

# **CHEMISTRY**

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## **A EUROPEAN JOURNAL**

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### Supporting Information

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#### **Using a Threading-Followed-by-Swelling Approach to Synthesize [2]Rotaxanes**

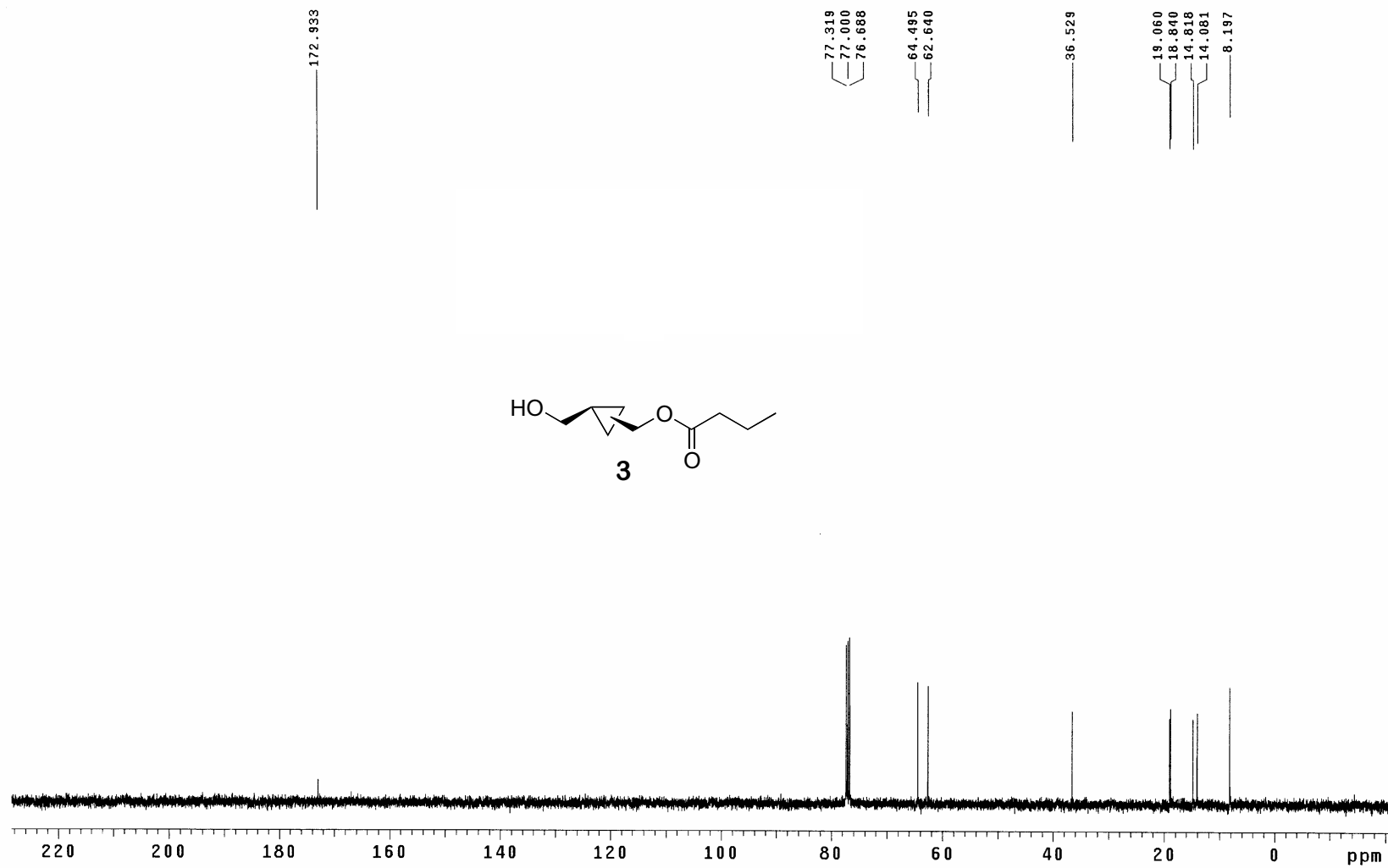
**Jia-Ling Ko,<sup>[a]</sup> Shau-Hua Ueng,<sup>[a]</sup> Ching-Wei Chiu,<sup>[a]</sup> Chien-Chen Lai,<sup>[b]</sup> Yi-Hung Liu,<sup>[a]</sup>  
Shie-Ming Peng,<sup>[a]</sup> and Sheng-Hsien Chiu\*<sup>[a]</sup>**

chem\_201000074\_sm\_miscellaneous\_information.pdf

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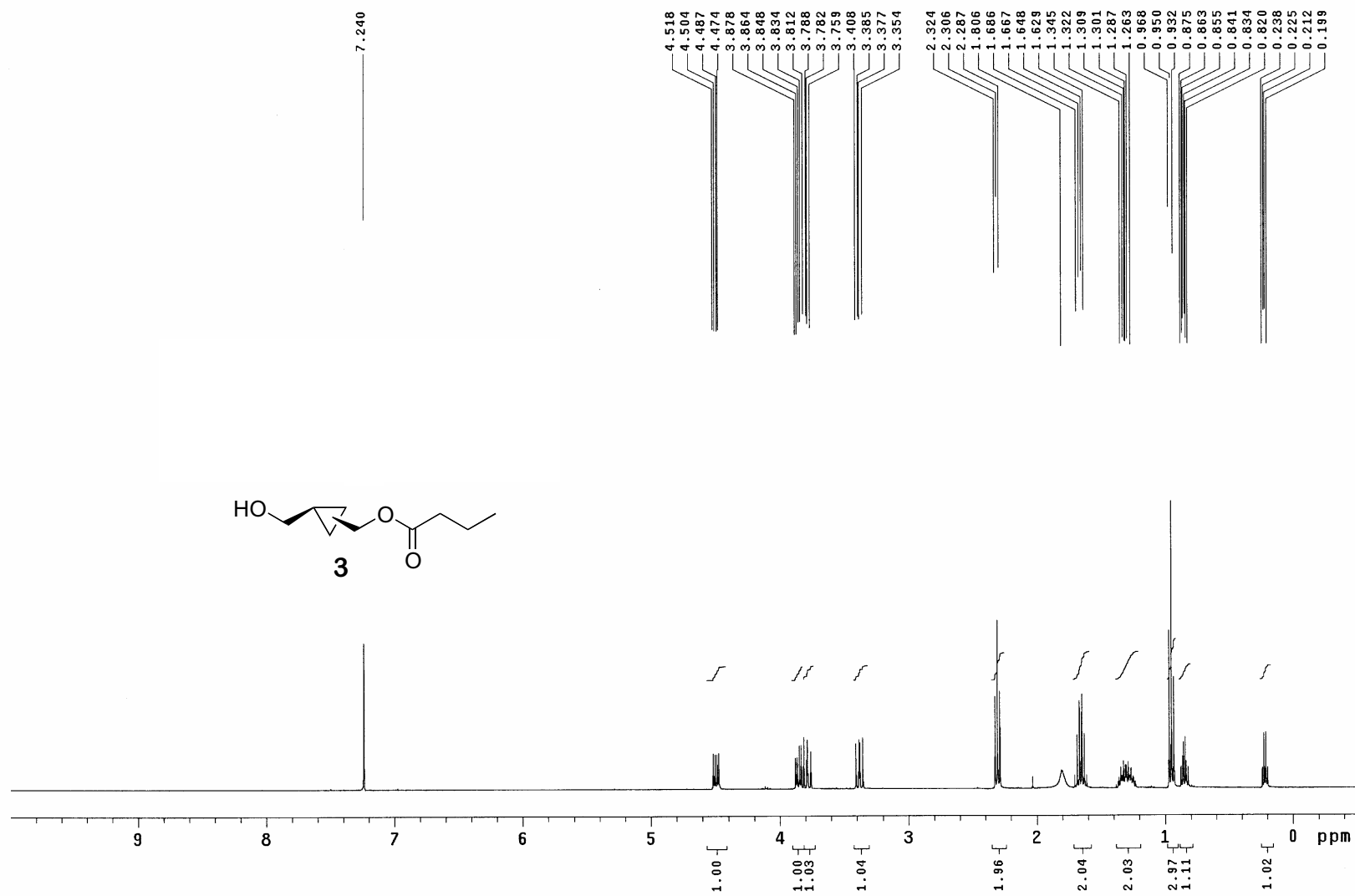
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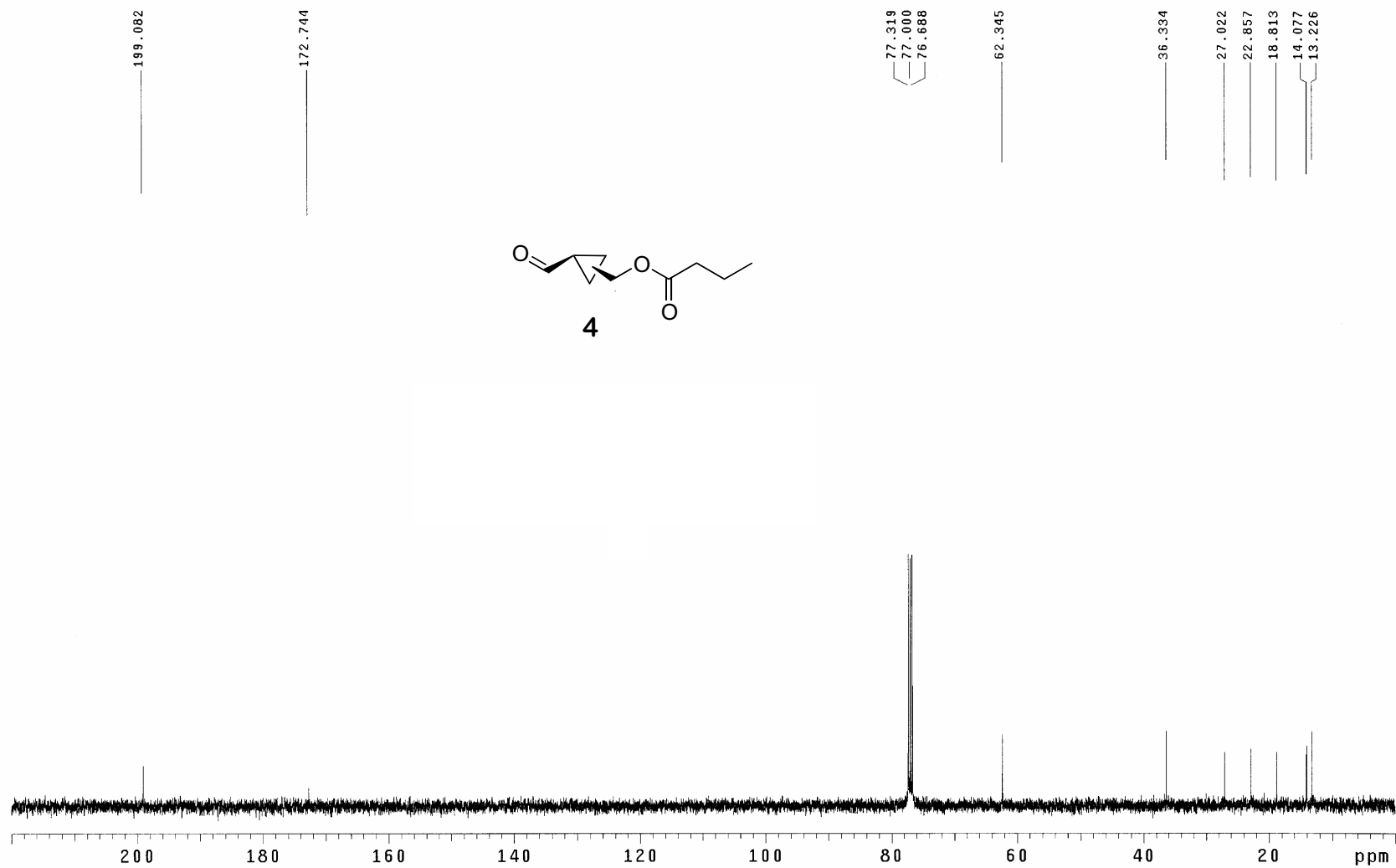
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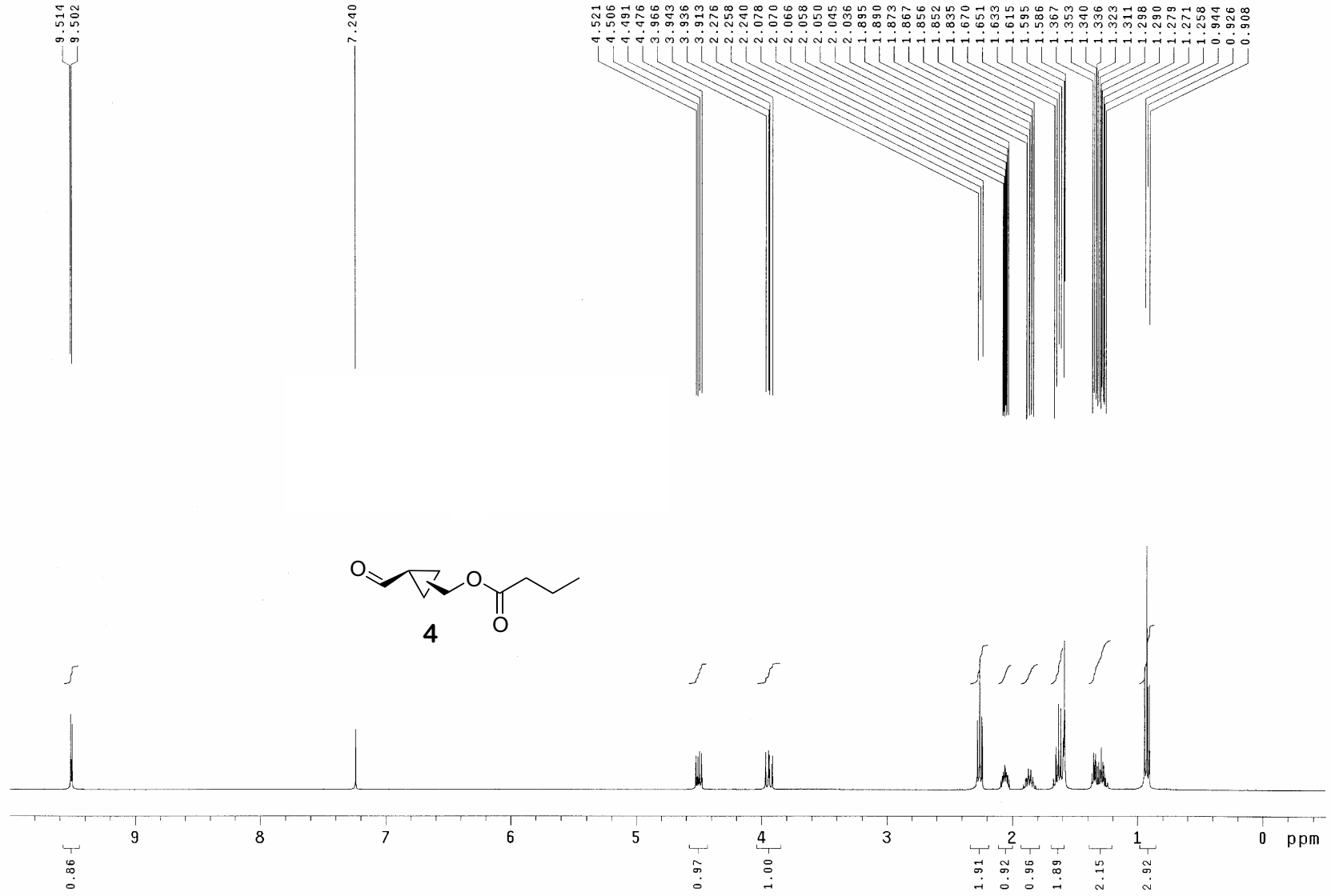
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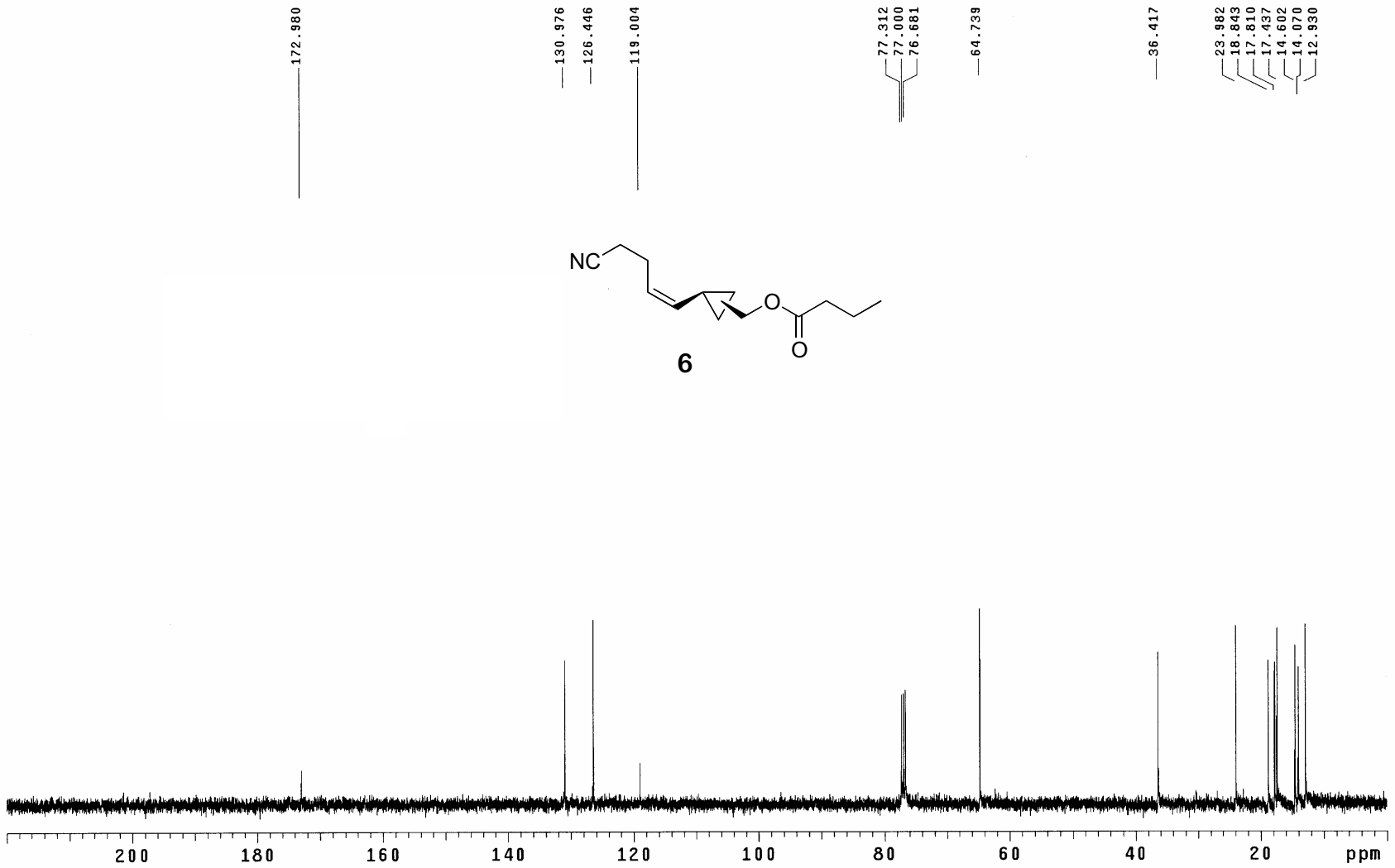
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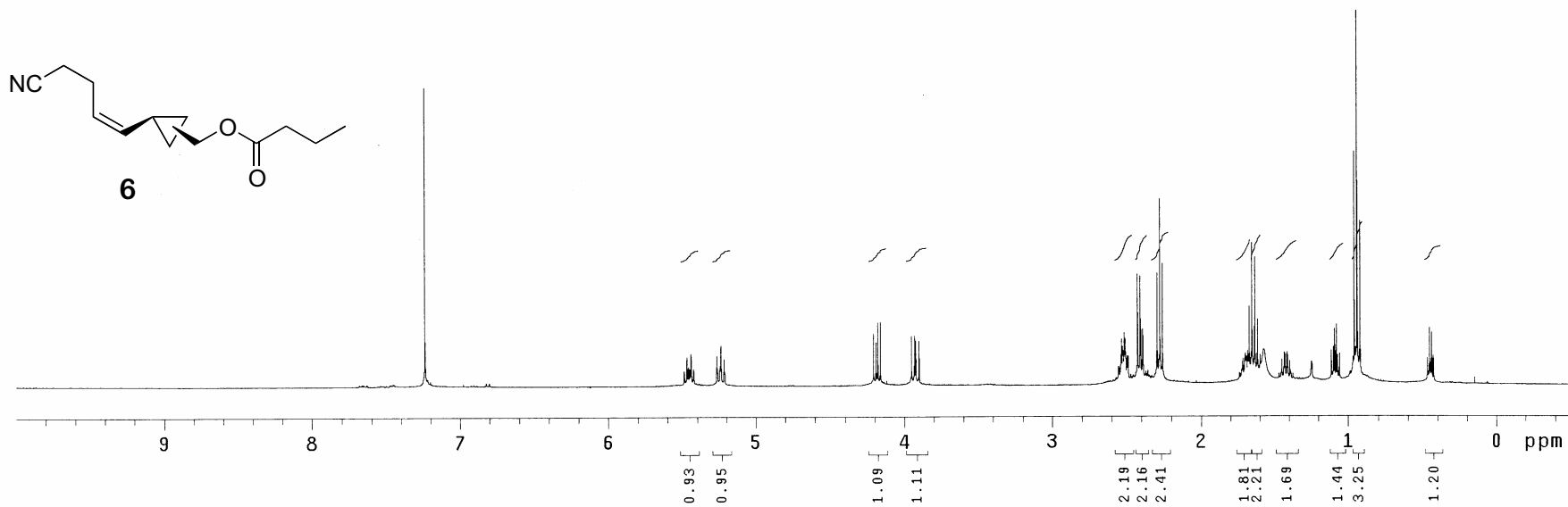
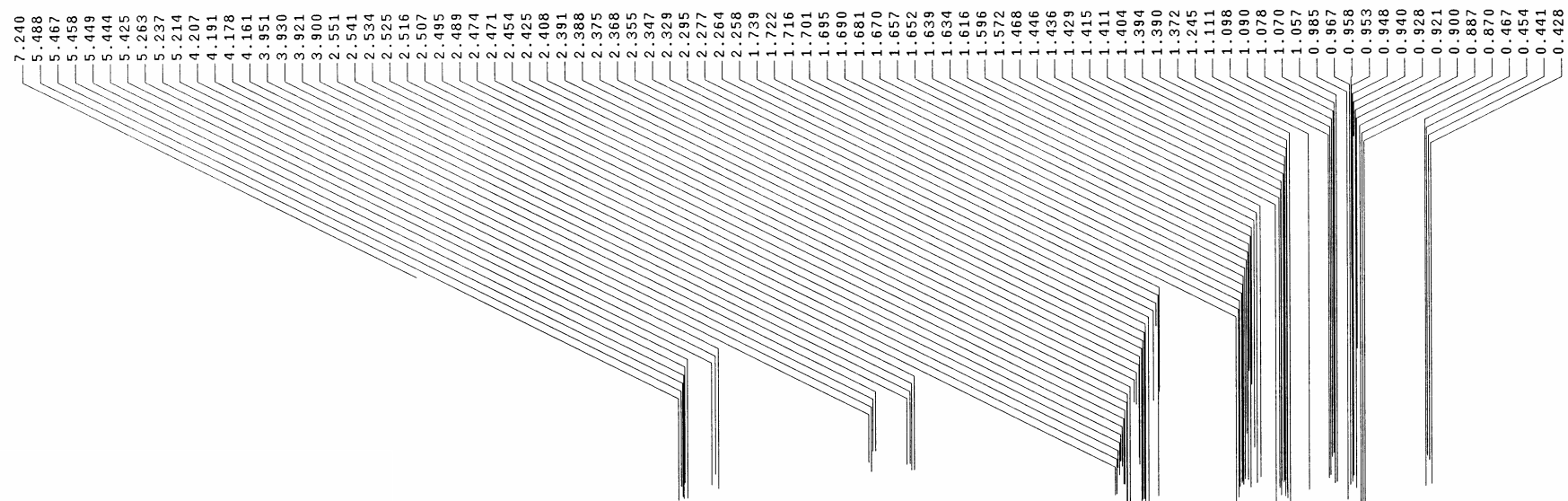
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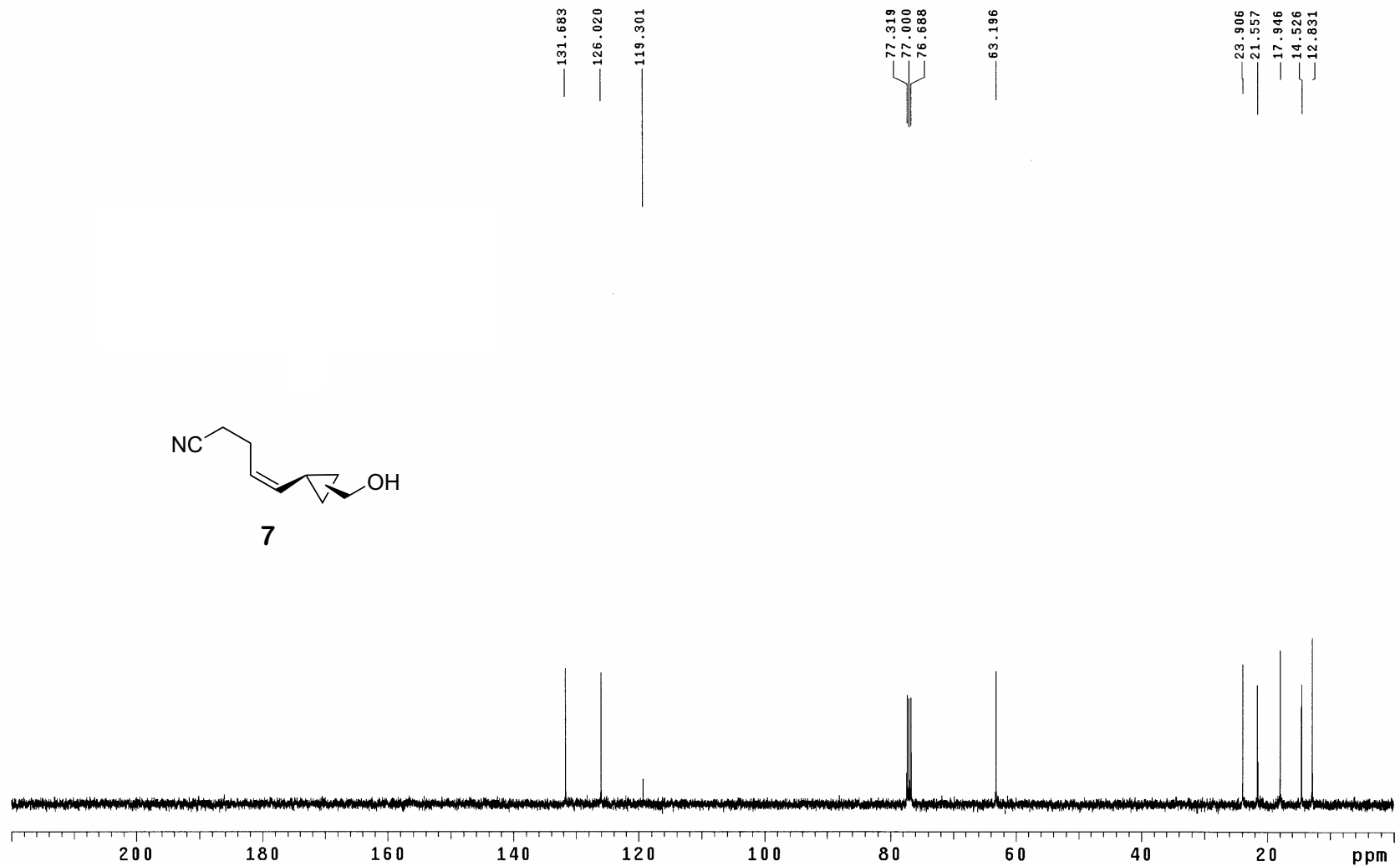
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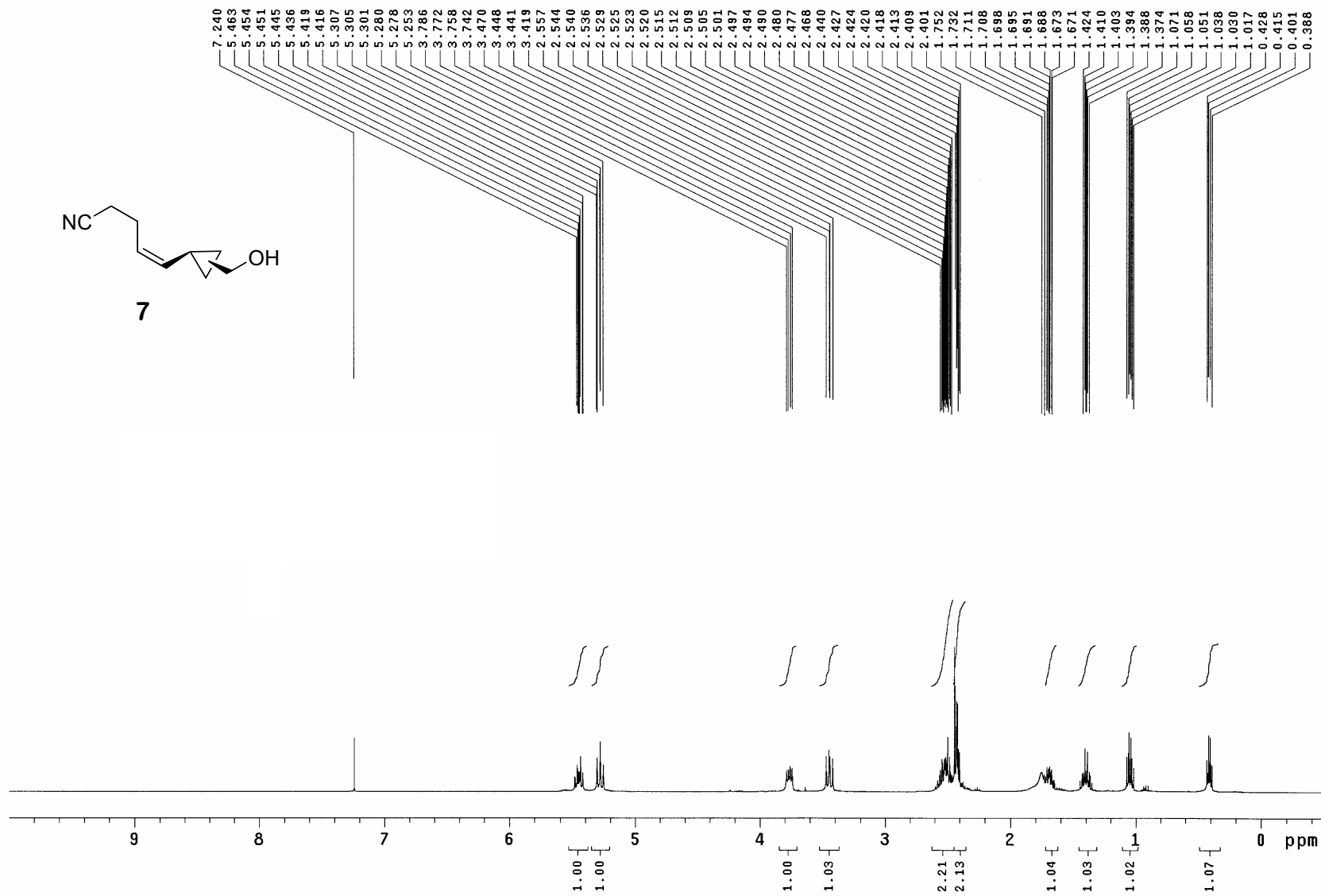
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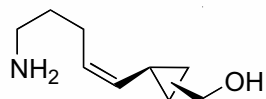
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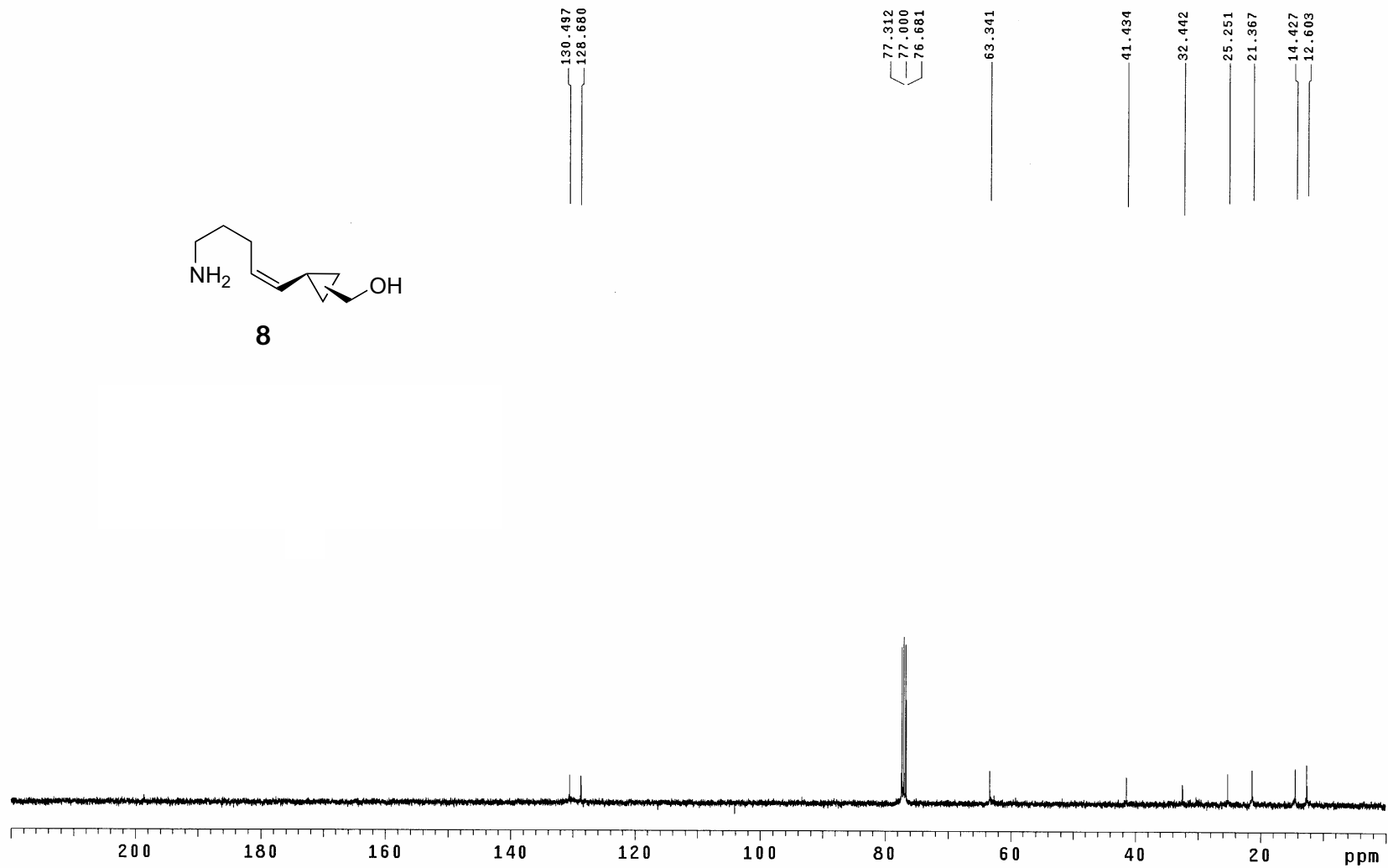


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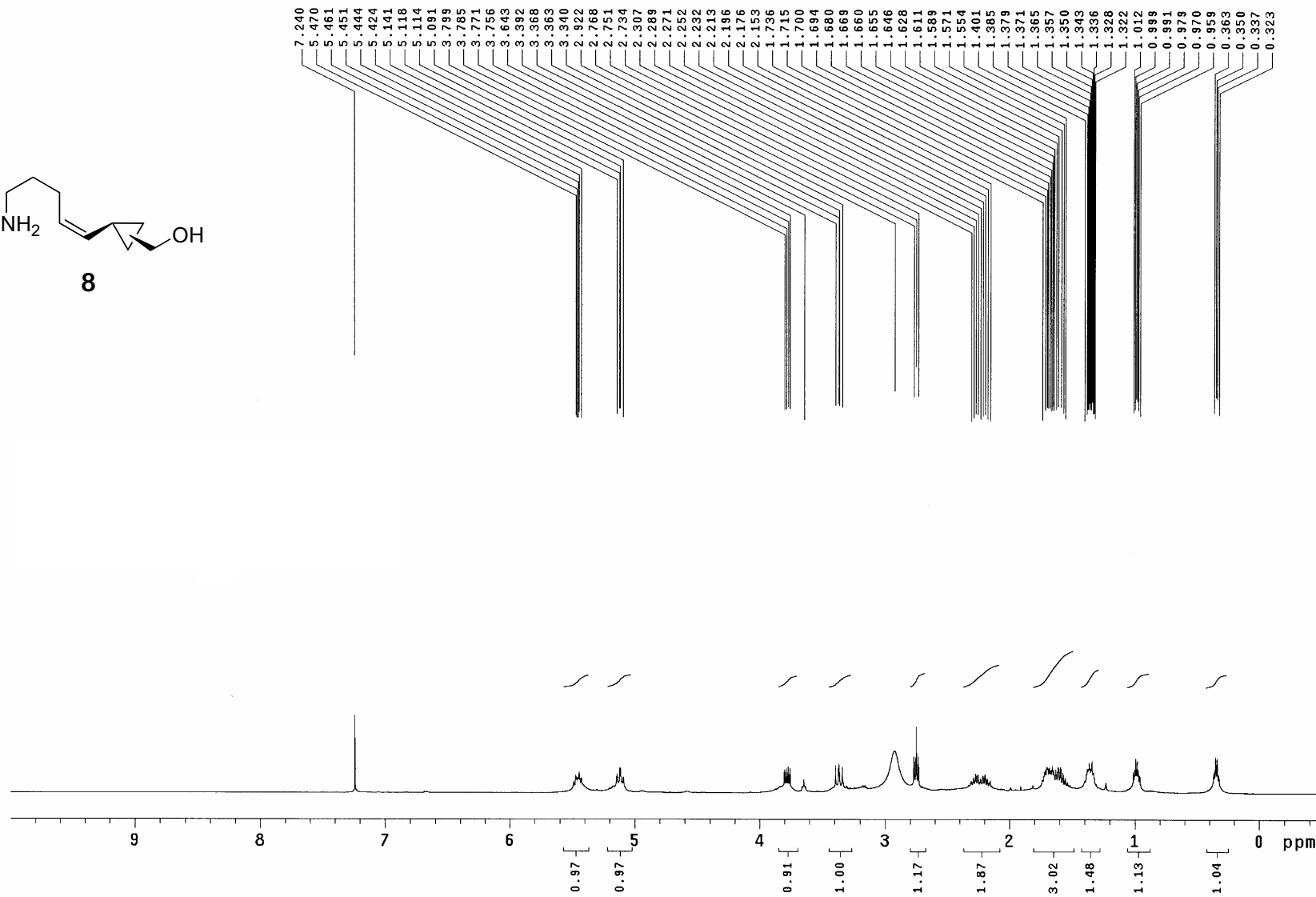
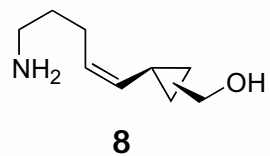


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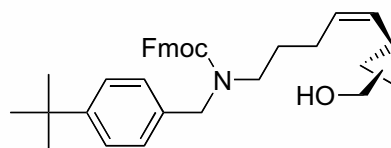
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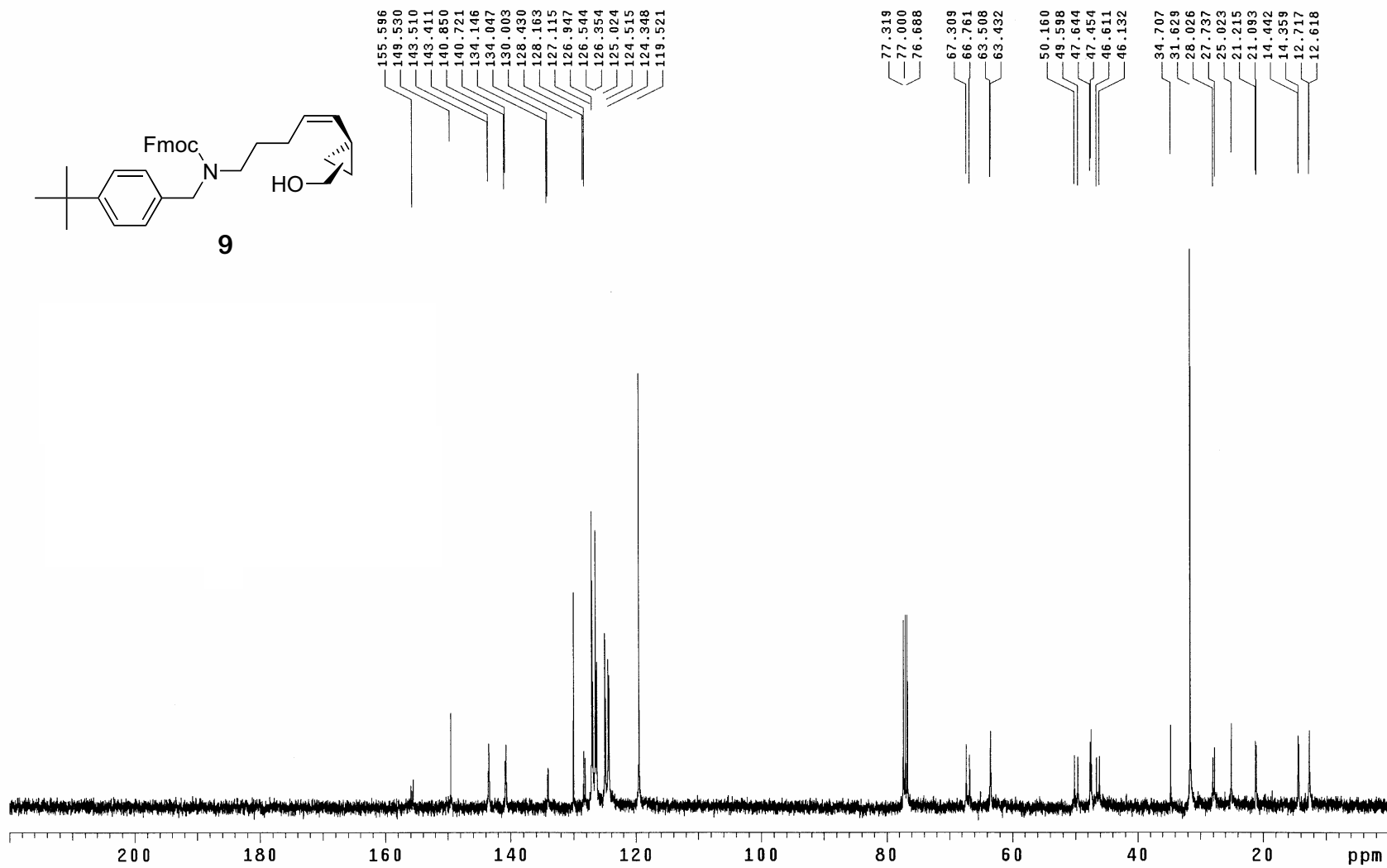


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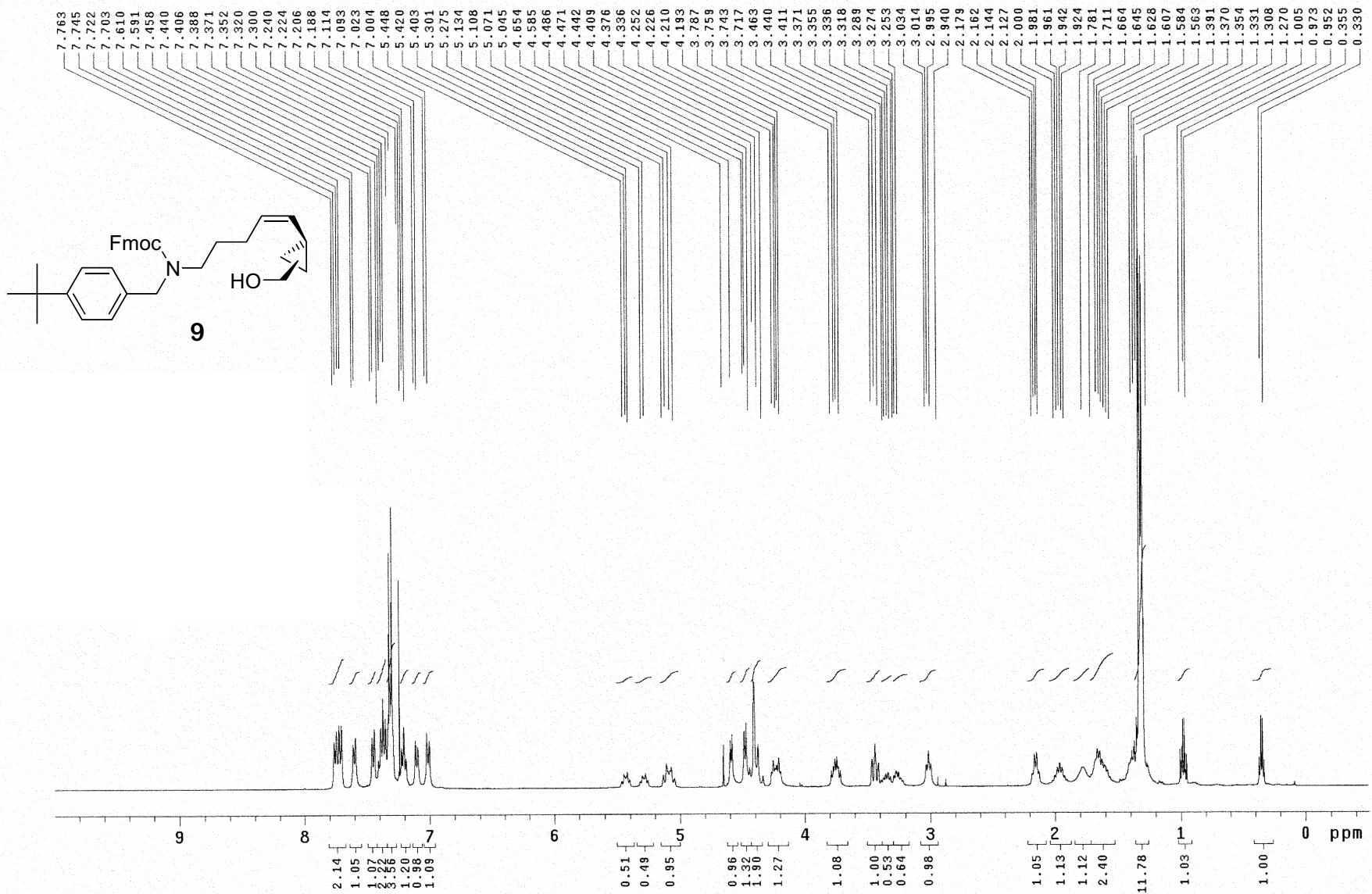


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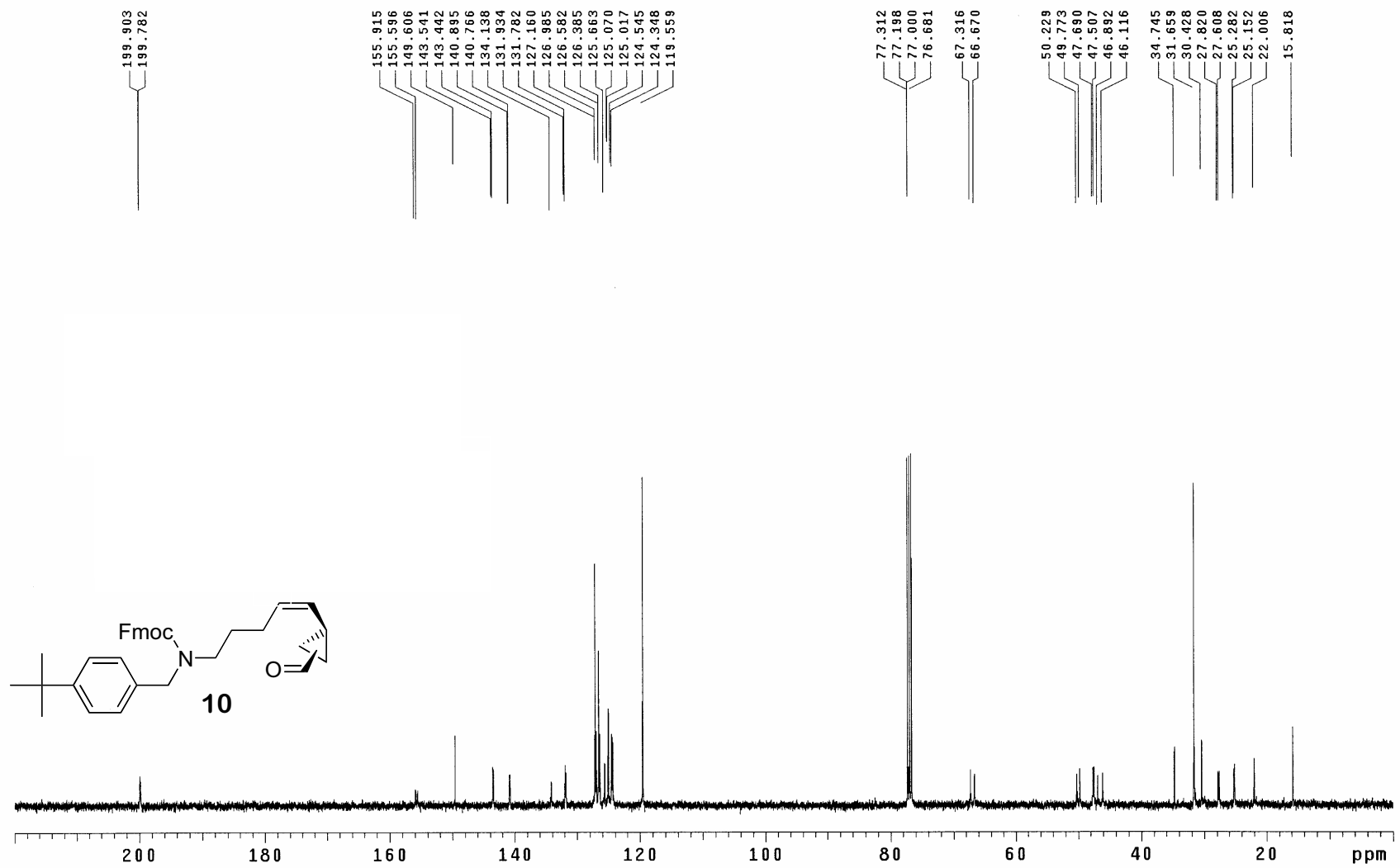
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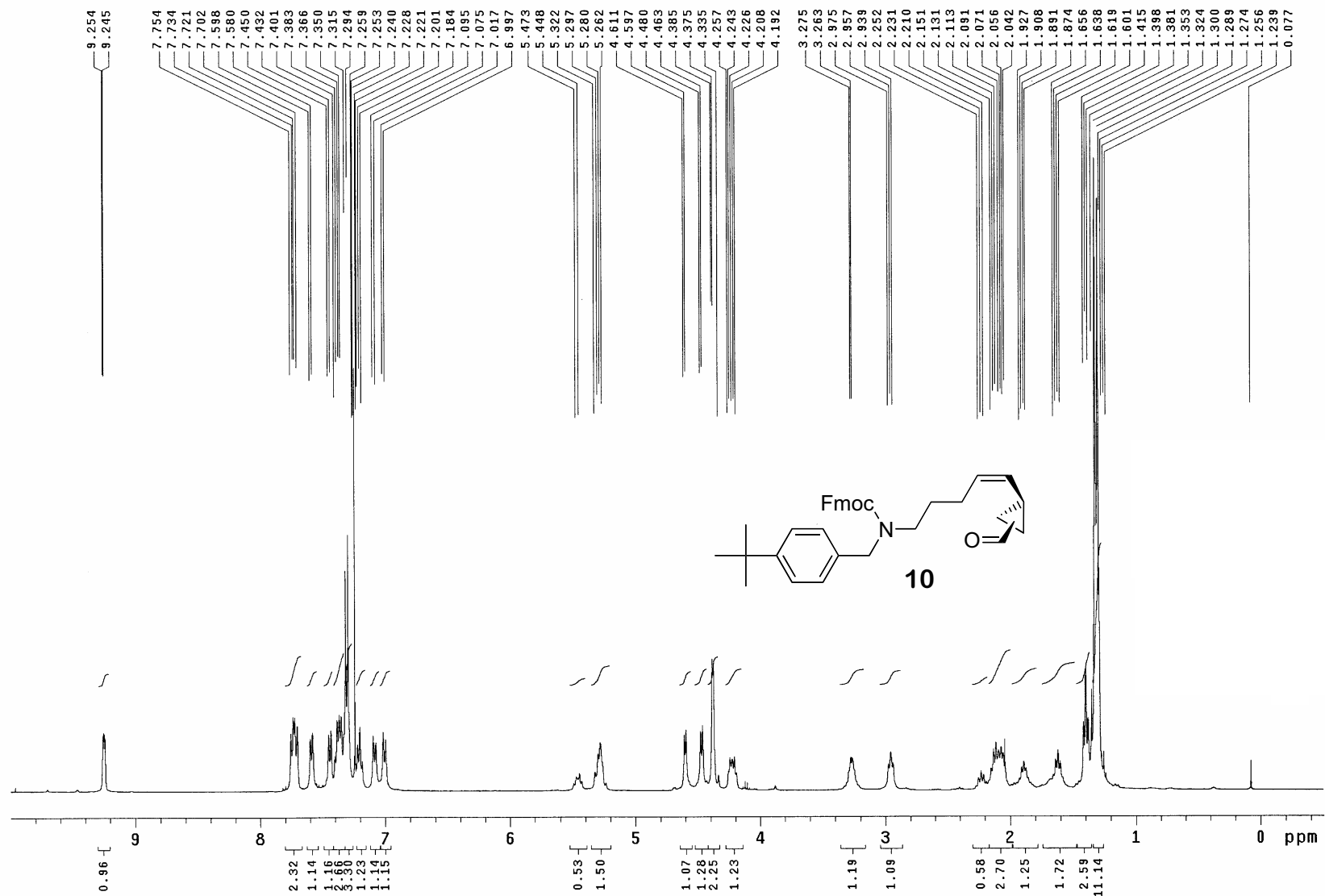
13C OBSERVE

Pulse Sequence: s2pu1



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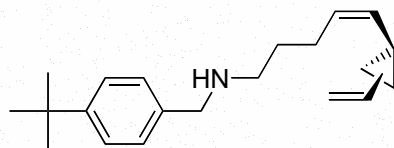
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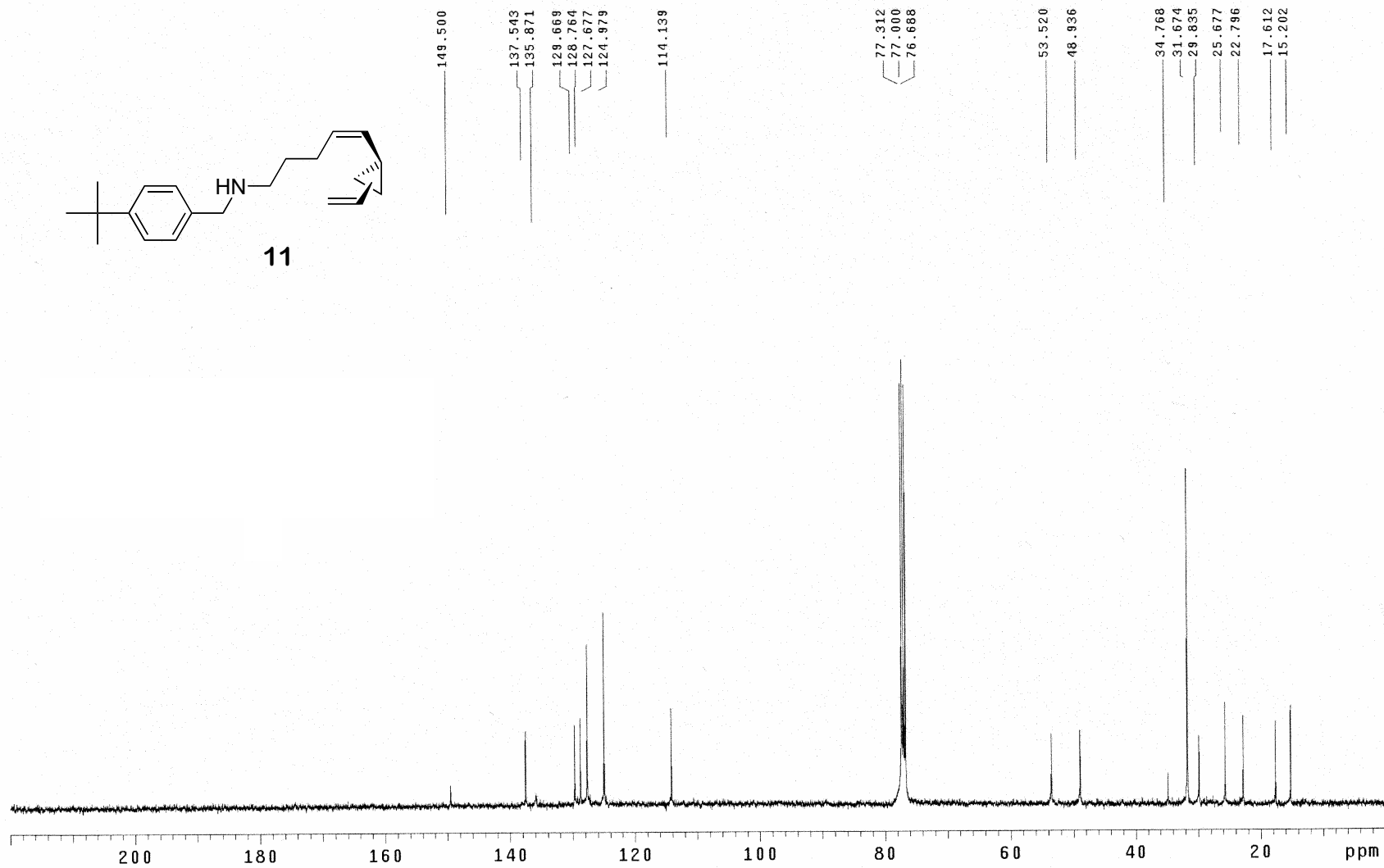


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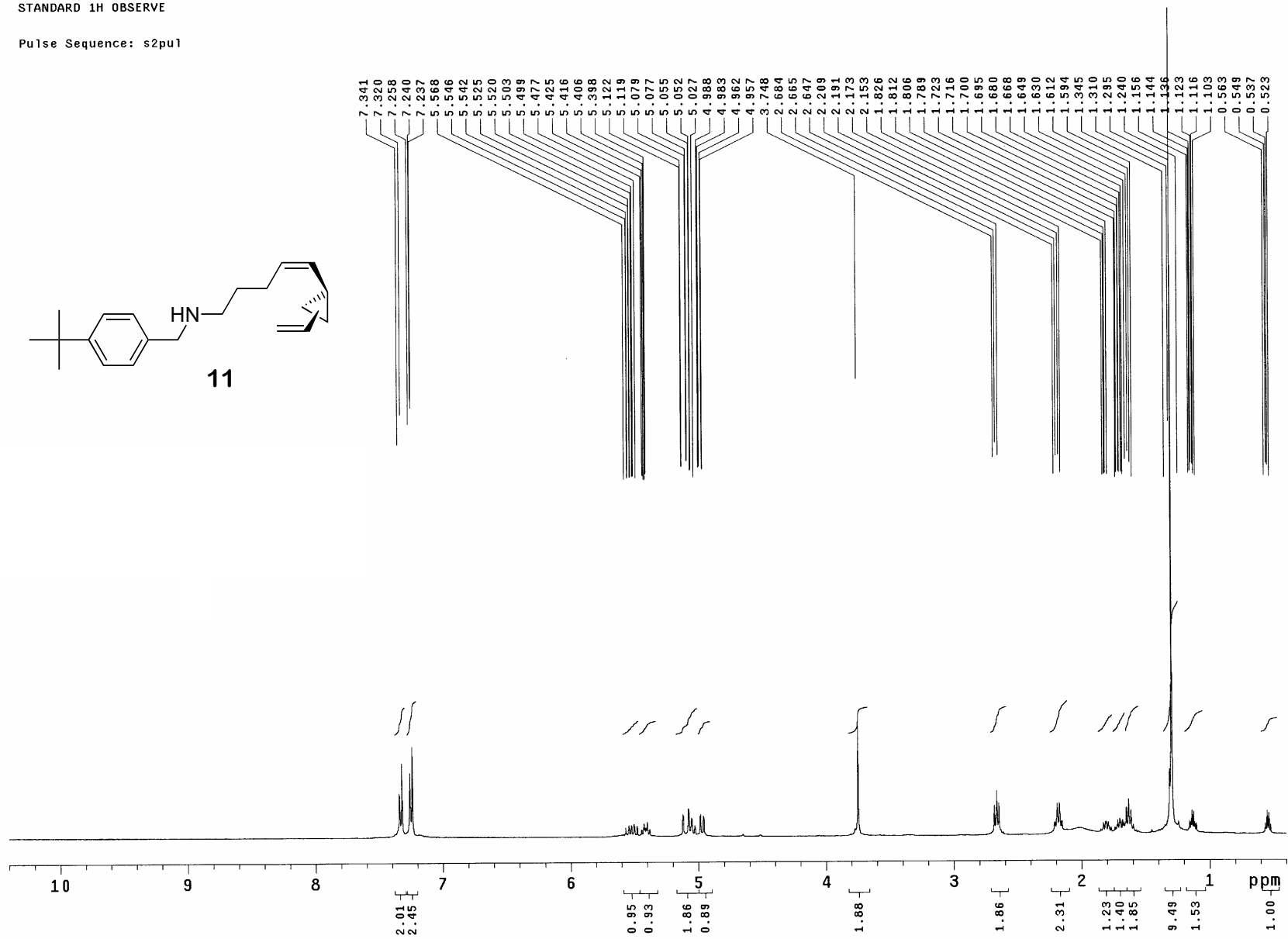


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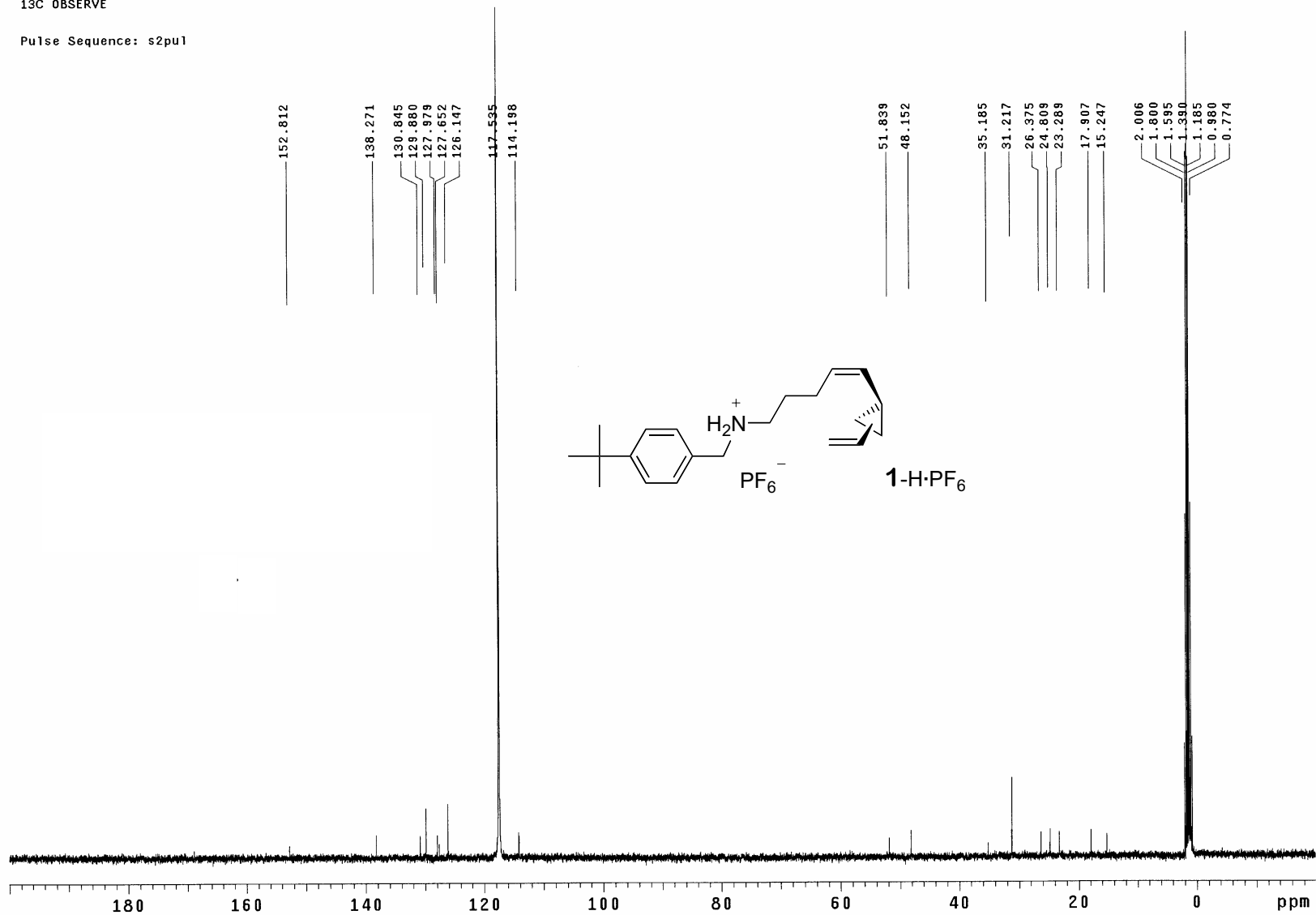
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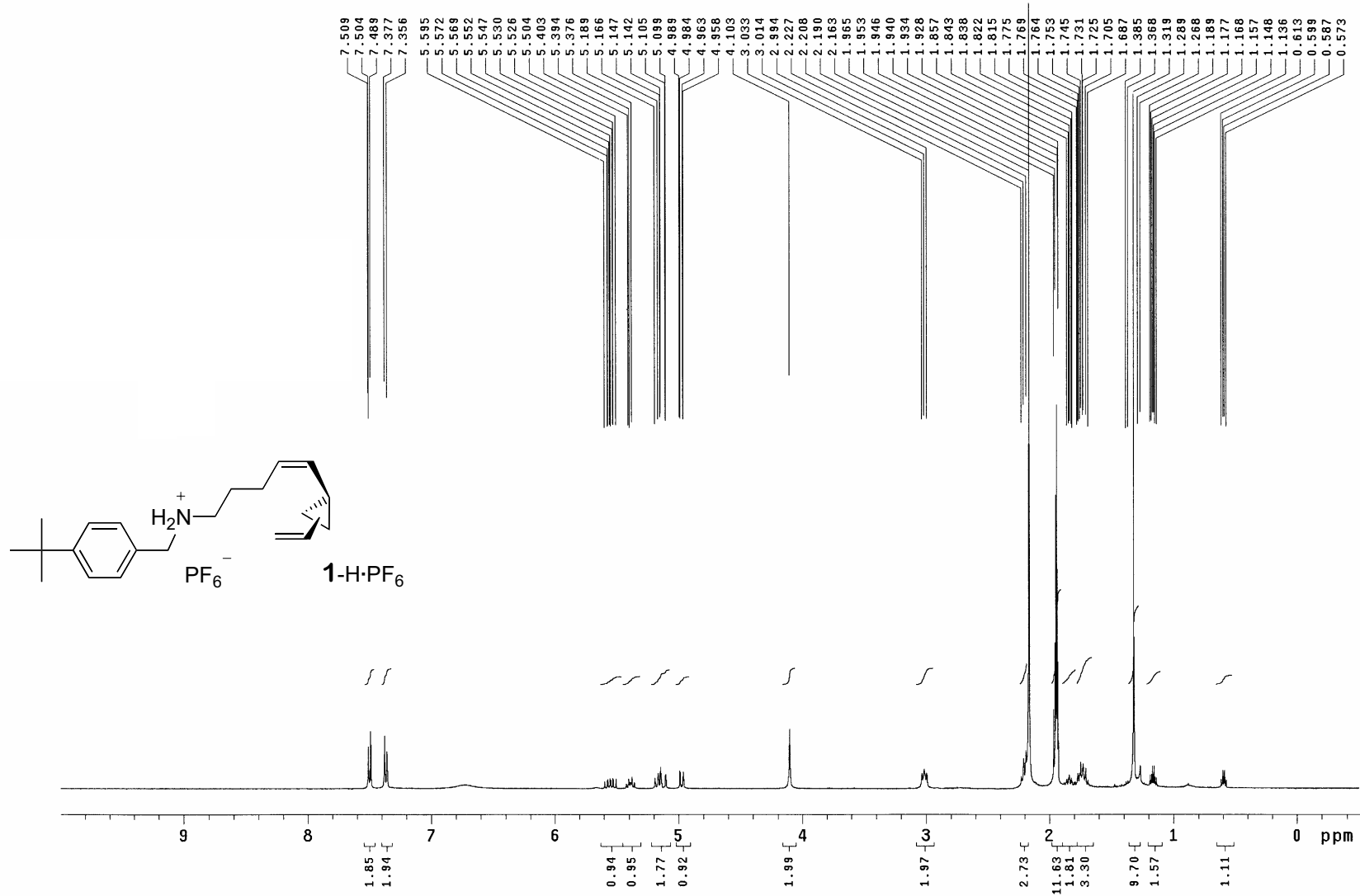
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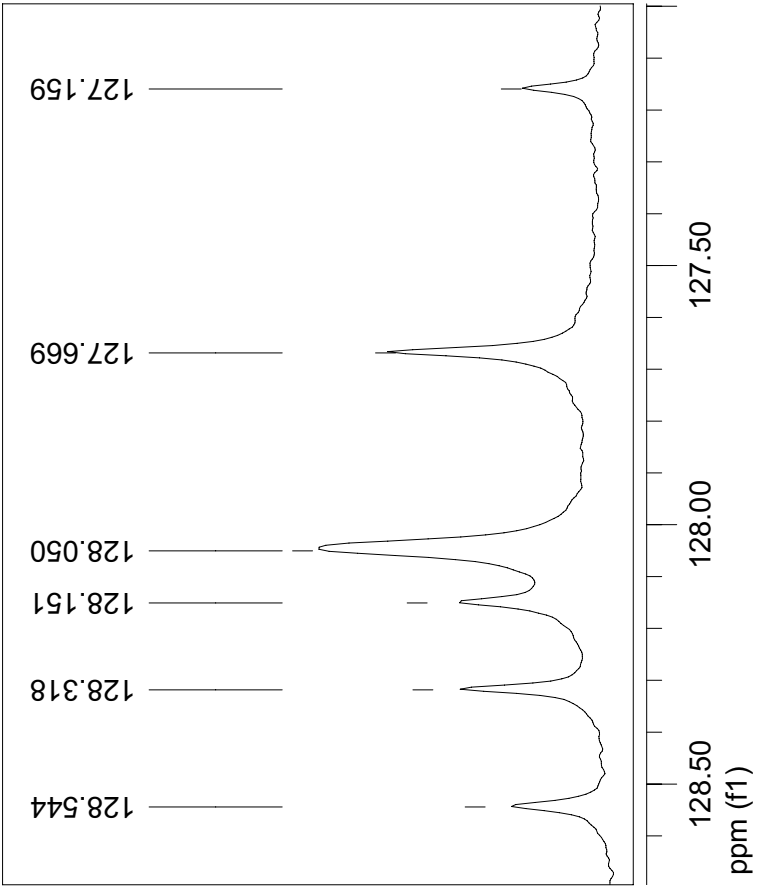
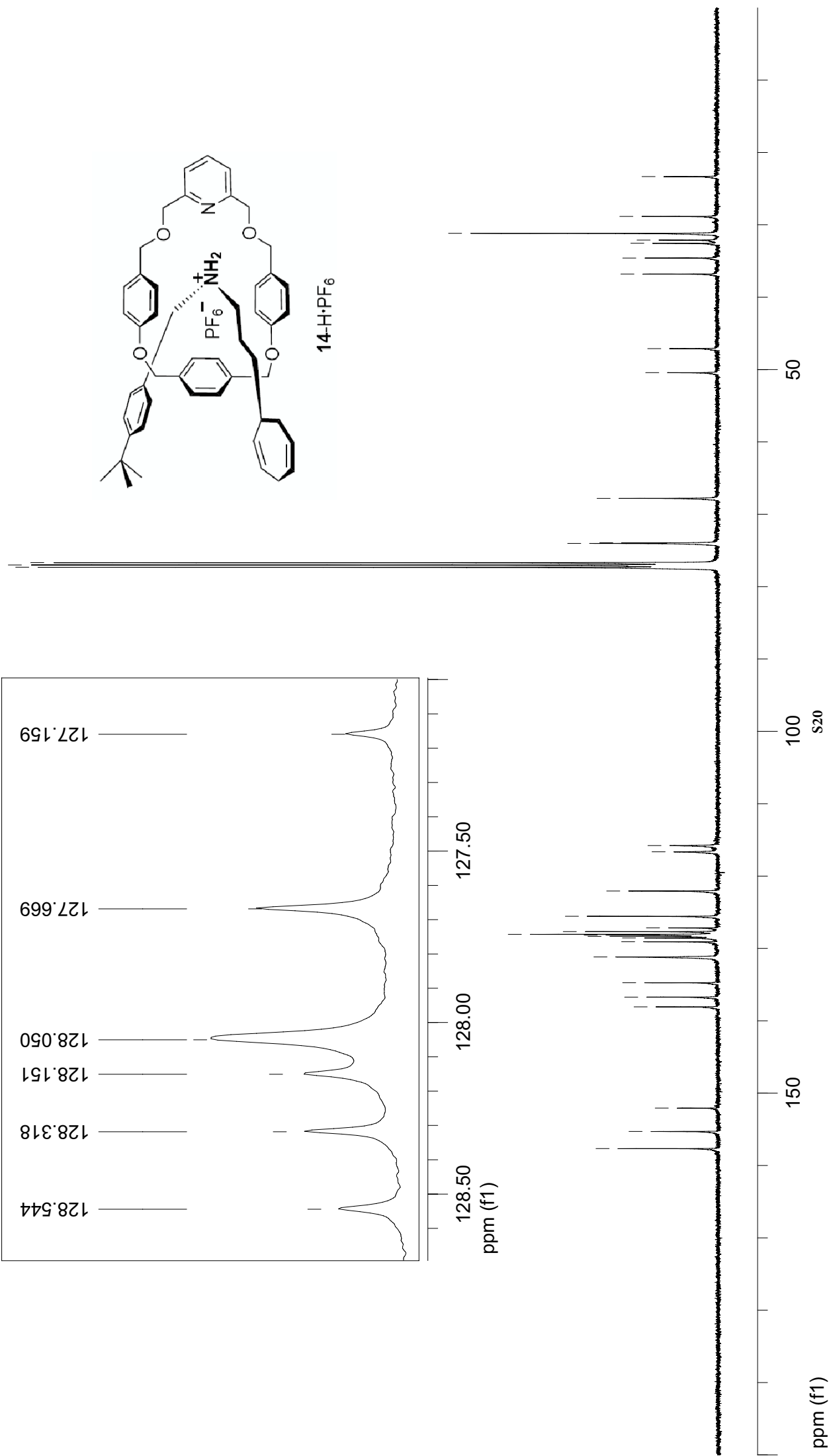
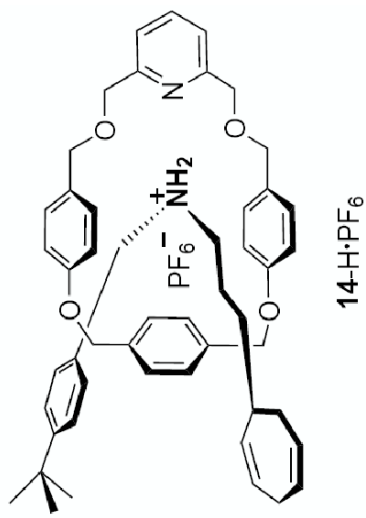
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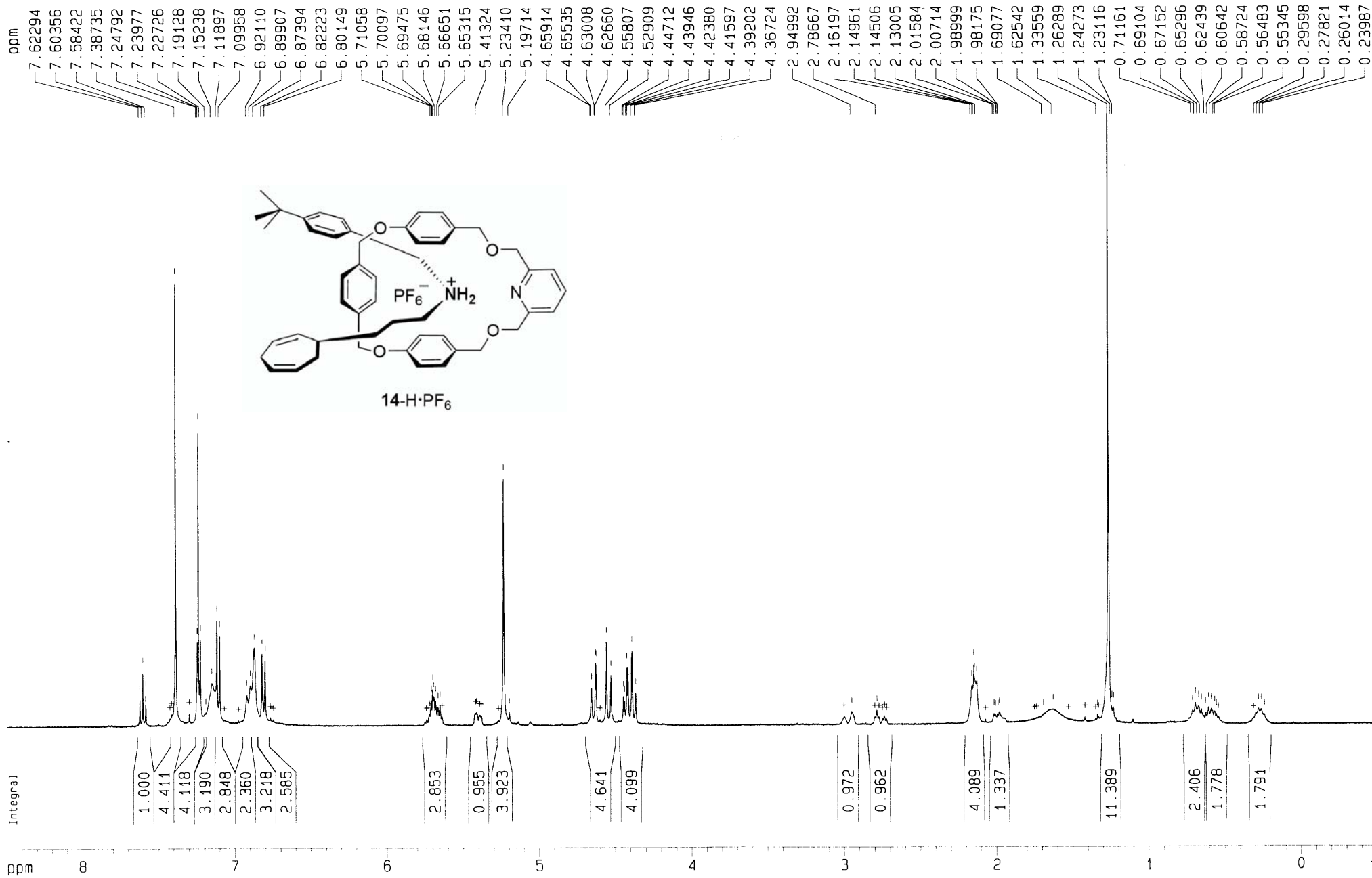
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- 23.324
- 28.828
- 31.167
- 32.100
- 32.545
- 34.572
- 36.793
- 47.098
- 50.425
- 67.791
- 73.943
- 74.024
- 76.682
- 77.000
- 77.318

- 115.782
- 116.654
- 122.061
- 125.554
- 127.159
- 127.669
- 128.050
- 128.151
- 128.318
- 128.544
- 129.076
- 131.179
- 134.736
- 136.727
- 138.071
- 152.071
- 155.300
- 157.657



13C OBSERVE

Pulse Sequence: s2pu1

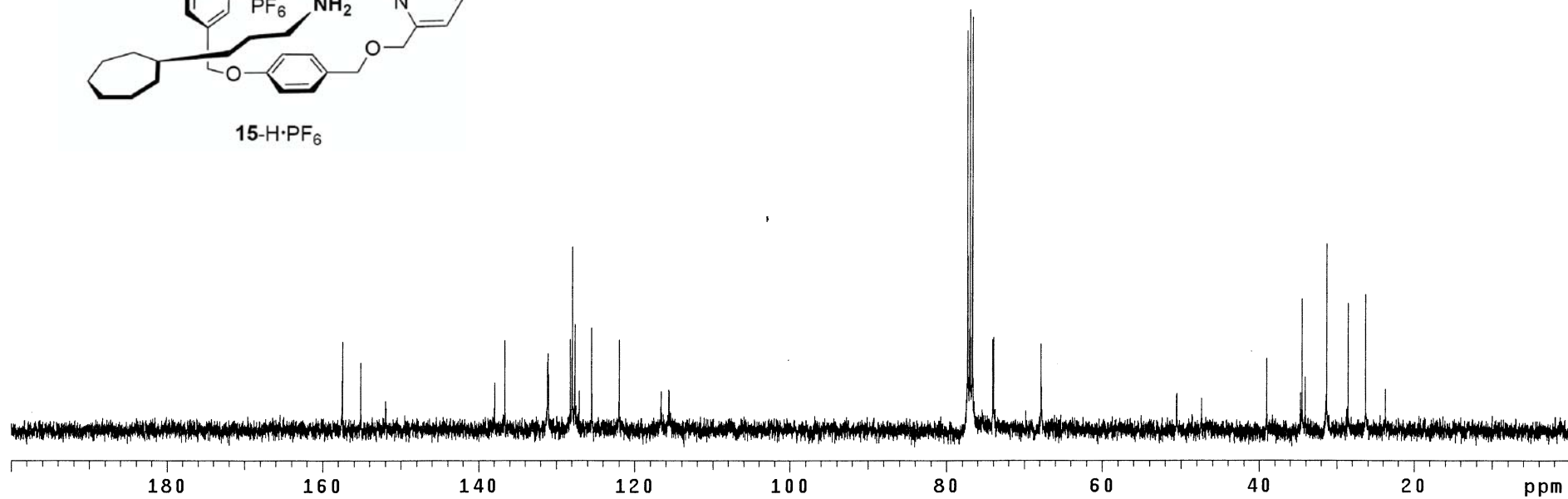
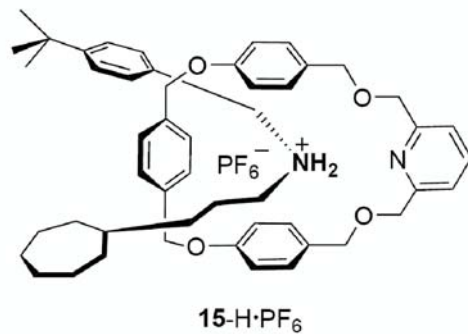
157.458  
155.117  
151.909

137.923  
136.586  
131.067  
128.201  
127.943  
127.609  
127.076  
125.465  
121.969  
116.602  
115.629

77.312  
77.000  
76.681  
74.058  
73.967  
67.818

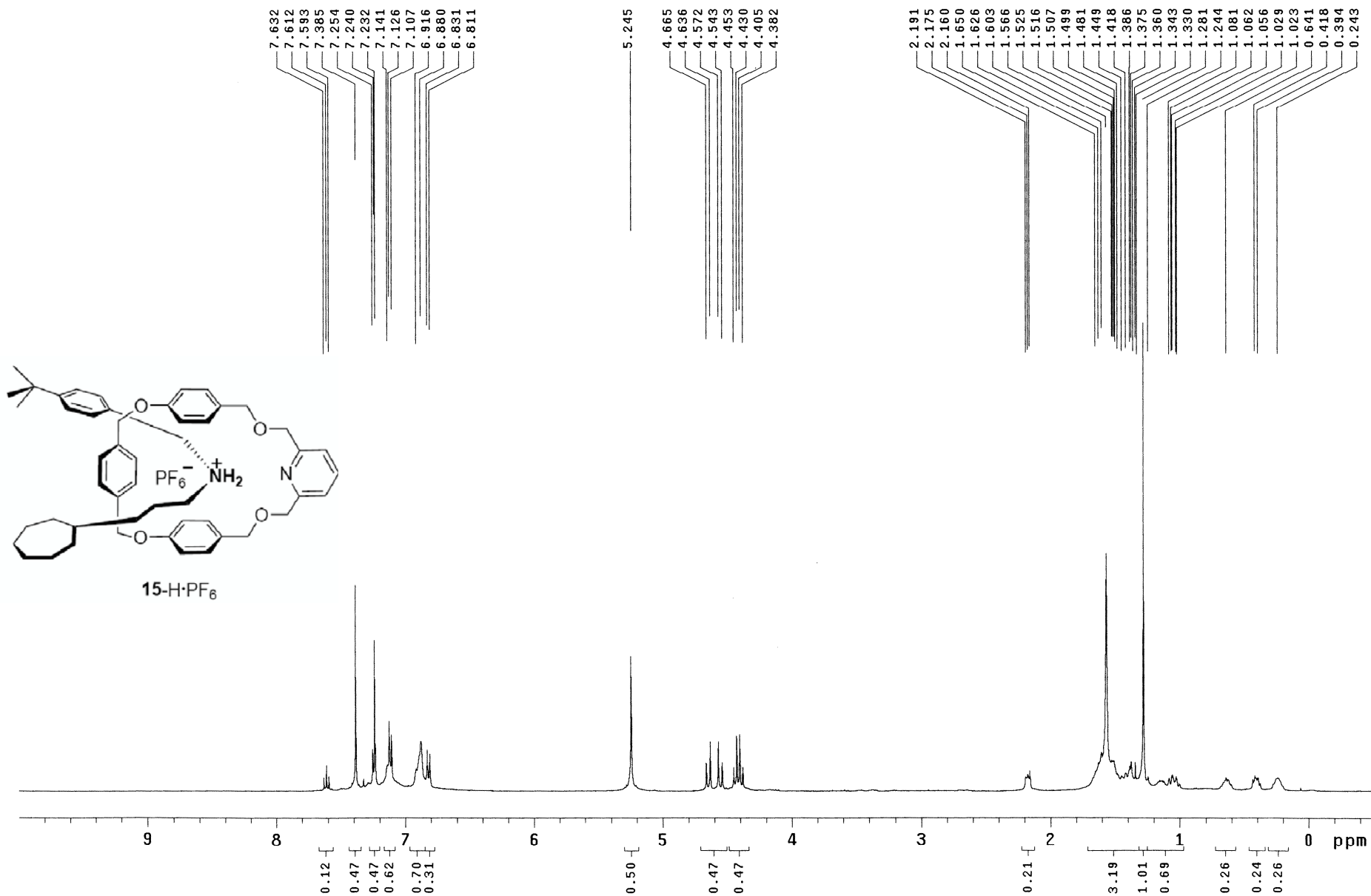
50.510  
47.348

39.032  
34.700  
34.471  
34.061  
31.302  
28.527  
26.255  
23.693



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1





13C OBSERVE

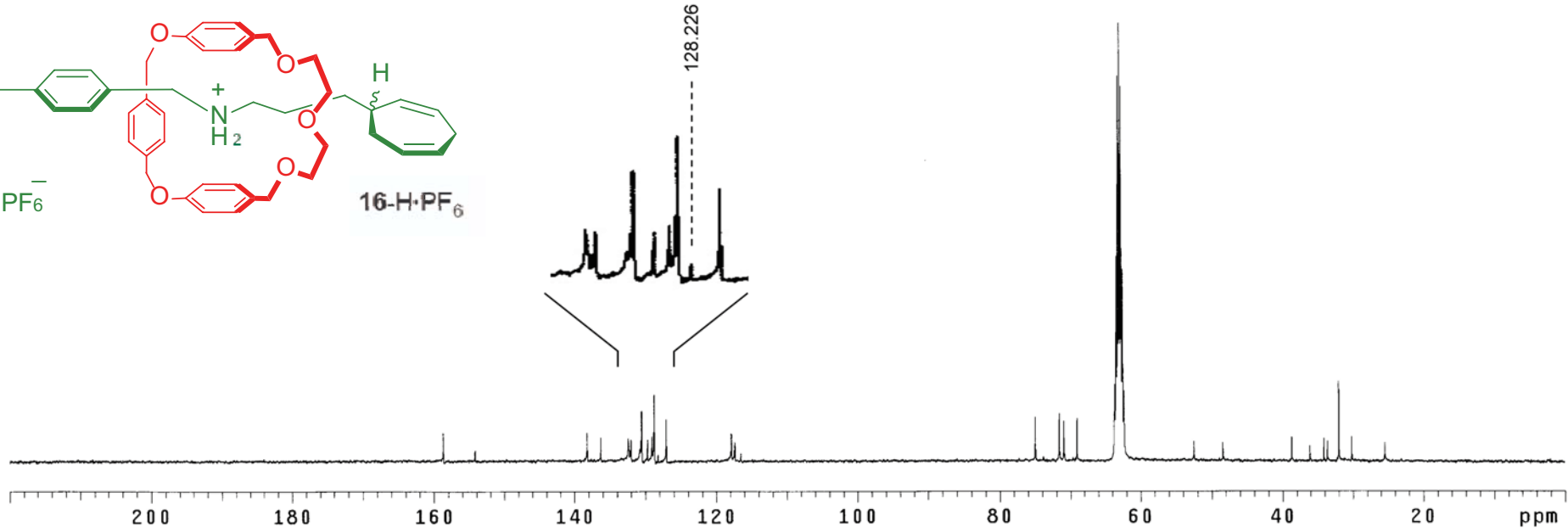
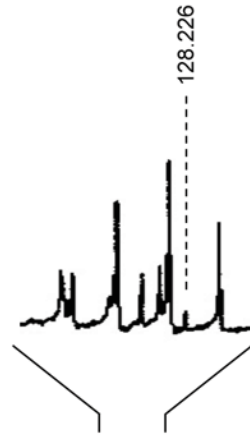
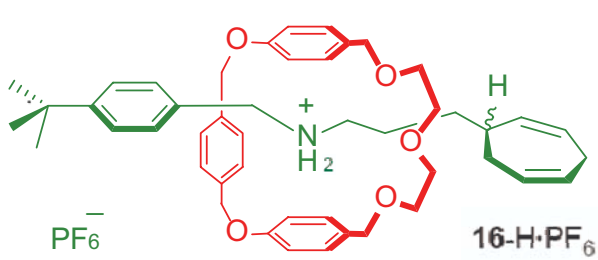
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158.691  
154.184

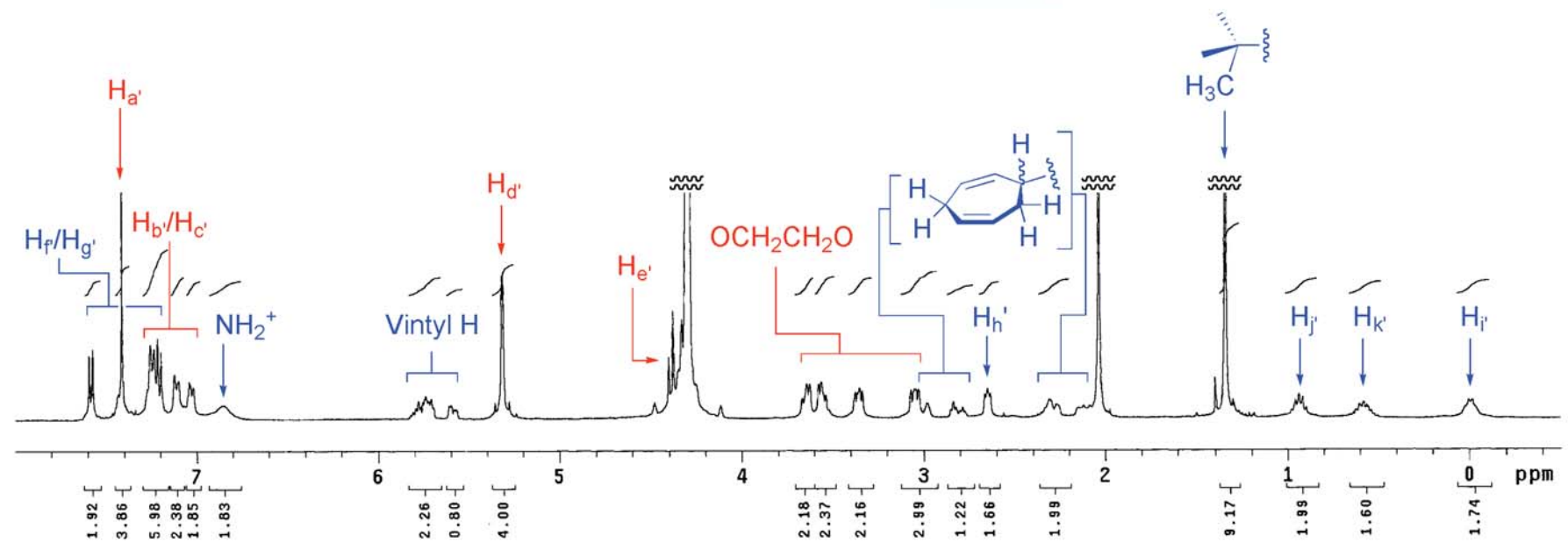
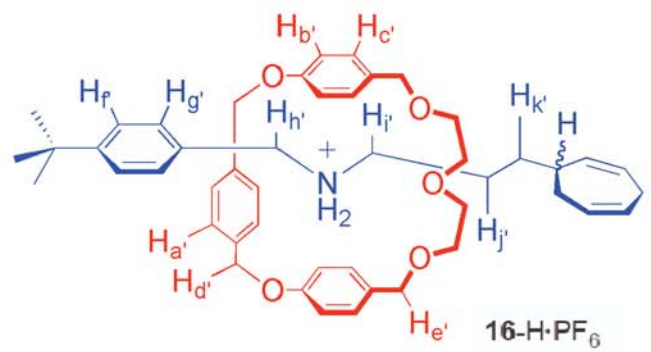
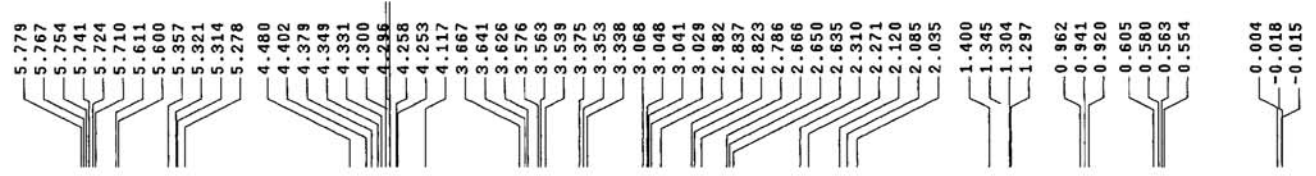
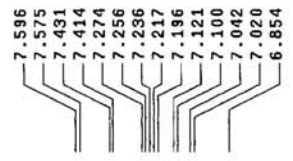
138.168  
136.252  
132.429  
132.057  
130.582  
130.506  
129.700  
129.100  
128.780  
127.055  
117.858  
117.379  
116.489

75.010  
71.597  
70.951  
69.066  
63.661  
63.441  
63.220  
63.000  
62.780  
62.559  
62.339  
52.472  
48.413

38.699  
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34.146  
33.622  
31.995  
30.163  
25.473

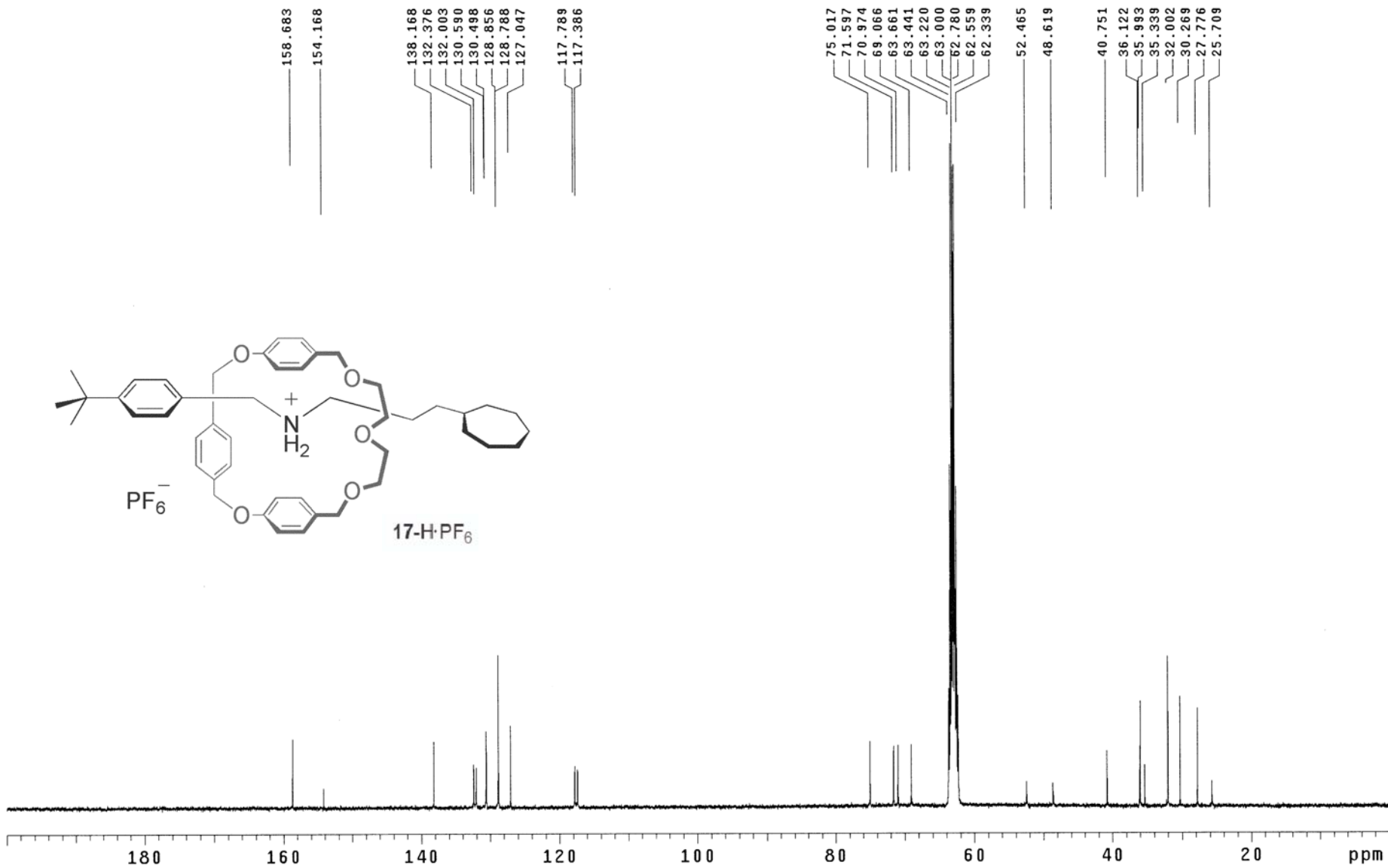


400MHz H<sup>1</sup> CD<sub>3</sub>NO<sub>2</sub>

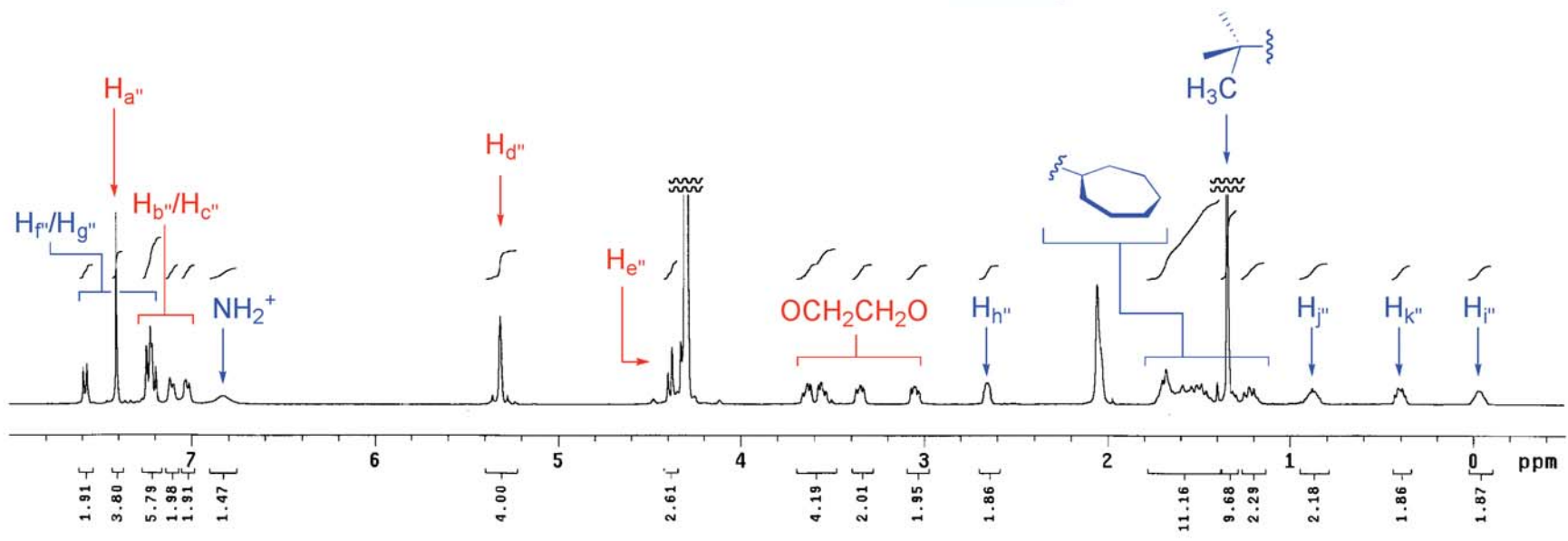
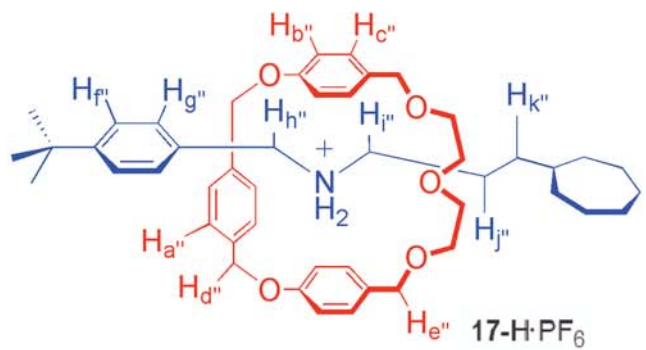
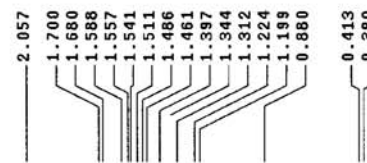
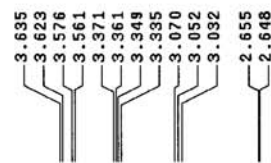
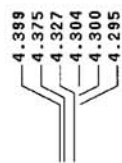
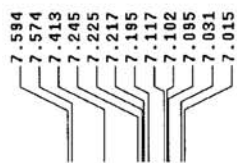


13C OBSERVE

Pulse Sequence: s2pu1

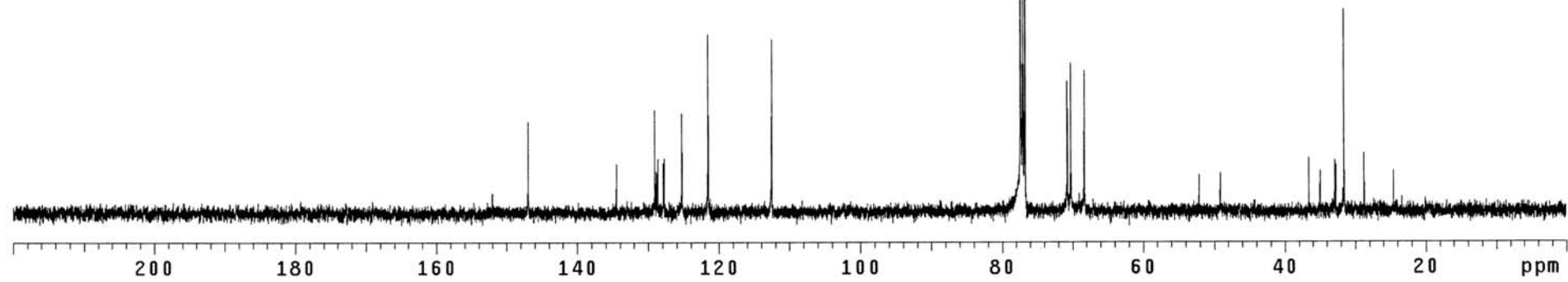
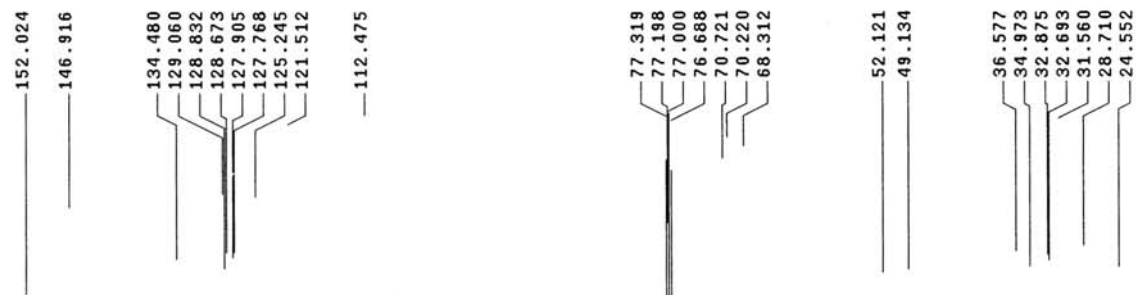
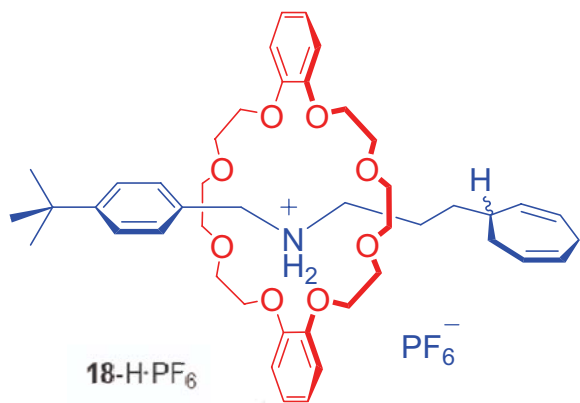


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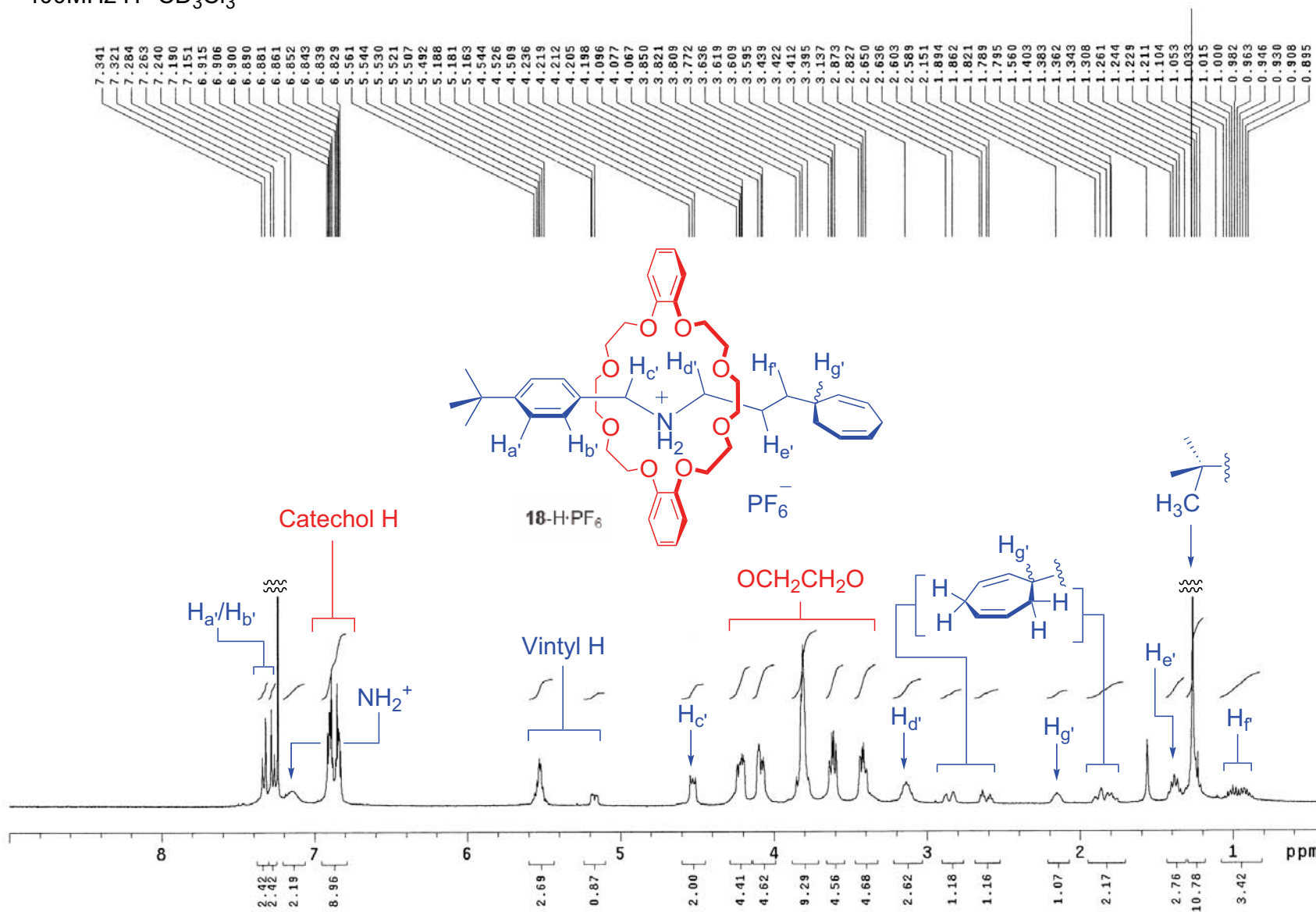


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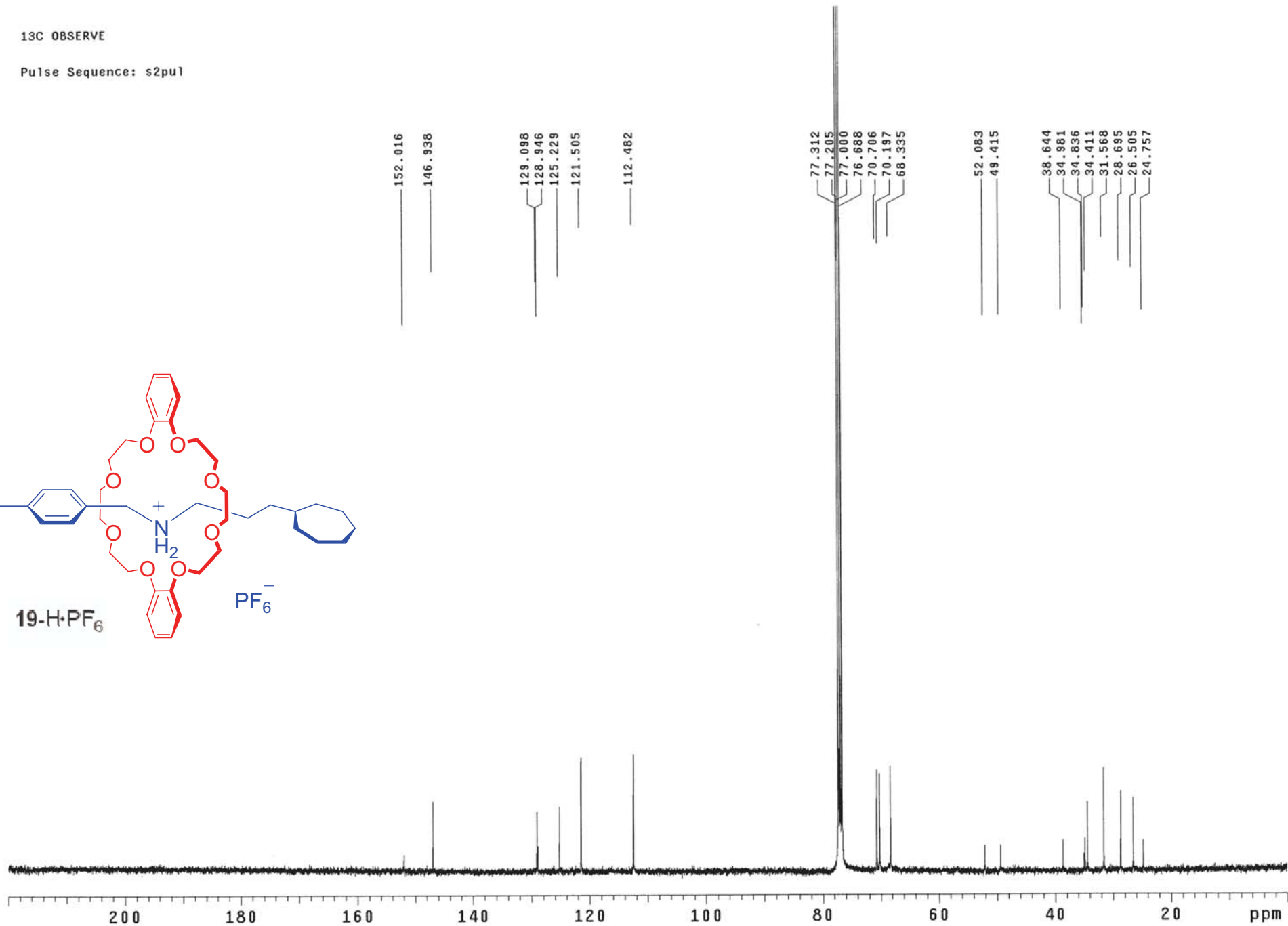
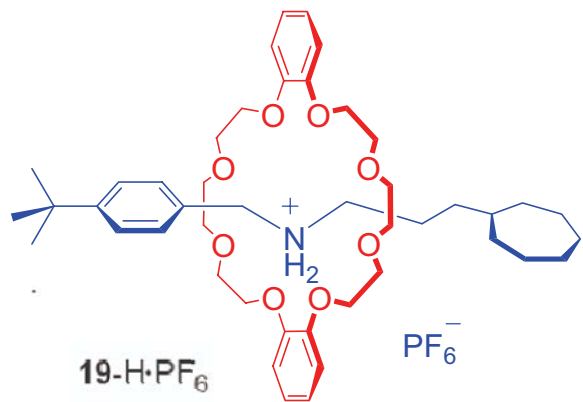


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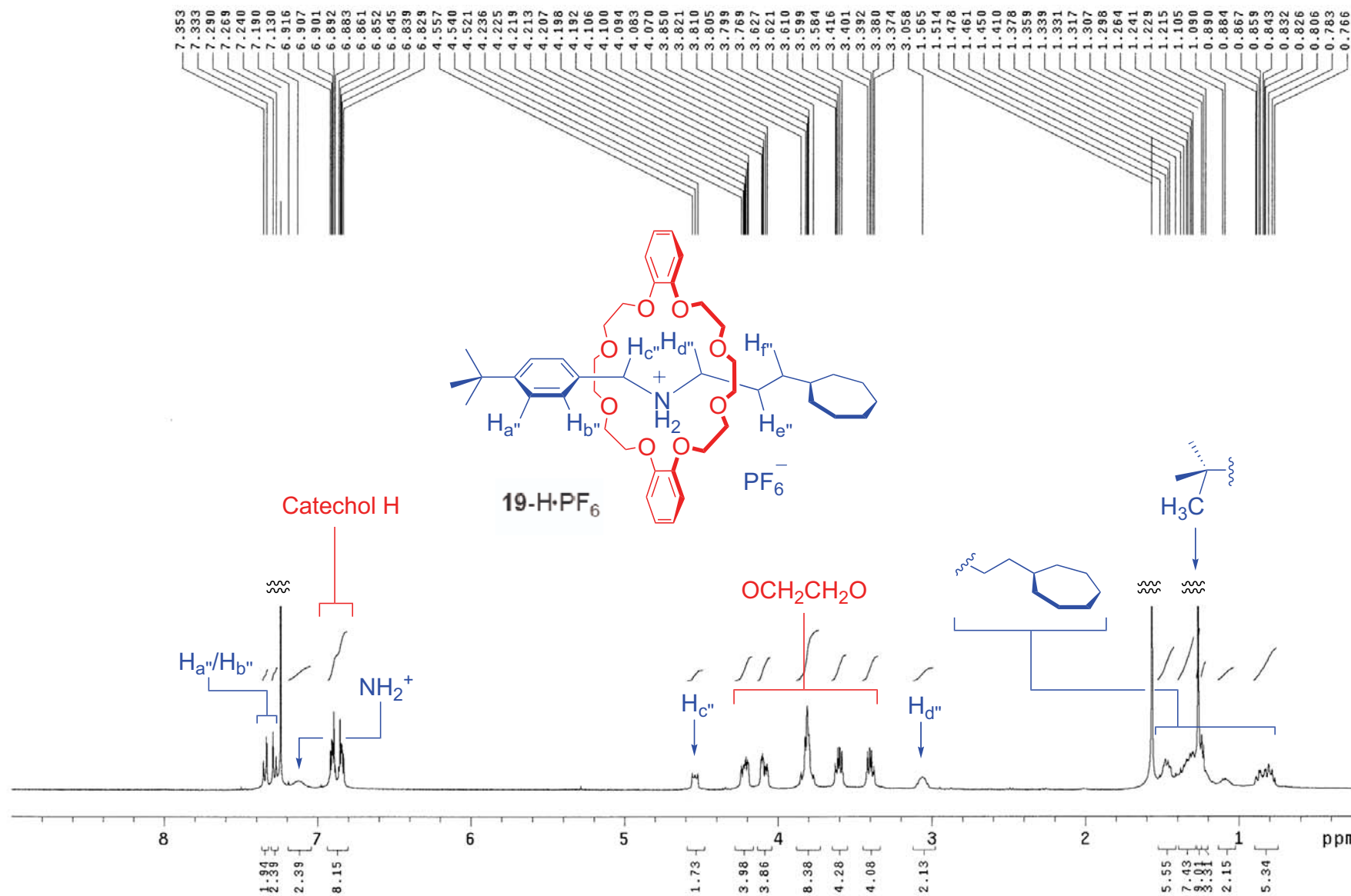


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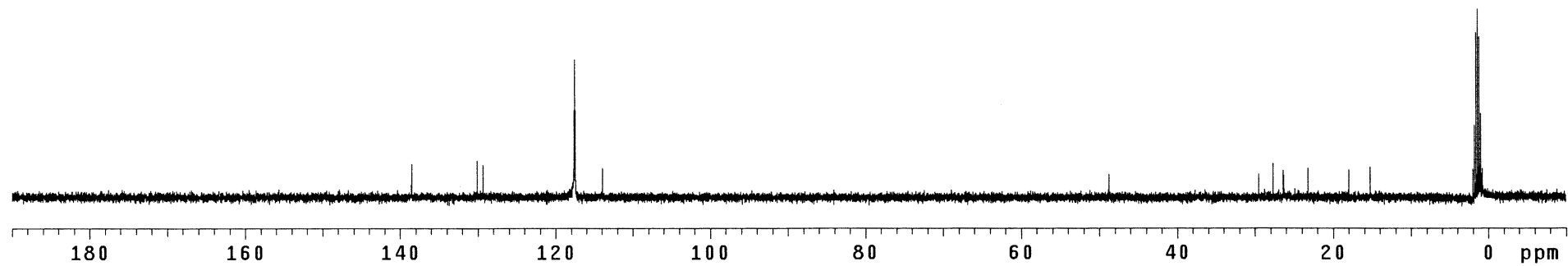
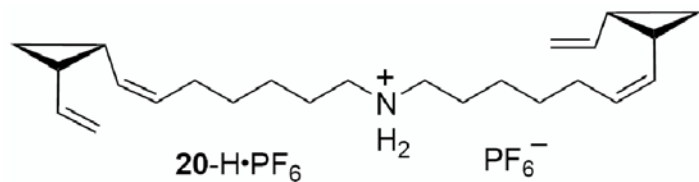
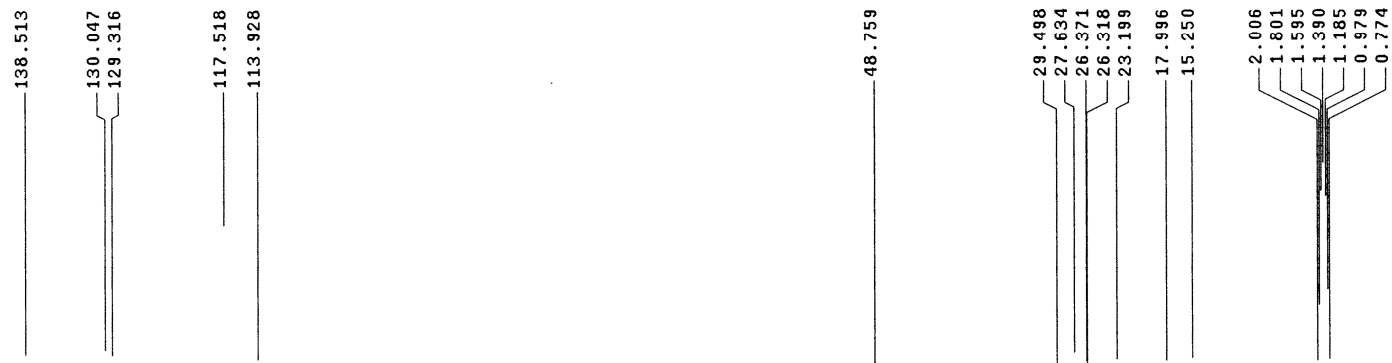
400MHz H<sup>1</sup> CD<sub>3</sub>Cl<sub>3</sub>





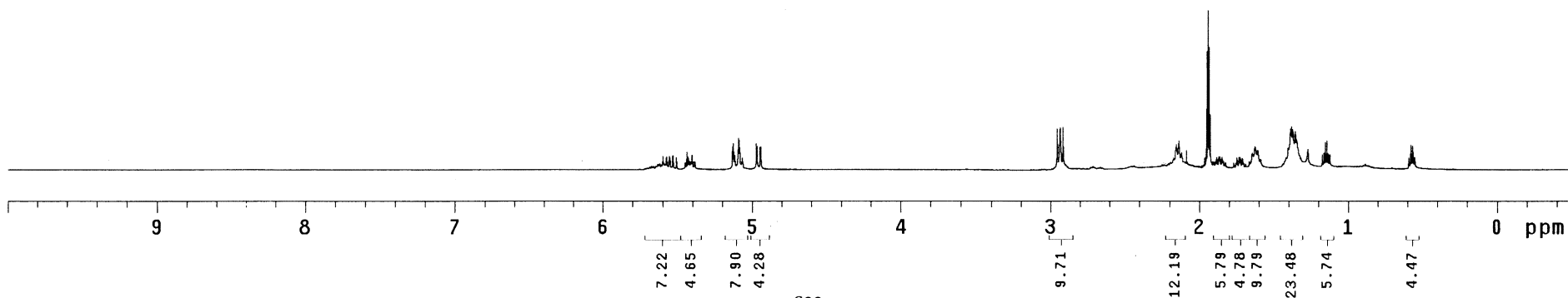
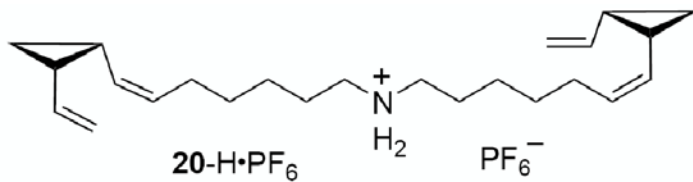
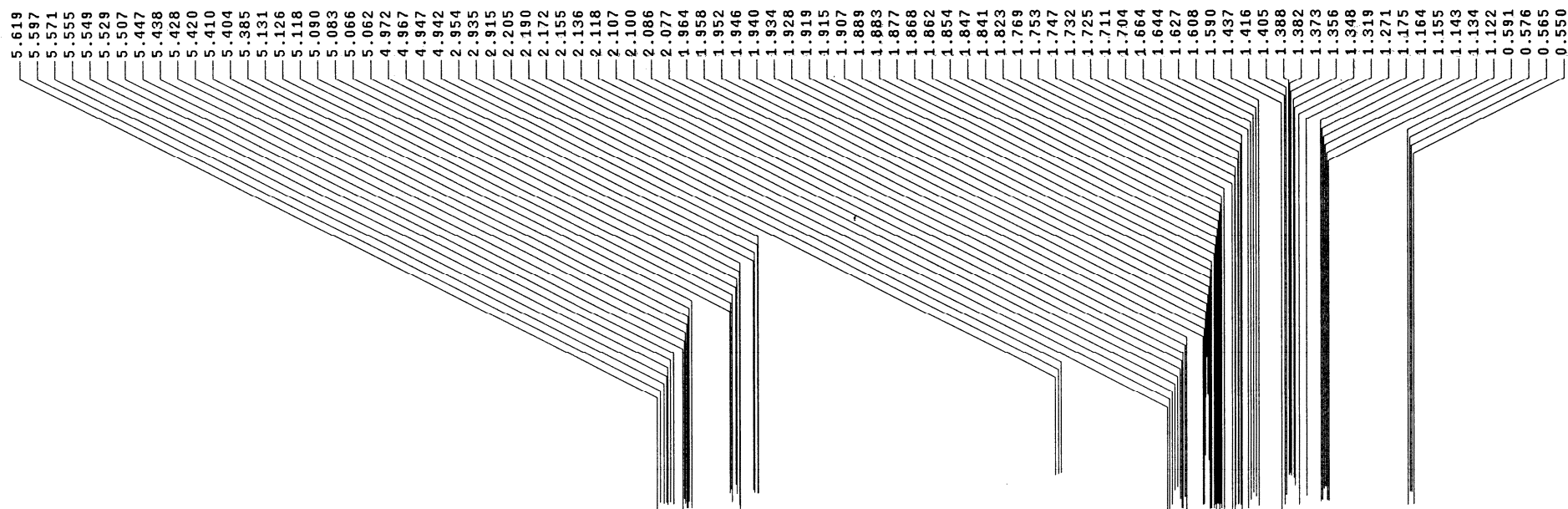
13C OBSERVE

Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



13C OBSERVE

Pulse Sequence: s2pu1

173.466

130.109  
128.452

119.498

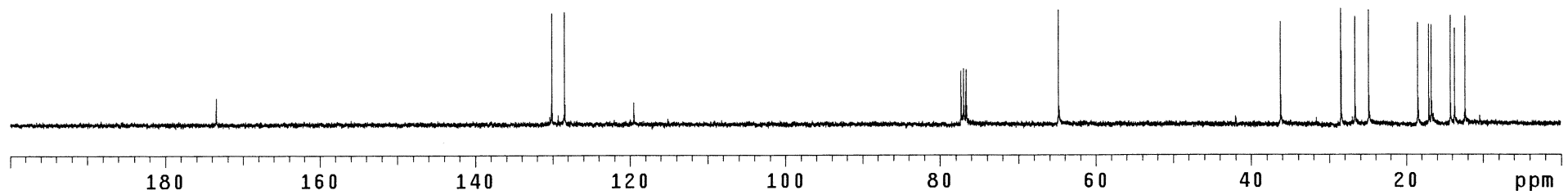
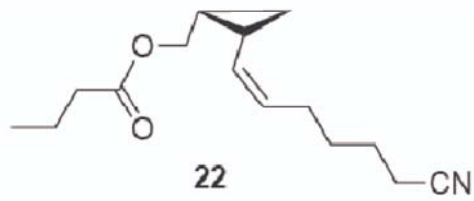
77.319  
77.000  
76.681

64.853

36.220

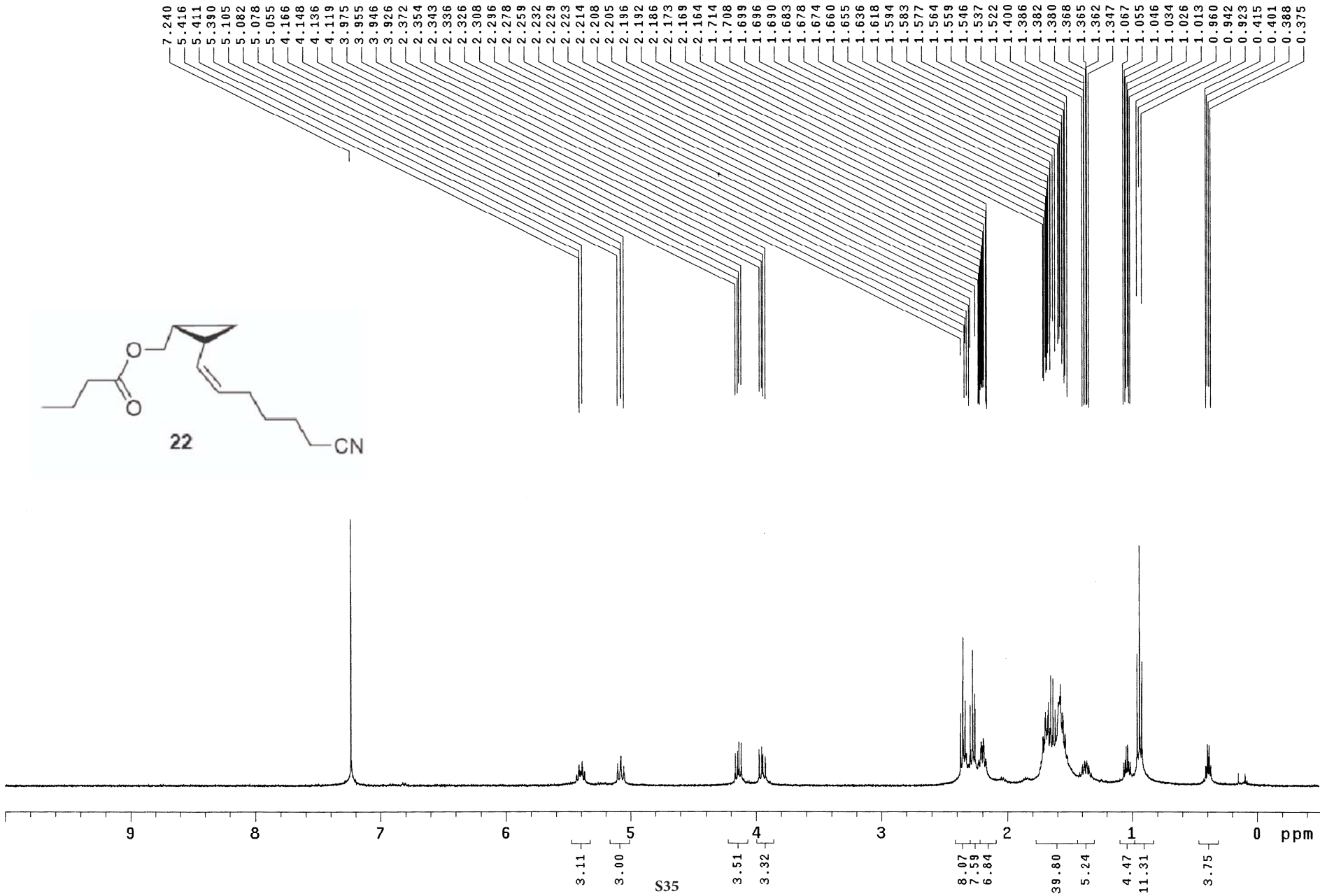
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26.673  
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18.524  
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16.761  
14.260  
13.720  
12.367



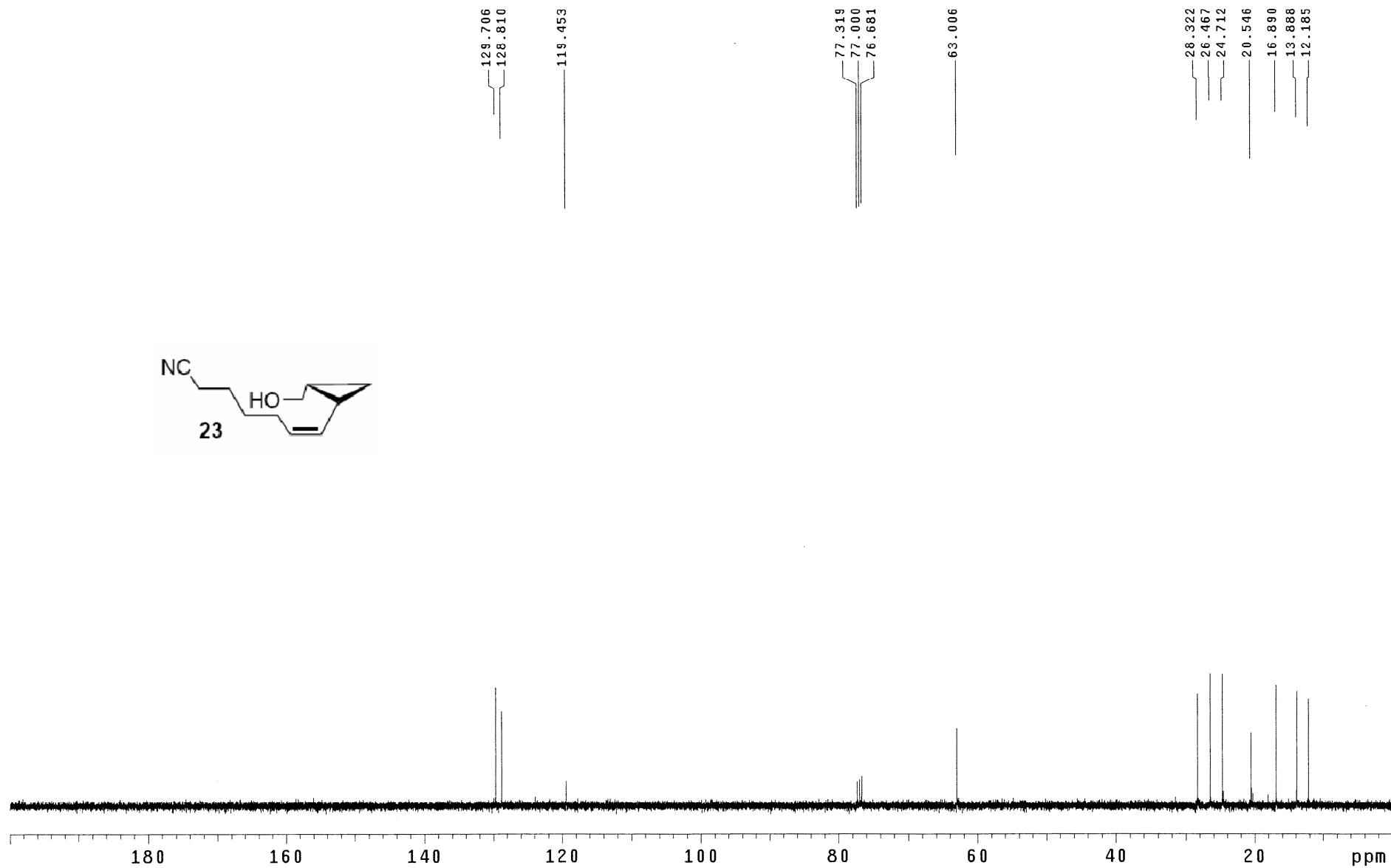
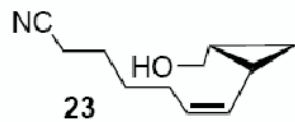
STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



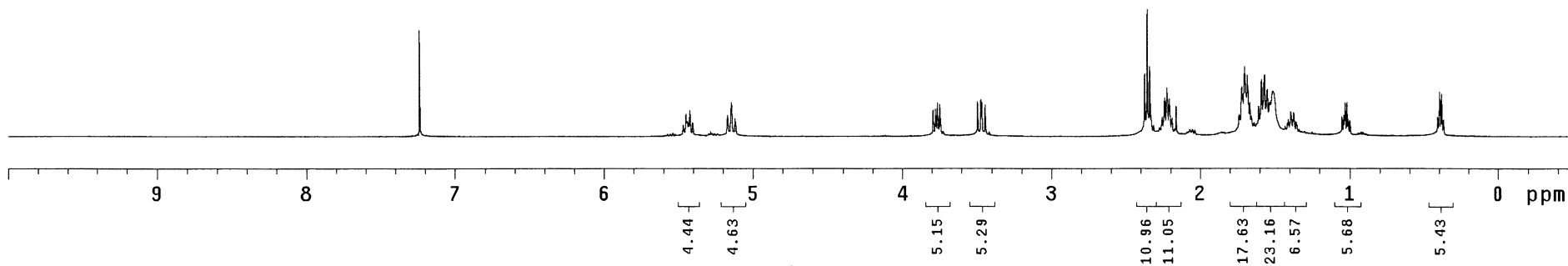
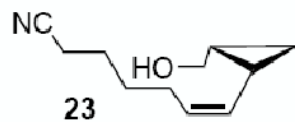
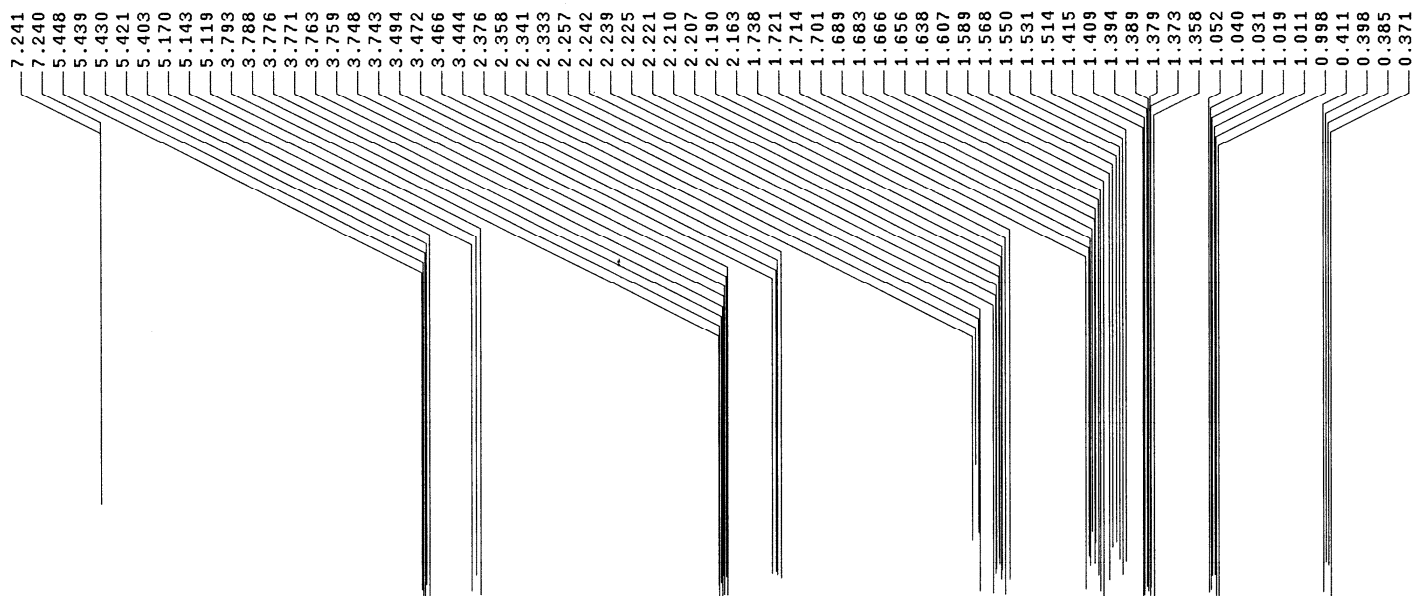
13C OBSERVE

Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



<sup>13</sup>C OBSERVE

Pulse Sequence: s2pu1

200.428

131.880

126.400

119.468

77.319

77.000

76.688

30.222

28.315

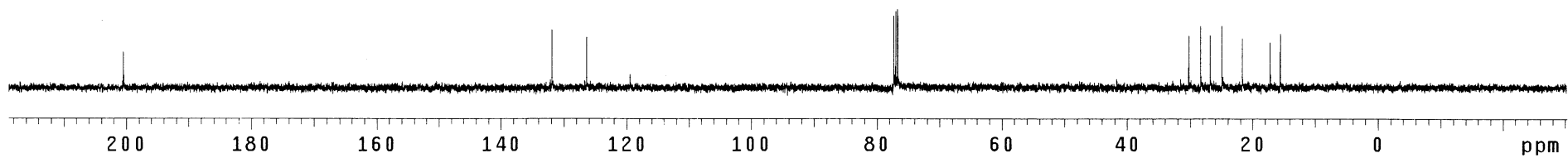
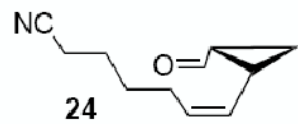
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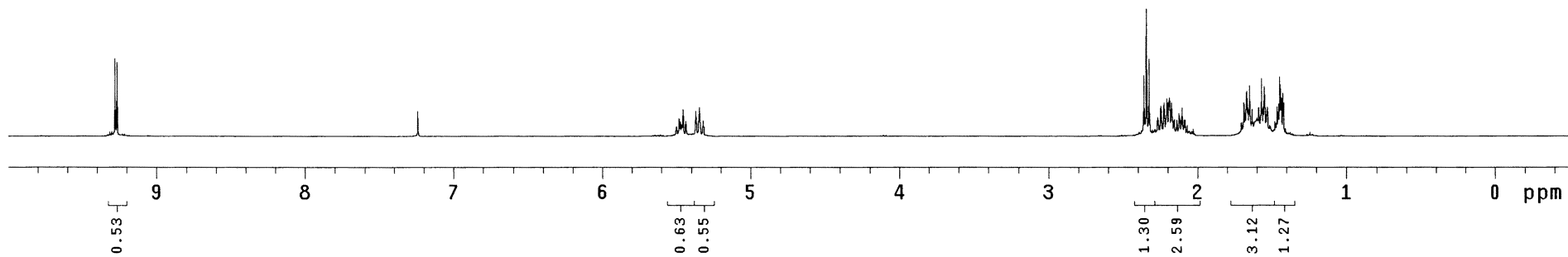
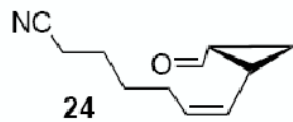
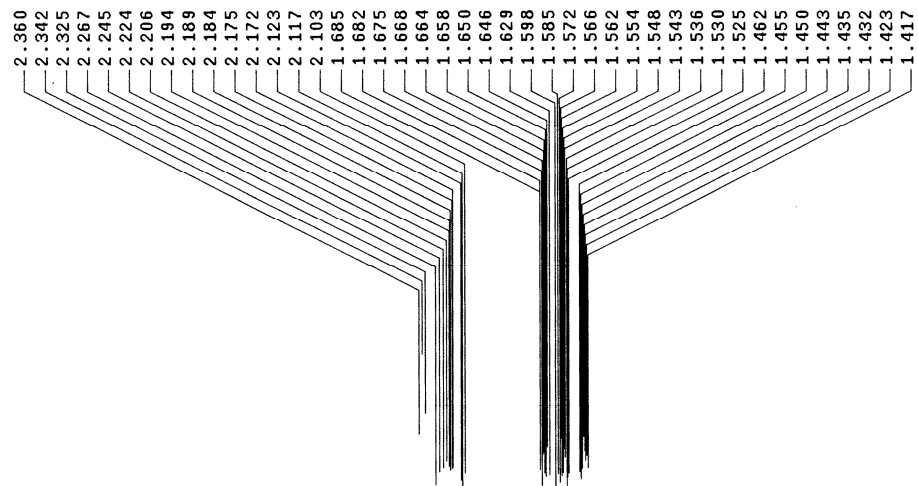
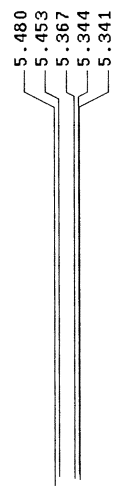
17.164

15.529



STANDARD 1H OBSERVE

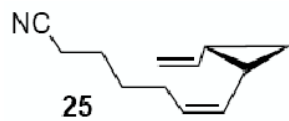
Pulse Sequence: s2pu1





13C OBSERVE

Pulse Sequence: s2pu1



137.688

129.562  
129.205

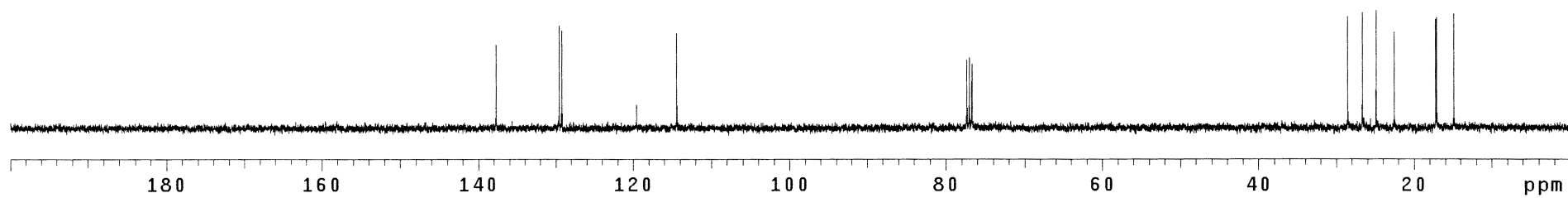
119.589

114.405

77.319  
77.000  
76.688

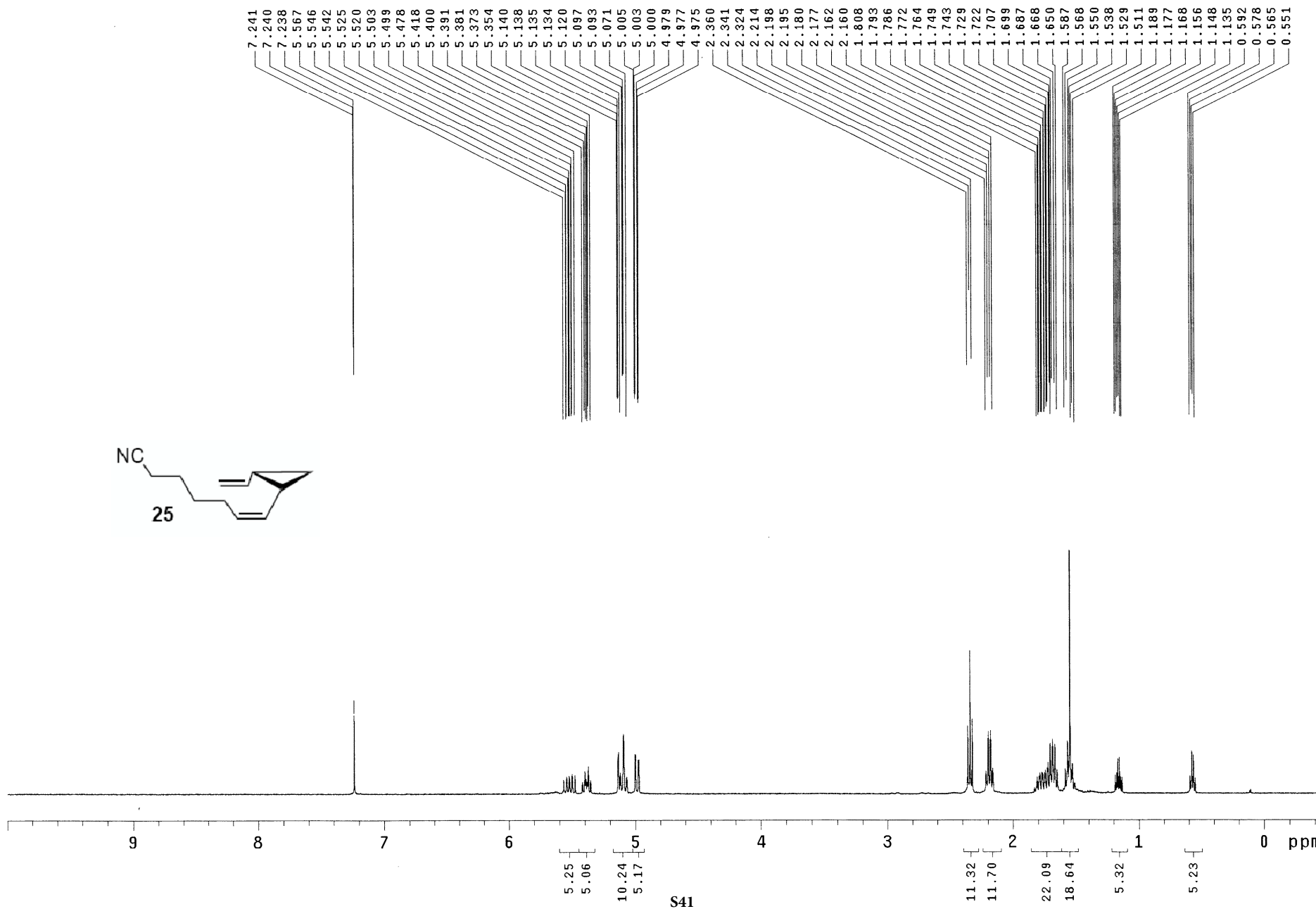
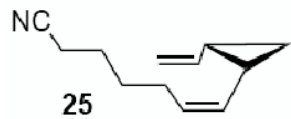
28.489  
26.619  
24.864  
22.522

17.217  
17.095  
14.868



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



13C OBSERVE

Pulse Sequence: s2pu1

202.457

137.840

129.927

129.030

114.307

77.319

77.000

76.688

43.806

29.143

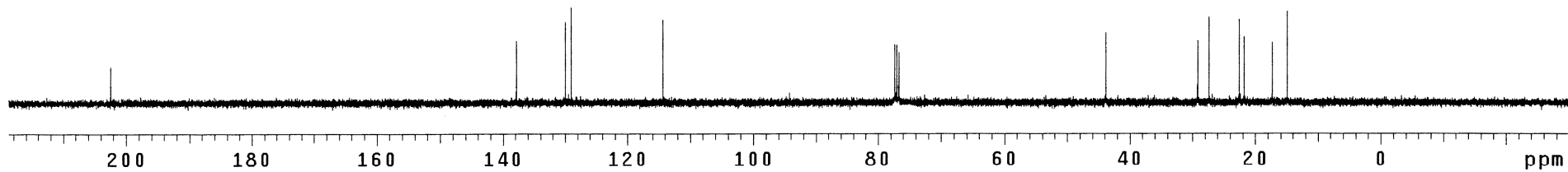
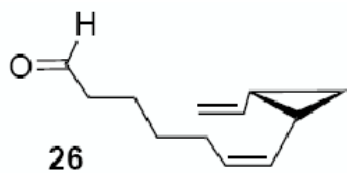
27.296

22.515

21.740

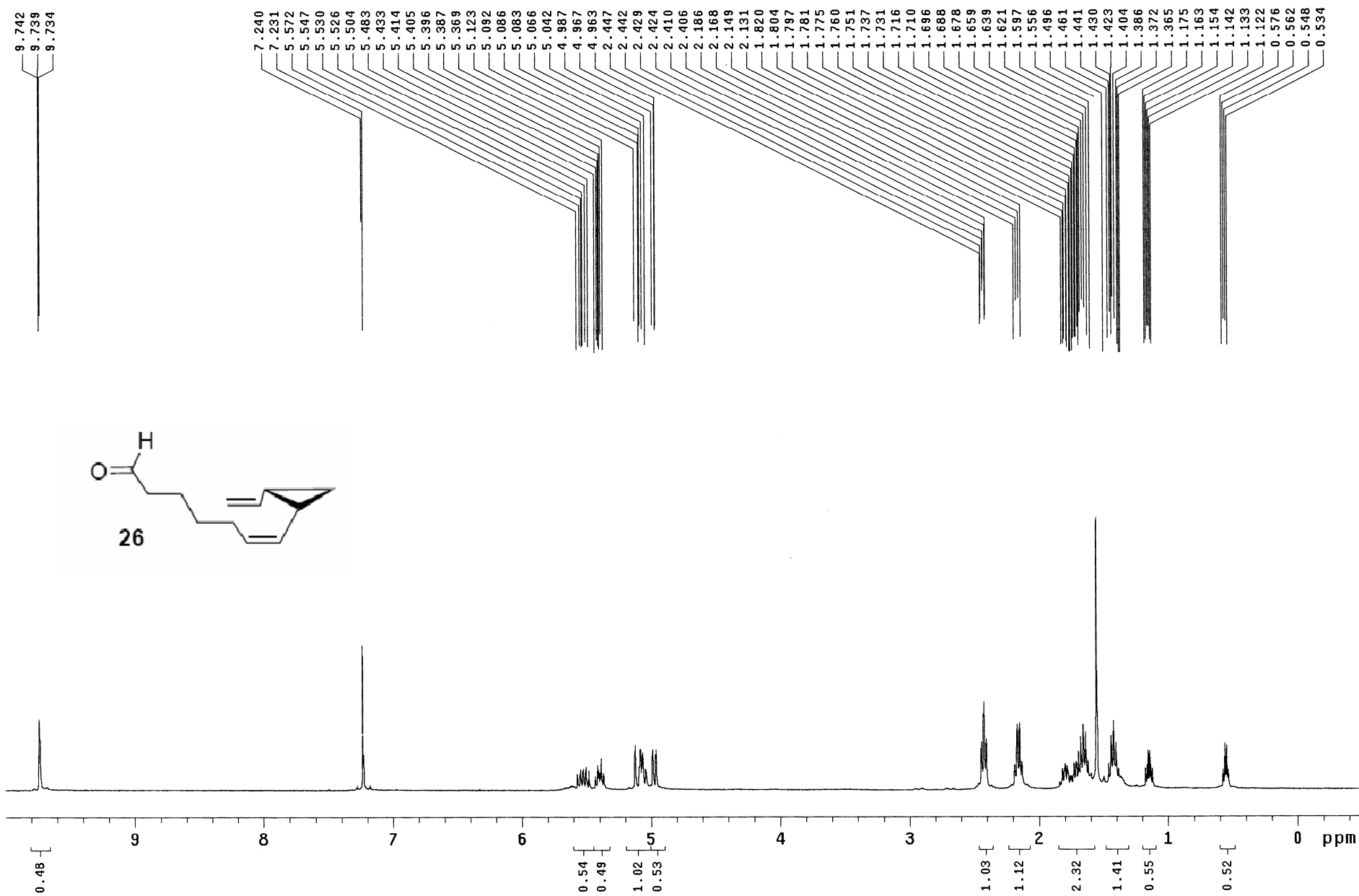
17.300

14.883



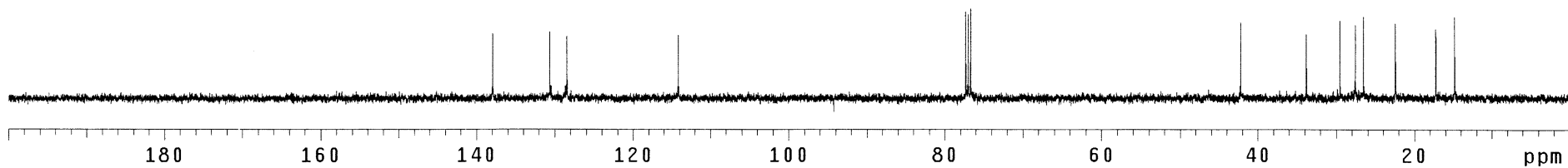
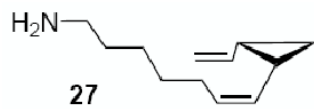
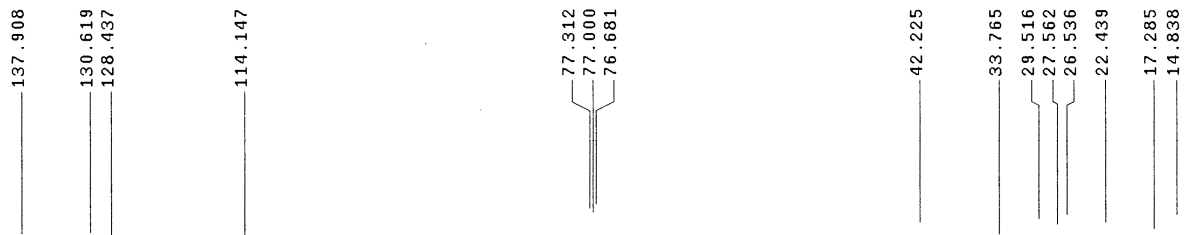
STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



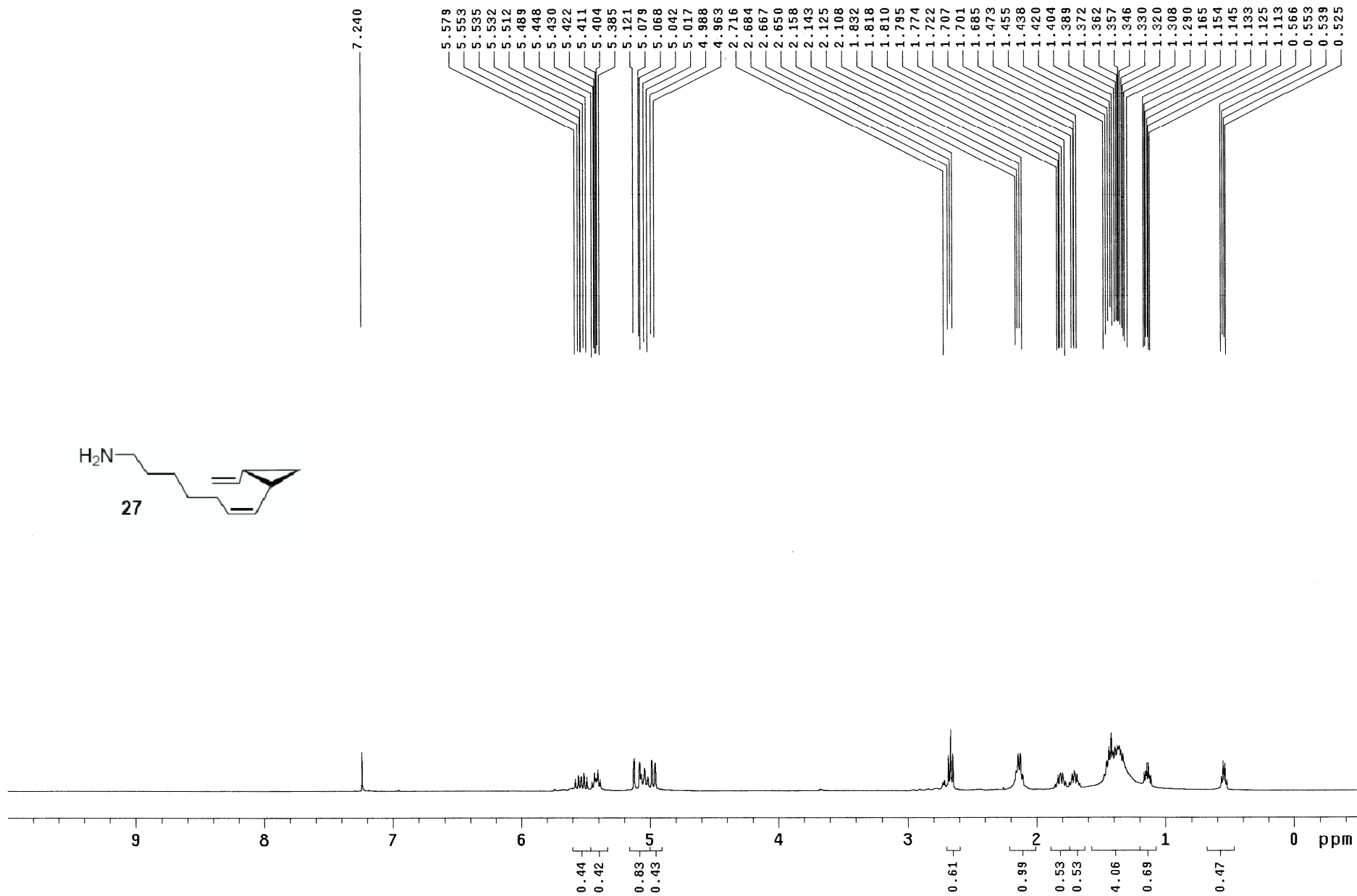
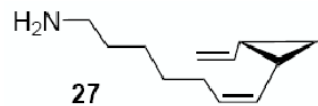
13C OBSERVE

Pulse Sequence: s2pu1



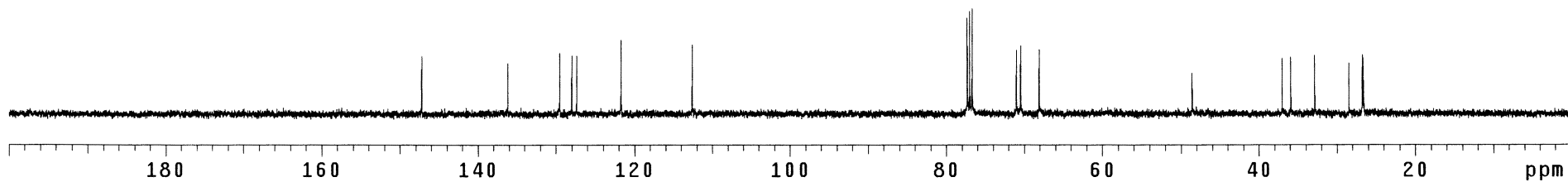
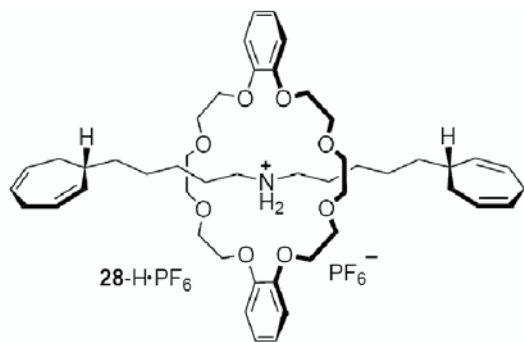
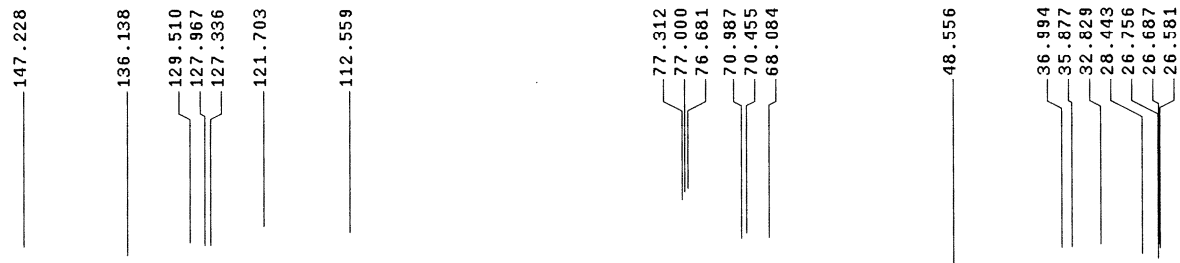
STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



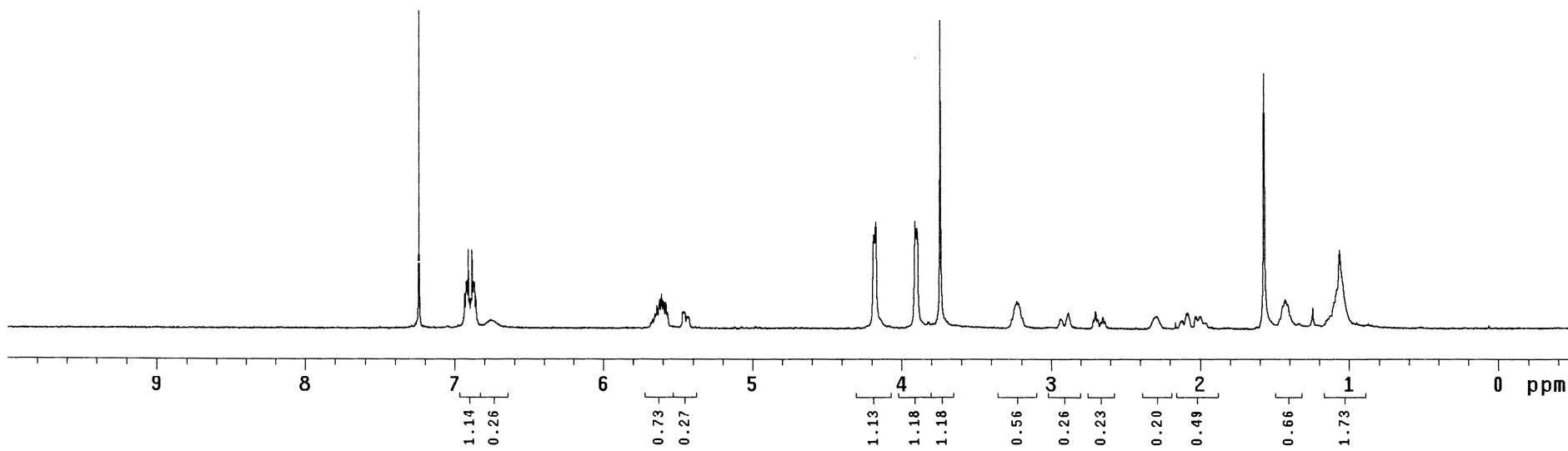
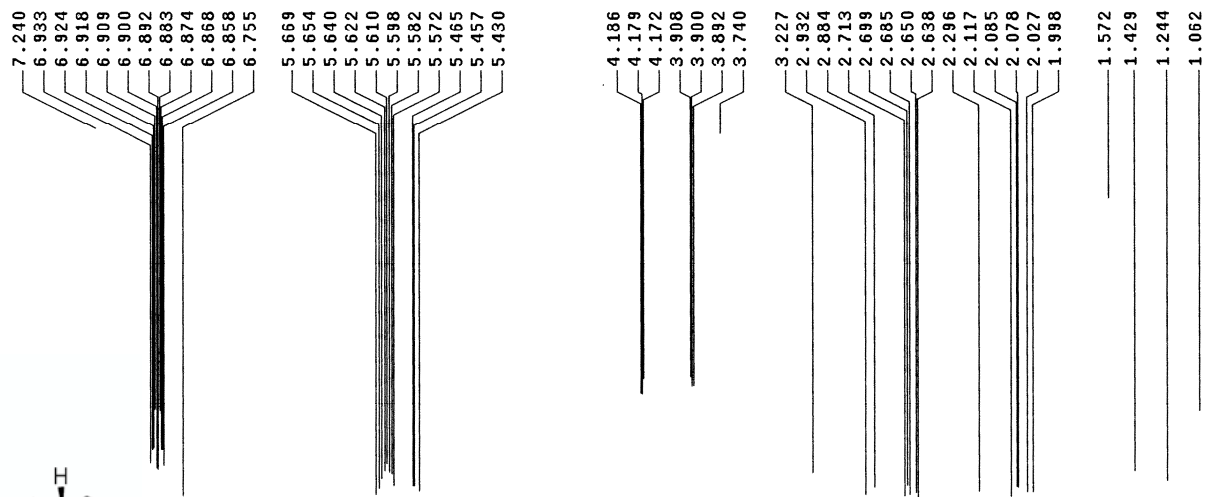
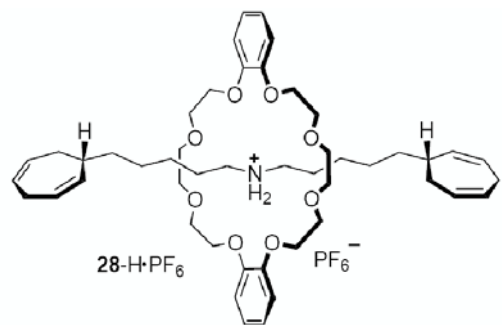
13C OBSERVE

Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

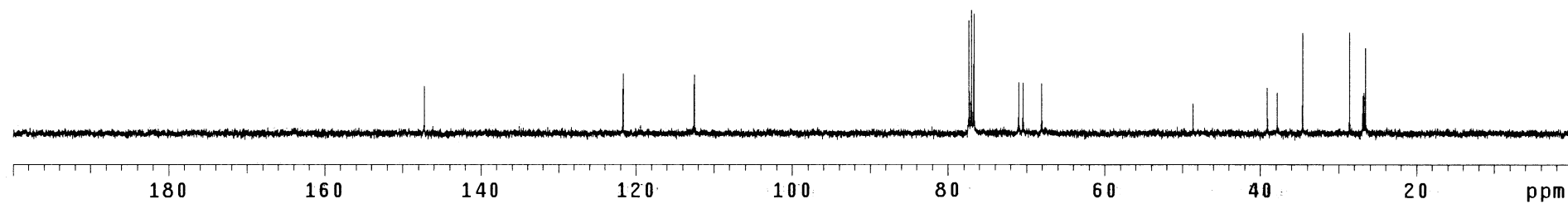
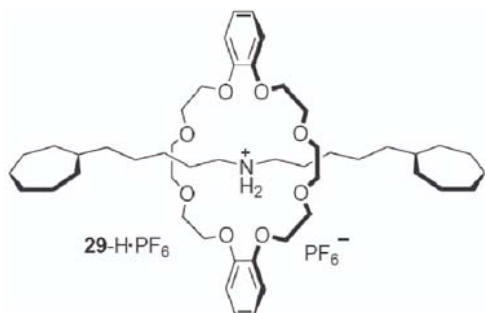
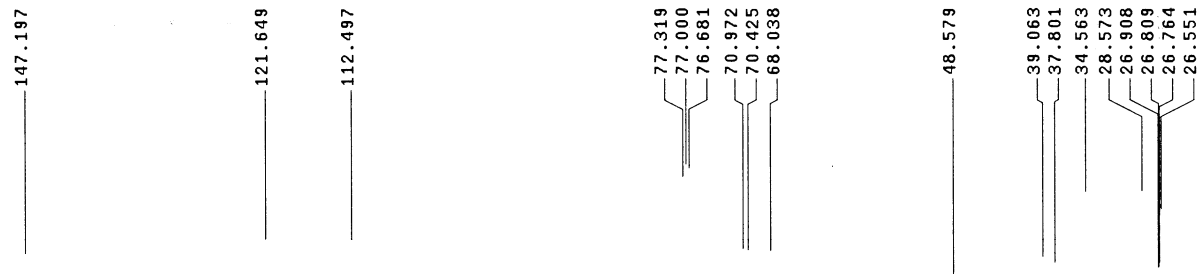
Pulse Sequence: s2pu1





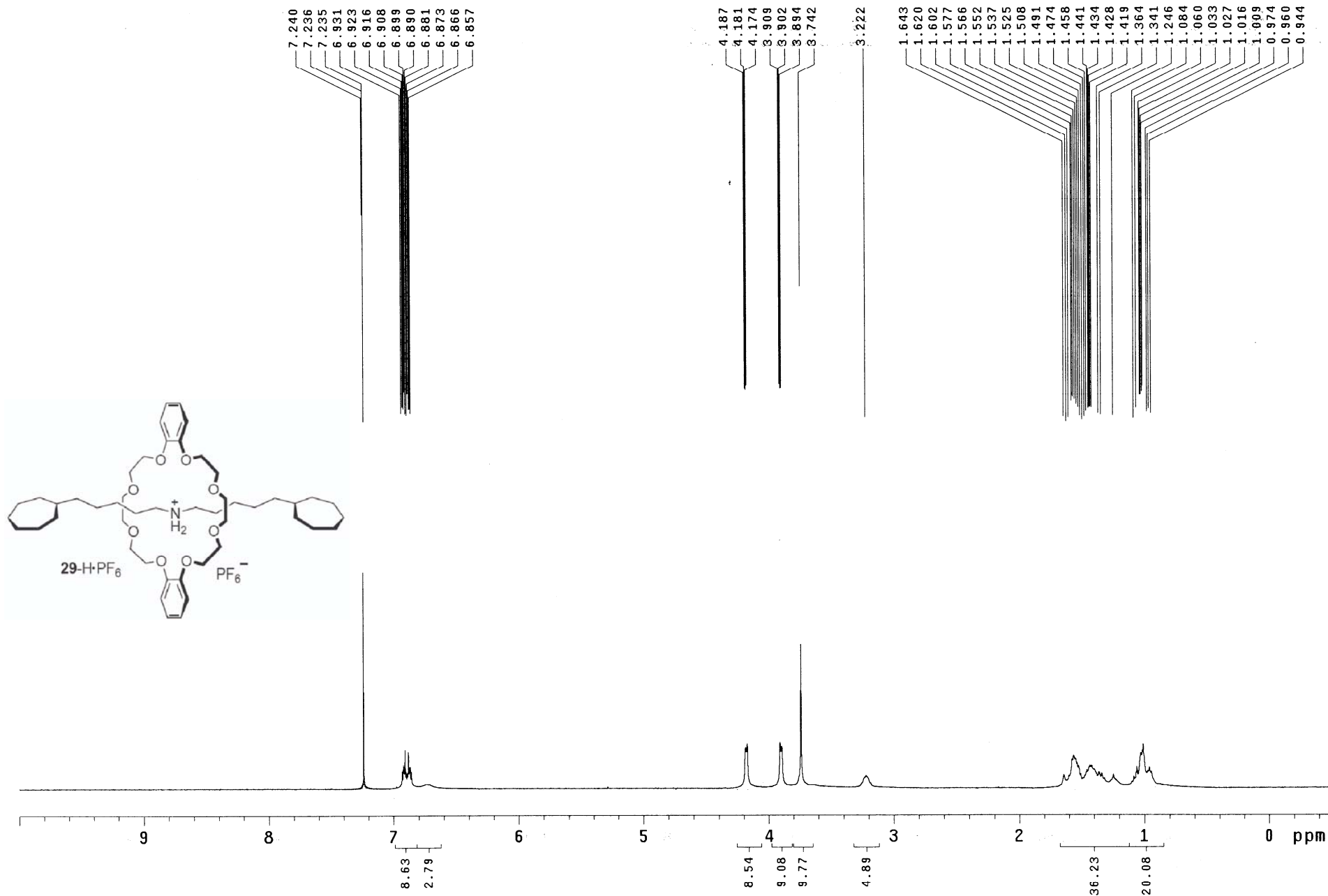
13C OBSERVE

Pulse Sequence: s2pu1



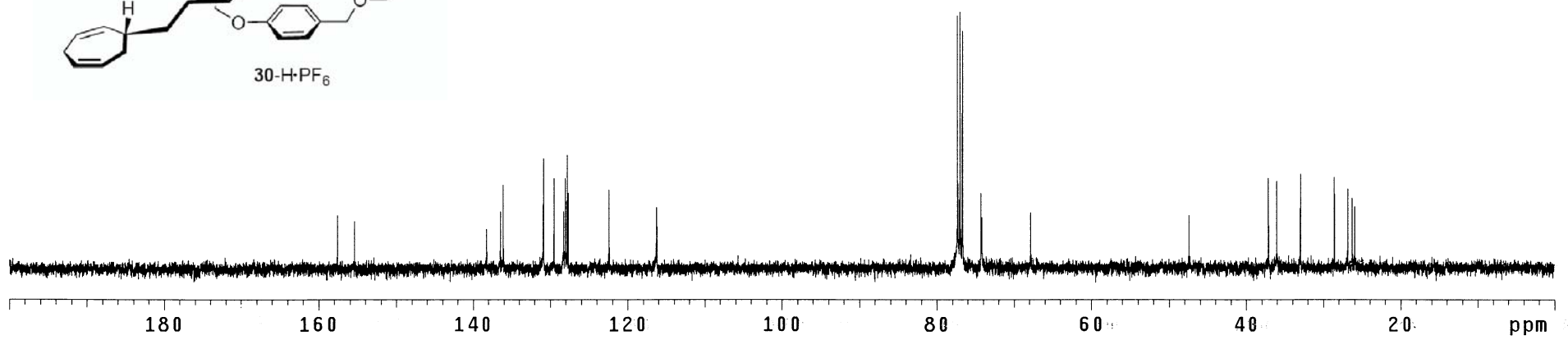
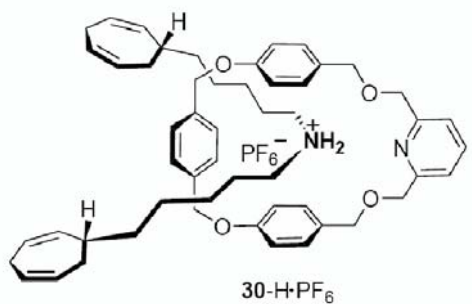
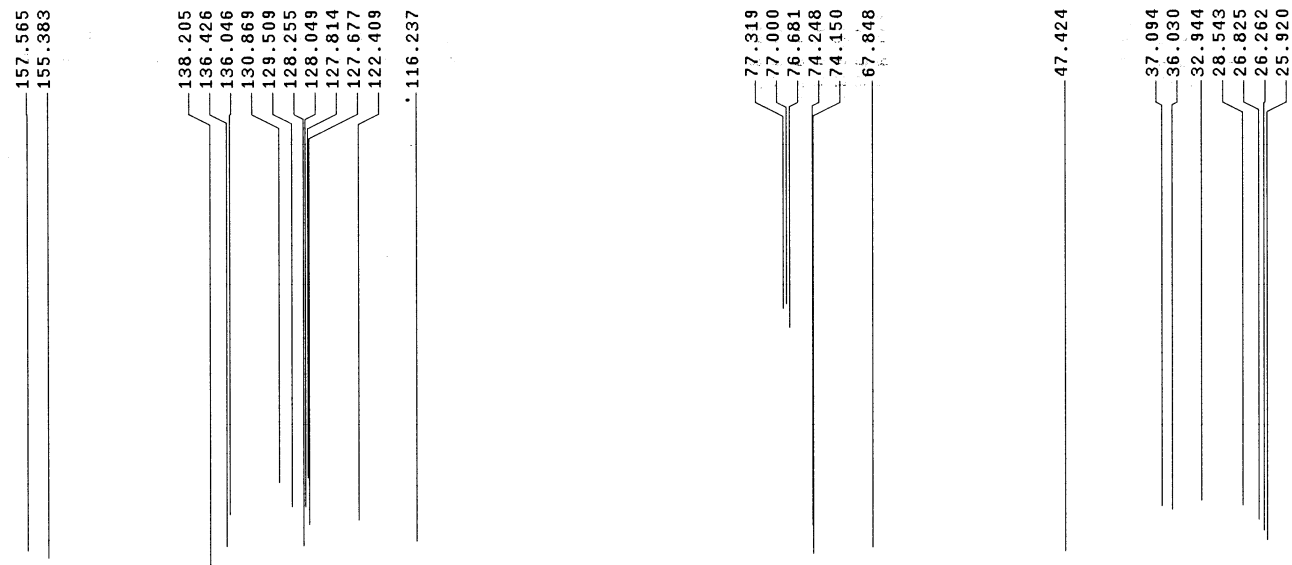
STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



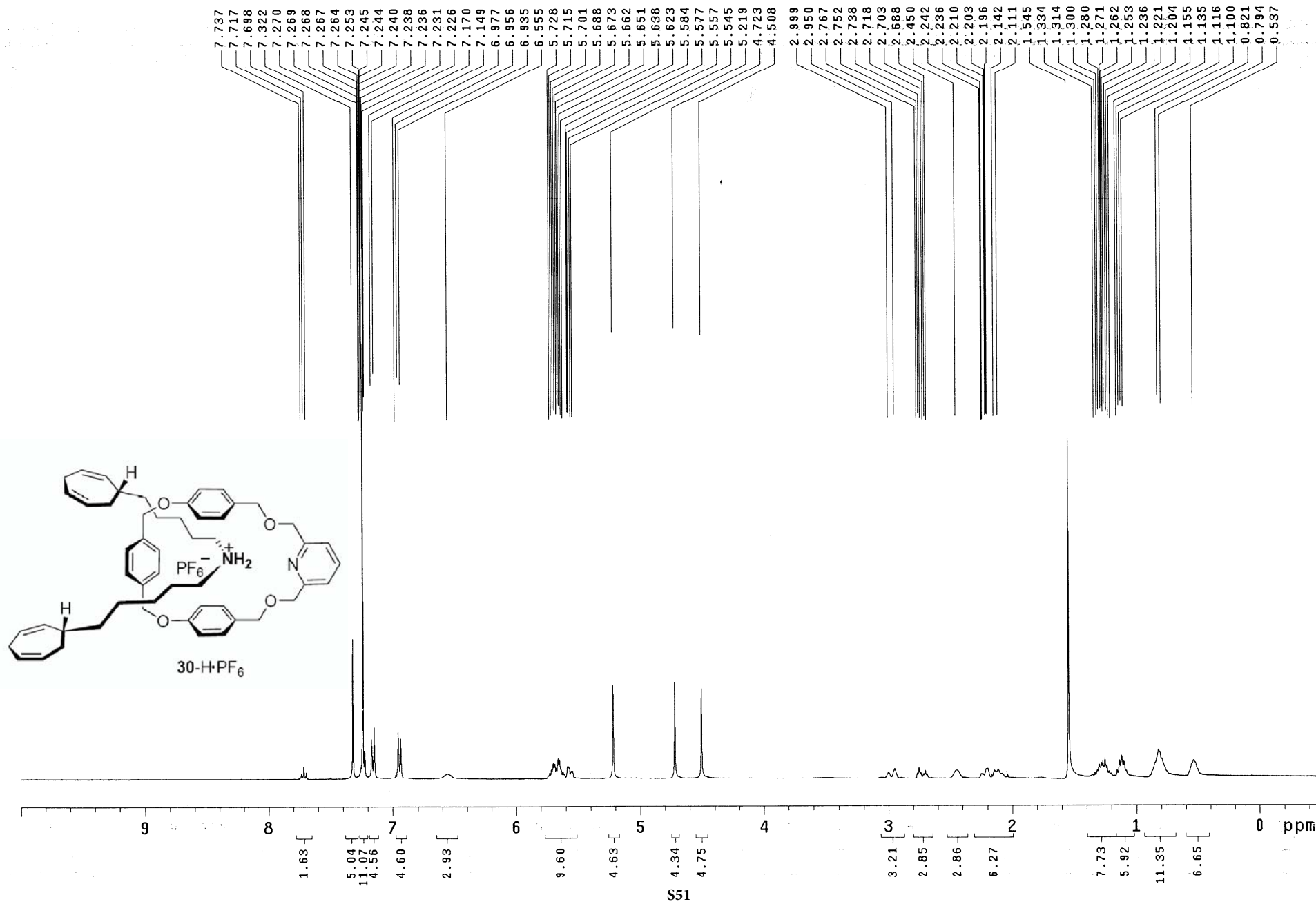
<sup>13</sup>C OBSERVE

Pulse Sequence: s2pu1



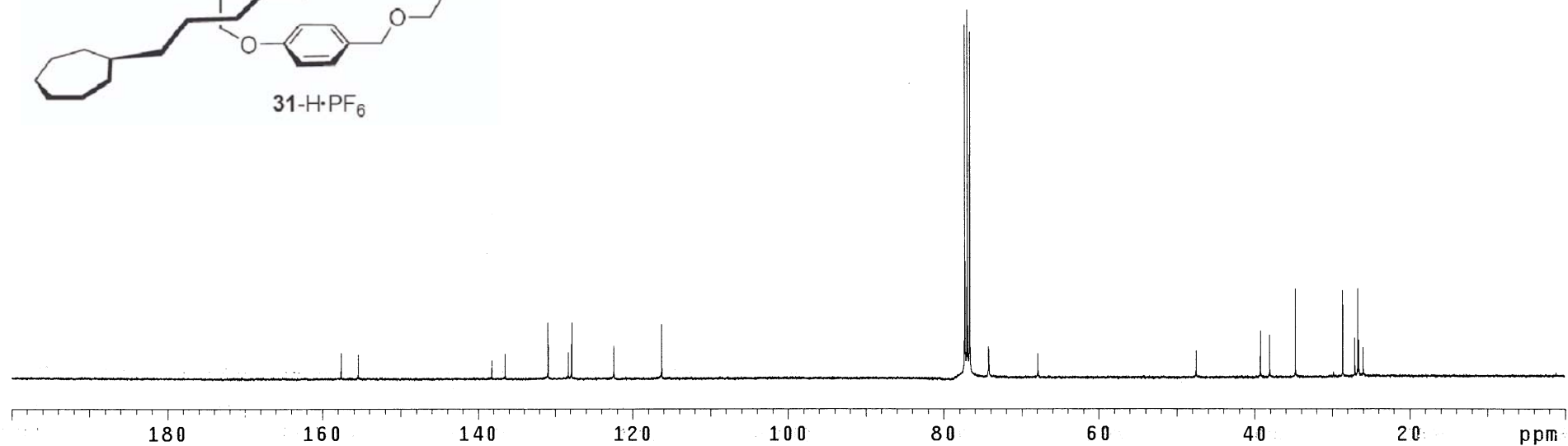
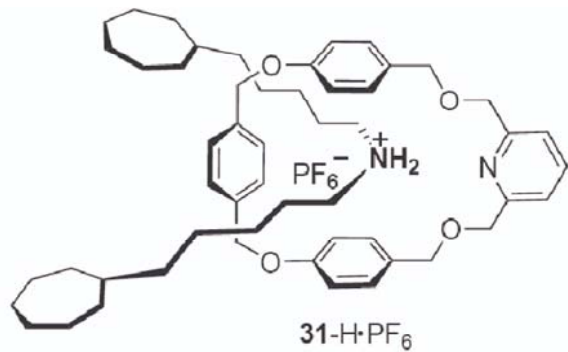
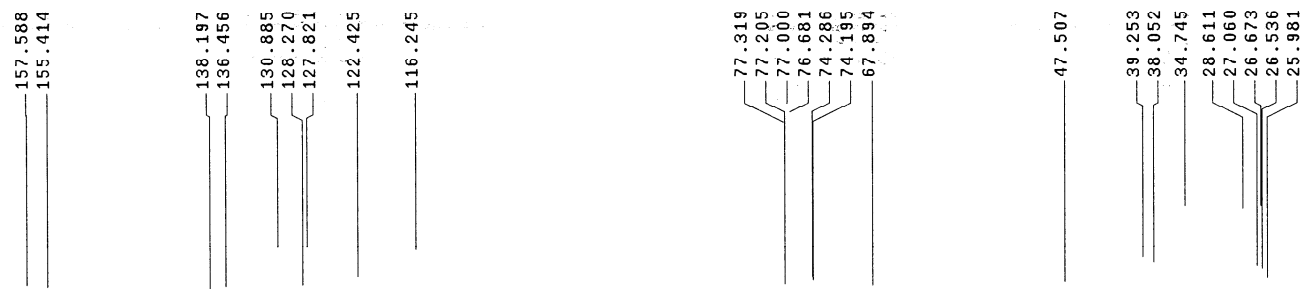
STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



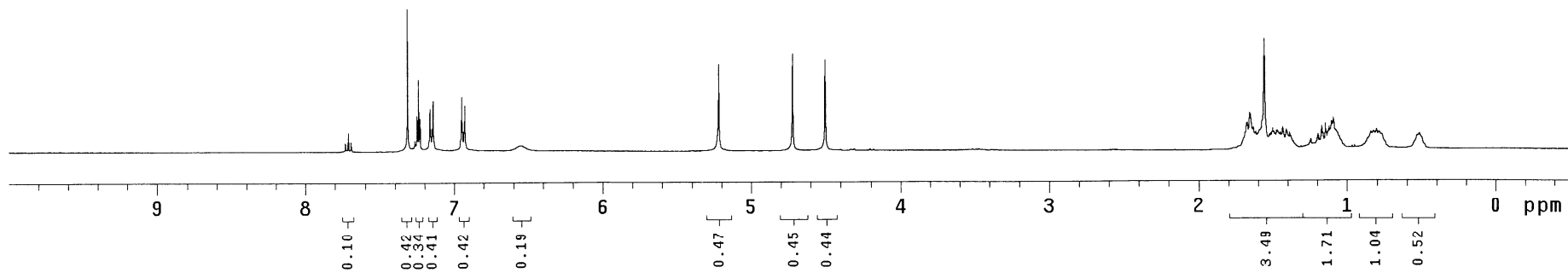
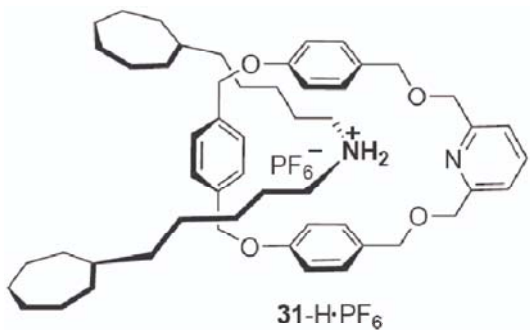
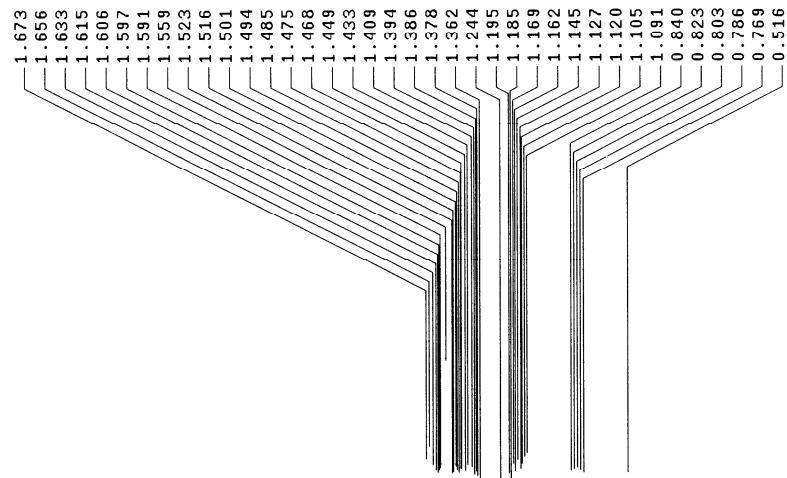
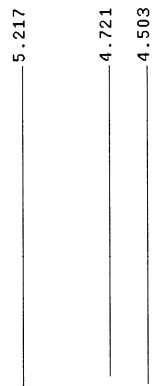
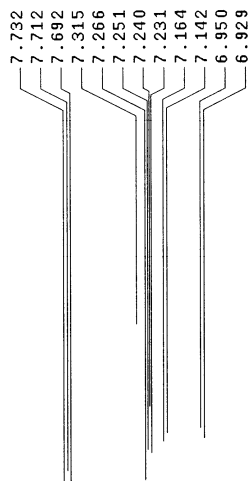
13C OBSERVE

Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1



13C OBSERVE

Pulse Sequence: s2pu1

159.170

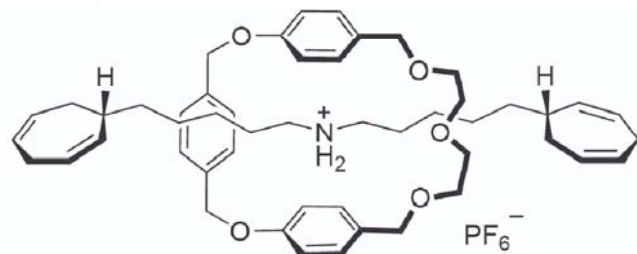
138.472  
137.917  
132.315  
131.175  
130.947  
129.639  
129.160  
129.062

117.888

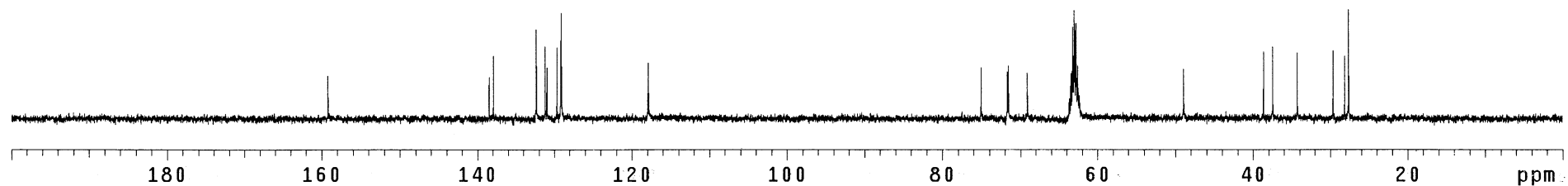
75.040  
71.896  
71.551  
69.104  
63.441  
63.228  
63.000  
62.780  
62.559

48.907

38.631  
37.445  
34.260  
29.608  
28.103  
27.601

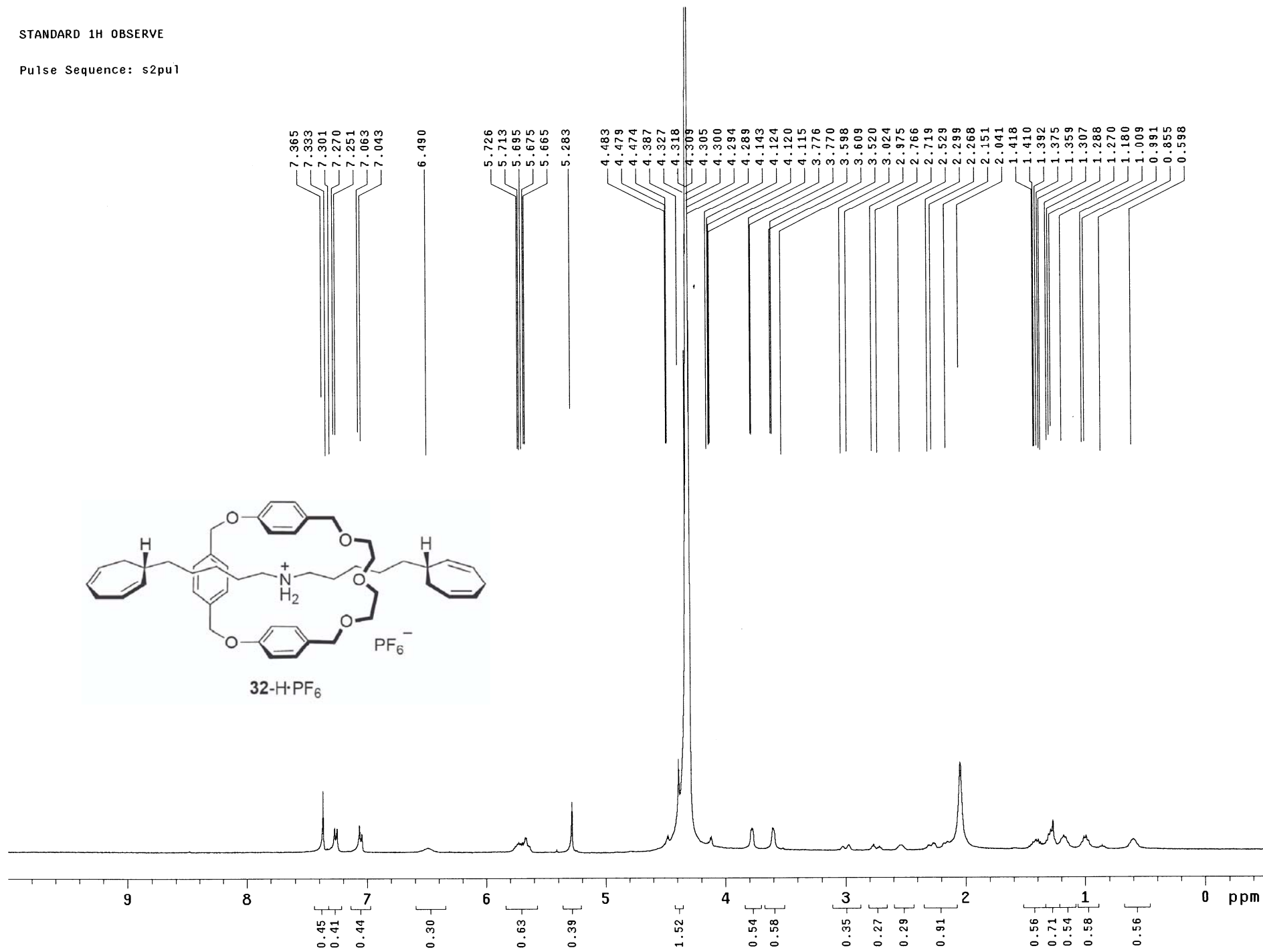


32-H<sup>+</sup>PF<sub>6</sub><sup>-</sup>



STANDARD 1H OBSERVE

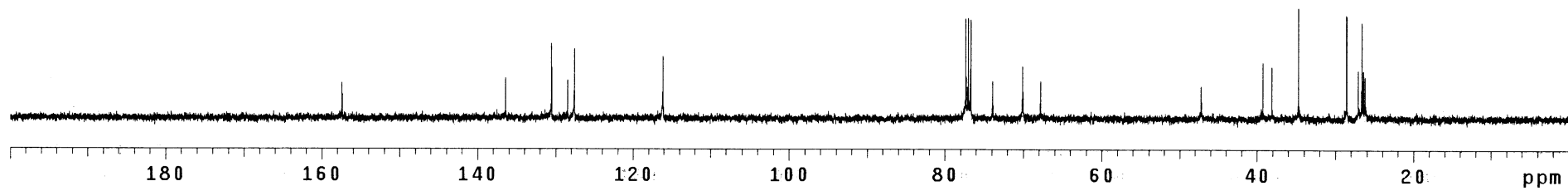
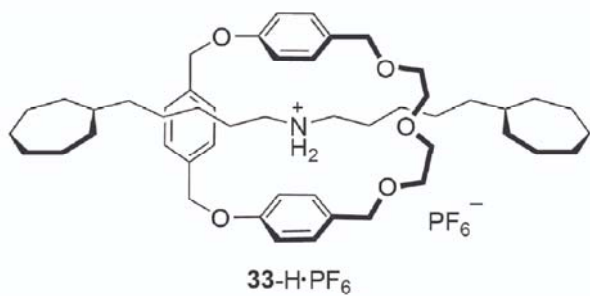
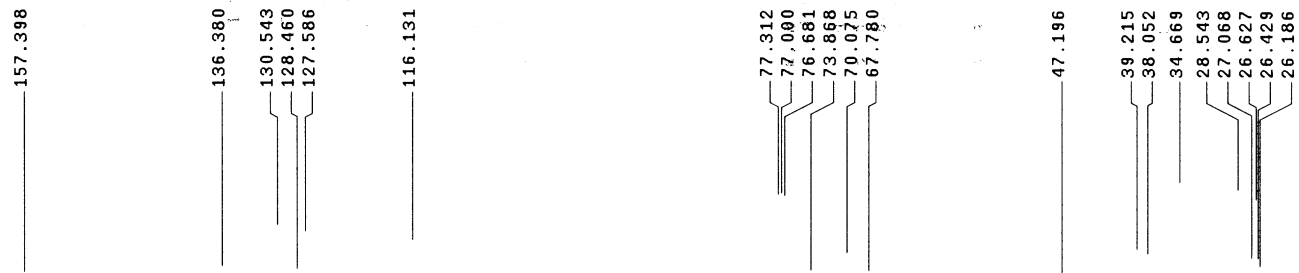
Pulse Sequence: s2pu1





13C OBSERVE

Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

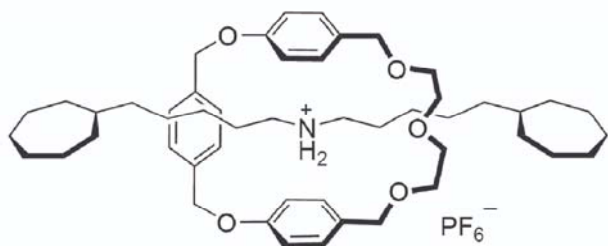
7.309  
7.305  
7.243  
7.241  
7.237  
7.132  
7.111  
6.944  
6.922

5.226

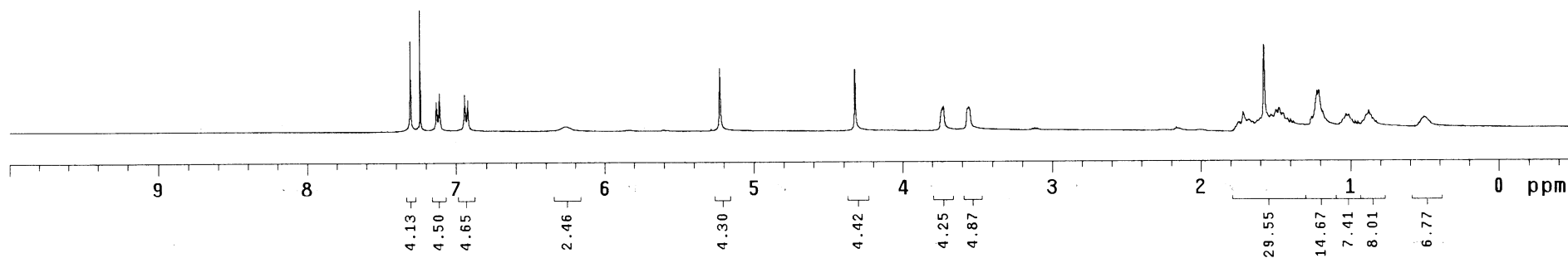
4.323

3.734  
3.724  
3.565  
3.555

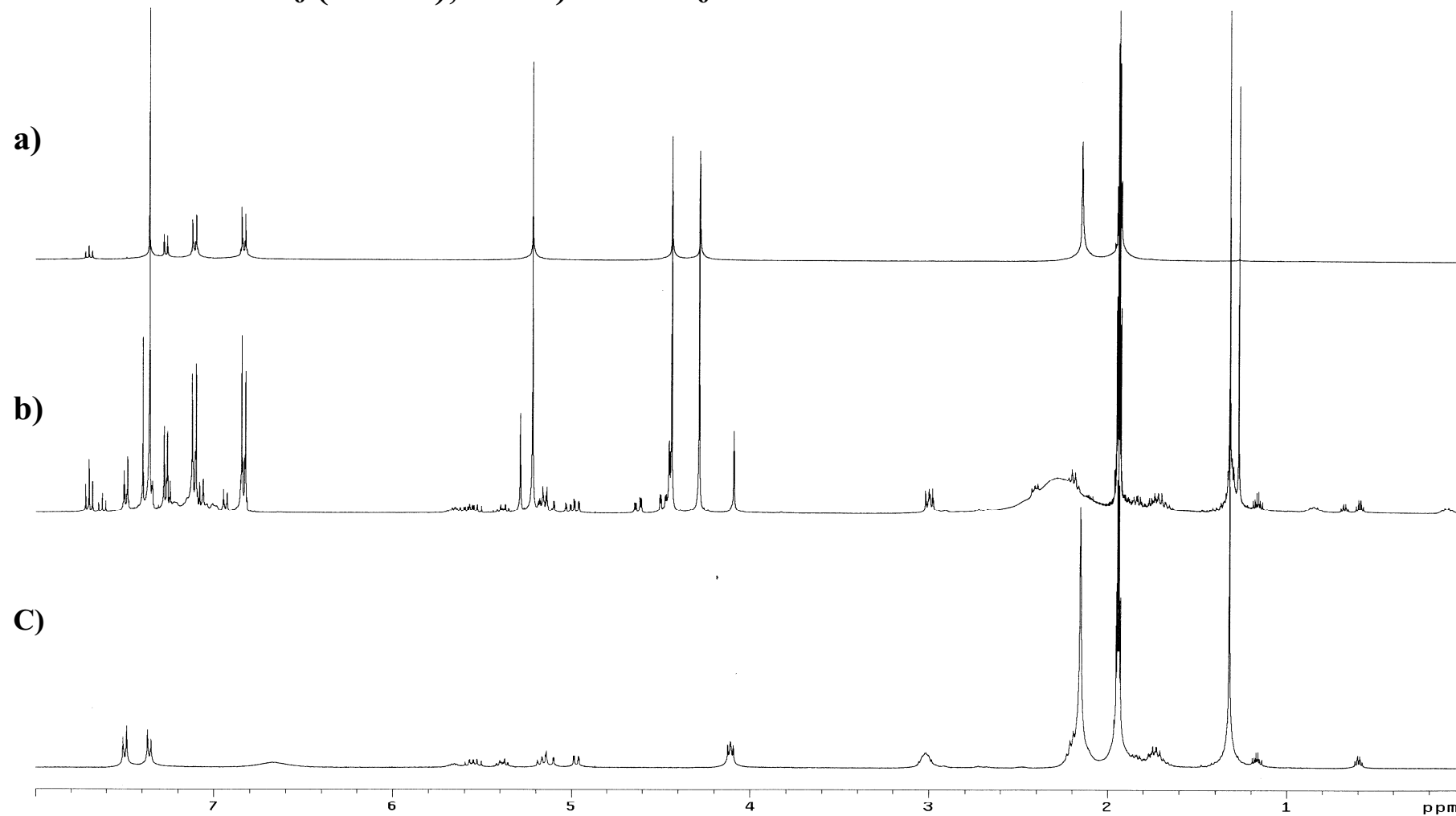
1.715  
1.576  
1.529  
1.496  
1.476  
1.453  
1.258  
1.223  
1.209  
1.184  
1.029  
1.009  
0.876  
0.864  
0.506



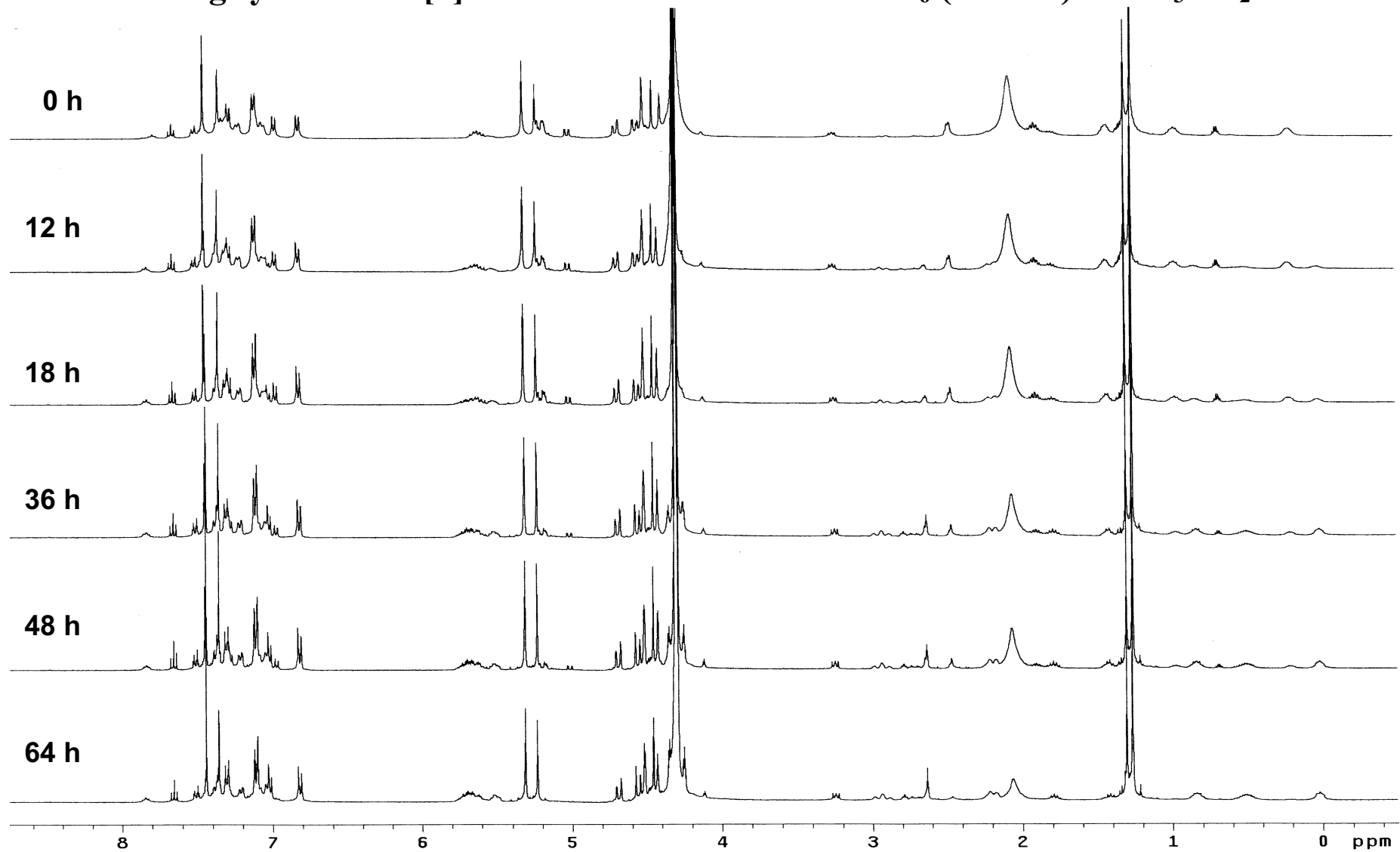
33-H<sup>+</sup>PF<sub>6</sub><sup>-</sup>



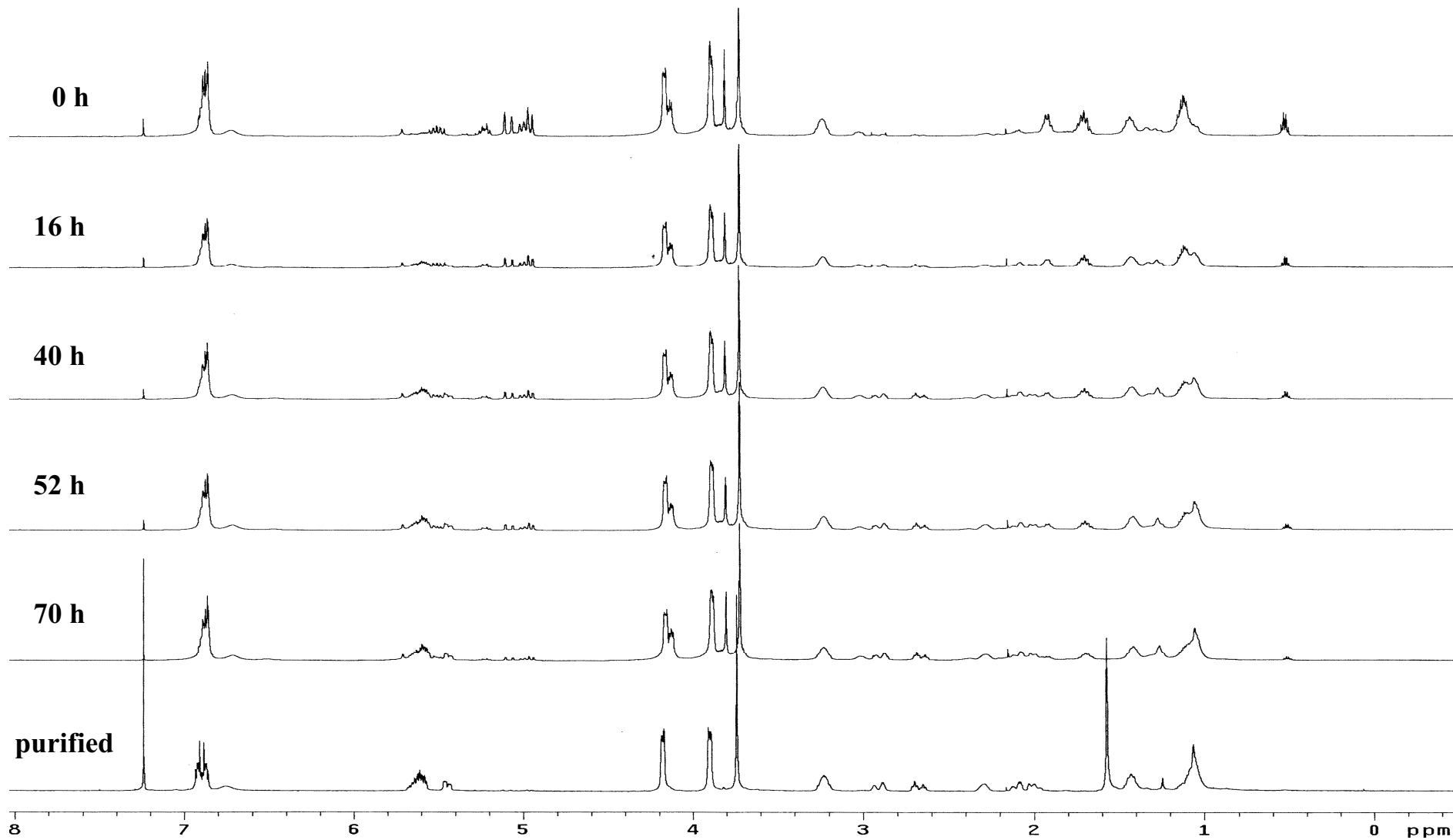
**$^1\text{H}$  NMR spectra (400 MHz,  $\text{CD}_3\text{CN}$ , 298 K) of a) macrocycle 12, b) an equimolar mixture of 12 and  $1\text{-H}\cdot\text{PF}_6$  (10mM), and c)  $1\text{-H}\cdot\text{PF}_6$ .**



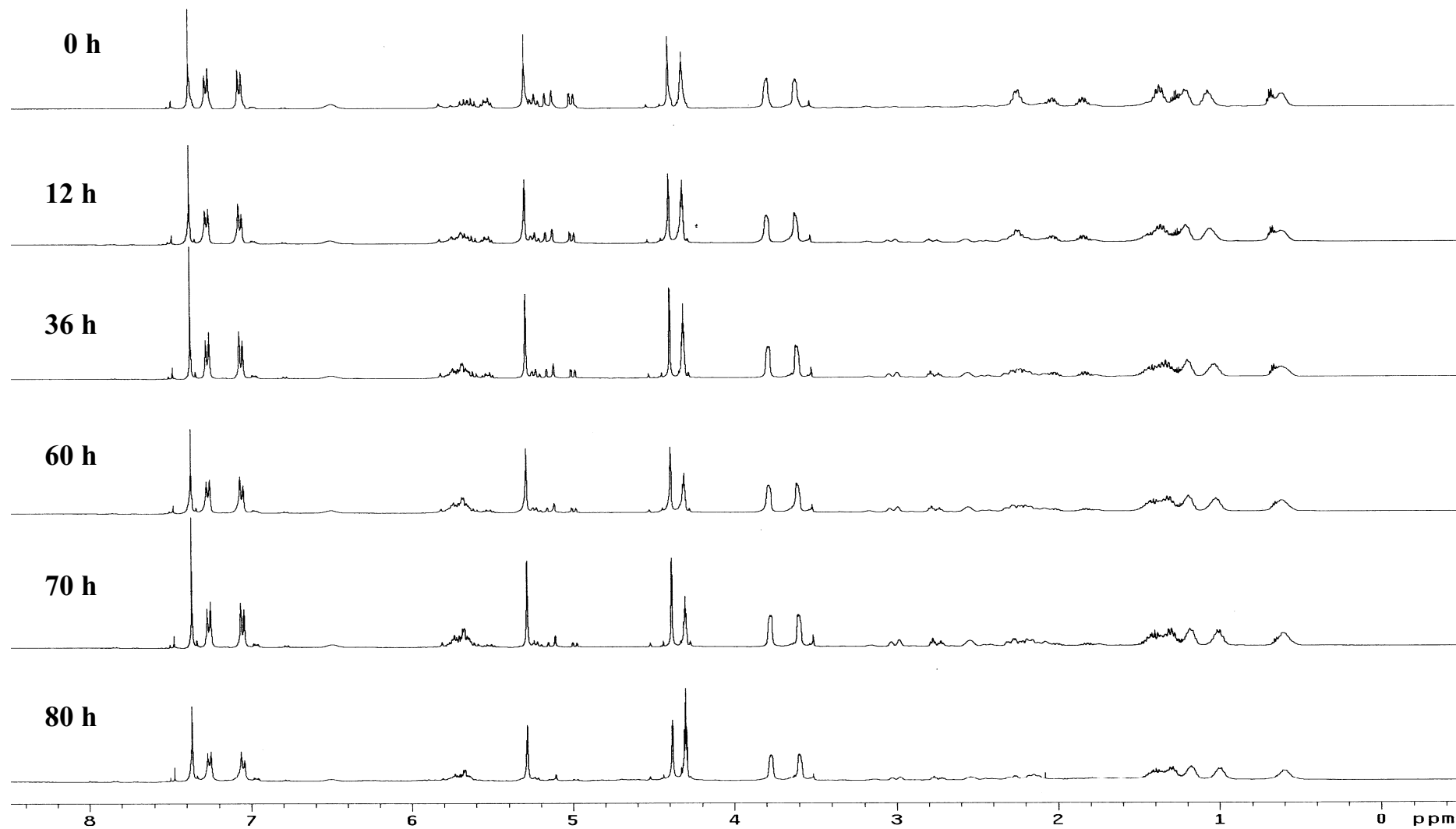
**The swelling synthesis of [2]rotaxane from 12 and 1-H·PF<sub>6</sub> (10 mM) in CD<sub>3</sub>NO<sub>2</sub> at 318 K**



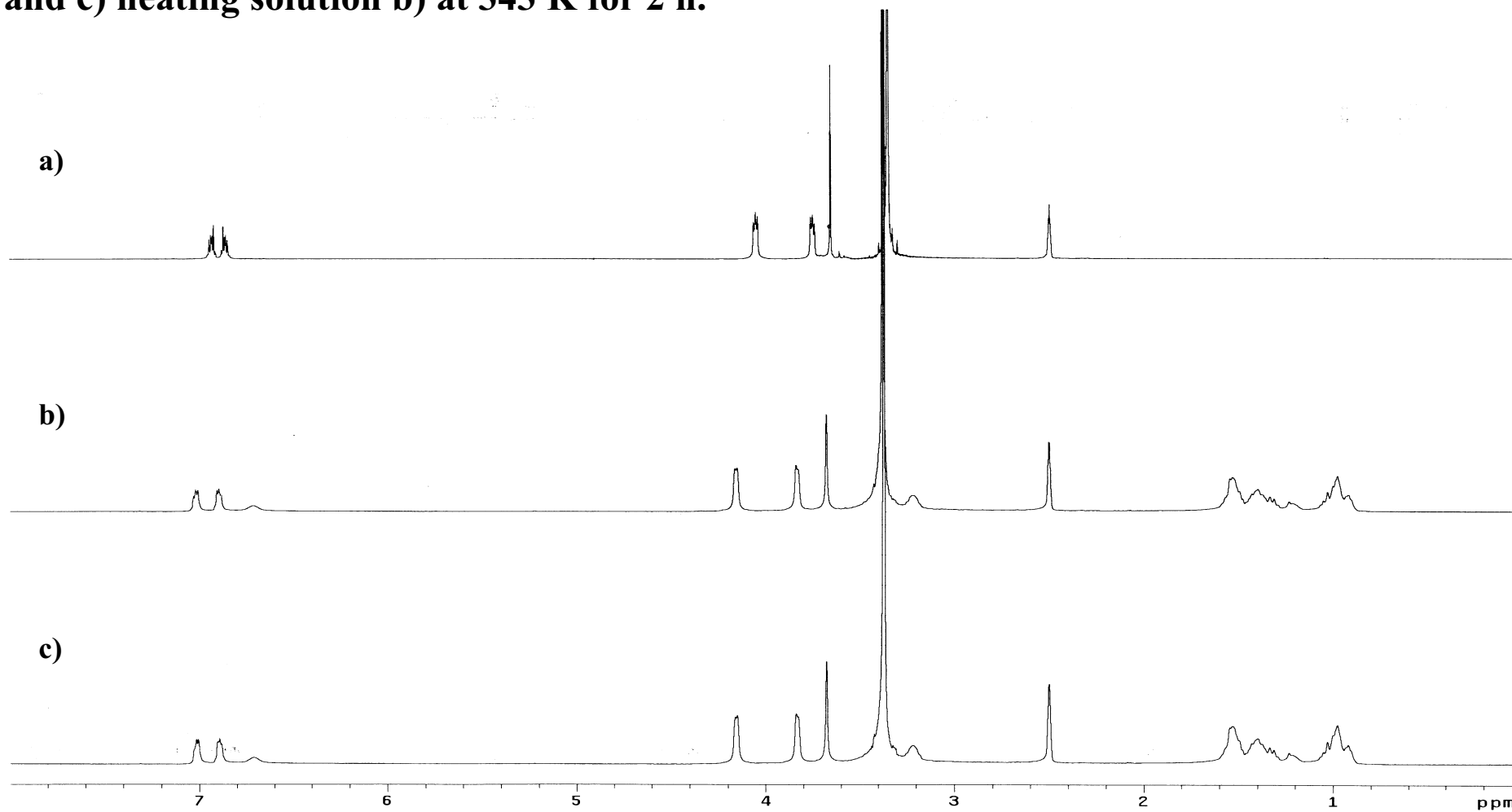
**The swelling synthesis of [2]rotaxane from DB24C8 and 20-H·PF<sub>6</sub> (70 mM) in CDCl<sub>3</sub> at 318 K**



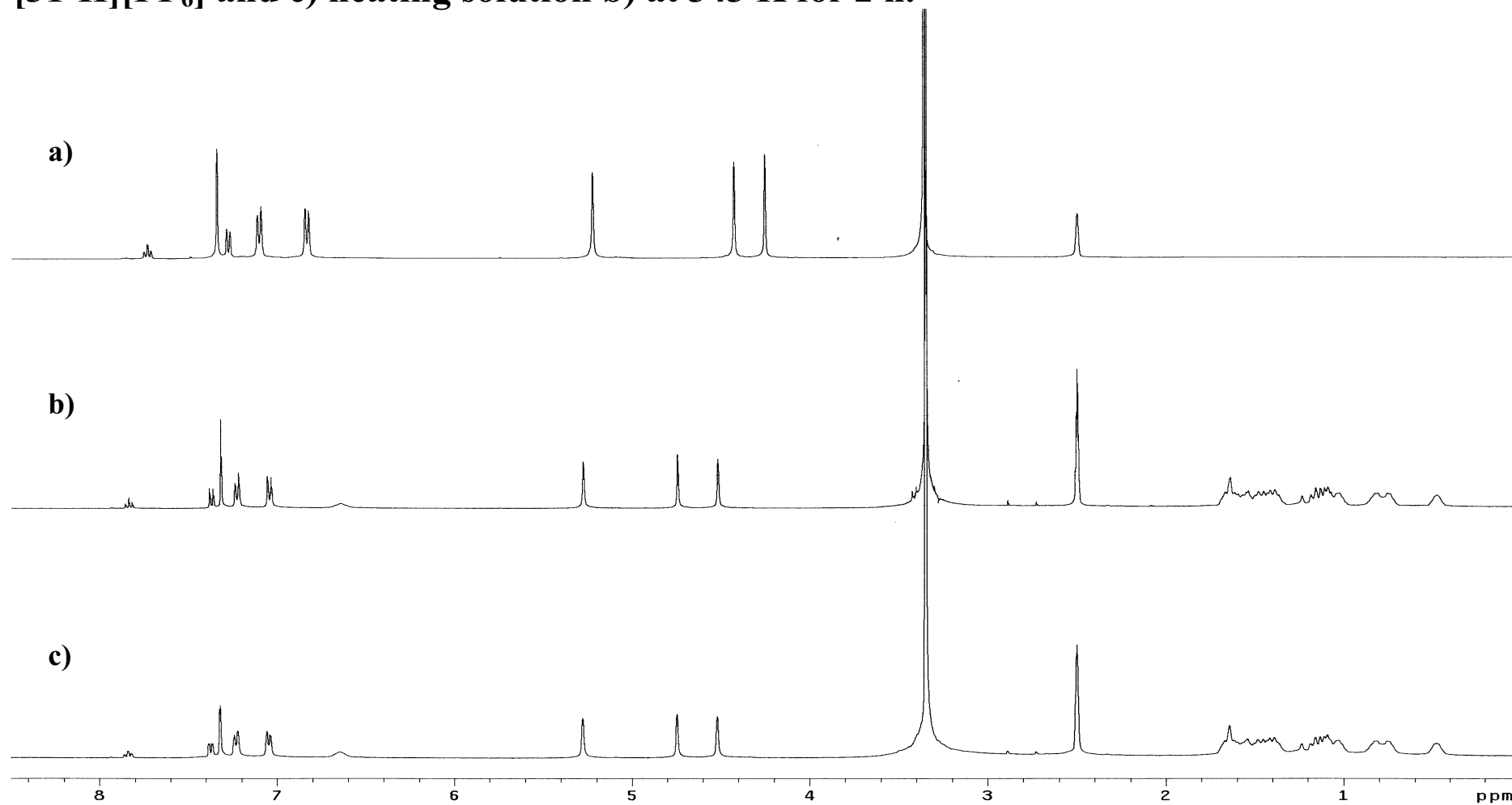
# The swelling synthesis of [2]rotaxane from 13 and 20-H·PF<sub>6</sub> (70 mM) in CD<sub>3</sub>NO<sub>2</sub> at 318 K



**$^1\text{H}$  NMR spectra (400 MHz,  $\text{CD}_3\text{SOCD}_3$ , 298 K) of a) DB24C8, b) [2]rotaxane [29-H][PF<sub>6</sub>] and c) heating solution b) at 343 K for 2 h.**

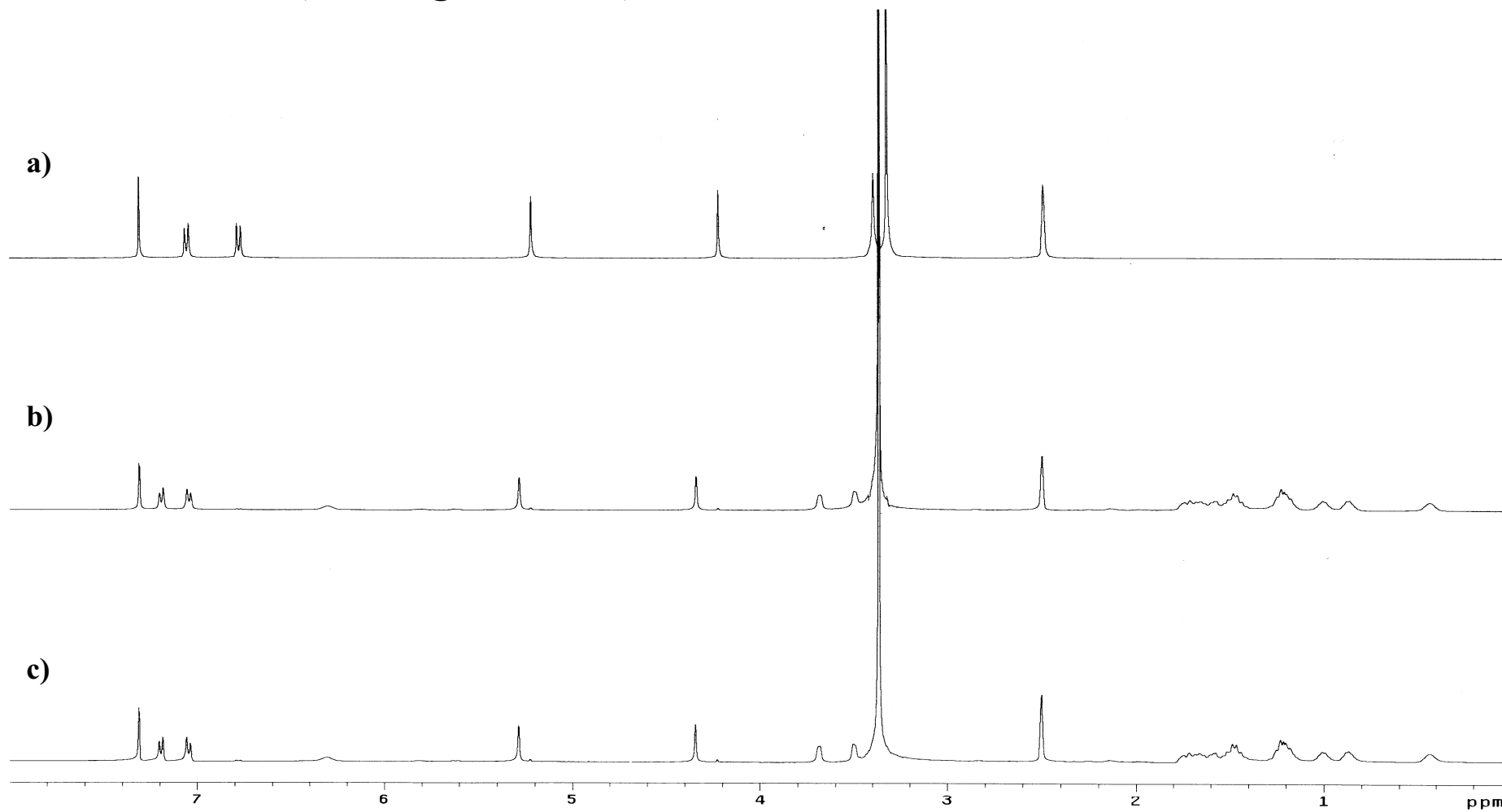


**$^1\text{H}$  NMR spectra (400 MHz,  $\text{CD}_3\text{SOCD}_3$ , 298 K) of a) macrocycle 12, b) [2]rotaxane [31-H][ $\text{PF}_6$ ] and c) heating solution b) at 343 K for 2 h.**





**$^1\text{H}$  NMR spectra (400 MHz,  $\text{CD}_3\text{SOCD}_3$ , 298 K) of a) macrocycle 13, b) [2]rotaxane [33-H][ $\text{PF}_6$ ] and c) heating solution b) at 343 K for 2 h.**



**$^1\text{H}$  NMR spectra (400 MHz,  $\text{CD}_3\text{SOCD}_3$ , 298 K) of a) macrocycle 12, b) [2]rotaxane [15-H][ $\text{PF}_6$ ] and c) heating solution b) at 343 K for 2 h.**

