Base-mediated Intramolecular Cyclization of α-Nitroethylallenic Esters as a Synthetic Route to 5-Hydroxy-3-pyrrolin-2-ones

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SUPPORTING INFORMATION

Contents:

X-Ray Data Collection and Structure Refinement Details for compound 2f:

A good quality single crystal of size 0.37 x 0.20 x 0.10 mm, was selected under a polarizing microscope and was mounted on a glass fiber for data collection. Single crystal X-ray data for compound **2f** were collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-Kα radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collection was performed using ω-scans of 0.5o steps at 293(2) K. Cell determination, data collection and data reduction was performed using the Rigaku CrystalClear-SM Expert 2.1 b24 software.[1] Structure solution and refinement were performed by using SHELXTL-NT.[2] Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.

Crystallization: The compound **2f** (5mg) was dissolved in a 1ml mixture of *n*-hexane/DCM (2:1) and placed in a cabinet to evaporate slowly. After two days, **2f** was obtained as white crystal.

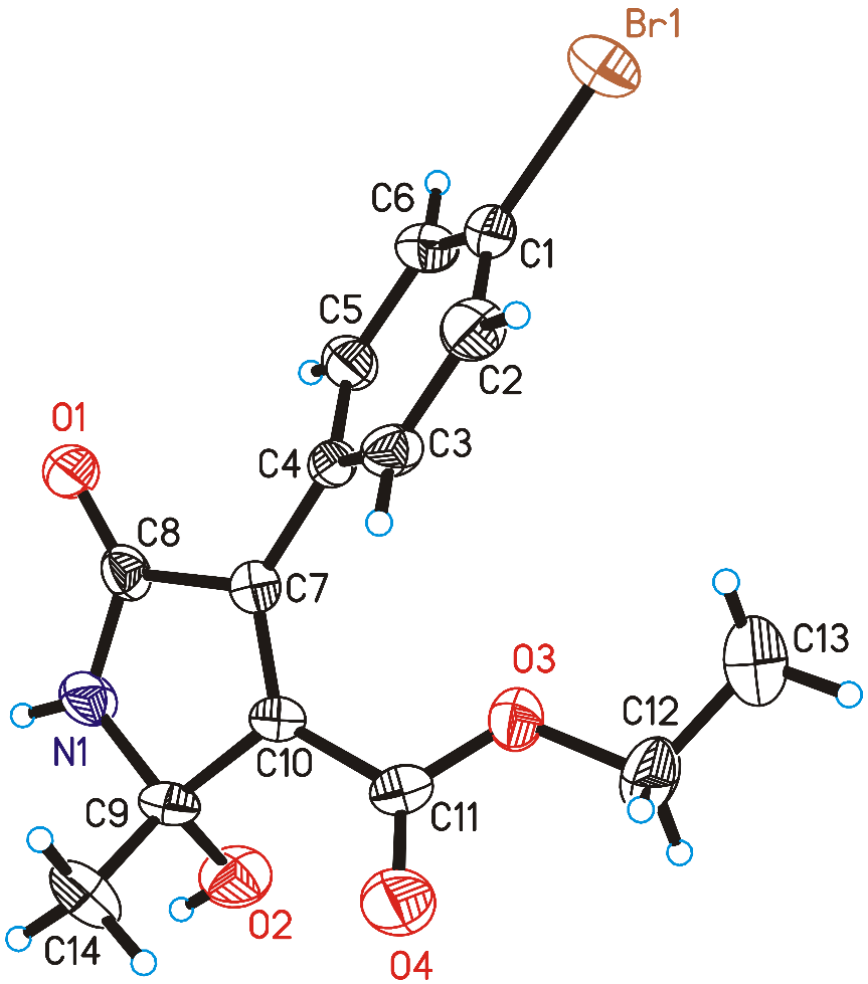


Figure S1. ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **2f** determined at 293 K.

Table S1 Crystal data and structure refinement details for **2f**.

Compound	2f
Empirical formula	C_{14} H_{14} Br N $O_{4.5}$
Formula weight	348.17
Crystal System	Monoclinic
Space group	$P\ 2_1/n$
a (Å)	9.838(4)
b (Å)	15.861(6)
c (Å)	20.361(9)
α (°)	90.00
β (°)	101.9810(10)
γ (°)	90.00
V (Å^3)	3108(2)
Z	8
D_c (g/cm^3)	1.488
F_{000}	1408
μ (mm^{-1})	2.660
θ_{max} (°)	25.36
Total reflections	19146
Unique reflections	5580
Reflections [$I > 2\sigma(I)$]	2173
Parameters	376
R_{int}	0.1136
Goodness-of-fit	1.007
R [$F^2 > 2\sigma(F^2)$]	0.0731
wR (F^2, all data)	0.1412
CCDC No.	2006864

X-Ray Data Collection and Structure Refinement Details for compound 4e:

A good quality single crystal of size 0.43 x 0.23x 0.19 mm, was selected under a polarizing microscope and was mounted on a glass fiber for data collection. Single crystal X-ray data for compound **4e** were collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-Kα radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collection was performed using ω-scans of 0.5^o steps at 293(2) K. Cell determination, data collection and data reduction was performed using the Rigaku CrystalClear-SM Expert 2.1 b24 software.[1] Structure solution and refinement were performed by using SHELXTL-NT.[2] Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.

Crystallization: The compound **4e (5mg)** was dissolved in a 1mL mixture of *n*-hexane/DCM/acetone (2:1:1) and placed in a cabinet to evaporate slowly. After two days, **4e** was obtained as white crystal.

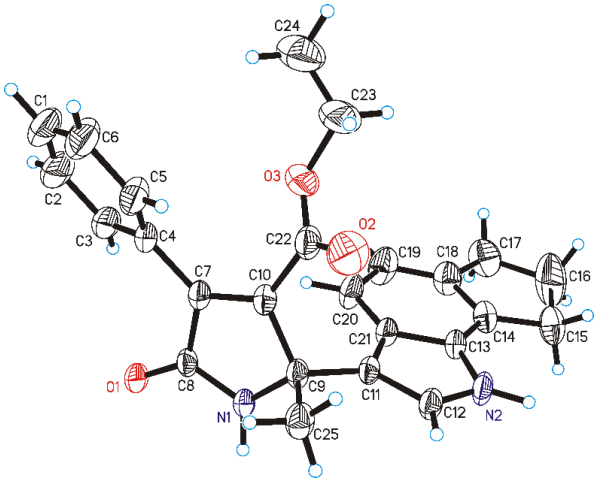
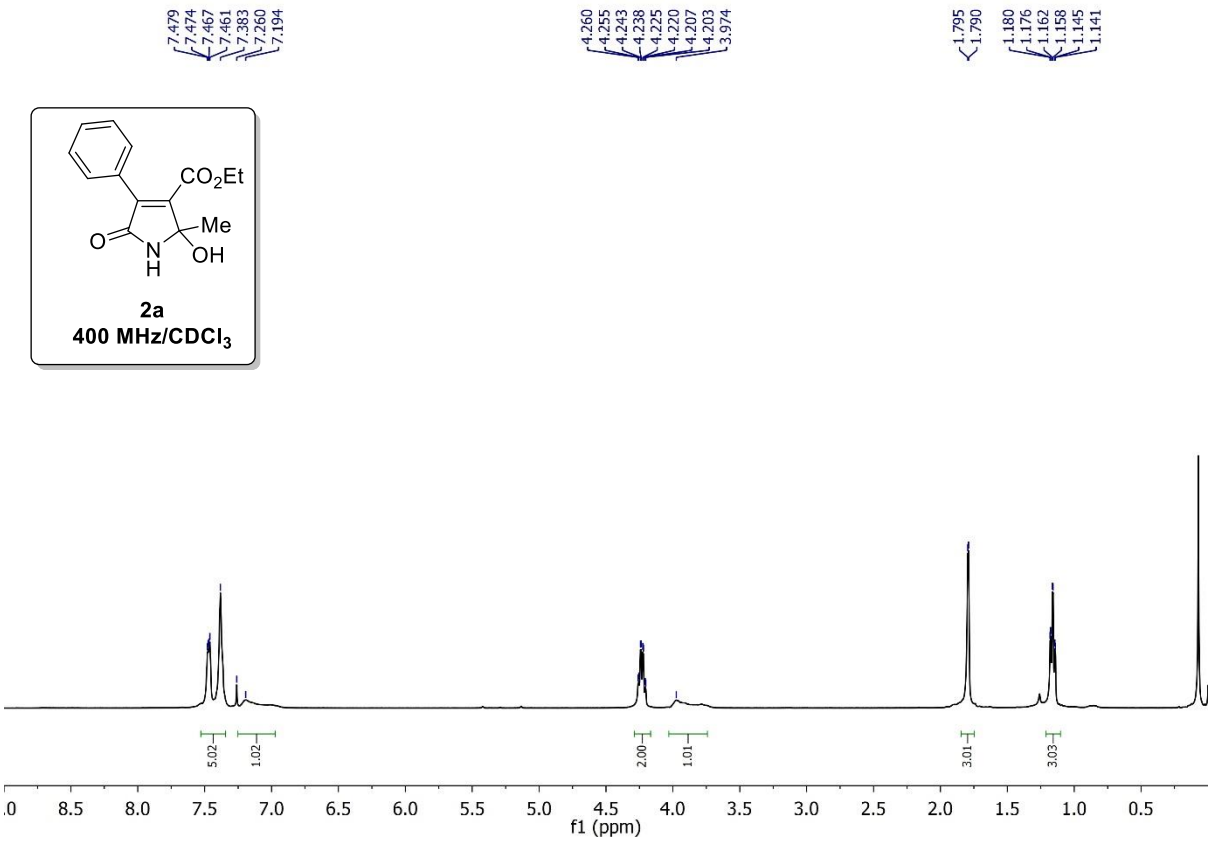


Figure S2. ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **4e** determined at 293 K.

Table S2 Crystal data and structure refinement details for **4e**.

Compound	**4e**
Empirical formula	$C_{25}H_{24}N_2O_3$
Formula weight	400.46
Crystal System	Triclinic
Space group	*P*-1
a (Å)	9.3094(3)
b (Å)	9.4648(3)
c (Å)	13.4239(4)
α (°)	70.898(3)
β (°)	76.491(3)
γ (°)	77.185(3
V (Å^3)	1073.07(6)
Z	2
D_c (g/cm^3)	1.239
F_{000}	424
μ (mm^{-1})	0.656
θ_{max} (°)	72.80
Total reflections	21777
Unique reflections	4045
Reflections [$I > 2\sigma(I)$]	3638
Parameters	282
R_{int}	0.1291
Goodness-of-fit	1.084
R [$F^2 > 2\sigma(F^2)$]	0.0756
wR (F^2, all data)	0.2164
CCDC No.	2009054

1. CrystalClear 2.1, Rigaku Corporation, Tokyo, Japan
2. Sheldrick, G. M. *Acta Crystallogr., Sect. A* **2008**, *64*, 112–122.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4.207
4.203
3.974
1.795
1.790
1.180
1.176
1.162
1.158
1.145
1.141
CO2Et
Me
OH
O
N
H
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1.01
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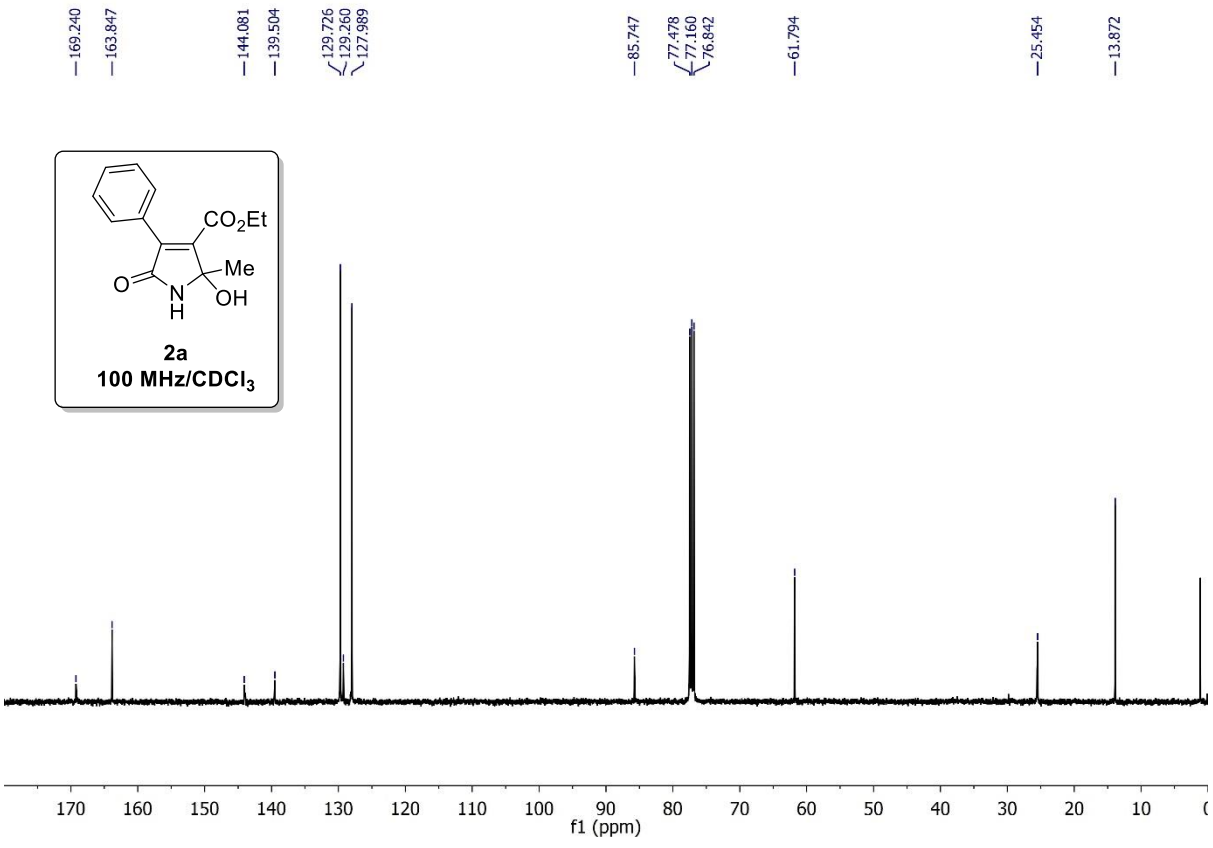
3.03
f1 (ppm)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H
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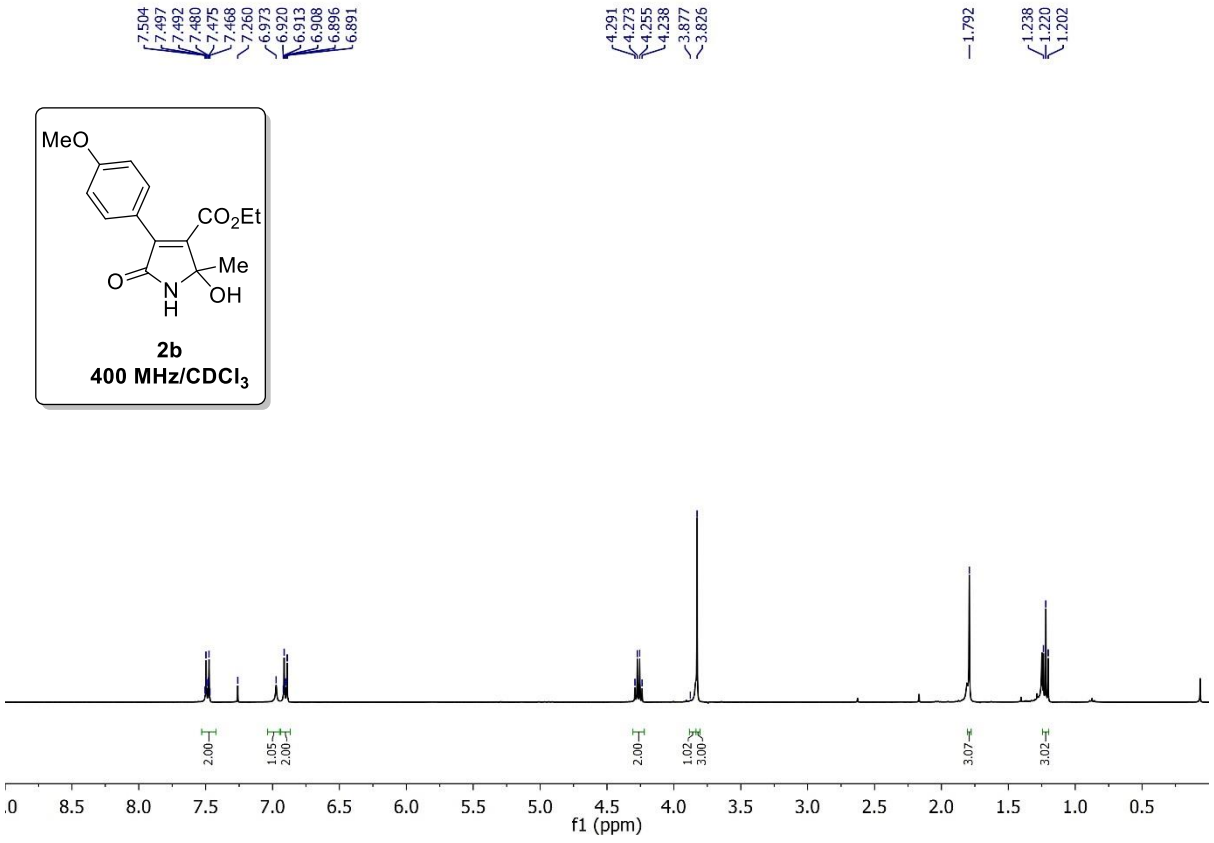
100 MHz/CDCl3
f1 (ppm)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4.238
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3.826
1.792
1.238
1.220
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CO2Et
Me
O
N
H
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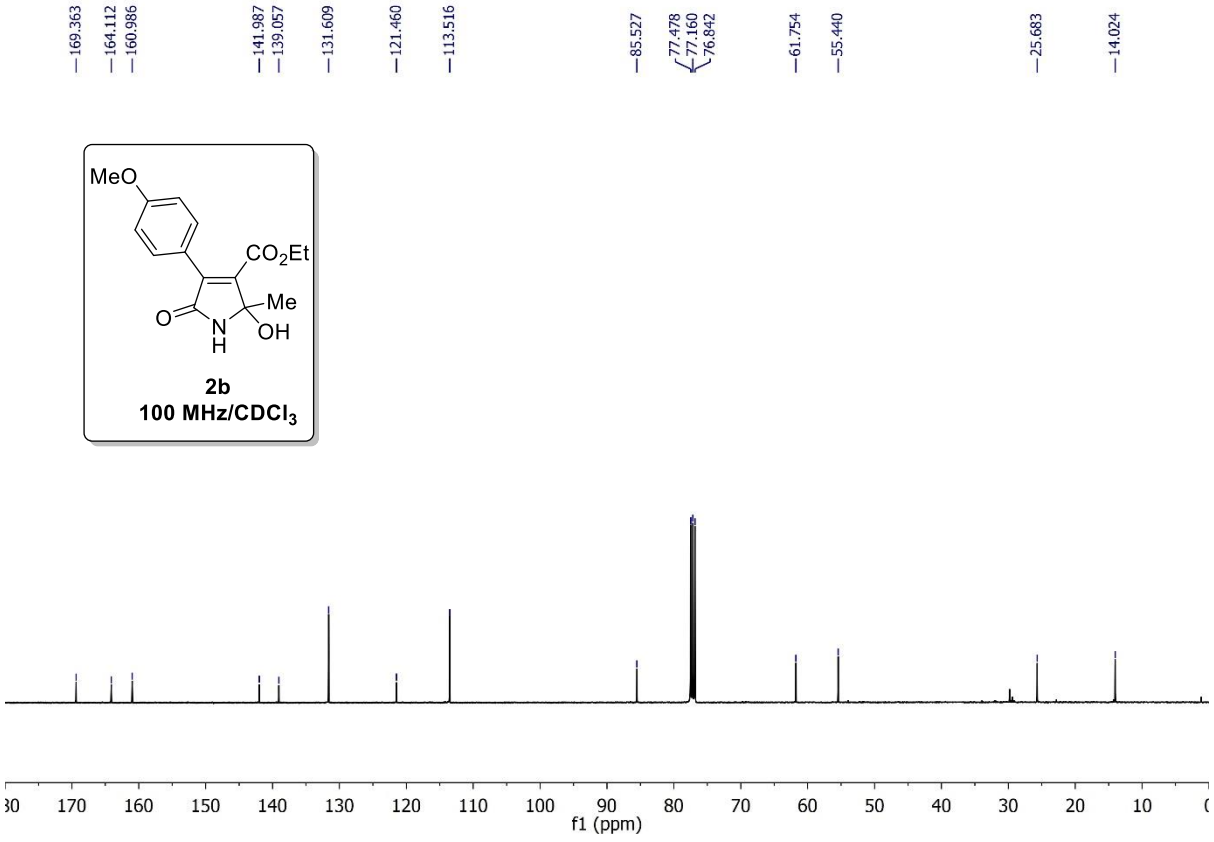
f1 (ppm)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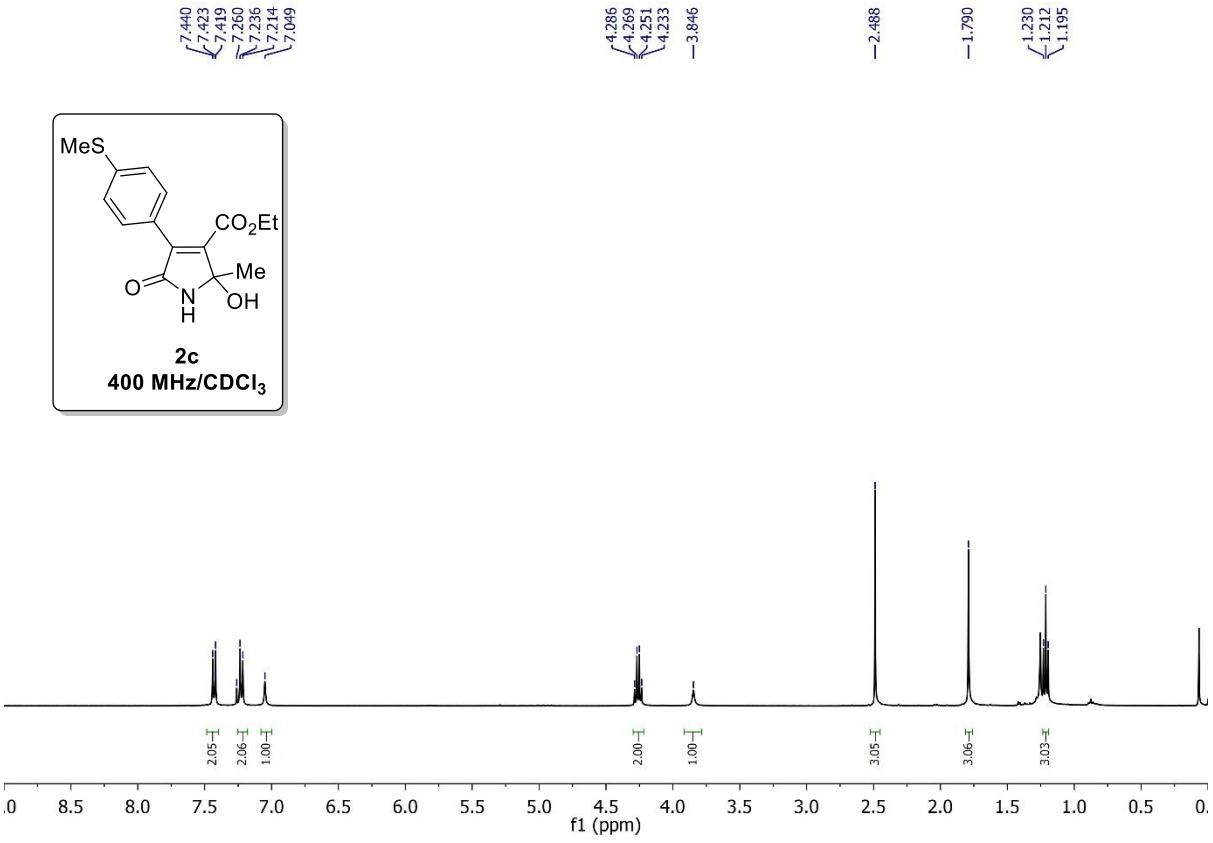
CO2Et
Me
O
N
H
OH
2b
100 MHz/CDCl3
f1 (ppm)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1.212
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MeS
CO2Et
Me
O
N
H
OH
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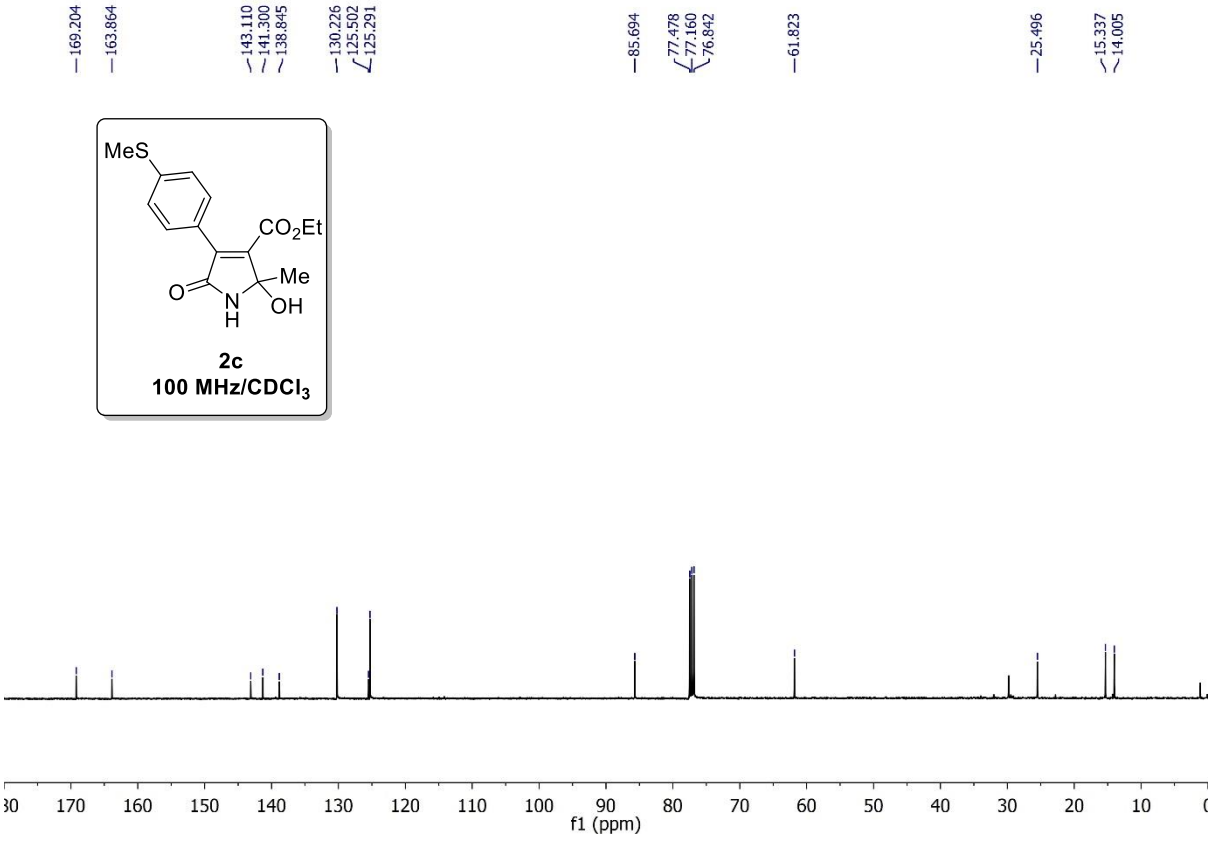
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H
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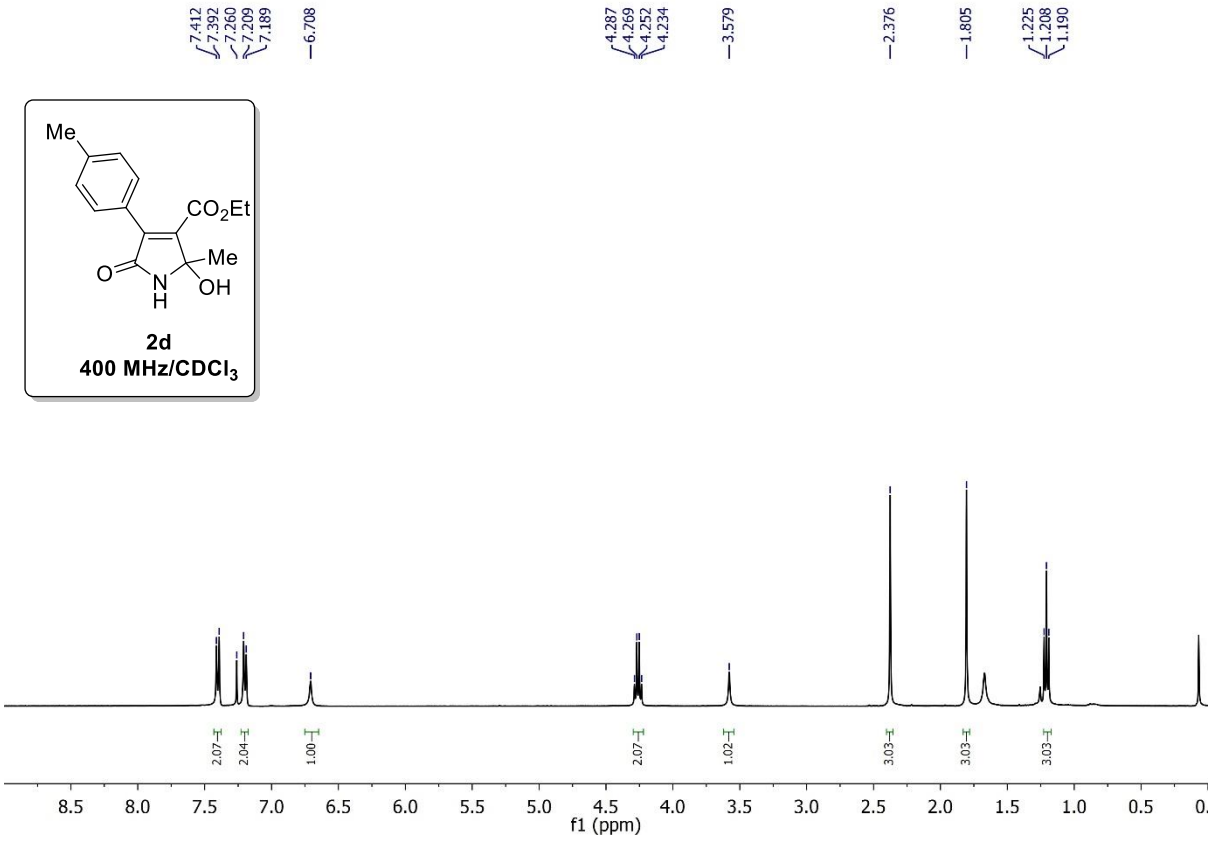
2c
100 MHz/CDCl3
f1 (ppm)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O
N
H
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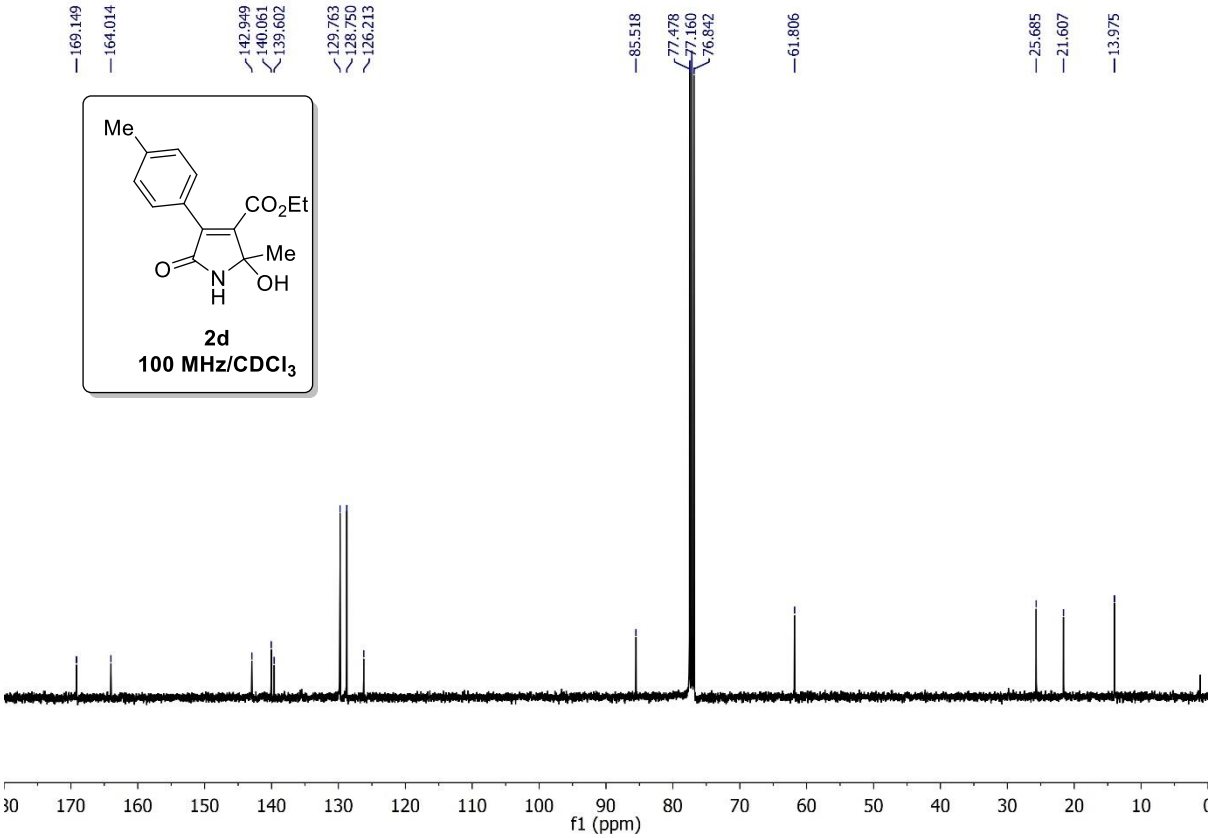
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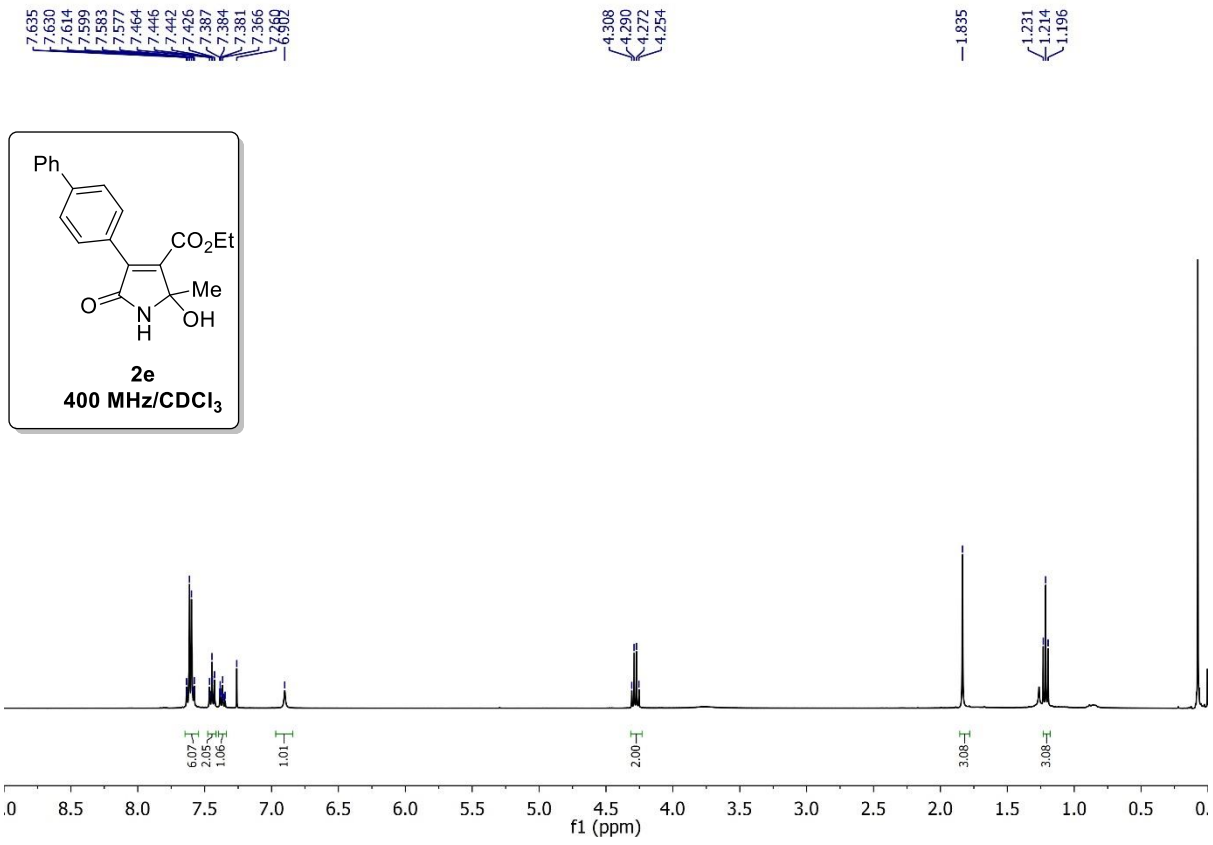
H
OH
2d
100 MHz/CDCl3
f1 (ppm)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1.231
1.214
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Ph
CO2Et
Me
OH
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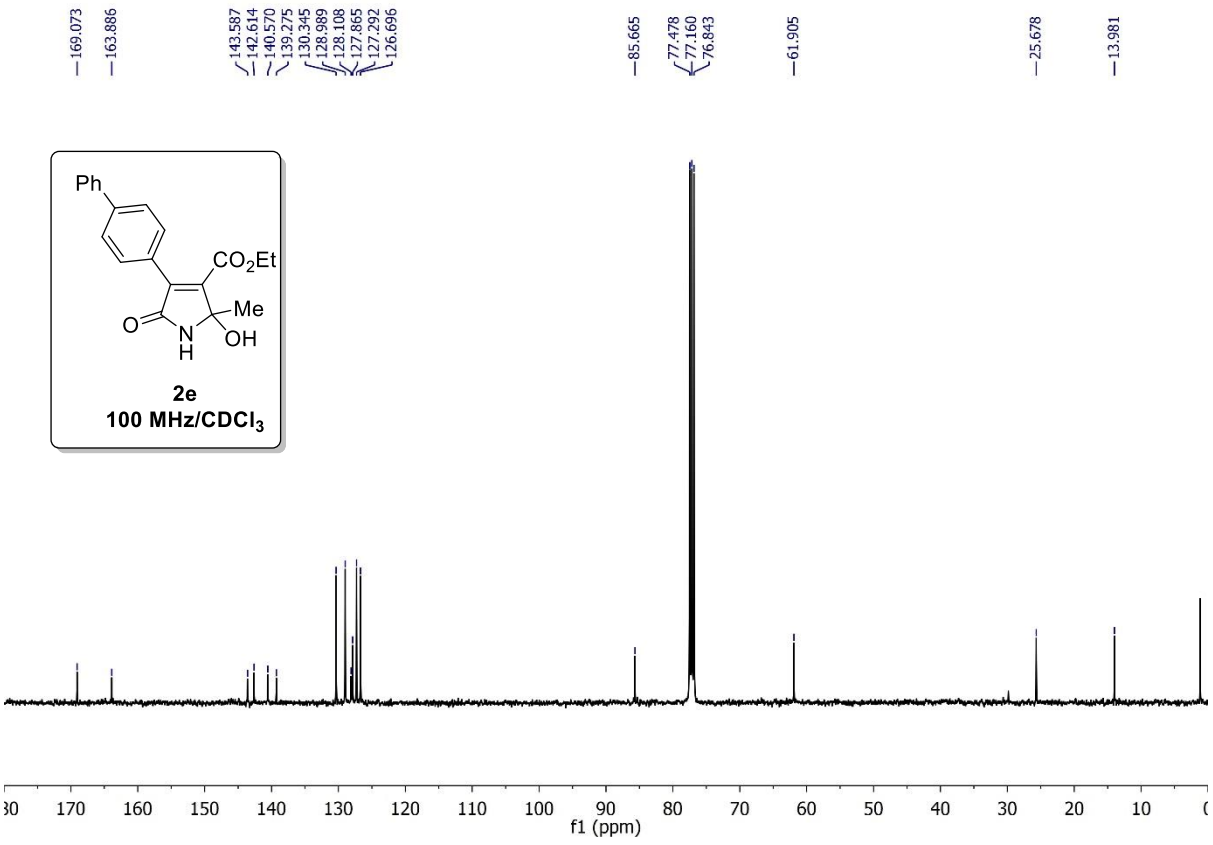
3.08
f1 (ppm)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H
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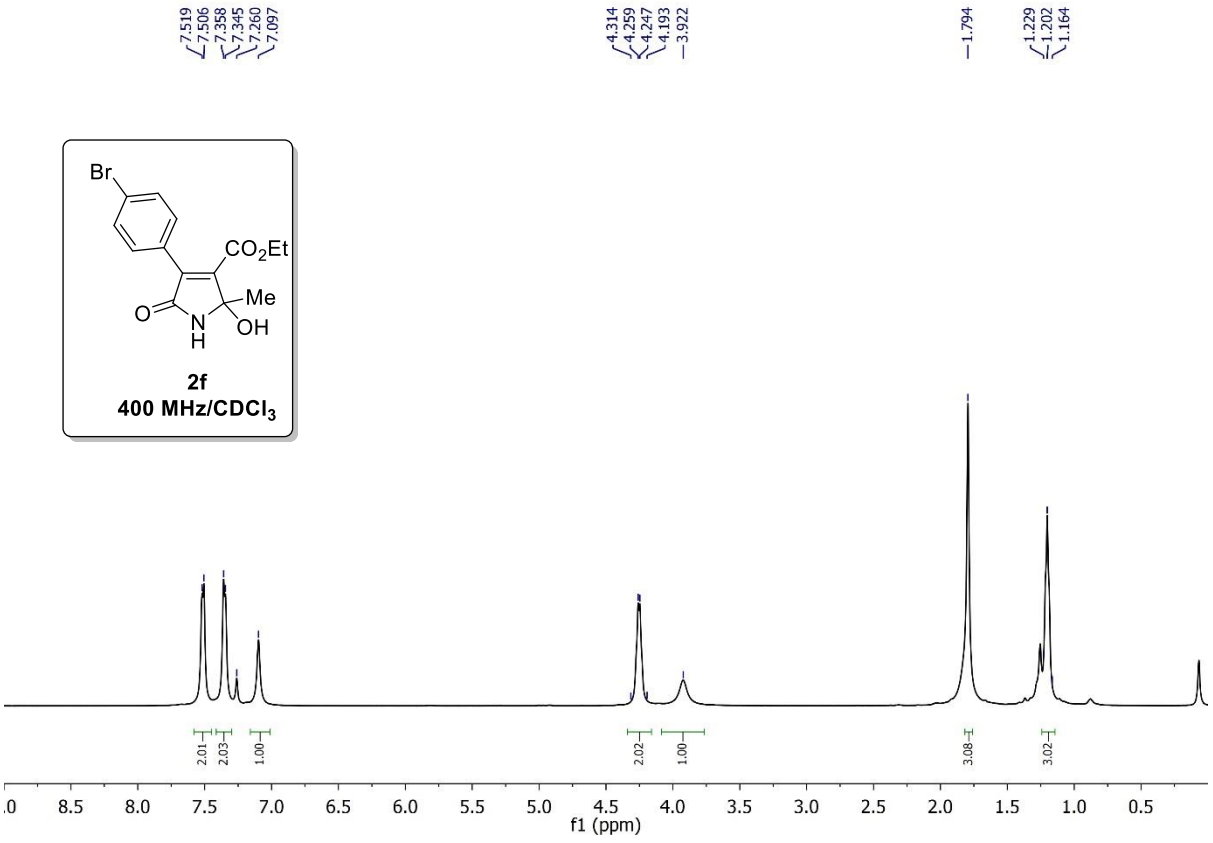
100 MHz/CDCl3
f1 (ppm)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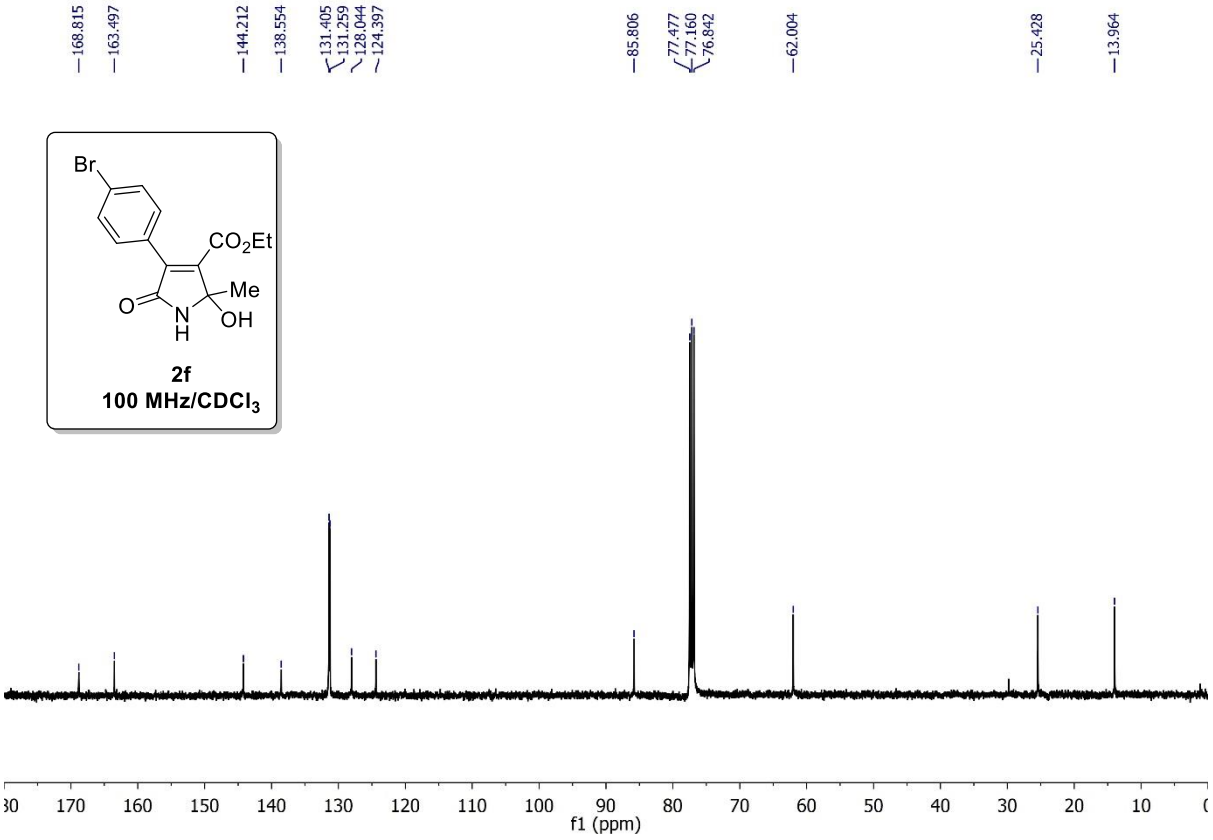
f1 (ppm)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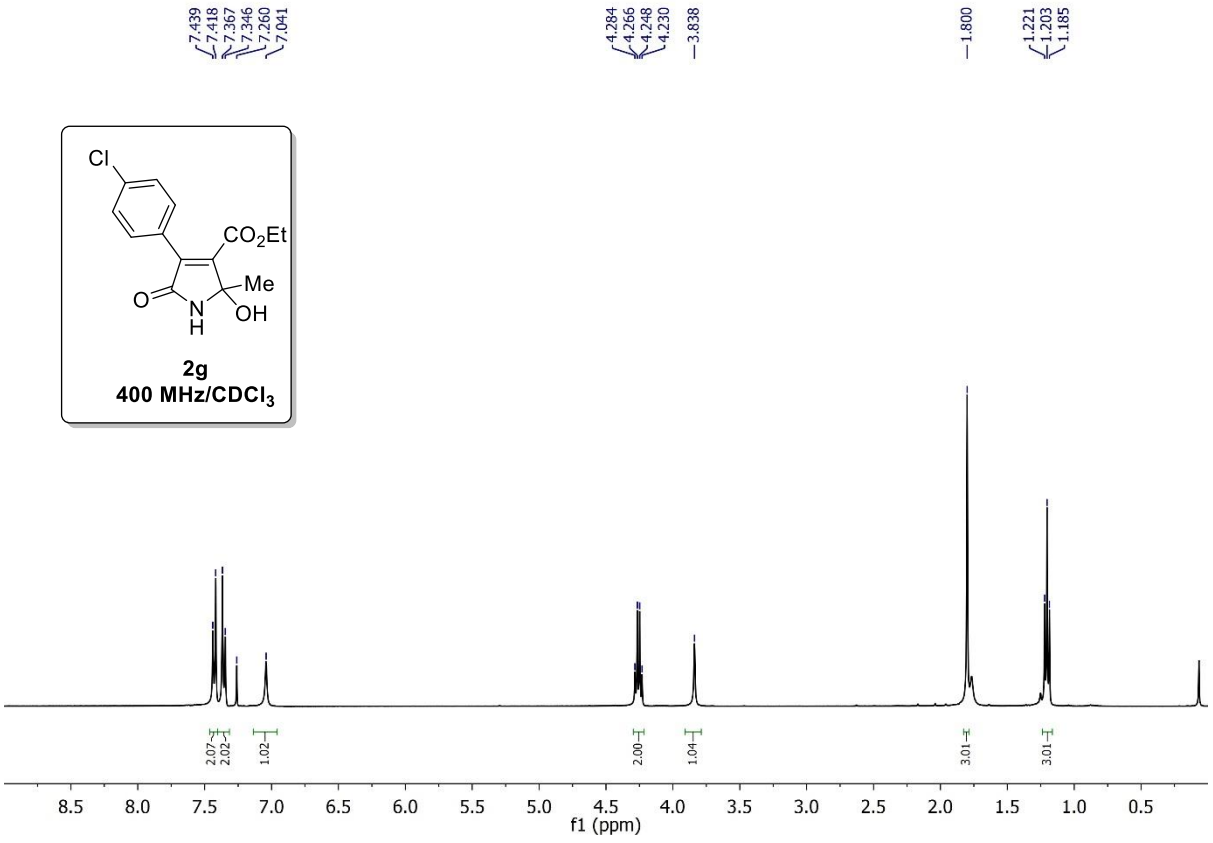
100 MHz/CDCl3
f1 (ppm)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CO2Et
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400 MHz/CDCl3
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1.02
2.00
1.04
3.01
3.01
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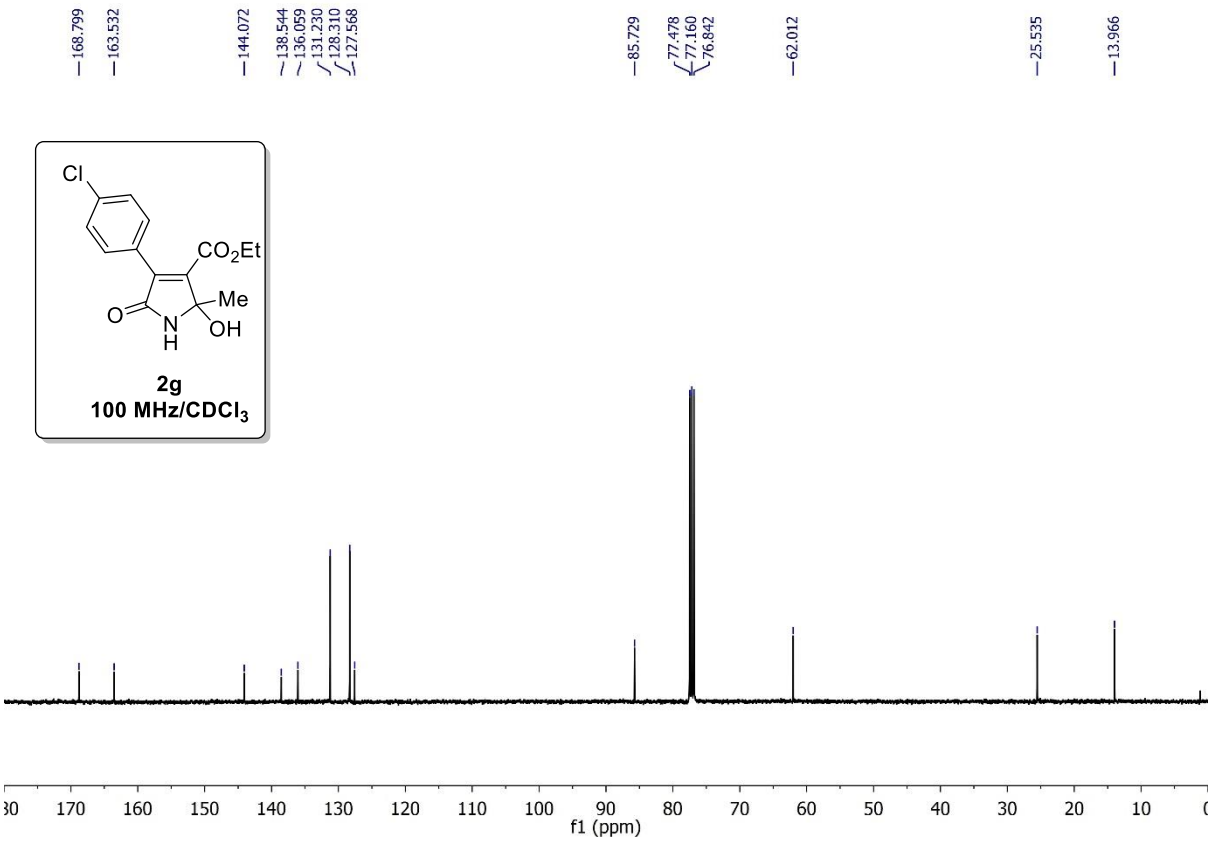
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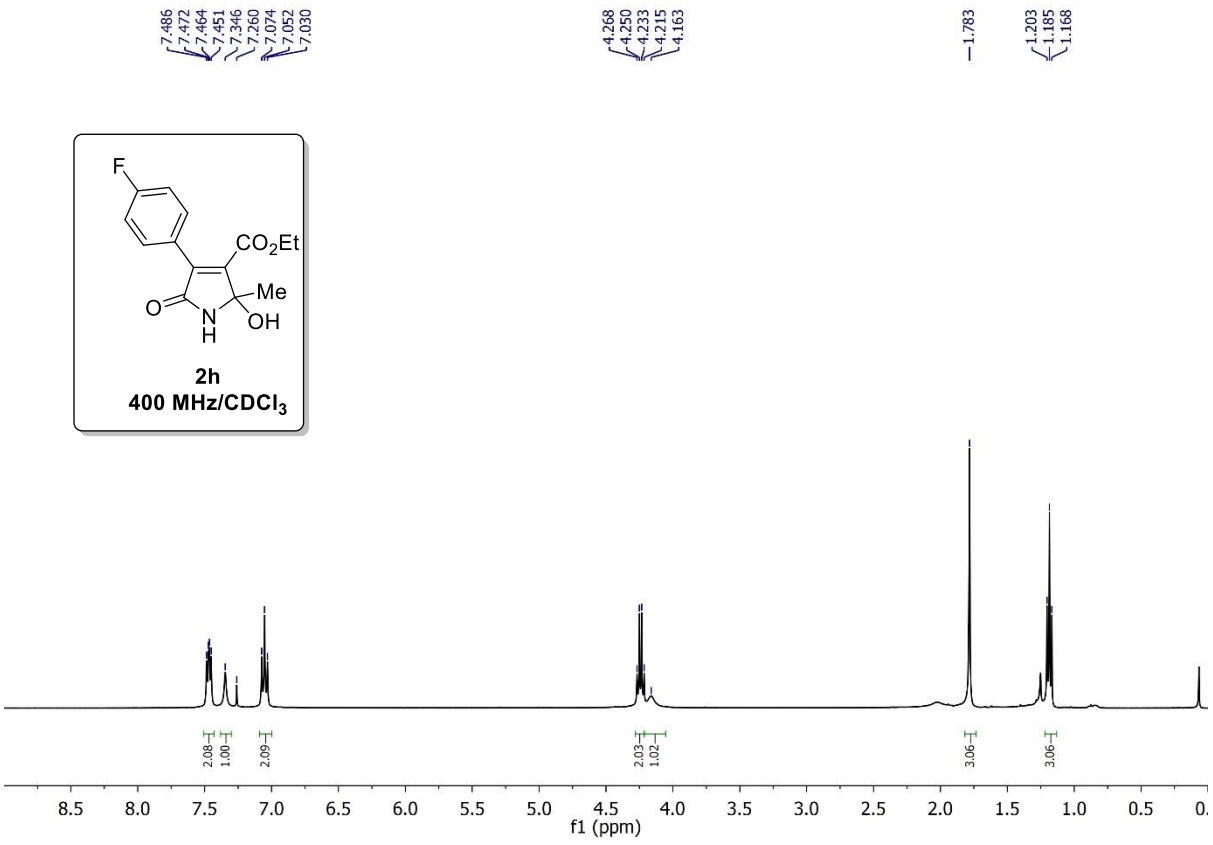
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1.185
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F
CO2Et
Me
O
N
H
OH
2h
400 MHz/CDCl3
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1.00
2.09
2.03
1.02
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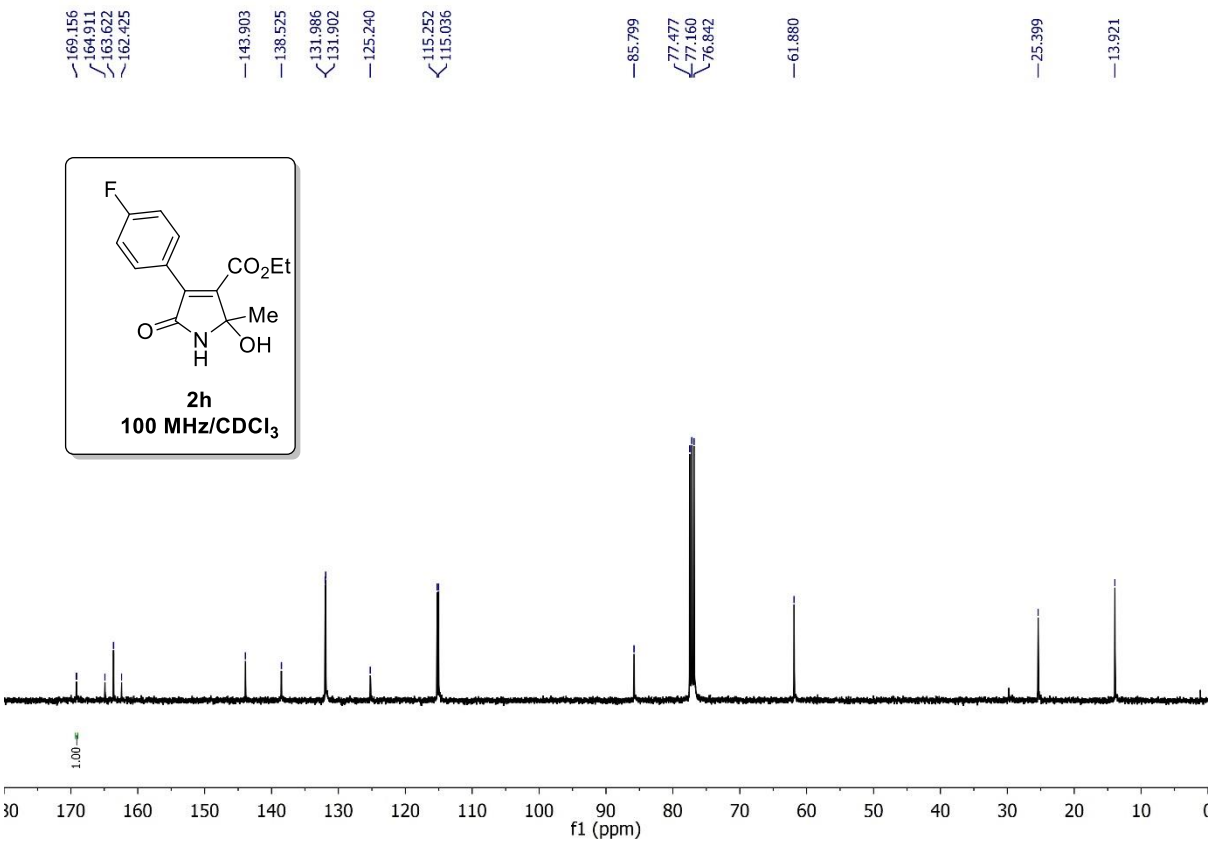
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100 MHz/CDCl3
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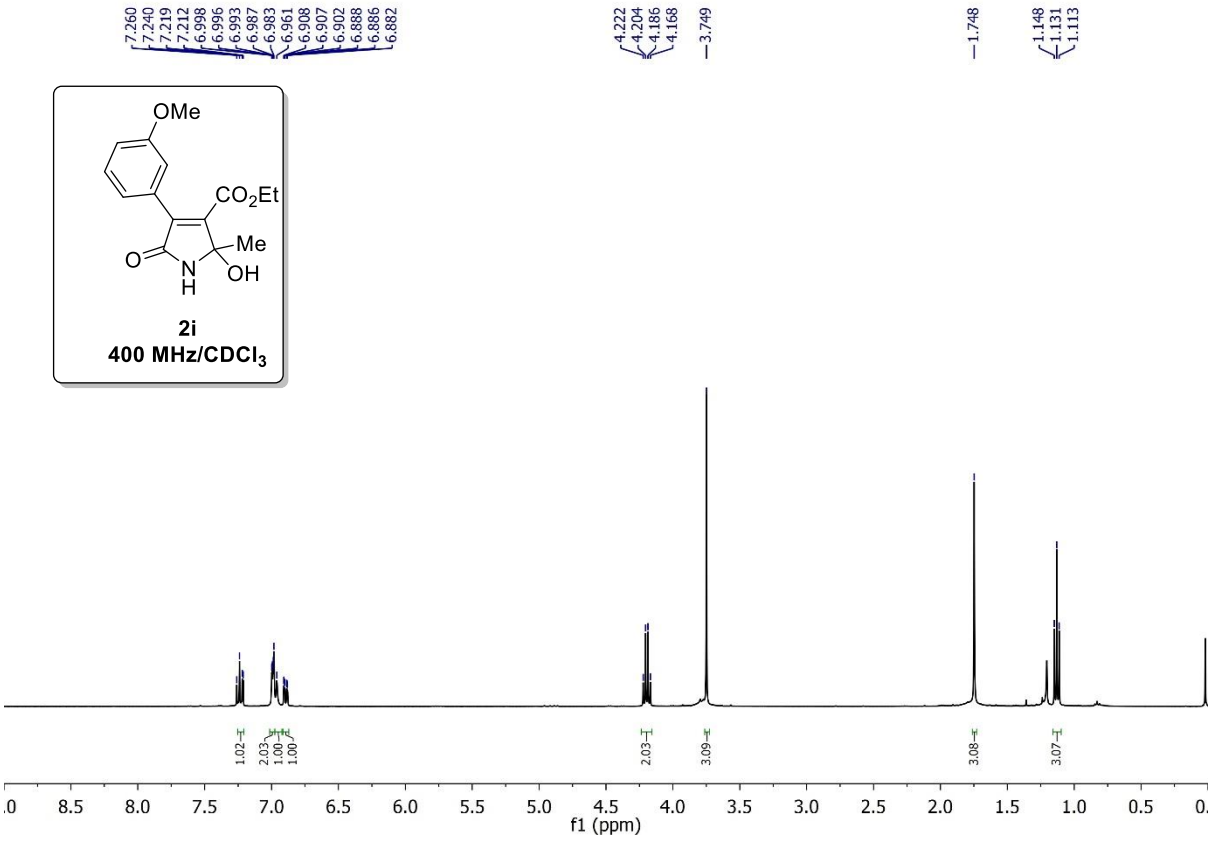
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O
N
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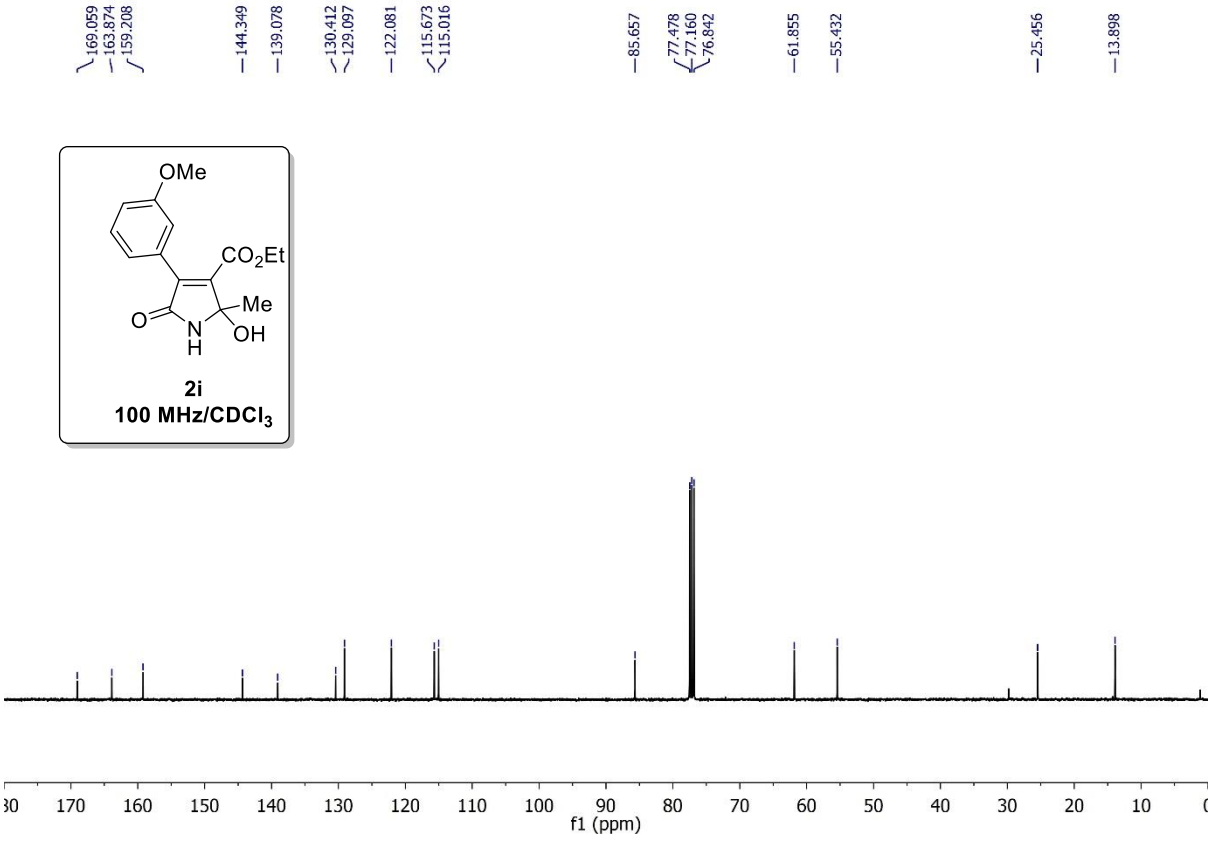
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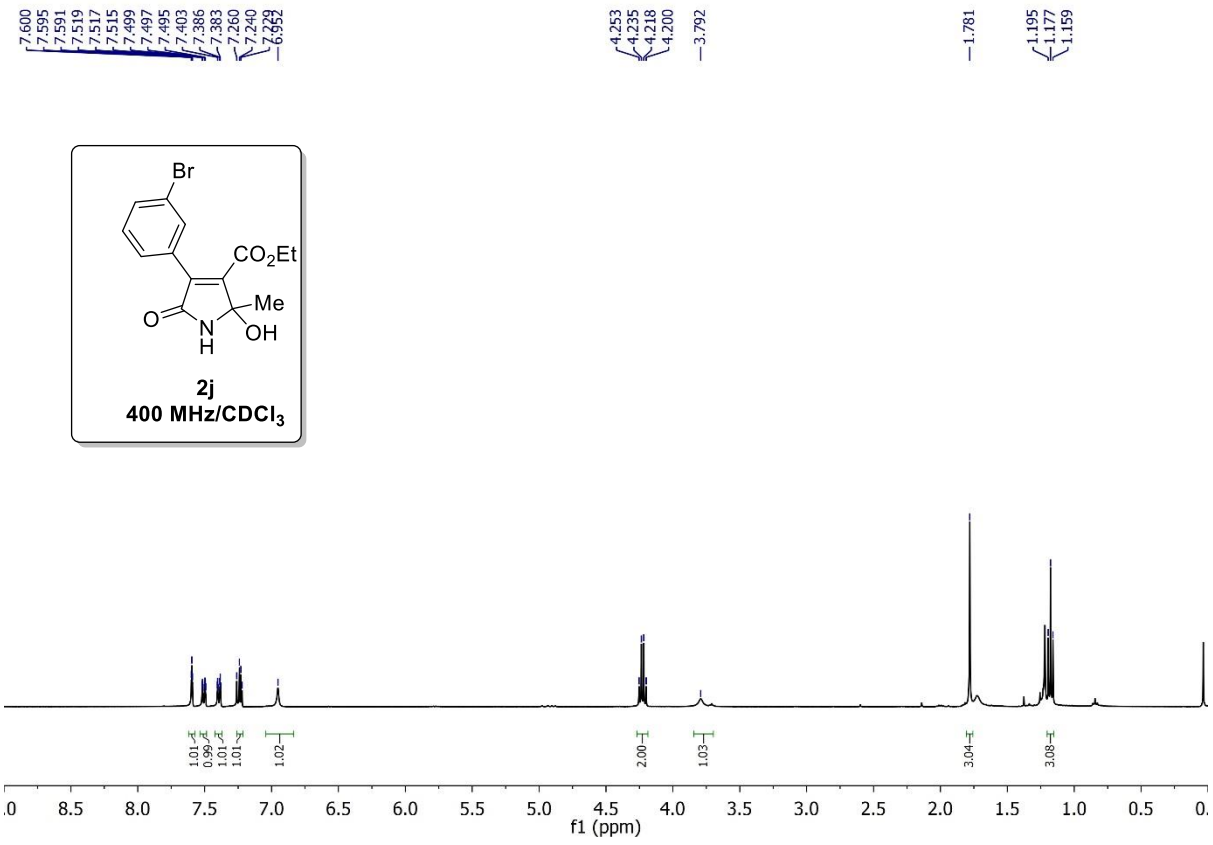
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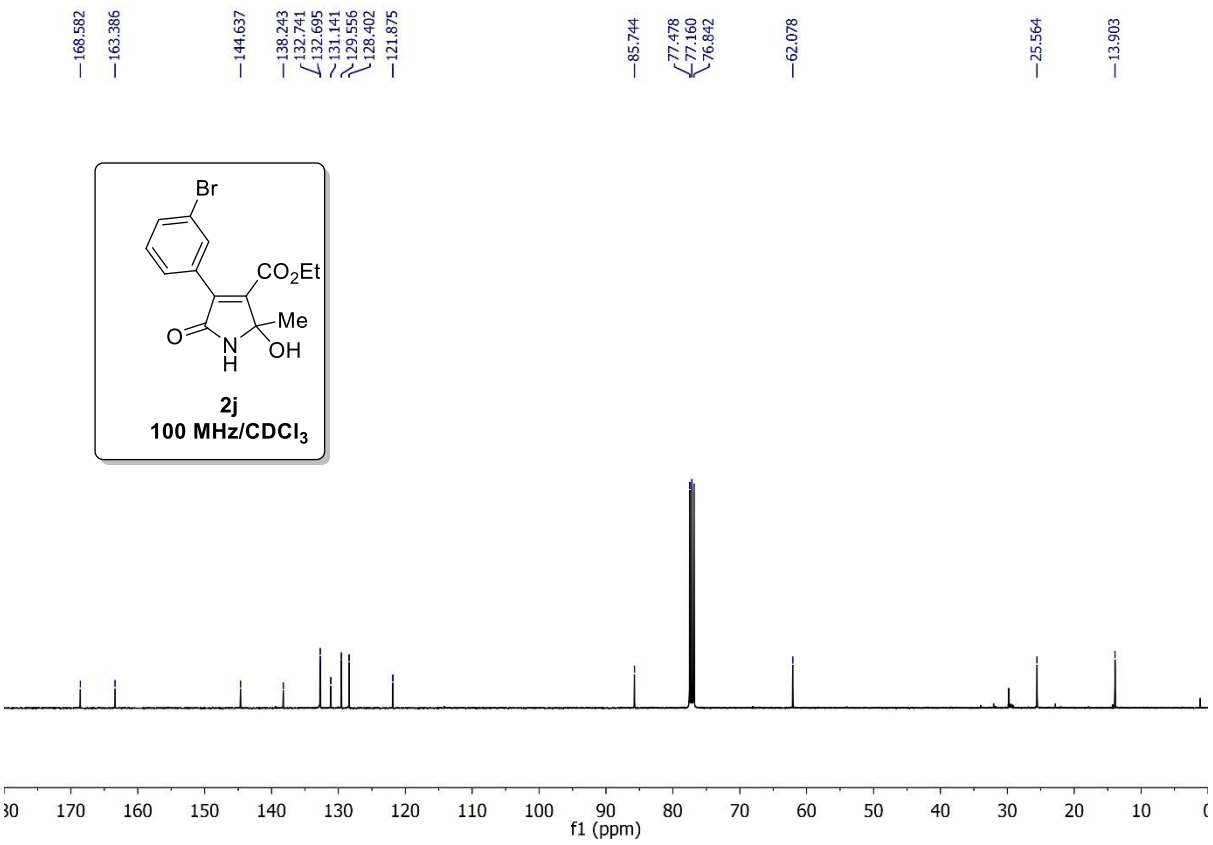
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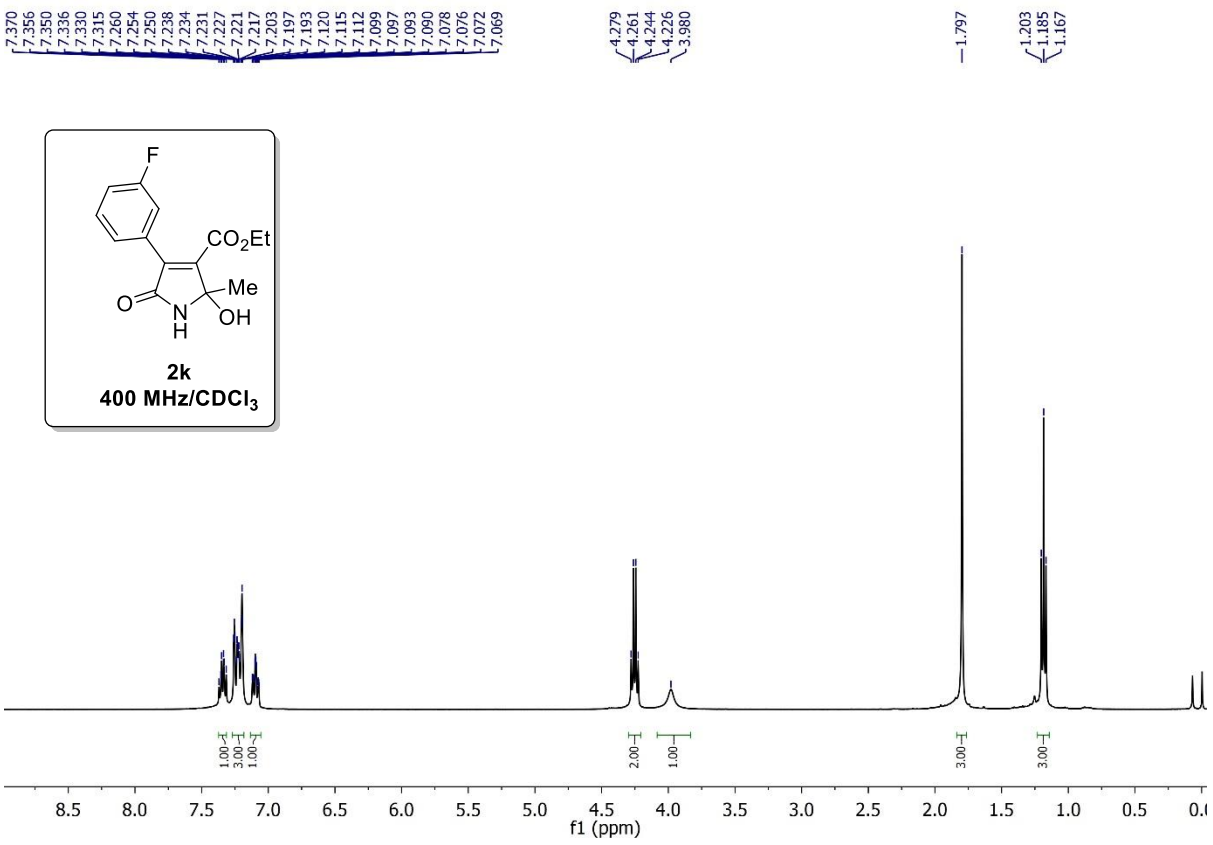
100 MHz/CDCl3
f1 (ppm)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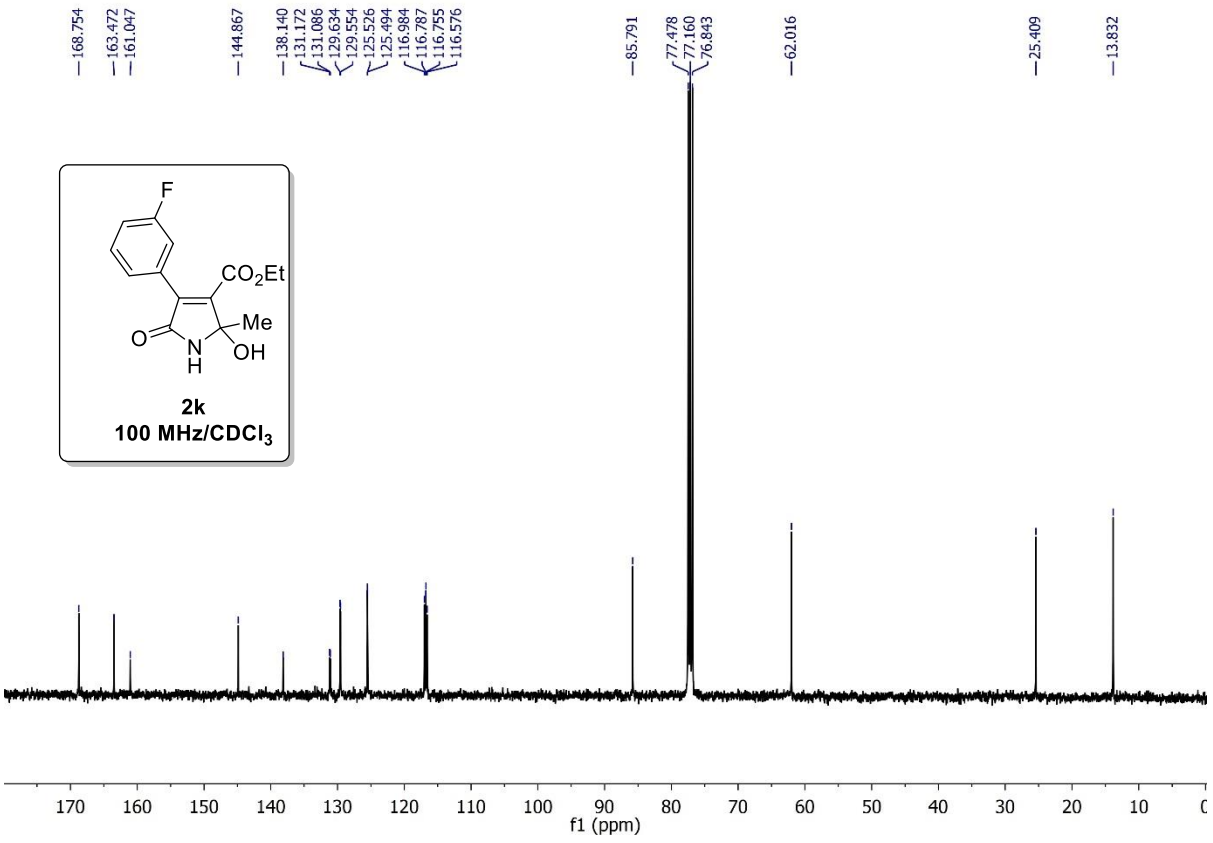
1.203
1.185
1.167
F
CO2Et
Me
O
N
H
OH
2k
400 MHz/CDCl3
1.00
3.00
1.00
2.00
1.00
3.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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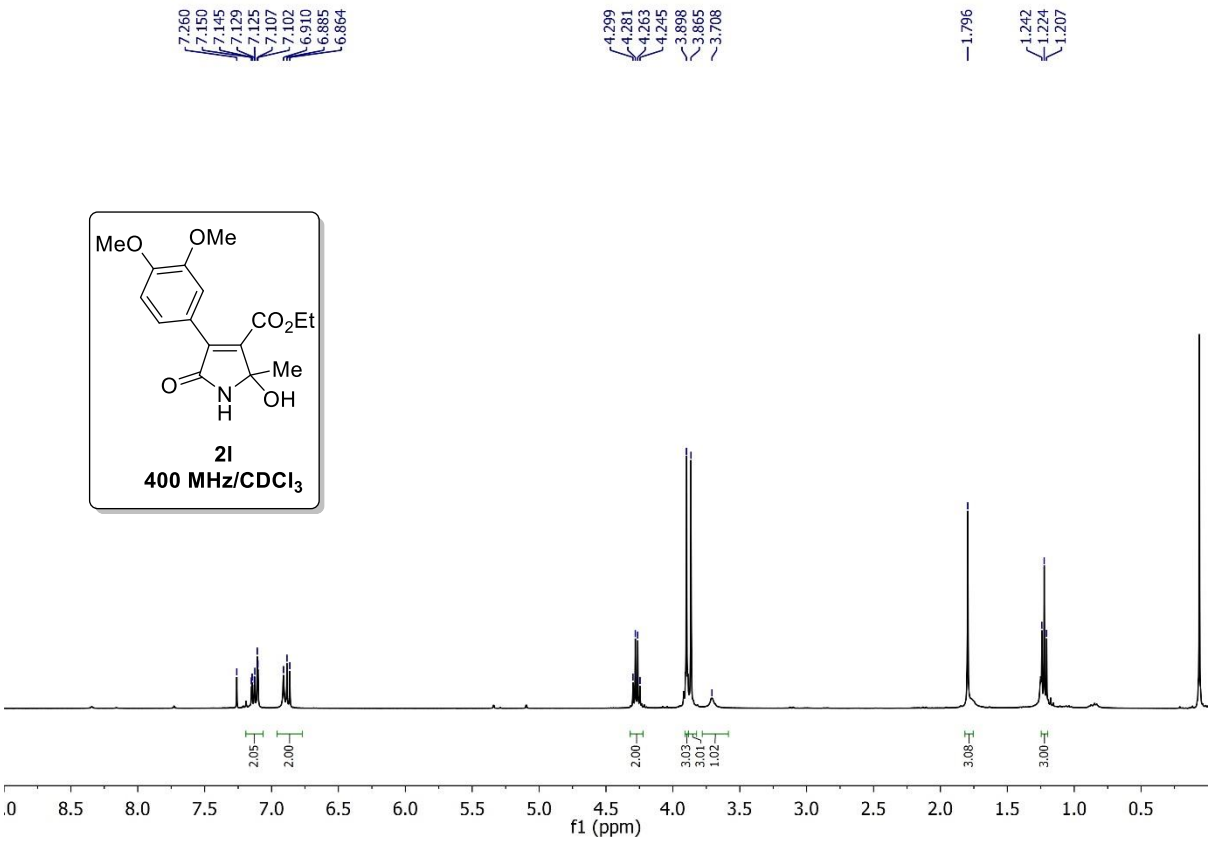
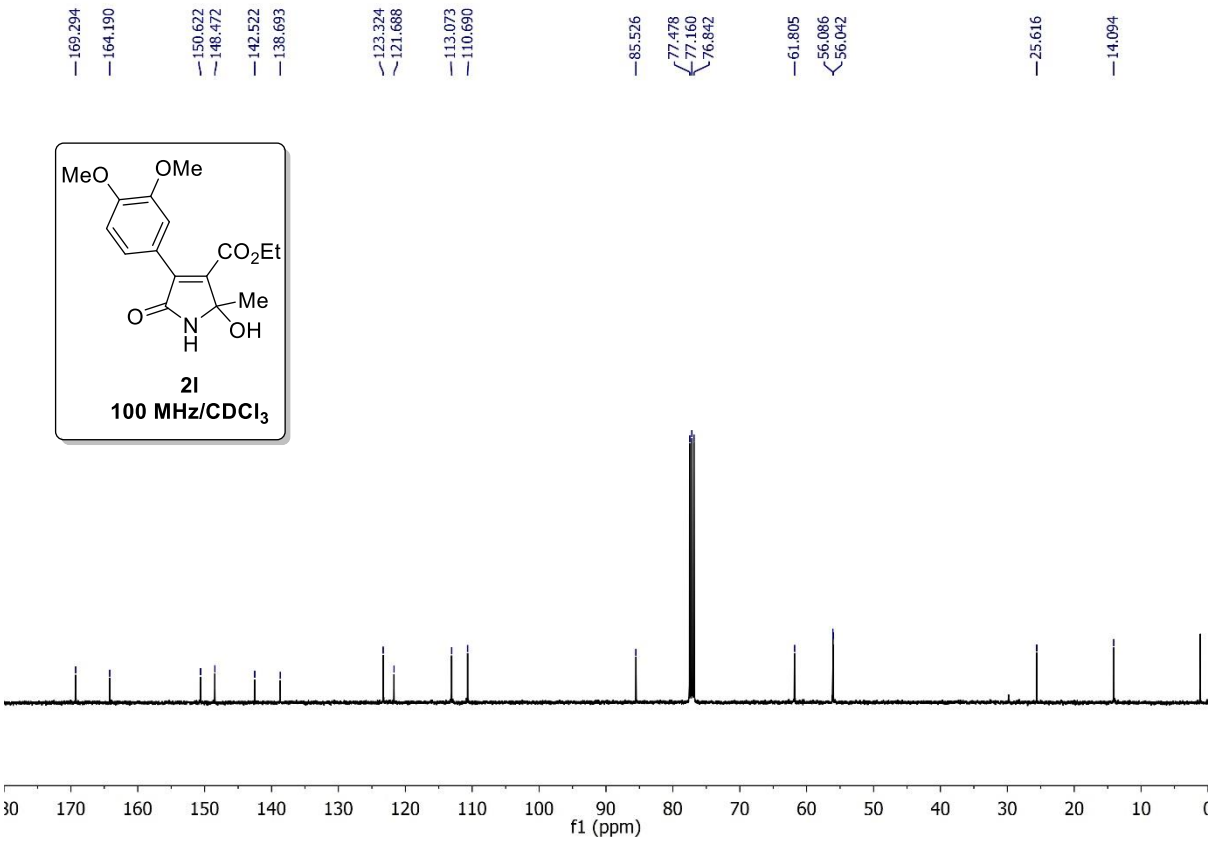
163.472
161.047
144.867
138.140
131.172
131.086
129.634
129.554
125.526
125.494
116.984
116.787
116.755
116.576
85.791
77.478
77.160
76.843
62.016
25.409
13.832
F
CO2Et
Me
O
N
H
OH
2k
100 MHz/CDCl3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.260
7.150
7.145
7.129
7.125
7.107
7.102
6.910
6.885
6.864
4.299
4.281
4.263
4.245
3.898
3.865
3.708
1.796
1.242
1.224
1.207
MeO
OMe
CO2Et
Me
OH
N
H
O
2l
400 MHz/CDCl3
2.05
2.00
2.00
3.03
3.01
1.02
3.08
3.00
f1 (ppm)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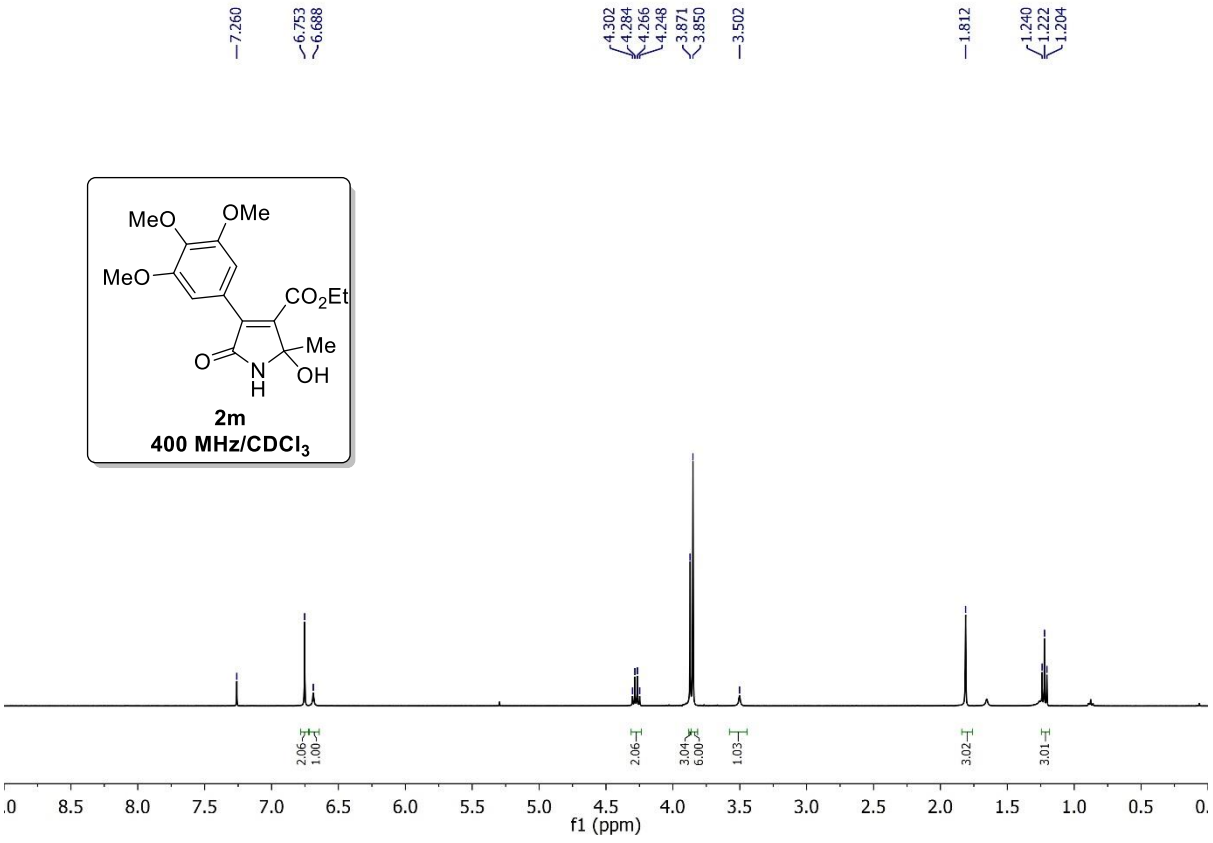
164.190
150.622
148.472
142.522
138.693
123.324
121.688
113.073
110.690
85.526
77.478
77.160
76.842
61.805
56.086
56.042
25.616
14.094
MeO
OMe
CO2Et
Me
OH
N
H
O
2l
100 MHz/CDCl3
f1 (ppm)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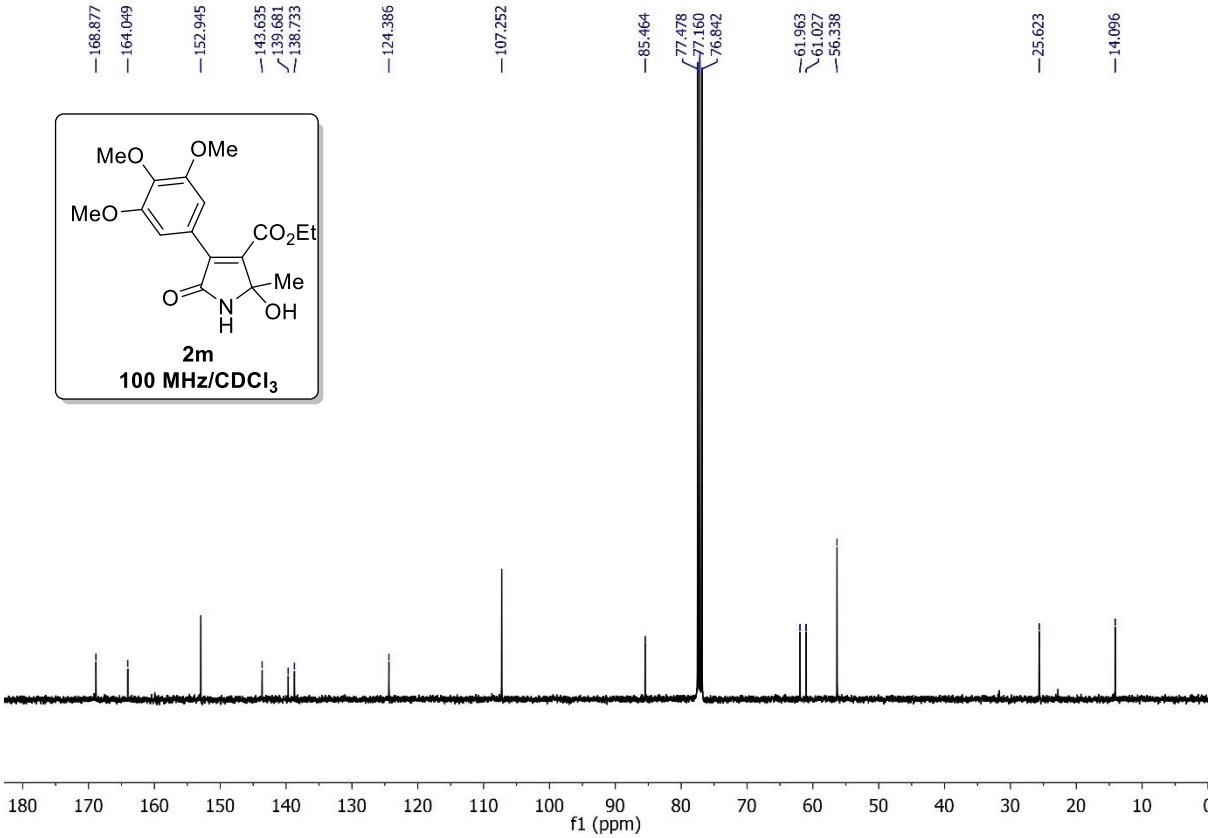
6.753
6.688
4.302
4.284
4.266
4.248
3.871
3.850
3.502
1.812
1.240
1.222
1.204
MeO
OMe
MeO
CO2Et
Me
O
N
H
OH
2m
400 MHz/CDCl3
2.06
1.00
2.06
3.04
6.00
1.03
3.02
3.01
f1 (ppm)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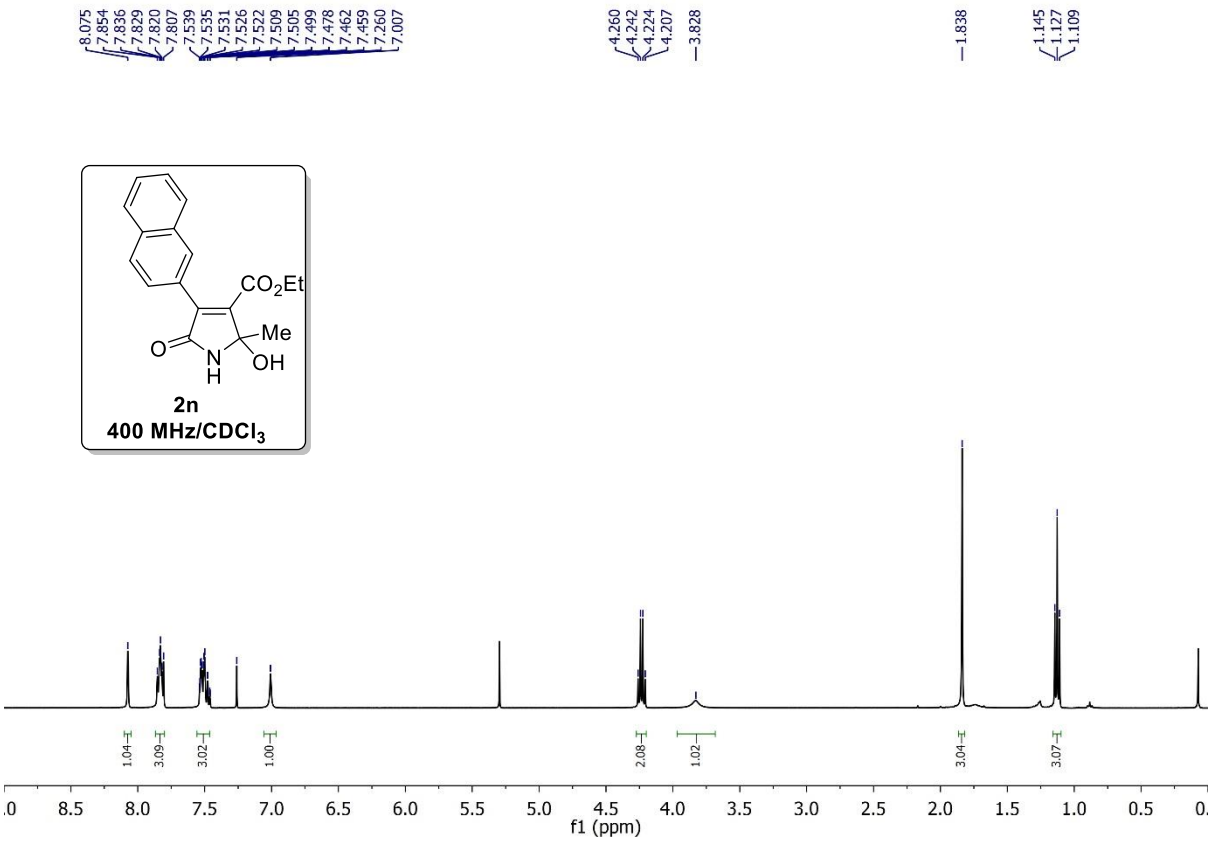
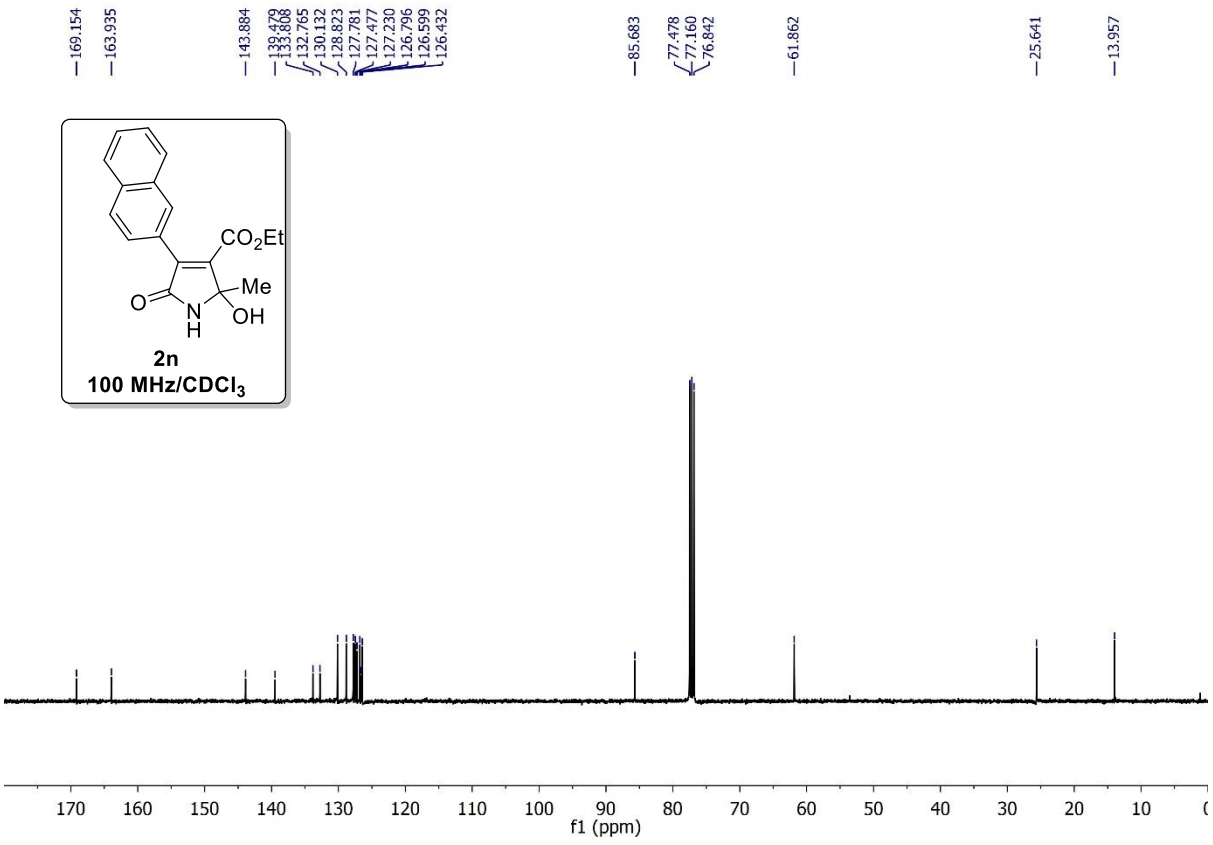
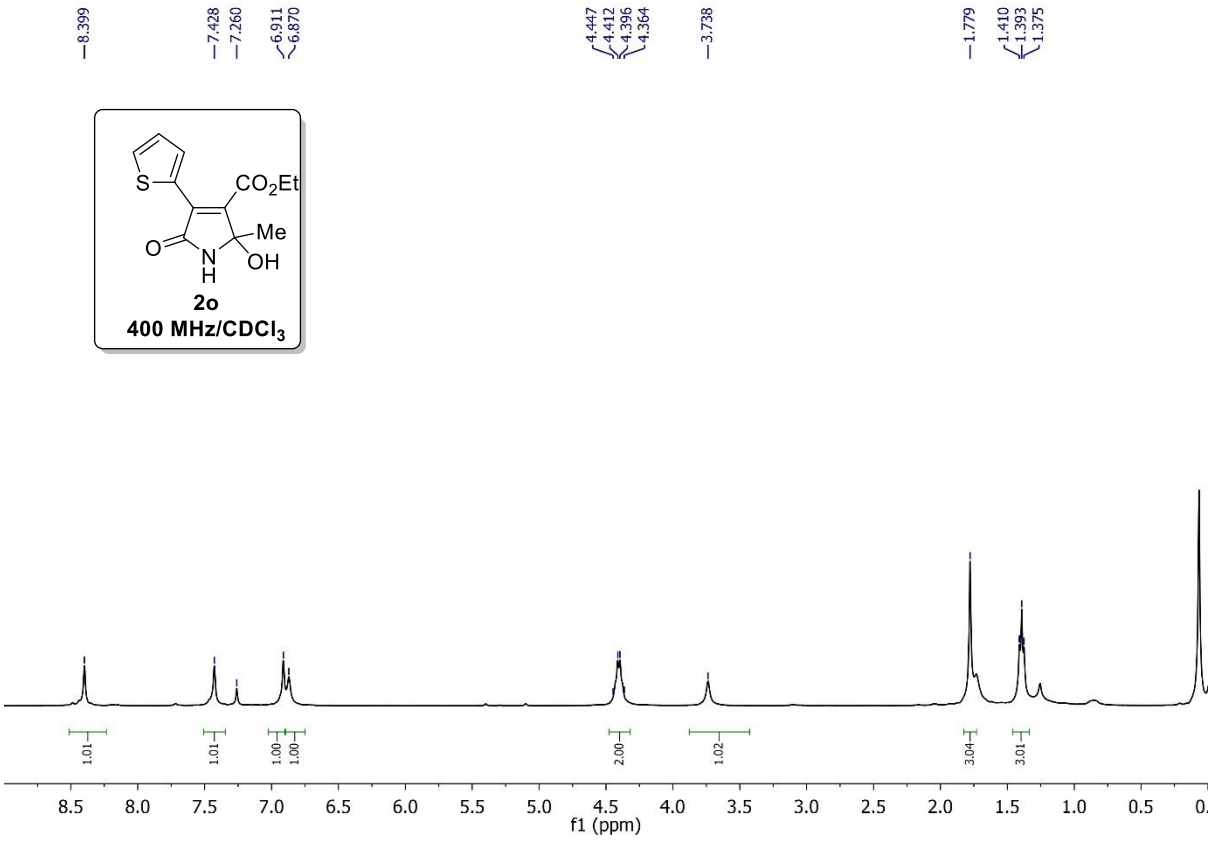
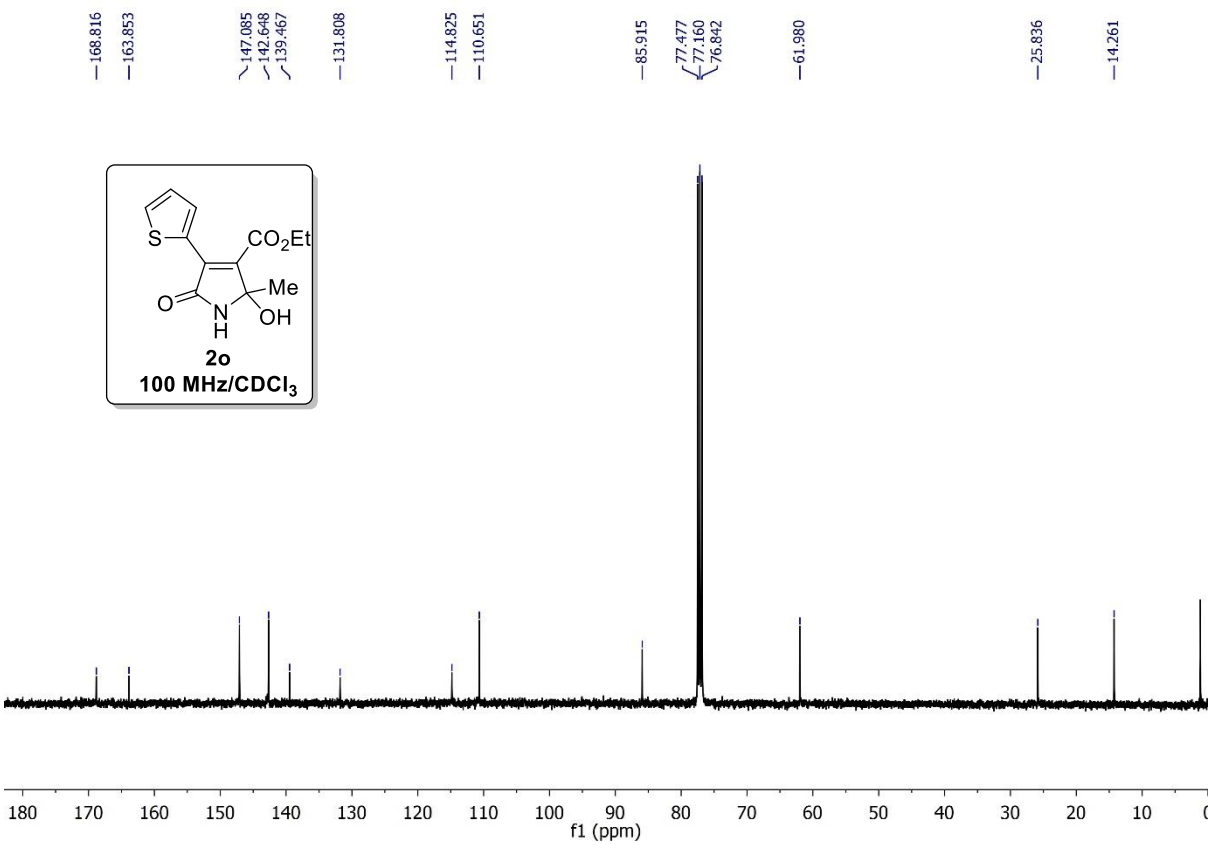
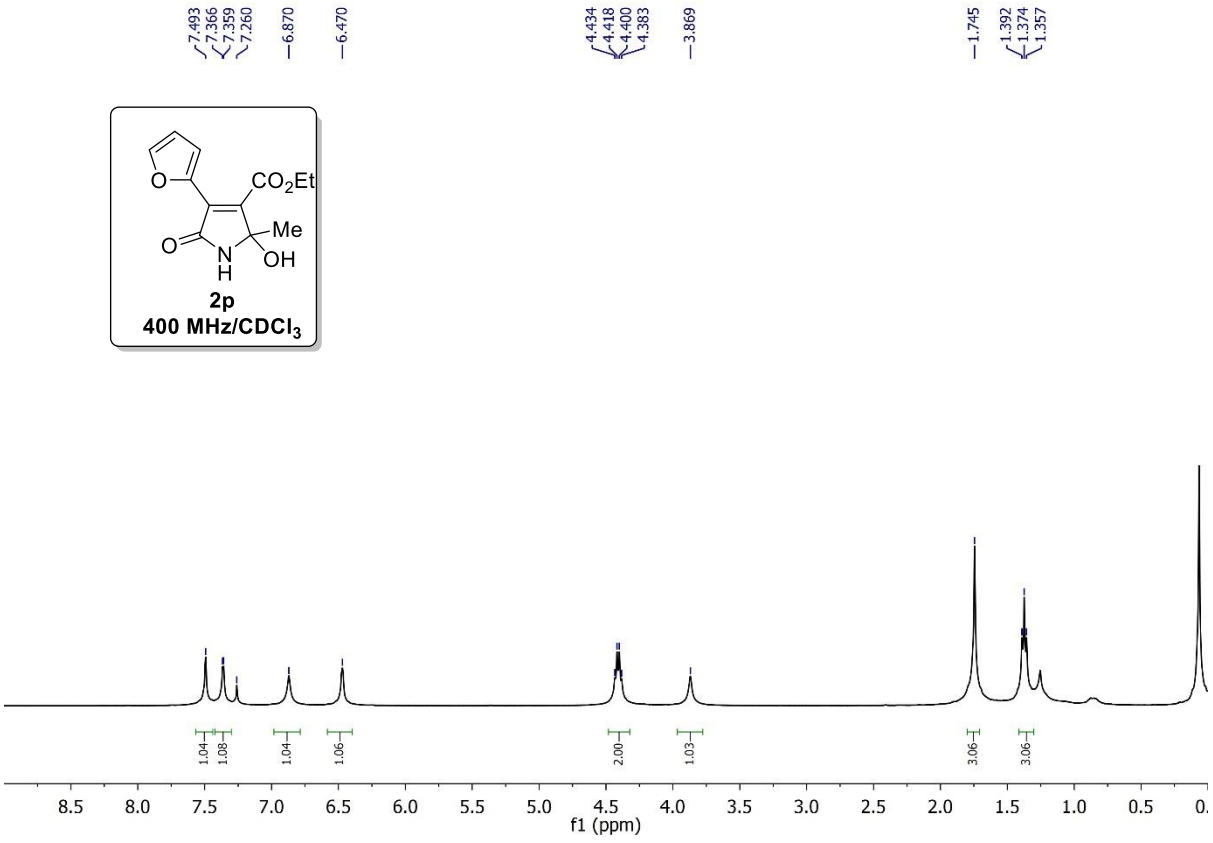
164.049
152.945
143.635
139.681
138.733
124.386
107.252
85.464
77.478
77.160
76.842
61.963
61.027
56.338
25.623
14.096
MeO
OMe
MeO
CO2Et
Me
O
N
H
OH
2m
100 MHz/CDCl3
f1 (ppm)


8.075
7.854
7.836
7.829
7.820
7.807
7.539
7.535
7.531
7.526
7.522
7.509
7.505
7.499
7.478
7.462
7.459
7.260
7.007
4.260
4.242
4.224
4.207
3.828
1.838
1.145
1.127
1.109
CO2Et
Me
OH
O
N
H
2n
400 MHz/CDCl3
1.04
3.09
3.02
1.00
2.08
1.02
3.04
3.07
f1 (ppm)


169.154
163.935
143.884
139.479
133.808
132.765
130.132
128.823
127.781
127.477
127.230
126.796
126.599
126.432
85.683
77.478
77.160
76.842
61.862
25.641
13.957
CO2Et
Me
OH
O
N
H
2n
100 MHz/CDCl3
f1 (ppm)


8.399
7.428
7.260
6.911
6.870
4.447
4.412
4.396
4.364
3.738
1.779
1.410
1.393
1.375
S
CO2Et
O
N
H
Me
OH
2o
400 MHz/CDCl3
1.01
1.01
1.00
1.00
2.00
1.02
3.04
3.01
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
f1 (ppm)


168.816
163.853
147.085
142.648
139.467
131.808
114.825
110.651
85.915
77.477
77.160
76.842
61.980
25.836
14.261
S
CO2Et
O
N
H
Me
OH
2o
100 MHz/CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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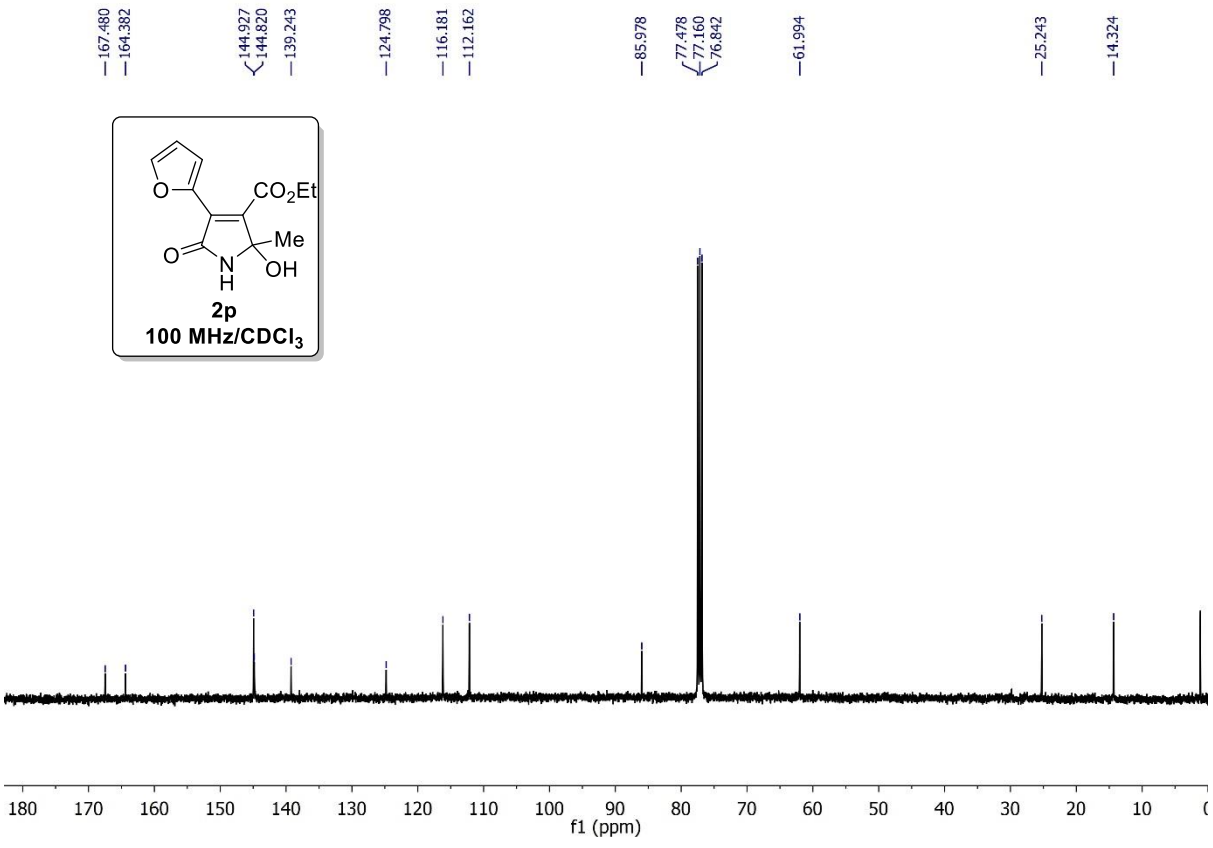
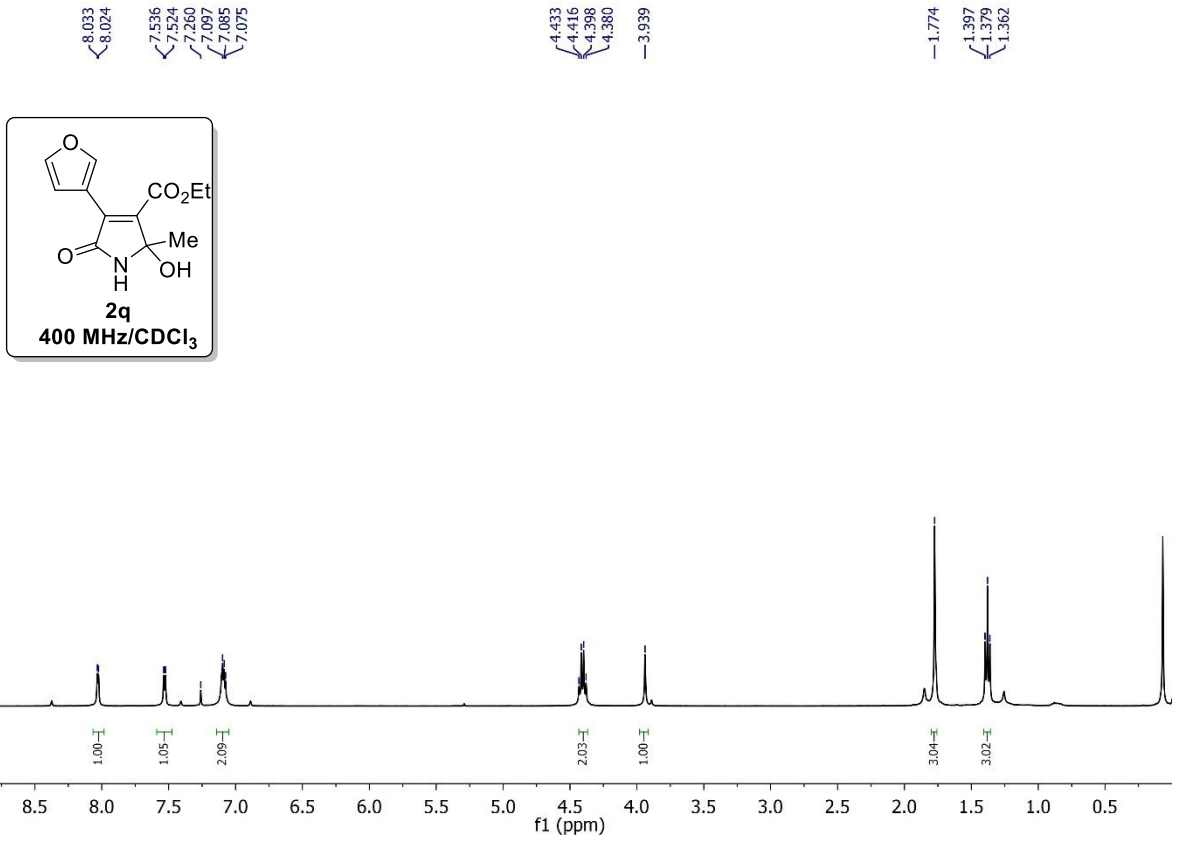
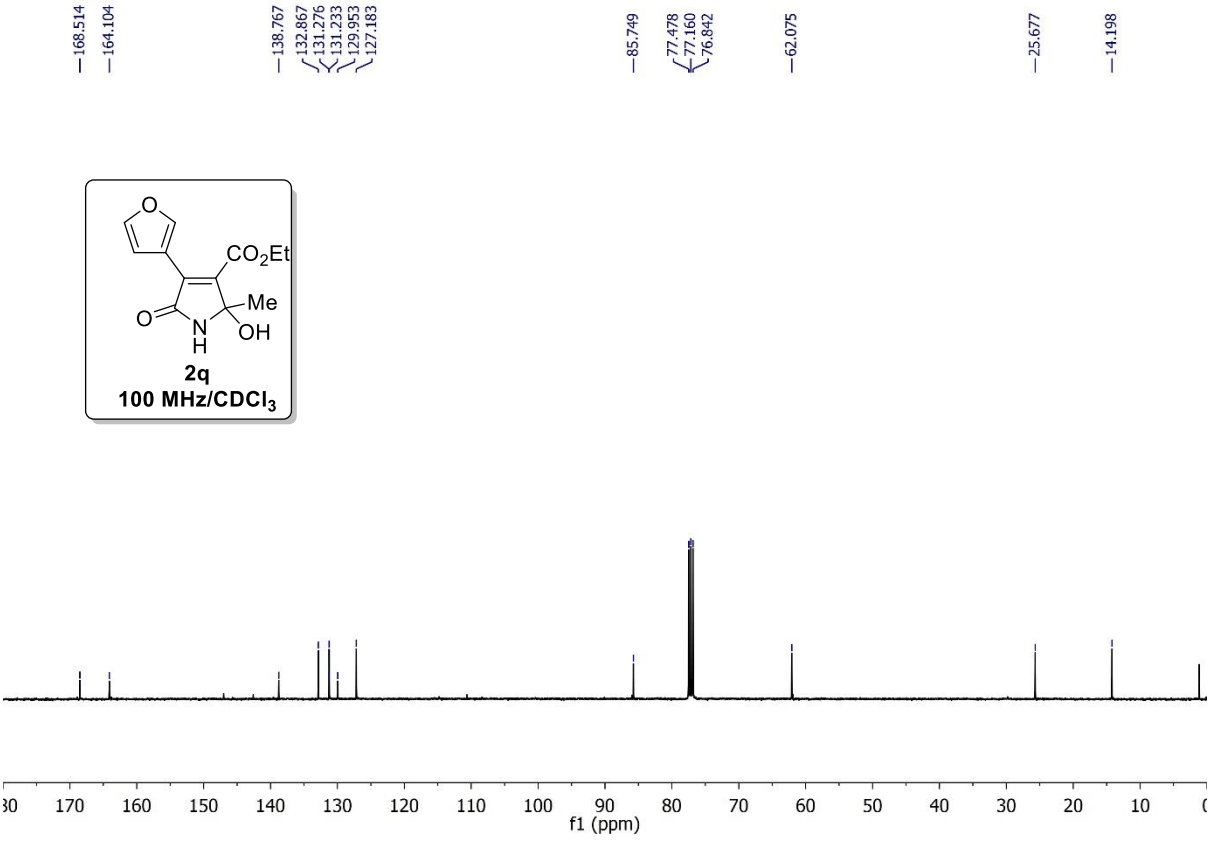
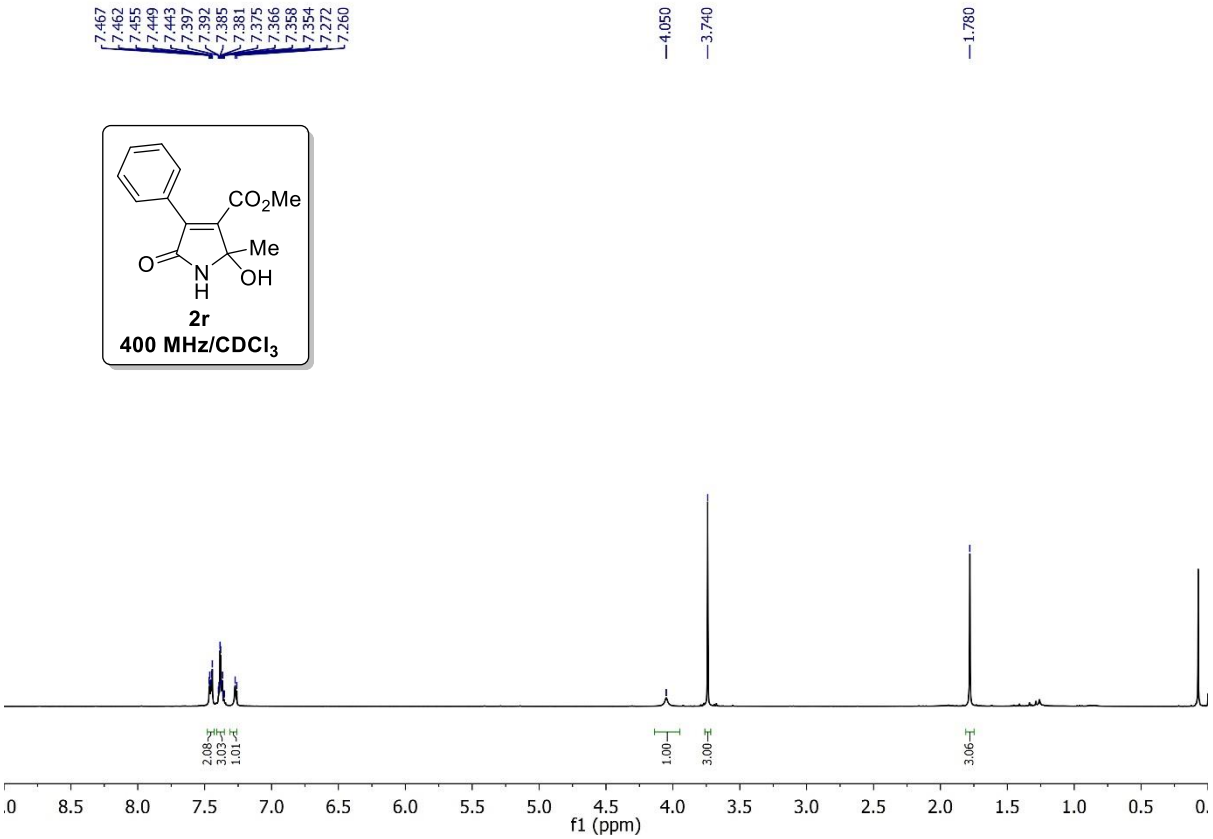
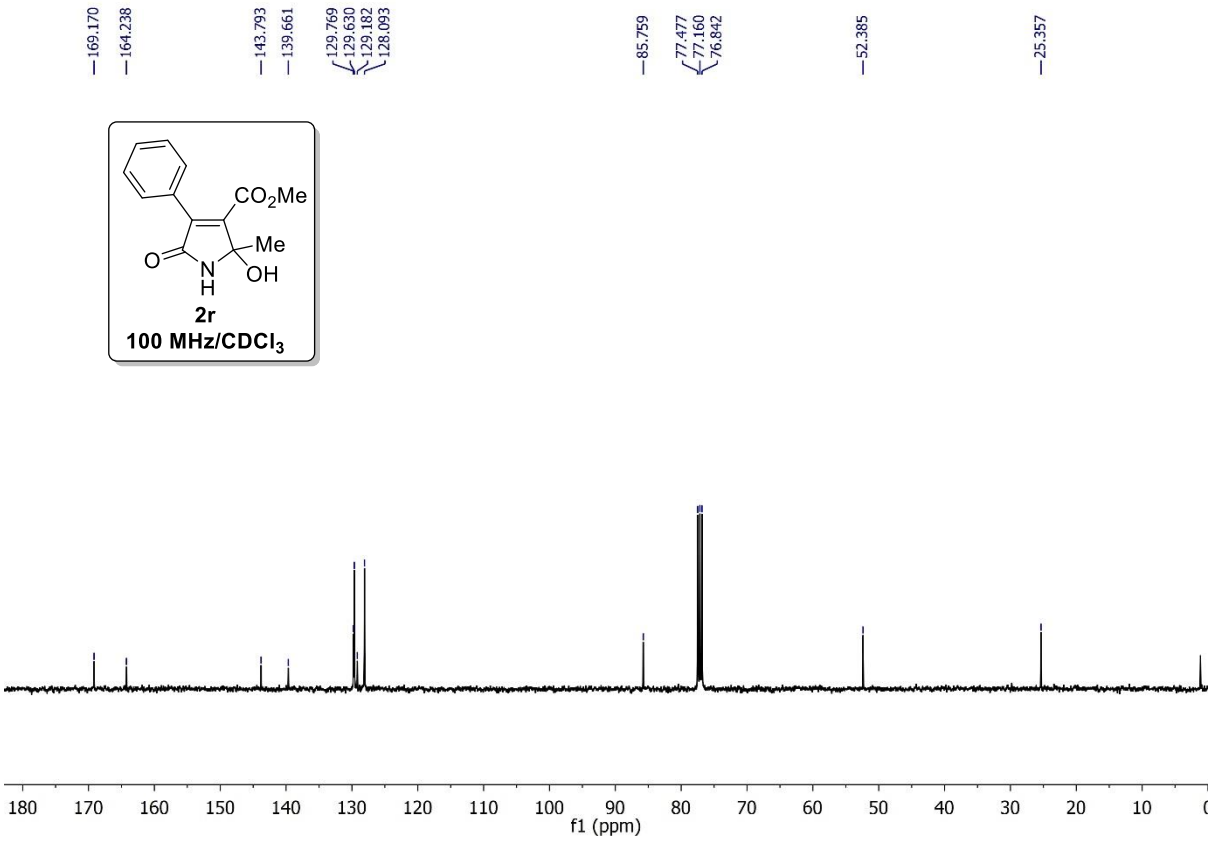
7.366
7.359
7.260
6.870
6.470
4.434
4.418
4.400
4.383
3.869
1.745
1.392
1.374
1.357
CO2Et
Me
OH
O
N
H
2p
400 MHz/CDCl3
1.04
1.08
1.04
1.06
2.00
1.03
3.06
3.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

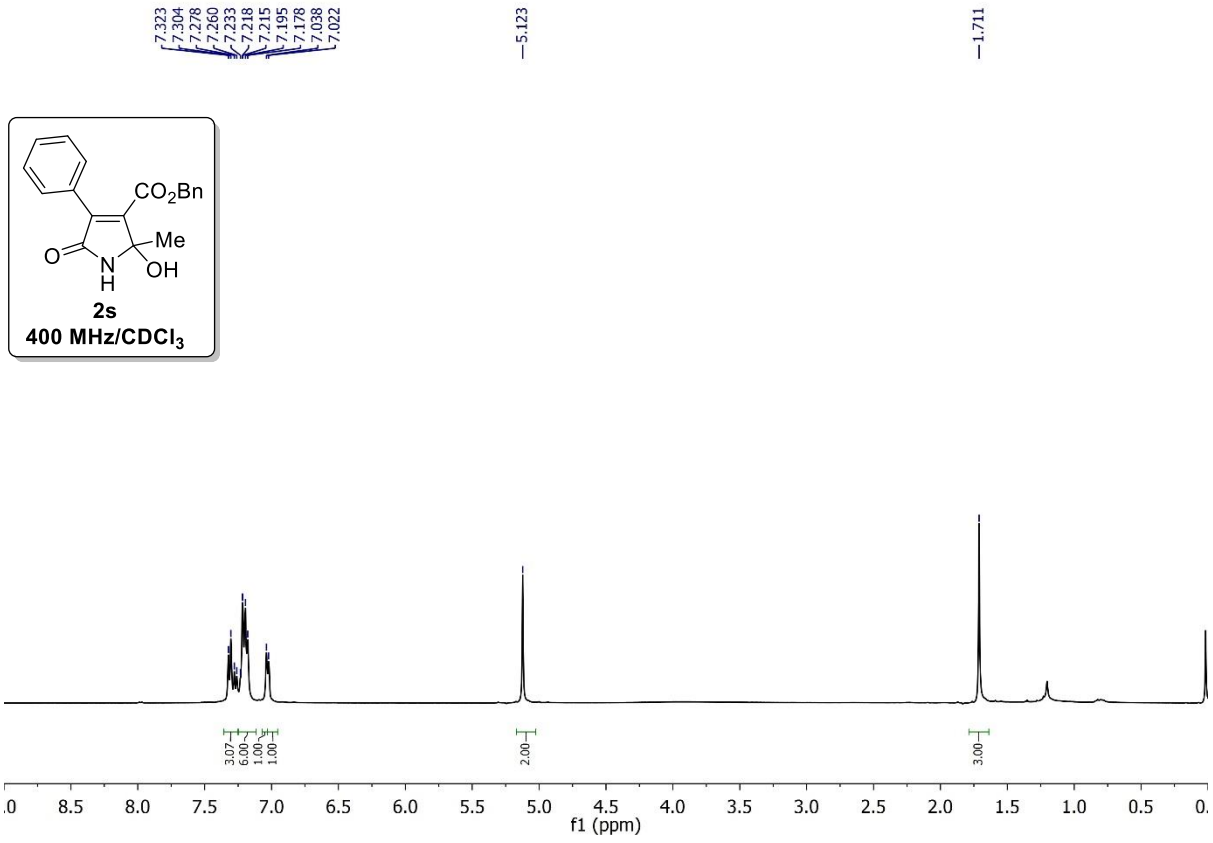

167.480
164.382
144.927
144.820
139.243
124.798
116.181
112.162
85.978
77.478
77.160
76.842
61.994
25.243
14.324
CO2Et
Me
OH
O
N
H
2p
100 MHz/CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

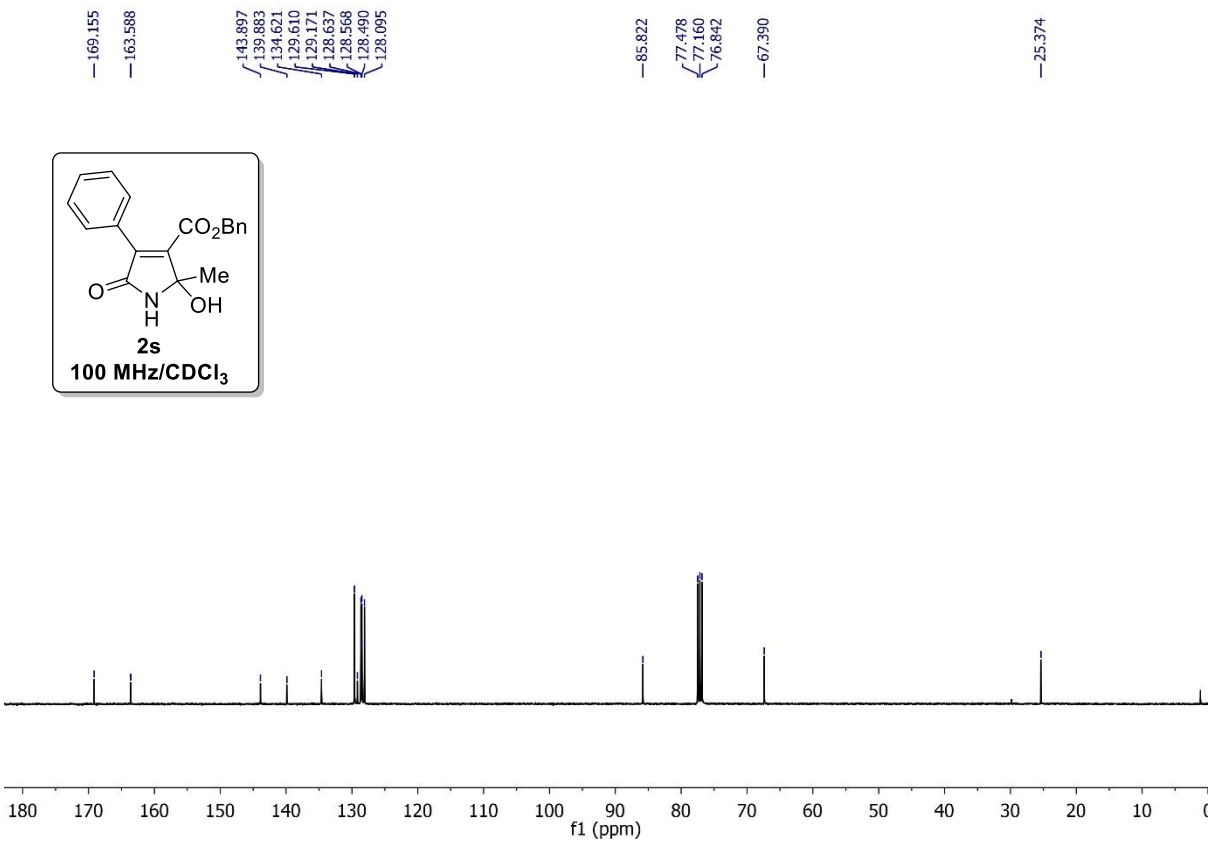

8.033
8.024
7.536
7.524
7.260
7.097
7.085
7.075
4.433
4.416
4.398
4.380
3.939
1.774
1.397
1.379
1.362
CO2Et
Me
OH
O
N
H
2q
400 MHz/CDCl3
1.00
1.05
2.09
2.03
1.00
3.04
3.02
f1 (ppm)


168.514
164.104
138.767
132.867
131.276
131.233
129.953
127.183
85.749
77.478
77.160
76.842
62.075
25.677
14.198
CO2Et
Me
OH
O
N
H
2q
100 MHz/CDCl3
f1 (ppm)


7.467
7.462
7.455
7.449
7.443
7.397
7.392
7.385
7.381
7.375
7.366
7.358
7.354
7.272
7.260
4.050
3.740
1.780
CO2Me
Me
O
N
H
OH
2r
400 MHz/CDCl3
2.08
3.03
1.01
1.00
3.00
3.06
f1 (ppm)


169.170
164.238
143.793
139.661
129.769
129.630
129.182
128.093
85.759
77.477
77.160
76.842
52.385
25.357
CO2Me
Me
O
N
H
OH
2r
100 MHz/CDCl3
f1 (ppm)


7.323
7.304
7.278
7.260
7.233
7.218
7.215
7.195
7.178
7.038
7.022
5.123
1.711
CO2Bn
Me
O
N
H
OH
2s
400 MHz/CDCl3
3.07
6.00
1.00
1.00
2.00
3.00
f1 (ppm)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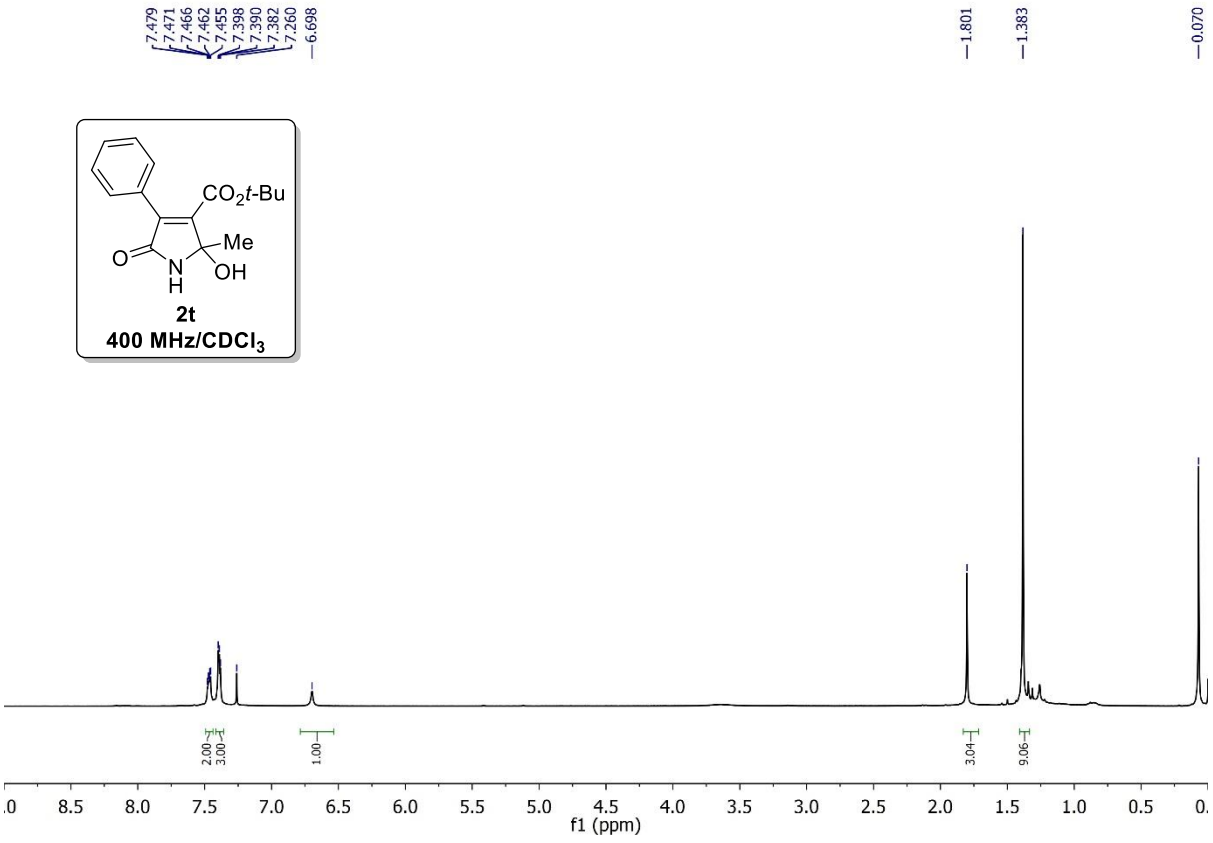
163.588
143.897
139.883
134.621
129.610
129.171
128.637
128.568
128.490
128.095
85.822
77.478
77.160
76.842
67.390
25.374
CO2Bn
Me
O
N
H
OH
2s
100 MHz/CDCl3
f1 (ppm)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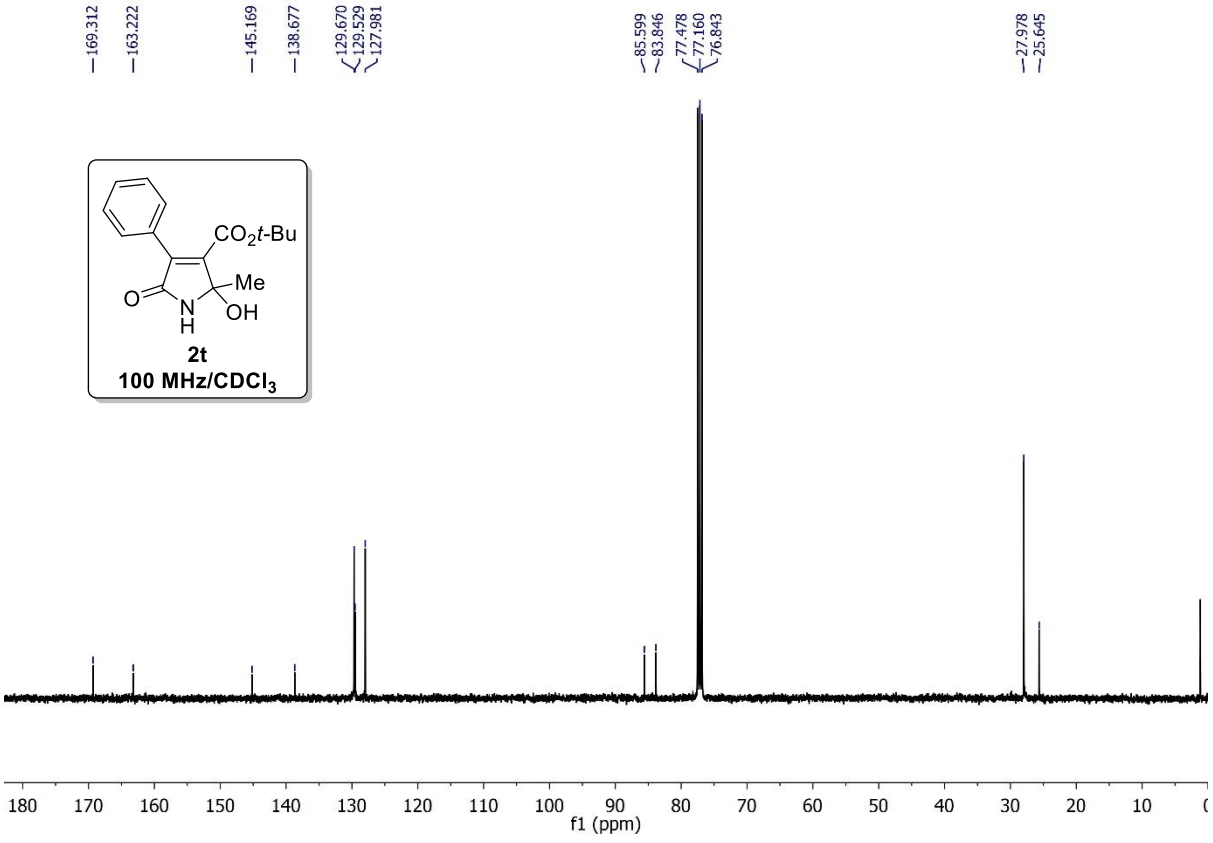
7.471
7.466
7.462
7.455
7.398
7.390
7.382
7.260
6.698
1.801
1.383
0.070
CO2t-Bu
Me
OH
O
N
H
2t
400 MHz/CDCl3
2.00
3.00
1.00
3.04
9.06
f1 (ppm)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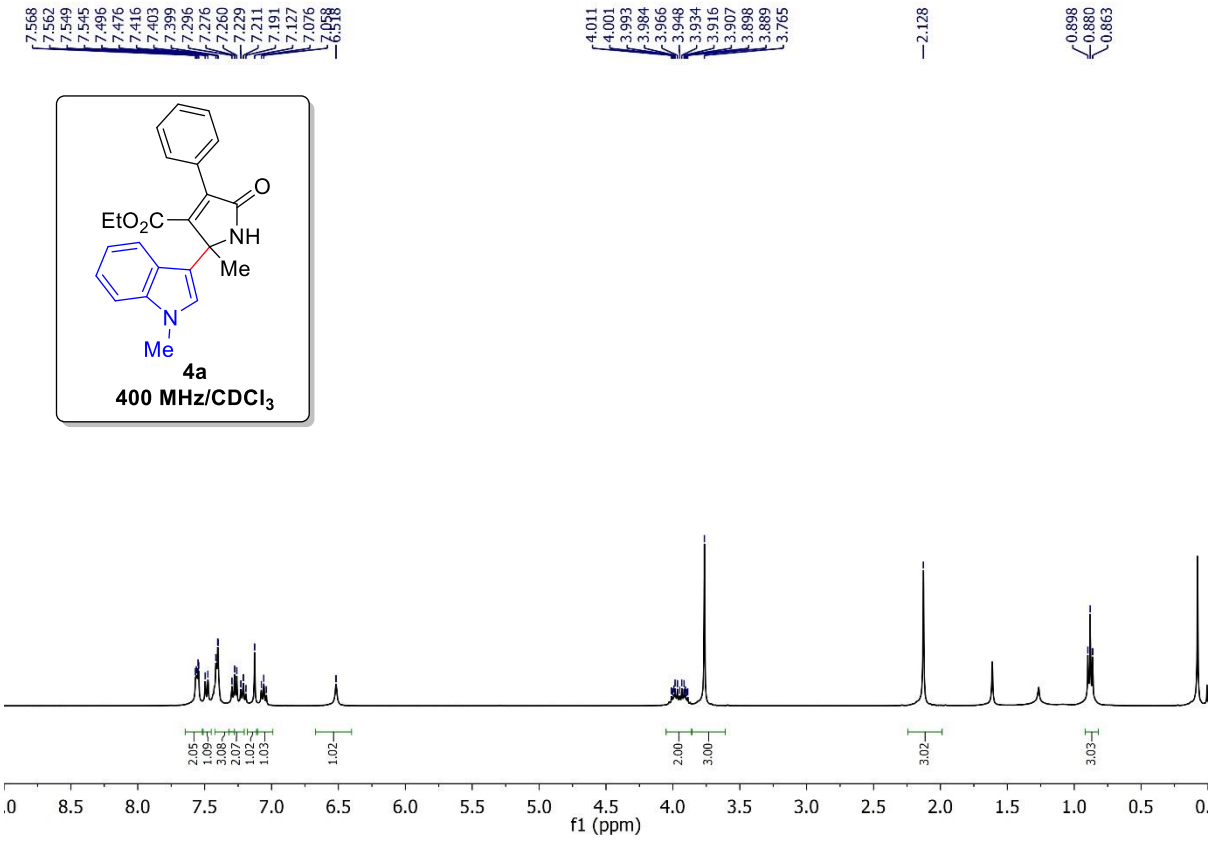
163.222
145.169
138.677
129.670
129.529
127.981
85.599
83.846
77.478
77.160
76.843
27.978
25.645
CO2t-Bu
Me
OH
O
N
H
2t
100 MHz/CDCl3
f1 (ppm)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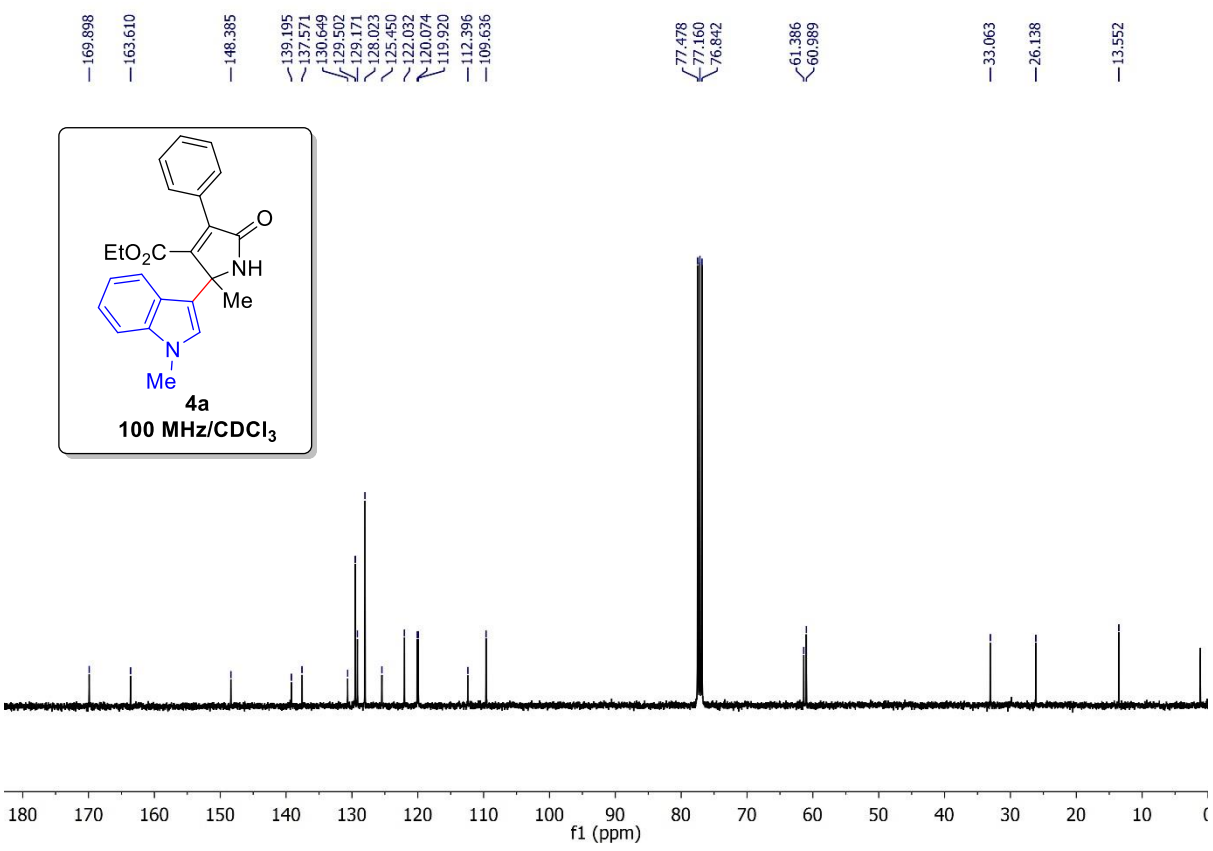
7.562
7.549
7.545
7.496
7.476
7.416
7.403
7.399
7.296
7.276
7.260
7.229
7.211
7.191
7.127
7.076
7.058
6.518
4.011
4.001
3.993
3.984
3.966
3.948
3.934
3.916
3.907
3.898
3.889
3.765
2.128
0.898
0.880
0.863
EtO2C
O
NH
Me
N
Me
4a
400 MHz/CDCl3
2.05
1.09
3.08
2.07
1.02
1.03
1.02
2.00
3.00
3.02
3.03
f1 (ppm)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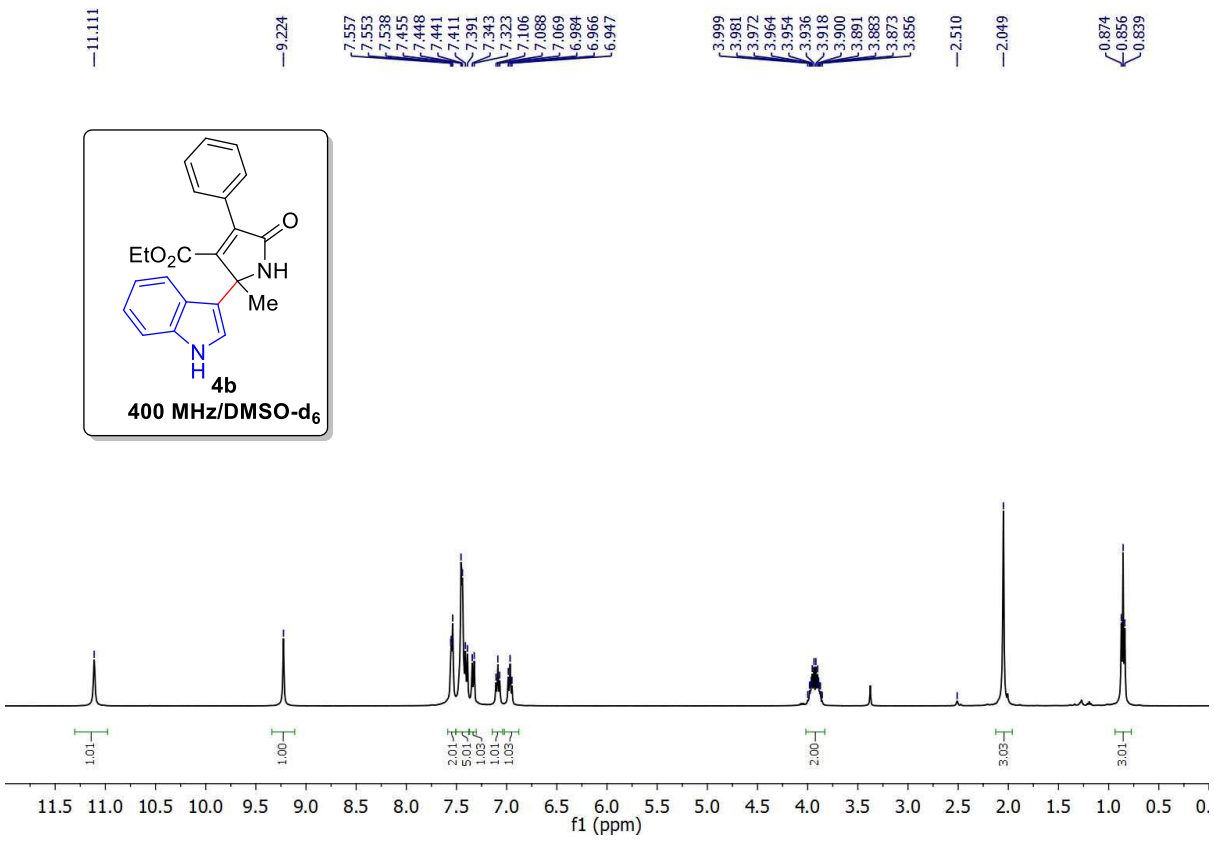
130.649
129.502
129.171
128.023
125.450
122.032
120.074
119.920
112.396
109.636
77.478
77.160
76.842
61.386
60.989
33.063
26.138
13.552
EtO2C
O
NH
Me
N
Me
4a
100 MHz/CDCl3
f1 (ppm)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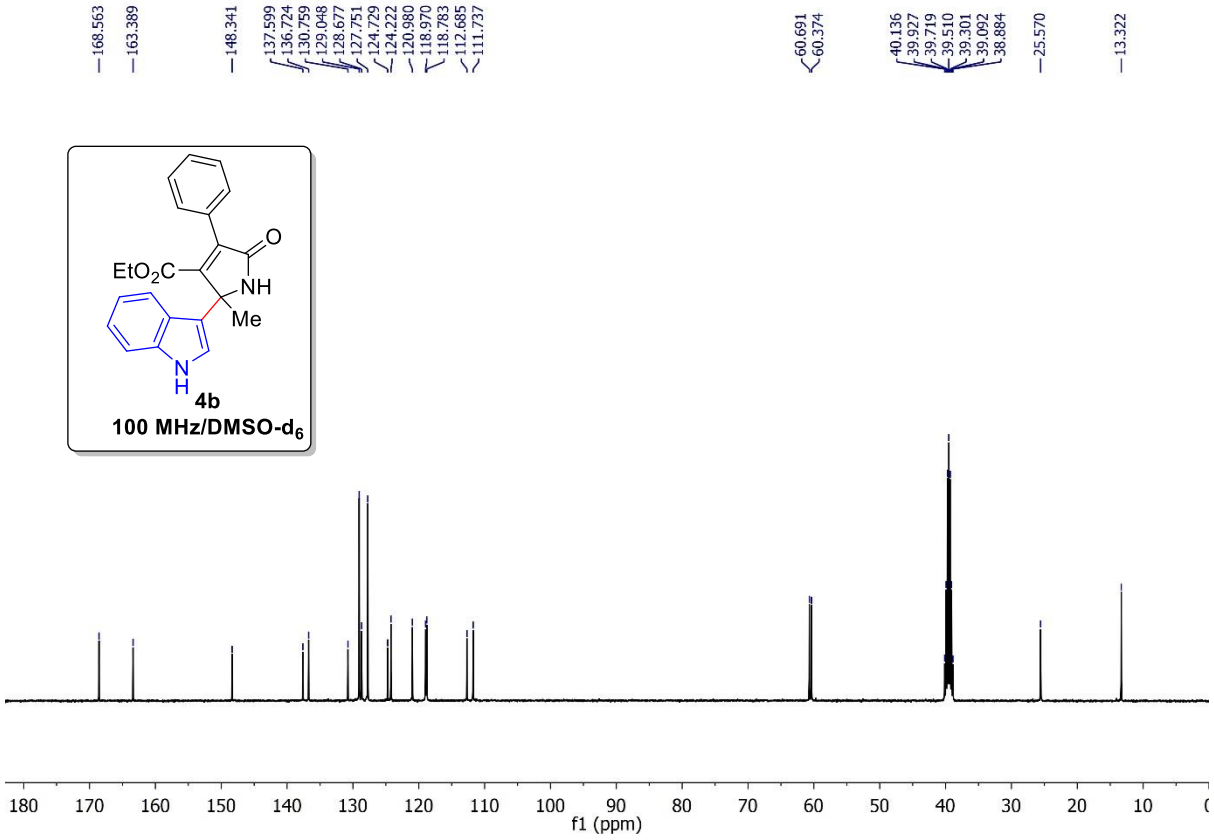
7.557
7.553
7.538
7.455
7.448
7.441
7.411
7.391
7.343
7.323
7.106
7.088
7.069
6.984
6.966
6.947
3.999
3.981
3.972
3.964
3.954
3.936
3.918
3.900
3.891
3.883
3.873
3.856
2.510
2.049
0.874
0.856
0.839
EtO2C
O
NH
Me
N
H
4b
400 MHz/DMSO-d6
1.01
1.00
2.01
5.01
1.03
1.01
1.03
2.00
3.03
3.01
11.5 11.0 10.5 10.0 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
f1 (ppm)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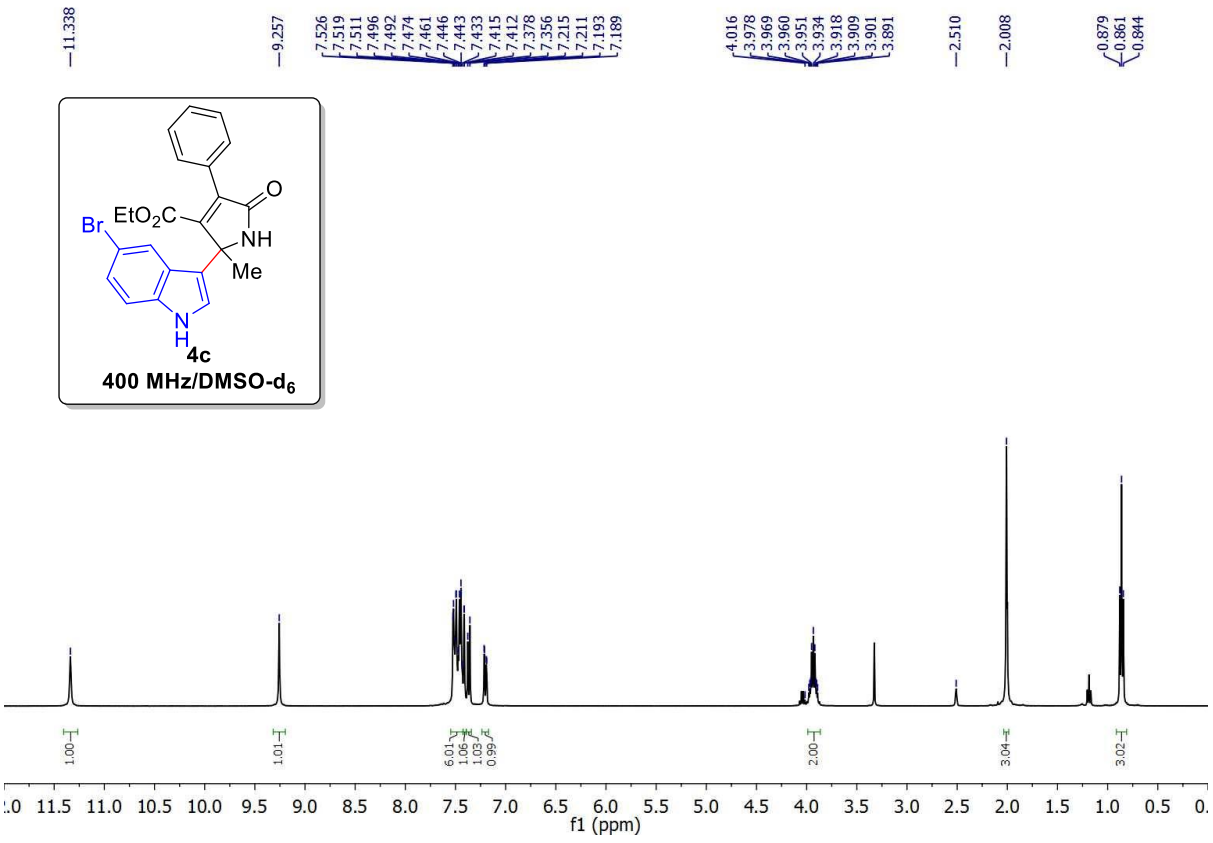
136.724
130.759
129.048
128.677
127.751
124.729
124.222
120.980
118.970
118.783
112.685
111.737
60.691
60.374
40.136
39.927
39.719
39.510
39.301
39.092
38.884
25.570
13.322
EtO2C
O
NH
Me
N
H
4b
100 MHz/DMSO-d6
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


11.338
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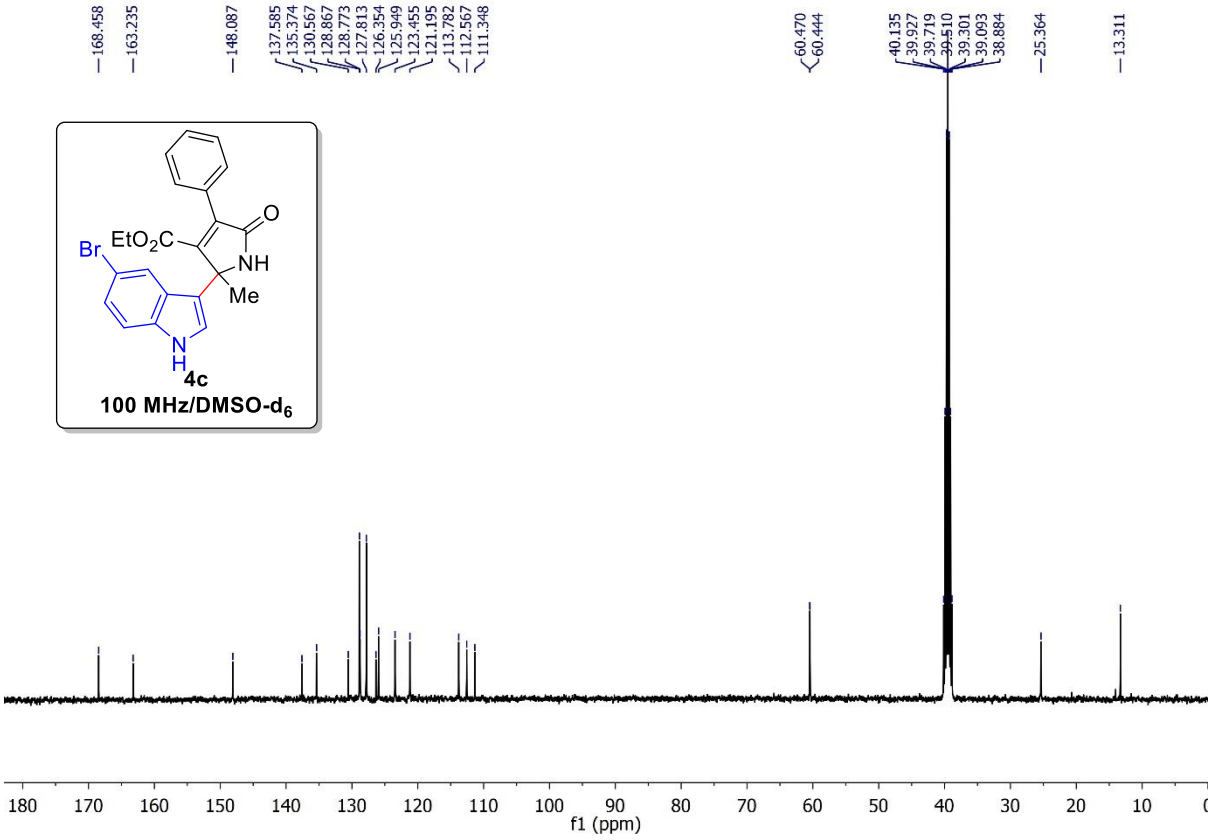
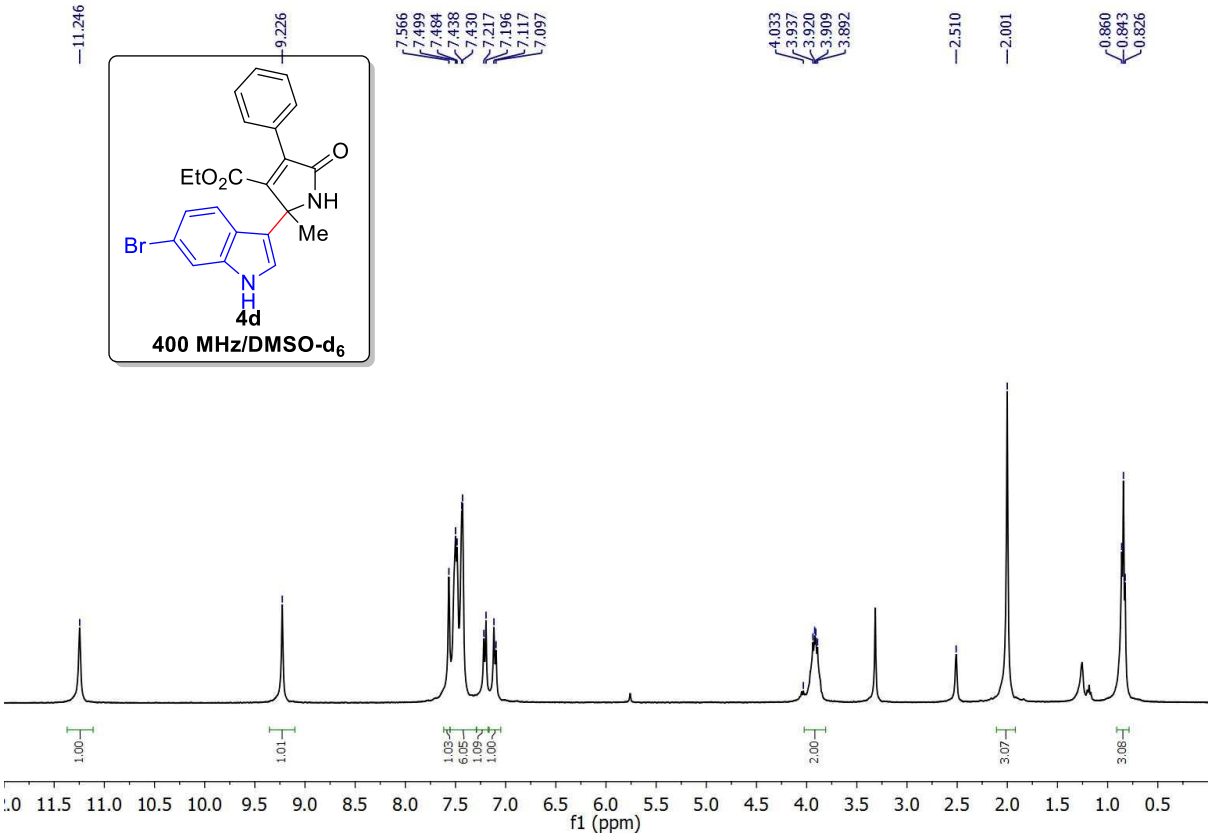
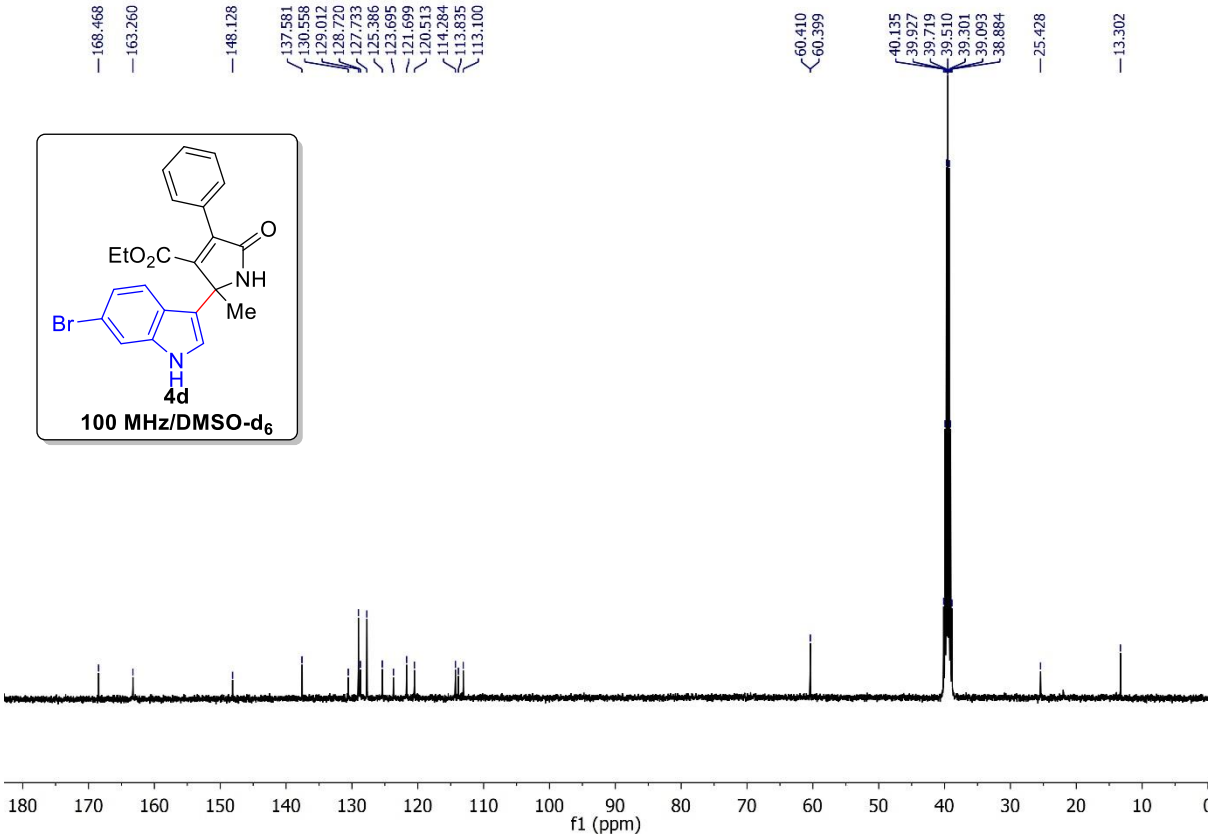
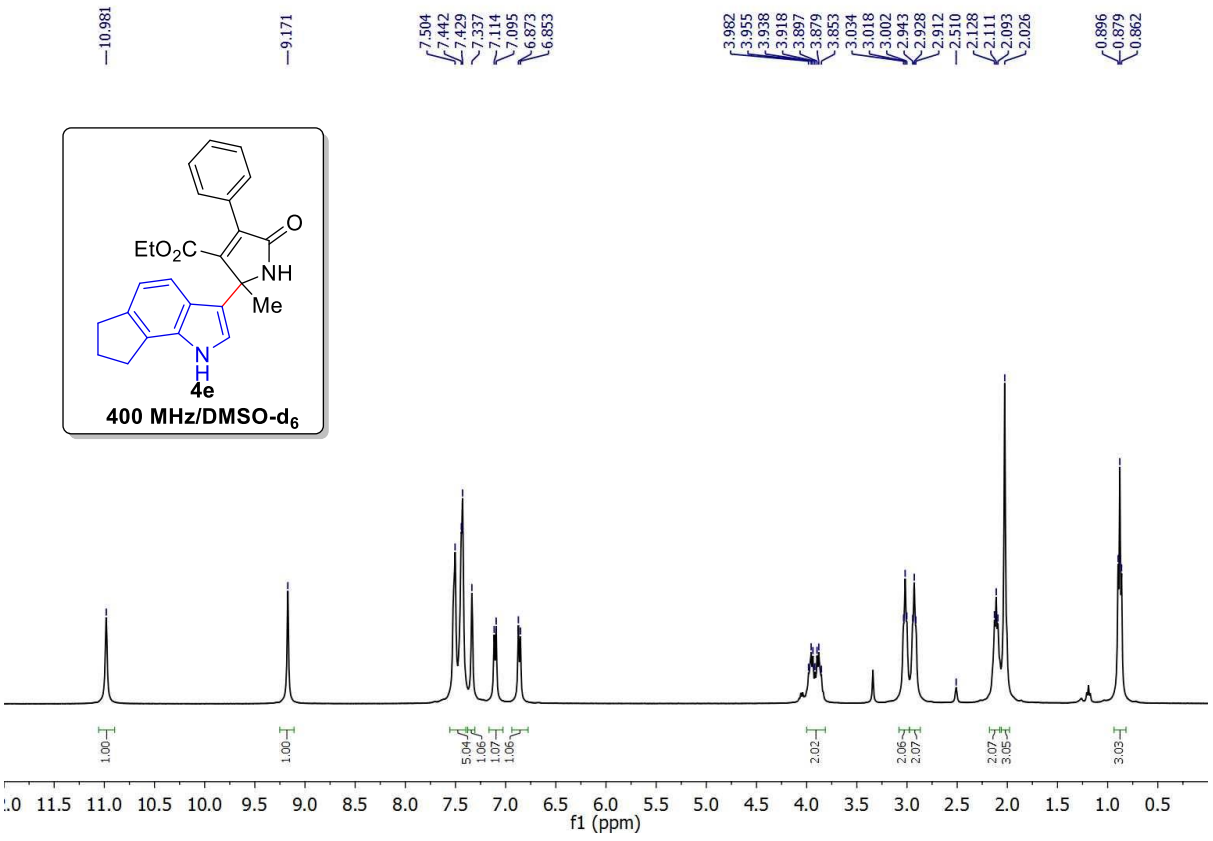
7.519
7.511
7.496
7.492
7.474
7.461
7.446
7.443
7.433
7.415
7.412
7.378
7.356
7.215
7.211
7.193
7.189
4.016
3.978
3.969
3.960
3.951
3.934
3.918
3.909
3.901
3.891
2.510
2.008
0.879
0.861
0.844
Br
EtO2C
O
NH
Me
N
H
4c
400 MHz/DMSO-d6
1.00
1.01
6.01
1.06
1.03
0.99
2.00
3.04
3.02
f1 (ppm)


168.458
163.235
148.087
137.585
135.374
130.567
128.867
128.773
127.813
126.354
125.949
123.455
121.195
113.782
112.567
111.348
60.470
60.444
40.135
39.927
39.719
39.510
39.301
39.093
38.884
25.364
13.311
Br
EtO2C
O
NH
Me
N
H
4c
100 MHz/DMSO-d6
f1 (ppm)


11.246
9.226
7.566
7.499
7.484
7.438
7.430
7.217
7.196
7.117
7.097
4.033
3.937
3.920
3.909
3.892
2.510
2.001
0.860
0.843
0.826
EtO2C
O
NH
Me
Br
N
H
4d
400 MHz/DMSO-d6
1.00
1.01
1.03
6.05
1.09
1.00
2.00
3.07
3.08
f1 (ppm)


168.468
163.260
148.128
137.581
130.558
129.012
128.720
127.733
125.386
123.695
121.699
120.513
114.284
113.835
113.100
60.410
60.399
40.135
39.927
39.719
39.510
39.301
39.093
38.884
25.428
13.302
EtO2C
O
NH
Me
Br
N
H
4d
100 MHz/DMSO-d6
f1 (ppm)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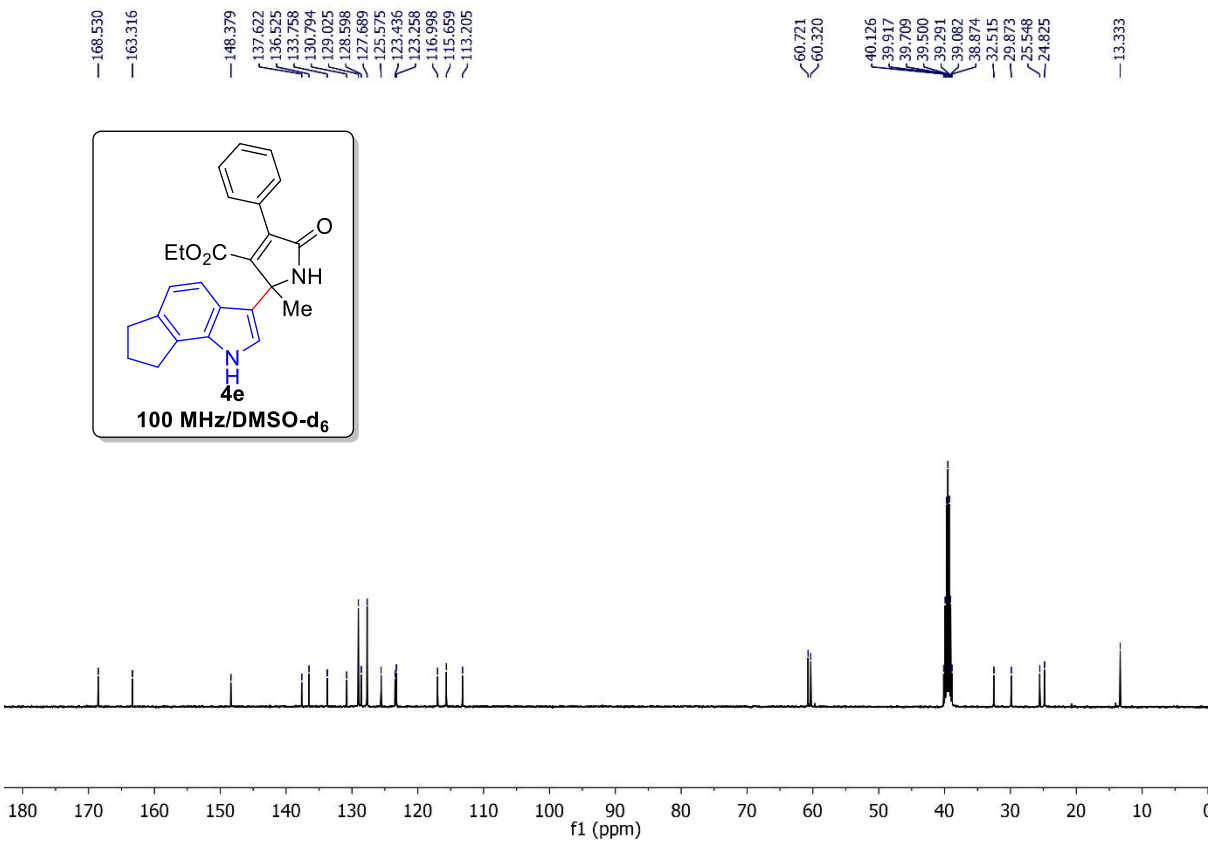
9.171
7.504
7.442
7.429
7.337
7.114
7.095
6.873
6.853
3.982
3.955
3.938
3.918
3.897
3.879
3.853
3.034
3.018
3.002
2.943
2.928
2.912
2.510
2.128
2.111
2.093
2.026
0.896
0.879
0.862
EtO2C
O
NH
Me
N
H
4e
400 MHz/DMSO-d6
1.00
1.00
5.04
1.06
1.07
1.06
2.02
2.06
2.07
2.07
3.05
3.03
f1 (ppm)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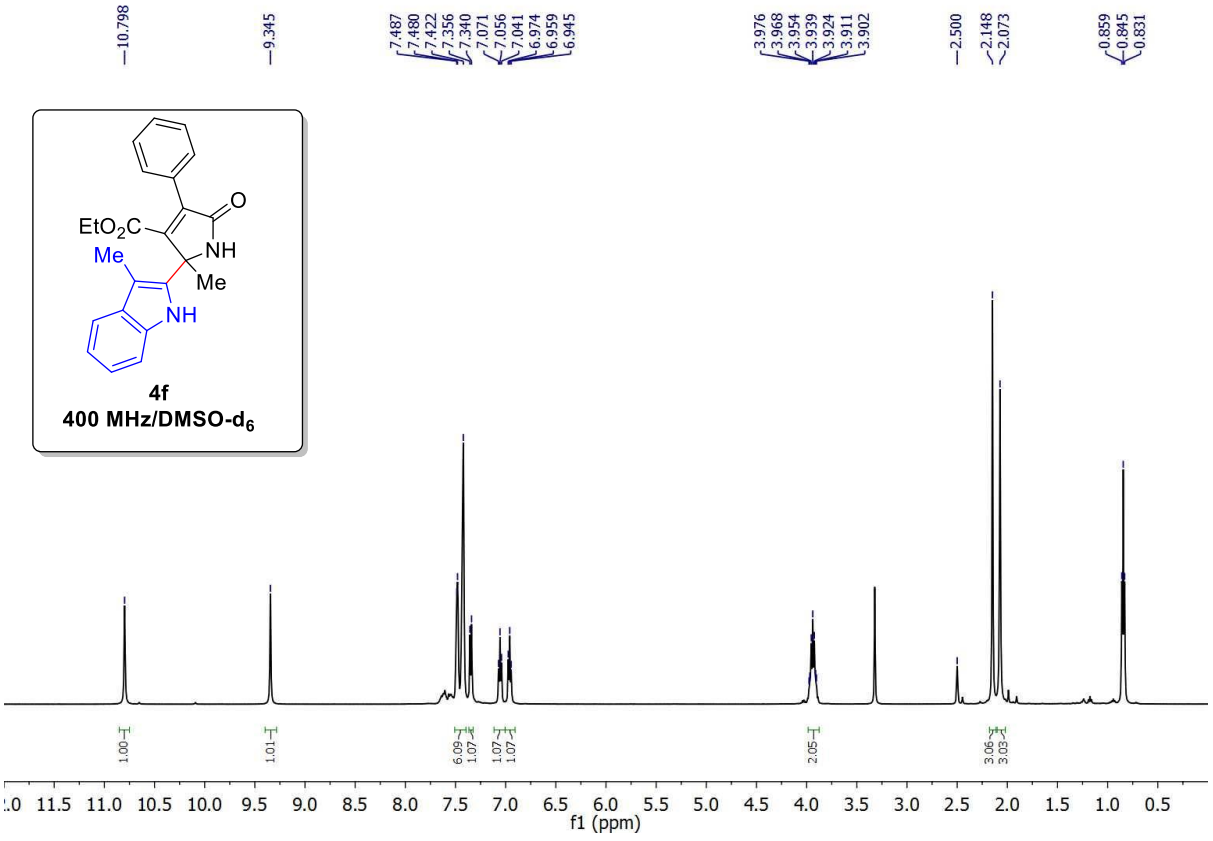
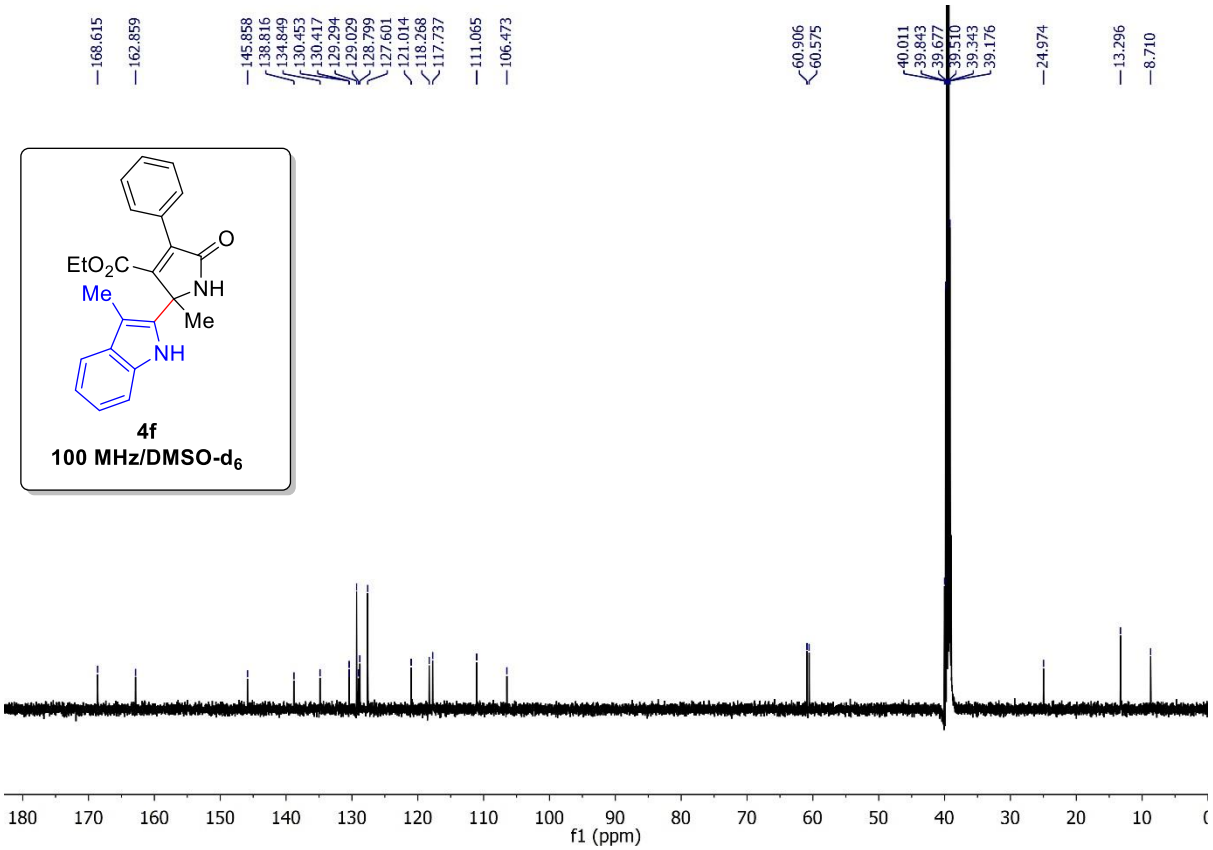
148.379
137.622
136.525
133.758
130.794
129.025
128.598
127.689
125.575
123.436
123.258
116.998
115.659
113.205
60.721
60.320
40.126
39.917
39.709
39.500
39.291
39.082
38.874
32.515
29.873
25.548
24.825
13.333
EtO2C
O
NH
Me
N
H
4e
100 MHz/DMSO-d6
f1 (ppm)


10.798
9.345
7.487
7.480
7.422
7.356
7.340
7.071
7.056
7.041
6.974
6.959
6.945
3.976
3.968
3.954
3.939
3.924
3.911
3.902
2.500
2.148
2.073
0.859
0.845
0.831
EtO2C
O
NH
Me
Me
NH
4f
400 MHz/DMSO-d6
1.00
1.01
6.09
1.07
1.07
1.07
2.05
3.06
3.03
f1 (ppm)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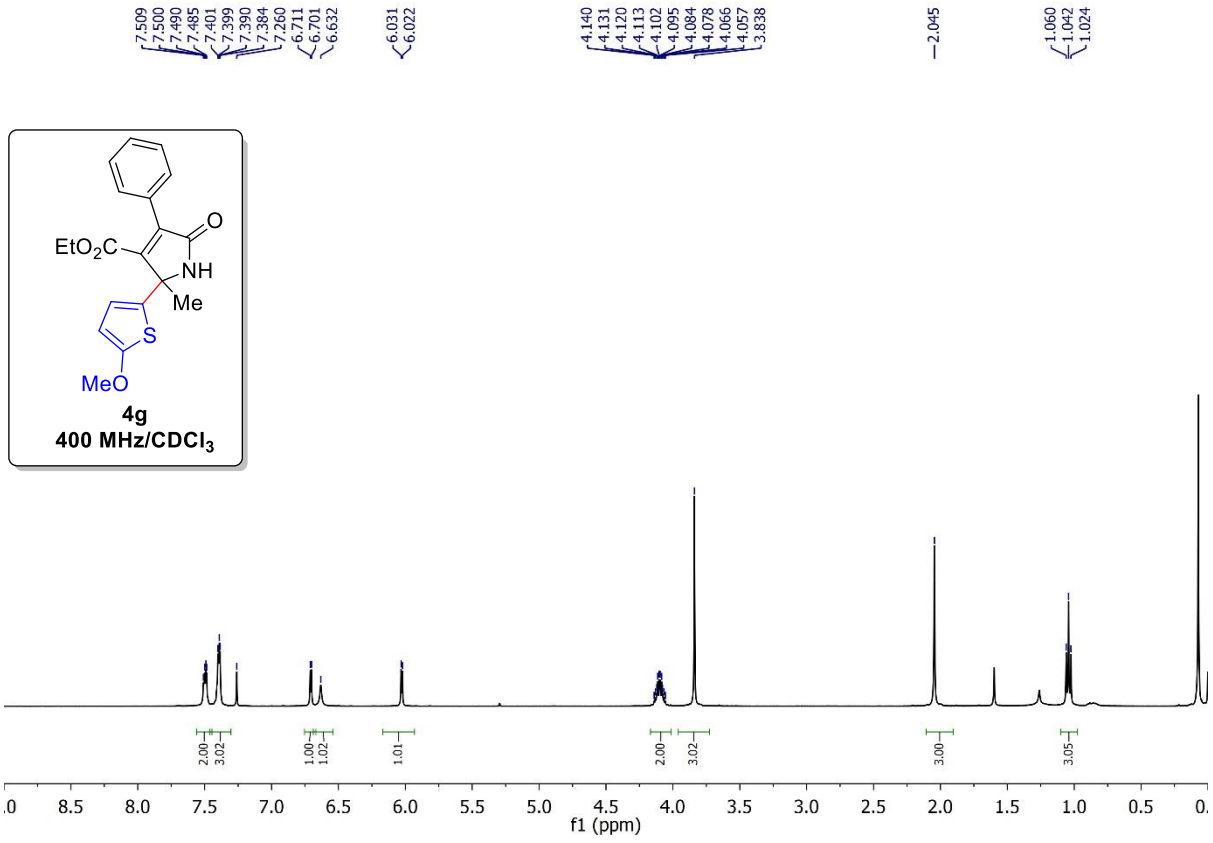
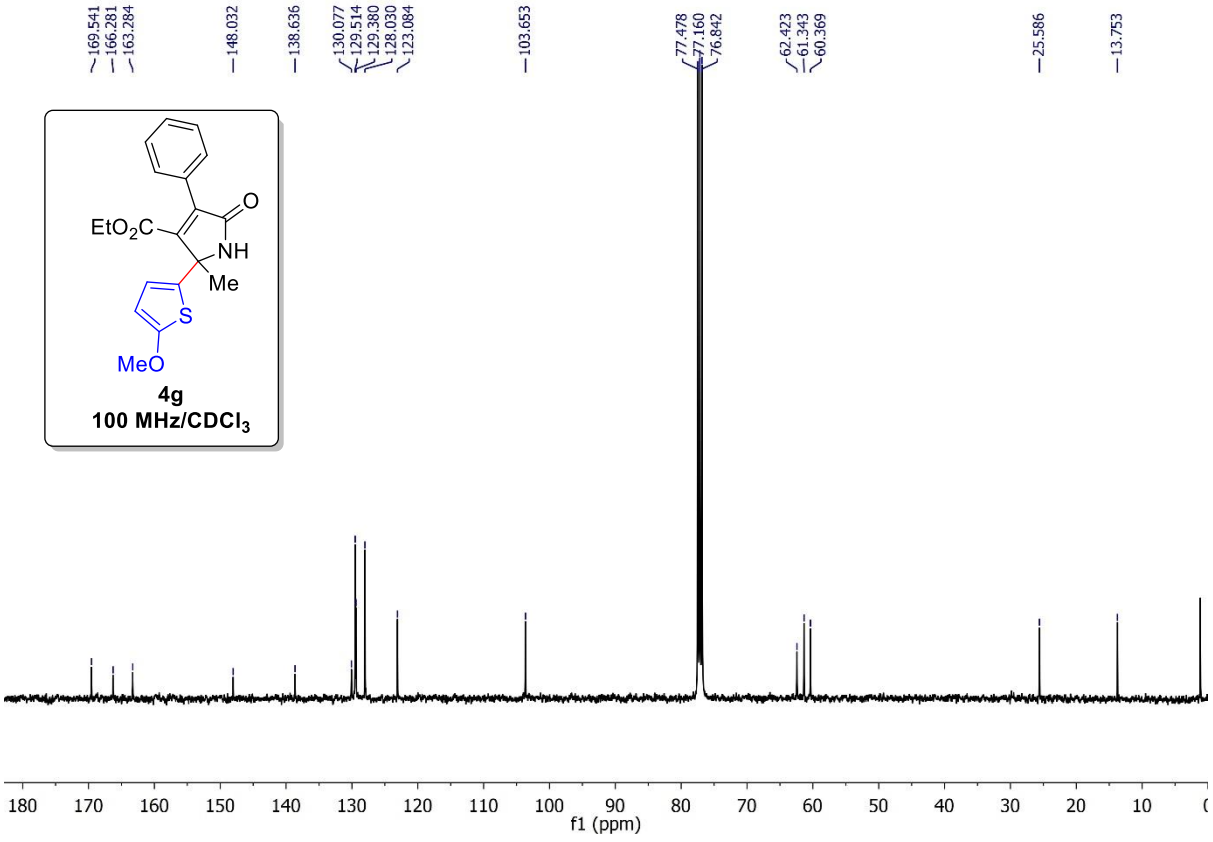
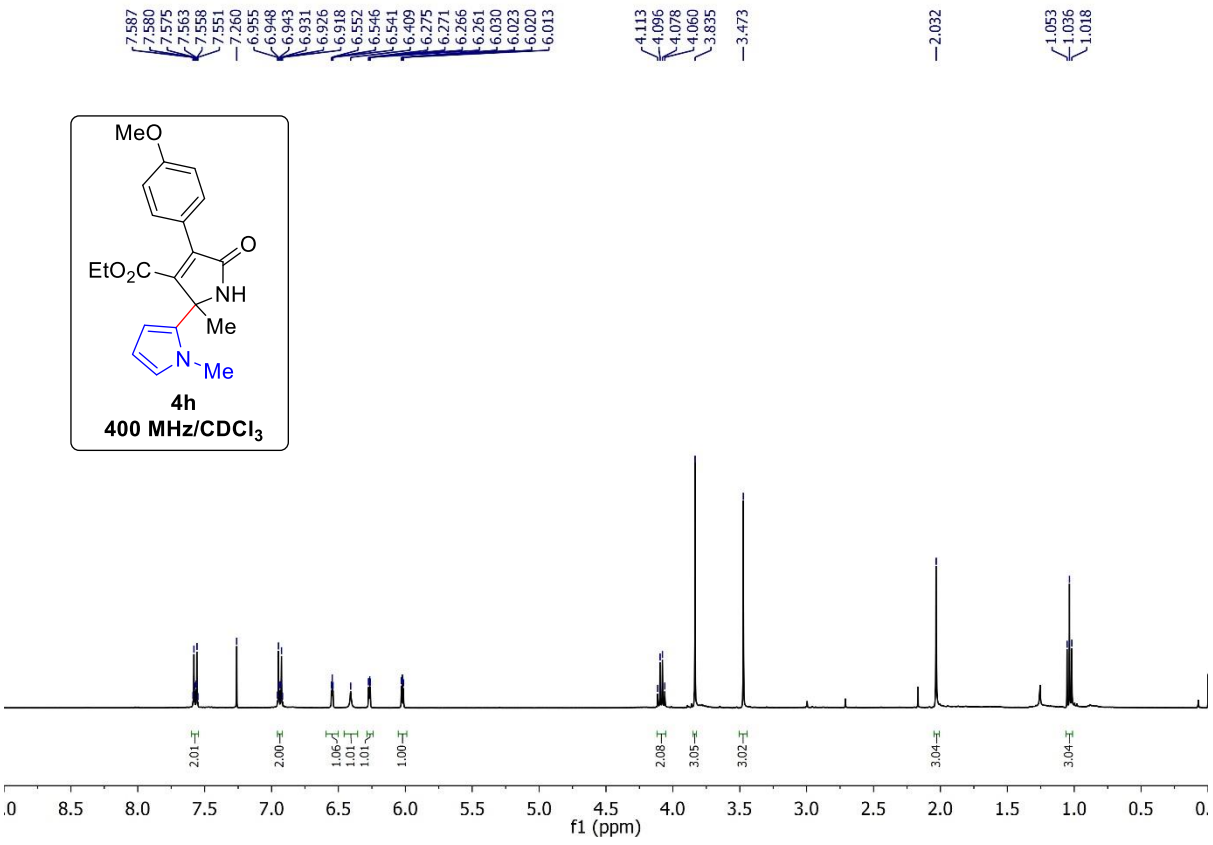
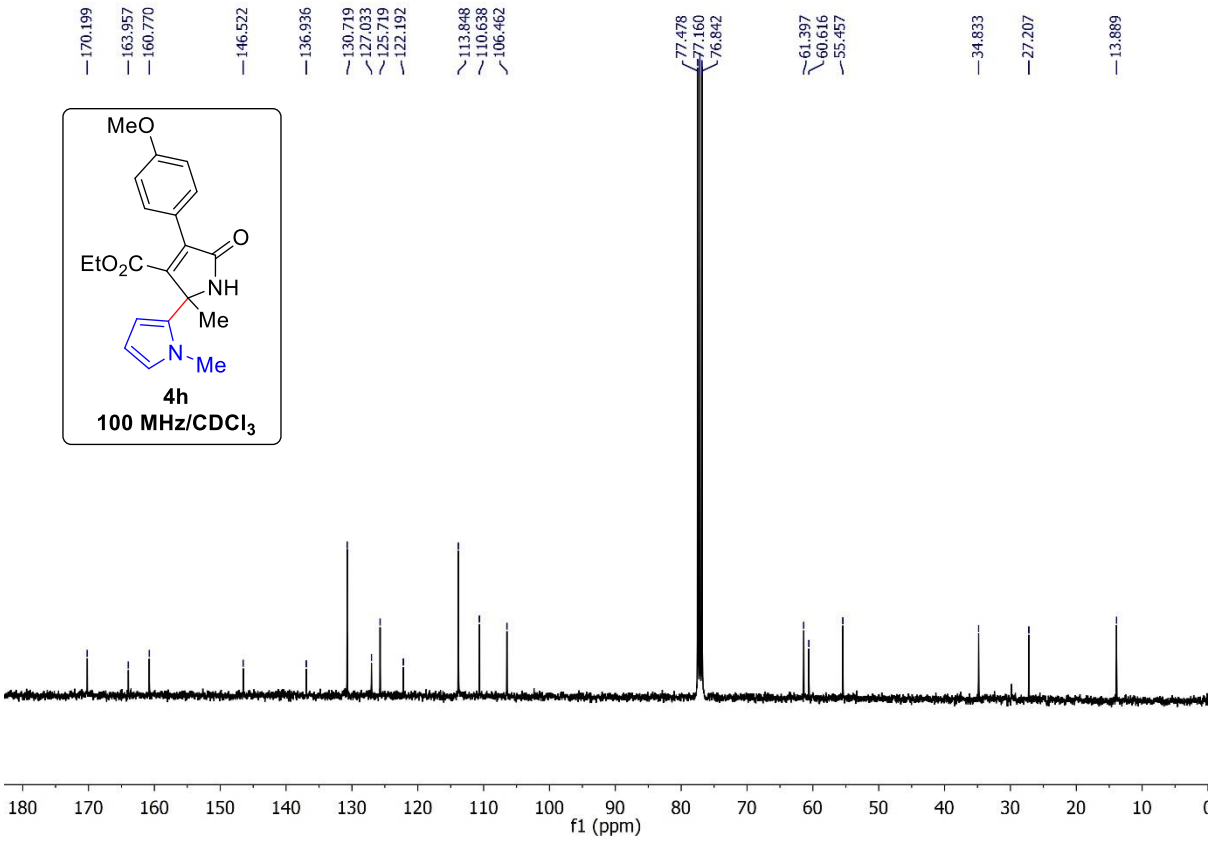
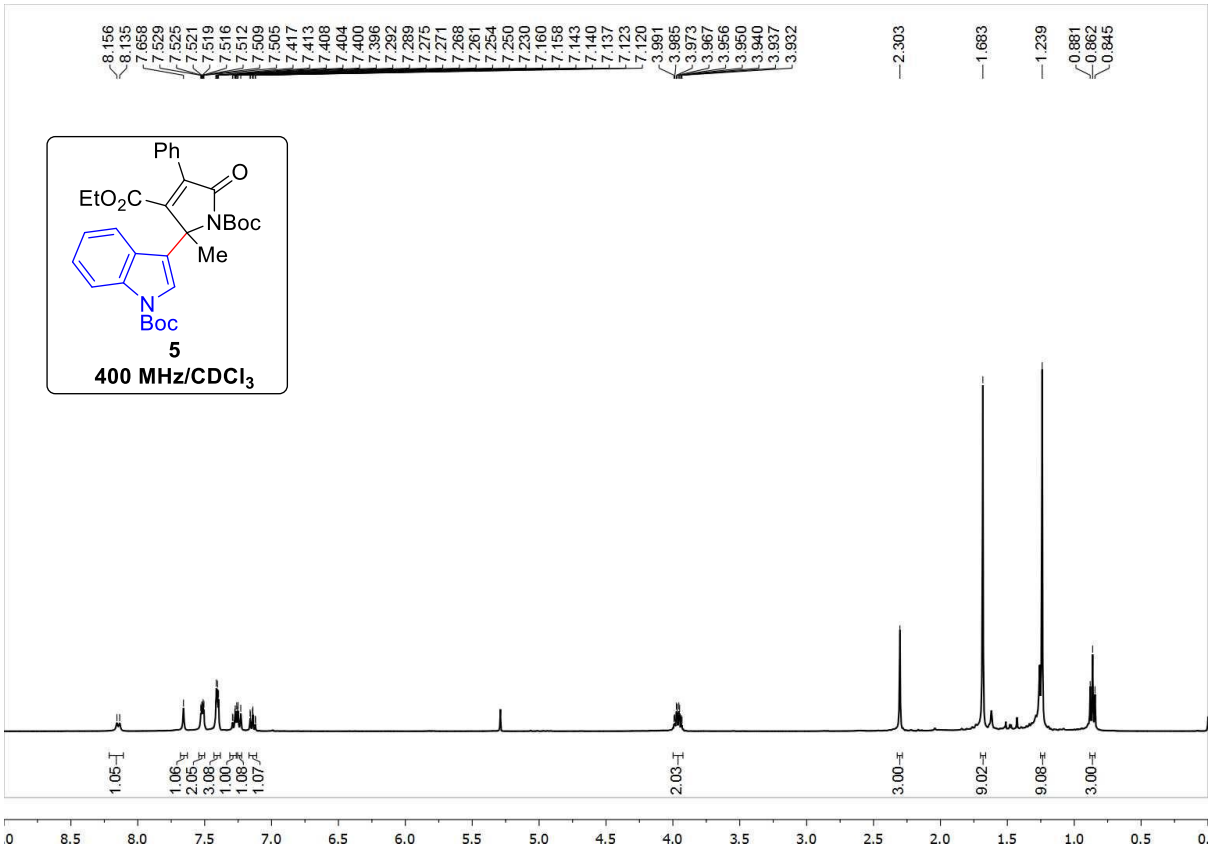
162.859
145.858
138.816
134.849
130.453
130.417
129.294
129.029
128.799
127.601
121.014
118.268
117.737
111.065
106.473
60.906
60.575
40.011
39.843
39.677
39.510
39.343
39.176
24.974
13.296
8.710
EtO2C
O
NH
Me
Me
NH
4f
100 MHz/DMSO-d6
f1 (ppm)

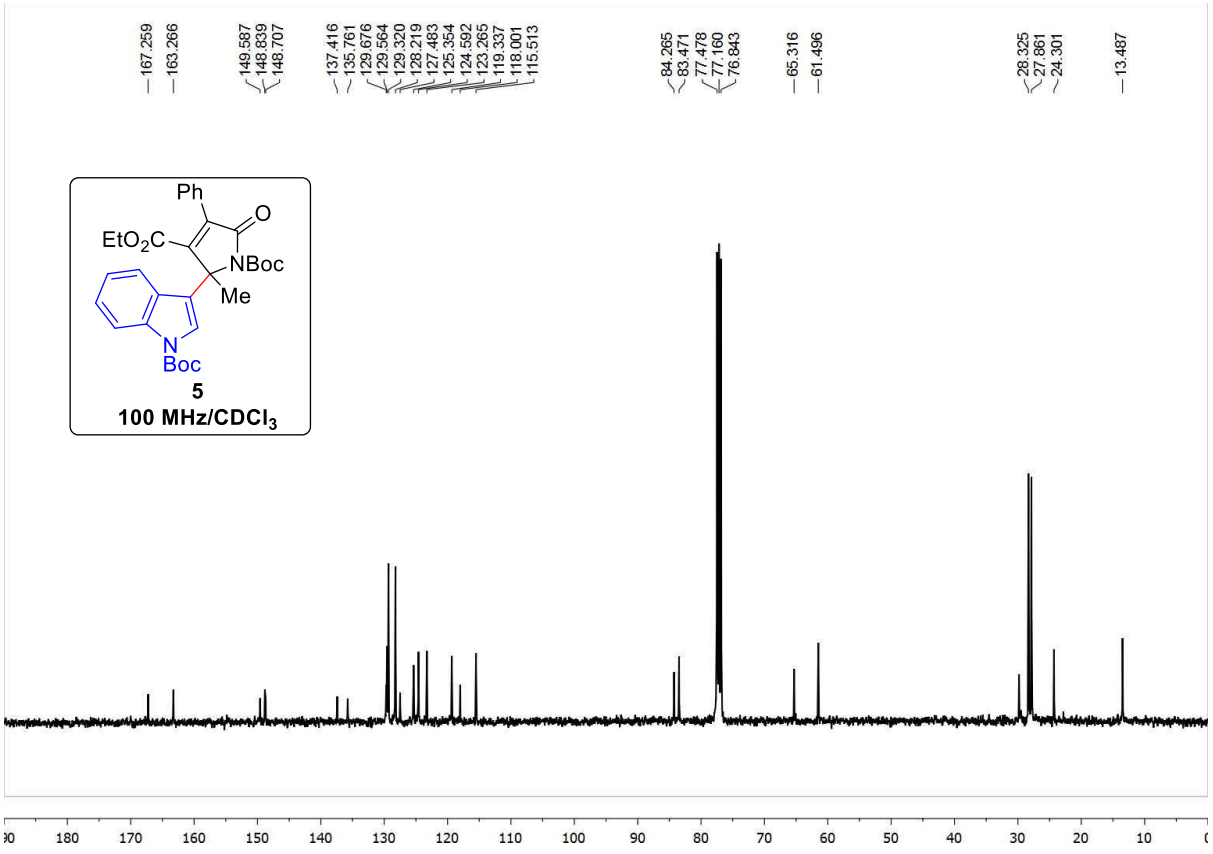

7.509
7.500
7.490
7.485
7.401
7.399
7.390
7.384
7.260
6.711
6.701
6.632
6.031
6.022
4.140
4.131
4.120
4.113
4.102
4.095
4.084
4.078
4.066
4.057
3.838
2.045
1.060
1.042
1.024
EtO2C
O
NH
Me
S
MeO
4g
400 MHz/CDCl3
2.00
3.02
1.00
1.02
1.01
2.00
3.02
3.00
3.05
f1 (ppm)

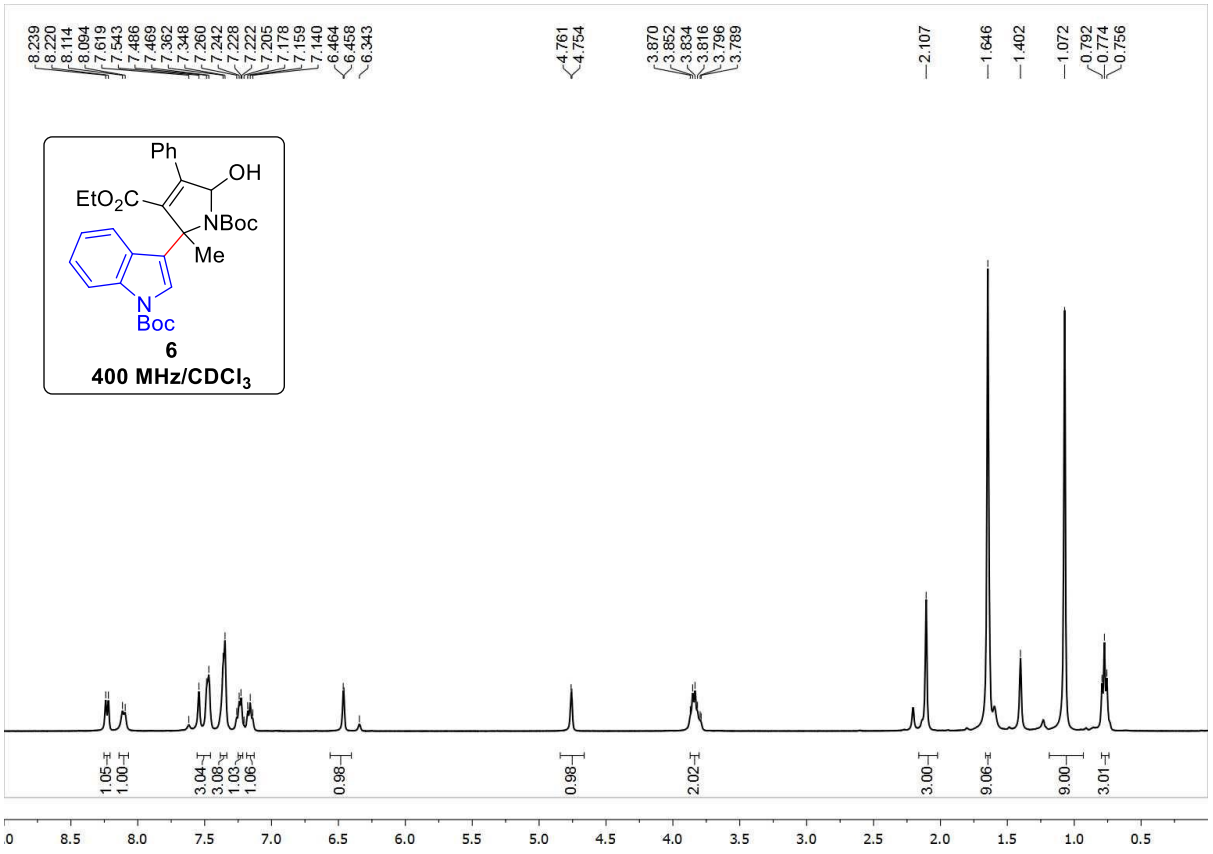

169.541
166.281
163.284
148.032
138.636
130.077
129.514
129.380
128.030
123.084
103.653
77.478
77.160
76.842
62.423
61.343
60.369
25.586
13.753
EtO2C
O
NH
Me
S
MeO
4g
100 MHz/CDCl3
f1 (ppm)

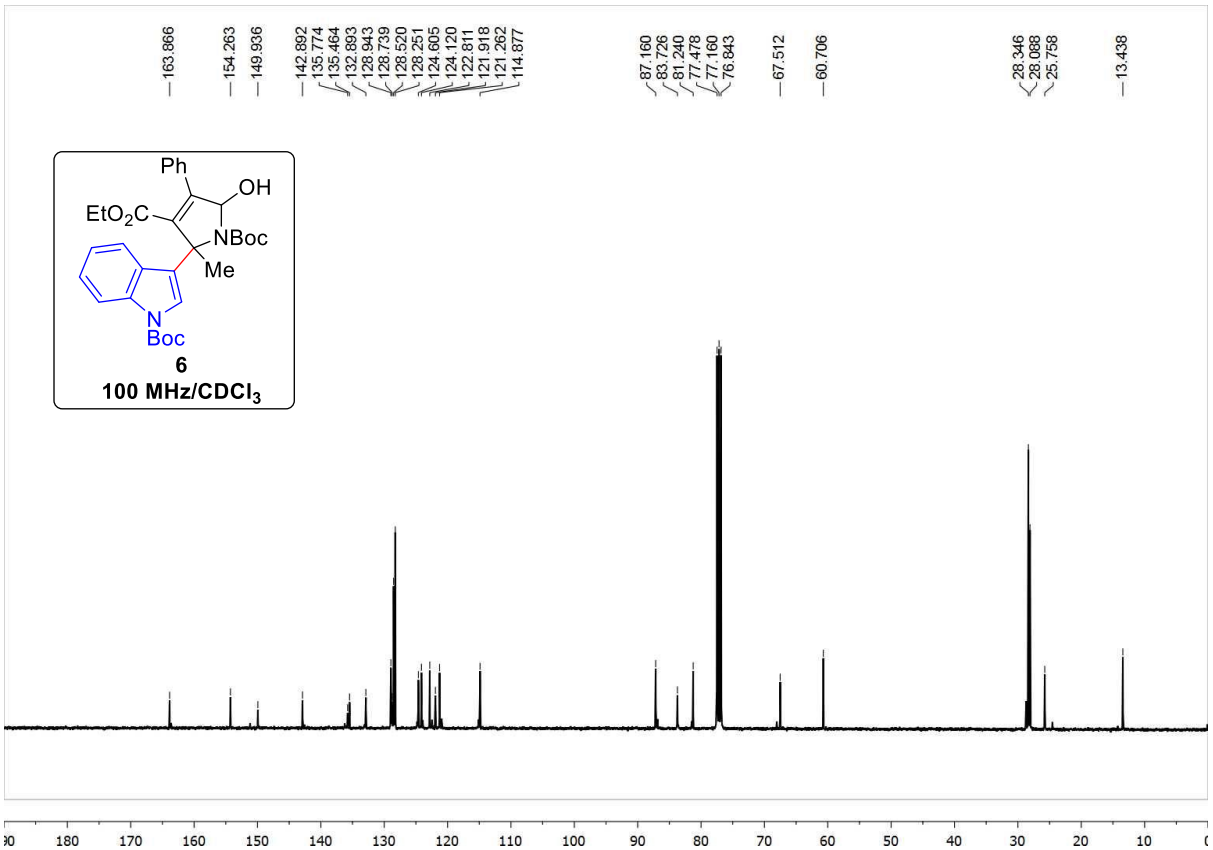

7.587
7.580
7.575
7.563
7.558
7.551
7.260
6.955
6.948
6.943
6.931
6.926
6.918
6.552
6.546
6.541
6.409
6.275
6.271
6.266
6.261
6.030
6.023
6.020
6.013
4.113
4.096
4.078
4.060
3.835
3.473
2.032
1.053
1.036
1.018
MeO
EtO2C
O
NH
Me
N
Me
4h
400 MHz/CDCl3
2.01
2.00
1.06
1.01
1.01
1.00
2.08
3.05
3.02
3.04
3.04
f1 (ppm)

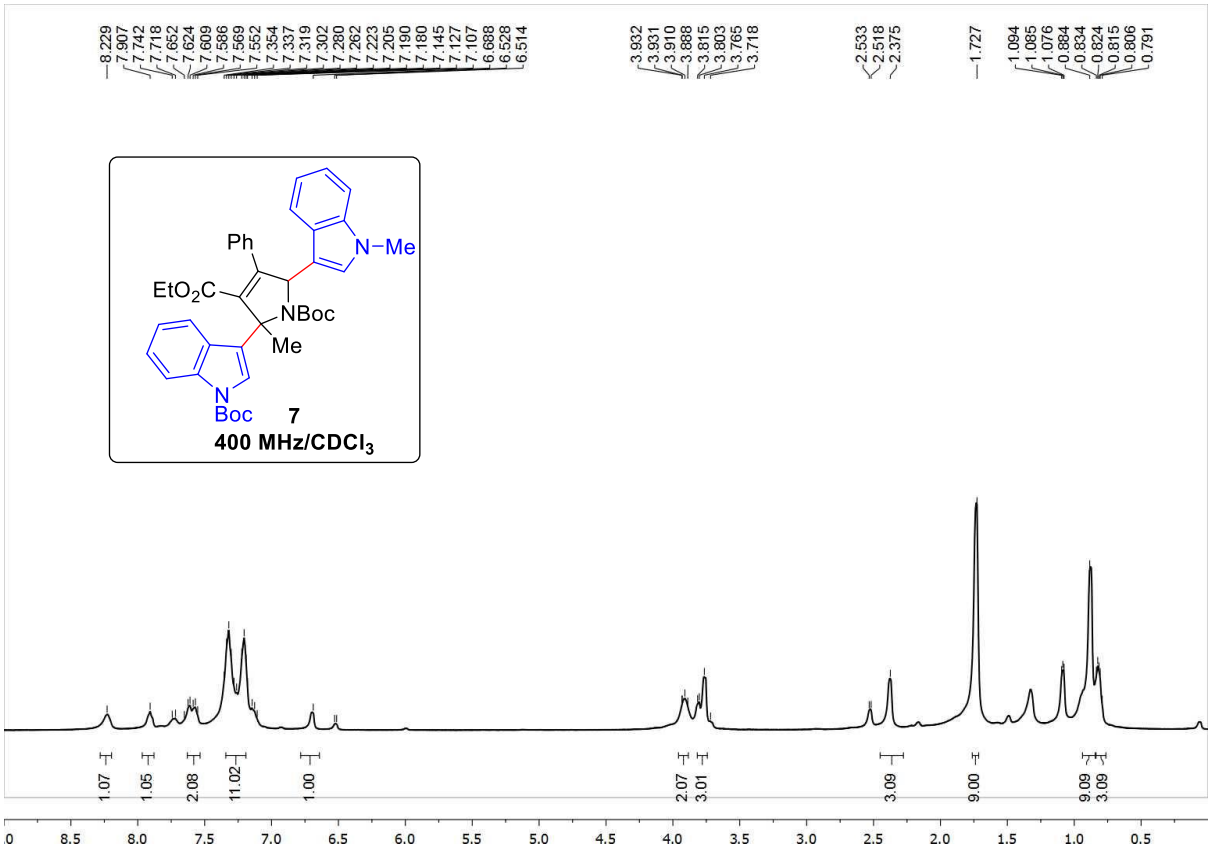

170.199
163.957
160.770
146.522
136.936
130.719
127.033
125.719
122.192
113.848
110.638
106.462
77.478
77.160
76.842
61.397
60.616
55.457
34.833
27.207
13.889
MeO
EtO2C
O
NH
Me
N
Me
4h
100 MHz/CDCl3
f1 (ppm)

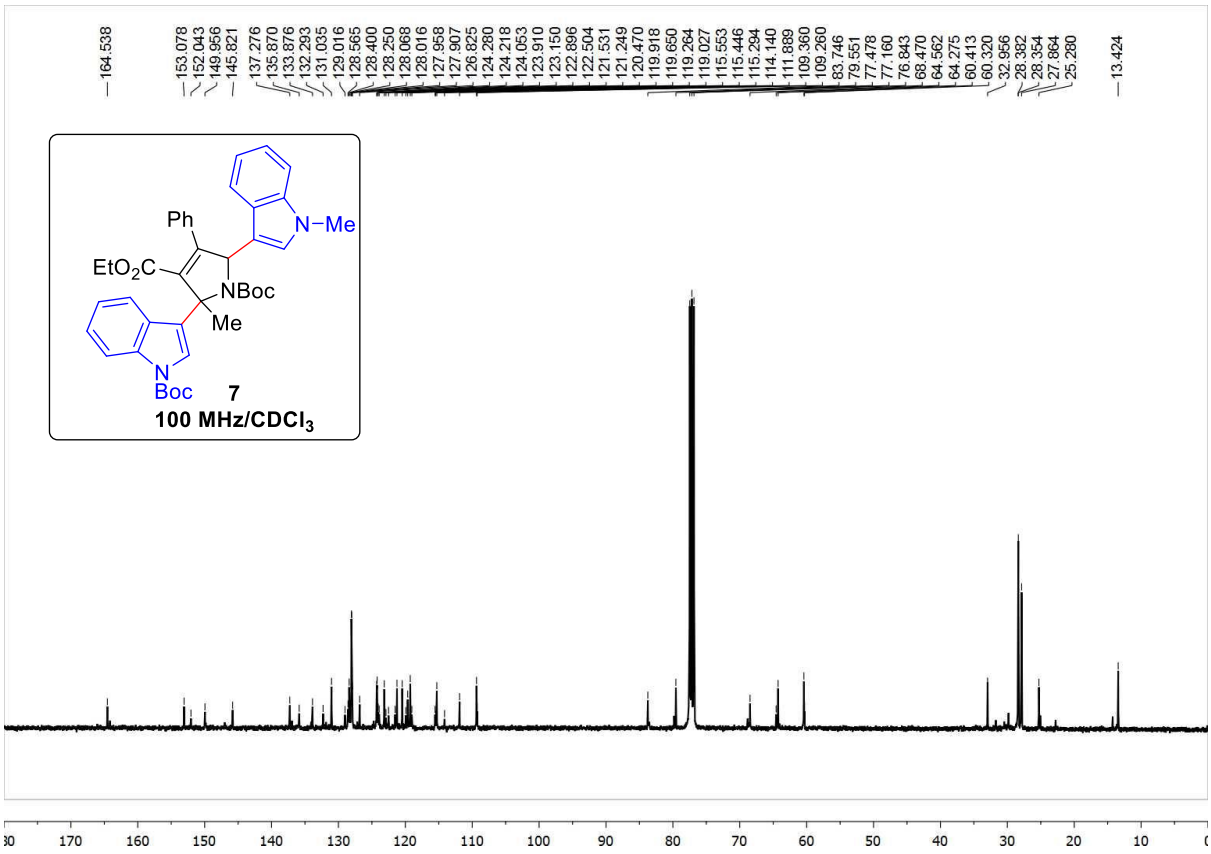

8.156
8.135
7.658
7.529
7.525
7.521
7.519
7.516
7.512
7.509
7.505
7.417
7.413
7.408
7.404
7.400
7.396
7.292
7.289
7.275
7.271
7.268
7.261
7.254
7.250
7.230
7.160
7.158
7.143
7.140
7.137
7.123
7.120
3.991
3.985
3.973
3.967
3.956
3.950
3.940
3.937
3.932
2.303
1.683
1.239
0.881
0.862
0.845
Ph
O
EtO2C
NBoc
Me
N
Boc
5
400 MHz/CDCl3
1.05
1.06
2.05
3.08
1.00
1.08
1.07
2.03
3.00
9.02
9.08
3.00
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

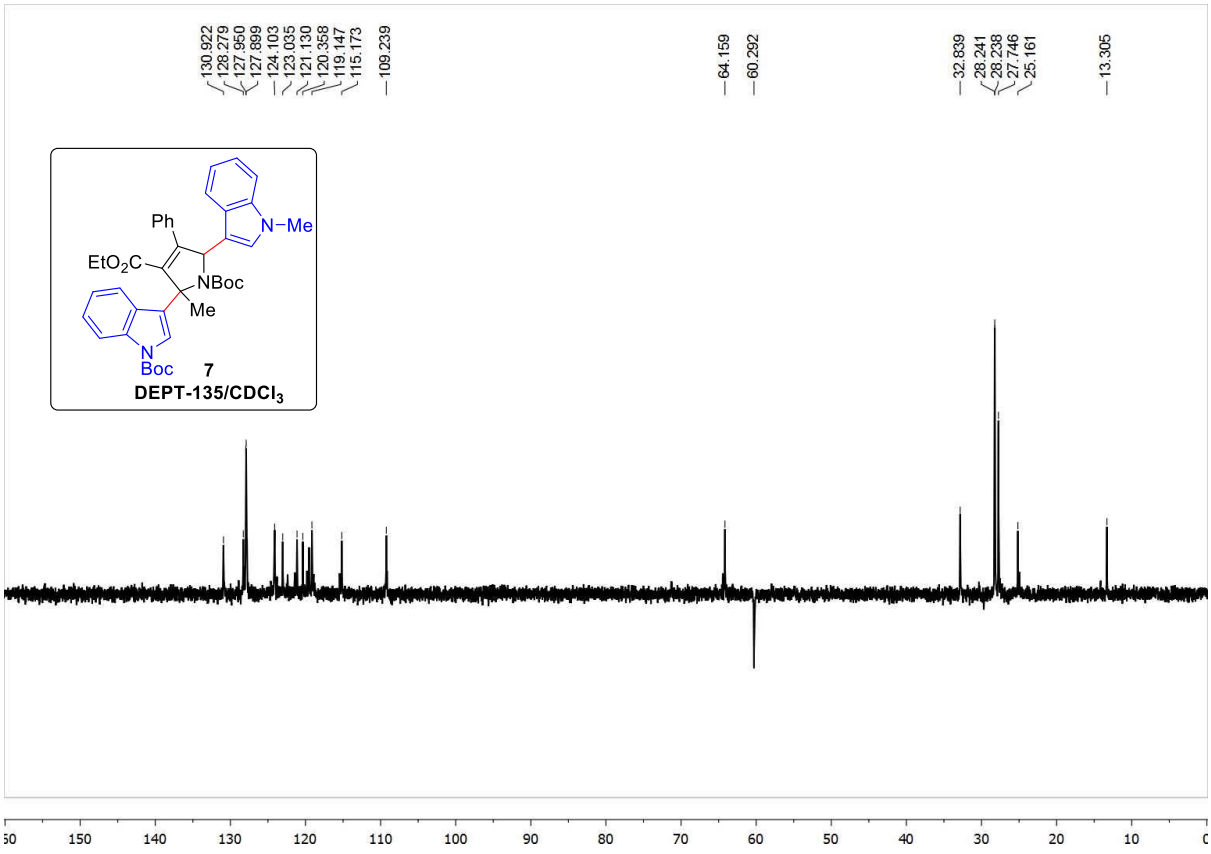

167.259
163.266
149.587
148.839
148.707
137.416
135.761
129.676
129.564
129.320
128.219
127.483
125.354
124.592
123.265
119.337
118.001
115.513
84.265
83.471
77.478
77.160
76.843
65.316
61.496
28.325
27.861
24.301
13.487
Ph
O
EtO2C
NBoc
Me
N
Boc
5
100 MHz/CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

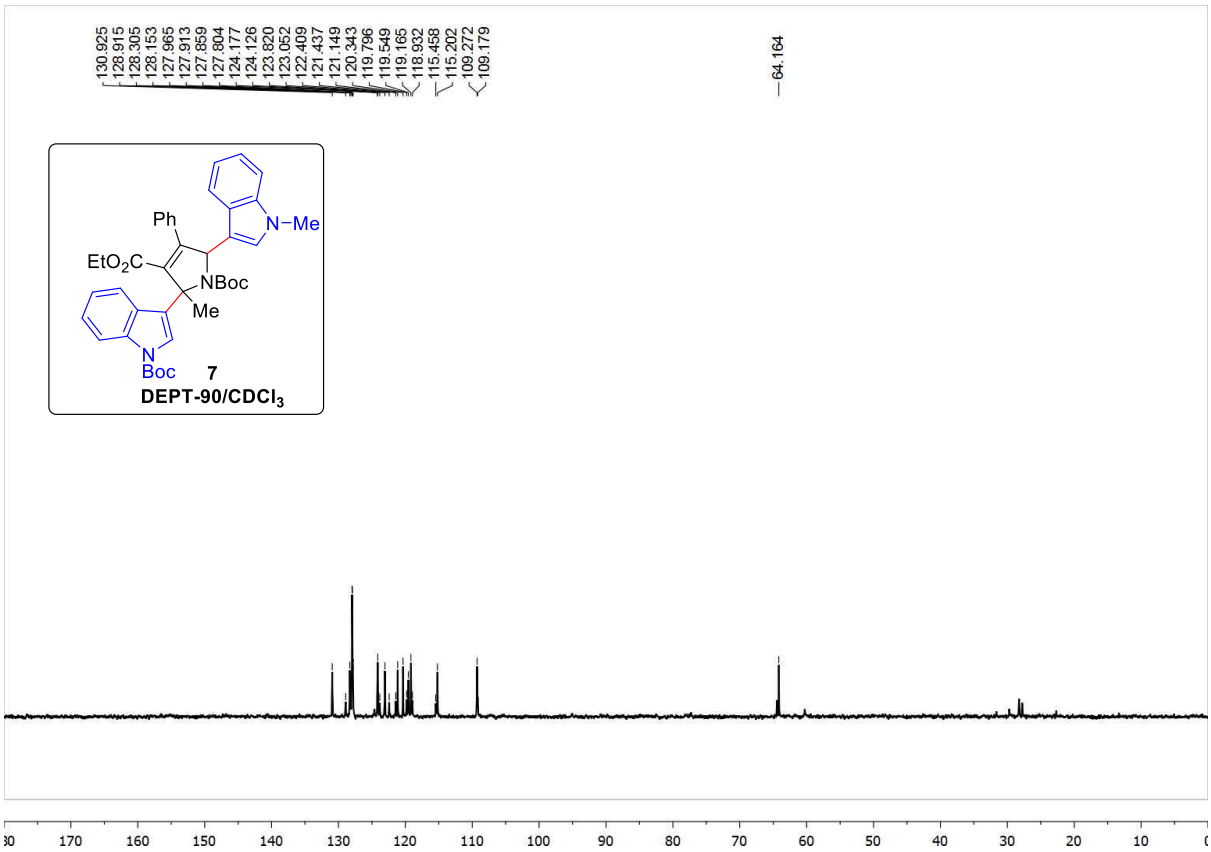

8.239
8.220
8.114
8.094
7.619
7.543
7.486
7.469
7.362
7.348
7.260
7.242
7.228
7.222
7.205
7.178
7.159
7.140
6.464
6.458
6.343
4.761
4.754
3.870
3.852
3.834
3.816
3.796
3.789
2.107
1.646
1.402
1.072
0.792
0.774
0.756
Ph
OH
EtO2C
NBoc
Me
N
Boc
6
400 MHz/CDCl3
1.05
1.00
3.04
3.08
1.03
1.06
0.98
0.98
2.02
3.00
9.06
9.00
3.01

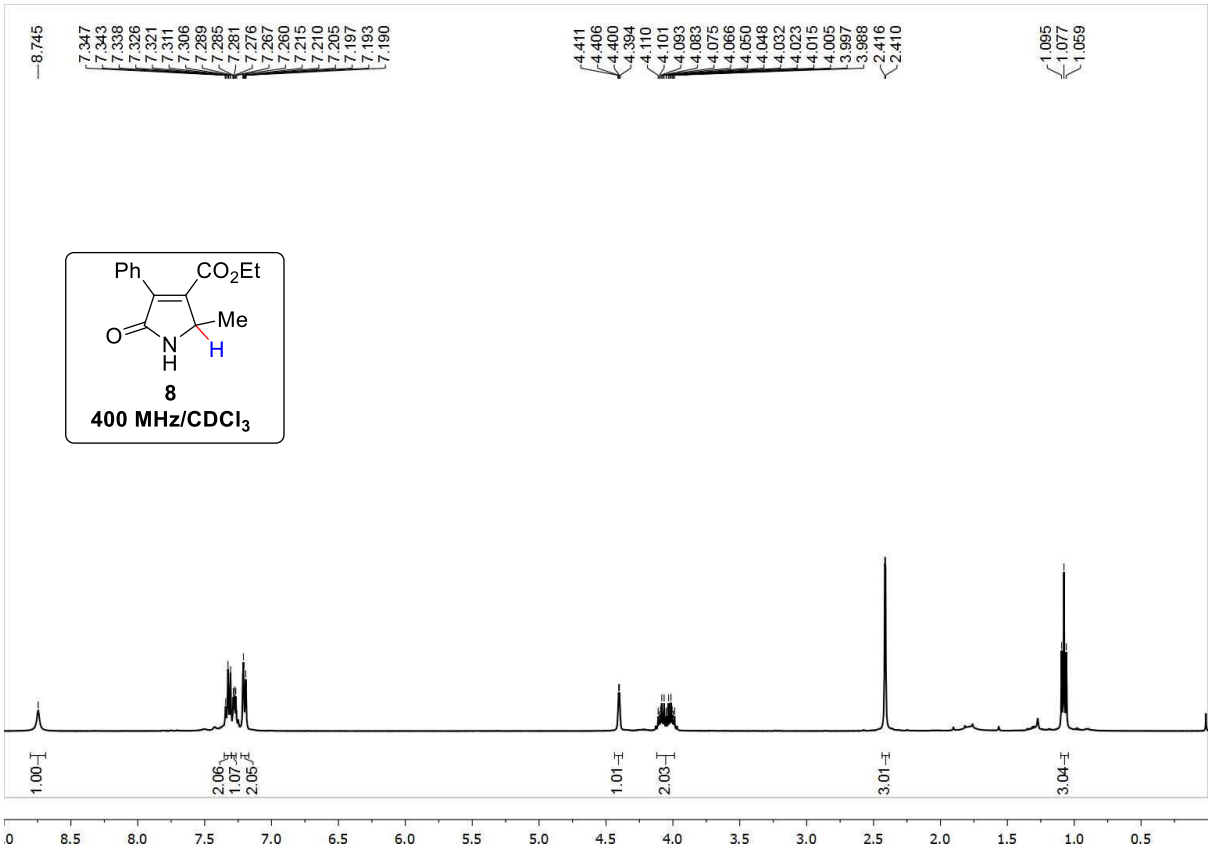

163.866
154.263
149.936
142.892
135.774
135.464
132.893
128.943
128.739
128.520
128.251
124.605
124.120
122.811
121.918
121.262
114.877
87.160
83.726
81.240
77.478
77.160
76.843
67.512
60.706
28.346
28.088
25.758
13.438
Ph
OH
EtO2C
NBoc
Me
N
Boc
6
100 MHz/CDCl3


Ph
N–Me
EtO2C
NBoc
Me
N
Boc
7
400 MHz/CDCl3


Ph
N–Me
EtO2C
NBoc
Me
N
Boc
7
100 MHz/CDCl3


130.922
128.279
127.950
127.899
124.103
123.035
121.130
120.358
119.147
115.173
109.239
64.159
60.292
32.839
28.241
28.238
27.746
25.161
13.305
Ph
N–Me
EtO2C
NBoc
Me
N
Boc
7
DEPT-135/CDCl3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


130.925
128.915
128.305
128.153
127.965
127.913
127.859
127.804
124.177
124.126
123.820
123.052
122.409
121.437
121.149
120.343
119.796
119.549
119.165
118.932
115.458
115.202
109.272
109.179
64.164
Ph
N–Me
EtO2C
NBoc
Me
N
Boc
7
DEPT-90/CDCl3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


8.745
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7.343
7.338
7.326
7.321
7.311
7.306
7.289
7.285
7.281
7.276
7.267
7.260
7.215
7.210
7.205
7.197
7.193
7.190
4.411
4.406
4.400
4.394
4.110
4.101
4.093
4.083
4.075
4.066
4.050
4.048
4.032
4.023
4.015
4.005
3.997
3.988
2.416
2.410
1.095
1.077
1.059
Ph
CO2Et
Me
O
N
H
H
8
400 MHz/CDCl3
1.00
2.06
1.07
2.05
1.01
2.03
3.01
3.04
0
8.5
8.0
7.5
7.0
6.5
6.0
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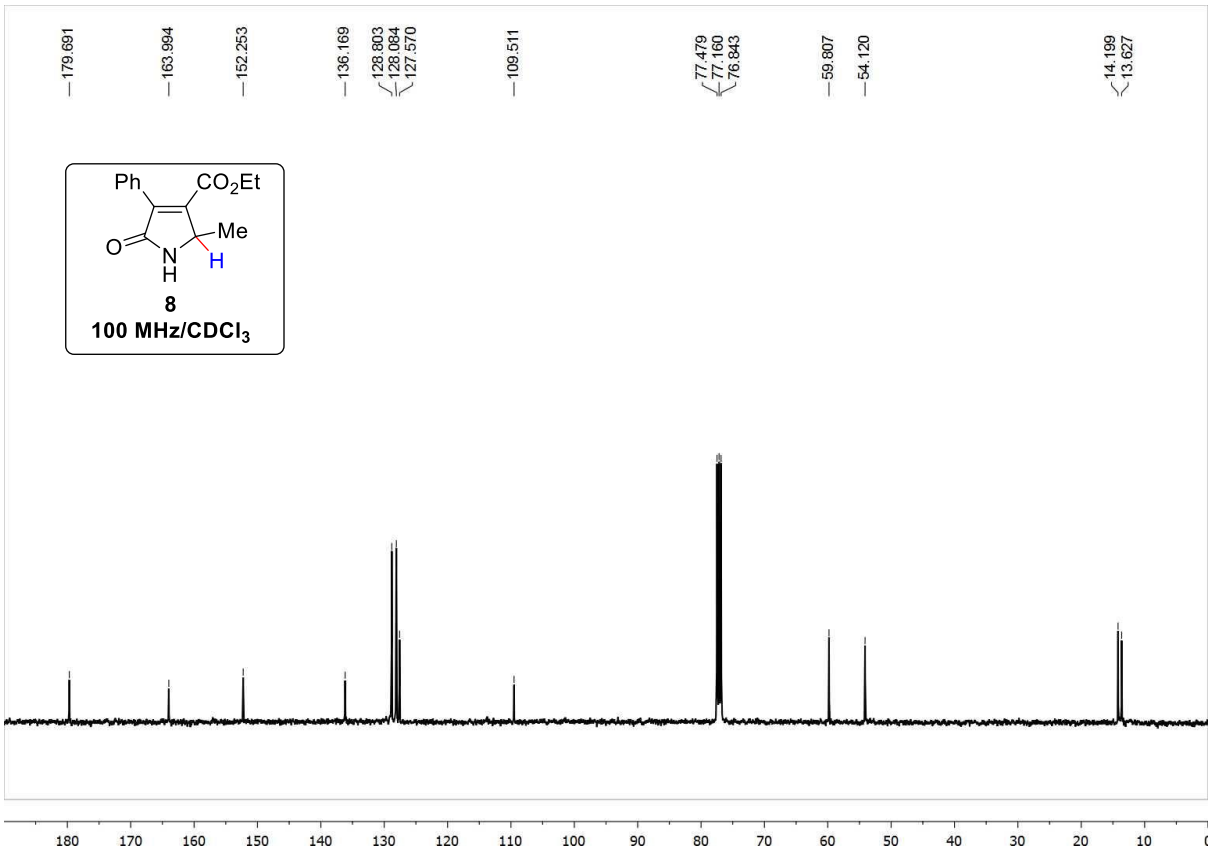
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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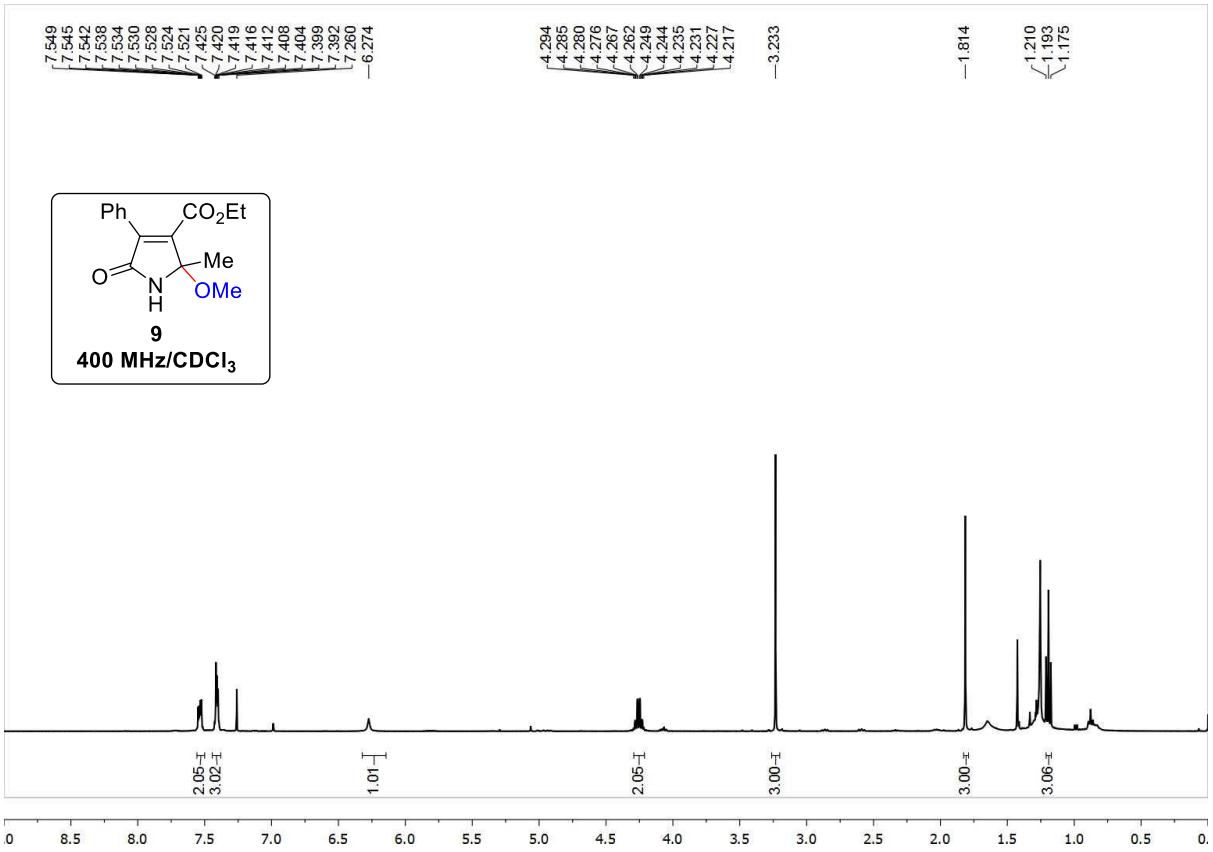
127.570
109.511
77.479
77.160
76.843
59.807
54.120
14.199
13.627
Ph
CO2Et
Me
O
N
H
H
8
100 MHz/CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10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7.545
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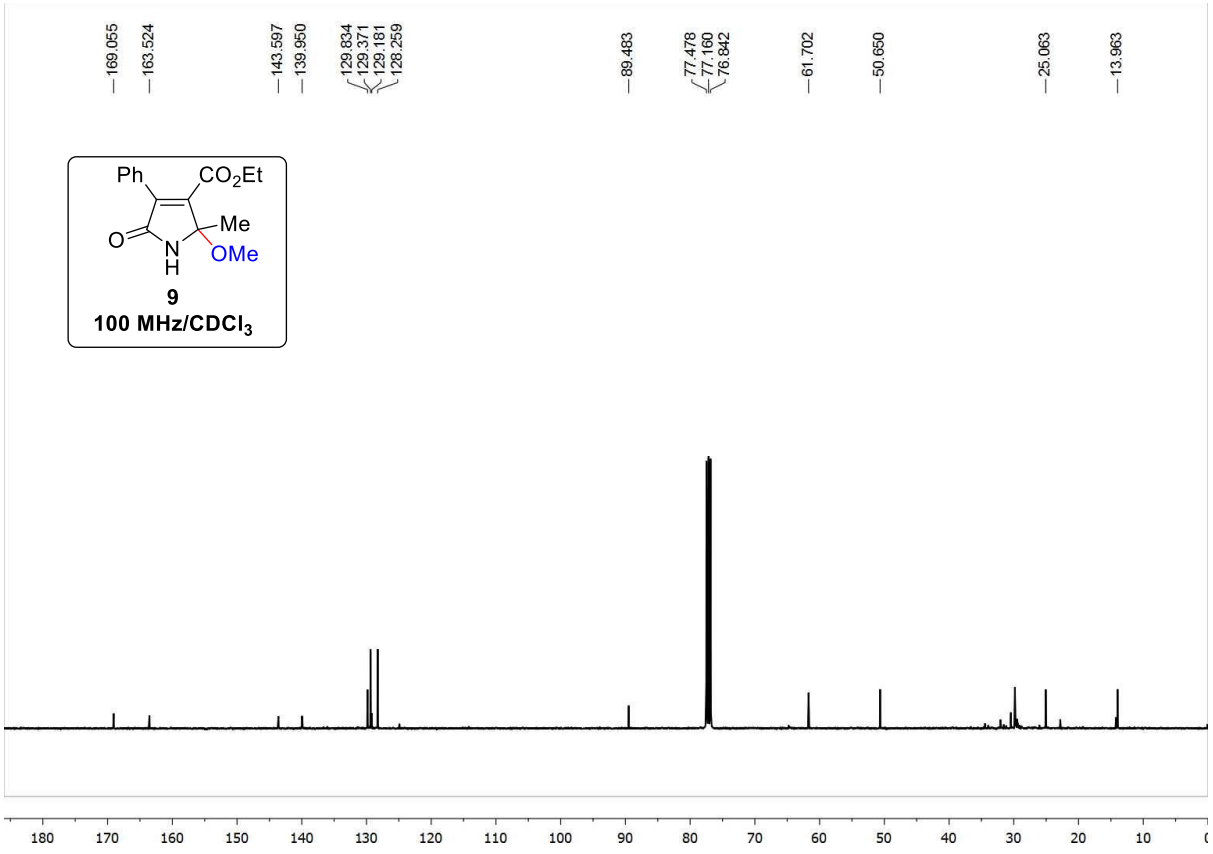
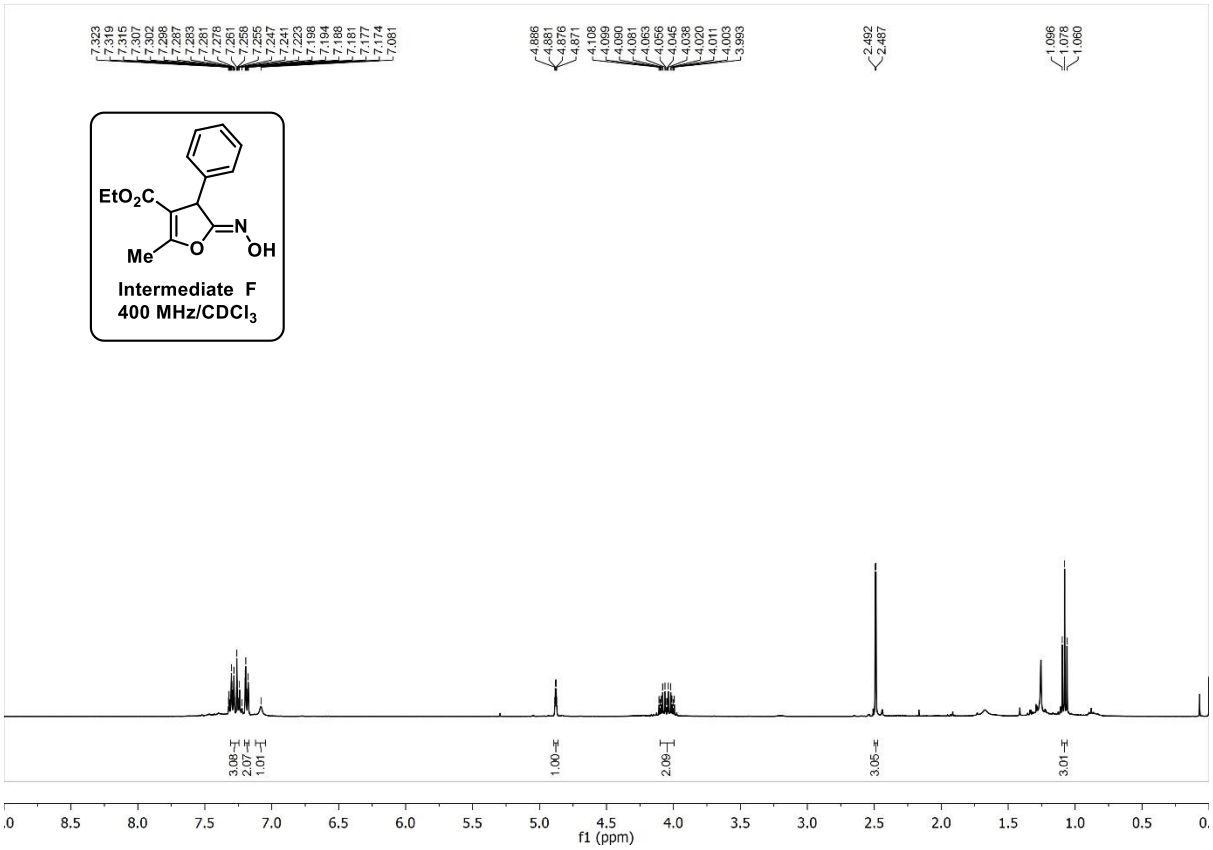
7.538
7.534
7.530
7.528
7.524
7.521
7.425
7.420
7.419
7.416
7.412
7.408
7.404
7.399
7.392
7.260
6.274
4.294
4.285
4.280
4.276
4.267
4.262
4.249
4.244
4.235
4.231
4.227
4.217
3.233
1.814
1.210
1.193
1.175
Ph
CO2Et
Me
O
N
H
OMe
9
400 MHz/CDCl3
2.05
3.02
1.01
2.05
3.00
3.00
3.06
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5


169.055
163.524
143.597
139.950
129.834
129.371
129.181
128.259
89.483
77.478
77.160
76.842
61.702
50.650
25.063
13.963
Ph
CO2Et
Me
O
N
H
OMe
9
100 MHz/CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


7.323
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7.307
7.302
7.298
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7.278
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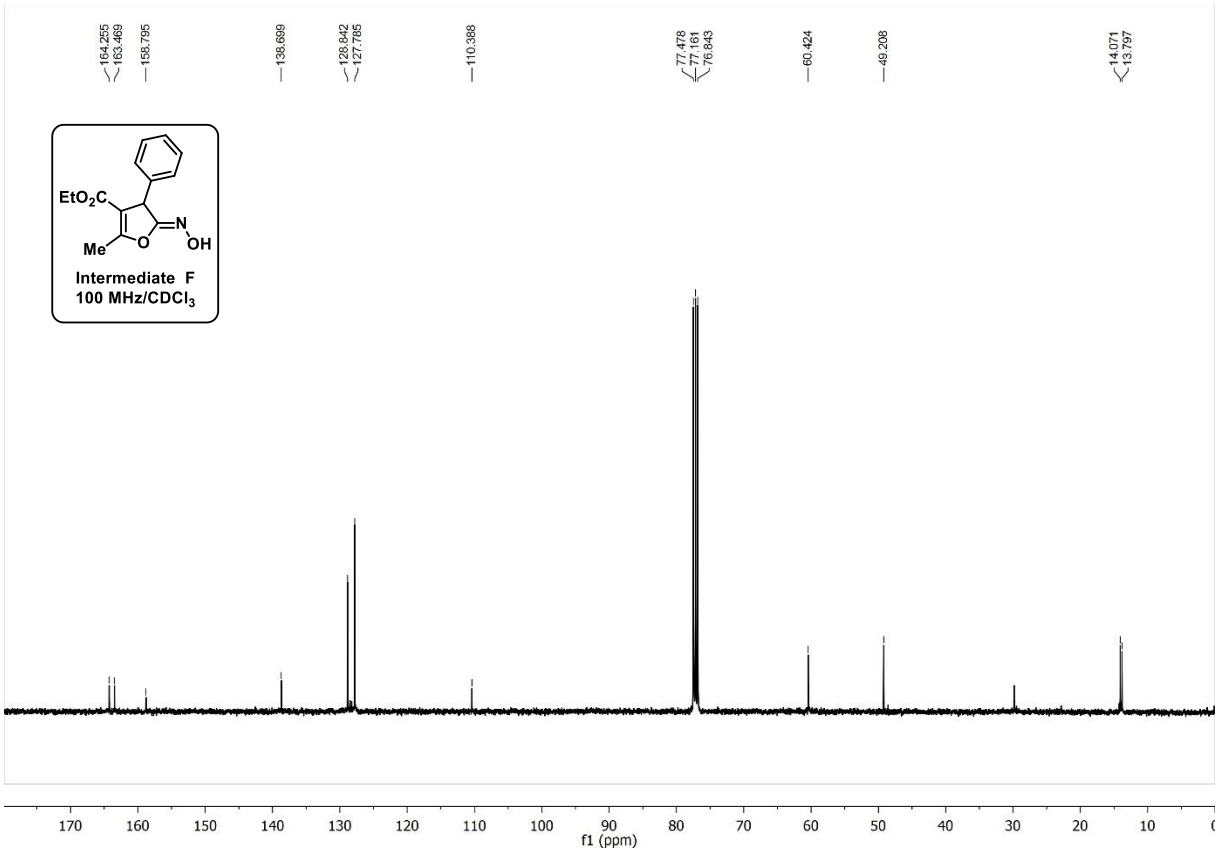
7.255
7.247
7.241
7.223
7.198
7.194
7.188
7.181
7.177
7.174
7.081
4.886
4.881
4.876
4.871
4.108
4.099
4.090
4.081
4.063
4.056
4.045
4.038
4.020
4.011
4.003
3.993
2.492
2.487
1.096
1.078
1.060
EtO2C
Me
O
N
OH
Intermediate F
400 MHz/CDCl3
3.08
2.07
1.01
1.00
2.09
3.05
3.01
f1 (ppm)


164.255
163.469
158.795
138.699
128.842
127.785
110.388
77.478
77.161
76.843
60.424
49.208
14.071
13.797
EtO2C
Me
O
N
OH
Intermediate F
100 MHz/CDCl3
f1 (ppm)