Electronic Supplementary Information

Stereoselective synthesis of cyclopentafullerenes: reaction of [60]fullerene with aldehydes and triethylamine promoted by magnesium perchlorate

Meng Zhang,‡[a] Hong-Yu Zhang,‡[a] Hui-Juan Wang,[b] Fa-Bao Li,*[a] Yongshun Huang,[a] Li Liu,*[a] Chao-Yang Liu,*[b] Abdullah M. Asiri,[c] and Khalid A. Alamry[c]

[a]*Hubei Collaborative Innovation Center for Advanced Organic Chemical Materials, Ministry of Education Key Laboratory for the Synthesis and Application of Organic Functional Molecules, Key Laboratory of Green Preparation and Application for Functional Materials, Ministry of Education, and School of Chemistry and Chemical Engineering, Hubei University, Wuhan 430062, P. R. China*

E-mail: lfb0615@hubu.edu.cn; liulihubei@hubu.edu.cn

[b]*State Key Laboratory of Magnetic Resonance and Atomic and Molecular Physics, Wuhan Center for Magnetic Resonance, Wuhan Institute of Physics and Mathematics, Chinese Academy of Sciences, Wuhan 430071, P. R. China*

E-mail: chyliu@wipm.ac.cn

[c]*Department of Chemistry, Faculty of Science, King Abdulaziz University, Jeddah 21589, Saudi Arabia*

‡*These authors contributed equally to this work.*

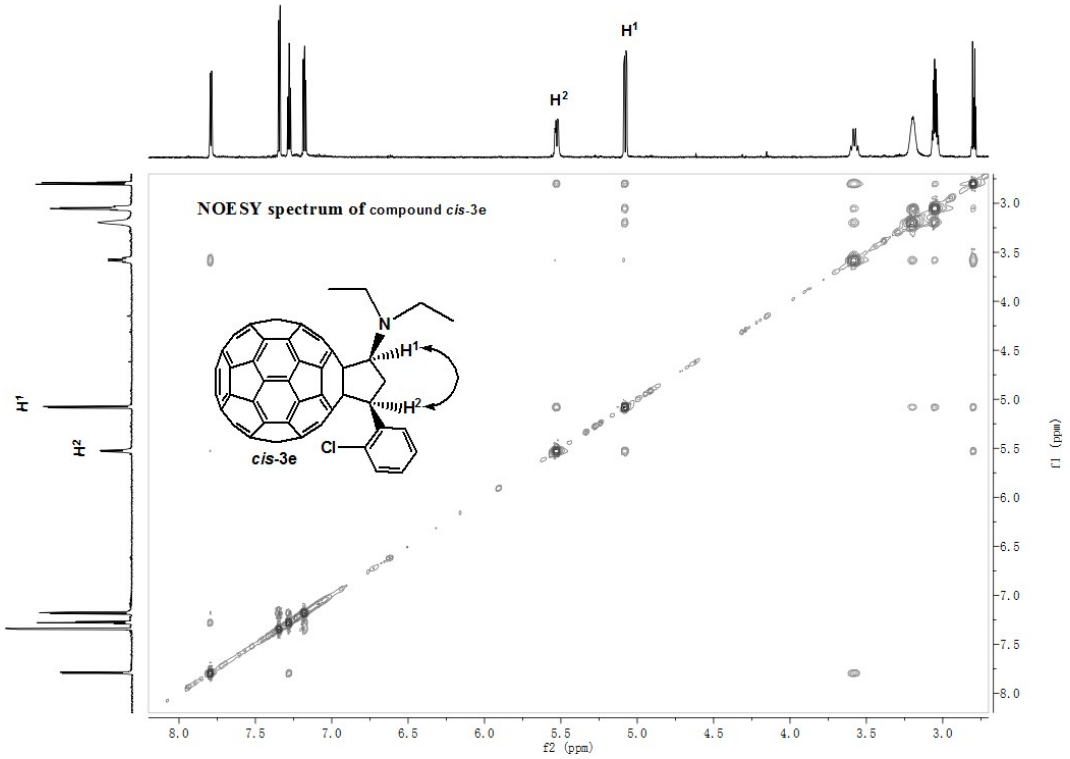


Figure S1. NOESY (800 MHz, CS_2/DMSO-d_6) spectrum of *cis*-**3e**, and the nuclear Overhauser effect between the two methine protons is indicated by the curved arrow.

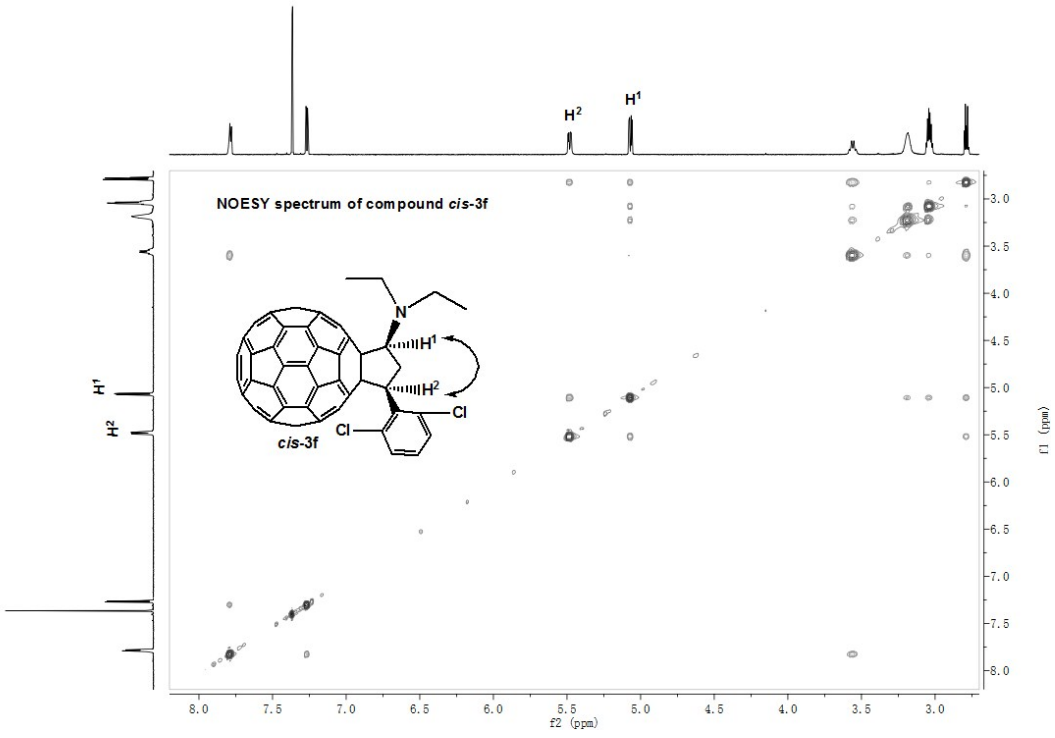
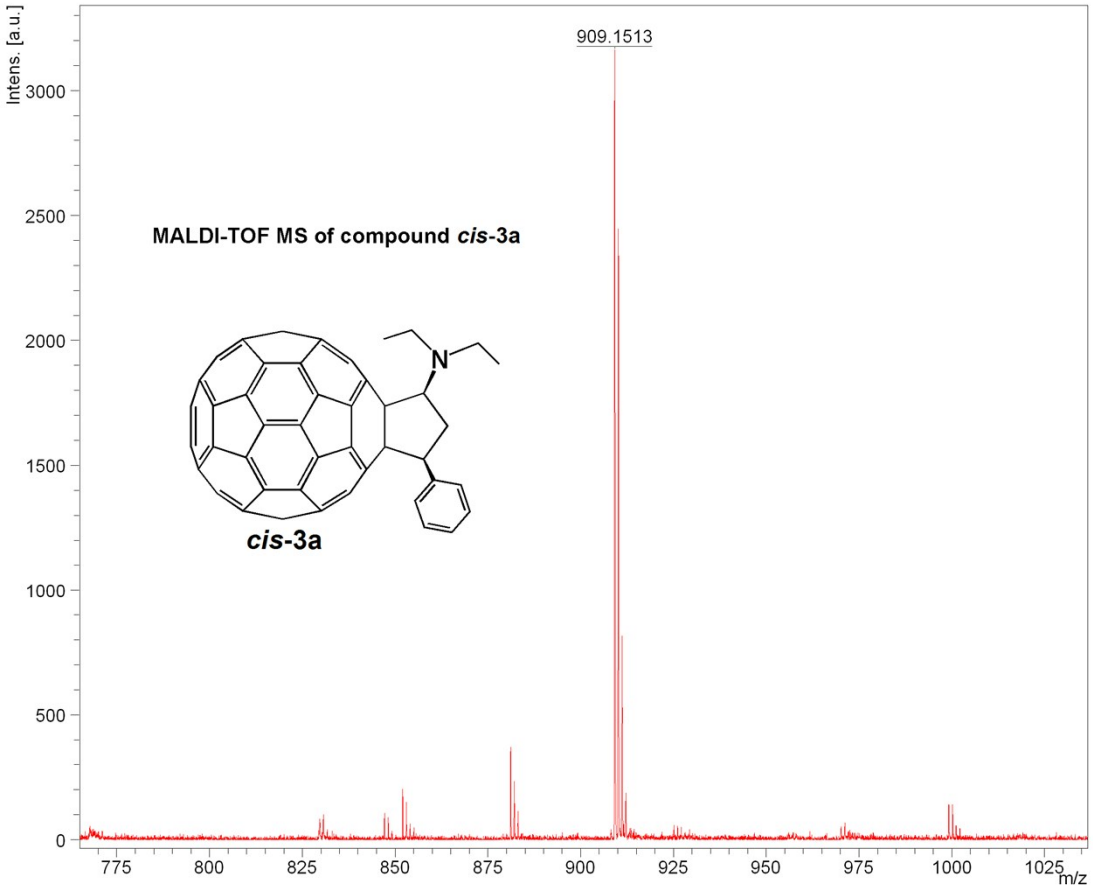
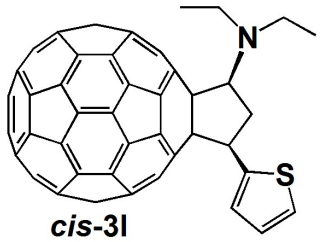


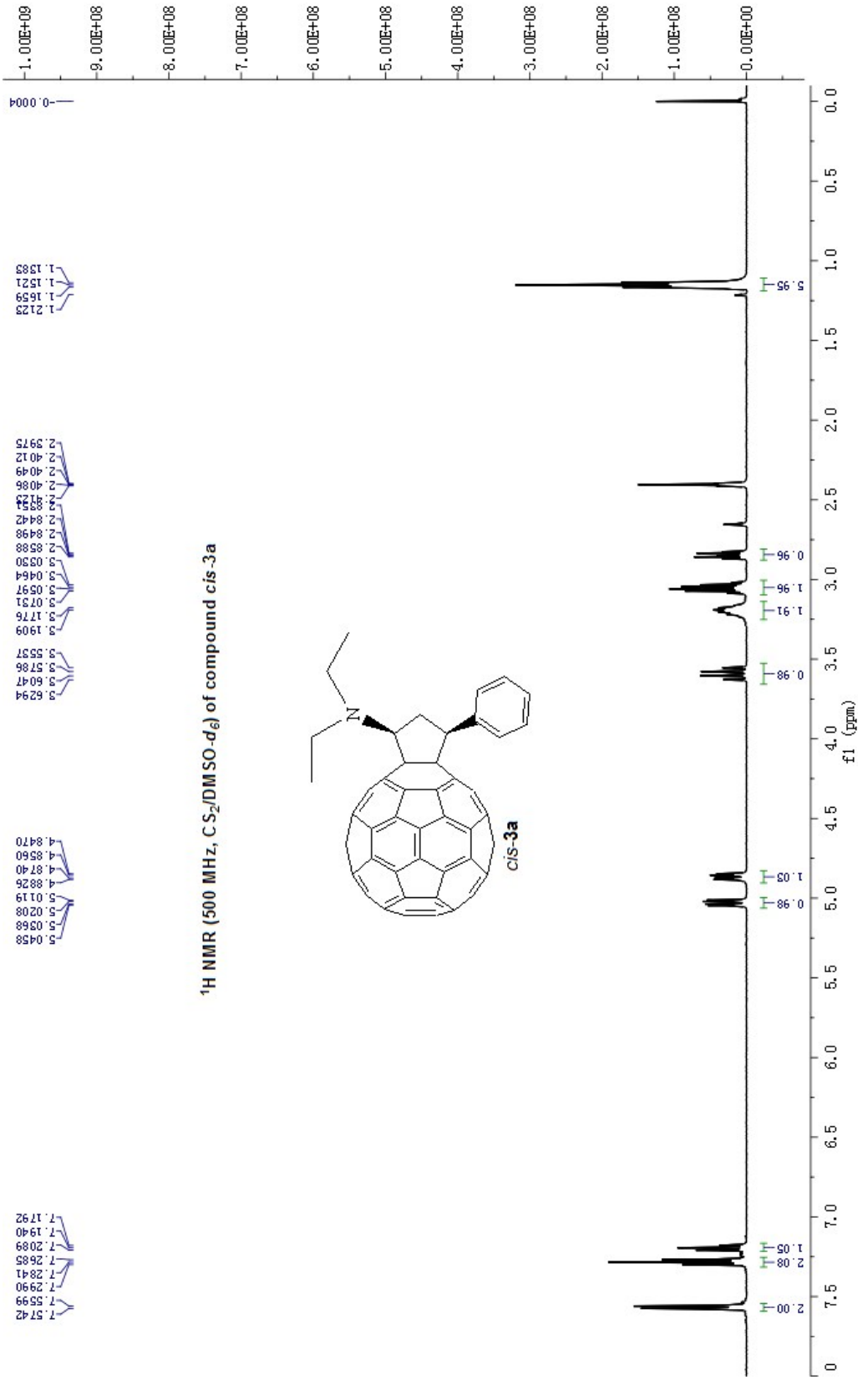
Figure S2. NOESY (800 MHz, CS_2/DMSO-d_6) spectrum of *cis*-**3f**, and the nuclear Overhauser effect between the two methine protons is indicated by the curved arrow.

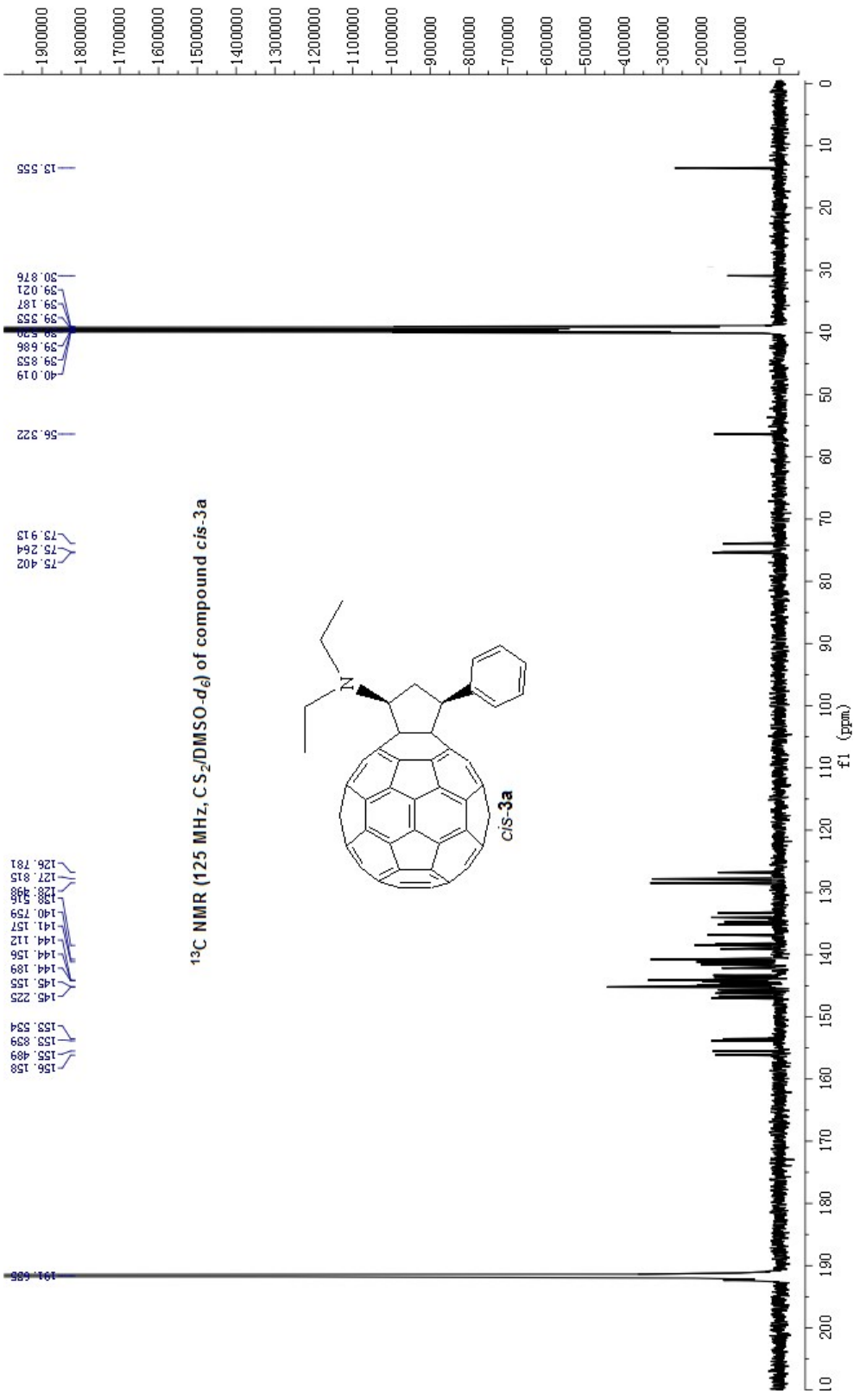

909.1513
MALDI-TOF MS of compound *cis*-3a
N
cis-3a
Intens. [a.u.]
3000
2500
2000
1500
1000
500
0
775
800
825
850
875
900
925
950
975
1000
1025
m/z

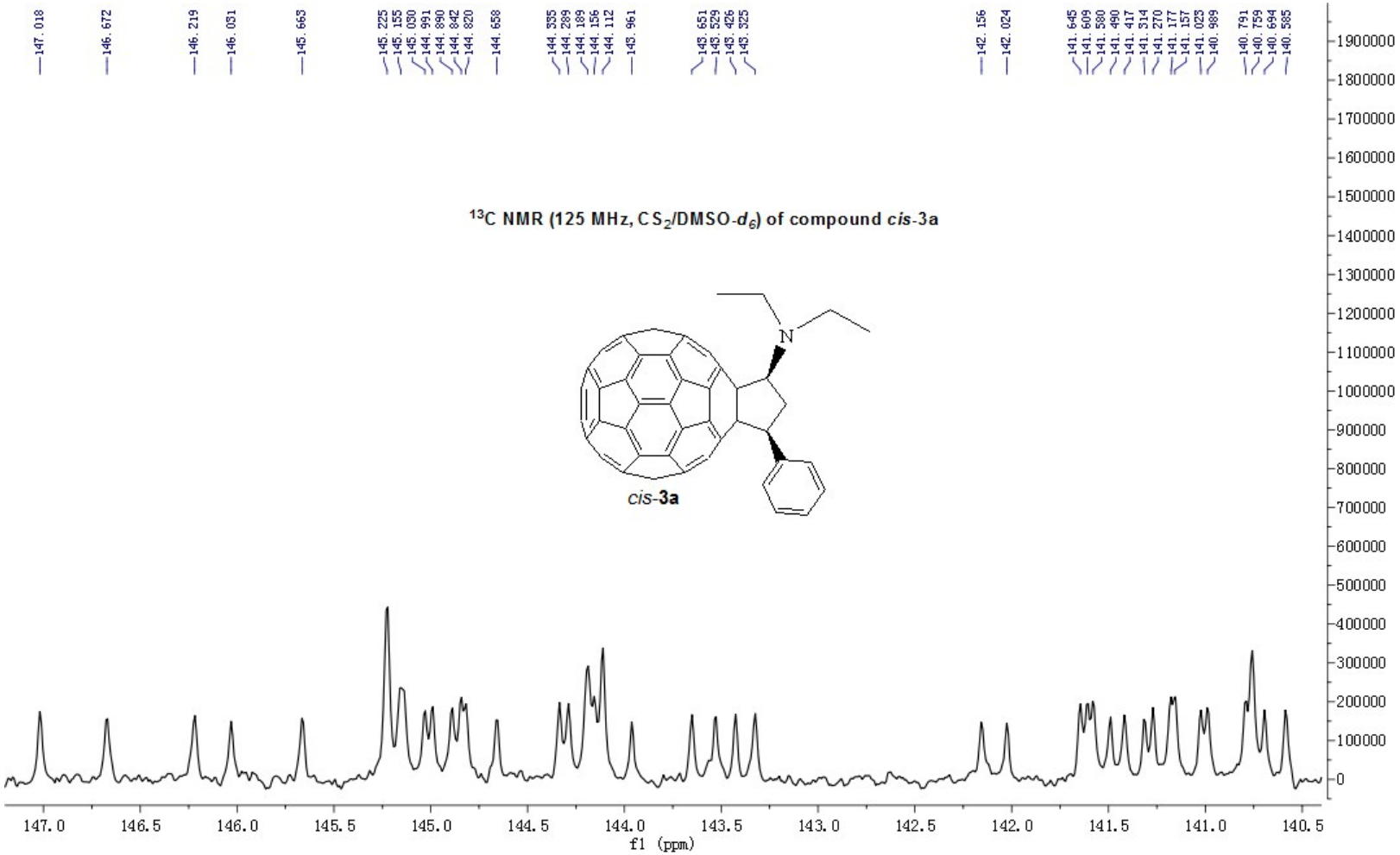
UV-vis spectrum of compound *cis*-3l in $CHCl_3$



***cis*-3l**

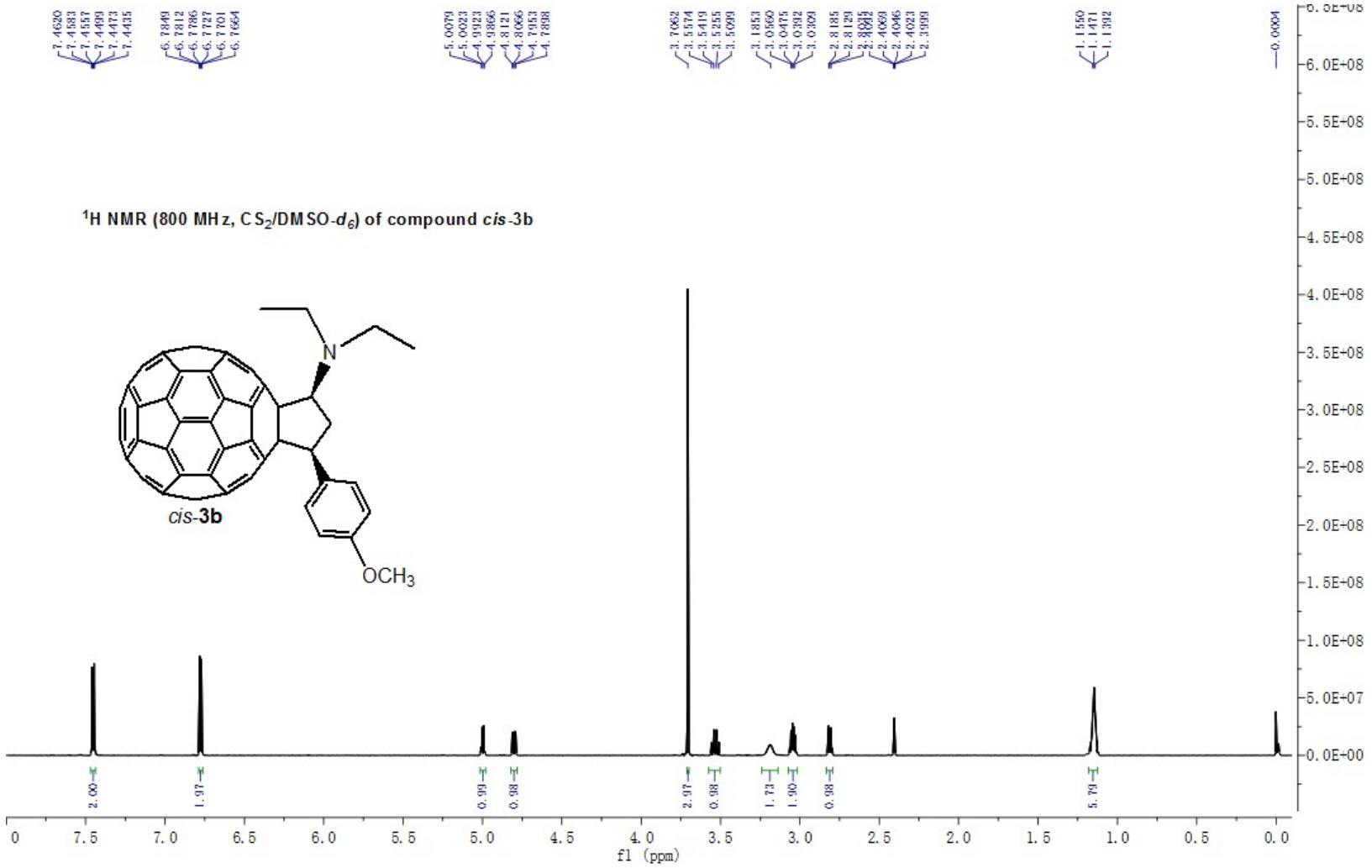

1H NMR (500 MHz, CS2/DMSO-d6) of compound cis-3a
cis-3a
f1 (ppm)


13.555
30.876
39.021
39.187
39.353
39.520
39.686
39.853
40.019
56.322
73.913
75.264
75.402
126.781
127.815
128.498
138.516
140.759
141.157
144.112
144.156
144.189
145.155
145.225
153.534
153.839
155.489
156.158
191.685
^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *cis*-3a
cis-3a
f1 (ppm)



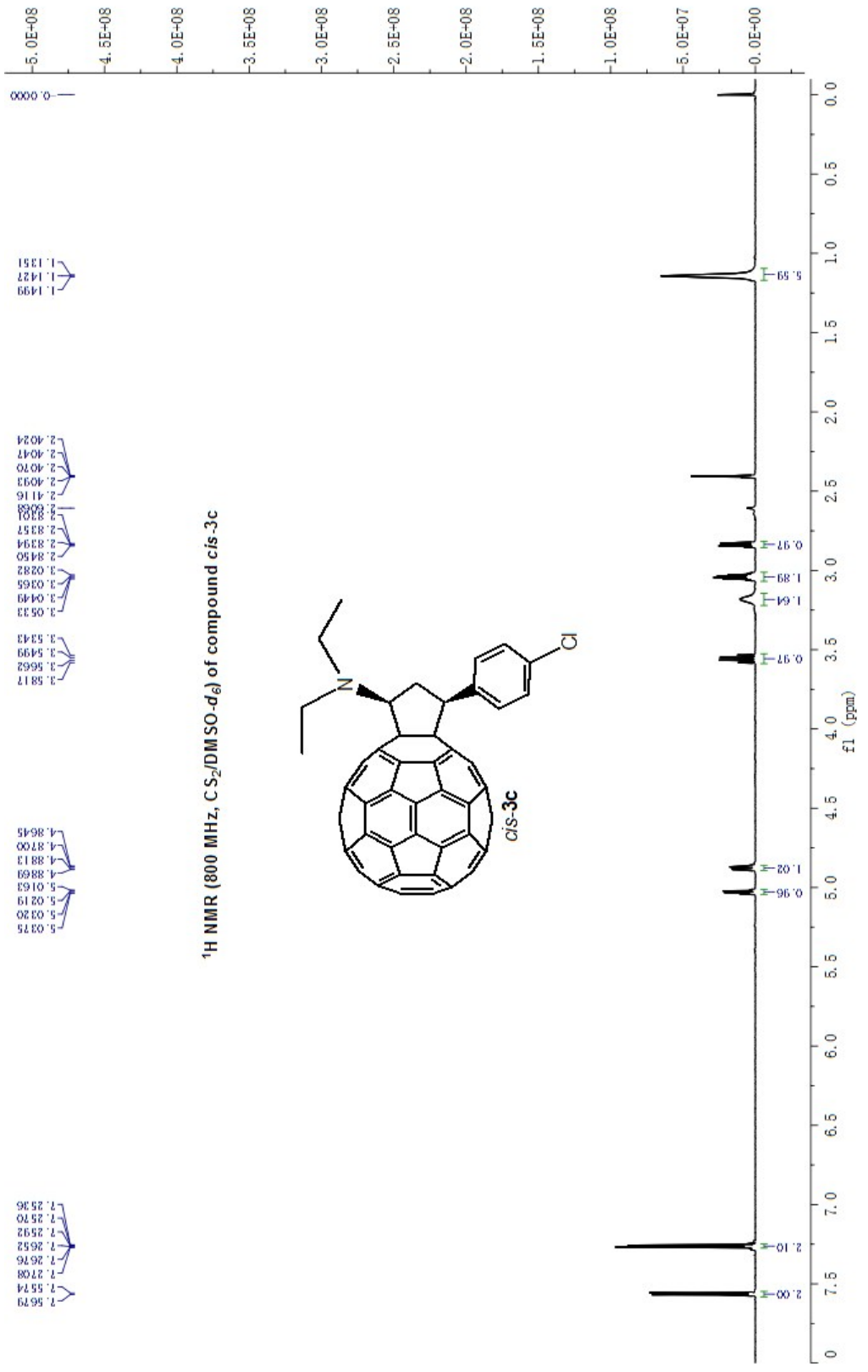
^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *cis*-3a

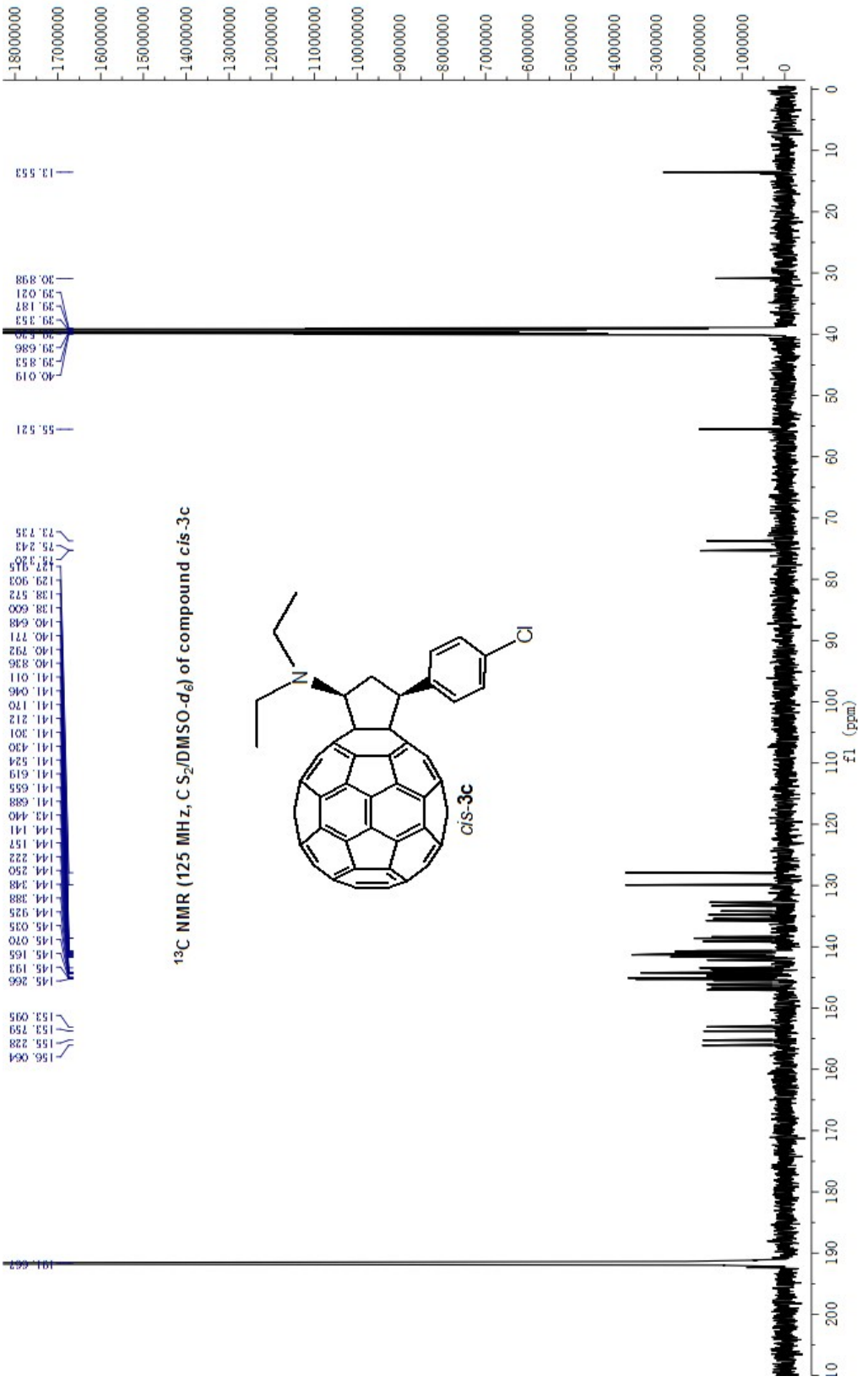
cis-**3a**

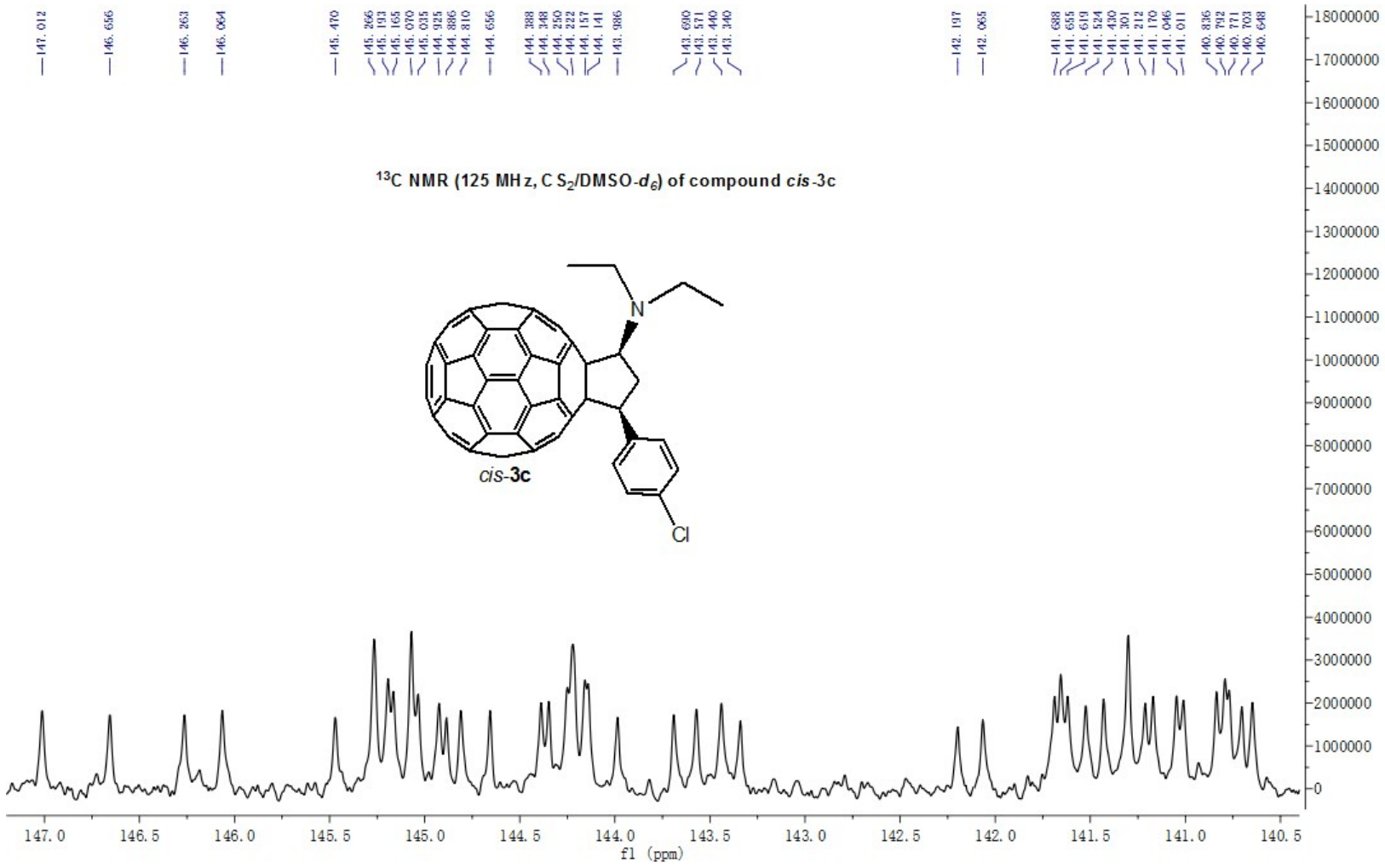


^{1}H NMR (800 MHz, CS_2/DMSO-d_6) of compound *cis*-3b

cis-**3b**

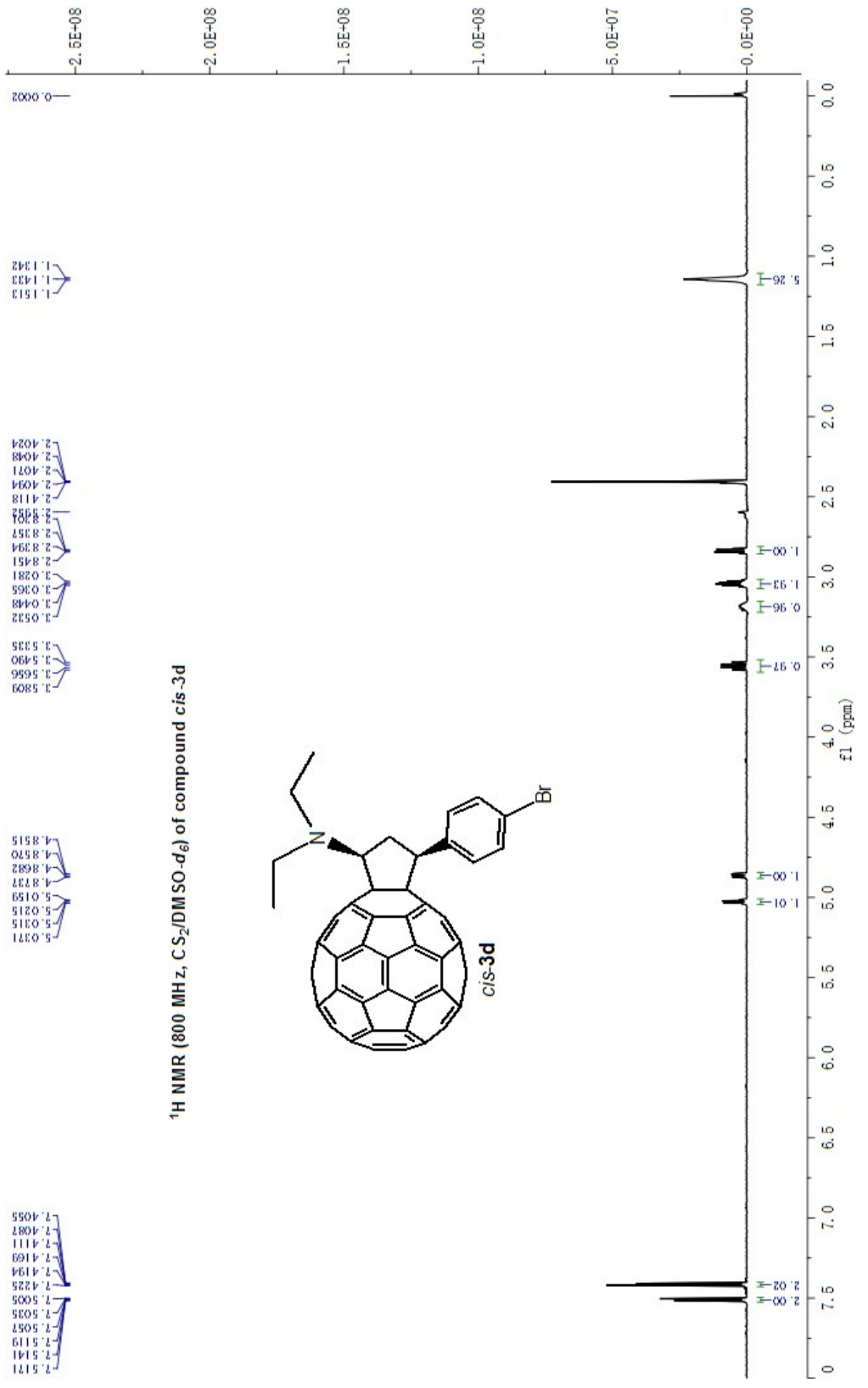

^{1}H NMR (800 MHz, CS_2/DMSO-d_6) of compound *cis*-3c
cis-3c
Cl
N
f1 (ppm)

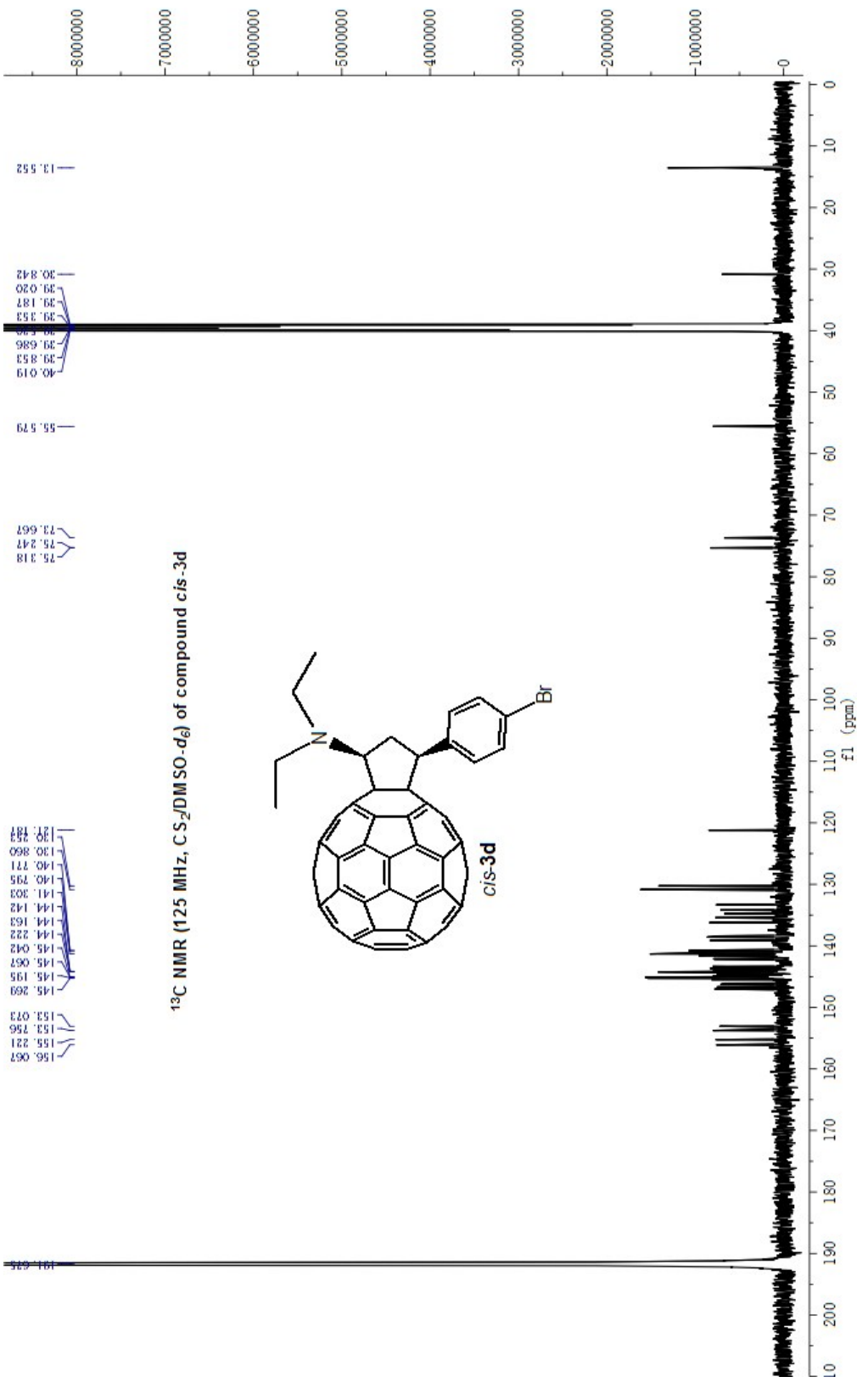

^{13}C NMR (125 MHz, C S_2/DMSO-d_6) of compound *cis*-3c
cis-3c
Cl
N
f1 (ppm)

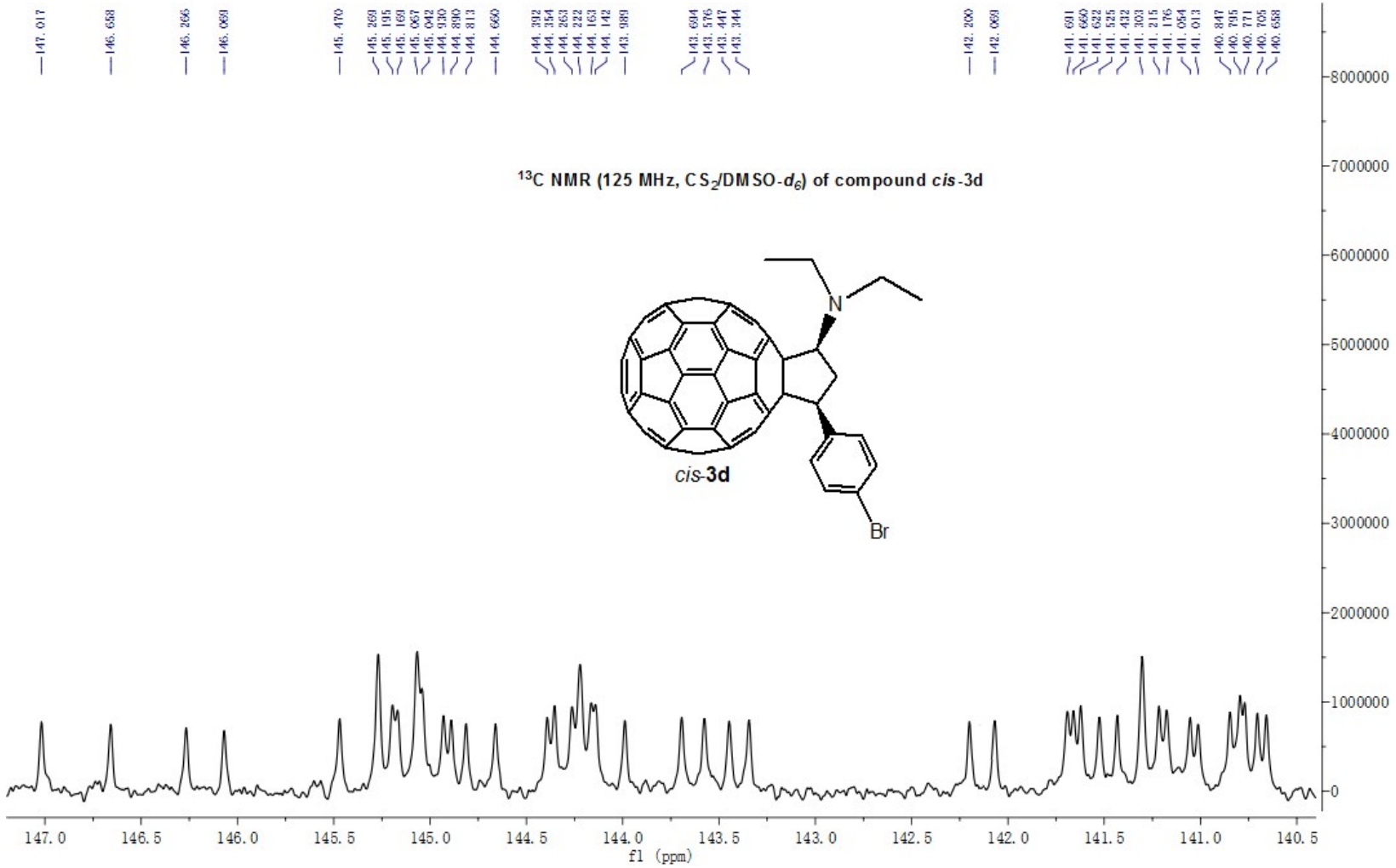


^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *cis*-3c

cis-**3c**


¹H NMR (800 MHz, CS2/DMSO-d6) of compound cis-3d
cis-3d
Br
N
f1 (ppm)


13.552
30.842
39.020
39.187
39.353
39.520
39.686
39.853
40.019
55.579
73.667
75.247
75.318
127.187
130.253
130.860
140.771
140.795
141.303
144.142
144.163
144.222
145.042
145.067
145.195
145.269
153.073
153.756
155.221
156.067
191.676
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3d
N
Br
cis-3d
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
f1 (ppm)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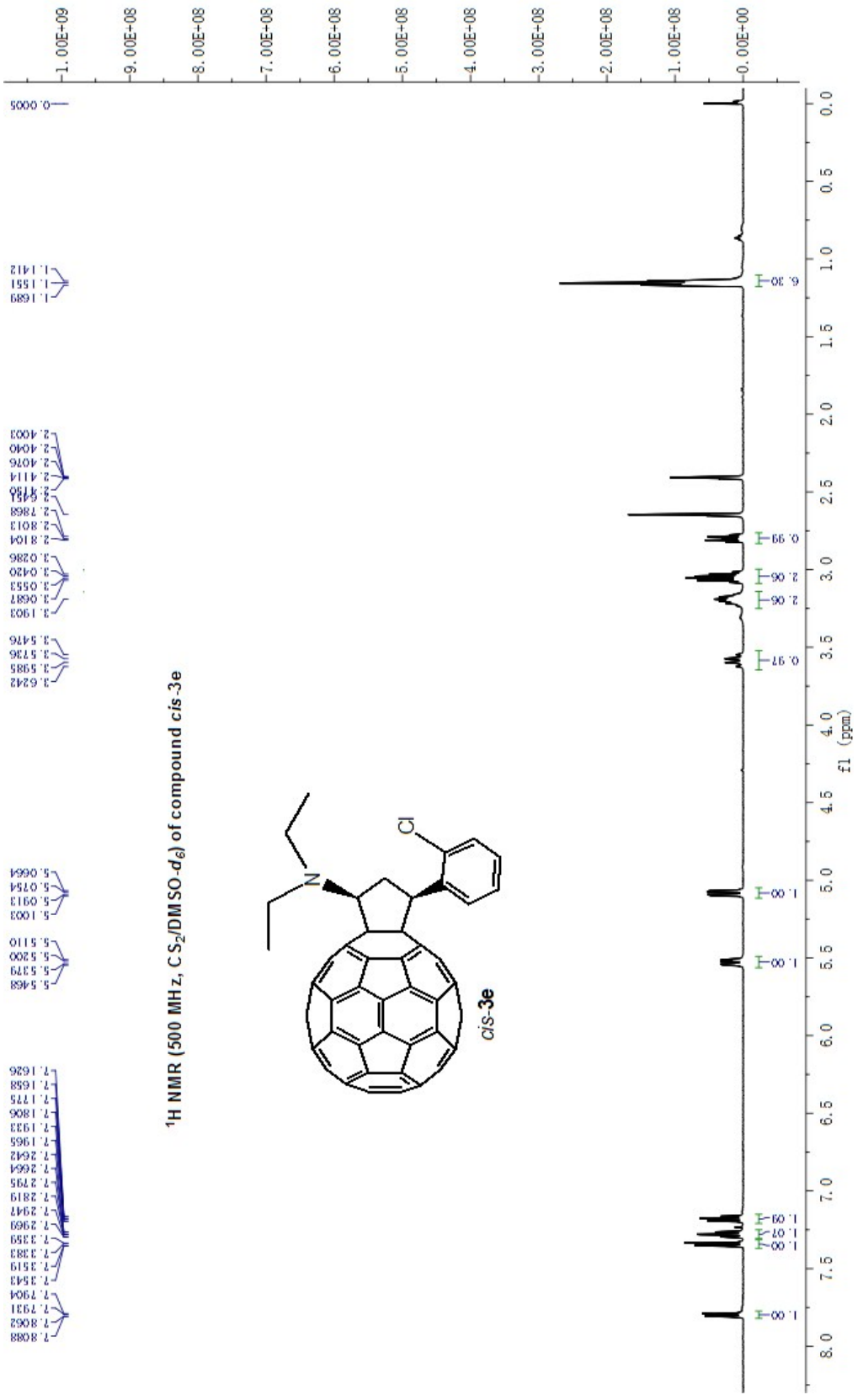
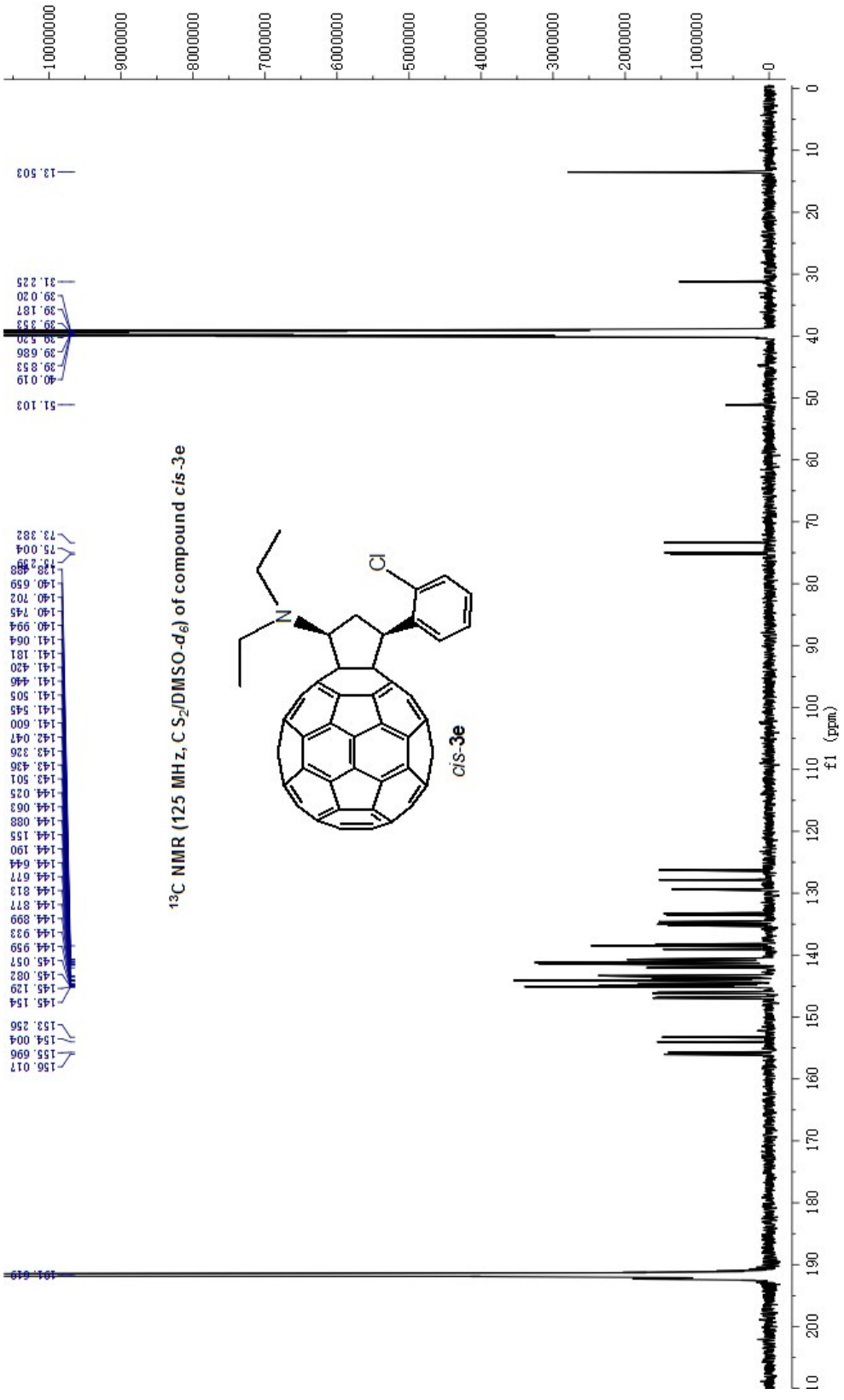
146.658
146.266
146.069
145.470
145.269
145.195
145.169
145.067
145.042
144.930
144.890
144.813
144.660
144.392
144.354
144.263
144.222
144.163
144.142
143.989
143.694
143.576
143.447
143.344
142.200
142.069
141.691
141.660
141.622
141.525
141.432
141.303
141.215
141.176
141.054
141.013
140.847
140.795
140.771
140.705
140.658
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3d
N
Br
cis-3d
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
147.0
146.5
146.0
145.5
145.0
144.5
144.0
143.5
143.0
142.5
142.0
141.5
141.0
140.5
f1 (ppm)


1H NMR (500 MHz, CS2/DMSO-d6) of compound cis-3e
cis-3e
Cl
N
f1 (ppm)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31.225
39.020
39.187
39.353
39.520
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39.853
40.019
51.103
73.382
75.004
128.488
140.659
140.702
140.745
140.994
141.064
141.181
141.420
141.446
141.505
141.545
141.600
142.047
143.326
143.436
143.501
144.025
144.063
144.088
144.155
144.190
144.644
144.677
144.813
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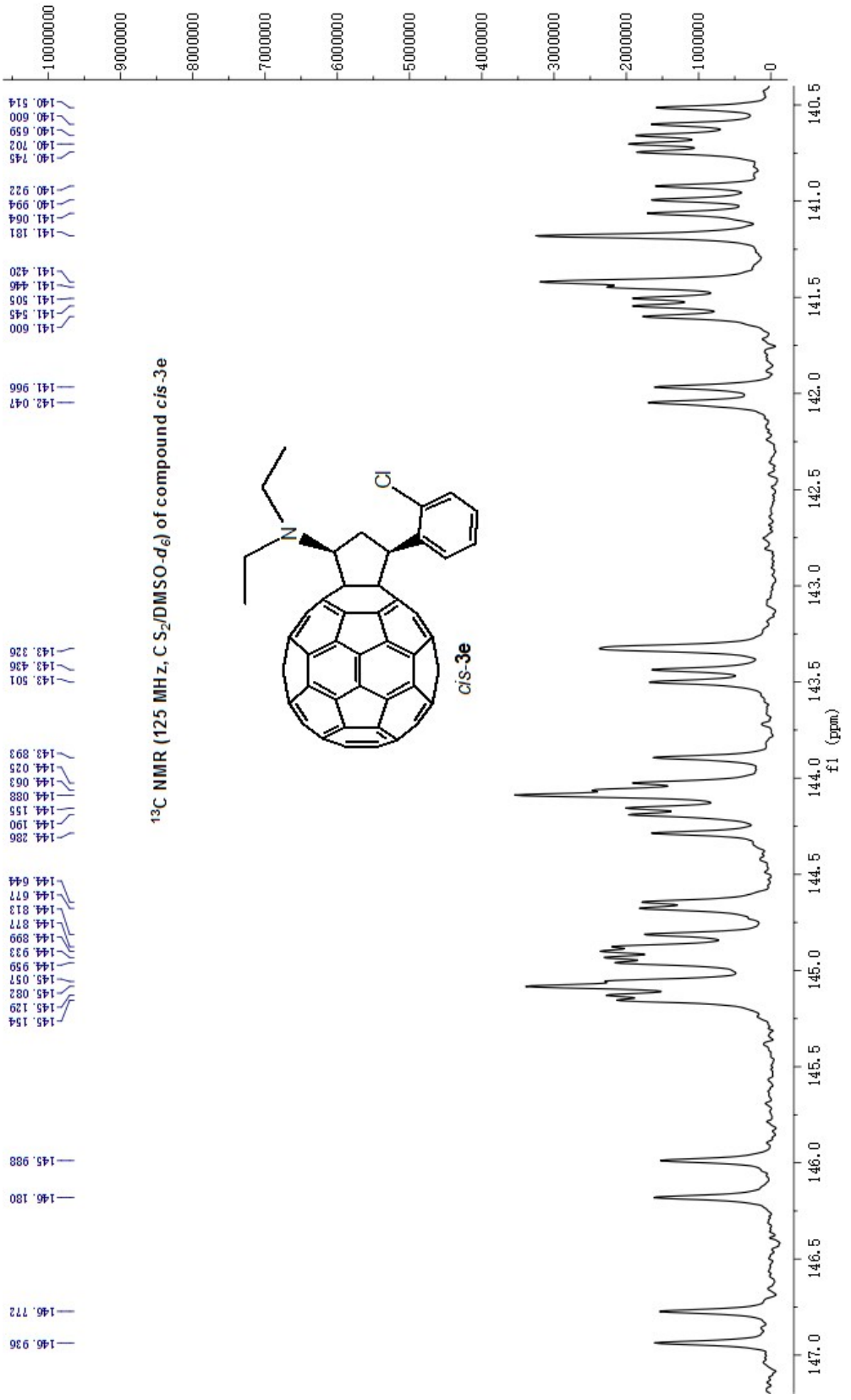
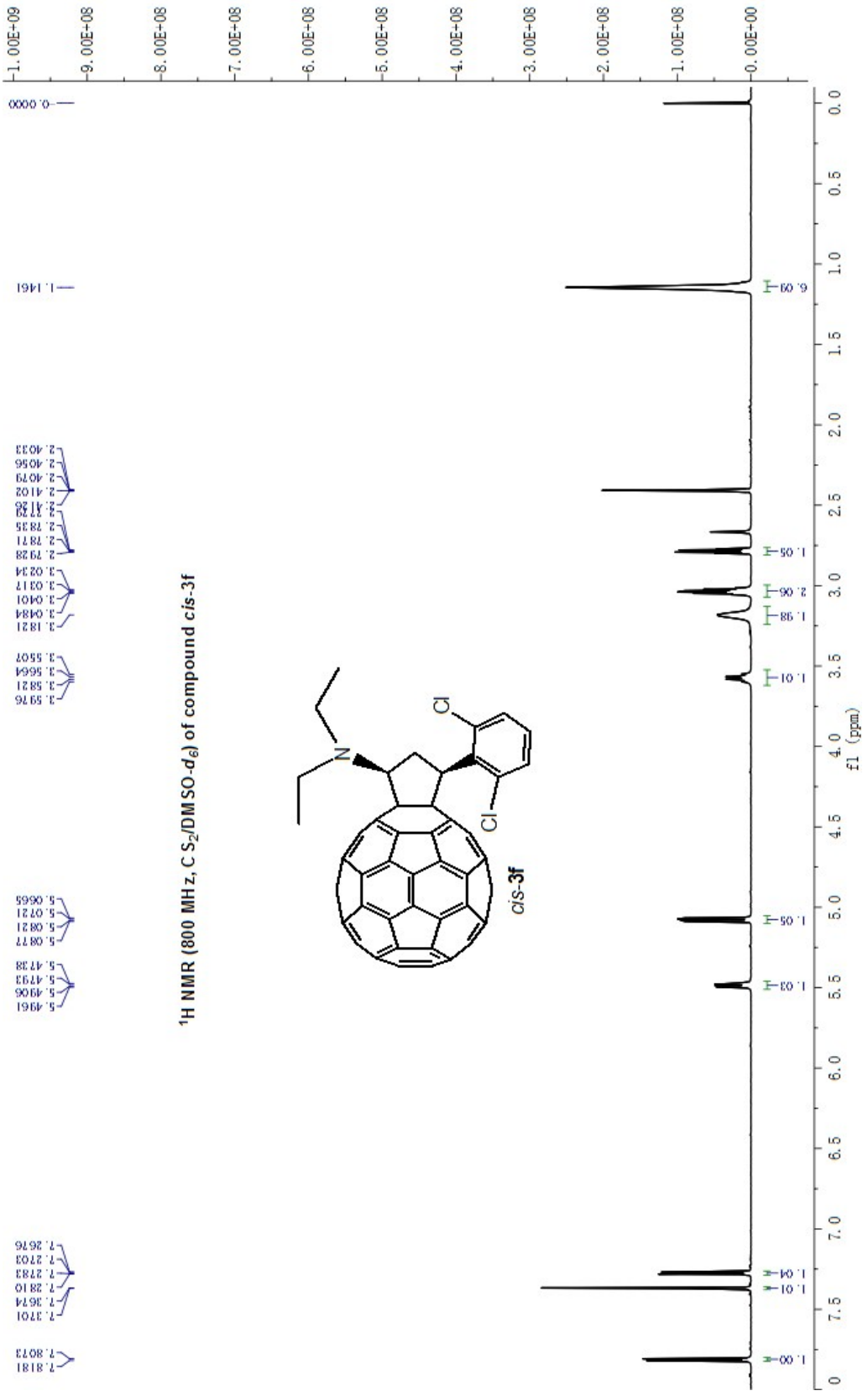
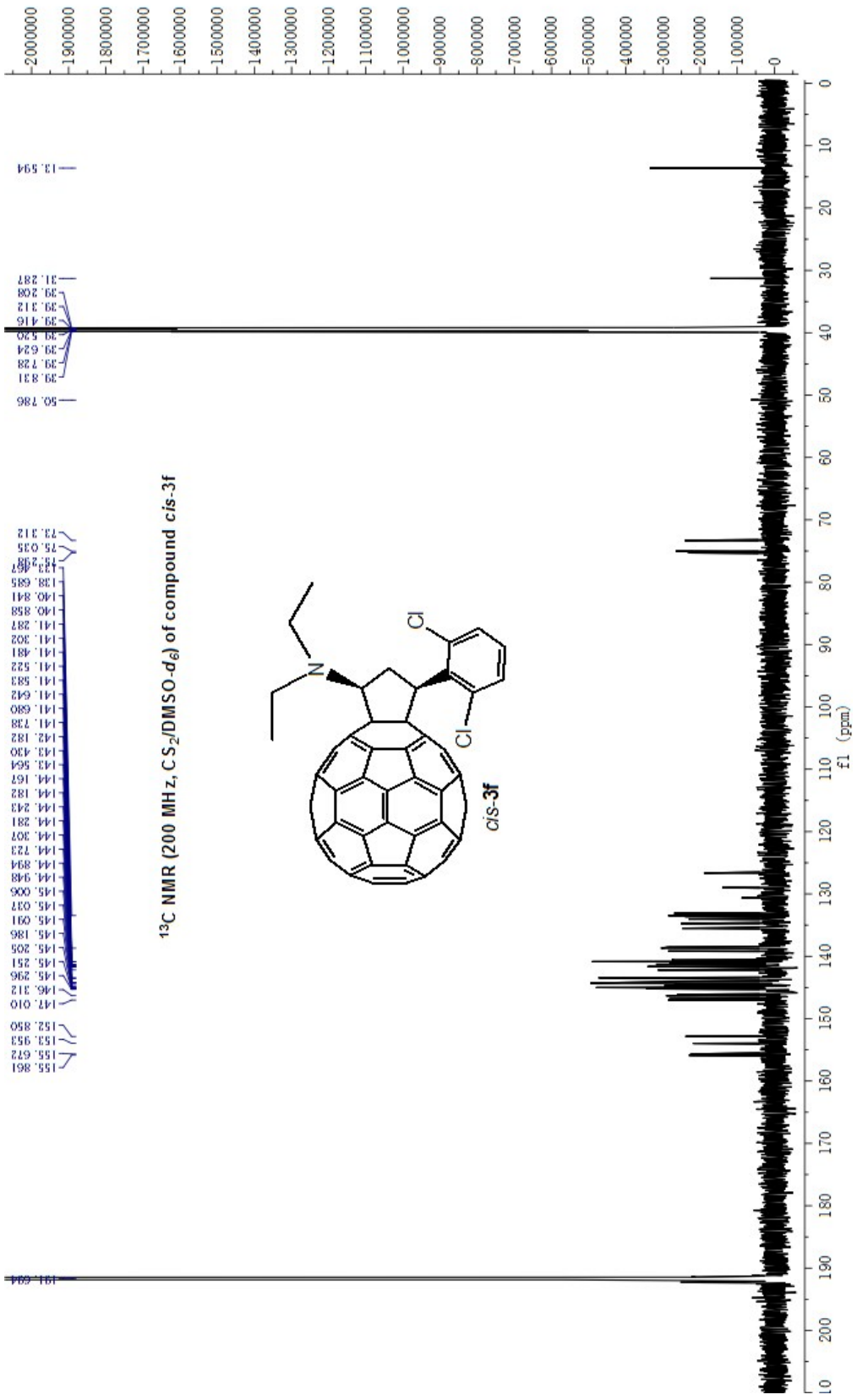
144.899
144.933
144.959
145.057
145.082
145.129
145.154
153.256
154.004
155.696
156.017
191.619
13C NMR (125 MHz, C S2/DMSO-d6) of compound cis-3e
N
Cl
cis-3e
f1 (ppm)

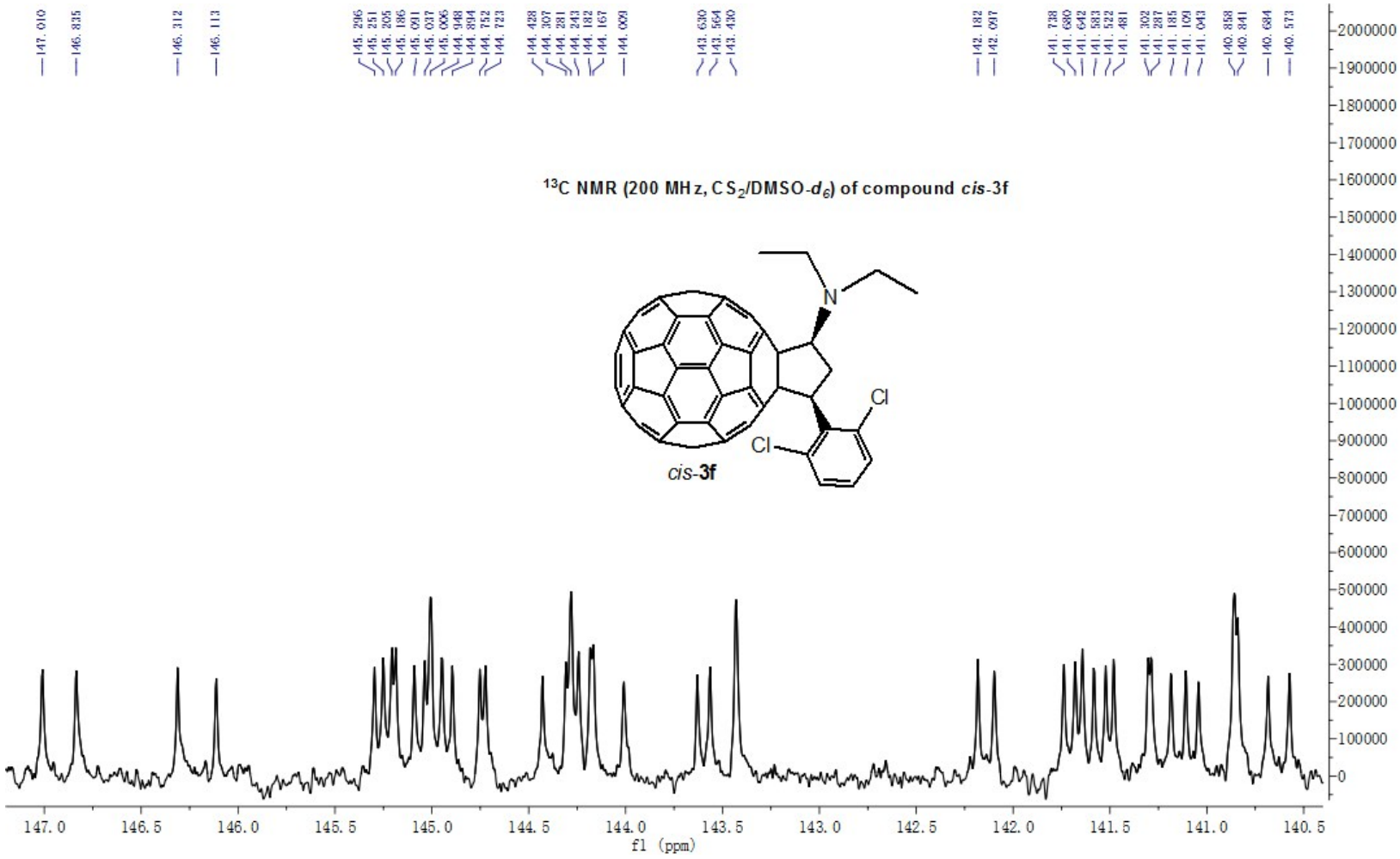

13C NMR (125 MHz, C S2/DMSO-d6) of compound cis-3e
Cl
N
cis-3e
f1 (ppm)


^{1}H NMR (800 MHz, C S_2/DMSO-d_6) of compound *cis*-3f
Cl
Cl
N
cis-3f
f1 (ppm)



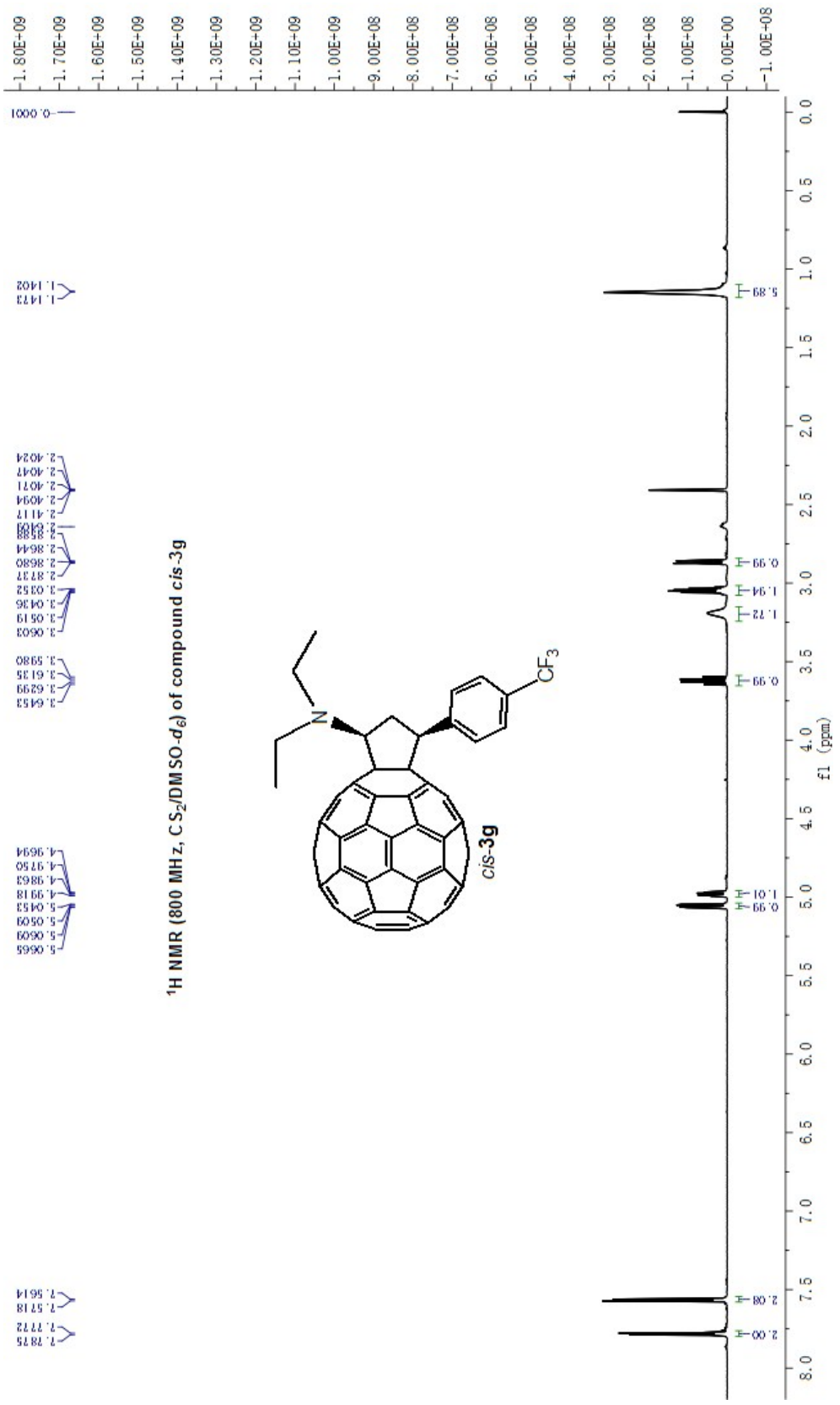
^{13}C NMR (200 MHz, CS_2/DMSO-d_6) of compound *cis*-3f

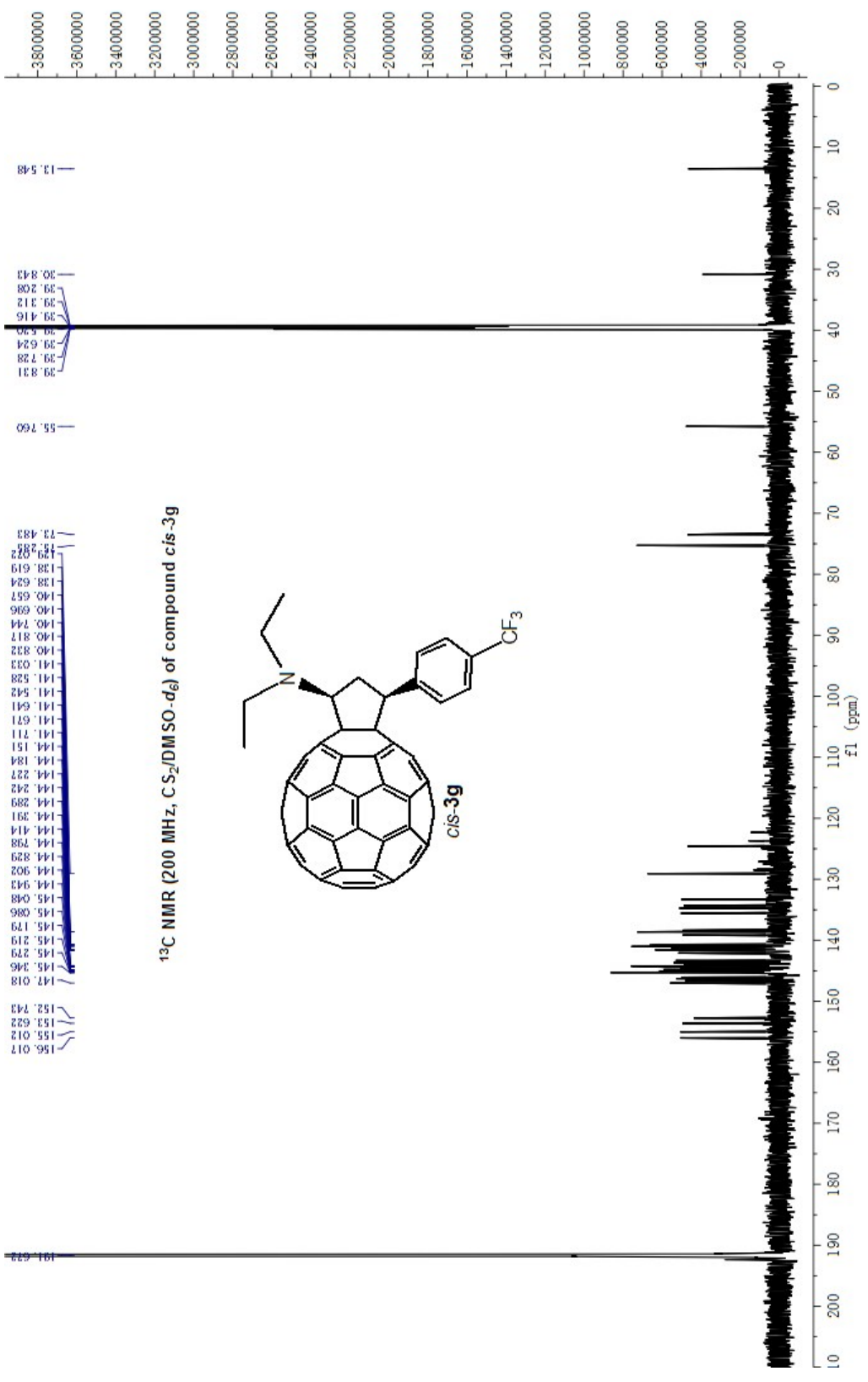
cis-**3f**



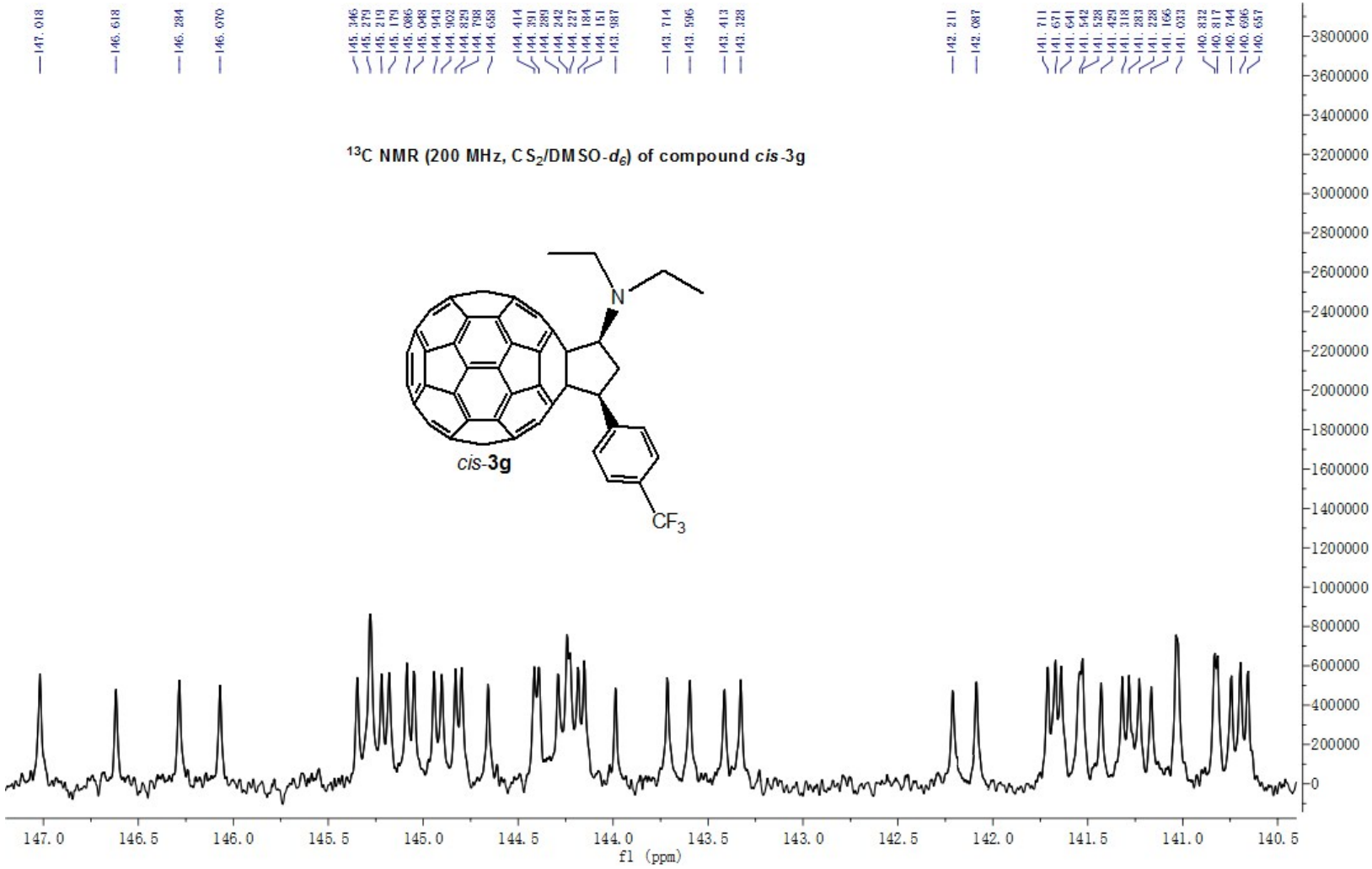
^{13}C NMR (200 MHz, CS_2/DMSO-d_6) of compound *cis*-3f

cis-**3f**


1H NMR (800 MHz, CS2/DMSO-d6) of compound cis-3g
cis-3g
CF3
N
f1 (ppm)

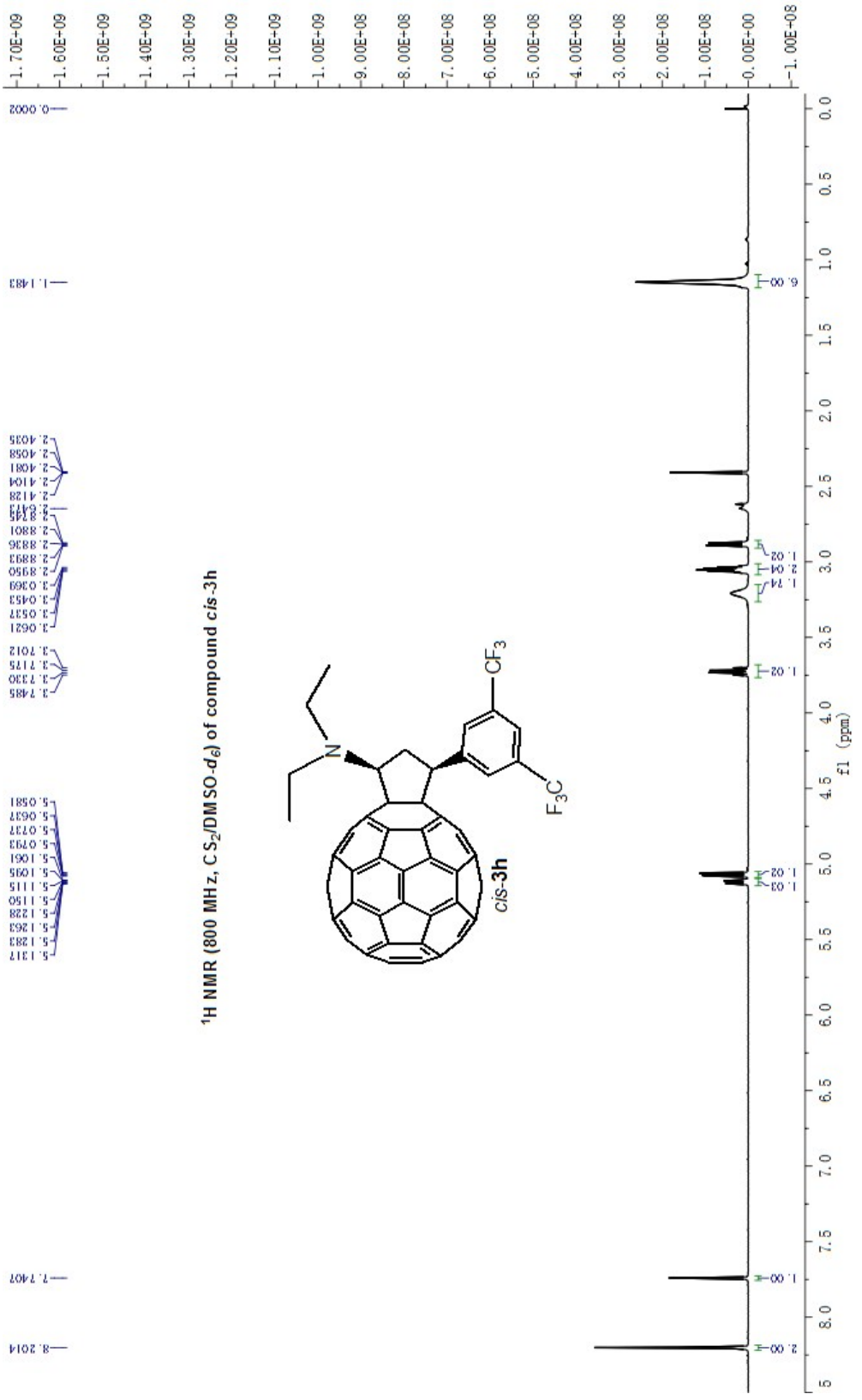


^{13}C NMR (200 MHz, CS_2/DMSO-d_6) of compound *cis*-3g



^{13}C NMR (200 MHz, CS_2/DMSO-d_6) of compound *cis*-3g

cis-**3g**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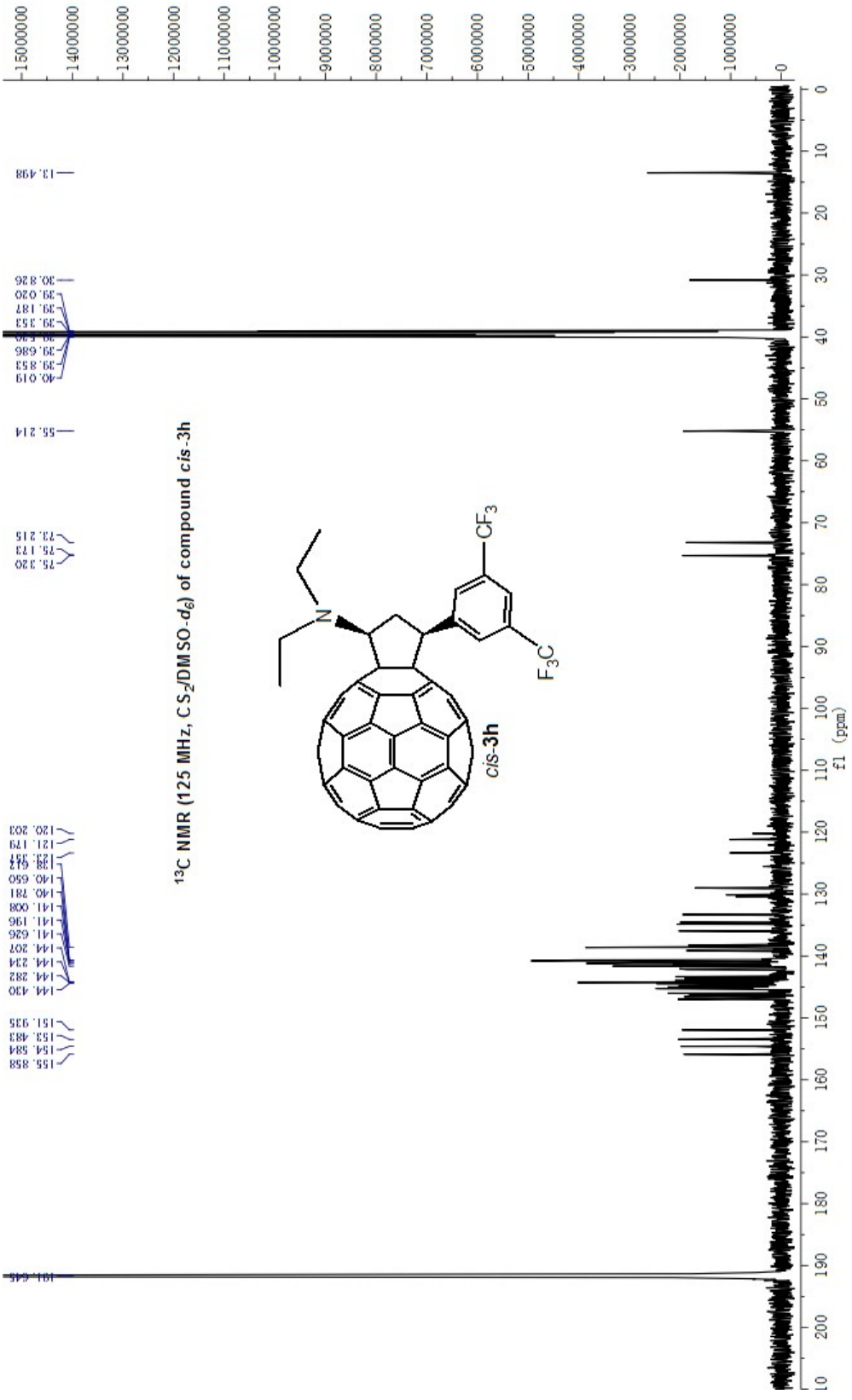
cis-3h
CF_3
F_3C
N
f1 (ppm)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30.826
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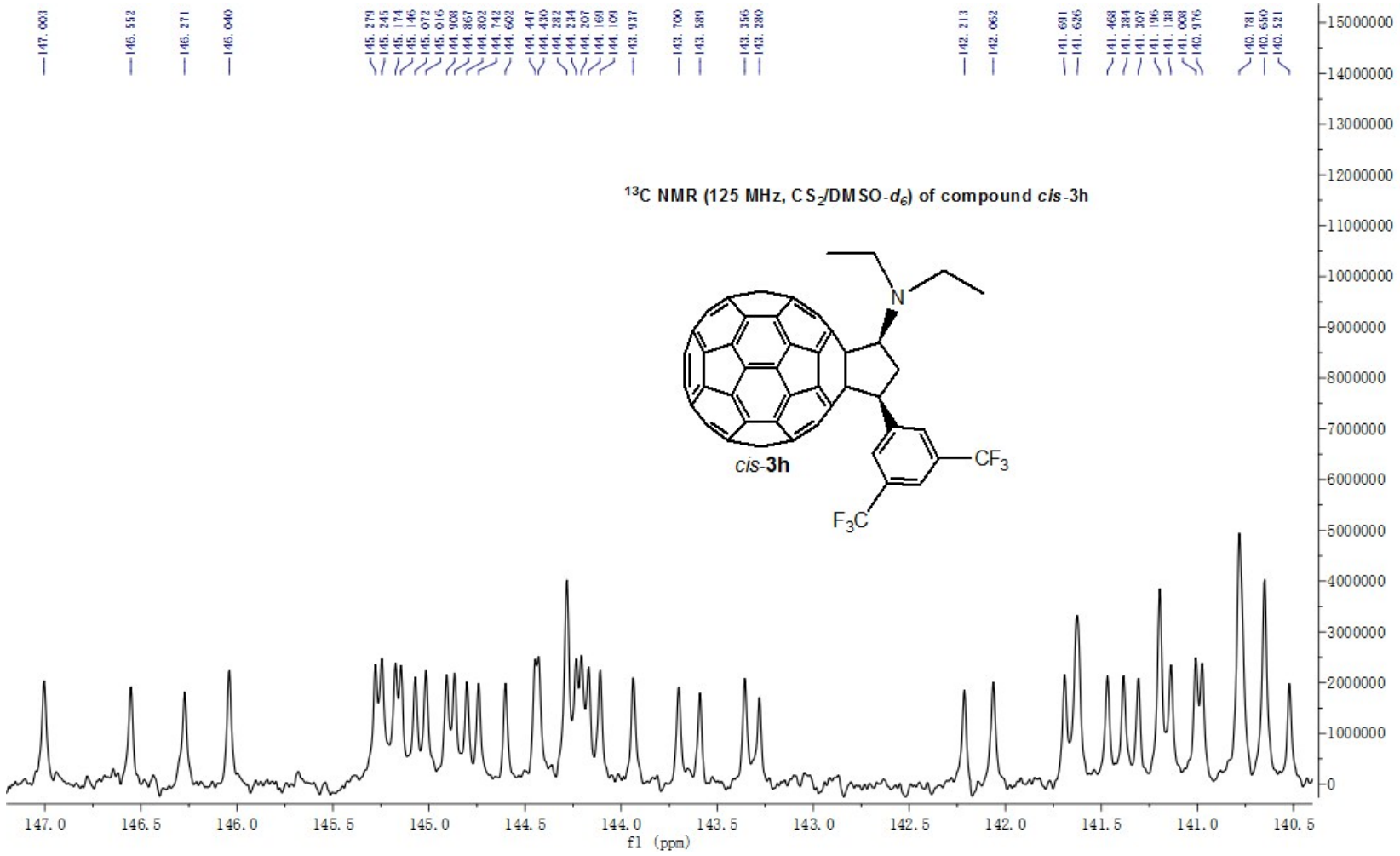
75.320
120.203
121.179
128.617
128.947
140.650
140.781
141.008
141.196
141.626
144.207
144.234
144.282
144.430
151.935
153.483
154.584
155.858
191.646
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3h
N
CF3
F3C
cis-3h
f1 (ppm)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145.146
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144.802
144.742
144.602
144.447
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144.282
144.234
144.207
144.169
144.109
143.937
143.700
143.589
143.356
143.280
142.213
142.062
141.691
141.626
141.468
141.384
141.307
141.196
141.138
141.008
140.976
140.781
140.650
140.521
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3h
N
CF3
F3C
cis-3h
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
147.0
146.5
146.0
145.5
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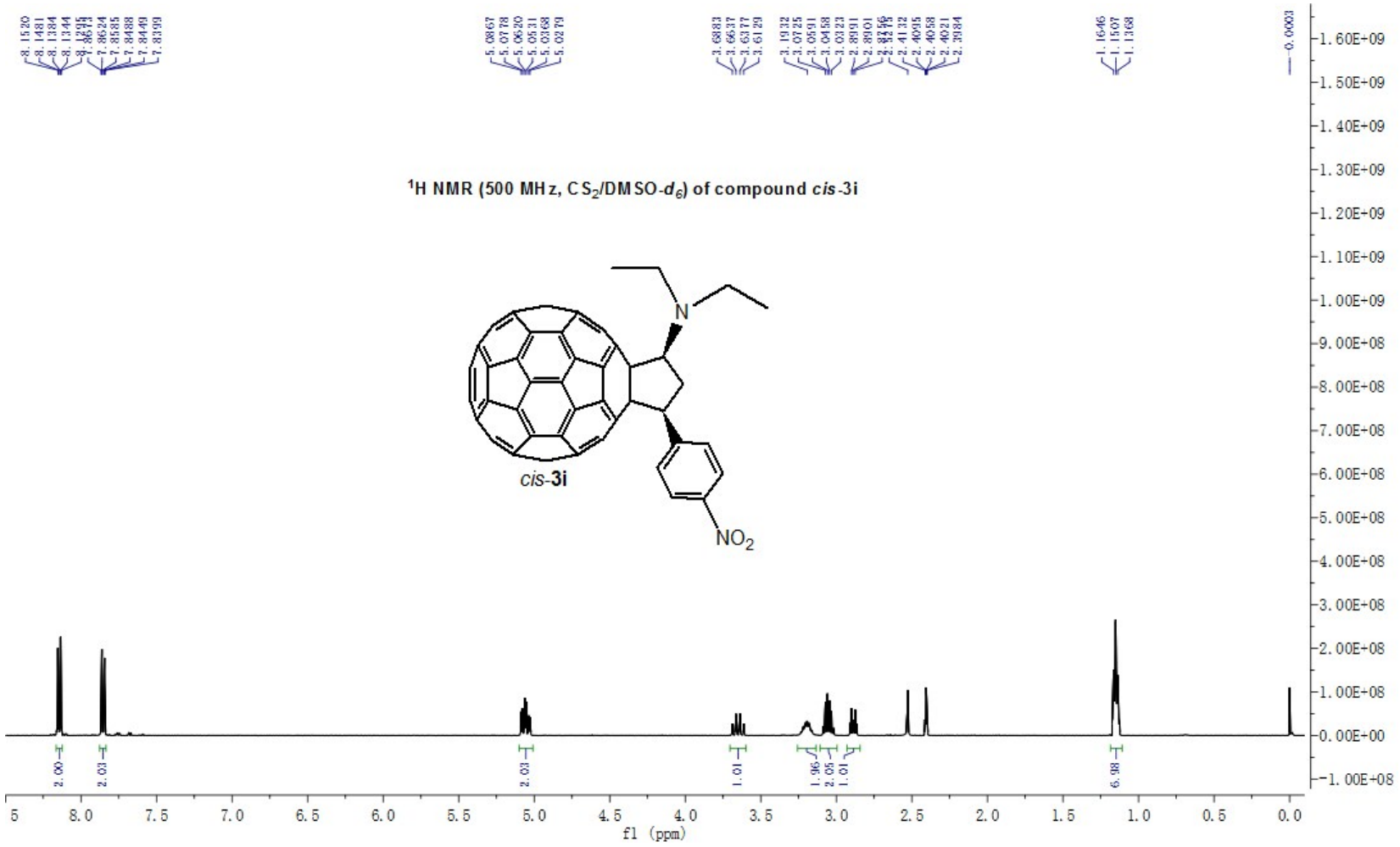
144.0
143.5
143.0
142.5
142.0
141.5
141.0
140.5
f1 (ppm)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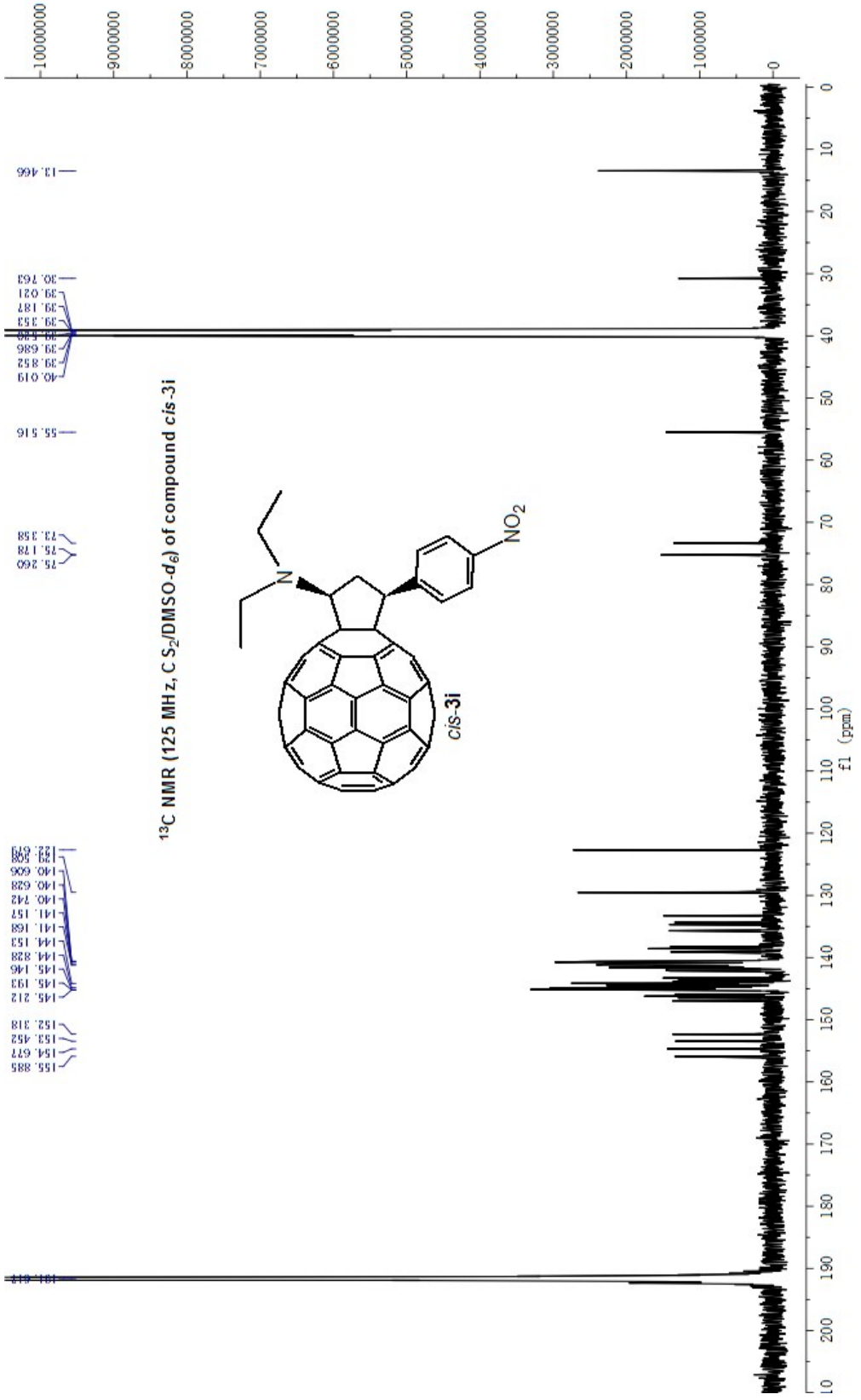
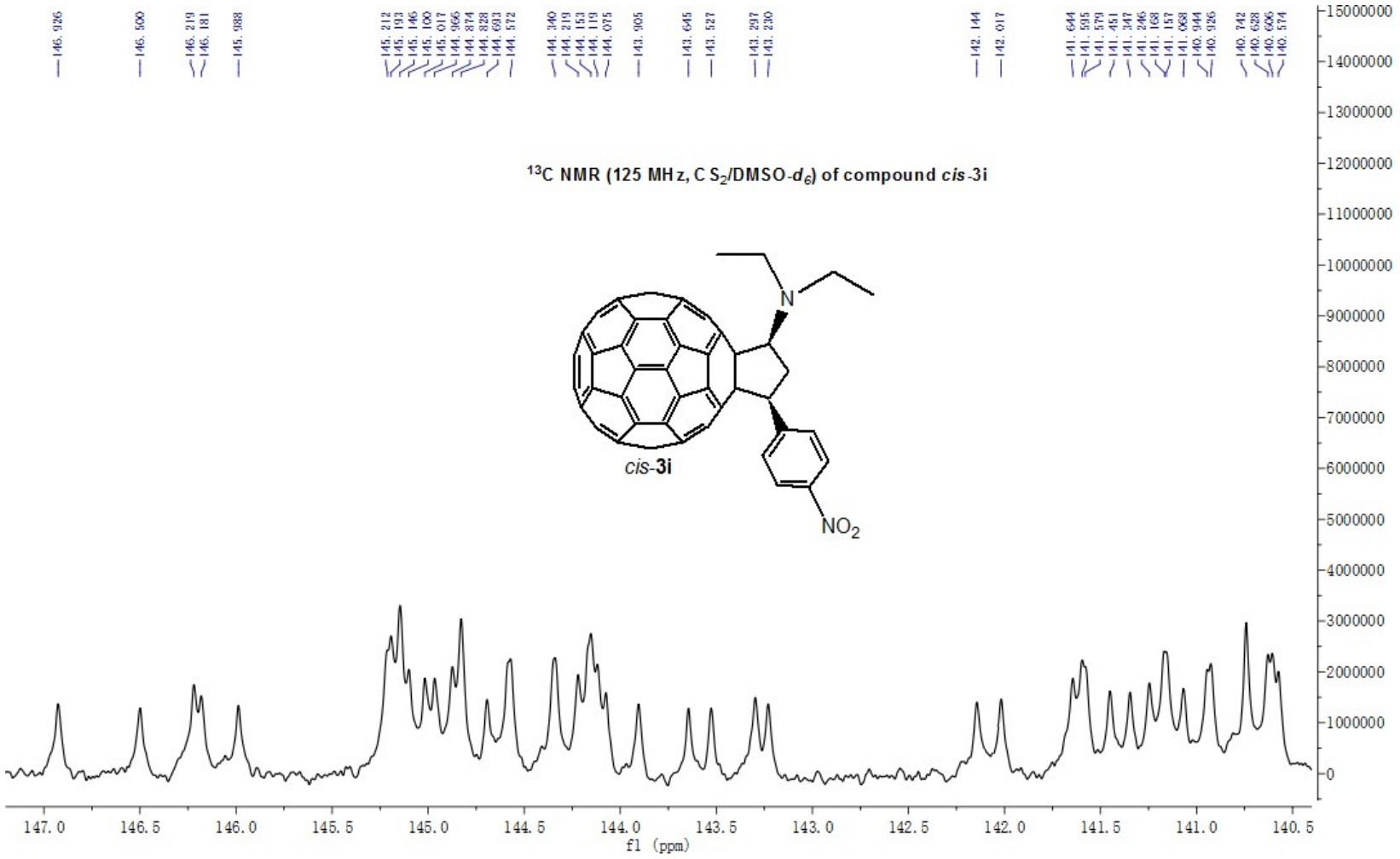
7.8488
7.8449
7.8399
5.0867
5.0778
5.0620
5.0531
5.0368
5.0279
3.6883
3.6637
3.6377
3.6129
3.1932
3.0725
3.0591
3.0458
3.0323
2.8991
2.8901
2.4132
2.4095
2.4058
2.4021
2.3984
1.1646
1.1507
1.1368
-0.0003
1H NMR (500 MHz, CS2/DMSO-d6) of compound cis-3i
N
cis-3i
NO2
2.00
2.03
2.03
1.01
1.96
2.05
1.01
6.98
1.60E+09
1.50E+09
1.40E+09
1.30E+09
1.20E+09
1.10E+09
1.00E+09
9.00E+08
8.00E+08
7.00E+08
6.00E+08
5.00E+08
4.00E+08
3.00E+08
2.00E+08
1.00E+08
0.00E+00
-1.00E+08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


^{13}C NMR (125 MHz, C S_2/DMSO-d_6) of compound *cis*-3i
cis-3i
NO_2
N
f1 (ppm)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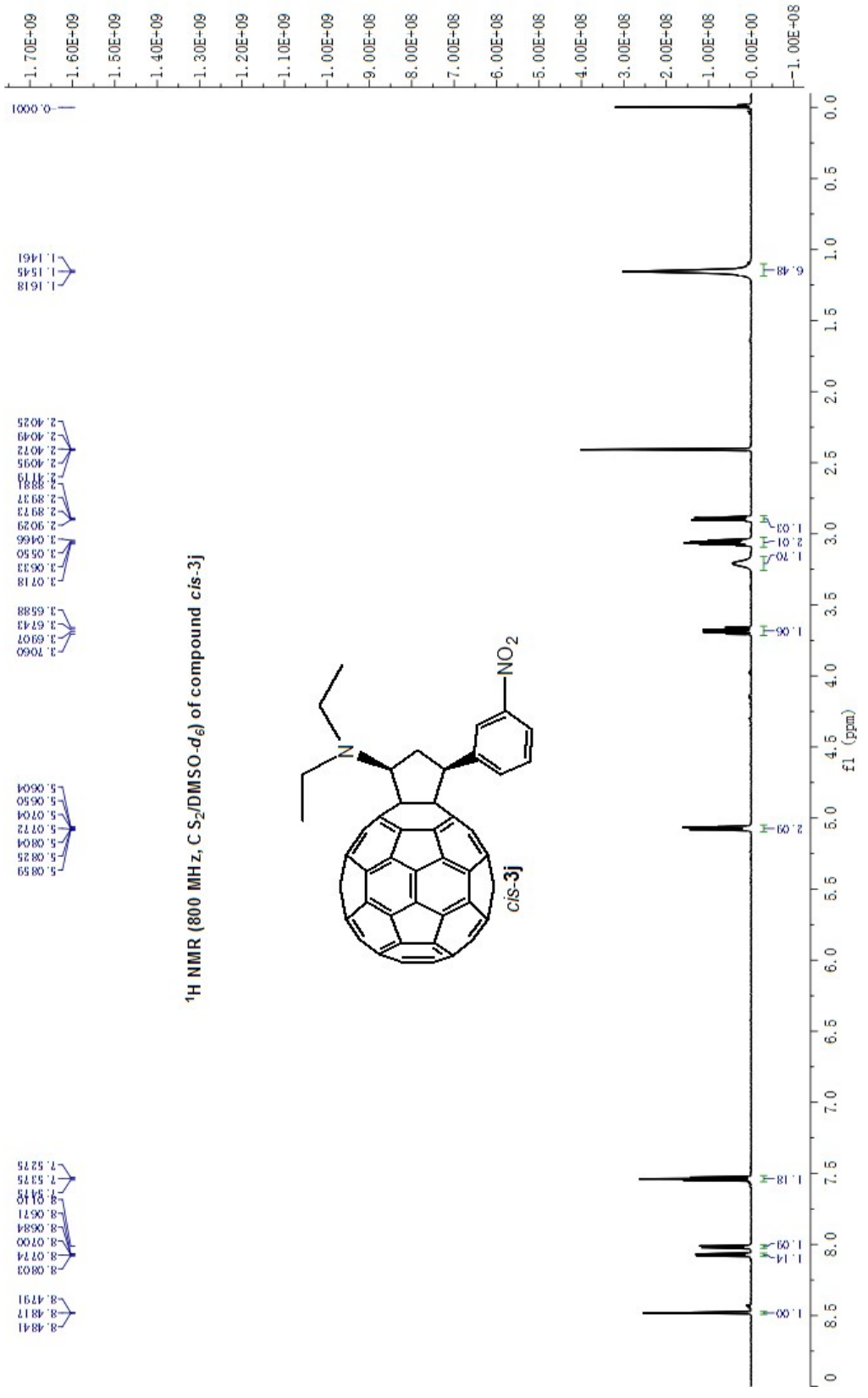
146.500
146.219
146.181
145.988
145.212
145.193
145.146
145.100
145.017
144.966
144.874
144.828
144.693
144.572
144.340
144.219
144.153
144.119
144.075
143.905
143.645
143.527
143.297
143.230
142.144
142.017
141.644
141.595
141.579
141.451
141.347
141.246
141.168
141.157
141.068
140.944
140.926
140.742
140.628
140.605
140.574
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3i
N
NO2
cis-3i
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
147.0
146.5
146.0
145.5
145.0
144.5
144.0
143.5
143.0
142.5
142.0
141.5
141.0
140.5
f1 (ppm)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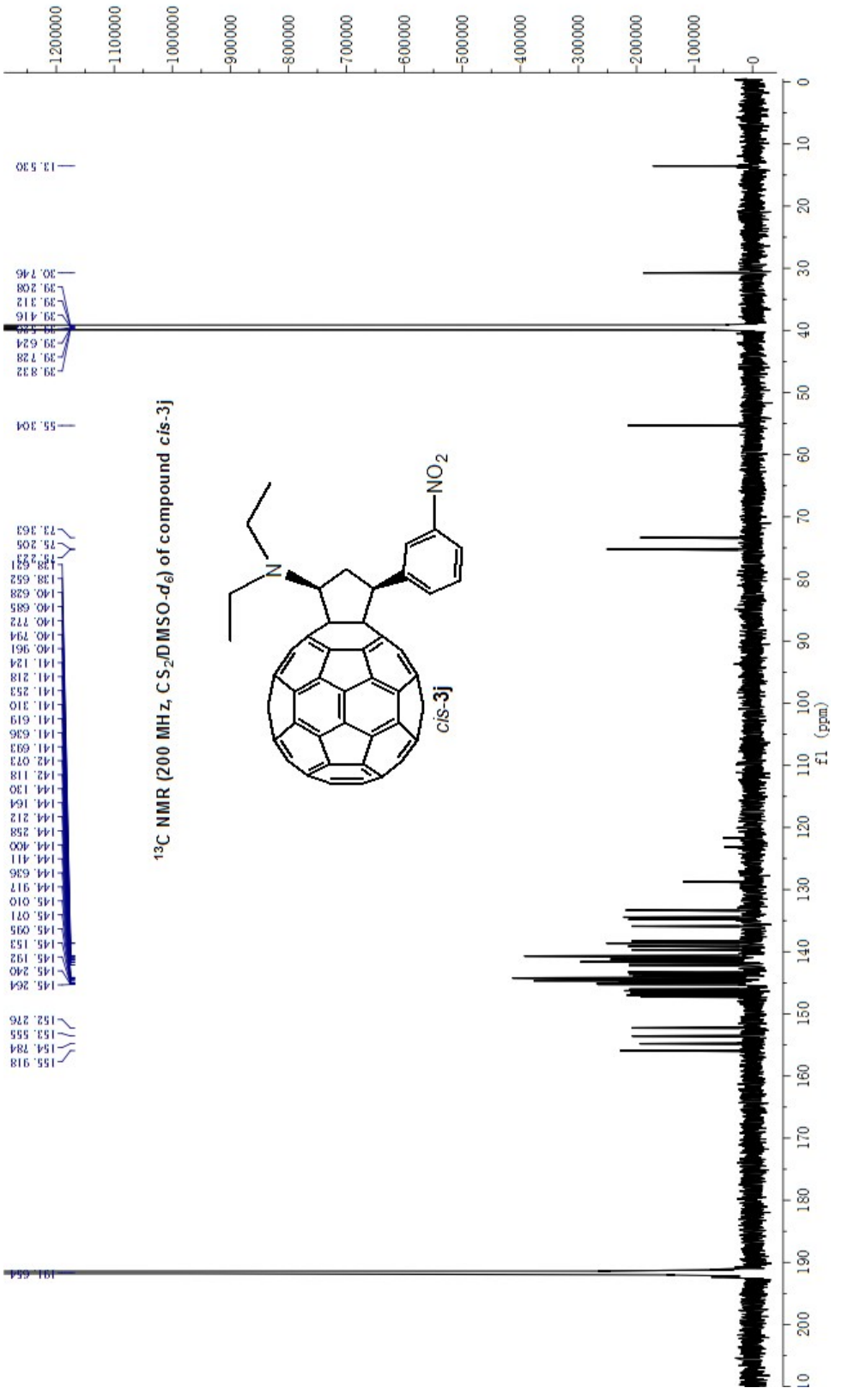
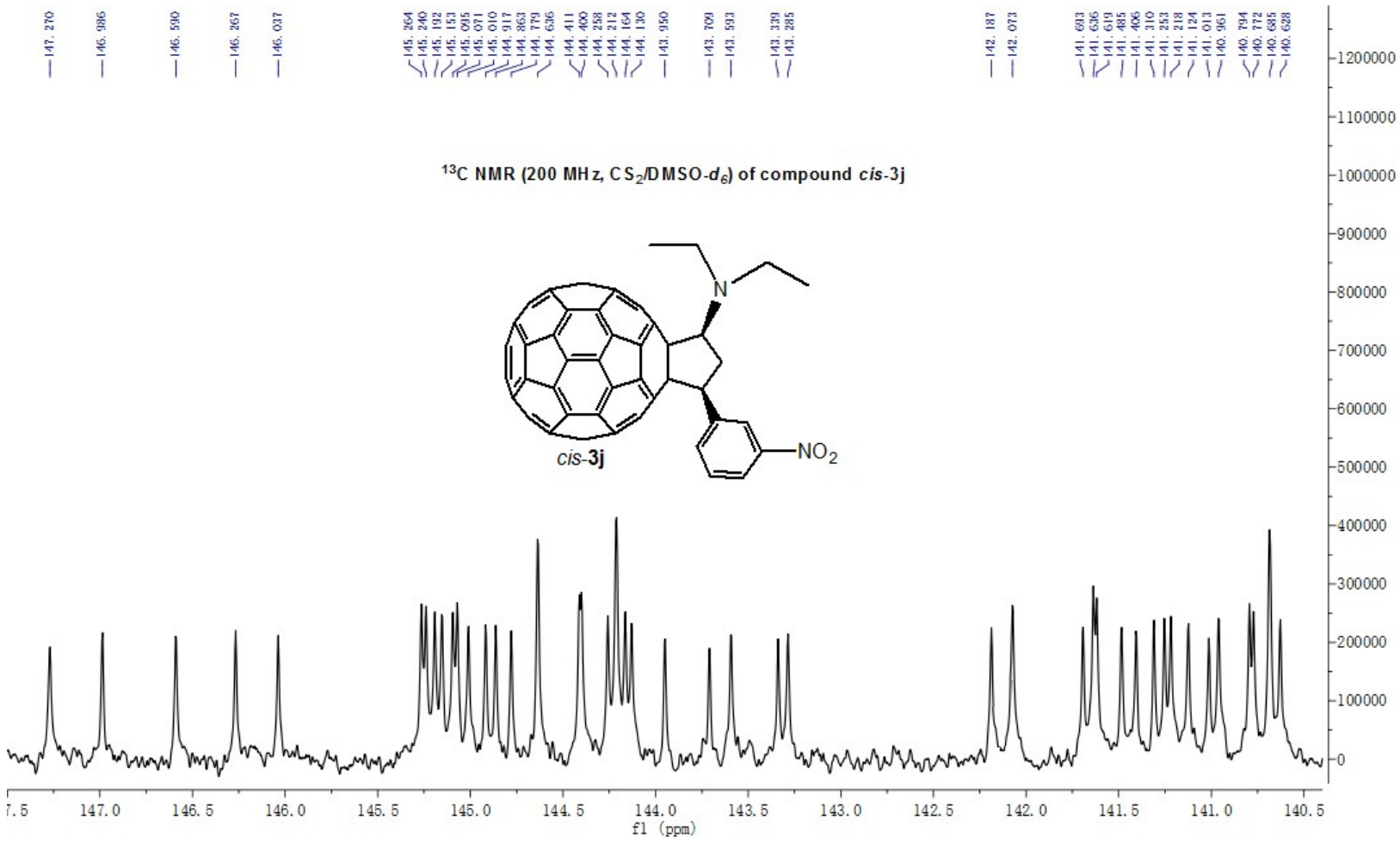
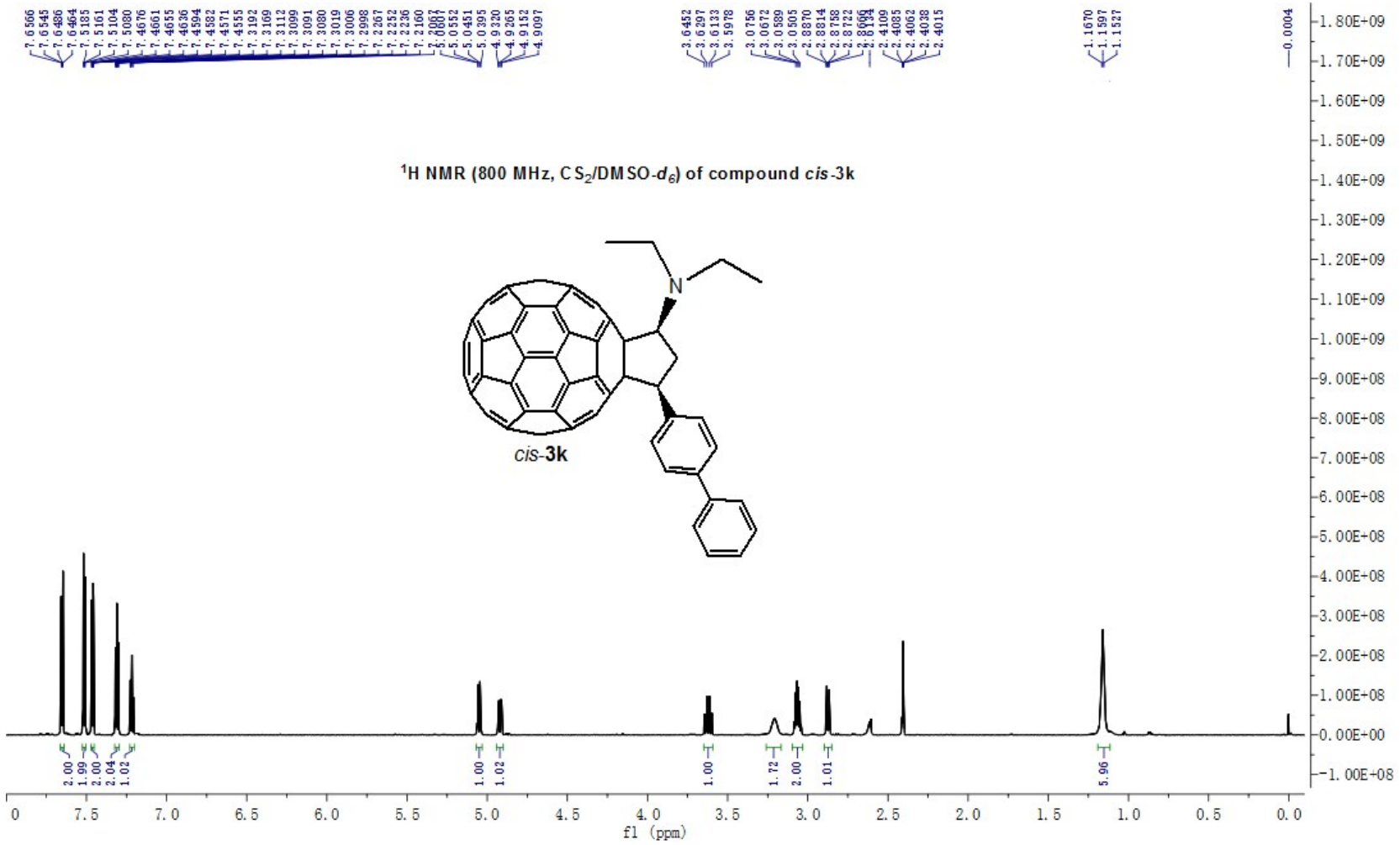
NO_2
N
cis-3j
f1 (ppm)


^{13}C NMR (200 MHz, CS_2/DMSO-d_6) of compound *cis*-3j
NO_2
N
cis-3j
f1 (ppm)



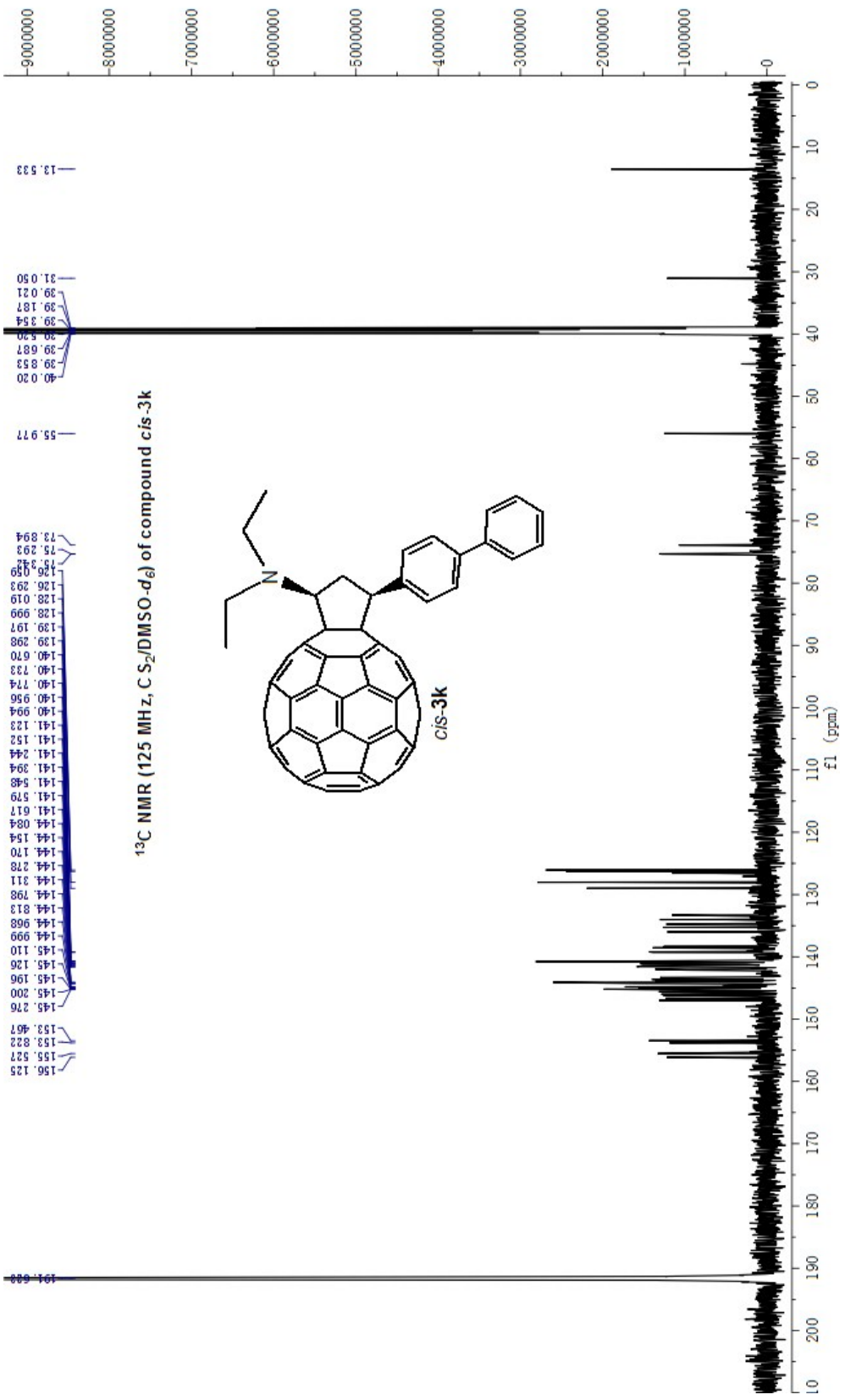
^{13}C NMR (200 MHz, CS_2/DMSO-d_6) of compound *cis*-3j

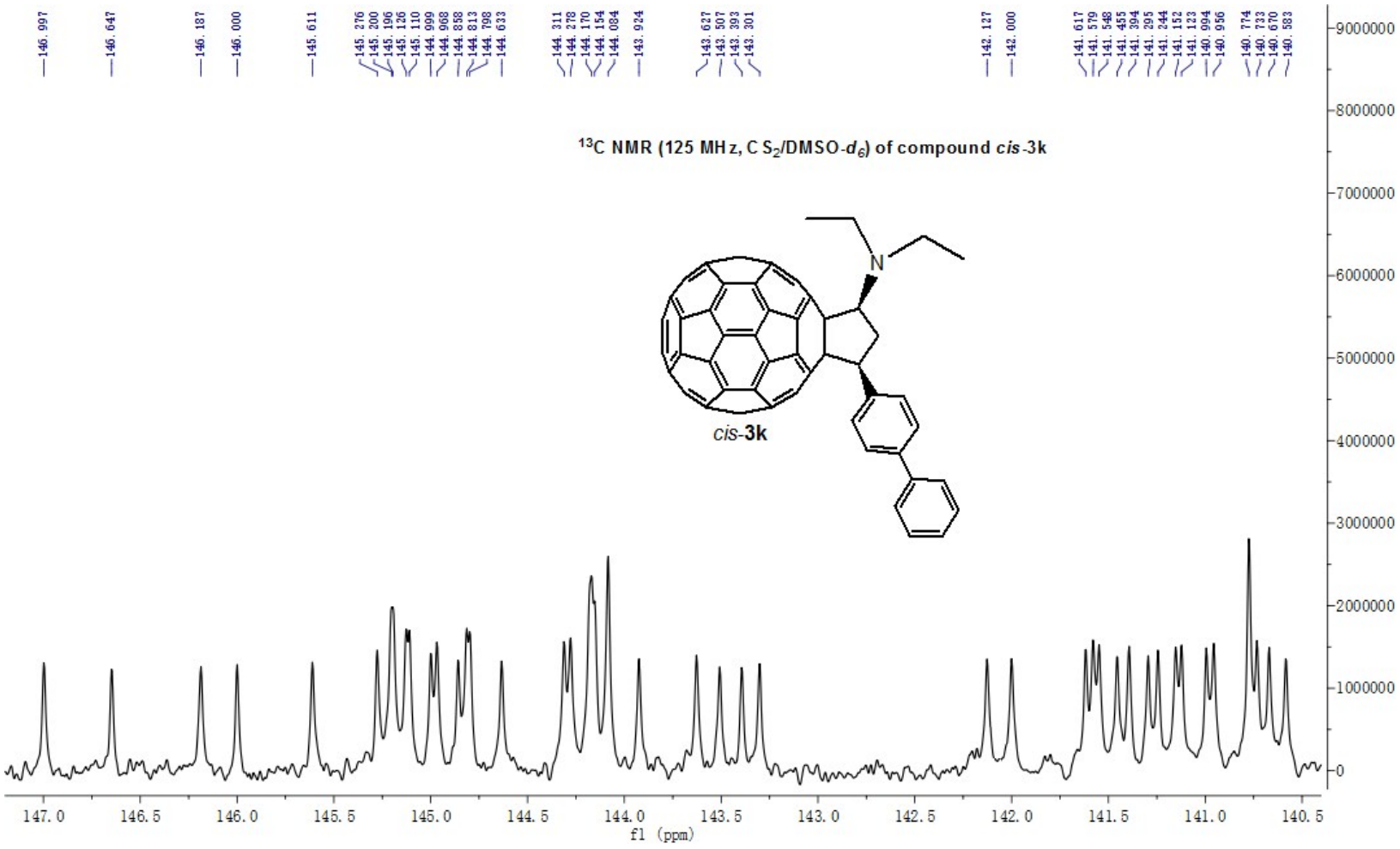
cis-**3j**

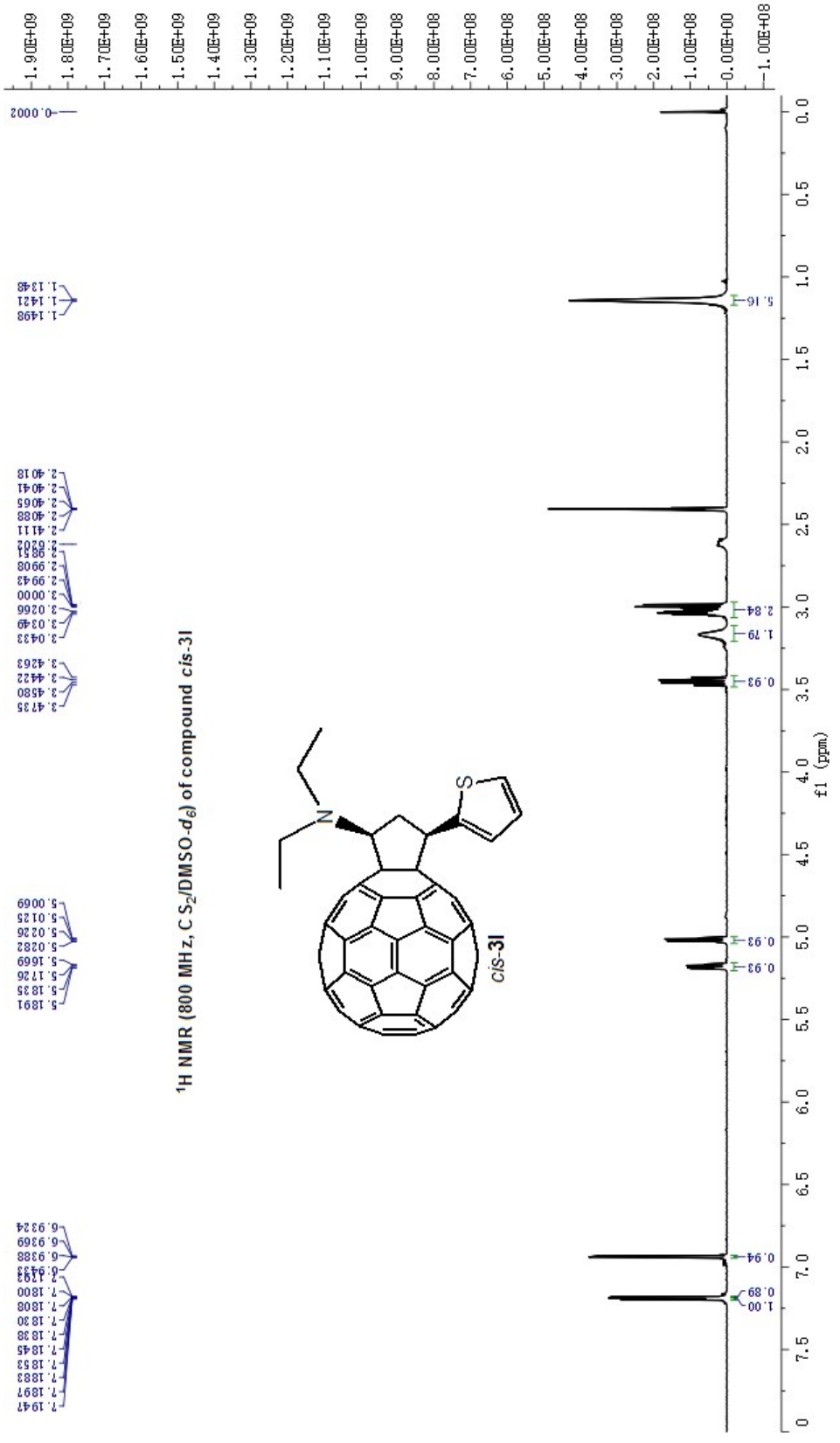


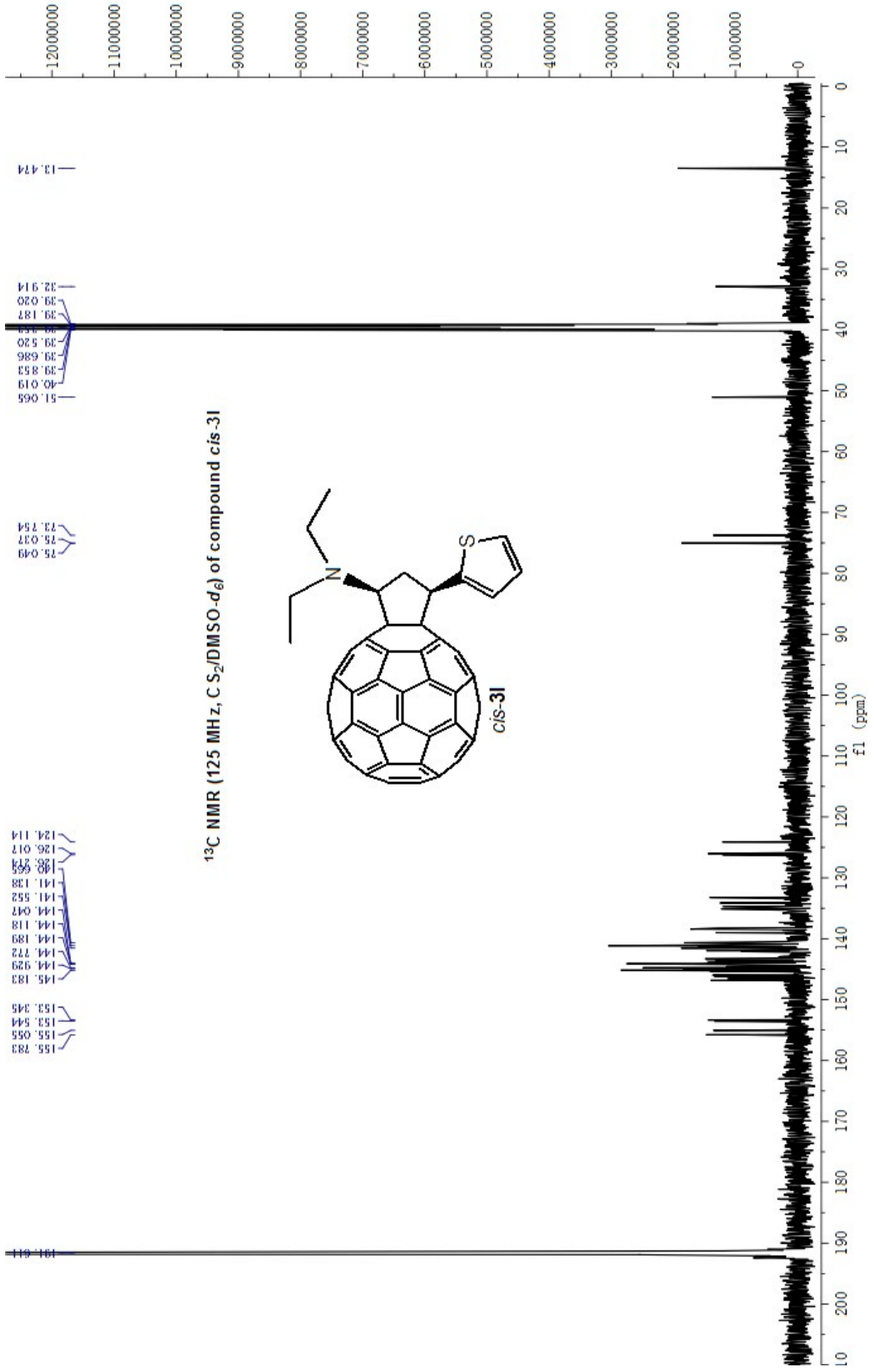
^{1}H NMR (800 MHz, CS_2/DMSO-d_6) of compound *cis*-3k

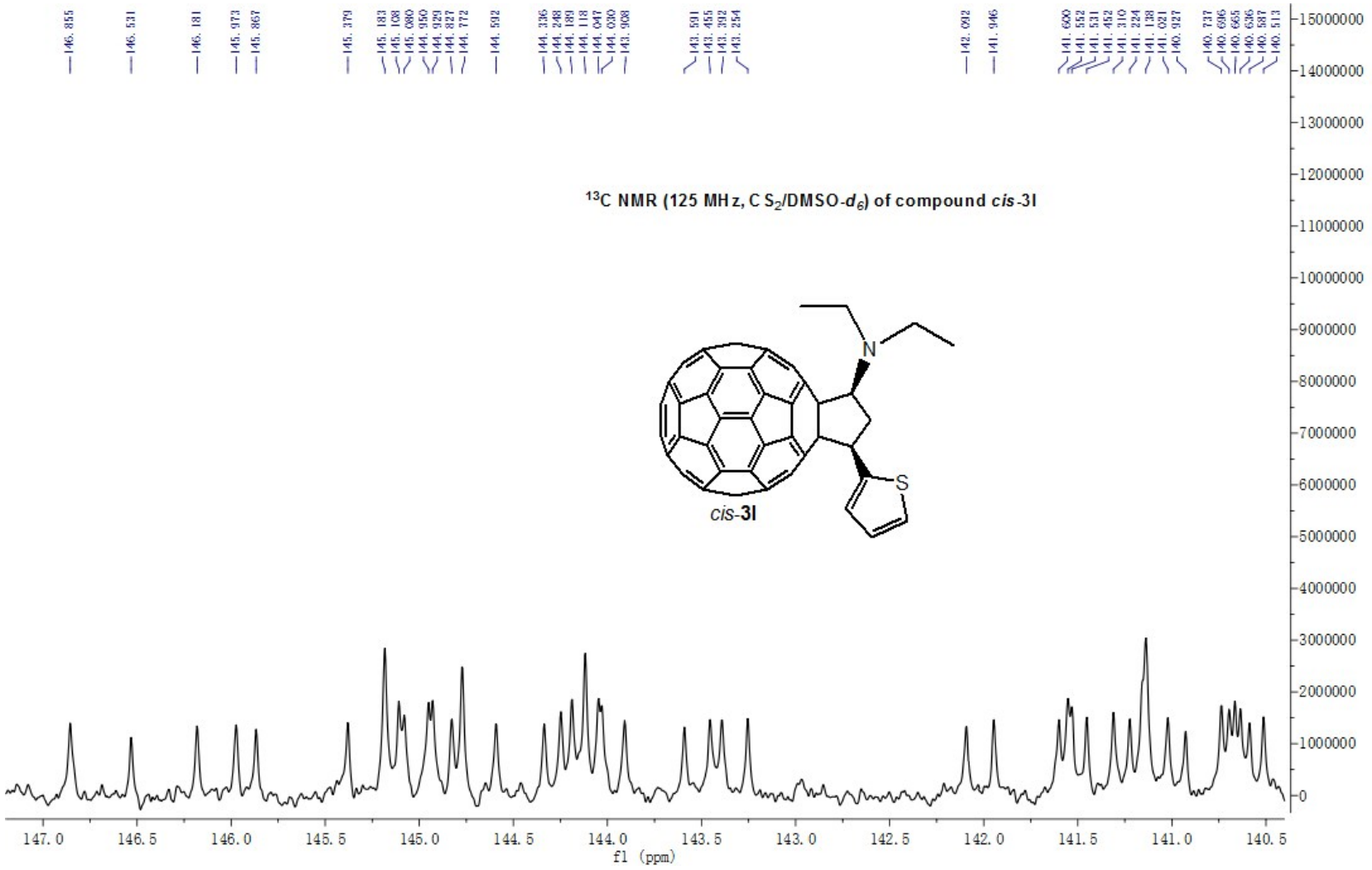
cis-**3k**


13.533
31.050
39.021
39.187
39.354
39.520
39.687
39.853
40.020
55.977
126.059
126.293
128.019
128.999
139.197
139.298
140.670
140.733
140.774
140.956
140.994
141.123
141.152
141.244
141.394
141.548
141.579
141.617
144.084
144.154
144.170
144.278
144.311
144.798
144.813
144.968
144.999
145.110
145.126
145.196
145.200
145.276
153.467
153.822
155.527
156.125
191.689
13C NMR (125 MHz, C S2/DMSO-d6) of compound cis-3k
cis-3k
f1 (ppm)


146.997
146.647
146.187
146.000
145.611
145.276
145.200
145.196
145.126
145.110
144.999
144.968
144.838
144.813
144.798
144.633
144.311
144.278
144.170
144.154
144.084
143.924
143.627
143.507
143.393
143.301
142.127
142.000
141.617
141.579
141.548
141.455
141.394
141.295
141.244
141.152
141.123
140.994
140.956
140.774
140.733
140.670
140.583
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3k
N
cis-3k
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
147.0
146.5
146.0
145.5
145.0
144.5
144.0
143.5
143.0
142.5
142.0
141.5
141.0
140.5
f1 (ppm)

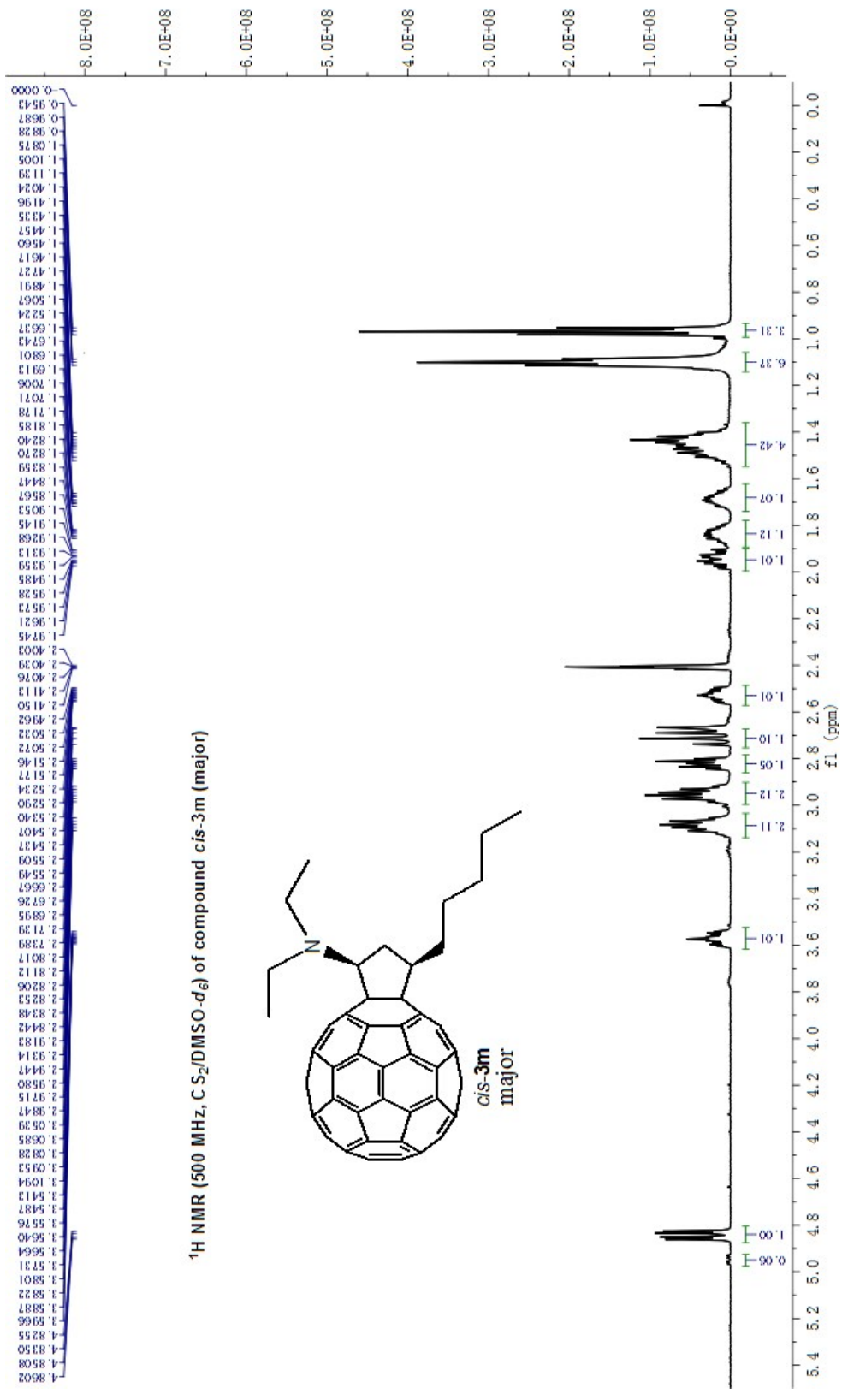

1H NMR (800 MHz, CS2/DMSO-d6) of compound cis-3l
cis-3l
f1 (ppm)

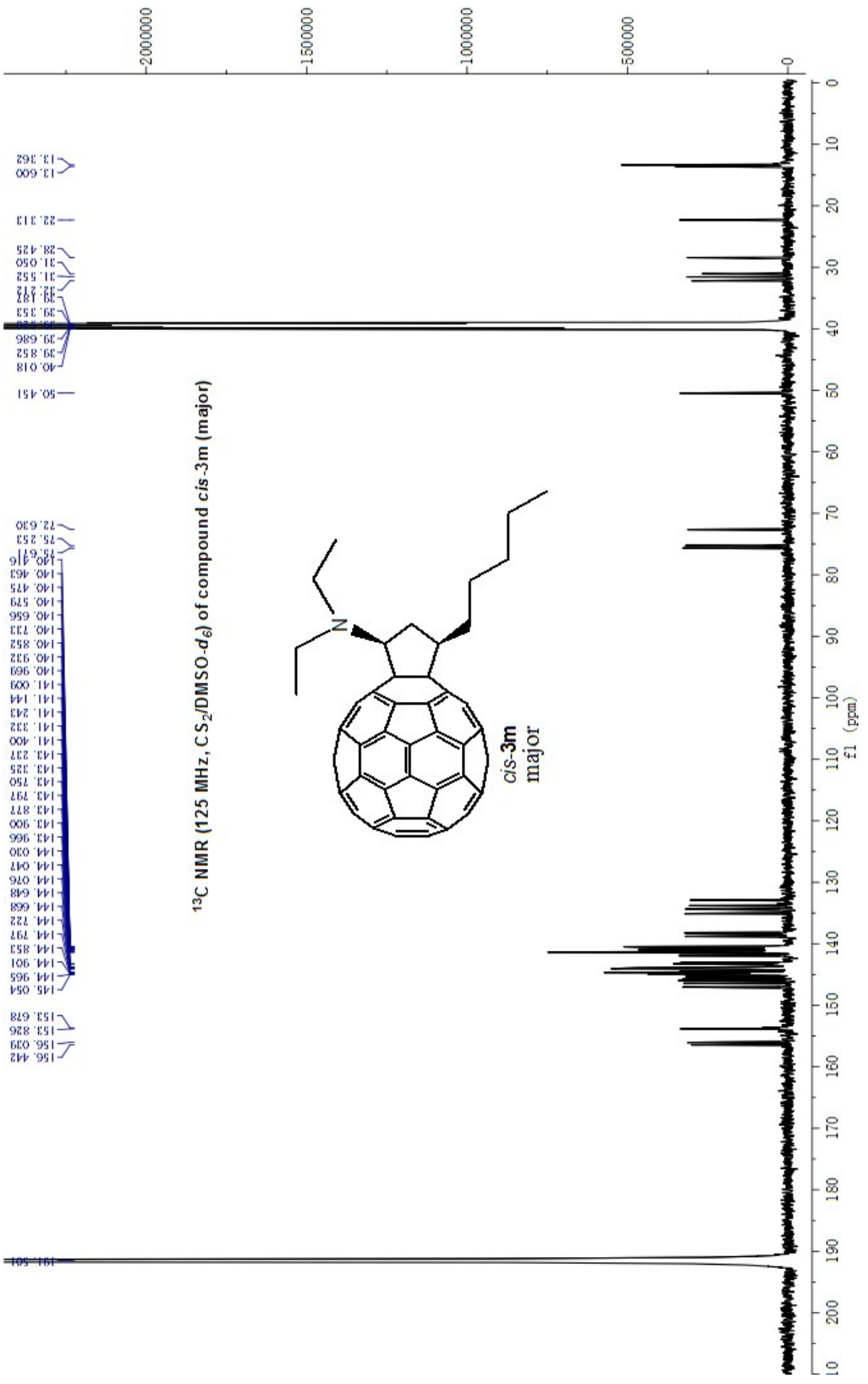

^{13}C NMR (125 MHz, C S_2/DMSO-d_6) of compound *cis*-3l
N
S
cis-3l
f1 (ppm)

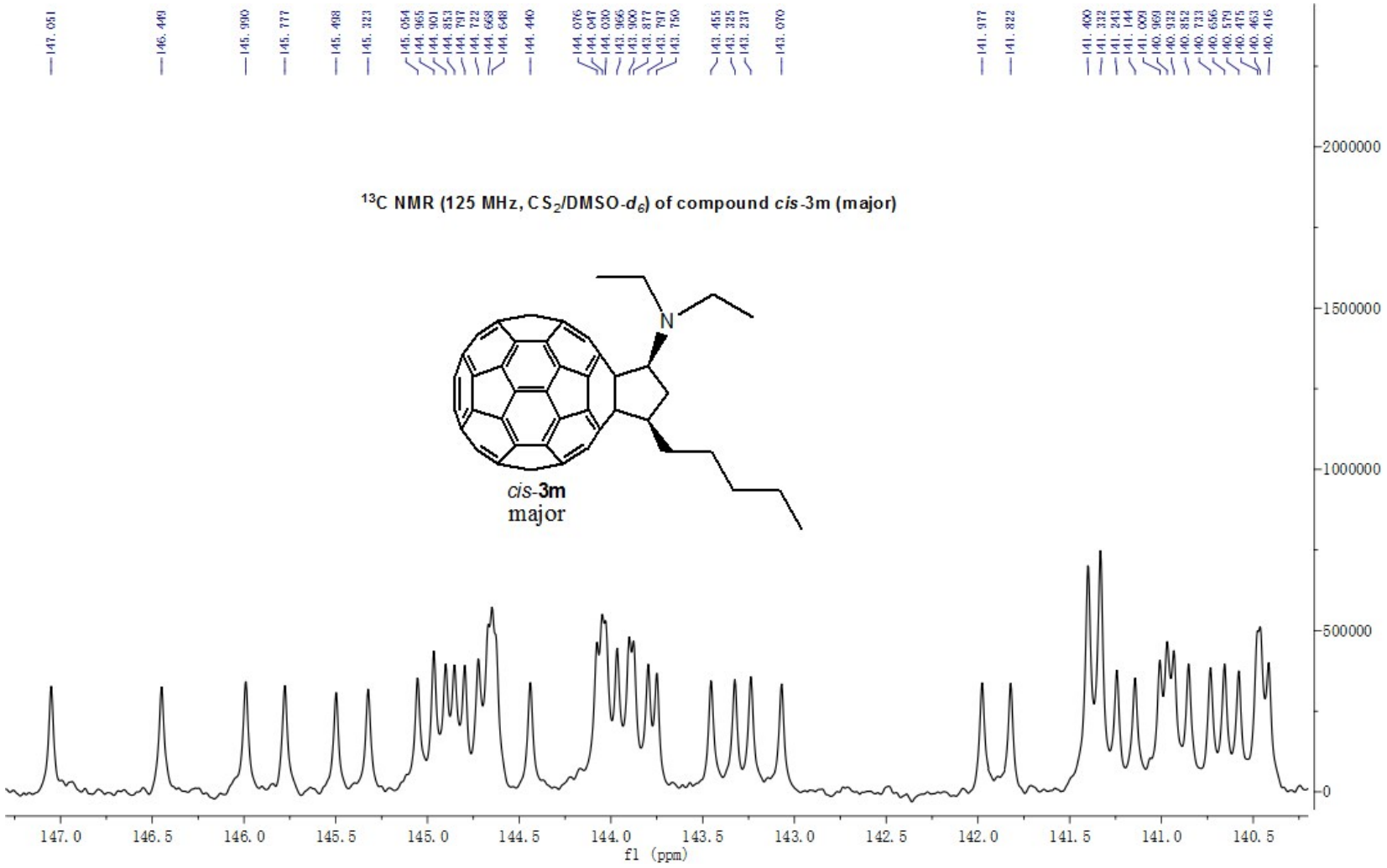


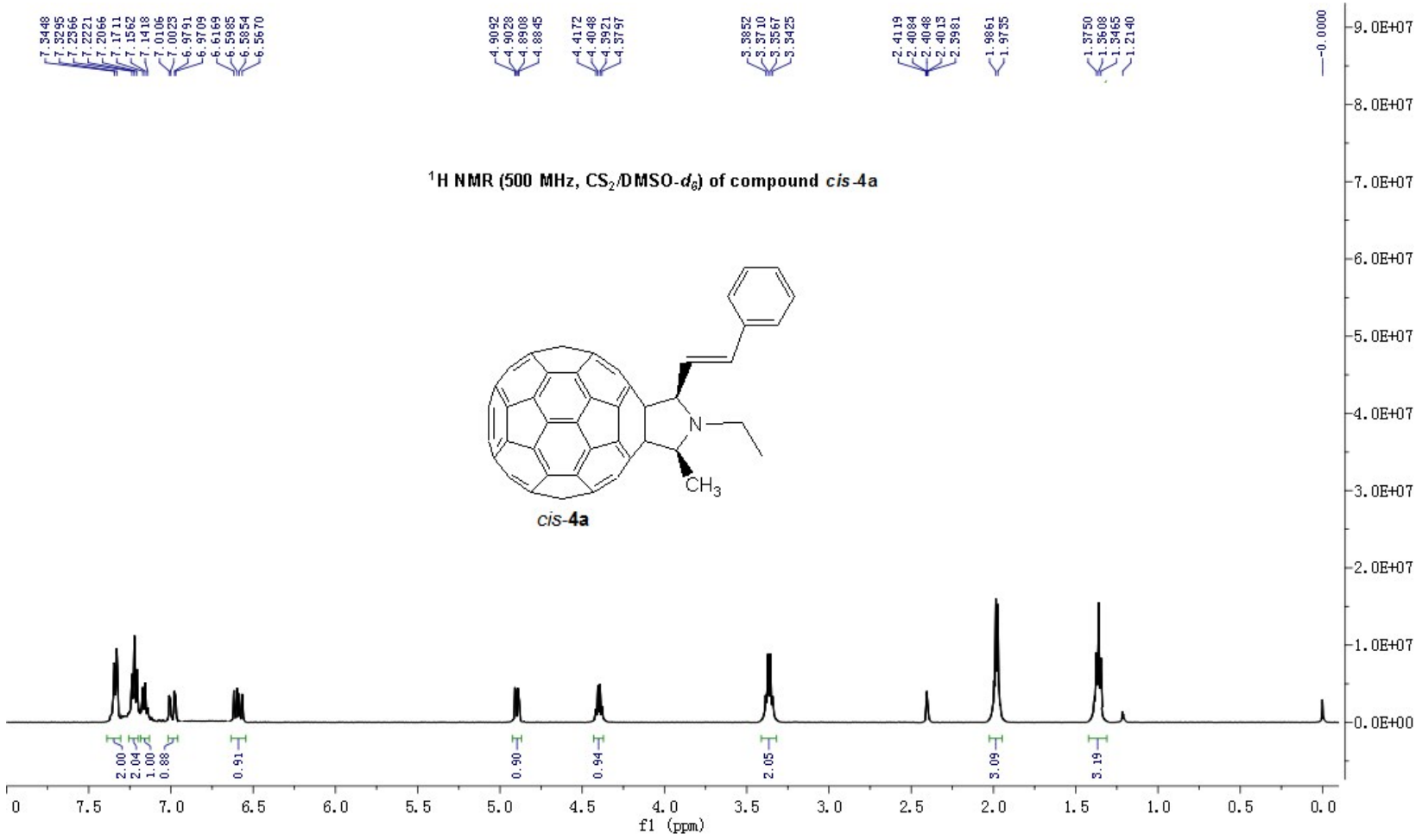
^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *cis*-3l

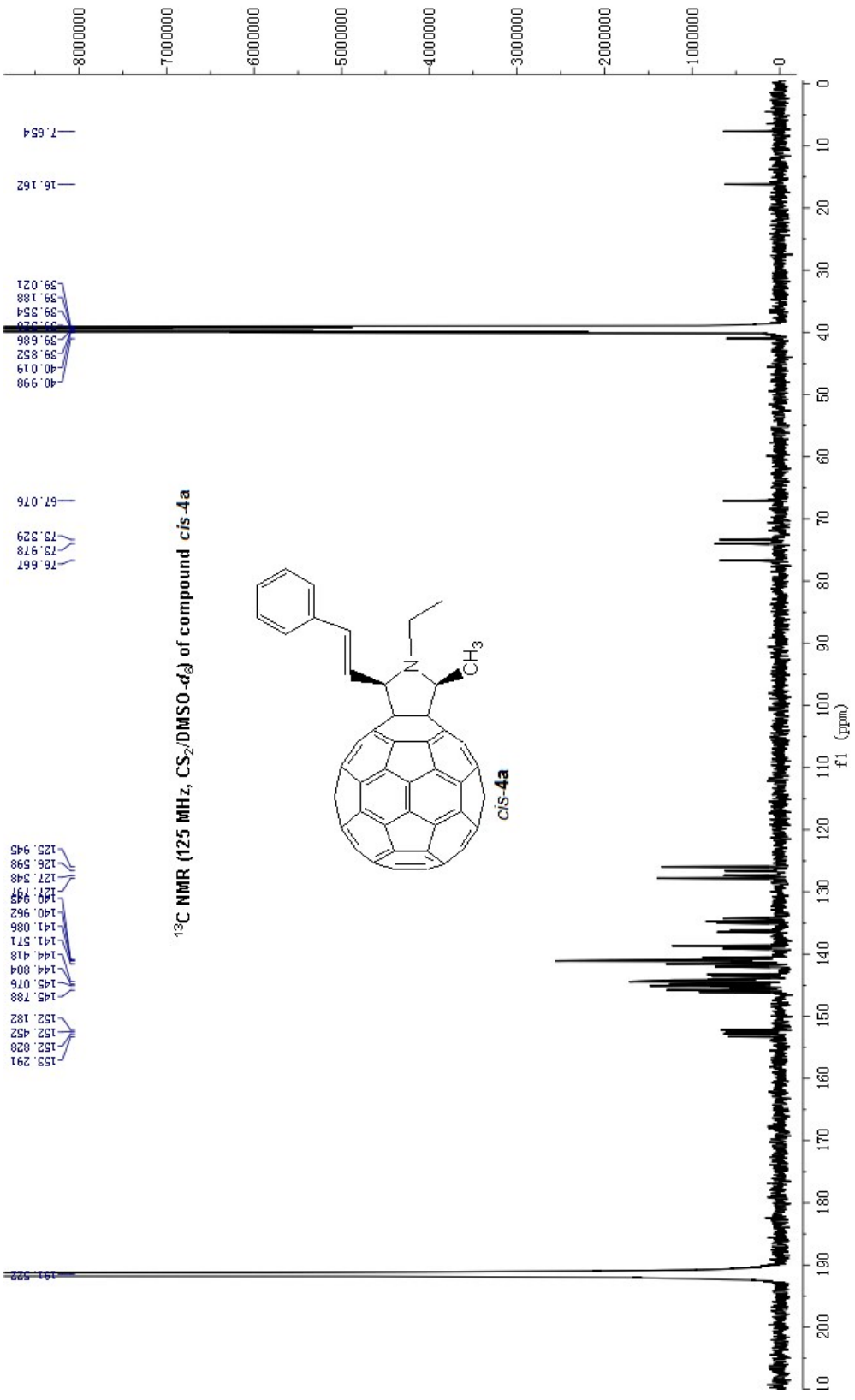
cis-**3l**

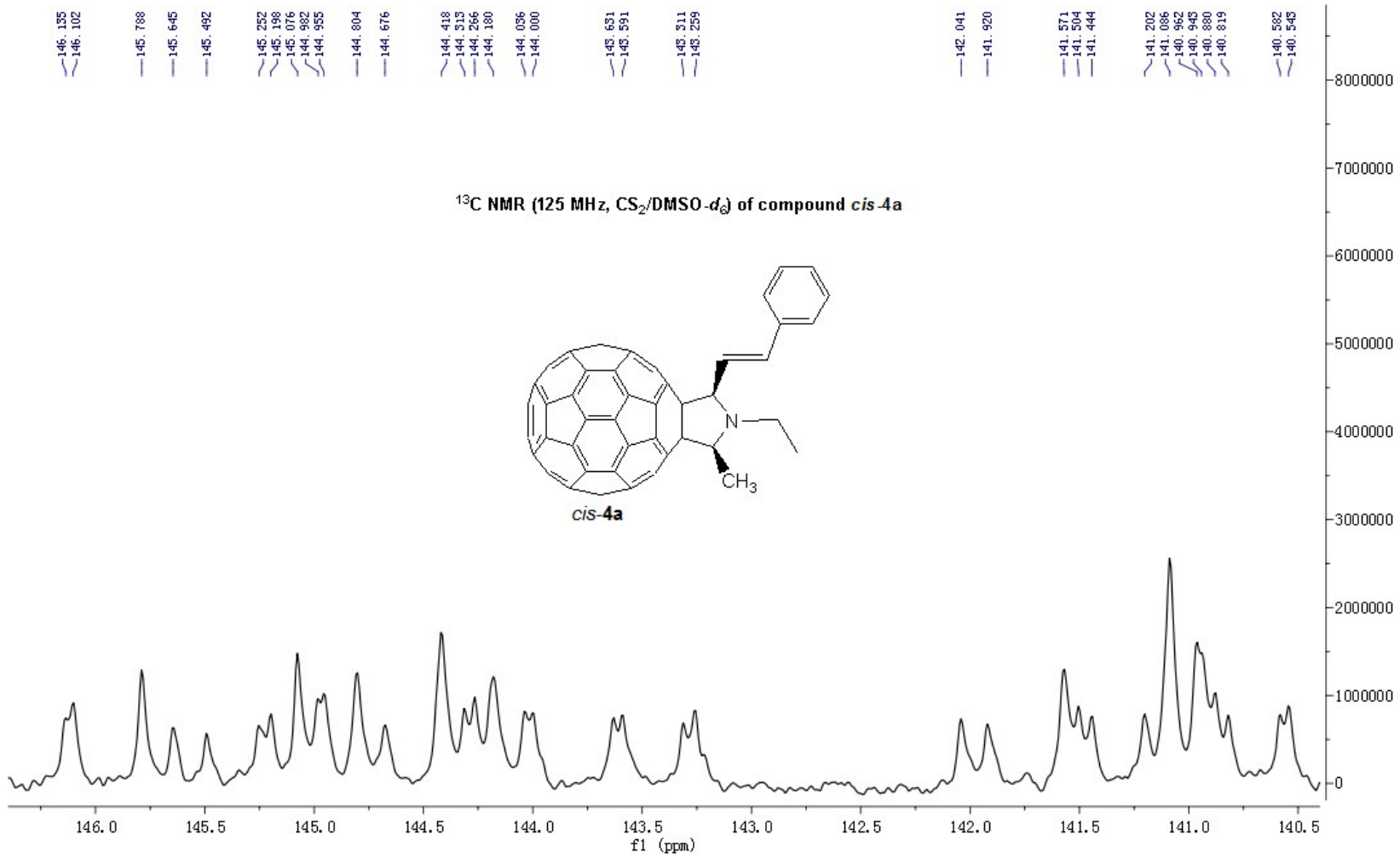

¹H NMR (500 MHz, C S2/DMSO-d6) of compound cis-3m (major)
cis-3m
major
f1 (ppm)


13.600
13.362
22.313
28.425
31.050
31.552
39.353
39.686
39.852
40.018
50.451
72.630
75.253
75.611
145.054
153.678
153.826
156.039
156.442
191.501
2000000
1500000
1000000
500000
0
f1 (ppm)
N
cis-3m
major
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3m (major)


147.051
146.449
145.990
145.777
145.498
145.323
145.054
144.965
144.901
144.853
144.797
144.722
144.668
144.648
144.440
144.076
144.047
144.030
143.966
143.900
143.877
143.797
143.750
143.455
143.325
143.237
143.070
141.977
141.822
141.400
141.332
141.243
141.144
141.009
140.969
140.932
140.852
140.733
140.656
140.579
140.475
140.463
140.416
13C NMR (125 MHz, CS2/DMSO-d6) of compound cis-3m (major)
N
cis-3m
major
2000000
1500000
1000000
500000
0
147.0
146.5
146.0
145.5
145.0
144.5
144.0
143.5
143.0
142.5
142.0
141.5
141.0
140.5
f1 (ppm)

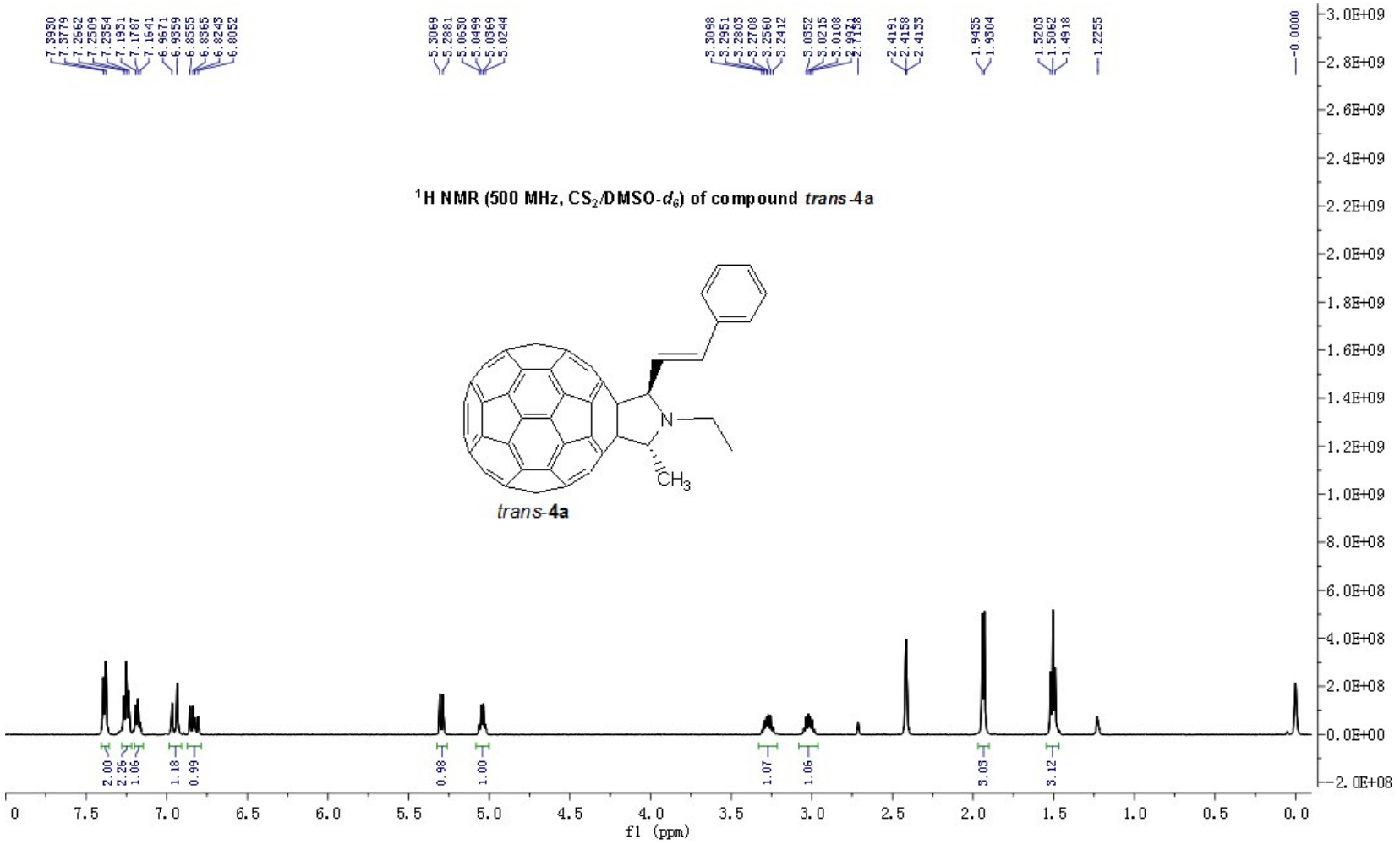

7.3448
7.3295
7.2966
7.2221
7.2066
7.1711
7.1562
7.1418
7.0106
7.0023
6.9791
6.9709
6.6169
6.5985
6.5854
6.5670
4.9092
4.9028
4.8908
4.8845
4.4172
4.4048
4.3921
4.3797
3.3852
3.3710
3.3567
3.3425
2.4119
2.4084
2.4048
2.4013
2.3981
1.9861
1.9735
1.3750
1.3608
1.3465
1.2140
0.0000
1H NMR (500 MHz, CS2/DMSO-d6) of compound cis-4a
N
CH3
cis-4a
2.00
2.04
1.00
0.88
0.91
0.90
0.94
2.05
3.09
3.19
f1 (ppm)

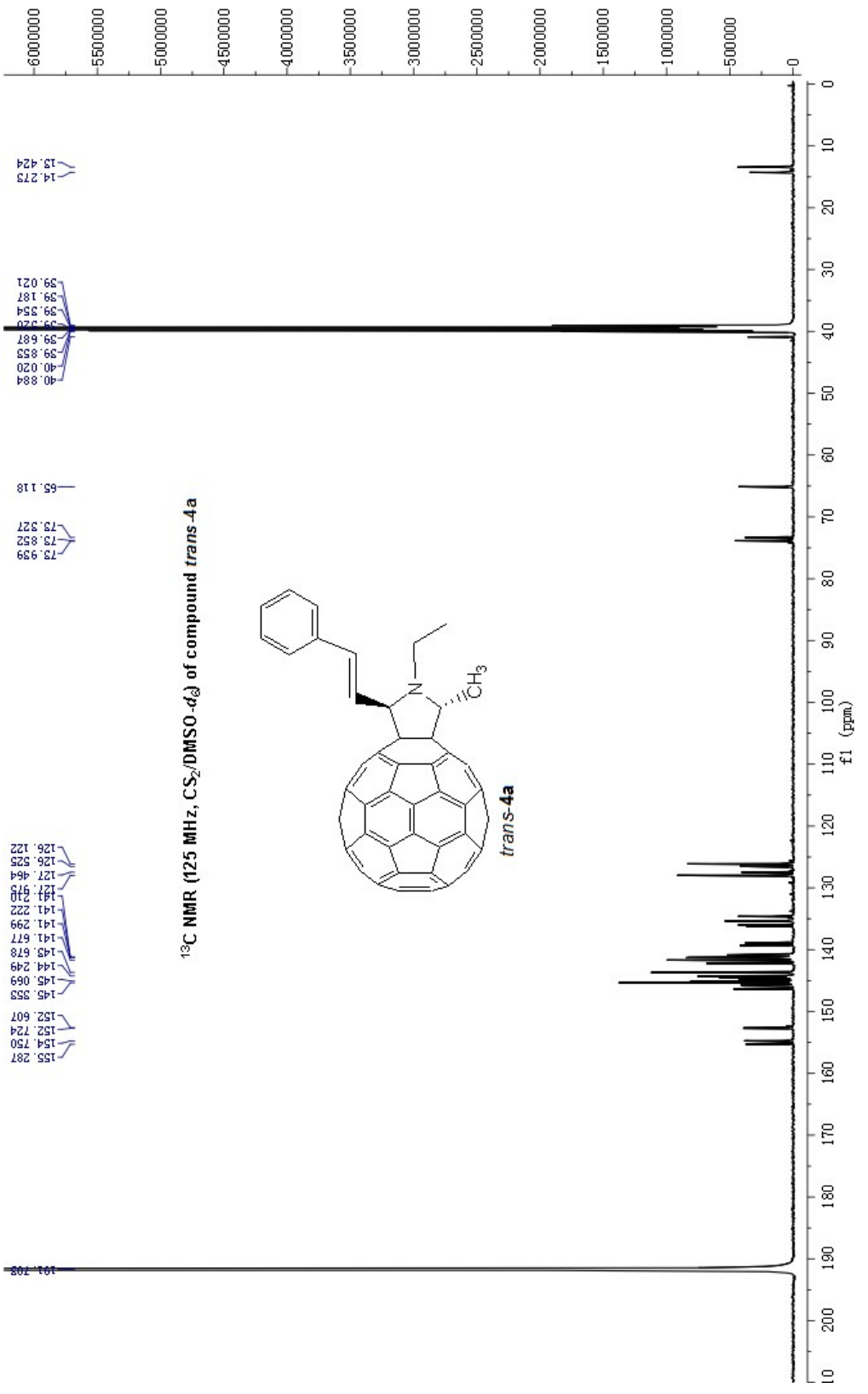

^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *cis*-4a
cis-4a
f1 (ppm)

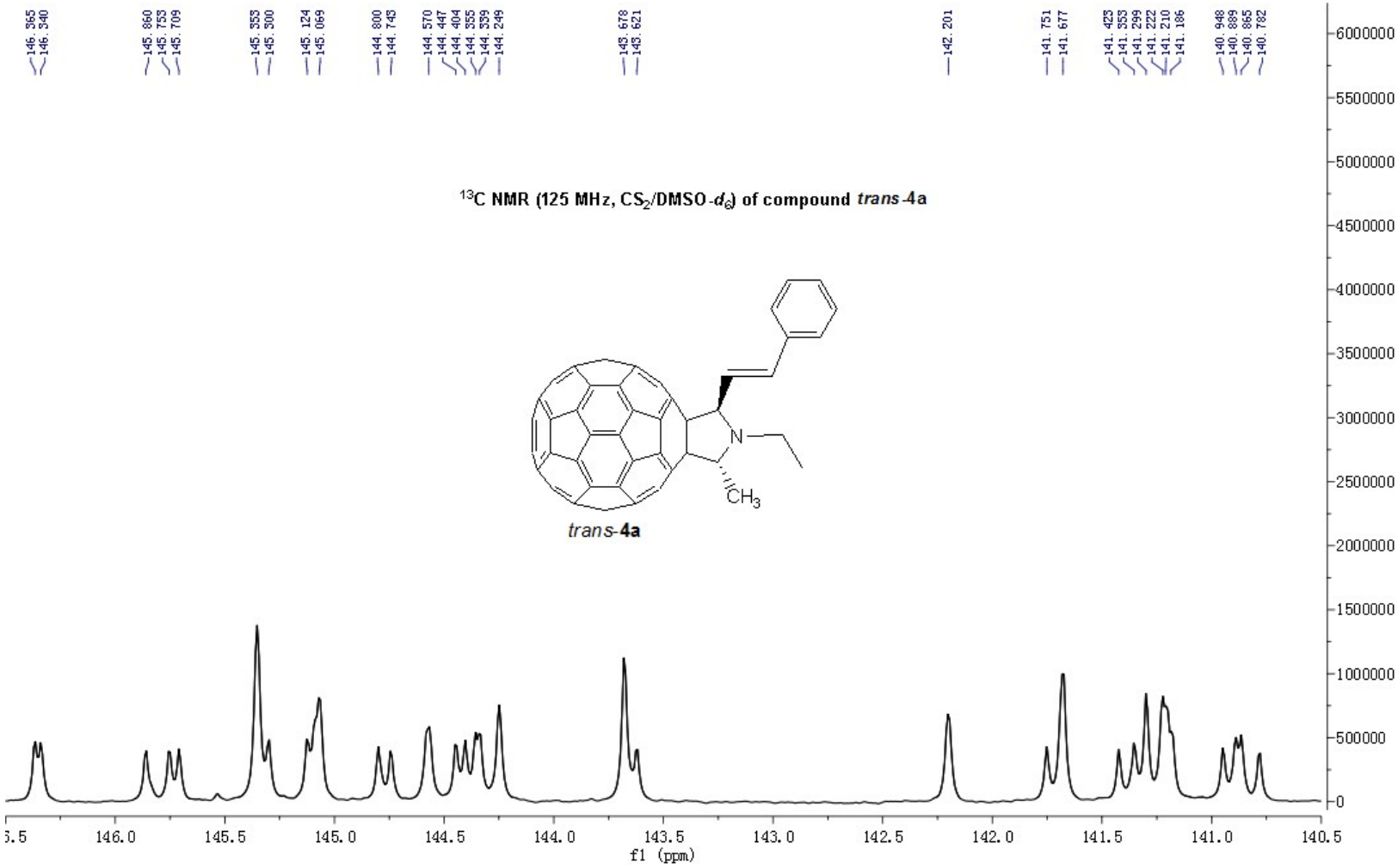


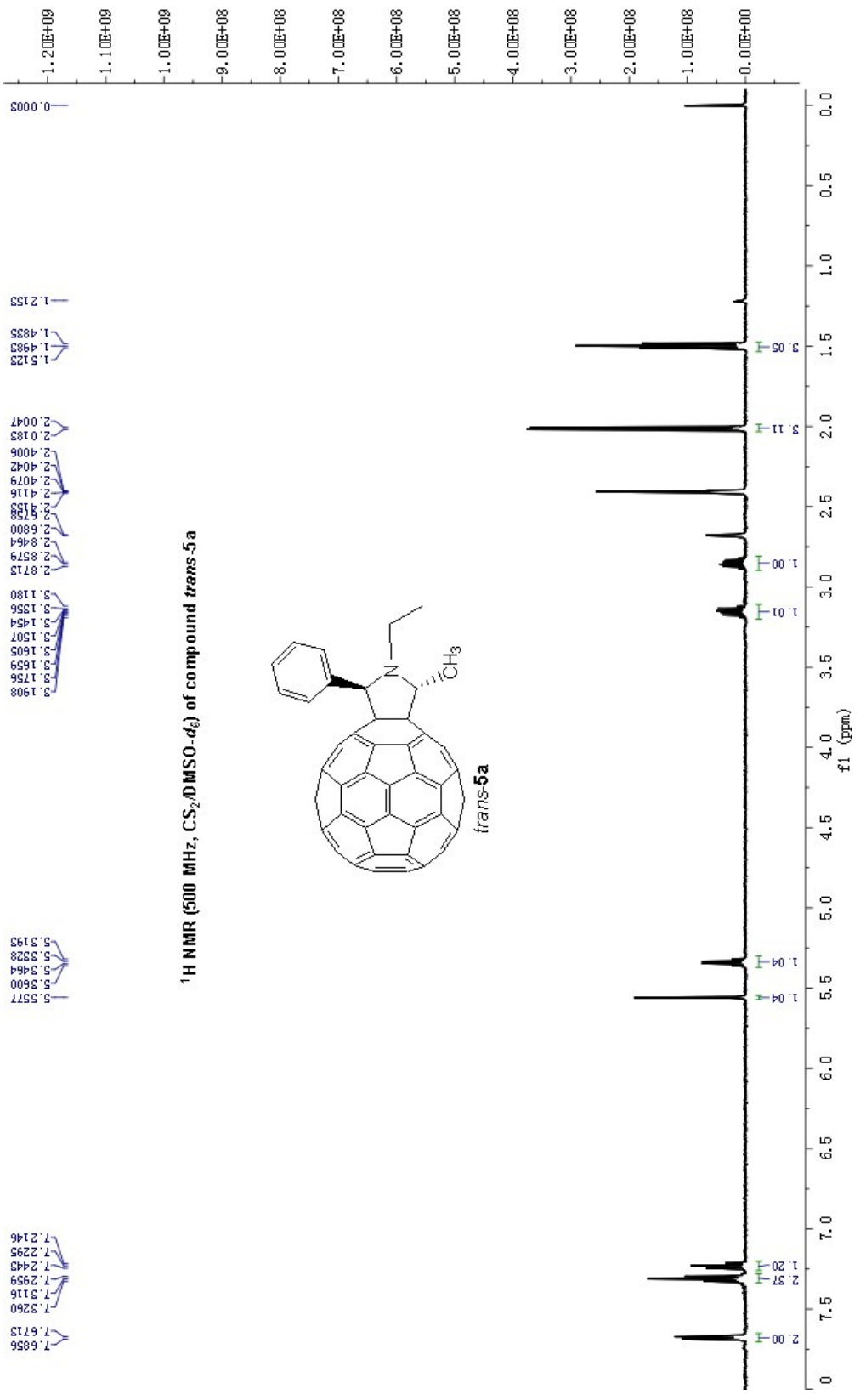
^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *cis*-**4a**

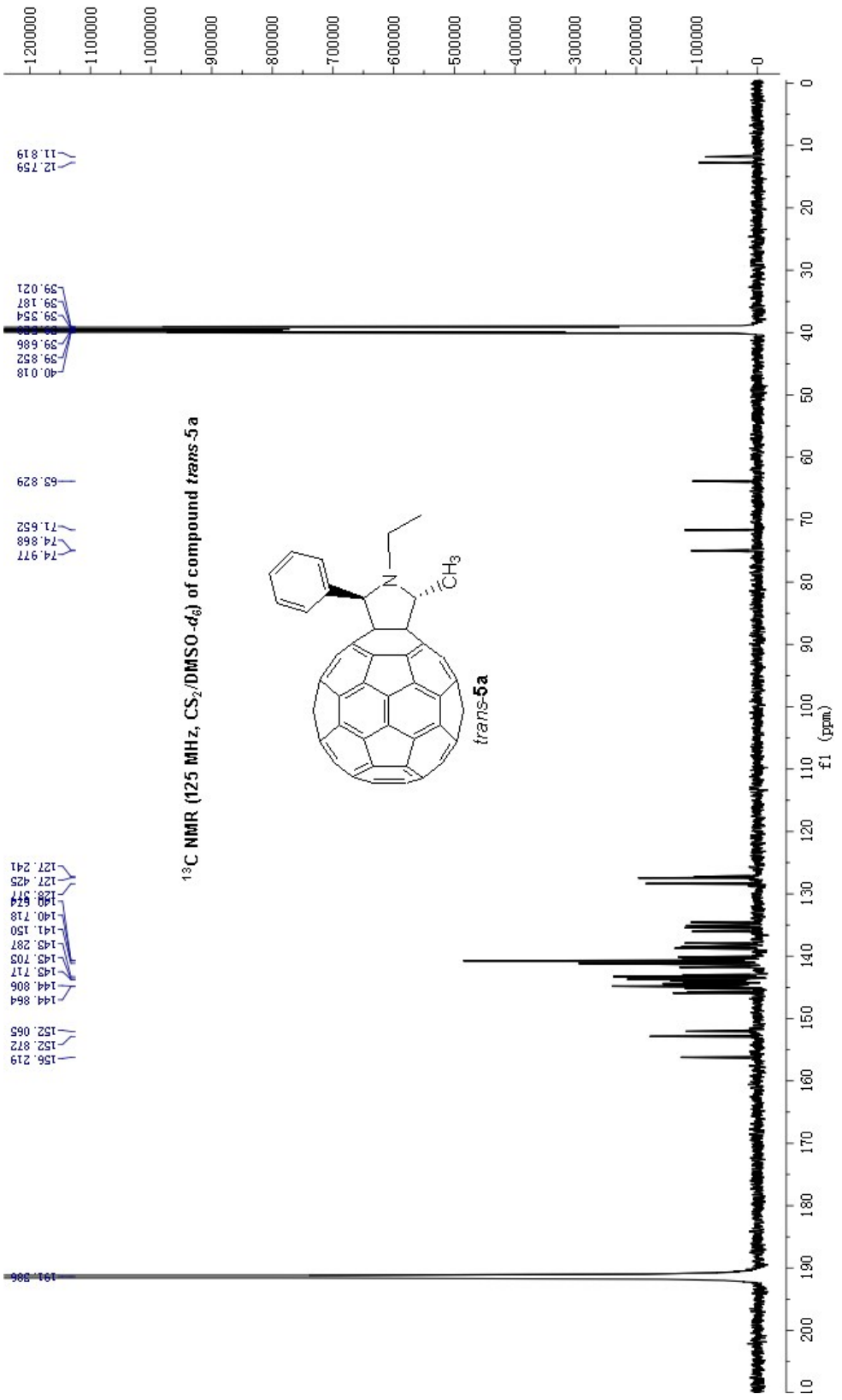
cis-**4a**

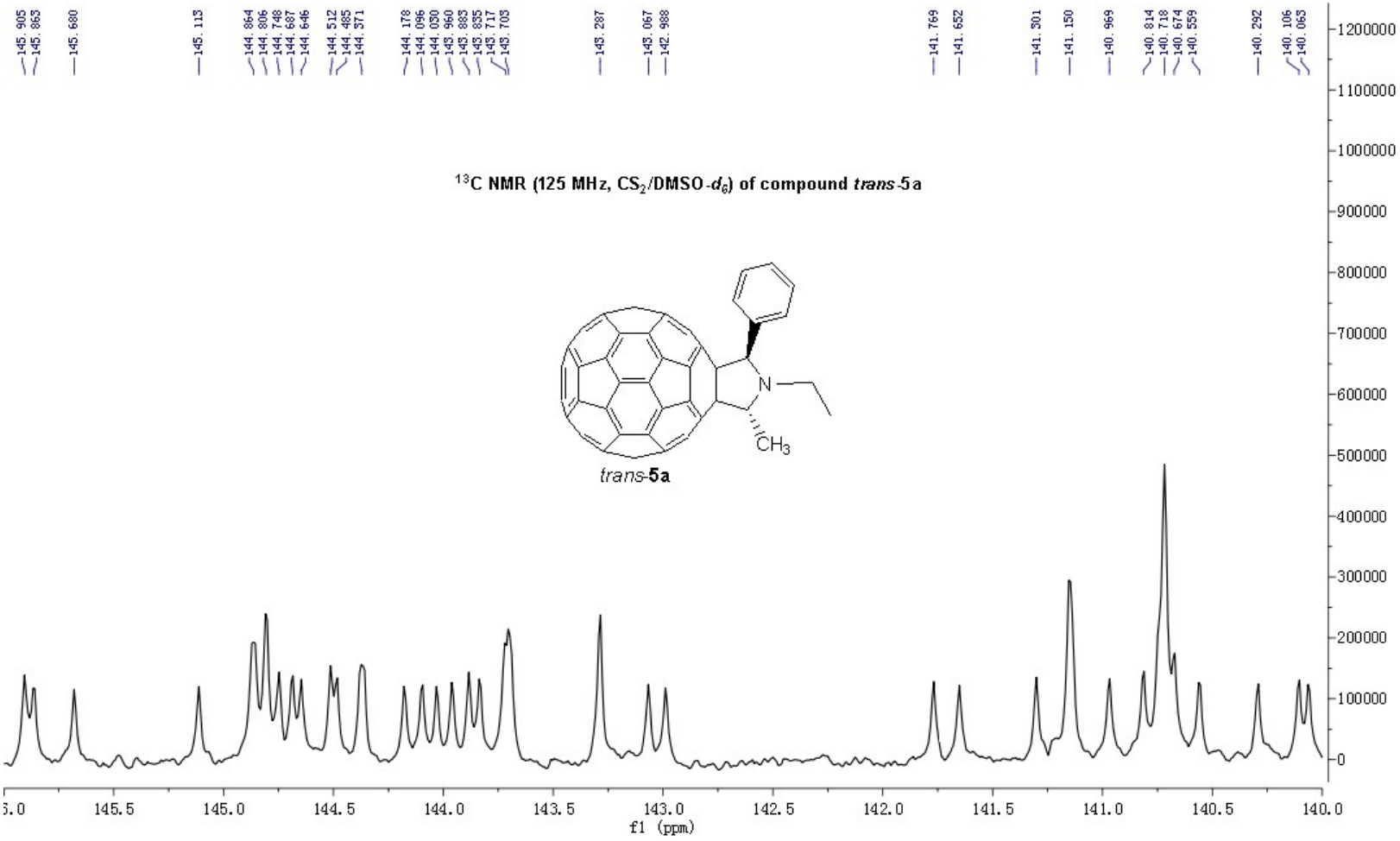

7.3930
7.3779
7.2662
7.2509
7.2354
7.1931
7.1787
7.1641
6.9671
6.9359
6.8555
6.8365
6.8245
6.8052
5.3069
5.2881
5.0630
5.0499
5.0369
5.0244
3.3098
3.2951
3.2803
3.2708
3.2560
3.2412
3.0852
3.0215
3.0108
2.9931
2.9758
2.4191
2.4158
2.4133
1.9455
1.9304
1.5203
1.5062
1.4918
1.2255
0.0000
^{1}H NMR (500 MHz, CS$_2$/DMSO-d_6) of compound *trans*-4a
N
CH$_3$
trans-**4a**
2.00
2.26
1.06
1.18
0.99
0.98
1.00
1.07
1.06
3.03
3.12
f1 (ppm)


13C NMR (125 MHz, CS2/DMSO-d6) of compound trans-4a
trans-4a
f1 (ppm)


146.365
146.340
145.860
145.753
145.709
145.353
145.300
145.124
145.069
144.800
144.745
144.570
144.447
144.404
144.355
144.339
144.249
143.678
143.621
142.201
141.751
141.677
141.423
141.353
141.299
141.222
141.210
141.186
140.948
140.889
140.865
140.782
^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *trans*-**4a**
N
CH_3
trans-**4a**
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
146.0
145.5
145.0
144.5
144.0
143.5
143.0
142.5
142.0
141.5
141.0
140.5
f1 (ppm)

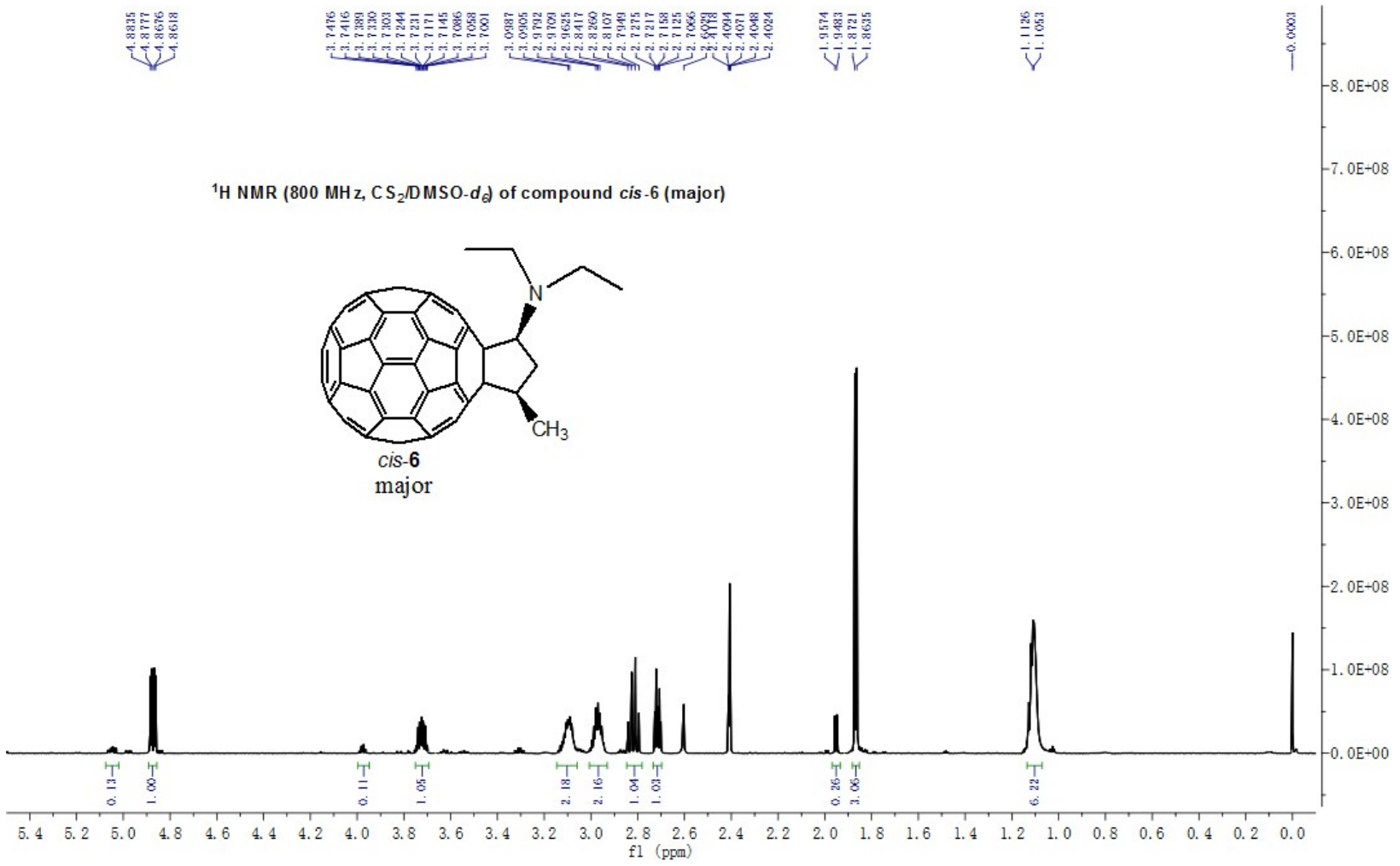

¹H NMR (500 MHz, CS2/DMSO-d6) of compound trans-5a
trans-5a
f1 (ppm)


^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *trans*-**5a**
trans-**5a**
f1 (ppm)



^{13}C NMR (125 MHz, CS_2/DMSO-d_6) of compound *trans*-**5a**

trans-**5a**


1H NMR (800 MHz, CS2/DMSO-d6) of compound cis-6 (major)
N
CH3
cis-6
major
f1 (ppm)