

Supporting Information

Synthesis of Pyrido[1,2-*a*]indole Malonates and Amines through Aryne Annulation

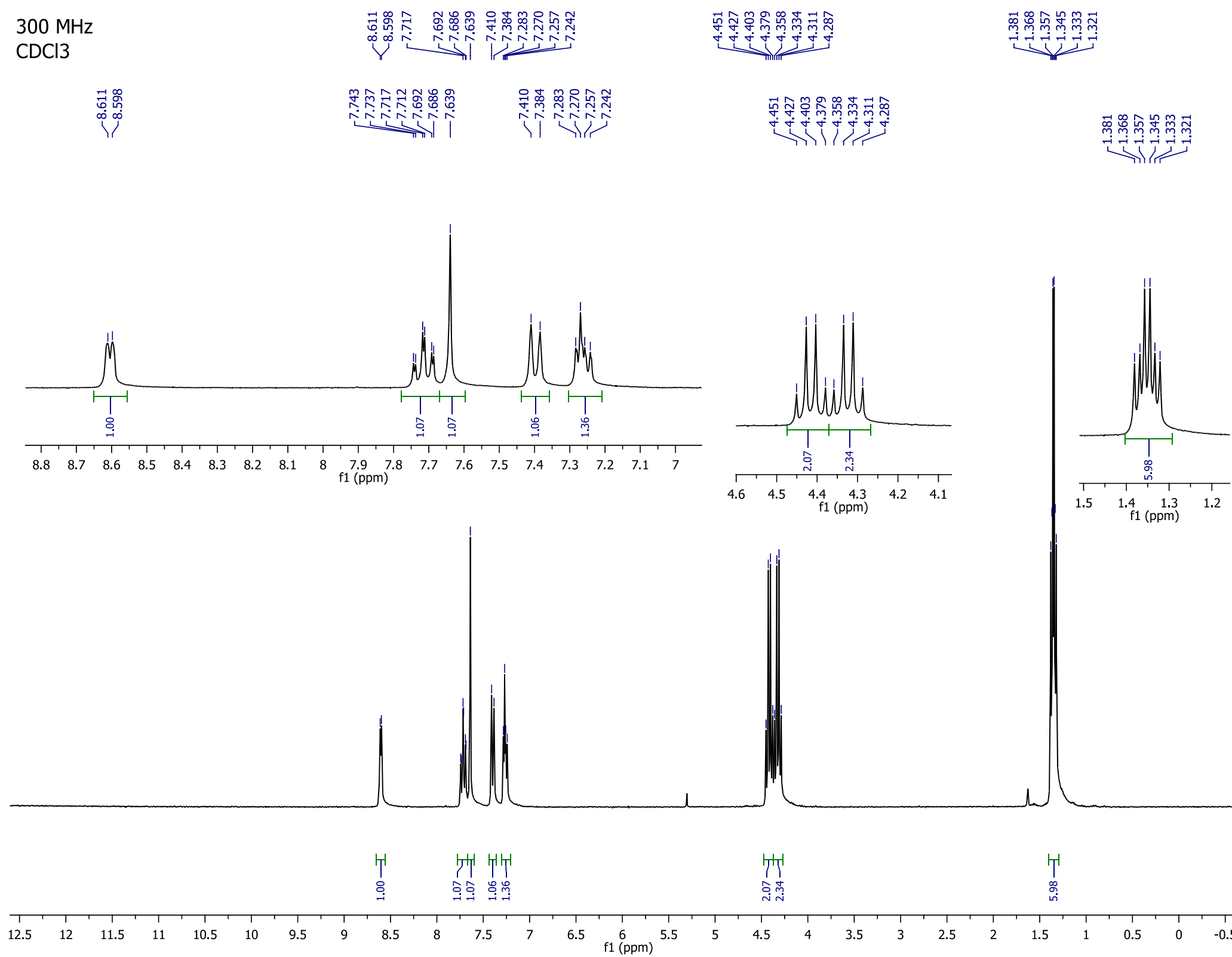
Donald C. Rogness, Nataliya A. Markina, Jesse P. Waldo and Richard C. Larock*

Department of Chemistry, Iowa State University, Ames, IA 50011

larock@iastate.edu

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Copies of ^1H and ^{13}C NMR spectra	S2-S144

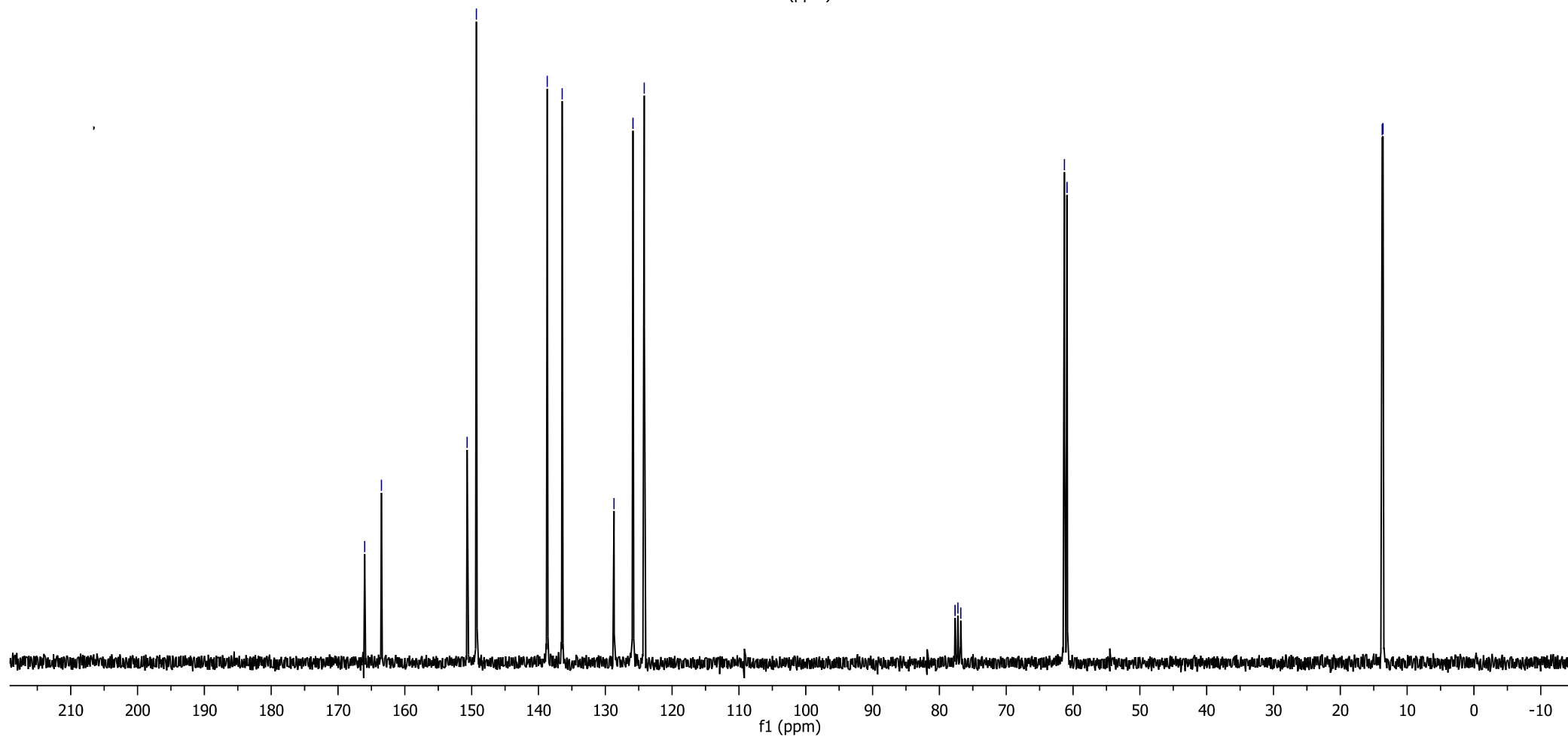
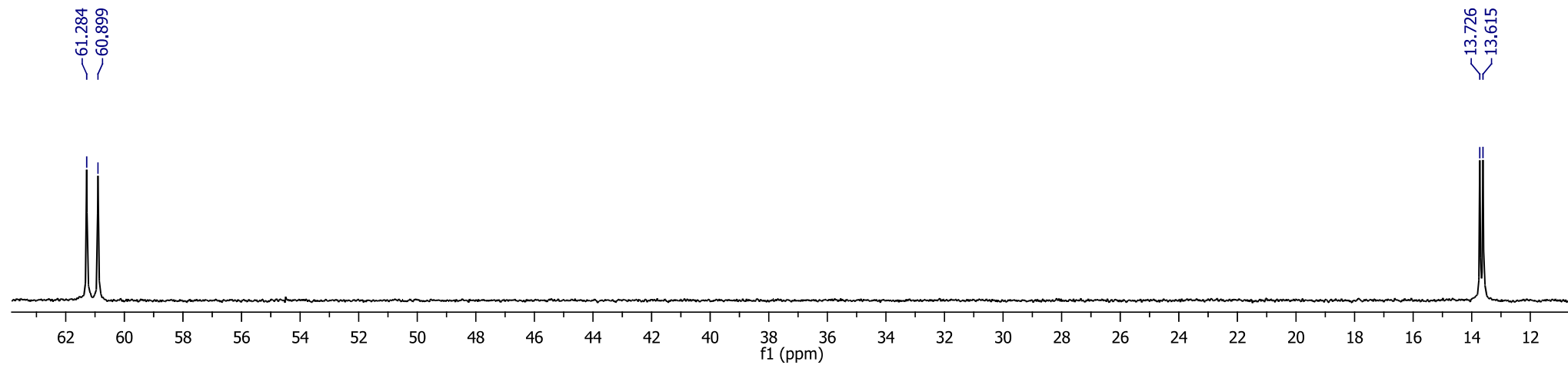
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75 MHz
CDCl₃

3

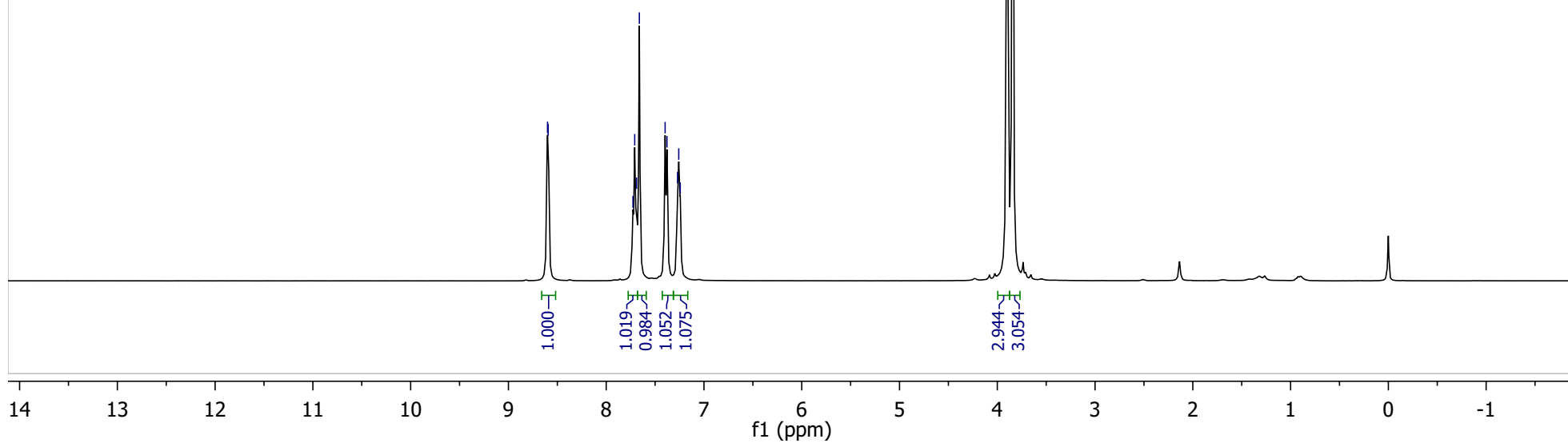
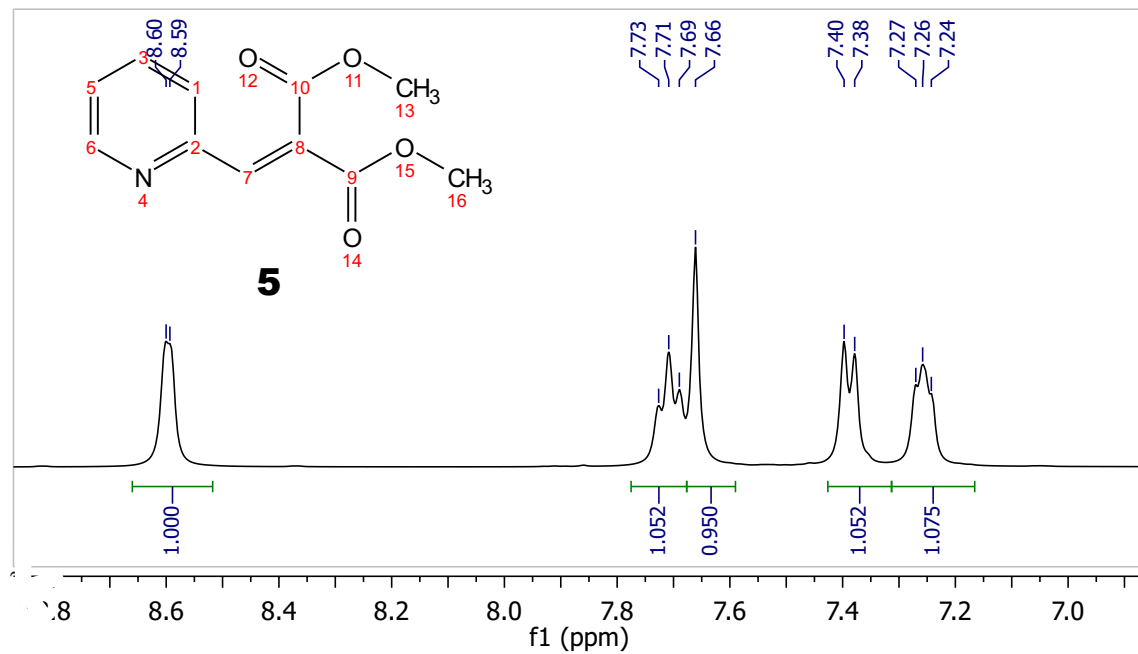
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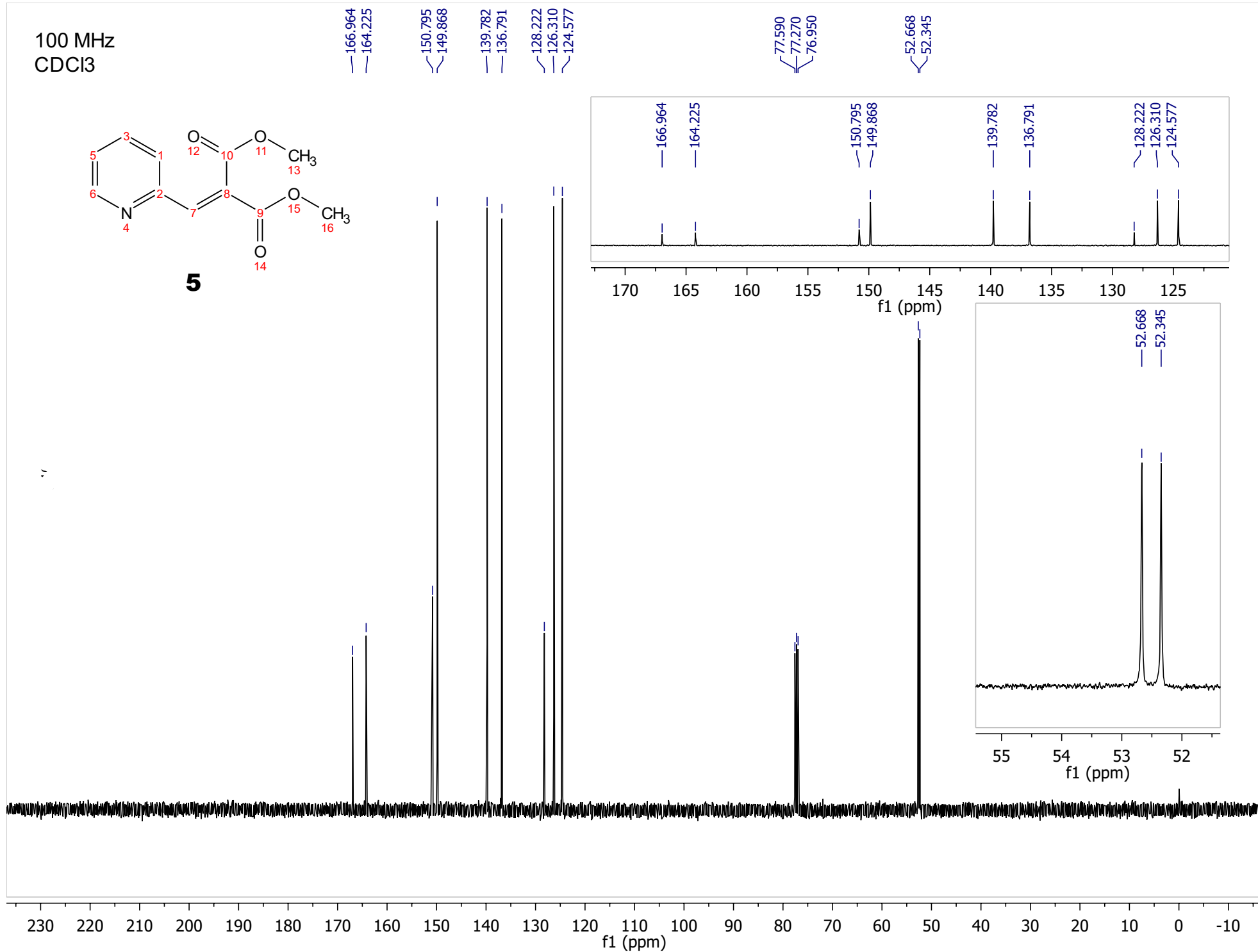
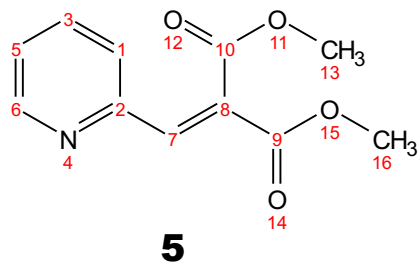
400 MHz
CDCl₃

8.60
8.59
7.73
7.71
7.69
7.66
7.40
7.38
7.27
7.26
7.24

3.90
3.84



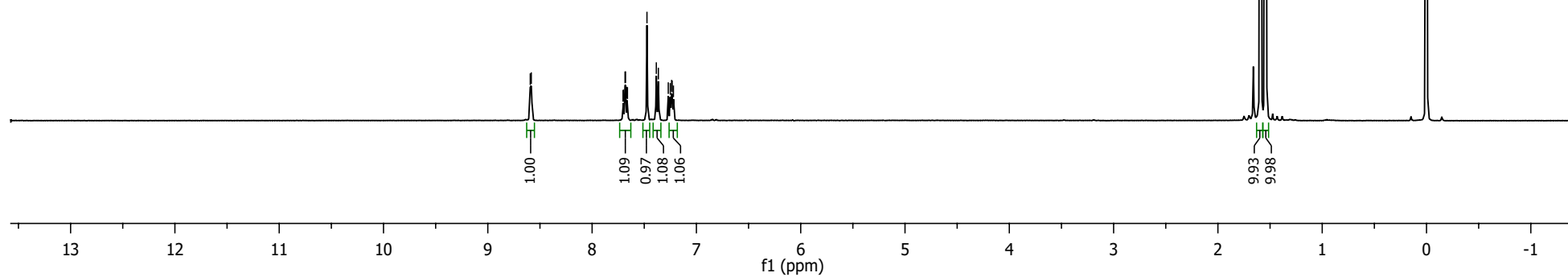
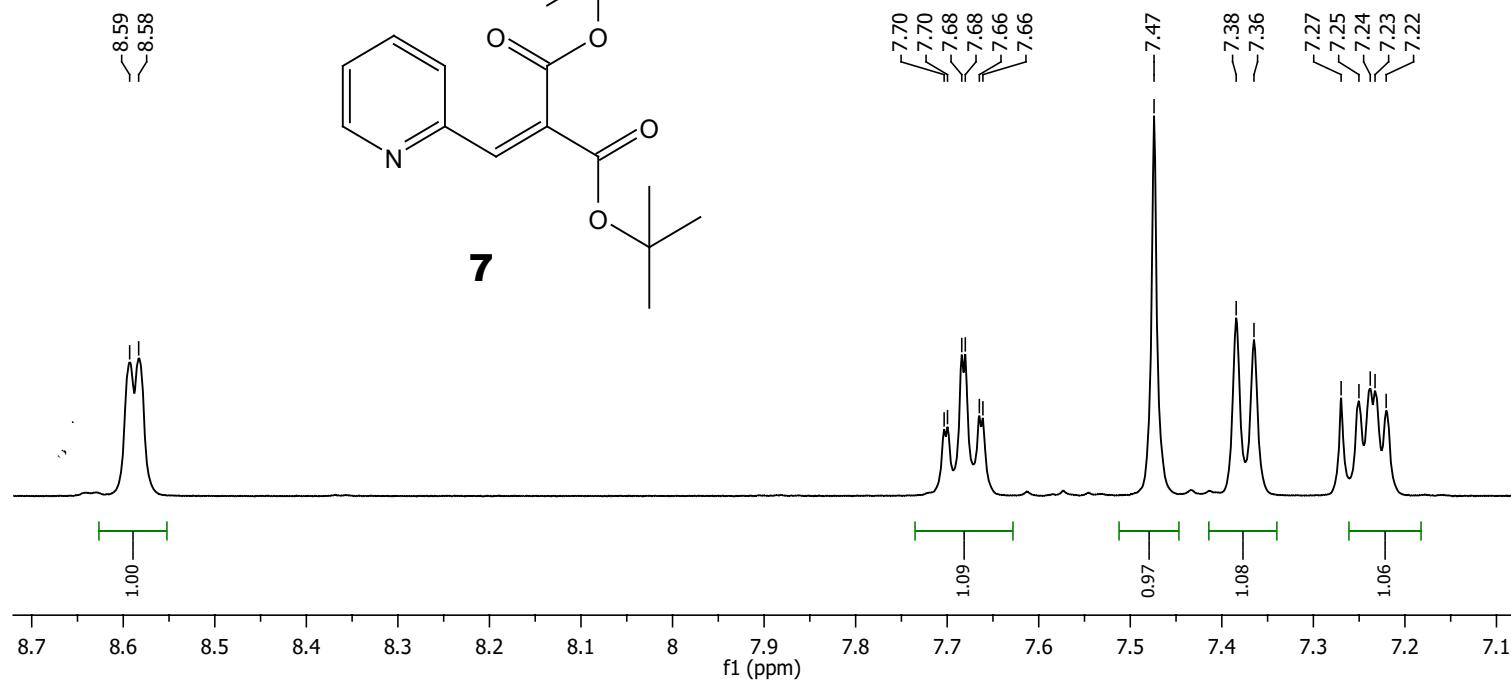
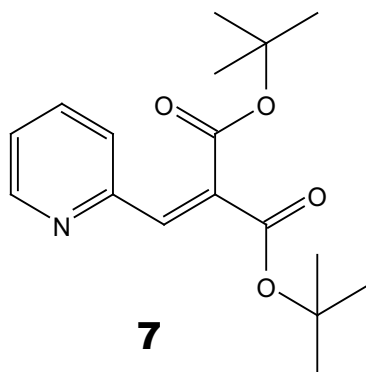
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CDCl₃



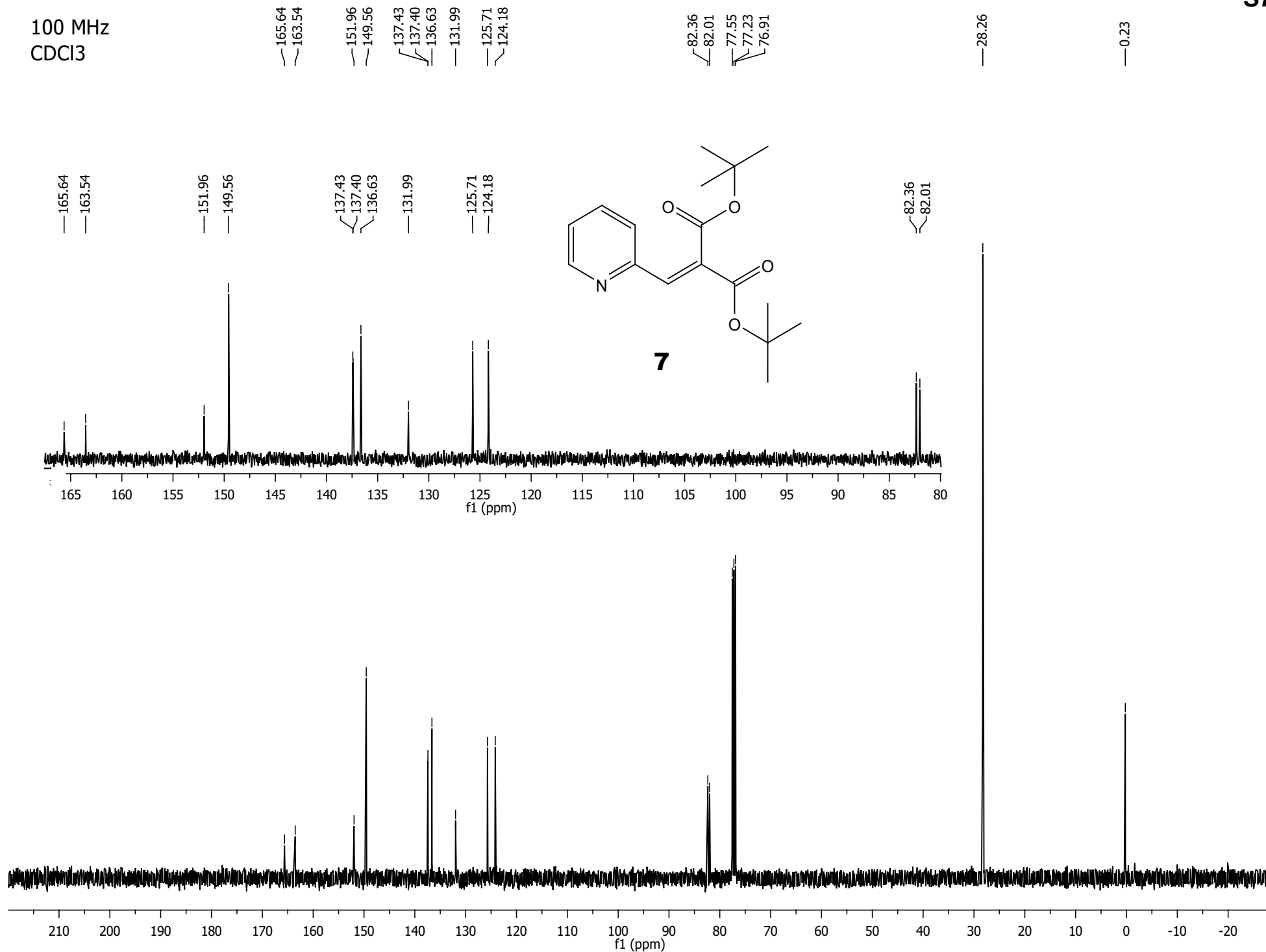
400 MHz
CDCl₃

8.59
8.58
7.70
7.70
7.68
7.68
7.66
7.66
7.47
7.38
7.36
7.27
7.25
7.24
7.23
7.22

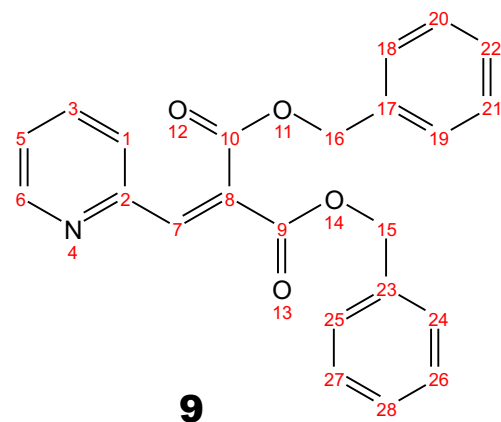
1.59
1.54



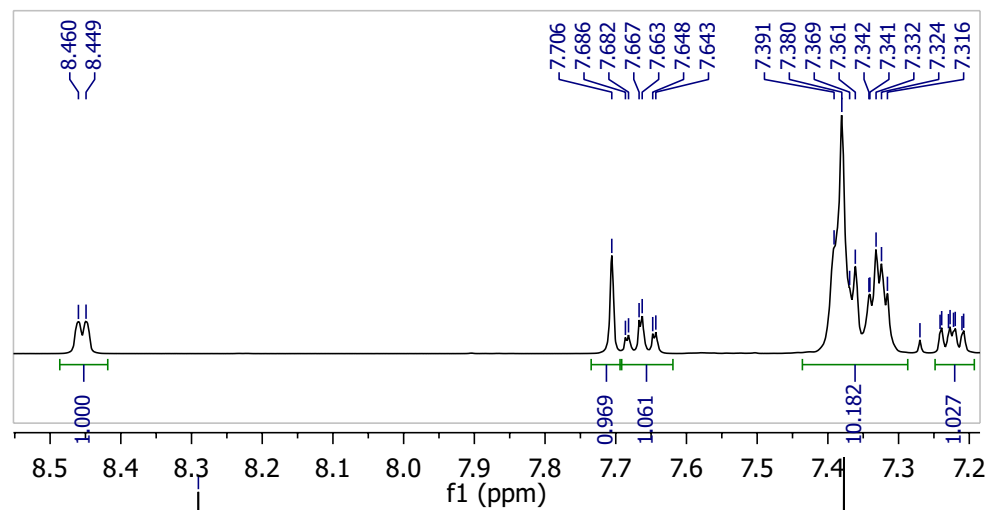
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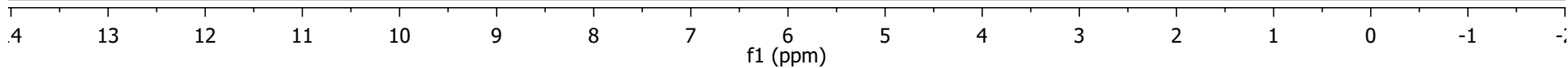
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CDCl₃

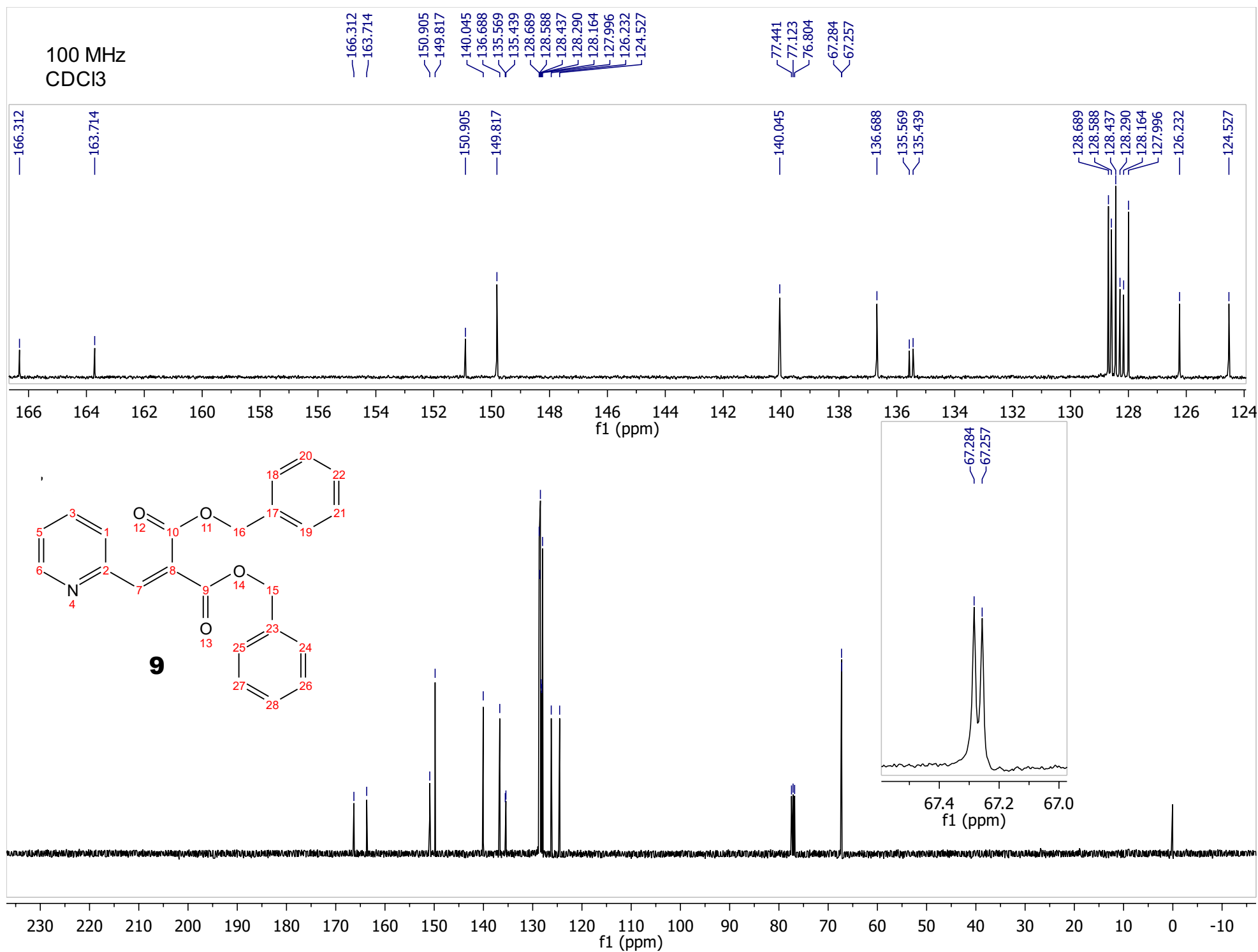


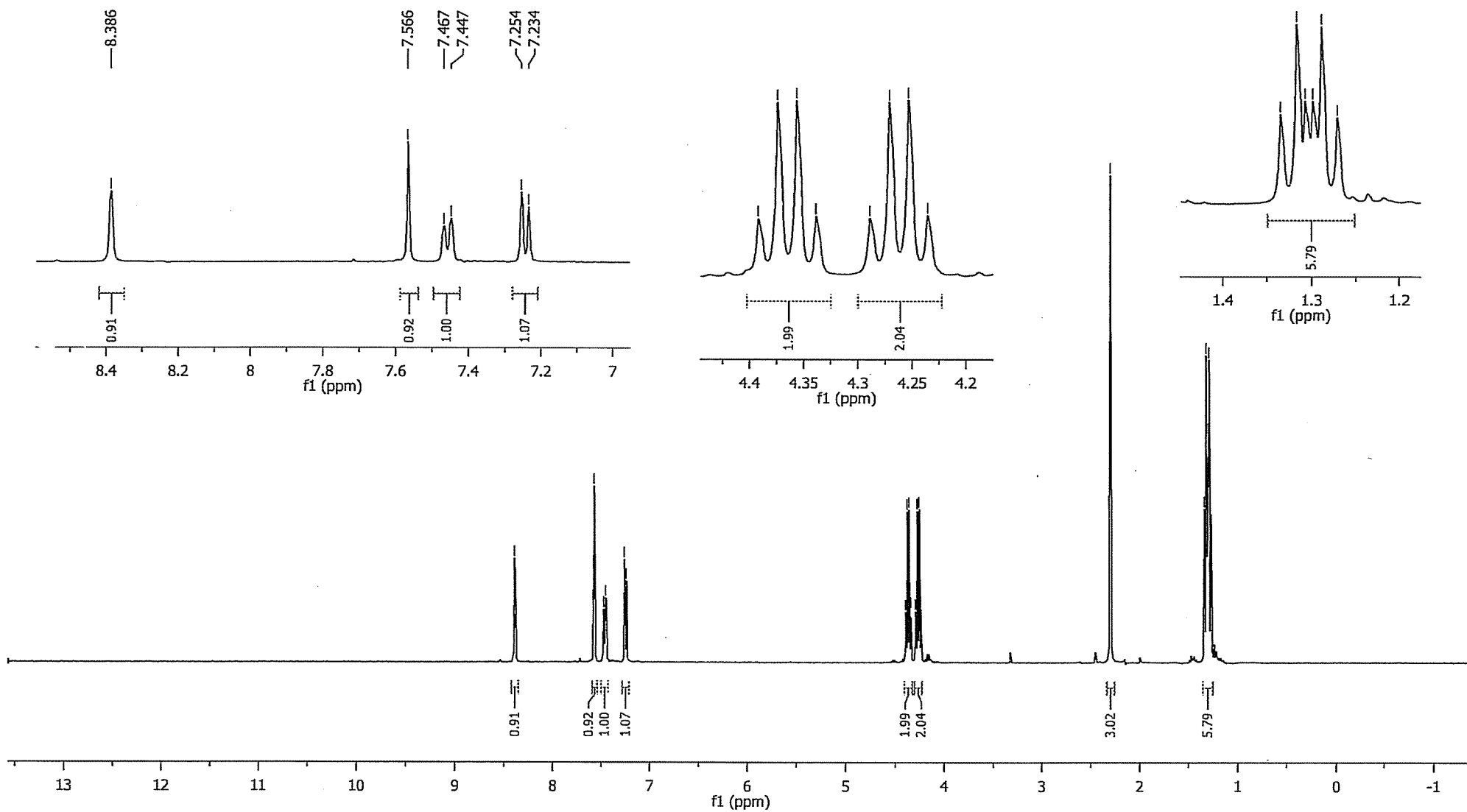
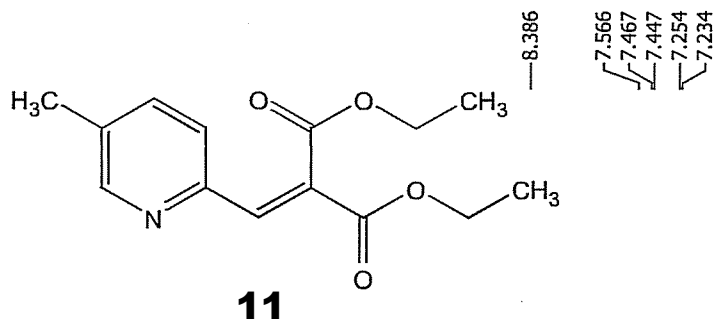
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8.449
7.706
7.667
7.663
7.391
7.380
7.369
7.361
7.342
7.341
7.332
7.324
7.316
7.227
5.322



1.000
0.969
1.061
10.182
1.027
1.921
1.966





400 MHz
CDCl₃

100 MHz
CDCl₃

166.746
164.157

150.532
148.538

139.302
136.963
134.715

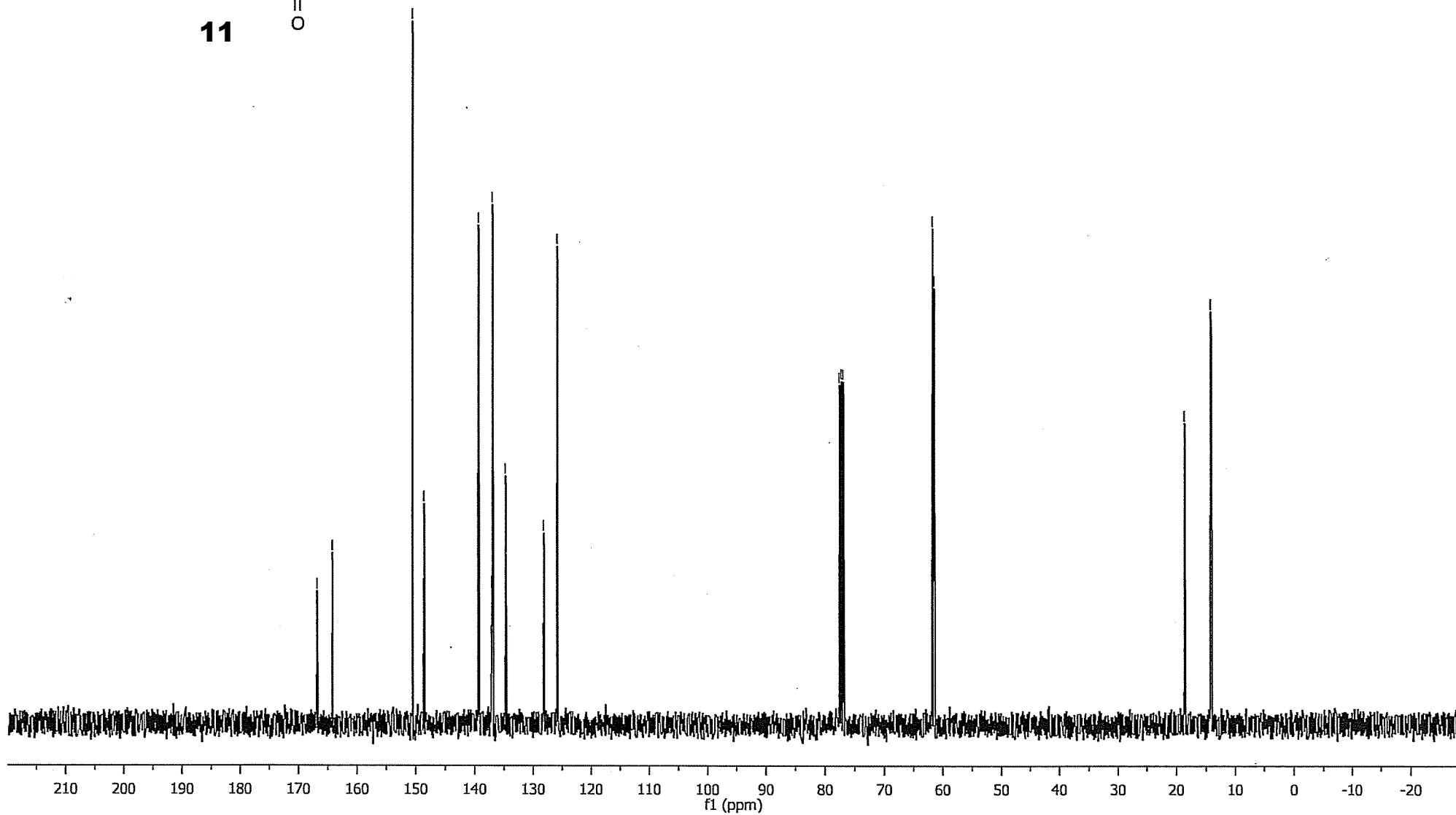
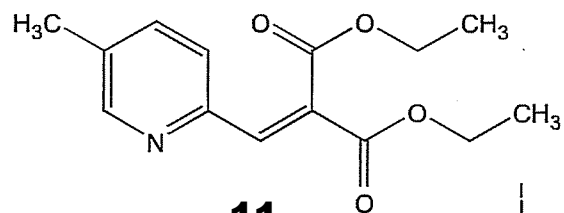
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125.806

77.548
77.230
76.911

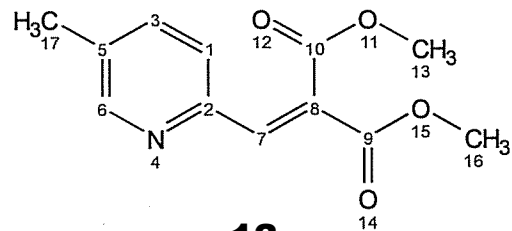
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61.402

18.619
14.221
14.126

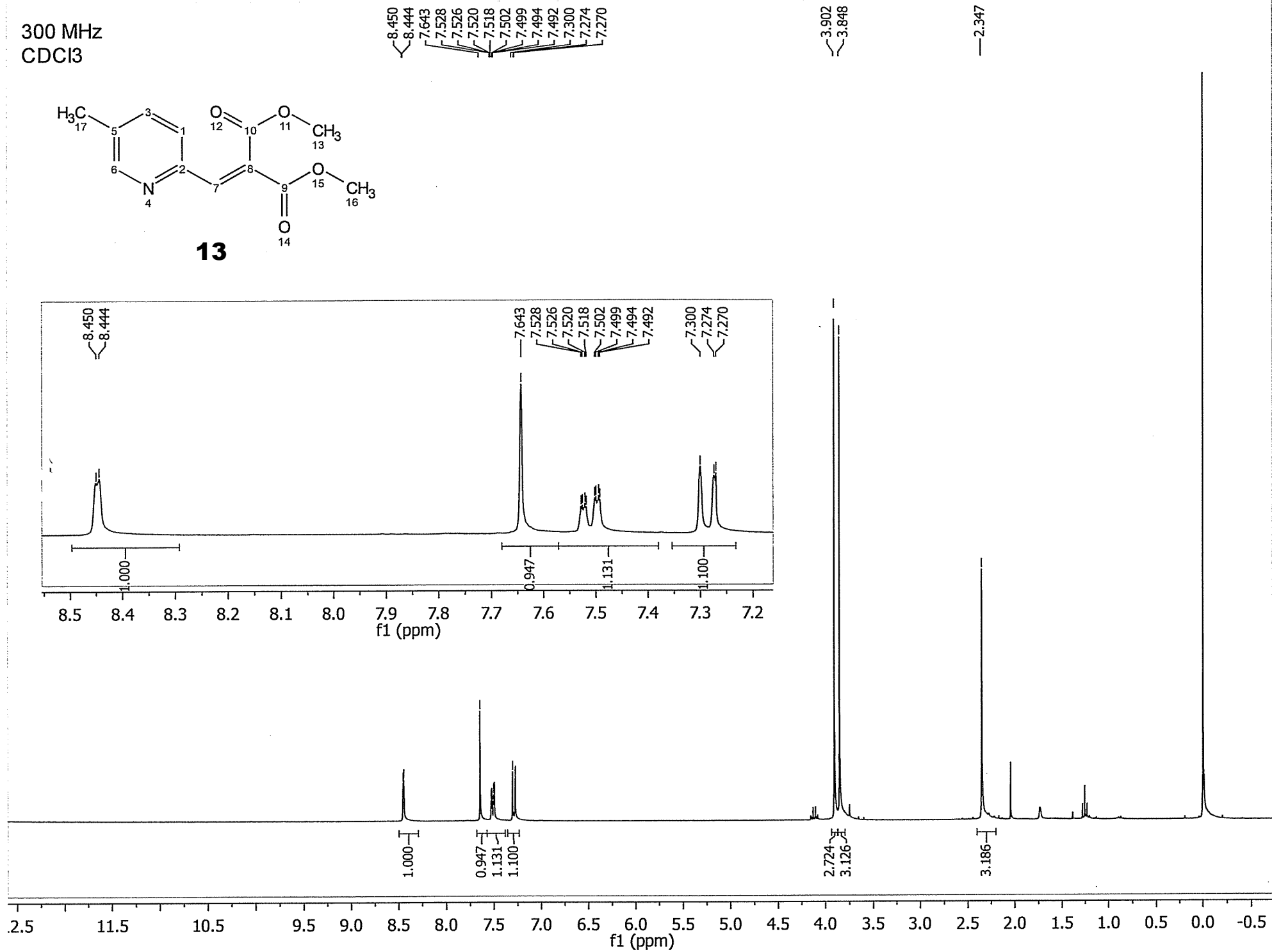
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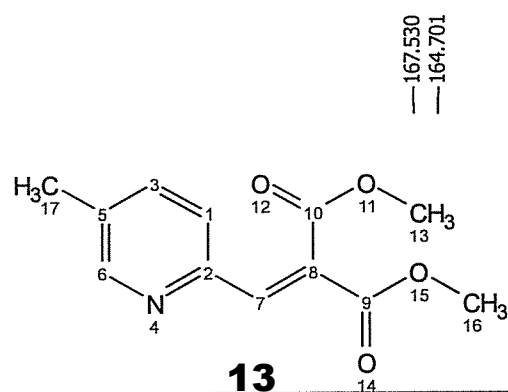


300 MHz
CDCl₃



13





167.530
164.701

150.981
148.481

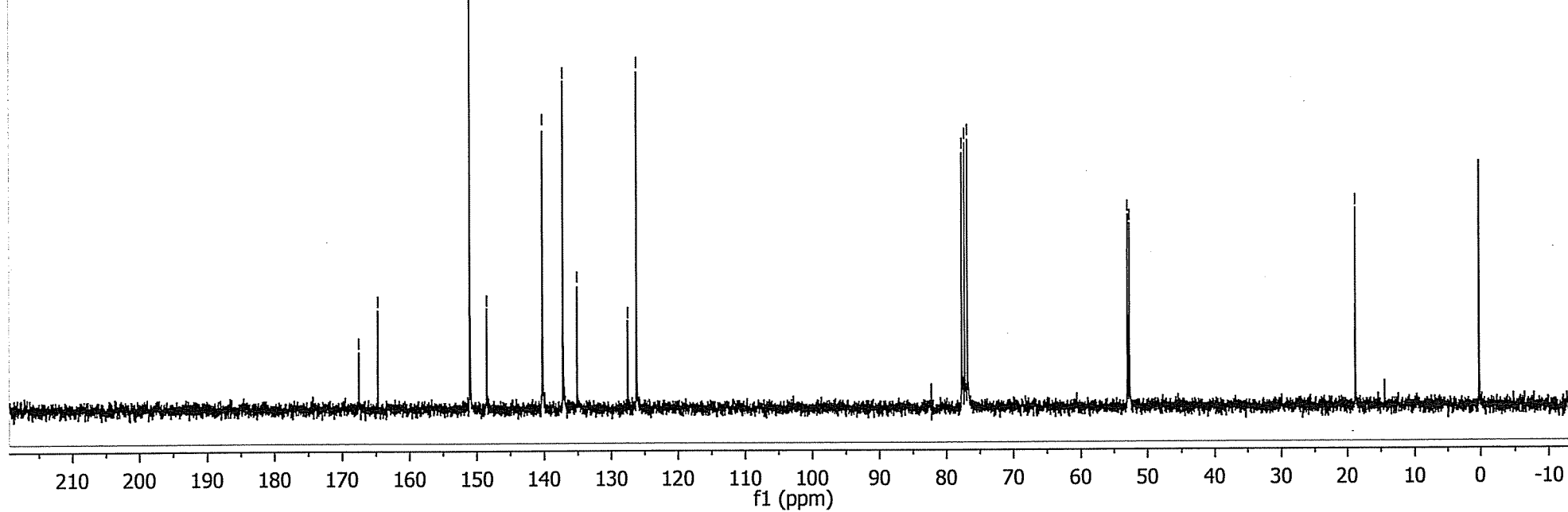
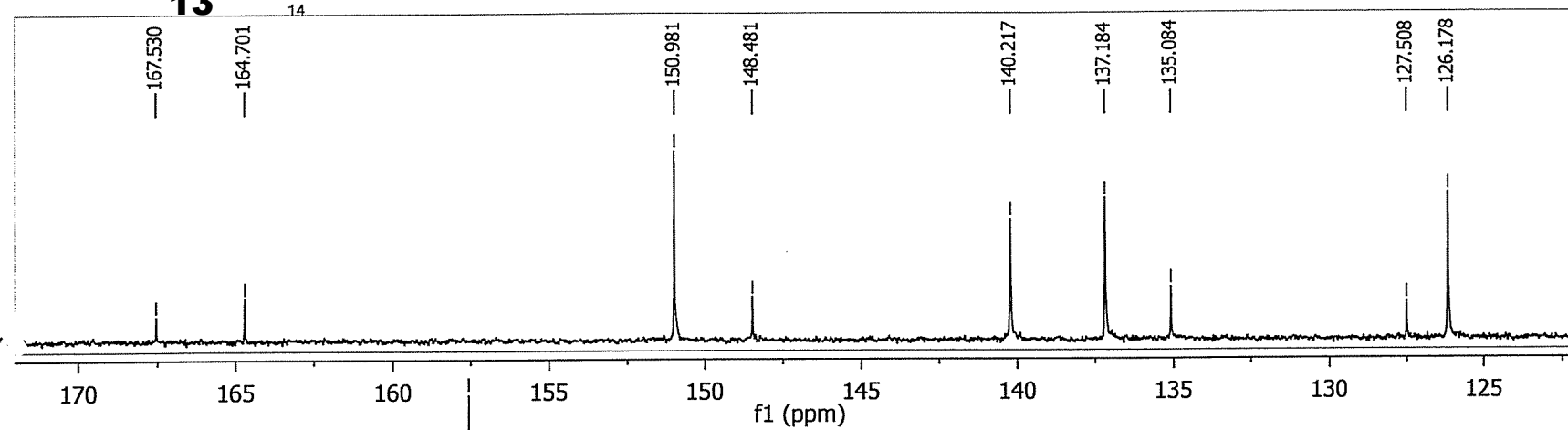
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137.184
135.084

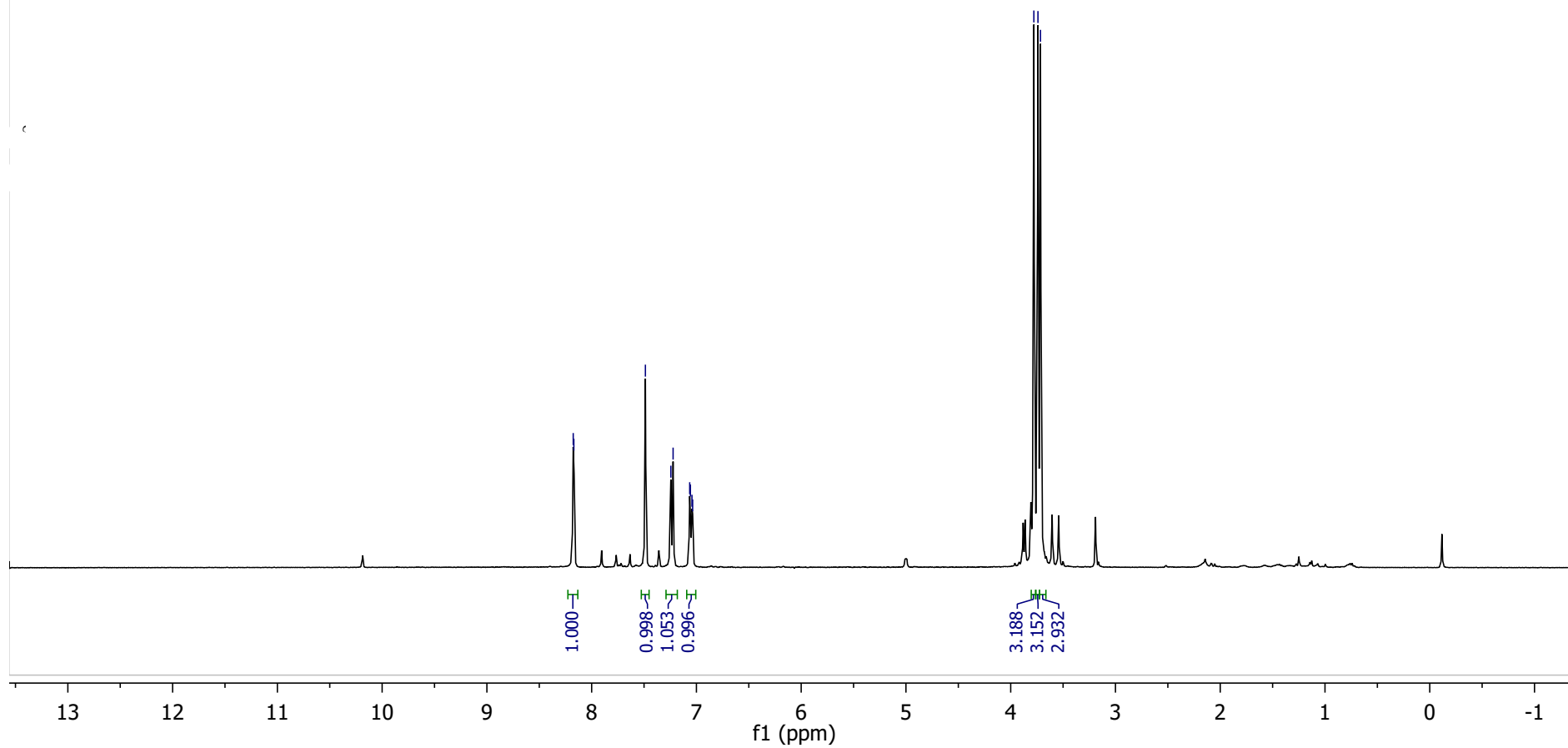
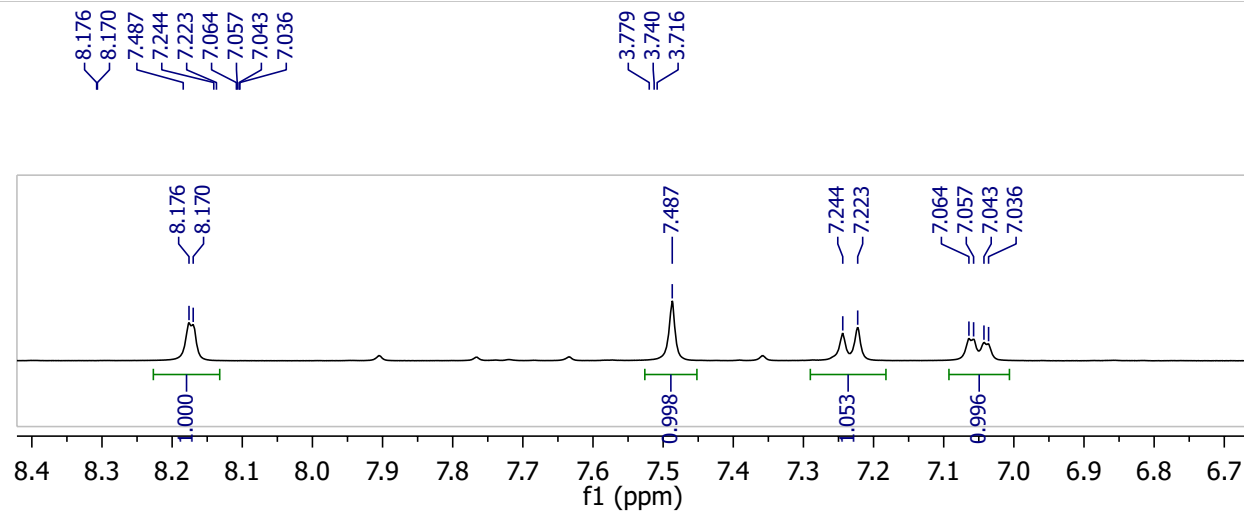
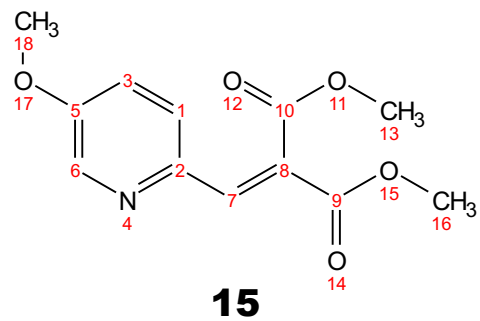
127.508
126.178

77.694
77.270
76.846

52.929
52.650

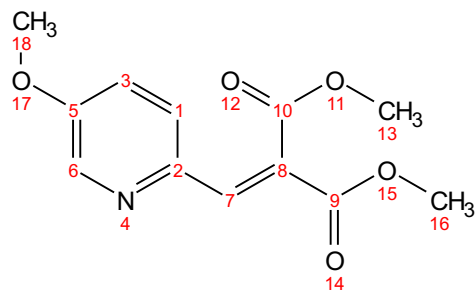
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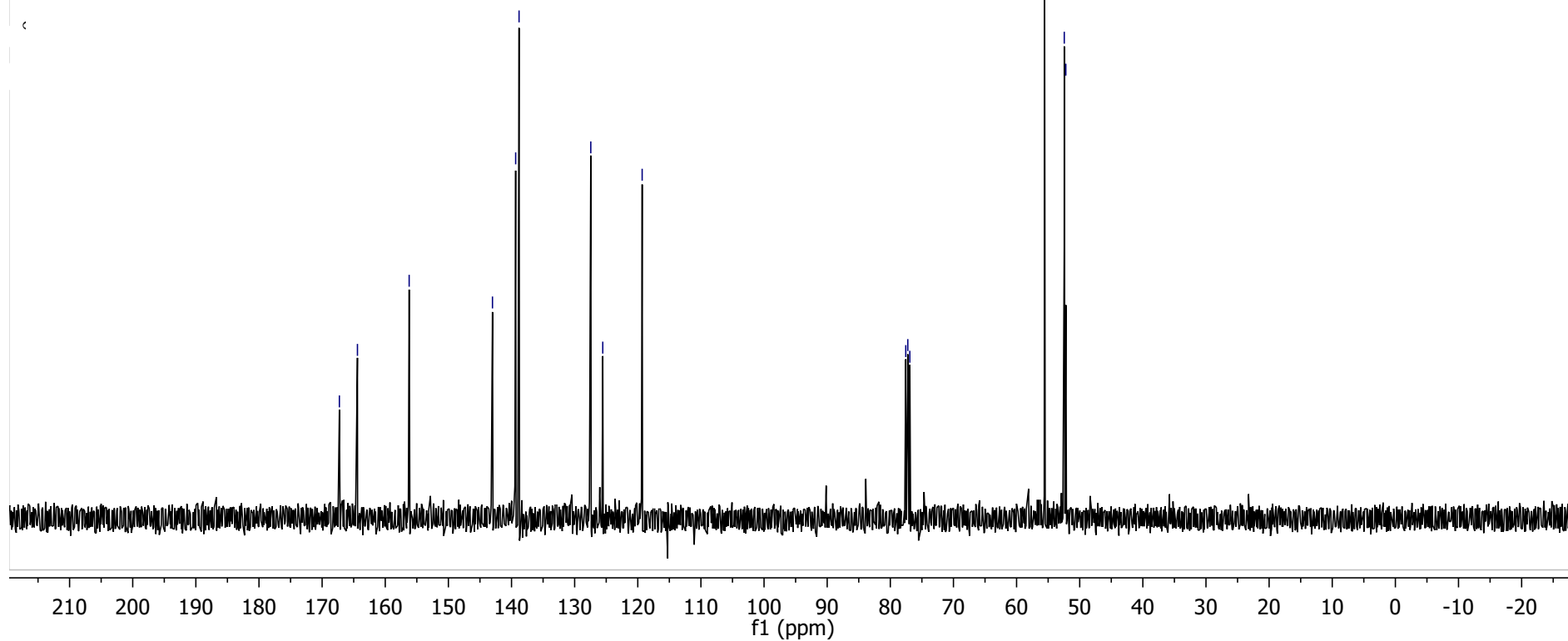


100 MHz
CDCl₃

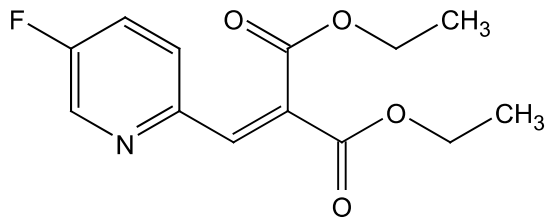
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156.212
143.016
139.343
138.805
127.445
125.546
119.307
77.551
77.230
76.910
55.561
52.446
52.199



15



300 Mhz
CDCl₃



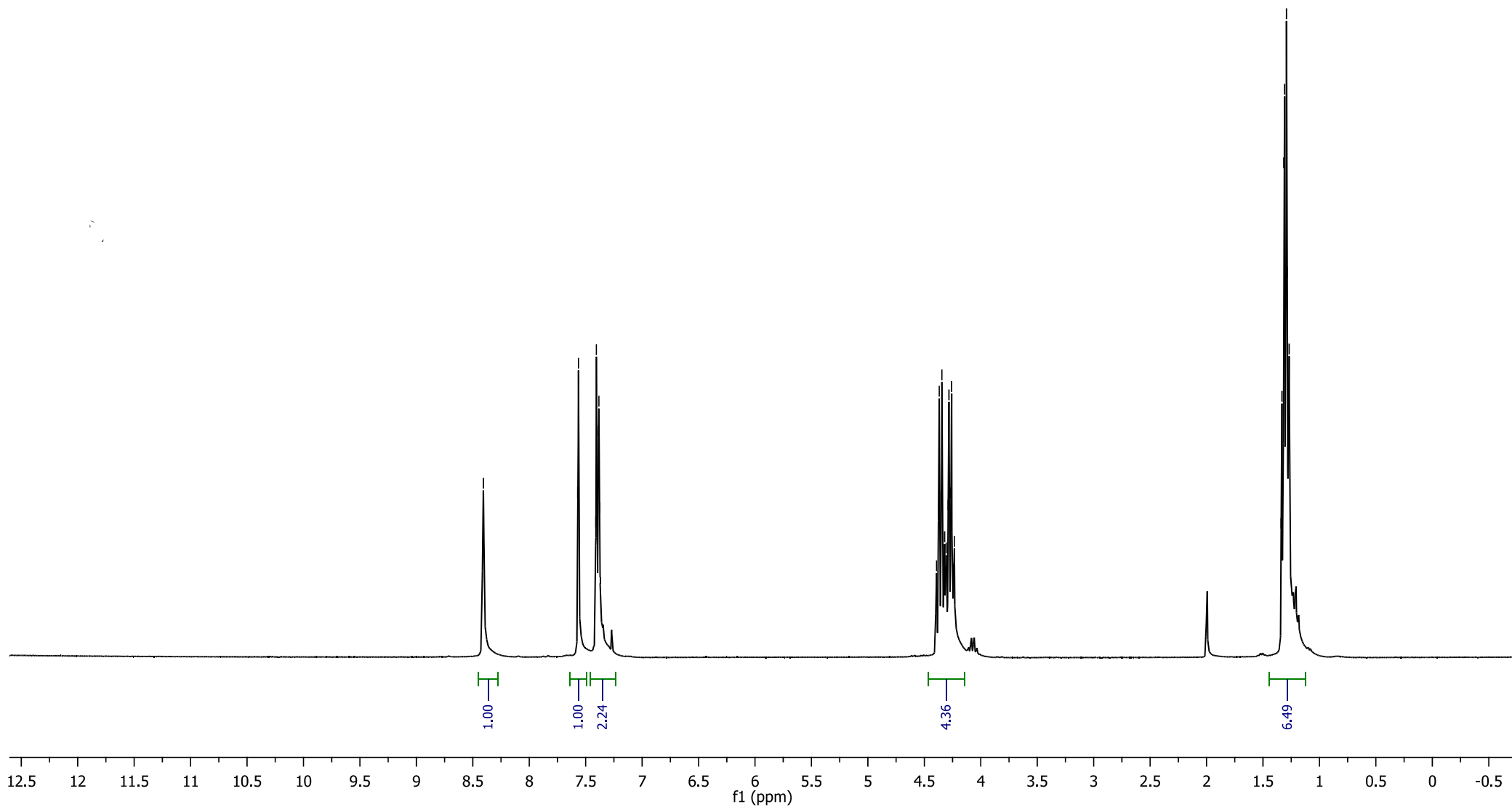
17

8.4064
7.5708
7.5639
7.4105
7.4052
7.3999
7.3861
7.3828
7.3751

4.3918
4.3751
4.3678
4.3515
4.3442
4.3275
4.3202
4.3137
4.3060
4.2897
4.2819
4.2657
4.2583
4.2421
4.2343

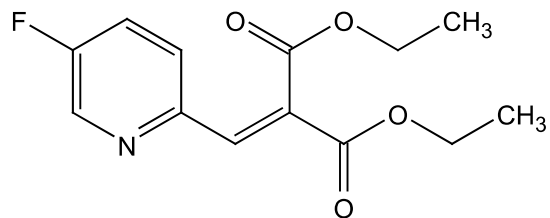
1.3394
1.3321
1.3235
1.3158
1.3085
1.2999
1.2918
1.2844
1.2763
1.2682

S16

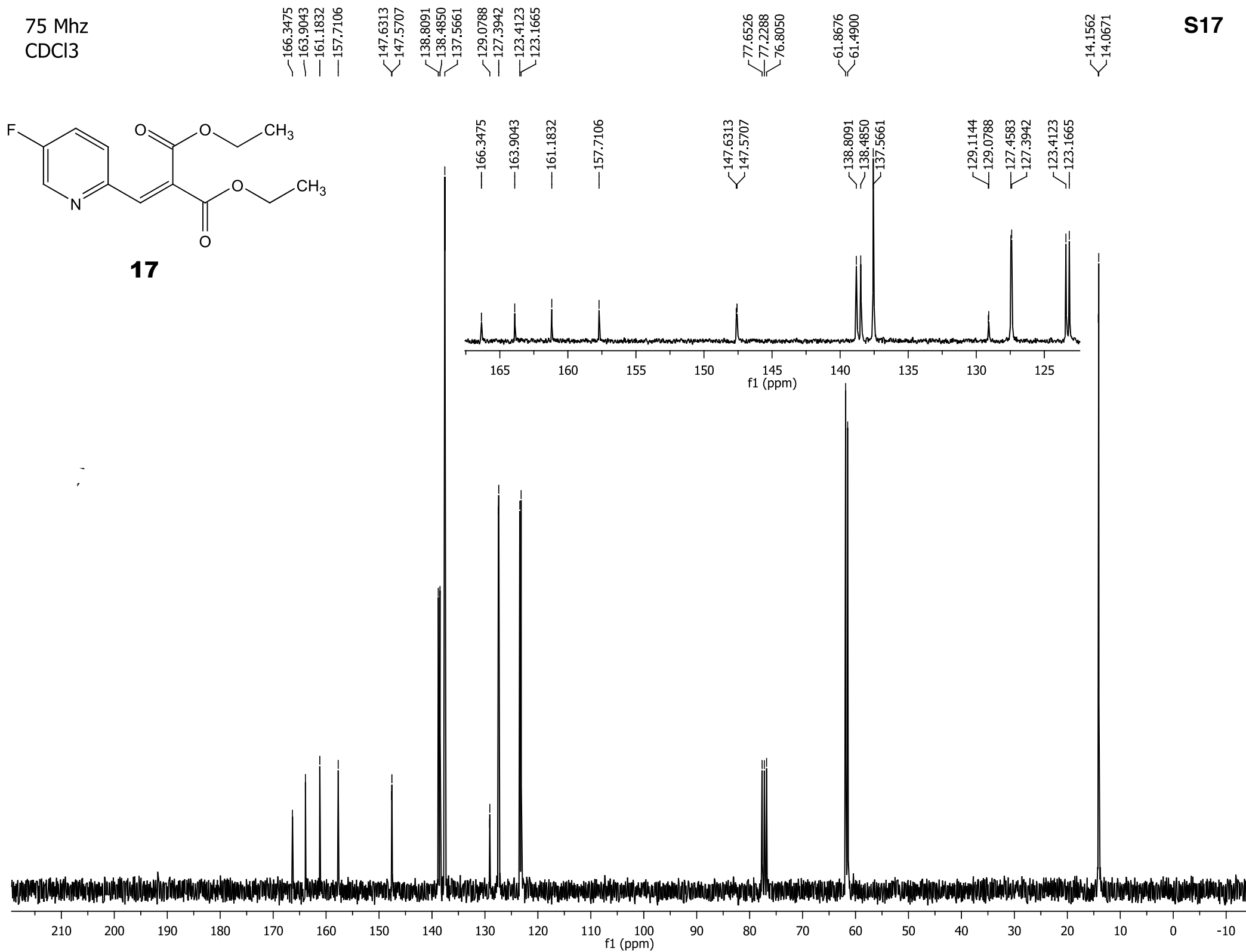


75 Mhz
CDCl3

S17

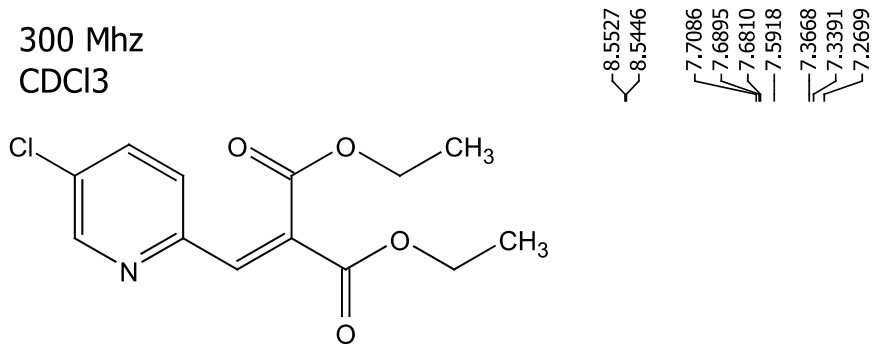


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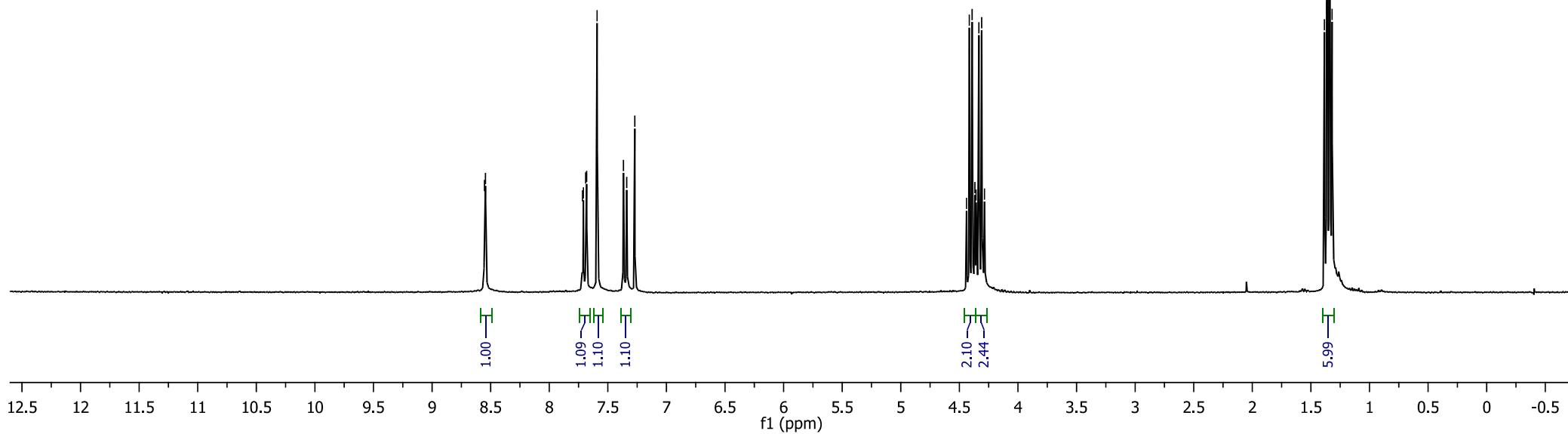
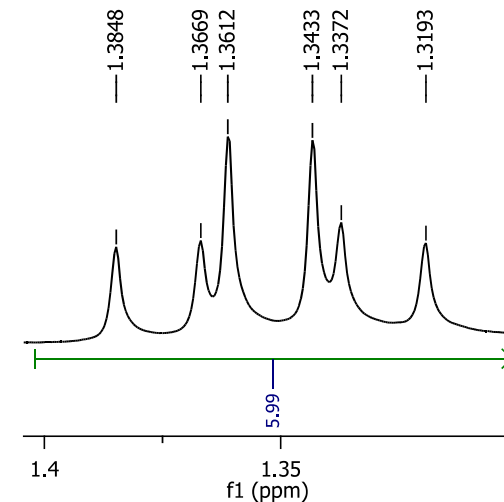
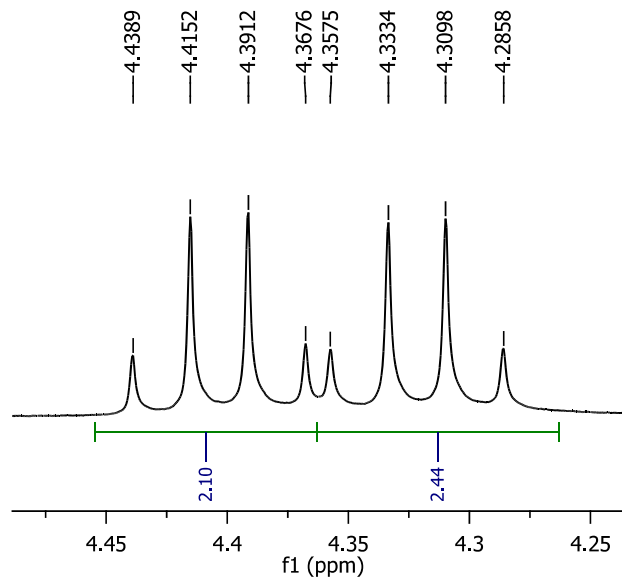
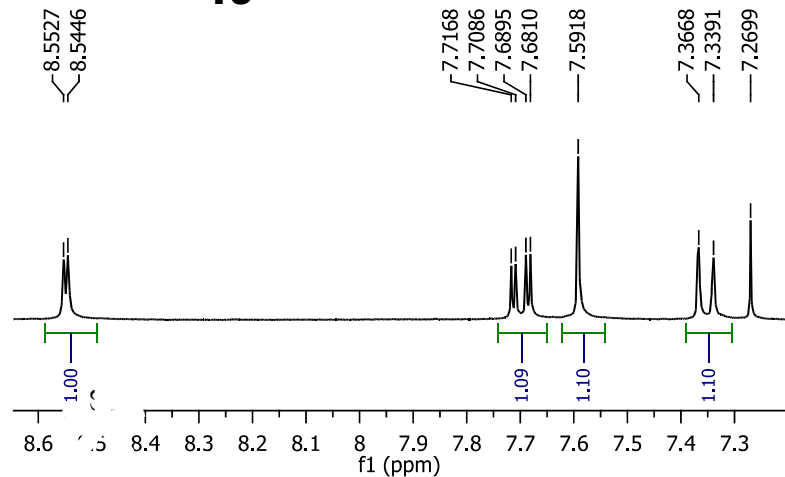


300 Mhz
CDCl3

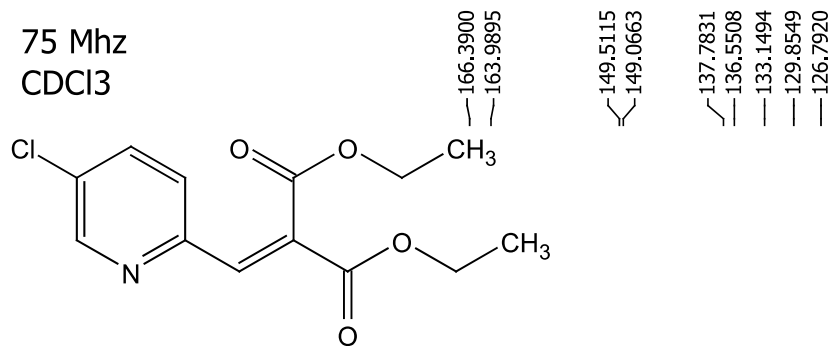
S18



19



75 Mhz
CDCl₃



19

149.5115
149.0663

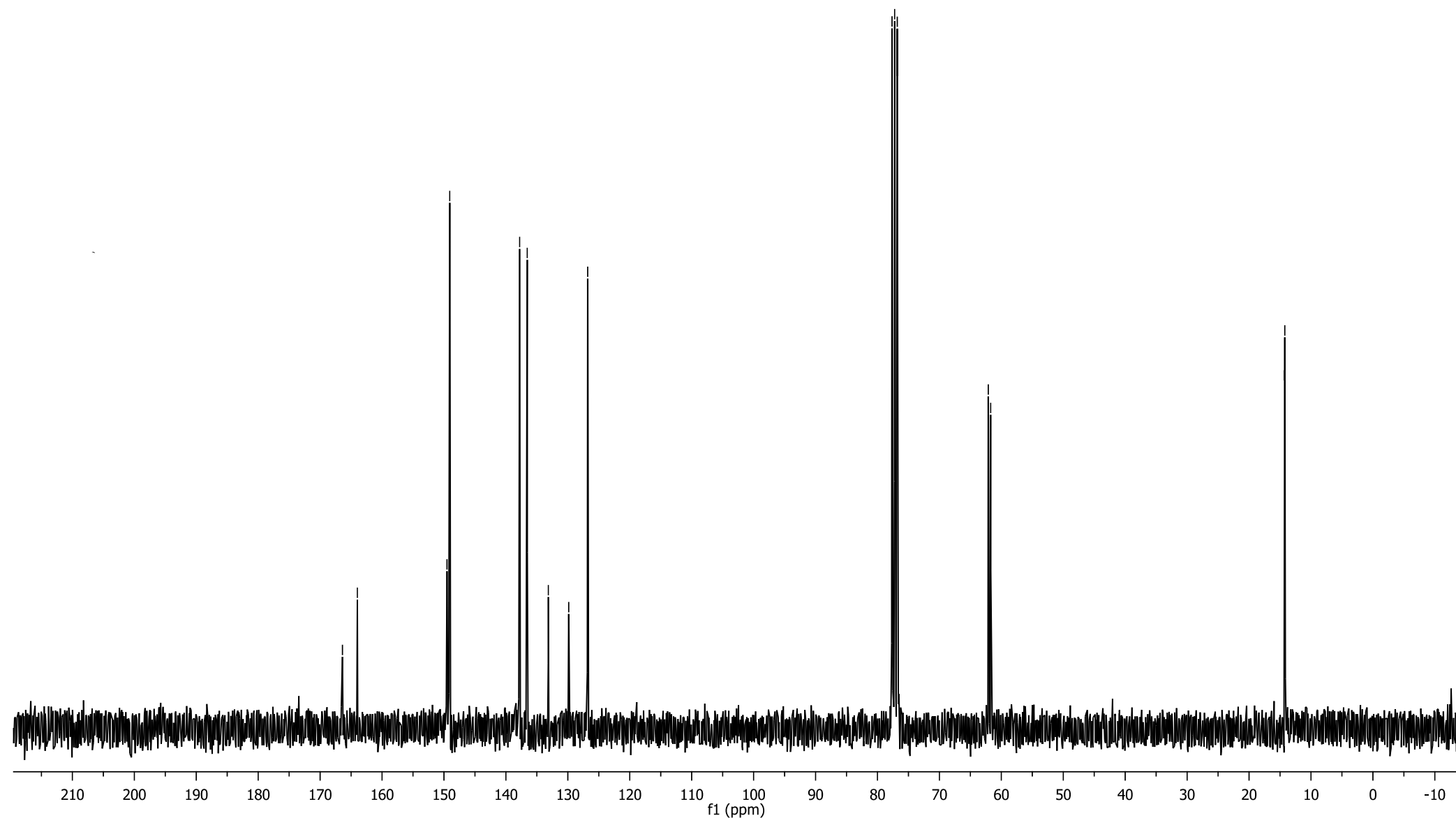
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136.5508
133.1494
129.8549
126.7920

77.6524
77.4814
77.2285
76.8047

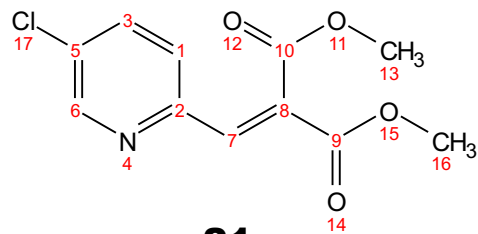
62.1059
61.7141

14.3090
14.2307

S19



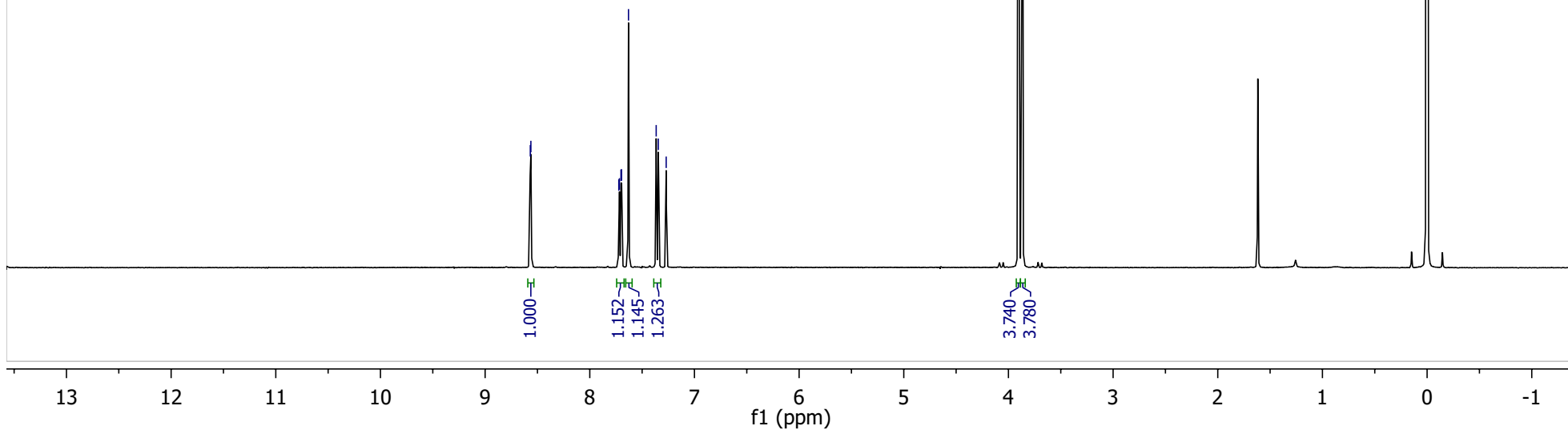
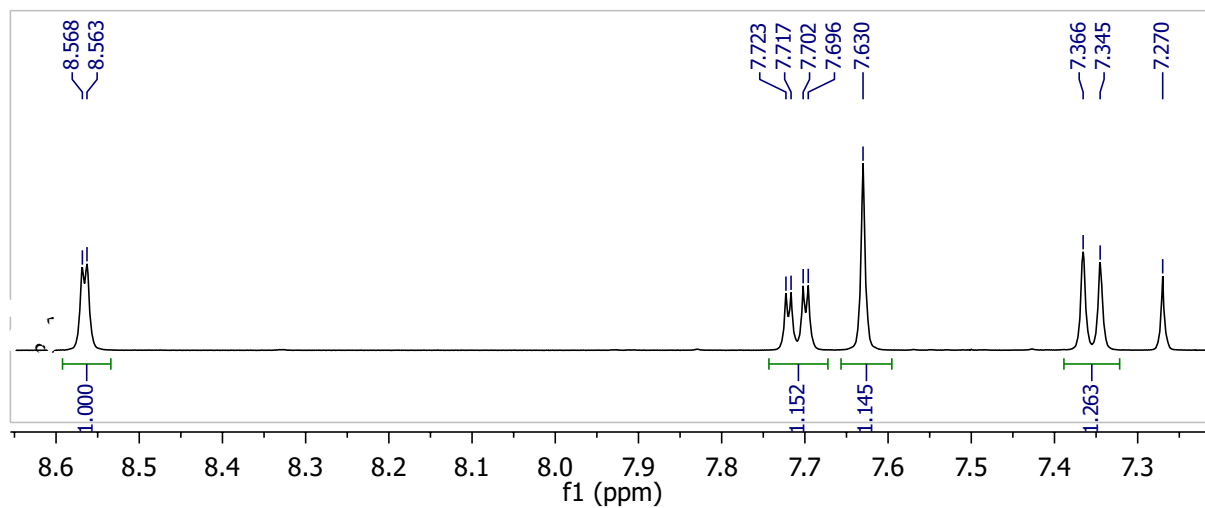
400 MHz
CDCl₃



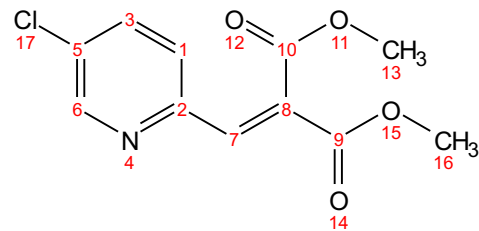
21

8.568
8.563
7.723
7.717
7.702
7.696
7.630
7.366
7.345
7.270

3.904
3.869



100 MHz
CDCl₃

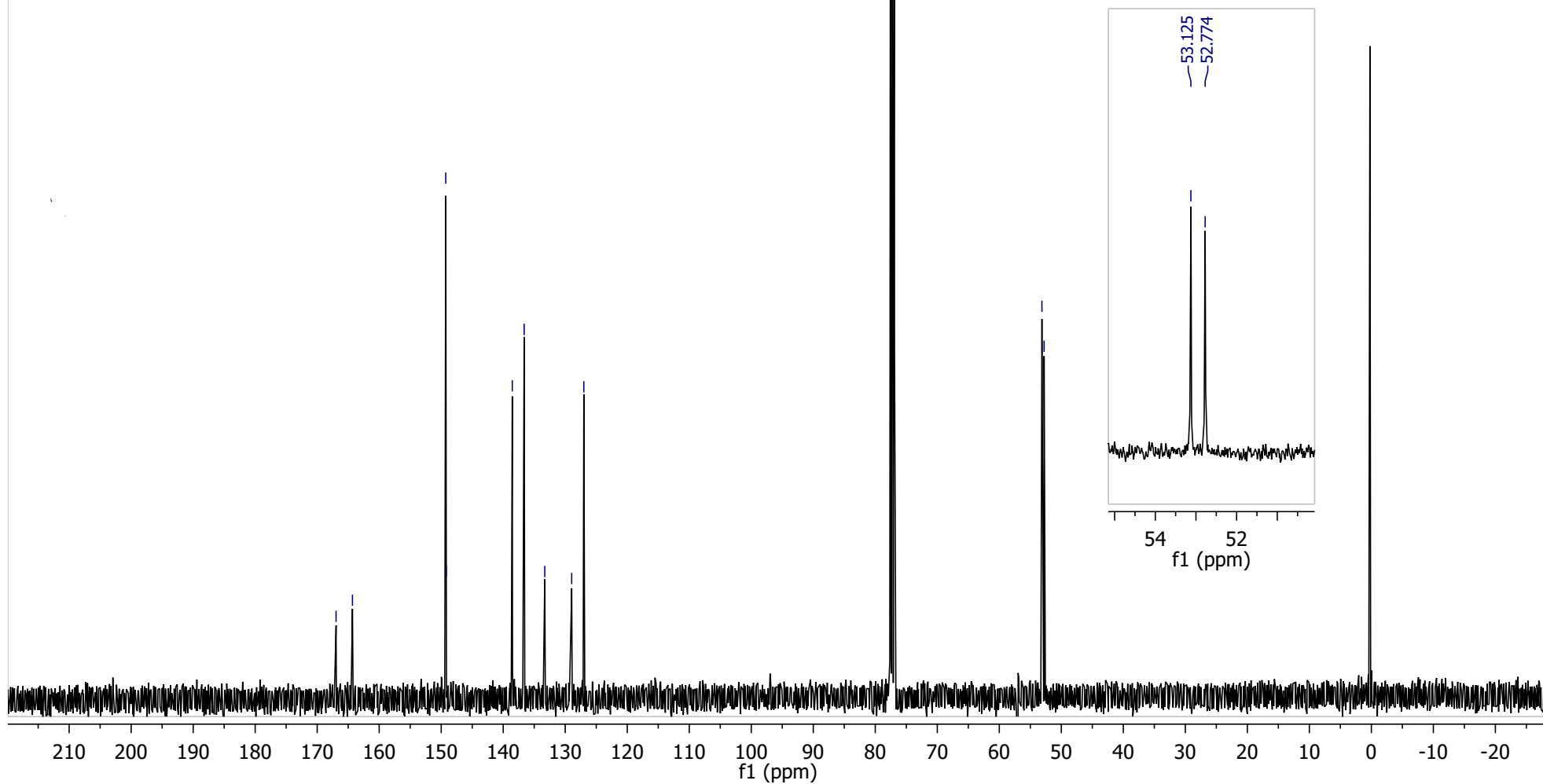


21

166.956
164.315
149.267
149.181
138.539
136.623
133.302
128.958
127.001

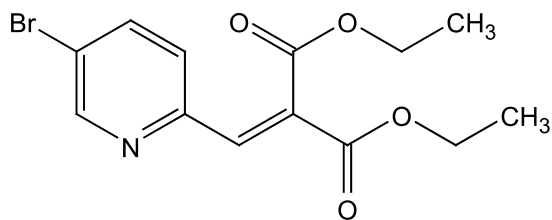
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77.230
76.913

53.125
52.774

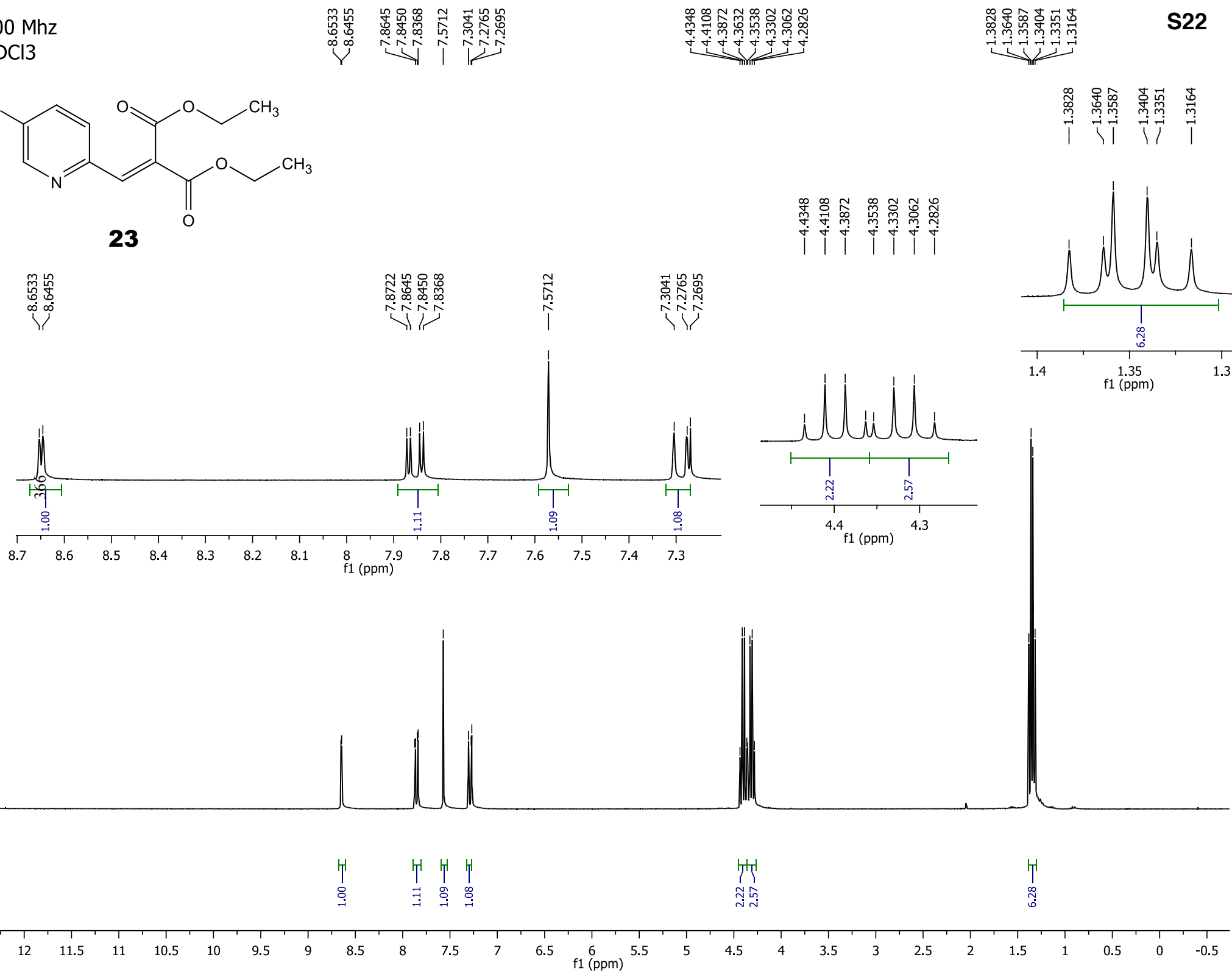


300 Mhz
CDCl3

S22



23



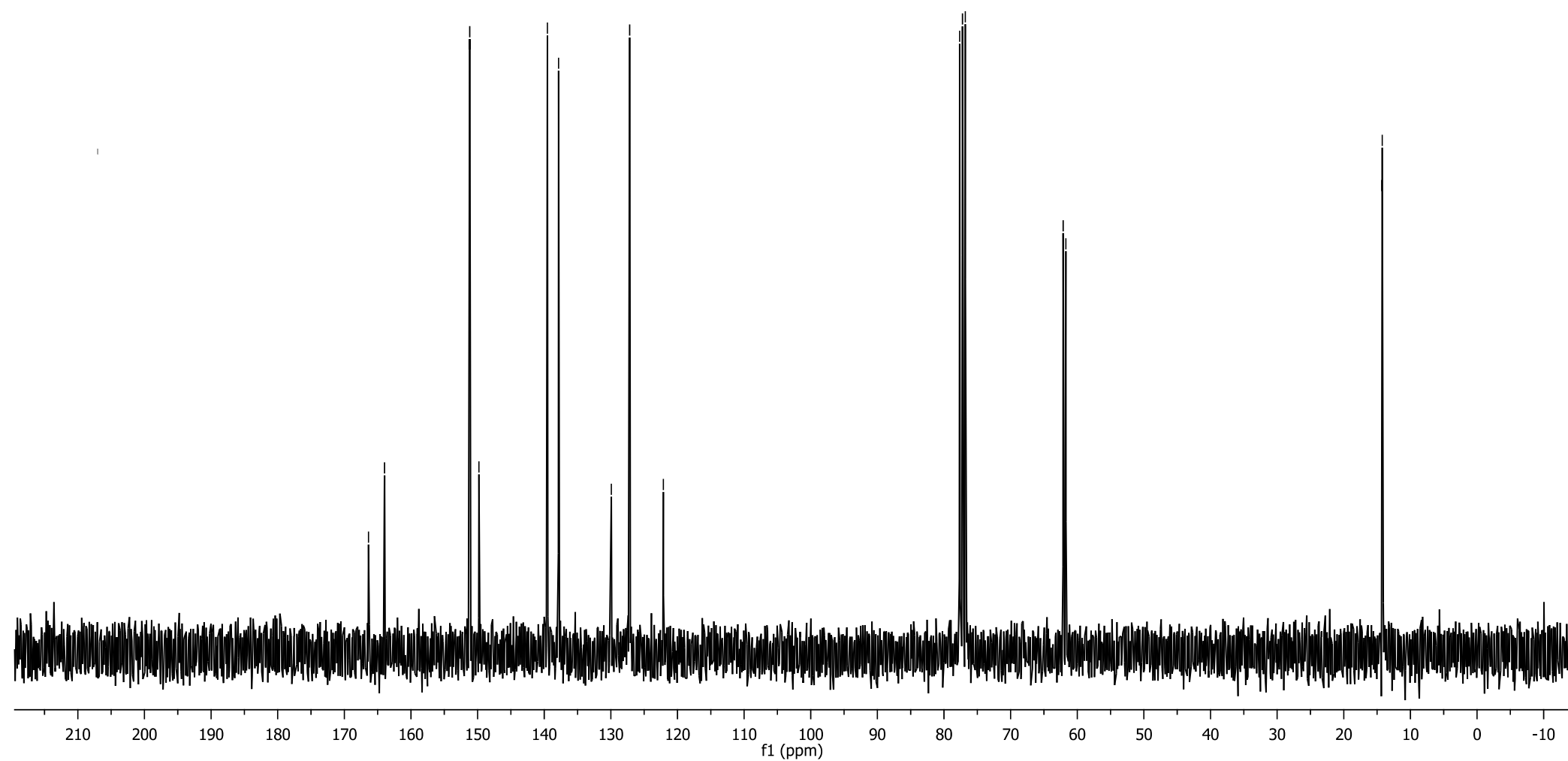
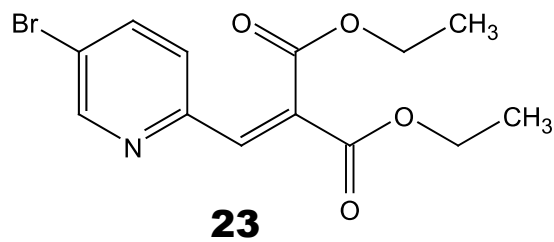
75 Mhz
CDCl₃

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137.8469
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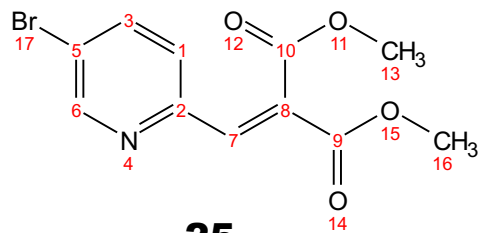
77.6520
77.2282
76.8044
62.1127
61.7103

14.2980
14.2232

S23



100 MHz
CDCl₃



166.928
164.298

151.390
149.484

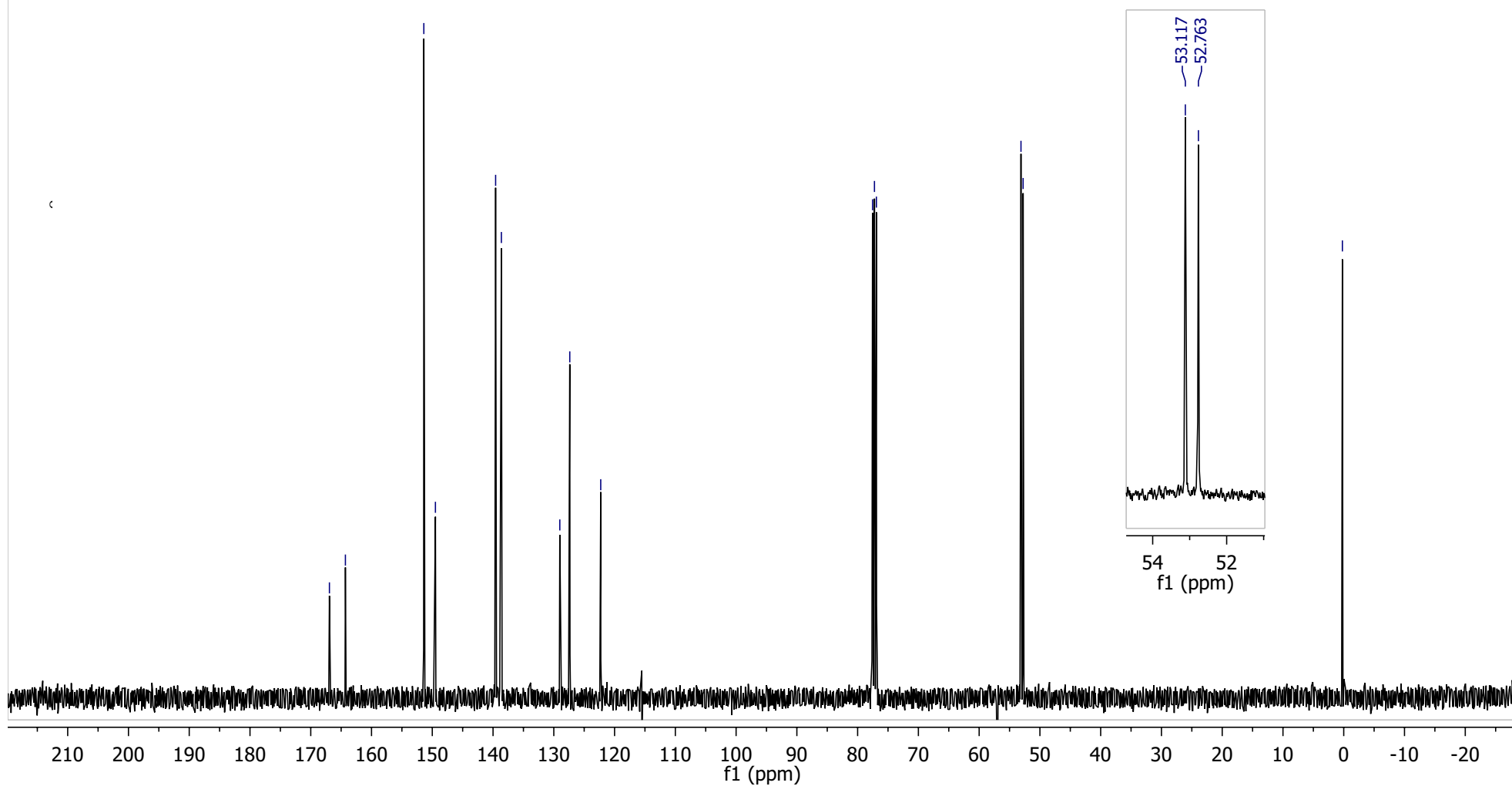
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138.627

129.013
127.373
122.278

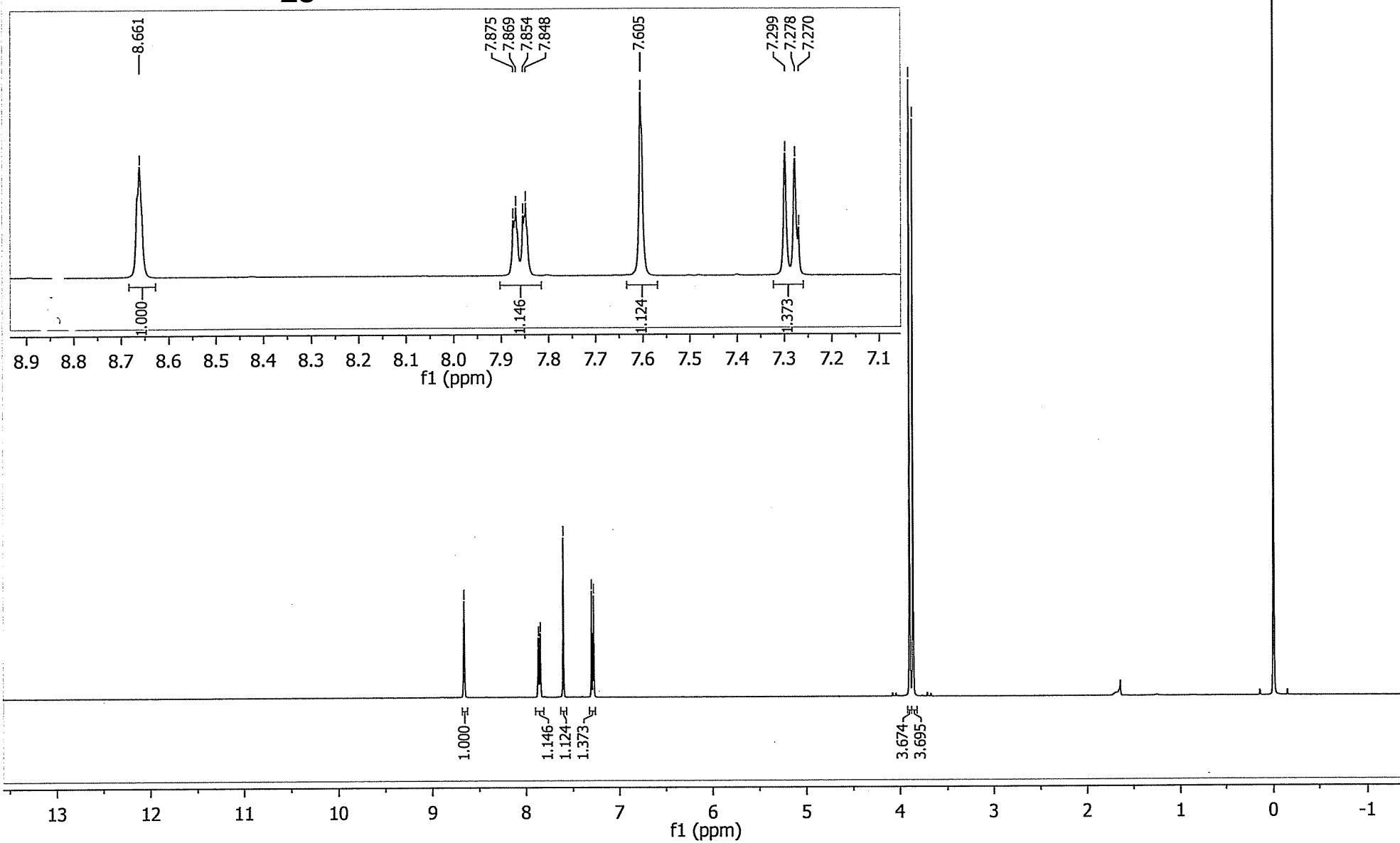
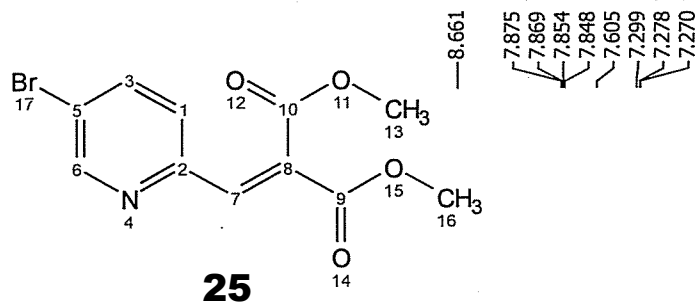
77.547
77.230
76.912

53.117
52.763

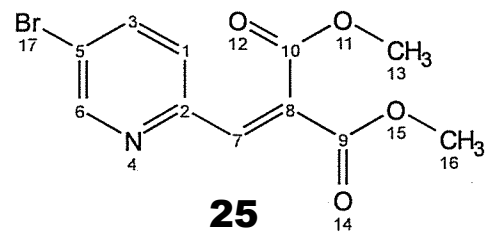
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400 MHz
CDCl₃



100 MHz
CDCl₃



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164.298

151.390
149.484

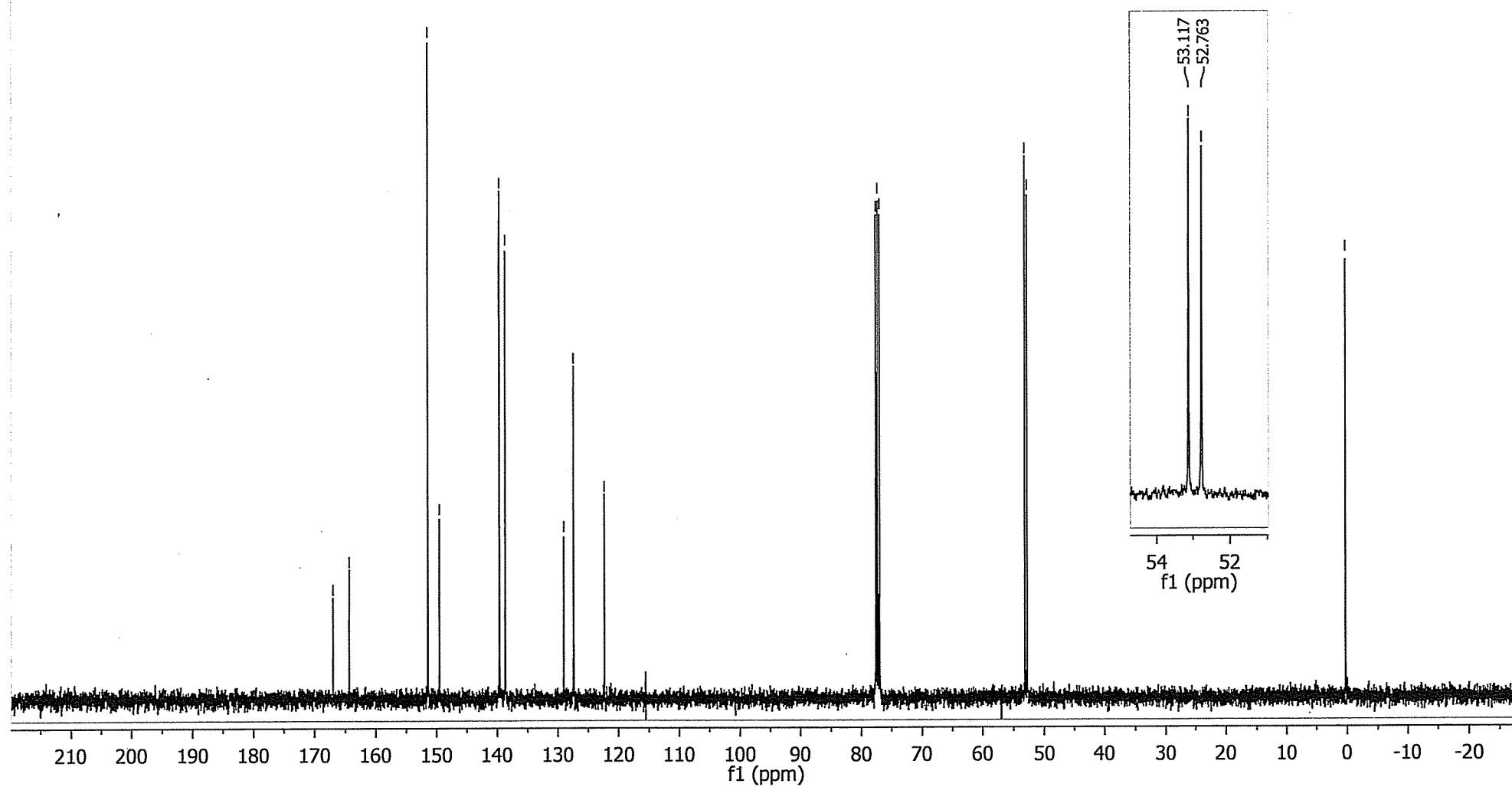
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138.627

129.013
127.373
122.278

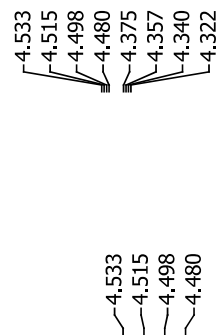
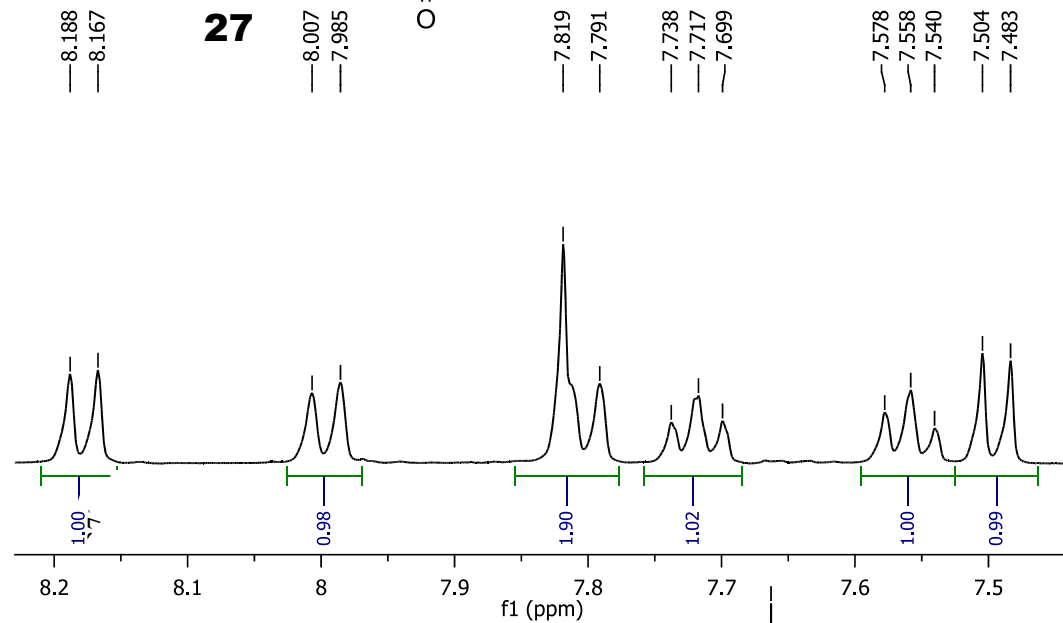
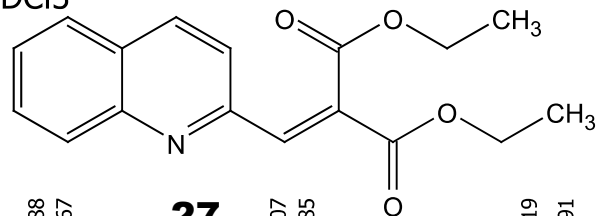
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77.230
76.912

53.117
52.763

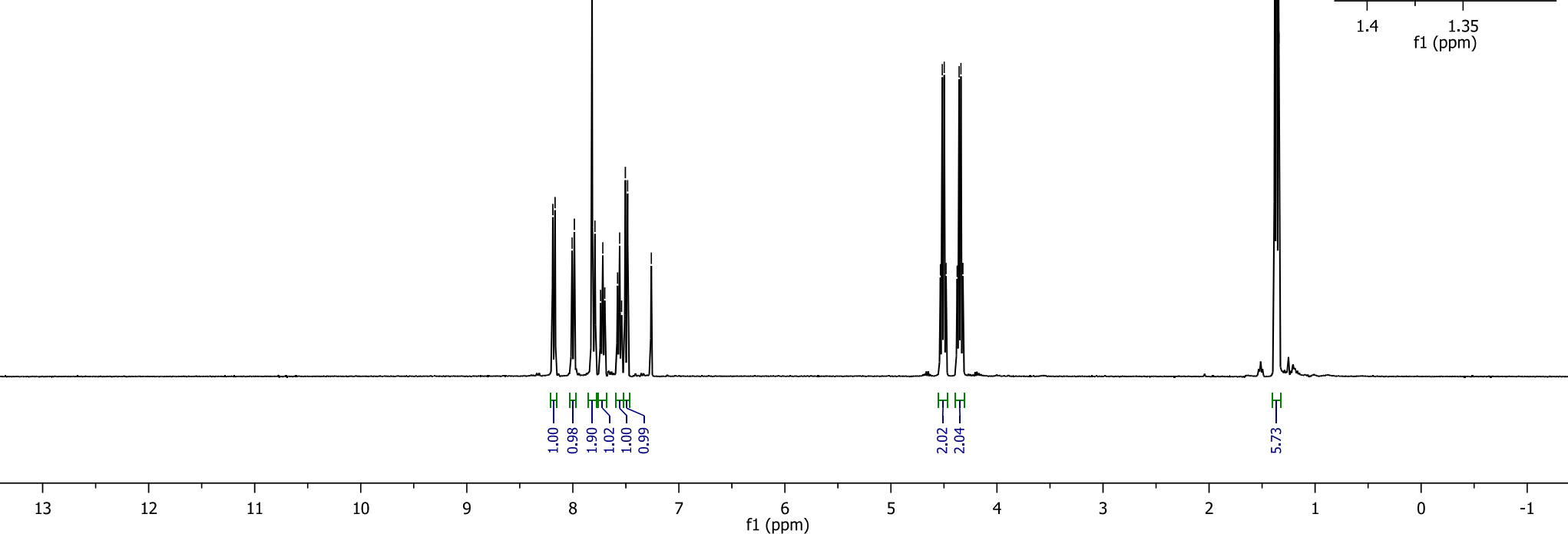
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400 MHz
CDCl₃

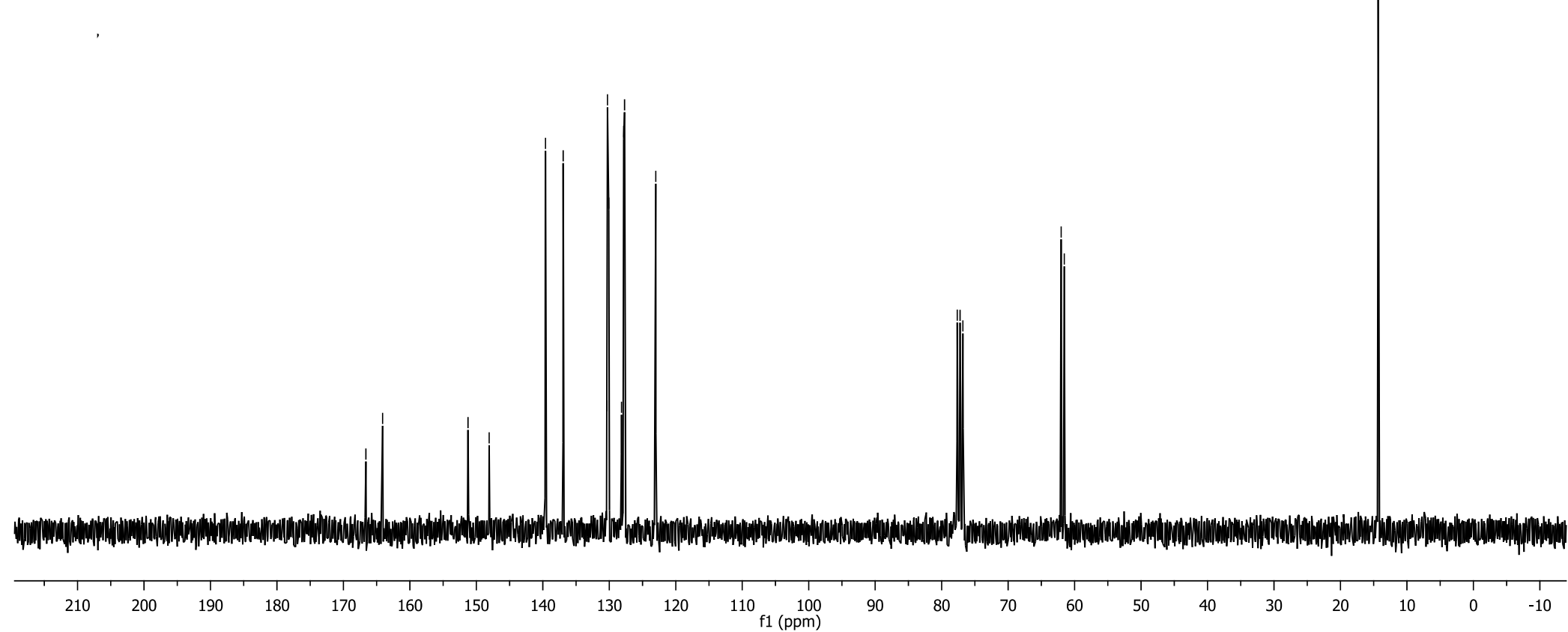
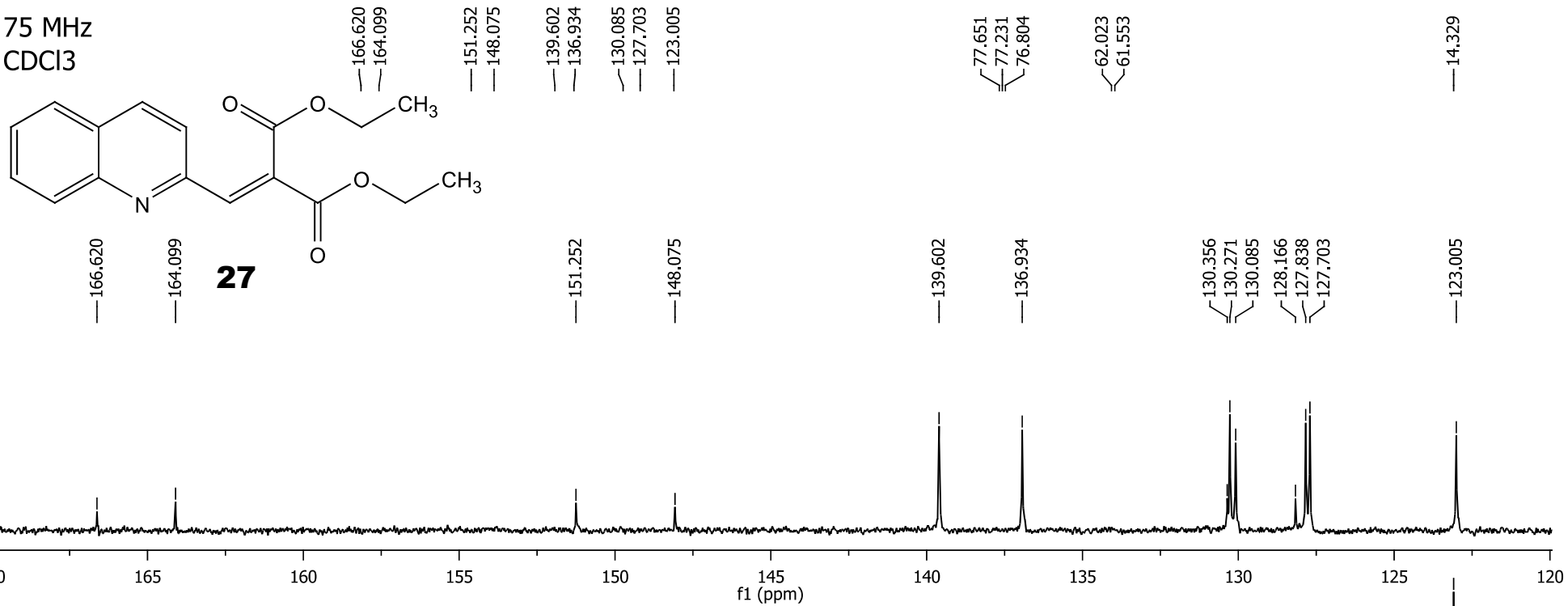


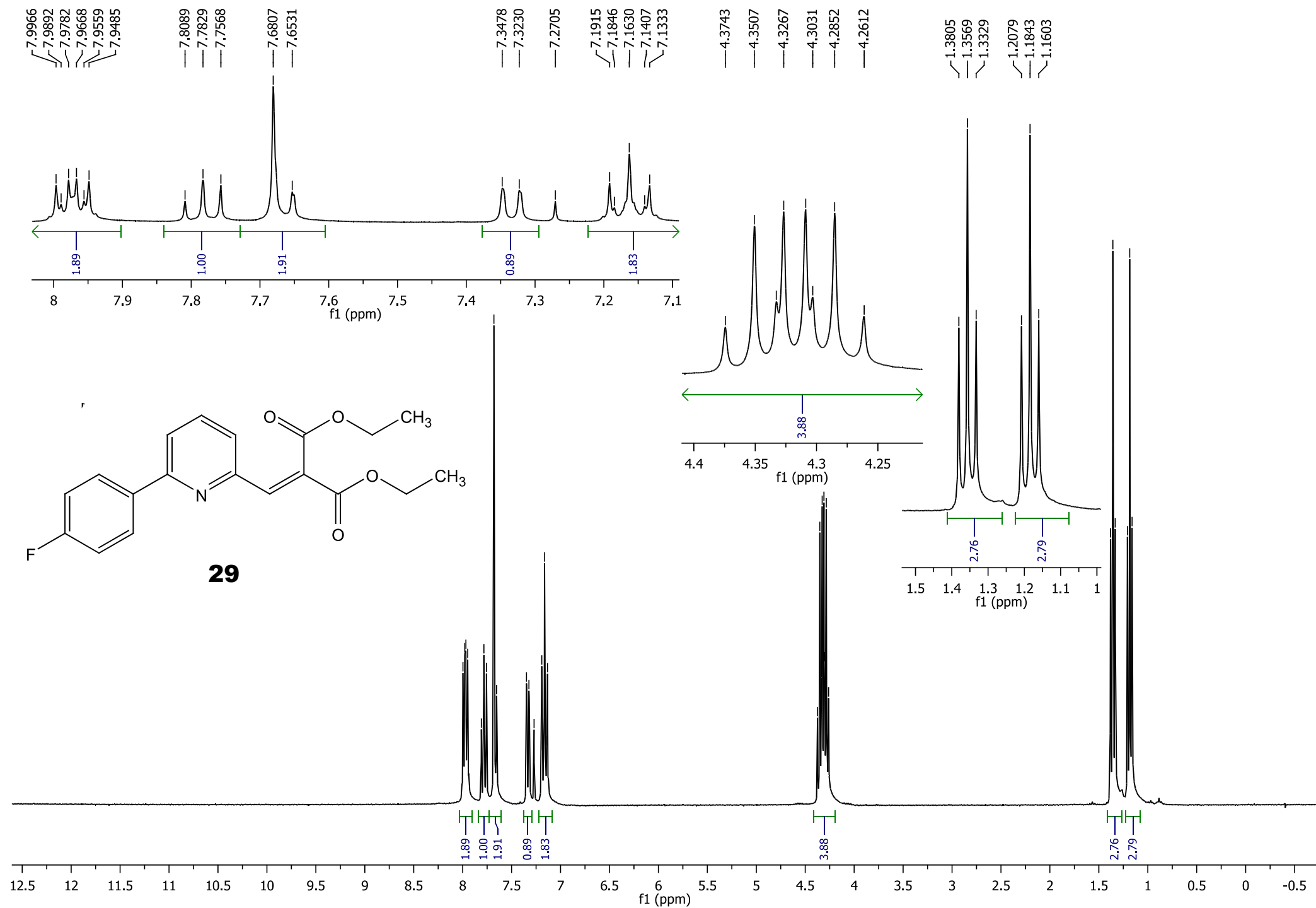
S27



75 MHz
CDCl₃

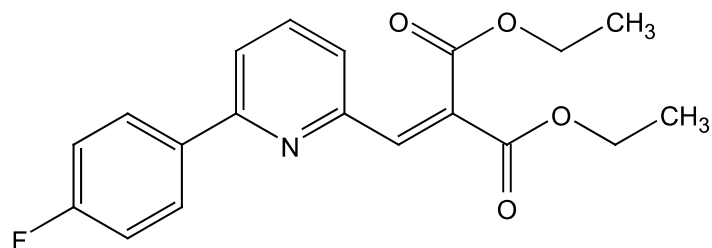
S28



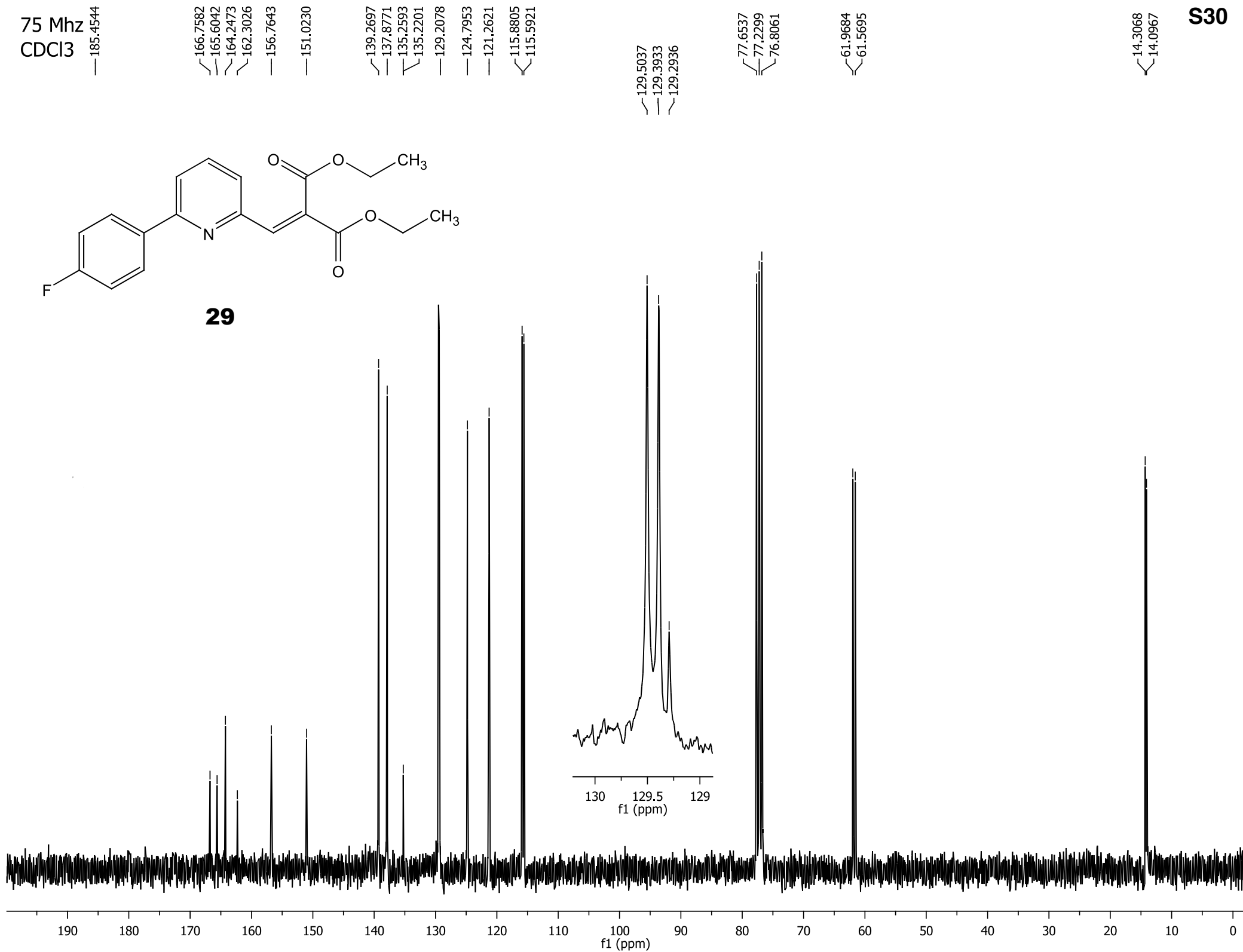


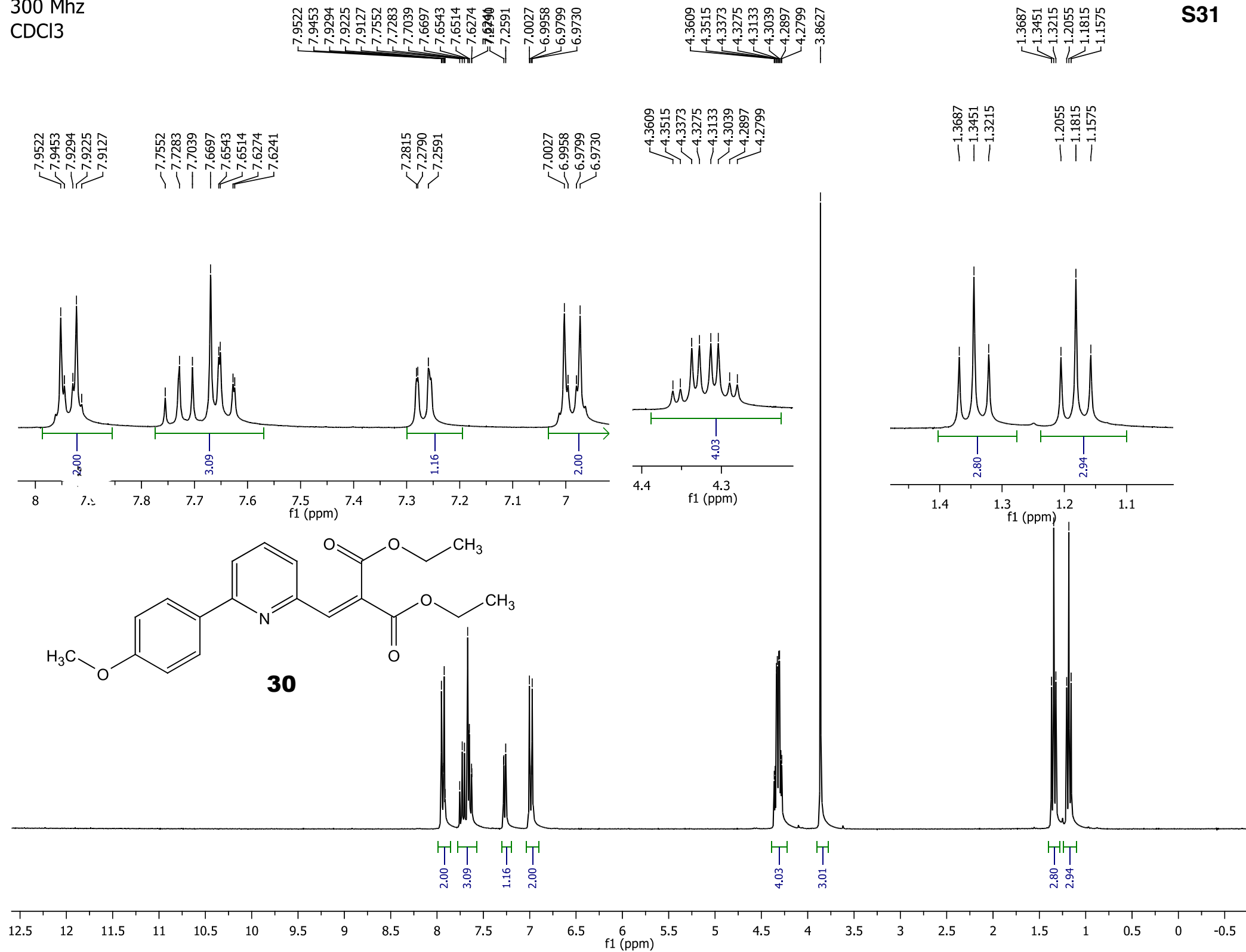
75 Mhz
CDCl₃

S30

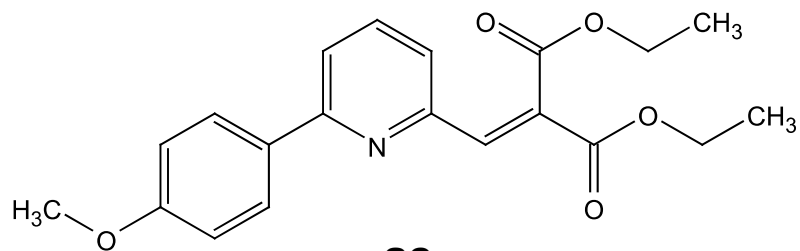


29



300 Mhz
CDCl₃

75 Mhz
CDCl3



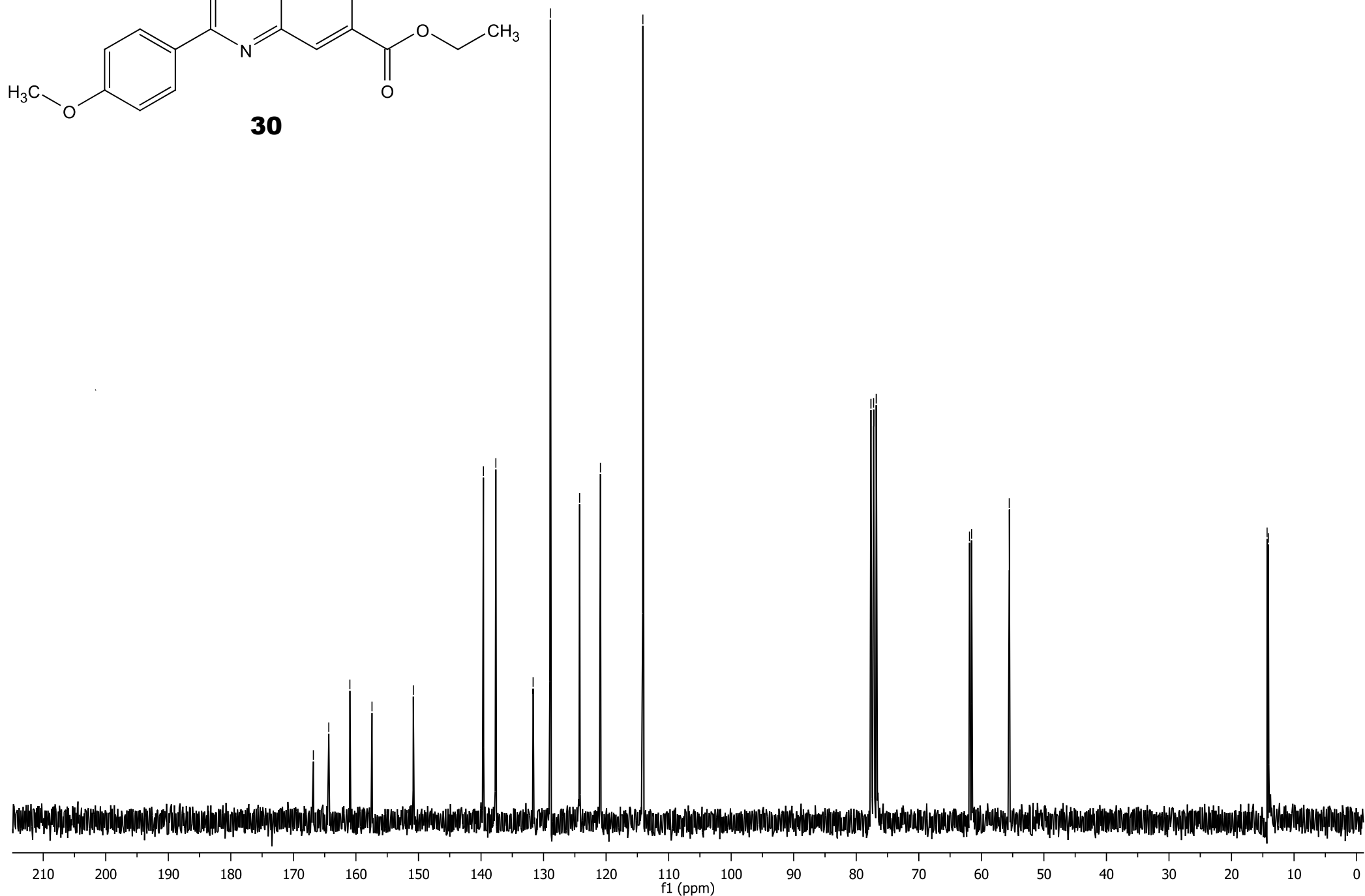
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164.3436
160.9636
157.4340
150.8272
139.6153
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124.2362
120.9097
114.1248

77.6539
77.2300
76.8062

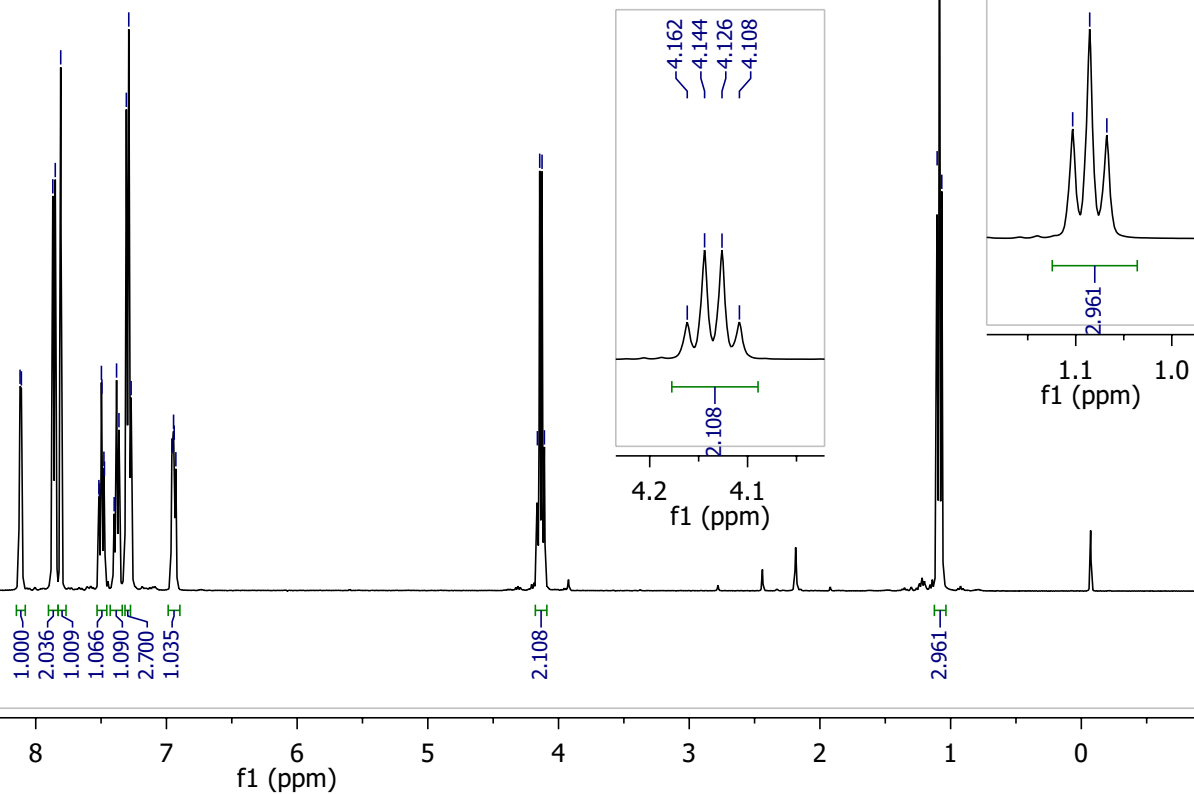
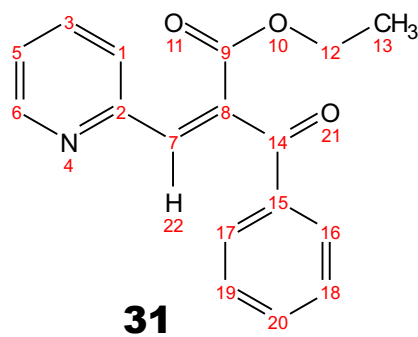
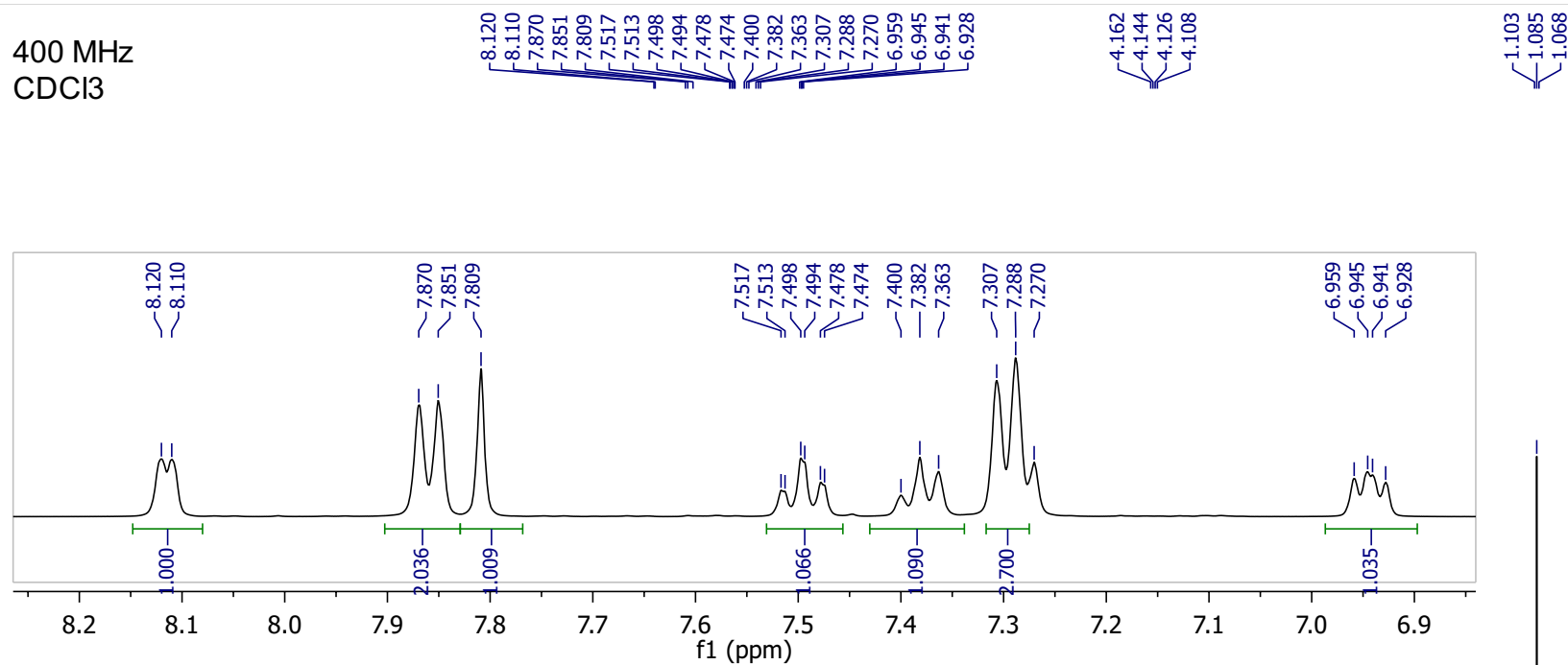
61.8937
61.5696
55.5398

14.3105
14.1253

S32



400 MHz
CDCl₃



100 MHz
CDCl₃

—194.311

—164.568

—150.945

—149.088

—139.737

—136.921

—136.500

—134.328

—132.573

—128.230

—125.846

—123.798

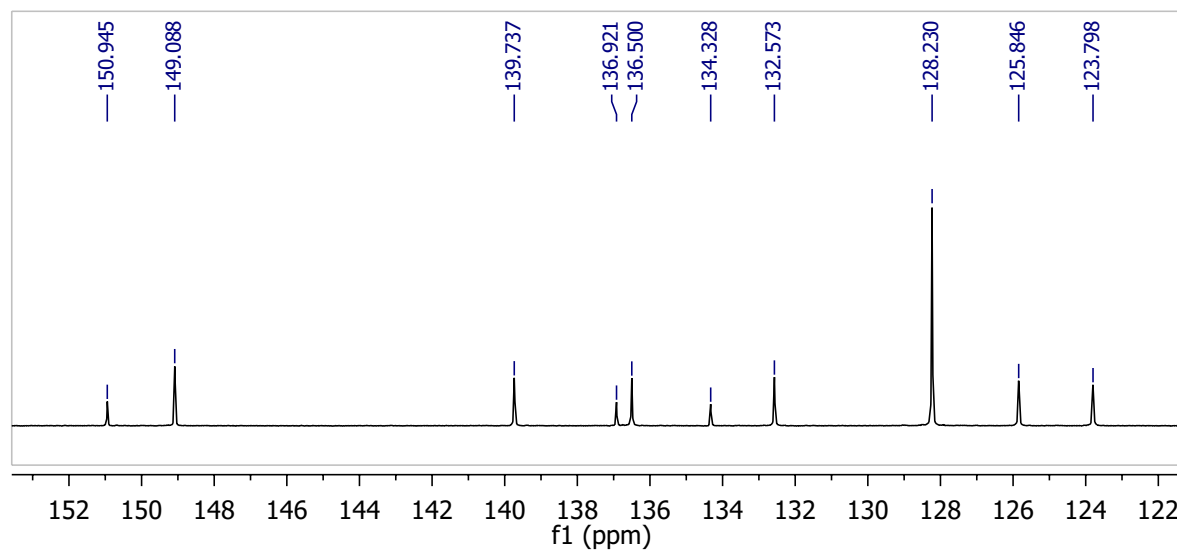
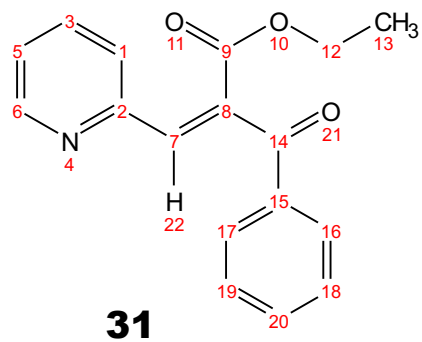
77.550

77.230

76.910

—61.418

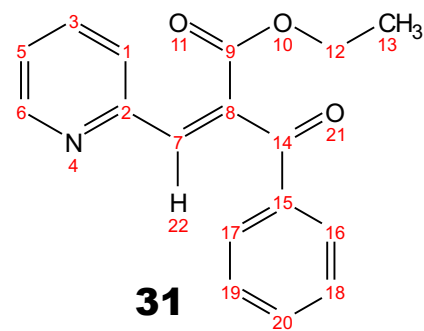
—13.857



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

f1 (ppm)

400 MHz
CDCl₃
1D-NOESY

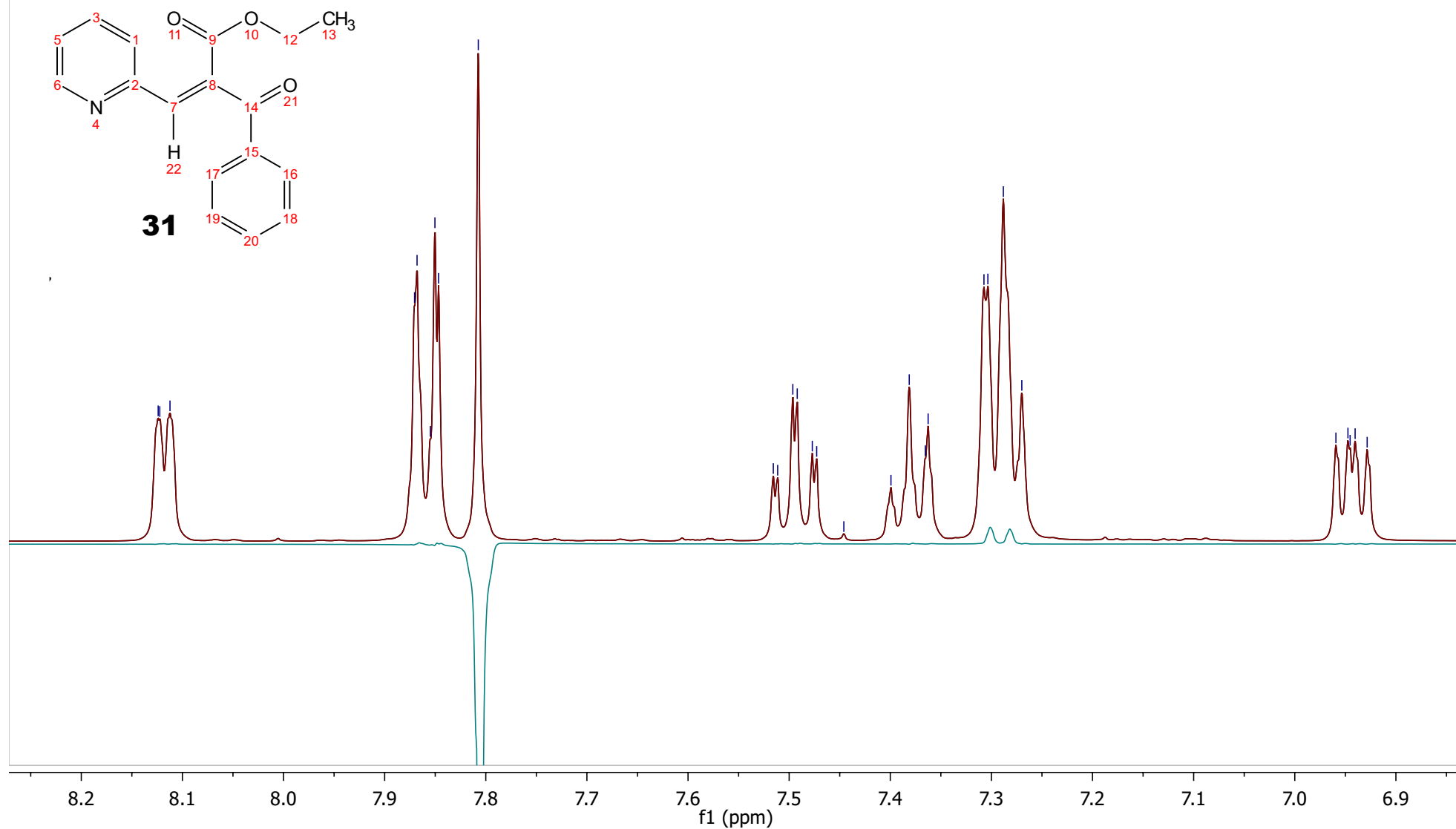


7.870
7.868
7.855
7.850
7.847
7.807

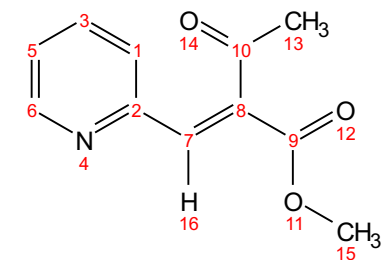
7.516
7.511
7.496
7.492
7.477
7.473
7.446
7.399
7.381
7.365
7.363

7.307
7.303
7.288
7.270

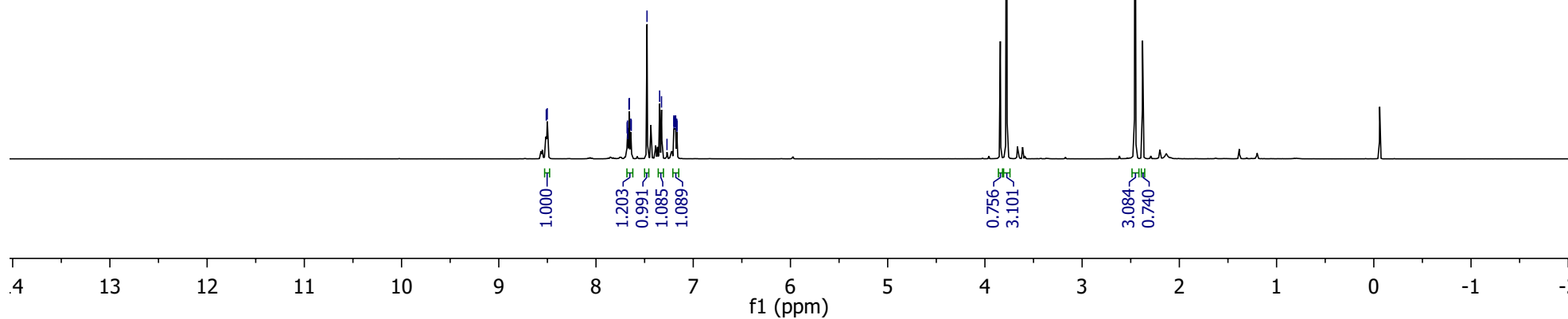
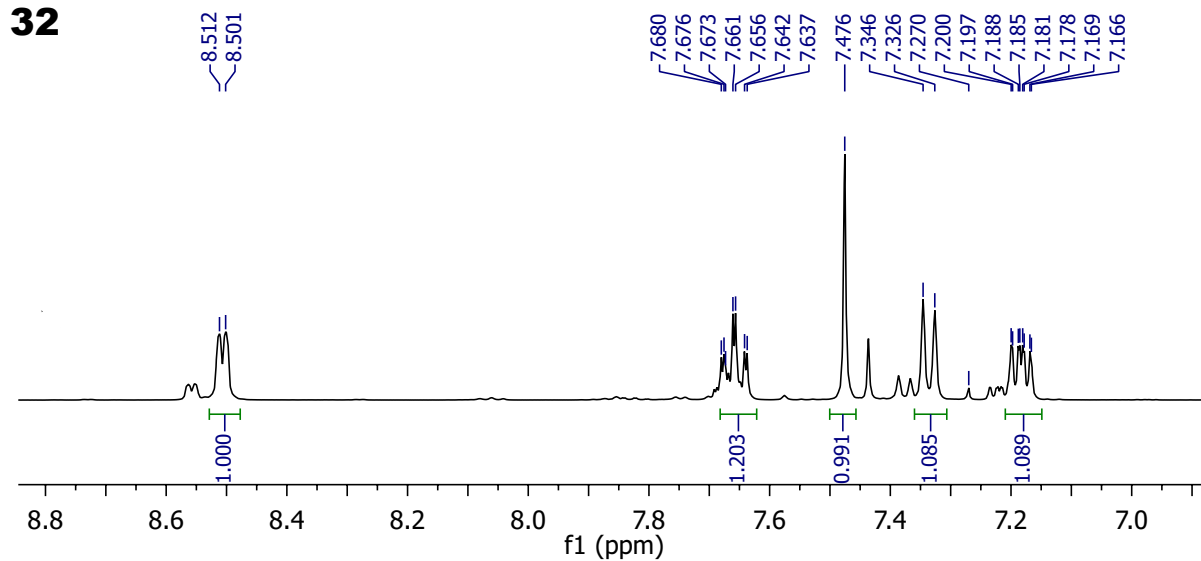
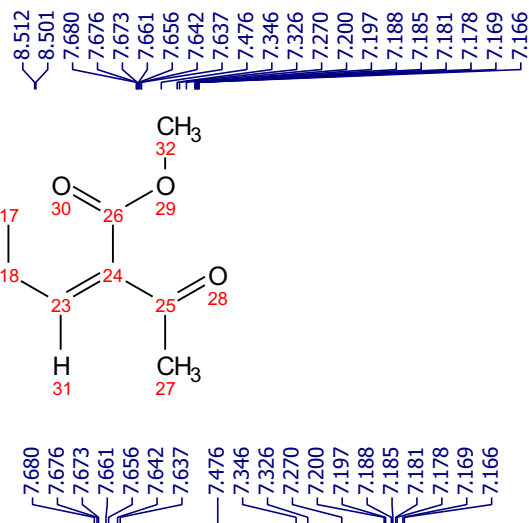
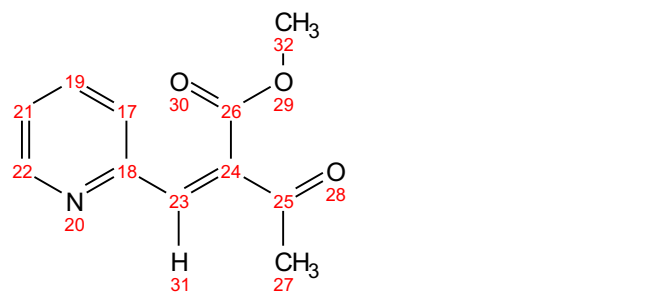
6.959
6.947
6.945
6.940
6.928



400 MHz
CDCl₃

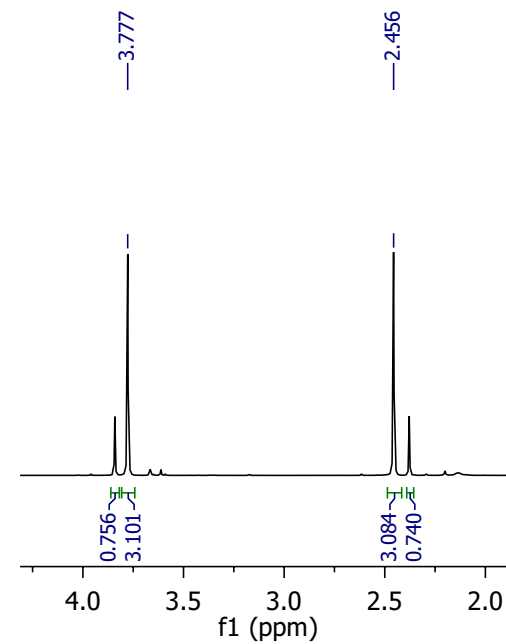


4:1



3.777

2.456



100 MHz
CDCl₃

— 202.468

— 194.657

— 168.289

— 164.857

— 151.332

— 151.055

— 149.967

— 149.534

— 137.579

— 136.927

— 136.525

— 126.158

— 124.667

— 124.163

— 77.549

— 77.230

— 76.910

— 76.043

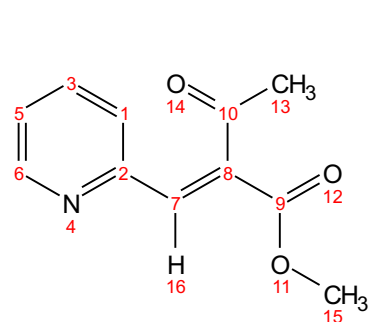
— 52.590

— 52.341

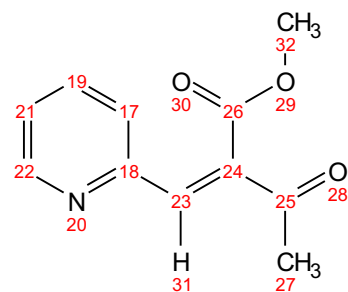
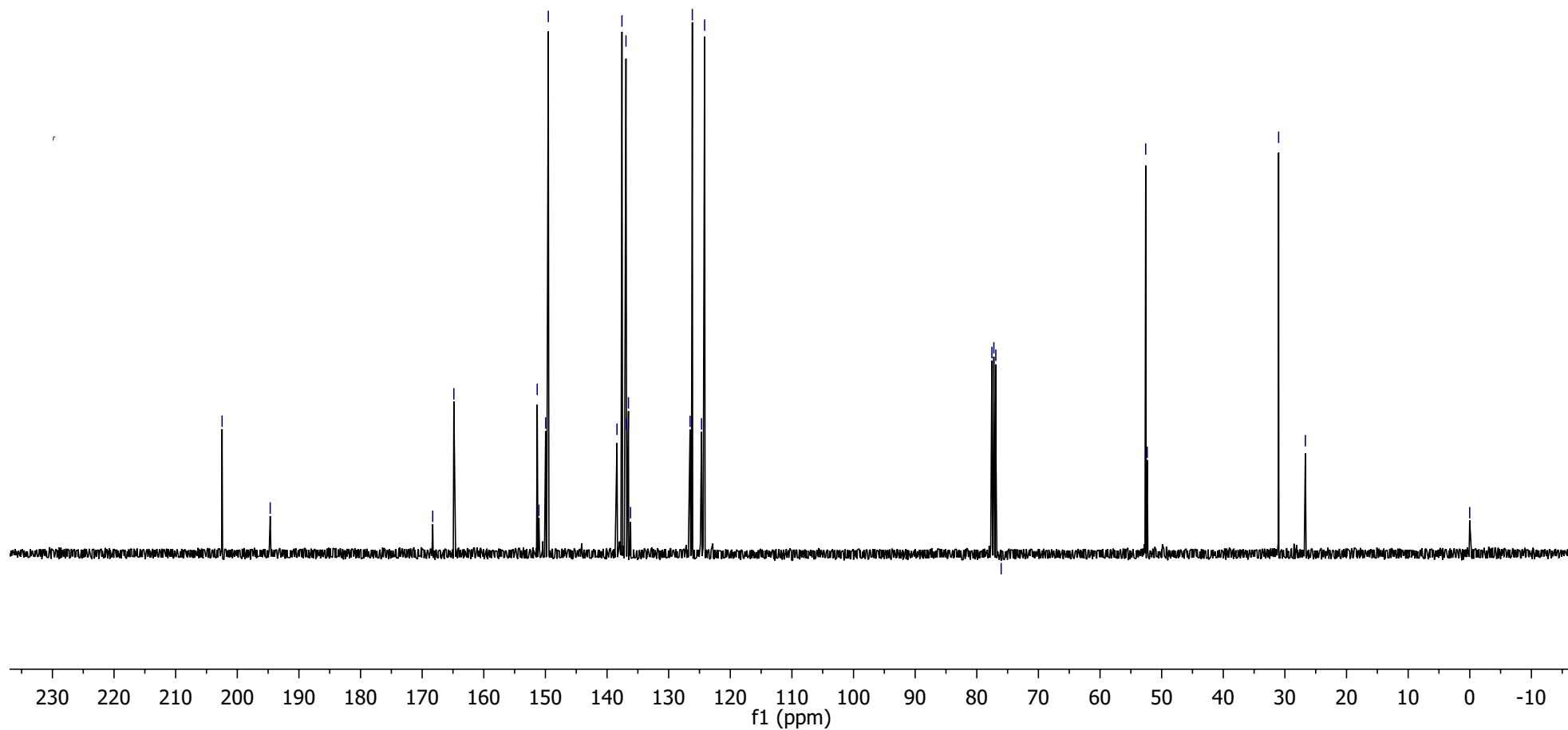
— 31.021

— 26.679

— 0.014



:
4:1

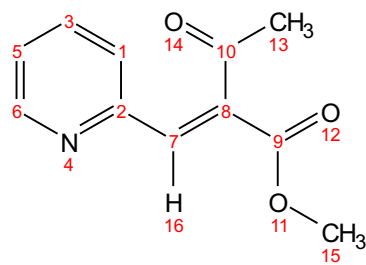
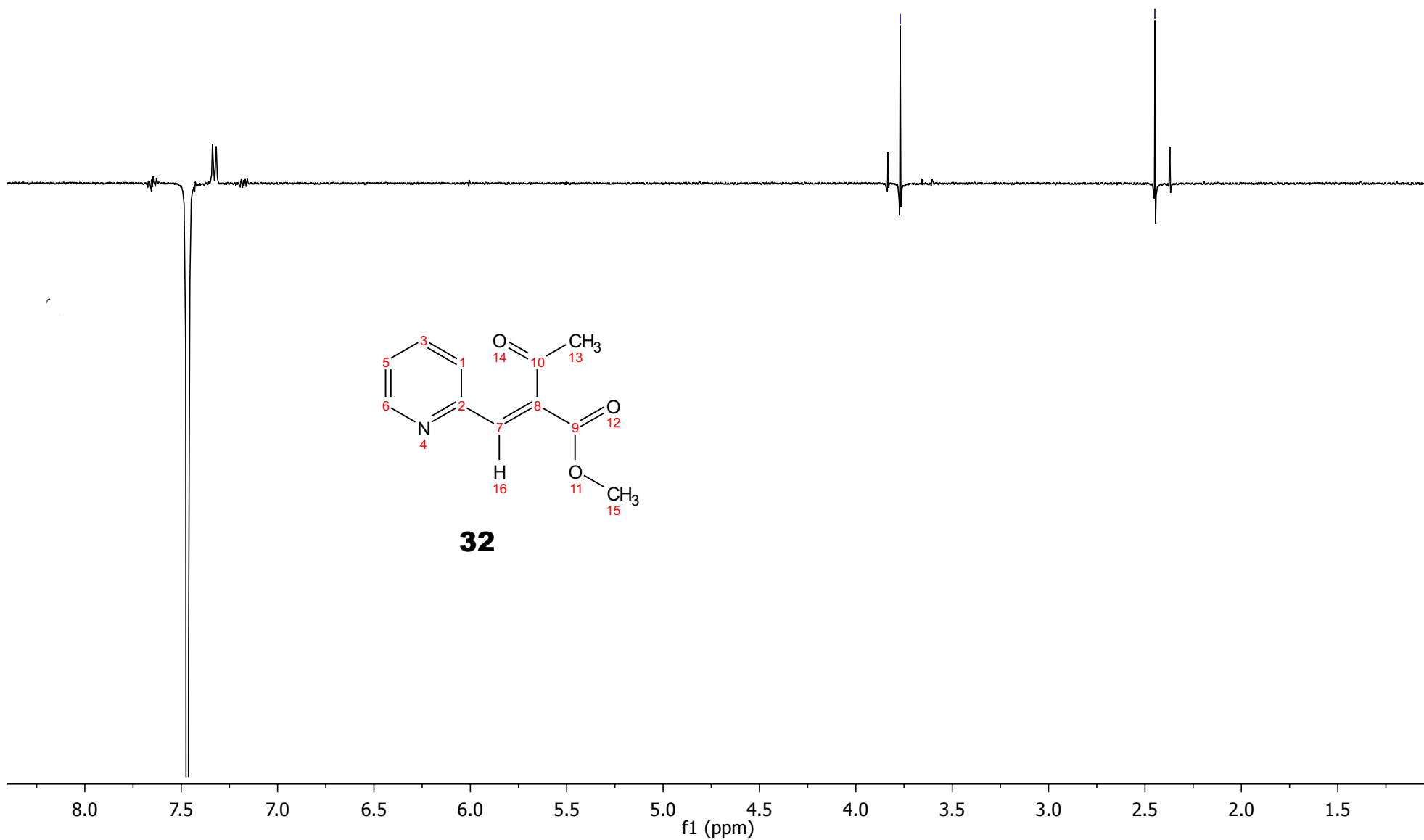
**32**

400 MHz
CDCl₃
1D-NOESY

7.468

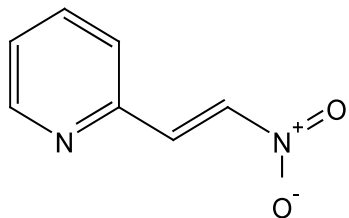
3.770

2.449

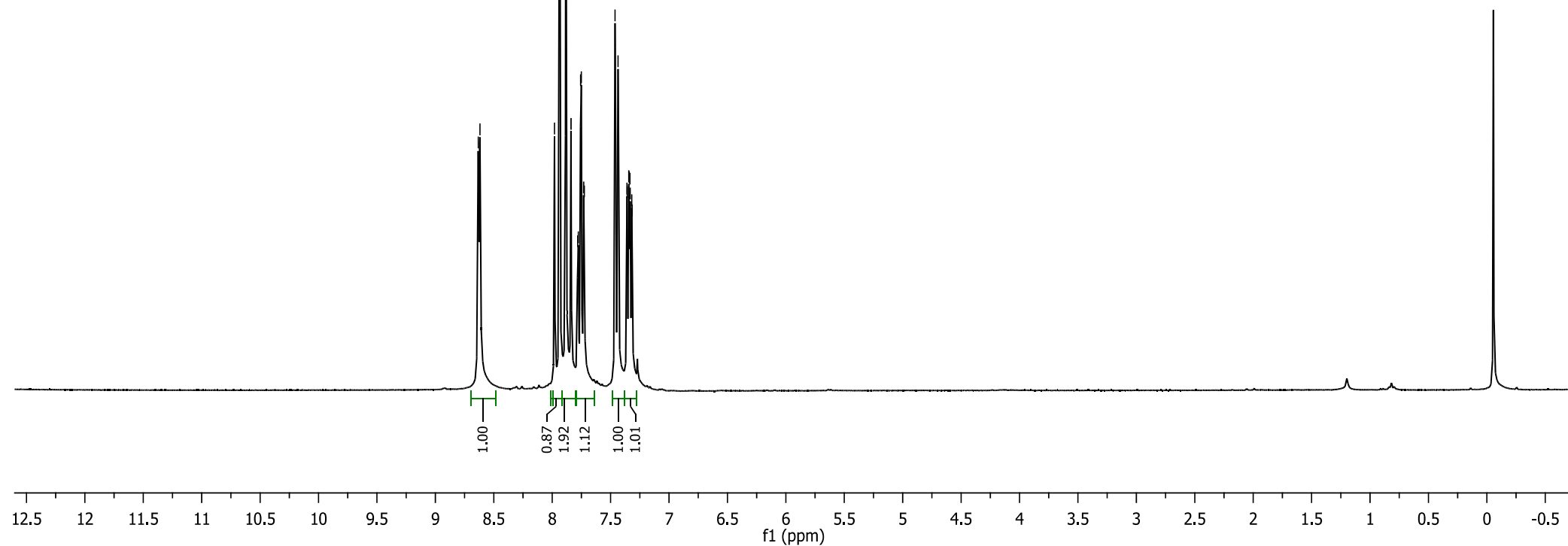
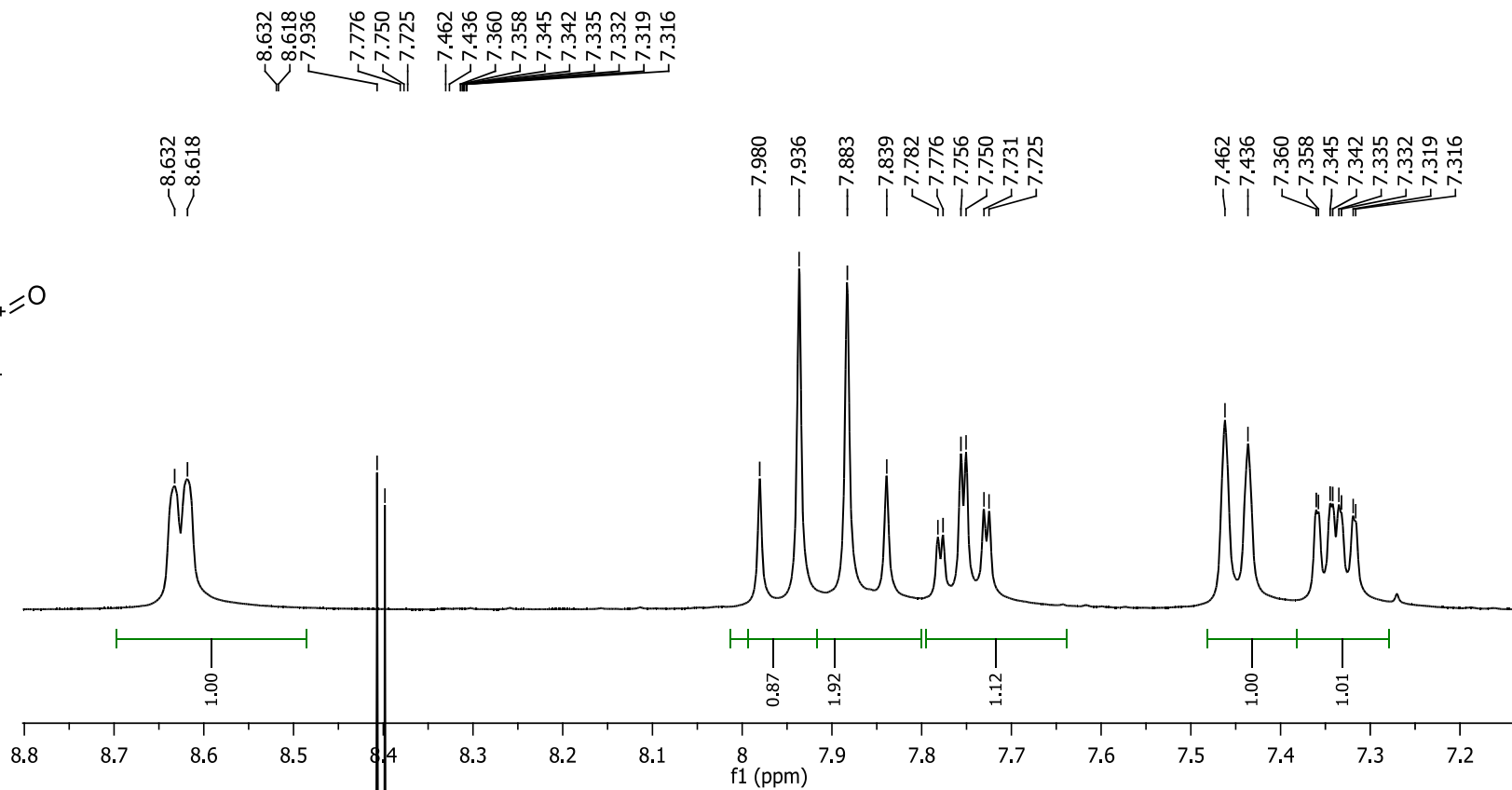
**32**

300 MHz
CDCl₃

S39



36



75 MHz
CDCl₃

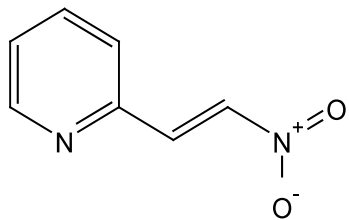
150.711
149.404

140.682
137.352
137.277

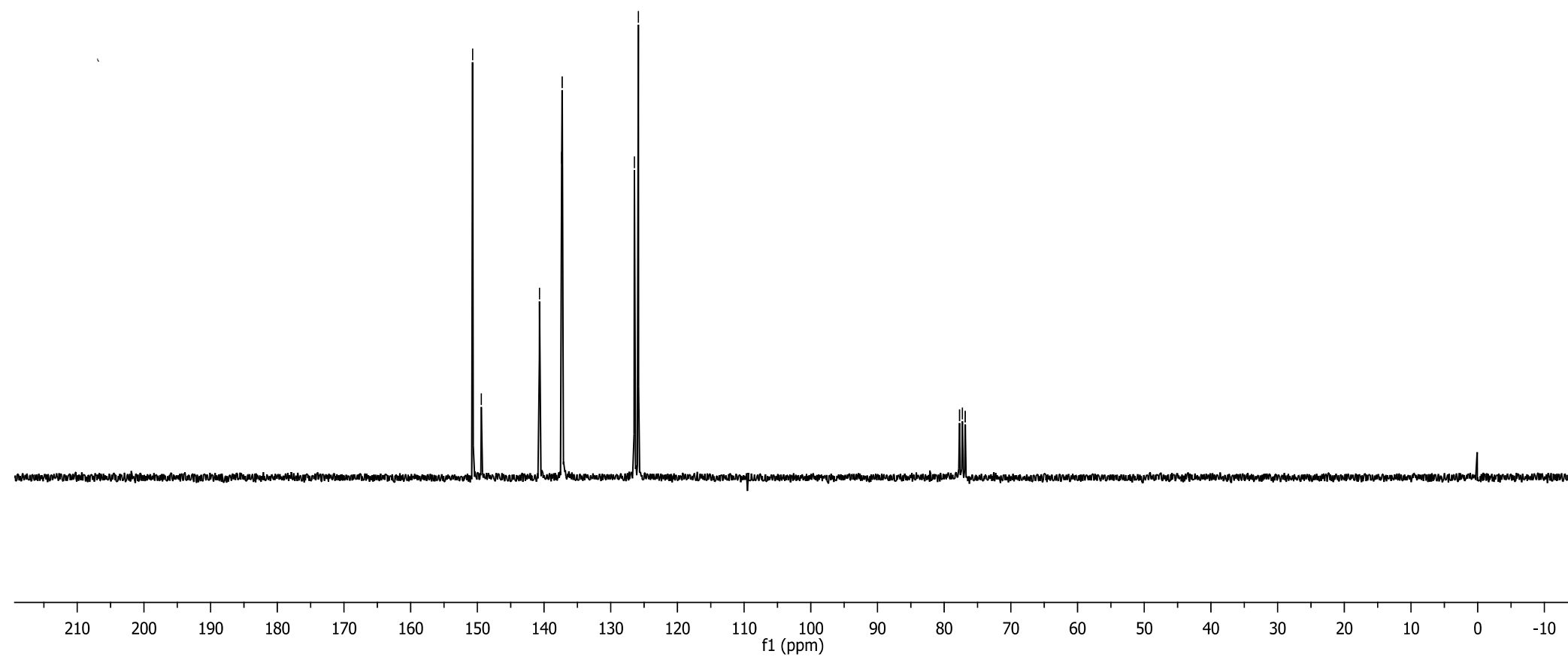
126.450
125.855

77.695
77.271
76.843

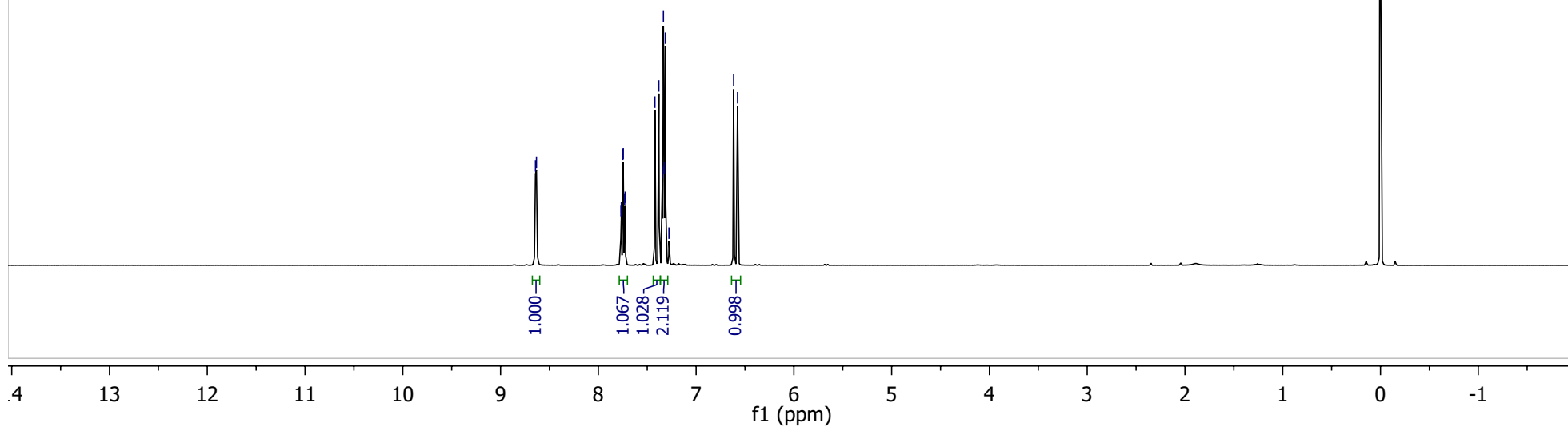
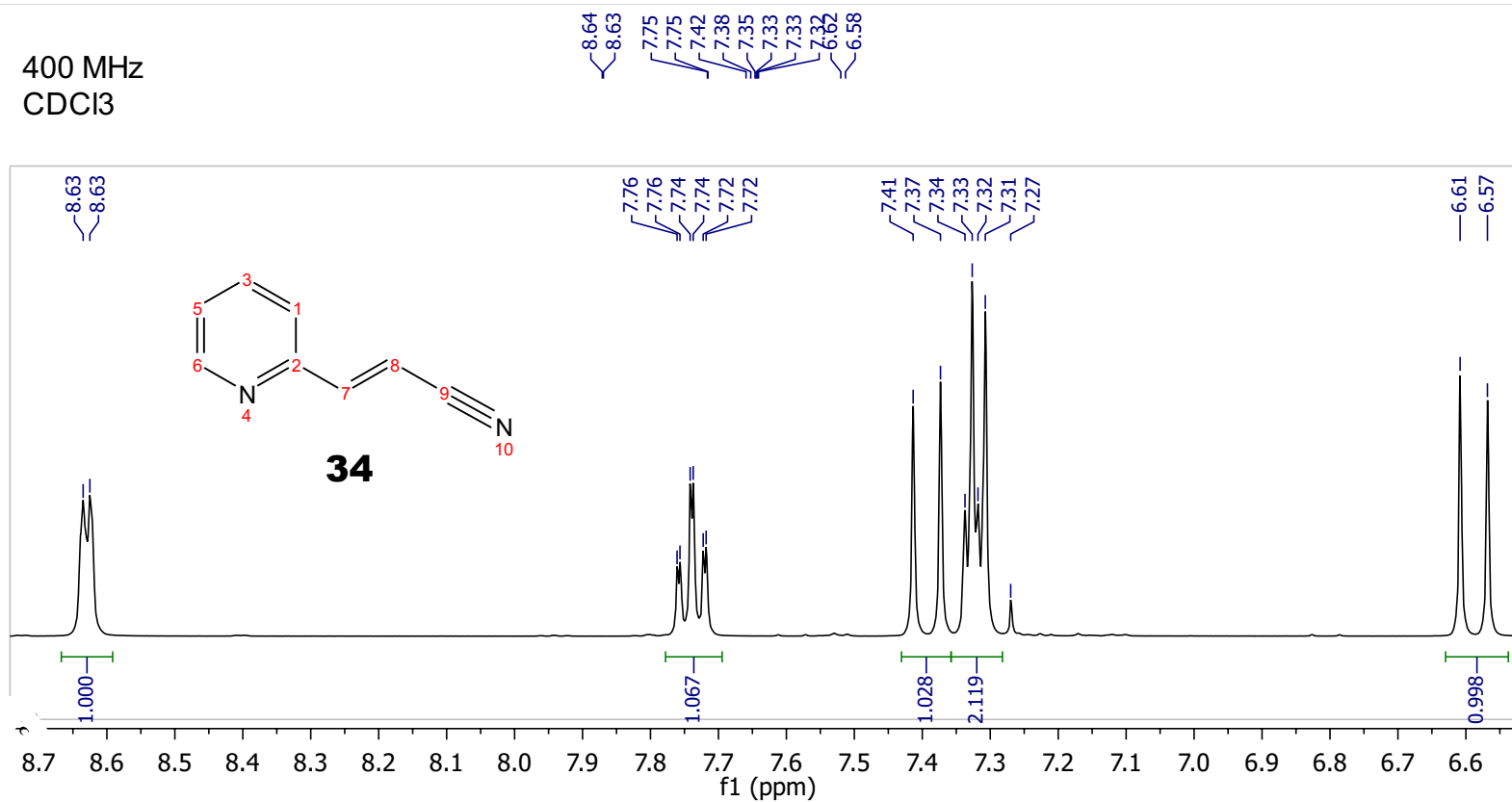
S40

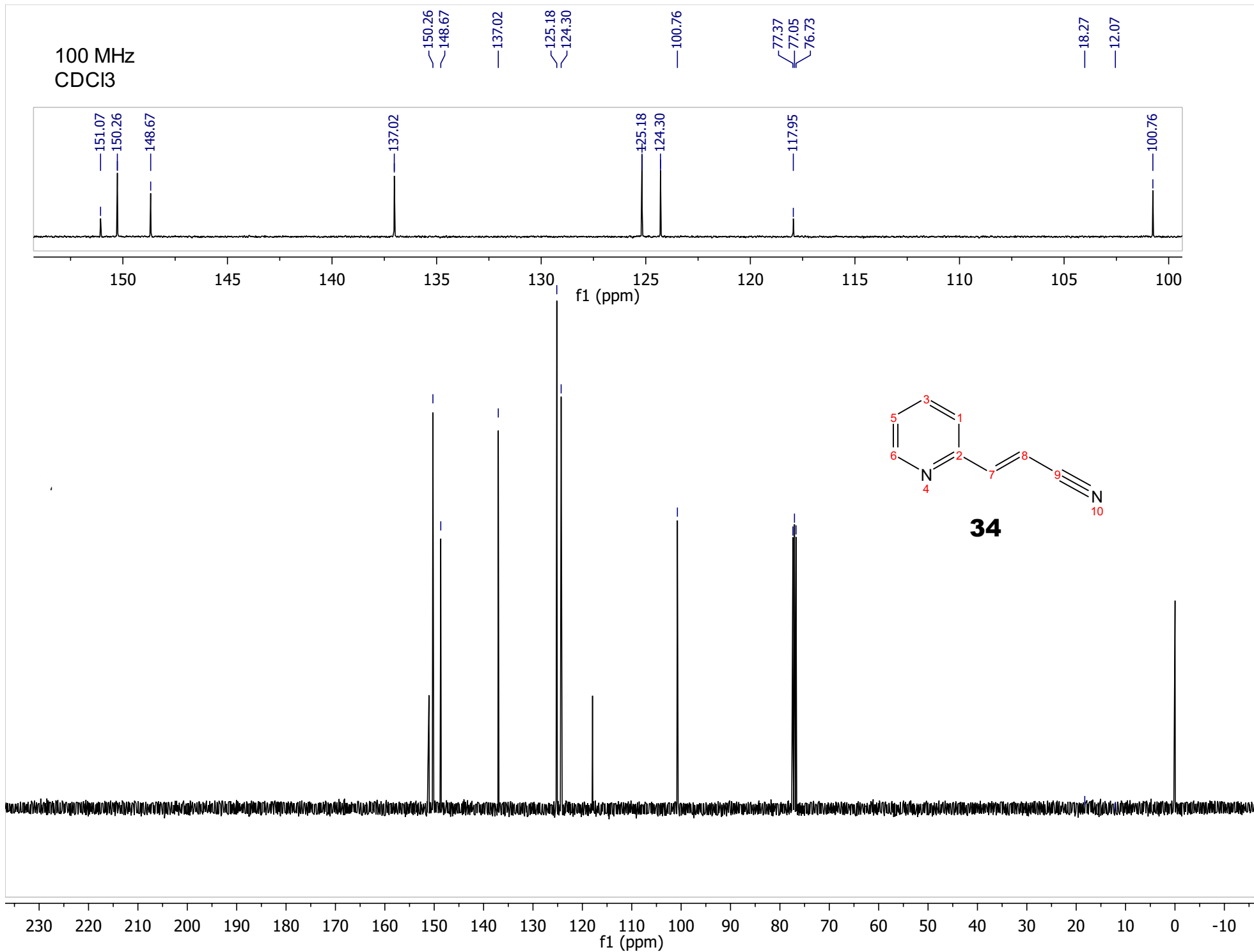


36

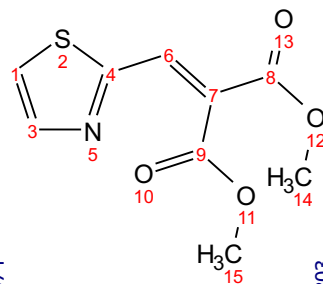


400 MHz
CDCl₃





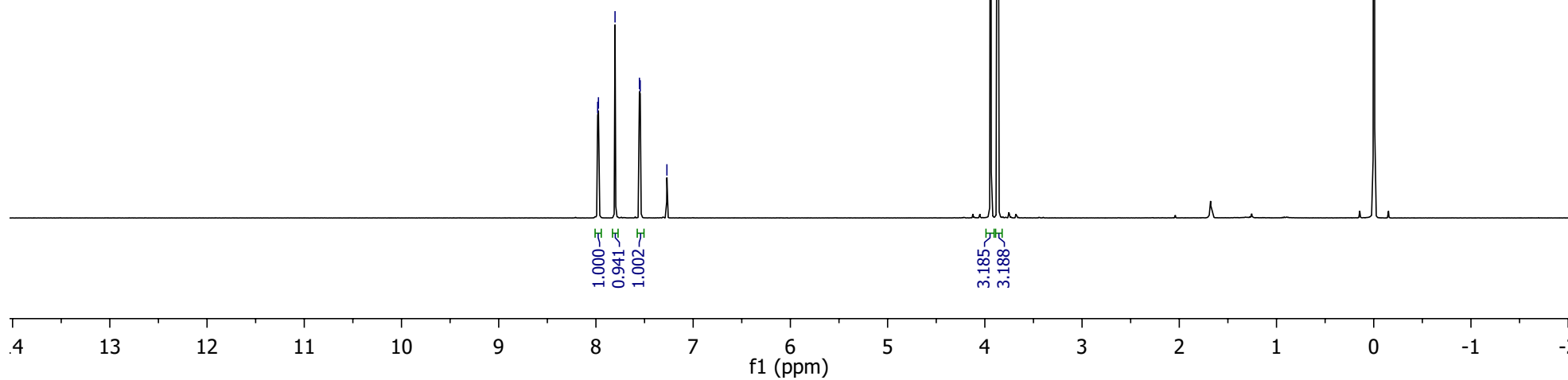
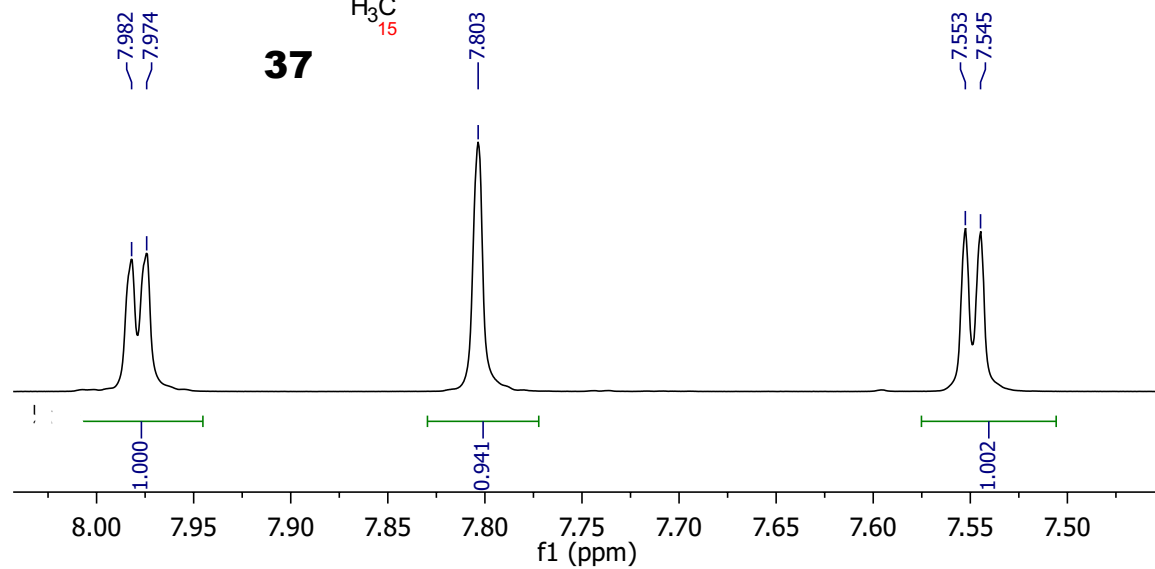
400 MHz
CDCl₃



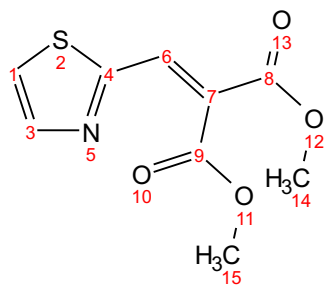
37

7.982
7.974
7.803
7.553
7.545
7.270

3.940
3.868



100 MHz
CDCl₃



37

—166.224
—164.024
—159.860

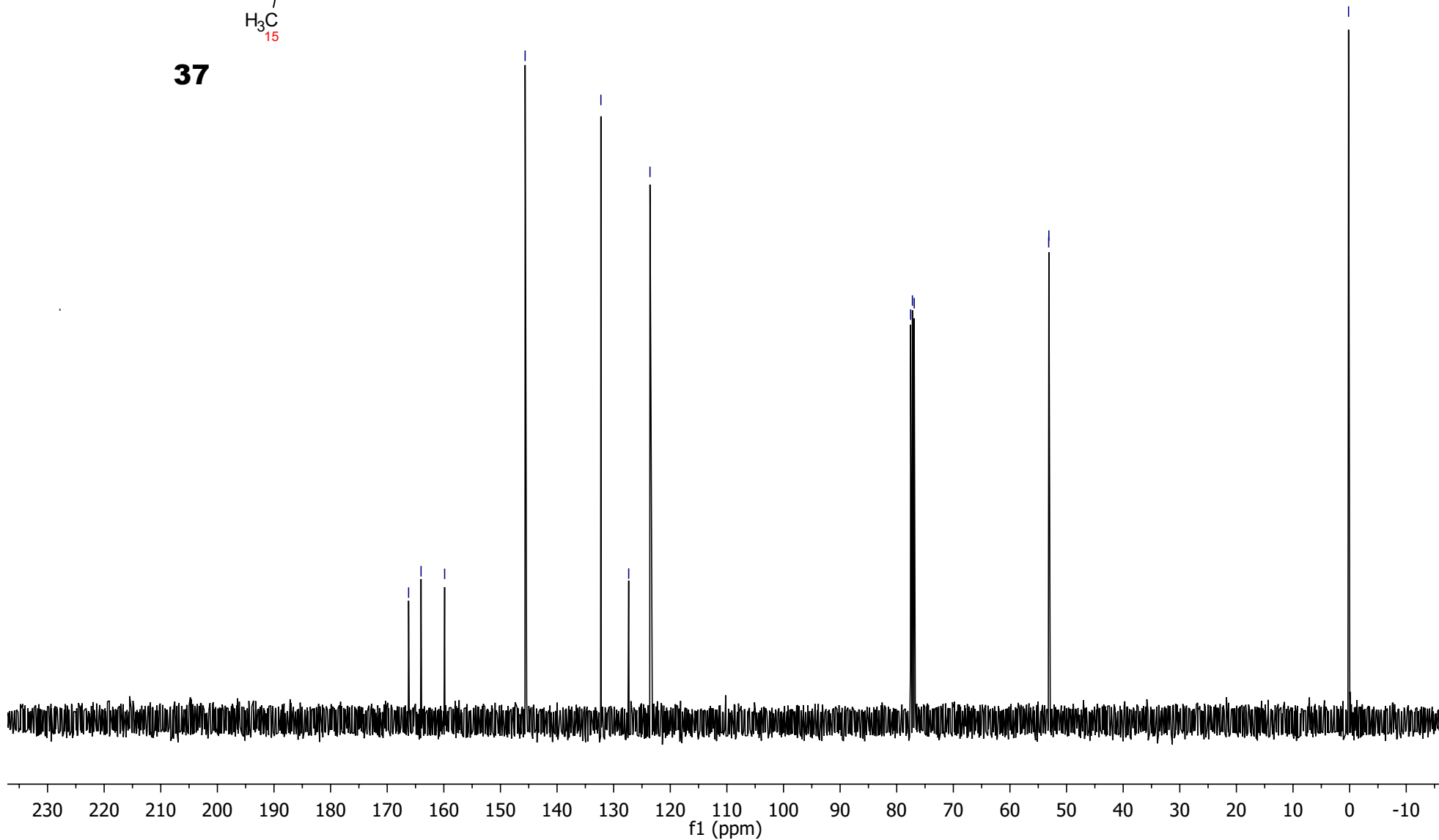
—145.641

—132.256
—127.347
—123.586

77.547
77.230
76.911

53.160
53.124

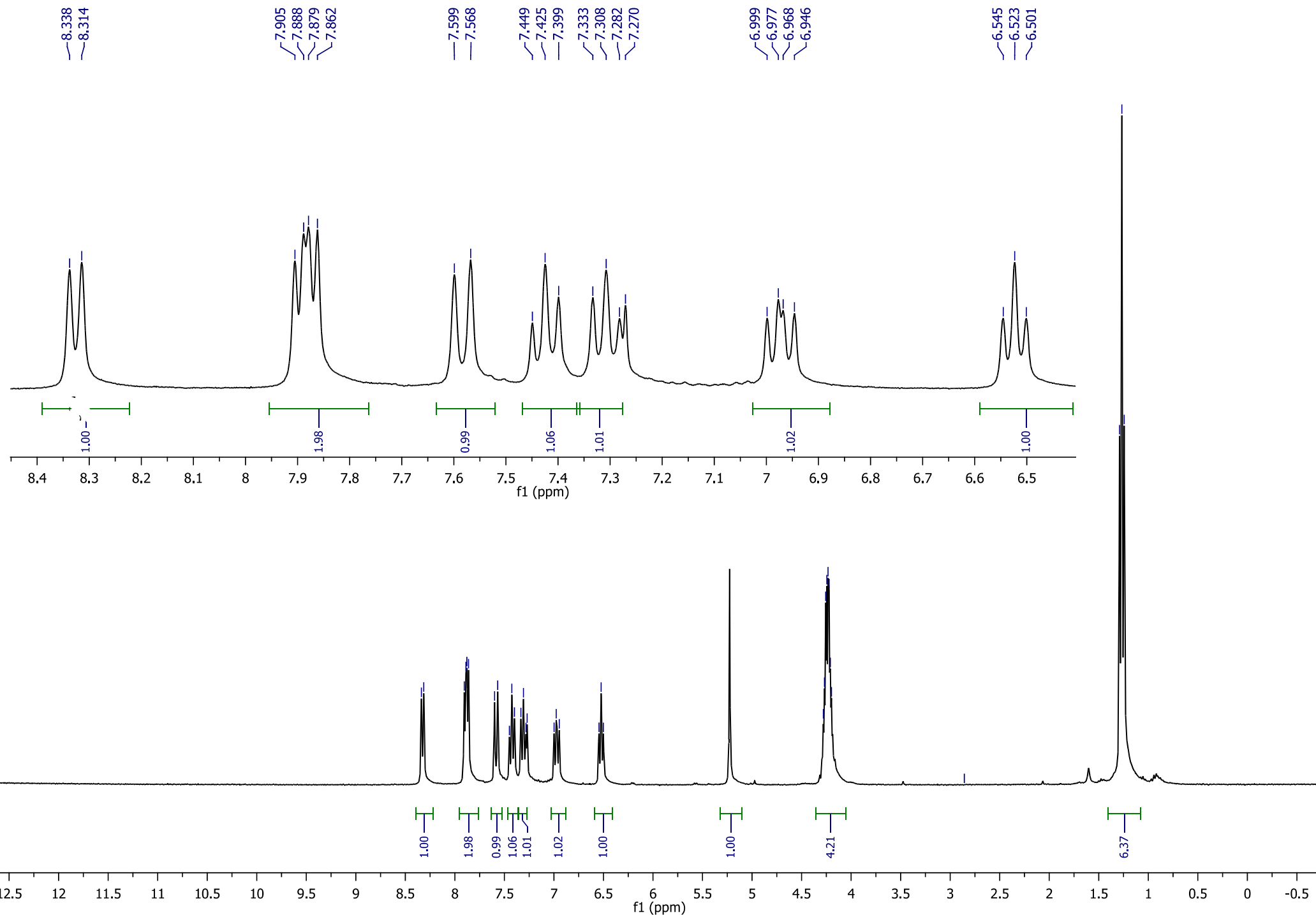
—0.185



300 MHz
CDCl₃

4

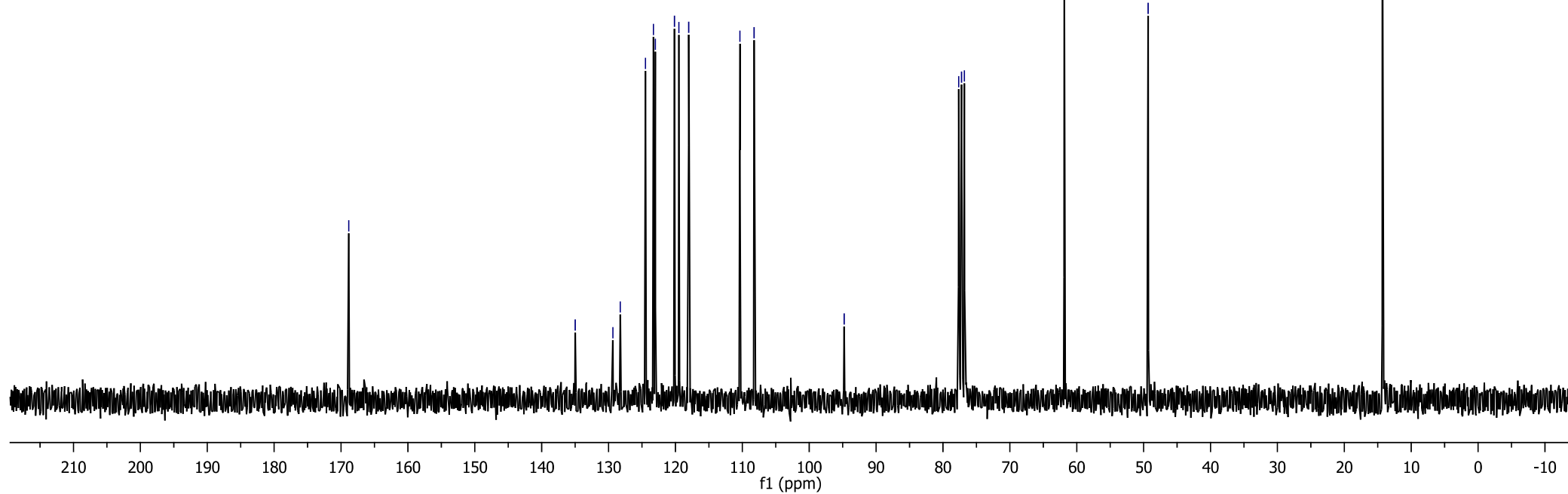
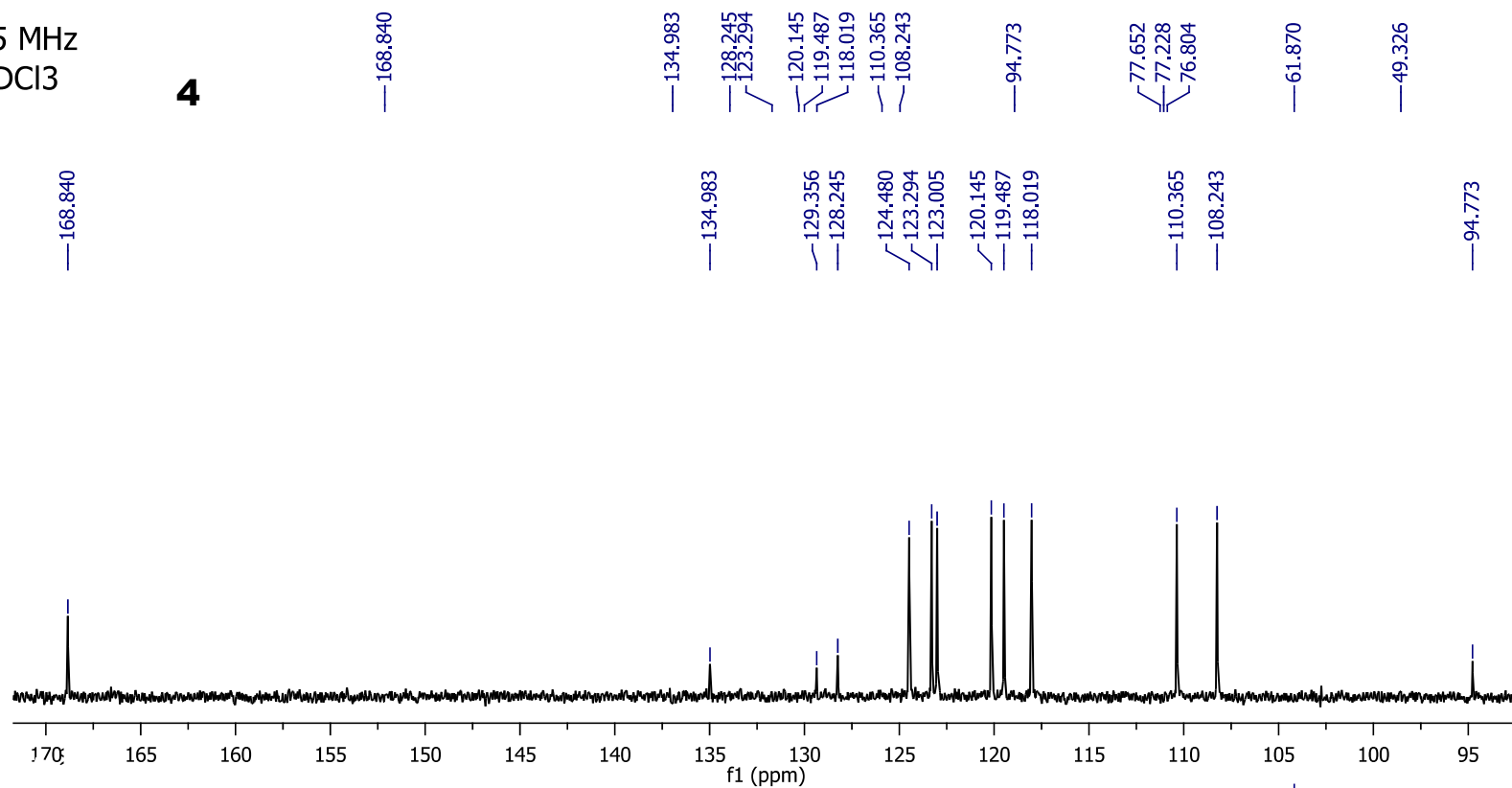
S45



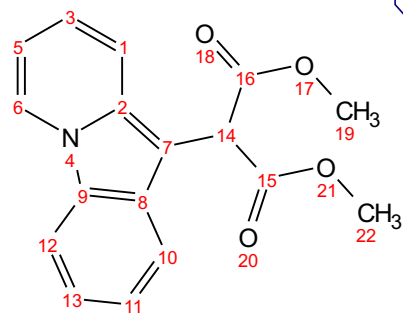
75 MHz
CDCl₃

4

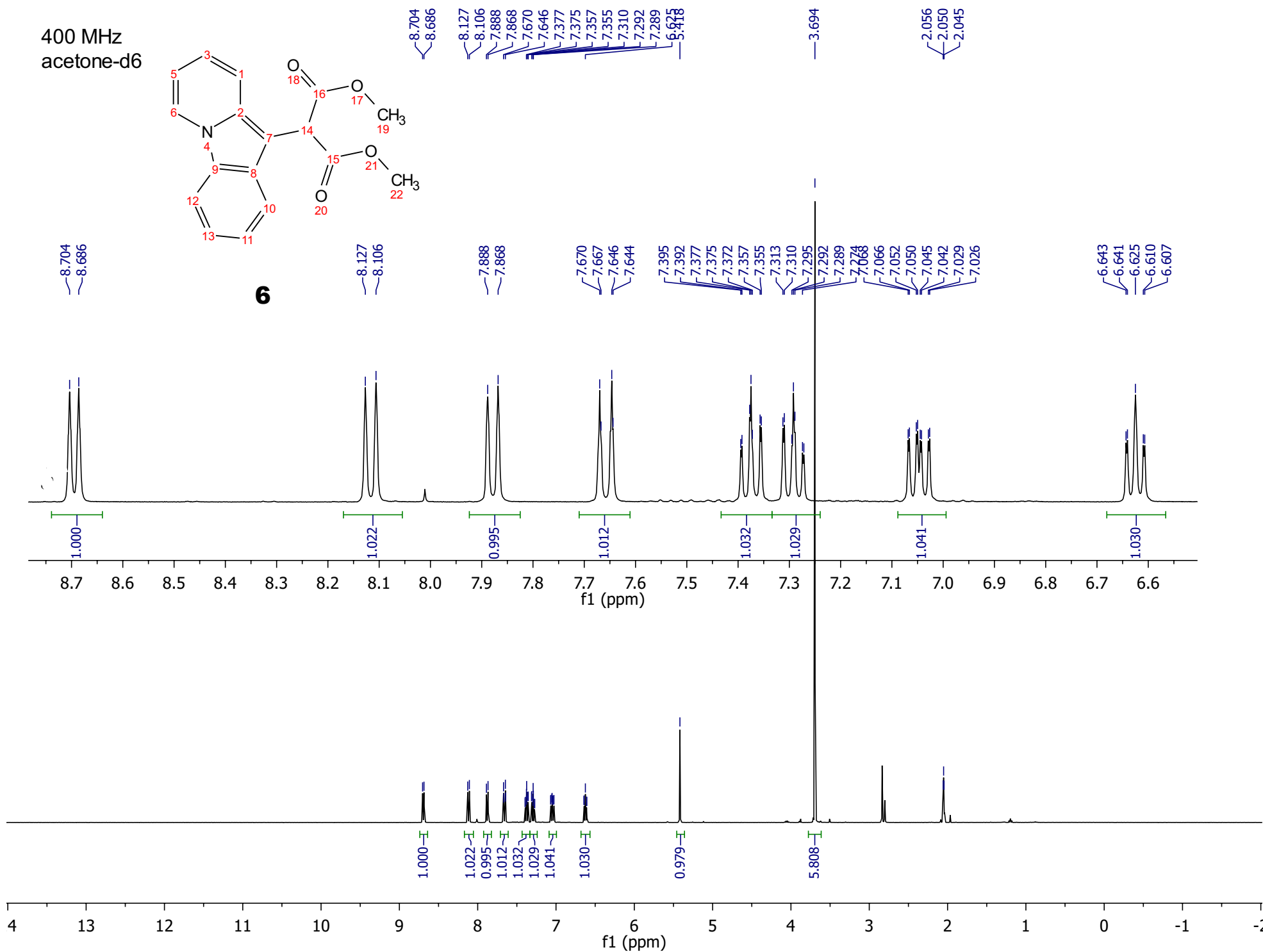
S46



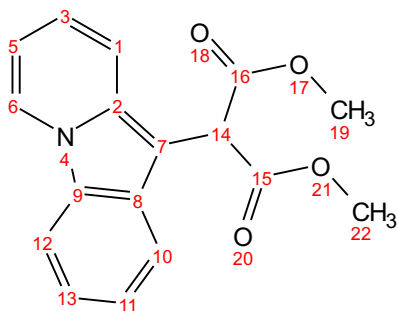
400 MHz
acetone-d₆



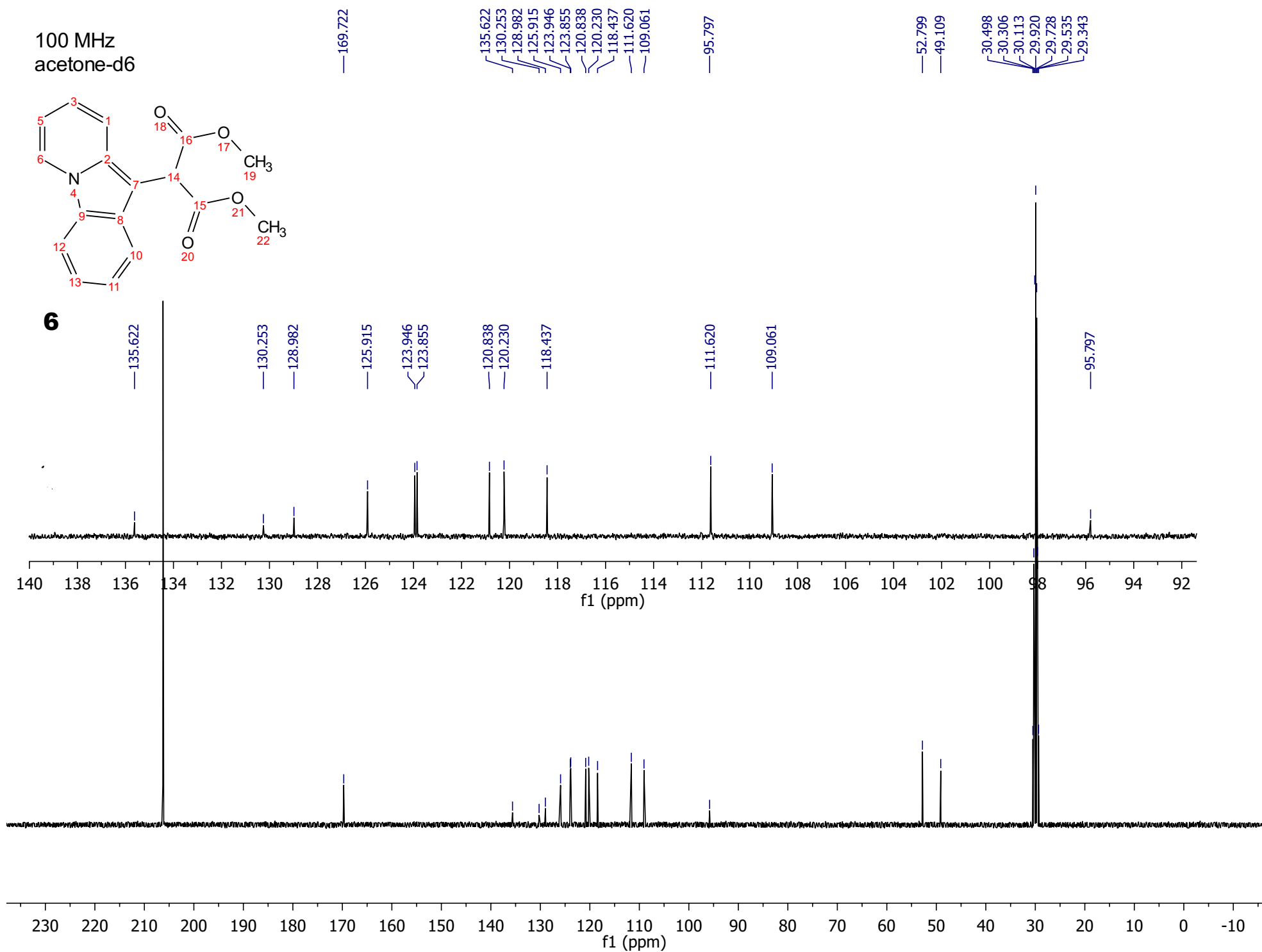
6



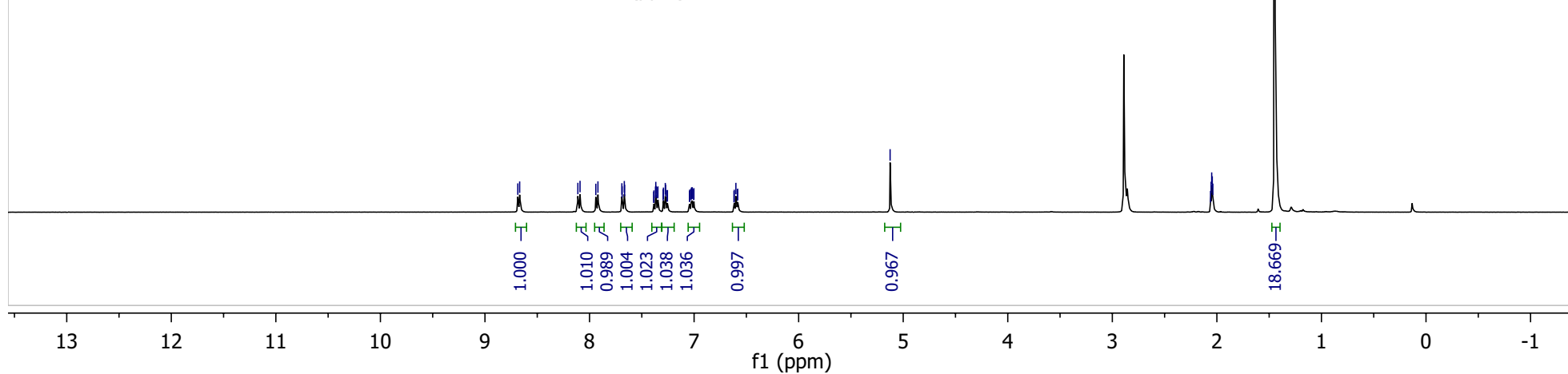
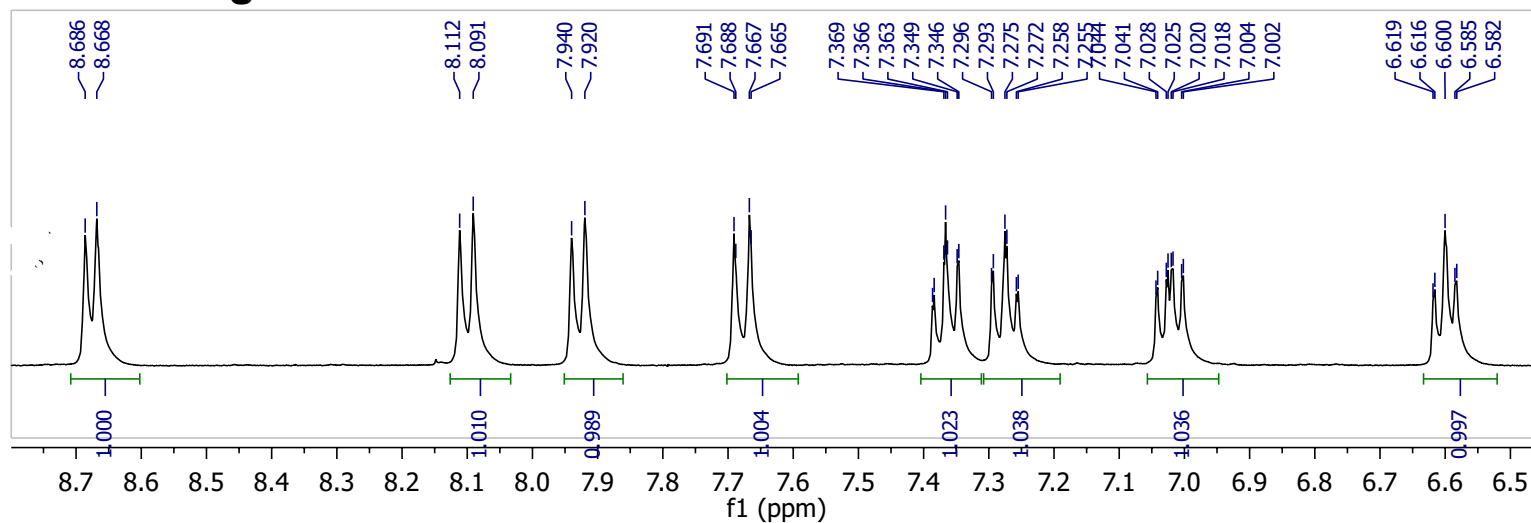
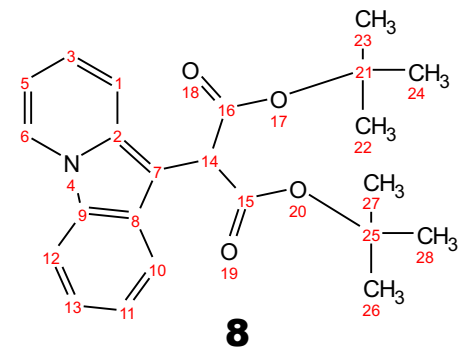
100 MHz
acetone-d₆

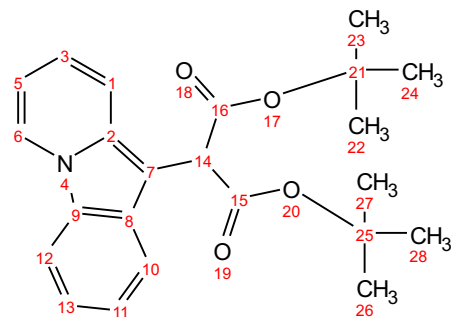
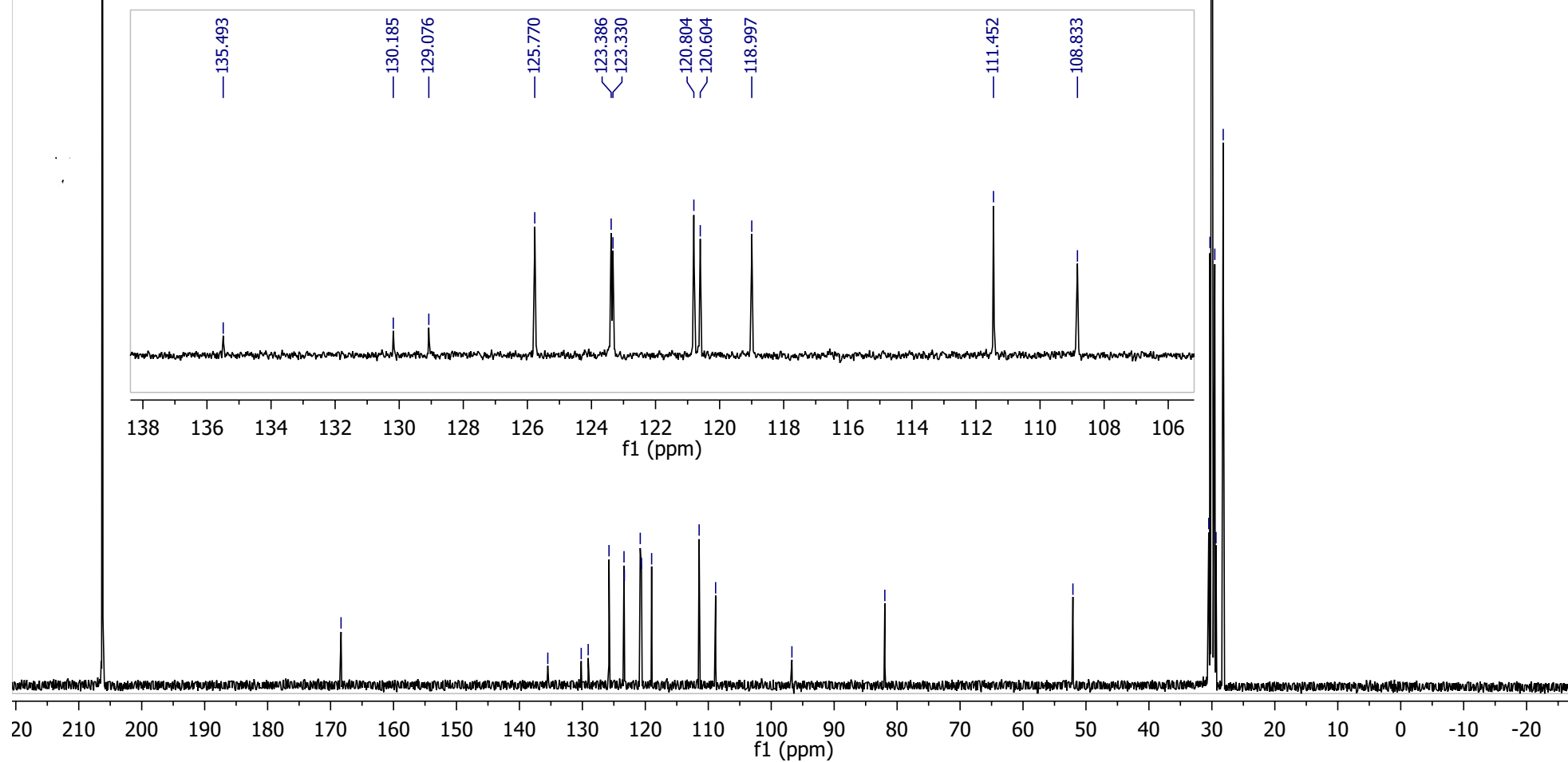


6

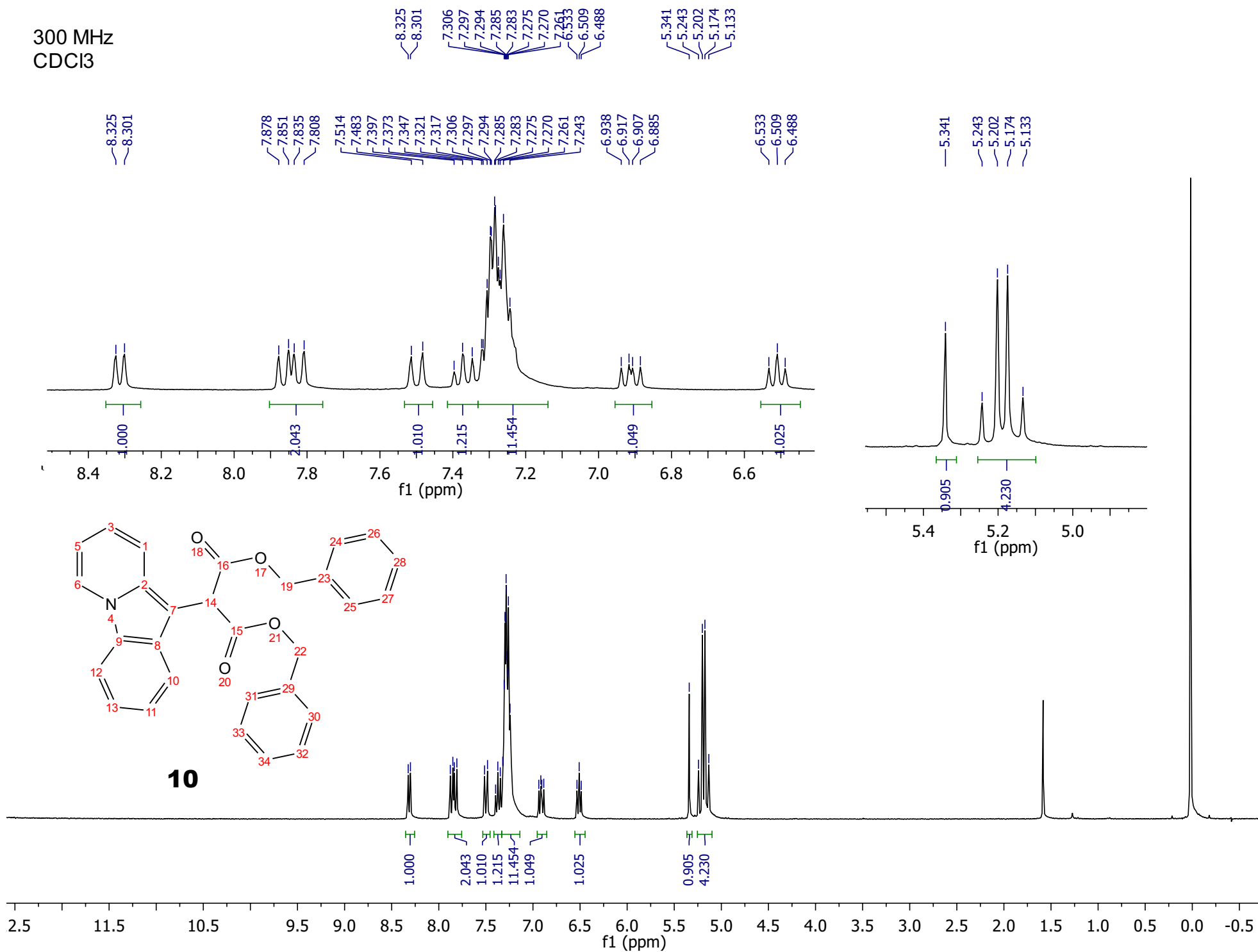


400 MHz
acetone-d₆

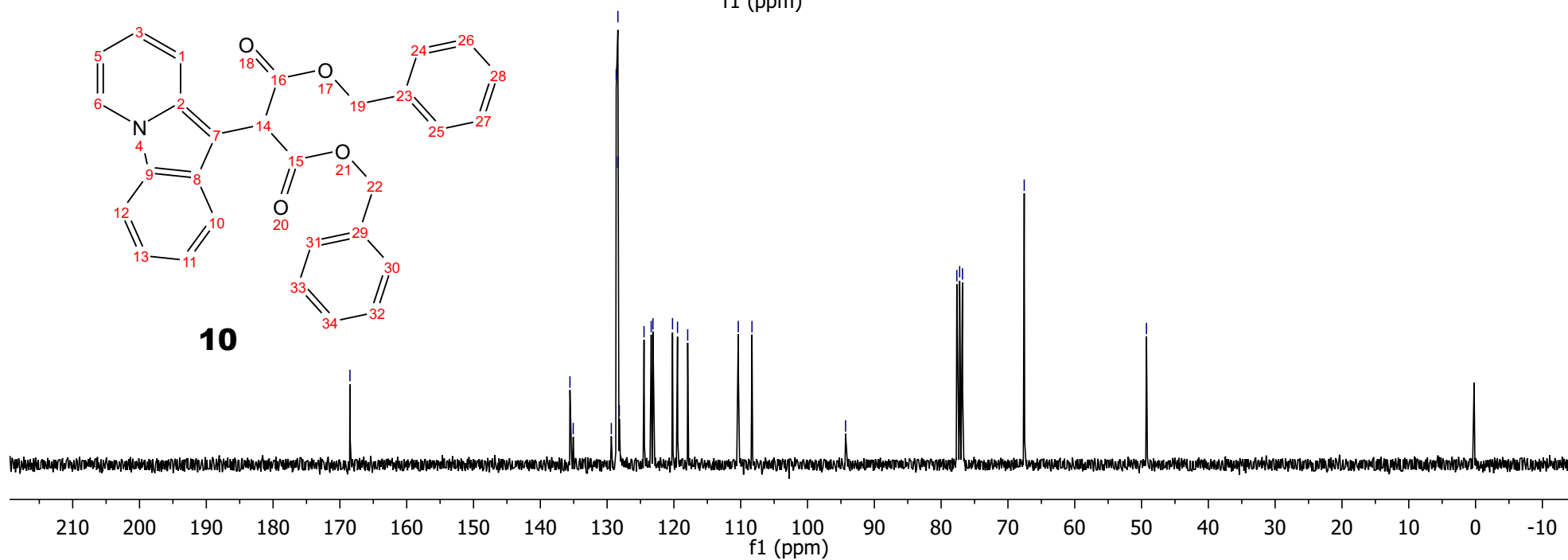
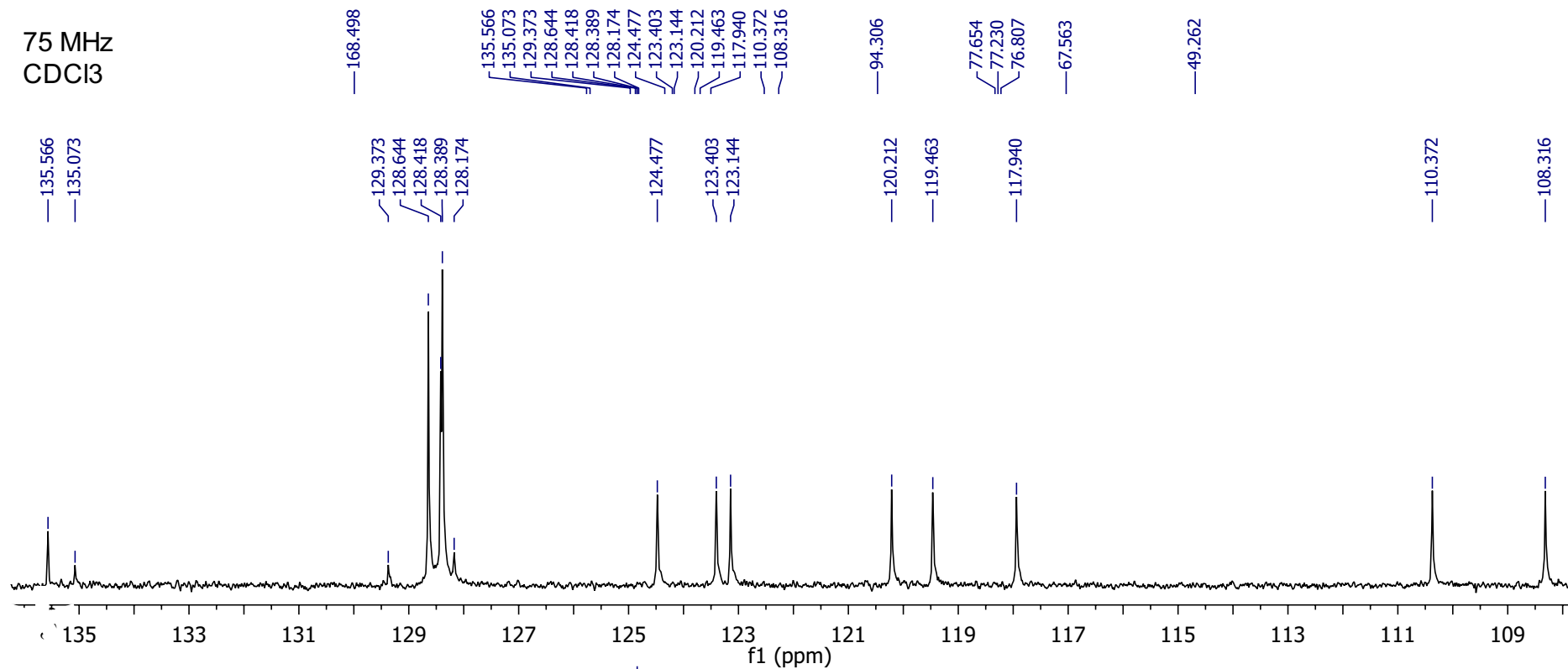


100 MHz
CDCl₃**8**

300 MHz
CDCl₃

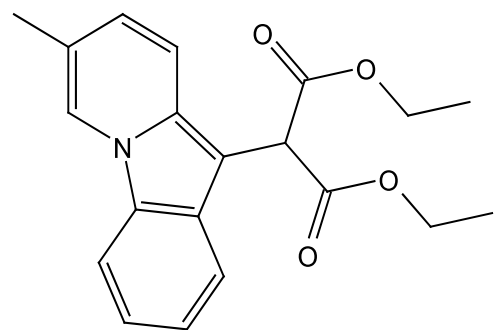
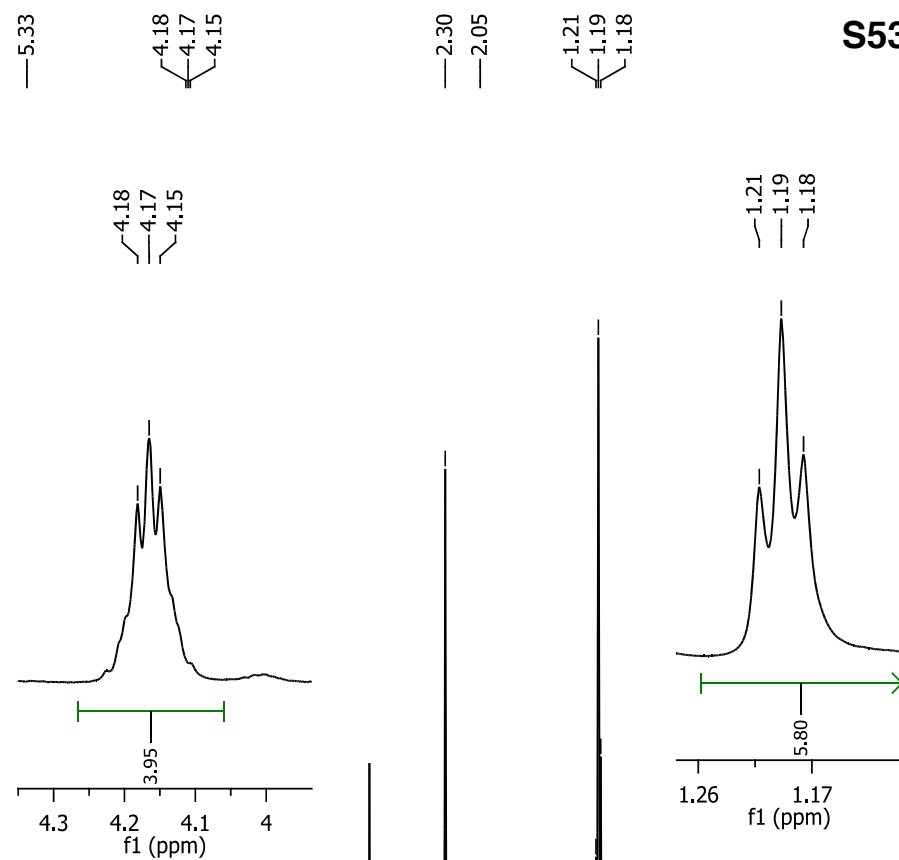
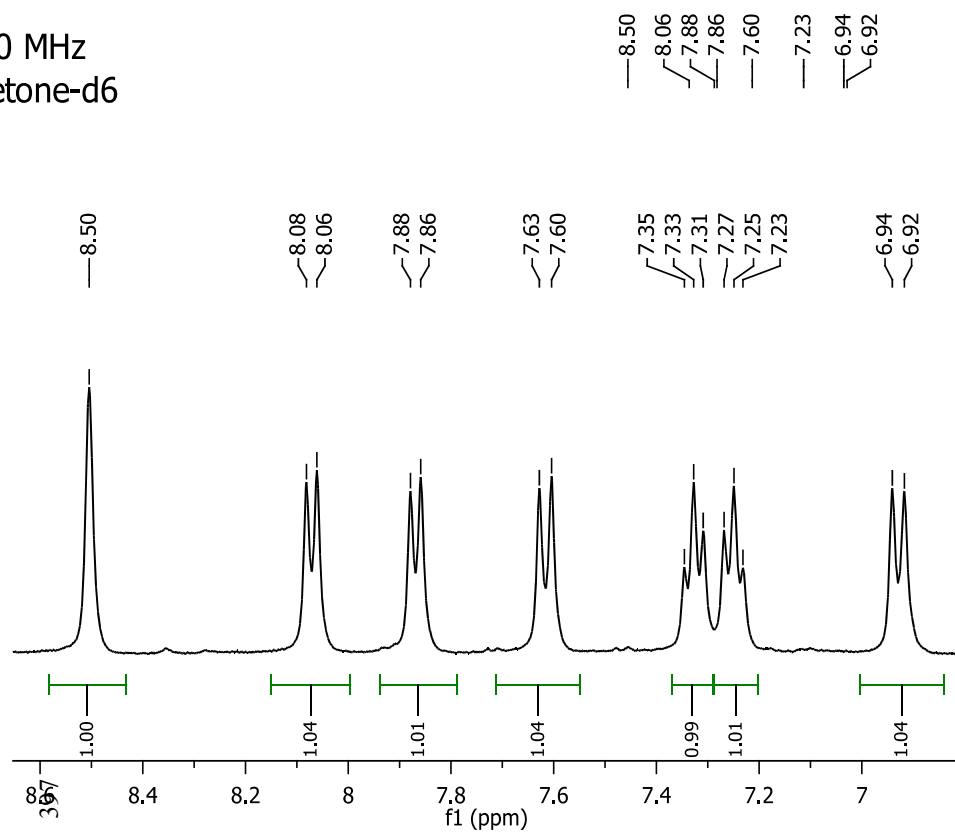


75 MHz
CDCl₃

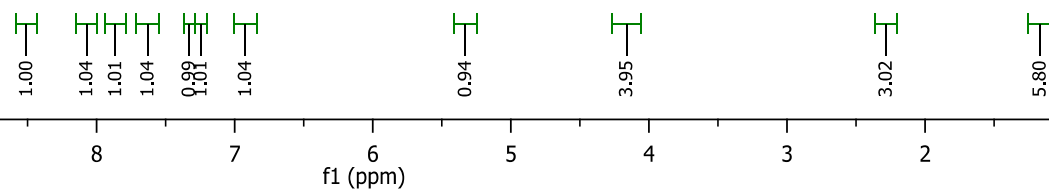


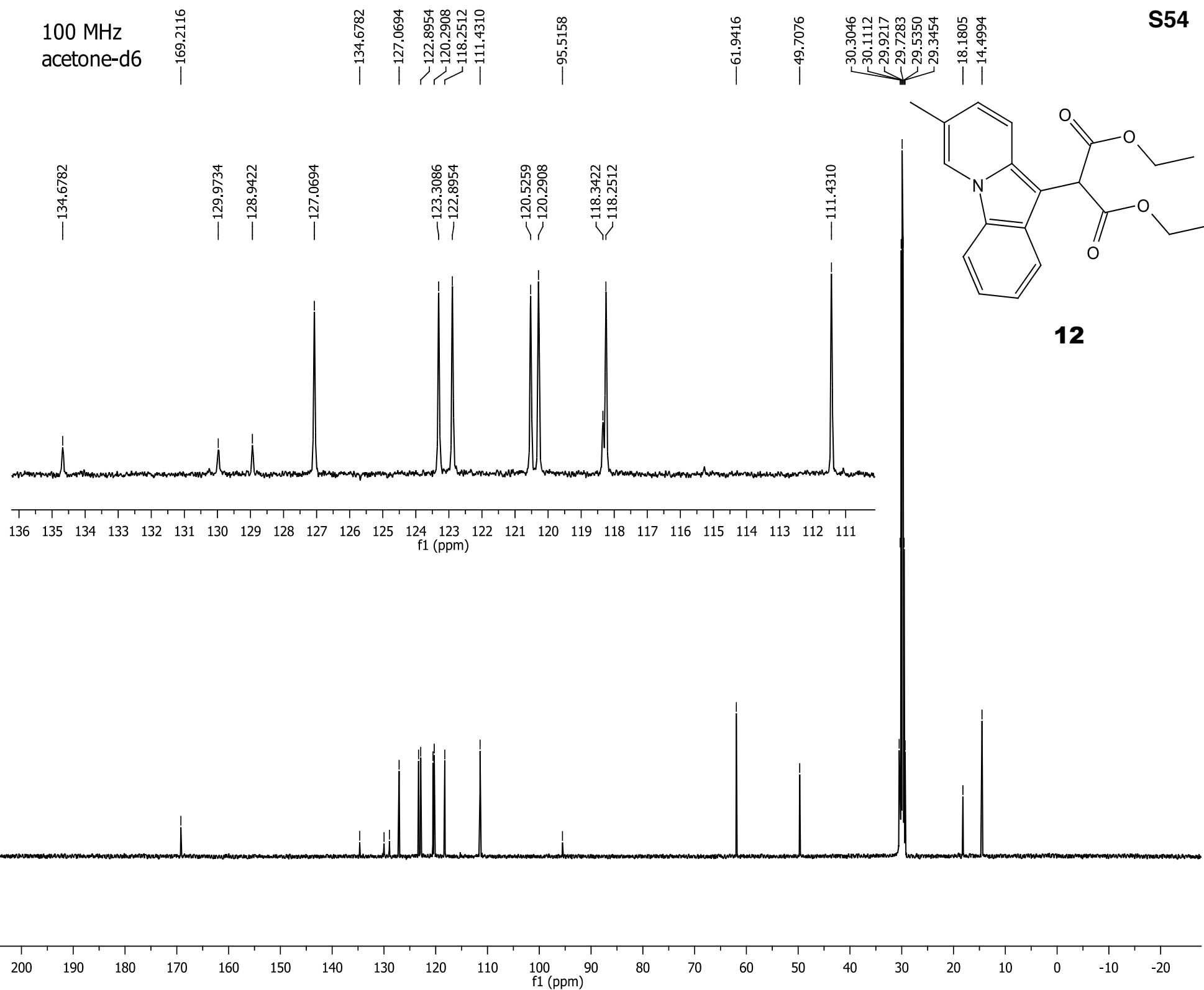
400 MHz
acetone-d₆

S53

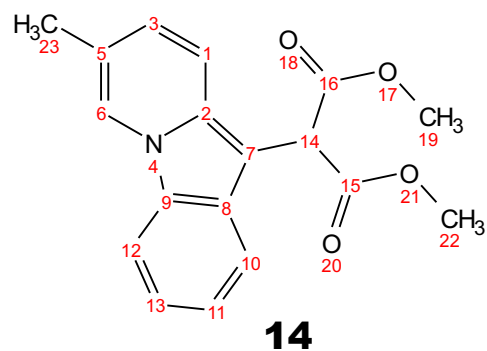


12

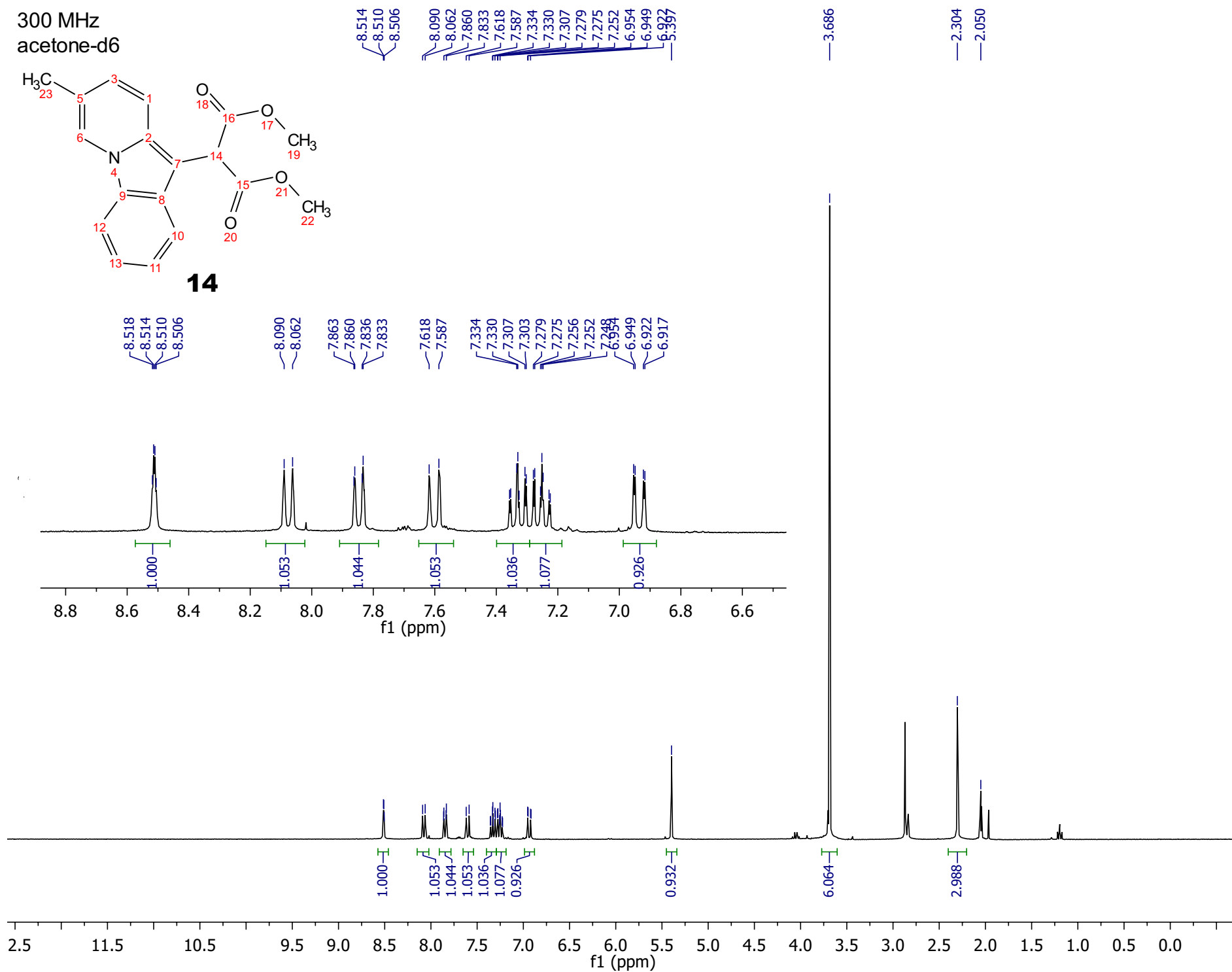


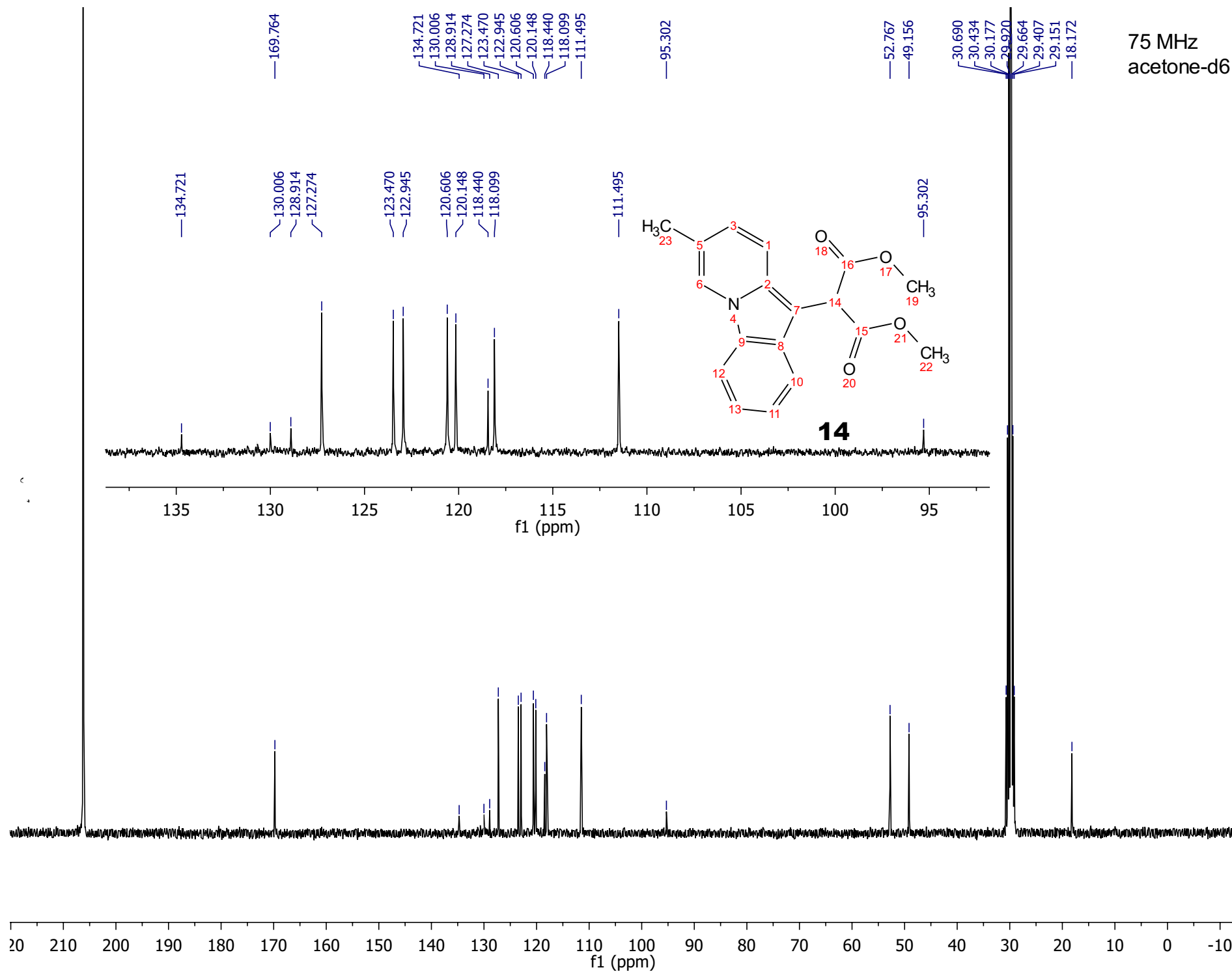
100 MHz
acetone-d₆

300 MHz
acetone-d₆

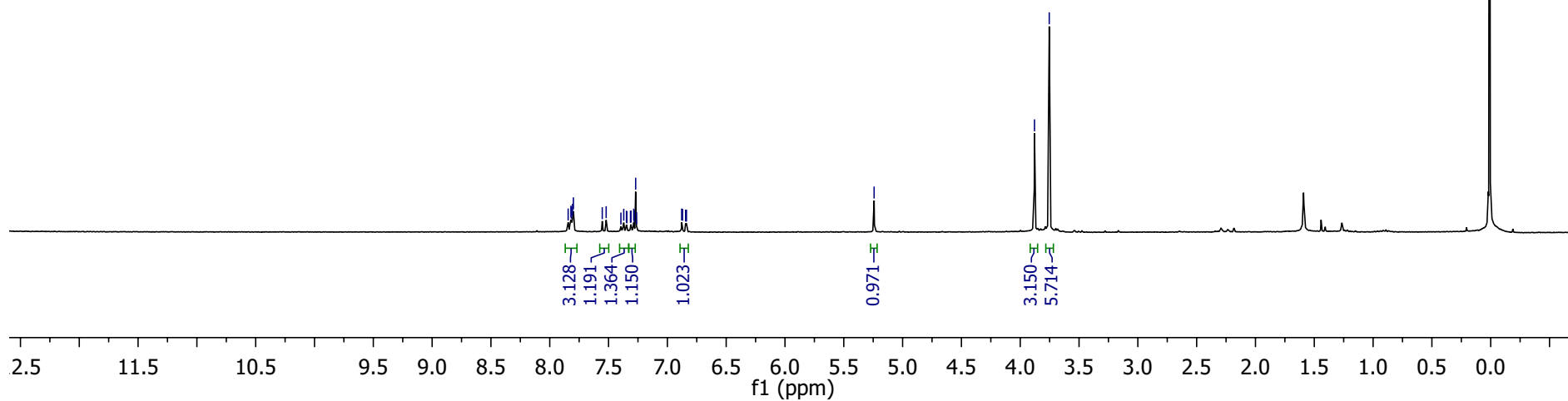
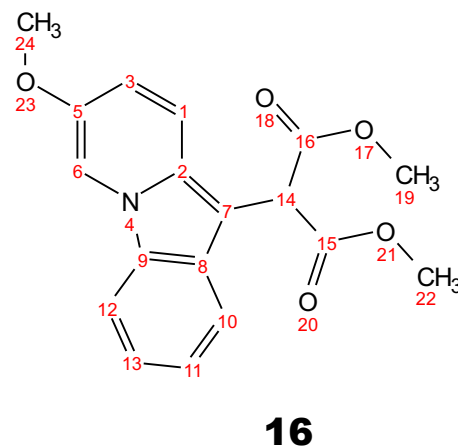
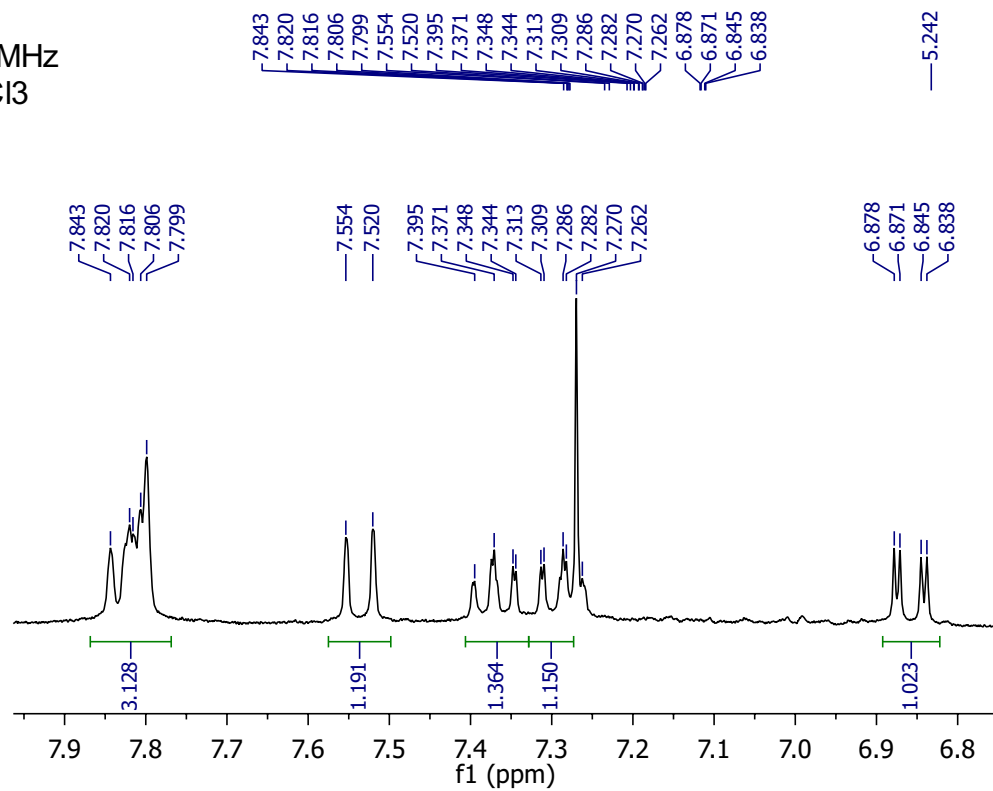


14

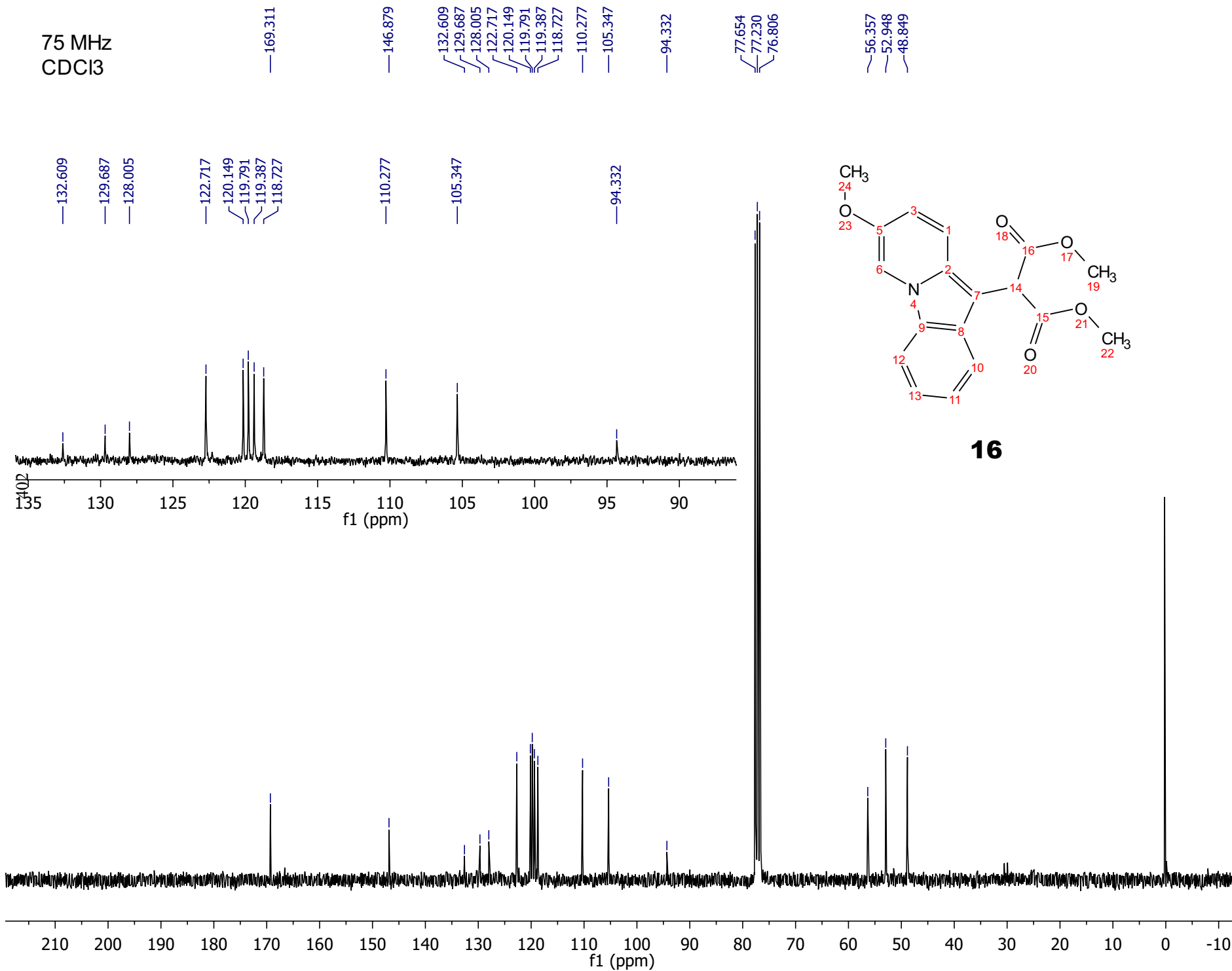


75 MHz
acetone-d₆

300 MHz
CDCl₃

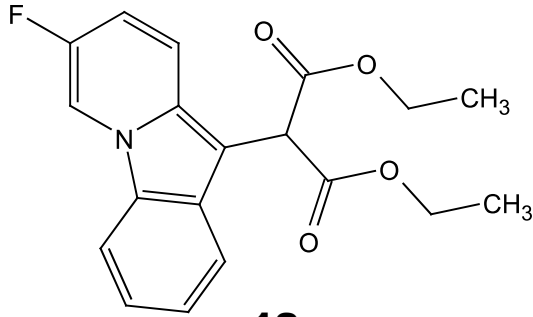


75 MHz
CDCl₃

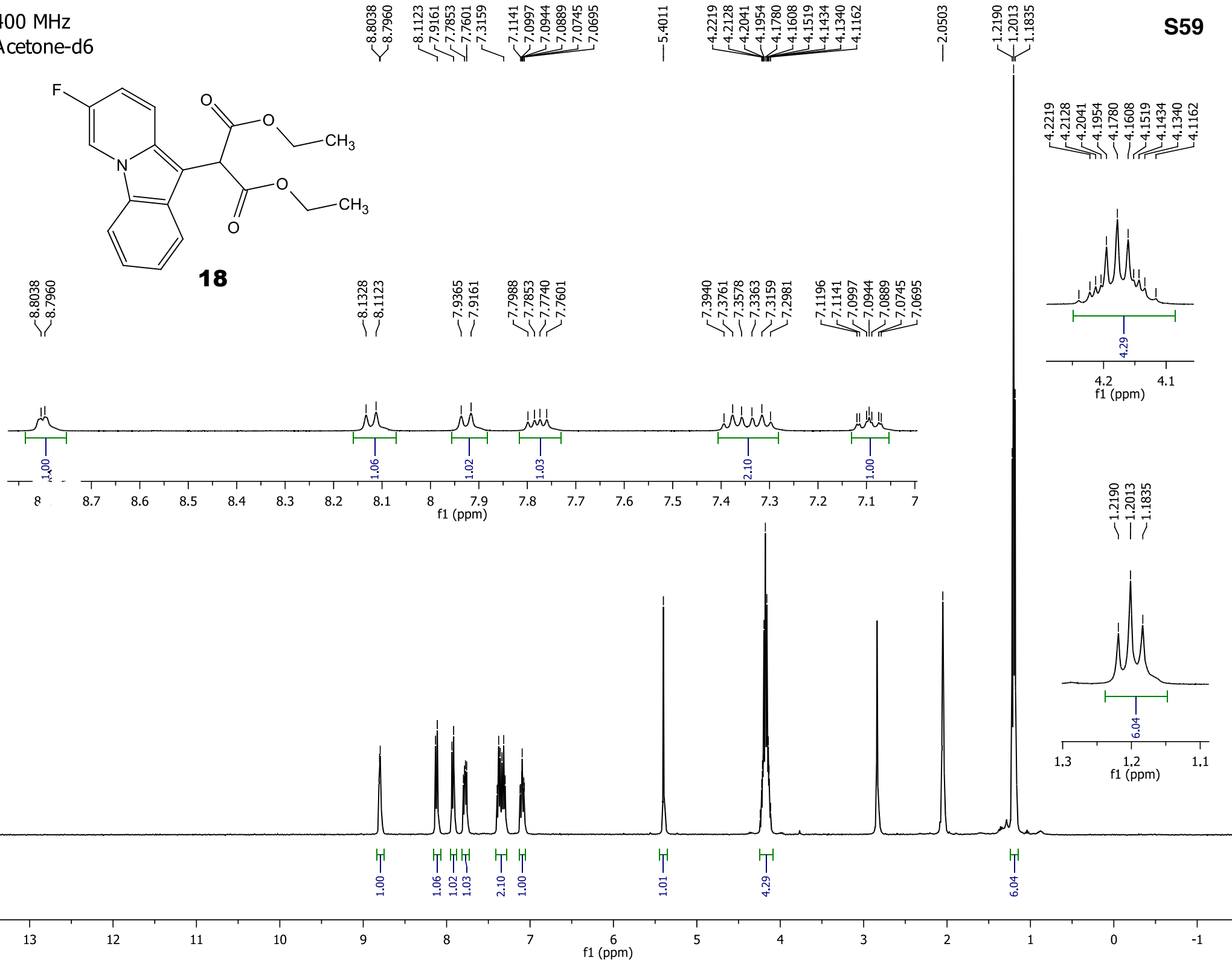


400 MHz
Acetone-d6

S59

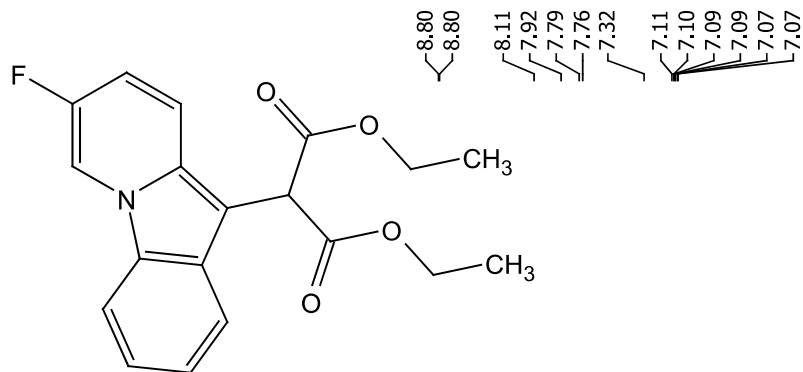


18

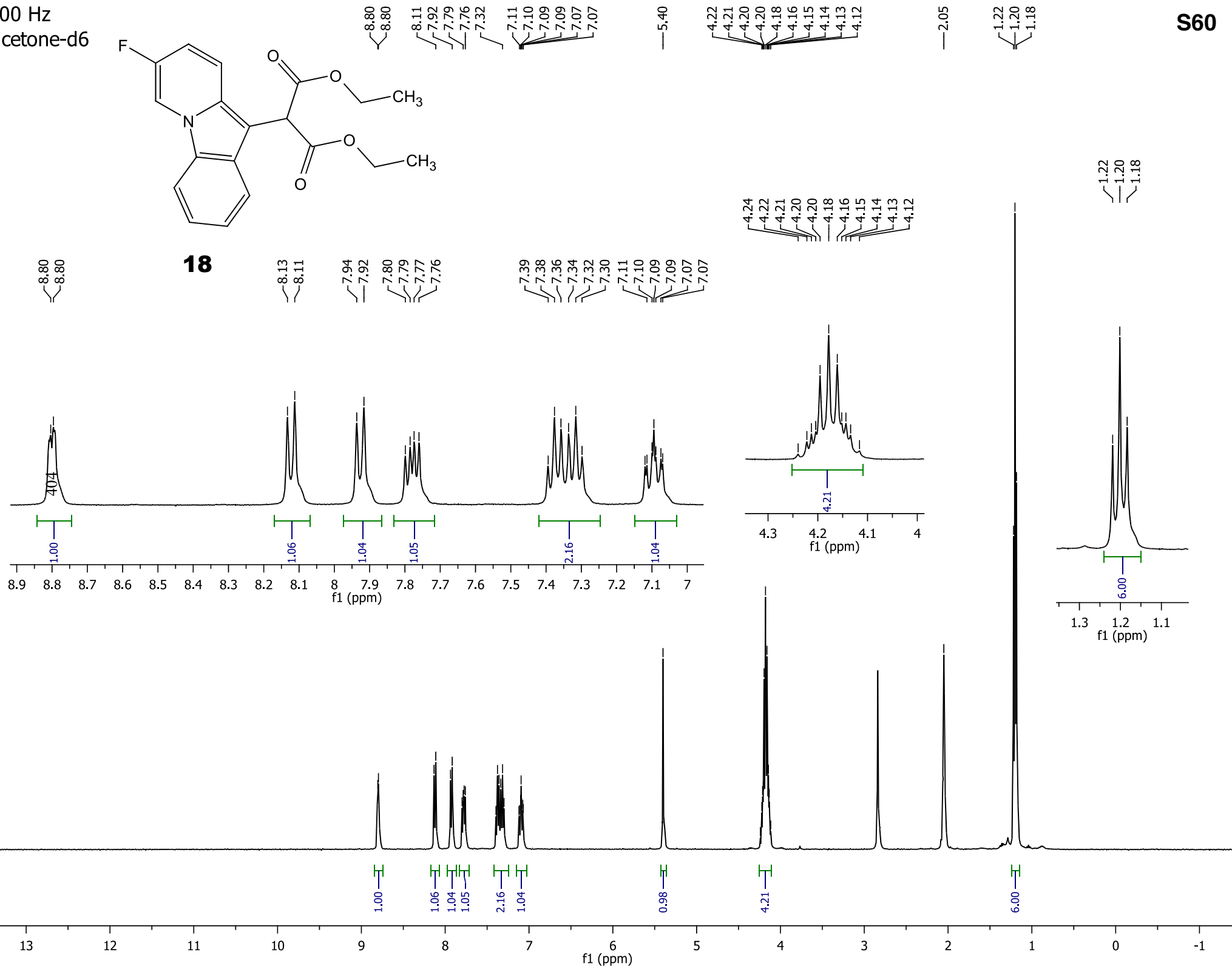


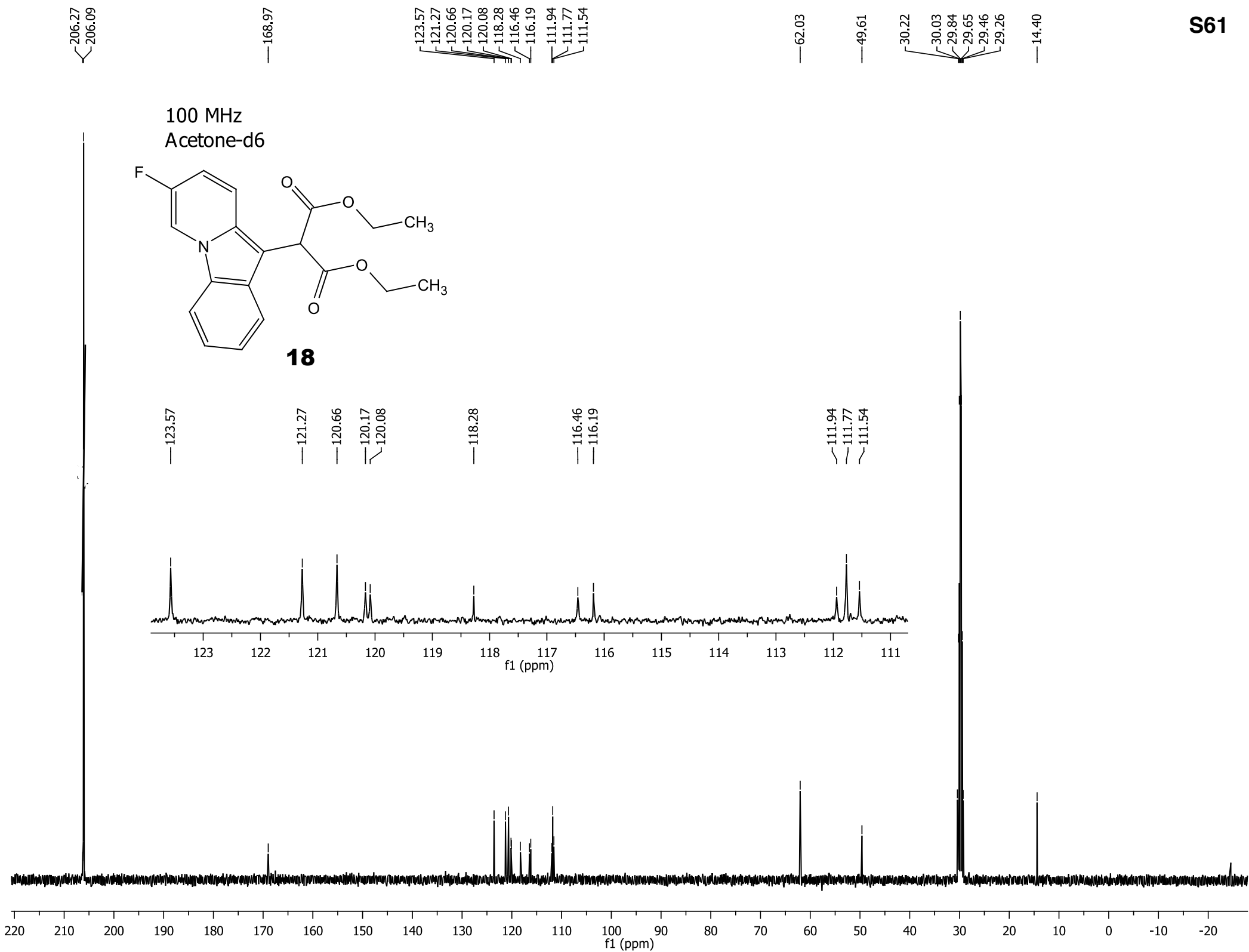
400 Hz
Acetone-d6

S60



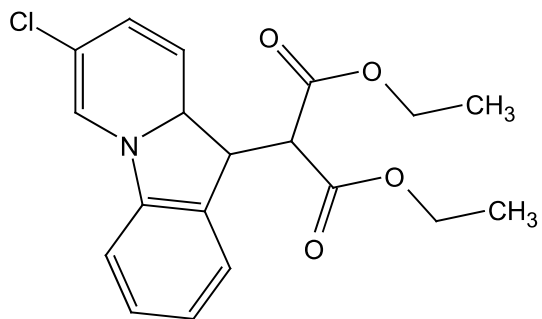
18



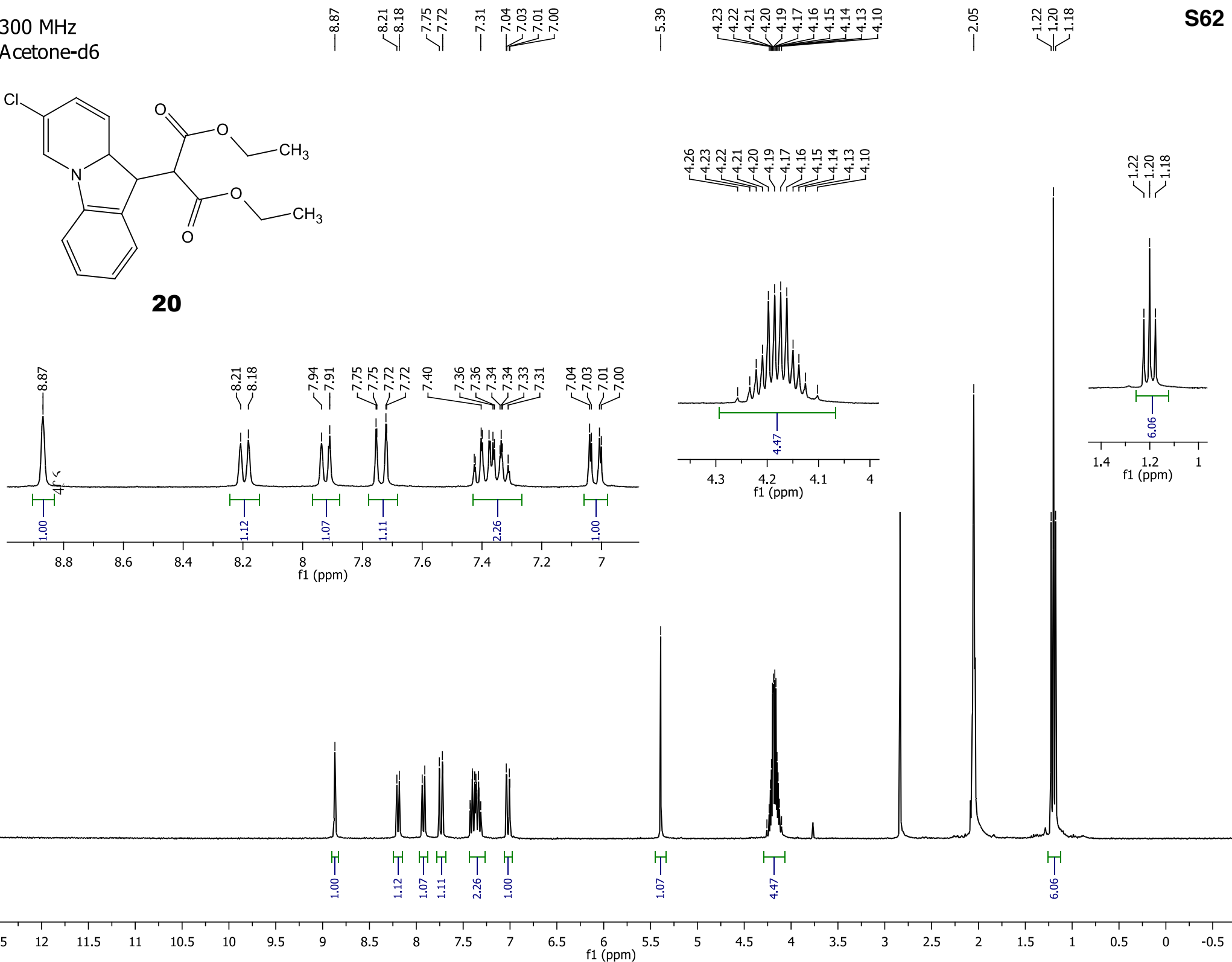


300 MHz
Acetone-d6

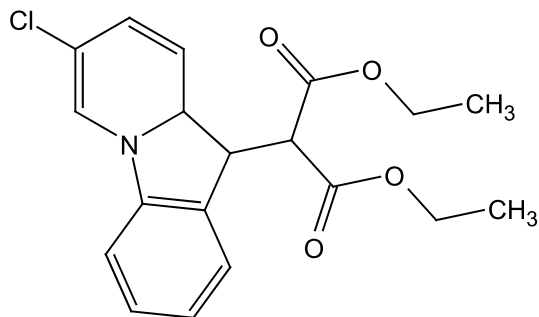
S62



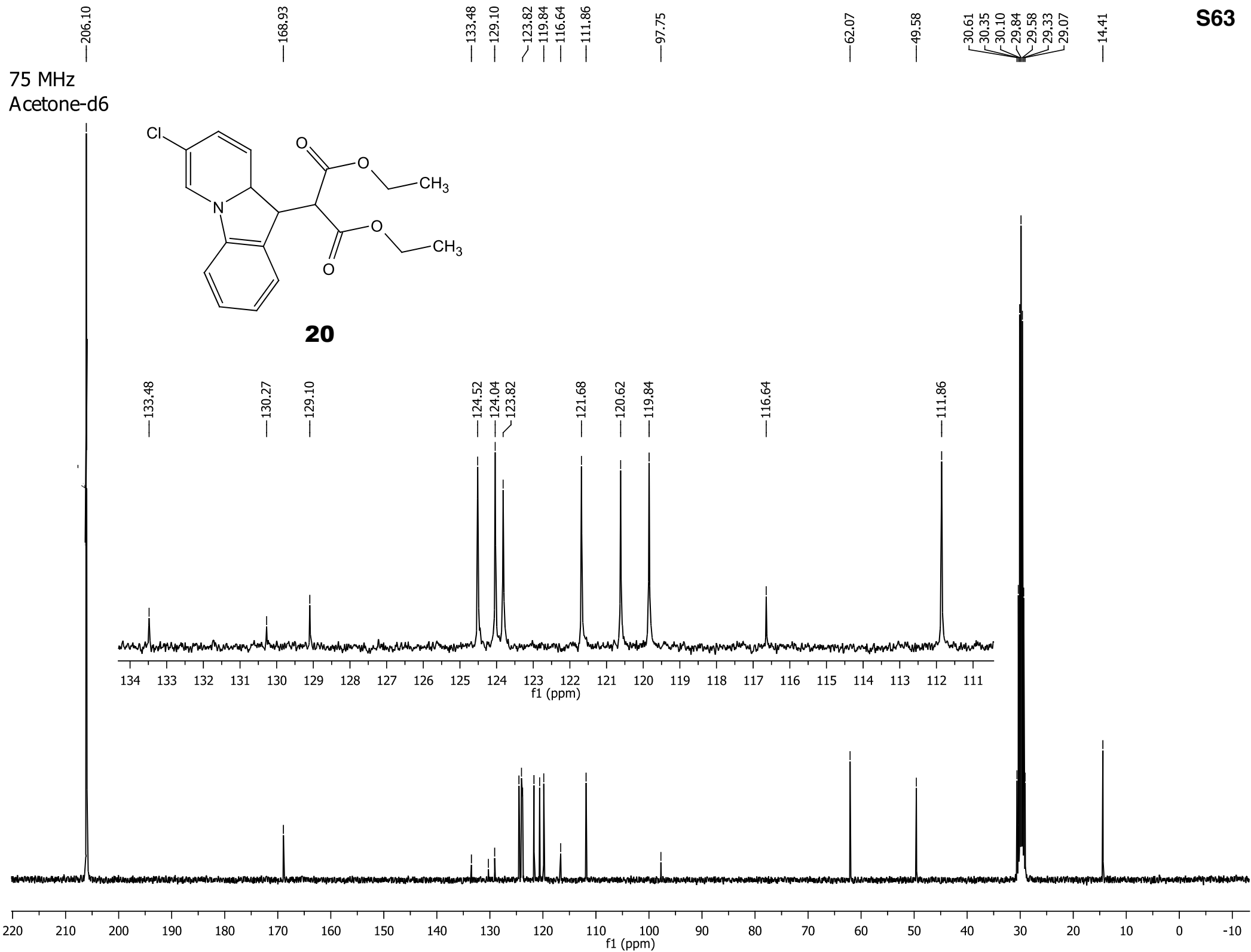
20

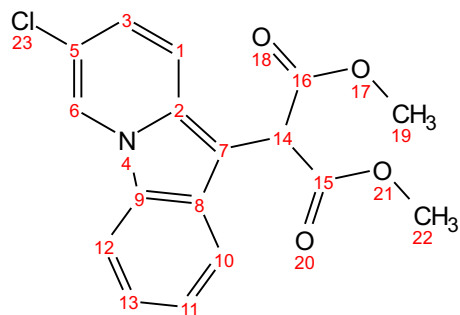


75 MHz
Acetone-d₆



20





— 8.217
— 8.191

7.916
7.914

7.890
7.887
7.883

7.741
7.738
7.709

7.424

7.402
7.379

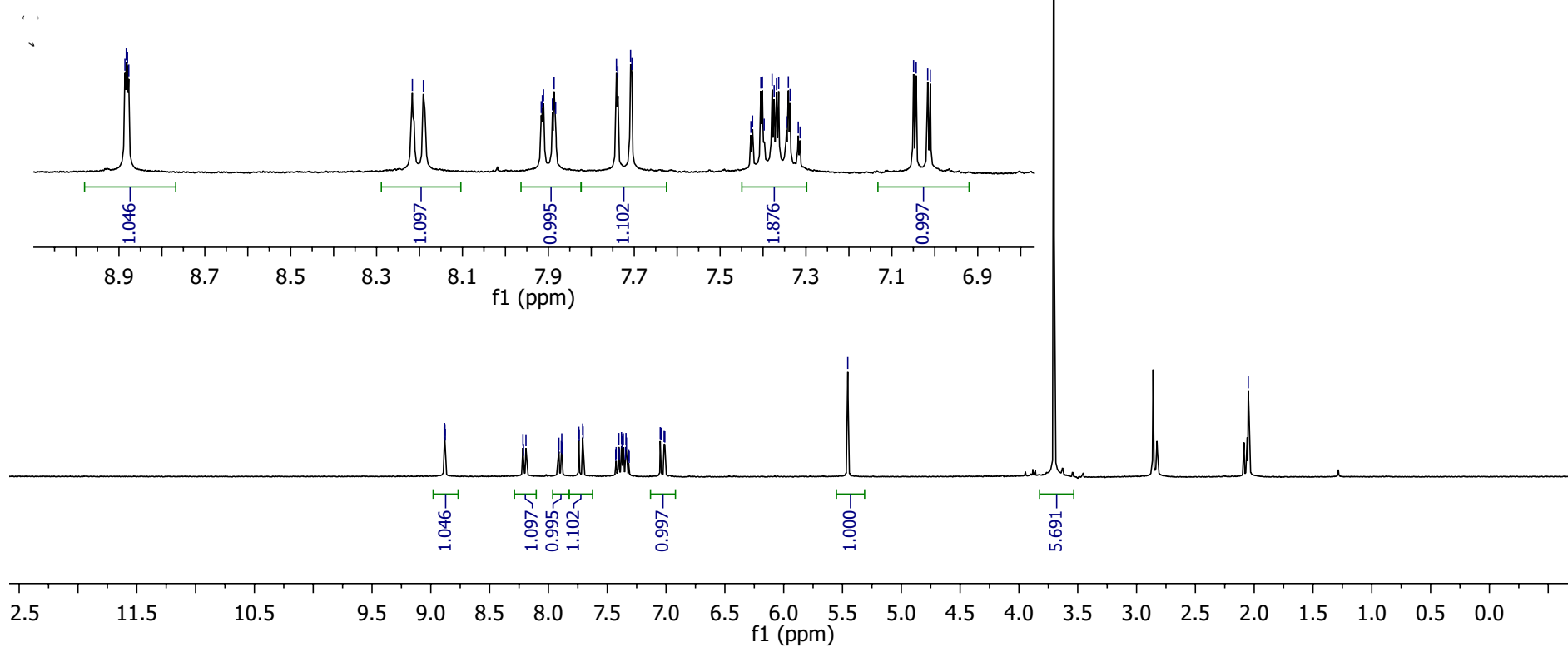
7.369
7.364
7.34E

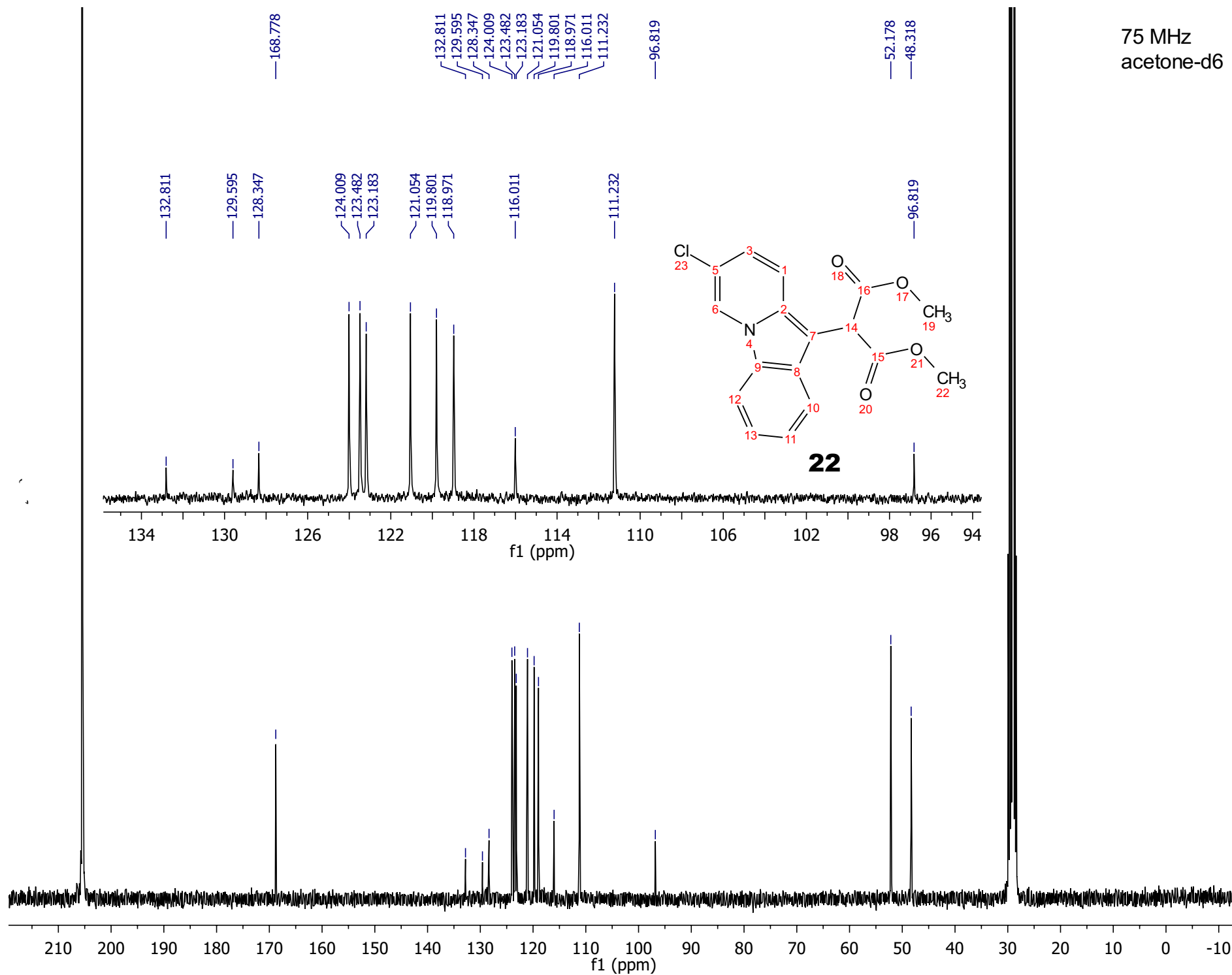
7.341
7.337
7.049

7.017
7.010

—3.701

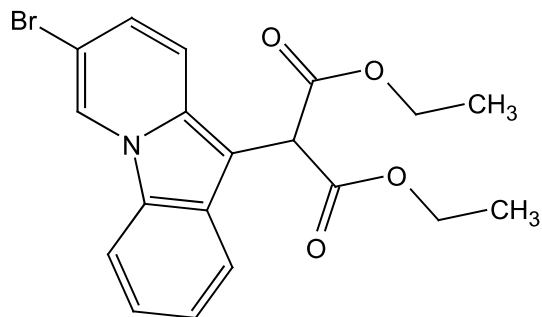
—2.050



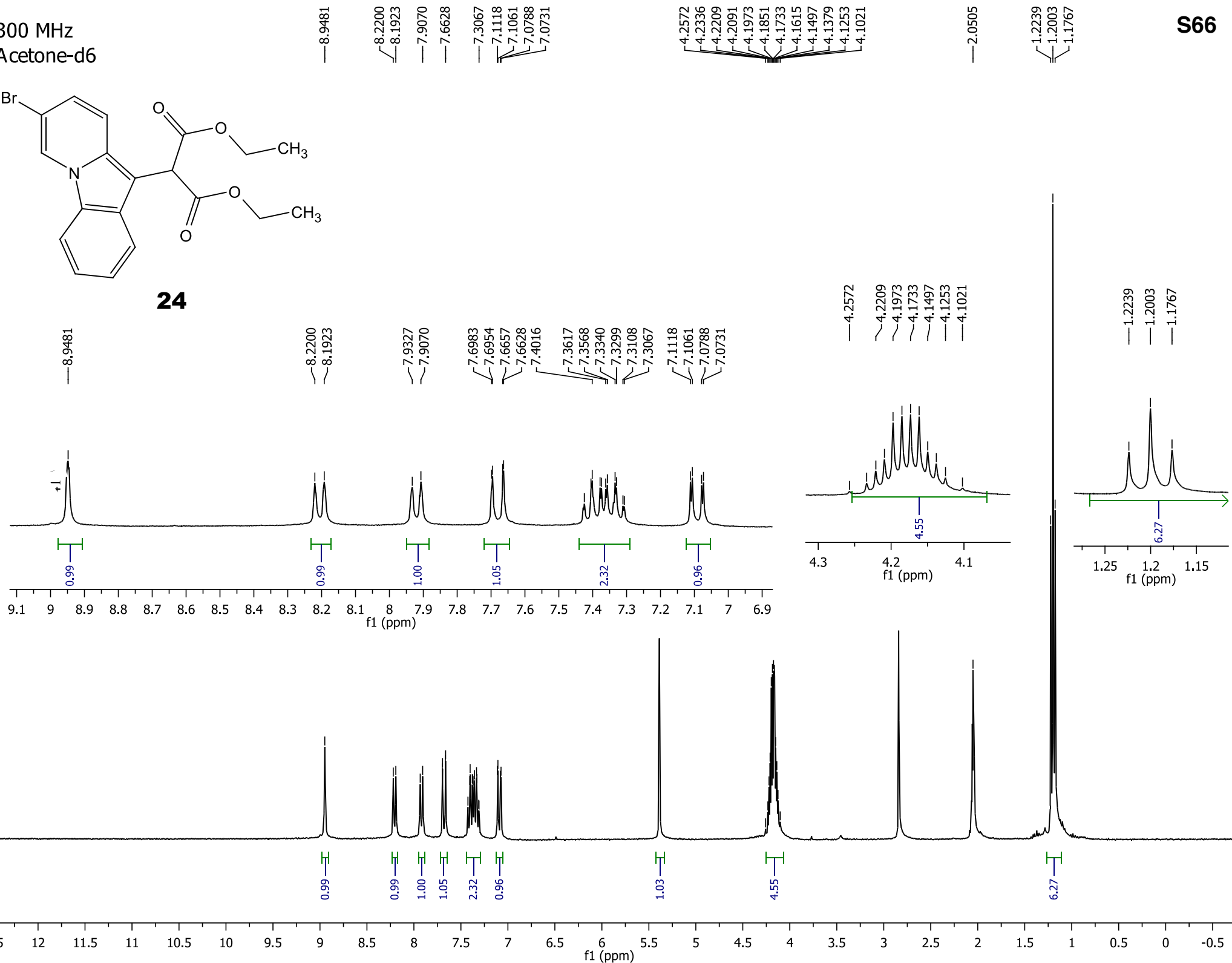
75 MHz
acetone-d₆

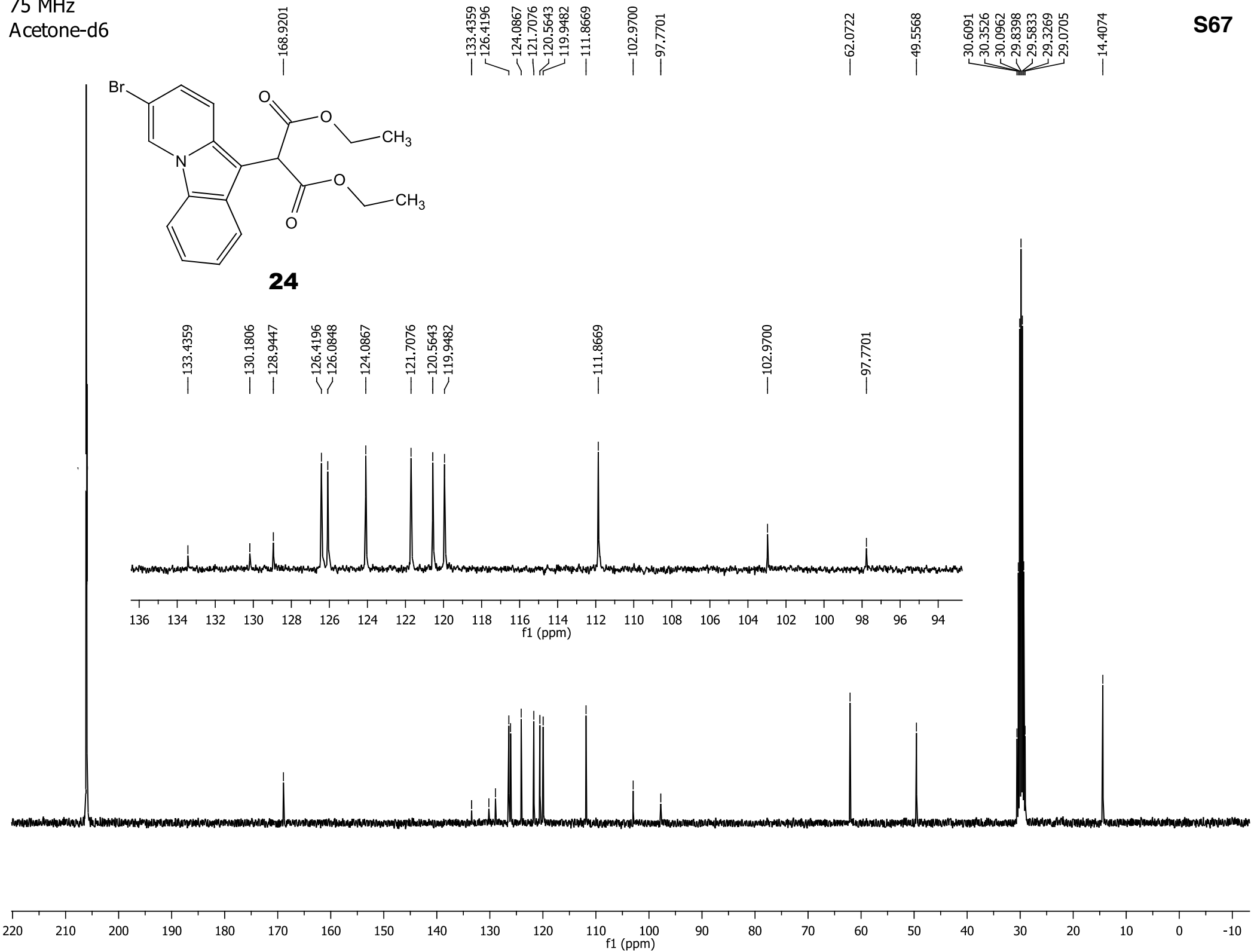
300 MHz
Acetone-d6

S66

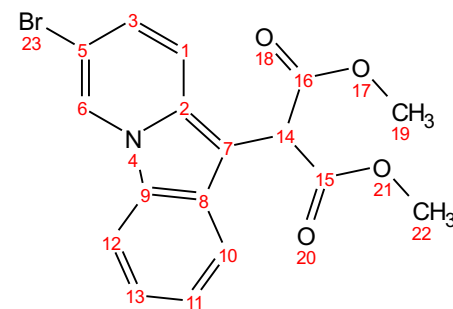
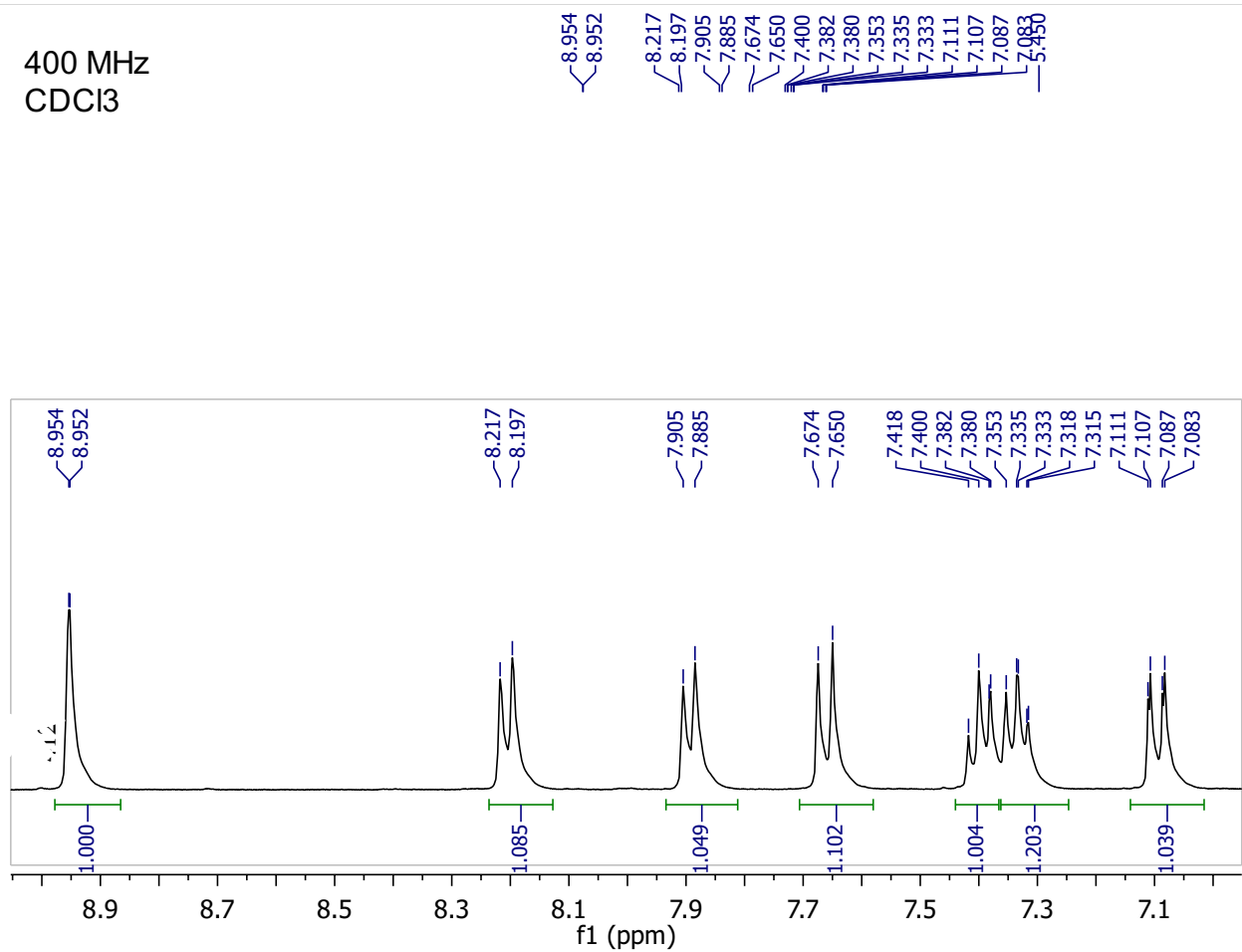


24

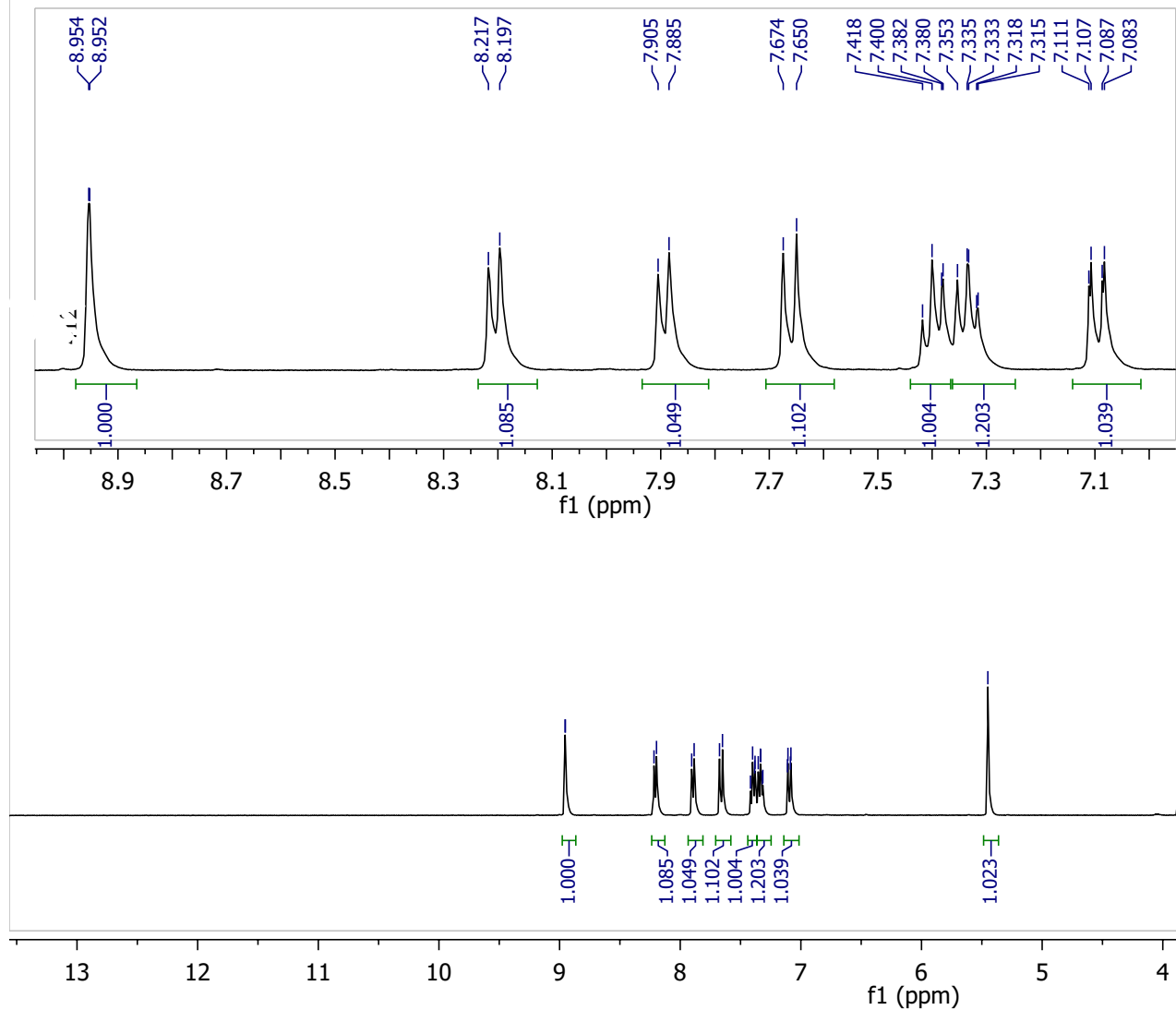


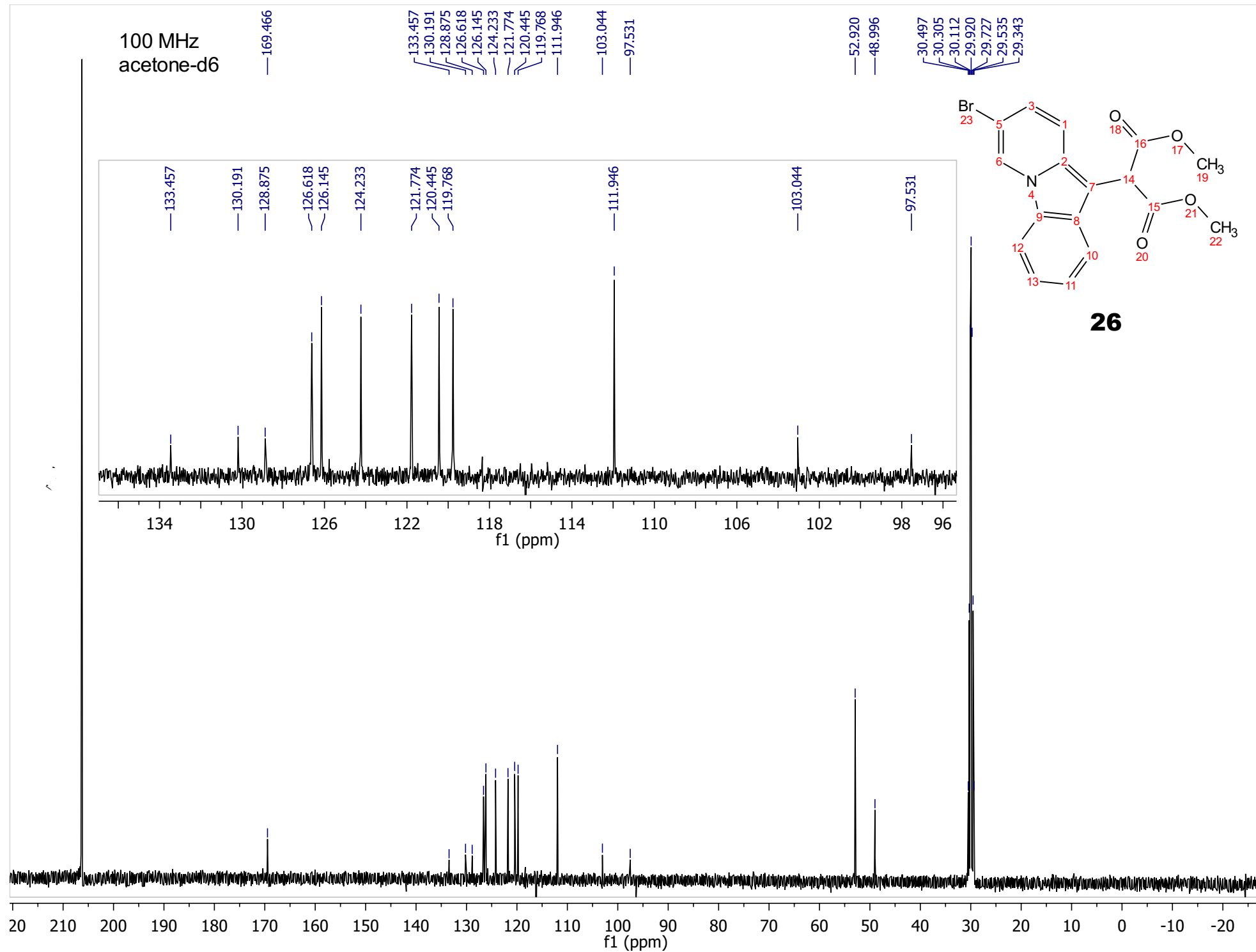


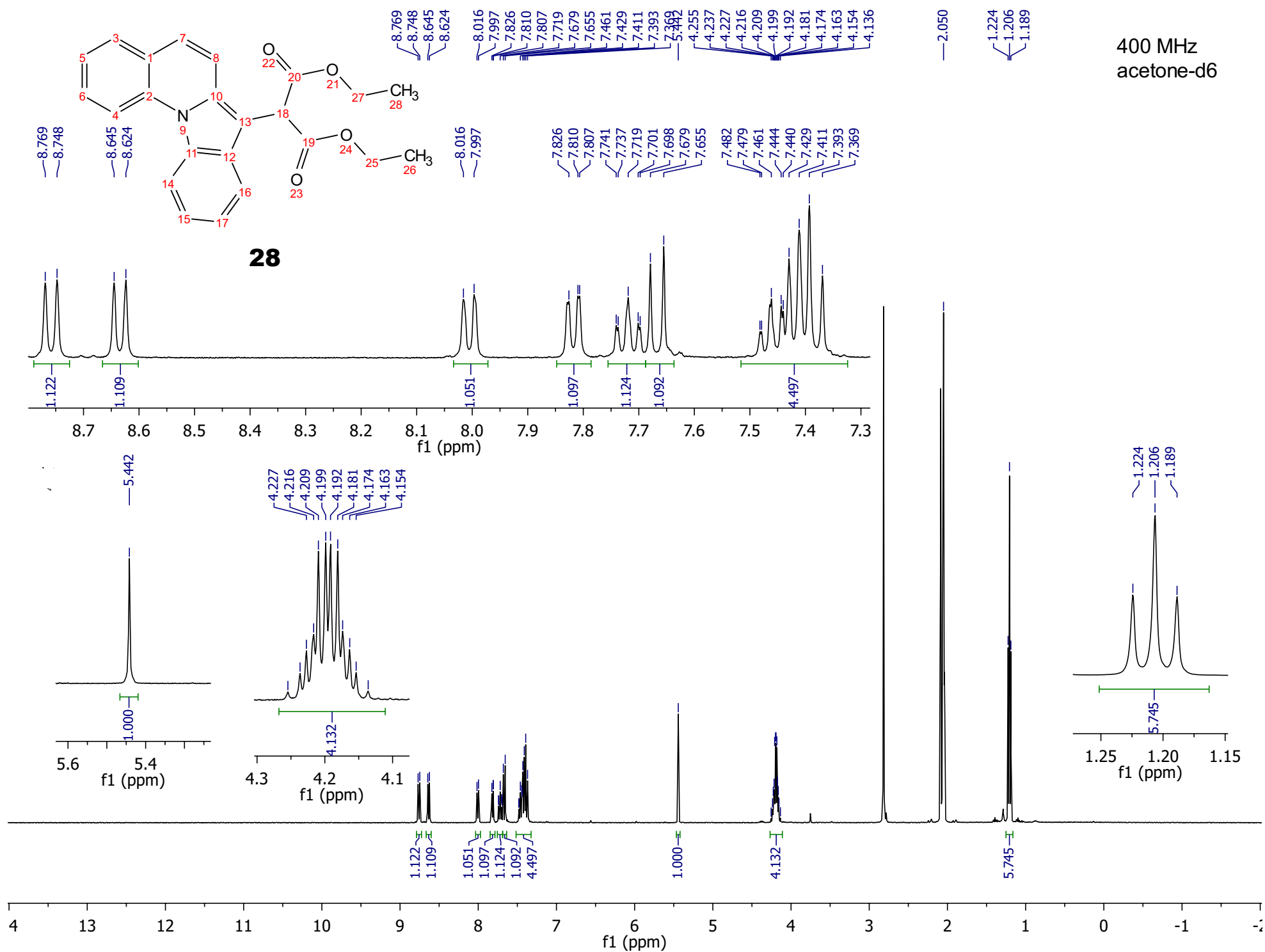
400 MHz
CDCl₃



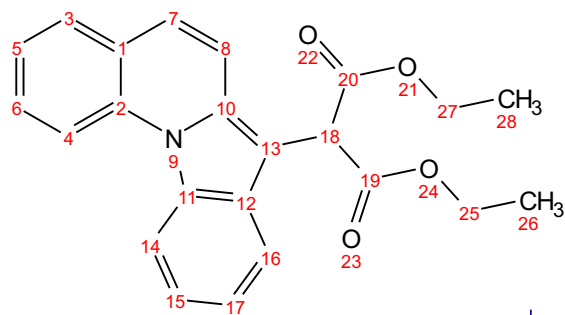
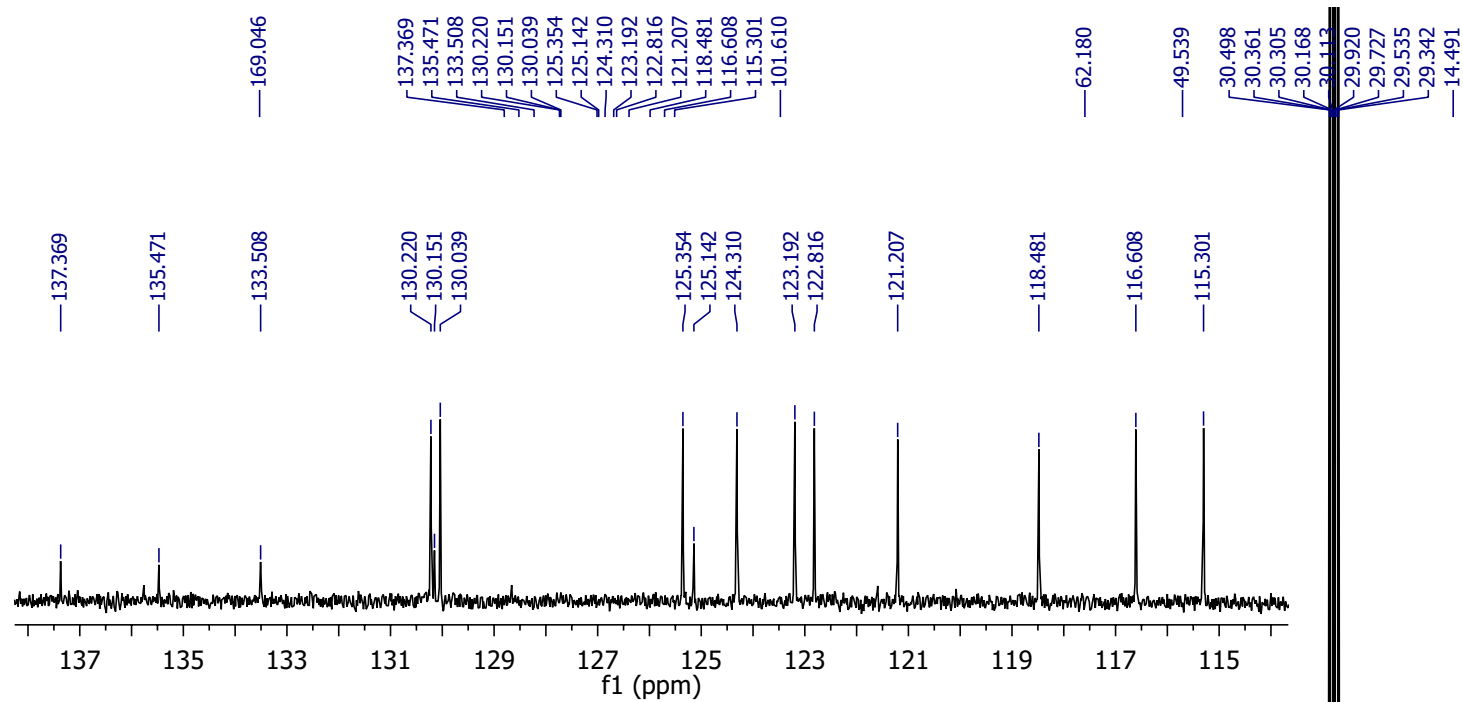
26



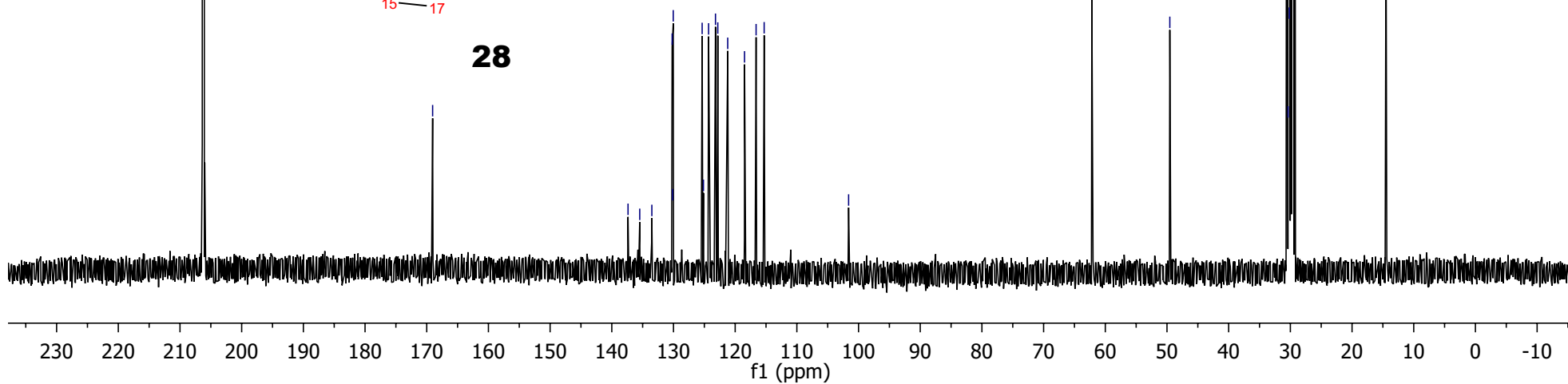


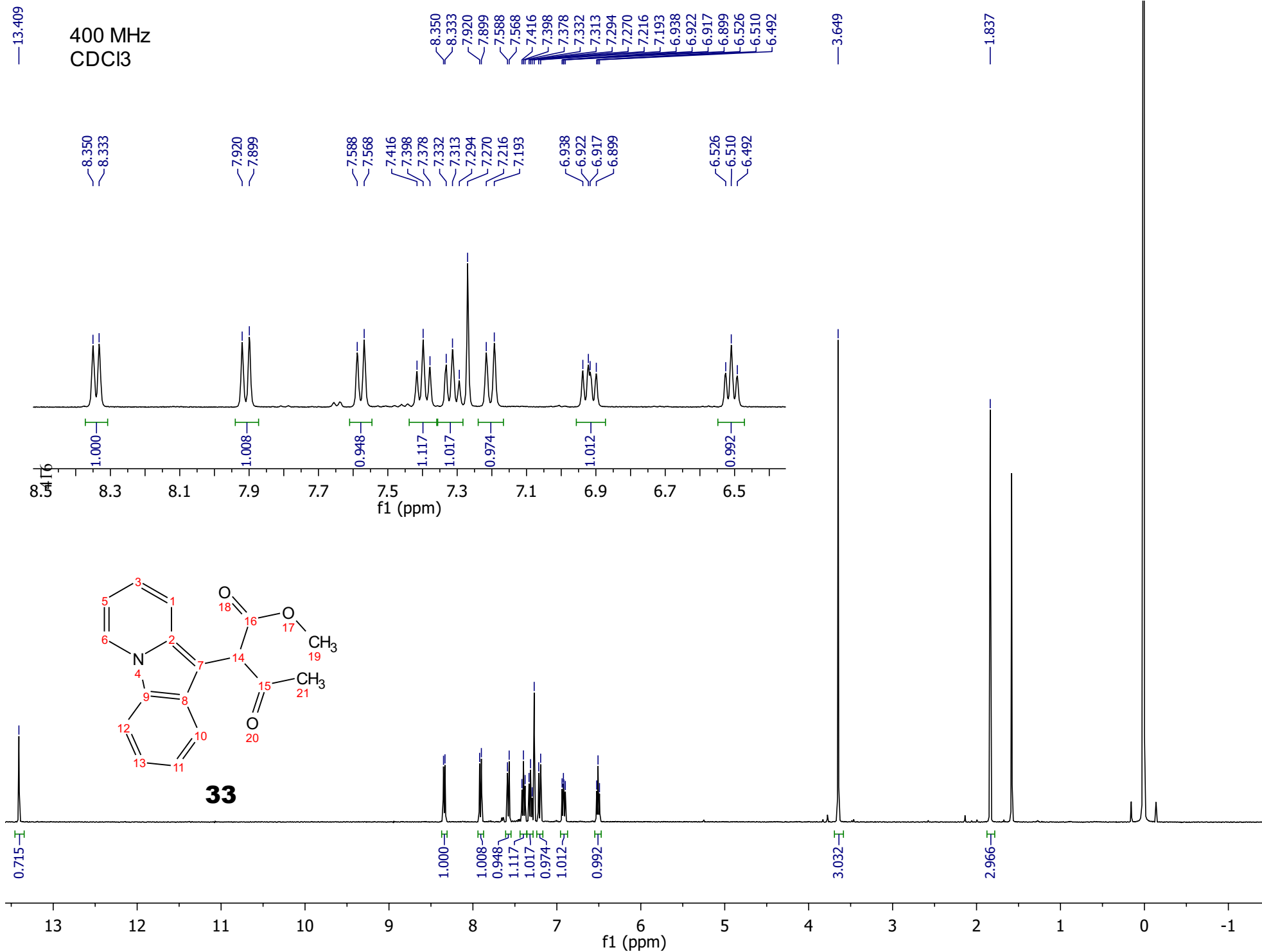
400 MHz
acetone-d₆**28**

100 MHz
acetone-d₆

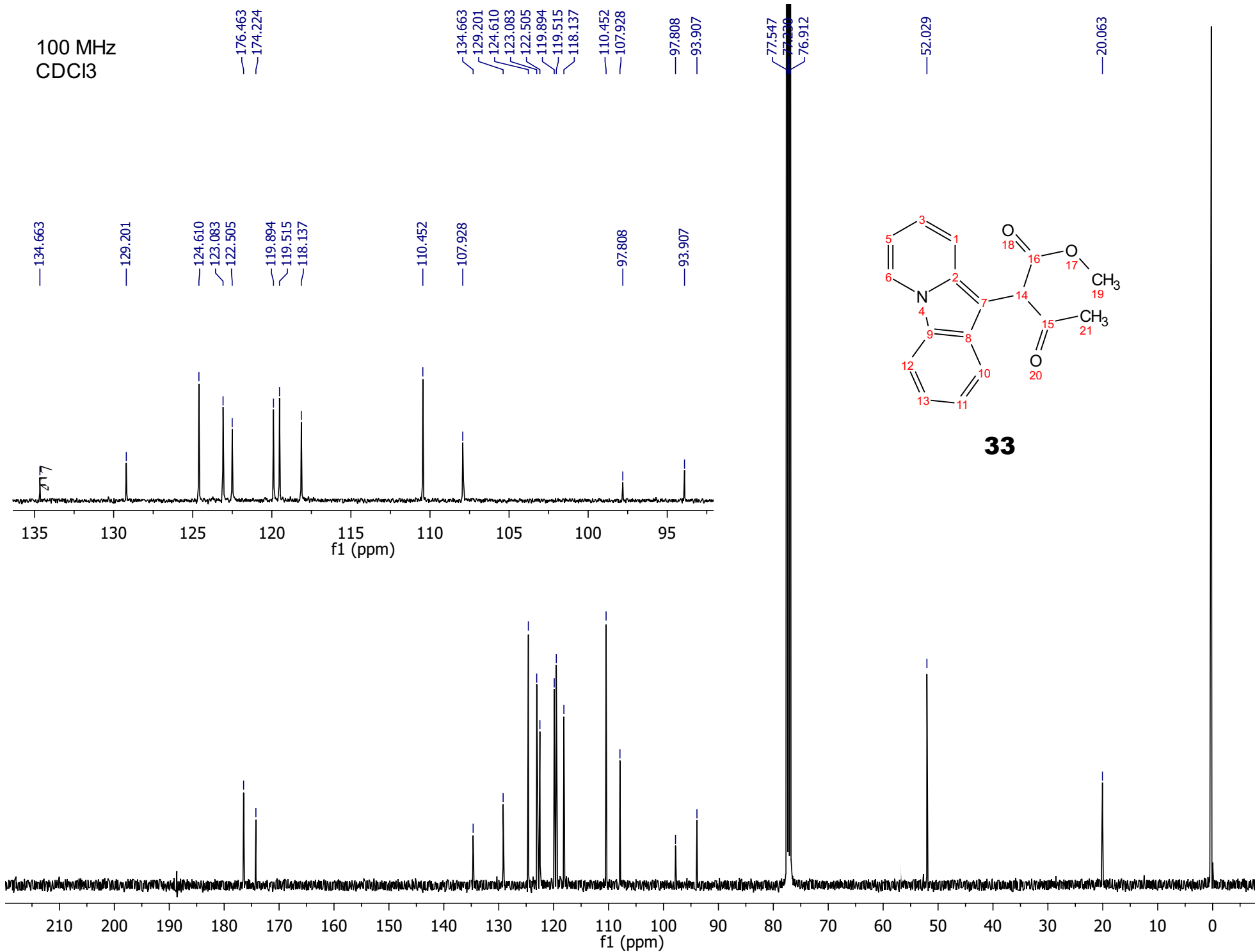


28

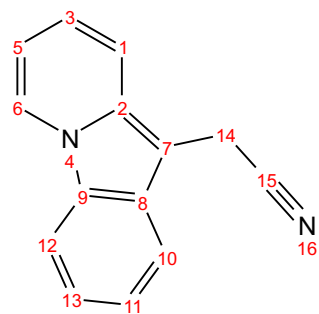
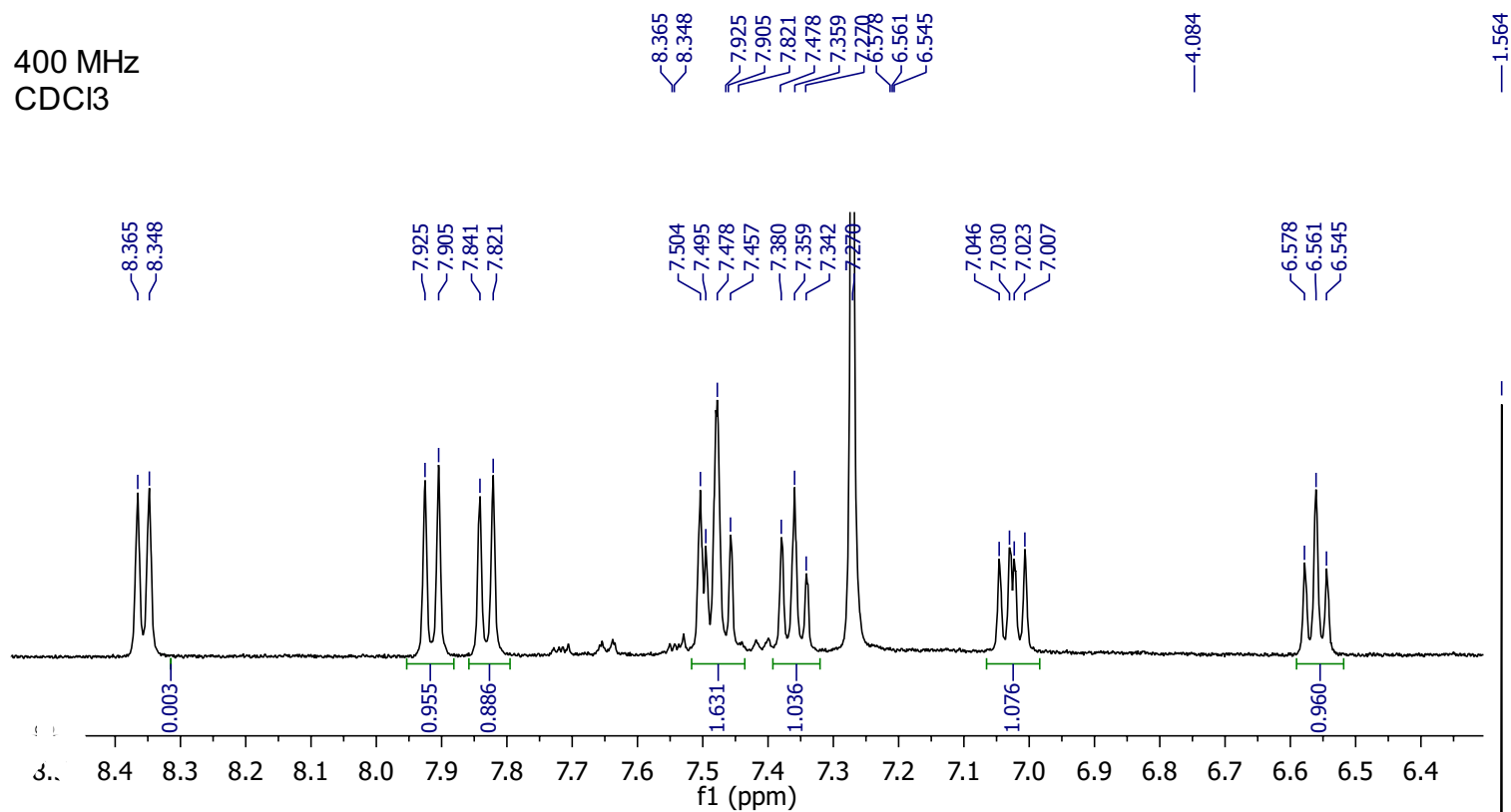




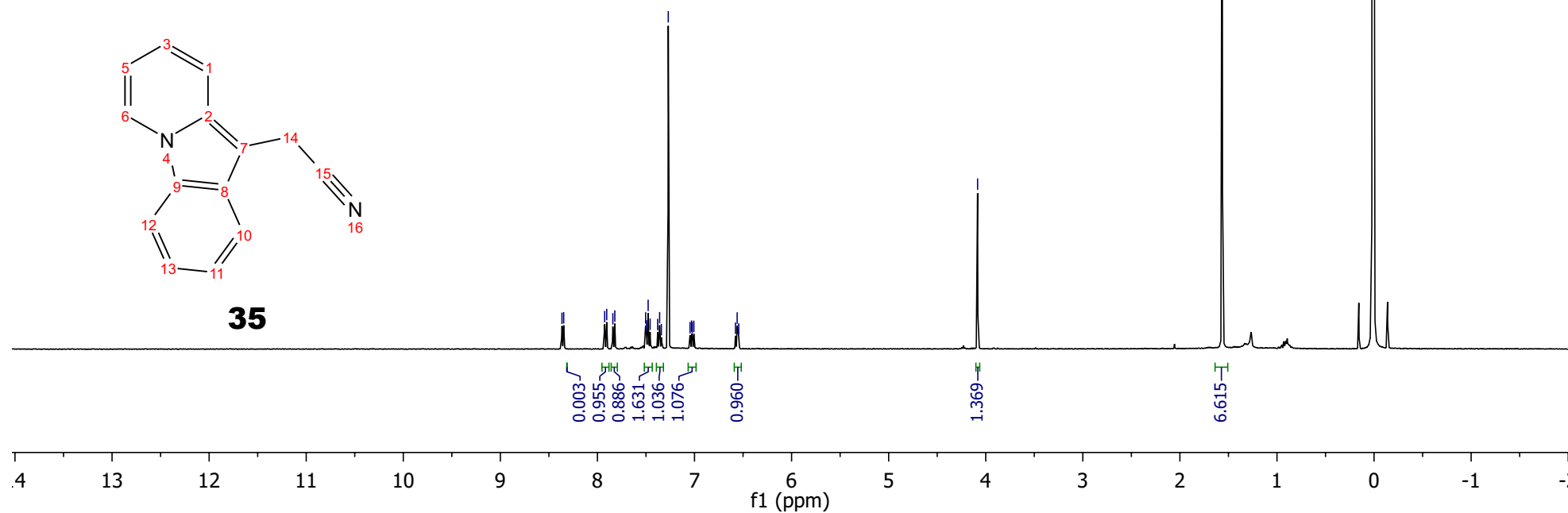
100 MHz
CDCl₃



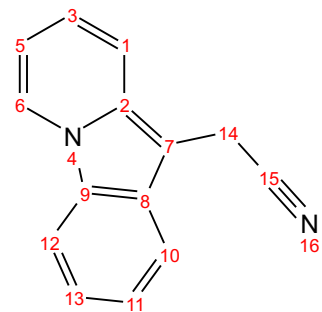
400 MHz
CDCl₃



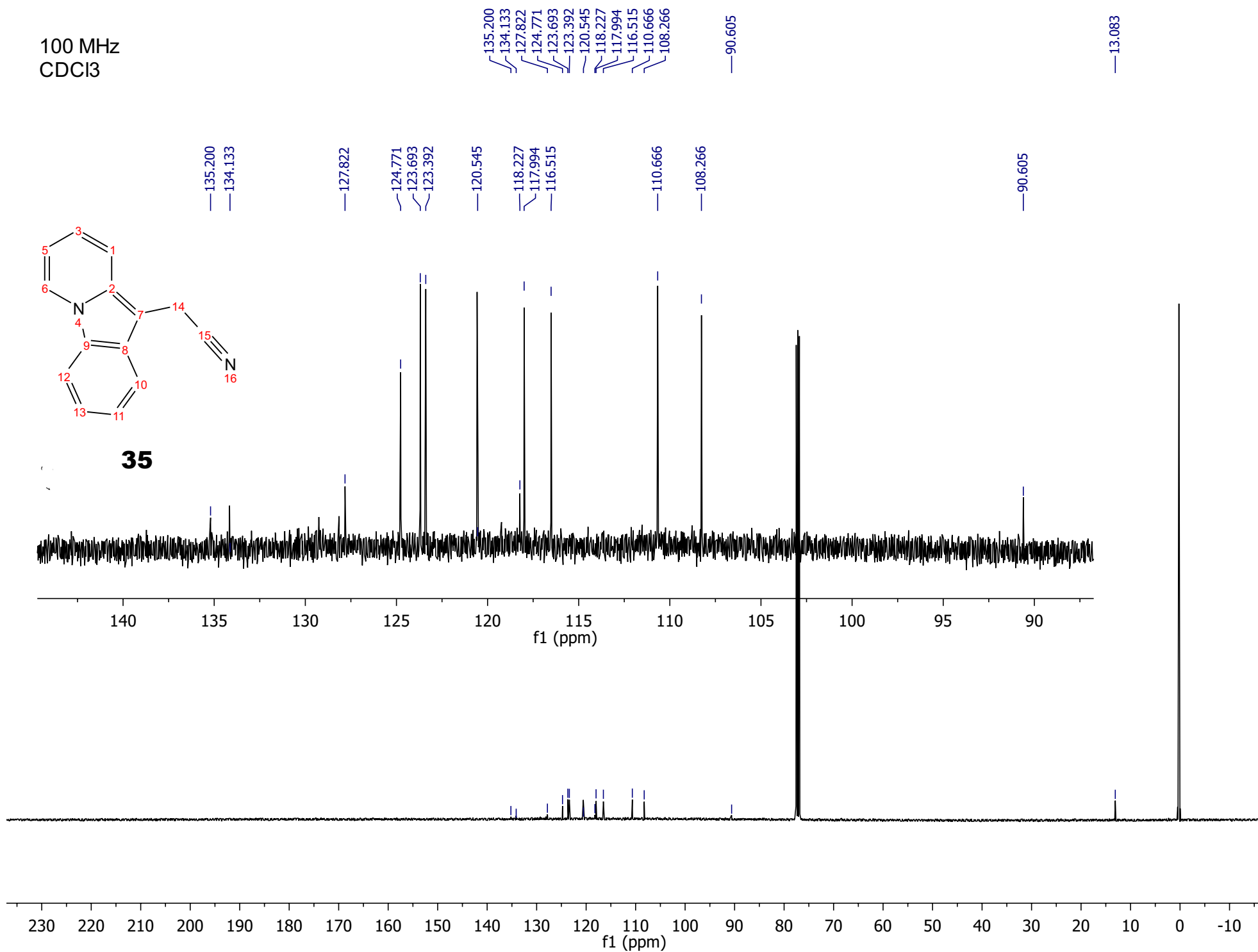
35



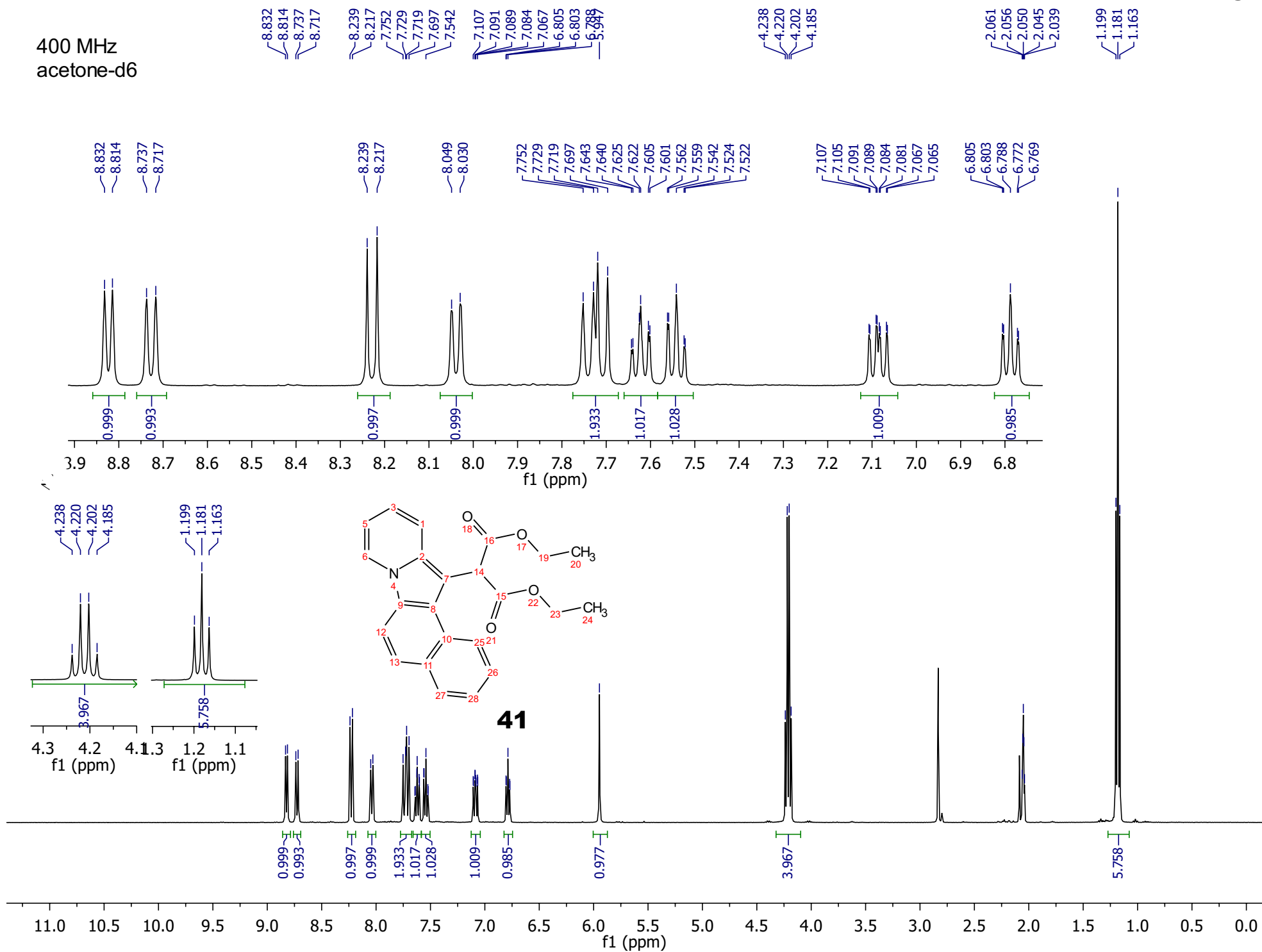
100 MHz
CDCl₃



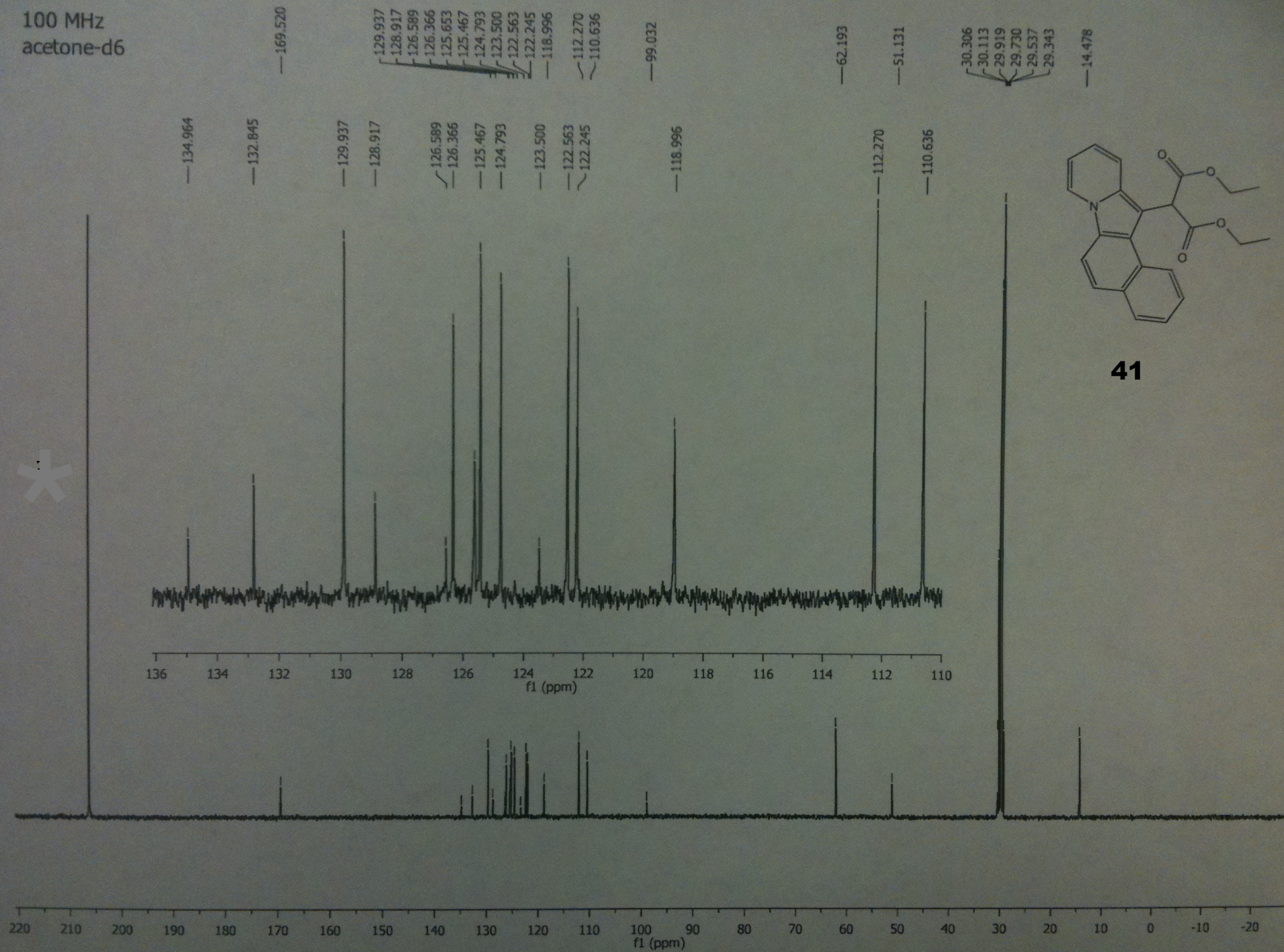
35



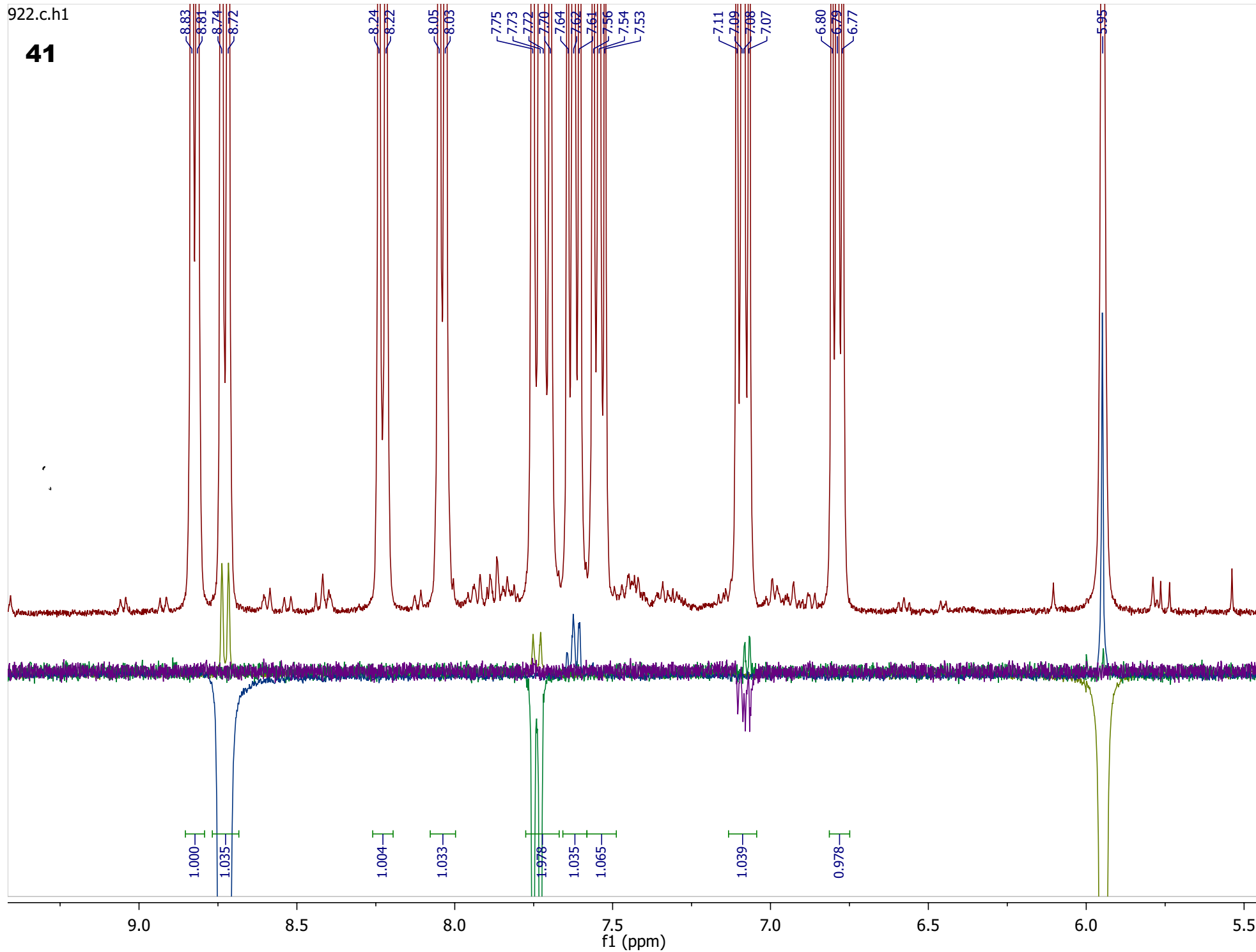
400 MHz
acetone-d₆



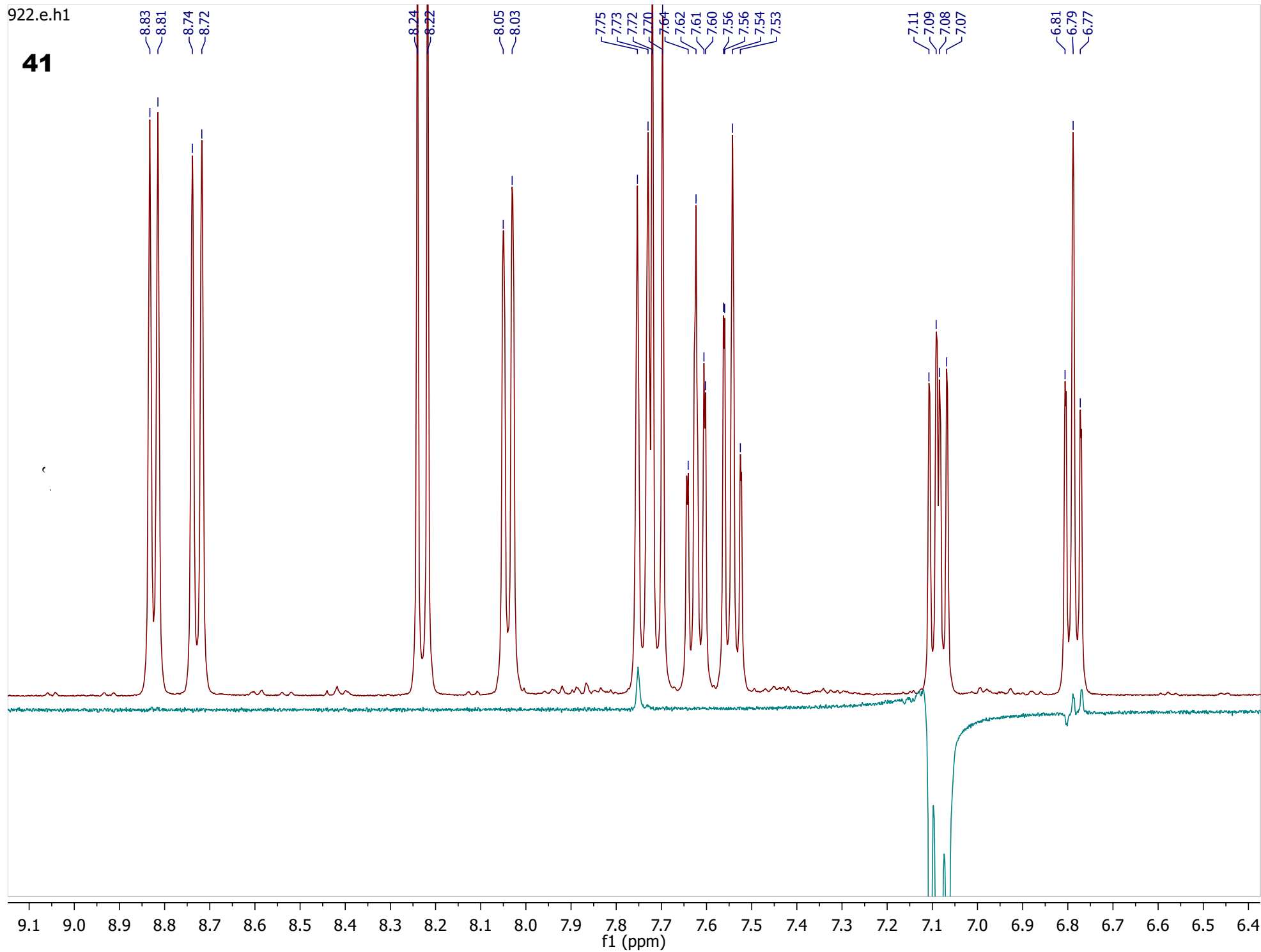
100 MHz
acetone-d₆



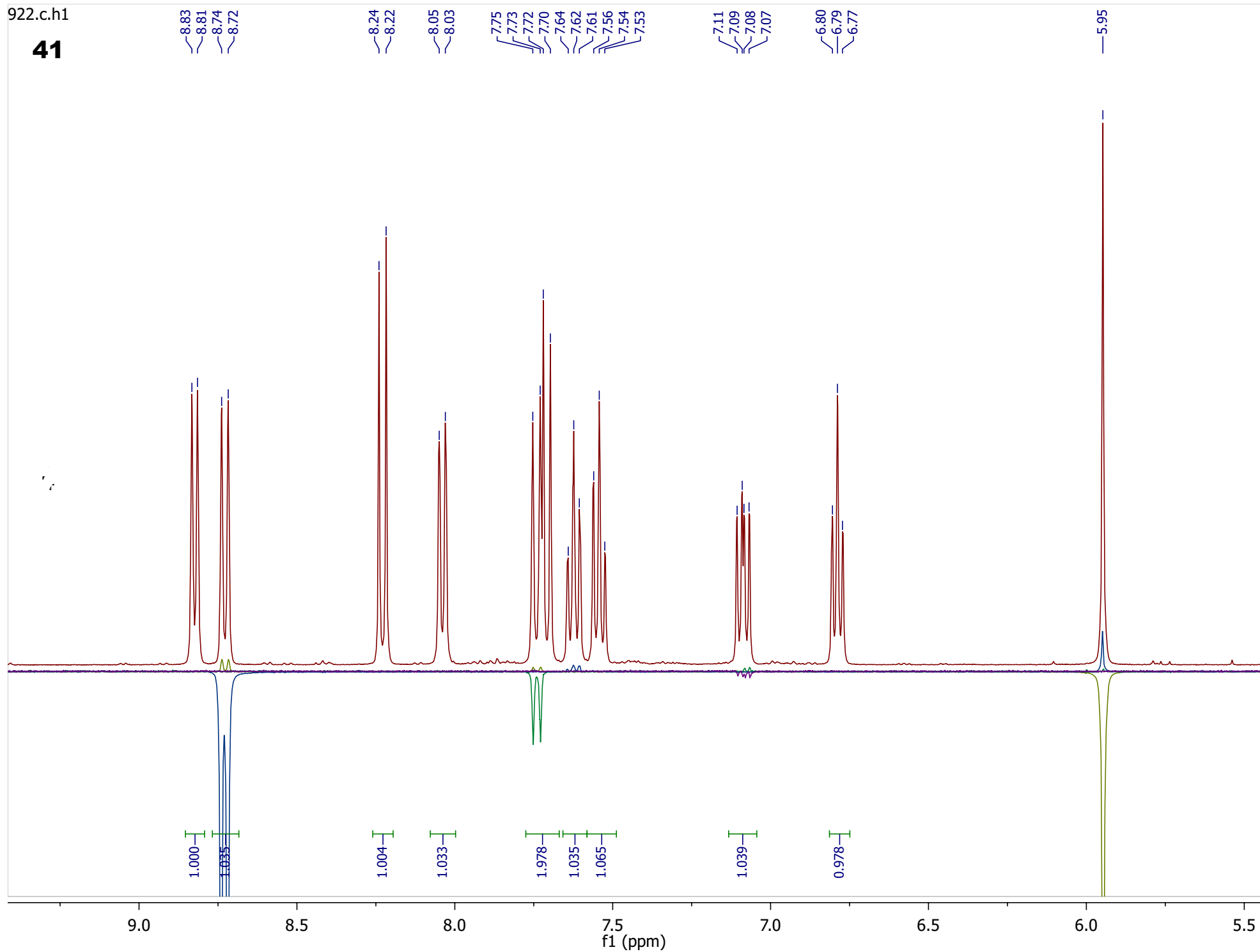
922.c.h1

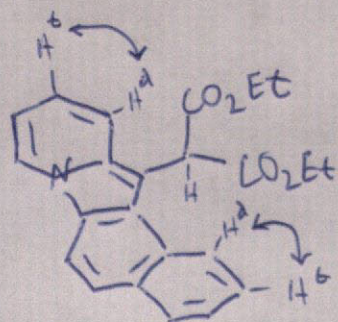
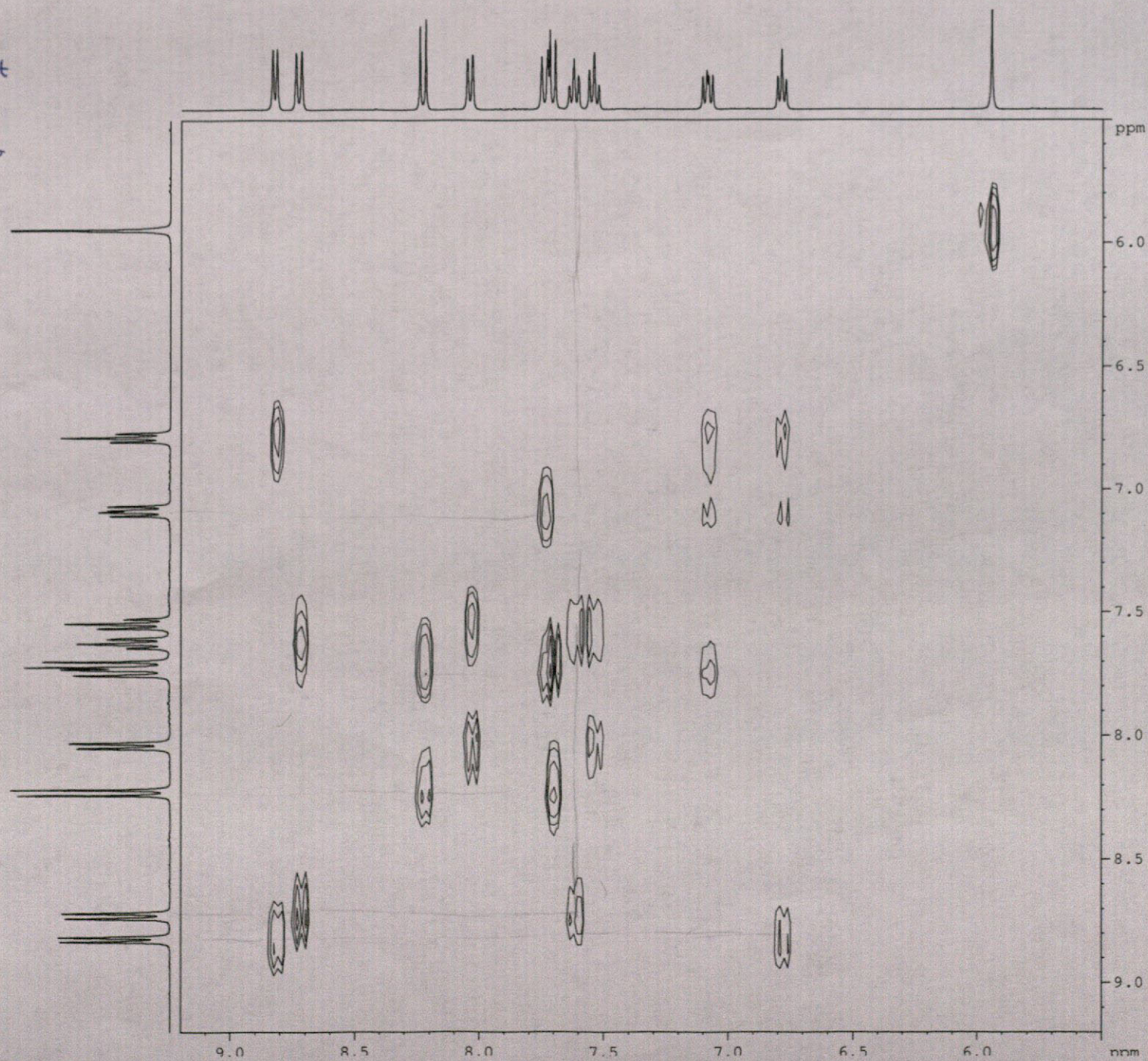
41

922.e.h1

41

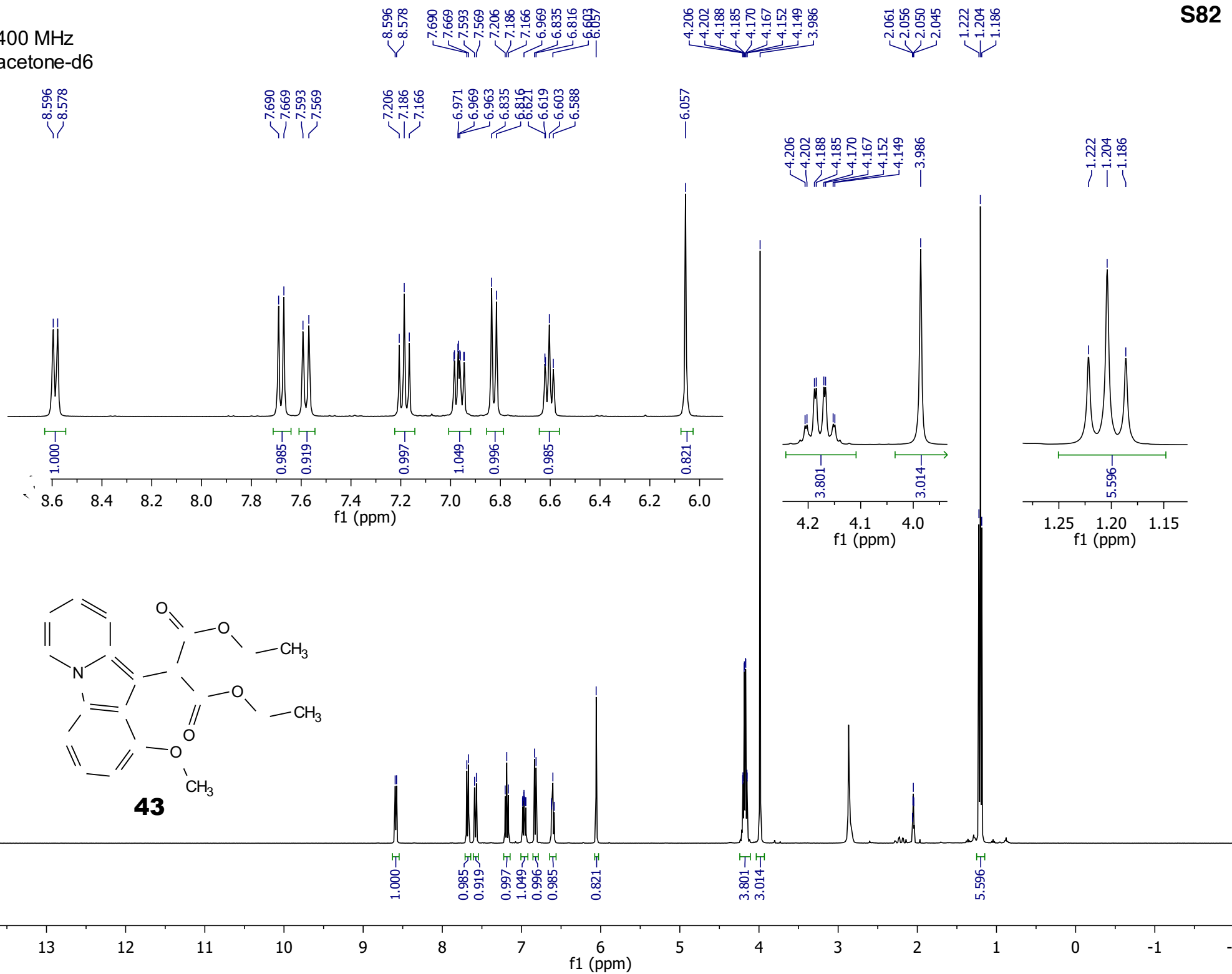
922.c.h1

41

**41**

400 MHz
acetone-d6

S82



100 MHz
acetone-d6

206.282
206.275

169.782

155.091

134.550
130.991
125.585
122.727
121.542
120.050
119.749

109.432

104.740

103.553

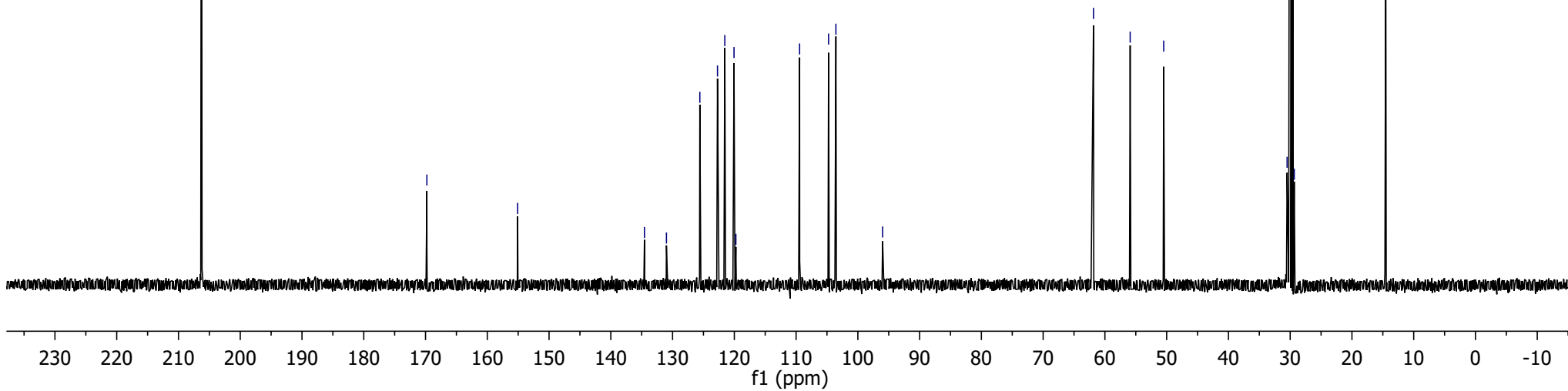
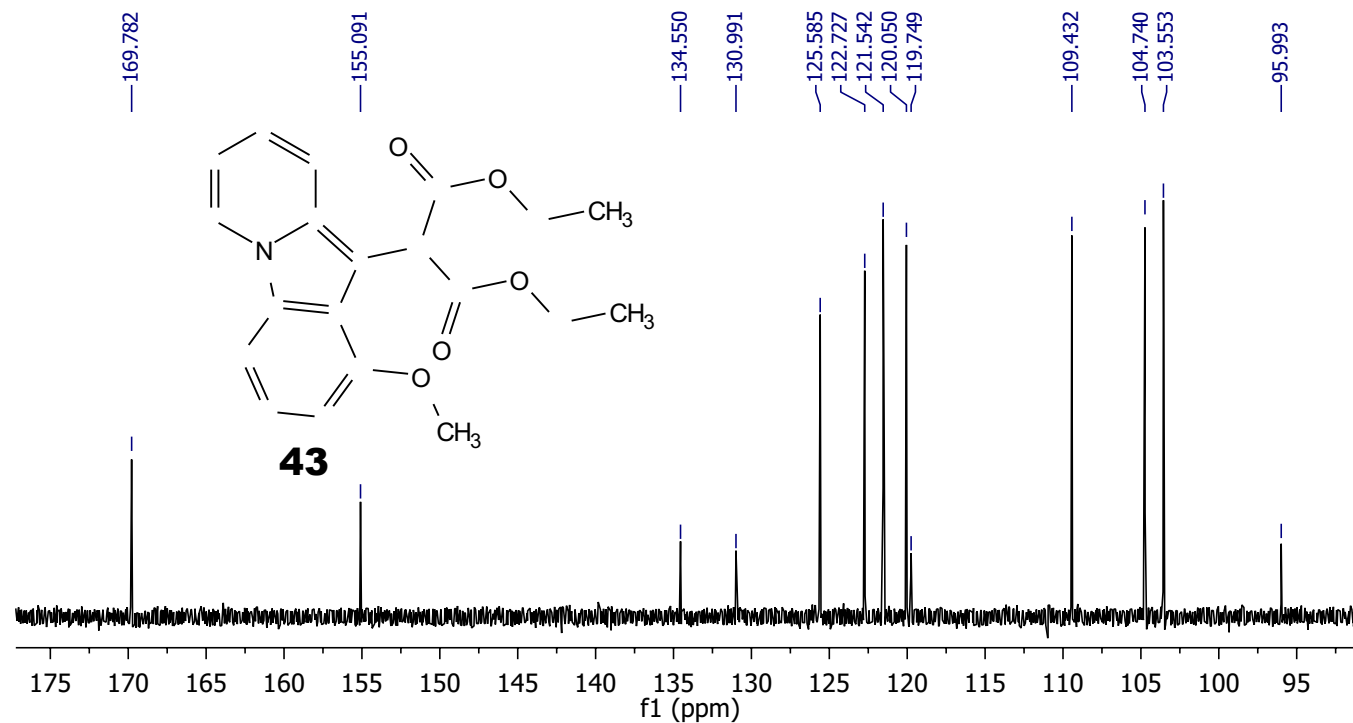
95.993

61.847
55.909
50.477

30.498
30.306
30.113
29.920
29.728
29.535
29.342

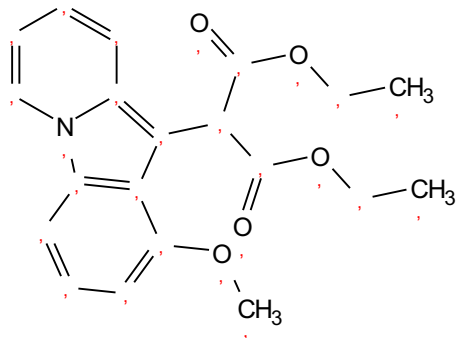
14.526

S83

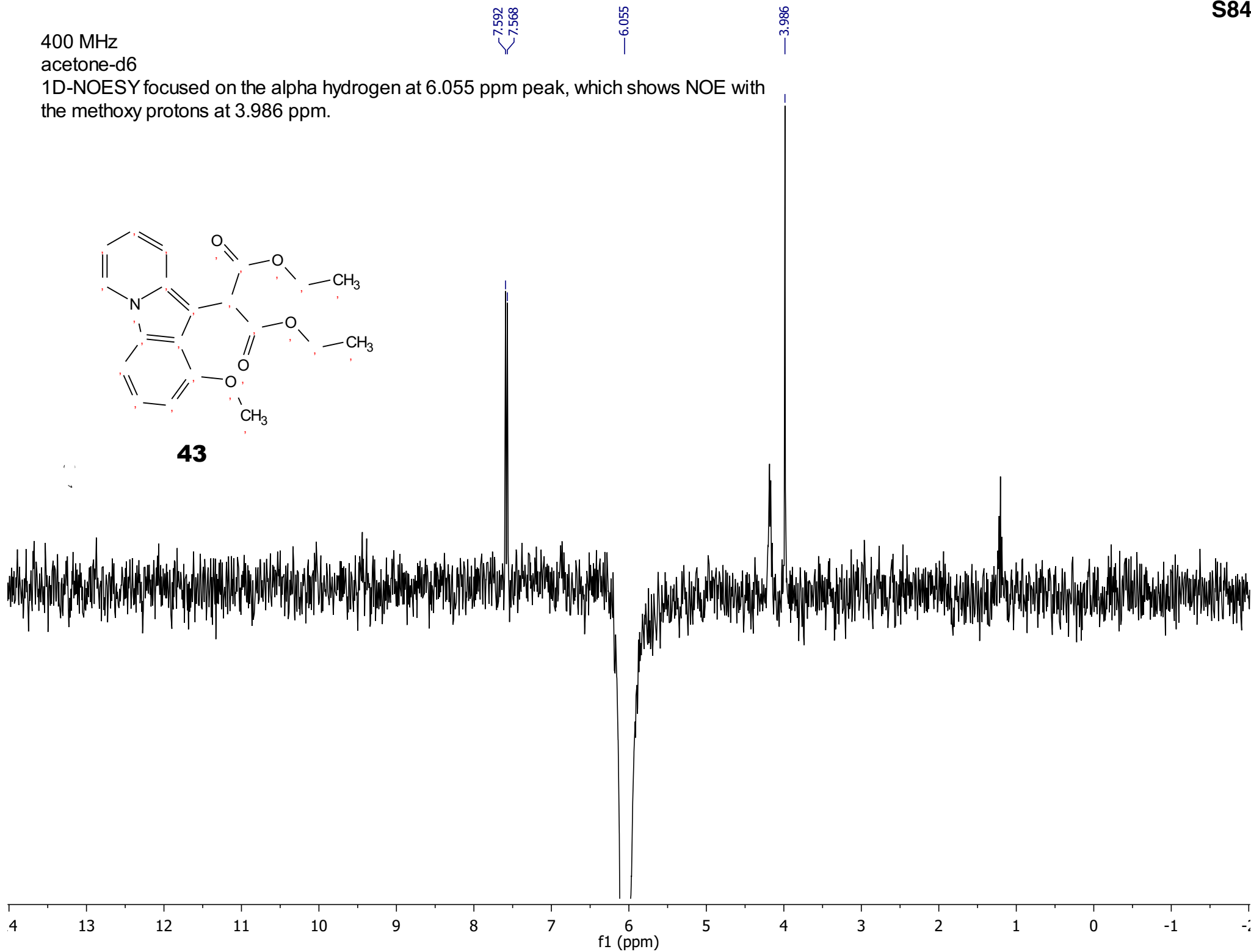


400 MHz
acetone-d₆

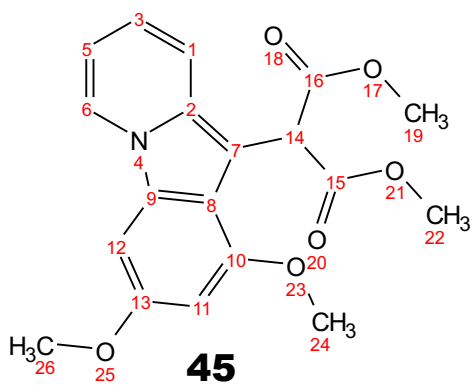
1D-NOESY focused on the alpha hydrogen at 6.055 ppm, which shows NOE with the methoxy protons at 3.986 ppm.



43



400 MHz
CDCl₃



8.106
8.088
7.516
7.493
7.270
6.832
6.817
6.476
6.454
6.004
3.961
3.934
3.913
3.752

8.106
8.088

7.516
7.493

7.270

6.832
6.817
6.809
6.793

6.494
6.476
6.454

6.004

1.090

1.028

2.023

2.023

0.938

8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0
f1 (ppm)

1.090

1.028

2.023

2.023

0.938

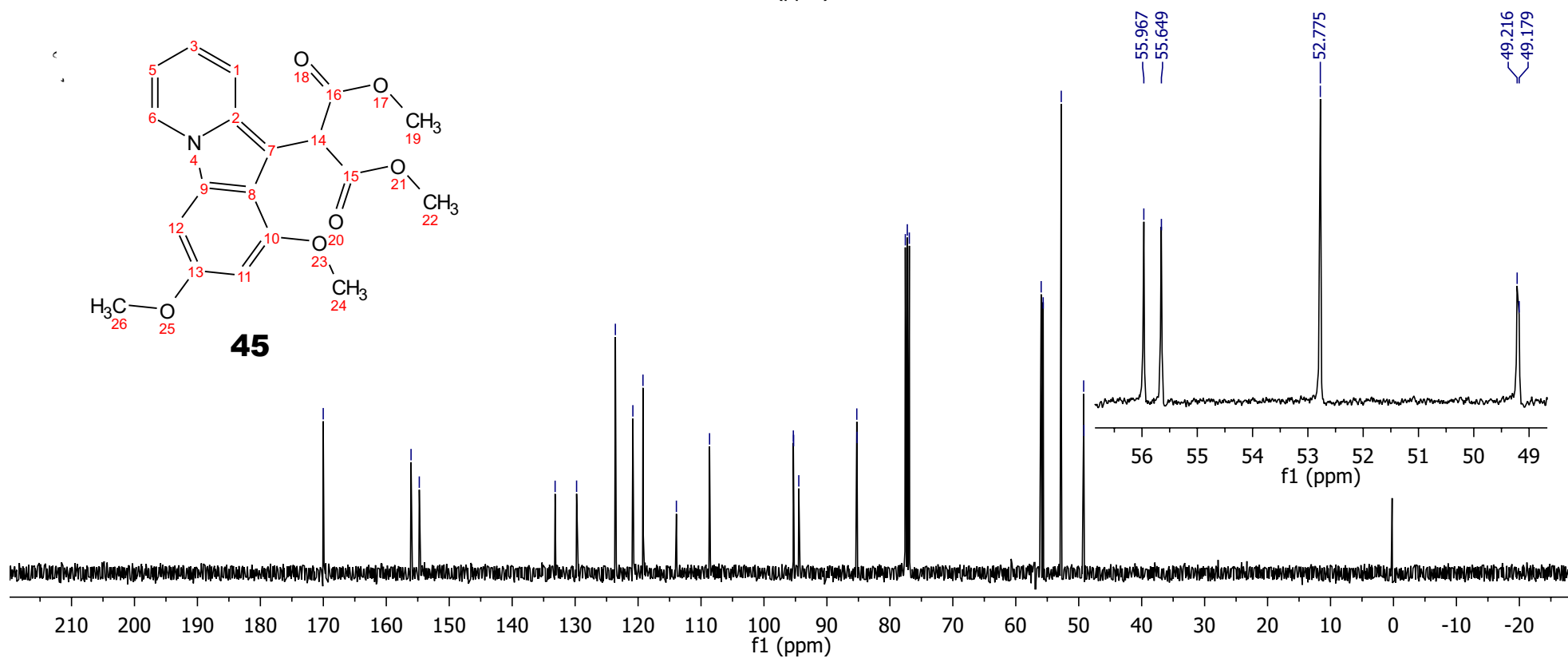
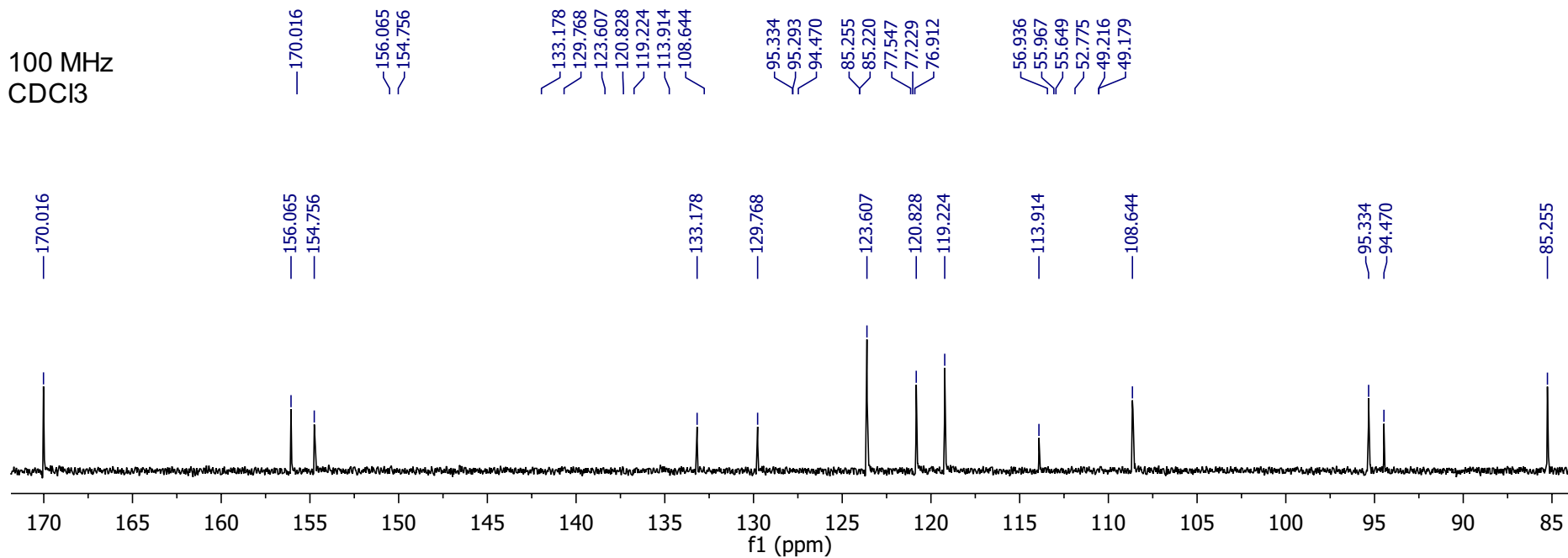
2.974

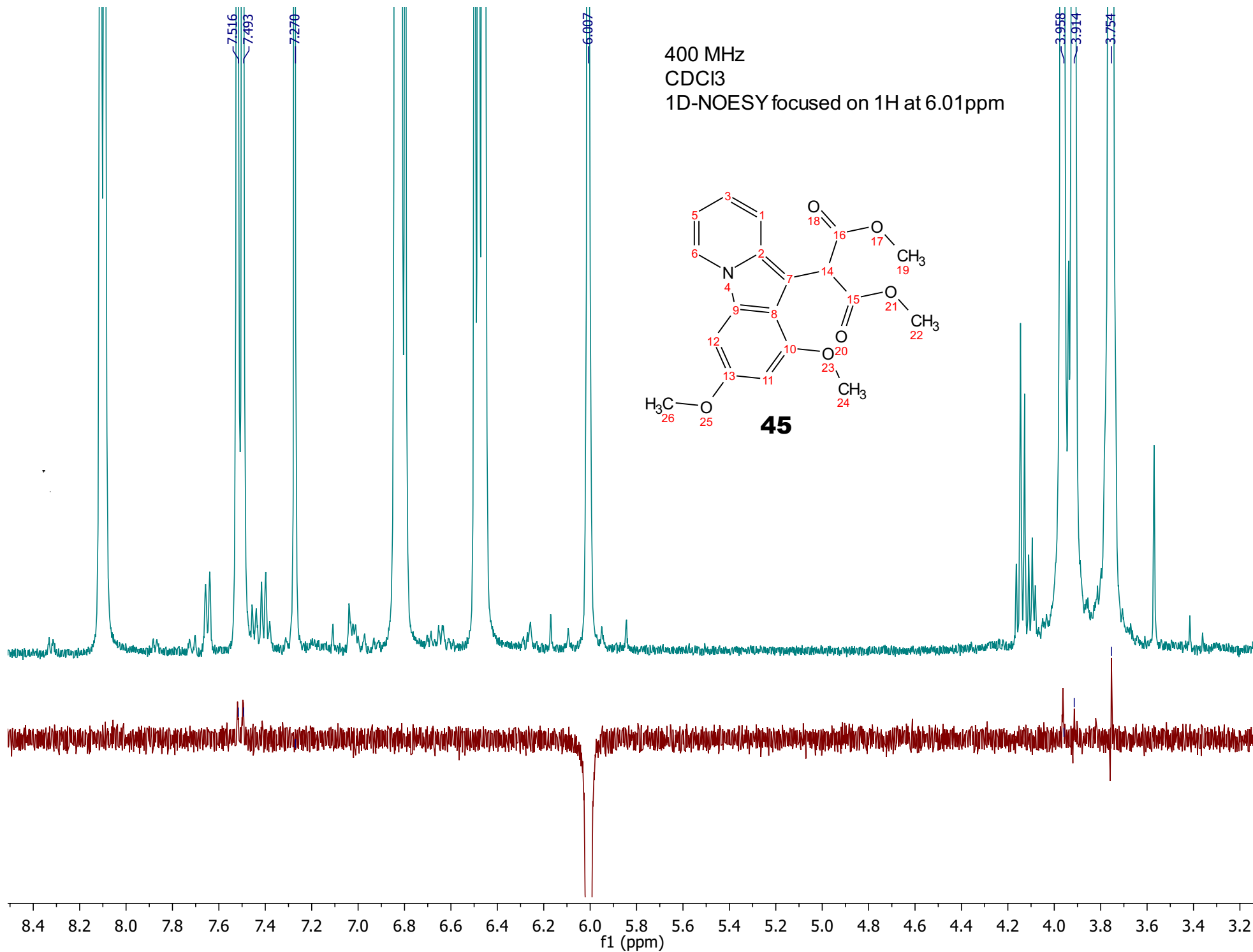
3.000

5.748

4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)

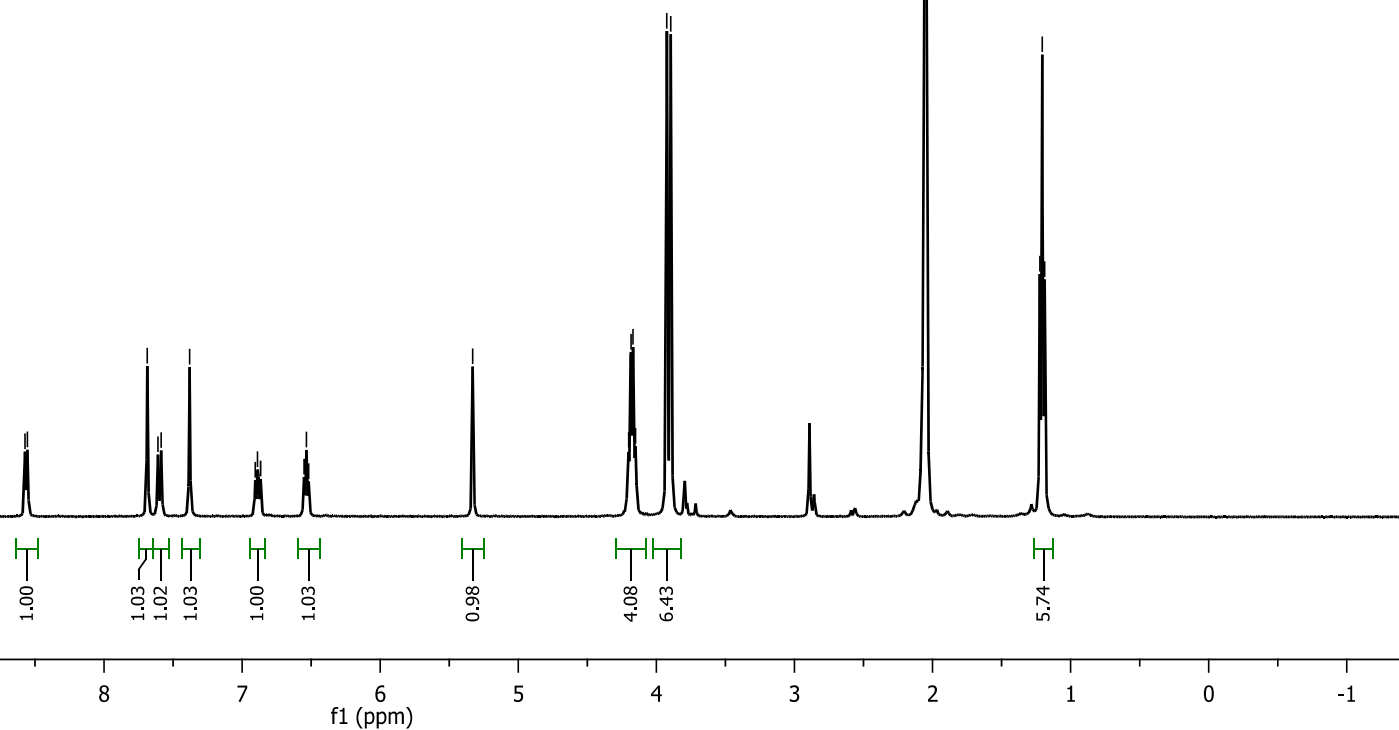
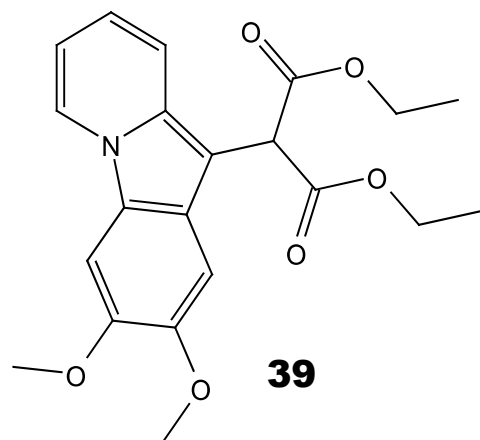
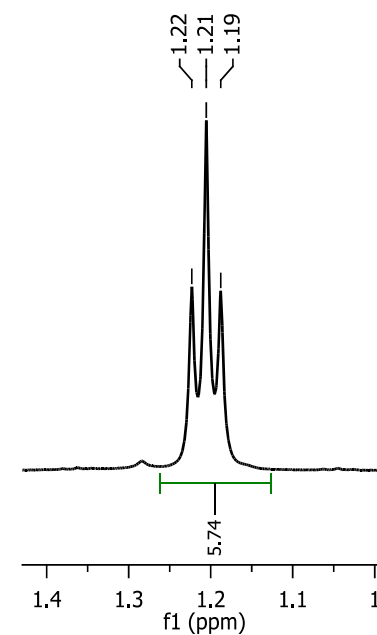
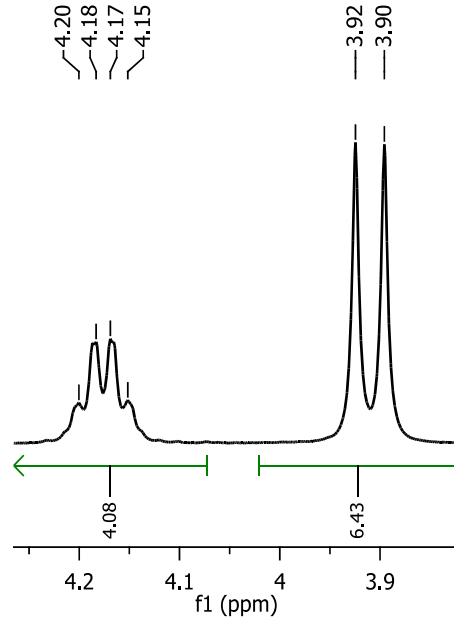
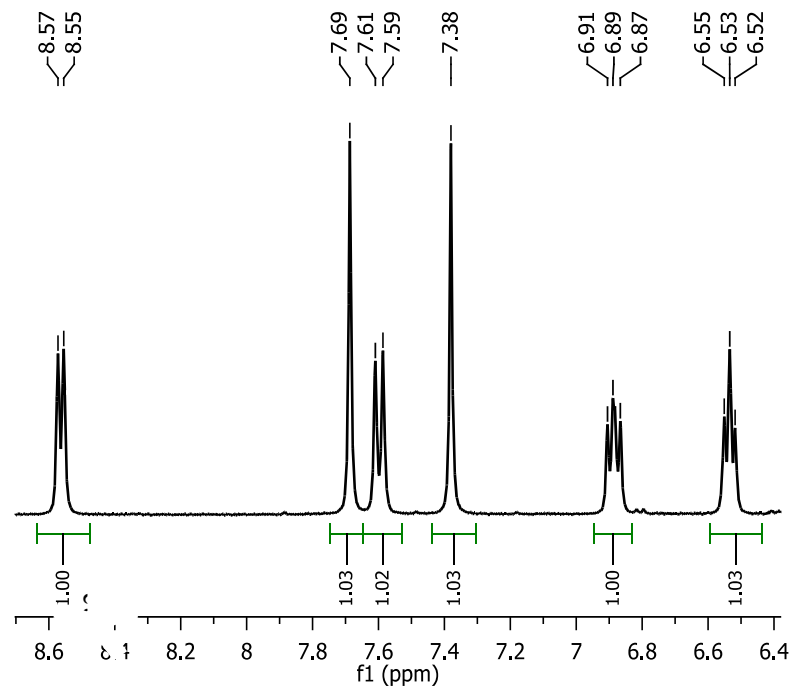
100 MHz
CDCl₃





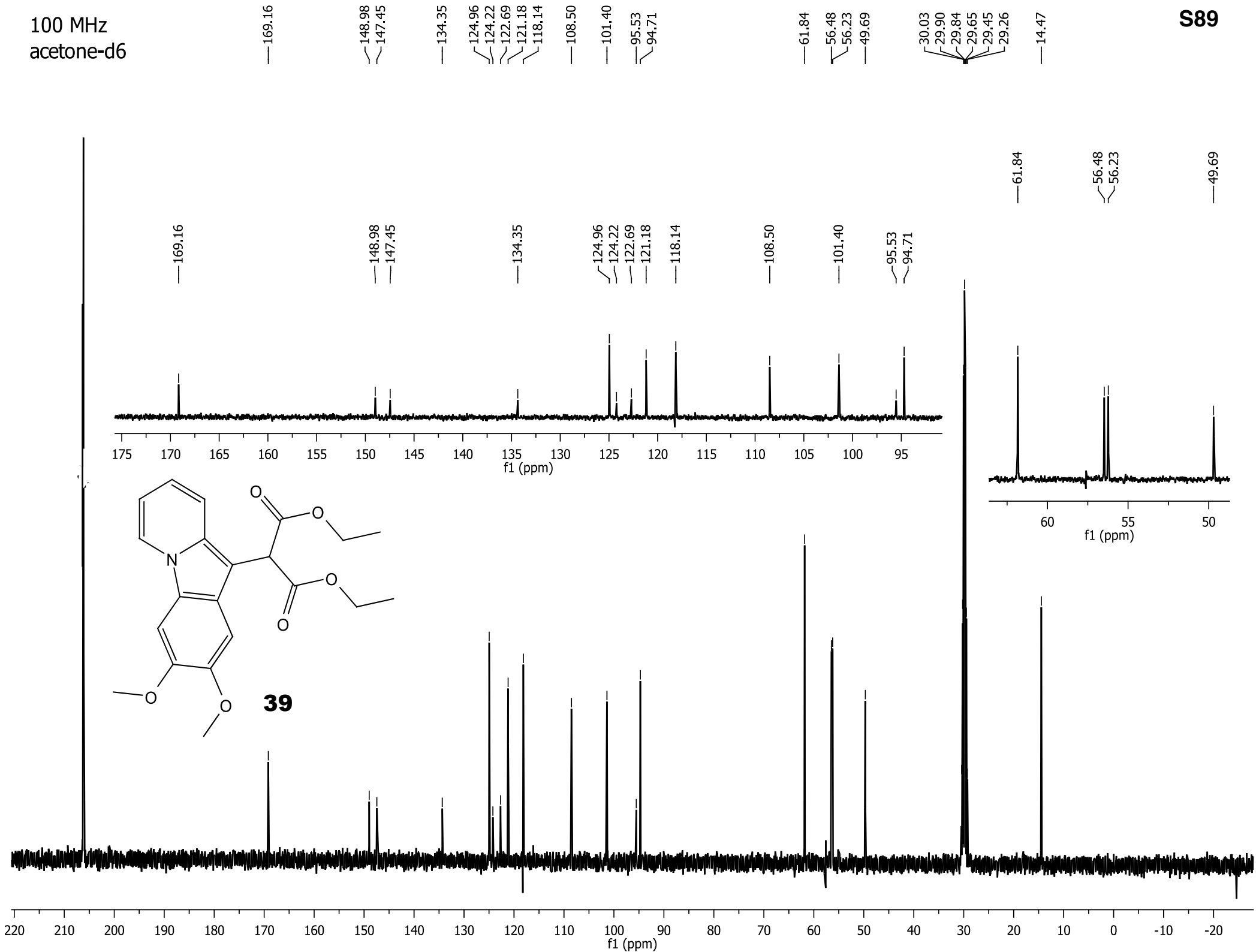
400 MHz
acetone-d₆

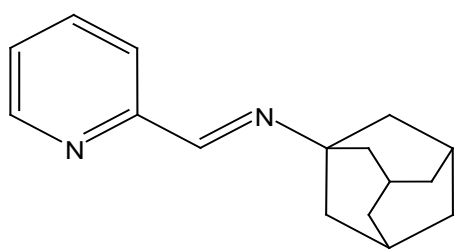
S88



100 MHz
acetone-d6

S89

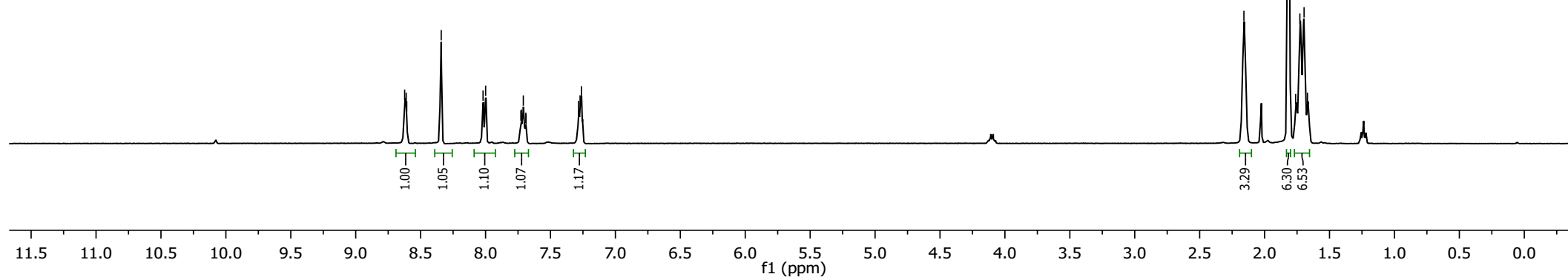
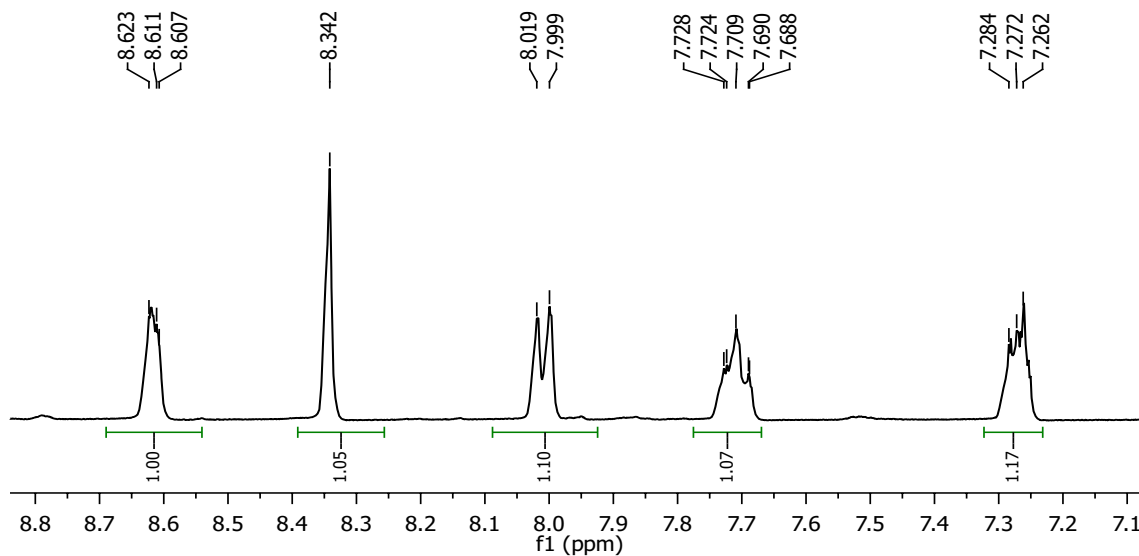


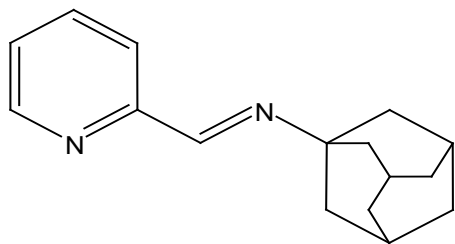


53

8.623
8.611
8.607
8.342
8.019
7.999
7.724
7.709
7.284
7.272
7.262

2.159
1.818
1.761
1.728
1.696
1.670





53

156.359

155.857

149.442

136.668

124.522

121.099

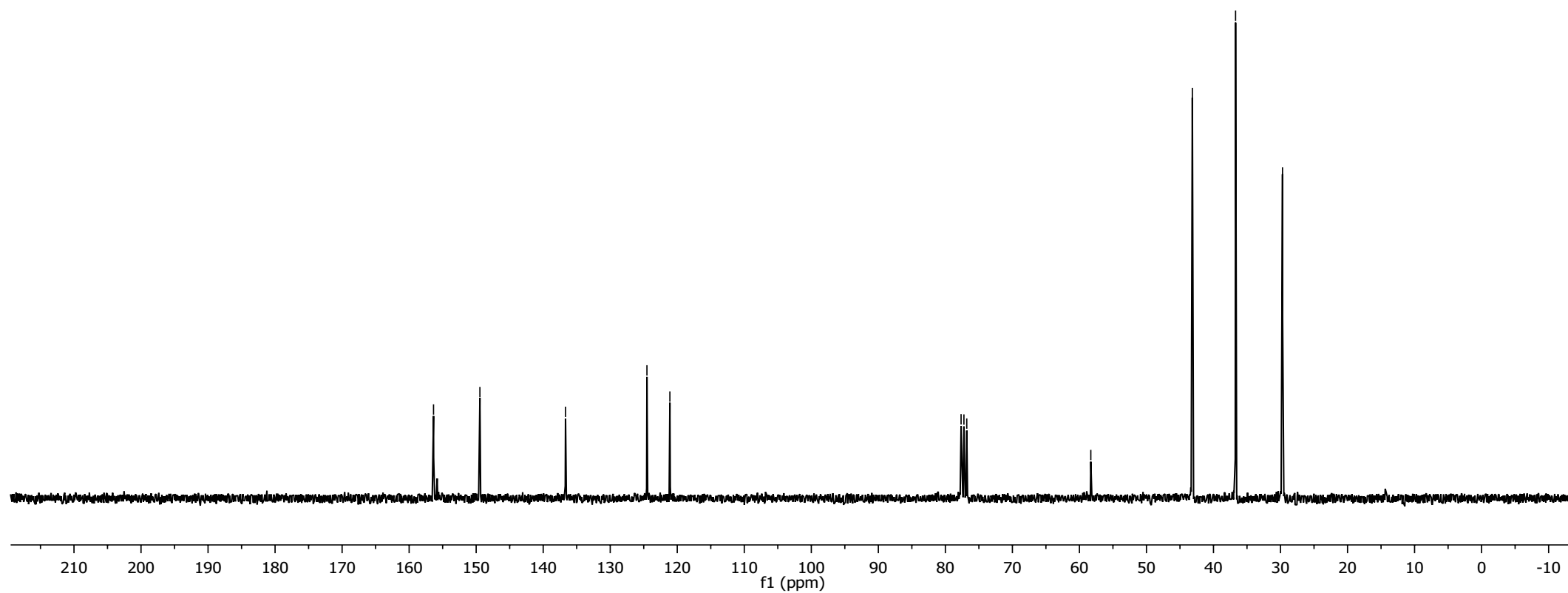
77.655 CDCl₃
77.231 CDCl₃
76.807 CDCl₃

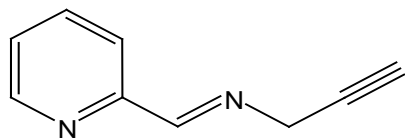
58.300

43.152

36.704

29.700



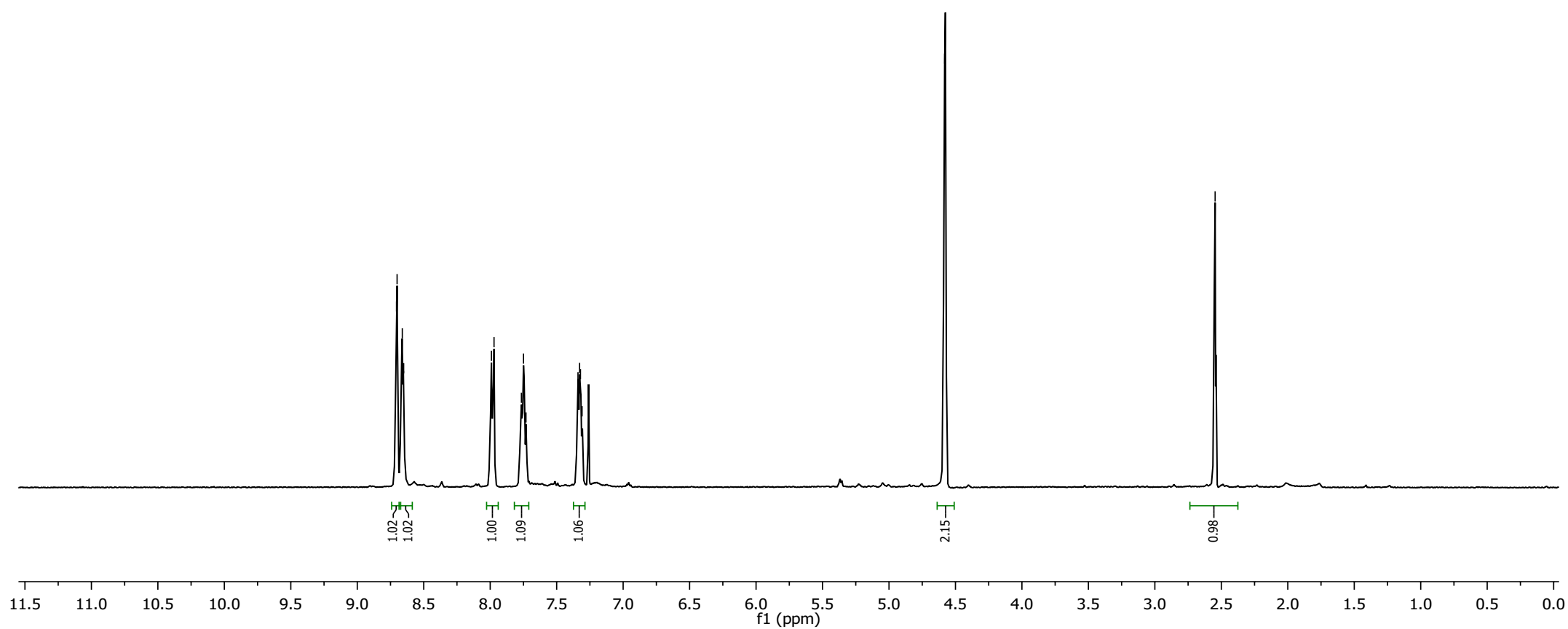


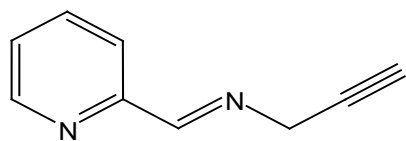
57

8.705
8.700
8.661
8.651
7.991
7.971
7.765
7.749
7.731
7.340
7.328
7.321
7.309

4.581

2.547



**57**

— 163.360

— 154.346

— 149.714

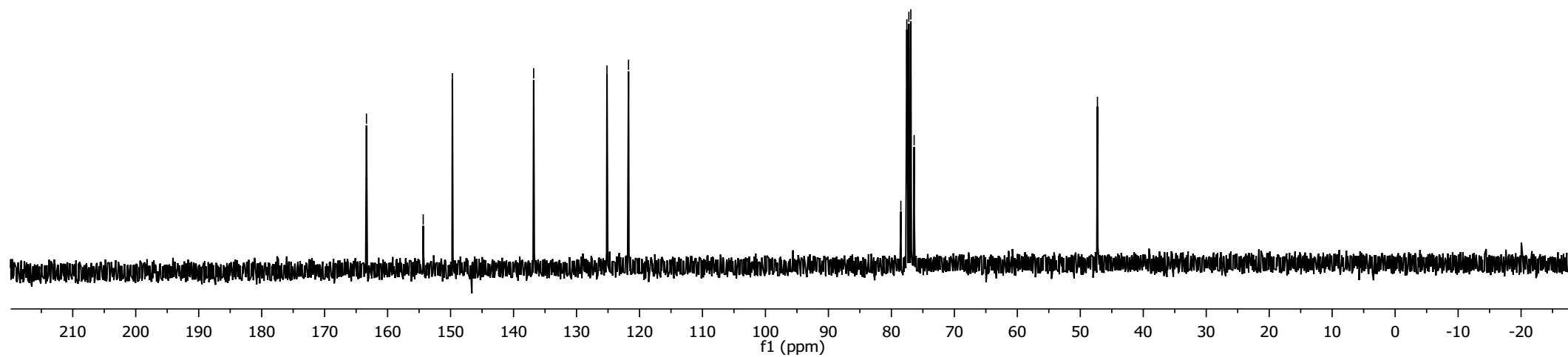
— 136.803

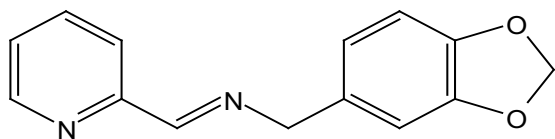
— 125.166

— 121.743

78.496
77.548
77.225
76.911
76.384

— 47.274

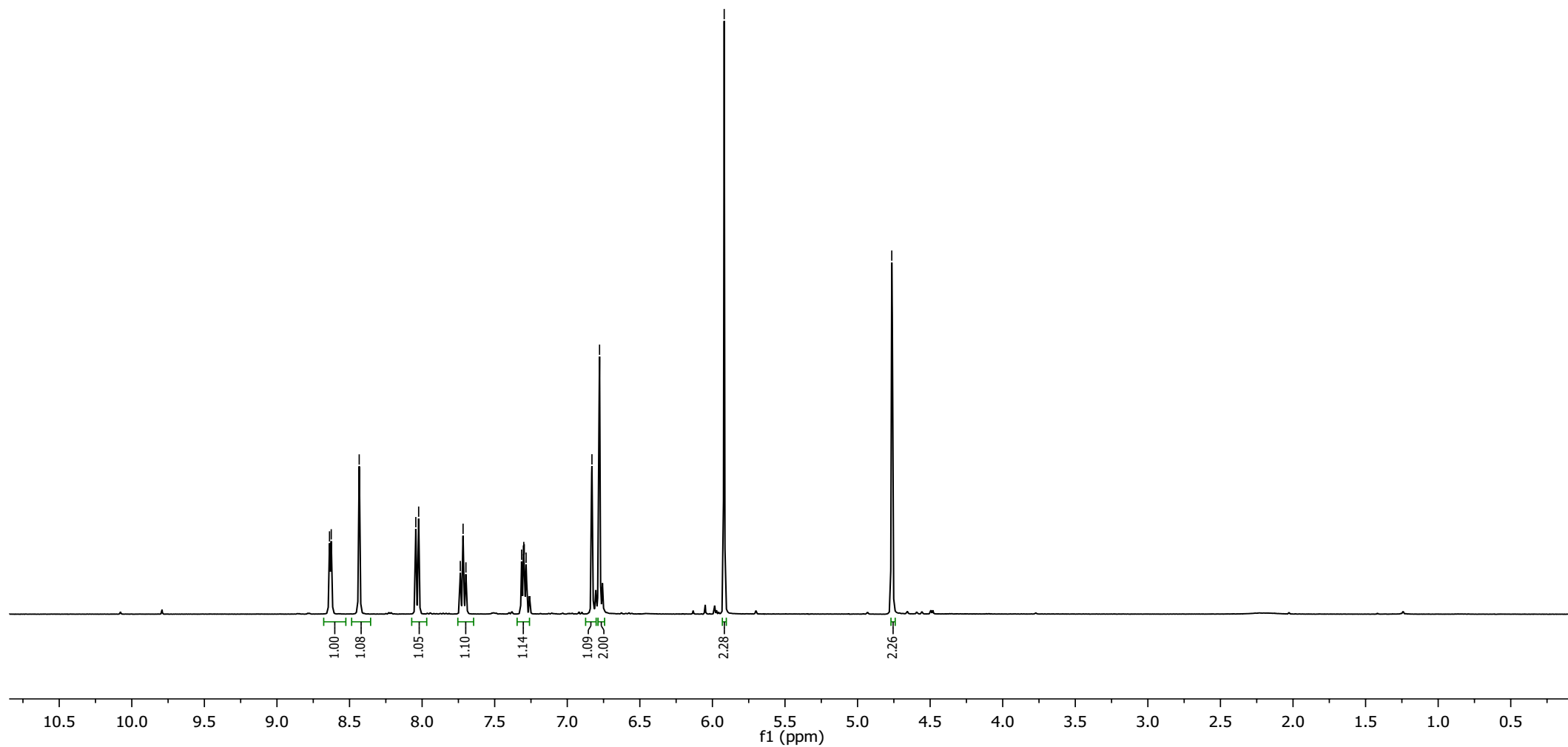


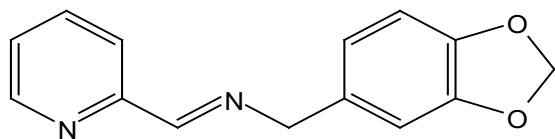
**58**

8.64
8.63
8.43
8.04
8.02
7.74
7.72
7.31
7.30
7.30
7.30
6.83
6.78

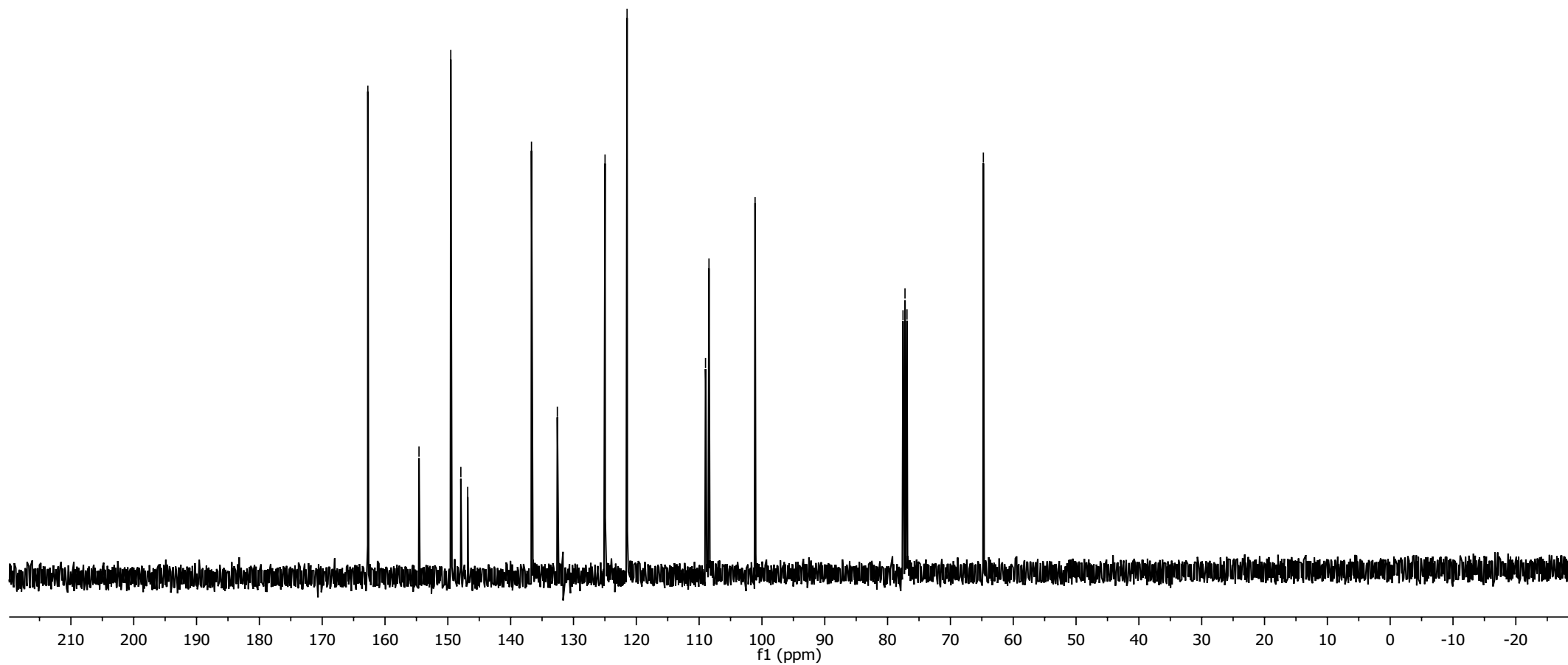
5.92

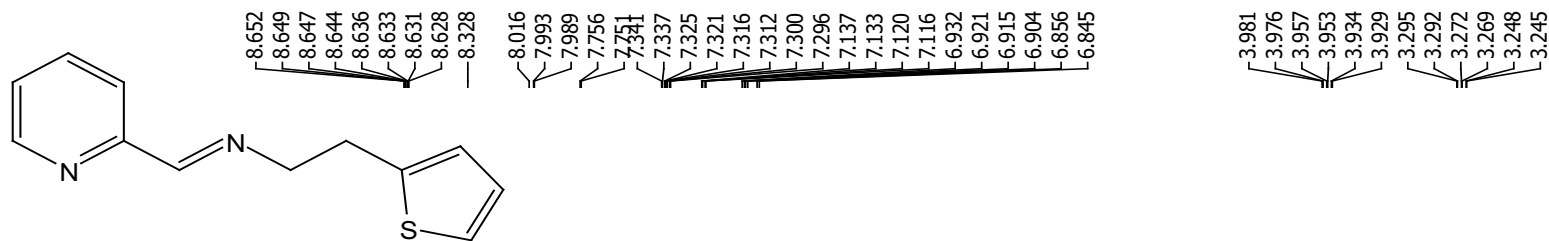
4.76



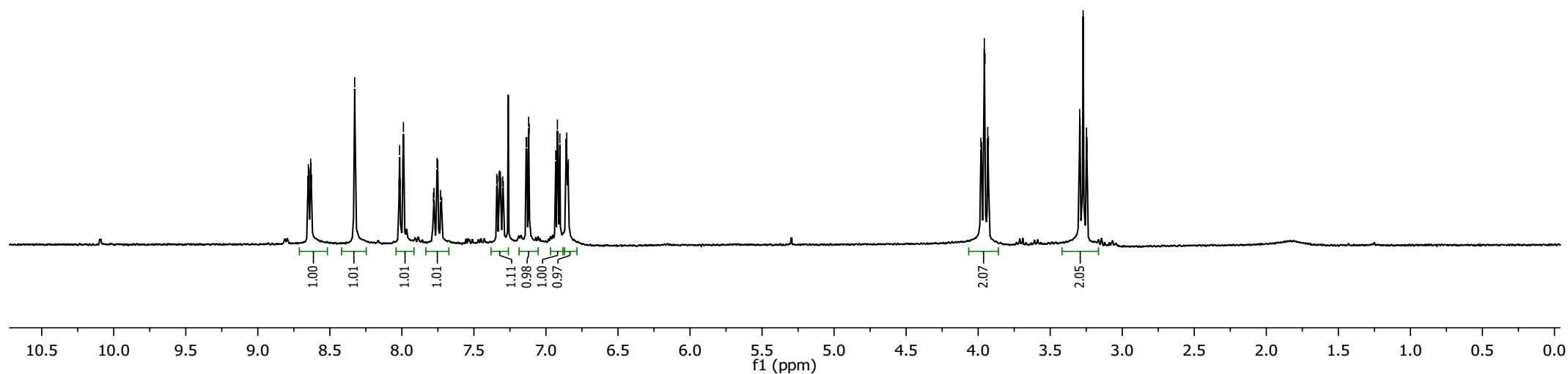
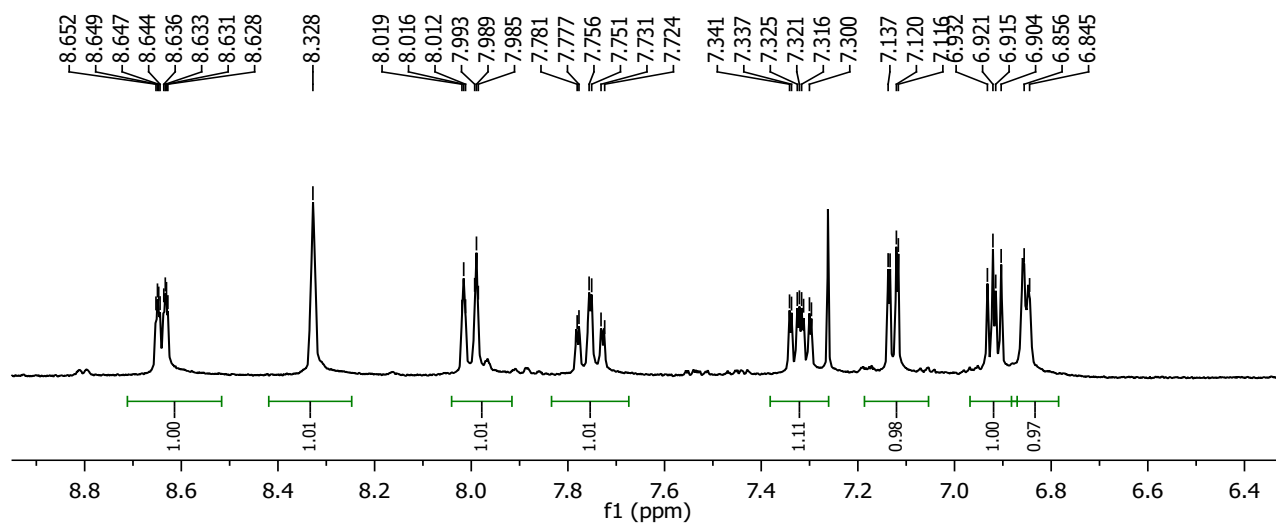
**58**

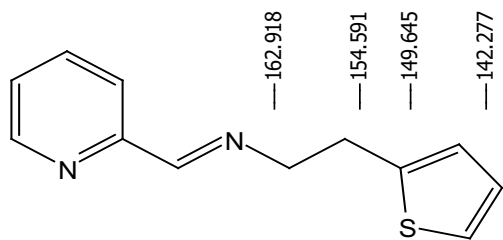
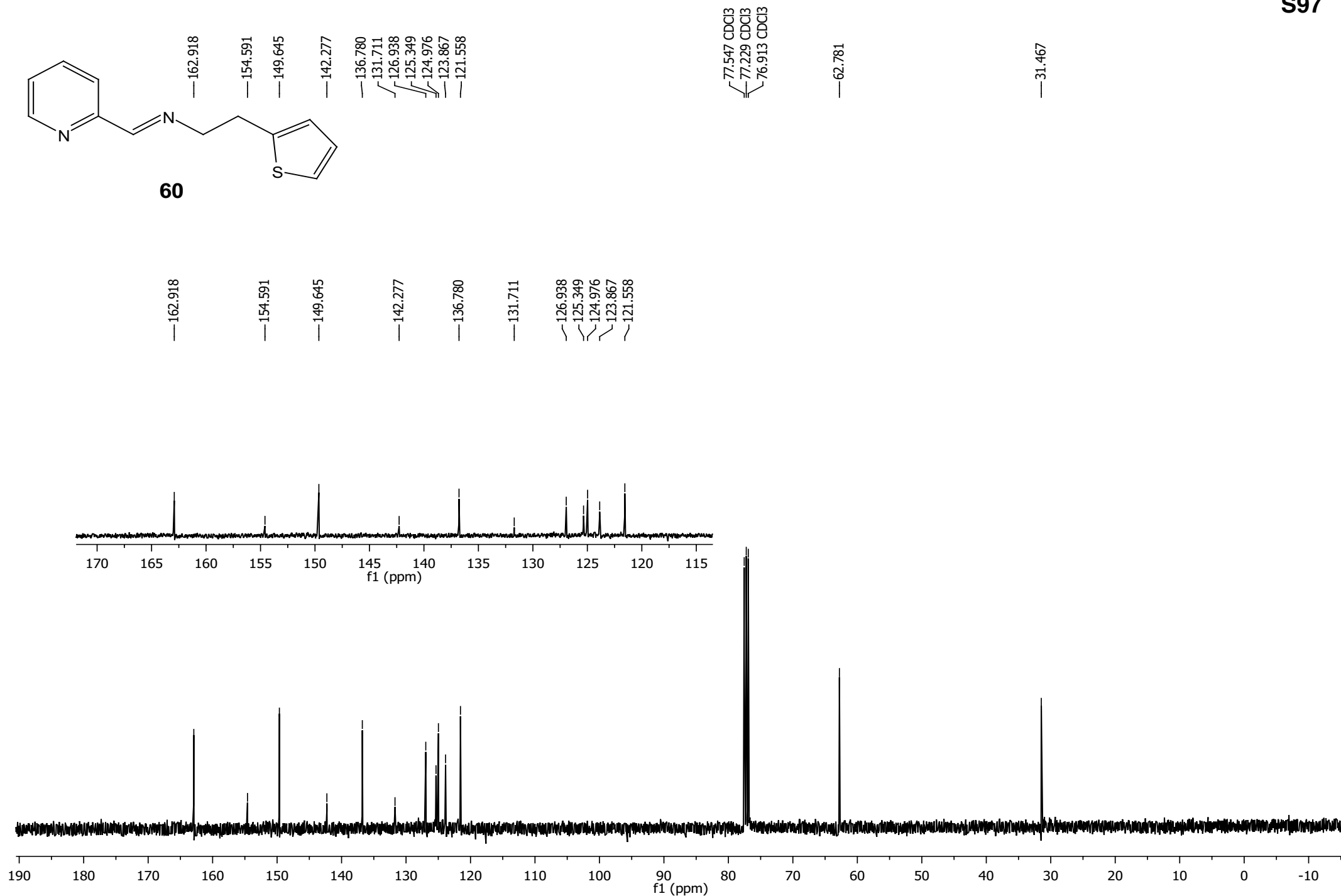
162.73
154.59
149.53
147.93
146.83
136.69
132.57
124.98
121.51
121.47
108.98
108.43
101.09
77.55 CDCl₃
77.23 CDCl₃
76.91 CDCl₃
64.76

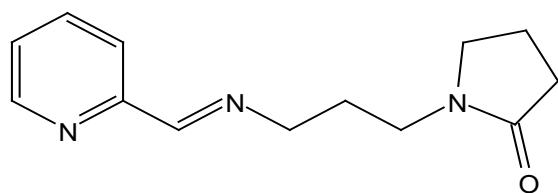




60



**60**

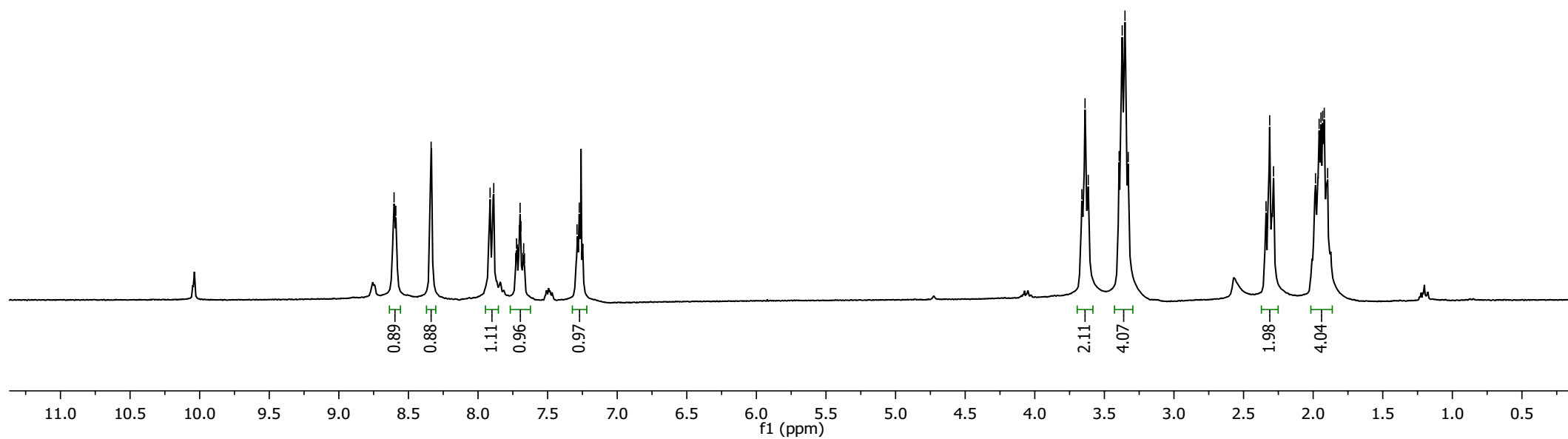


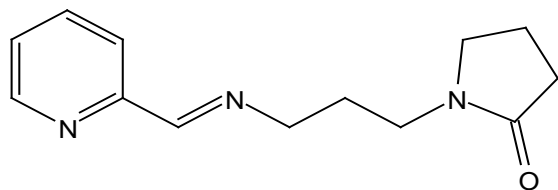
62

8.603
8.591
8.337
7.913
7.887
7.704
7.698
7.692
7.289
7.273
7.252

3.662
3.639
3.616
3.394
3.372
3.352
3.329

2.339
2.312
2.285
1.983
1.957
1.944
1.931
1.920
1.896





62

— 175.079

— 162.479

— 154.429

— 149.613

— 136.670

— 124.855

— 121.567

77.647 CDCl₃
77.221 CDCl₃
76.800 CDCl₃

— 59.021

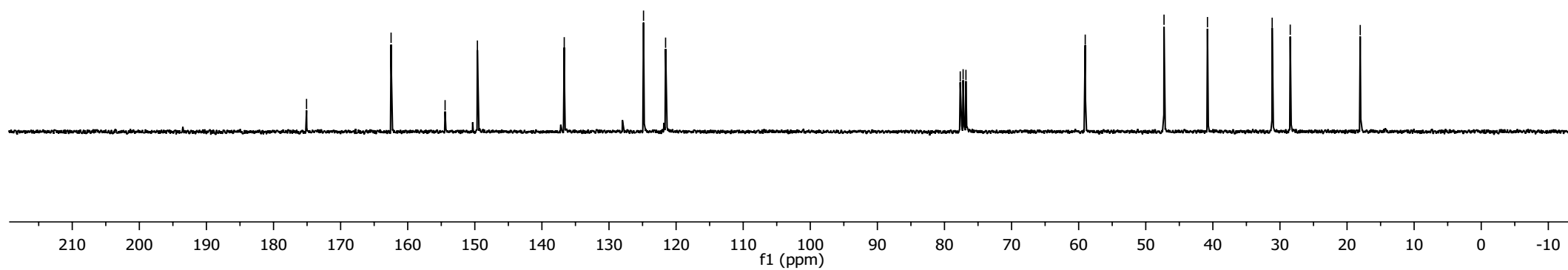
— 47.277

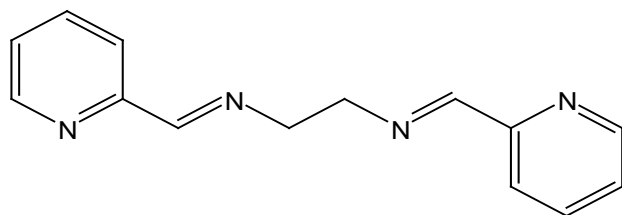
— 40.802

— 31.157

— 28.465

— 18.033

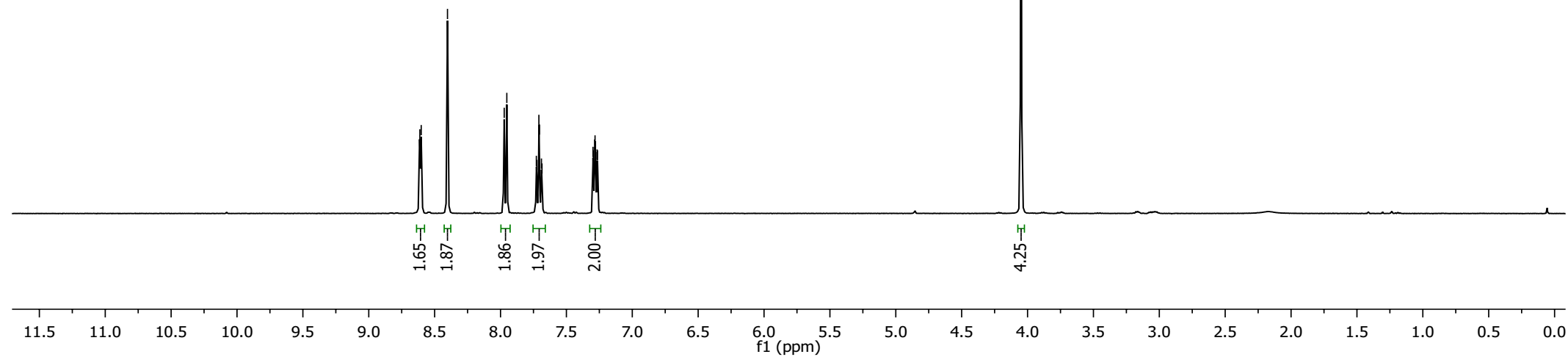
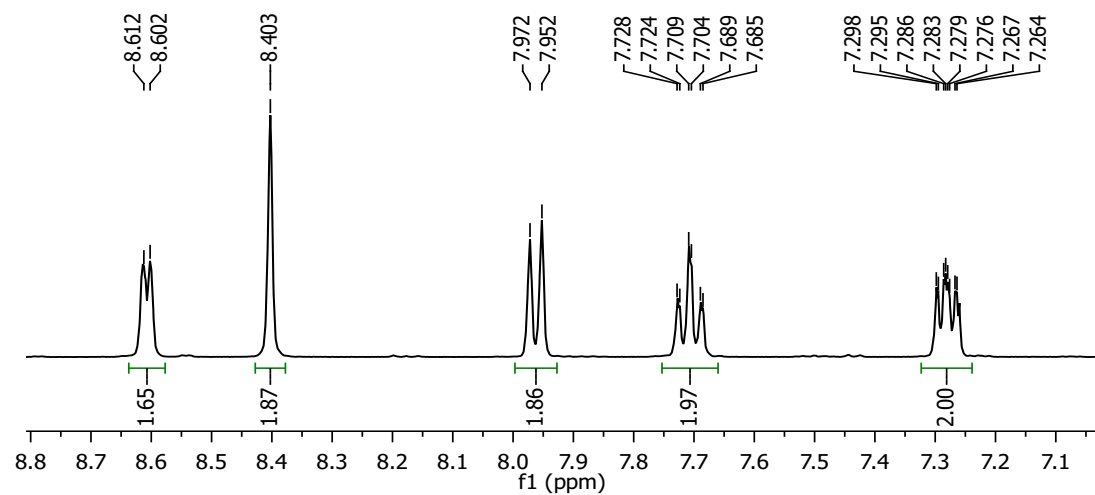


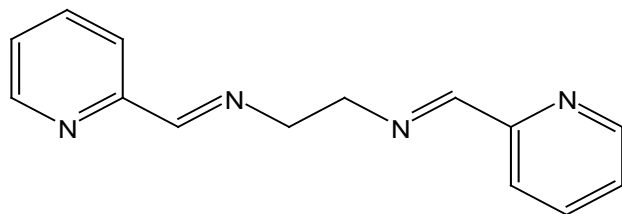


64

8.612
8.602
8.403
7.972
7.952
7.728
7.709
7.704
7.689
7.685
7.298
7.295
7.286
7.283
7.279
7.276
7.267
7.264

4.049



**64**

—163.600

—154.524

—149.564

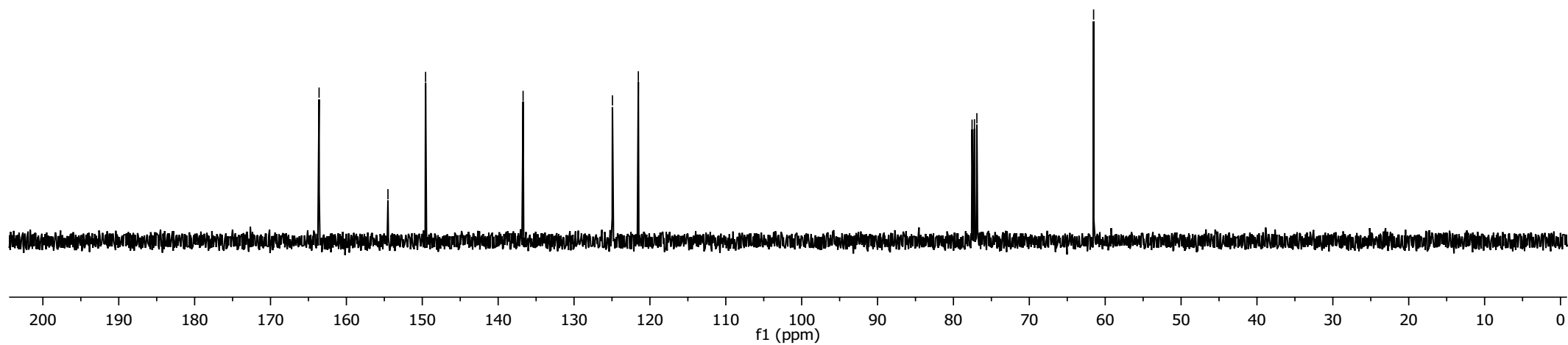
—136.714

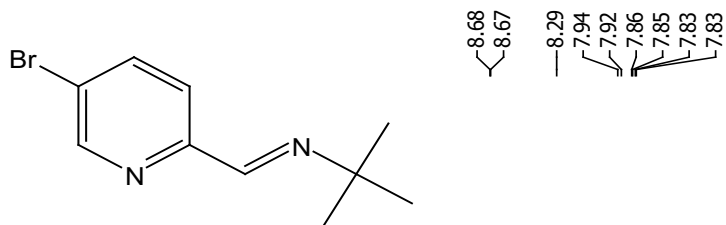
—124.938

—121.526

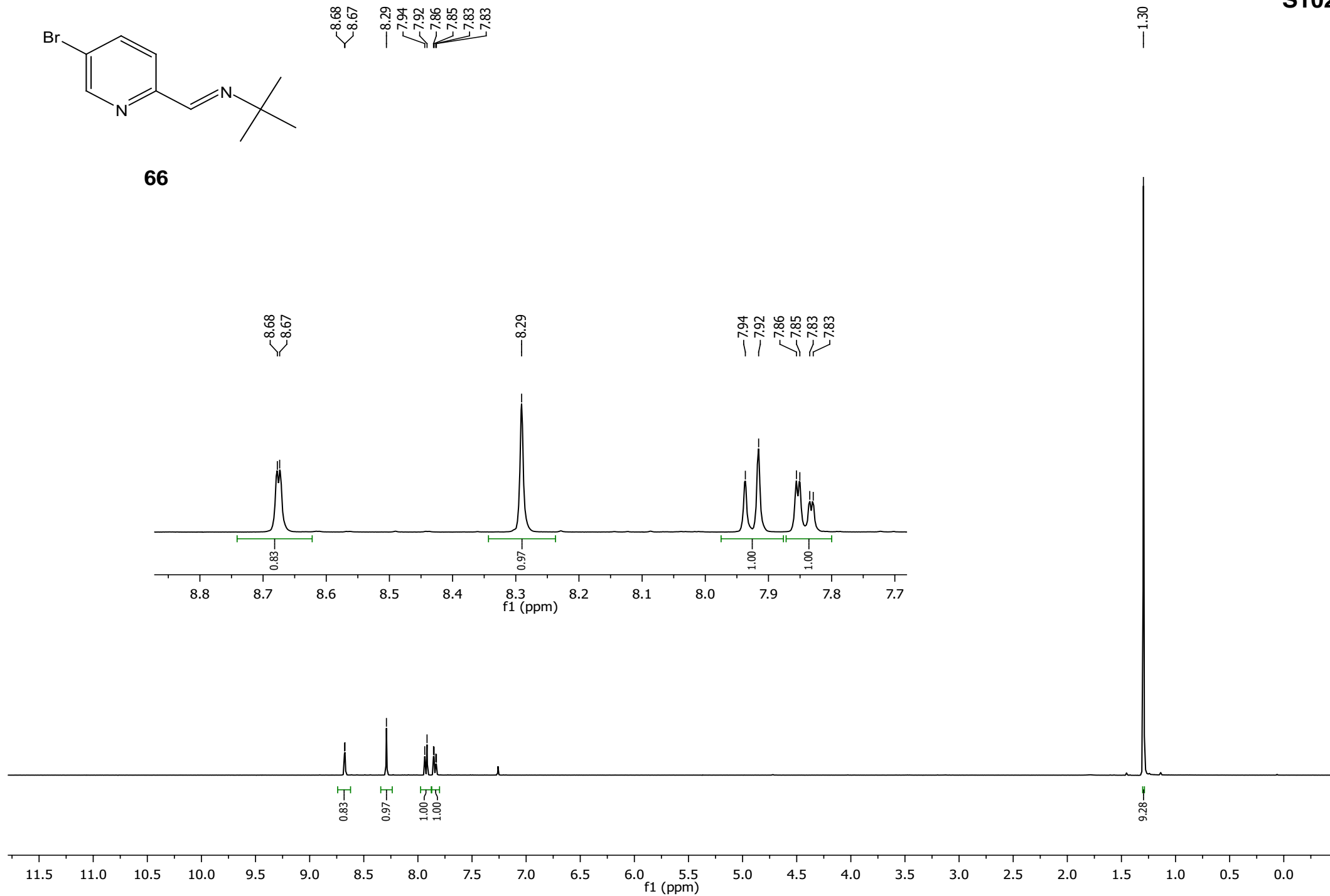
77.547 CDCl₃
77.230 CDCl₃
76.912 CDCl₃

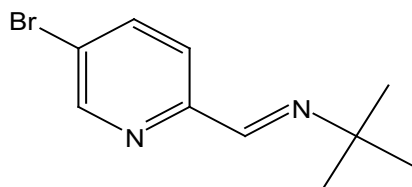
—61.528



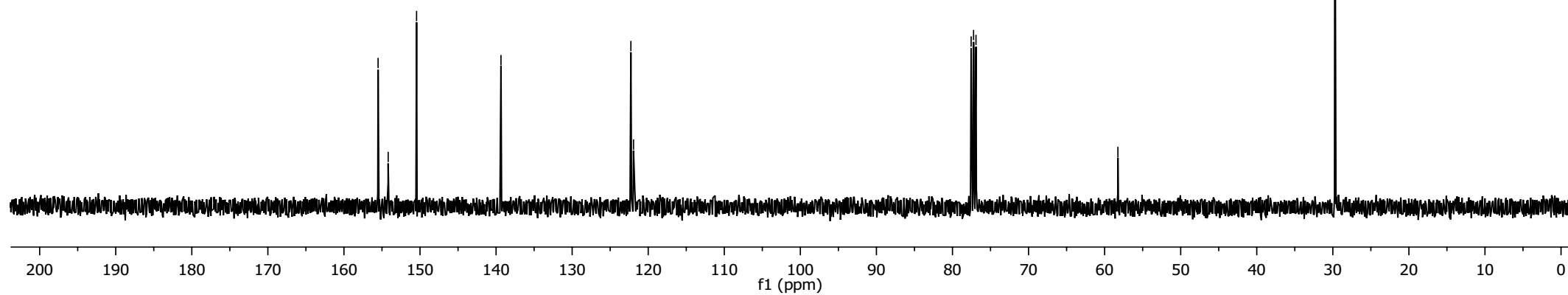
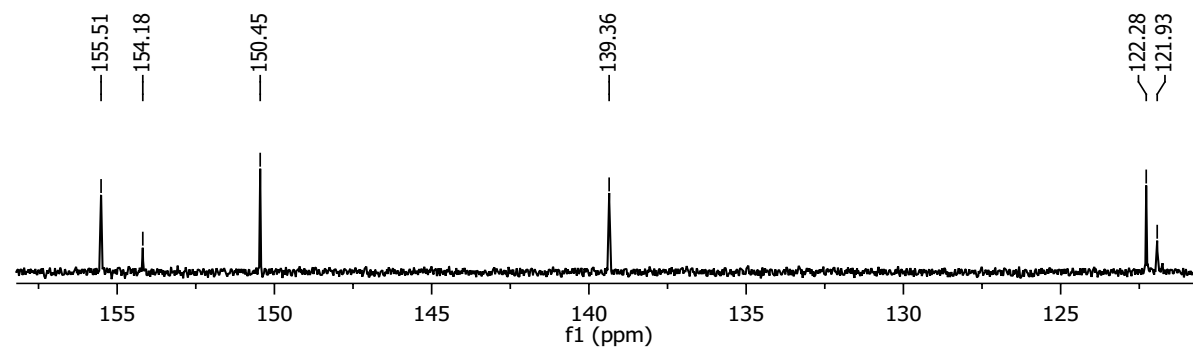


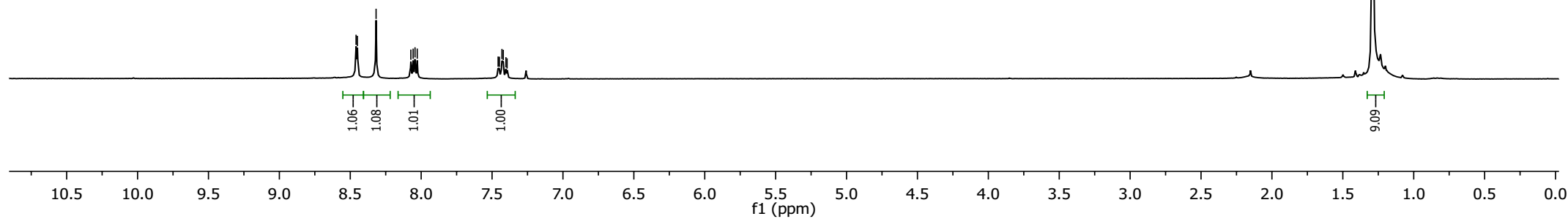
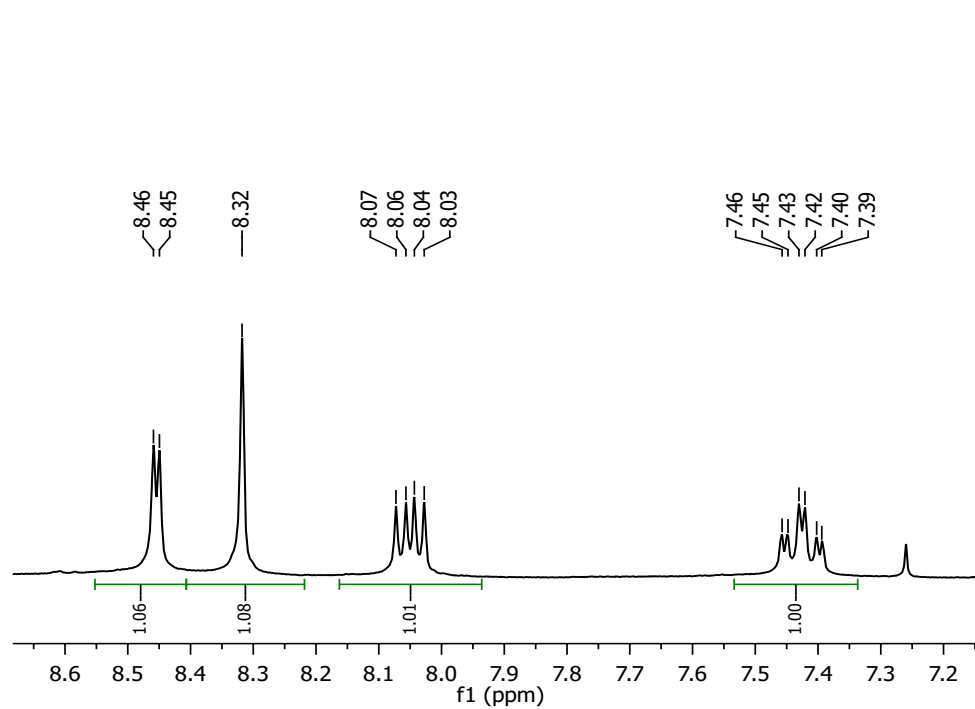
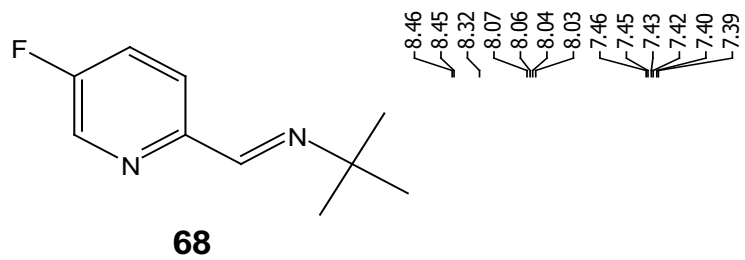
66

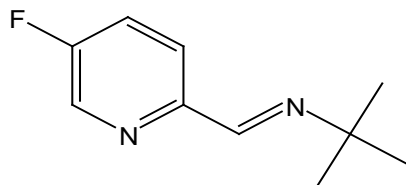




66







68

161.44
158.87
155.17
152.14
152.10

137.63
137.39

123.73
123.54
122.35
122.31

77.55 CDCl₃
77.23 CDCl₃
76.91 CDCl₃

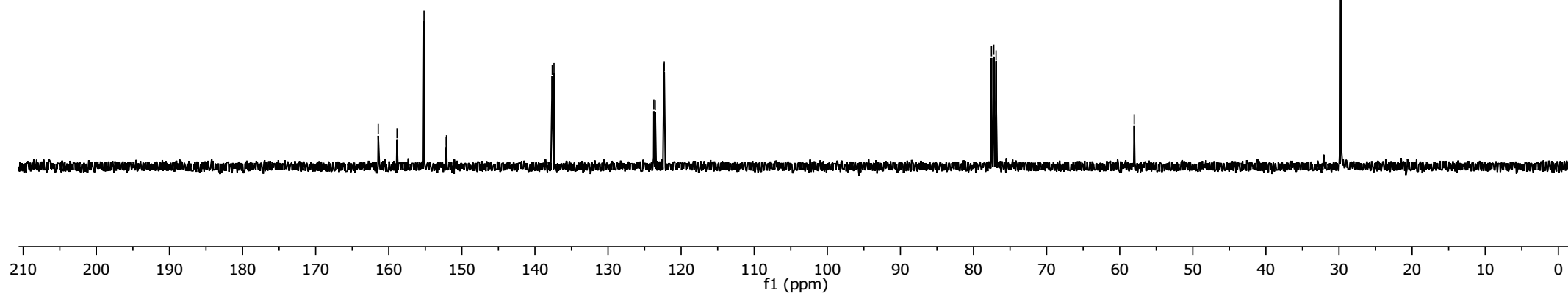
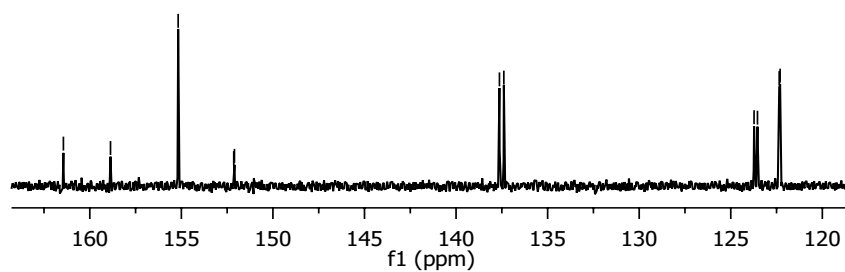
58.02

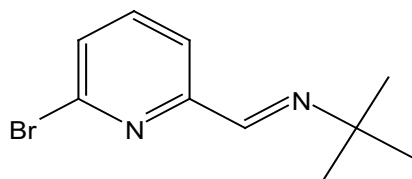
29.76

161.44
158.87
155.17
152.14
152.10

137.63
137.39

123.73
123.54
122.35
122.31

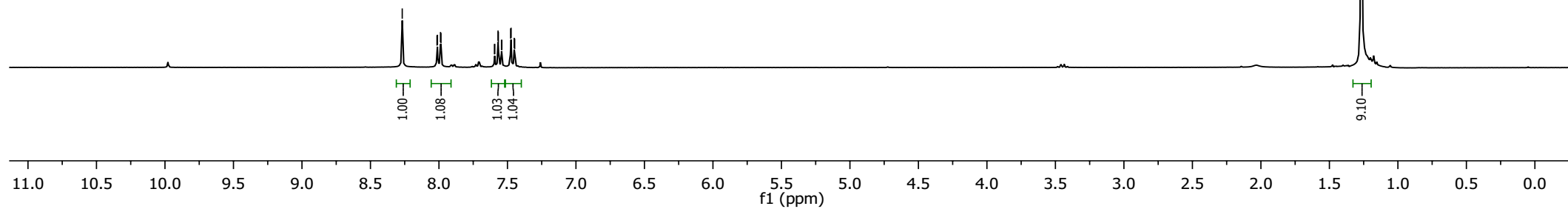
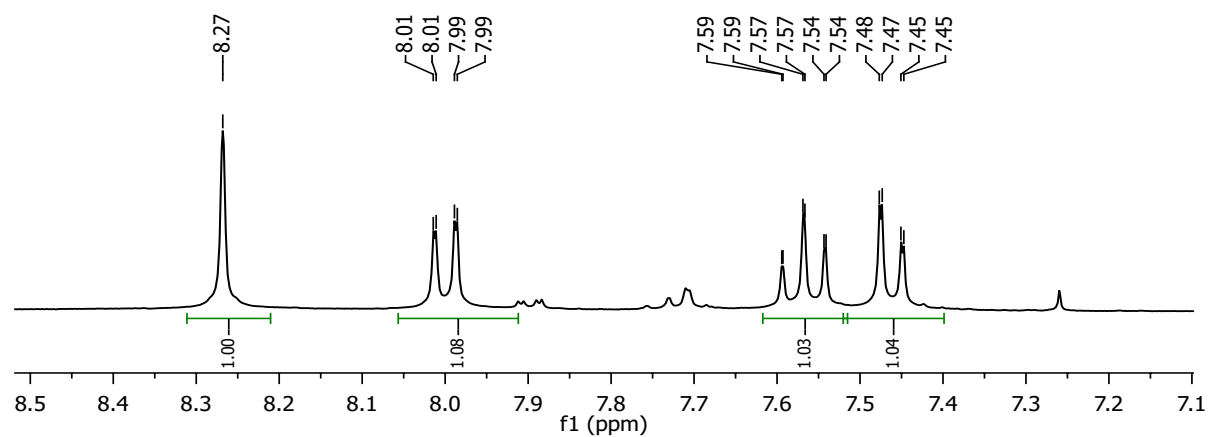


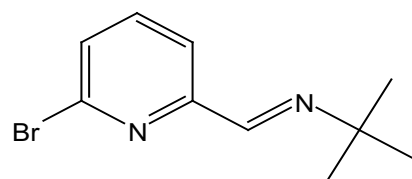


70

8.27
8.01
8.01
7.99
7.99
7.59
7.59
7.57
7.57
7.54
7.54
7.48
7.47
7.45
7.45

1.27
1.27



**70**~156.92
~155.40~141.43
~138.99

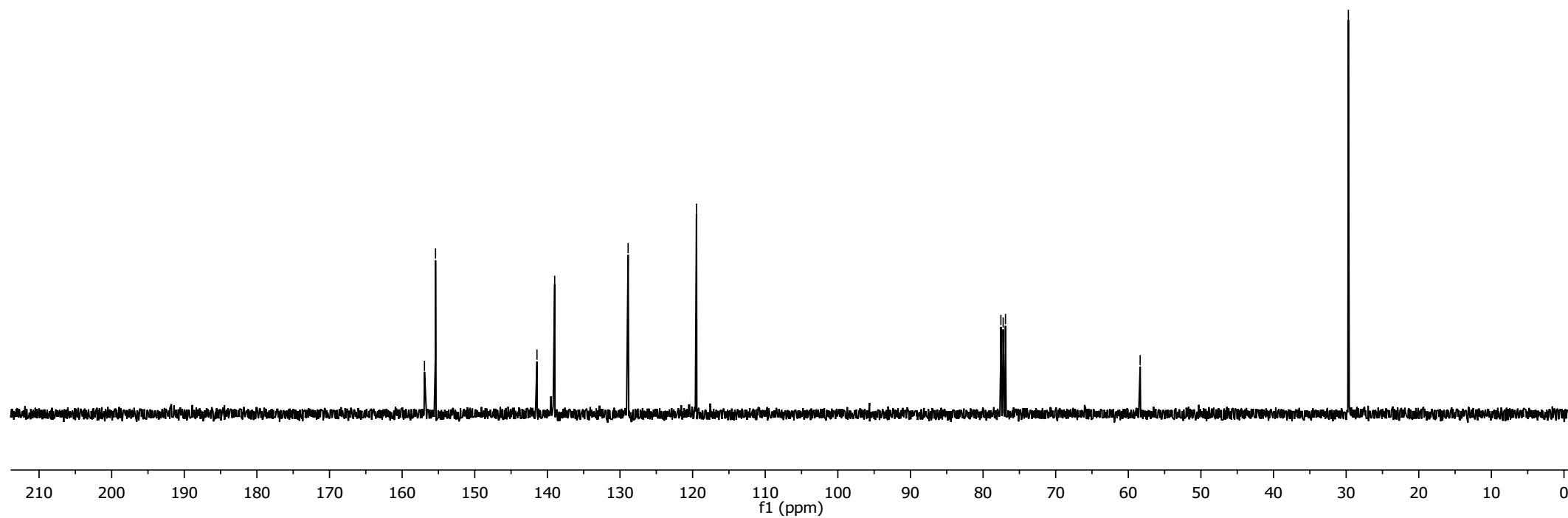
—128.88

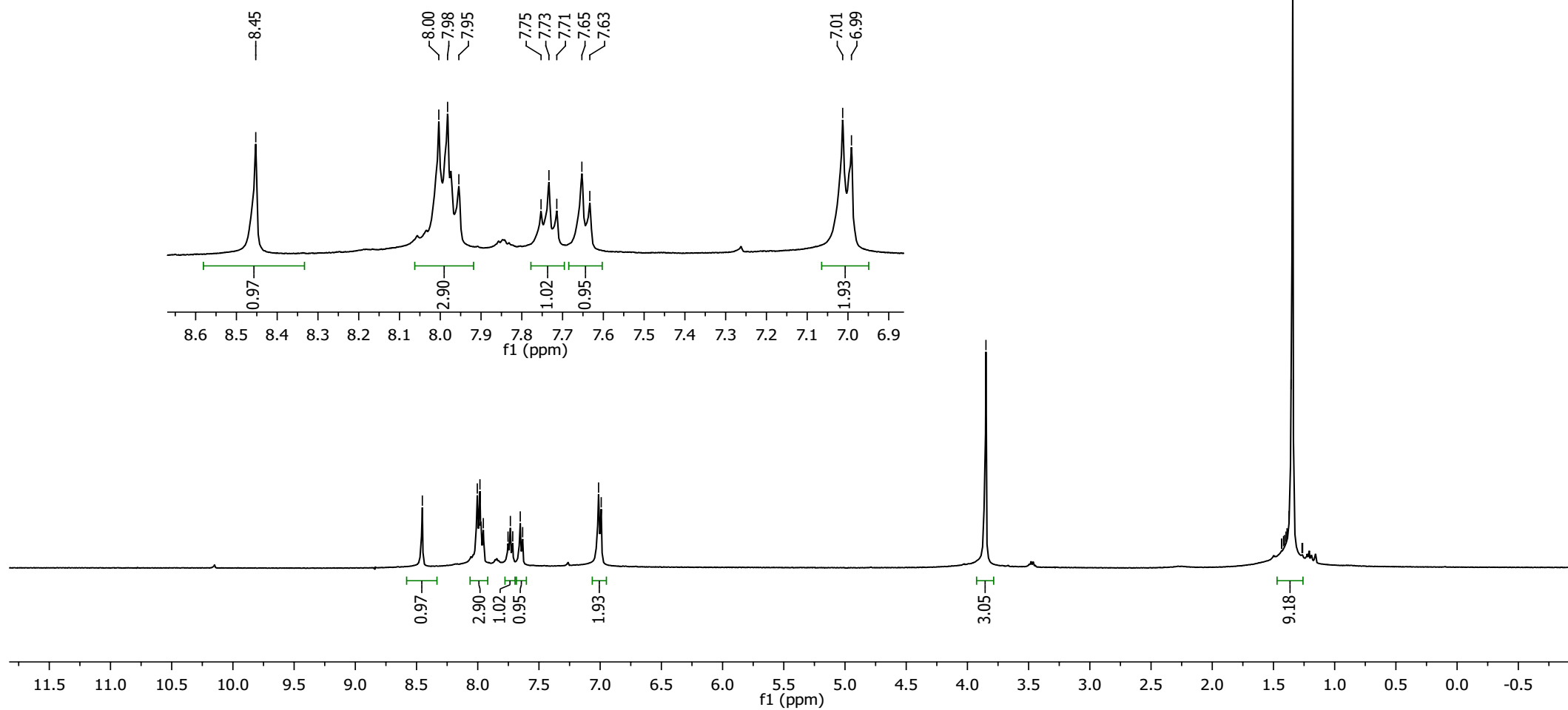
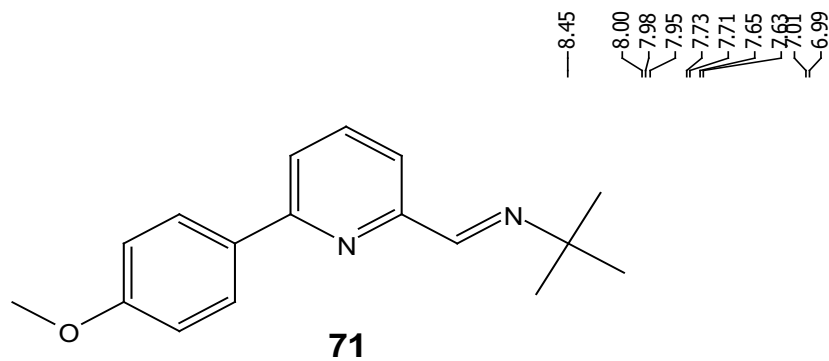
—119.46

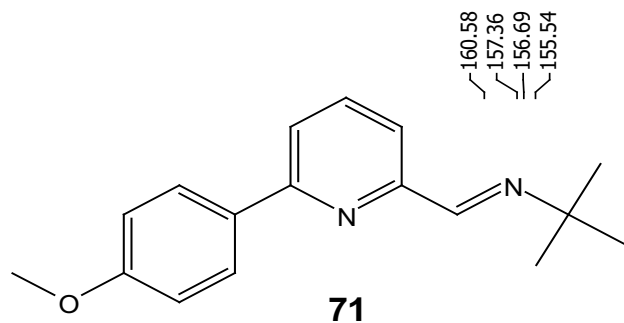
77.55 CDCl₃
77.23 CDCl₃
76.91 CDCl₃

—58.37

—29.69







160.58
157.36
156.69
155.54

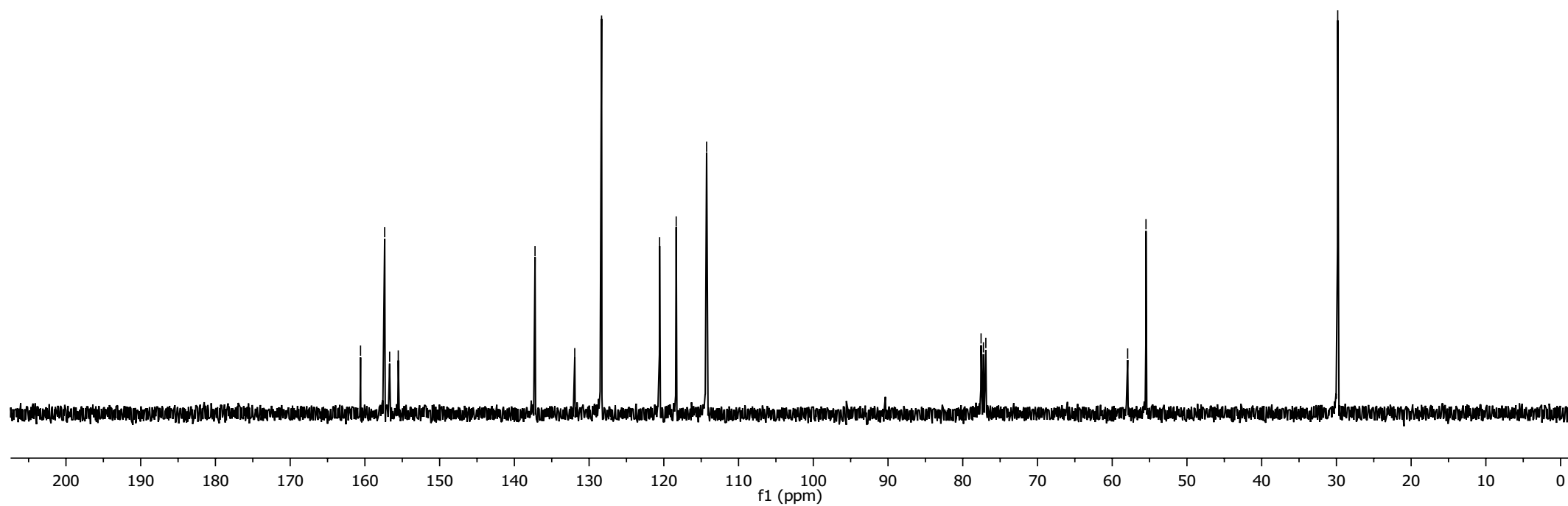
137.24
131.92
128.33

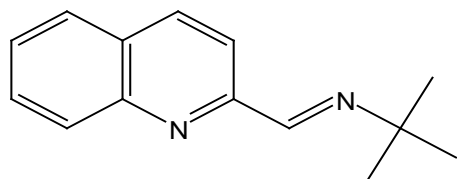
120.56
118.34
114.27

77.55 CDCl₃
77.23 CDCl₃
76.91 CDCl₃

57.93
55.48

29.82

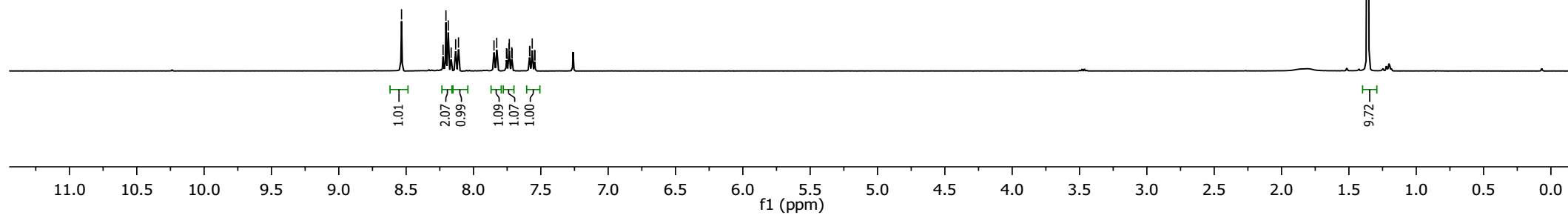
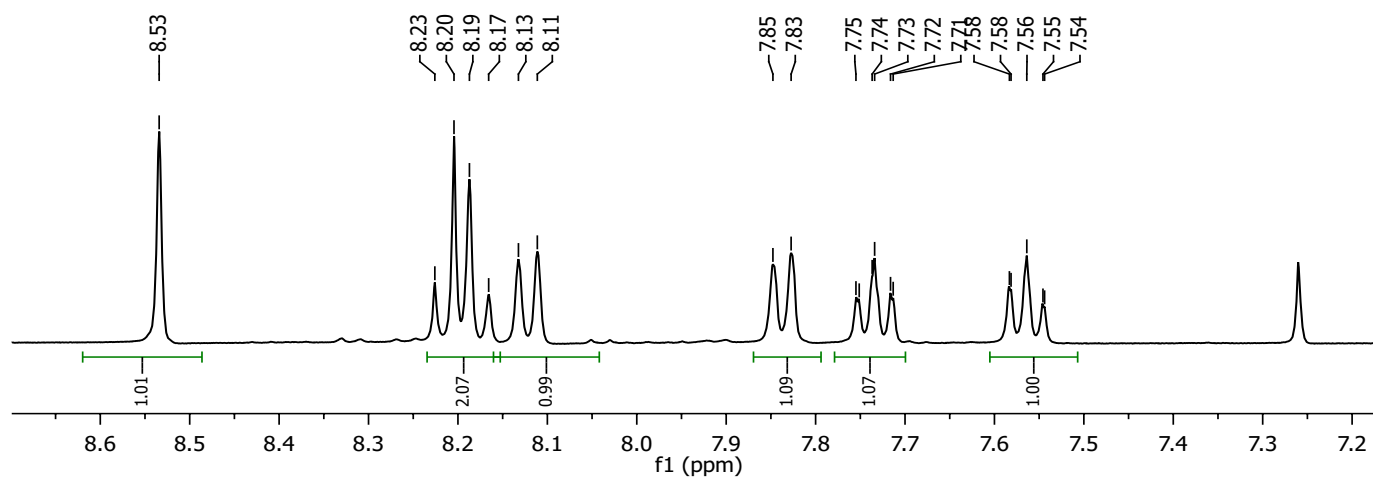


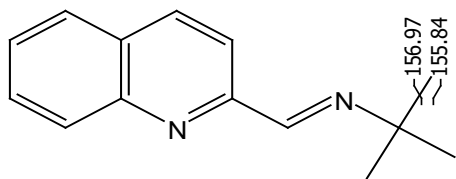
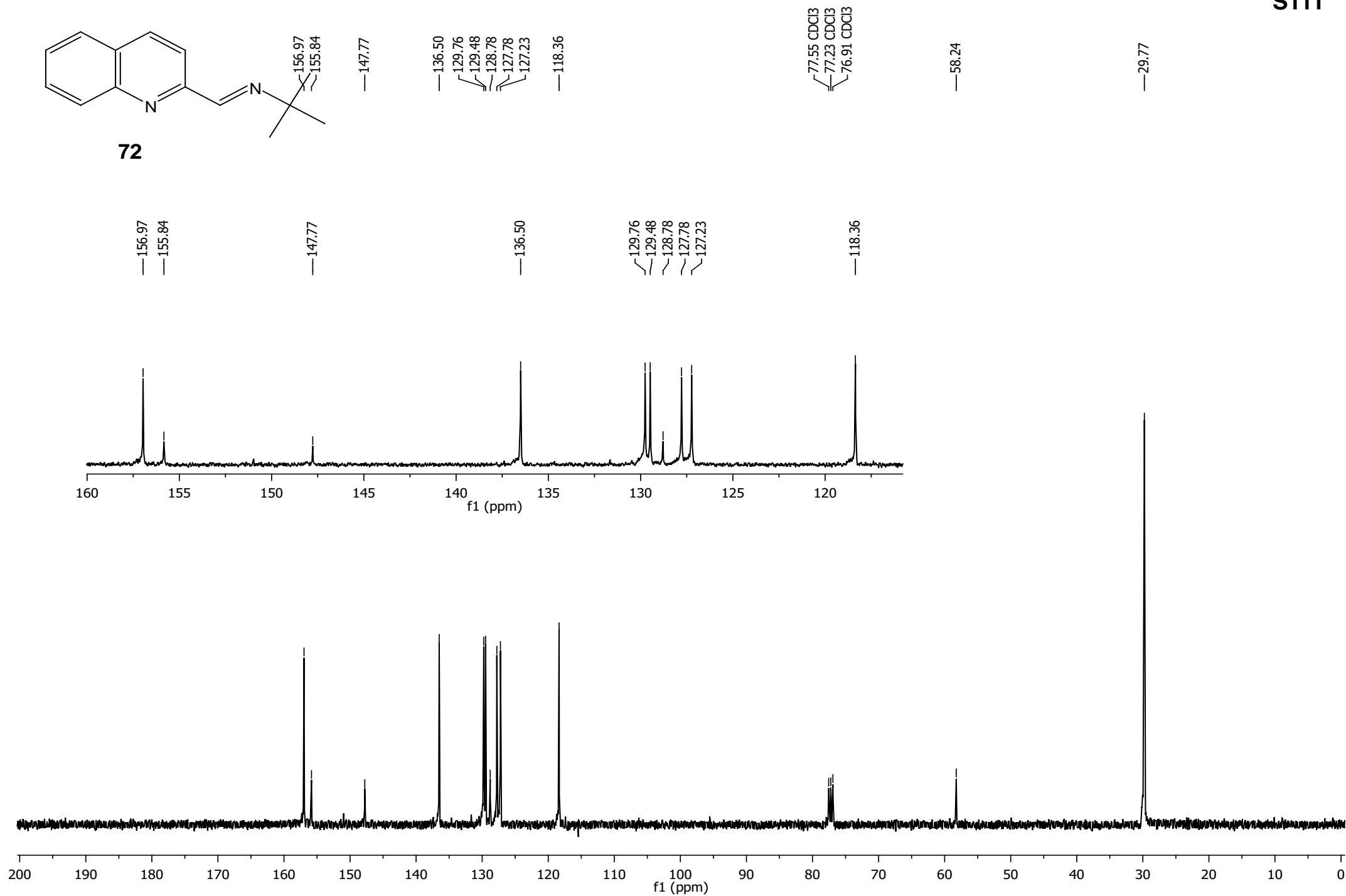


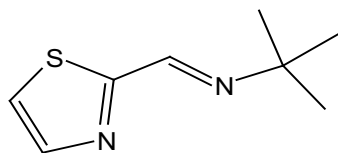
72

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7.71
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7.55
7.54

1.36



**72**

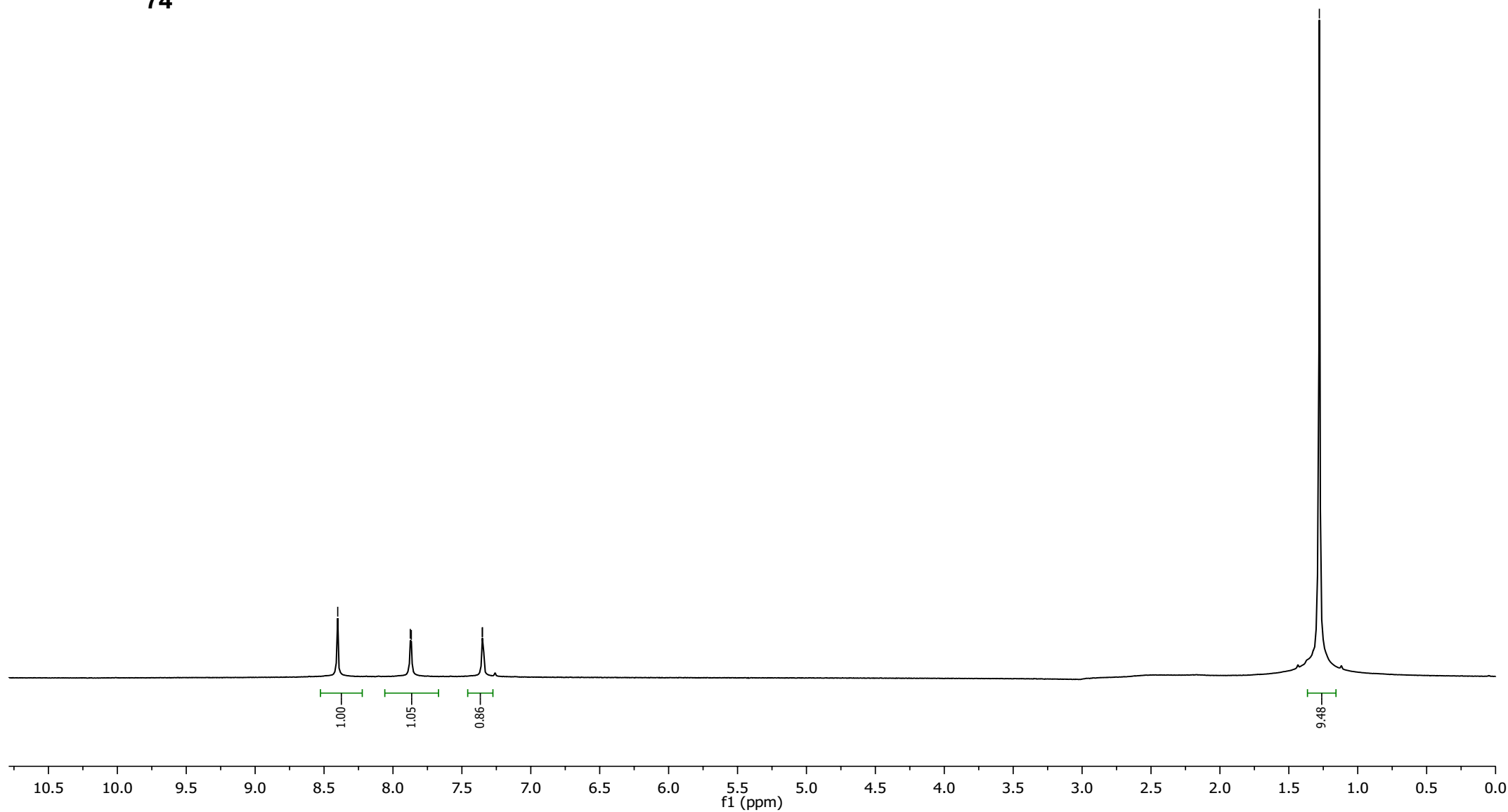


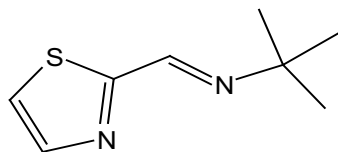
74

8.40

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1.28





74

—149.95

—143.86

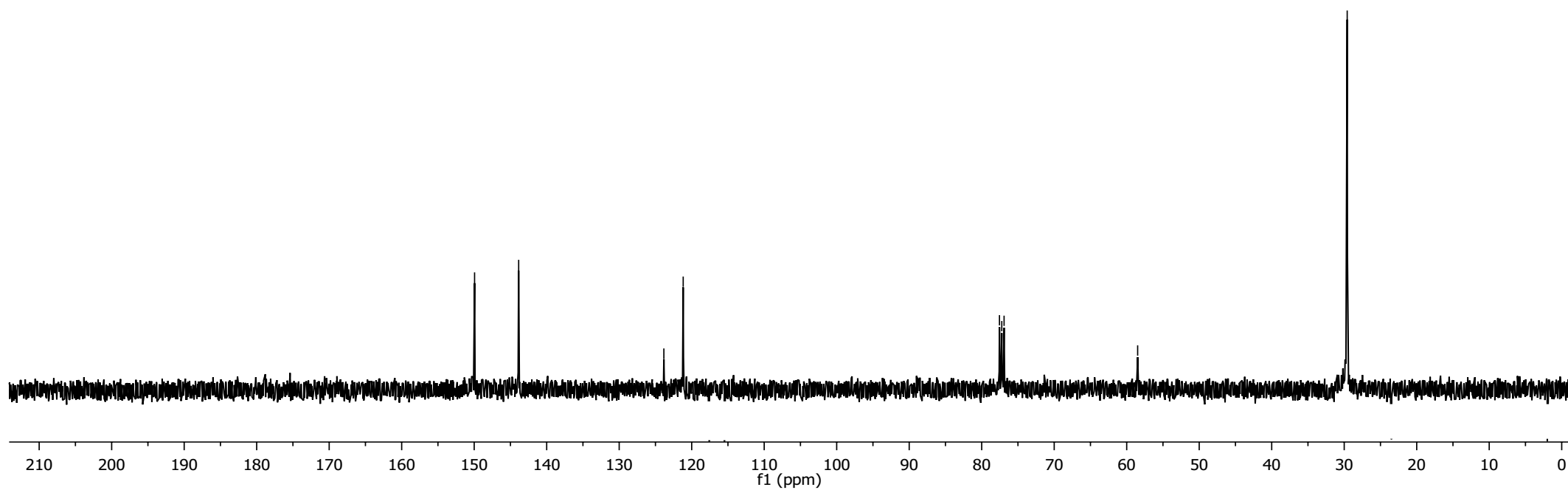
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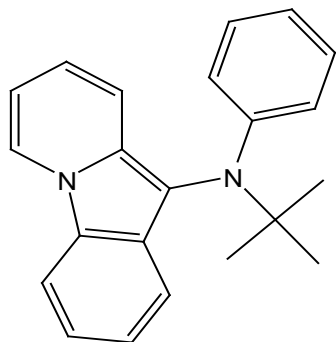
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77.23 CDCl₃
76.91 CDCl₃

—58.49

—29.59

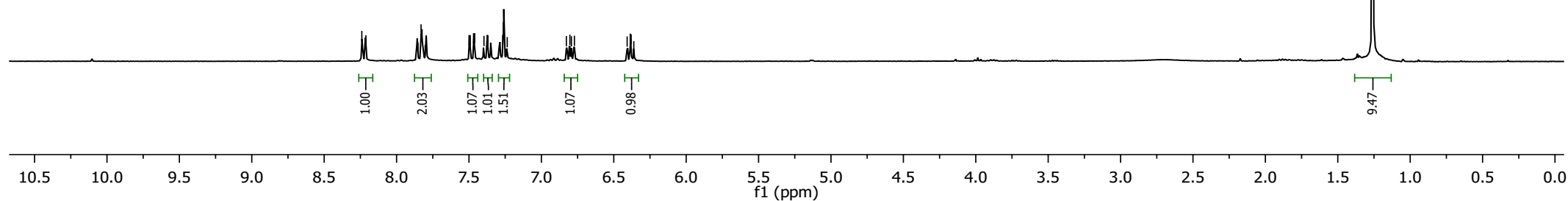
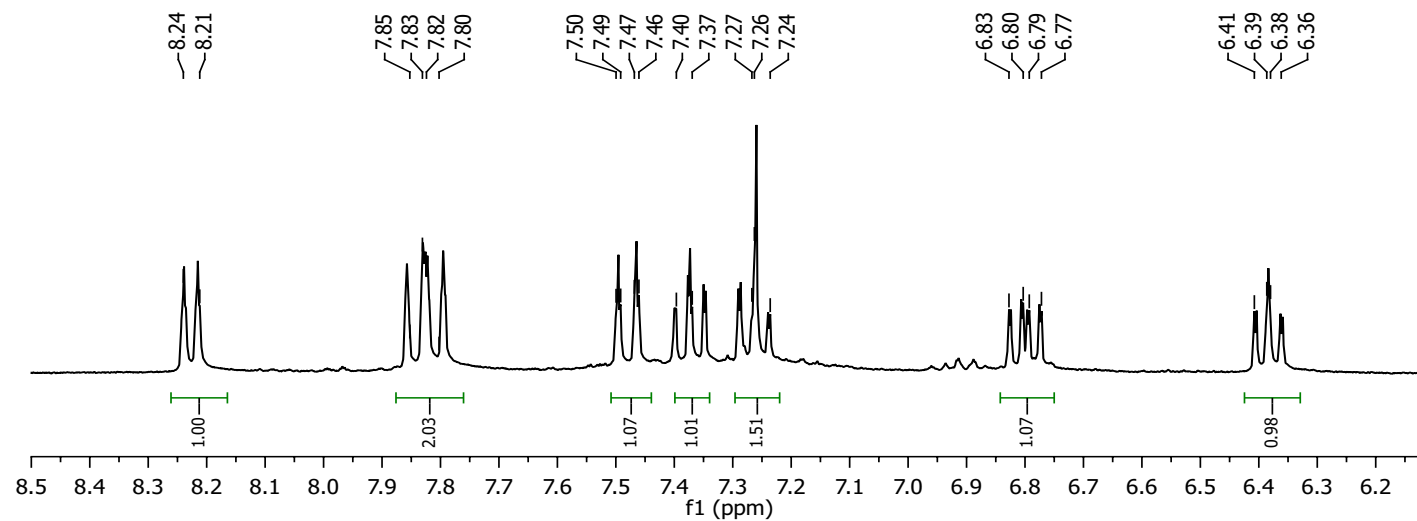


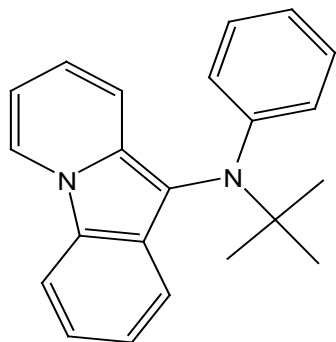


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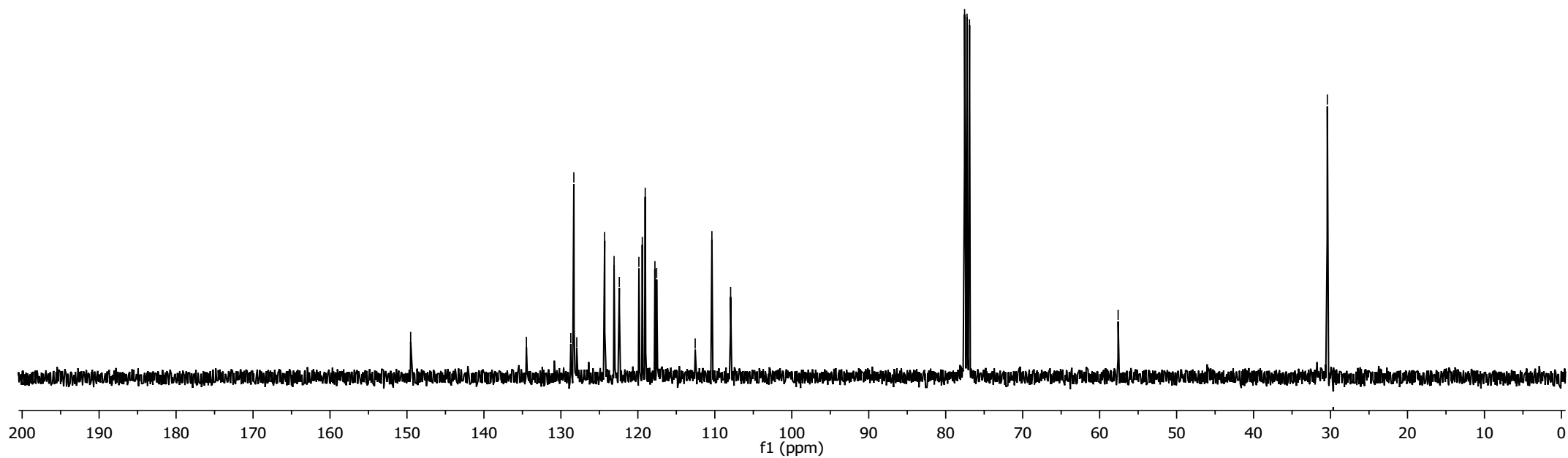
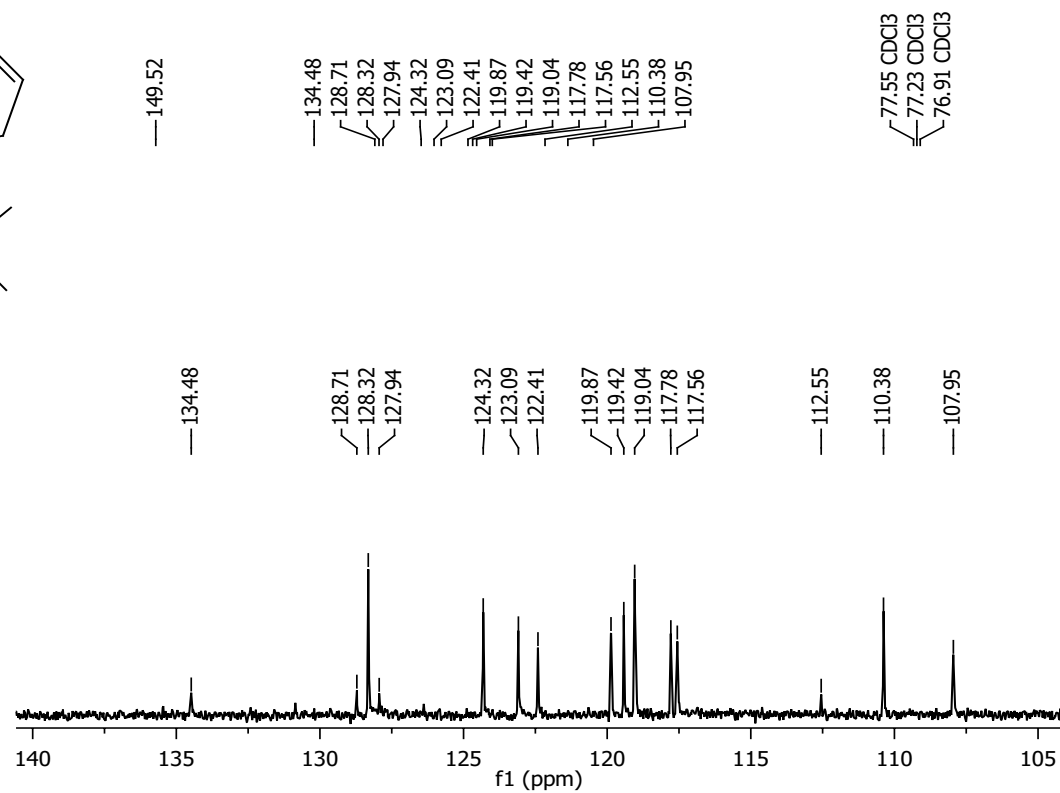
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6.36

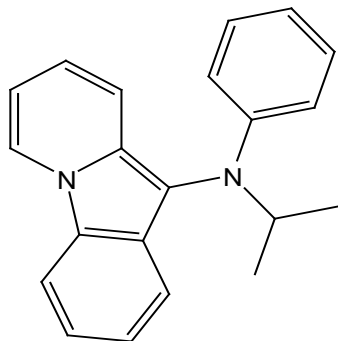
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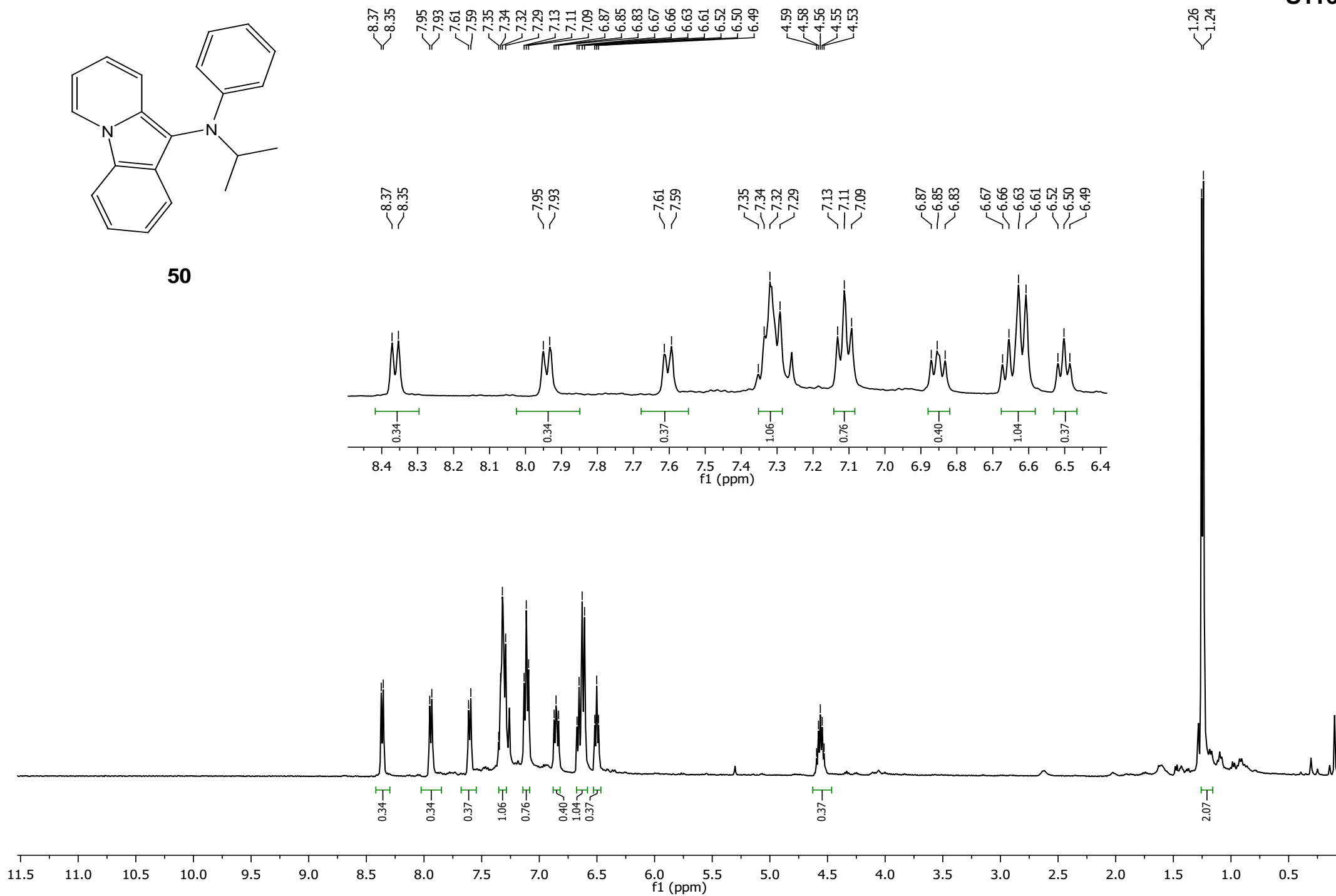


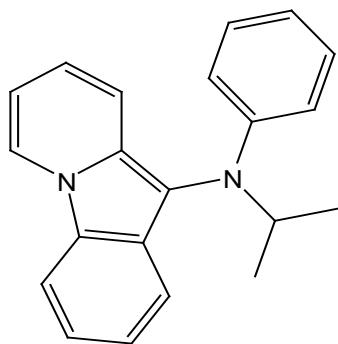
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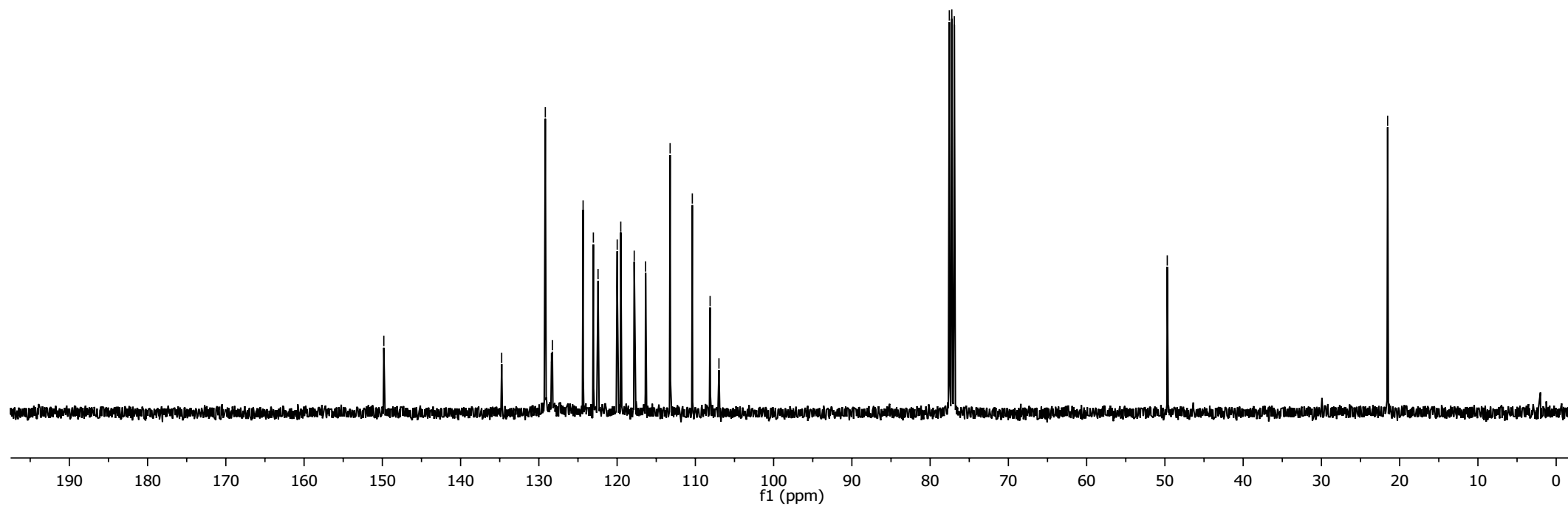
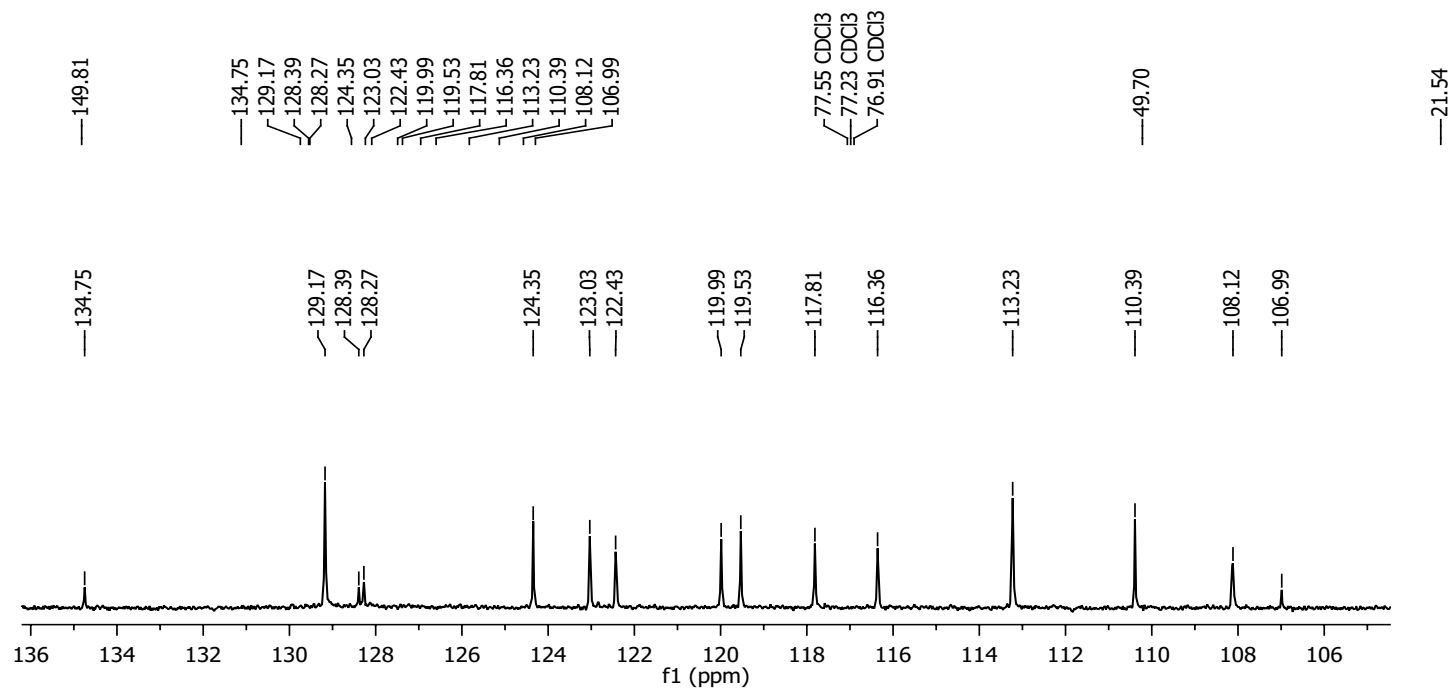


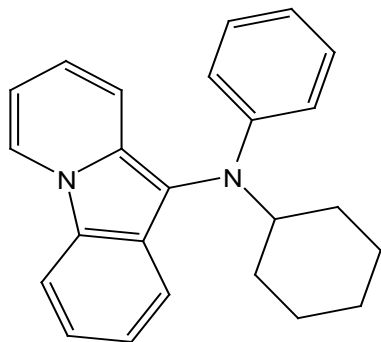
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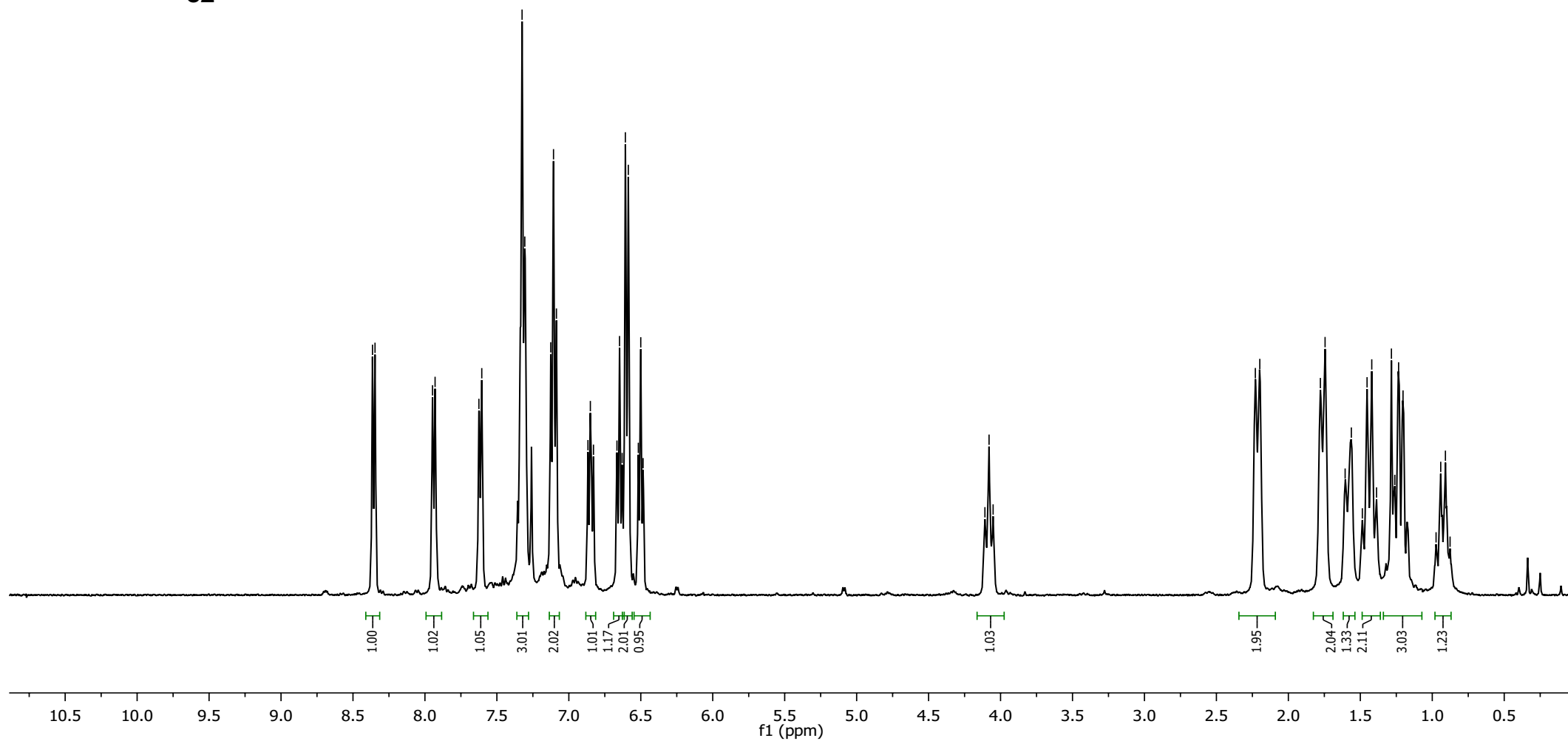


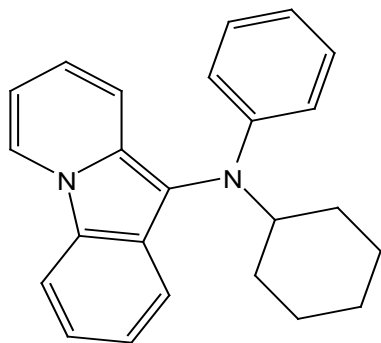
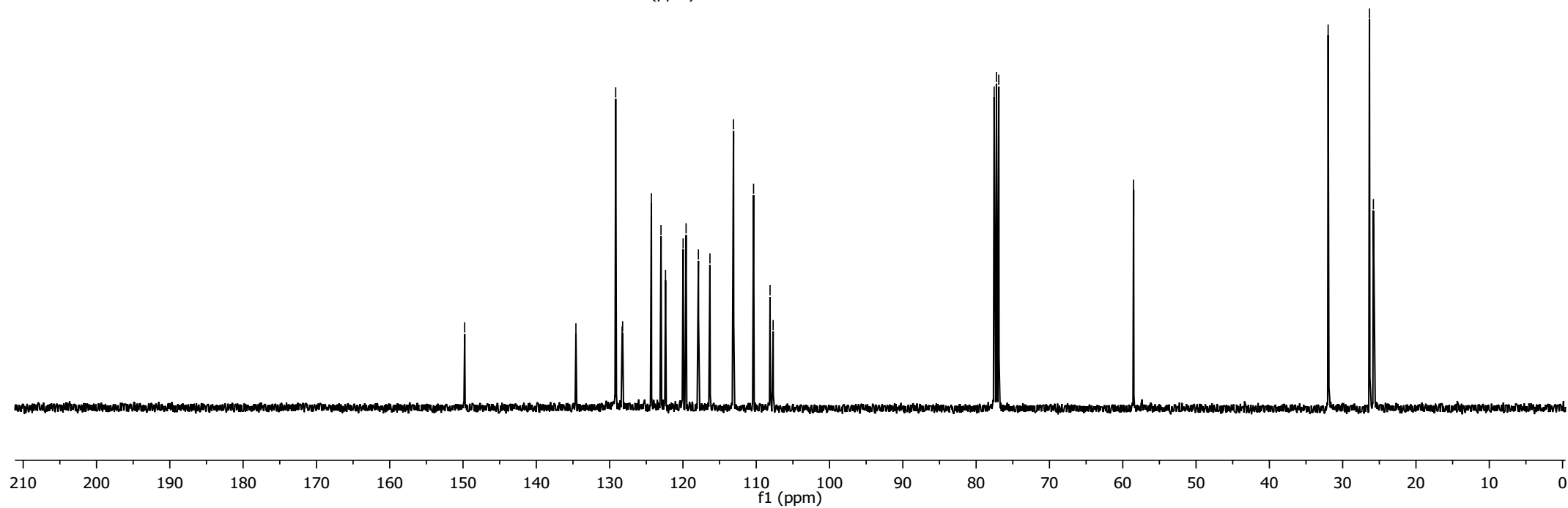
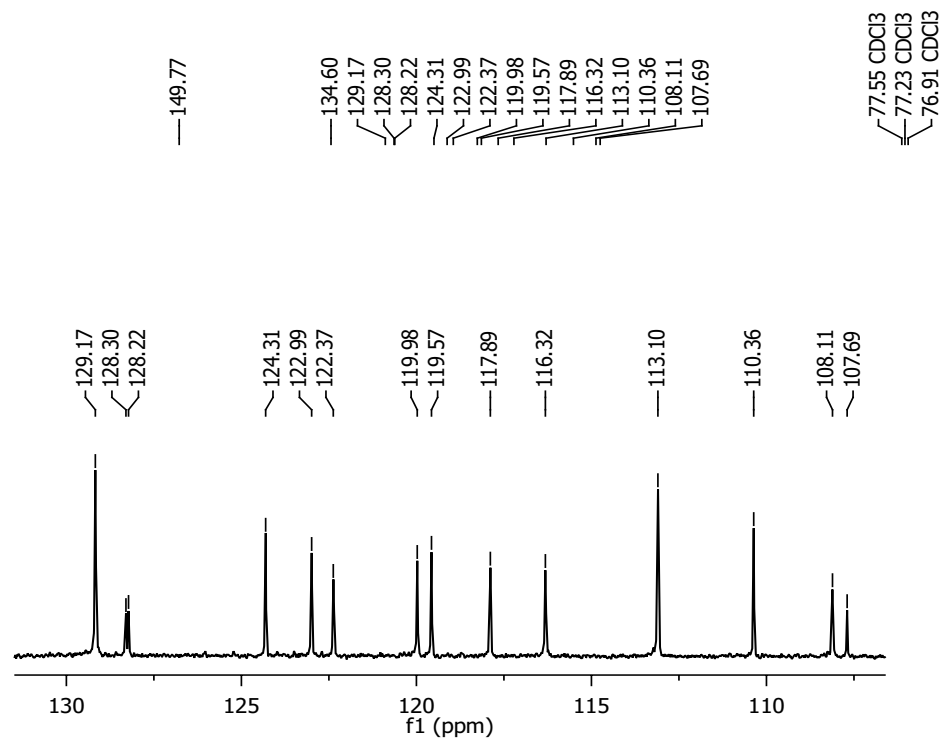
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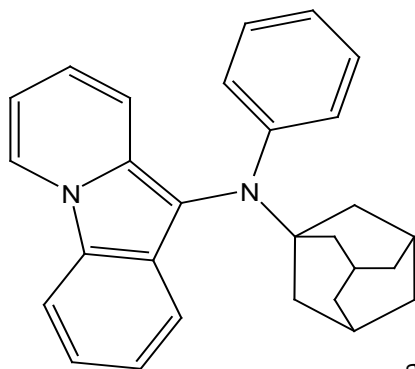
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4.08
4.05

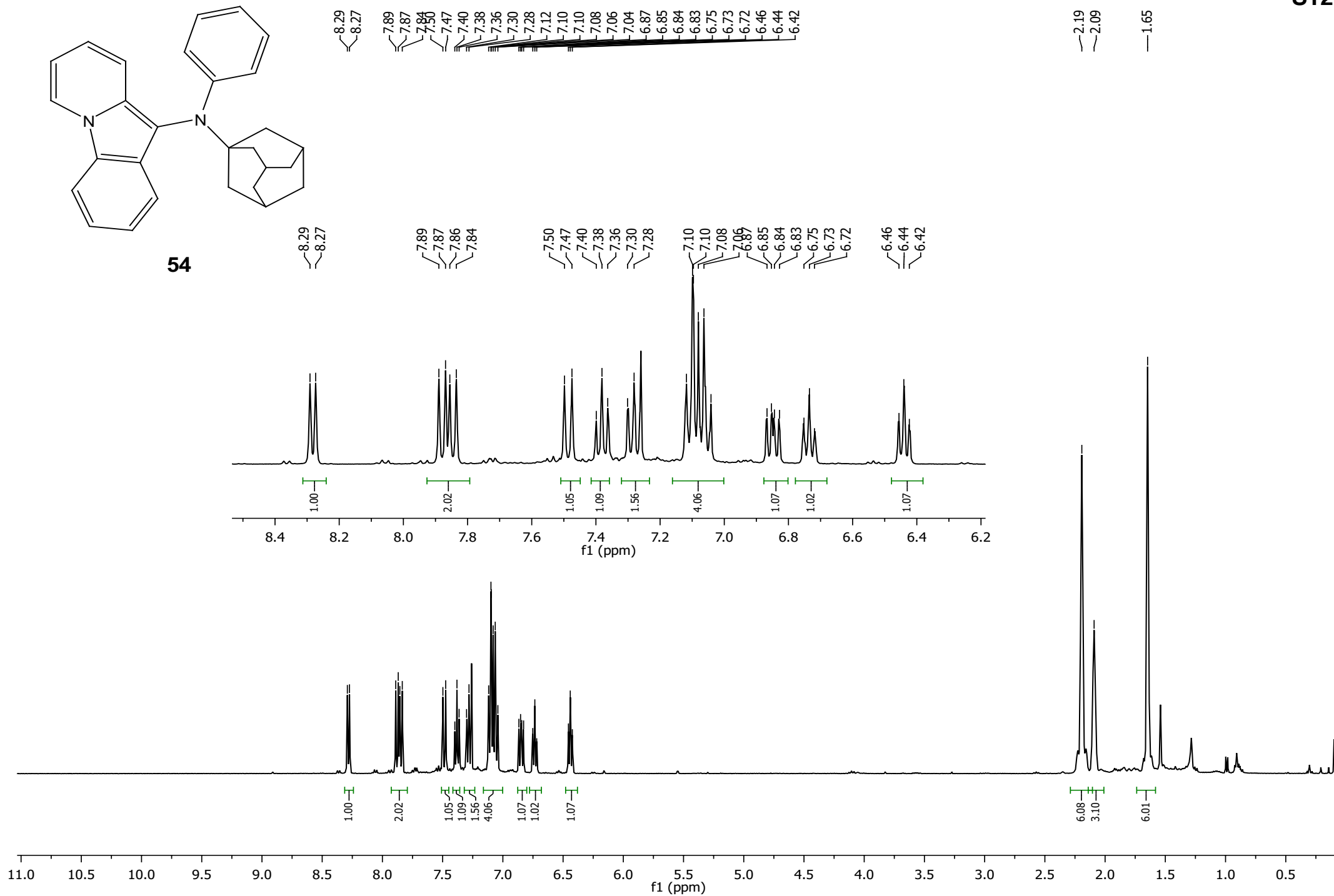
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0.91

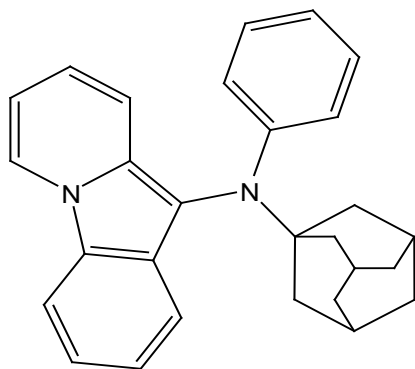
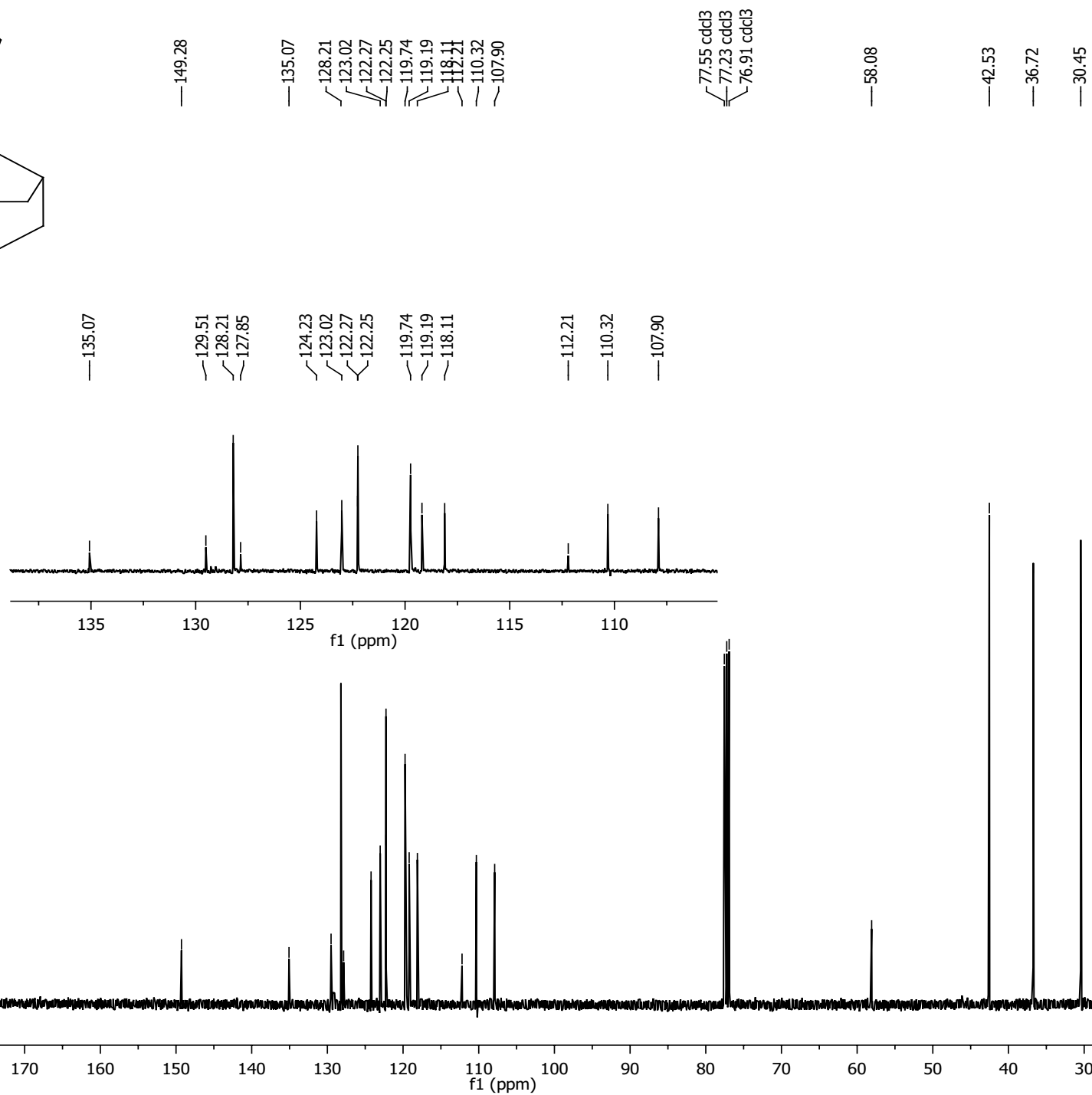


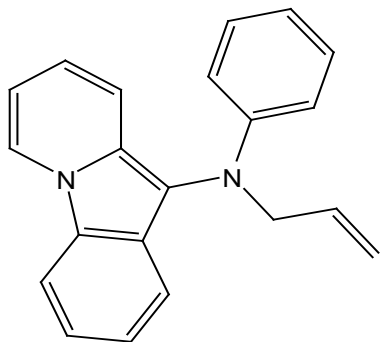
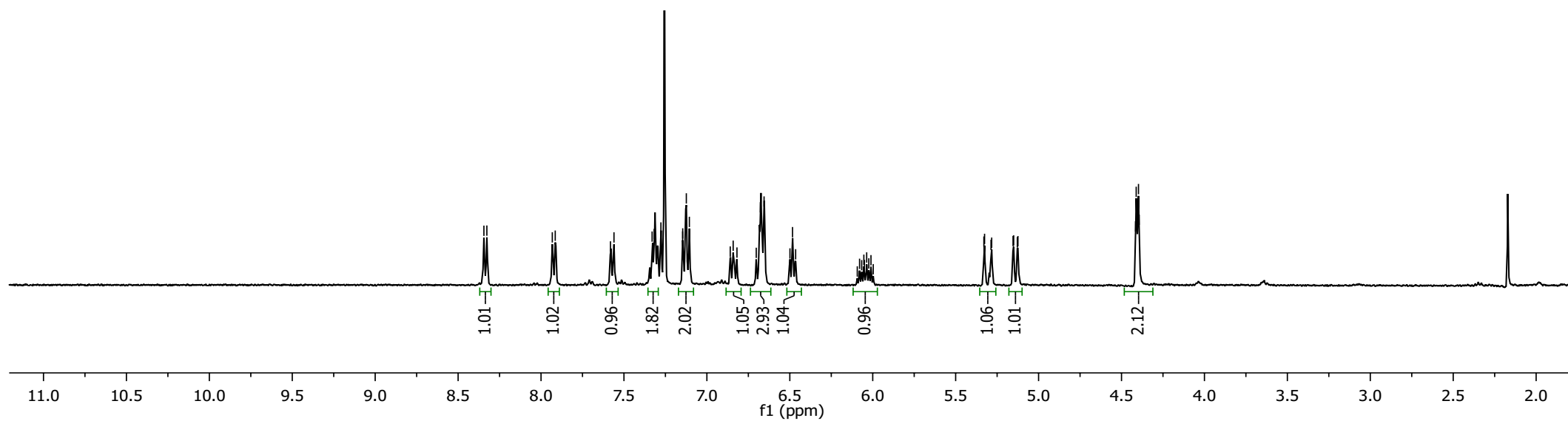
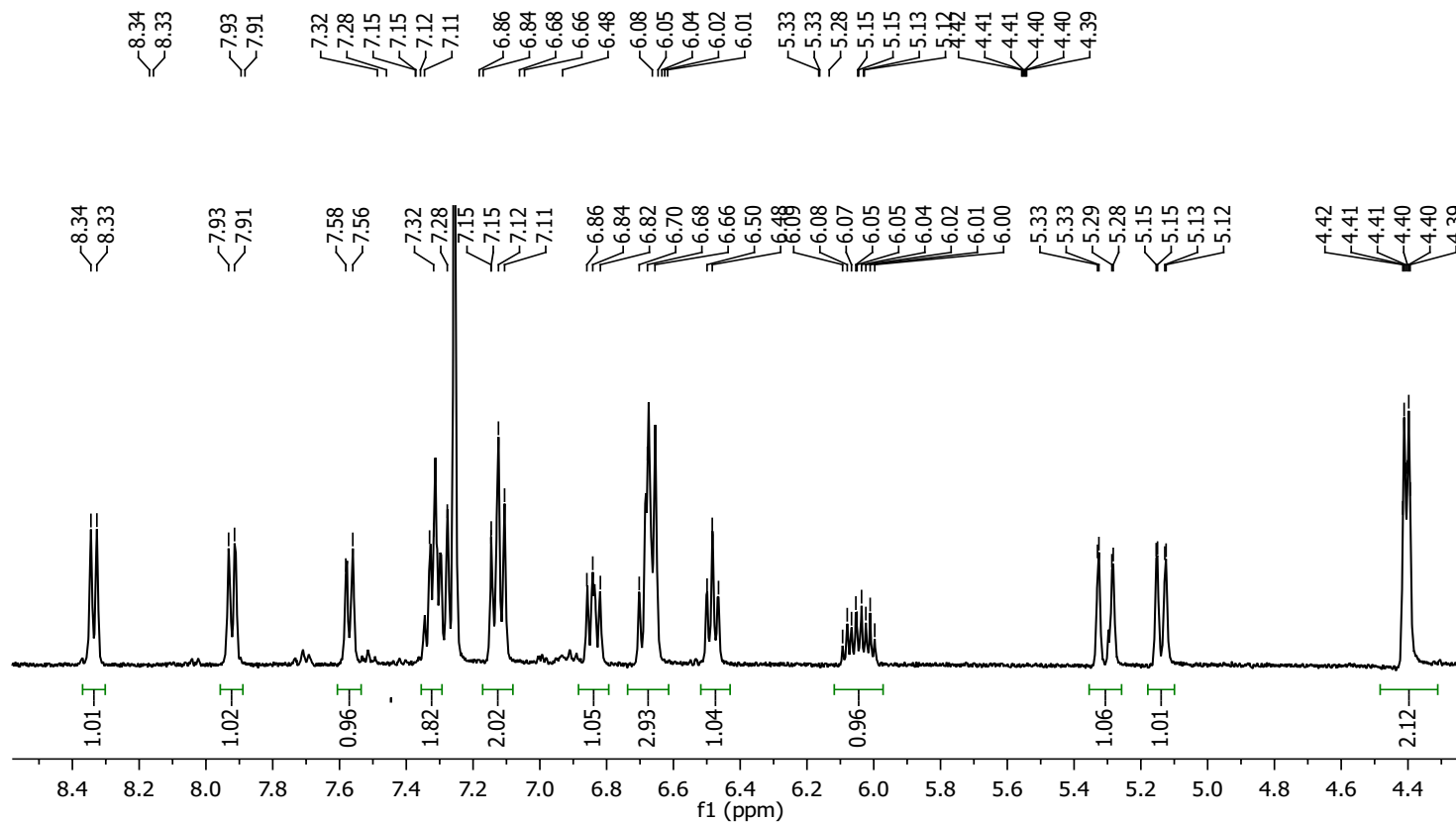
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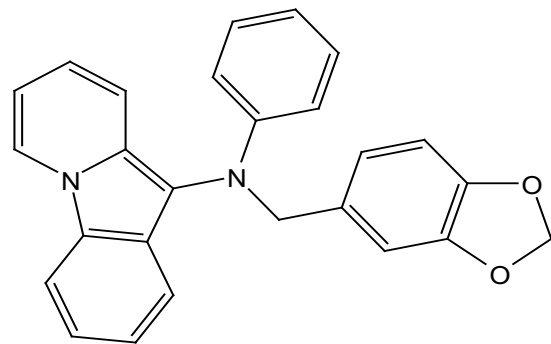


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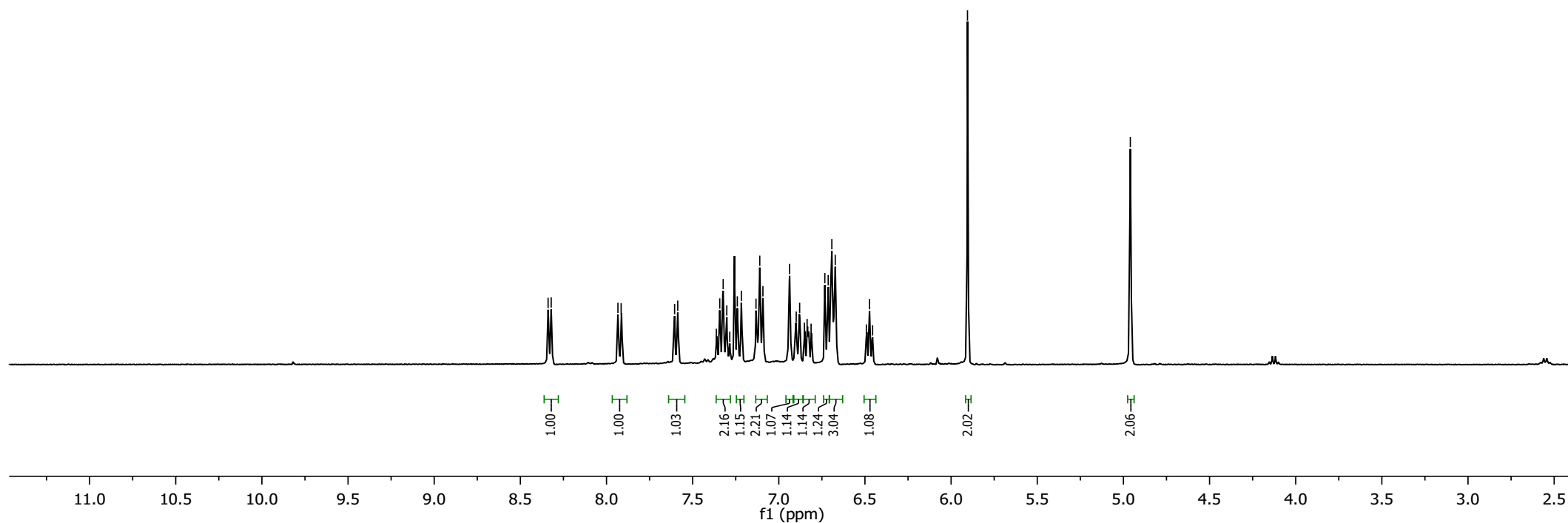
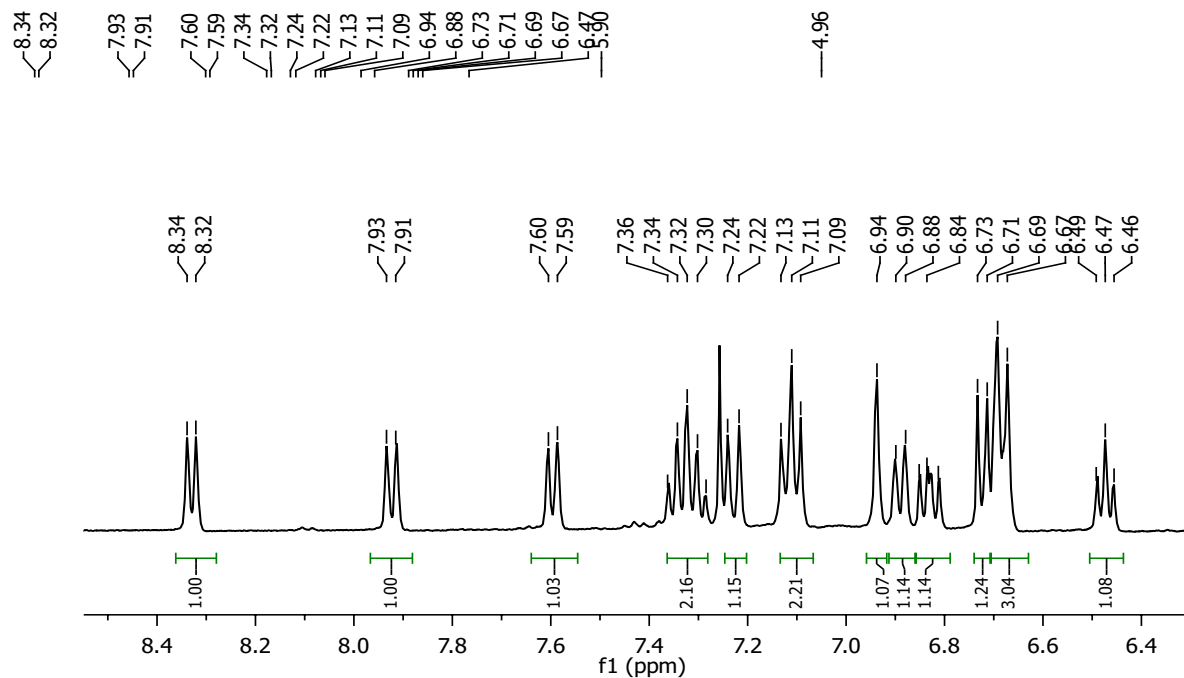


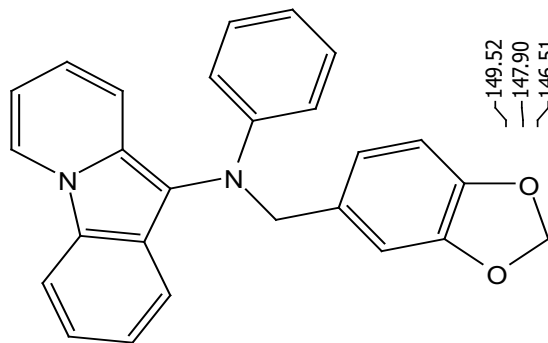
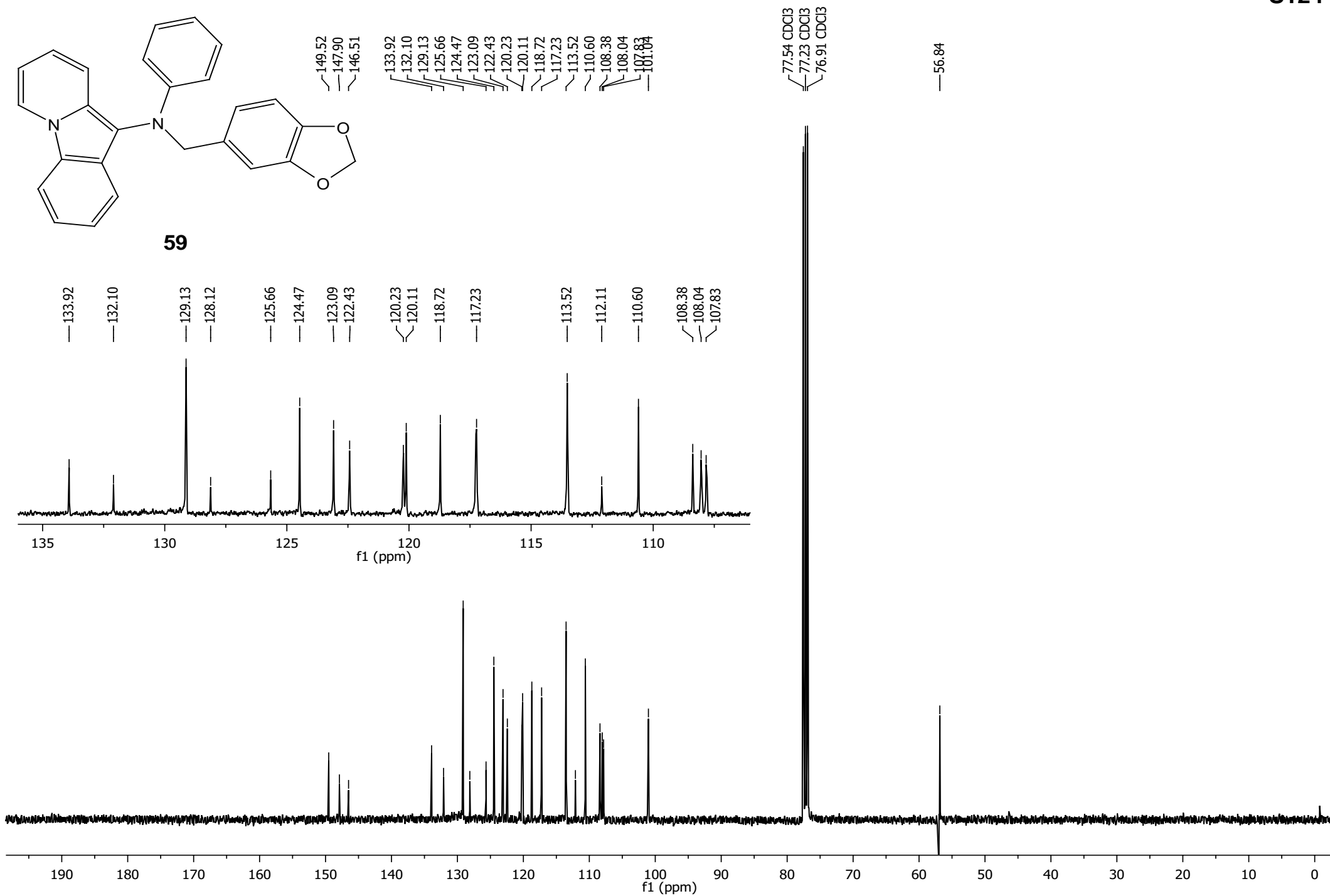
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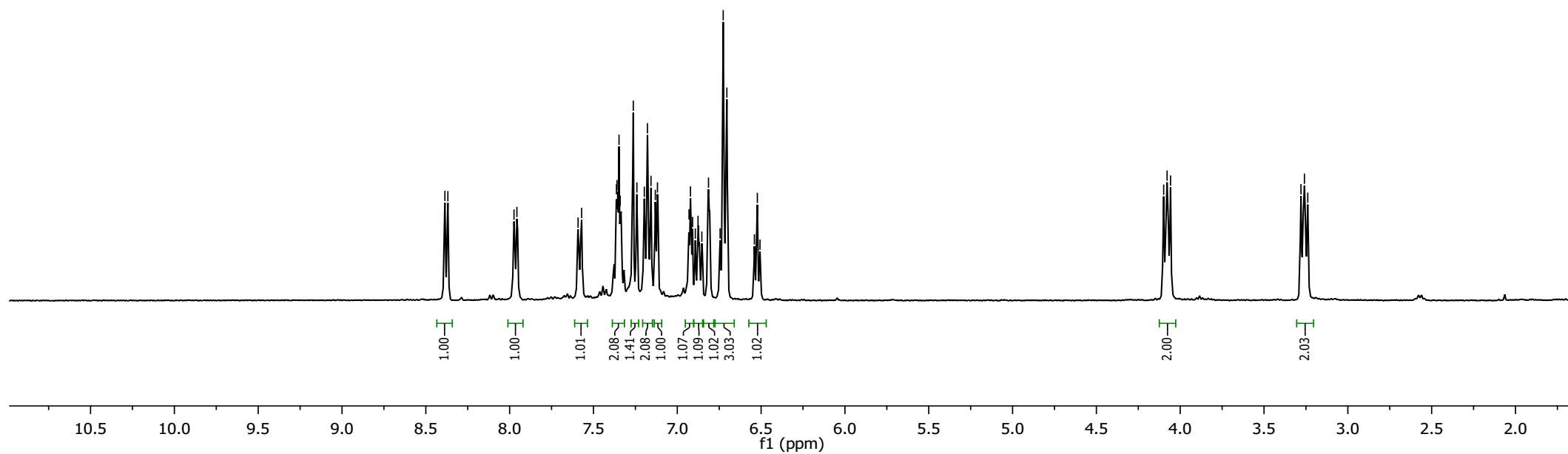
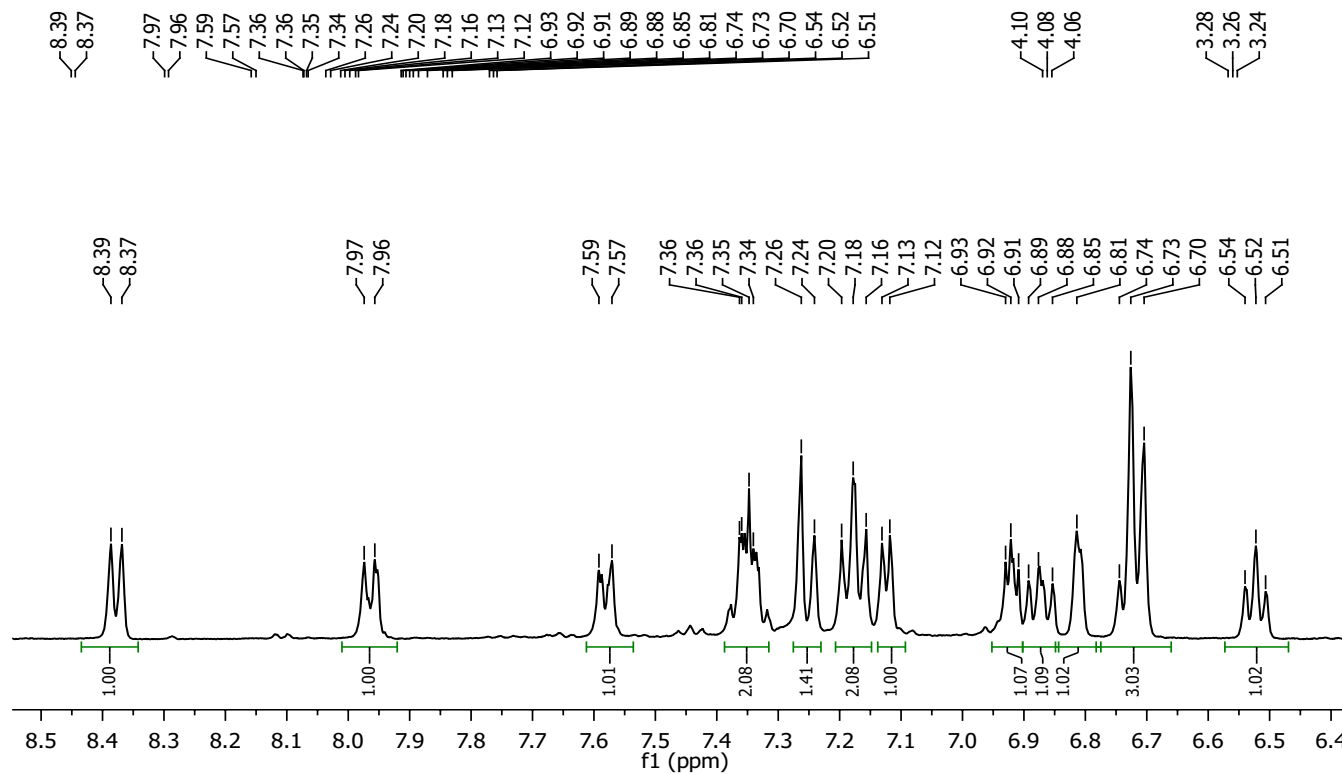
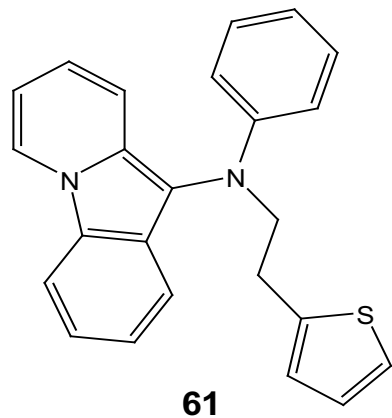
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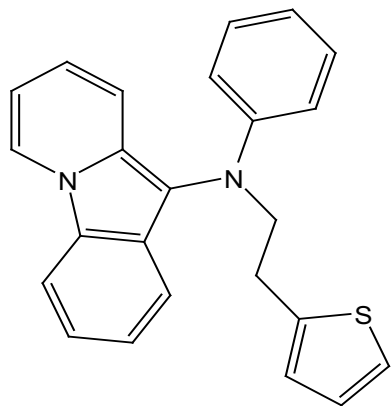


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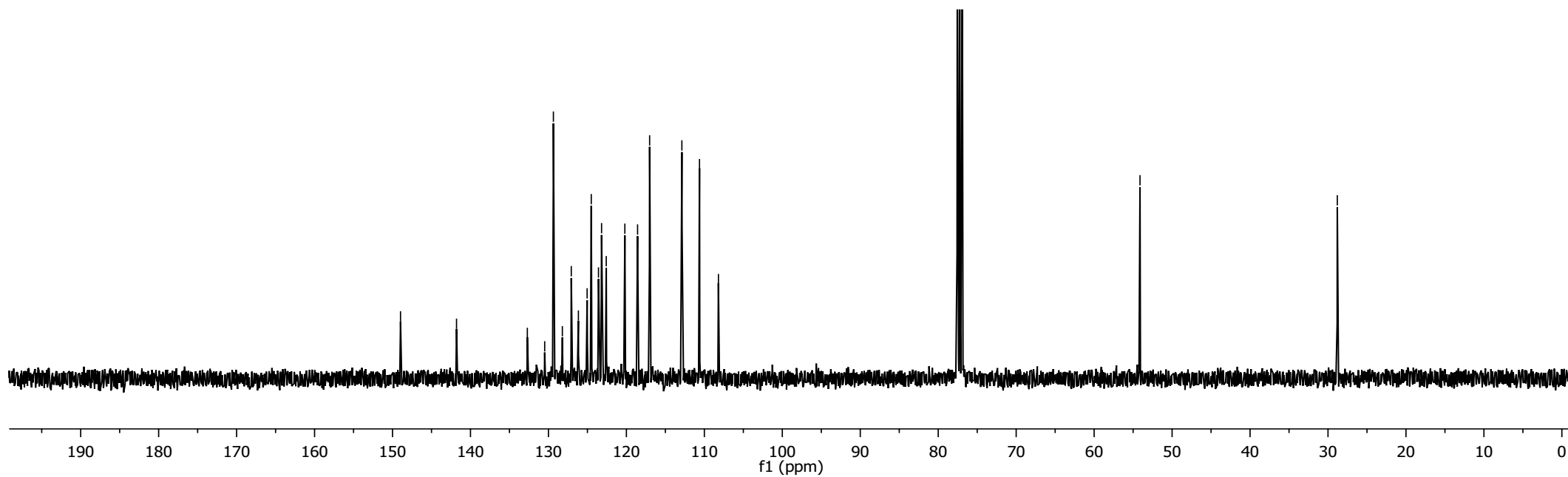
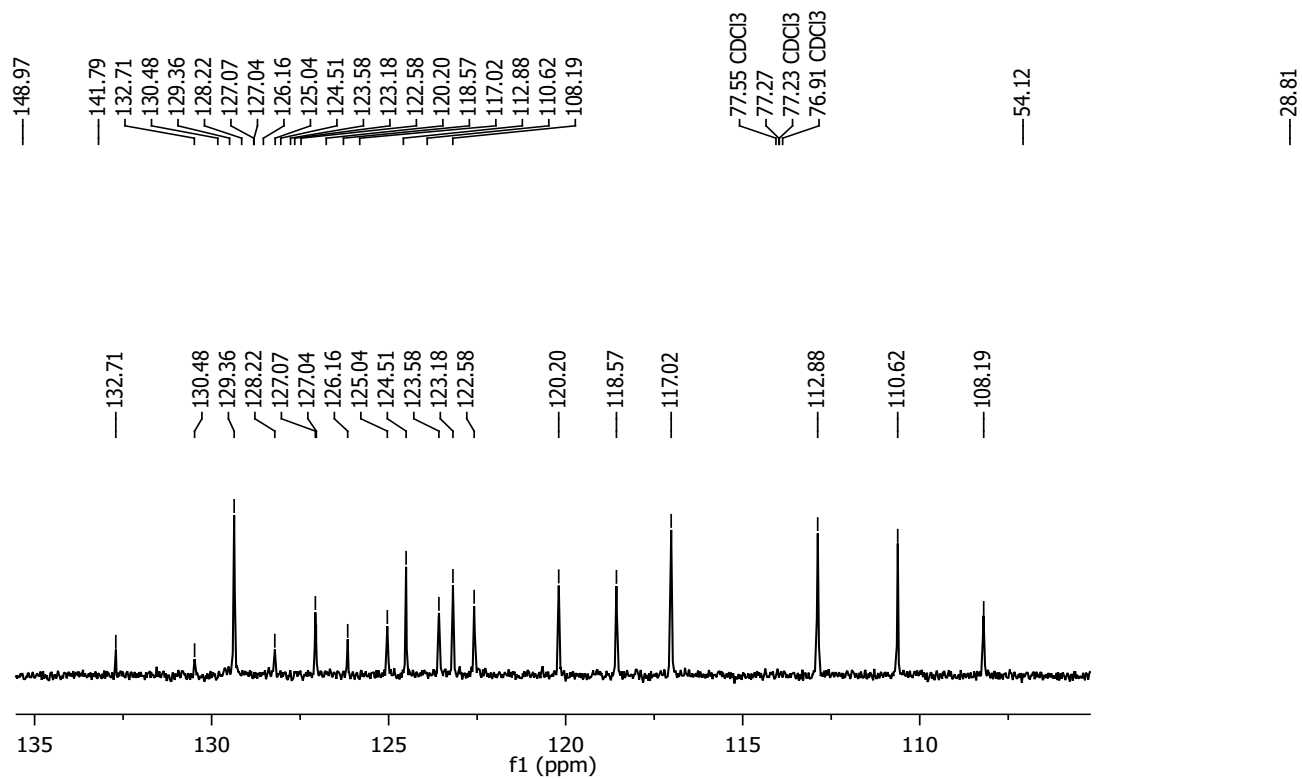


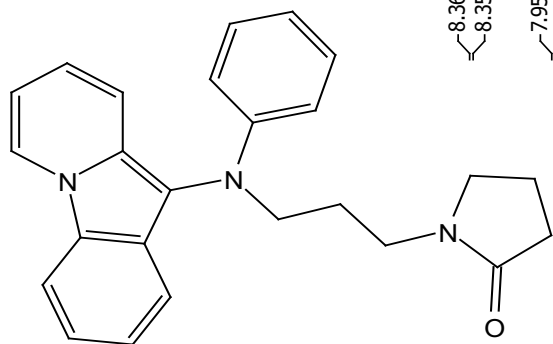
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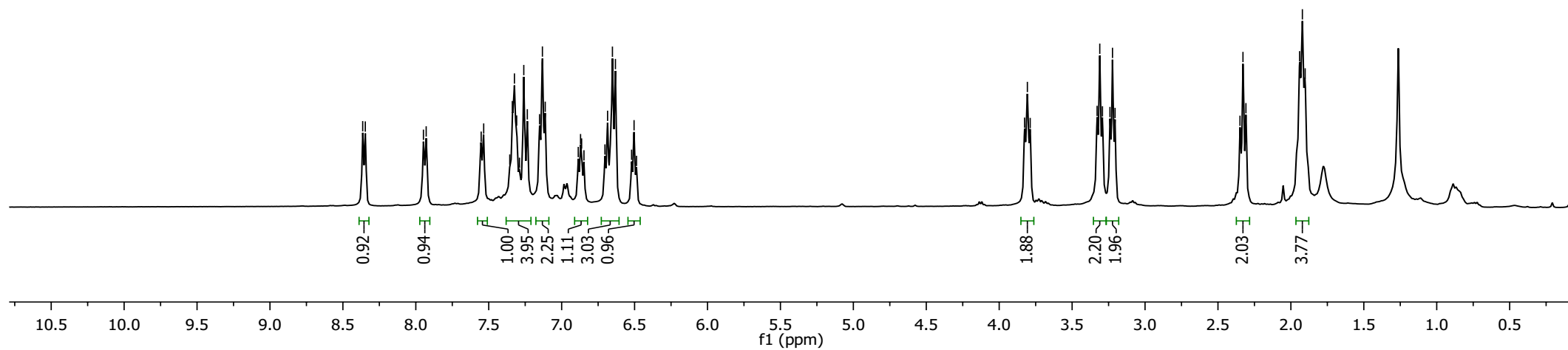


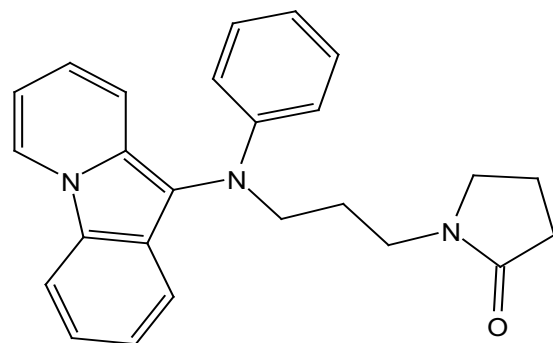
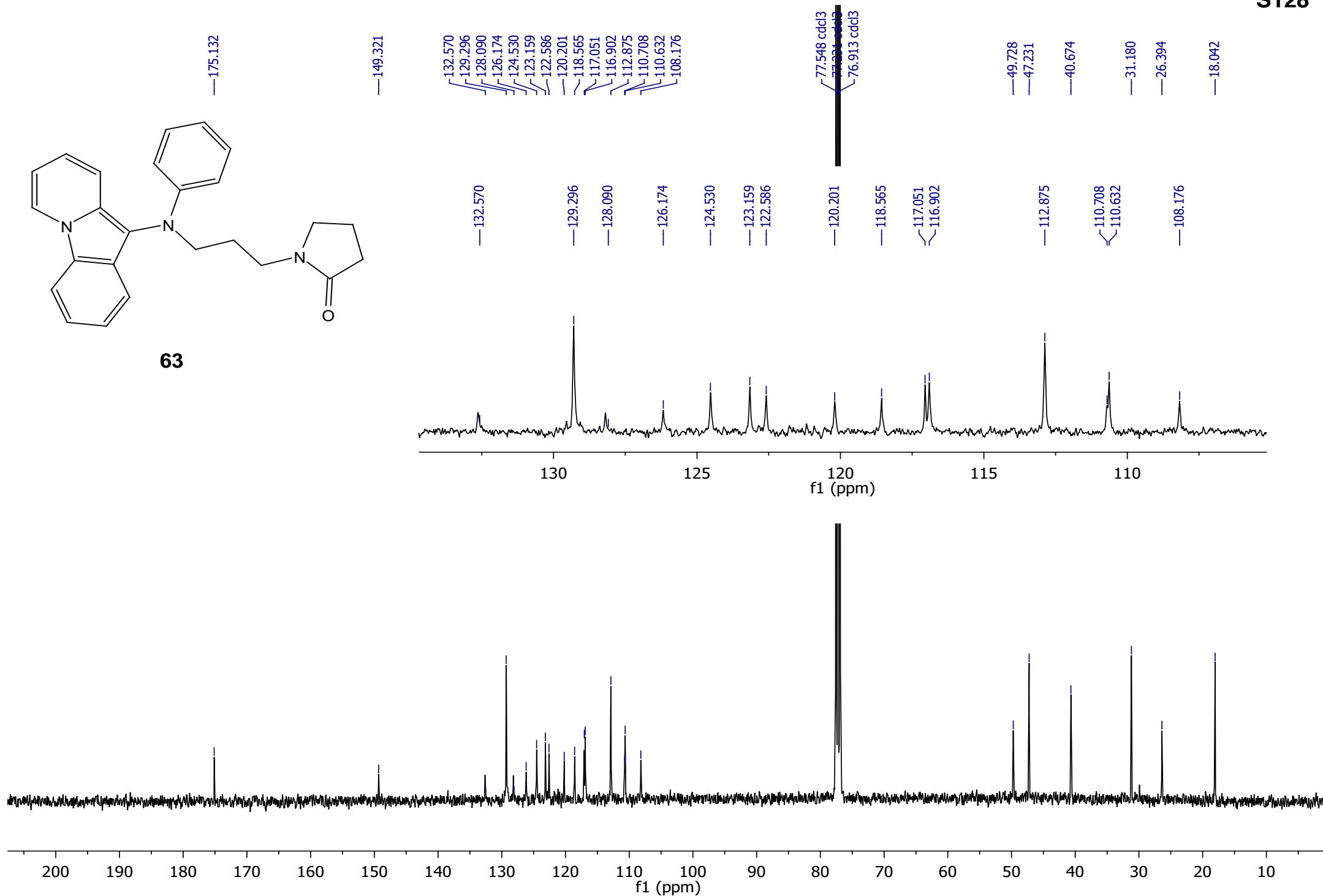
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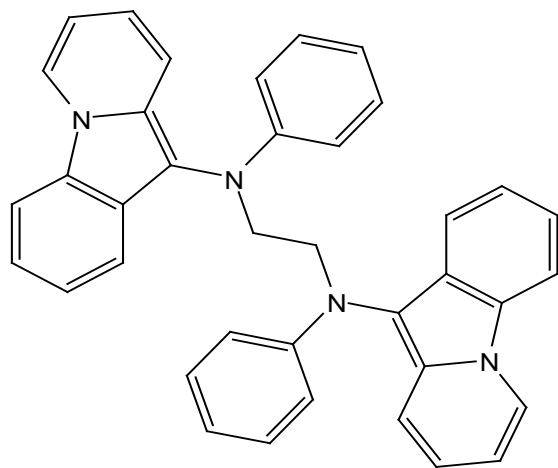
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6.65
6.63
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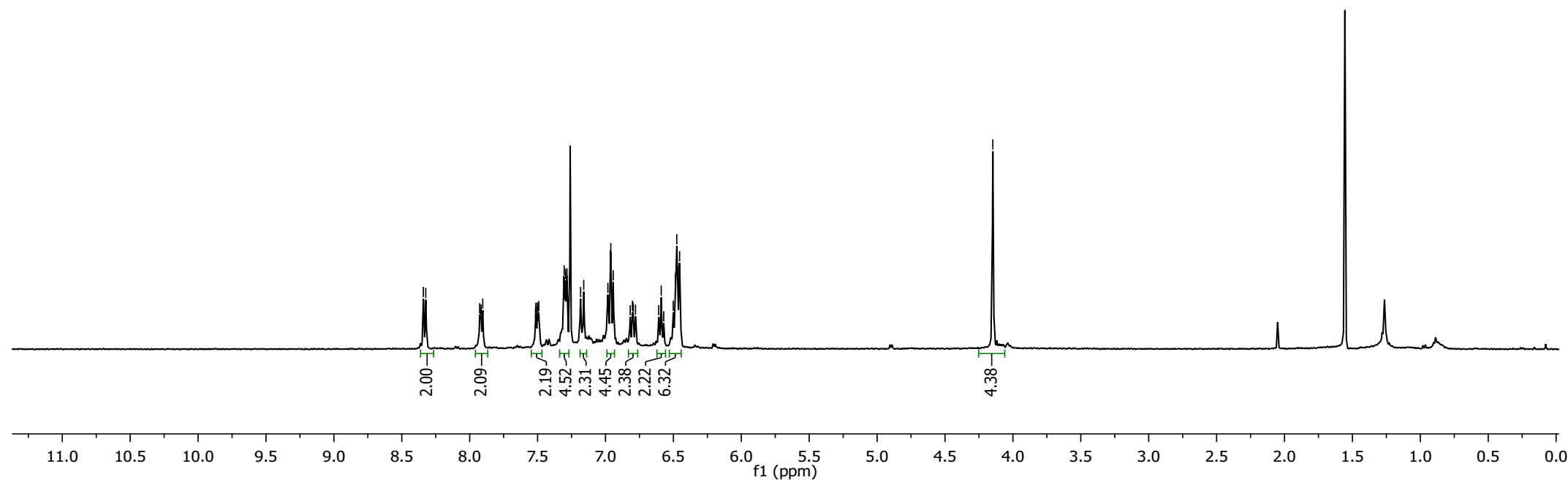
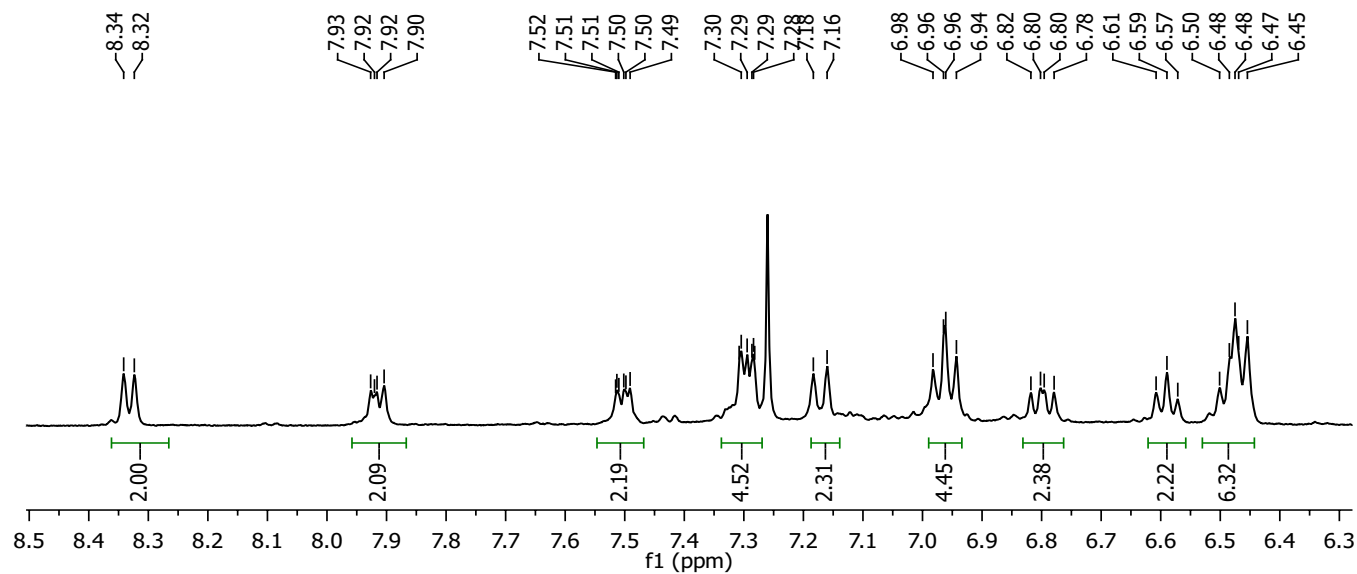
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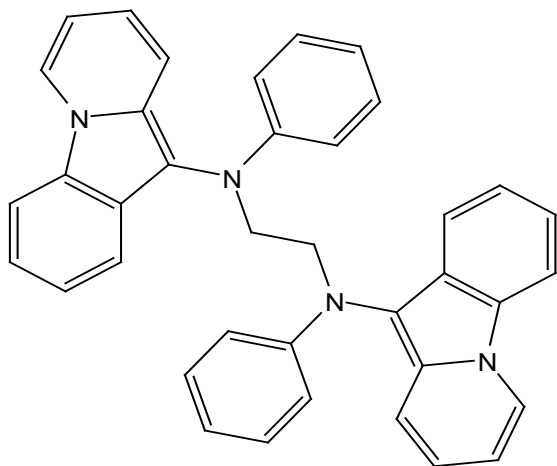


**63**

**65**

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7.30
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6.94
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6.80
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6.50
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6.47
6.45
6.45





65

—149.19

132.55
129.18
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123.18
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110.57
108.11

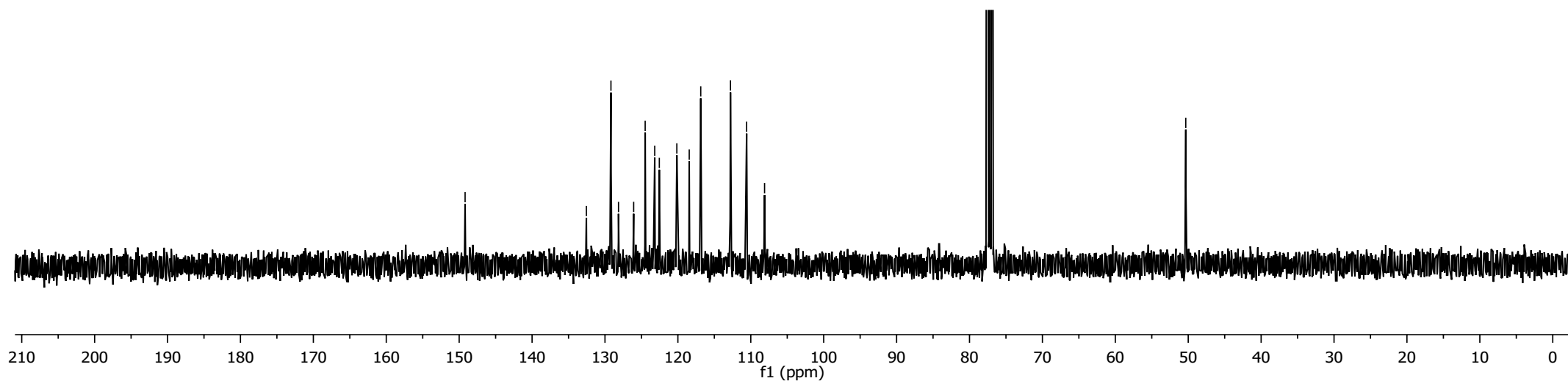
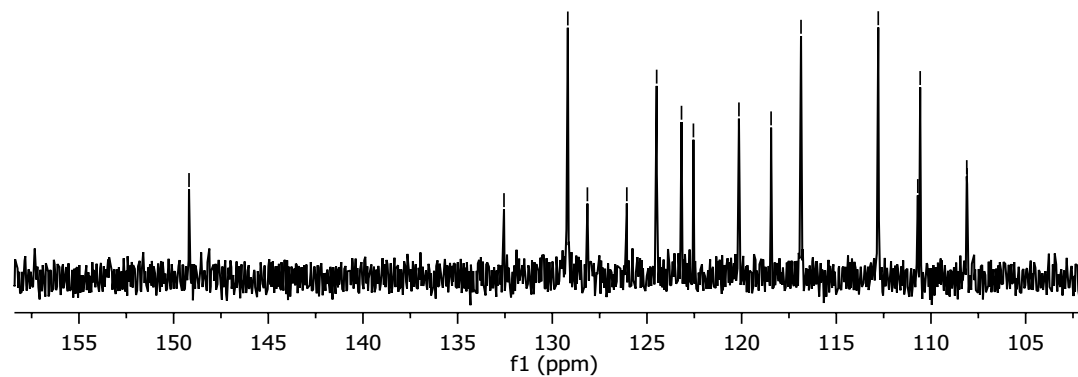
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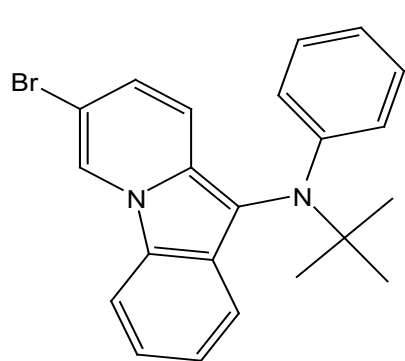
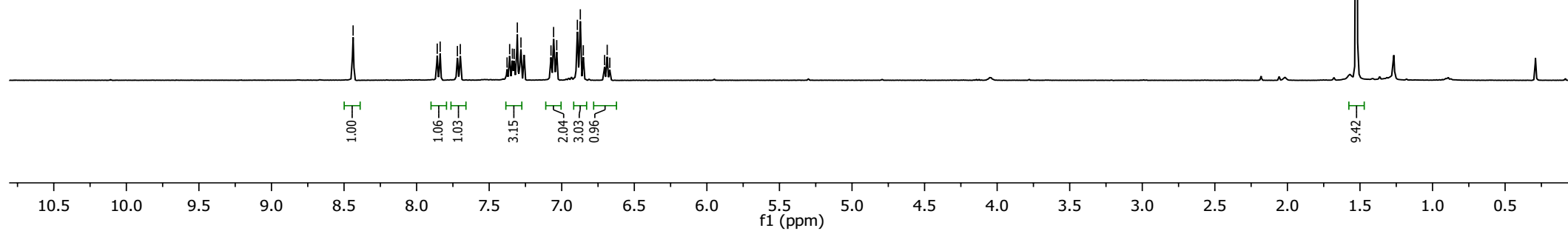
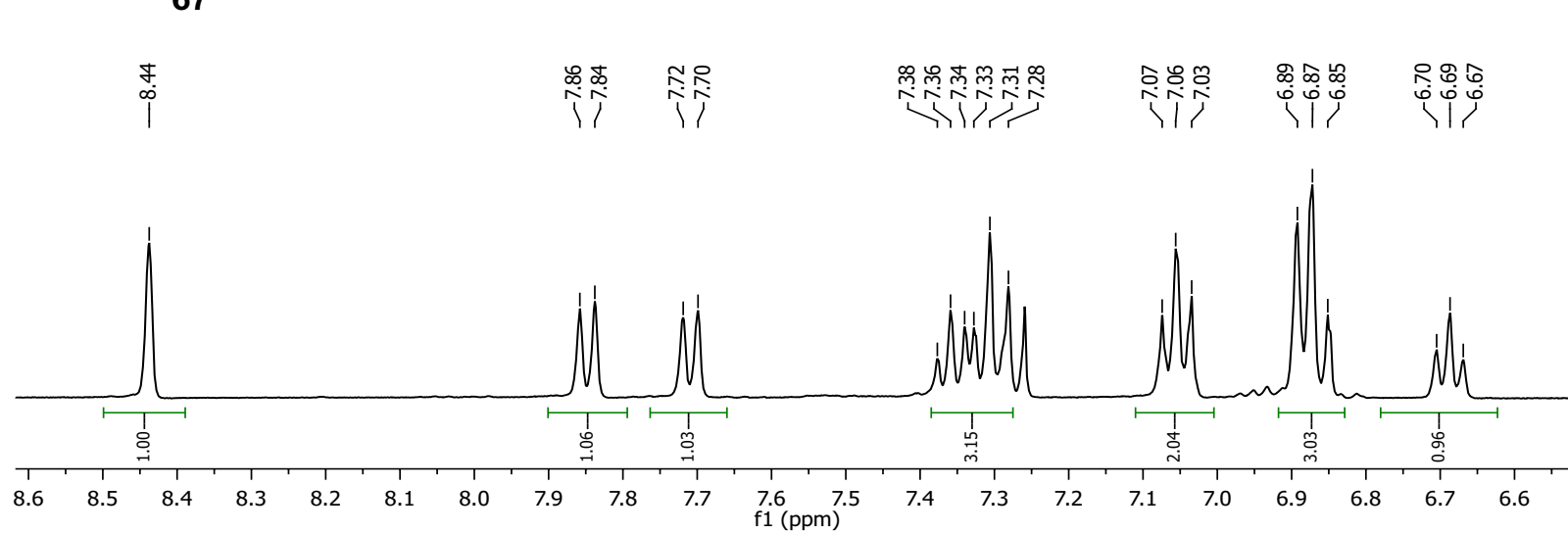
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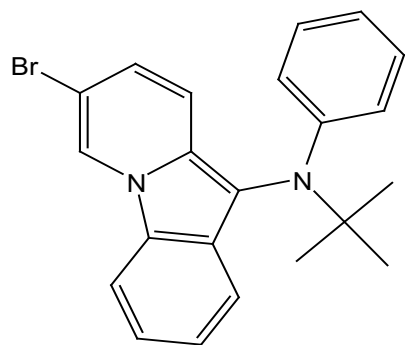
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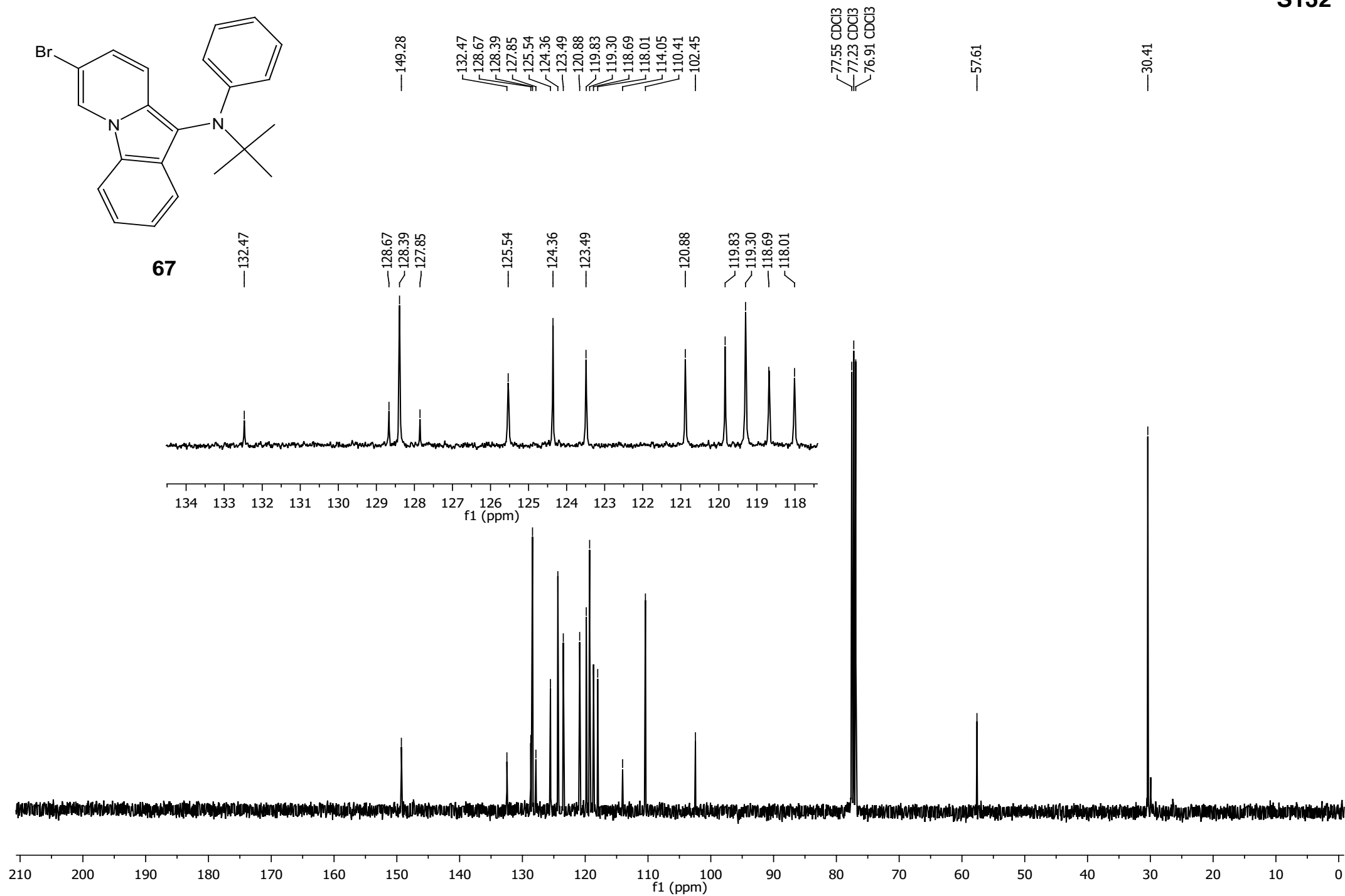
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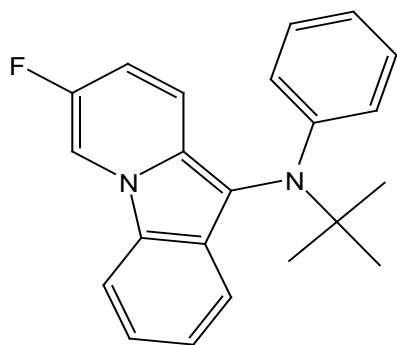


**67**



67

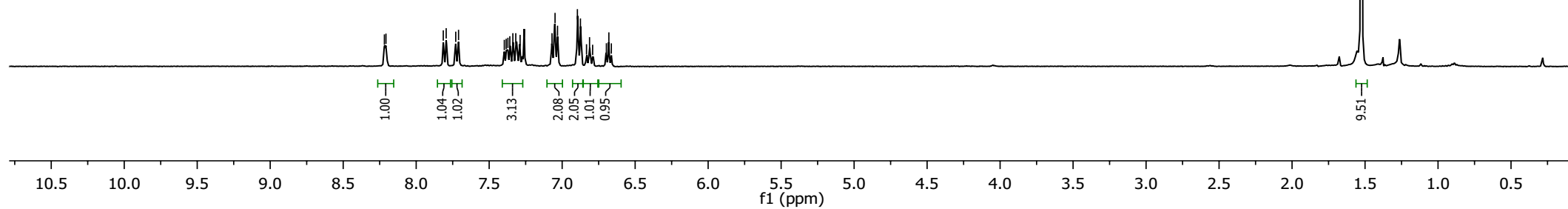
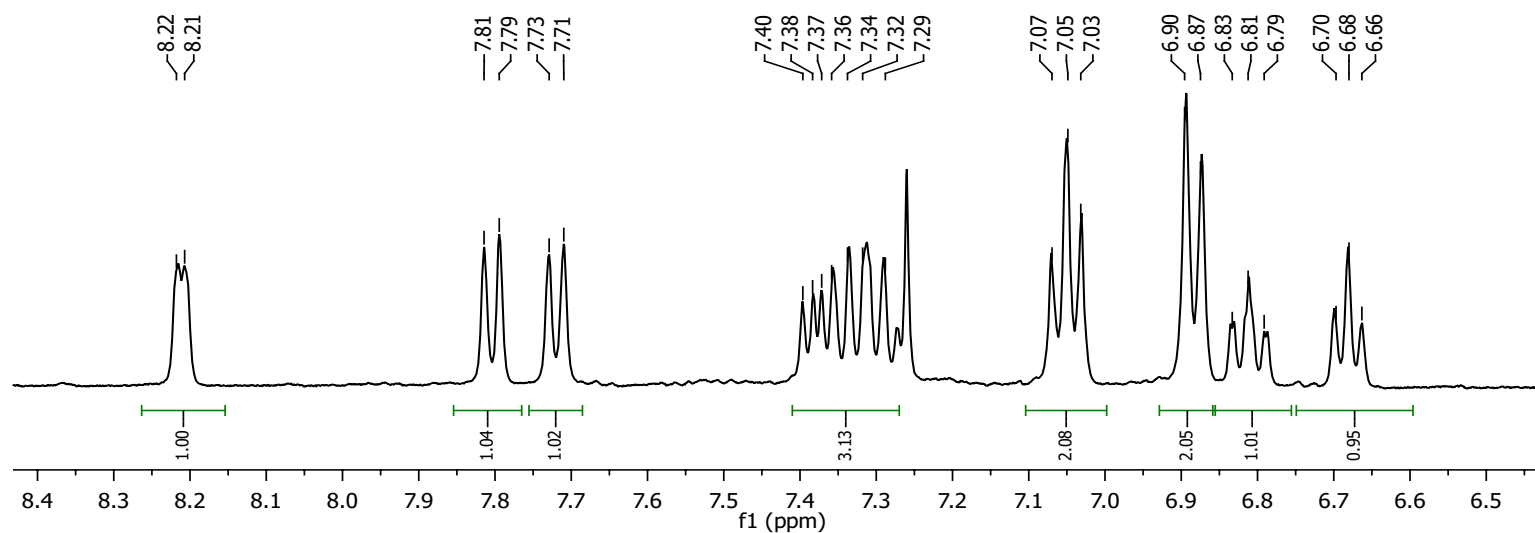


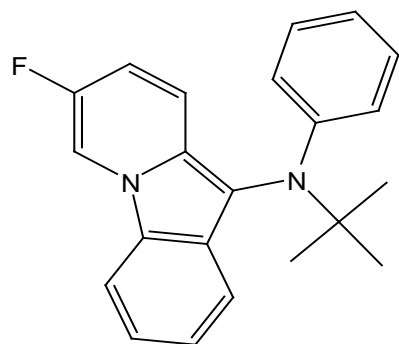


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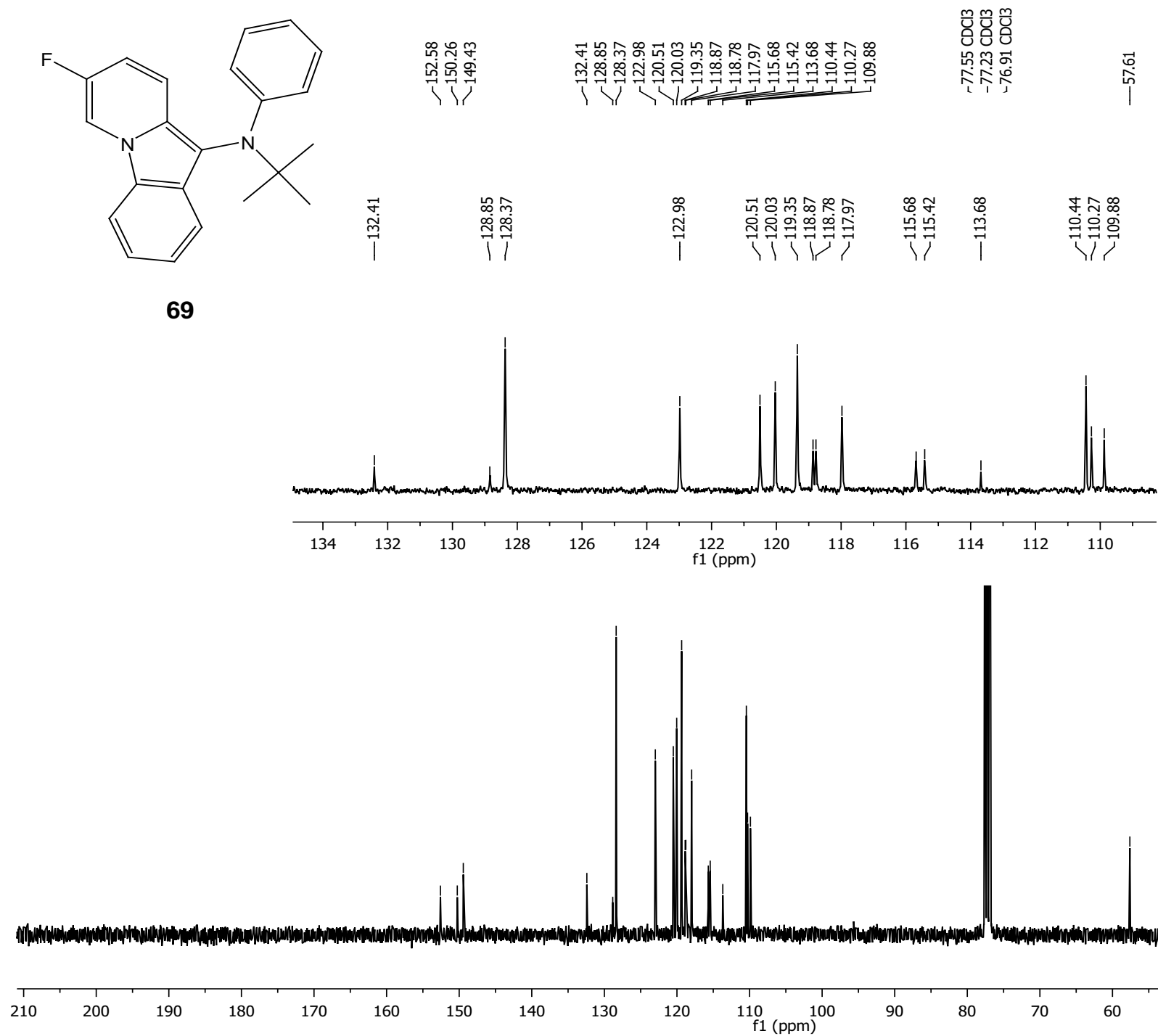
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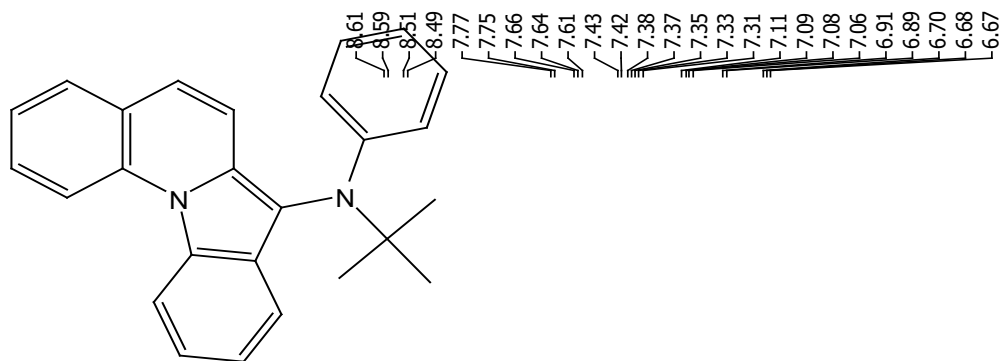
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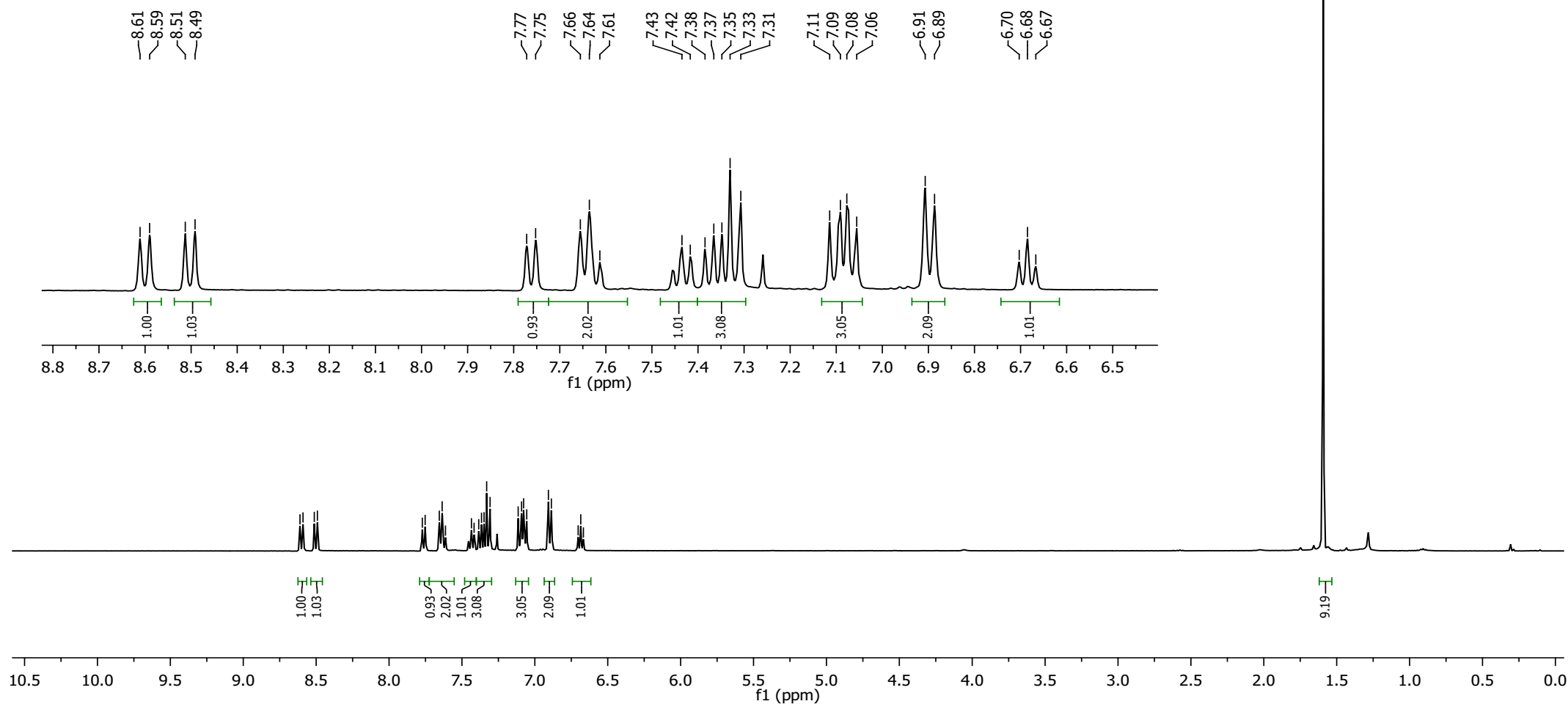


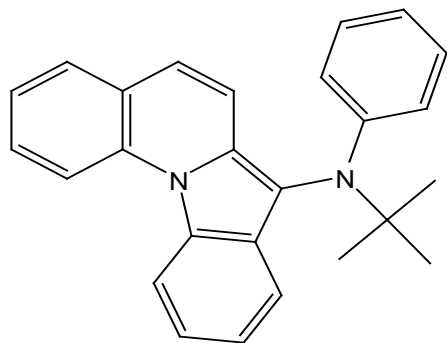
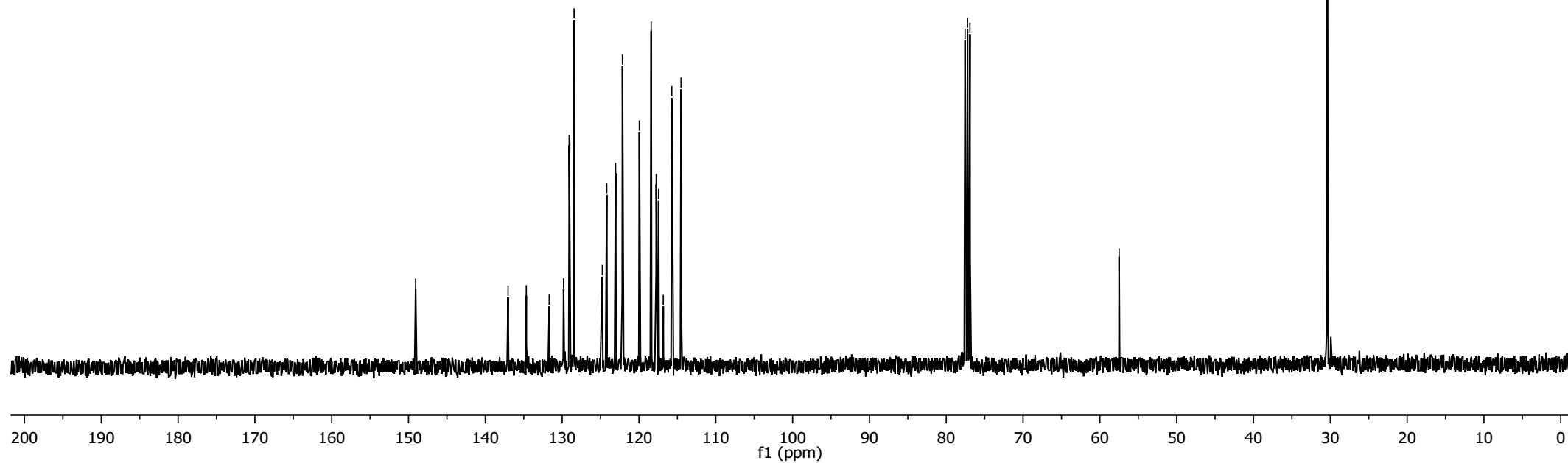
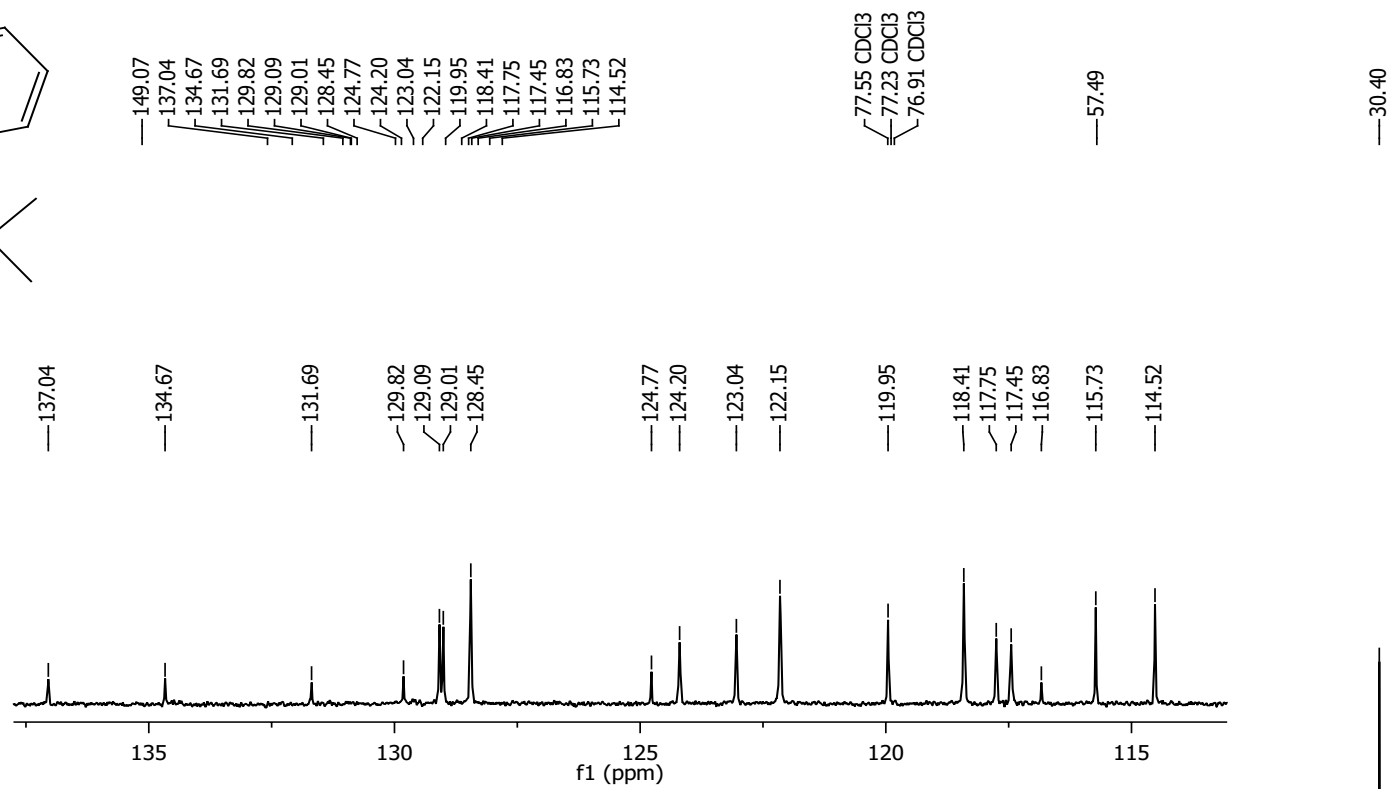
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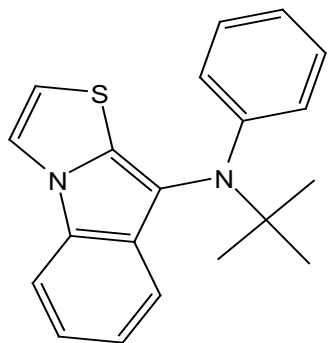




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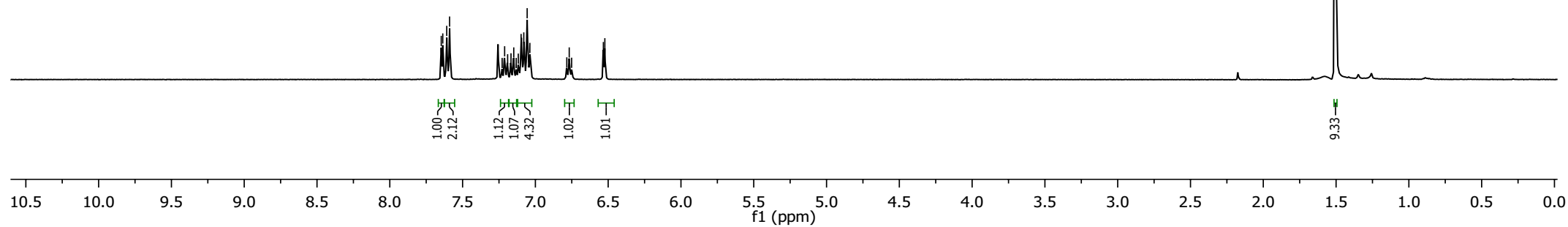
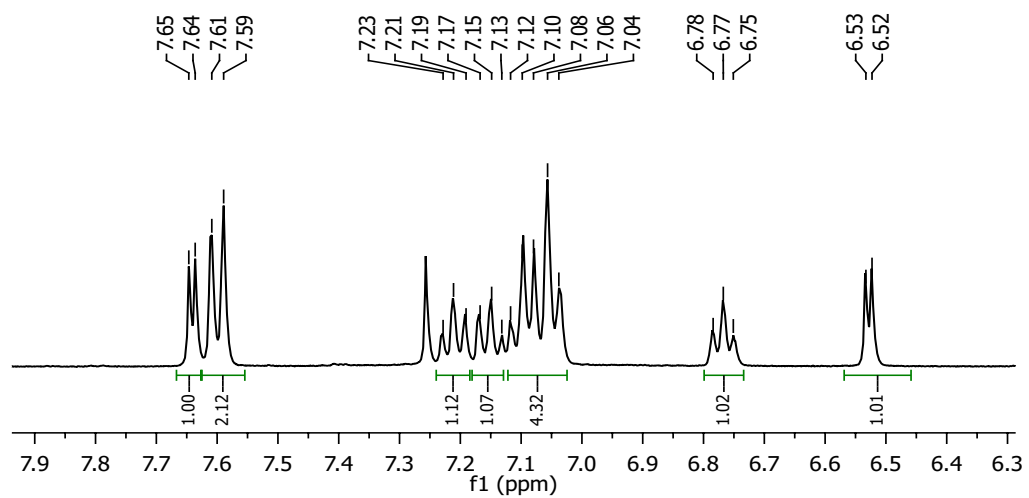
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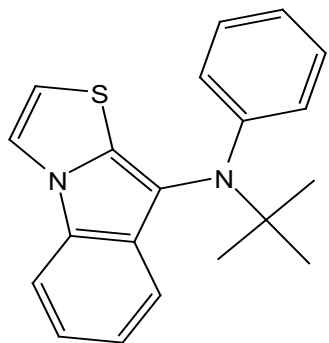


75

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7.13
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7.10
7.08
7.06
7.04
6.78
6.77
6.75
6.53
6.52

—1.51



**75**

—148.66

—136.27

—132.24

—128.37

—128.17

—121.75

—121.52

—119.56

—119.12

—118.74

—114.88

—110.30

—108.79

—108.74

77.55 CDCl₃
77.23 CDCl₃
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—58.42

—30.46

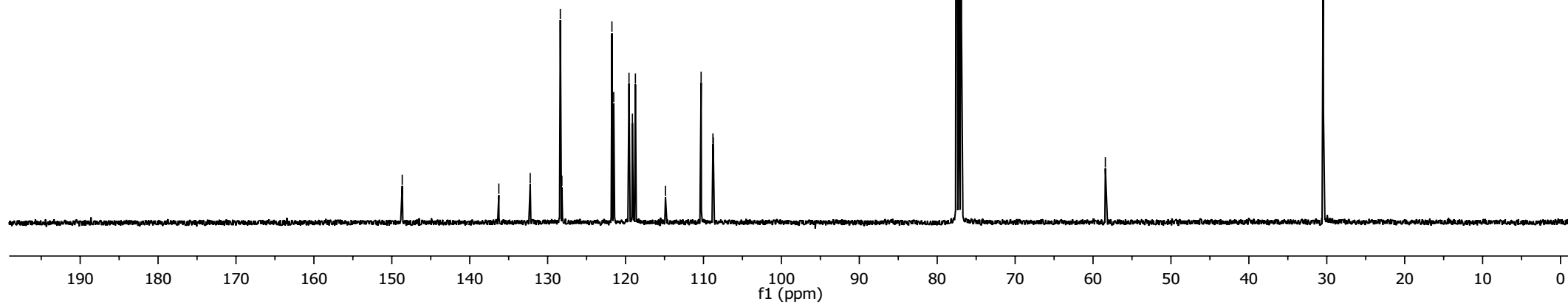
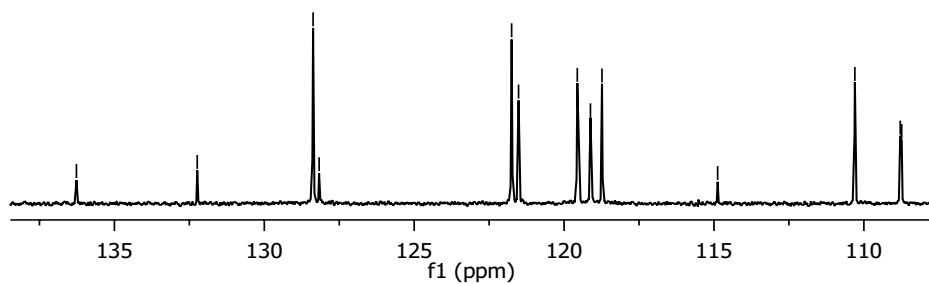
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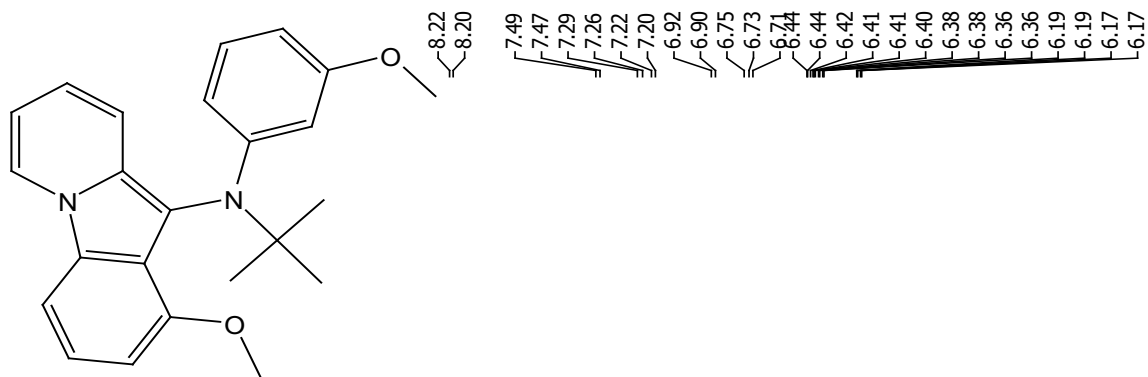
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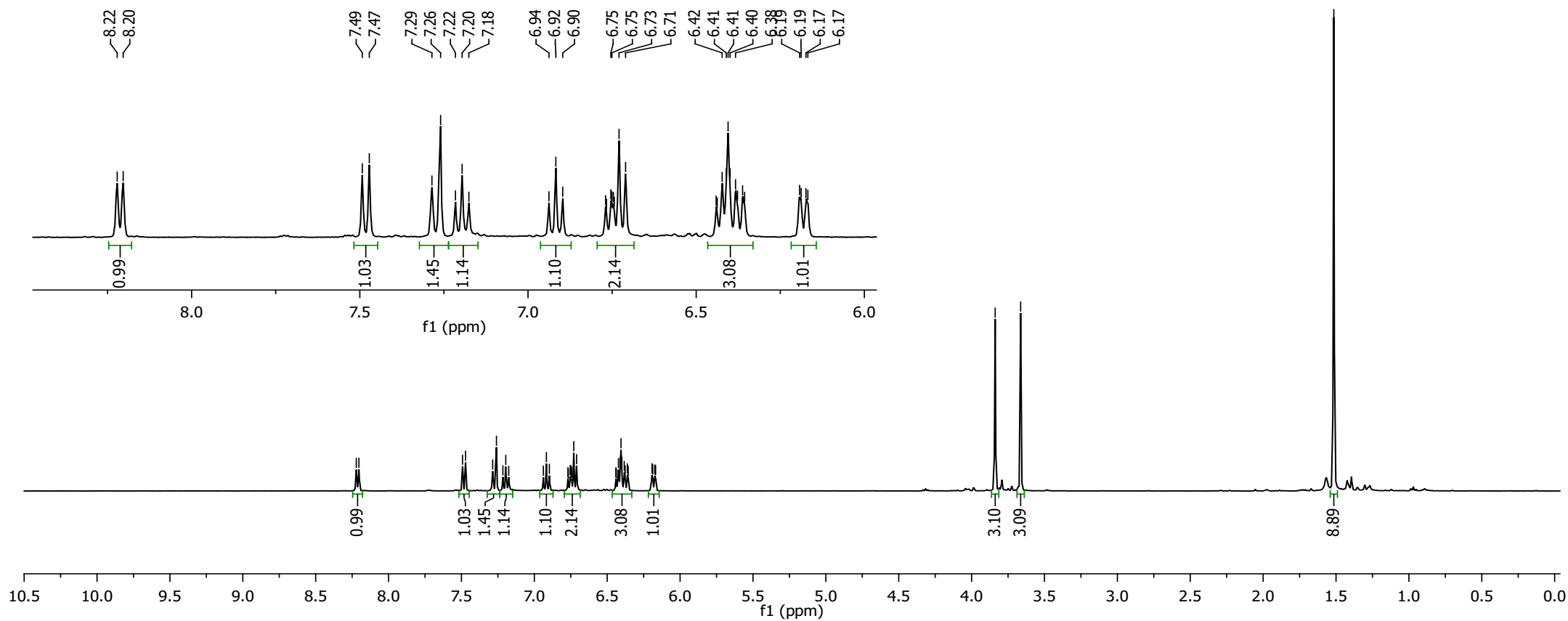
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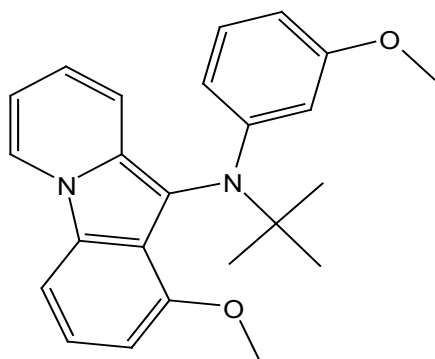
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77





77

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77.55 CDCl₃
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29.96

132.98

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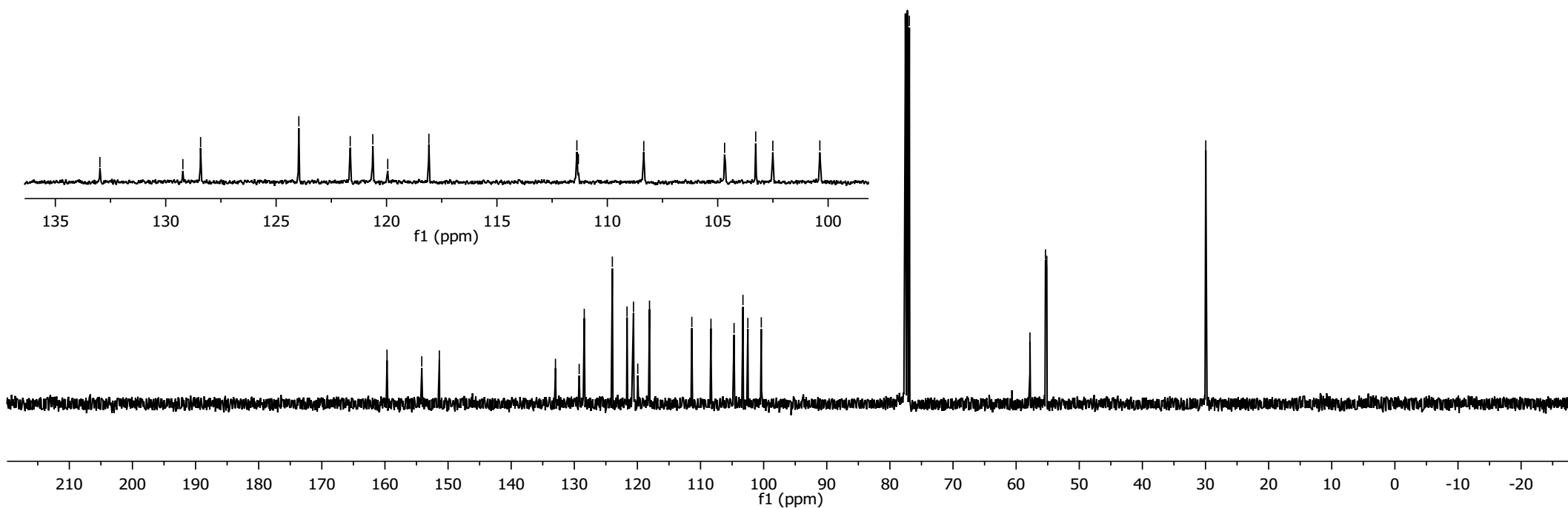
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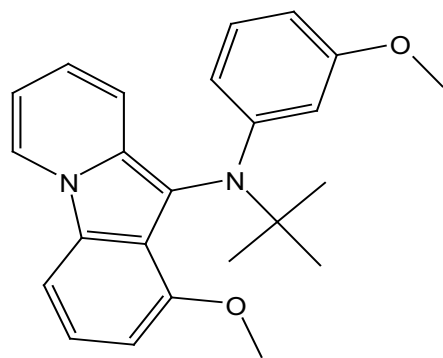
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111.31

108.35

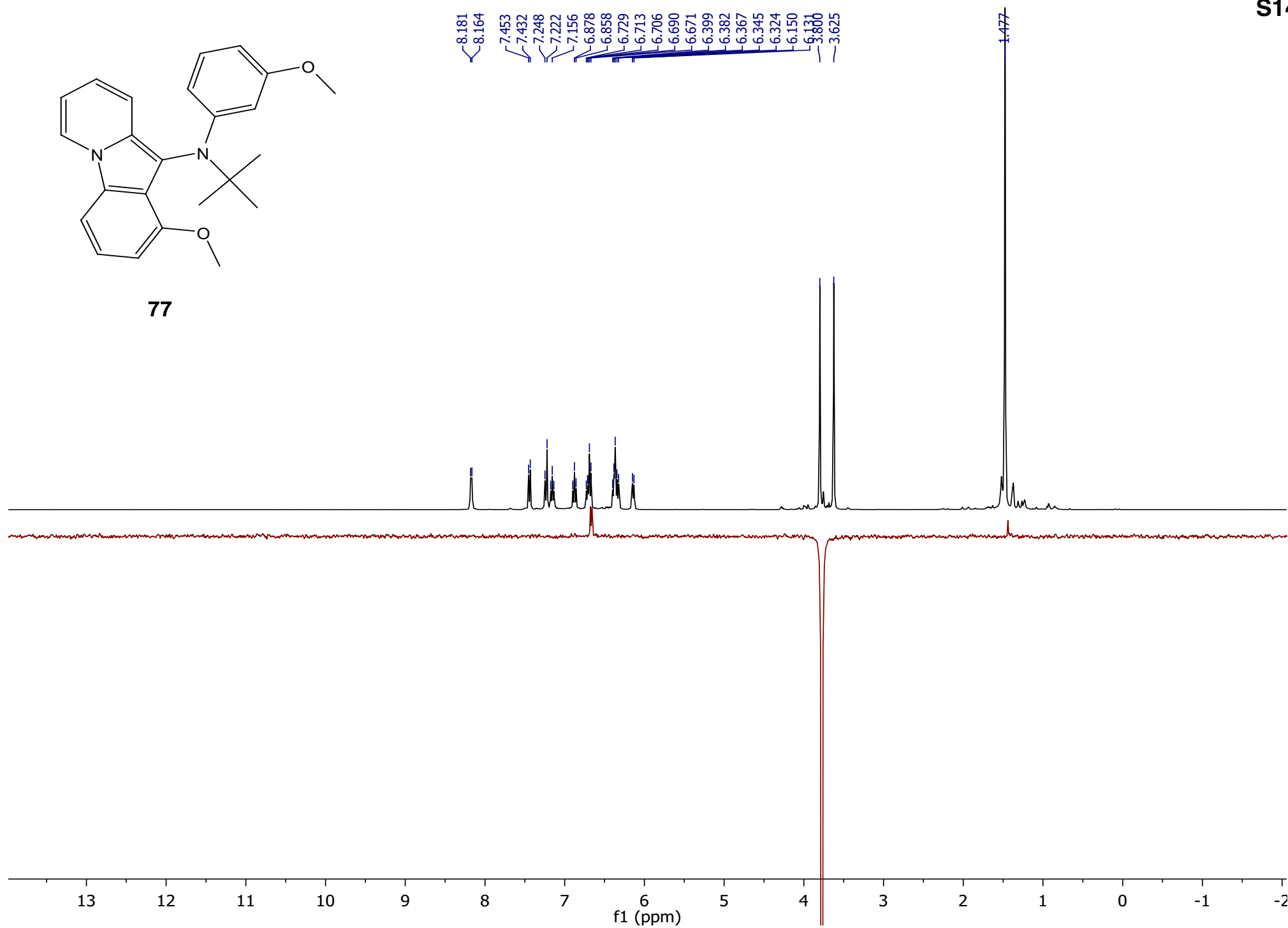
104.69
103.28
102.50

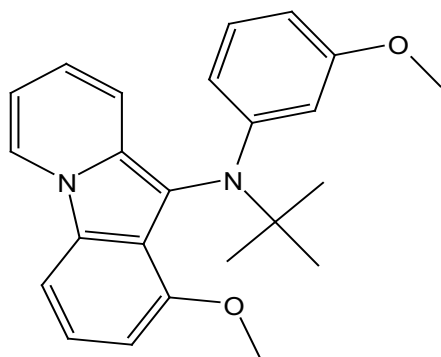
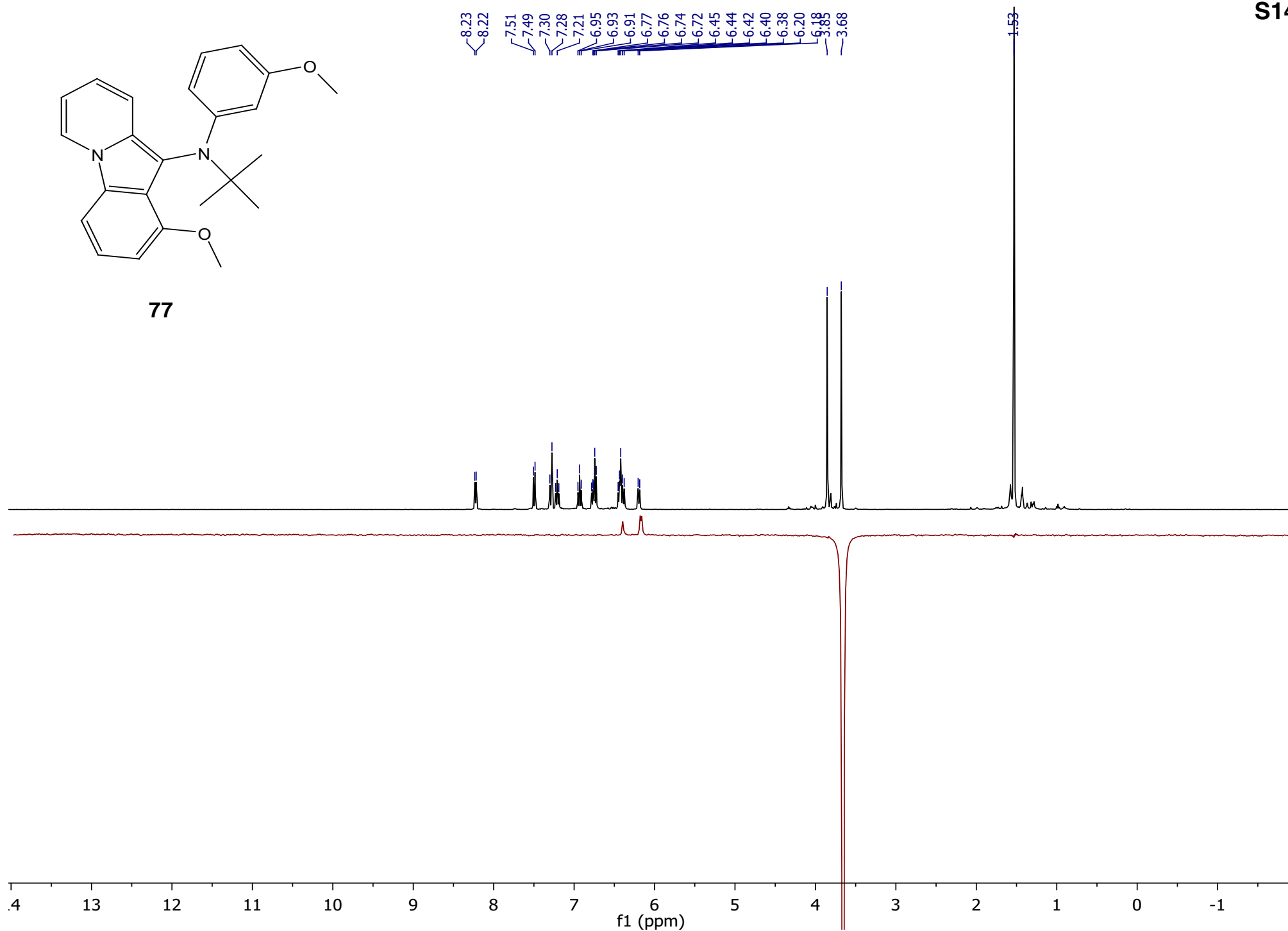
100.37

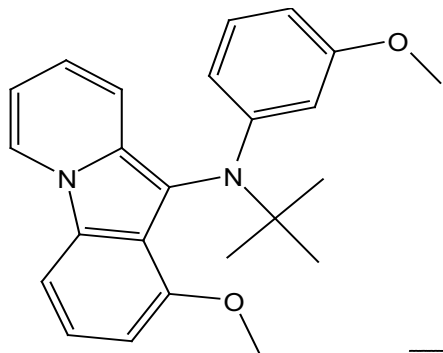




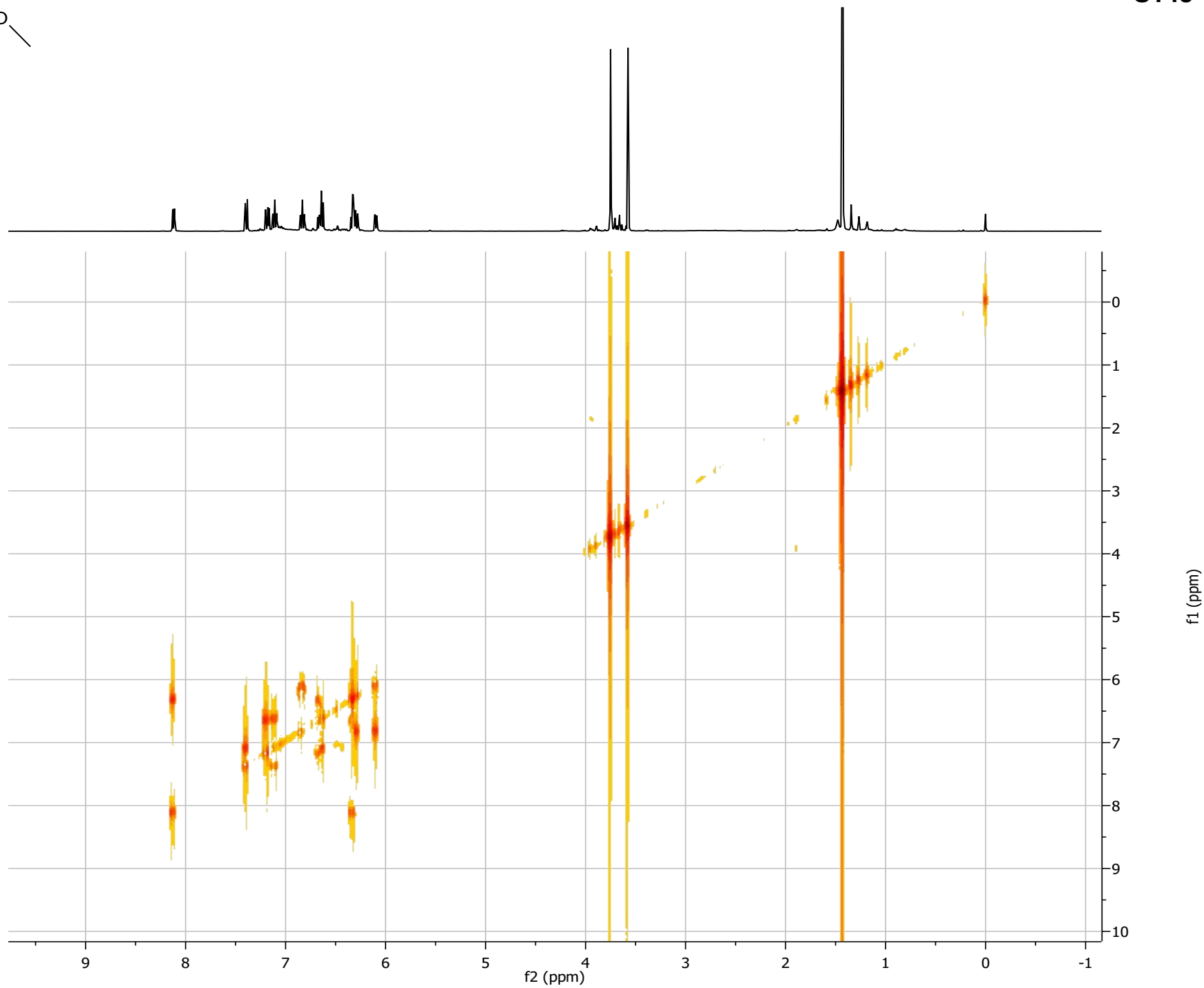
77

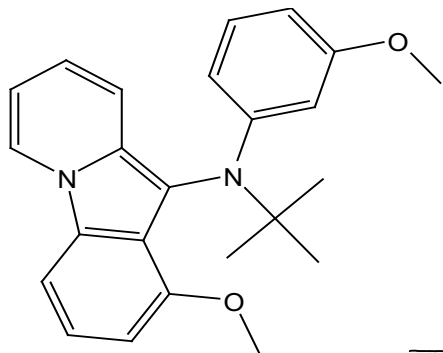


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