Transformation of 3-(Furan-2-yl)-1,3-di(het)arylpropan-1-ones to the Corresponding Prop-2-en-1-ones *via* the Oxidative Furan Dearomatization/2-Ene-1,4,7-triones Cyclization

Roman O. Shcherbakov,[†] Diana A. Eshmemet'eva,[†] Anton A. Merkushev,[†] Igor V. Trushkov,[§,‡] Maxim G. Uchuskin*[†]

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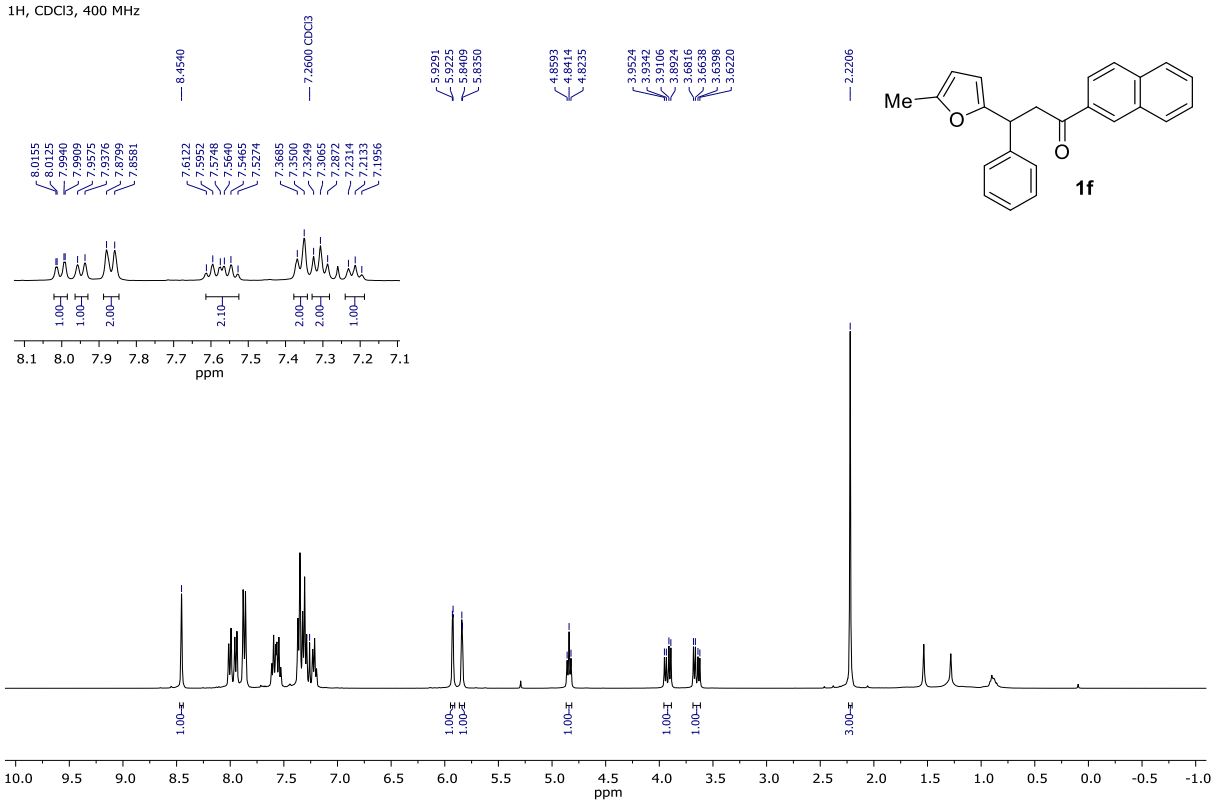
Supporting Information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4.8235
3.9524
3.9342
3.9106
3.8924
3.6816
3.6638
3.6398
3.6220
2.2206
8.0155
8.0125
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7.9909
7.9575
7.9376
7.8799
7.8581
7.6122
7.5952
7.5748
7.5640
7.5465
7.5274
7.3685
7.3500
7.3249
7.3065
7.2872
7.2314
7.2133
7.1956
1.00
1.00
2.00
2.10
2.00
2.00
1.00
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1
ppm
1.00
1.00
1.00
1.00
1.00
3.00
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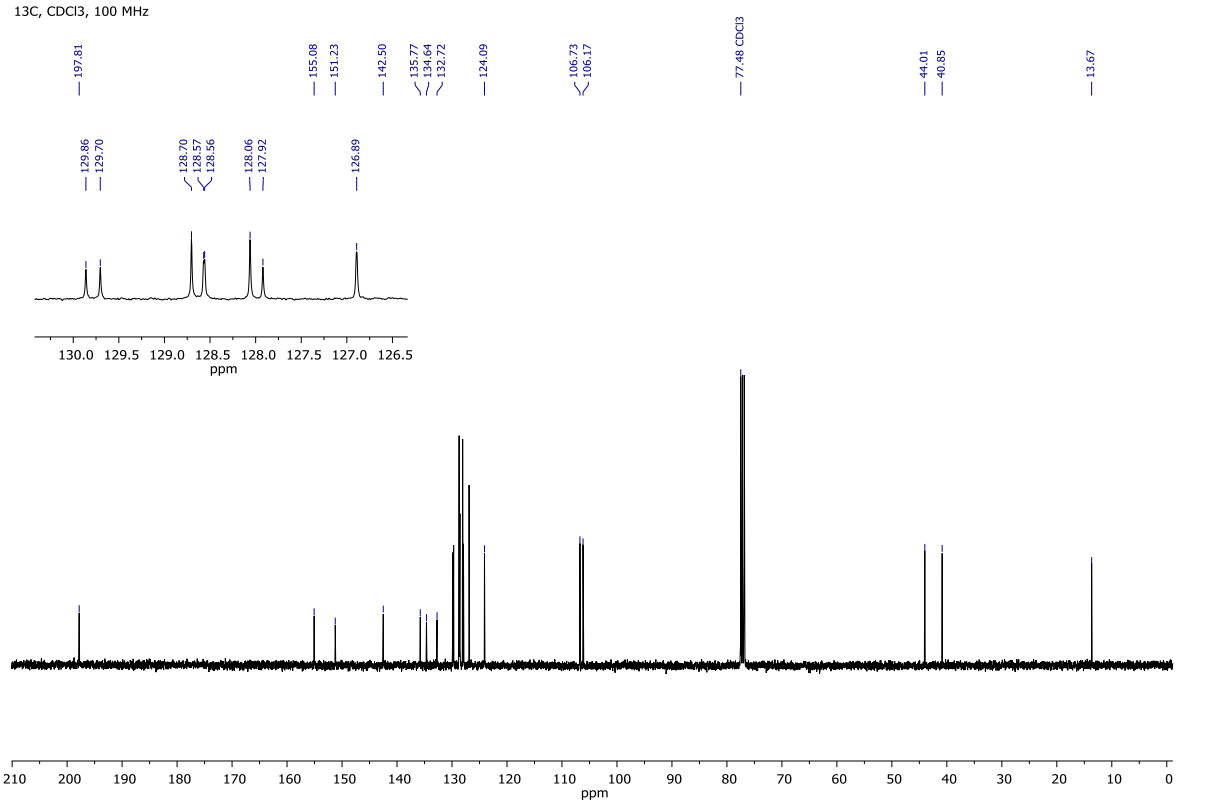
O
O
1f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134.64
132.72
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106.17
77.48 CDCl3
44.01
40.85
13.67
129.86
129.70
128.70
128.57
128.56
128.06
127.92
126.89
130.0 129.5 129.0 128.5 128.0 127.5 127.0 126.5
ppm
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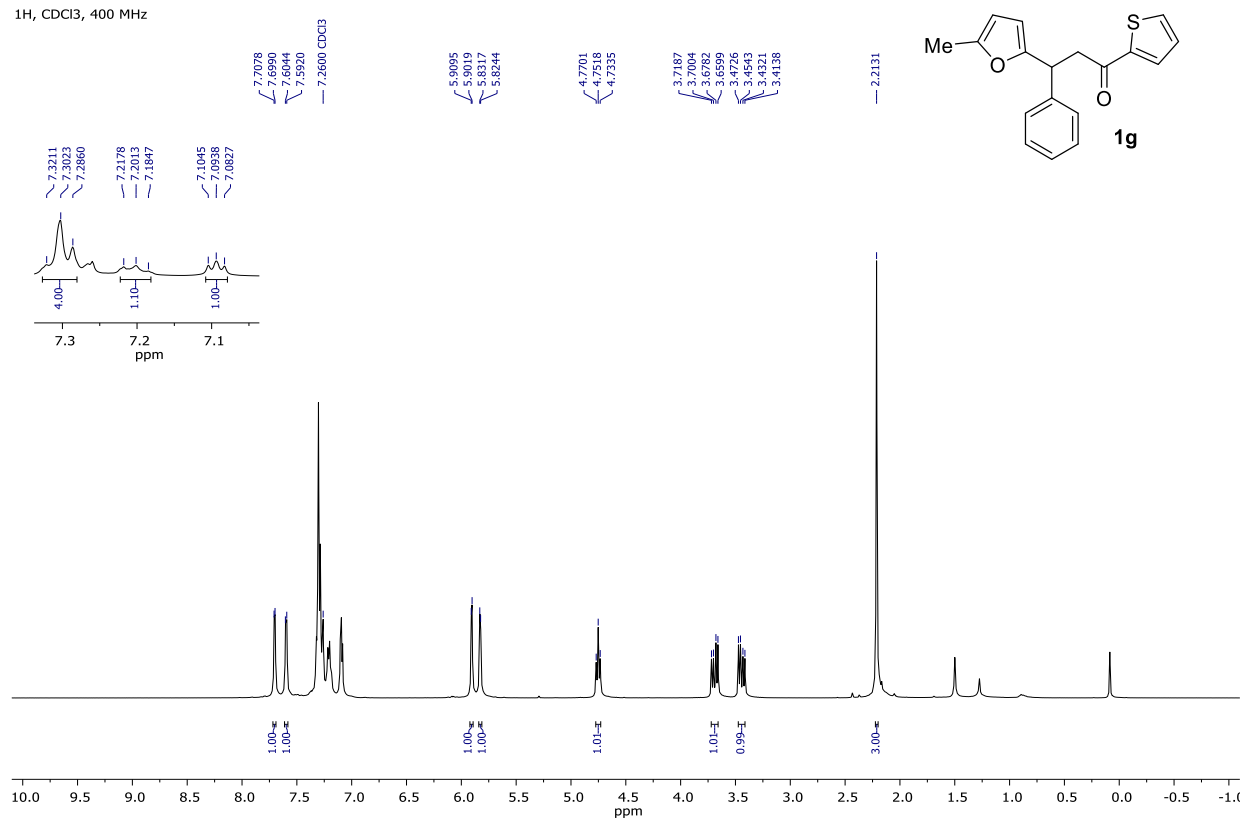
ppm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3.7187
3.7004
3.6782
3.6599
3.4726
3.4543
3.4321
3.4138
2.2131
7.3211
7.3023
7.2860
7.2178
7.2013
7.1847
7.1045
7.0938
7.0827
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1.10
1.00
7.3
7.2
7.1
ppm
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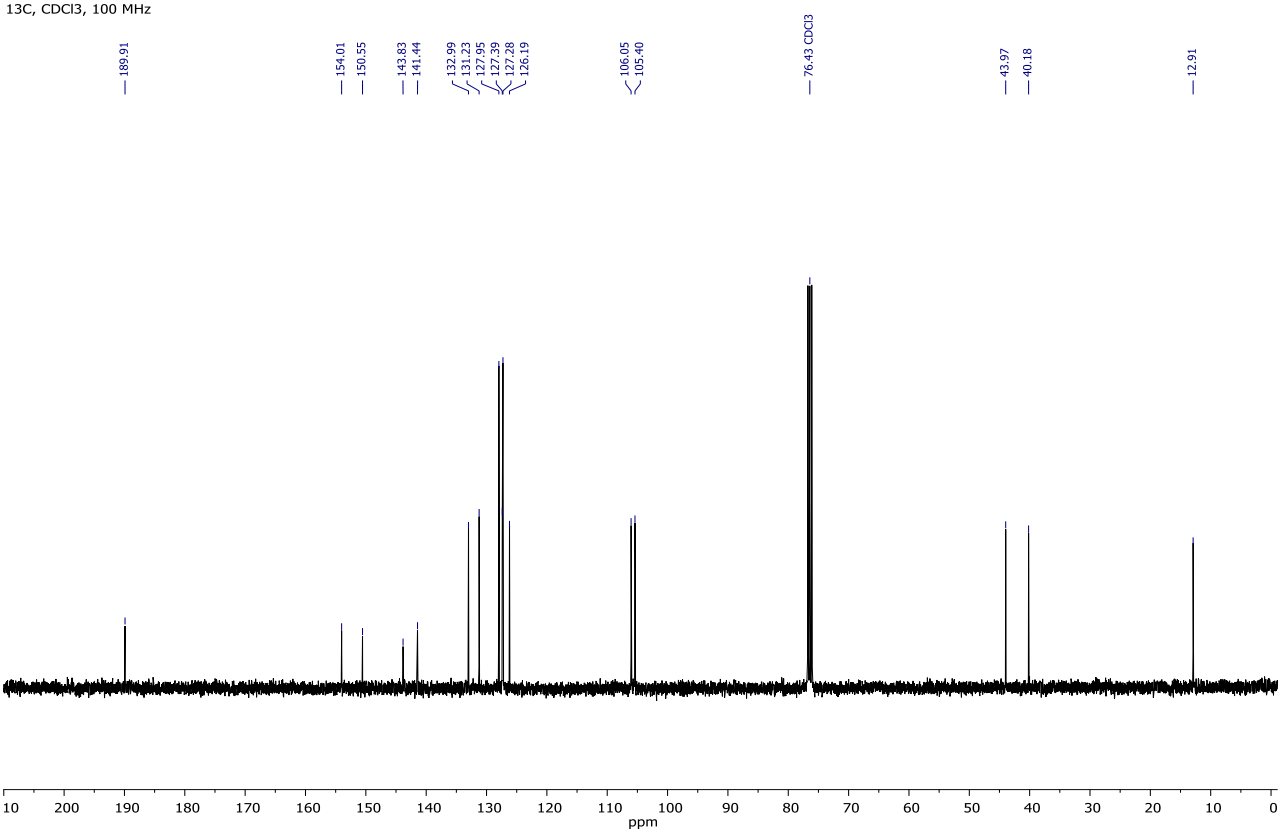
1.00
1.00
1.00
1.01
1.01
0.99
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
Me
O
S
O
1g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143.83
141.44
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127.95
127.39
127.28
126.19
106.05
105.40
76.43 CDCl3
43.97
40.18
12.91
10
200
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180
170
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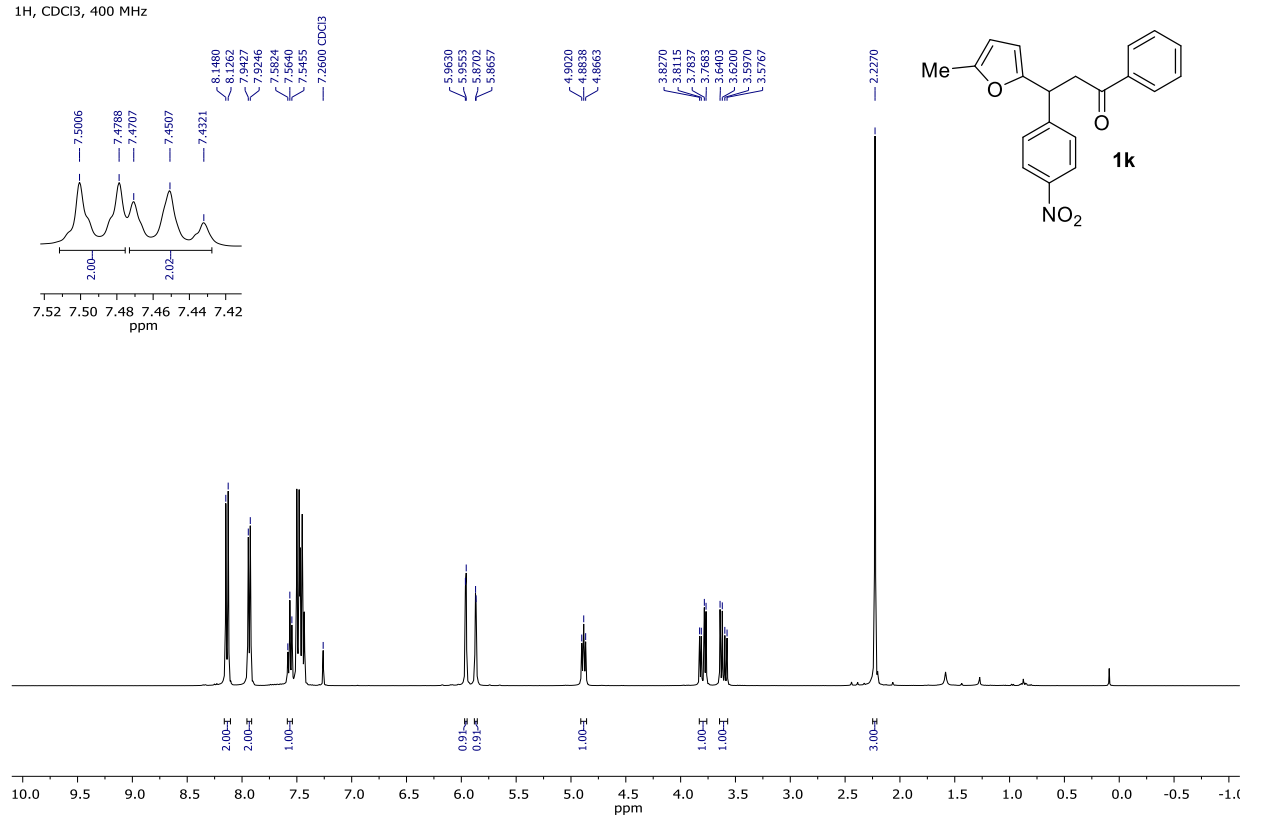
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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5.9553
5.8702
5.8657
4.9020
4.8838
4.8663
3.8270
3.8115
3.7837
3.7683
3.6403
3.6200
3.5970
3.5767
2.2270
7.5006
7.4788
7.4707
7.4507
7.4321
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2.02
7.52 7.50 7.48 7.46 7.44 7.42
ppm
Me
O
O
NO2
1k
2.00
2.00
1.00
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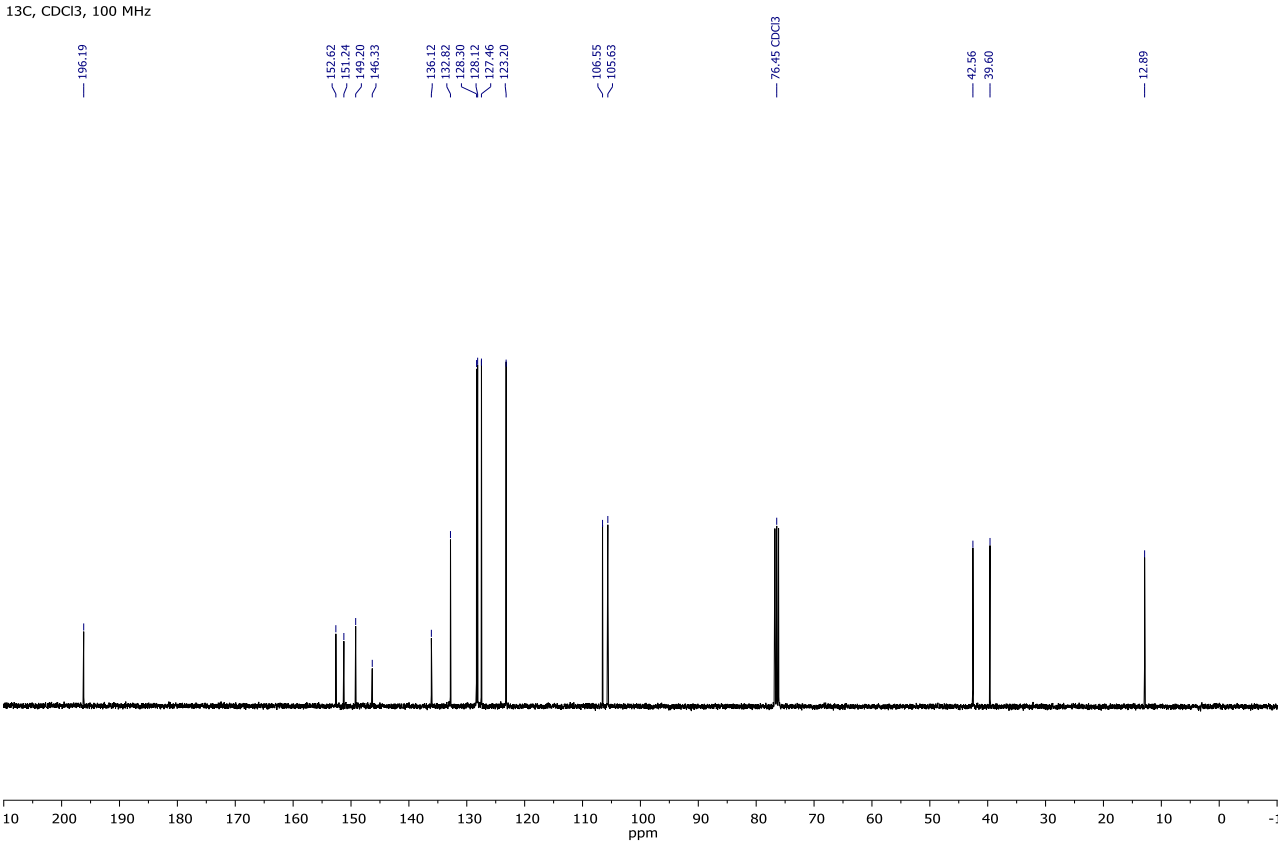
0.91
1.00
1.00
1.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
ppm


13C, CDCl3, 100 MHz
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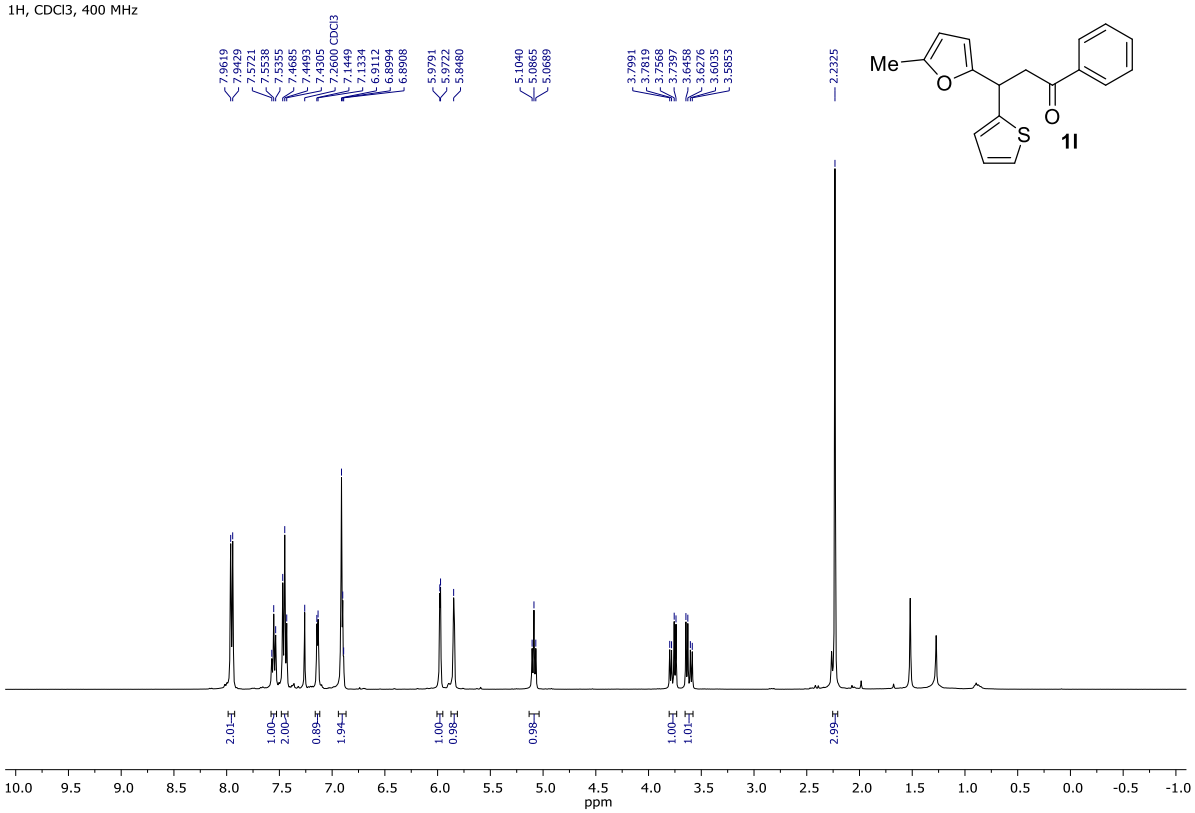
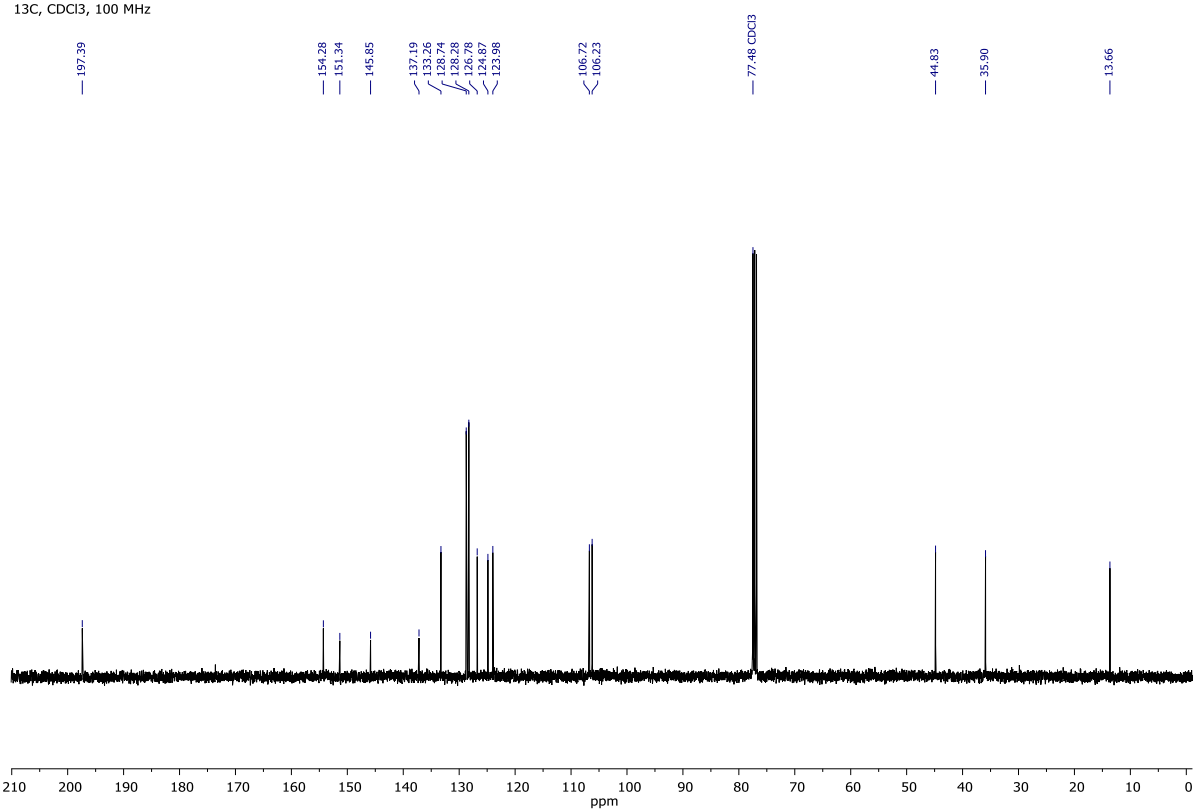
152.62
151.24
149.20
146.33
136.12
132.82
128.30
128.12
127.46
123.20
106.55
105.63
76.45 CDCl3
42.56
39.60
12.89
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

1H, CDCl3, 400 MHz

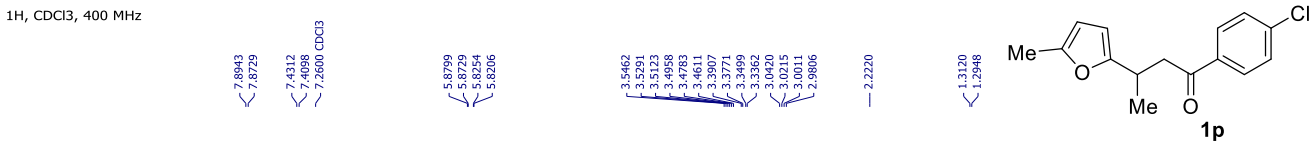


1l

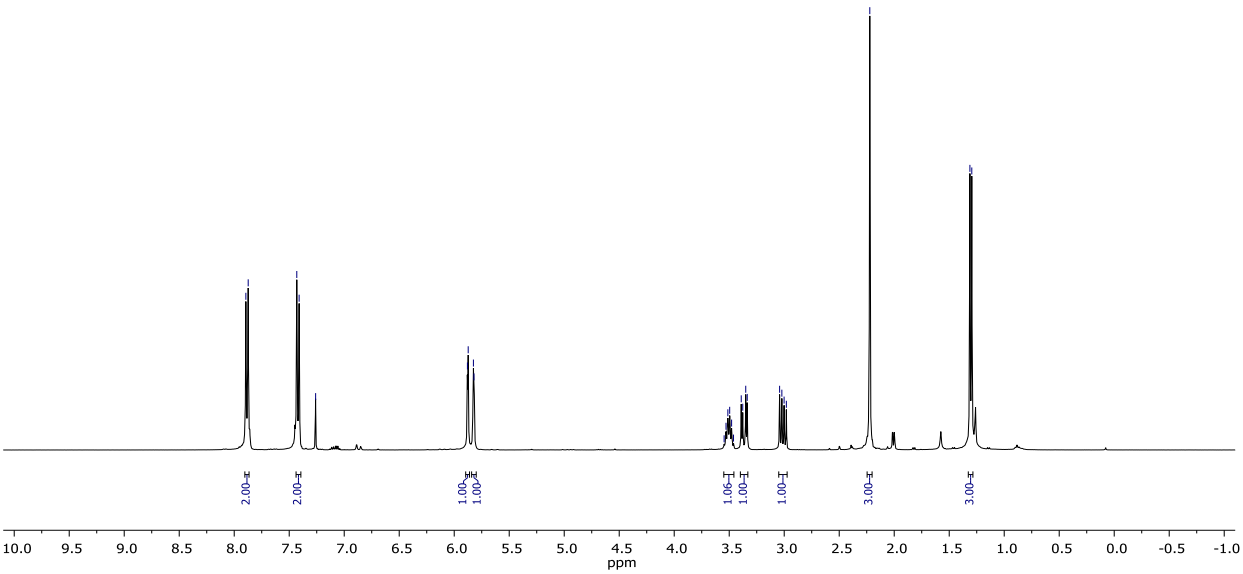
13C, CDCl3, 100 MHz



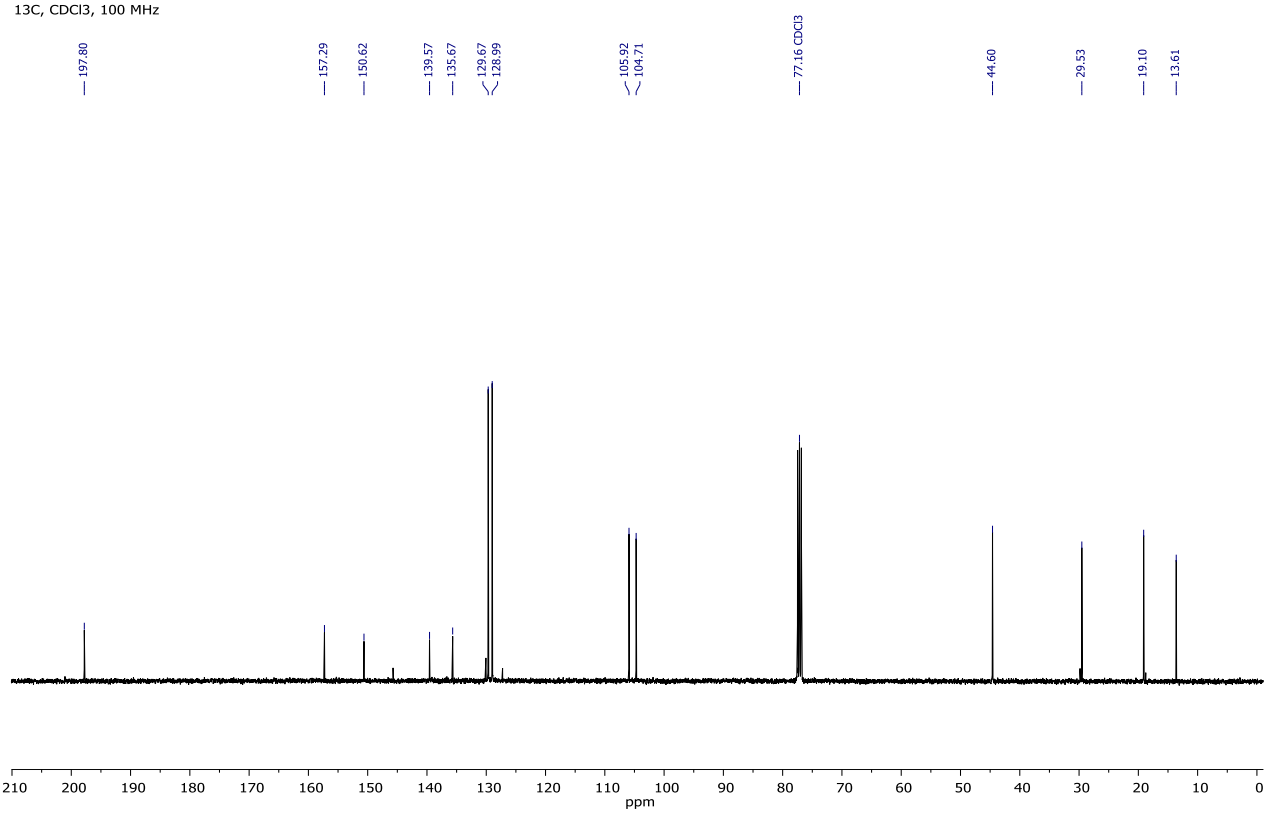
1H, CDCl3, 400 MHz



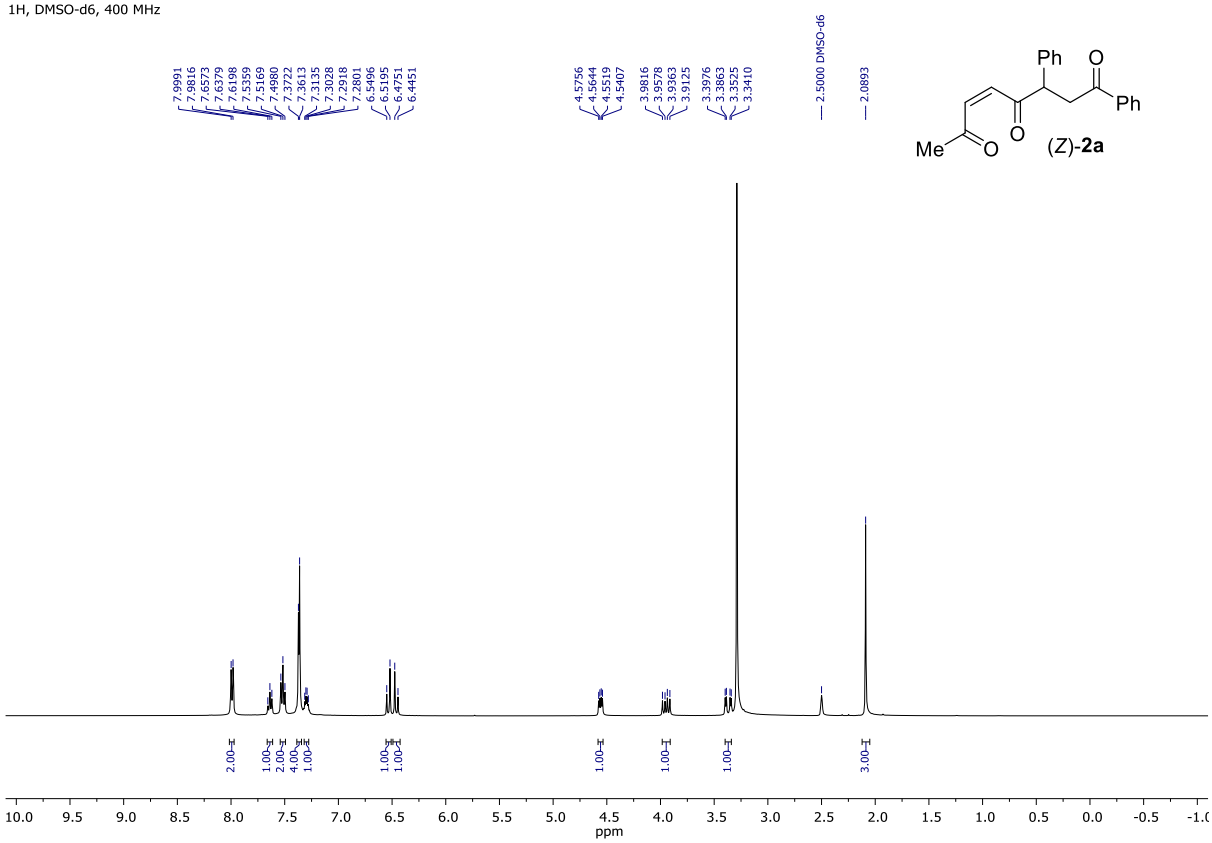
1p



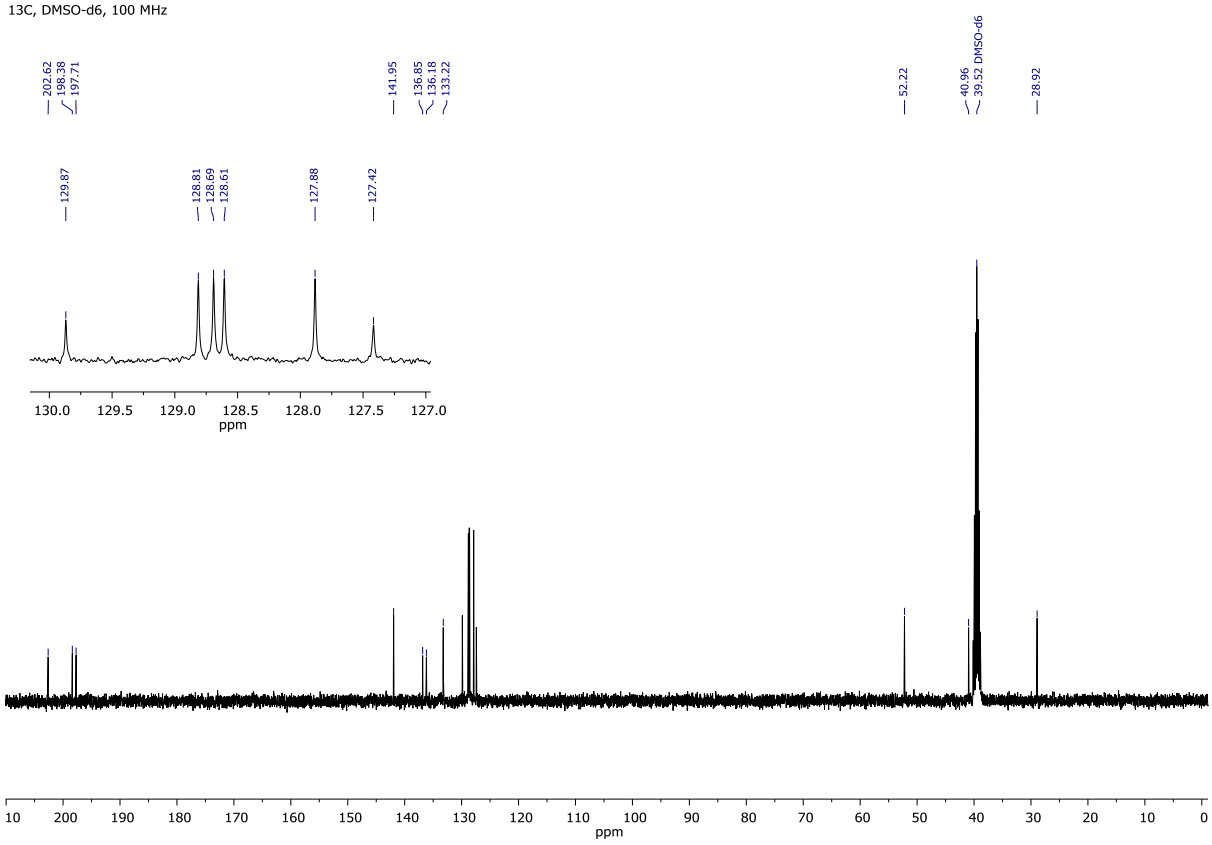
13C, CDCl3, 100 MHz

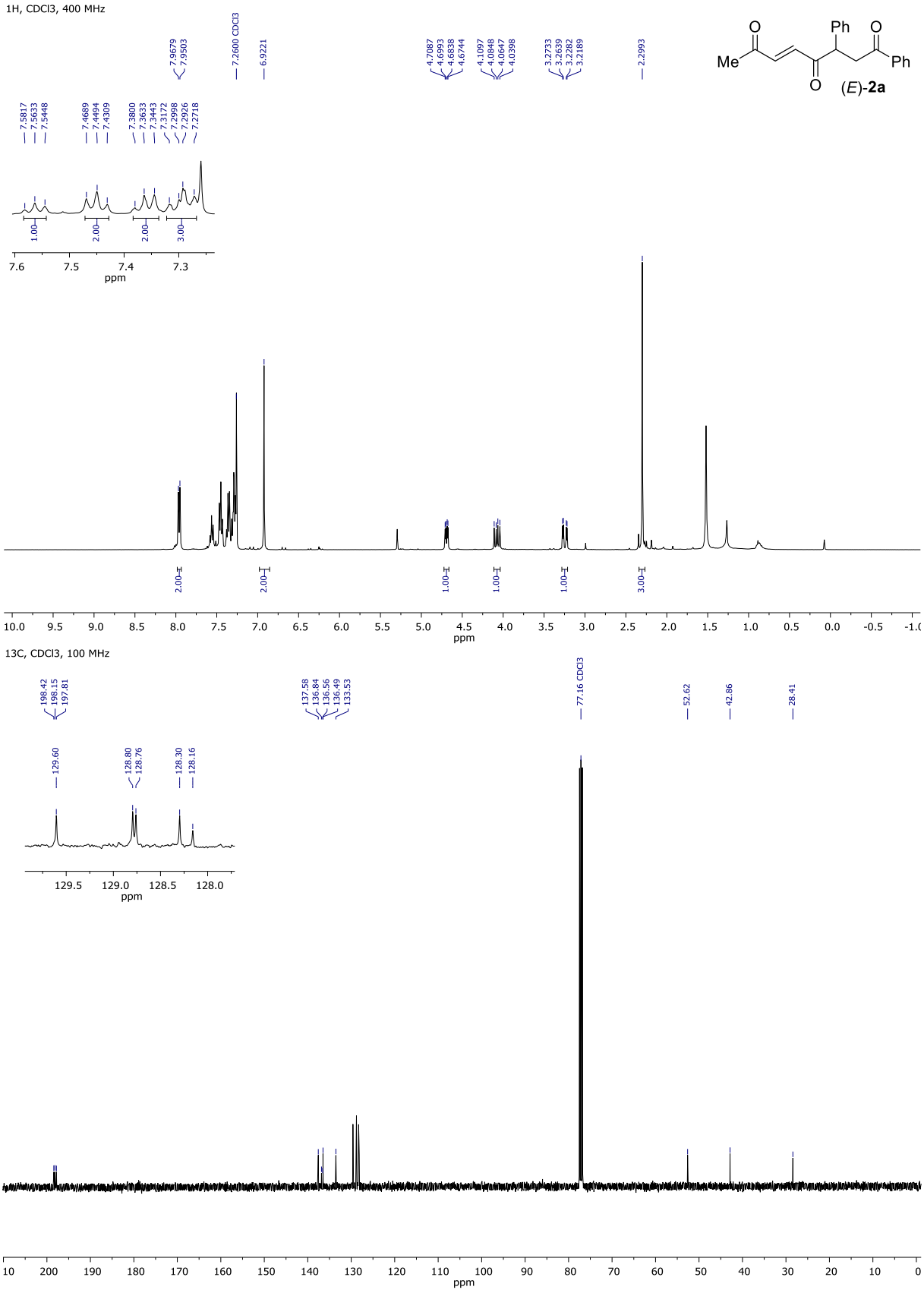


1H, DMSO-d6, 400 MHz



13C, DMSO-d6, 100 MHz

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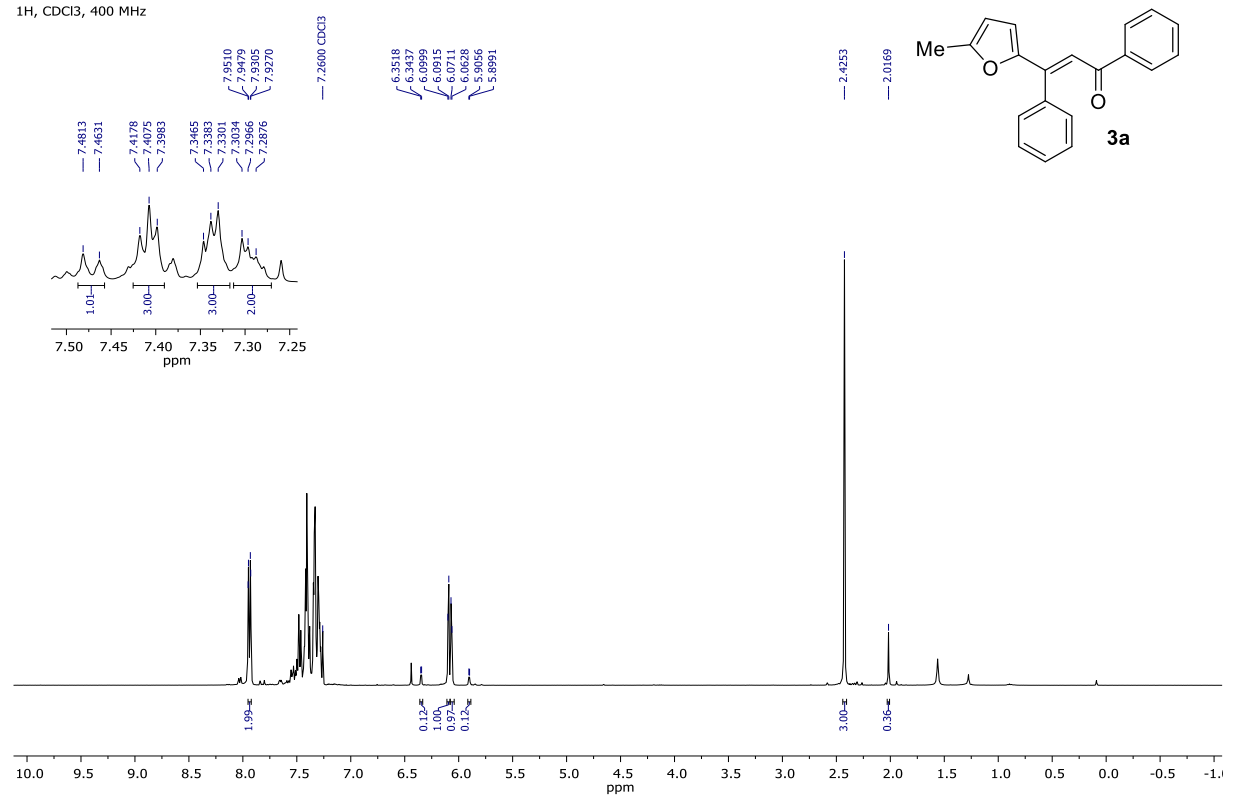
4.6744
4.1097
4.0848
4.0647
4.0398
3.2733
3.2639
3.2282
3.2189
2.2993
7.5817
7.5633
7.5448
7.4689
7.4494
7.4309
7.3800
7.3633
7.3443
7.3172
7.2998
7.2926
7.2718
1.00
2.00
2.00
3.00
7.6
7.5
7.4
7.3
ppm
2.00
2.00
1.00
1.00
1.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
Me
Ph
O
O
O
Ph
(E)-2a
13C, CDCl3, 100 MHz
198.42
198.15
197.81
137.58
136.84
136.66
136.49
133.53
77.16 CDCl3
52.62
42.86
28.41
129.60
128.80
128.76
128.30
128.16
129.5
129.0
128.5
128.0
ppm
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


1H, CDCl3, 400 MHz
Me
O
O
3a
7.9510
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7.2600 CDCl3
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6.3437
6.0999
6.0915
6.0711
6.0628
5.9056
5.8991
2.4253
2.0169
7.4813
7.4631
7.4178
7.4075
7.3983
7.3465
7.3383
7.3301
7.3034
7.2966
7.2876
1.01
3.00
3.00
2.00
7.50 7.45 7.40 7.35 7.30 7.25
ppm
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0.12
1.00
0.97
0.12
3.00
0.36
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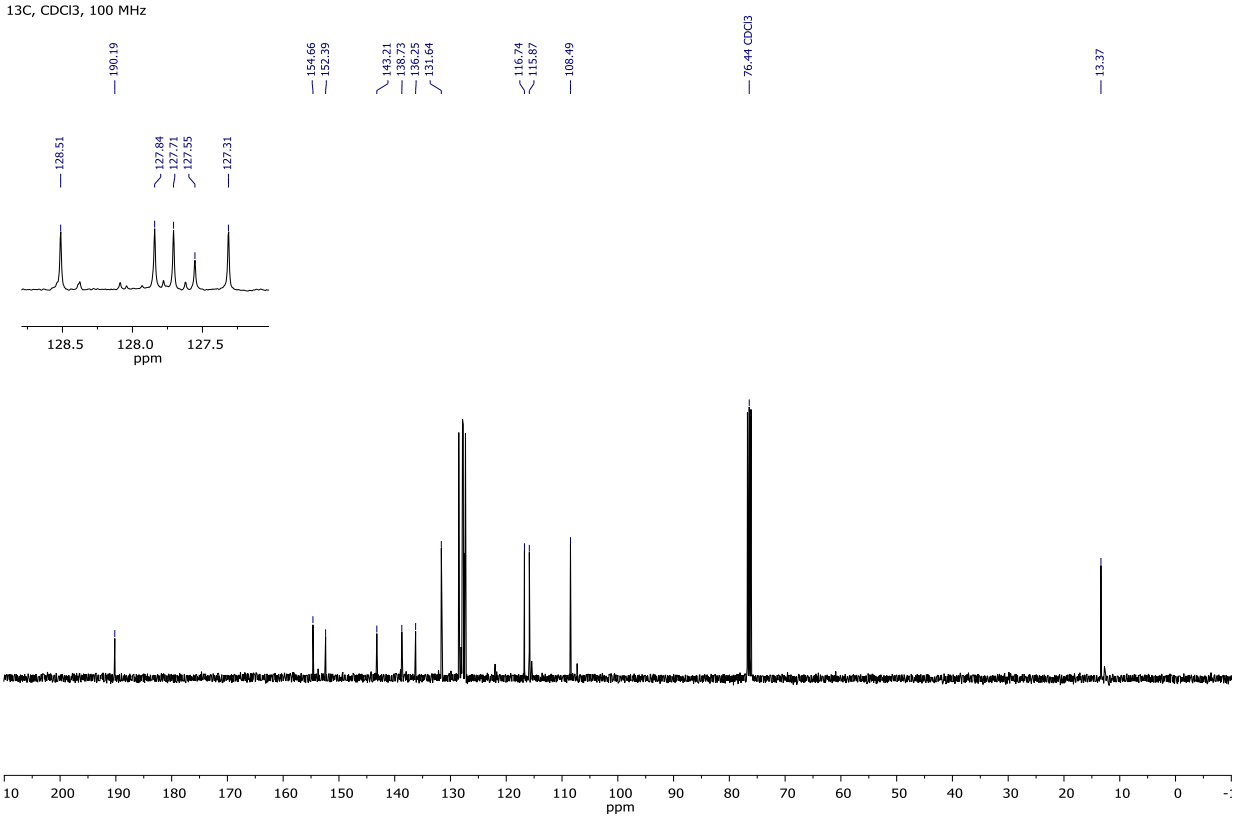
ppm


13C, CDCl3, 100 MHz
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143.21
138.73
136.25
131.64
116.74
115.87
108.49
76.44 CDCl3
13.37
128.51
127.84
127.71
127.55
127.31
128.5 128.0 127.5
ppm
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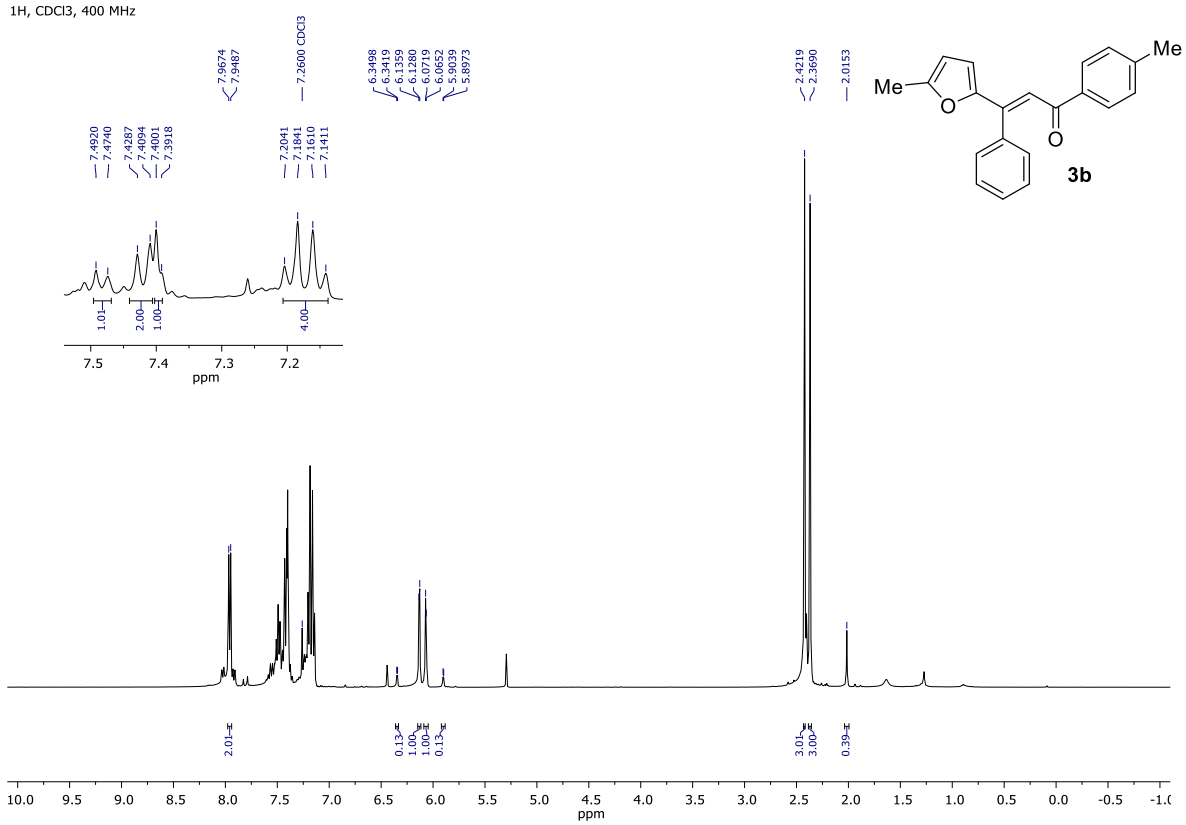
ppm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5.9039
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2.3690
2.0153
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7.4740
7.4287
7.4094
7.4001
7.3918
7.2041
7.1841
7.1610
7.1411
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2.00
1.00
4.00
7.5
7.4
7.3
7.2
ppm
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1.00
1.00
0.13
3.01
3.00
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8.0
7.5
7.0
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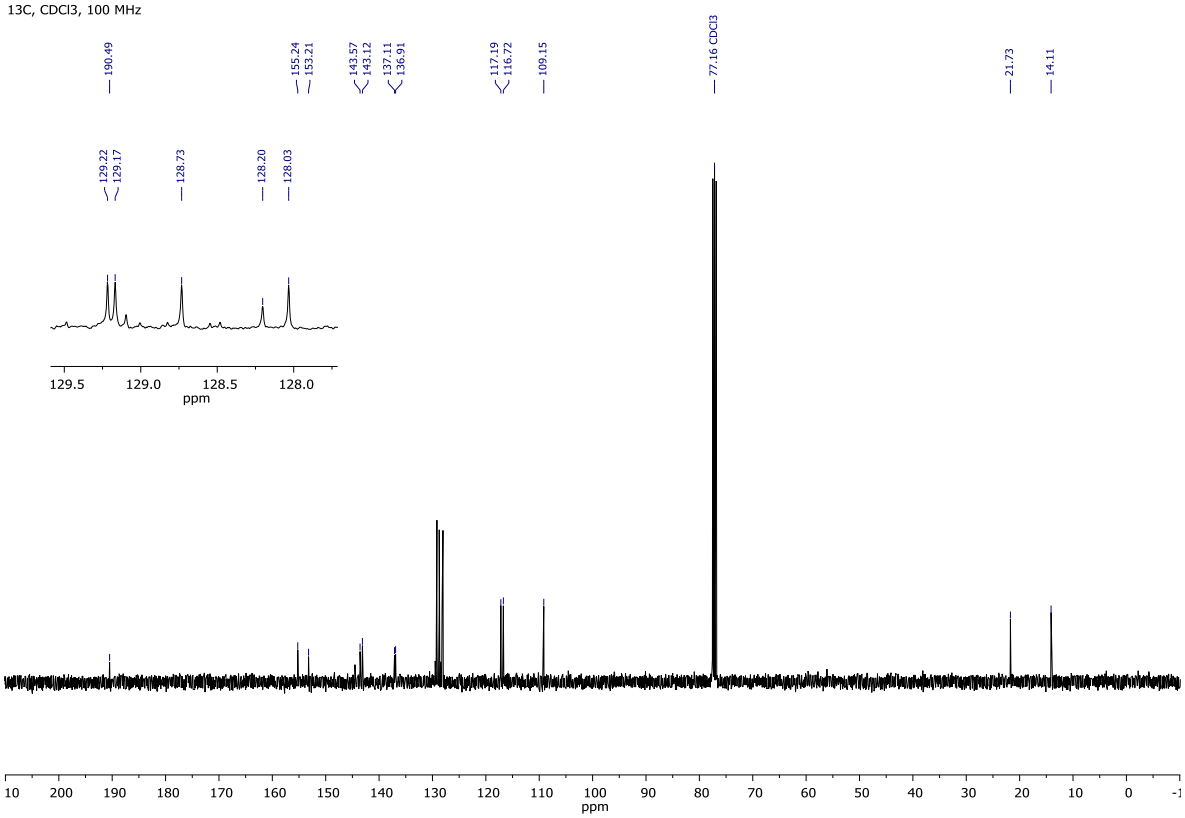
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
Me
Me
O
O
3b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117.19
116.72
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21.73
14.11
129.22
129.17
128.73
128.20
128.03
129.5
129.0
128.5
128.0
ppm
10
200
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180
170
160
150
140
130
120
110
100
90
80
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60
50
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20
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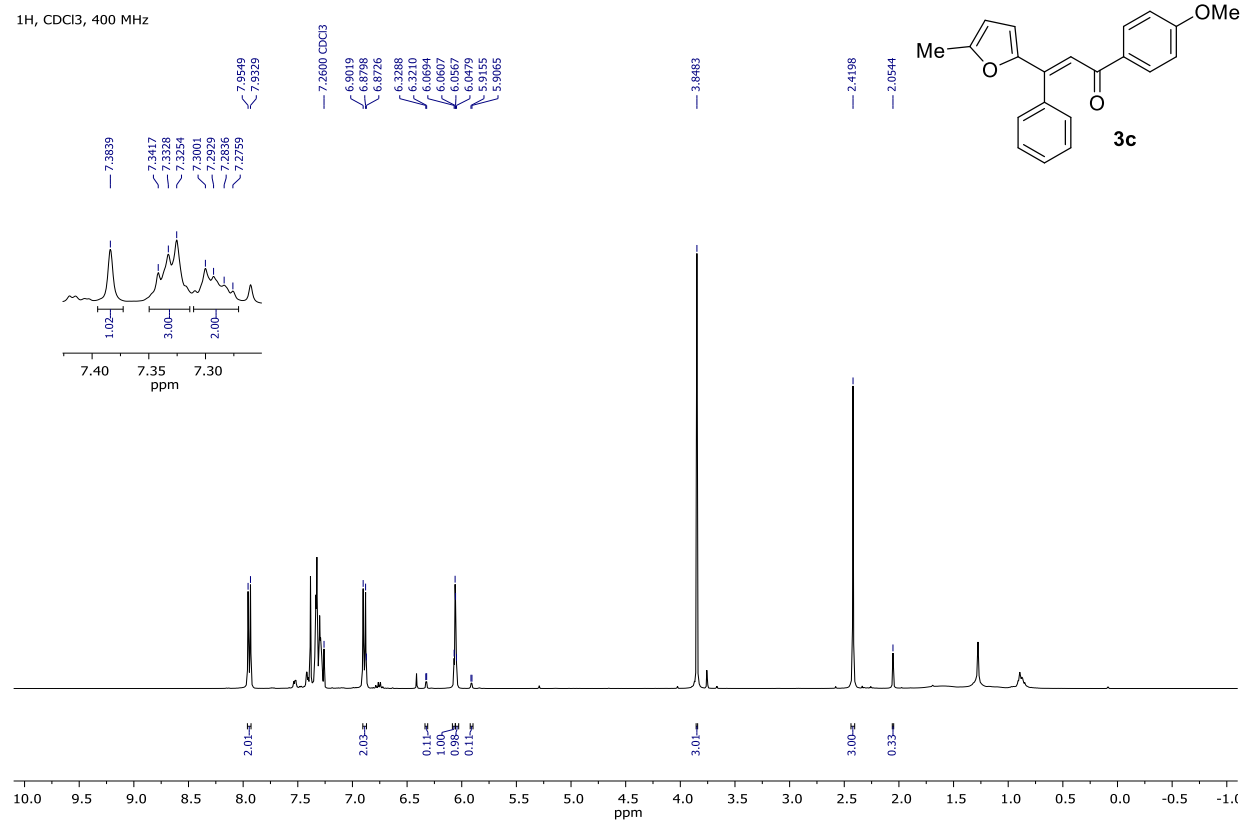
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6.0567
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5.9065
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2.0544
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3.01
3.00
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3.5
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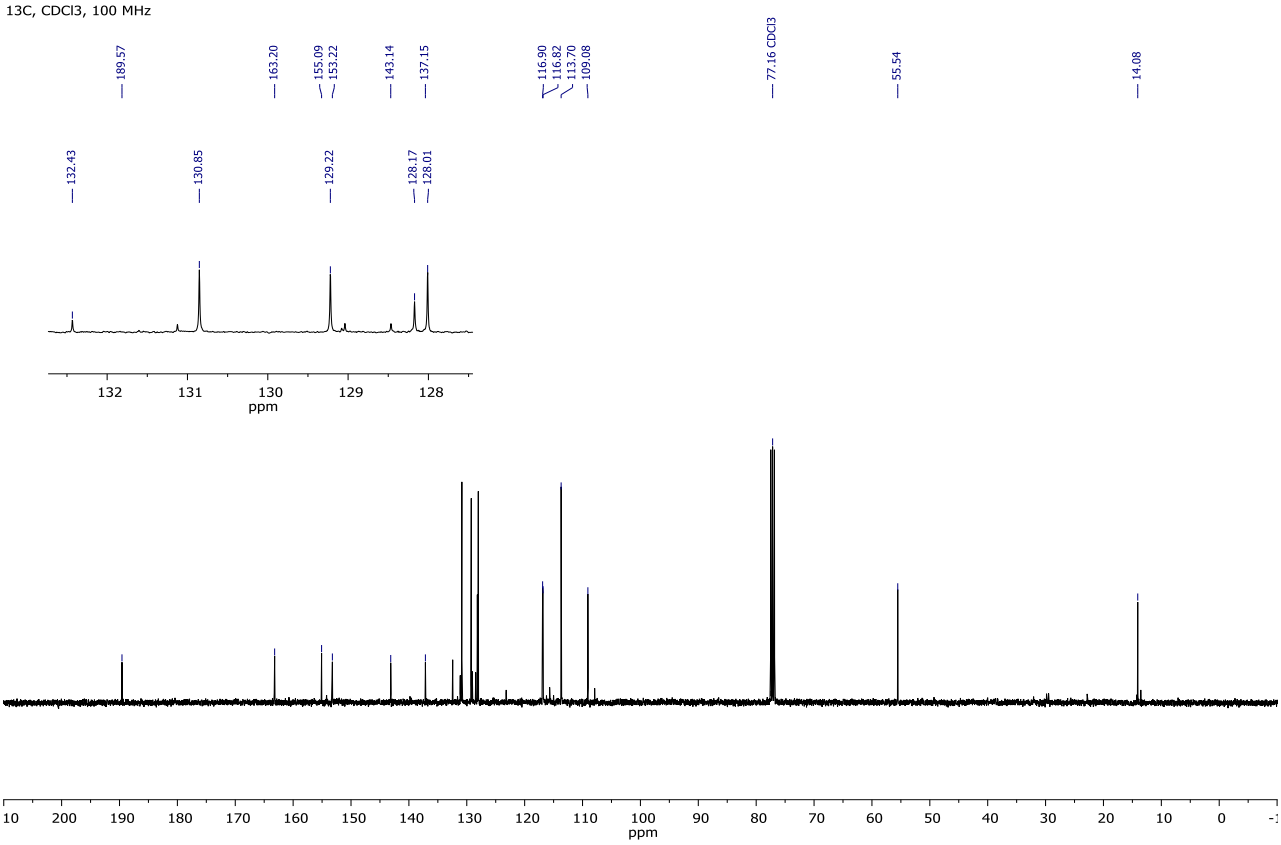
O
OMe
3c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116.82
113.70
109.08
77.16 CDCl3
55.54
14.08
132.43
130.85
129.22
128.17
128.01
132
131
130
129
128
ppm
10
200
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130
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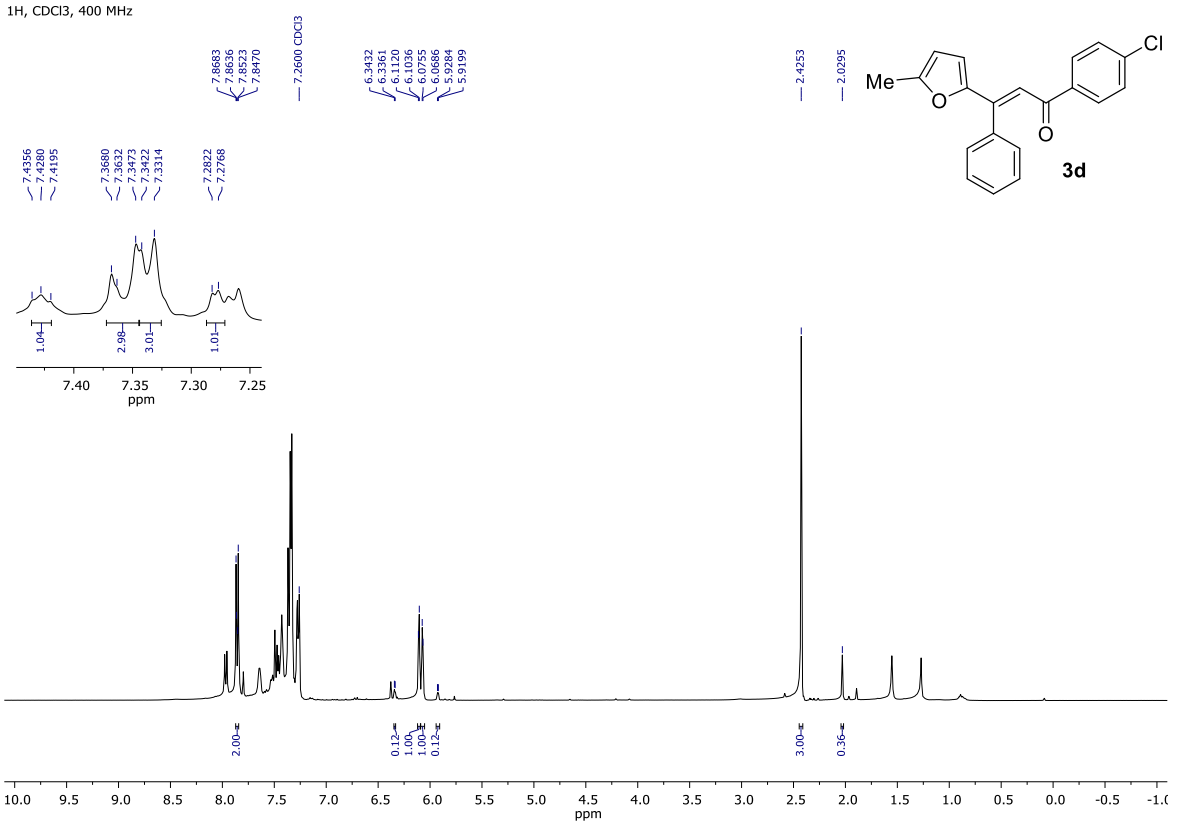
90
80
70
60
50
40
30
20
10
0
ppm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6.0686
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5.9199
2.4253
2.0295
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7.4195
7.3680
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7.25
ppm
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3.00
0.36
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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
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2.0
1.5
1.0
0.5
0.0
-0.5
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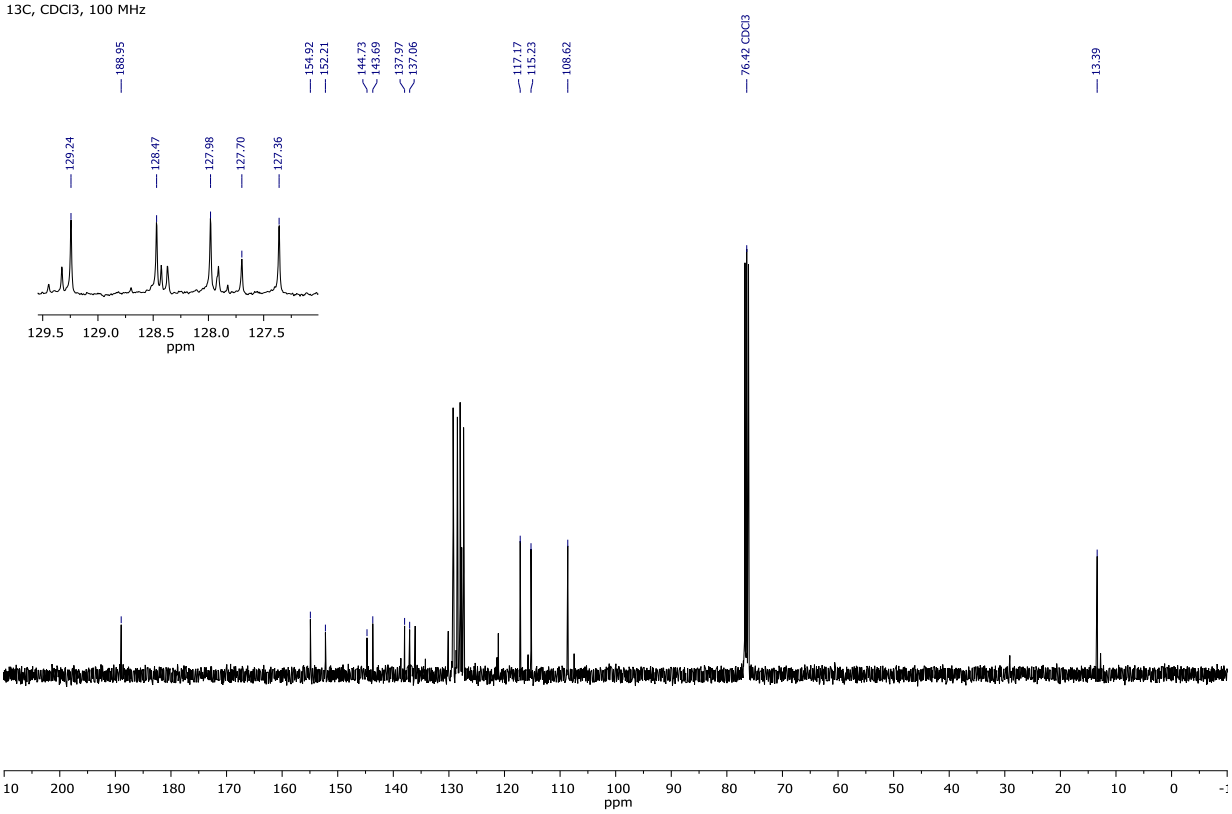
Me
O
O
Cl
3d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137.97
137.06
117.17
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108.62
76.42 CDCl3
13.39
129.24
128.47
127.98
127.70
127.36
129.5
129.0
128.5
128.0
127.5
ppm
10
200
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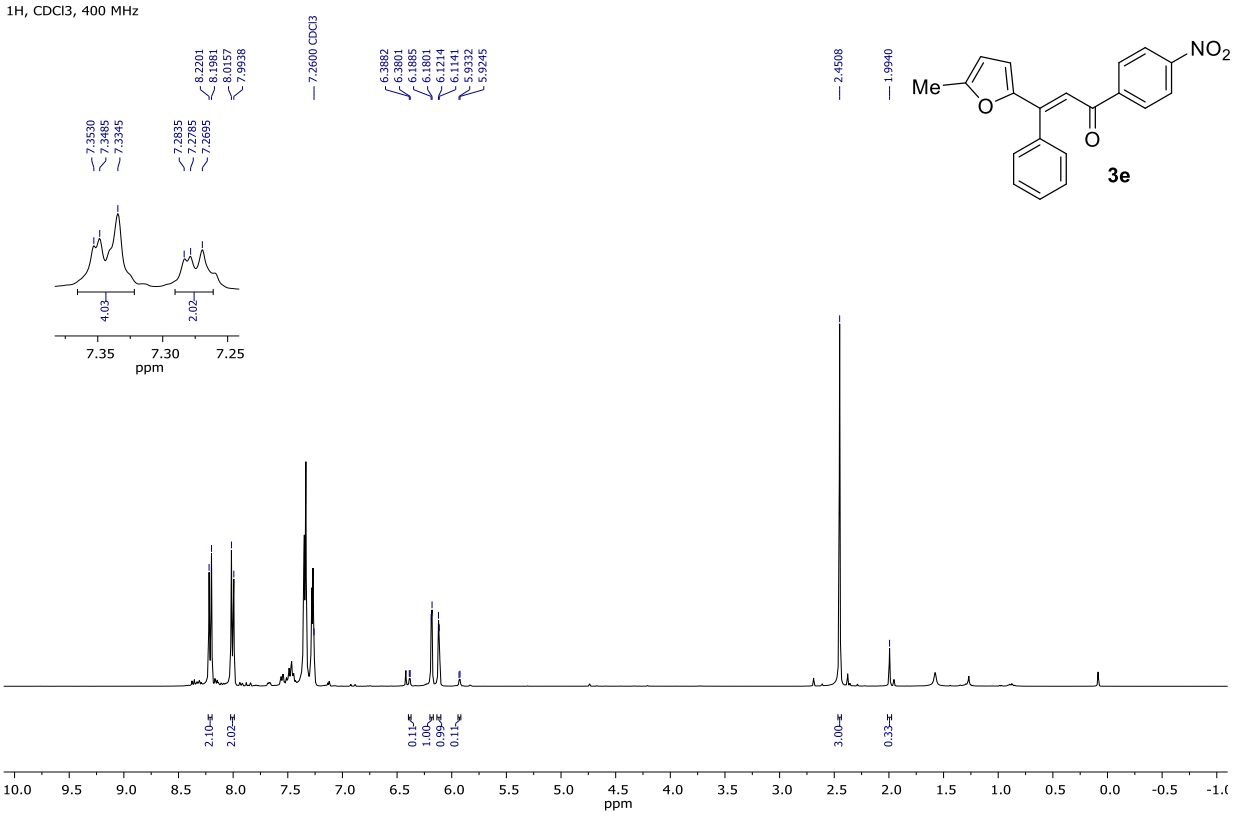
1H, CDCl3, 400 MHz
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8.1961
8.0157
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7.2600 CDCl3
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6.3801
6.1885
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7.2785
7.2695
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2.02
7.35
7.30
7.25
ppm
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2.02
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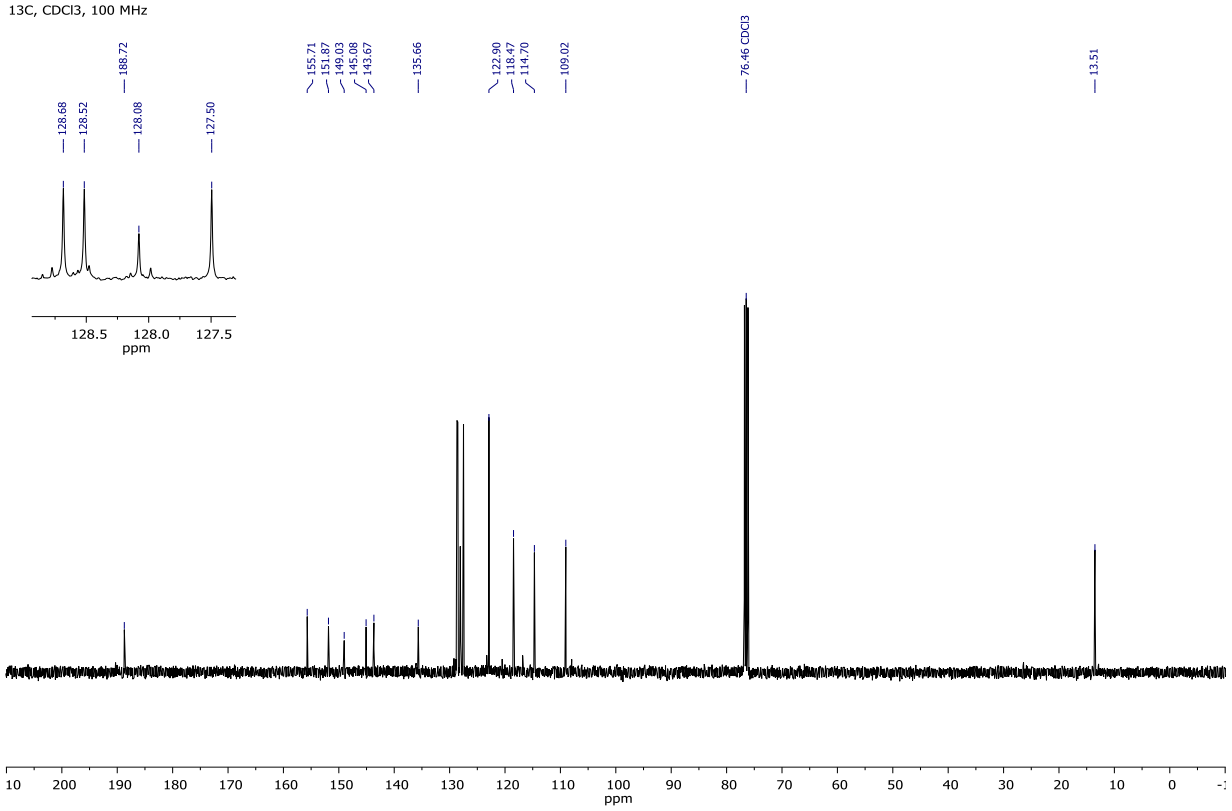
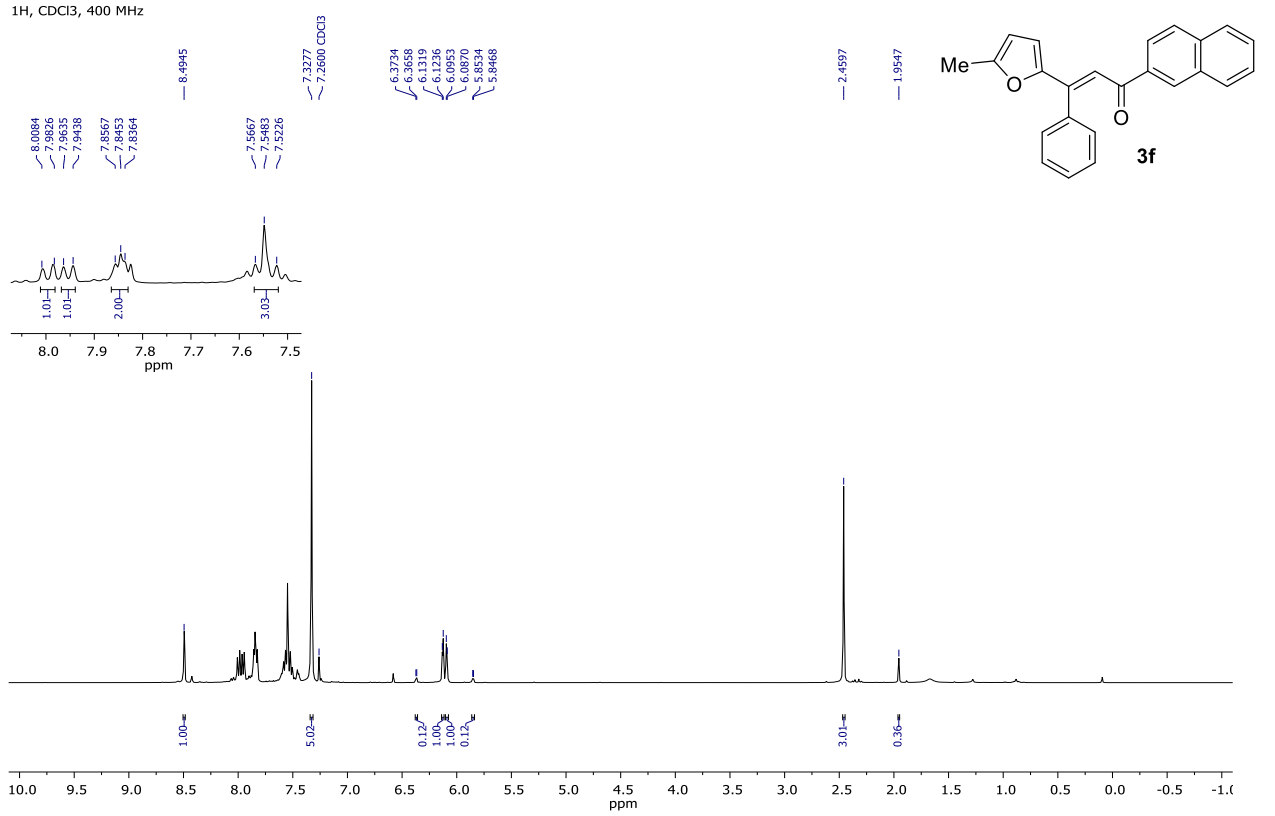
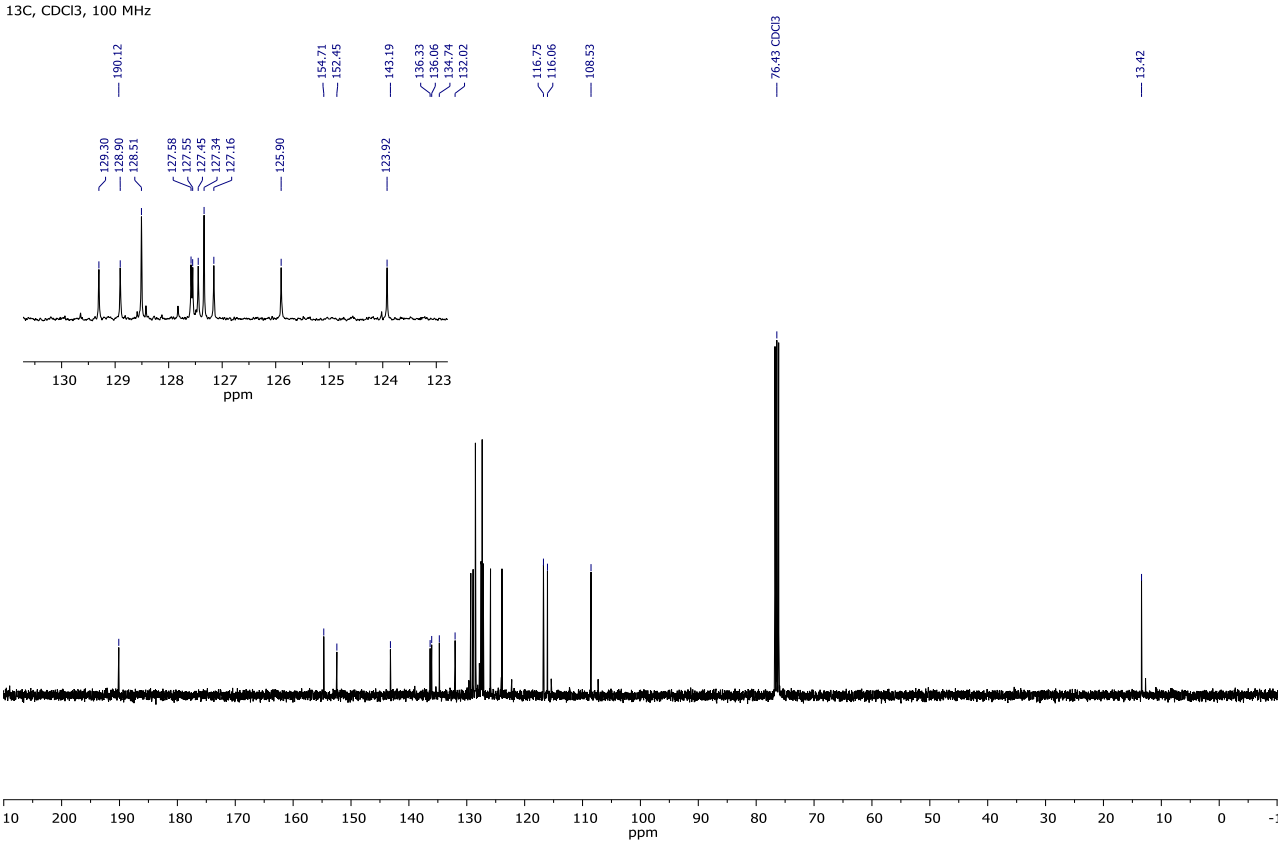
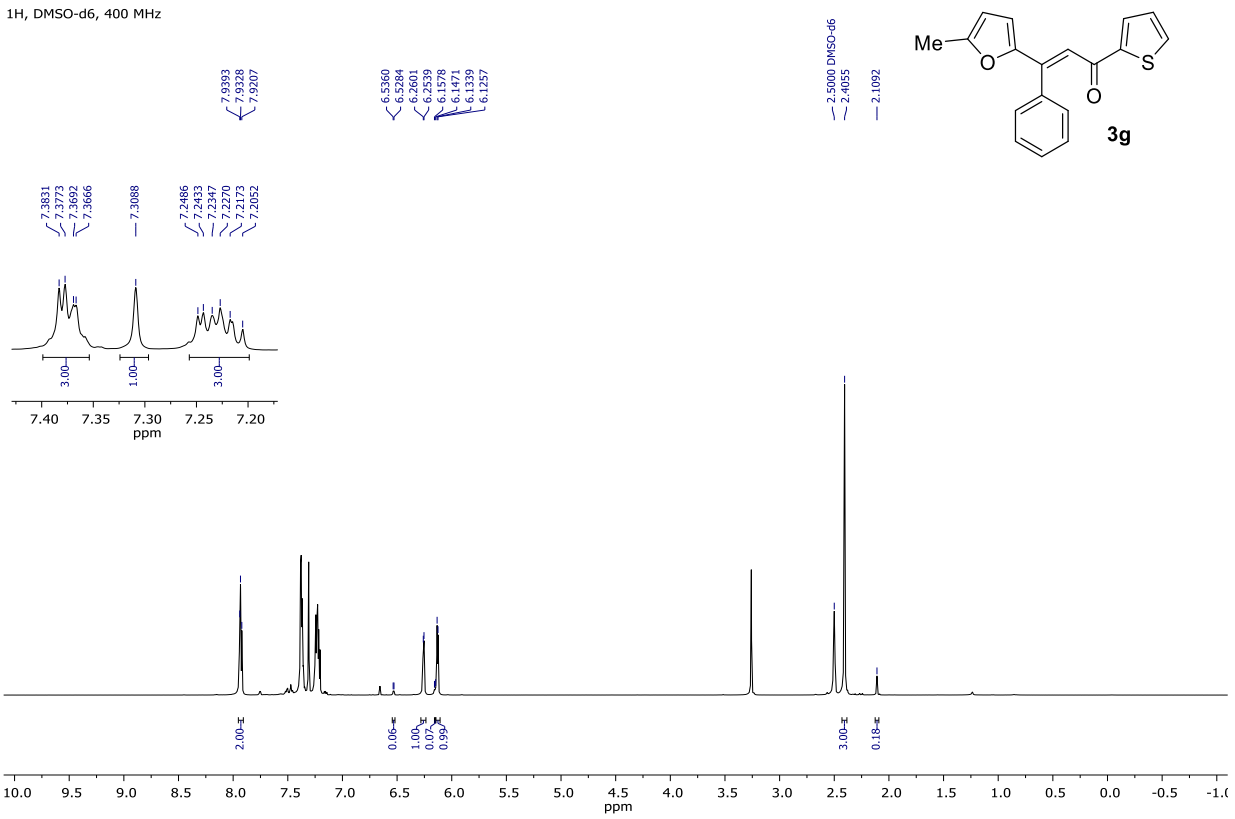
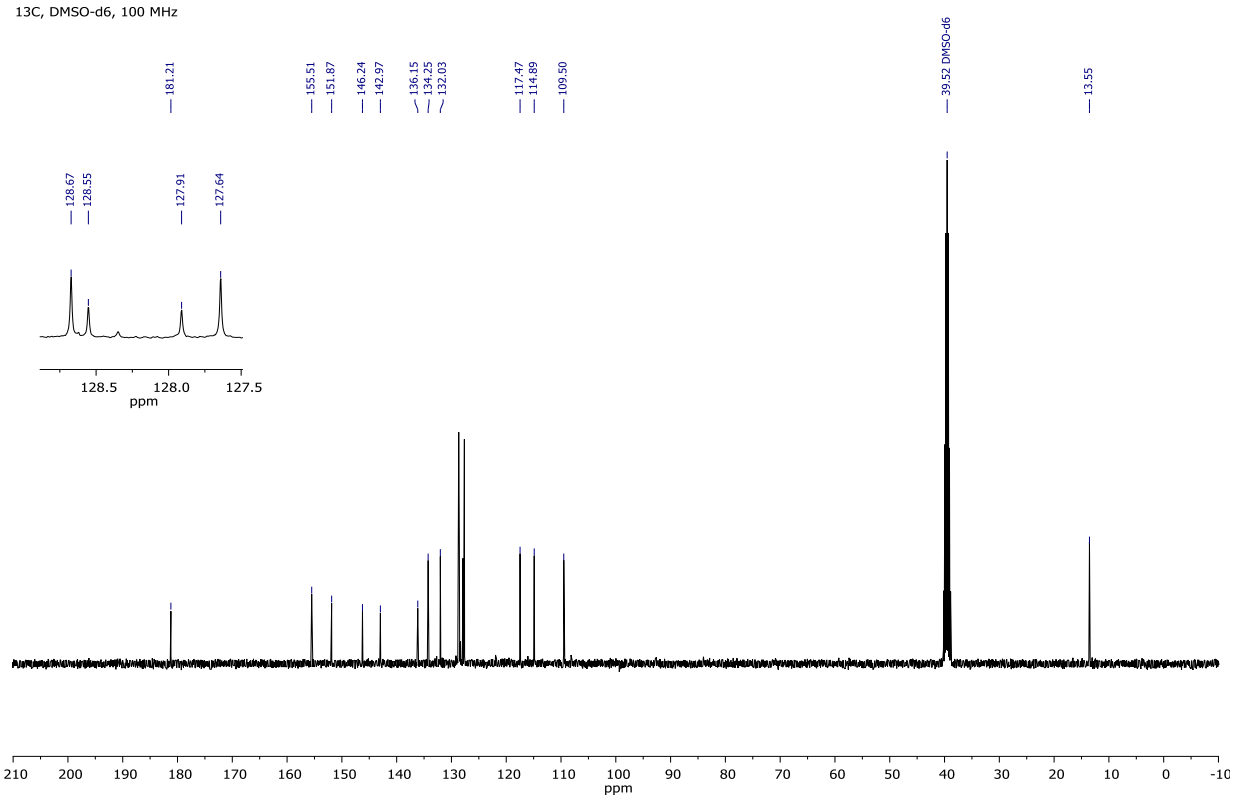
0.11
3.00
0.33
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
Me
O
O
NO2
3e

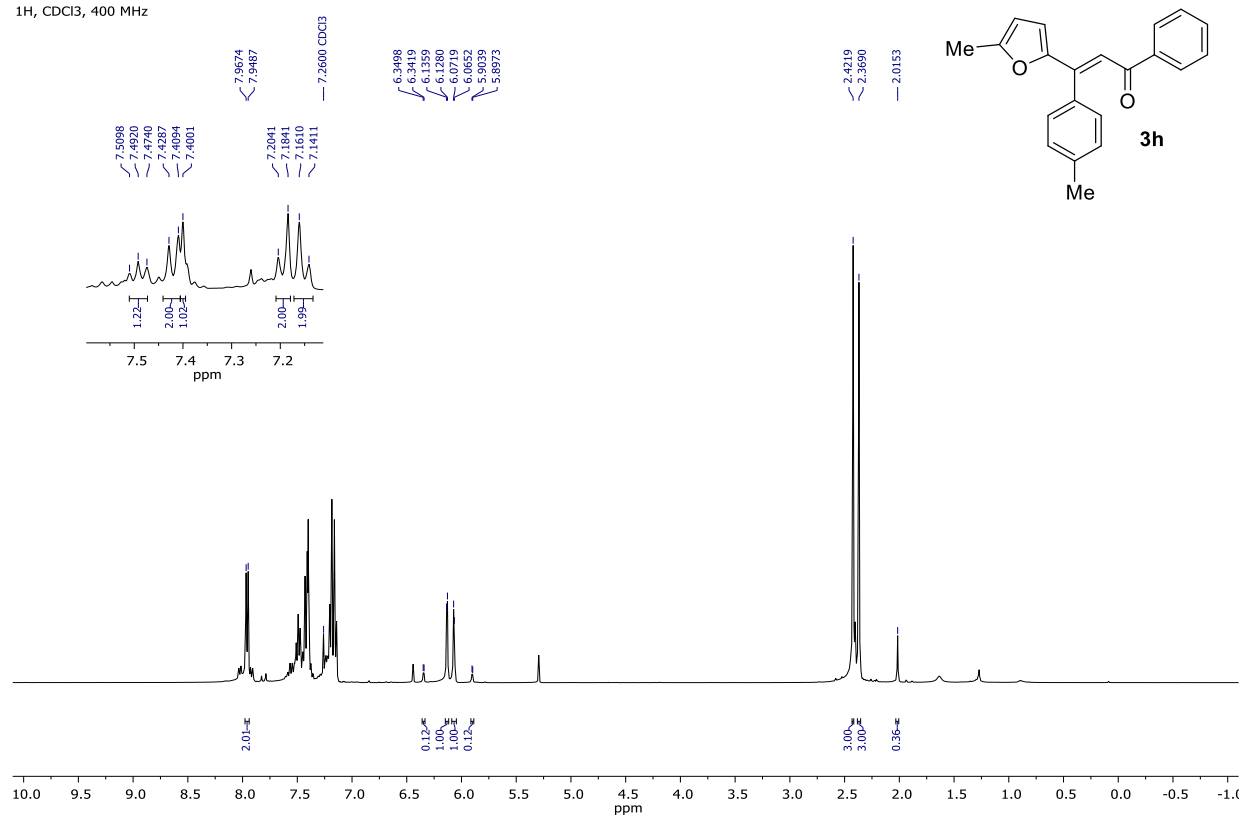

13C, CDCl3, 100 MHz
188.72
155.71
151.87
149.03
145.08
143.67
135.66
122.90
118.47
114.70
109.02
76.46 CDCl3
13.51
128.68
128.52
128.08
127.50
128.5
128.0
127.5
ppm
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

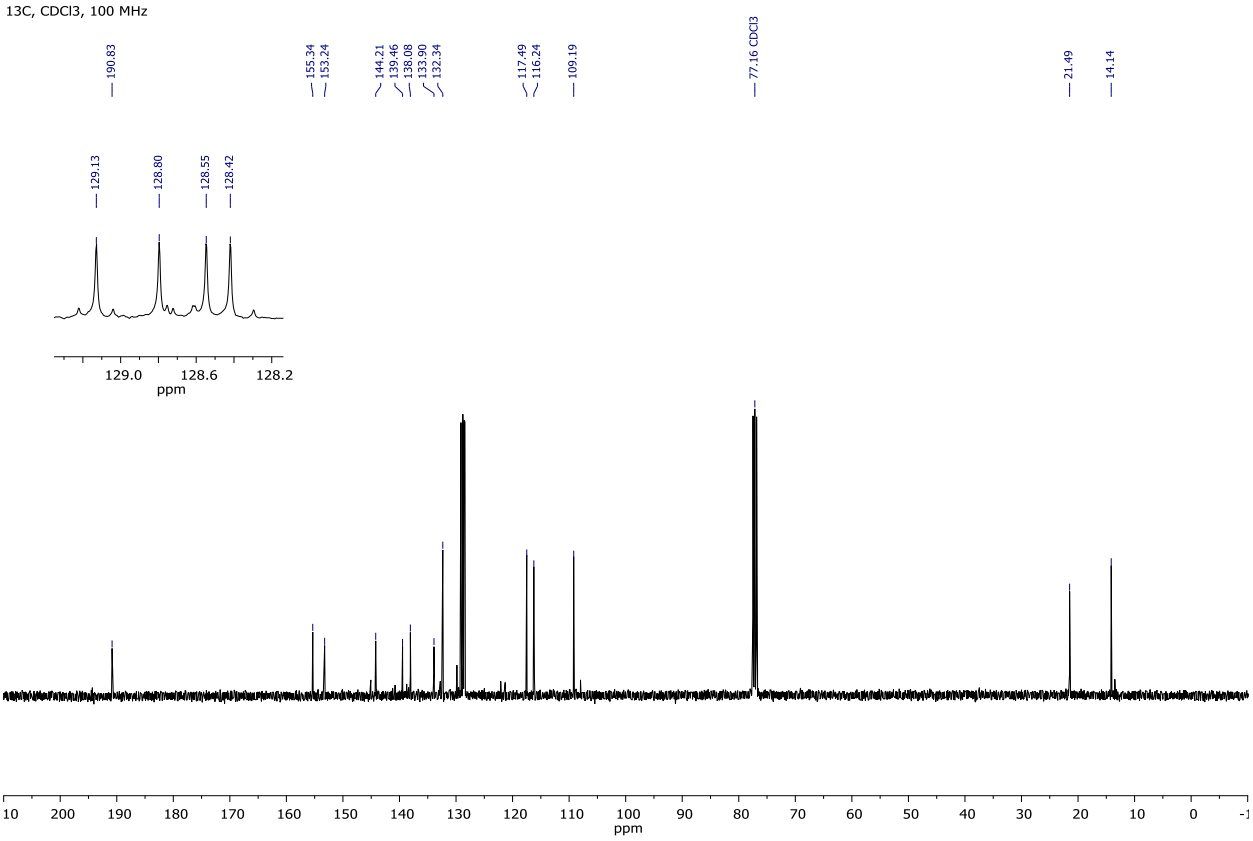

1H, CDCl3, 400 MHz
8.4945
7.3277
7.2600 CDCl3
6.3734
6.3658
6.1319
6.1236
6.0953
6.0870
5.8534
5.8468
2.4597
1.9547
8.0084
7.9826
7.9635
7.9438
7.8567
7.8453
7.8364
7.5667
7.5483
7.5226
Me
O
O
3f
1.01
1.01
2.00
3.03
ppm
1.00
5.02
0.12
1.00
1.00
0.12
3.01
0.36
ppm

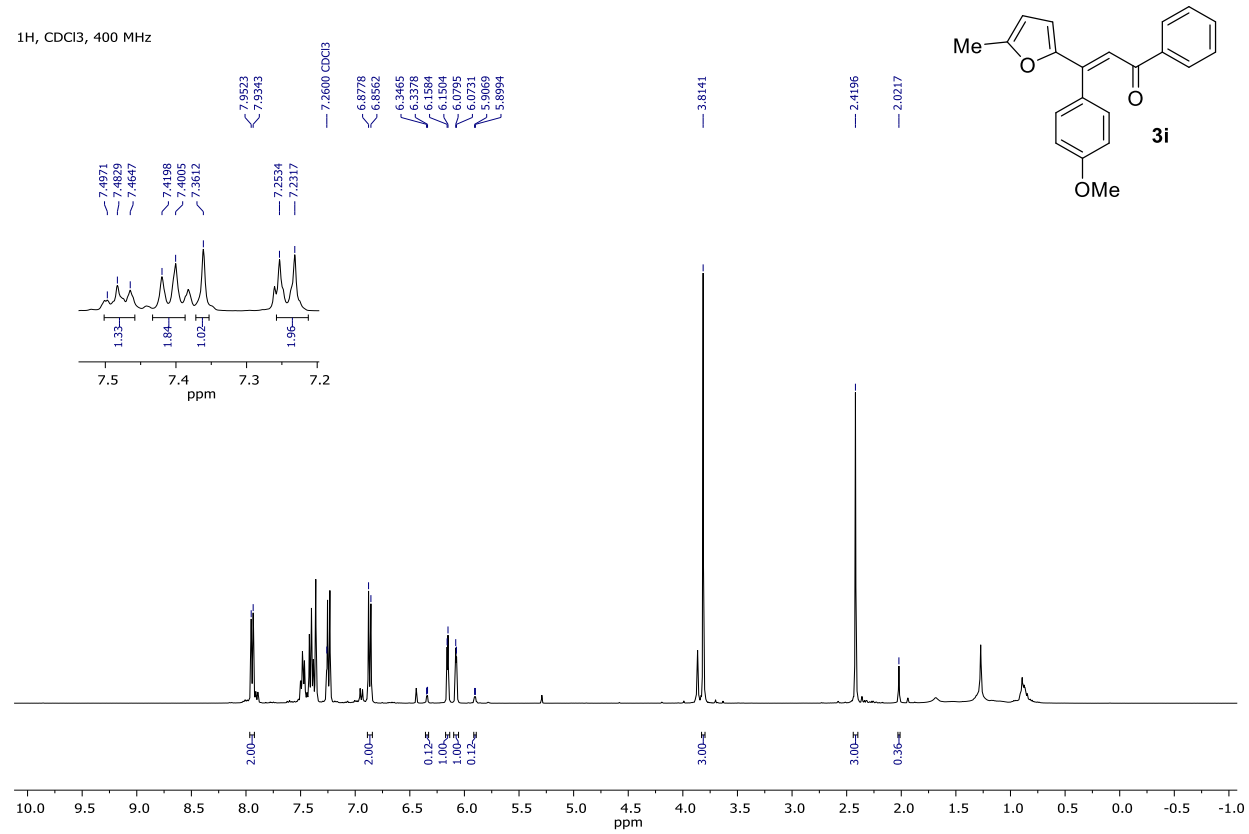

13C, CDCl3, 100 MHz
190.12
154.71
152.45
143.19
136.33
136.06
134.74
132.02
116.75
116.06
108.53
76.43 CDCl3
13.42
129.30
128.90
128.51
127.58
127.55
127.45
127.34
127.16
125.90
123.92
ppm
ppm

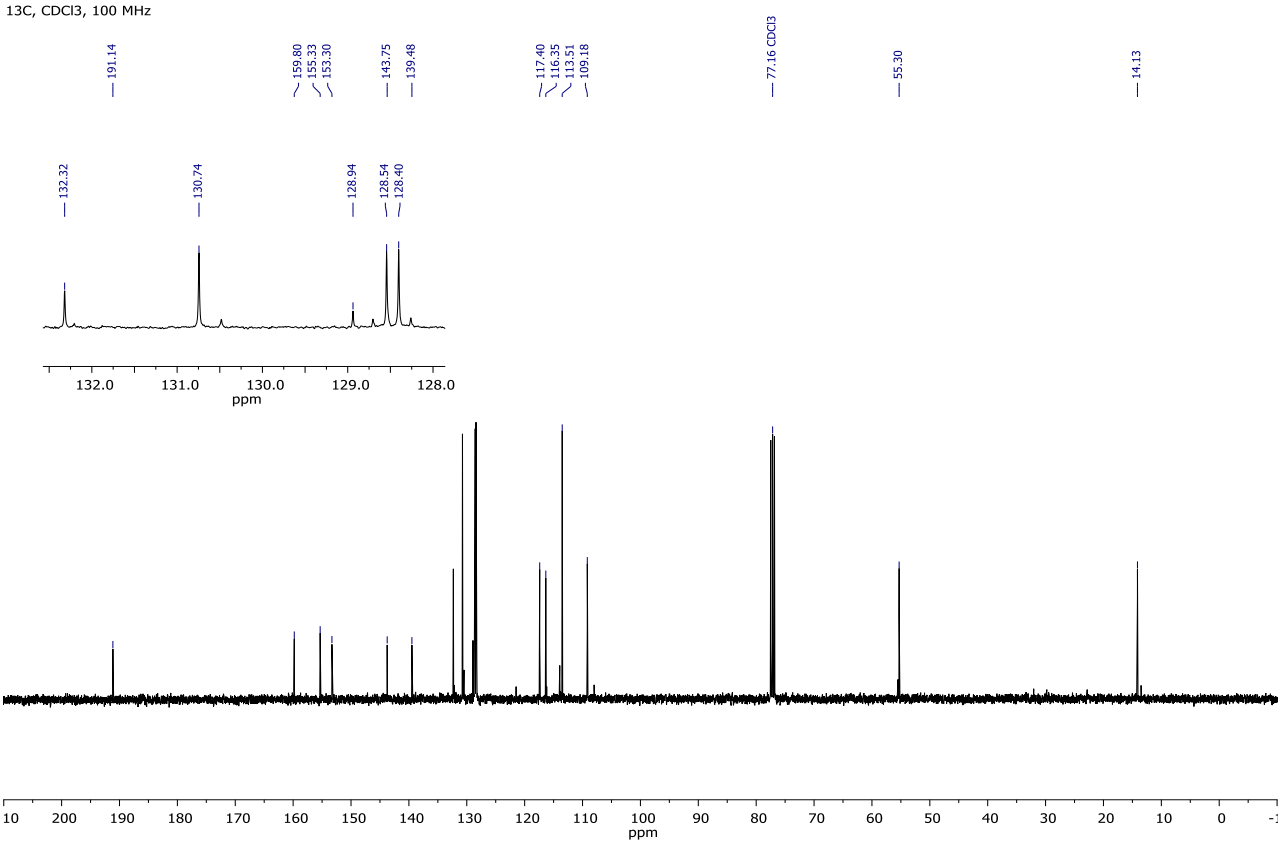

1H, DMSO-d6, 400 MHz
Me
O
S
O
3g
2.5000 DMSO-d6
2.4055
2.1092
ppm

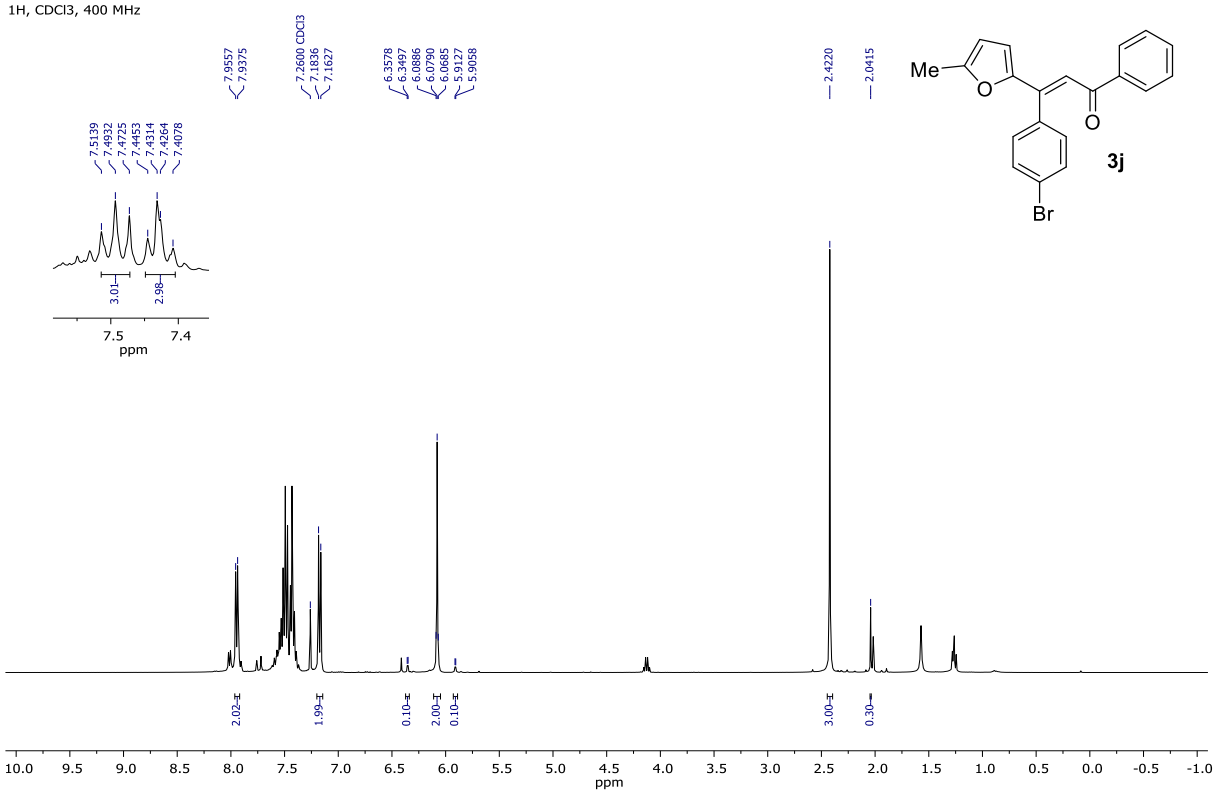

13C, DMSO-d6, 100 MHz
39.52 DMSO-d6
ppm

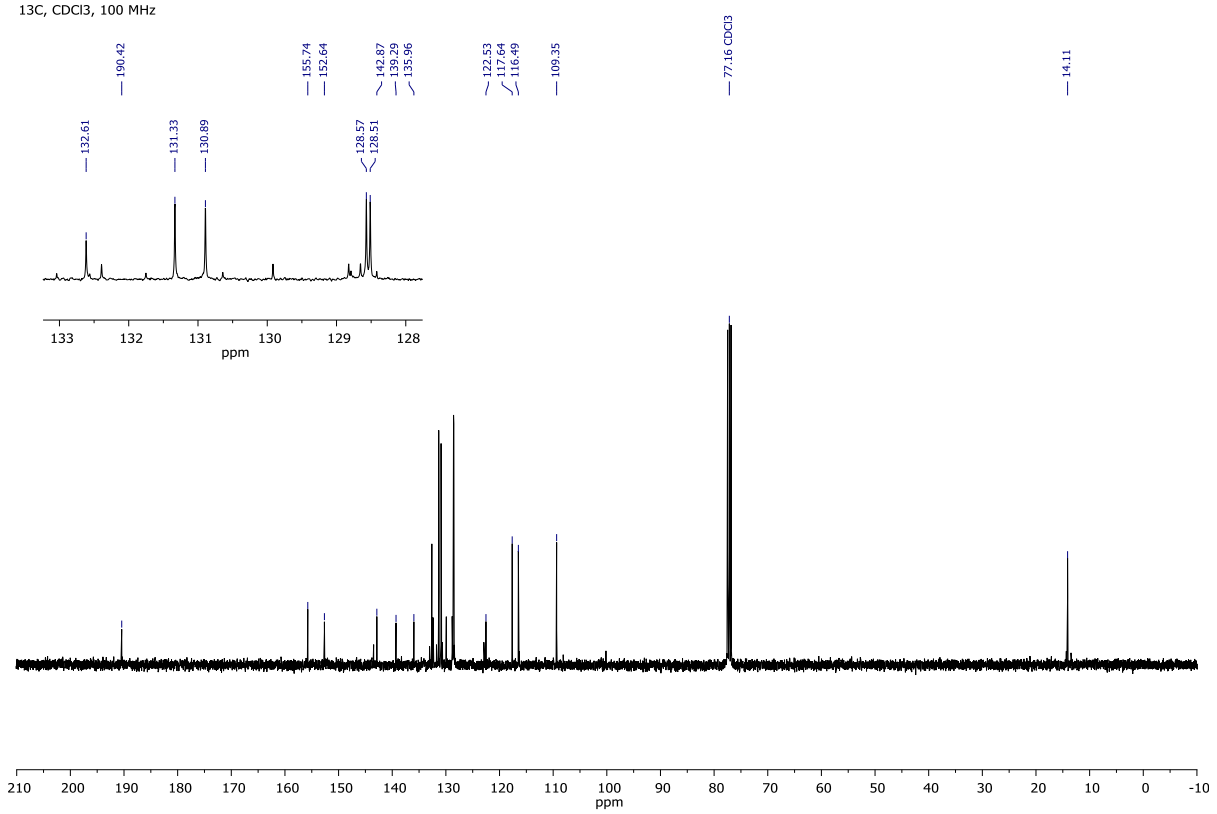

1H, CDCl3, 400 MHz
Me
O
O
Me
3h
ppm

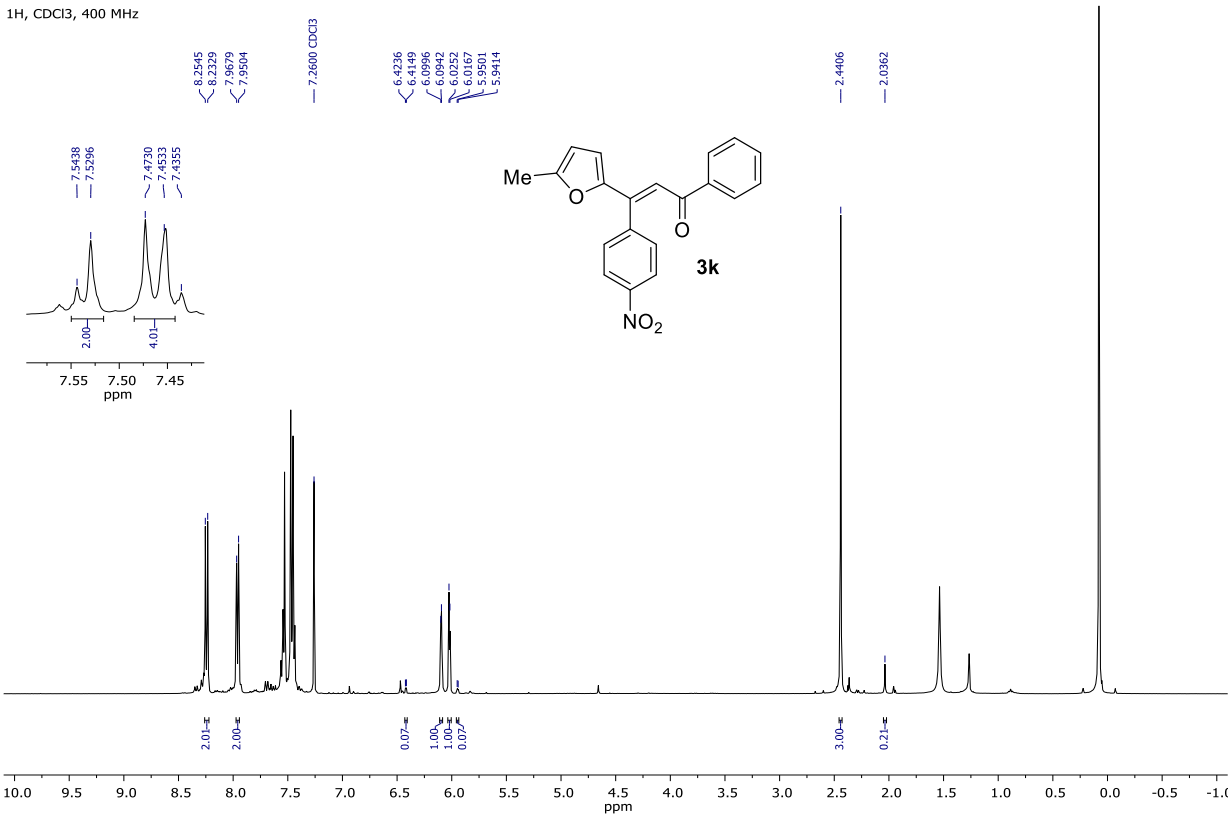

13C, CDCl3, 100 MHz
ppm

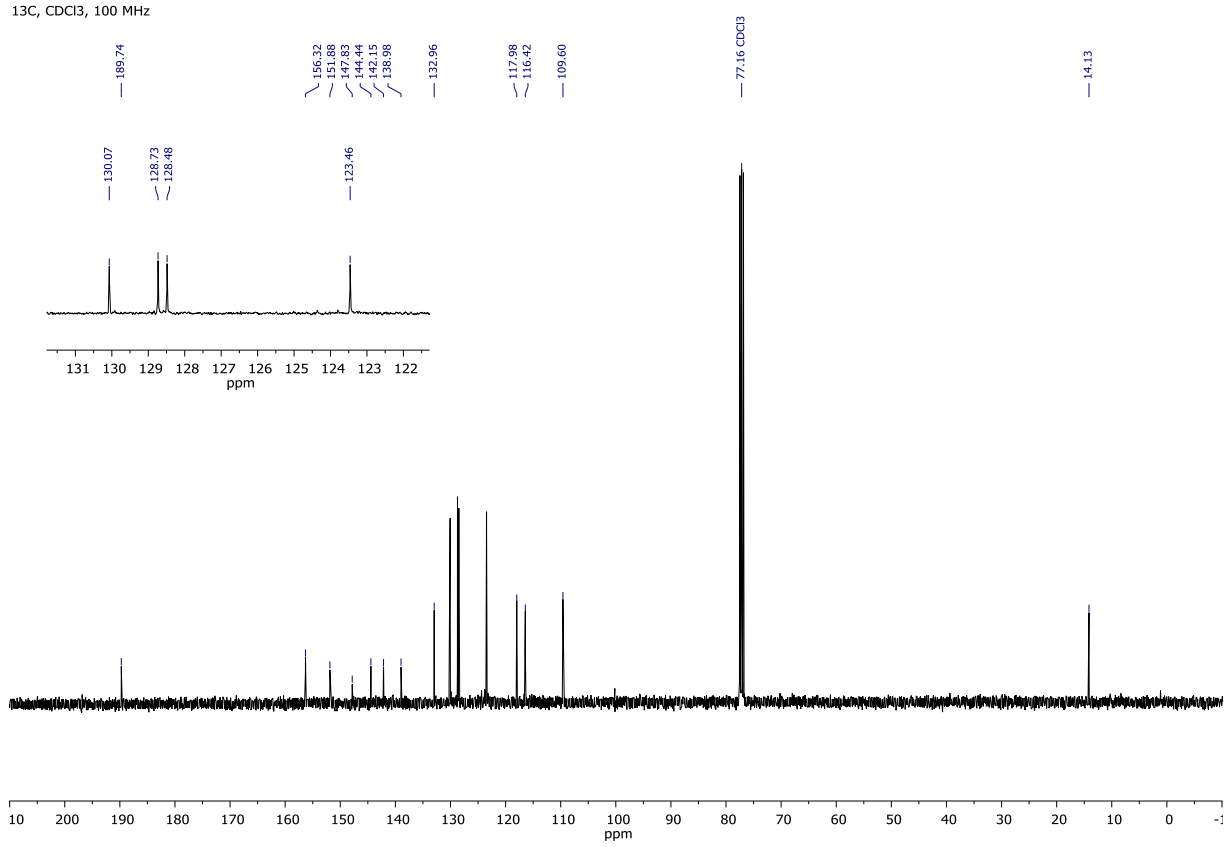

1H, CDCl3, 400 MHz
Me
O
O
OMe
3i
7.9523
7.9343
7.2600 CDCl3
6.8778
6.8562
6.3465
6.3378
6.1584
6.1504
6.0795
6.0731
5.9069
5.8994
3.8141
2.4196
2.0217
7.4971
7.4829
7.4647
7.4198
7.4005
7.3612
7.2534
7.2317
1.33
1.84
1.02
1.96
2.00
2.00
0.12
1.00
1.00
0.12
3.00
3.00
0.36
ppm

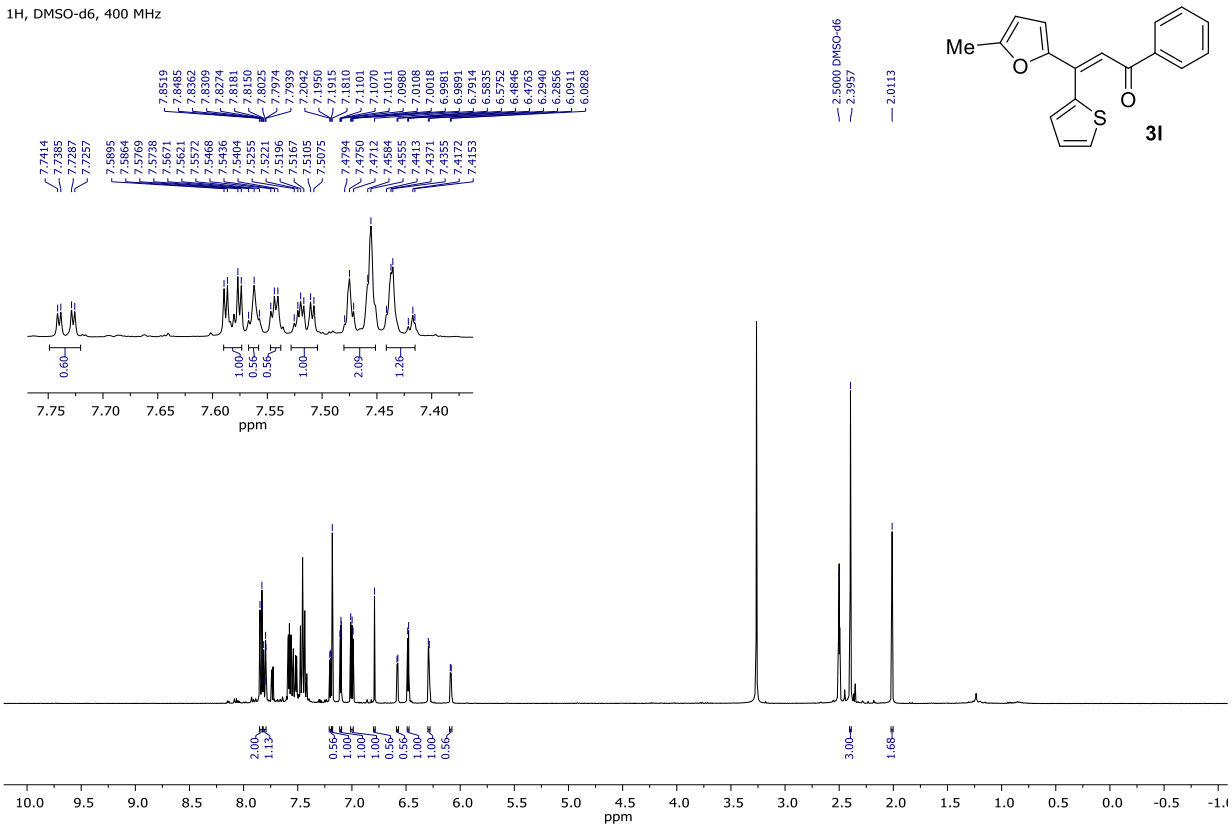

13C, CDCl3, 100 MHz
191.14
159.80
155.33
153.30
143.75
139.48
117.40
116.35
113.51
109.18
77.16 CDCl3
55.30
14.13
132.32
130.74
128.94
128.54
128.40
ppm

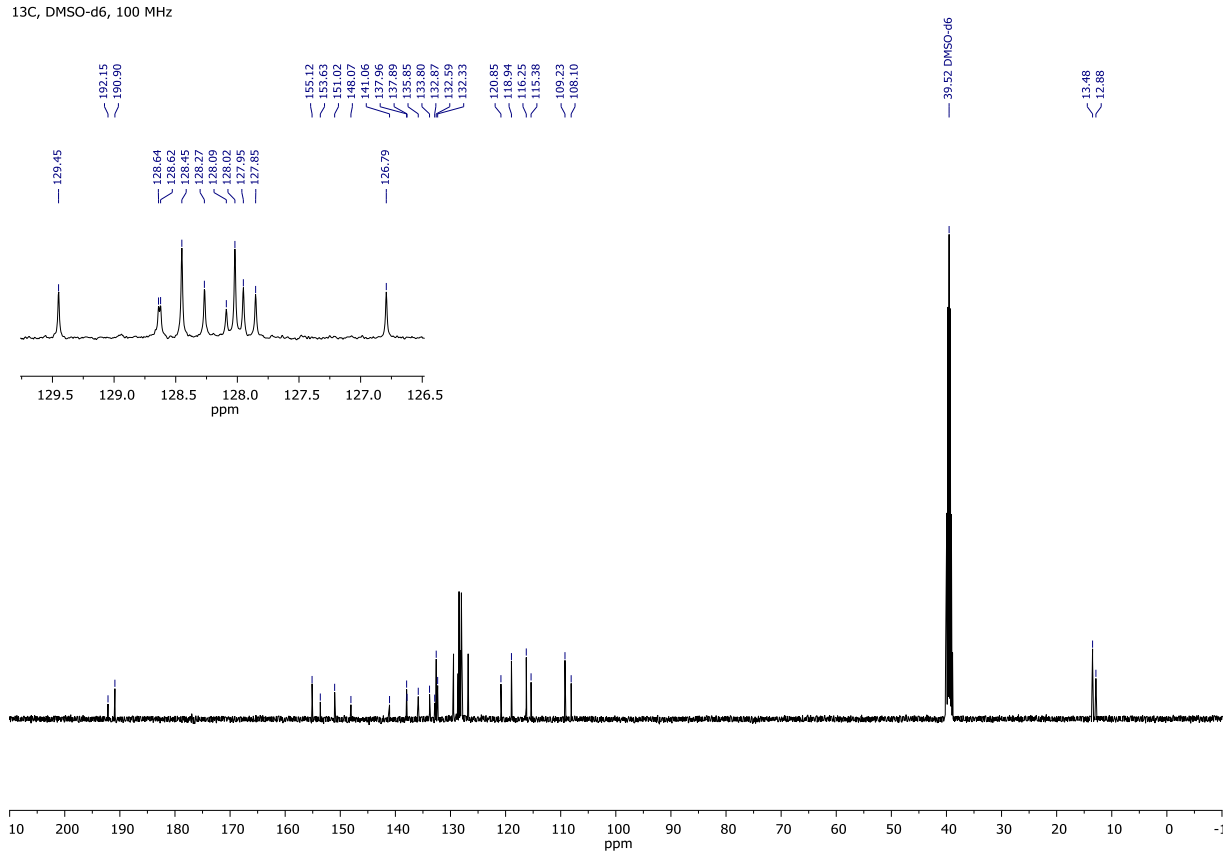

1H, CDCl3, 400 MHz
7.9557
7.9375
7.7600 CDCl3
7.1836
7.1627
6.3578
6.3497
6.0886
6.0790
6.0685
5.9127
5.9058
2.4220
2.0415
7.5139
7.4932
7.4725
7.4453
7.4314
7.4264
7.4078
3.01
2.98
7.5
7.4
ppm
Me
O
O
Br
3j
2.02
1.99
0.10
2.00
0.10
3.00
0.30
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
ppm

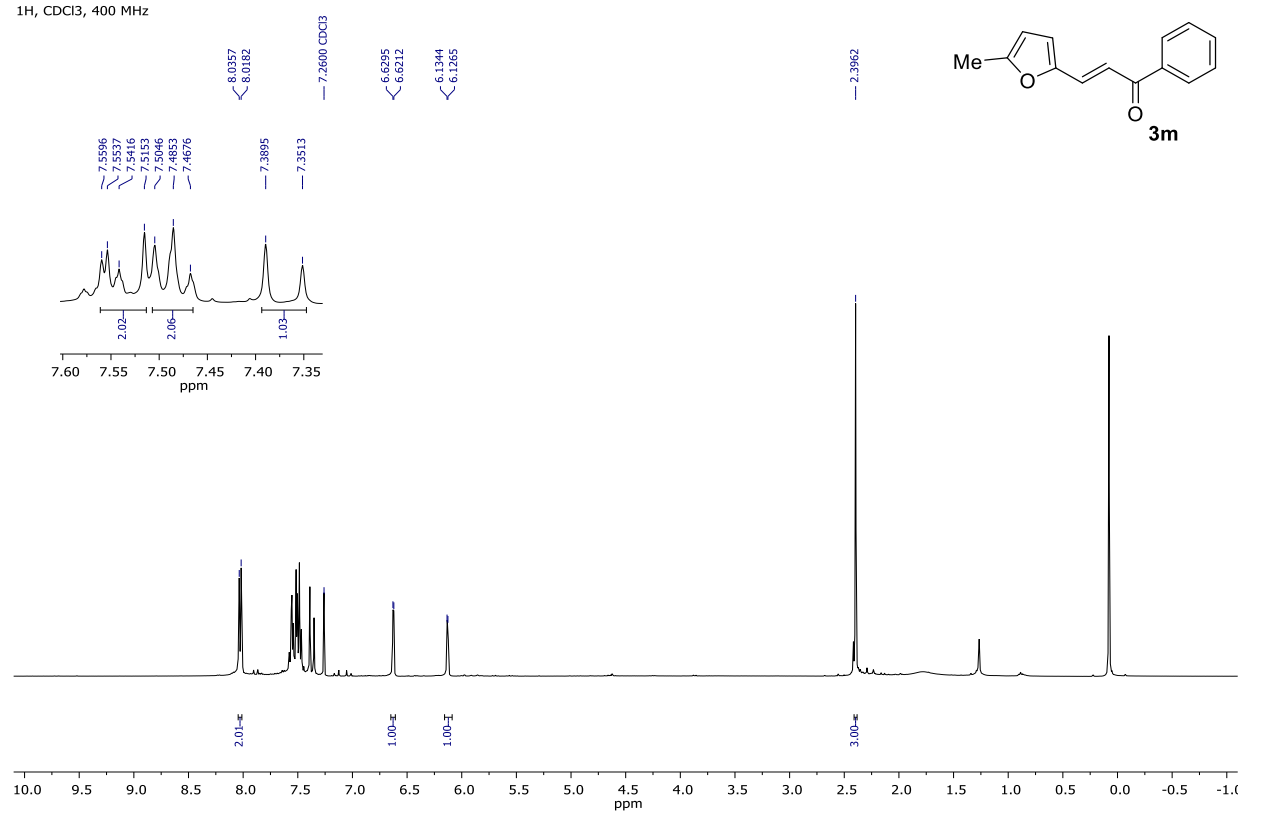

13C, CDCl3, 100 MHz
190.42
155.74
152.64
142.87
139.29
135.96
122.53
117.64
116.49
109.35
77.16 CDCl3
14.11
132.61
131.33
130.89
128.57
128.51
133 132 131 130 129 128
ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm

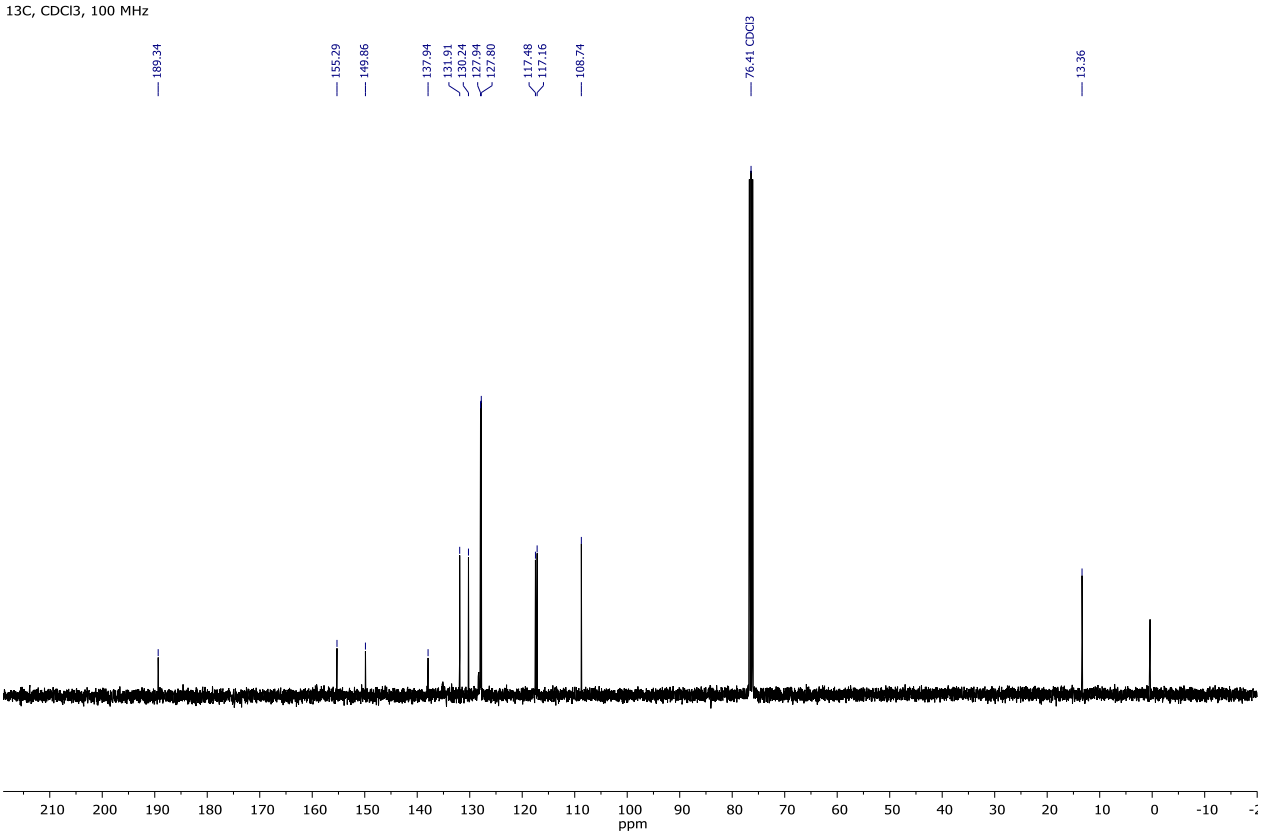

1H, CDCl3, 400 MHz
8.2545
8.2329
7.9679
7.9504
7.2600 CDCl3
6.4236
6.4149
6.0996
6.0942
6.0252
6.0167
5.9501
5.9414
2.4406
2.0362
7.5438
7.5296
7.4730
7.4533
7.4355
2.00
4.01
7.55 7.50 7.45
ppm
Me
O
O
NO2
3k
2.01
2.00
0.07
1.00
1.00
0.07
3.00
0.21
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
ppm


13C, CDCl3, 100 MHz
189.74
156.32
151.88
147.83
144.44
142.15
138.98
132.96
117.98
116.42
109.60
77.16 CDCl3
14.13
130.07
128.73
128.48
123.46
131 130 129 128 127 126 125 124 123 122
ppm
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

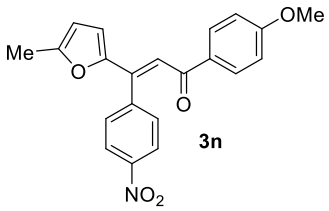

1H, DMSO-d6, 400 MHz
Me
O
O
S
3l
ppm


13C, DMSO-d6, 100 MHz
ppm

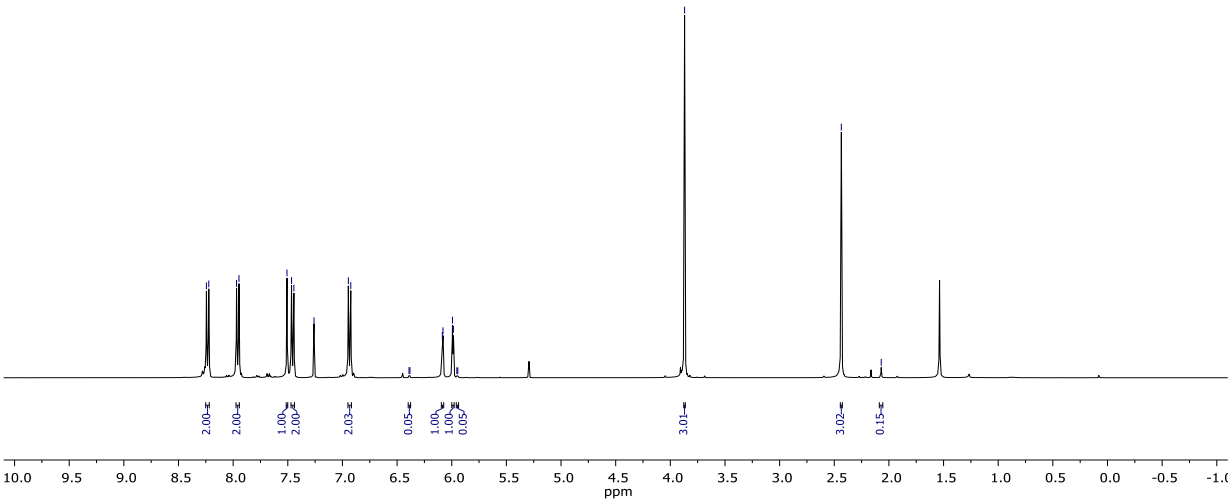

1H, CDCl3, 400 MHz
8.0357
8.0182
7.2600 CDCl3
6.6295
6.6212
6.1344
6.1265
2.3962
7.5596
7.5537
7.5416
7.5153
7.5046
7.4853
7.4676
7.3895
7.3513
2.02
2.06
1.03
7.60 7.55 7.50 7.45 7.40 7.35
ppm
Me
O
O
3m
2.01
1.00
1.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
ppm


13C, CDCl3, 100 MHz
189.34
155.29
149.86
137.94
131.91
130.24
127.94
127.80
117.48
117.16
108.74
76.41 CDCl3
13.36
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm

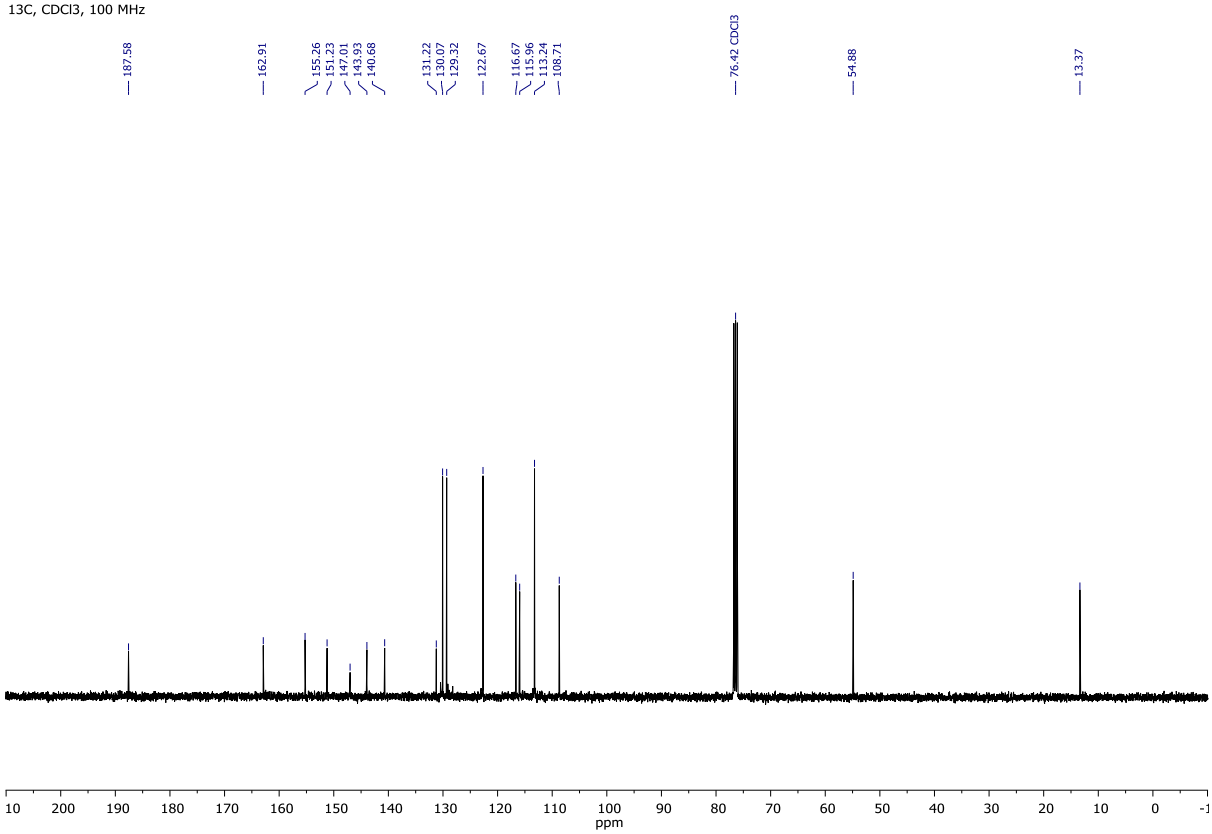
1H, CDCl3, 400 MHz



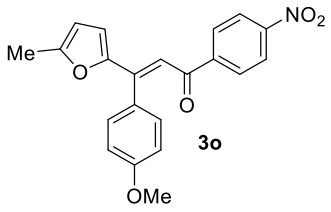
3n



13C, CDCl3, 100 MHz

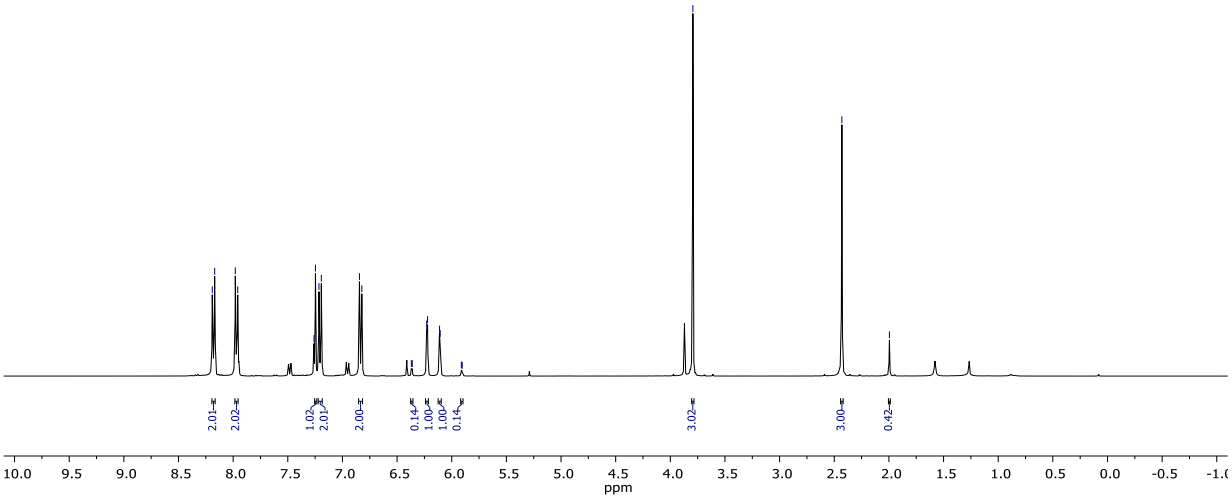


1H, CDCl3, 400 MHz



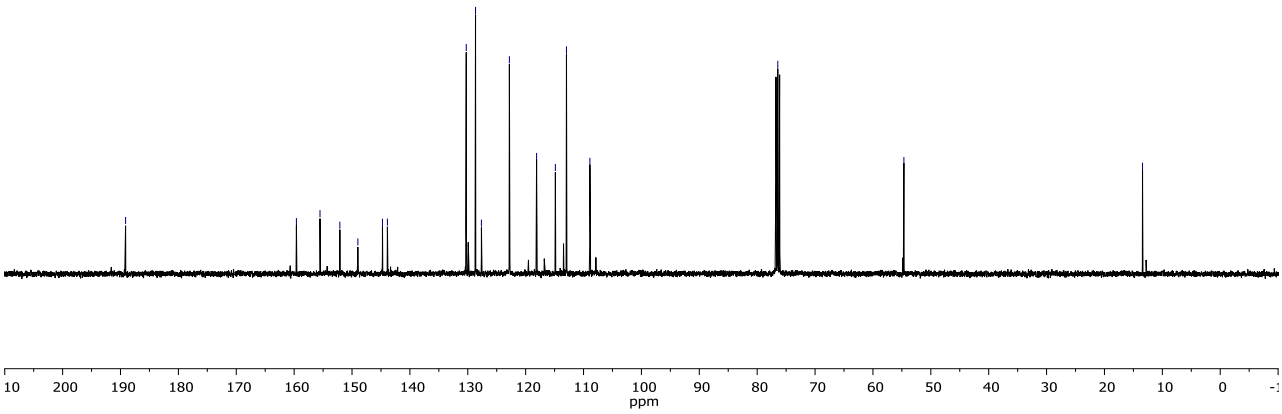
3o

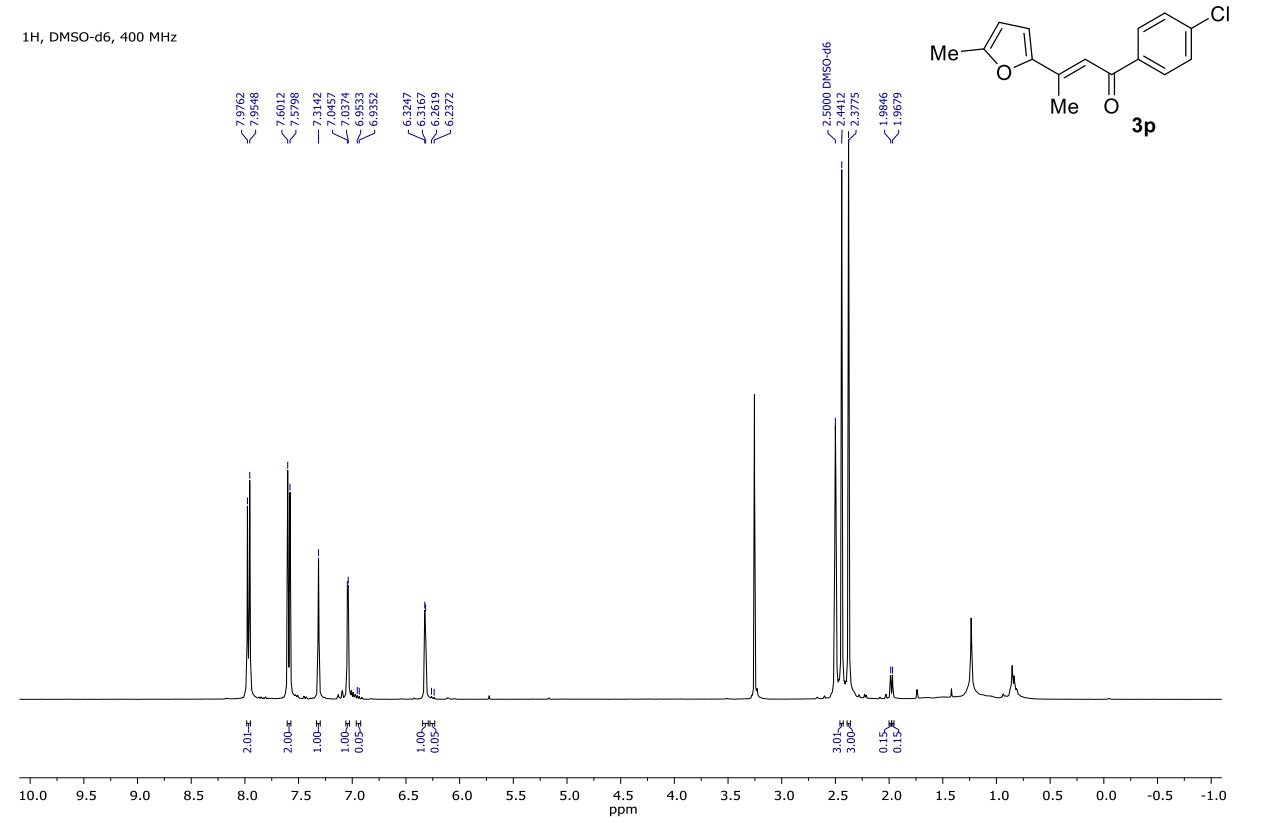
8.1910, 8.1691, 7.9802, 7.9582, 7.2600 CDCl3, 7.2470, 7.2149, 7.1932, 6.8445, 6.8228, 6.3694, 6.3613, 6.2295, 6.2211, 6.1115, 6.1059, 5.9120, 5.9043, 3.7935, 2.4303, 1.9954

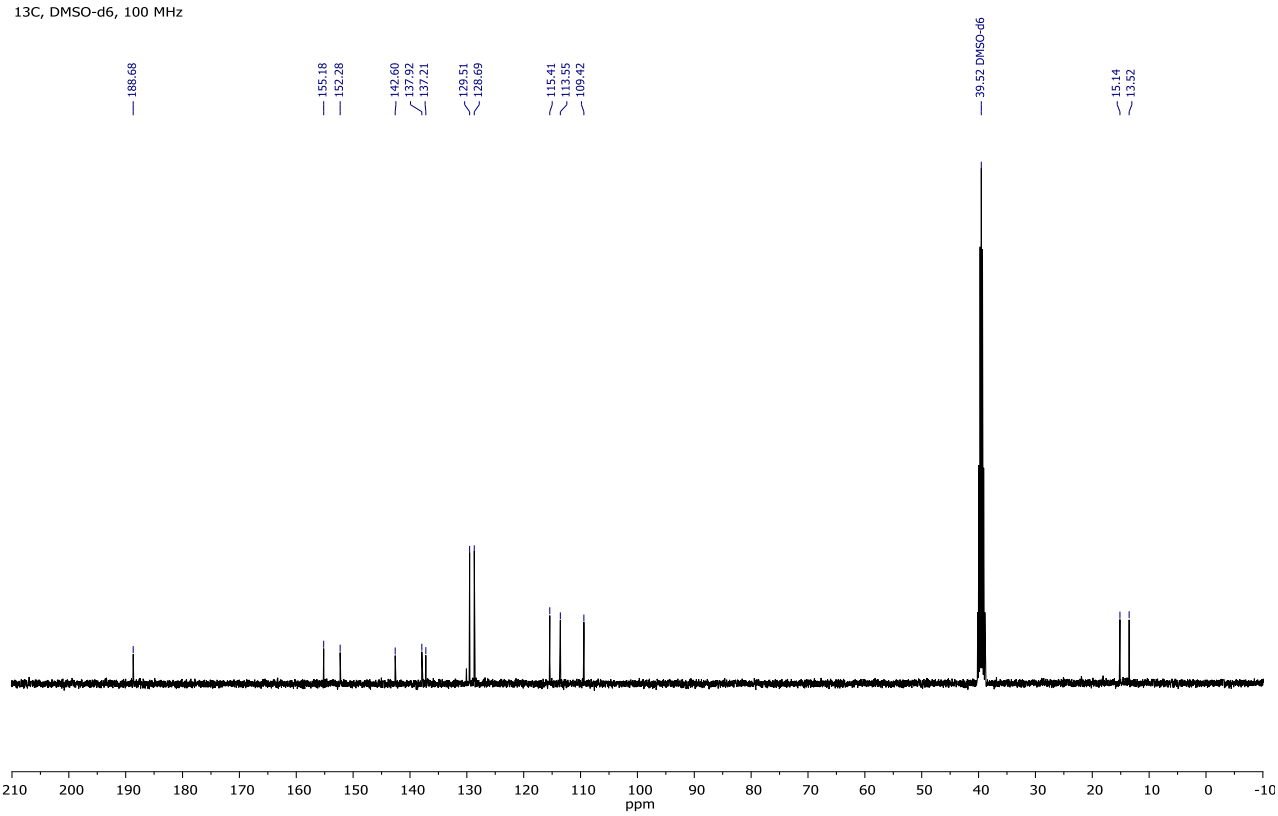


13C, CDCl3, 100 MHz

189.12, 159.61, 155.53, 152.10, 148.99, 144.74, 143.88, 130.26, 128.66, 127.64, 122.81, 118.11, 114.87, 112.95, 108.90, 76.44 CDCl3, 54.65, 13.42




1H, DMSO-d6, 400 MHz
Me
O
Me
O
Cl
3p
7.9762
7.9548
7.6012
7.5798
7.3142
7.0457
7.0374
6.9533
6.9352
6.3247
6.3167
6.2619
6.2372
2.5000 DMSO-d6
2.4412
2.3775
1.9846
1.9679
2.01
2.00
1.00
1.00
0.05
1.00
0.05
3.01
3.00
0.15
0.15
ppm


13C, DMSO-d6, 100 MHz
188.68
155.18
152.28
142.60
137.92
137.21
129.51
128.69
115.41
113.55
109.42
39.52 DMSO-d6
15.14
13.52
ppm