

## Supplementary Information

### Nucleophilic Sulfur Controlled Efficient Ketothioamide Synthesis from Tribromomethyl Carbinols

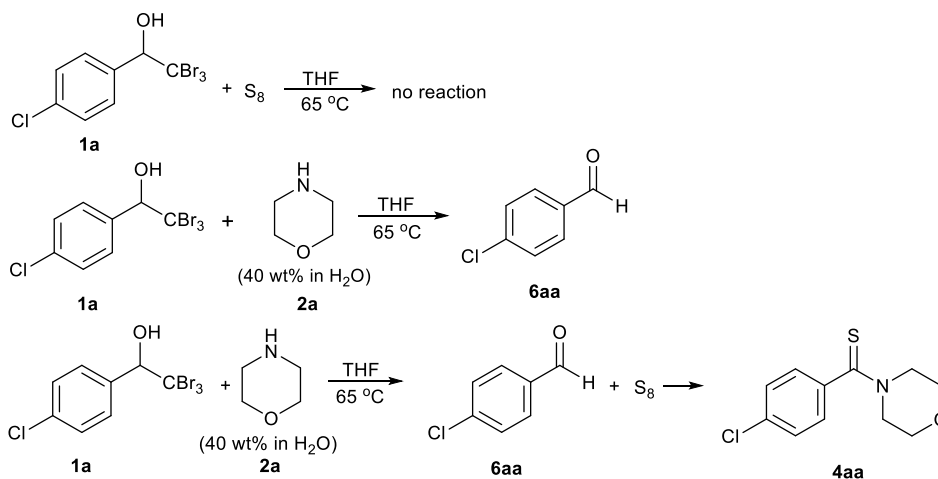
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[gururaja.n@cug.ac.in](mailto:gururaja.n@cug.ac.in)

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## 1. Control experiments



**Experiment no. 1:** The tribromomethyl carbinol **1a** (0.5 mmol, 1.0 equiv) dissolved in THF (1.0 mL) was added to elemental sulfur (2.0 mmol, 4.0 equiv) in the round bottom flask equipped with a reflux condenser. The resulting reaction mixture was stirred at a temp of 65 °C.

**Inference:** No reaction.

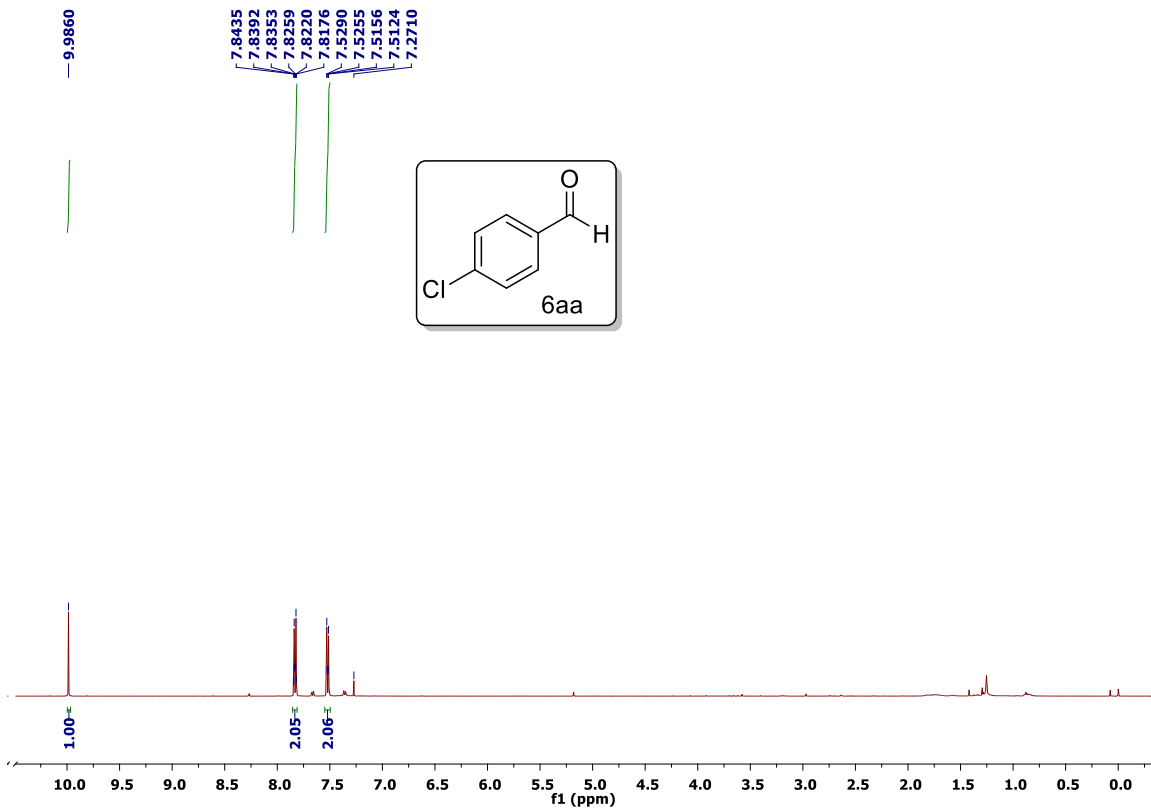
**Experiment no. 2:** The tribromomethyl carbinol **1a** (0.5 mmol, 1.0 equiv) dissolved in THF (1.0 mL) was added to amine **2a** (40 wt% in water) (10.0 mmol, 20.0 equiv) in the round bottom flask equipped with a reflux condenser. The resulting reaction mixture was stirred at a temp of 65 °C.

**Inference:** Formation of aldehyde and was separated as an intermediate **6aa**.

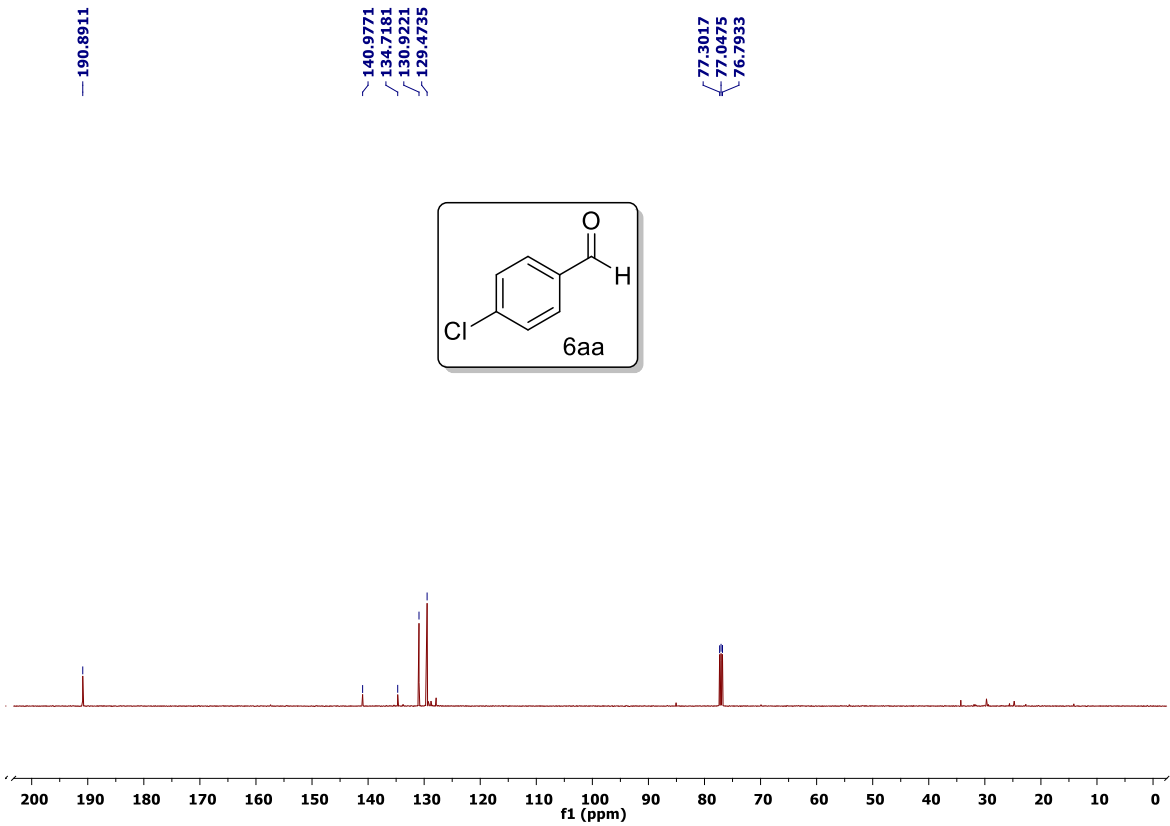
**Experiment no. 3:** The tribromomethyl carbinol **1a** (0.5 mmol, 1.0 equiv) dissolved in THF (1.0 mL) was added to amine **2a** (40 wt% in water) (10.0 mmol, 20.0 equiv) in the round bottom flask equipped with a reflux condenser. The resulting reaction mixture was stirred at a temp of 65 °C. After one hour of reaction elemental sulfur (2.0 mmol) was added.

**Inference:** Formation of thioamide **4aa**.

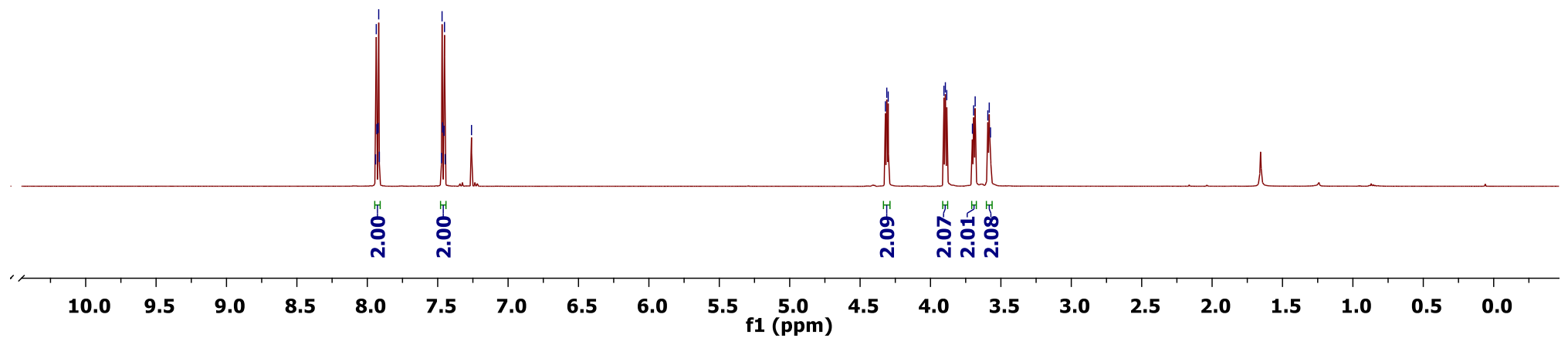
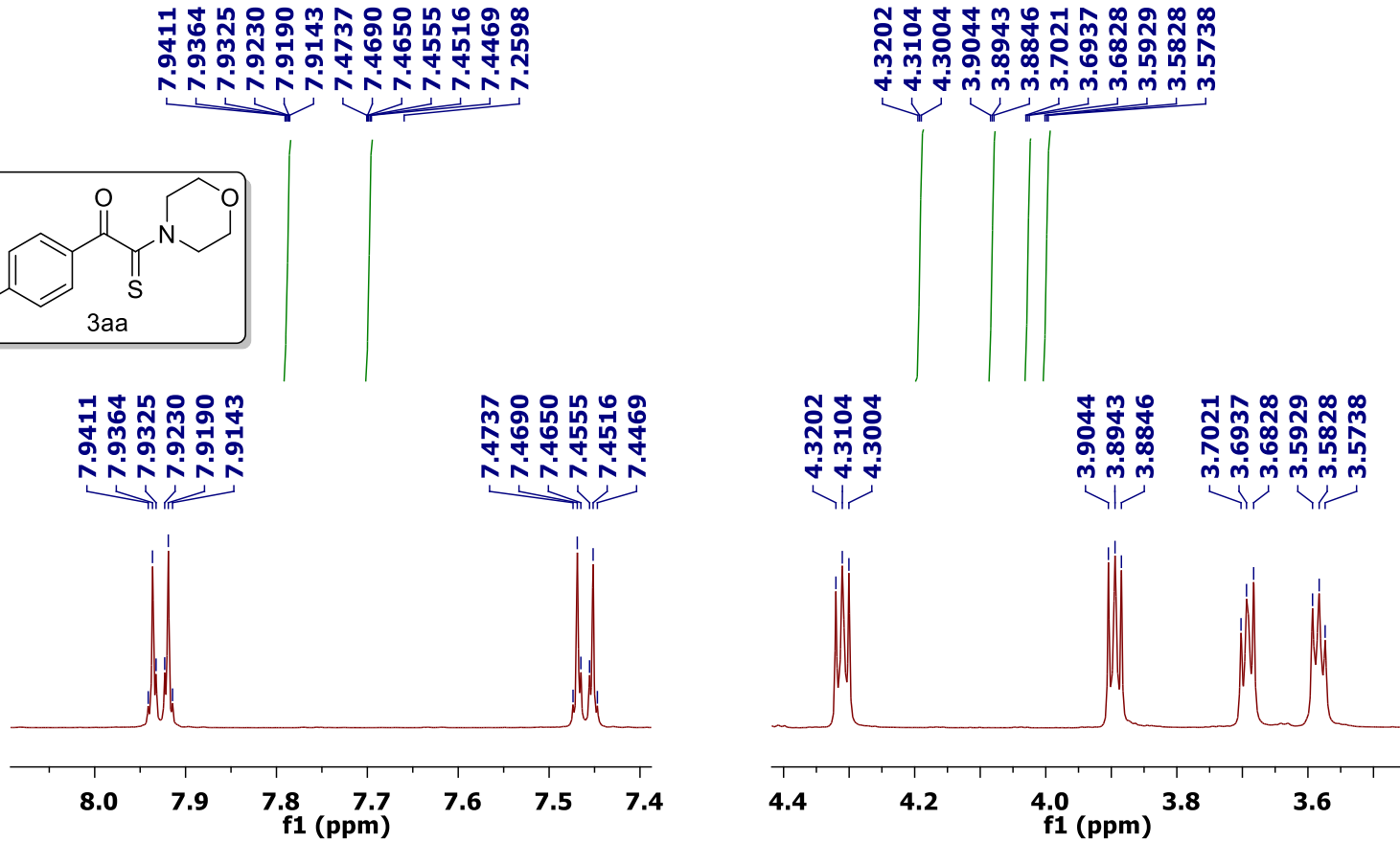
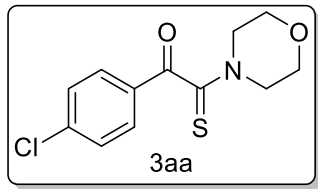
**$^1\text{H}$  NMR for compound 6aa (500 MHz,  $\text{CDCl}_3$ )**



**$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 6aa (125 MHz,  $\text{CDCl}_3$ )**



<sup>1</sup>H NMR for compound 3aa (500 MHz, CDCl<sub>3</sub>)





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3aa (125 MHz,  $\text{CDCl}_3$ )

— 195.0293

— 186.5357

— 141.1324

{ 131.8416

{ 131.2989

{ 129.4373

{ 77.4108

{ 77.1566

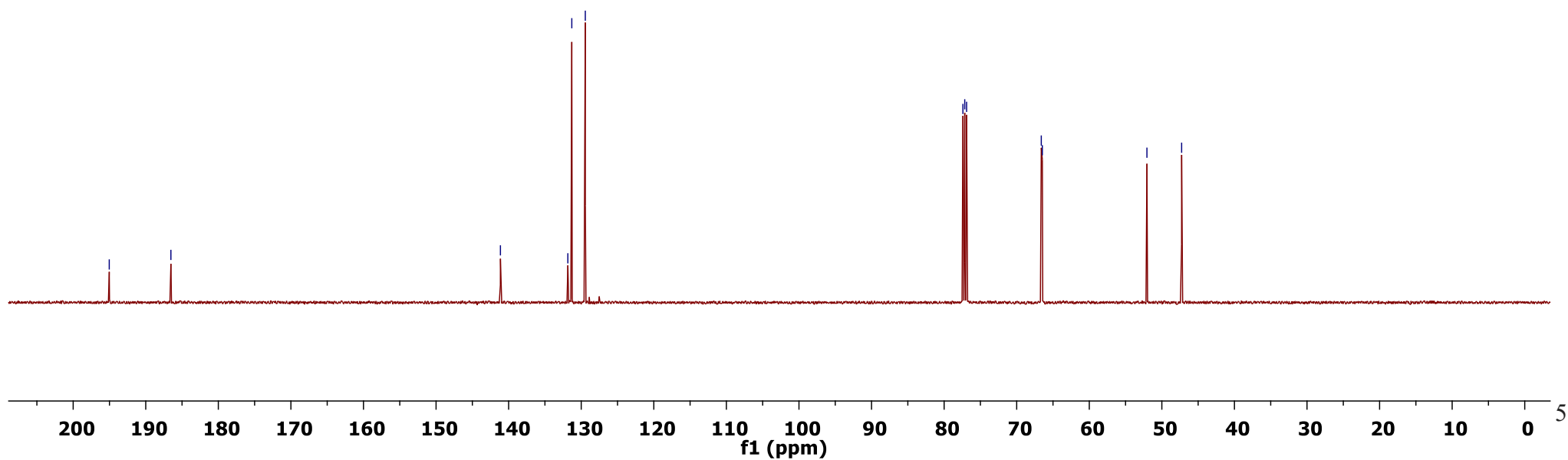
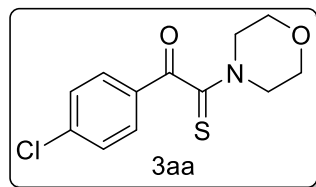
{ 76.9024

{ 66.6135

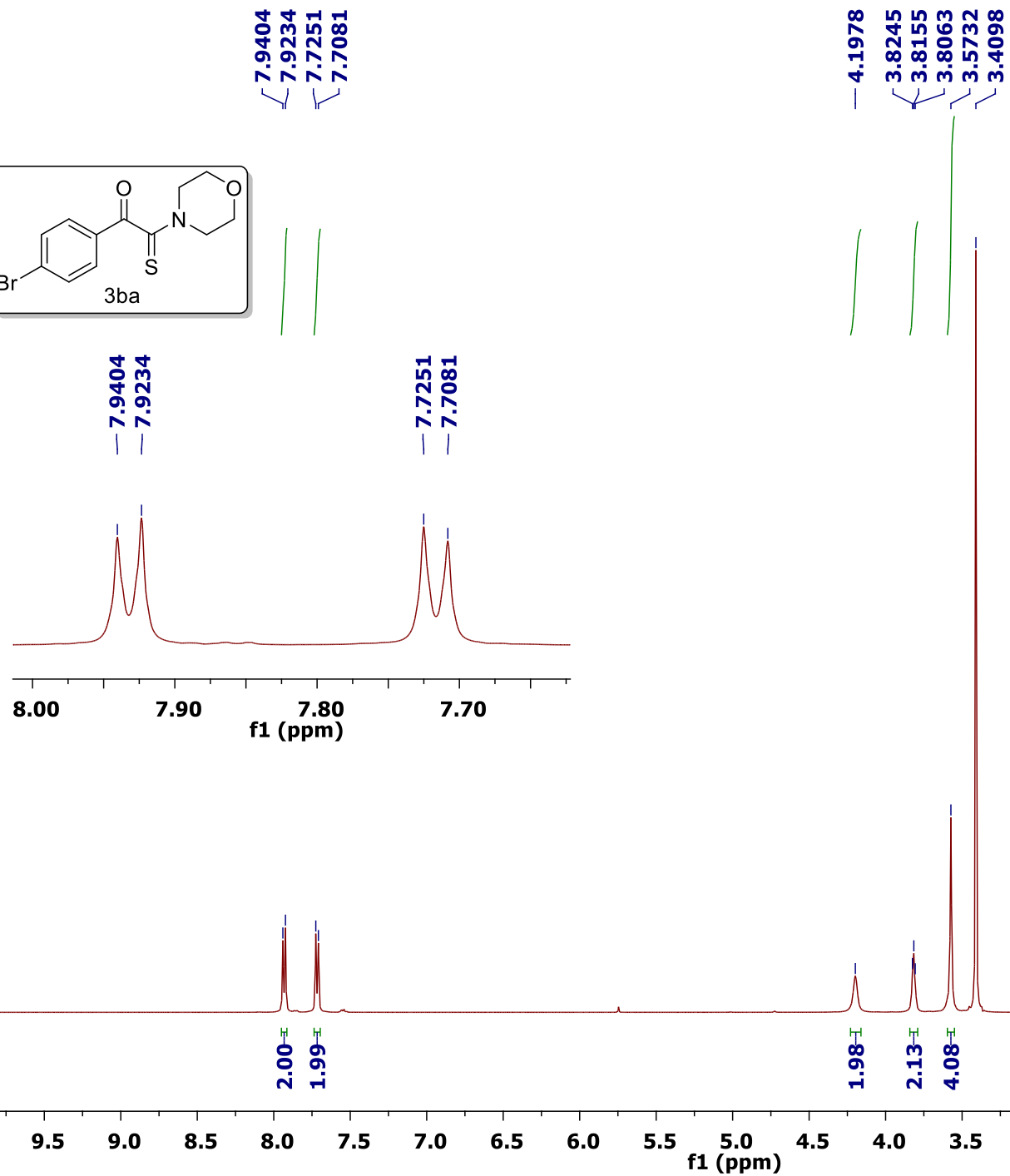
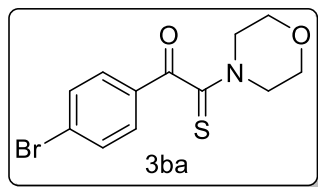
{ 66.4660

— 52.0548

— 47.2746



<sup>1</sup>H NMR for compound 3ba (500 MHz, DMSO-d<sub>6</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ba (125 MHz, DMSO- $d_6$ )

— 193.7217

— 186.5764

— 143.2930

— 132.2496

— 130.9818

— 126.6258

— 66.3486

— 65.9988

— 52.3248

— 47.4252

— 40.4460

— 40.2789

— 40.2014

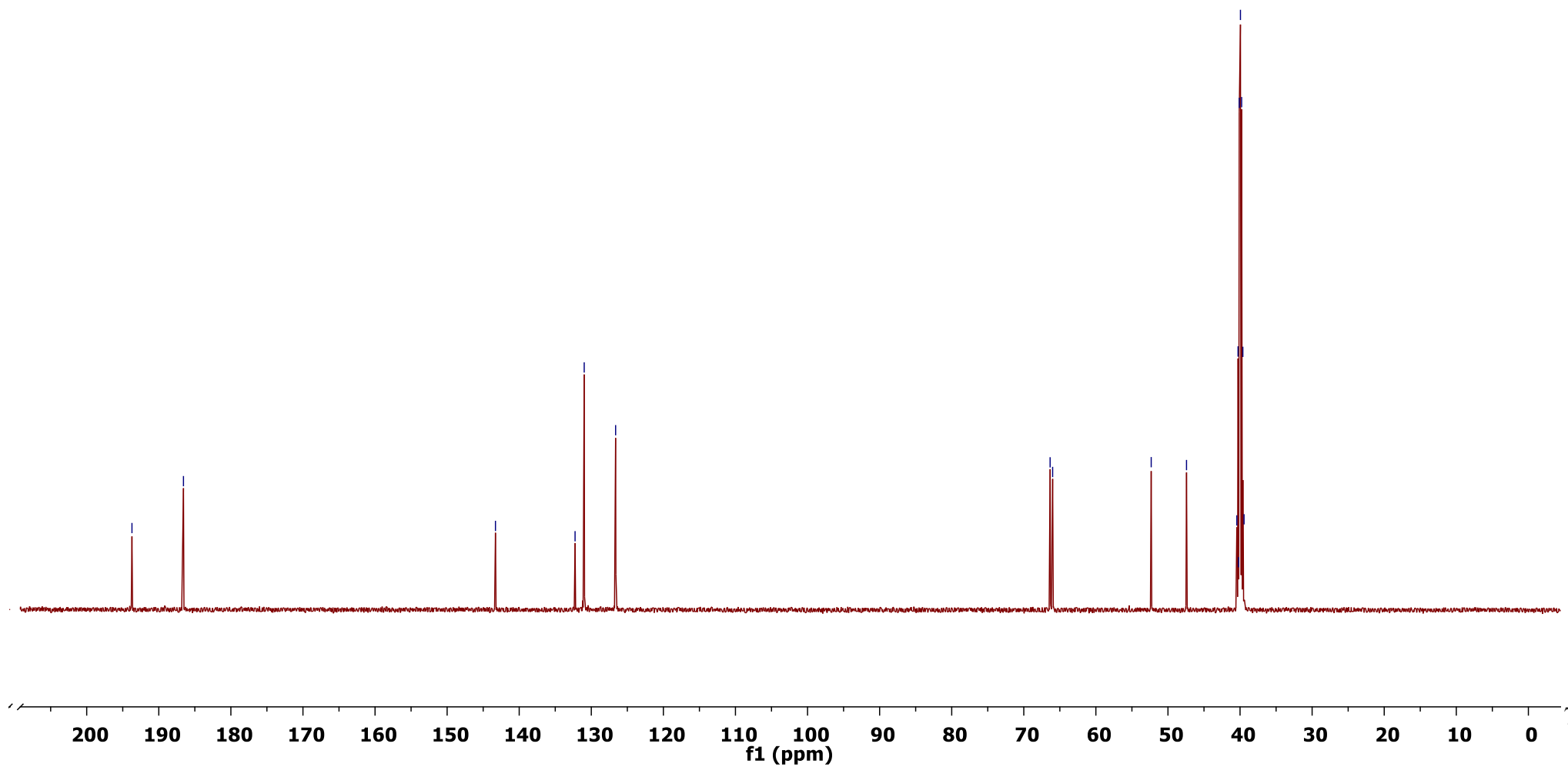
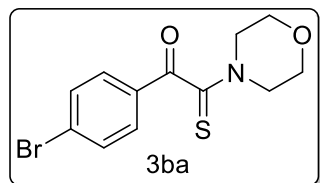
— 40.1120

— 39.9450

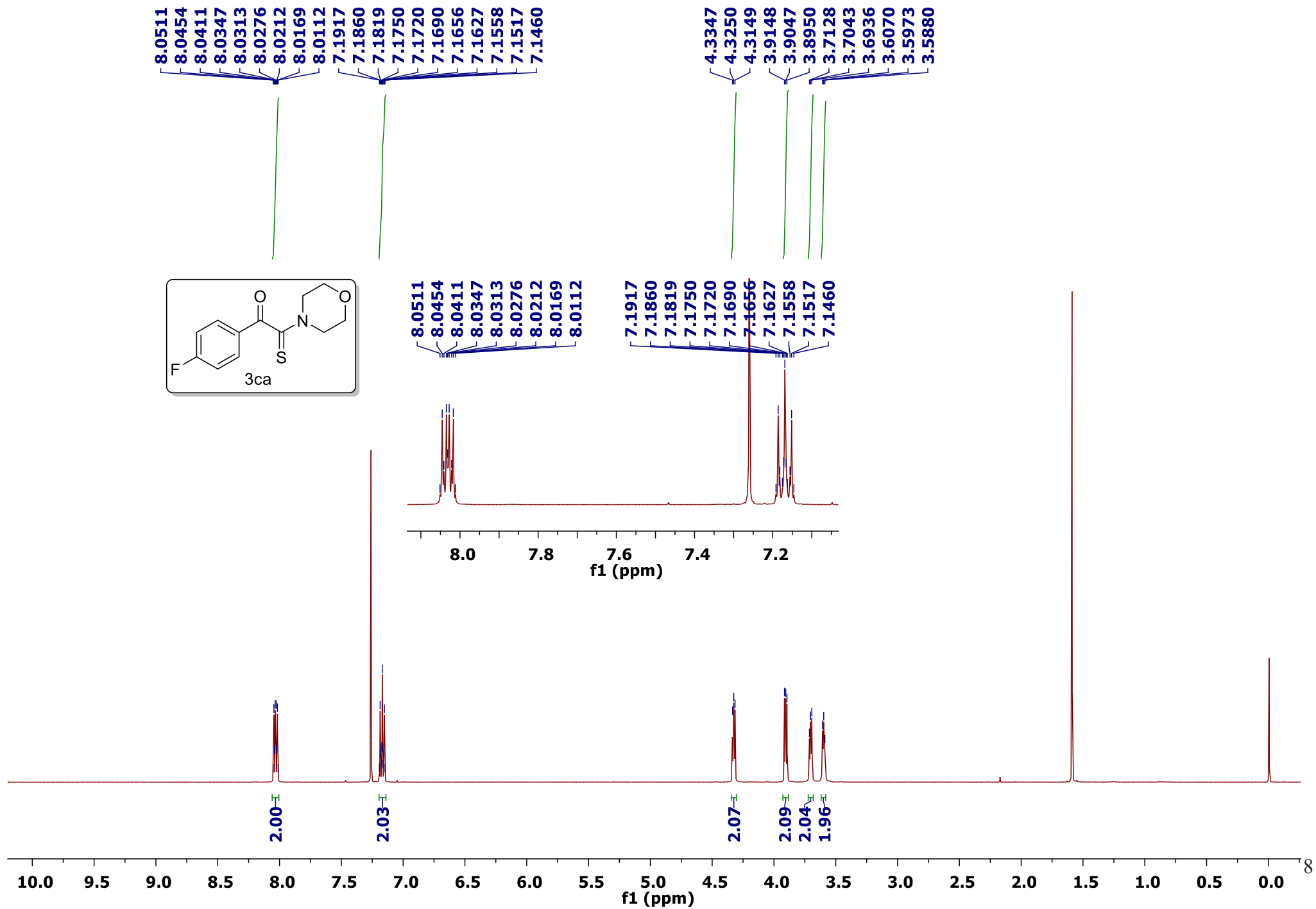
— 39.7780

— 39.6113

— 39.4445



<sup>1</sup>H NMR for compound 3ca (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ca (125 MHz,  $\text{CDCl}_3$ )

— 195.3422

— 186.5066

— 167.6163

— 165.5667

— 132.8409

— 132.7635

— 129.8662

— 116.5089

— 116.3320

77.4132

77.1591

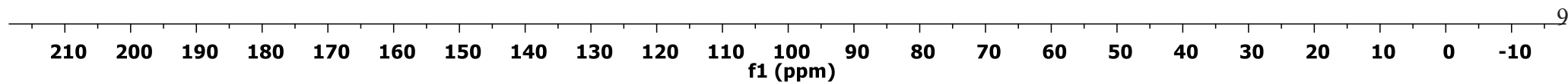
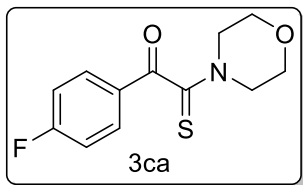
76.9050

66.6633

66.5150

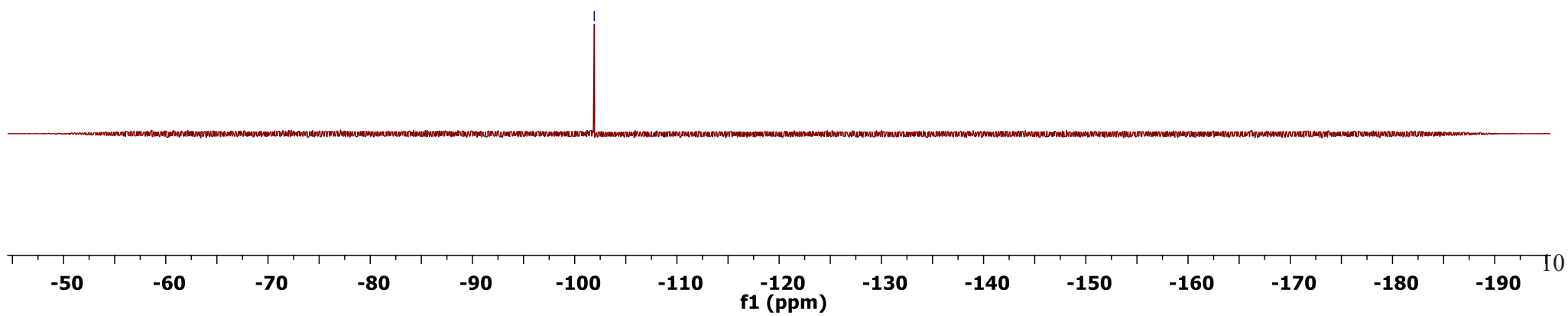
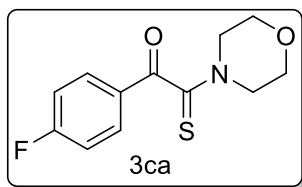
— 52.0896

— 47.3019

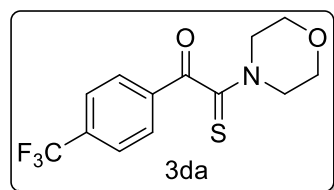


$^{19}\text{F}$  NMR for compound 3ca (470 MHz,  $\text{CDCl}_3$ )

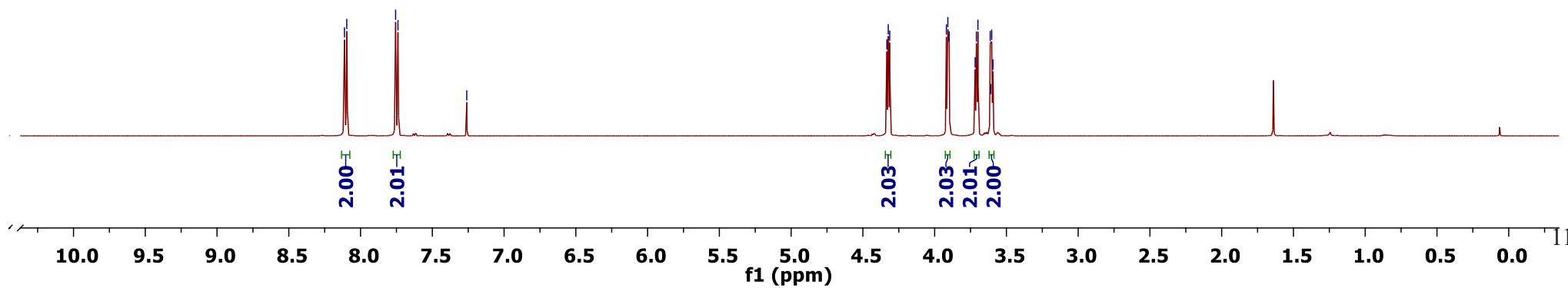
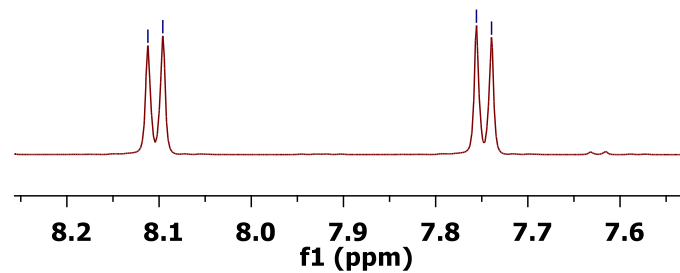
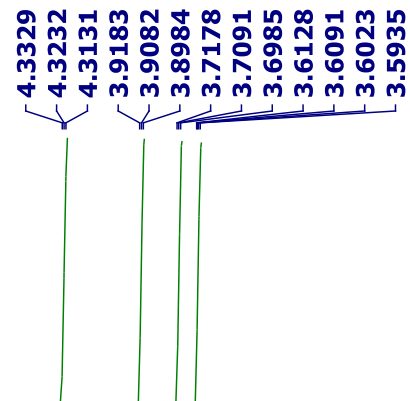
— -101.9167



<sup>1</sup>H NMR for compound 3da (500 MHz, CDCl<sub>3</sub>)



8.1121  
8.0959  
7.7558  
7.7394



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3da (125 MHz,  $\text{CDCl}_3$ )

— 194.5405

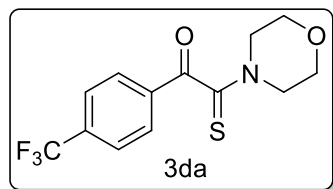
— 185.9335

136.4146  
135.8992  
135.6378  
135.3770  
135.1155  
130.2612  
128.3349  
126.7289  
126.2661  
126.1182  
126.0893  
126.0597  
126.0299  
124.5586  
122.3881  
120.2177  
119.3226

77.4141  
77.1600  
76.9057

66.6319  
66.4791

— 52.1172  
— 47.3481



— 136.4146

— 135.8992

— 135.6378

— 135.3770

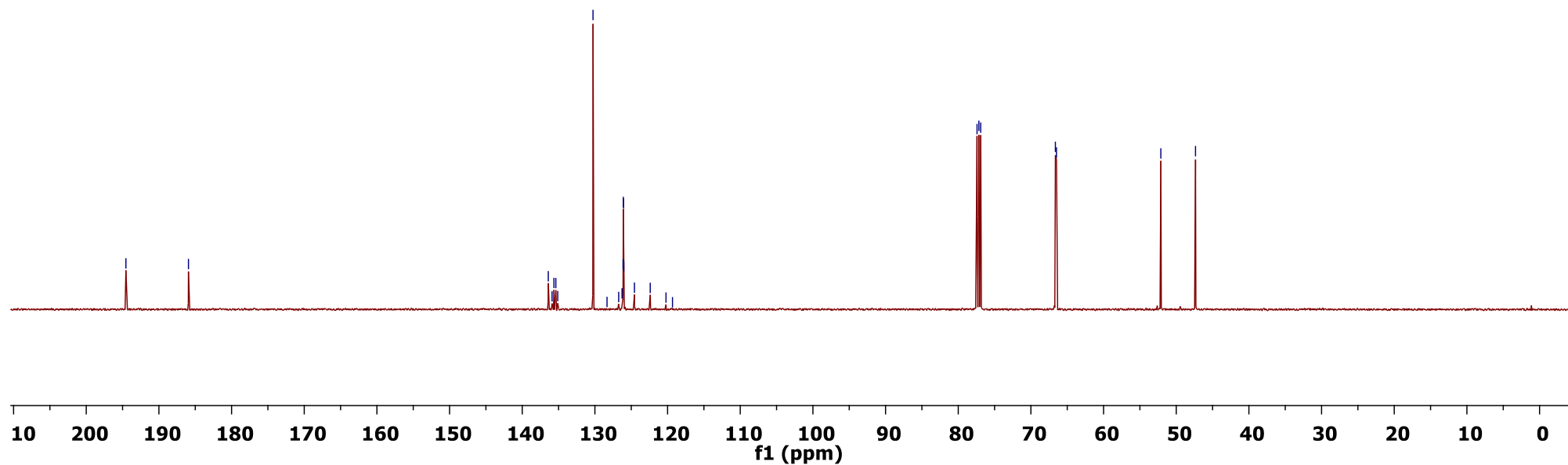
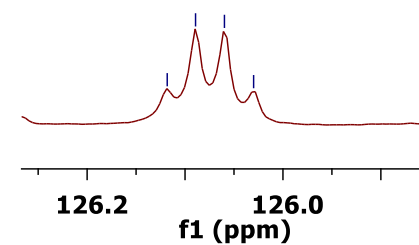
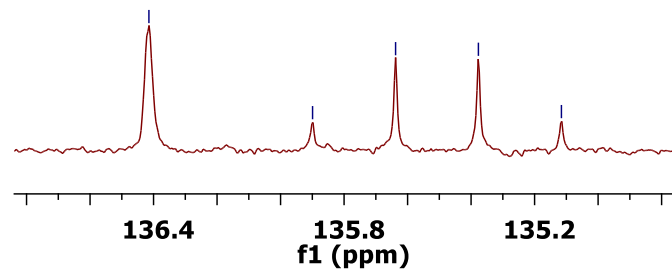
— 135.1155

— 126.1182

— 126.0893

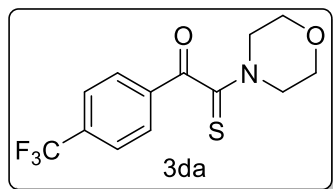
— 126.0597

— 126.0299

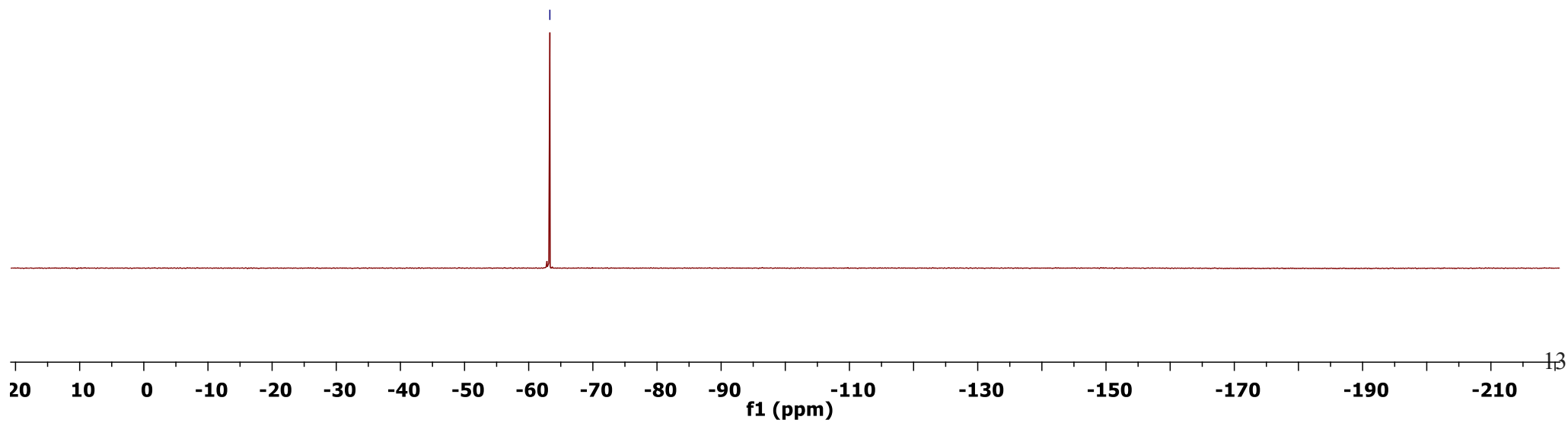




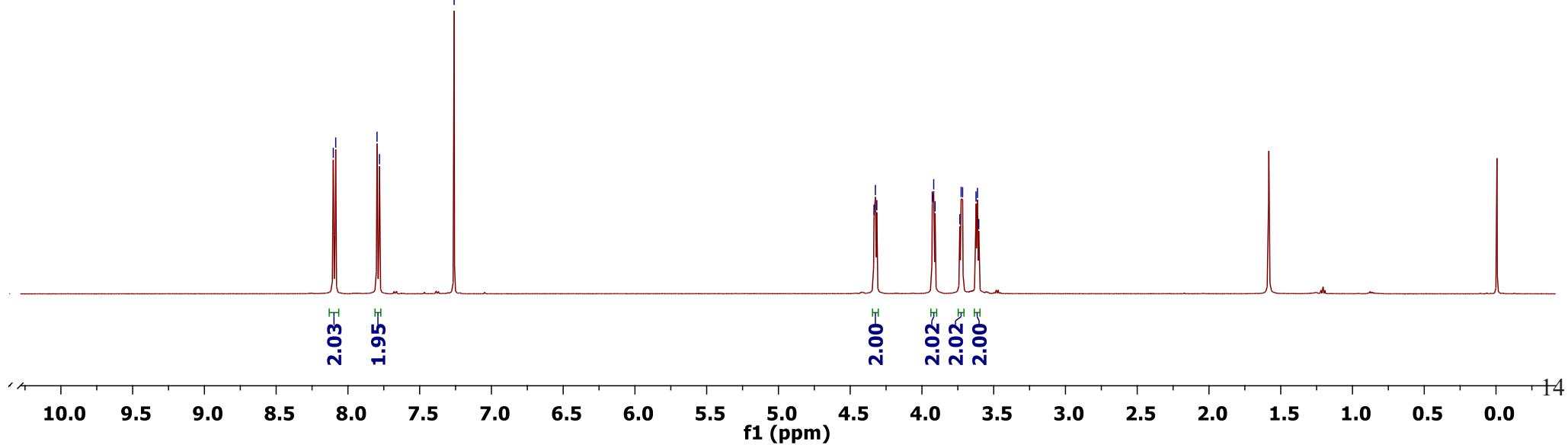
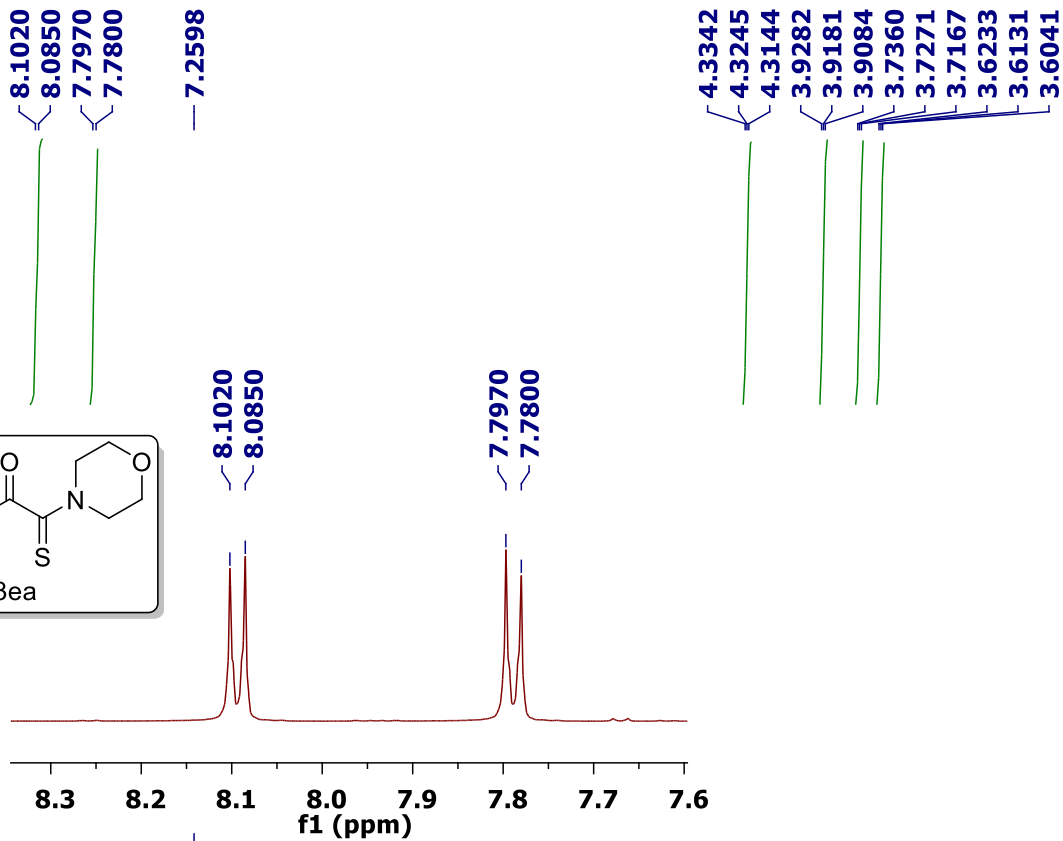
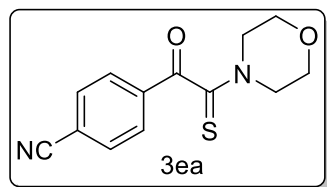
**$^{19}\text{F}$  NMR for compound 3da (470 MHz,  $\text{CDCl}_3$ )**



— -63.2986



<sup>1</sup>H NMR for compound 3ea (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ea (125 MHz,  $\text{CDCl}_3$ )

— 193.9704

— 185.0804

— 136.9546

— 132.7641

— 130.2714

— 117.7900

— 117.4525

77.4128

77.1587

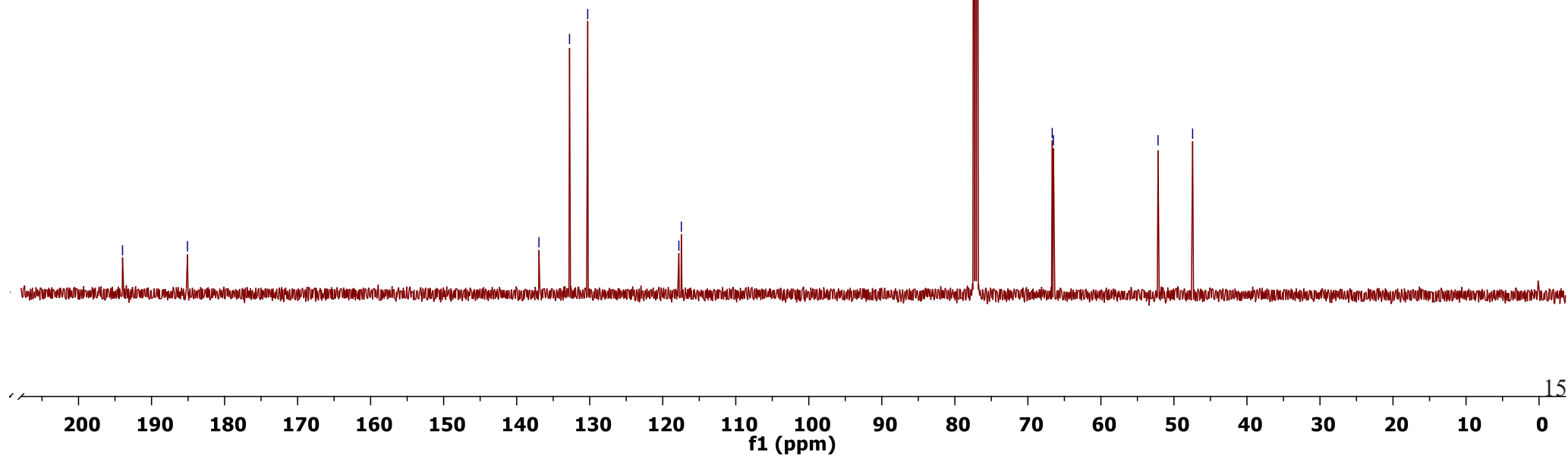
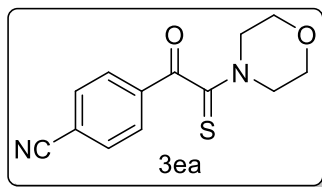
76.9046

66.6677

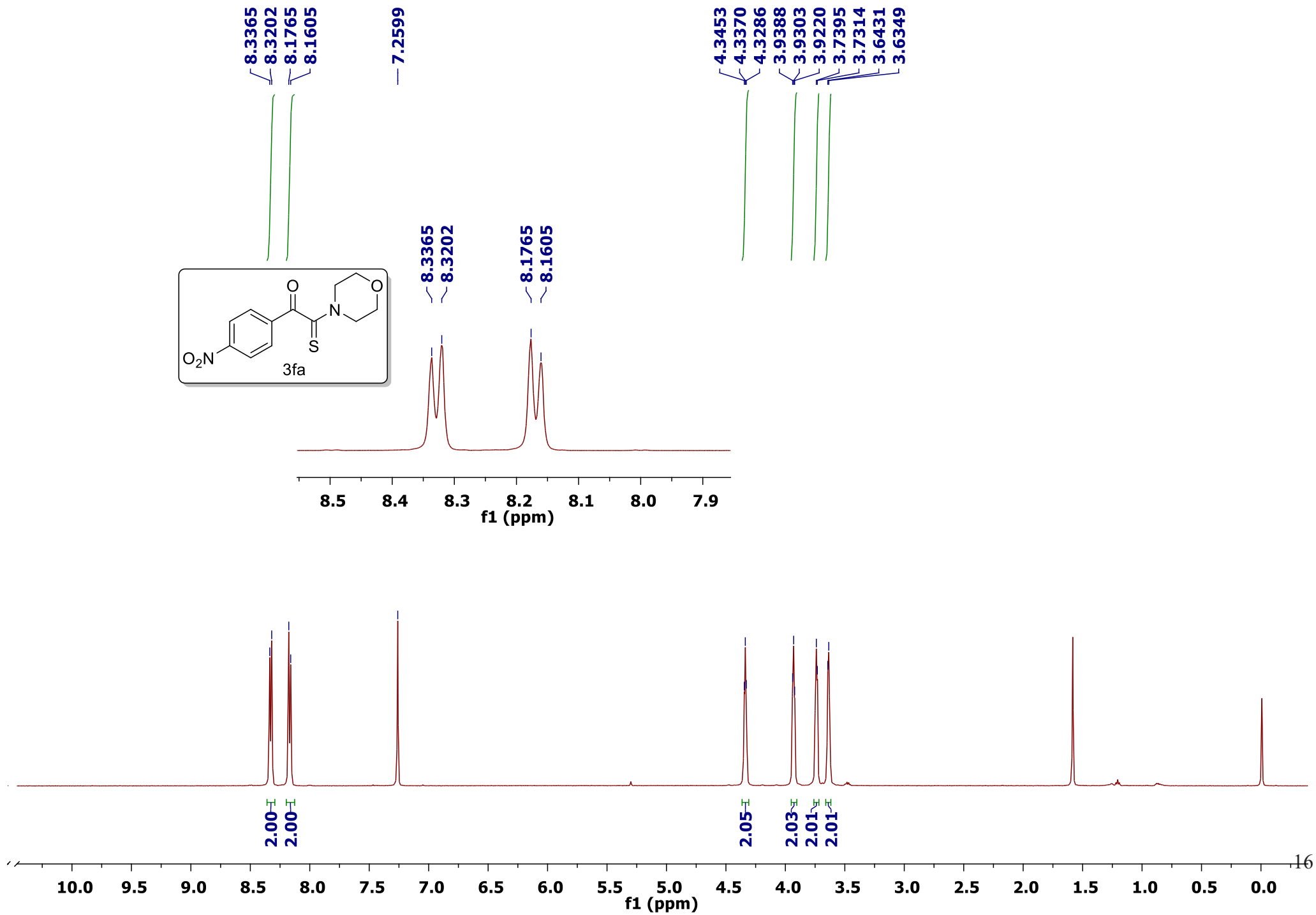
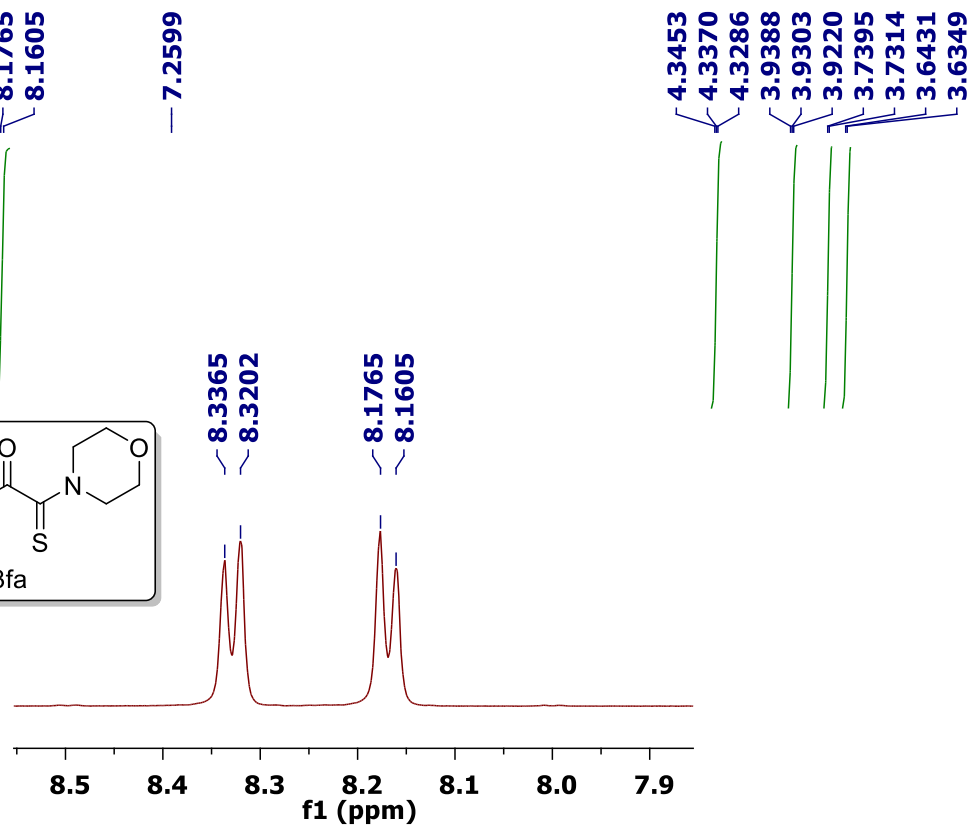
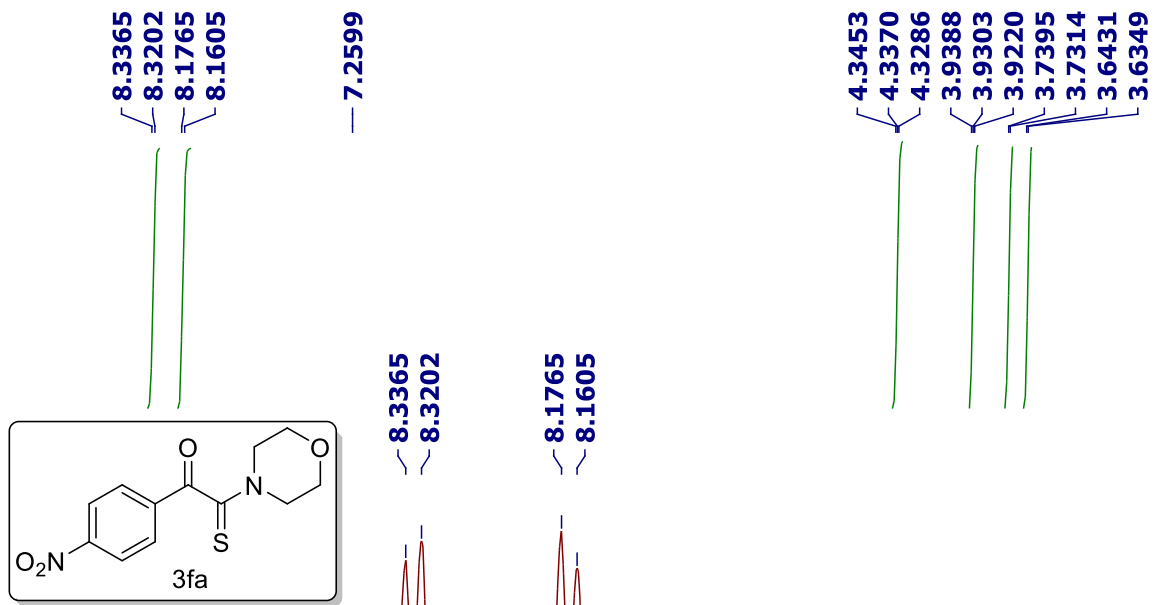
66.4968

— 52.1742

— 47.4406



<sup>1</sup>H NMR for compound 3fa (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3fa (125 MHz,  $\text{CDCl}_3$ )

— 193.8279

— 184.6090

— 150.9075

— 138.4868

— 130.9655

— 124.1522

77.4136

77.1597

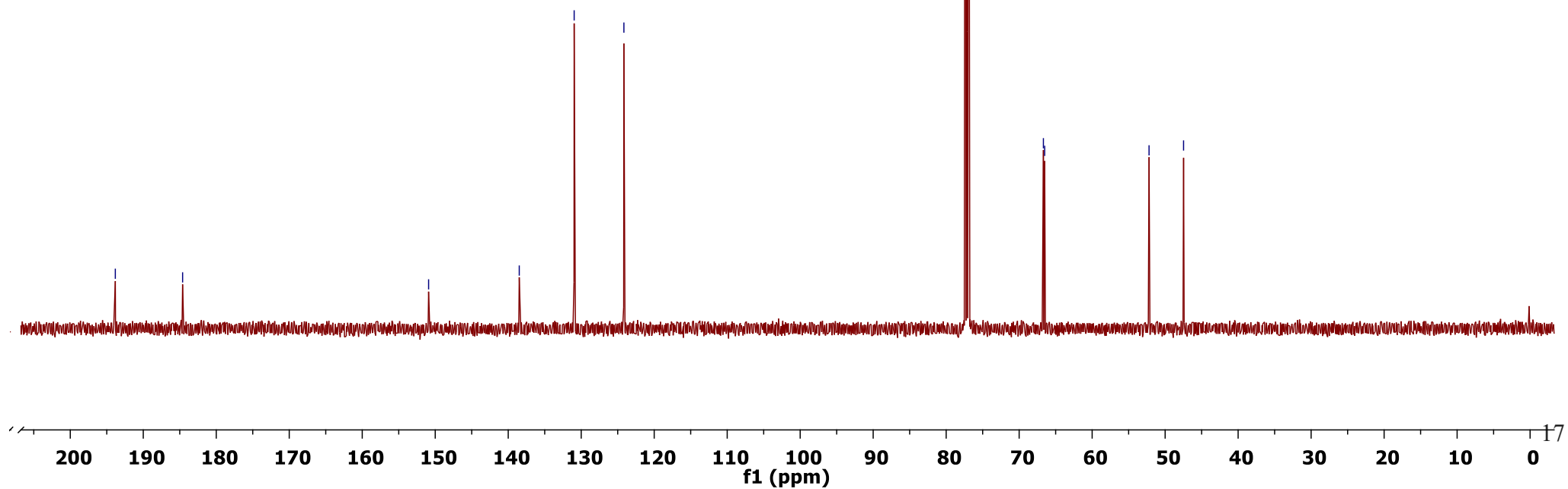
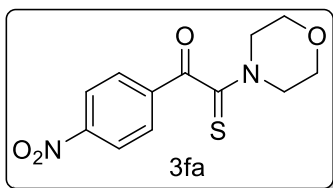
76.9056

66.6783

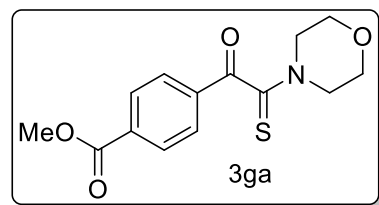
66.5061

— 52.2046

— 47.4754



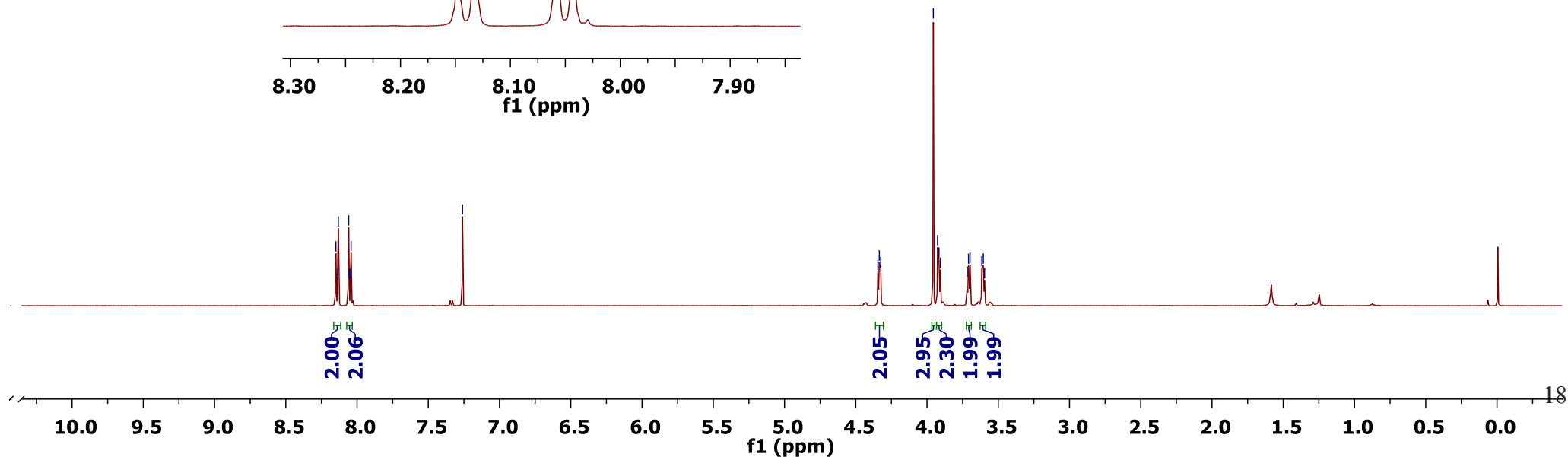
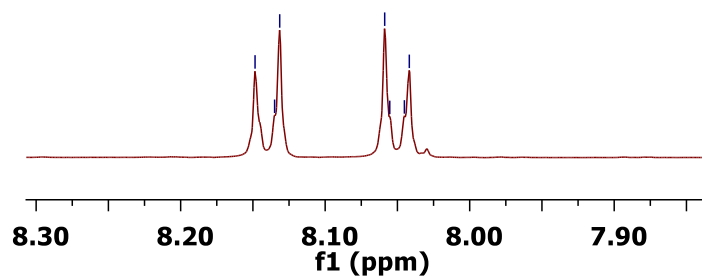
<sup>1</sup>H NMR for compound 3ga (500 MHz, CDCl<sub>3</sub>)



8.1485  
8.1350  
8.1315  
8.0588  
8.0553  
8.0452  
8.0418

8.1485  
8.1350  
8.1315  
8.0588  
8.0553  
8.0452  
8.0418

4.3444  
4.3348  
4.3246  
3.9555  
3.9259  
3.9162  
3.9065  
3.7173  
3.7086  
3.6981  
3.6153  
3.6051  
3.5962



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ga (125 MHz,  $\text{CDCl}_3$ )

— 194.8517

— 186.5259

— 165.9480

— 136.7954

— 134.9538

— 130.0752

— 129.7637

— 77.4036

— 77.1493

— 76.8948

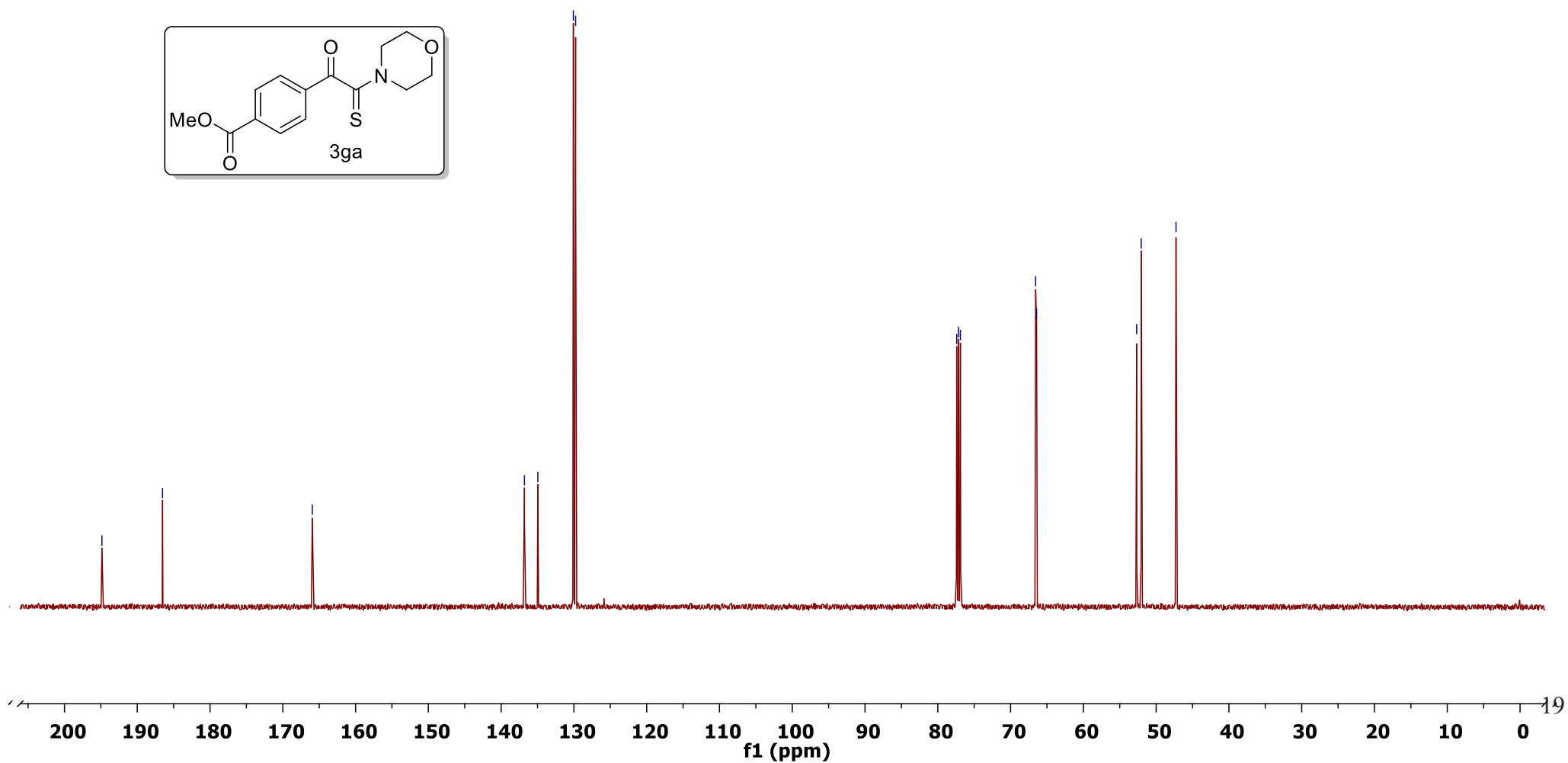
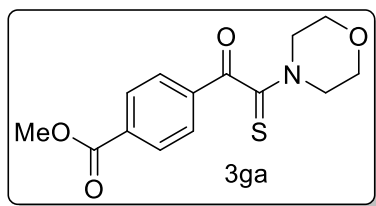
— 66.5537

— 66.4254

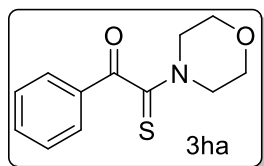
— 52.6748

— 52.0478

— 47.2696



<sup>1</sup>H NMR for compound 3ha (500 MHz, CDCl<sub>3</sub>)

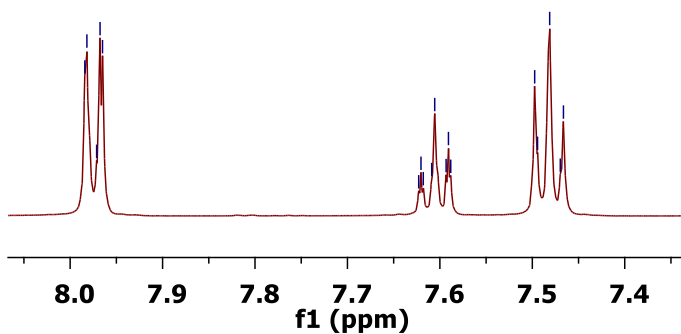


7.9841  
7.9819  
7.9714  
7.9676  
7.9650  
7.6229  
7.6204  
7.6180  
7.6088  
7.6056  
7.5932  
7.5907  
7.5883  
7.4974  
7.4943  
7.4812  
7.4697  
7.4663  
7.2598

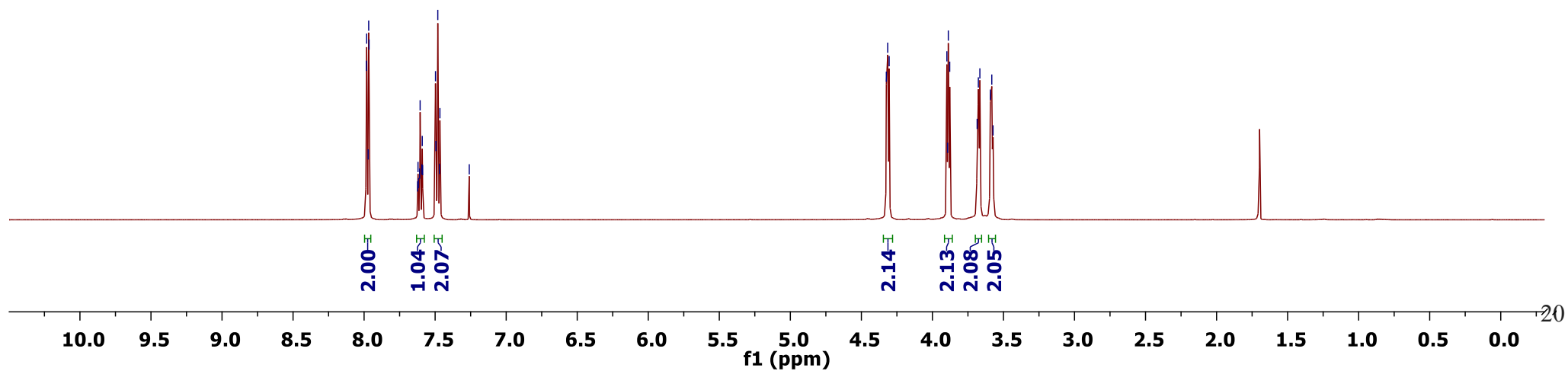
4.3244  
4.3146  
4.3046  
3.8981  
3.8933  
3.8881  
3.8783  
3.6852  
3.6767  
3.6661  
3.5921  
3.5822  
3.5732



7.9841  
7.9819  
7.9714  
7.9676  
7.9650  
7.6229  
7.6204  
7.6180  
7.6088  
7.6056  
7.5932  
7.5907  
7.5883  
7.4974  
7.4943  
7.4812  
7.4697  
7.4663



2.00  
1.04  
2.07  
2.14  
2.13  
2.08  
2.05





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ha (125 MHz,  $\text{CDCl}_3$ )

— 195.7165

— 187.9491

— 134.5250

— 133.2924

— 129.8887

— 129.0260

— 77.4115

— 77.1571

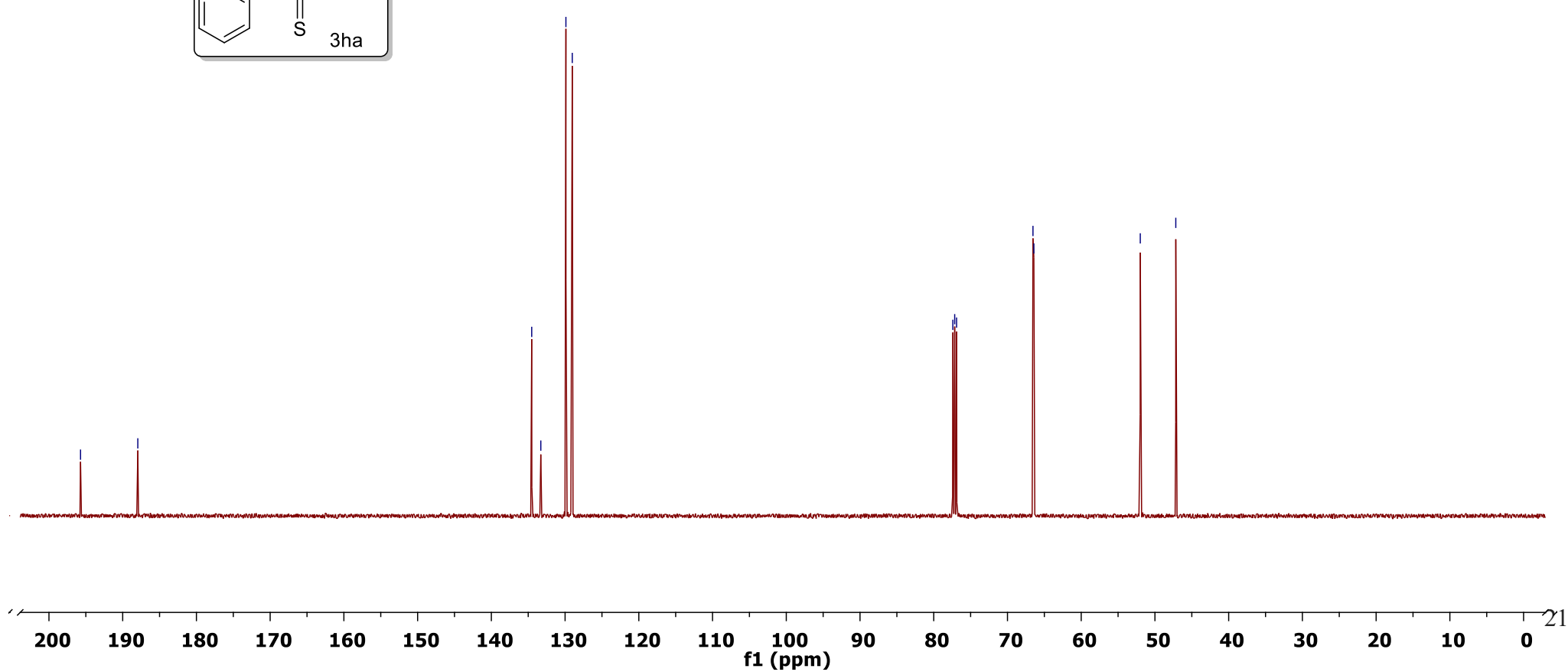
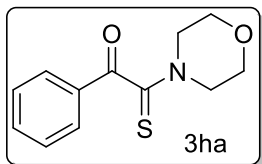
— 76.9029

— 66.5412

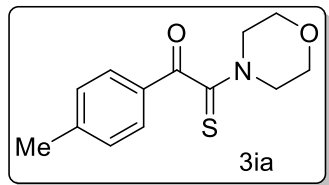
— 66.4224

— 51.9892

— 47.1719

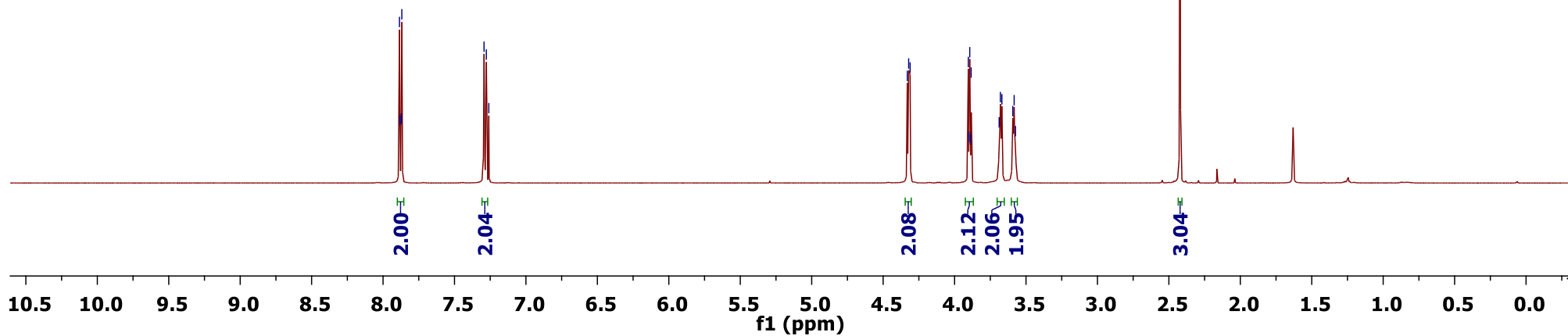
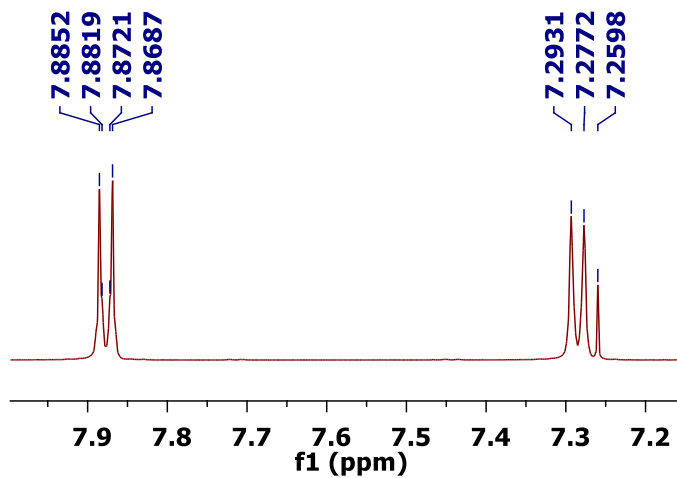


<sup>1</sup>H NMR for compound 3ia (500 MHz, CDCl<sub>3</sub>)



7.8852  
7.8819  
7.8721  
7.8687  
7.2931  
7.2772  
7.2598

4.3309  
4.3212  
4.3111  
3.9038  
3.8937  
3.8840  
3.6879  
3.6792  
3.6689  
3.5923  
3.5829  
3.5737



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ia (125 MHz,  $\text{CDCl}_3$ )

— 196.1352

— 188.0043

— 145.8416

130.8498

130.0898

129.7966

77.4136

77.1593

76.9051

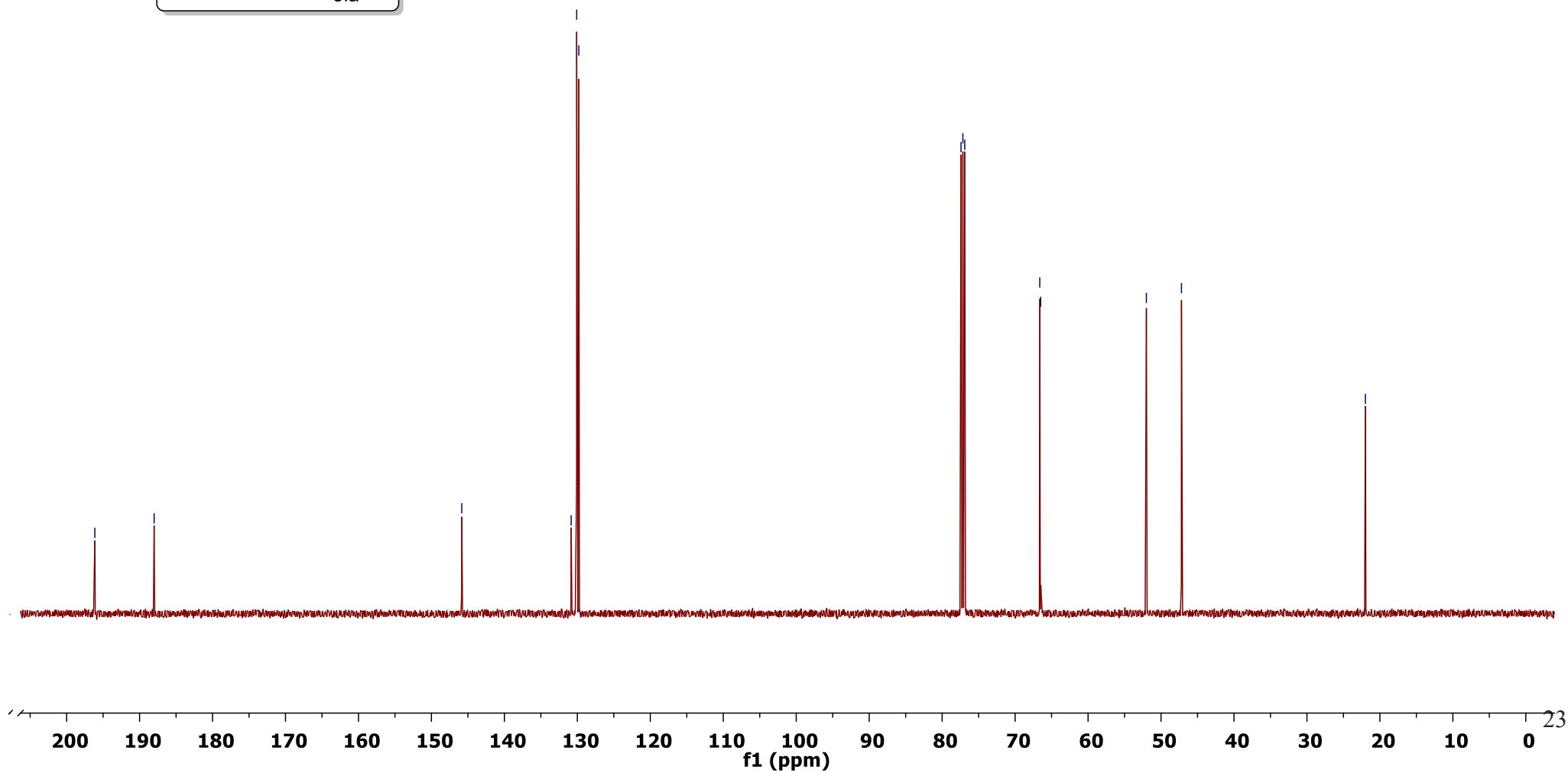
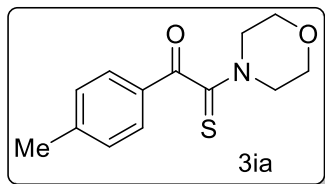
66.6189

66.4953

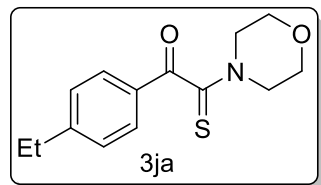
— 52.0065

— 47.1981

— 21.9860



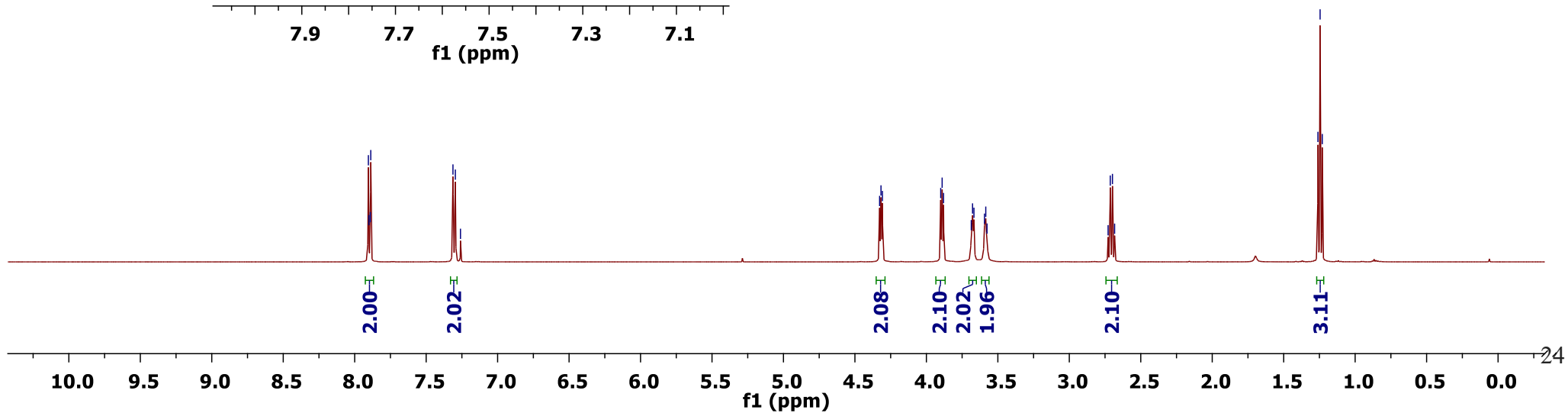
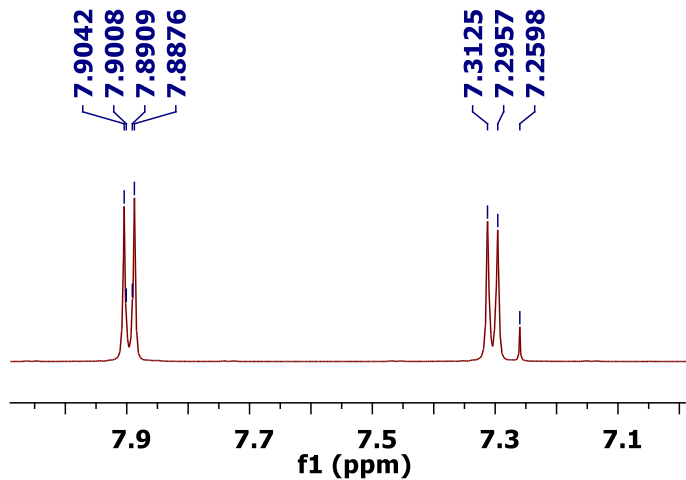
<sup>1</sup>H NMR for compound 3ja (500 MHz, CDCl<sub>3</sub>)



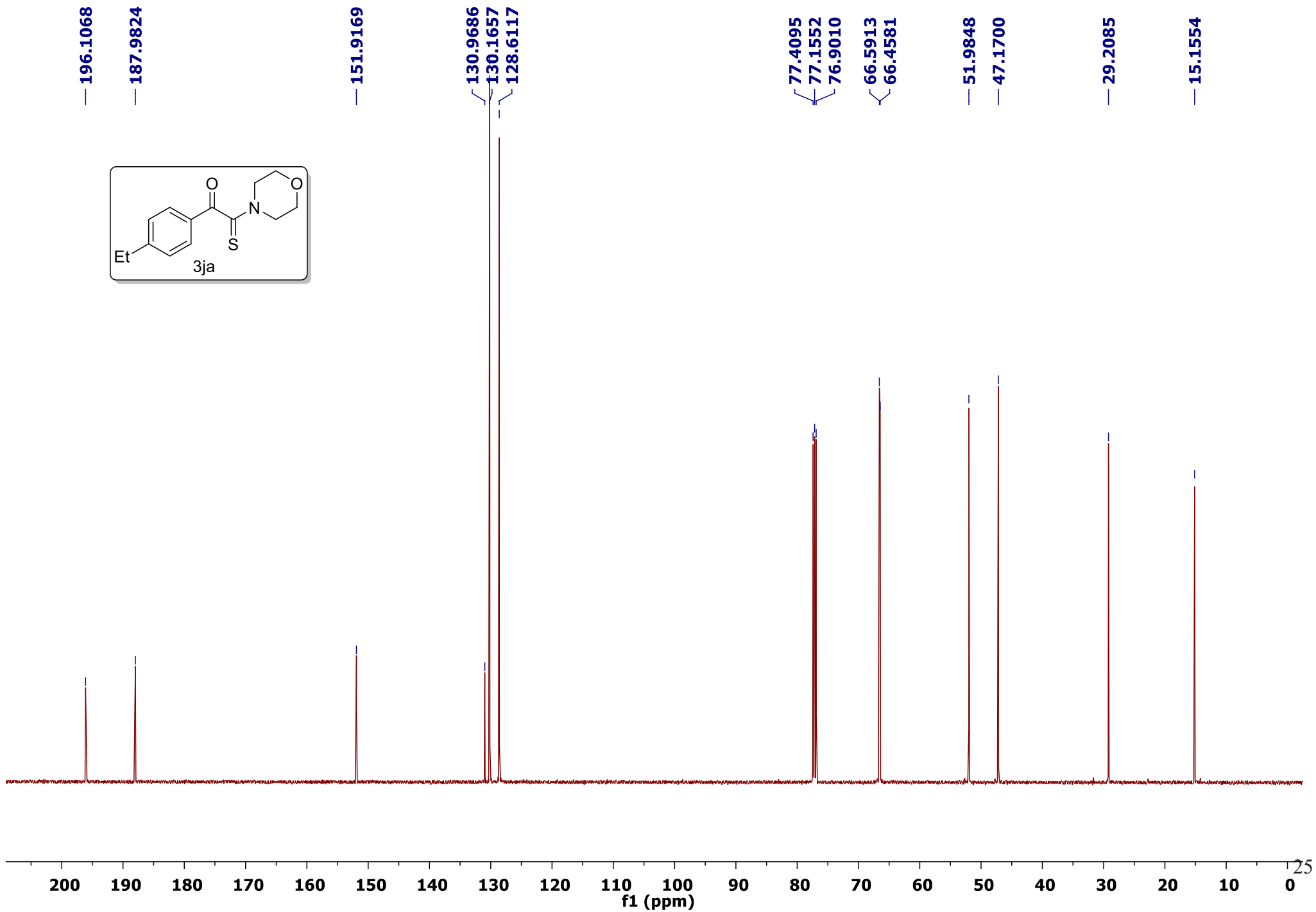
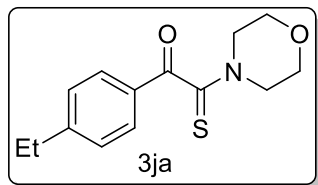
7.9042  
7.9008  
7.8909  
7.8876  
7.3125  
7.2957  
7.2598

4.3273  
4.3176  
4.3075  
3.8997  
3.8897  
3.8799  
3.6770  
3.6673  
3.5886  
2.7133  
2.6981  
2.6829

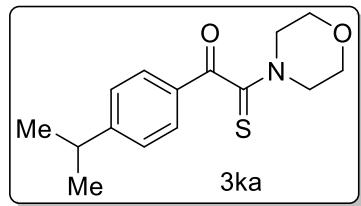
1.2605  
1.2453  
1.2300



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ja (125 MHz,  $\text{CDCl}_3$ )



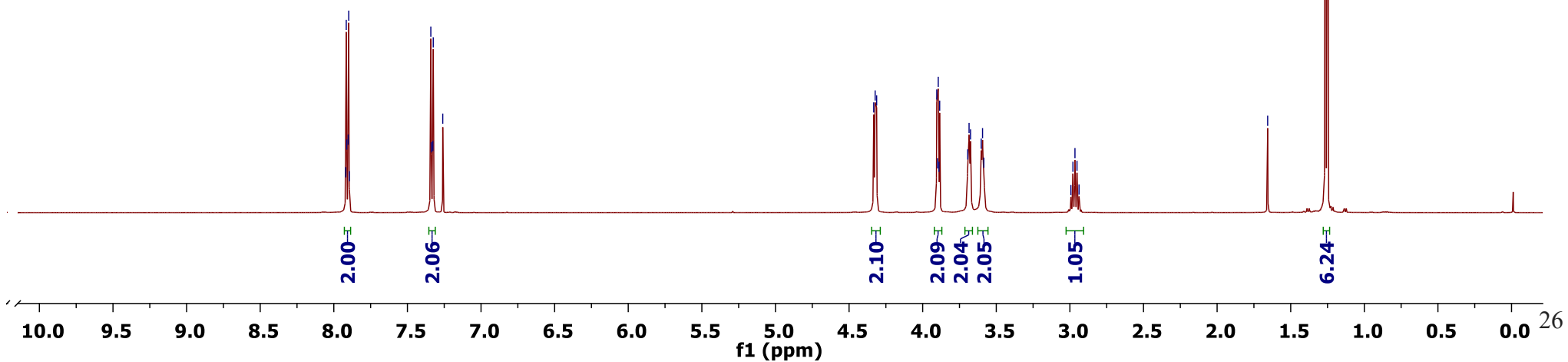
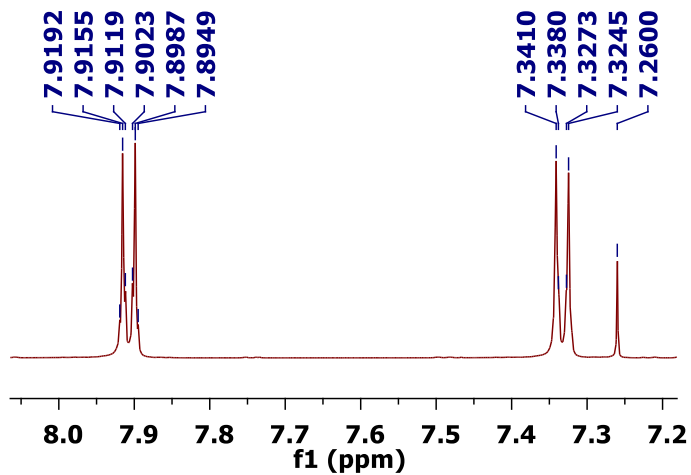
<sup>1</sup>H NMR for compound 3ka (500 MHz, CDCl<sub>3</sub>)



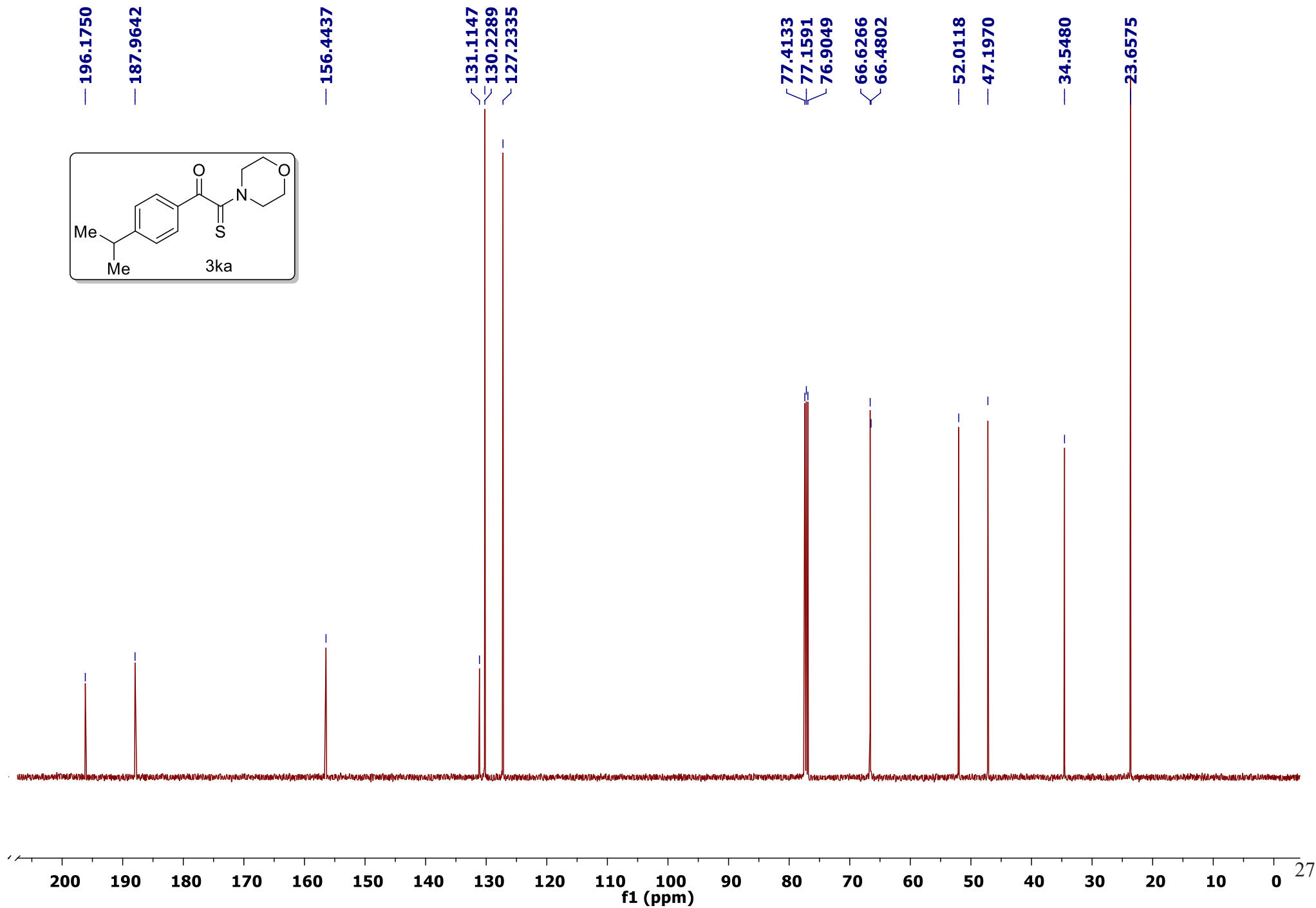
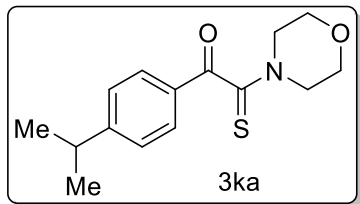
7.9192  
7.9155  
7.9119  
7.9023  
7.8987  
7.8949  
7.3410  
7.3380  
7.3273  
7.3245  
7.2600

4.3325  
4.3228  
4.3127  
3.9043  
3.8942  
3.8844  
3.6849  
3.5933  
2.9793  
2.9655  
2.9517  
2.9379

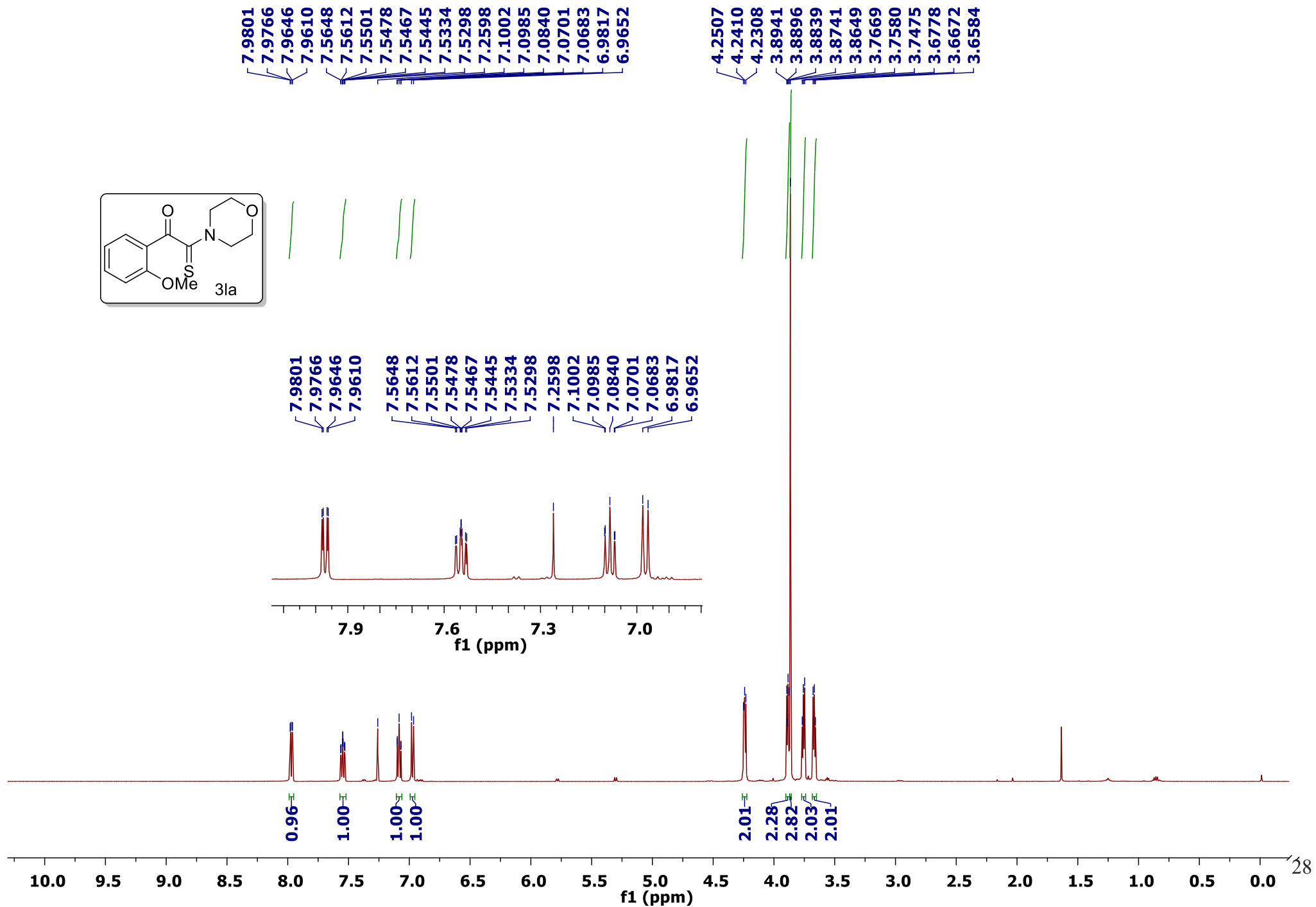
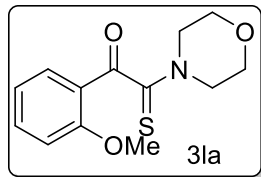
1.6565  
1.2645  
1.2506



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ka (125 MHz,  $\text{CDCl}_3$ )



<sup>1</sup>H NMR for compound 3la (500 MHz, CDCl<sub>3</sub>)





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3la (125 MHz,  $\text{CDCl}_3$ )

— 198.4120

— 186.6998

— 159.2368

— 135.6394

— 131.9006

— 124.4739

— 121.6216

— 112.6509

77.4150

77.1610

76.9068

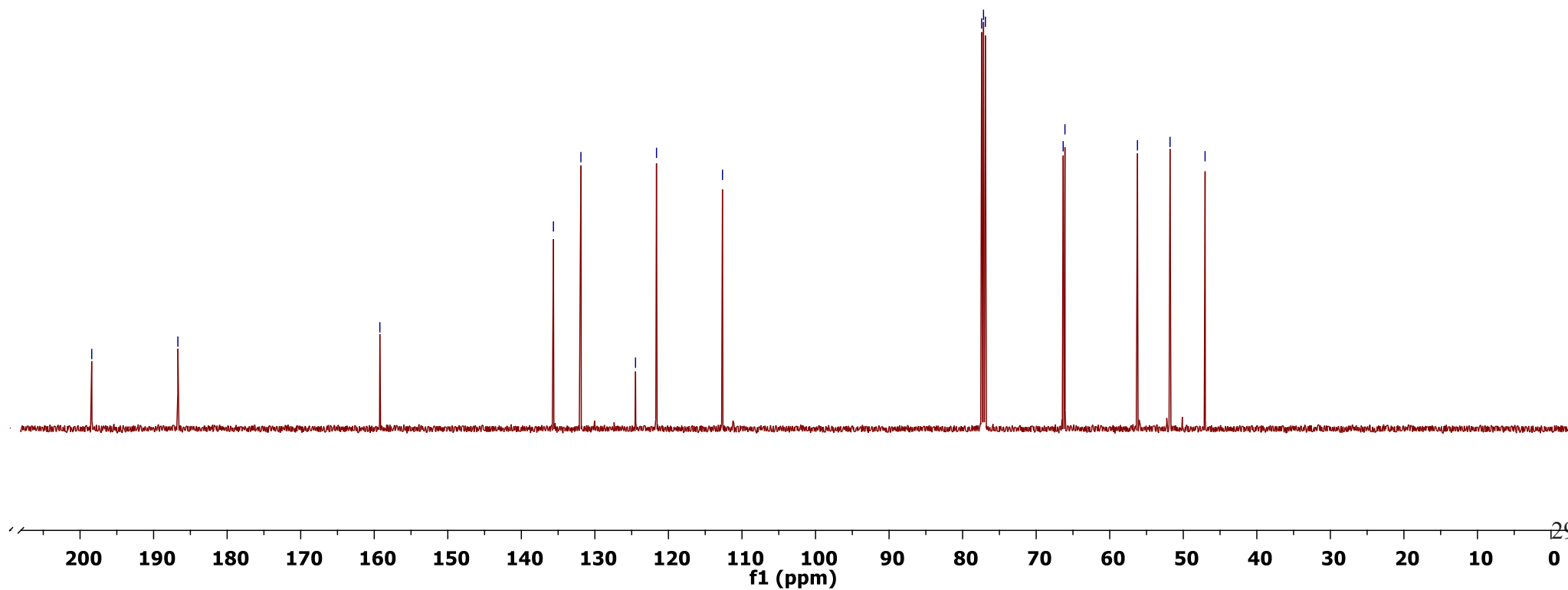
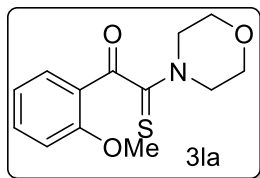
66.3306

66.0875

56.2303

51.7997

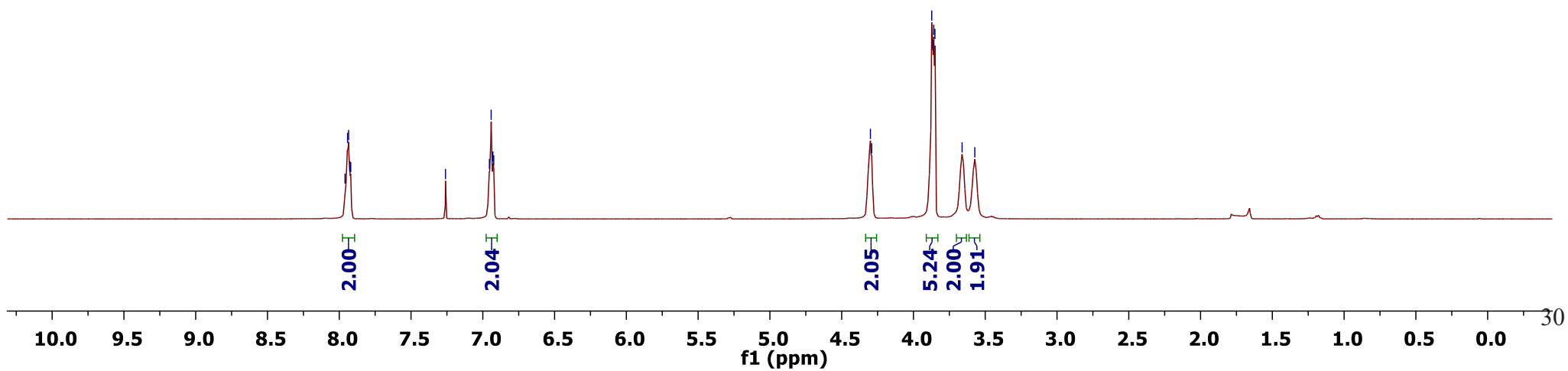
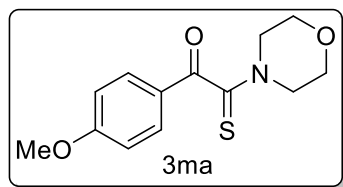
47.0348



<sup>1</sup>H NMR for compound 3ma (500 MHz, CDCl<sub>3</sub>)

7.9597  
7.9421  
7.9349  
7.9270  
7.9201  
7.2599  
6.9537  
6.9417  
6.9301  
6.9240

4.2996  
4.2910  
3.8727  
3.8657  
3.8585  
3.8505  
3.6611  
3.5725



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ma (125 MHz,  $\text{CDCl}_3$ )

— 196.1986

— 187.3353

— 164.7041

— 132.3723

— 126.0718

— 114.3606

{ 77.4149

{ 77.1605

{ 76.9060

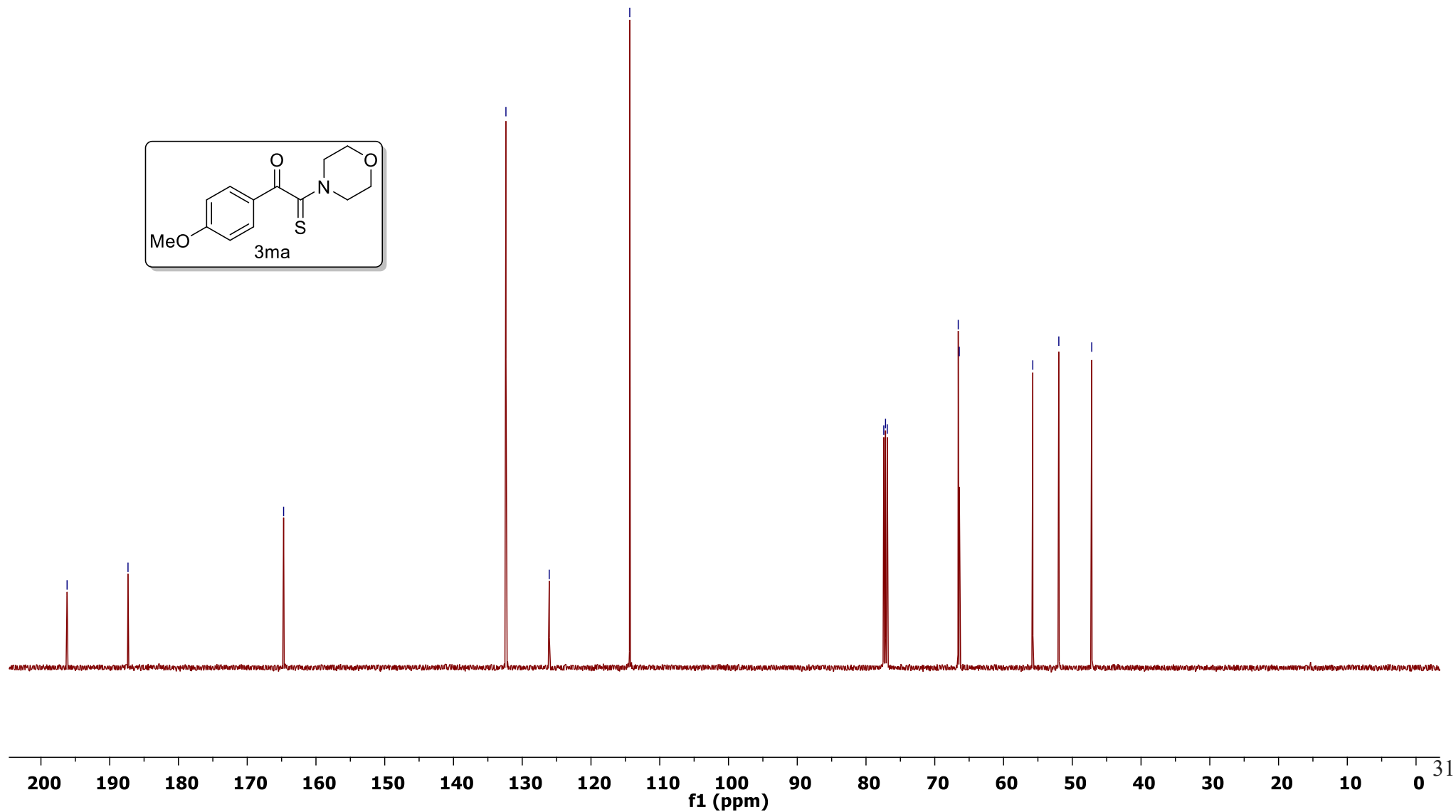
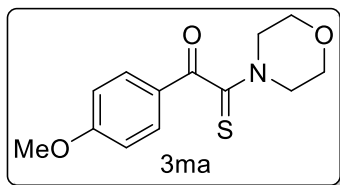
{ 66.5877

{ 66.4350

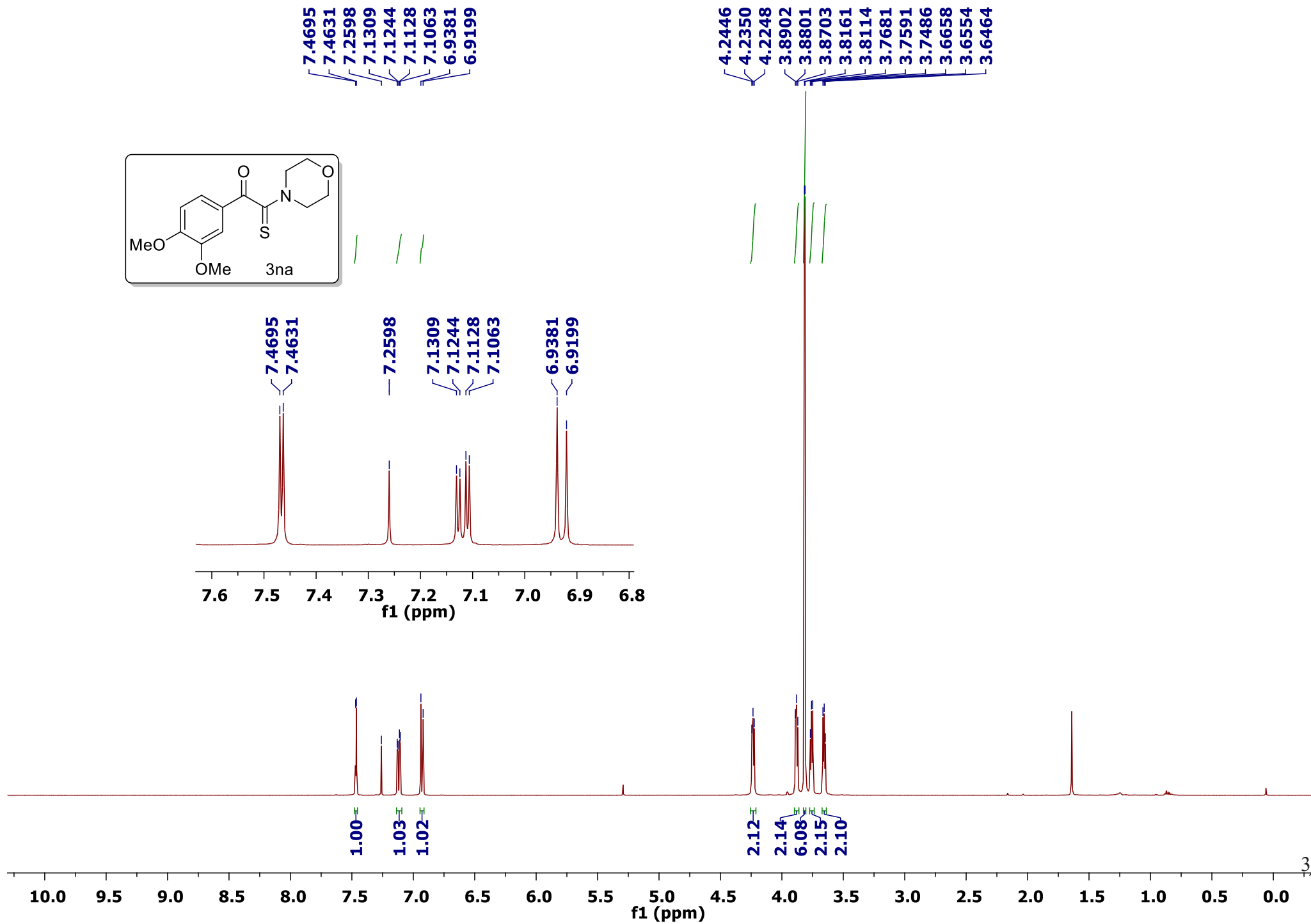
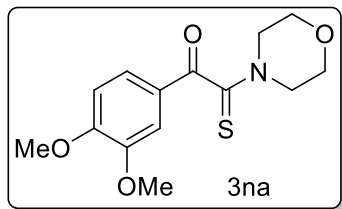
{ 55.7437

{ 51.9576

{ 47.1725



<sup>1</sup>H NMR for compound 3na (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3na (125 MHz,  $\text{CDCl}_3$ )

— 198.2460

— 186.3243

— 154.2990

— 153.8641

— 124.8329

— 123.3085

— 114.7268

— 113.7912

— 77.4142

— 77.1601

— 76.9058

— 66.3021

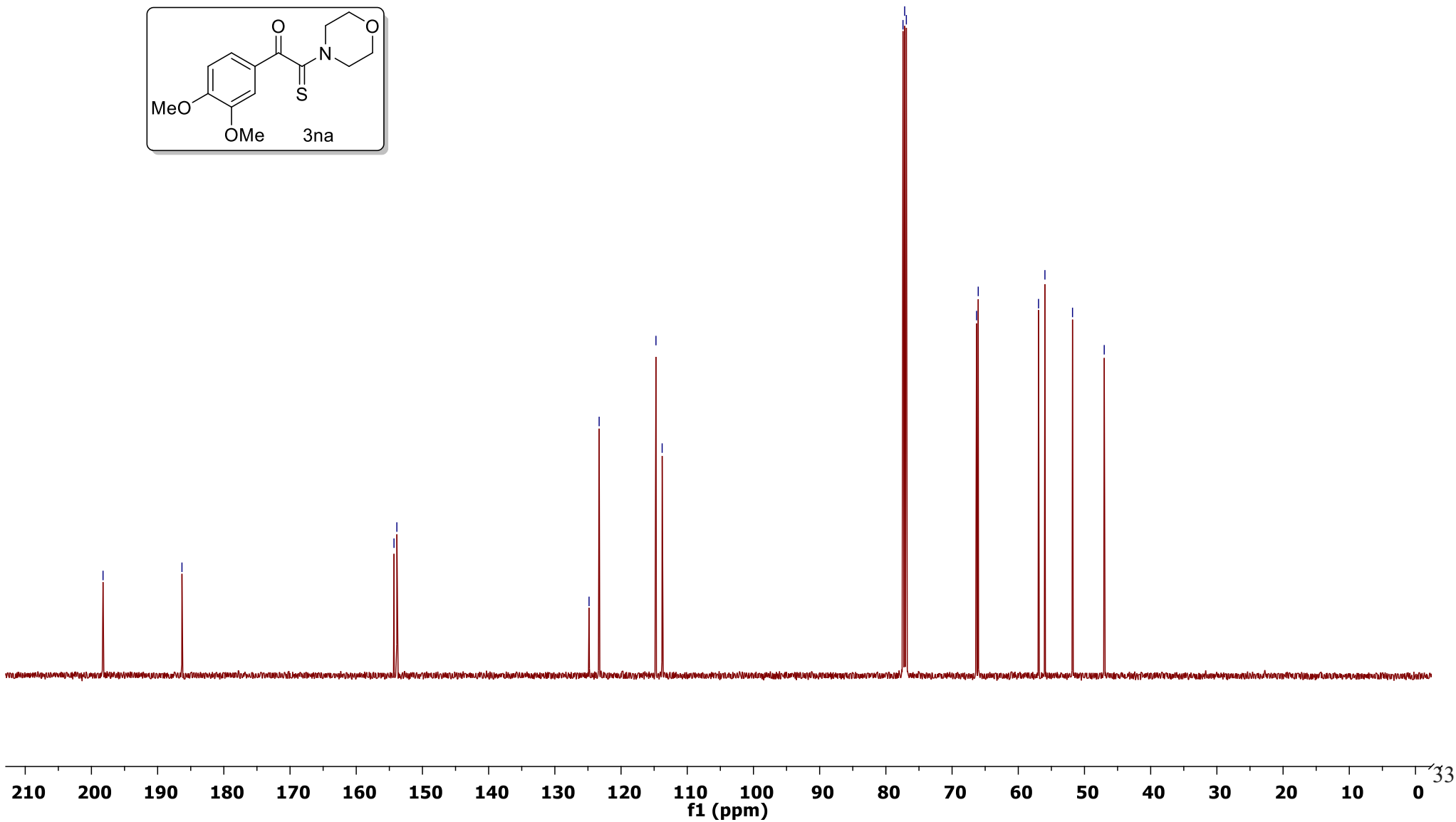
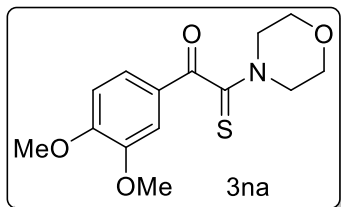
— 66.0494

— 56.9388

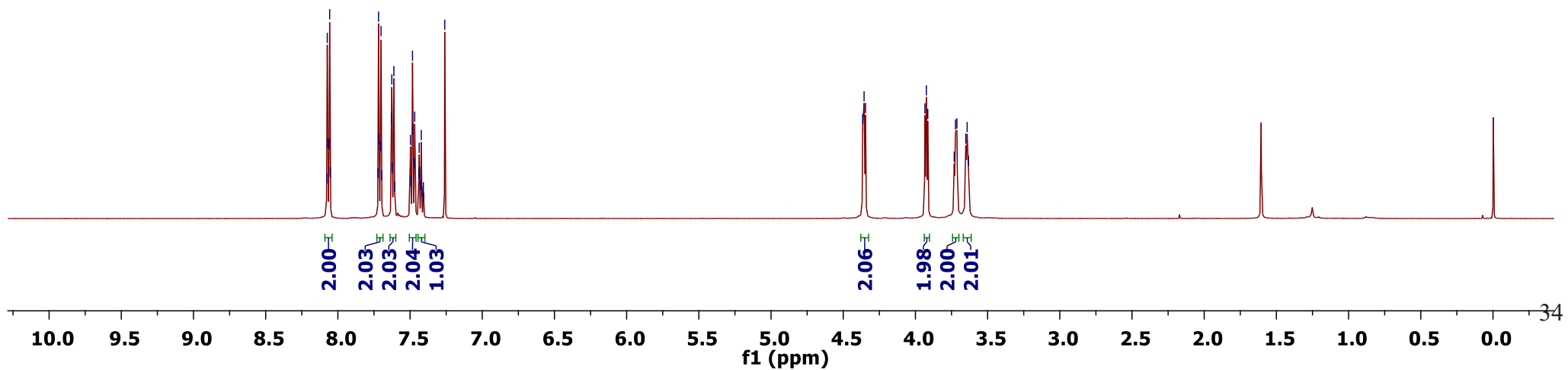
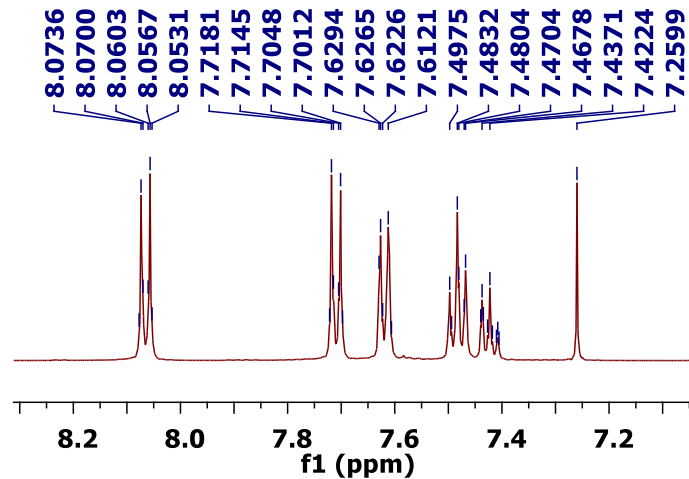
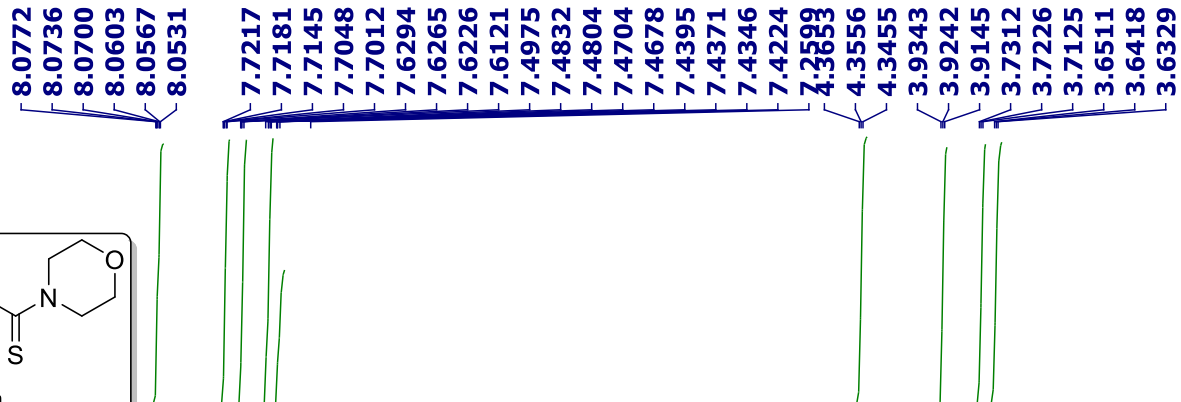
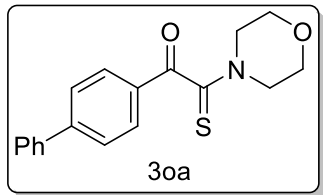
— 55.9752

— 51.8032

— 47.0158



<sup>1</sup>H NMR for compound 30a (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3oa (125 MHz,  $\text{CDCl}_3$ )

— 195.8799

— 187.6754

— 147.3494

— 139.6678

— 132.0867

— 130.5735

— 129.1792

— 128.7201

— 127.7387

— 127.4616

— 77.4132

— 77.1592

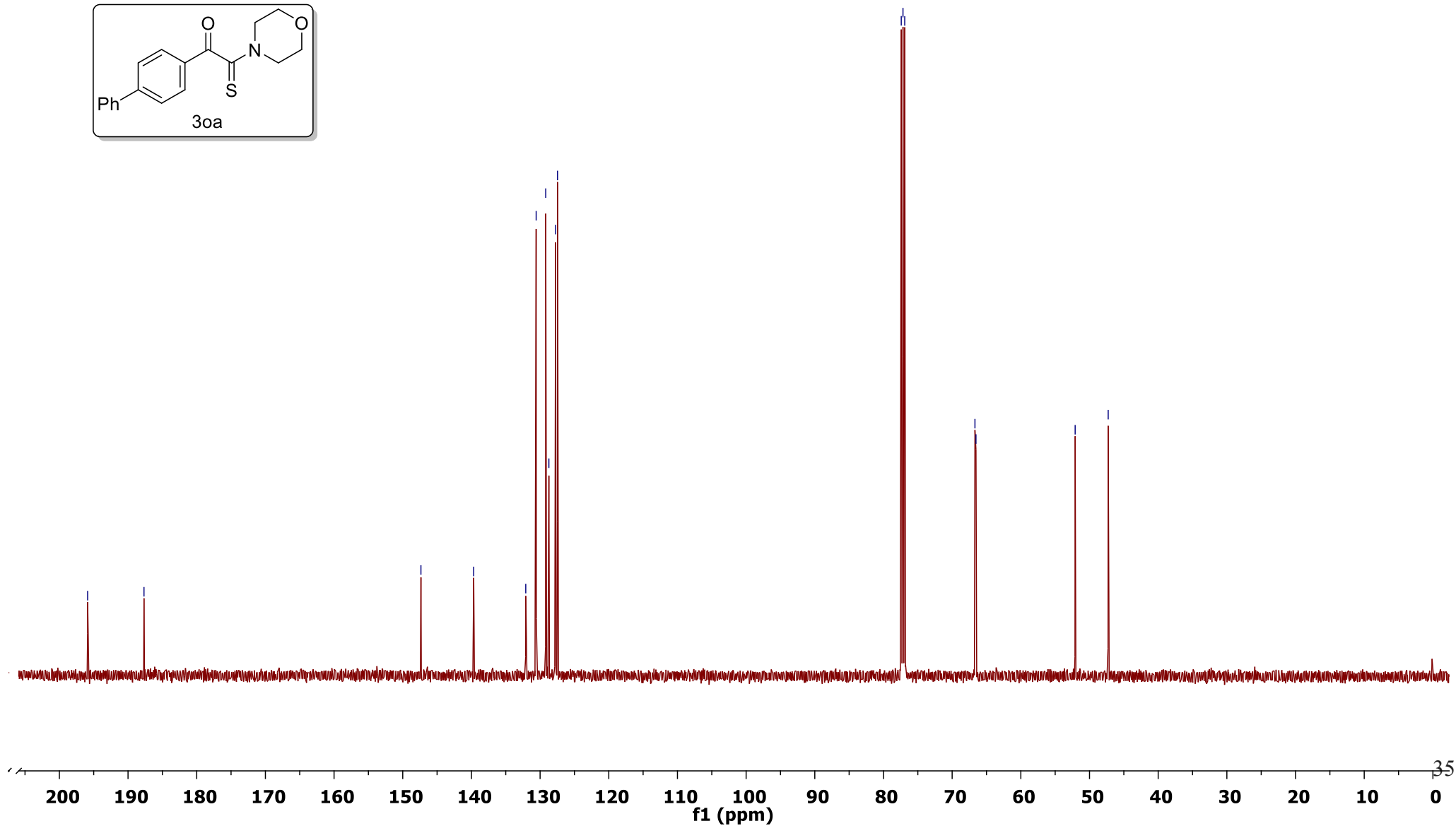
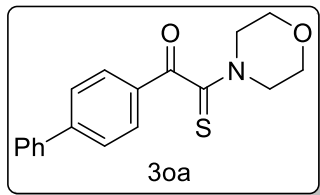
— 76.9049

— 66.6816

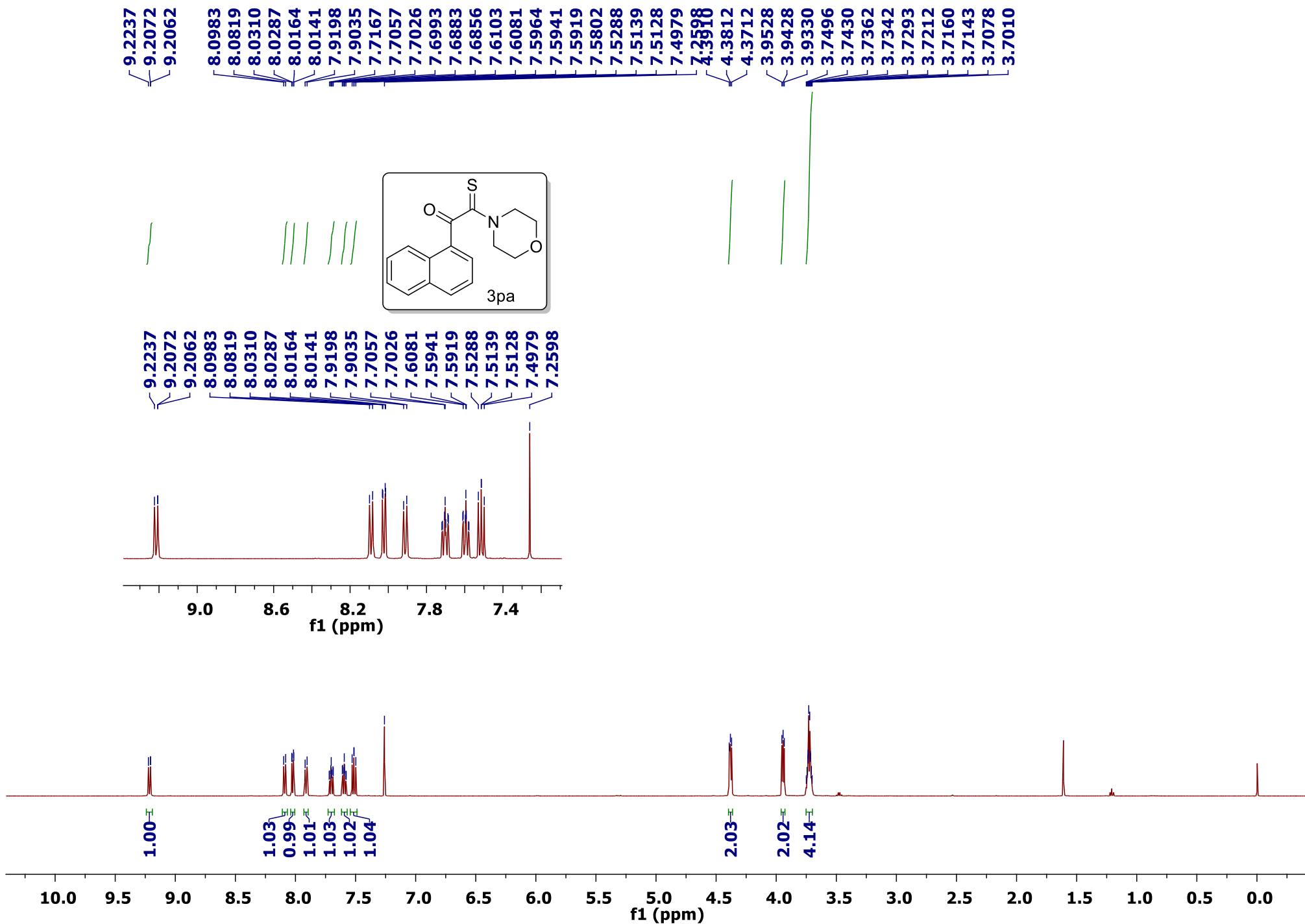
— 66.5321

— 52.0921

— 47.2797



<sup>1</sup>H NMR for compound 3pa (500 MHz, CDCl<sub>3</sub>)





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3pa (125 MHz,  $\text{CDCl}_3$ )

— 196.9260

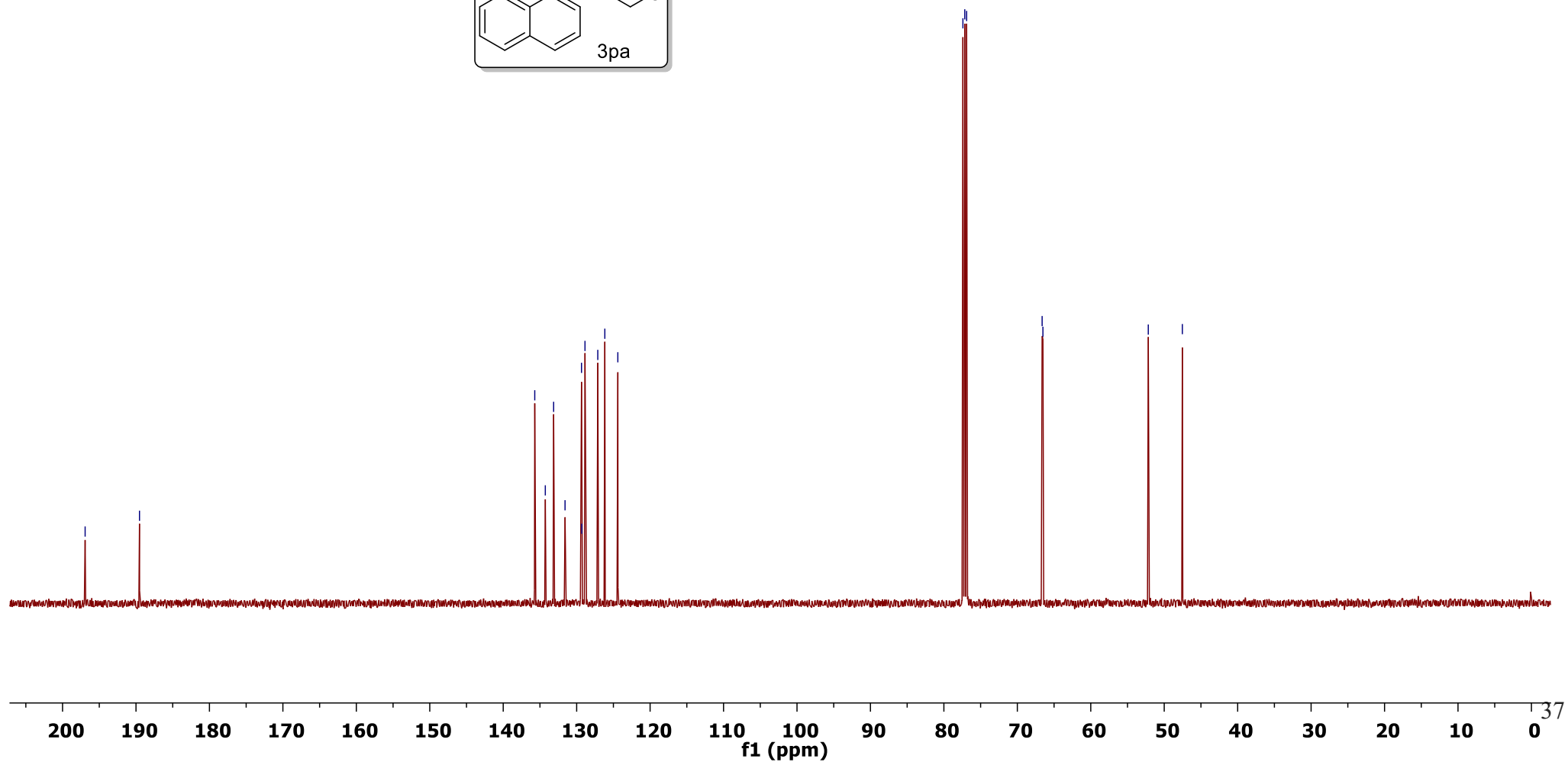
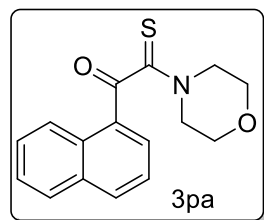
— 189.5237

135.7059  
134.2705  
133.1360  
131.5809  
129.3386  
129.3004  
128.8673  
127.1233  
126.1654  
124.3942

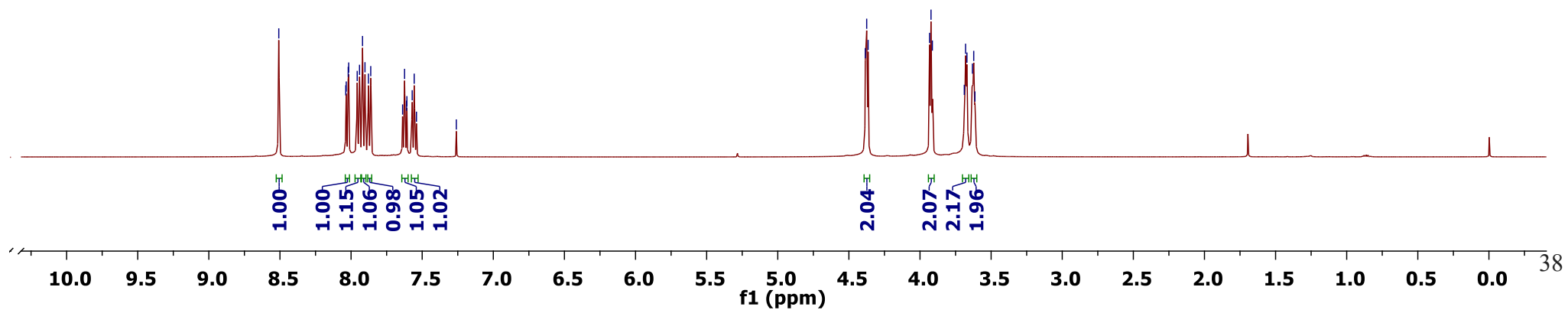
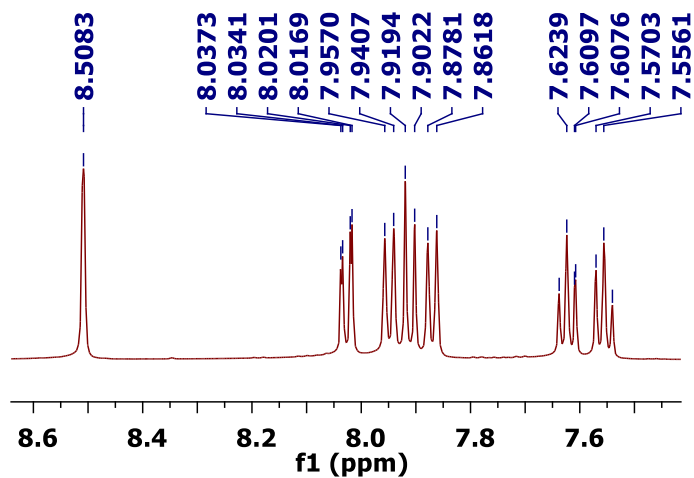
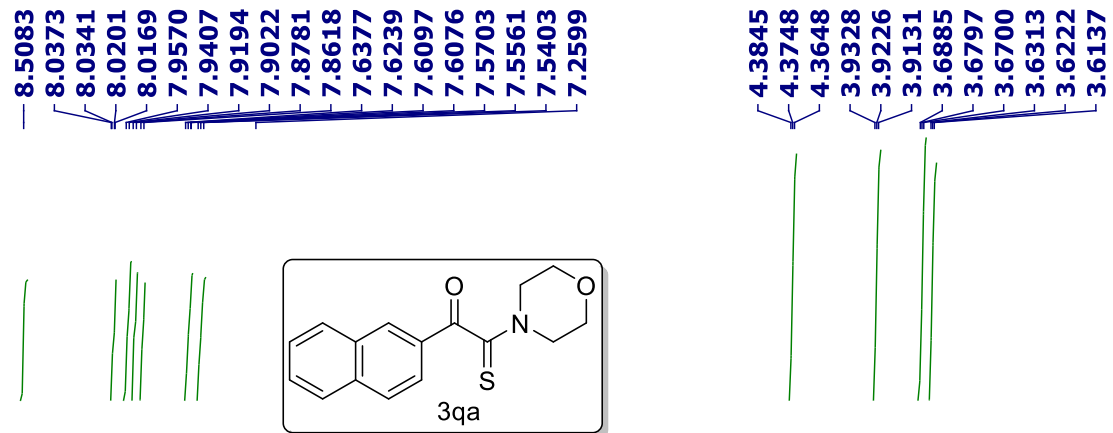
77.4095  
77.1554  
76.9013

66.6228  
66.5159

— 52.1682  
— 47.5300



<sup>1</sup>H NMR for compound 3qa (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3qa (125 MHz,  $\text{CDCl}_3$ )

— 195.8206

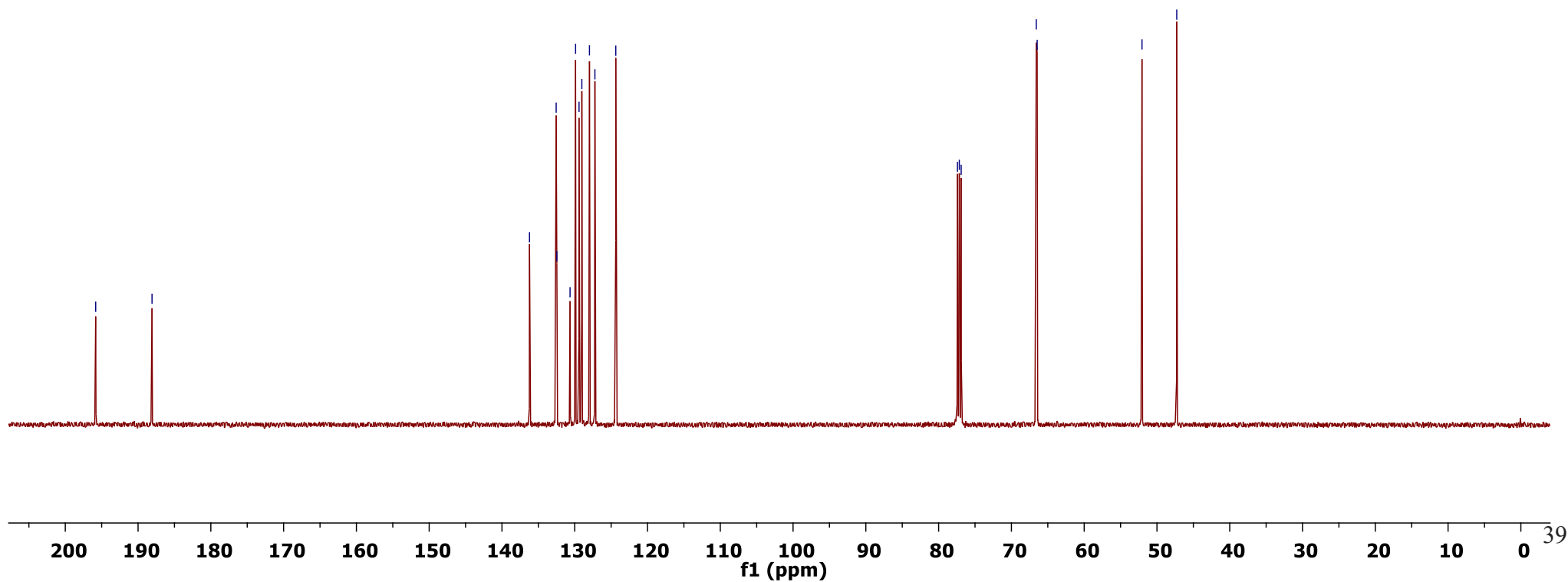
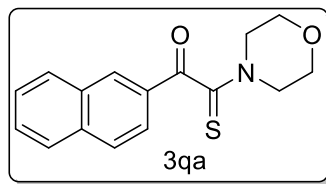
— 188.0819

136.2154  
132.5496  
132.4558  
130.6423  
129.8841  
129.4094  
129.0222  
127.9755  
127.2269  
124.3602

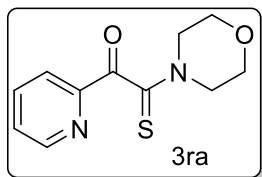
77.4109  
77.1567  
76.9023

66.5825  
66.4610

— 52.0549  
— 47.2820

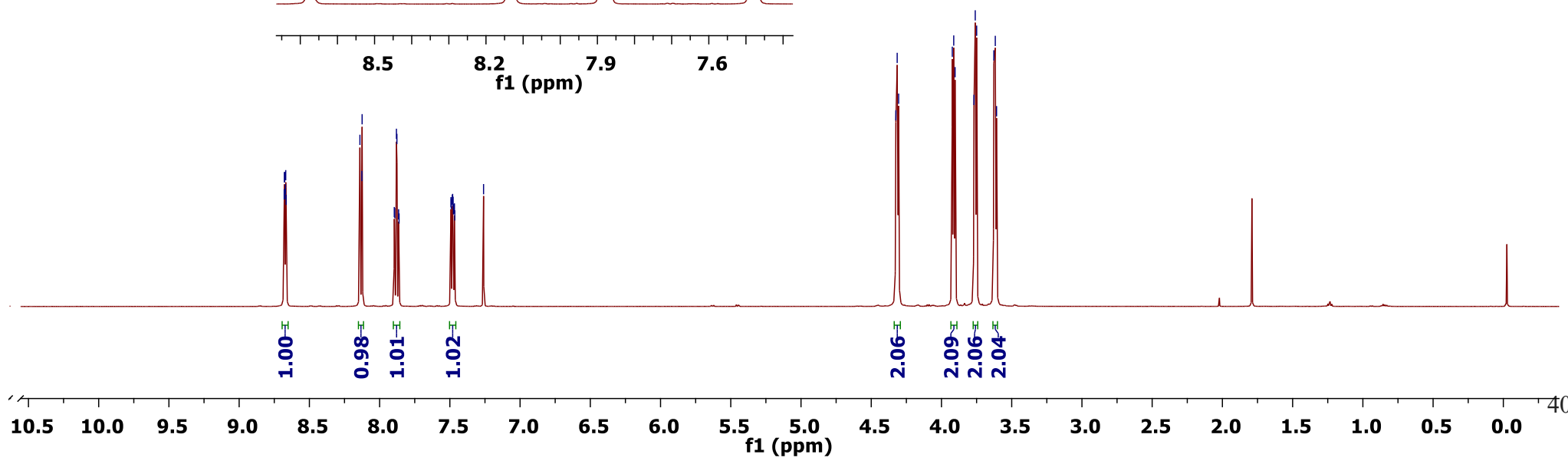
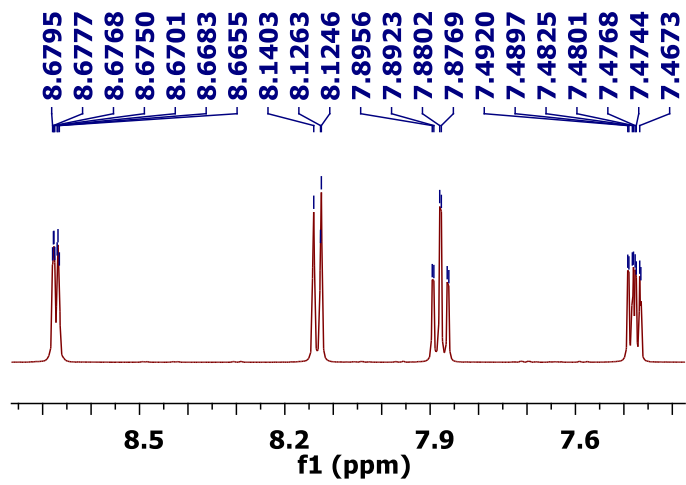
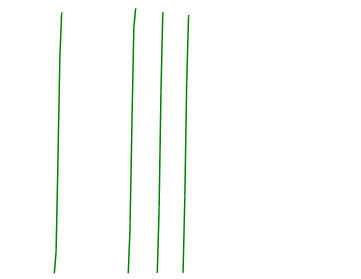
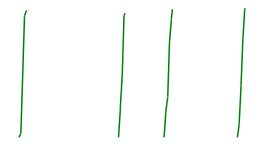


<sup>1</sup>H NMR for compound 3ra (500 MHz, CDCl<sub>3</sub>)

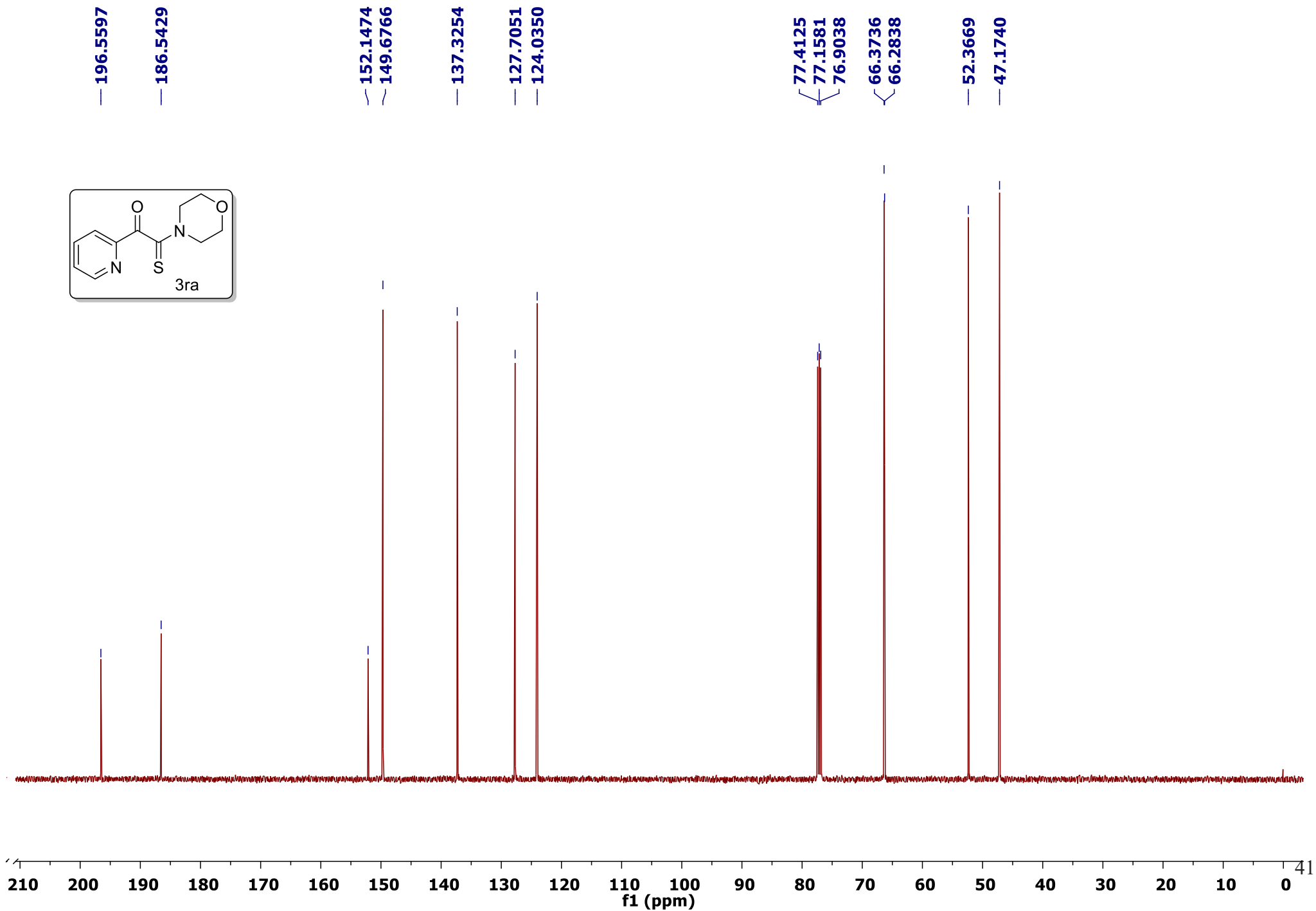
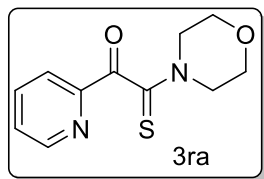


8.6795  
8.6777  
8.6768  
8.6750  
8.6701  
8.6683  
8.6655  
8.1403  
8.1246  
7.8802  
7.8769  
7.8720  
7.4897  
7.4825  
7.4801  
7.4768  
7.4744  
7.4673  
7.4649  
7.2599

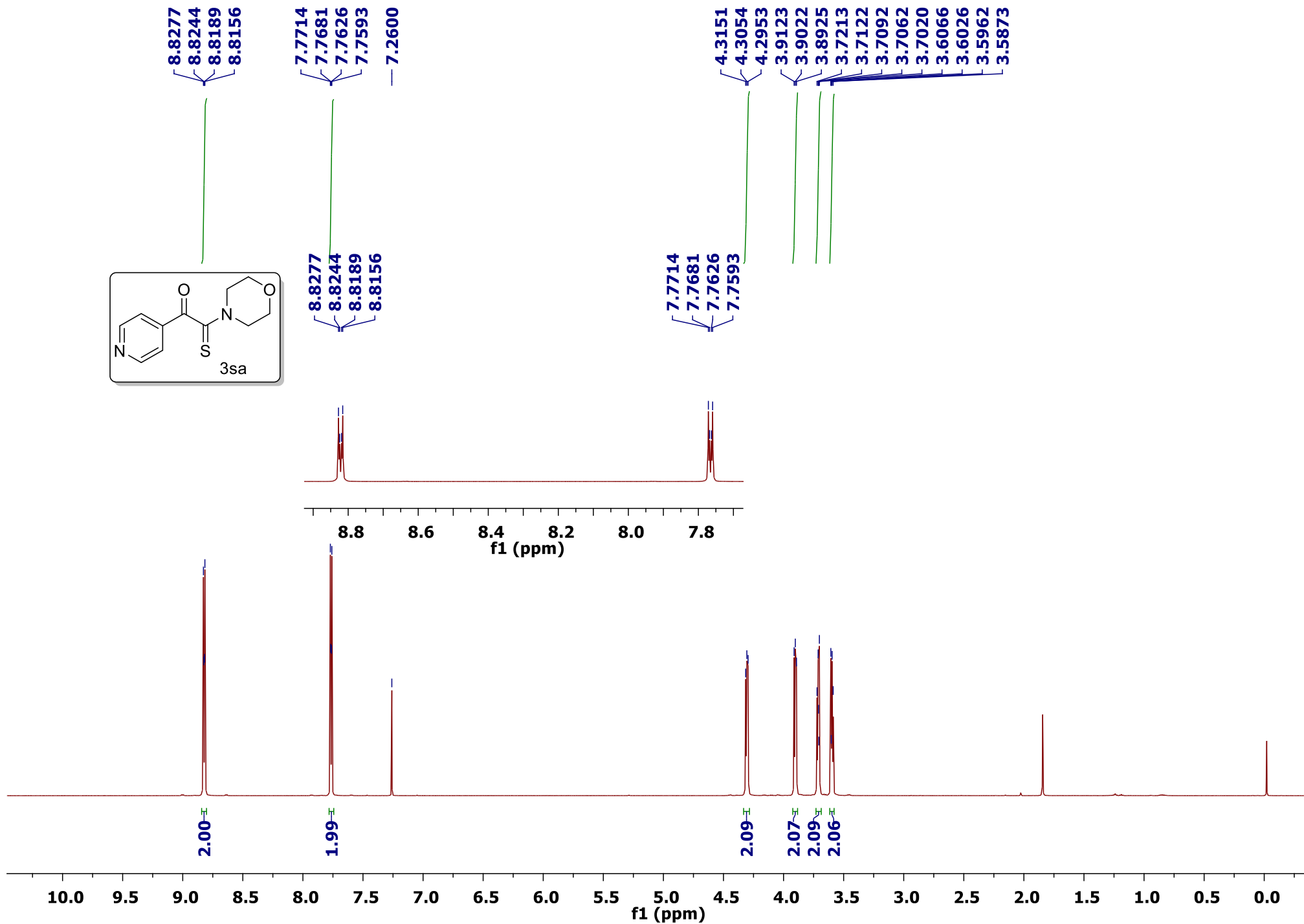
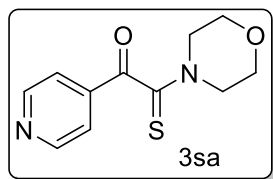
4.3249  
4.3152  
4.3050  
3.9229  
3.9127  
3.9030  
3.7688  
3.7595  
3.7494  
3.6272  
3.6169  
3.6078



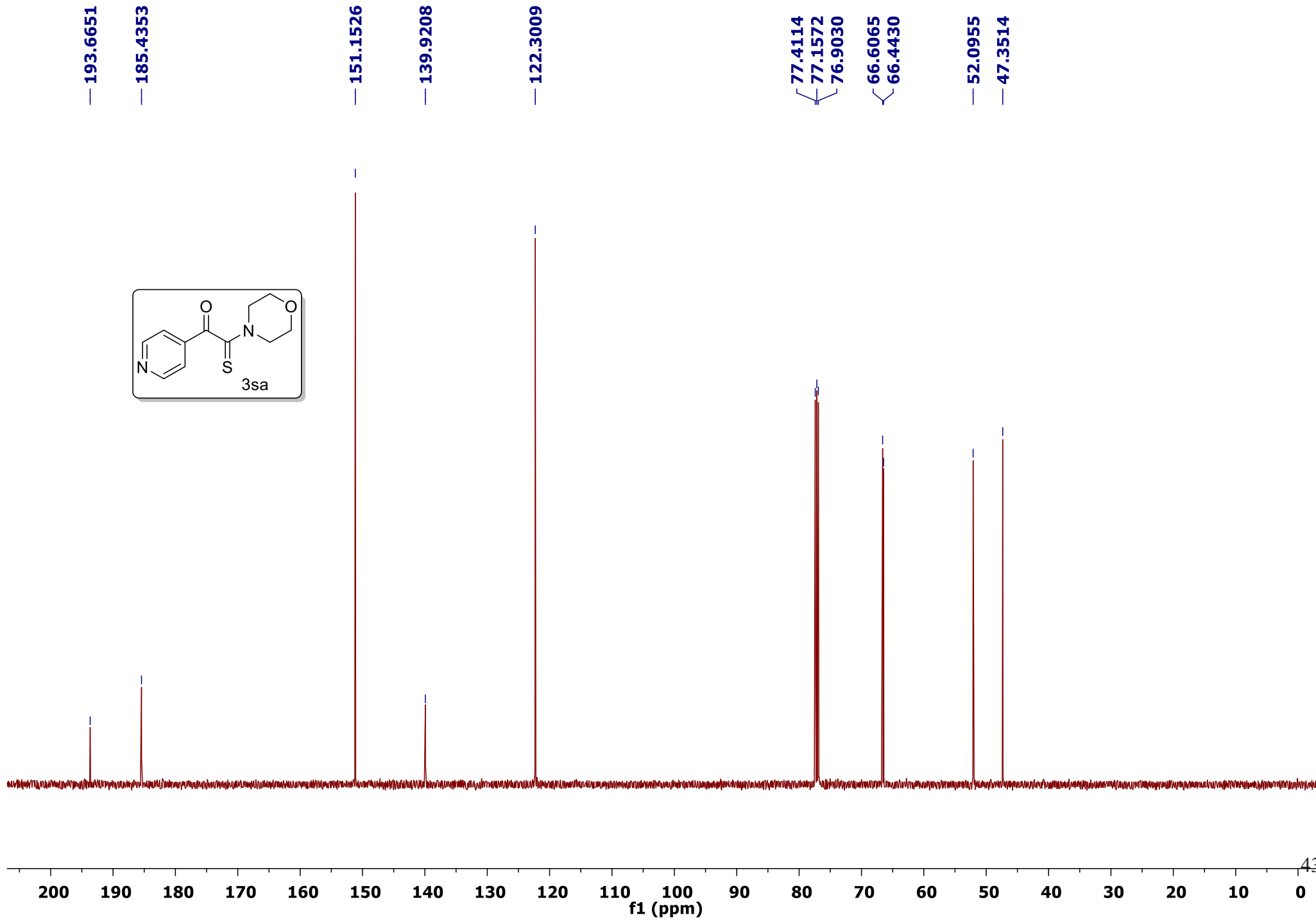
$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ra (125 MHz,  $\text{CDCl}_3$ )



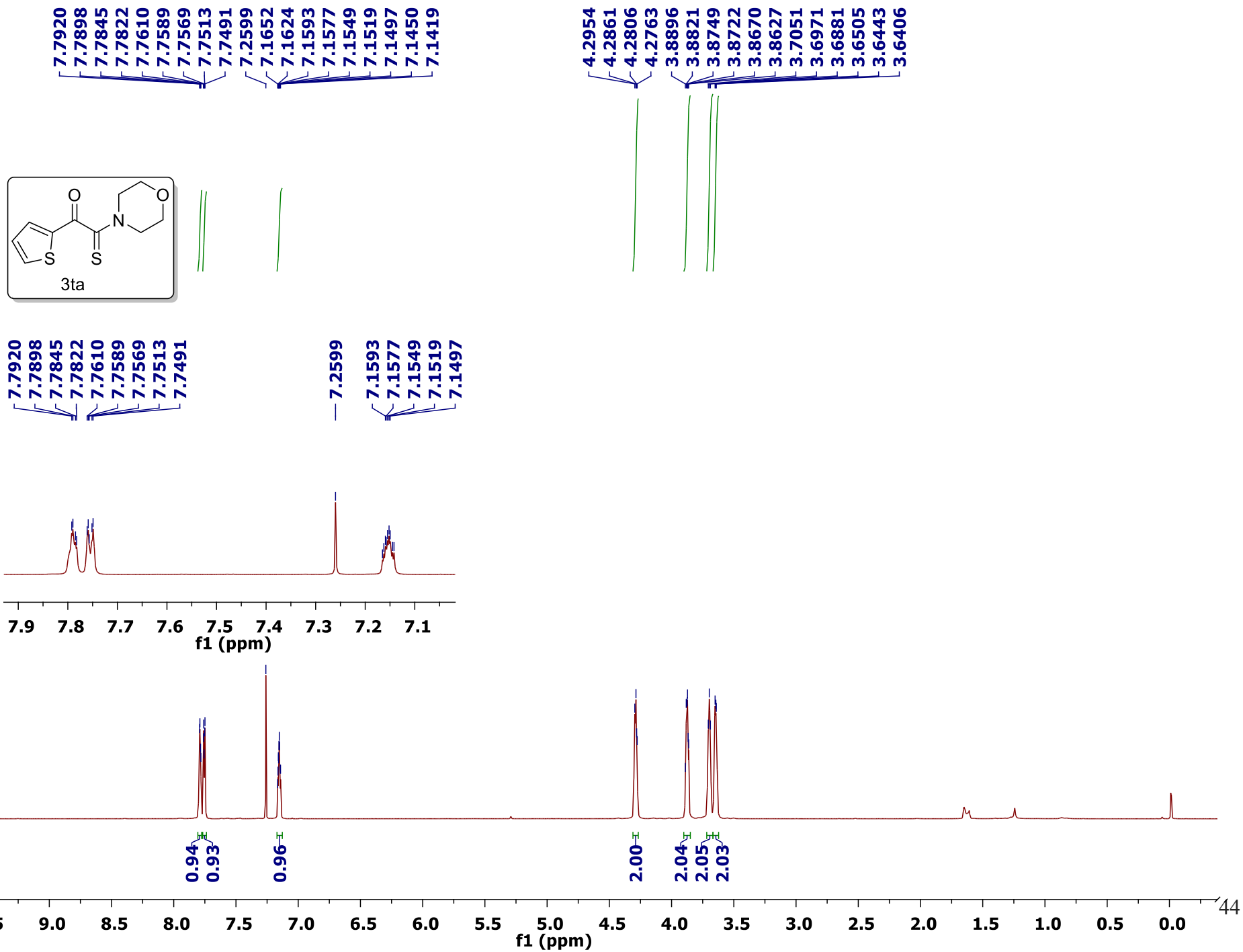
<sup>1</sup>H NMR for compound 3sa (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3sa (125 MHz,  $\text{CDCl}_3$ )



<sup>1</sup>H NMR for compound 3ta (500 MHz, CDCl<sub>3</sub>)





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ta (125 MHz,  $\text{CDCl}_3$ )

— 194.3602

— 181.1271

— 140.3685

— 136.3148

— 135.7200

— 128.7589

— 77.4109

— 77.1569

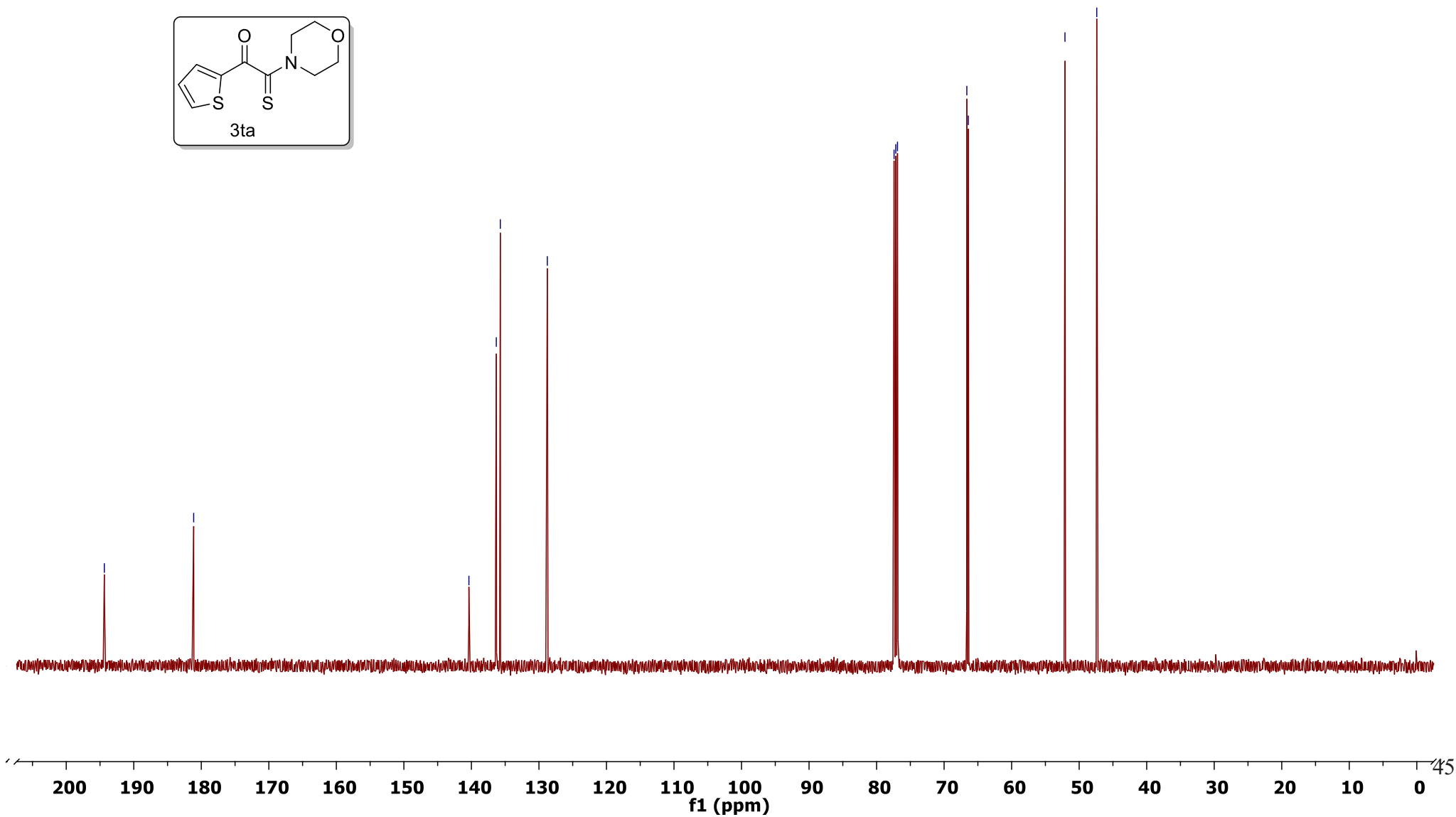
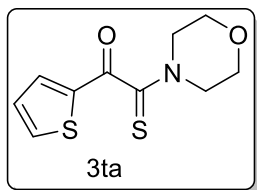
— 76.9024

— 66.6353

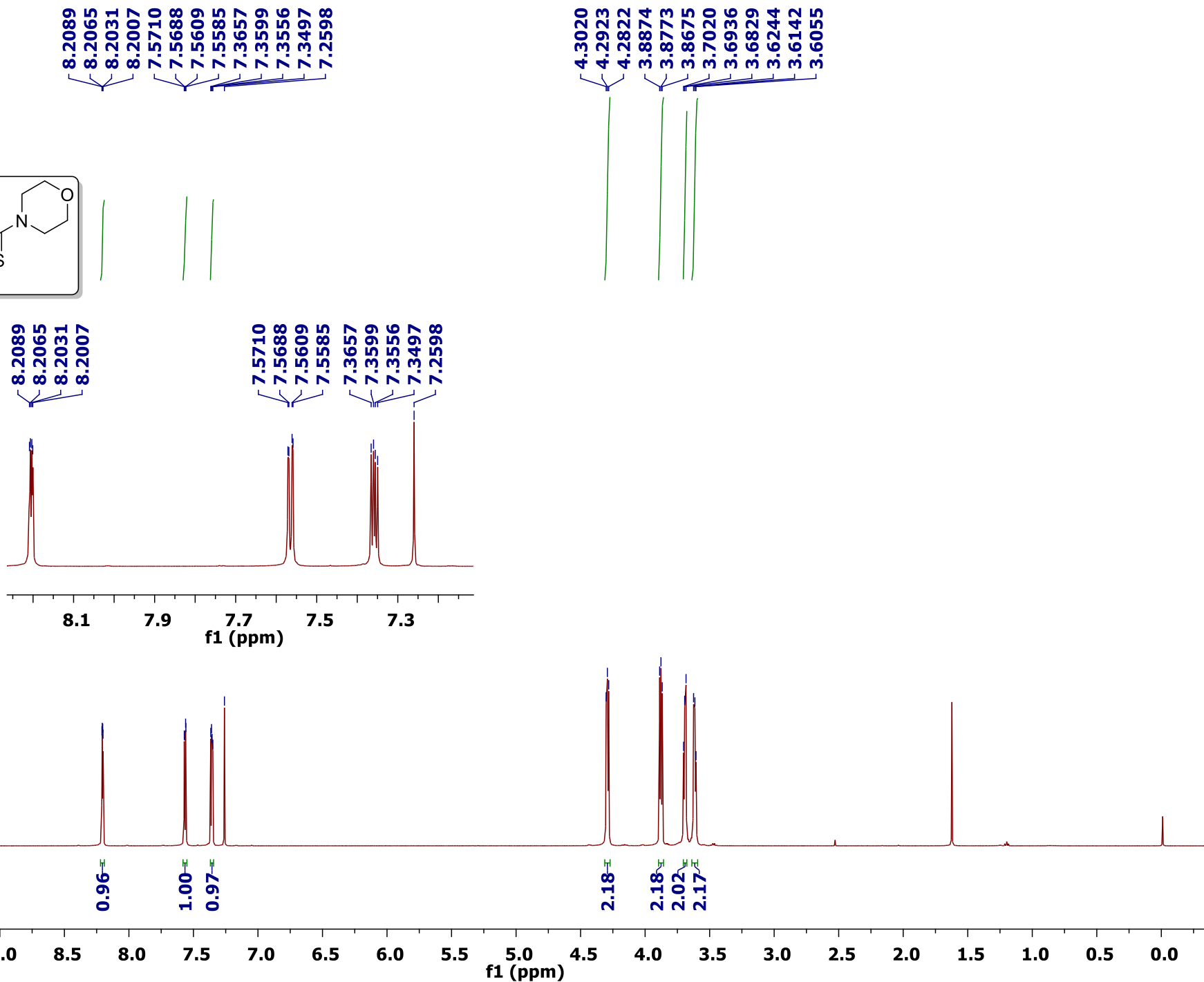
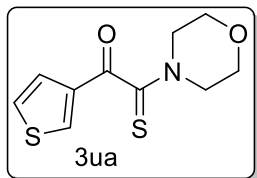
— 66.4115

— 52.1018

— 47.3943



<sup>1</sup>H NMR for compound 3ua (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ua (125 MHz,  $\text{CDCl}_3$ )

— 195.6206

— 182.2842

— 138.4881

— 135.8775

— 127.6266

— 127.2829

— 77.4121

— 77.1580

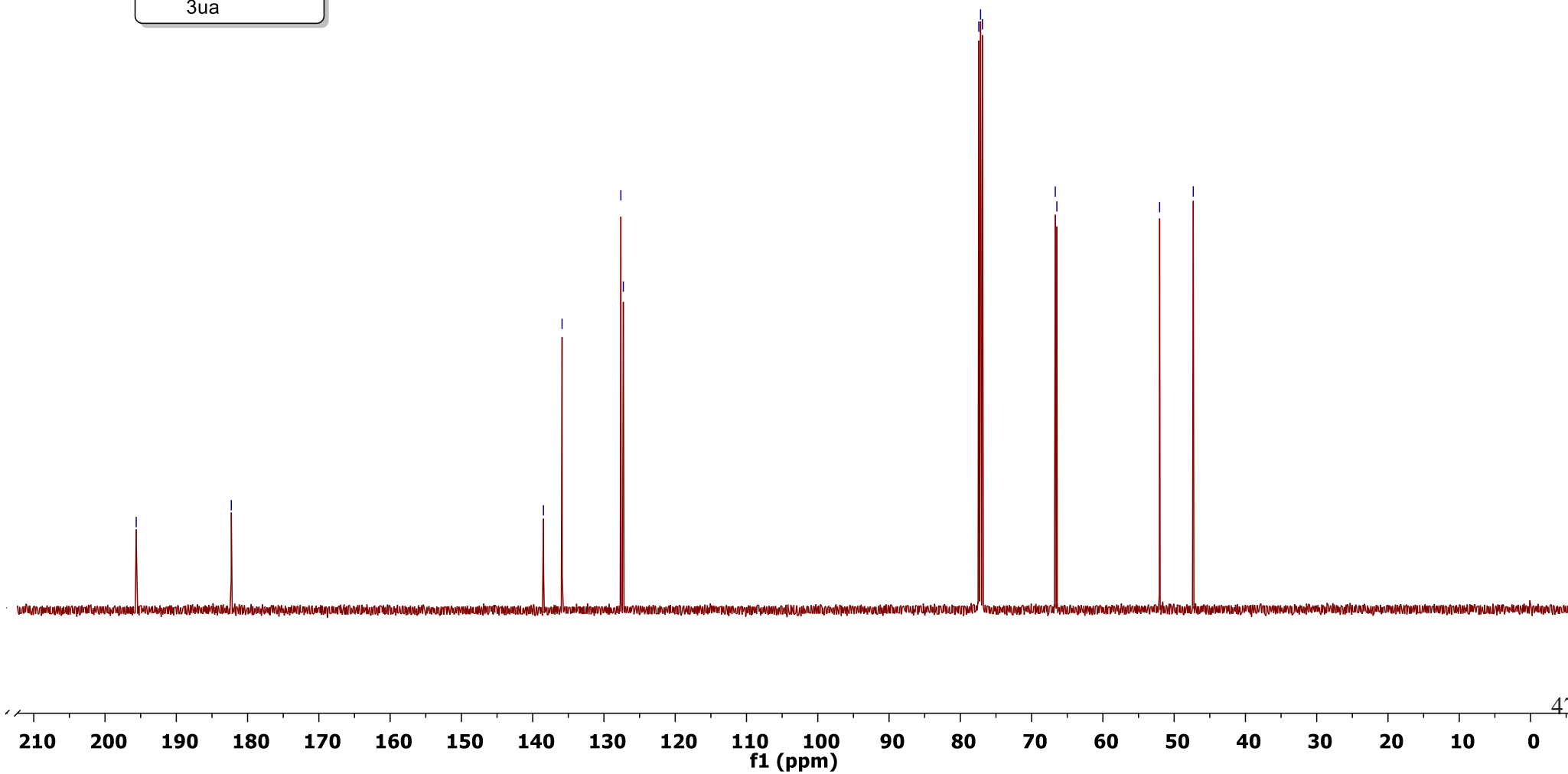
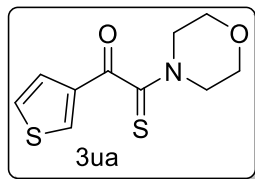
— 76.9039

— 66.6761

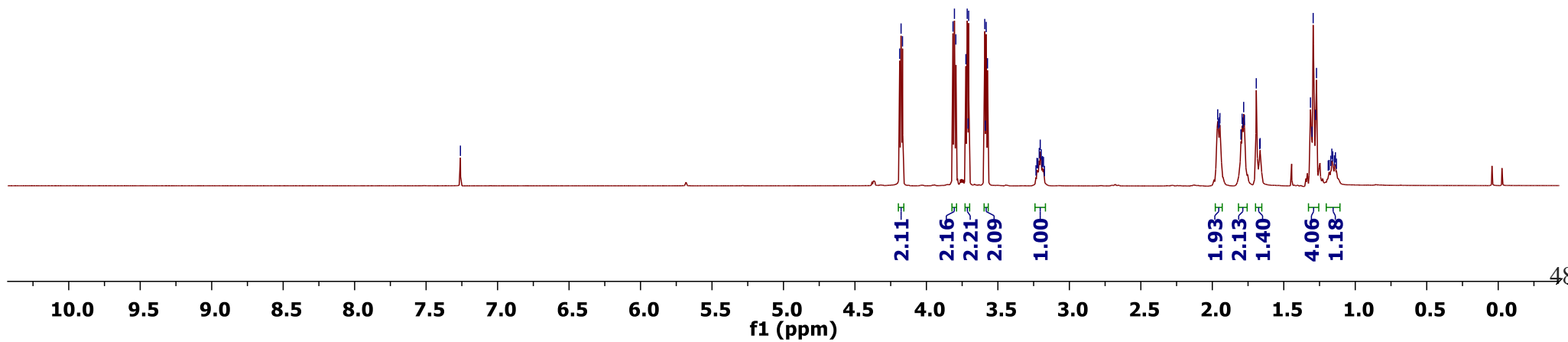
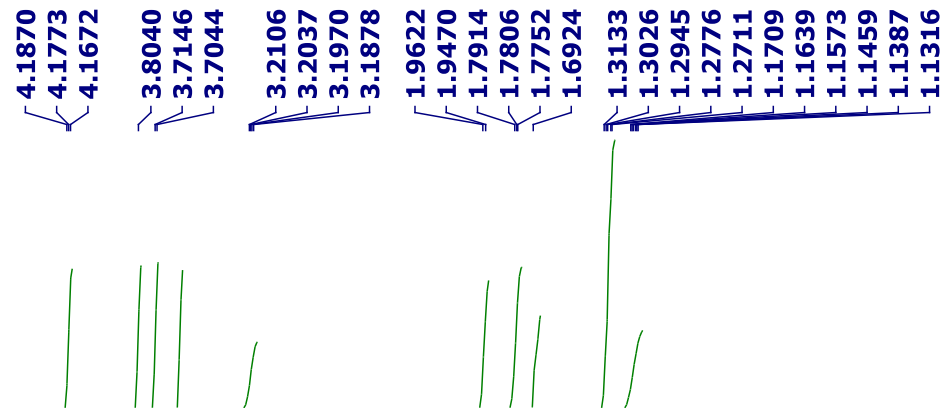
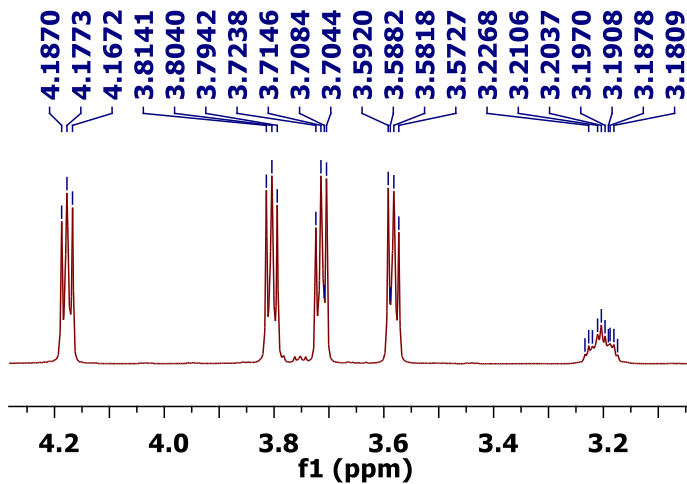
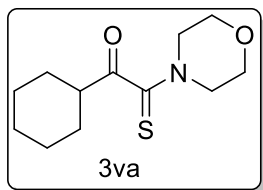
— 66.4562

— 52.0374

— 47.3178



<sup>1</sup>H NMR for compound 3va (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3va (125 MHz,  $\text{CDCl}_3$ )

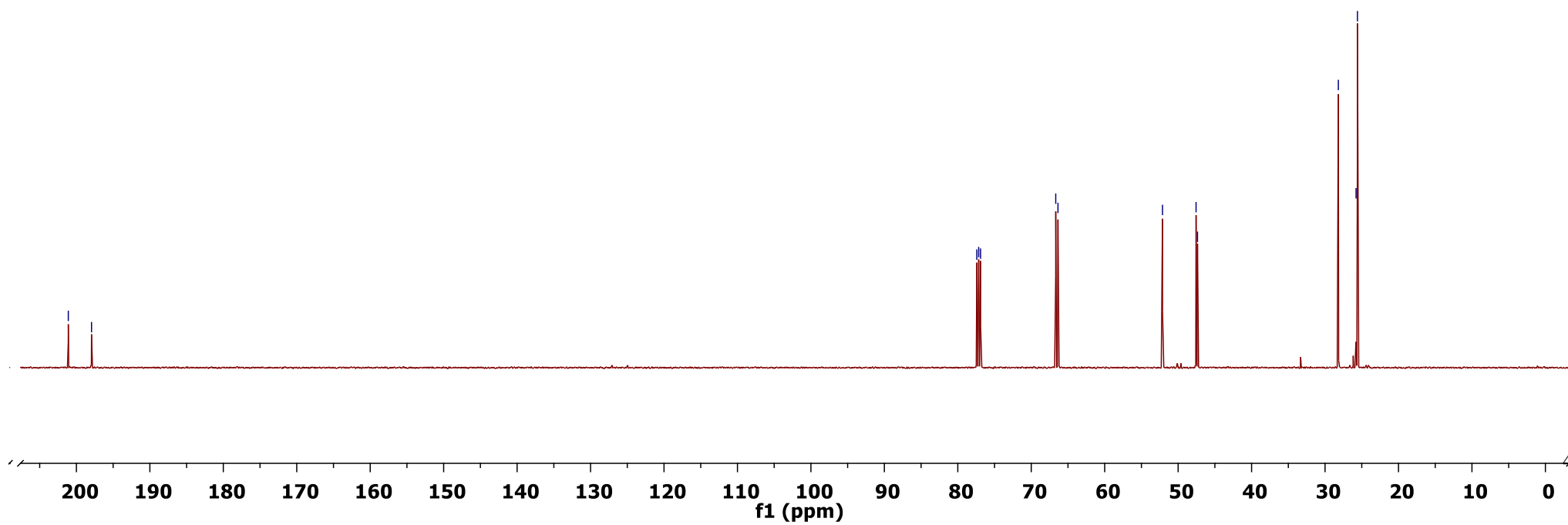
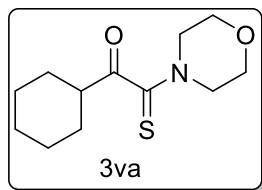
— 201.1021  
— 197.9417

{ 77.4120  
{ 77.1577  
{ 76.9035

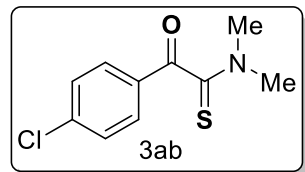
{ 66.6649  
{ 66.3644

{ 52.1321  
{ 47.5601  
{ 47.3644

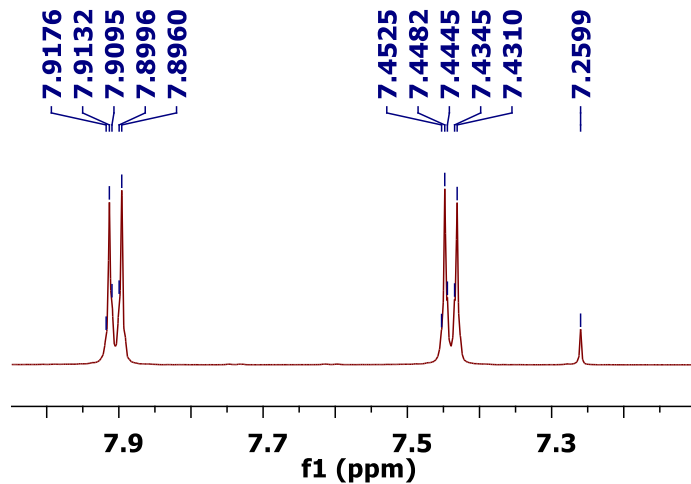
{ 28.1882  
{ 25.7786  
{ 25.5887



<sup>1</sup>H NMR for compound 3ab (500 MHz, CDCl<sub>3</sub>)



7.9176  
7.9132  
7.9095  
7.8996  
7.8960  
7.4525  
7.4482  
7.4445  
7.4345  
7.4310  
7.2599



2.00

1.97

3.00

3.06

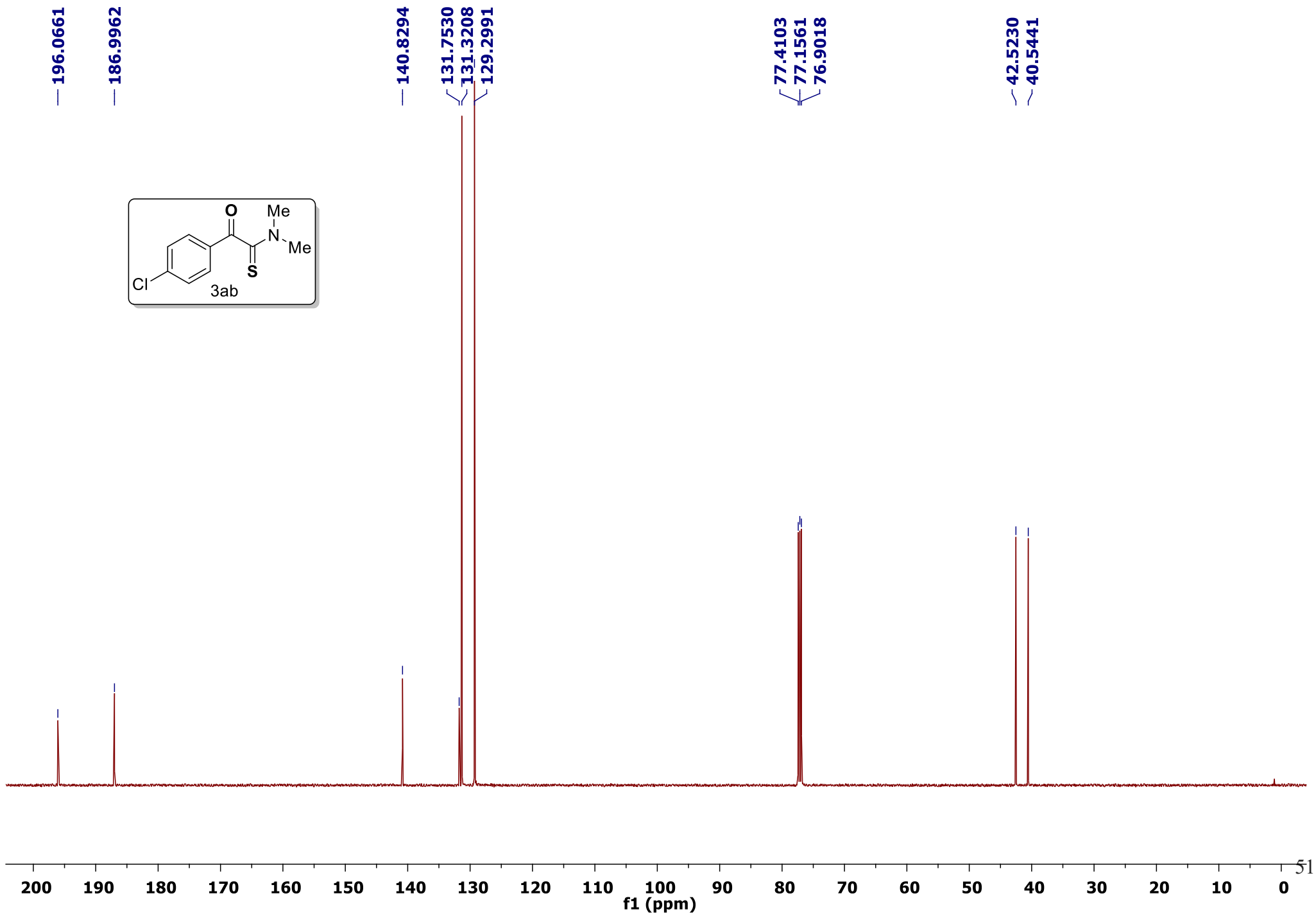
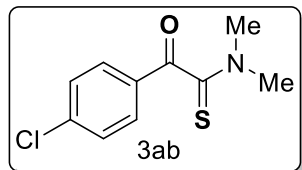
3.5304

3.2093

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

f1 (ppm)

$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ab (125 MHz,  $\text{CDCl}_3$ )

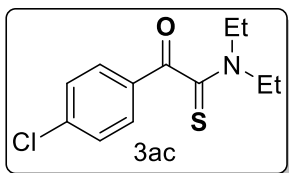


<sup>1</sup>H NMR for compound 3ac (500 MHz, CDCl<sub>3</sub>)

7.9112  
7.9066  
7.9029  
7.8932  
7.8894  
7.8848  
7.4410  
7.4364  
7.4327  
7.4229  
7.4192  
7.4146  
7.2599

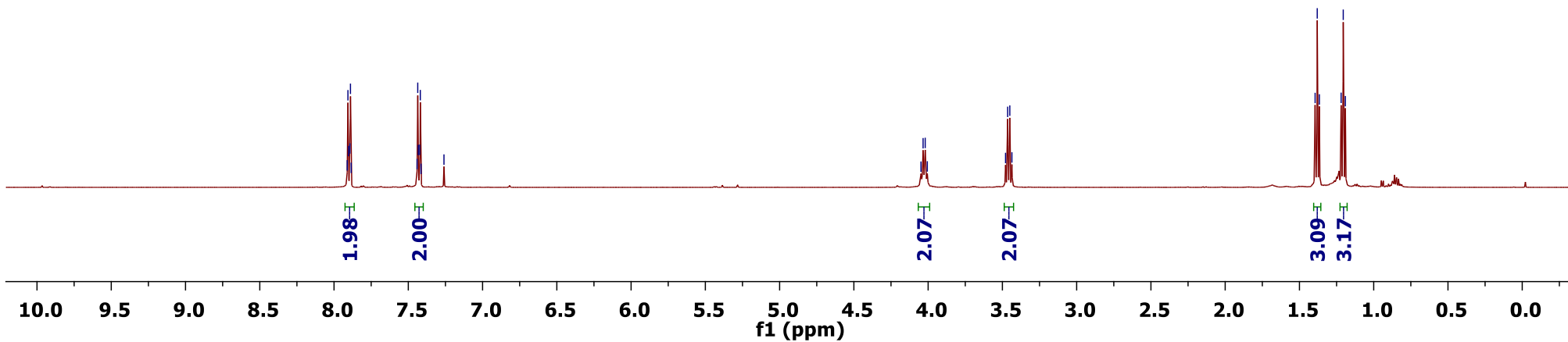
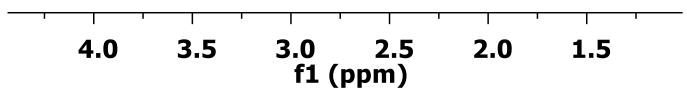
4.0482  
4.0340  
4.0197  
4.0056  
3.4788  
3.4645  
3.4502  
3.4359

1.3946  
1.3803  
1.3659  
1.2194  
1.2051  
1.1908



4.0482  
4.0340  
4.0197  
4.0056  
3.4788  
3.4645  
3.4502  
3.4359

1.3946  
1.3803  
1.3659  
1.2194  
1.2051  
1.1908





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ac (125 MHz,  $\text{CDCl}_3$ )

— 194.9125

— 186.0382

— 140.5682

— 132.0843

— 131.3141

— 129.1833

— 77.4123

— 77.1580

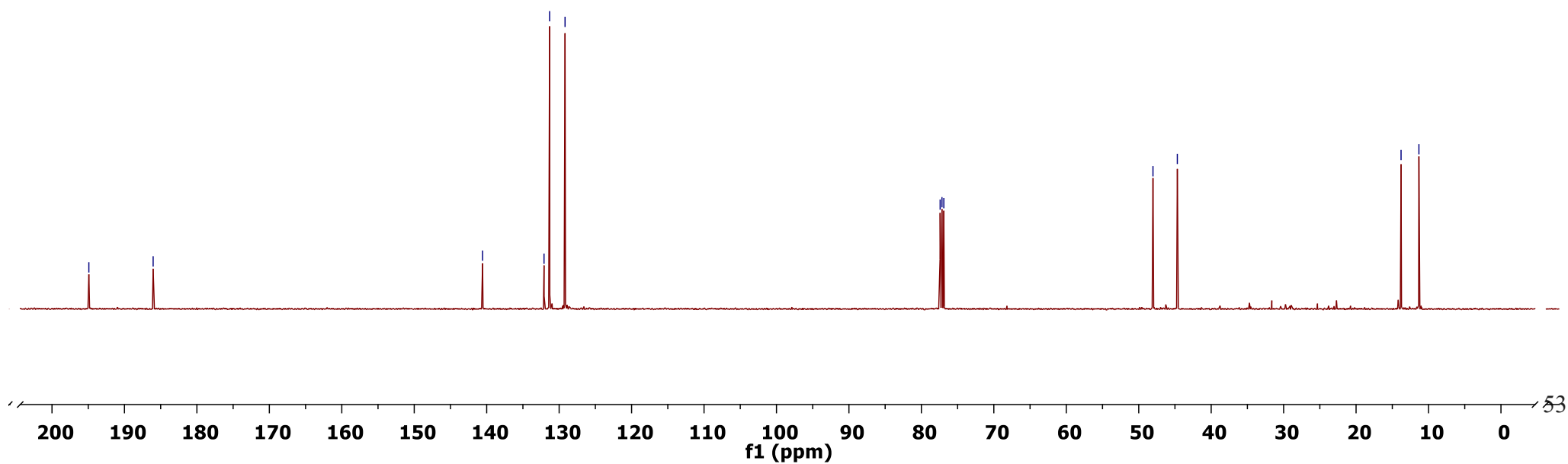
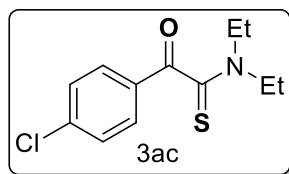
— 76.9038

— 48.0333

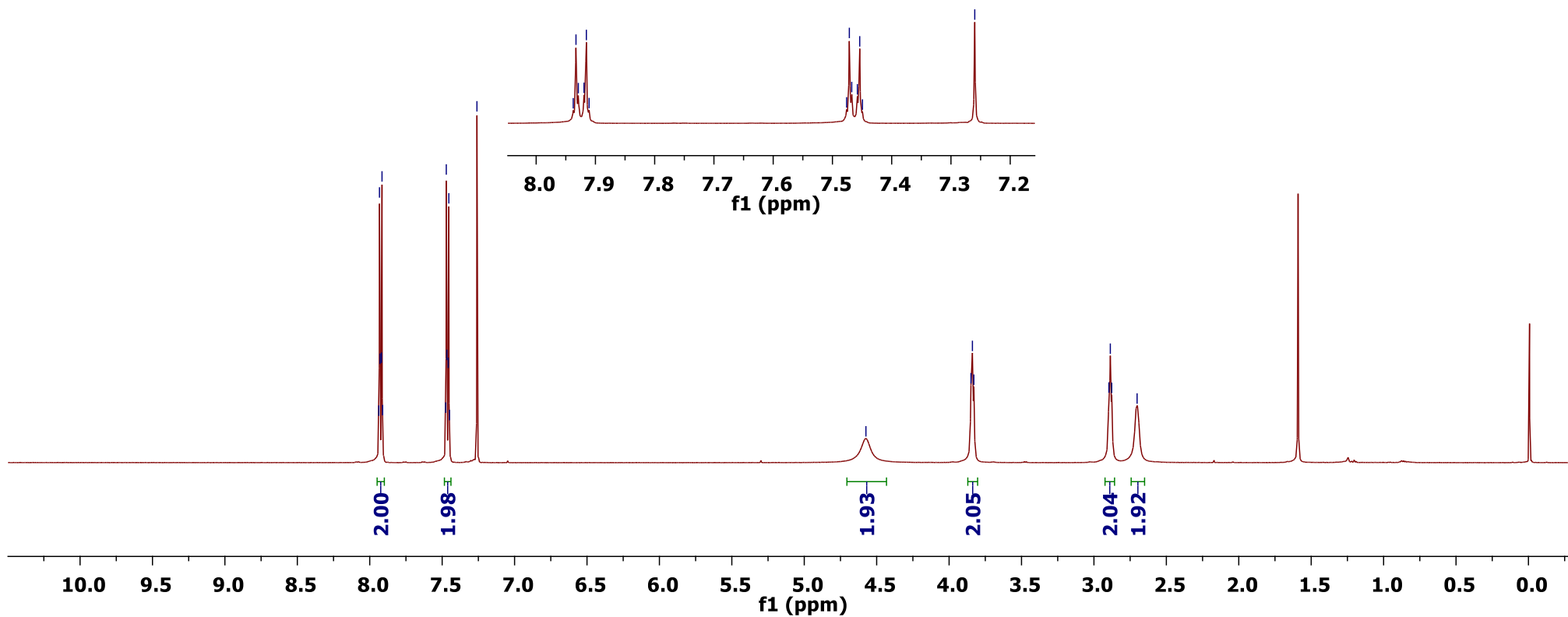
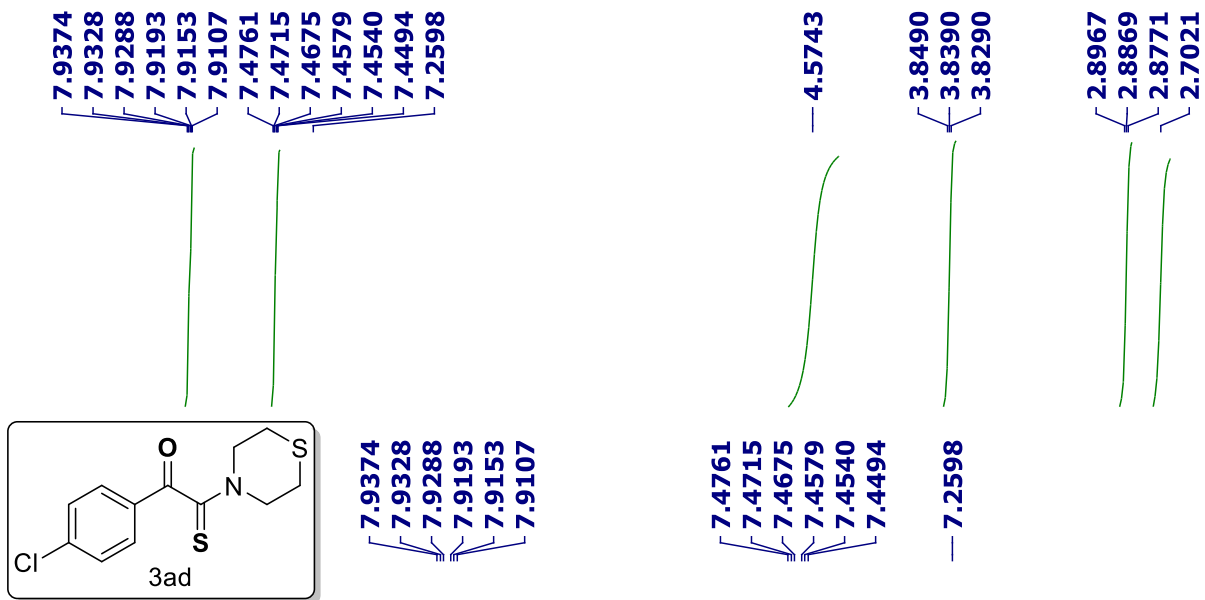
— 44.6727

— 13.7894

— 11.3258



<sup>1</sup>H NMR for compound 3ad (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ad (125 MHz,  $\text{CDCl}_3$ )

— 195.4168

— 186.4013

— 141.1354

— 131.7942

— 131.3712

— 129.4338

— 77.4117

— 77.1576

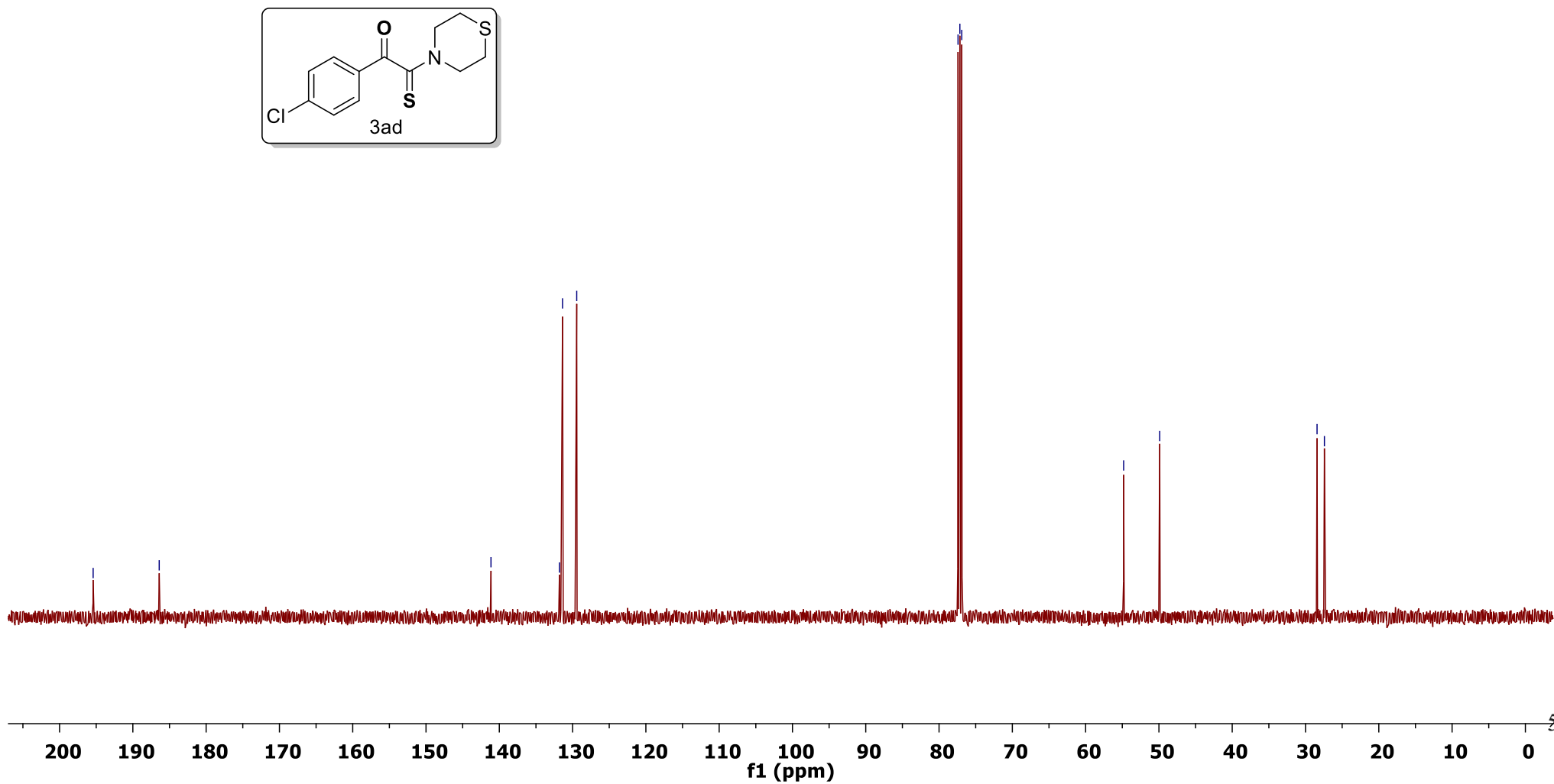
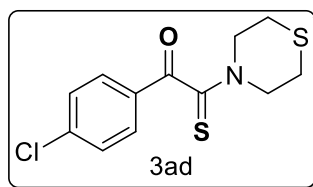
— 76.9037

— 54.8125

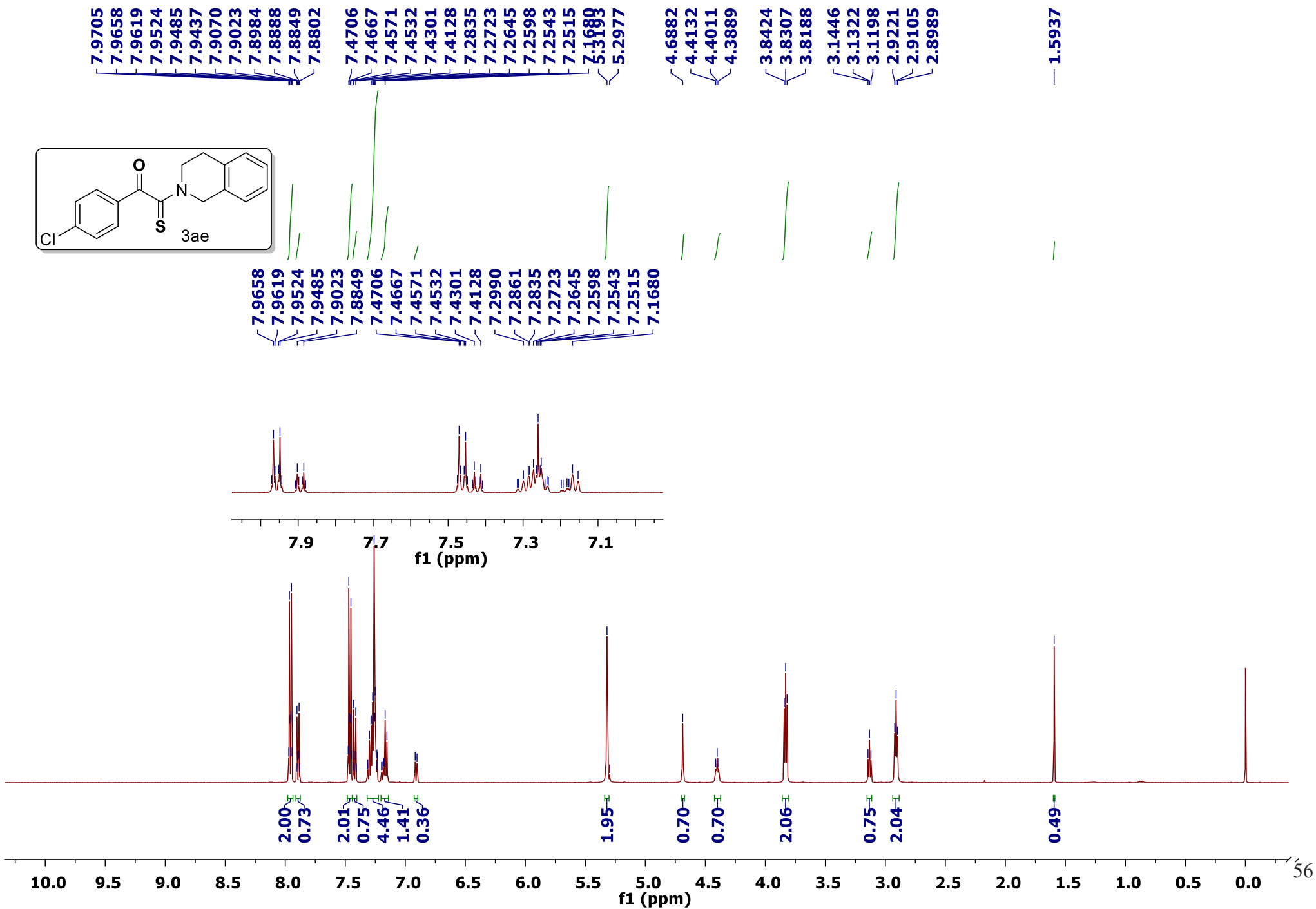
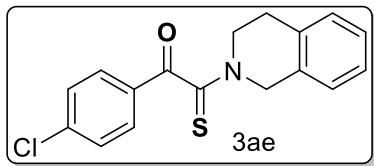
— 49.8935

— 28.4232

— 27.4207



<sup>1</sup>H NMR for compound 3ae (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ae (125 MHz,  $\text{CDCl}_3$ )

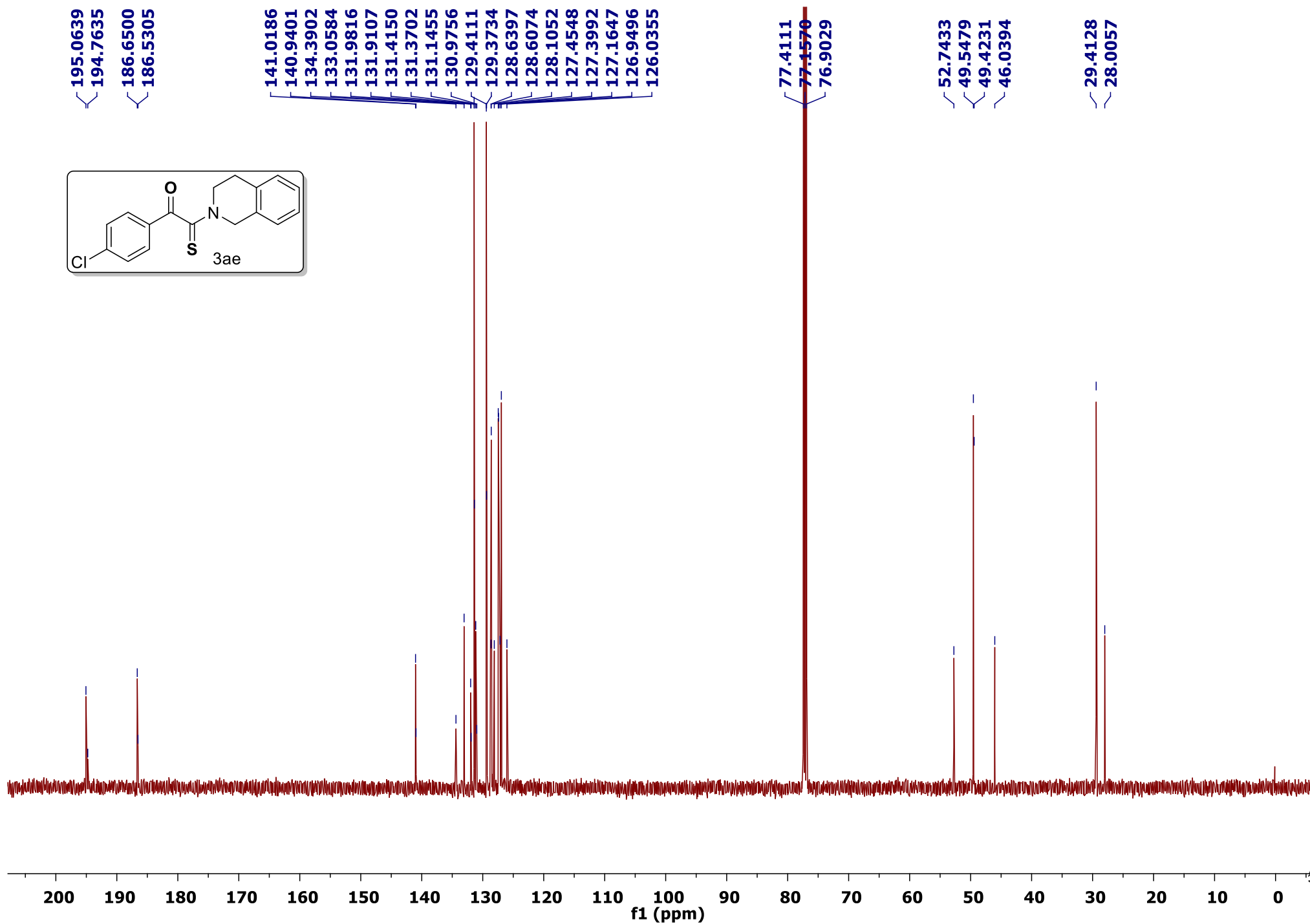
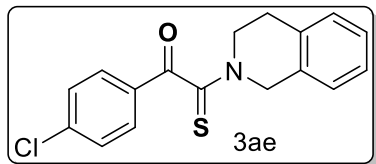
195.0639  
194.7635  
186.6500  
186.5305

141.0186  
140.9401  
134.3902  
133.0584  
131.9816  
131.9107  
131.4150  
131.3702  
131.1455  
130.9756  
129.4111  
129.3734  
128.6397  
128.6074  
128.1052  
127.4548  
127.3992  
127.1647  
126.9496  
126.0355

77.4111  
77.1570  
76.9029

52.7433  
49.5479  
49.4231  
46.0394

29.4128  
28.0057

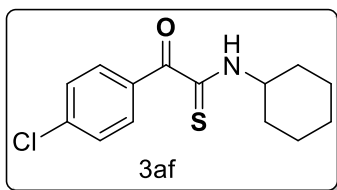


<sup>1</sup>H NMR for compound 3af (500 MHz, CDCl<sub>3</sub>)

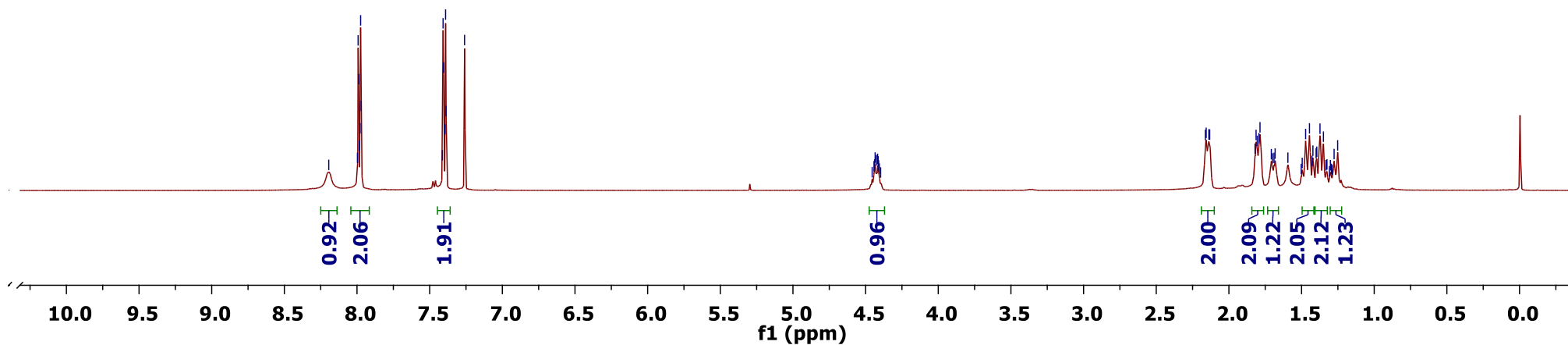
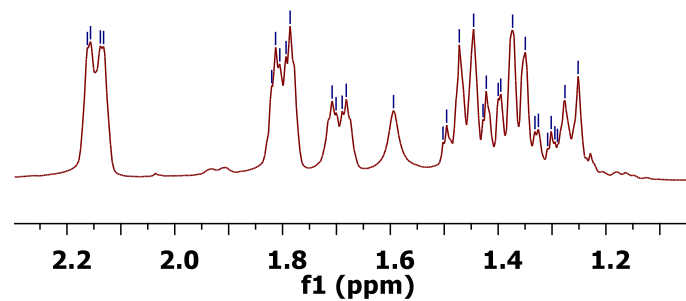
8.1946  
7.9971  
7.9925  
7.9888  
7.9850  
7.9790  
7.9752  
7.9715  
7.4126  
7.4078  
7.4041  
7.3944  
7.3905  
7.3873  
7.2599

4.4550  
4.4422  
4.4383  
4.4343  
4.4258  
4.4174  
4.4138  
4.4099  
4.4055  
4.3966

2.1621  
2.1562  
2.1380  
2.1320  
1.8200  
1.8129  
1.8051  
1.7935  
1.7859  
1.7080  
1.7007  
1.6894  
1.6818  
1.5941  
1.4720  
1.4455  
1.4219  
1.3999  
1.3951  
1.3731  
1.3495  
1.2760  
1.2516



2.1621  
2.1562  
2.1380  
2.1320  
1.8200  
1.8129  
1.8051  
1.7935  
1.7859  
1.7080  
1.6818  
1.5941  
1.4720  
1.4455  
1.4219  
1.3999  
1.3951  
1.3731  
1.3495  
1.2760  
1.2516



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3af (125 MHz,  $\text{CDCl}_3$ )

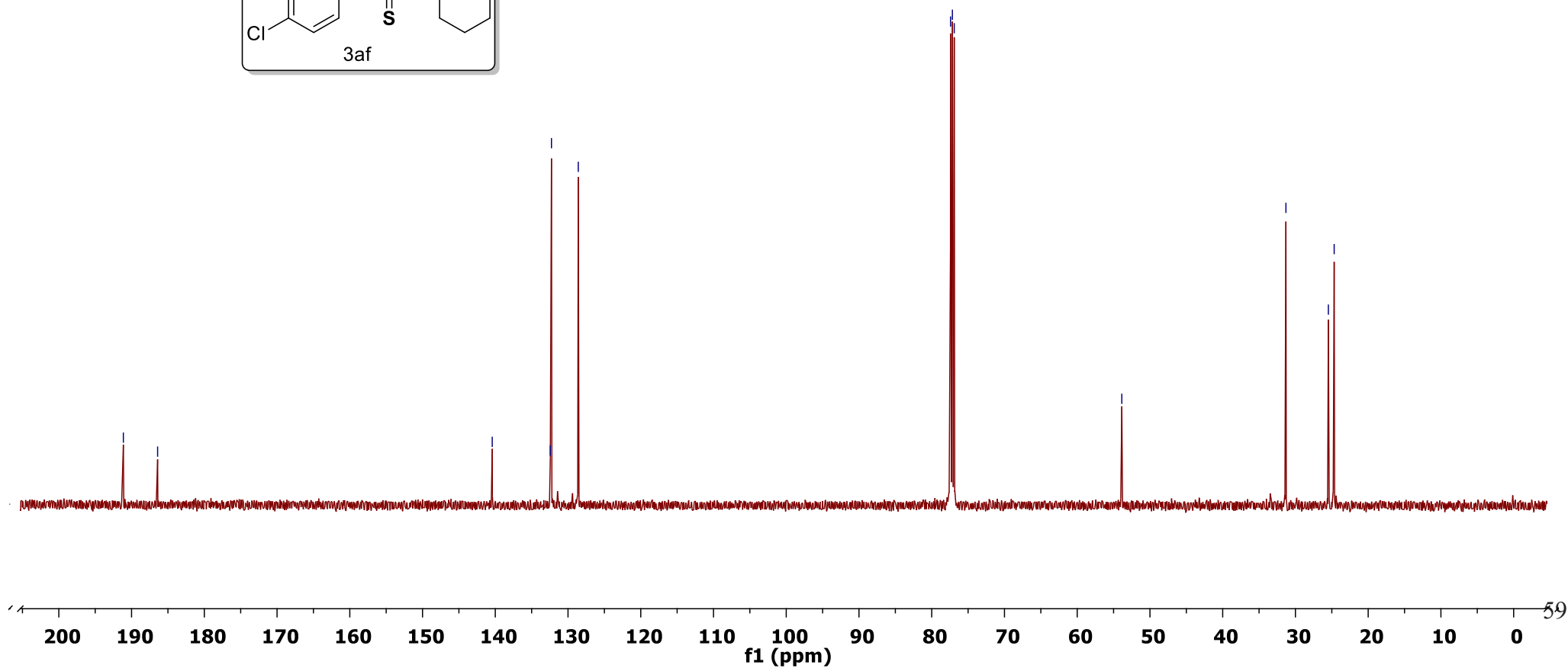
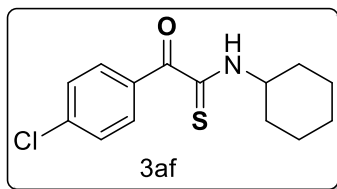
— 191.1277  
— 186.4193

— 140.4191  
— 132.4355  
— 132.2741  
— 128.5891

— 77.4117  
— 77.1576  
— 76.9037

— 53.8698

— 31.3080  
— 25.4713  
— 24.6739



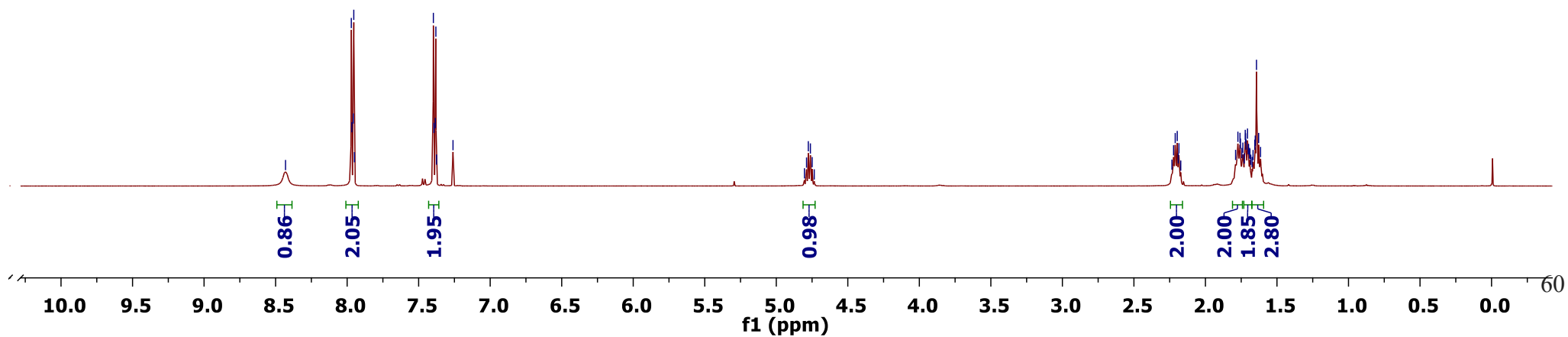
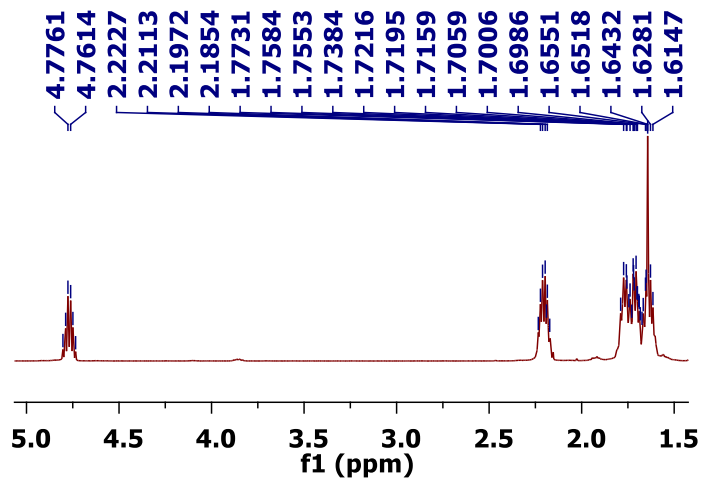
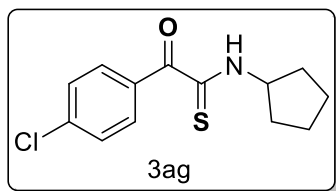
<sup>1</sup>H NMR for compound 3ag (500 MHz, CDCl<sub>3</sub>)

8.4307  
7.9708  
7.9670  
7.9572  
7.9534  
7.3932  
7.3833  
7.3797  
7.3750  
7.2598

4.8032  
4.7884  
4.7761  
4.7614  
4.7492  
4.7346

2.2343  
2.2227  
2.2113  
2.1972  
2.1901  
2.1854  
2.1725

1.7731  
1.7584  
1.7553  
1.7384  
1.7216  
1.7195  
1.7159  
1.7059  
1.7006  
1.6986  
1.6551  
1.6518  
1.6432  
1.6281  
1.6147





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ag (125 MHz,  $\text{CDCl}_3$ )

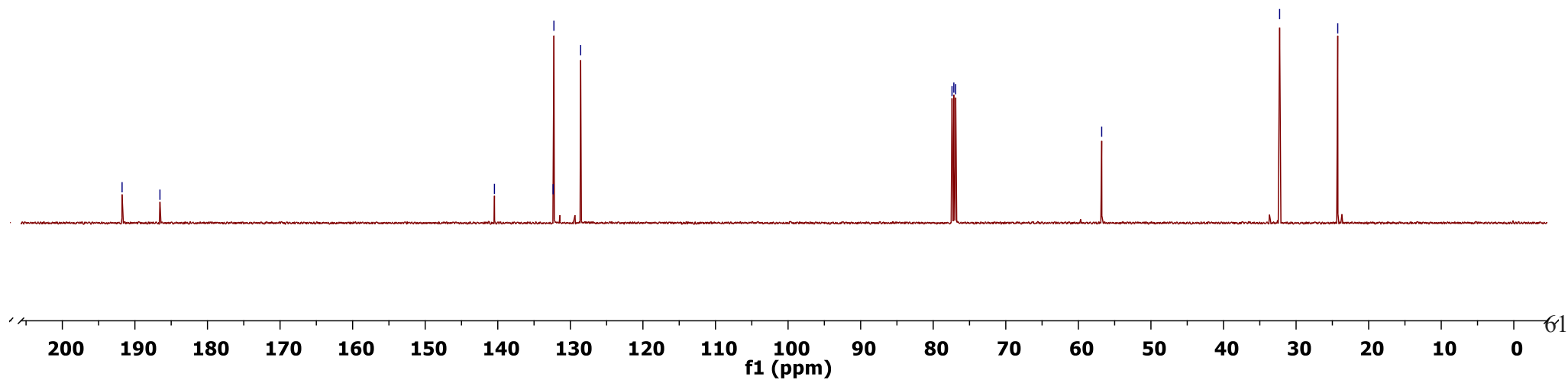
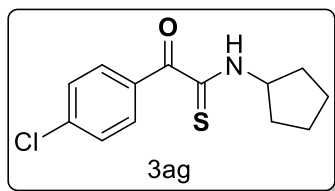
— 191.7661  
— 186.5504

— 140.4596  
{ 132.3731  
{ 132.2642  
{ 128.5805

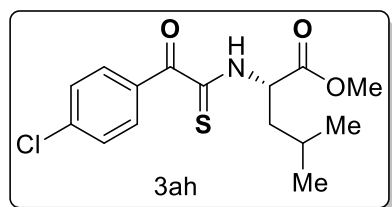
{ 77.4115  
{ 77.1574  
{ 76.9033

— 56.7852

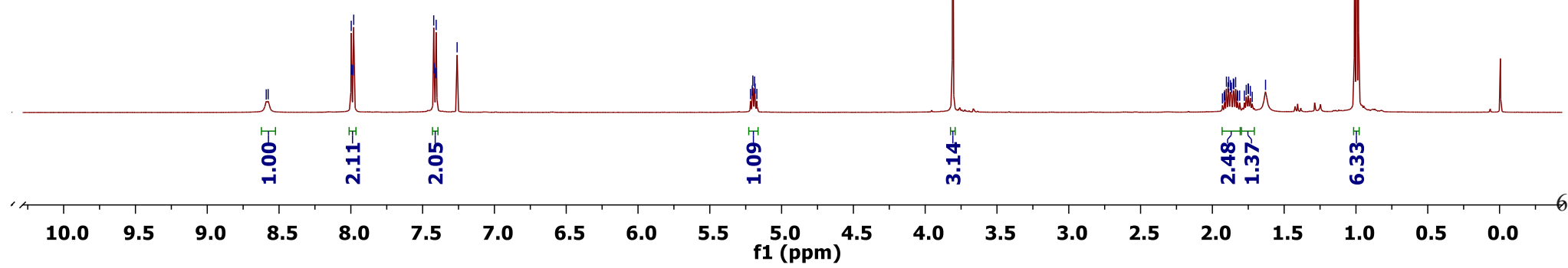
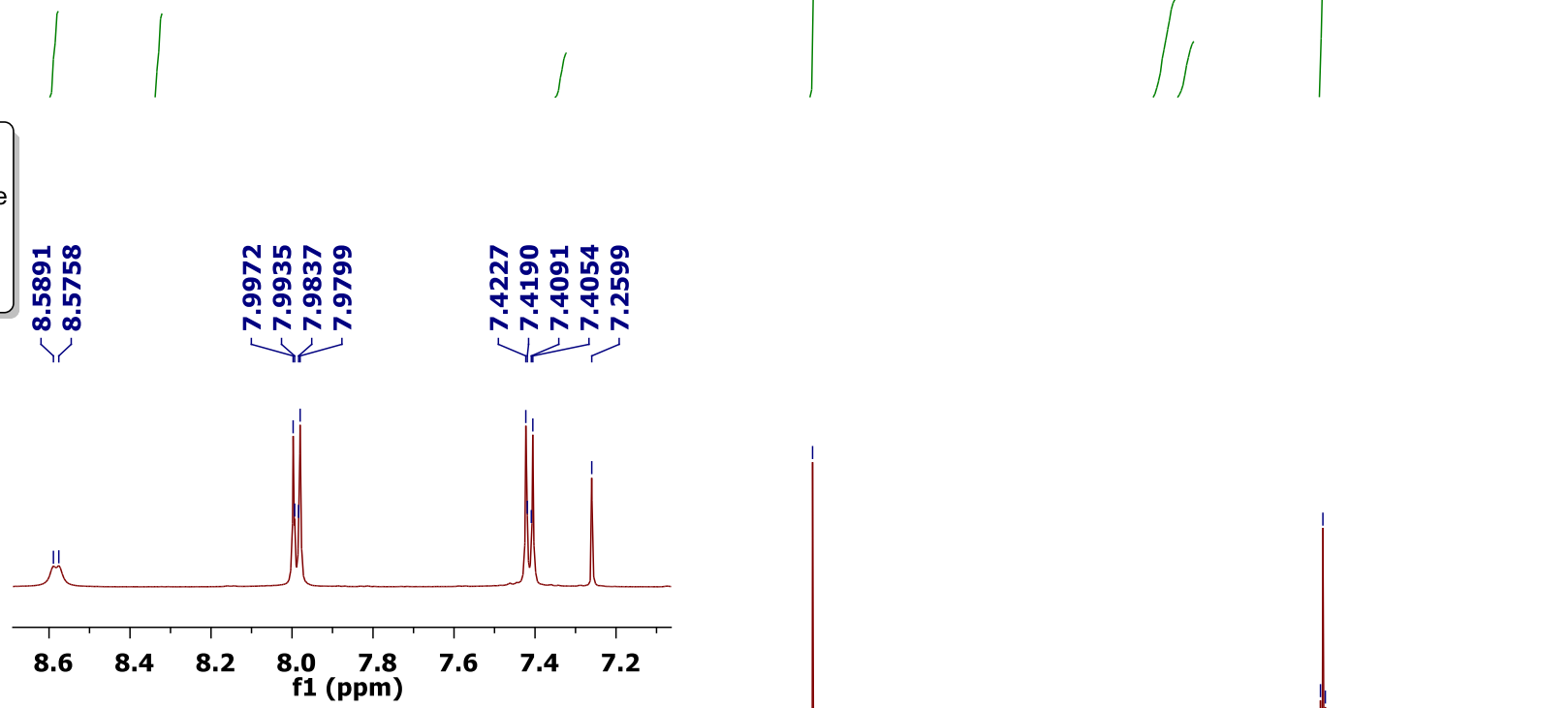
— 32.2687  
— 24.2579



<sup>1</sup>H NMR for compound 3ah (500 MHz, CDCl<sub>3</sub>)



8.5891  
8.5758  
7.9972  
7.9935  
7.9837  
7.9799  
7.4227  
7.4190  
7.4091  
7.4054  
7.2599  
5.2164  
5.2044  
5.2003  
5.1885  
5.1844  
5.1724  
3.8087  
1.9020  
1.8901  
1.8862  
1.8743  
1.8671  
1.8543  
1.8508  
1.8381  
1.8264  
1.7641  
1.7510  
1.7489  
1.7356  
1.6797  
0.9966  
0.9833



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ah (125 MHz,  $\text{CDCl}_3$ )

— 193.3278

— 186.0206

— 171.5546

— 140.6387

{ 132.3223

{ 132.2386

{ 128.6961

{ 77.4139

{ 77.1600

{ 76.9058

— 55.9773

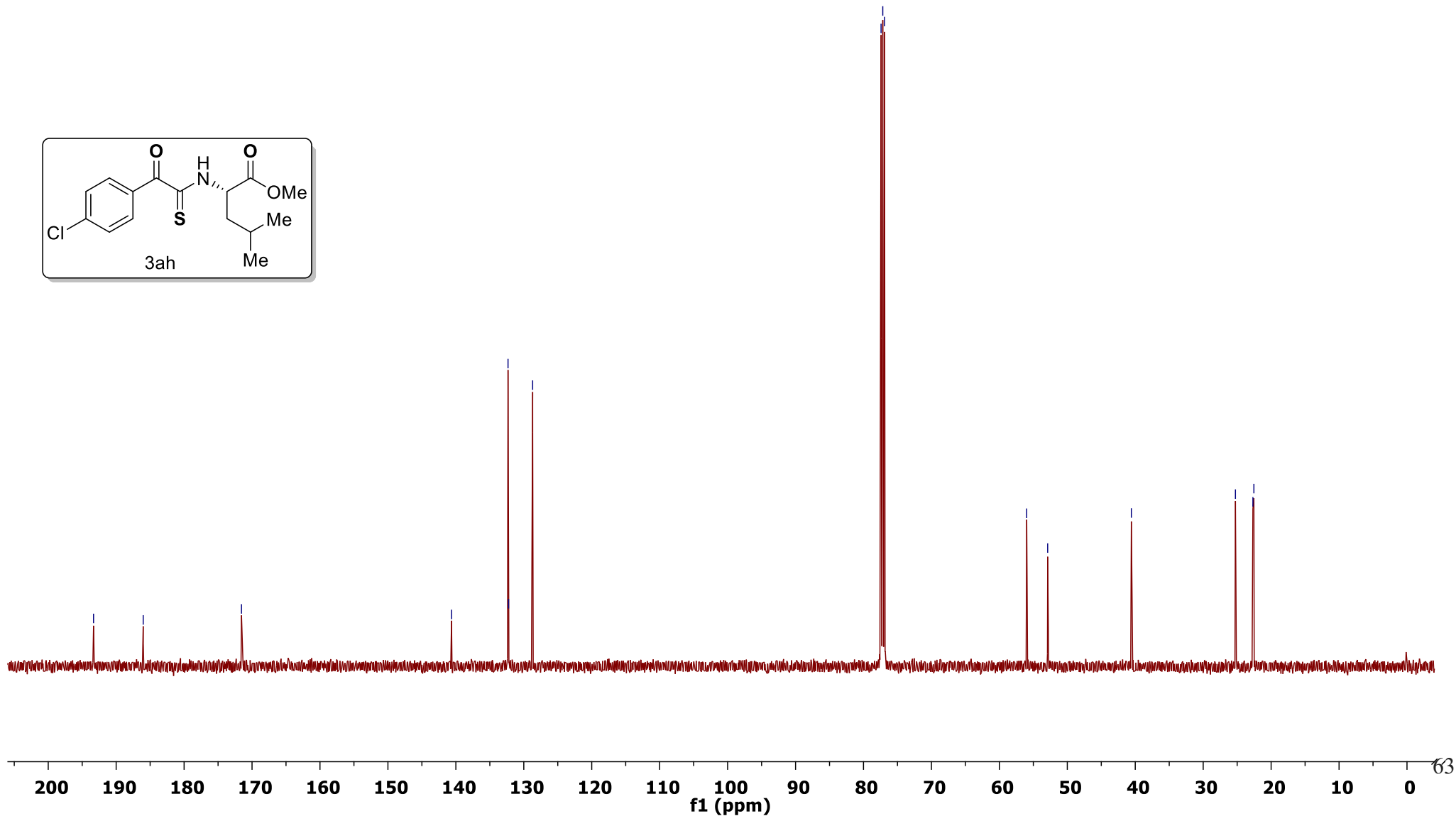
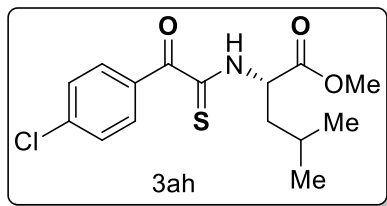
— 52.8886

— 40.5711

{ 25.2539

{ 22.6817

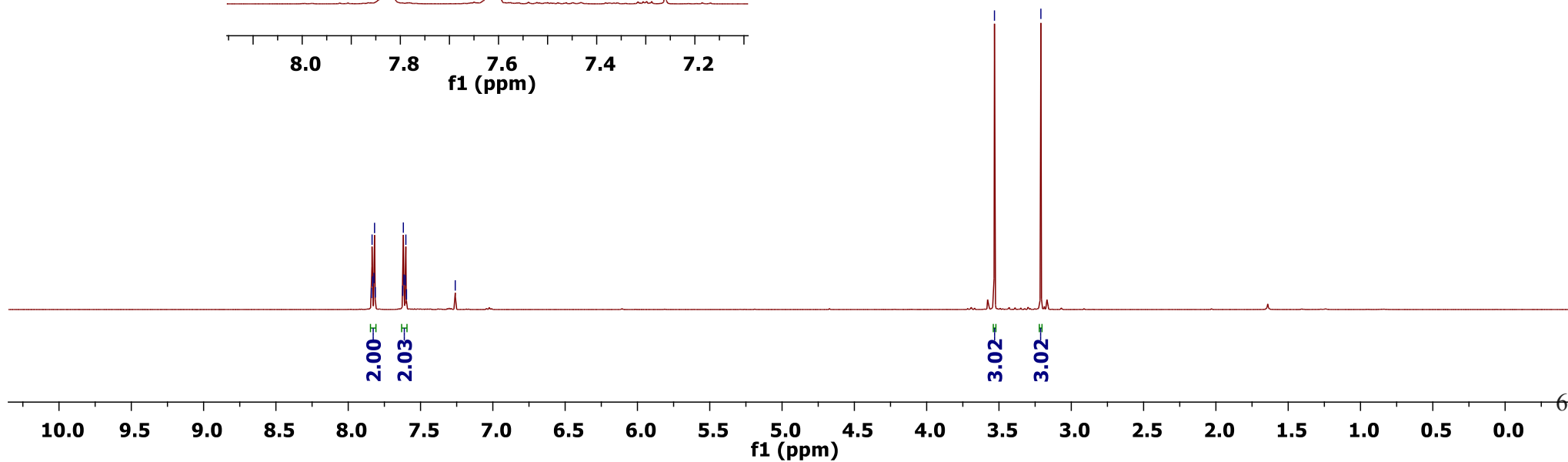
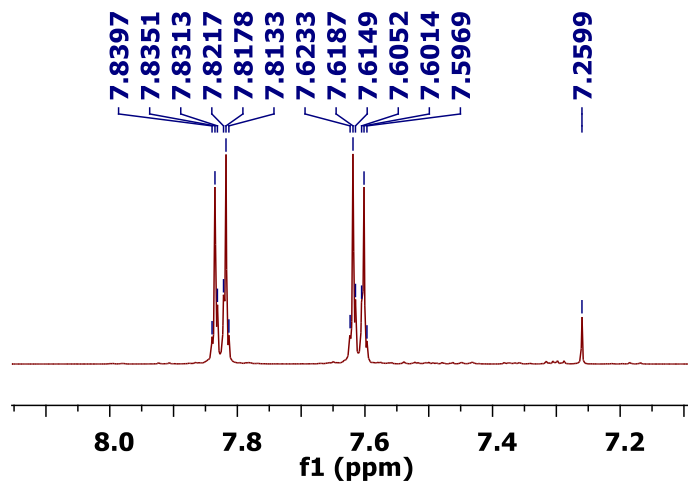
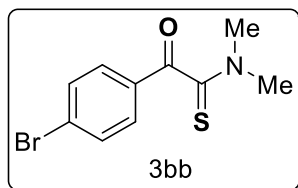
{ 22.5367



<sup>1</sup>H NMR for compound 3bb (500 MHz, CDCl<sub>3</sub>)

7.8397  
7.8351  
7.8313  
7.8217  
7.8178  
7.8133  
7.6233  
7.6187  
7.6149  
7.6052  
7.6014  
7.5969  
7.2599

3.5311  
3.2102



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3bb (125 MHz,  $\text{CDCl}_3$ )

— 195.9828

— 187.1497

132.2938

132.1535

131.3767

129.6937

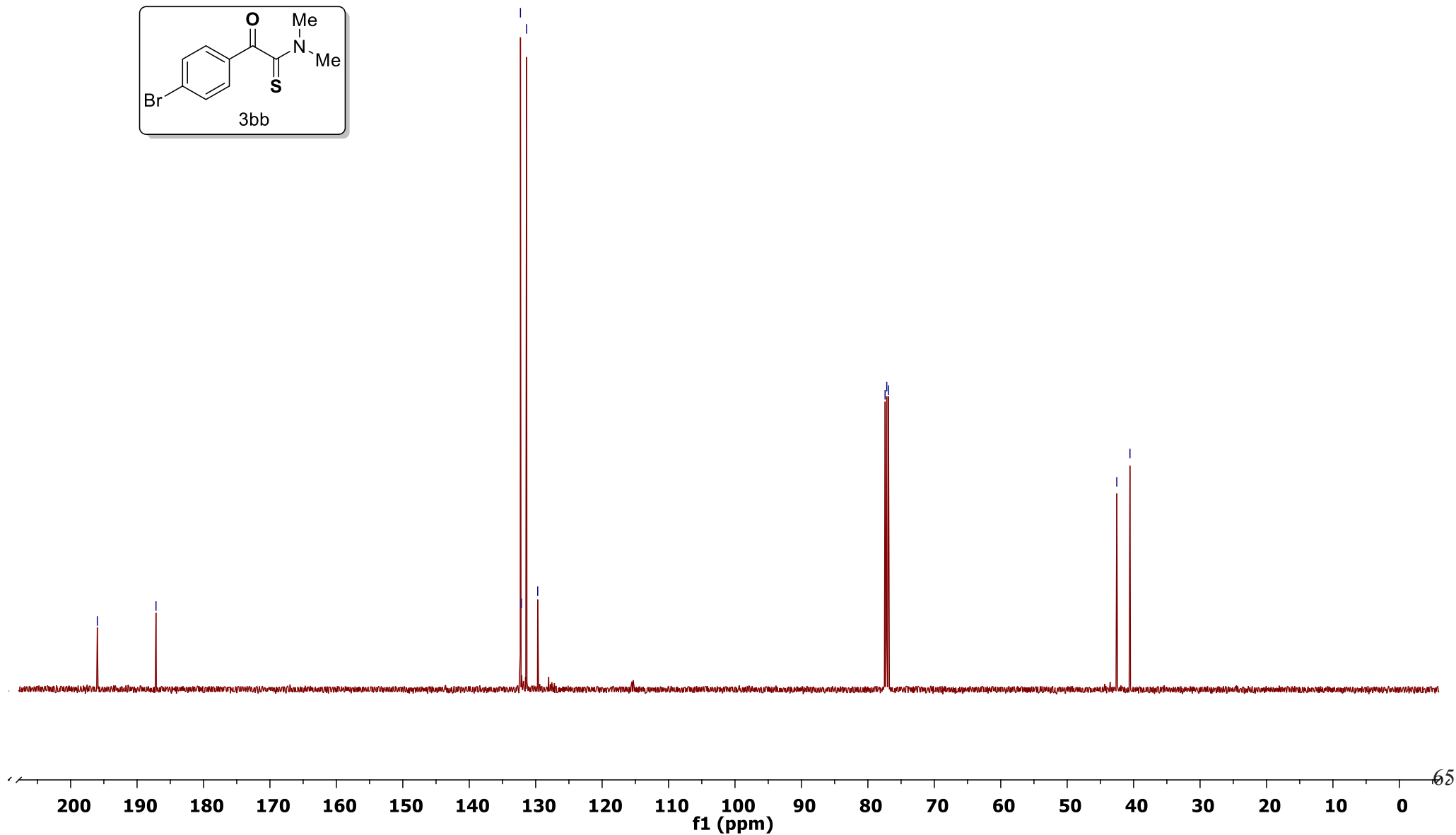
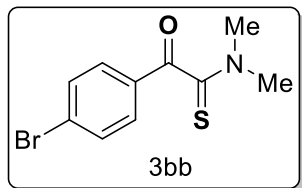
77.4123

77.1580

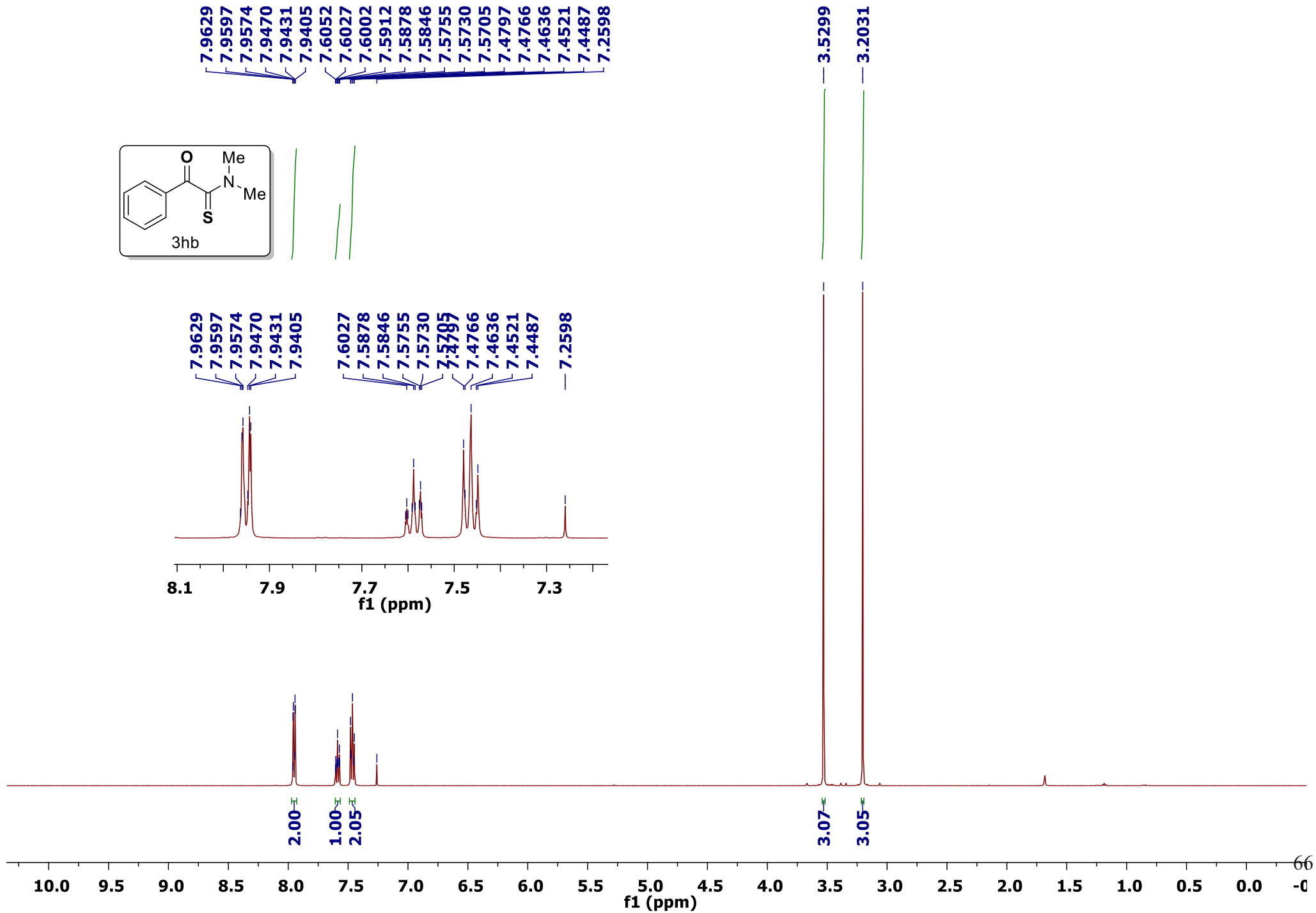
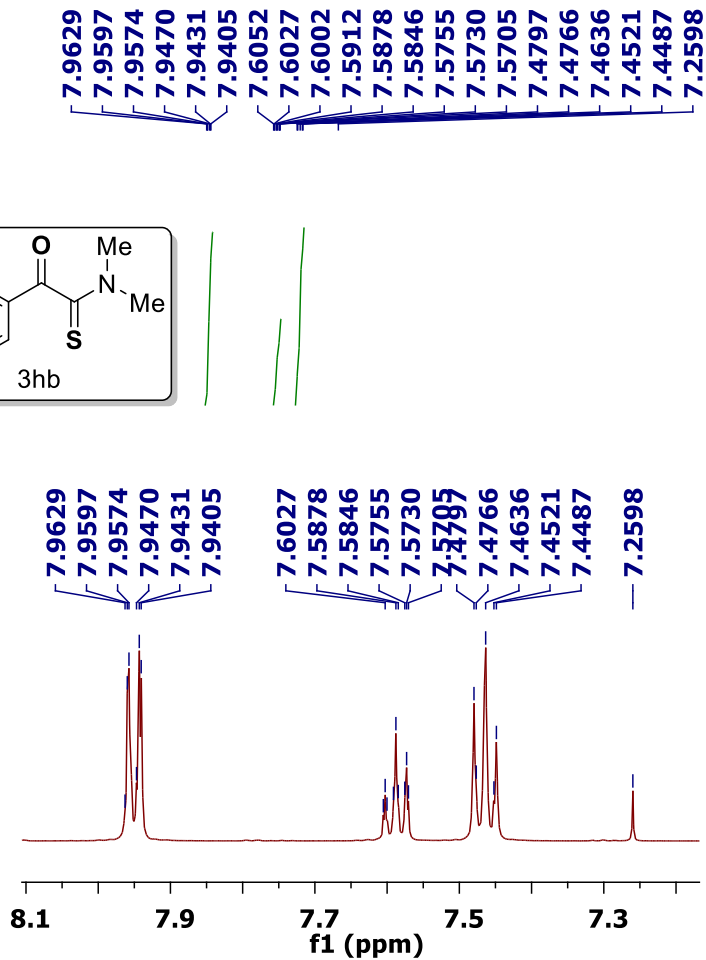
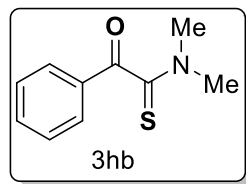
76.9038

42.5375

40.5511



<sup>1</sup>H NMR for compound 3hb (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3hb (125 MHz,  $\text{CDCl}_3$ )

— 196.7548

— 188.4731

— 134.3118

— 133.1802

— 129.9064

— 128.9238

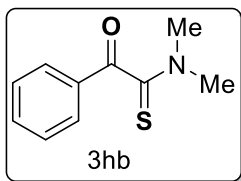
— 77.4109

— 77.1563

— 76.9021

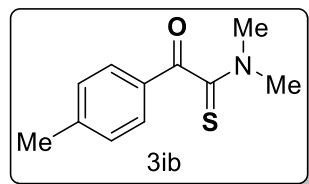
— 42.5183

— 40.4728



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

<sup>1</sup>H NMR for compound 3ib (500 MHz, CDCl<sub>3</sub>)



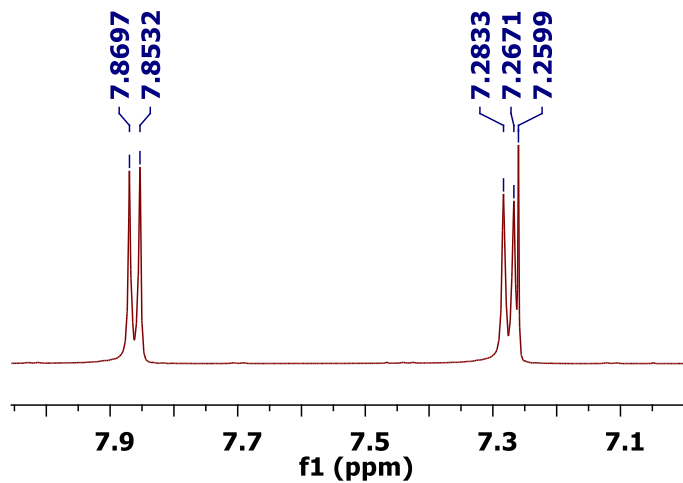
7.8697  
7.8532

7.2833  
7.2671  
7.2599

3.5449

3.2149

2.4187



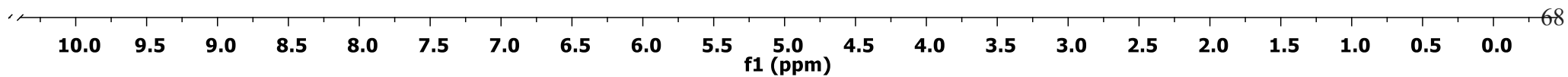
2.00

2.05

3.06

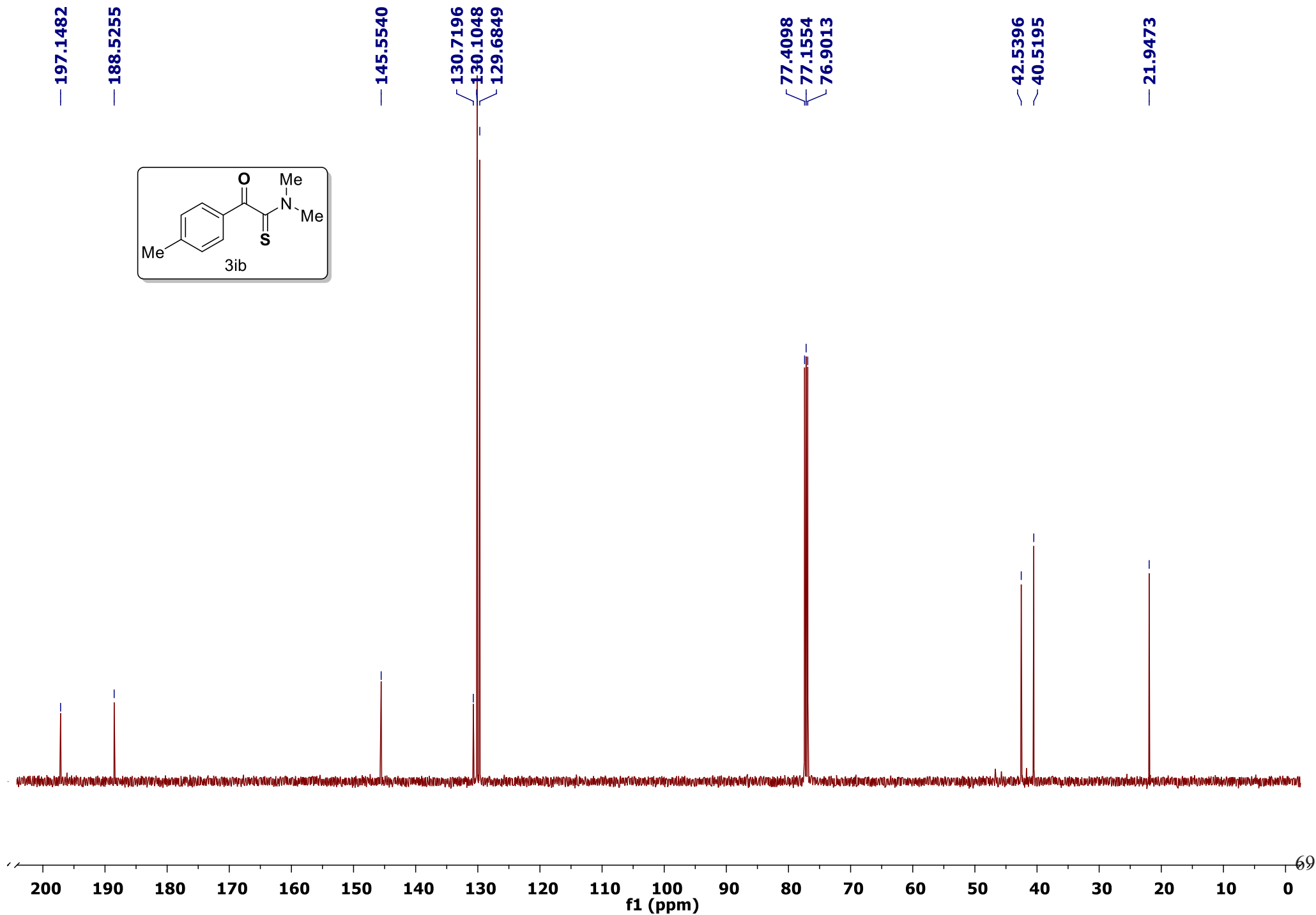
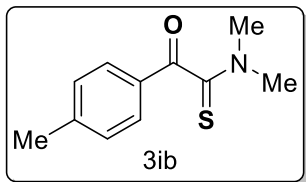
2.92

3.06

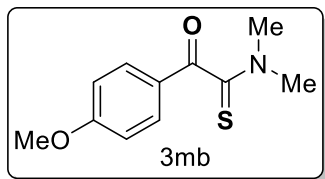




$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ib (125 MHz,  $\text{CDCl}_3$ )

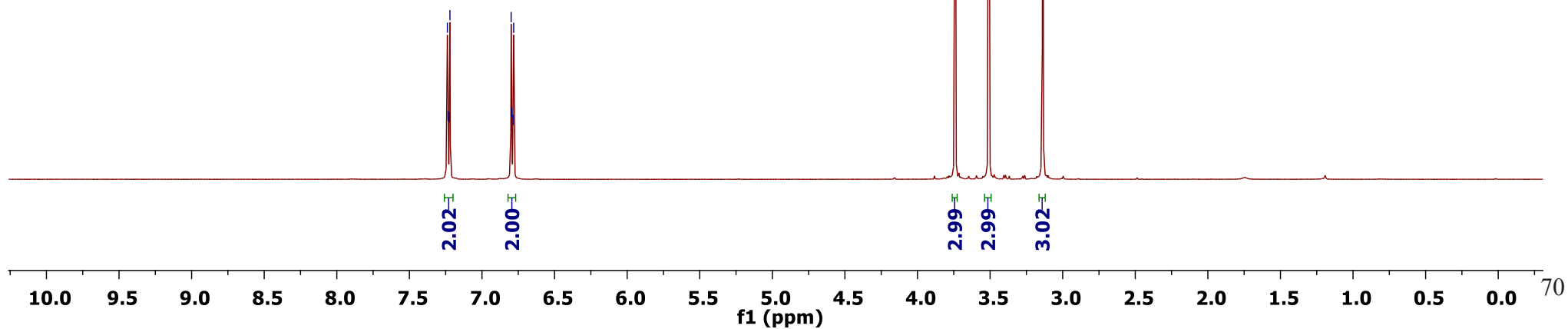
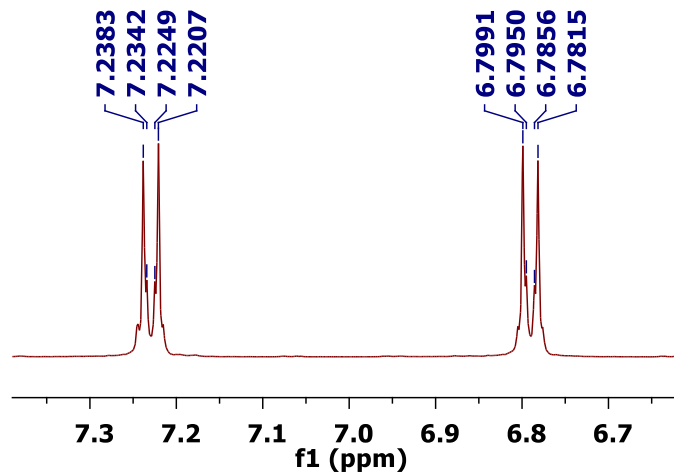


<sup>1</sup>H NMR for compound 3mb (500 MHz, CDCl<sub>3</sub>)



7.2383  
7.2342  
7.2249  
7.2207  
6.7991  
6.7950  
6.7856  
6.7815

3.7412  
3.5093  
3.1376



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3mb (125 MHz,  $\text{CDCl}_3$ )

— 197.2851

— 187.9147

— 164.5668

— 132.4345

— 126.0317

— 114.2835

{ 77.4111

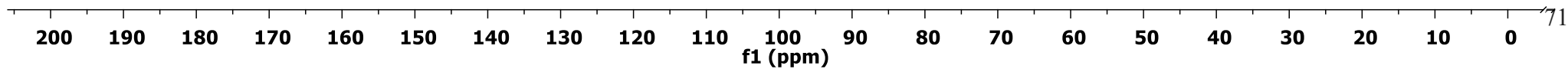
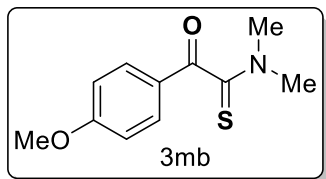
{ 77.1569

{ 76.9026

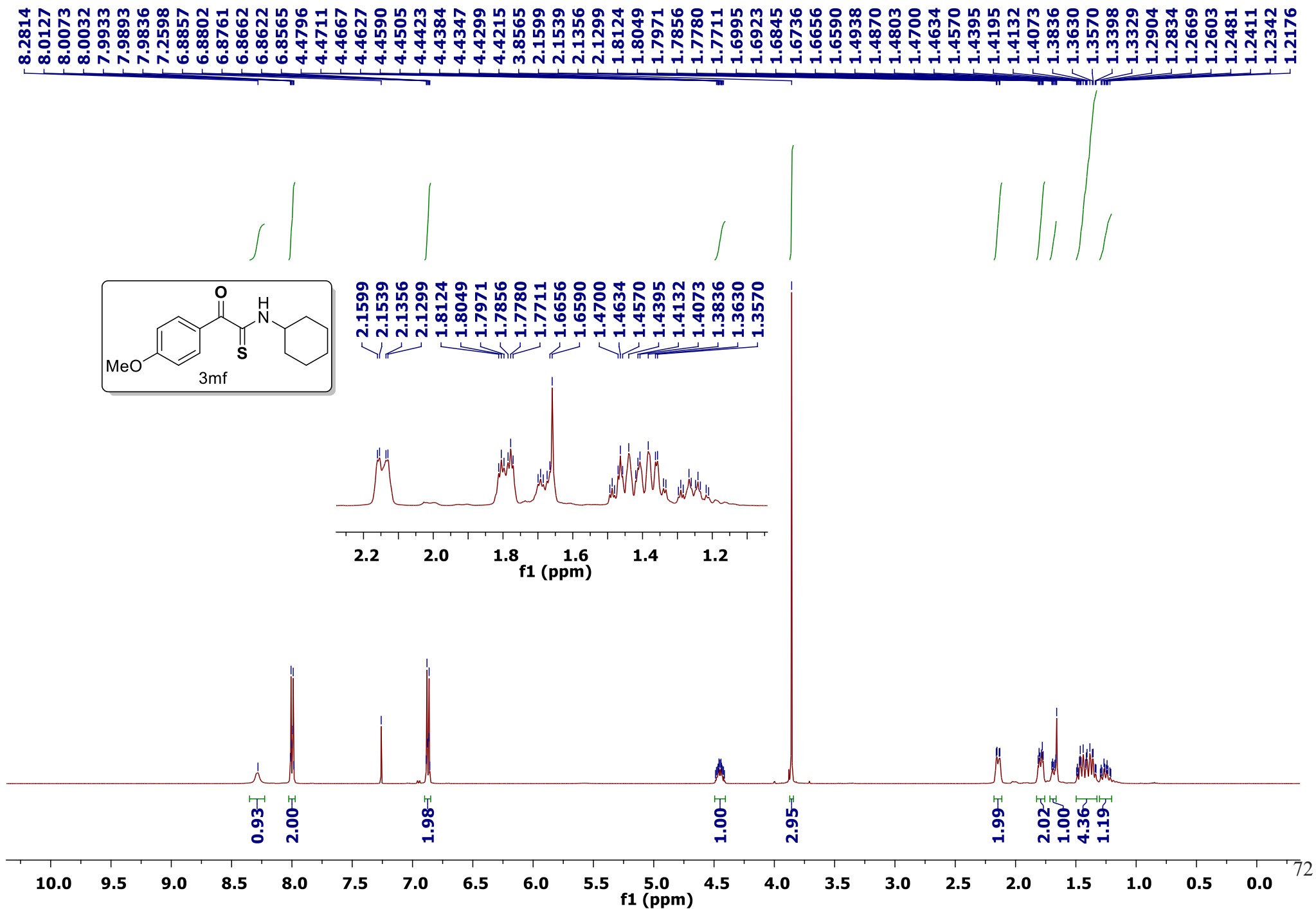
— 55.7376

~ 42.5529

~ 40.5722



<sup>1</sup>H NMR for compound 3mf (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3mf (125 MHz,  $\text{CDCl}_3$ )

— 193.3920

— 187.3758

— 164.3635

— 133.3806

— 126.3262

— 113.6786

77.4108

77.1567

76.9024

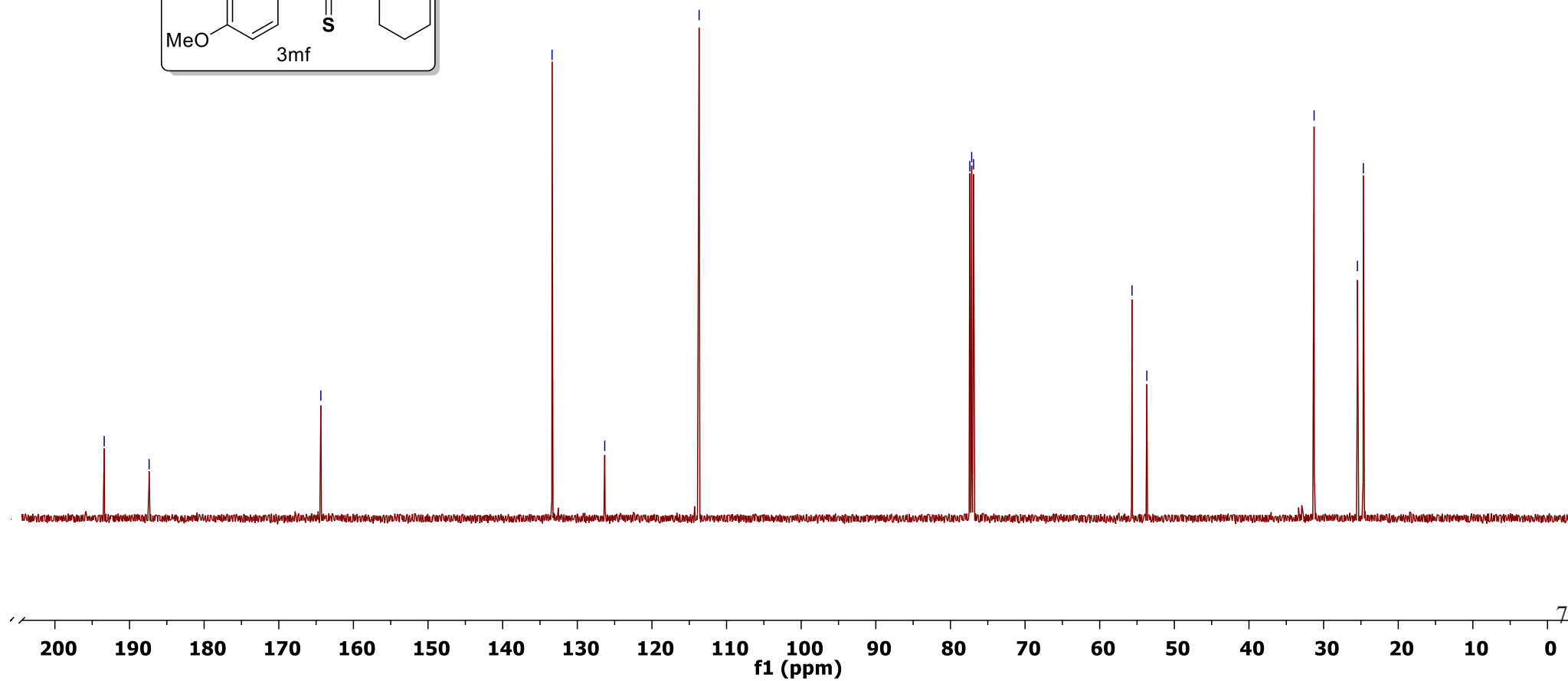
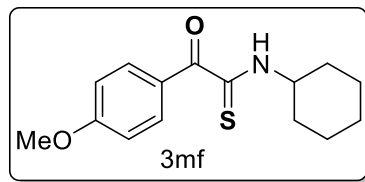
55.6701

53.6861

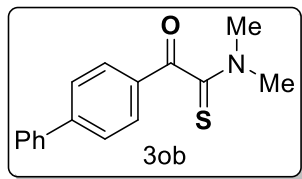
31.2746

25.4689

24.6757

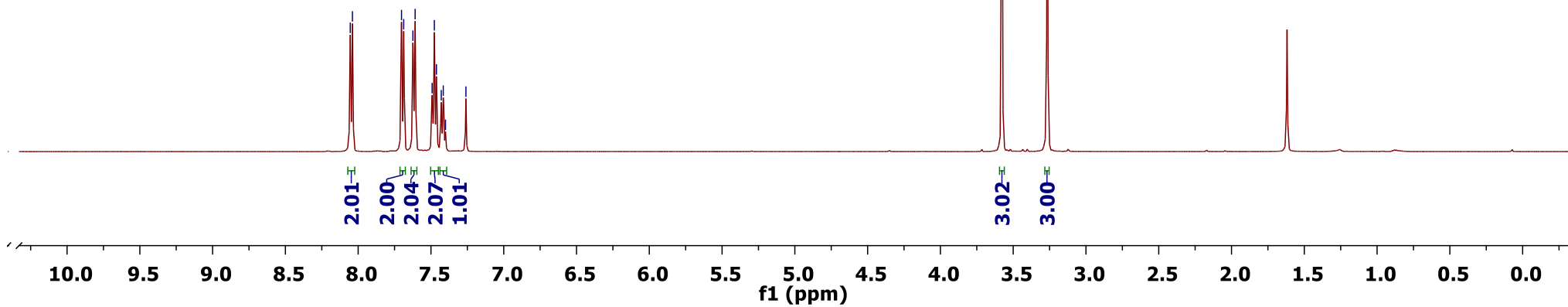
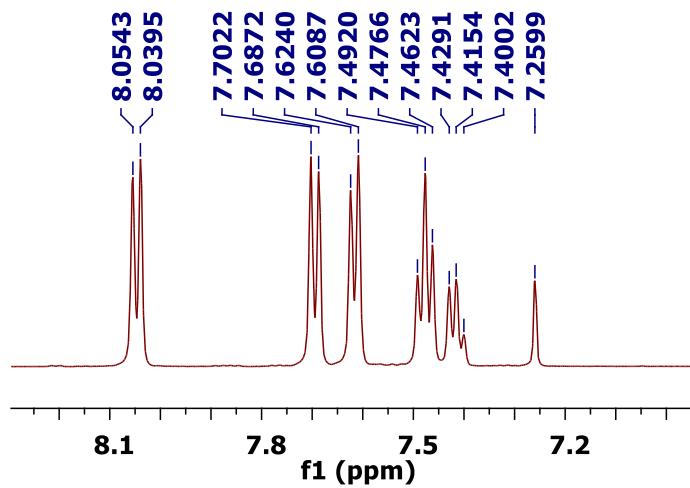


<sup>1</sup>H NMR for compound 3ob (500 MHz, CDCl<sub>3</sub>)



8.0543  
8.0395  
7.7022  
7.6872  
7.6240  
7.6087  
7.4920  
7.4766  
7.4623  
7.4291  
7.4154  
7.4002  
7.2599

3.5770  
3.2656



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ob (125 MHz,  $\text{CDCl}_3$ )

— 196.9293

— 188.1891

— 147.1141

— 139.7672

— 132.0138

— 130.6084

— 129.1415

— 128.6272

— 127.6492

— 127.4538

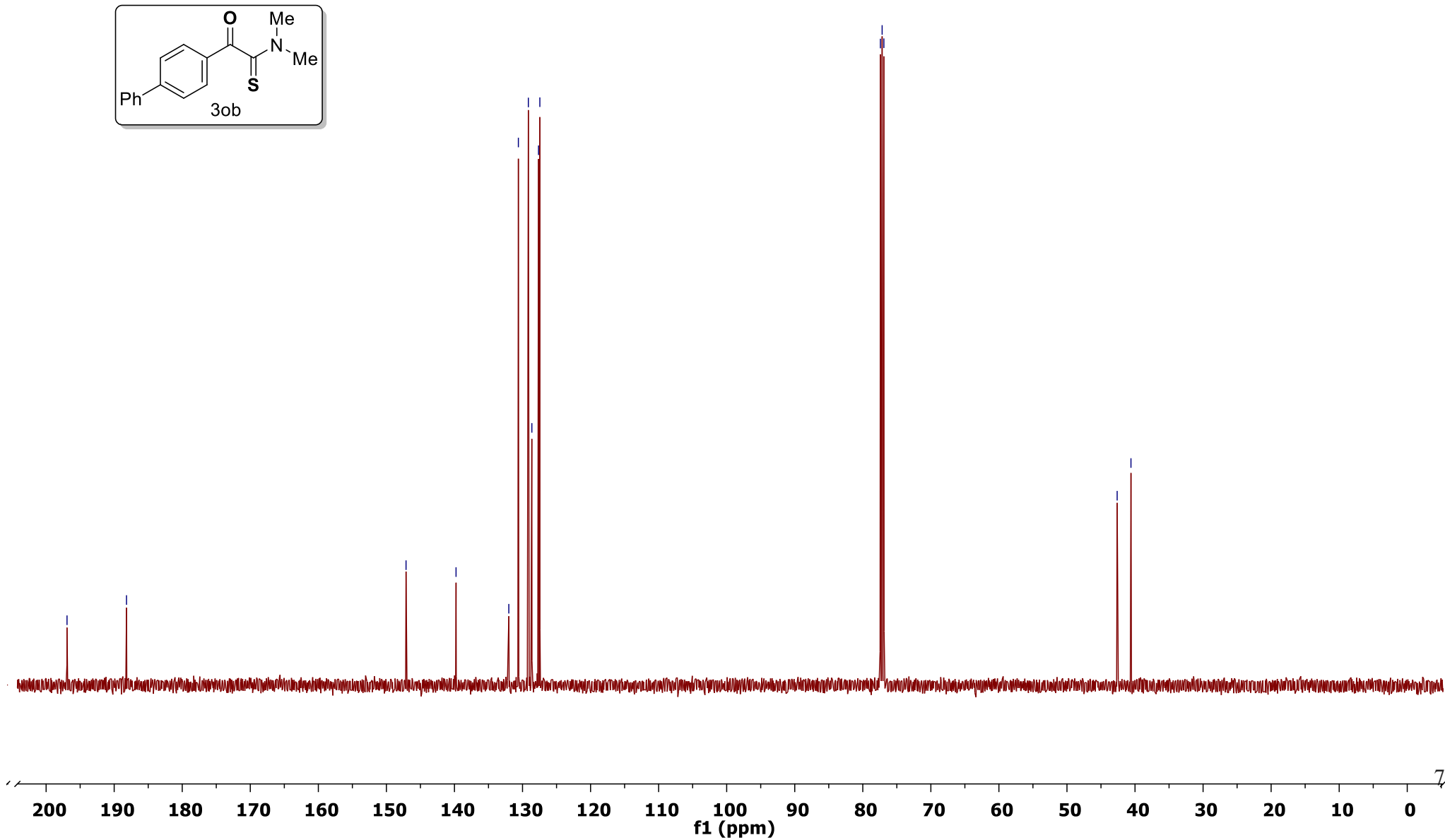
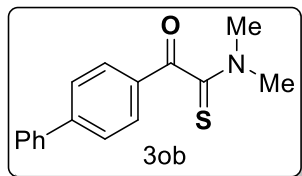
77.4118

77.1578

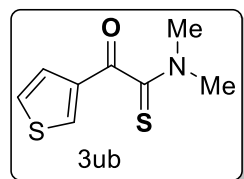
76.9037

42.6244

40.5954

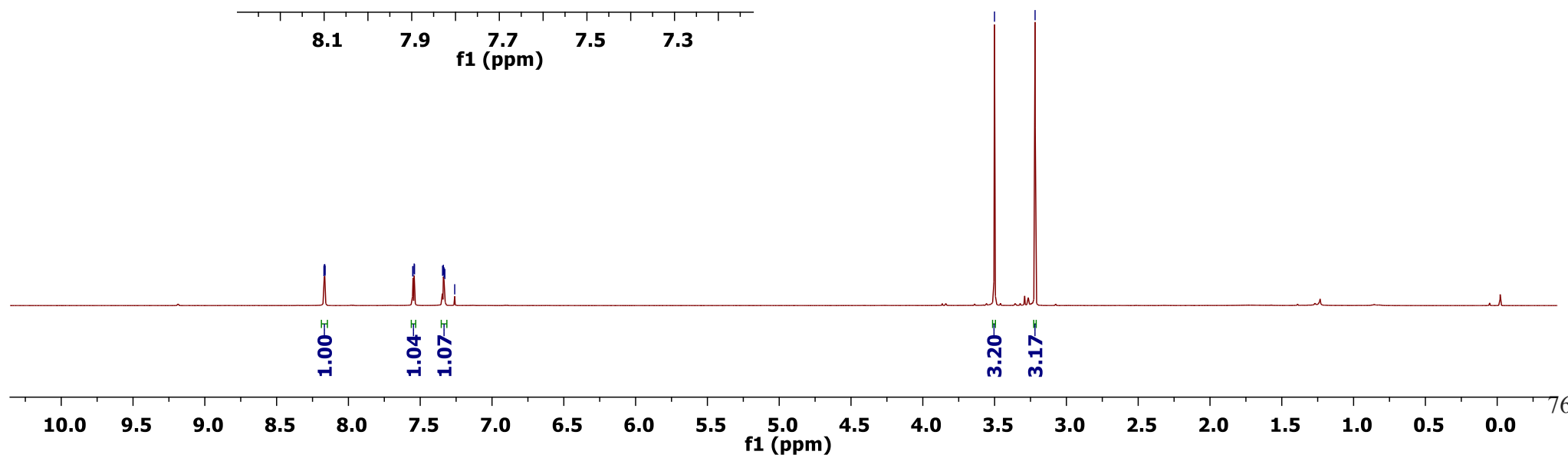
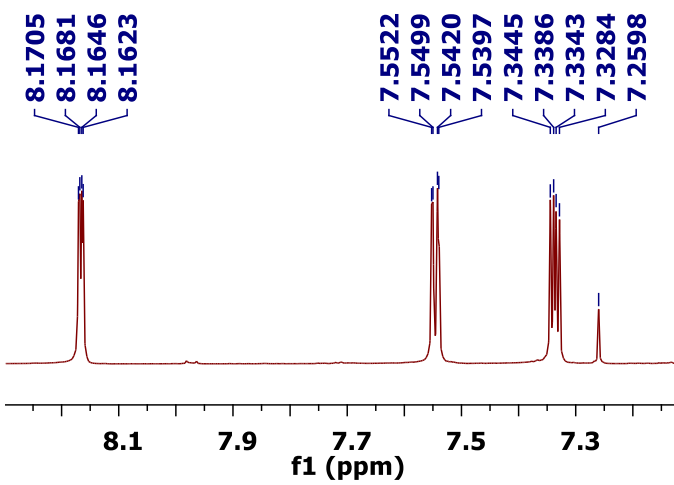


<sup>1</sup>H NMR for compound 3ub (500 MHz, CDCl<sub>3</sub>)



8.1705  
8.1681  
8.1646  
8.1623  
7.5522  
7.5499  
7.5420  
7.5397  
7.3445  
7.3386  
7.3343  
7.3284  
7.2598

3.5008  
3.2180





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3ub (125 MHz,  $\text{CDCl}_3$ )

— 196.5313

— 182.8208

— 138.3124

— 135.7769

— 127.6265

— 127.0818

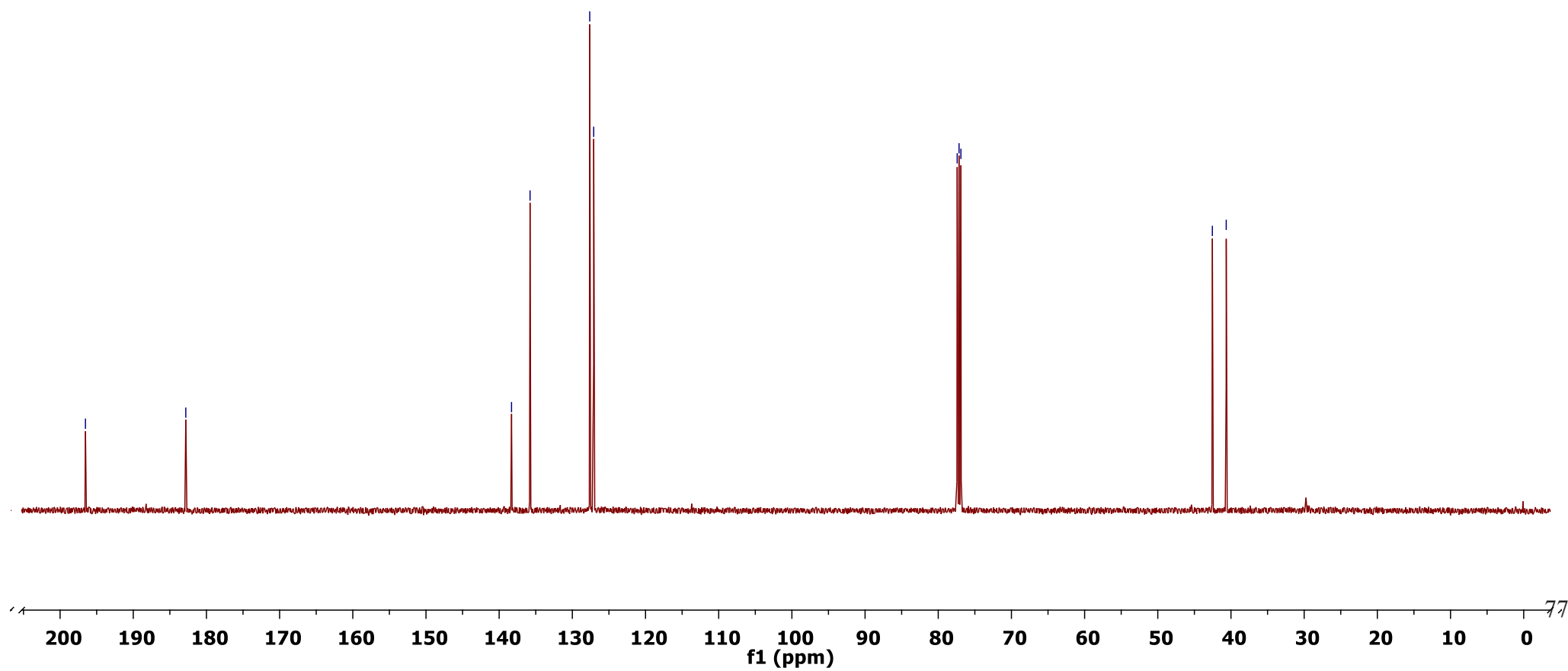
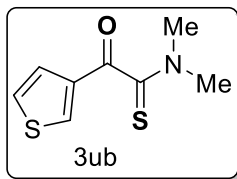
— 77.4140

— 77.1597

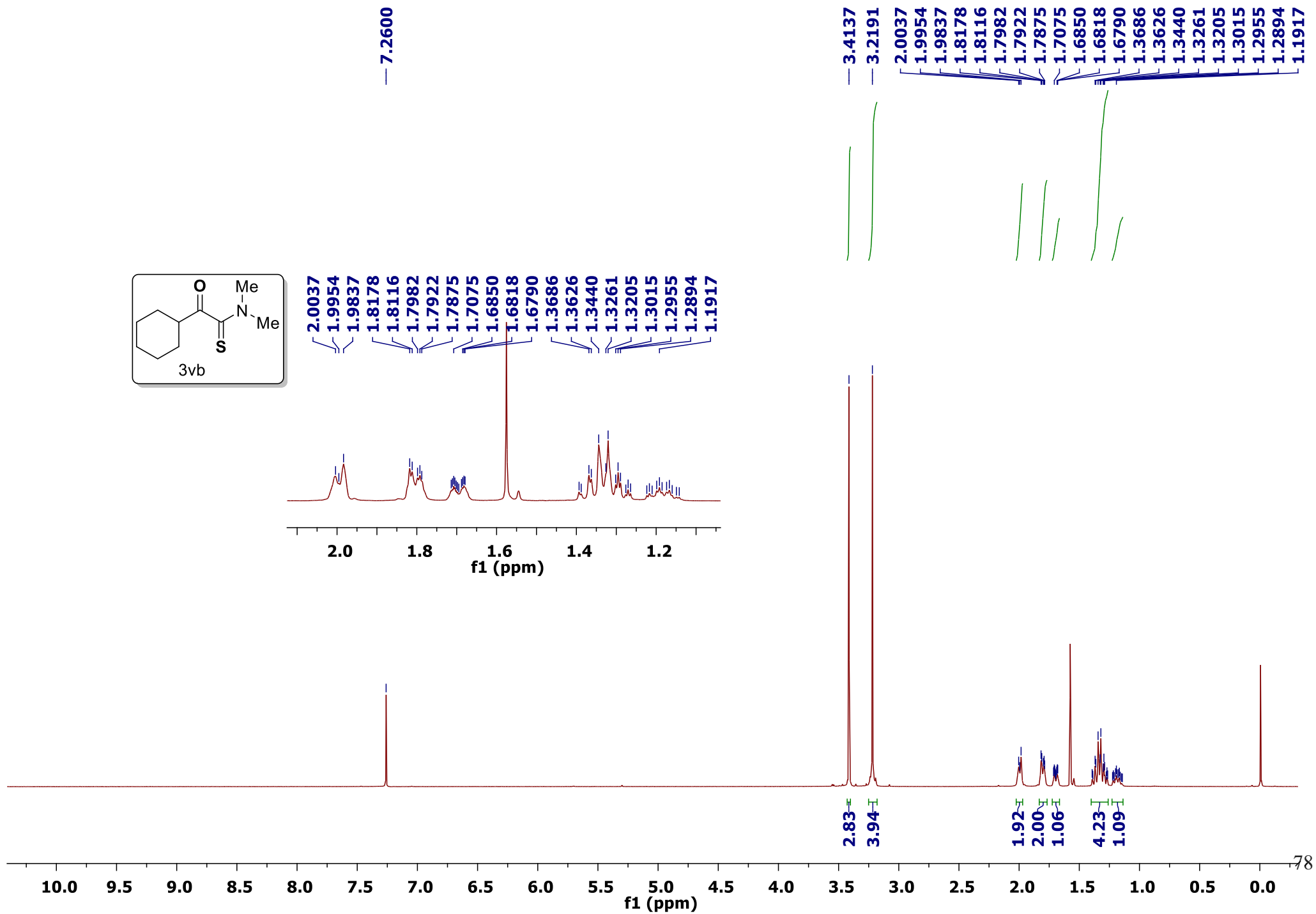
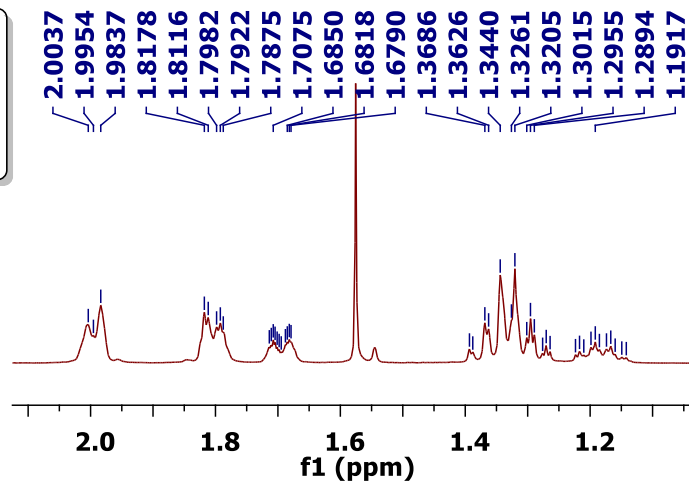
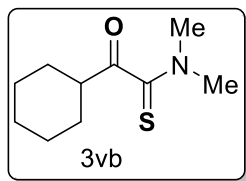
— 76.9055

— 42.5436

— 40.6487



<sup>1</sup>H NMR for compound 3vb (500 MHz, CDCl<sub>3</sub>)



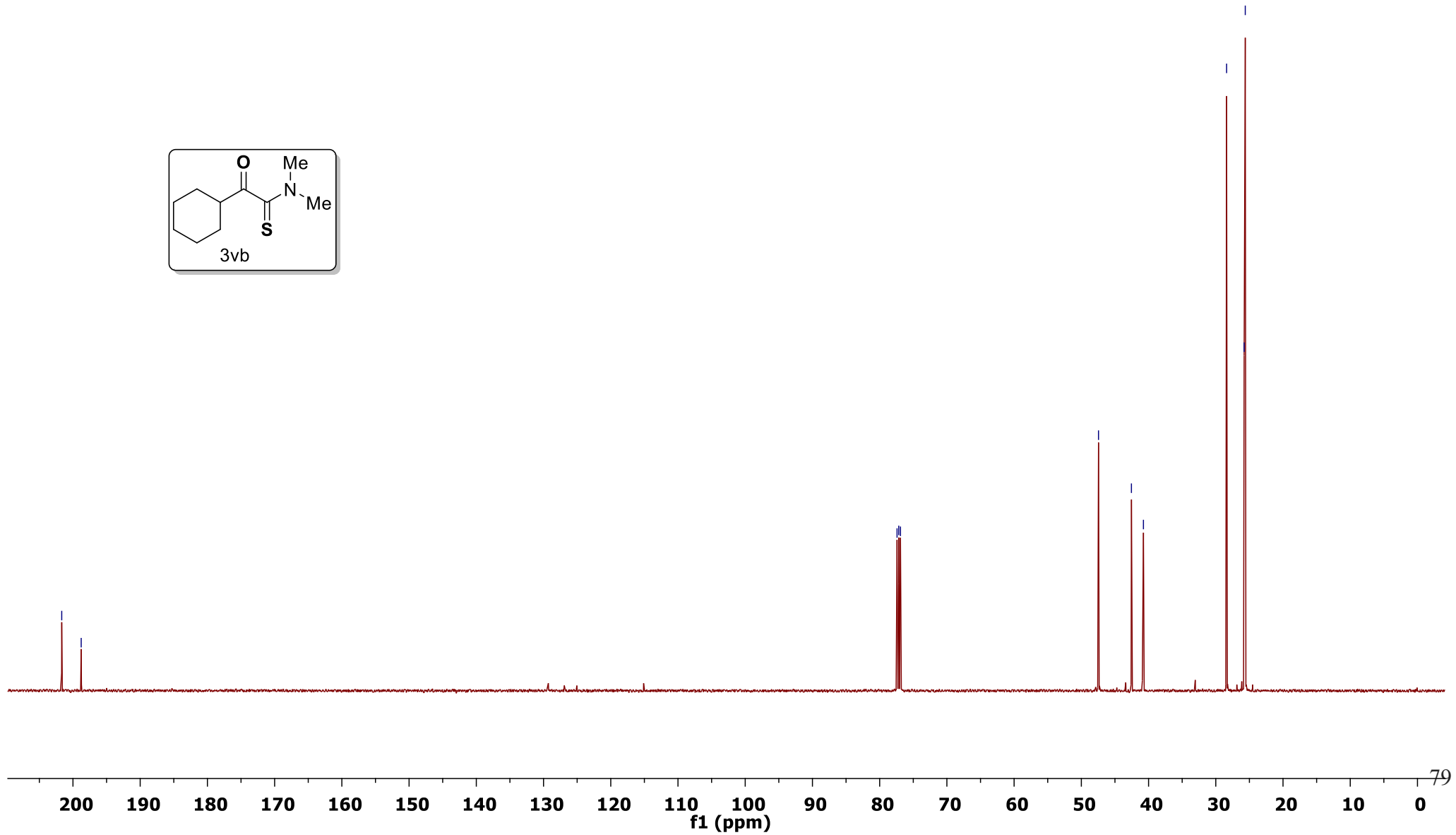
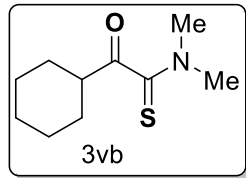
$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 3vb (125 MHz,  $\text{CDCl}_3$ )

— 201.6694  
— 198.7753

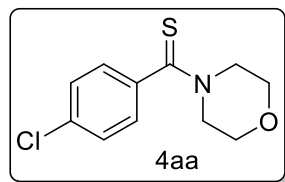
— 77.4133  
— 77.1591  
— 76.9046

— 47.4455  
— 42.5496  
— 40.7627

— 28.3907  
— 25.7842  
— 25.6137

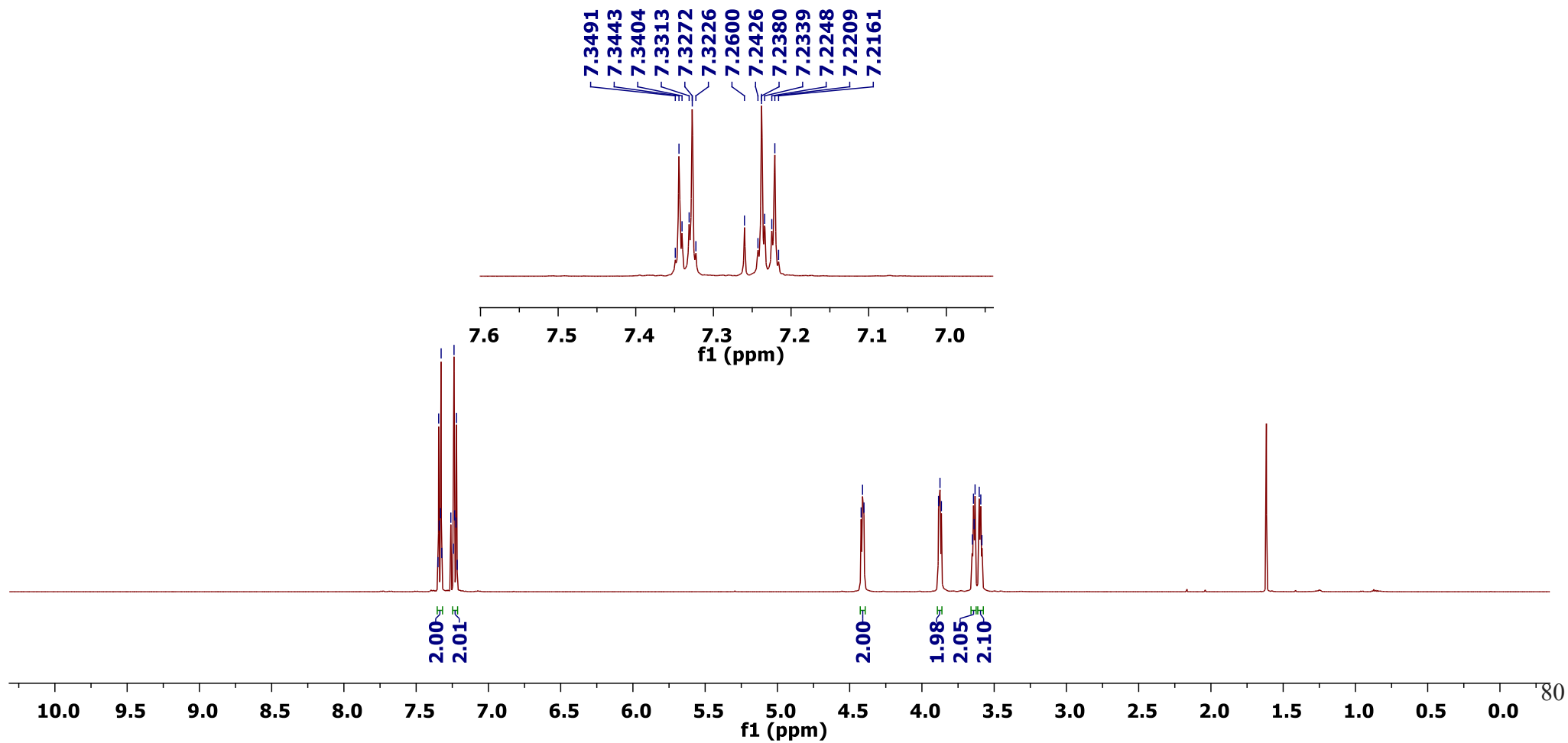


<sup>1</sup>H NMR for compound 4aa (500 MHz, CDCl<sub>3</sub>)

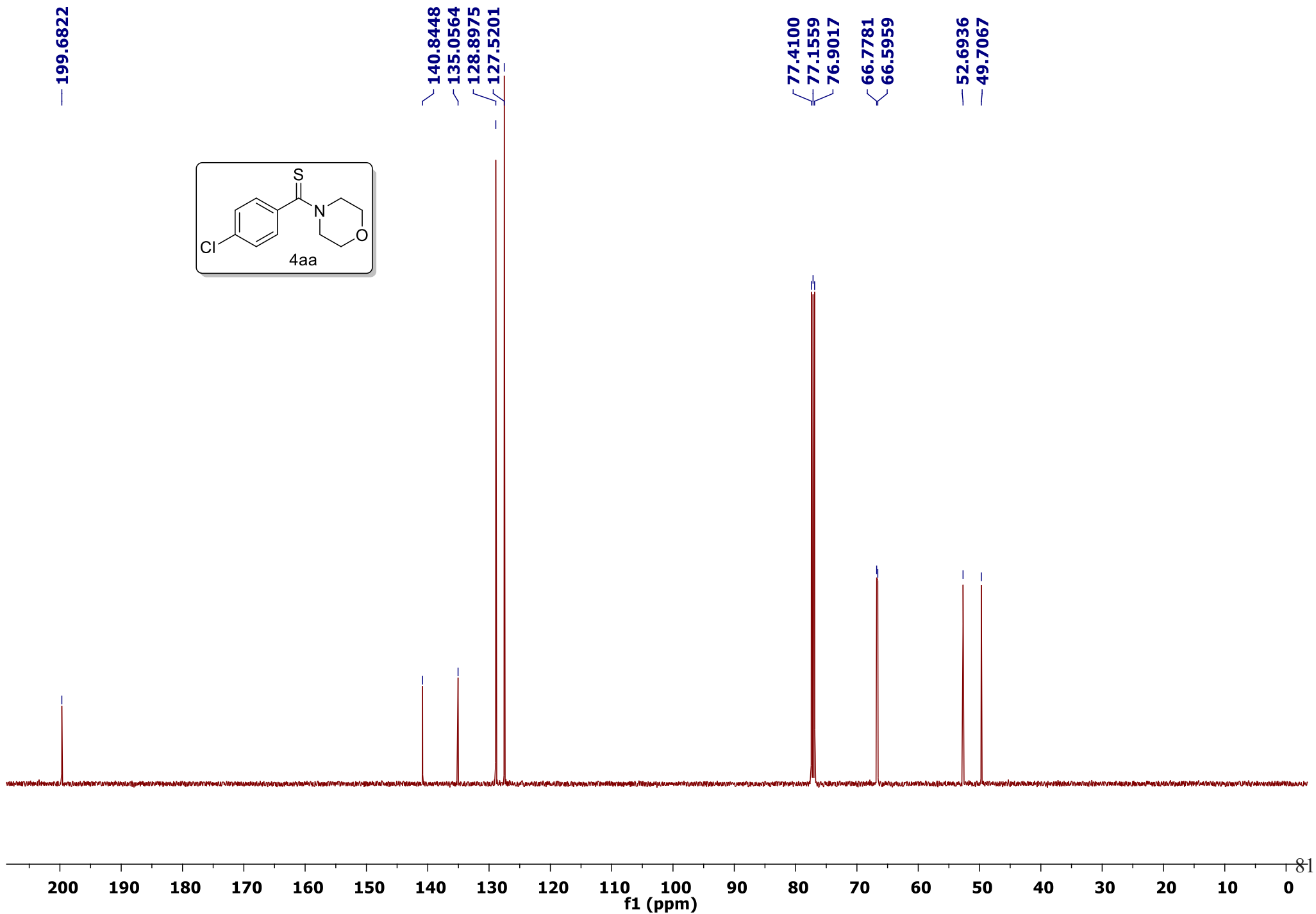
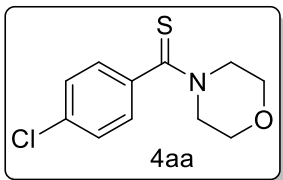


7.3491  
7.3443  
7.3404  
7.3313  
7.3272  
7.3226  
7.2600  
7.2426  
7.2380  
7.2339  
7.2248  
7.2209  
7.2161

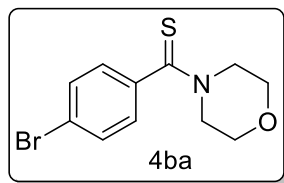
4.4206  
4.4109  
4.4010  
3.8849  
3.8750  
3.8652  
3.6506  
3.6431  
3.6390  
3.6323  
3.6034  
3.5925  
3.5848



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4aa (125 MHz,  $\text{CDCl}_3$ )



<sup>1</sup>H NMR for compound 4ba (500 MHz, CDCl<sub>3</sub>)



7.4970  
7.4924  
7.4885  
7.4792  
7.4753  
7.4707  
7.2599  
7.1681  
7.1637  
7.1598  
7.1505  
7.1467  
7.1419  
4.4039  
4.3944  
4.3844  
3.8709  
3.8612  
3.8514  
3.6364  
3.6292  
3.6249  
3.6186  
3.5892  
3.5782  
3.5706

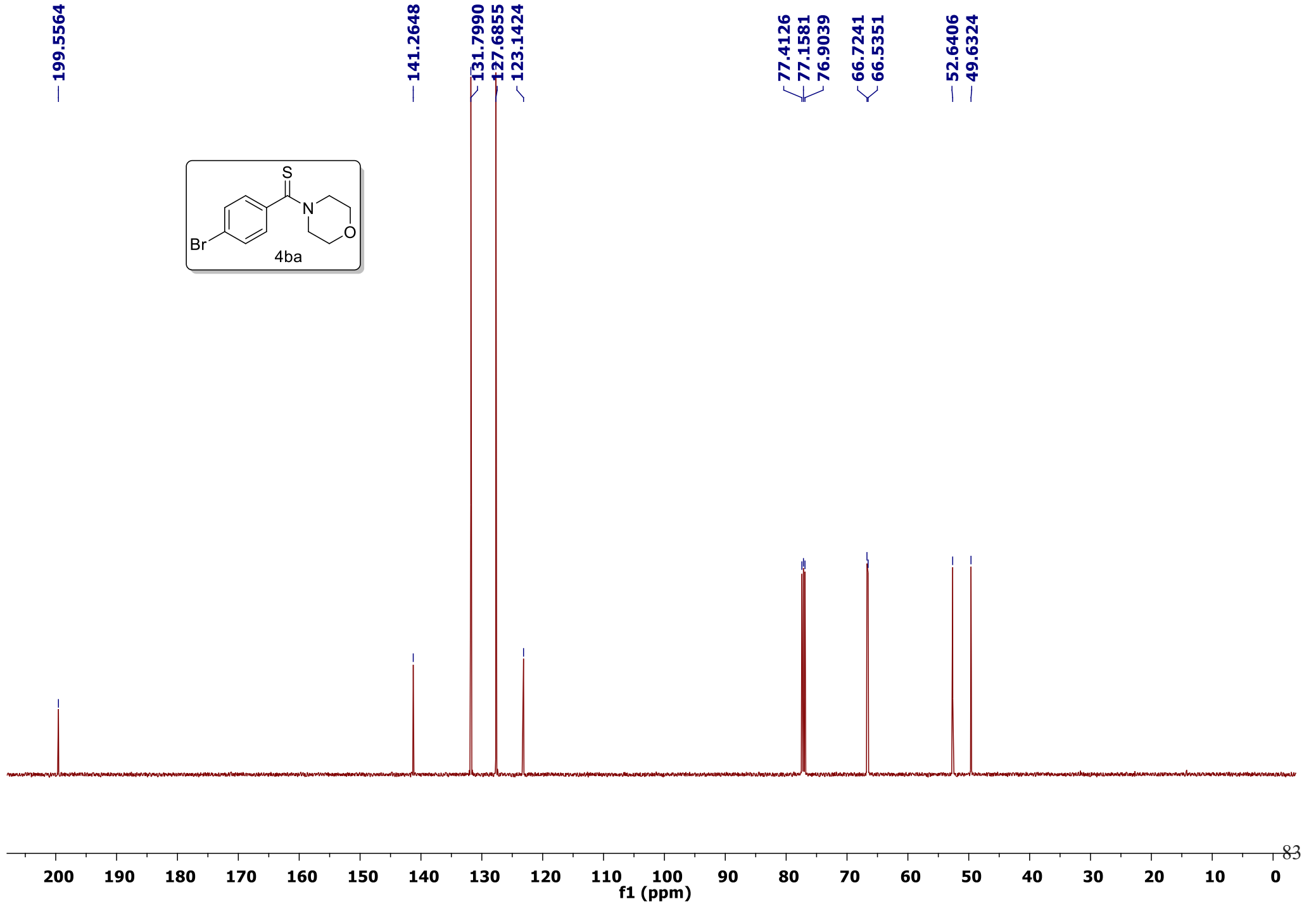
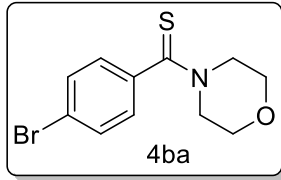
7.4970  
7.4924  
7.4885  
7.4792  
7.4753  
7.4707  
7.2599  
7.1681  
7.1637  
7.1598  
7.1505  
7.1467  
7.1419

7.6  
7.5  
7.4  
7.3  
7.2  
7.1  
7.0  
f1 (ppm)

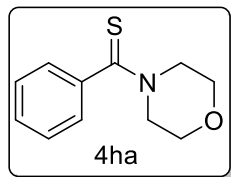
2.00  
2.02  
2.07  
2.03  
2.05  
2.04

10.0  
9.5  
9.0  
8.5  
8.0  
7.5  
7.0  
6.5  
6.0  
5.5  
5.0  
4.5  
4.0  
3.5  
3.0  
2.5  
2.0  
1.5  
1.0  
0.5  
0.0  
f1 (ppm)

$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4ba (125 MHz,  $\text{CDCl}_3$ )

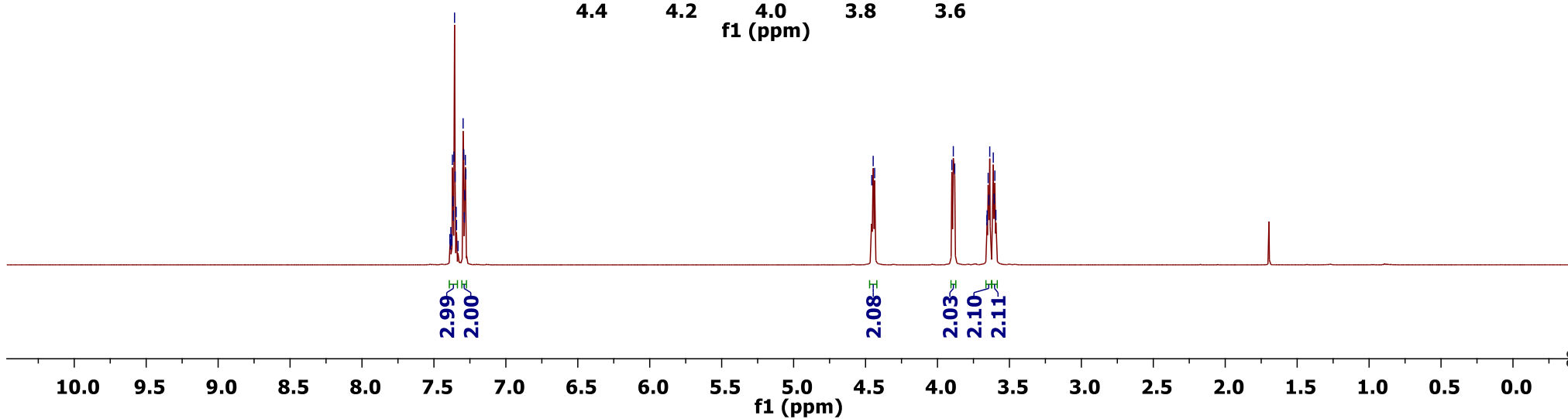
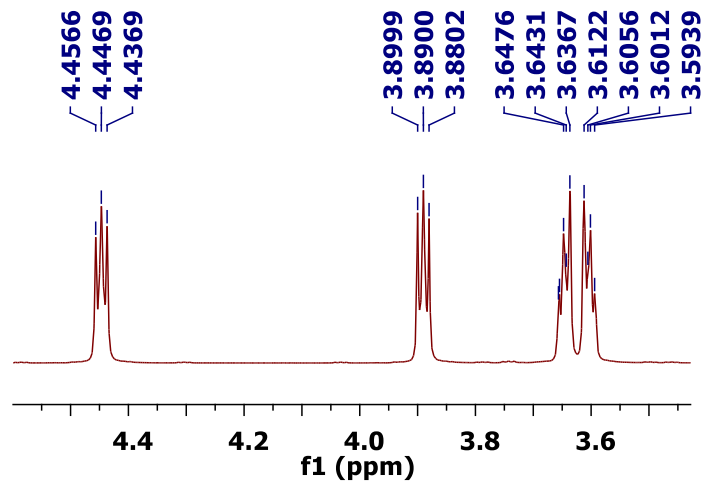


<sup>1</sup>H NMR for compound 4ha (500 MHz, CDCl<sub>3</sub>)



7.3889  
7.3852  
7.3825  
7.3794  
7.3766  
7.3755  
7.3716  
7.3679  
7.3642  
7.3608  
7.3565  
7.3526  
7.3453  
7.3430  
7.3329  
7.2971  
7.2928  
7.2887  
7.2858  
7.2814  
7.2781

4.4566  
4.4469  
4.4369  
3.8999  
3.8900  
3.8802  
3.6476  
3.6431  
3.6367  
3.6122  
3.6056  
3.6012  
3.6476  
3.6431  
3.6367  
3.6122  
3.6056  
3.6012  
3.5939





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4ha (125 MHz,  $\text{CDCl}_3$ )

— 201.0133

— 142.5046

{ 128.8998

{ 128.5734

{ 125.9090

{ 77.4122

{ 77.1578

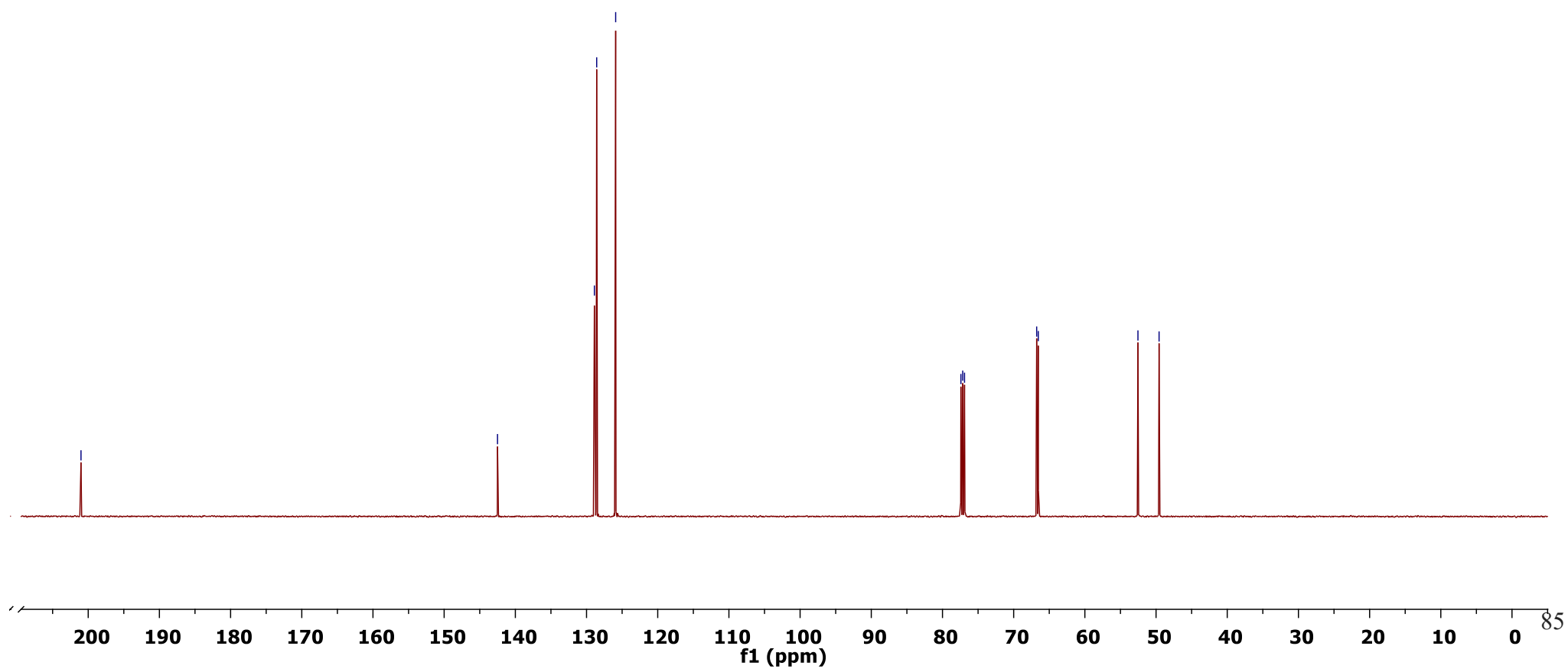
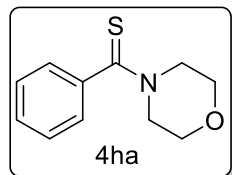
{ 76.9035

{ 66.7677

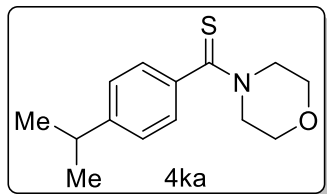
{ 66.5423

— 52.5433

— 49.5743



<sup>1</sup>H NMR for compound 4ka (500 MHz, CDCl<sub>3</sub>)



7.2597  
7.2272  
7.2252  
7.2214  
7.2098  
7.2071  
7.2011  
7.1951  
7.1914  
7.1895

4.4431  
4.4335  
4.4235  
3.8891  
3.8792  
3.8695  
3.6399  
2.9253  
2.9115  
2.8976  
2.8838  
2.8700

1.2415  
1.2277

4.4431  
4.4335  
4.4235  
3.8891  
3.8792  
3.8695  
3.6399  
2.9253  
2.9115  
2.8976  
2.8838  
2.8700

4.5  
4.0  
3.5  
3.0  
f1 (ppm)

3.93

2.00

2.02

3.92

1.00

6.16

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0  
f1 (ppm)

$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4ka (125 MHz,  $\text{CDCl}_3$ )

— 201.5675

— 150.0672

— 140.0760

{ 126.6844  
126.2093

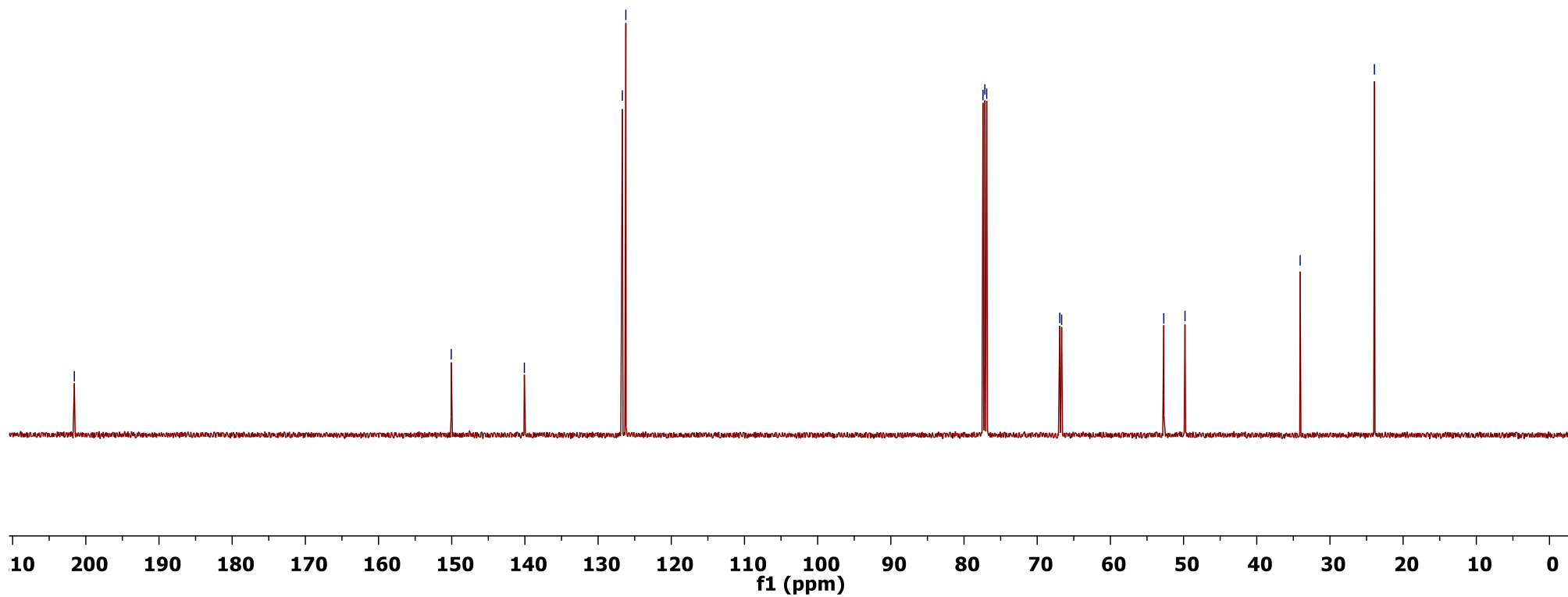
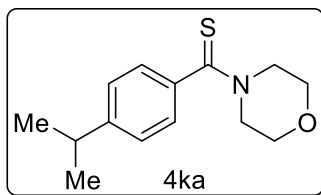
{ 77.4100  
77.1558  
76.9017

{ 66.9153  
66.6593

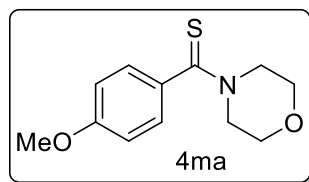
— 52.7065  
— 49.8051

— 34.0712

— 23.9261

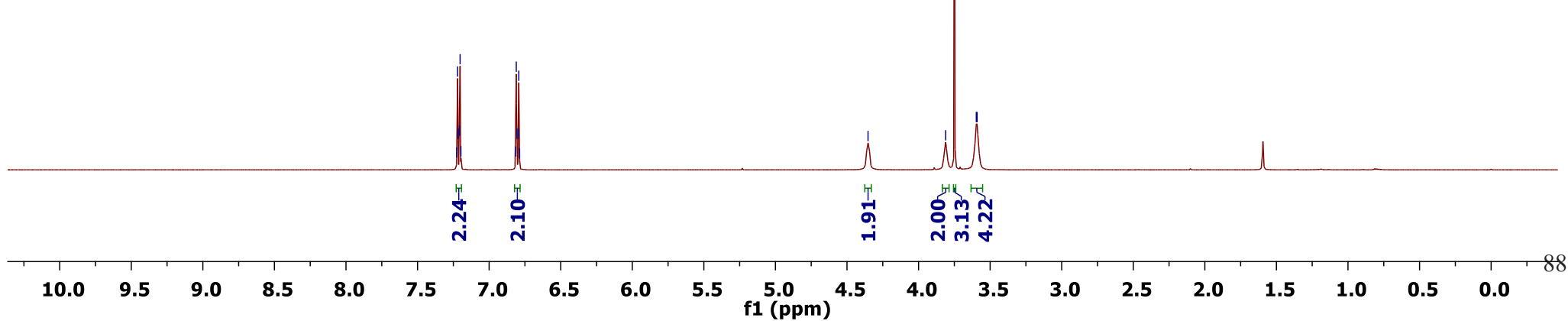
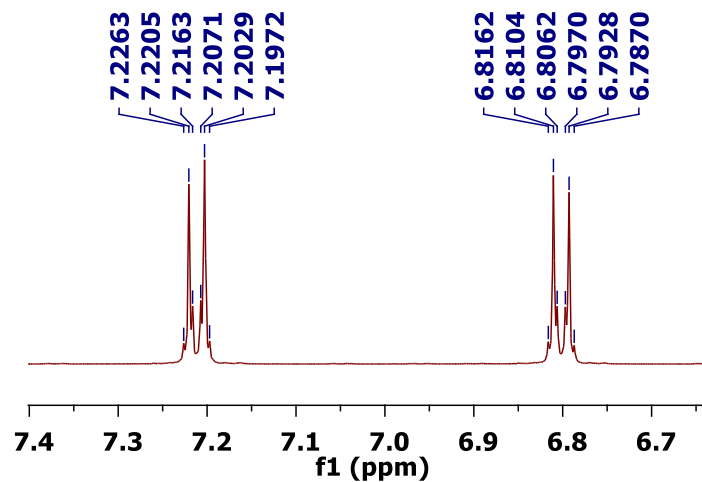


<sup>1</sup>H NMR for compound 4ma (500 MHz, CDCl<sub>3</sub>)

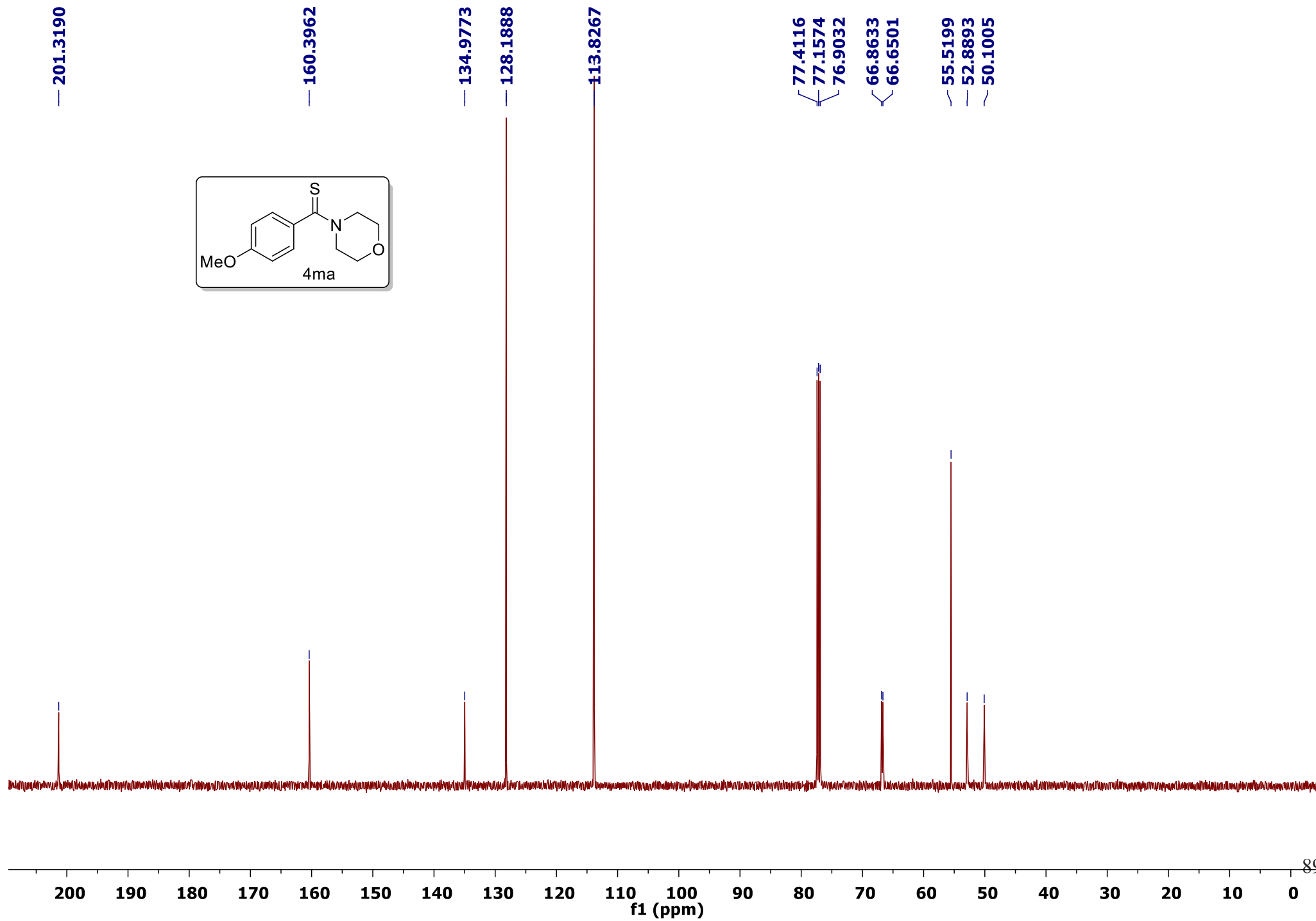
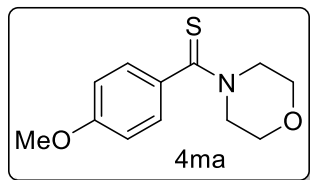


7.2263  
7.2205  
7.2163  
7.2071  
7.2029  
7.1972  
6.8162  
6.8104  
6.8062  
6.7970  
6.7928  
6.7870

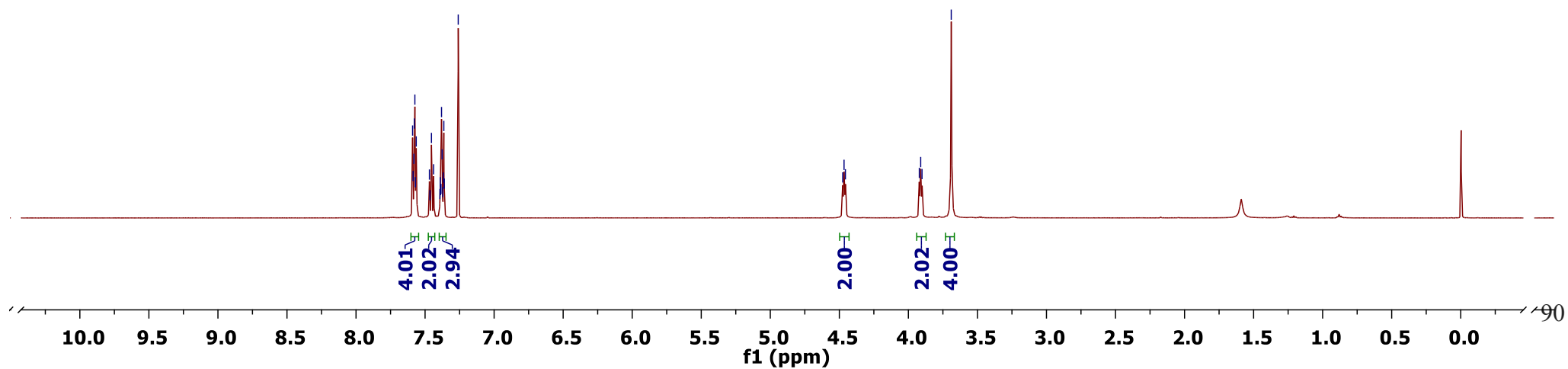
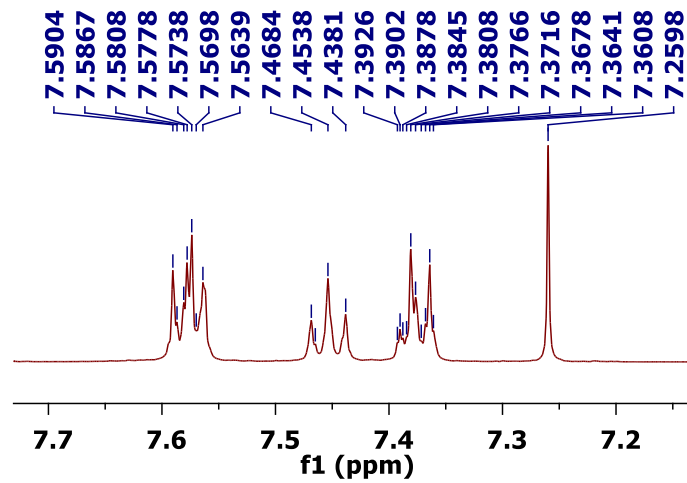
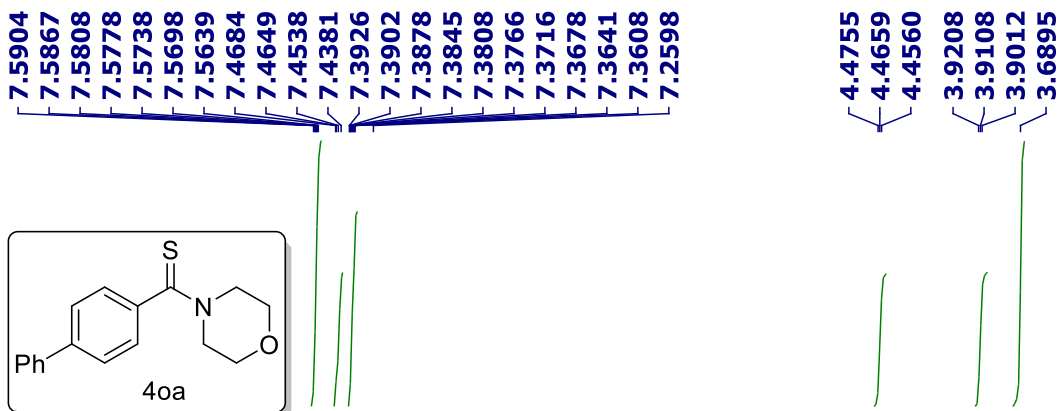
4.3530  
3.8108  
3.7489  
3.5967  
3.5910



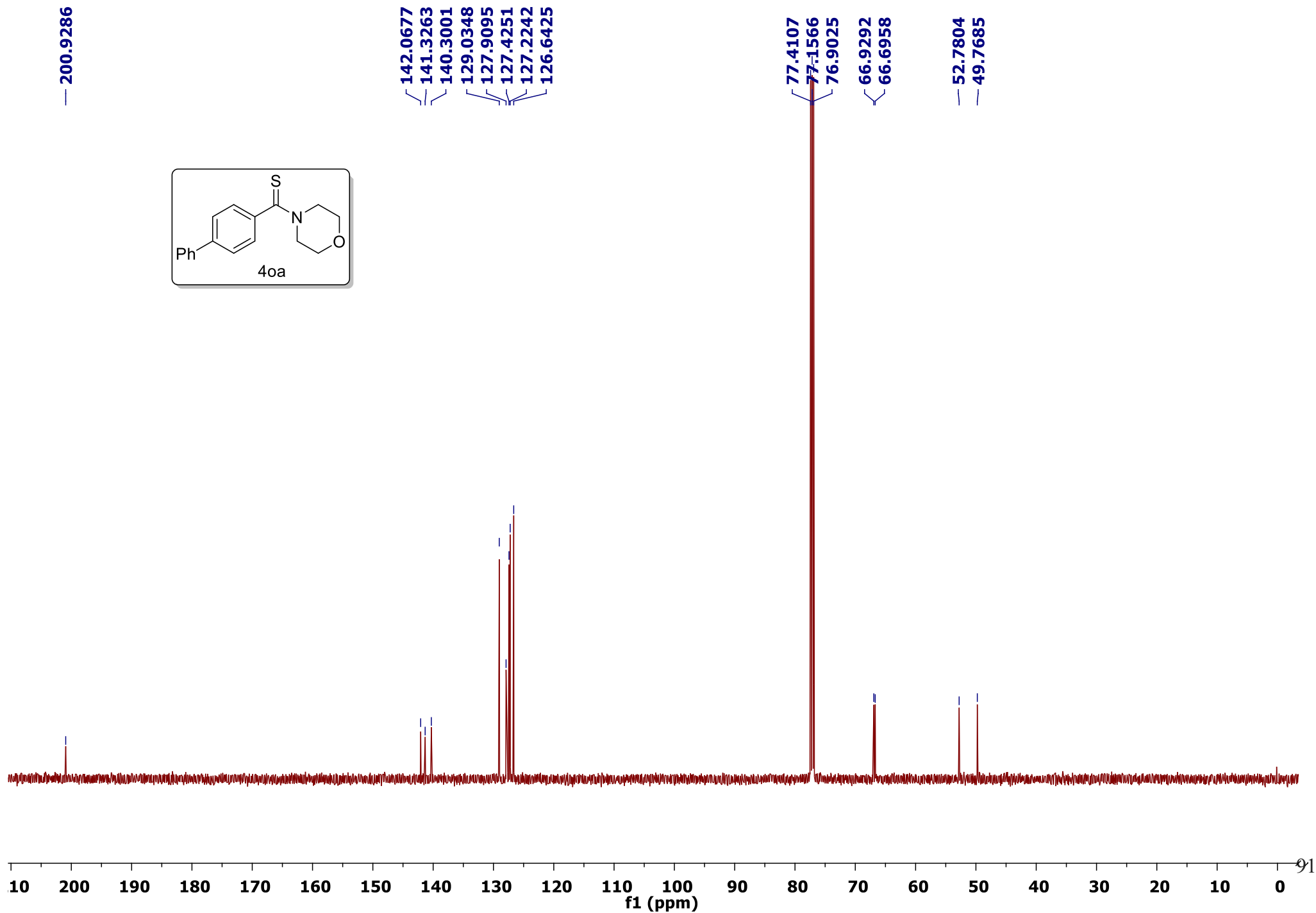
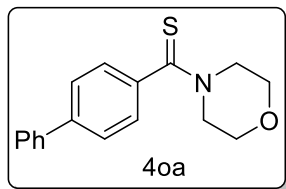
$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4ma (125 MHz,  $\text{CDCl}_3$ )



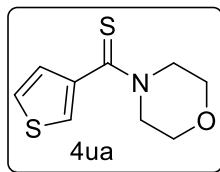
<sup>1</sup>H NMR for compound 40a (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4oa (125 MHz,  $\text{CDCl}_3$ )

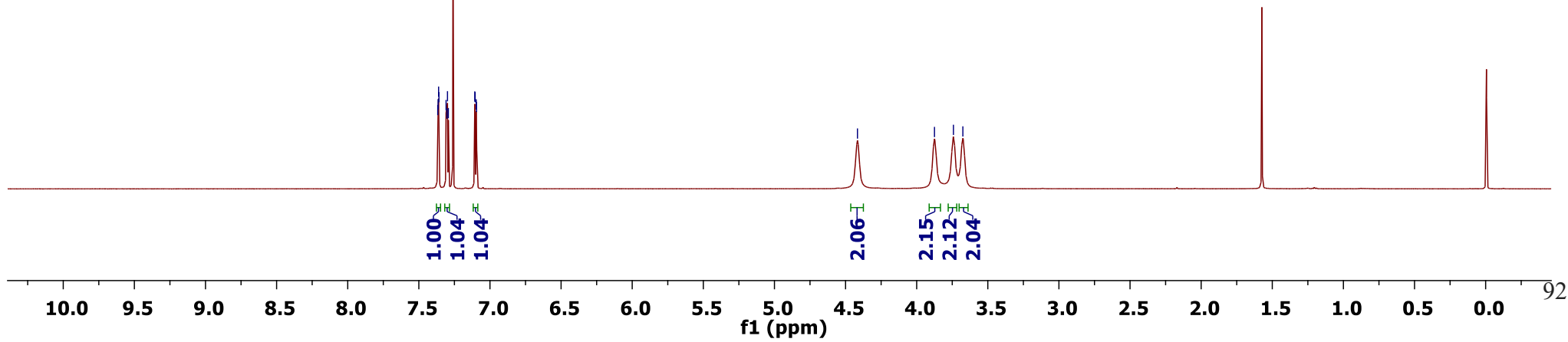
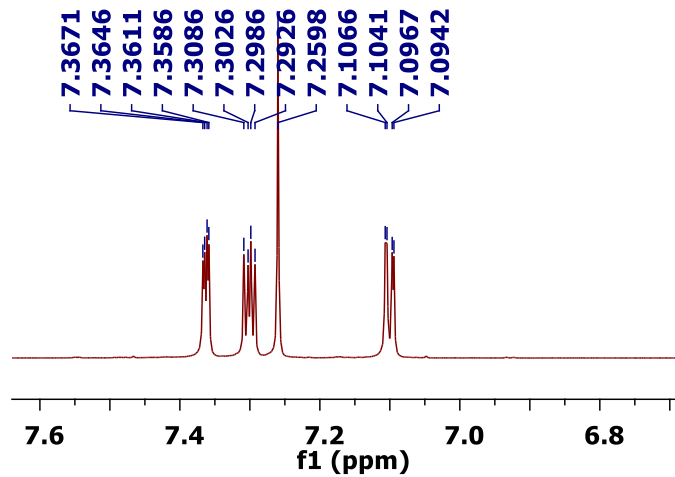


<sup>1</sup>H NMR for compound 4ua (500 MHz, CDCl<sub>3</sub>)



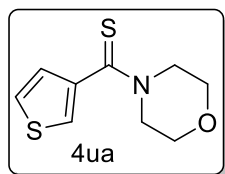
7.3671  
7.3646  
7.3611  
7.3586  
7.3086  
7.3026  
7.2986  
7.2926  
7.2598  
7.1066  
7.1041  
7.0967  
7.0942

4.4163  
3.8749  
3.7417  
3.6753





$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4ua (125 MHz,  $\text{CDCl}_3$ )



— 195.3307

— 142.6478

{ 127.0405

{ 126.1299

{ 124.3682

{ 77.4147

{ 77.1607

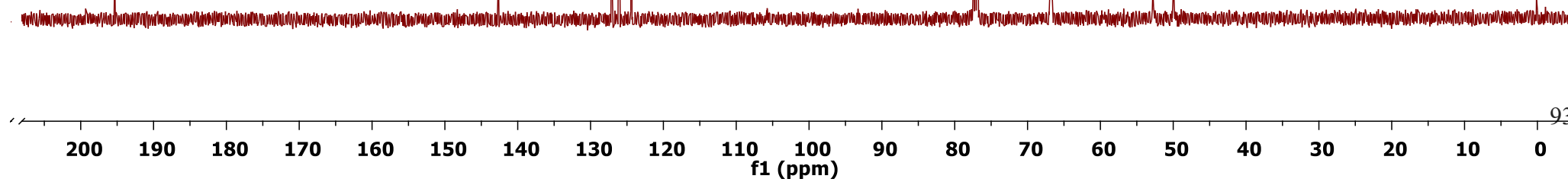
{ 76.9067

{ 66.8772

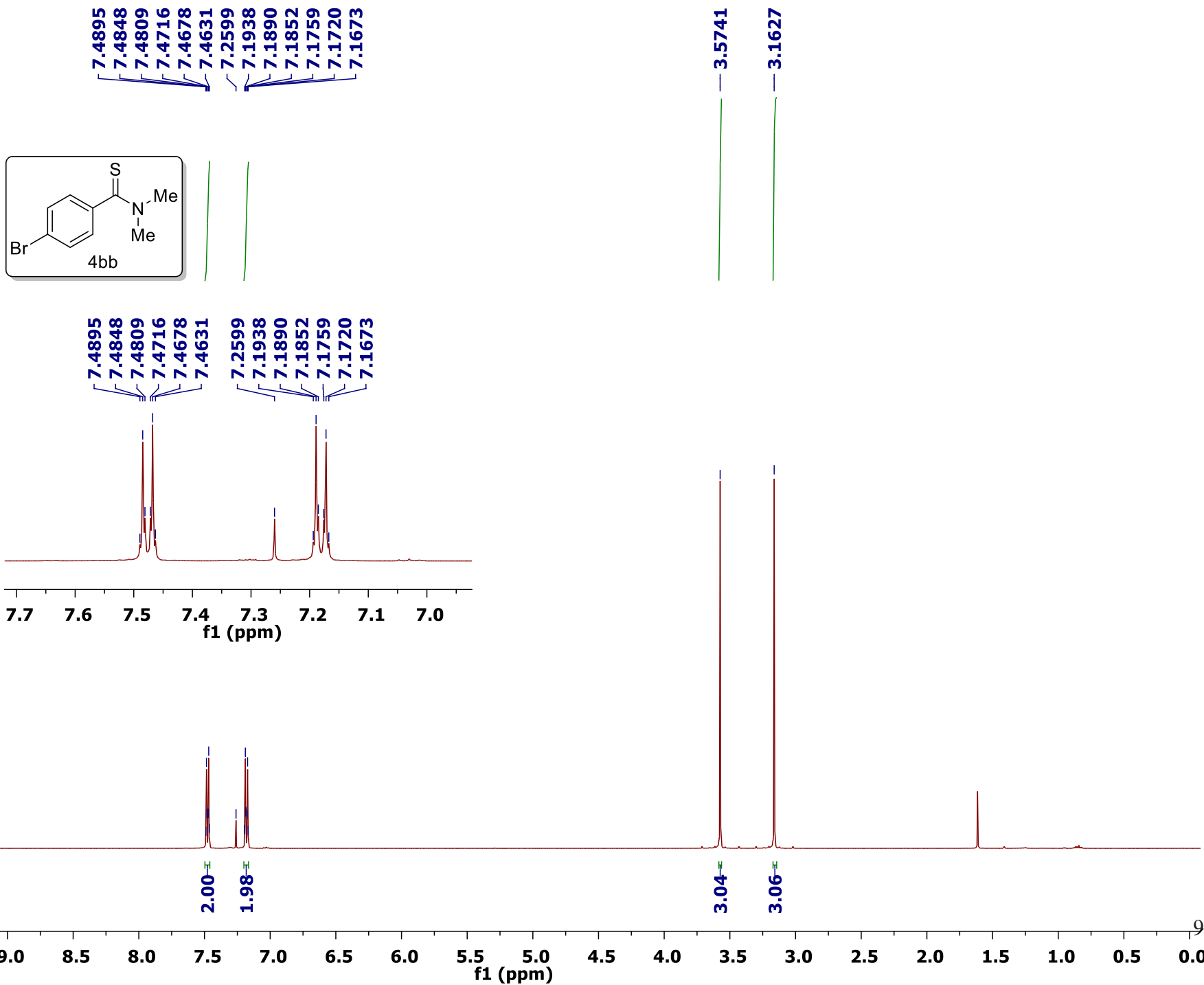
{ 66.6851

{ 52.7974

{ 49.9485



<sup>1</sup>H NMR for compound 4bb (500 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4bb (125 MHz,  $\text{CDCl}_3$ )

— 199.9223

— 142.1904

— 131.6175

— 127.5921

— 122.8359

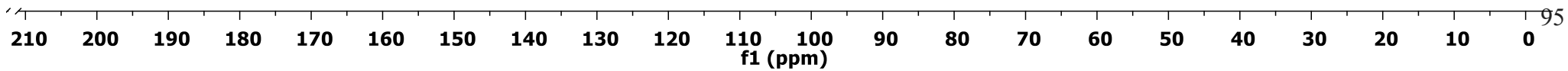
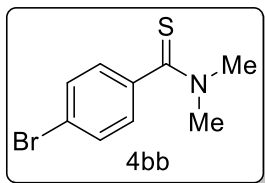
77.4114

77.1574

76.9032

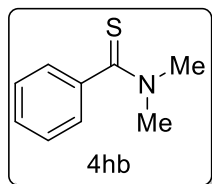
44.2661

43.3793



<sup>1</sup>H NMR for compound 4hb (500 MHz, CDCl<sub>3</sub>)

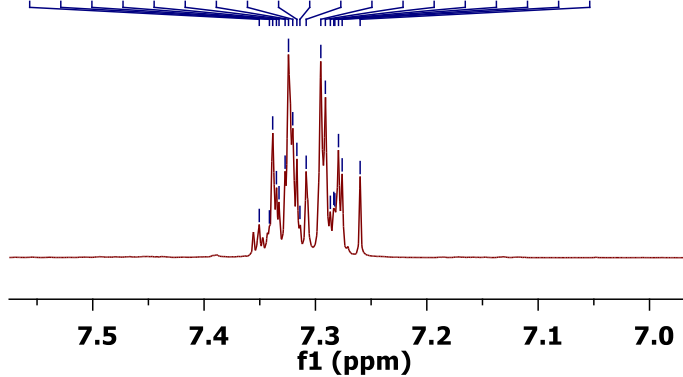
7.3504  
7.3413  
7.3384  
7.3350  
7.3327  
7.3272  
7.3242  
7.3204  
7.3167  
7.3139  
7.3083  
7.2952  
7.2911  
7.2866  
7.2835  
7.2826  
7.2793  
7.2760  
7.2599



3.5903

3.1529

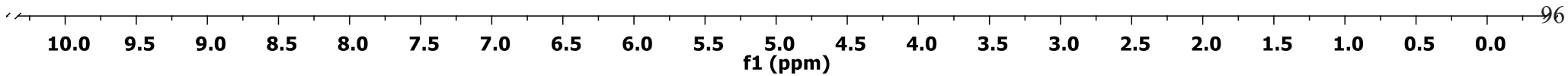
7.3504  
7.3413  
7.3384  
7.3350  
7.3327  
7.3272  
7.3242  
7.3204  
7.3167  
7.3139  
7.3083  
7.2952  
7.2911  
7.2866  
7.2835  
7.2826  
7.2793  
7.2760  
7.2599



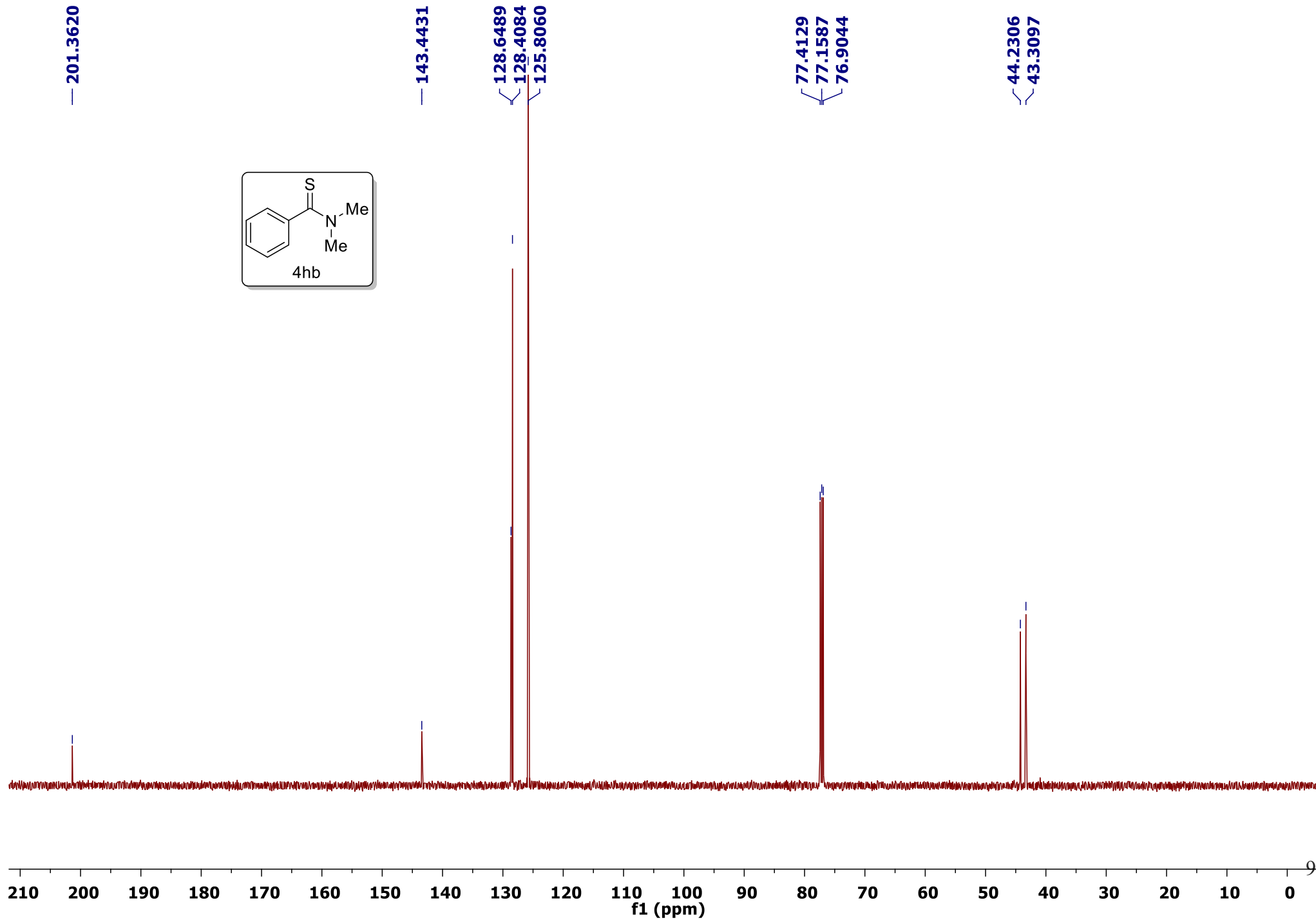
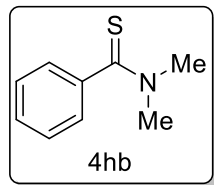
1.00  
1.99  
2.06

3.09

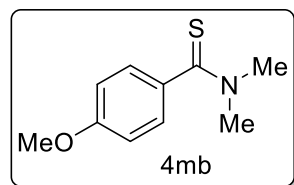
3.08



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4hb (125 MHz,  $\text{CDCl}_3$ )

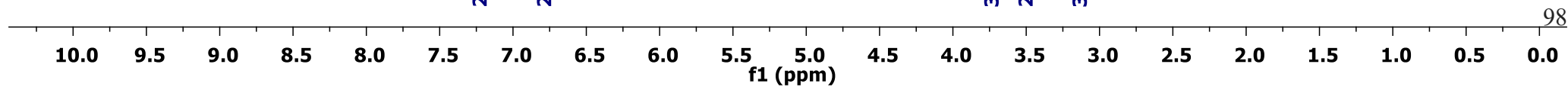
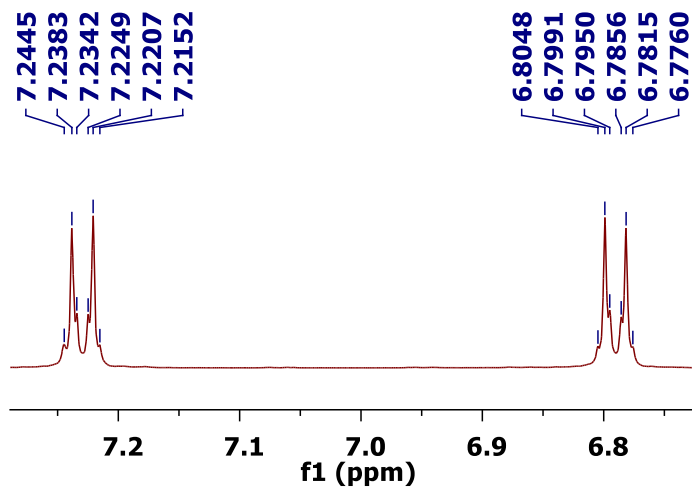


<sup>1</sup>H NMR for compound 4mb (500 MHz, CDCl<sub>3</sub>)

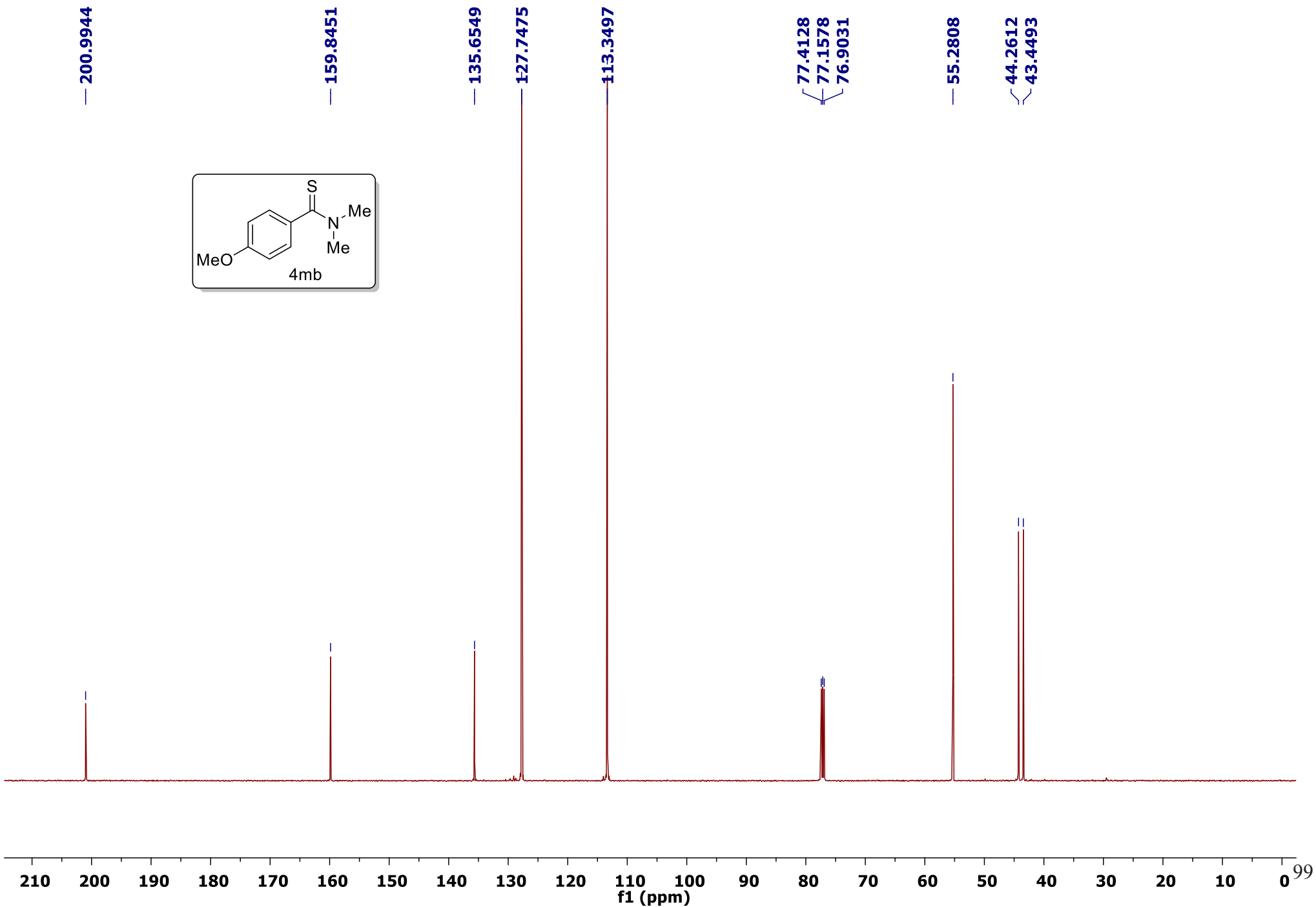
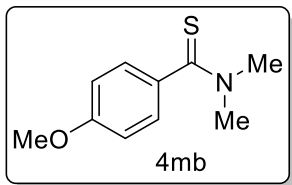


7.2445  
7.2383  
7.2342  
7.2249  
7.2207  
7.2152  
6.8048  
6.7991  
6.7950  
6.7856  
6.7815  
6.7760

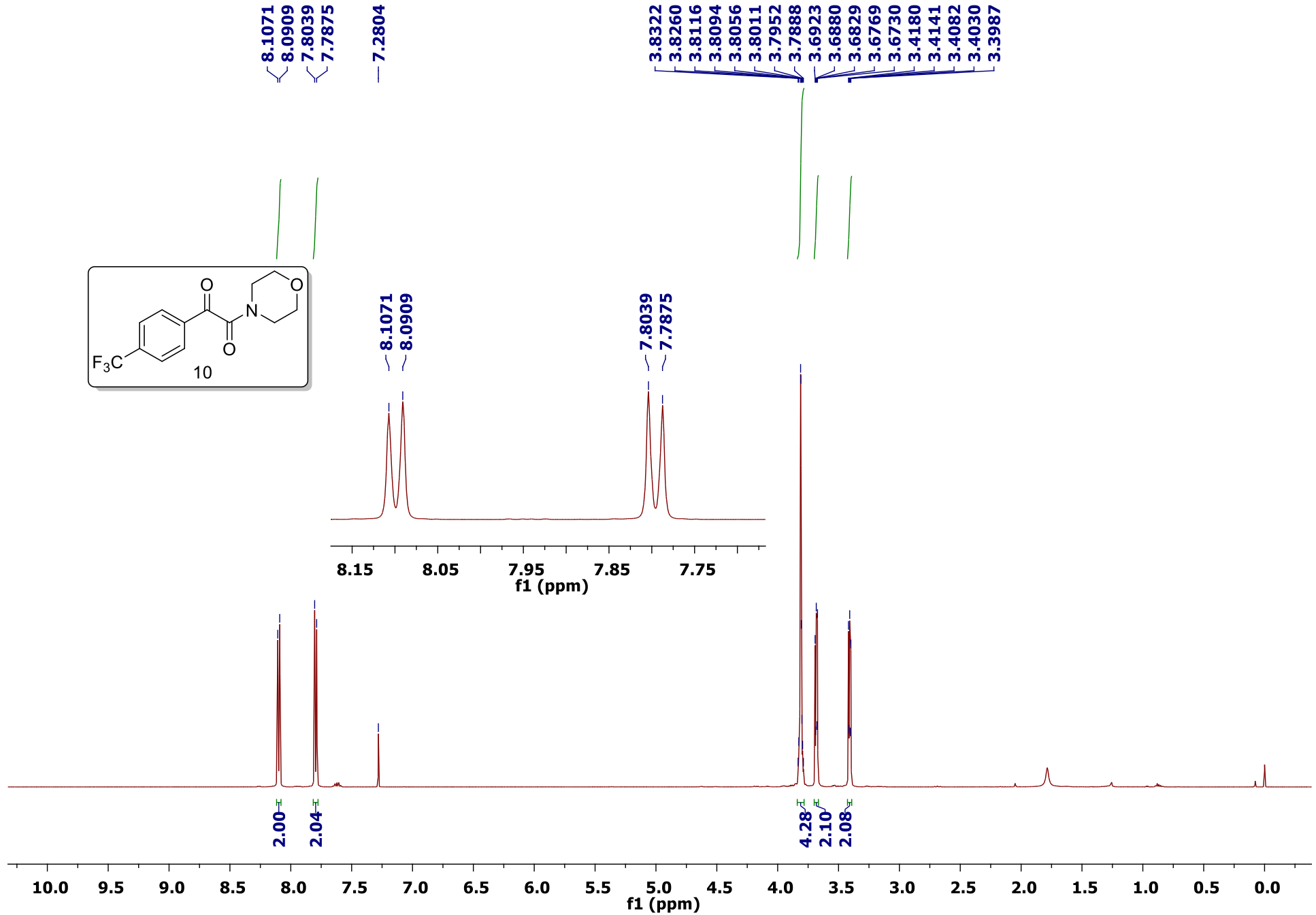
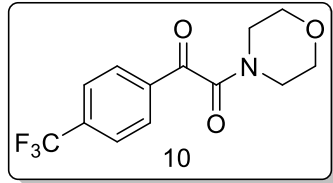
3.7412  
3.5093  
3.1376



$^{13}\text{C}\{^1\text{H}\}$  NMR for compound 4mb (125 MHz,  $\text{CDCl}_3$ )



<sup>1</sup>H NMR for compound 10 (500 MHz, CDCl<sub>3</sub>)





<sup>13</sup>C NMR for compound 10 (125 MHz, CDCl<sub>3</sub>)

— 189.6527

— 164.5405

136.3035

136.0415

135.7800

135.7415

135.5184

— 130.0655

126.5686

126.1297

126.1002

124.3977

122.2266

120.0555

77.2991

77.0449

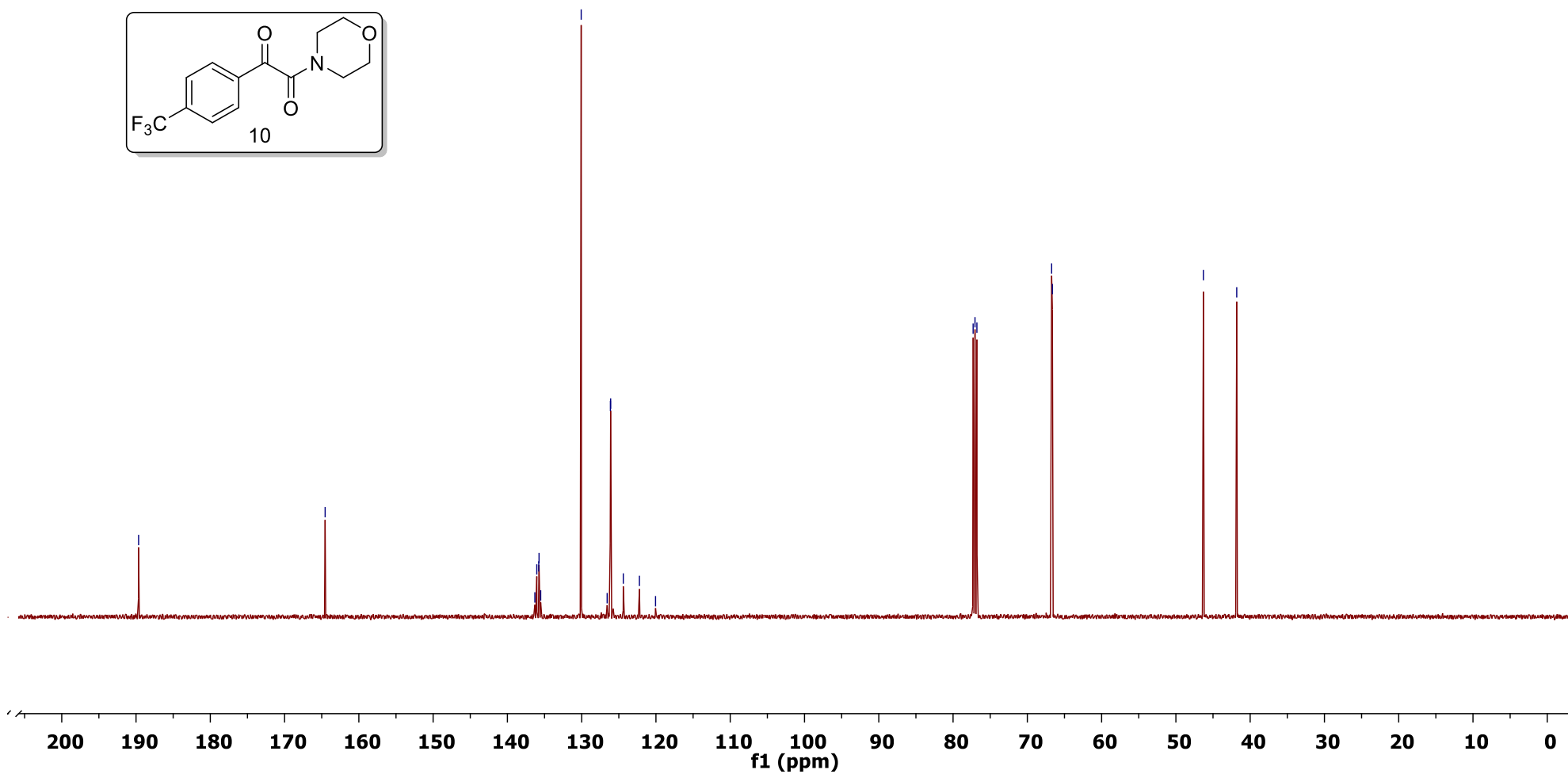
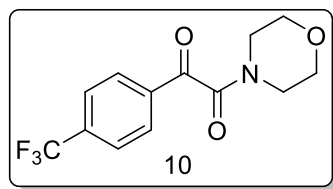
76.7906

66.7281

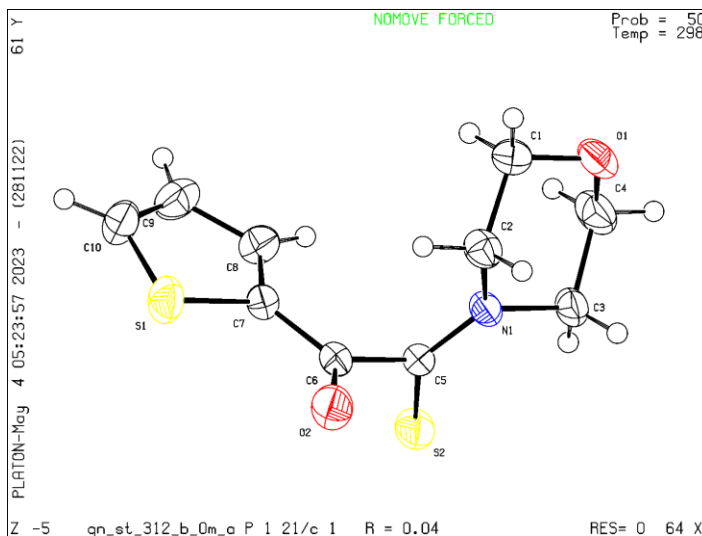
66.6287

— 46.2935

— 41.8077



### 3. Crystallographic data of 3ta (CCDC No. 2259851)



Bond precision: C-C = 0.0017 Å Wavelength=0.71073

Cell: a=10.8649(7) b=8.9005(5) c=11.3860(7)  
 alpha=90 beta=91.427(4) gamma=90

Temperature: 298 K

	Calculated	Reported
Volume Space	1100.72(12)	1100.72(12)
groupHall	P 21/c	P 1 21/c 1
group	-P 2ybc	-P 2ybc
Moiety formula	C10 H11 N O2 S2	C10 H11 N O2 S2
Sum formula	C10 H11 N O2 S2	C10 H11 N O2 S2
Mr	241.32	241.32
Dx, g cm <sup>-3</sup>	1.456	1.456
Z	4	4
Mu (mm <sup>-1</sup> )	0.462	0.462
F000	504.0	504.0
F000'	505.18	
h,k,lmax	18,14,19	18,14,19
Nref	5383	5360
Tmin,Tmax	0.797,0.827	0.655,0.747
Tmin'	0.797	

Correction method= # Reported T Limits: Tmin=0.655 Tmax=0.747 AbsCorr = MULTI-SCAN  
 Data completeness= 0.996 Theta(max)= 36.451  
 R(reflections)= 0.0419( 4038) wR2(reflection) = 0.1162( 5360)  
 S = 1.062 Npar= 136