

Synthesis of 2,2-Difunctionalized 2*H*-Azirines by I₂-Mediated Annulation of Enamines

Qing Lu, Yanmin He, Junbiao Chang,* and Wenquan Yu*

College of Chemistry, Pingyuan Laboratory, State Key Laboratory of Antiviral Drugs, Zhengzhou University, Zhengzhou 450001, China

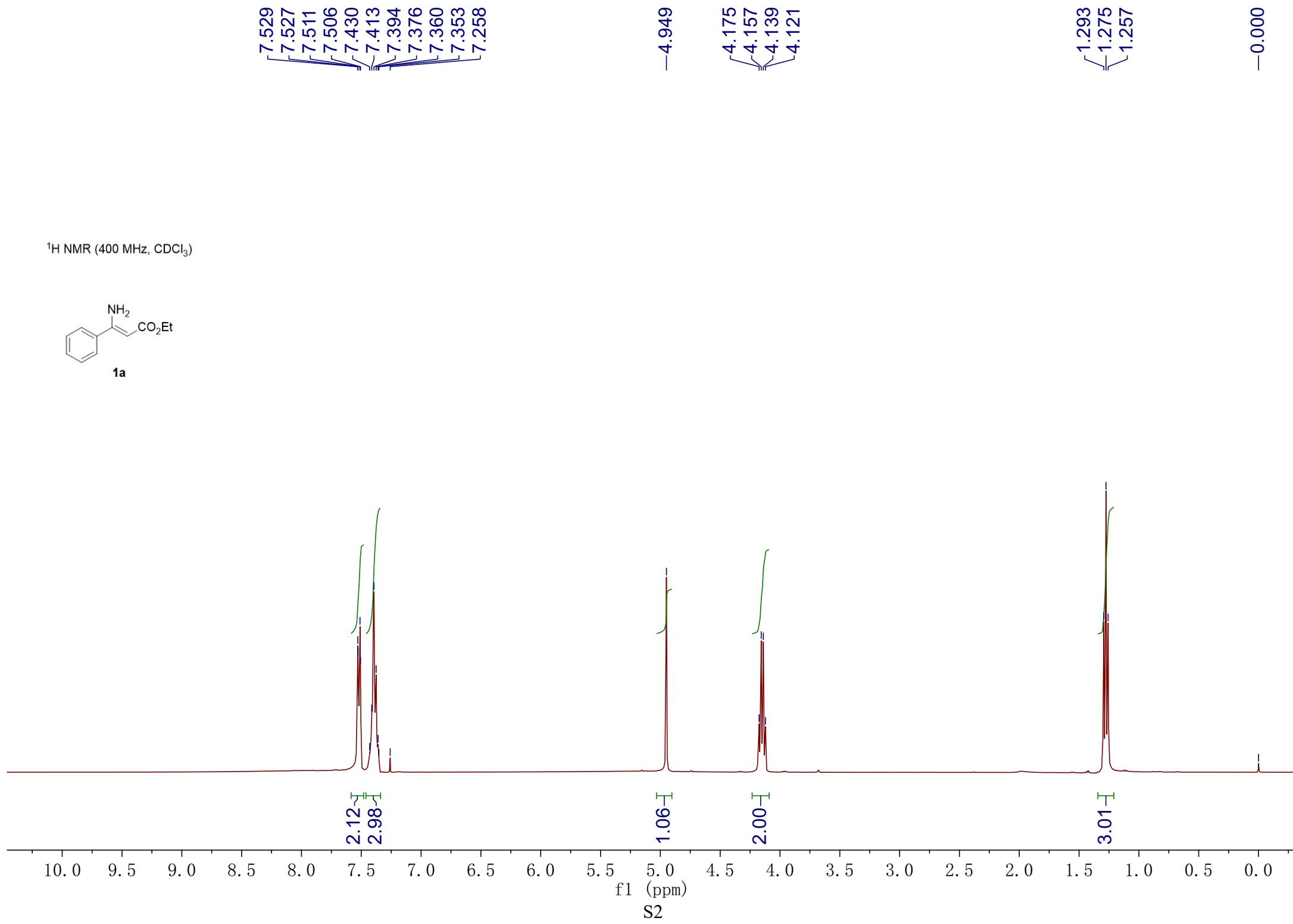
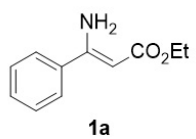
Email: wenquan_yu@zzu.edu.cn; changjunbiao@zzu.edu.cn

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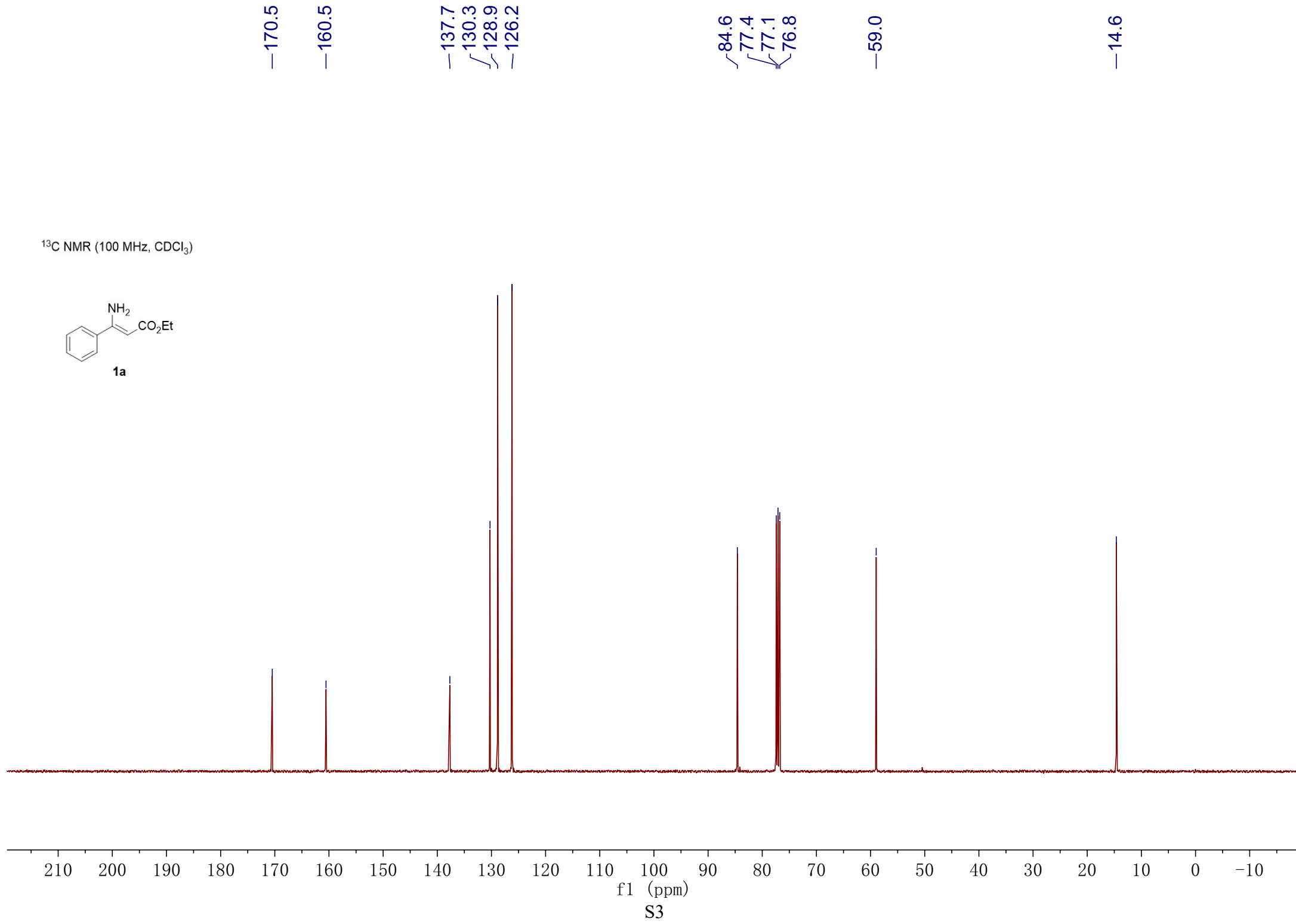
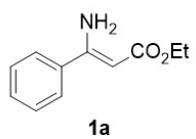
1. NMR Spectra of Isolated Compounds

S2

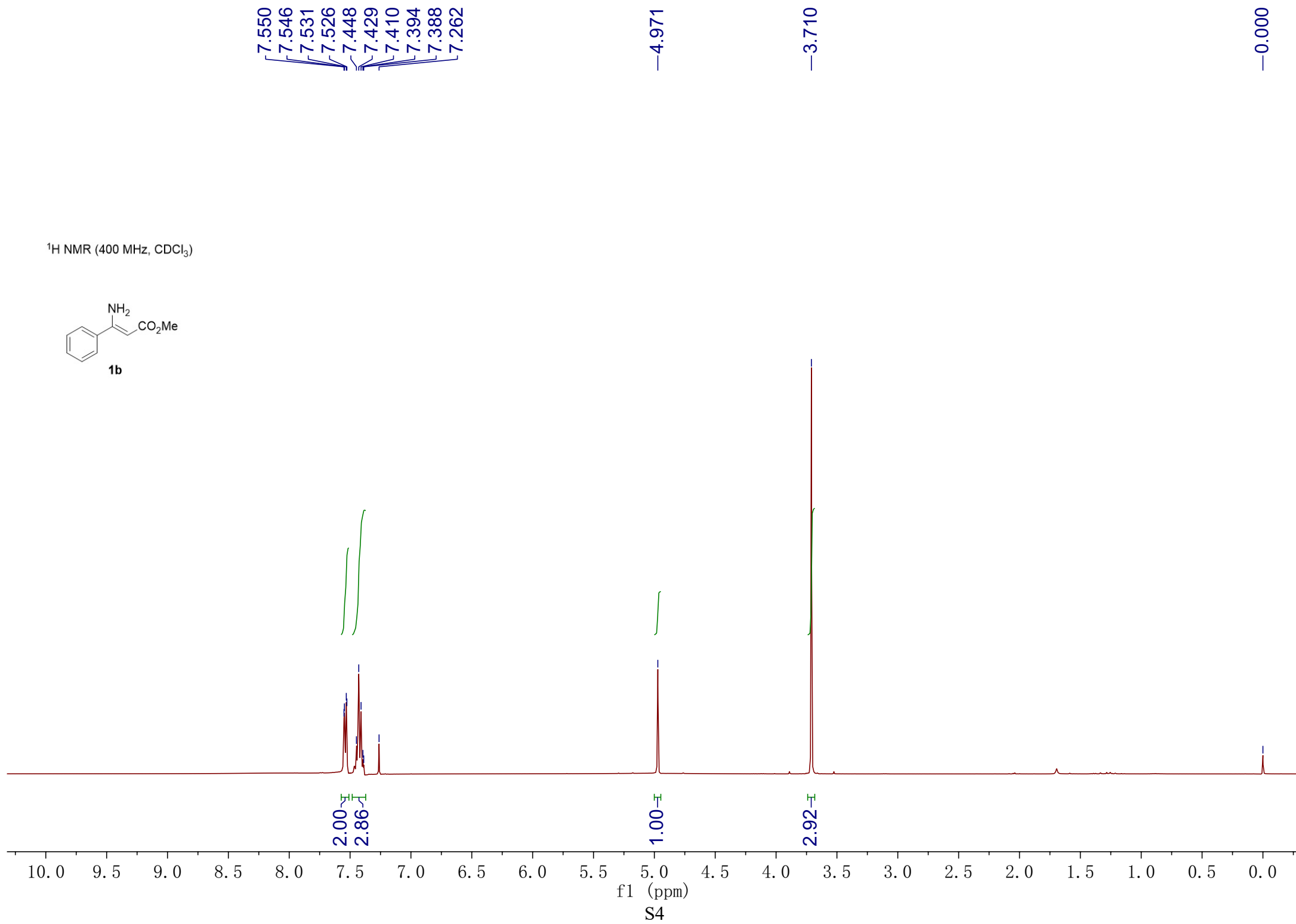
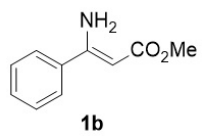
¹H NMR (400 MHz, CDCl₃)



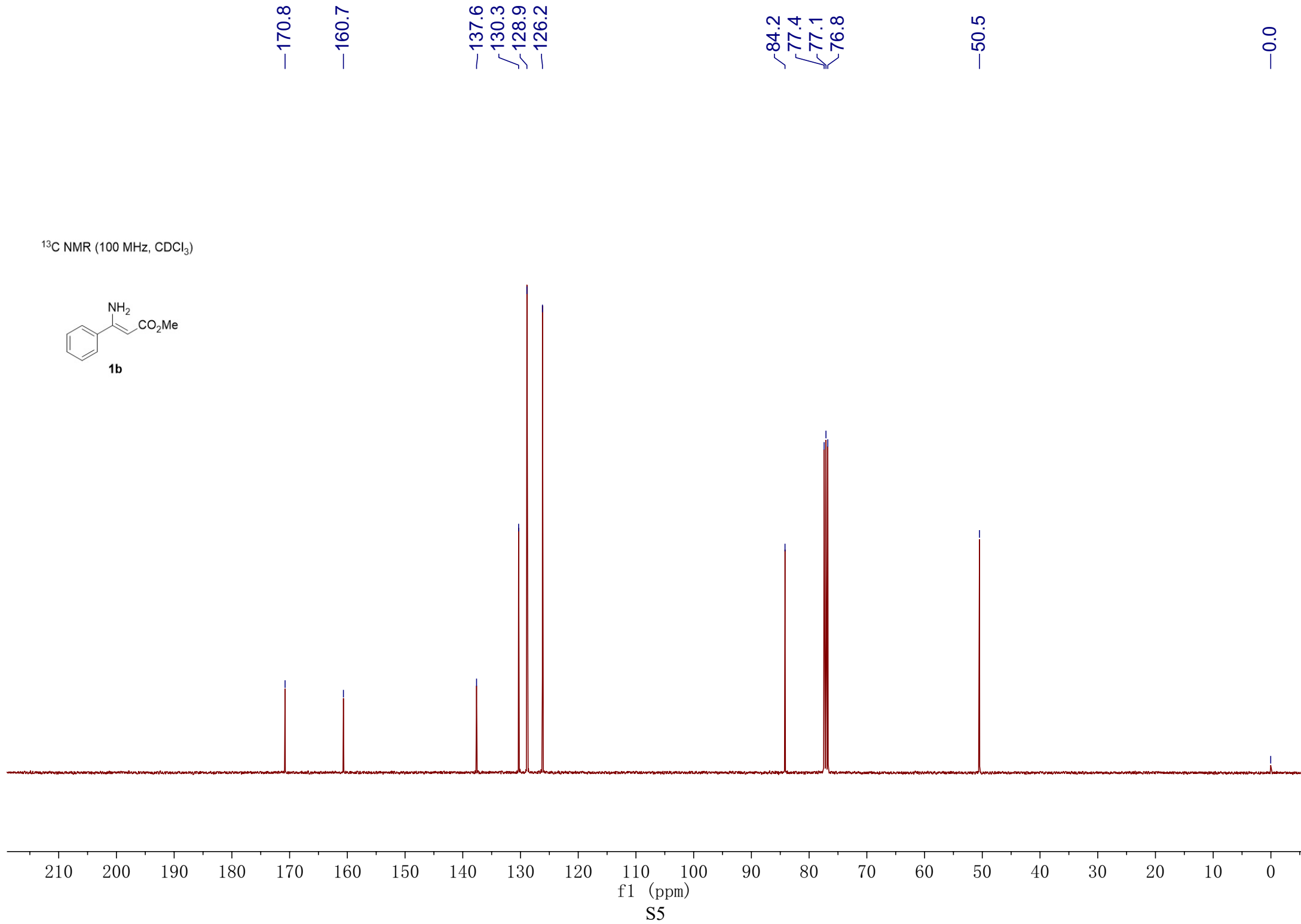
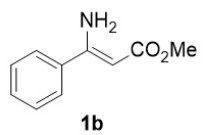
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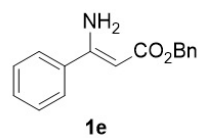


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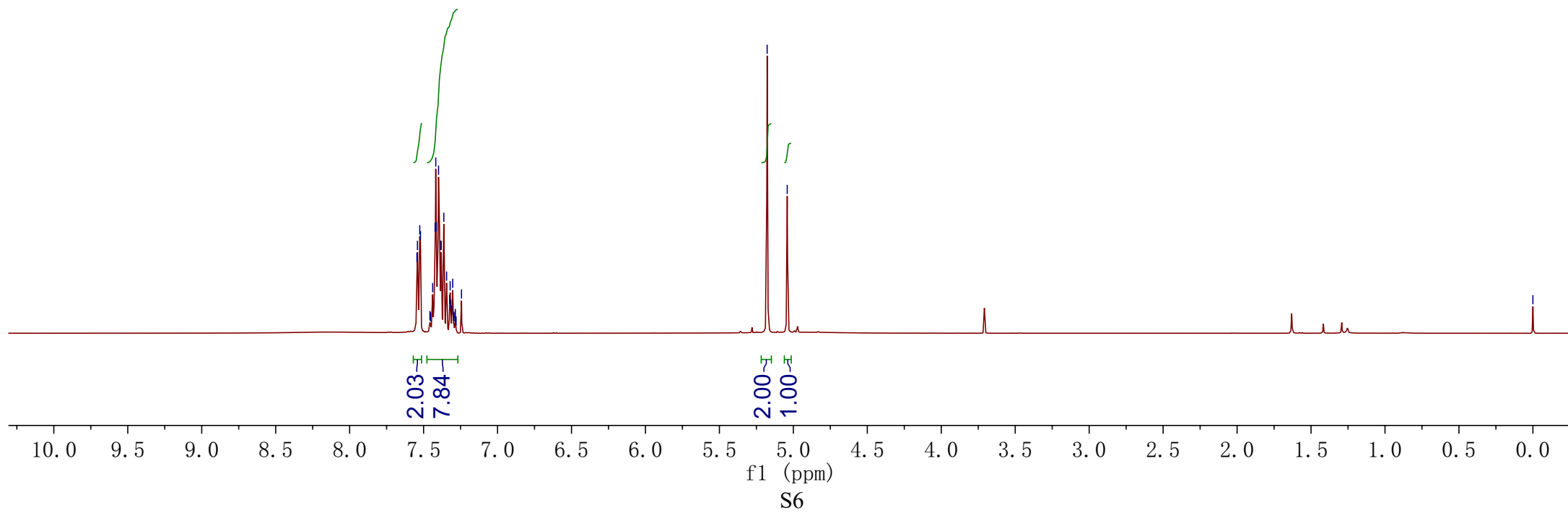


^{13}C NMR (100 MHz, CDCl_3)





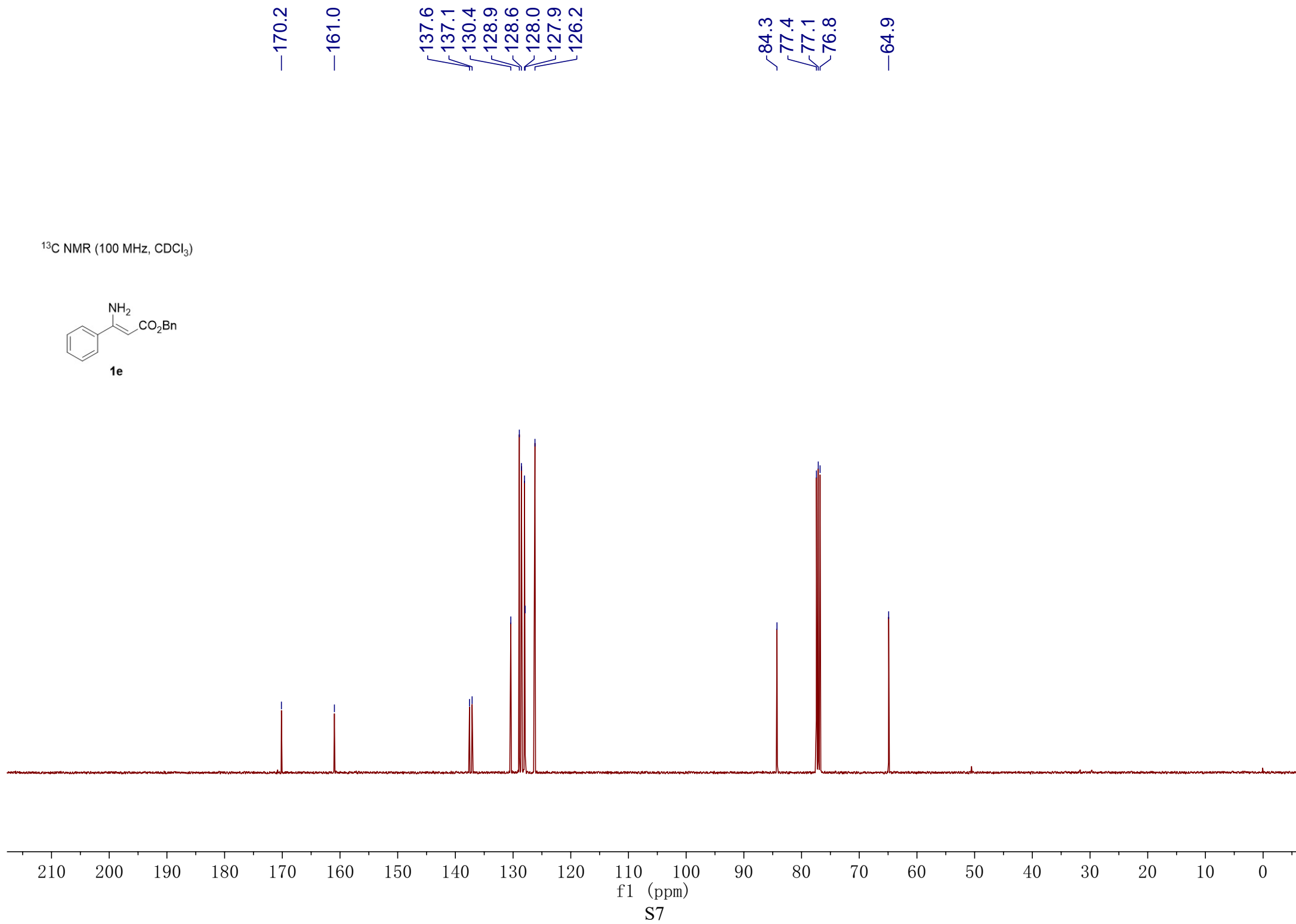
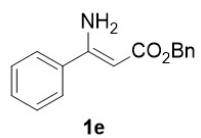
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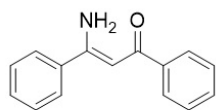


7.545
 7.541
 7.526
 7.521
 7.457
 7.453
 7.439
 7.422
 7.418
 7.414
 7.399
 7.383
 7.381
 7.363
 7.344
 7.324
 7.320
 7.316
 7.309
 7.303
 7.296
 7.289
 7.285
 7.280
 7.244
 ~5.177
 ~5.041

—0.000

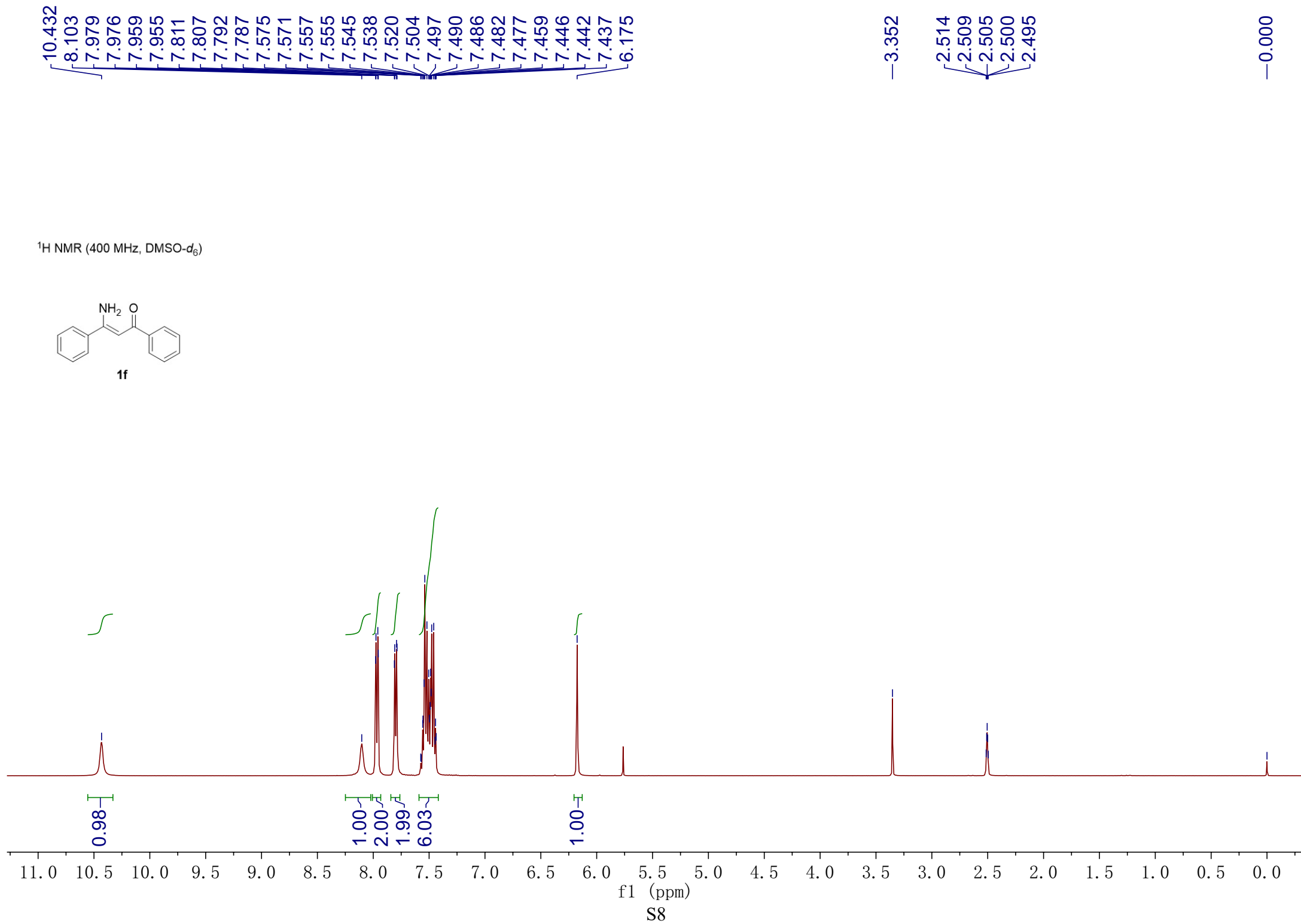
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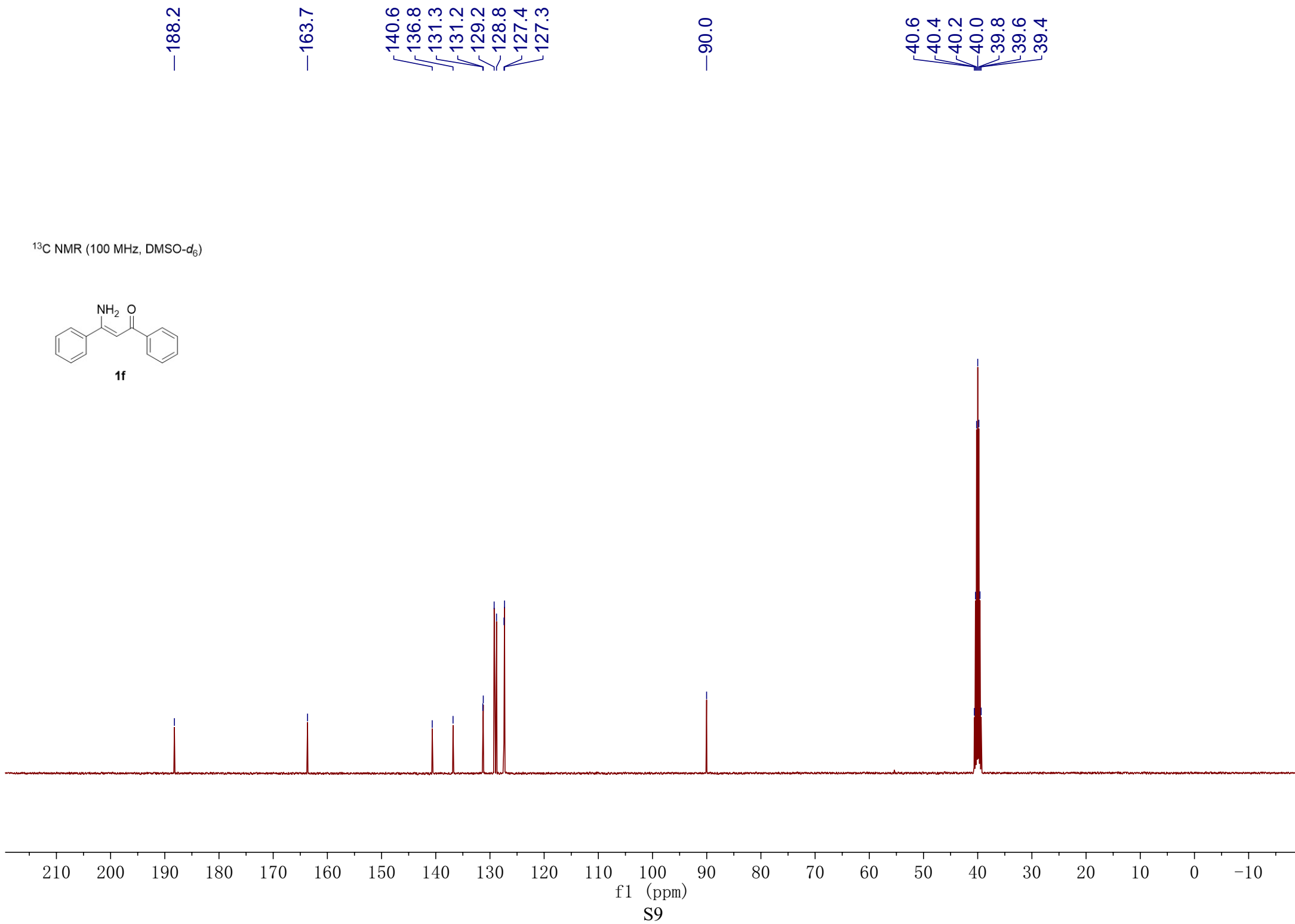
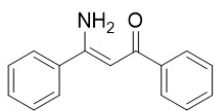


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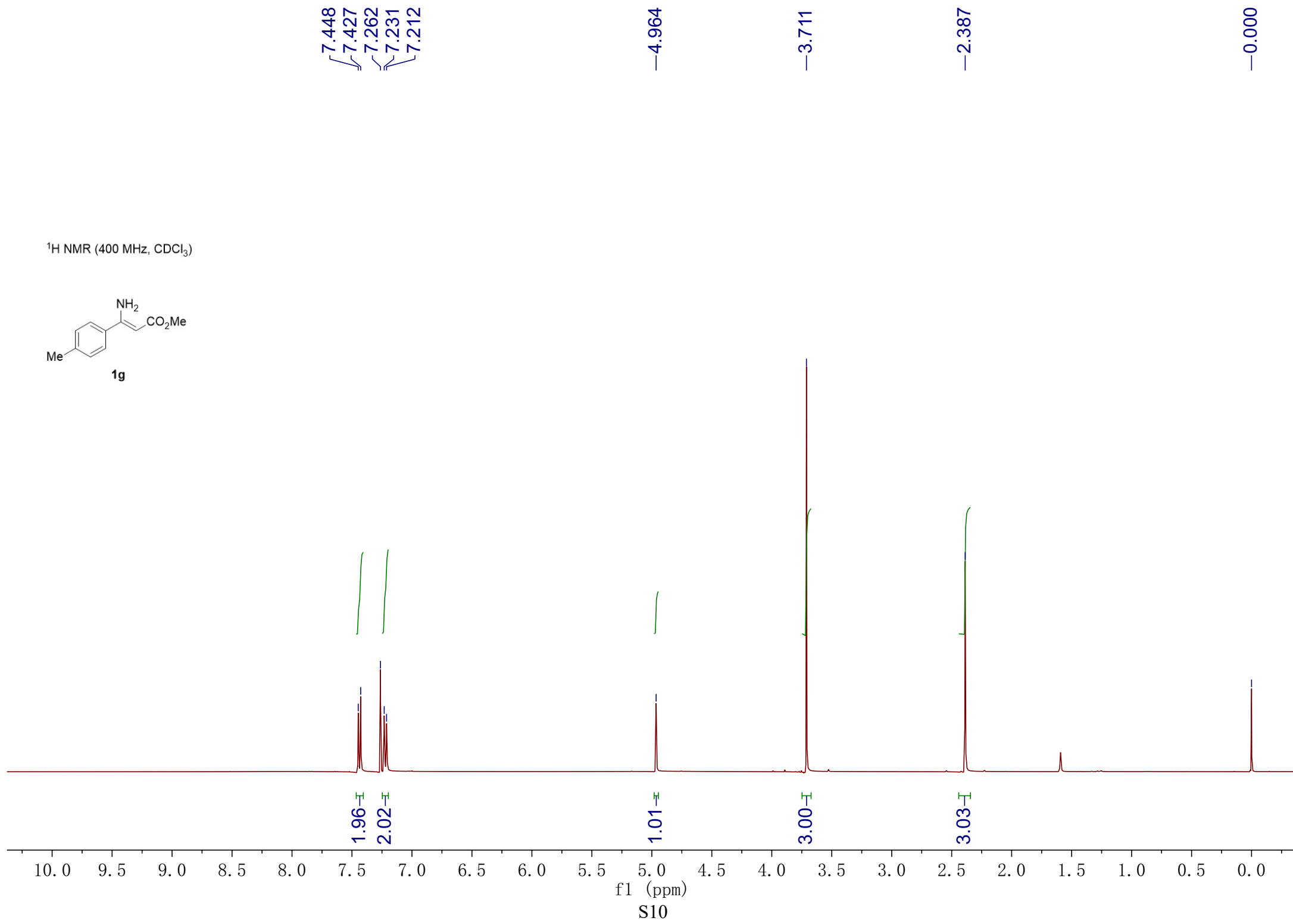
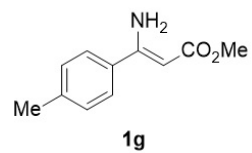
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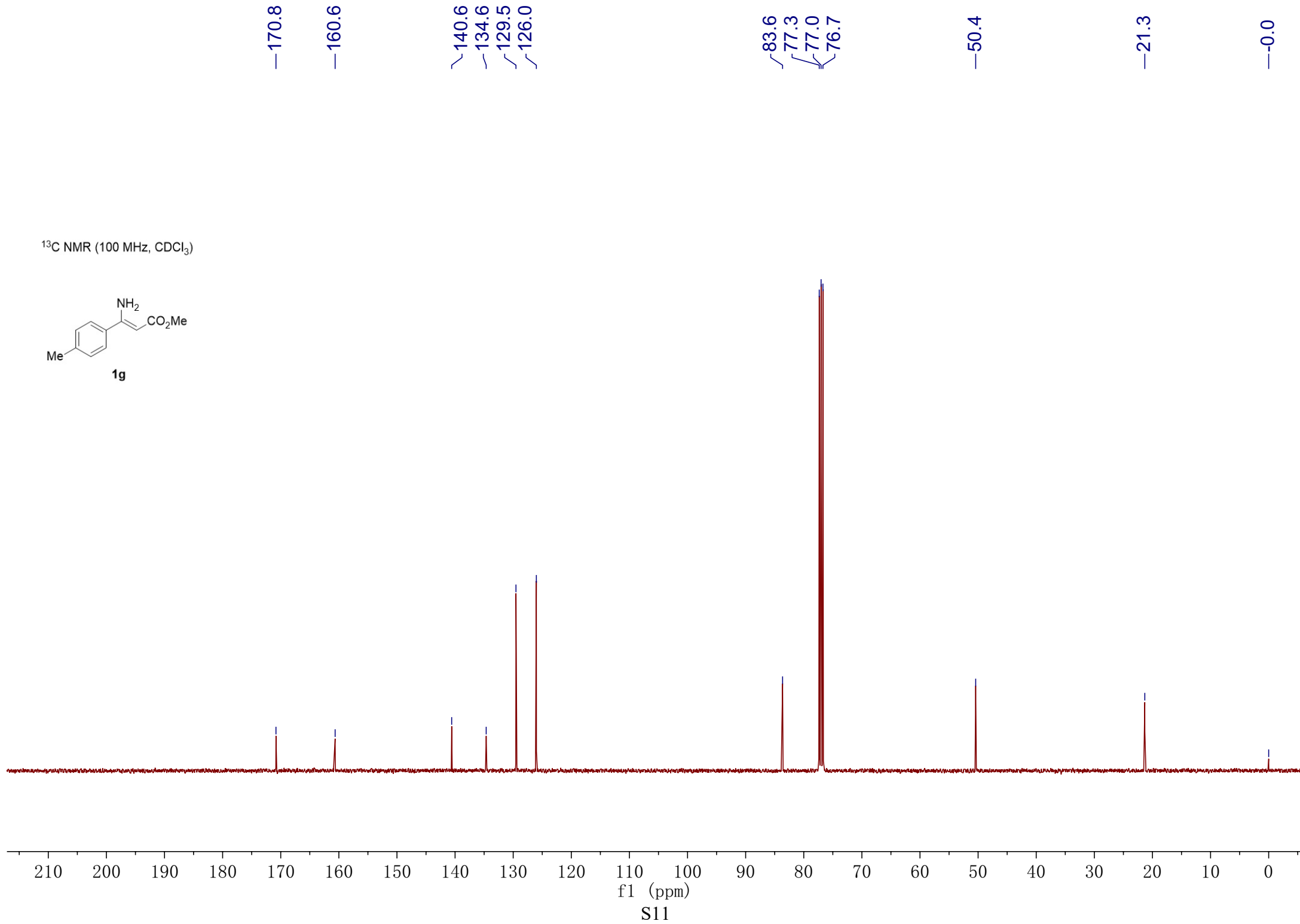
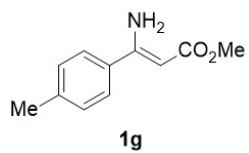
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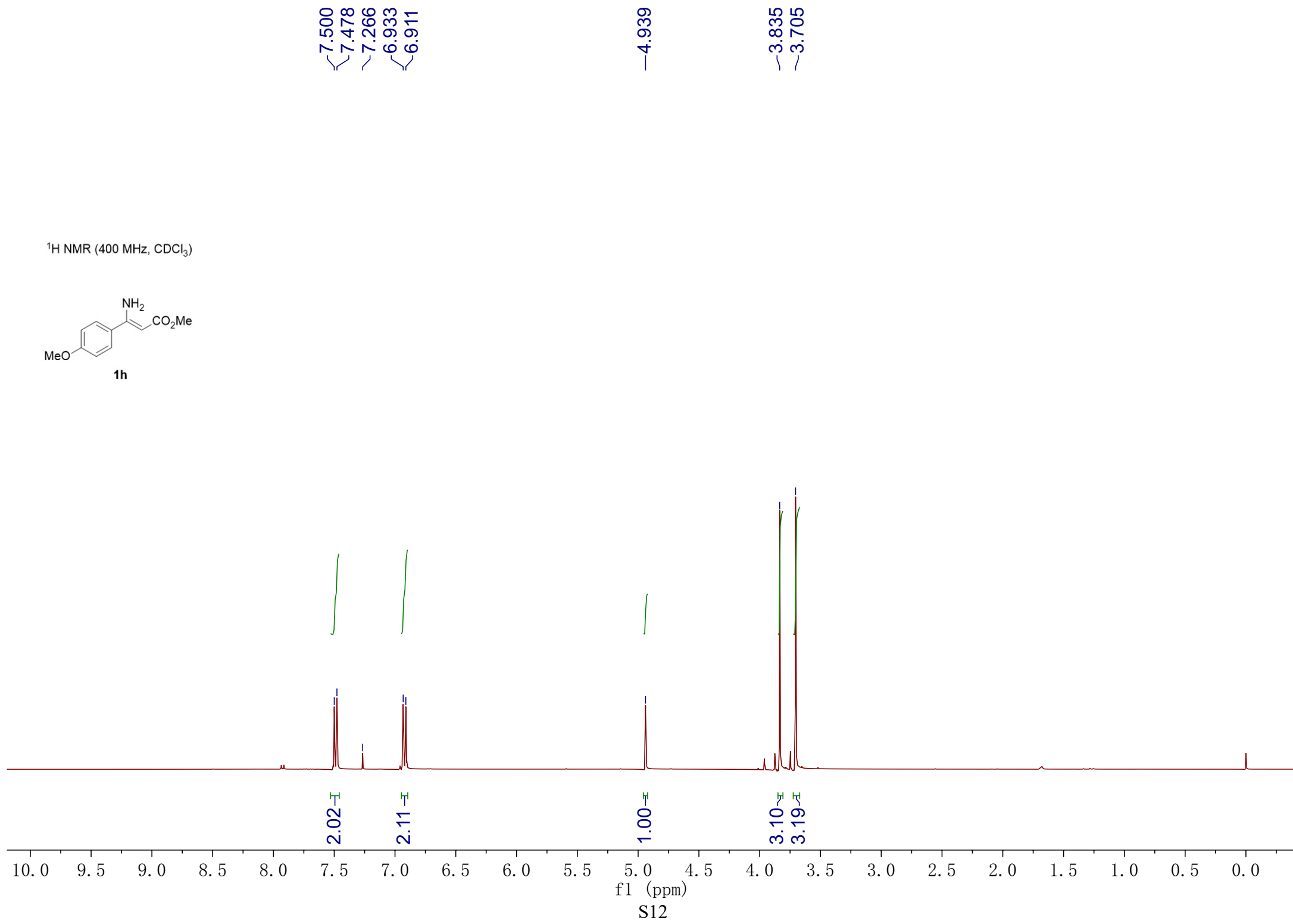
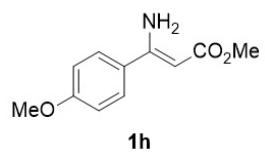
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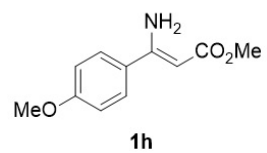
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¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)



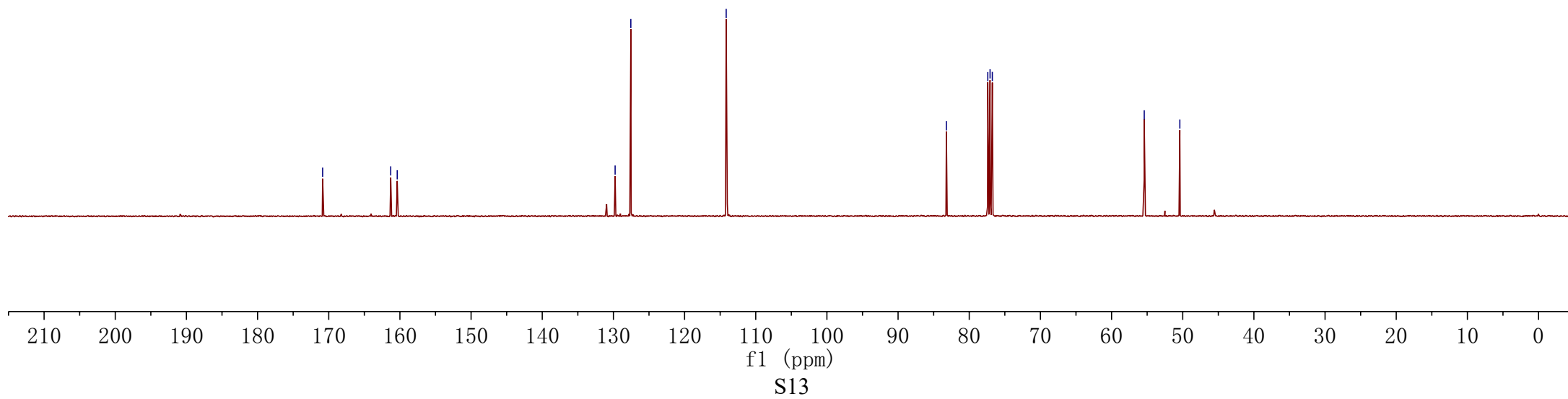
—170.9
—161.3
—160.4

—129.8
—127.6

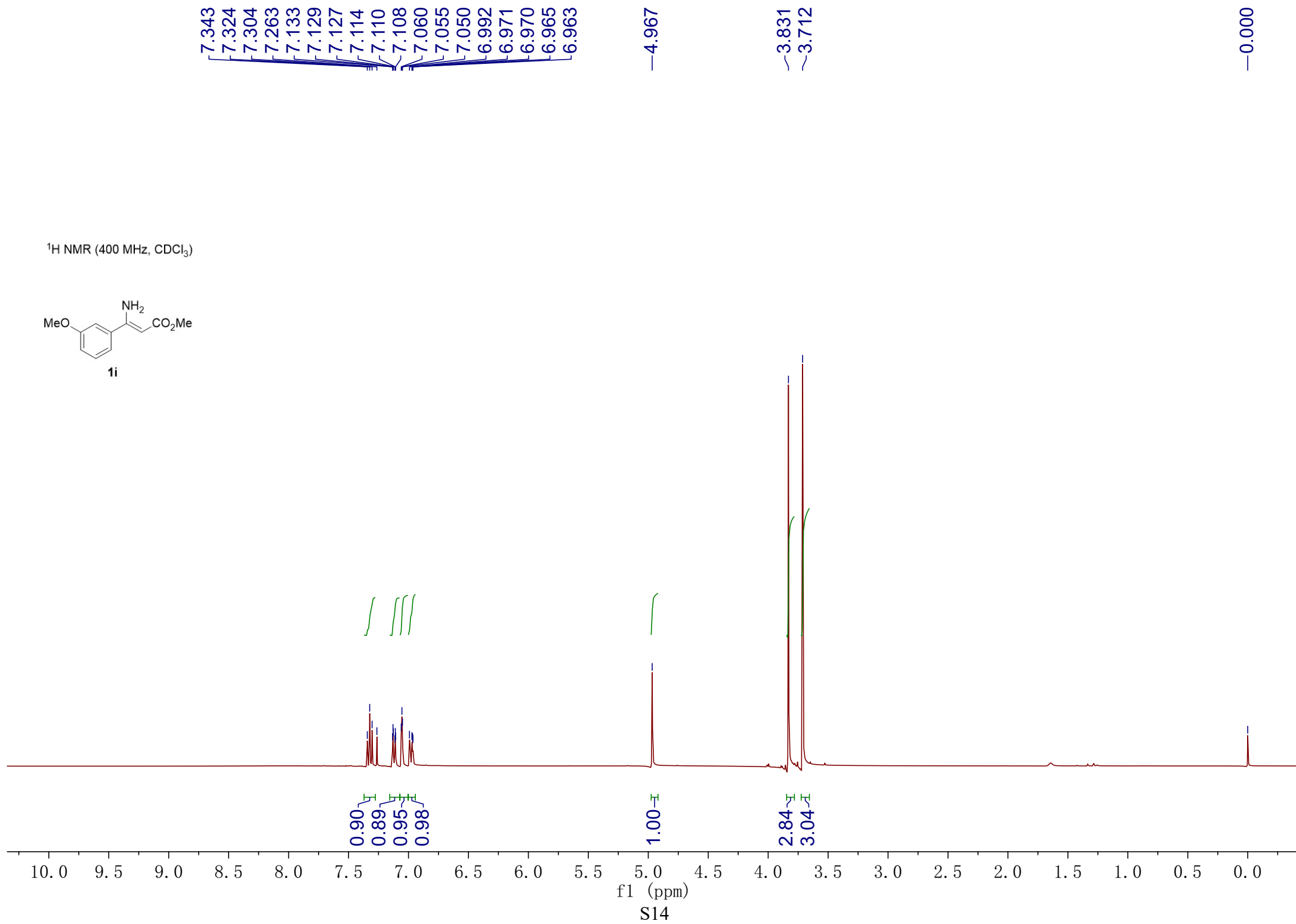
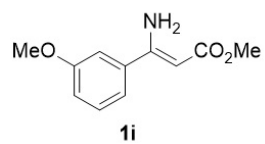
—114.2

—83.2
—77.4
—77.1
—76.8

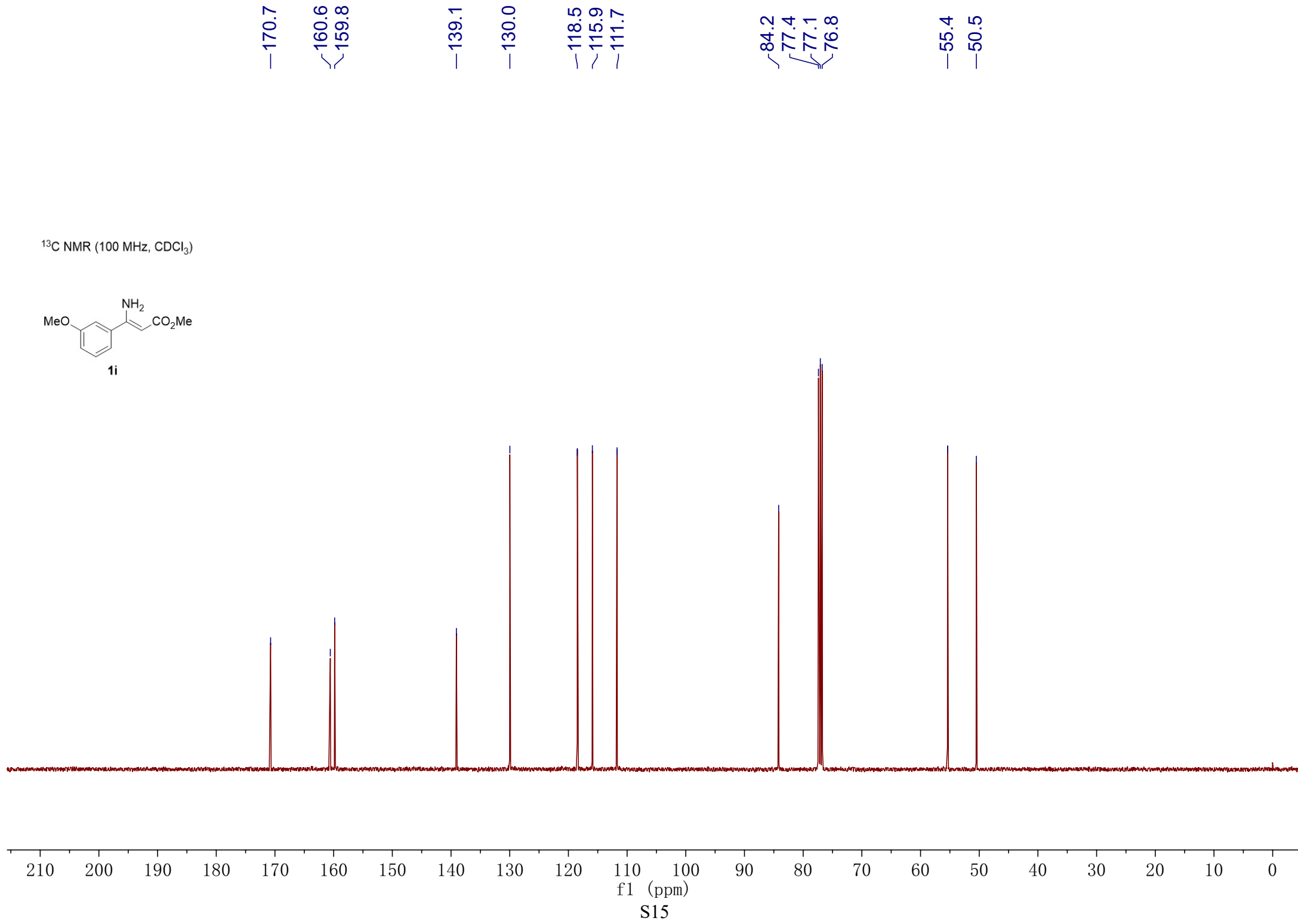
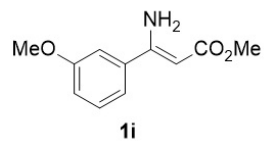
—55.4
—50.4



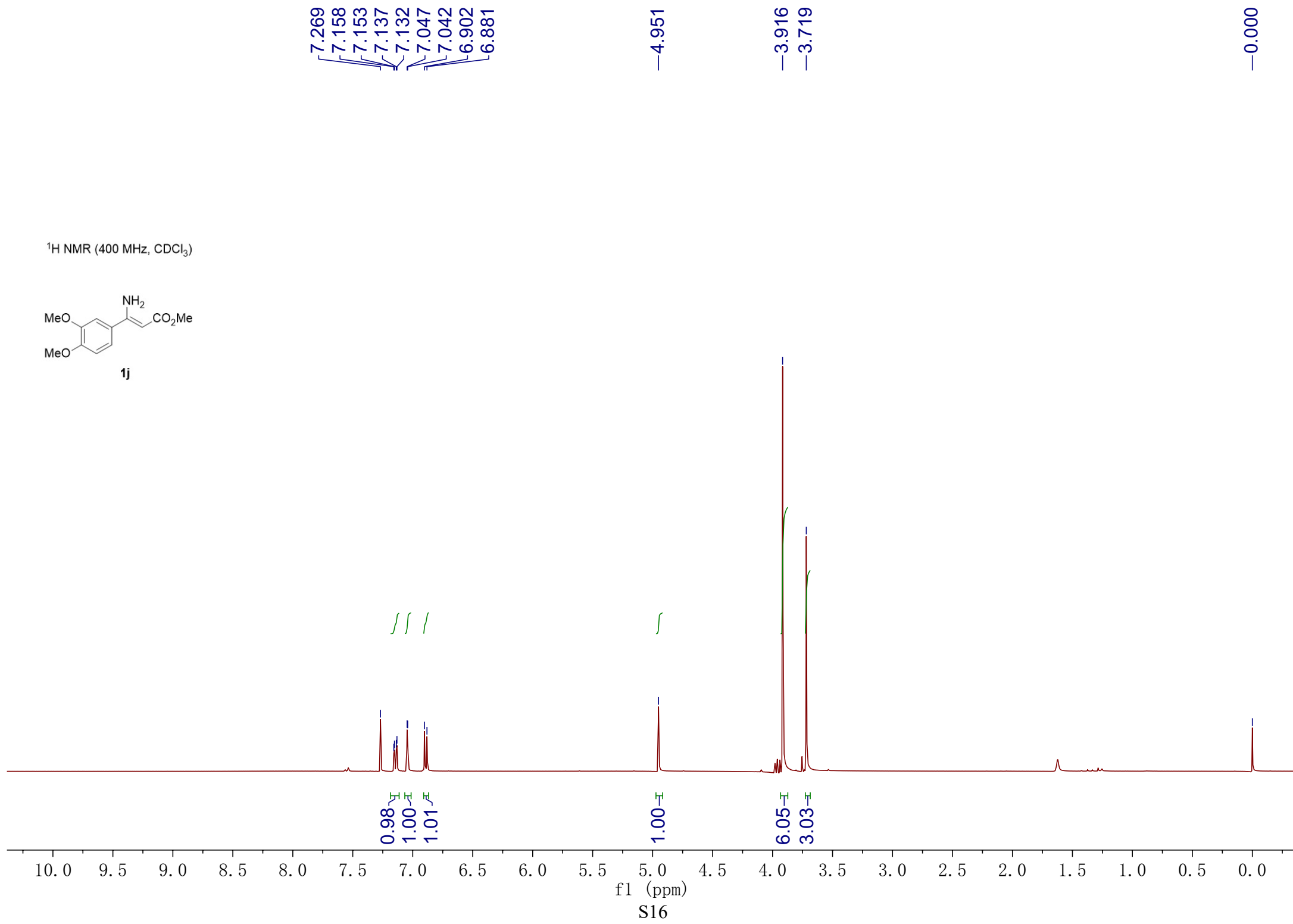
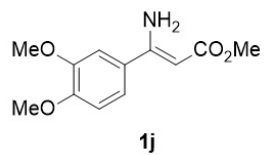
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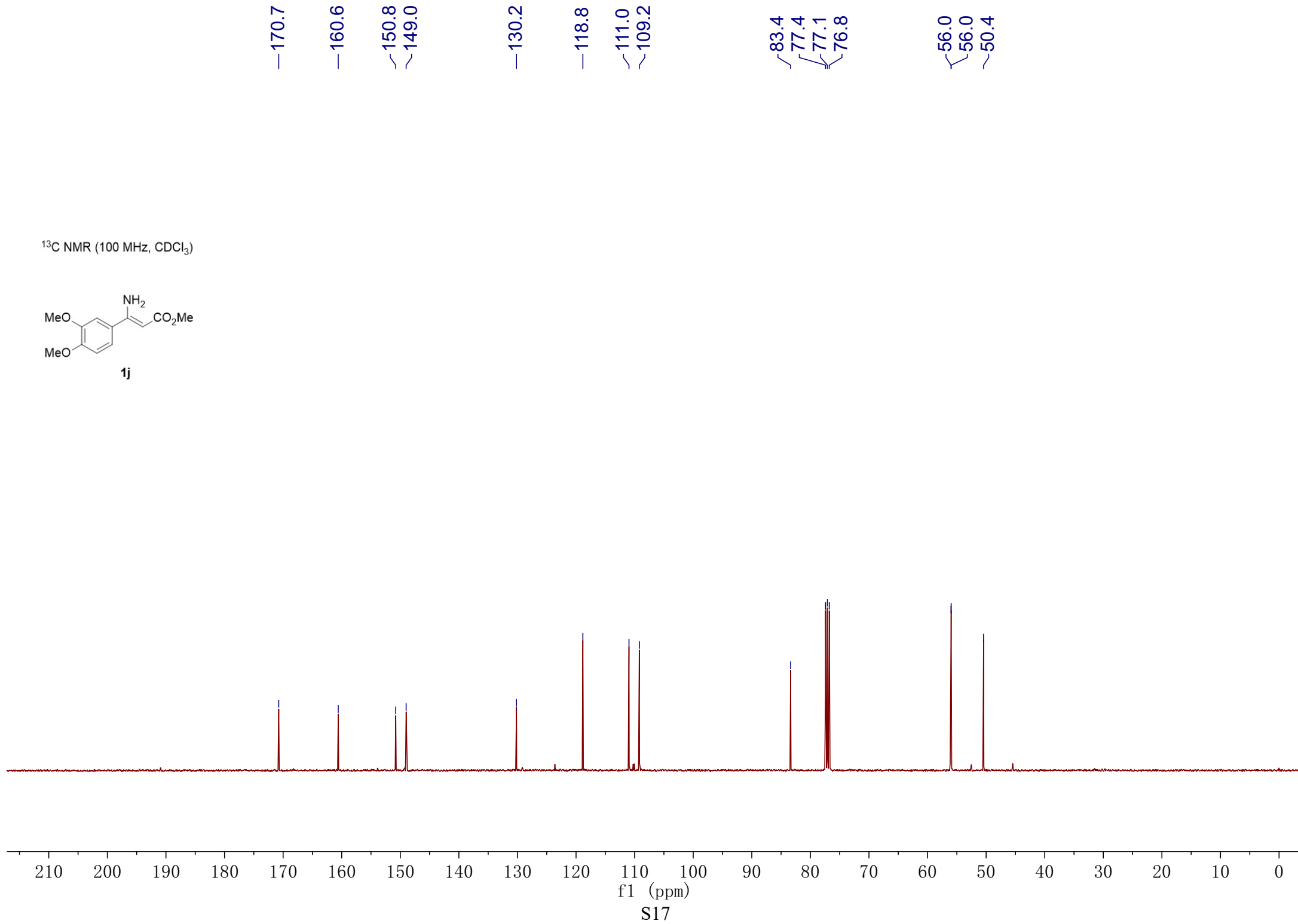
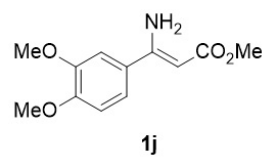
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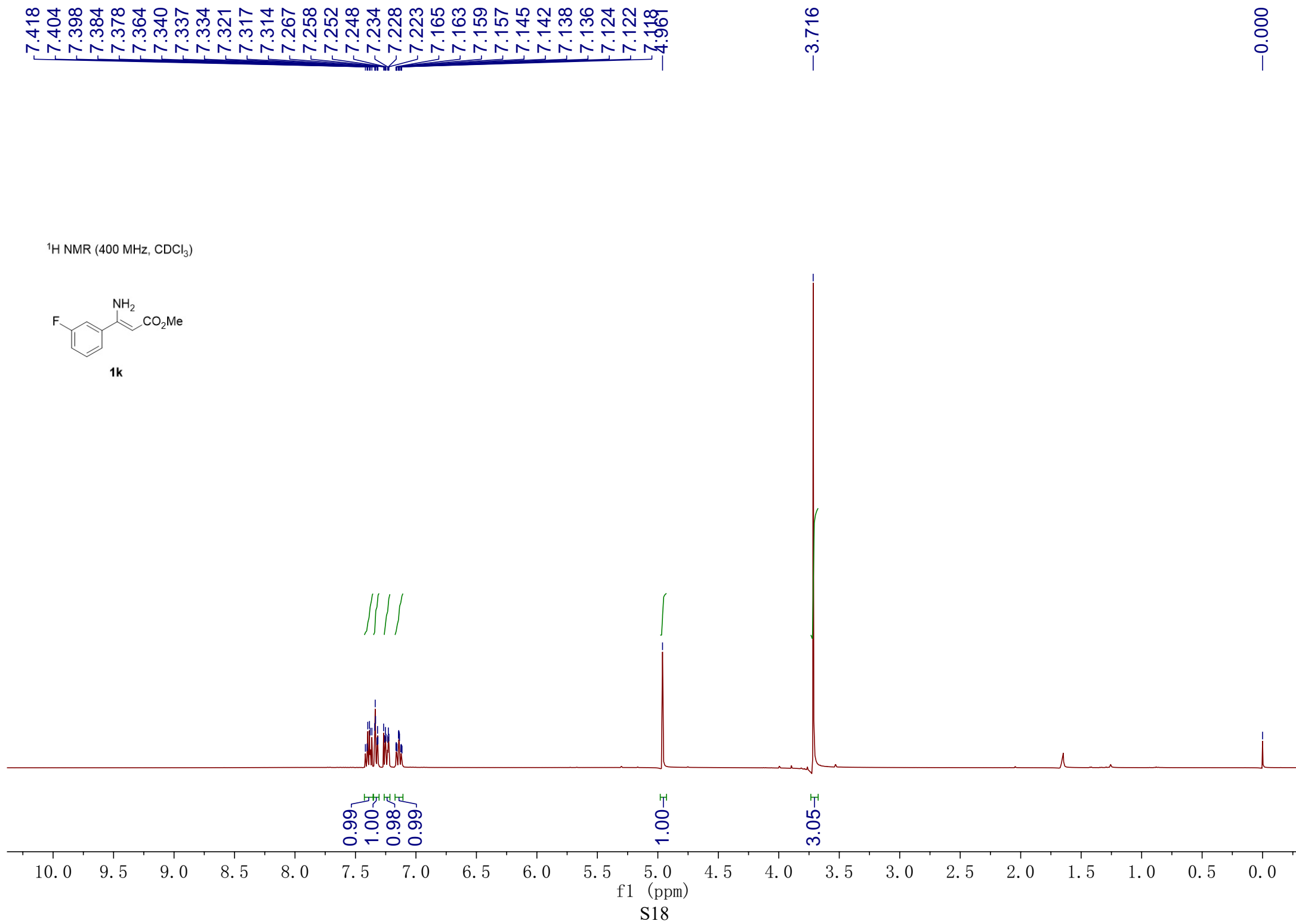
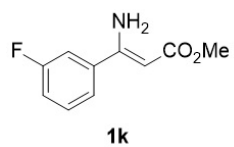
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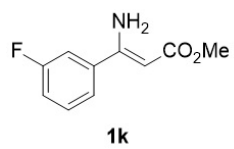


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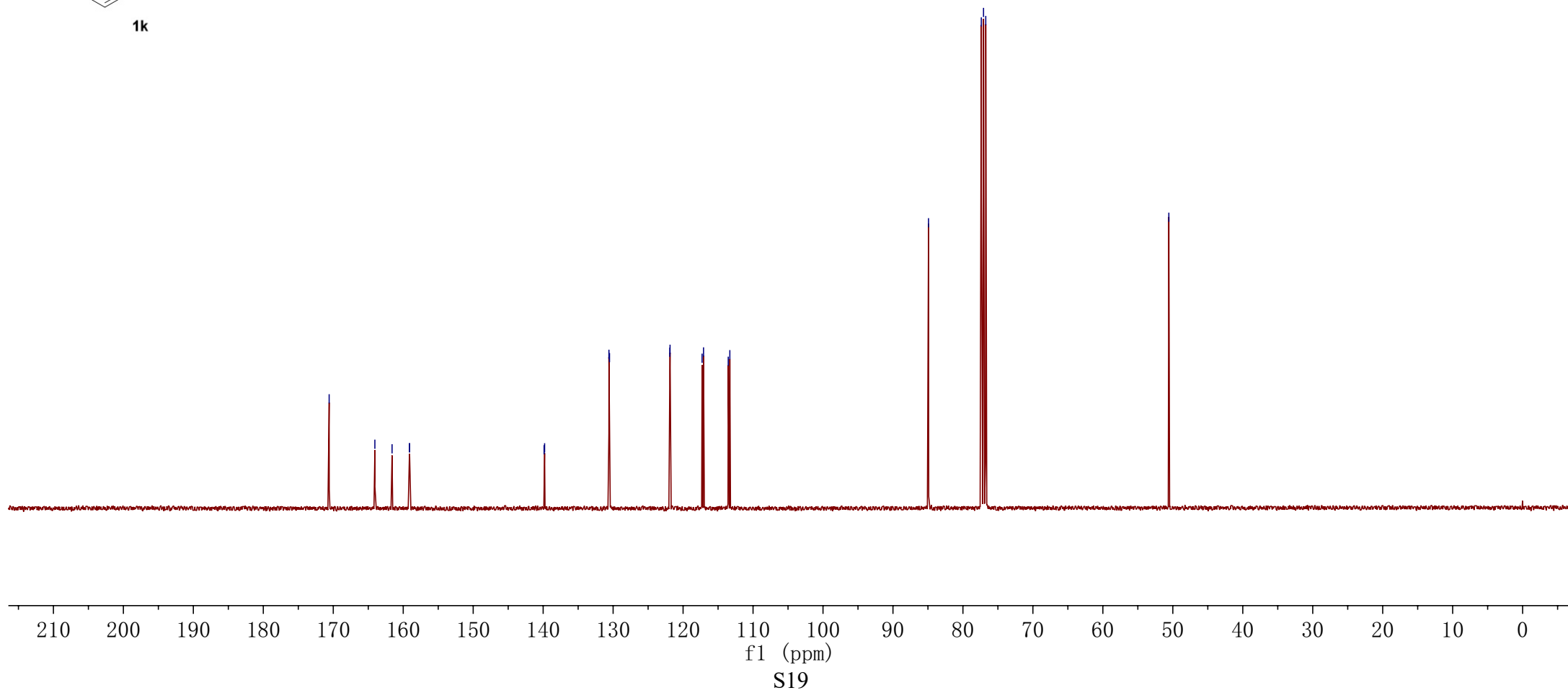
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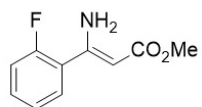




^{13}C NMR (100 MHz, CDCl_3)

170.6
164.1
161.6
159.1
159.1
139.9
139.8
130.6
130.5
121.9
121.9
117.3
117.1
113.5
113.3
84.9
77.4
77.1
76.7
50.6





11

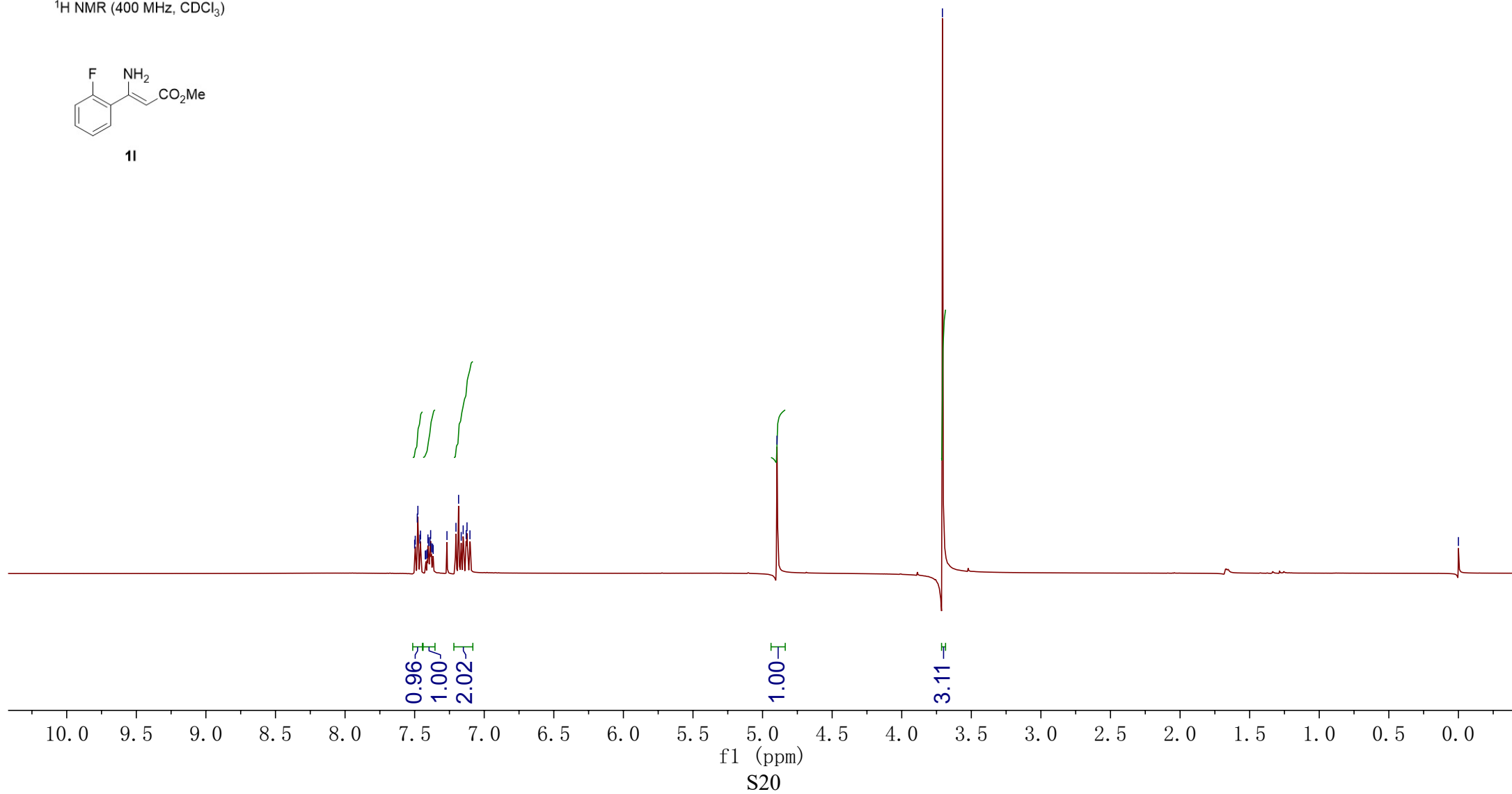
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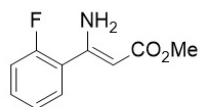
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7.481
7.477
7.462
7.458
7.423
7.419
7.410
7.405
7.400
7.390
7.385
7.380
7.371
7.367
7.268
7.203
7.184
7.165
7.151
7.129
7.124
7.103

—4.896

—3.708

—0.000

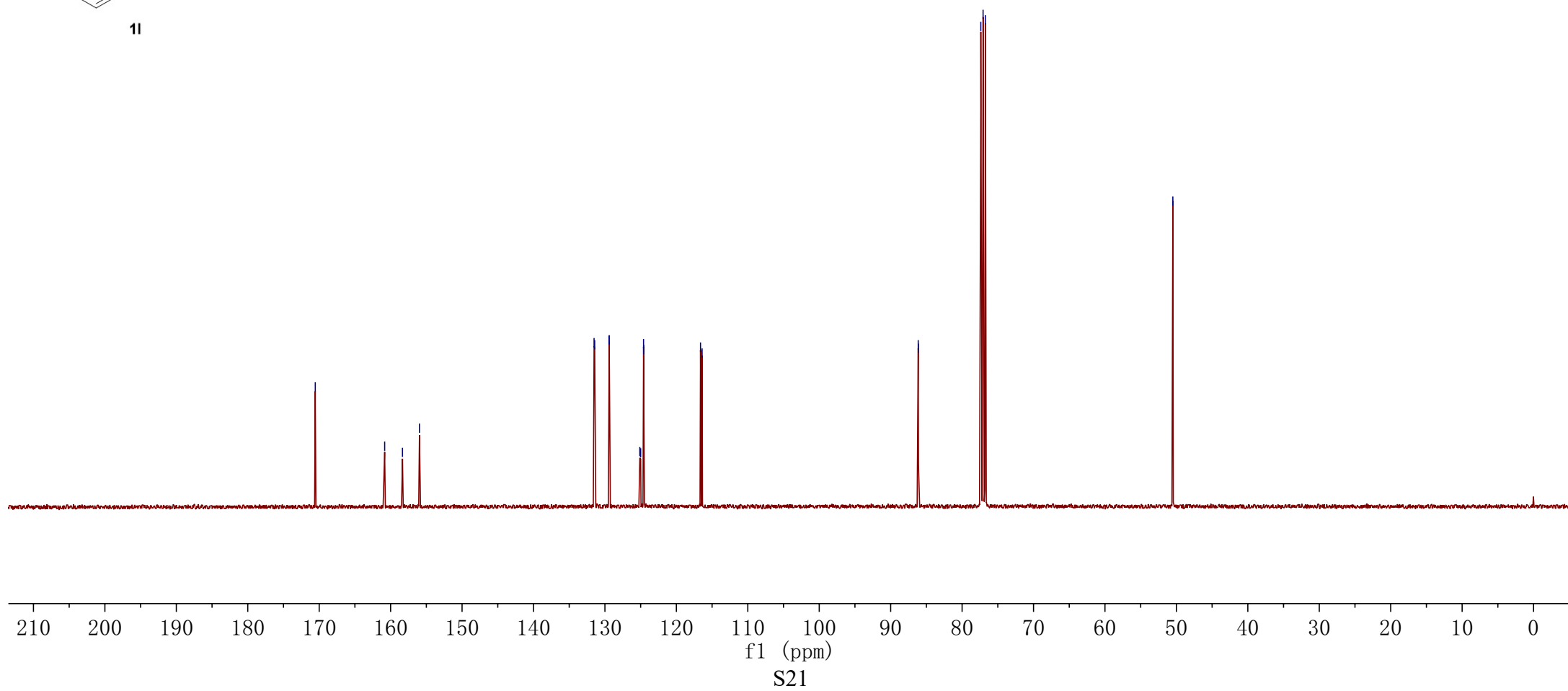




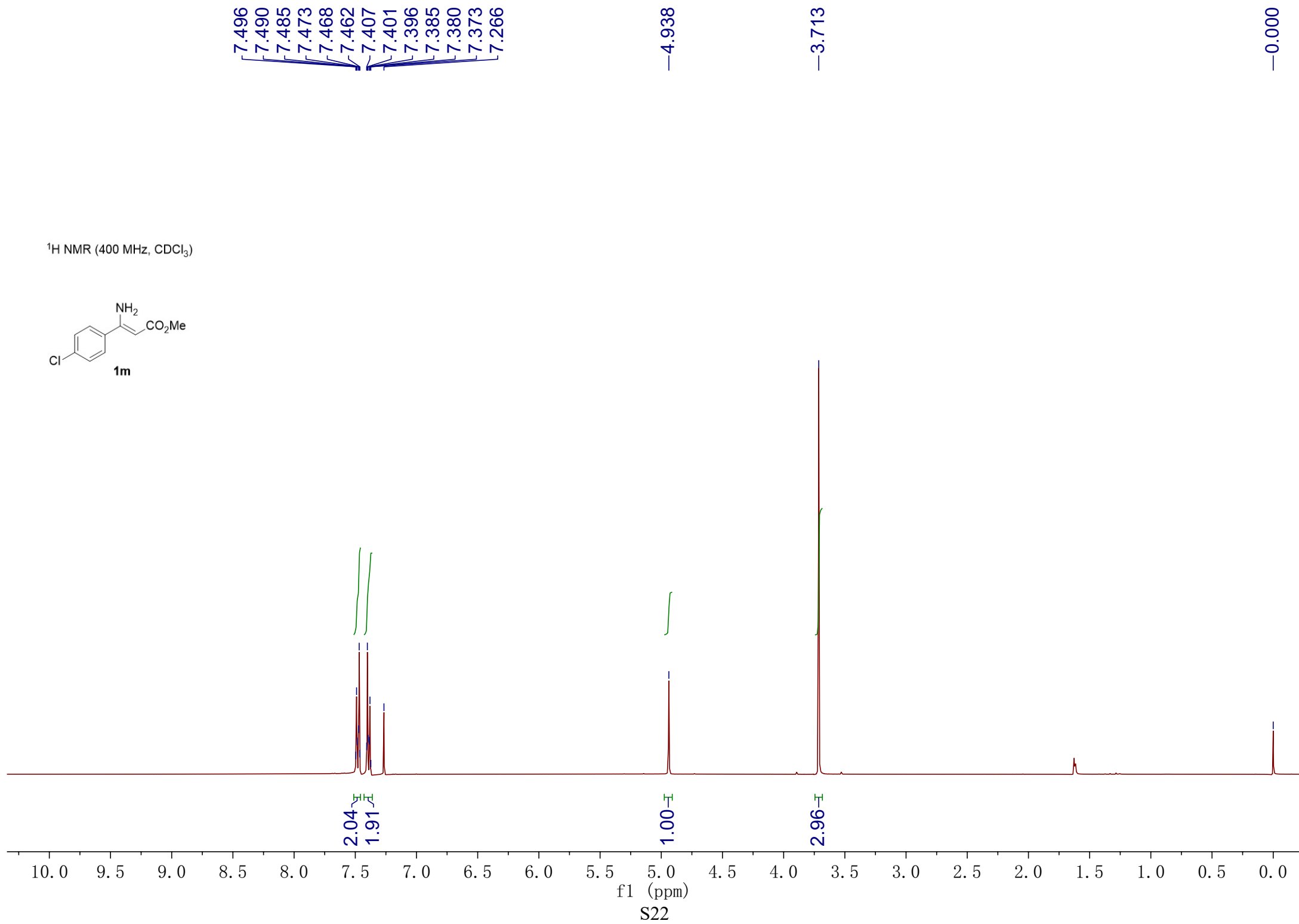
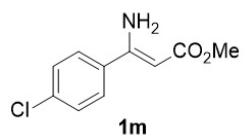
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^{13}C NMR (100 MHz, CDCl_3)

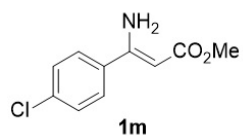
170.6
160.8
158.3
156.0
131.5
131.4
129.4
129.4
125.1
125.0
124.6
124.6
116.6
116.4
86.1
86.1
77.4
77.1
76.7
50.5



¹H NMR (400 MHz, CDCl₃)



^{13}C NMR (100 MHz, CDCl_3)



—170.6

—159.3

—136.3

—136.0

—129.1

—127.5

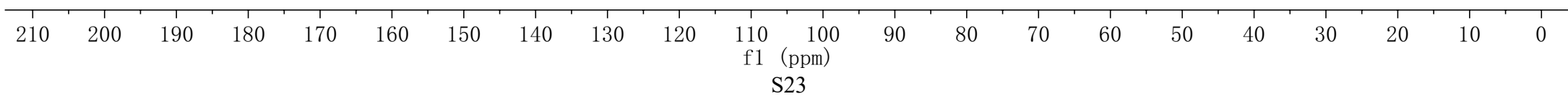
—84.7

—77.4

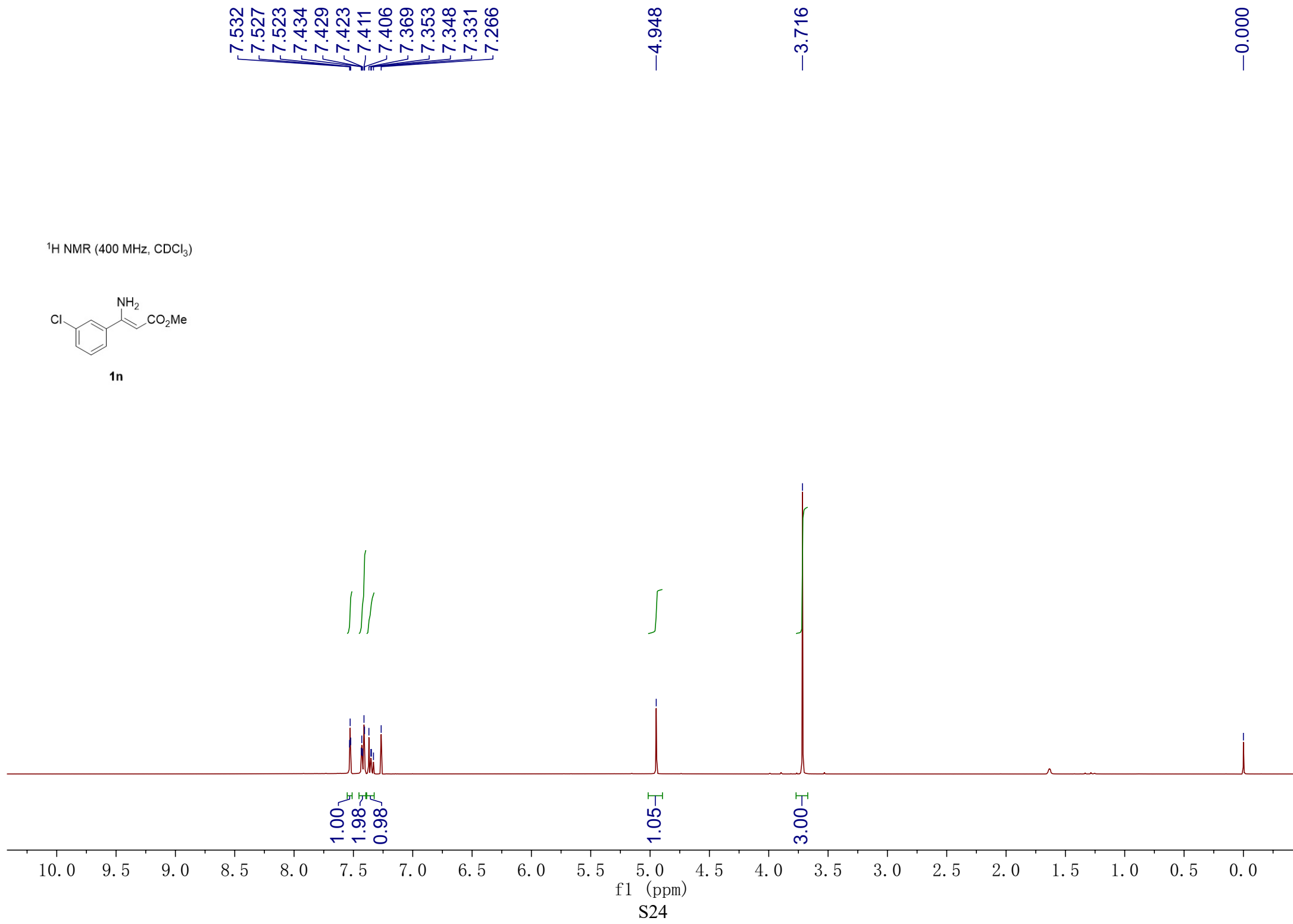
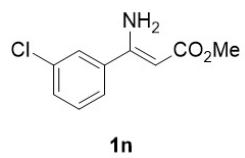
—77.1

—76.7

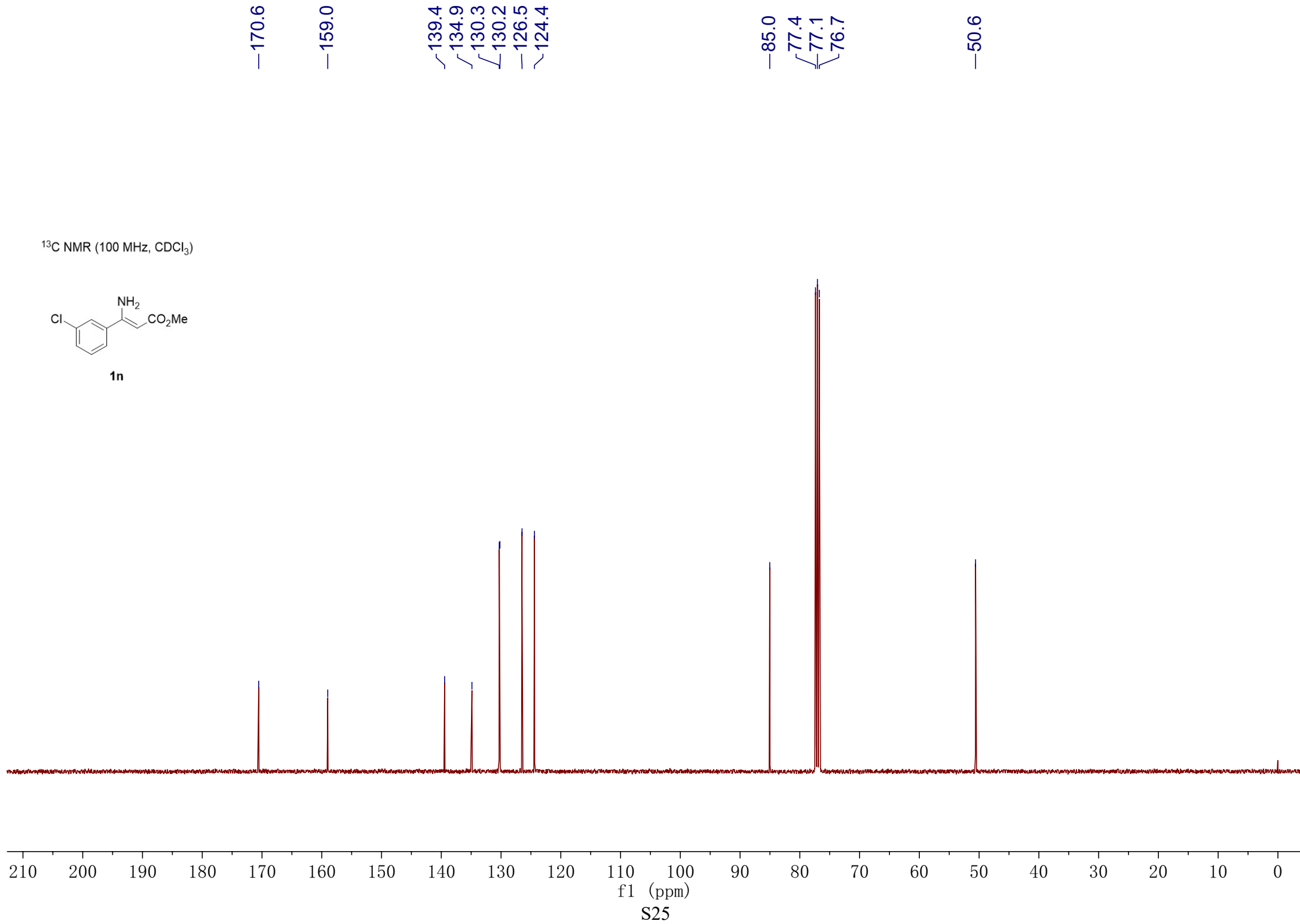
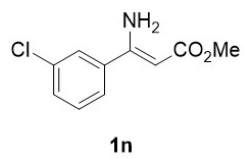
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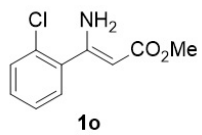


¹H NMR (400 MHz, CDCl₃)

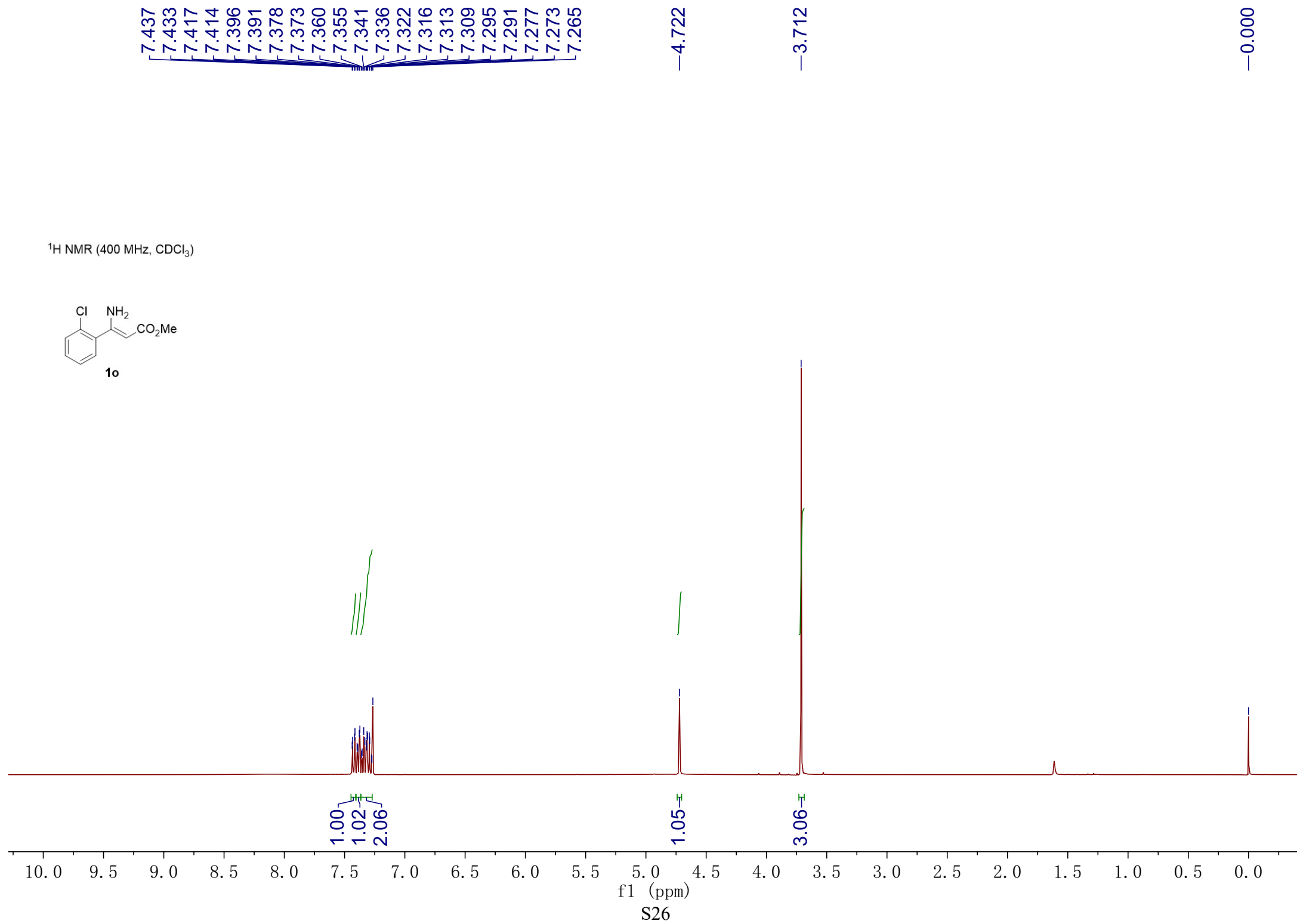


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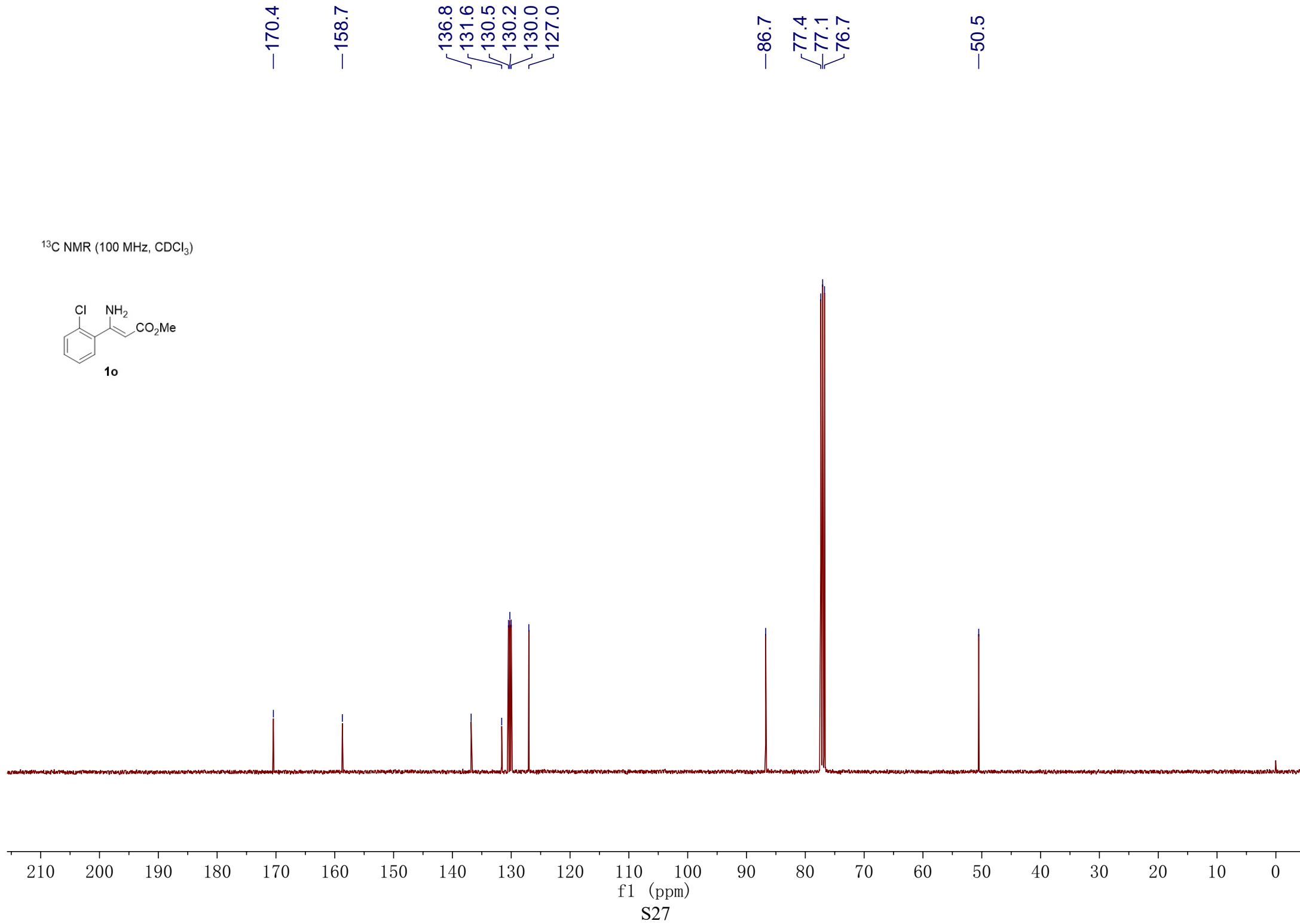
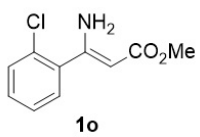




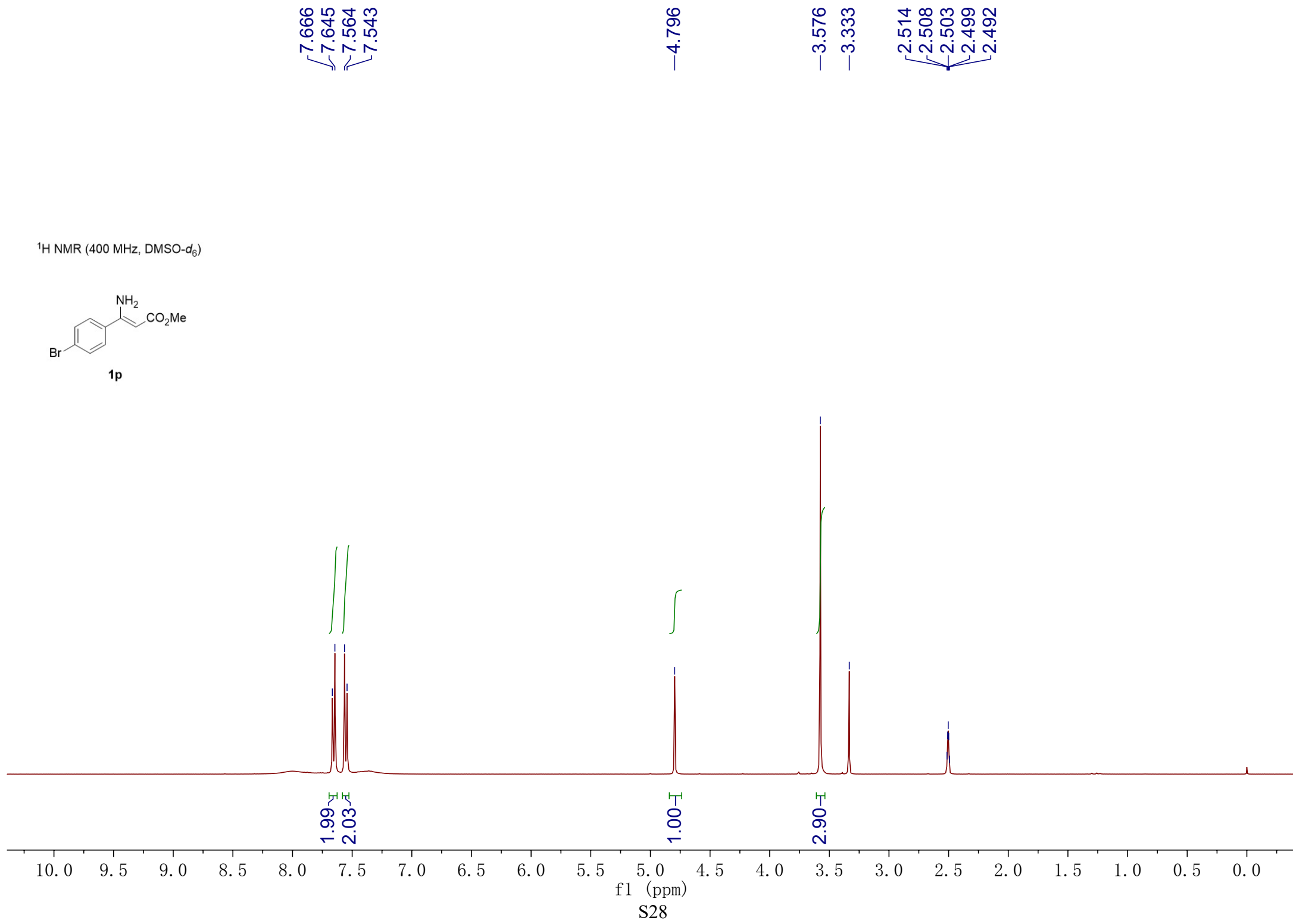
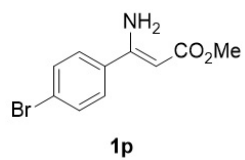
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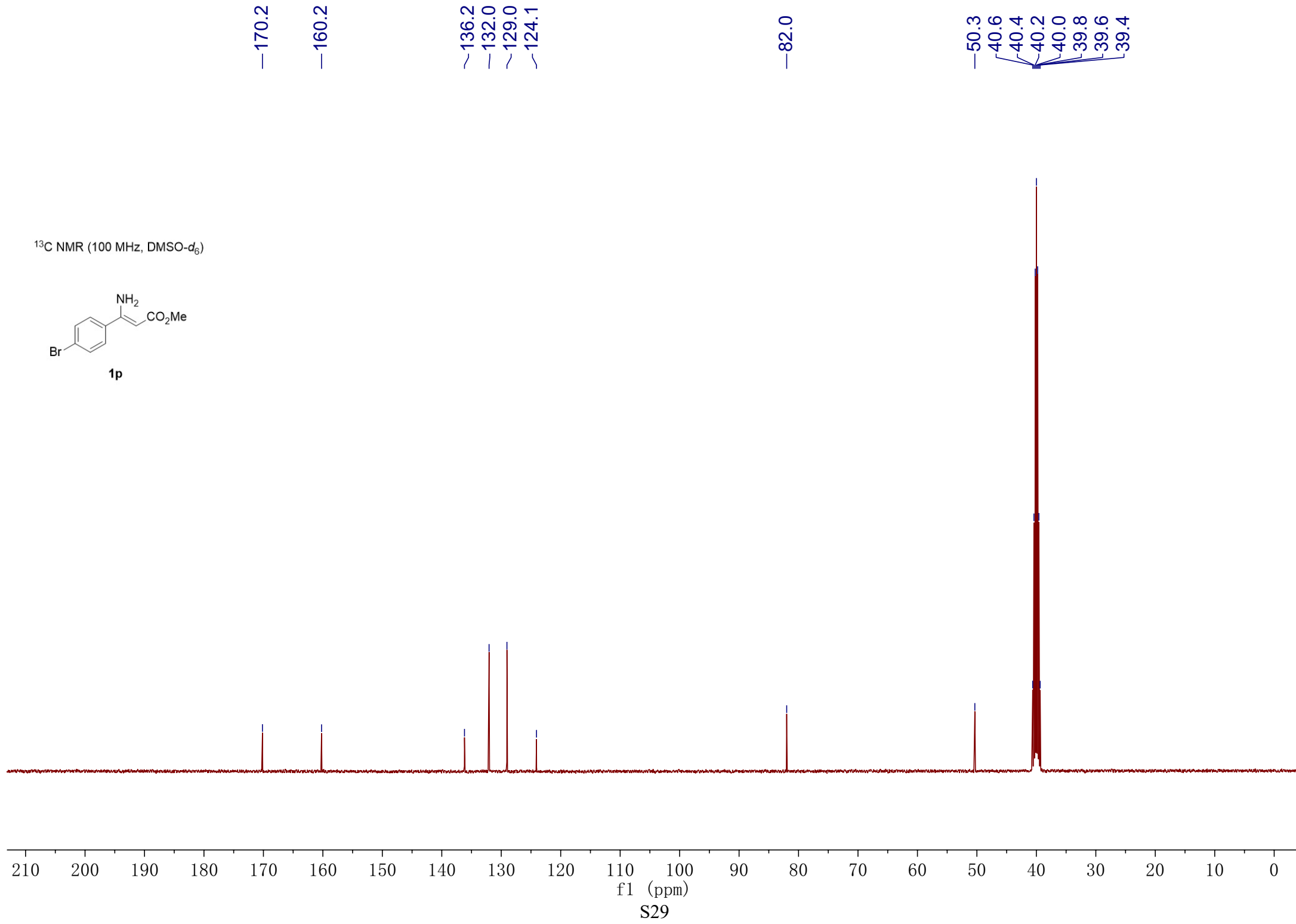
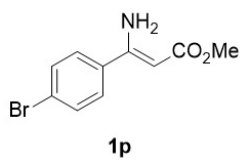
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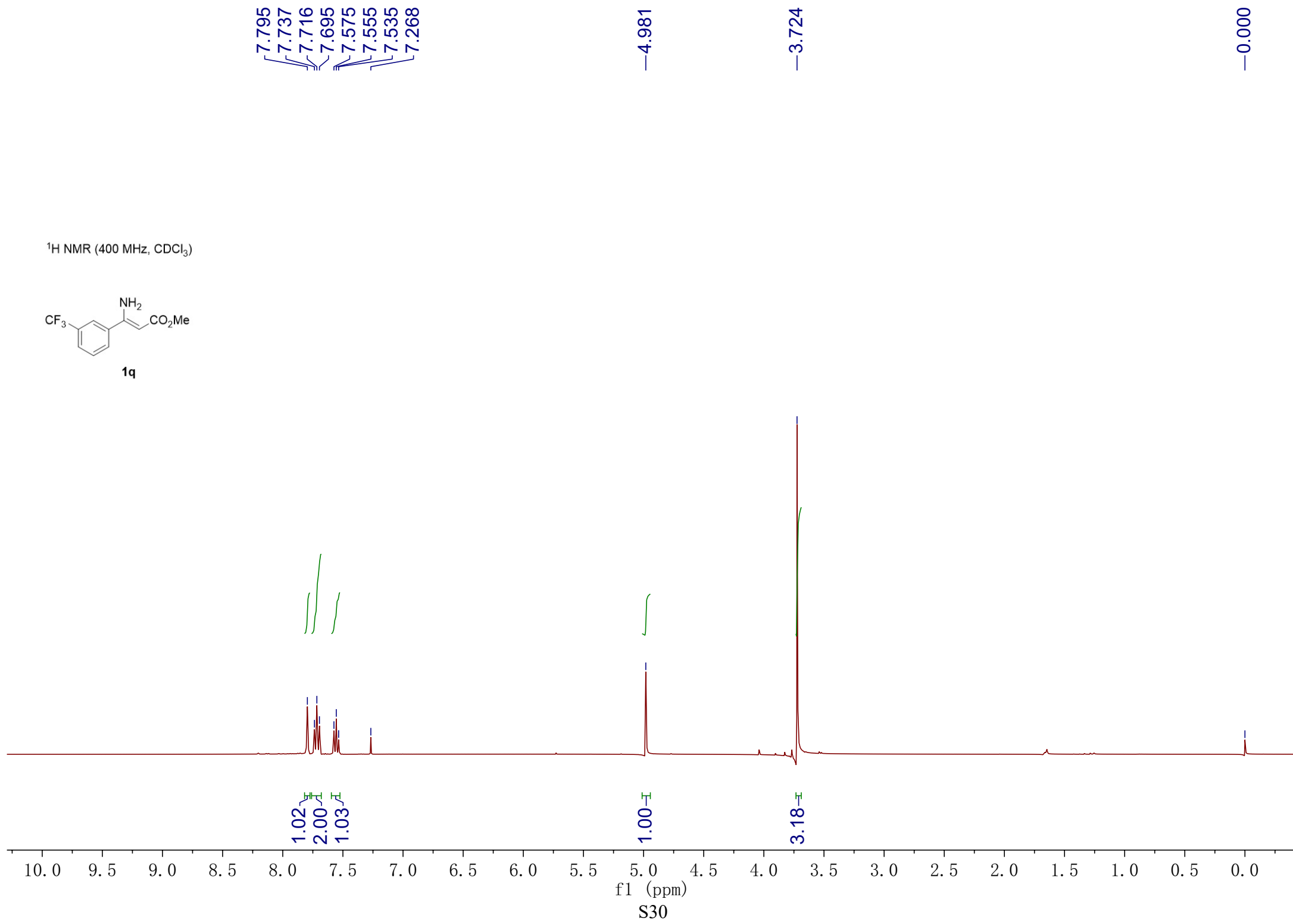
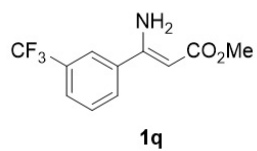
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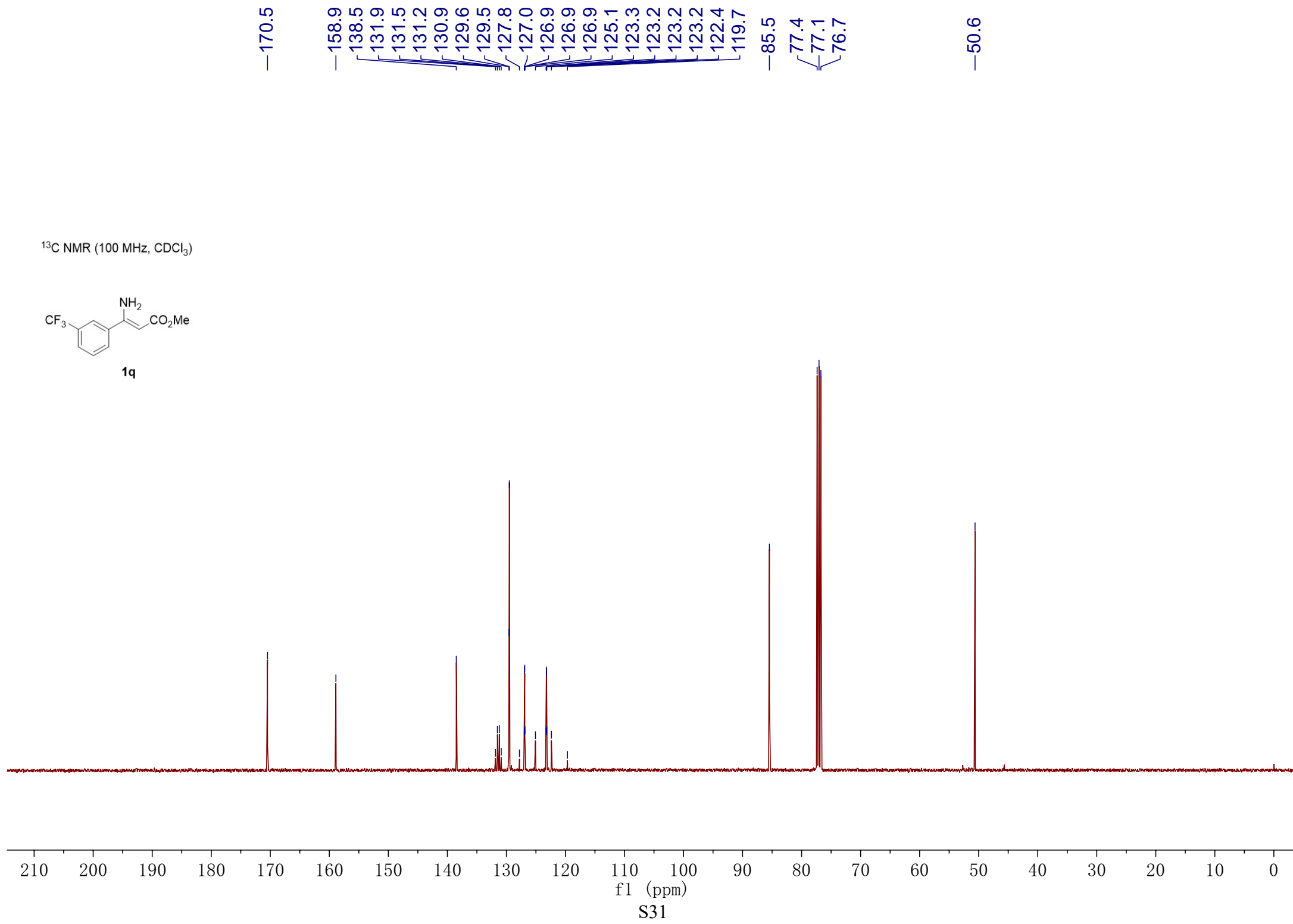
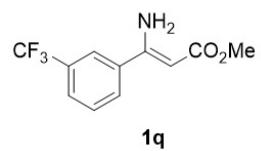
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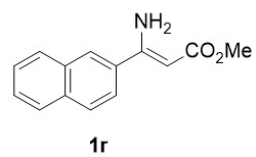


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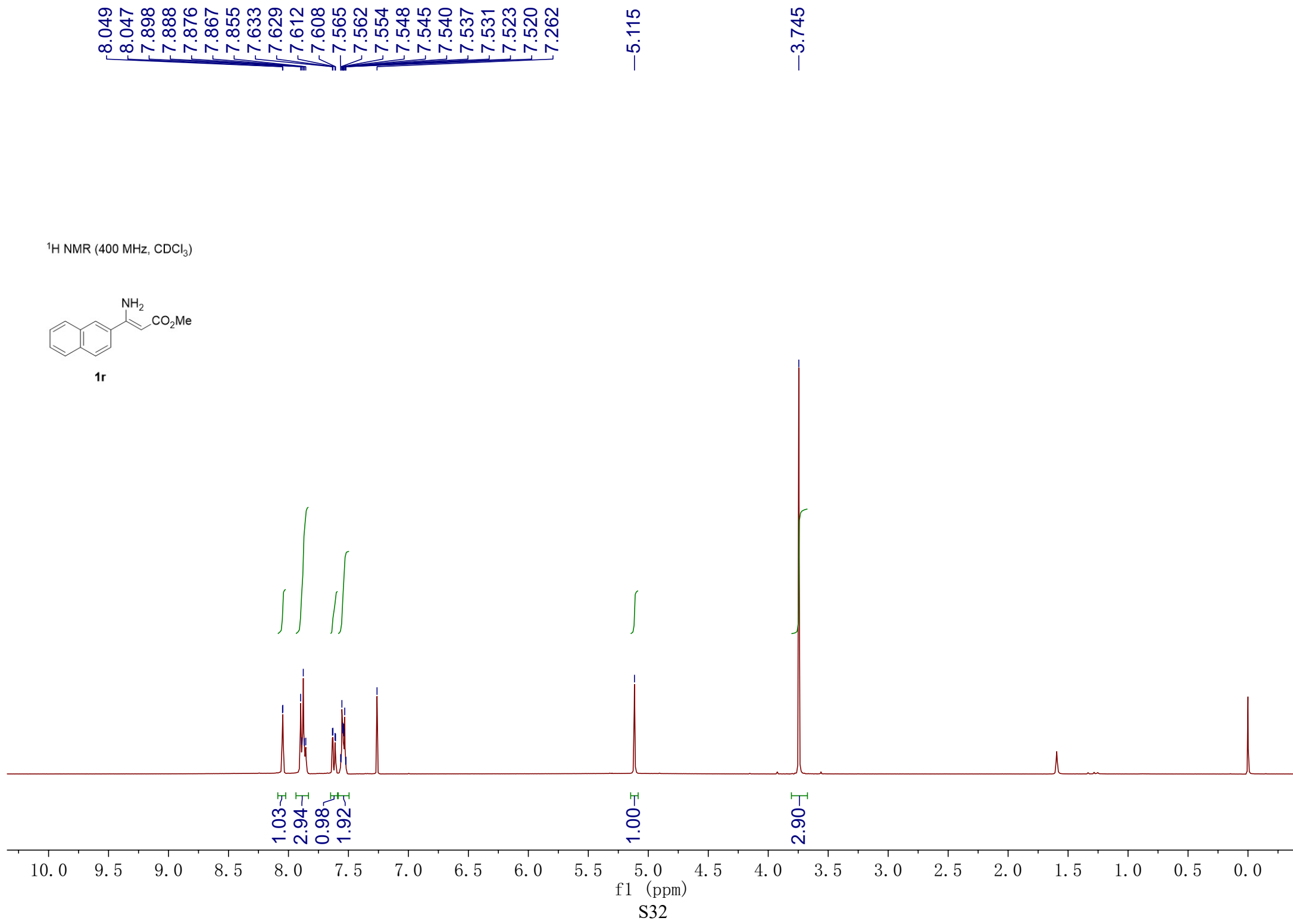


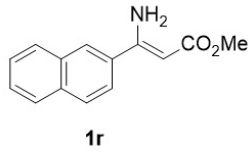
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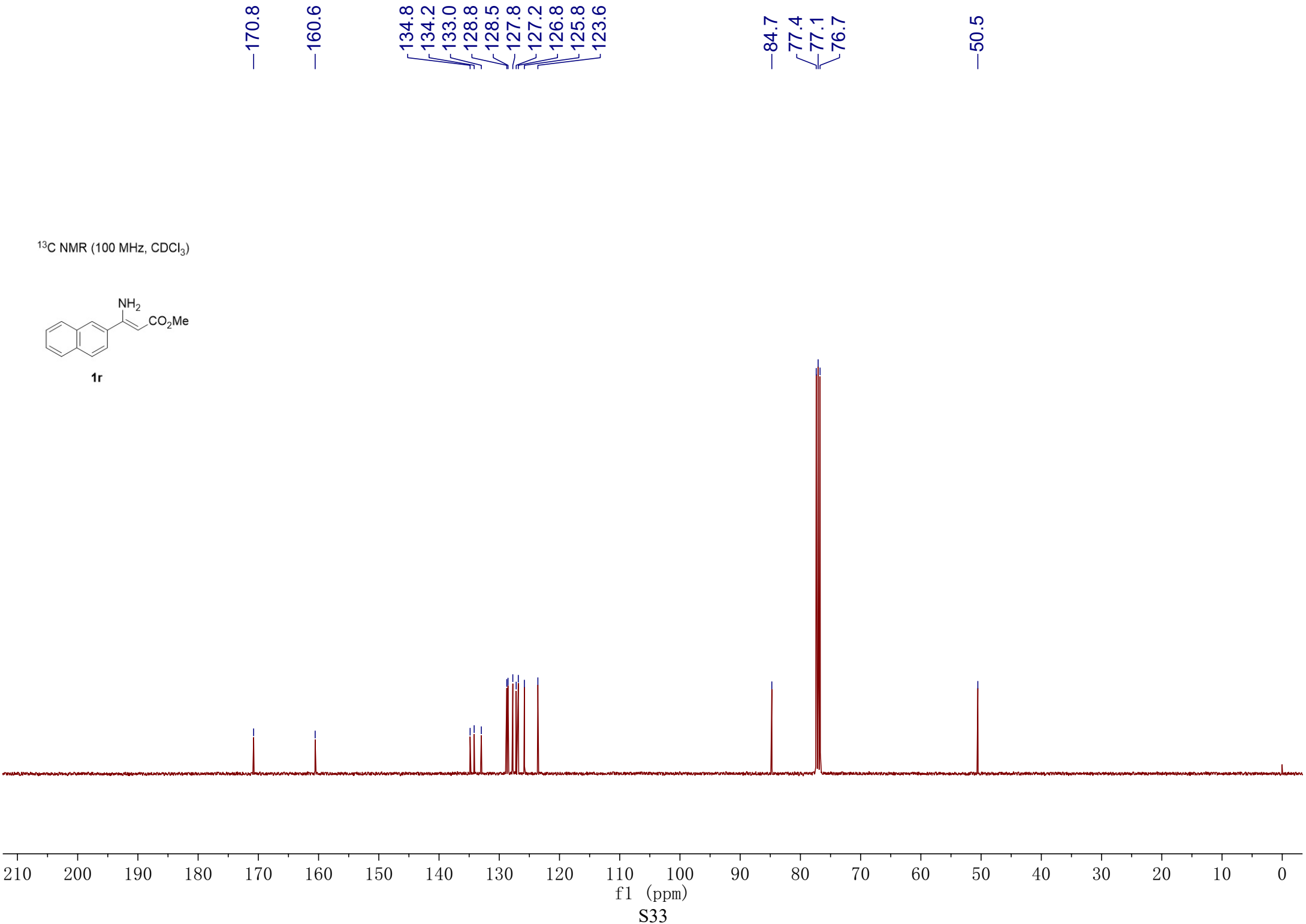


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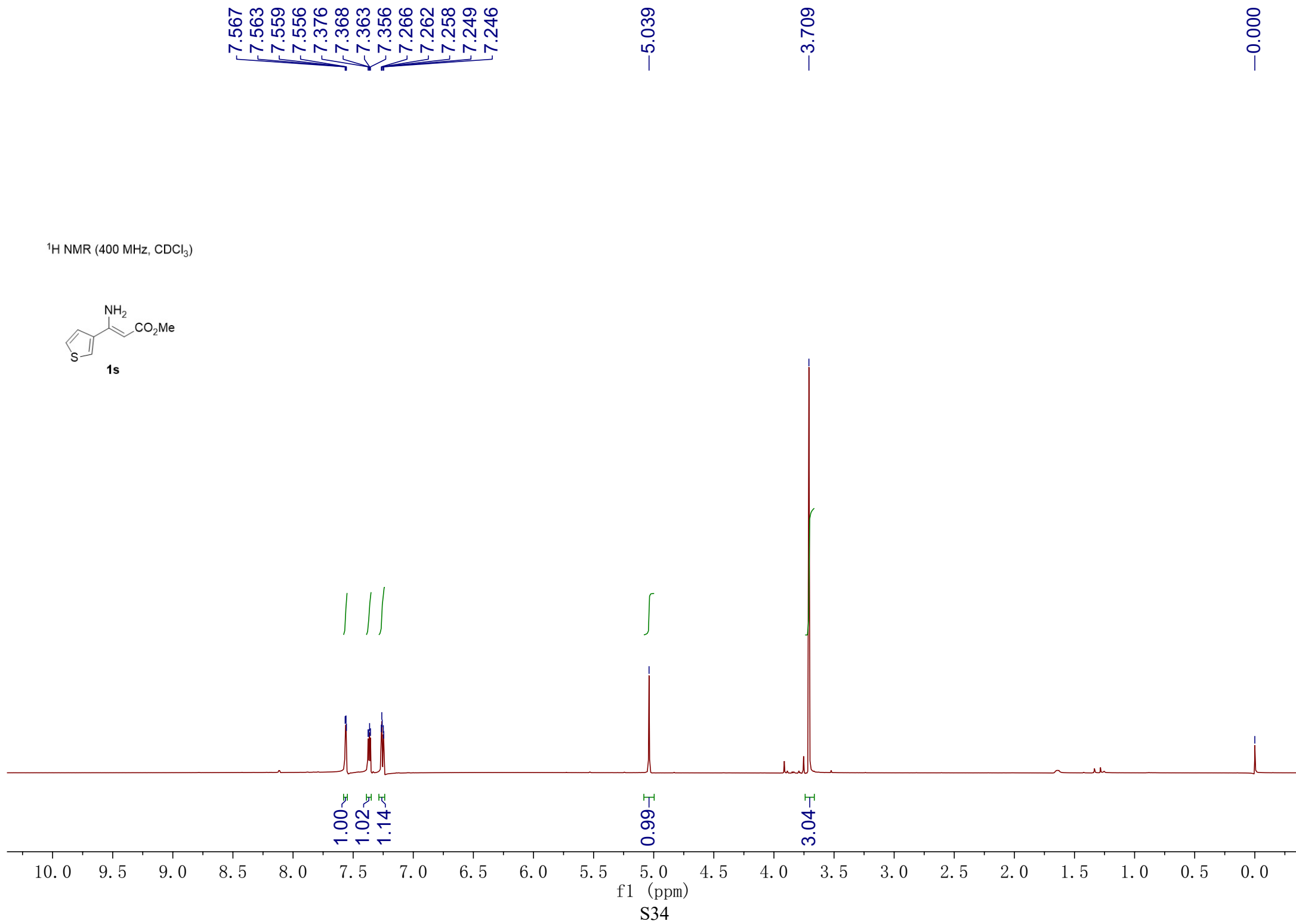
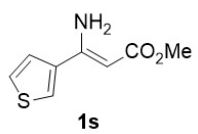




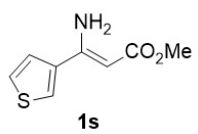
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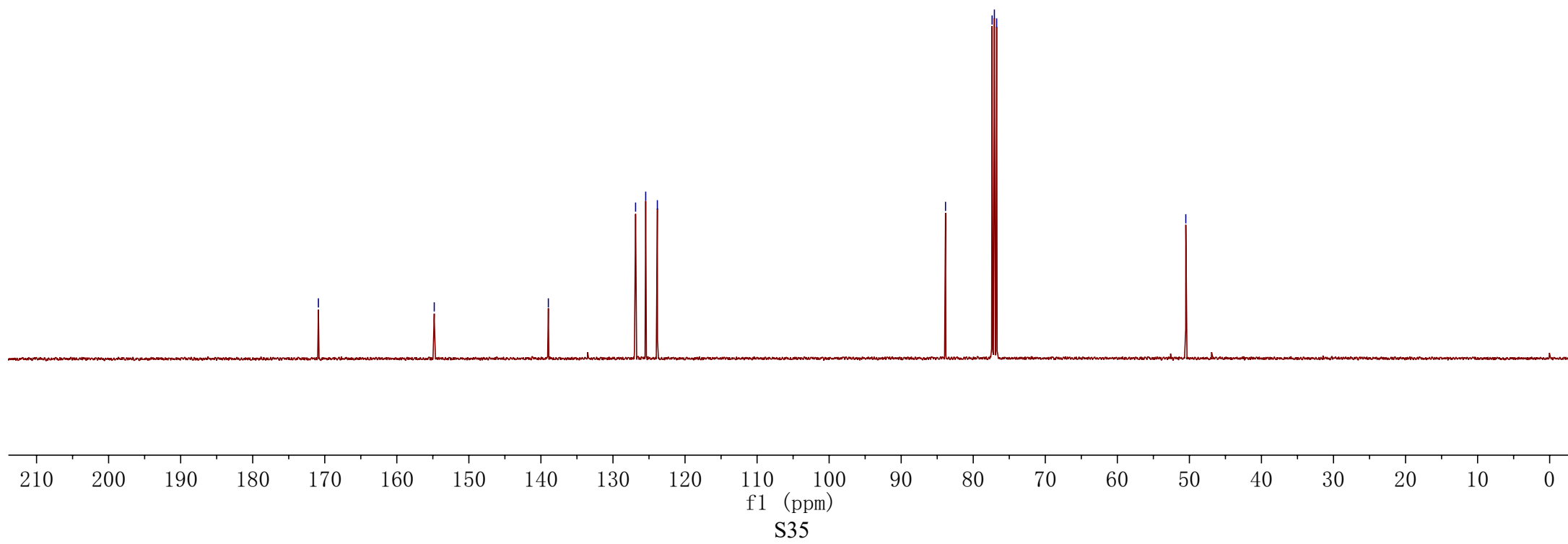
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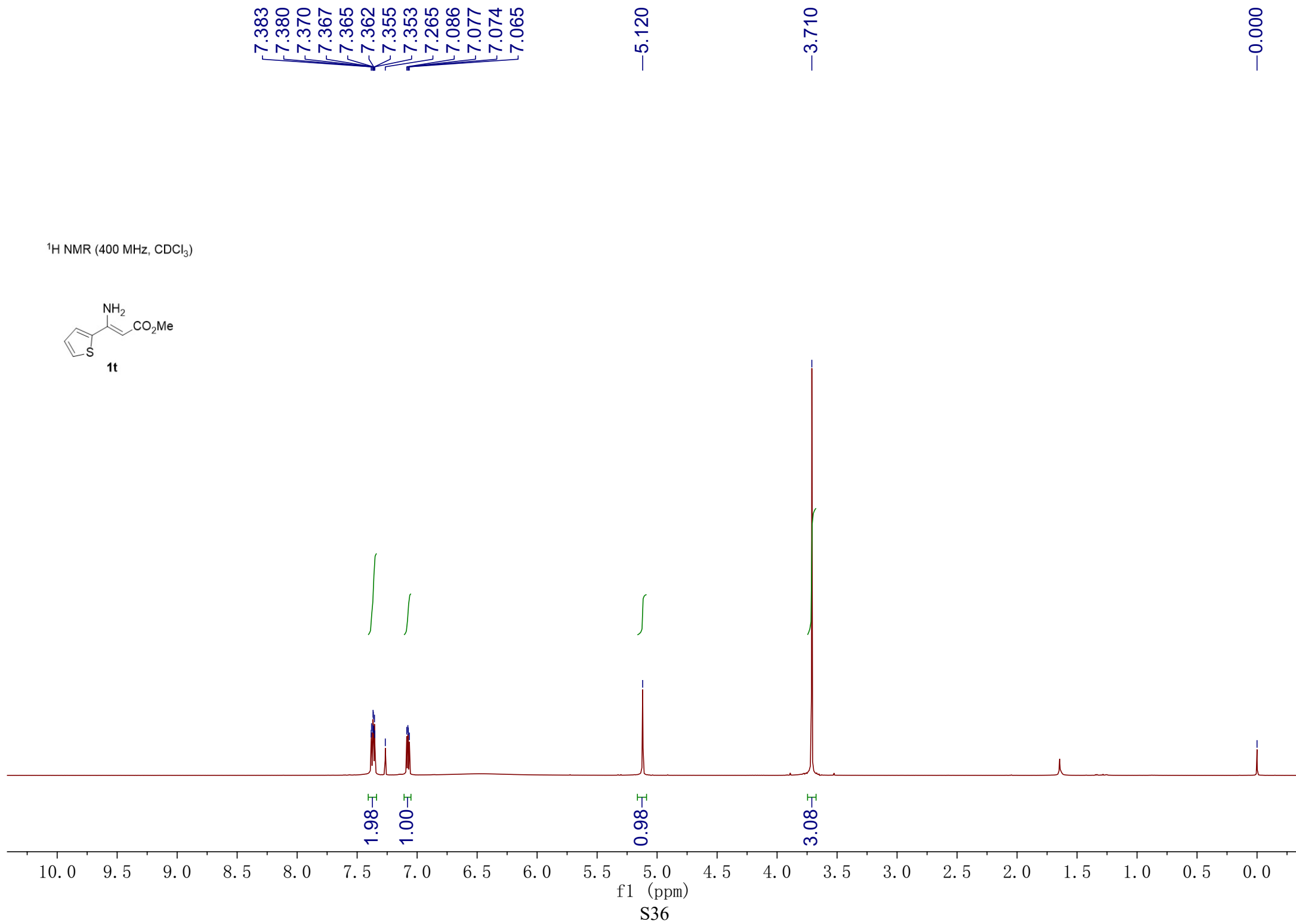
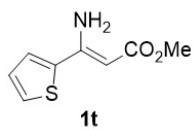
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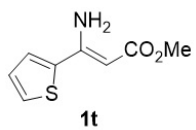
—170.9 —154.8 —139.0
126.9 125.5 123.8
83.8 77.4 77.1 76.7
—50.5



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)



—170.6

—153.2

—140.0

—127.9

—127.4

—125.7

—84.0

—77.4

—77.1

—76.7

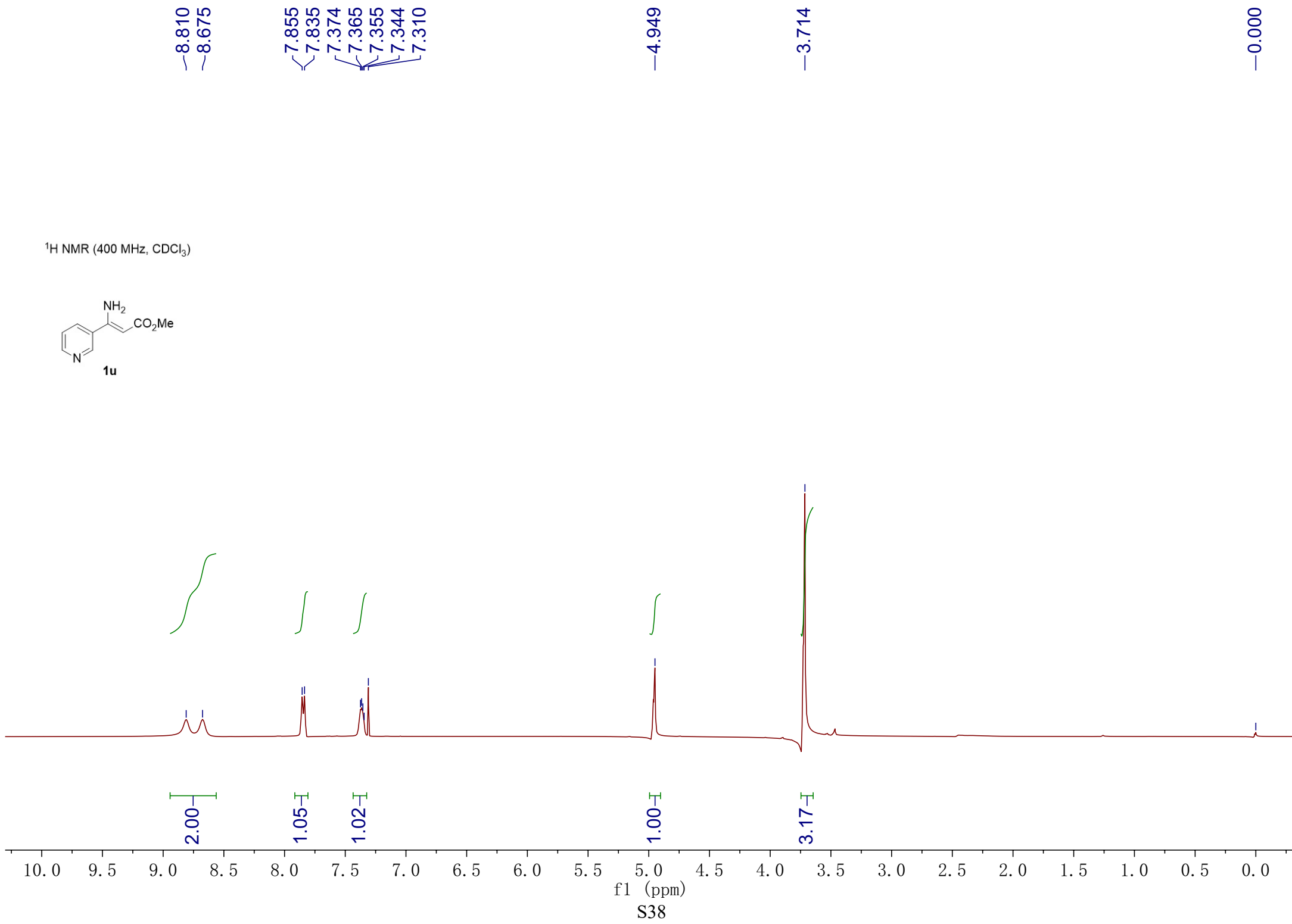
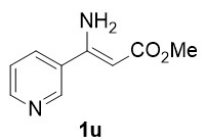
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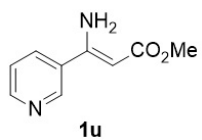
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S37

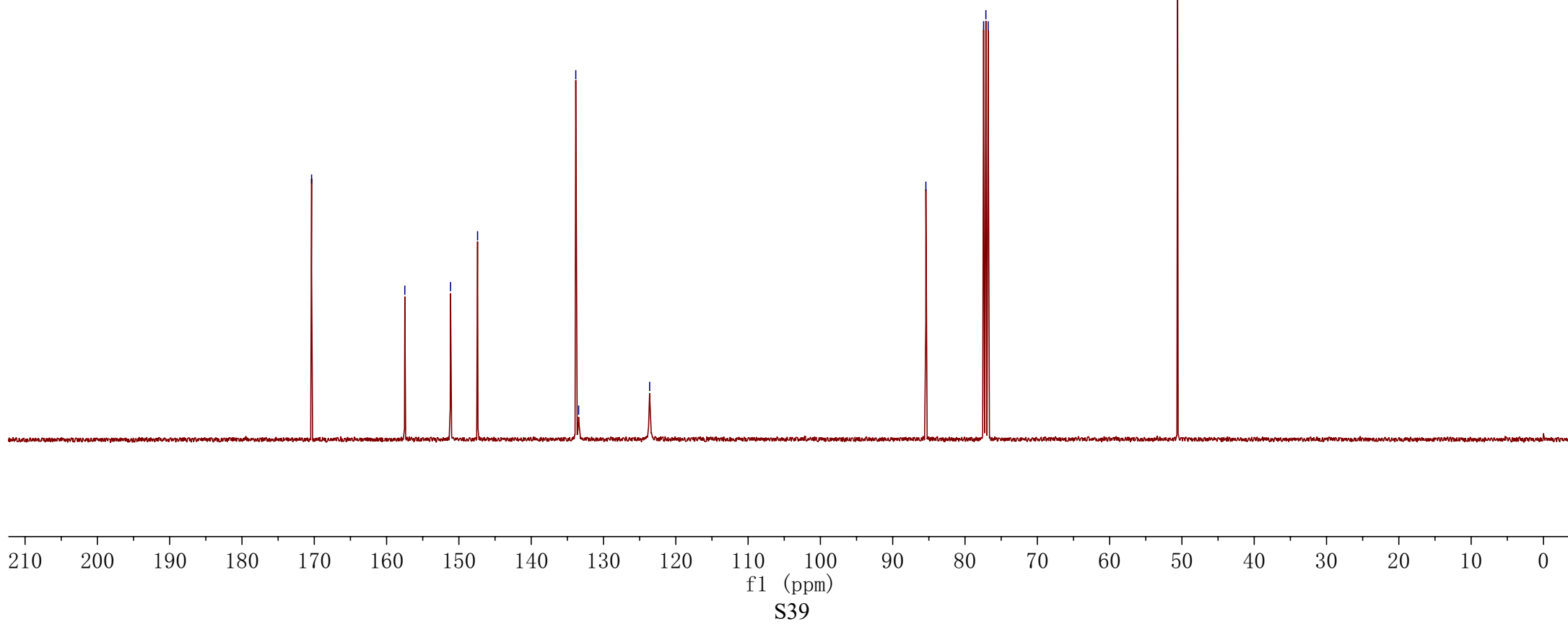
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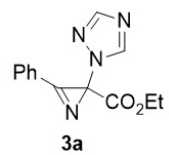
¹³C NMR (100 MHz, CDCl₃)



—170.4
—157.5
—151.2
—147.4
—133.8
—133.4
—123.6
—85.4
—77.4
—77.1
—76.8
—50.6



¹H NMR (400 MHz, CDCl₃)

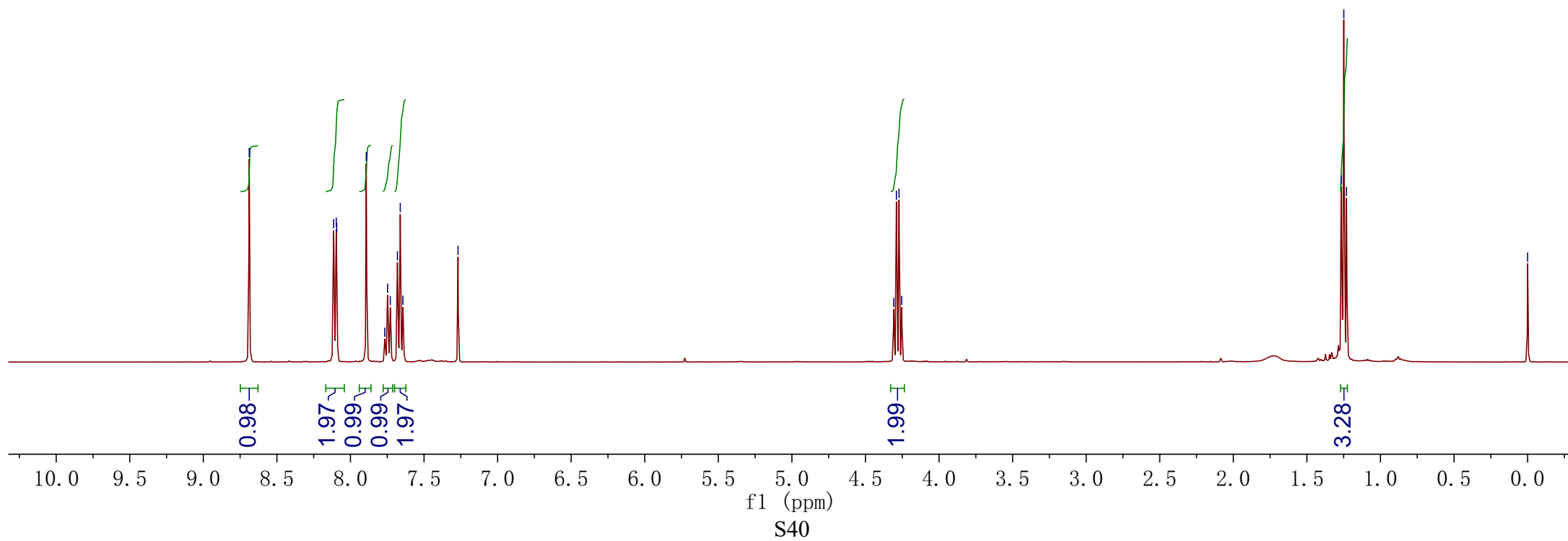


8.688
8.114
8.097
8.093
7.893
7.767
7.748
7.729
7.681
7.662
7.644
— 7.269

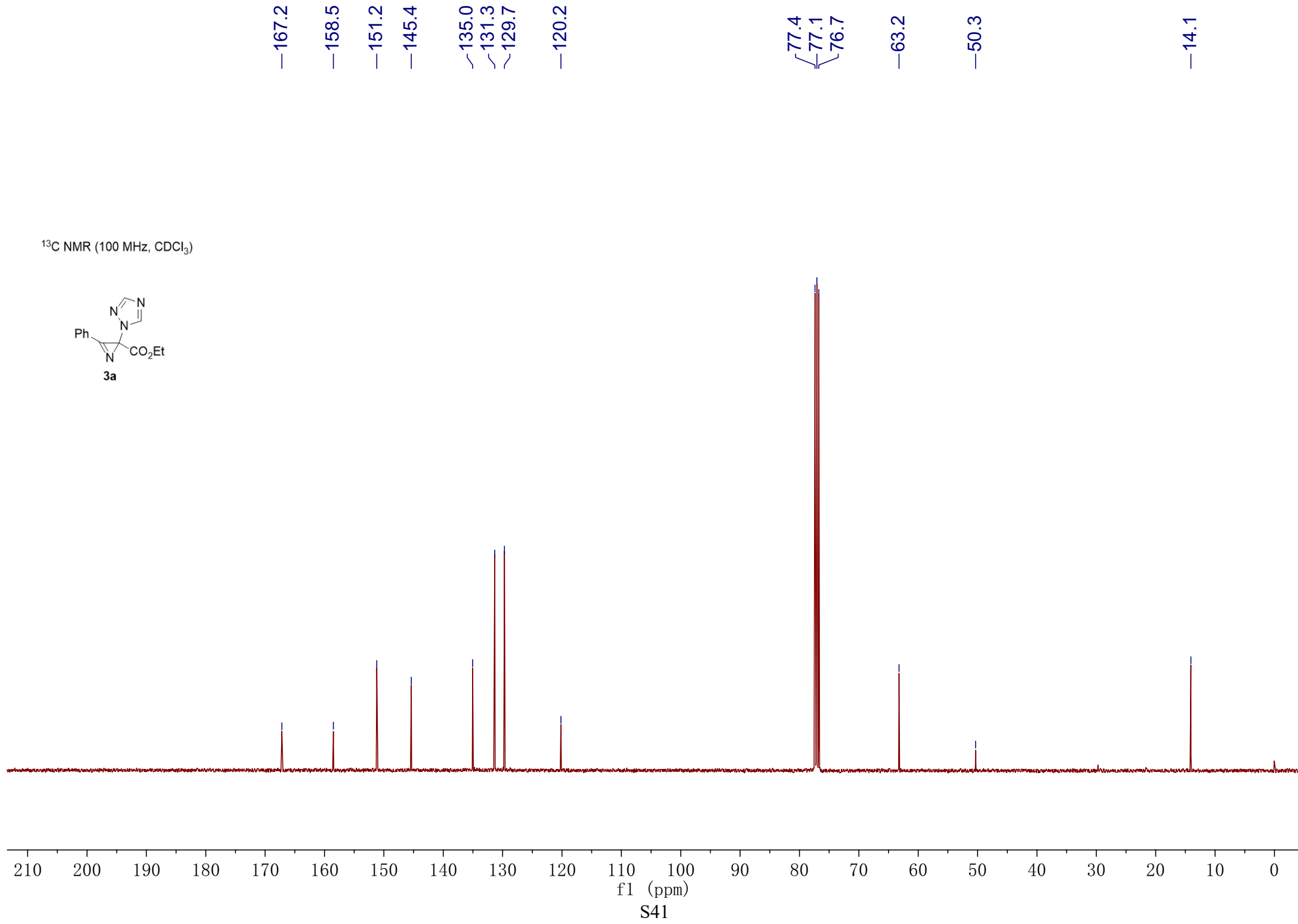
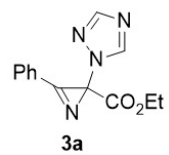
4.307
4.290
4.272
4.254

1.267
1.249
1.231

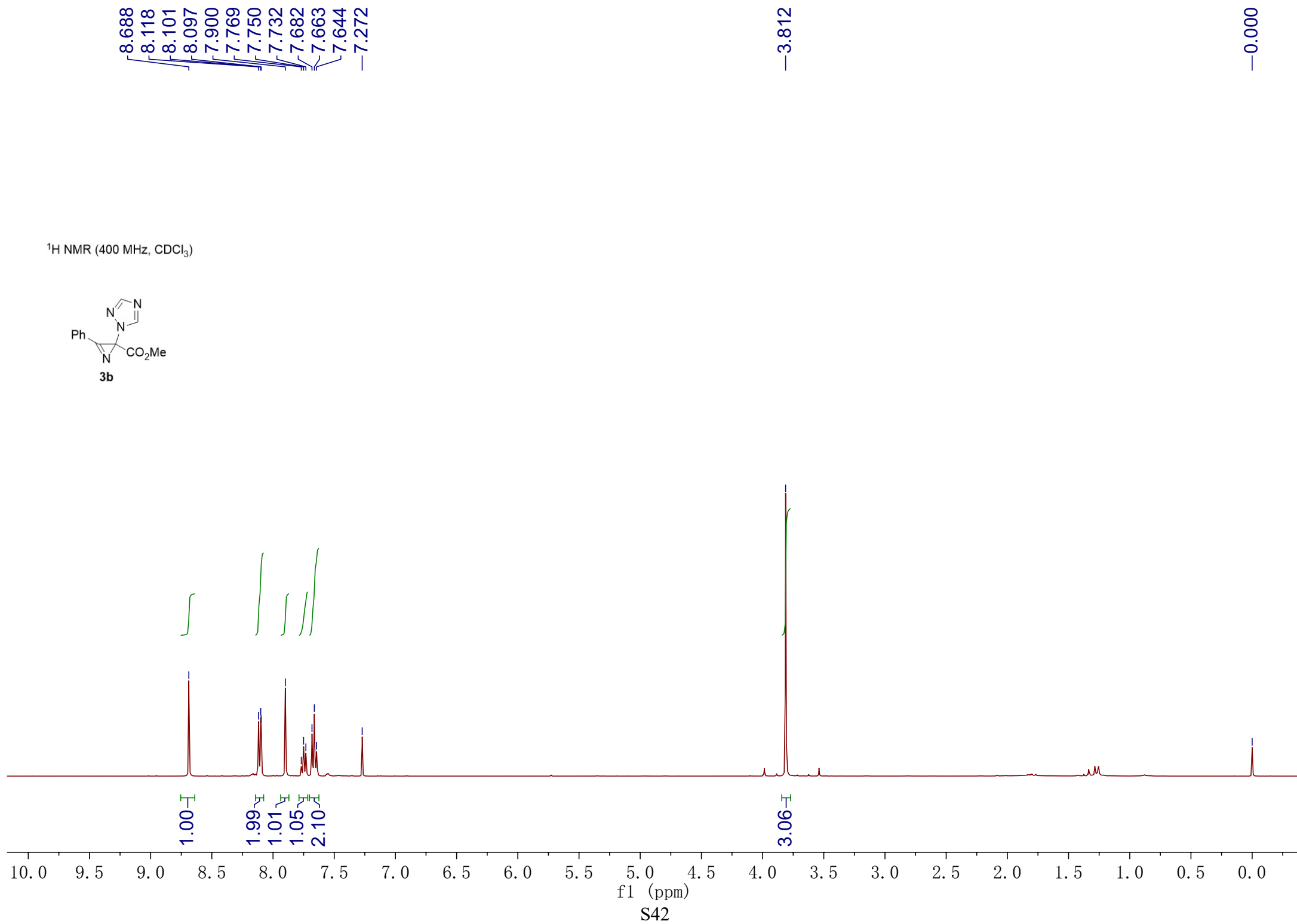
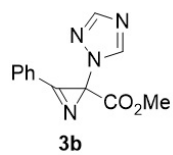
— 0.000



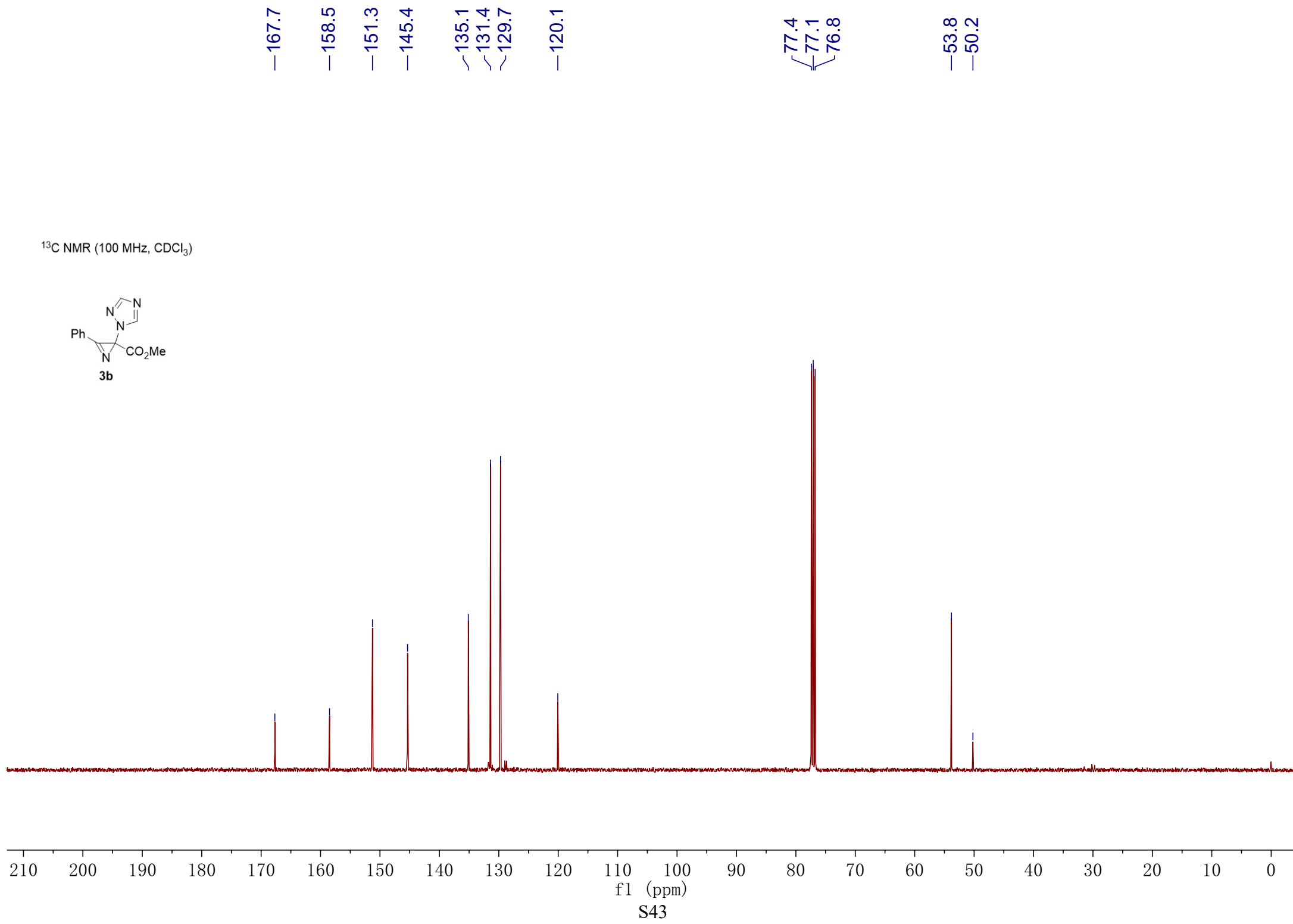
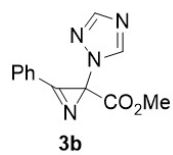
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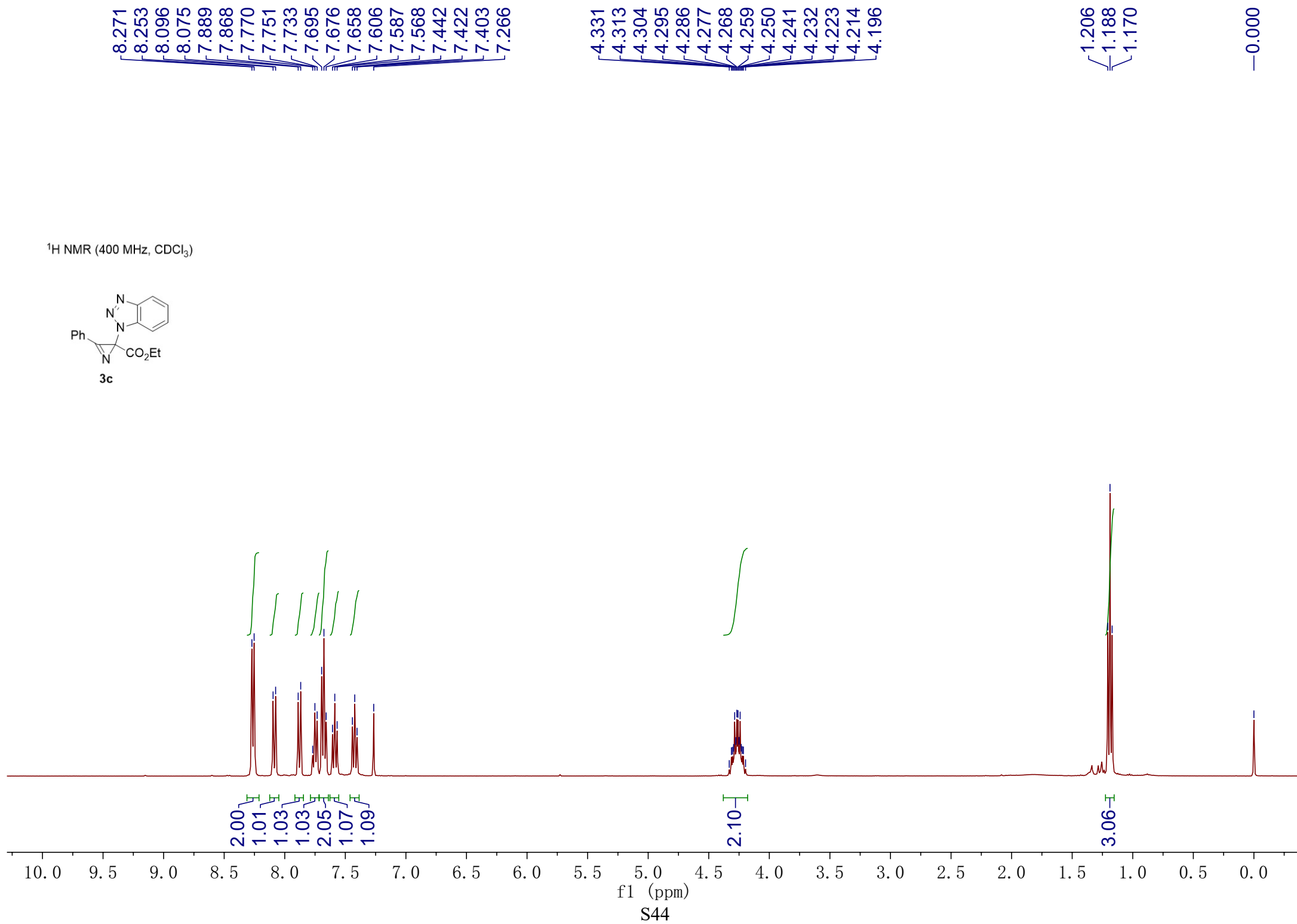
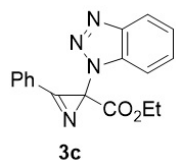
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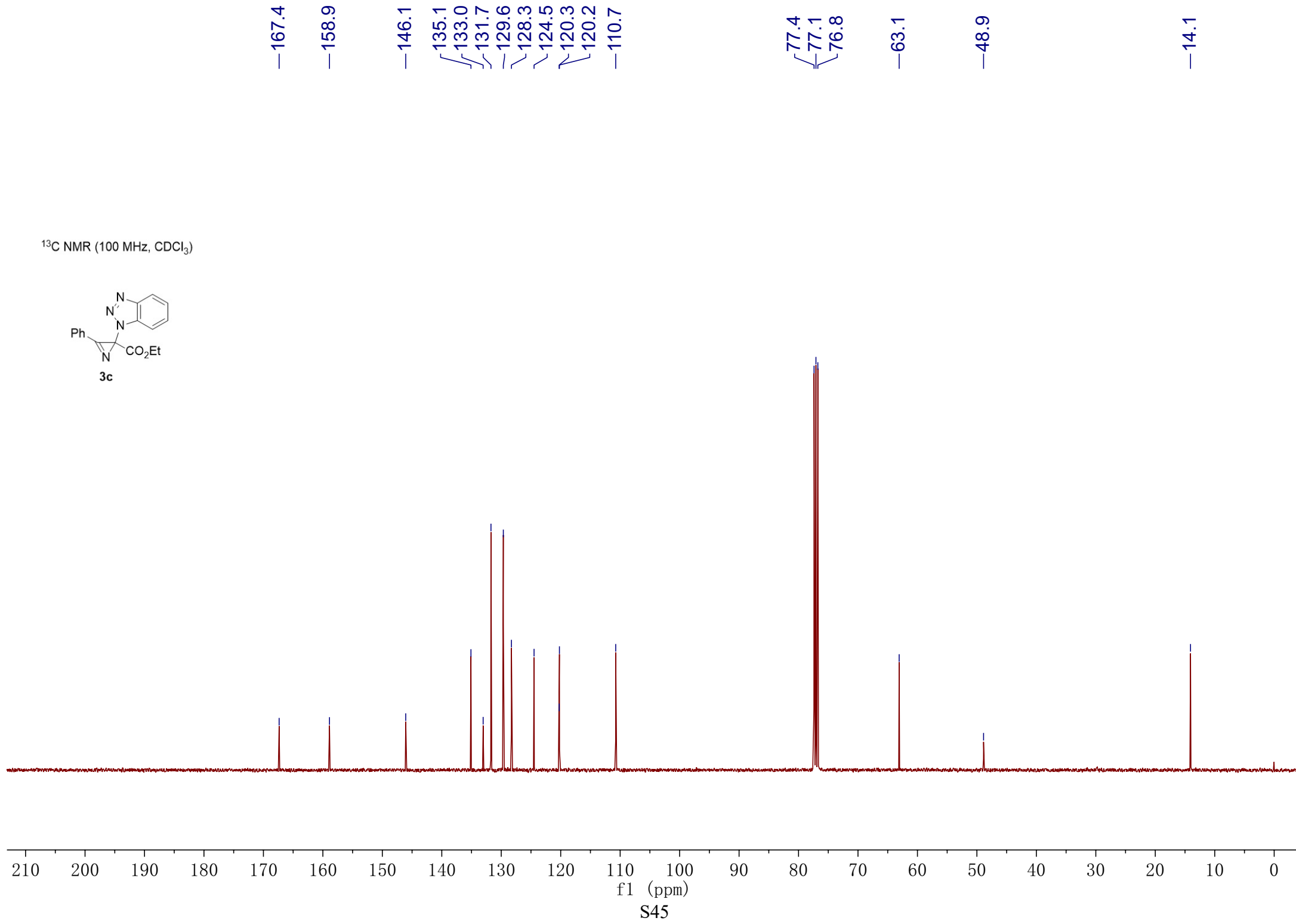
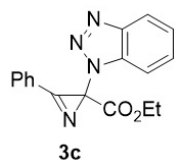
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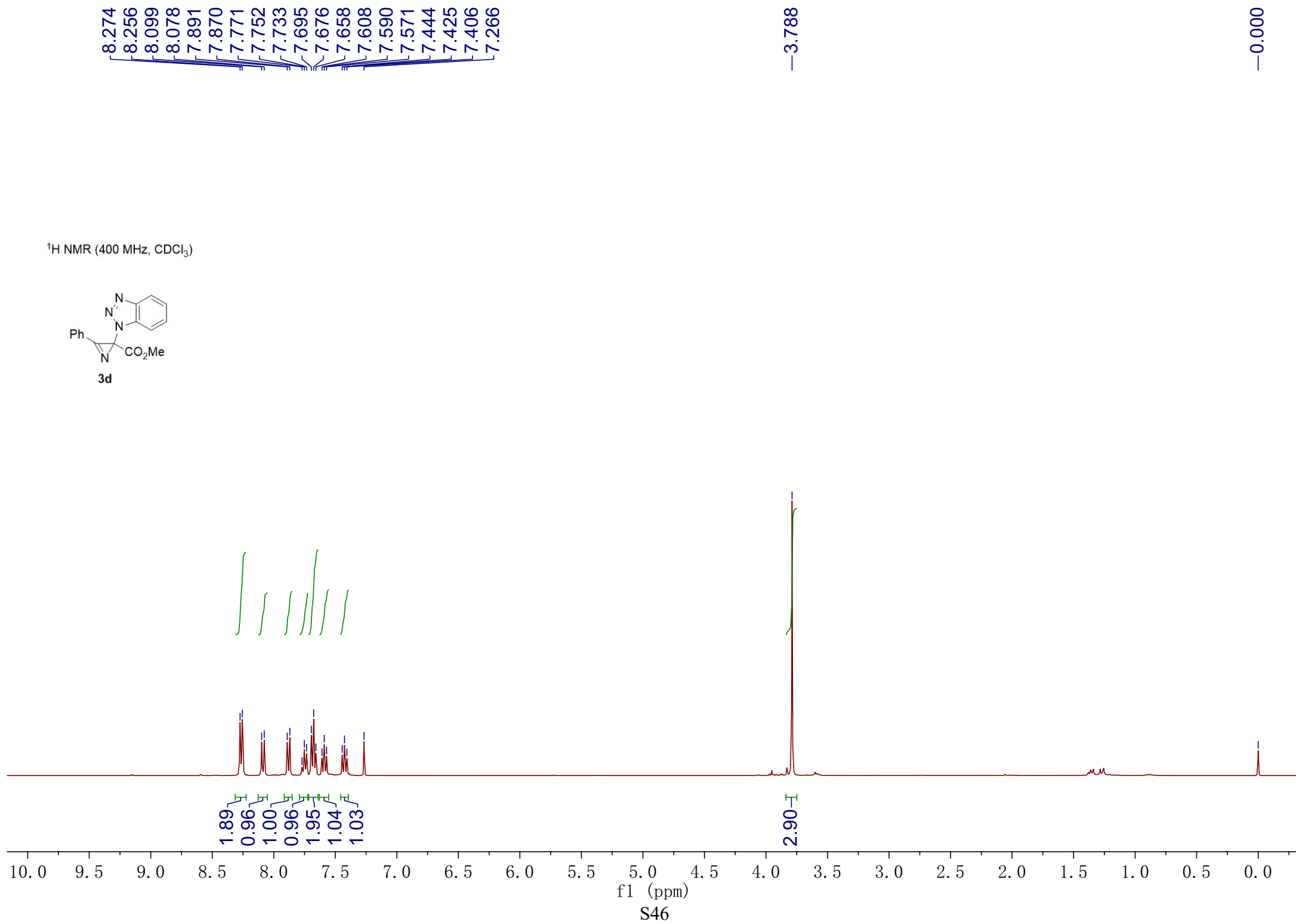
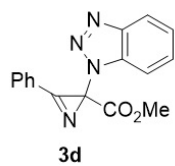
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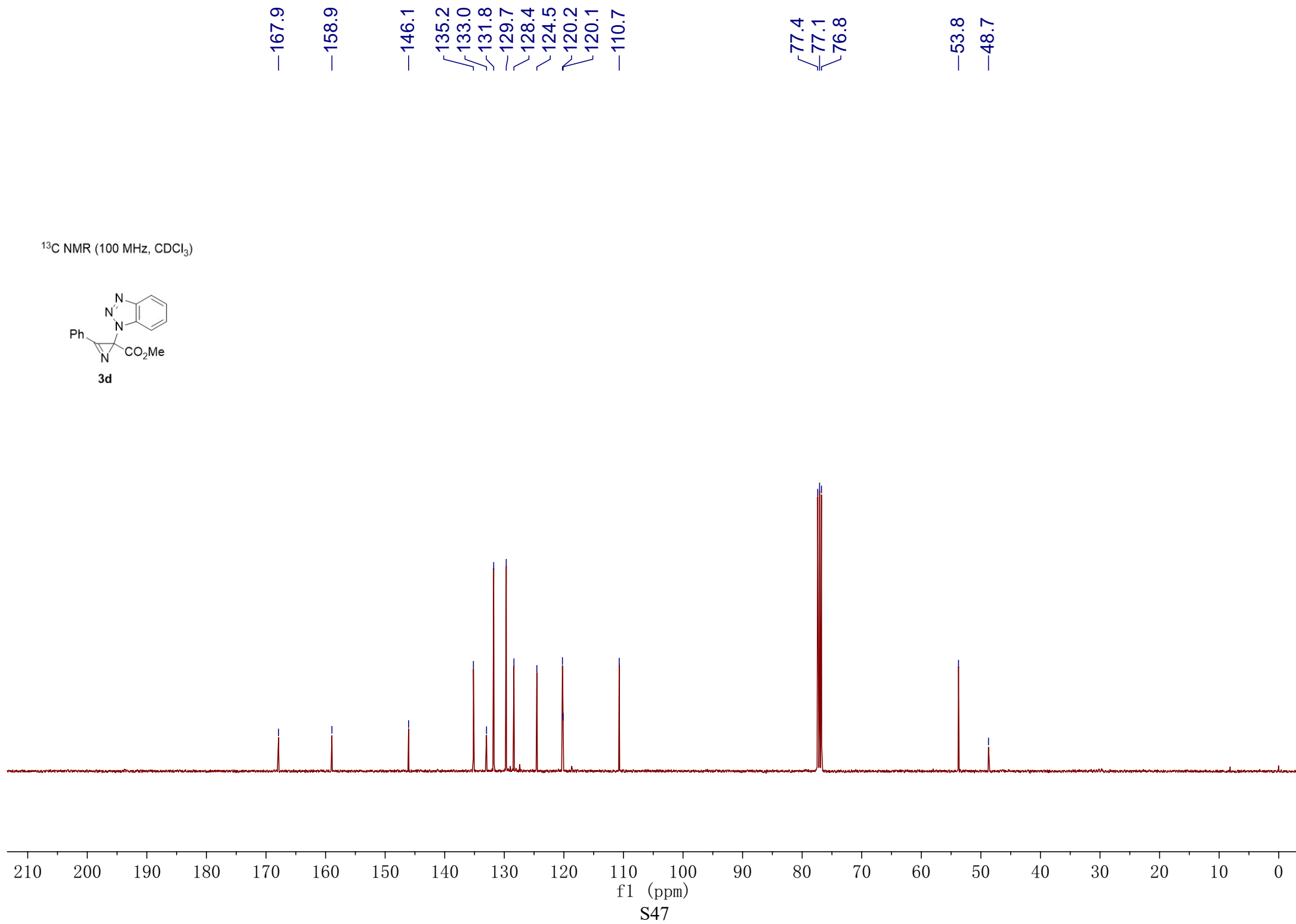
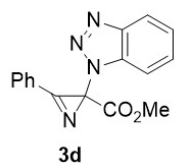
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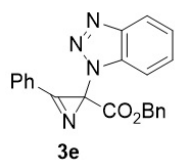


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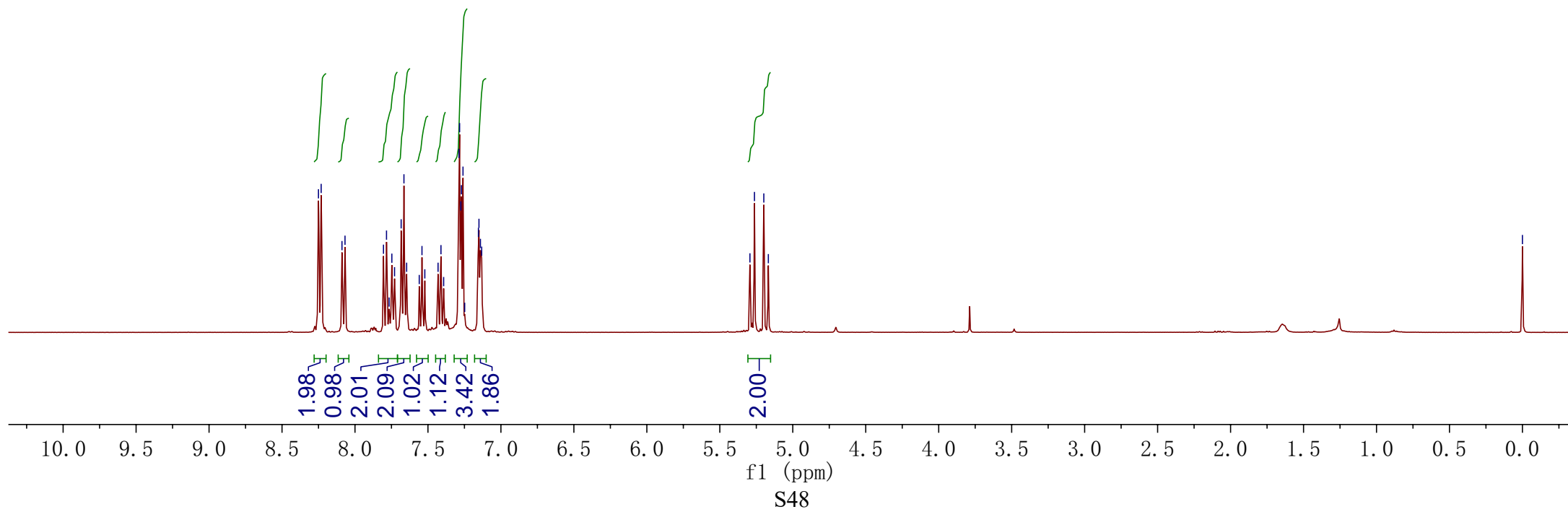


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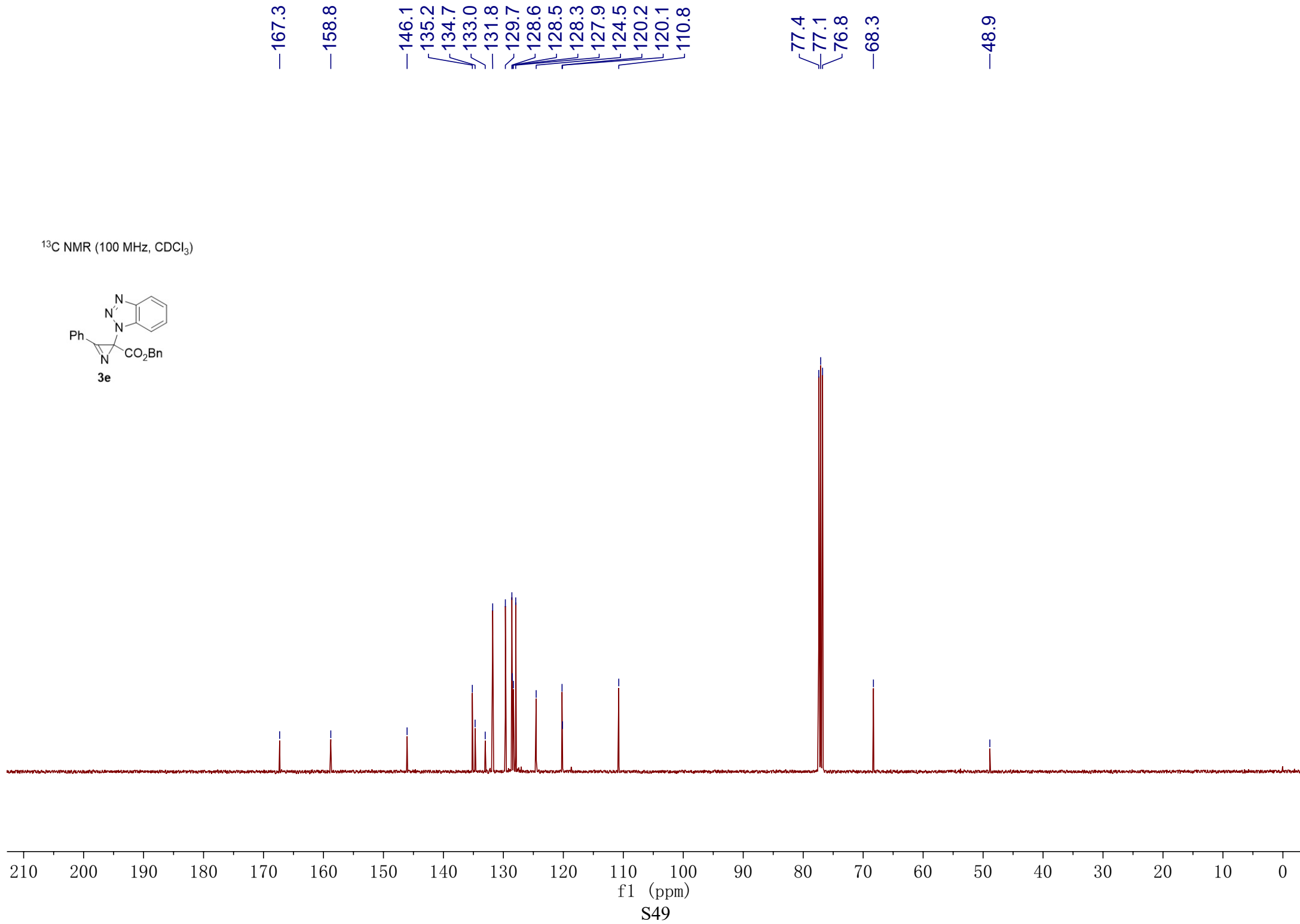
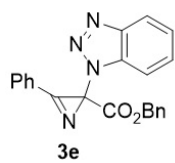


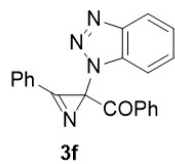
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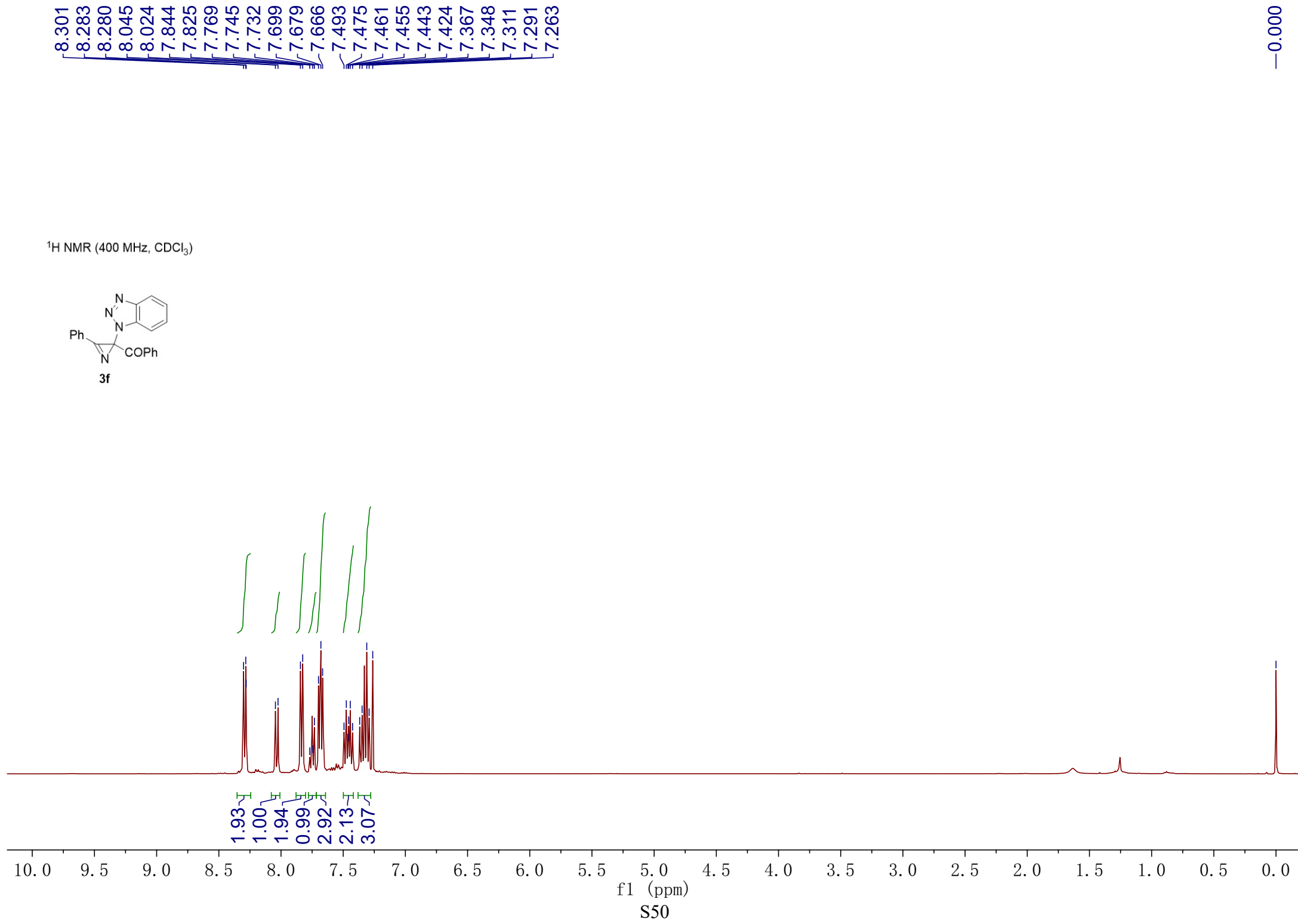
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¹³C NMR (100 MHz, CDCl₃)

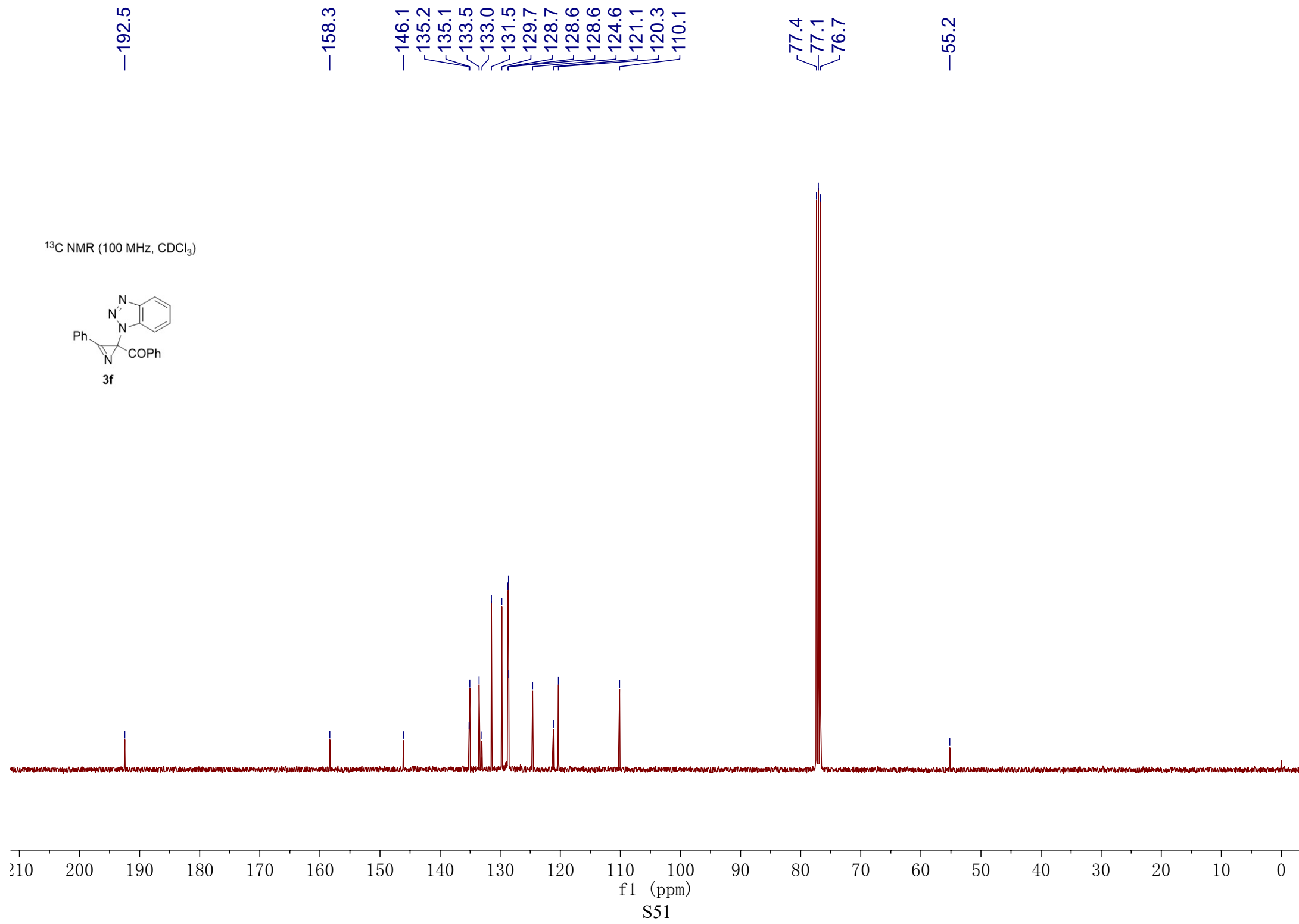
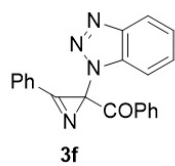




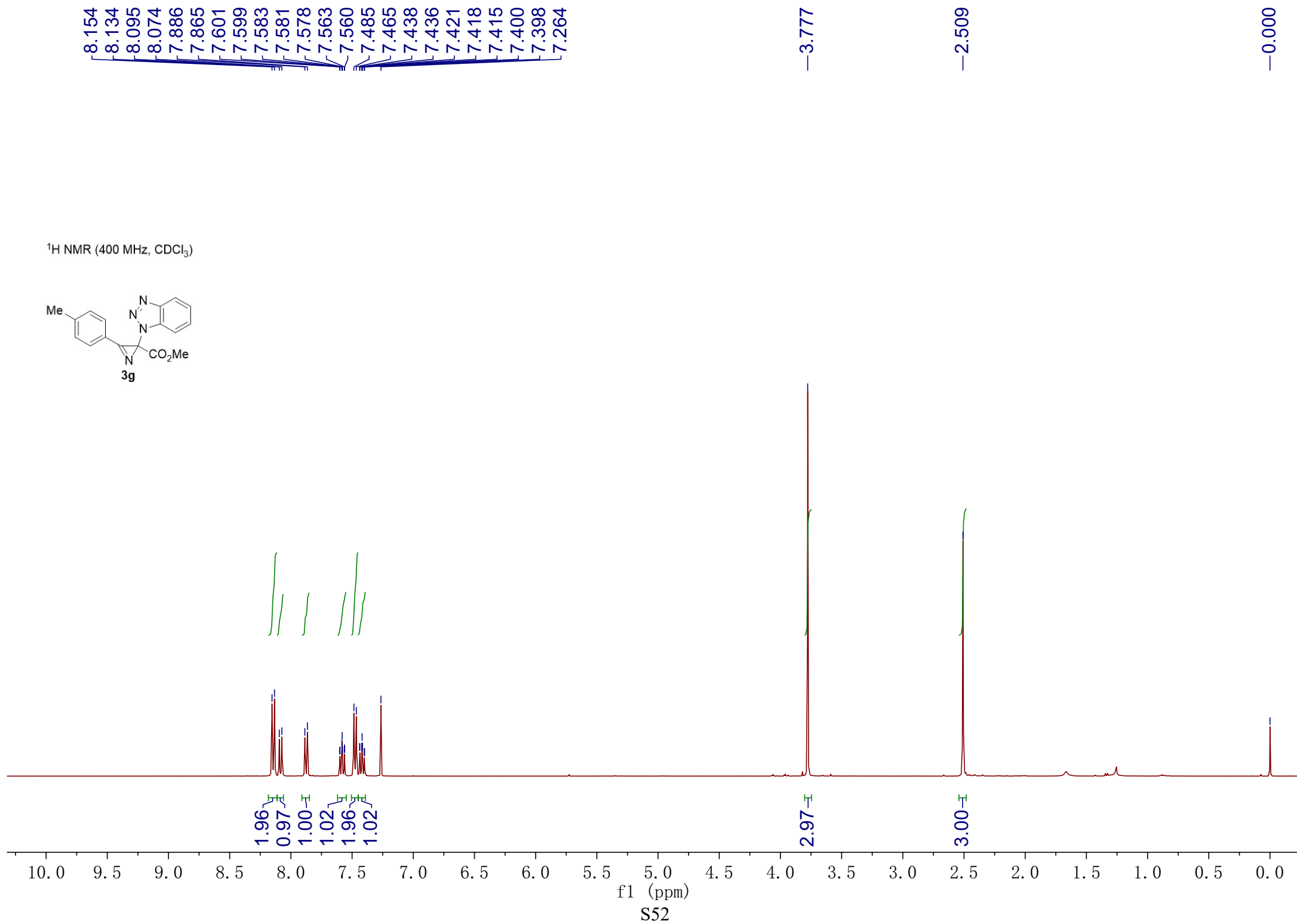
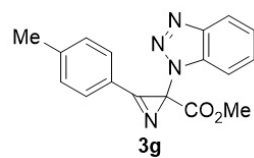
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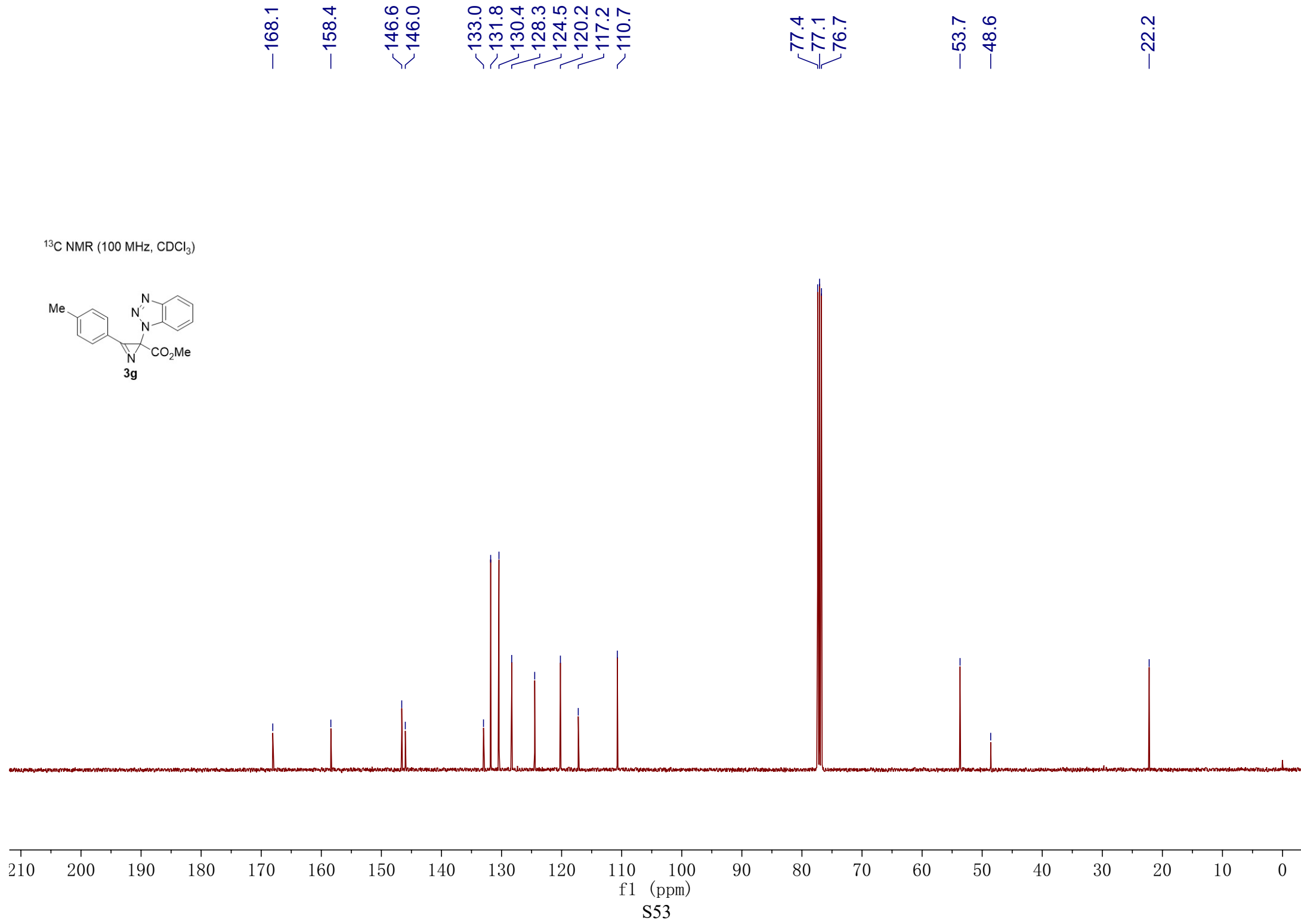
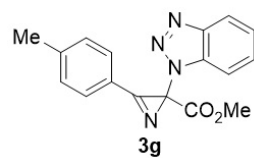
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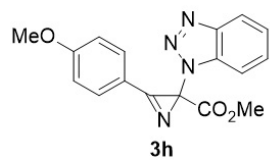
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¹³C NMR (100 MHz, CDCl₃)



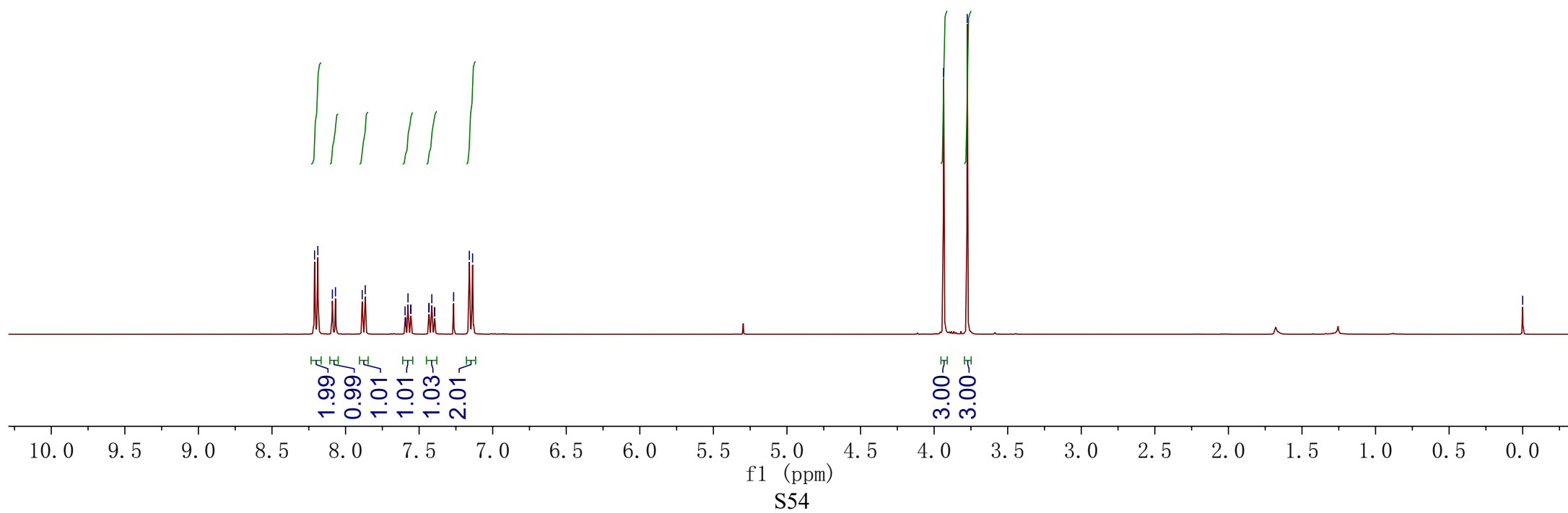
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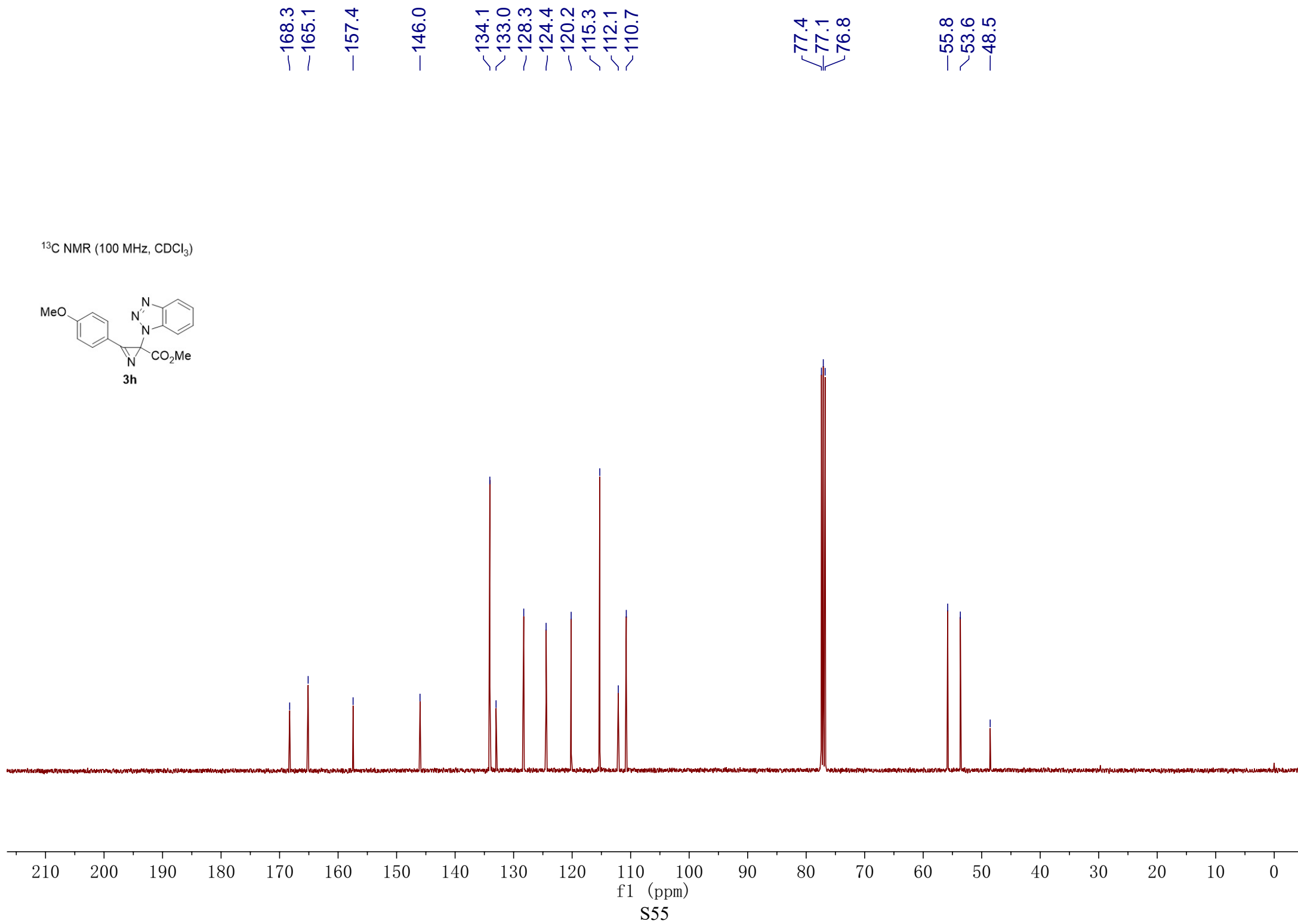
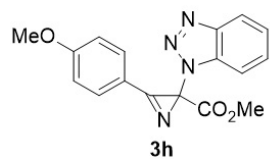
8.211
8.188
8.089
8.069
7.887
7.866
7.596
7.591
7.576
7.557
7.555
7.434
7.432
7.414
7.396
7.394
7.266
7.159
7.137

3.935
3.775

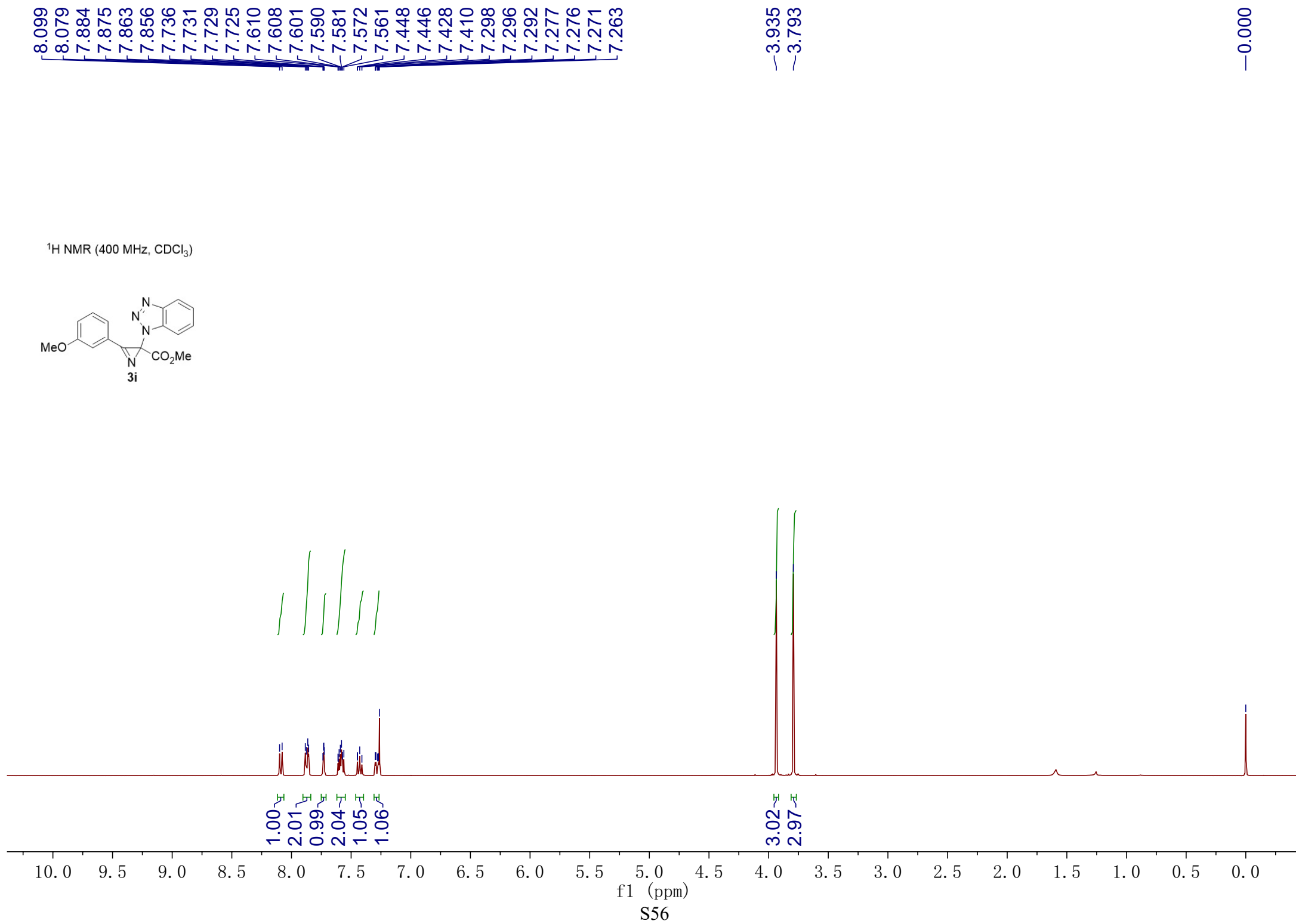
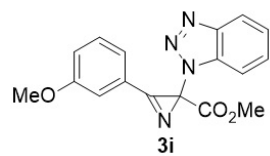
0.000



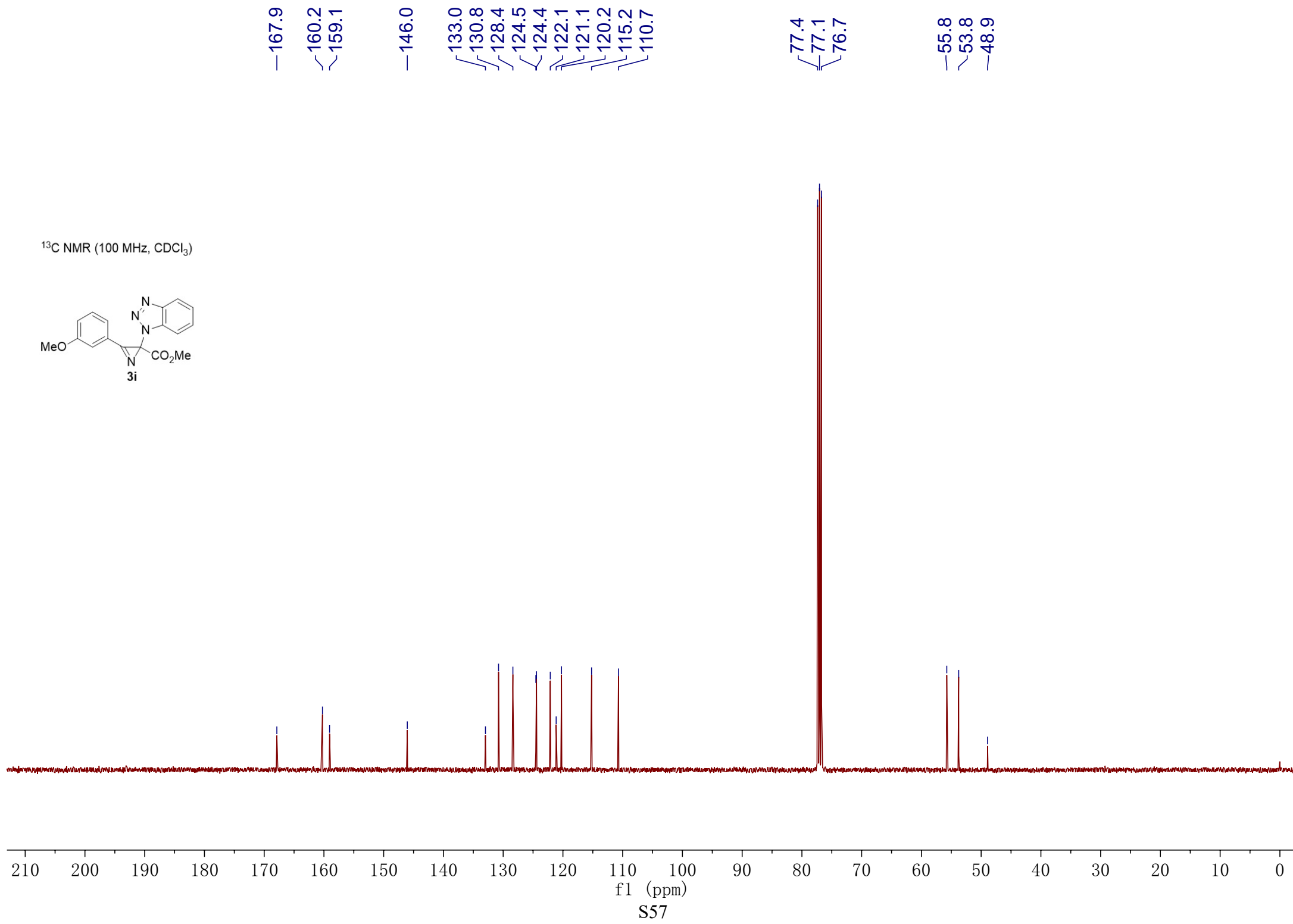
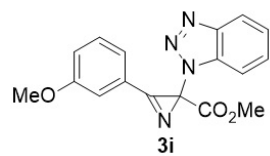
¹³C NMR (100 MHz, CDCl₃)



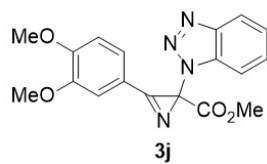
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¹³C NMR (100 MHz, CDCl₃)



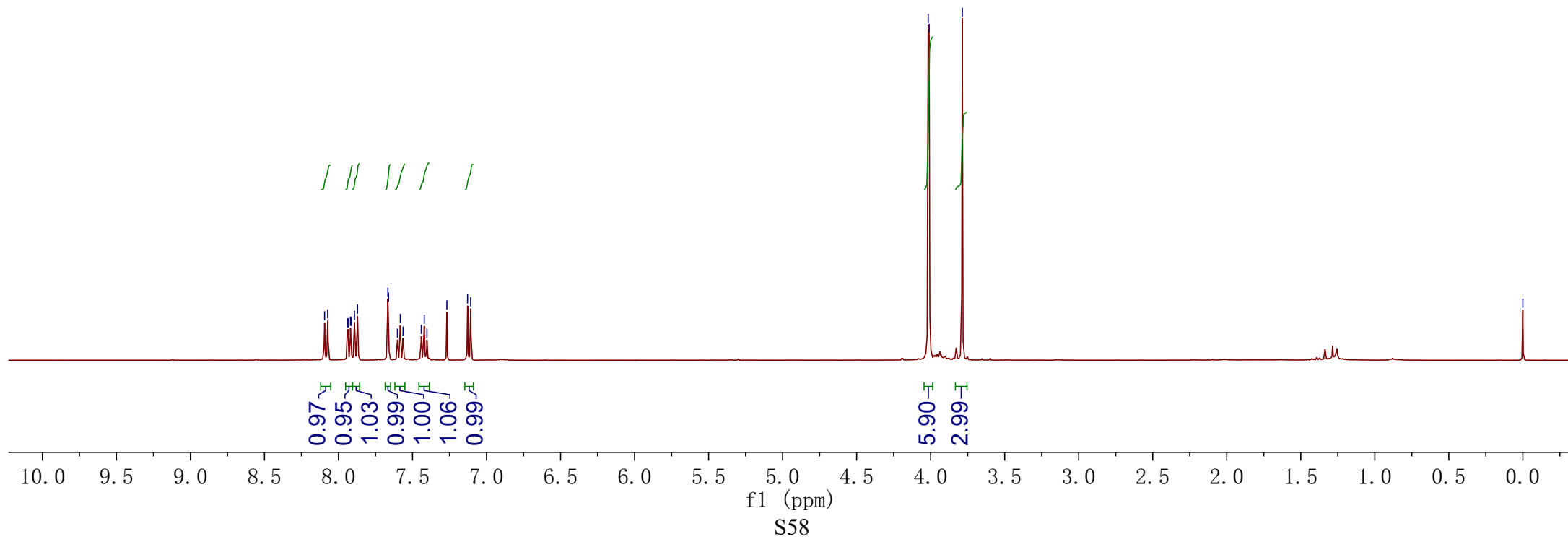
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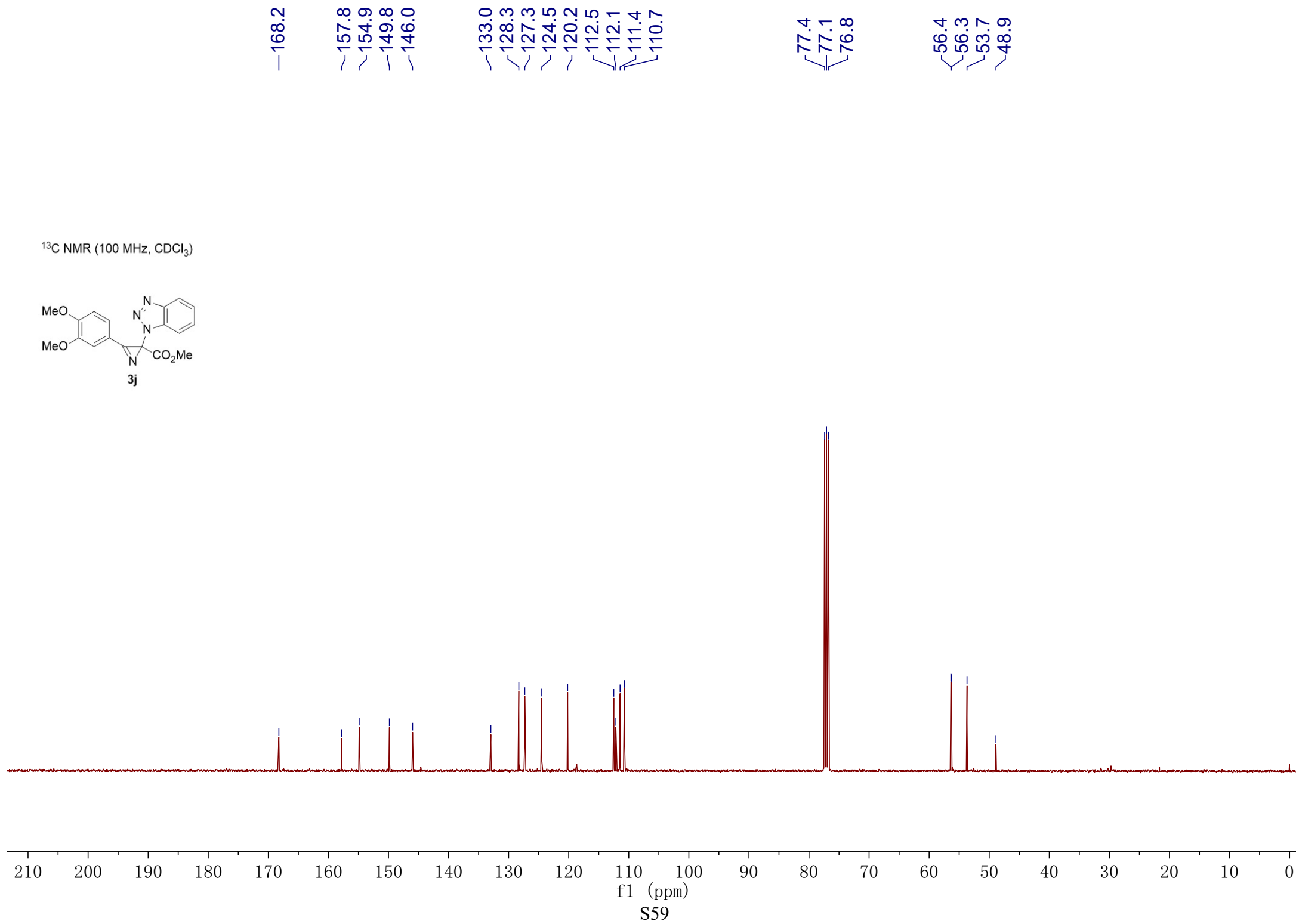
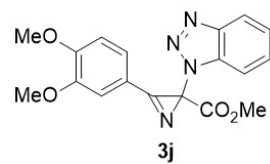
8.094
8.073
7.940
7.935
7.919
7.914
7.892
7.871
7.666
7.662
7.602
7.583
7.564
7.441
7.421
7.403
7.268
7.128
7.107

4.017
4.010
3.786

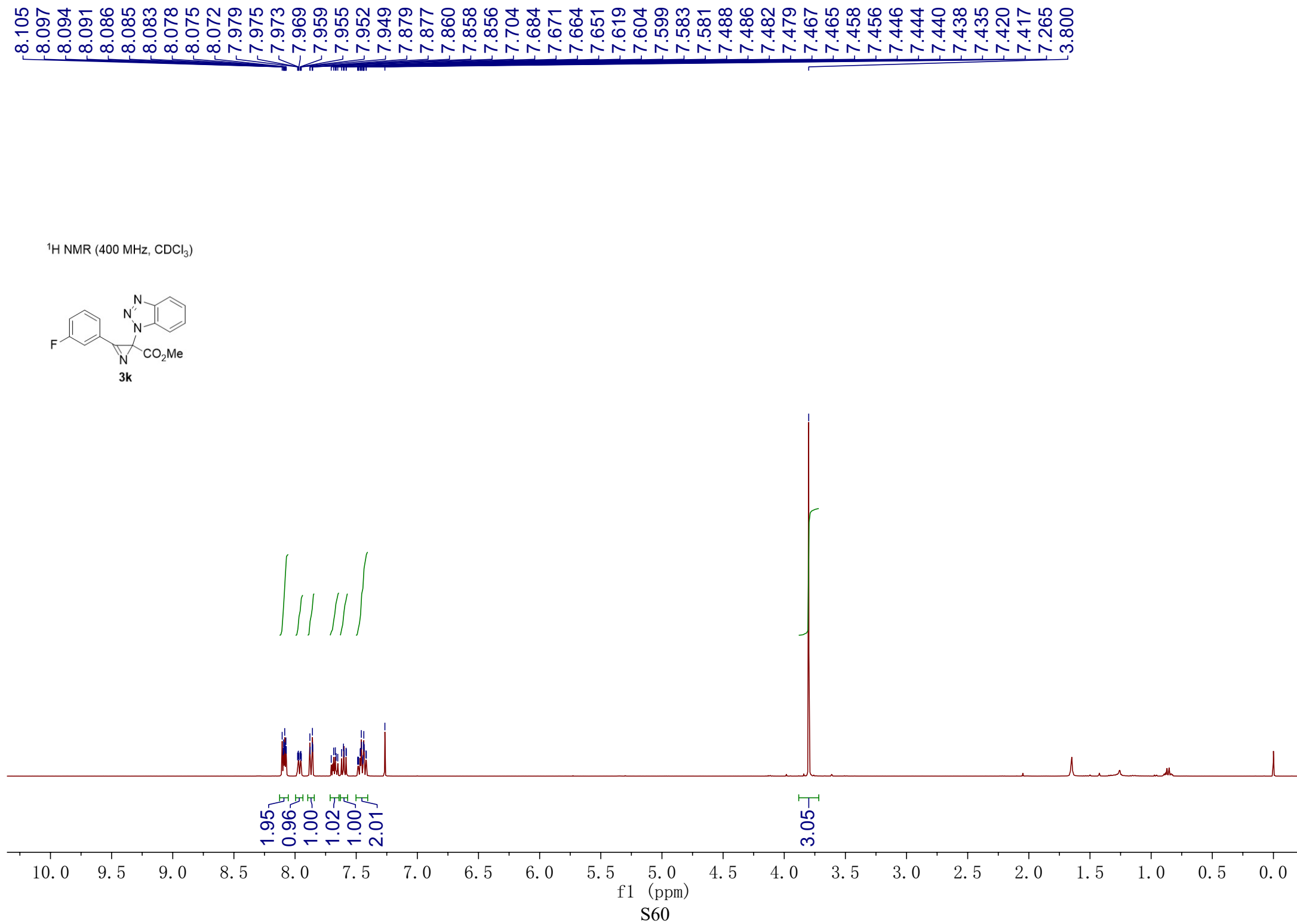
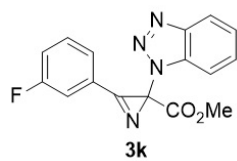
0.000



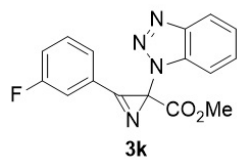
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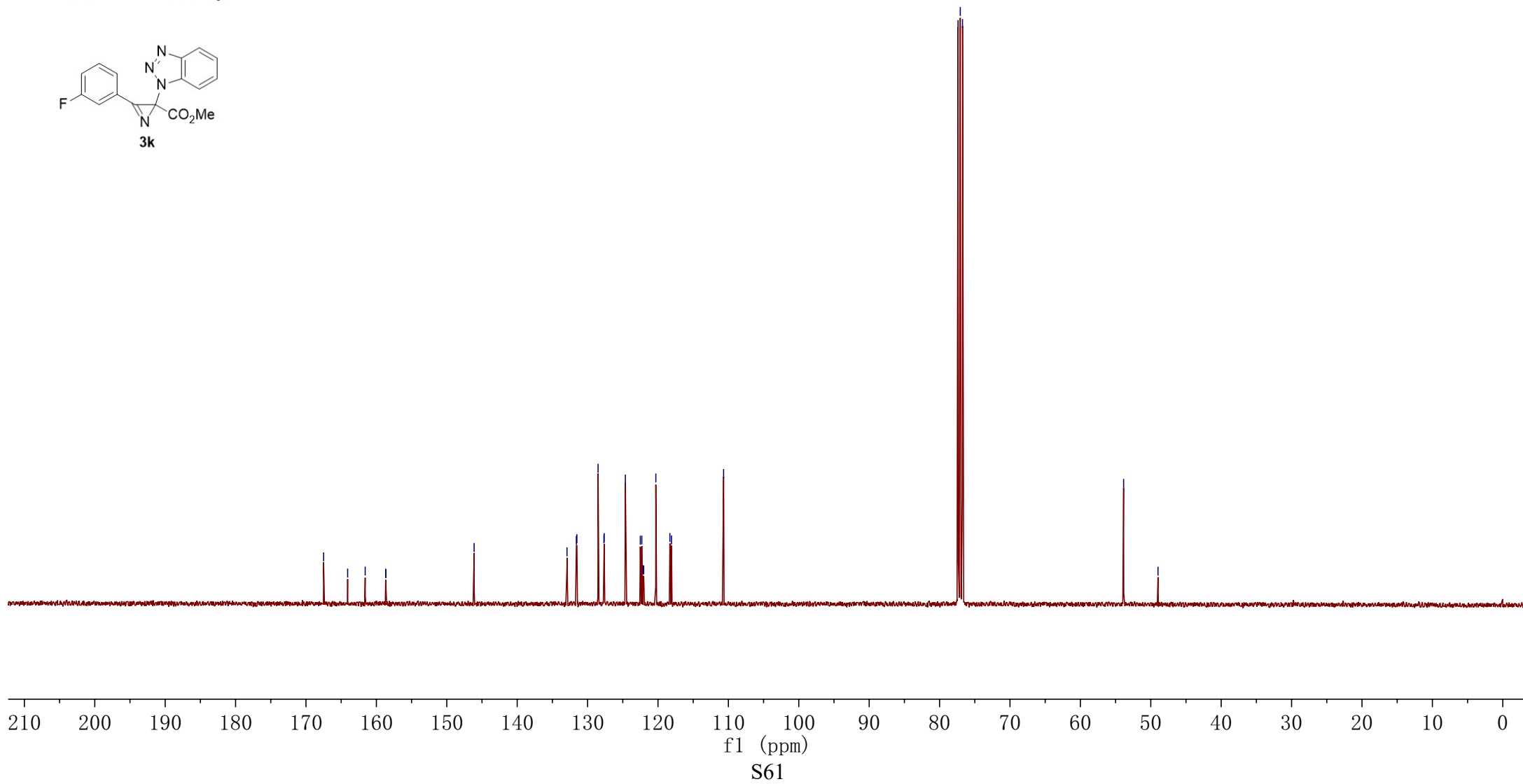
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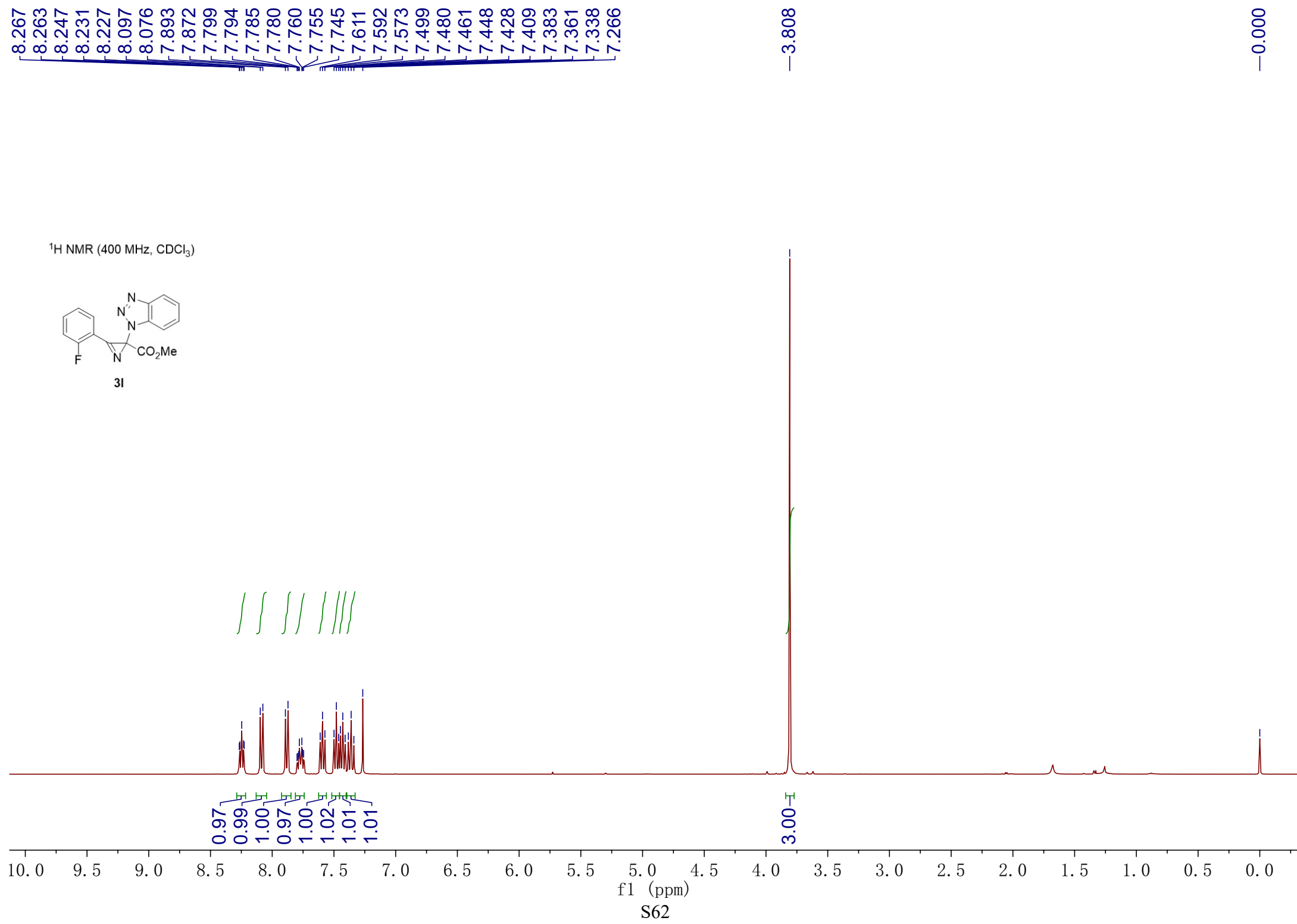


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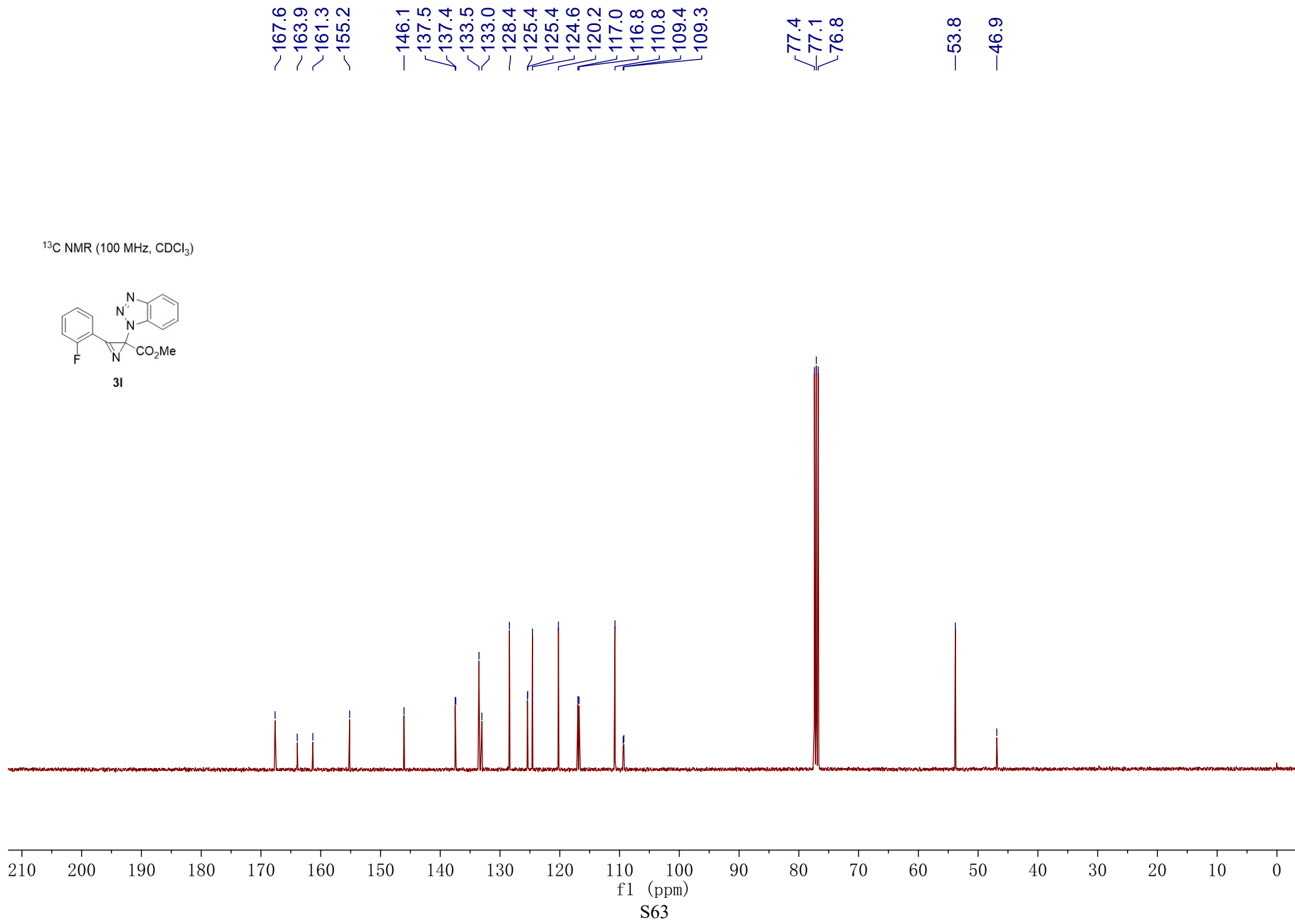
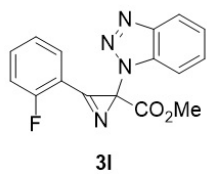


167.5
164.1
161.6
158.7
158.6
— 146.1
132.9
131.6
131.5
128.5
127.7
127.6
124.6
122.5
122.3
122.1
122.0
120.3
118.3
118.1
110.7
77.4
77.1
76.7
— 53.9
— 49.0

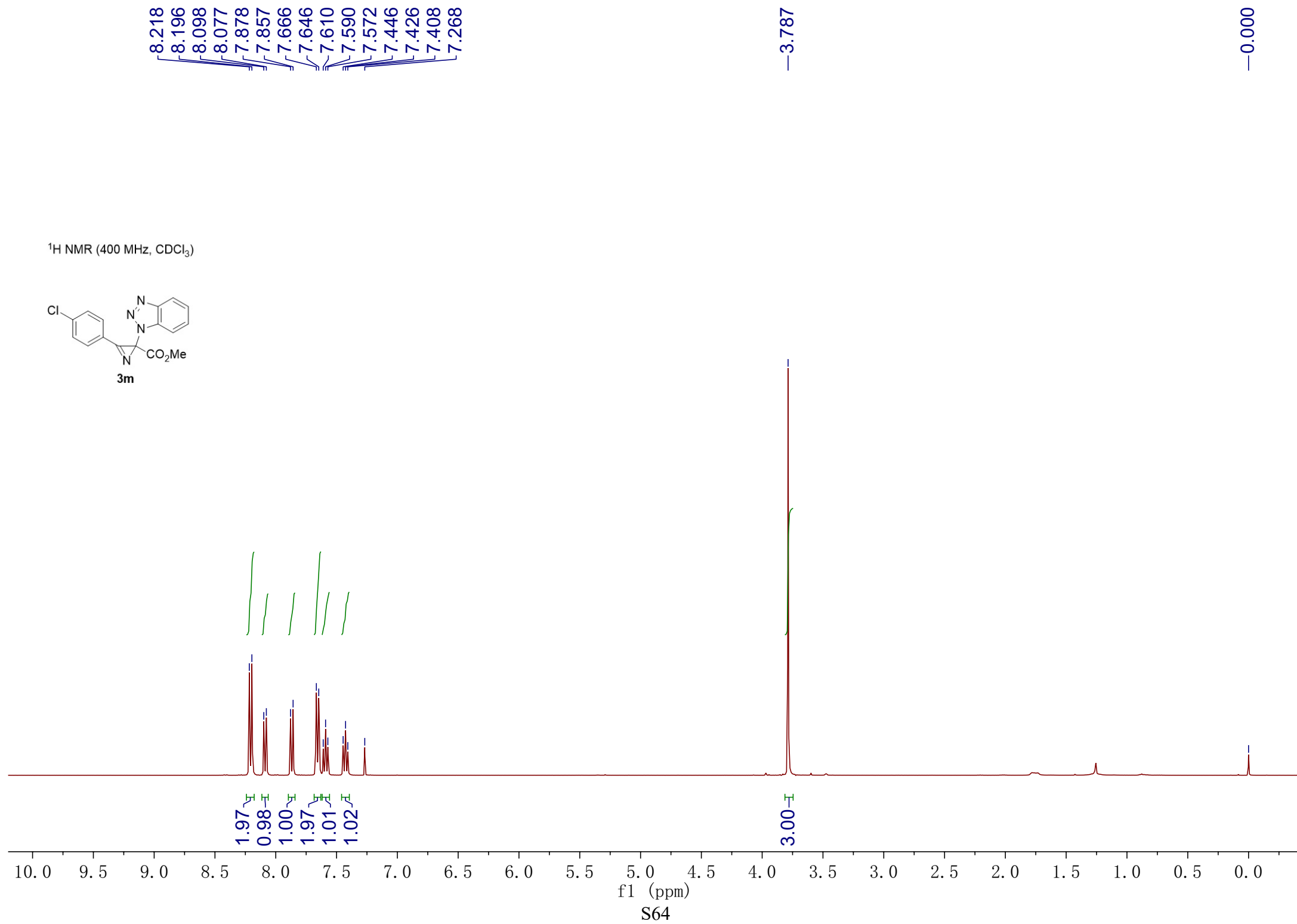
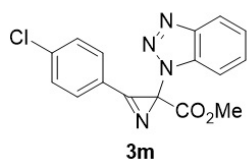




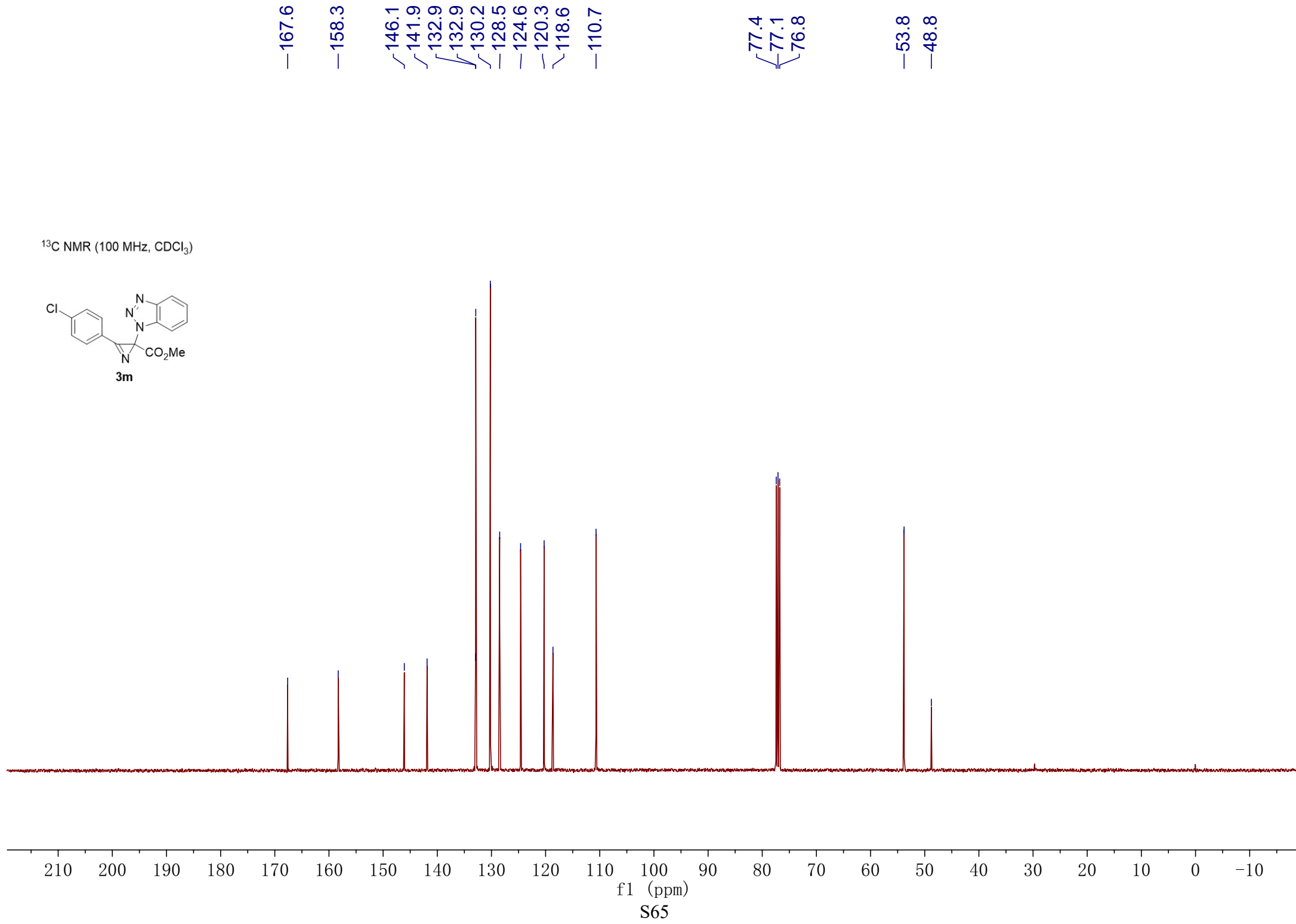
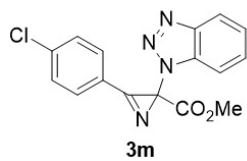
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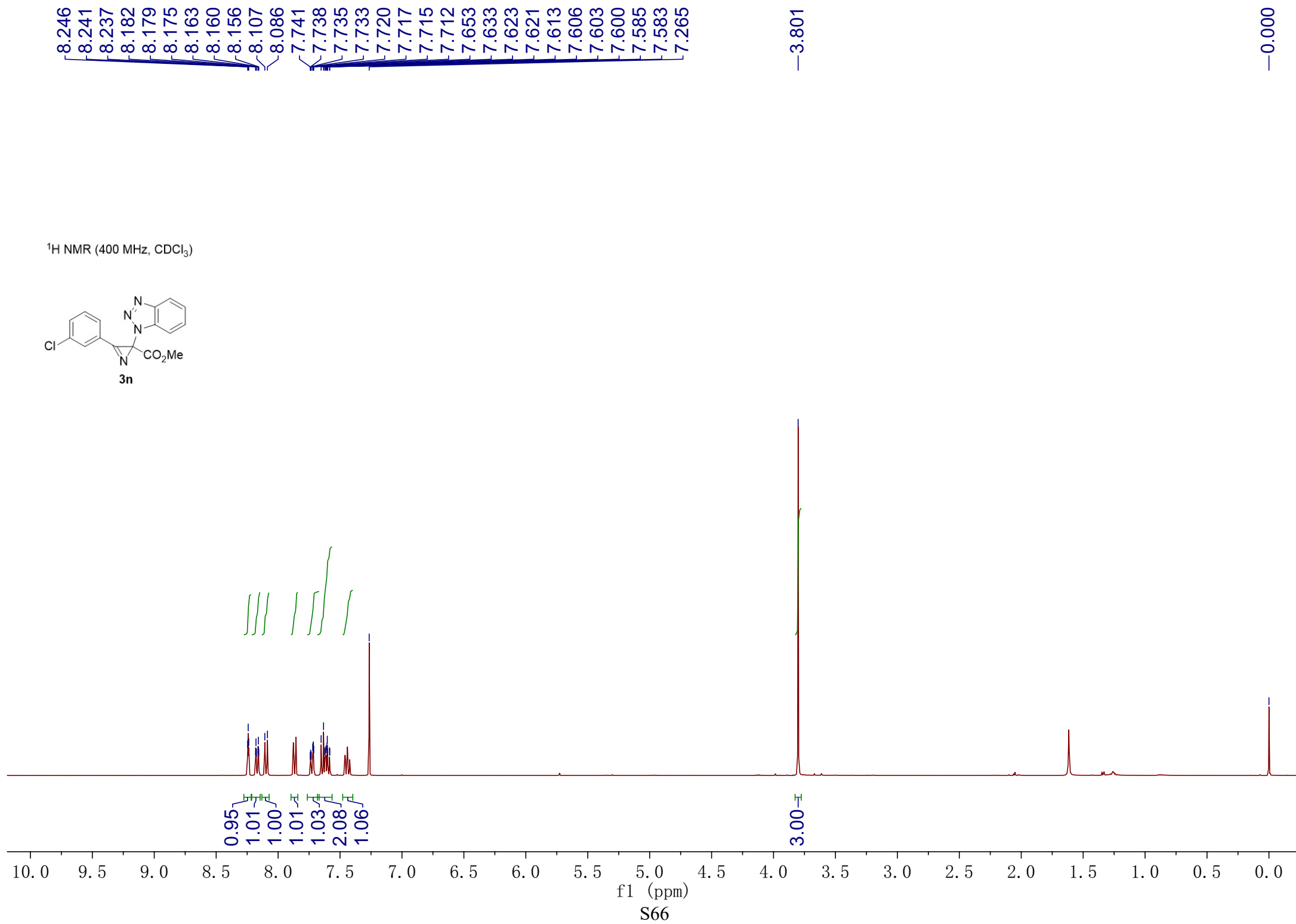
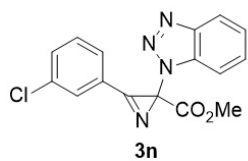
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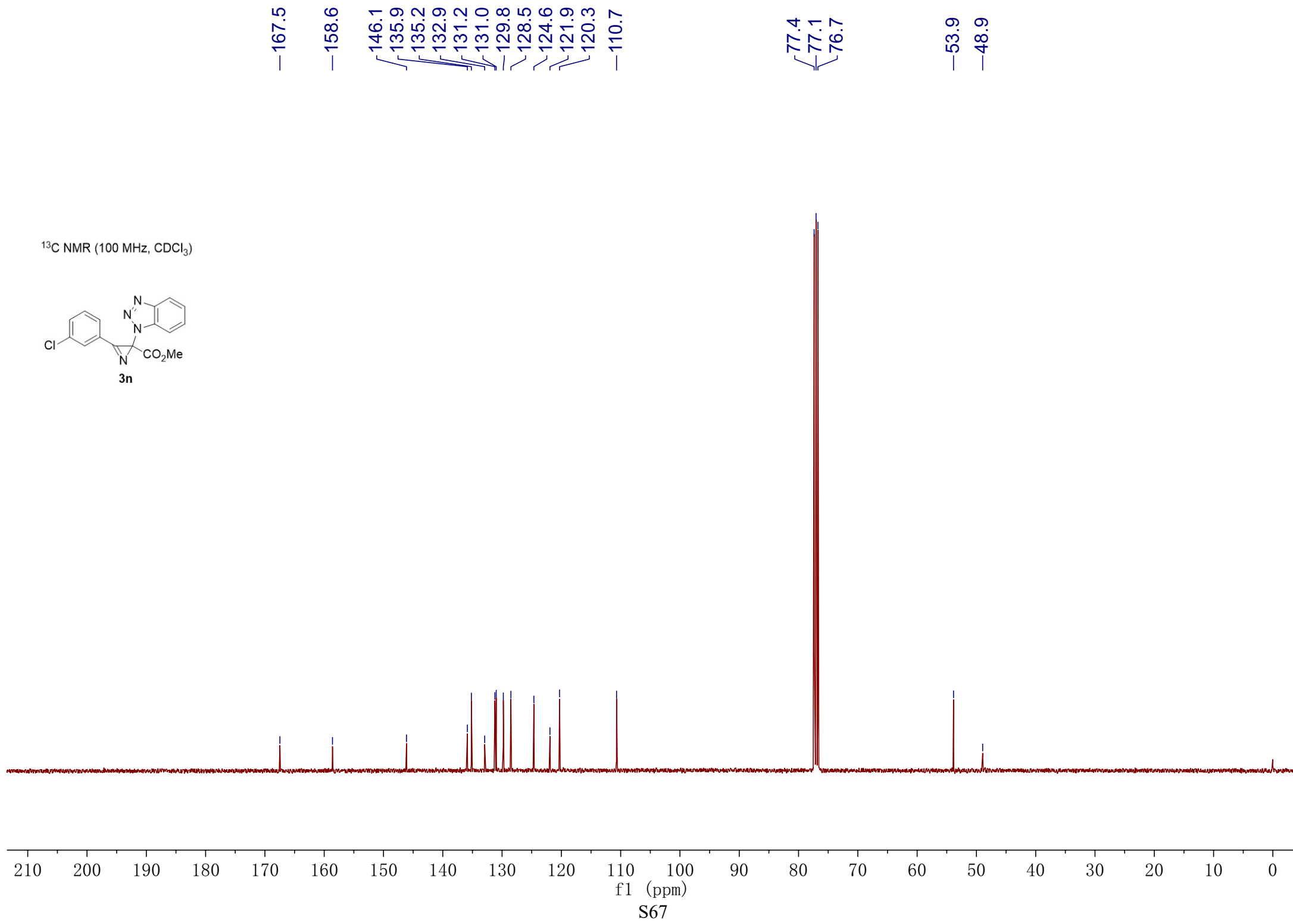
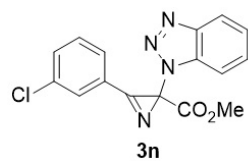
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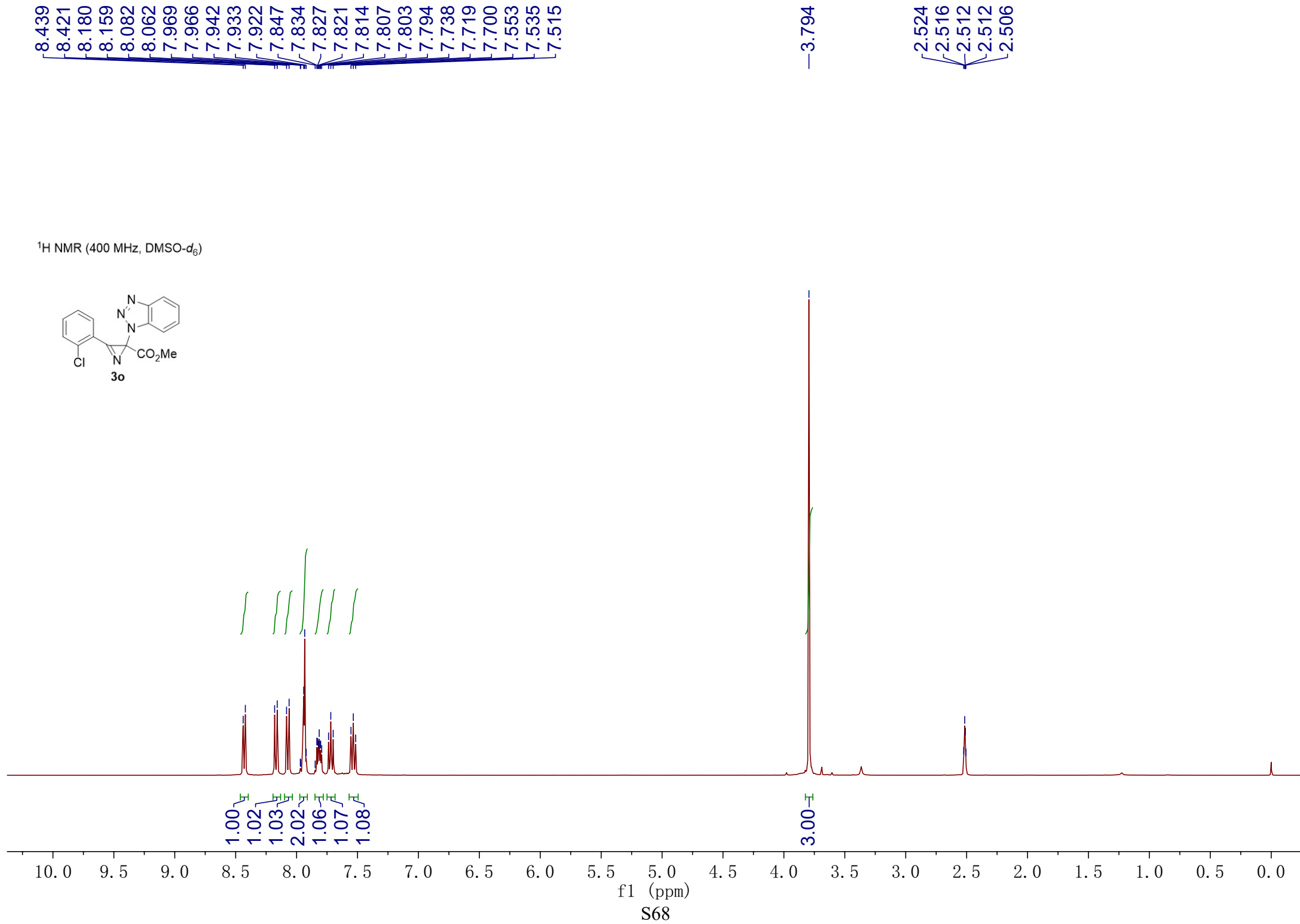
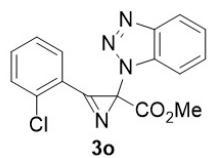
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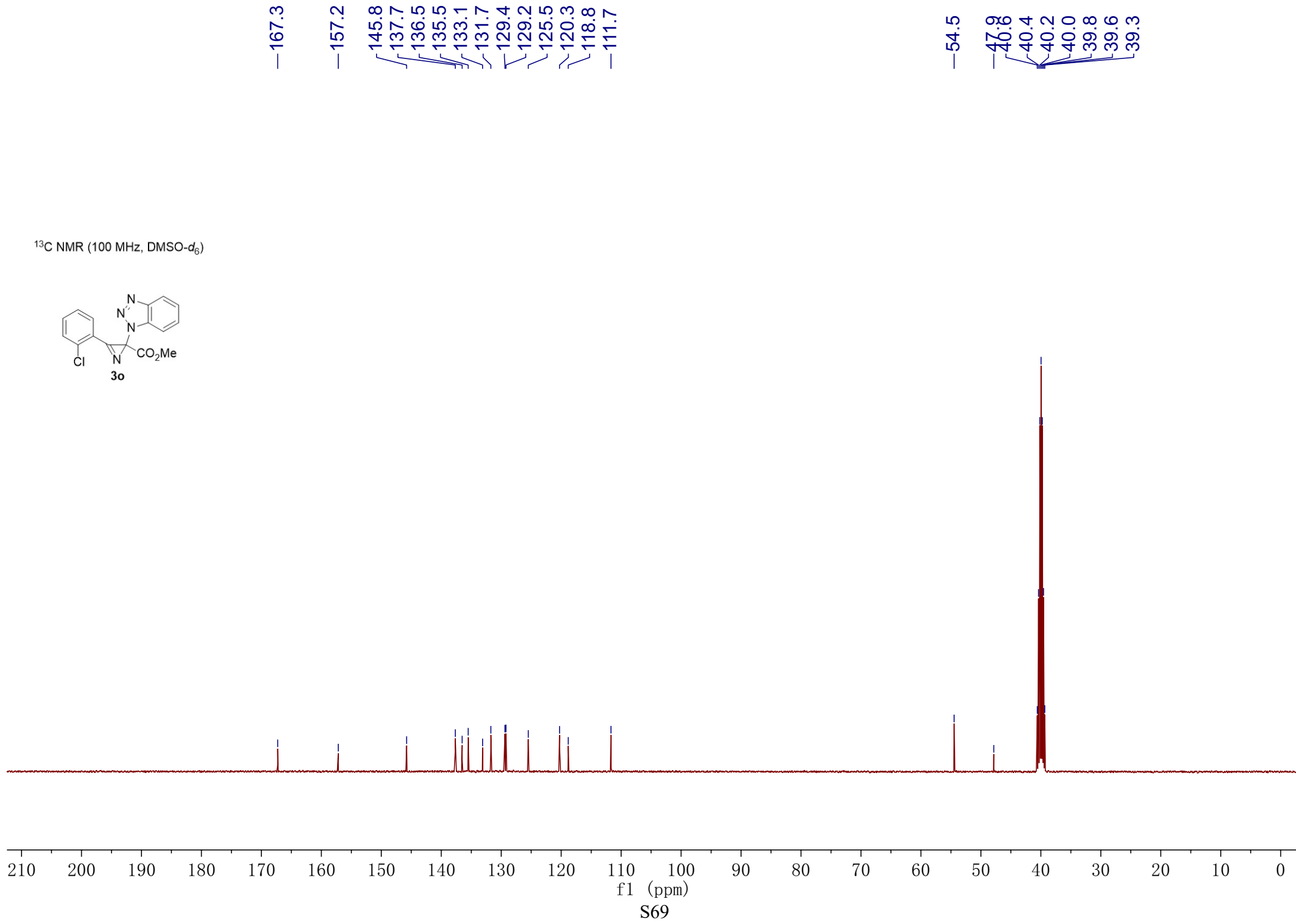
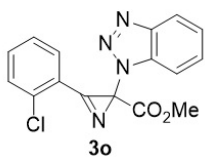
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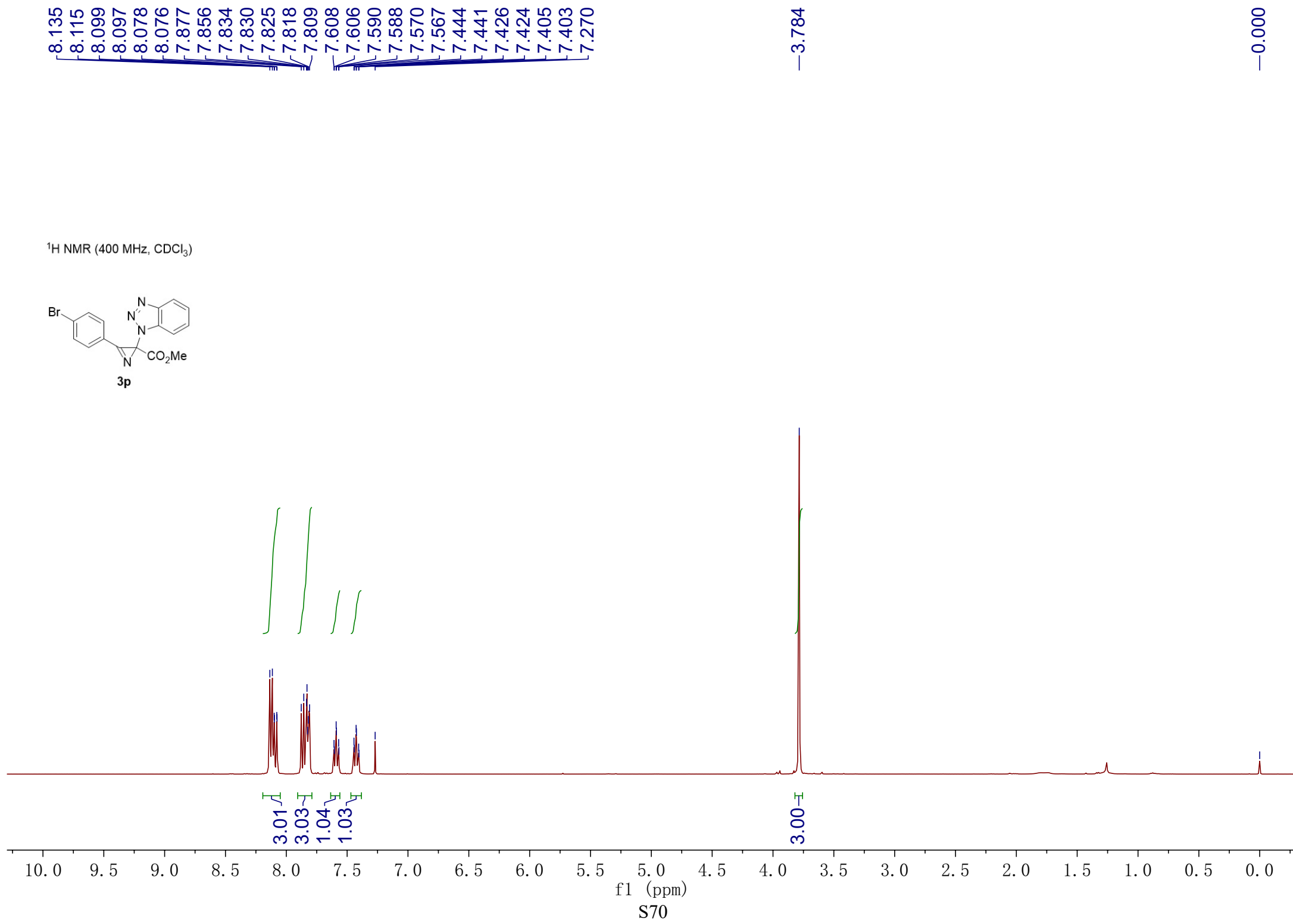
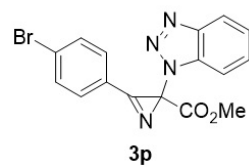
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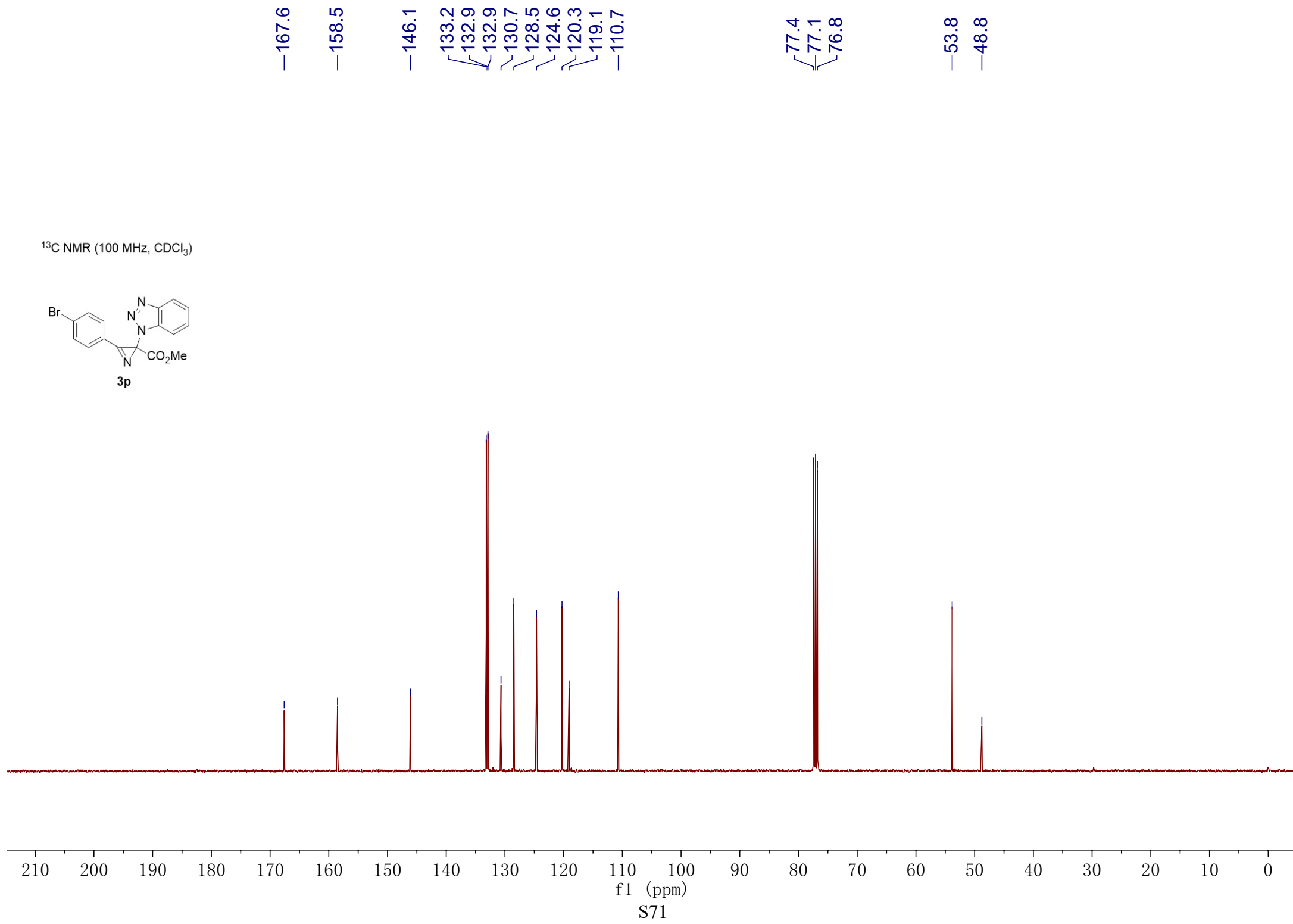
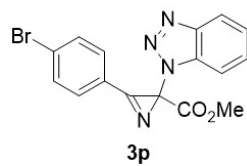
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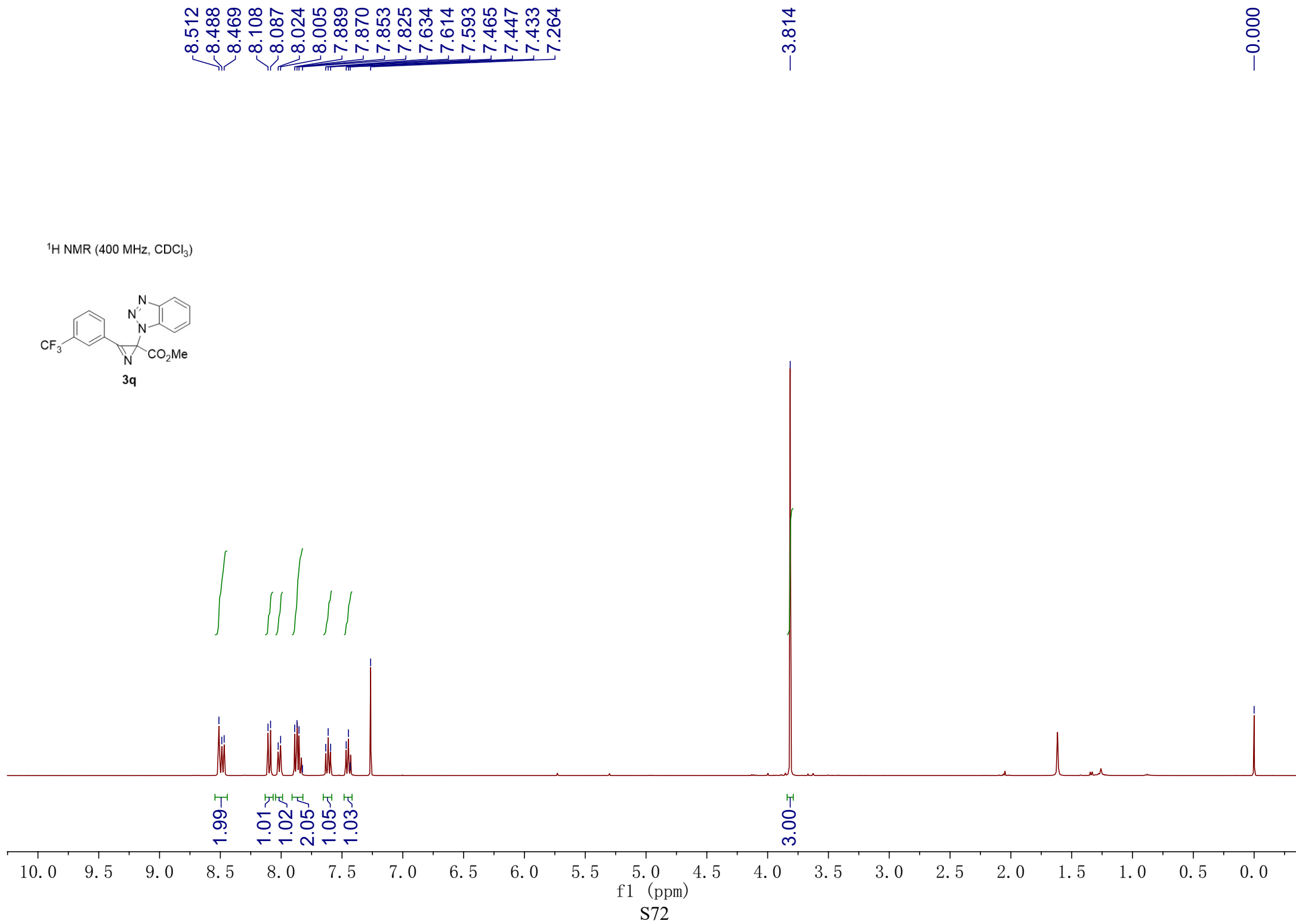
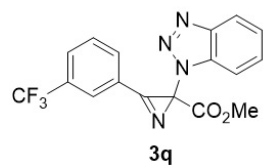
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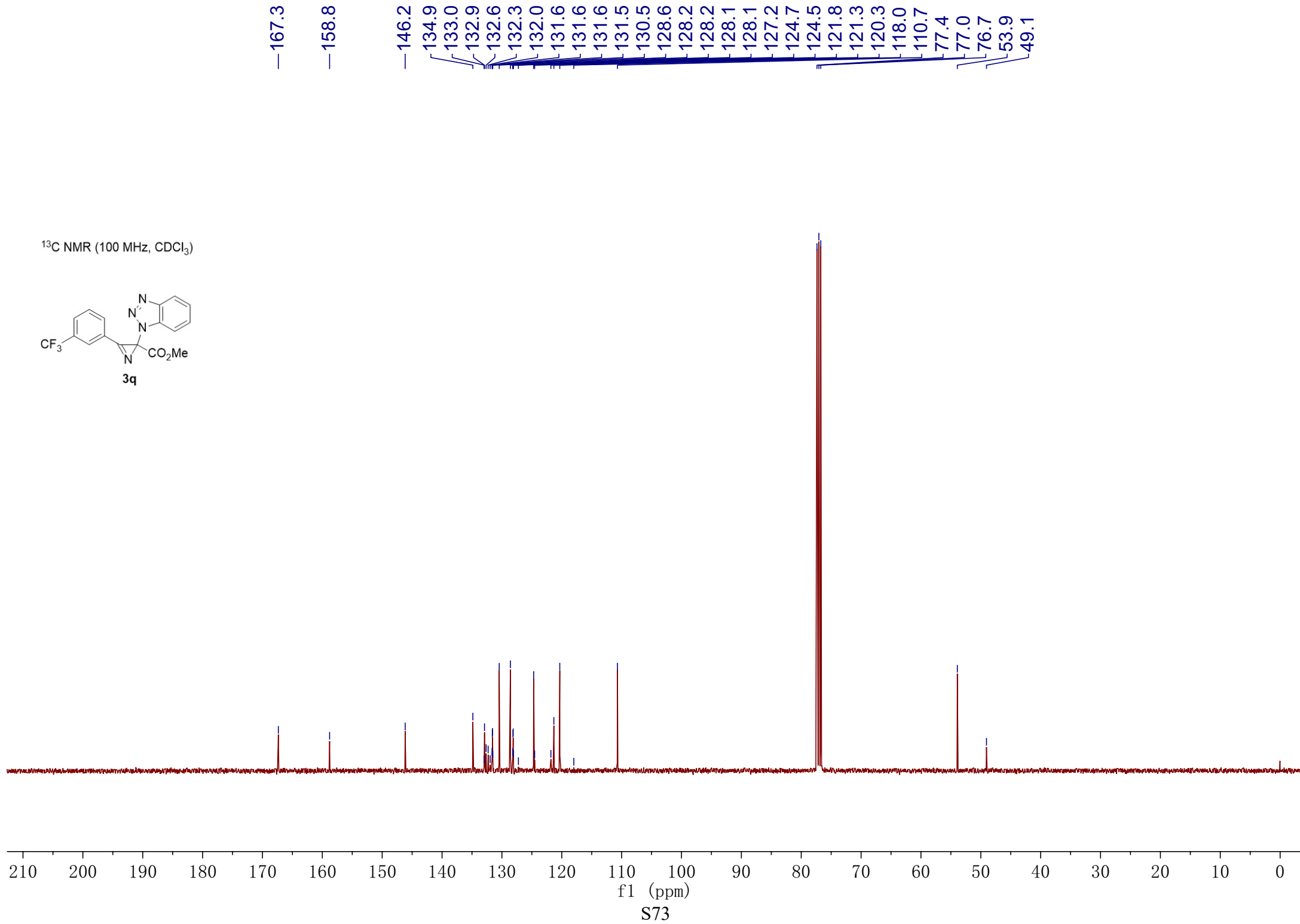
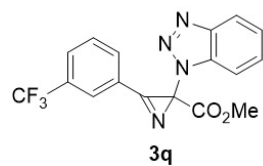
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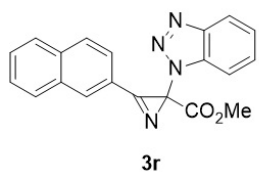


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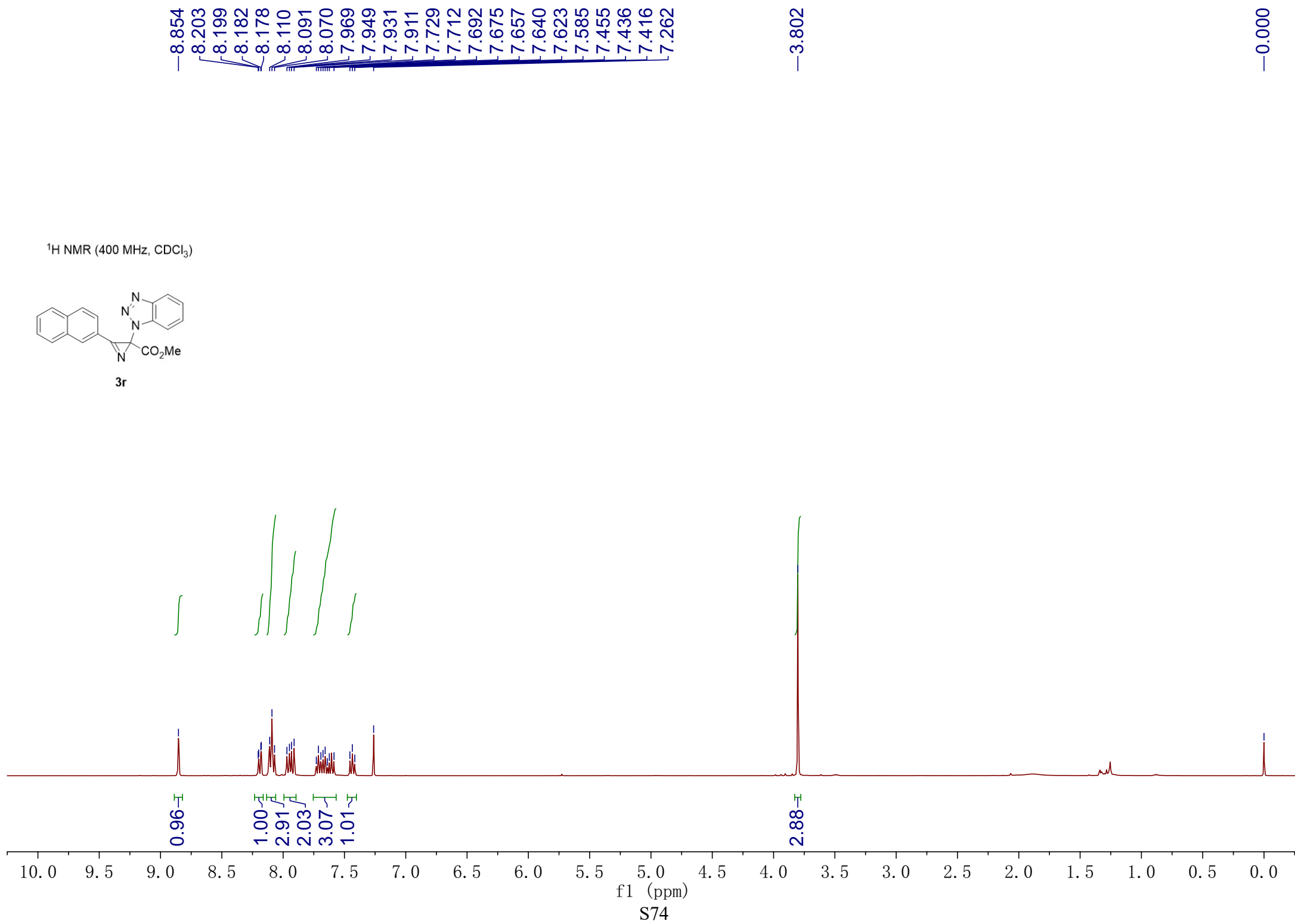


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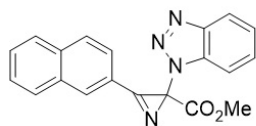




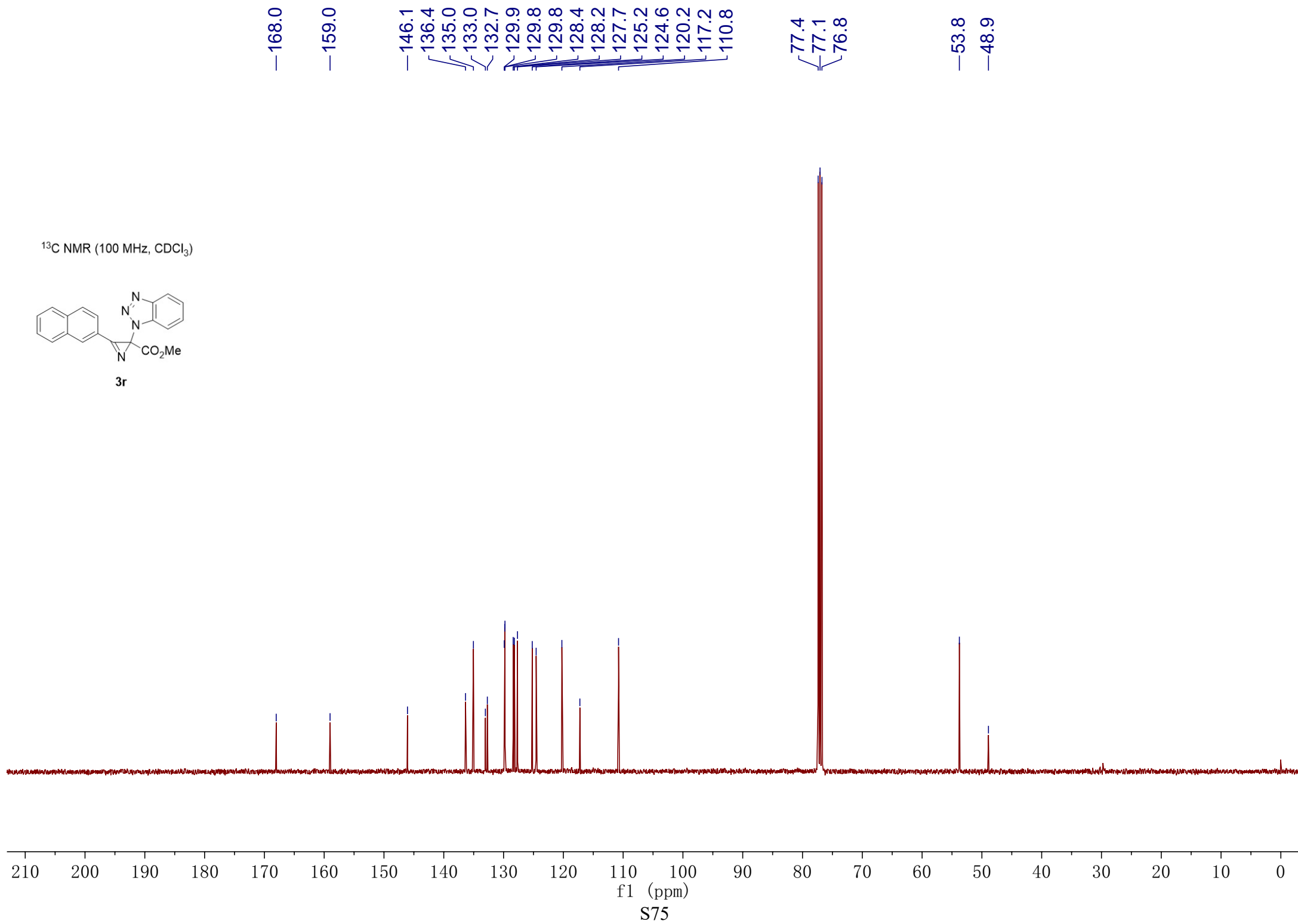
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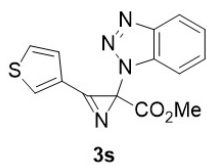
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3r



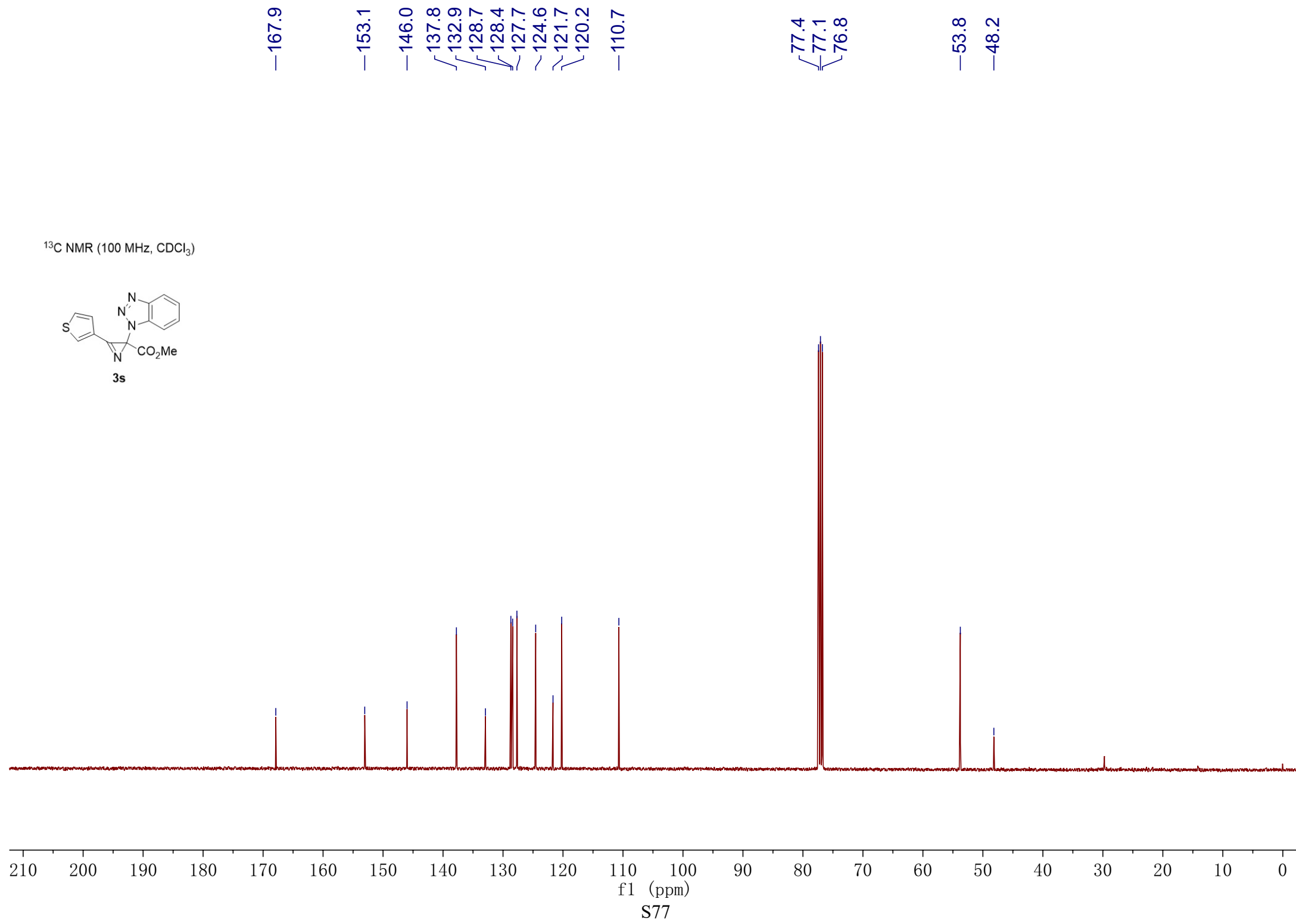
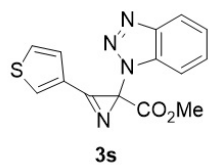
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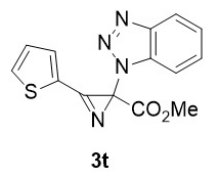


f1 (ppm)

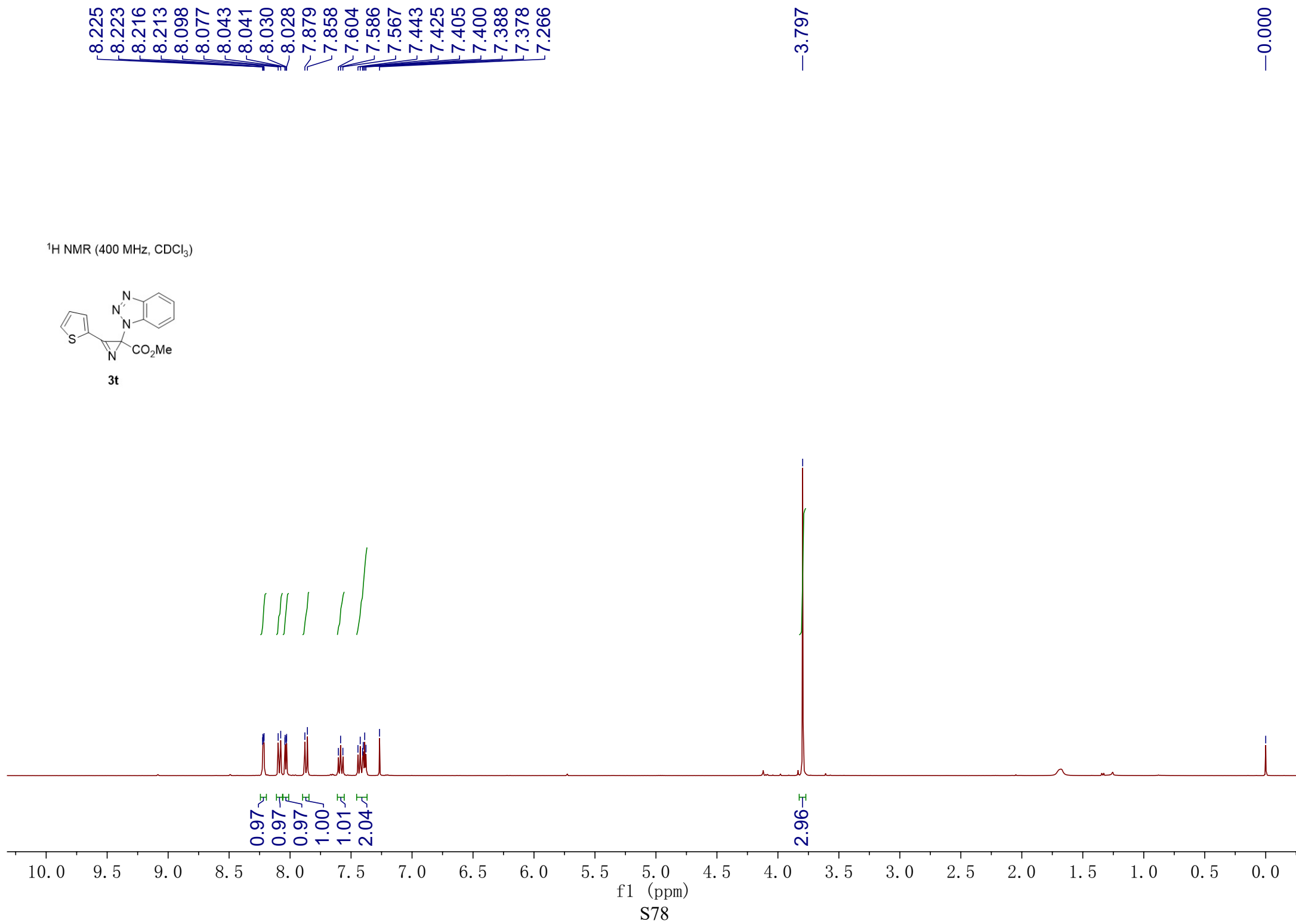
S76

¹³C NMR (100 MHz, CDCl₃)

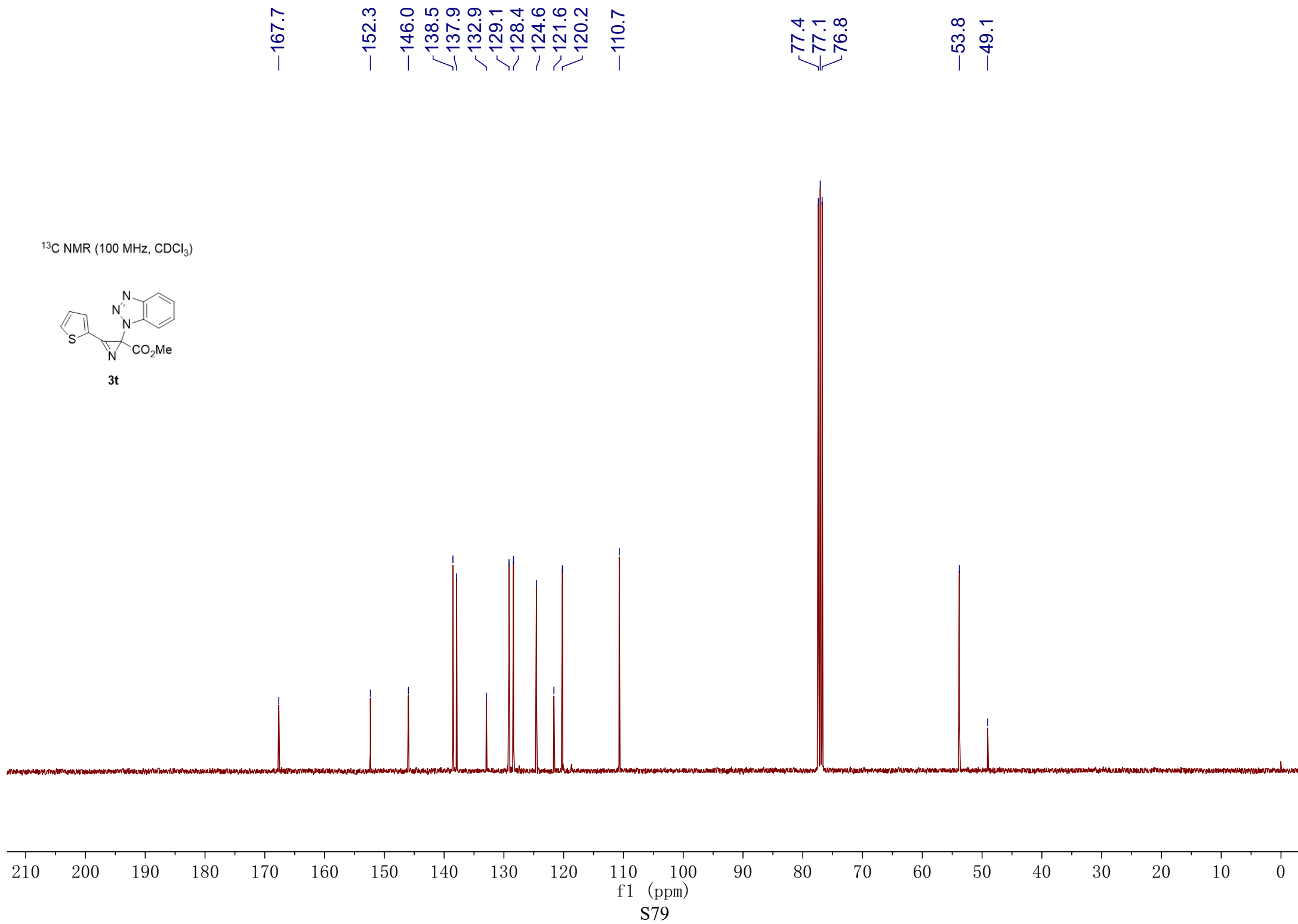
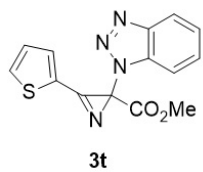




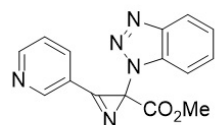
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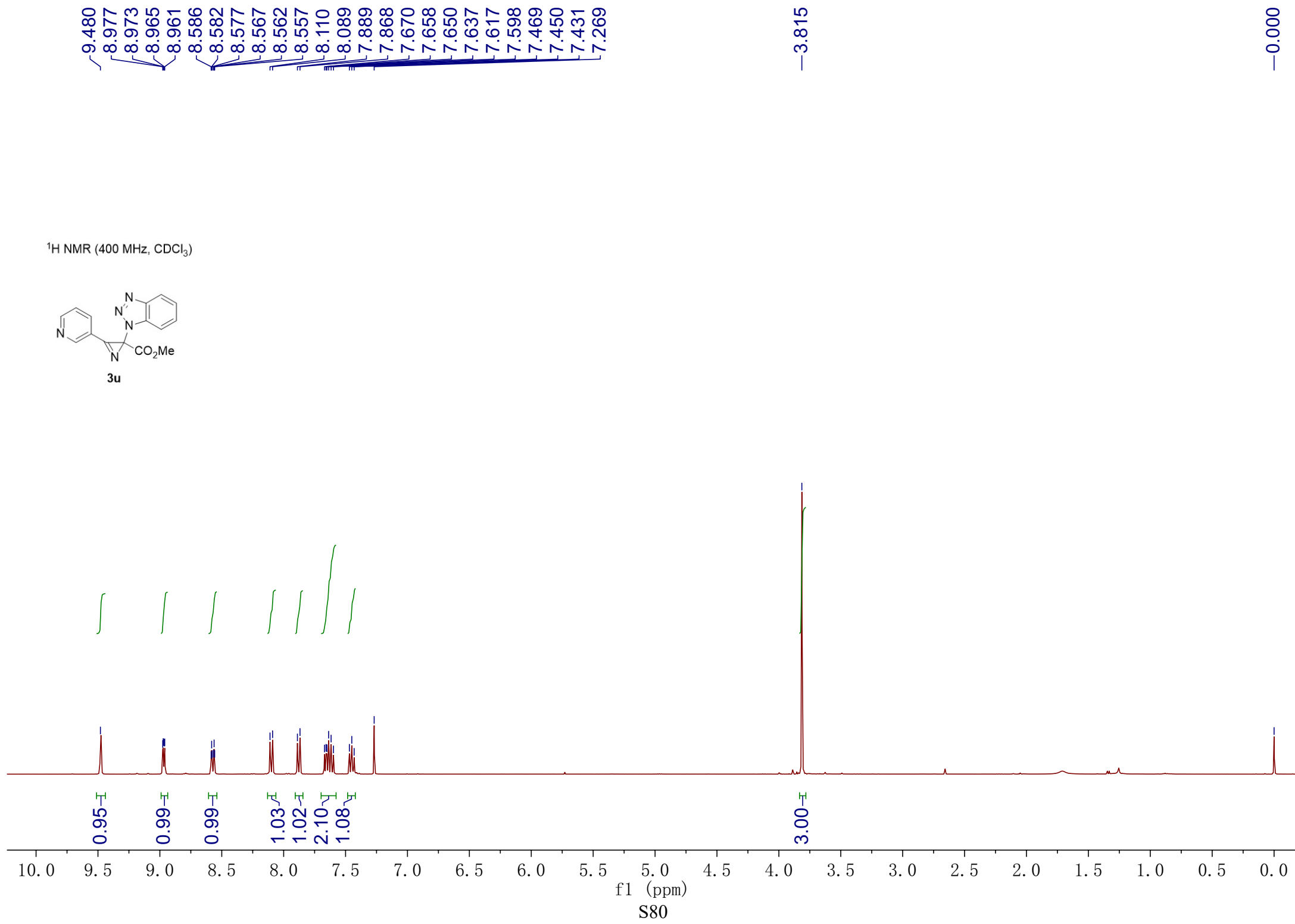
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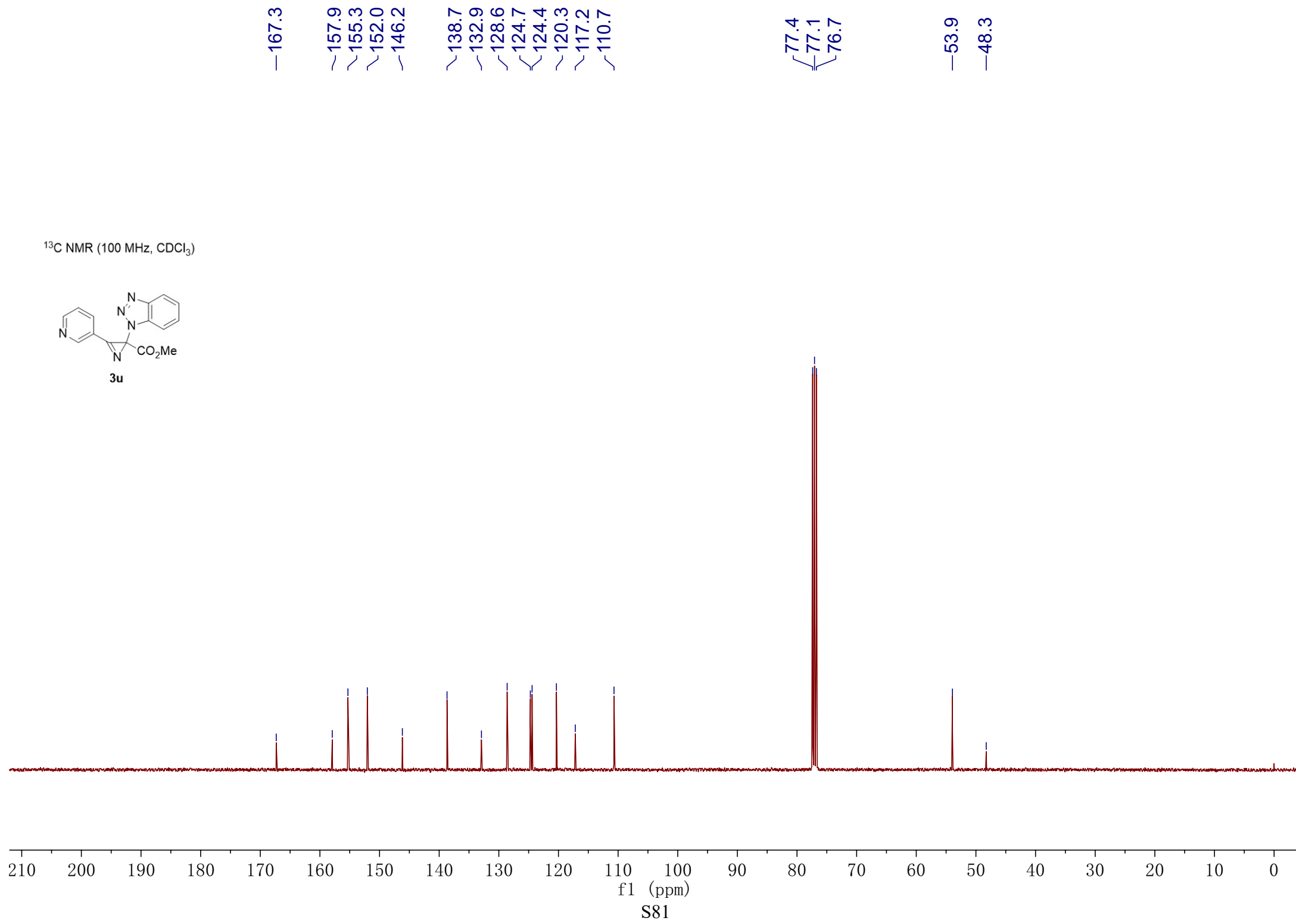
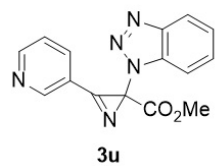
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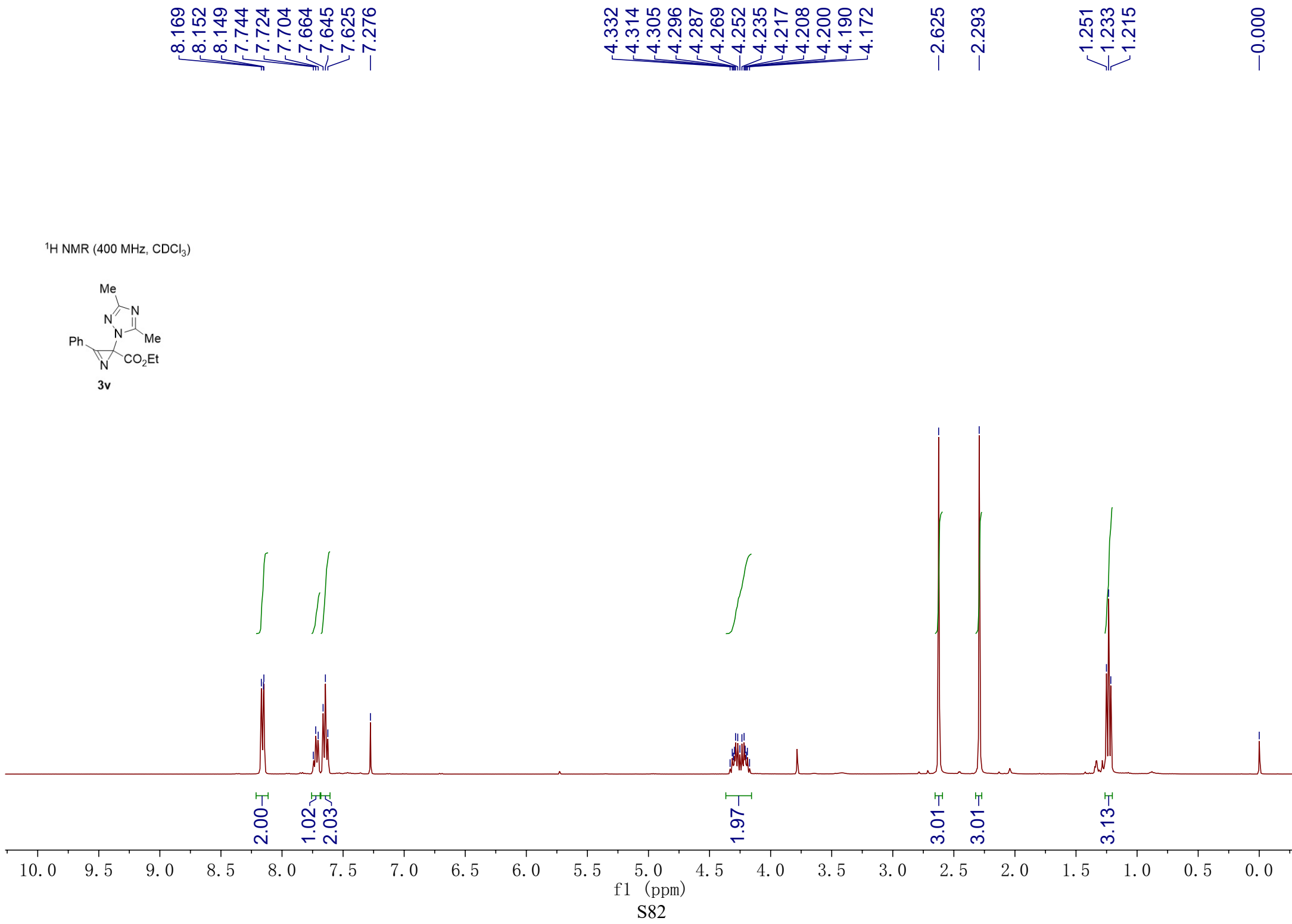
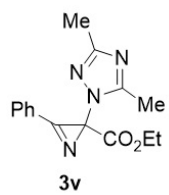
3u



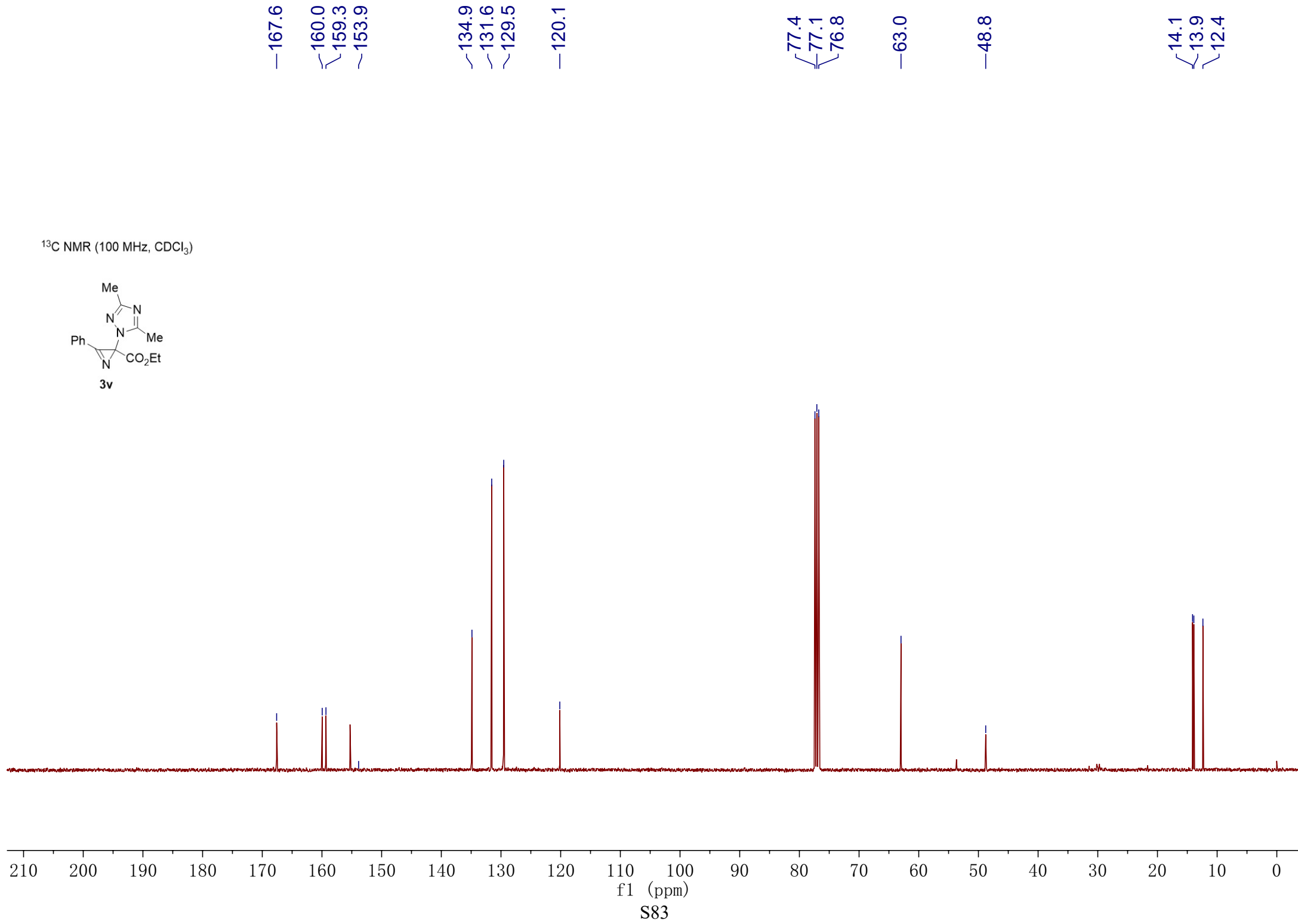
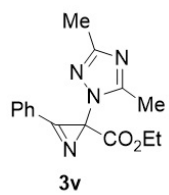
¹³C NMR (100 MHz, CDCl₃)

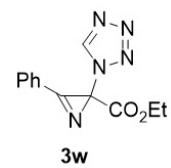


¹H NMR (400 MHz, CDCl₃)

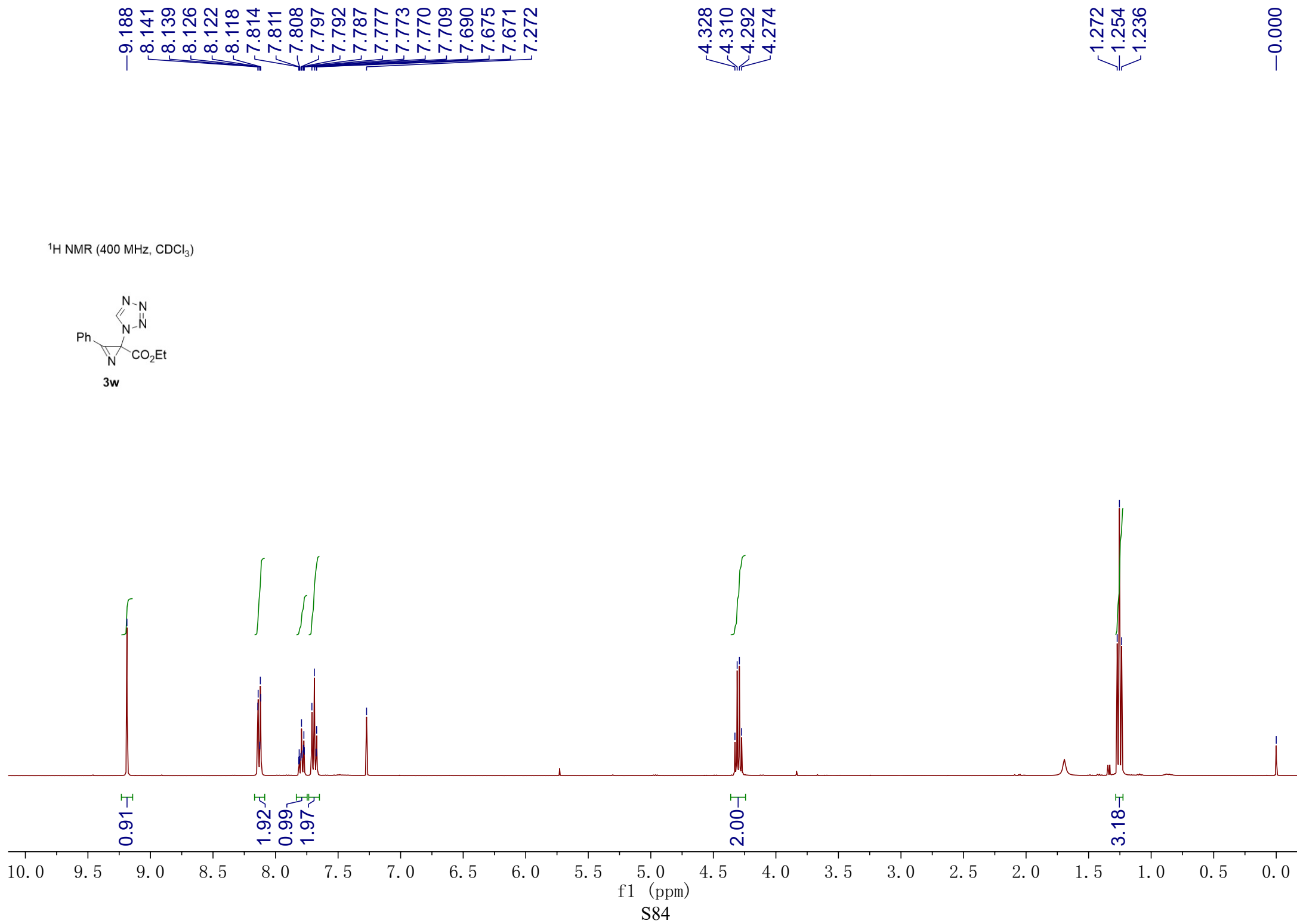


¹³C NMR (100 MHz, CDCl₃)

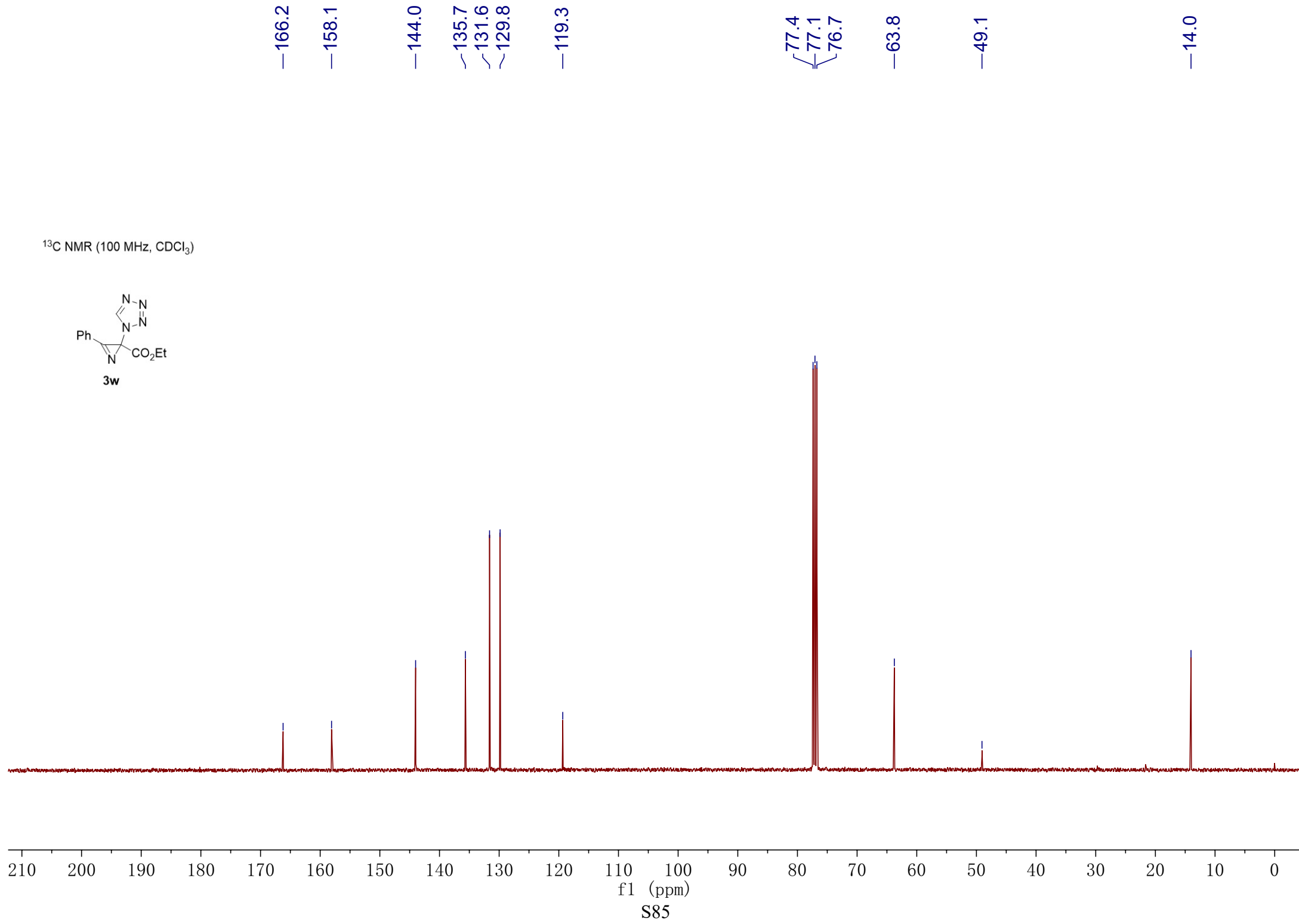
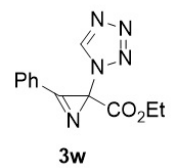




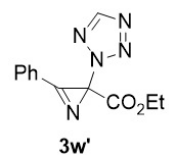
¹H NMR (400 MHz, CDCl₃)



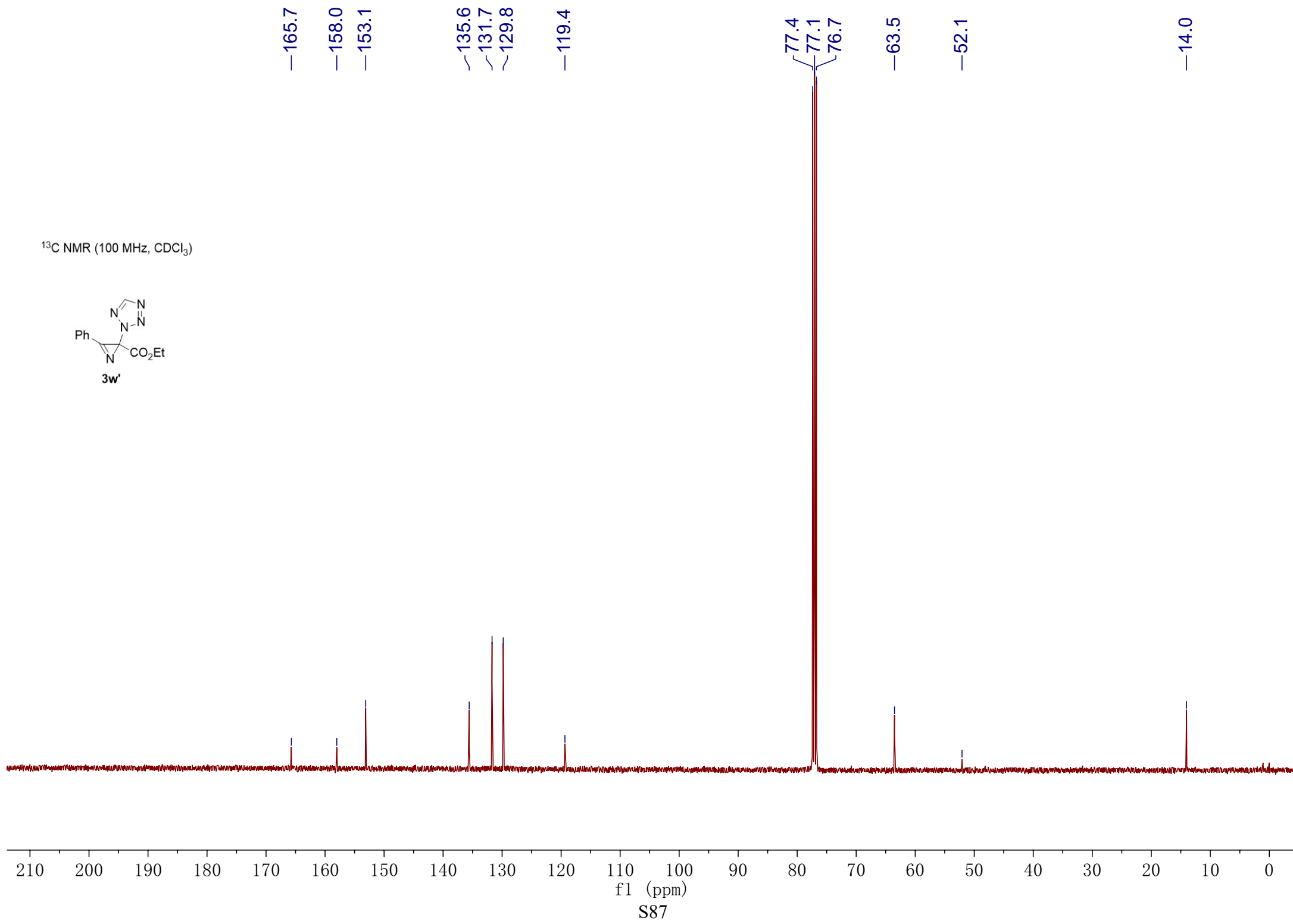
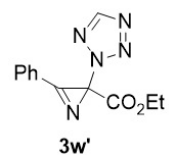
¹³C NMR (100 MHz, CDCl₃)



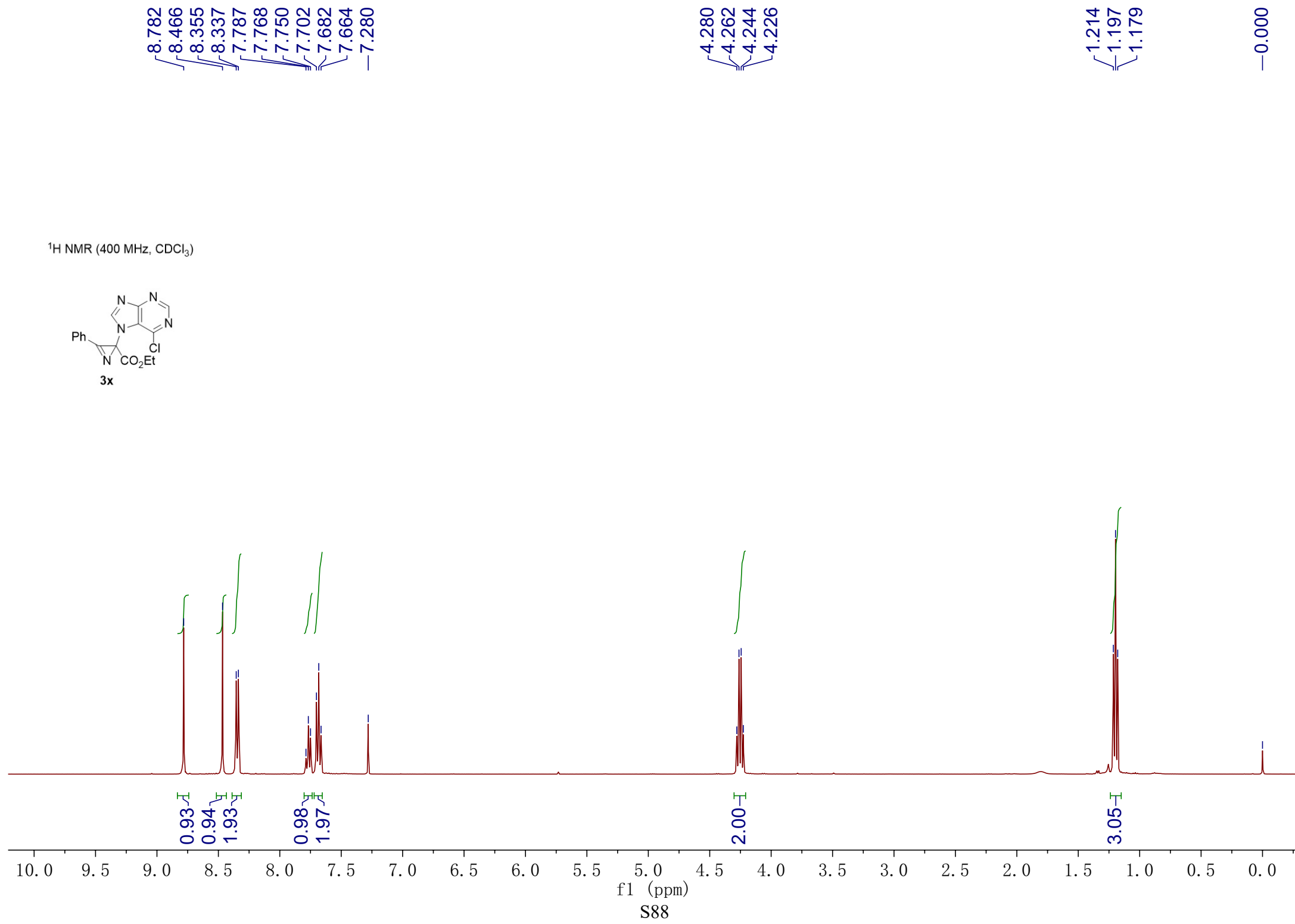
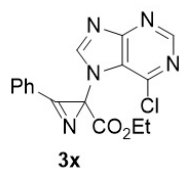
¹H NMR (400 MHz, CDCl₃)



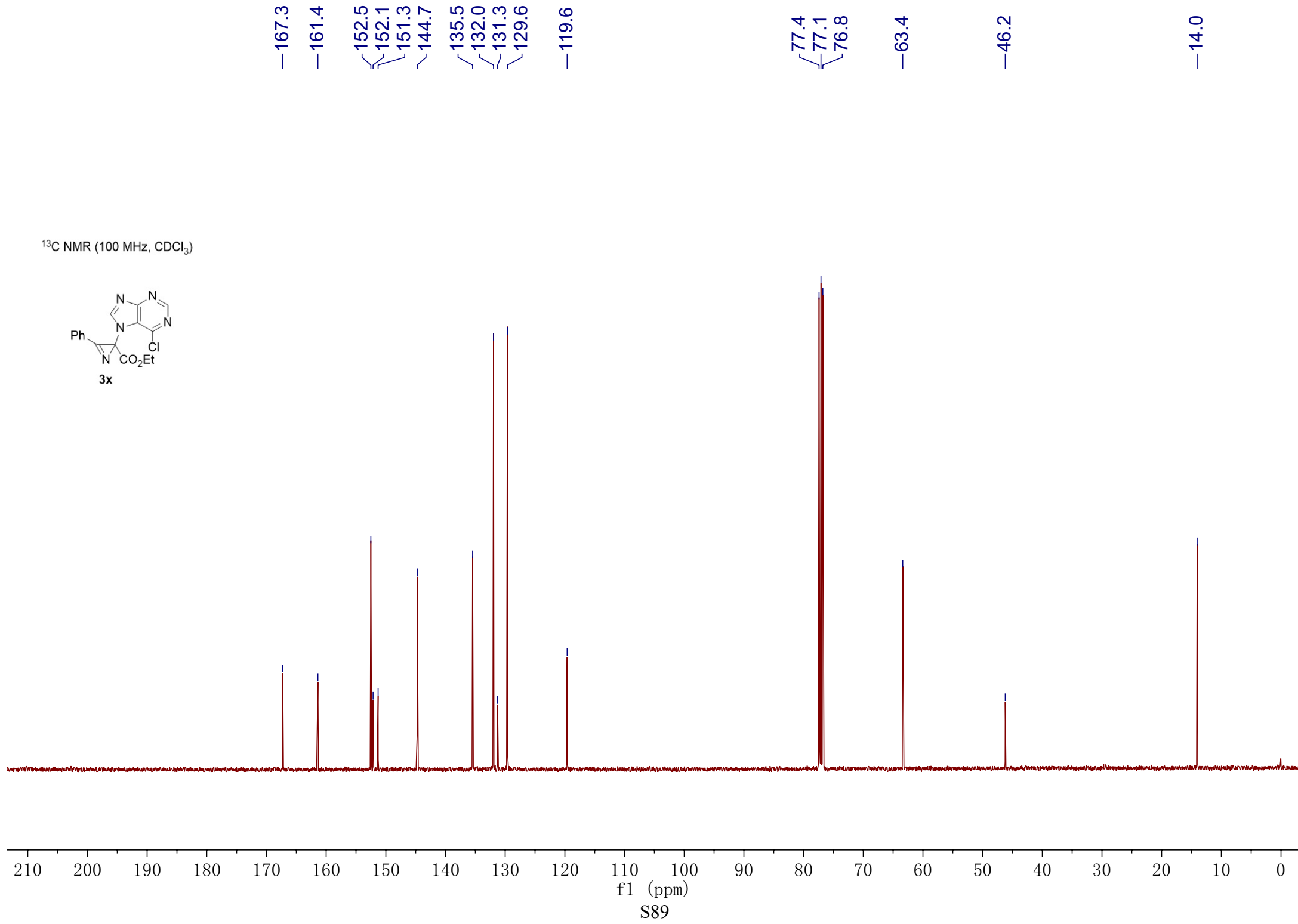
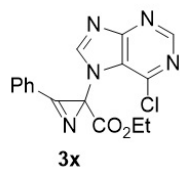
¹³C NMR (100 MHz, CDCl₃)



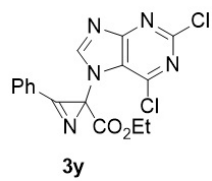
¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)



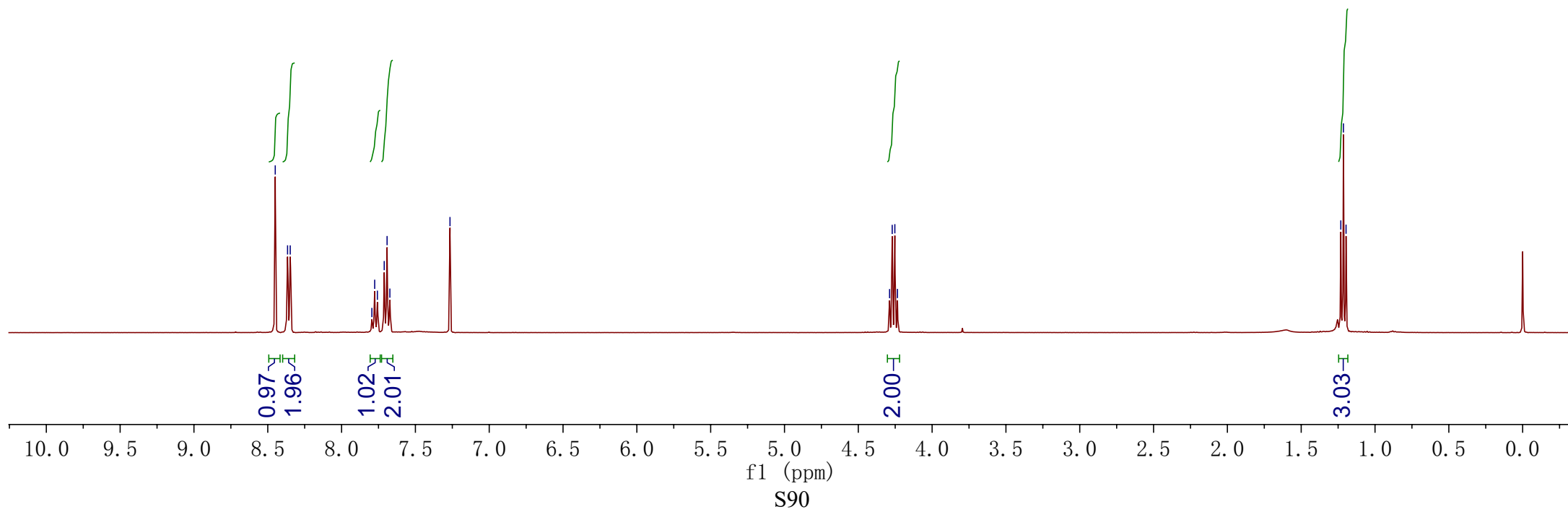
¹H NMR (400 MHz, CDCl₃)



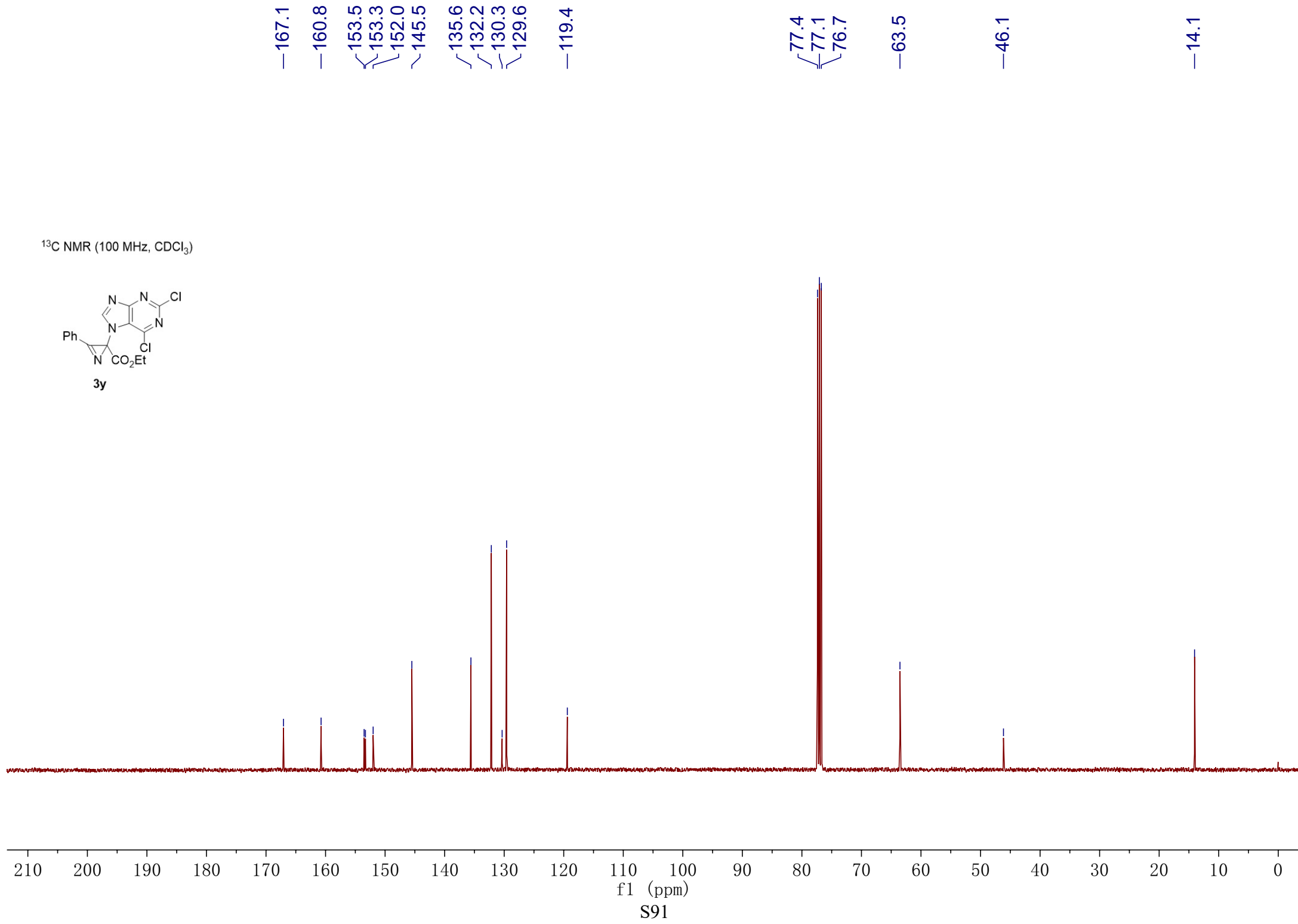
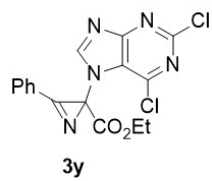
8.449
8.366
8.348
7.795
7.776
7.758
7.711
7.692
7.673
7.266

4.288
4.270
4.252
4.235

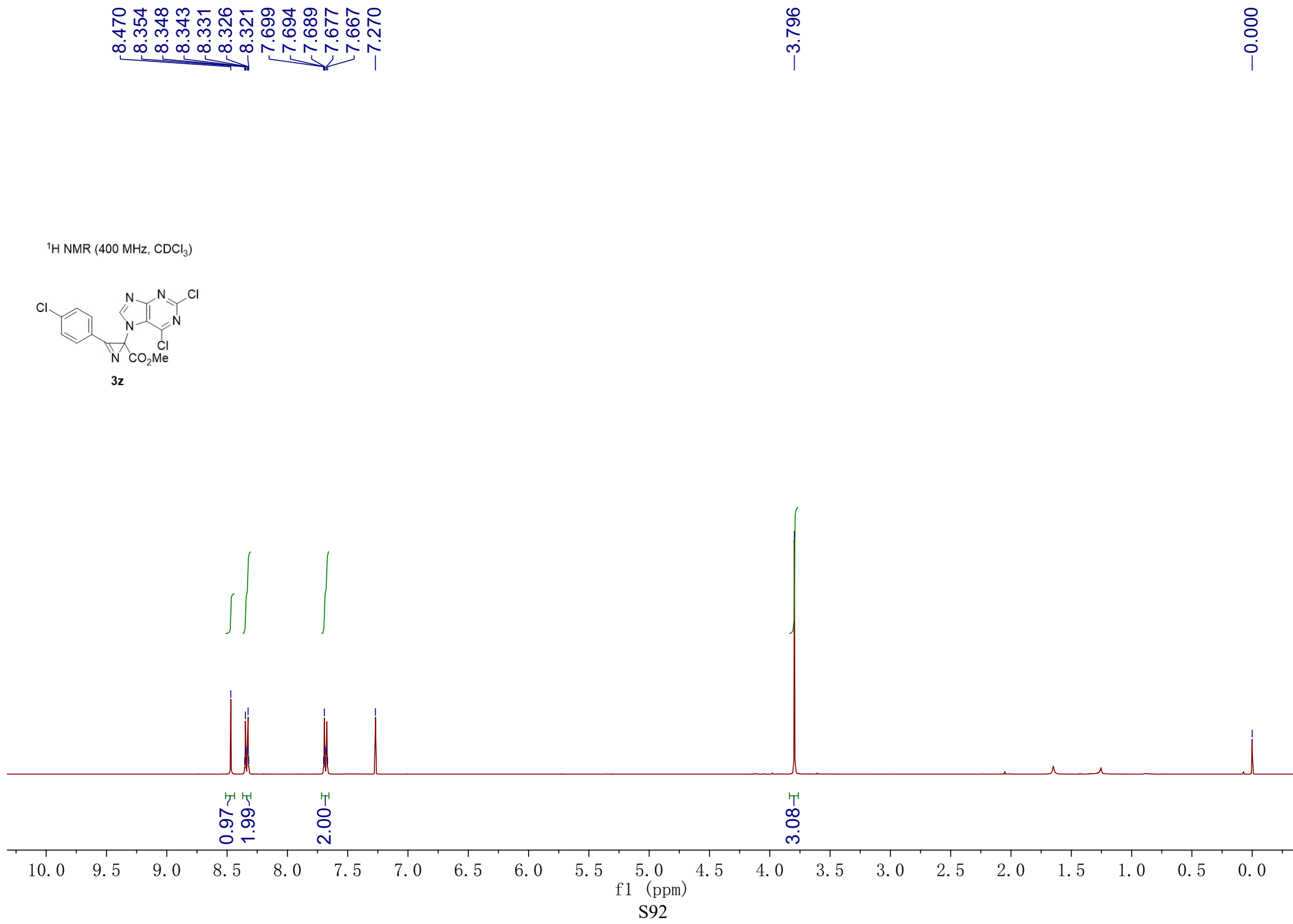
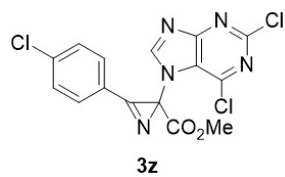
1.232
1.214
1.196



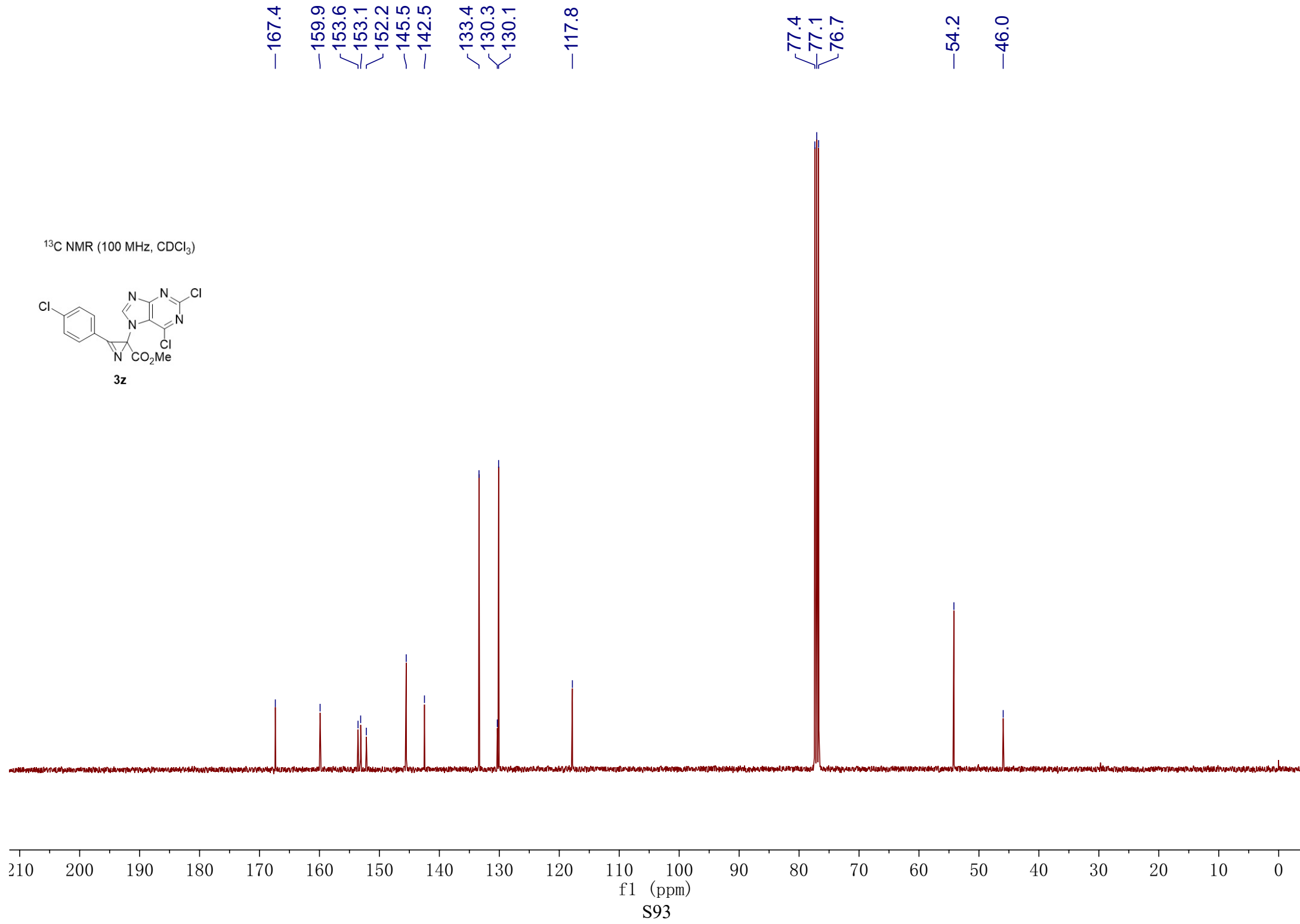
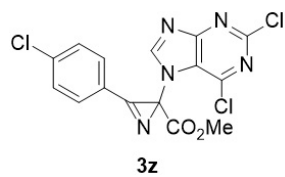
¹³C NMR (100 MHz, CDCl₃)



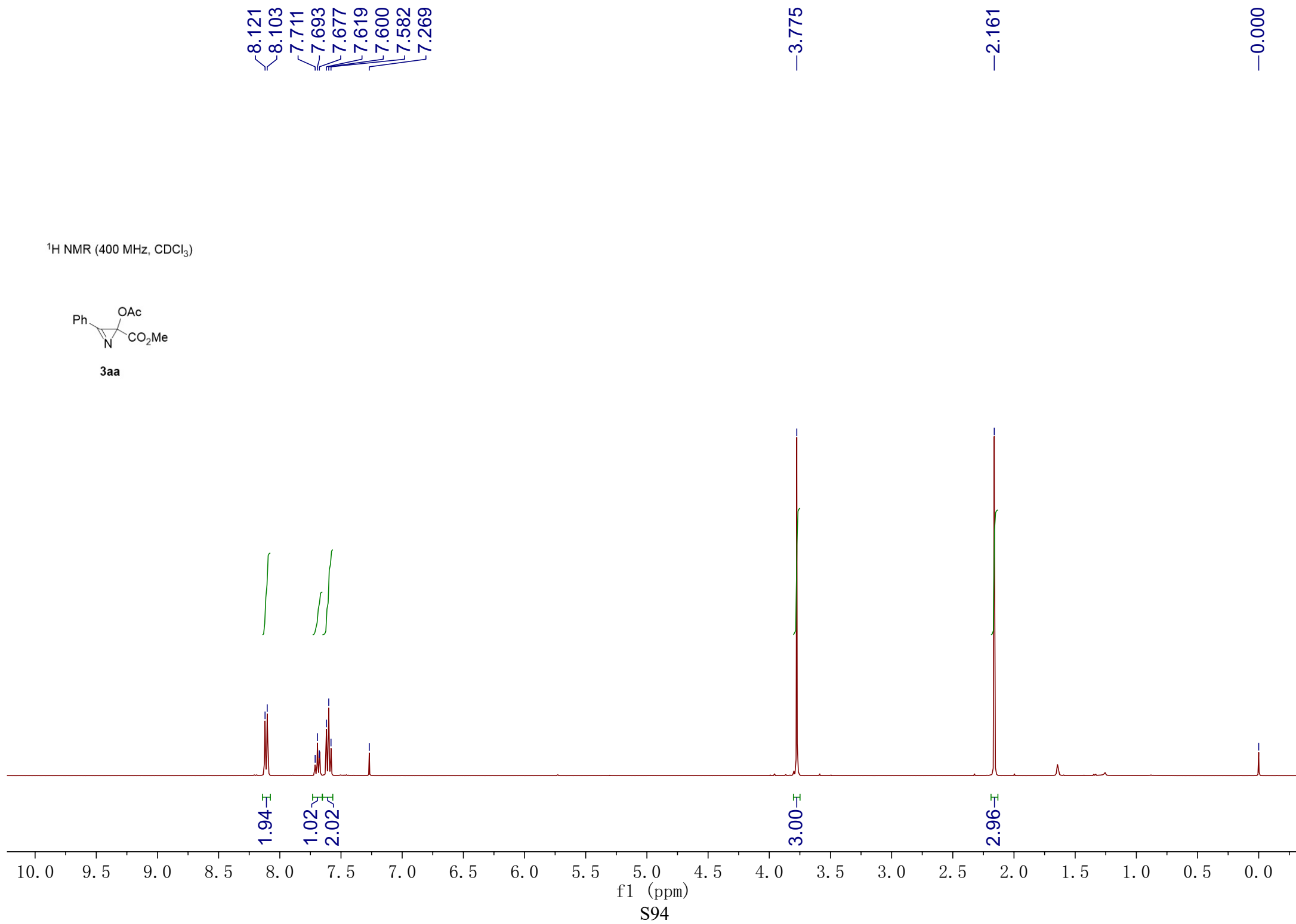
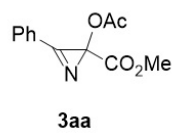
¹H NMR (400 MHz, CDCl₃)



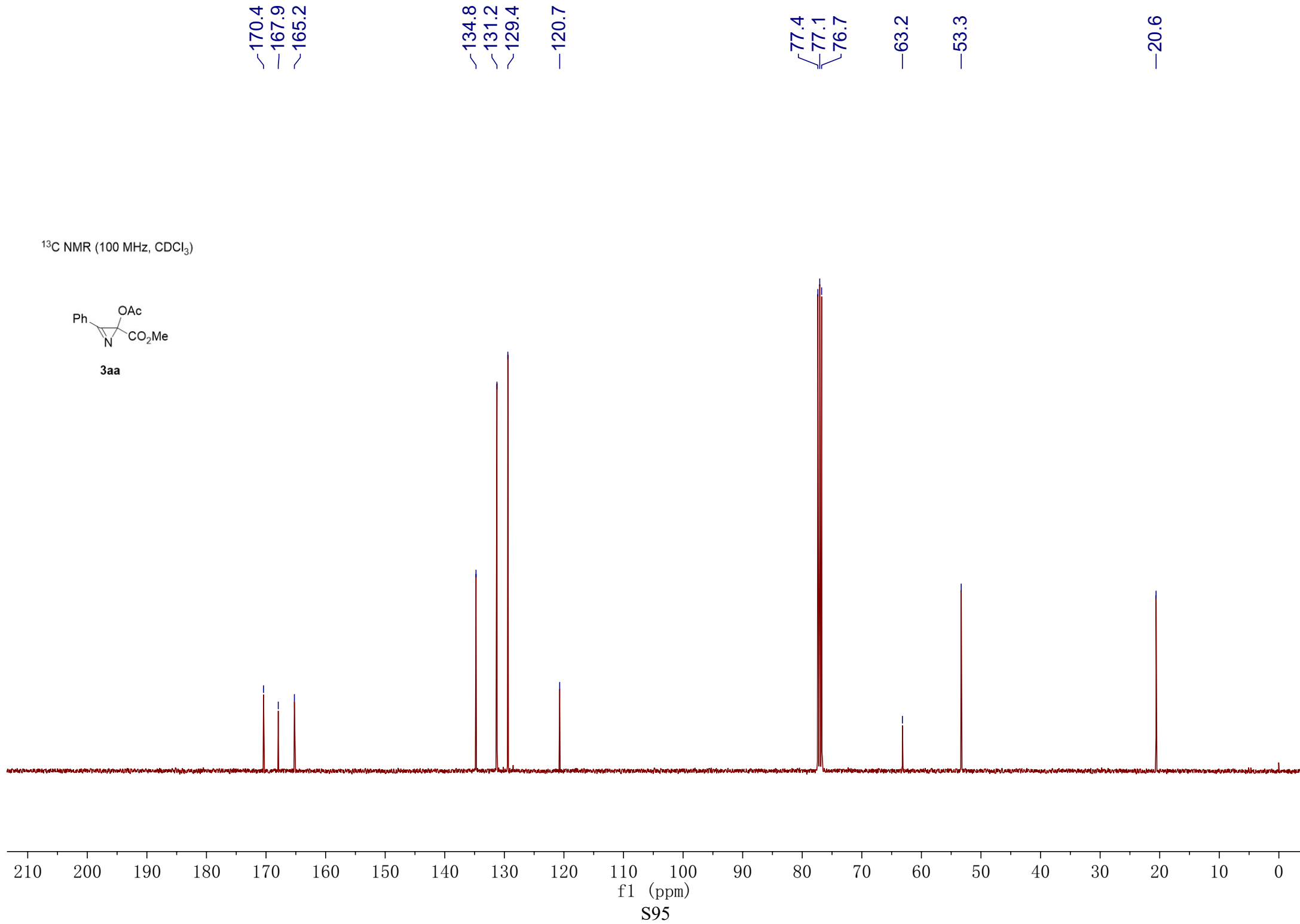
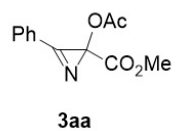
¹³C NMR (100 MHz, CDCl₃)



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)



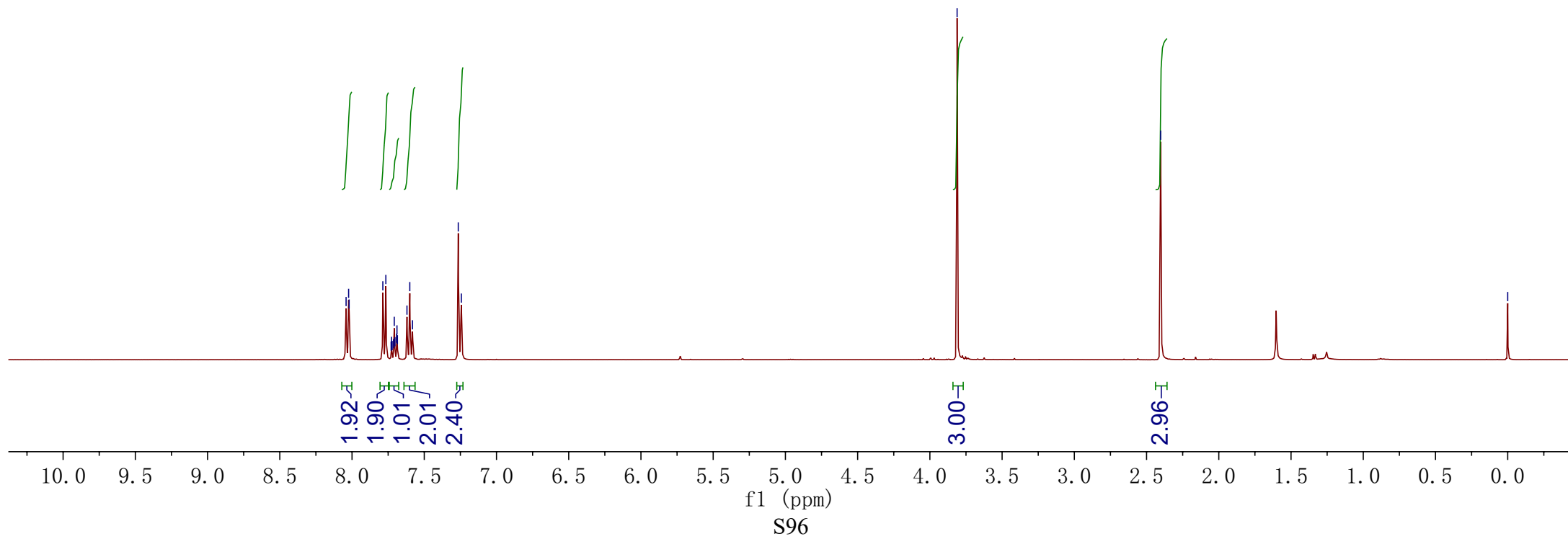
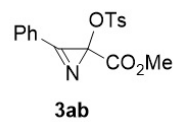
8.041
8.024
8.020
7.787
7.766
7.729
7.726
7.723
7.723
7.713
7.708
7.703
7.692
7.689
7.686
7.620
7.600
7.582
7.265
7.243

—3.812

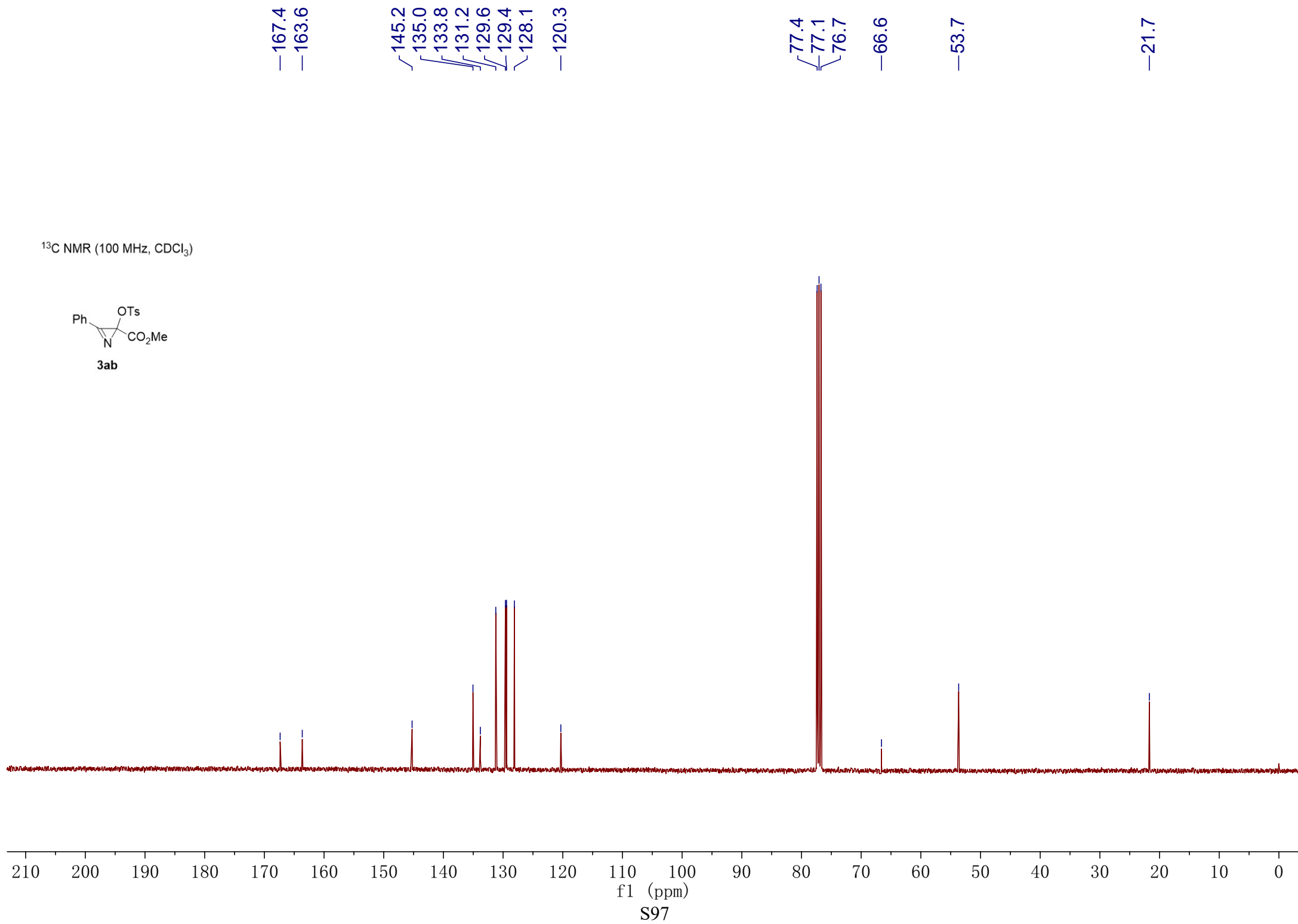
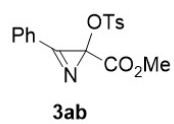
—2.403

—0.000

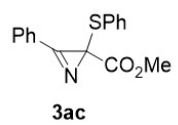
¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)

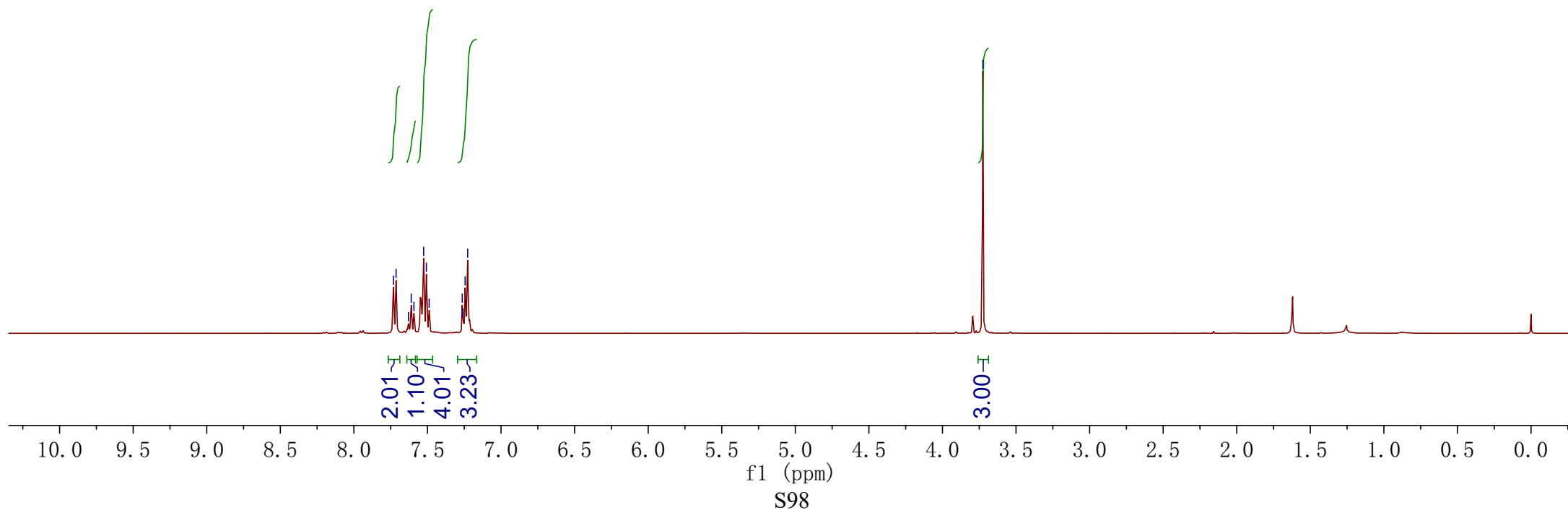


¹H NMR (400 MHz, CDCl₃)

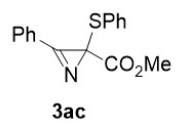


7.731
7.713
7.629
7.610
7.592
7.526
7.507
7.489
7.264
7.258
7.244
7.227

3.726



¹³C NMR (100 MHz, CDCl₃)



—170.2

—160.6

134.2

133.0

131.6

130.5

129.3

129.0

128.0

121.3

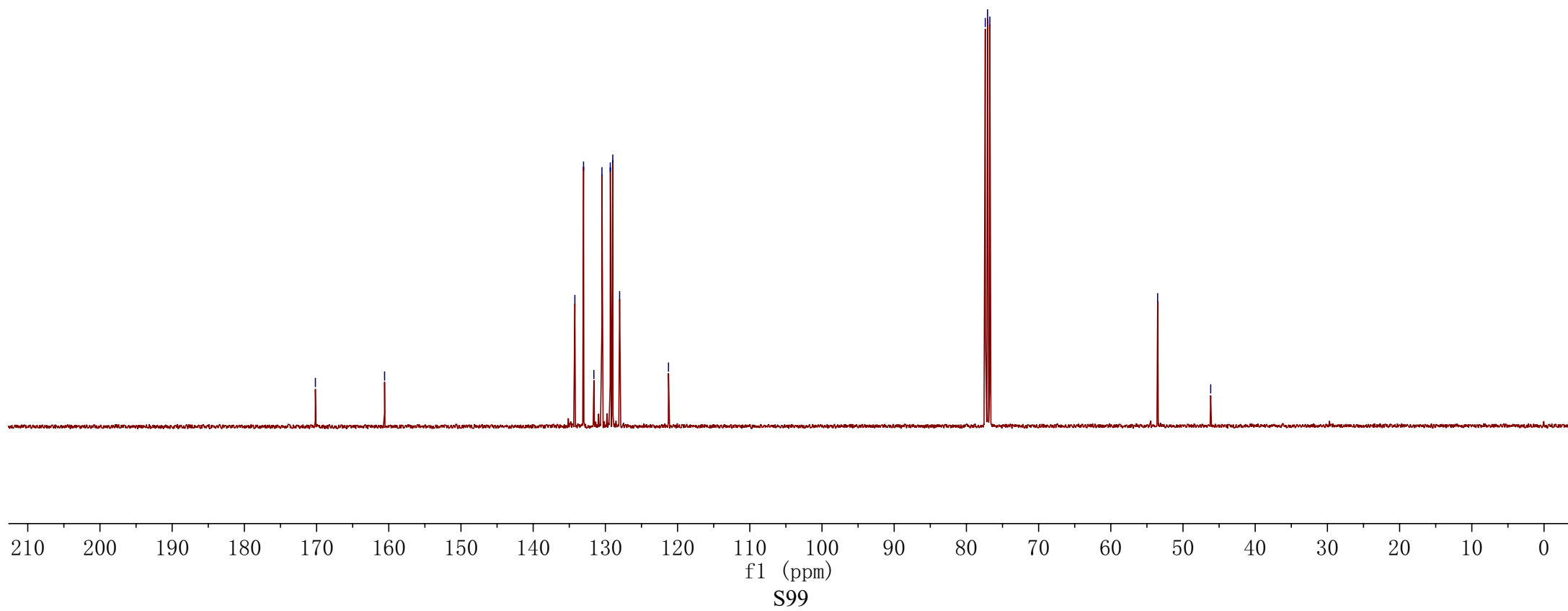
77.4

77.1

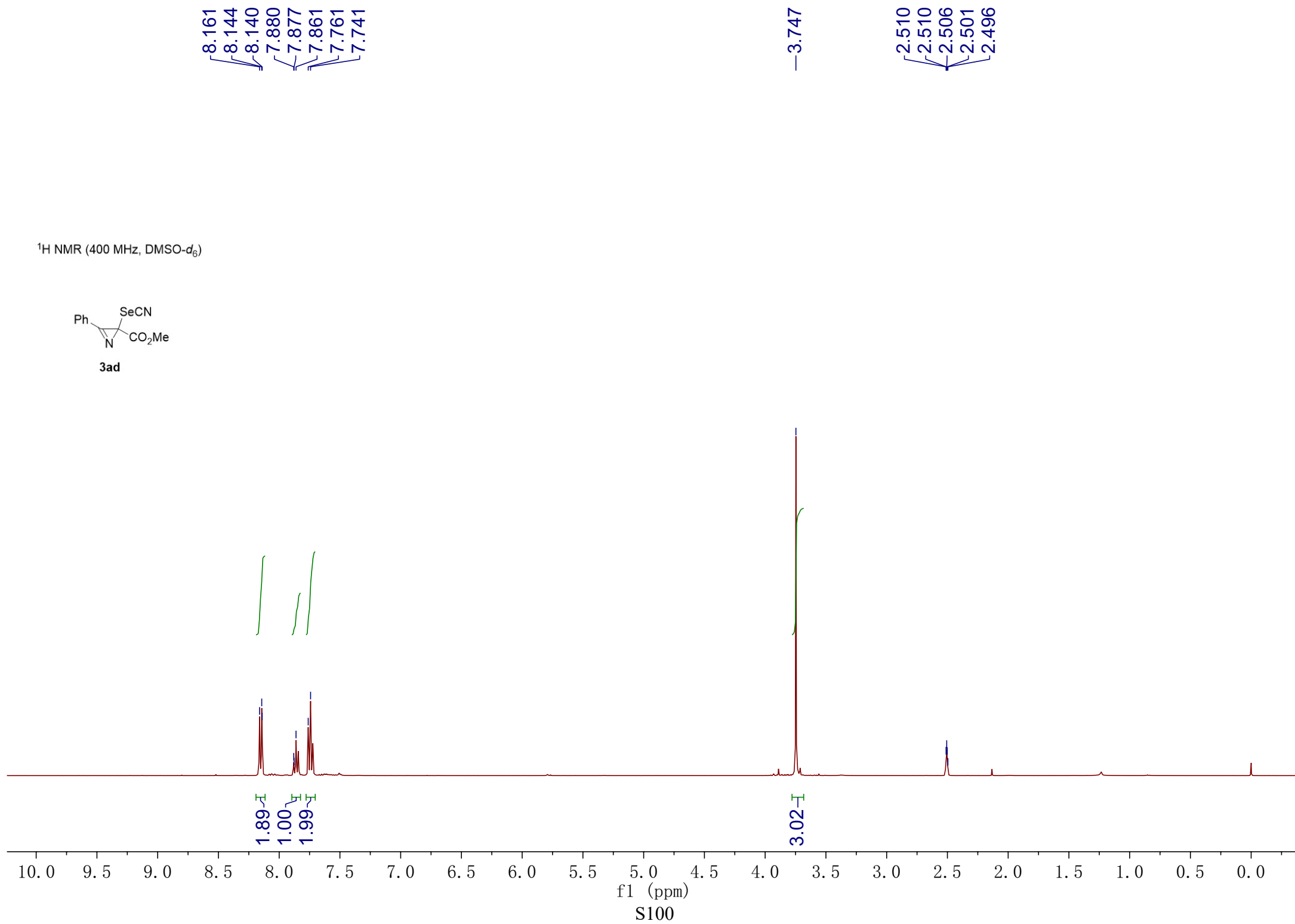
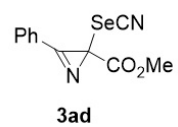
76.8

—53.5

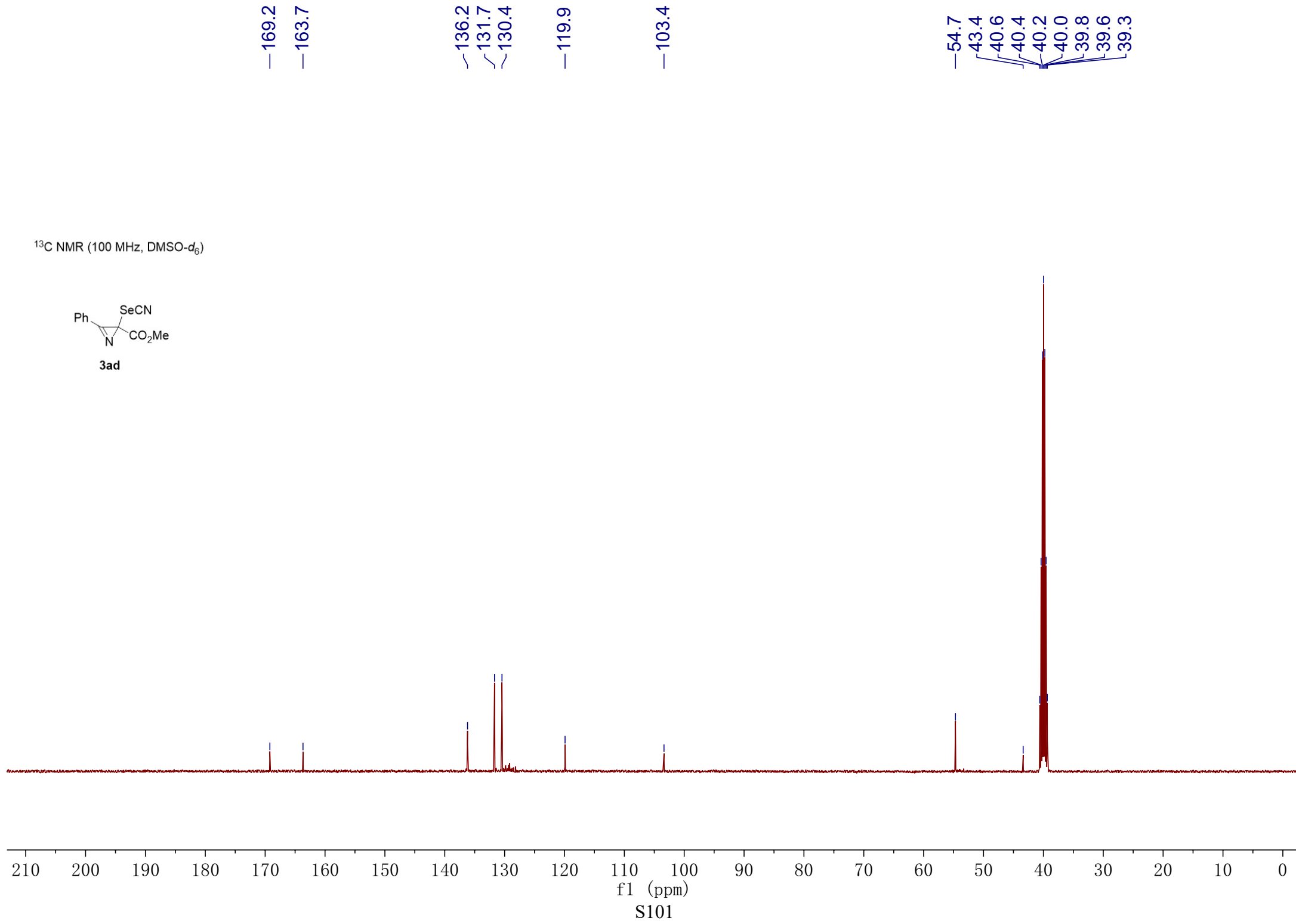
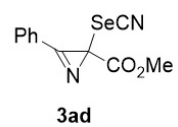
—46.2



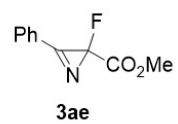
¹H NMR (400 MHz, DMSO-*d*₆)



^{13}C NMR (100 MHz, $\text{DMSO}-d_6$)



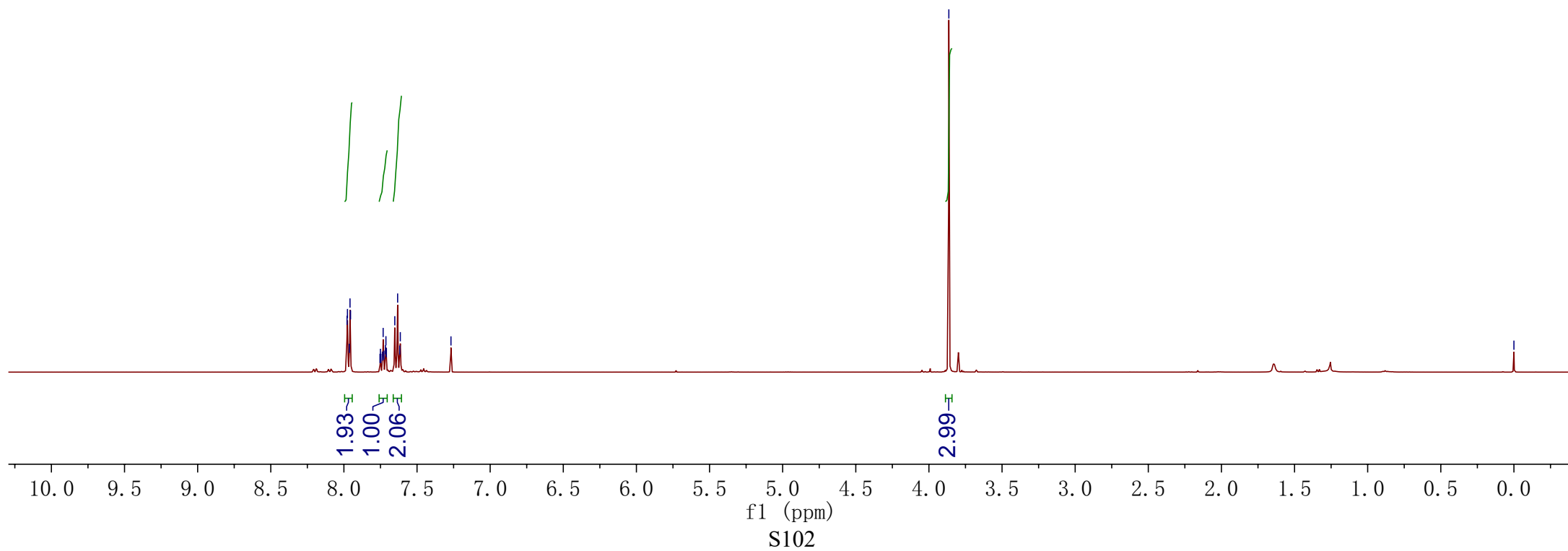
¹H NMR (400 MHz, CDCl₃)



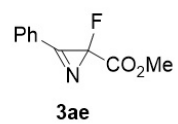
7.978
7.976
7.963
7.958
7.955
7.753
7.750
7.747
7.737
7.732
7.726
7.716
7.713
7.709
7.652
7.633
7.618
7.614
7.268

3.864

0.000



¹³C NMR (100 MHz, CDCl₃)



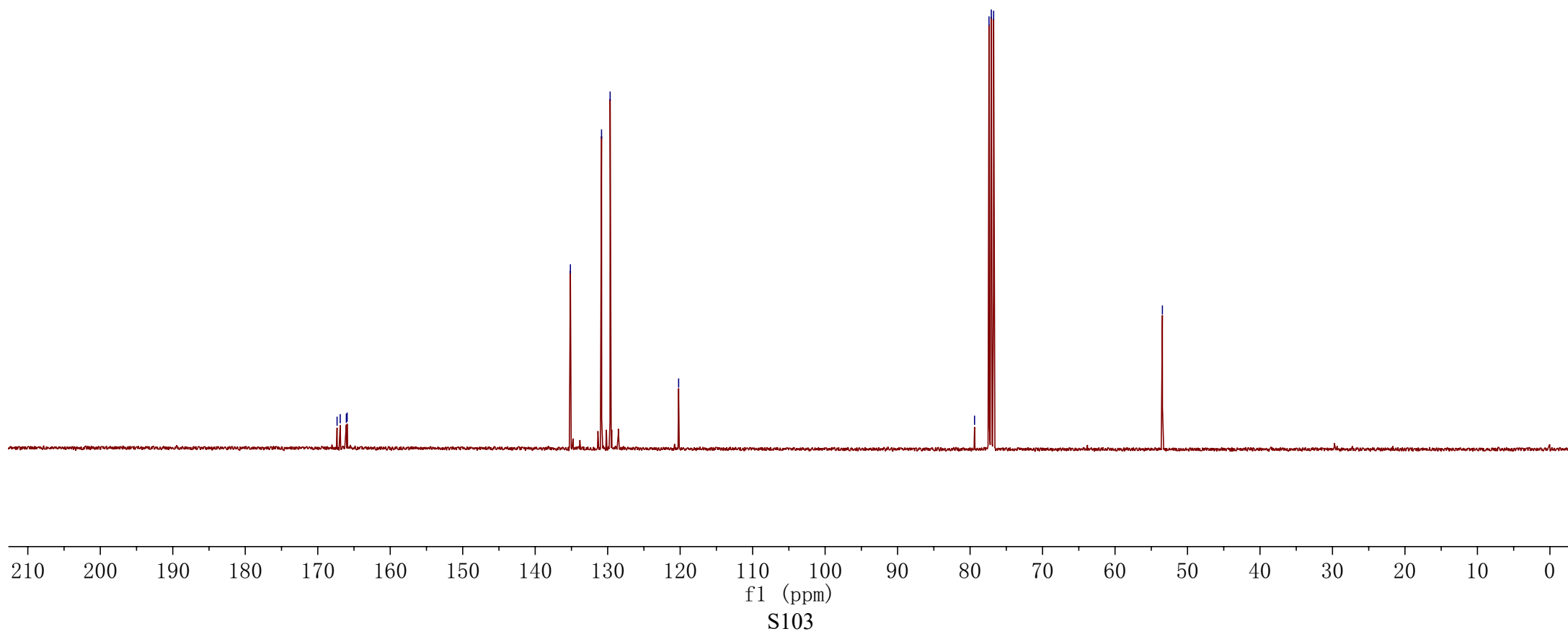
167.3
166.9
166.1
165.9

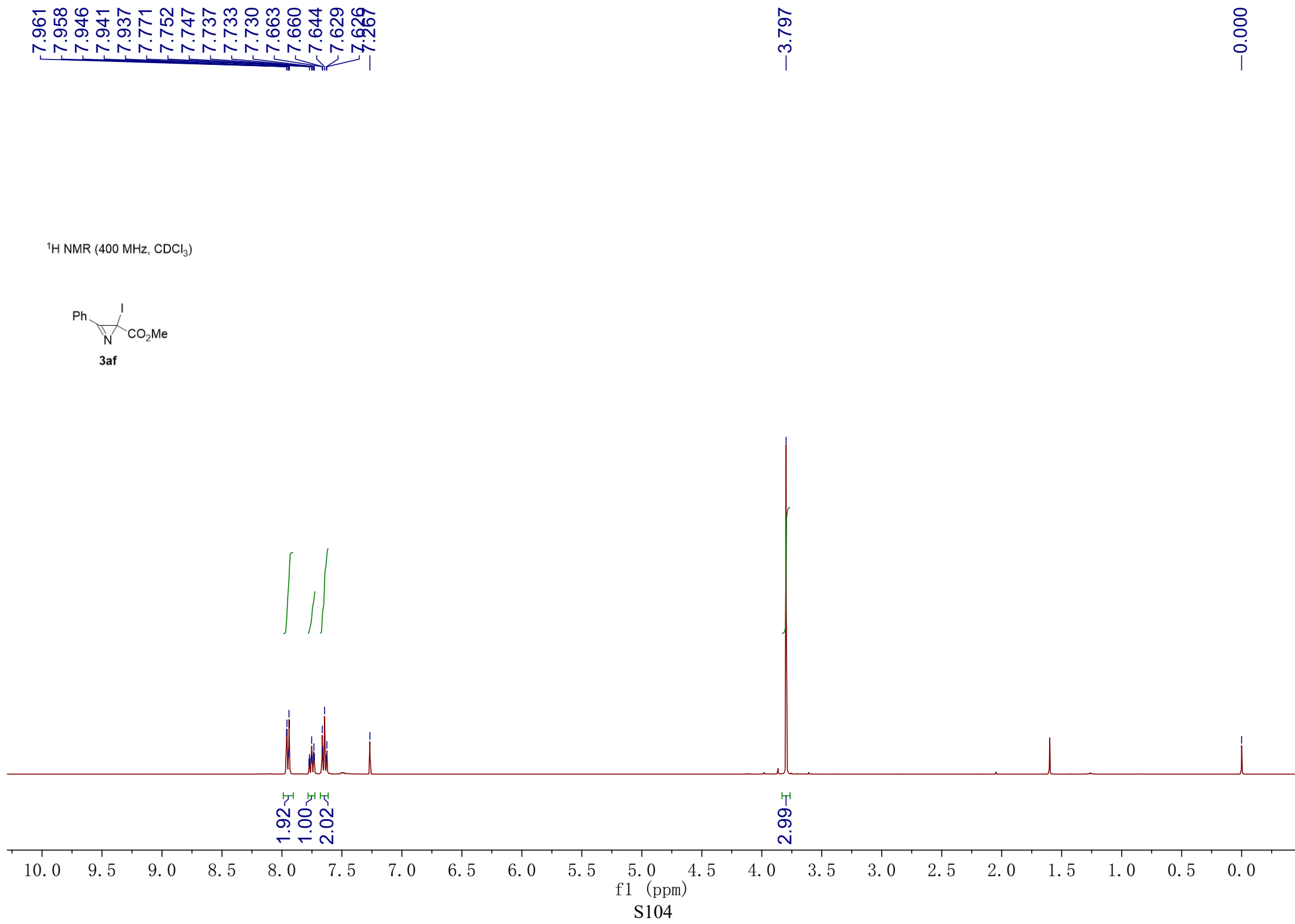
135.1
130.8
129.7

120.2

79.4
77.4
77.1
76.8

53.5





¹³C NMR (100 MHz, CDCl₃)

