

Electronic Supplementary Information

Pd-Catalyzed Sequential B(3)–I/B(4)–H Bond Activation for the Synthesis of 3,4-Benzo-*o*-Carboranes

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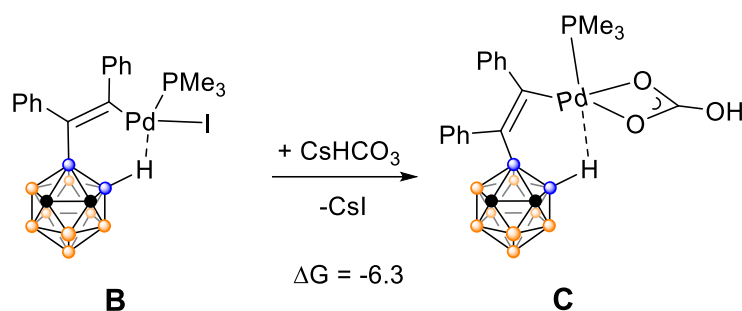
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DFT Calculation



Scheme S1. Energy (ΔG_{gas} , kcal/mol) calculated for the formation of intermediate **C** at the B3LYP-D3/6-31g** level of theory.

Computational Details. Geometry optimizations were carried out with the Gaussian09 program, Revision D.01¹ at the B3LYP²-D3³ level of density functional theory. The Los Alamos National Laboratory (LANL) effective core potentials (ECP) with the appropriate valence basis set of double- ζ quality (denoted LANL2DZ)⁴ was used for Pd, Cs and I while the 6-31G(d,p) basis set was used for all other atoms. Frequency calculations were made to determine the characteristics of all stationary points as energy minima. Orbital energies of compound **5a** were calculated at the B3LYP/6-311+G(d,p) level of theory. The graphics of the molecular orbitals were produced by using the visualizing software VMD.⁵

The Cartesian coordinates of **B**

0 1

B	-1.83516400	-1.61190300	0.75960100
B	-3.15619900	-2.72201300	0.35431100
B	-3.55117300	-0.24127500	-1.07000500
B	-4.23954100	-1.86021600	-0.77651900
B	-4.91108800	-0.44523400	0.06911400
B	-4.26791900	-0.44172000	1.72942400
B	-2.47024700	-1.64320700	-0.90456800
B	-2.02909500	-0.08924100	-0.15543400
H	-0.74329000	-1.87694100	1.22107300

H	-2.98685900	-3.89333100	0.32866800
H	-3.68849900	0.44817400	-2.02232000
H	-4.85936400	-2.41540100	-1.62187300
H	-5.93259100	0.11736500	-0.13247800
H	-4.75129800	0.14840000	2.63108400
H	-1.84034800	-2.04252900	-1.82616400
B	-3.17853900	-1.84057300	1.90199300
H	-2.96155500	-2.24777400	2.99099300
B	-4.68004900	-1.97606000	0.95606000
H	-5.60408600	-2.60770400	1.34650200
C	-3.54569000	0.48395300	0.46247500
C	-2.58094700	-0.29612900	1.49561900
H	-3.59690200	1.55976100	0.57912000
H	-2.04801000	0.32366800	2.20637600
C	-0.80125200	0.86974300	-0.31553300
C	0.44061400	0.32365300	-0.38066600
C	-1.05466000	2.33645600	-0.15747800
C	-0.49621000	3.05563500	0.91273800
C	-1.91323500	3.01357300	-1.04098600
C	-0.77551900	4.41086600	1.08598500
H	0.17237700	2.54324100	1.59801700
C	-2.19123500	4.37066700	-0.86867600
H	-2.35447000	2.46997400	-1.87131000
C	-1.62364900	5.07427000	0.19578000
H	-0.33130900	4.94952200	1.91828600
H	-2.85054300	4.87776400	-1.56763500
H	-1.84152700	6.12950100	0.33148600
C	1.69123800	1.11354300	-0.44025600
C	1.82527400	2.21658100	-1.30700400
C	2.80463200	0.74915700	0.34097900

C	3.02550400	2.91928900	-1.39241300
H	0.98157000	2.50995100	-1.92213700
C	4.00191100	1.46103300	0.26433900
H	2.72296500	-0.10543800	1.00733000
C	4.12028200	2.54601500	-0.60772400
H	3.10699700	3.76016500	-2.07553400
H	4.84363500	1.16461000	0.88392400
H	5.05516200	3.09439000	-0.67626100
Pd	0.61682000	-1.72503800	-0.17333200
I	1.05910300	-4.35885200	0.60529400
P	1.98934900	-1.94403500	-1.98319200
C	3.73004000	-2.29237500	-1.52817500
H	3.74482700	-3.16118100	-0.86686300
H	4.33074000	-2.48225200	-2.42350200
H	4.12843900	-1.42573400	-0.99477300
C	1.45455800	-3.36104900	-3.02295900
H	2.10533000	-3.44827100	-3.89935600
H	1.48921400	-4.27322400	-2.42643900
H	0.42377100	-3.19513400	-3.34549100
C	2.11710400	-0.58572500	-3.21331500
H	2.67261000	-0.94796000	-4.08408800
H	1.11564000	-0.27695200	-3.52151200
H	2.63184500	0.27337100	-2.78495400

The Cartesian coordinates of **CsHCO₃**

0 1

C	2.90238900	-3.35135800	-0.58674000
O	2.62446600	-2.70424100	-1.63944000
O	2.14670200	-4.06164500	0.11507100
Cs	-0.22150200	-3.46234300	-1.49139400

O	4.21821600	-3.25982300	-0.14774000
H	4.63797400	-2.68216000	-0.80072900

The Cartesian coordinates of **CsI**

0 1

Cs	-0.52125400	-3.14224100	-0.93358600
I	-4.17029600	-3.14224100	-0.93358600

The Cartesian coordinates of **C**

0 1

B	-1.82924300	-1.62933900	1.26027100
B	-2.98385800	-2.76780100	0.52743700
B	-2.46691000	-0.67214600	-1.37549200
B	-3.38182500	-2.17425900	-1.10886700
B	-4.19722800	-0.60619500	-0.93856000
B	-4.31930900	-0.23574100	0.79802600
B	-1.71406500	-2.00705700	-0.47683000
B	-1.45673900	-0.33405900	0.06910900
H	-1.02940200	-1.83749800	2.10366700
H	-2.95281000	-3.92231900	0.79943500
H	-2.10990800	-0.20445700	-2.40459100
H	-3.63322600	-2.90092100	-2.01346400
H	-4.96550400	-0.08525100	-1.67364400
H	-5.07256400	0.54900200	1.25915400
H	-0.81560100	-2.63628800	-0.92443600
B	-3.55925800	-1.56369400	1.70107100
H	-3.88000200	-1.72527800	2.82894300
B	-4.52783000	-1.89771200	0.24240100
H	-5.59068500	-2.41995800	0.30889600
C	-3.03758000	0.36802400	-0.17465300

C	-2.67846300	-0.16589700	1.30700000
H	-3.02665000	1.44240900	-0.30421500
H	-2.44680000	0.60098900	2.03638300
C	-0.22038700	0.64068400	0.13962100
C	1.05071200	0.21588700	0.31758400
C	-0.51333000	2.11354500	0.13279800
C	-0.33692300	2.87991300	1.29502100
C	-0.99408300	2.75186400	-1.02334500
C	-0.62568700	4.24535200	1.30101600
H	0.04730300	2.39539000	2.18797900
C	-1.28333400	4.11743100	-1.01825300
H	-1.13287100	2.16869800	-1.93036600
C	-1.10108700	4.86862600	0.14544000
H	-0.47795900	4.82281900	2.20929100
H	-1.64758200	4.59497000	-1.92368000
H	-1.32656200	5.93096200	0.15037500
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C	3.13758500	1.02792300	1.45307300
C	3.70385900	2.74295100	-0.67647300
H	1.84887600	2.05258600	-1.52412600
C	4.28973200	1.81126600	1.47266100
H	2.91749200	0.35337000	2.27596700
C	4.58282400	2.66607800	0.40590600
H	3.91836600	3.40987700	-1.50701900
H	4.96460300	1.75099500	2.32189600
H	5.48637700	3.26860200	0.42021700
Pd	1.55065900	-1.69141100	0.66034400
C	1.82923200	-3.11991600	2.74056200
O	2.11537100	-3.62820500	1.61991100

O	1.41816500	-1.91960700	2.88249900
O	1.96726900	-3.89333300	3.83502000
H	1.69281900	-3.34964100	4.58886500
P	2.08415600	-1.91735500	-1.51990200
C	3.81309200	-1.38748300	-1.84690800
H	3.90388900	-0.32169500	-1.62289300
H	4.48456200	-1.94054500	-1.18586700
H	4.08879300	-1.57176600	-2.89024400
C	1.11257300	-1.05815100	-2.81731700
H	0.06995700	-1.37321500	-2.76122100
H	1.15748100	0.01785000	-2.64183800
H	1.51602900	-1.28749200	-3.80838300
C	2.03495500	-3.69090700	-1.99052200
H	2.60231400	-4.26240500	-1.25189000
H	0.99845100	-4.03577200	-1.96528700
H	2.45221200	-3.84625500	-2.99040600

The Cartesian coordinates of **5a**

0 1

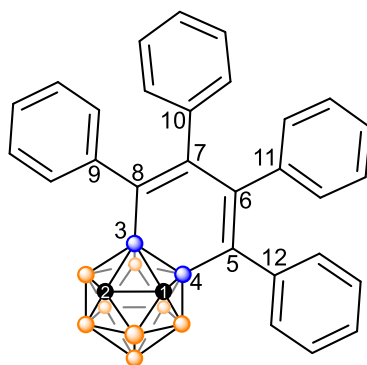
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C	1.67420800	-3.06176800	0.64932600
H	2.61317400	-2.81415200	1.13034300
C	1.53254100	-0.32326900	-0.19306000
C	0.67577500	0.73184200	-0.03402200
C	-0.81331100	0.61237700	0.00352200
C	-1.49818100	-0.57091100	0.06405200
C	-2.98692700	-0.61515100	-0.02810100
C	-3.75903100	-1.12768100	1.02479200
H	-3.26689600	-1.46243700	1.93191300

C	-5.14852400	-1.19195100	0.92513800
H	-5.72992700	-1.58033400	1.75649300
C	-5.78906800	-0.76311600	-0.23826000
H	-6.87066600	-0.81856000	-0.31891100
C	-5.02956900	-0.26504900	-1.29874600
H	-5.51836200	0.06785800	-2.20983200
C	-3.64220900	-0.18983100	-1.19390600
H	-3.05312300	0.20672400	-2.01419700
C	-1.58463200	1.89766100	-0.05728600
C	-2.42270300	2.27723600	0.99895100
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C	-3.14991000	3.46504200	0.94035100
H	-3.79443000	3.74363600	1.76907500
C	-3.05316900	4.29074200	-0.18066400
H	-3.62018800	5.21578000	-0.22851000
C	-2.22379900	3.92011100	-1.24111200
H	-2.14374700	4.55536300	-2.11847200
C	-1.49294600	2.73544200	-1.17718600
H	-0.84026300	2.45483000	-1.99801600
C	1.24322900	2.11269800	0.10301600
C	1.02828500	2.86203100	1.26809300
H	0.41750400	2.44601300	2.06337900
C	1.57773300	4.13513800	1.40591800
H	1.40345400	4.70131400	2.31627000
C	2.34320300	4.68400800	0.37503500
H	2.76632600	5.67871700	0.47966600
C	2.55837300	3.94865300	-0.79140800
H	3.15110000	4.36787200	-1.59920500
C	2.01503000	2.67175700	-0.92385900
H	2.19112100	2.09462400	-1.82597200

C	3.01543400	-0.17600400	-0.15789200
C	3.79929100	-0.58478900	-1.24881600
H	3.30973800	-0.96103400	-2.14110500
C	5.18996000	-0.48996200	-1.20341000
H	5.77777000	-0.79850300	-2.06308300
C	5.82436600	-0.00218100	-0.05959200
H	6.90739600	0.06792300	-0.02311400
C	5.05690300	0.39595400	1.03691600
H	5.54086500	0.77837300	1.93099700
C	3.66646500	0.30985400	0.98830400
H	3.07072600	0.64062500	1.83330400
B	0.96529000	-1.76948500	-0.25815400
B	-0.76625100	-1.95623100	0.04057700
B	-1.01986900	-3.33753600	1.15820900
H	-1.82083300	-3.26585200	2.02535500
B	0.57162000	-4.01929600	1.57706900
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B	1.56728300	-3.26338200	-1.03444500
H	2.56447500	-3.20456400	-1.66592800
B	-0.02738800	-2.60256800	-1.45915800
H	-0.23059600	-2.07008500	-2.49935700
B	-1.27086800	-3.55203600	-0.58959200
H	-2.35917300	-3.71246100	-1.03117100
B	-0.43285800	-4.81946600	0.36740500
H	-0.90595800	-5.88107000	0.60217700
B	1.32597700	-4.62651100	0.08792000
H	2.16038100	-5.45936000	0.18952700
B	0.18204700	-4.35910900	-1.25195200
H	0.13969600	-5.10489700	-2.17364800

Table S1. Selected experimental and calculated structural parameters of **5a**.

(calculated at the B3LYP-D3/6-31G(d, p) level of theory)



5a

Bond Length (Å)	Experimental	Computational
C(1)-C(2)	1.625(2)	1.617
B(3)-B(4)	1.764(2)	1.769
B(4)-C(5)	1.563(2)	1.561
C(5)-C(6)	1.358(2)	1.371
C(6)-C(7)	1.498(2)	1.501
C(7)-C(8)	1.366(2)	1.371
C(8)-B(3)	1.558(2)	1.574
C(8)-C(9)	1.502(2)	1.499
C(7)-C(10)	1.500(2)	1.506
C(6)-C(11)	1.504(2)	1.504
C(5)-C(12)	1.493(2)	1.498

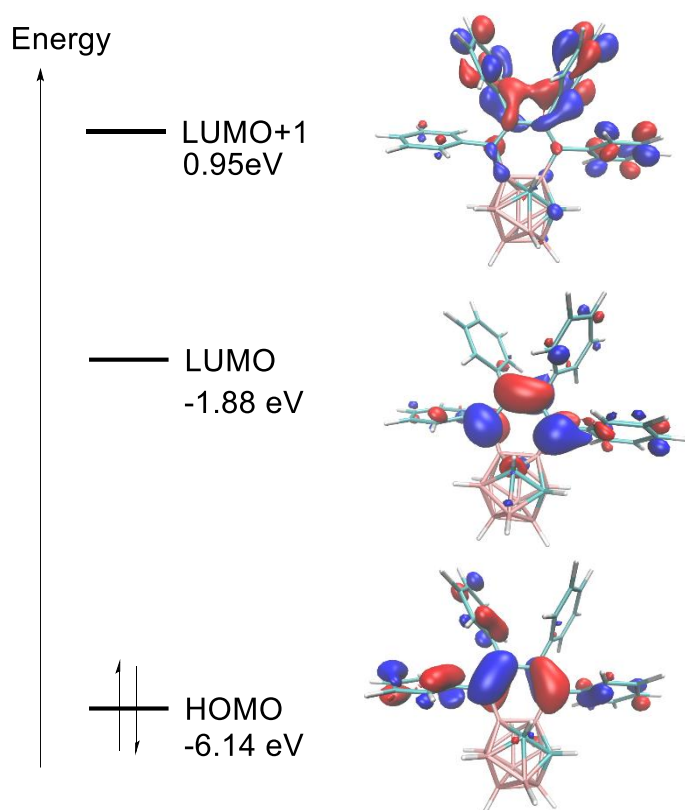


Figure S1. Plots of the LUMO+1, LUMO and HOMO orbitals of **5a** calculated at the B3LYP/6-311+G(d, p) level of theory. These molecular orbitals display the orbital features of π and π^* orbitals in typical 1,3-dienes.

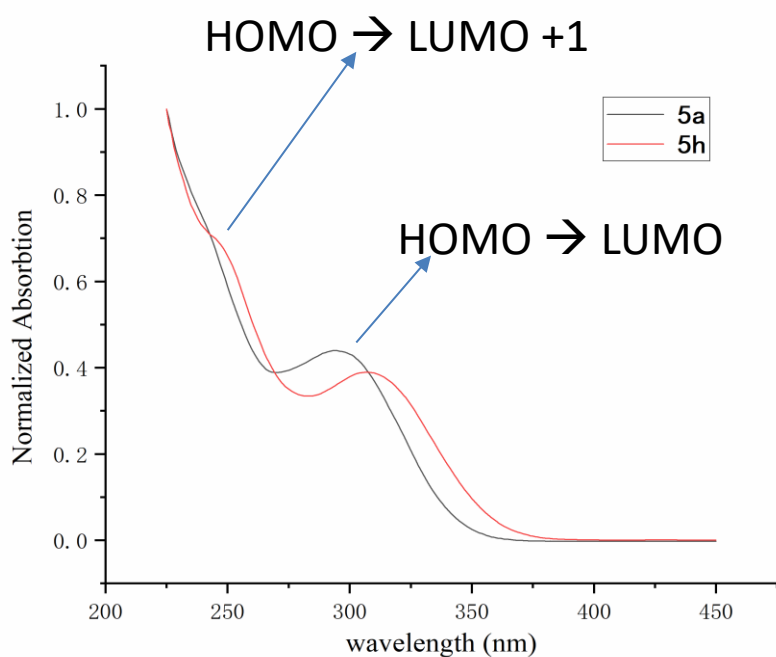


Figure S2. UV–visible spectra of **5a** and **5h** in CH_2Cl_2 (1×10^{-4} M) at room temperature.

Table S2. Crystal Data and Summary of Data Collection and Refinement for **4a**, **5a** and **5h**.

Compound No.	4a	5a	5h
formula	C ₁₆ H ₂₂ B ₁₀	C ₃₀ H ₃₀ B ₁₀	C ₄₆ H ₆₂ B ₁₀
crystal size(mm ³)	0.12 x 0.08 x 0.02	0.25 x 0.2 x 0.16	0.2 x 0.1 x 0.05
fw	322.43	498.64	723.05
crystal system	Monoclinic	triclinic	triclinic
space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> -1	<i>P</i> -1
<i>a</i> , Å	9.733(2)	11.347(1)	12.911(2)
<i>b</i> , Å	21.183(4)	15.270(1)	15.490(3)
<i>c</i> , Å	10.197(2)	17.571(2)	26.271(4)
α , deg	90	85.718(2)	96.100(3)
β , deg	117.947(4)	75.442(2)	90.510(3)
γ , deg	90	76.838(2)	112.747(3)
<i>V</i> , Å ³	1857.2(6)	2868.8(5)	4810.8(13)
<i>Z</i>	4	4	4
<i>D</i> _{calcd} , Mg/m ³	1.153	1.154	0.998
radiation(λ)Å	0.71073	0.71073	0.71073
2 θ range, deg	3.8 to 61.3	3.6 to 61.7	3.1 to 51.0
μ , mm ⁻¹	0.057	0.060	0.053
F(000)	672	1040	1552
no. of obsd reflns	5718	17777	17903
no. of params refnd	235	721	1123
goodness of fit	1.022	0.998	0.988
R1	0.0528	0.0599	0.0839
wR2	0.1271	0.1313	0.2168

References

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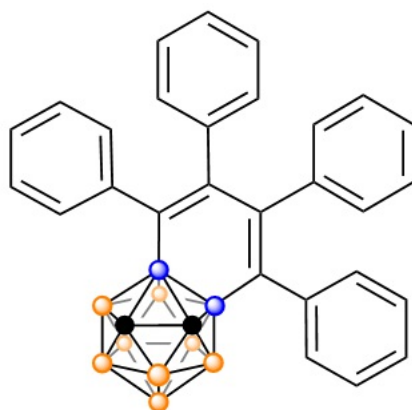
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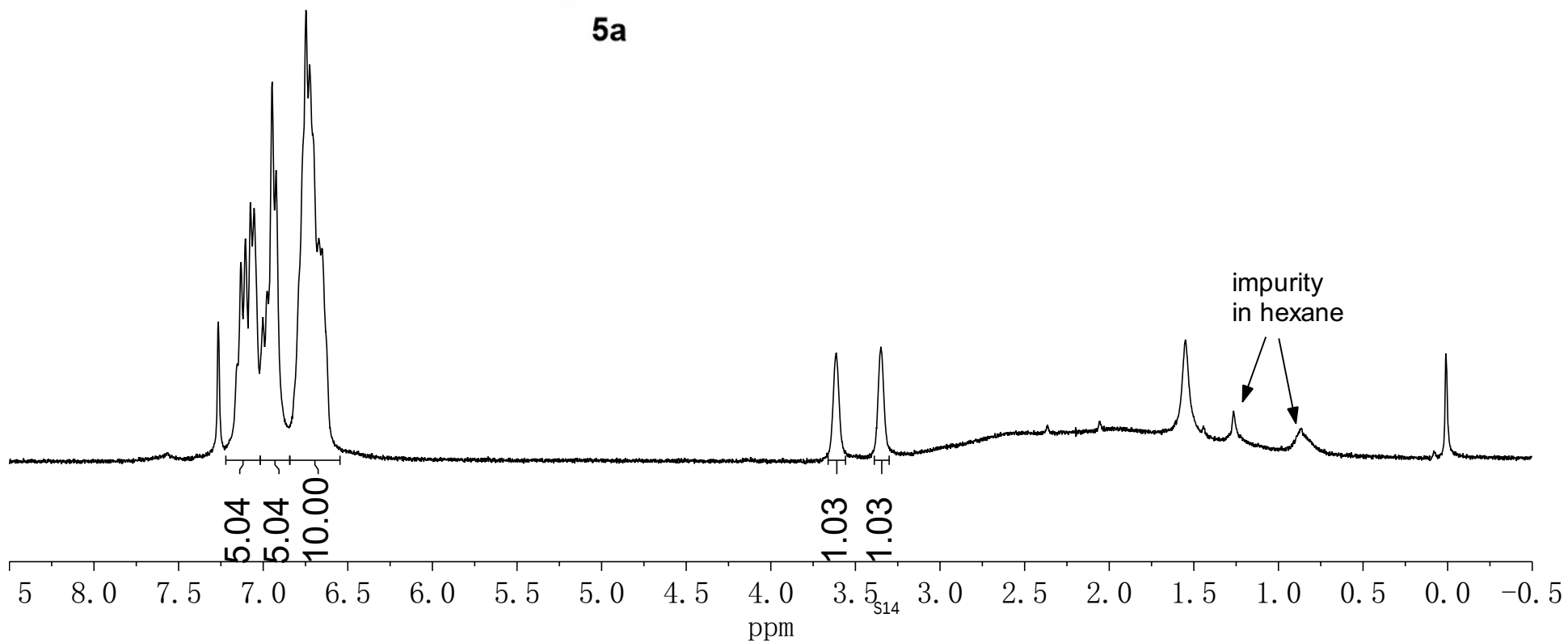
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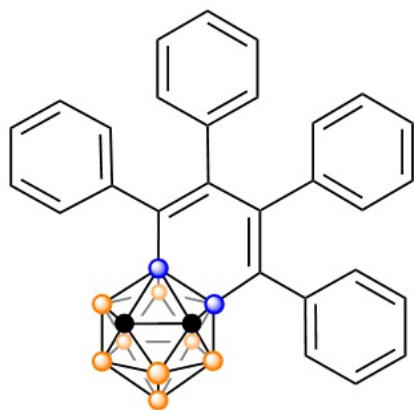
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Origin	Varian
Spectrometer	mercury
Solvent	CDCl3
Temperature	29.0
Experiment	1D
Probe	SW_BB
Number of Scans	8
Receiver Gain	28
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.0000
Spectrometer Frequency	300.03
Spectral Width	5494.5
Lowest Frequency	-708.7
Nucleus	1H
Acquired Size	10989
Spectral Size	32768



5a

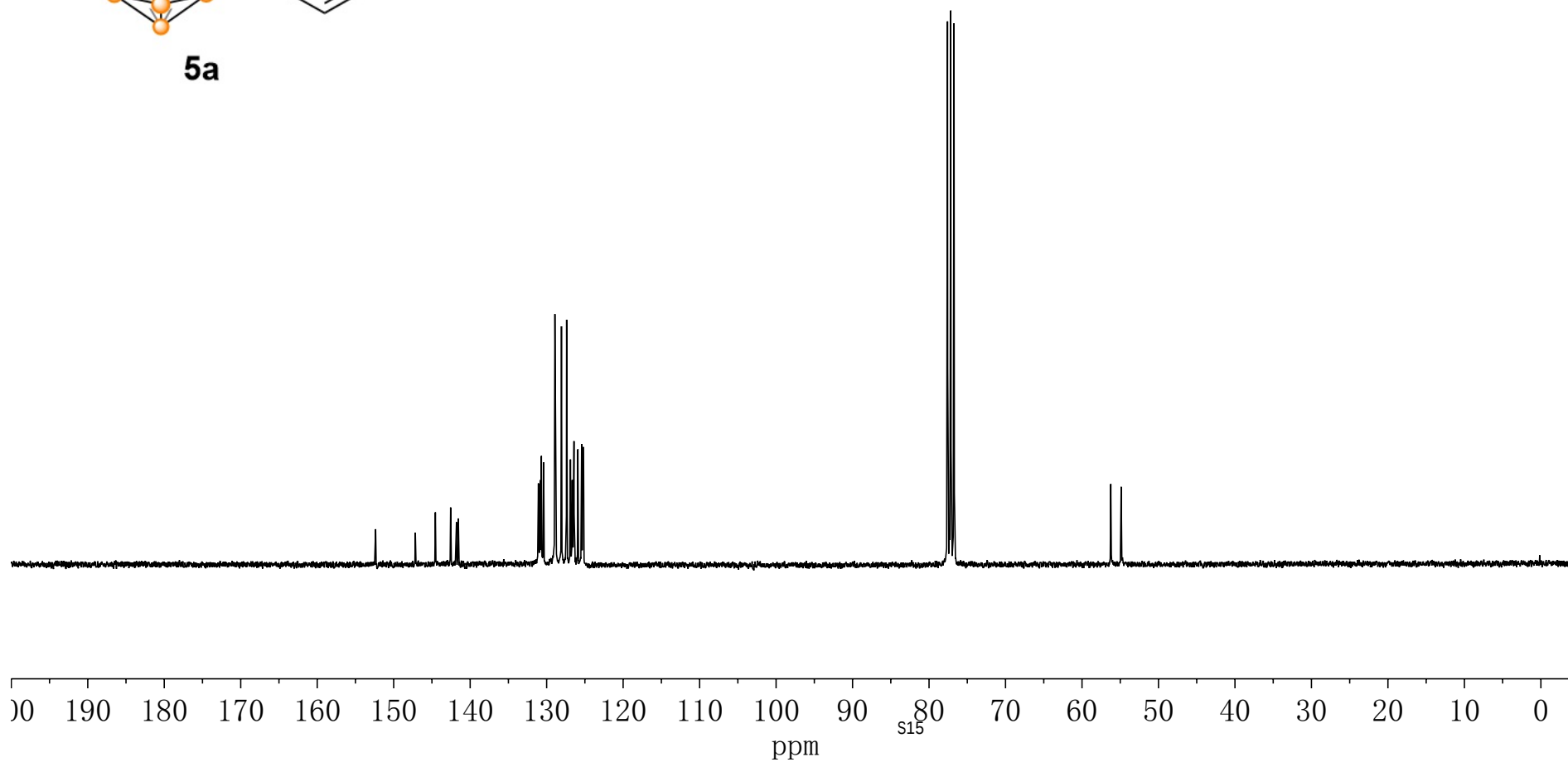


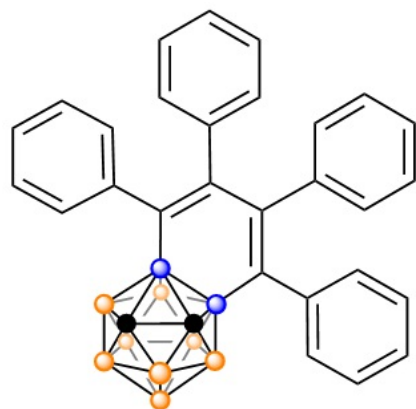


5a

152.36
147.17
144.55
142.56
141.79
141.55
131.07
130.83
130.69
130.39
128.92
128.85
128.06
127.39
126.89
126.65
126.46
126.43
125.92
125.42
125.28
125.18
77.58
77.16
76.74
56.24
54.88

Origin	Varian
Spectrometer	mercury
Solvent	CDC13
Temperature	29.0
Experiment	1D
Probe	SW_BB
Number of Scans	11332
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.5000
Spectrometer Frequency	75.45
Spectral Width	19011.4
Lowest Frequency	-1758.8
Nucleus	¹³ C
Acquired Size	28517
Spectral Size	65536

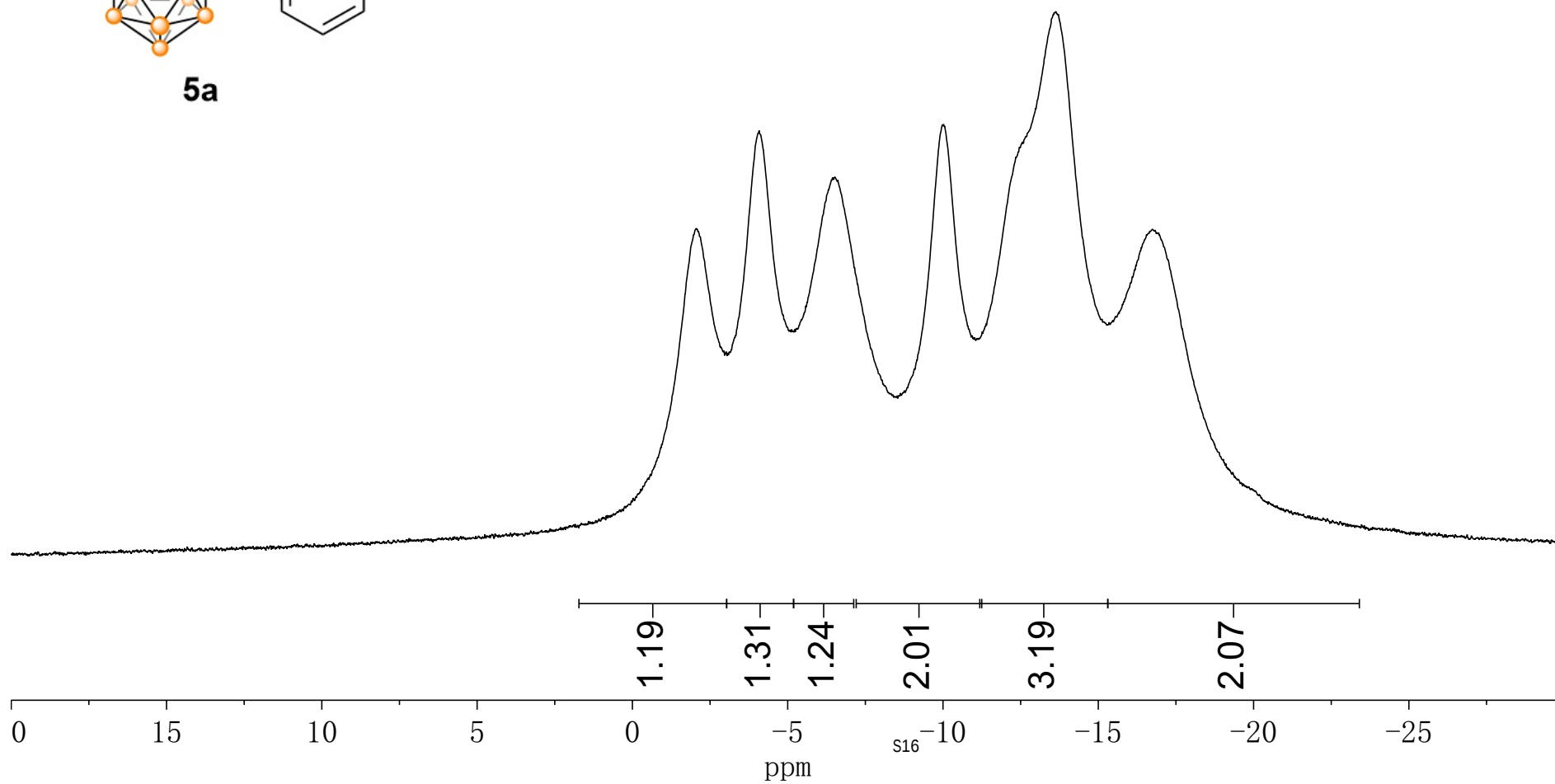


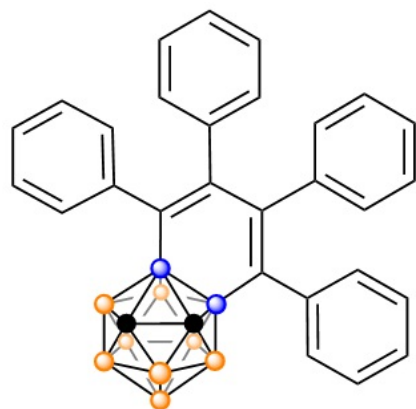


5a

--2.05
--4.08
--6.52
--10.02
--13.63
--16.75

Solvent	CDC13
Number of Scans	256
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	8.5000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16415.3
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

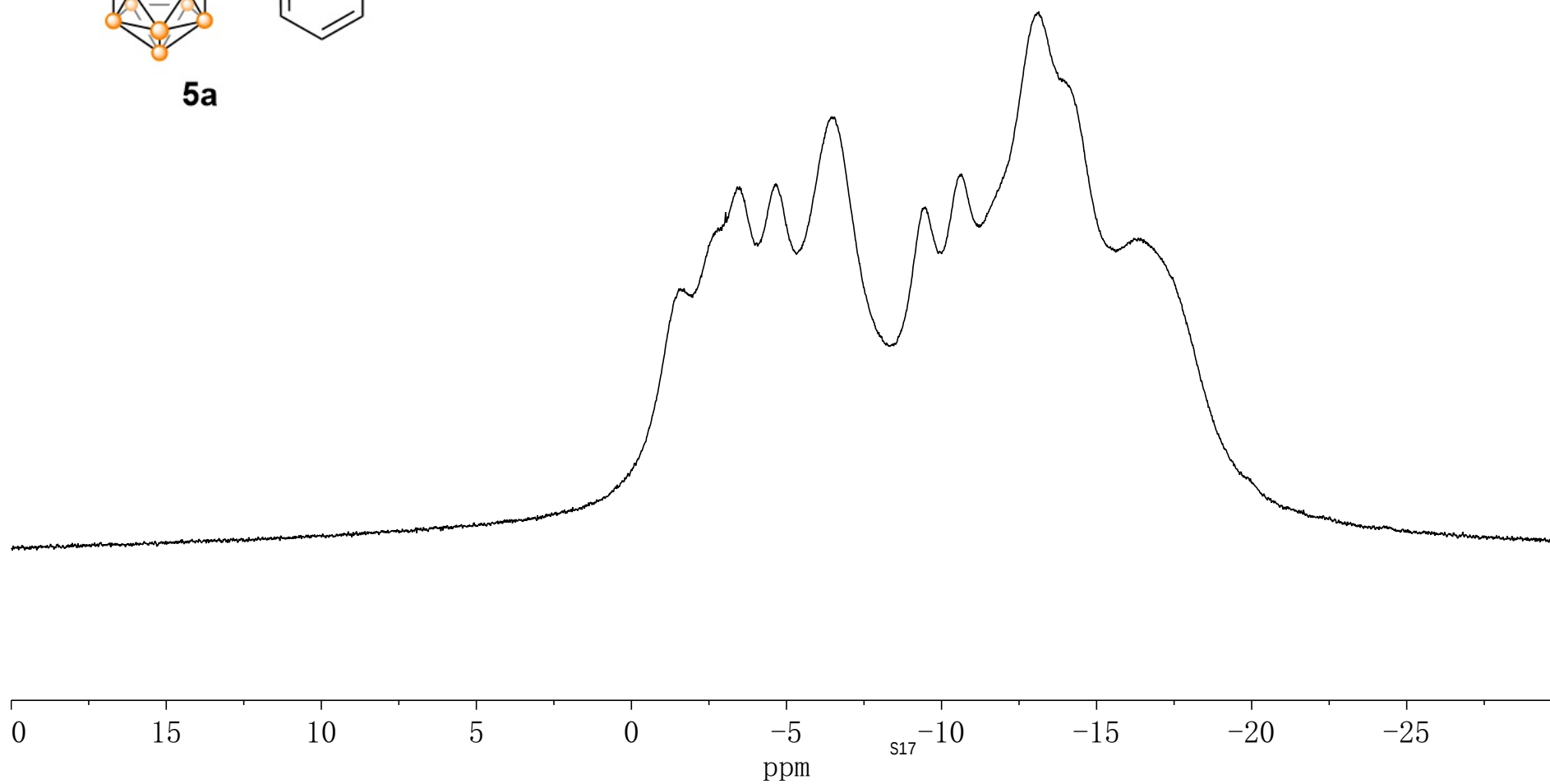




5a

1.48
 3.48
 4.66
 6.47
 9.47
 10.64
 13.13
 16.43

Solvent	CDC13
Number of Scans	256
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	8.5000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16415.3
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



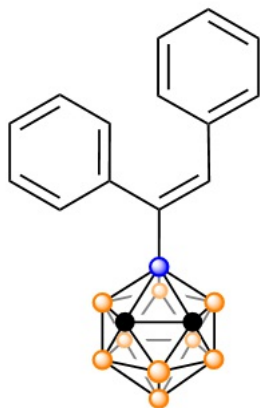
7.413
7.389
7.365
7.332
7.309
7.284
7.260
7.242
7.131
7.124
7.112
7.069
7.045
6.958
6.945
6.933

—3.243

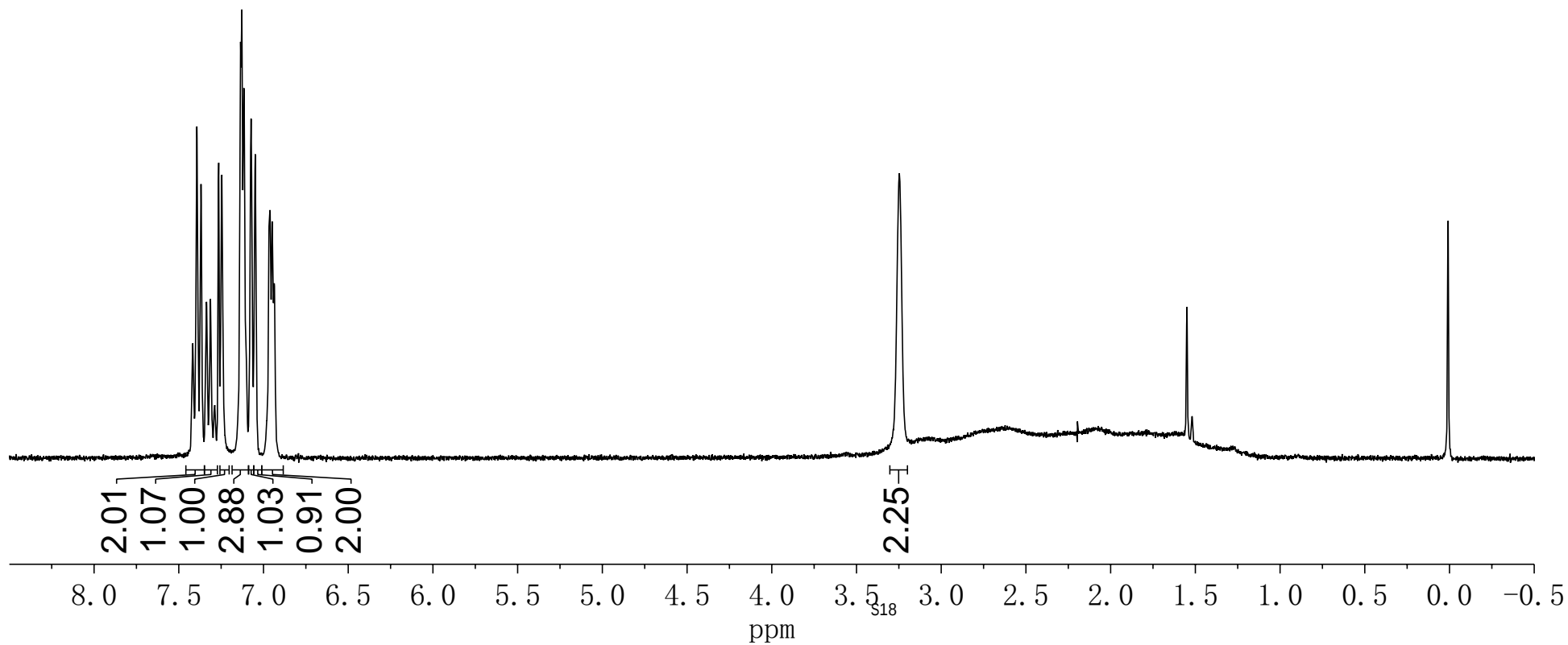
—1.550

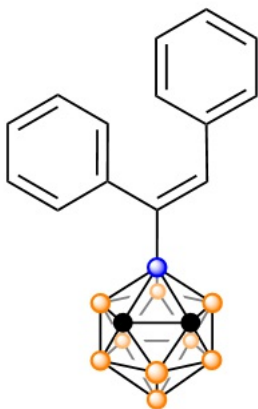
—0.004

Origin	Varian
Spectrometer	mercury
Solvent	CDCl ₃
Temperature	29.0
Experiment	1D
Probe	SW_BB
Number of Scans	4
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.0000
Spectrometer Frequency	300.03
Spectral Width	5494.5
Lowest Frequency	-709.3
Nucleus	¹ H
Acquired Size	10989
Spectral Size	32768



4a





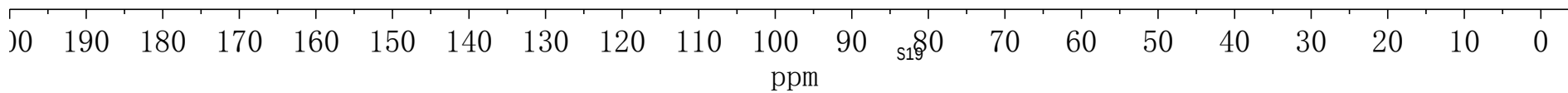
4a

141.90
141.53
136.80
129.83
129.69
128.21
127.87
127.48
127.29

77.48
77.16
76.84

—55.50

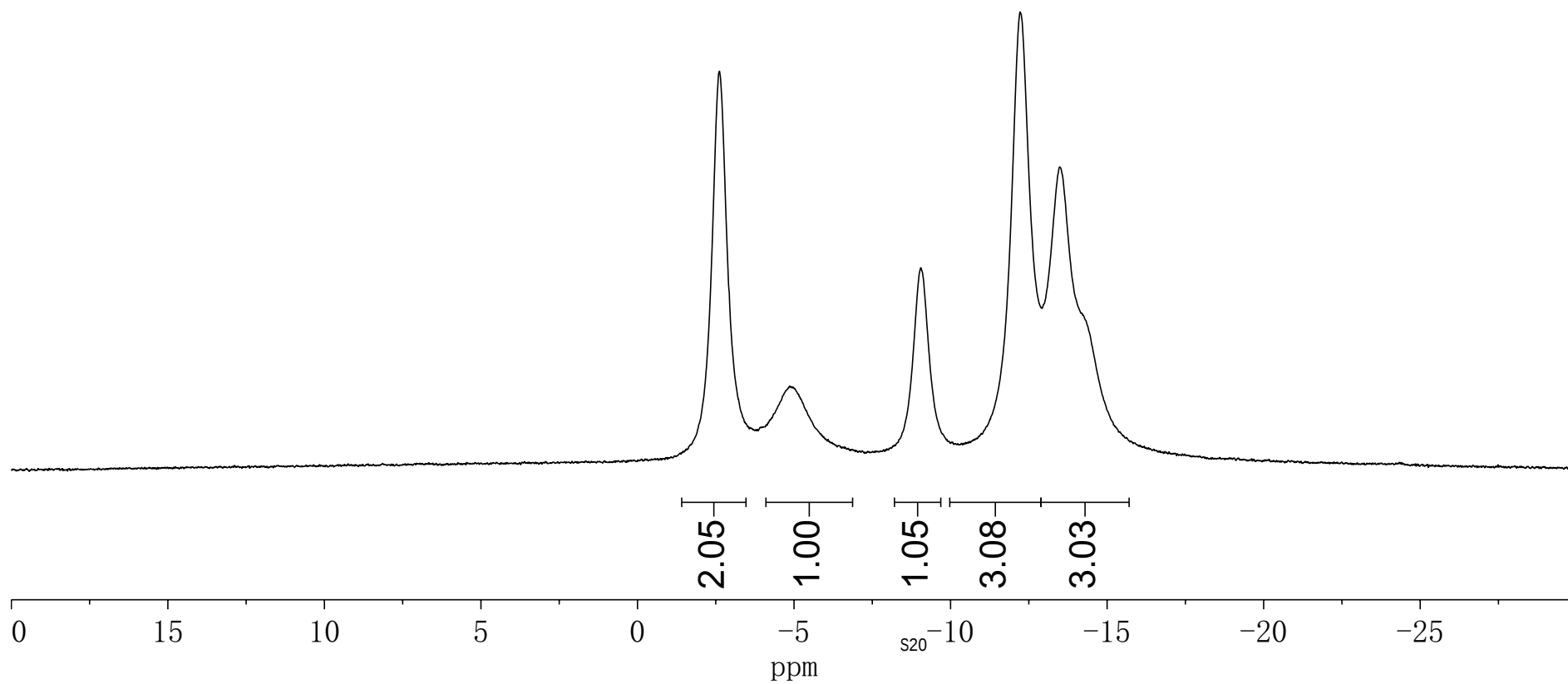
Solvent	CDCl ₃
Number of Scans	1500
Receiver Gain	16384
Relaxation Delay	3.0000
Pulse Width	4.0000
Spectrometer Frequency	100.61
Spectral Width	35211.3
Lowest Frequency	-6906.8
Nucleus	¹³ C
Acquired Size	16384
Spectral Size	65536

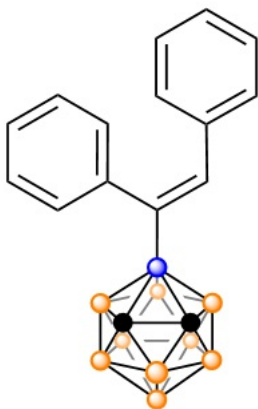




—13.49

Solvent	CDC13
Number of Scans	256
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	8.5000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16401.2
Nucleus	¹ H
Acquired Size	16384
Spectral Size	32768

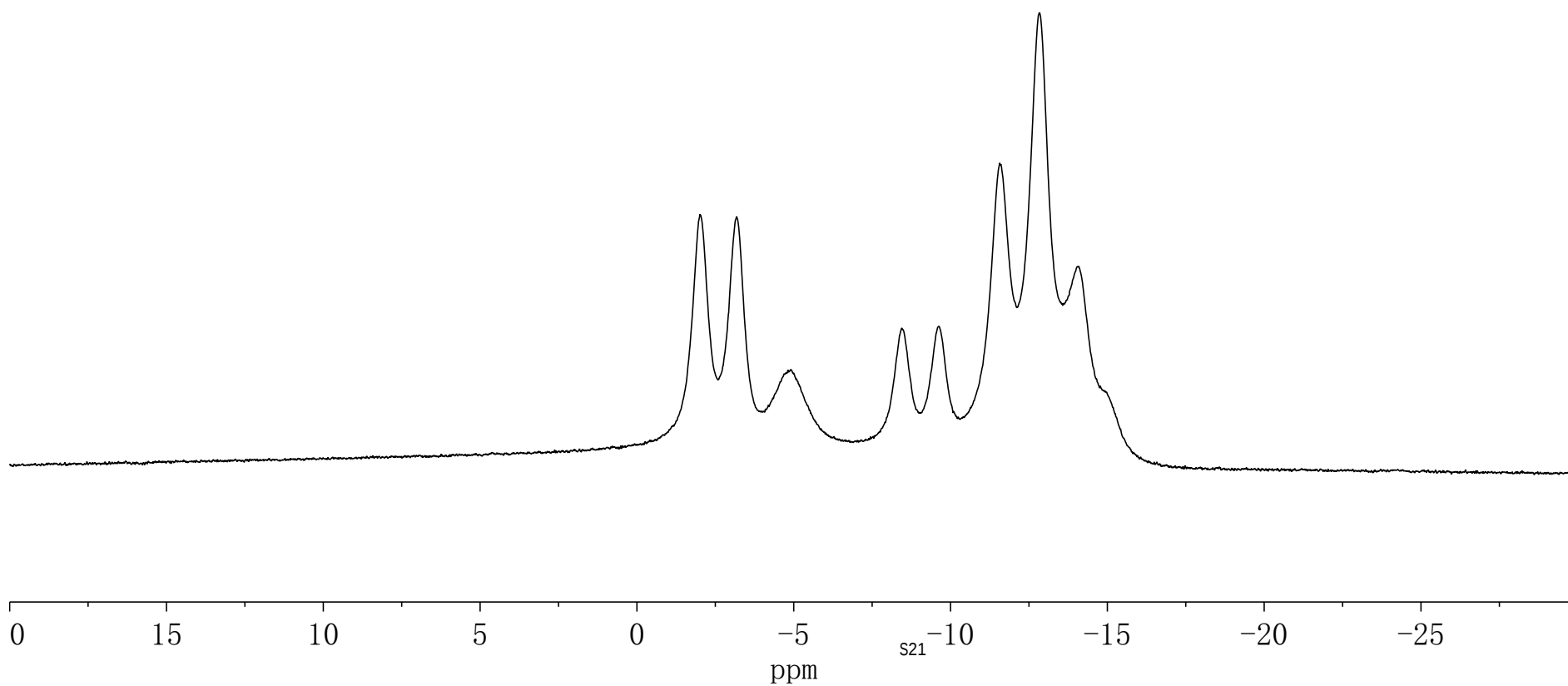


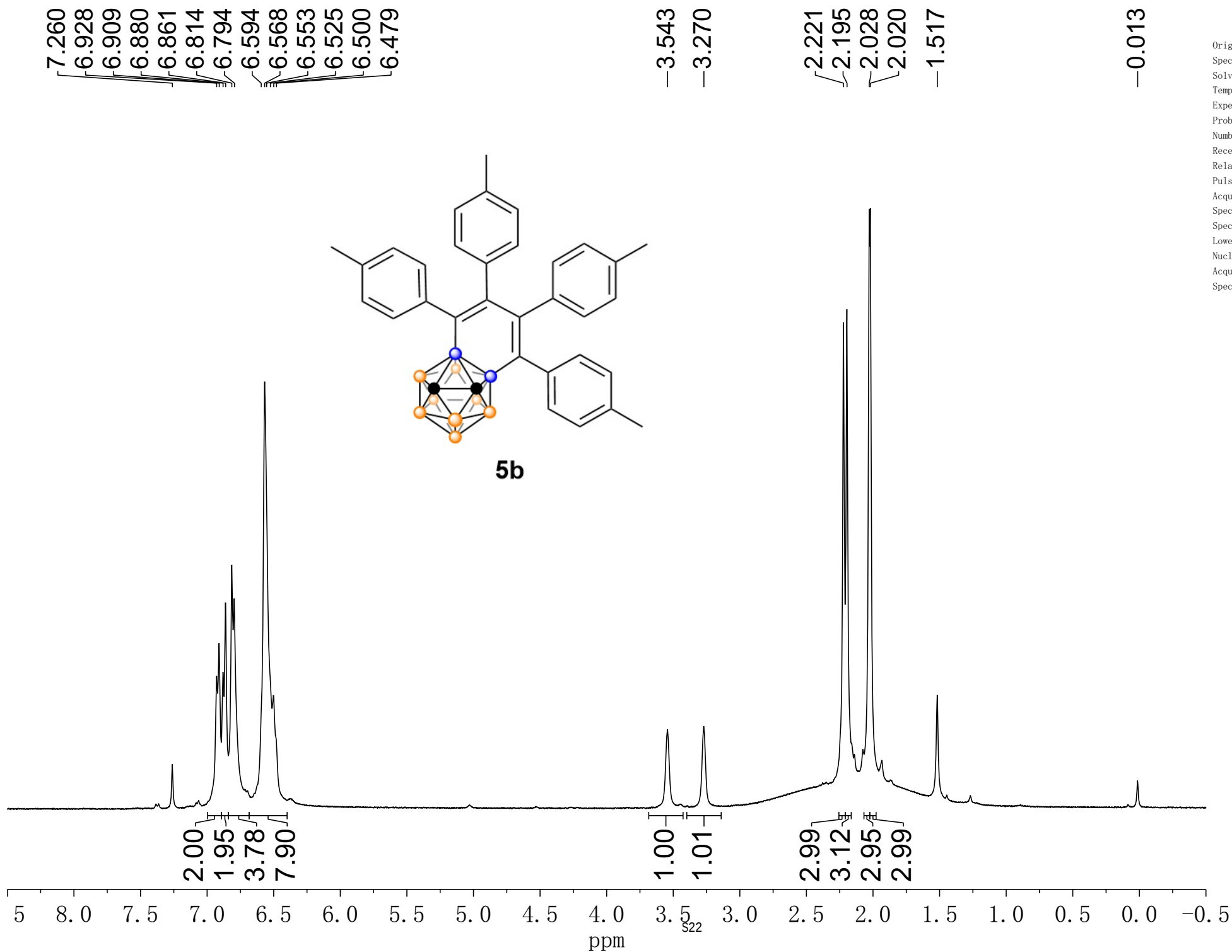


4a

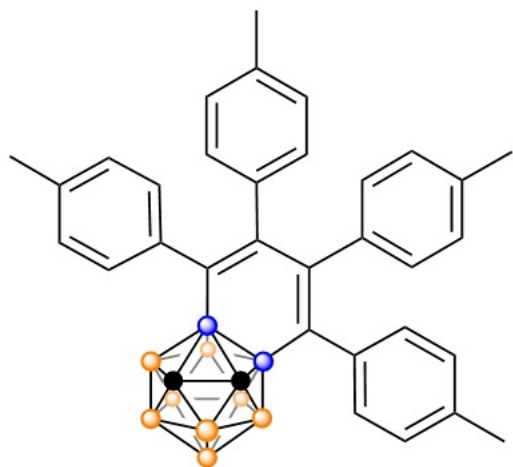
~2.01
~3.18
~4.91
~8.46
~9.63
~11.57
~12.83
~14.09

Solvent	CDC13
Number of Scans	256
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	8.5000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16401.2
Nucleus	11B
Acquired Size	16384
Spectral Size	32768





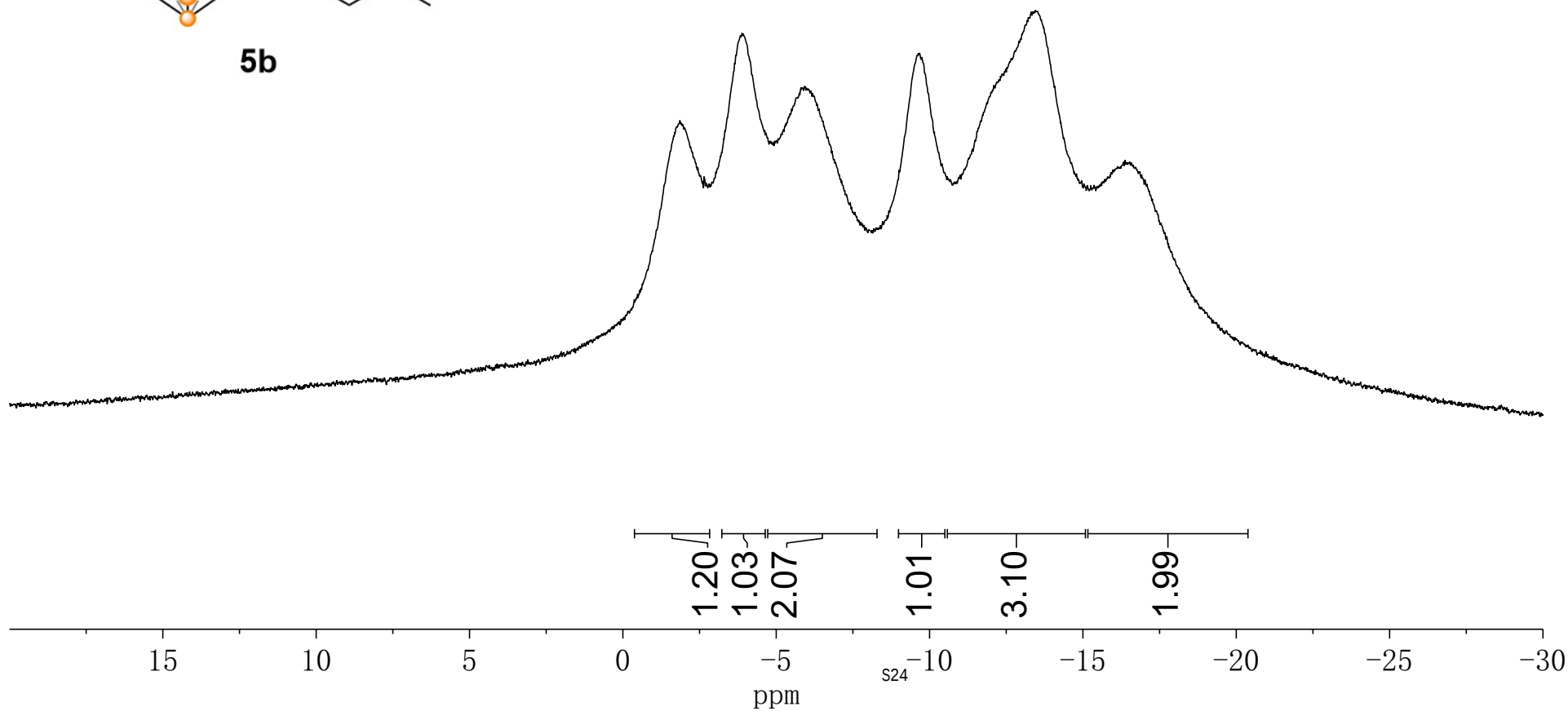
Origin	Varian
Spectrometer	vnmr5
Solvent	CDCl ₃
Temperature	21.0
Experiment	1D
Probe	4nuc
Number of Scans	8
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	399.72
Spectral Width	7183.9
Lowest Frequency	-793.1
Nucleus	¹ H
Acquired Size	21552
Spectral Size	65536

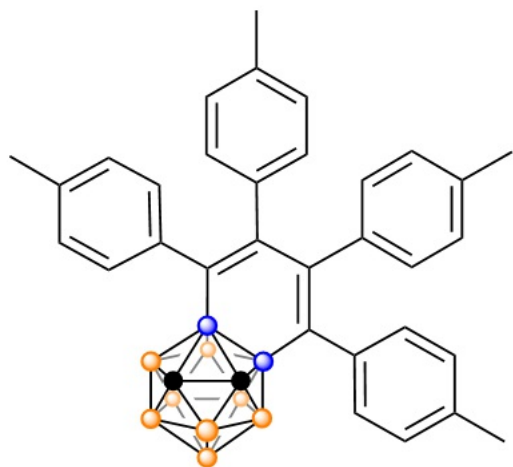


5b

δ 1.82
 δ 3.86
 δ 6.01
 δ 9.63
 δ 13.34
 δ 16.67

Solvent	CDC13
Number of Scans	1024
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16365.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

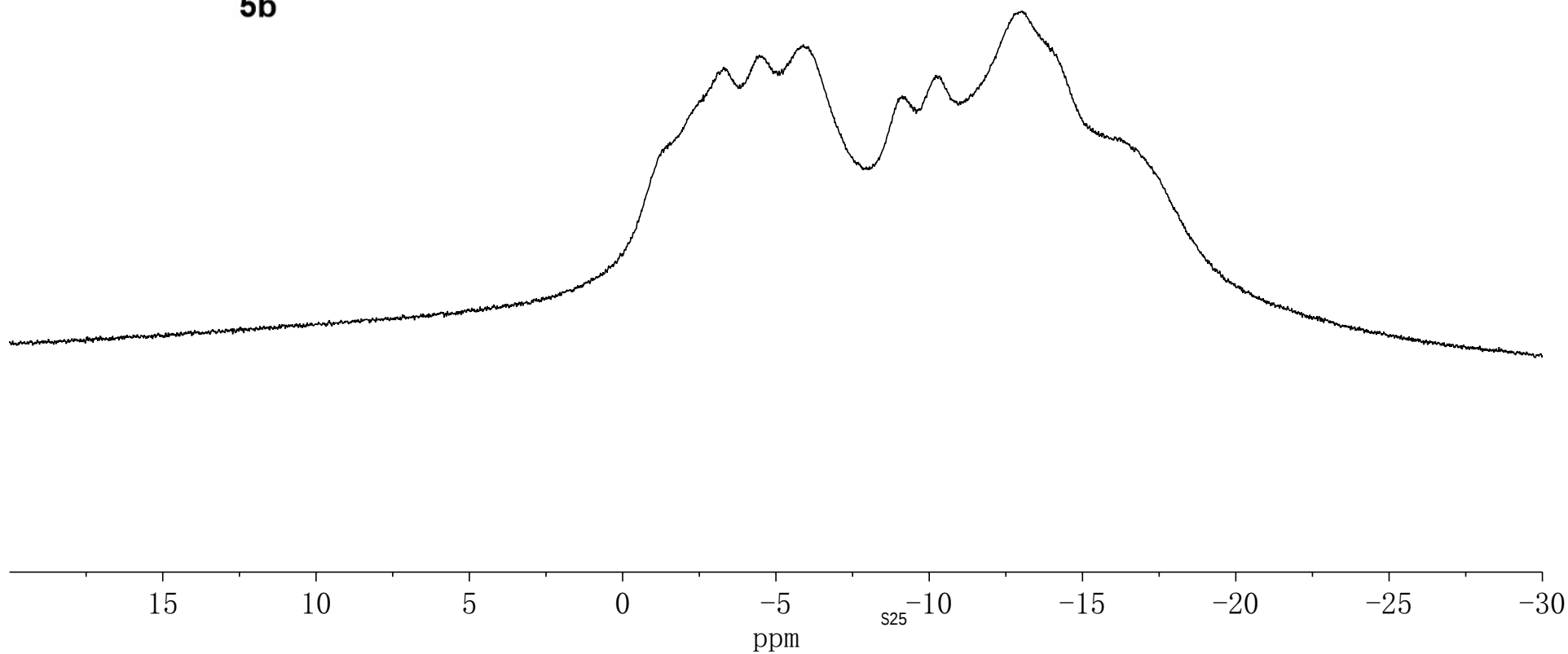




5b

~-1.32
 ~-3.03
 ~-4.47
 ~-6.06
 --9.05
 --10.19
 ~-12.80
 ~-12.98
 --16.82

Solvent	CDC13
Number of Scans	1169
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16365.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

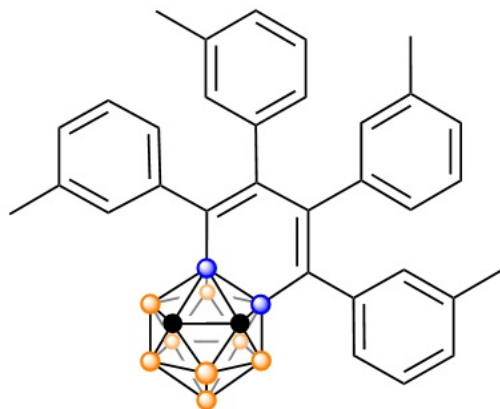


7.260
7.004
6.985
6.962
6.938
6.919
6.839
6.820
6.779
6.760
6.697
6.664
6.643
6.623
6.604
6.537
6.520
6.497
6.482
6.434
6.386

3.615
3.567
3.269

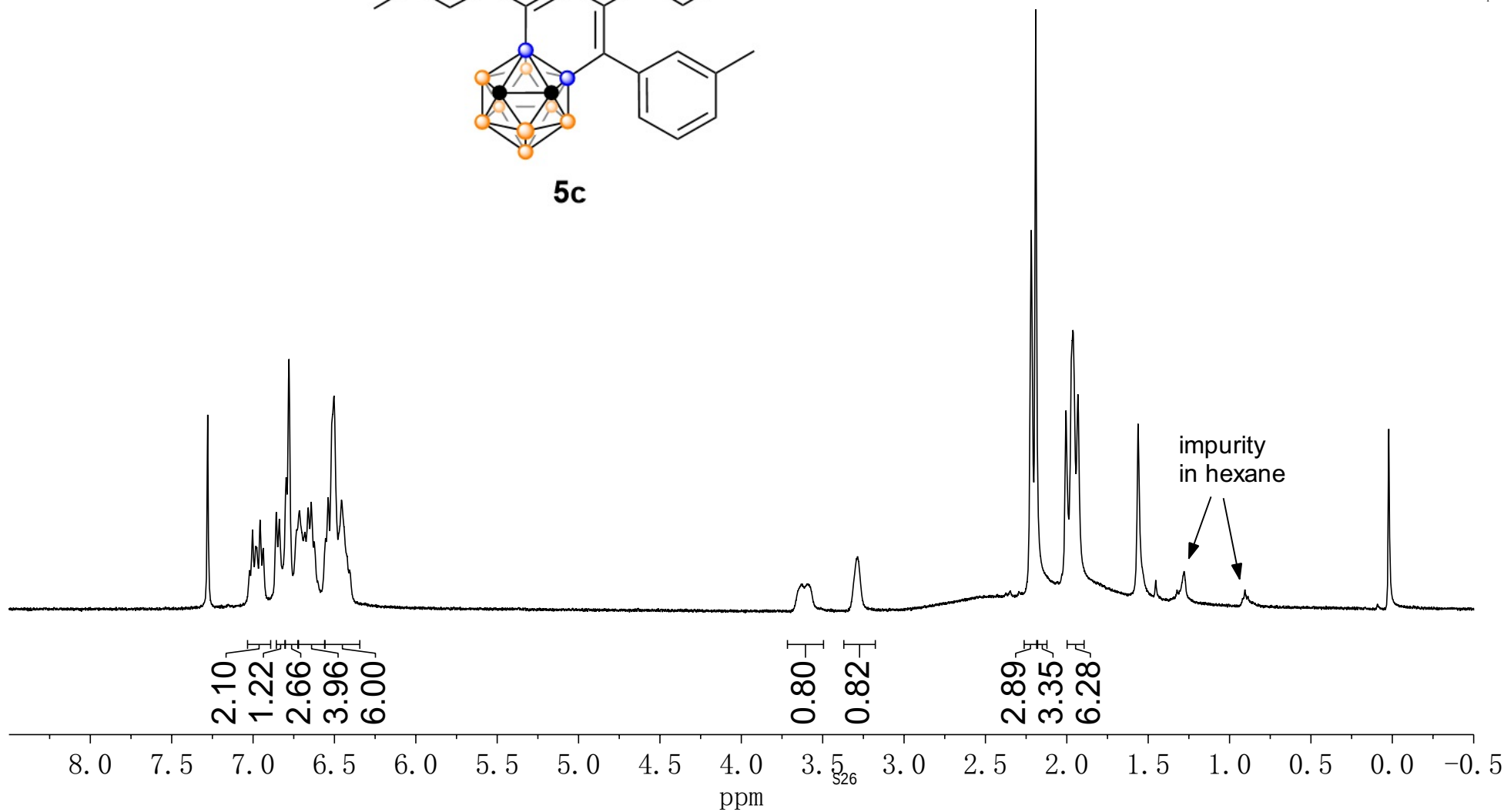
2.200
2.172
1.986
1.940
1.912
1.542

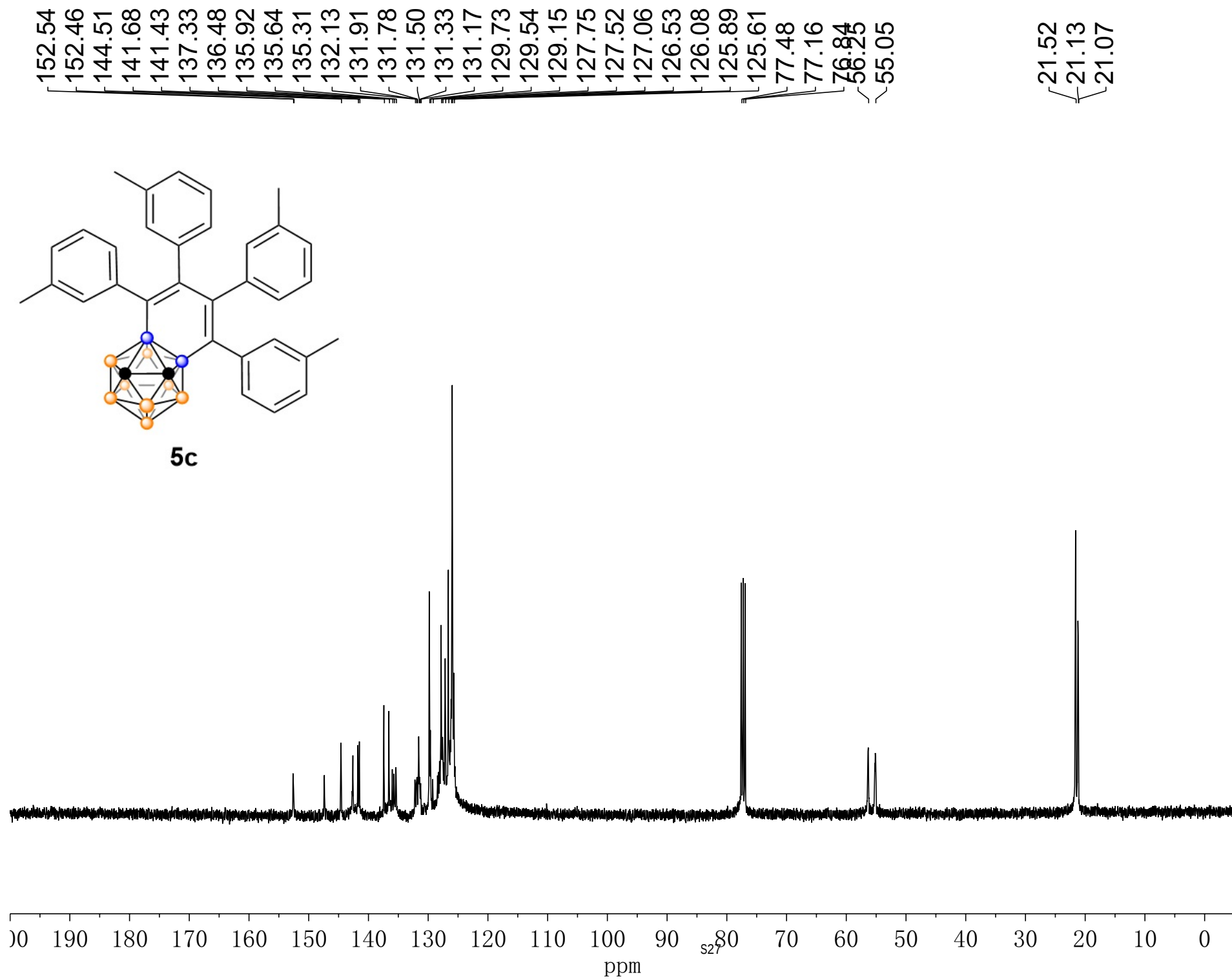
0.003



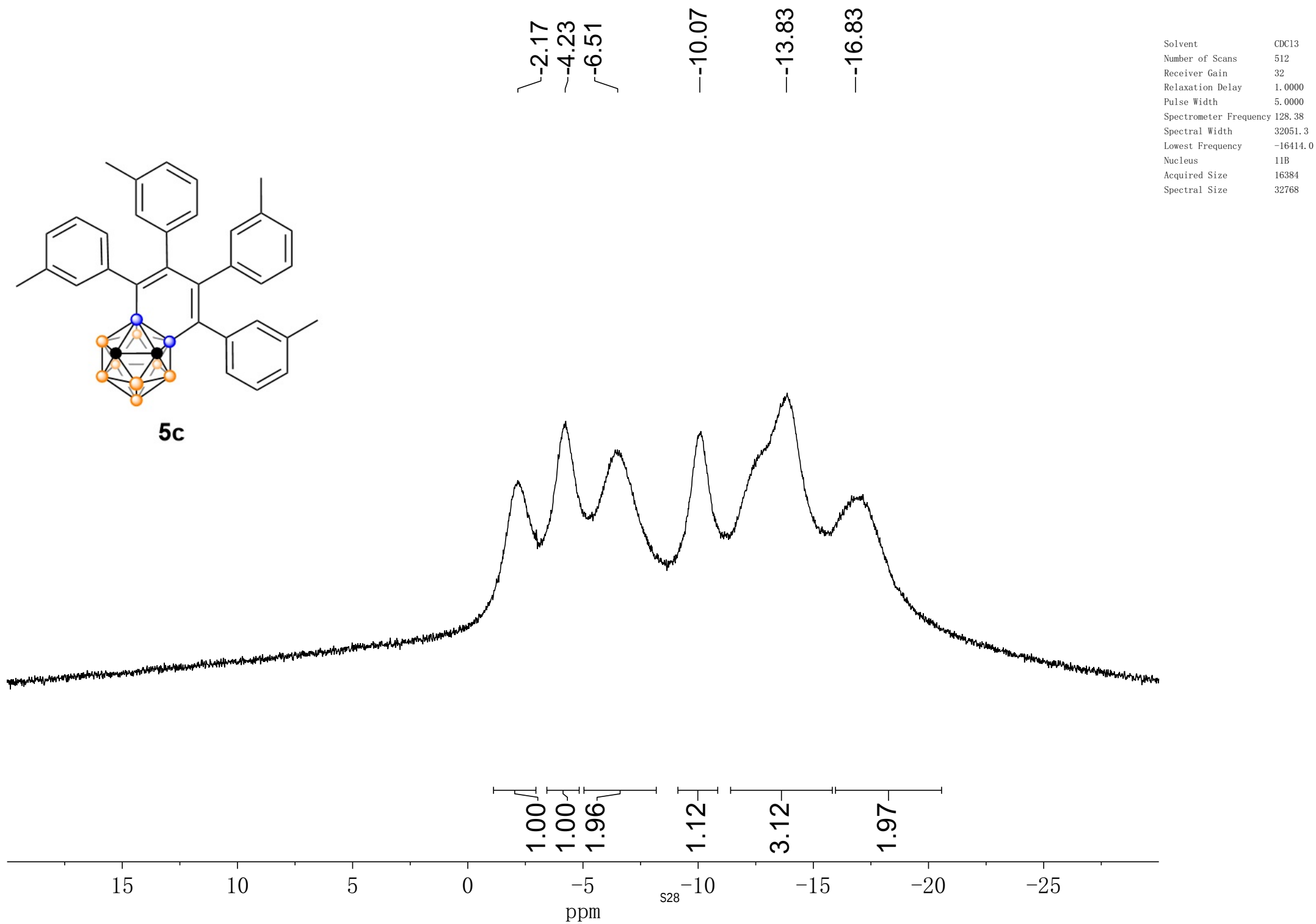
5c

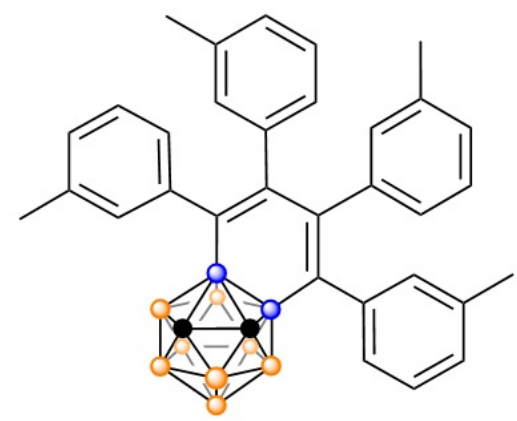
Origin	Varian
Spectrometer	vnmr5
Solvent	CDC13
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	20
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	400.03
Spectral Width	7267.4
Lowest Frequency	-838.3
Nucleus	¹ H
Acquired Size	21802
Spectral Size	65536





Origin	Varian
Spectrometer	nmr
Solvent	cdcl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	1212
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3000
Spectrometer Frequency	100.60
Spectral Width	26595.7
Lowest Frequency	-2214.3
Nucleus	13C
Acquired Size	34575
Spectral Size	131072

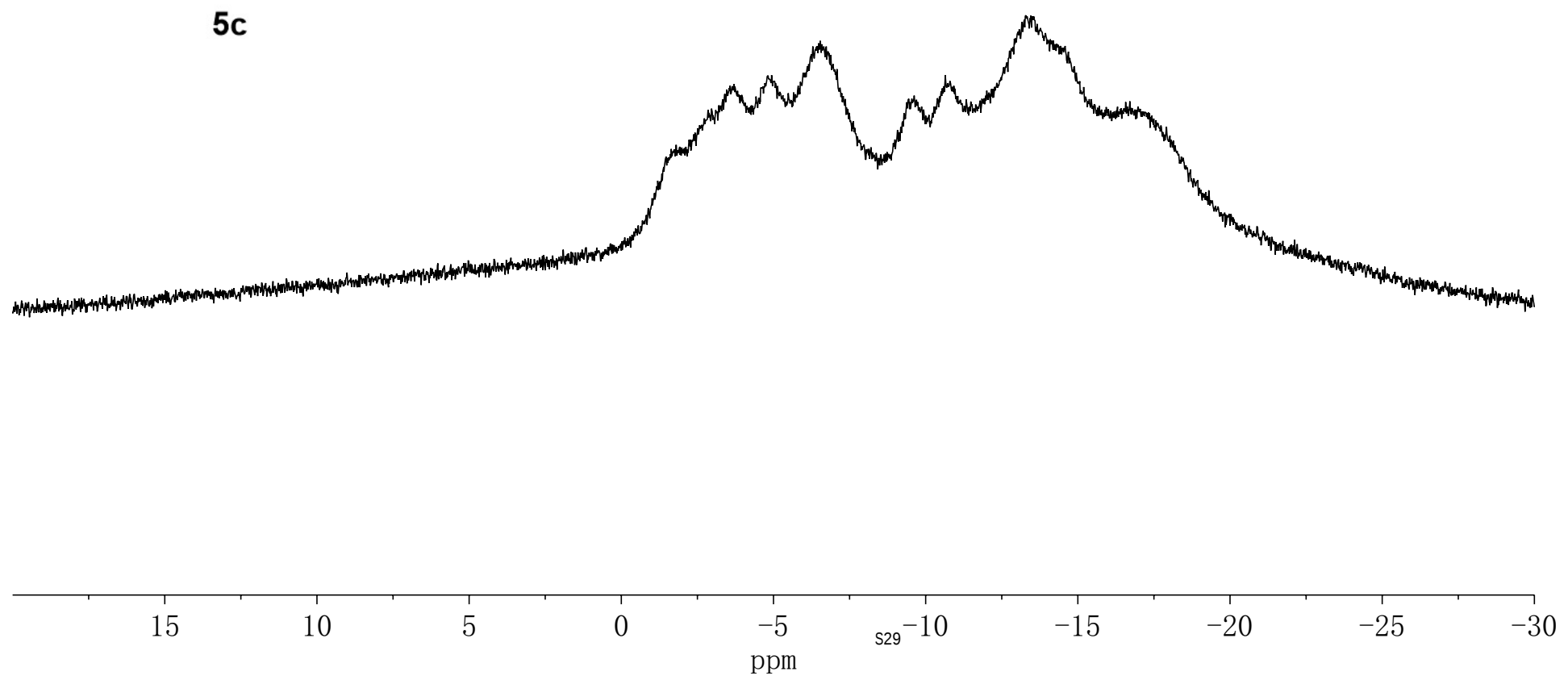


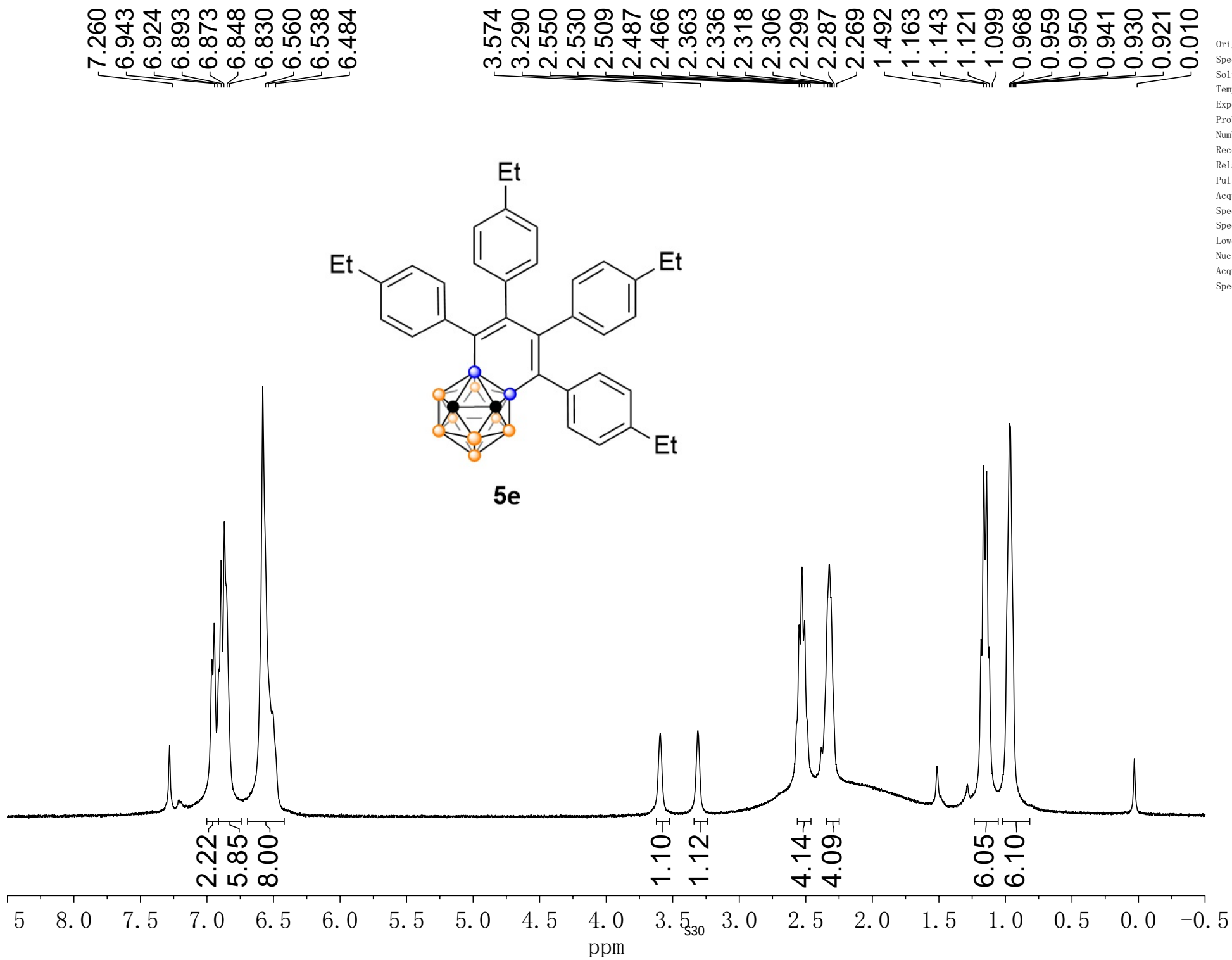


5c

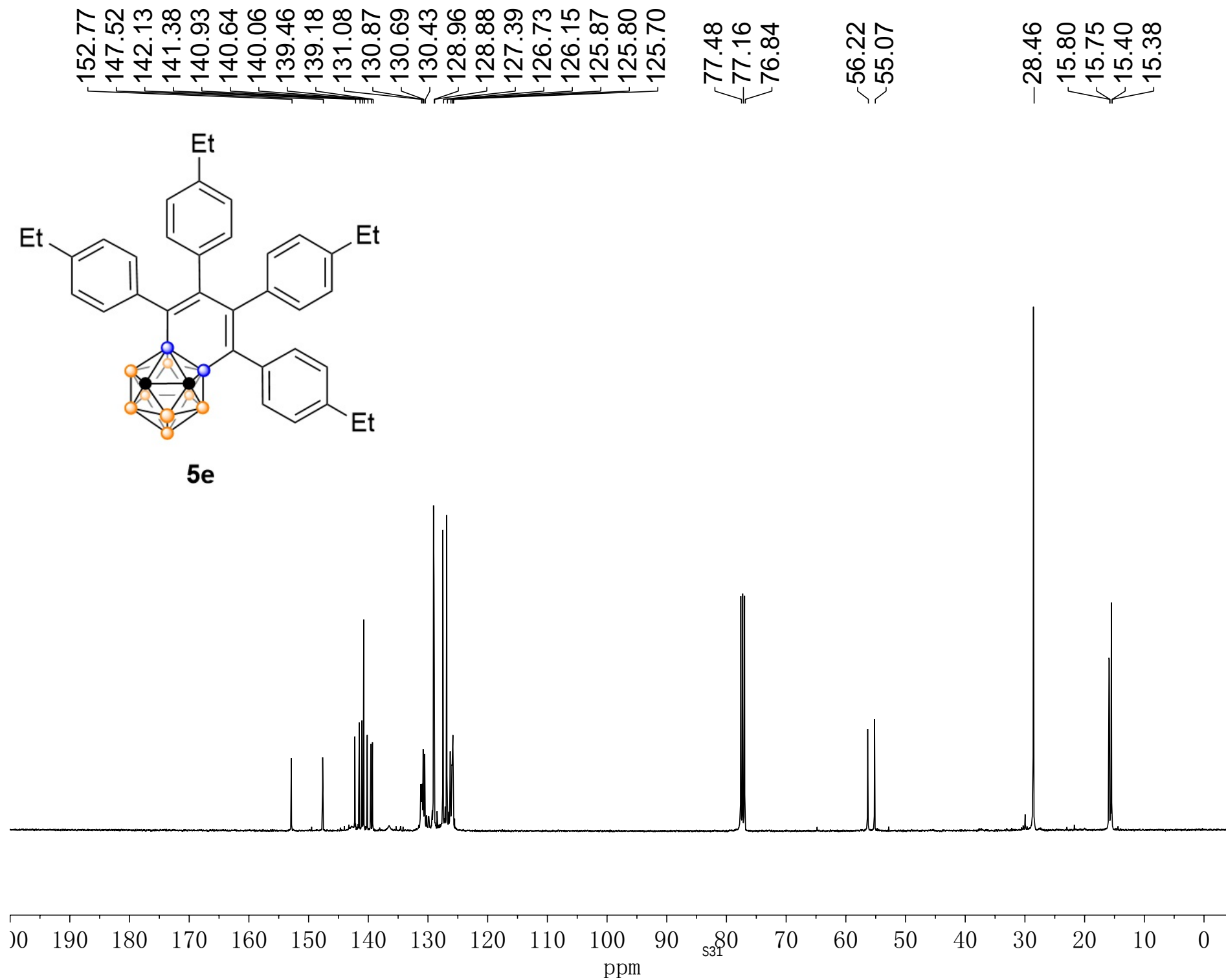
1.76
 3.53
 4.91
 6.70
 9.59
 10.73
 13.54
 16.93

Solvent	CDC13
Number of Scans	256
Receiver Gain	575
Relaxation Delay	1.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16419.8
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

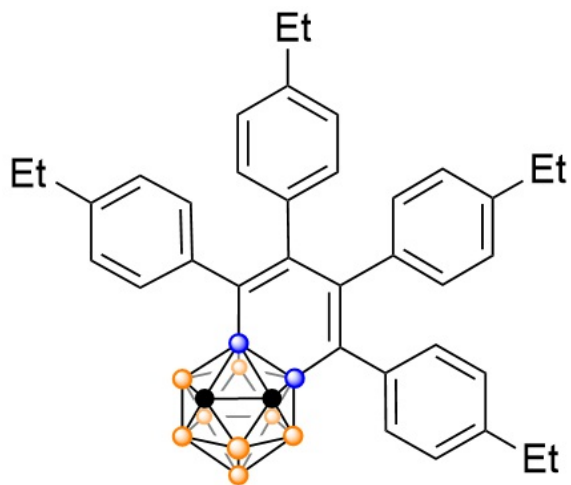




Origin	Varian
Spectrometer	nmrs
Solvent	CDCl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	8
Receiver Gain	28
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	400.03
Spectral Width	7225.4
Lowest Frequency	-795.7
Nucleus	1H
Acquired Size	21676
Spectral Size	65536



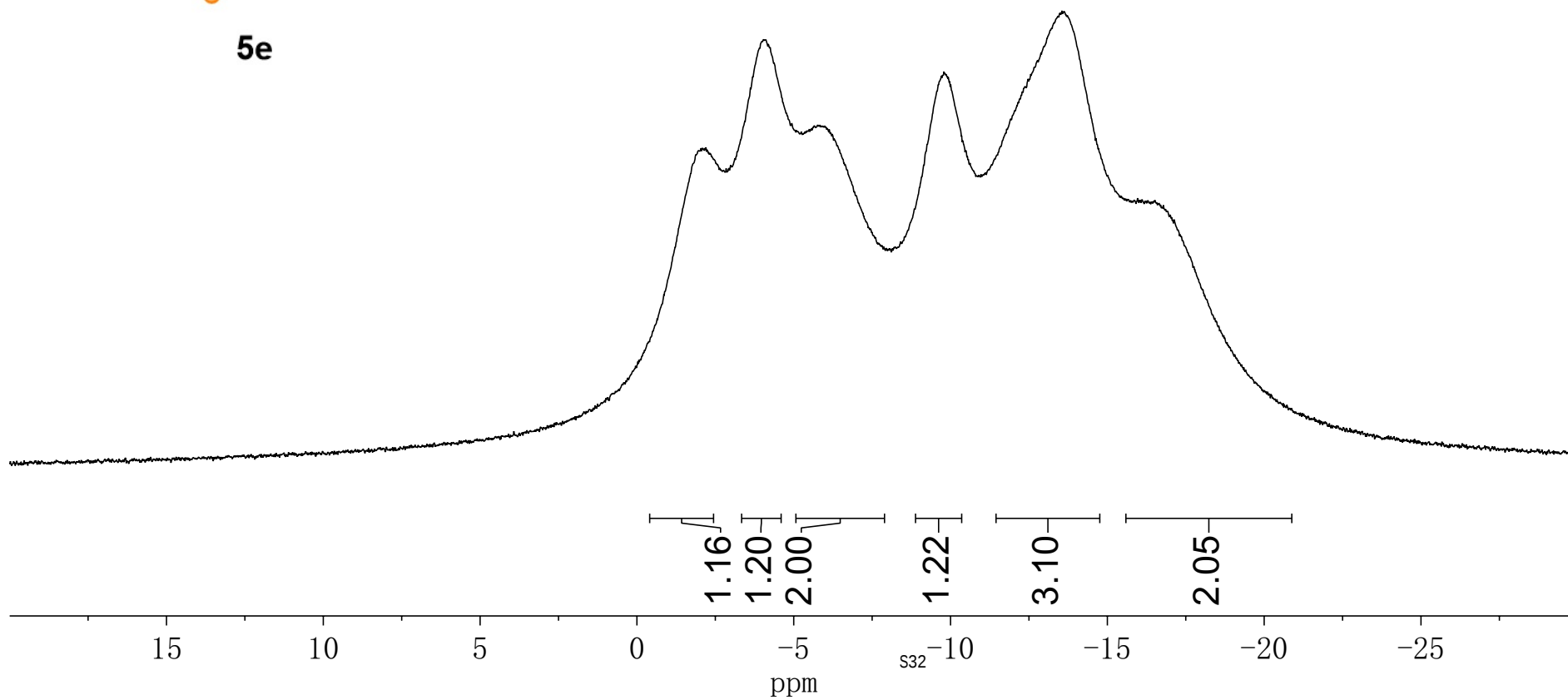
Origin	Varian
Spectrometer	nmr5
Solvent	cdcl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	13640
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3005
Spectrometer Frequency	100.60
Spectral Width	24509.8
Lowest Frequency	-1669.3
Nucleus	13C
Acquired Size	31875
Spectral Size	65536

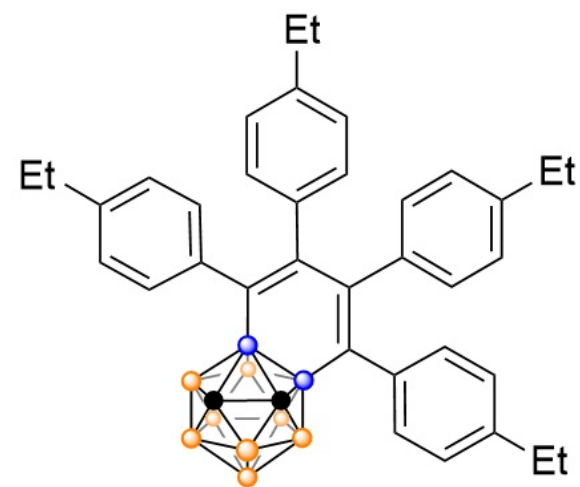


5e

δ -1.95
 δ -4.07
 δ -5.83
 δ -9.81
 δ -13.72
 δ -16.43

Solvent	CDC13
Number of Scans	256
Receiver Gain	228
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16391.5
Nucleus	^{11}B
Acquired Size	16384
Spectral Size	32768

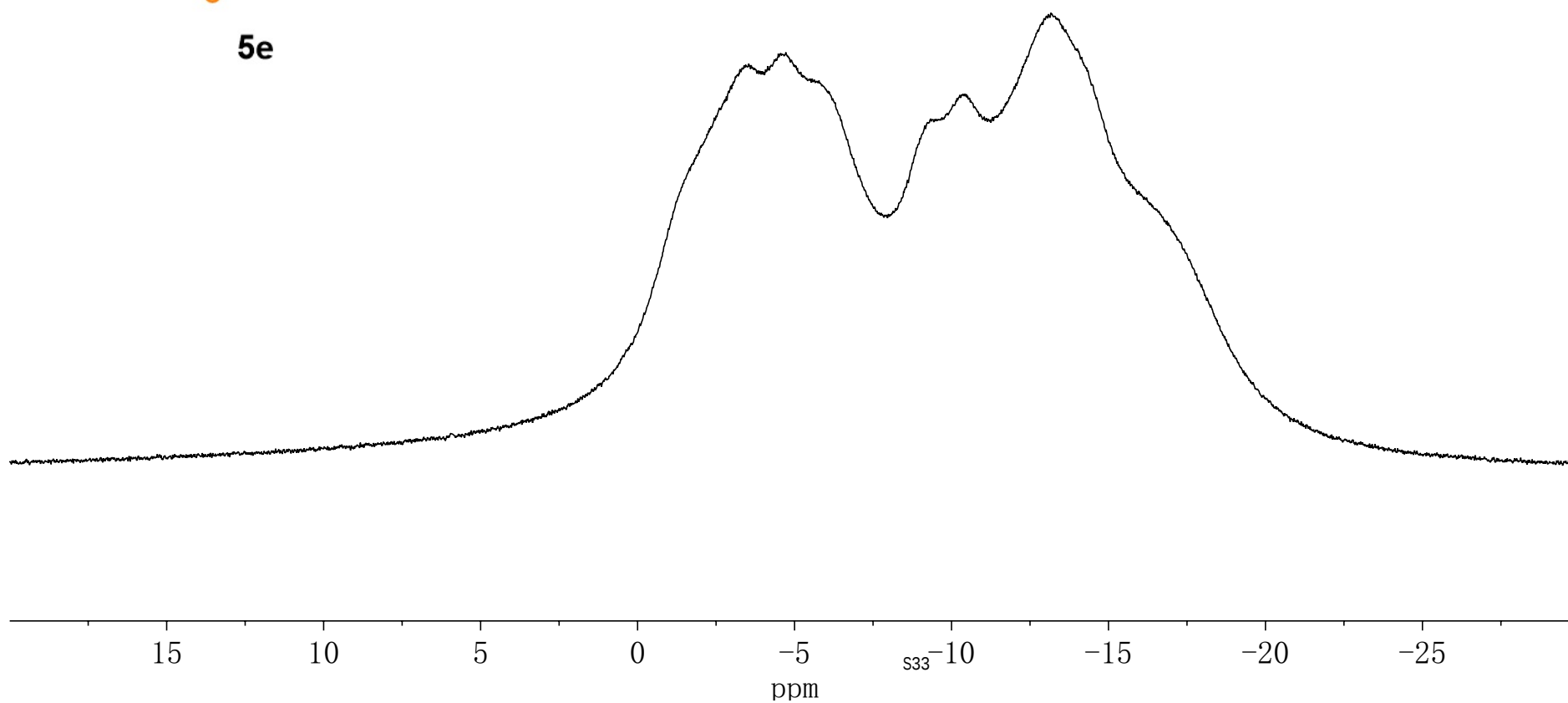


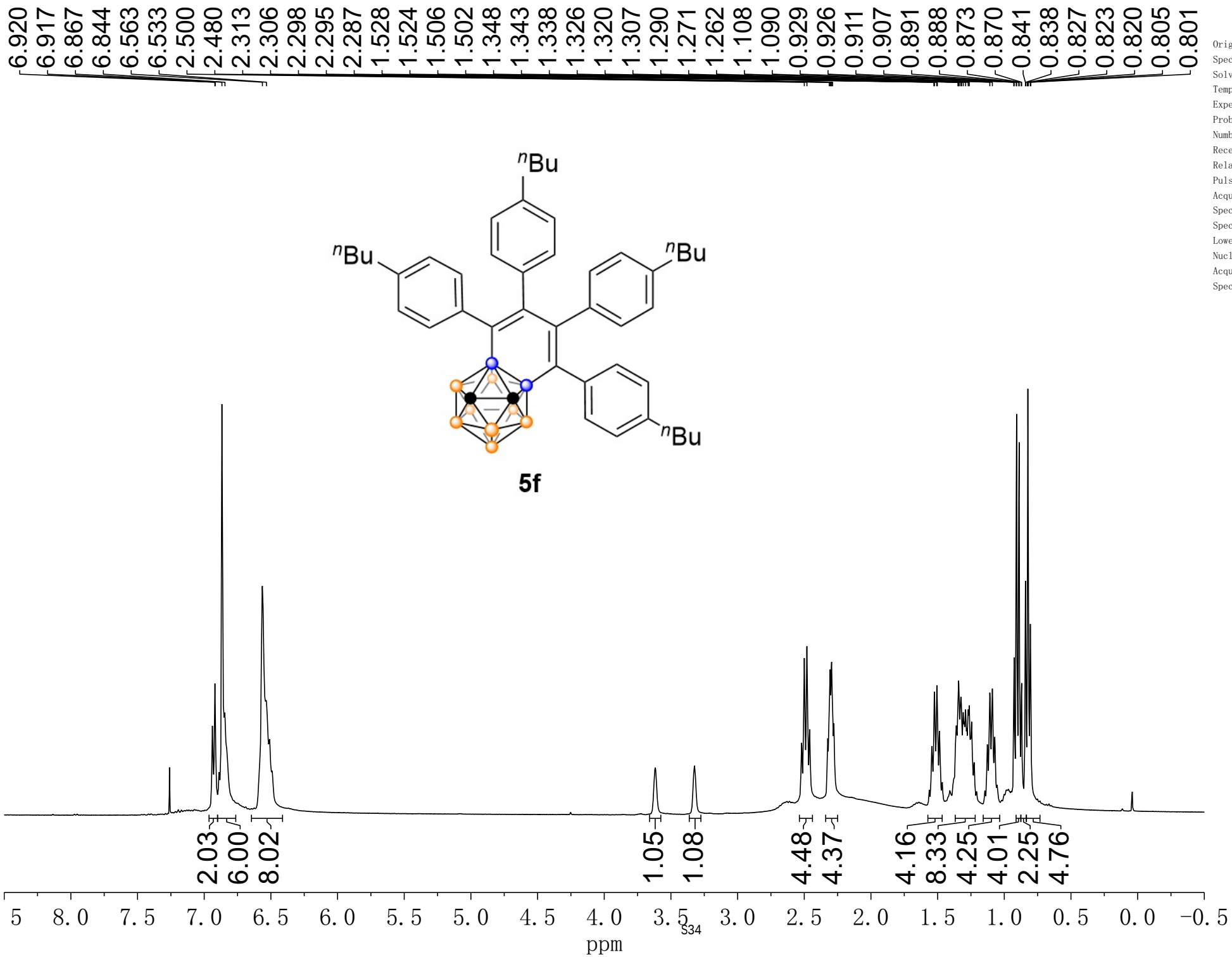


5e

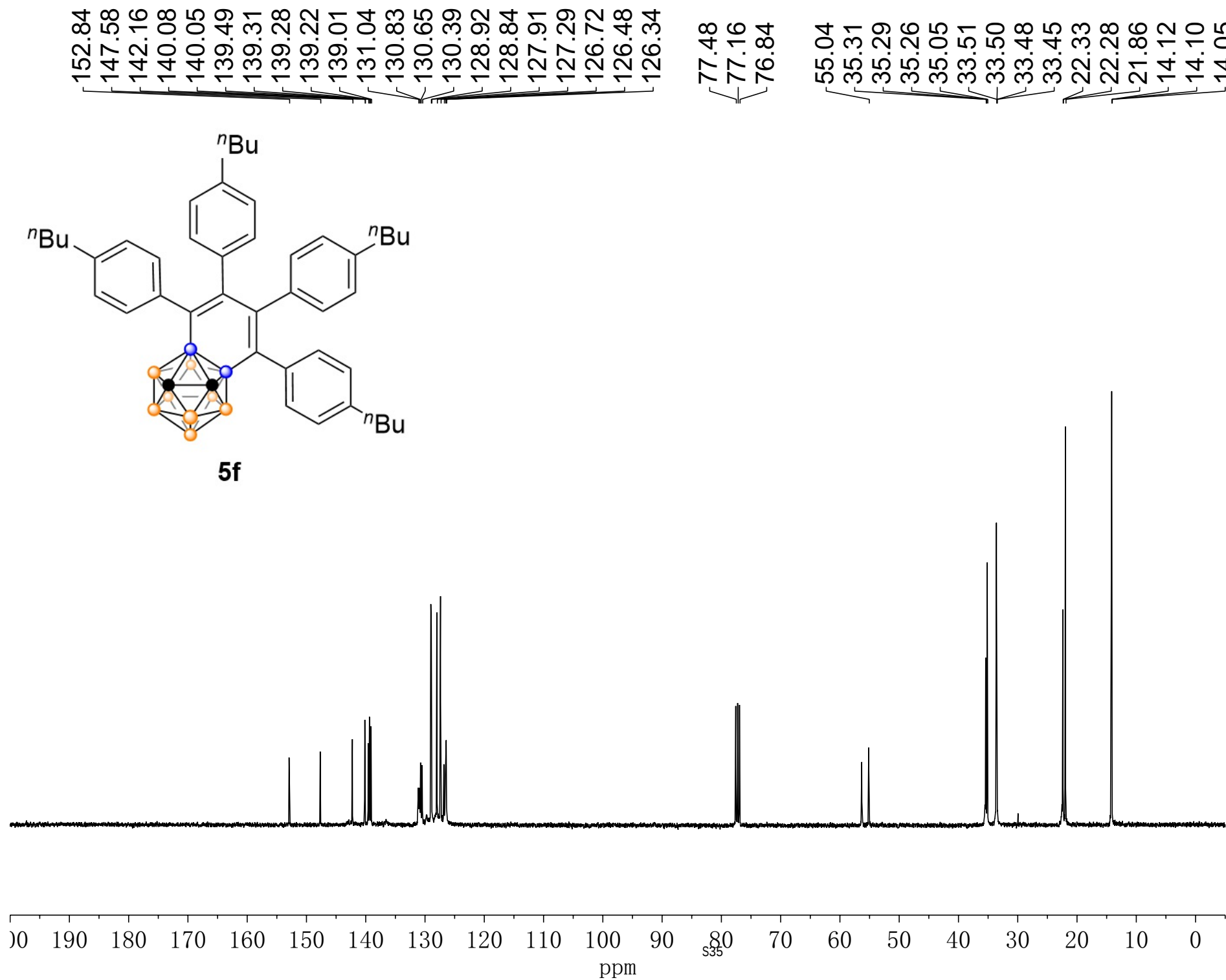
1.38
 3.42
 4.55
 5.78
 9.20
 10.28
 13.25
 13.26
 16.26

Solvent	CDC13
Number of Scans	256
Receiver Gain	203
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16391.5
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

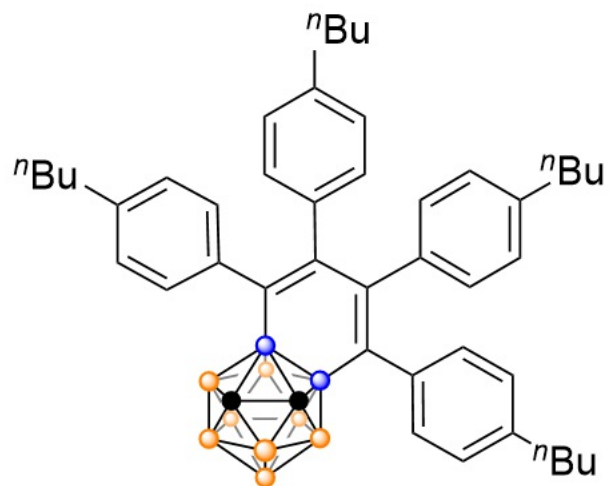




Origin	Varian
Spectrometer	vnmr5
Solvent	CDCl3
Temperature	25.0
Experiment	1D
Probe	4nuc
Number of Scans	8
Receiver Gain	26
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	399.72
Spectral Width	7183.9
Lowest Frequency	-784.6
Nucleus	1H
Acquired Size	21552
Spectral Size	65536



Origin	Varian
Spectrometer	nmrs
Solvent	cdcl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	424
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3005
Spectrometer Frequency	100.60
Spectral Width	24509.8
Lowest Frequency	-1673.6
Nucleus	13C
Acquired Size	31875
Spectral Size	65536



5f

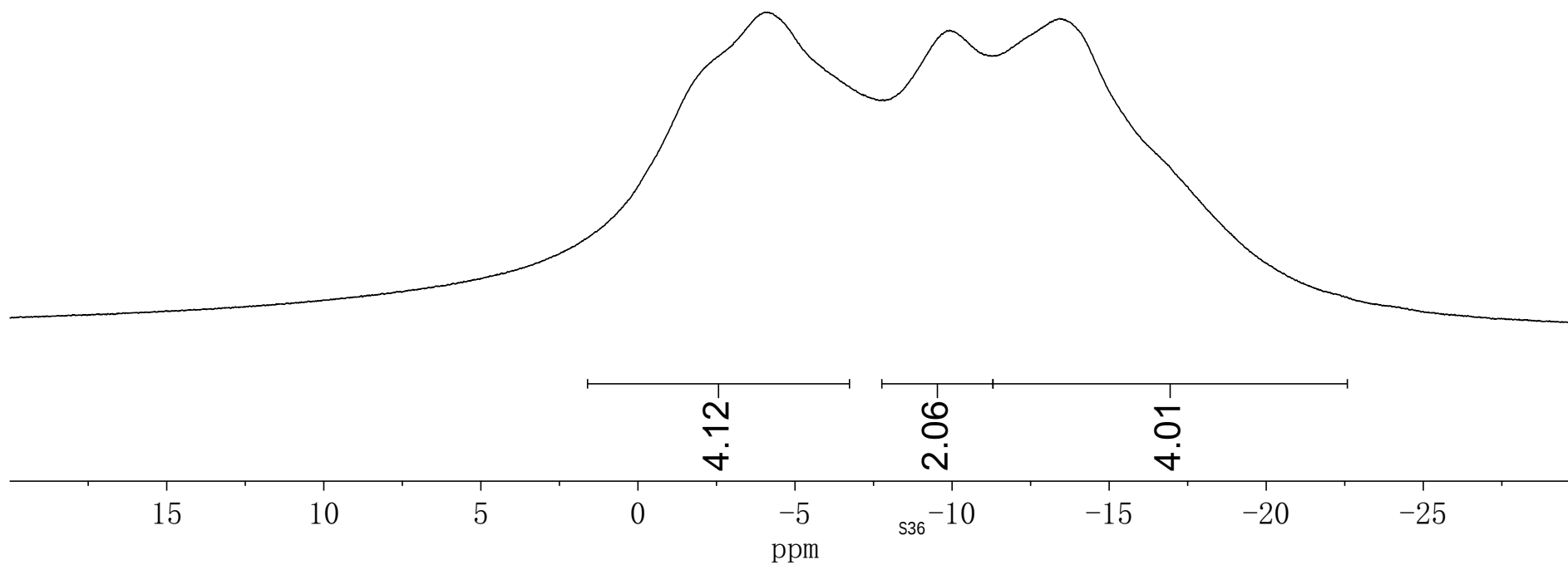
--1.77

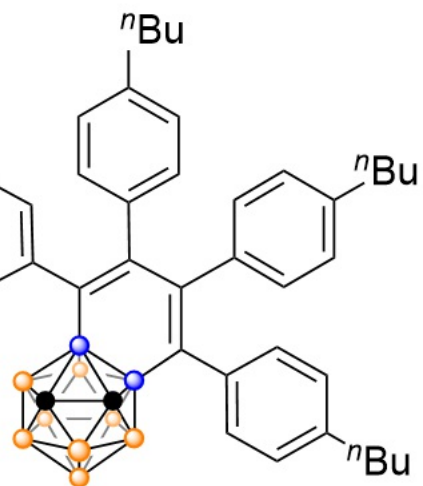
--4.05

--9.84

--13.44

Solvent	CDC13
Number of Scans	1024
Receiver Gain	128
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16416.9
Nucleus	11B
Acquired Size	16384
Spectral Size	32768





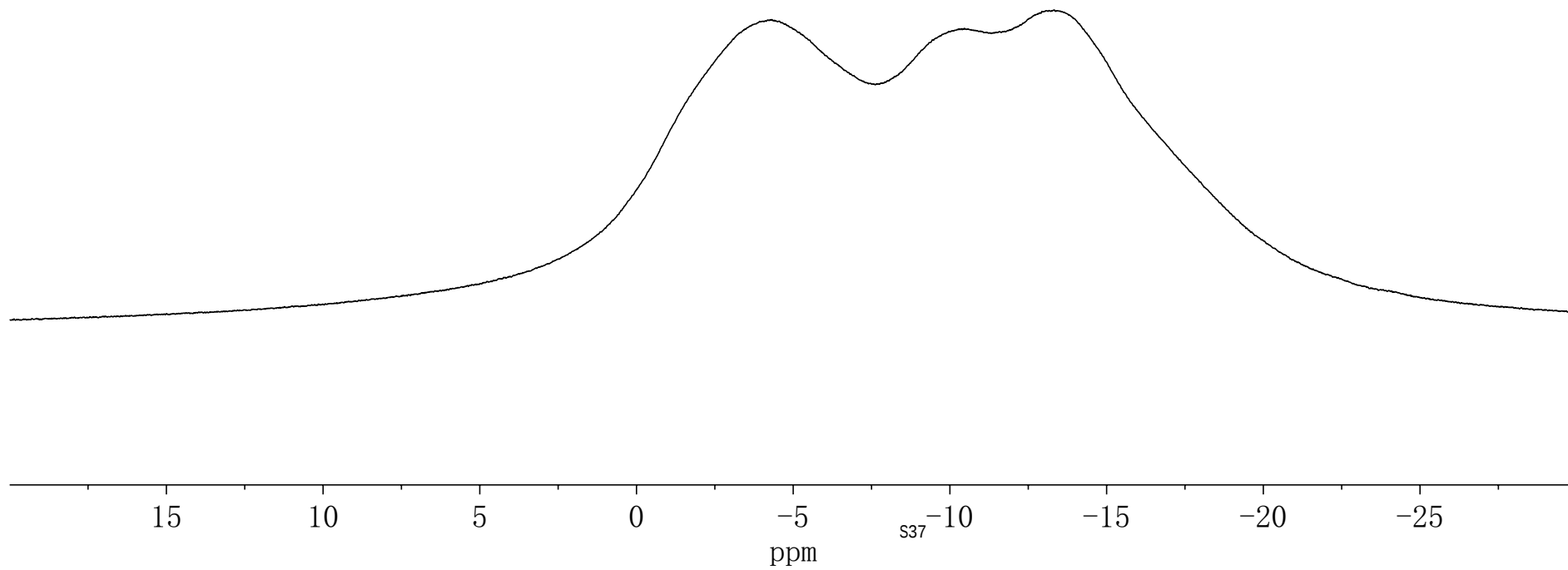
5f

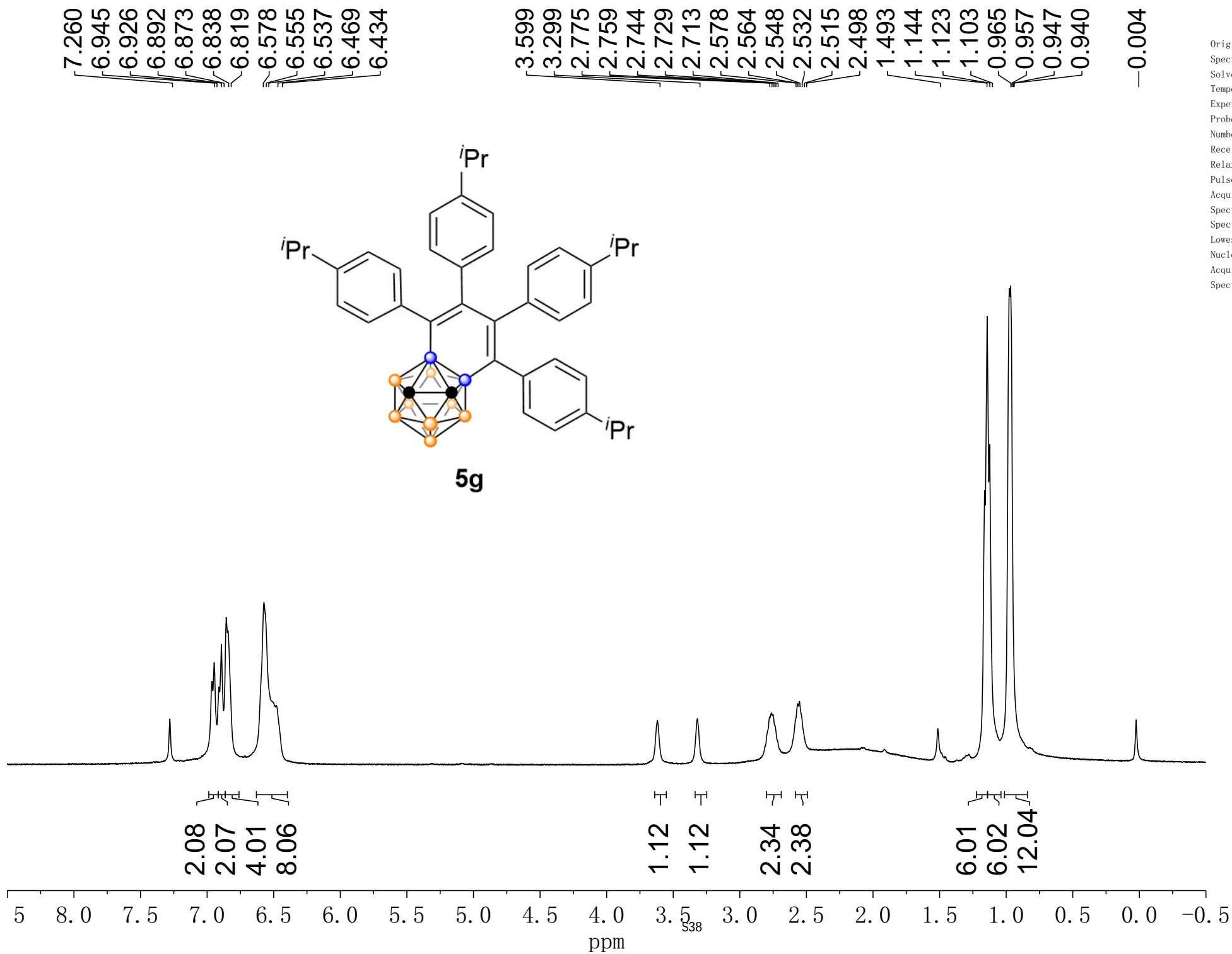
--4.33

--10.41

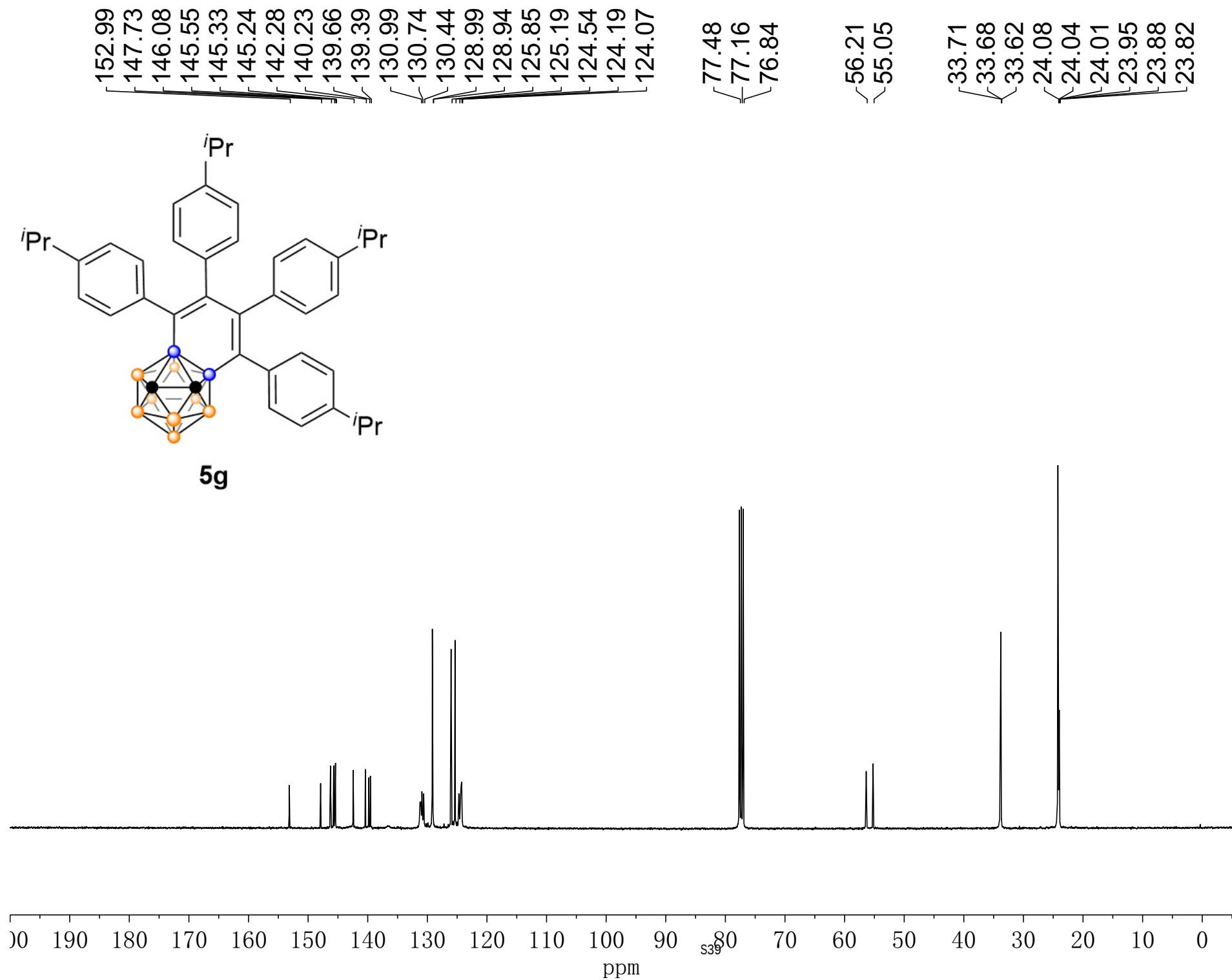
--13.17

Solvent	CDC13
Number of Scans	1024
Receiver Gain	128
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16416.9
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

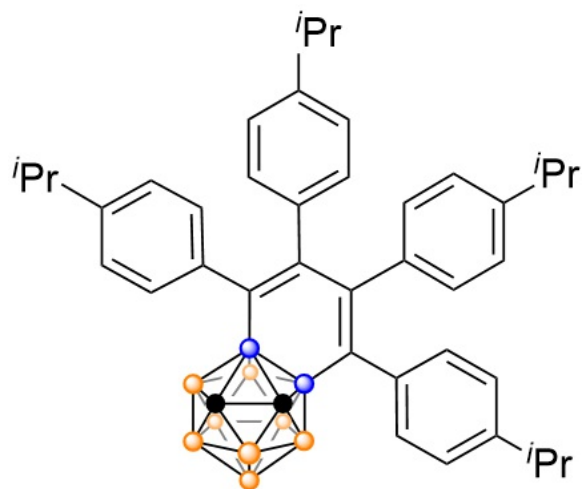




Origin	Varian
Spectrometer	vnmrs
Solvent	CDCl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	20
Receiver Gain	28
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	400.03
Spectral Width	7225.4
Lowest Frequency	-796.5
Nucleus	¹ H
Acquired Size	21676
Spectral Size	65536



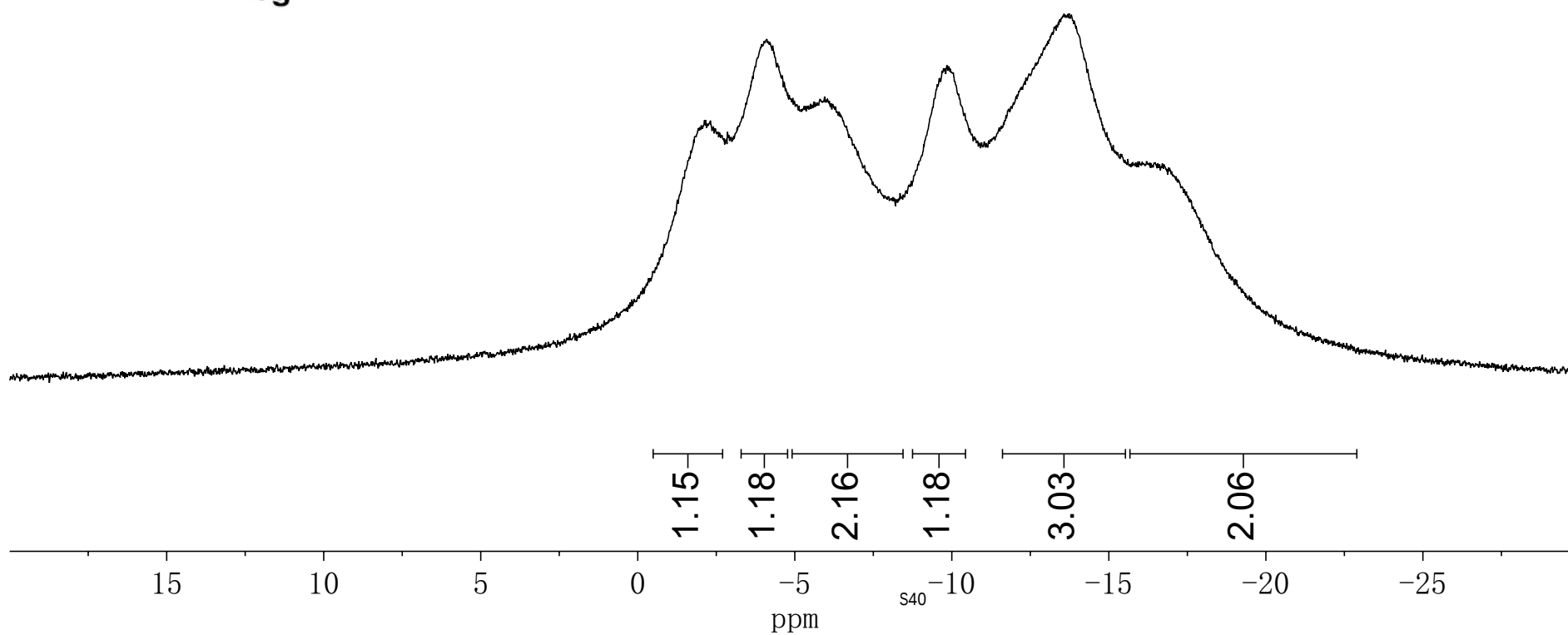
Origin	Varian
Spectrometer	nmrs
Solvent	cdcl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	13832
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3005
Spectrometer Frequency	100.60
Spectral Width	24509.8
Lowest Frequency	-1659.5
Nucleus	13C
Acquired Size	31875
Spectral Size	65536

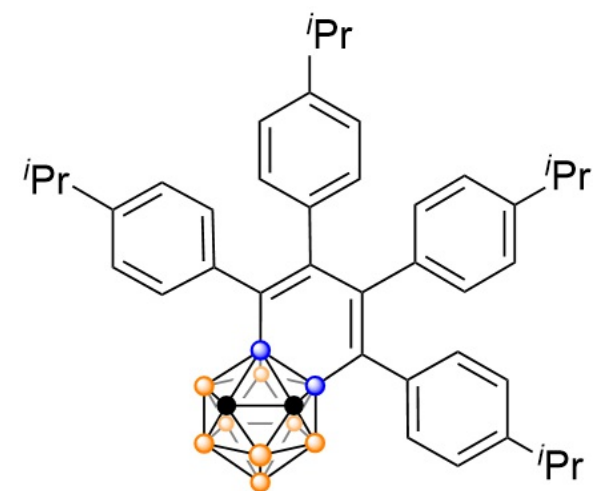


5g

δ -2.16
 δ -4.11
 δ -5.98
 δ -9.84
 δ -13.61
 δ -16.34

Solvent	CDC13
Number of Scans	256
Receiver Gain	228
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16391.5
Nucleus	¹¹ B
Acquired Size	16384
Spectral Size	32768

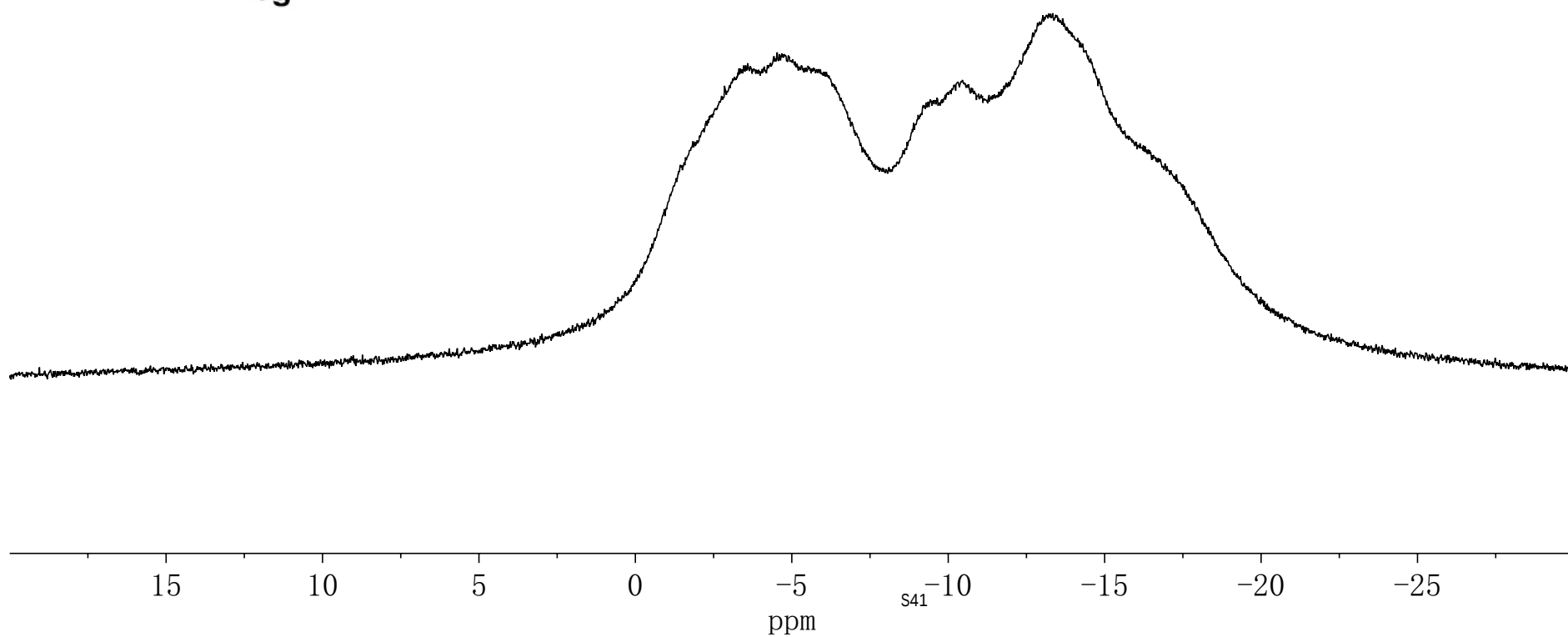


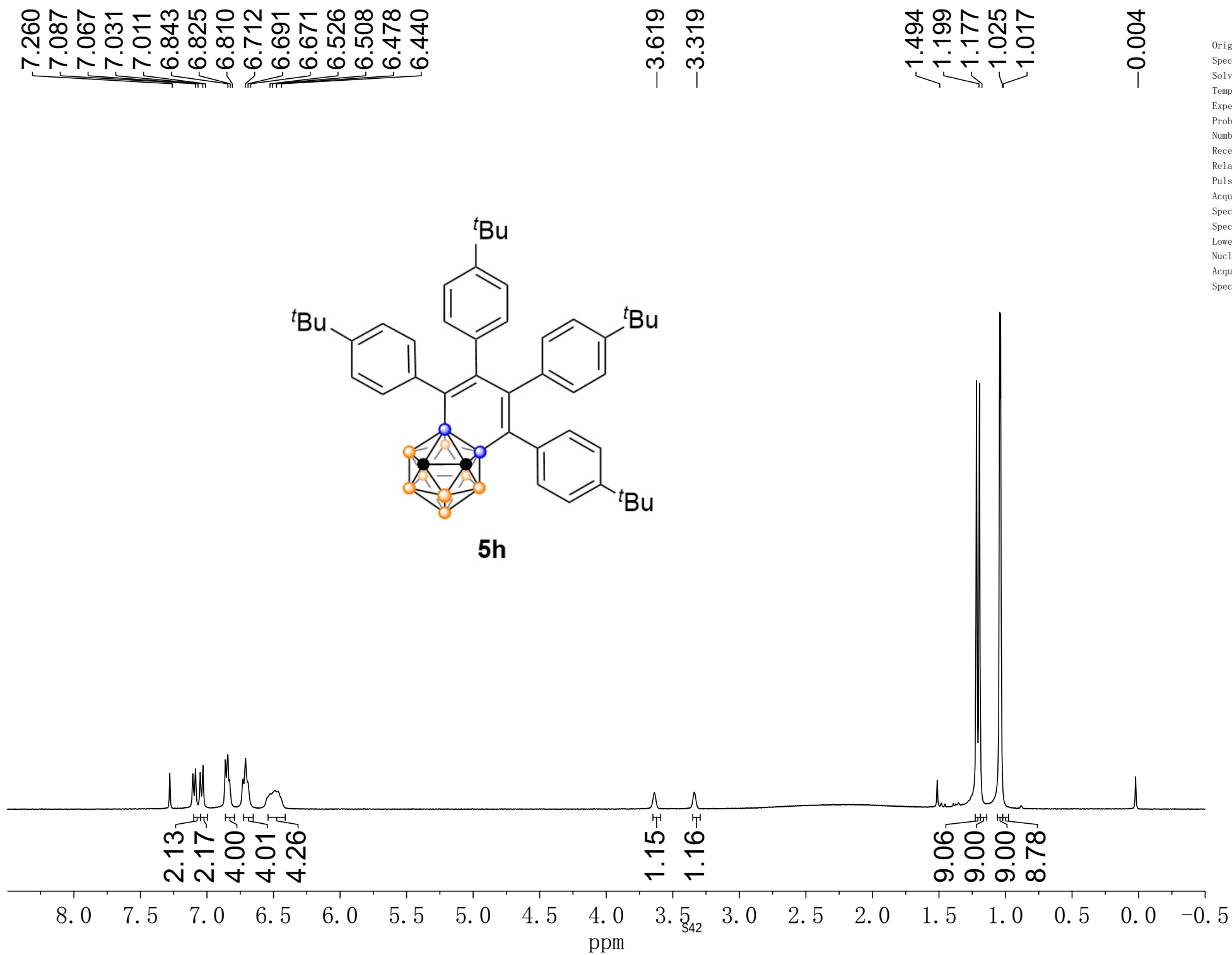


5g

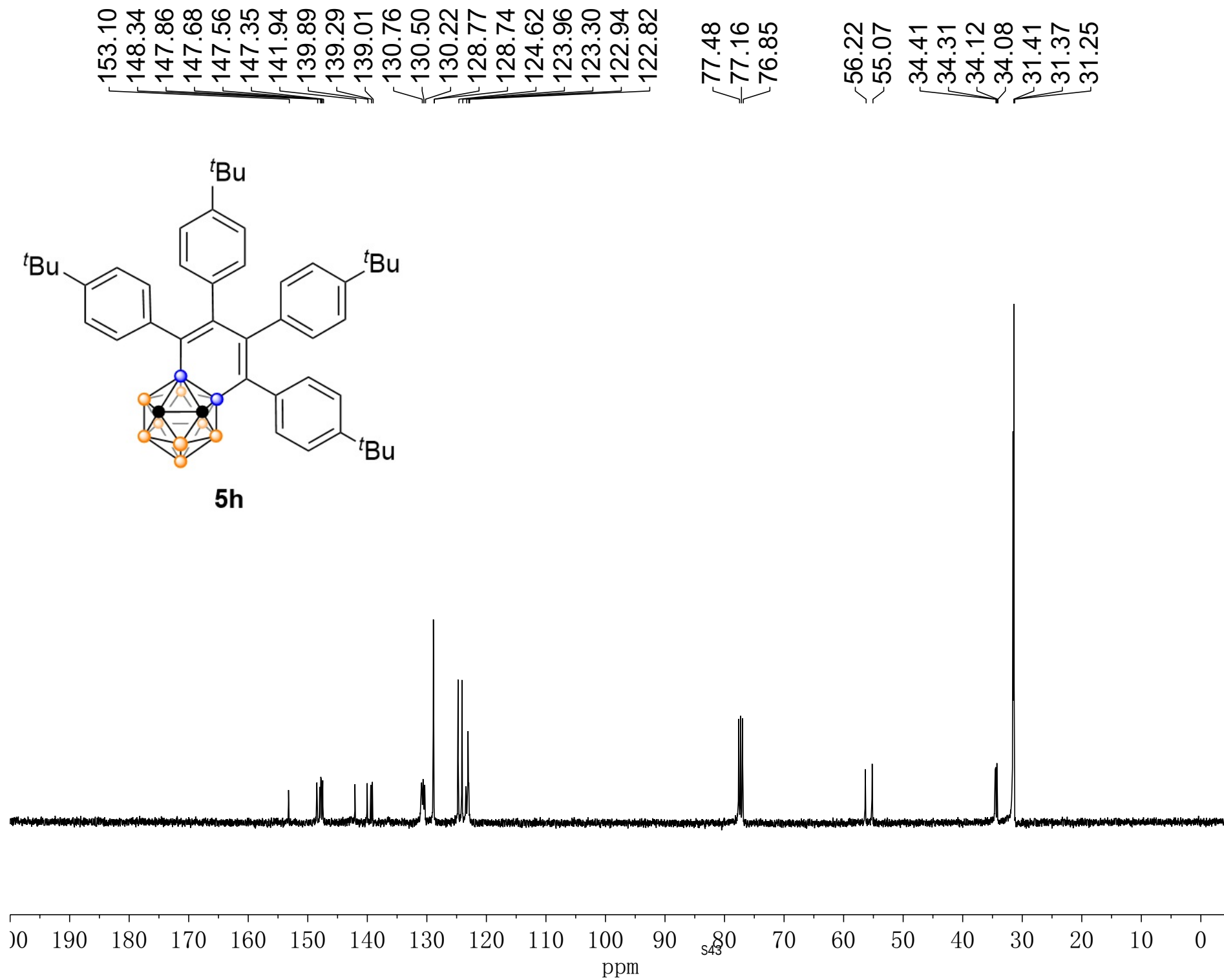
~-1.43
 ~-3.46
 ~-4.61
 ~-5.85
 ~-9.46
 ~-10.41
 --13.32
 --16.47

Solvent	CDC13
Number of Scans	256
Receiver Gain	287
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16391.5
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

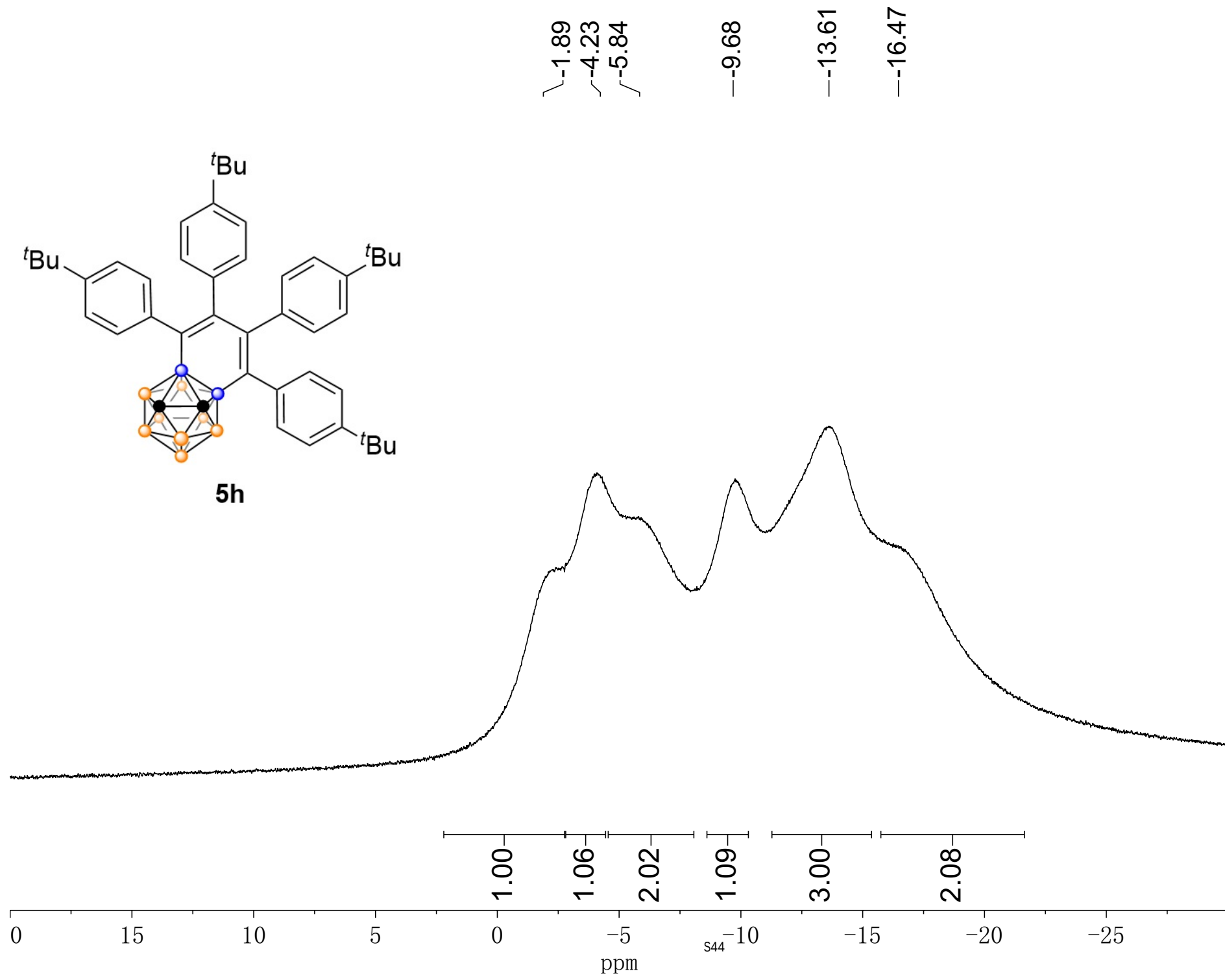




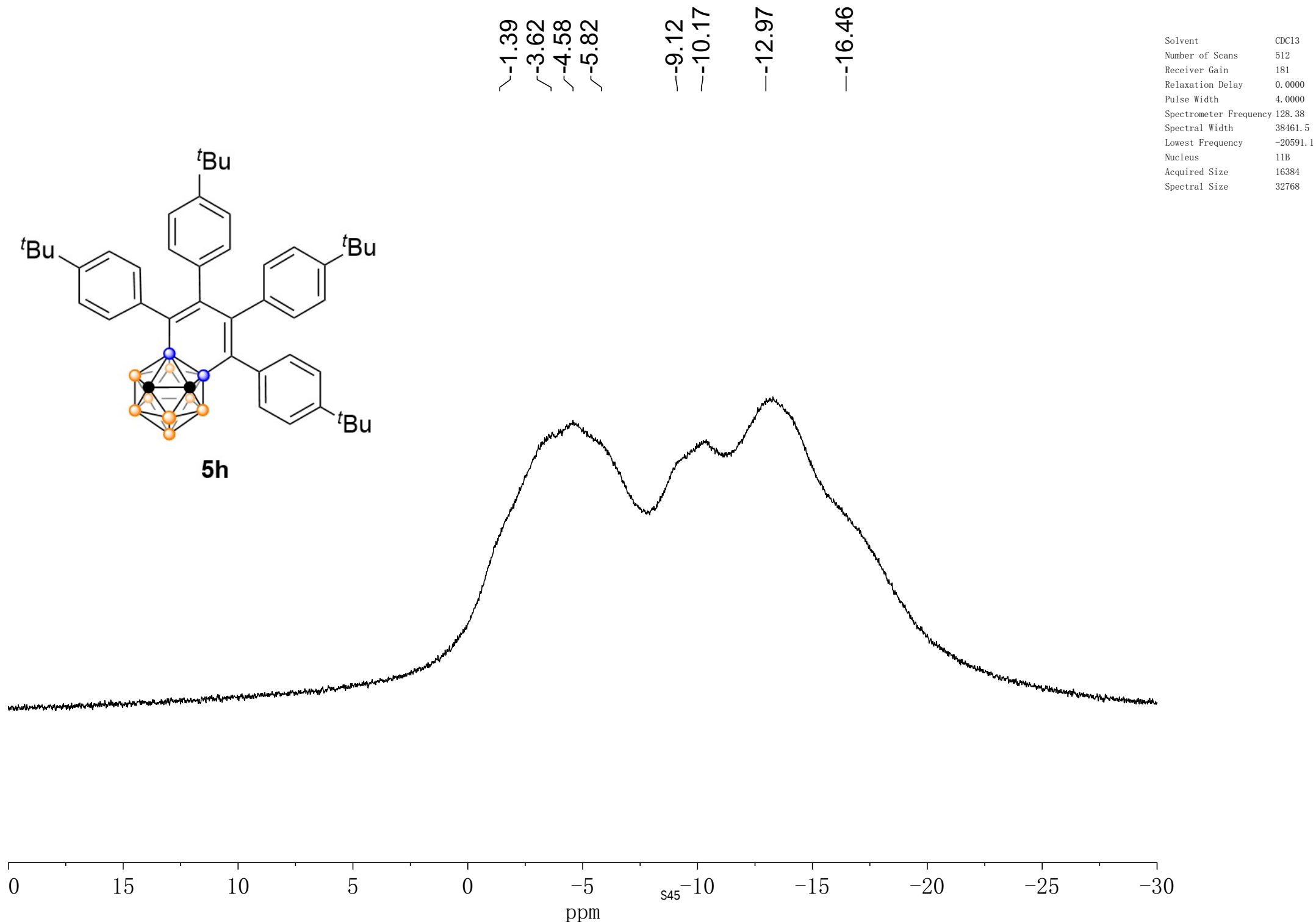
Origin	Varian
Spectrometer	vnmrs
Solvent	CDCl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	8
Receiver Gain	24
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	400.03
Spectral Width	7267.4
Lowest Frequency	-838.3
Nucleus	1H
Acquired Size	21802
Spectral Size	65536

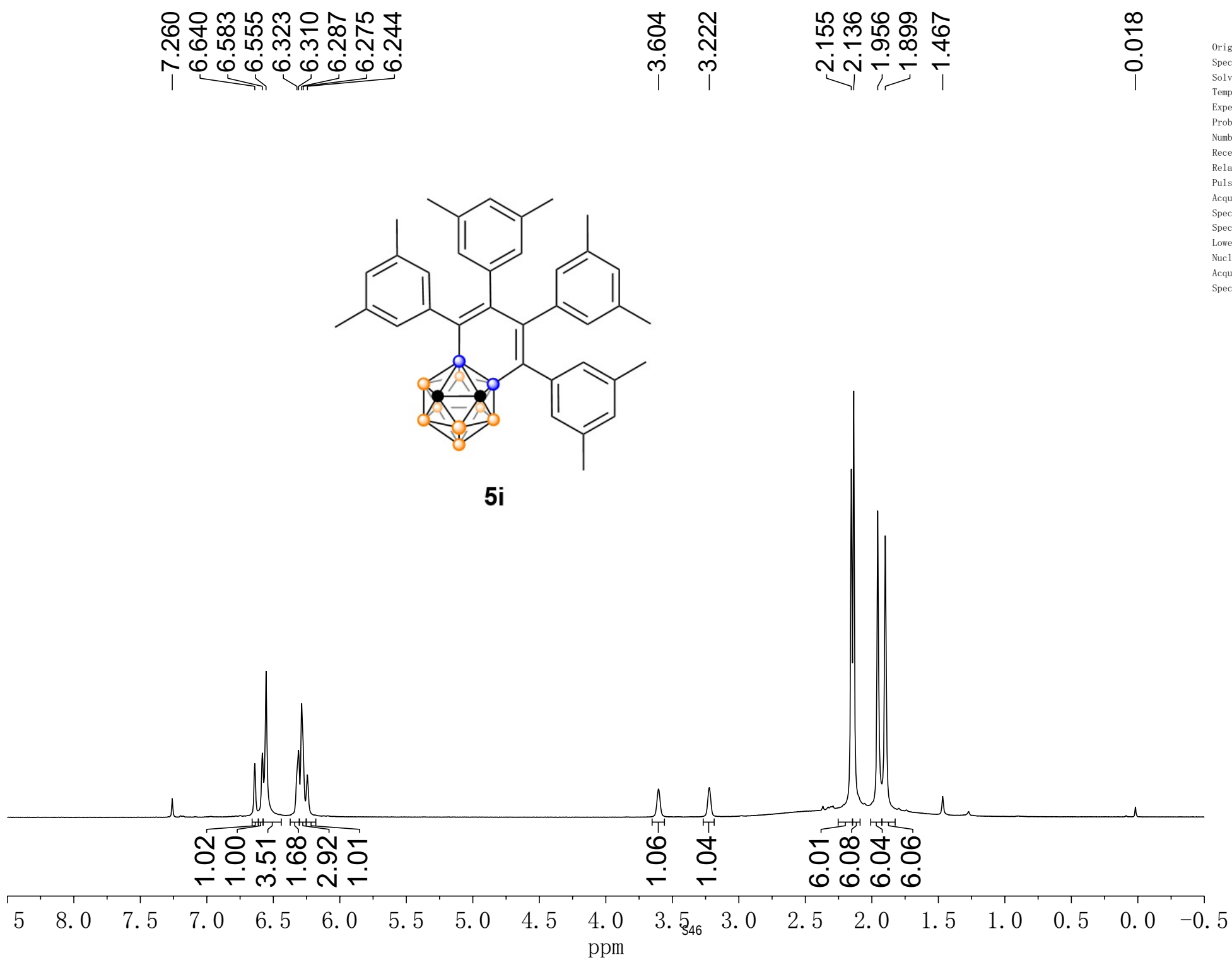


Origin	Varian
Spectrometer	vnmrs
Solvent	CDC13
Temperature	21.0
Experiment	1D
Probe	4nuc
Number of Scans	404
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3000
Spectrometer Frequency	100.52
Spectral Width	25000.0
Lowest Frequency	-1416.8
Nucleus	13C
Acquired Size	32500
Spectral Size	65536

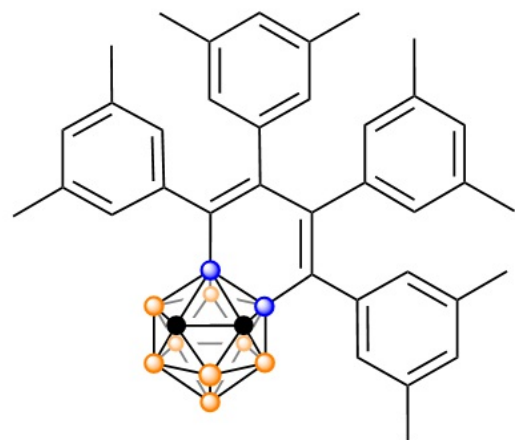


Solvent	CDCl3
Number of Scans	600
Receiver Gain	406
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16380.7
Nucleus	11B
Acquired Size	16384
Spectral Size	32768





Origin	Varian
Spectrometer	nmrs
Solvent	CDCl ₃
Temperature	21.0
Experiment	1D
Probe	4nuc
Number of Scans	8
Receiver Gain	26
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	399.72
Spectral Width	7183.9
Lowest Frequency	-793.9
Nucleus	¹ H
Acquired Size	21552
Spectral Size	65536



5i

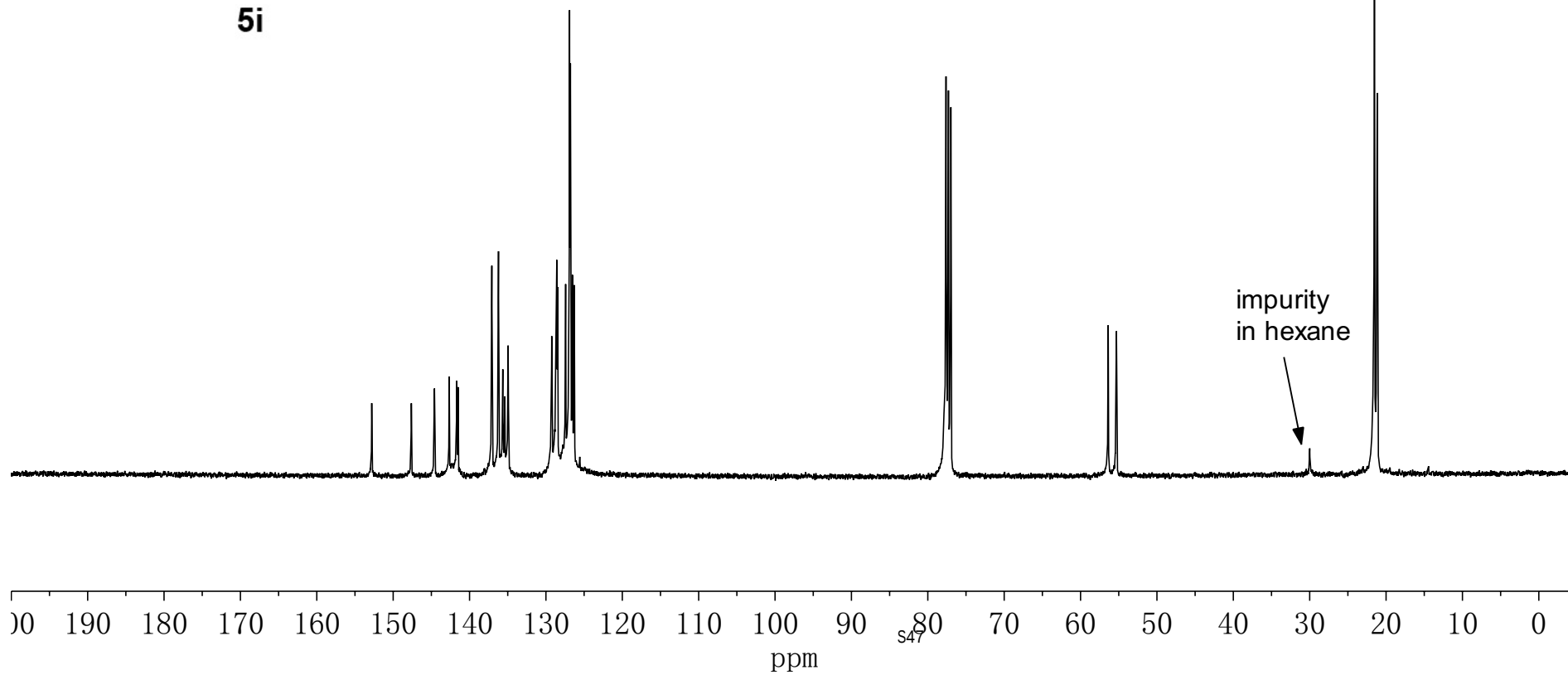
152.67
147.48
144.46
142.53
141.54
141.36
136.94
136.07
135.51
135.24
134.81
129.17
129.10
128.52
128.44
128.32
127.28
126.77
126.66
126.39
126.15

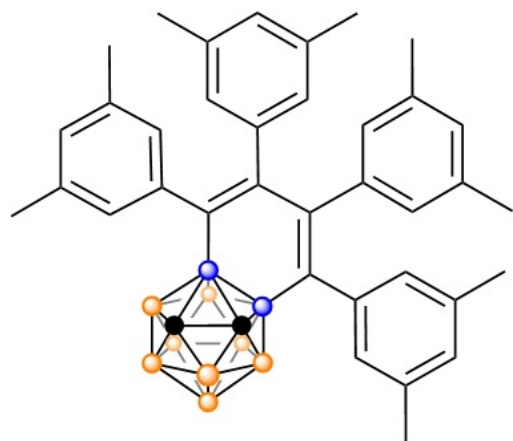
77.48
77.16
76.84

56.24
55.17

21.40
21.38
21.02
20.97

Origin	Varian
Spectrometer	nmrs
Solvent	cdcl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	16076
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3005
Spectrometer Frequency	100.60
Spectral Width	24509.8
Lowest Frequency	-1665.5
Nucleus	¹³ C
Acquired Size	31875
Spectral Size	65536

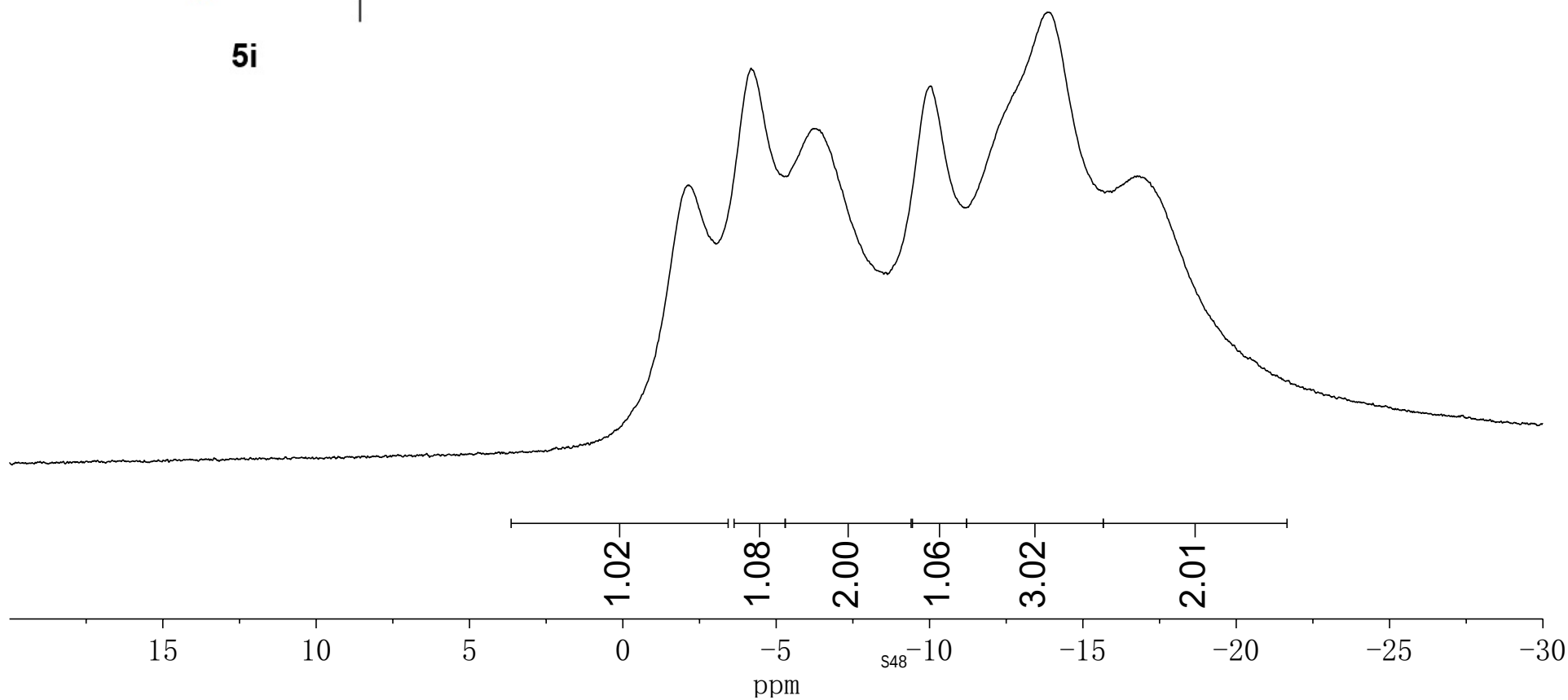


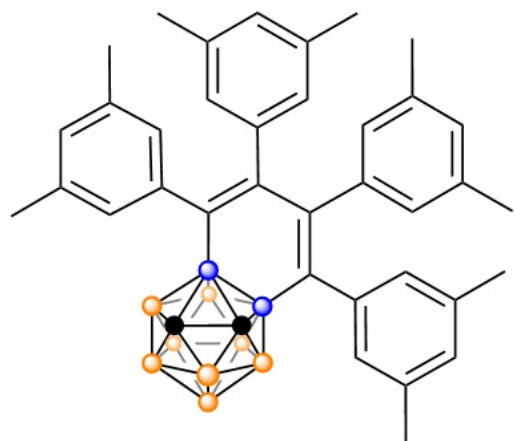


5i

-2.08
 -4.17
 -6.35
 -10.00
 -13.94
 -16.75

Solvent	CDC13
Number of Scans	814
Receiver Gain	256
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-12696.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

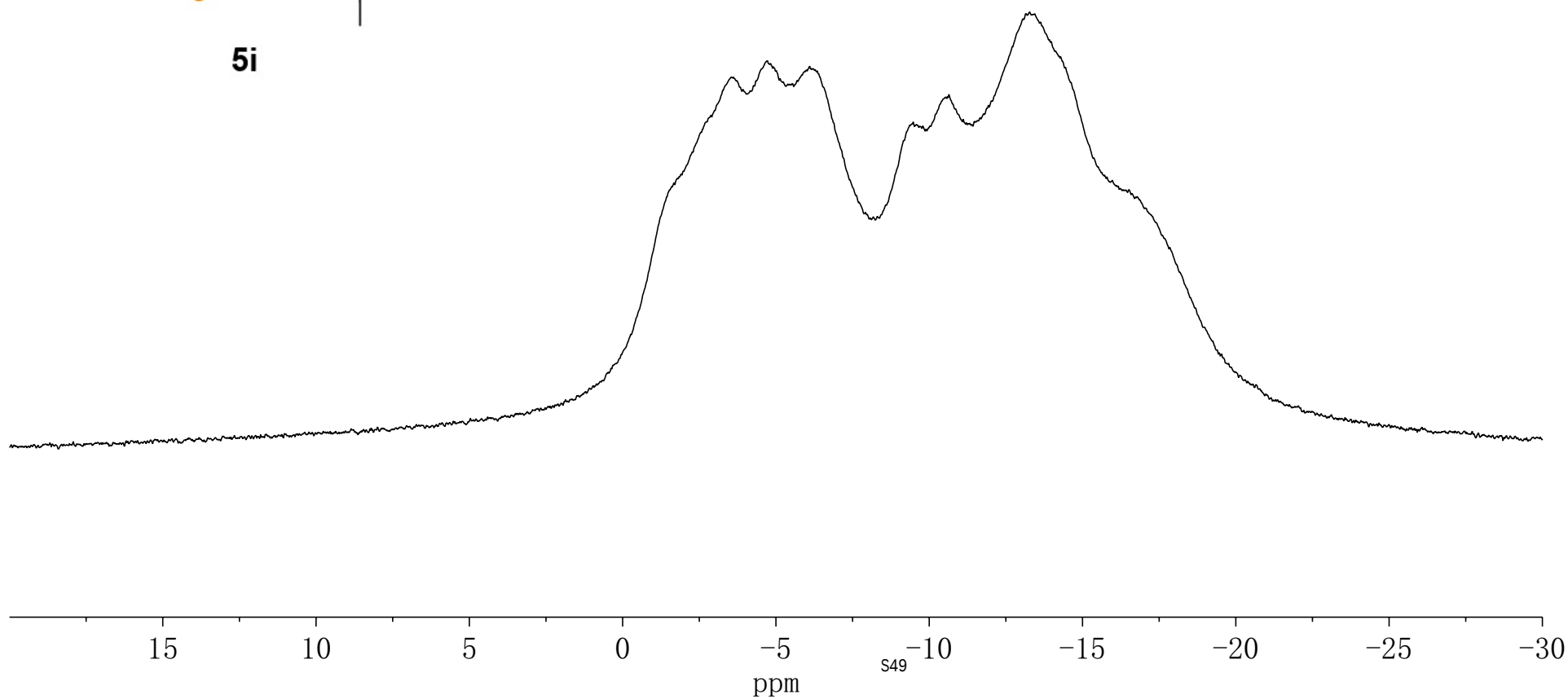


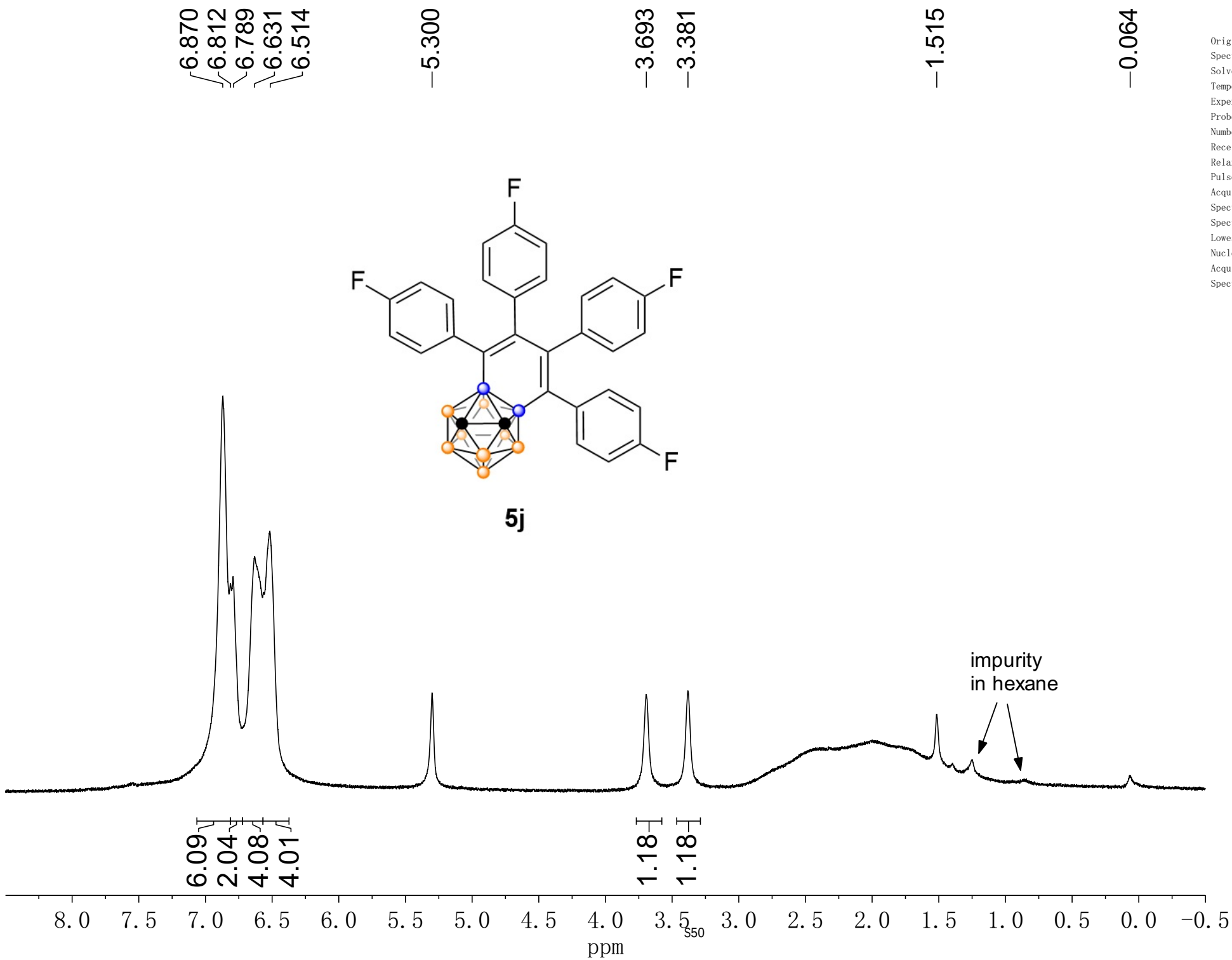


5i

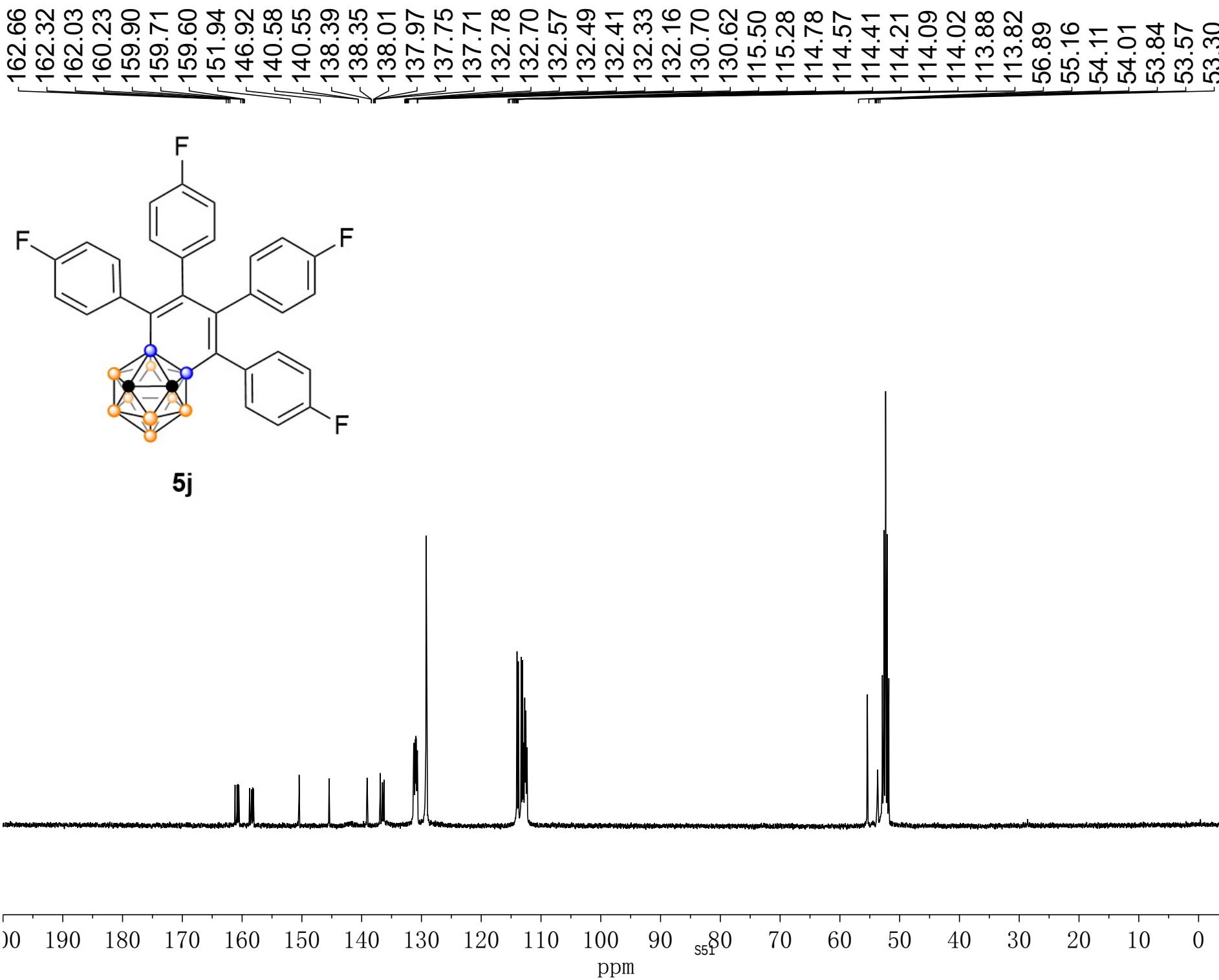
1.42
 3.53
 4.80
 6.39
 9.39
 10.56
 13.46
 17.38

Solvent	CDC13
Number of Scans	368
Receiver Gain	512
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-12696.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

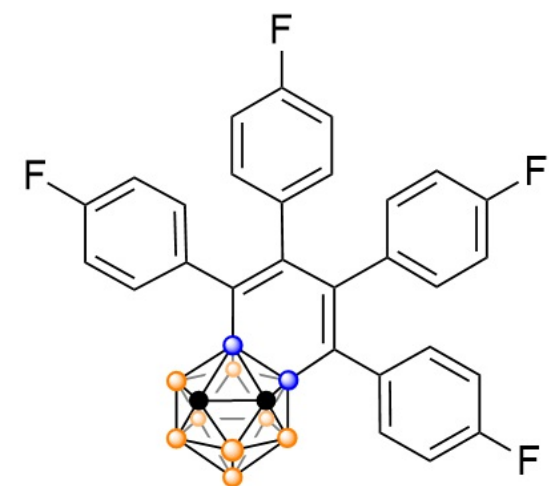




Origin	Varian
Spectrometer	vnmr5
Solvent	CD2Cl2
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	8
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	400.03
Spectral Width	7267.4
Lowest Frequency	-1628.8
Nucleus	1H
Acquired Size	21802
Spectral Size	65536



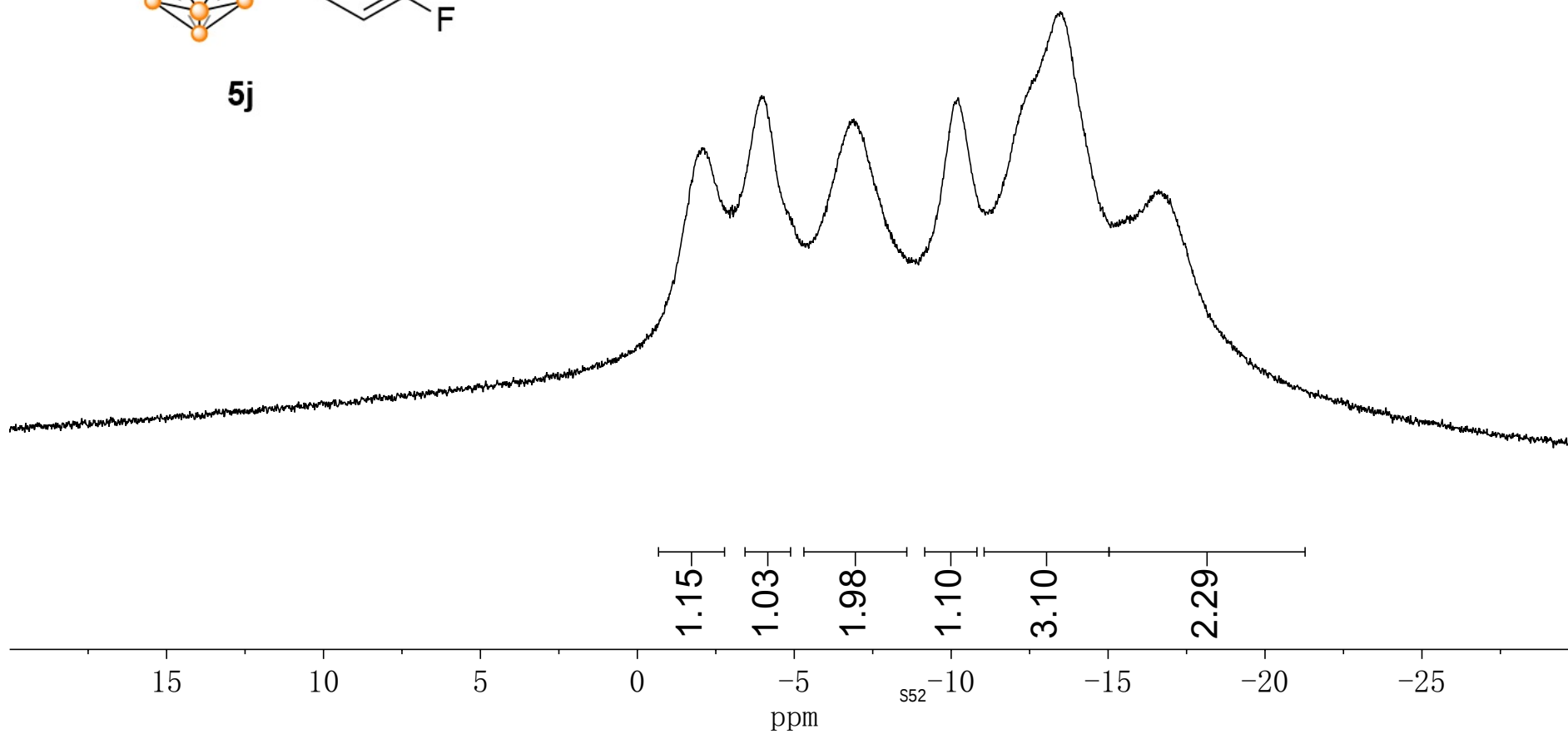
Origin	Varian
Spectrometer	nmr
Solvent	cd2cl2
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	11796
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3000
Spectrometer Frequency	100.60
Spectral Width	26595.7
Lowest Frequency	-2532.0
Nucleus	¹³ C
Acquired Size	34575
Spectral Size	131072

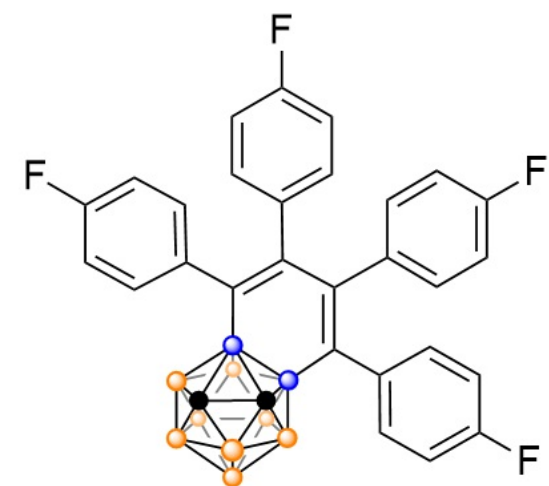


5j

--2.00
--3.97
--6.83
--10.13
--13.18
--16.79

Solvent	CD2Cl2
Number of Scans	512
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16414.0
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

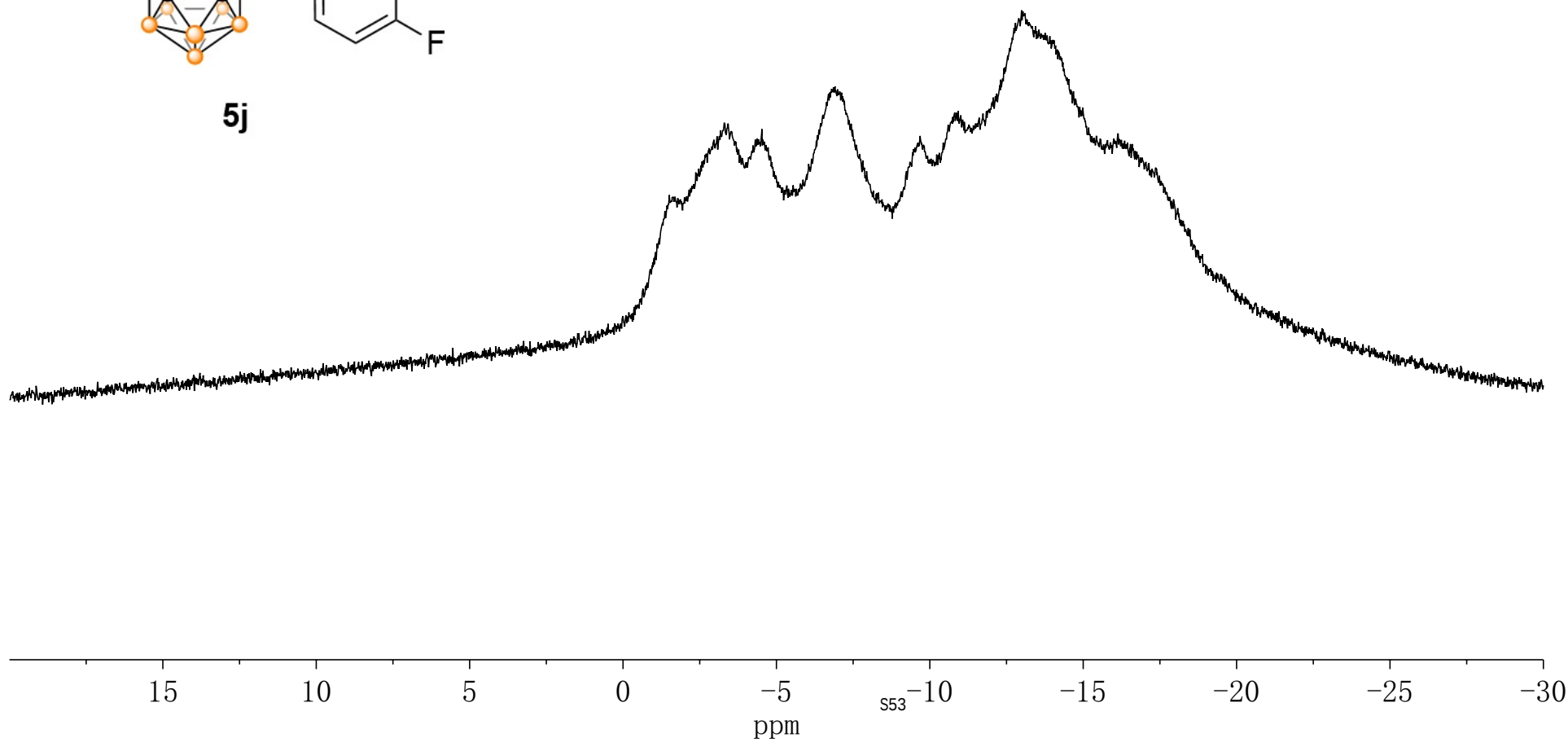


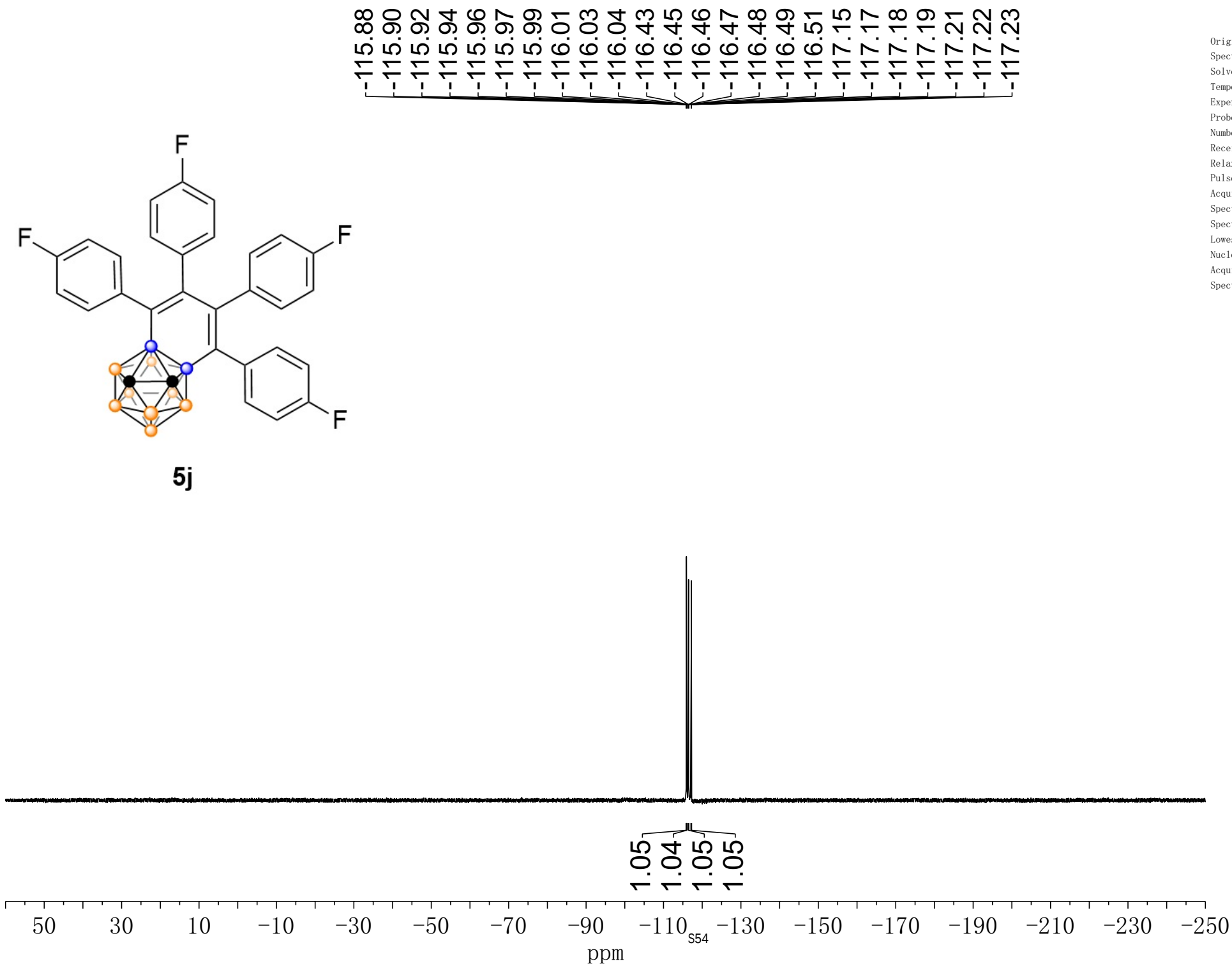


5j

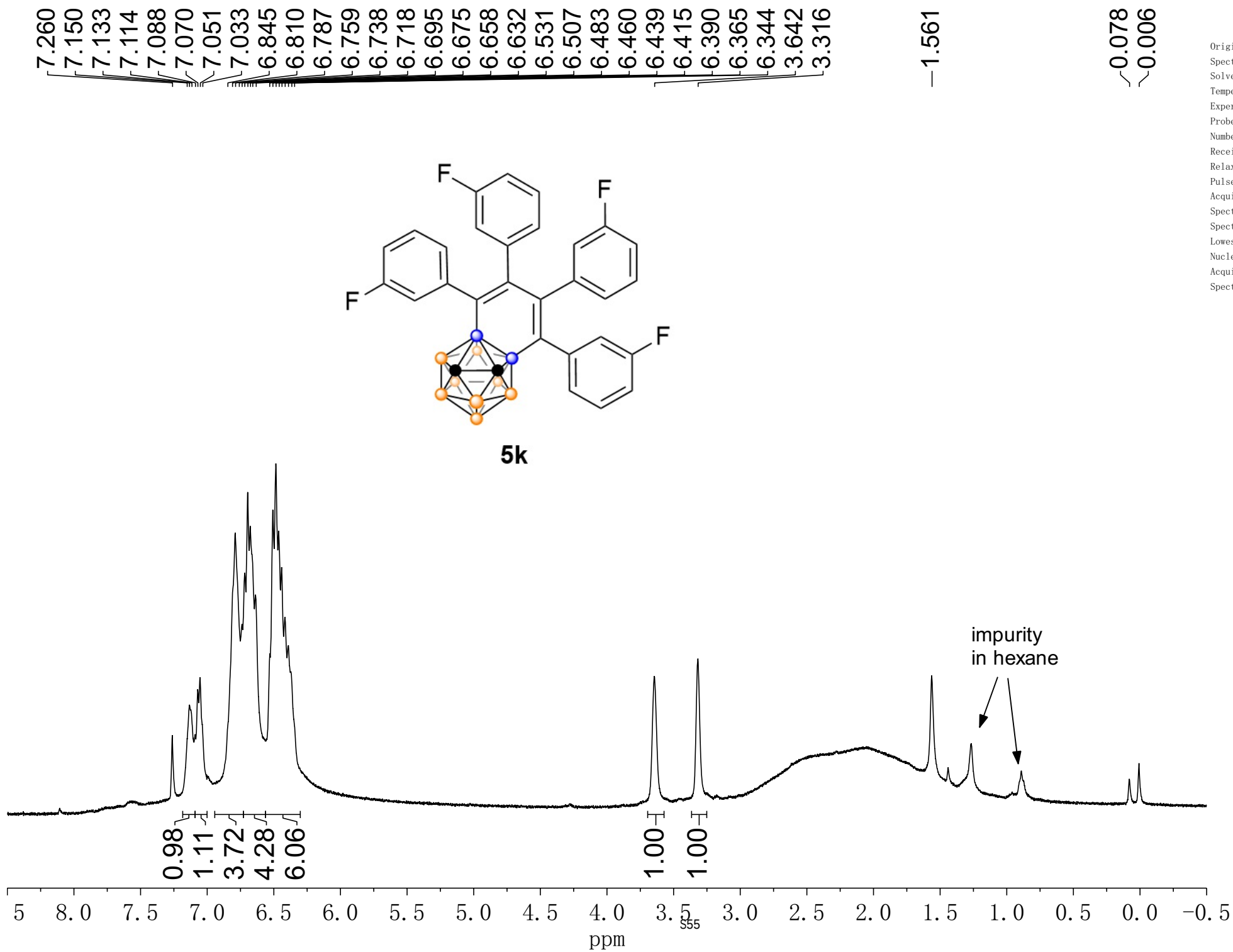
~-1.69
 ~-3.24
 ~-4.63
 ---7.06
 ---9.68
 ---10.81
 ---13.38
 ---17.65

Solvent	CD2Cl2
Number of Scans	256
Receiver Gain	406
Relaxation Delay	1.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16419.8
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

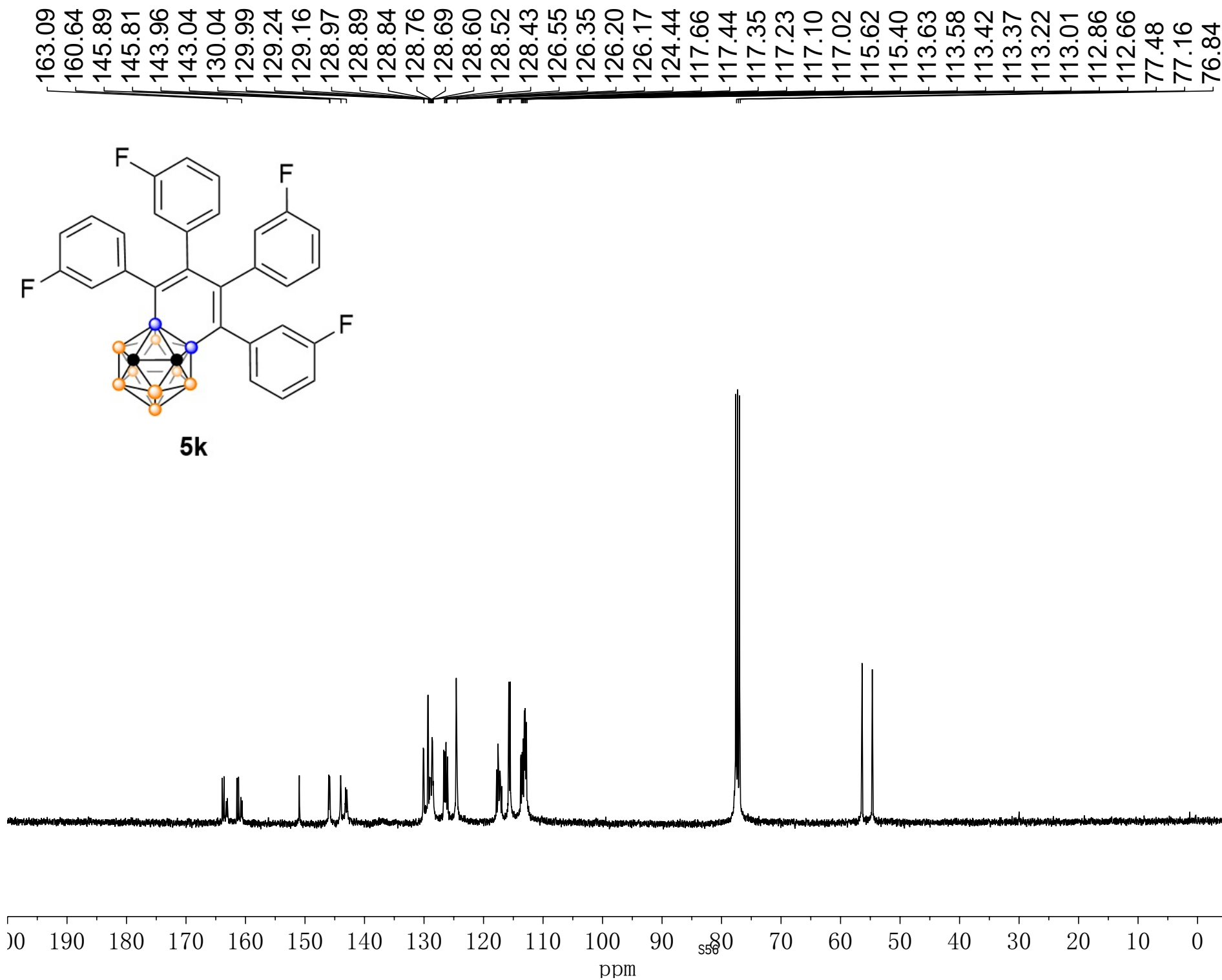




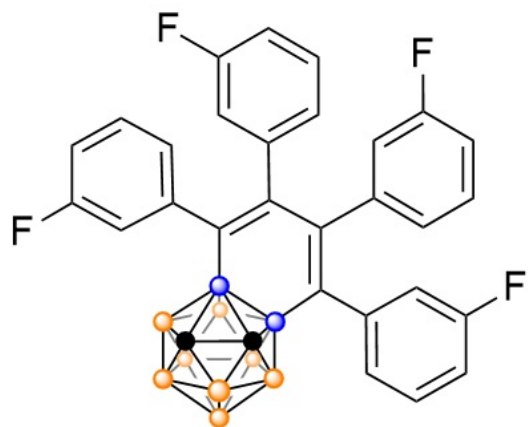
Origin	Varian
Spectrometer	vnmrs
Solvent	CD2Cl2
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	40
Receiver Gain	58
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.0000
Spectrometer Frequency	376.36
Spectral Width	138888.9
Lowest Frequency	-114612.8
Nucleus	19F
Acquired Size	138889
Spectral Size	524288



Origin	Varian
Spectrometer	vnmr5
Solvent	CDCl3
Temperature	21.0
Experiment	1D
Probe	4nuc
Number of Scans	8
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	399.72
Spectral Width	7183.9
Lowest Frequency	-792.3
Nucleus	1H
Acquired Size	21552
Spectral Size	65536



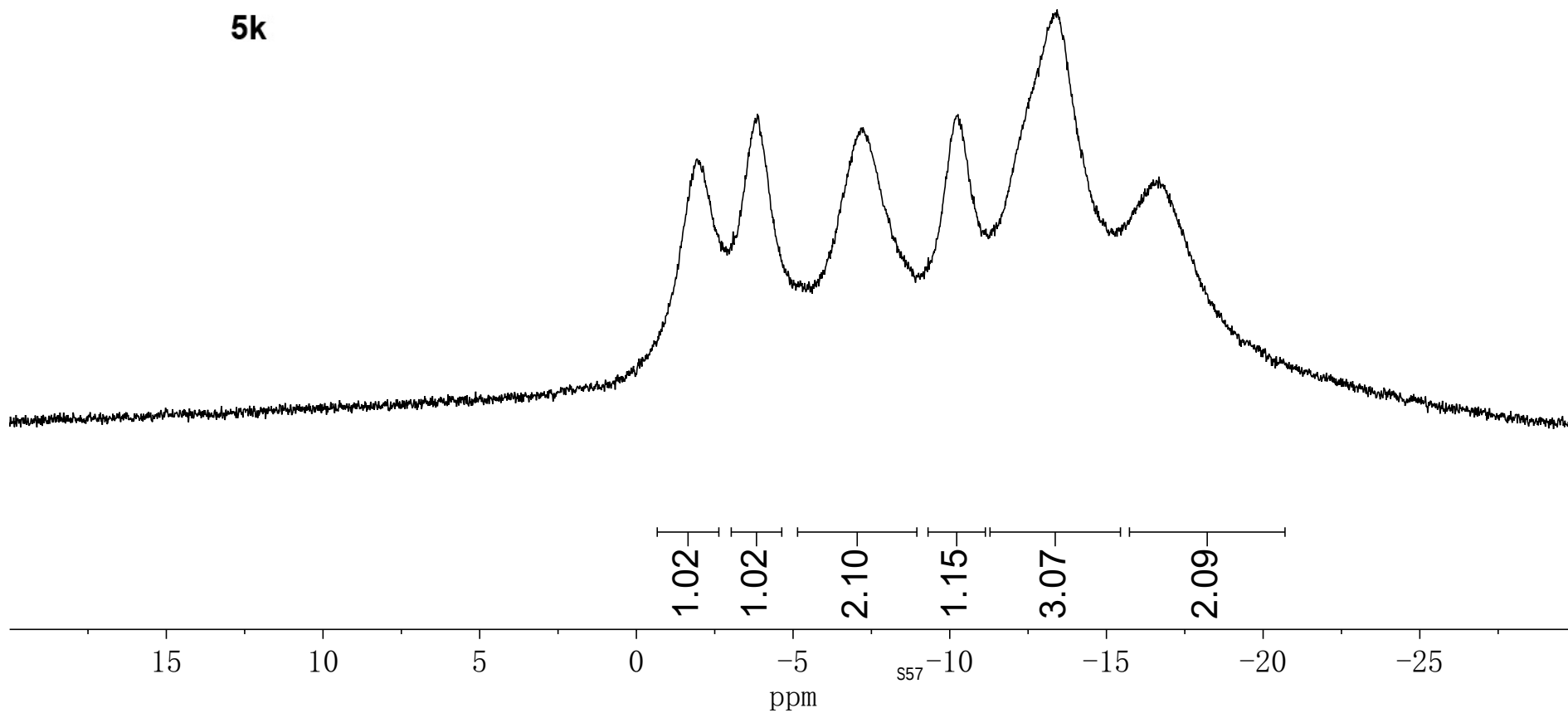
Origin	Varian
Spectrometer	vnmrs
Solvent	CDC13
Temperature	21.0
Experiment	1D
Probe	4nuc
Number of Scans	10760
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3000
Spectrometer Frequency	100.52
Spectral Width	24509.8
Lowest Frequency	-1393.3
Nucleus	13C
Acquired Size	31863
Spectral Size	65536

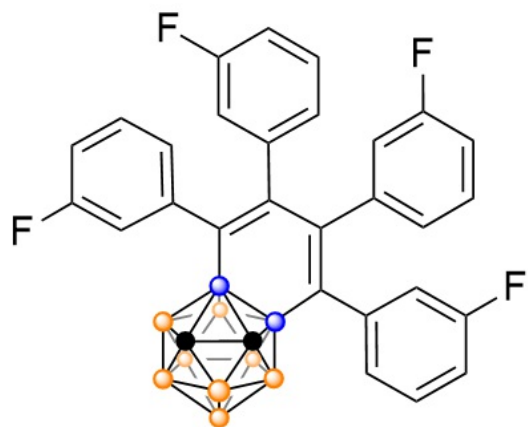


5k

--1.92
--3.82
--7.15
--10.26
--13.24
--16.66

Solvent	CDC13
Number of Scans	485
Receiver Gain	256
Relaxation Delay	0.1000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16421.9
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

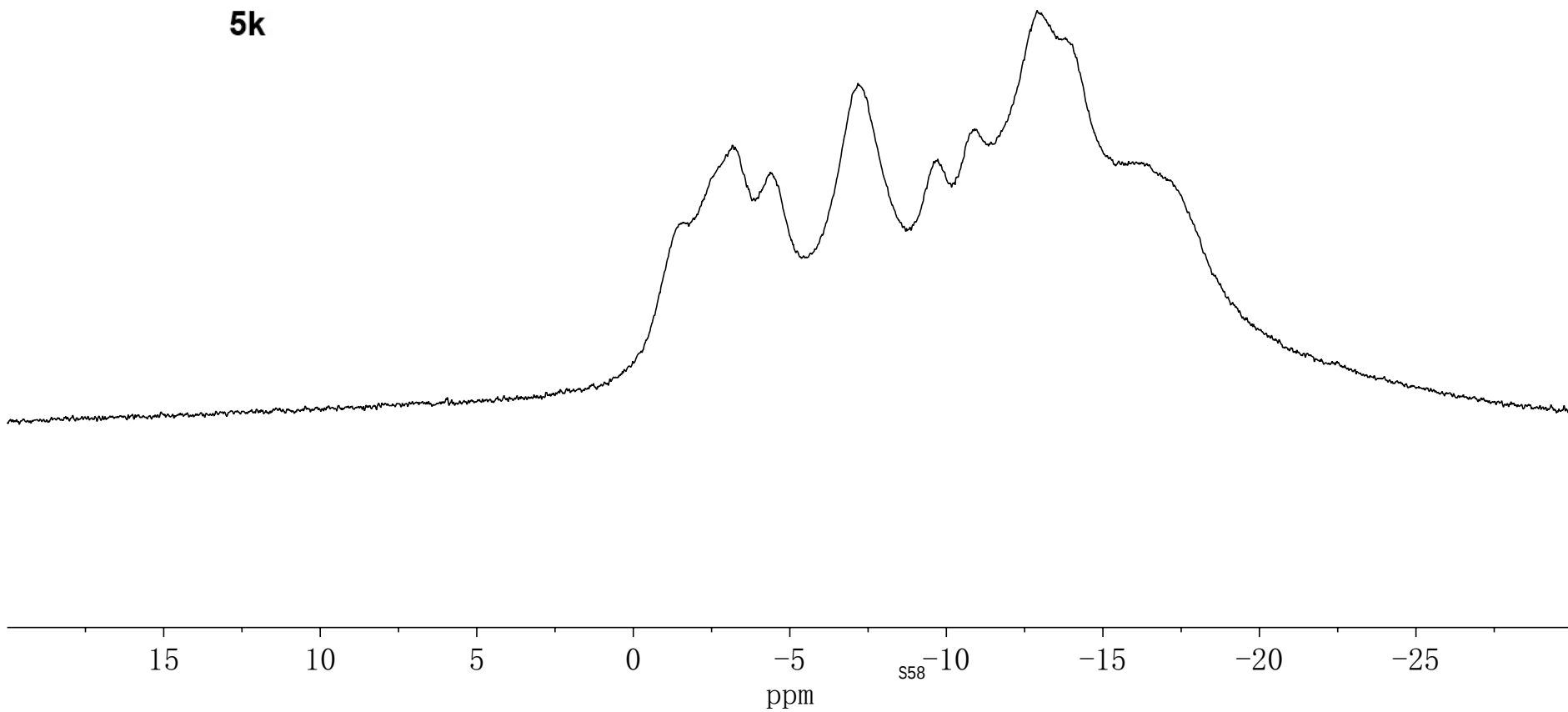


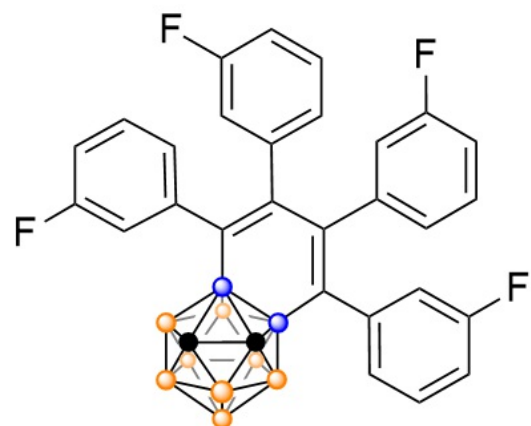


5k

~-1.40
 ~-3.29
 ~-4.47
 --7.19
 ~-9.61
 ~-10.96
 ~-12.92
 ~-14.07
 --17.11

Solvent	CDC13
Number of Scans	708
Receiver Gain	512
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	38461.5
Lowest Frequency	-20552.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

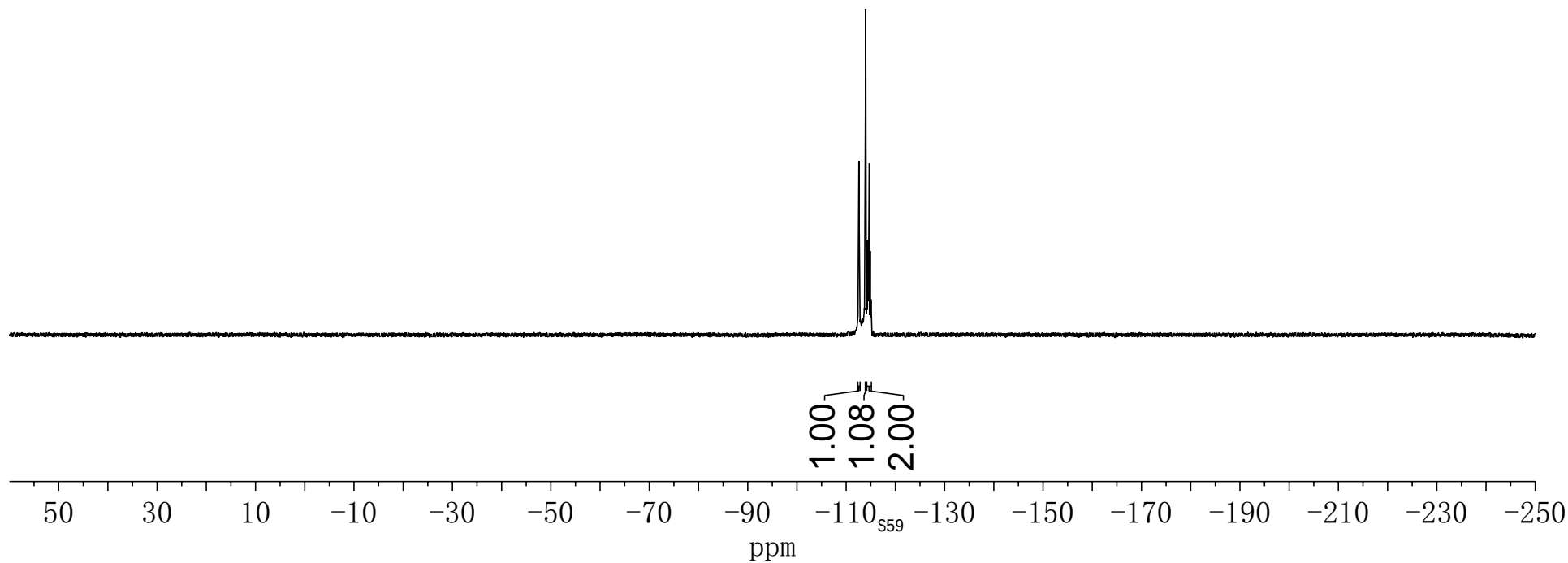




5k

112.60
 113.92
 113.94
 113.97
 113.99
 114.01
 114.22
 114.24
 114.26
 114.28
 114.41
 114.43
 114.45
 114.47
 114.53
 114.56
 114.58
 114.69
 114.71
 114.73
 114.75
 114.87
 114.90
 114.92
 114.98
 115.01
 115.03
 115.05

Origin	Varian
Spectrometer	vnmrs
Solvent	CDC13
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	80
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.6291
Spectrometer Frequency	376.41
Spectral Width	416666.7
Lowest Frequency	-206451.3
Nucleus	19F
Acquired Size	262144
Spectral Size	262144



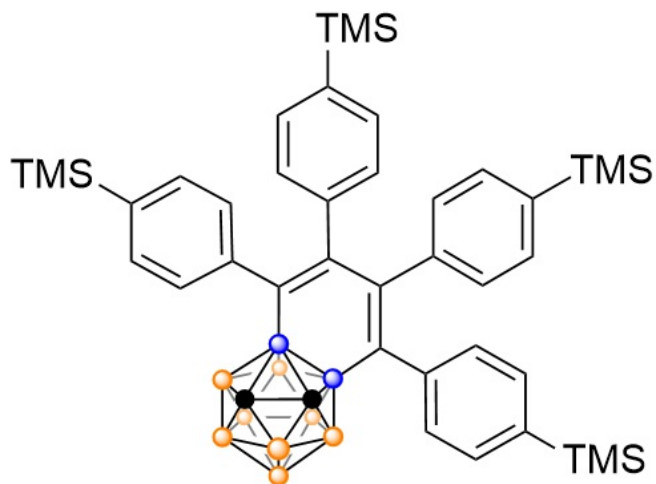
7.260
7.244
7.225
7.191
7.172
6.910
6.890
6.874
6.857
6.842
6.624
6.605
6.546
6.543
6.538

-3.612
-3.343

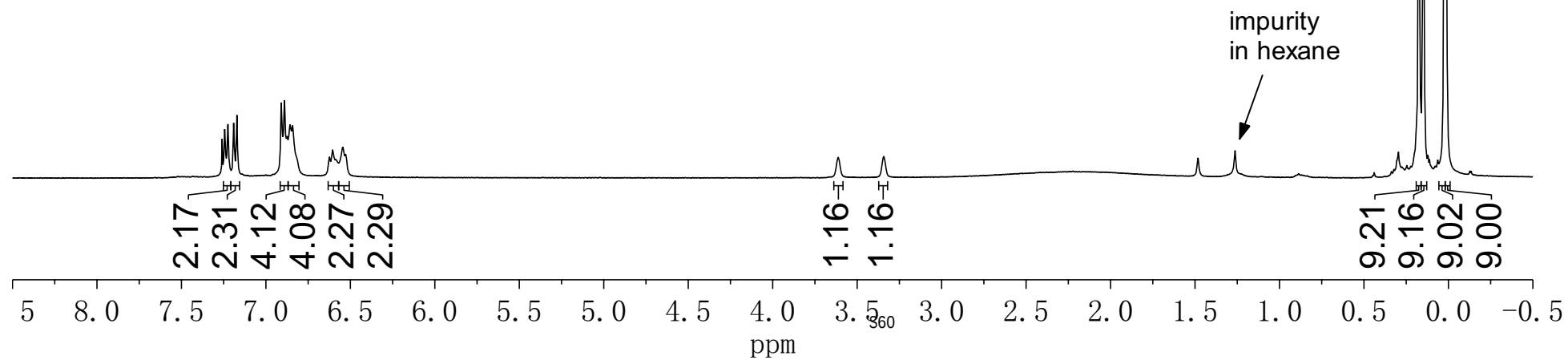
-1.482

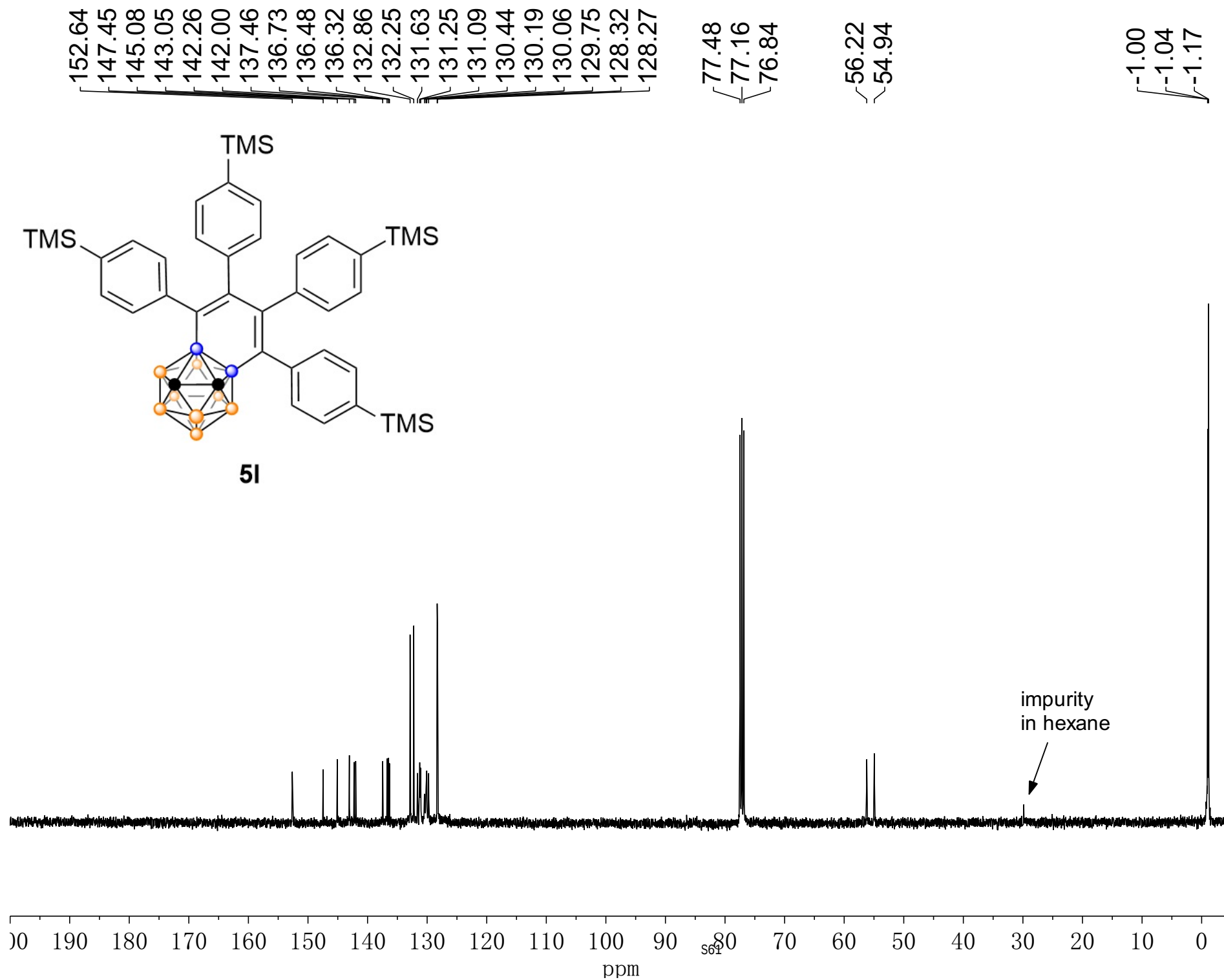
0.175
0.149
0.024
0.016

Origin	Varian
Spectrometer	nmrs
Solvent	CDCl ₃
Temperature	25.0
Experiment	1D
Probe	4nuc
Number of Scans	4
Receiver Gain	36
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	399.72
Spectral Width	7183.9
Lowest Frequency	-793.9
Nucleus	¹ H
Acquired Size	21552
Spectral Size	65536

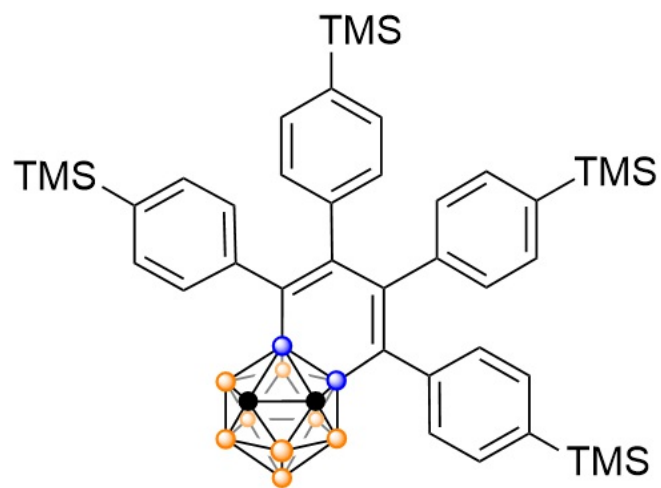


5I





Origin	Varian
Spectrometer	nmrs
Solvent	cdcl3
Temperature	25.0
Experiment	1D
Probe	4nuc
Number of Scans	940
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3369
Spectrometer Frequency	100.52
Spectral Width	24509.8
Lowest Frequency	-1319.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

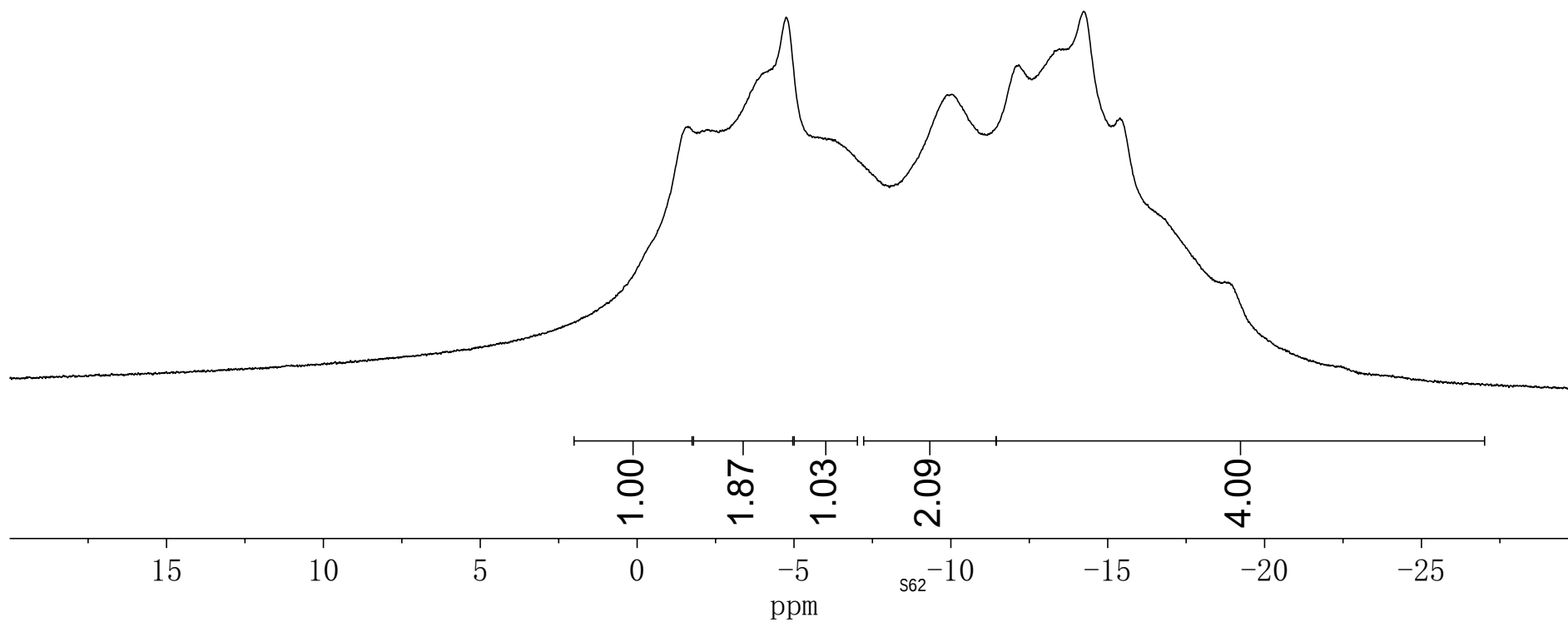


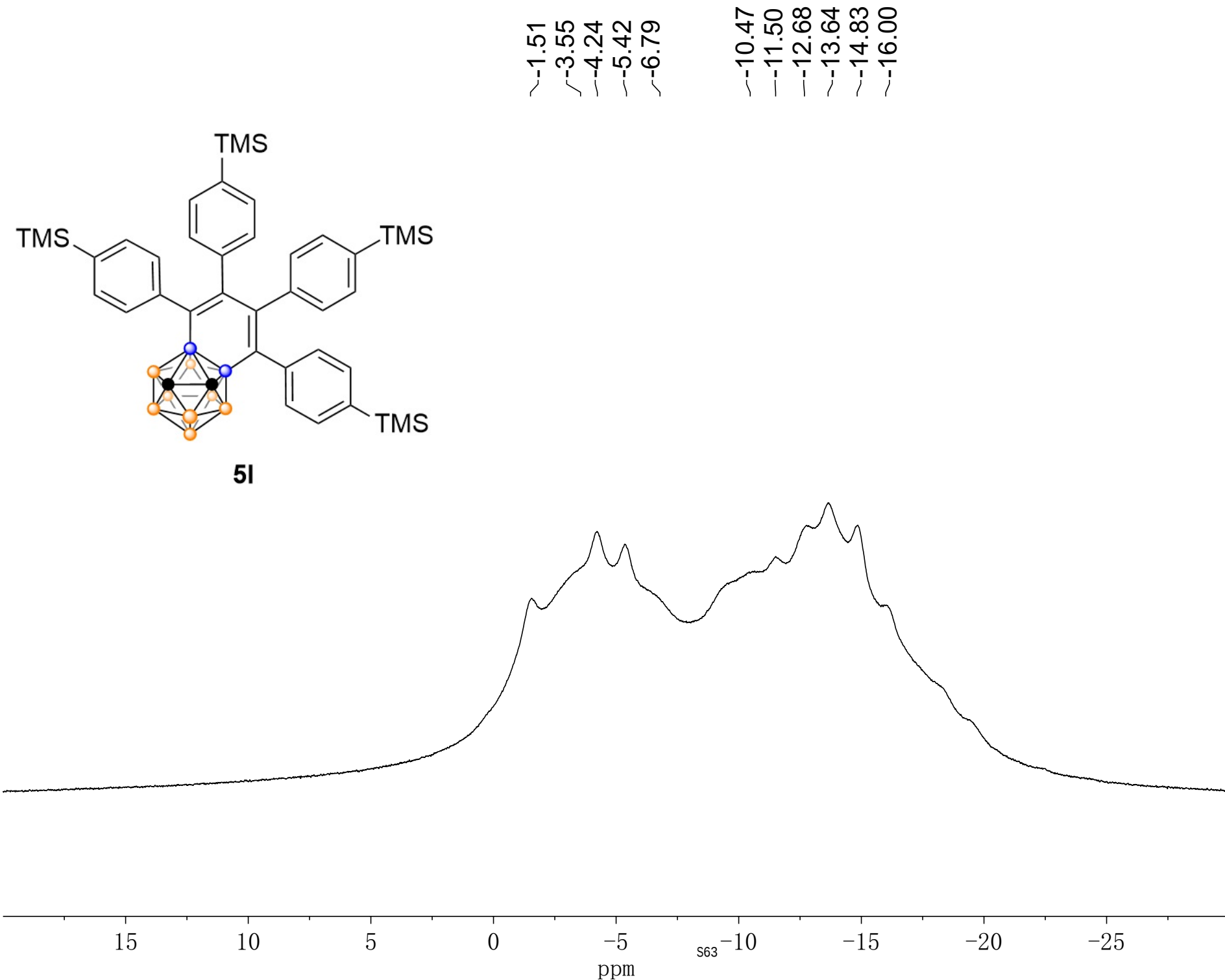
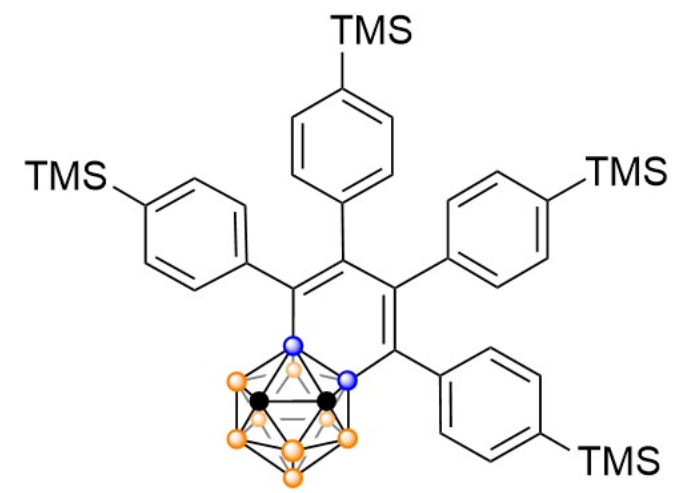
5I

~1.55
~2.36
~4.08
~4.79
~6.49

~-9.96
~-12.08
~-13.44
~-14.29
~-15.43

Solvent	CDC13
Number of Scans	1024
Receiver Gain	203
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16416.9
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



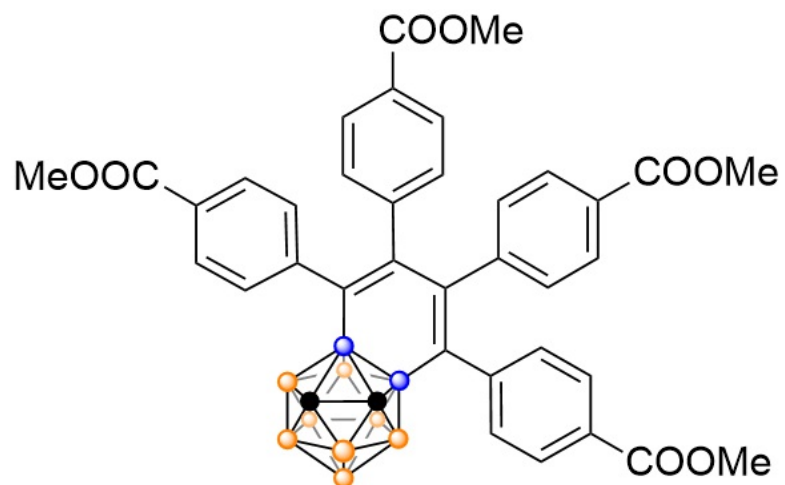


Solvent	CDCl ₃
Number of Scans	1024
Receiver Gain	203
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-16416.9
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

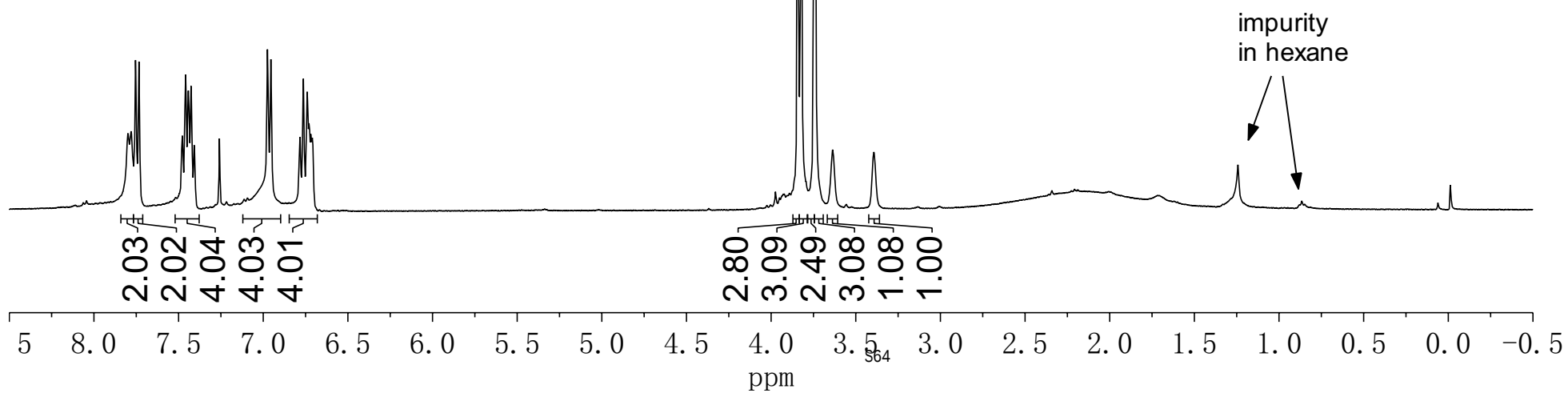
7.801
7.781
7.755
7.735
7.483
7.478
7.463
7.459
7.447
7.444
7.440
7.428
7.424
7.410
7.406
7.260
6.976
6.956
6.788
6.783
6.768
6.763
6.741
6.732
6.727
6.721
6.717
6.711
6.707
3.845
3.824
3.748
3.742
3.637
3.394

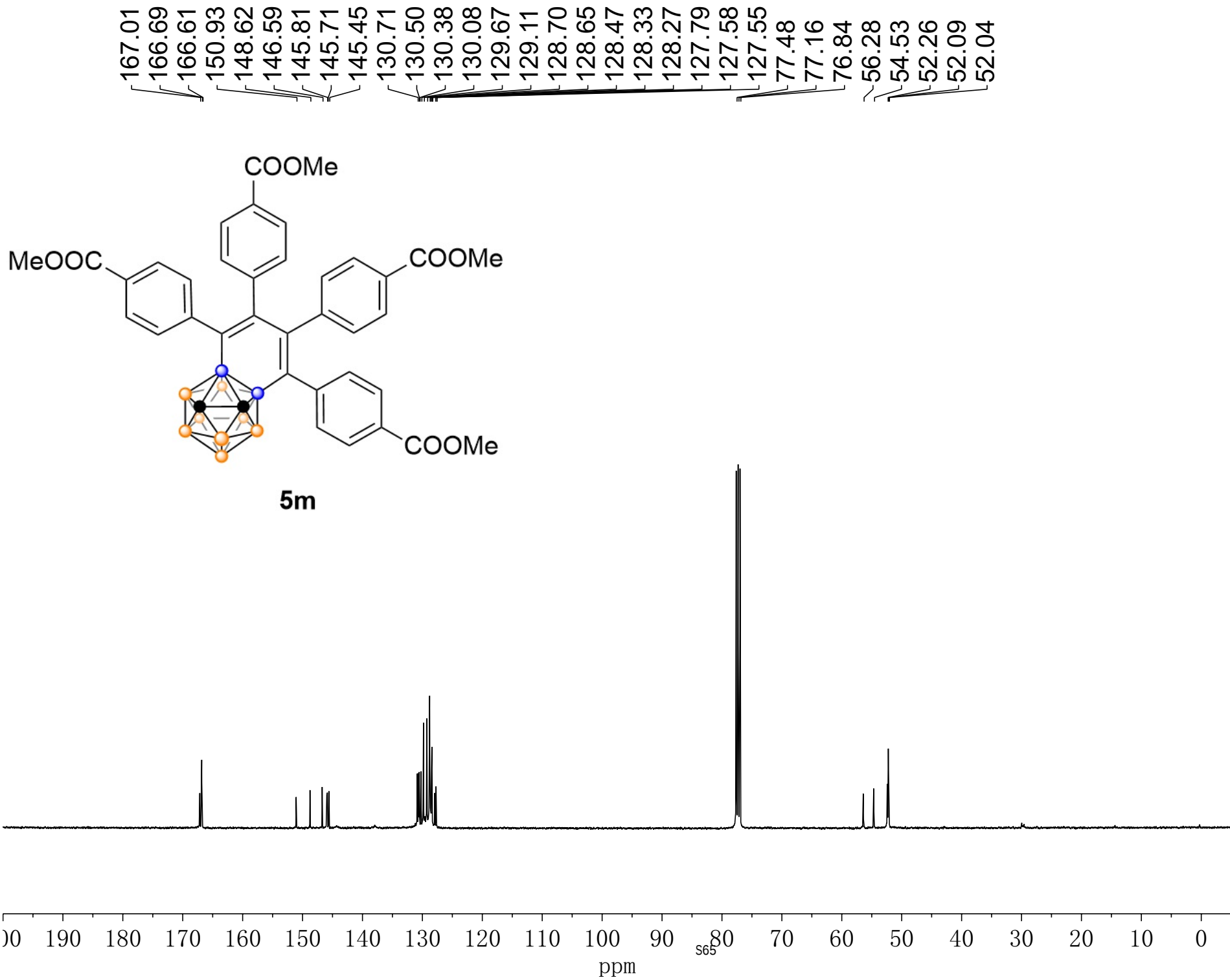
--0.012

Origin	Varian
Spectrometer	nmrs
Solvent	CDCl3
Temperature	21.0
Experiment	1D
Probe	4nuc
Number of Scans	8
Receiver Gain	40
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Spectrometer Frequency	399.72
Spectral Width	7183.9
Lowest Frequency	-794.7
Nucleus	1H
Acquired Size	21552
Spectral Size	65536

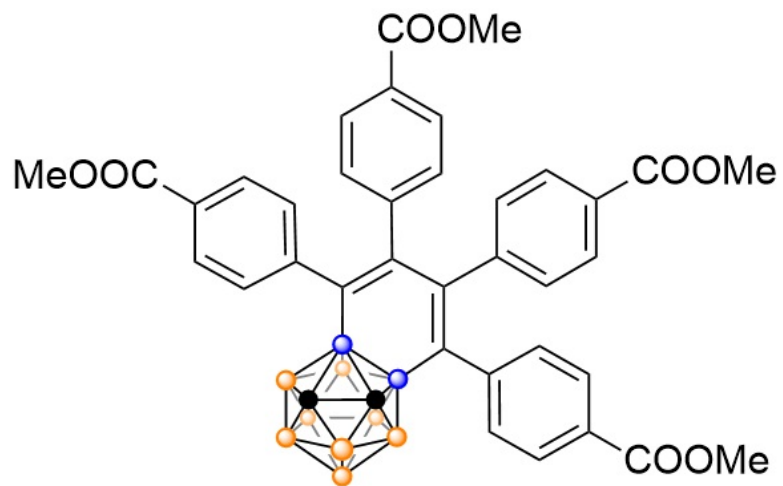


5m





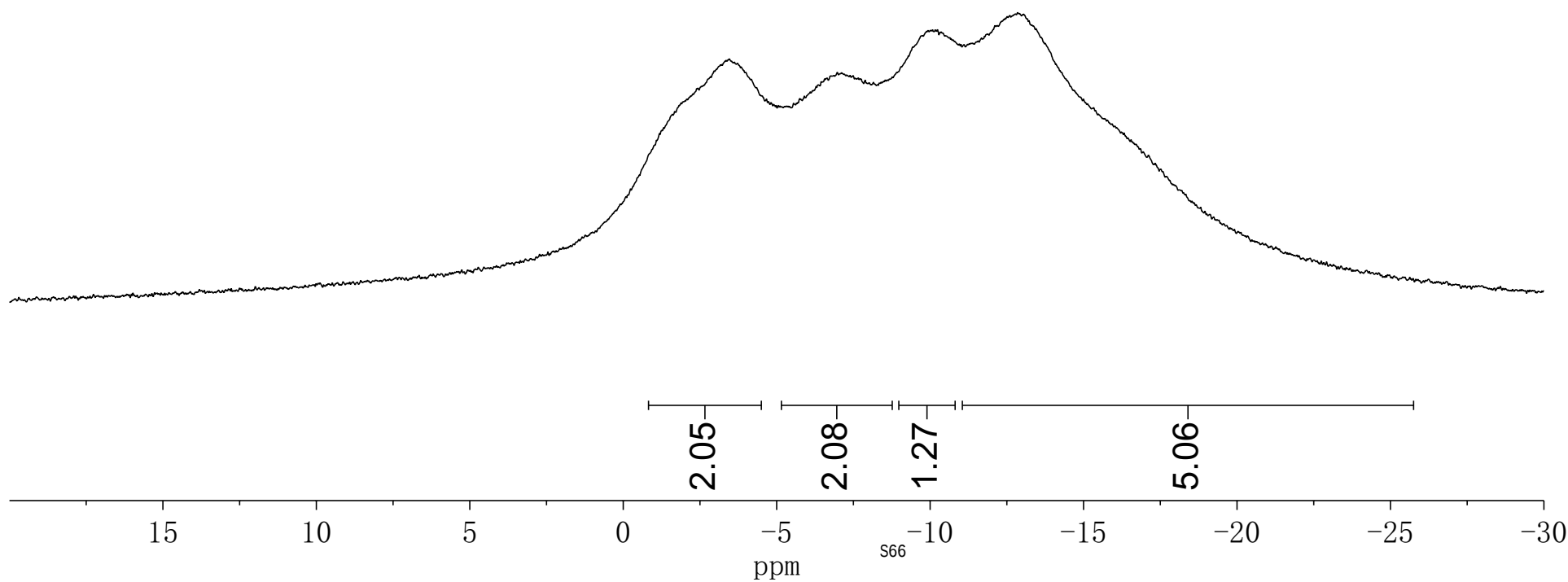
Origin	Varian
Spectrometer	vnmrs
Solvent	CDCl3
Temperature	20.0
Experiment	1D
Probe	AutoSW
Number of Scans	15632
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.3005
Spectrometer Frequency	100.60
Spectral Width	24509.8
Lowest Frequency	-1664.7
Nucleus	13C
Acquired Size	31875
Spectral Size	65536

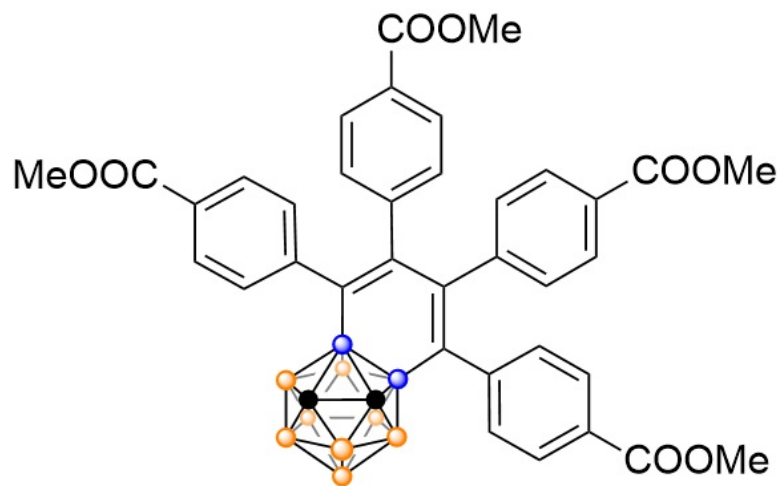


5m

--1.81
--3.57
--6.86
--10.22
--13.41

Solvent	CDC13
Number of Scans	587
Receiver Gain	256
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-12696.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768





5m

--3.09

--6.96

--9.60

--13.05

Solvent	CDC13
Number of Scans	953
Receiver Gain	322
Relaxation Delay	0.0000
Pulse Width	5.0000
Spectrometer Frequency	128.38
Spectral Width	32051.3
Lowest Frequency	-12696.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

