Synthesis

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Supporting Information

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Supporting Information

Efficient Access to Isoquinolines via Rhodium-Catalyzed Oxidative Annulation of Pyridyl C-H Bonds Directed by the Carbonyl with Internal Alkyne

Lijun Shi[ab], Mingshan Wen[a], Fuwei Li[a]*

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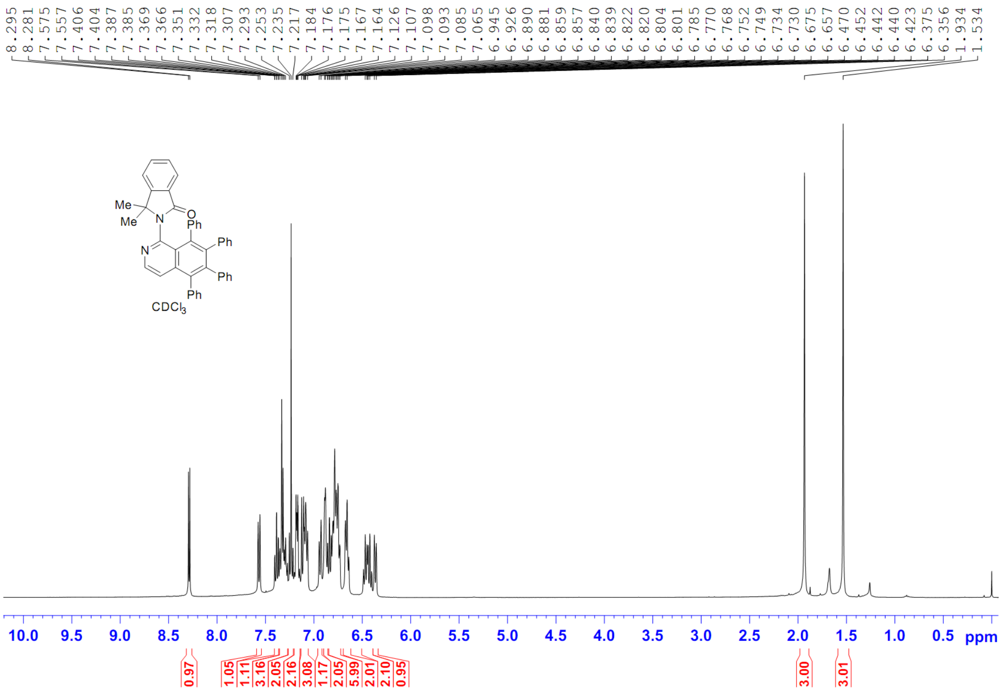
[b] University of the Chinese Academy of Sciences, Beijing 100049, China.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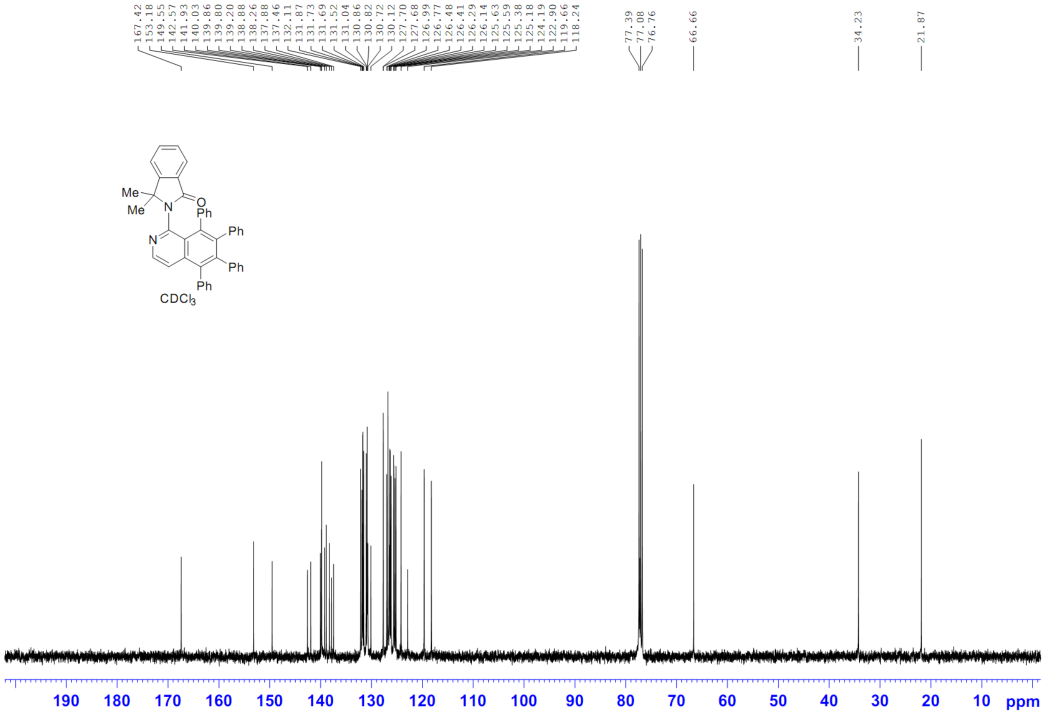
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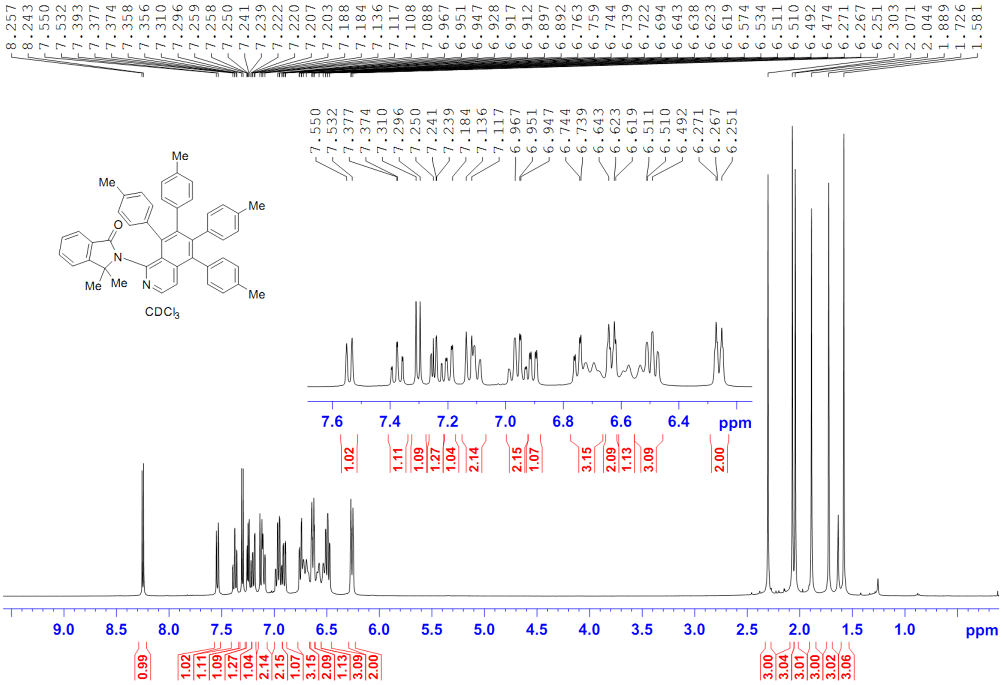
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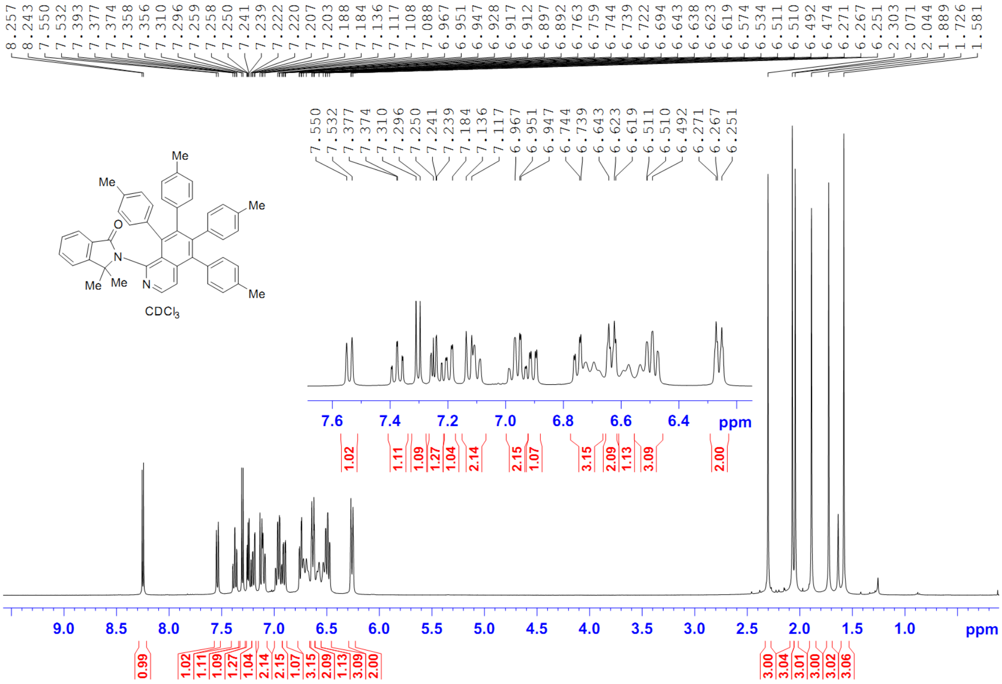
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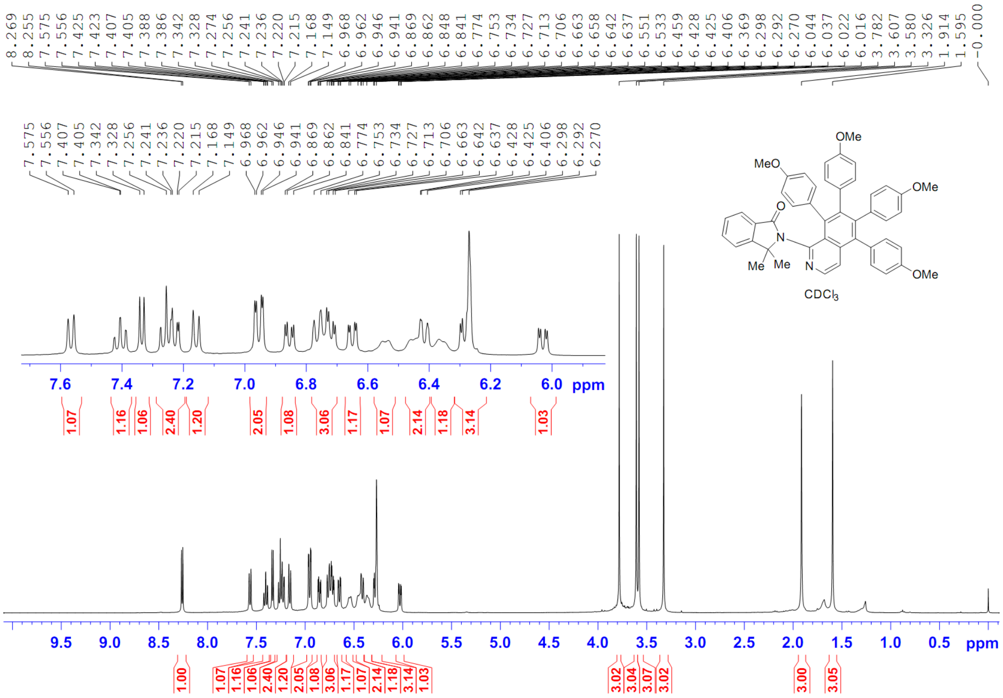
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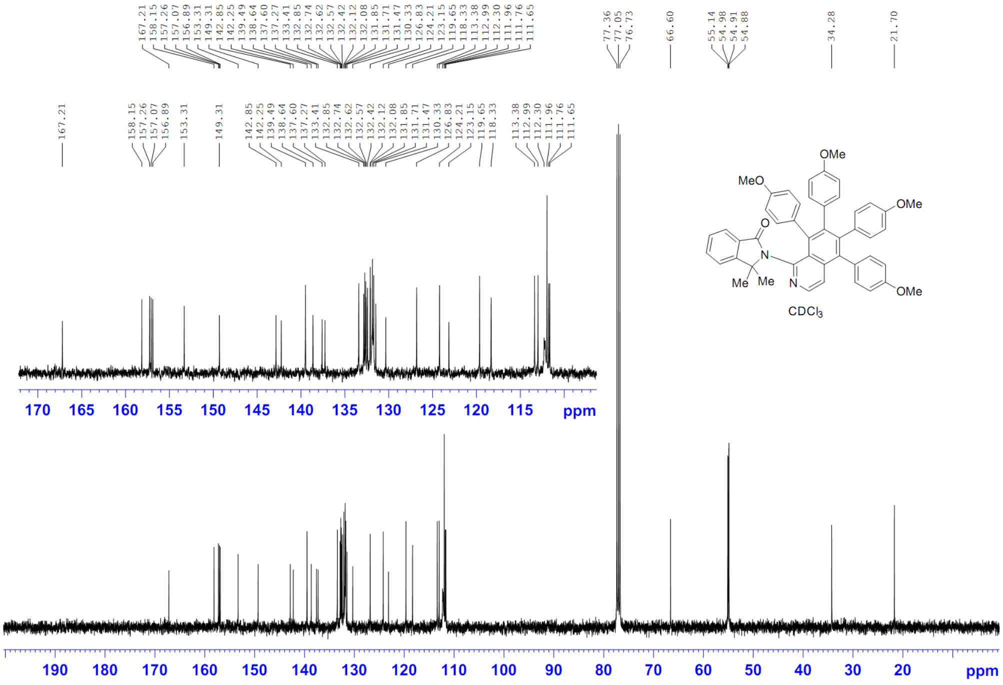
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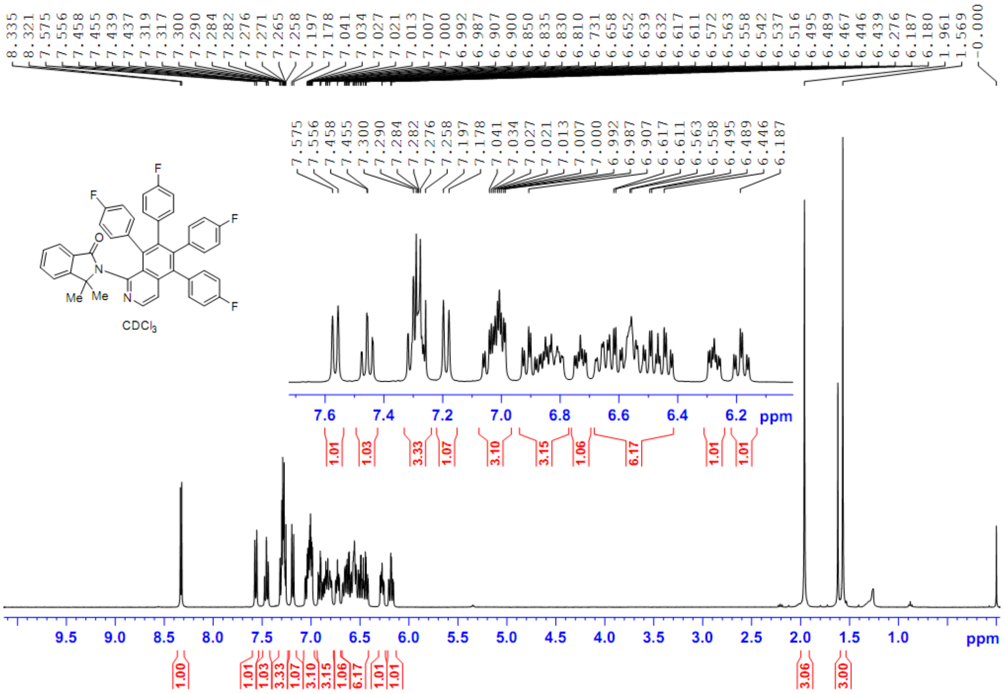
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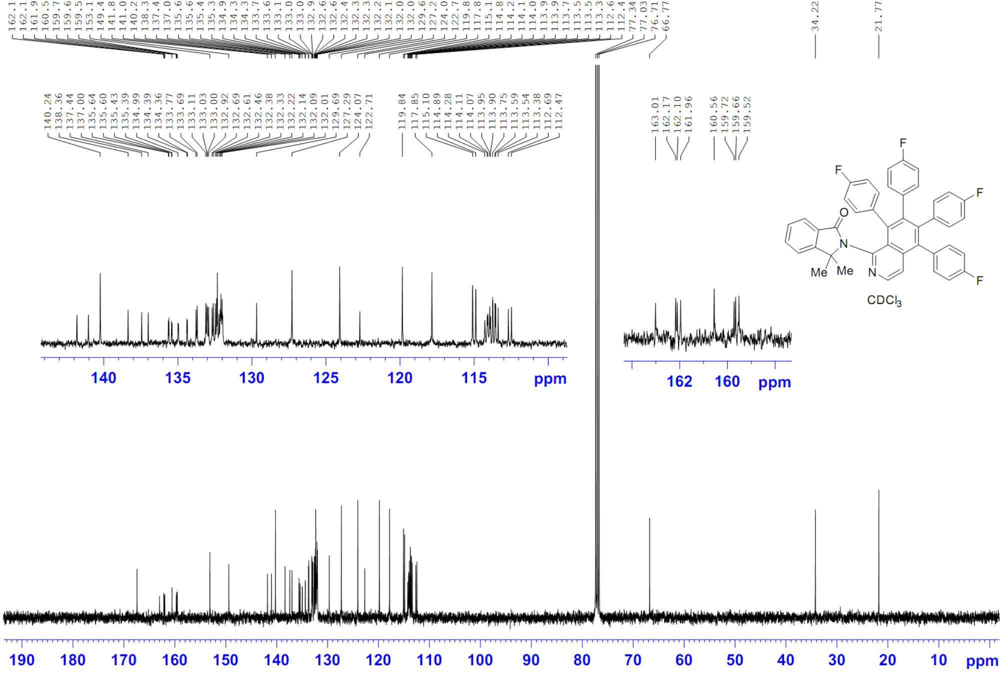
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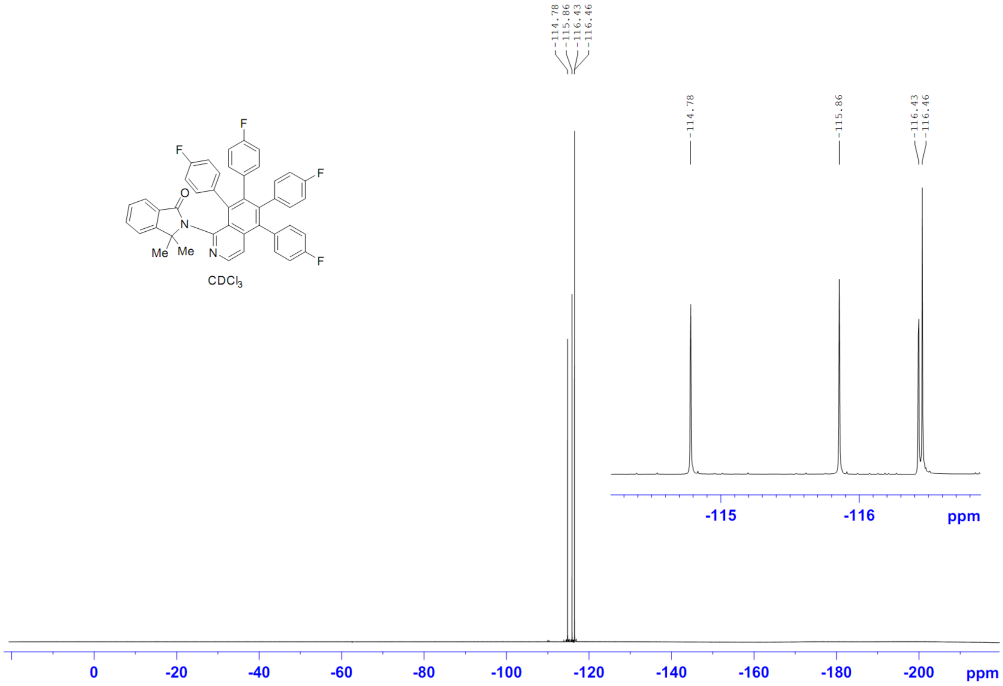
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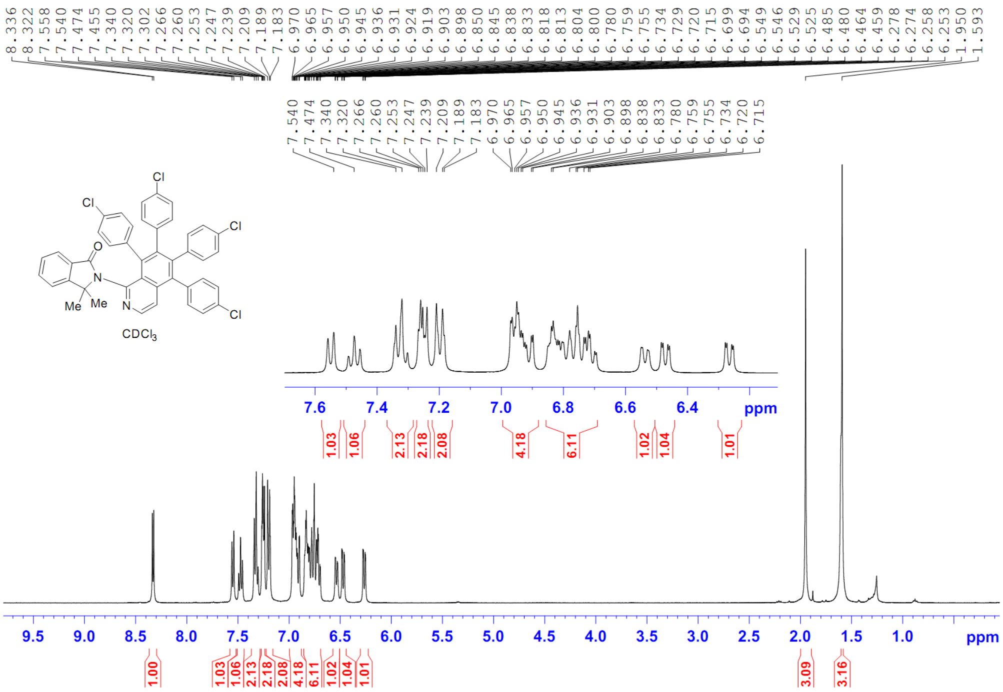
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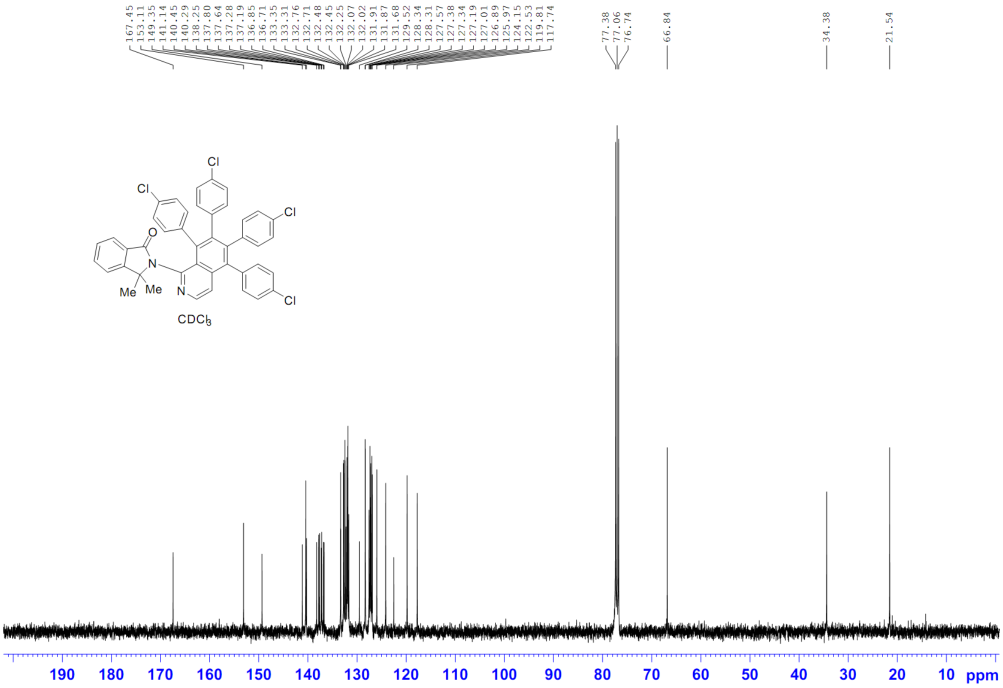
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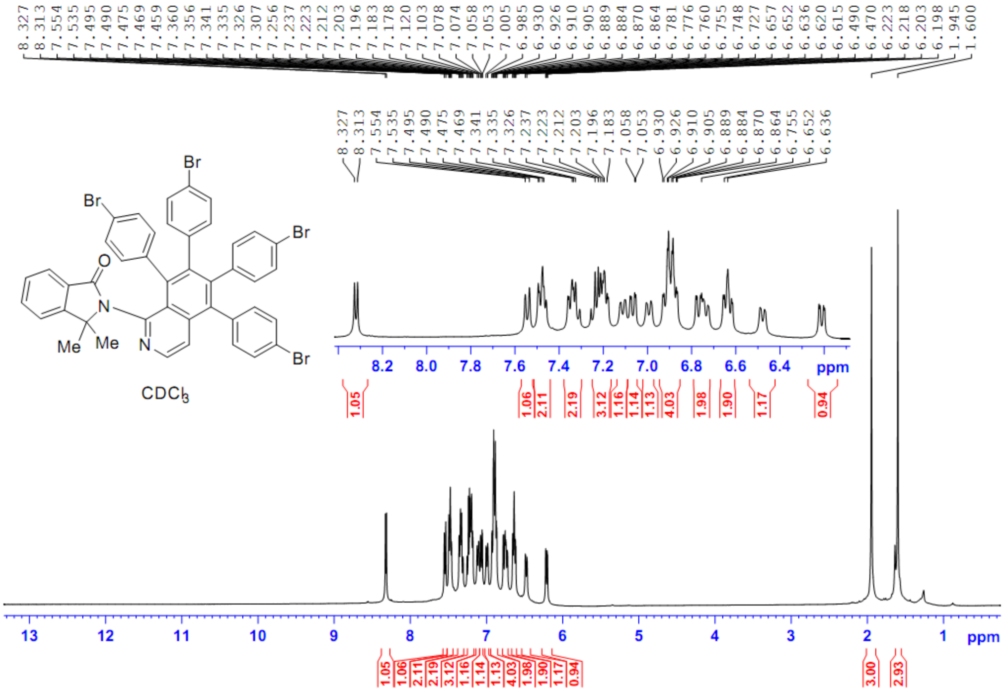
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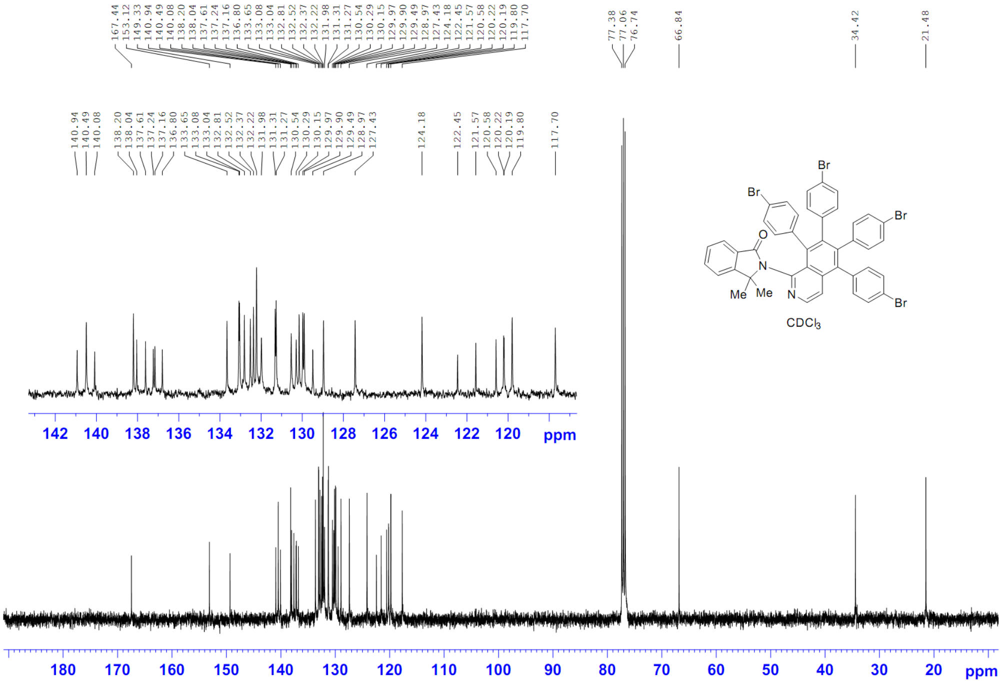
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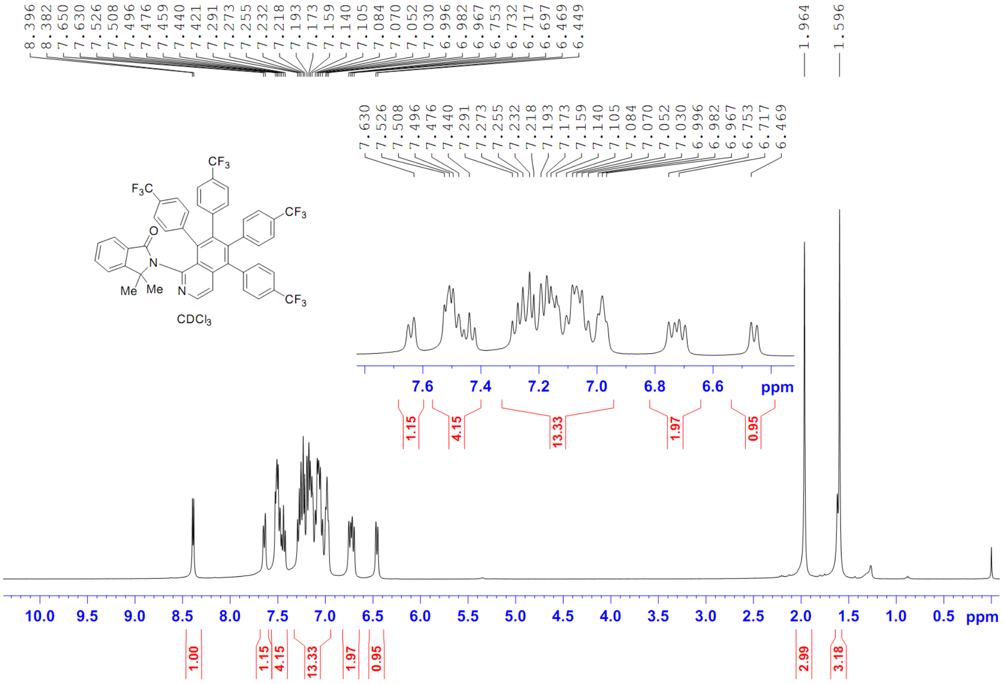
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7.159
7.140
7.105
7.084
7.070
7.052
7.030
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6.967
6.753
6.732
6.717
6.697
6.469
6.449
1.964
1.596
7.630
7.526
7.508
7.496
7.476
7.440
7.291
7.273
7.255
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7.218
7.193
7.173
7.159
7.140
7.105
7.084
7.070
7.052
7.030
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6.982
6.967
6.753
6.717
6.469
CF3
F3C
CF3
O
N
CF3
Me
Me
N
CDCl3
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7.4
7.2
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6.8
6.6
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0.95
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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
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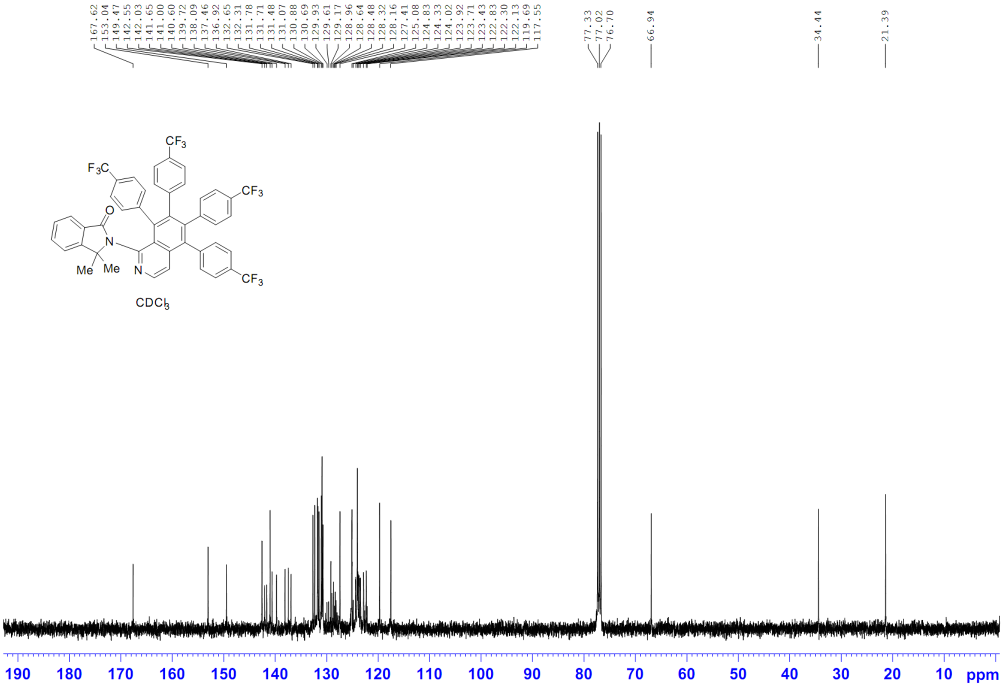
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131.78
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129.61
129.17
128.96
128.64
128.48
128.32
128.16
127.41
125.08
124.83
124.33
124.02
123.92
123.71
123.43
122.83
122.30
122.13
119.69
117.55
77.33
77.02
76.70
66.94
34.44
21.39
CF3
F3C
CF3
O
N
CF3
Me
Me
N
CDCl3
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150
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100
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70
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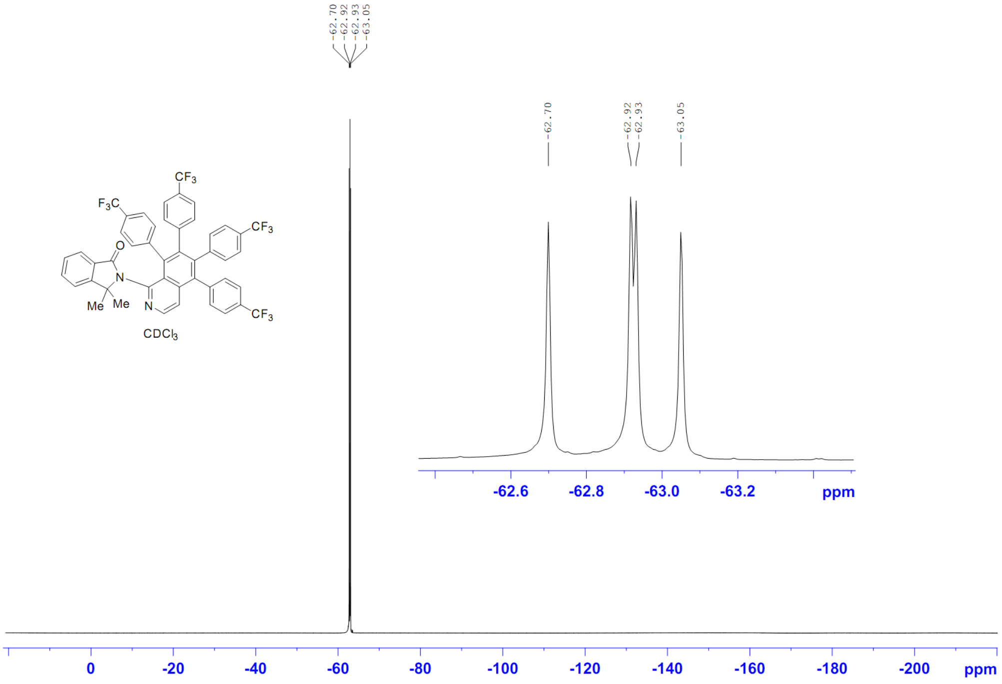
20
10
ppm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CF3
CF3
CF3
O
N
N
Me
Me
CDCl3
-62.70
-62.92
-62.93
-63.05
-62.6
-62.8
-63.0
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ppm
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-80
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-120
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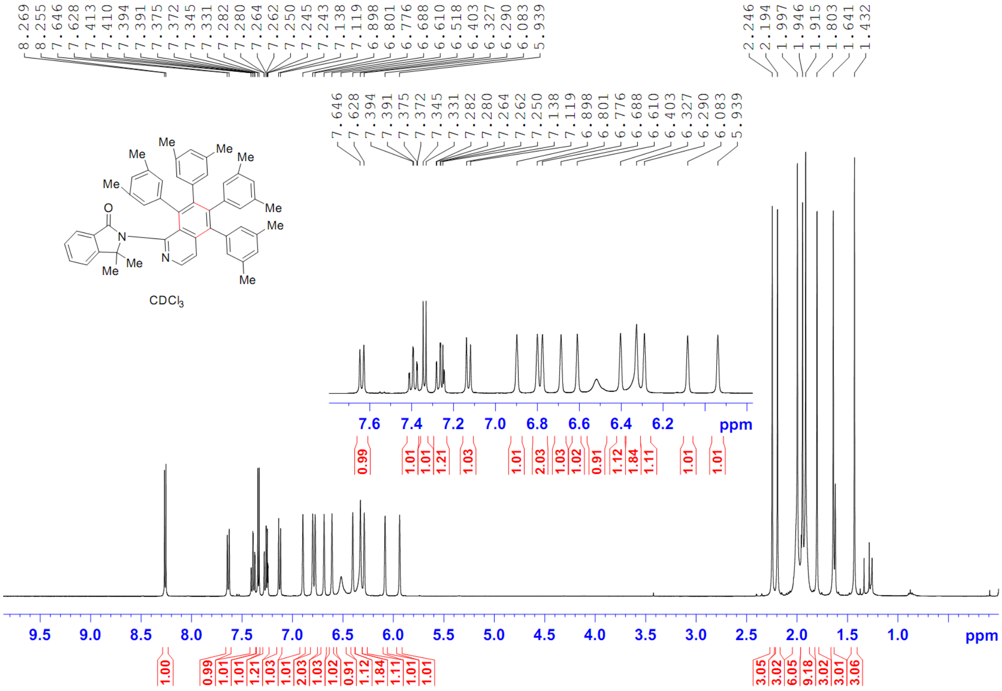
-180
-200
ppm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7.280
7.264
7.262
7.250
7.245
7.243
7.138
7.119
6.898
6.801
6.776
6.688
6.610
6.518
6.403
6.327
6.290
6.083
5.939
2.246
2.194
1.997
1.946
1.915
1.803
1.641
1.432
7.646
7.628
7.394
7.391
7.375
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7.345
7.331
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7.250
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6.801
6.776
6.688
6.610
6.403
6.327
6.290
6.083
5.939
Me
Me
Me
Me
Me
Me
Me
Me
O
N
N
Me
Me
CDCl3
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
ppm
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1.01
1.21
1.03
1.01
2.03
1.03
1.02
0.91
1.12
1.84
1.11
1.01
1.01
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
ppm
1.00
0.99
1.01
1.01
1.21
1.03
1.01
2.03
1.03
1.02
0.91
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1.01
1.01
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3.02
6.05
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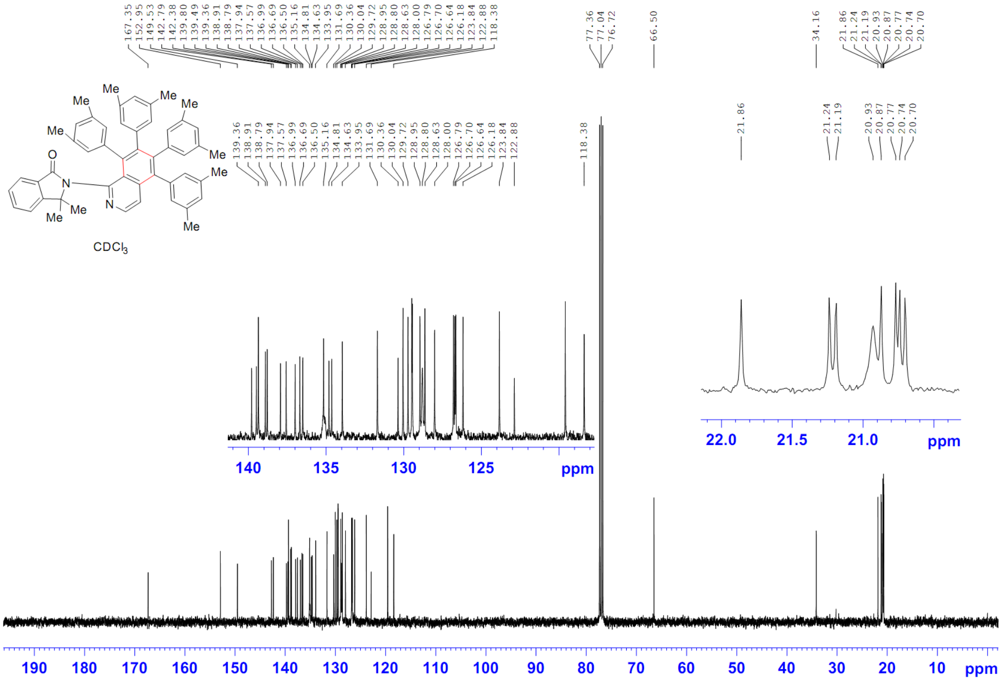
3.02
3.01
3.06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136.50
135.16
134.81
134.63
133.95
131.69
130.36
130.04
129.72
128.95
128.80
128.63
128.00
126.79
126.70
126.64
126.18
123.84
122.88
118.38
77.36
77.04
76.72
66.50
34.16
21.86
21.24
21.19
20.93
20.87
20.77
20.74
20.70
139.36
138.91
138.79
137.94
137.57
136.99
136.69
136.50
135.16
134.81
134.63
133.95
131.69
130.36
130.04
129.72
128.95
128.80
128.63
128.00
126.79
126.70
126.64
126.18
123.84
122.88
118.38
21.86
21.24
21.19
20.93
20.87
20.77
20.74
20.70
Me
Me
Me
Me
Me
Me
Me
Me
O
N
N
Me
Me
CDCl3
140
135
130
125
ppm
22.0
21.5
21.0
ppm
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180
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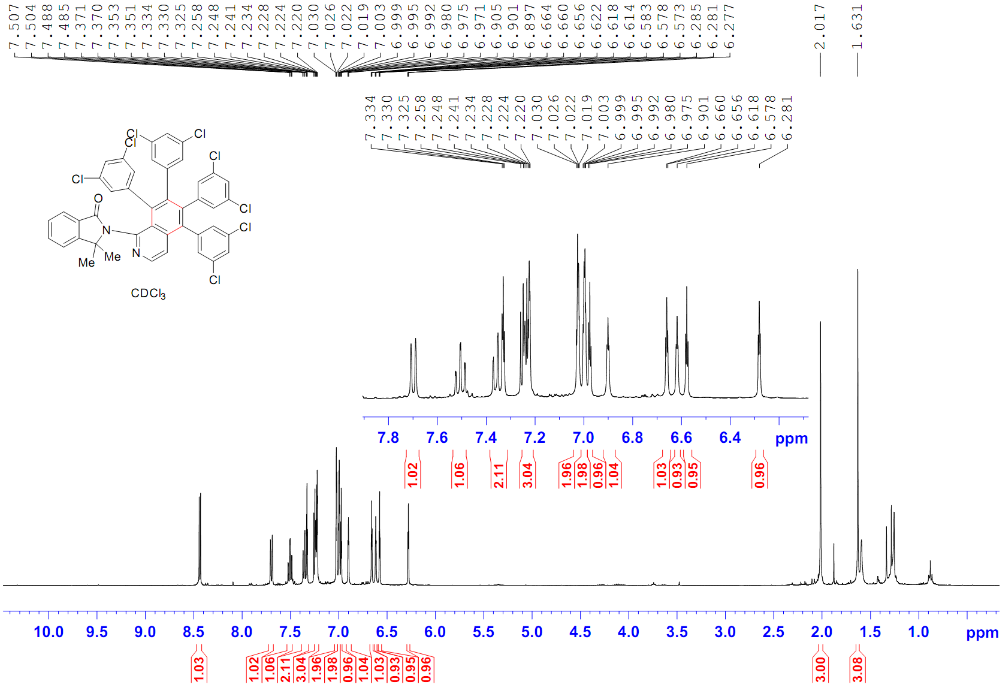
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.370
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7.351
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7.330
7.325
7.258
7.258
7.248
7.241
7.234
7.228
7.224
7.220
7.030
7.026
7.022
7.019
7.003
6.999
6.995
6.992
6.980
6.975
6.971
6.905
6.901
6.897
6.664
6.660
6.656
6.622
6.618
6.614
6.583
6.578
6.573
6.285
6.281
6.277
2.017
1.631
7.334
7.330
7.325
7.258
7.248
7.241
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7.228
7.224
7.220
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7.022
7.019
7.003
6.999
6.995
6.992
6.980
6.975
6.901
6.660
6.656
6.618
6.578
6.281
Cl
Cl
Cl
Cl
Cl
Cl
Cl
Cl
O
N
Me
Me
N
CDCl3
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
ppm
1.02
1.06
2.11
3.04
1.96
1.98
0.96
1.04
1.03
0.93
0.95
0.96
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
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2.5
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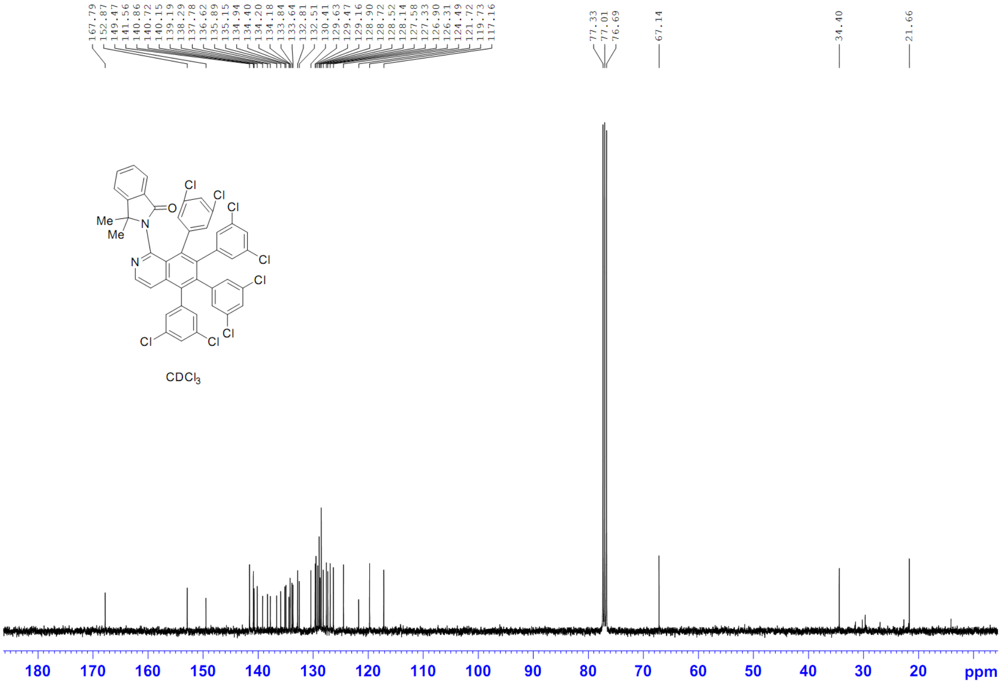
1.0
ppm
1.03
1.02
1.06
2.11
3.04
1.96
1.98
0.96
1.04
1.03
0.93
0.95
0.96
3.00
3.08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135.15
134.94
134.40
134.20
134.18
133.84
133.64
132.81
132.51
130.41
129.63
129.47
129.16
128.90
128.72
128.52
128.14
127.58
127.33
126.90
126.31
124.49
121.72
119.73
117.16
77.33
77.01
76.69
67.14
34.40
21.66
O
Me
Me
N
N
Cl
Cl
Cl
Cl
Cl
Cl
Cl
Cl
CDCl3
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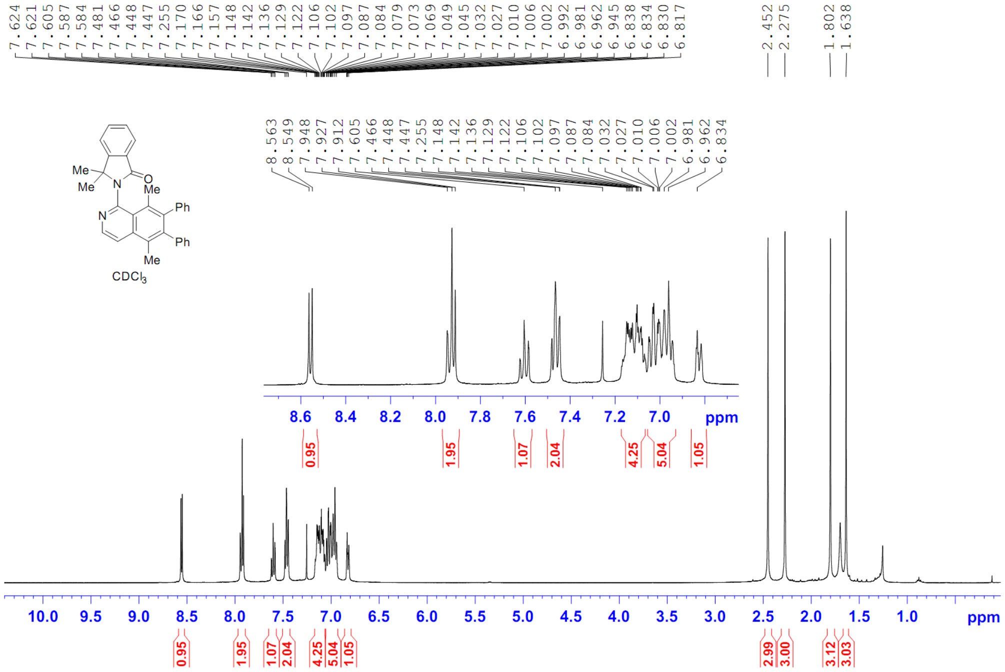
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.447
7.255
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7.166
7.157
7.148
7.142
7.136
7.129
7.122
7.106
7.102
7.097
7.087
7.084
7.079
7.073
7.069
7.049
7.045
7.032
7.027
7.010
7.006
7.002
6.992
6.981
6.962
6.945
6.838
6.834
6.830
6.817
2.452
2.275
1.802
1.638
8.563
8.549
7.948
7.927
7.912
7.605
7.466
7.448
7.447
7.255
7.148
7.142
7.136
7.129
7.122
7.106
7.102
7.097
7.087
7.084
7.032
7.027
7.010
7.006
7.002
6.981
6.962
6.834
Me
Me
N
O
Me
Ph
N
Ph
Me
CDCl3
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
ppm
0.95
1.95
1.07
2.04
4.25
5.04
1.05
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
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1.95
1.07
2.04
4.25
5.04
1.05
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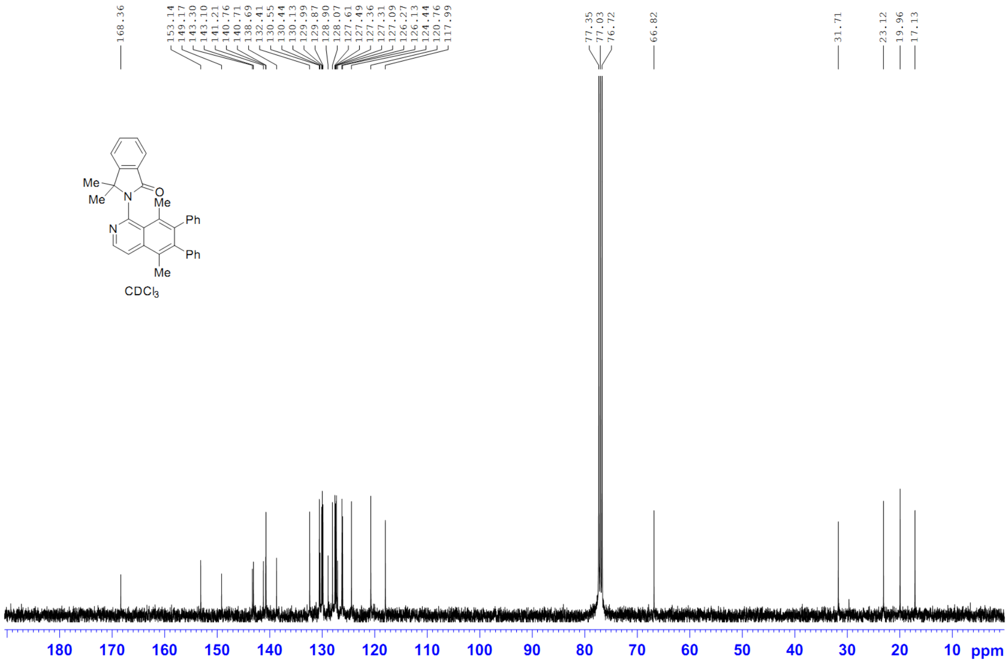
3.00
3.12
3.03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132.41
130.55
130.44
130.13
129.99
129.87
128.90
128.07
127.61
127.49
127.36
127.31
127.09
126.27
126.13
124.44
120.76
117.99
77.35
77.03
76.72
66.82
31.71
23.12
19.96
17.13
Me
Me
N
O
Me
Ph
N
Ph
Me
CDCl3
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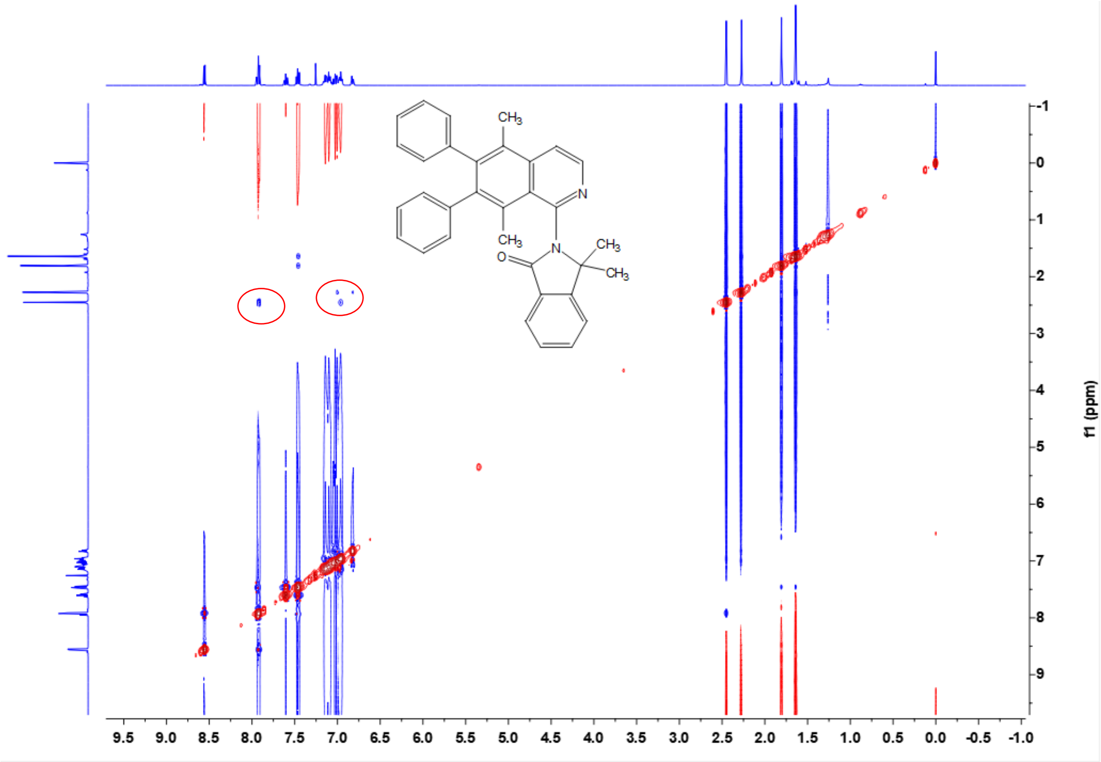
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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CH3
N
O
N
CH3
CH3
-1
0
1
2
3
4
5
6
7
8
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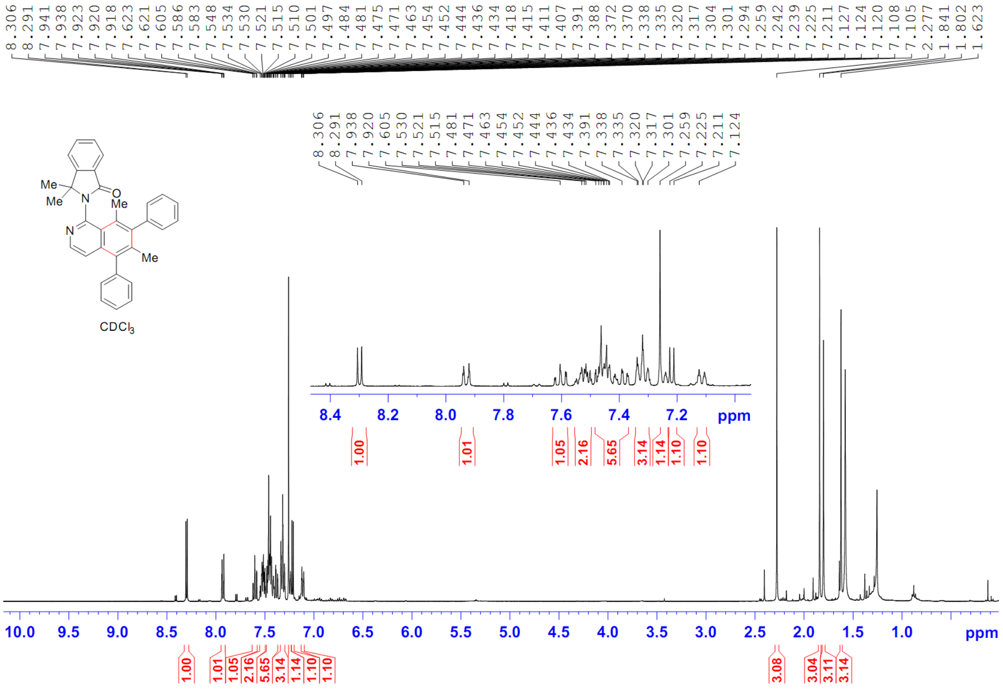
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0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8.306 8.291 7.938 7.920 7.605 7.530 7.521 7.515 7.481 7.471 7.463 7.454 7.452 7.444 7.436 7.434 7.391 7.338 7.335 7.320 7.317 7.301 7.259 7.225 7.211 7.124
Me
Me
N
O
Me
N
Me
CDCl3
8.4 8.2 8.0 7.8 7.6 7.4 7.2 ppm
1.00 1.01 1.05 2.16 5.65 3.14 1.14 1.10 1.10
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 ppm
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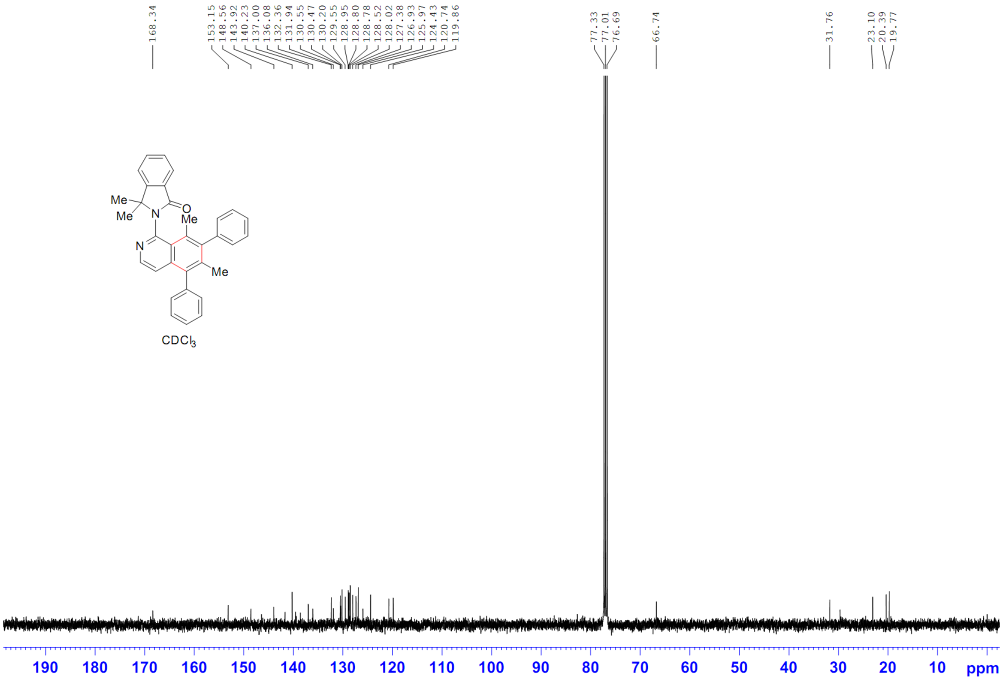
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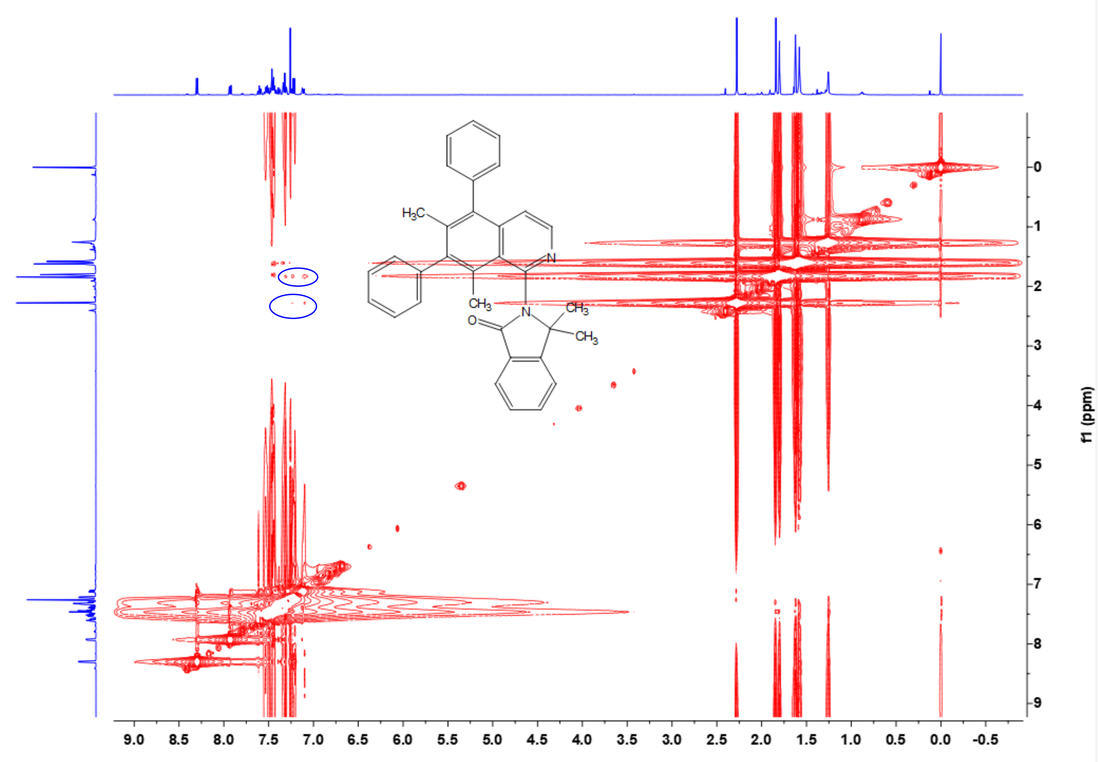
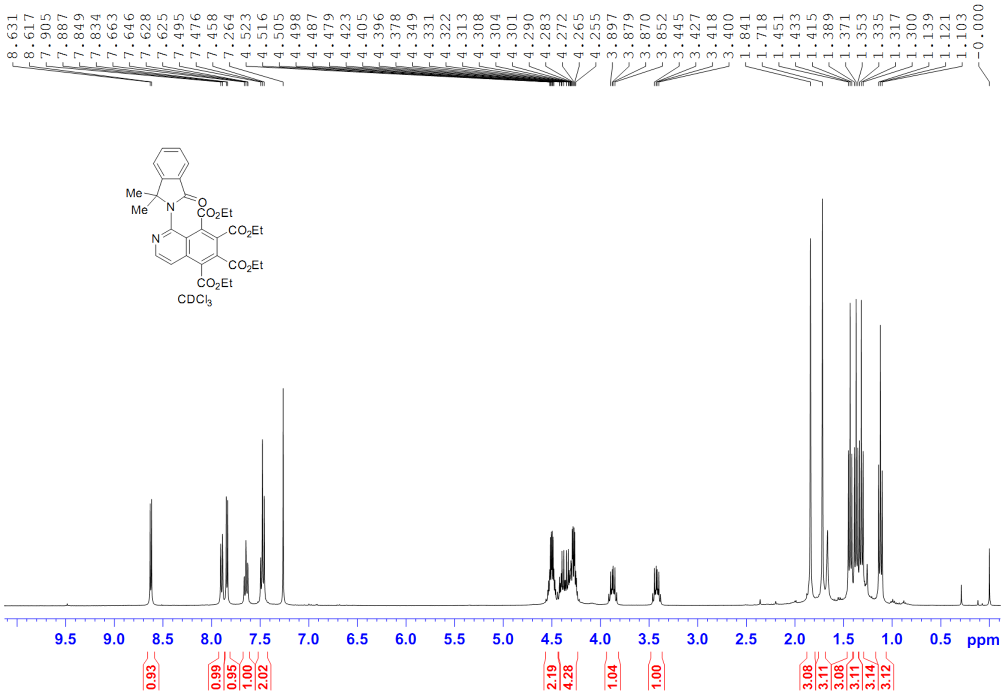
128.80
128.78
128.52
128.02
127.38
126.93
125.97
124.43
120.74
119.86
77.33
77.01
76.69
66.74
31.76
23.10
20.39
19.77
Me
Me
O
N
Me
N
Me
CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


H3C
N
CH3
O
N
CH3
CH3
0
1
2
3
4
5
6
7
8
9
f1 (ppm)
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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7.834
7.663
7.646
7.628
7.625
7.495
7.476
7.458
7.264
4.523
4.516
4.505
4.498
4.487
4.479
4.423
4.405
4.396
4.378
4.349
4.331
4.322
4.313
4.308
4.304
4.301
4.290
4.283
4.272
4.265
4.255
3.897
3.879
3.870
3.852
3.445
3.427
3.418
3.400
1.841
1.718
1.451
1.433
1.415
1.389
1.371
1.353
1.335
1.317
1.300
1.139
1.121
1.103
-0.000
Me
Me
N
O
N
CO_2Et
CO_2Et
CO_2Et
CO_2Et
$CDCl_3$
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
ppm
0.93
0.99
0.95
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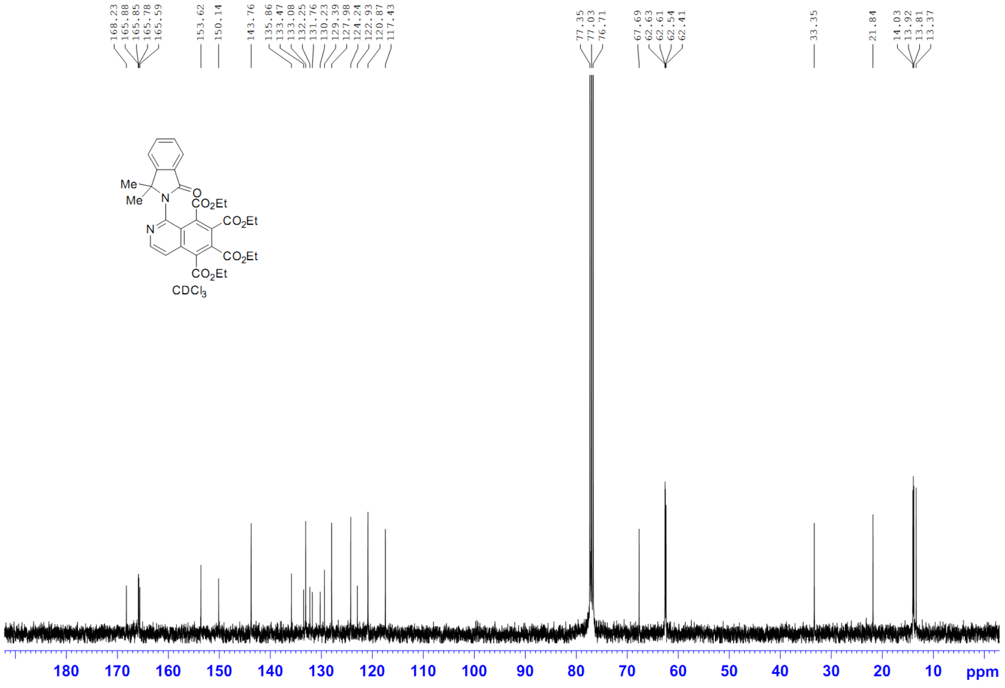
4.28
1.04
1.00
3.08
3.11
3.08
3.11
3.14
3.12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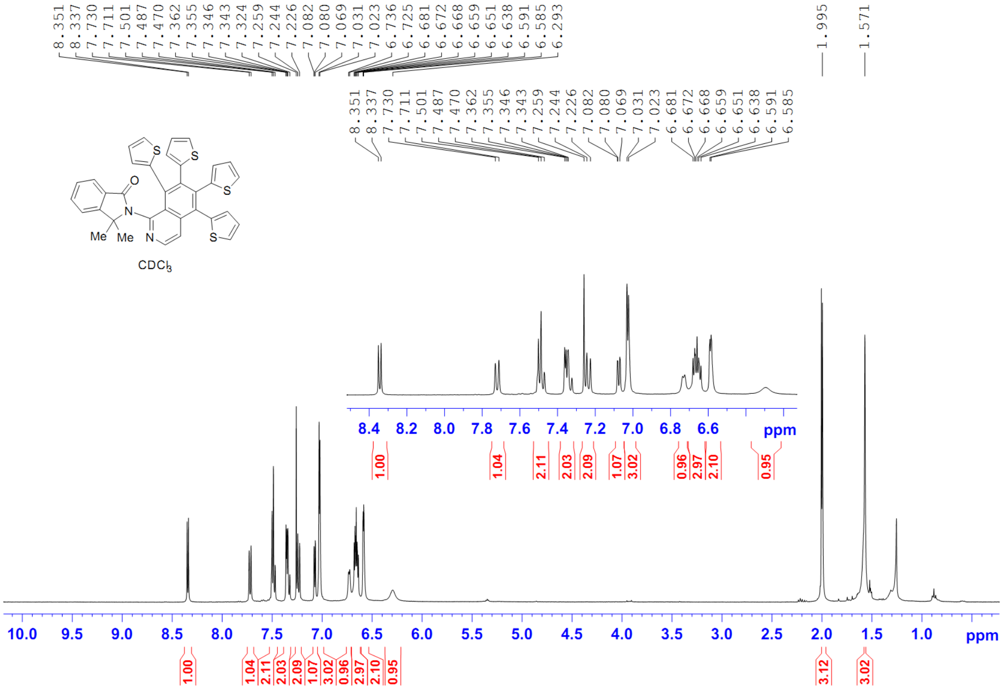
165.85
165.78
165.59
153.62
150.14
143.76
135.86
133.47
133.08
132.25
131.76
130.23
129.39
127.98
124.24
122.93
120.87
117.43
77.35
77.03
76.71
67.69
62.63
62.61
62.54
62.41
33.35
21.84
14.03
13.92
13.81
13.37
Me
Me
N
O
N
CO_2Et
CO_2Et
CO_2Et
CO_2Et
$CDCl_3$
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



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7.355
7.346
7.343
7.324
7.259
7.244
7.226
7.082
7.080
7.069
7.031
7.023
6.736
6.725
6.681
6.672
6.668
6.659
6.651
6.638
6.591
6.585
6.293
1.995
1.571
S
O
N
Me
Me
CDCl3
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
ppm
1.00
1.04
2.11
2.03
2.09
1.07
3.02
0.96
2.97
2.10
0.95
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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4.0
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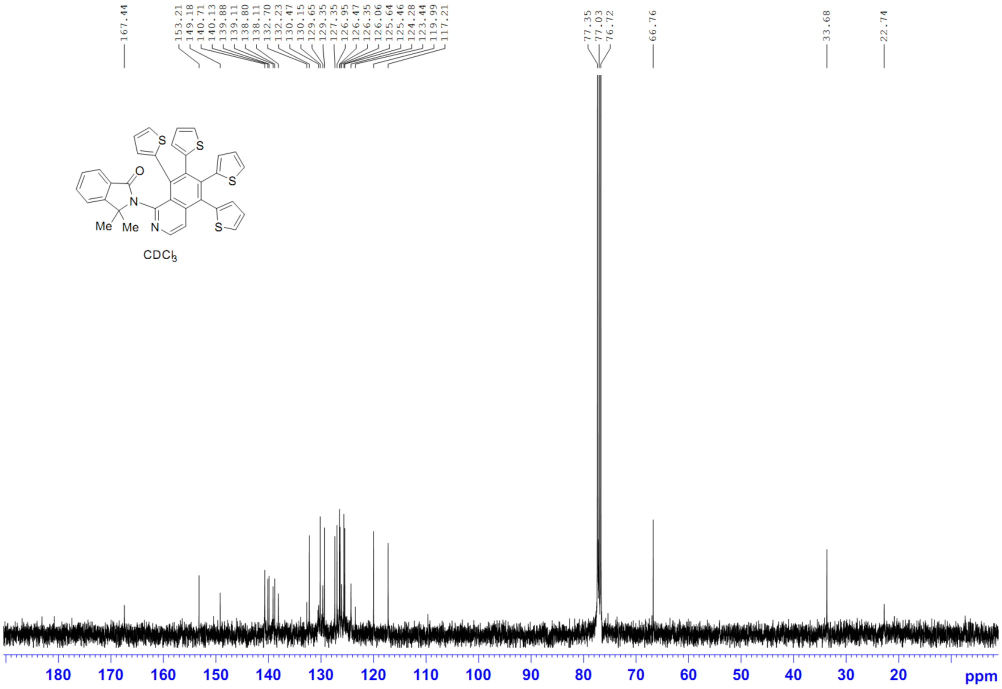
1.0
3.12
3.02



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139.88
139.11
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138.11
132.70
132.23
130.47
130.15
129.65
129.35
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126.95
126.47
126.35
126.06
125.64
125.46
124.28
123.44
119.99
117.21
77.35
77.03
76.72
66.76
33.68
22.74
S
O
N
Me
Me
CDCl3
180
170
160
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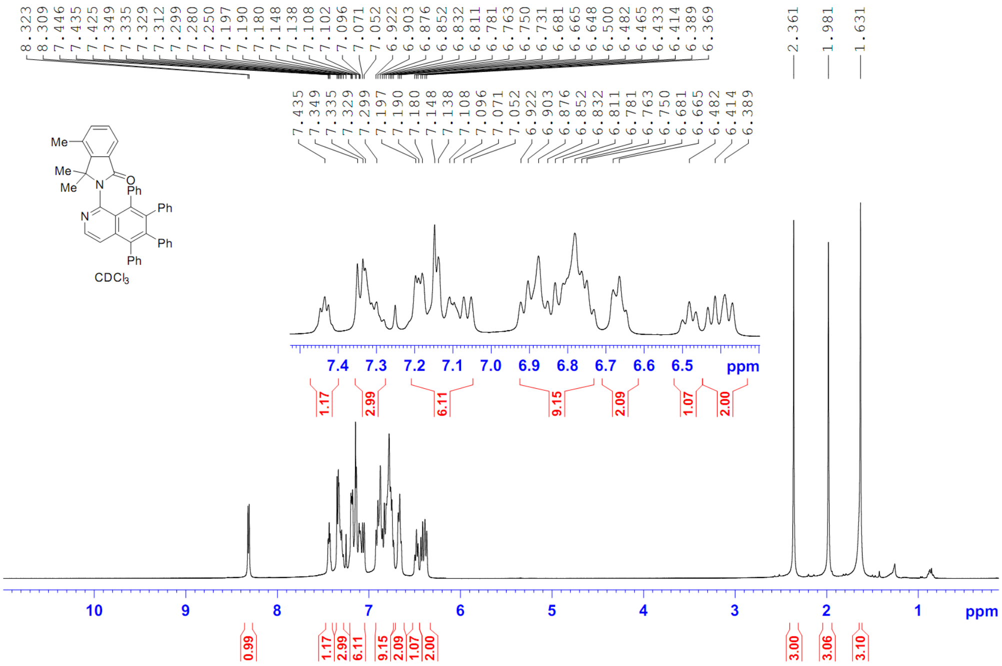
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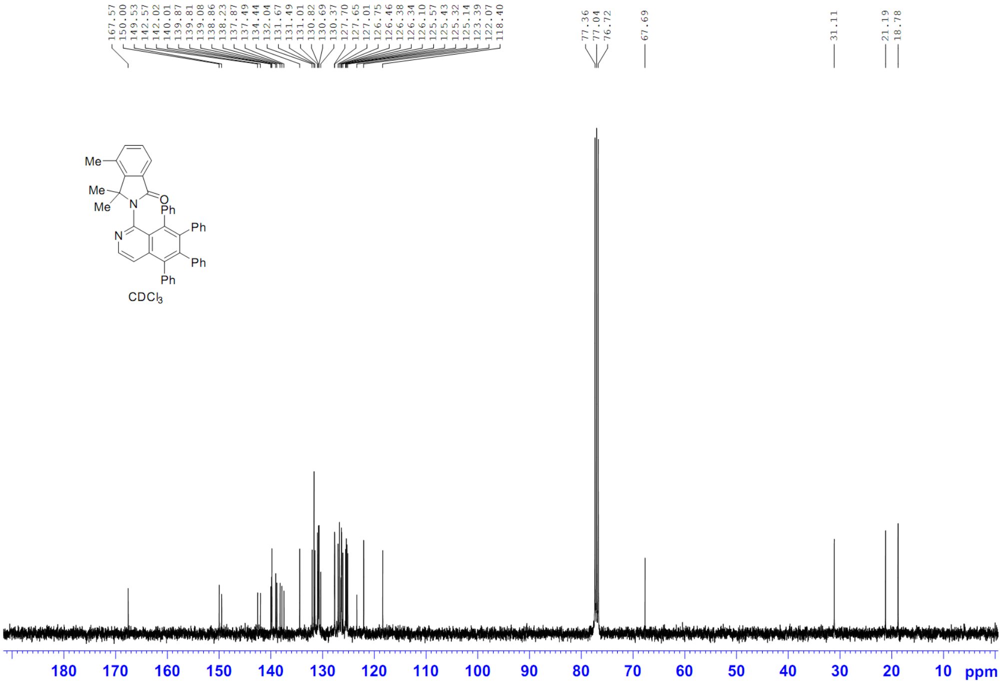
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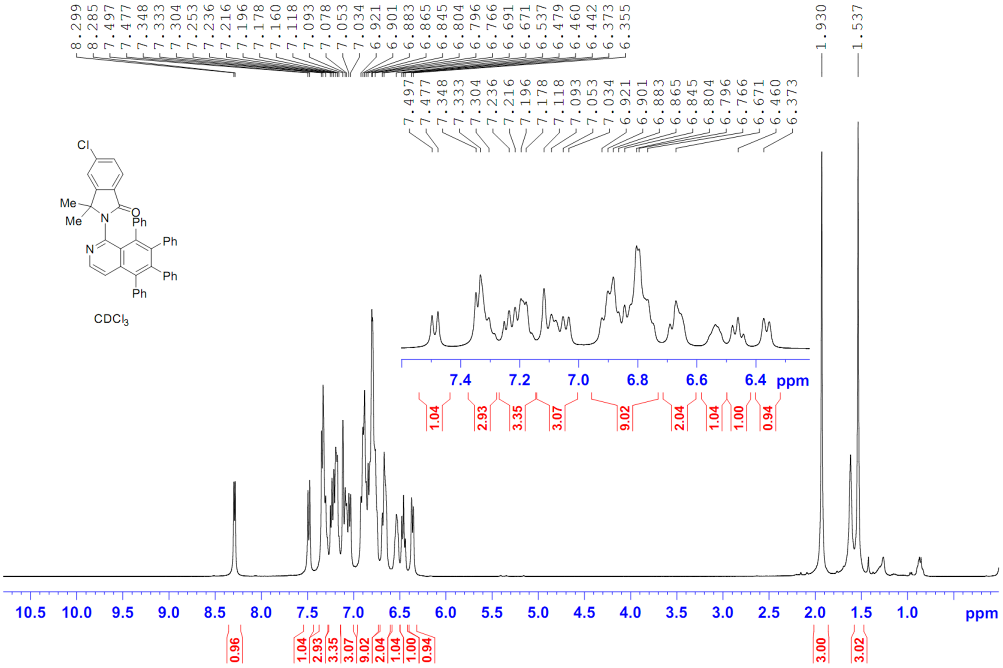
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6.804
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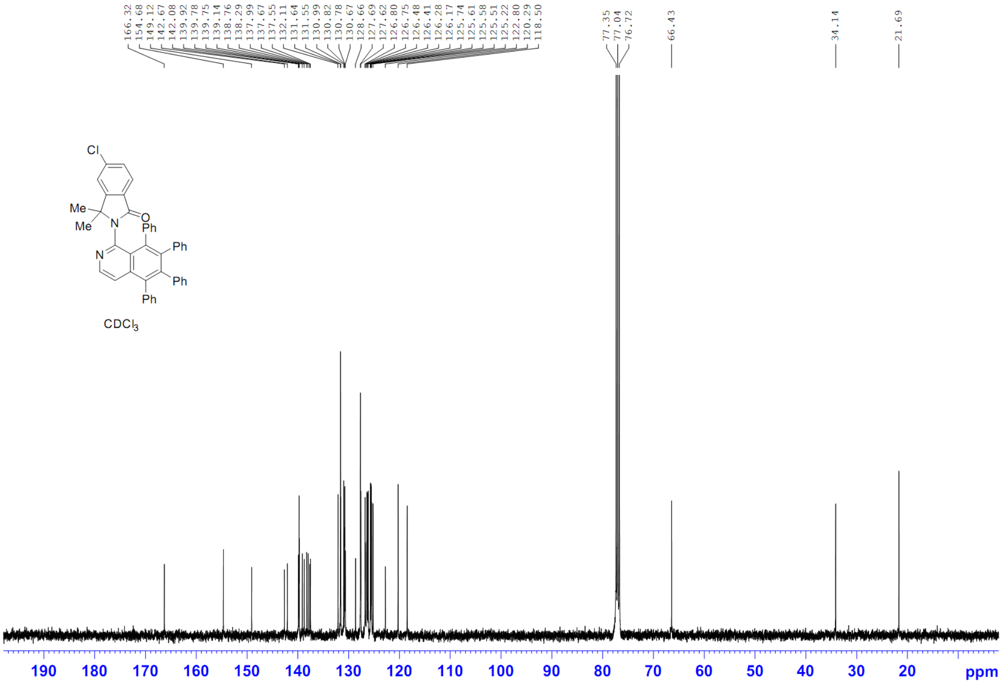
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77.04
76.72
66.43
34.14
21.69
Cl
Me
Me
N
O
Ph
N
Ph
Ph
Ph
CDCl$_3$
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40
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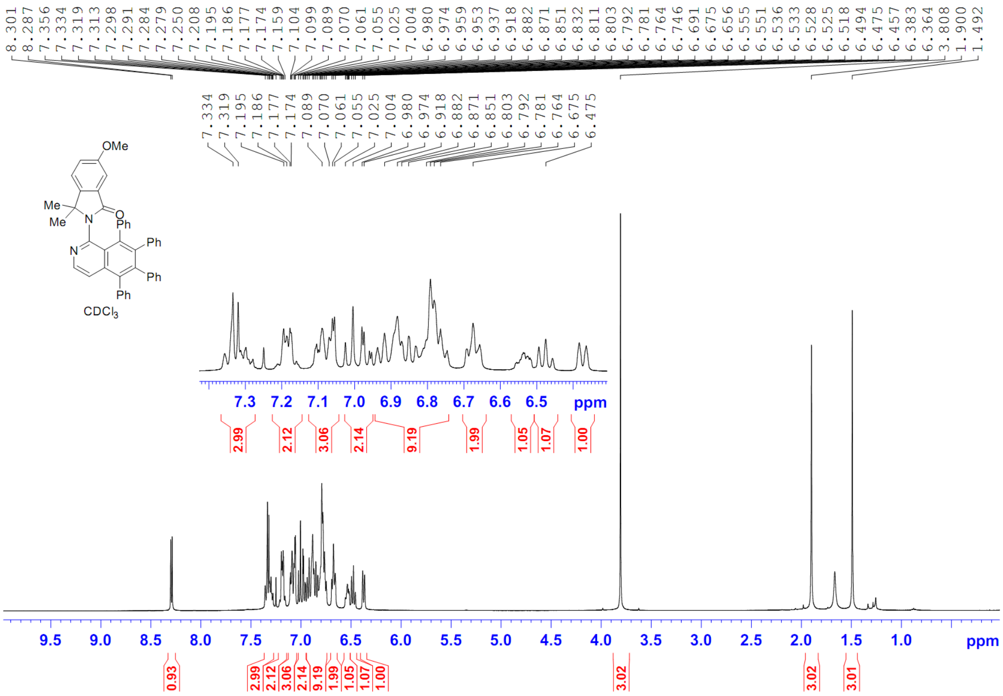
20
ppm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7.195
7.186
7.177
7.174
7.159
7.104
7.099
7.089
7.070
7.061
7.055
7.025
7.004
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6.959
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6.832
6.811
6.803
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6.746
6.691
6.675
6.656
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6.551
6.536
6.533
6.528
6.525
6.518
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6.475
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6.364
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1.492
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7.319
7.195
7.186
7.177
7.174
7.089
7.070
7.061
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6.980
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6.781
6.764
6.675
6.475
OMe
Me
Me
N
O
Ph
Ph
Ph
Ph
N
CDCl3
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2.99 2.12 3.06 2.14 9.19 1.99 1.05 1.07 1.00
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 ppm
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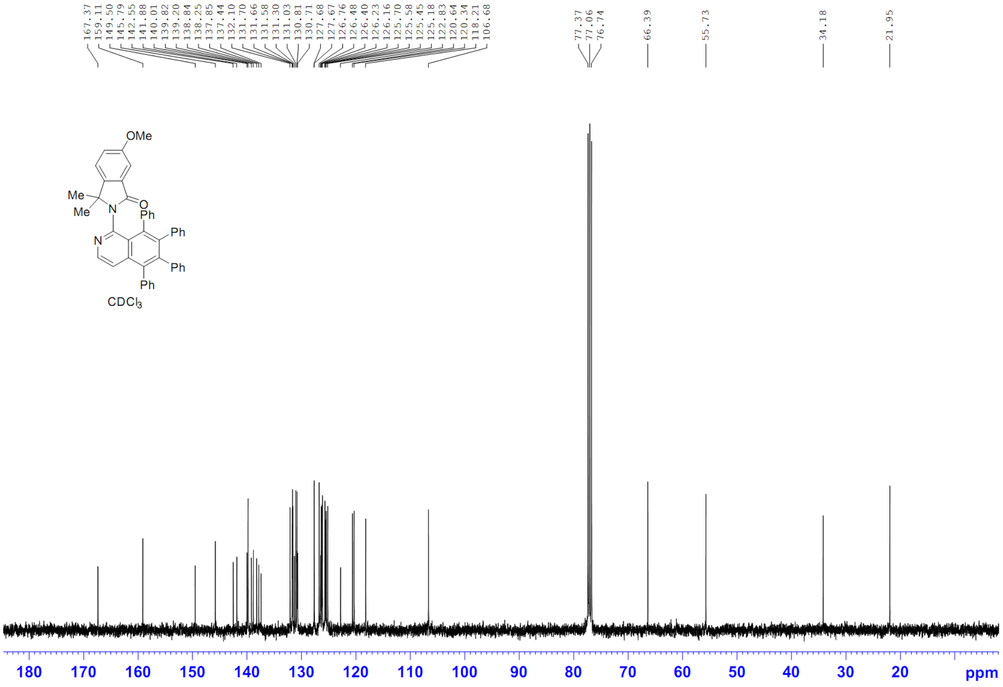
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131.66
131.58
131.30
131.03
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130.71
127.68
127.67
126.76
126.48
126.40
126.23
126.16
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125.58
125.45
125.18
122.83
120.64
120.34
118.21
106.68
77.37
77.06
76.74
66.39
55.73
34.18
21.95
OMe
Me
Me
N
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Ph
Ph
Ph
Ph
N
CDCl3
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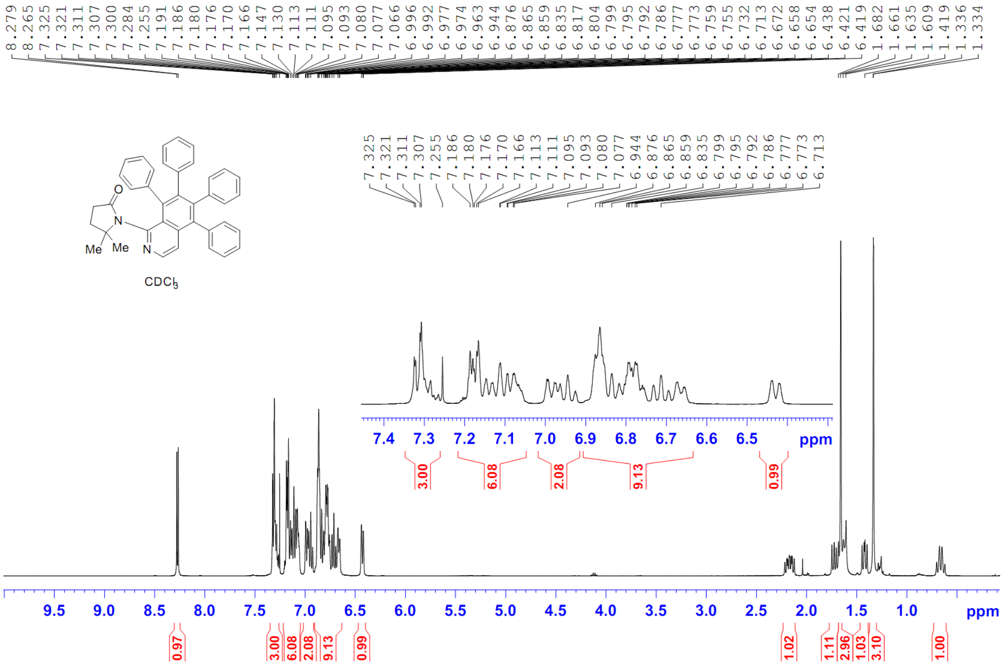
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7.170
7.166
7.147
7.130
7.113
7.111
7.095
7.093
7.080
7.077
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6.992
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6.974
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6.654
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6.419
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1.609
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1.336
1.334
O
N
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Me
N
CDCl3
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6.795
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7.0
6.9
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6.7
6.6
6.5
ppm
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6.08
2.08
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9.0
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4.5
4.0
3.5
3.0
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2.0
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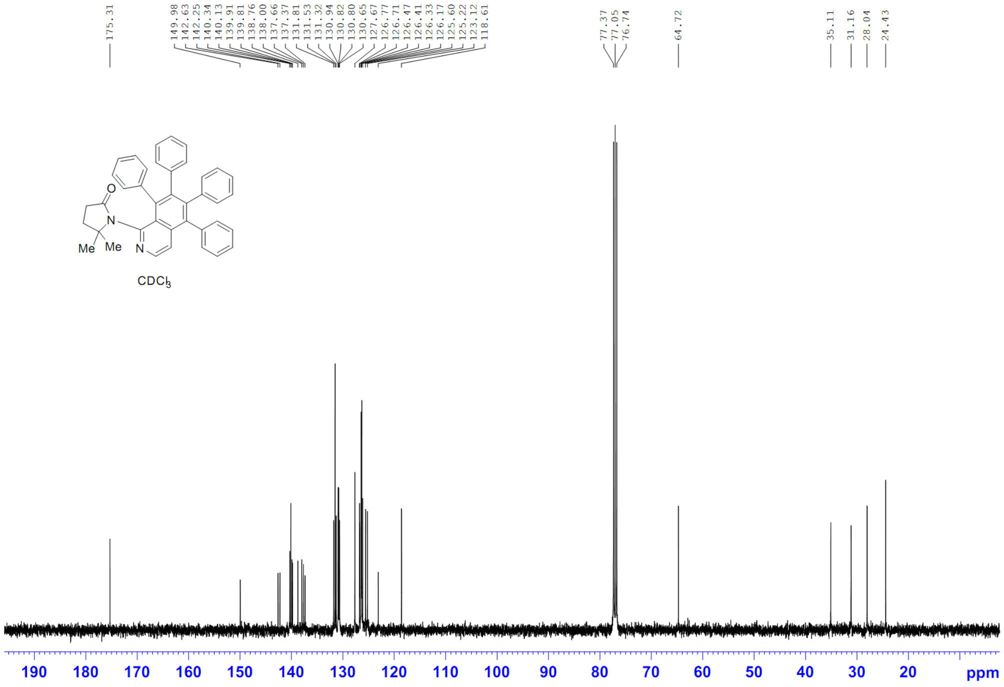
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77.37
77.05
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64.72
35.11
31.16
28.04
24.43
O
N
Me
Me
N
CDCl3
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120
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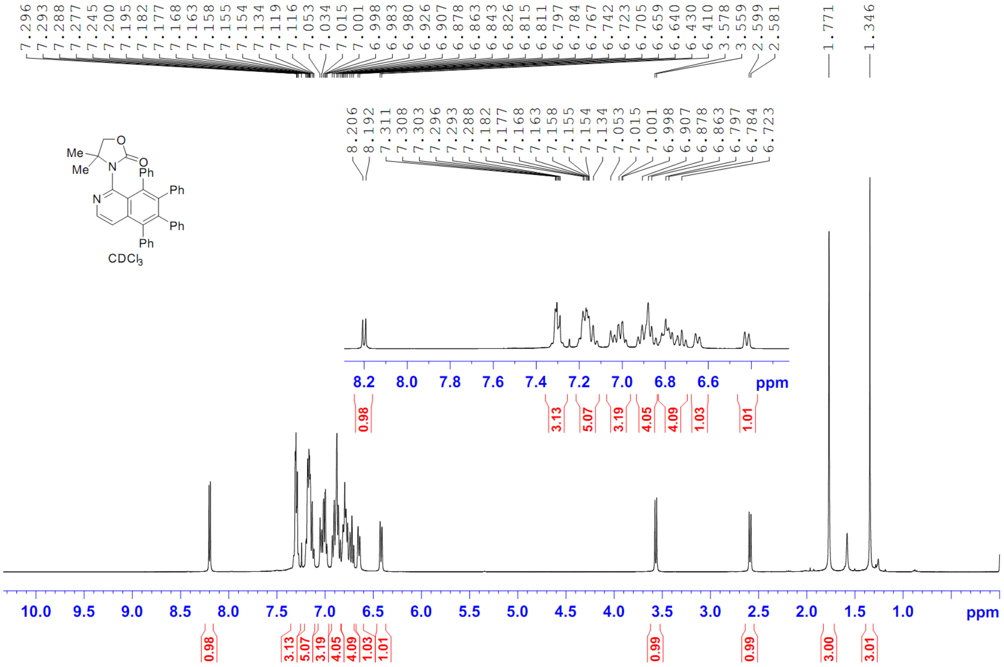
20
ppm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1.771
1.346
8.206 8.192 7.311 7.308 7.303 7.296 7.293 7.288 7.182 7.177 7.168 7.163 7.158 7.155 7.154 7.134 7.053 7.015 7.001 6.998 6.907 6.878 6.863 6.797 6.784 6.723
Me
Me
O
O
N
Ph
Ph
Ph
Ph
N
CDCl3
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0.98 3.13 5.07 3.19 4.05 4.09 1.03 1.01
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 ppm
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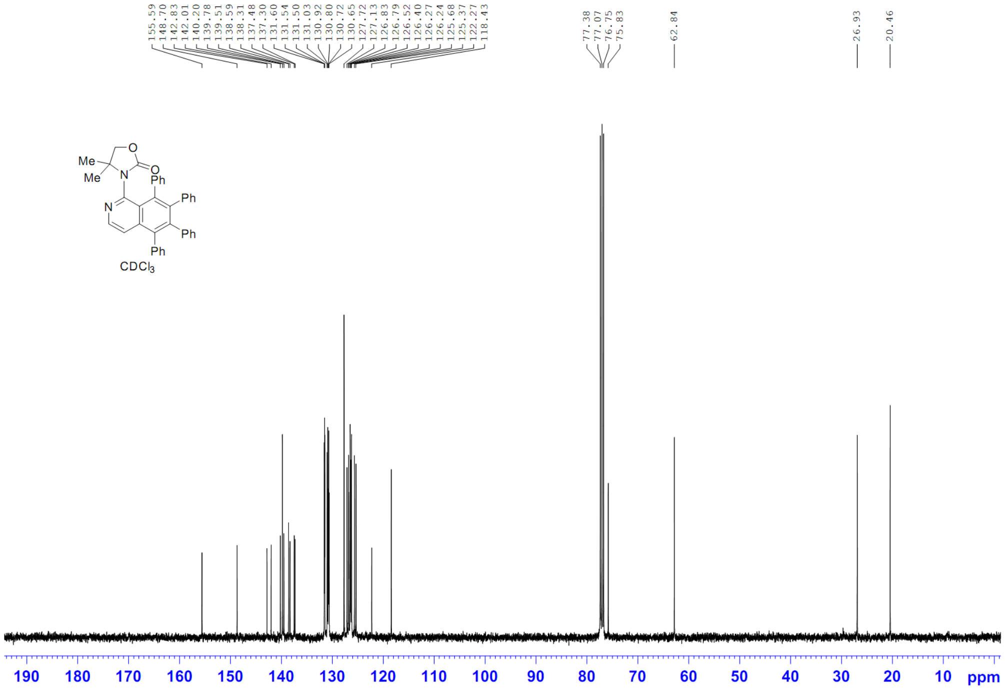
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77.38 77.07 76.75 75.83
62.84
26.93
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Ph
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N
CDCl3
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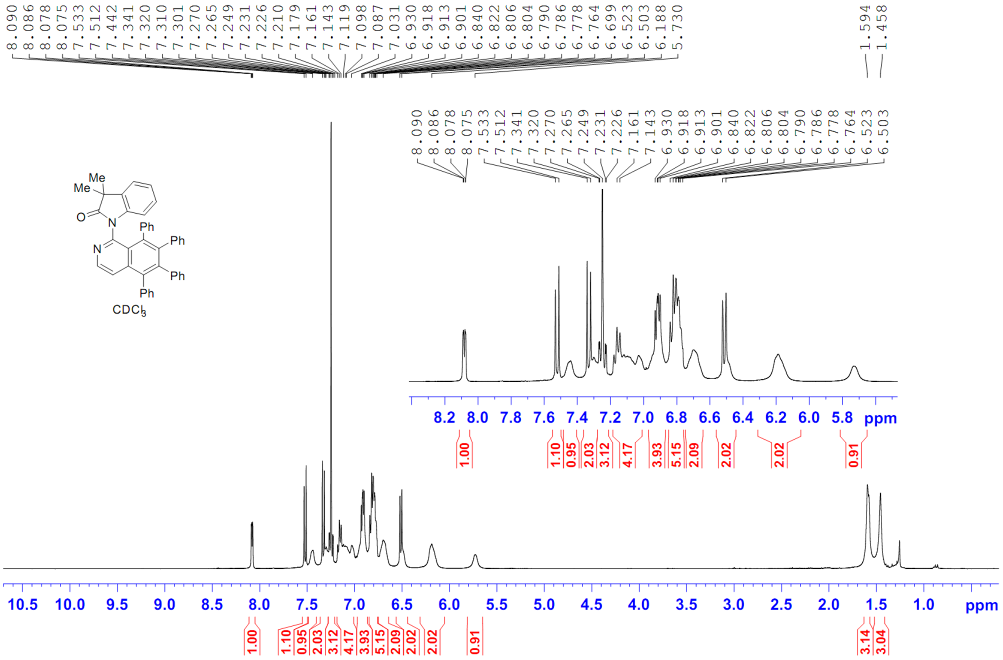
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Ph
Ph
CDCl3
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7.265
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7.143
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6.913
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2.02
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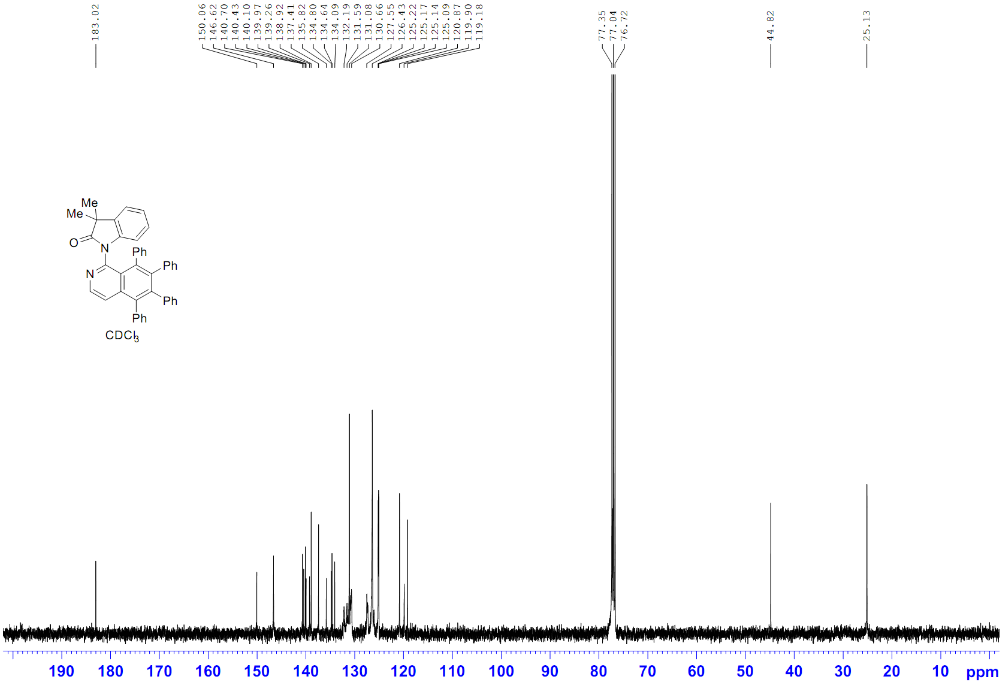
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125.22
125.17
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125.09
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119.18
77.35
77.04
76.72
44.82
25.13
Me
Me
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N
Ph
Ph
N
Ph
Ph
CDCl3
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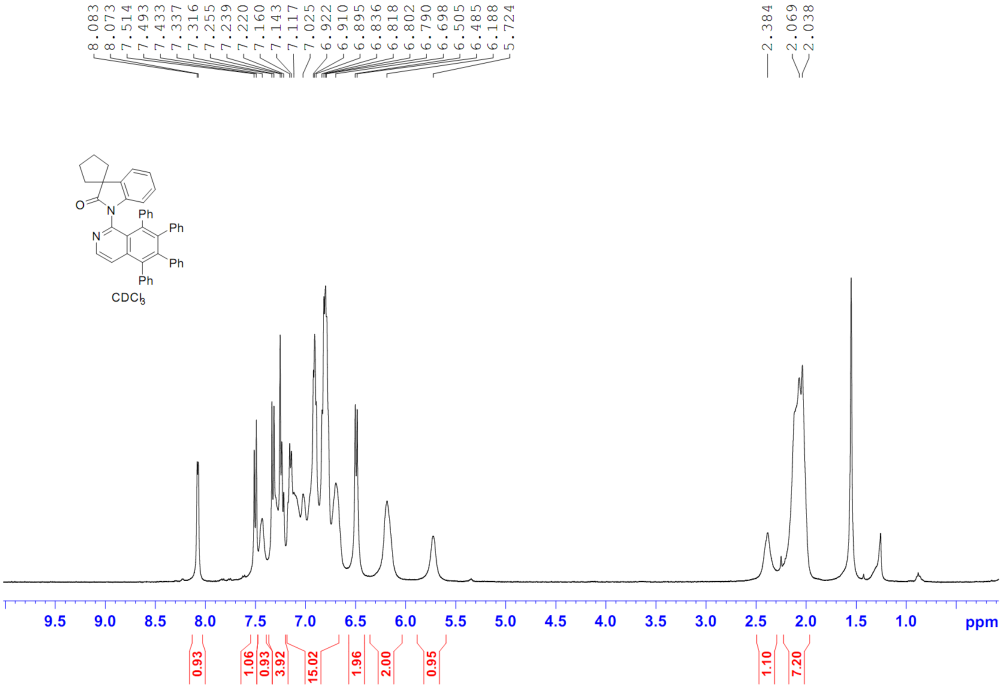
ppm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2.069
2.038
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Ph
Ph
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Ph
CDCl3
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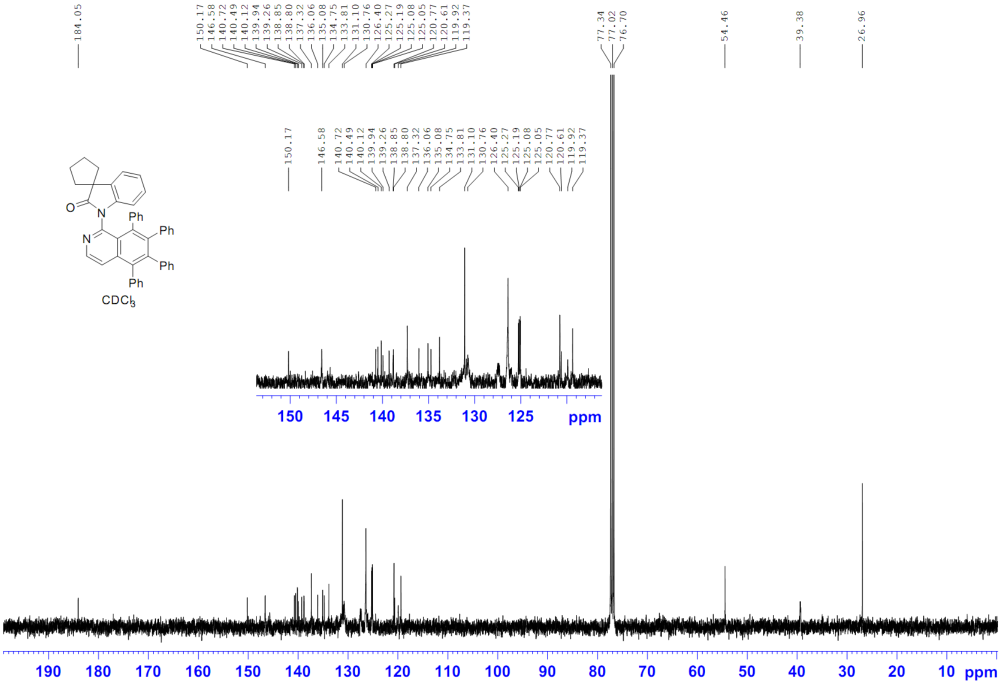
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Ph
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125
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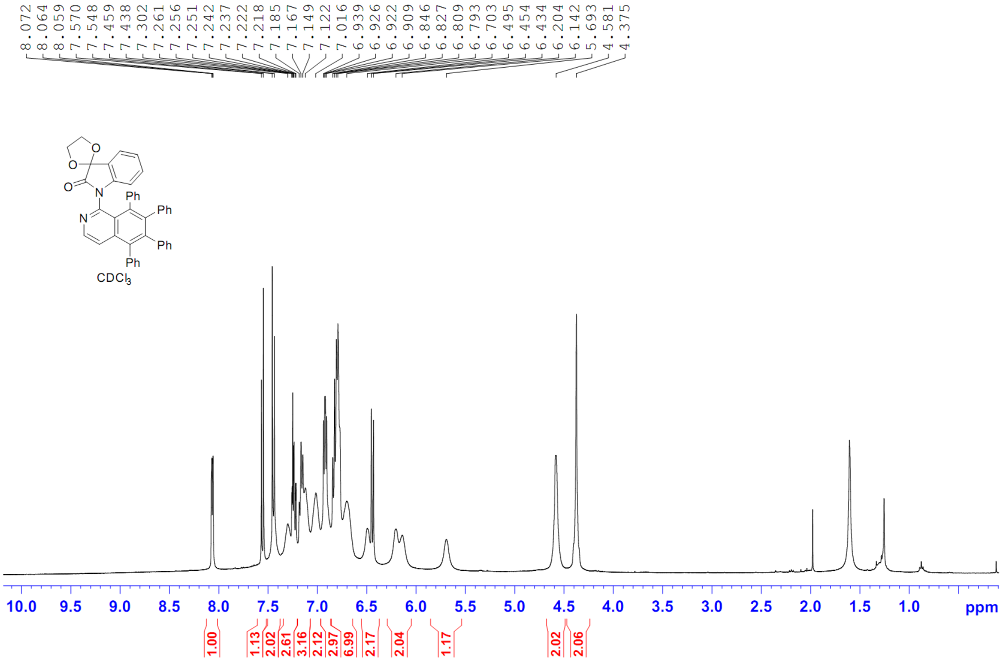
ppm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Ph
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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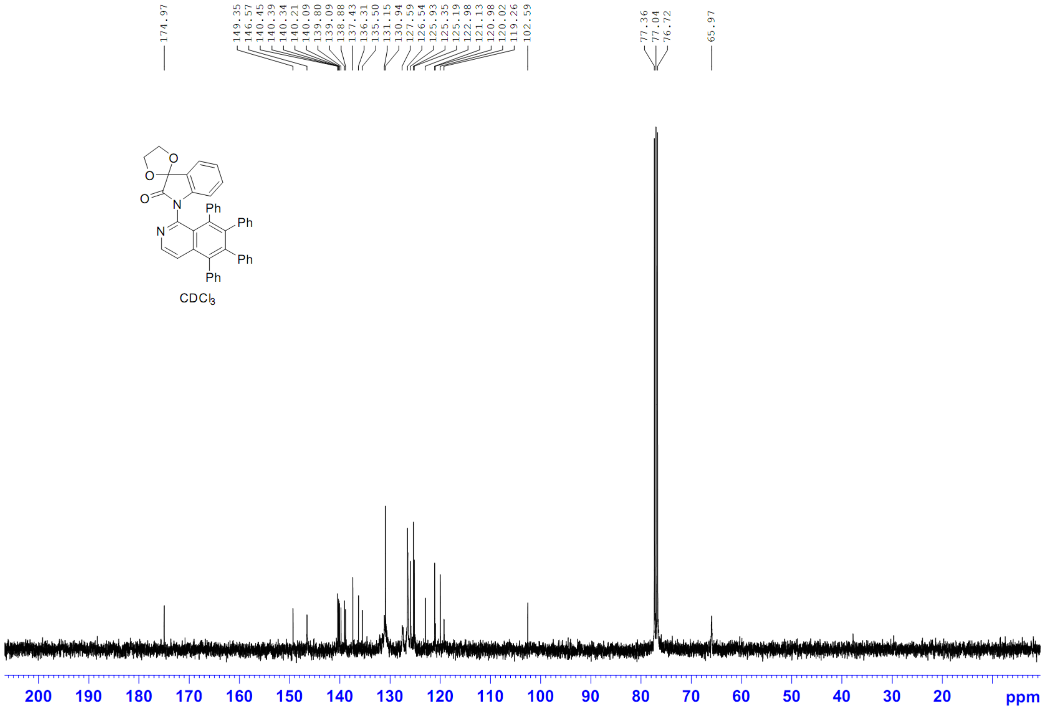
1.17
2.02
2.06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125.35
125.19
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121.13
120.98
120.02
119.26
102.59
77.36
77.04
76.72
65.97
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O
O
N
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Ph
Ph
Ph
Ph
CDCl3
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50
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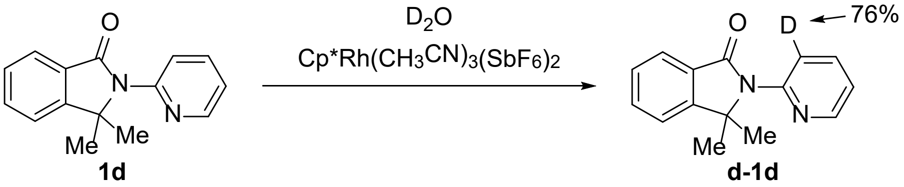
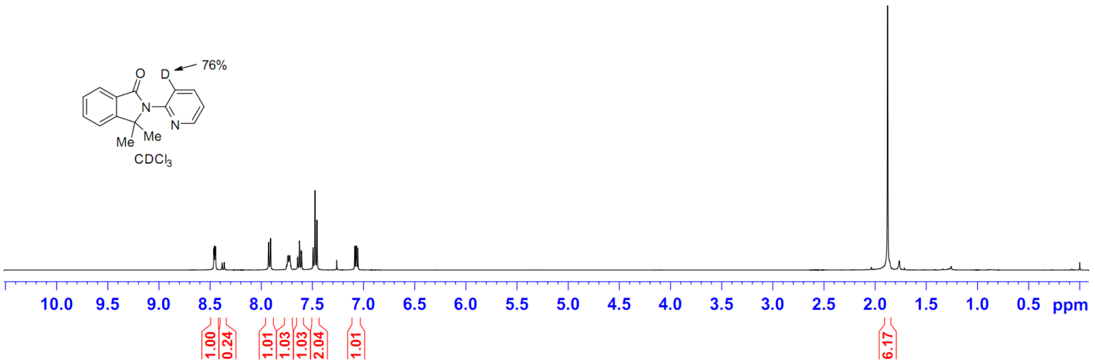
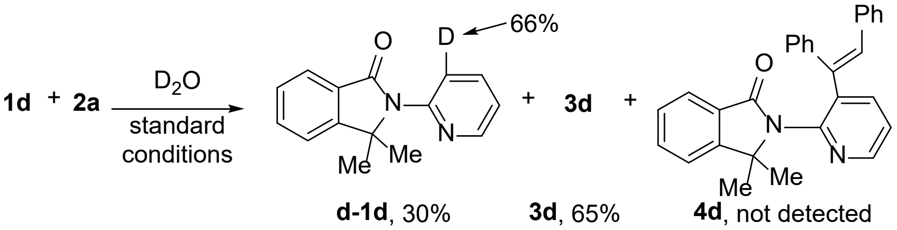
30
20
ppm

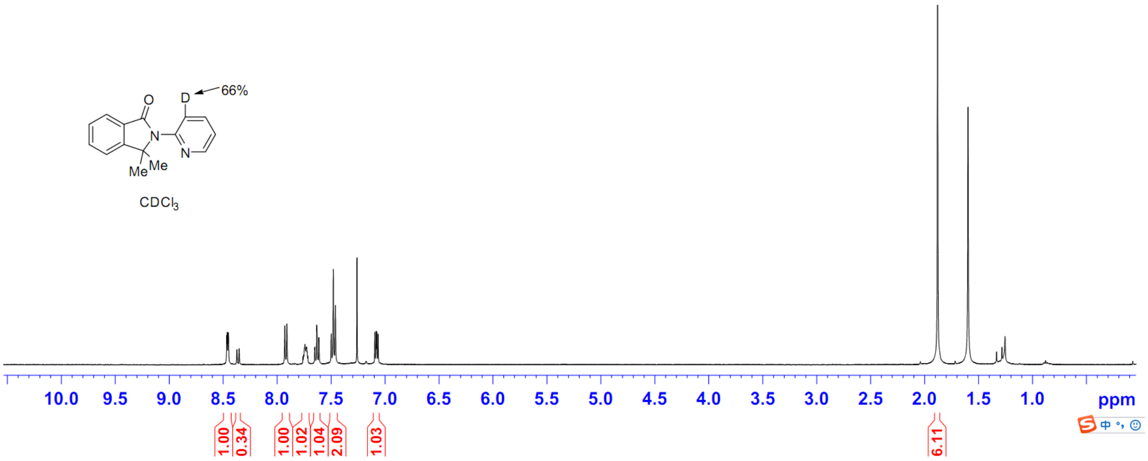
(Eq 1) In a 25 mL Schlenk tube was changed of **1d** (0.2 mmol, 1.0 equiv), D_2O (2 equiv), $Cp^*Rh(CH_3CN)_3(SbF_6)_2$ (5 mol%) and AgOAc (2.2 equiv) in DCE (1 mL). The mixture was stirred at 100 ^{o}C for 24 h. After cooling to room temperature, the reaction mixture was directly loaded onto a silica gel column which was then eluted with petroleum ether/ethyl acetate (PE/EA = 5/1, v/v) to afford the **d-1d**.



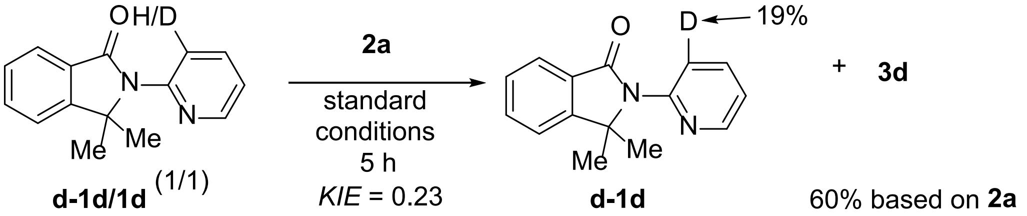


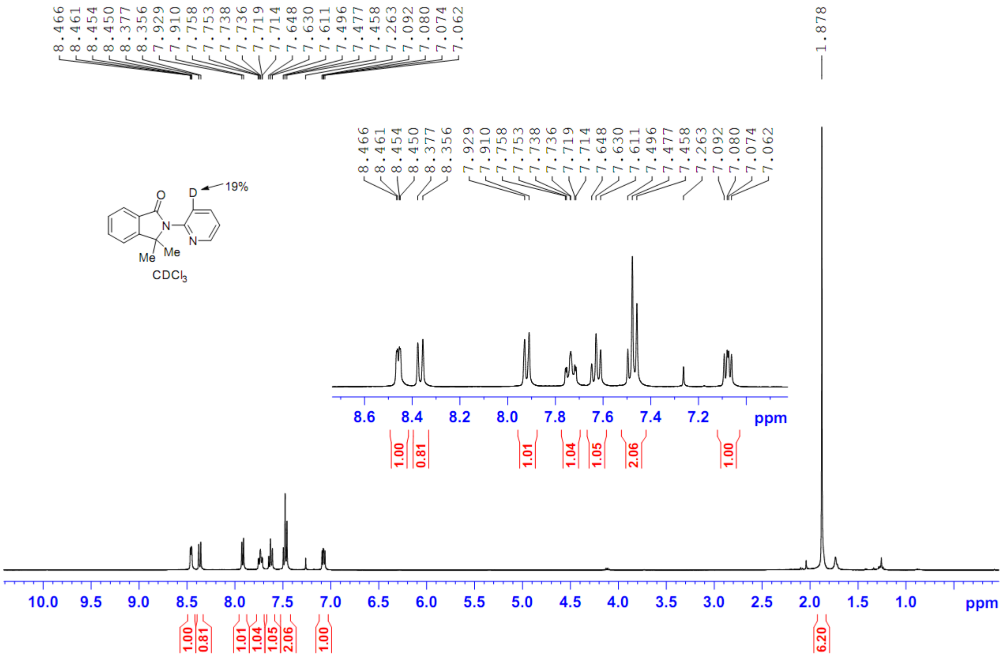
(eq 2) In a 25 mL Schlenk tube was changed of **1d** (0.2 mmol, 1.0 equiv), **2a** (2.4 equiv), D_2O (2 equiv), $Cp^*Rh(CH_3CN)_3(SbF_6)_2$ (5 mol%) and AgOAc (2.2 equiv) in DCE (1 mL). The mixture was stirred at 100 ^{o}C for 24 h. After cooling to room temperature, the reaction mixture was directly loaded onto a silica gel column which was then eluted with petroleum ether/ethyl acetate (PE/EA = 5/1, v/v) to afford the **d-1d** (30%) and **3d** (65%).



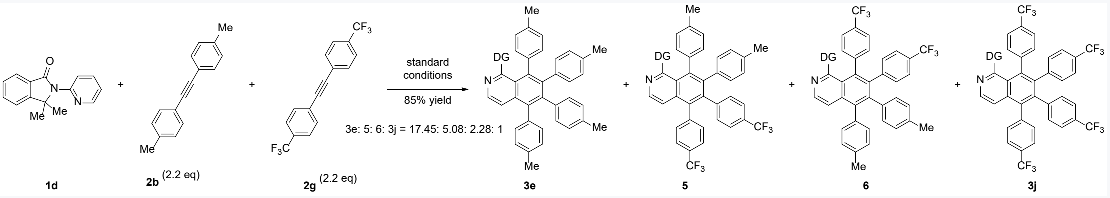


(eq 3) According to the standard reaction condition, the competitive reactions were conducted by employing 1:1 ratio of **1d** (23.8 mg, 0.1 mmol, 1 equiv.) and **d-1d** (23.9 mg, 0.1 mmol, 1 equiv.) and **2a** (1 equiv). The mixture was stirred at 100 °C for 5 h. The starting material **1d** and **d-1d** were recovered and detected by ^{1}H NMR.





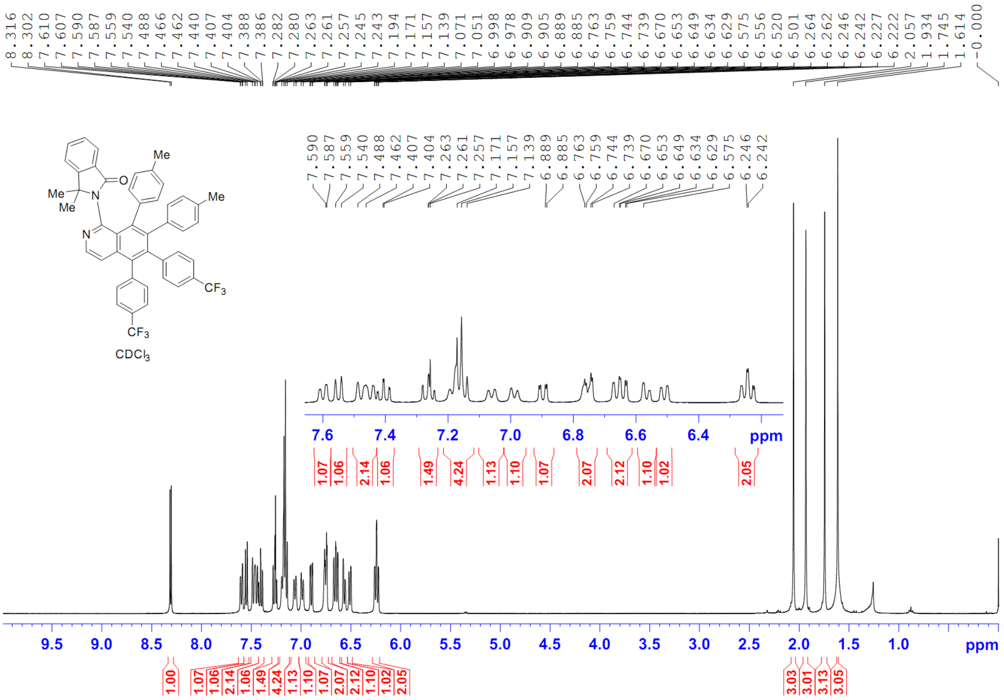
(eq 4) In a 25 mL Schlenk tube was changed of **1d** (0.2 mmol, 1.0 equiv), **2b** (2.2 equiv), **2g** (2.2 equiv), Cp*Rh(CH$_3$CN)$_3$(SbF$_6$)$_2$ (5 mol%) and AgOAc (2.2 equiv) in DCE (1 mL). The mixture was stirred at 100 ^{o}C for 24 h. After cooling to room temperature, the reaction mixture was filtered by a short silica gel column to remove the inorganic solid. The filtrate was concentrated and further purified by prep-HPLC. The ratio of four products was determined by HPLC analysis.

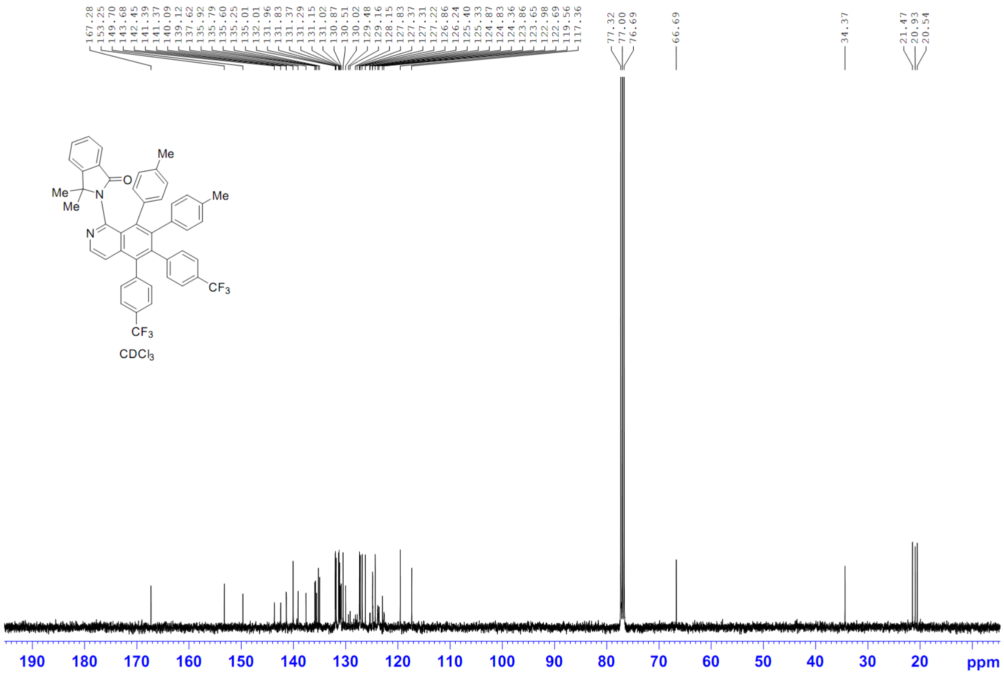


2-(7,8-di-p-tolyl-5,6-bis(4-(trifluoromethyl)phenyl)isoquinolin-1-yl)-3,3-dimethylisoindolin-1-one (5): White solid, R$_f$ = 0.43, (PE/EA = 10/1, v/v). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.31 (d, *J* = 5.6 Hz, 1H), 7.60 (dd, *J* = 8.0, 1.9 Hz, 1H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.49-7.44 (m, 2H), 7.41 (td, *J* = 7.5, 1.2 Hz, 1H), 7.28-7.24

(m, 1H), 7.19-7.14 (m, 4H), 7.06 (d, J = 8.0 Hz, 1H), 6.99 (d, J = 8.0 Hz, 1H), 6.90 (dd, J = 8.0, 2.0 Hz, 1H), 6.76-6.74 (m, 2H), 6.67-6.63 (m, 2H), 6.57 (d, J = 7.8 Hz, 1H), 6.51 (d, J = 7.9 Hz, 1H), 6.24 (td, J = 7.7, 1.9 Hz, 2H), 2.06 (s, 3H), 1.93 (s, 3H), 1.74 (s, 3H), 1.61 (s, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 167.28, 153.25, 149.70, 143.68, 142.45, 141.39, 141.37, 140.09, 139.12, 137.62, 135.92, 135.79, 135.60, 135.24, 135.01, 132.01, 131.96, 131.83, 131.37, 131.29, 131.15, 131.02, 130.87, 130.51, 130.02, 129.48, 129.16, 128.15, 127.83, 127.37, 127.31, 127.22, 126.86, 126.24, 125.36 (q, J = 7 Hz), 124.85 (q, J = 3.9 Hz), 124.36, 123.86, 123.65, 122.98, 122.69, 119.56, 117.36, 66.69, 34.37, 21.47, 20.93, 20.54. ^{19}F NMR (376 MHz, CDCl$_3$) δ -62.62 (s), -62.67 (s). HRMS (ESI) Calcd for $C_{47}H_{34}F_6N_2ONa$ [M+Na]$^+$ 779.2473, Found 779.2459.

2-(5,6-di-p-tolyl-7,8-bis(4-(trifluoromethyl)phenyl)isoquinolin-1-yl)-3,3-dimethylisoindolin-1-one (6): Oil, R$_f$ = 0.56, (PE/EA = 10/1, v/v). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.33 (d, J = 5.7 Hz, 1H), 7.50 (d, J = 7.5 Hz, 1H), 7.42 (td, J = 7.5, 1.2 Hz, 1H), 7.36 (d, J = 5.6 Hz, 1H), 7.27-7.21 (m, 2H), 7.14 (t, J = 6.6 Hz, 4H), 7.04-7.00 (m, 3H), 6.95 (t, J = 7.8 Hz, 3H), 6.76-6.69 (m, 3H), 6.60 (d, J = 7.9 Hz, 1H), 6.46 (t, J = 8.4 Hz, 2H), 2.33 (s, 3H), 2.09 (s, 3H), 1.93 (s, 3H), 1.57 (s, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 167.58, 153.02, 149.12, 140.27, 138.89, 138.63, 136.51, 136.16, 136.03, 135.45, 135.36, 132.49, 132.33, 131.84, 131.63, 131.37, 131.02, 130.63, 129.45, 128.54, 128.51, 127.28, 123.97, 123.21, 119.59, 118.53, 66.77, 34.45, 21.43, 21.22, 20.98. ^{19}F NMR (376 MHz, CDCl$_3$) δ -62.71 (s), -62.97 (s). HRMS (ESI) Calcd for $C_{47}H_{34}F_6N_2ONa$ [M+Na]$^+$ 779.2473, Found 779.2465.

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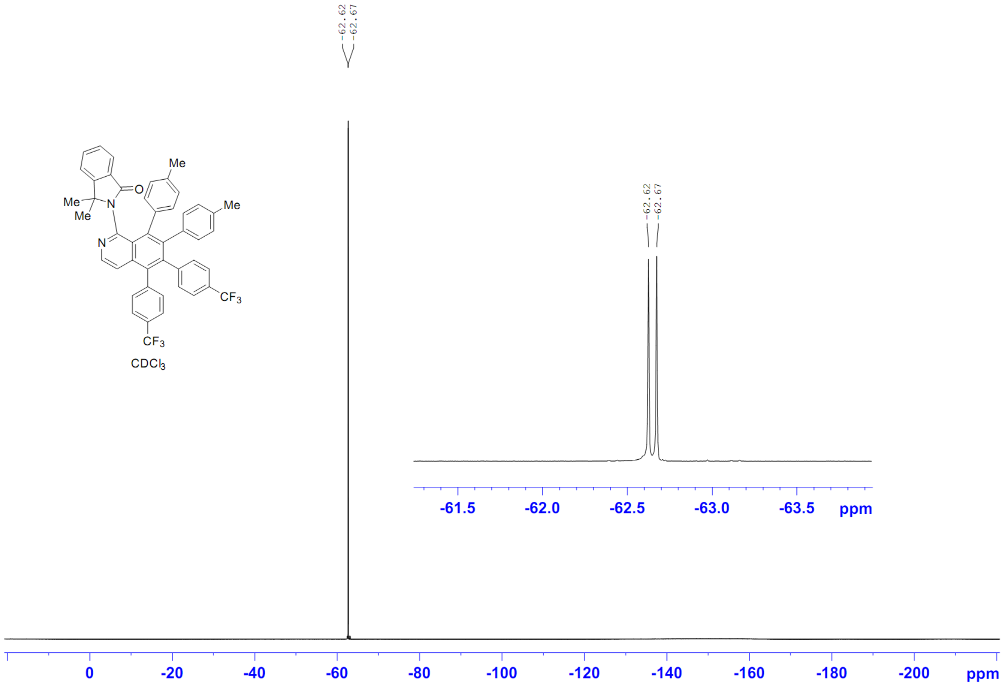
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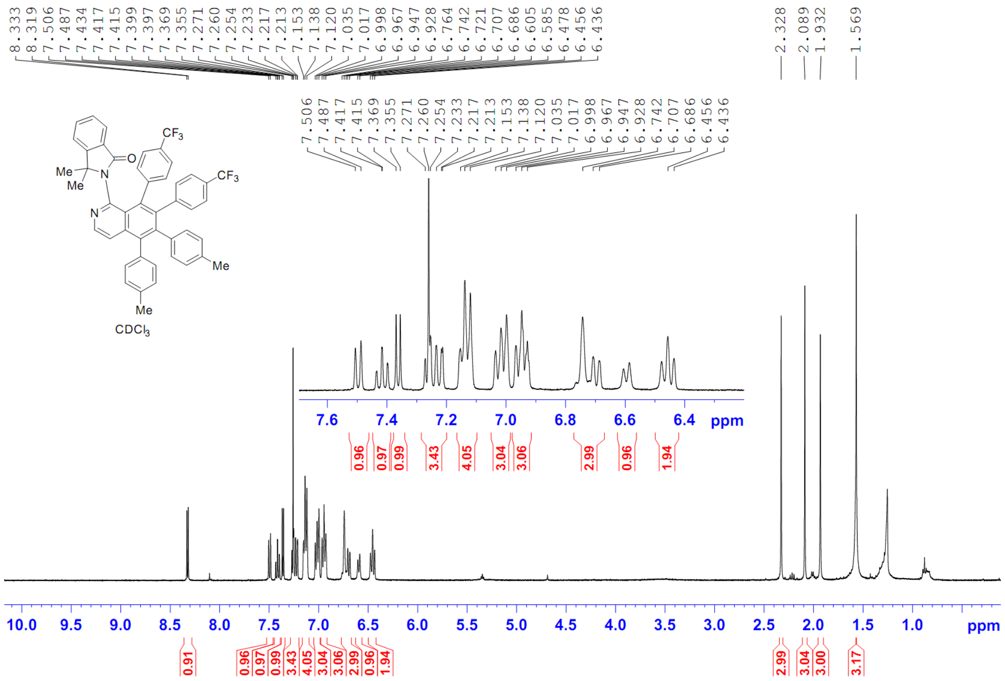
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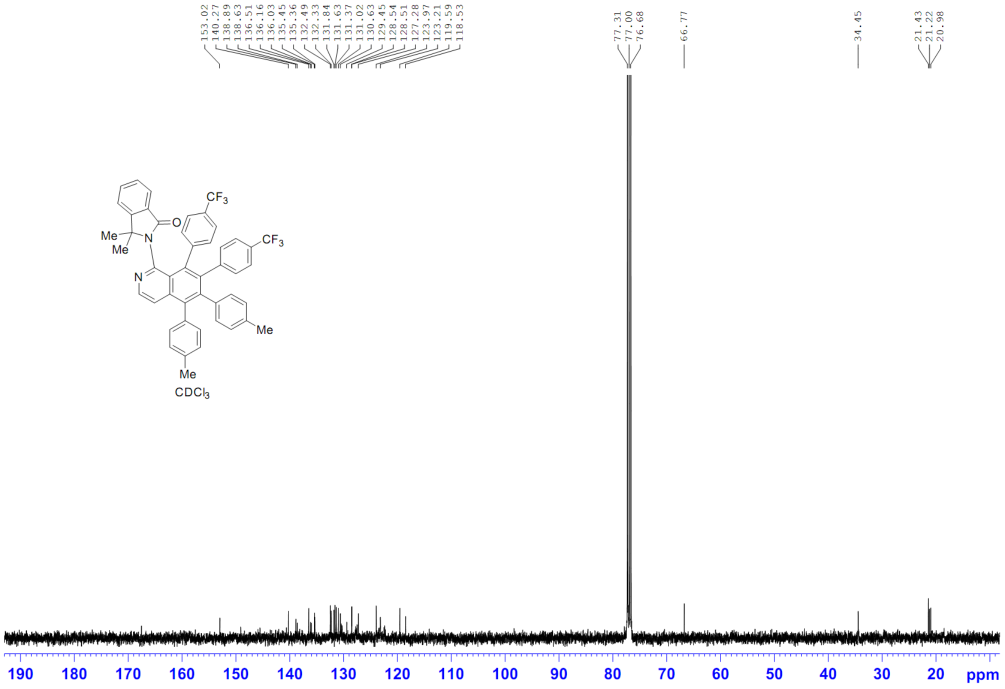
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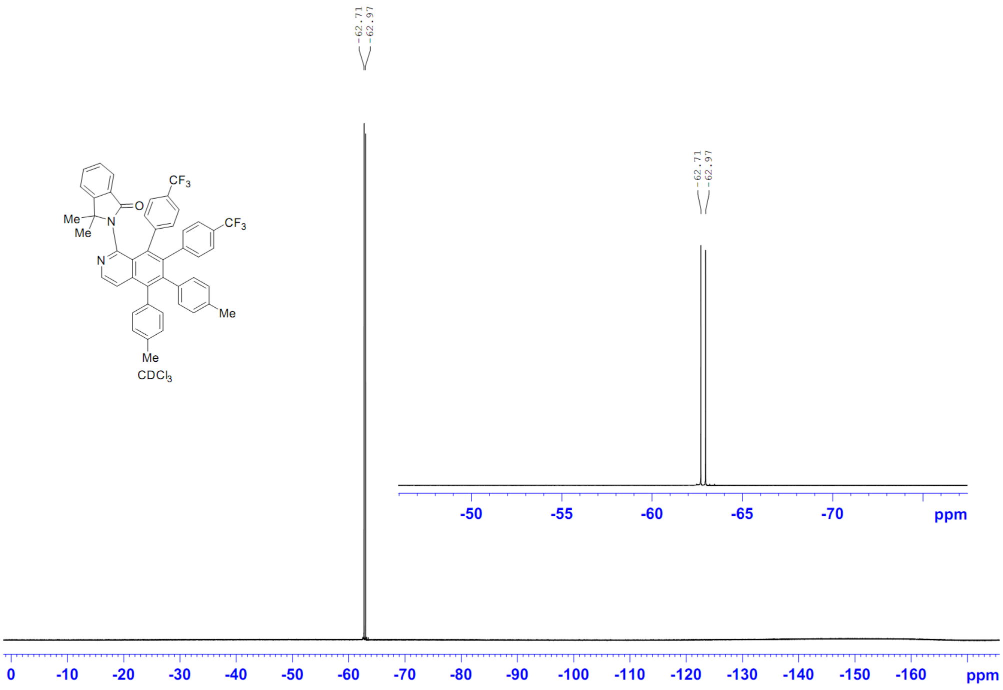
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