

AMINOPHENYLPYRROLE SYNTHESIS AND APPLICATION TO PYRROLO[1,2-*c*]QUINAZOLINONE SYNTHESIS.

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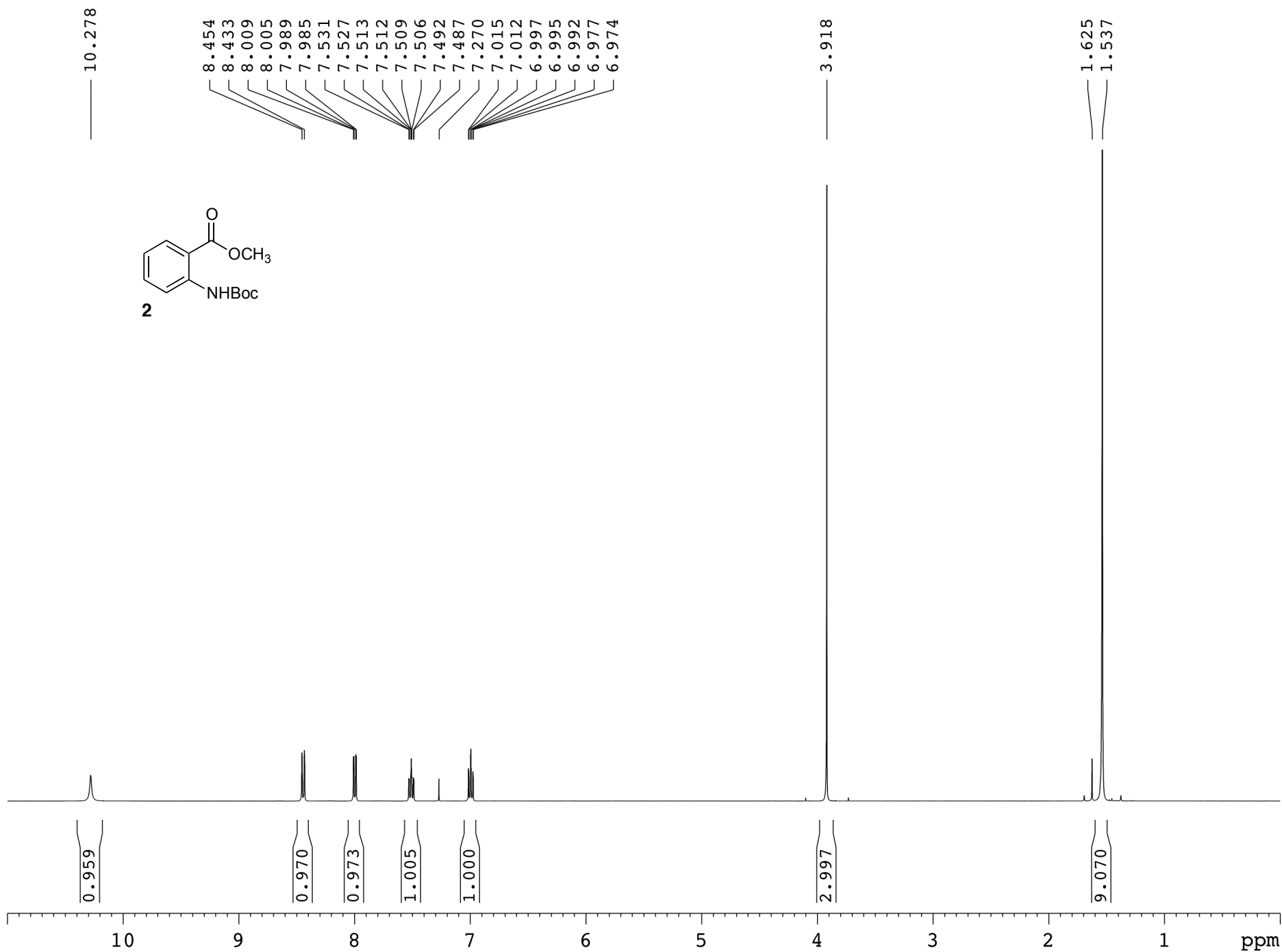
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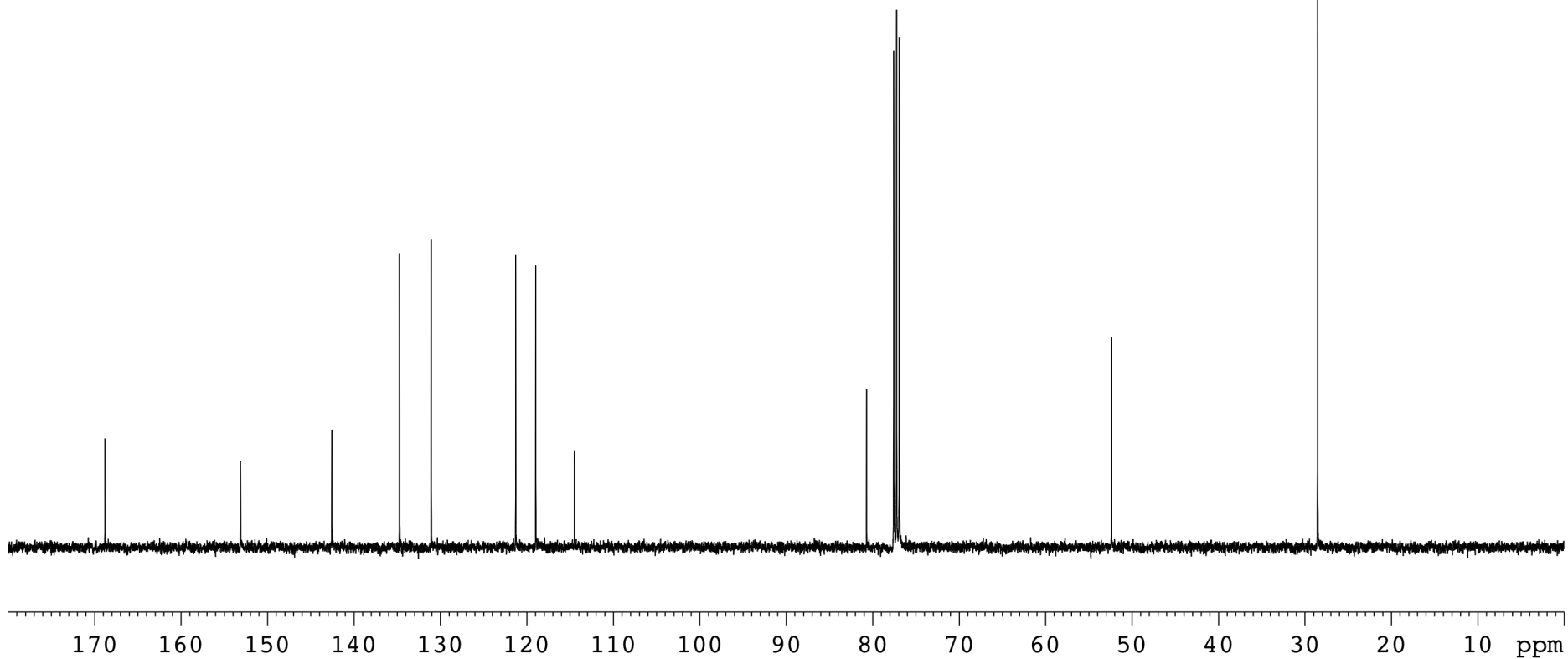
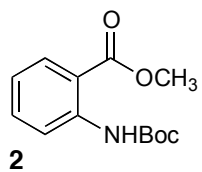
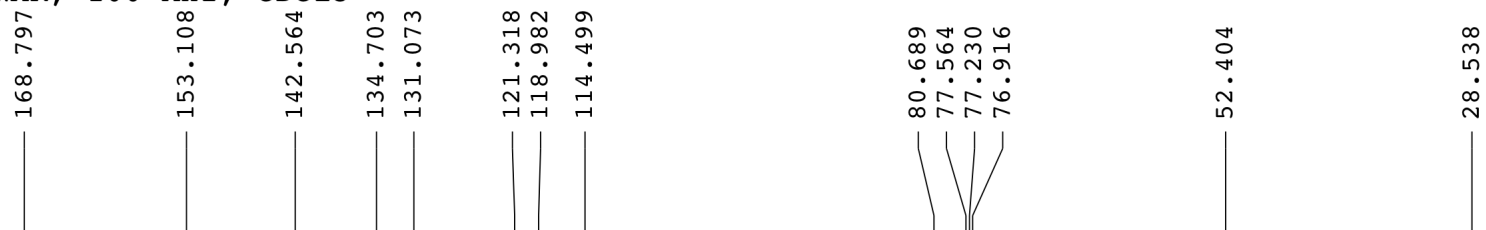
Proton NMR spectrum for **2**

¹H NMR, 400 MHz, CDCl₃



Carbon NMR spectrum for **2**

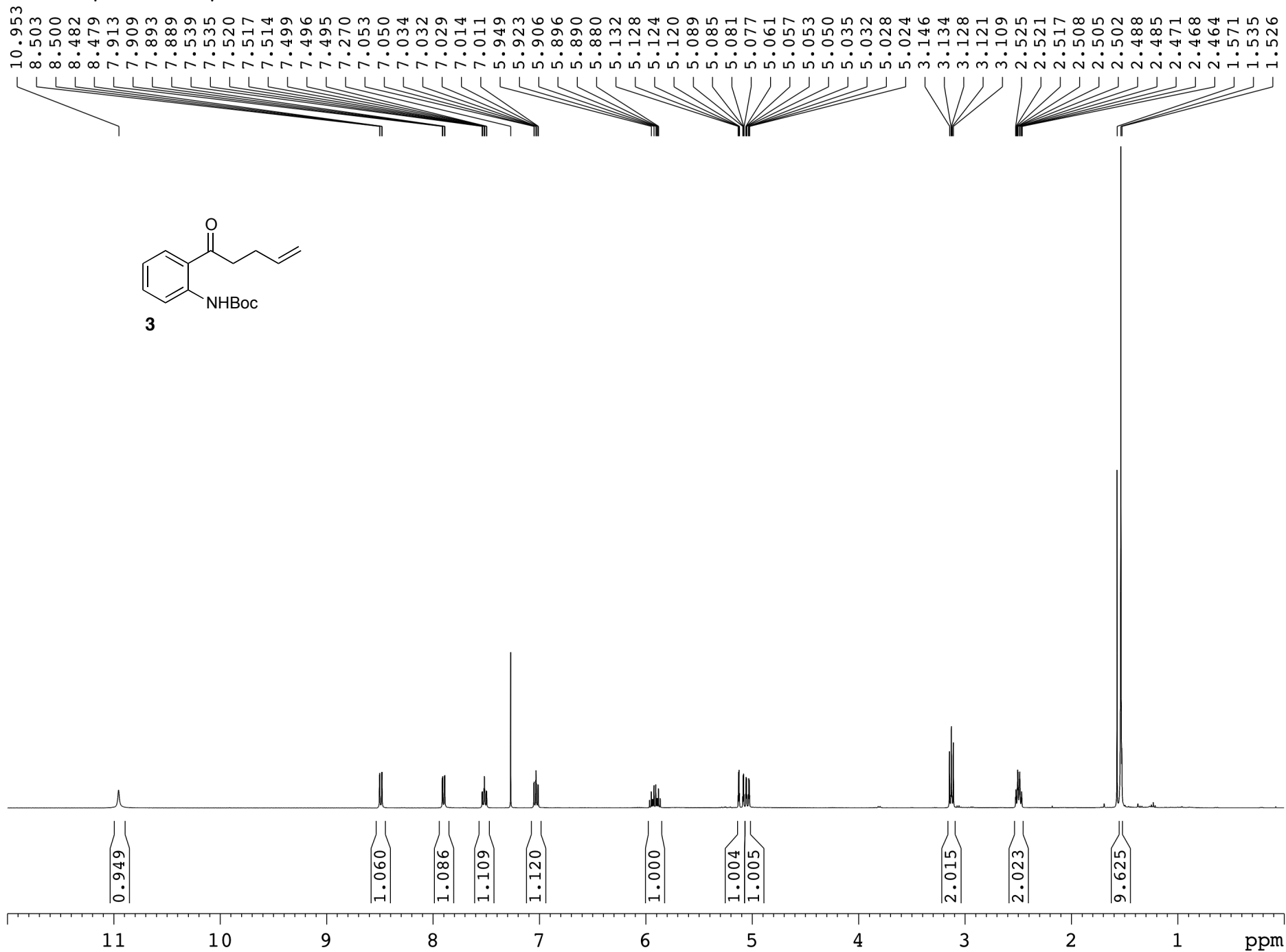
^{13}C NMR, 100 MHz, CDCl_3



Proton NMR spectrum for **3**

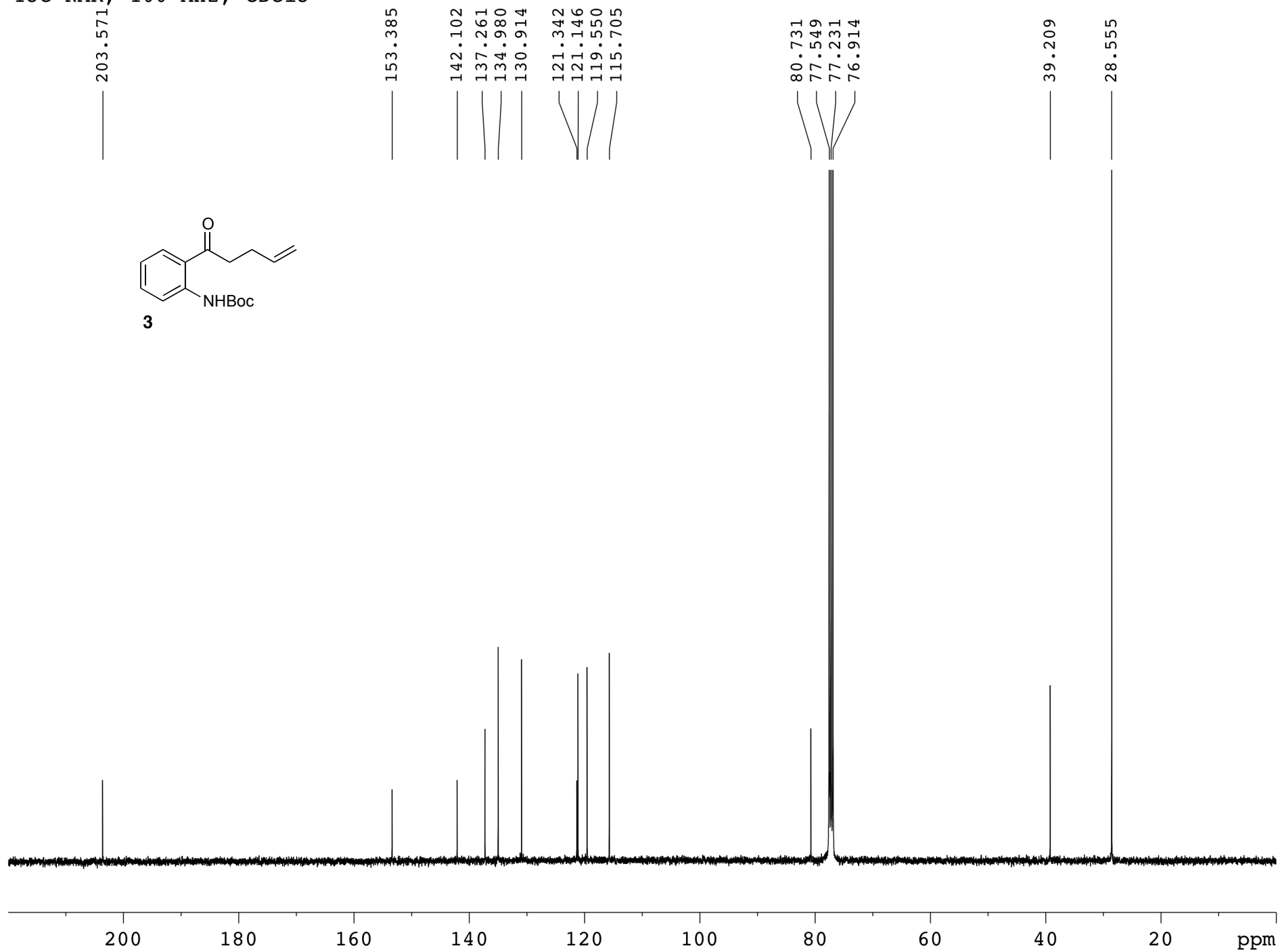
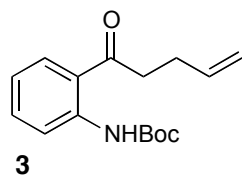
SI 6

¹H NMR, 400 MHz, CDCl₃



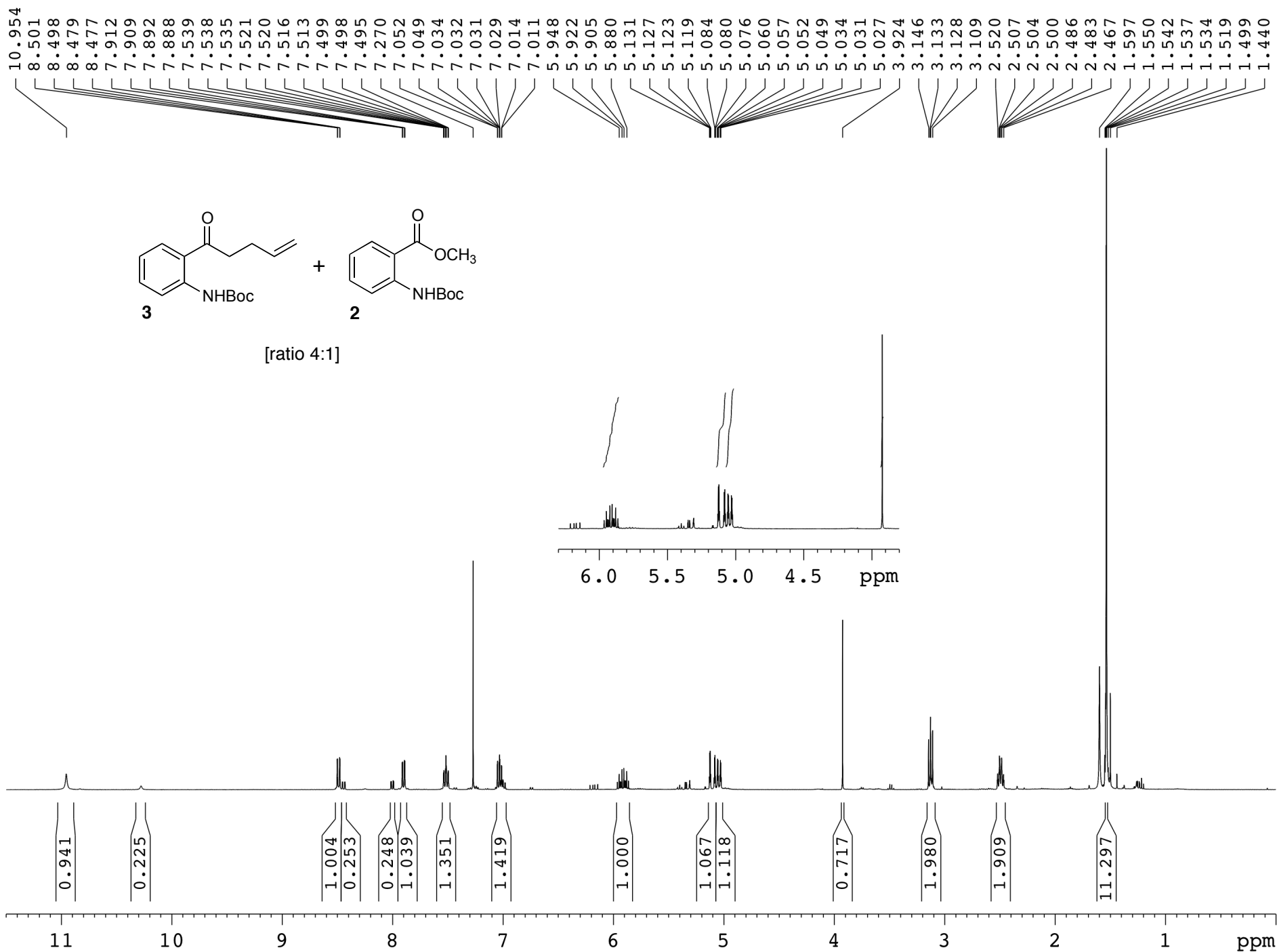
Carbon NMR spectrum for **3**

^{13}C NMR, 100 MHz, CDCl_3



Proton NMR spectrum for 4:1 mixture of **3** and **2**

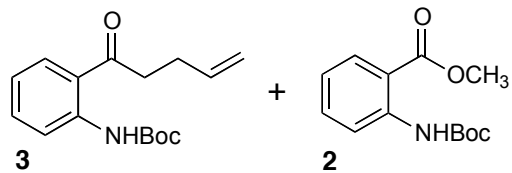
¹H NMR, 400 MHz, CDCl₃



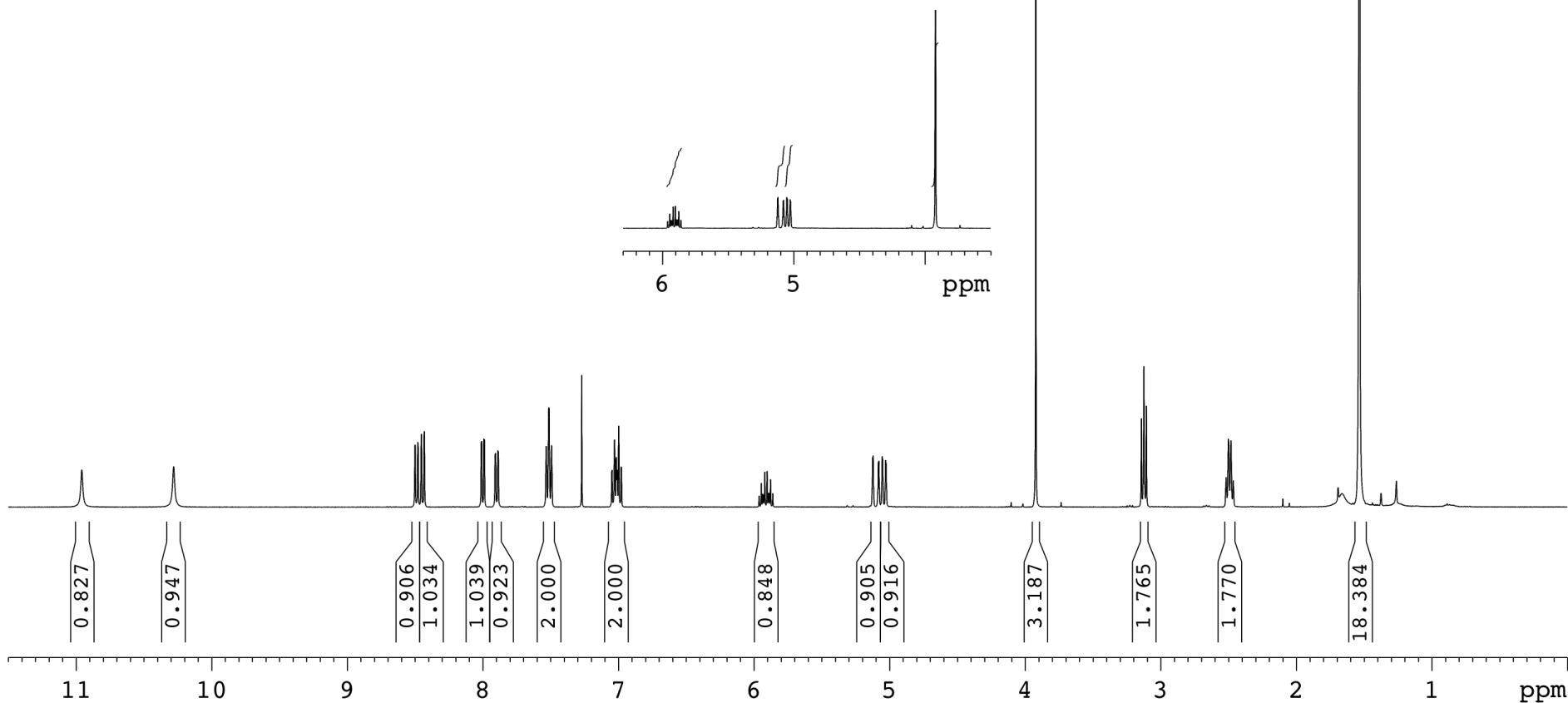
Proton NMR spectrum for 1:1 mixture of **3** and **2**

¹H NMR, 400 MHz, CDCl₃

10.956, 10.278, 8.499, 8.479, 8.452, 8.432, 8.012, 8.008, 7.992, 7.988, 7.909, 7.906, 7.889, 7.886, 7.532, 7.514, 7.511, 7.493, 7.270, 7.049, 7.046, 7.029, 7.018, 7.015, 7.011, 7.008, 6.997, 6.980, 6.977, 5.962, 5.946, 5.937, 5.930, 5.920, 5.903, 5.894, 5.887, 5.878, 5.861, 5.129, 5.125, 5.121, 5.117, 5.086, 5.082, 5.078, 5.074, 5.055, 5.051, 5.029, 5.025, 3.735, 3.142, 3.124, 3.106, 2.518, 2.501, 2.499, 2.482, 2.465, 2.099, 1.692, 1.663, 1.438, 1.375, 1.261

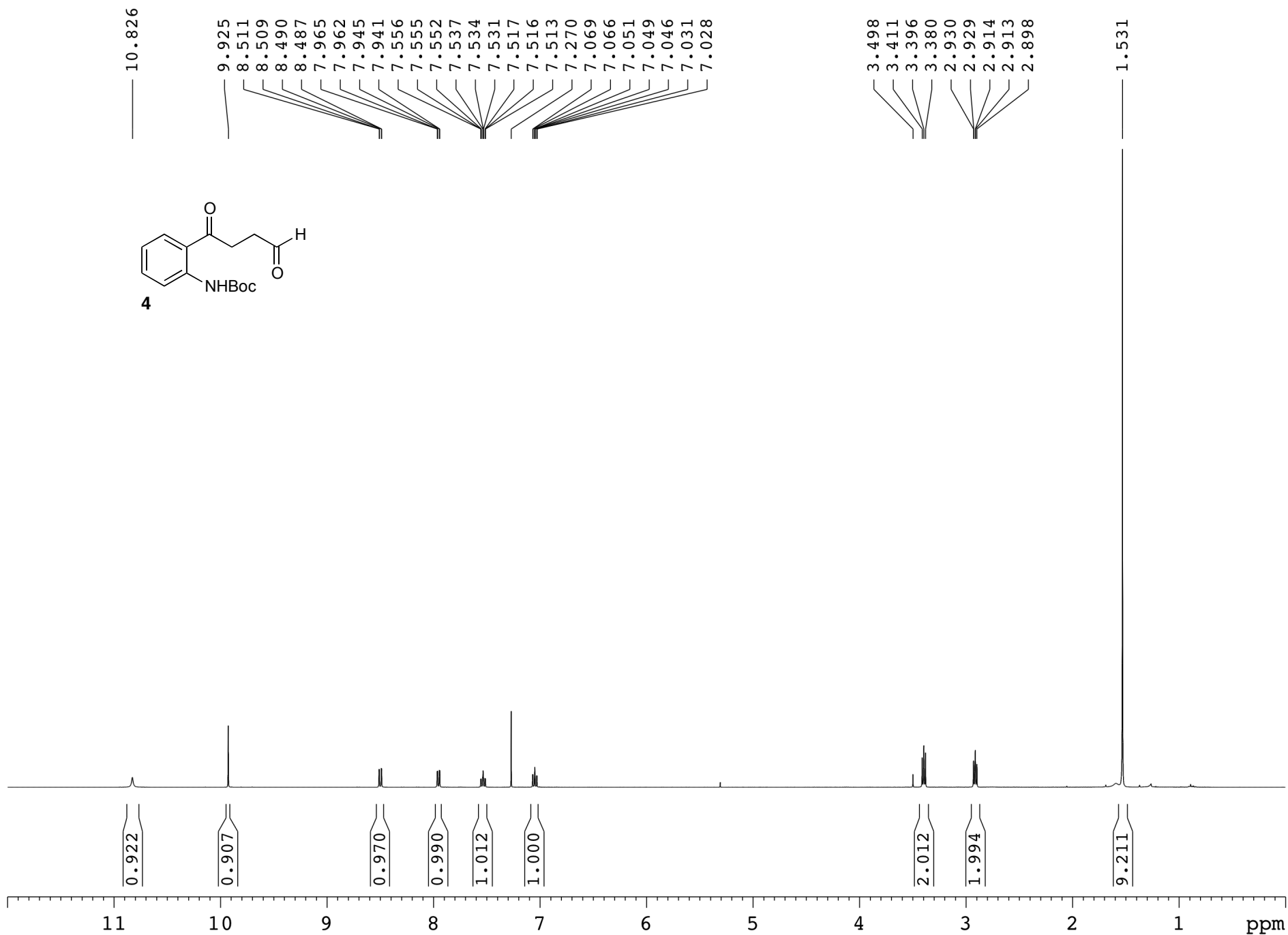


[ratio 1:1]



Proton NMR spectrum for **4**

¹H NMR, 400 MHz, CDCl₃



Carbon NMR spectrum for 4

^{13}C NMR, 100 MHz, CDCl_3

201.522
200.672

153.273

142.166

135.290

130.882

121.227

120.899

119.522

80.814

77.547

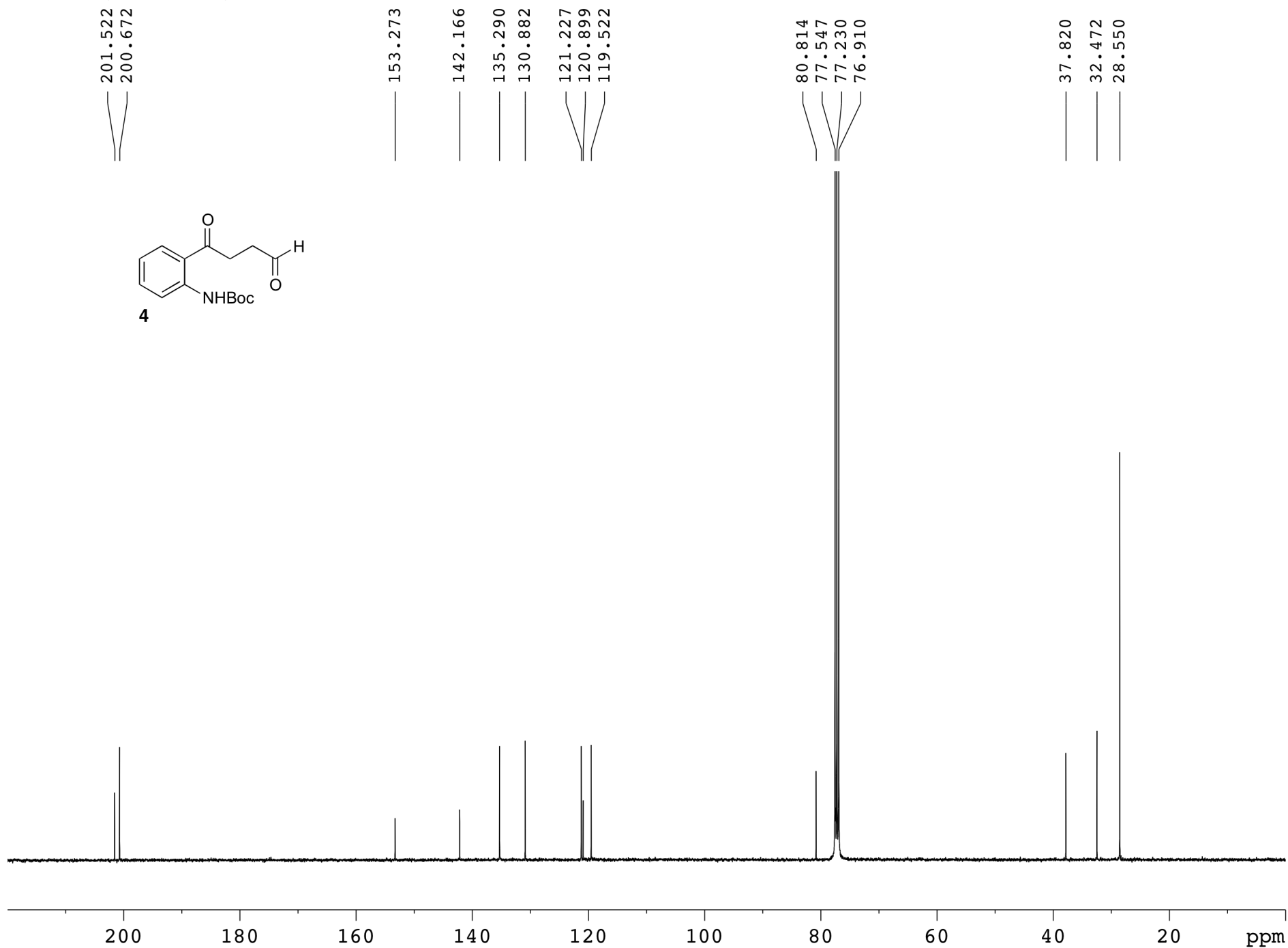
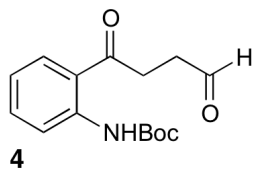
77.230

76.910

37.820

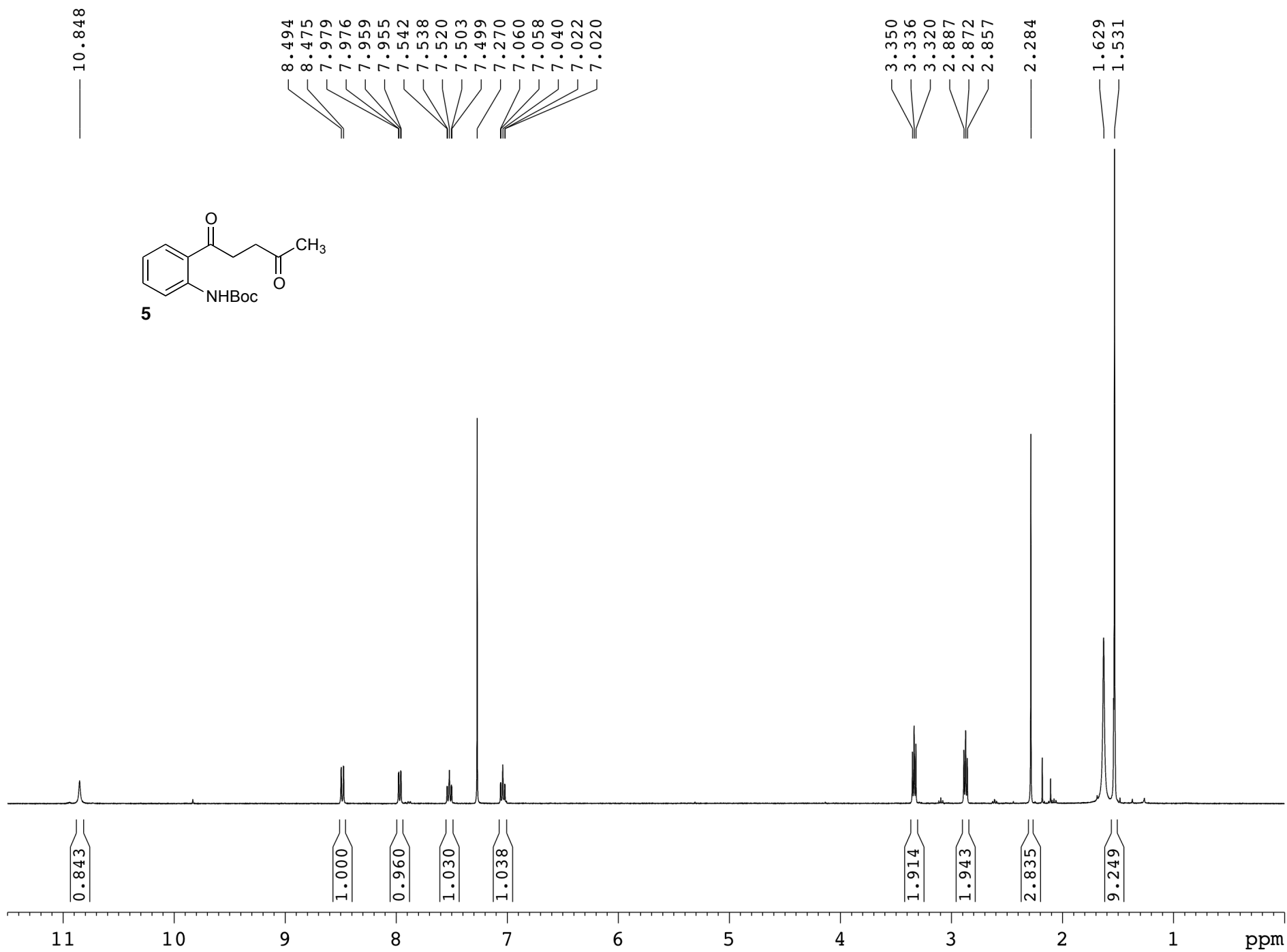
32.472

28.550

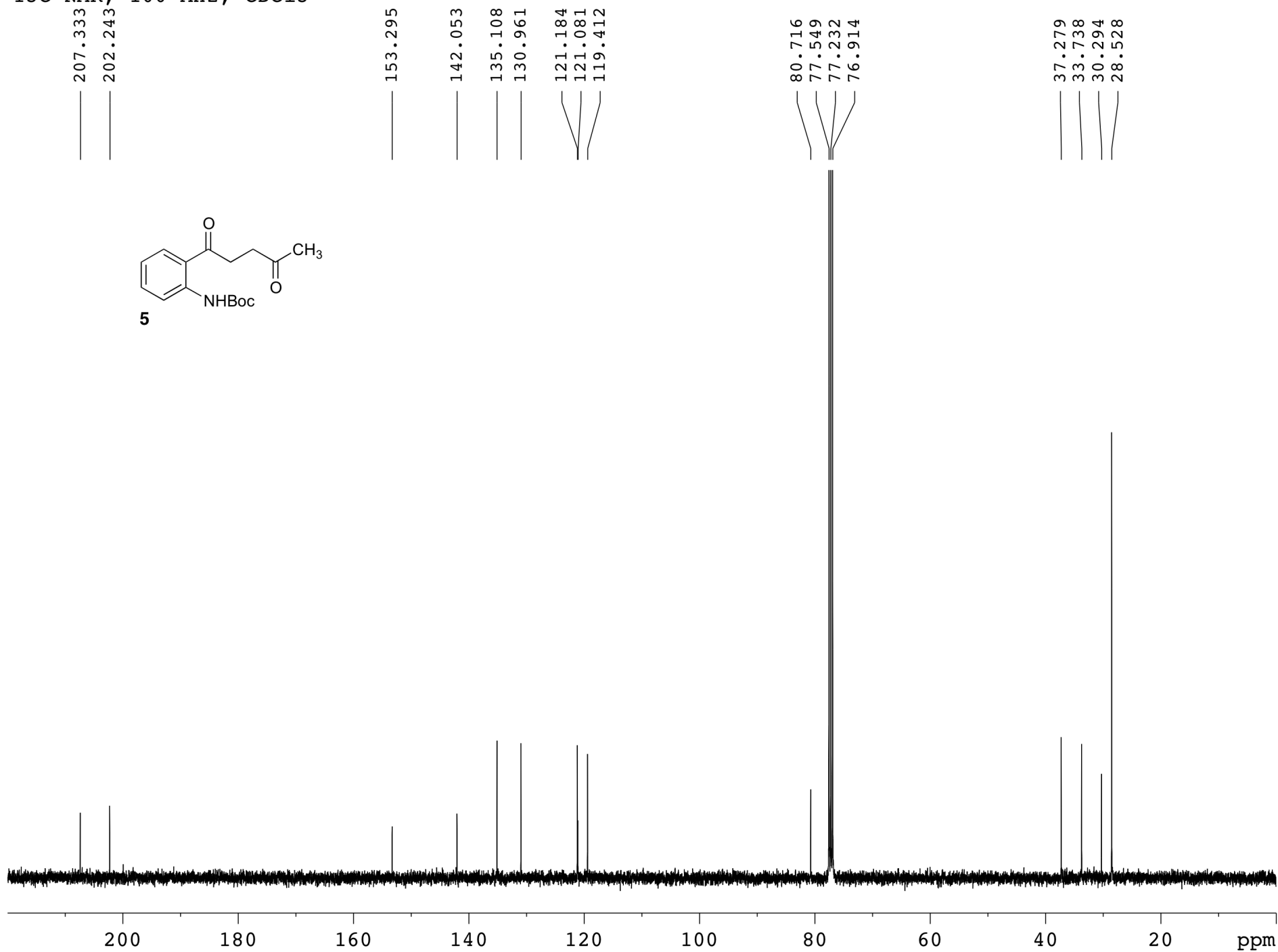
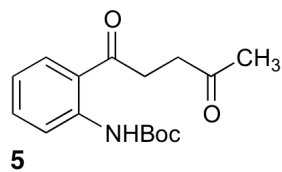


Proton NMR spectrum for **5**

¹H NMR, 400 MHz, CDCl₃

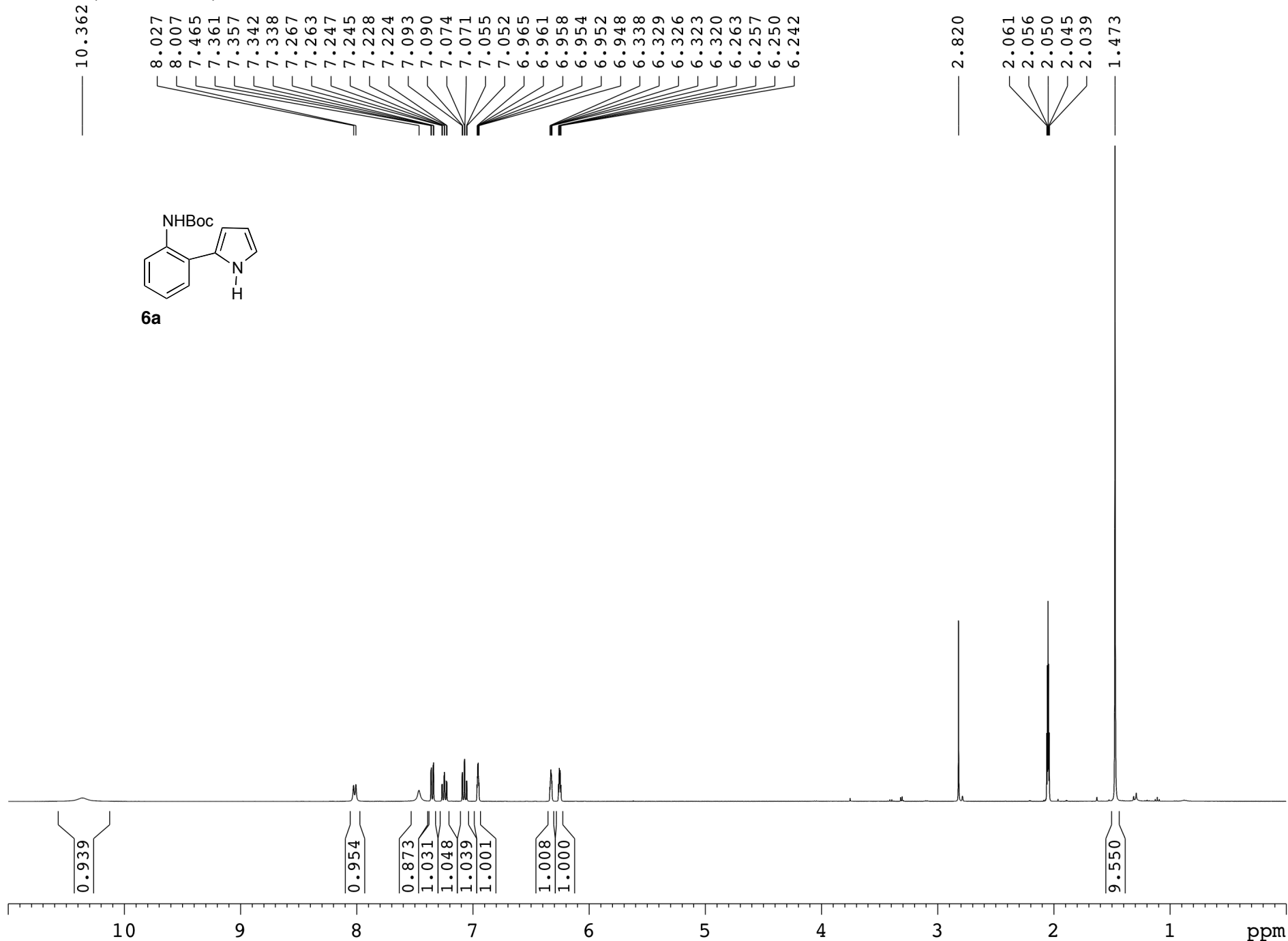


Carbon NMR spectrum for **5**
13C NMR, 100 MHz, CDCl3



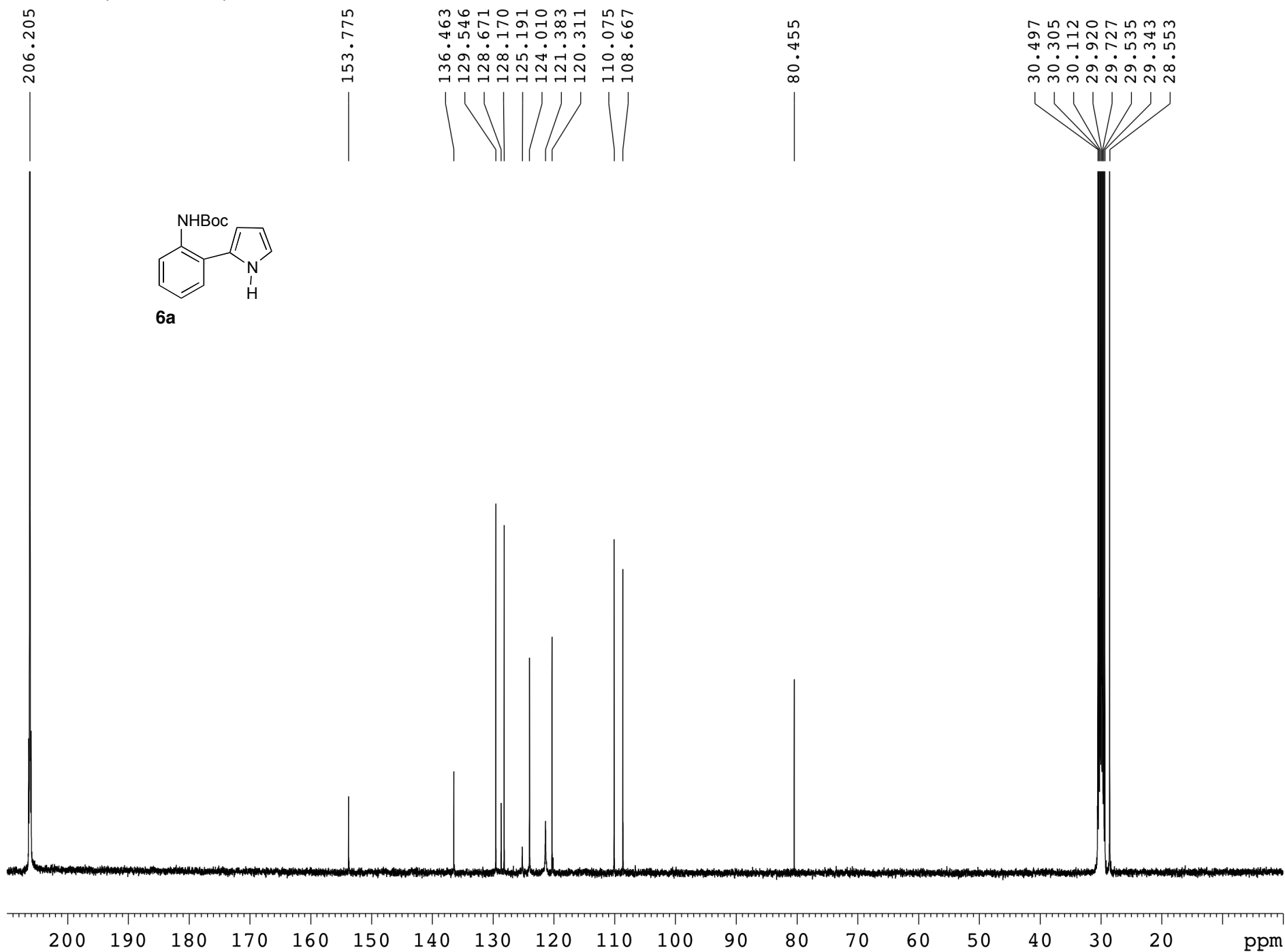
Proton NMR spectrum for **6a**

¹H NMR, 400 MHz, acetone-d₆



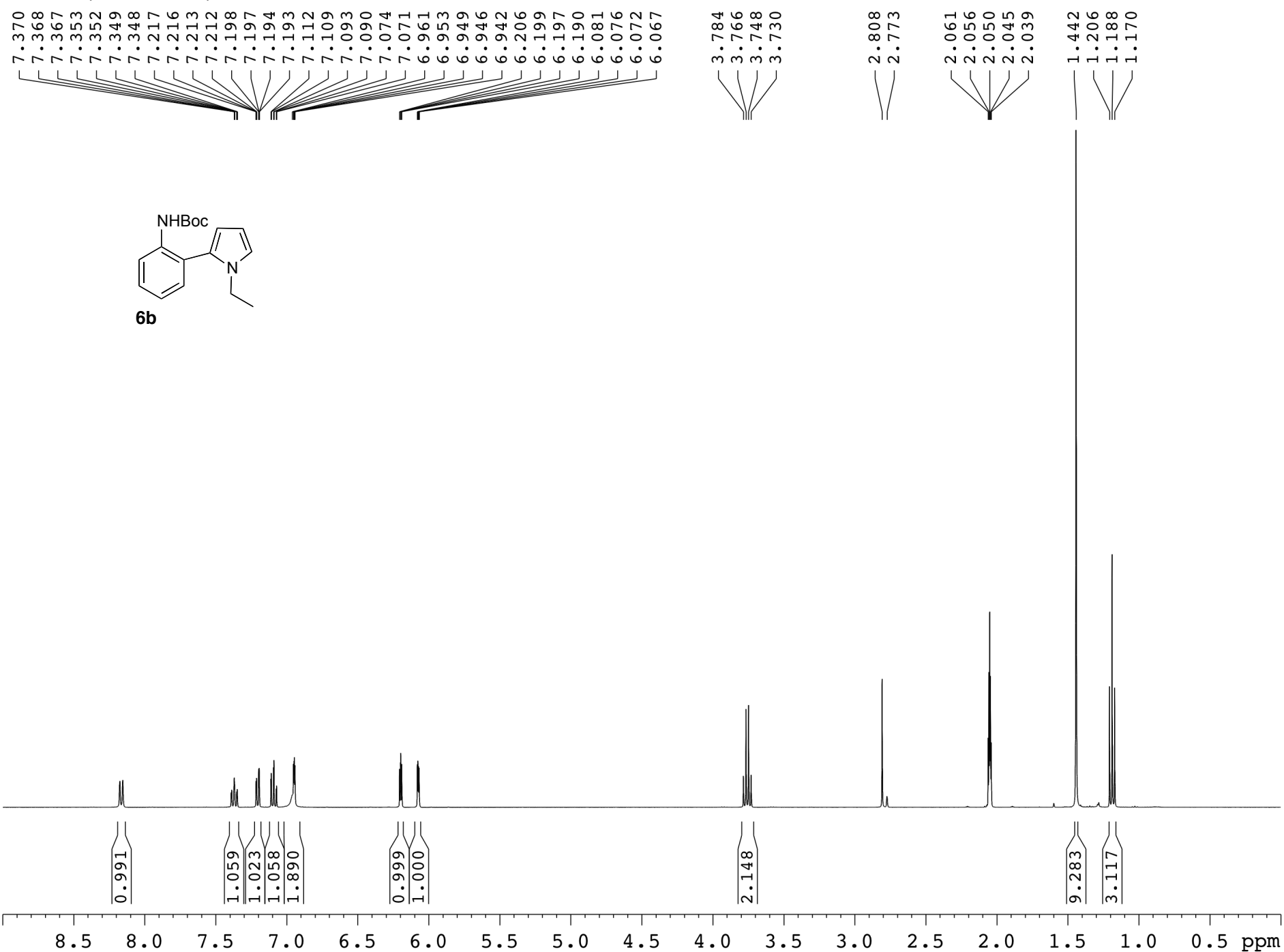
Carbon NMR spectrum for **6a**

^{13}C NMR, 100 MHz, acedone- d_6



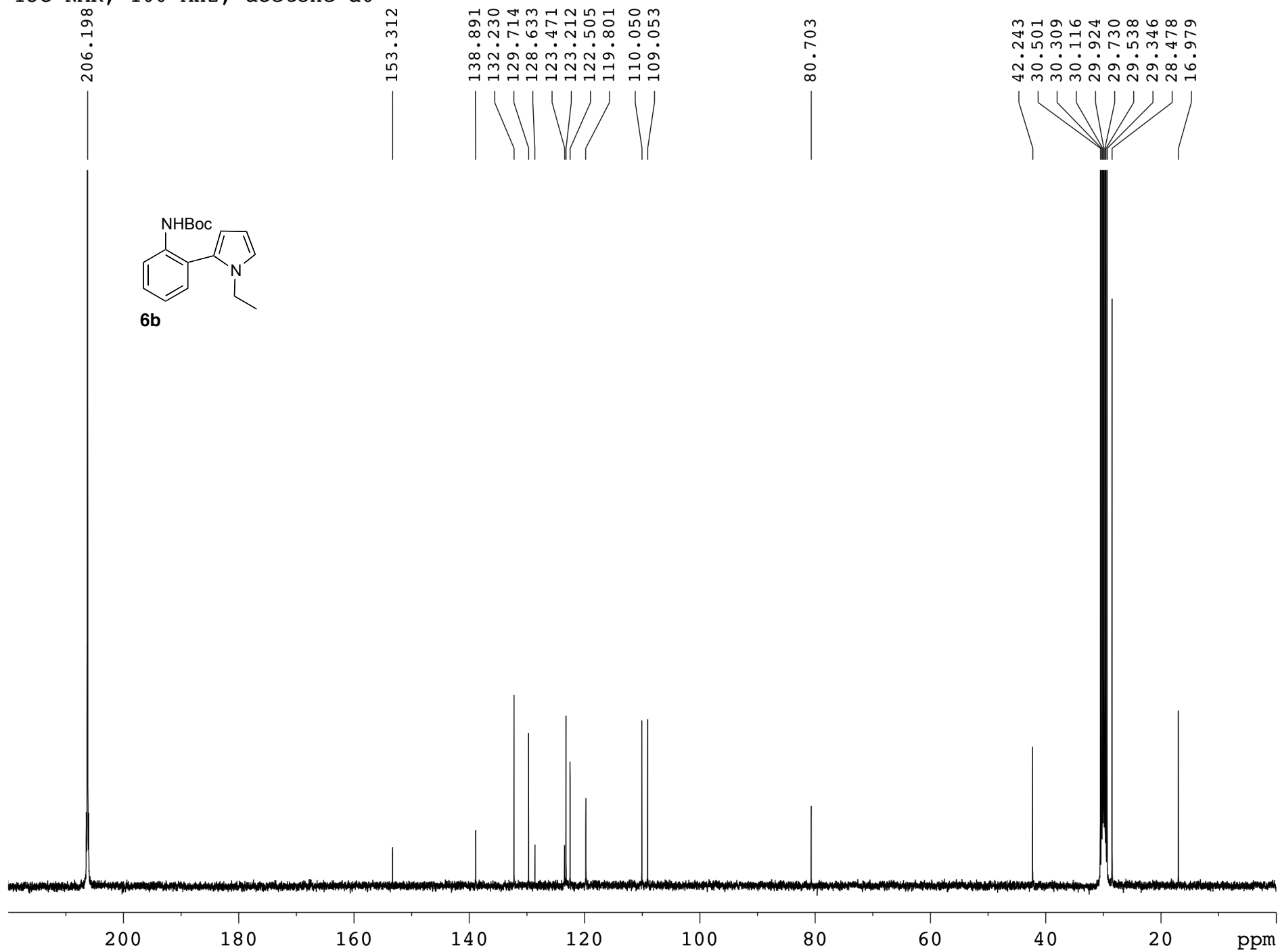
Proton NMR spectrum for **6b**

¹H NMR, 400 MHz, acetone-d₆



Carbon NMR spectrum for **6b**

¹³C NMR, 100 MHz, acetone-d₆

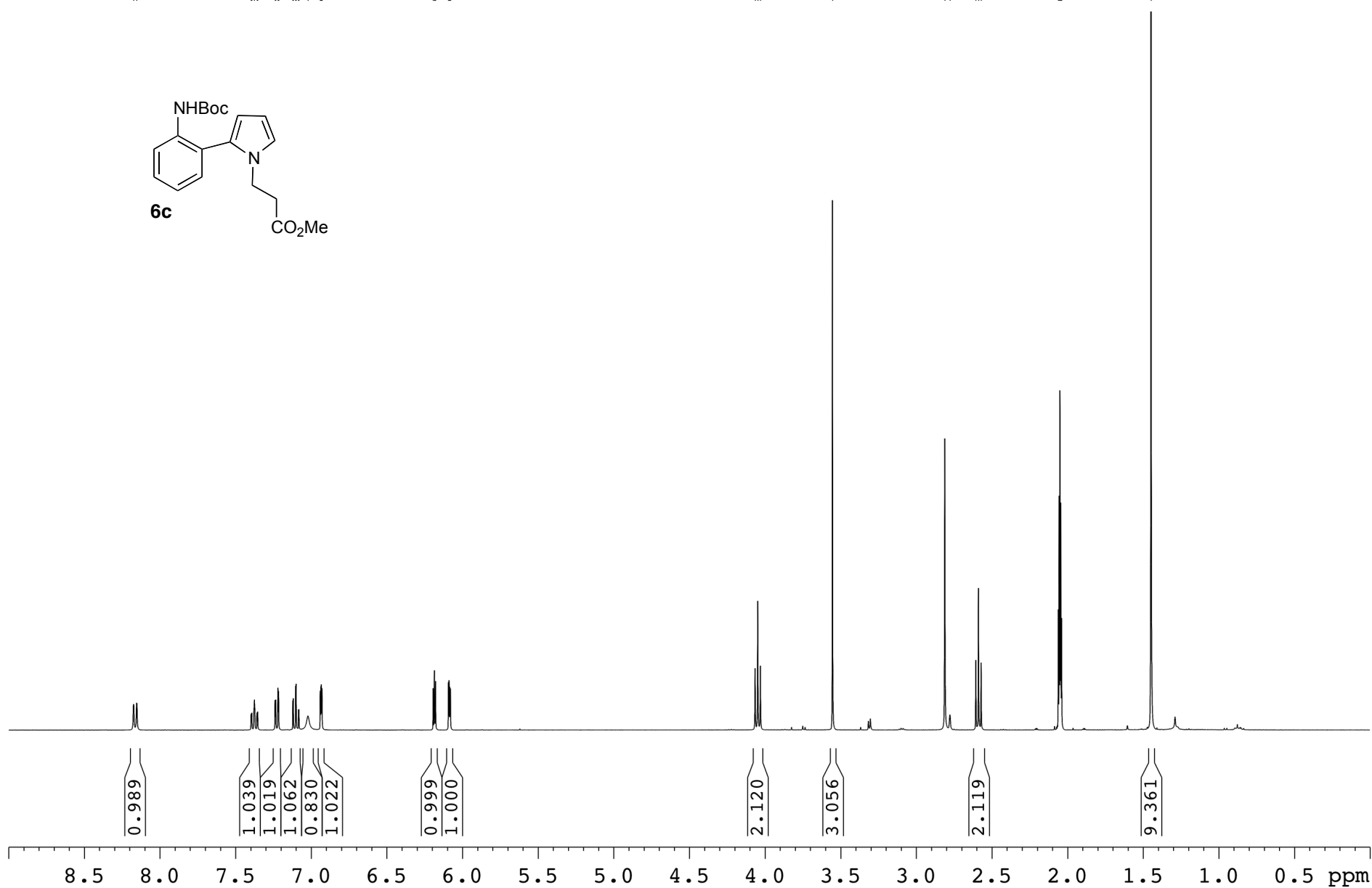
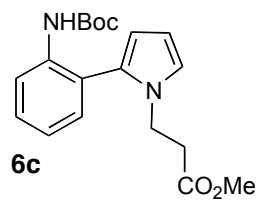


Proton NMR spectrum for **6c**

¹H NMR, 400 MHz, acetone-d₆

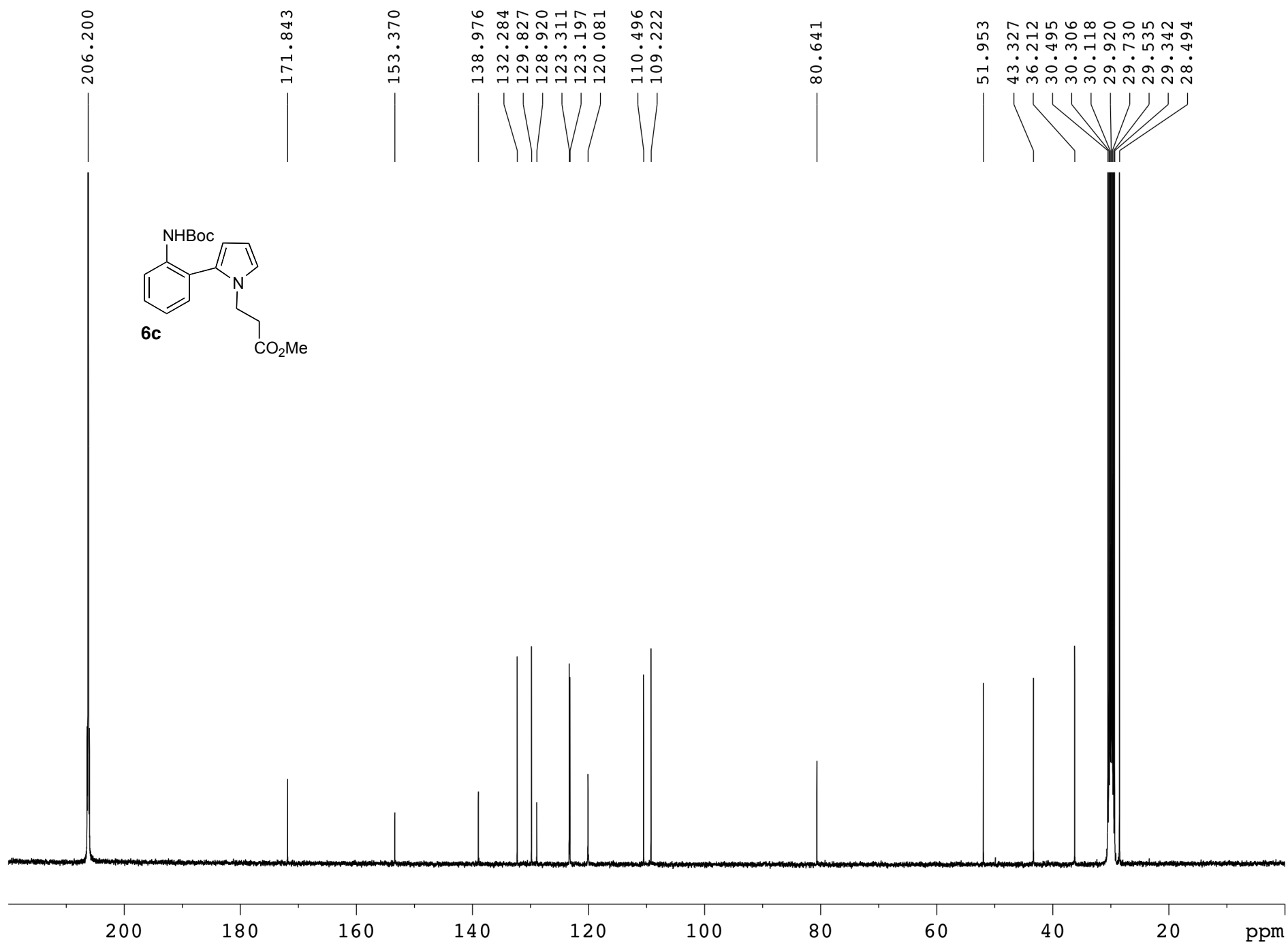
8.174
8.154
7.397
7.393
7.376
7.358
7.354
7.239
7.235
7.220
7.216
7.122
7.119
7.103
7.100
7.084
7.081
7.022
7.022
6.940
6.936
6.933
6.929
6.194
6.187
6.185
6.178
6.093
6.089
6.084
6.080

4.066
4.048
4.031
3.554
2.812
2.778
2.606
2.589
2.571
2.061
2.056
2.050
2.045
2.039
1.448



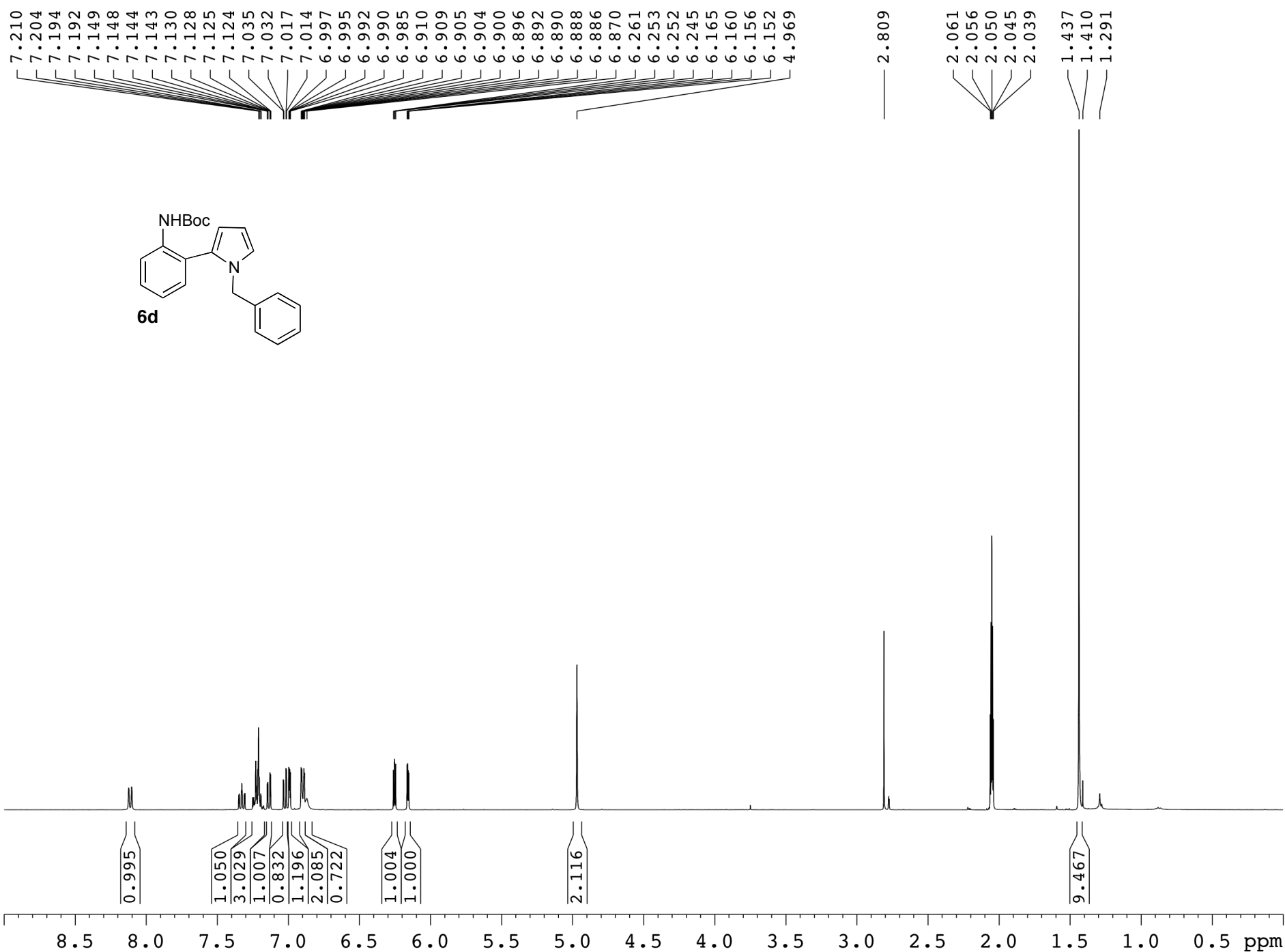
Carbon NMR spectrum for **6c**

¹³C NMR, 100 MHz, acetone-d₆



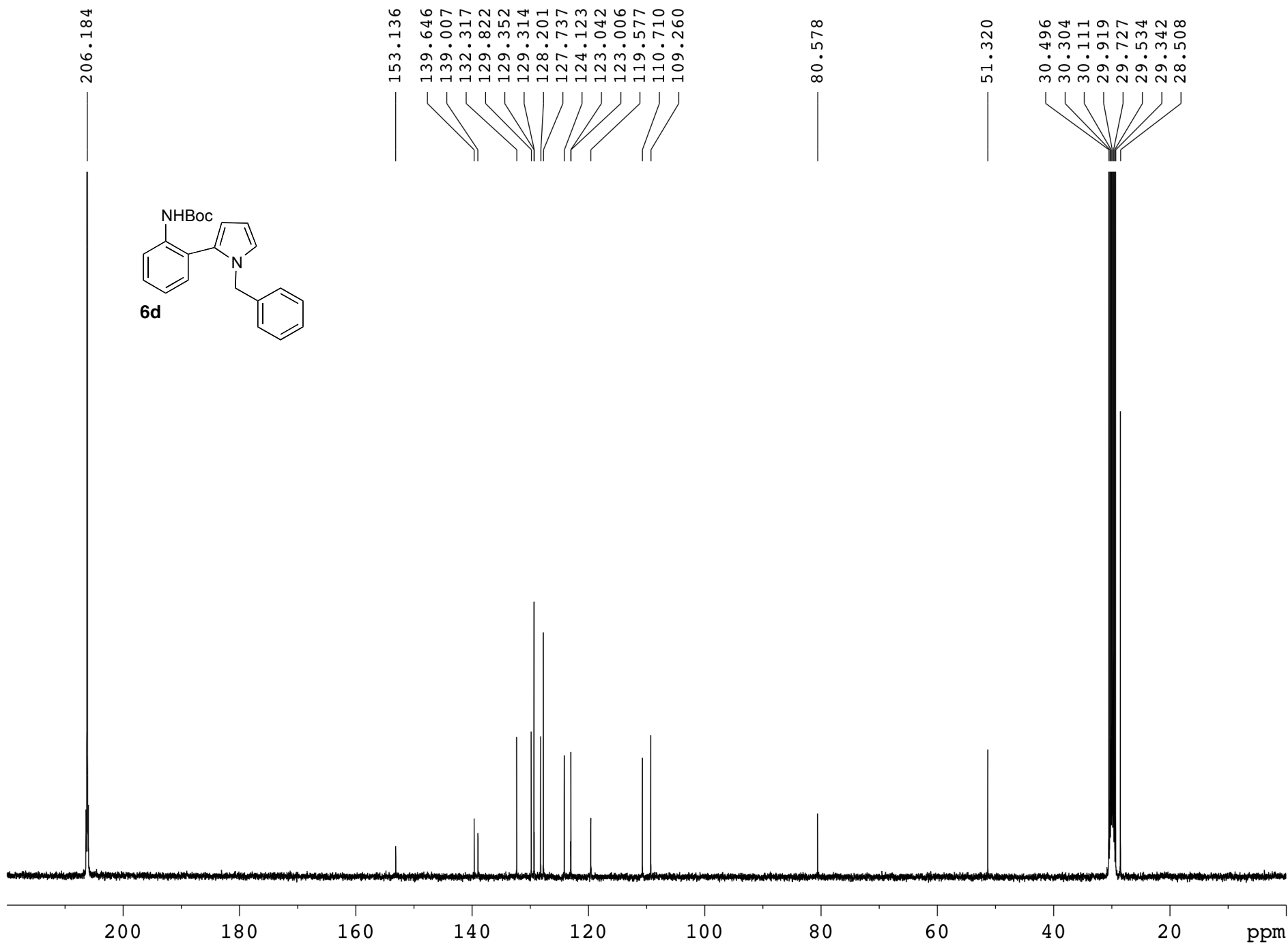
Proton NMR spectrum for **6d**

¹H NMR, 400 MHz, acetone-d₆



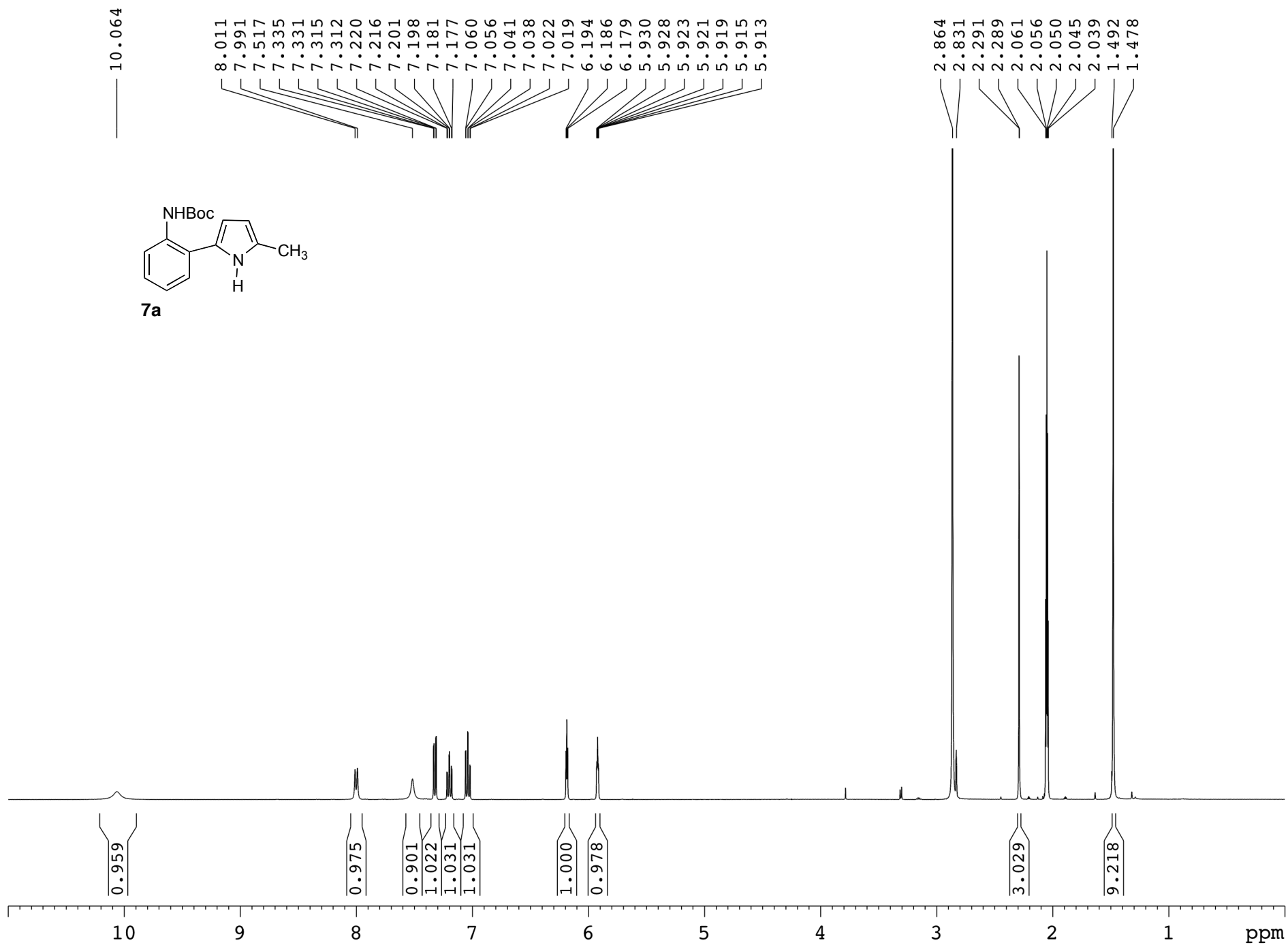
Carbon NMR spectrum for **6d**

^{13}C NMR, 100 MHz, acetone- d_6



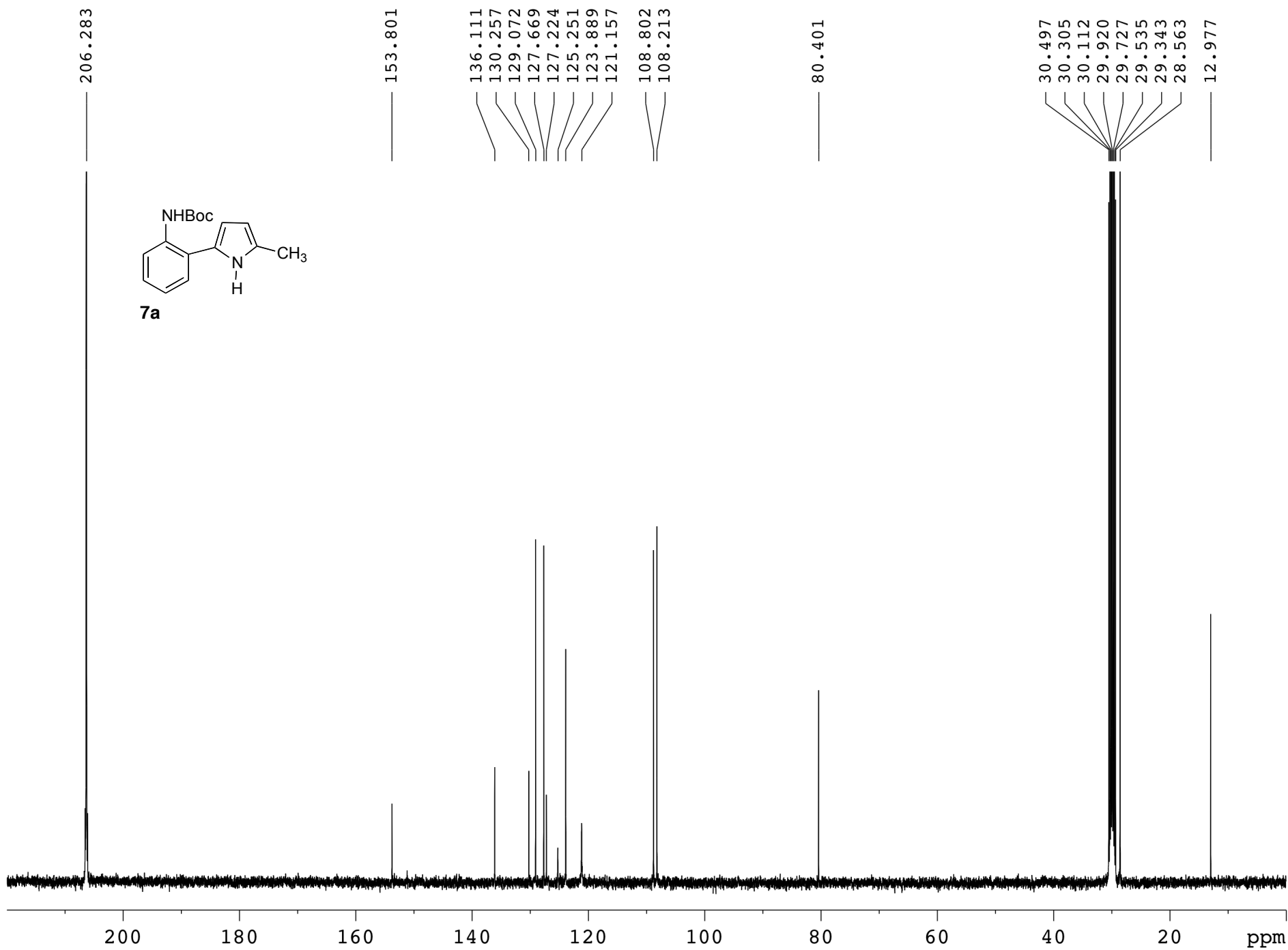
Proton NMR spectrum for **7a**

¹H NMR, 400 MHz, acetone-d₆



Carbon NMR spectrum for **7a**

^{13}C NMR, 100 MHz, acetone- d_6



Proton NMR spectrum for **7b**

¹H NMR, 400 MHz, acetone-d₆

8.193
8.172
7.378
7.374
7.356
7.339
7.335
7.208
7.204
7.189
7.185
7.096
7.093
7.078
7.075
7.059
7.056
7.013
5.976
5.968
5.941
5.939
5.933
5.931

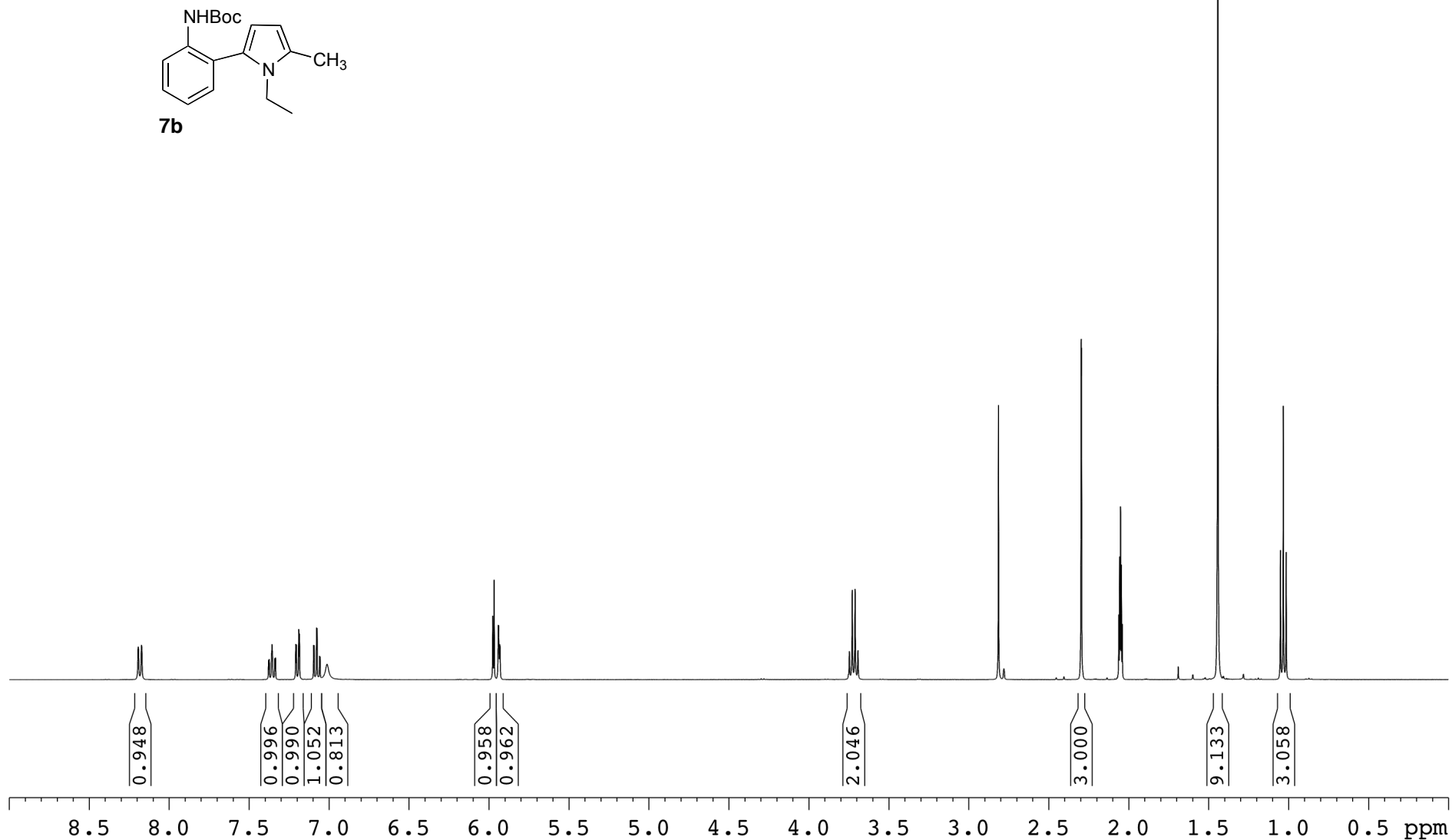
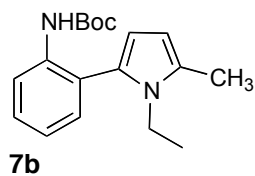
3.746
3.728
3.710
3.692

2.813

2.296
2.295
2.061
2.056
2.050
2.045
2.039

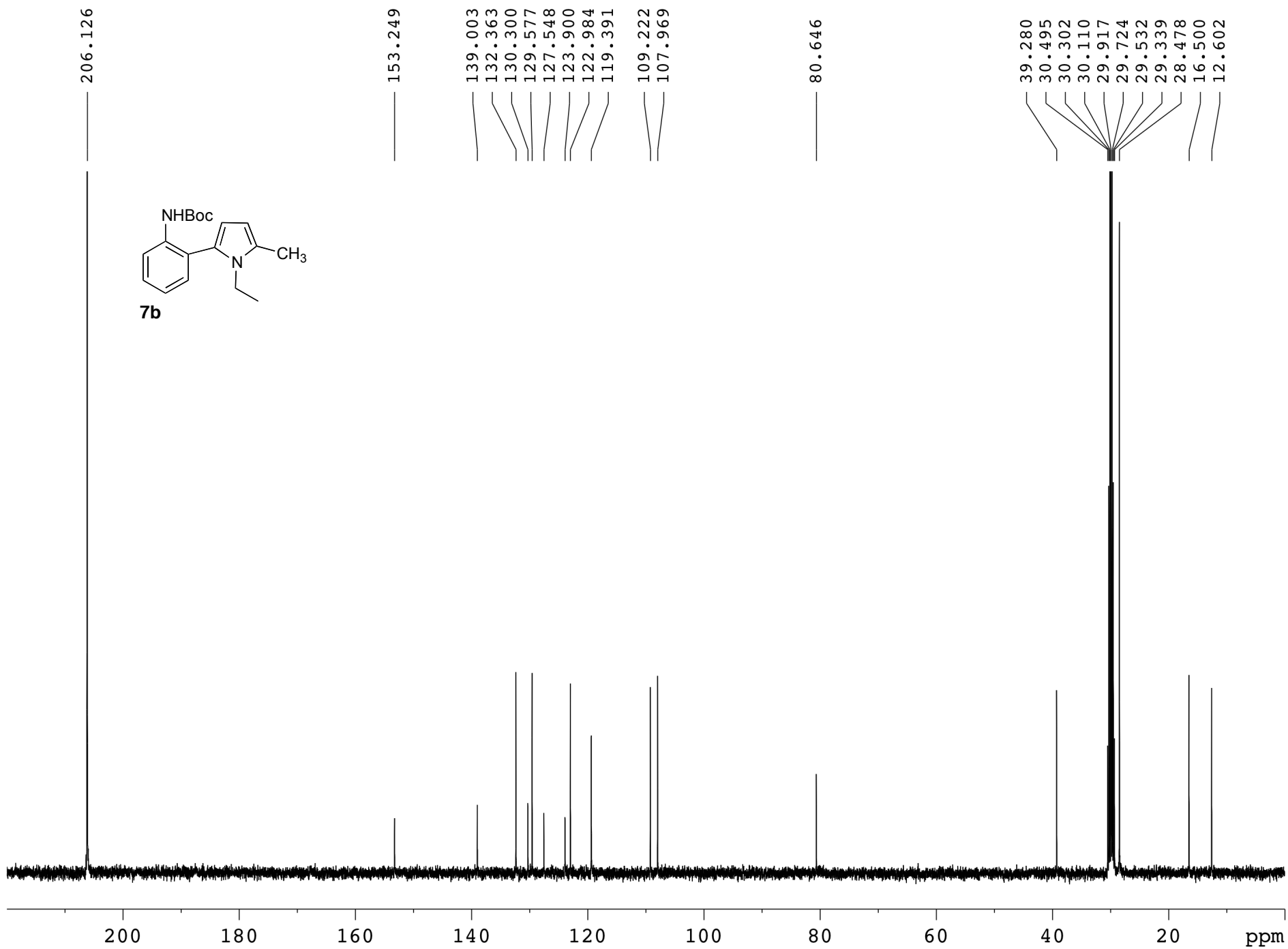
1.442

1.050
1.032
1.014



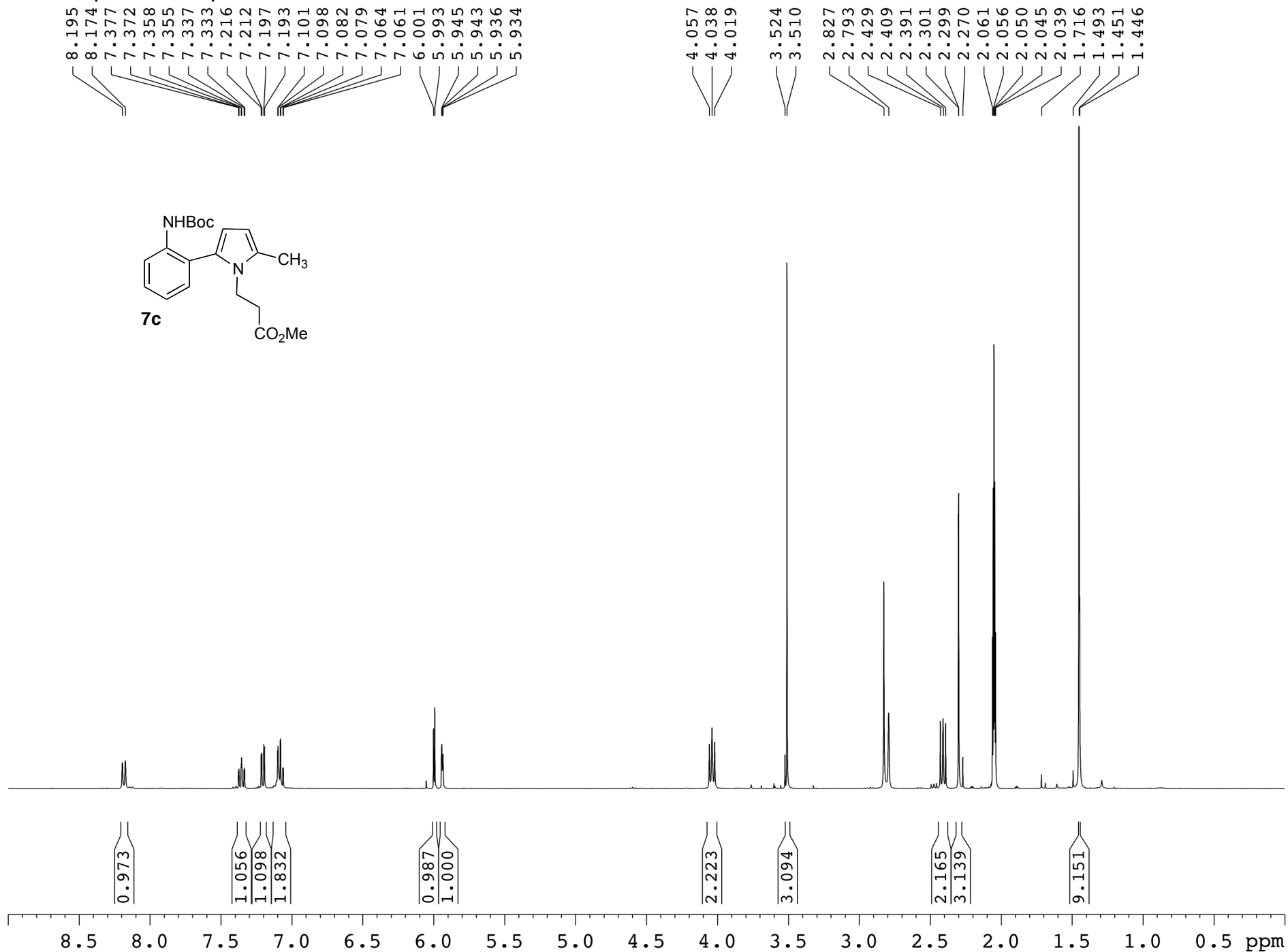
Carbon NMR spectrum for **7b**

¹³C NMR, 100 MHz, acetone-d₆



Proton NMR spectrum for **7c**

¹H NMR, 400 MHz, acetone-d₆



Carbon NMR spectrum for **7c**

^{13}C NMR, 100 MHz, acetone- d_6

206.428
206.235
206.035

171.601

153.358

138.949

132.236

130.877

129.665

127.845

123.728

123.169

119.735

109.882

108.225

80.639

51.921

40.388

35.661

30.558

30.500

30.364

30.307

30.167

30.115

29.923

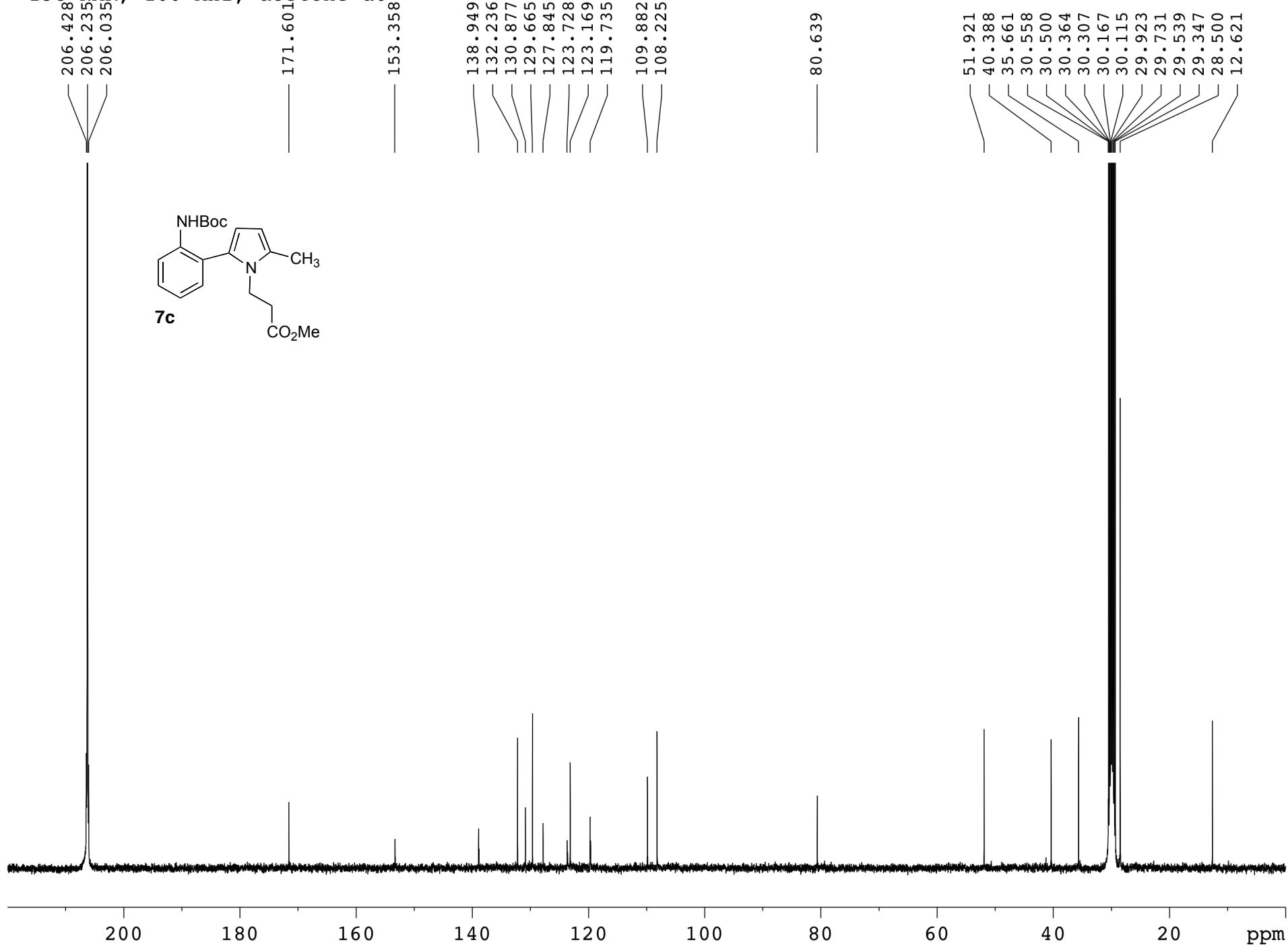
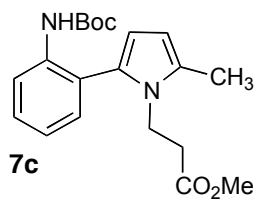
29.731

29.539

29.347

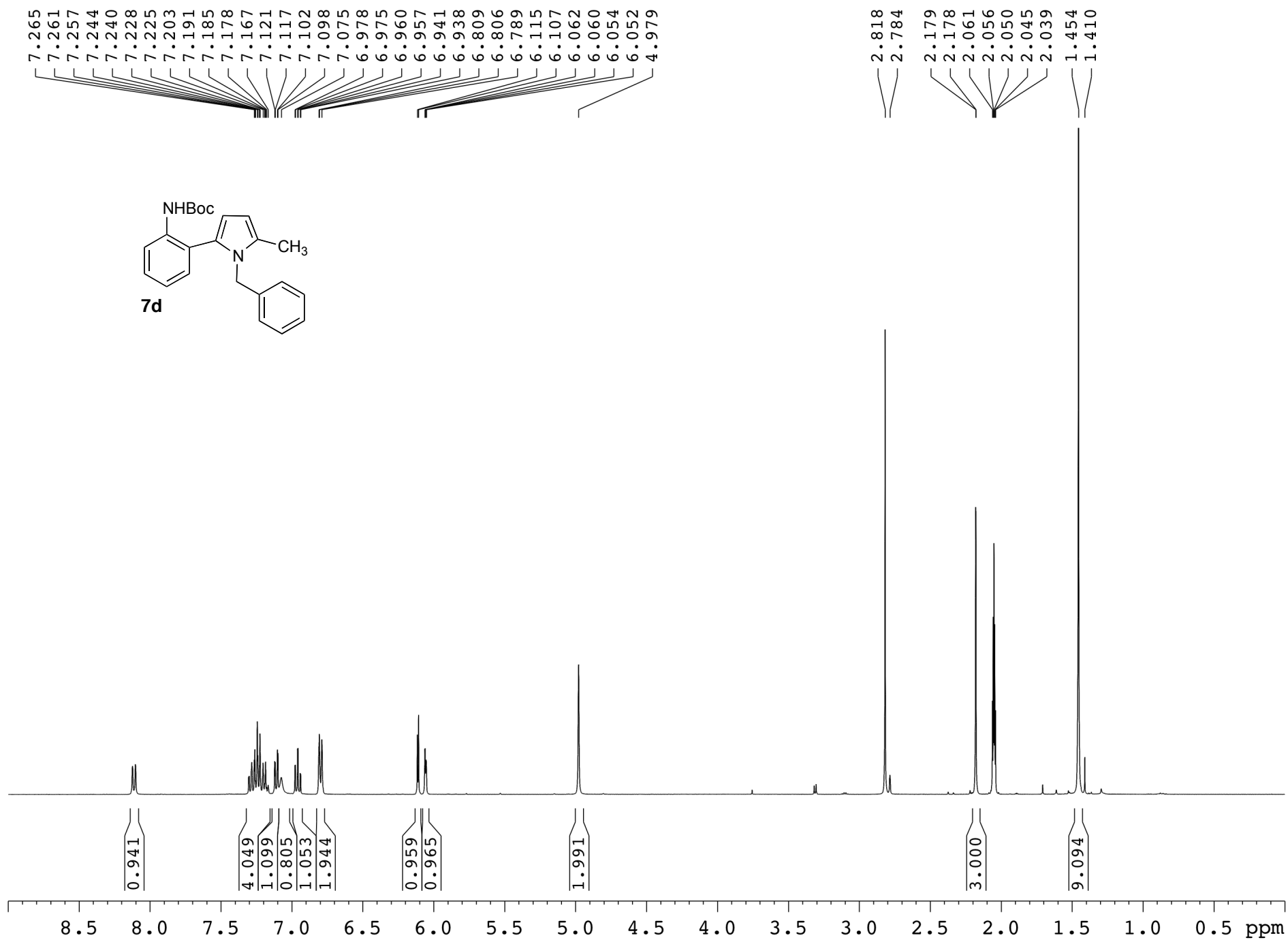
28.500

12.621



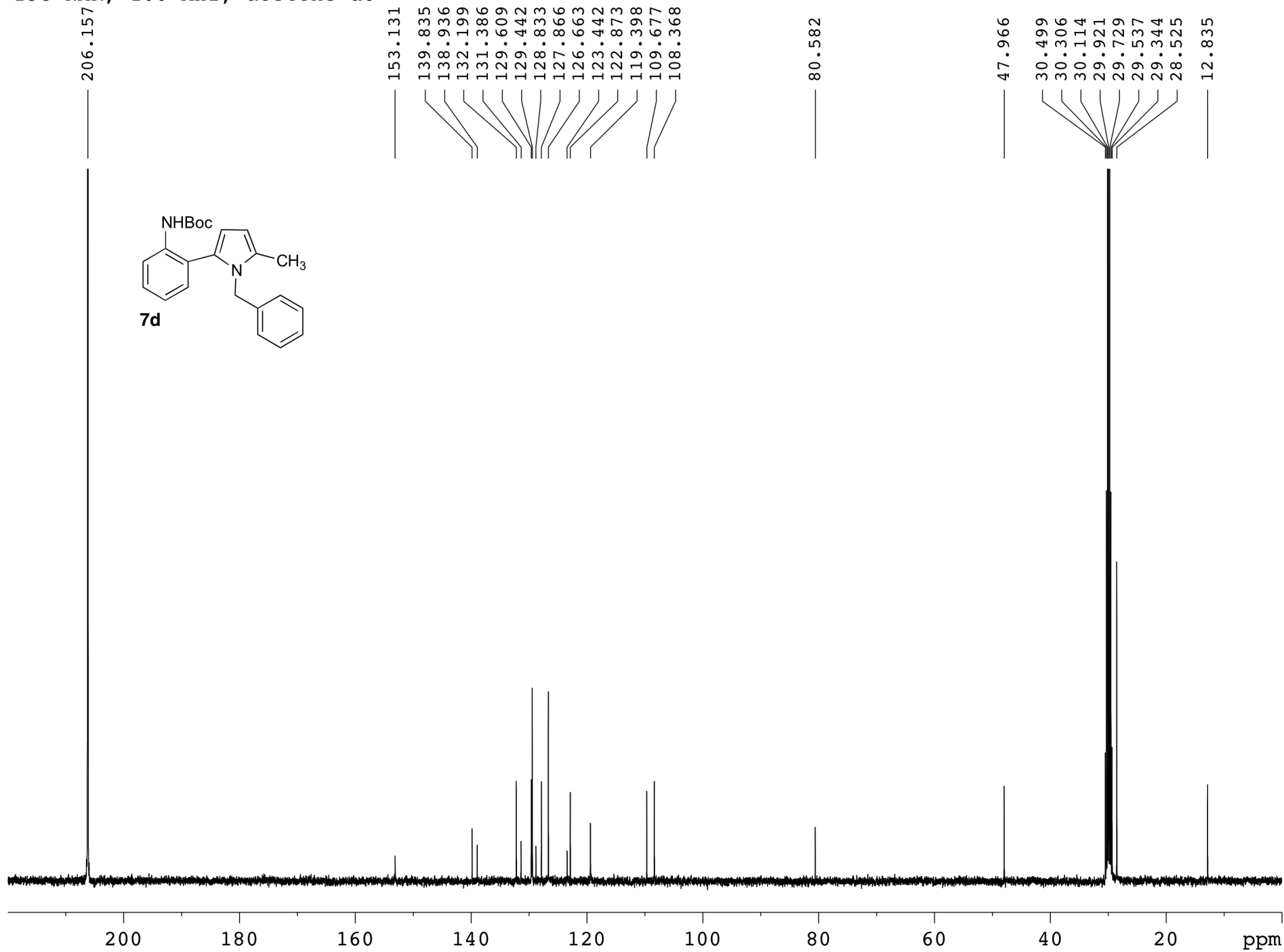
Proton NMR spectrum for **7d**

¹H NMR, 400 MHz, acetone-d₆



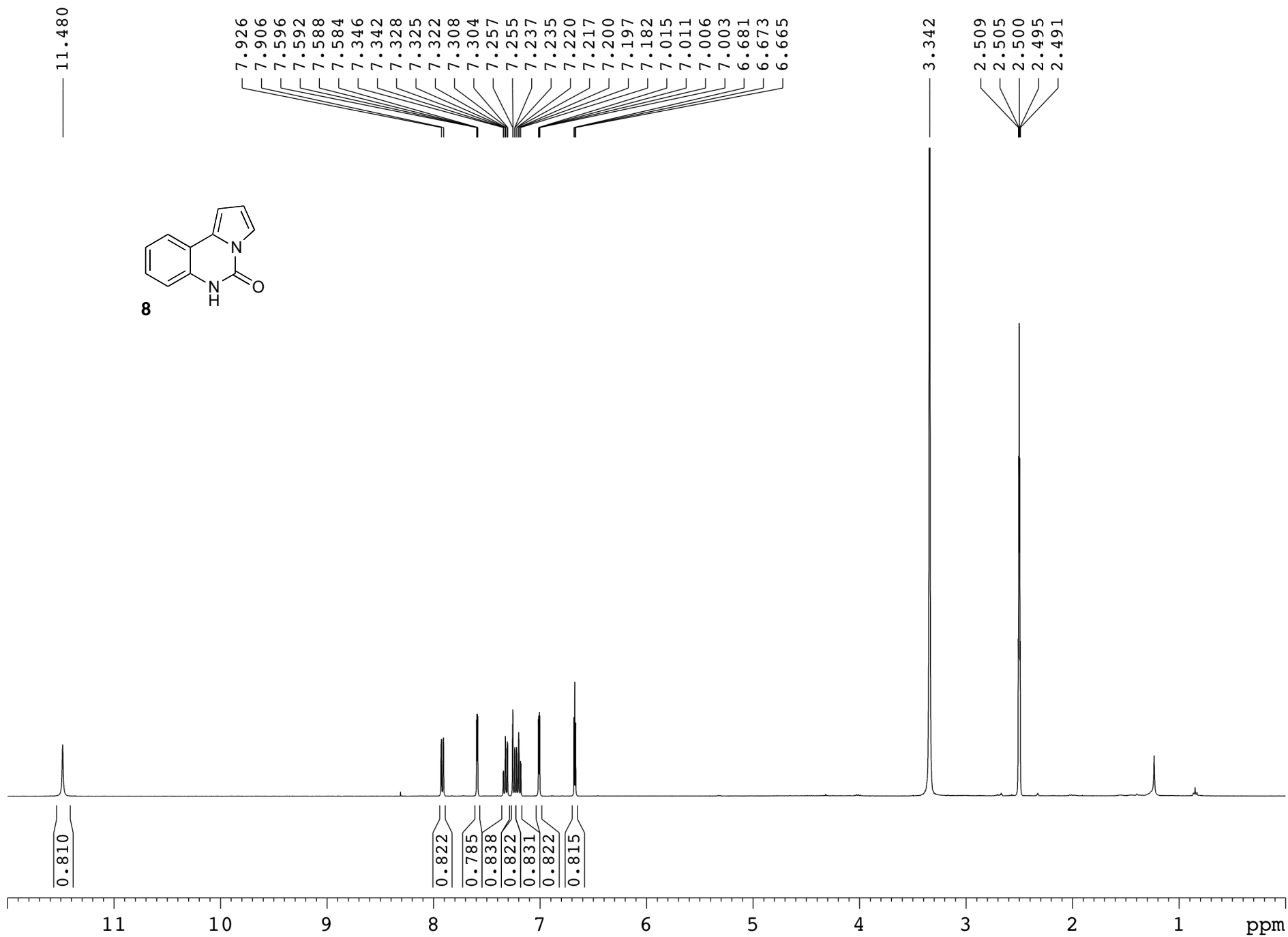
Carbon NMR spectrum for **7d**

¹³C NMR, 100 MHz, acetone-d₆



Proton NMR spectrum for **8**

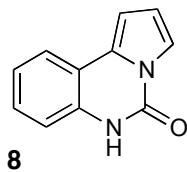
¹H NMR, 400 MHz, dmsO-d6



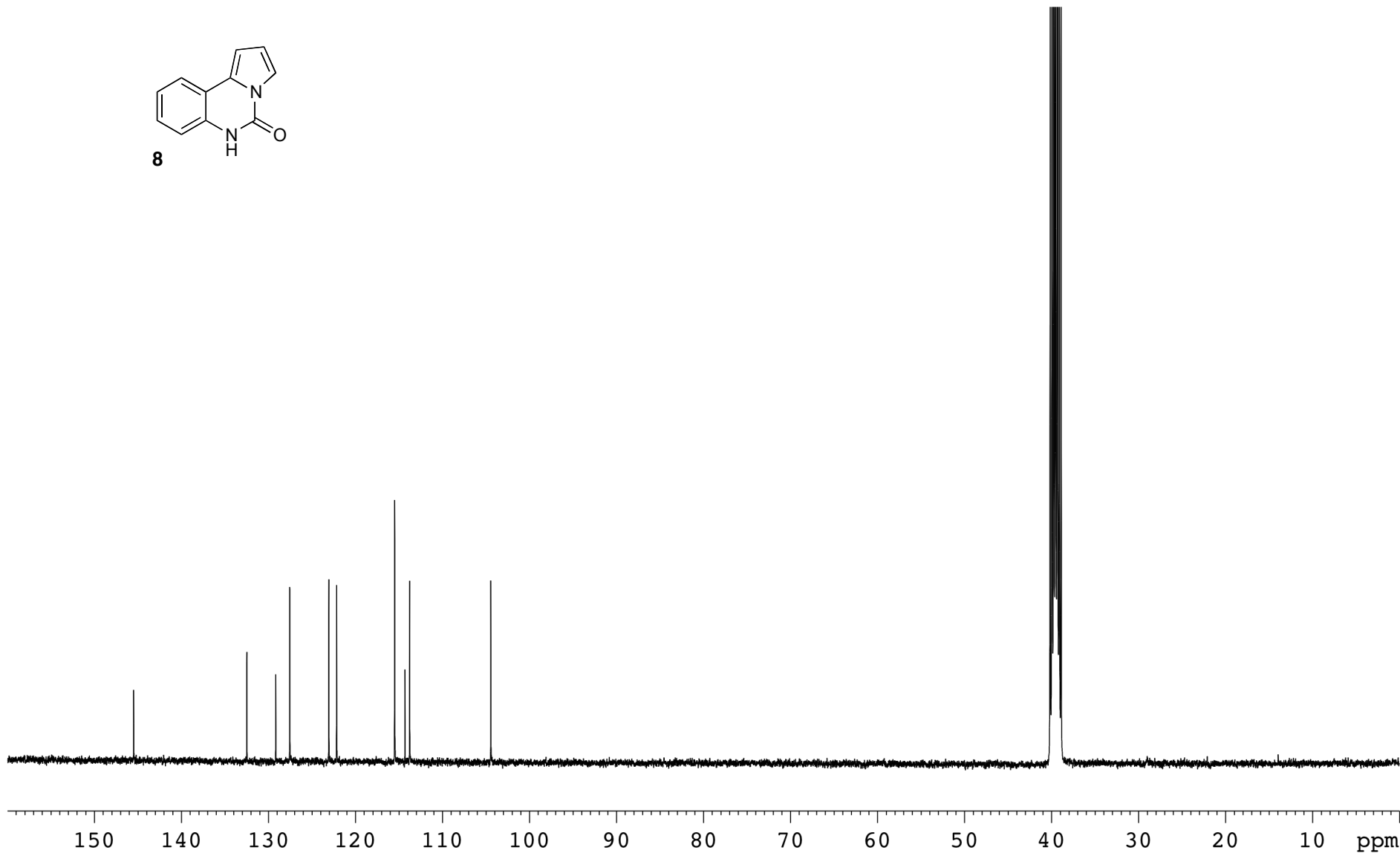
Carbon NMR spectrum for **8**

^{13}C NMR, 100 MHz, dmsO-d_6

145.489
132.479
129.157
127.550
123.059
122.162
115.491
114.305
113.769
104.441

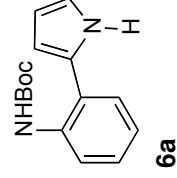
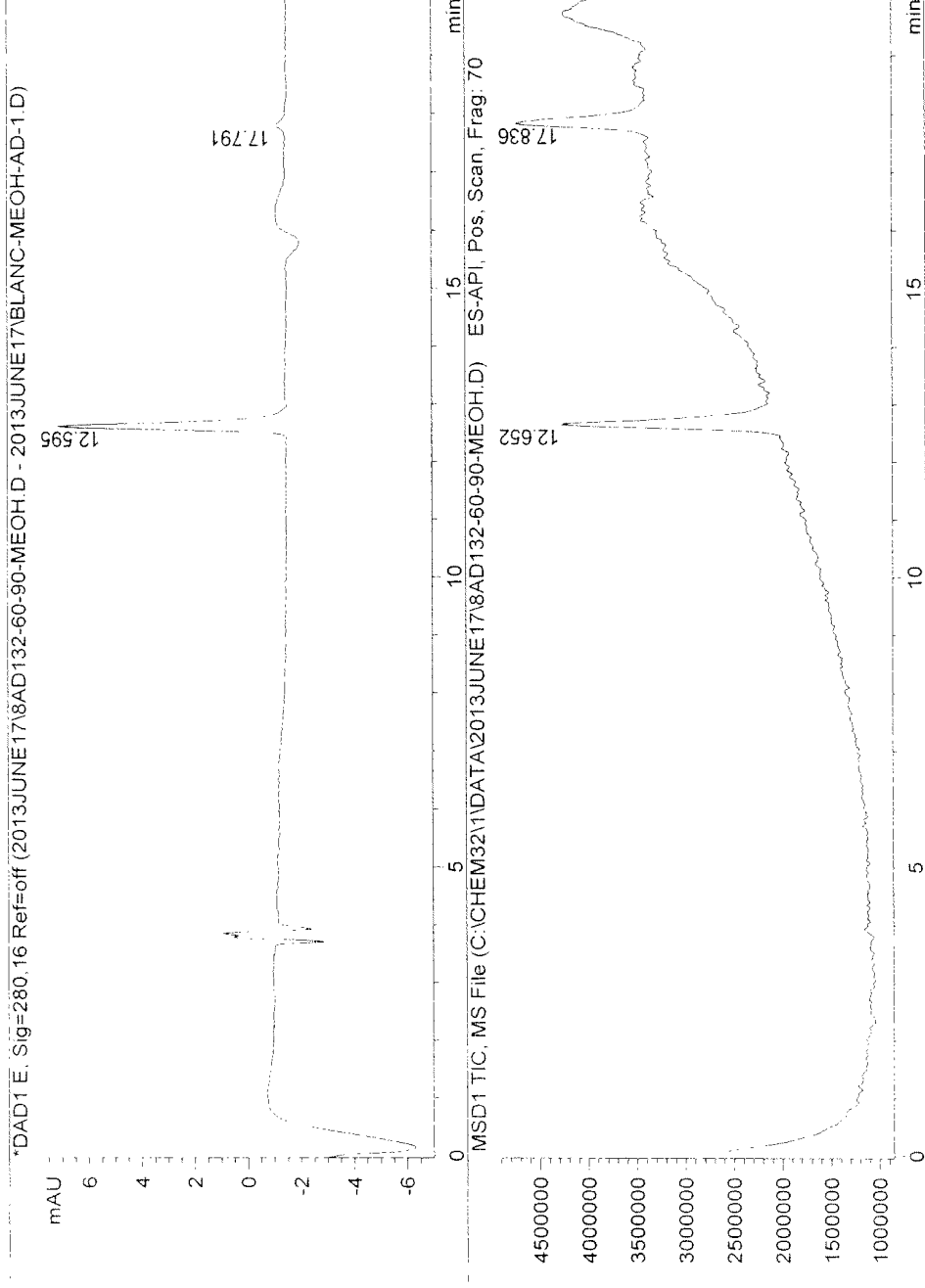


40.148
39.939
39.730
39.522
39.313
39.104
38.896



Reverse-phase HPLC profile of 6a at 280 nM

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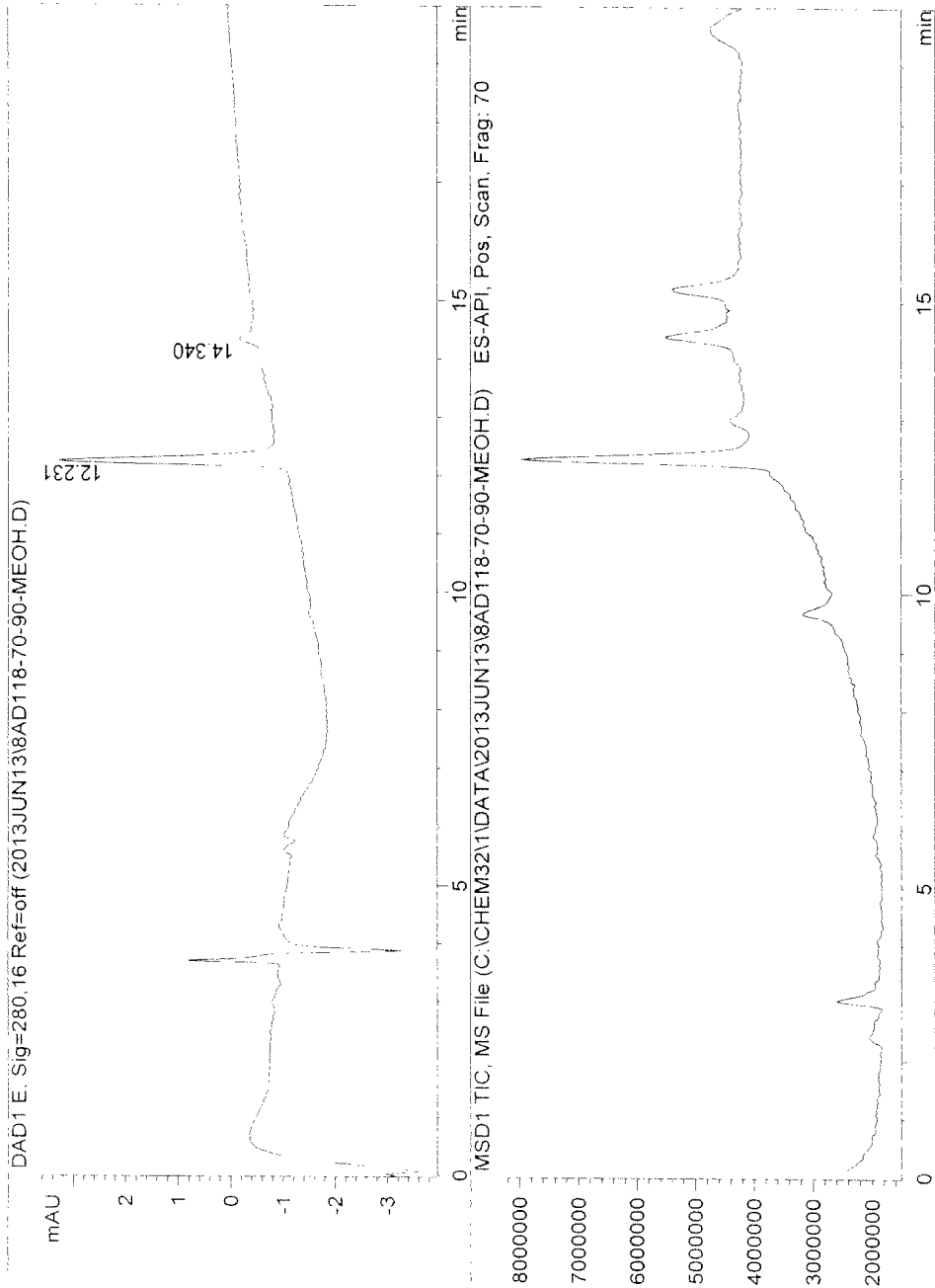
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MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
12.652	2.241e6	2.635e7	62.321
17.836	1.327e6	1.593e7	37.679

Reverse-phase HPLC profile of **6b** at 280 nm

Data File name: C:\CHEM32\1\DATA\2013JUN13\8AD118-70-90-MEOH.D
 Date: 13 June 2013
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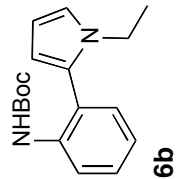


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14.340	0.421	5.733	12.389

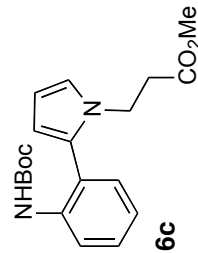
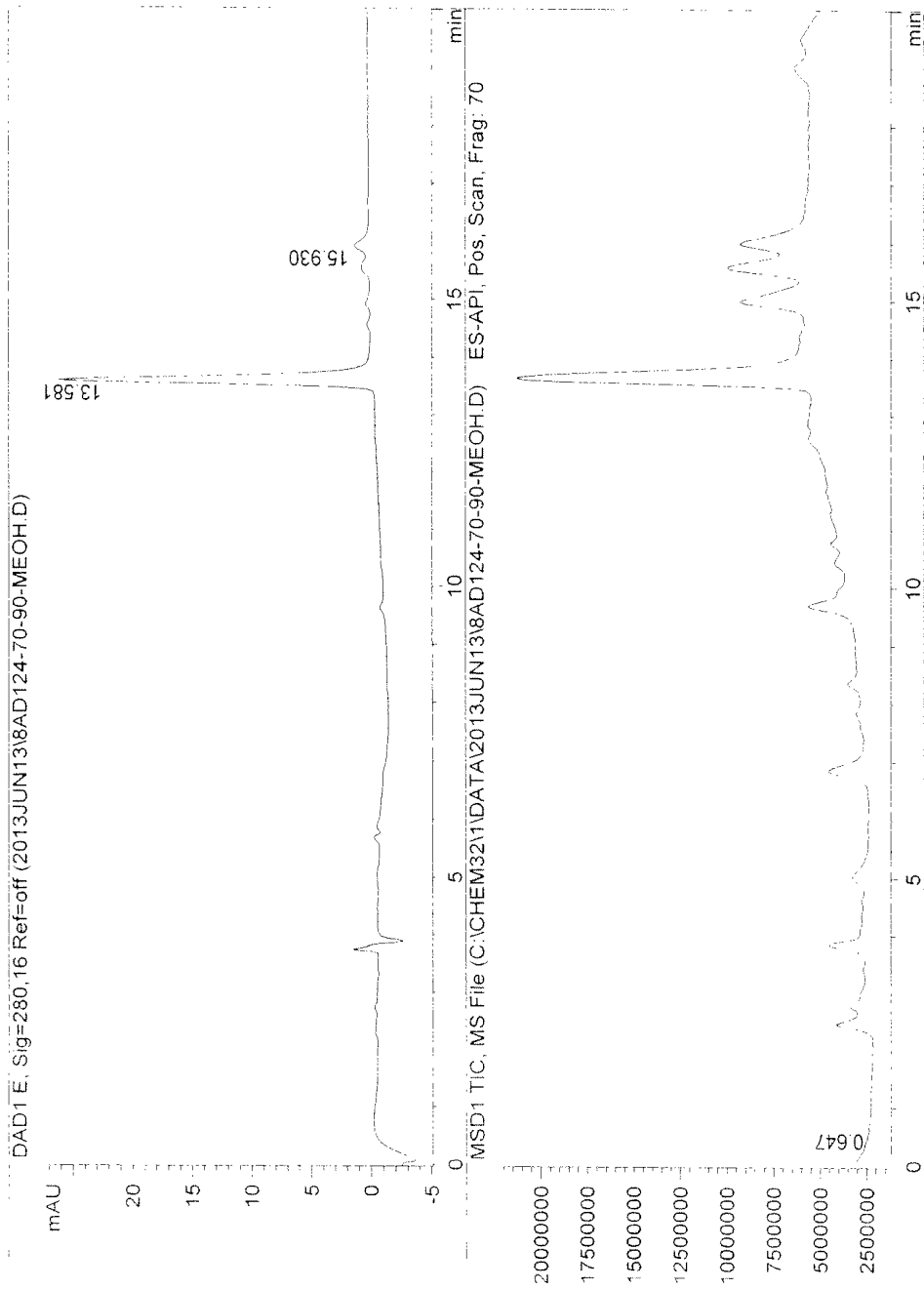
MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
4.324	0.421	5.733	12.389
14.340	4.324	40.540	87.611



Reverse-phase HPLC profile of 6c at 280 nM

Data File name: C:\CHEM32\1\DATA\2013JUN13\8AD124-70-90-MEOH.D
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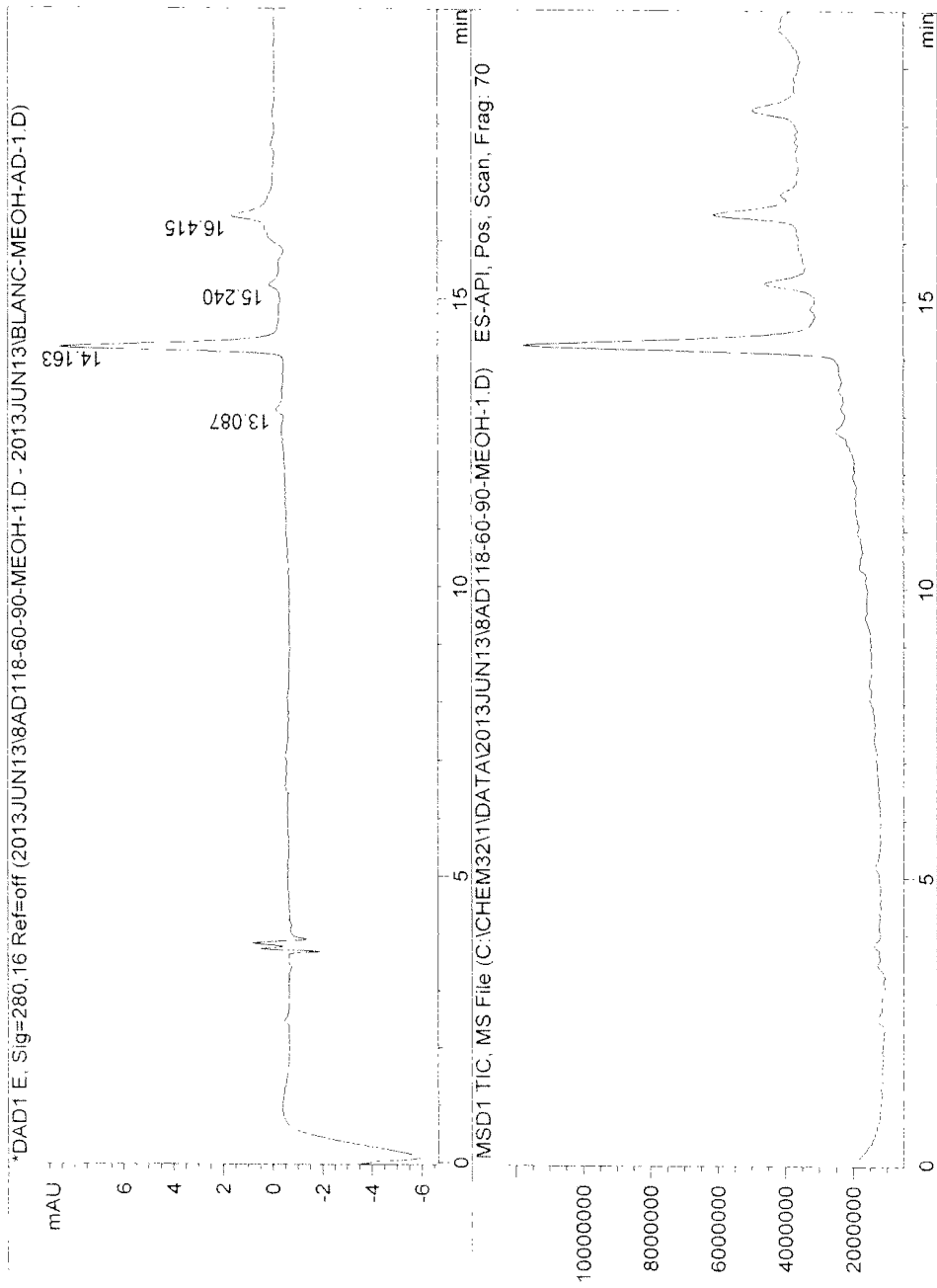
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15.930	1.240	29.662	9.972

MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
0.647	22095.771	84260.484	100.000

Reverse-phase HPLC profile of 6d at 280 nm

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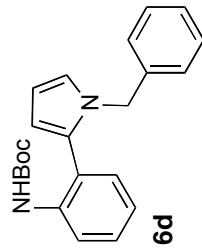


DAD1 E, Sig=280,16 Ref=off

Ret. Time	Height	Area	Area %
13.087	0.407	5.096	4.822
14.163	8.994	82.200	77.771
15.240	0.489	5.392	5.102
16.415	1.361	13.006	12.305

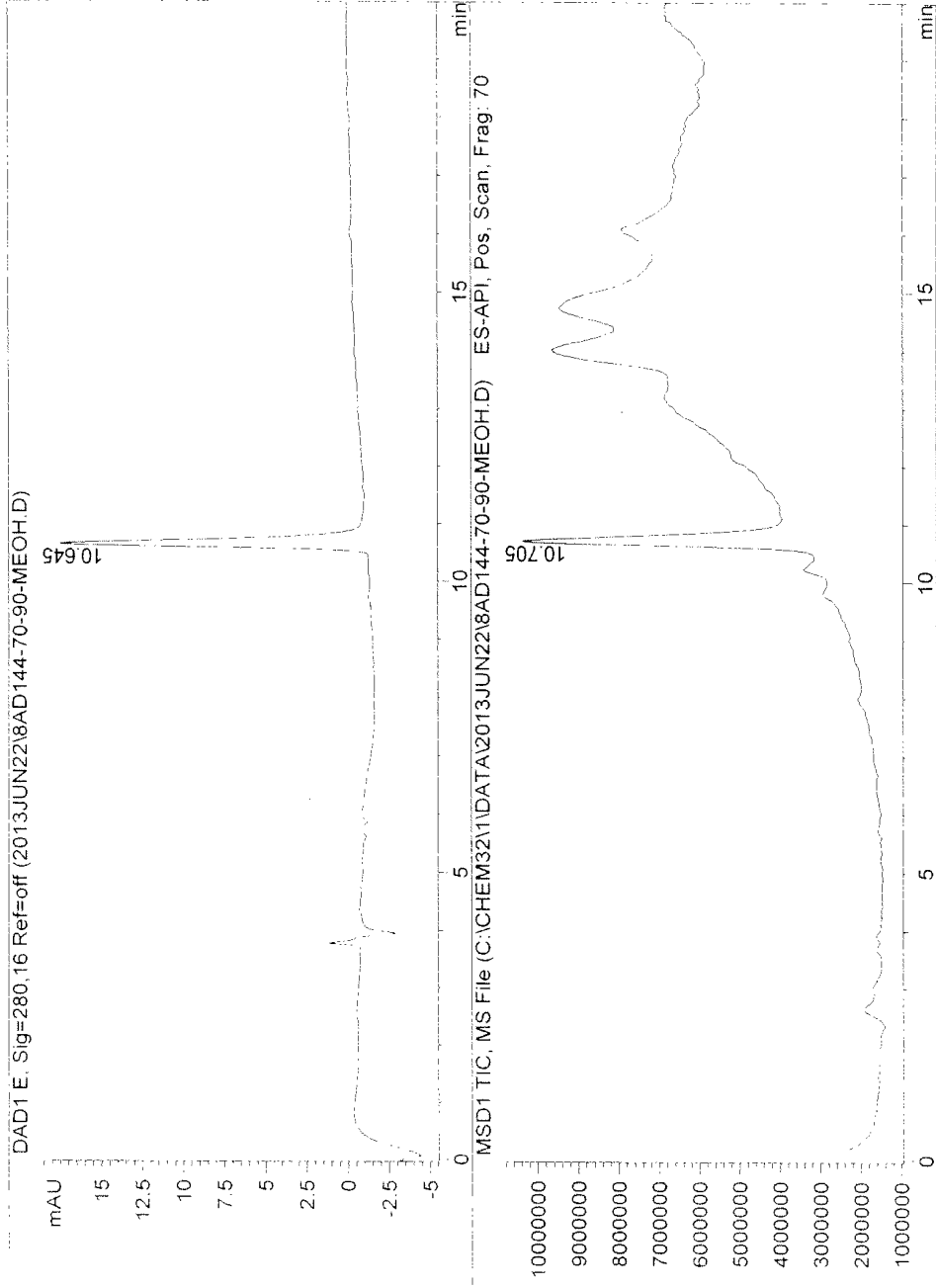
MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
13.087	0.407	5.096	4.822
14.163	8.994	82.200	77.771
15.240	0.489	5.392	5.102
16.415	1.361	13.006	12.305



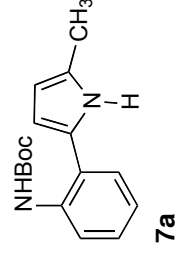
Reverse-phase HPLC profile of **7a** at 280 nm

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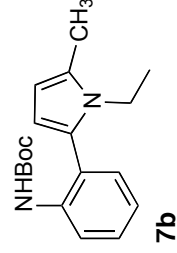
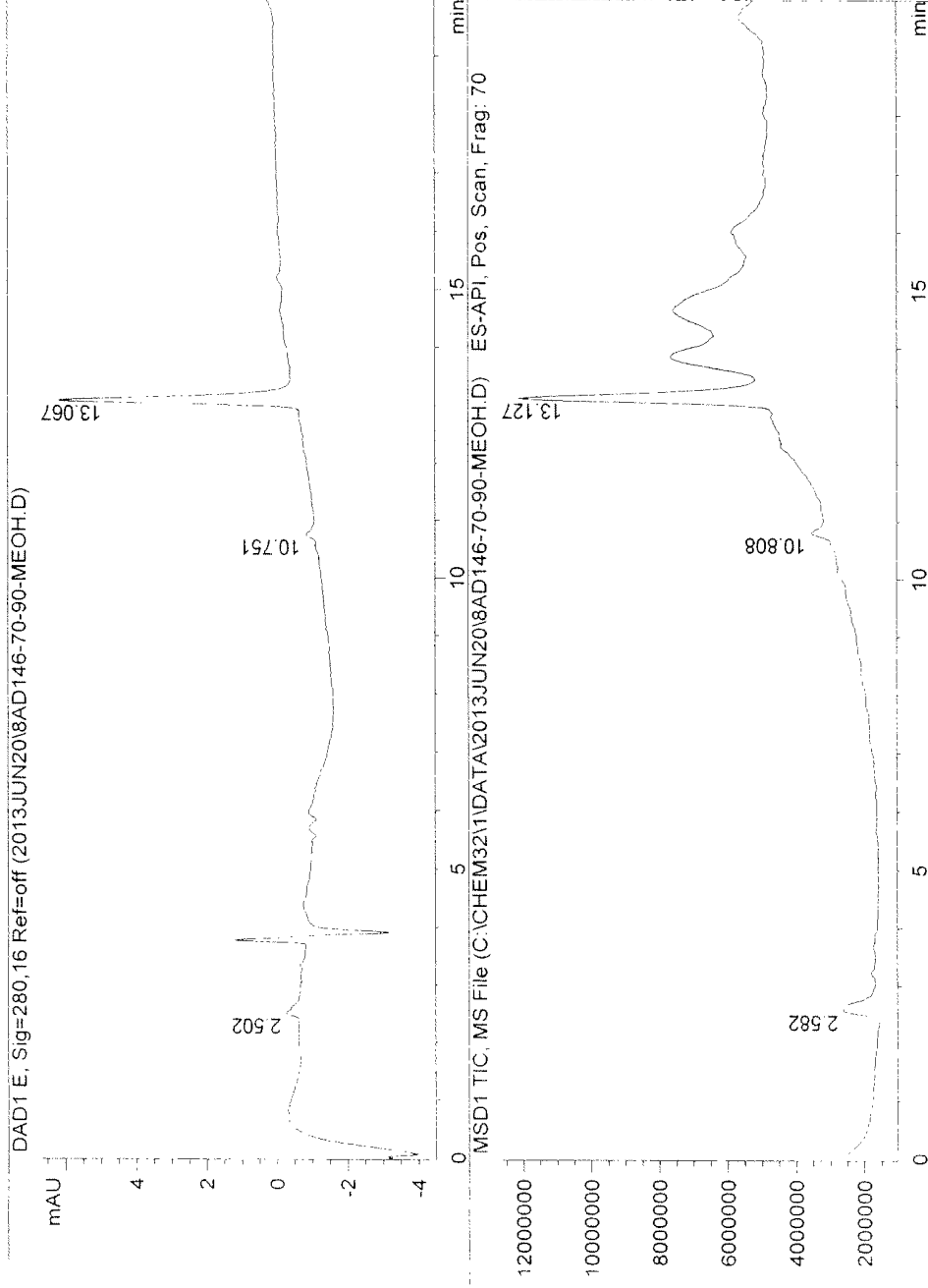


MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
10.705	7.043e6	8.783e7	100.000

Reverse-phase HPLC profile of **7b** at 280 nm

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 Date: 20 June 2013
 Acq. method: C:\CHEM32\1\METHODS\LC_70_90_7MIN_20MN_



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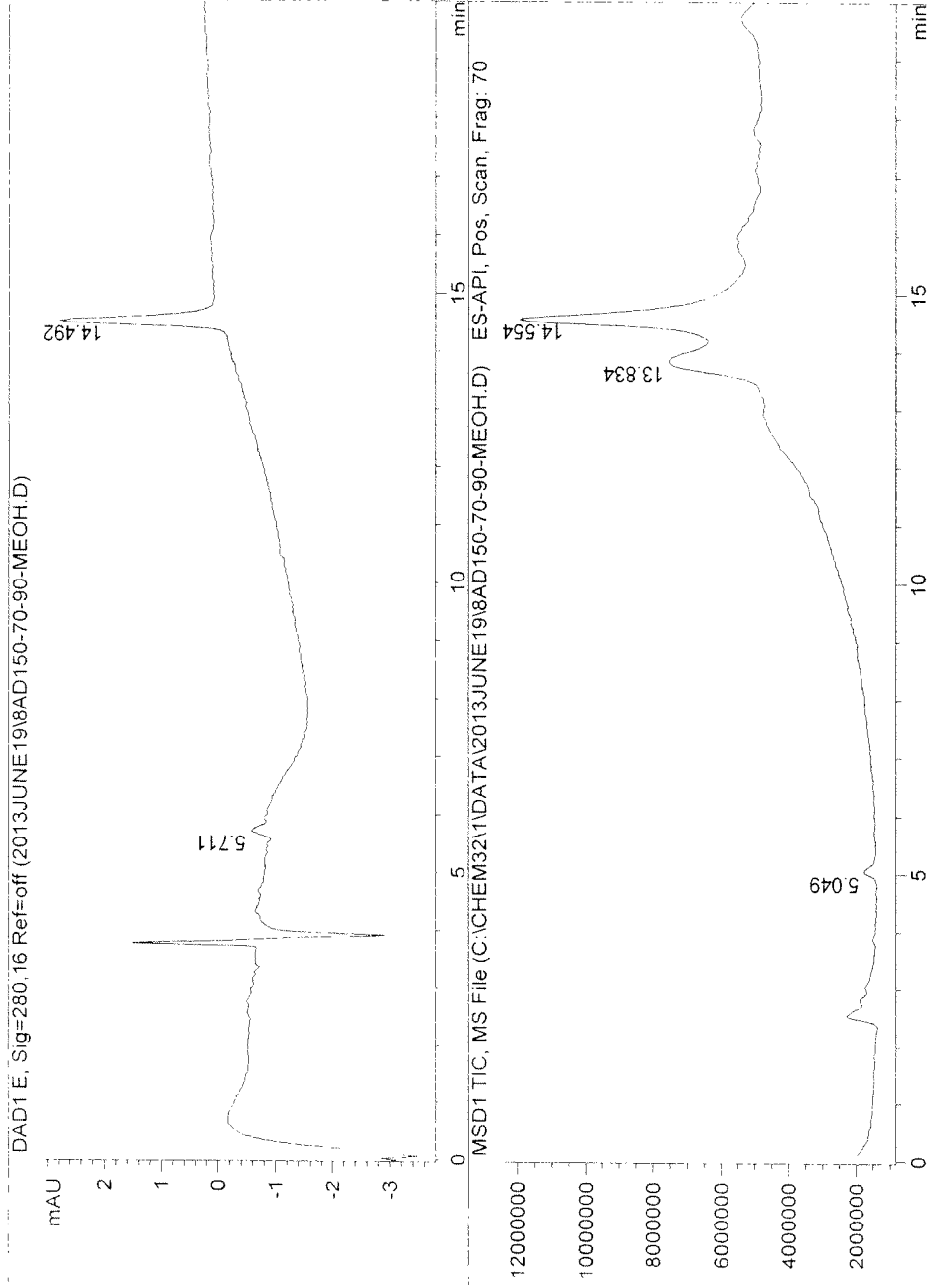
Ret. Time	Height	Area	Area %
2.502	0.378	3.472	4.985
10.751	0.286	2.777	3.987
13.067	6.809	63.403	91.028

MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
2.582	1.056e6	1.665e7	16.123
10.808	500422.281	4.675e6	4.526
13.127	7.321e6	8.196e7	79.351

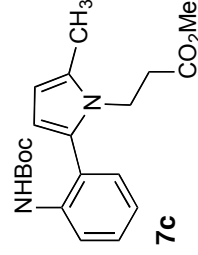
Reverse-phase HPLC profile of 7c at 280 nm

Data File name: C:\CHEM32\1\DATA\2013JUNE19\8AD150-70-90-MEOH.D
 Date: 19 June 2013
 Acq method: C:\CHEM32\1\METHODS\LC_70_90_7MIN_20MN



DAD1 E, Sig=280.16 Ref=off

Ret. Time	Height	Area	Area %
5.711	0.454	5.147	13.702
14.492	2.893	32.419	86.298

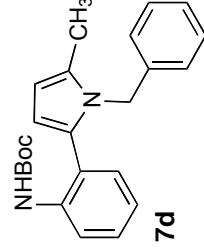
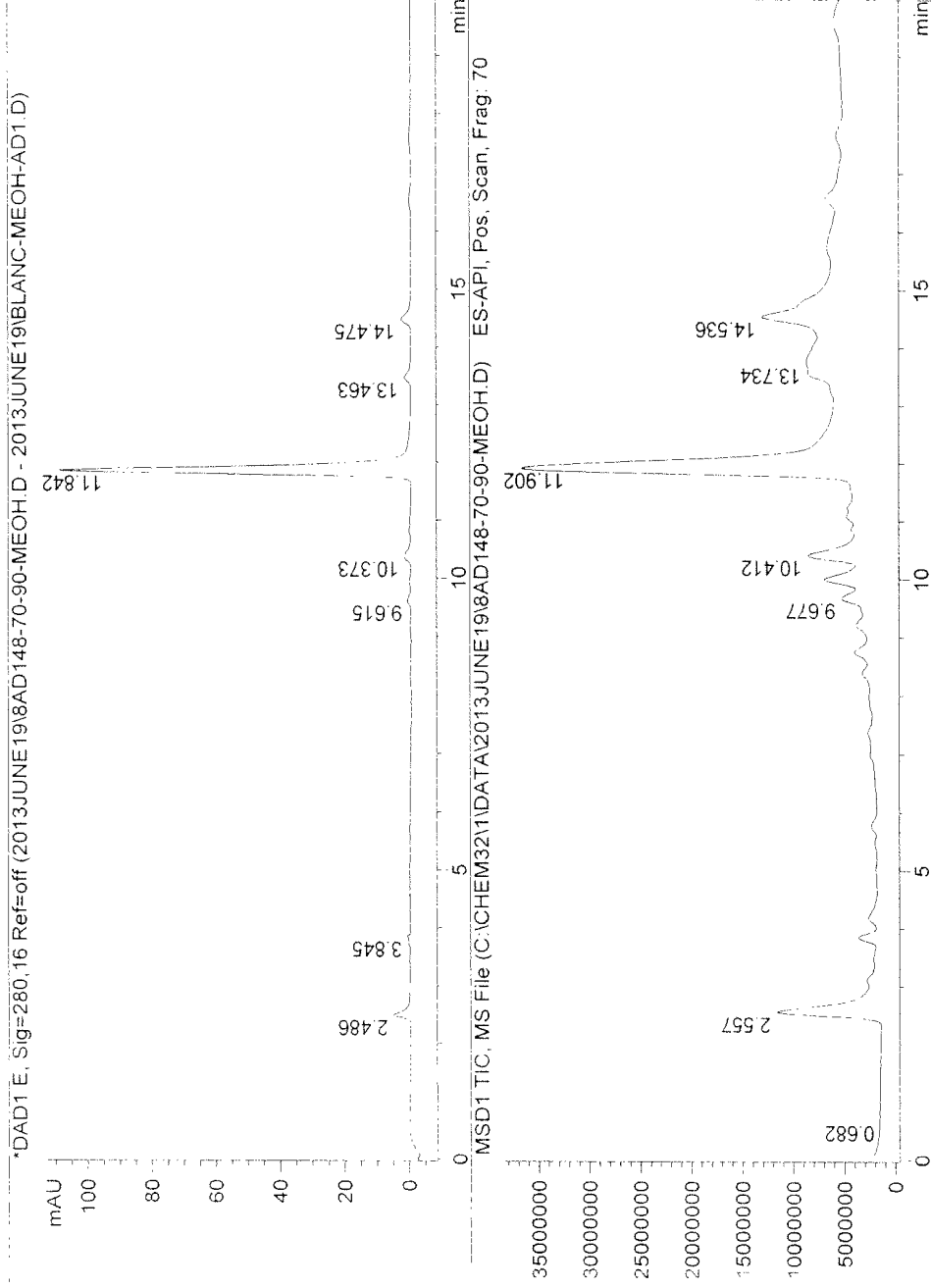


MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
5.049	416412.281	4.931e6	1.853
13.834	2.730e6	8.559e7	32.169
14.554	7.135e6	1.755e8	65.978

Reverse-phase HPLC profile of **7d** at 280 nm

Data File name: C:\CHEM32\1\DATA\2013JUNE19\8AD148-70-90-MEOH.D
 Date: 19 June 2013
 Acq method: C:\CHEM32\1\METHODS\IC_70_90_7MIN_20MN_



DAD1 E, Sig=280,16 Ref=off

Ret. Time	Height	Area	Area %
2.486	5.254	40.725	3.416
3.845	1.056	5.952	0.499
9.615	1.027	9.613	0.806
10.373	1.980	23.674	1.986
11.842	110.250	1057.239	88.694
13.463	1.992	19.902	1.670
14.475	3.010	34.902	2.928

MSD1 TIC, MS File

Ret. Time	Height	Area	Area %
0.682	17151.857	79538.266	0.008
2.557	9.898e6	1.252e8	13.271
9.677	1.842e6	1.919e7	2.034
9.998	3.278e6	3.686e7	3.907
10.412	4.896e6	6.624e7	7.020
11.902	3.164e7	5.356e8	56.761
13.734	1.767e6	5.436e7	5.761
14.536	5.697e6	1.060e8	11.239