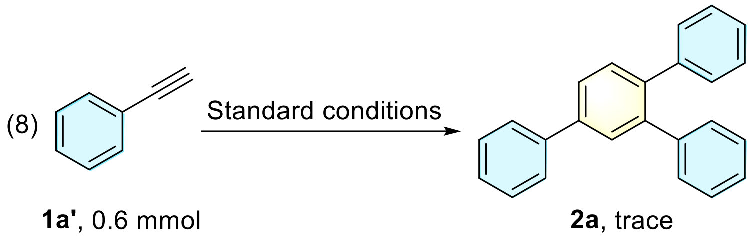
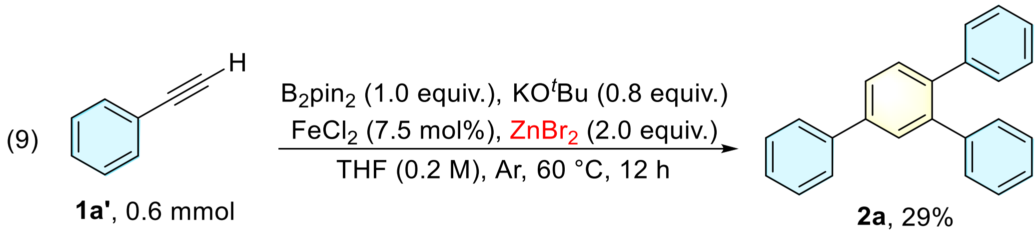


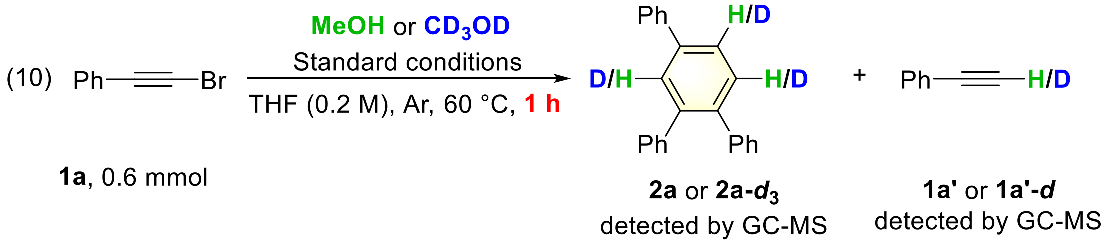
Figure S8. MS spectra of the reaction mixture of experiment (7b) (**1a'-*d*** was found)



Phenylacetylene **1a'** (61.3 mg, 0.6 mmol), B_2pin_2 (152.4 mg, 1.0 equiv.), Zn (78.5 mg, 2.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), KO^tBu (53.9 mg, 0.8 equiv.) and THF (3 mL, 0.2 M) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the reaction was detected by TLC and only trace amount of **2a** was generated under the standard reaction conditions.



Phenylacetylene **1a'** (61.3 mg, 0.6 mmol), B_2pin_2 (152.4 mg, 1.0 equiv.), $ZnBr_2$ (270.3 mg, 2.0 equiv.), $FeCl_2$ (7.6 mg, 10 mol%), KO^tBu (53.9 mg, 0.8 equiv.) and THF (3 mL, 0.2 M) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford **2d** in 29% yield (17.8 mg).



(Bromoethynyl)benzene **1a** (108.6 mg, 0.6 mmol), B_2pin_2 (152.4 mg, 1.0 equiv.), Zn

(78.5 mg, 2.0 equiv.), FeCl$_2$ (7.6 mg, 10 mol%) or (5.7 mg, 7.5 mol%), KO^{t}Bu (53.9 mg, 0.8 equiv.), THF (3 mL, 0.2 M) and **MeOH** (19.2 mg, 1.0 equiv.) [or **CD$_3$OD** (32.5 mg, 1.5 equiv.)] were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 1 hour under the argon atmosphere. Some of the reaction mixtures of experiment (**10-MeOH** or **10-CD$_3$OD**) were taken and analyzed by GC-MS, phenylacetylene **1a'** (found RT: 6.06 min, m/z: 102, **Figure S9** and **S10**) and terminal deuterium-substituted phenylacetylene **1a'-*d*** (found RT: 6.05 min, m/z: 103, **Figure S13** and **S14**) were successfully formed. Meanwhile, small amount of 1,2,4-triphenylbenzene **2a** (found RT: 26.7 min, m/z: 306, **Figure S11** and **S12**) and 1,2,4-triphenyl-3,5,6-trideuterobenzene **2a-*d*$_3$** (found RT: 26.71 min, m/z: 309, **Figure S15** and **S16**) were detected by GC-MS.

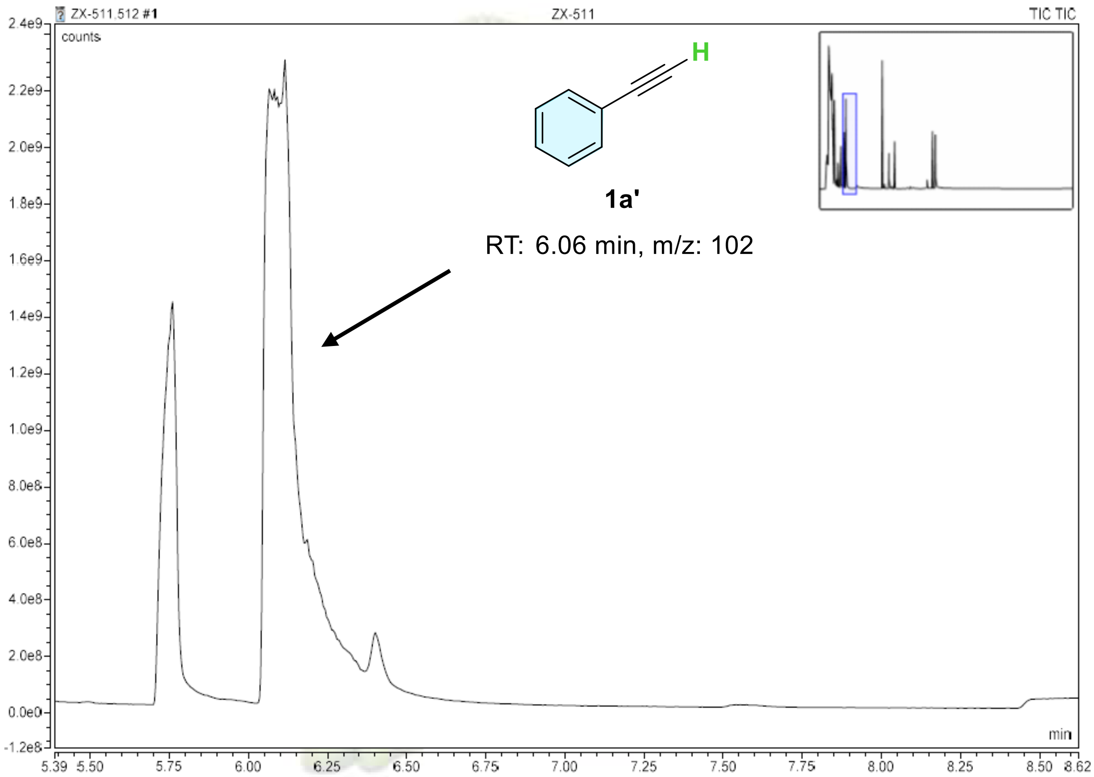


Figure S9. GC-MS chromatogram of the reaction mixture of experiment (**10-MeOH**) (**1a'** was found)

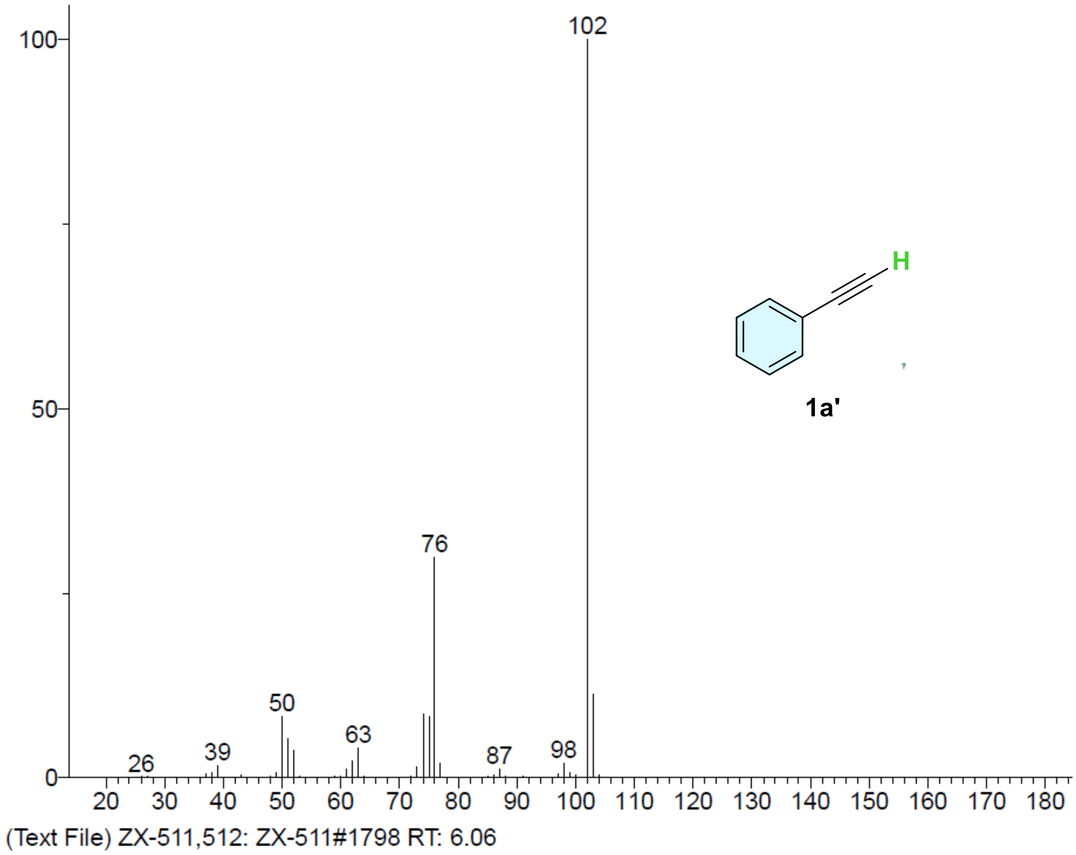


Figure S10. MS spectra of the reaction mixture of experiment (**10-MeOH**) (**1a'** was found)

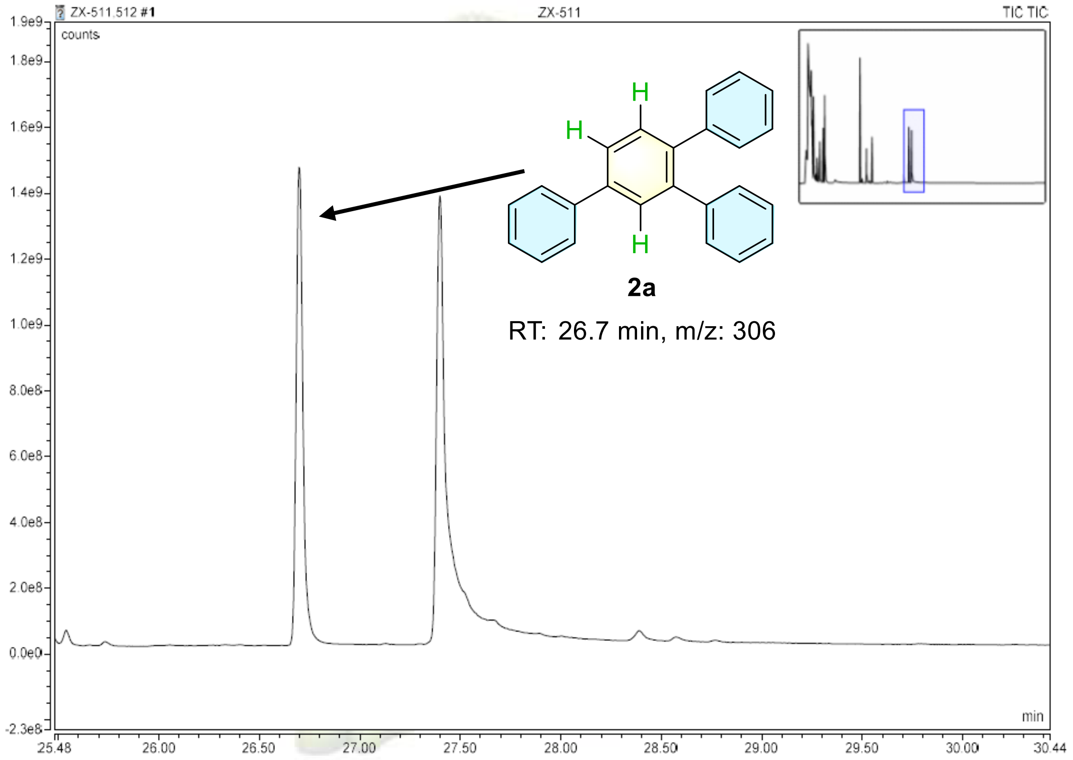


Figure S11. GC-MS chromatogram of the reaction mixture of experiment (**10-MeOH**) (**2a** was found)

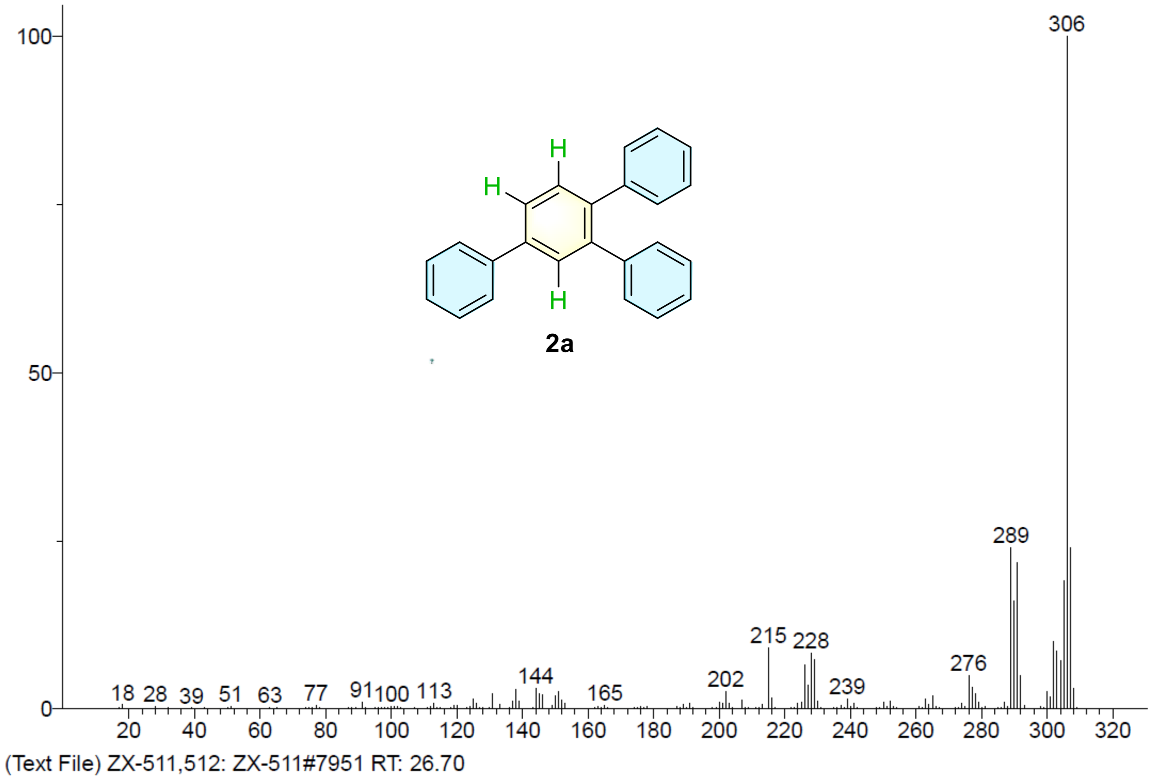


Figure S12. MS spectra of the reaction mixture of experiment (**10-MeOH**) (**2a** was found)

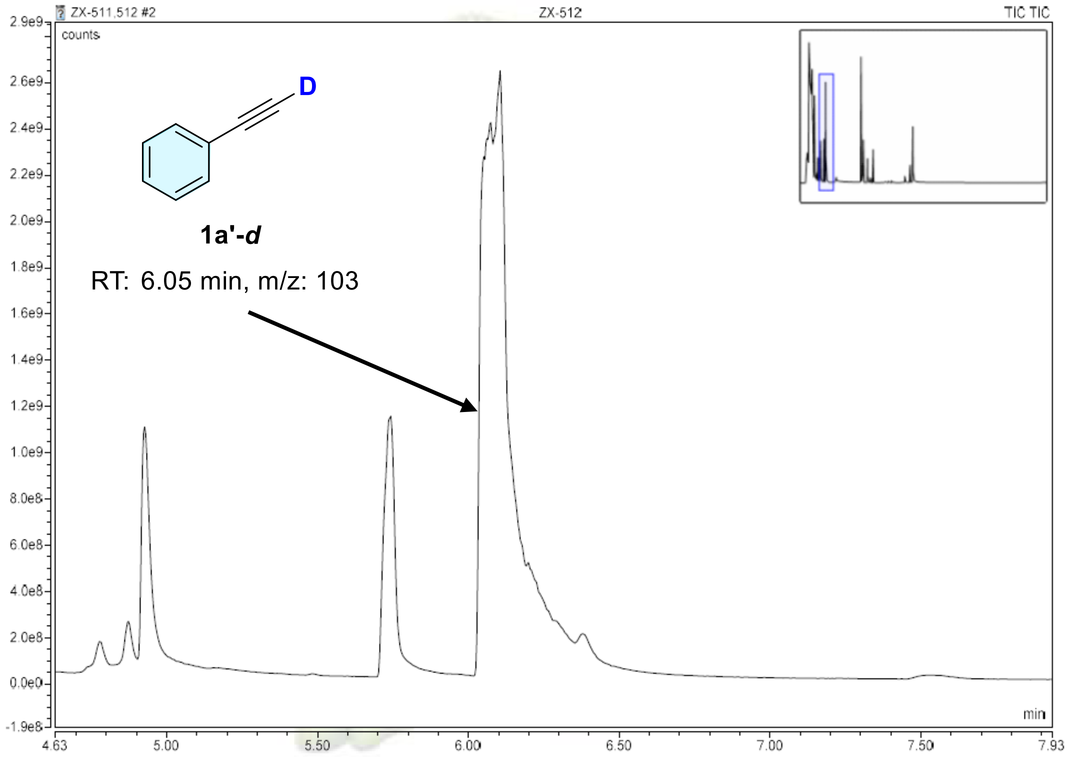


Figure S13. GC-MS Chromatogram of the reaction mixture of experiment (**10-CD$_3$OD**) (**1a’-*d*** was found)

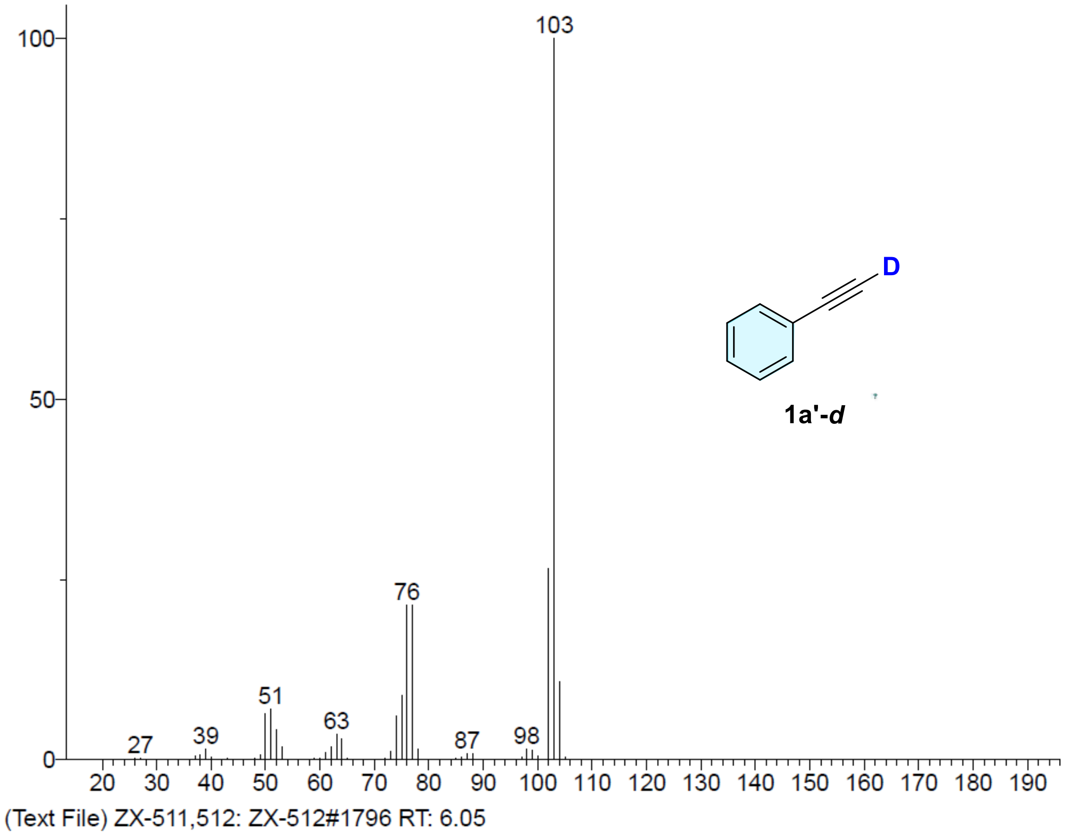


Figure S14. MS spectra of the reaction mixture of experiment (**10-CD$_3$OD**) (**1a'-*d*** was found)

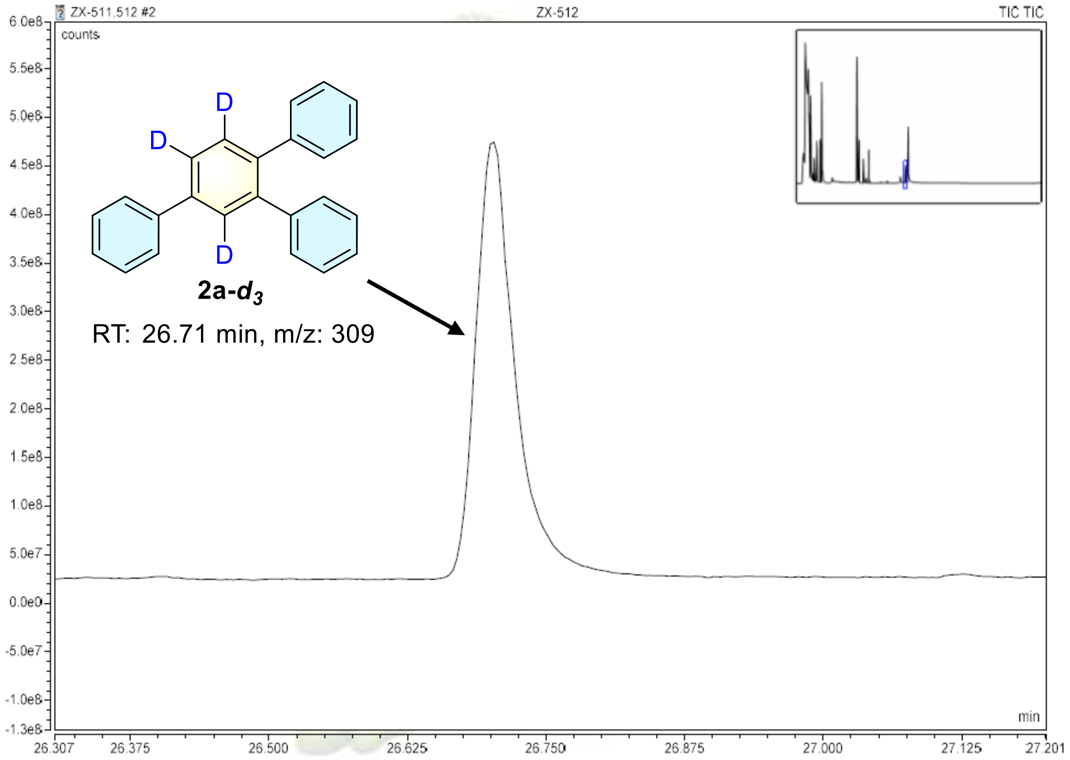
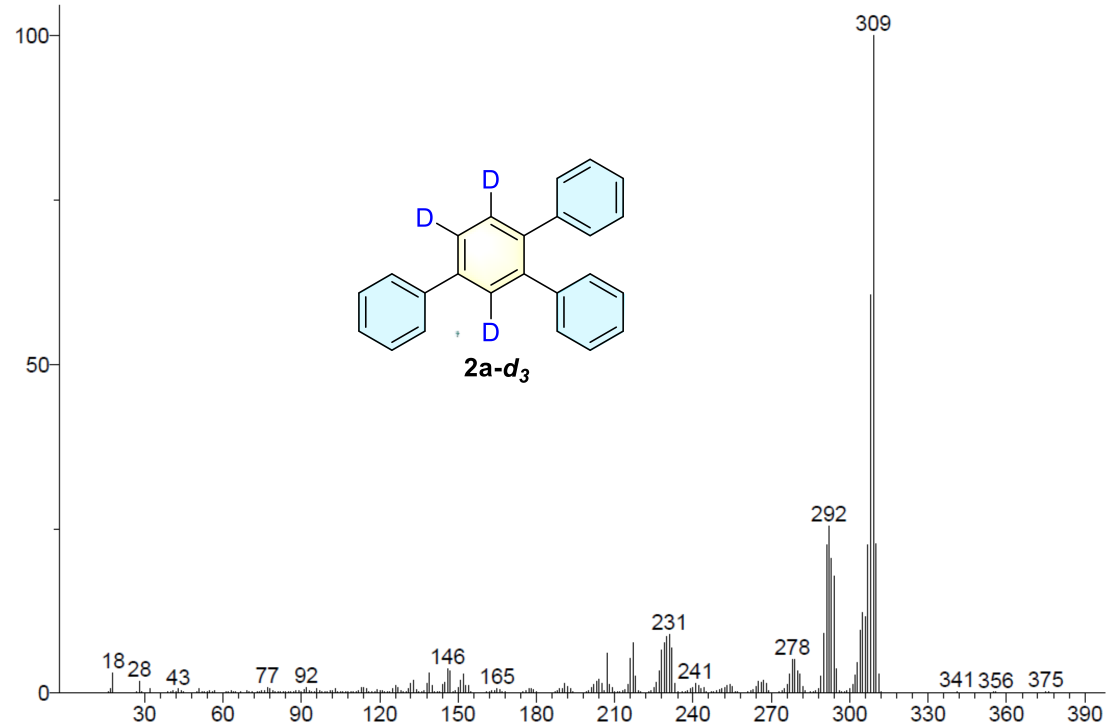


Figure S15. GC-MS Chromatogram of the reaction mixture of experiment (**10-CD$_3$OD**) (**2a-*d*$_3$** was found)

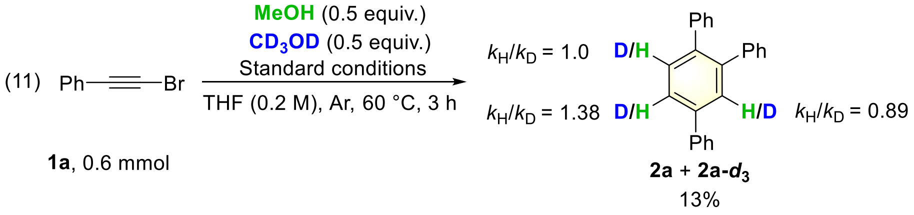


(Text File) ZX-511,512: ZX-512#7954 RT: 26.71

Figure S16. MS spectra of the reaction mixture of experiment (**10-CD$_3$OD**) (**2a-** ***d*$_3$** was found)

Experiments (6), (7), (8), (9) and (10) indicated that the terminal alkyne might be the intermediate in this reductive cyclotrimerization reaction.

KIE experiment:



(Bromoethynyl)benzene **1a** (108.6 mg, 0.6 mmol), B$_2$pin$_2$ (152.4 mg, 1.0 equiv.), Zn (78.5 mg, 2.0 equiv.), FeCl$_2$ (7.6 mg, 10 mol%) or (5.7 mg, 7.5 mol%), KO^{t}Bu (53.9 mg, 0.8 equiv.), THF (3 mL, 0.2 M), MeOH (9.6 mg, 0.5 equiv.) and CD$_3$OD (16.3 mg, 0.5 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 3 hours under the argon atmosphere. The competition reaction products **2a**+**2a-*d*$_3$** were isolated as a mixture within 13% yield at a low conversion and revealed little kinetic isotope effects (C$_3$: $k_H/k_D = 0.89$, C$_5$: $k_H/k_D = 1.38$, C$_6$: $k_H/k_D = 1.0$), indicating that the formation of C(sp)-H bond might be not involved in the rate-limiting step of this transformation (**Figure S17**). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.69-7.67 (m, 2.5H), 7.66-7.65 (m, 0.47H), 7.52-7.51 (m, 0.58H), 7.46 (t, J = 7.5 Hz, 2H), 7.37 (t, J = 7.0 Hz, 1H),7.26-7.17 (m, 10H).

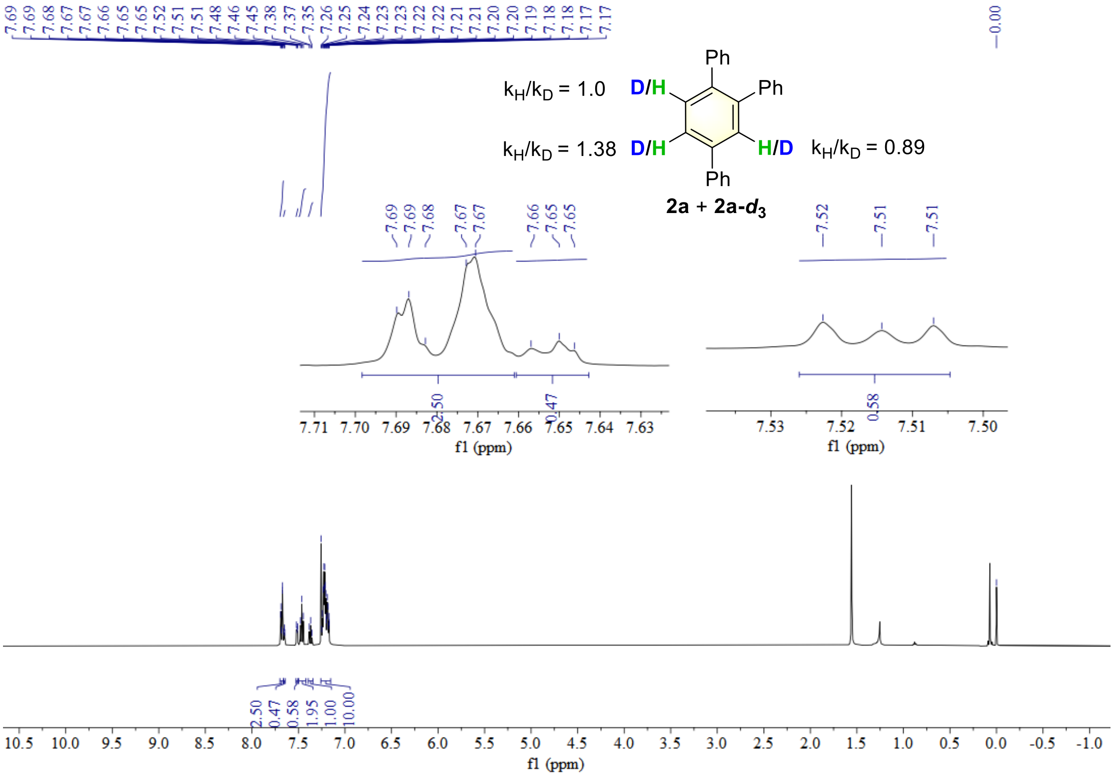
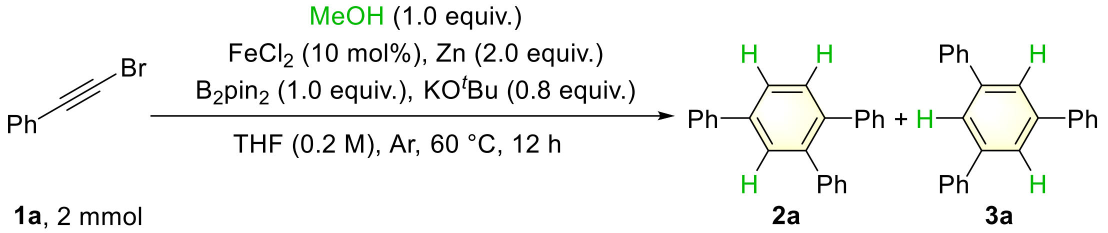


Figure S17. ^{1}H NMR spectra of **2a**+**2a-d_3**

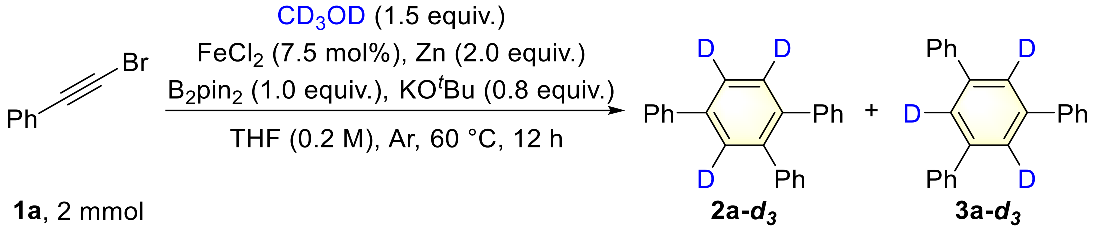
B) Scale-up synthesis:

Scale-up synthesis of 2a



(Bromoethynyl)benzene **1a** (362 mg, 2 mmol), B_2pin_2 (507.9 mg, 1.0 equiv.), Zn (261.6 mg, 2.0 equiv.), $FeCl_2$ (25.3 mg, 10 mol%), KO*t*Bu (179.5 mg, 0.8 equiv.), THF (10 mL, 0.2 M) and MeOH (64 mg, 1.0 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford **2a** and **3a** in 50% yield (101.2 mg). Amount of the 1,3,5-regioisomer = 1%.

Scale-up synthesis of 2a-*d*3



(Bromoethynyl)benzene **1a** (362 mg, 2 mmol), B_2pin_2 (507.9 mg, 1.0 equiv.), Zn (261.6 mg, 2.0 equiv.), $FeCl_2$ (18.9 mg, 7.5 mol%), KO*t*Bu (179.5 mg, 0.8 equiv.), THF (10 mL, 0.2 M) and CD_3OD (108 mg, 1.5 equiv.) were added in sequence to a flame-dried 25 mL Schlenk tube in the glove box. The mixture was stirred at 60 ºC for 12 hours under the argon atmosphere. Upon completion of the reaction, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel and eluted with petroleum ether to afford **2a-*d*3** and **3a-*d*3** in 43% yield (89 mg). Amount of the 1,3,5-regioisomer = 1%. D-inc. determined by ^1H NMR: 3-D: 99%, 5-D: 97%, 6-D: 99%.

9. References

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[2] K.-L. Liang, L.-J. Lu, X. Liu, D.-L. Yang, S.-C. Wang, Y.-M. Gao, H. Alhumade, H. Yi, and A.-W. Lei, Electrochemical Cobalt-catalyzed Cyclotrimerization of Alkynes to 1,2,4-Substituted Arenes, *ACS Catal.*, 2021, **11**, 14892−14897.

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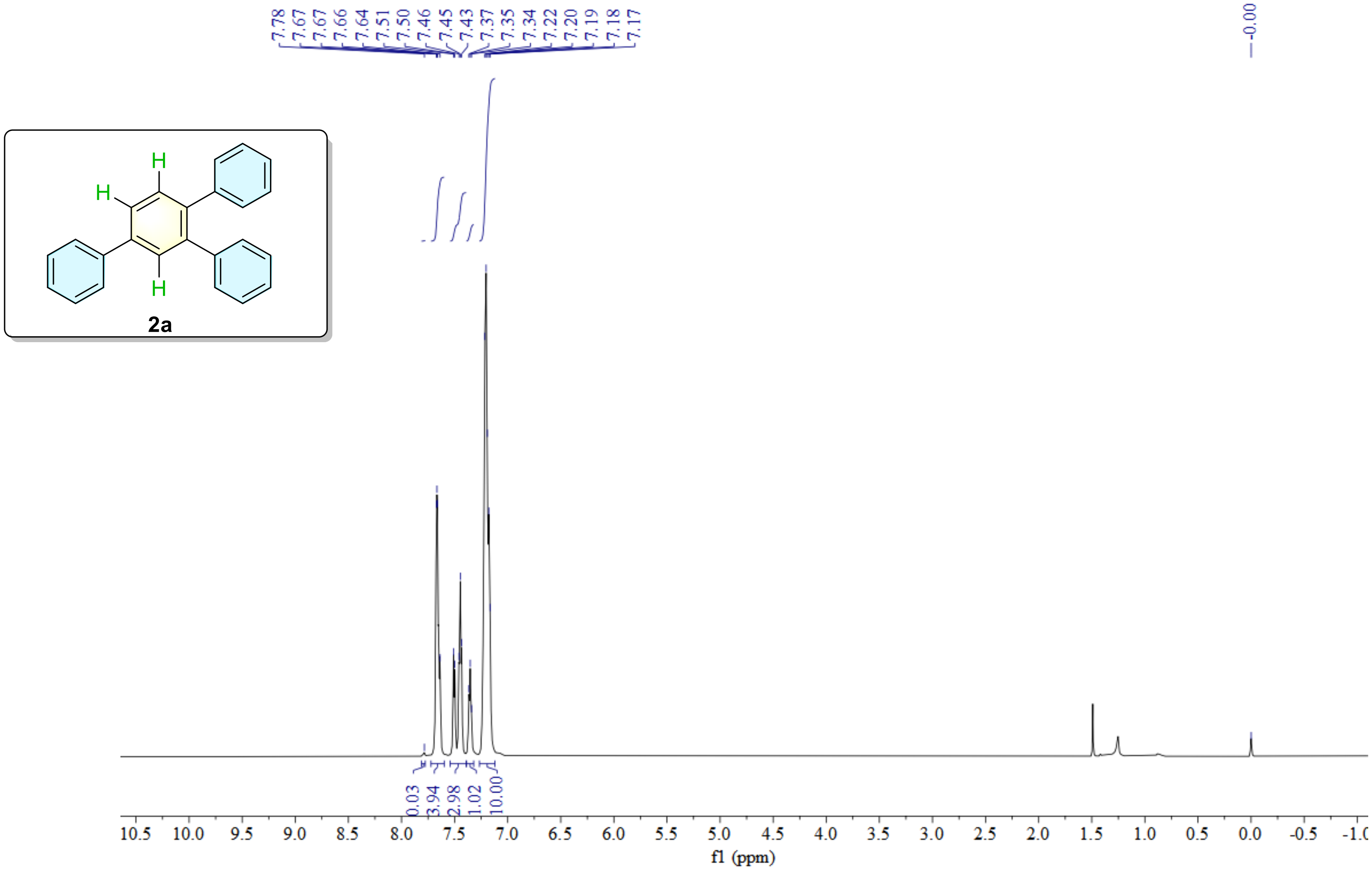
10. Copies for ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of the products



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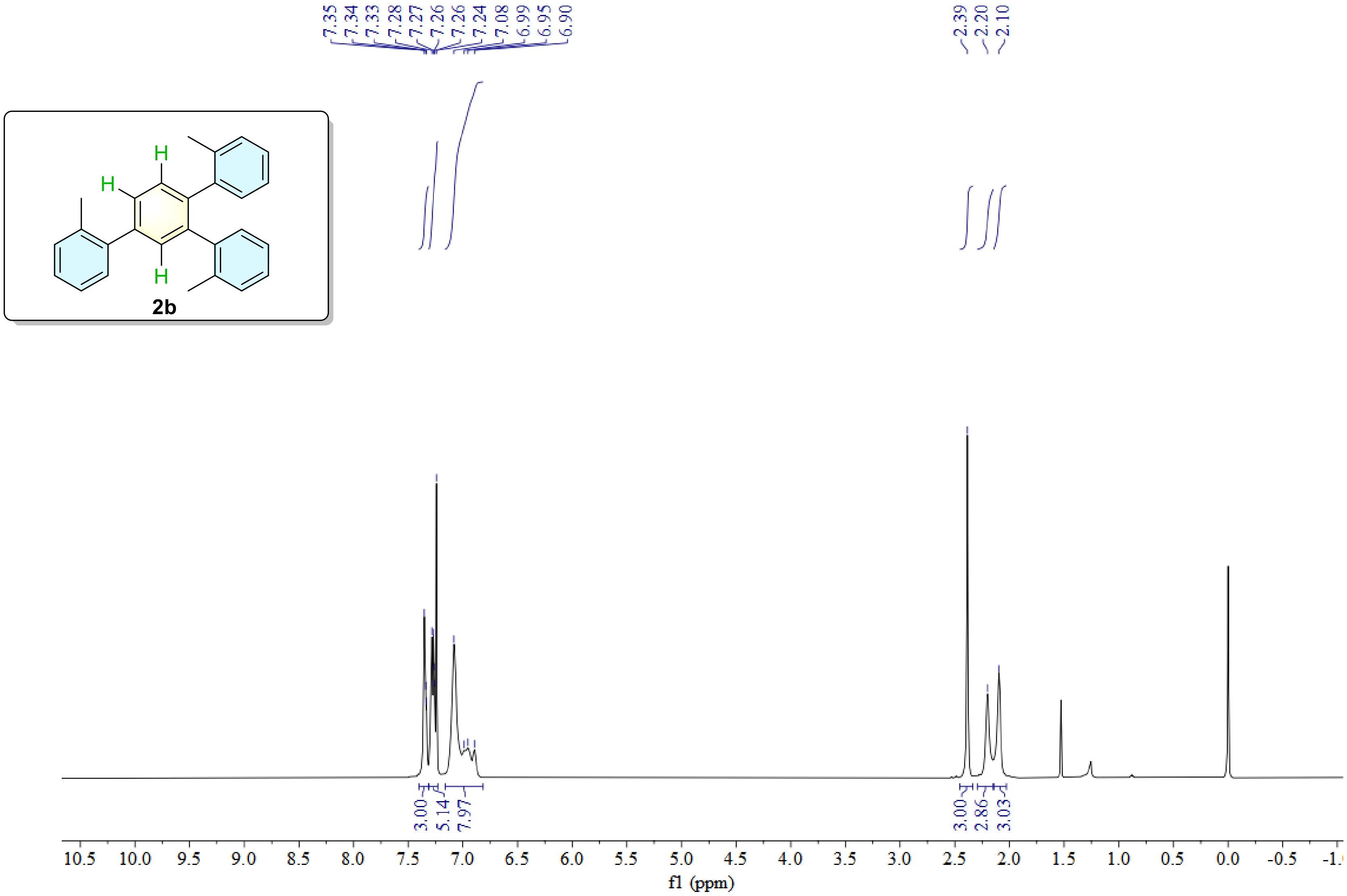
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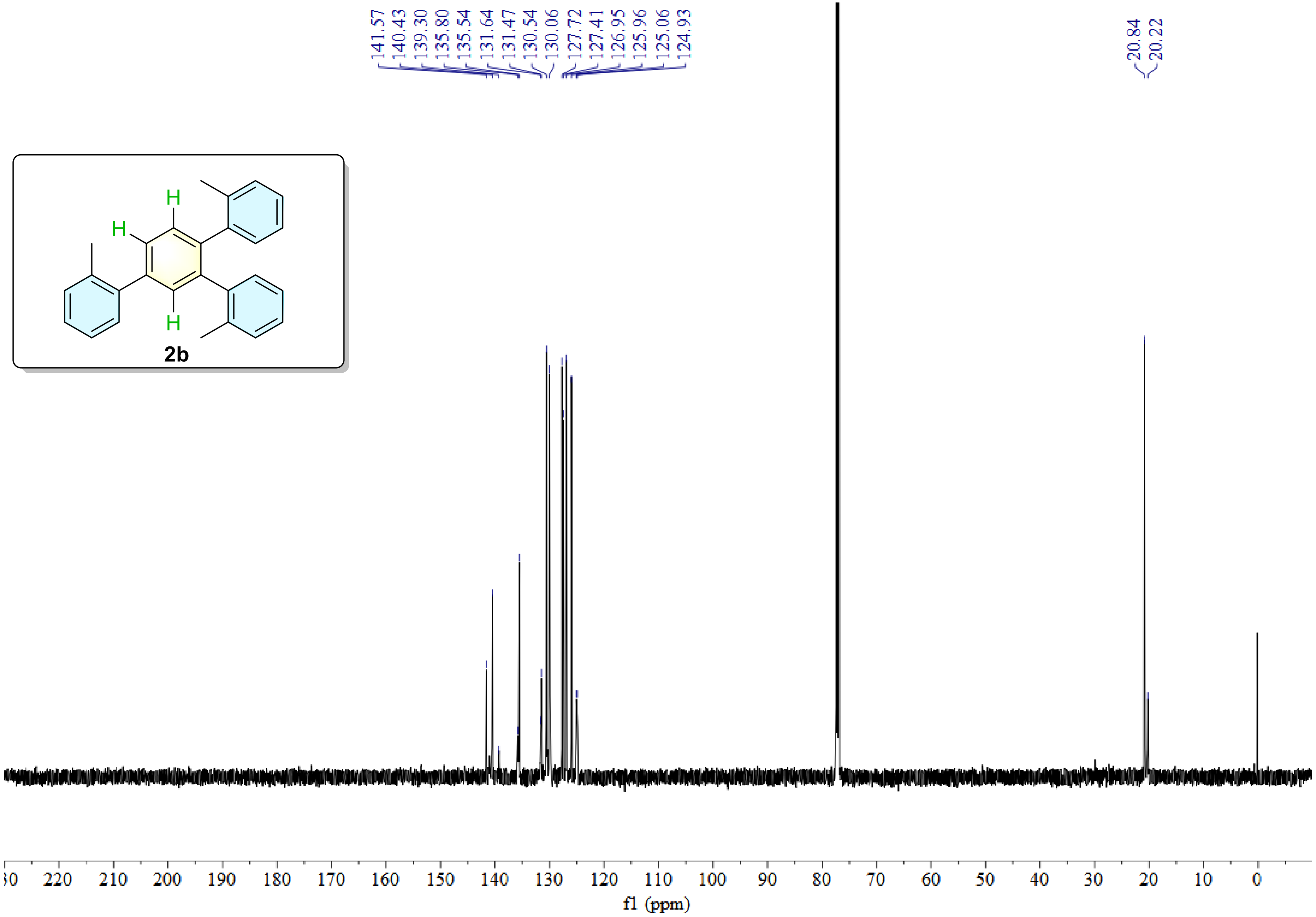
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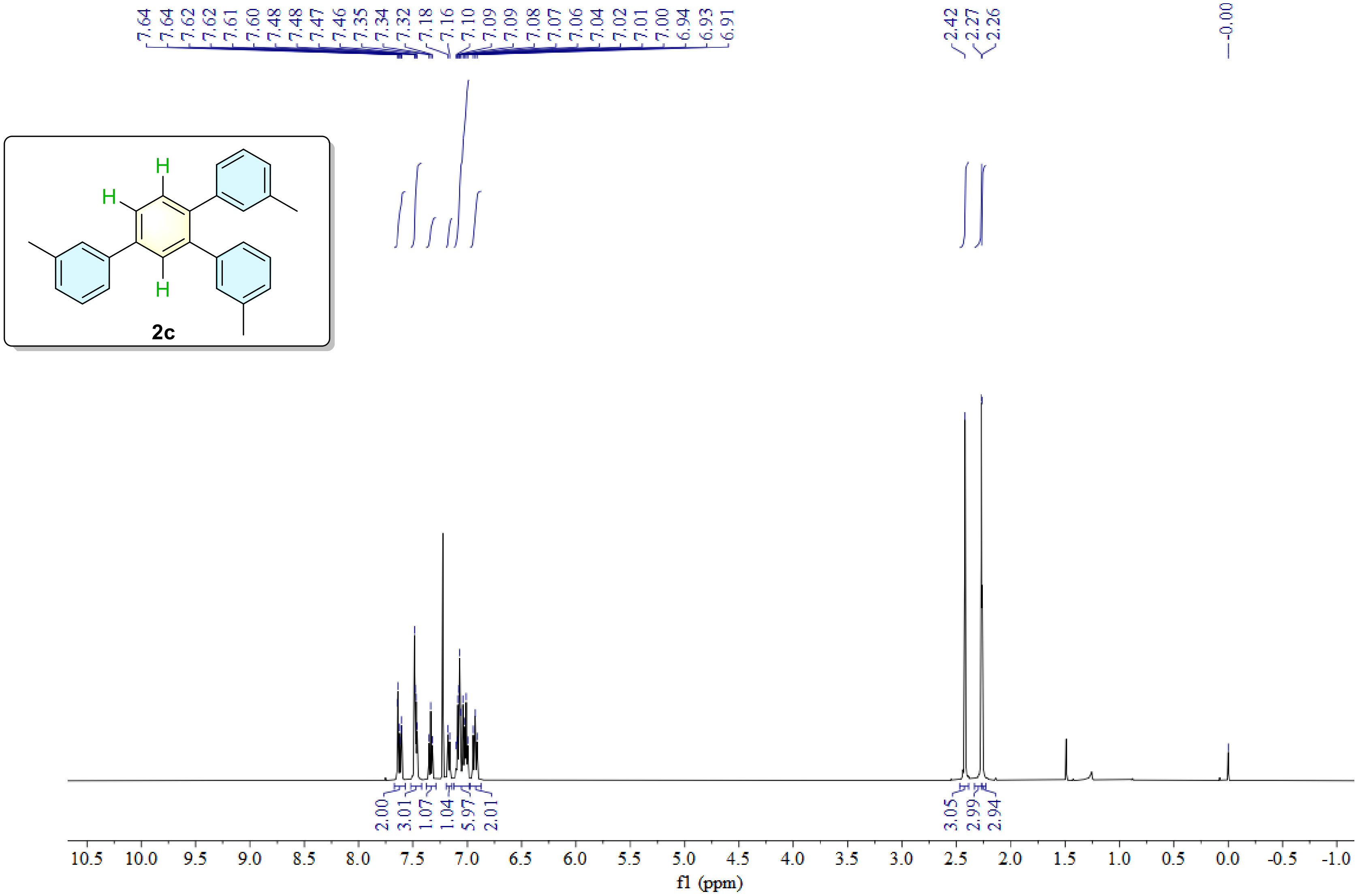
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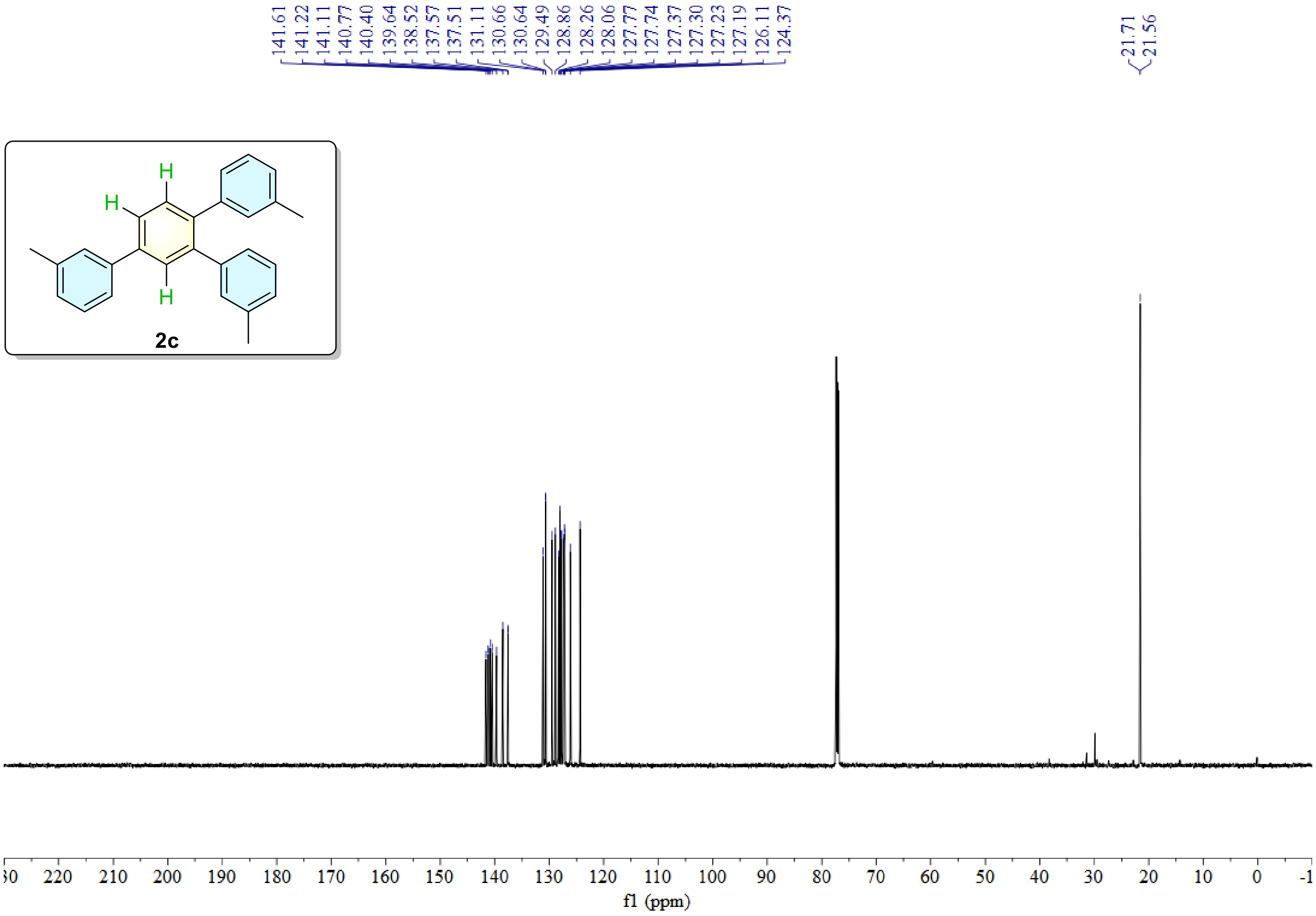
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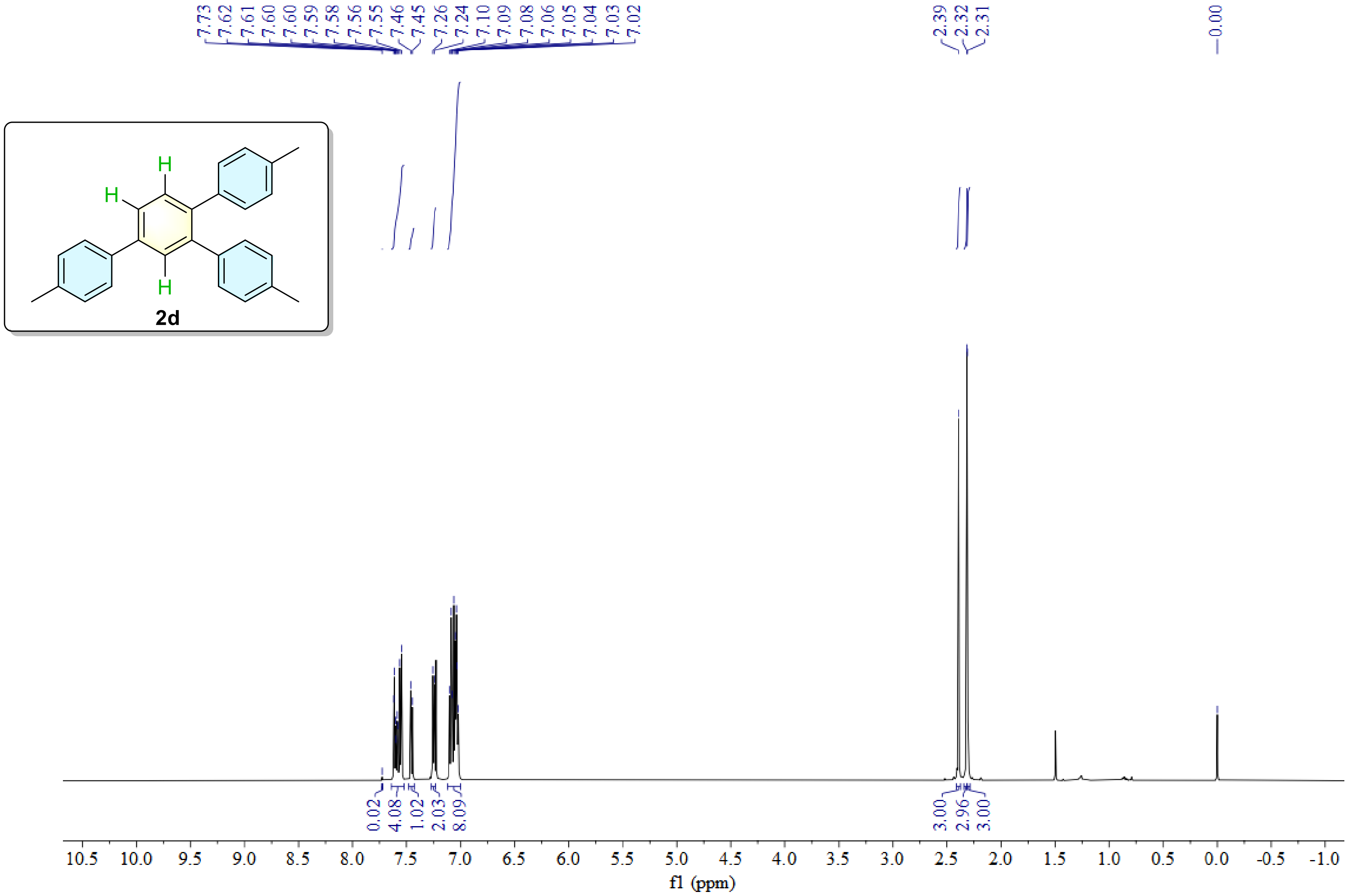
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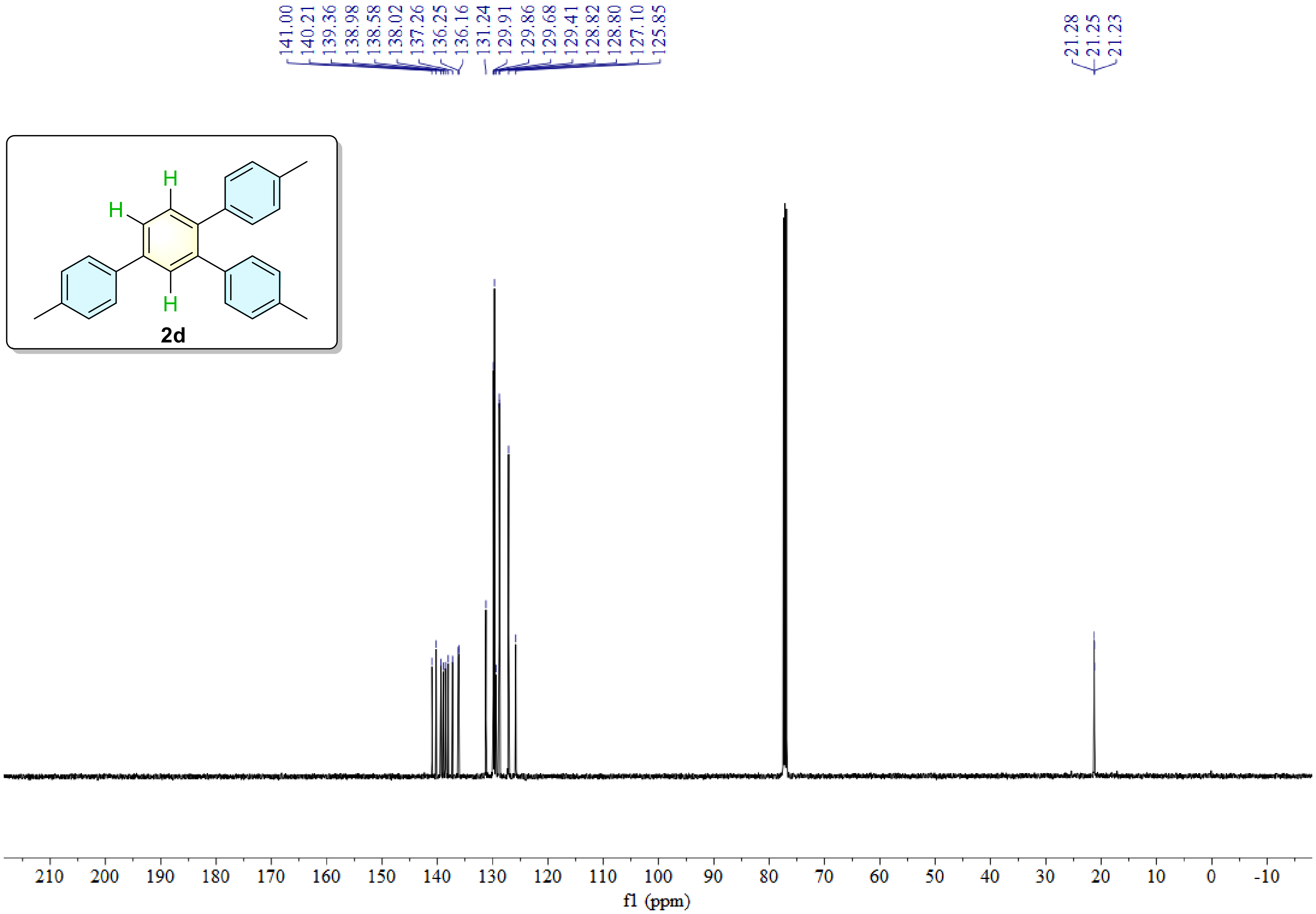
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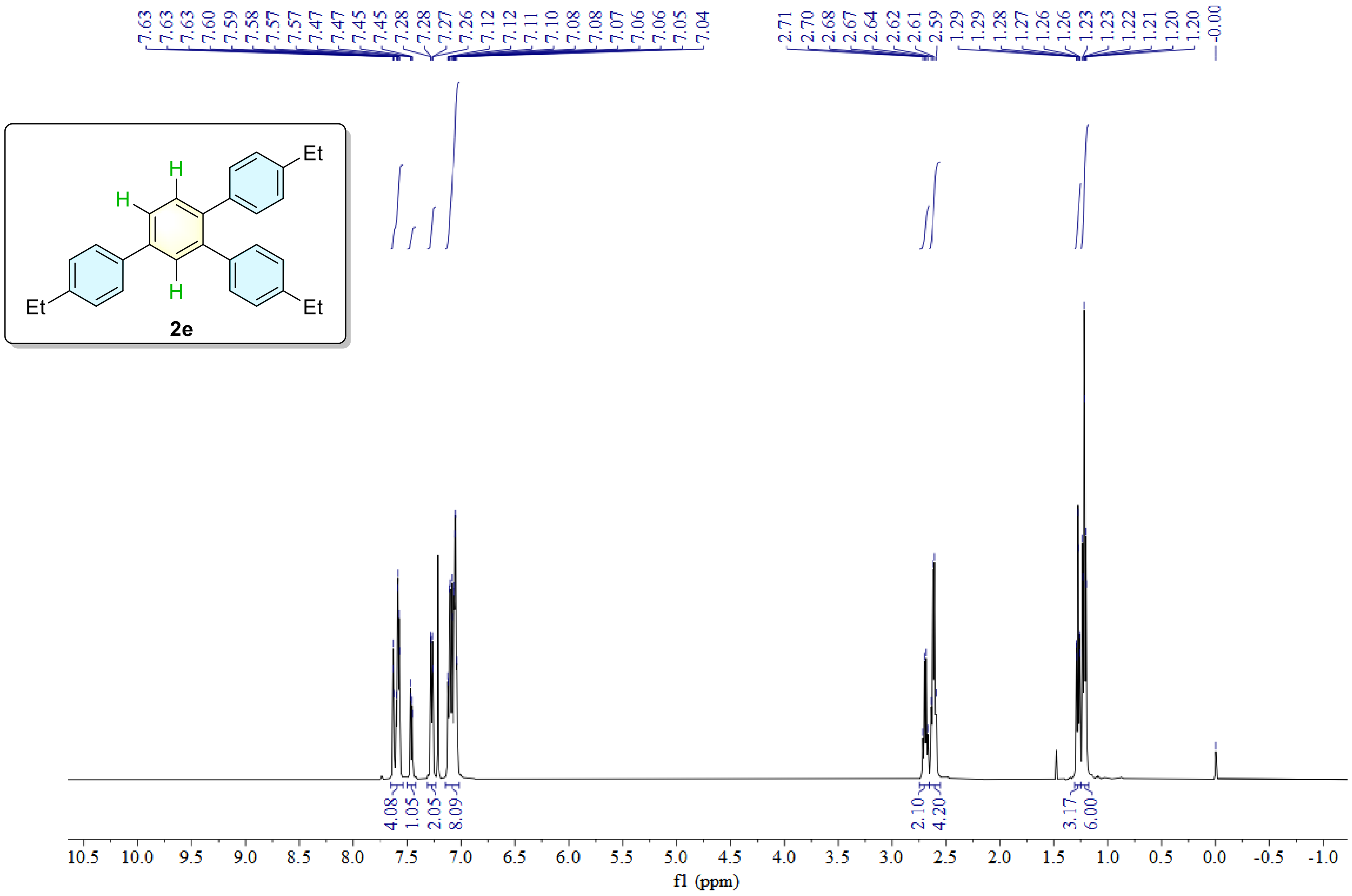
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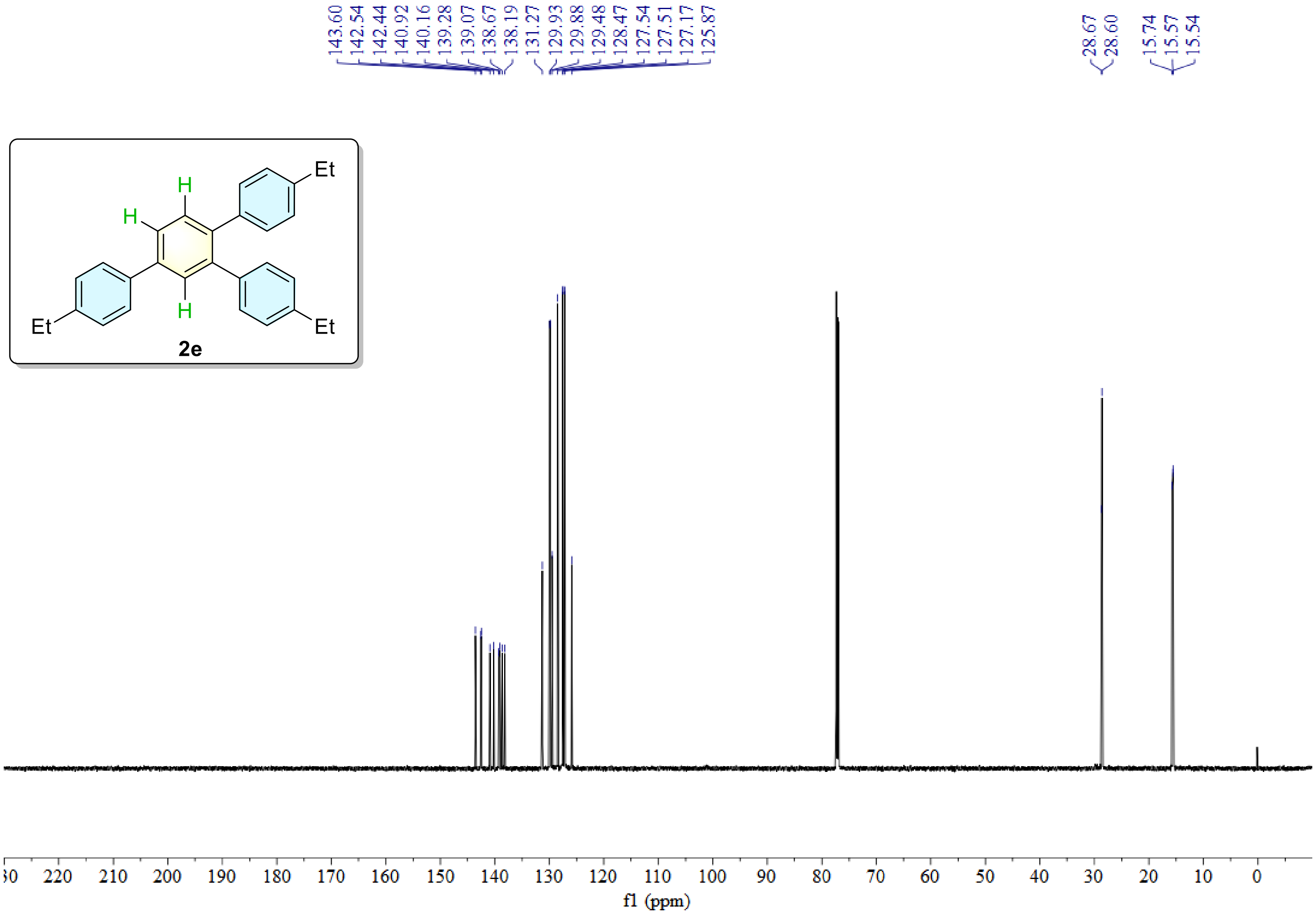
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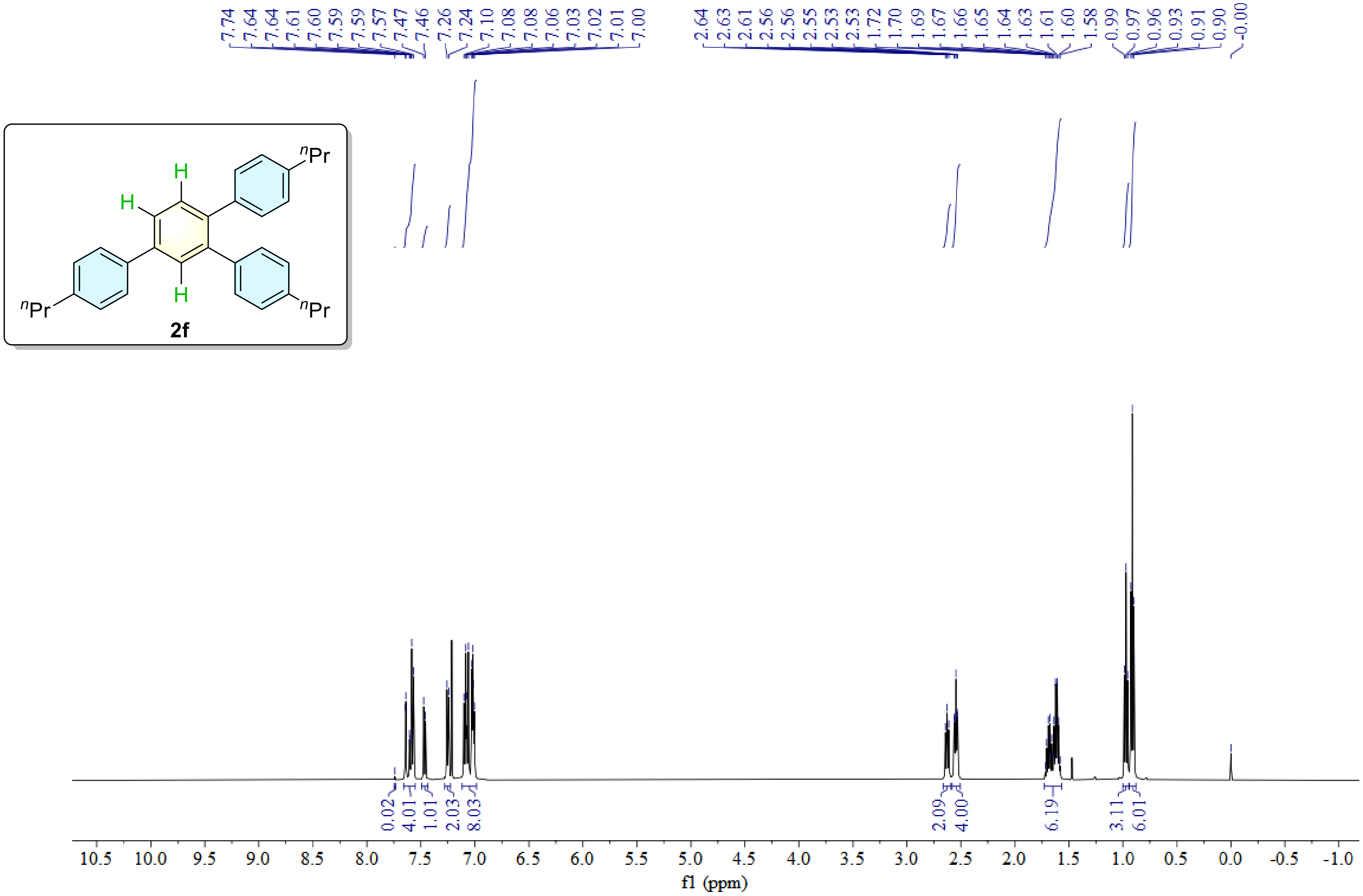
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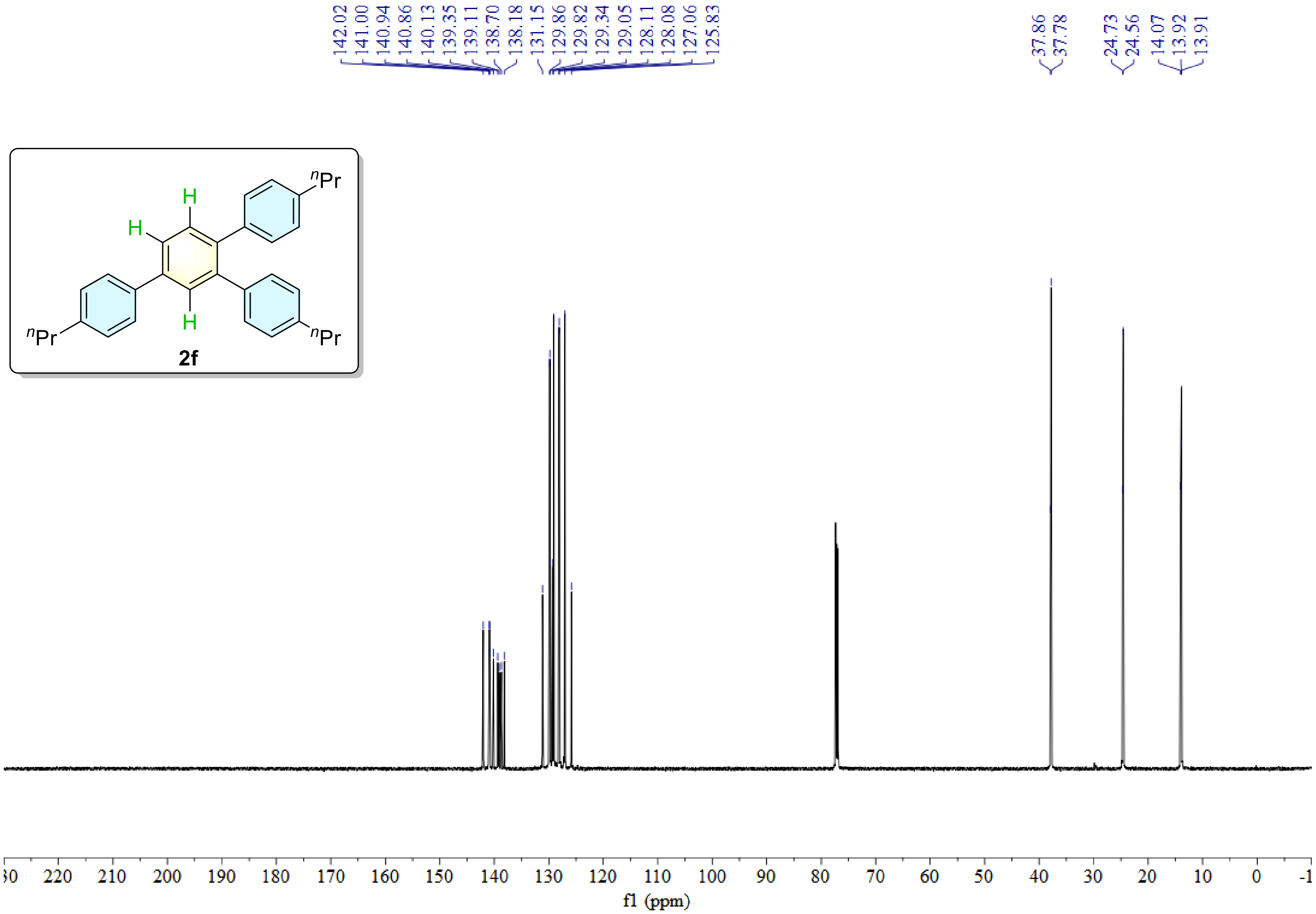
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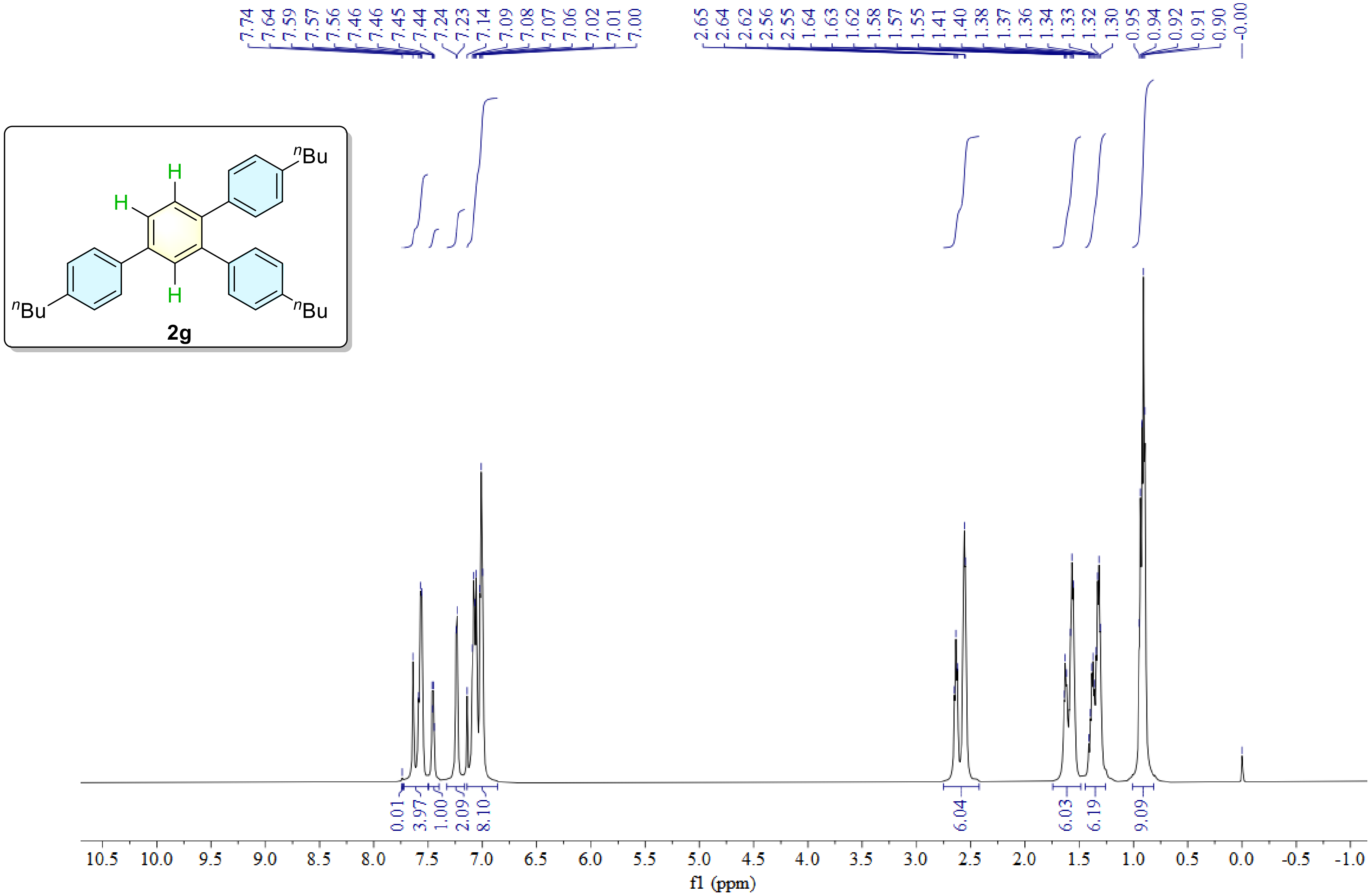

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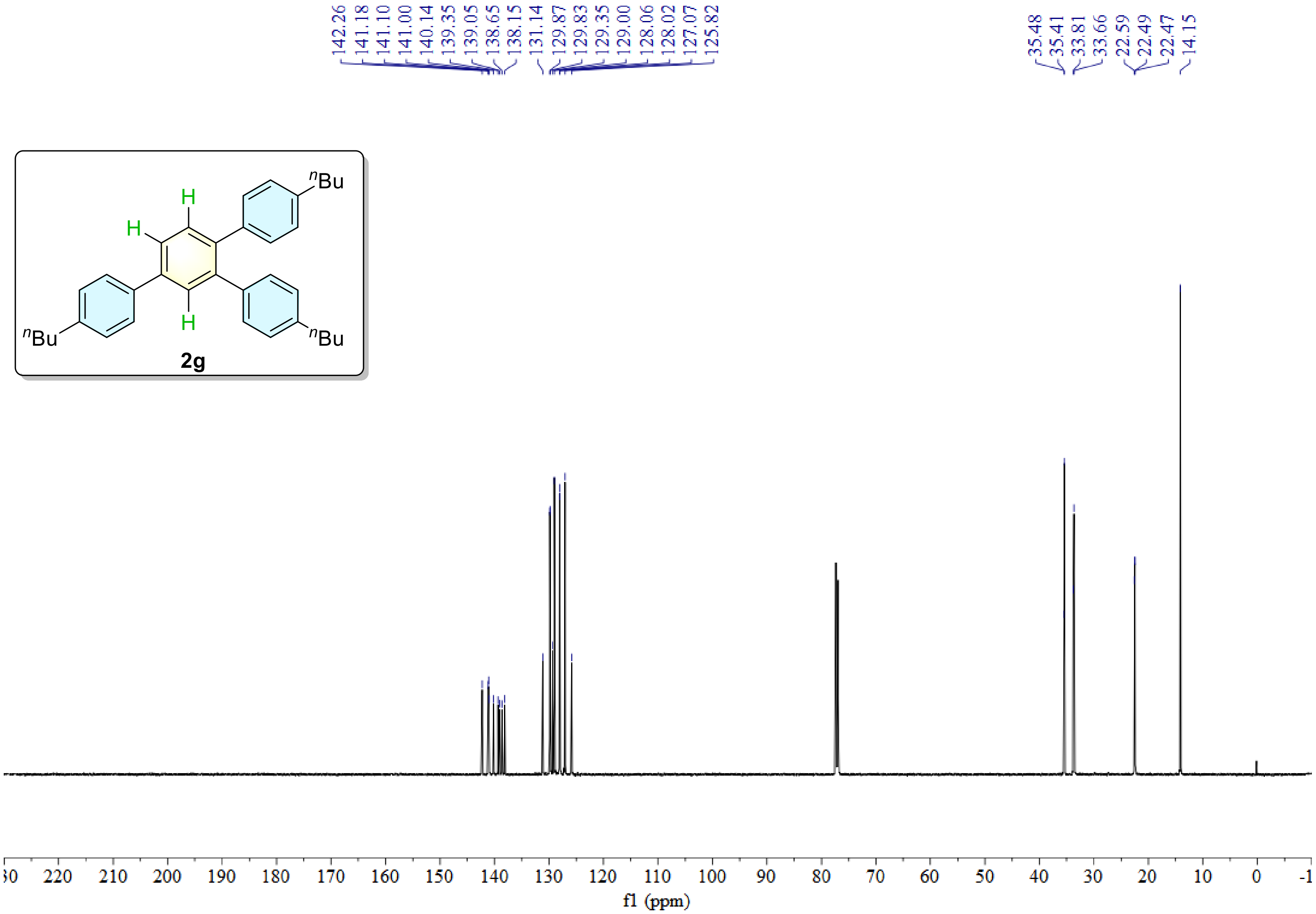
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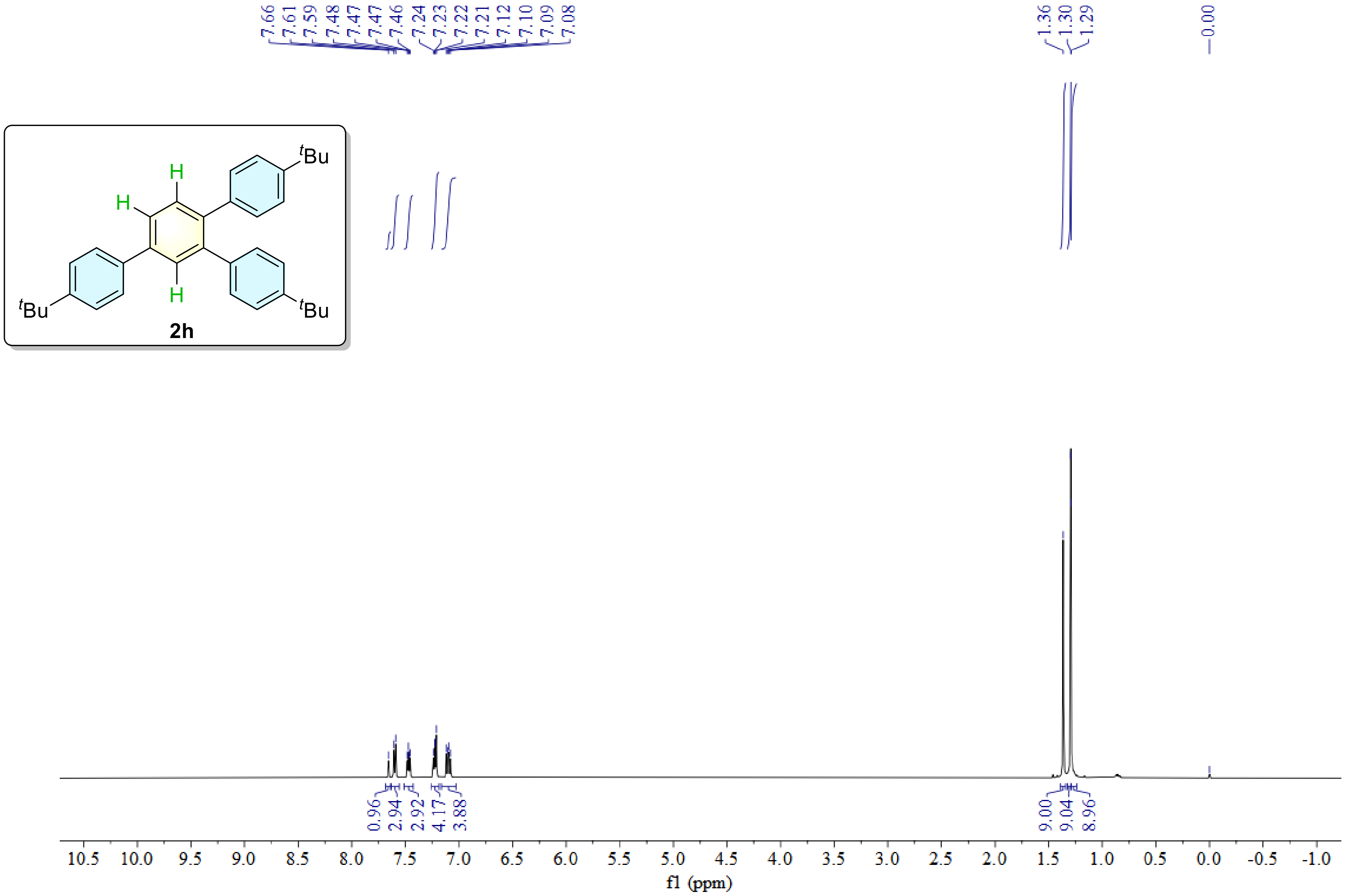
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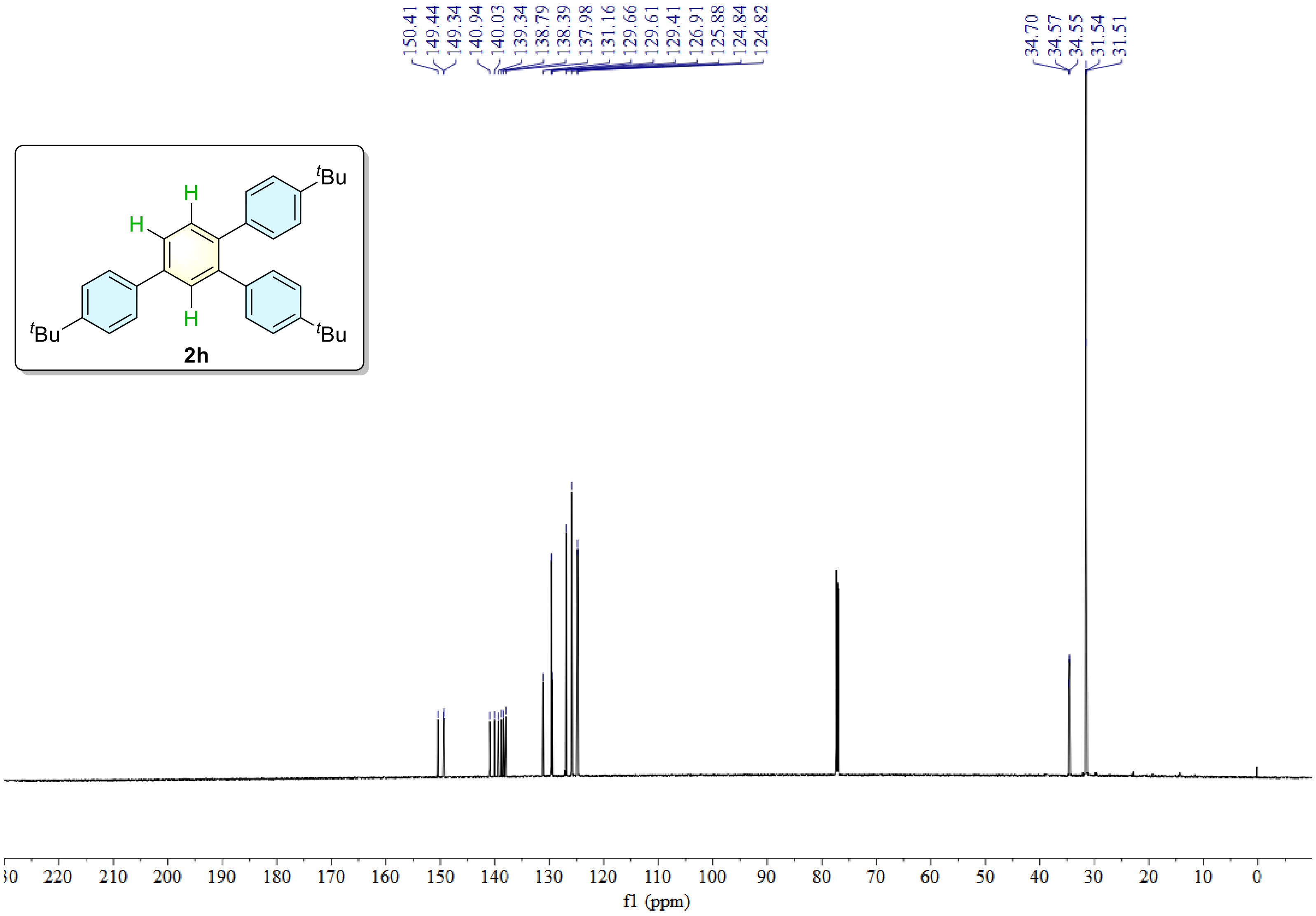
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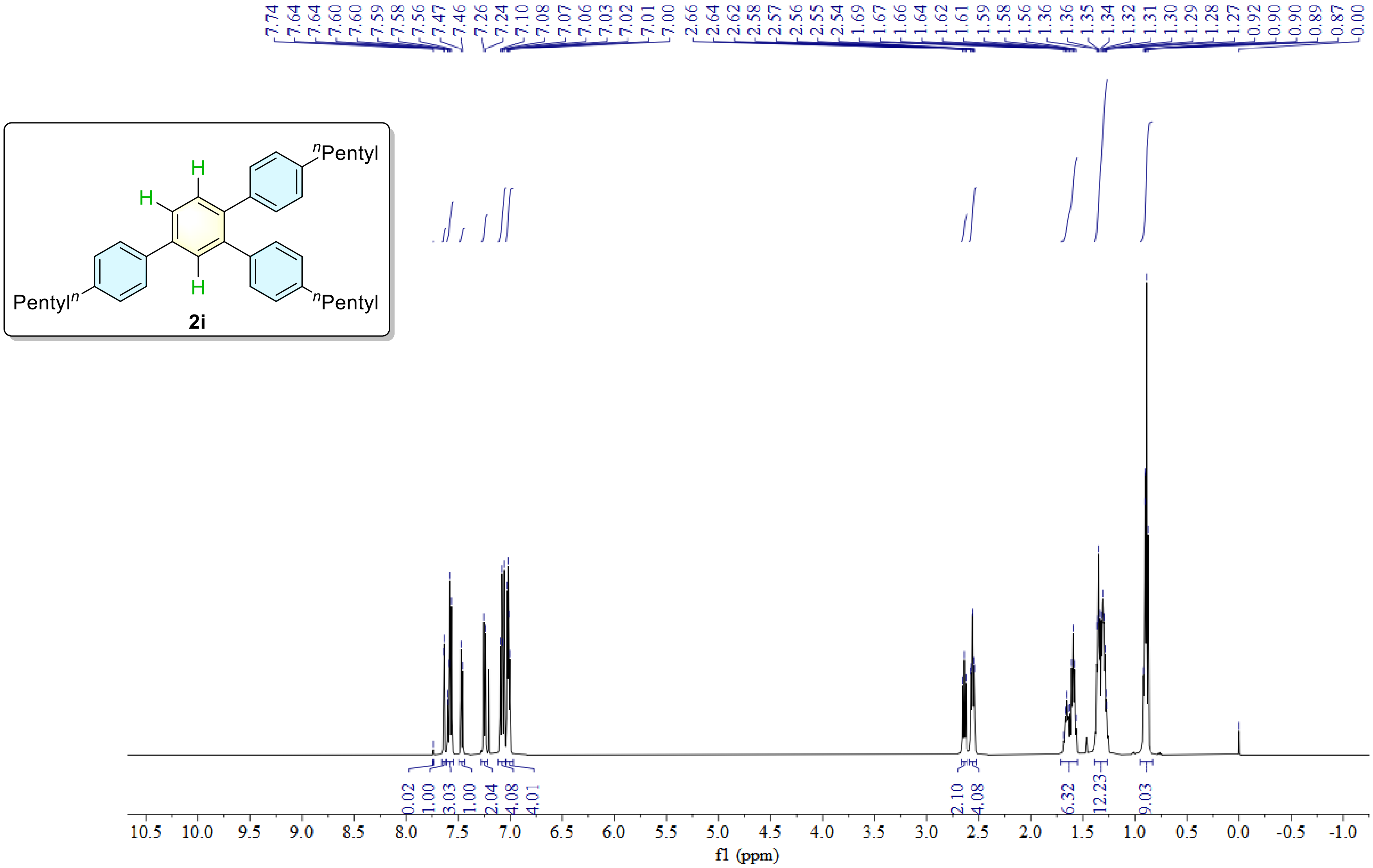
2i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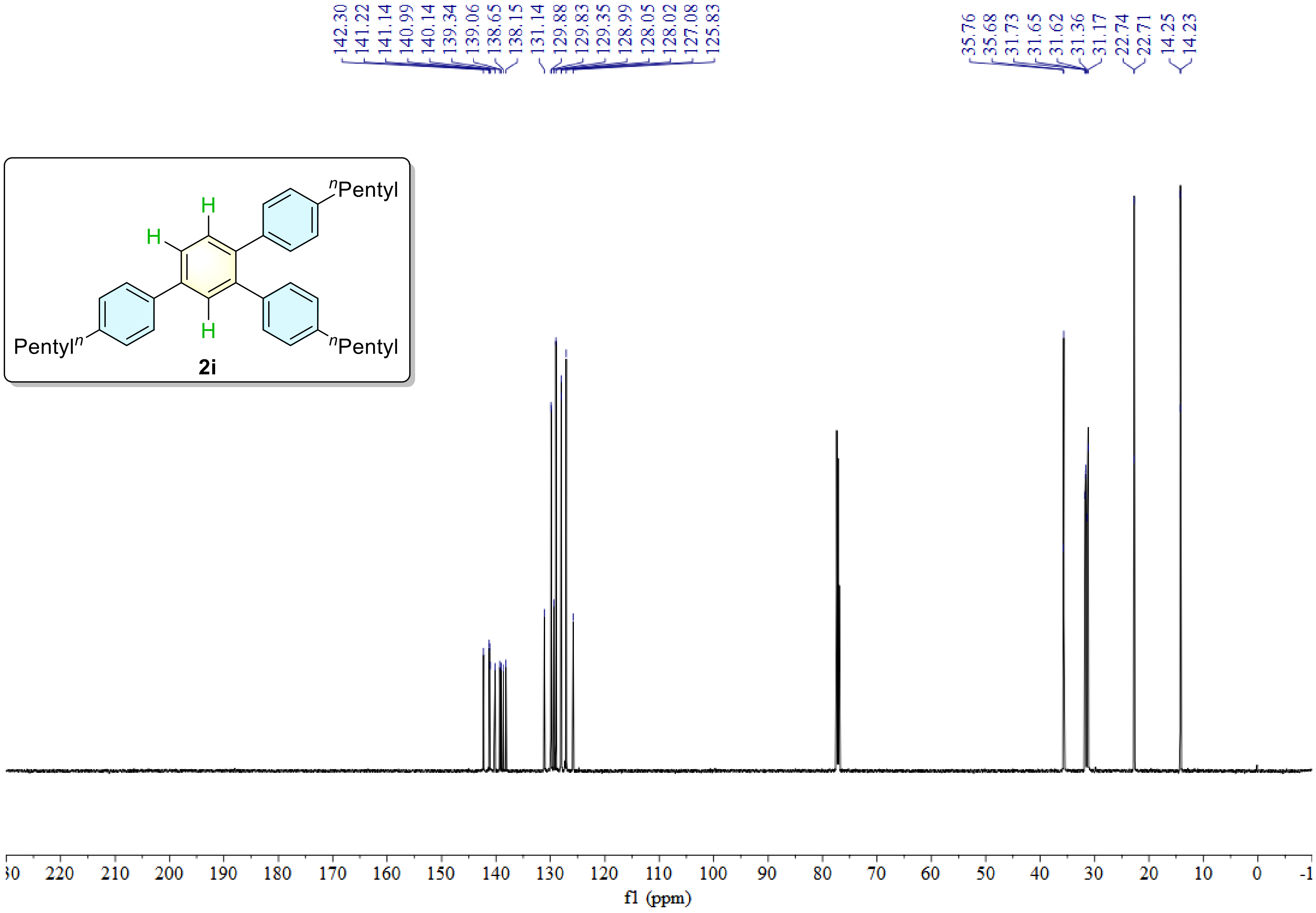
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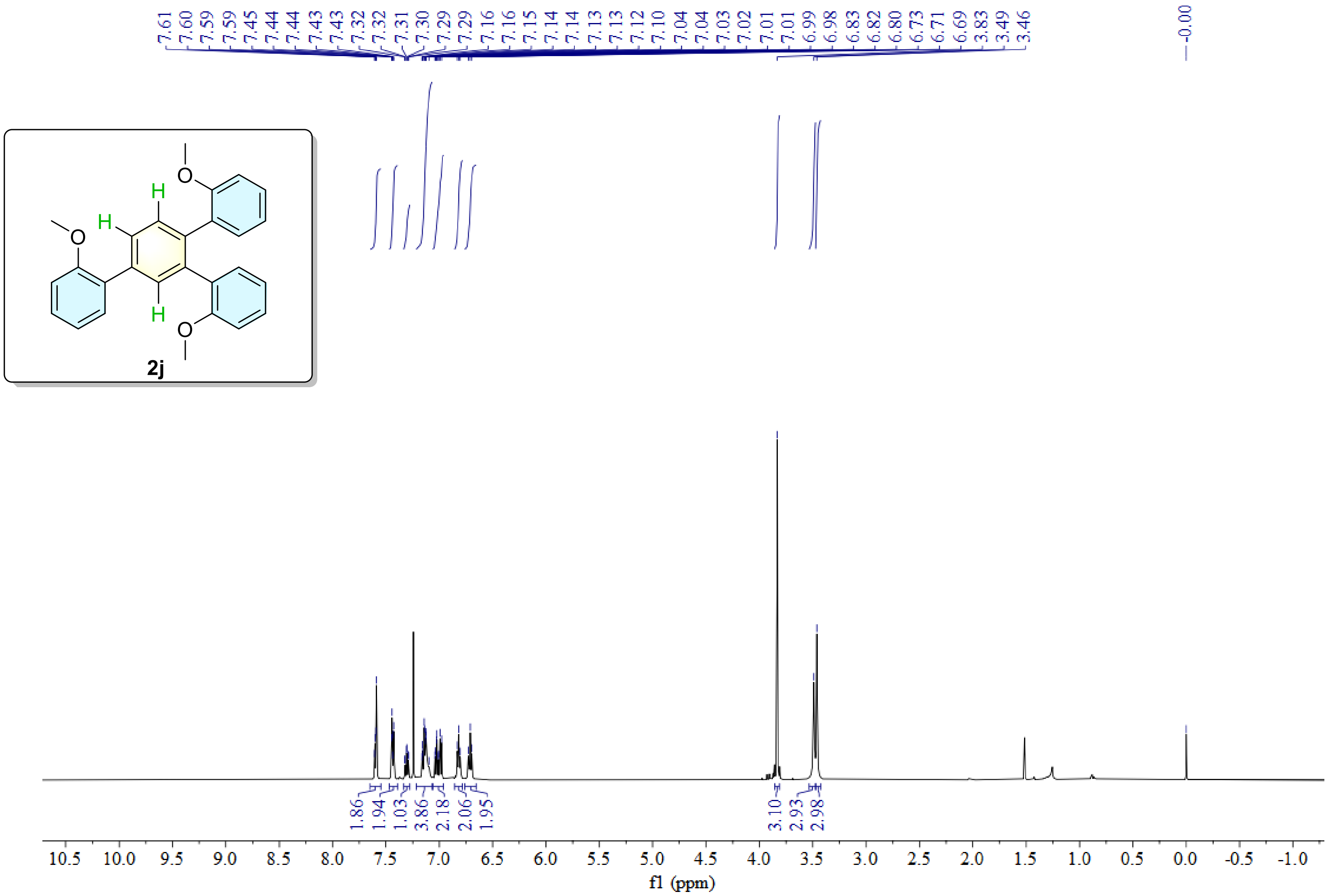
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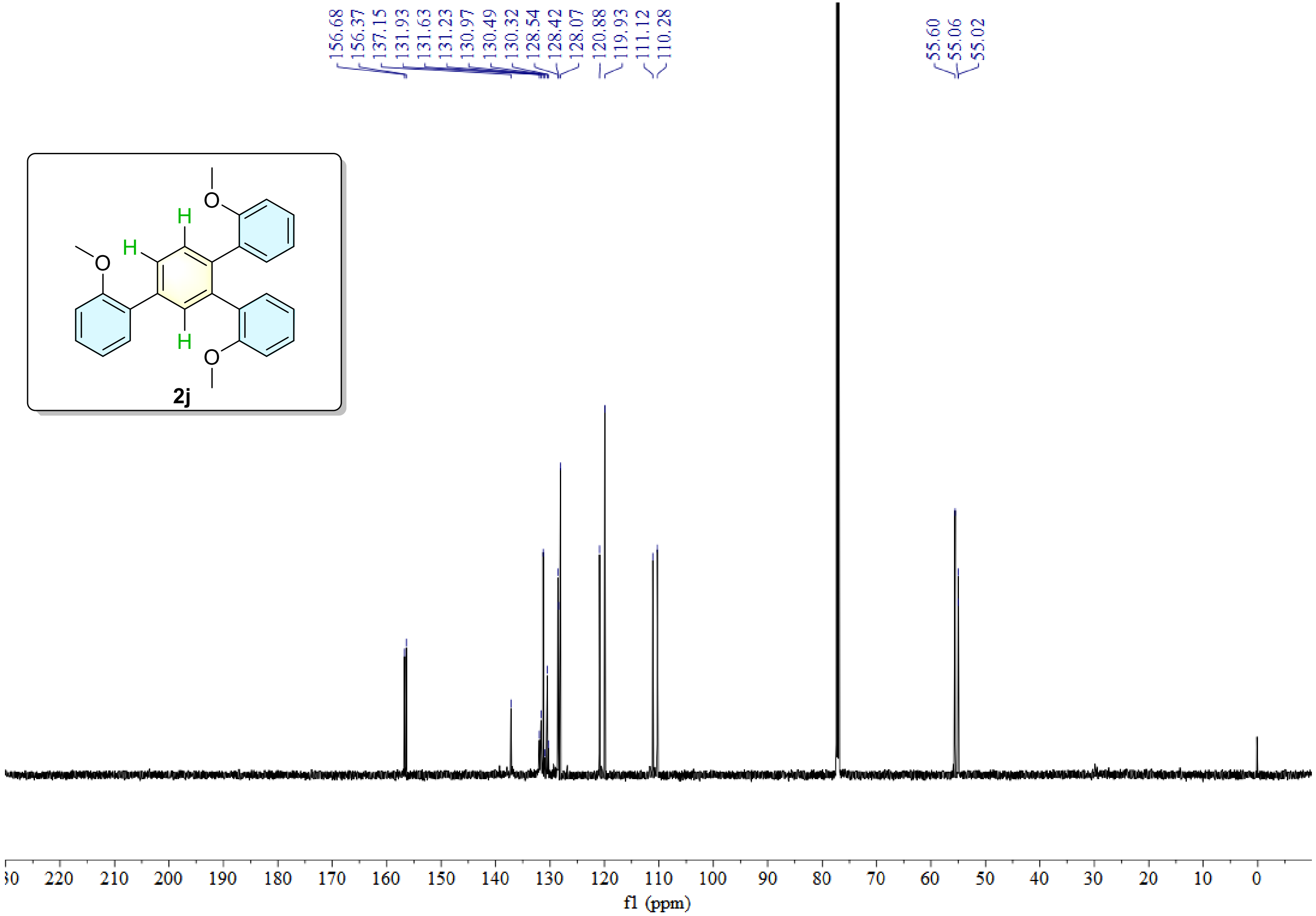
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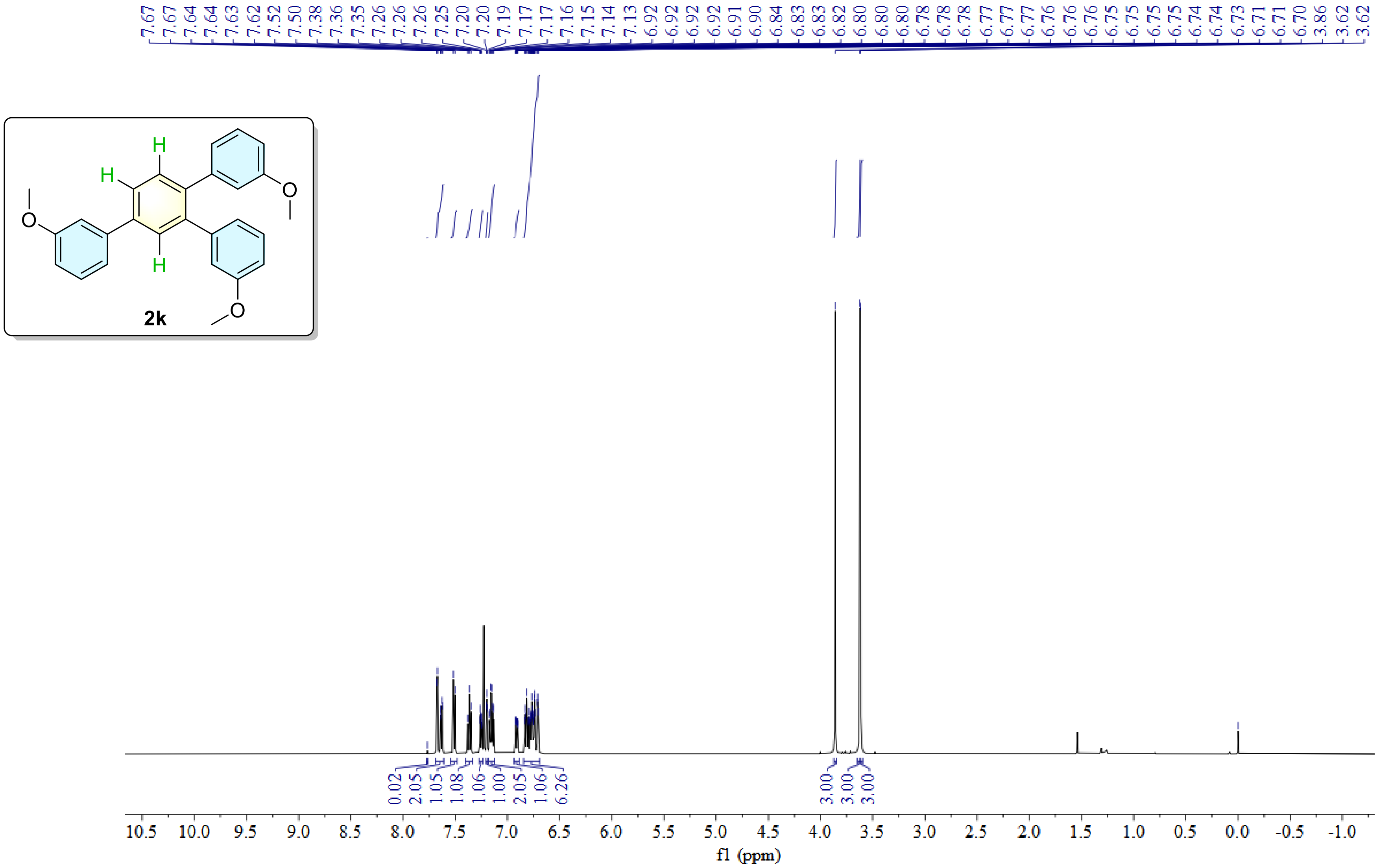
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3.62
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H
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O
O
O
2k
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1.08
1.06
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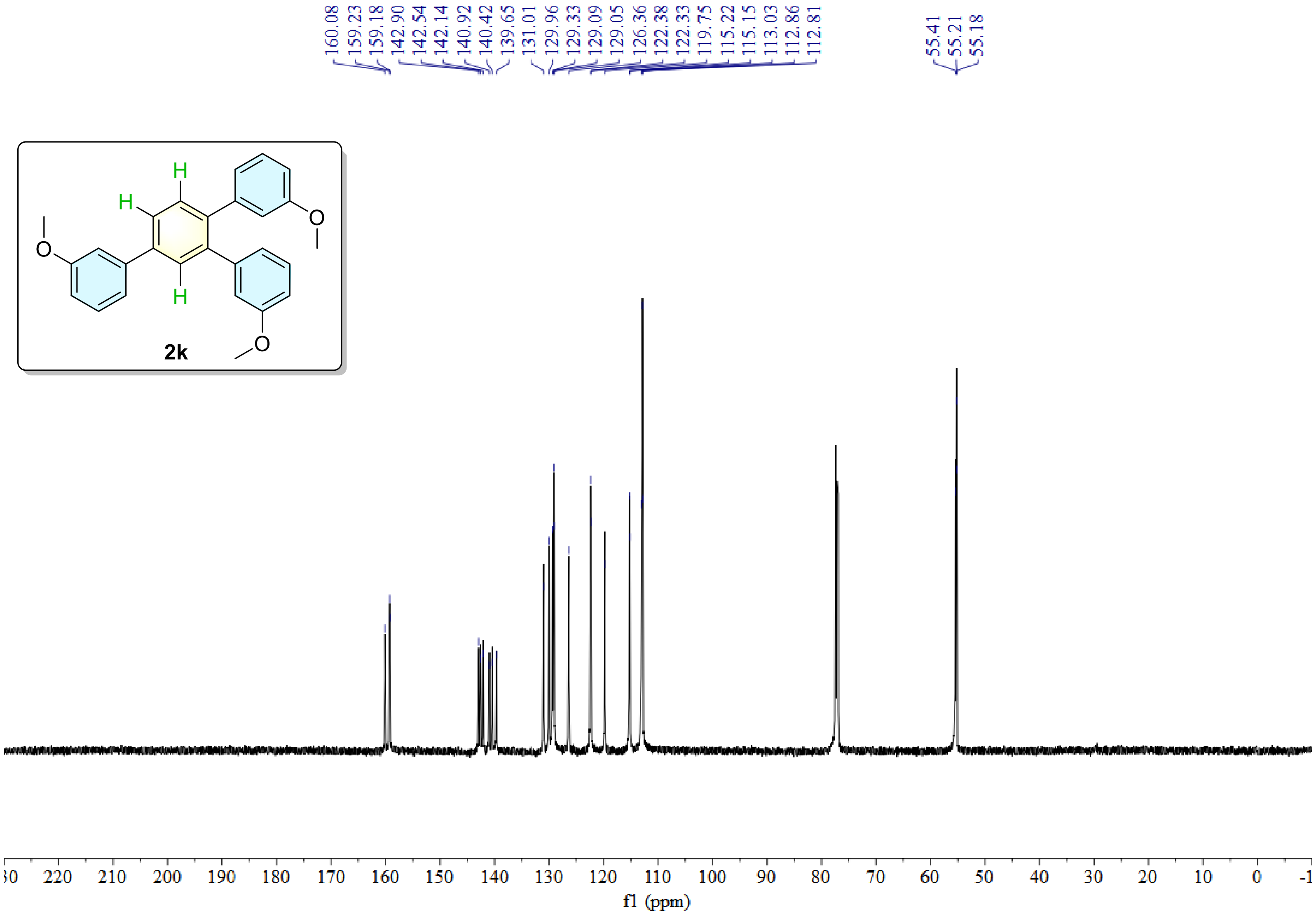
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113.03
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O
O
O
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180
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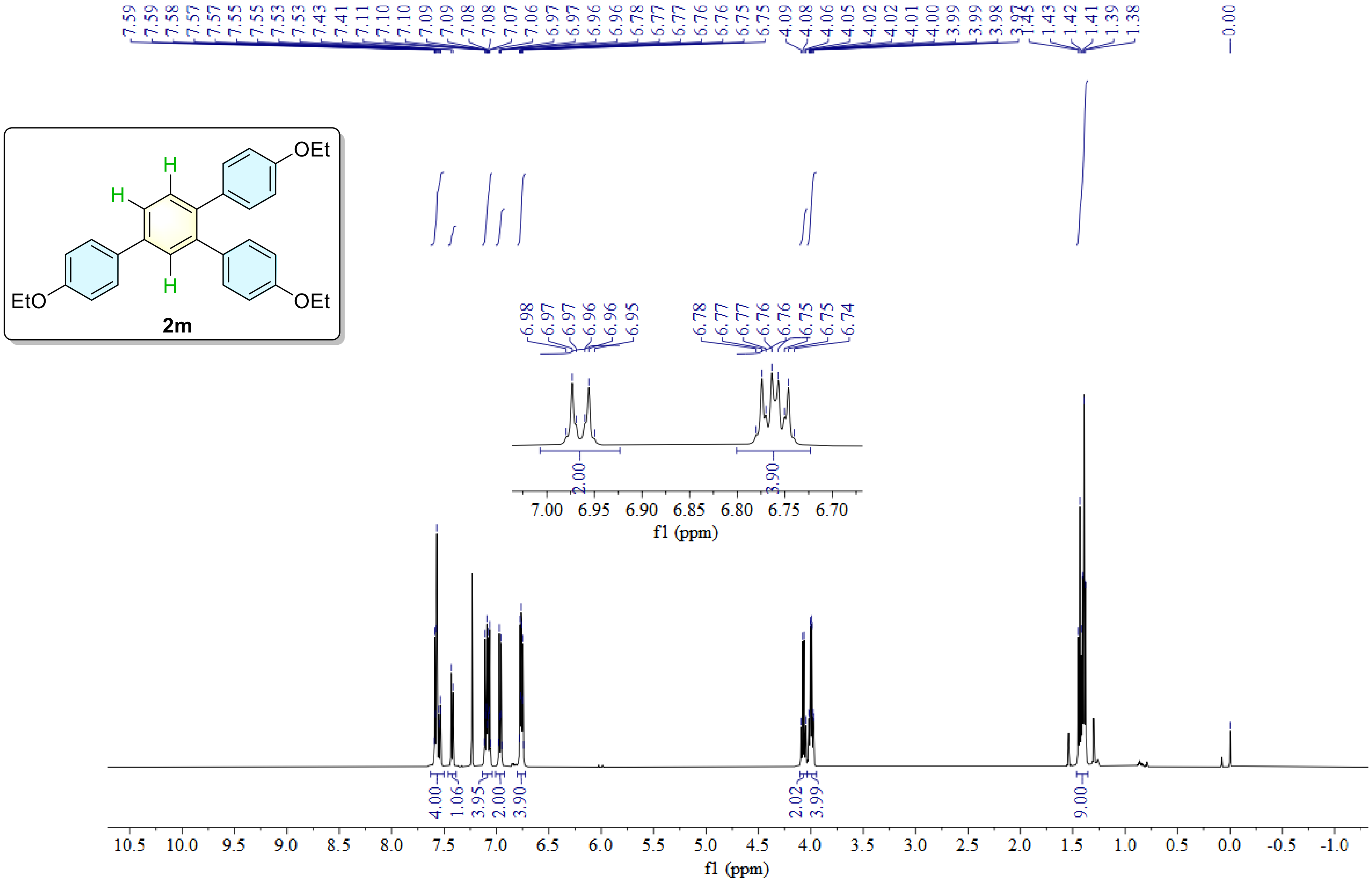
f1 (ppm)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H
H
H
OEt
OEt
EtO
2m
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6.77
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6.76
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6.80
6.75
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f1 (ppm)
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1.06
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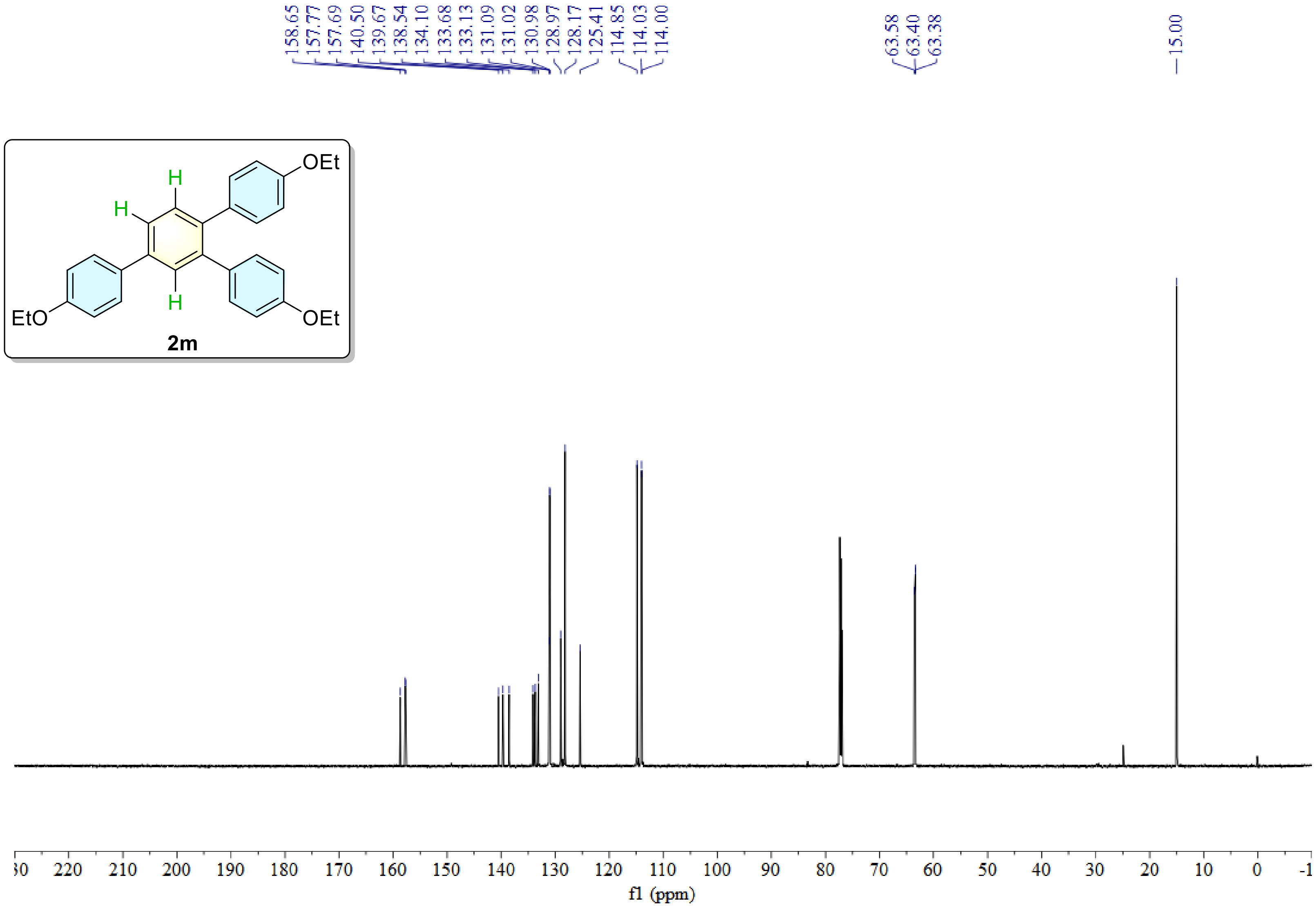
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125.41
114.85
114.03
114.00
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63.40
63.38
15.00
OEt
H
H
H
EtO
OEt
2m
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210
200
190
180
170
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100
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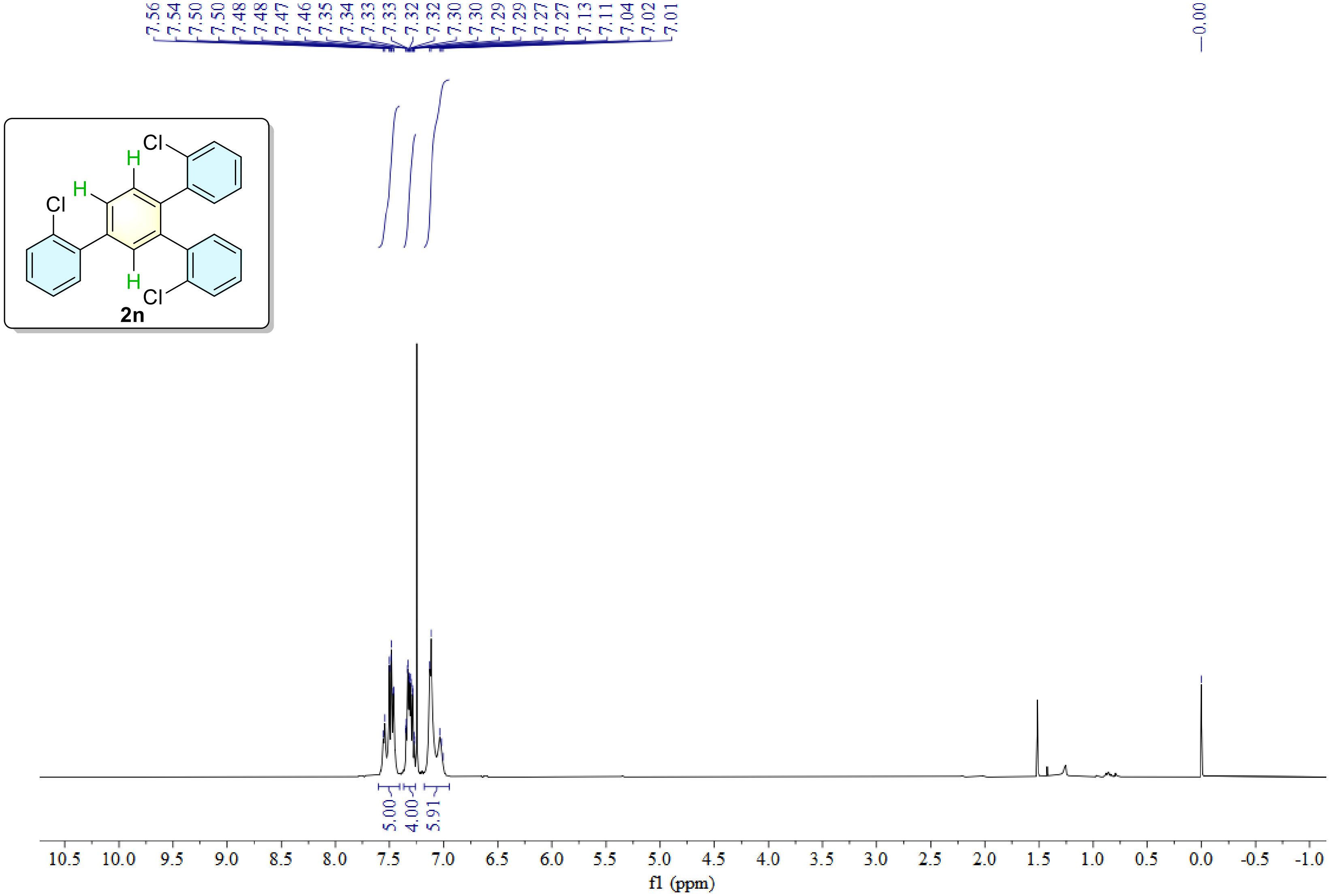
f1 (ppm)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H
H
Cl
H
Cl
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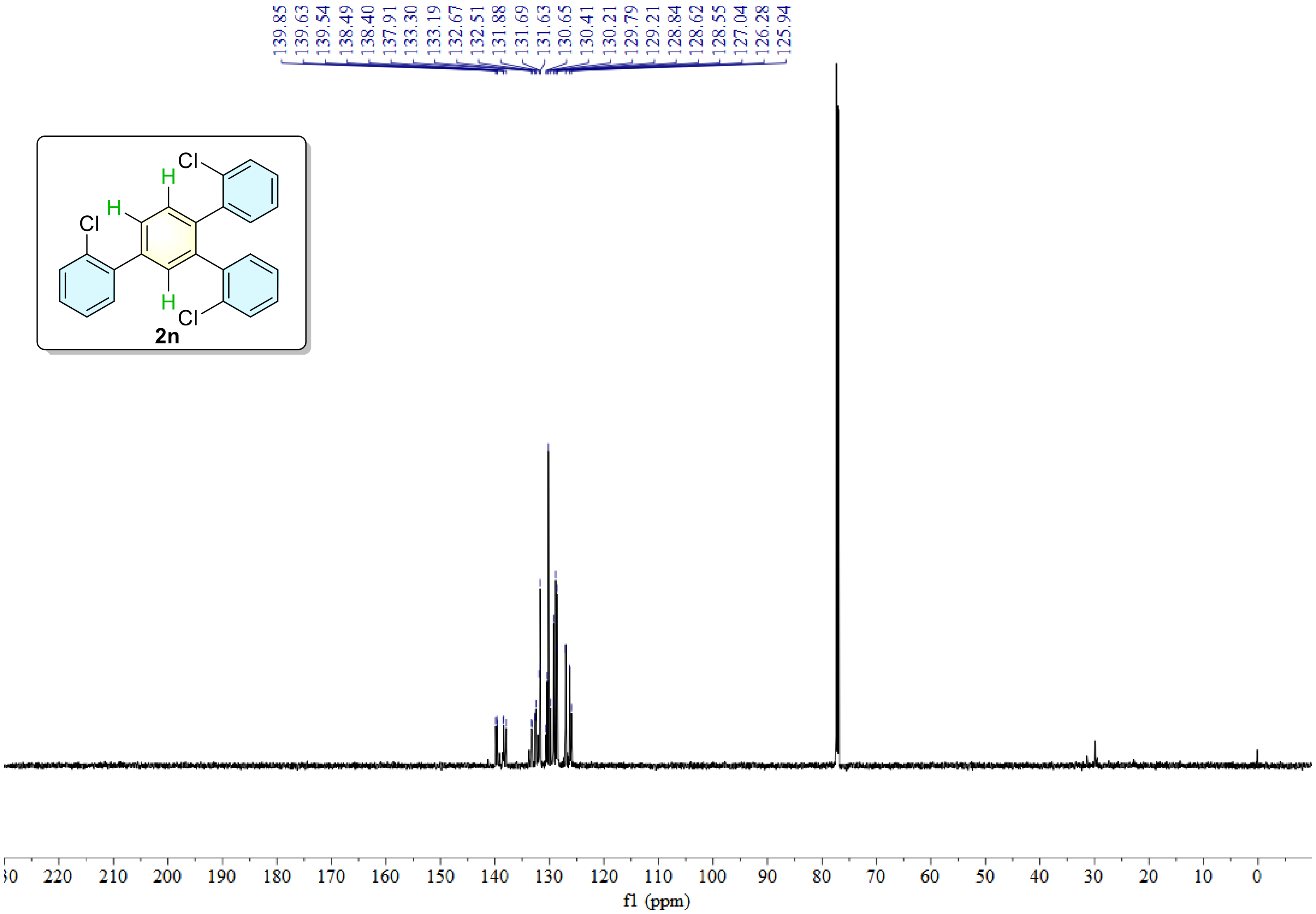
f1 (ppm)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128.55
127.04
126.28
125.94
Cl
H
H
Cl
H
Cl
2n
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210
200
190
180
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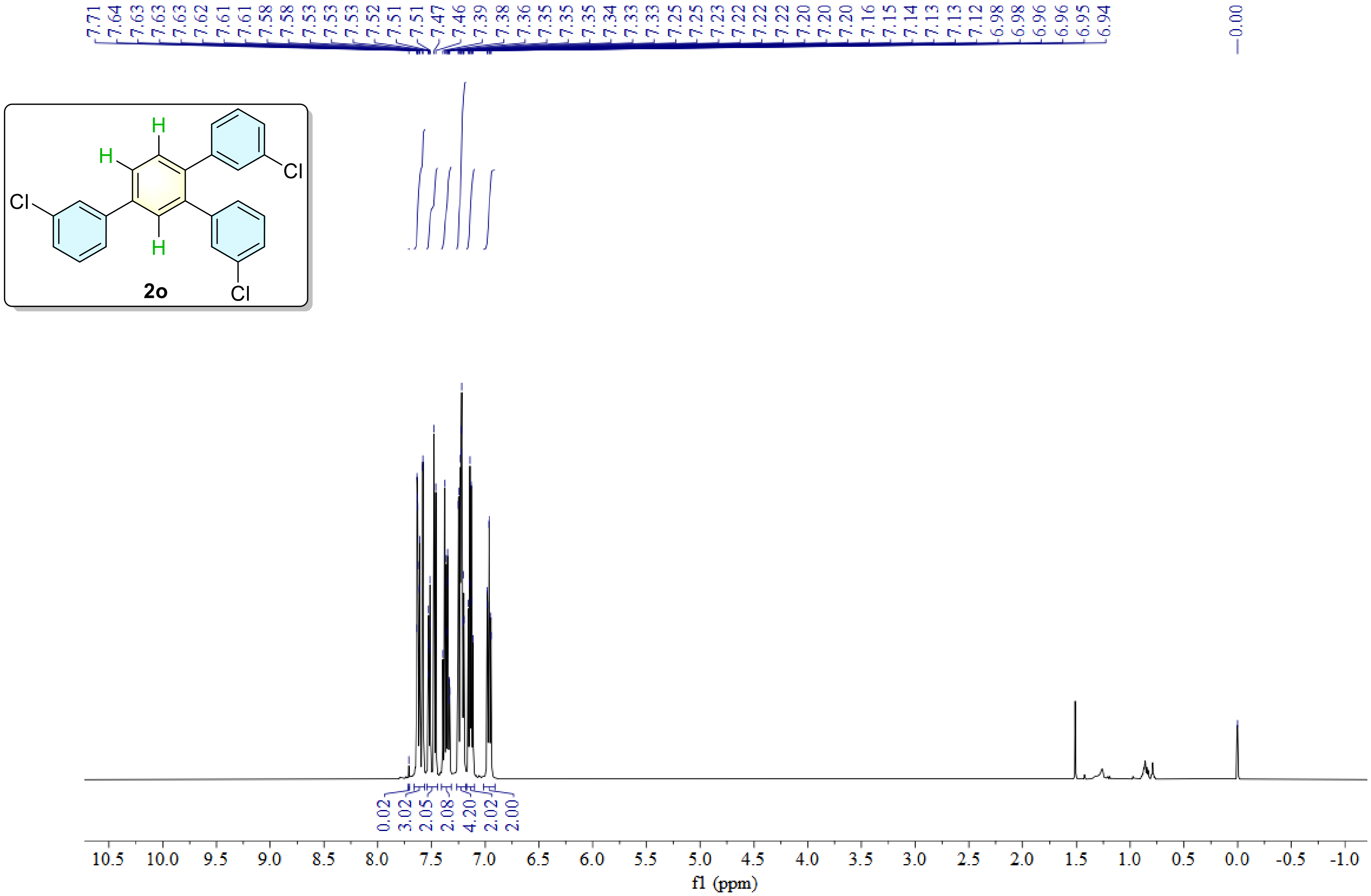
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7.51
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7.38
7.36
7.35
7.35
7.35
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6.96
6.96
6.95
6.94
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H
H
Cl
Cl
Cl
2o
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2.08
4.20
2.02
2.00
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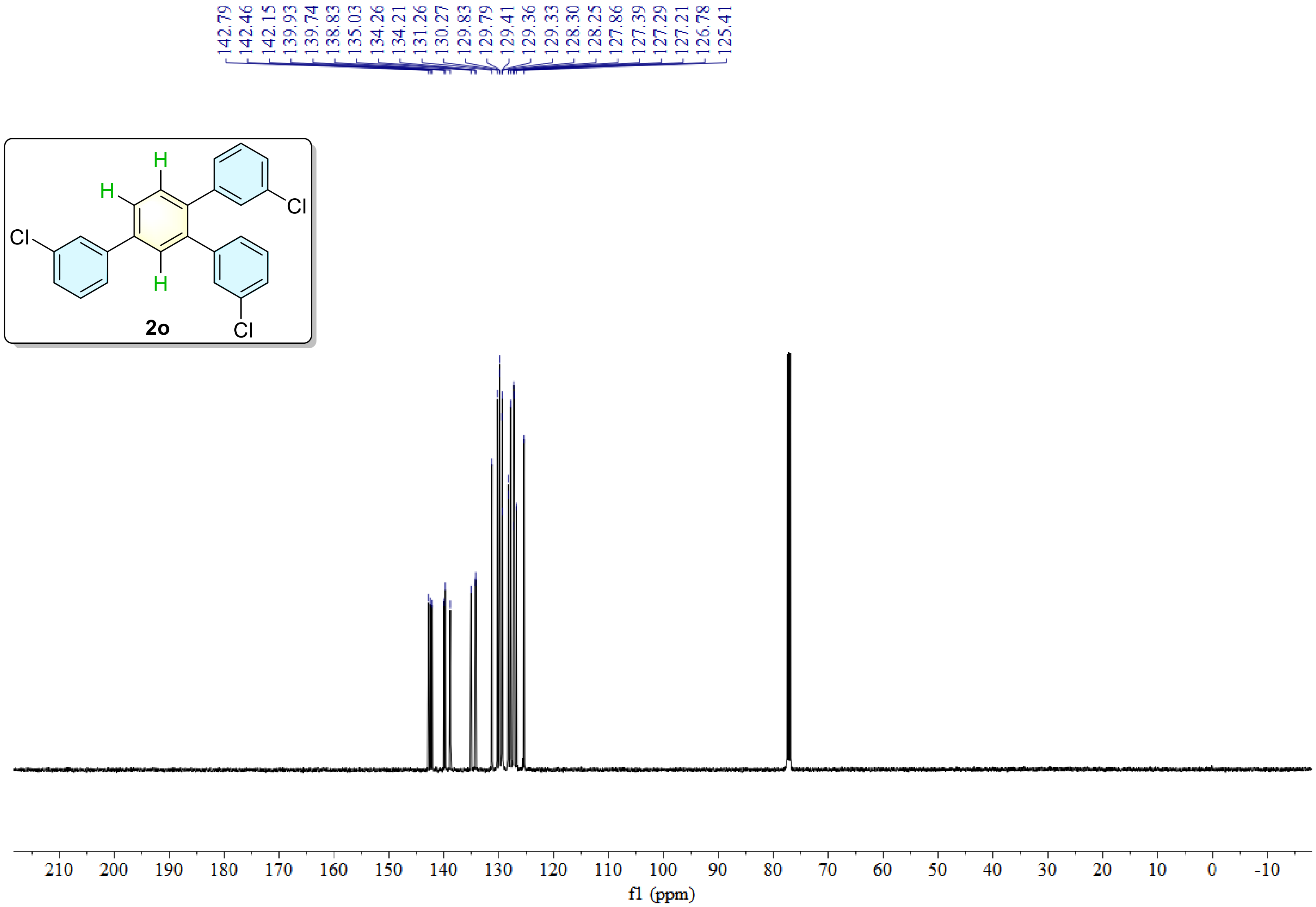
-1.0
f1 (ppm)



2o

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129.79
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129.36
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128.30
128.25
127.86
127.39
127.29
127.21
126.78
125.41
H
H
H
Cl
Cl
Cl
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200
190
180
170
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100
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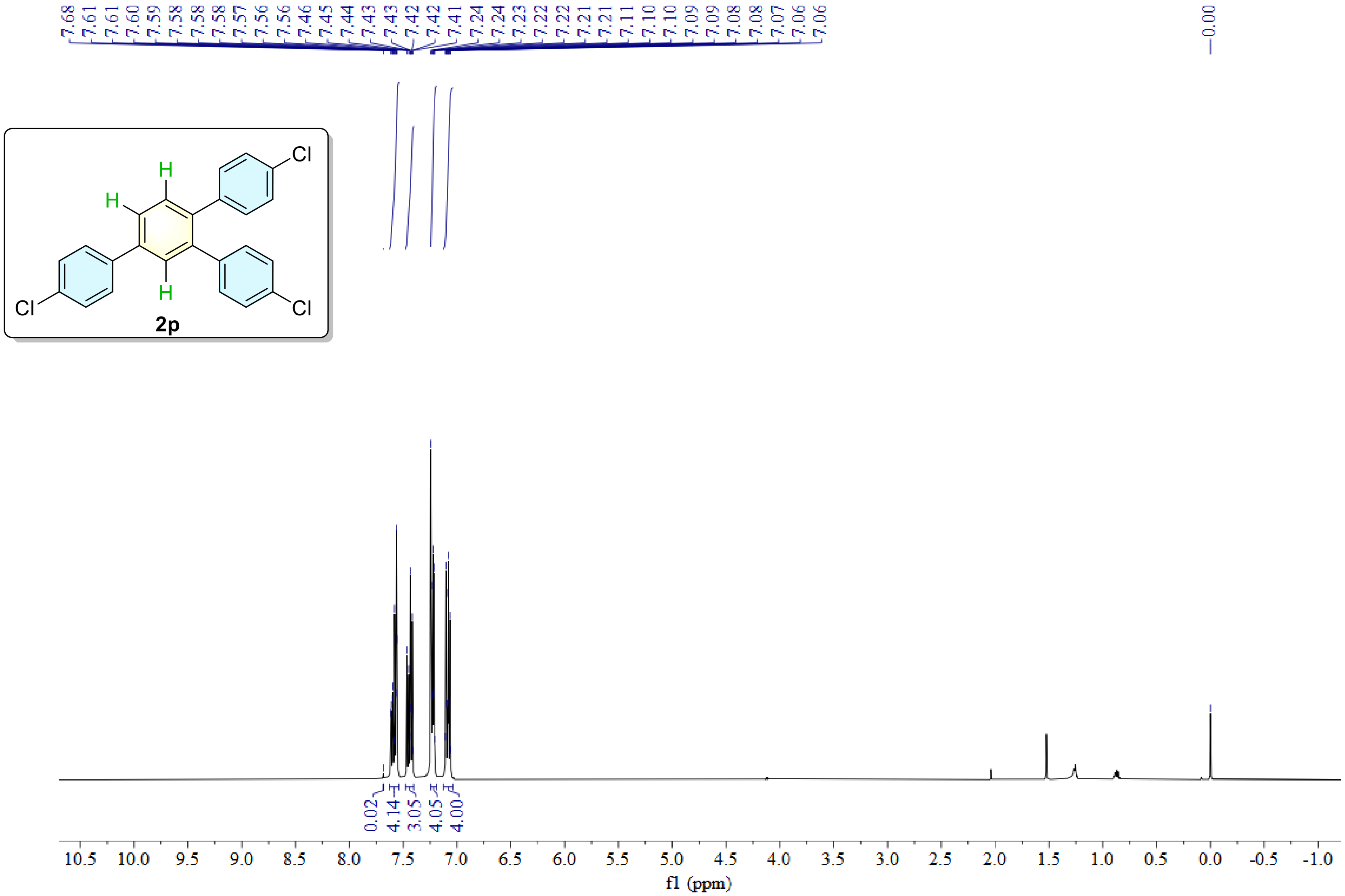
f1 (ppm)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7.43
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7.22
7.21
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7.09
7.08
7.08
7.07
7.06
7.06
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H
H
Cl
Cl
Cl
2p
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4.05
4.00
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4.0
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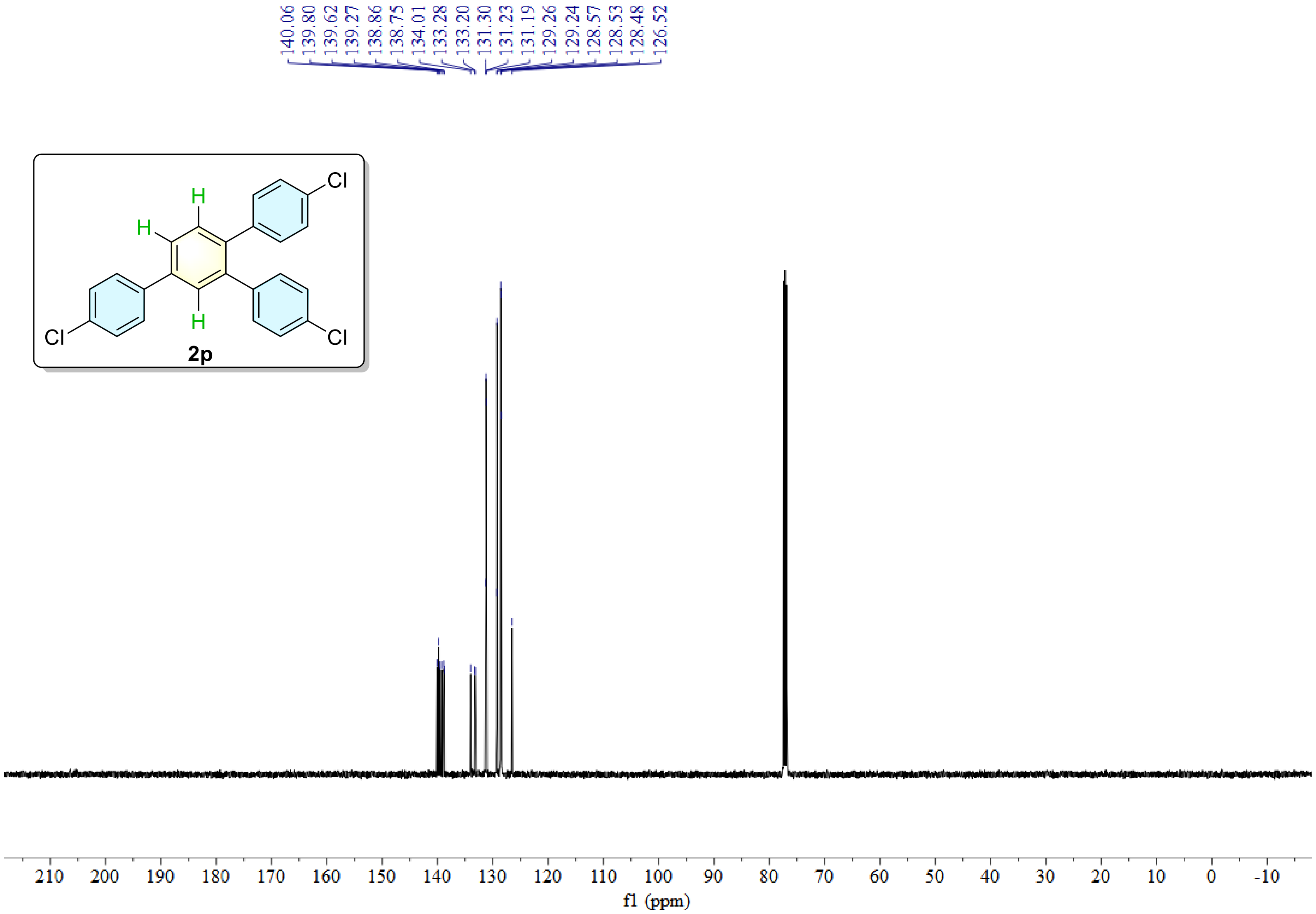
-1.0
f1 (ppm)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129.26
129.24
128.57
128.53
128.48
126.52
H
H
H
Cl
Cl
Cl
2p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
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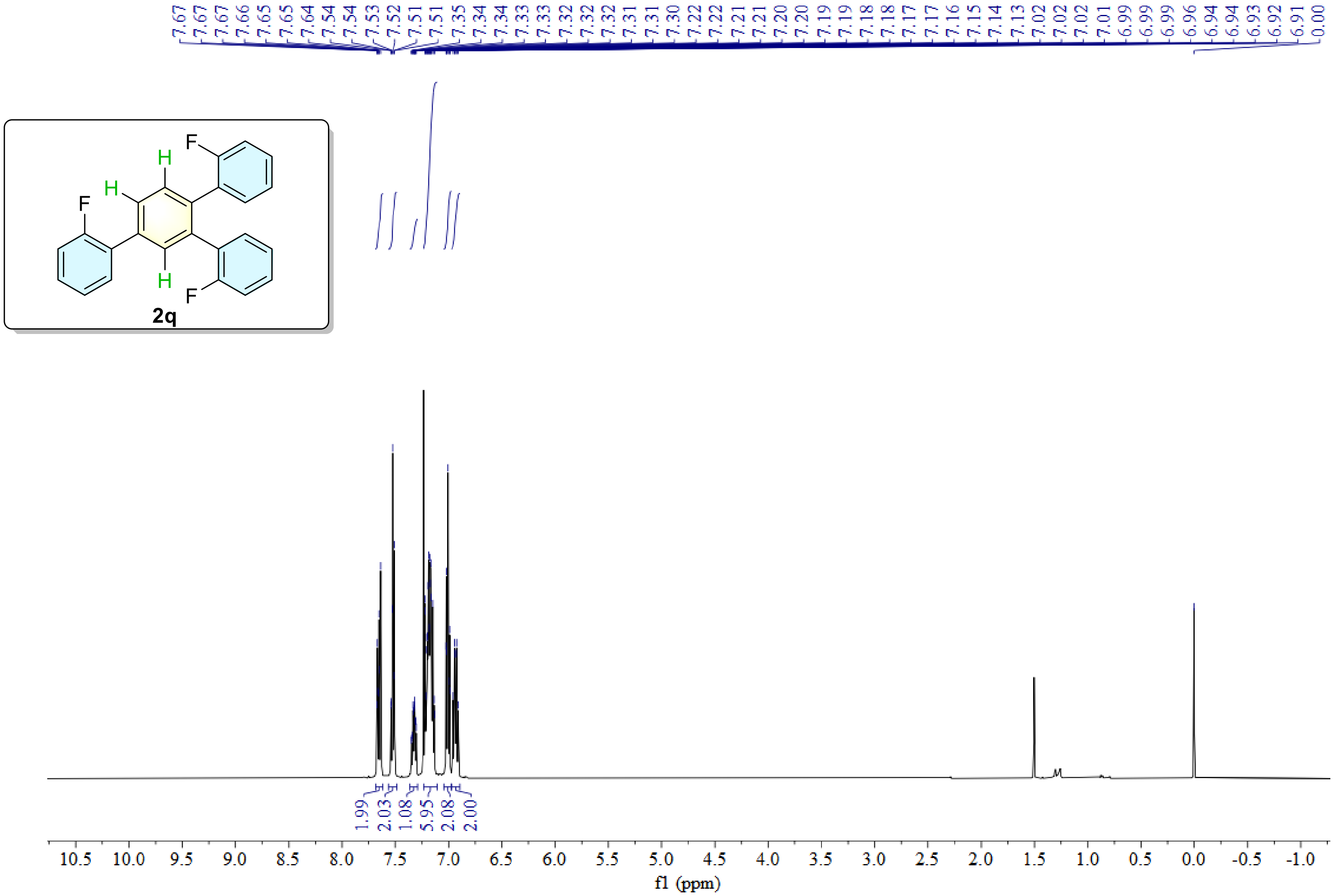
50
40
30
20
10
0
-10
f1 (ppm)


F
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H
F
H
F

2q

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7.14
7.13
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7.02
7.02
7.01
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6.99
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6.94
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6.92
6.91
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6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
1.5
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0.0
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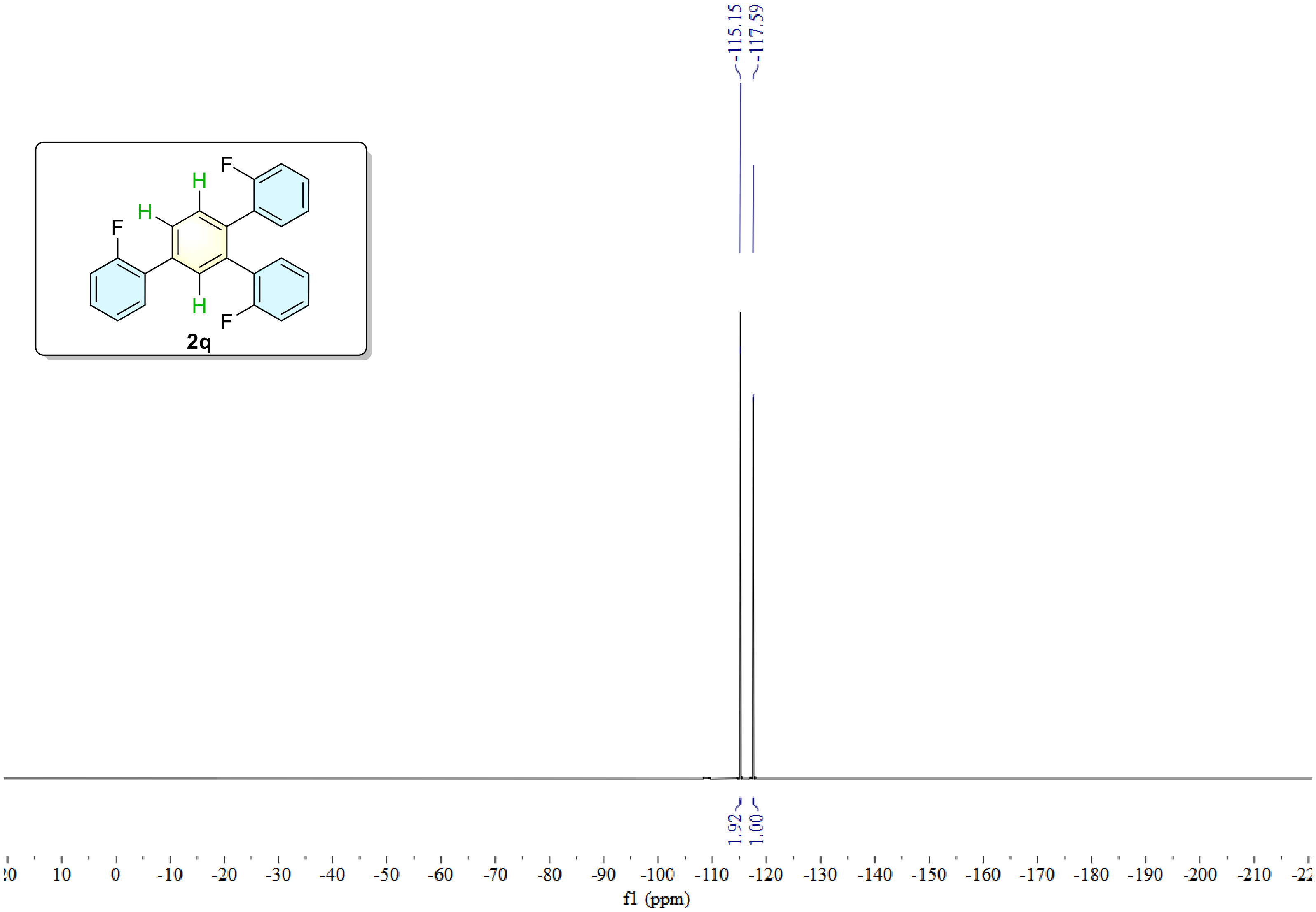
-1.0
f1 (ppm)



2q

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-117.59
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-80
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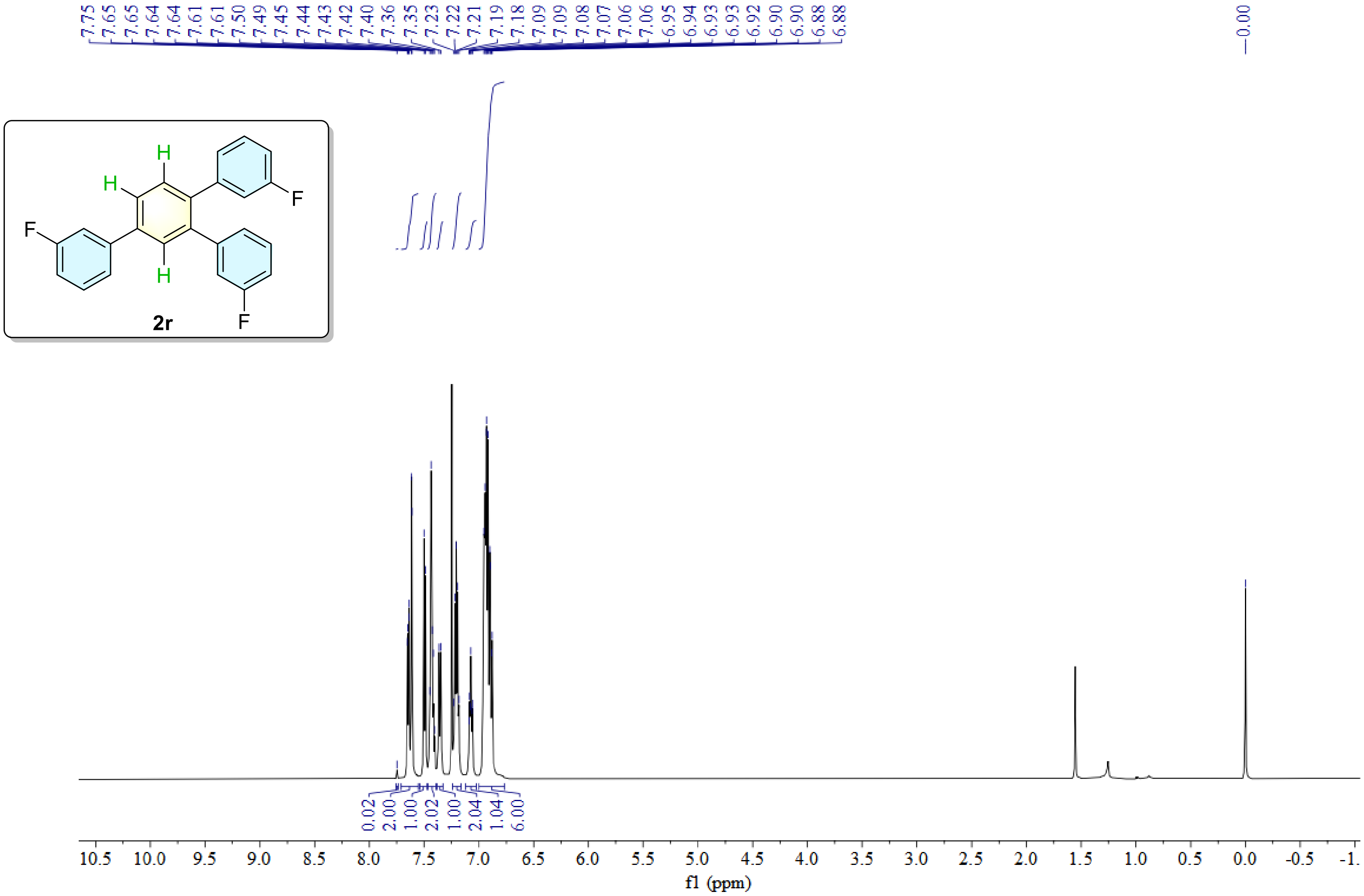
-190
-200
-210
f1 (ppm)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7.08
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7.06
7.06
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6.90
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H
H
F
F
F
2r
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2.02
1.00
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0.0
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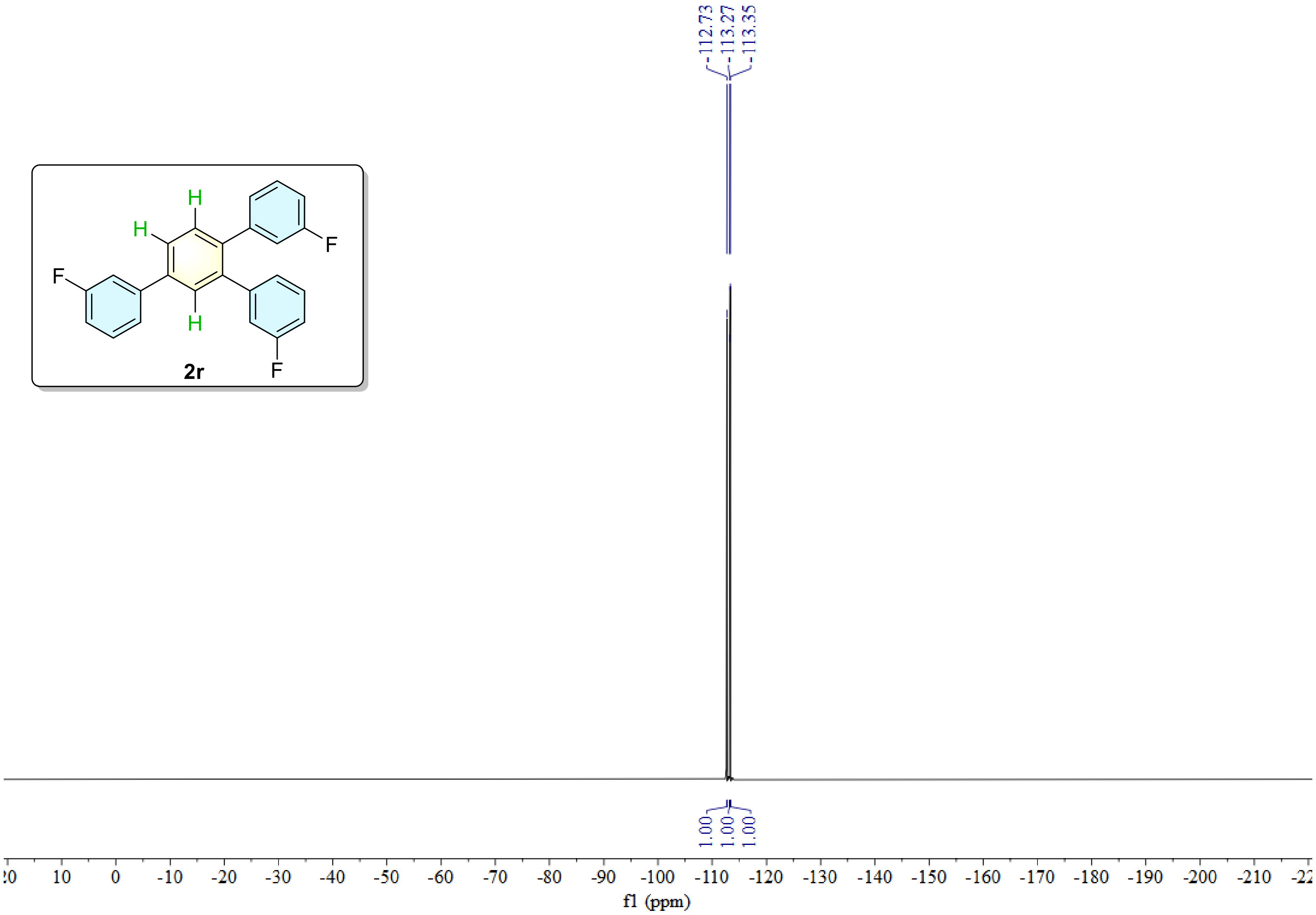
-1.
f1 (ppm)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H
H
F
F
F
2r
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1.00
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
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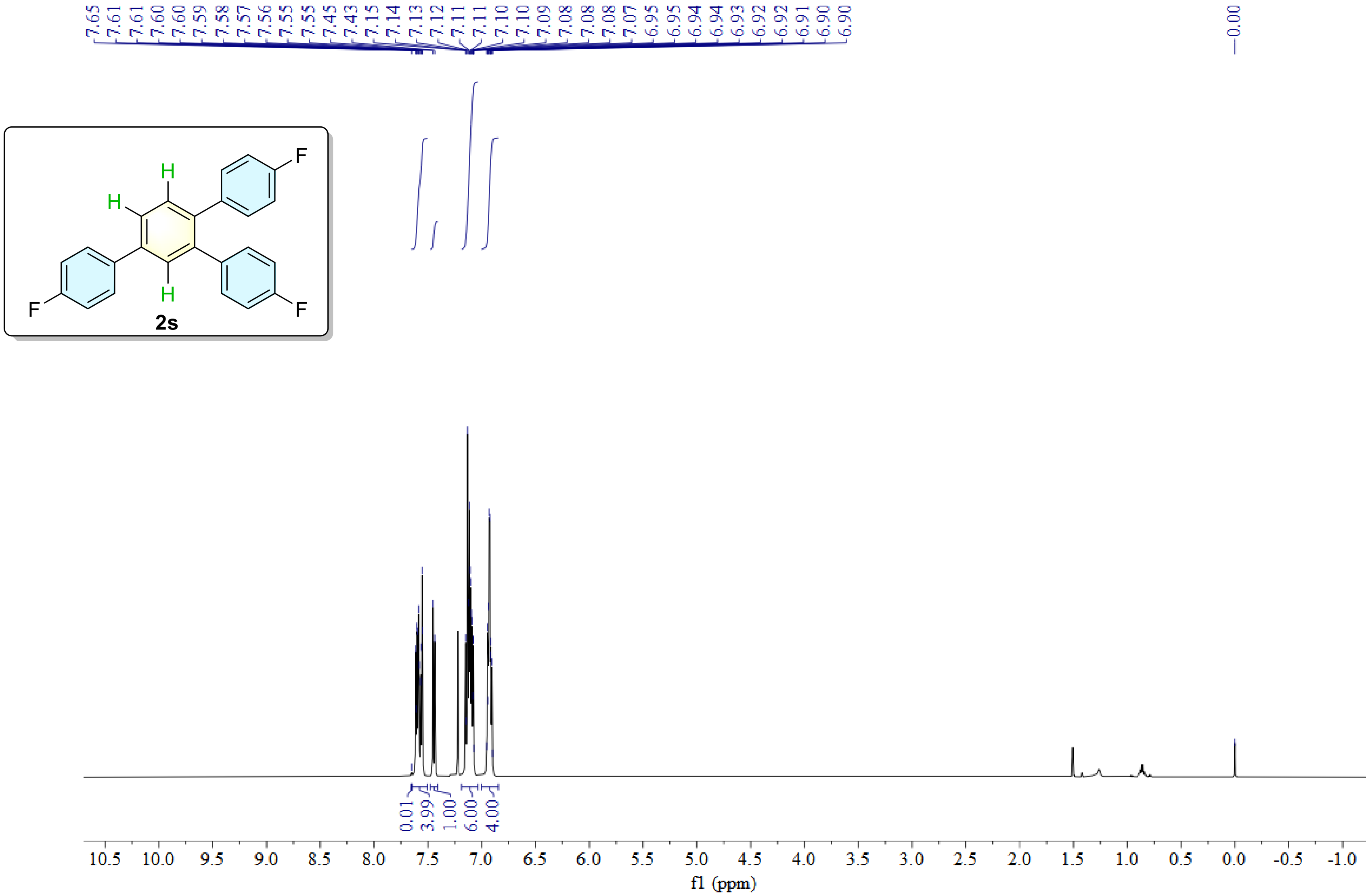
-200
-210
-220
f1 (ppm)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7.08
7.07
6.95
6.95
6.94
6.94
6.93
6.92
6.92
6.91
6.90
6.90
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H
H
H
F
F
F
2s
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3.99
1.00
6.00
4.00
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9.5
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7.5
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4.5
4.0
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2.0
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0.0
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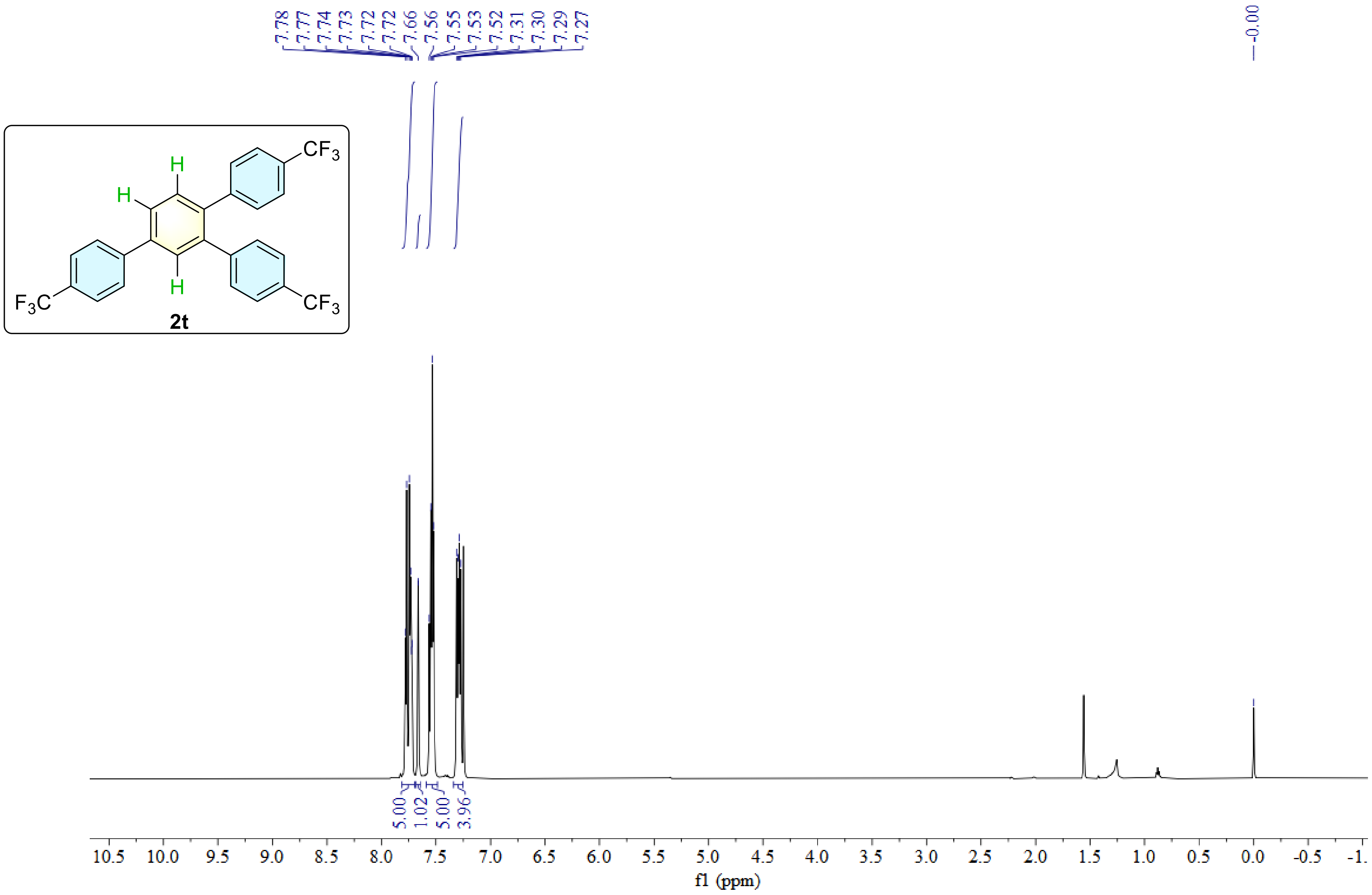
-1.0
f1 (ppm)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H
H
CF3
CF3
F3C
2t
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1.02
5.00
3.96
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
4.5
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2.5
2.0
1.5
1.0
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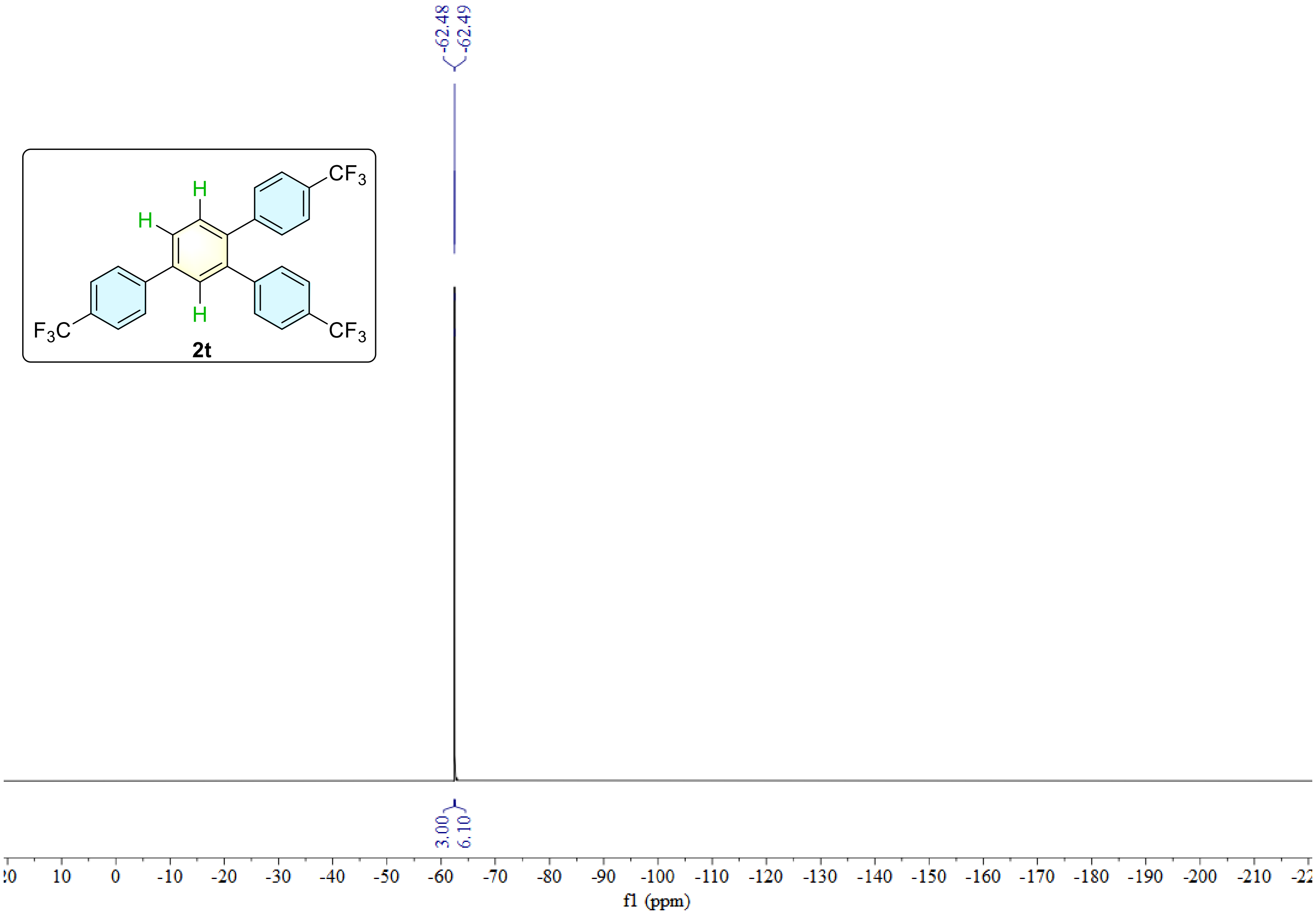
0.0
-0.5
-1.
f1 (ppm)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CF3
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H
H
F3C
CF3
2t
3.00
6.10
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
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-120
-130
-140
-150
-160
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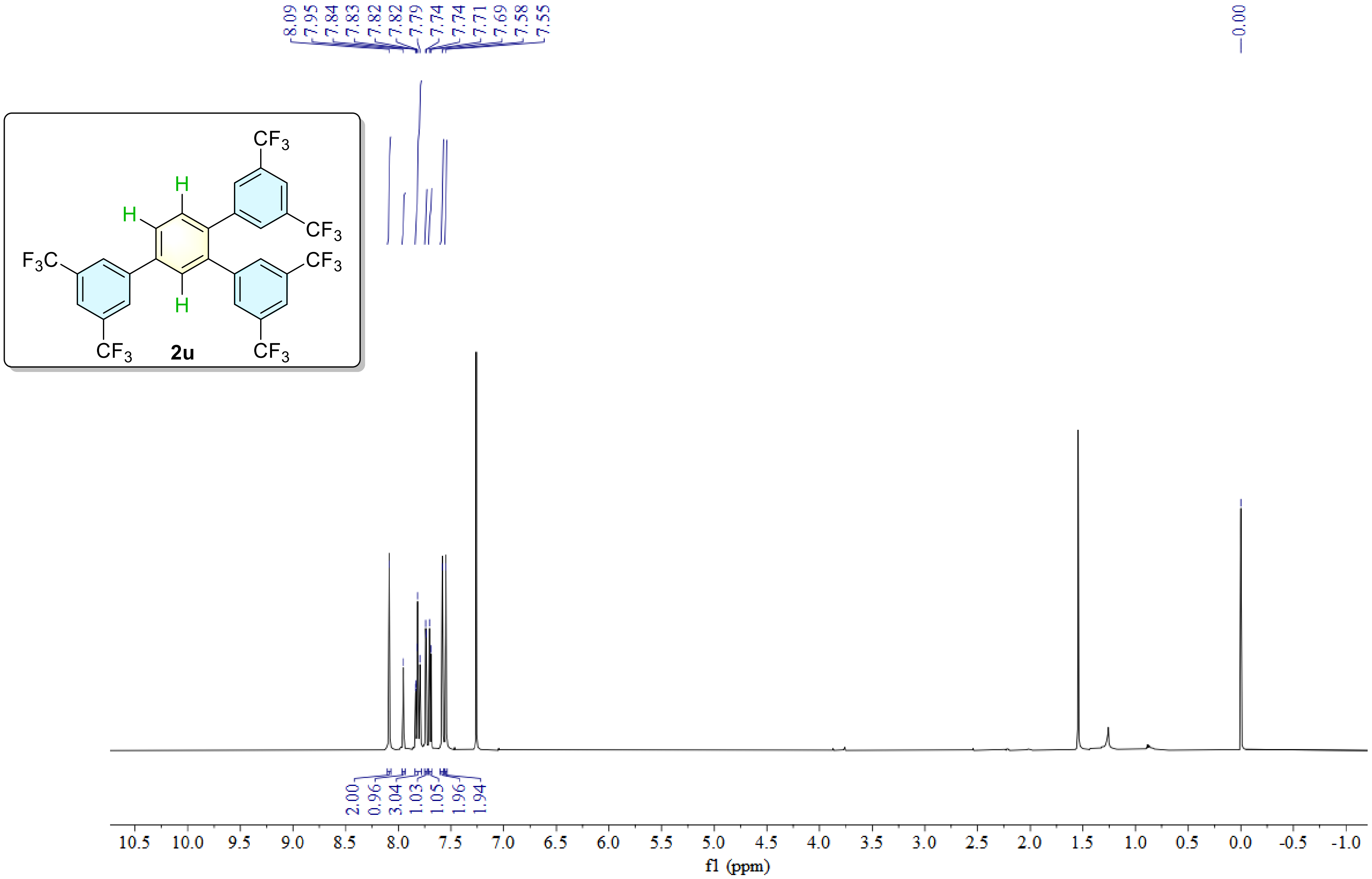
-200
-210
-22
f1 (ppm)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7.55
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CF3
CF3
F3C
CF3
CF3
CF3
H
H
H
2u
2.00
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1.05
1.96
1.94
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10.0
9.5
9.0
8.5
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7.5
7.0
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6.0
5.5
5.0
4.5
4.0
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2.0
1.5
1.0
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0.0
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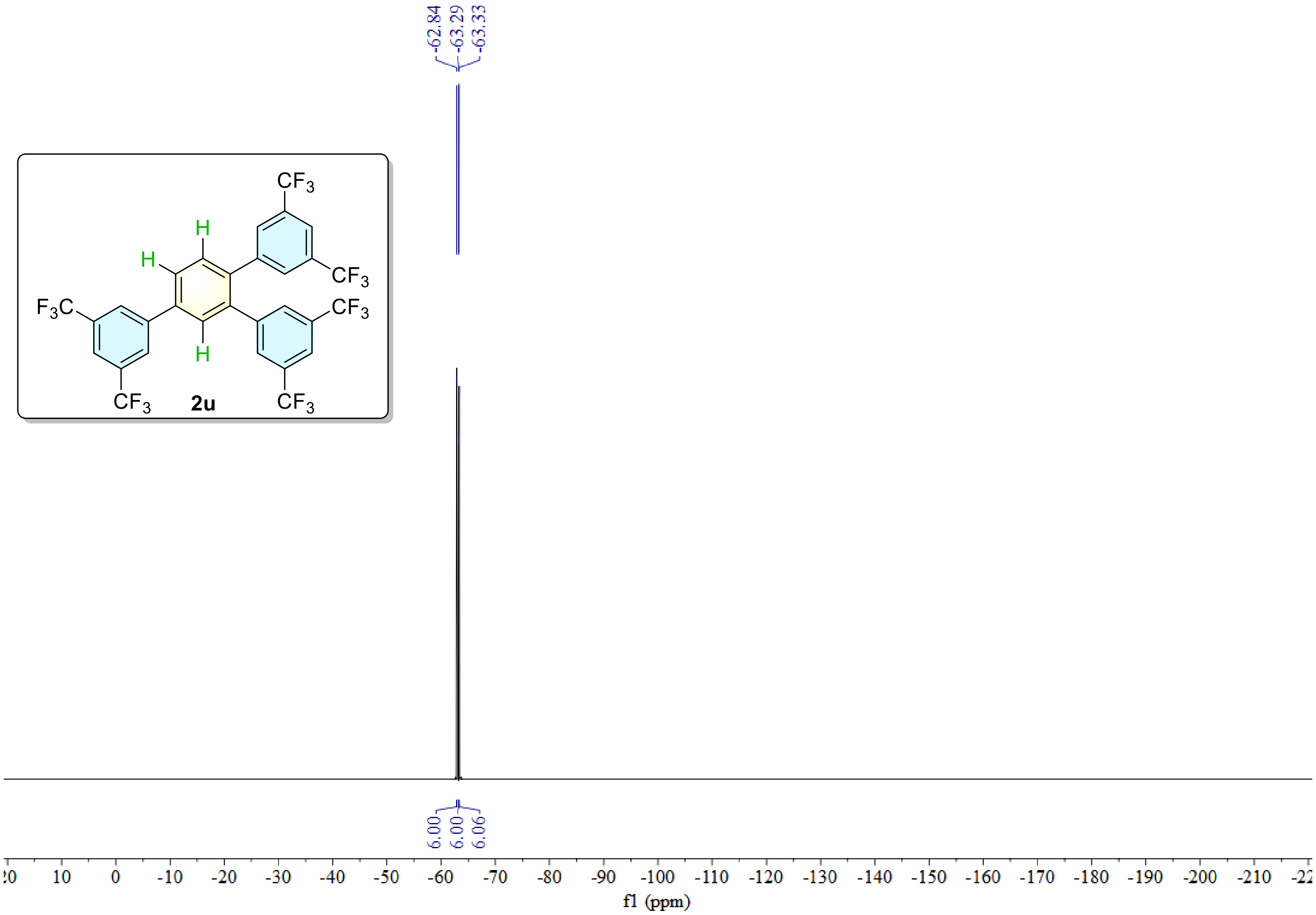
-1.0
f1 (ppm)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CF3
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H
F3C
CF3
CF3
H
CF3
CF3
2u
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6.00
6.06
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
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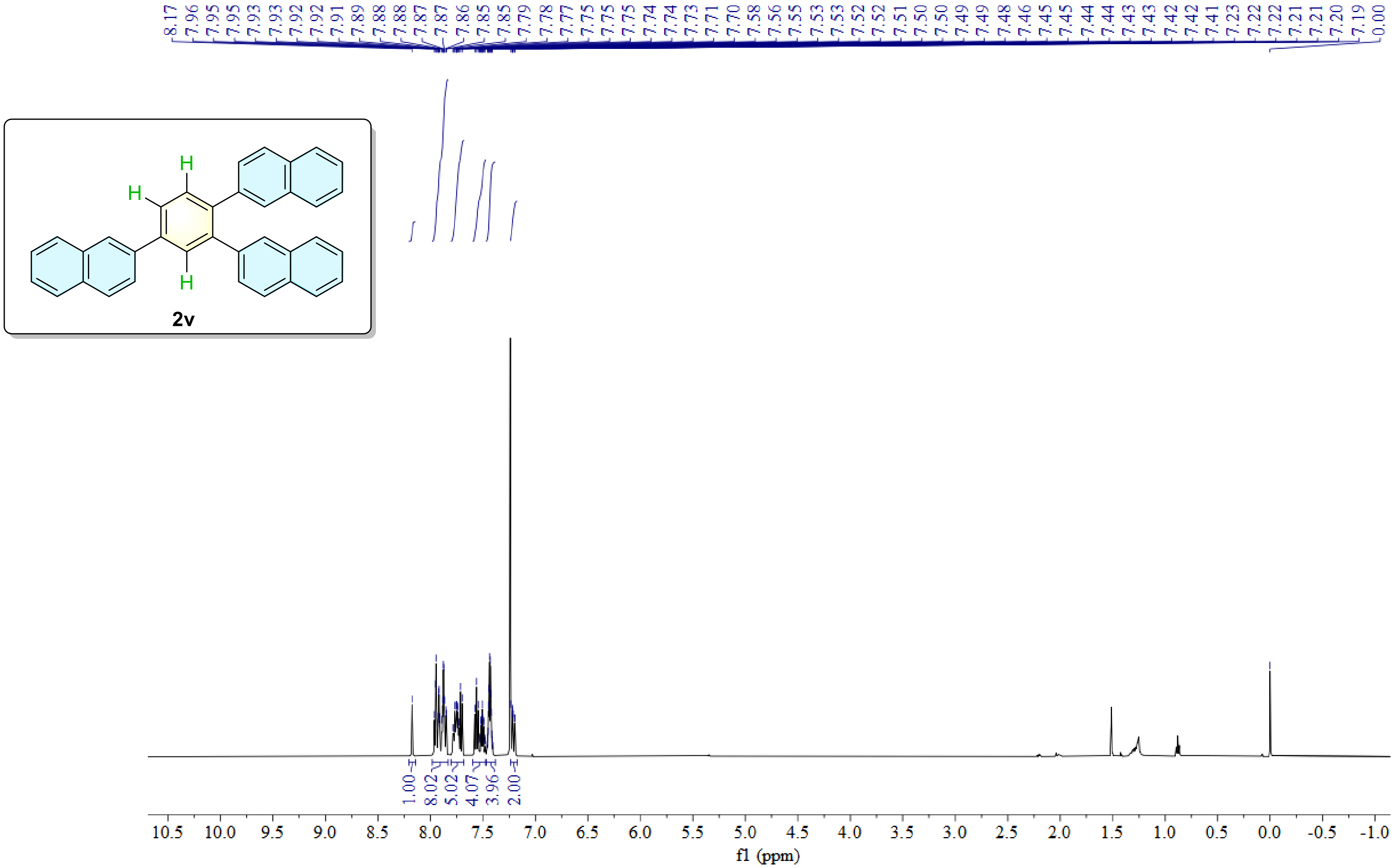
-130
-140
-150
-160
-170
-180
-190
-200
-210
-22
f1 (ppm)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H
H
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2.00
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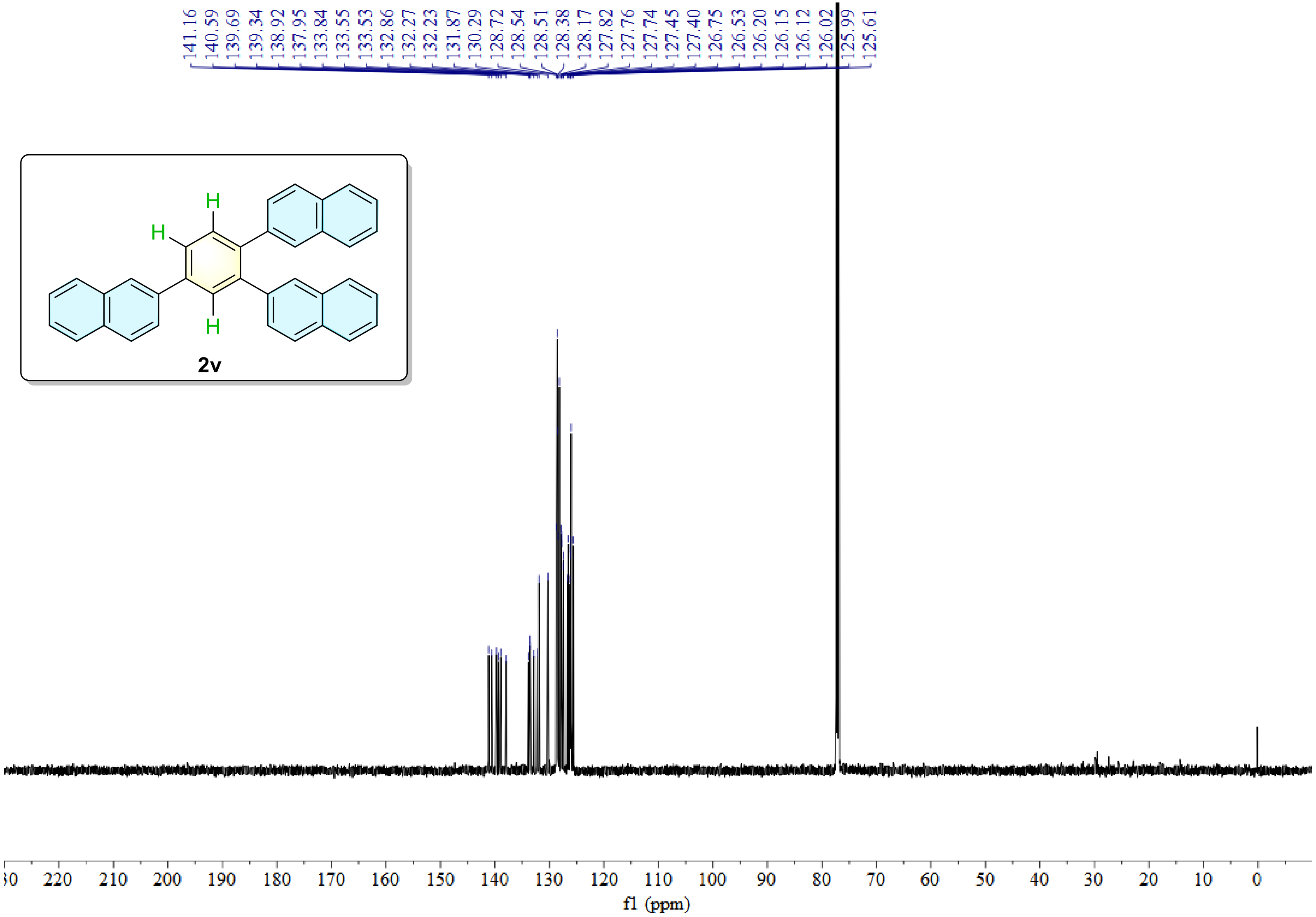
-1.0
f1 (ppm)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126.20
126.15
126.12
126.02
125.99
125.61
H
H
H
2v
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200
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140
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110
100
90
80
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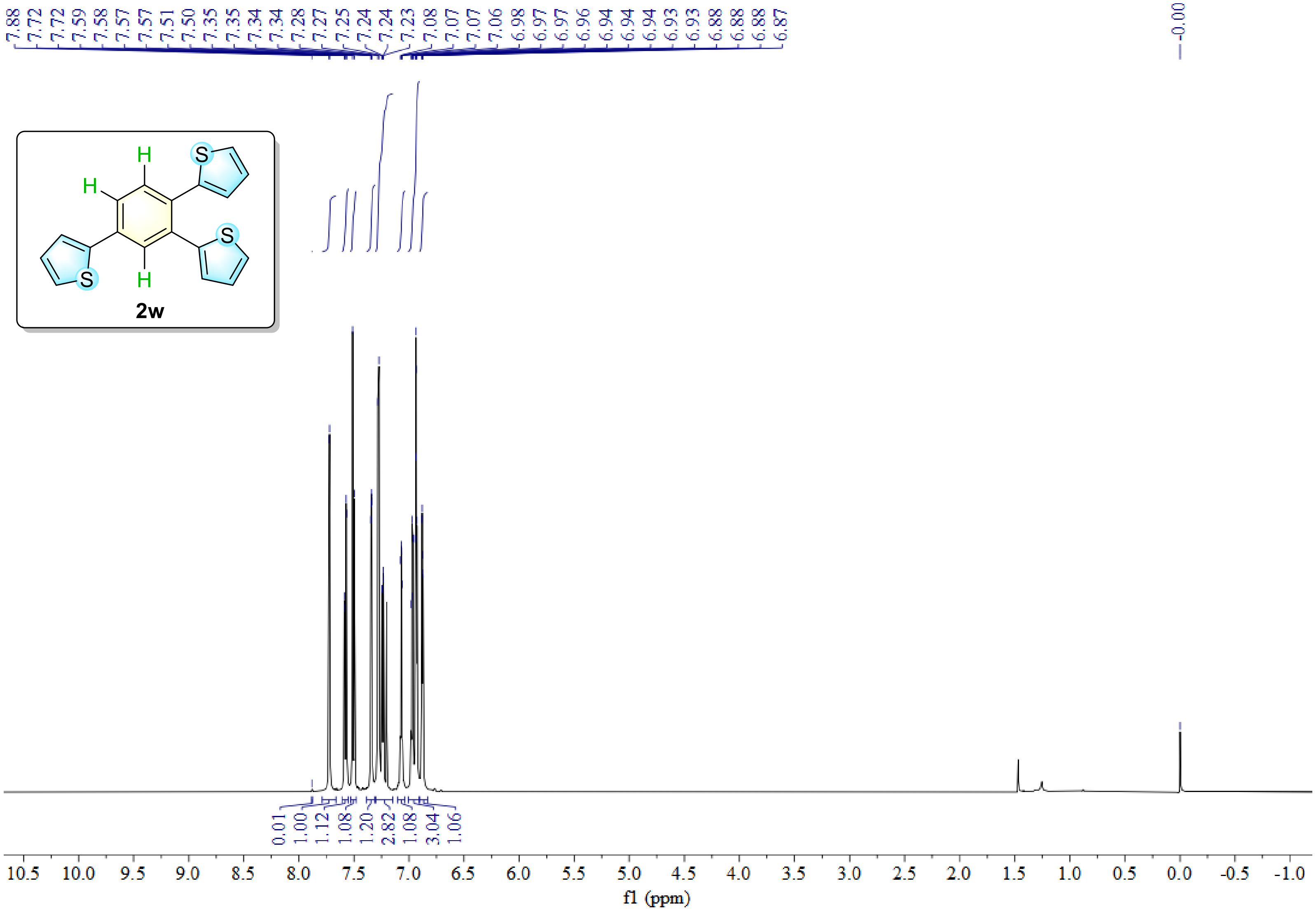
0
f1 (ppm)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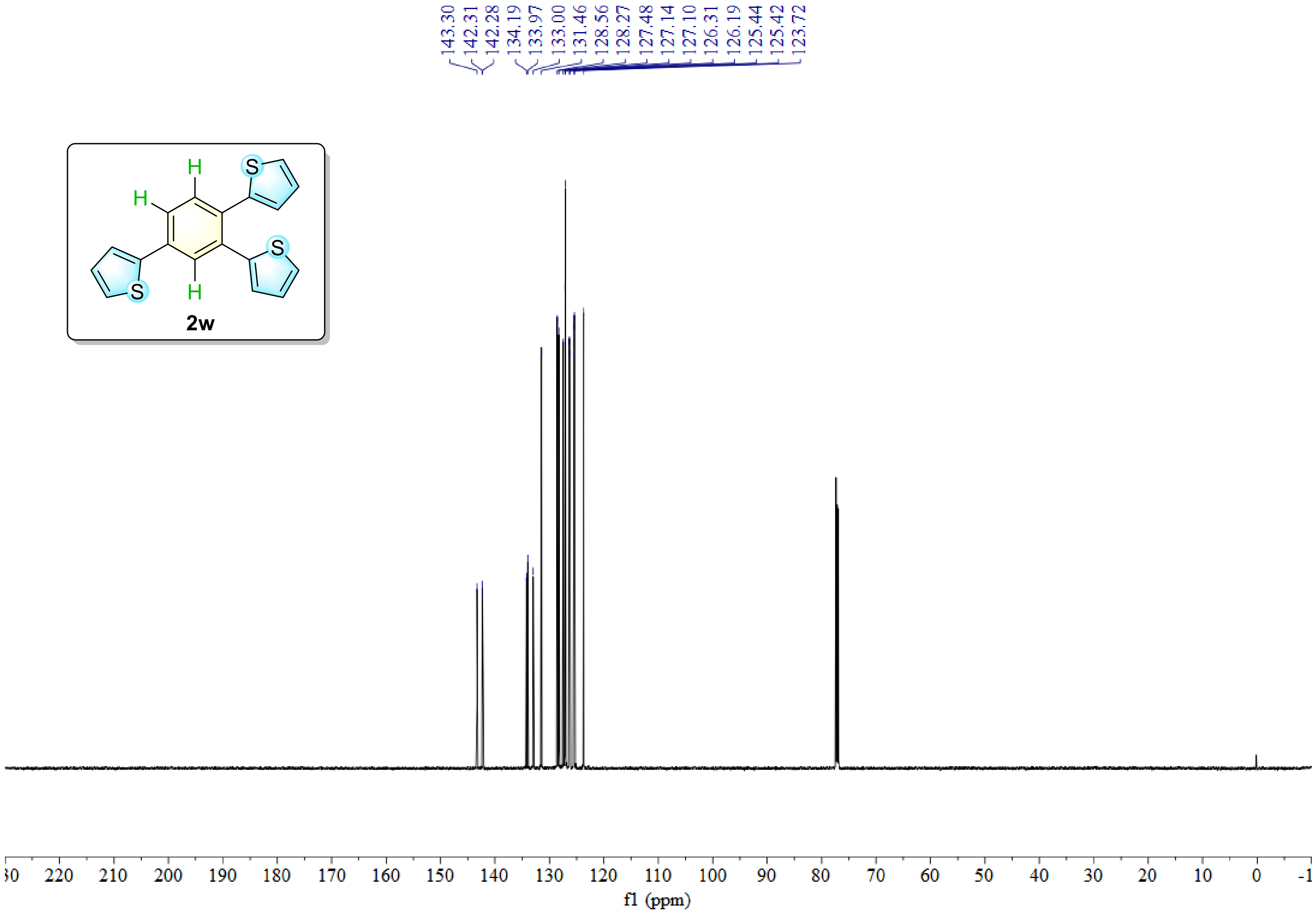
7.72
7.72
7.59
7.58
7.57
7.57
7.51
7.50
7.35
7.35
7.34
7.34
7.28
7.27
7.25
7.24
7.24
7.23
7.08
7.07
7.07
7.06
6.98
6.97
6.97
6.96
6.94
6.94
6.94
6.93
6.93
6.88
6.88
6.88
6.87
-0.00
H
H
H
S
S
S
2w
0.01
1.00
1.12
1.08
1.20
2.82
1.08
3.04
1.06
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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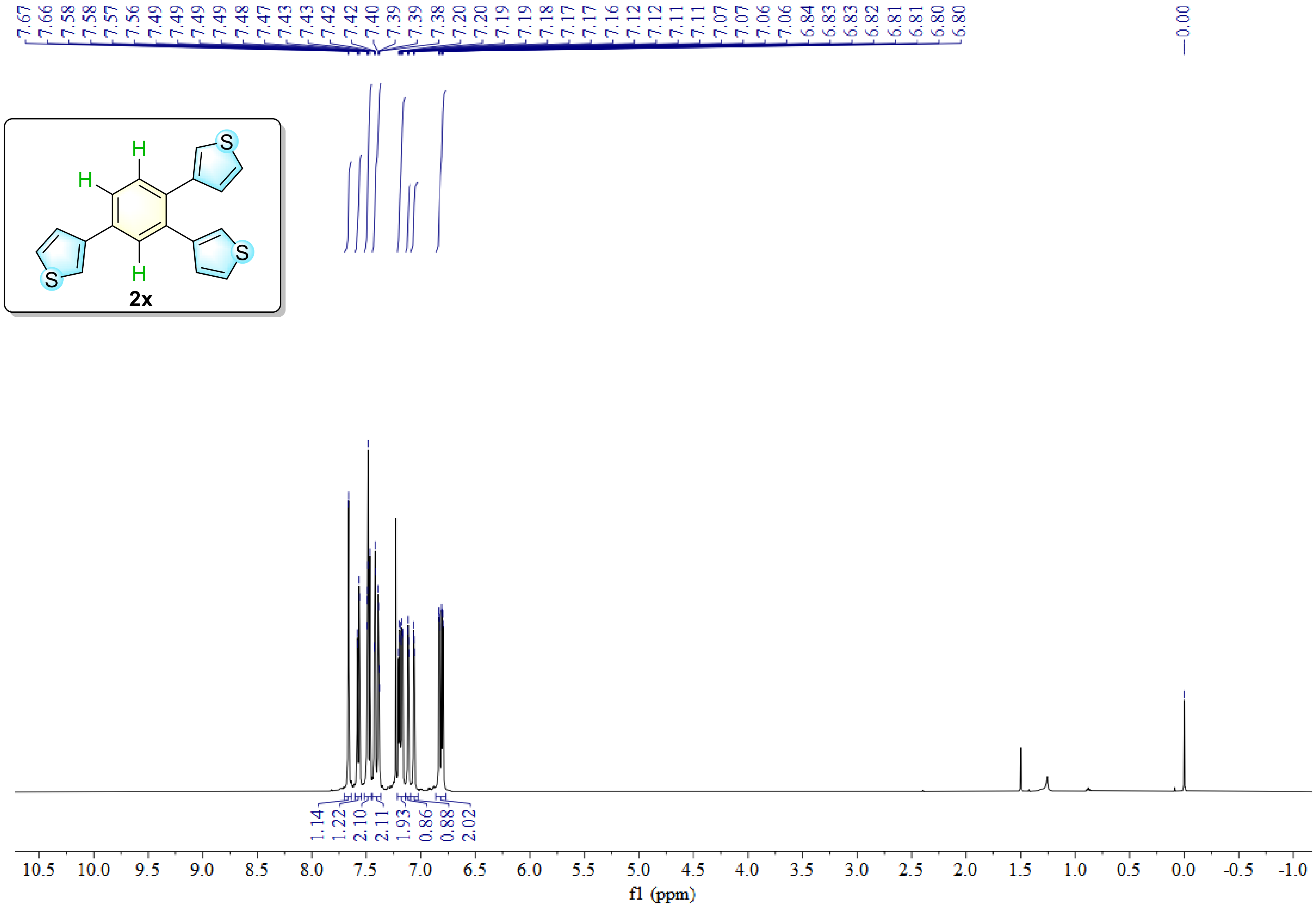
142.28
134.19
133.97
133.00
131.46
128.56
128.27
127.48
127.14
127.10
126.31
126.19
125.44
125.42
123.72
H
H
H
S
S
S
2w
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


7.67
7.66
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7.58
7.57
7.56
7.49
7.49
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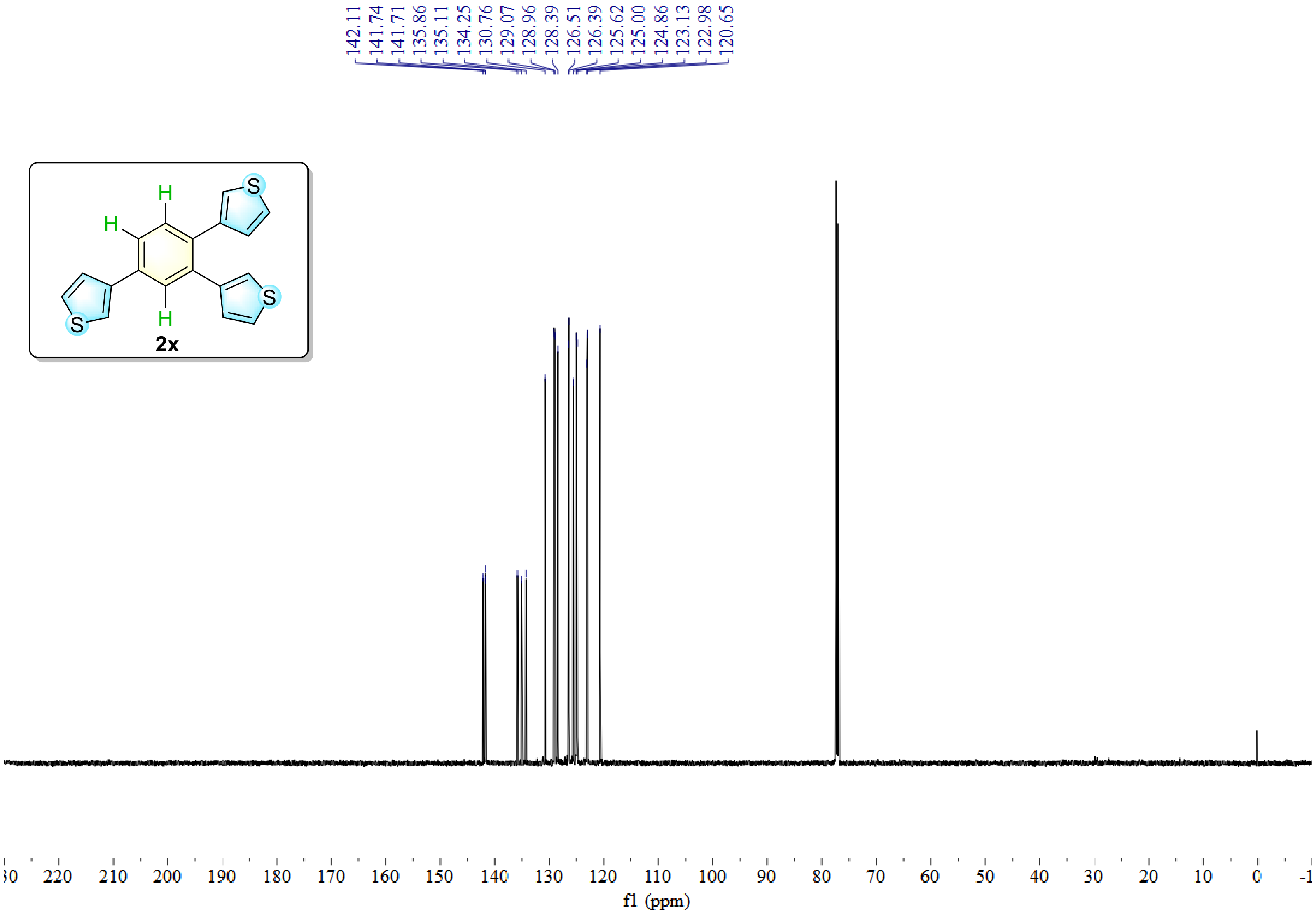
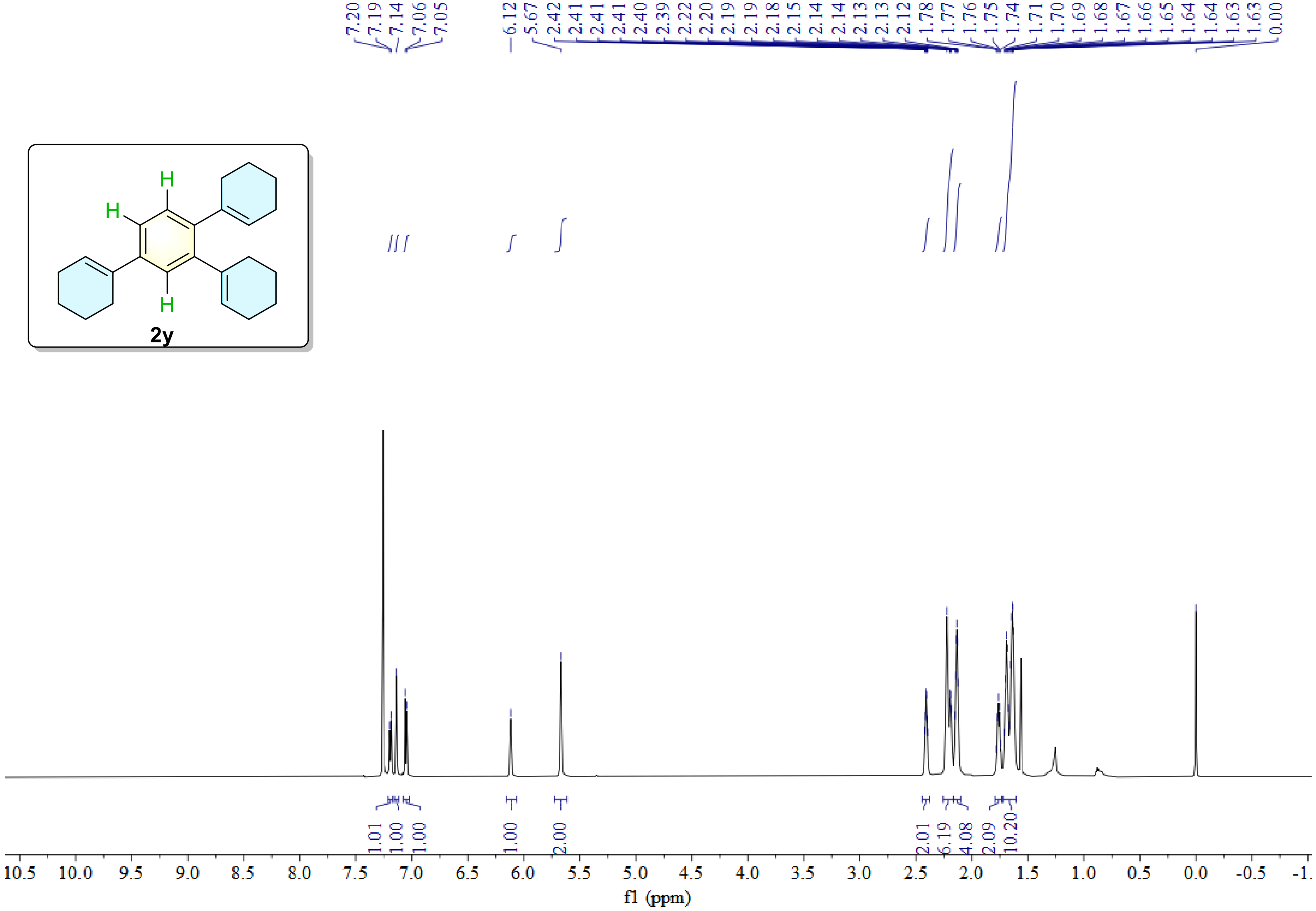
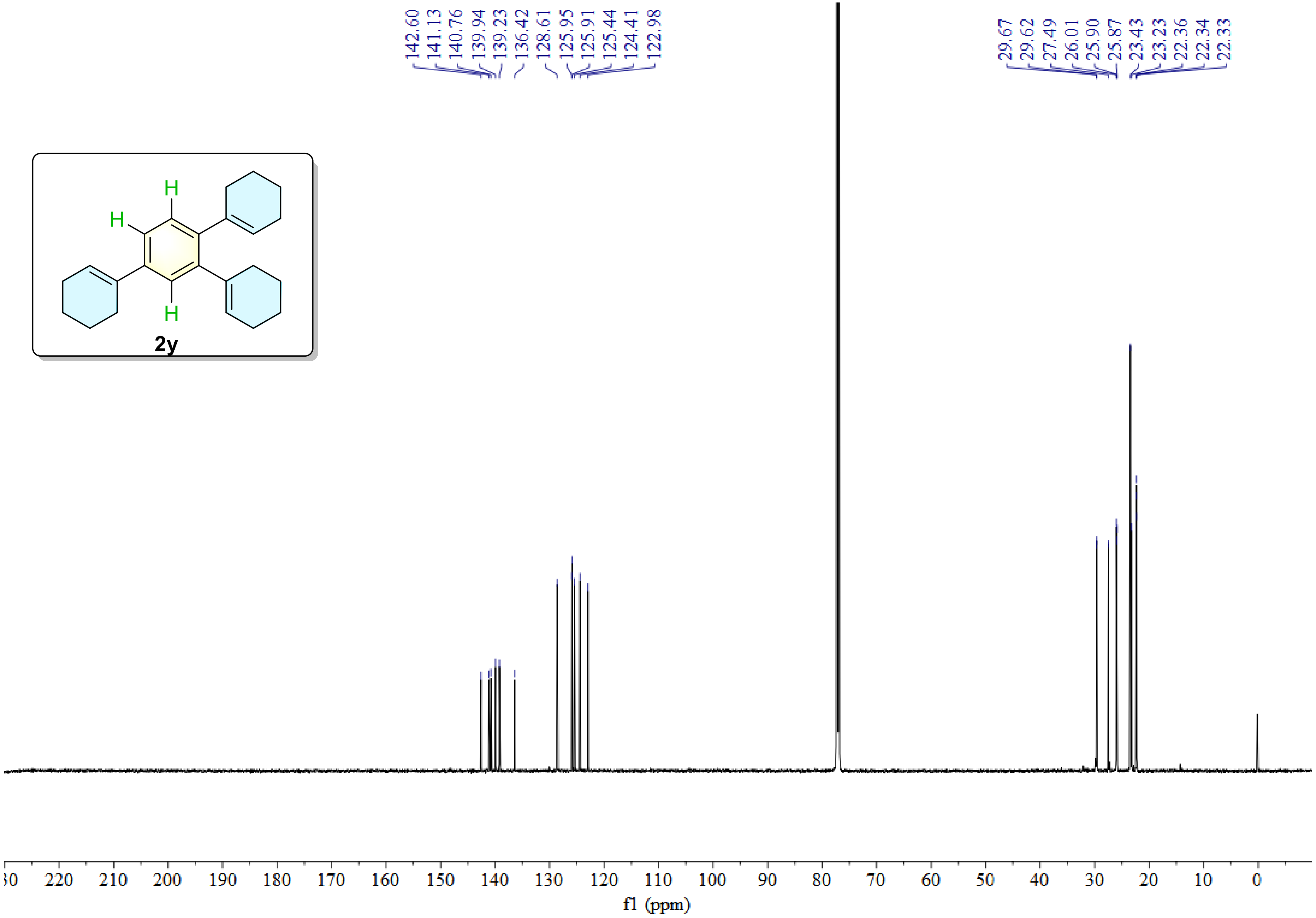
7.49
7.48
7.47
7.43
7.43
7.42
7.42
7.40
7.39
7.39
7.38
7.20
7.20
7.19
7.19
7.18
7.17
7.17
7.16
7.12
7.12
7.11
7.11
7.07
7.07
7.06
7.06
6.84
6.83
6.83
6.82
6.81
6.81
6.80
6.80
-0.00
H
H
H
S
S
S
1.14
1.22
2.10
2.11
1.93
0.86
0.88
2.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

2x


142.11
141.74
141.71
135.86
135.11
134.25
130.76
129.07
128.96
128.39
126.51
126.39
125.62
125.00
124.86
123.13
122.98
120.65
H
H
H
S
S
S
2x
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

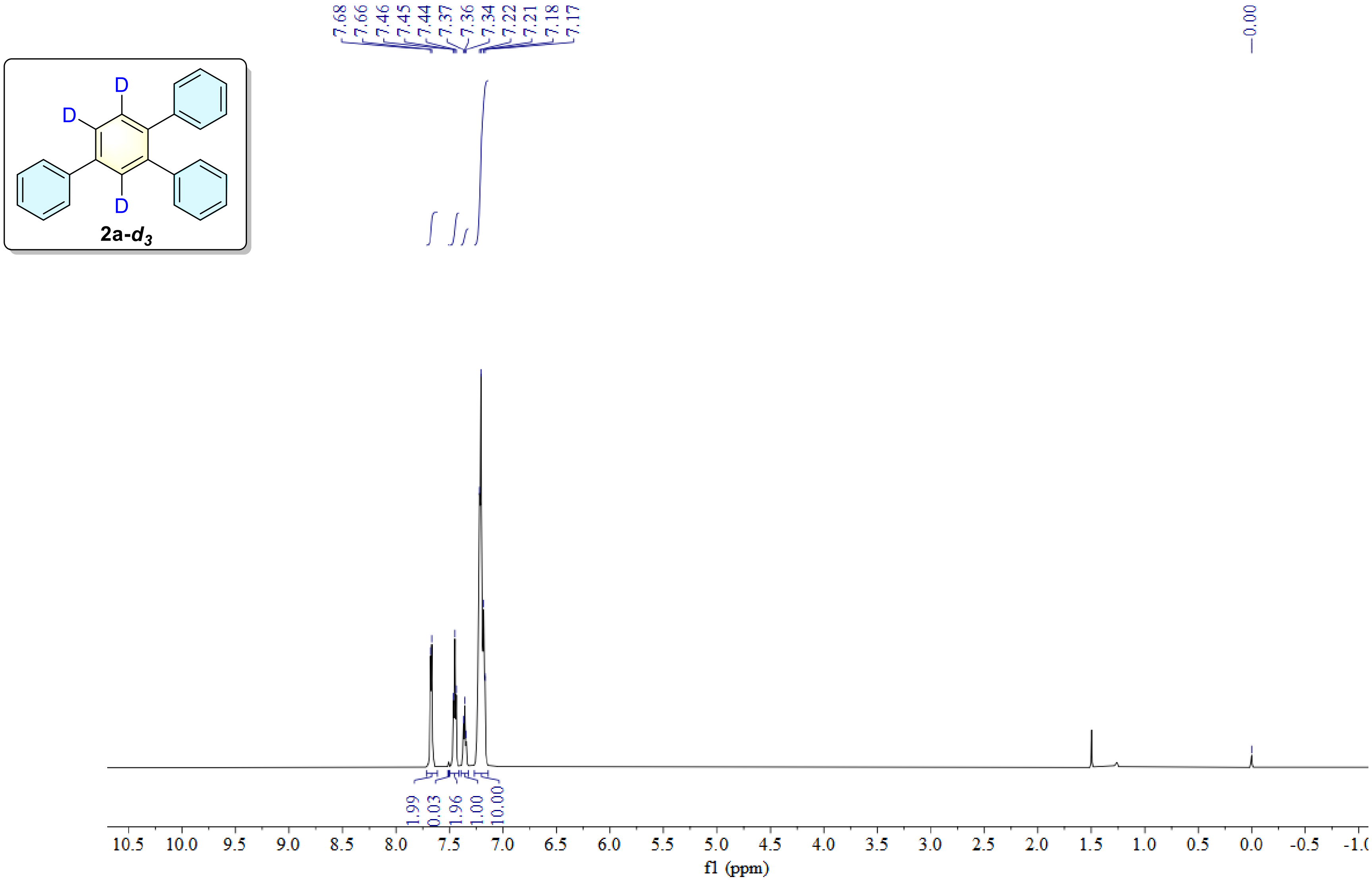


2y
7.20
7.19
7.14
7.06
7.05
6.12
5.67
2.42
2.41
2.41
2.41
2.40
2.39
2.22
2.20
2.19
2.19
2.18
2.15
2.14
2.14
2.13
2.13
2.12
1.78
1.77
1.76
1.75
1.74
1.71
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.64
1.63
1.63
0.00
1.01
1.00
1.00
1.00
2.00
2.01
6.19
4.08
2.09
10.20
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)



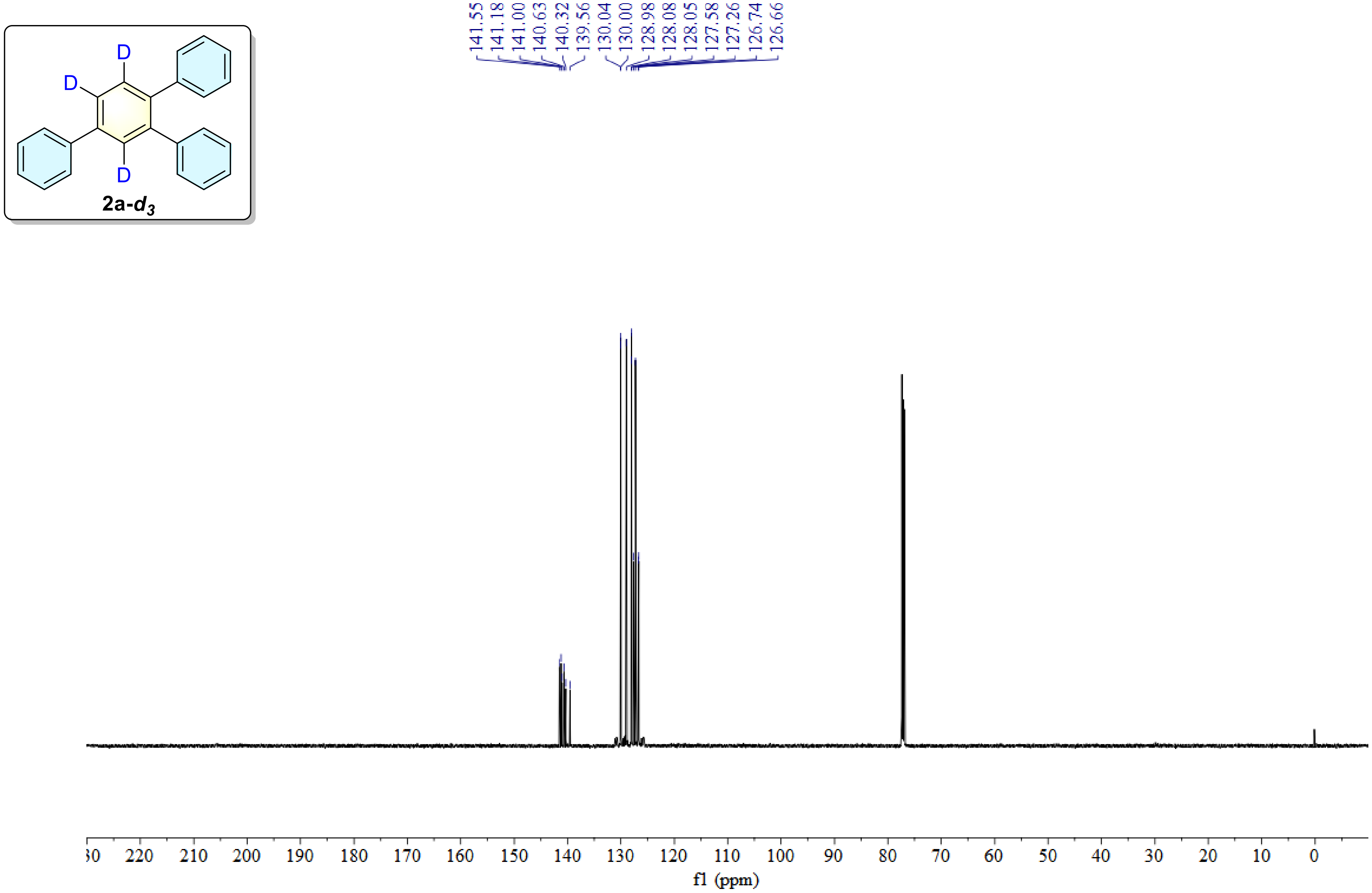
2y

142.60
141.13
140.76
139.94
139.23
136.42
128.61
125.95
125.91
125.44
124.41
122.98
29.67
29.62
27.49
26.01
25.90
25.87
23.43
23.23
22.36
22.34
22.33
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D
D
D

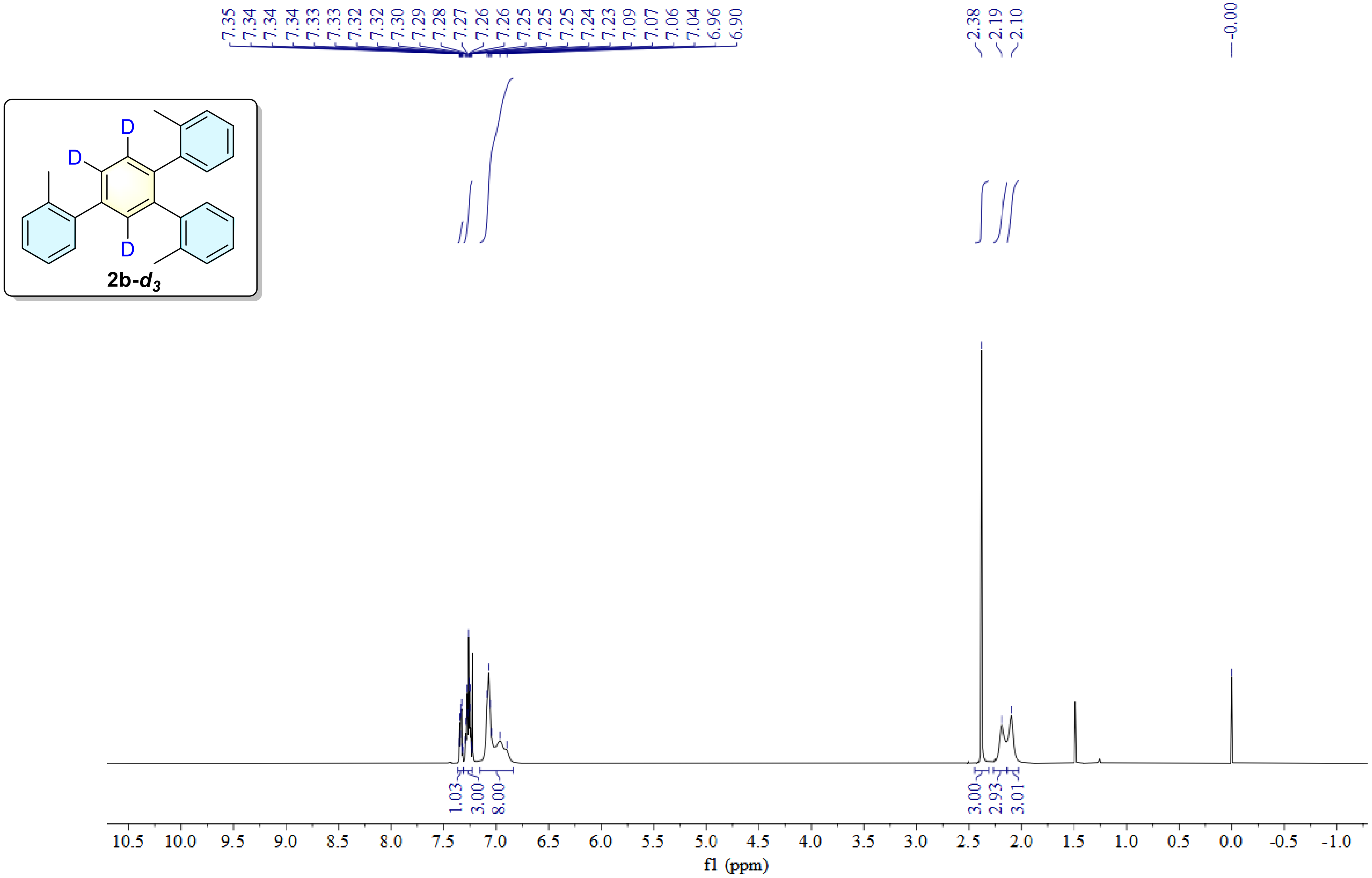
2a-*d*$_3$

7.68
7.66
7.46
7.45
7.44
7.37
7.36
7.34
7.22
7.21
7.18
7.17
0.00
1.99
0.03
1.96
1.00
10.00
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
-0.5
-1.0
f1 (ppm)


D
D
D

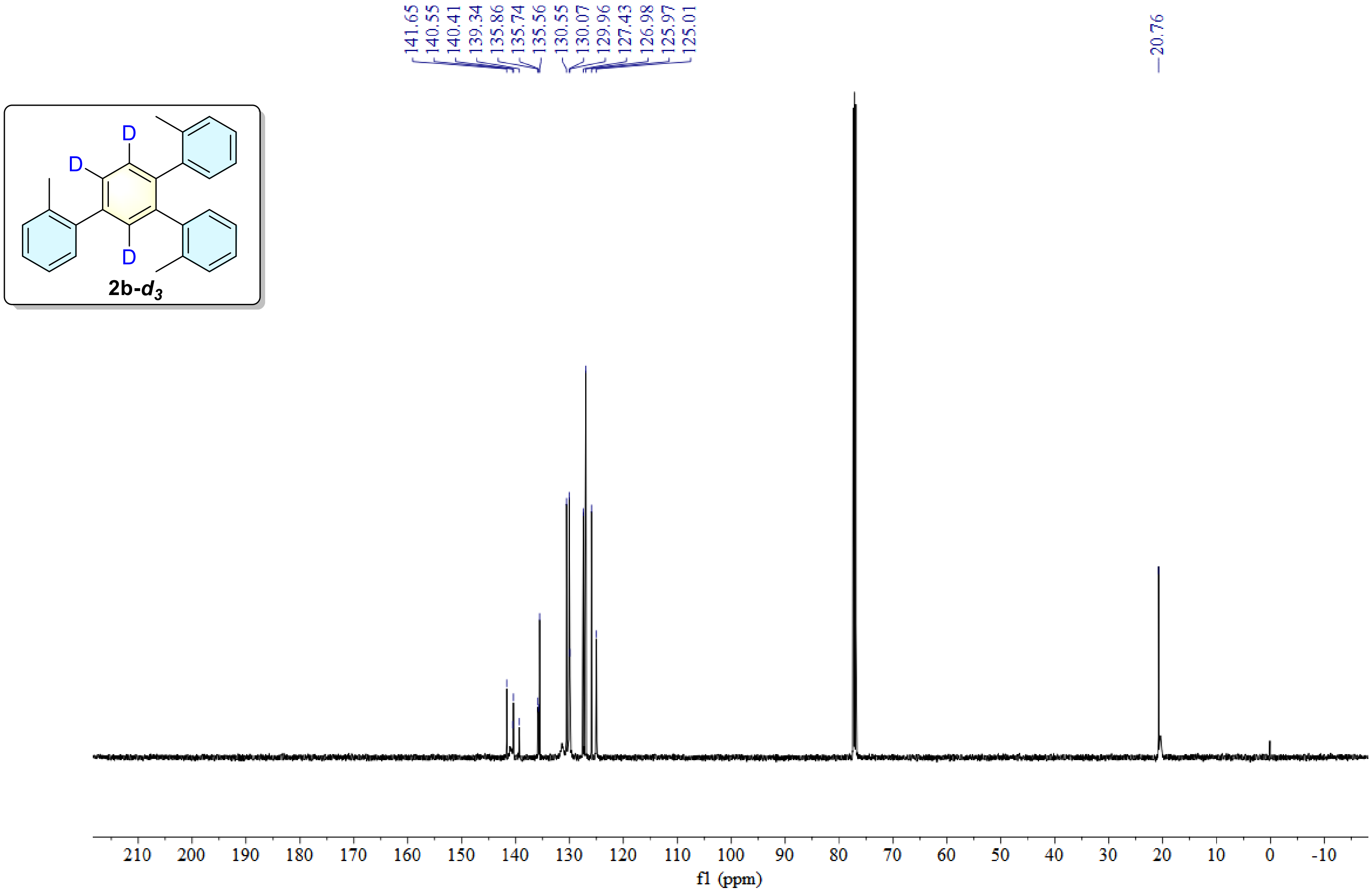
2a-d_3

141.55
141.18
141.00
140.63
140.32
139.56
130.04
130.00
128.98
128.08
128.05
127.58
127.26
126.74
126.66
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



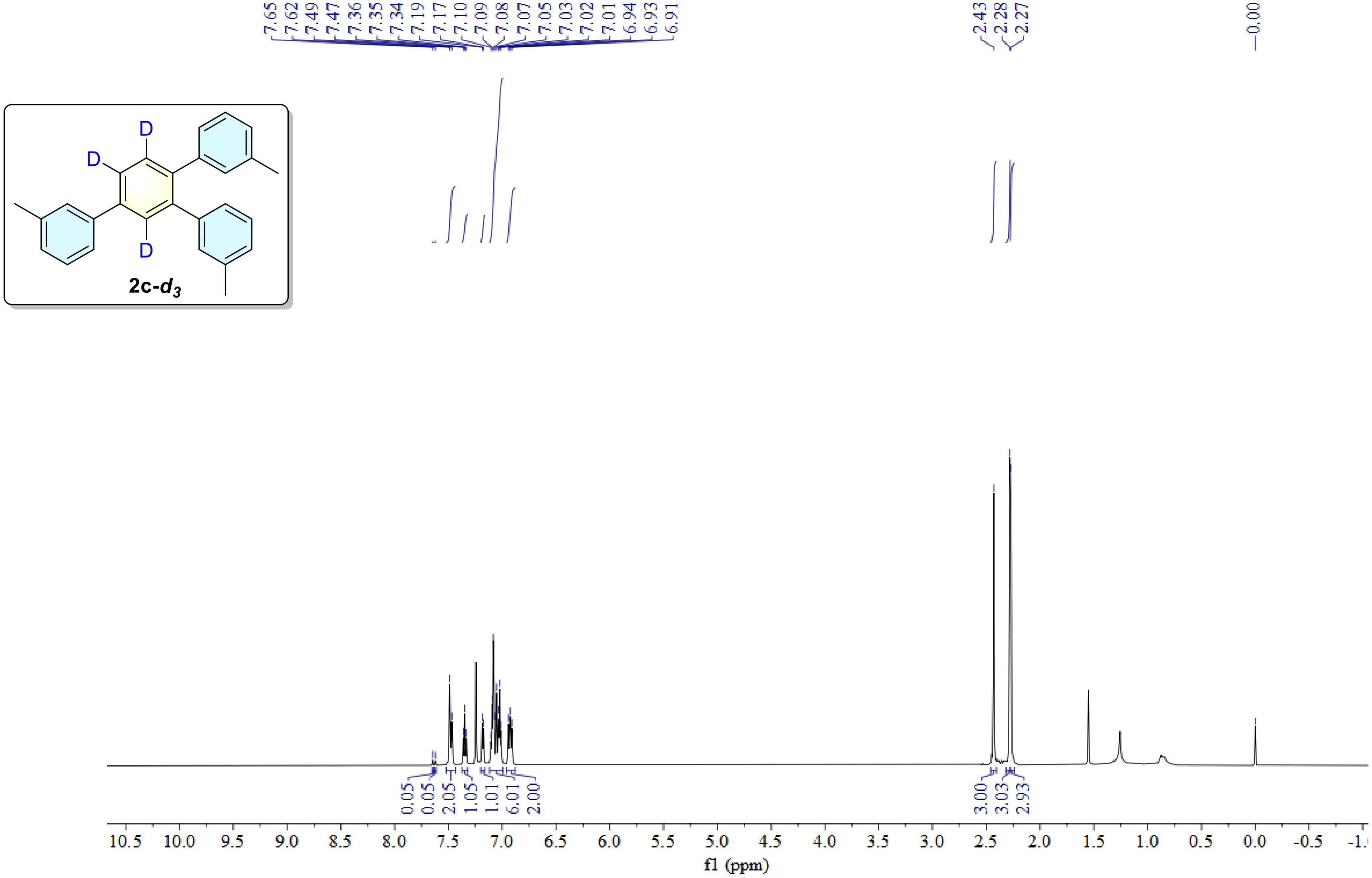
2b-d_3

D
D
D
7.35
7.34
7.34
7.34
7.33
7.33
7.32
7.32
7.30
7.29
7.28
7.27
7.26
7.26
7.25
7.25
7.25
7.24
7.23
7.09
7.07
7.06
7.04
6.96
6.90
2.38
2.19
2.10
-0.00
1.03
3.00
8.00
3.00
2.93
3.01
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
-0.5
-1.0
f1 (ppm)



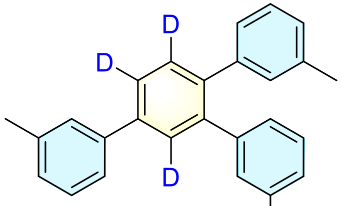
2b-*d*$_3$

141.65
140.55
140.41
139.34
135.86
135.74
135.56
130.55
130.07
129.96
127.43
126.98
125.97
125.01
20.76
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)

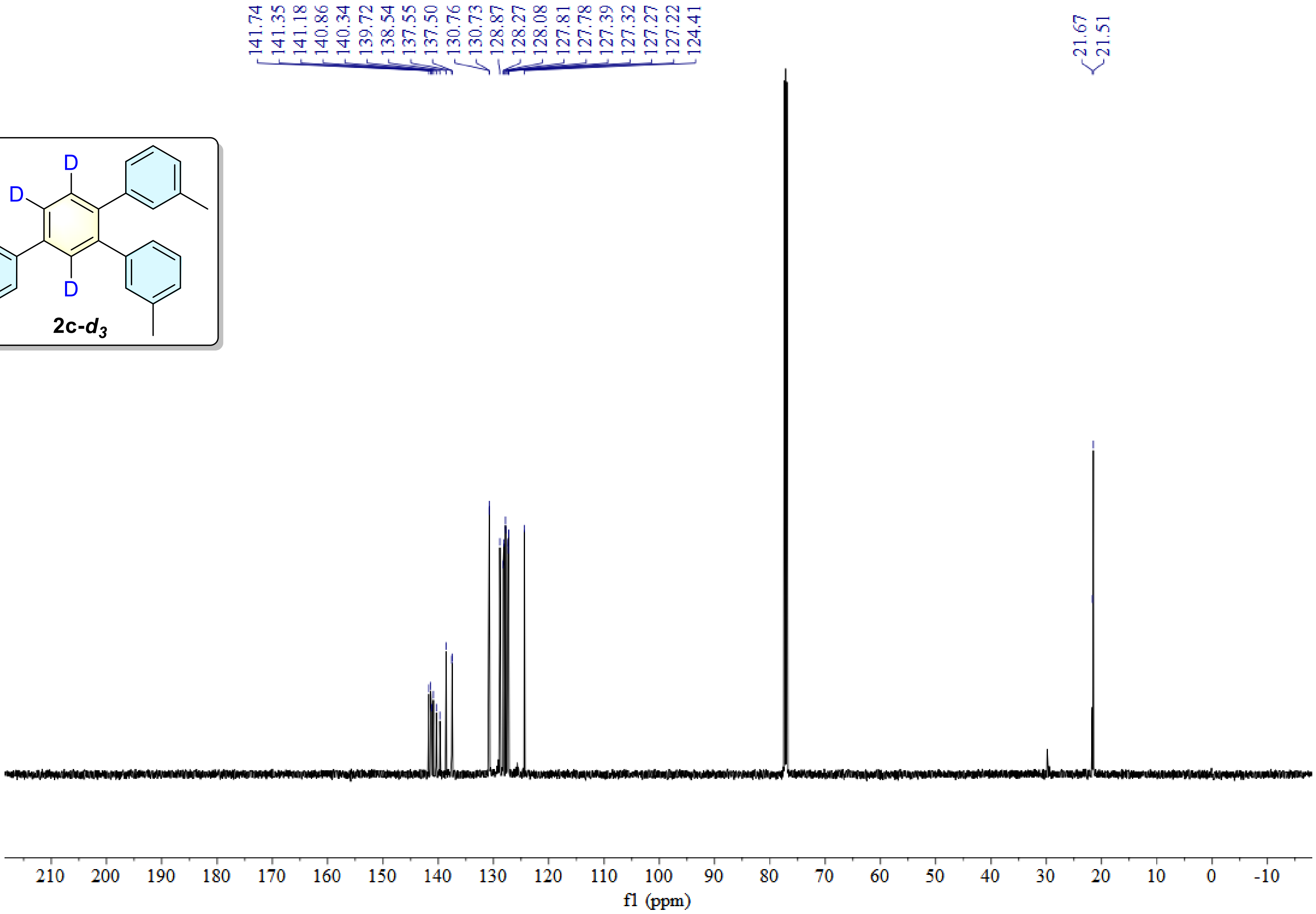

D
D
D

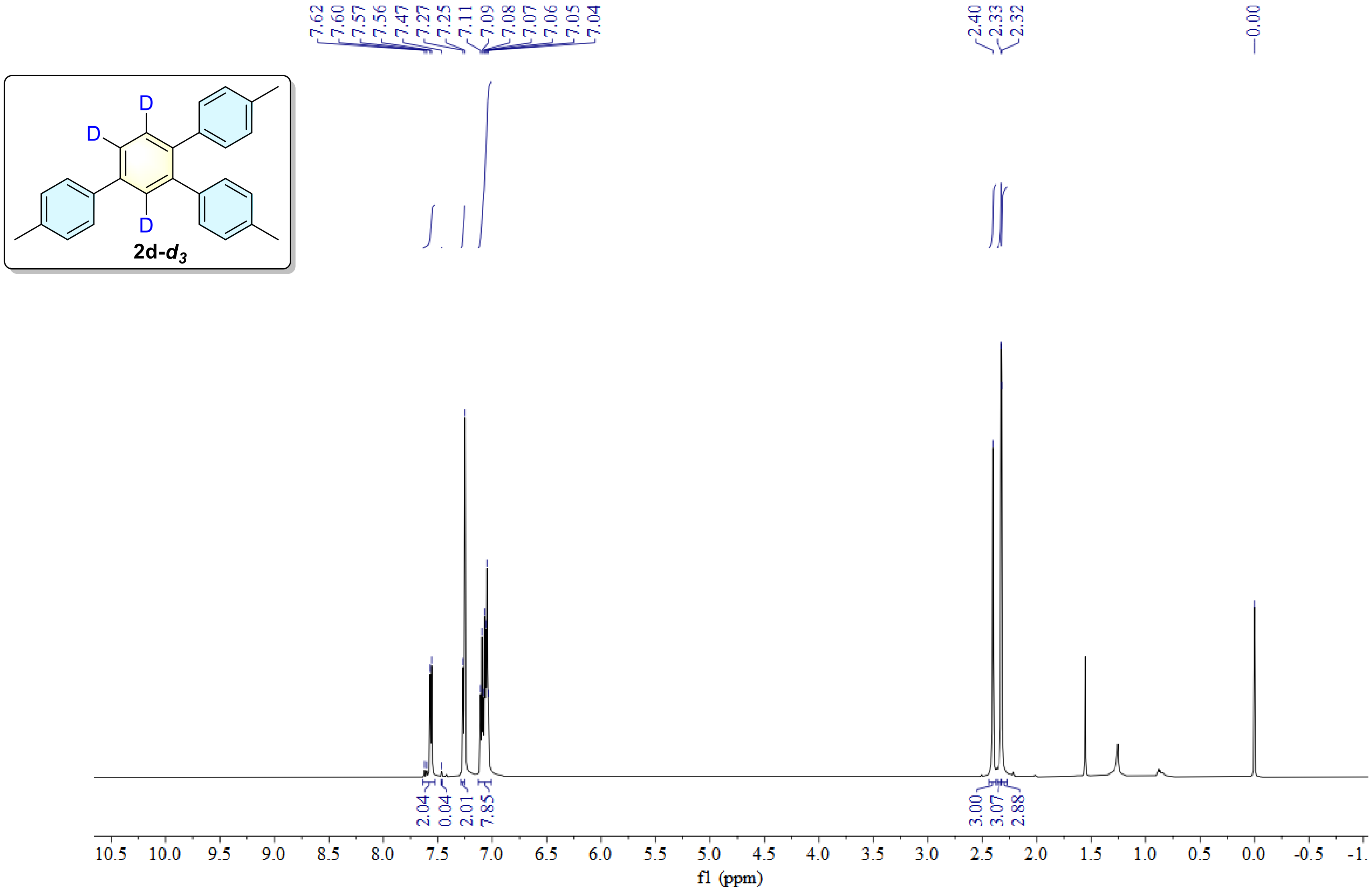
2c-*d*$_3$

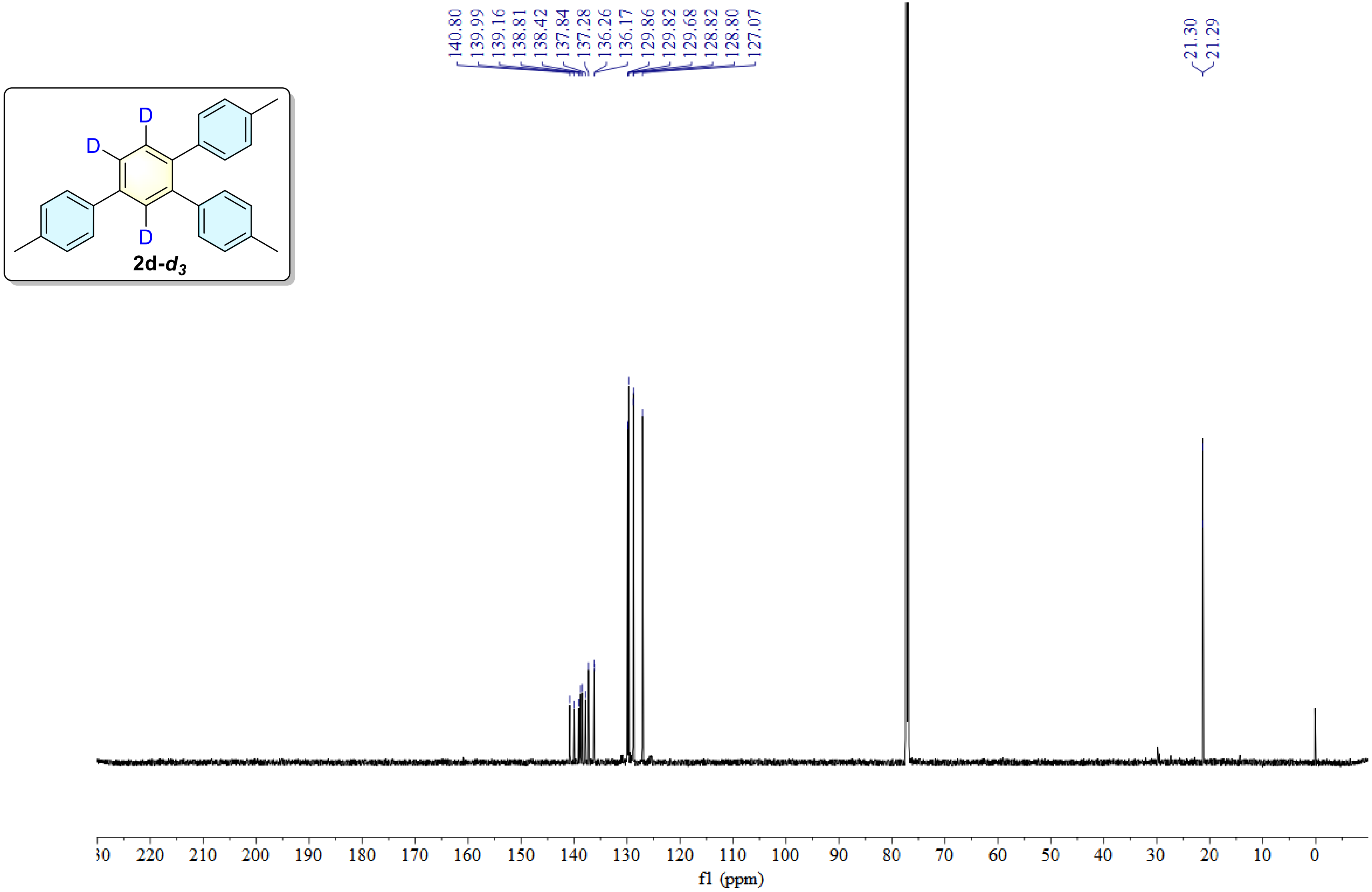
7.65
7.62
7.49
7.47
7.36
7.35
7.34
7.19
7.17
7.10
7.09
7.08
7.07
7.05
7.03
7.02
7.01
6.94
6.93
6.91
2.43
2.28
2.27
0.00
0.05
0.05
2.05
1.05
1.01
6.01
2.00
3.00
3.03
2.93
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


D
D
D

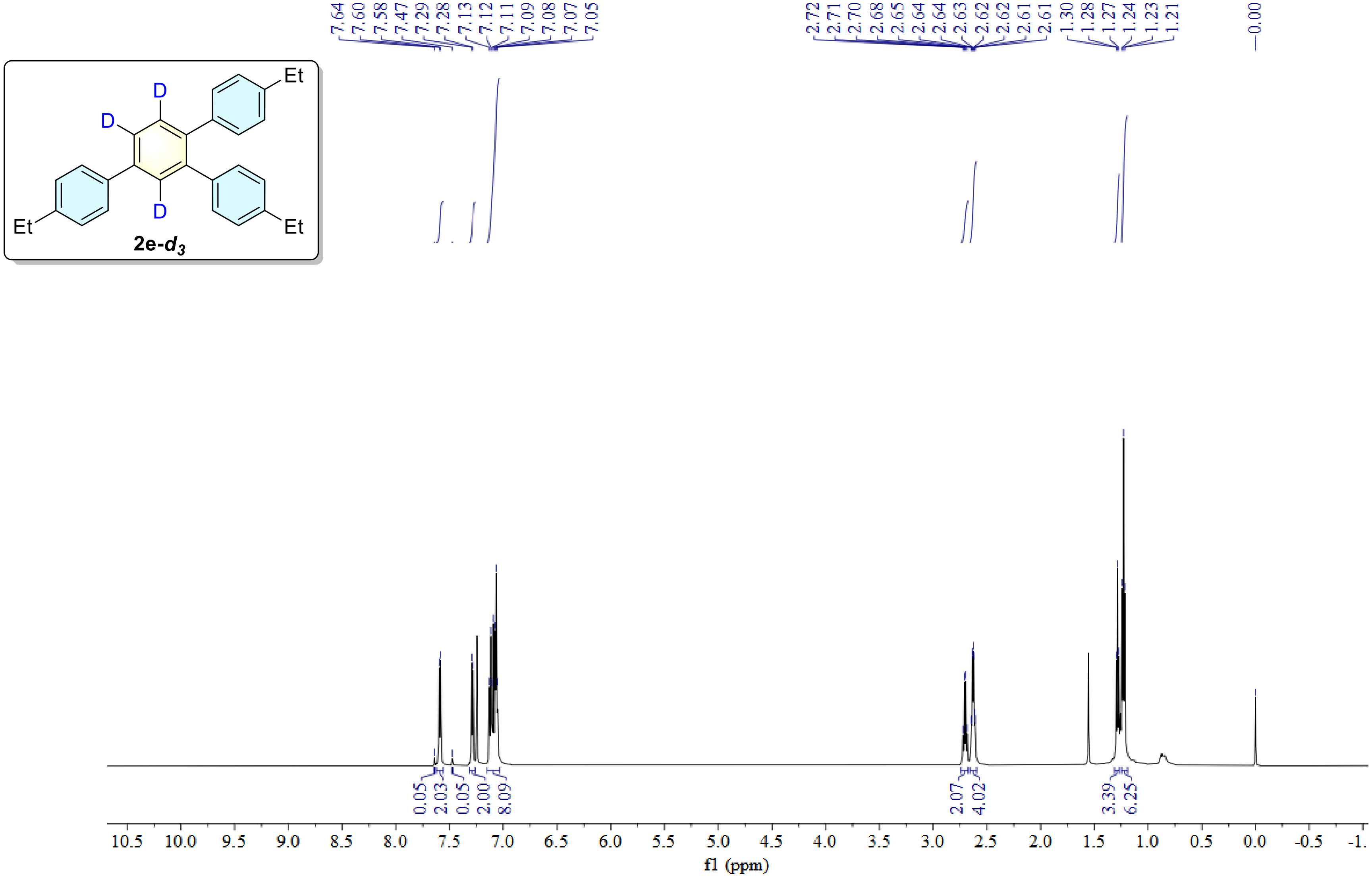
2c-d_3


141.74
141.35
141.18
140.86
140.34
139.72
138.54
137.55
137.50
130.76
130.73
128.87
128.27
128.08
127.81
127.78
127.39
127.32
127.27
127.22
124.41
21.67
21.51
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)

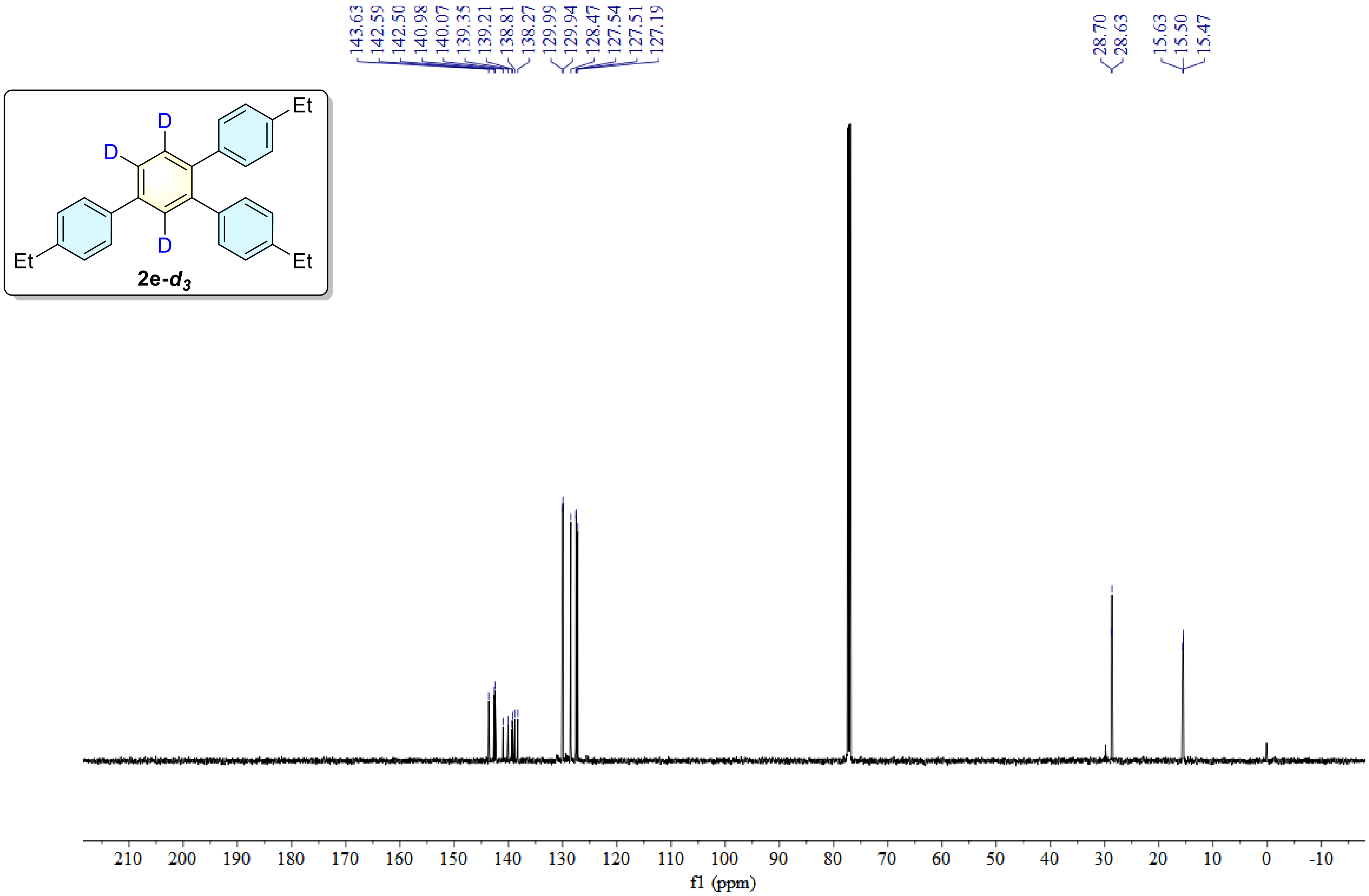

7.62
7.60
7.57
7.56
7.47
7.27
7.25
7.11
7.09
7.08
7.07
7.06
7.05
7.04
2.40
2.33
2.32
-0.00
D
D
D
2d-d_3
2.04
0.04
2.01
7.85
3.00
3.07
2.88
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)



2d-*d*$_3$
140.80
139.99
139.16
138.81
138.42
137.84
137.28
136.26
136.17
129.86
129.82
129.68
128.82
128.80
127.07
21.30
21.29
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

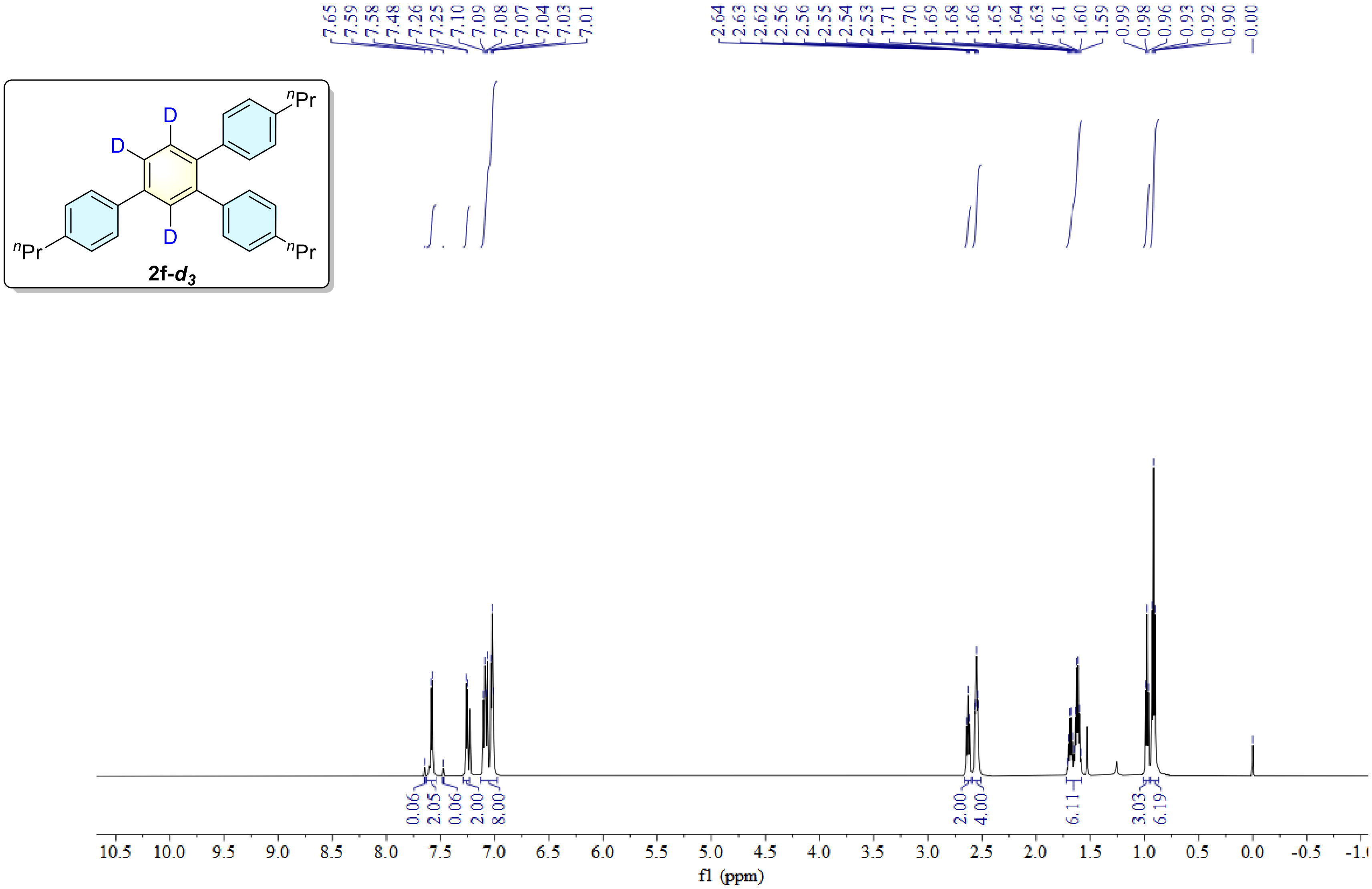

2e-*d*$_3$

D
D
D
Et
Et
Et
7.64
7.60
7.58
7.47
7.29
7.28
7.13
7.12
7.11
7.09
7.08
7.07
7.05
2.72
2.71
2.70
2.68
2.65
2.64
2.64
2.63
2.62
2.62
2.61
2.61
1.30
1.28
1.27
1.24
1.23
1.21
0.00
0.05
2.03
0.05
2.00
8.09
2.07
4.02
3.39
6.25
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
-0.5
-1.
f1 (ppm)

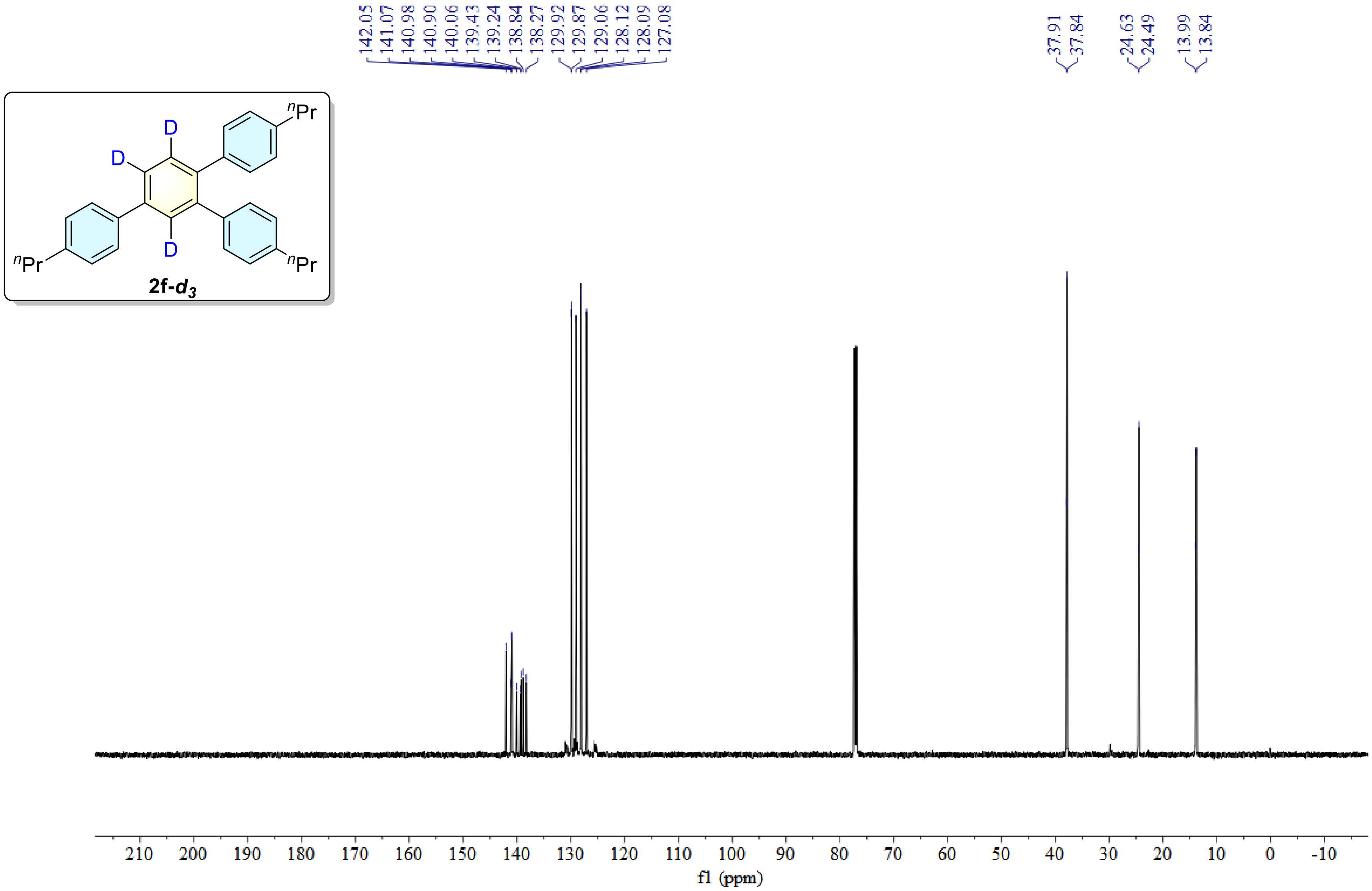

D
D
D
Et
Et
Et

2e-d_3

143.63
142.59
142.50
140.98
140.07
139.35
139.21
138.81
138.27
129.99
129.94
128.47
127.54
127.51
127.19
28.70
28.63
15.63
15.50
15.47
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)

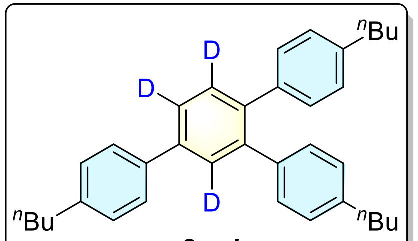


7.65
7.59
7.58
7.48
7.26
7.25
7.10
7.09
7.08
7.07
7.04
7.03
7.01
2.64
2.63
2.62
2.56
2.56
2.55
2.54
2.53
1.71
1.70
1.69
1.68
1.66
1.65
1.64
1.63
1.61
1.60
1.59
0.99
0.98
0.96
0.93
0.92
0.90
0.00
D
D
D
^{n}Pr
^{n}Pr
^{n}Pr
2f-d_3
0.06
2.05
0.06
2.00
8.00
2.00
4.00
6.11
3.03
6.19
10.5
10.0
9.5
9.0
8.5
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
-0.5
-1.0
f1 (ppm)

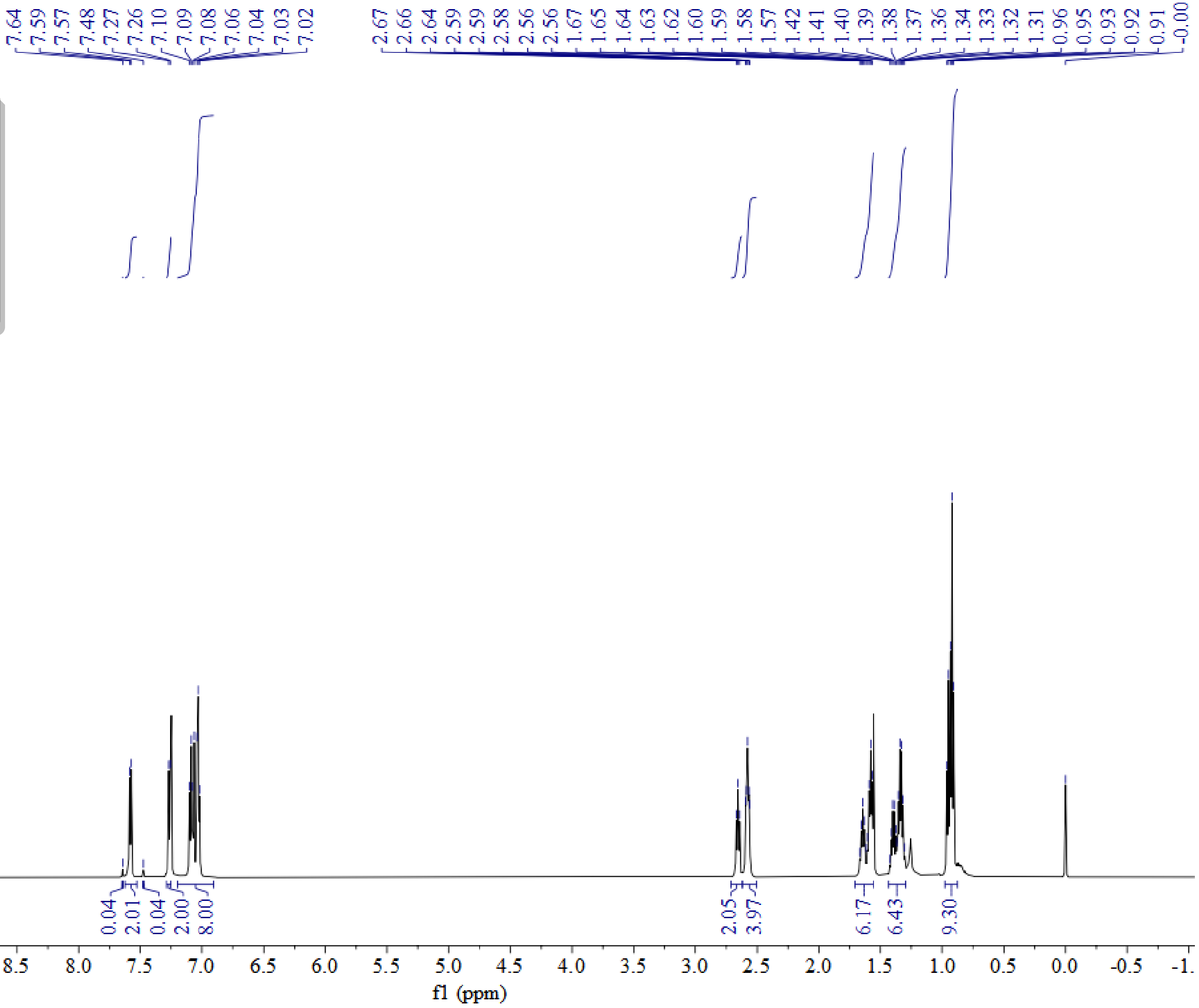


2f-*d*$_3$

142.05
141.07
140.98
140.90
140.06
139.43
139.24
138.84
138.27
129.92
129.87
129.06
128.12
128.09
127.08
37.91
37.84
24.63
24.49
13.99
13.84
D
D
D
nPr
nPr
nPr
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)


D
D
D
^{n}Bu
^{n}Bu
^{n}Bu

2g-d_3


7.64
7.59
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7.27
7.26
7.10
7.09
7.08
7.06
7.04
7.03
7.02
2.67
2.66
2.64
2.59
2.59
2.58
2.56
2.56
1.67
1.65
1.64
1.63
1.62
1.60
1.59
1.58
1.57
1.42
1.41
1.40
1.39
1.38
1.37
1.36
1.34
1.33
1.32
1.31
0.96
0.95
0.93
0.92
0.91
-0.00
0.04
2.01
0.04
2.00
8.00
2.05
3.97
6.17
6.43
9.30
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
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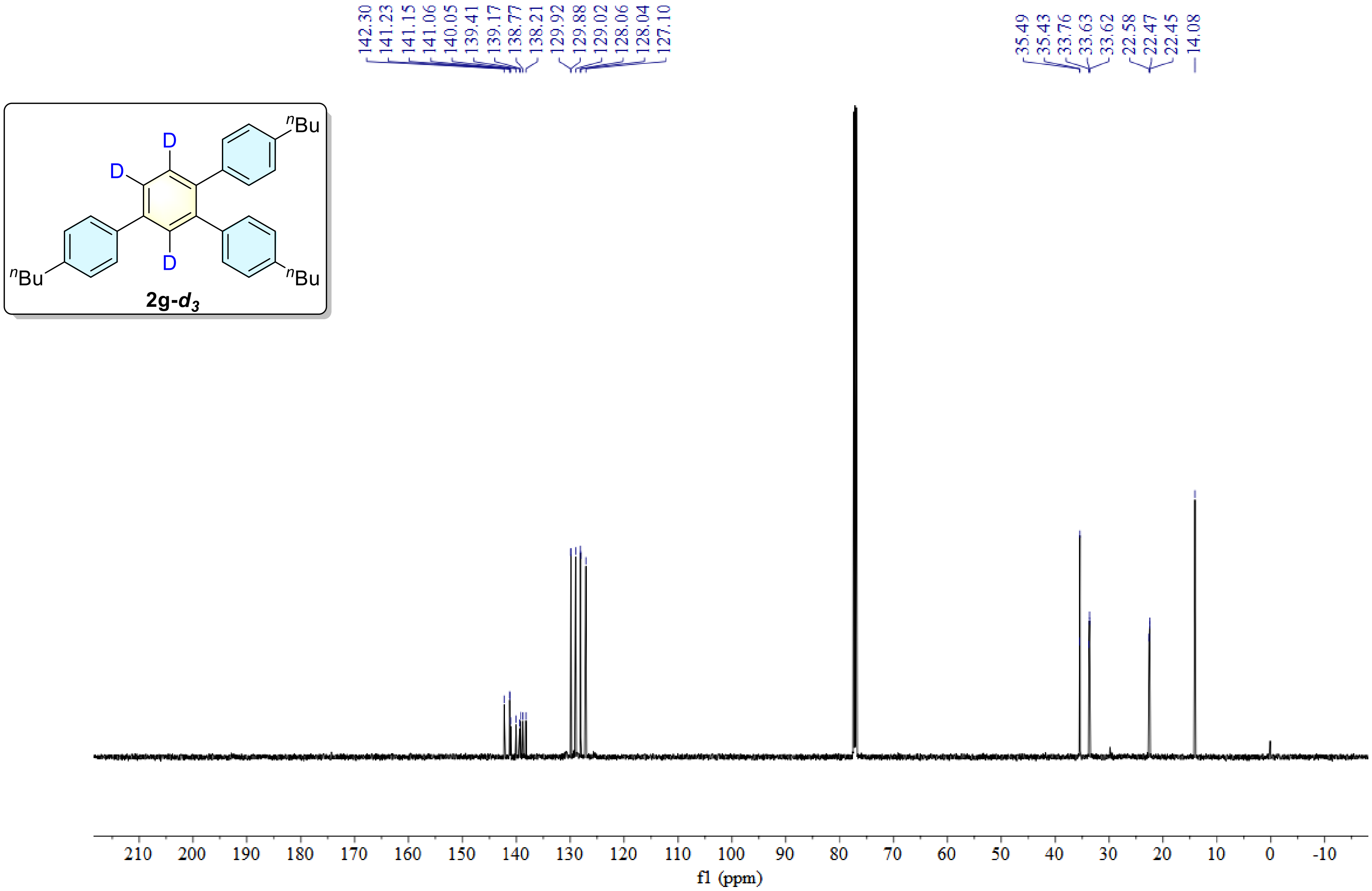
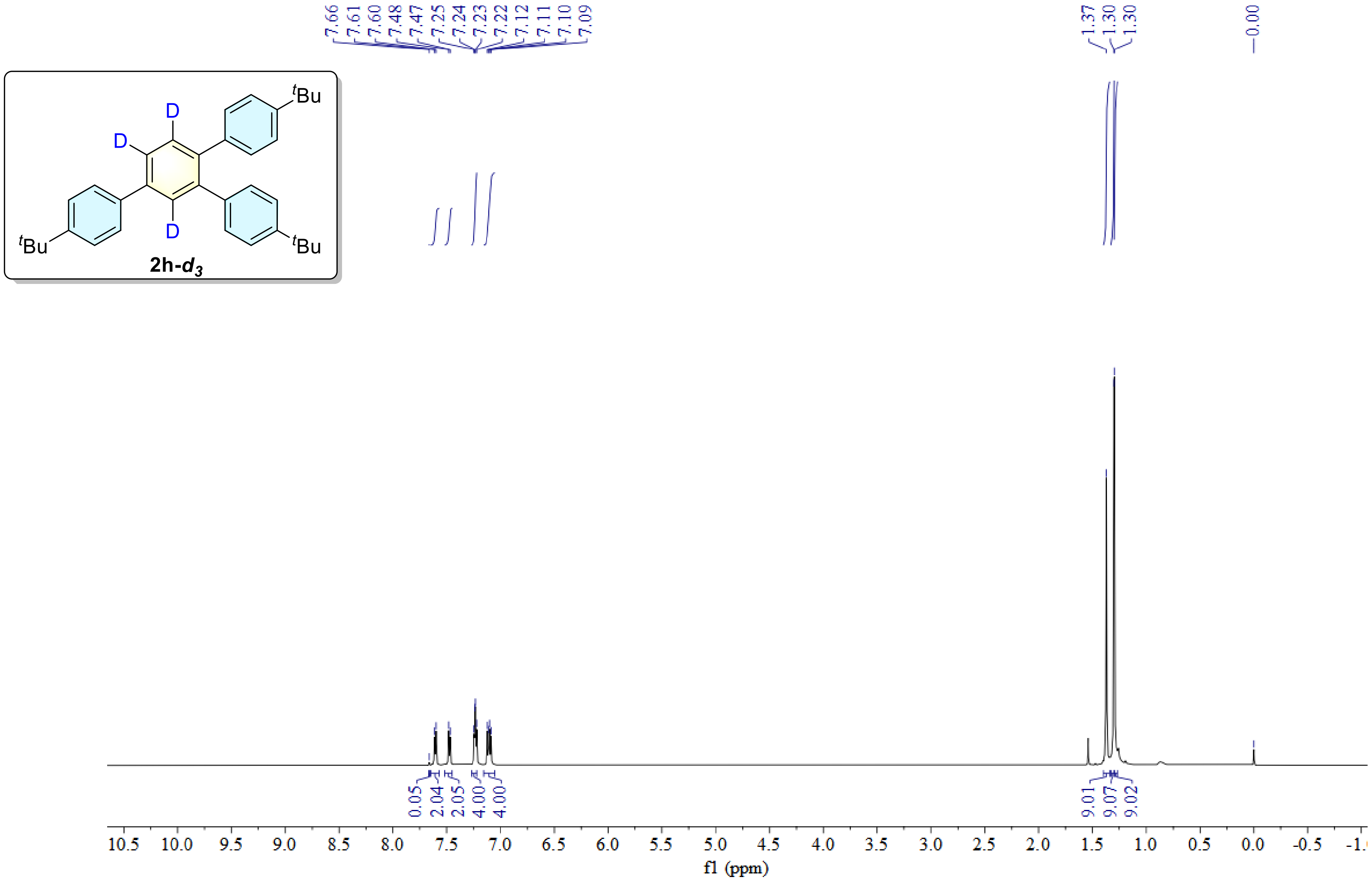
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)


142.30
141.23
141.15
141.06
140.05
139.41
139.17
138.77
138.21
129.92
129.88
129.02
128.06
128.04
127.10
35.49
35.43
33.76
33.63
33.62
22.58
22.47
22.45
14.08
D
D
D
^{n}Bu
^{n}Bu
^{n}Bu
2g-d_3
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)


7.66
7.61
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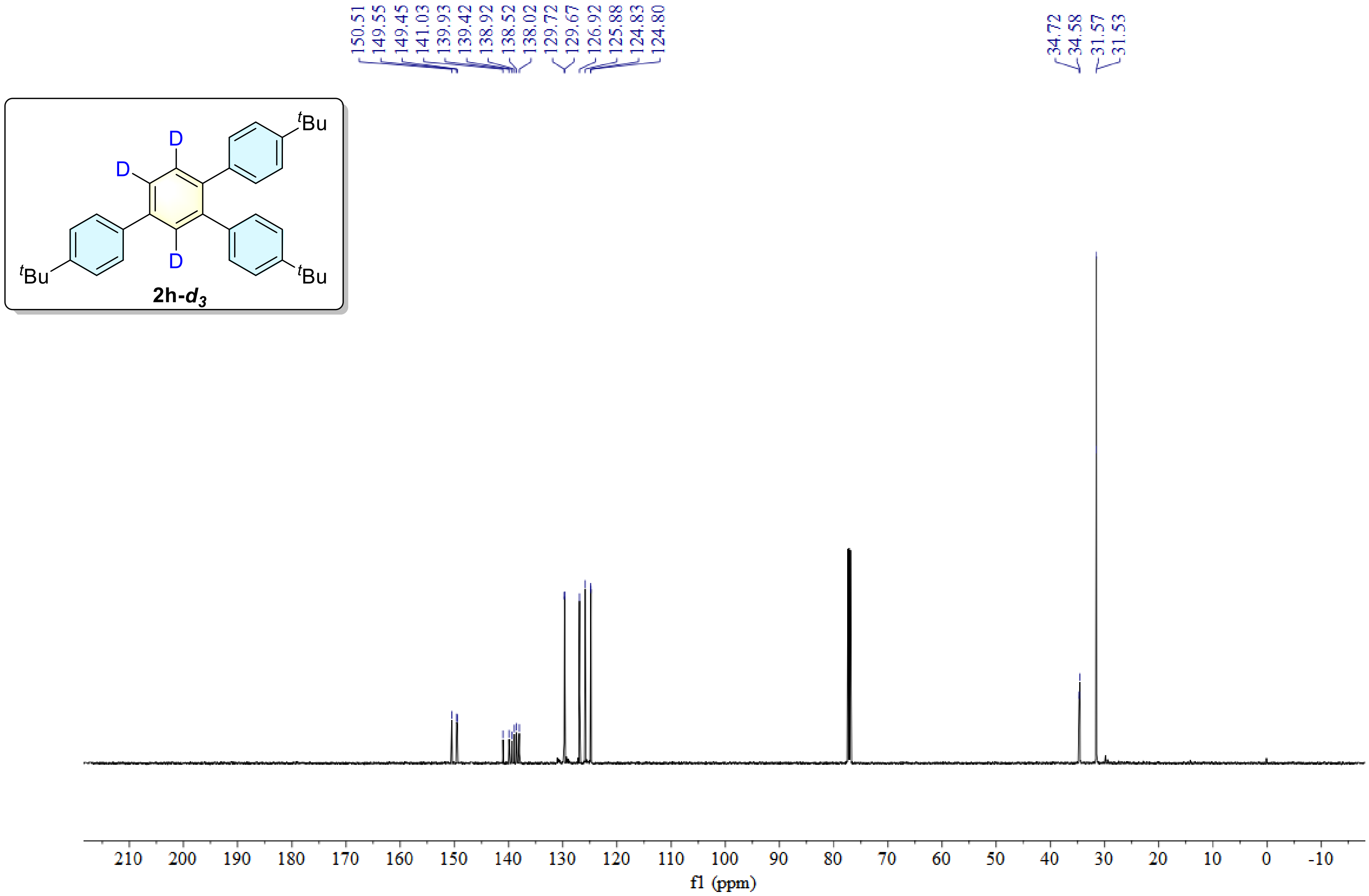
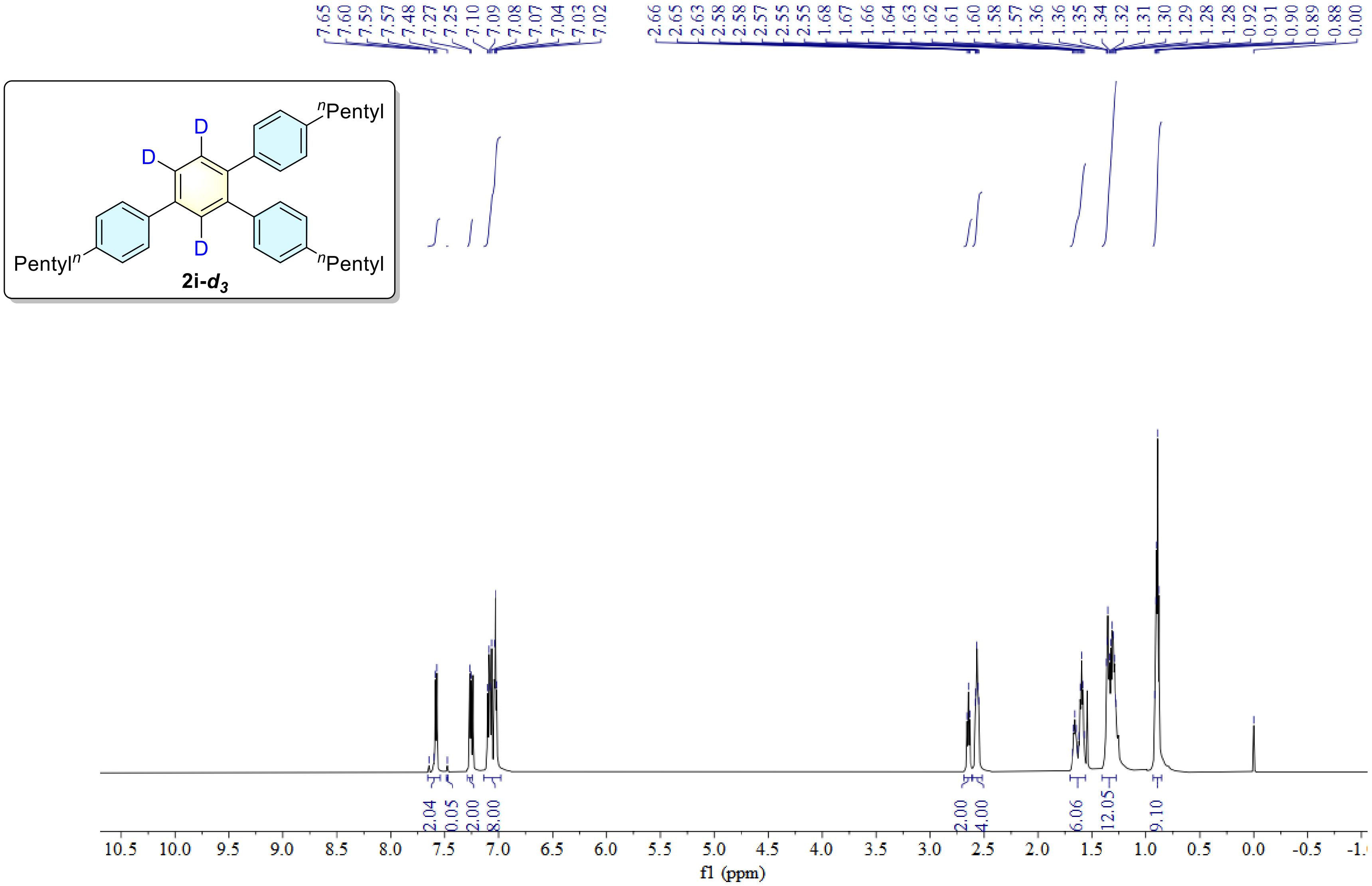
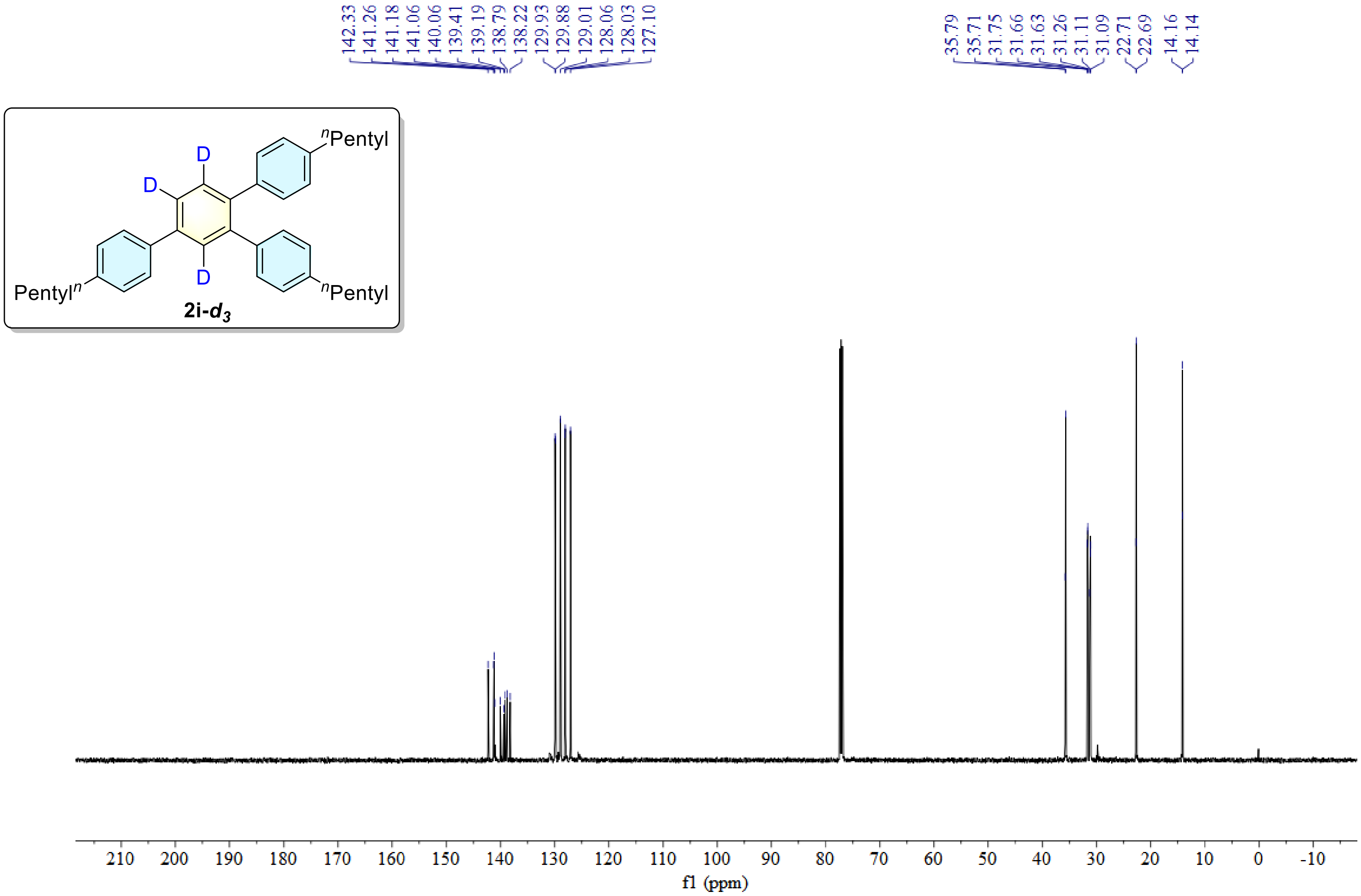
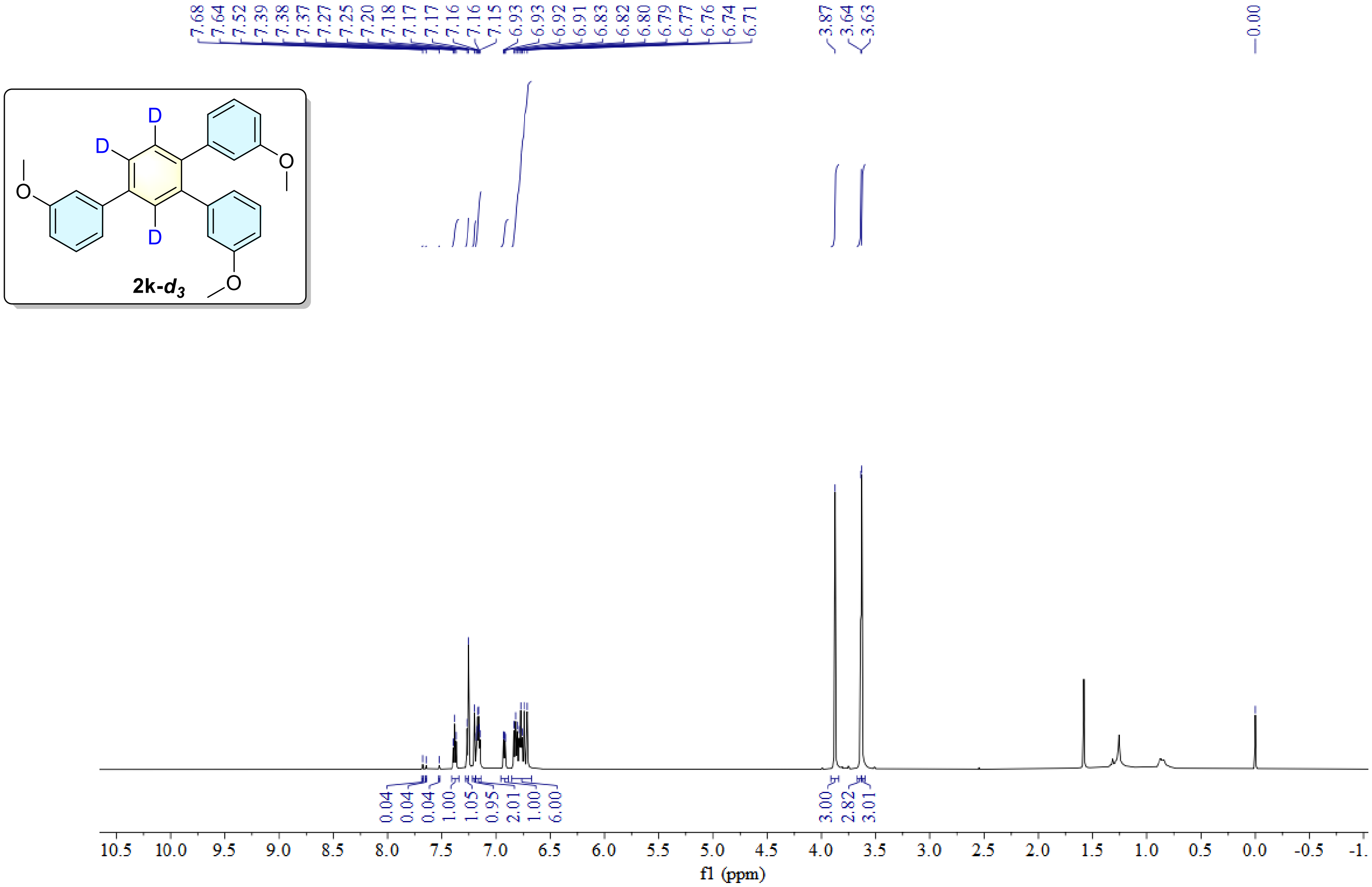
7.48
7.47
7.25
7.24
7.23
7.22
7.12
7.11
7.10
7.09
1.37
1.30
1.30
0.00
D
D
D
tBu
tBu
tBu
2h-d3
0.05
2.04
2.05
4.00
4.00
9.01
9.07
9.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


150.51
149.55
149.45
141.03
139.93
139.42
138.92
138.52
138.02
129.72
129.67
126.92
125.88
124.83
124.80
34.72
34.58
31.57
31.53
D
D
D
^{t}Bu
^{t}Bu
^{t}Bu
2h-d_3
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)


7.65
7.60
7.59
7.57
7.48
7.27
7.25
7.10
7.09
7.08
7.07
7.04
7.03
7.02
2.66
2.65
2.63
2.58
2.58
2.57
2.55
2.55
1.68
1.67
1.66
1.64
1.63
1.62
1.61
1.60
1.58
1.57
1.36
1.36
1.35
1.34
1.32
1.31
1.30
1.29
1.28
1.28
0.92
0.91
0.90
0.89
0.88
0.00
D
D
D
nPentyl
nPentyl
Pentyln
2.04
0.05
2.00
8.00
2.00
4.00
6.06
12.05
9.10
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
-0.5
-1.
f1 (ppm)

2i-*d*$_3$


142.33
141.26
141.18
141.06
140.06
139.41
139.19
138.79
138.22
129.93
129.88
129.01
128.06
128.03
127.10
35.79
35.71
31.75
31.66
31.63
31.26
31.11
31.09
22.71
22.69
14.16
14.14
D
D
D
ⁿPentyl
Pentylⁿ
ⁿPentyl
2i-d_3
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)


7.68
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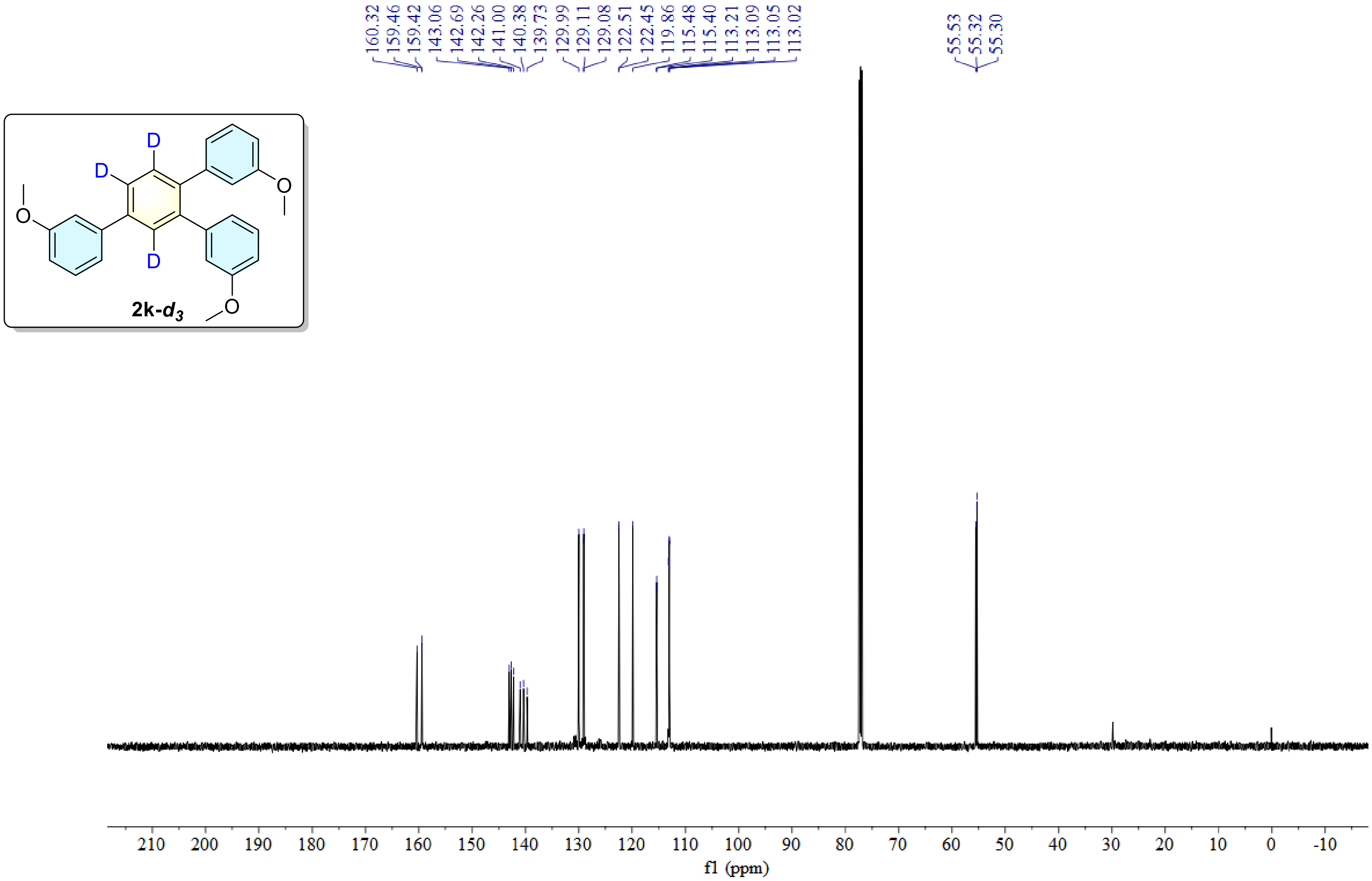
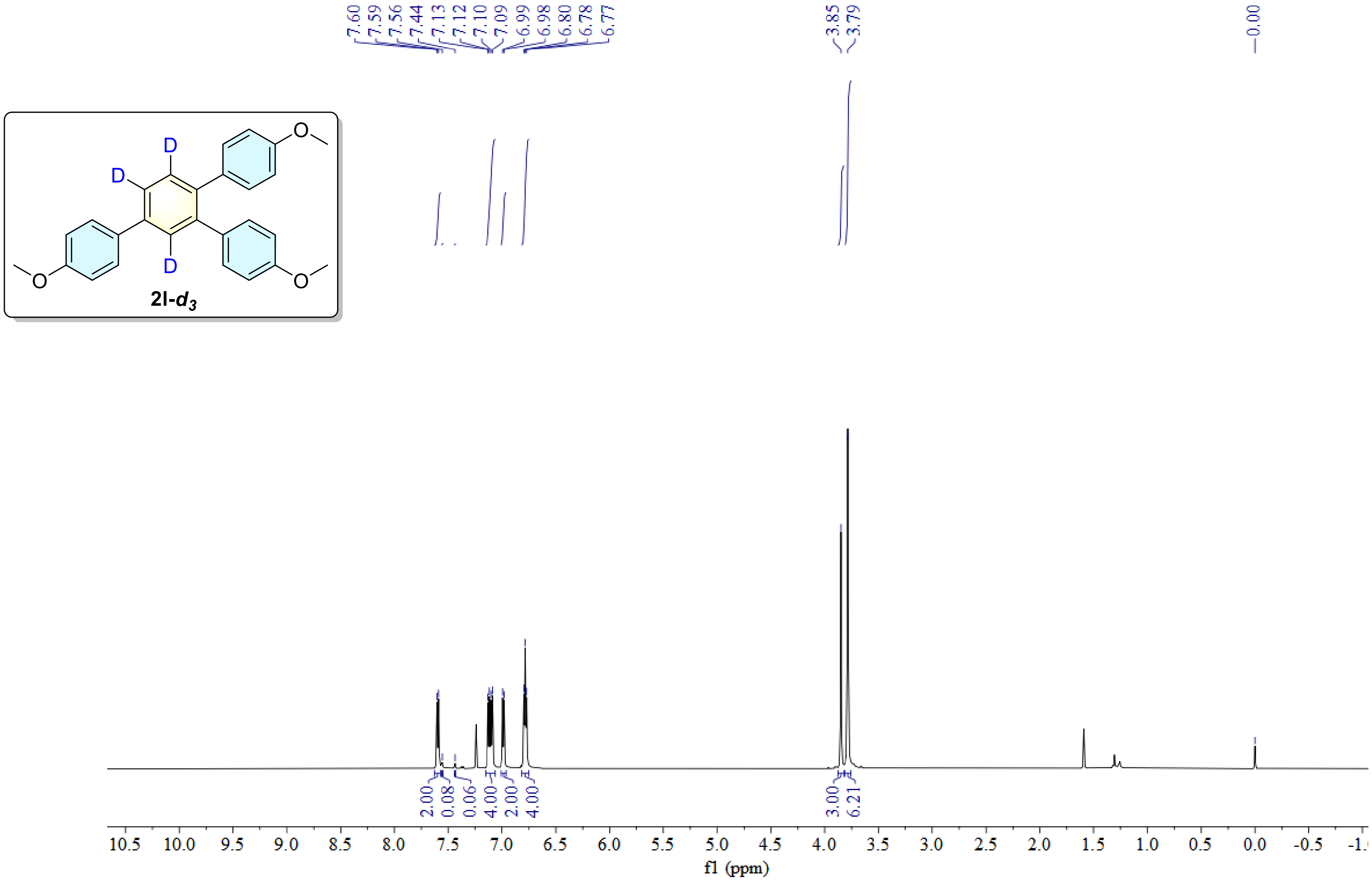
7.52
7.39
7.38
7.37
7.27
7.25
7.20
7.18
7.17
7.17
7.16
7.16
7.15
6.93
6.93
6.92
6.91
6.83
6.82
6.80
6.79
6.77
6.76
6.74
6.71
3.87
3.64
3.63
0.00
D
D
D
O
O
O
0.04
0.04
0.04
1.00
1.05
0.95
2.01
1.00
6.00
3.00
2.82
3.01
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)

2k-d_3


D
D
O
O
D
O

2k-d_3
160.32
159.46
159.42
143.06
142.69
142.26
141.00
140.38
139.73
129.99
129.11
129.08
122.51
122.45
119.86
115.48
115.40
113.21
113.09
113.05
113.02
55.53
55.32
55.30
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)

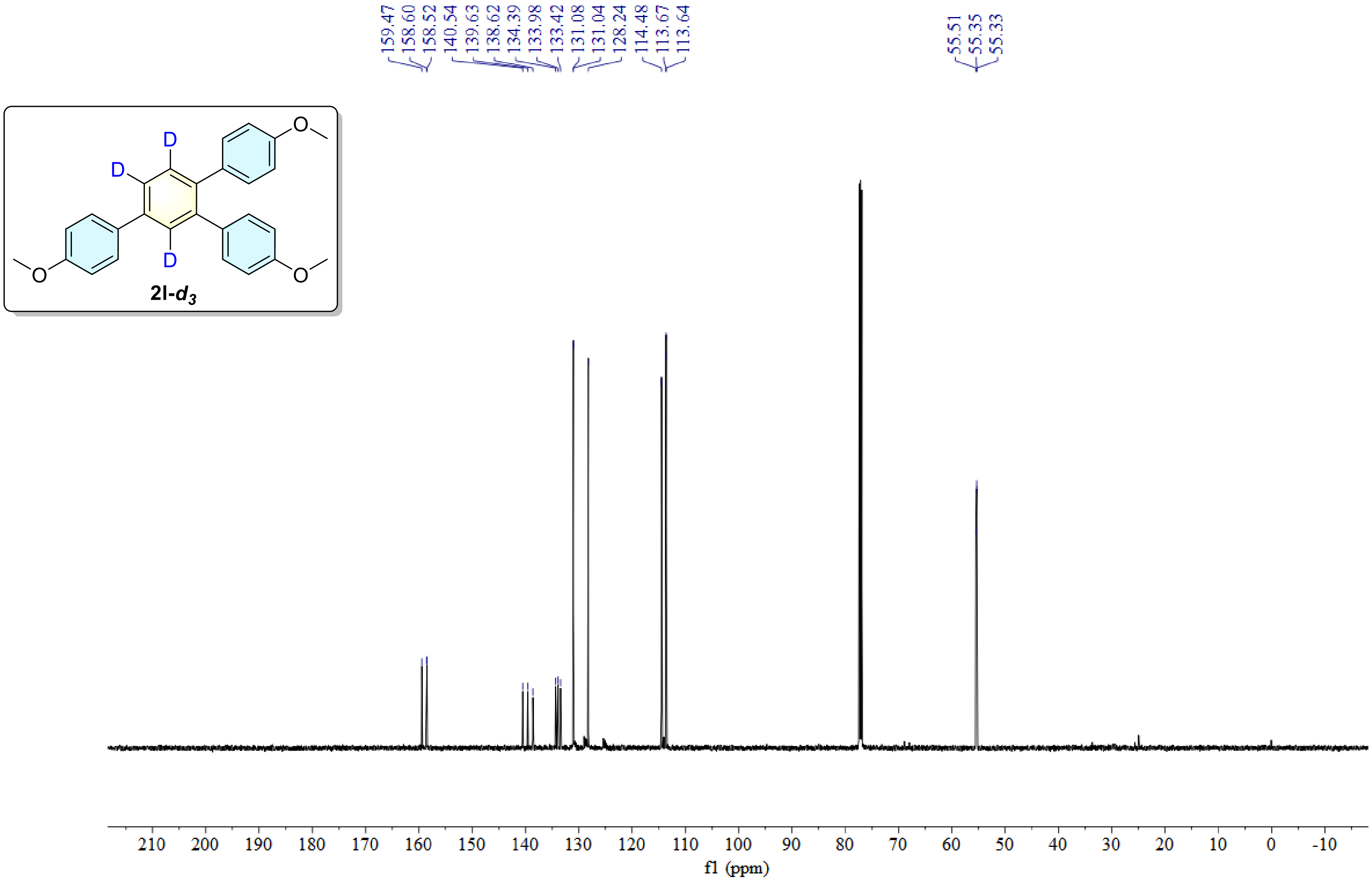

D
D
D
O
O
O

2l-d_3

7.60
7.59
7.56
7.44
7.13
7.12
7.10
7.09
6.99
6.98
6.80
6.78
6.77
3.85
3.79
0.00
2.00
0.08
0.06
4.00
2.00
4.00
3.00
6.21
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
-0.5
f1 (ppm)

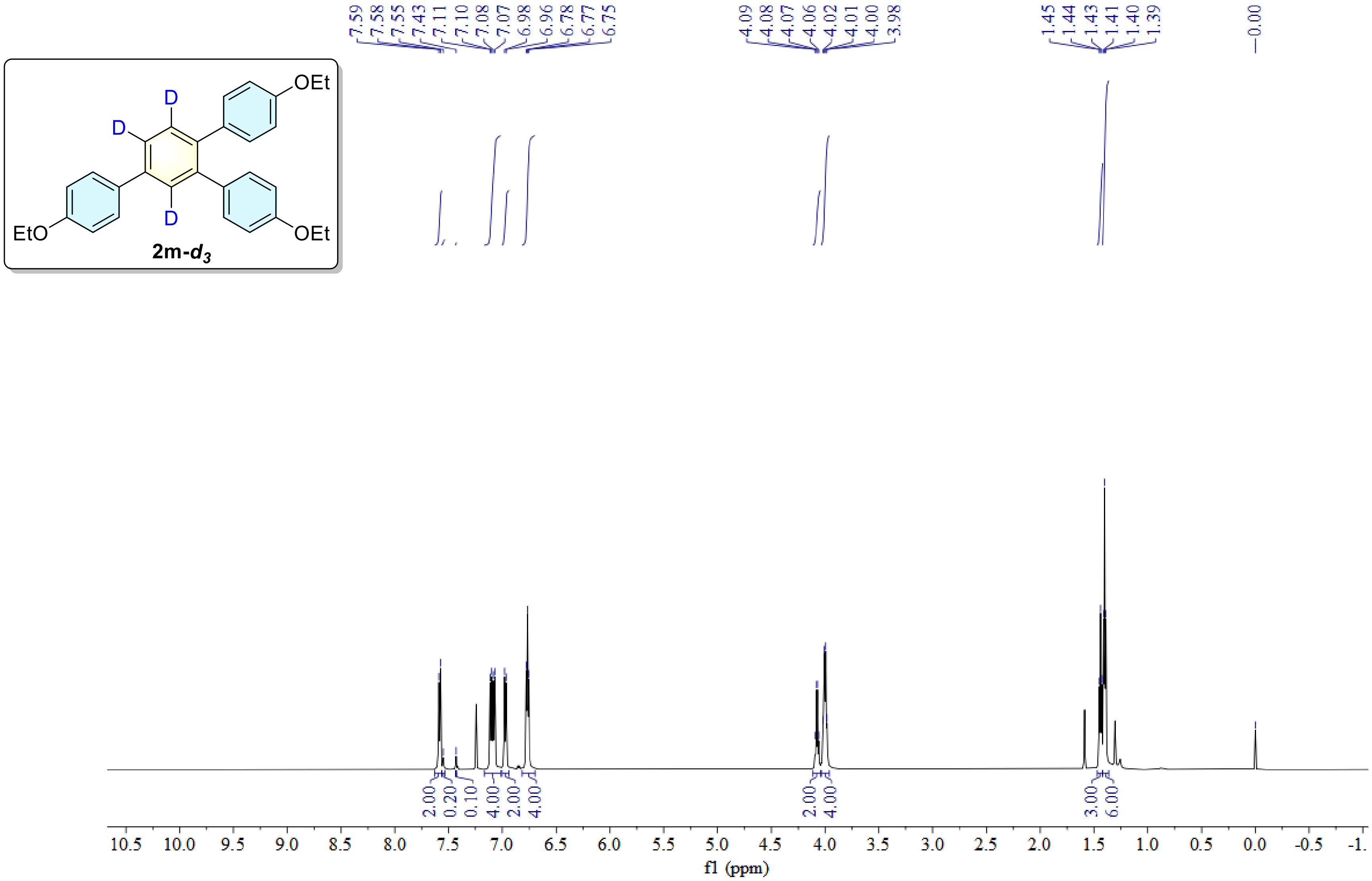
D
D
D
O
O
O

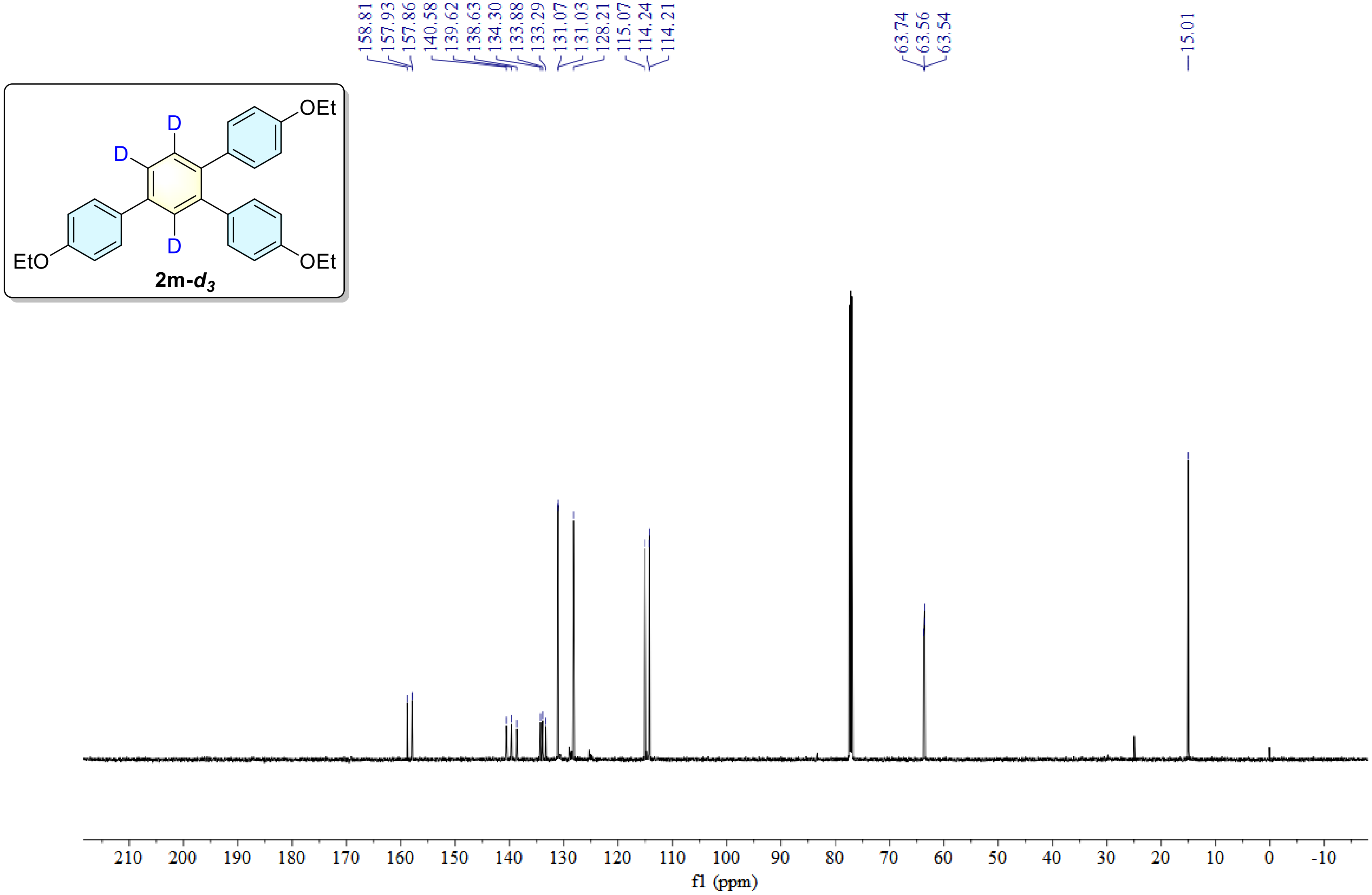
2l-d_3

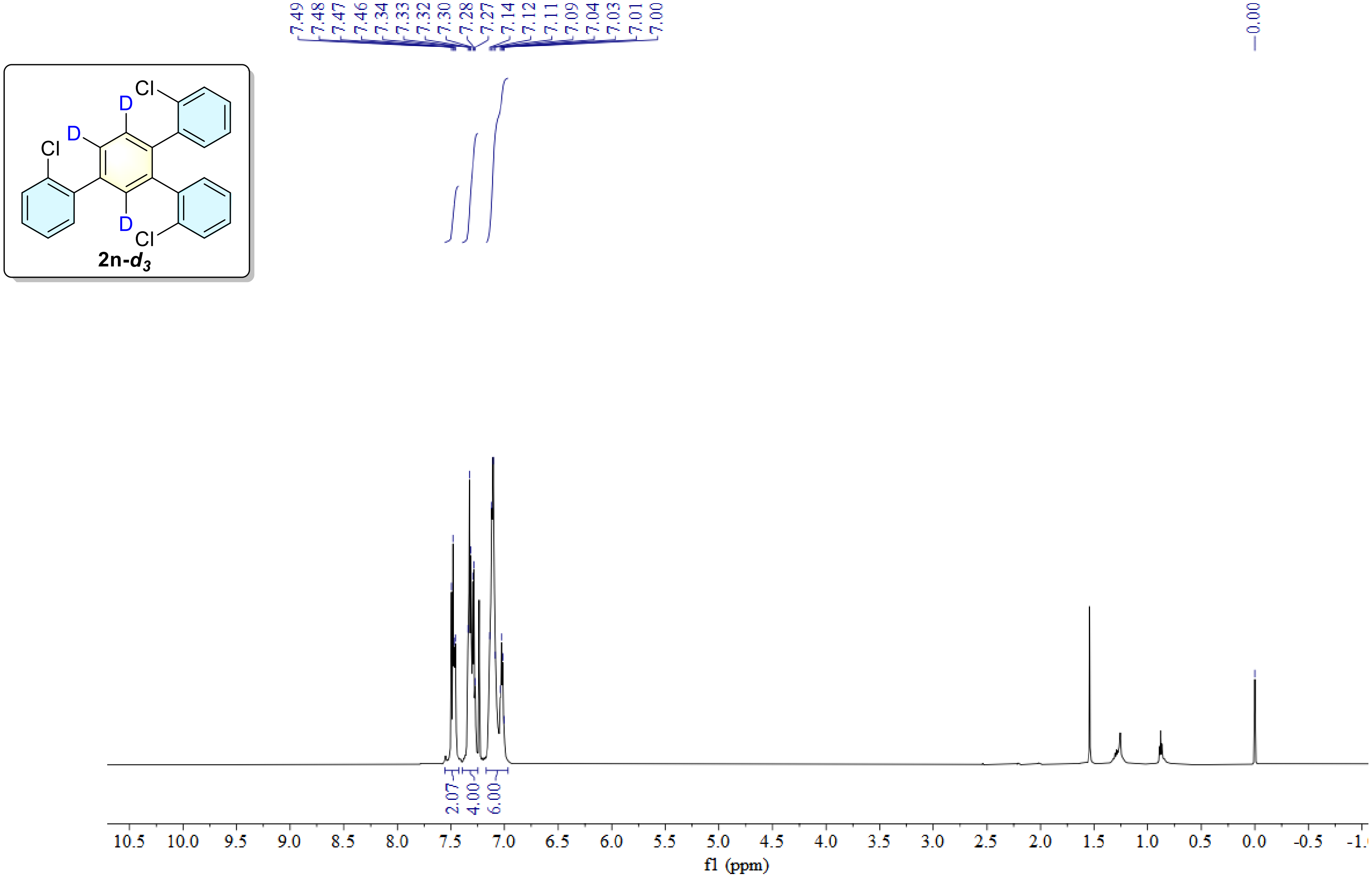

159.47
158.60
158.52
140.54
139.63
138.62
134.39
133.98
133.42
131.08
131.04
128.24
114.48
113.67
113.64
55.51
55.35
55.33
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

D
D
D
OEt
OEt
EtO

2m-d_3


7.59
7.58
7.55
7.43
7.11
7.10
7.08
7.07
6.98
6.96
6.78
6.77
6.75
4.09
4.08
4.07
4.06
4.02
4.01
4.00
3.98
1.45
1.44
1.43
1.41
1.40
1.39
0.00
2.00
0.20
0.10
4.00
2.00
4.00
2.00
4.00
3.00
6.00
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
-0.5
-1.
f1 (ppm)

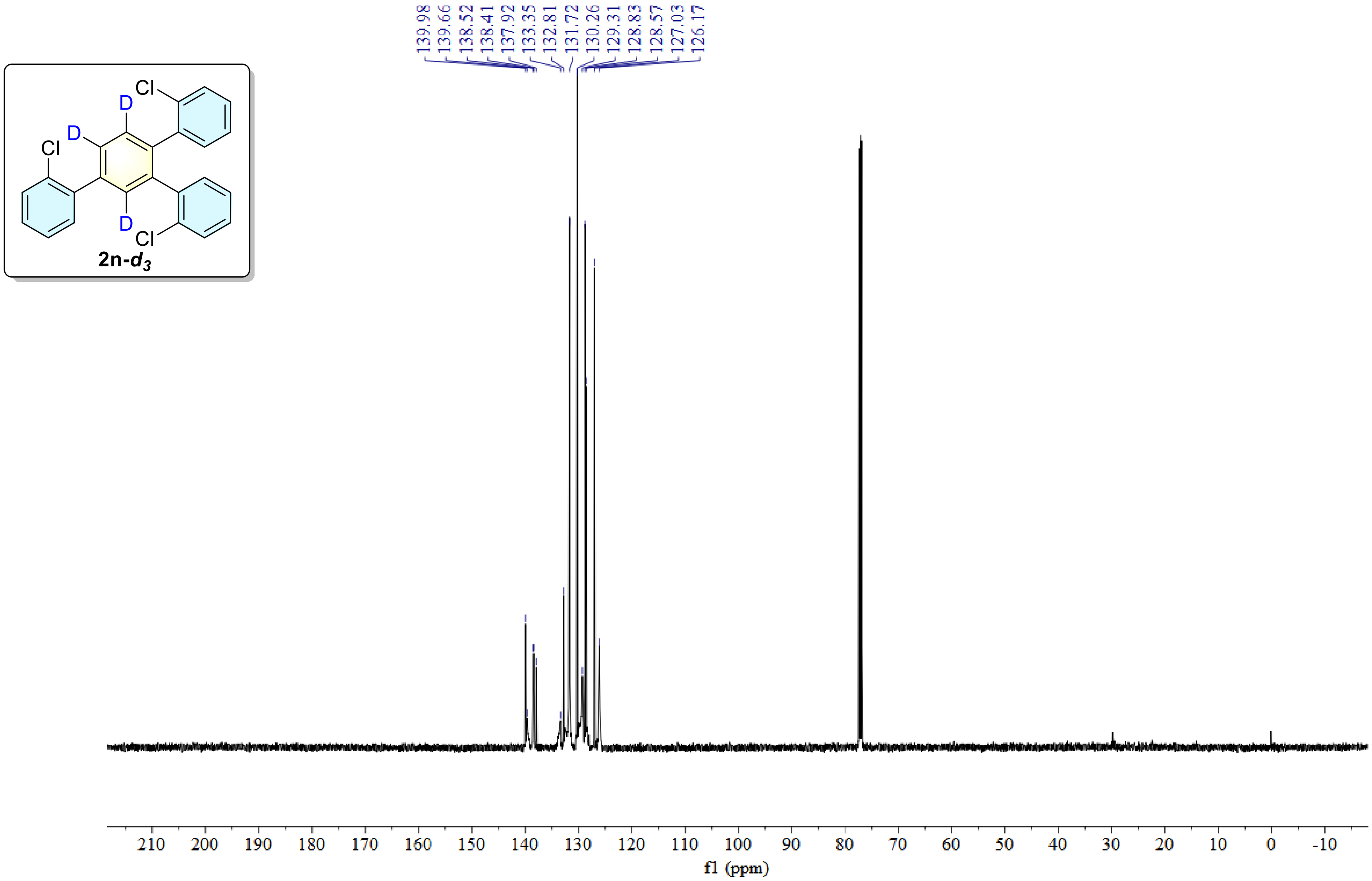

158.81
157.93
157.86
140.58
139.62
138.63
134.30
133.88
133.29
131.07
131.03
128.21
115.07
114.24
114.21
63.74
63.56
63.54
15.01
D
D
D
OEt
OEt
EtO
2m-d_3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



Cl
D
D
Cl
D
Cl

2n-d_3

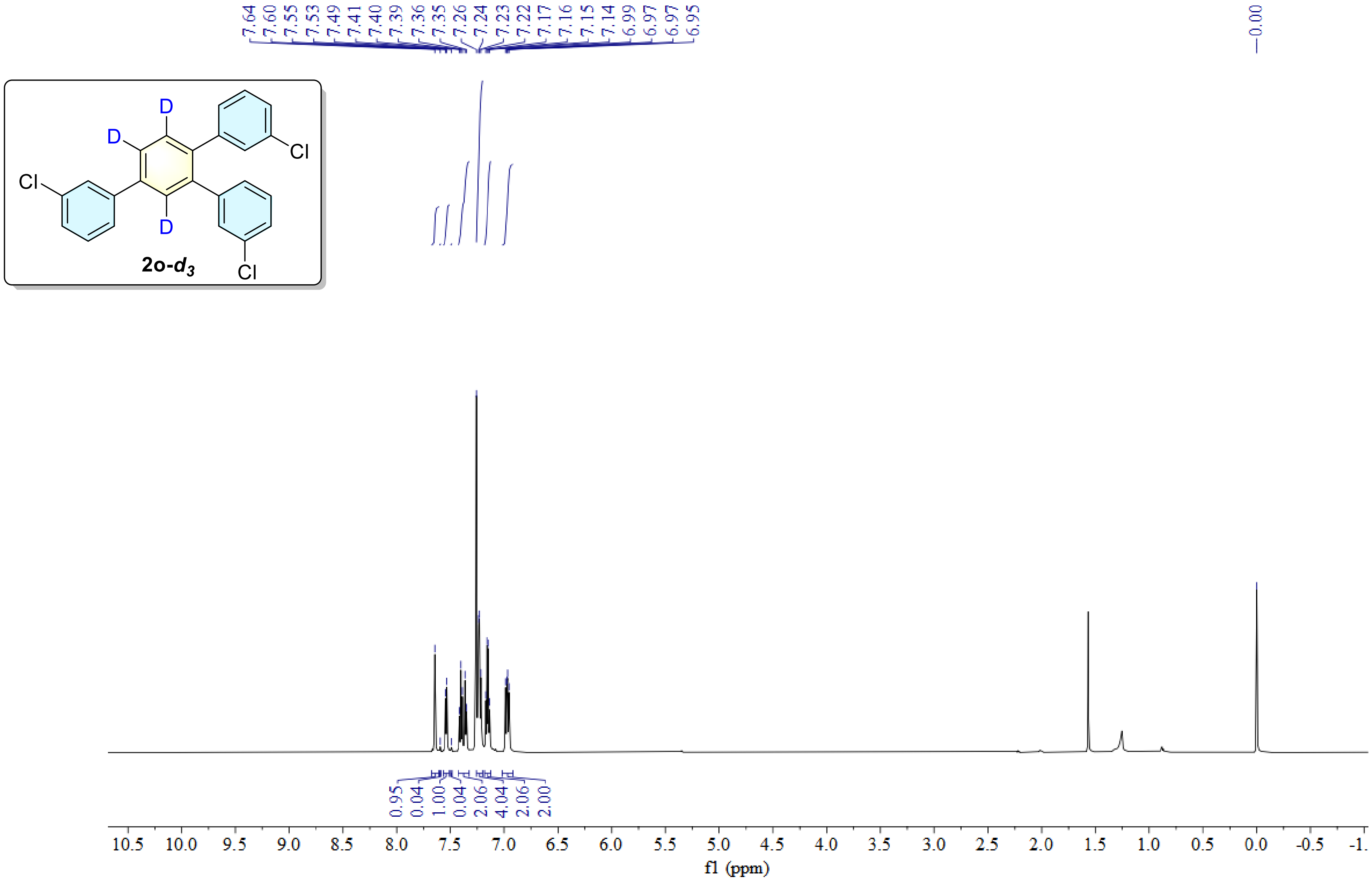
7.49
7.48
7.47
7.46
7.34
7.33
7.32
7.30
7.28
7.27
7.14
7.12
7.11
7.09
7.04
7.03
7.01
7.00
0.00
2.07
4.00
6.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)

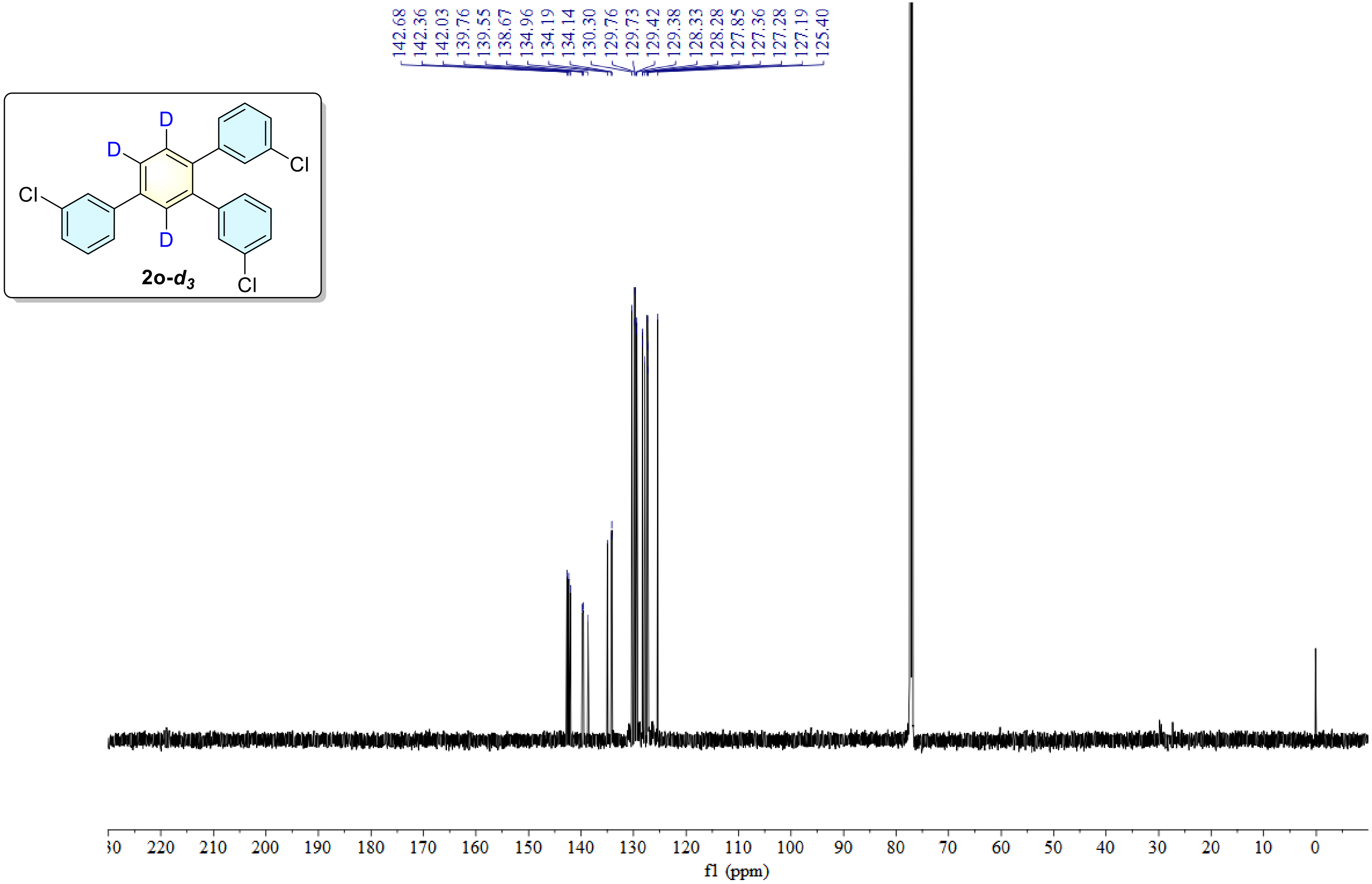


Cl
D
D
Cl
D
Cl

2n-d_3

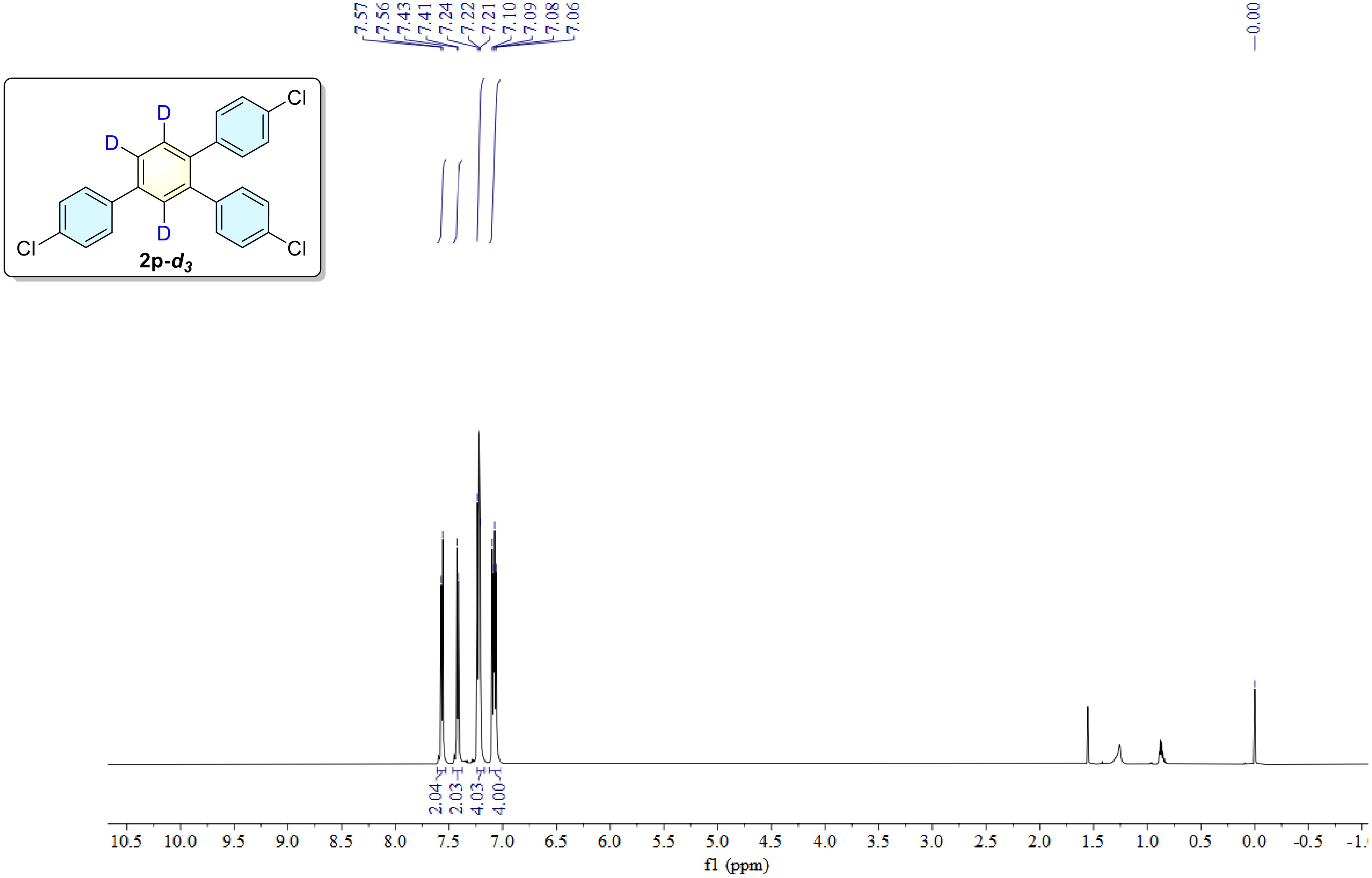
139.98
139.66
138.52
138.41
137.92
133.35
132.81
131.72
130.26
129.31
128.83
128.57
127.03
126.17
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.64
7.60
7.55
7.53
7.49
7.41
7.40
7.39
7.36
7.35
7.26
7.24
7.23
7.22
7.17
7.16
7.15
7.14
6.99
6.97
6.97
6.95
0.00
D
D
D
Cl
Cl
Cl
2o-*d*$_3$
0.95
0.04
1.00
0.04
2.06
4.04
2.06
2.00
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
-0.5
-1.
f1 (ppm)


D
D
D
Cl
Cl
Cl

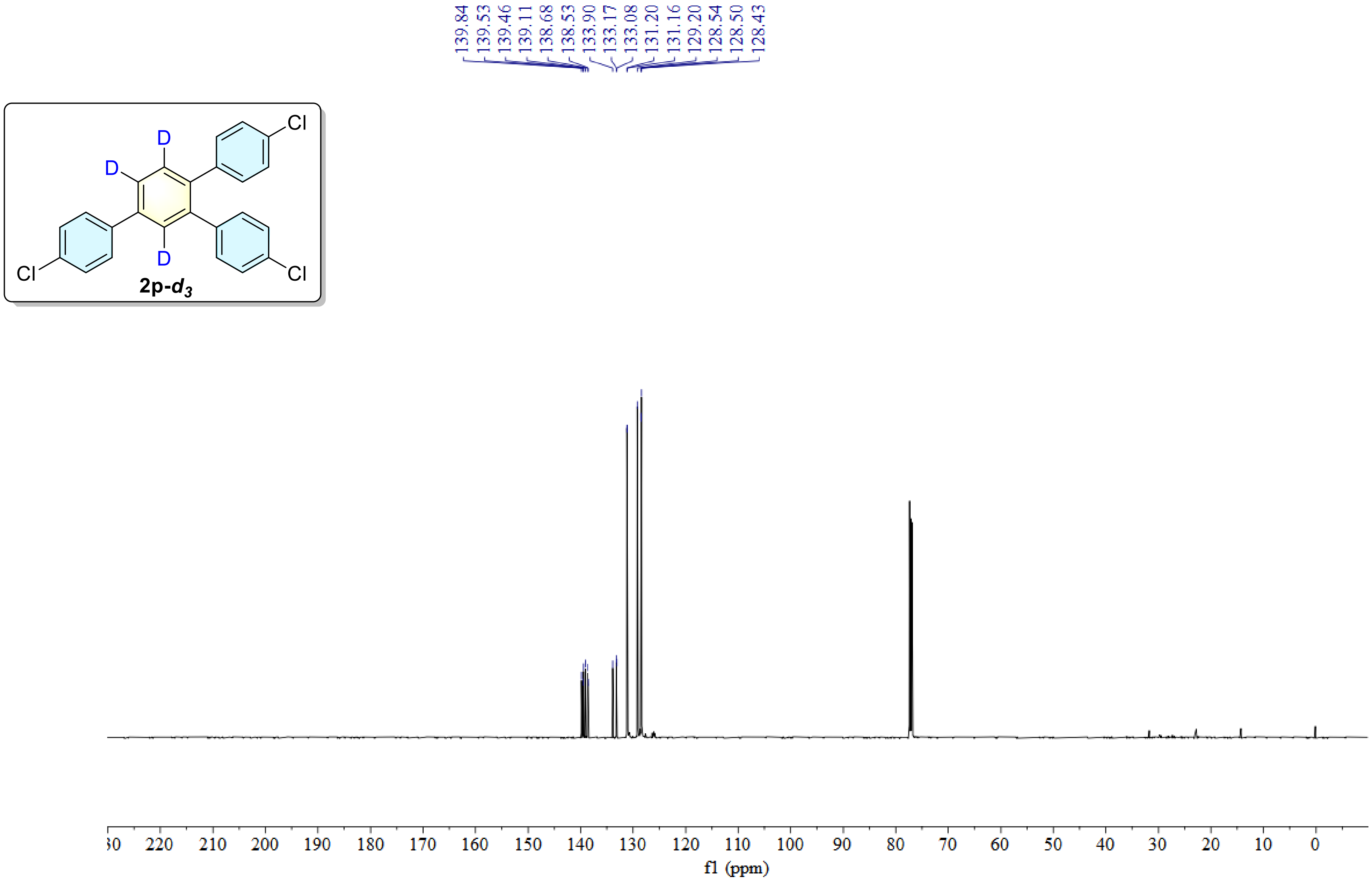
2o-d_3

142.68
142.36
142.03
139.76
139.55
138.67
134.96
134.19
134.14
130.30
129.76
129.73
129.42
129.38
128.33
128.28
127.85
127.36
127.28
127.19
125.40
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



2p-d_3

D
D
D
Cl
Cl
Cl
7.57
7.56
7.43
7.41
7.24
7.22
7.21
7.10
7.09
7.08
7.06
0.00
2.04
2.03
4.03
4.00
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
-0.5
-1.
f1 (ppm)

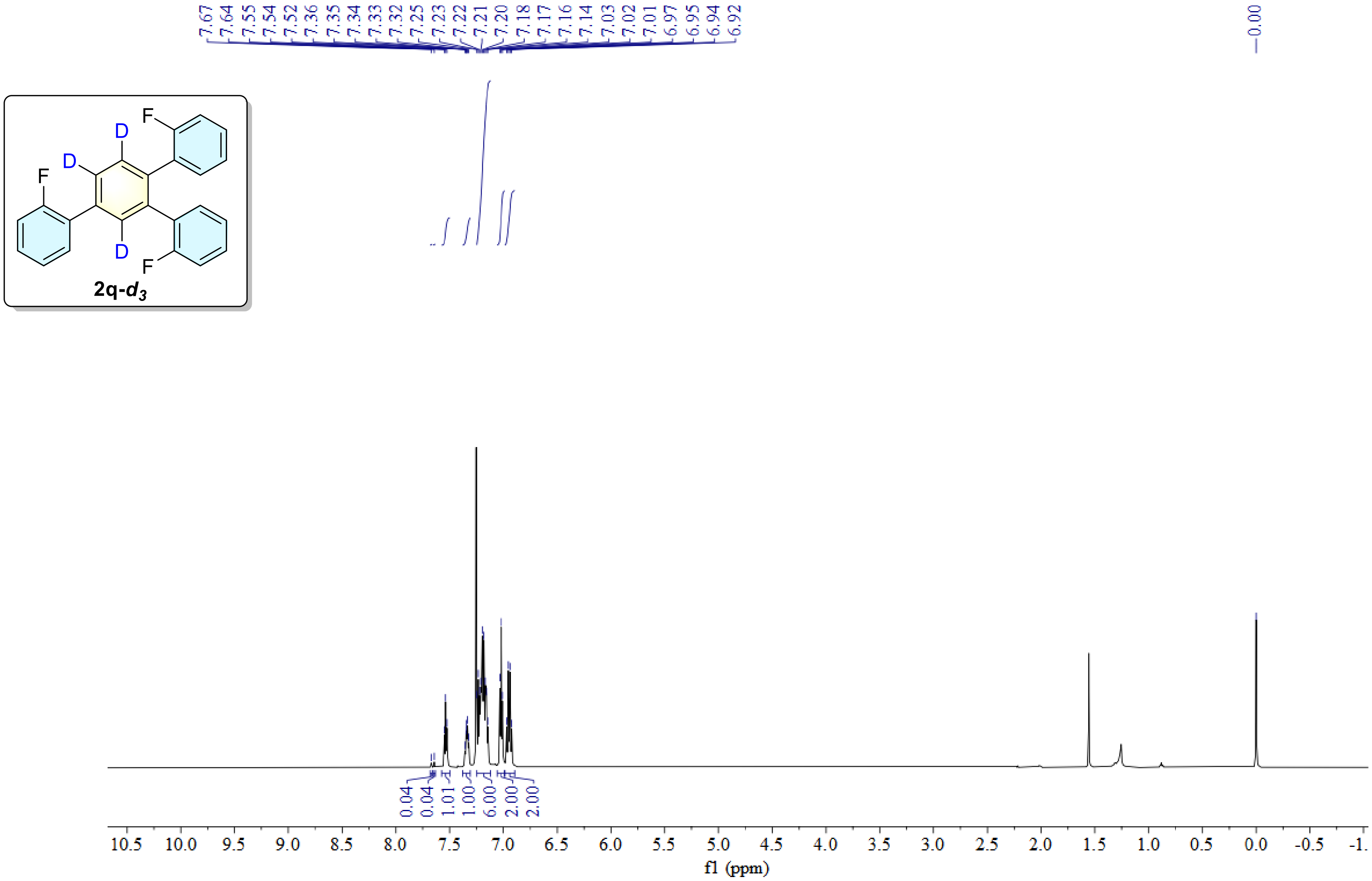

D
D
D
Cl
Cl
Cl

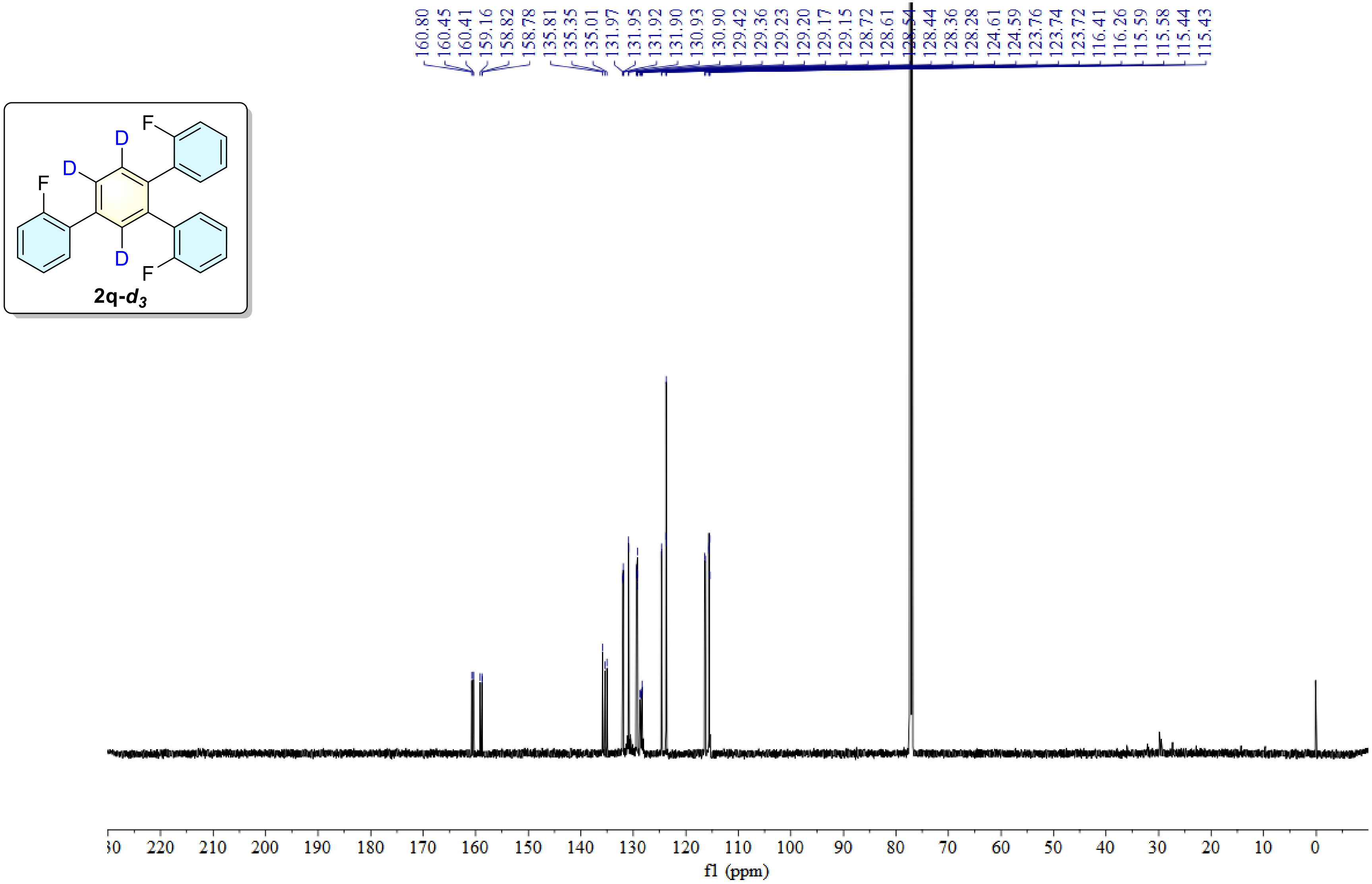
2p-*d*$_3$

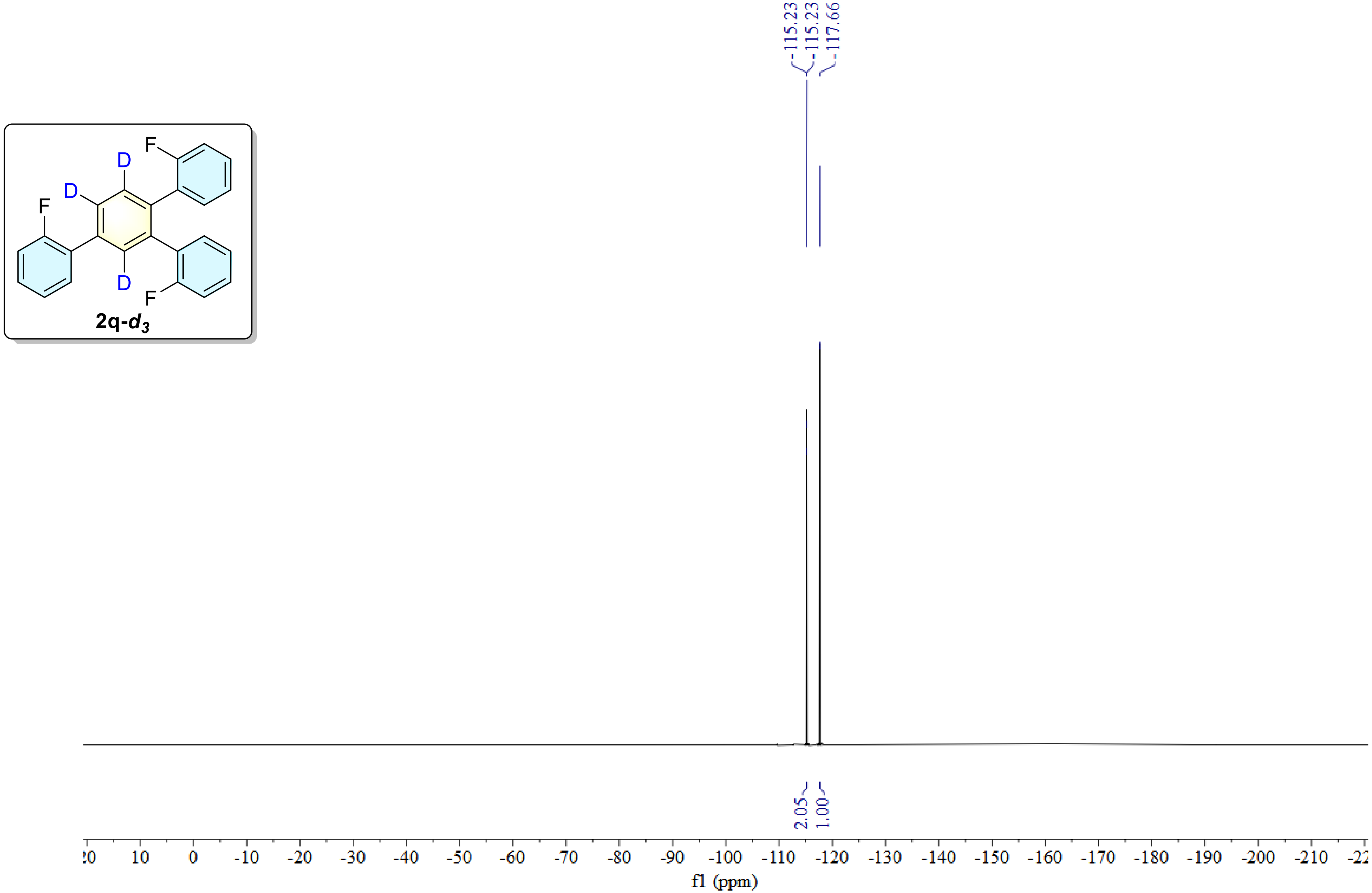
139.84
139.53
139.46
139.11
138.68
138.53
133.90
133.17
133.08
131.20
131.16
129.20
128.54
128.50
128.43
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

F
D
D
F
D
F

2q-d_3

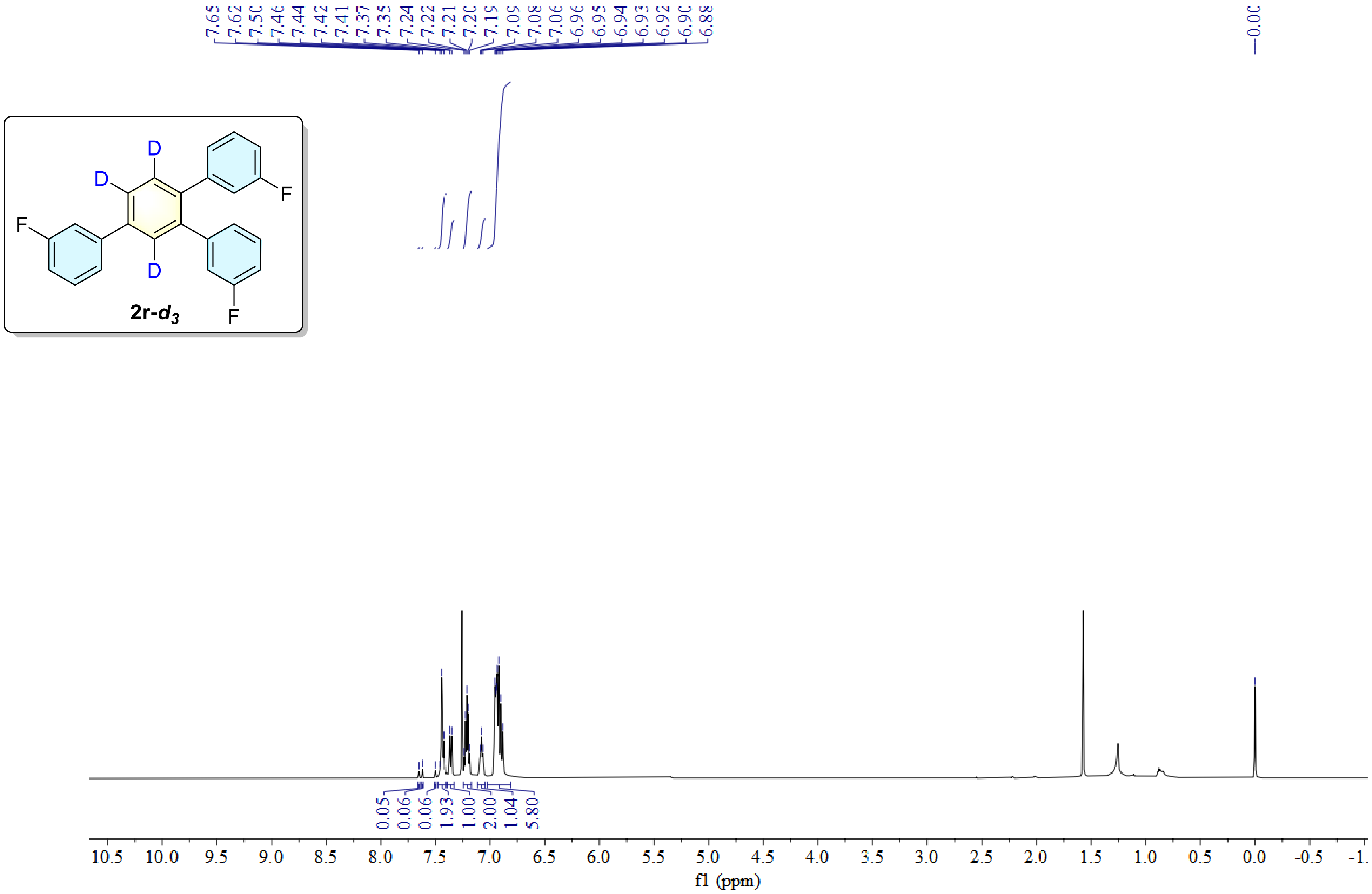

7.67
7.64
7.55
7.54
7.52
7.36
7.35
7.34
7.33
7.32
7.25
7.23
7.22
7.21
7.20
7.18
7.17
7.16
7.14
7.03
7.02
7.01
6.97
6.95
6.94
6.92
0.00
0.04
0.04
1.01
1.00
6.00
2.00
2.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
f1 (ppm)


160.80
160.45
160.41
159.16
158.82
158.78
135.81
135.35
135.01
131.97
131.95
131.92
131.90
130.93
130.90
129.42
129.36
129.23
129.20
129.17
129.15
128.72
128.61
128.54
128.44
128.36
128.28
124.61
124.59
123.76
123.74
123.72
116.41
116.26
115.59
115.58
115.44
115.43
F
D
D
F
D
F
2q-d_3
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



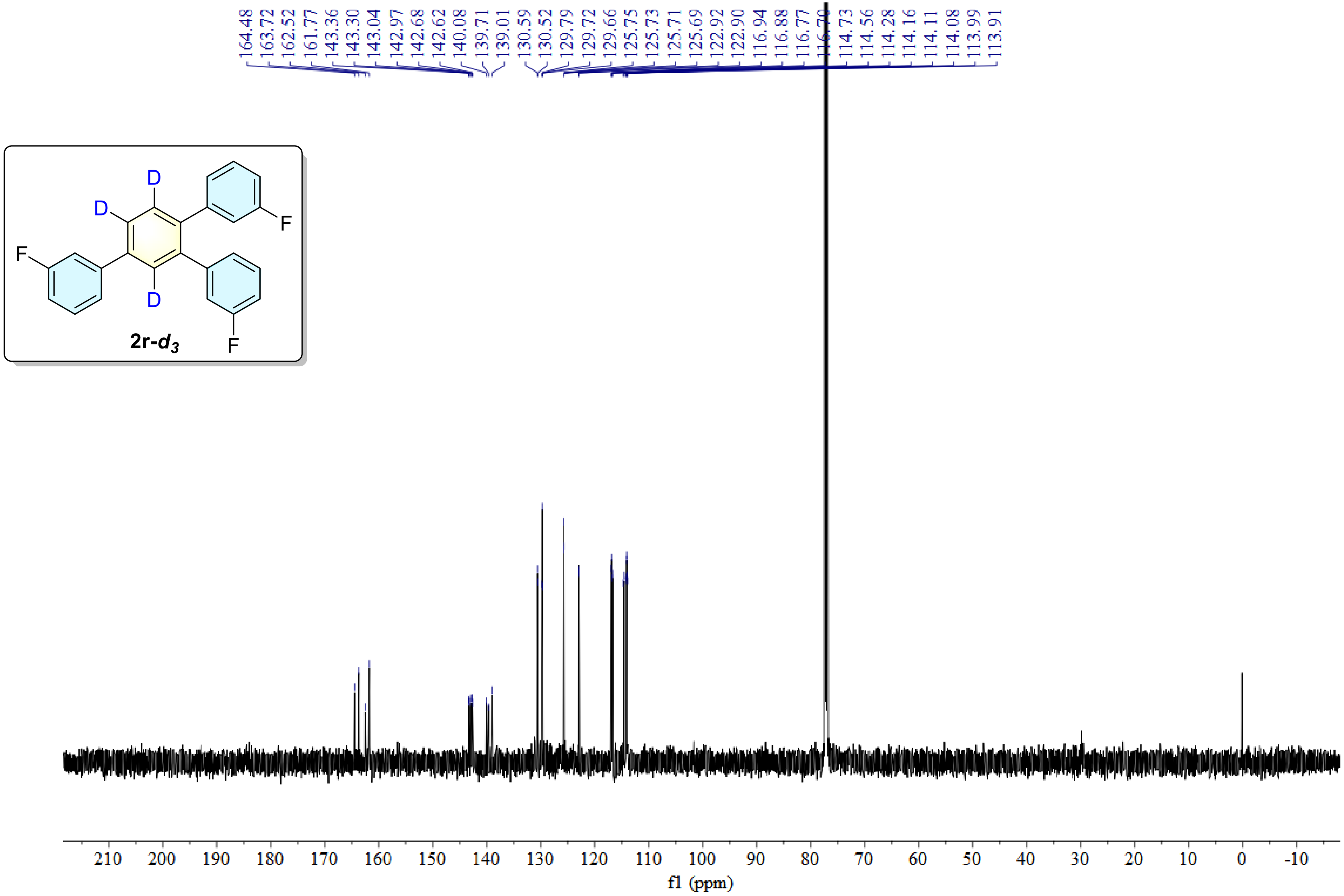
2q-*d*$_3$

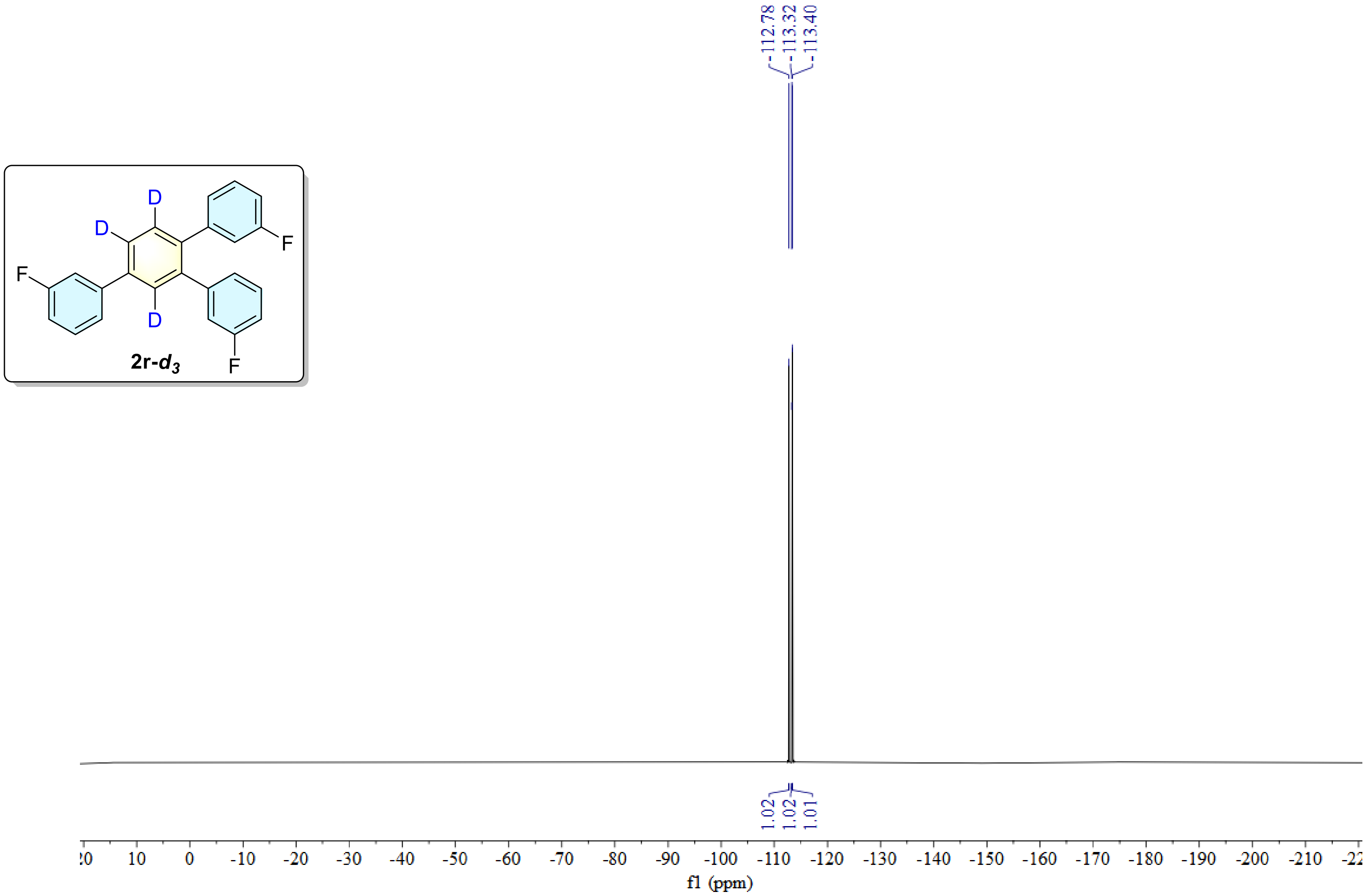
-115.23
-115.23
-117.66
2.05
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
f1 (ppm)

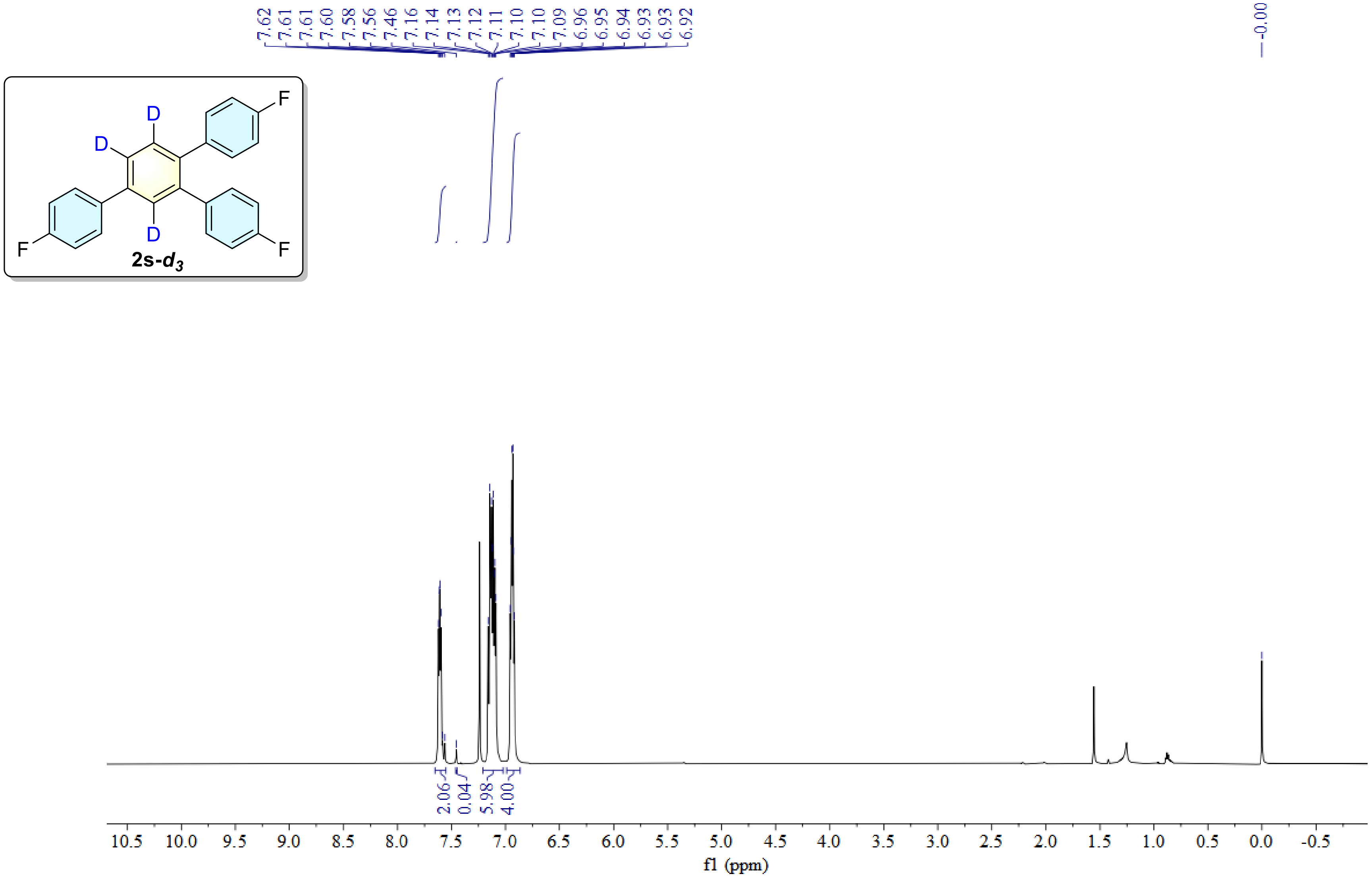


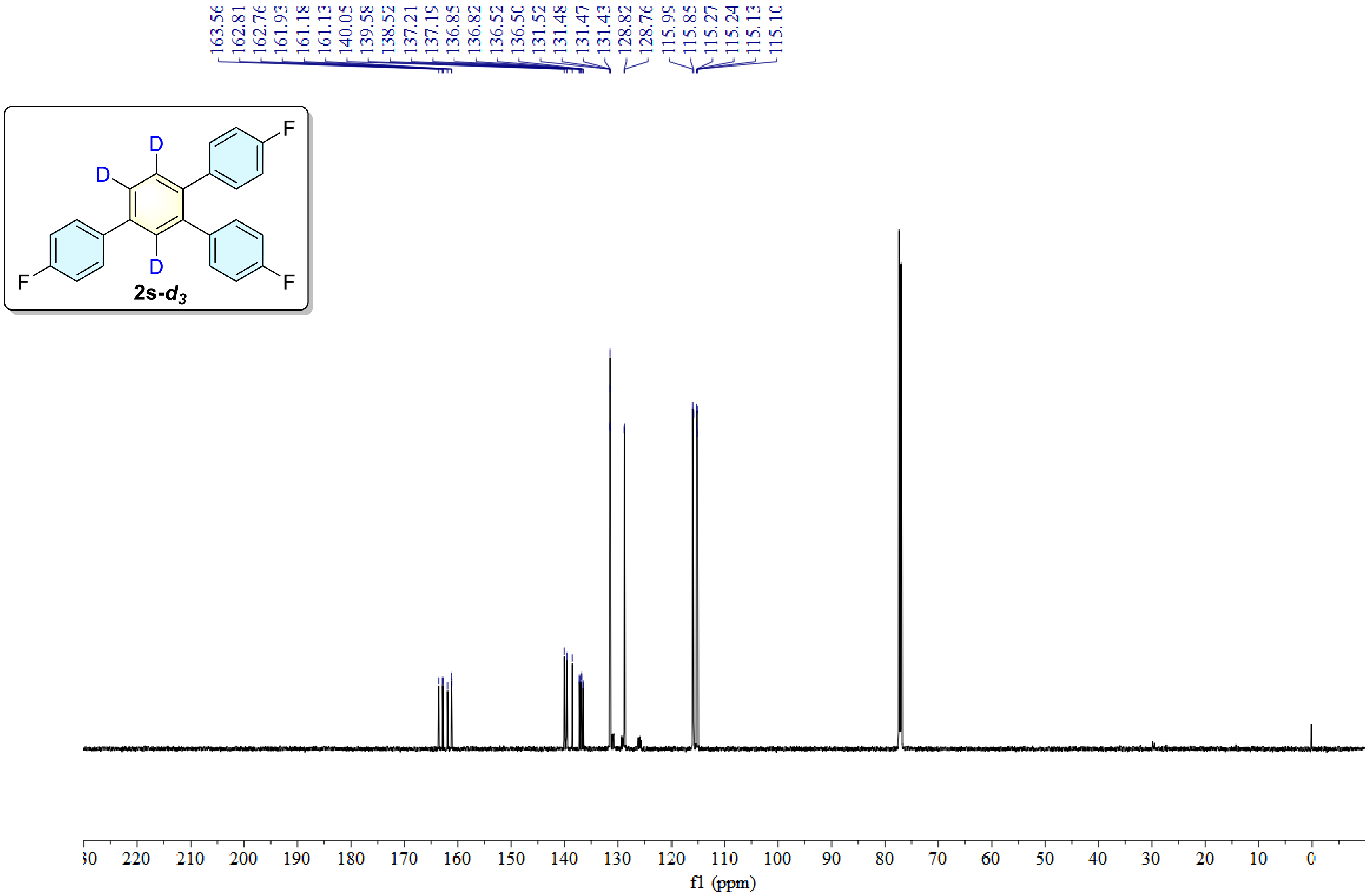
2r-*d*$_3$

D
D
D
F
F
F
7.65
7.62
7.50
7.46
7.44
7.42
7.41
7.37
7.35
7.24
7.22
7.21
7.20
7.19
7.09
7.08
7.06
6.96
6.95
6.94
6.93
6.92
6.90
6.88
0.00
0.05
0.06
0.06
1.93
1.00
2.00
1.04
5.80
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
-0.5
-1.
f1 (ppm)

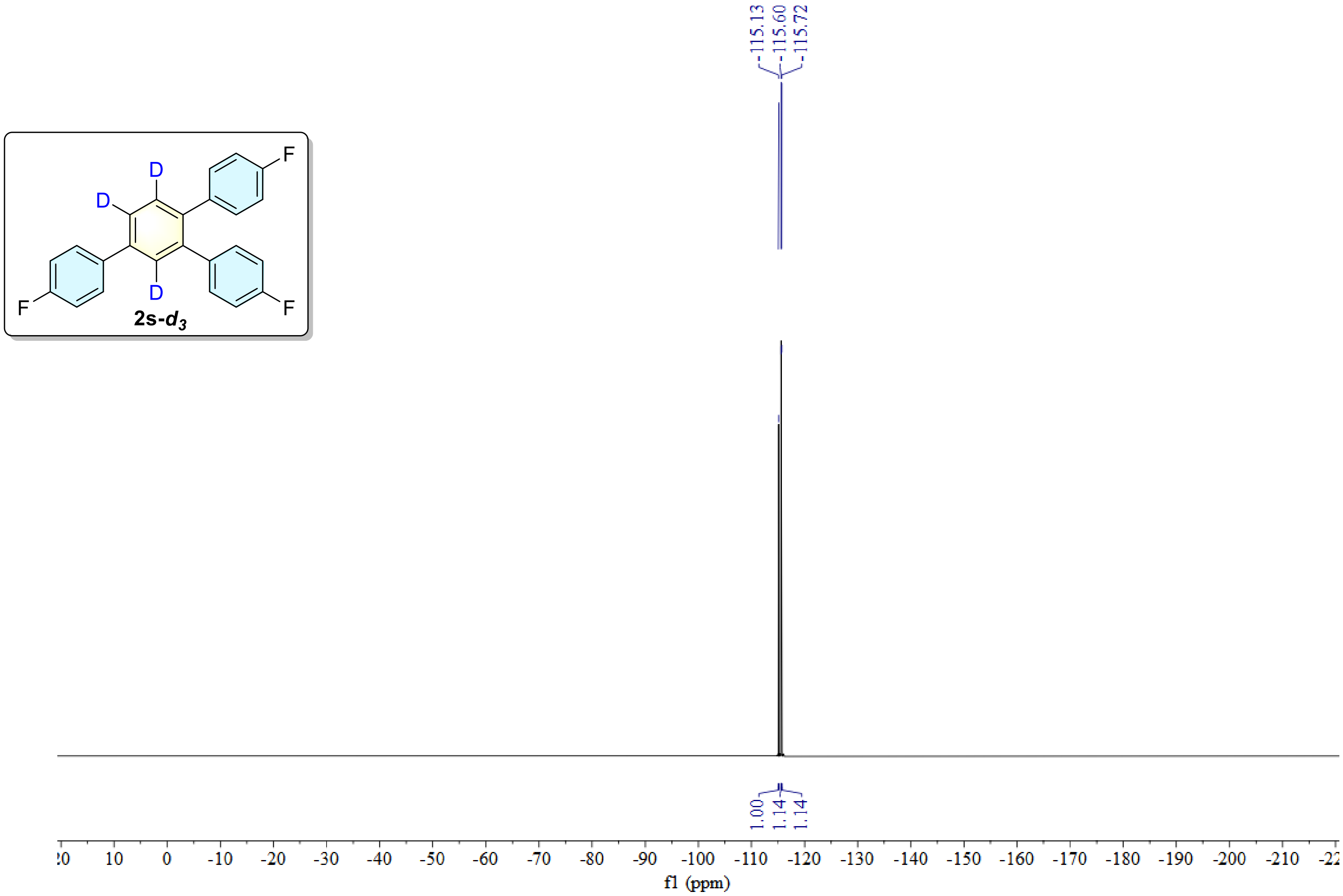

164.48
163.72
162.52
161.77
143.36
143.30
143.04
142.97
142.68
142.62
140.08
139.71
139.01
130.59
130.52
129.79
129.72
129.66
125.75
125.73
125.71
125.69
122.92
122.90
116.94
116.88
116.77
116.70
114.73
114.56
114.28
114.16
114.11
114.08
113.99
113.91
D
D
D
F
F
F
2r-d_3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

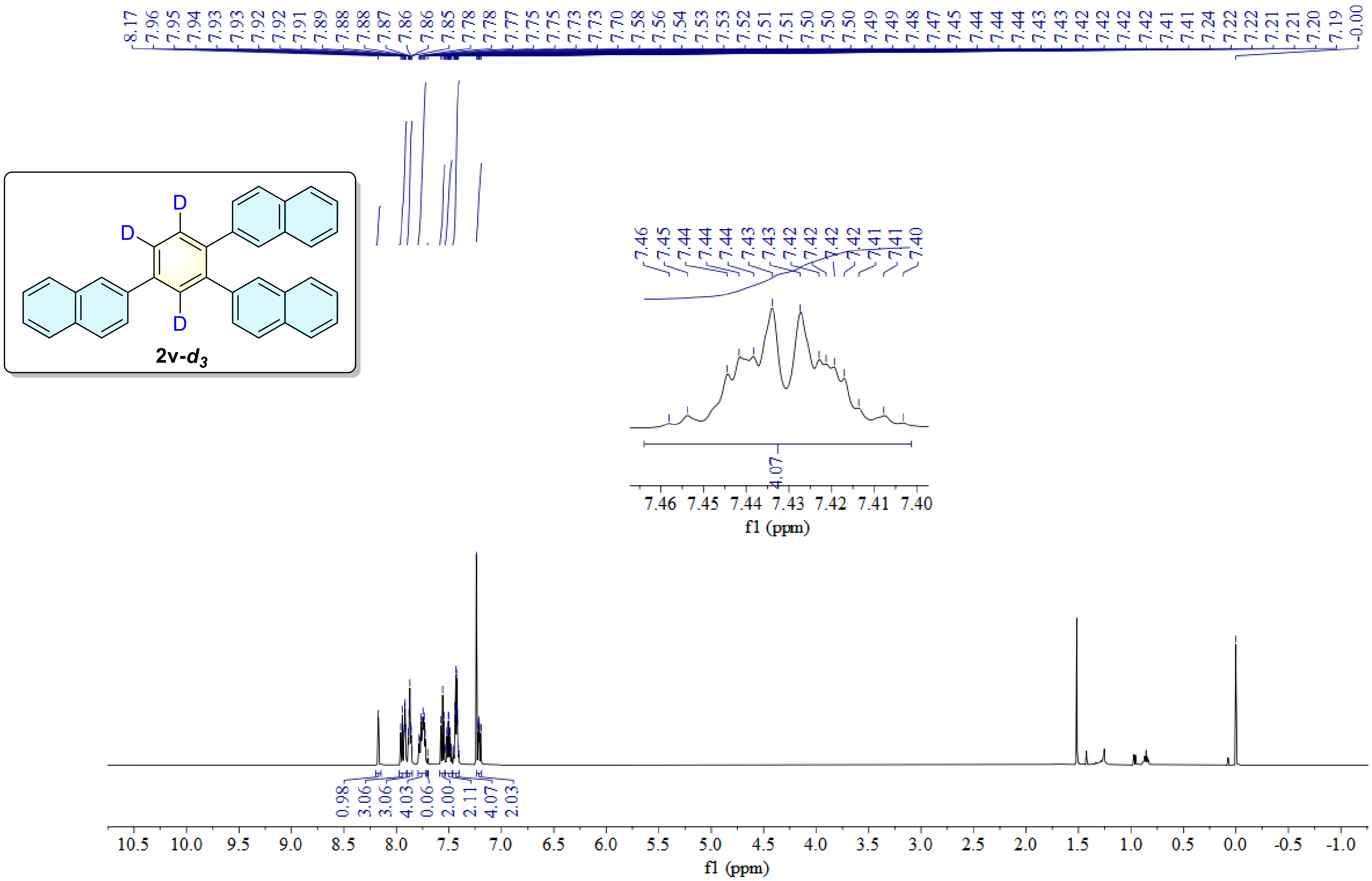

-112.78
-113.32
-113.40
D
D
D
F
F
F
2r-d_3
1.02
1.02
1.01
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
f1 (ppm)


7.62
7.61
7.61
7.60
7.58
7.56
7.46
7.16
7.14
7.13
7.12
7.11
7.10
7.10
7.09
6.96
6.95
6.94
6.93
6.93
6.92
-0.00
D
D
D
F
F
F
2s-d_3
2.06
0.04
5.98
4.00
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
-0.5
f1 (ppm)

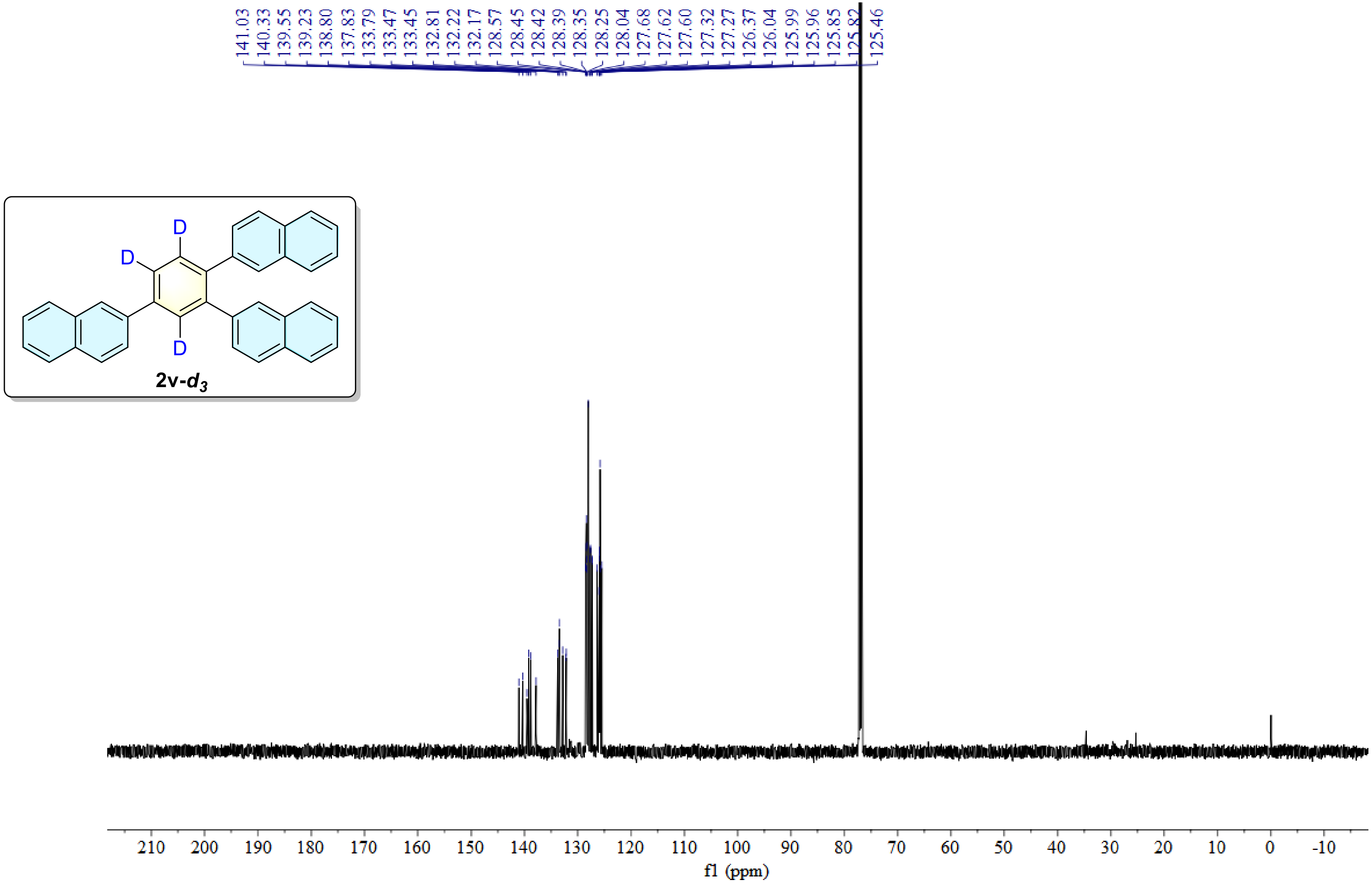

163.56
162.81
162.76
161.93
161.18
161.13
140.05
139.58
138.52
137.21
137.19
136.85
136.82
136.52
136.50
131.52
131.48
131.47
131.43
128.82
128.76
115.99
115.85
115.27
115.24
115.13
115.10
D
D
D
F
F
F
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2s-*d*$_3$


-115.13
-115.60
-115.72
D
D
D
F
F
F
2s-d_3
1.00
1.14
1.14
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-22
f1 (ppm)


8.17
7.96
7.95
7.94
7.93
7.93
7.92
7.92
7.91
7.89
7.88
7.88
7.87
7.86
7.86
7.85
7.78
7.78
7.77
7.75
7.75
7.73
7.73
7.70
7.58
7.56
7.54
7.53
7.53
7.52
7.51
7.51
7.50
7.50
7.50
7.49
7.49
7.48
7.47
7.45
7.44
7.44
7.44
7.43
7.43
7.42
7.42
7.42
7.42
7.41
7.41
7.24
7.22
7.22
7.21
7.21
7.20
7.19
-0.00
D
D
D
7.46
7.45
7.44
7.44
7.44
7.43
7.43
7.42
7.42
7.42
7.42
7.41
7.41
7.40
4.07
7.46 7.45 7.44 7.43 7.42 7.41 7.40
f1 (ppm)
0.98
3.06
3.06
4.03
0.06
2.00
2.11
4.07
2.03
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 -0.5 -1.0
f1 (ppm)

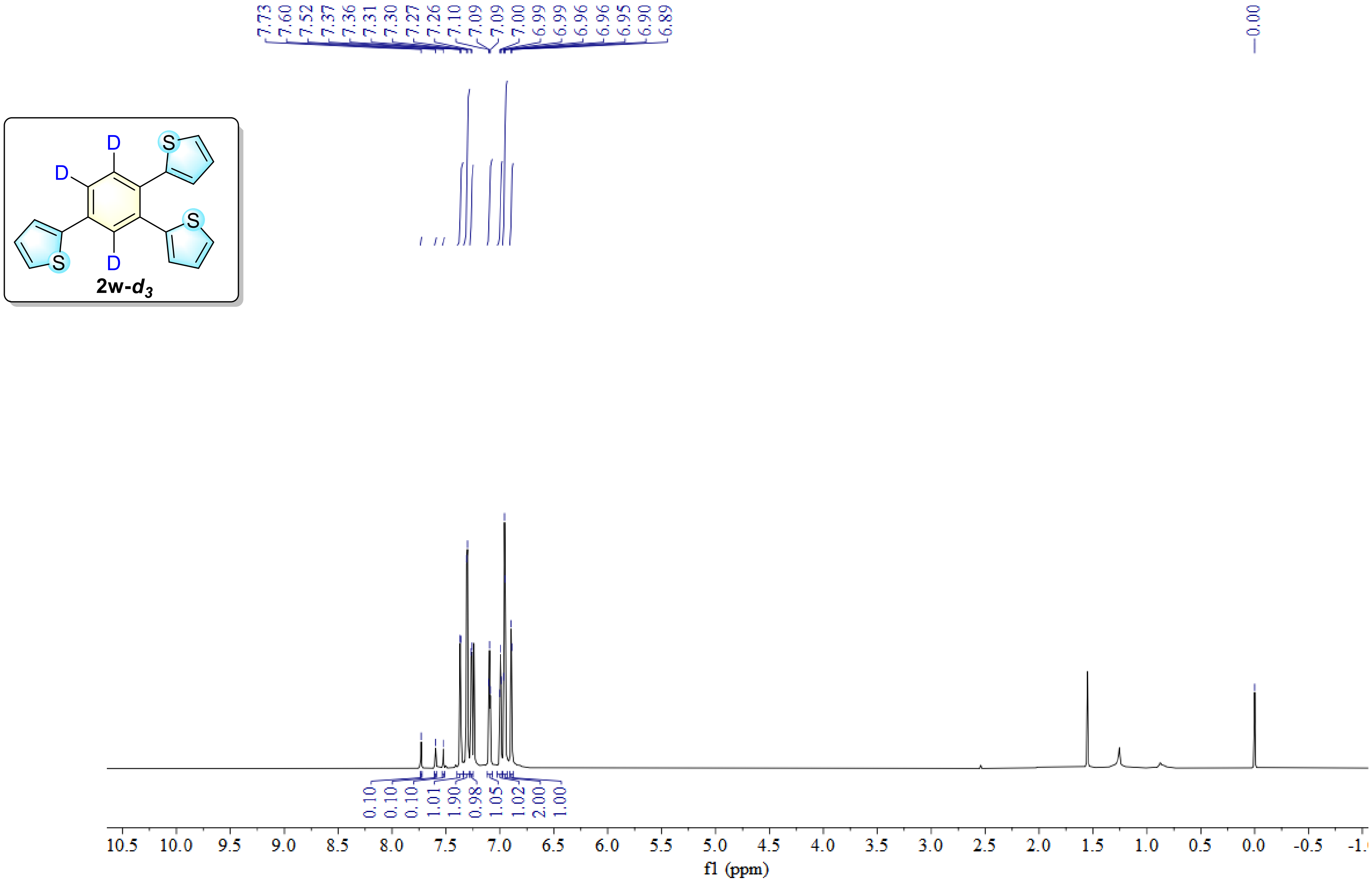
2v-d_3

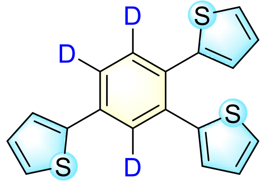


2v-*d*$_3$
141.03
140.33
139.55
139.23
138.80
137.83
133.79
133.47
133.45
132.81
132.22
132.17
128.57
128.45
128.42
128.39
128.35
128.25
128.04
127.68
127.62
127.60
127.32
127.27
126.37
126.04
125.99
125.96
125.85
125.82
125.46
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

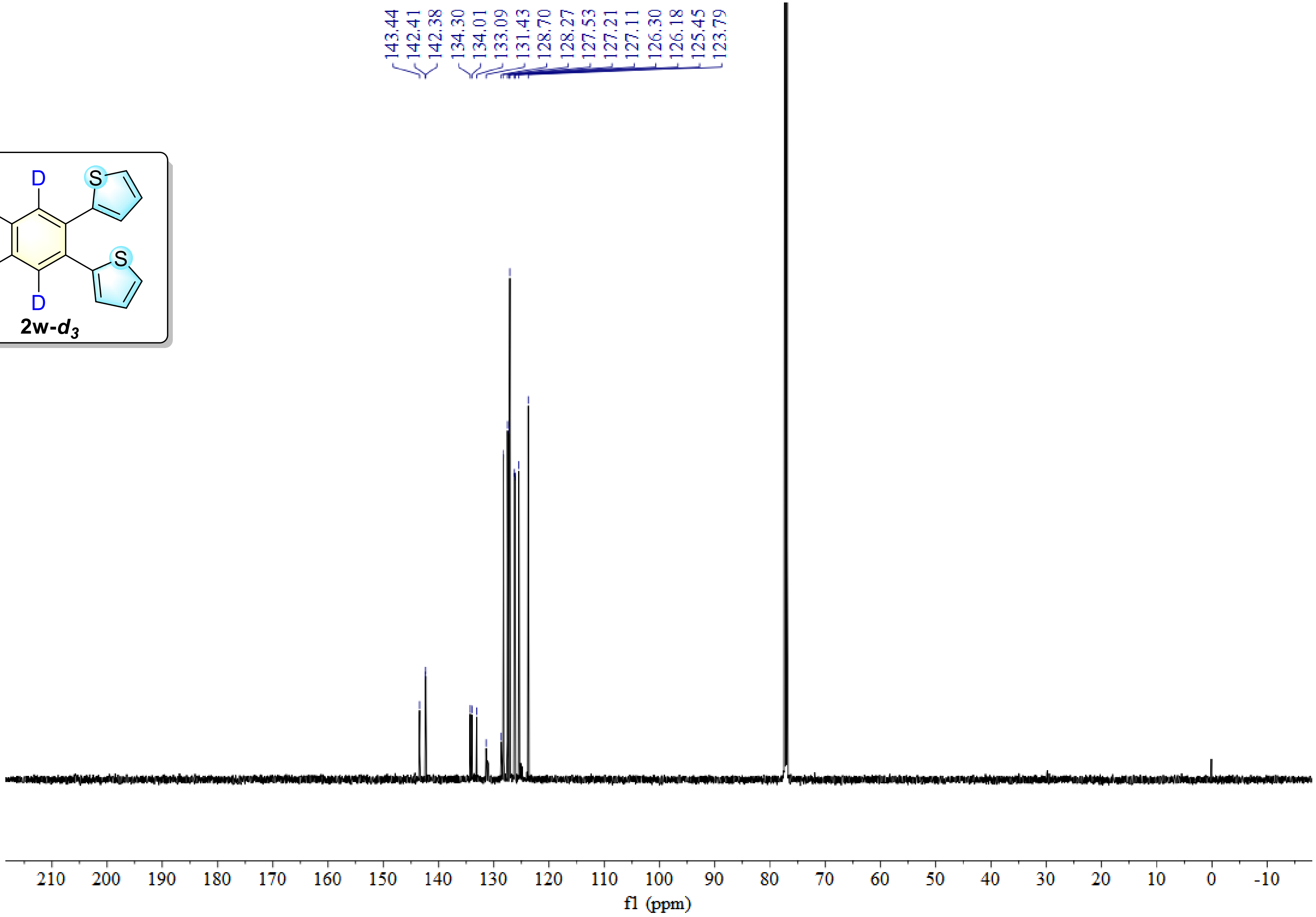
D
D
D
S
S
S

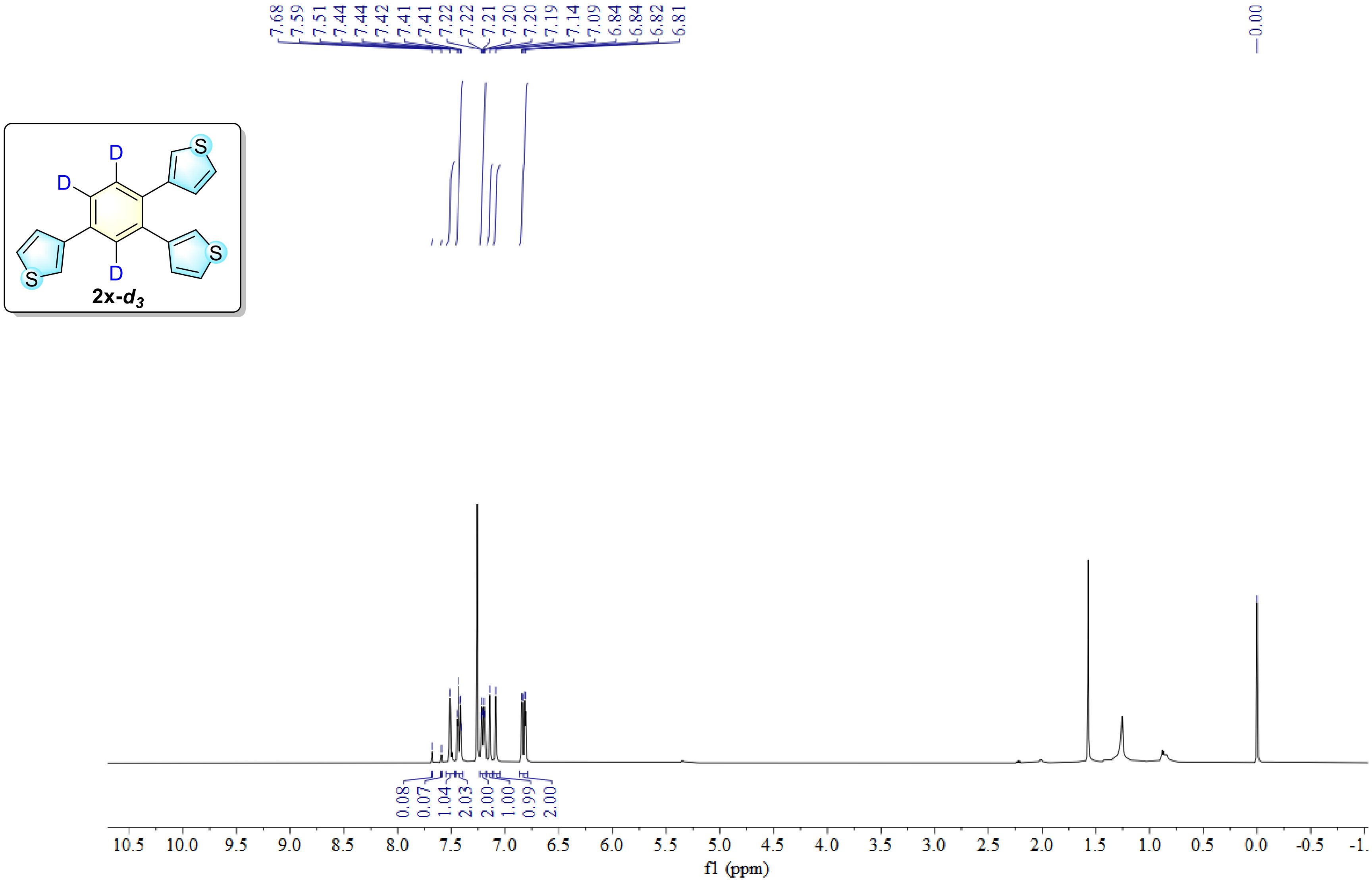
2w-d_3


7.73
7.60
7.52
7.37
7.36
7.31
7.30
7.27
7.26
7.10
7.09
7.09
7.00
6.99
6.99
6.96
6.96
6.95
6.90
6.89
0.00
0.10
0.10
0.10
1.01
1.90
0.98
1.05
1.02
2.00
1.00
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
-0.5
-1.
f1 (ppm)


D
D
S
S
S
D

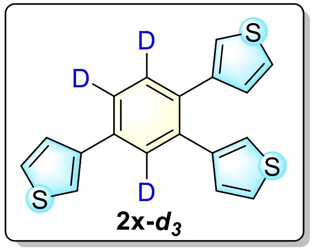
2w-d_3


143.44
142.41
142.38
134.30
134.01
133.09
131.43
128.70
128.27
127.53
127.21
127.11
126.30
126.18
125.45
123.79
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)

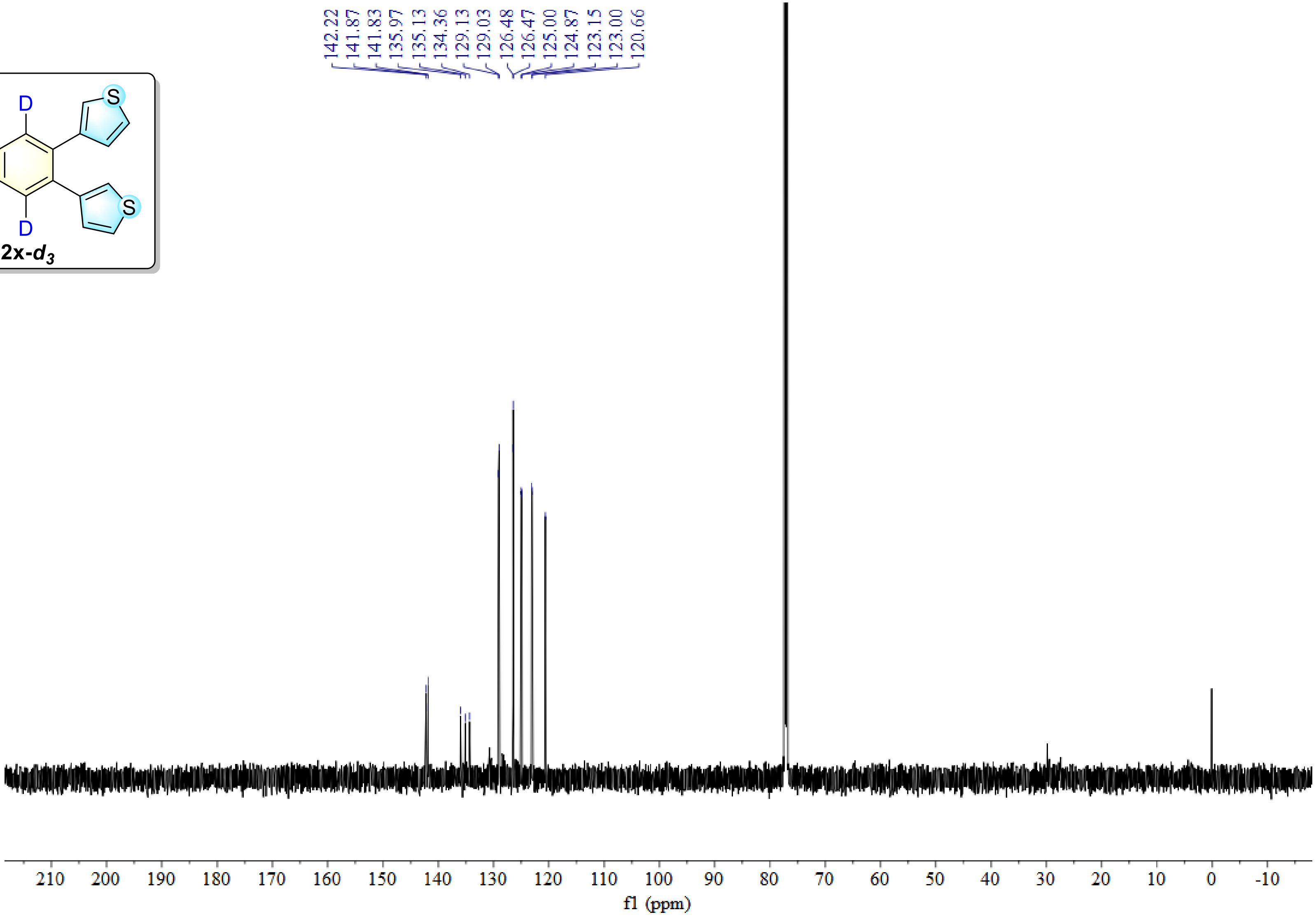

D
D
D
S
S
S

2x-*d*$_3$

7.68
7.59
7.51
7.44
7.44
7.42
7.41
7.41
7.22
7.22
7.21
7.20
7.20
7.19
7.14
7.09
6.84
6.84
6.82
6.81
0.00
0.08
0.07
1.04
2.03
2.00
1.00
0.99
2.00
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
-0.5
-1.
f1 (ppm)


D
D
D
S
S
S

2x-d_3


142.22
141.87
141.83
135.97
135.13
134.36
129.13
129.03
126.48
126.47
125.00
124.87
123.15
123.00
120.66
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)