Supporting Information

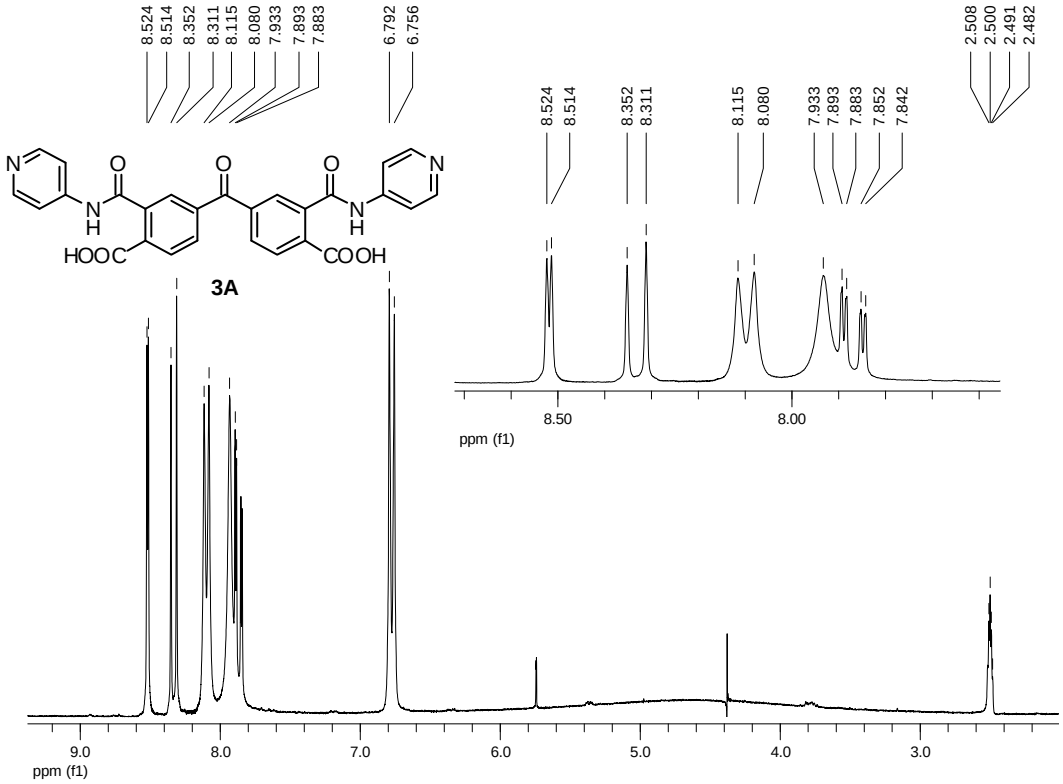
A novel and highly regioselective synthesis of new carbamoylcarboxylic acids from dianhydrides

Adrián Ochoa-Teran[1]*, Jesús Estrada-Manjarrez[2], Marisela Martínez-Quiroz[1], Marco Antonio Landey-Álvarez[1], Eleazar Alcántar Zavala[3], Georgina Pina-Luis[1], Hisila Santacruz-Ortega[4], Luis Enrique Gómez-Pineda[1], José Z. Ramírez[4], Daniel Chávez[1], Julio Montes Ávila[3], Victoria Labastida-Galván[5] and Mario Ordoñez[5]

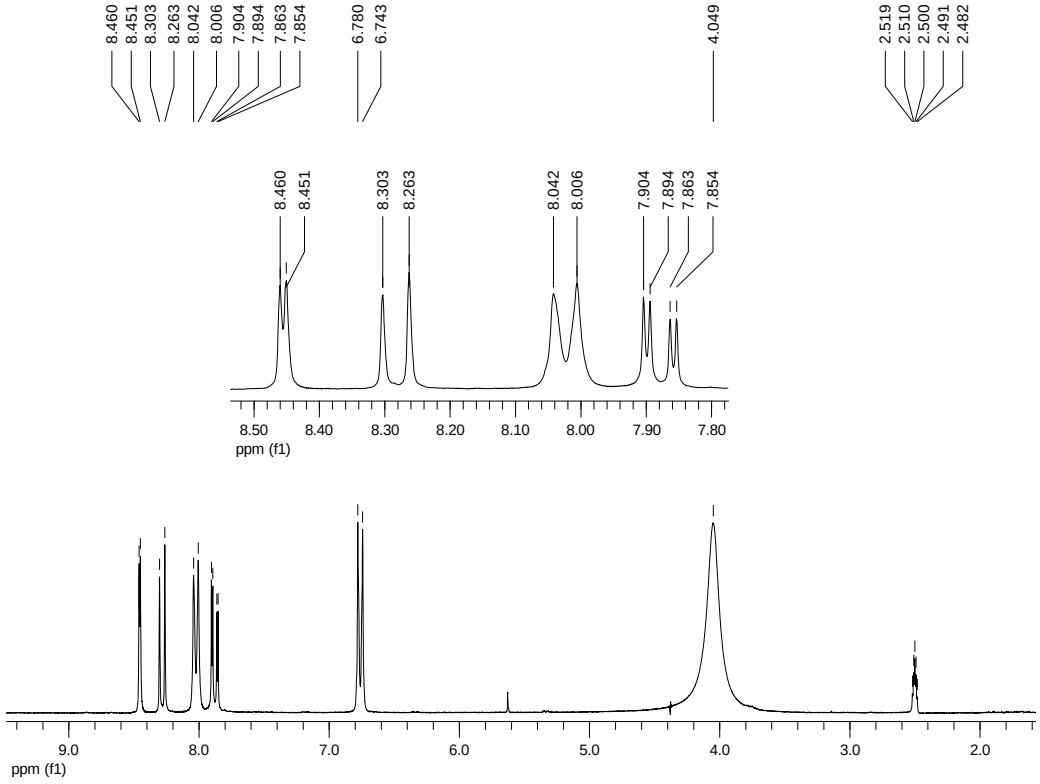
[1] Centro de Graduados e Investigación, Instituto Tecnológico de Tijuana. Tijuana, B. C., 22510, México.
[2] Departamento de Ingeniería Bioquímica, Instituto Tecnológico de Culiacán. Culiacán, Sin., 80220. México.
[3] Facultad de Ciencias Químico Biológicas, Universidad Autónoma de Sinaloa, Culiacán, Sin. 80010, México.
[4] Departamento de Polímeros y Materiales, Universidad de Sonora, Hermosillo, Son. 83000, México.
[5] Centro de Investigaciones Químicas, Universidad Autónoma del Estado de Morelos. Cuernavaca, Mor. 62209. México.

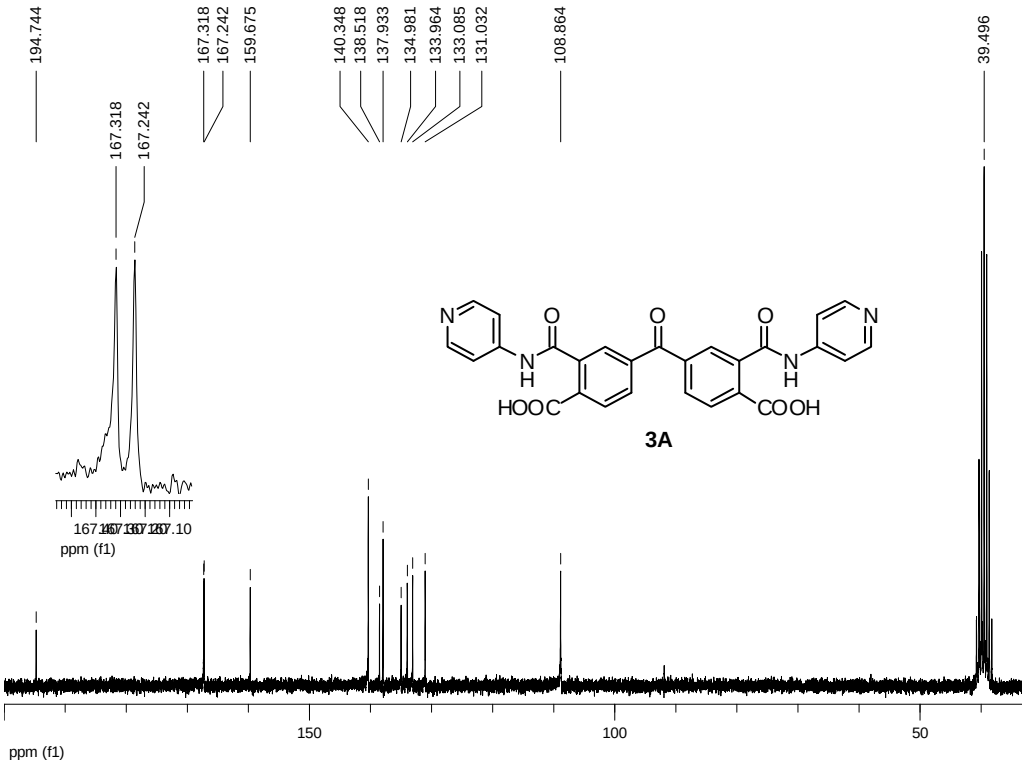
Content **Page No.**

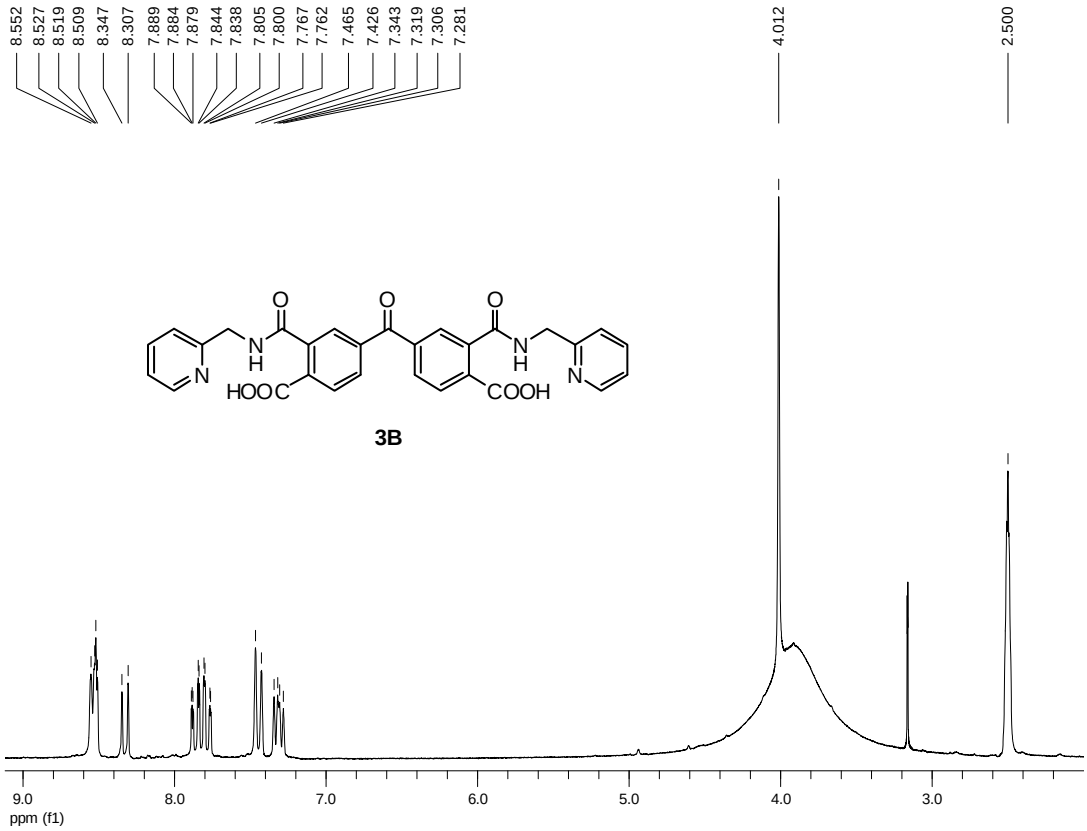
I. Selected NMR spectra of compounds 3

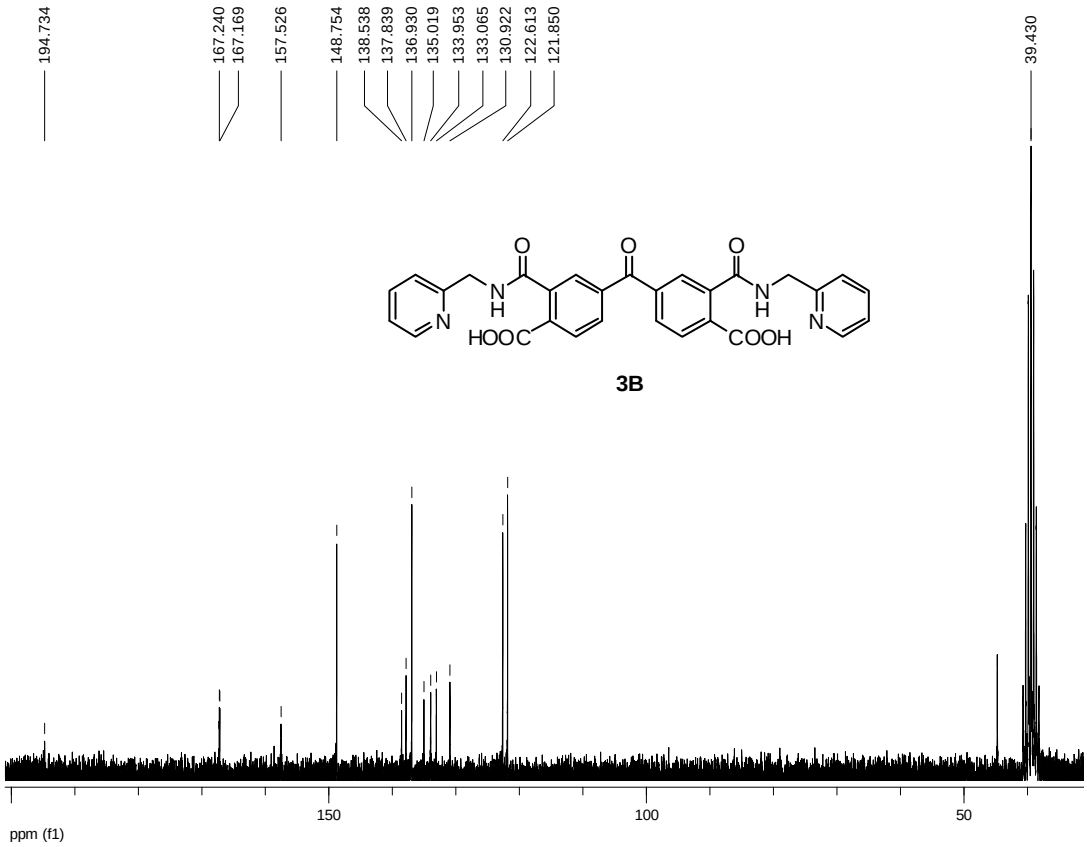


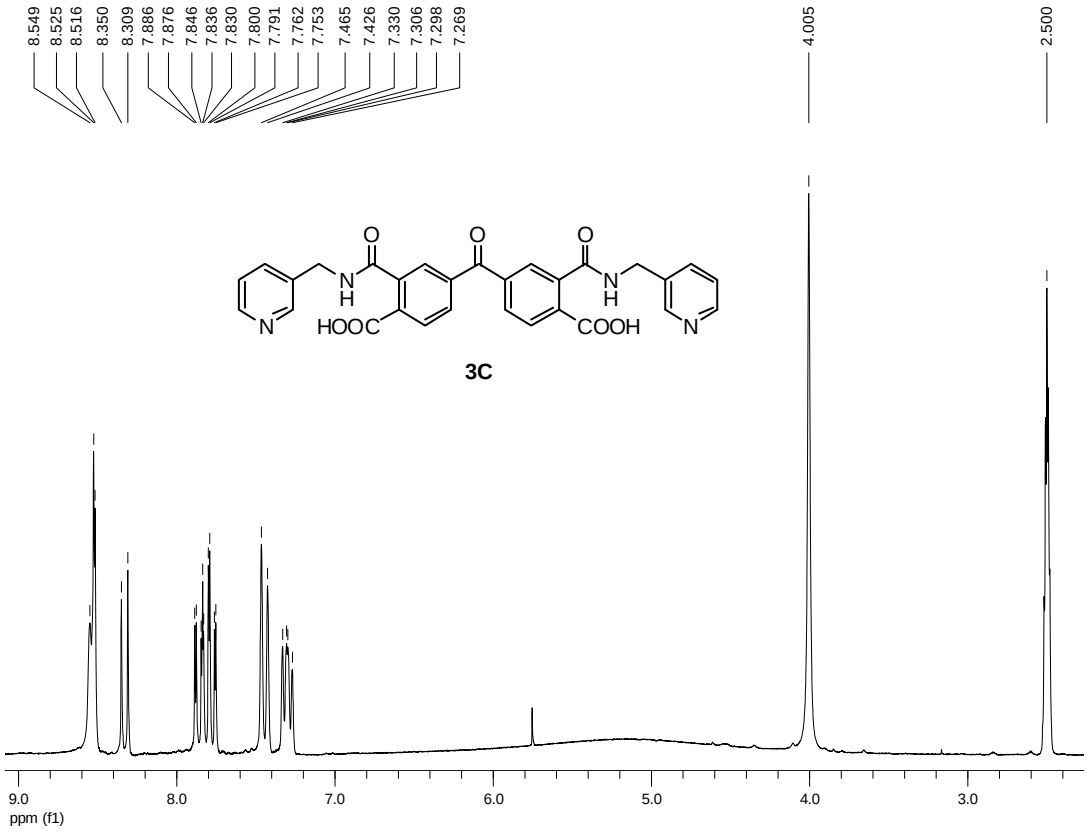
With two drops of D_2O



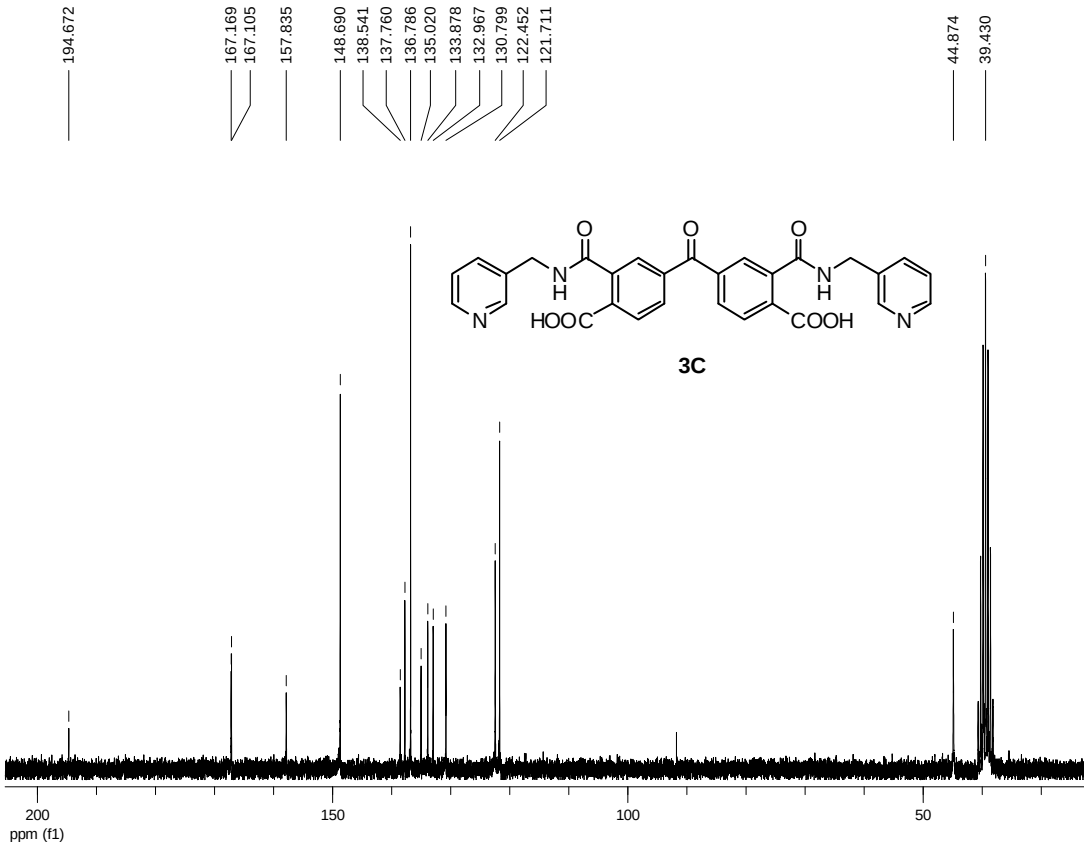

194.744
167.318
167.242
159.675
140.348
138.518
137.933
134.981
133.964
133.085
131.032
108.864
39.496
ppm (f1)
150
100
50
ppm (f1)
HOOC
COOH
3A


8.552
8.527
8.519
8.509
8.347
8.307
7.889
7.884
7.879
7.844
7.838
7.805
7.800
7.767
7.762
7.465
7.426
7.343
7.319
7.306
7.281
4.012
2.500
O
O
O
N
H
N
H
N
N
HOOC
COOH
3B
9.0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (f1)

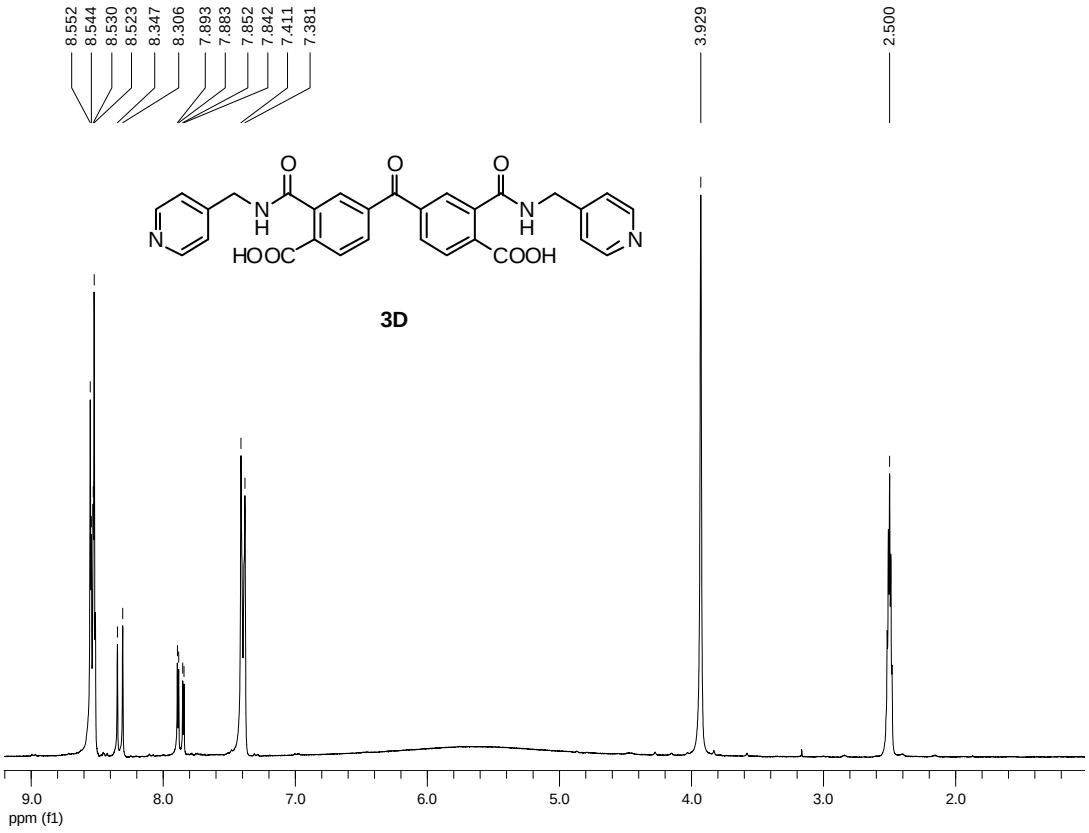

194.734
167.240
167.169
157.526
148.754
138.538
137.839
136.930
135.019
133.953
133.065
130.922
122.613
121.850
39.430
O
O
O
N
H
N
H
N
N
HOOC
COOH
3B
150
100
50
ppm (f1)


8.549
8.525
8.516
8.350
8.309
7.886
7.876
7.846
7.836
7.830
7.800
7.791
7.762
7.753
7.465
7.426
7.330
7.306
7.298
7.269
4.005
2.500
HOOC
COOH
9.0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (f1)

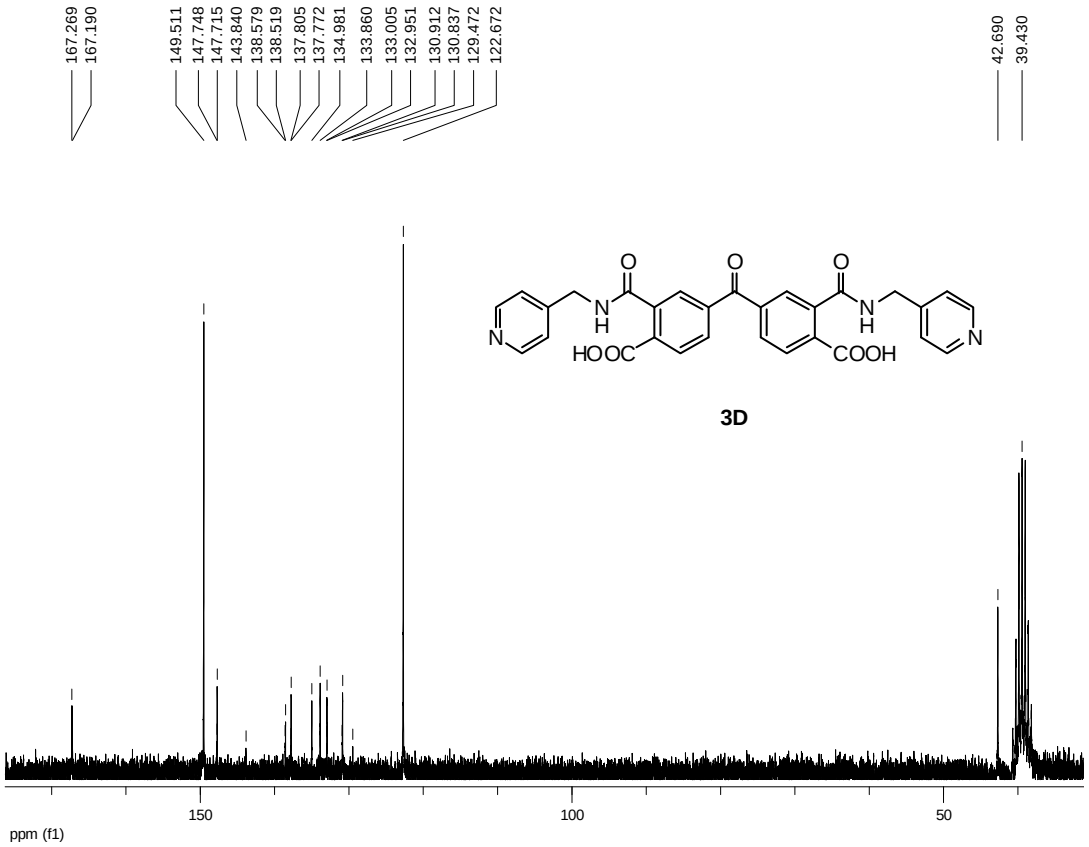
3C


194.672
167.169
167.105
157.835
148.690
138.541
137.760
136.786
135.020
133.878
132.967
130.799
122.452
121.711
44.874
39.430
HOOC
COOH
200
150
100
50
ppm (f1)

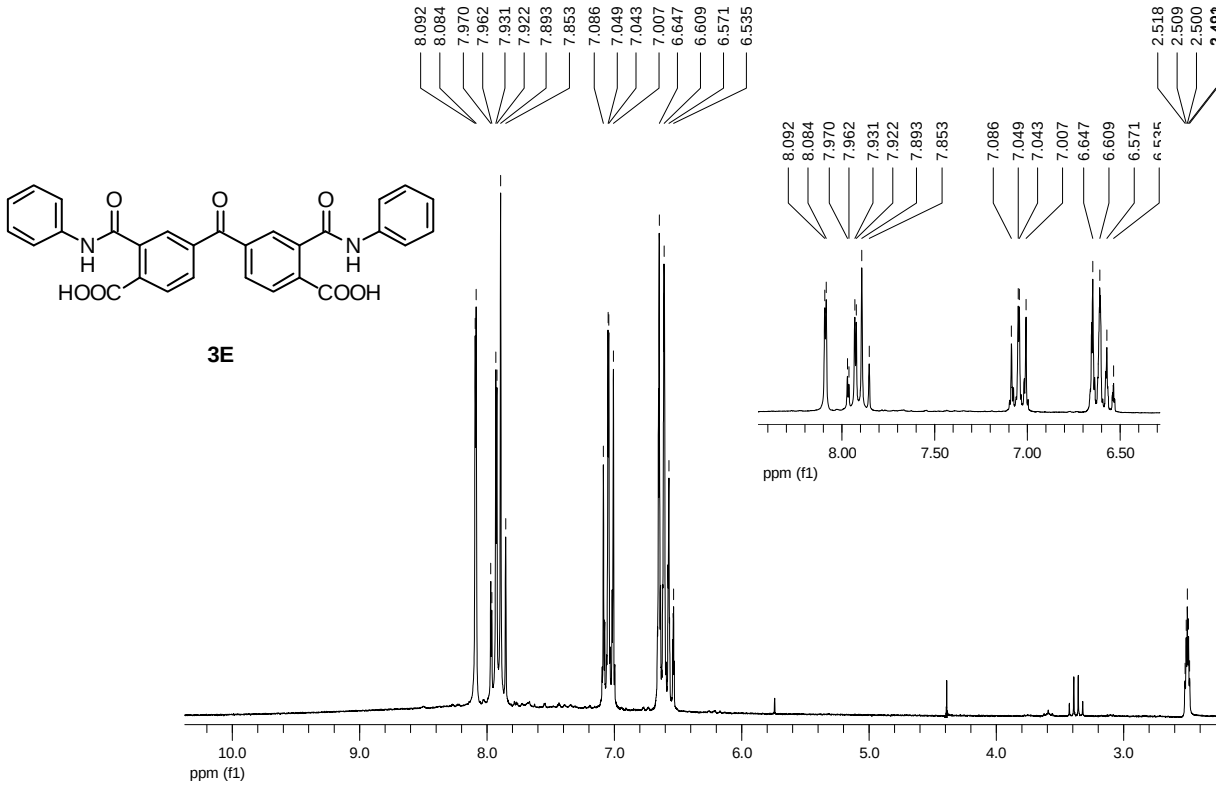
3C


8.552
8.544
8.530
8.523
8.347
8.306
7.893
7.883
7.852
7.842
7.411
7.381
3.929
2.500
HOOC
COOH
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)

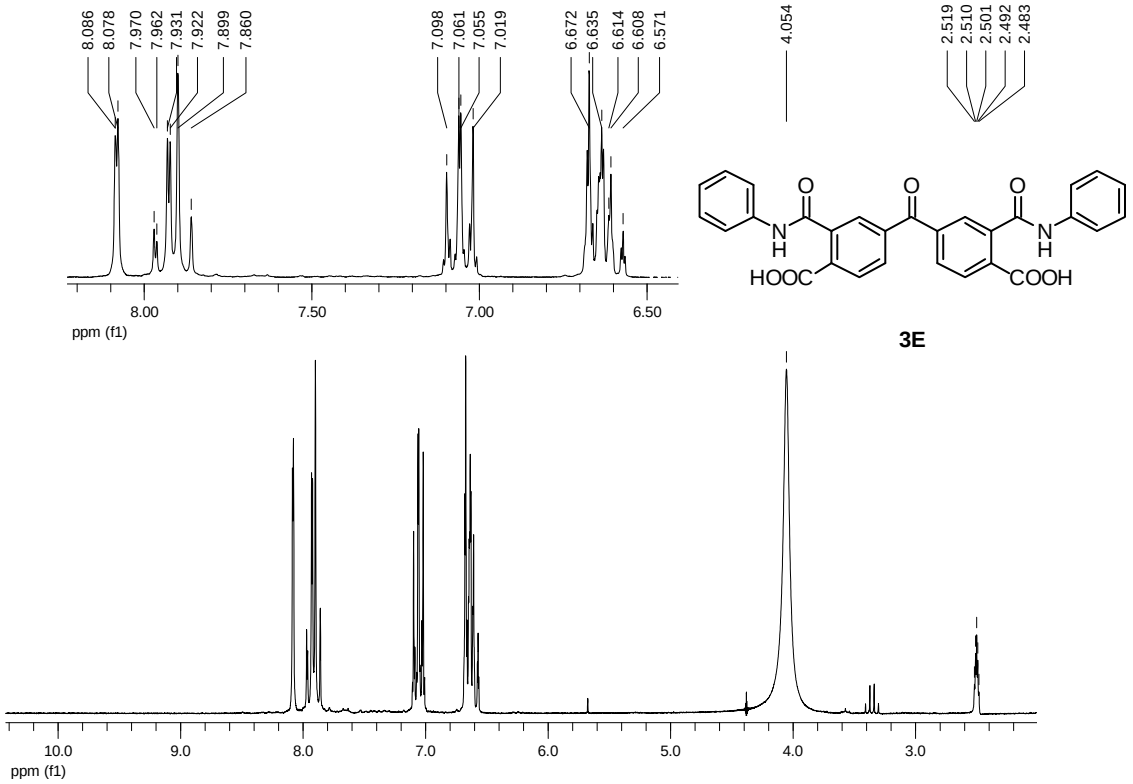
3D

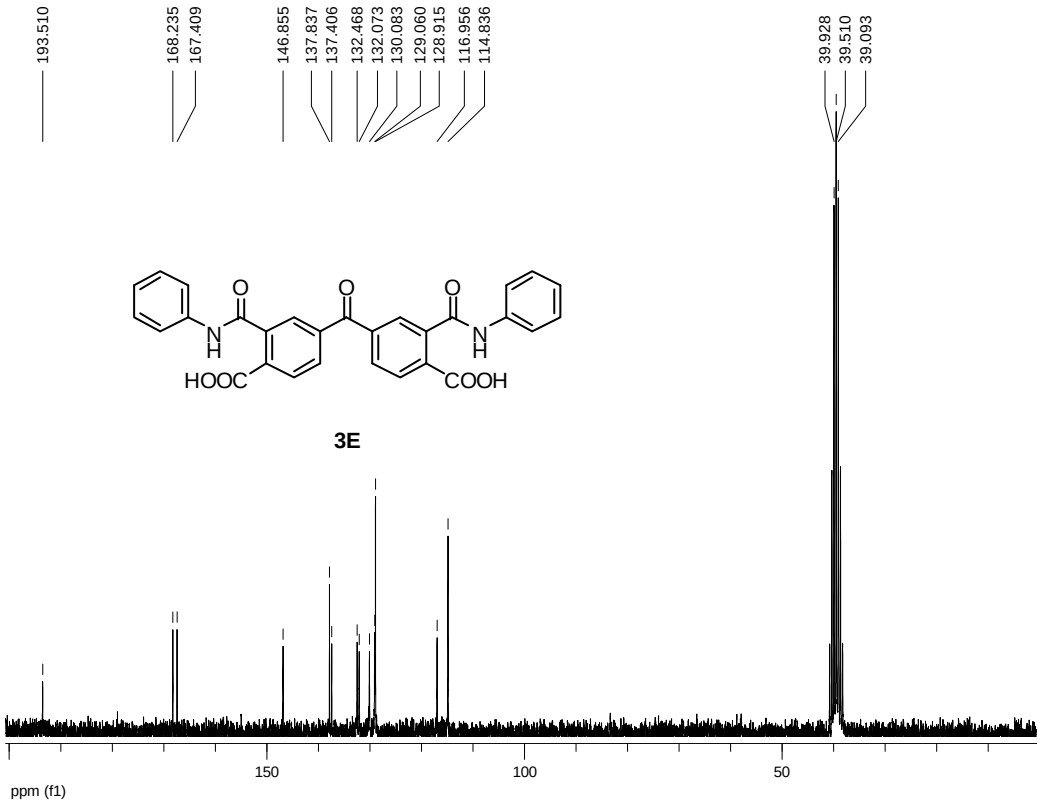

167.269
167.190
149.511
147.748
147.715
143.840
138.579
138.519
137.805
137.772
134.981
133.860
133.005
132.951
130.912
130.837
129.472
122.672
42.690
39.430
HOOC
COOH
150
100
50
ppm (f1)

3D



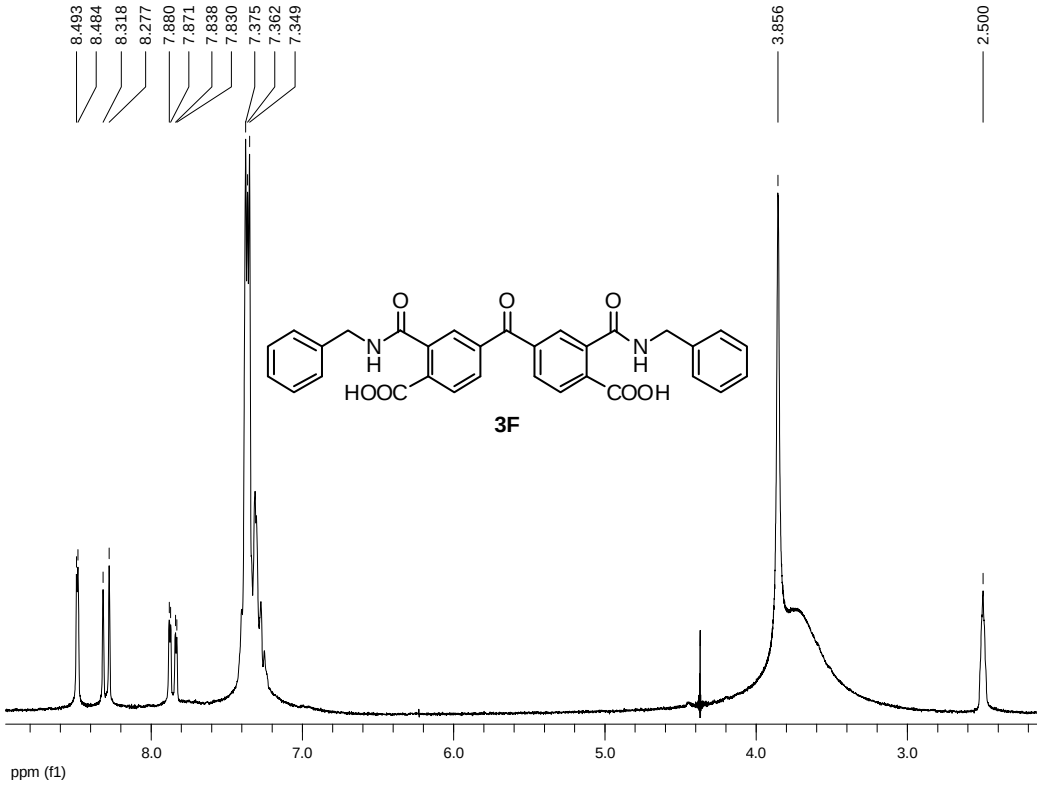
With one drop of D_2O

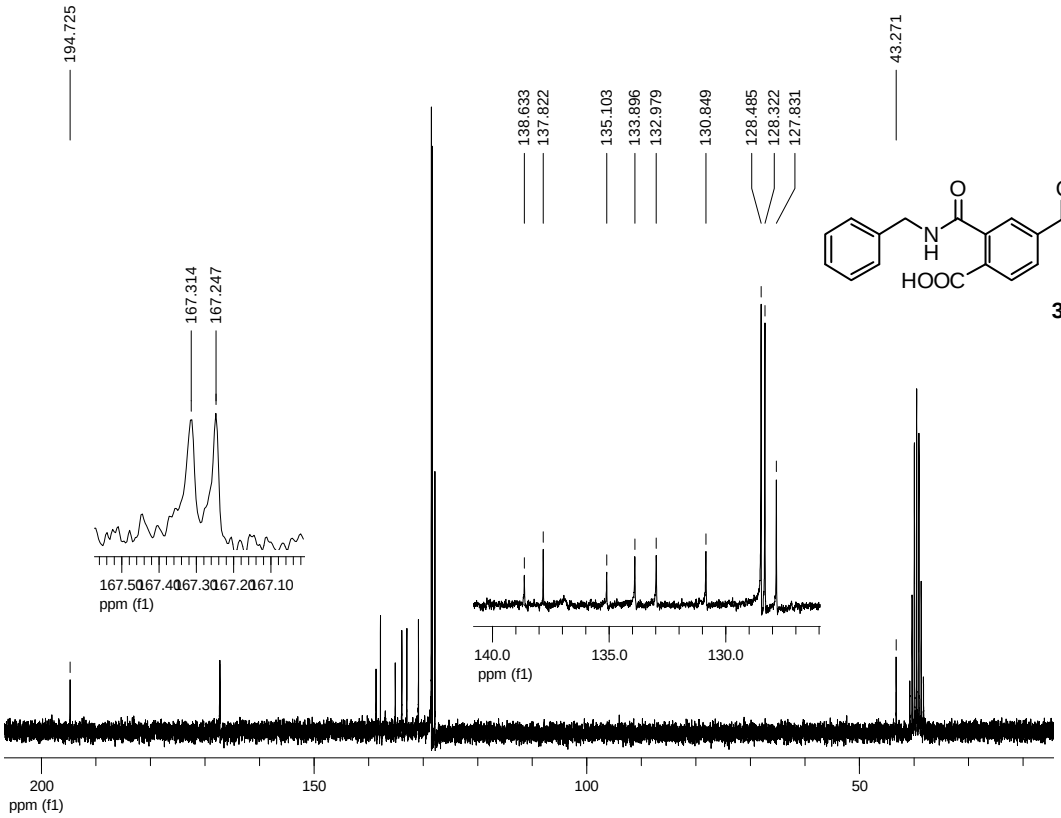



193.510
168.235
167.409
146.855
137.837
137.406
132.468
132.073
130.083
129.060
128.915
116.956
114.836
39.928
39.510
39.093
O
O
O
N
H
N
H
HOOC
COOH
150
100
50
ppm (f1)

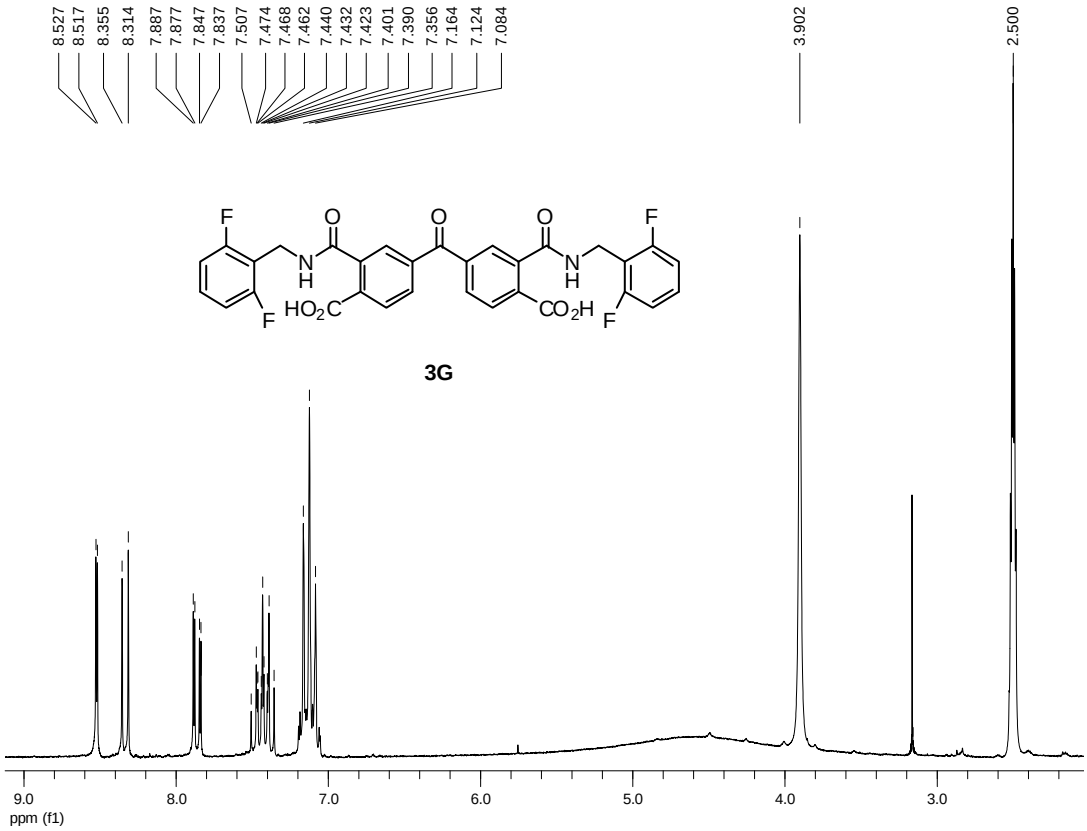
3E

With one drop of D_2O

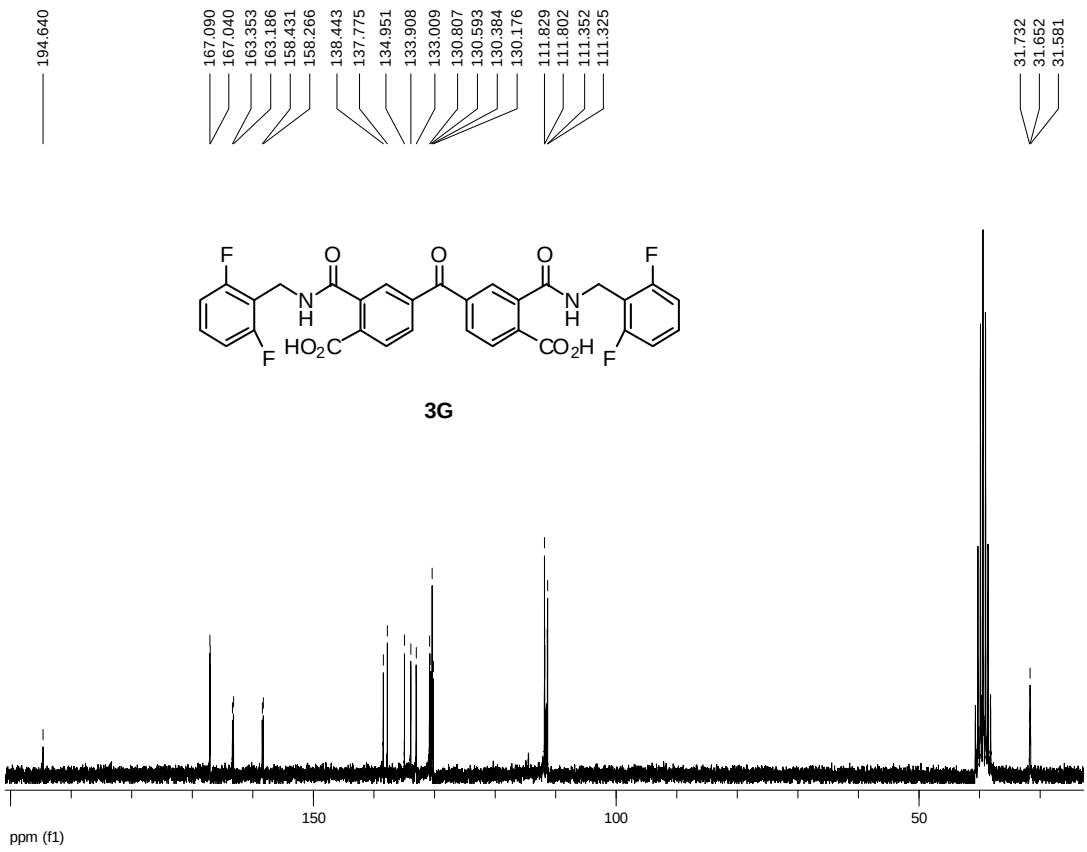



194.725
138.633
137.822
135.103
133.896
132.979
130.849
128.485
128.322
127.831
43.271
167.314
167.247
167.50
167.40
167.30
167.20
167.10
ppm (f1)
140.0
135.0
130.0
ppm (f1)
200
150
100
50
ppm (f1)
O
O
O
N
H
N
H
HOOC
COOH

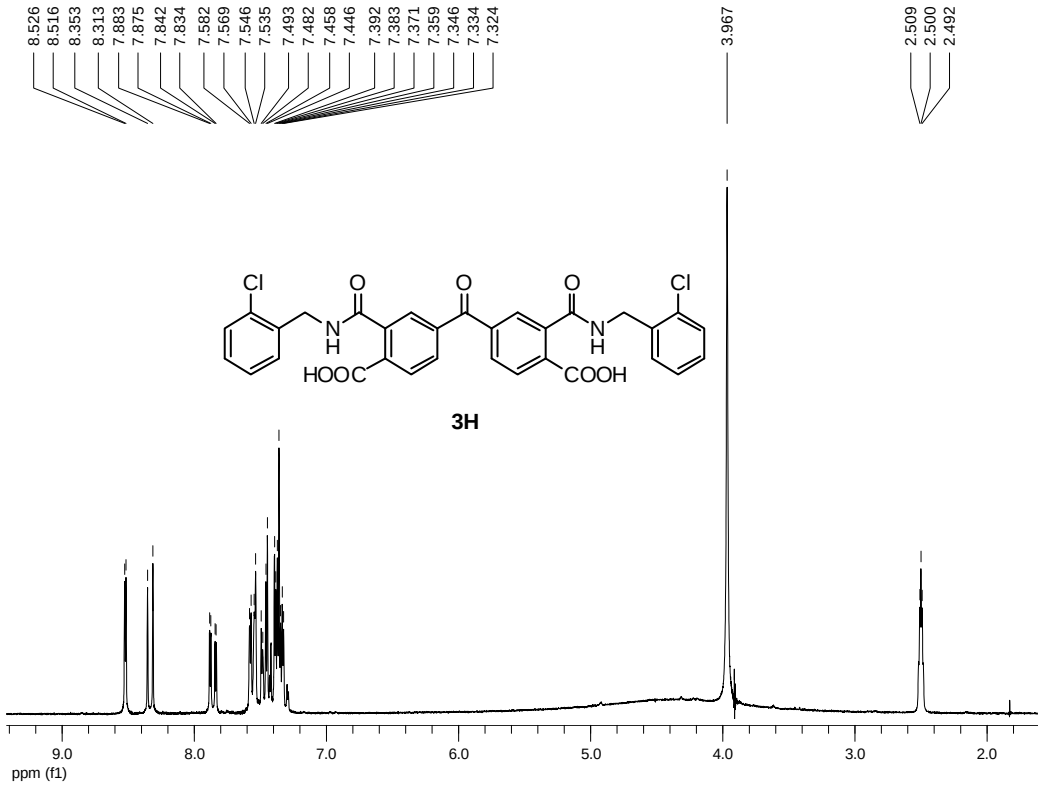
3F


8.527
8.517
8.355
8.314
7.887
7.877
7.847
7.837
7.507
7.474
7.468
7.462
7.440
7.432
7.423
7.401
7.390
7.356
7.164
7.124
7.084
3.902
2.500
F
O
O
O
F
N
H
N
H
F
HO2C
CO2H
F
9.0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (f1)

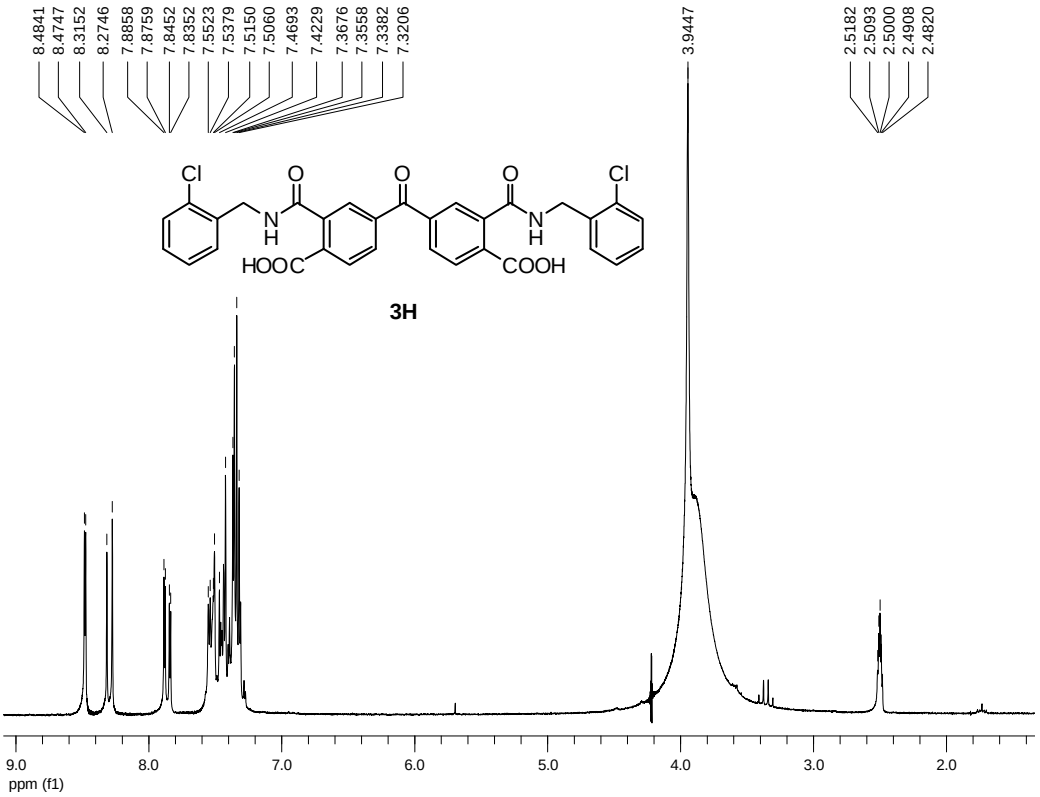
3G

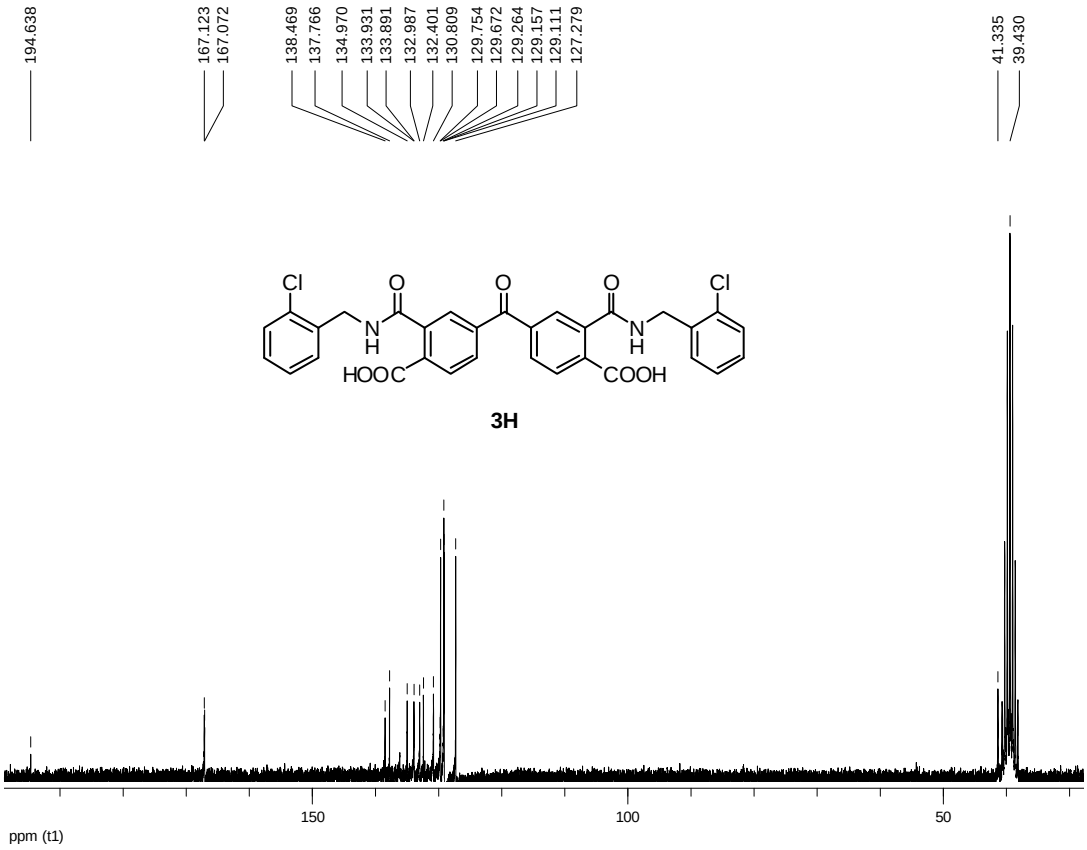

194.640
167.090
167.040
163.353
163.186
158.431
158.266
138.443
137.775
134.951
133.908
133.009
130.807
130.593
130.384
130.176
111.829
111.802
111.352
111.325
31.732
31.652
31.581
F
O
O
O
F
N
H
N
H
F
HO2C
CO2H
F
150
100
50
ppm (f1)

3G

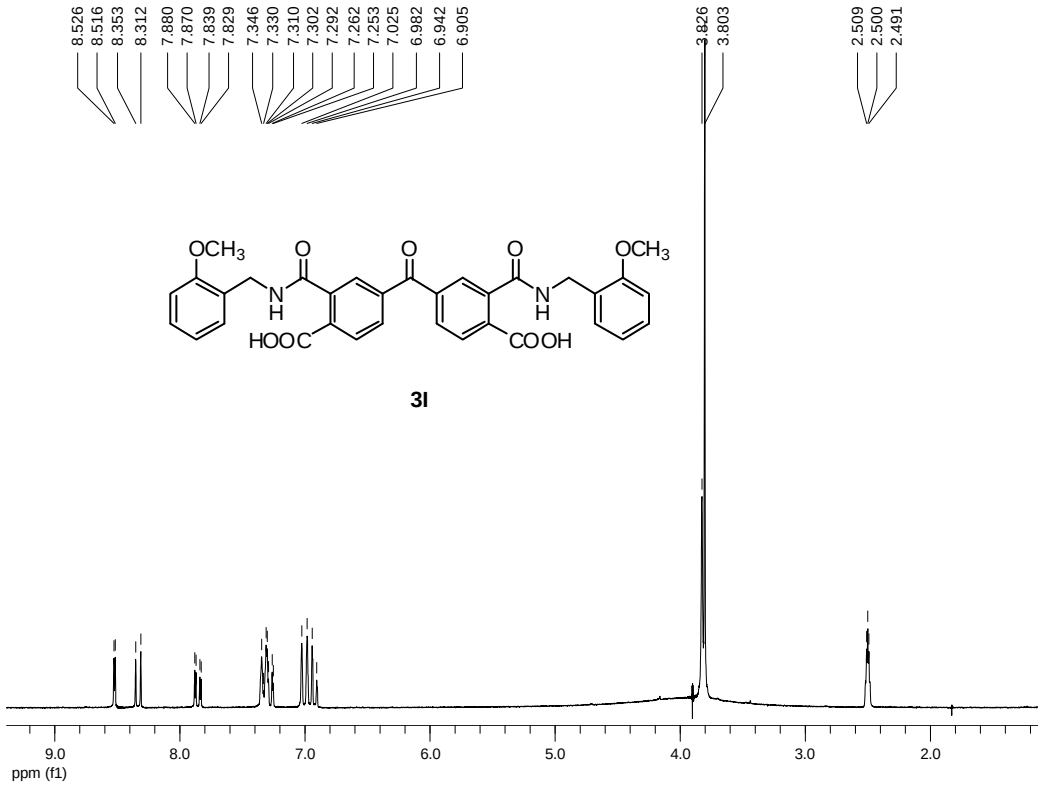


With two drops of D_2O added

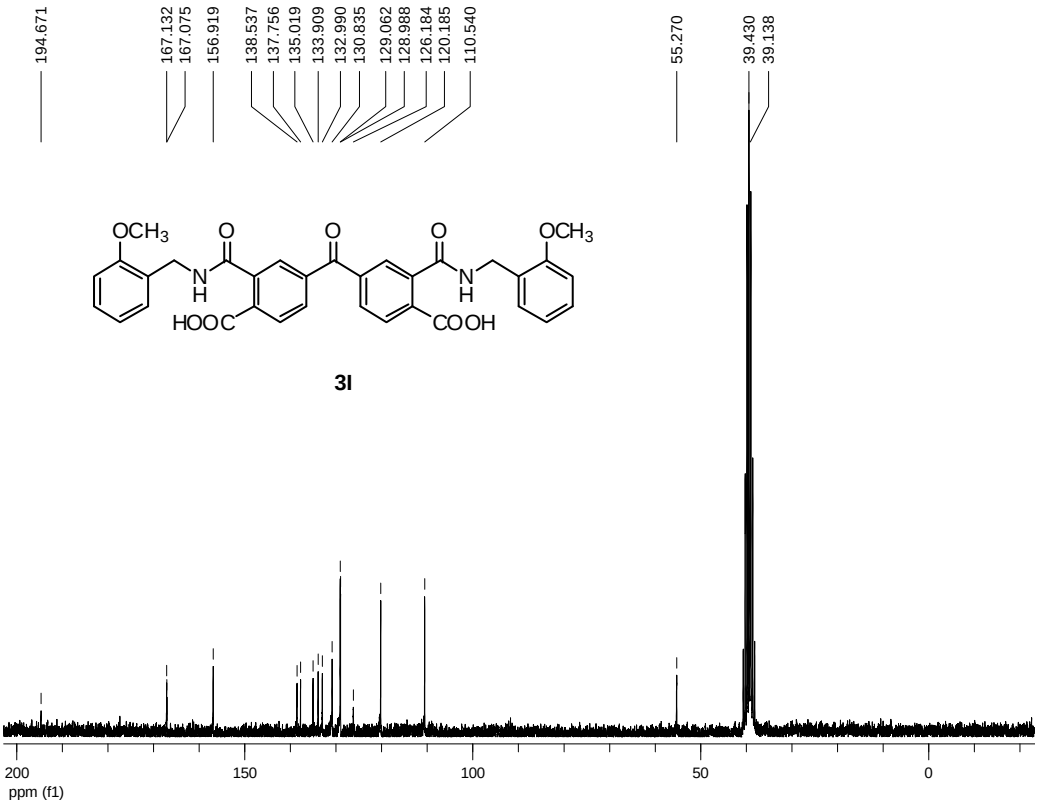



194.638
167.123
167.072
138.469
137.766
134.970
133.931
133.891
132.987
132.401
130.809
129.754
129.672
129.264
129.157
129.111
127.279
41.335
39.430
Cl
O
O
O
Cl
N
H
N
H
HOOC
COOH
150
100
50
ppm (t1)

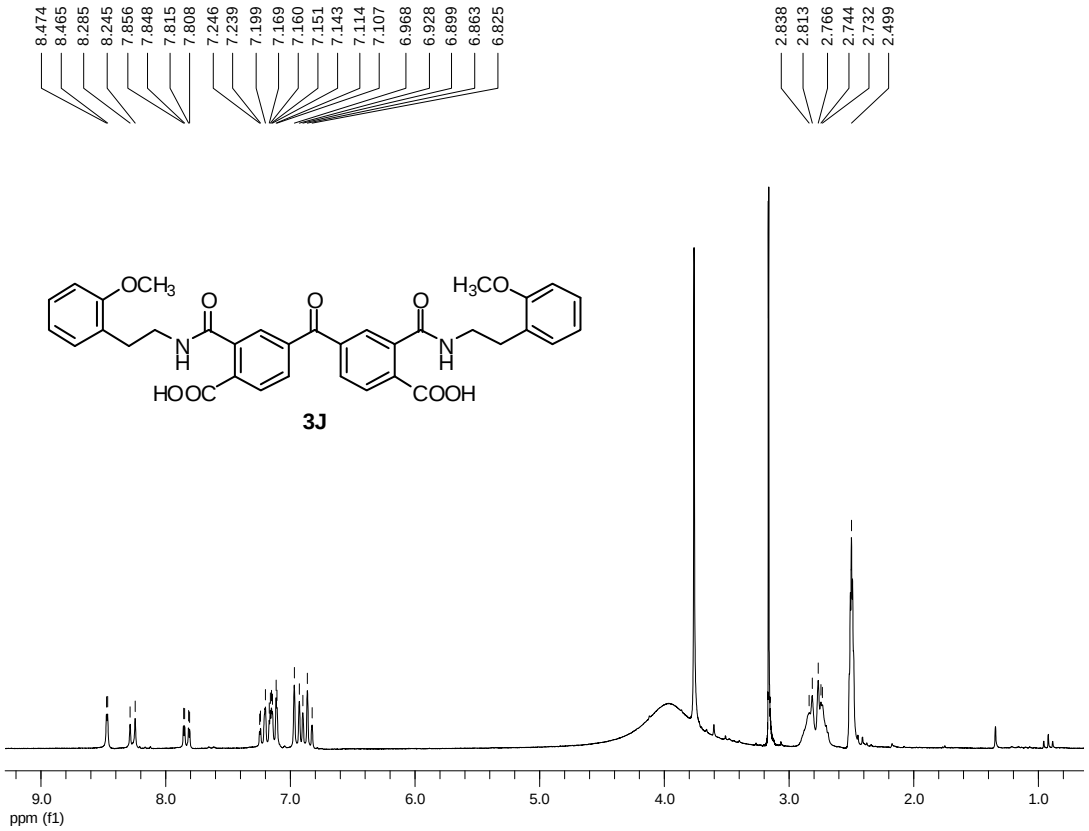
3H

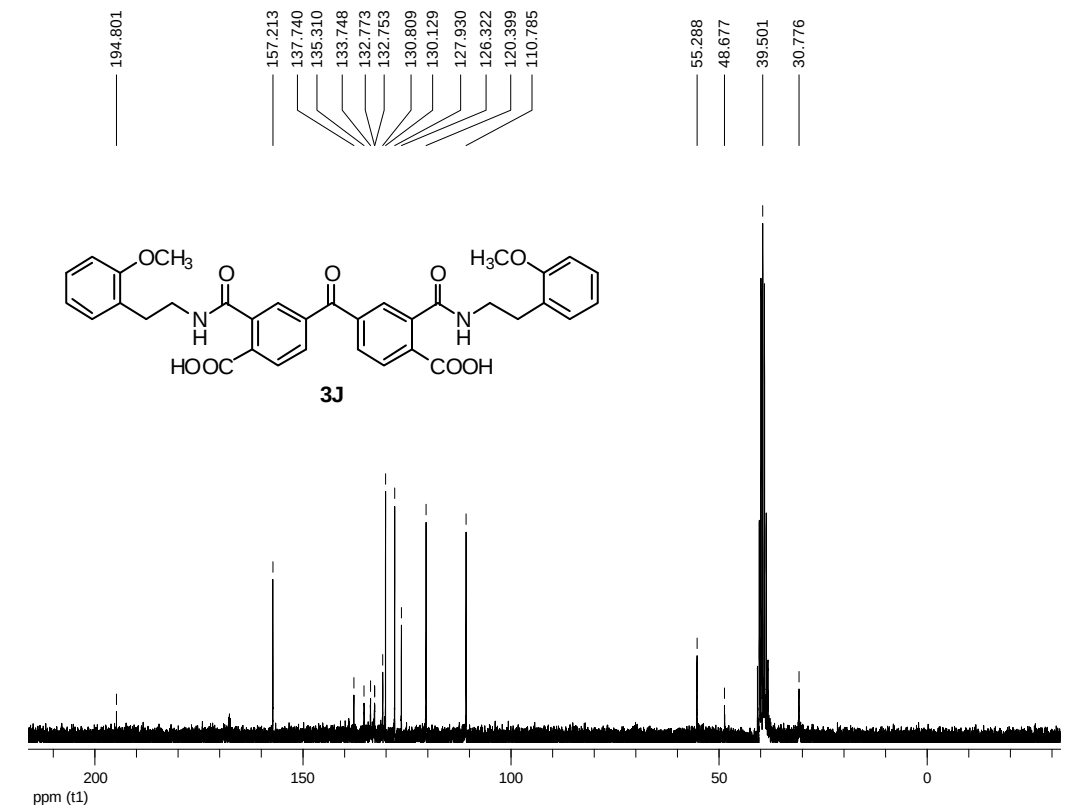

8.526
8.516
8.353
8.312
7.880
7.870
7.839
7.829
7.346
7.330
7.310
7.302
7.292
7.262
7.253
7.025
6.982
6.942
6.905
3.826
3.803
2.509
2.500
2.491
OCH3
O
O
O
OCH3
N
H
N
H
HOOC
COOH
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)

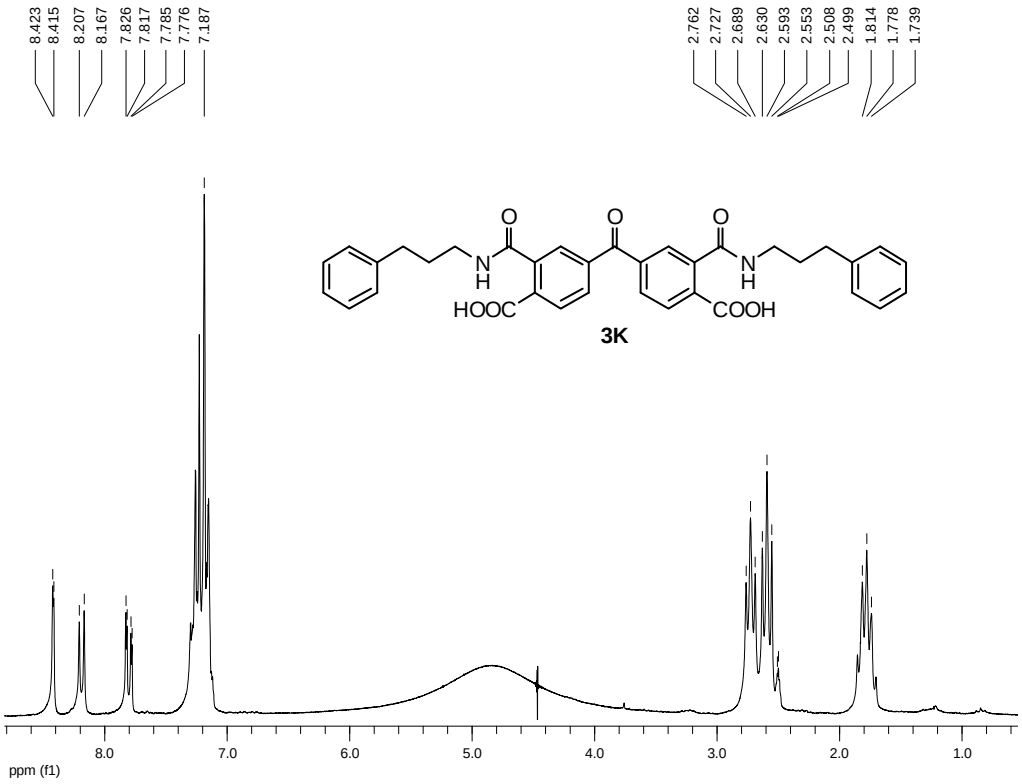
3l

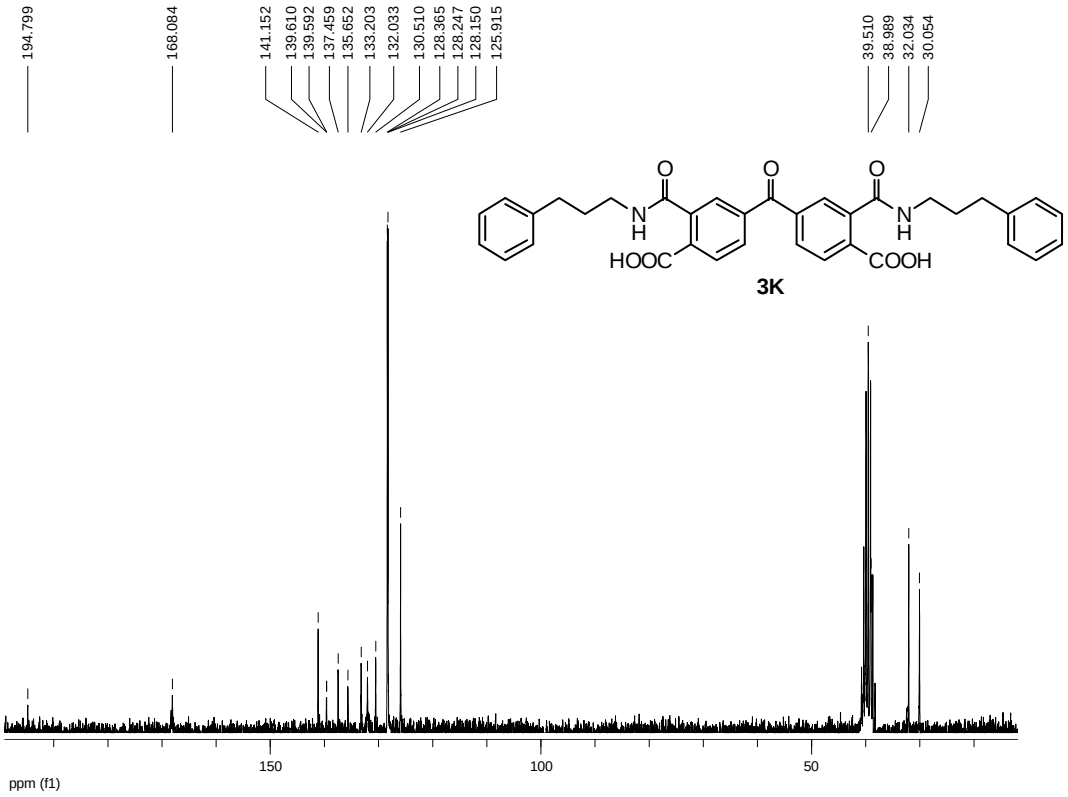

194.671
167.132
167.075
156.919
138.537
137.756
135.019
133.909
132.990
130.835
129.062
128.988
126.184
120.185
110.540
55.270
39.430
39.138
OCH3
O
O
O
OCH3
N
H
N
H
HOOC
COOH
200
150
100
50
0
ppm (f1)

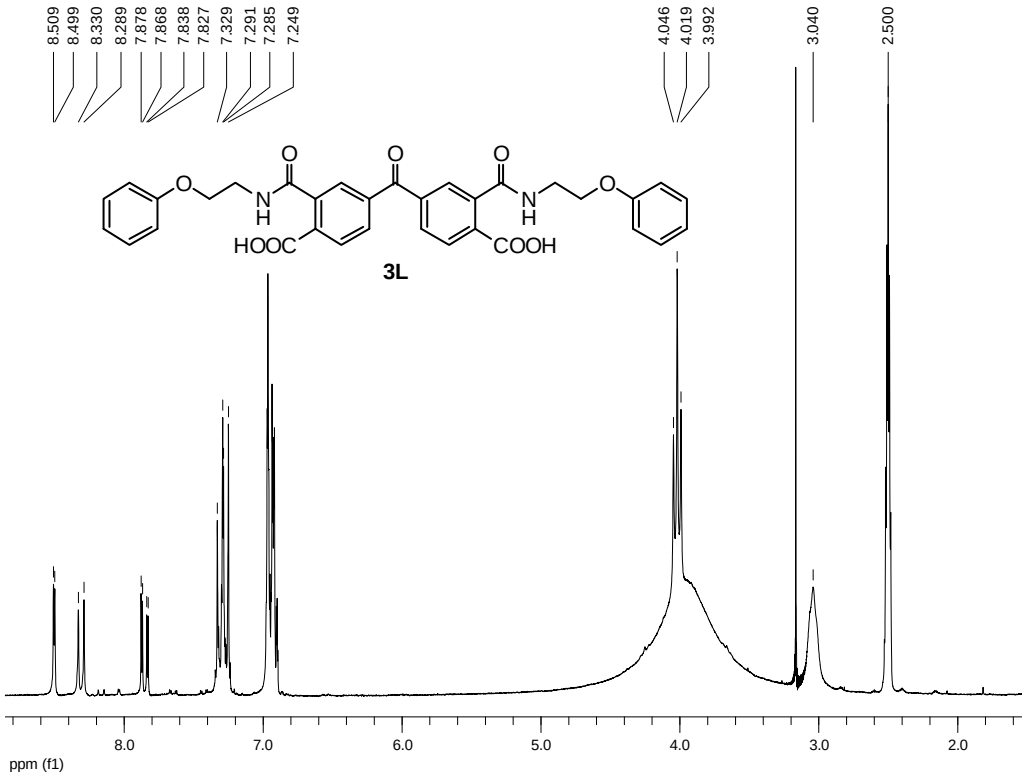
3l

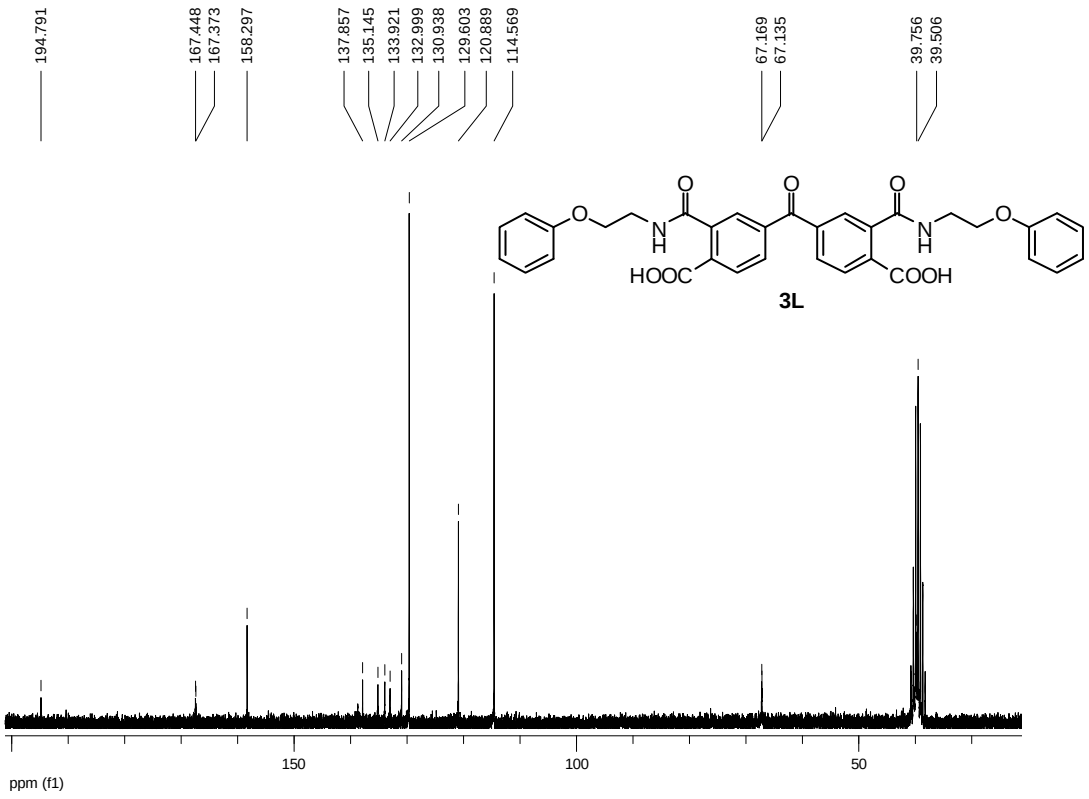

8.474
8.465
8.285
8.245
7.856
7.848
7.815
7.808
7.246
7.239
7.199
7.169
7.160
7.151
7.143
7.114
7.107
6.968
6.928
6.899
6.863
6.825
2.838
2.813
2.766
2.744
2.732
2.499
OCH3
H3CO
HOOC
COOH
3J
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

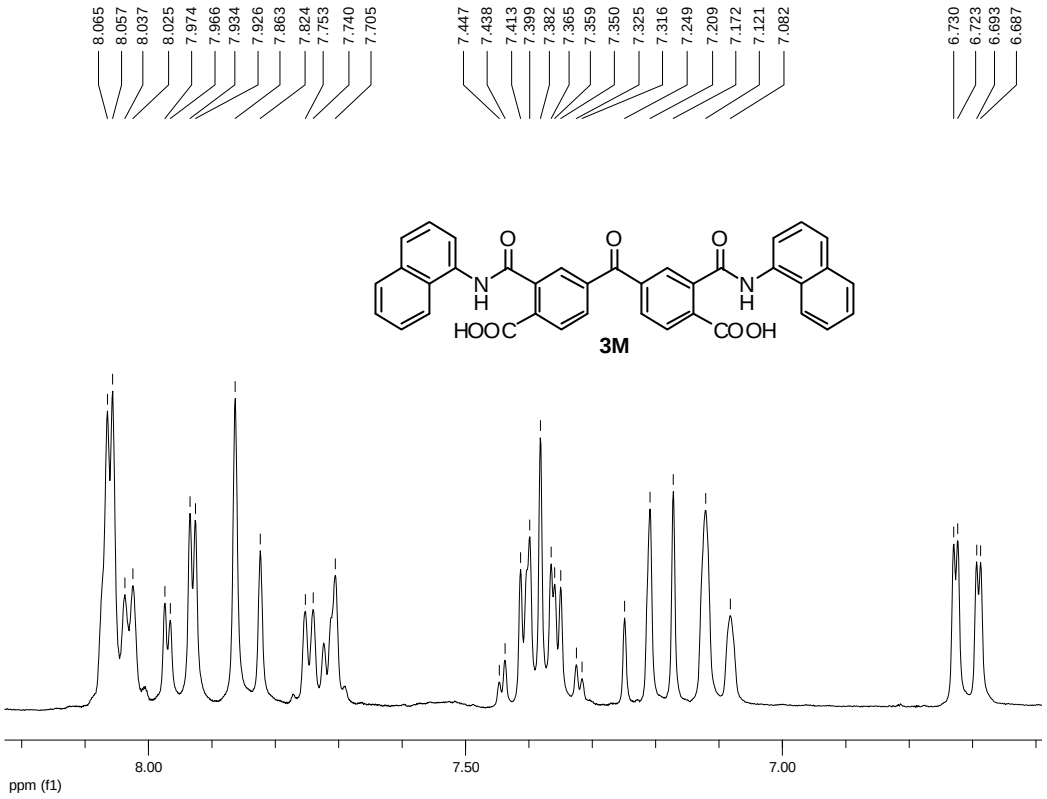

194.801
157.213
137.740
135.310
133.748
132.773
132.753
130.809
130.129
127.930
126.322
120.399
110.785
55.288
48.677
39.501
30.776
OCH3
H3CO
HOOC
COOH
3J
200
150
100
50
0
ppm (t1)

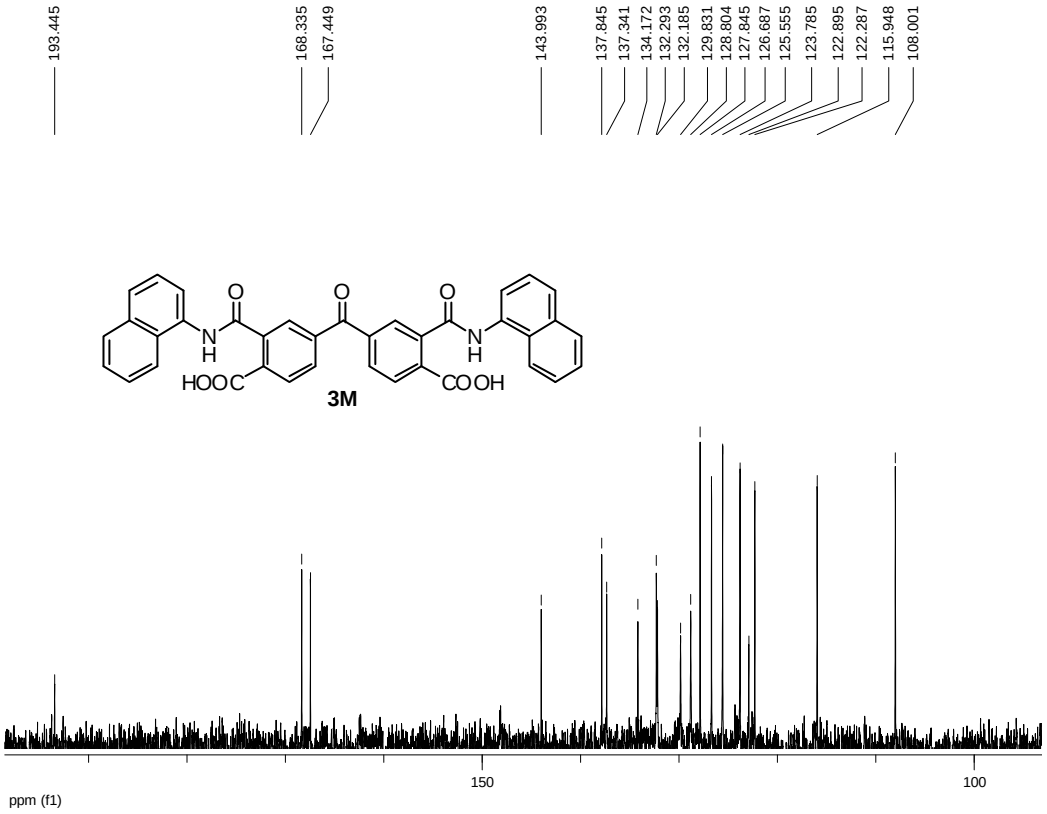

8.423
8.415
8.207
8.167
7.826
7.817
7.785
7.776
7.187
2.762
2.727
2.689
2.630
2.593
2.553
2.508
2.499
1.814
1.778
1.739
O
O
O
N
H
N
H
HOOC
COOH
3K
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

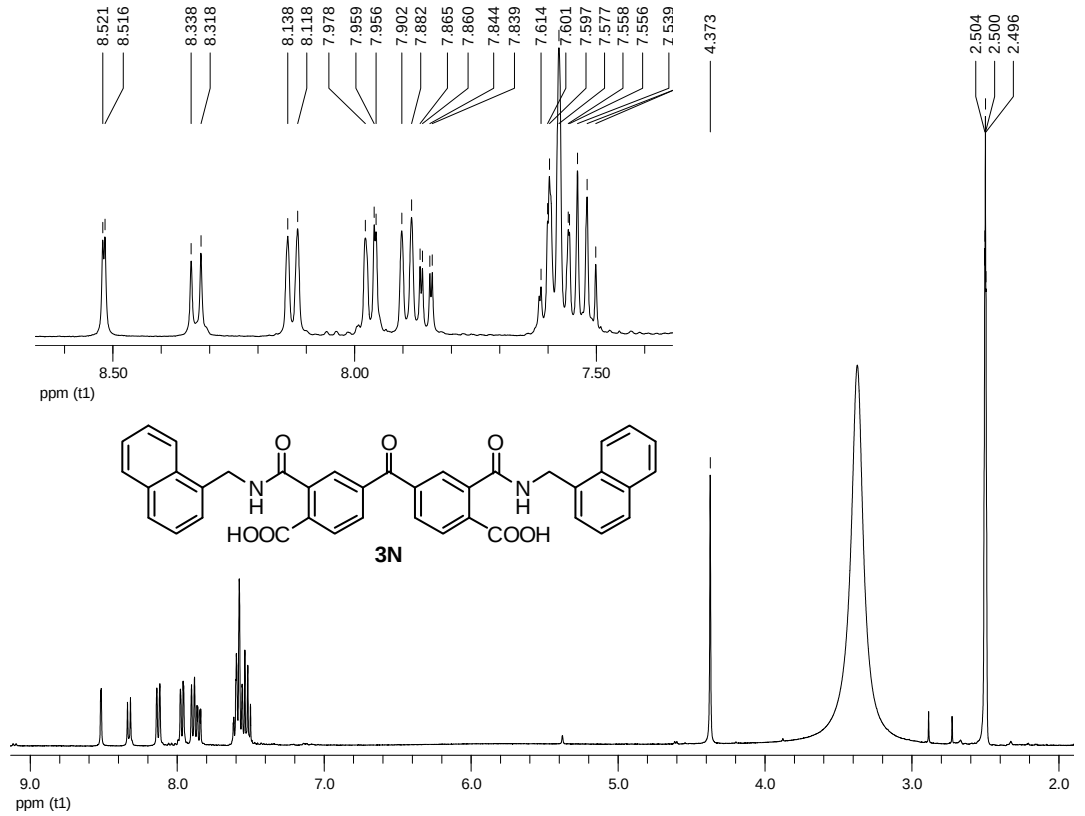

194.799
168.084
141.152
139.610
139.592
137.459
135.652
133.203
132.033
130.510
128.365
128.247
128.150
125.915
39.510
38.989
32.034
30.054
O
O
O
N
H
N
H
HOOC
COOH
3K
150
100
50
ppm (f1)

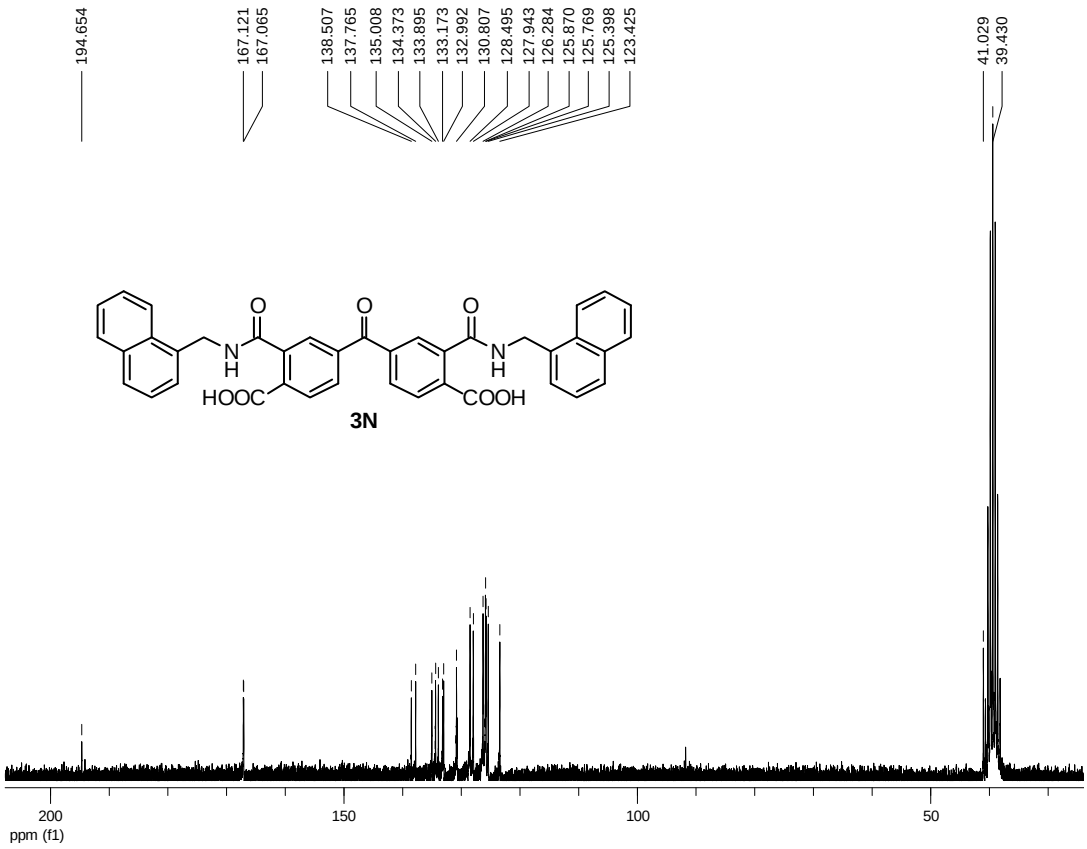

8.509
8.499
8.330
8.289
7.878
7.868
7.838
7.827
7.329
7.291
7.285
7.249
4.046
4.019
3.992
3.040
2.500
HOOC
COOH
3L
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)


194.791
167.448
167.373
158.297
137.857
135.145
133.921
132.999
130.938
129.603
120.889
114.569
67.169
67.135
39.756
39.506
HOOC
COOH
3L
150
100
50
ppm (f1)

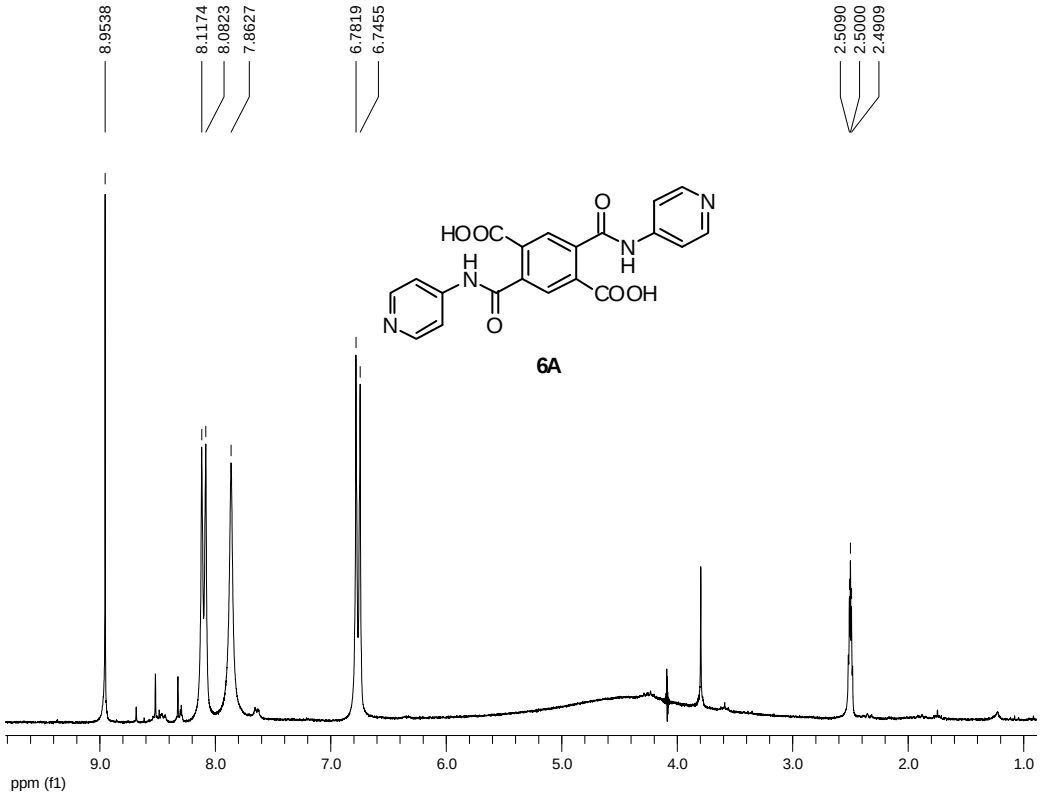

8.065
8.057
8.037
8.025
7.974
7.966
7.934
7.926
7.863
7.824
7.753
7.740
7.705
7.447
7.438
7.413
7.399
7.382
7.365
7.359
7.350
7.325
7.316
7.249
7.209
7.172
7.121
7.082
6.730
6.723
6.693
6.687
O
O
O
N
H
N
H
HOOC
COOH
3M
8.00
7.50
7.00
ppm (f1)


193.445
168.335
167.449
143.993
137.845
137.341
134.172
132.293
132.185
129.831
128.804
127.845
126.687
125.555
123.785
122.895
122.287
115.948
108.001
O
O
O
N
H
N
H
HOOC
COOH
3M
150
100
ppm (f1)

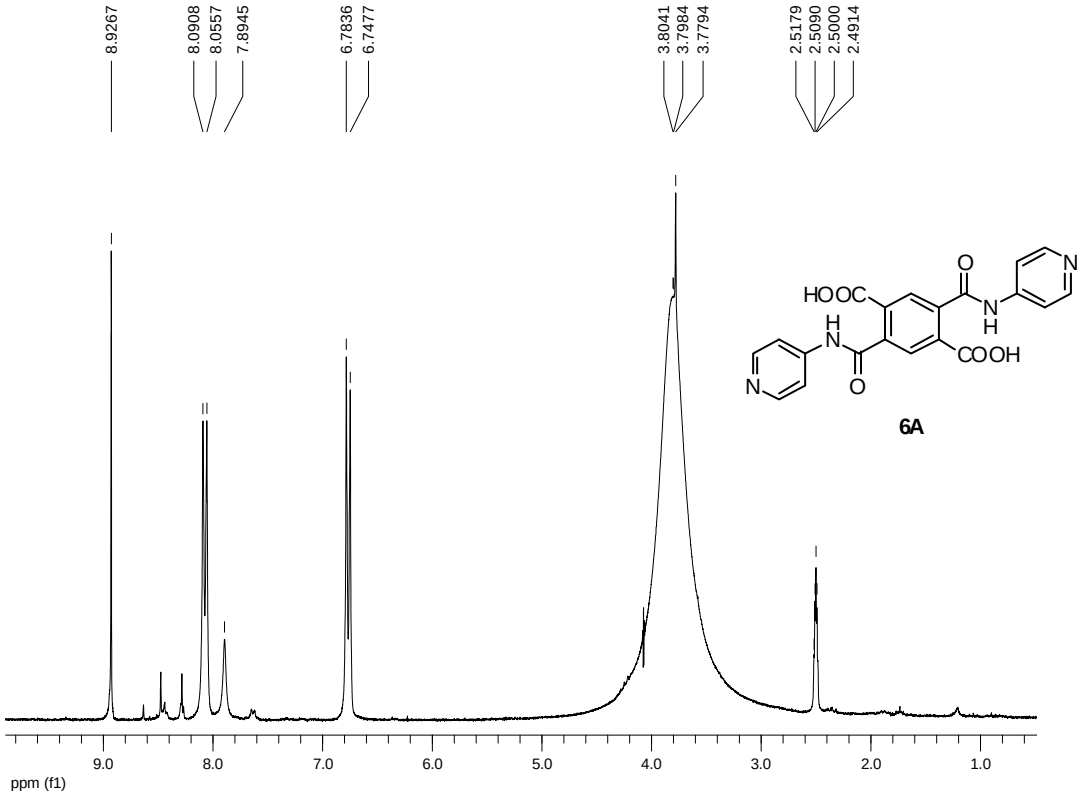

8.521
8.516
8.338
8.318
8.138
8.118
7.978
7.959
7.956
7.902
7.882
7.865
7.860
7.844
7.839
7.614
7.601
7.597
7.577
7.558
7.556
7.539
4.373
2.504
2.500
2.496
8.50
8.00
7.50
ppm (t1)
HOOC
COOH
3N
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)

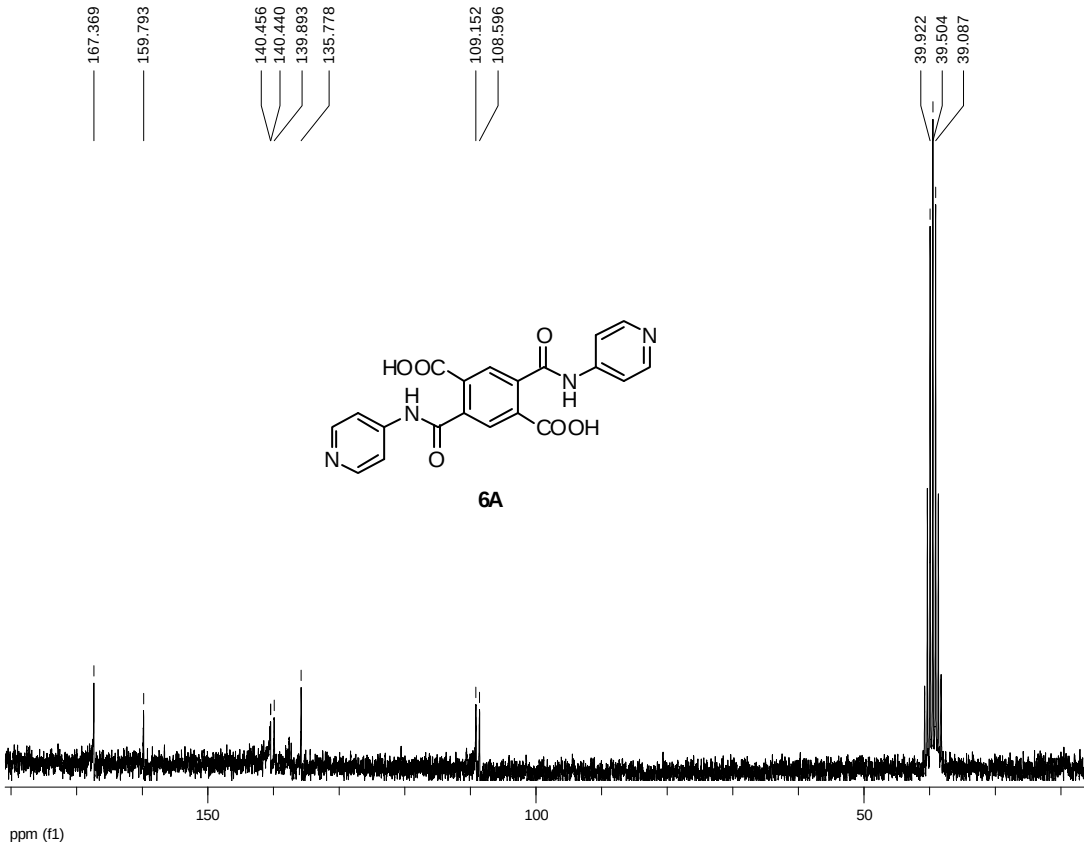

194.654
167.121
167.065
138.507
137.765
135.008
134.373
133.895
133.173
132.992
130.807
128.495
127.943
126.284
125.870
125.769
125.398
123.425
41.029
39.430
HOOC
COOH
3N
200
150
100
50
ppm (f1)

II. Selected NMR spectra of compounds 6



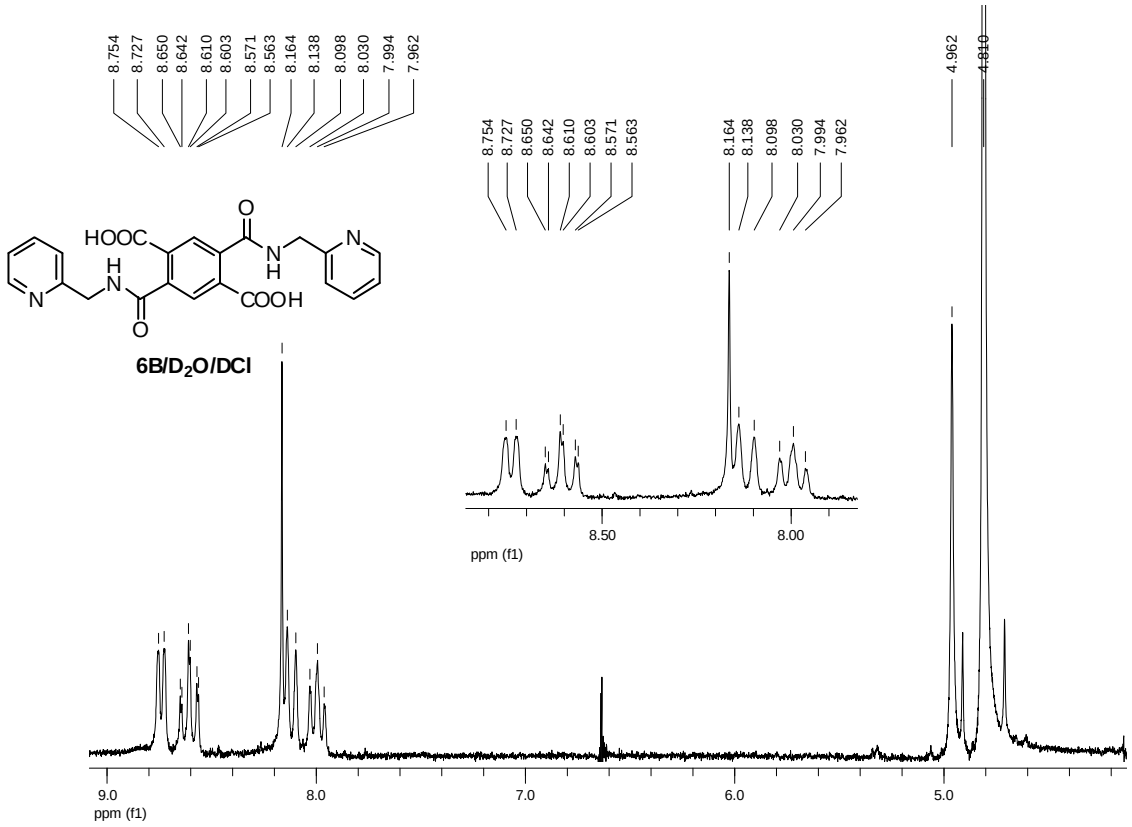
With two drops of D_2O added



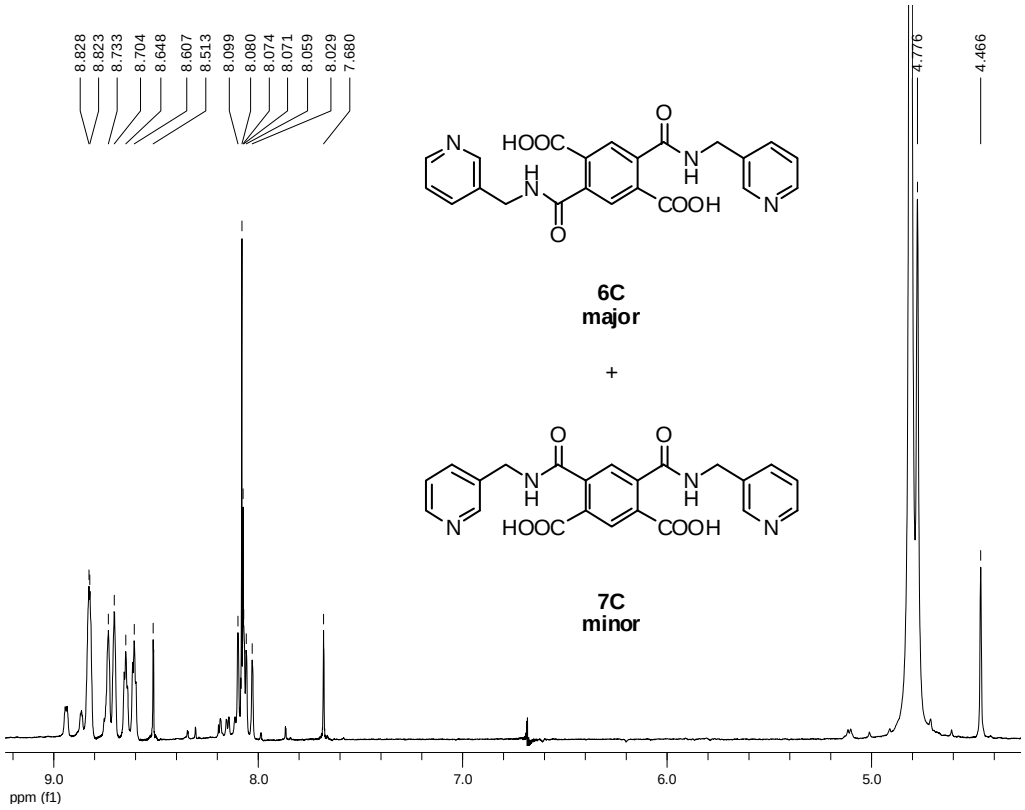

167.369
159.793
140.456
140.440
139.893
135.778
109.152
108.596
39.922
39.504
39.087
HOOC
H
N
O
N
H
N
O
COOH
150
100
50
ppm (f1)

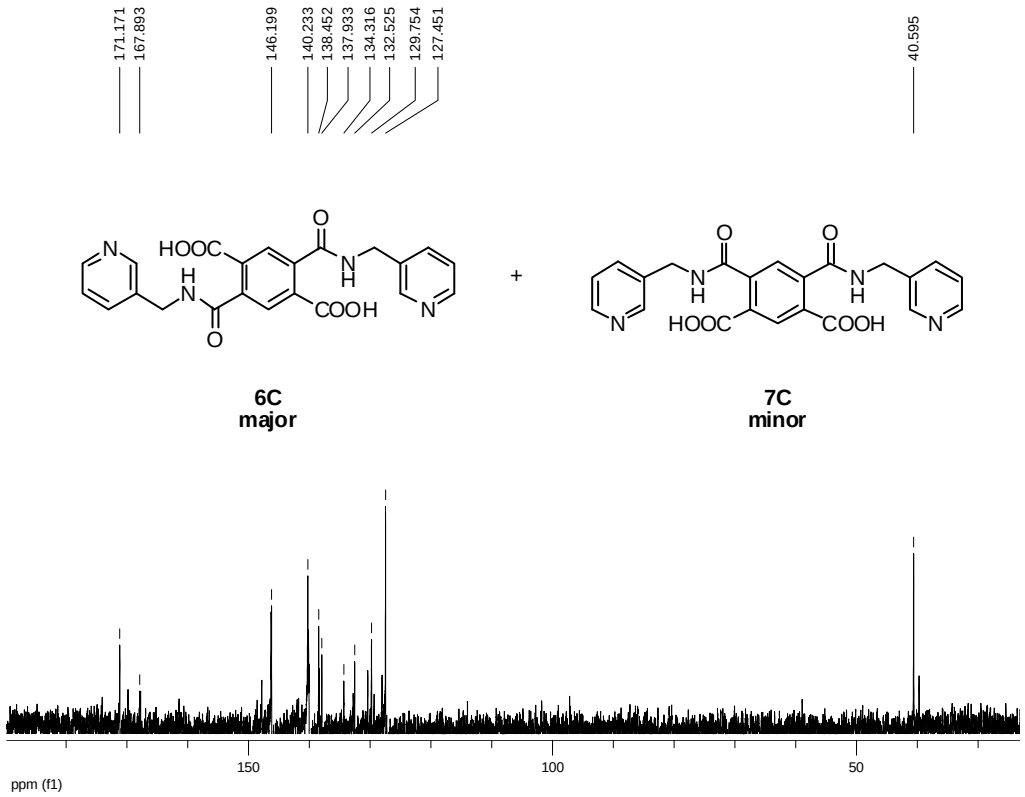
6A

Measured in D_2O/DCl

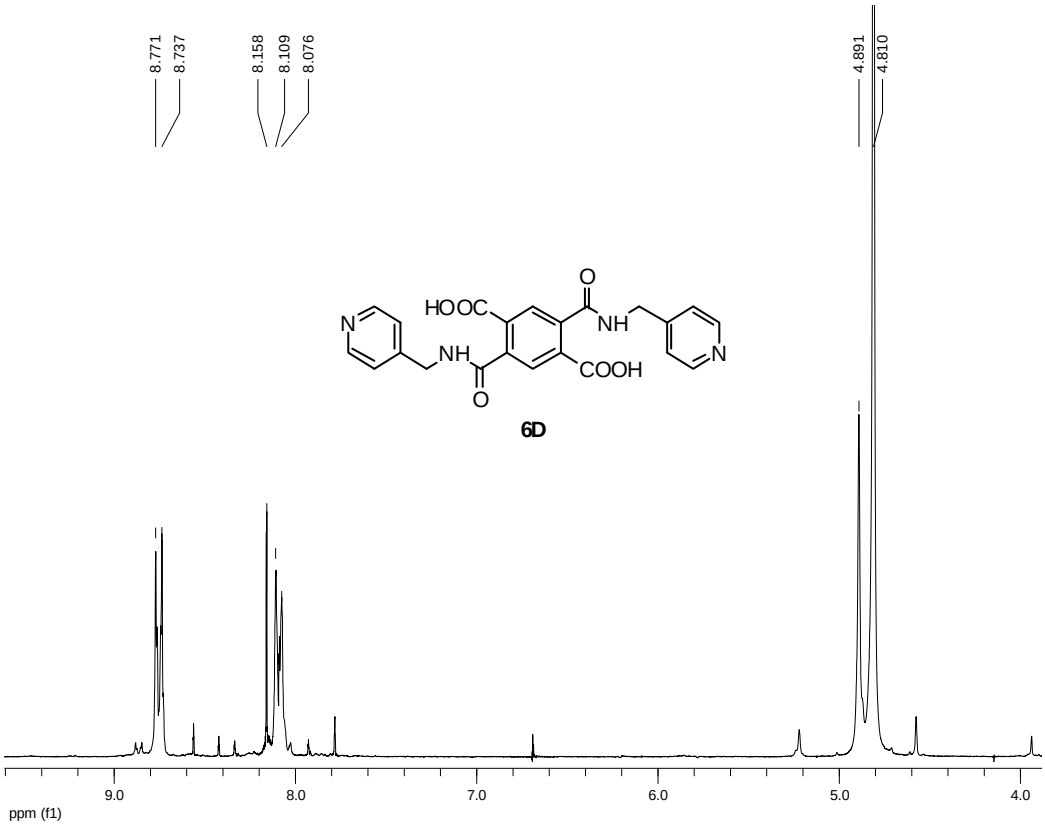


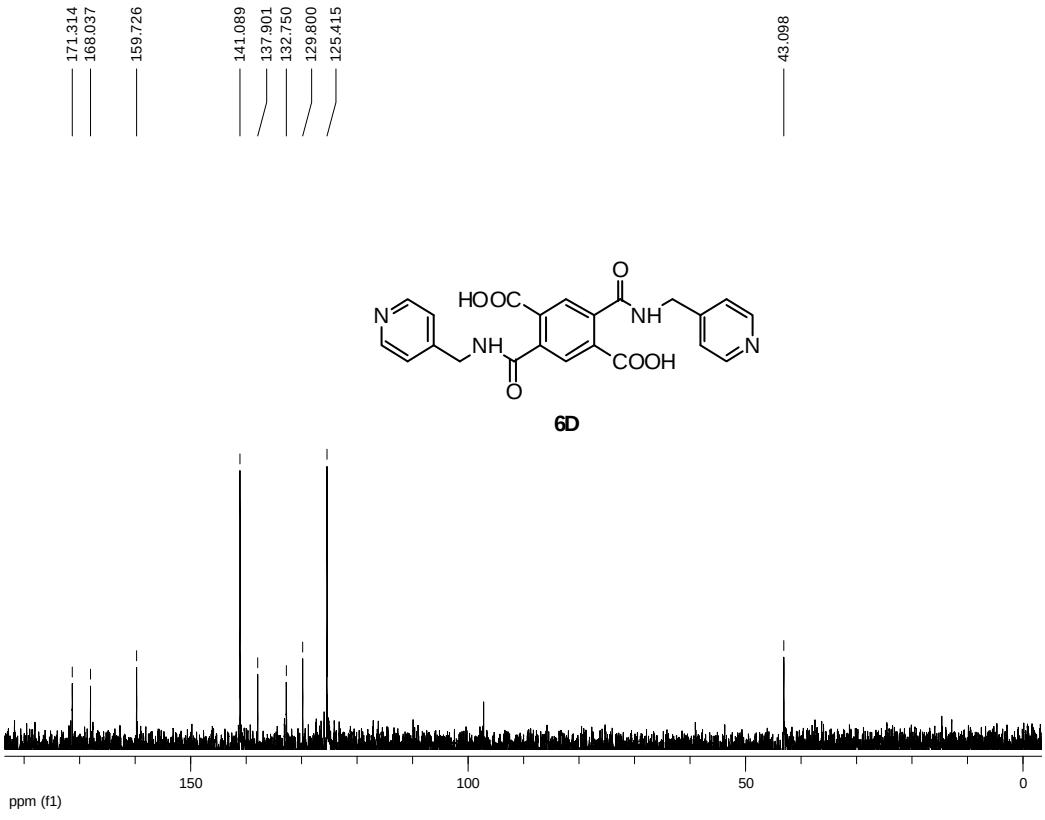
Measured in D_2O/DCl



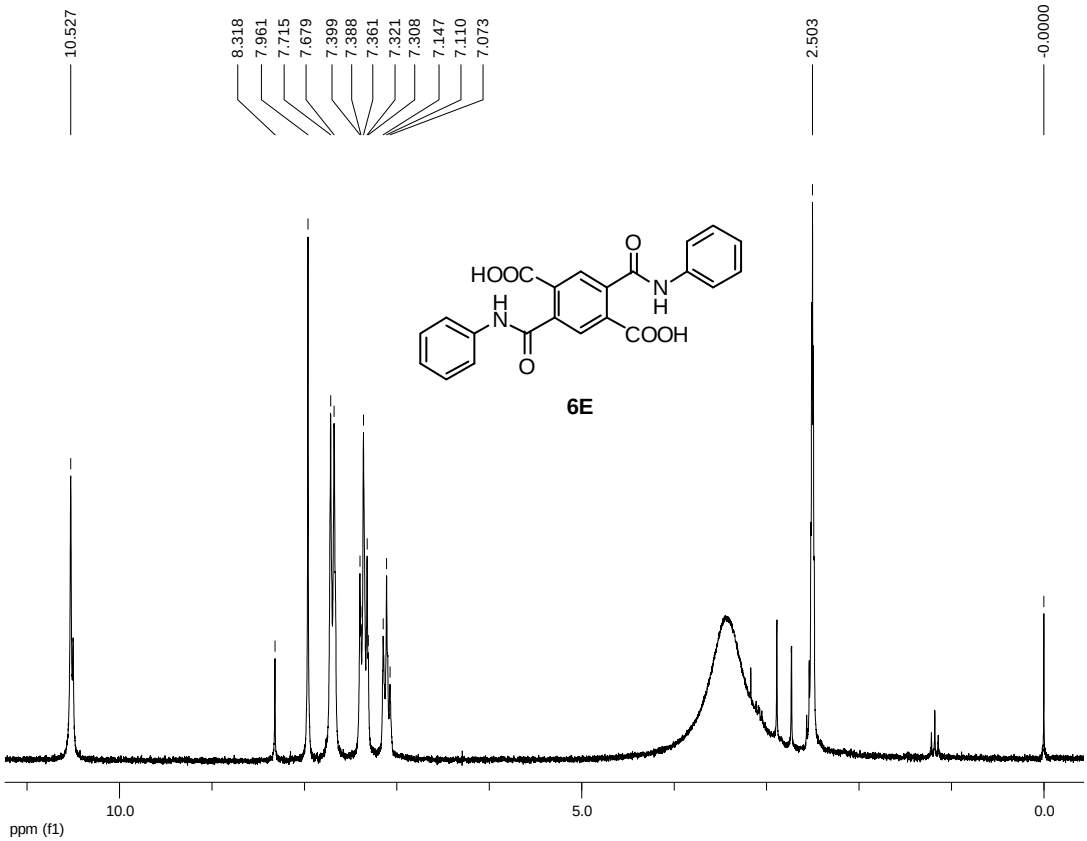


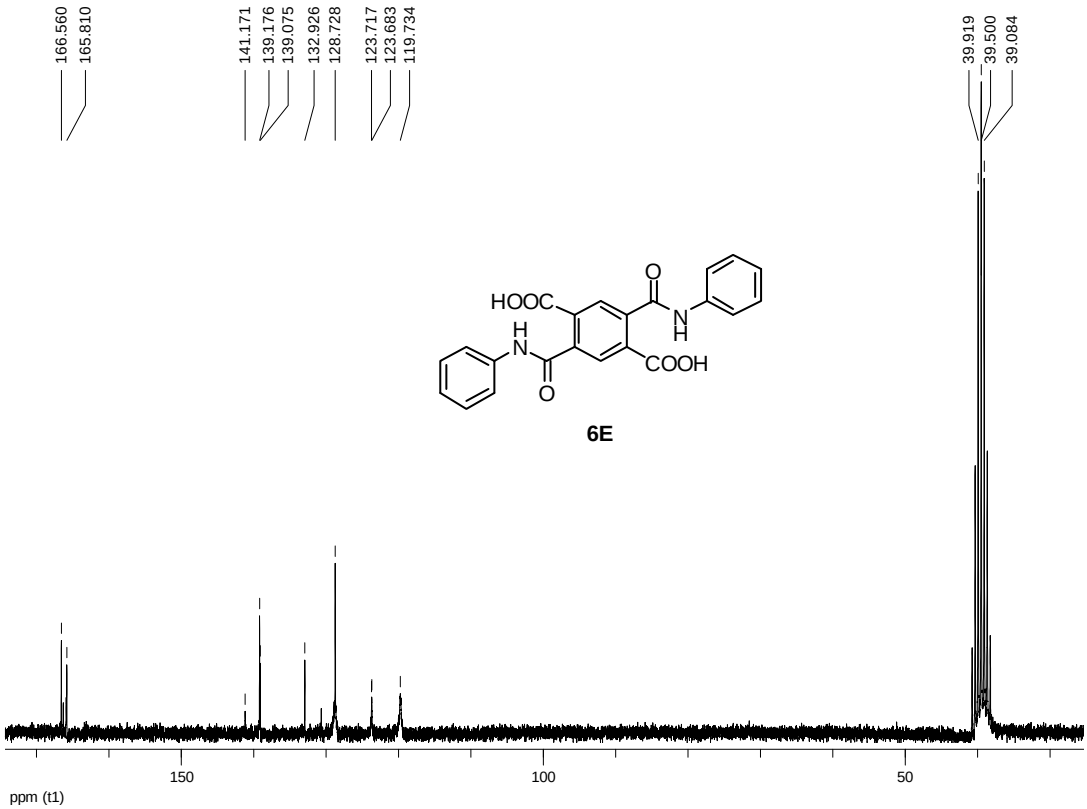
Measured in D_2O/DCl



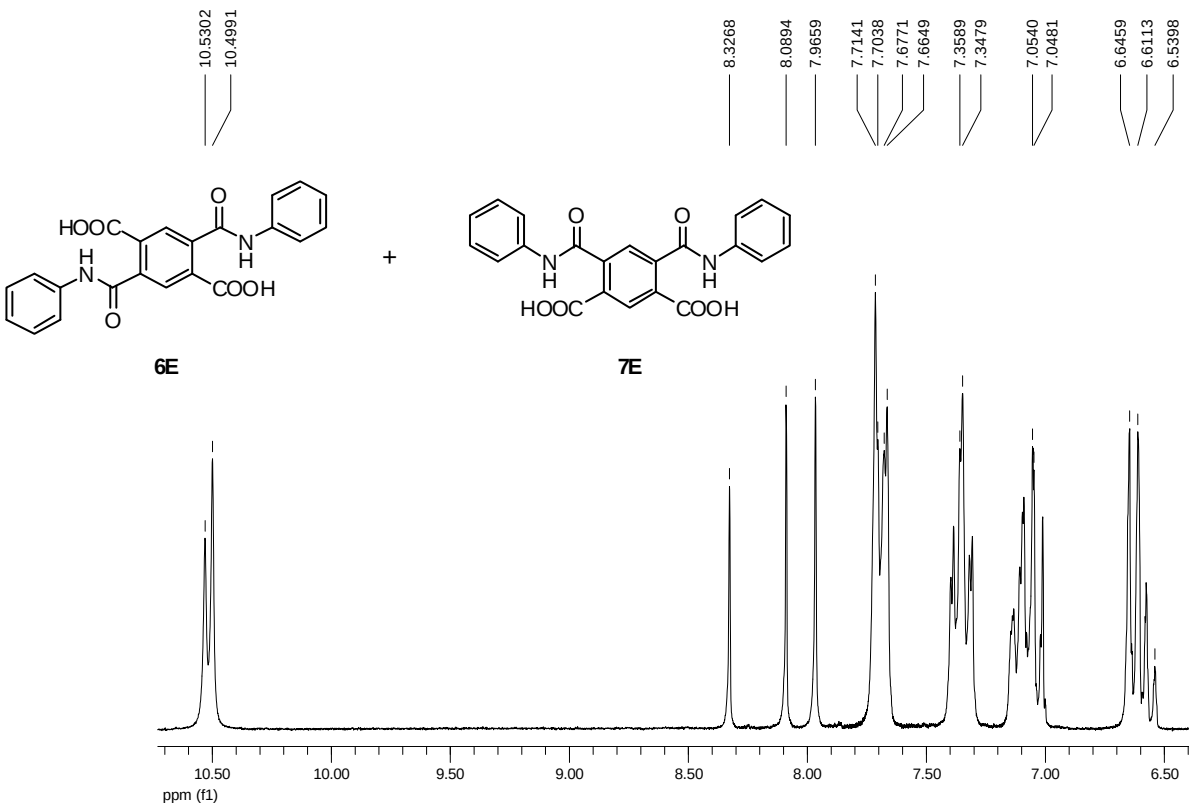


Reaction in THF

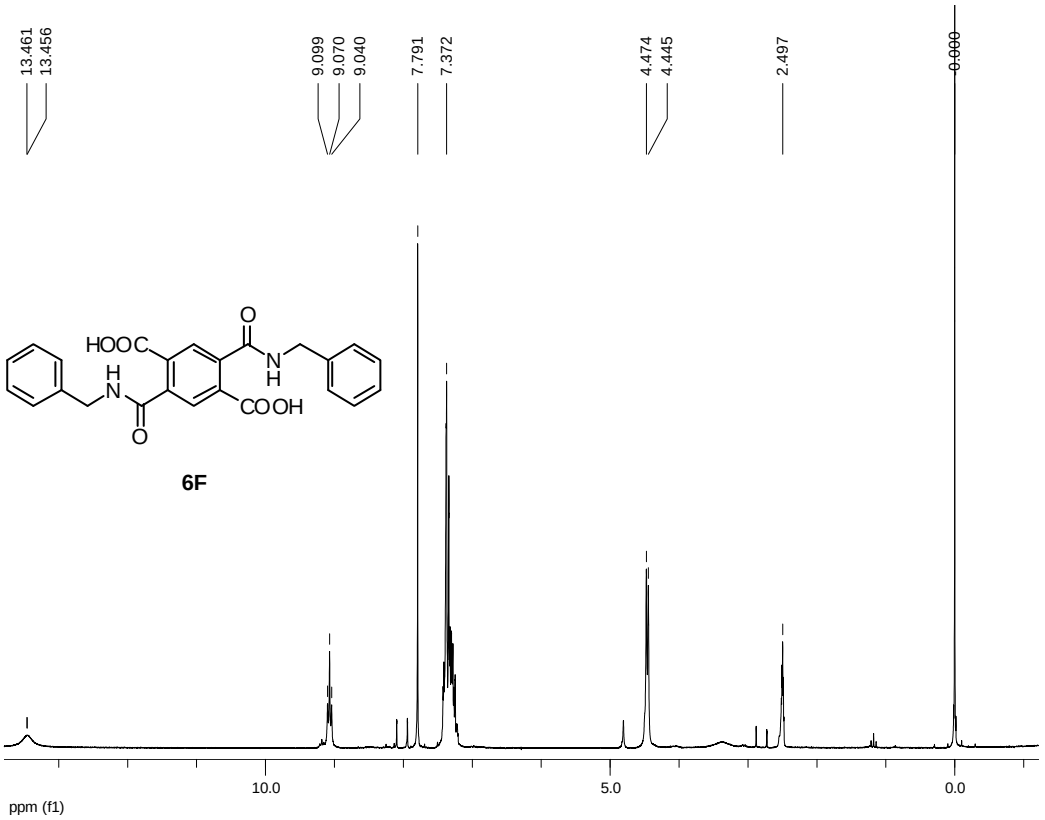




Reaction in Ethanol

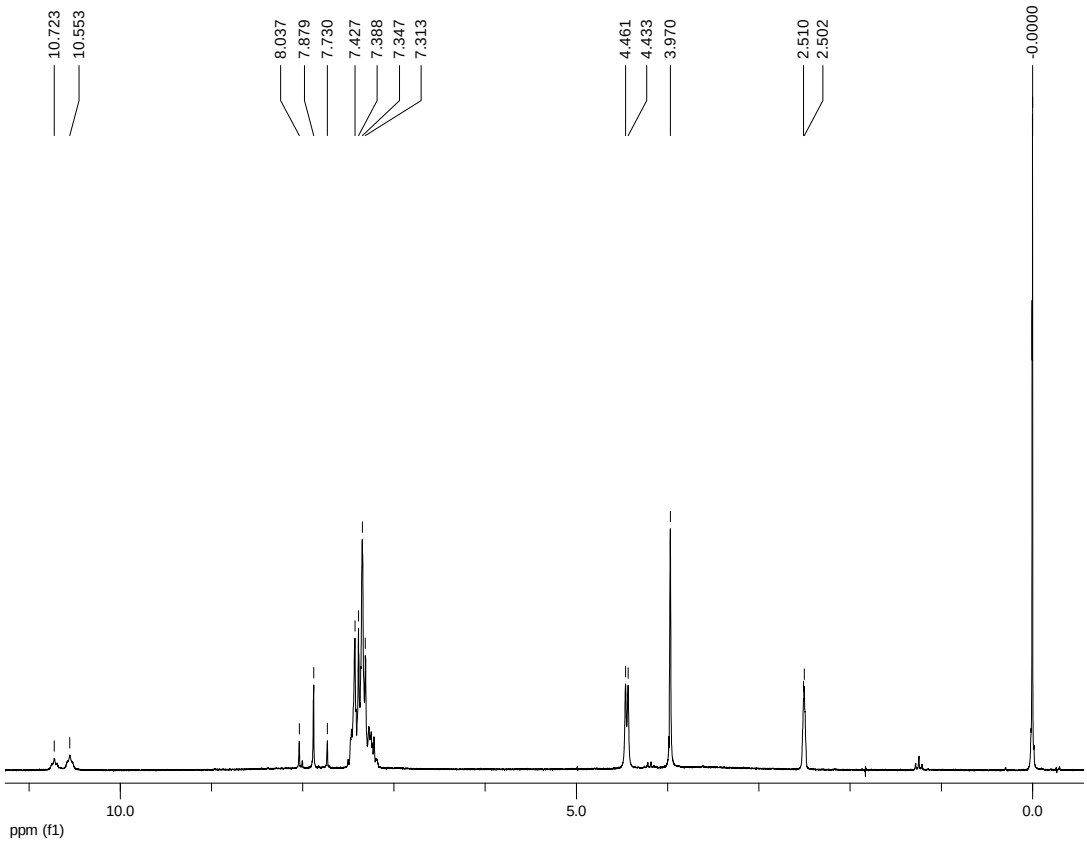


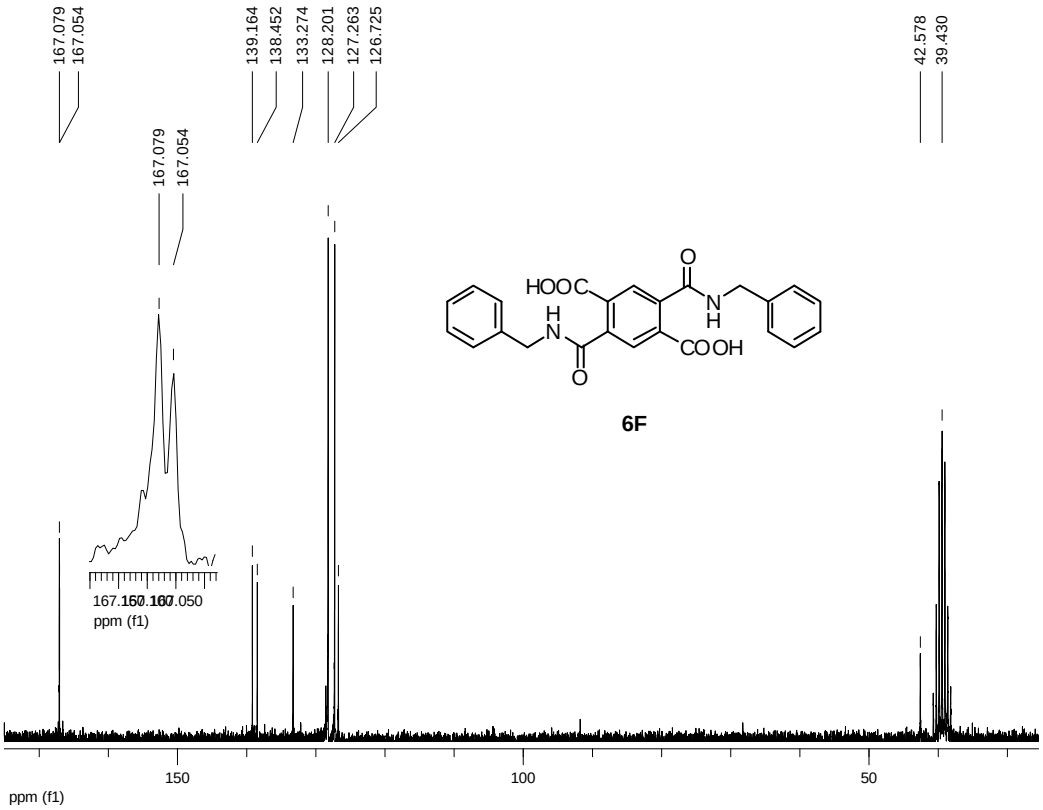
Reaction in THF

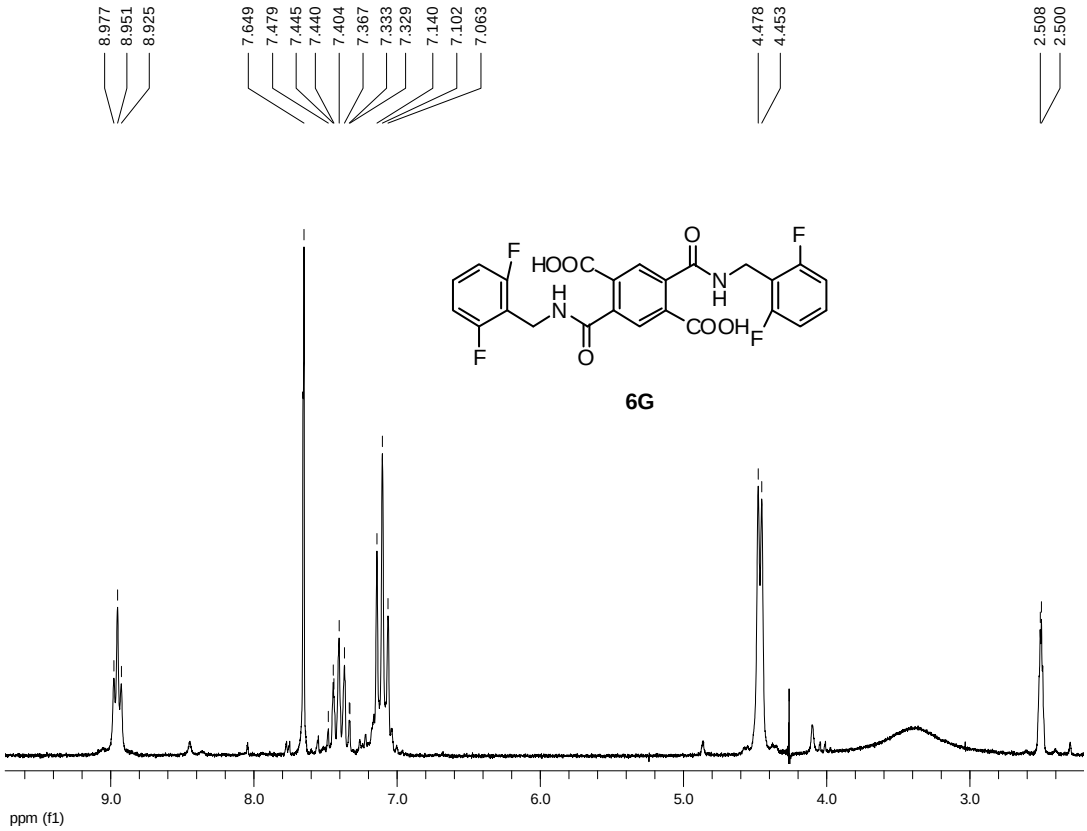


6F

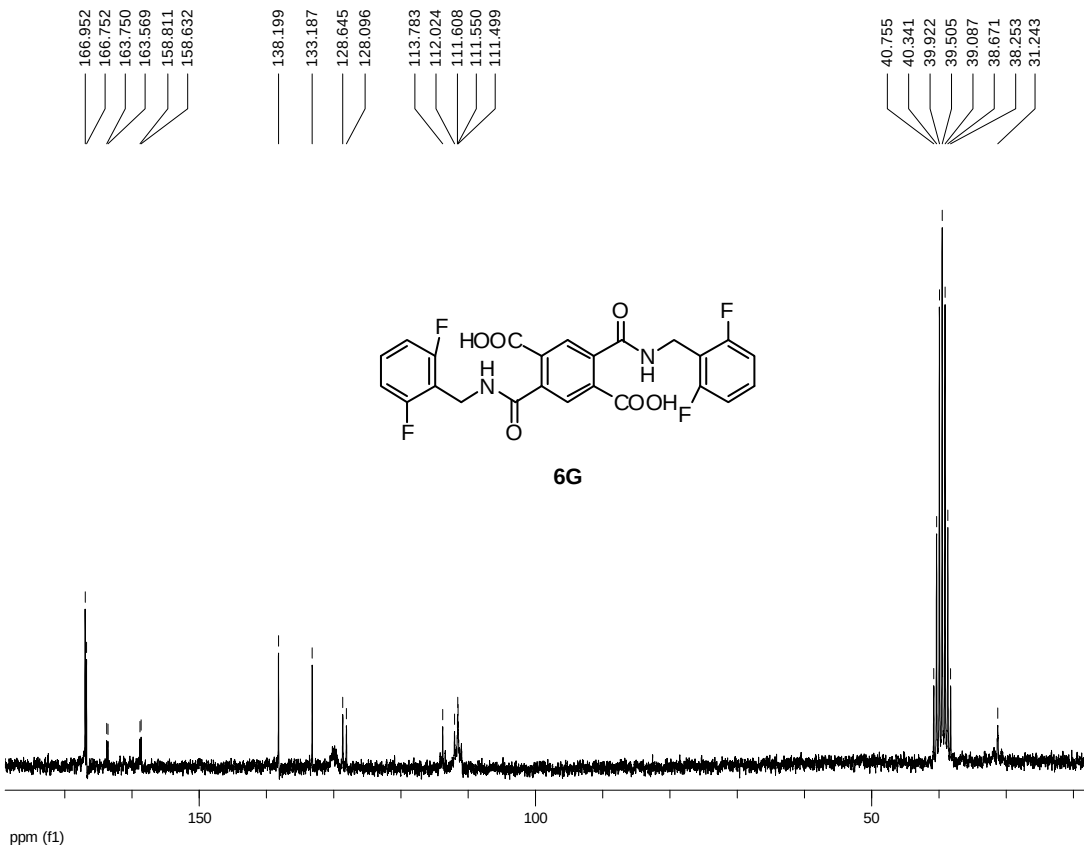
Reaction in Ethanol



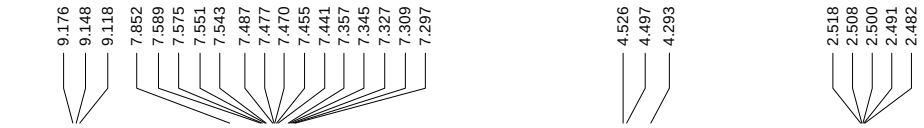

167.079
167.054
139.164
138.452
133.274
128.201
127.263
126.725
42.578
39.430
HOOC
O
N
H
H
N
CO OH
O
6F
167.160
167.050
ppm (f1)
150
100
50
ppm (f1)

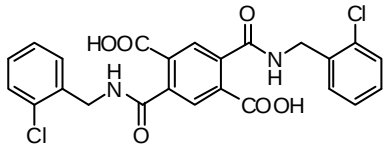

8.977
8.951
8.925
7.649
7.479
7.445
7.440
7.404
7.367
7.333
7.329
7.140
7.102
7.063
4.478
4.453
2.508
2.500
F
HOOC
O
N
H
F
H
N
COOH
F
F
O
9.0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (f1)

6G

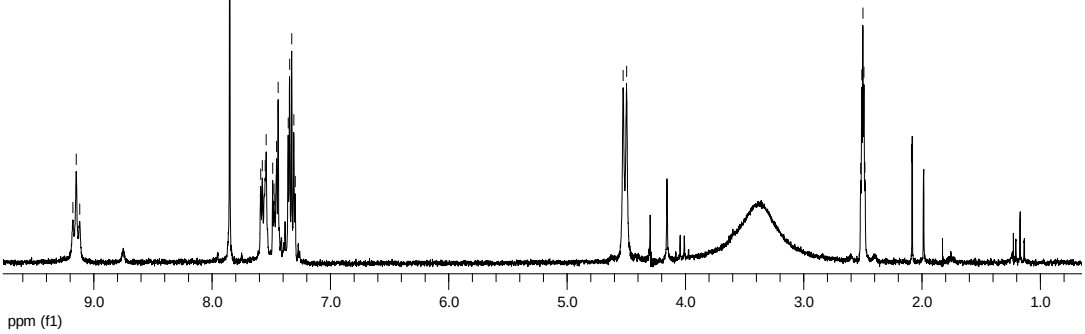

166.952
166.752
163.750
163.569
158.811
158.632
138.199
133.187
128.645
128.096
113.783
112.024
111.608
111.550
111.499
40.755
40.341
39.922
39.505
39.087
38.671
38.253
31.243
F
HOOC
O
N
H
F
H
N
COOH
F
F
O
150
100
50
ppm (f1)

6G

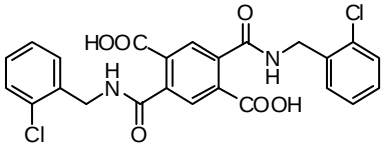

9.176
9.148
9.118
7.852
7.589
7.575
7.551
7.543
7.487
7.477
7.470
7.455
7.441
7.357
7.345
7.327
7.309
7.297
4.526
4.497
4.293
2.518
2.508
2.500
2.491
2.482


HOOC
H
N
O
Cl
O
N
H
Cl
COOH
O

6H

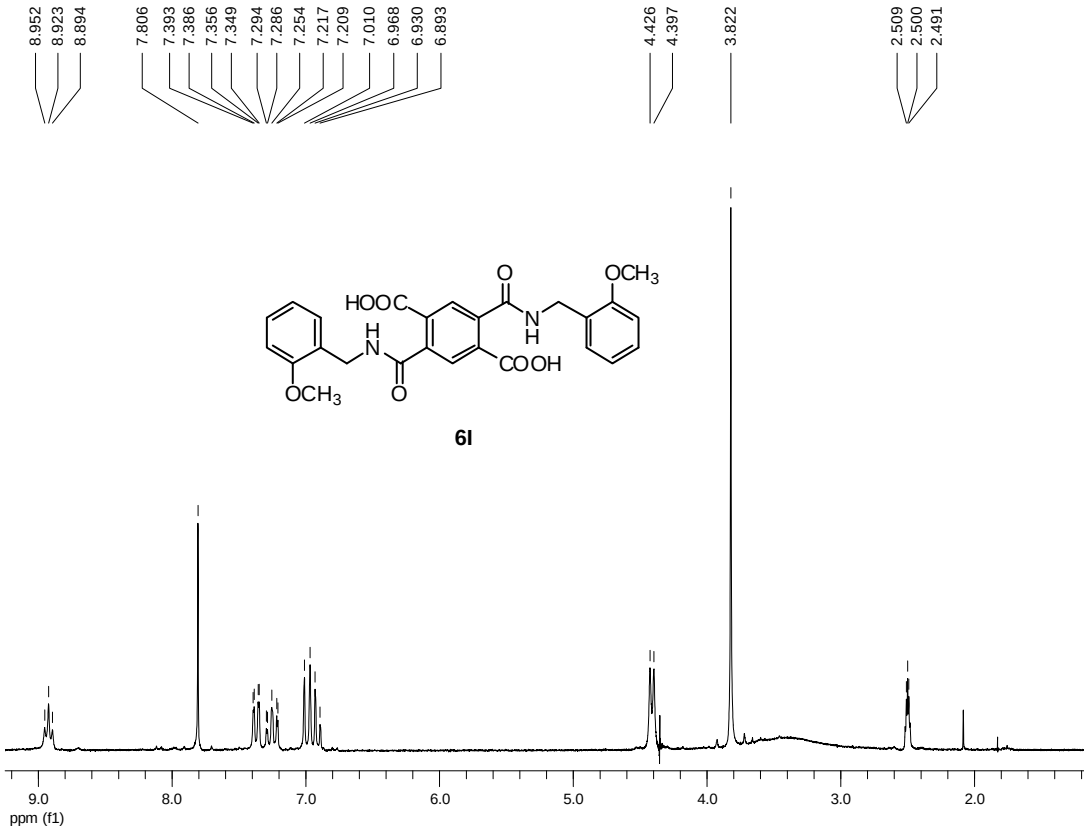

9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


167.422
167.181
138.358
136.042
133.512
131.942
129.344
129.293
128.773
128.242
127.508
127.482
126.935
40.748
40.635
40.331
39.910
39.494
39.077
38.657
38.233

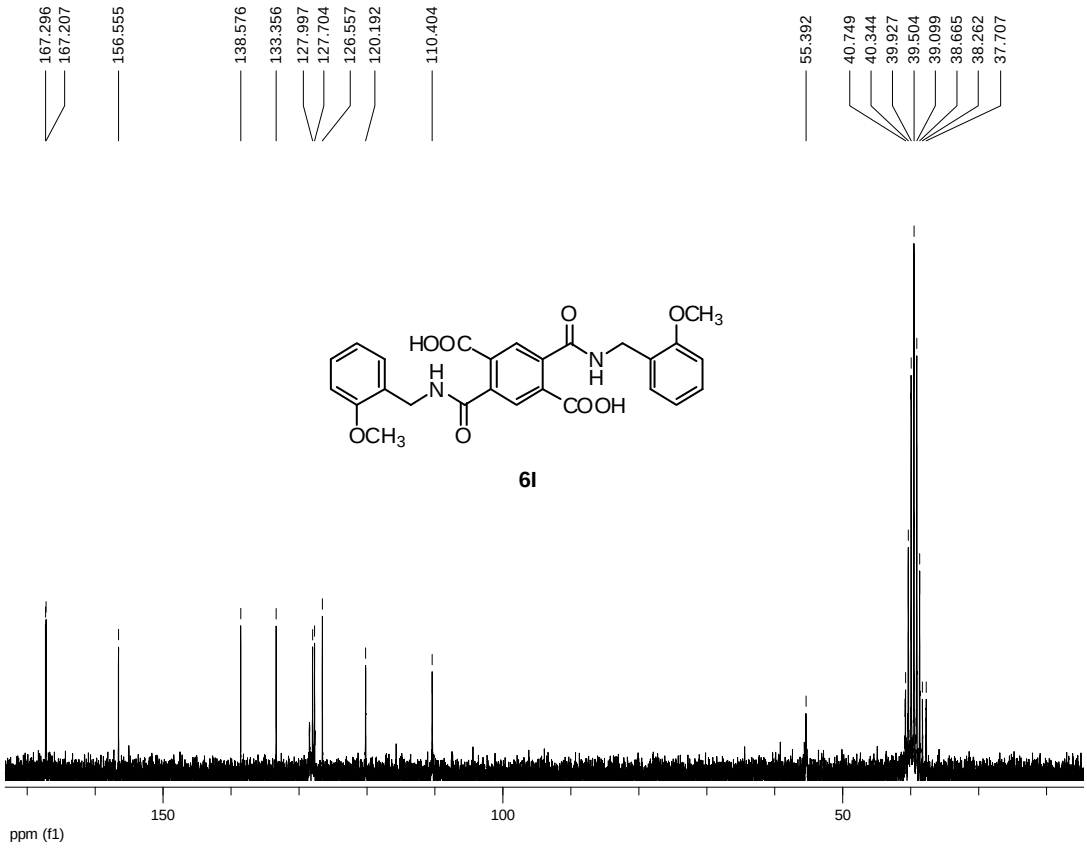

HOOC
H
N
O
Cl
O
N
H
Cl
COOH
O

6H

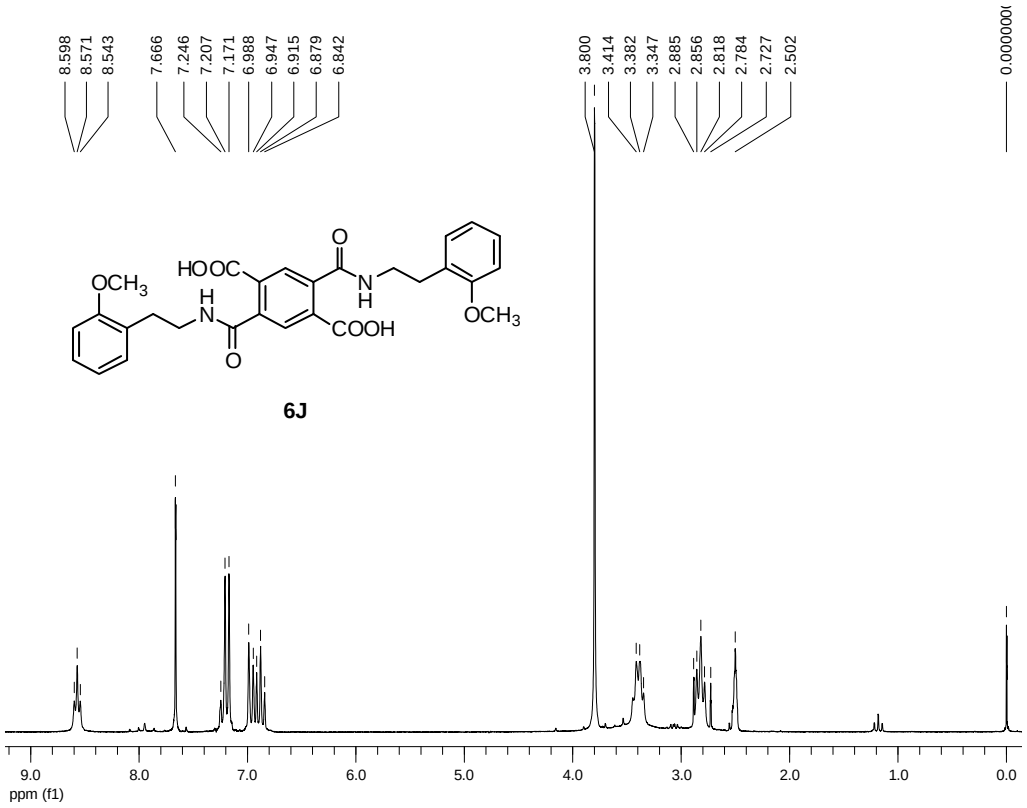
150
100
50
ppm (f1)


8.952
8.923
8.894
7.806
7.393
7.386
7.356
7.349
7.294
7.286
7.254
7.217
7.209
7.010
6.968
6.930
6.893
4.426
4.397
3.822
2.509
2.500
2.491
HOOC
O
OCH3
N
H
COOH
OCH3
O
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)

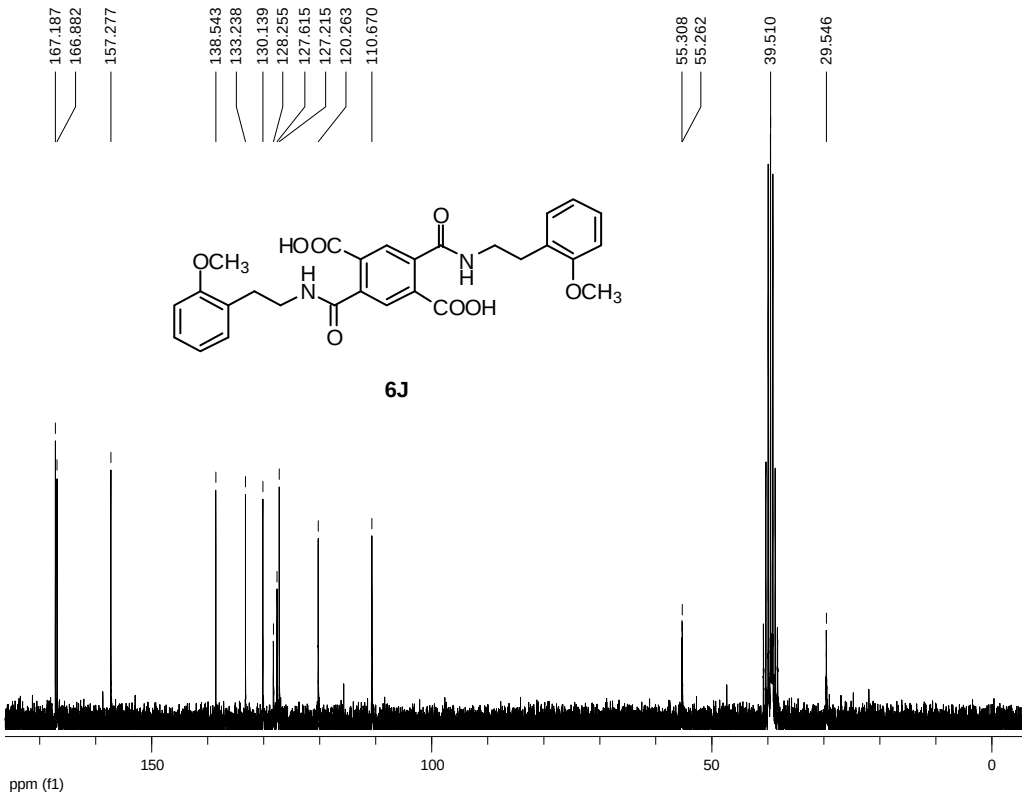
6l


167.296
167.207
156.555
138.576
133.356
127.997
127.704
126.557
120.192
110.404
55.392
40.749
40.344
39.927
39.504
39.099
38.665
38.262
37.707
HOOC
O
OCH3
N
H
COOH
OCH3
O
150
100
50
ppm (f1)

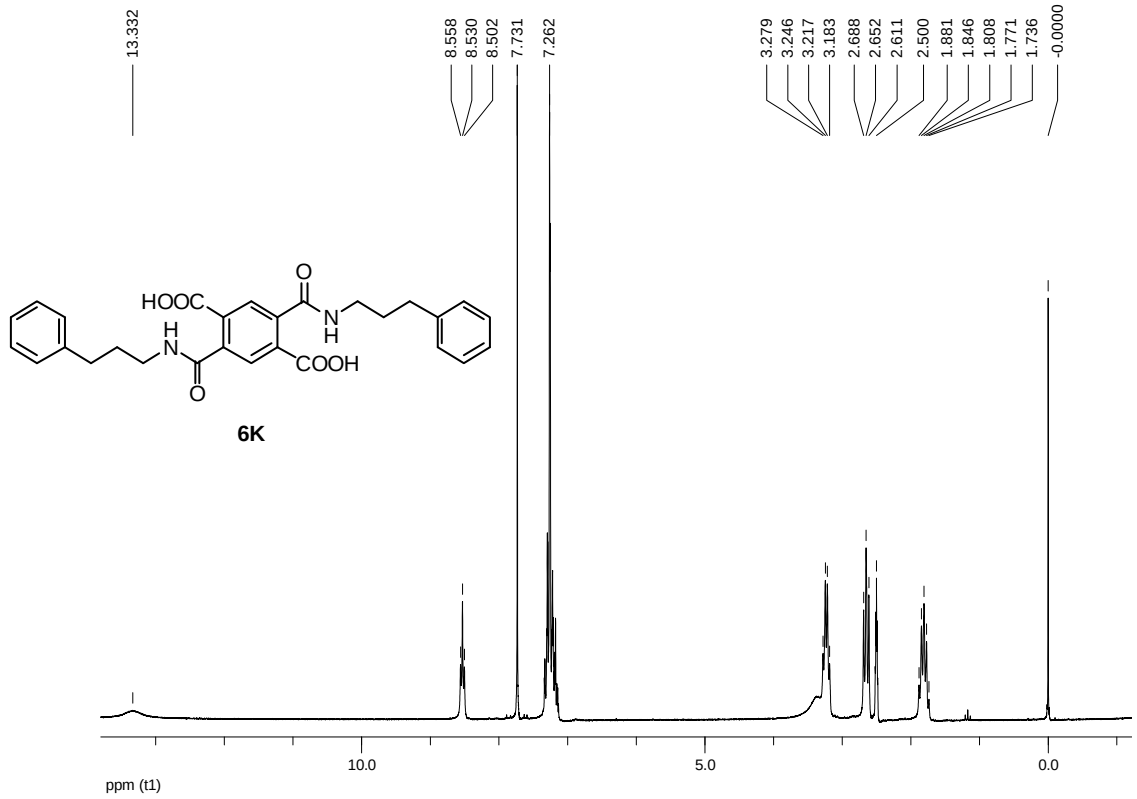
6l


8.598
8.571
8.543
7.666
7.246
7.207
7.171
6.988
6.947
6.915
6.879
6.842
3.800
3.414
3.382
3.347
2.885
2.856
2.818
2.784
2.727
2.502
0.000000
HOOC
OCH3
H
N
O
COOH
N
H
OCH3
O
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

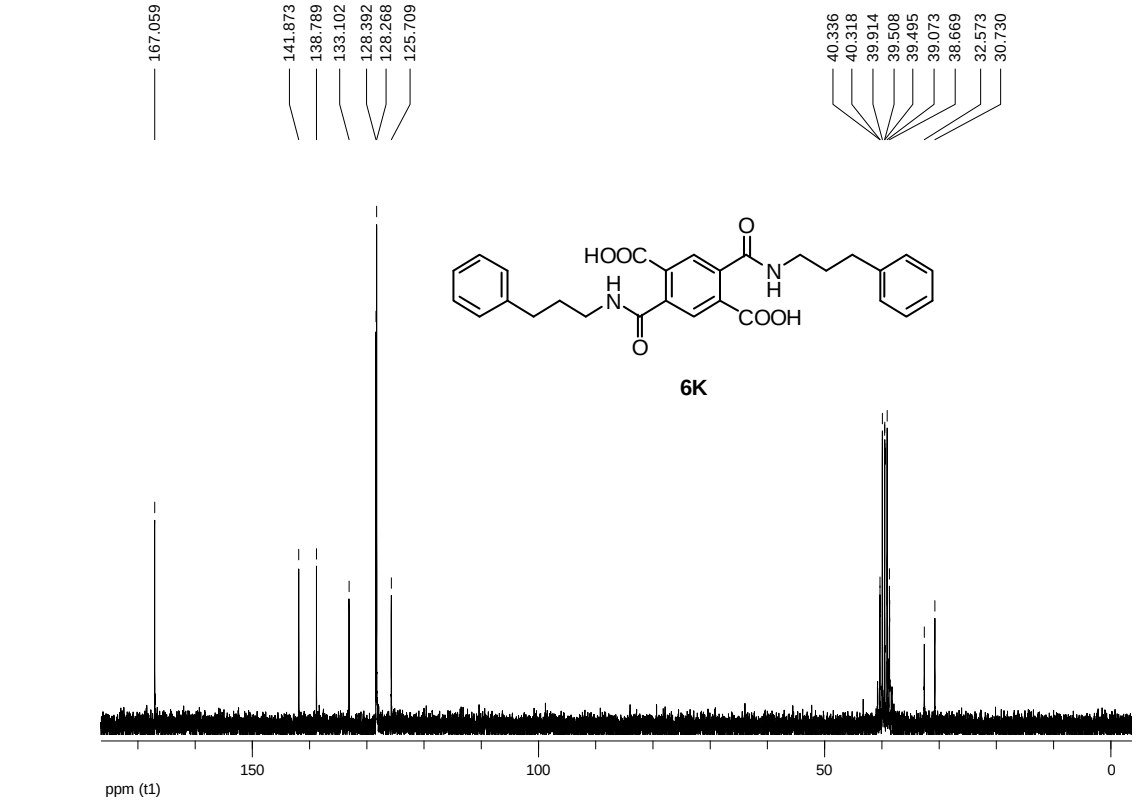
6J


167.187
166.882
157.277
138.543
133.238
130.139
128.255
127.615
127.215
120.263
110.670
55.308
55.262
39.510
29.546
HOOC
OCH3
H
N
O
COOH
N
H
OCH3
O
150
100
50
0
ppm (f1)

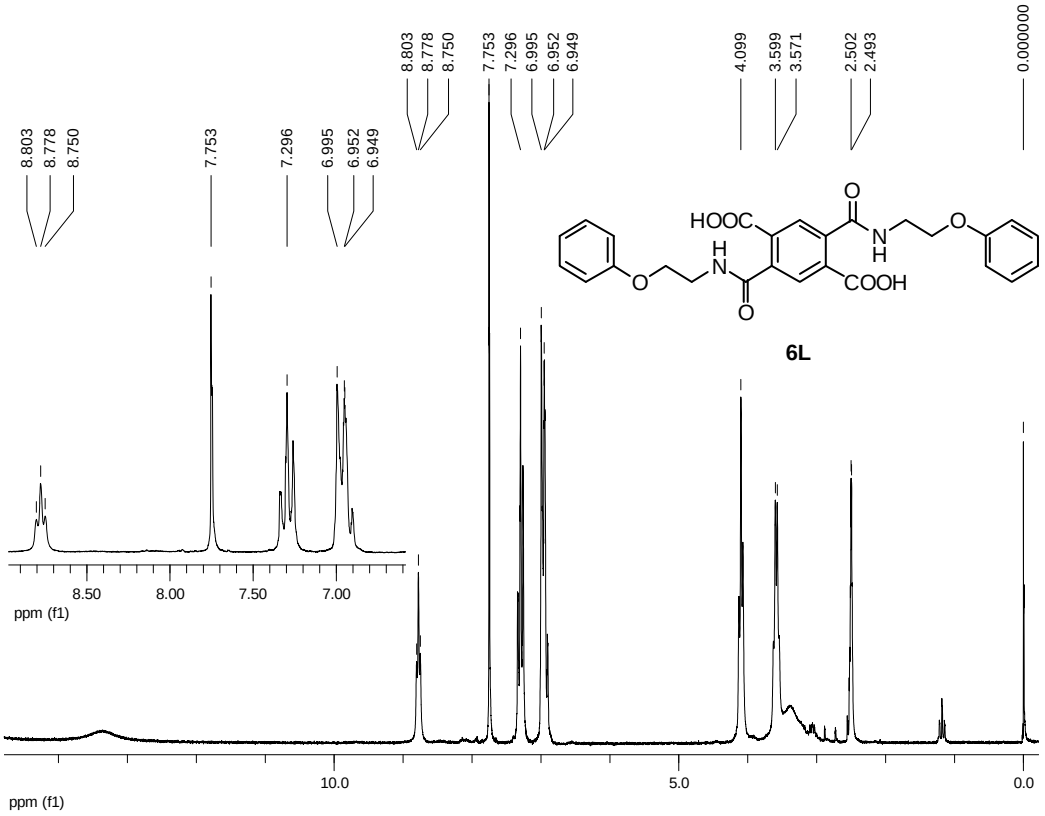
6J

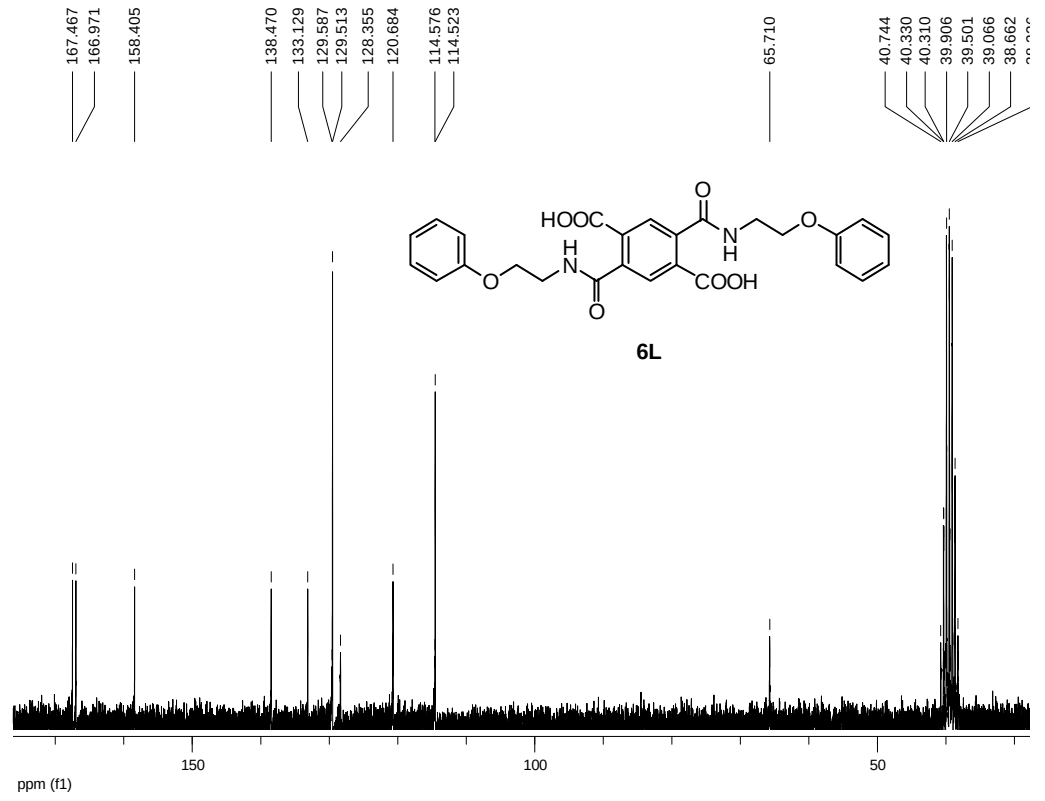

13.332
8.558
8.530
8.502
7.731
7.262
3.279
3.246
3.217
3.183
2.688
2.652
2.611
2.500
1.881
1.846
1.808
1.771
1.736
-0.0000
HOOC
COOH
10.0
5.0
0.0
ppm (t1)

6K

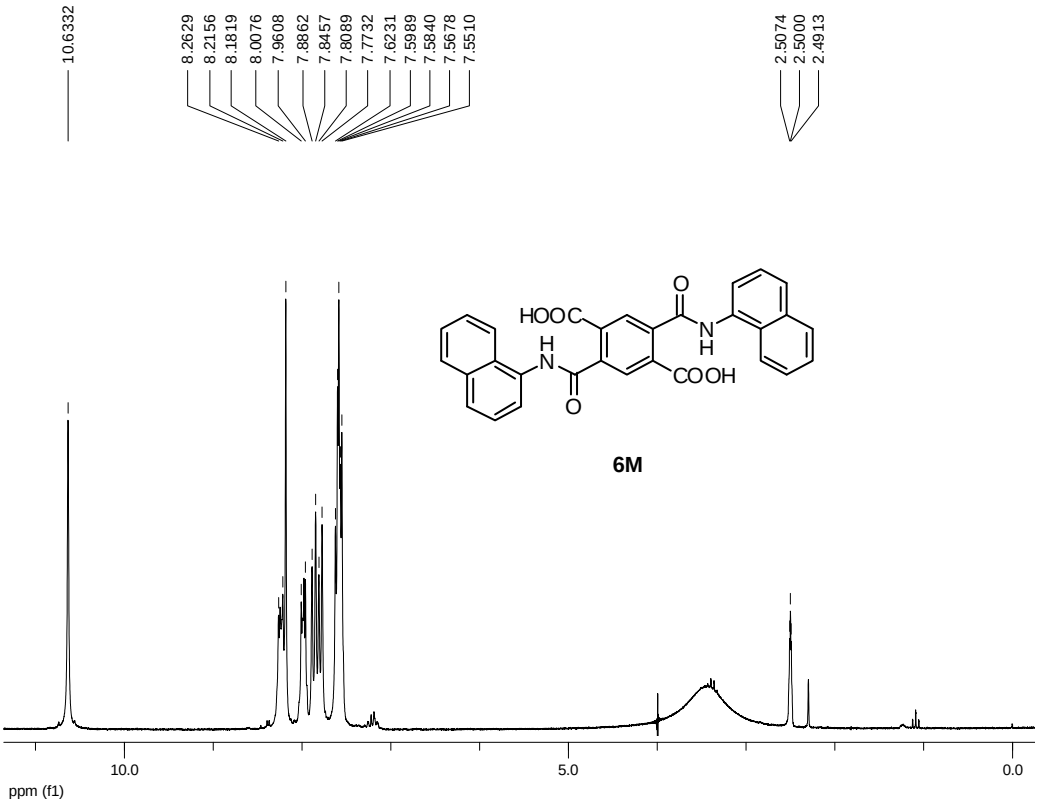

167.059
141.873
138.789
133.102
128.392
128.268
125.709
40.336
40.318
39.914
39.508
39.495
39.073
38.669
32.573
30.730
HOOC
COOH
150
100
50
0
ppm (t1)

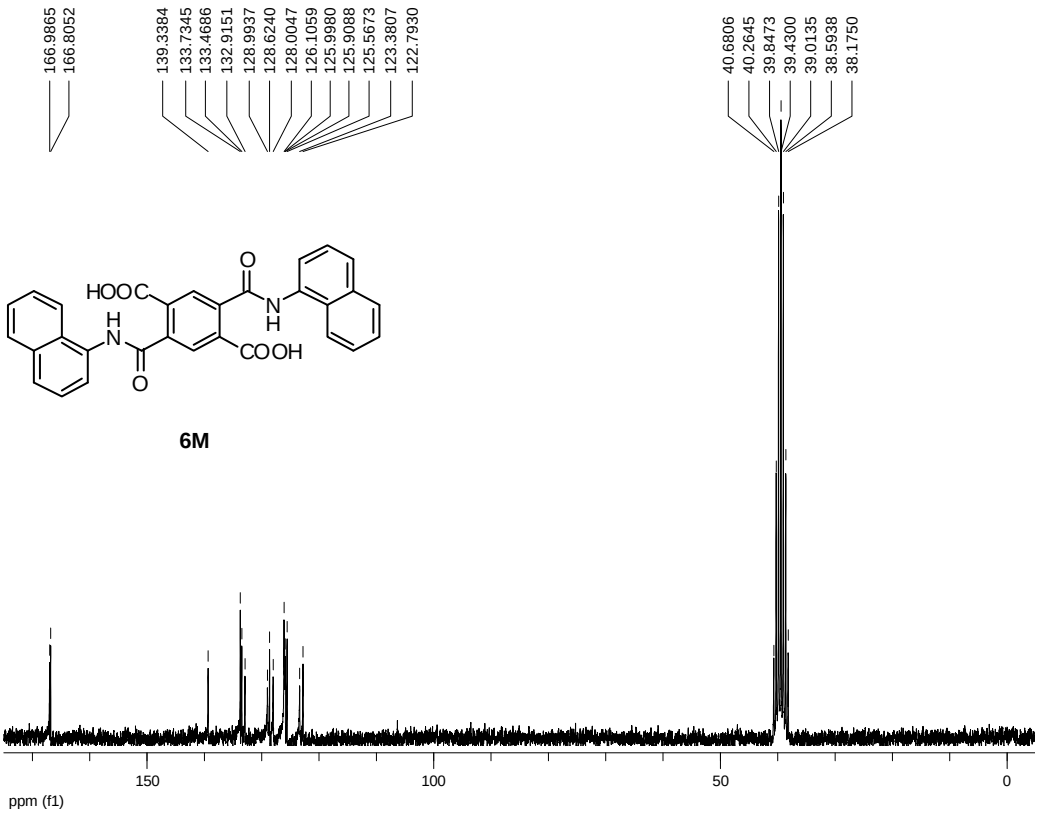
6K


8.803
8.778
8.750
7.753
7.296
6.995
6.952
6.949
4.099
3.599
3.571
2.502
2.493
0.000000
HOOC
COOH
N
H
O
6L
8.50
8.00
7.50
7.00
ppm (f1)
10.0
5.0
0.0
ppm (f1)

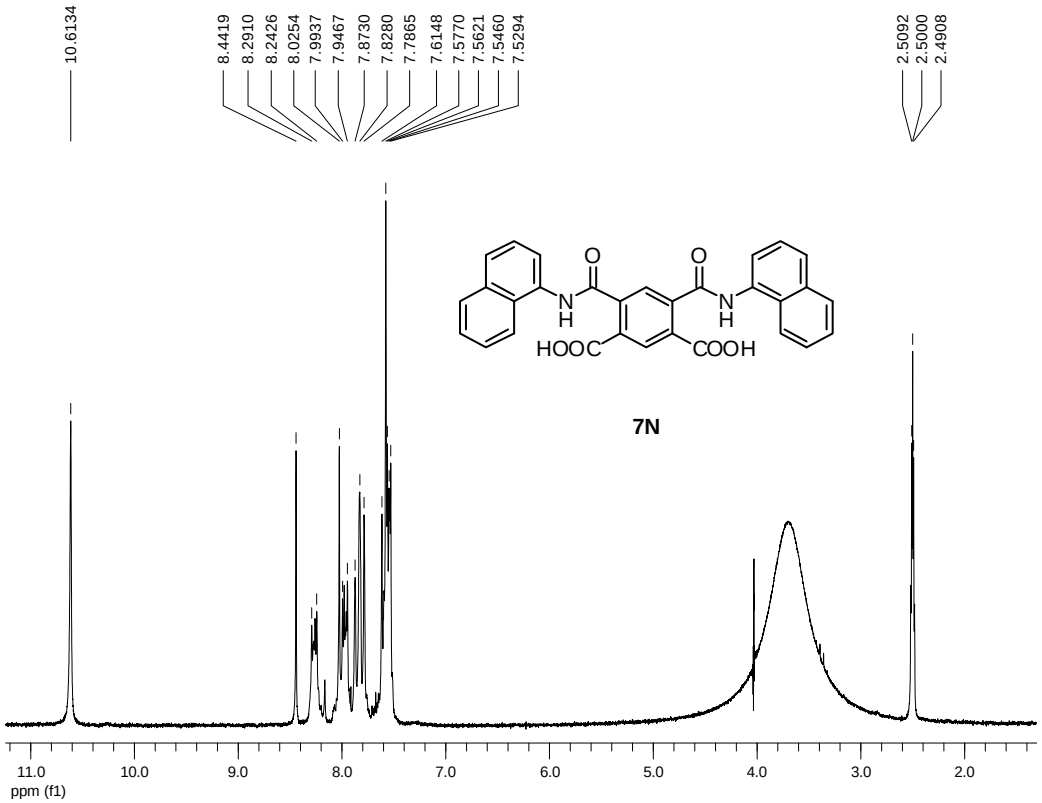

167.467
166.971
158.405
138.470
133.129
129.587
129.513
128.355
120.684
114.576
114.523
65.710
40.744
40.330
40.310
39.906
39.501
39.066
38.662
HOOC
COOH
N
H
O
6L
150
100
50
ppm (f1)

Reaction in Toluene

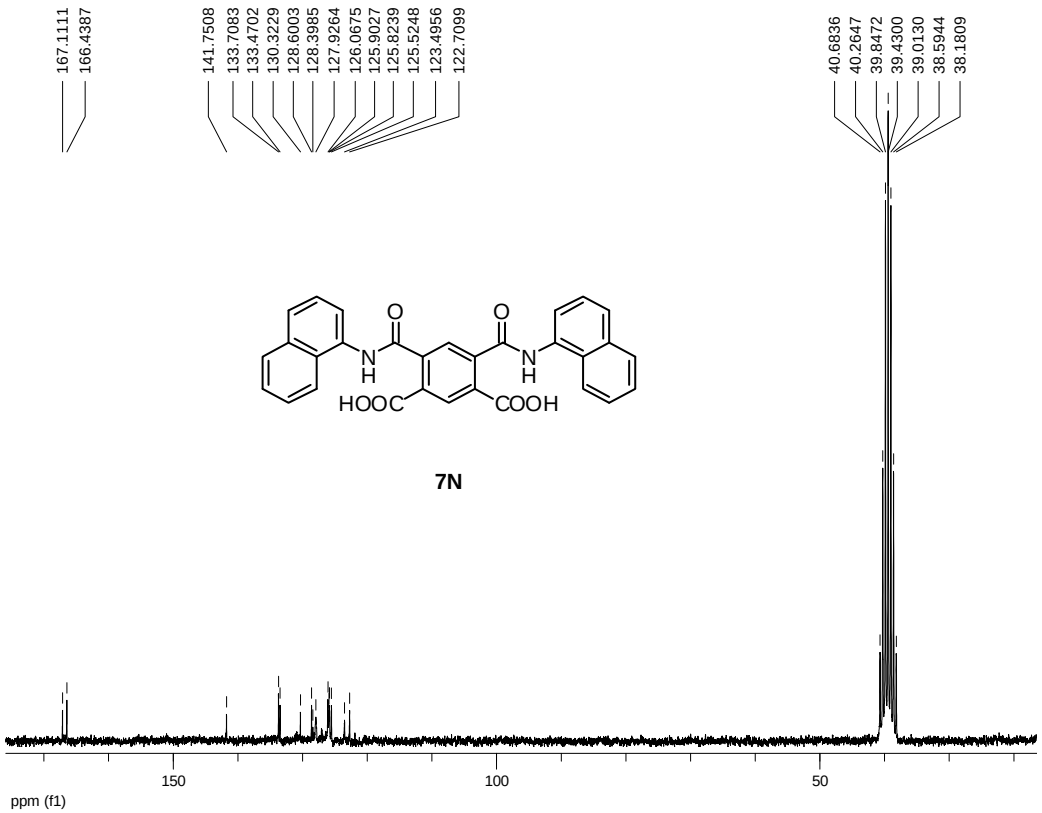




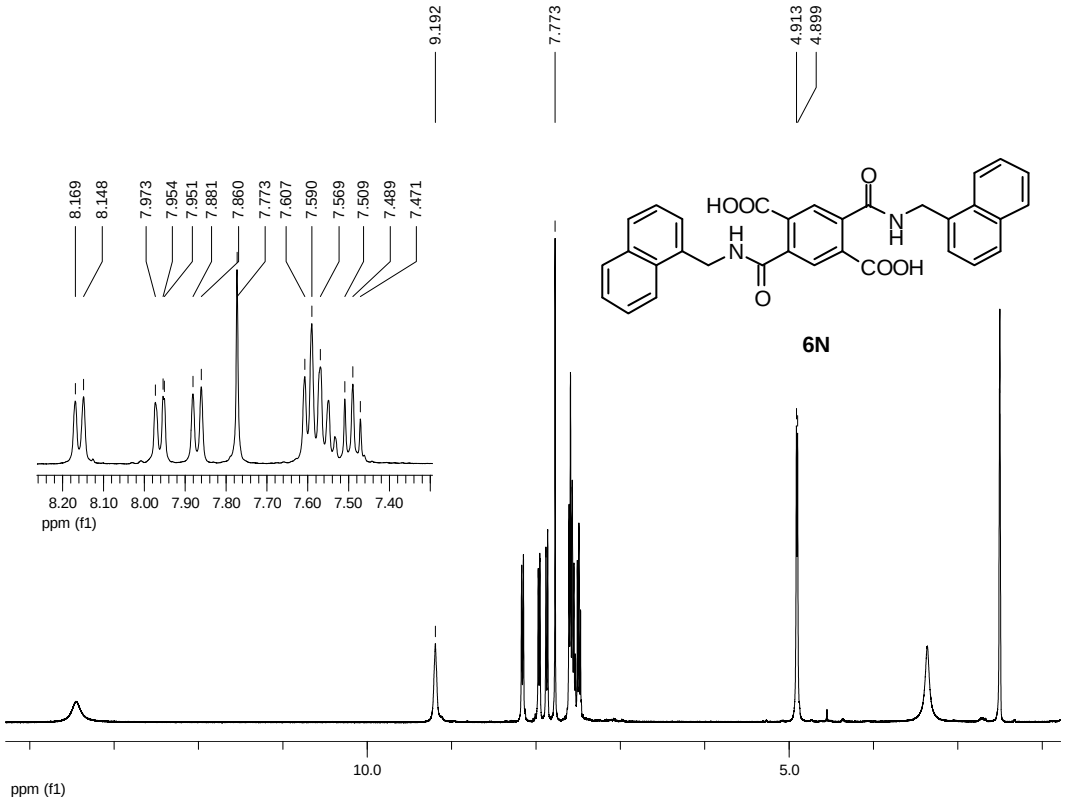
Reaction in Ethanol

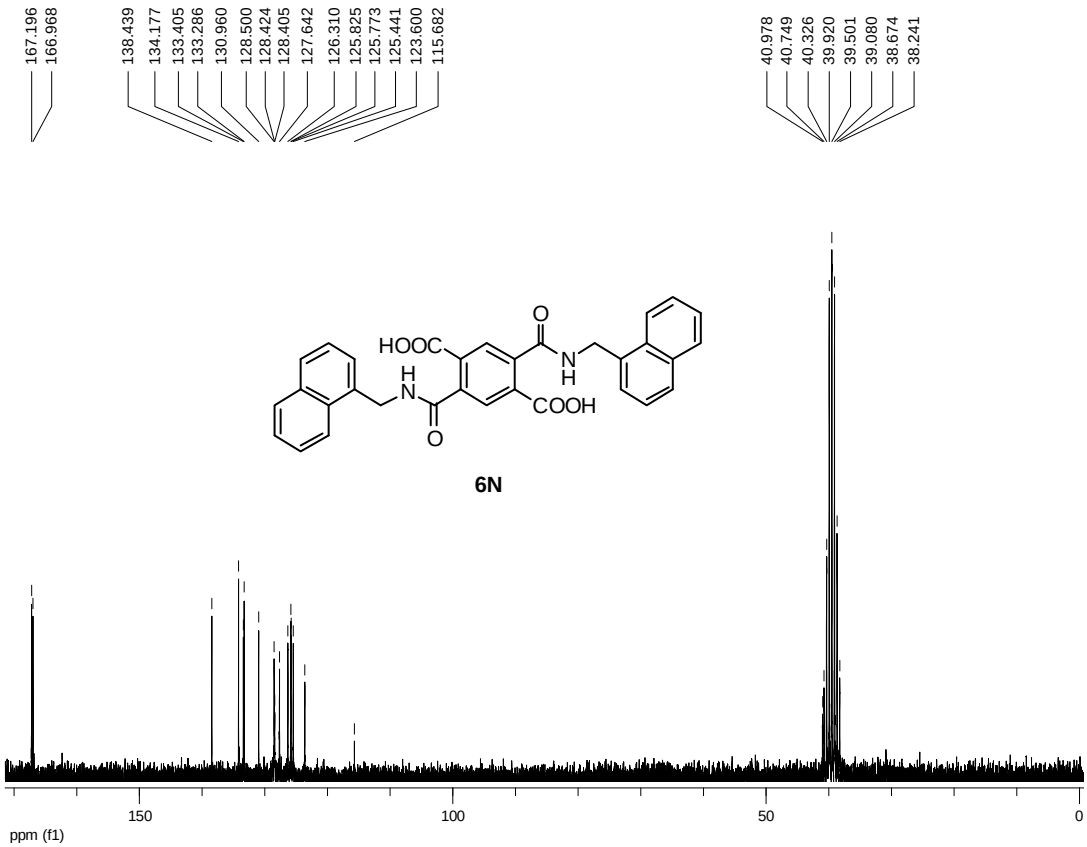


7N



7N


9.192
7.773
4.913
4.899
8.169
8.148
7.973
7.954
7.951
7.881
7.860
7.773
7.607
7.590
7.569
7.509
7.489
7.471
8.20 8.10 8.00 7.90 7.80 7.70 7.60 7.50 7.40
ppm (f1)
HOOC
H
N
O
O
N
H
COOH
6N
10.0
5.0
ppm (f1)


167.196
166.968
138.439
134.177
133.405
133.286
130.960
128.500
128.424
128.405
127.642
126.310
125.825
125.773
125.441
123.600
115.682
40.978
40.749
40.326
39.920
39.501
39.080
38.674
38.241
HOOC
H
N
O
O
N
H
COOH
6N
150
100
50
0
ppm (f1)