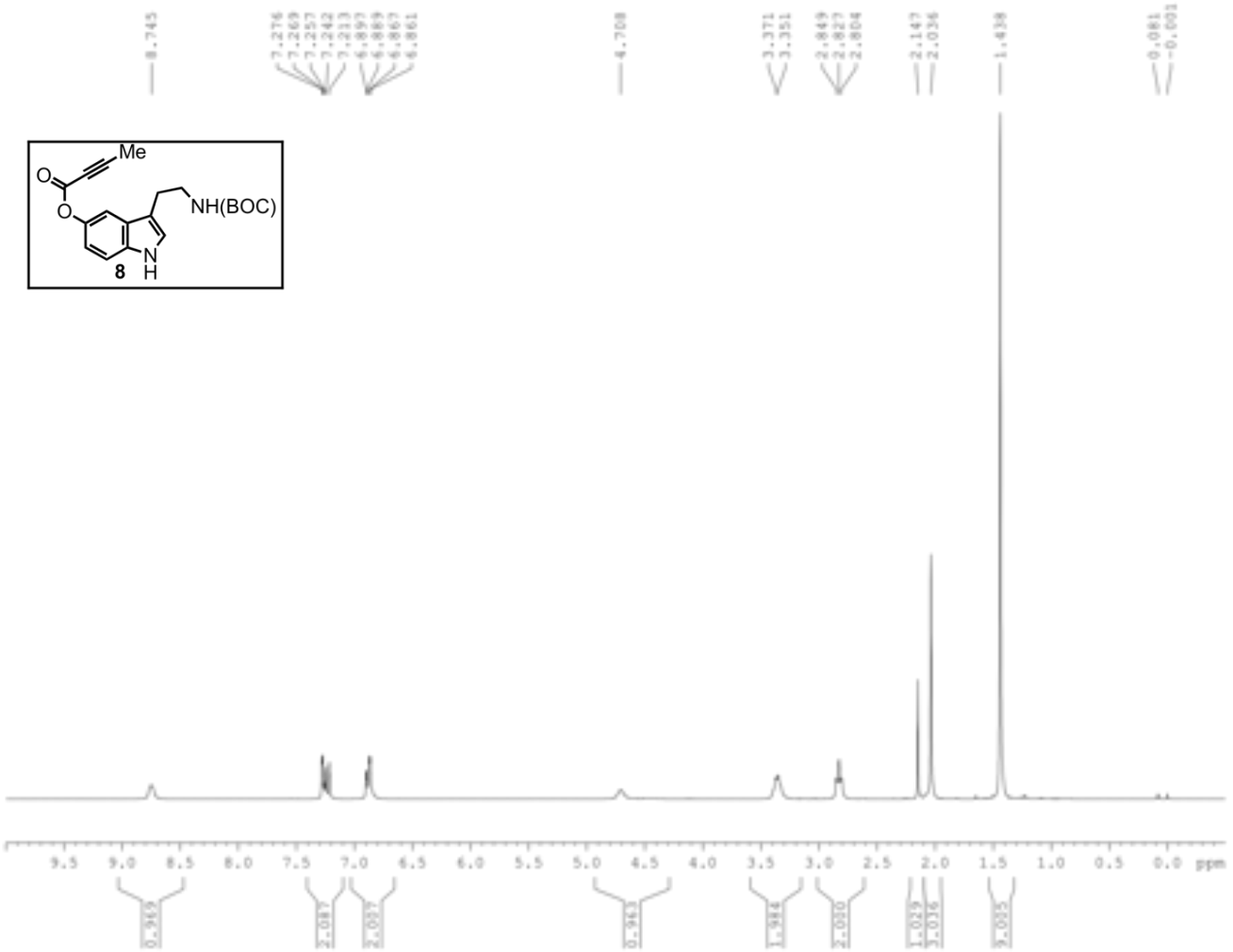
Catalytic Coupling of Arene C-H Bonds and Alkynes for the Synthesis of Coumarins: the Substrate Scope and Application to the Development of Neuroimaging Agents

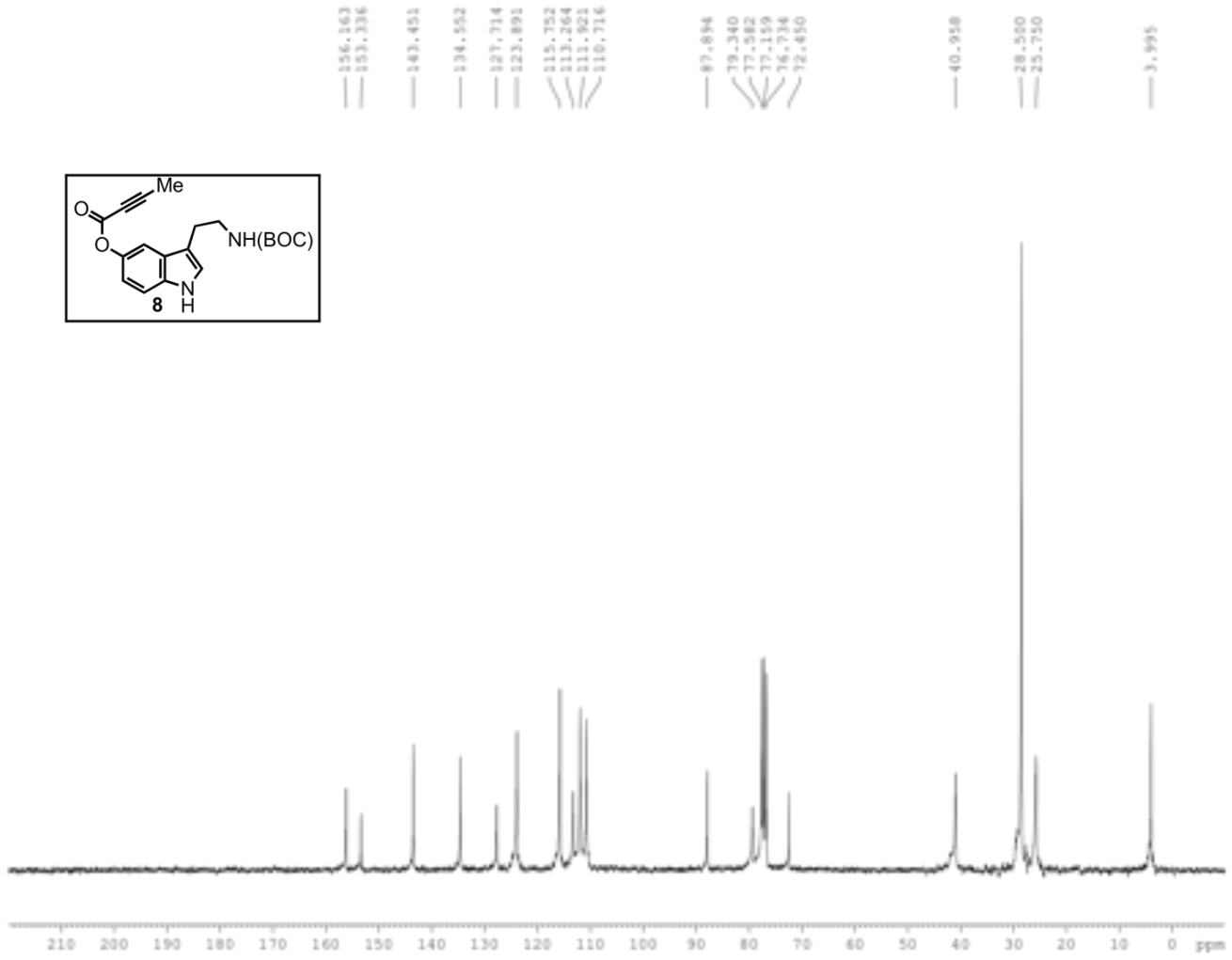
Paul A. Vadola and Dalibor Sames*

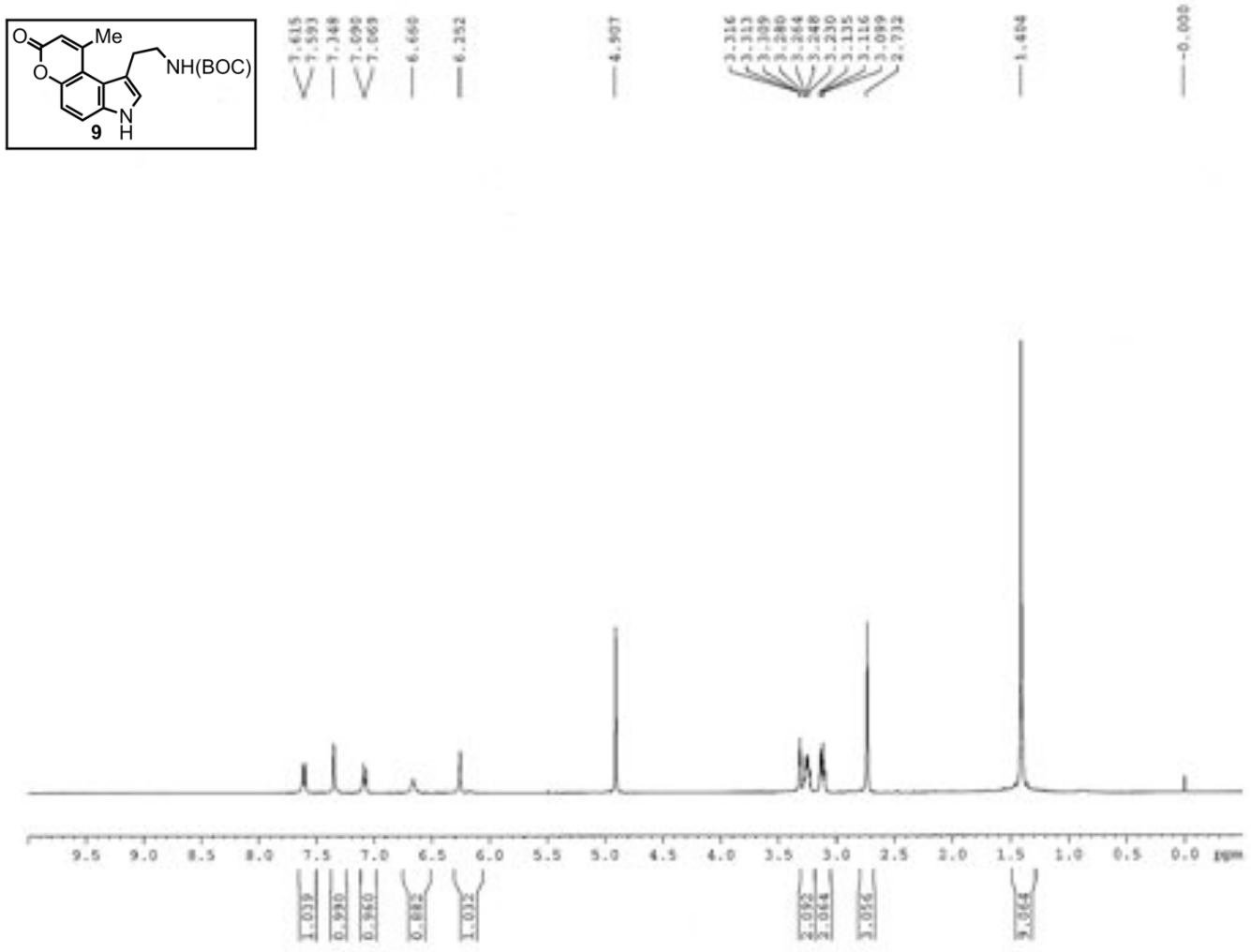
Department of Chemistry, Columbia University,
3000 Broadway, New York, New York, 10027

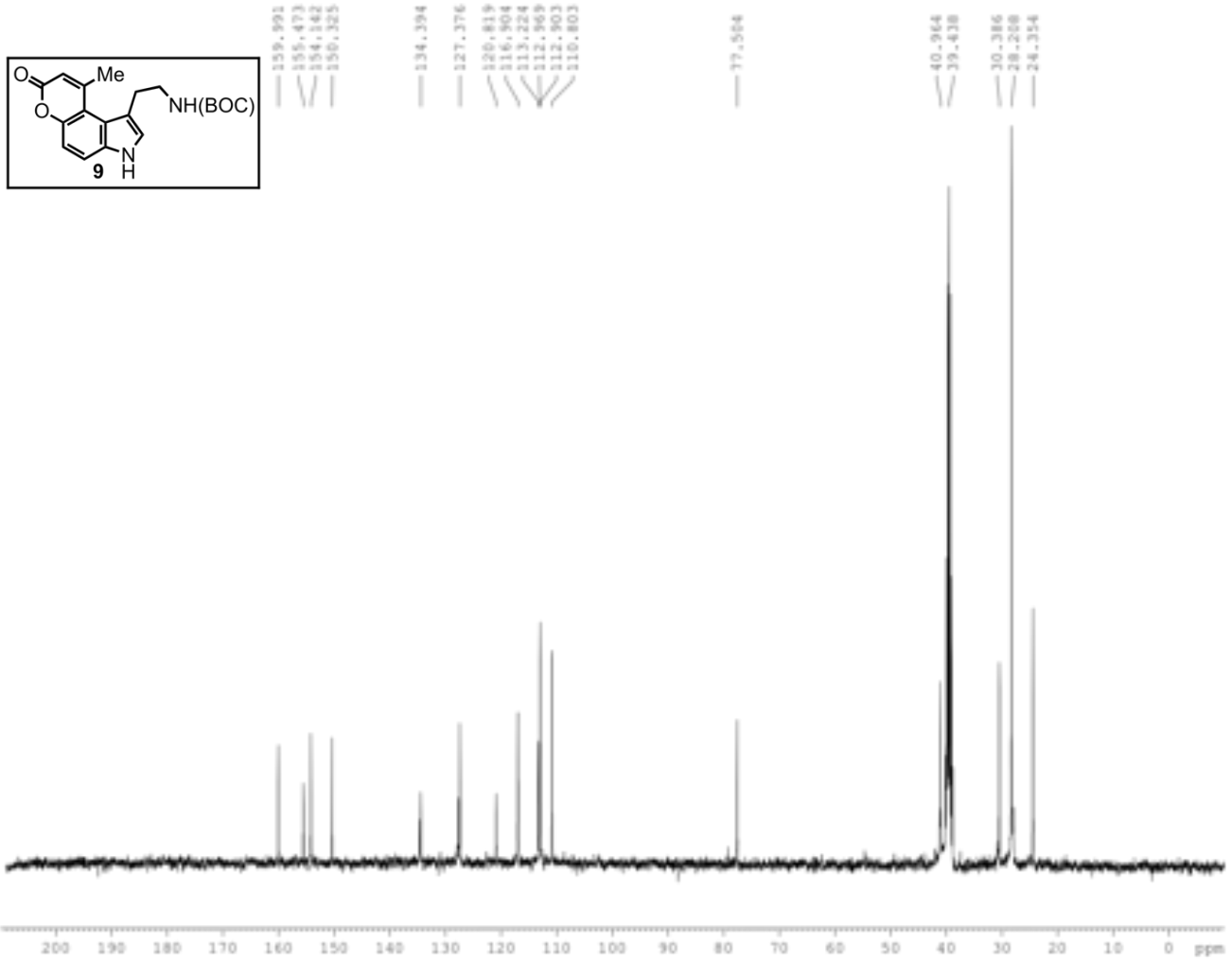
NMR Spectra

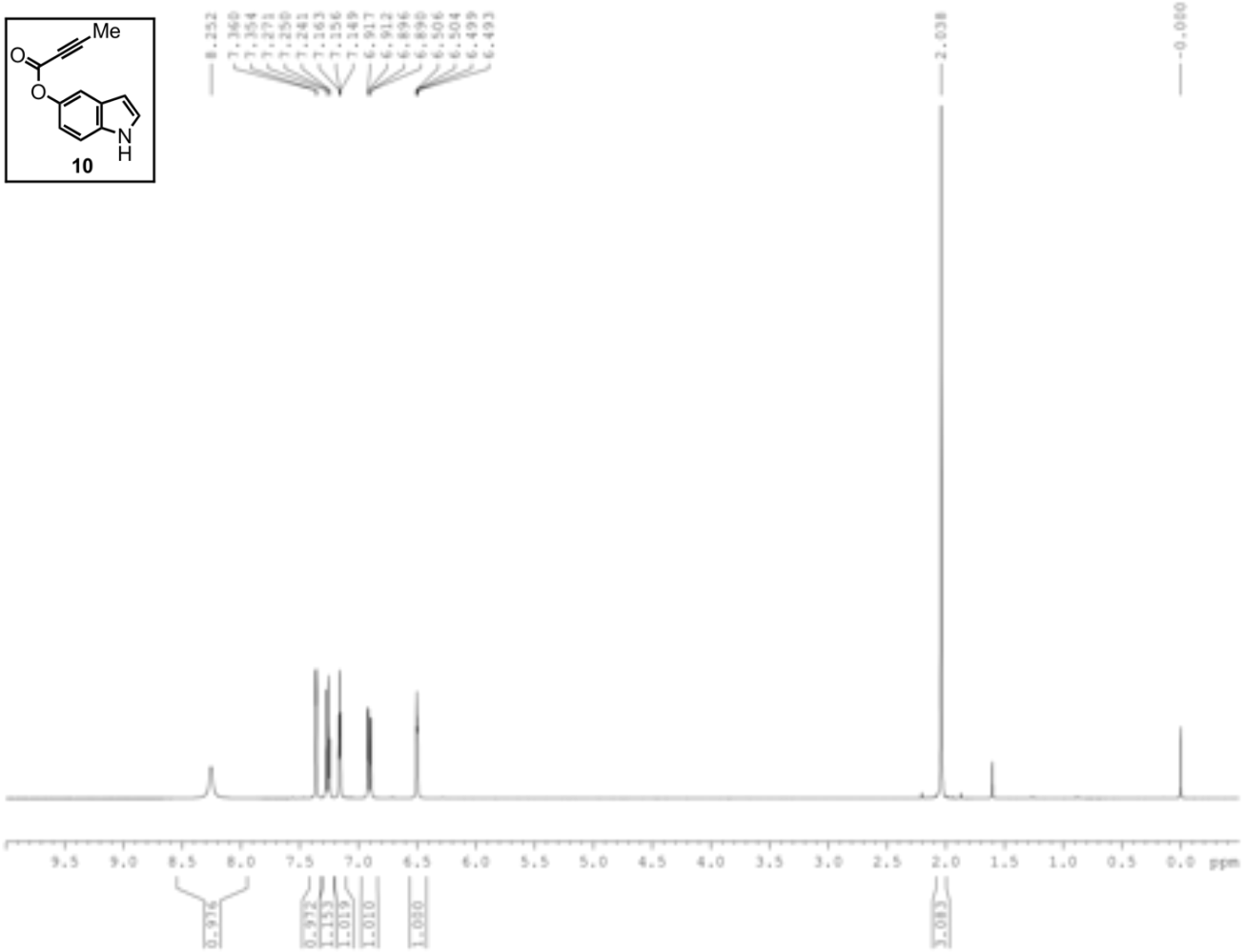
Compound.	Proton Spectrum	Carbon Spectrum
3-(2-(*tert*-butoxycarbonylamino)ethyl)-1*H*-indol-5-yl but-2-ynoate (8).	5	6
tert-butyl-9-methyl-pyrano[3,2-*e*]indol-7(*3H*)-one-1-ethylcarbamate (9)	7	8
1*H*-indol-5-yl but-2-ynoate (10).	9	10
9-methyl-pyrano[3,2-*e*]indol-7(*3H*)-one (11)	11	12
3-(2-(*tert*-butoxycarbonylamino)ethyl)-1*H*-indol-5-yl 3-phenylprop-2-ynoate (15).	13	14
tert-butyl-9-phenyl-pyrano[3,2-*e*]indol-7(*3H*)-one-1-ethylcarbamate (14)	15	16
3-(2-(*tert*-butoxycarbonylamino)-3-methoxy-3-oxopropyl)-1*H*-indol-5-yl but-2-ynoate (16)	17	18
1-(2-(*tert*-butoxycarbonylamino)-methyl-propanoate)-9-methyl-pyrano[3,2-*e*]indol-7(*3H*)-one (17)	19	20
3-(*tert*-butoxycarbonylamino)phenyl but-2-ynoate (18)	21	22
tert-butyl 4-methyl-2-oxo-2*H*-chromen-7-ylcarbamate (19)	23	24
tert-butyl 4-methyl-2-oxo-2*H*-chromen-5-ylcarbamate (20)	25	26
3-aminophenyl but-2-ynoate (21).	27	28
7-amino-4-methyl-2*H*-chromen-2-one (22)	29	30
29,30. 5-amino-4-methyl-2*H*-chromen-2-one (23)	31	32

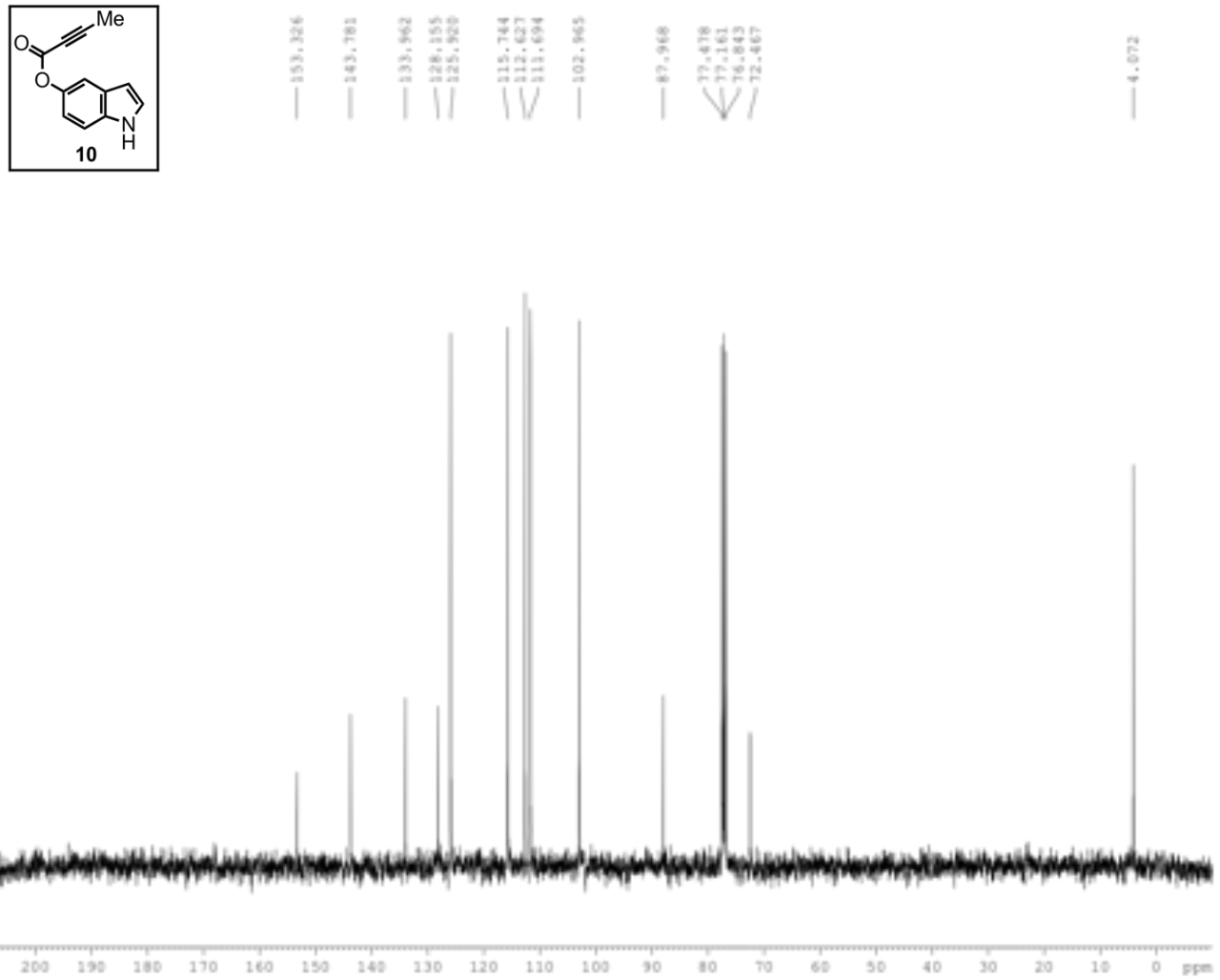

8.745
7.276
7.269
7.257
7.242
7.233
6.893
6.889
6.867
6.861
4.708
3.371
3.351
2.849
2.827
2.804
2.147
2.036
1.438
0.081
-0.001
Me
O
O
NH(BOC)
N
H
8
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.969
2.087
2.037
0.943
1.984
2.000
1.029
3.016
9.035

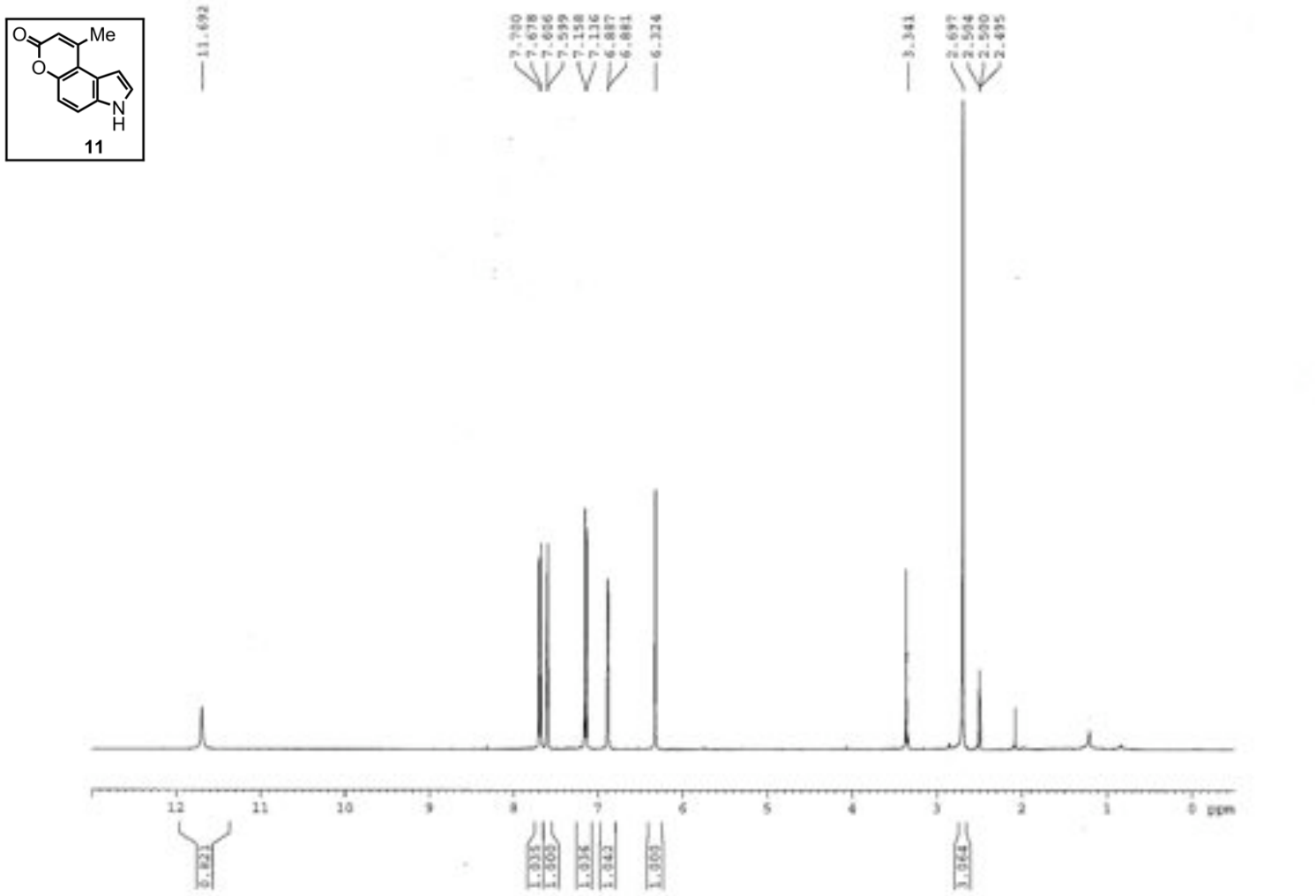

156.163
153.336
143.451
134.552
127.714
123.891
115.752
113.264
111.921
110.716
87.894
79.340
77.582
77.159
76.734
72.450
40.958
28.500
25.750
3.995
Me
O
O
NH(BOC)
N
H
8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

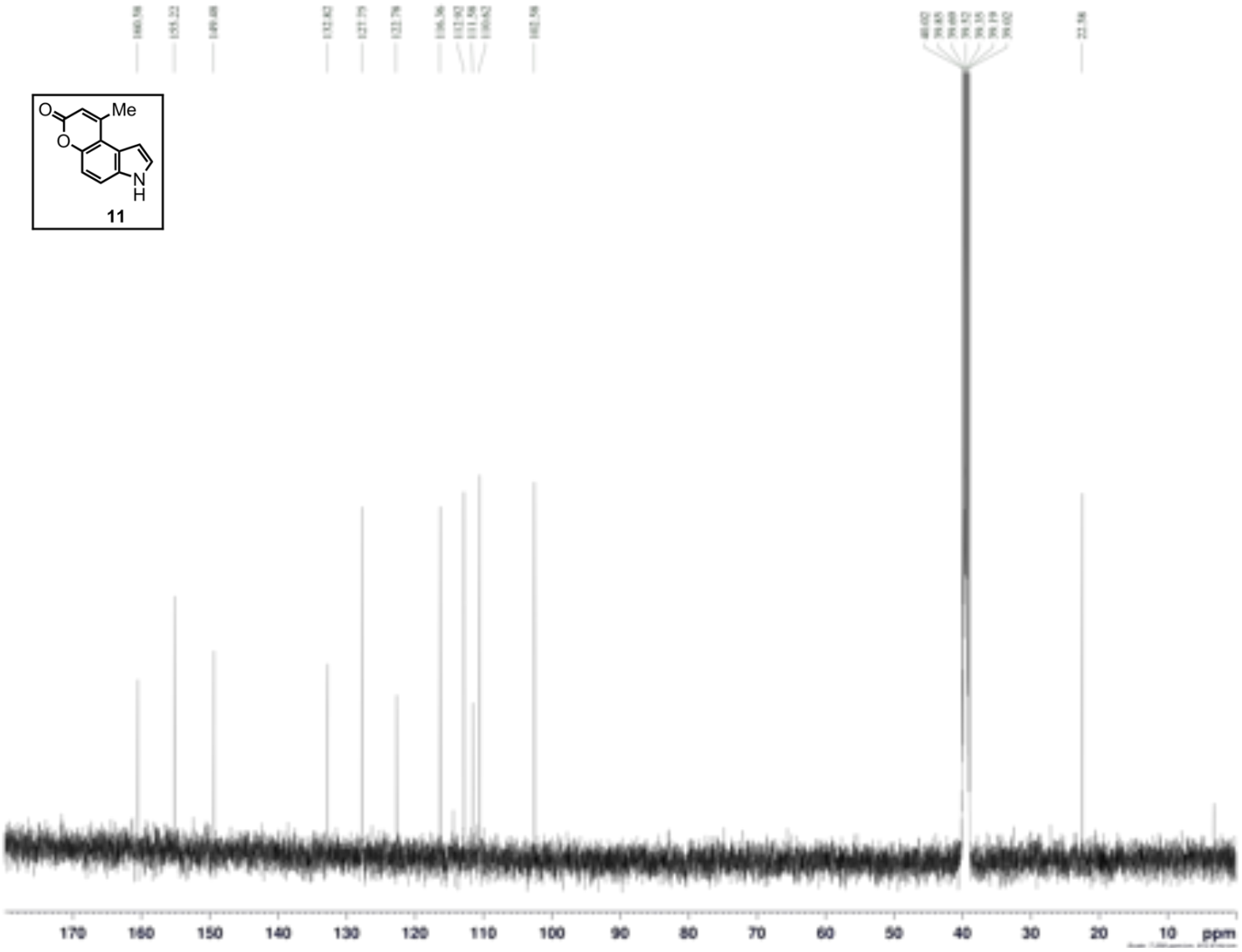

Me
O
O
NH(BOC)
N
H
9
7.615
7.593
7.348
7.090
7.069
6.660
6.252
4.907
2.316
3.313
3.309
3.280
3.264
3.248
3.230
3.135
3.116
3.099
2.732
1.404
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.019
0.990
0.960
0.885
1.017
2.002
2.004
1.000
4.000

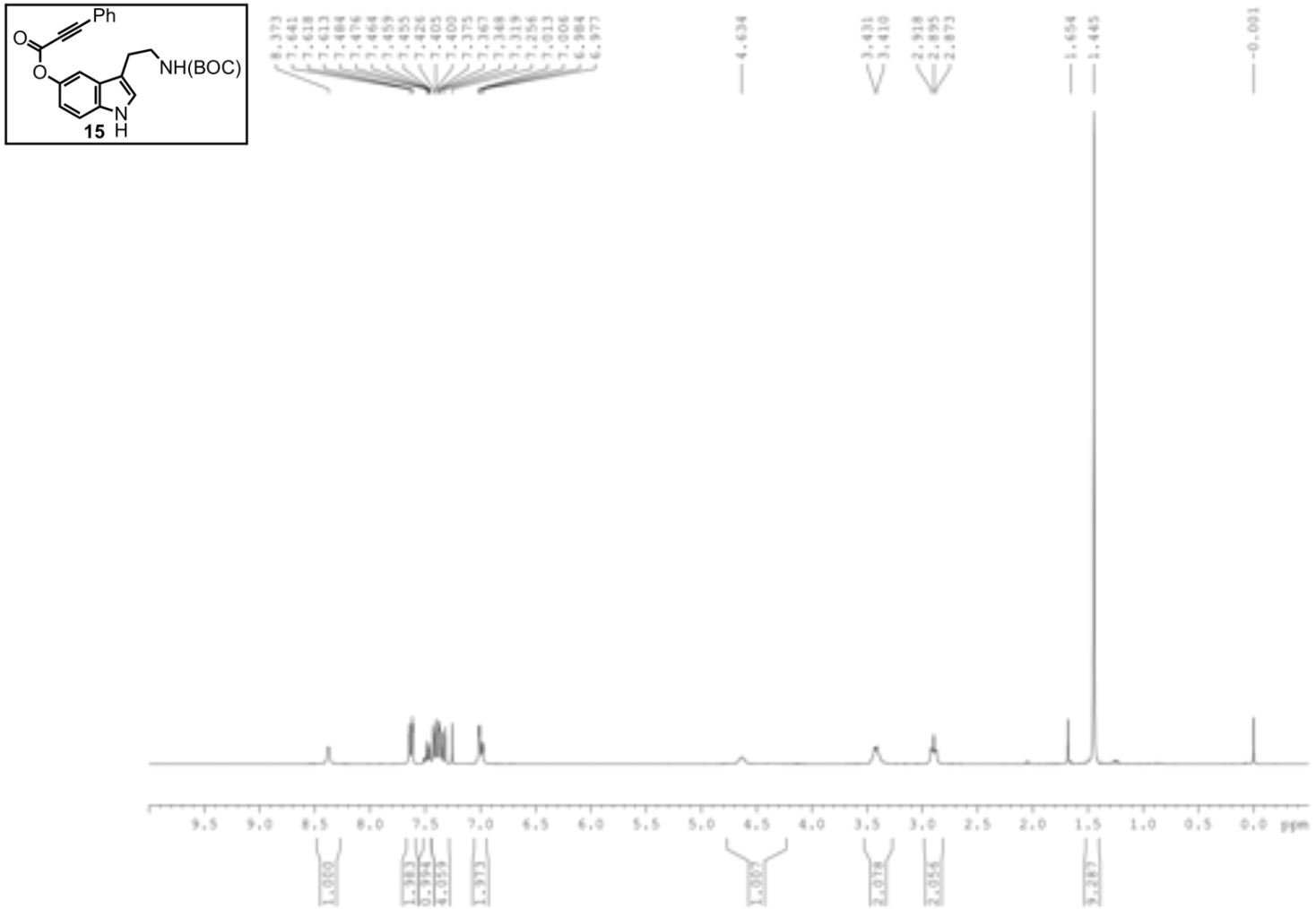

159.991
155.473
154.142
150.325
134.394
127.376
120.819
116.904
113.224
112.969
112.903
110.803
77.504
40.964
39.438
30.386
28.268
24.354
Me
O
O
NH(BOC)
N
H
9
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

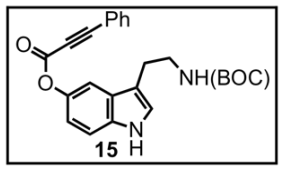

Me
O
O
N
H
10
8.252
7.360
7.354
7.271
7.250
7.241
7.163
7.156
7.149
6.917
6.912
6.896
6.890
6.506
6.504
6.499
6.493
2.038
-0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.976
0.972
1.153
1.019
1.010
1.020
3.083

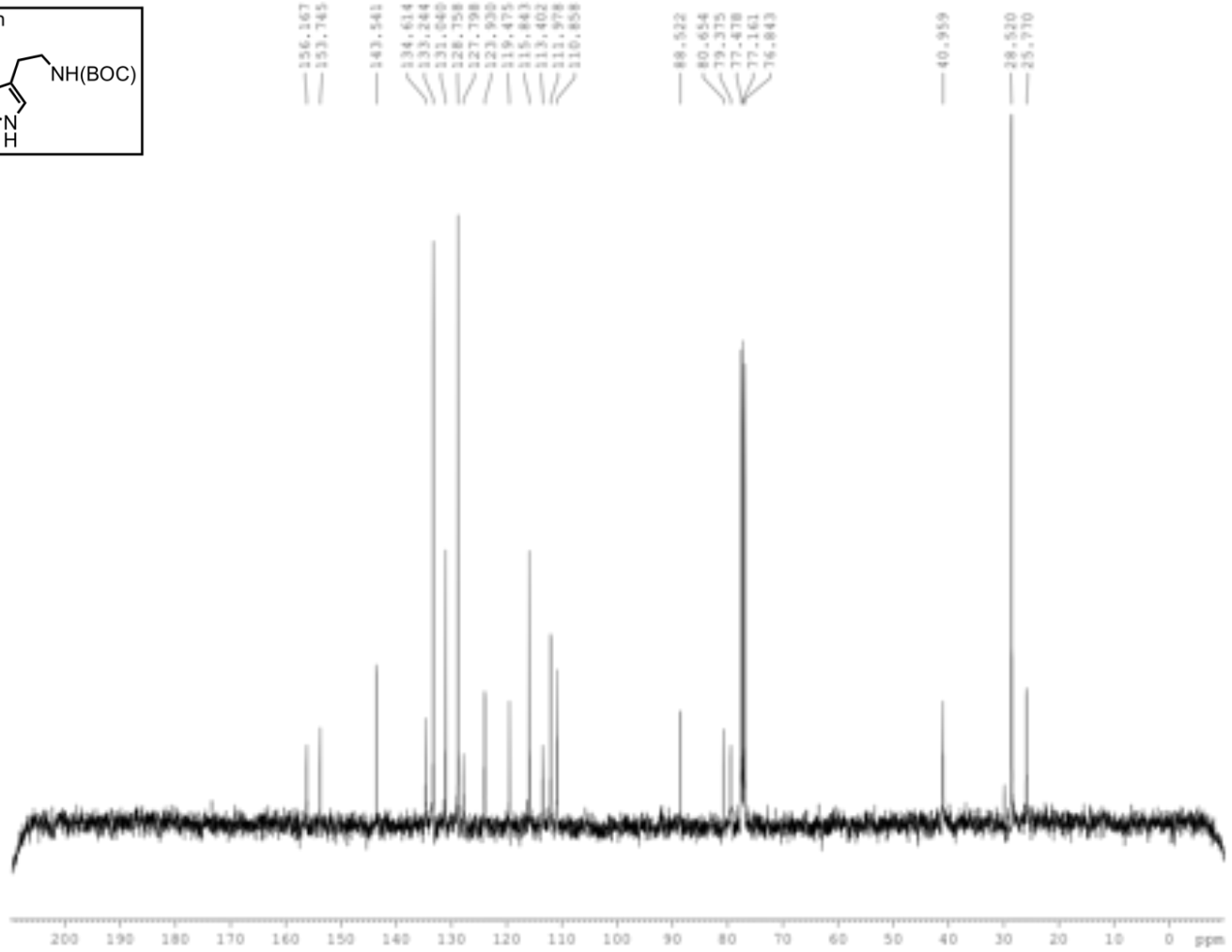

Me
O
O
N
H
10
153.326
143.781
133.962
128.155
125.920
115.744
112.627
111.694
102.965
87.968
77.478
77.161
76.843
72.467
4.072
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

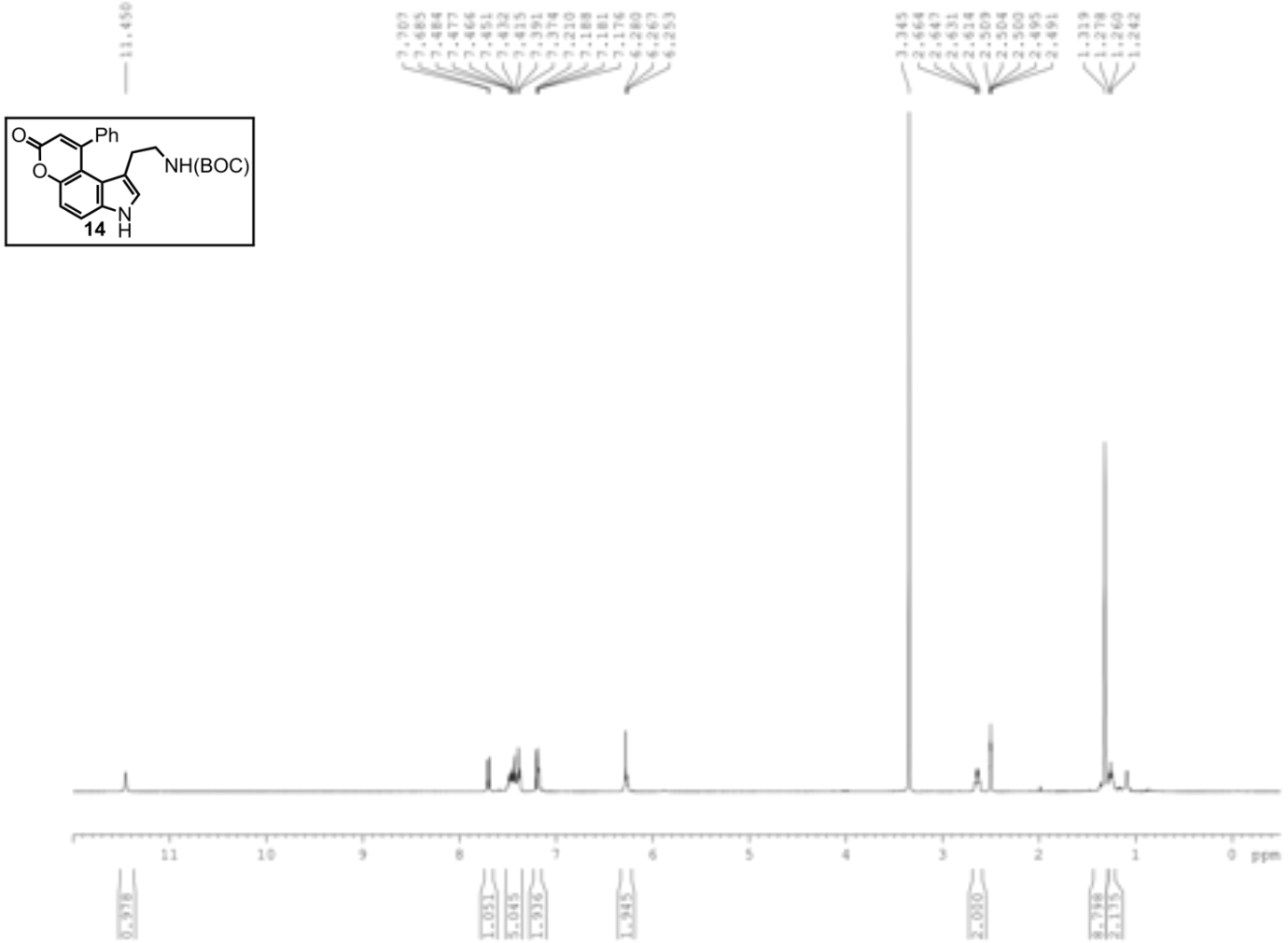

O
Me
O
N
H
11
11.692
7.700
7.678
7.606
7.599
7.158
7.136
6.887
6.881
6.324
3.341
2.697
2.504
2.500
2.495
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.822
1.035
1.000
1.036
1.042
1.000
1.964

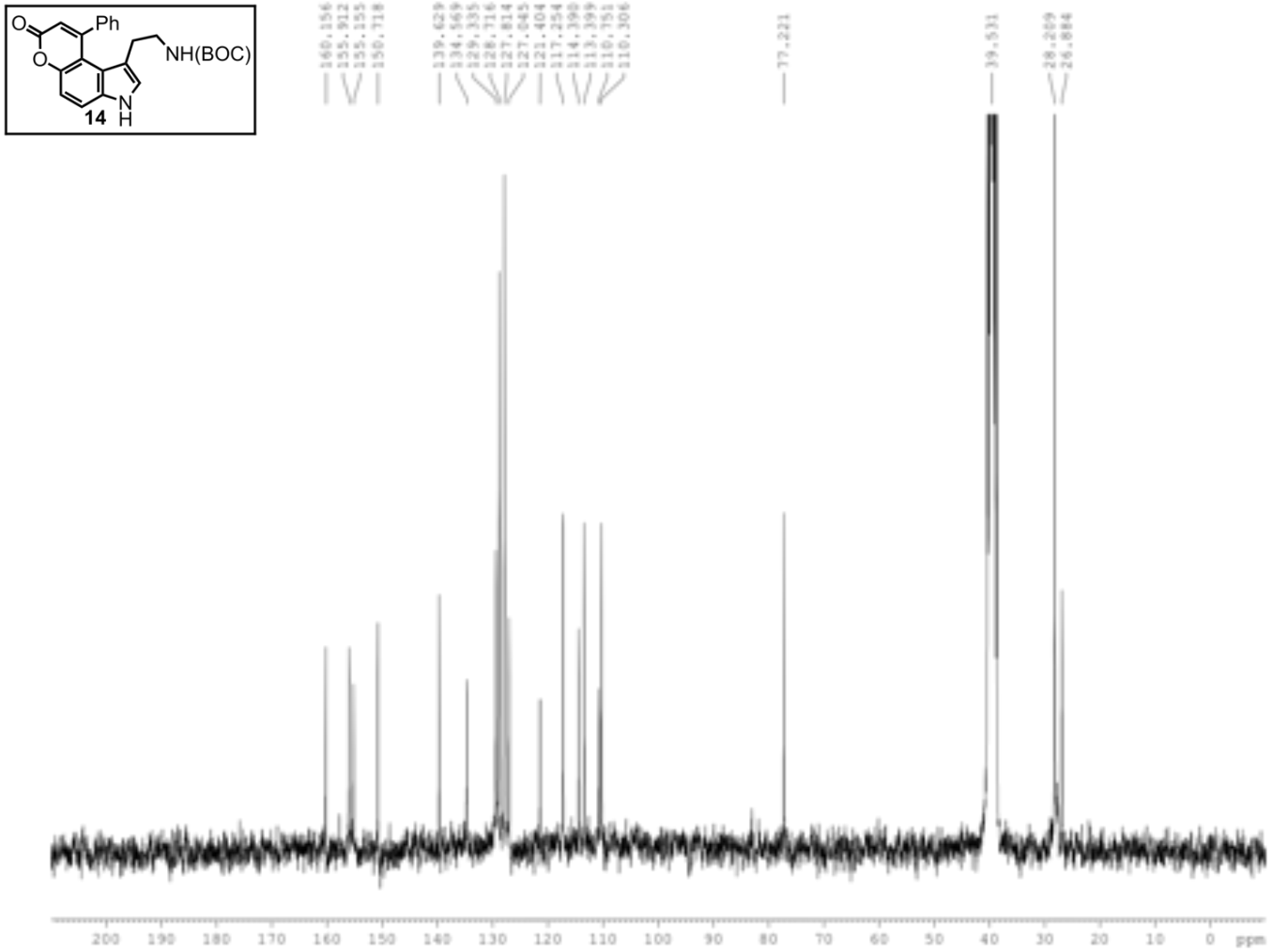

O
Me
O
N
H
11
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

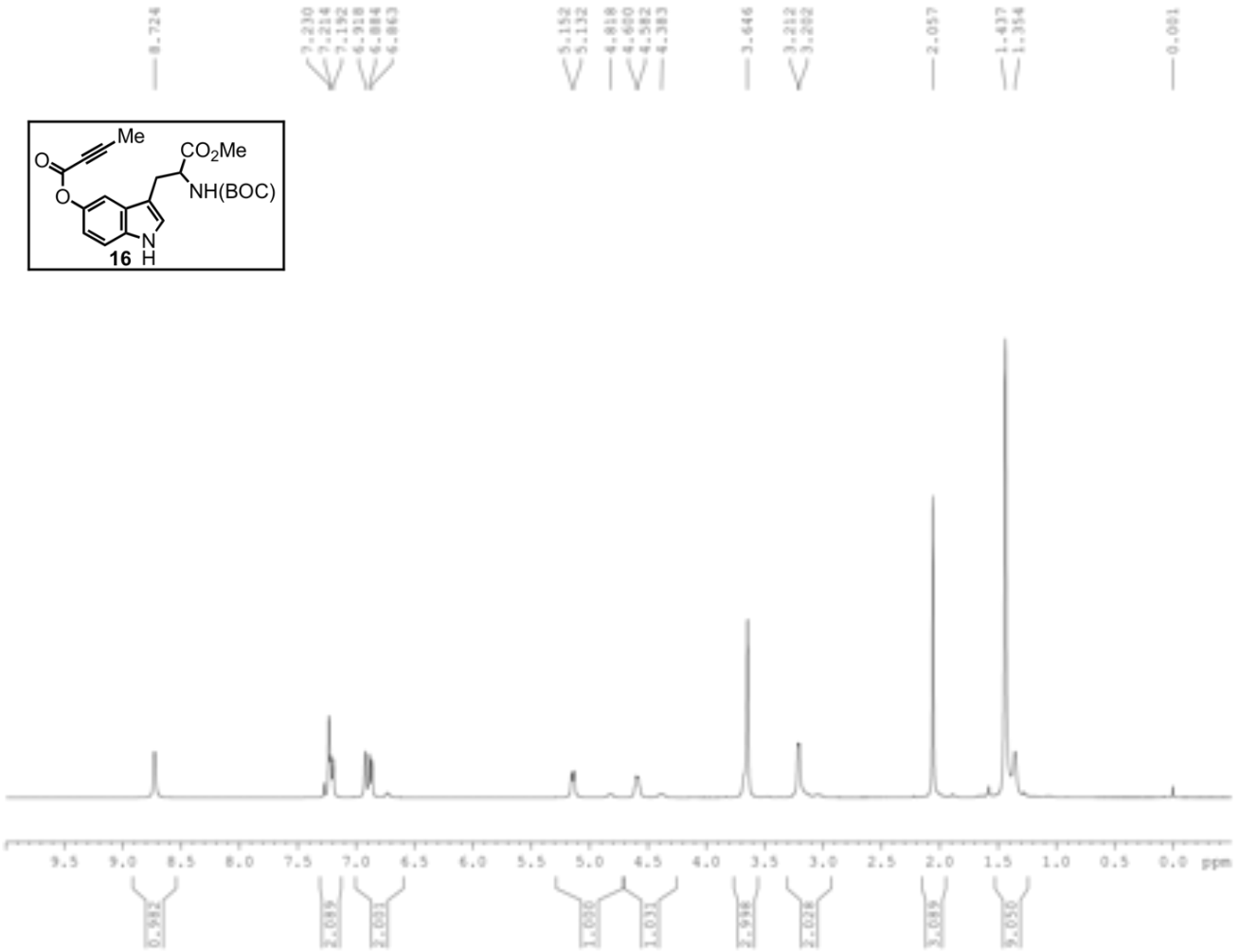

Ph
O
O
NH(BOC)
N
H
15
8.373
7.641
7.618
7.613
7.484
7.476
7.464
7.459
7.455
7.426
7.405
7.400
7.375
7.367
7.348
7.319
7.256
7.013
7.006
6.984
6.977
4.634
3.431
3.410
2.918
2.895
2.873
1.654
1.445
-0.001
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
1.983
0.994
4.059
1.913
1.000
2.018
2.056
9.287

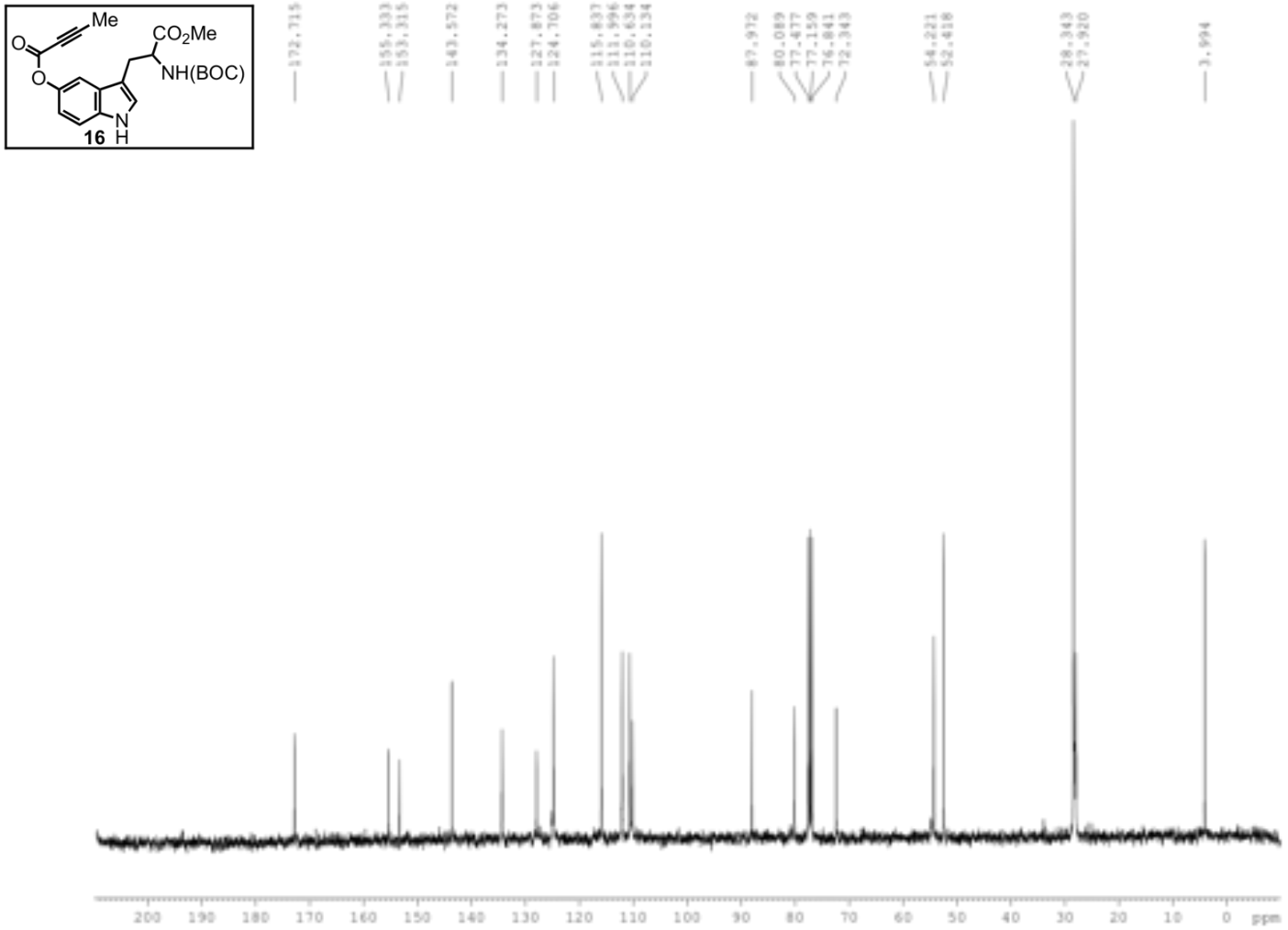

Ph
O
O
NH(BOC)
N
H
15

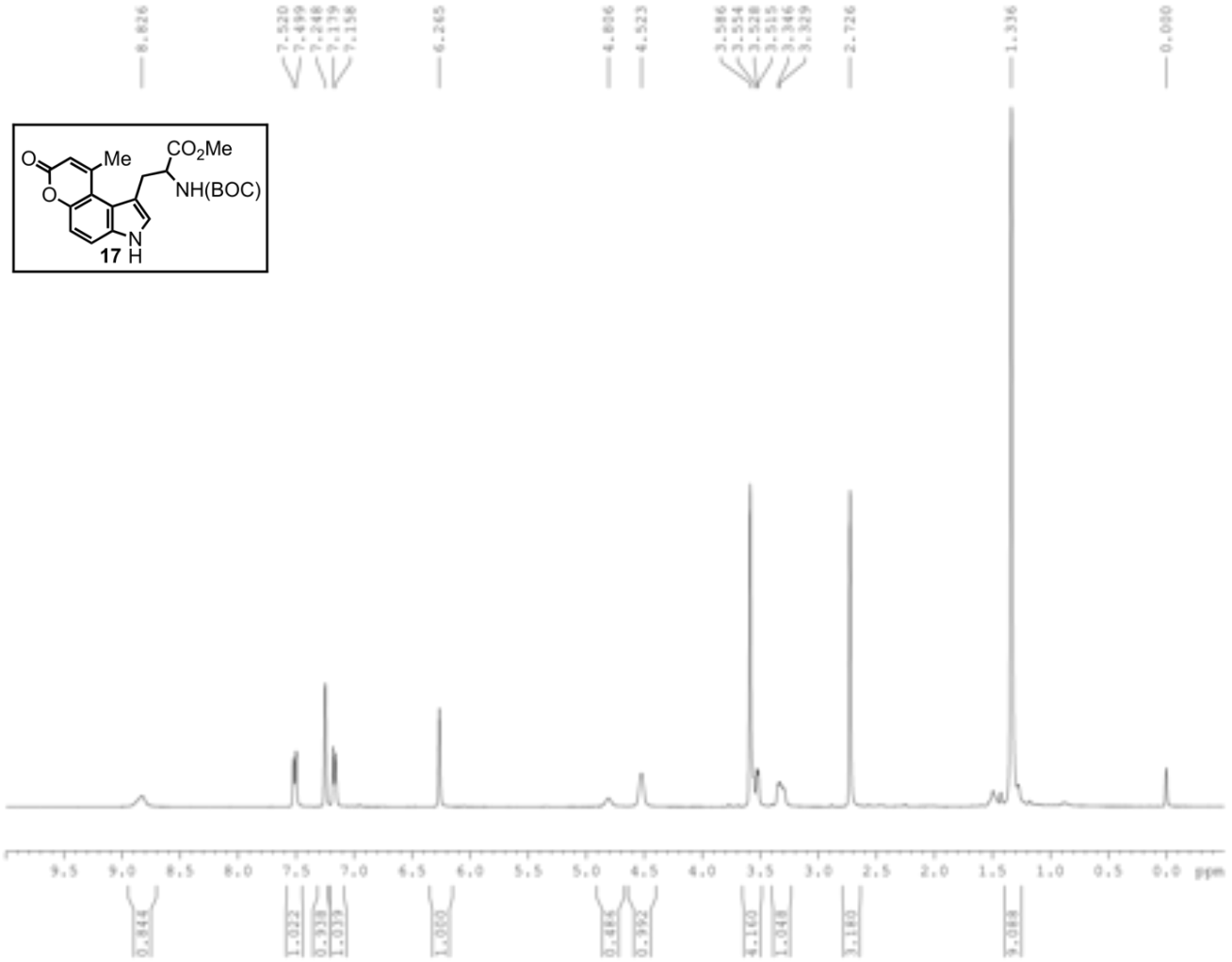

156.167
153.745
143.541
134.614
133.244
131.640
128.758
127.798
123.930
119.475
115.843
113.402
111.978
110.858
88.522
80.654
79.375
77.478
77.161
76.843
40.959
28.520
25.770
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

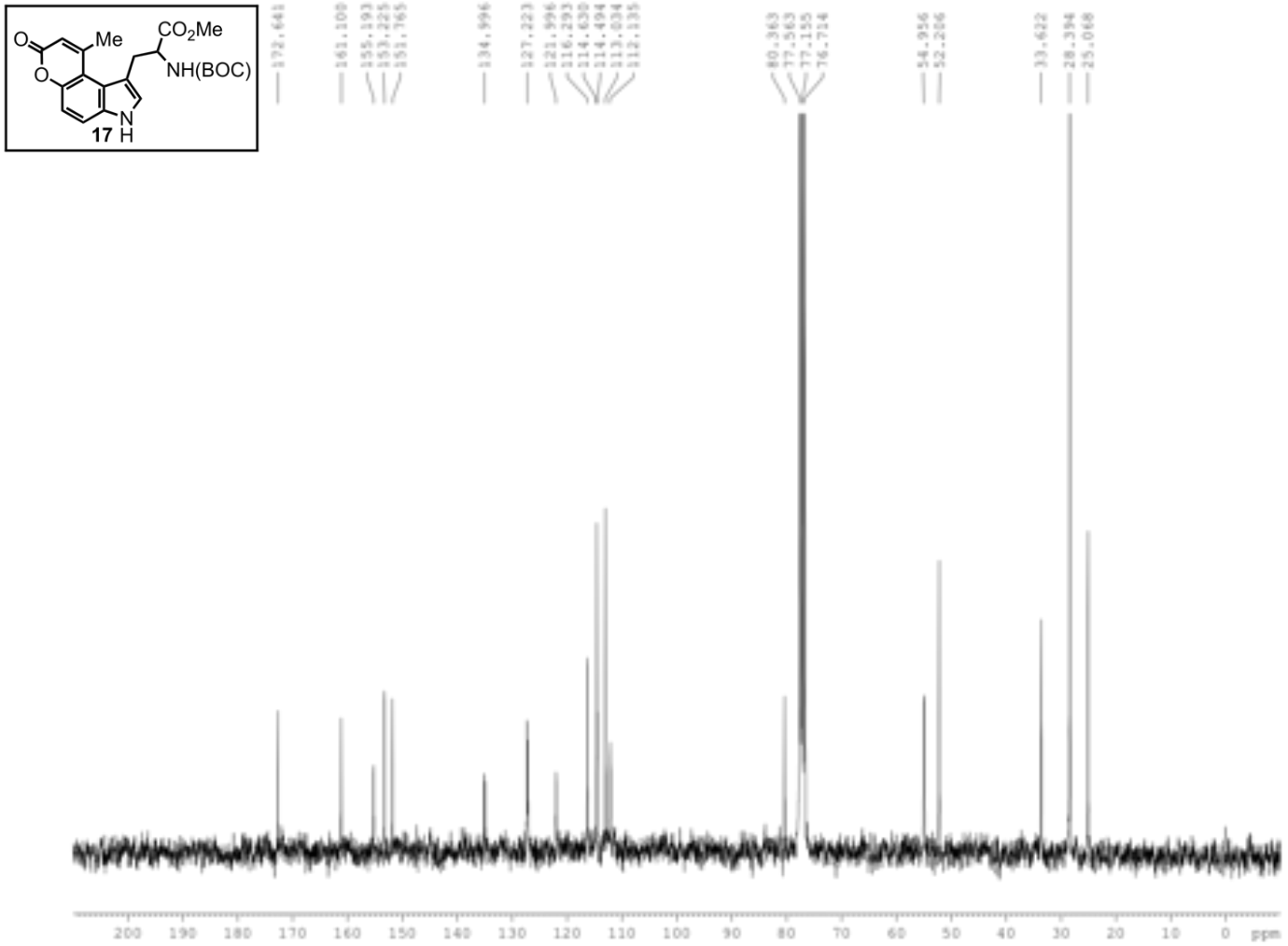

11.450
7.707
7.685
7.484
7.477
7.466
7.451
7.432
7.415
7.391
7.374
7.210
7.188
7.181
7.176
6.280
6.267
6.253
3.345
2.664
2.647
2.631
2.614
2.509
2.504
2.500
2.495
2.491
1.319
1.278
1.260
1.242
O
O
Ph
NH(BOC)
N
H
14
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.978
1.051
5.045
1.936
1.945
2.000
8.298
2.115

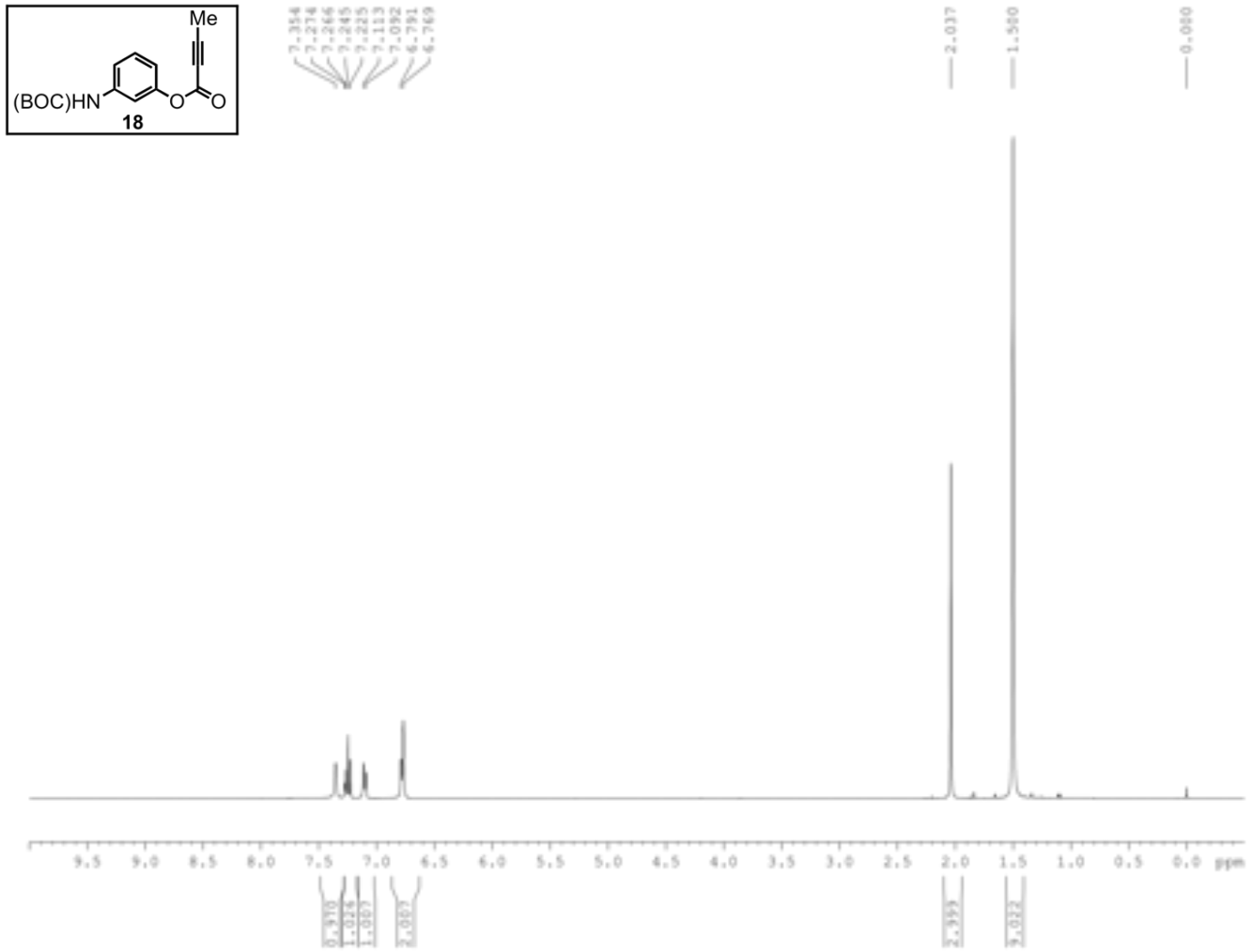

O
Ph
O
NH(BOC)
N
H
14
160.156
155.912
155.155
150.718
139.629
134.569
129.335
128.716
127.814
127.645
121.404
117.254
114.390
113.399
110.751
110.306
77.221
39.531
28.269
26.884
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

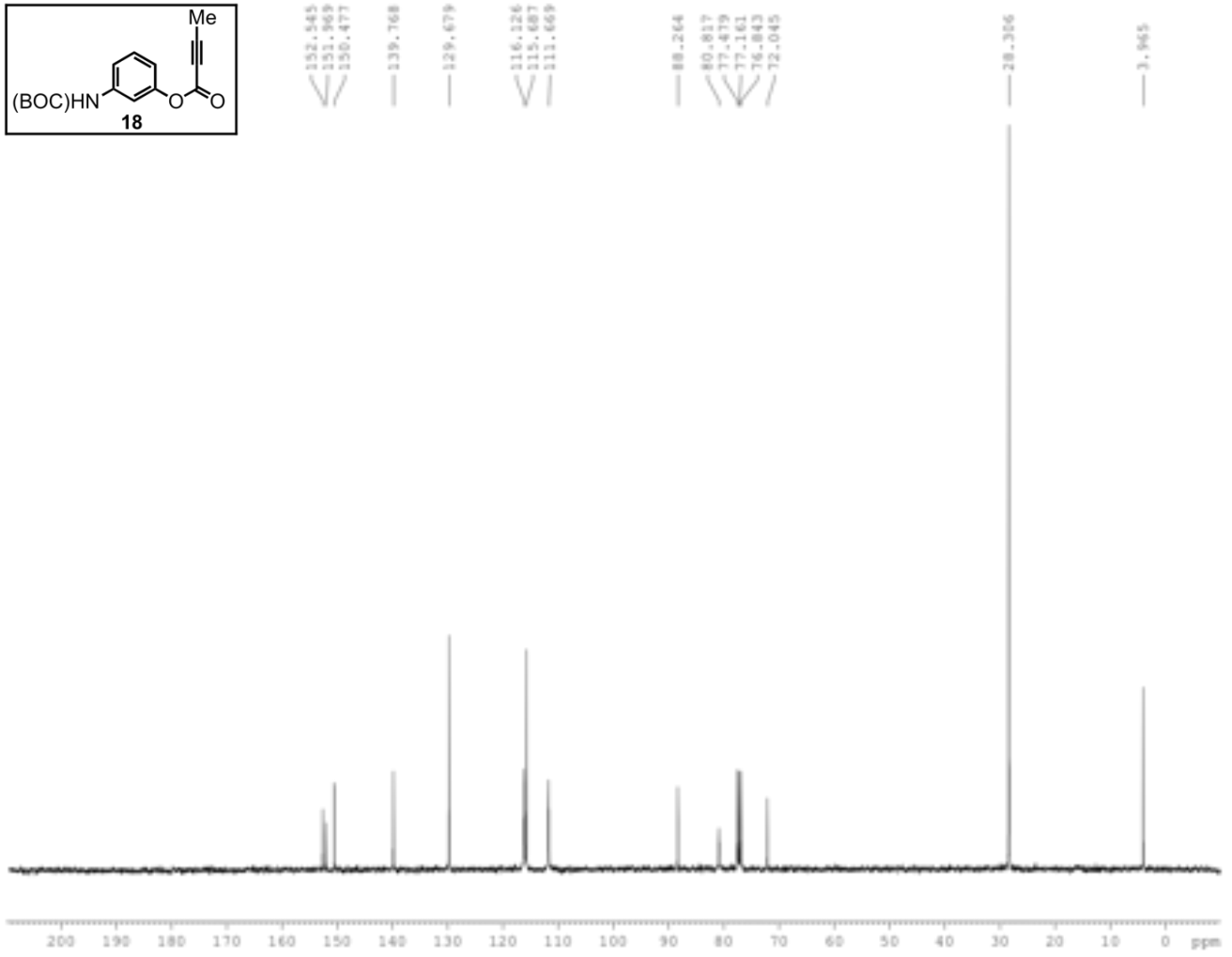

8.724
7.230
7.214
7.192
6.918
6.884
6.863
5.152
5.132
4.618
4.600
4.582
4.383
3.646
3.212
3.202
2.057
1.437
1.354
0.001
Me
O
O
CO2Me
NH(BOC)
N
H
16
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.982
2.089
2.001
1.000
1.033
2.938
2.028
3.089
3.050

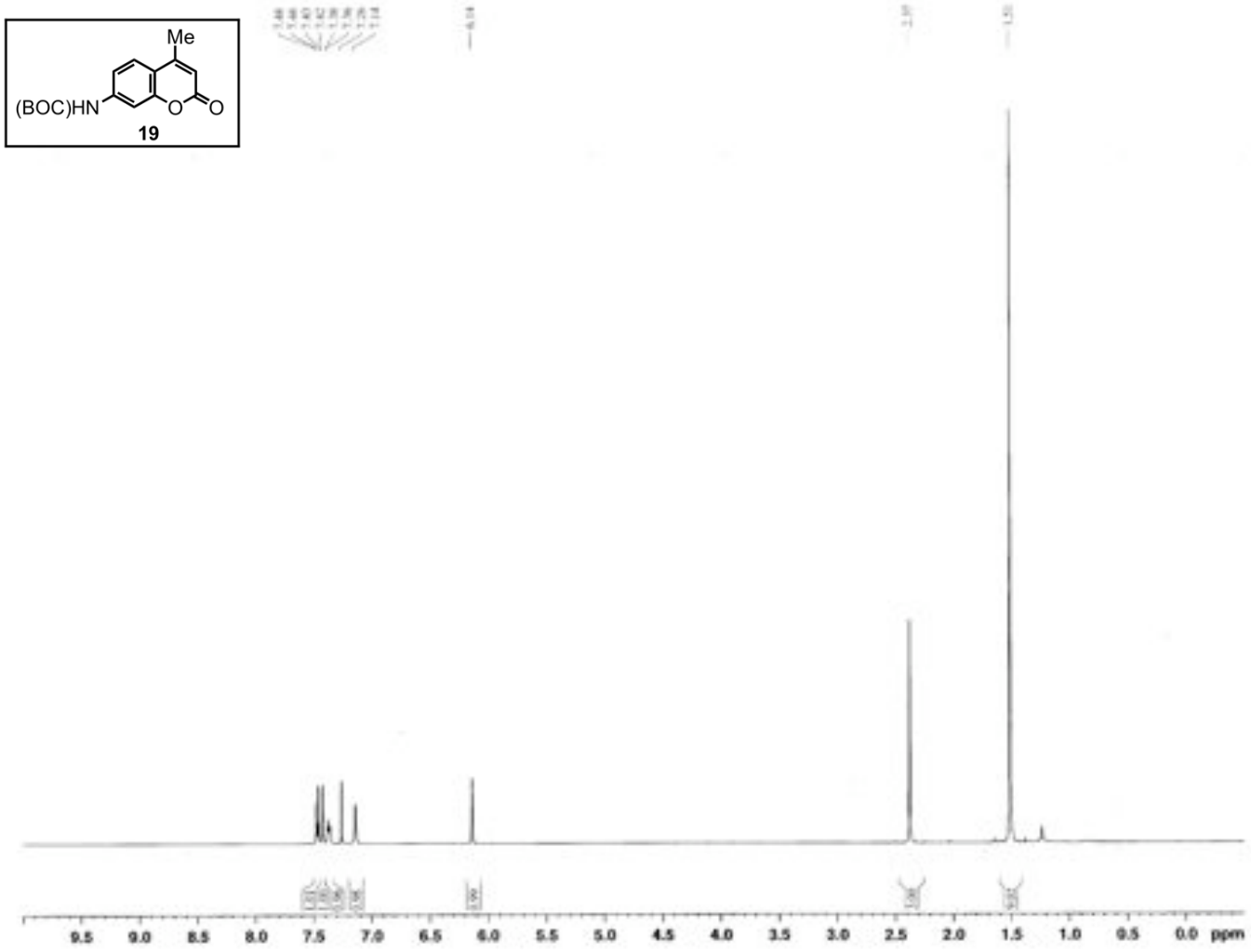

Me
O
O
CO2Me
NH(BOC)
N
H
16
172.715
155.333
153.315
143.572
134.273
127.873
124.706
115.837
111.996
110.634
110.134
87.972
80.089
77.477
77.159
76.841
72.343
54.221
52.418
28.343
27.920
3.994
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

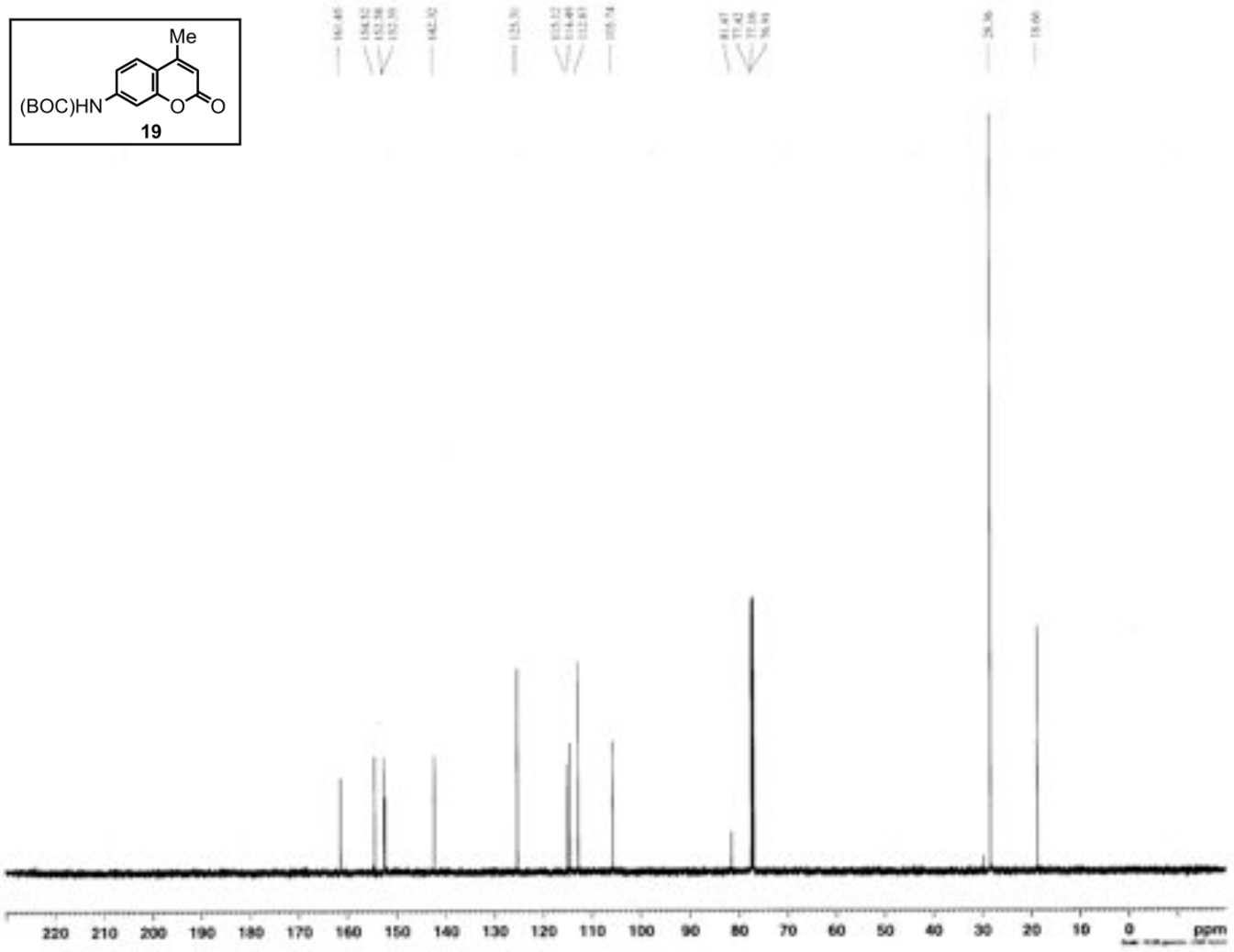

8.826
7.520
7.499
7.248
7.179
7.158
6.265
4.806
4.523
3.586
3.554
3.528
3.515
3.346
3.329
2.726
1.336
0.000
Me
CO2Me
NH(BOC)
O
O
N
H
17
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.844
1.022
0.938
1.039
1.000
0.484
0.392
4.160
1.048
3.180
9.088

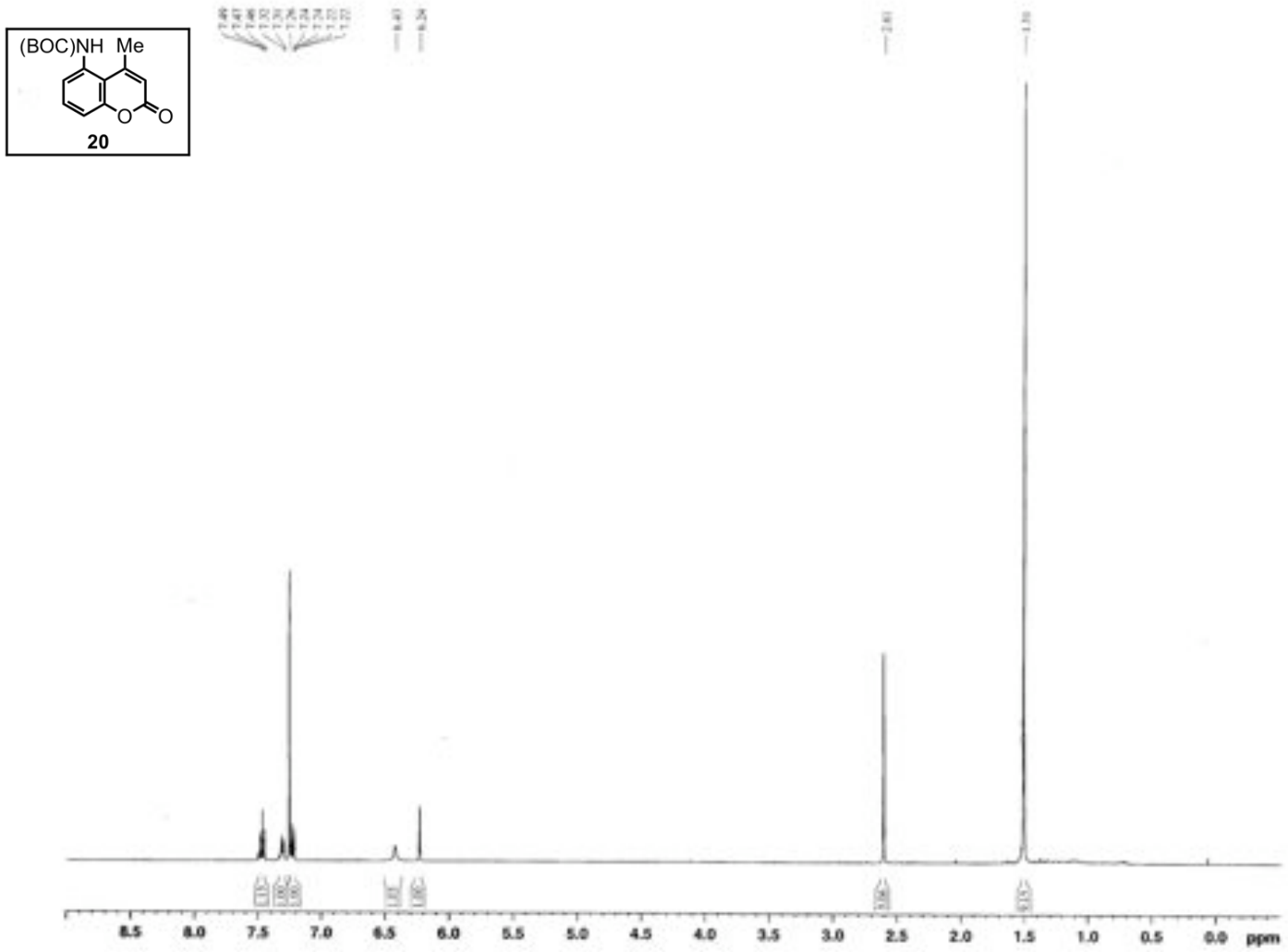

O
Me
CO2Me
NH(BOC)
O
N
H
17
172.641
161.108
155.193
153.225
151.765
134.996
127.223
121.996
116.293
114.630
114.494
113.034
112.135
80.363
77.563
77.155
76.714
54.956
52.206
33.622
28.394
25.068
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

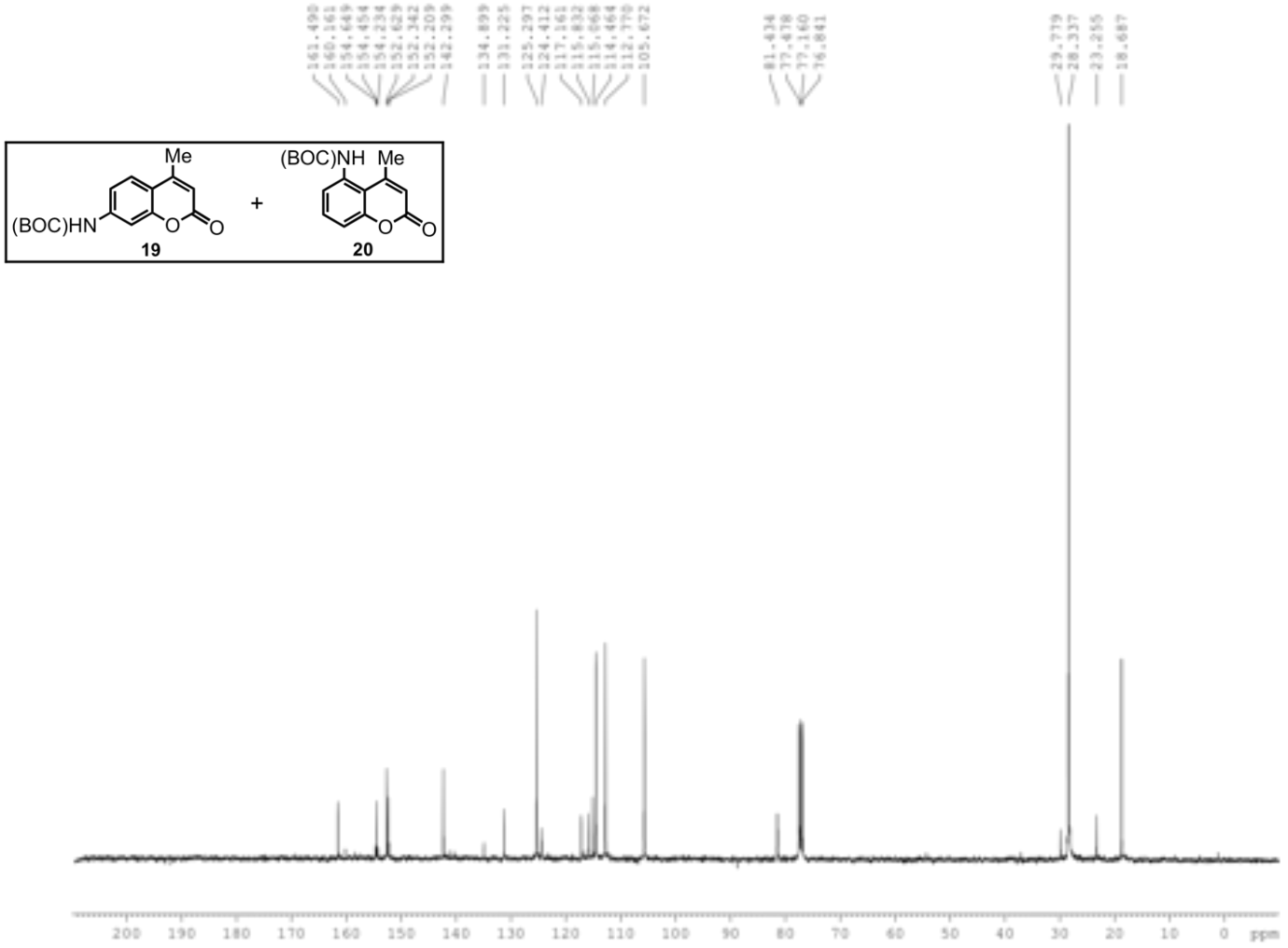

Me
(BOC)HN
O
O
18
7.354
7.274
7.266
7.245
7.225
7.113
7.092
6.791
6.769
2.037
1.500
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.910
1.026
1.007
2.007
2.359
3.022

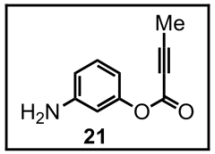

Me
(BOC)HN
O
O
18
152.545
151.969
150.477
139.768
129.679
116.126
115.687
111.669
80.264
80.017
77.479
77.161
76.843
72.045
28.306
3.965
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

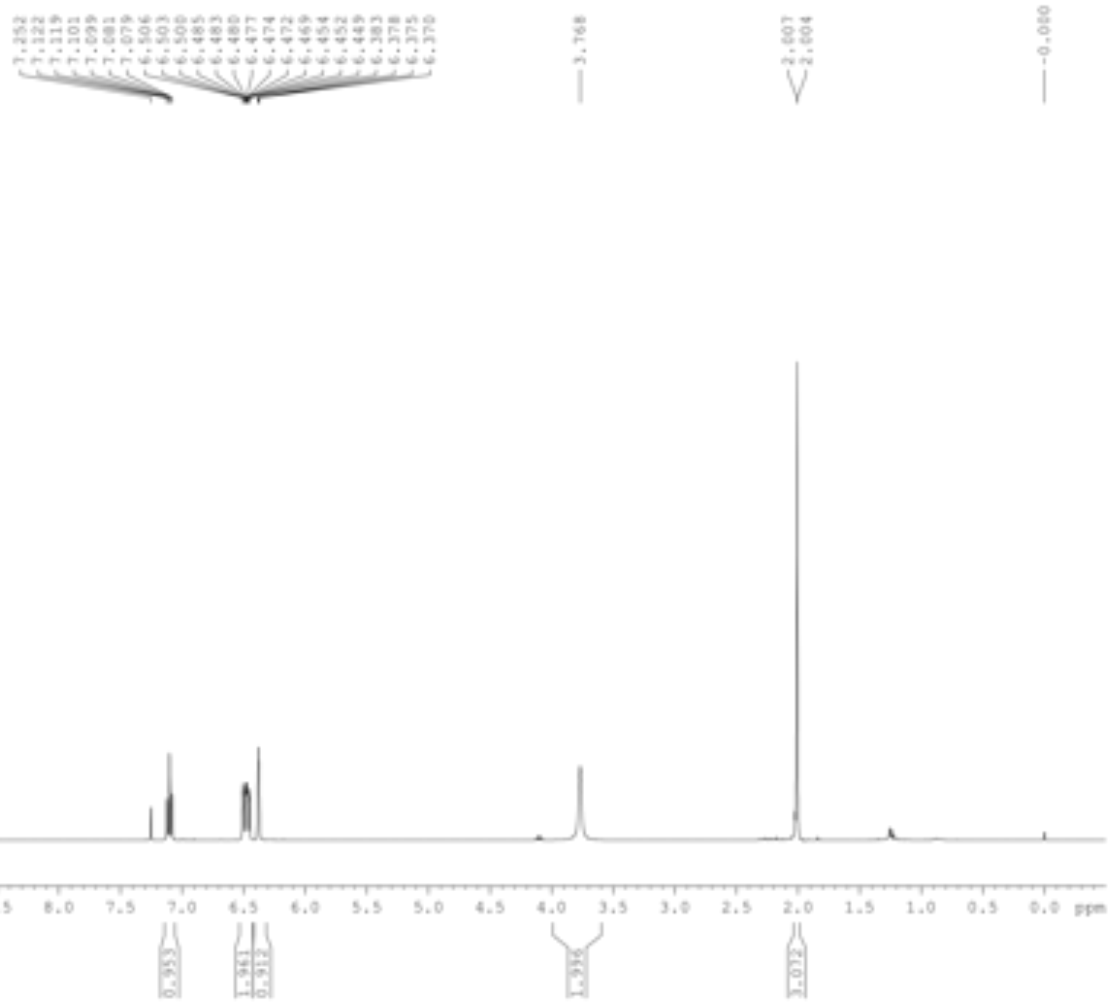

Me
(BOC)HN
O
O
19
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

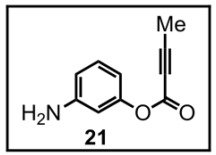

Me
(BOC)HN
O
O
19
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

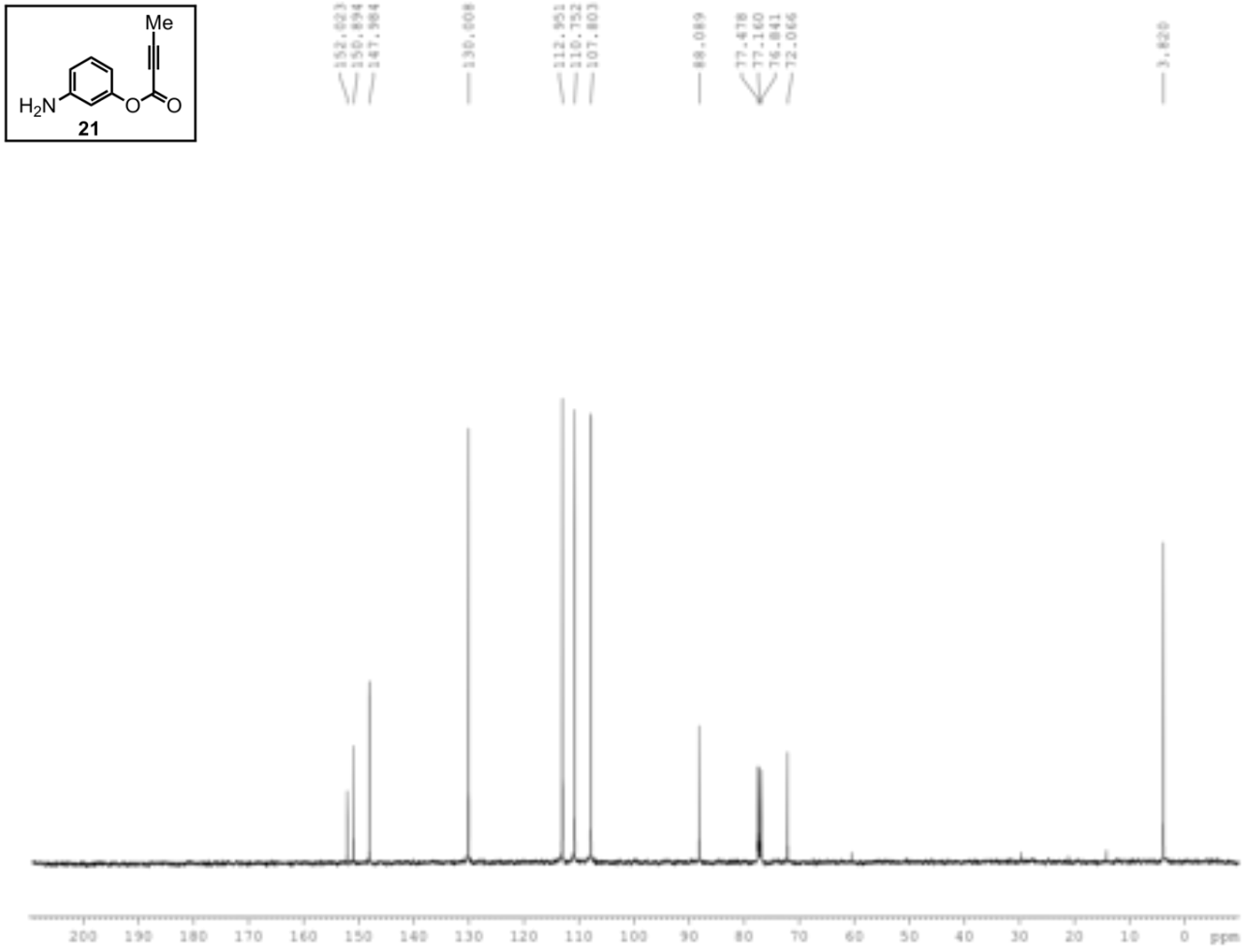

(BOC)NH Me
O
O
20
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

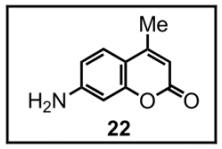

161.490
160.161
154.649
154.454
154.234
152.629
152.342
152.209
142.299
134.899
131.225
125.297
124.412
117.161
115.832
115.668
114.464
112.770
105.672
81.434
77.478
77.160
76.841
29.779
28.337
23.255
18.687
Me
(BOC)HN
O
O
19
+
(BOC)NH
Me
O
O
20
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


Me
H_2N
O
O
21

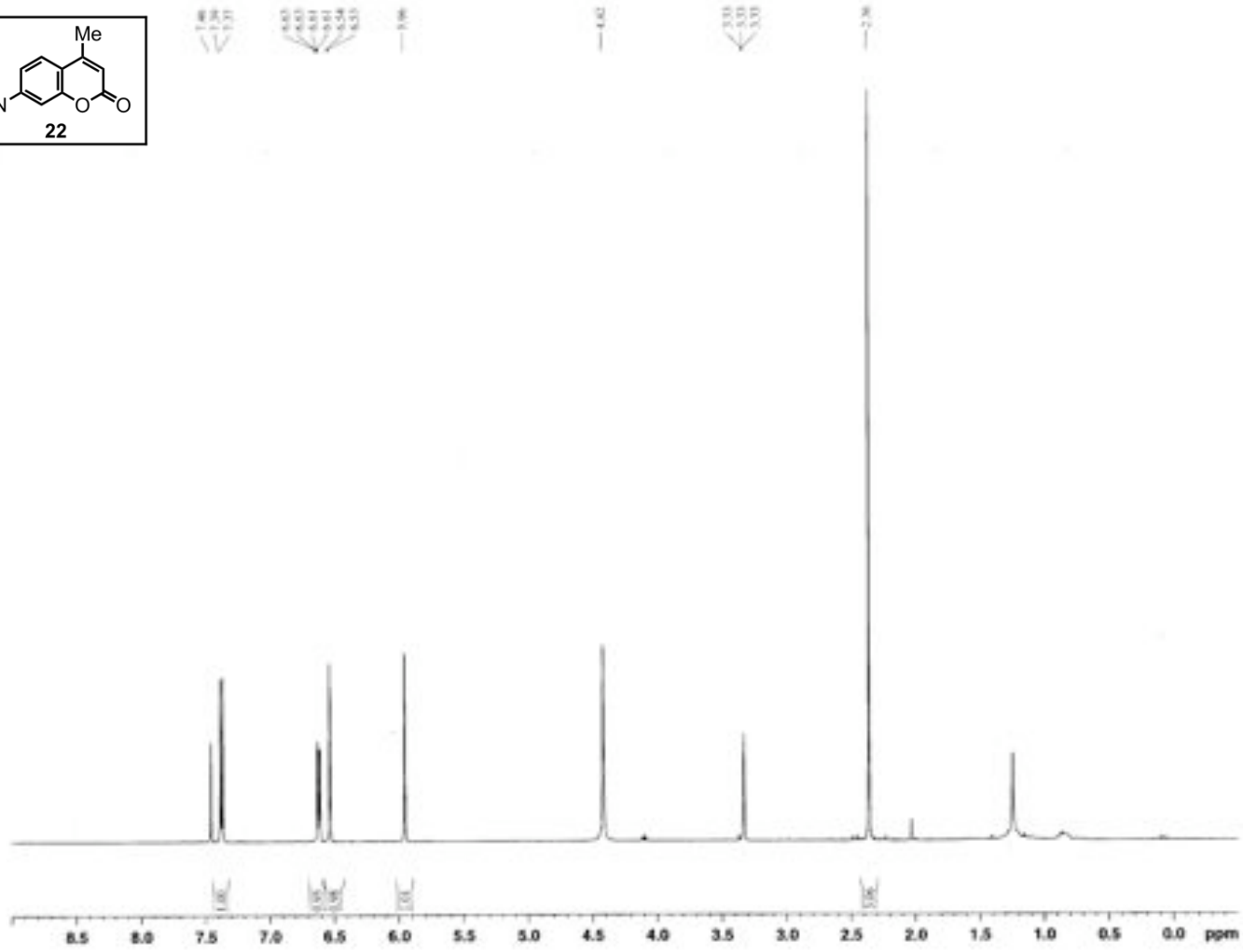

7.252
7.122
7.119
7.101
7.099
7.081
7.079
6.506
6.503
6.500
6.485
6.483
6.480
6.477
6.474
6.472
6.469
6.454
6.452
6.449
6.383
6.378
6.375
6.370
3.768
2.607
2.604
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.953
1.941
0.912
3.936
3.072


Me
H_2N
O
O
21


152.023
150.894
147.984
130.008
112.951
110.752
107.803
88.089
77.478
77.160
76.841
72.066
3.820
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

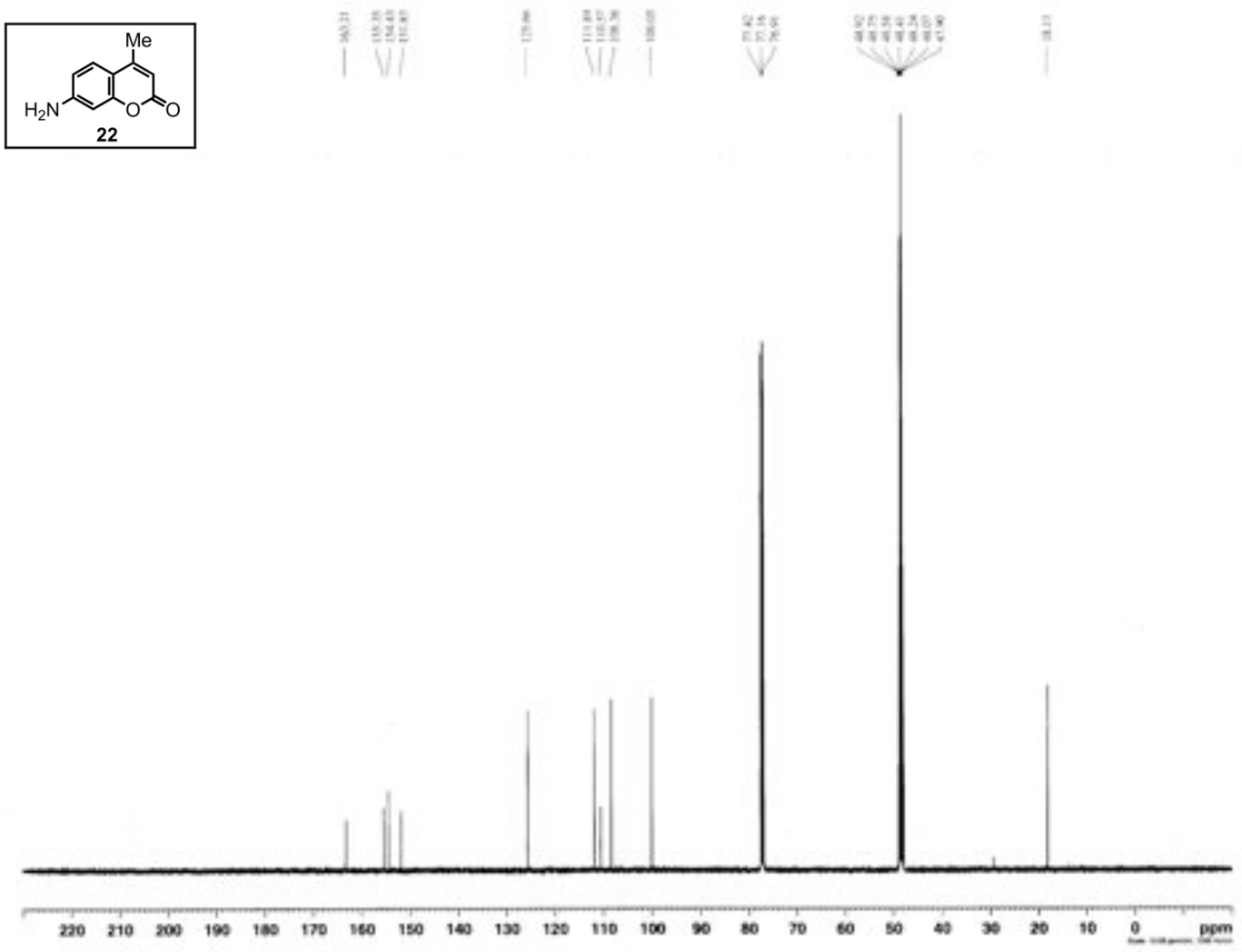


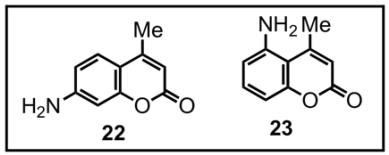
22

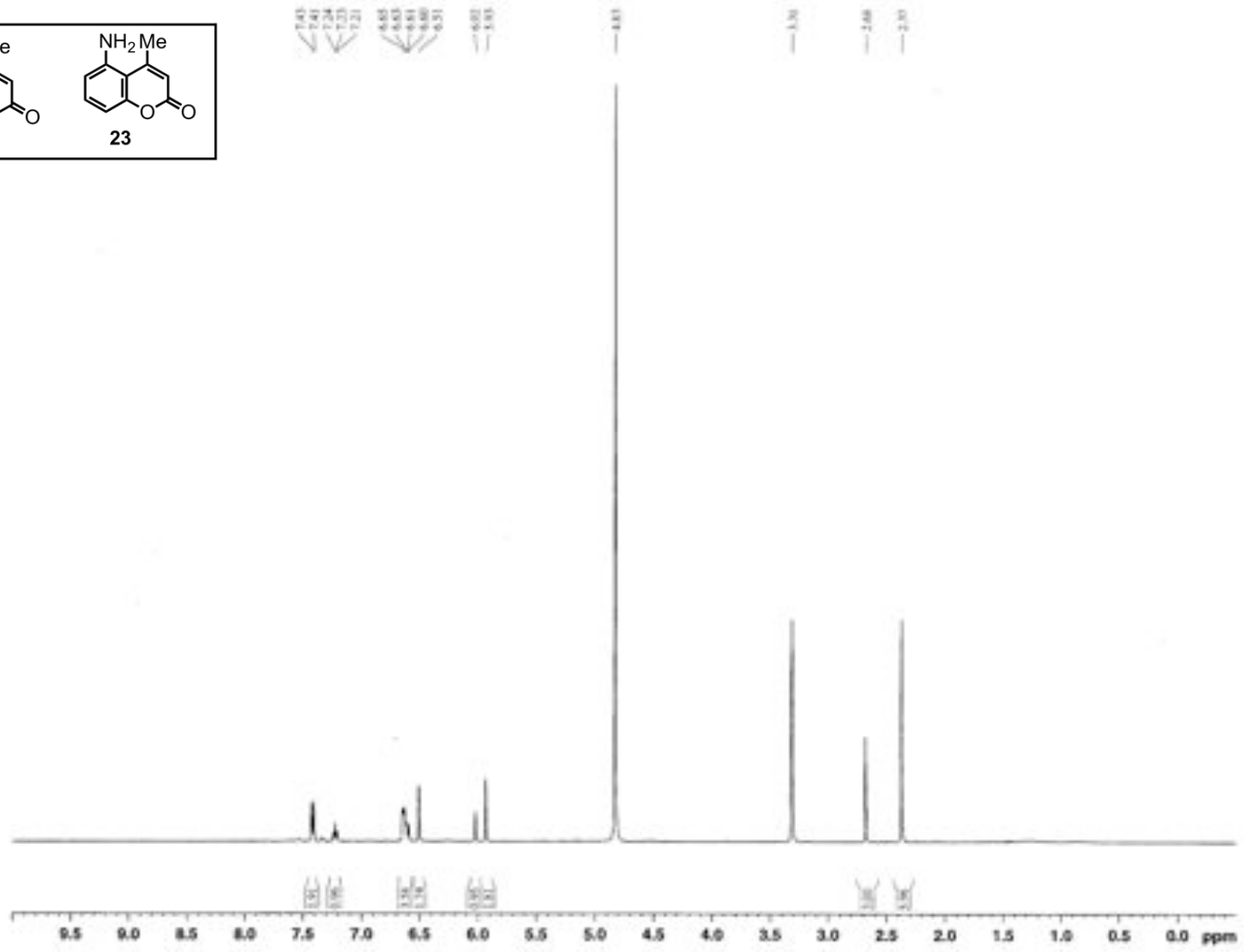


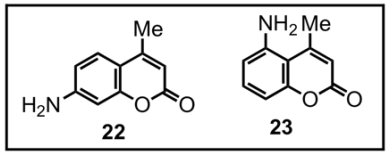
Me
H_2N
O
O

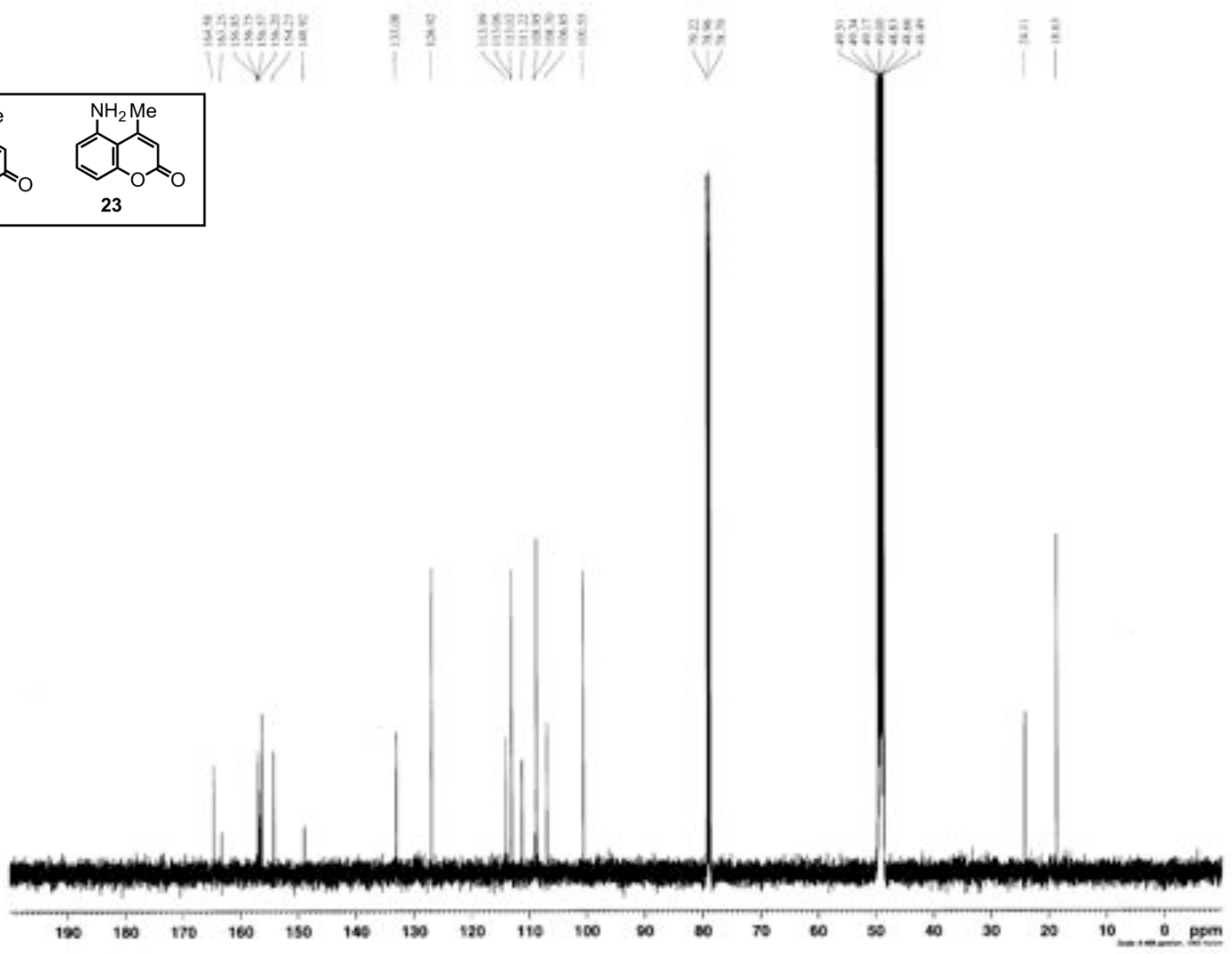
22

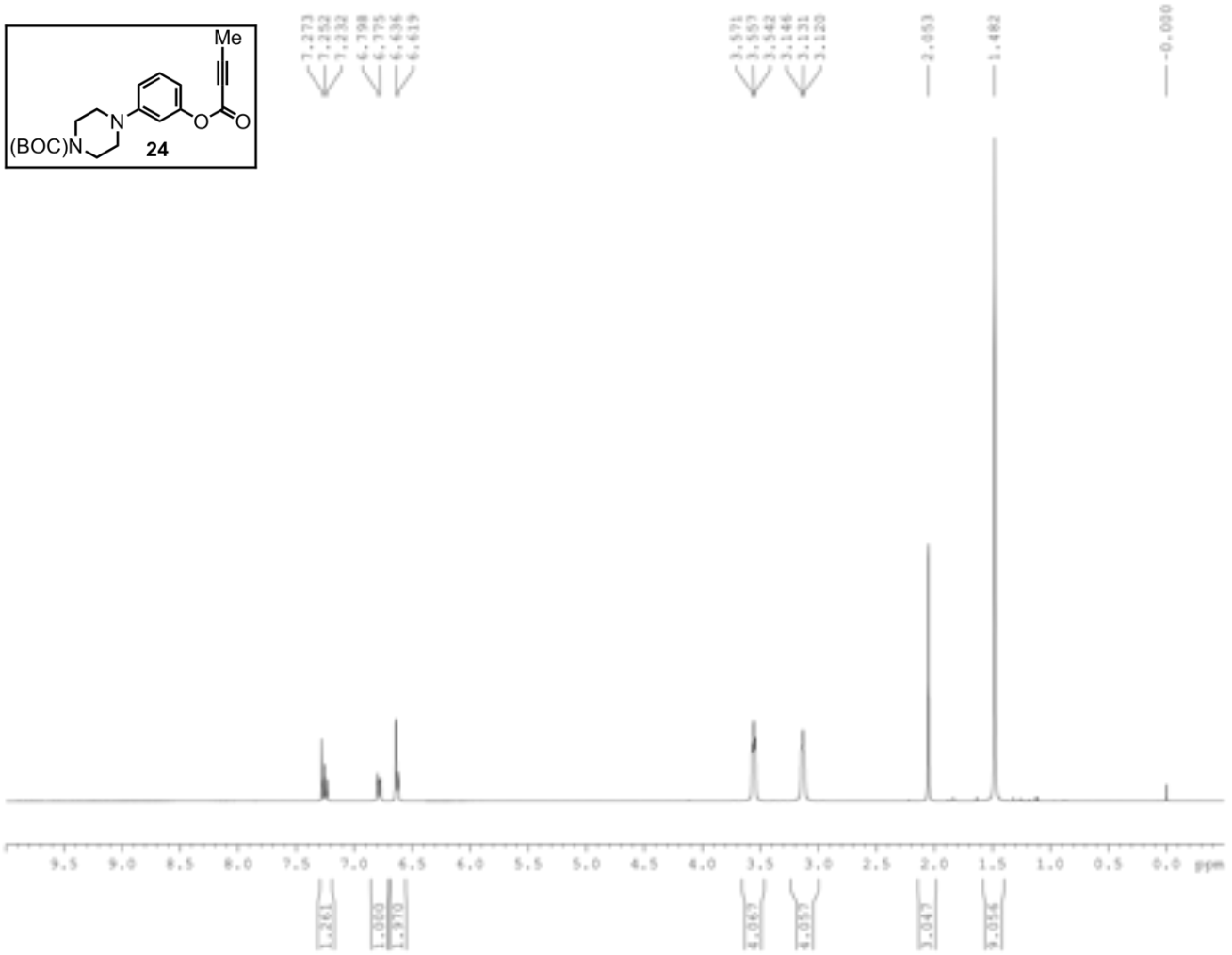

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

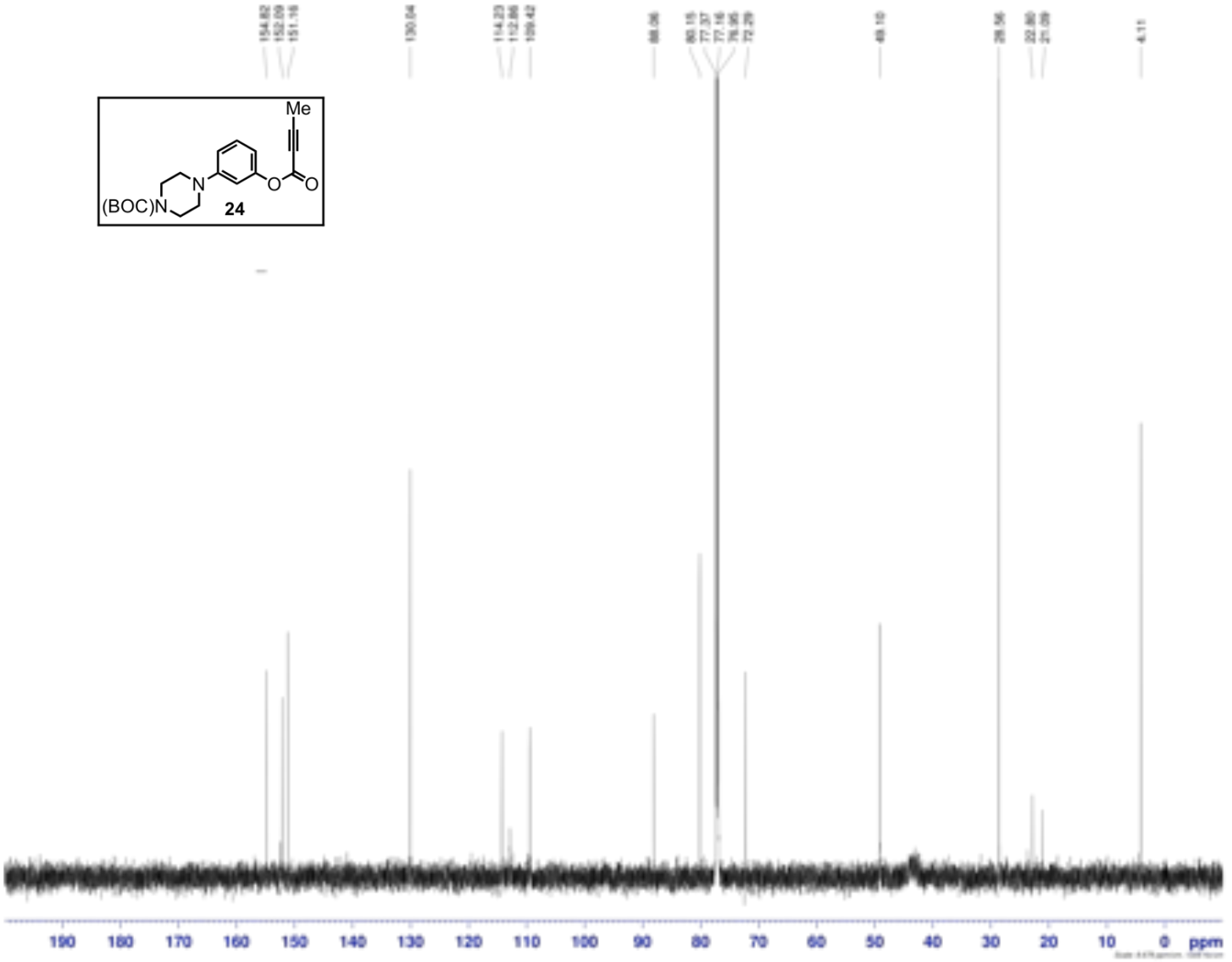

Me
H2N
O
O
22
NH2 Me
O
O
23

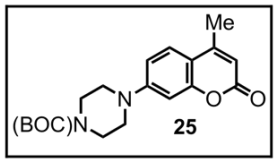

9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

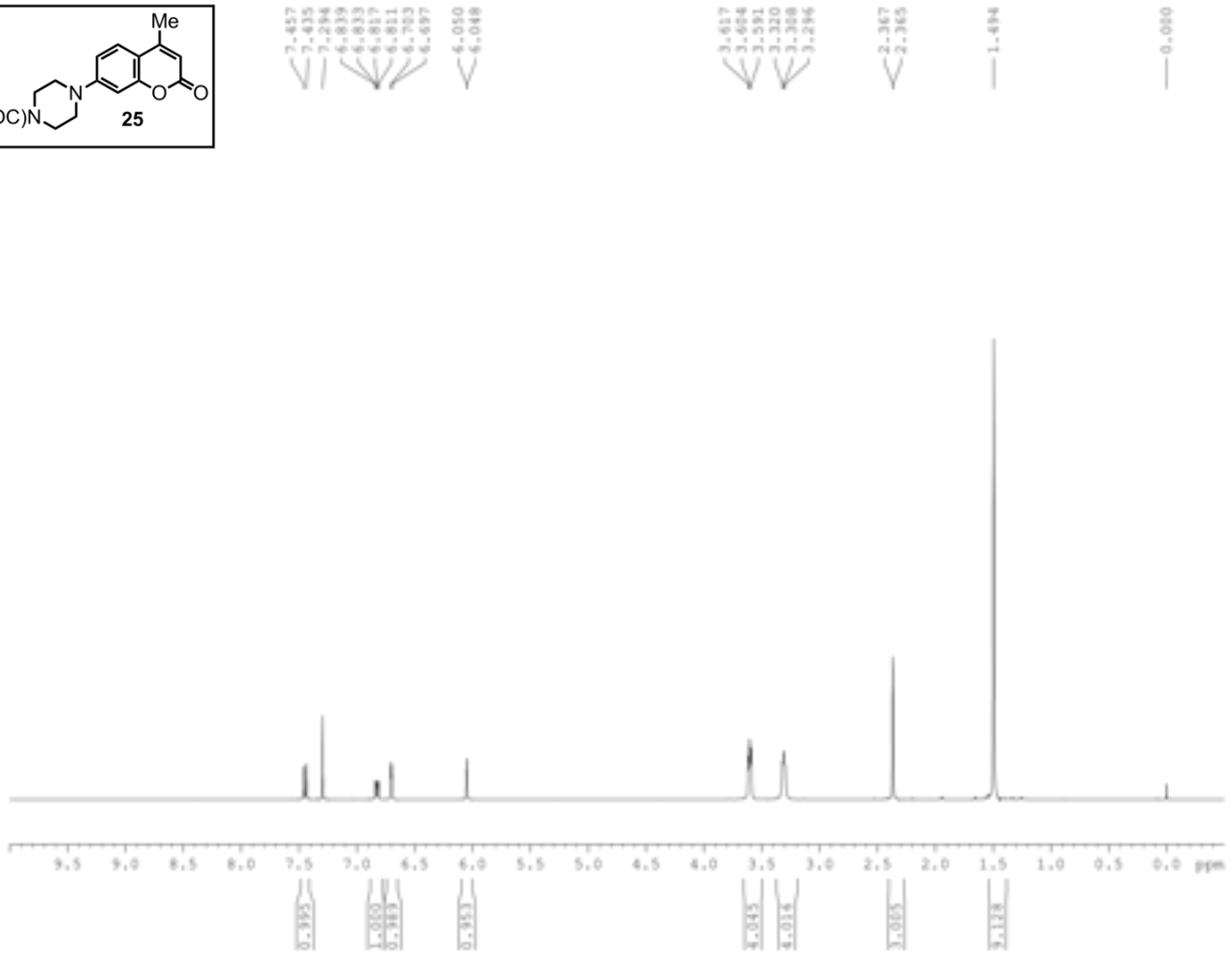

Me
H_2N
O
O
22
NH_2 Me
O
O
23

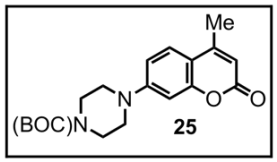

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

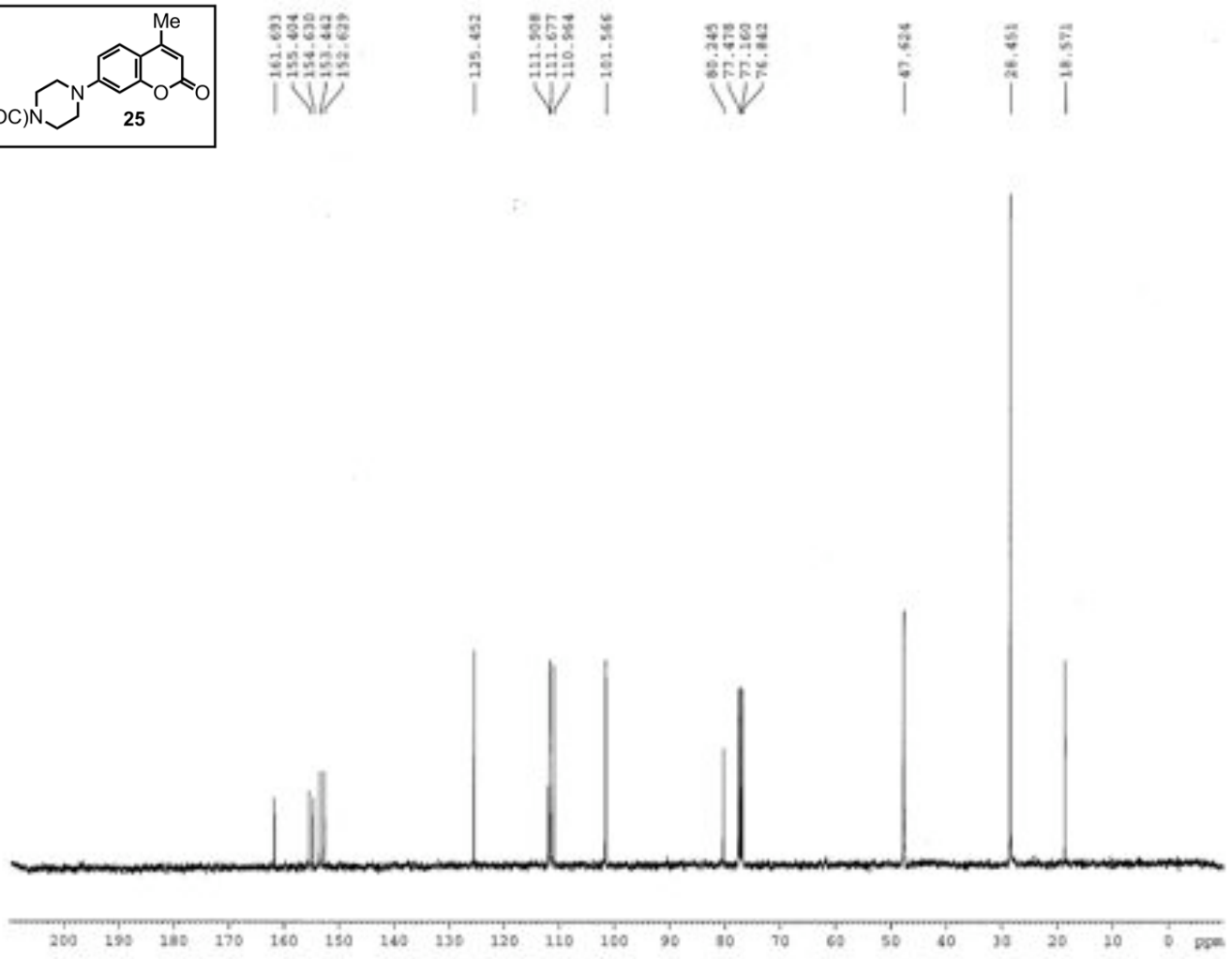

Me
N
O
O
(BOC)N
24
7.273
7.252
7.232
6.798
6.775
6.636
6.619
3.571
3.557
3.542
3.146
3.131
3.120
2.053
1.462
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.261
1.000
1.570
4.067
4.057
3.047
9.056

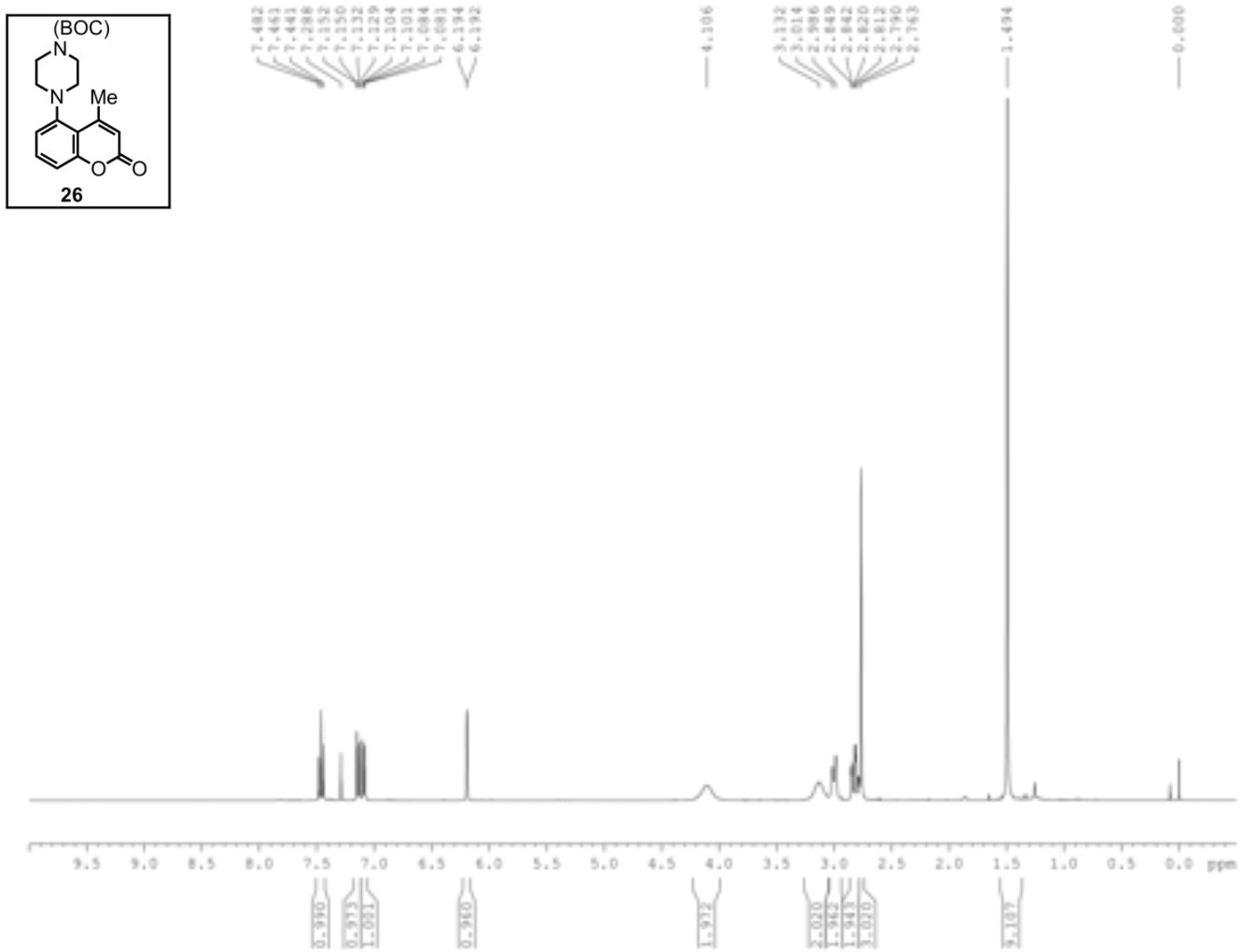

Me
(BOC)N
24
154.82
152.09
151.16
130.04
114.23
112.86
109.42
88.06
80.15
77.37
77.16
76.95
72.29
49.10
28.56
22.80
21.09
4.11
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

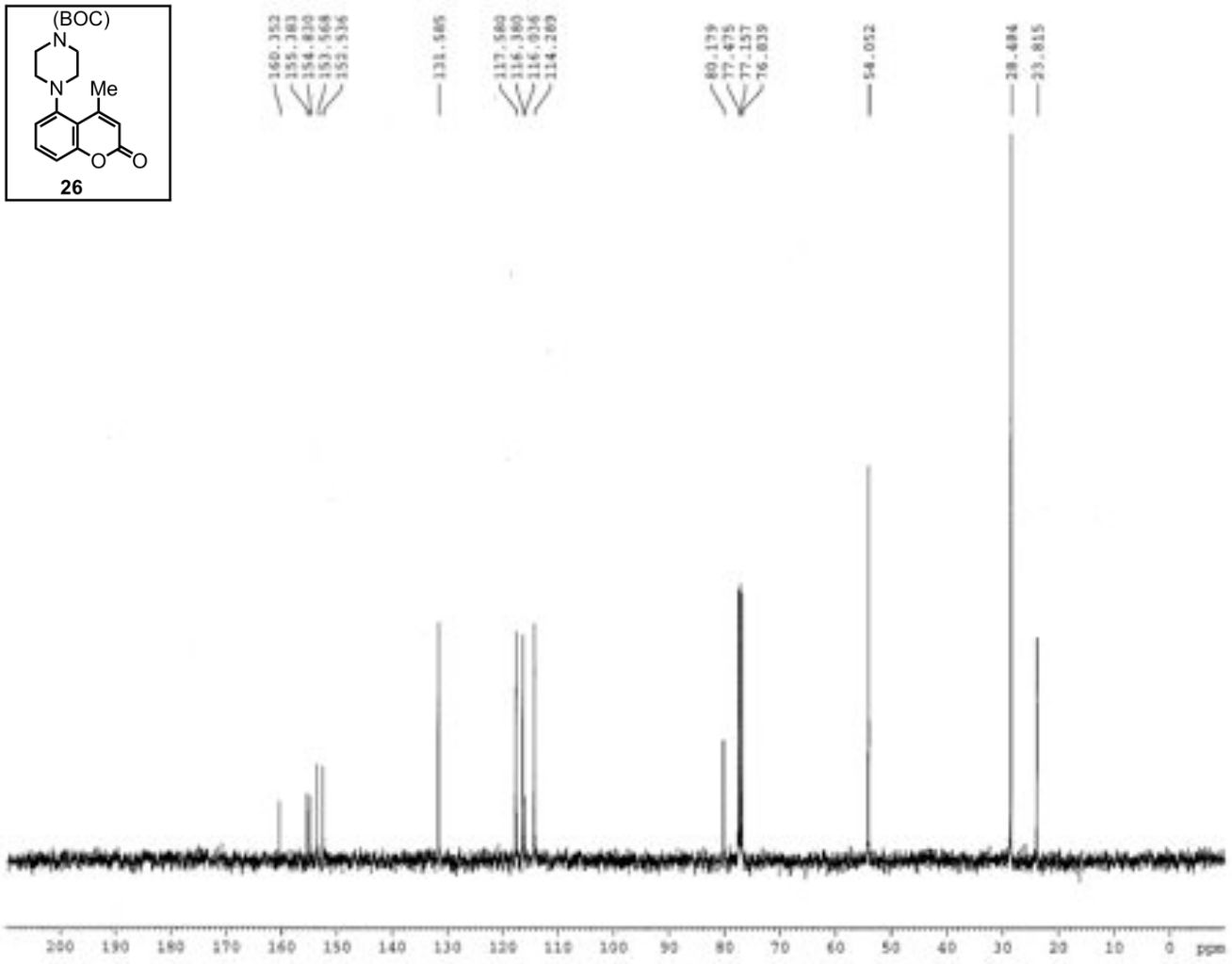

Me
N
O
O
(BOC)N
25

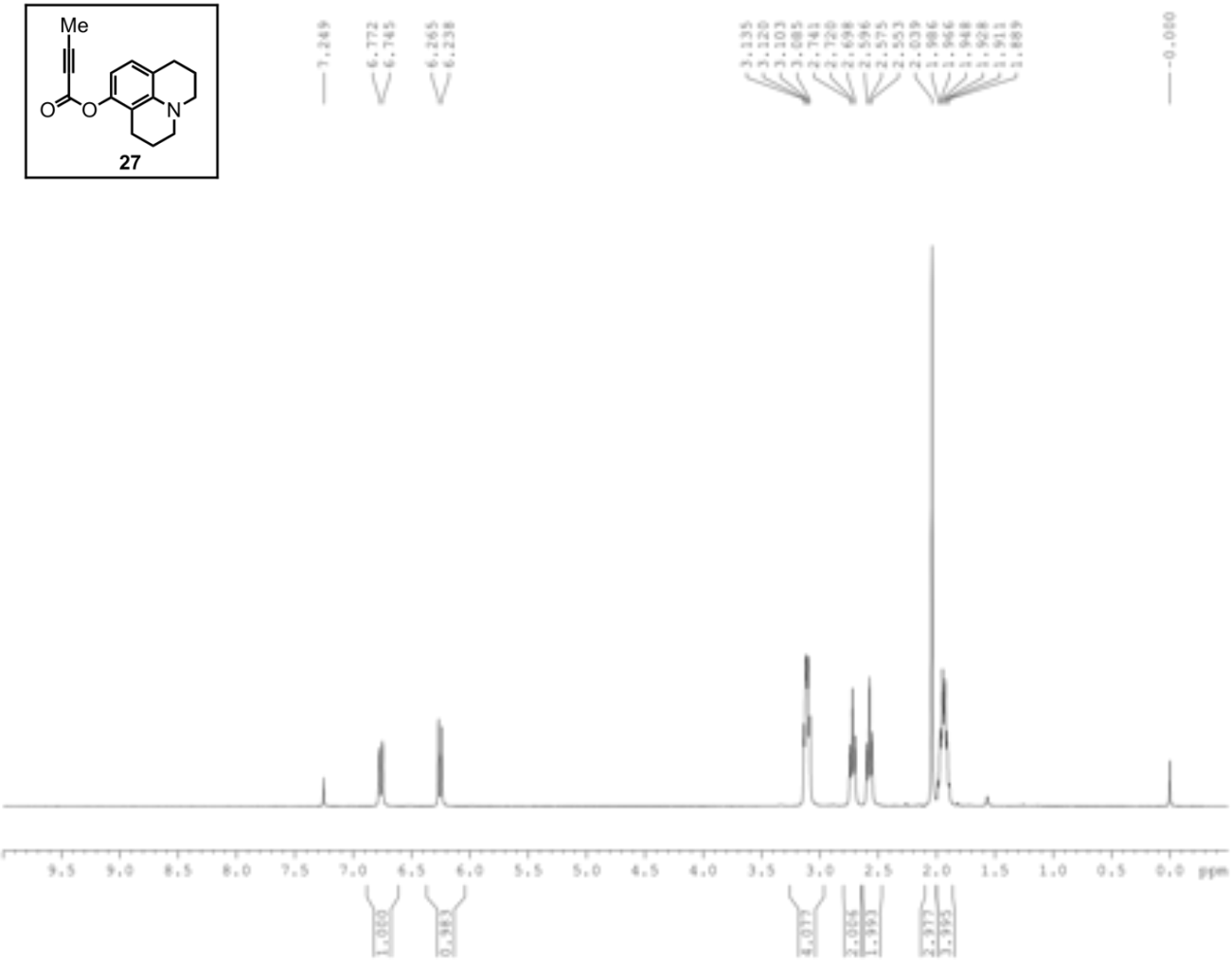

7.457
7.435
7.294
6.839
6.833
6.817
6.811
6.703
6.697
6.050
6.048
3.617
3.604
3.591
3.320
3.308
3.296
2.367
2.365
1.494
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.395
1.000
0.389
0.953
4.045
4.014
3.005
9.178

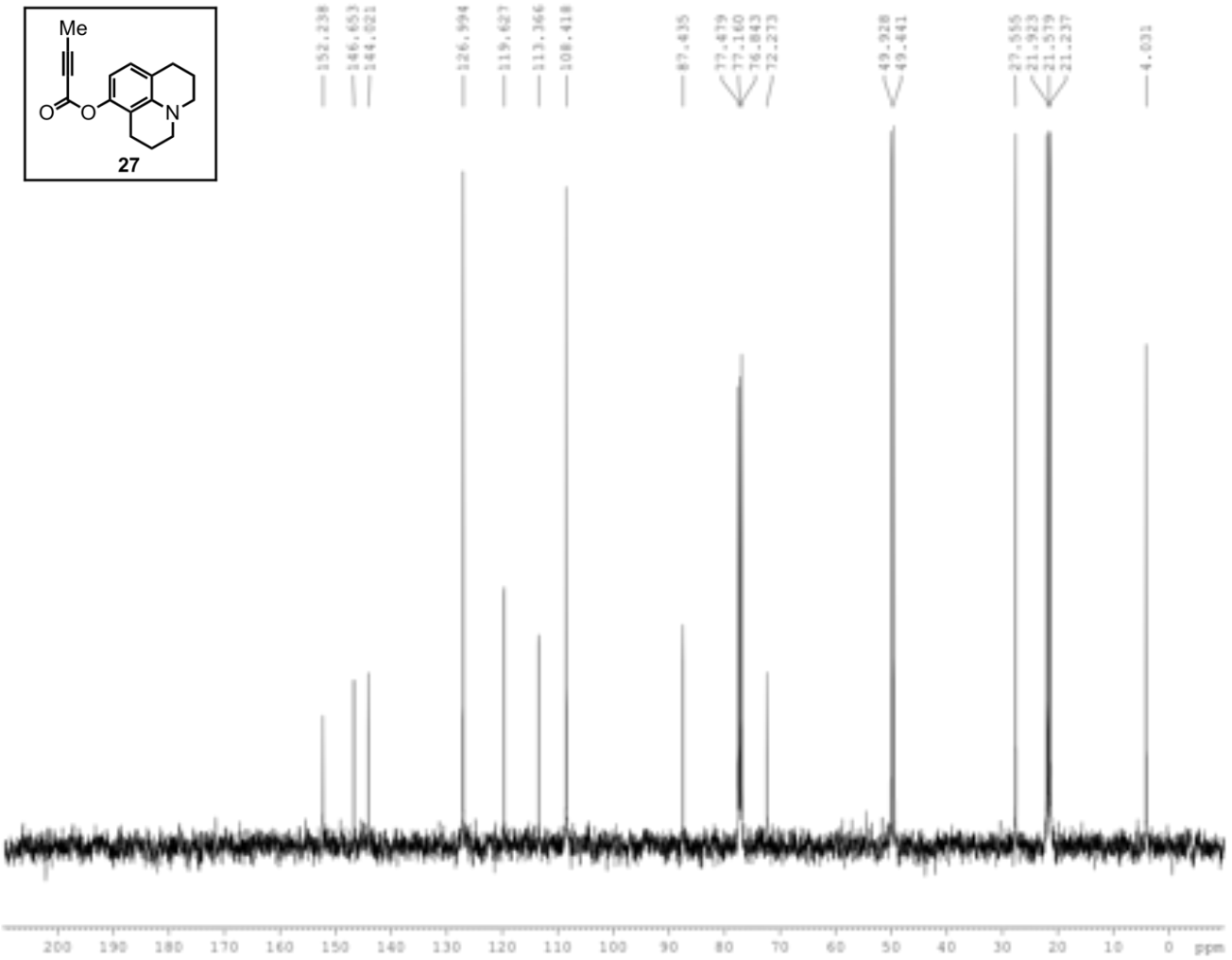

Me
N
O
O
(BOC)N
25


161.693
155.404
154.630
153.442
152.629
125.452
111.908
111.677
110.964
101.566
80.245
77.478
77.160
76.842
47.624
28.451
18.571
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

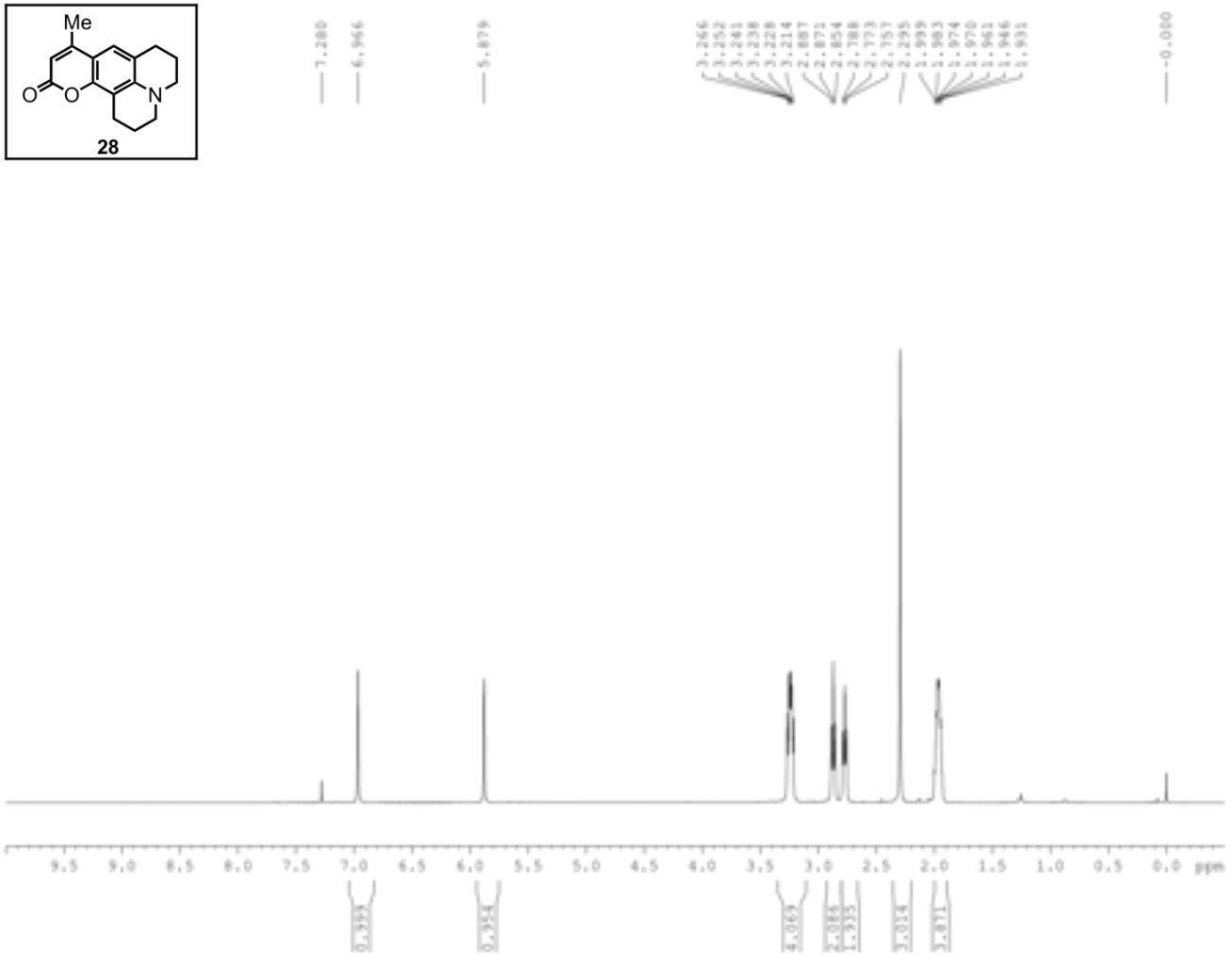

(BOC)
N
N
Me
O
O
26
7.482
7.461
7.441
7.288
7.252
7.150
7.132
7.129
7.104
7.101
7.084
7.081
6.194
6.192
4.106
3.132
3.014
2.986
2.849
2.842
2.820
2.812
2.790
2.763
1.494
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.990
0.973
1.001
0.960
1.972
2.020
1.942
1.943
3.020
9.107

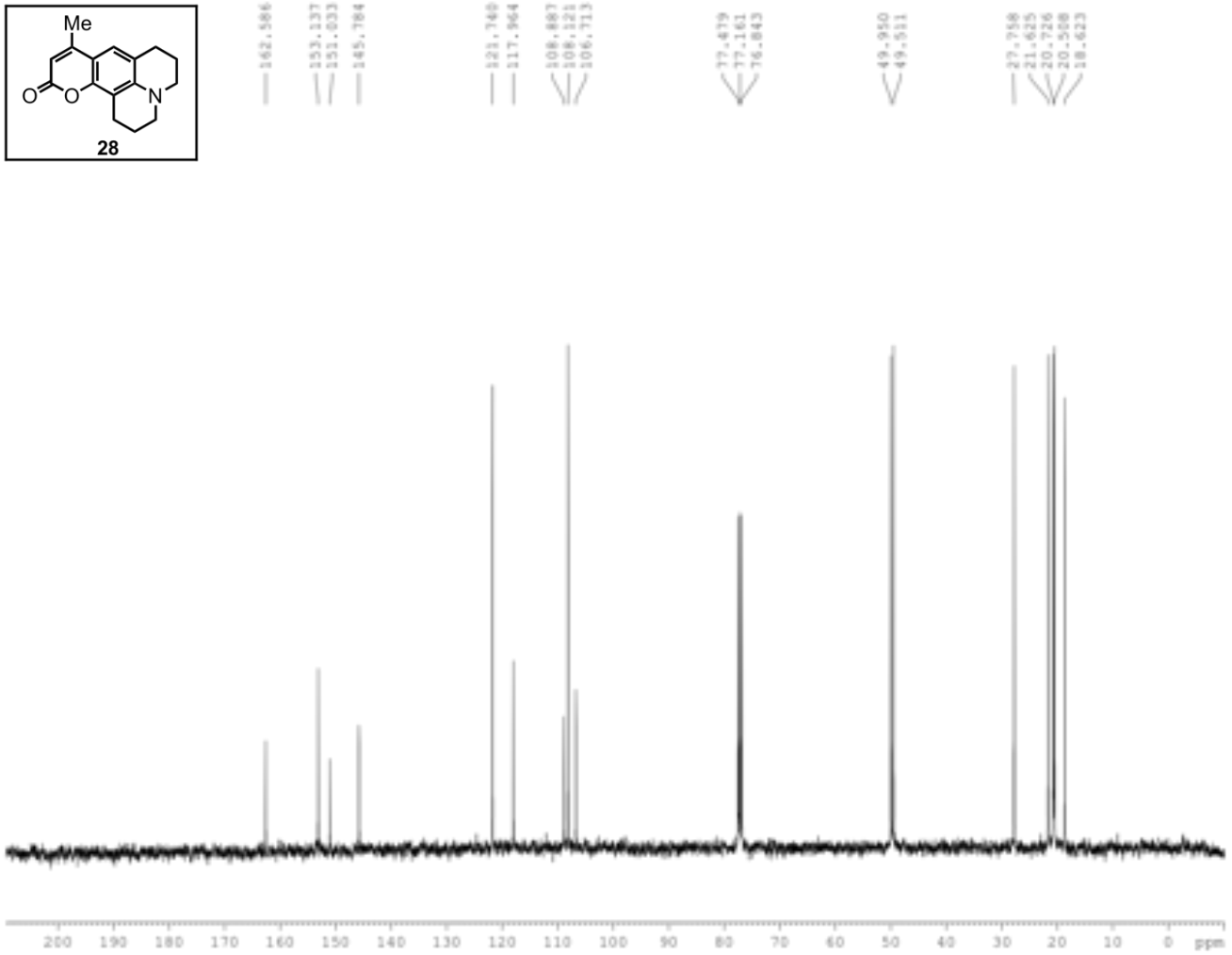

(BOC)
N
N
Me
O
O
26
160.352
155.383
154.830
153.564
152.536
131.585
117.580
116.380
116.034
114.269
80.179
77.475
77.157
76.839
54.052
28.484
23.815
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

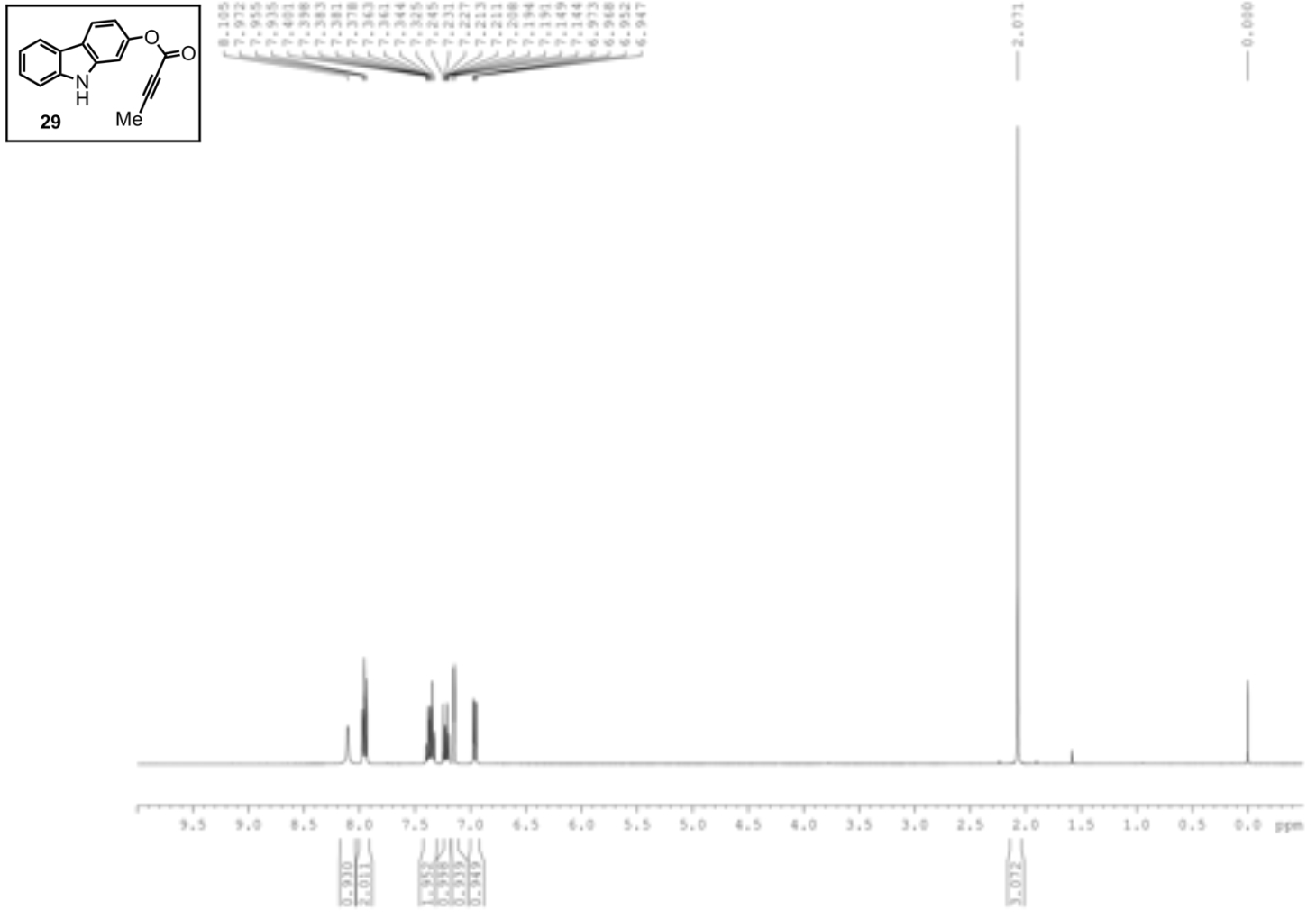

Me
O
O
N
27
7.249
6.772
6.745
6.265
6.238
3.135
3.120
3.103
3.085
2.741
2.720
2.698
2.596
2.575
2.553
2.039
1.986
1.966
1.948
1.928
1.911
1.889
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
0.383
4.017
2.006
1.993
2.977
2.995

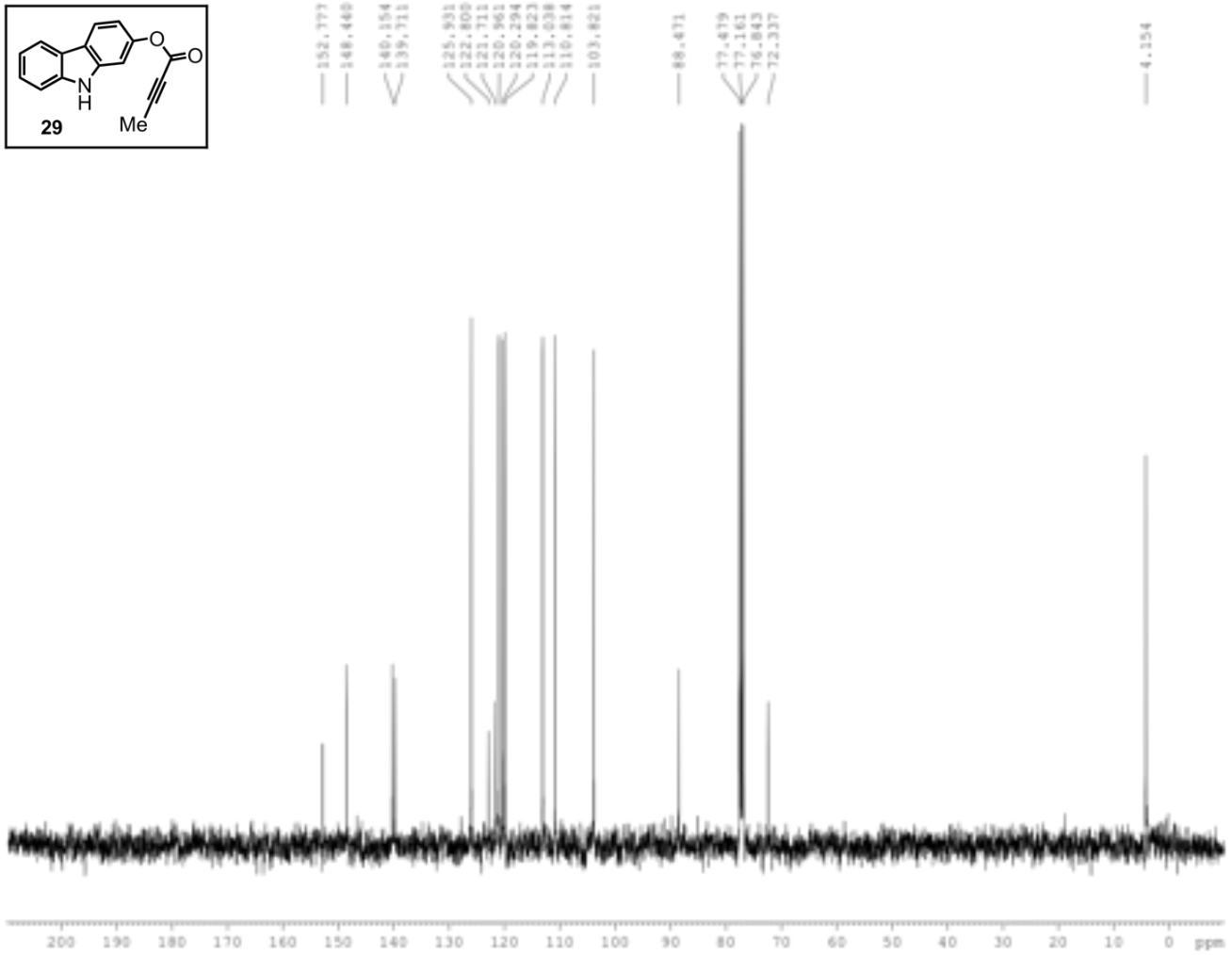

Me
O
O
N
27
152.238
146.653
144.021
126.994
119.627
113.366
108.418
87.435
77.479
77.160
76.843
72.273
49.928
49.441
27.555
21.923
21.579
21.237
4.031
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

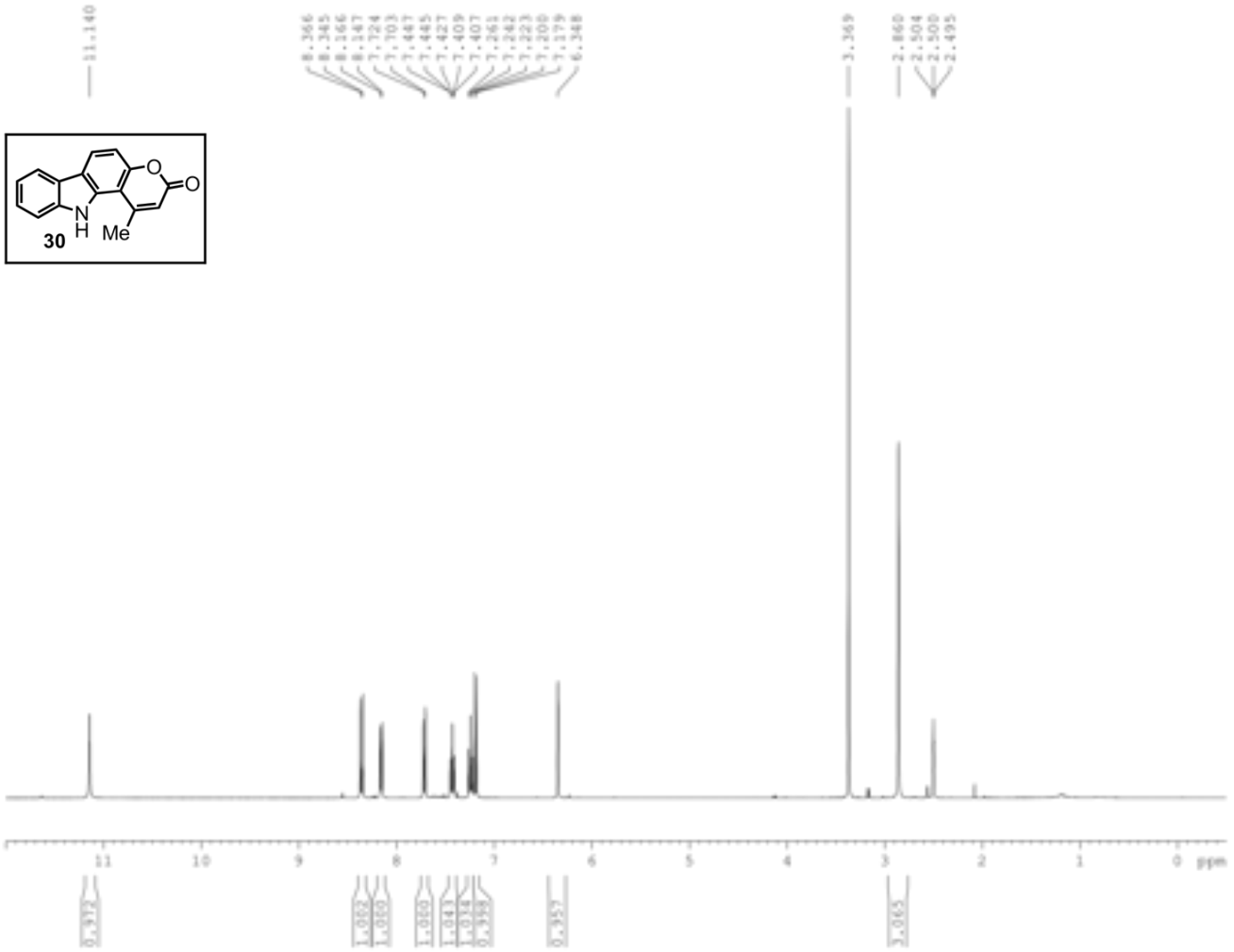
Me
O
O
N
28

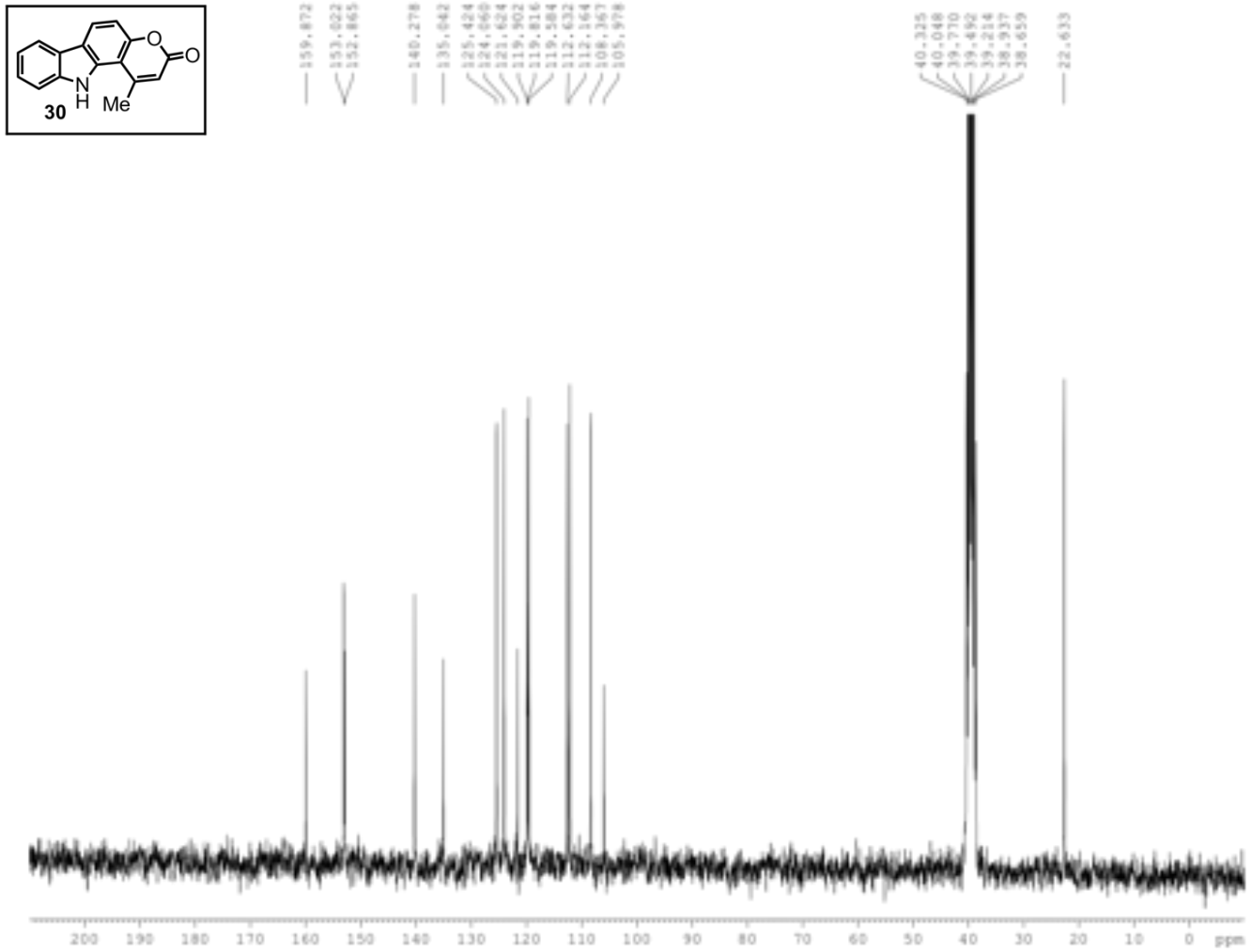

7.280
6.966
5.879
3.266
3.252
3.241
3.238
3.228
3.214
2.887
2.871
2.854
2.788
2.773
2.757
2.295
1.999
1.983
1.974
1.970
1.961
1.946
1.931
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.999
0.954
4.069
2.046
1.935
3.014
3.071

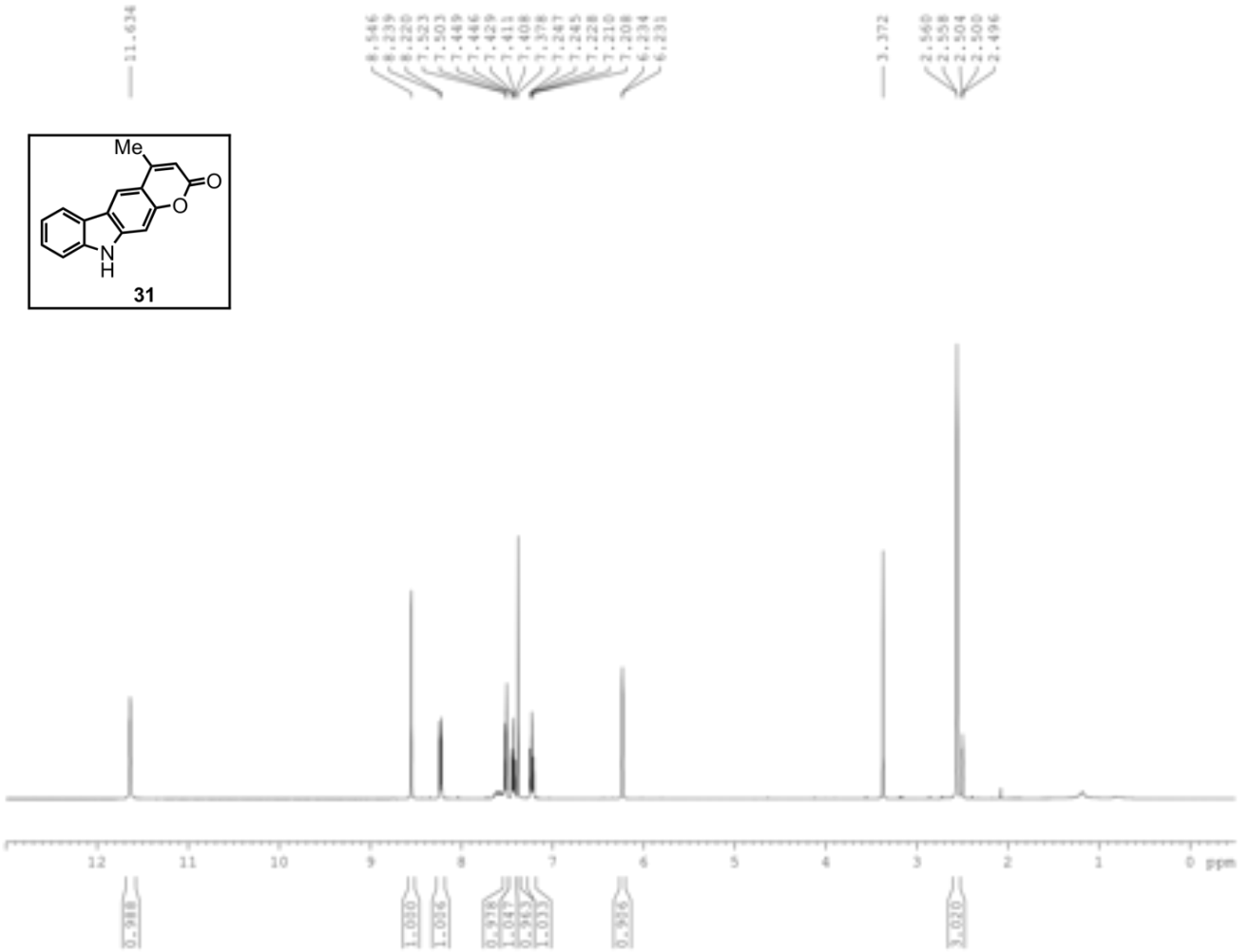

Me
O
O
N
28
162.586
153.137
151.033
145.784
121.740
117.964
108.887
108.121
106.713
77.479
77.161
76.843
49.950
49.511
27.758
21.625
20.726
20.508
18.623
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

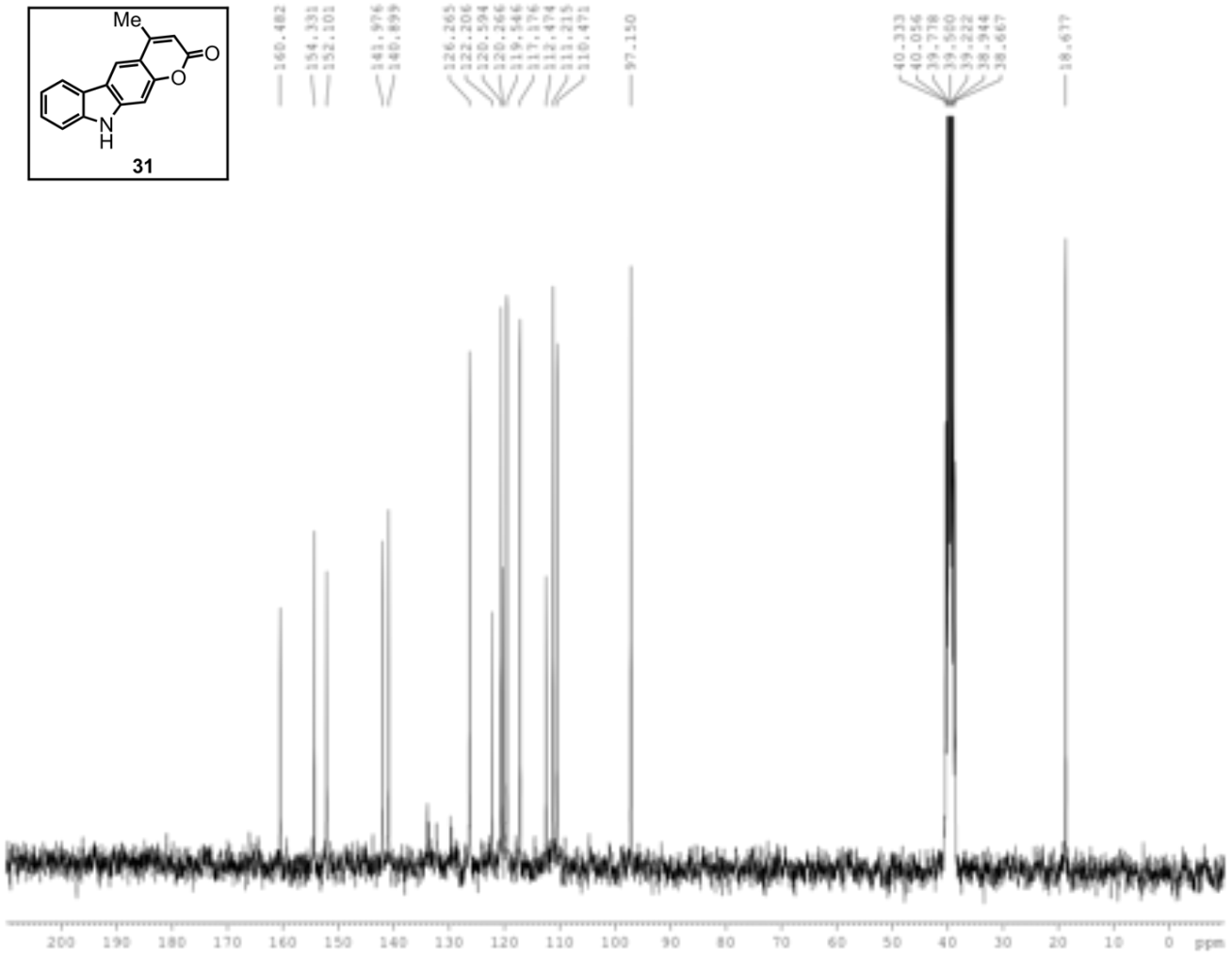

29
Me
8.105
7.972
7.955
7.935
7.401
7.398
7.383
7.381
7.378
7.363
7.361
7.344
7.325
7.245
7.231
7.227
7.213
7.211
7.208
7.194
7.191
7.149
7.144
6.973
6.968
6.952
6.947
2.071
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.930
2.011
1.952
1.938
0.933
0.949
3.072

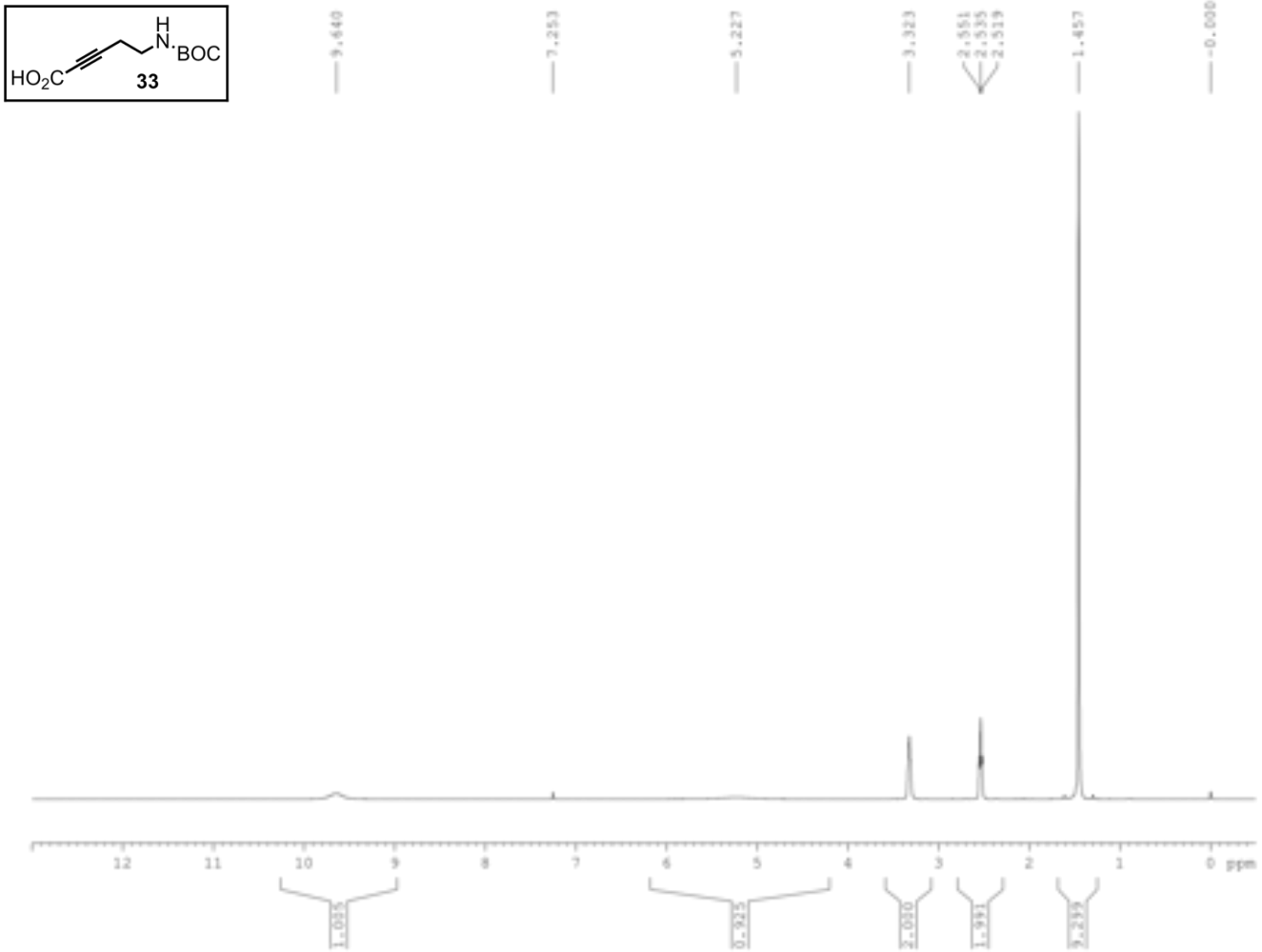

29
Me
152.777
148.440
140.154
139.711
125.931
122.800
121.711
120.961
120.294
119.823
113.038
110.814
103.821
88.471
77.479
77.161
76.843
72.337
4.154
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

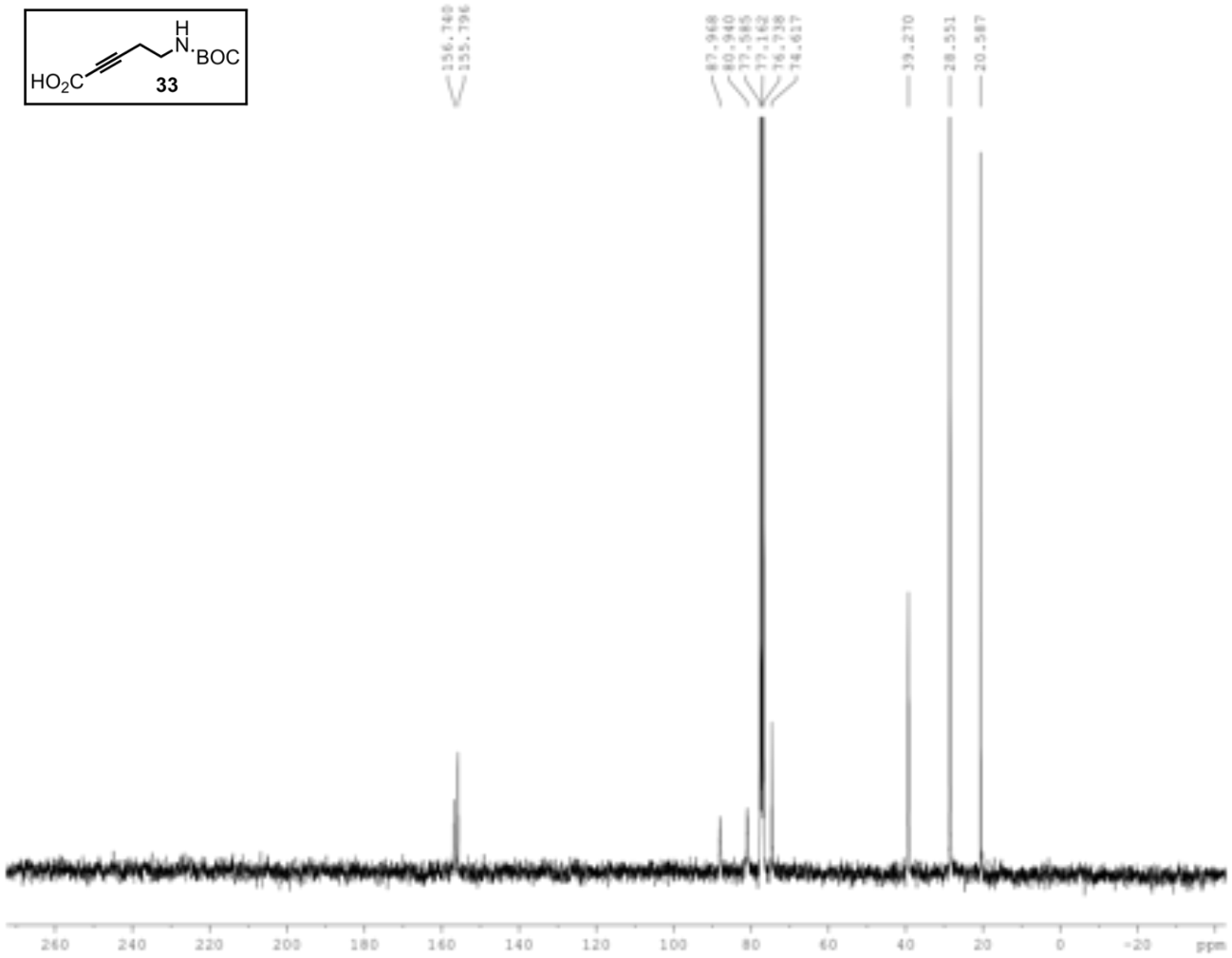

11.140
8.366
8.345
8.166
8.147
7.724
7.703
7.447
7.445
7.427
7.409
7.407
7.261
7.242
7.223
7.200
7.179
6.348
3.369
2.860
2.504
2.500
2.495
O
O
N
H
Me
30
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.332
1.000
1.000
1.080
1.041
1.014
0.998
0.957
3.065

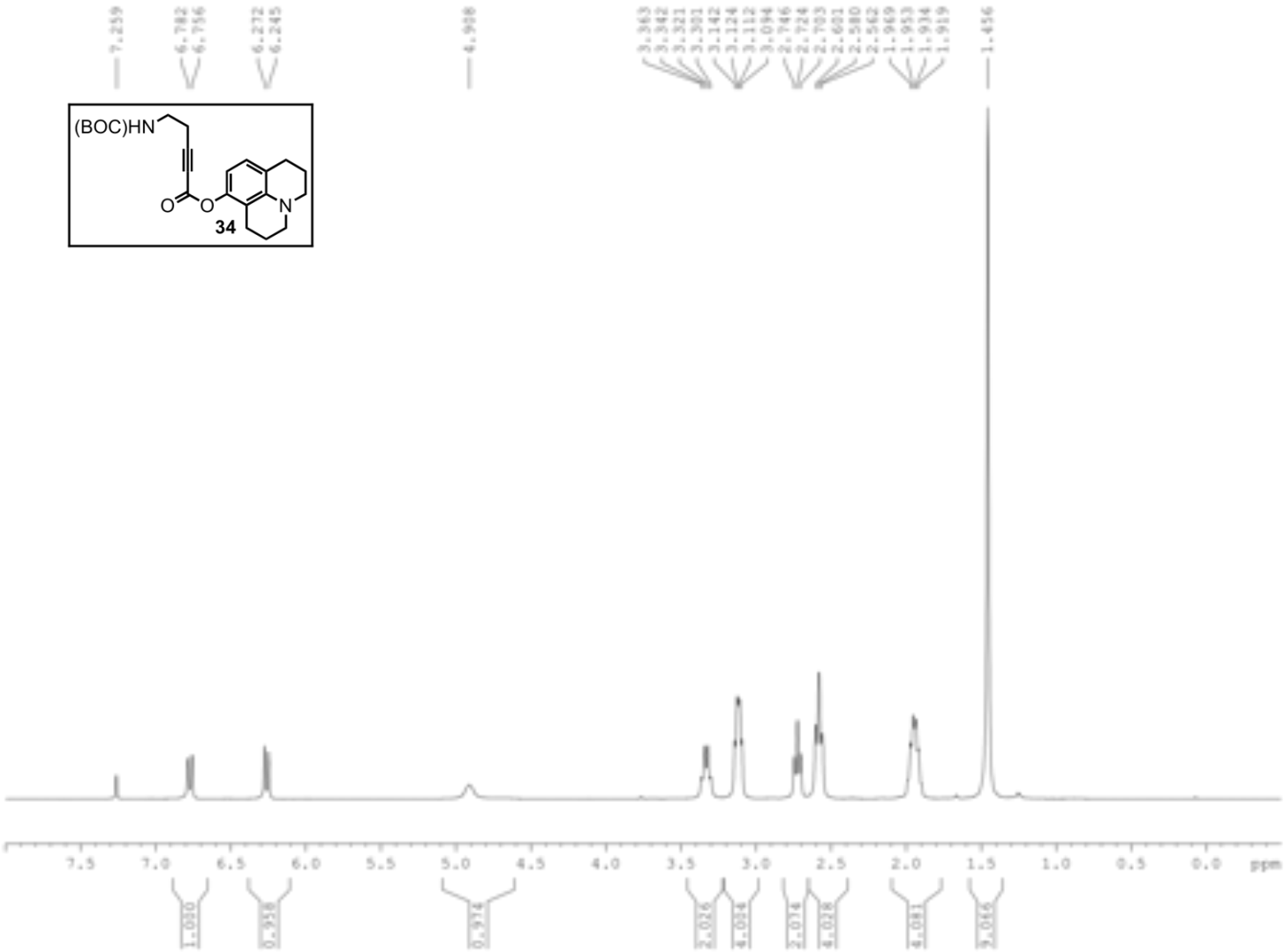

30
N
H
Me
O
O
159.872
153.022
152.865
140.278
135.042
125.424
124.660
121.624
119.902
119.816
119.584
112.632
112.164
108.367
105.978
40.325
40.048
39.770
39.492
39.214
38.937
38.659
22.633
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

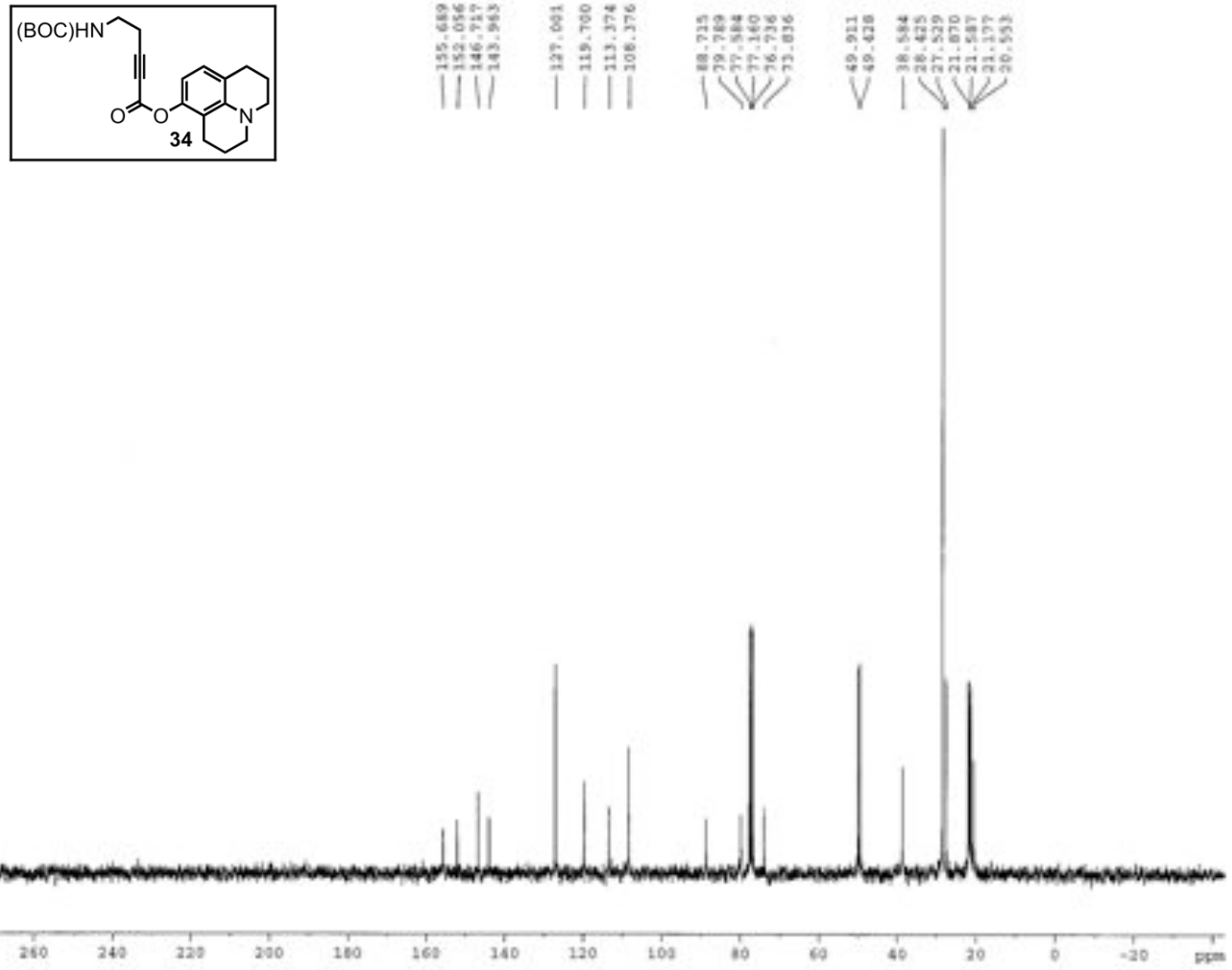

11.634
8.546
8.239
8.220
7.523
7.503
7.449
7.446
7.429
7.411
7.408
7.378
7.247
7.245
7.228
7.210
7.208
6.234
6.231
3.372
2.560
2.558
2.504
2.500
2.496
Me
O
O
N
H
31
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.988
1.000
1.026
0.978
1.047
0.963
1.013
0.936
3.010

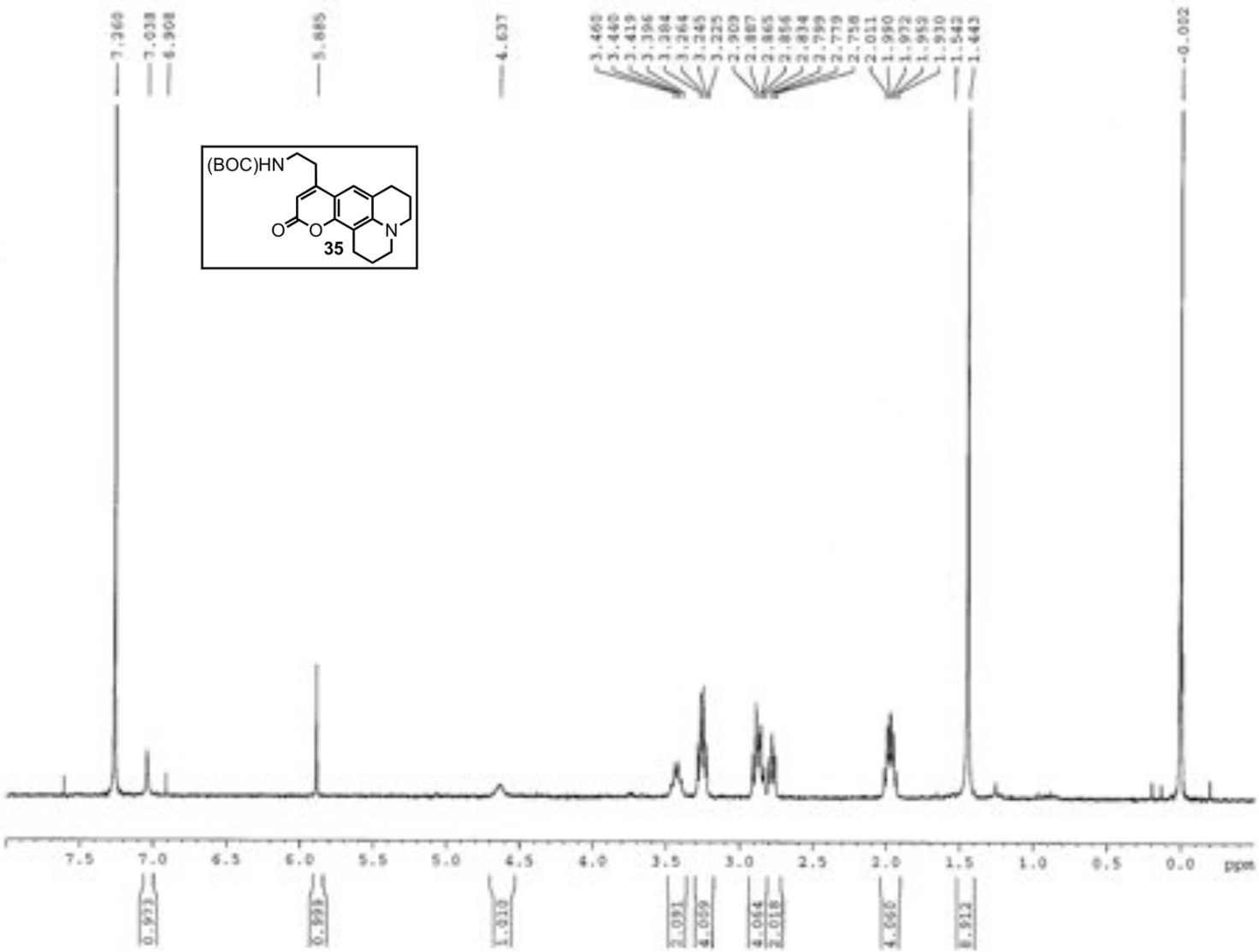

Me
O
O
N
H
31
160.482
154.331
152.101
141.976
140.899
126.265
122.206
120.594
120.266
119.546
117.176
112.474
111.215
110.471
97.150
40.333
40.056
39.778
39.500
39.222
38.944
38.667
18.677
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

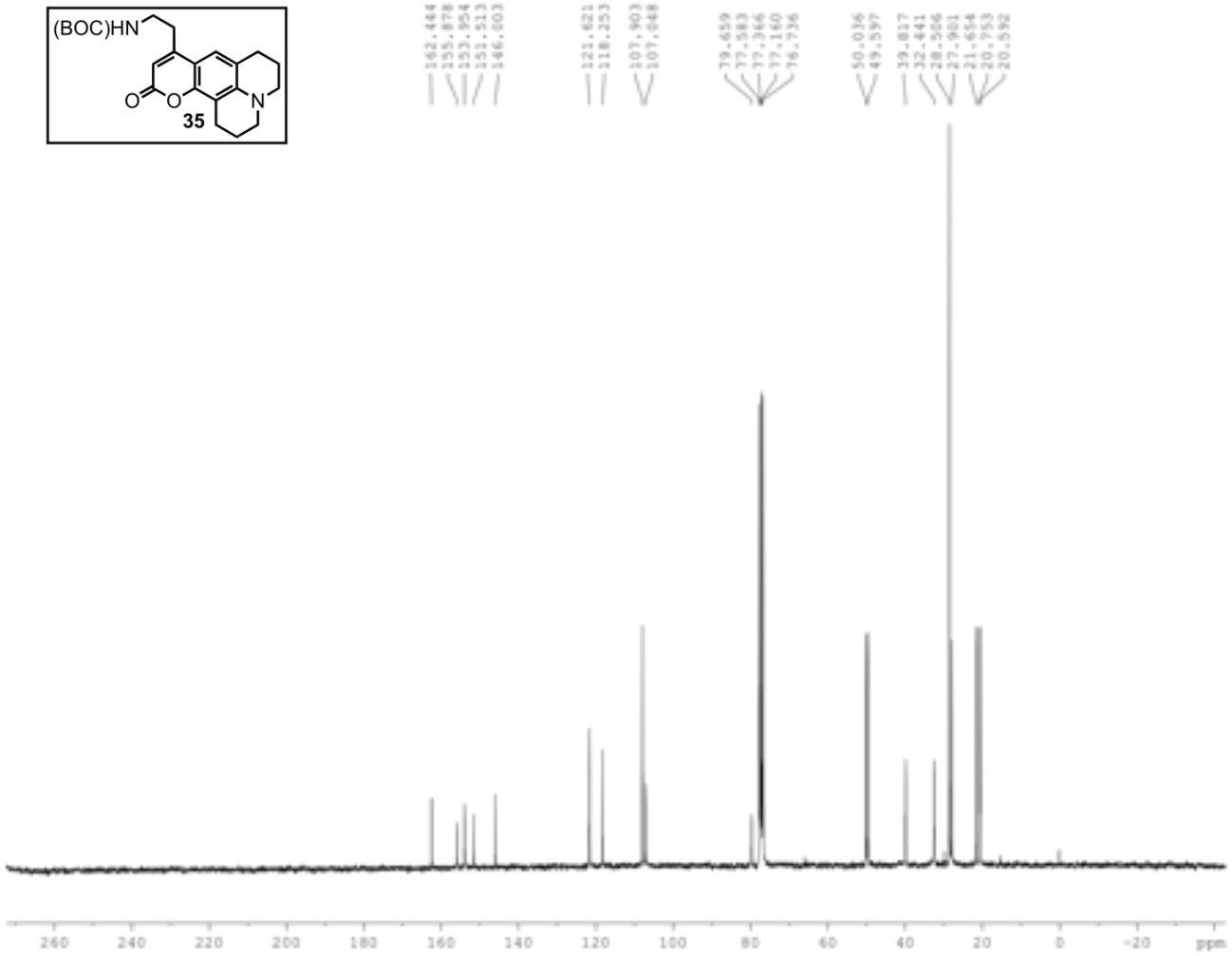

HO2C
H
N
BOC
33
9.640
7.253
5.227
3.323
2.551
2.535
2.519
1.457
0.000
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
1.085
0.925
2.000
1.991
9.439

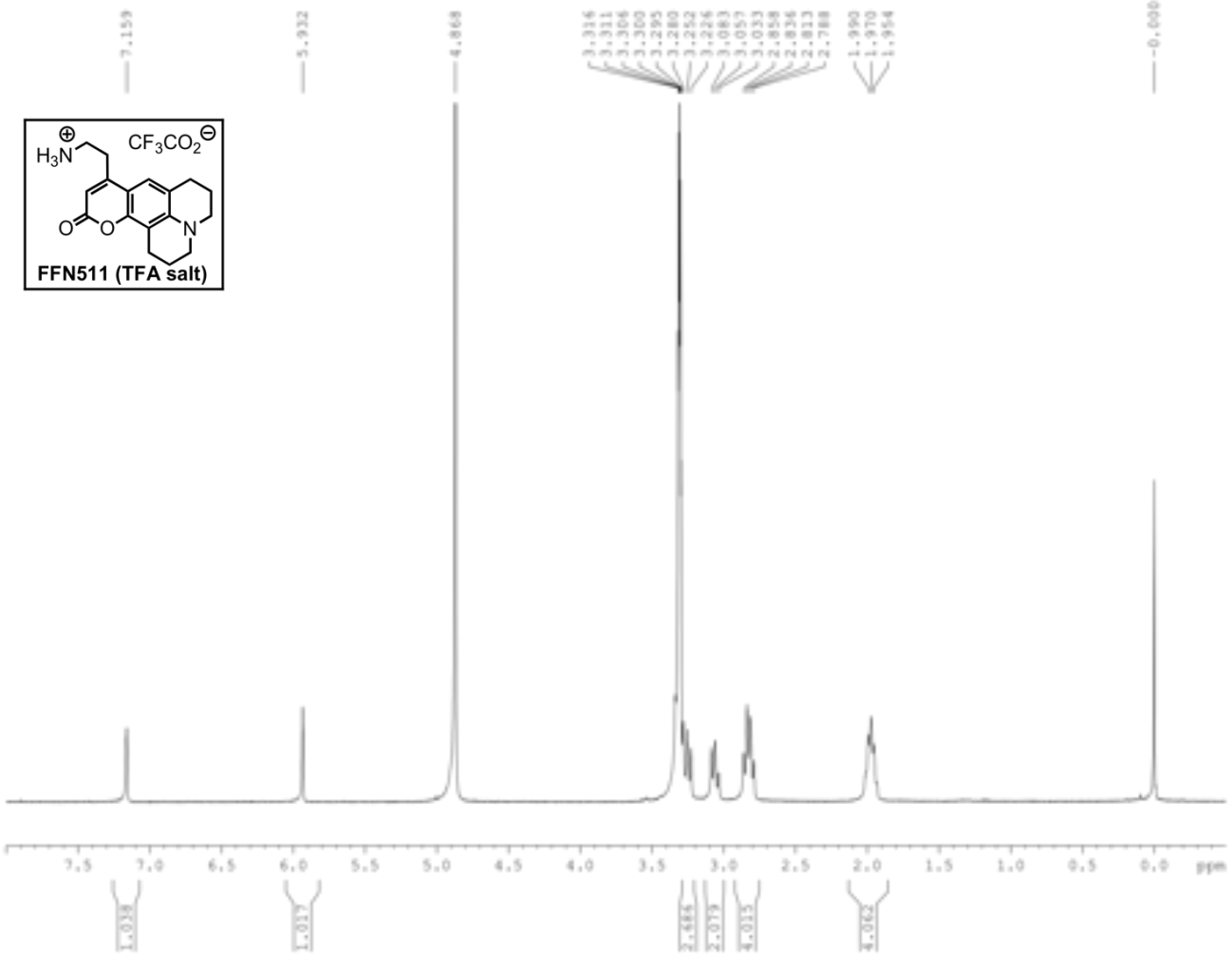

HO2C
H
N
BOC
33
156.740
155.796
87.968
80.940
77.585
77.162
76.738
74.617
39.270
28.551
20.587
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

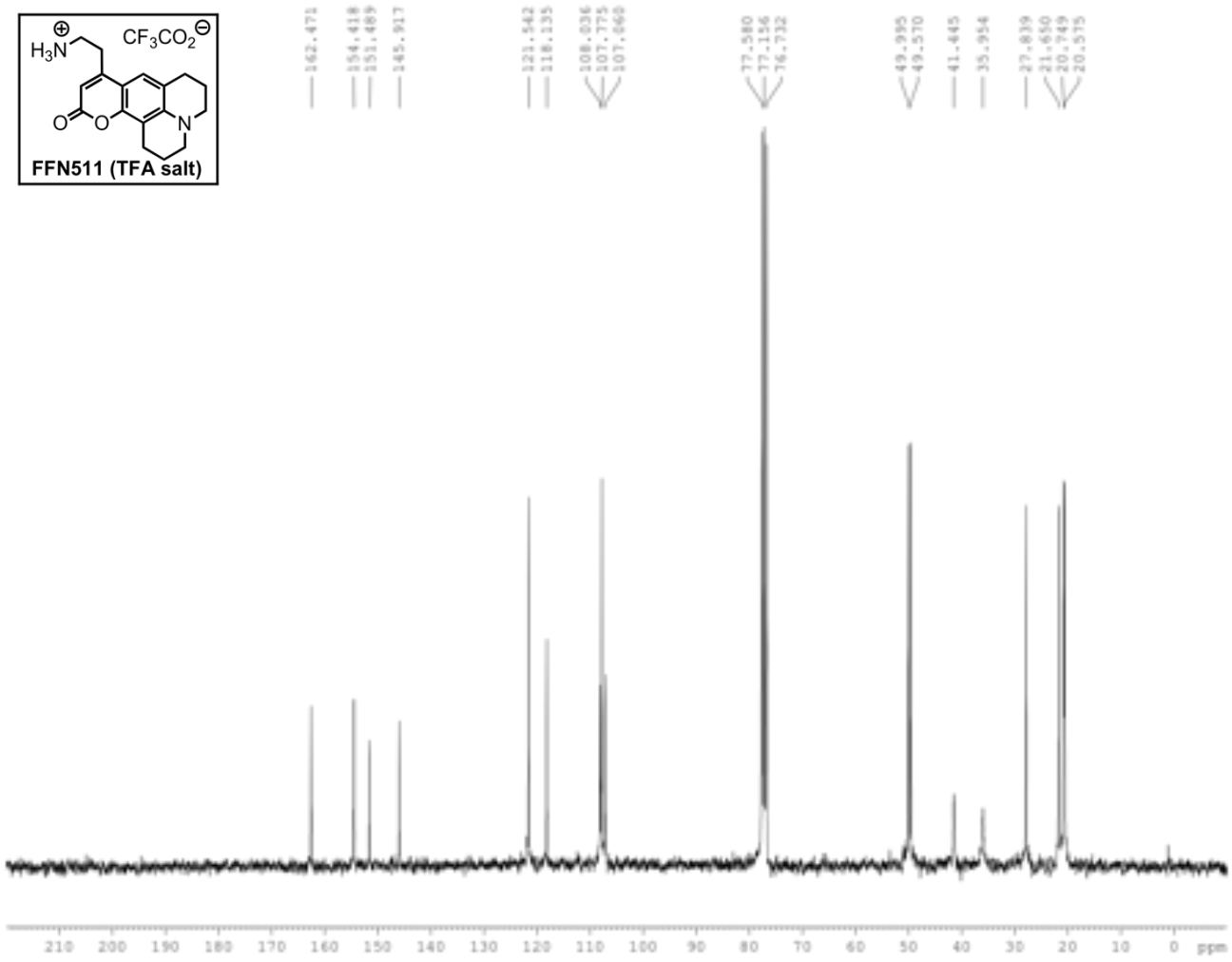

7.259
6.782
6.756
6.272
6.245
4.908
3.363
3.342
3.321
3.301
3.142
3.124
3.112
3.094
2.746
2.724
2.703
2.601
2.580
2.562
1.969
1.953
1.934
1.919
1.456
(BOC)HN
O
O
N
34
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.020
0.958
0.974
2.026
4.004
2.074
4.028
4.081
9.046

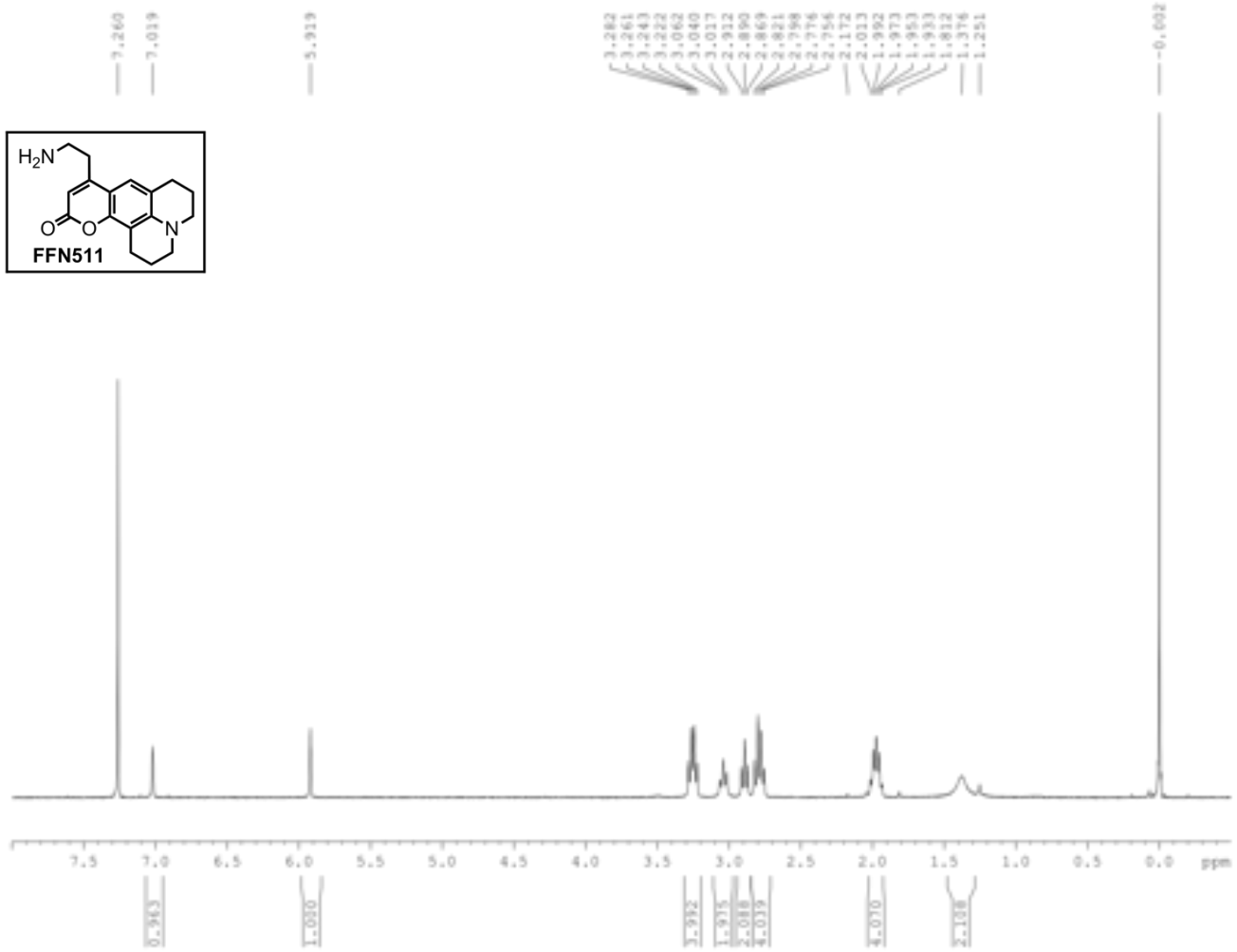

(BOC)HN
O
O
N
34
155.689
152.056
146.717
143.963
137.001
119.700
113.374
108.376
88.735
79.789
77.584
77.160
76.736
73.836
49.911
49.428
38.584
28.425
27.529
21.870
21.587
21.177
20.553
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

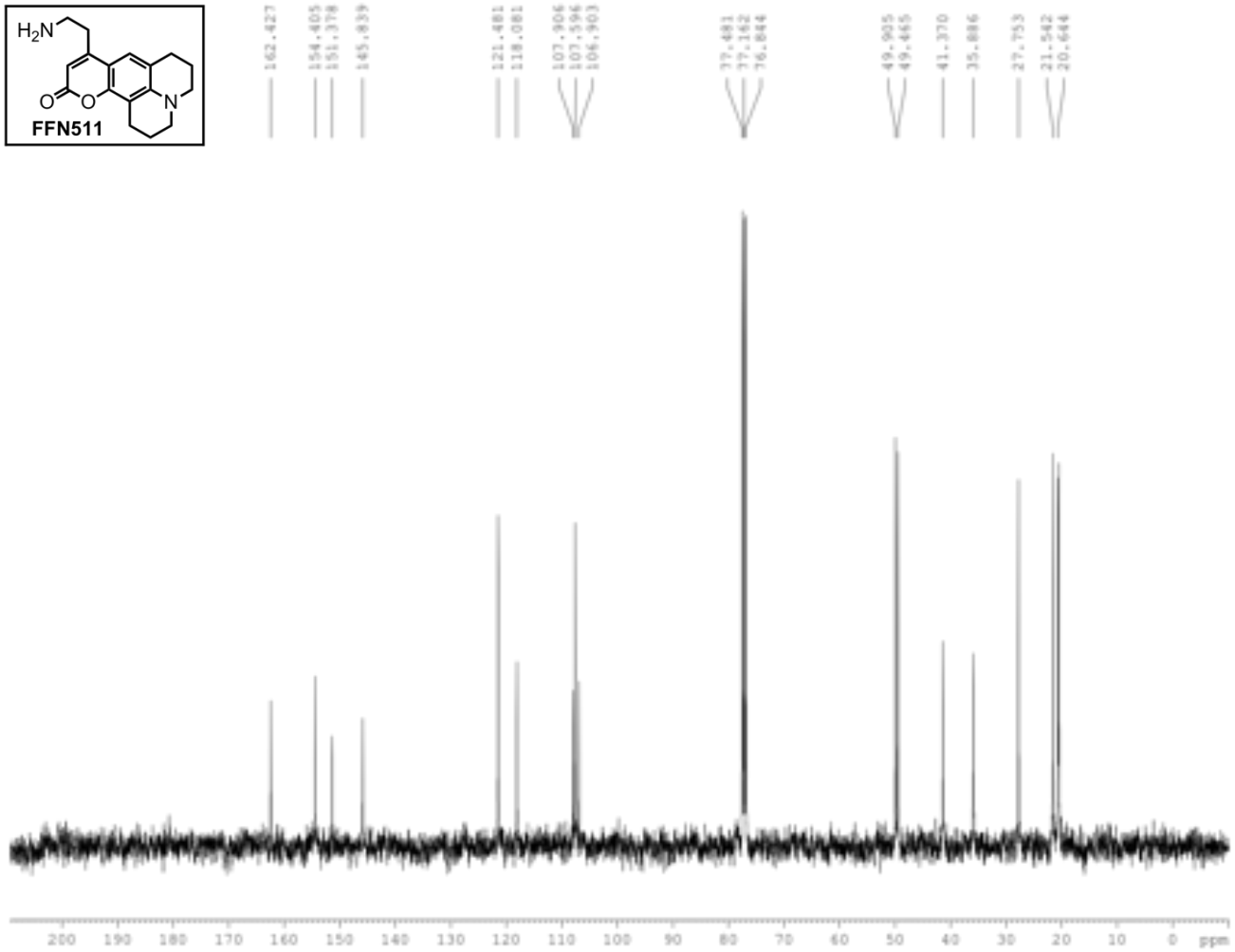

7.369
7.038
6.908
5.885
4.637
3.460
3.440
3.419
3.396
3.384
3.264
3.245
3.225
2.909
2.887
2.865
2.856
2.834
2.799
2.779
2.758
2.011
1.990
1.972
1.953
1.930
1.542
1.443
0.000
(BOC)HN
O
O
N
35
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.973
0.999
1.010
2.091
4.029
4.044
2.018
4.560
8.912

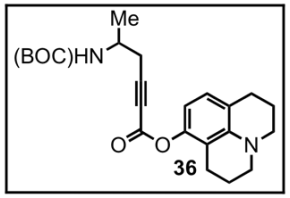

(BOC)HN
O
O
N
35
162.444
155.878
153.954
151.513
146.003
121.621
118.253
107.903
107.648
79.659
77.583
77.366
77.160
76.736
50.036
49.597
39.817
32.441
28.506
27.901
21.654
20.753
20.592
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

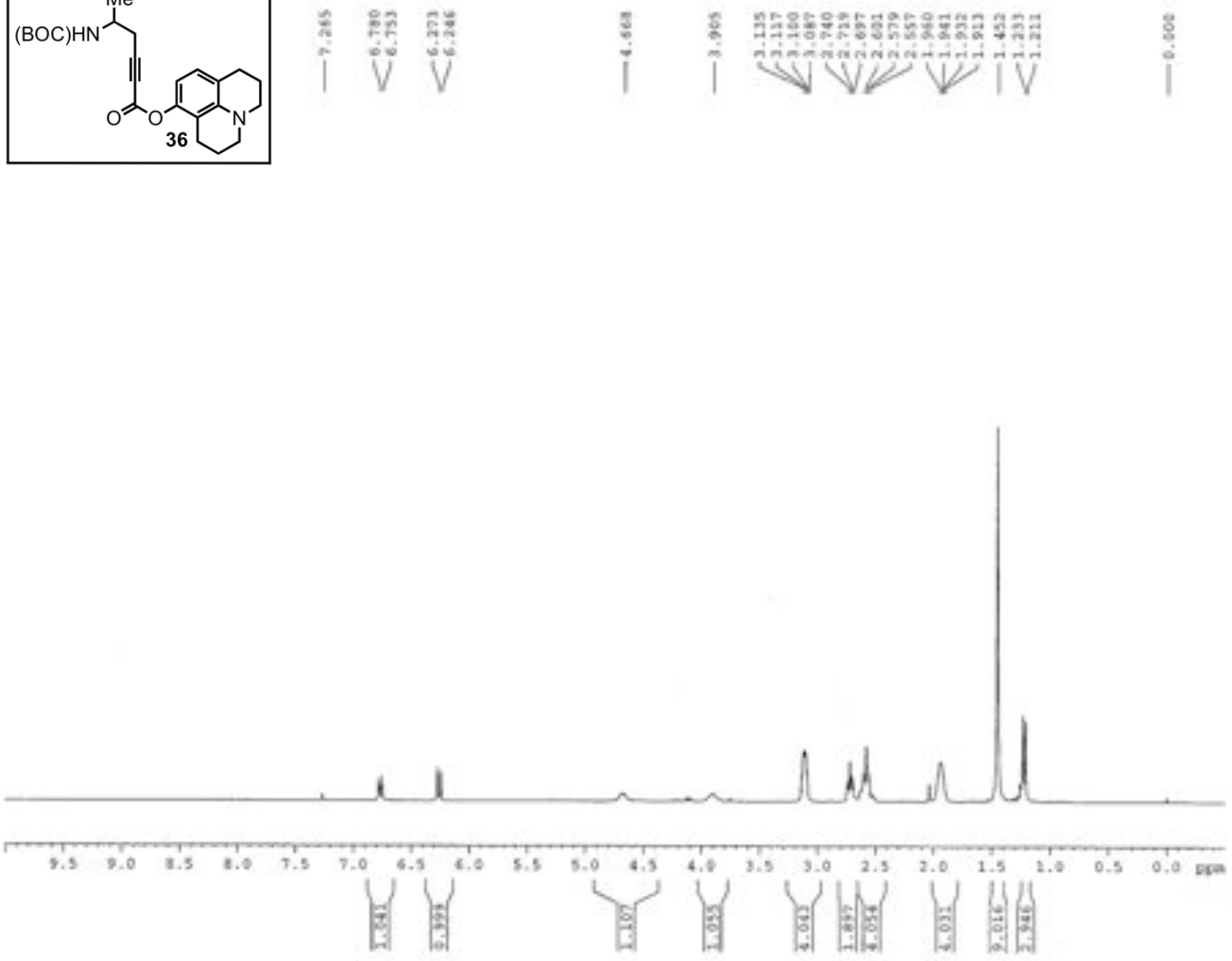

7.159
5.932
4.868
3.316
3.311
3.306
3.300
3.295
3.280
3.252
3.226
3.083
3.057
3.033
2.858
2.836
2.813
2.788
1.990
1.970
1.954
0.000
FFN511 (TFA salt)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.038
1.017
2.466
2.019
4.015
4.062

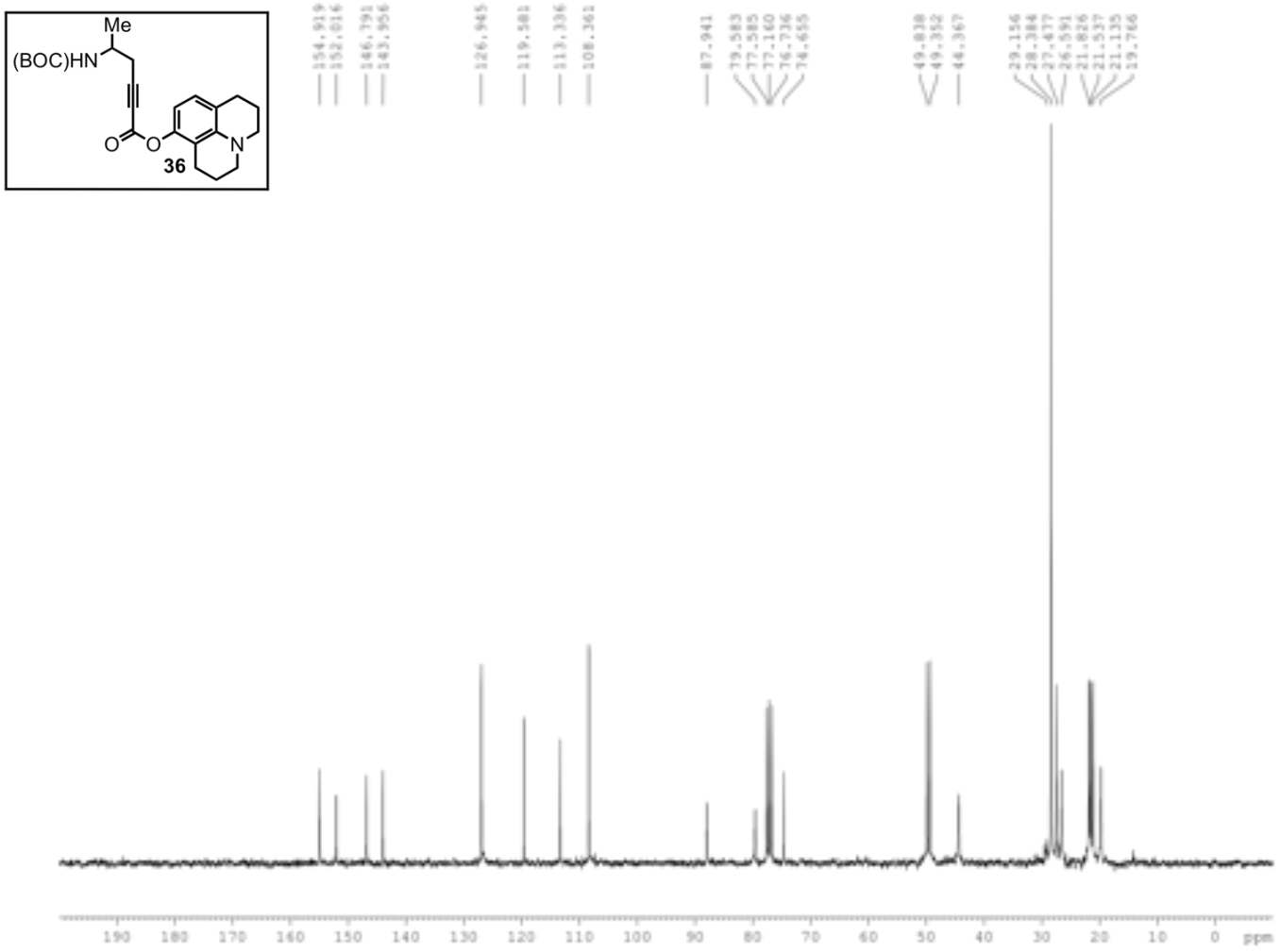

FFN511 (TFA salt)
162.471
154.418
151.489
145.917
121.542
118.135
108.036
107.775
107.660
77.580
77.156
76.732
49.995
49.570
41.445
35.954
27.839
21.650
20.749
20.575
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

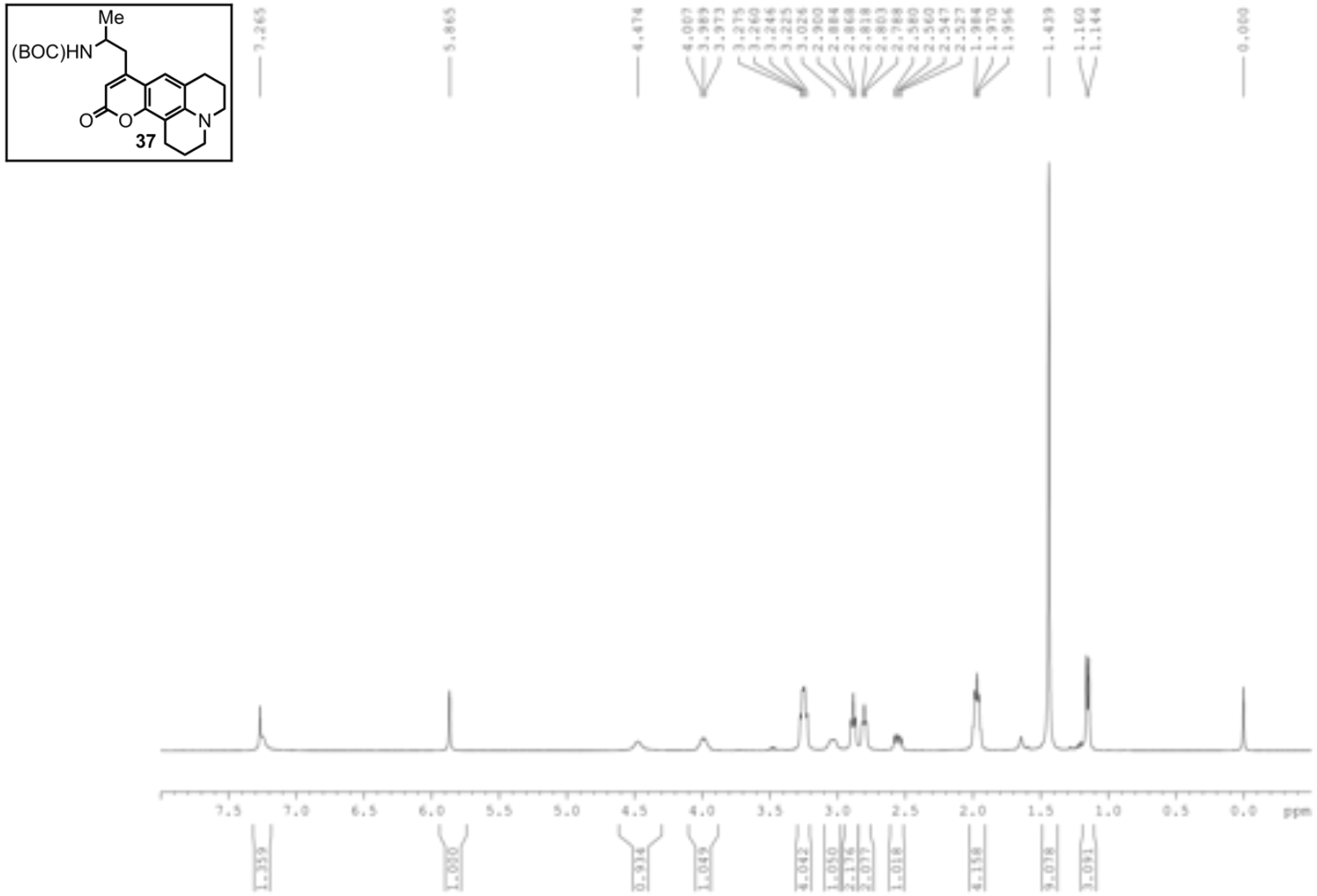

7.260
7.019
5.919
3.282
3.261
3.243
3.222
3.062
3.040
3.017
2.912
2.890
2.869
2.821
2.798
2.776
2.756
2.172
2.013
1.992
1.973
1.953
1.933
1.812
1.376
1.251
0.002
H2N
O
O
N
FFN511
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.963
1.000
3.992
1.915
2.088
4.019
4.070
2.108

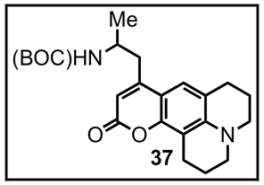

H2N
O
O
N
FFN511
162.427
154.405
151.378
145.839
121.481
118.081
107.906
107.596
106.903
77.481
77.162
76.844
49.905
49.465
41.370
35.886
27.753
21.542
20.644
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

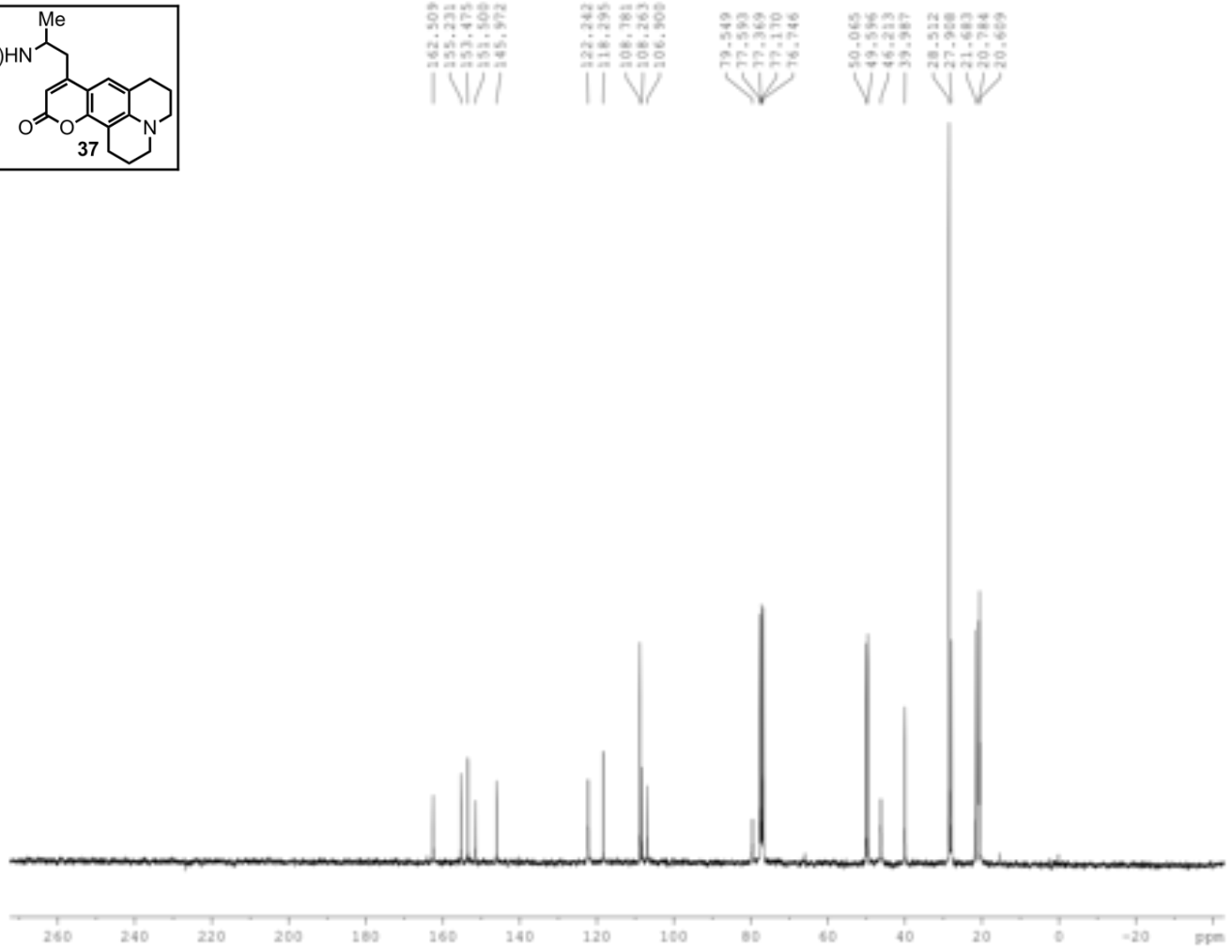

Me
(BOC)HN
O
O
N
36

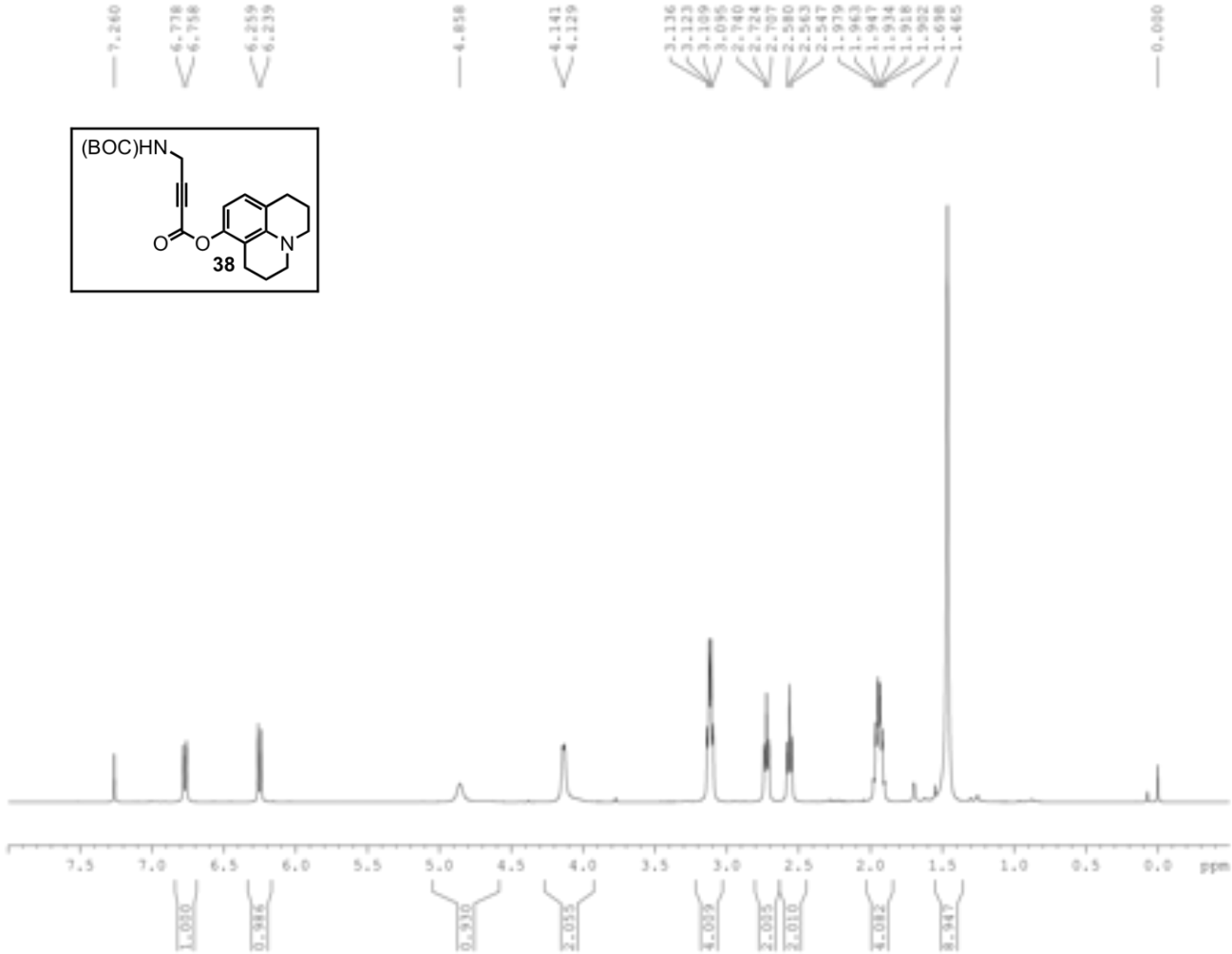

7.265
6.780
6.753
6.273
6.246
4.668
3.905
3.135
3.117
3.100
3.087
2.740
2.719
2.697
2.601
2.579
2.557
1.960
1.941
1.932
1.913
1.452
1.233
1.211
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.041
1.393
3.107
1.055
4.043
1.897
4.054
3.031
2.016
2.946

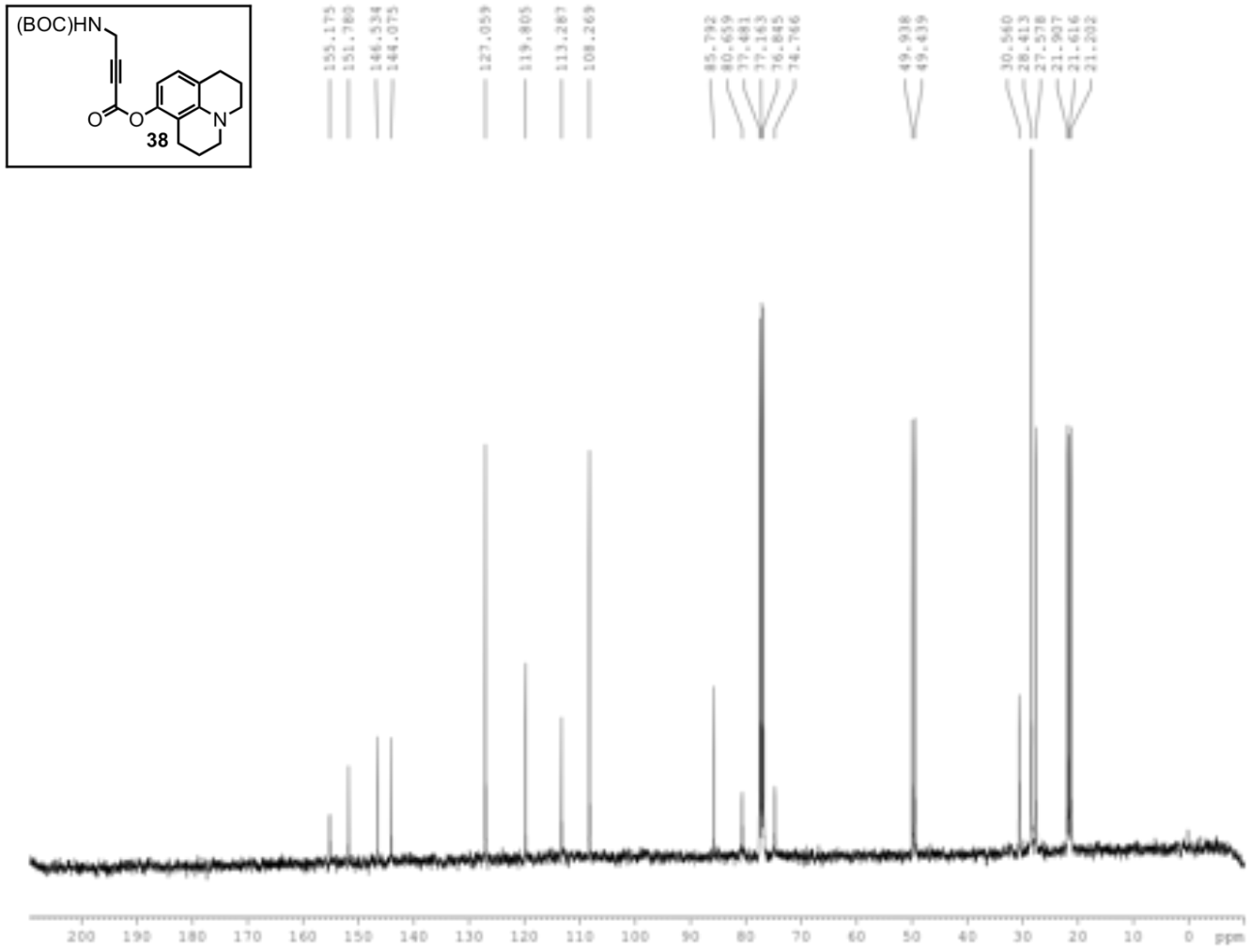

Me
(BOC)HN
O
O
N
36
154.919
152.016
146.791
143.956
126.945
119.581
113.336
108.361
87.941
79.583
77.585
77.160
76.736
74.655
49.838
49.352
44.367
29.156
28.384
27.477
26.591
21.826
21.537
21.135
19.766
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

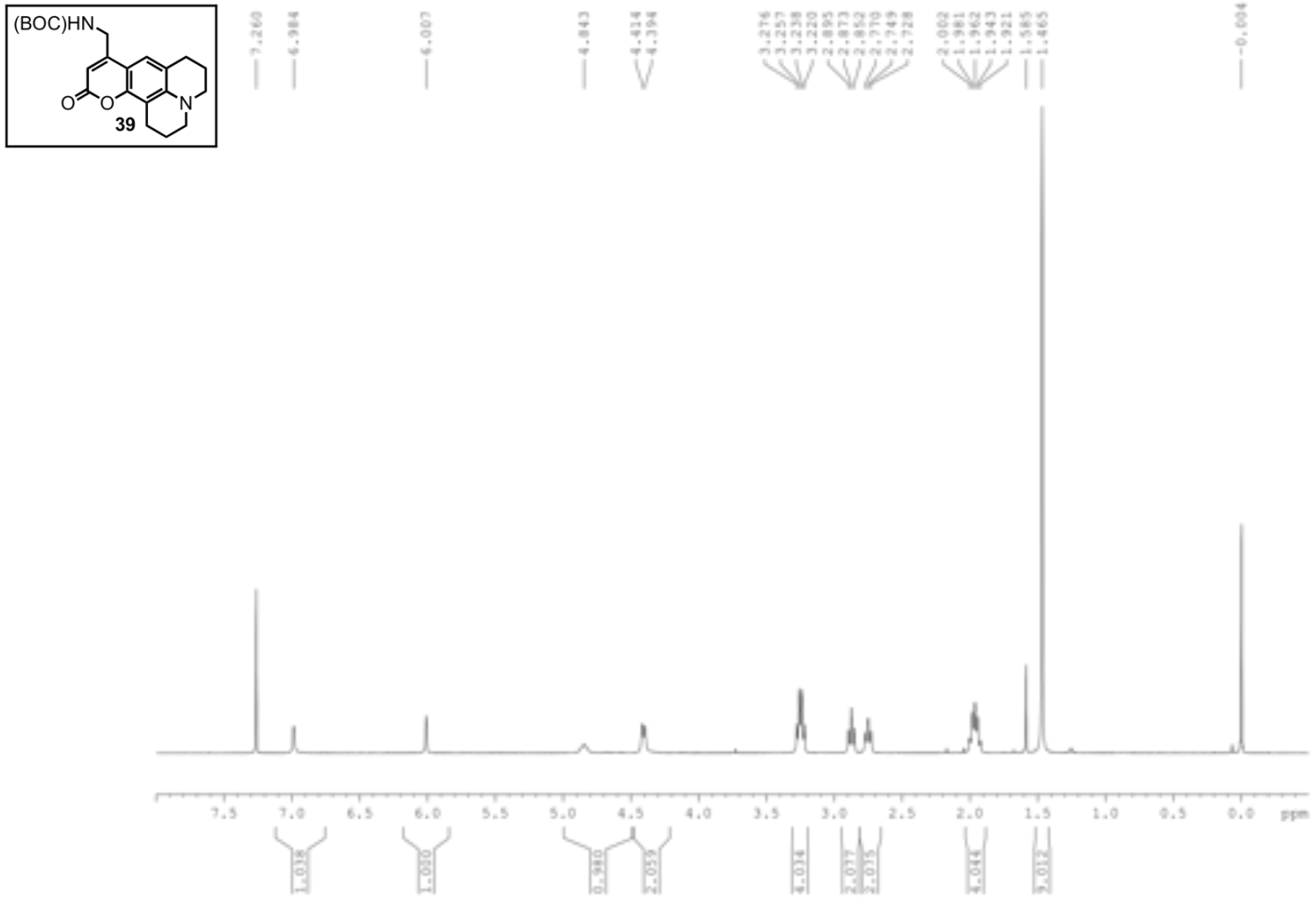

Me
(BOC)HN
O
O
N
37
7.265
5.865
4.474
4.007
3.989
3.973
3.275
3.260
3.246
3.225
3.026
2.900
2.884
2.868
2.818
2.803
2.788
2.580
2.560
2.547
2.527
1.984
1.970
1.956
1.439
1.160
1.144
0.000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.359
1.000
0.914
1.045
4.042
1.050
1.116
2.017
1.018
4.158
4.078
3.091

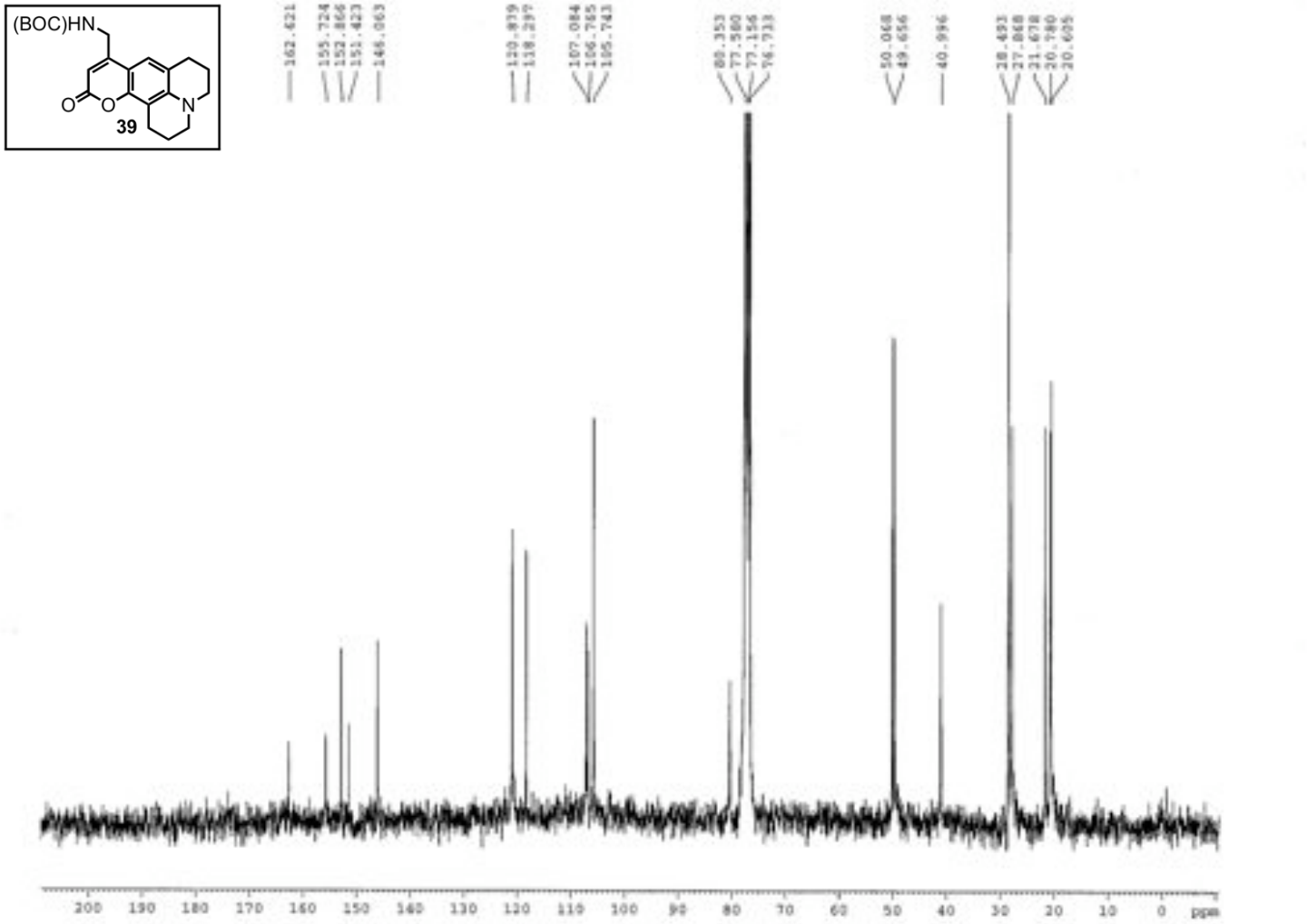

Me
(BOC)HN
O
O
N
37

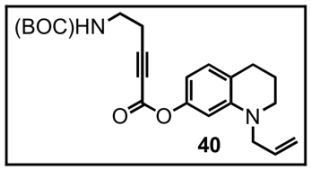

162.509
155.231
153.475
151.500
145.972
122.242
118.295
108.781
108.263
106.900
79.549
77.593
77.369
77.170
76.746
50.065
49.596
46.213
39.987
28.512
27.908
21.683
20.784
20.609
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

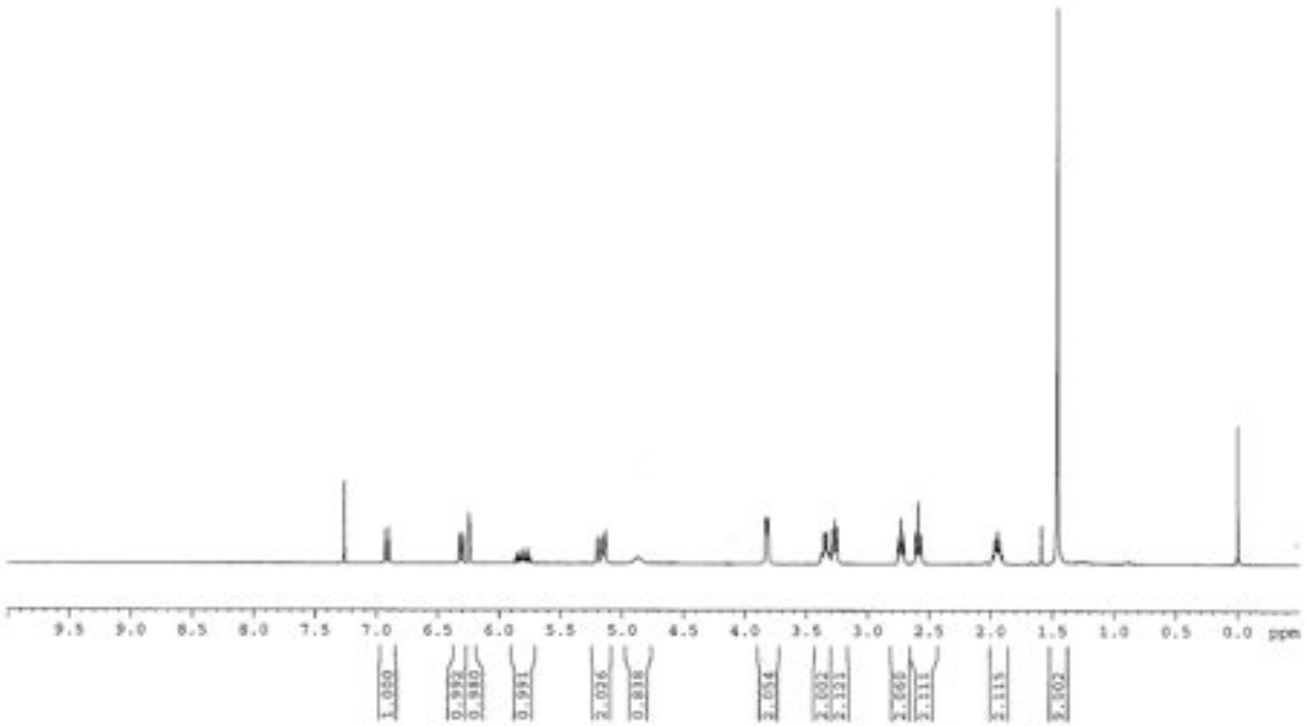

7.260
6.778
6.758
6.259
6.239
4.858
4.141
4.129
3.136
3.123
3.109
3.095
2.740
2.724
2.707
2.580
2.563
2.547
1.979
1.963
1.947
1.934
1.918
1.902
1.698
1.465
0.000
(BOC)HN
O
O
N
38
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.010
0.986
0.930
2.055
4.009
2.005
2.010
4.082
8.947

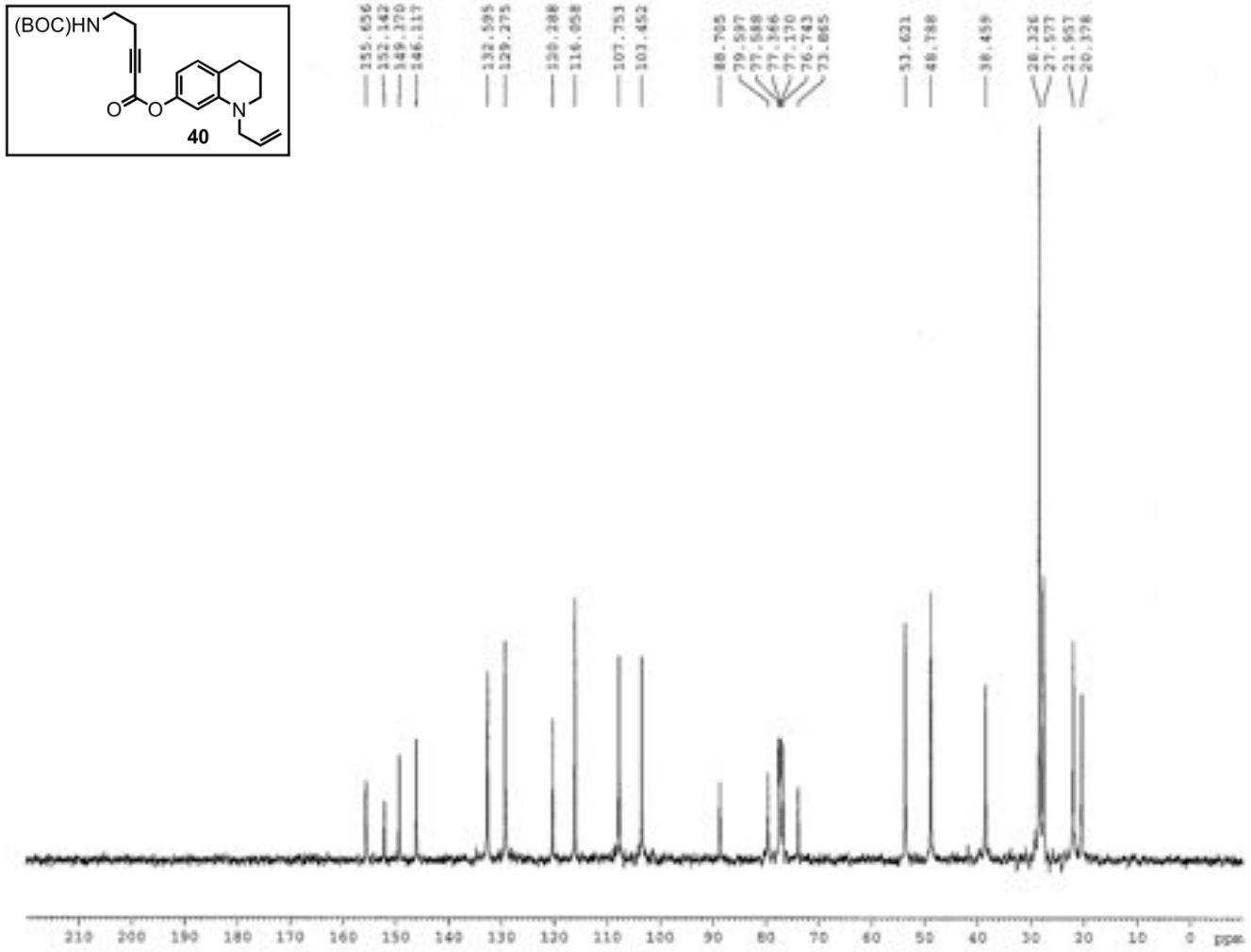

(BOC)HN
O
O
N
38
155.175
151.780
146.534
144.075
127.059
119.805
113.287
108.269
85.792
80.659
77.481
77.163
76.845
74.766
49.938
49.439
30.560
28.413
27.578
21.997
21.616
21.202
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

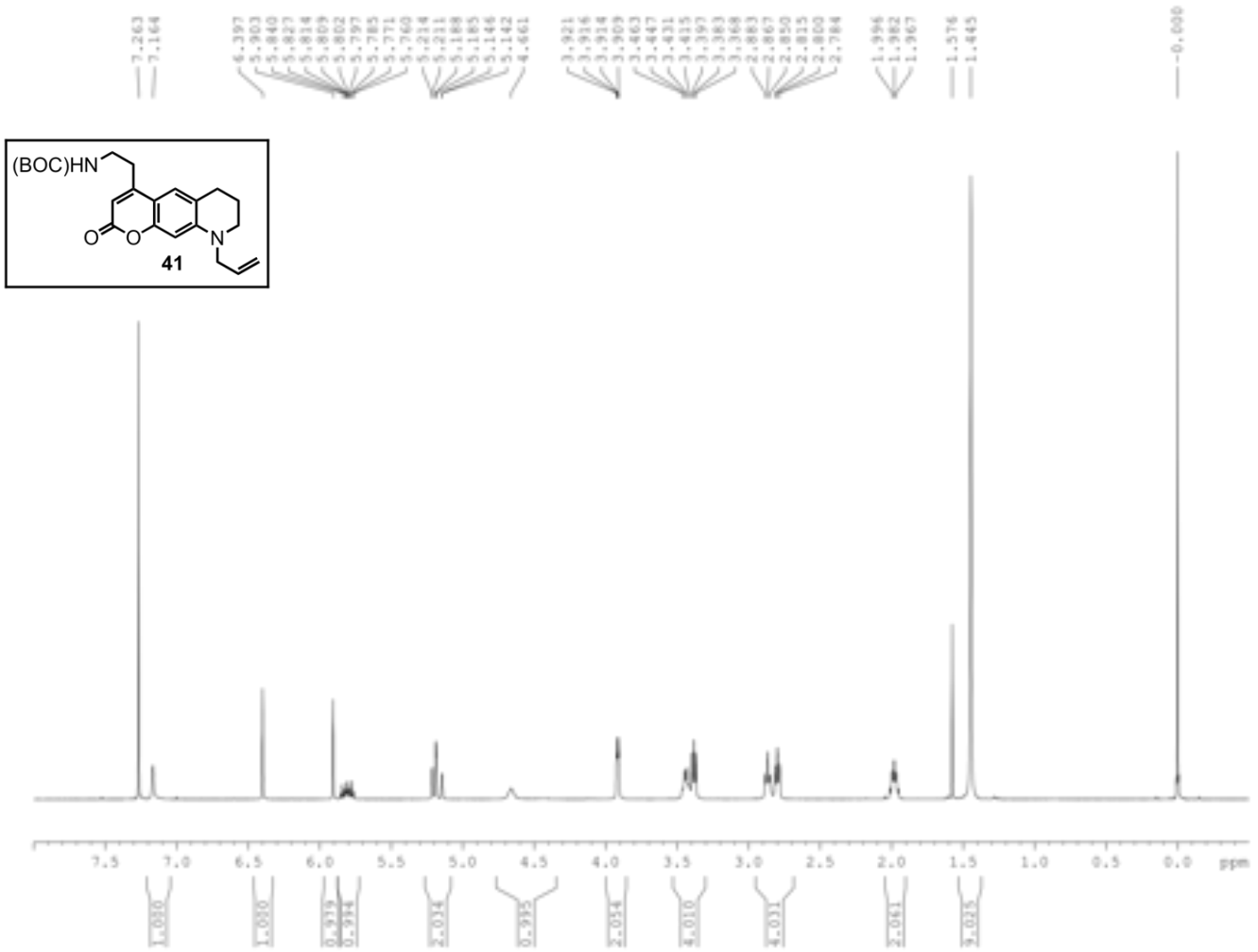

(BOC)HN
O
O
N
39
7.260
6.984
6.007
4.843
4.414
4.394
3.276
3.257
3.238
3.220
2.895
2.873
2.852
2.770
2.749
2.728
2.002
1.981
1.962
1.943
1.921
1.585
1.465
0.004
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.016
1.000
0.940
2.059
3.014
2.017
2.015
3.044
9.012

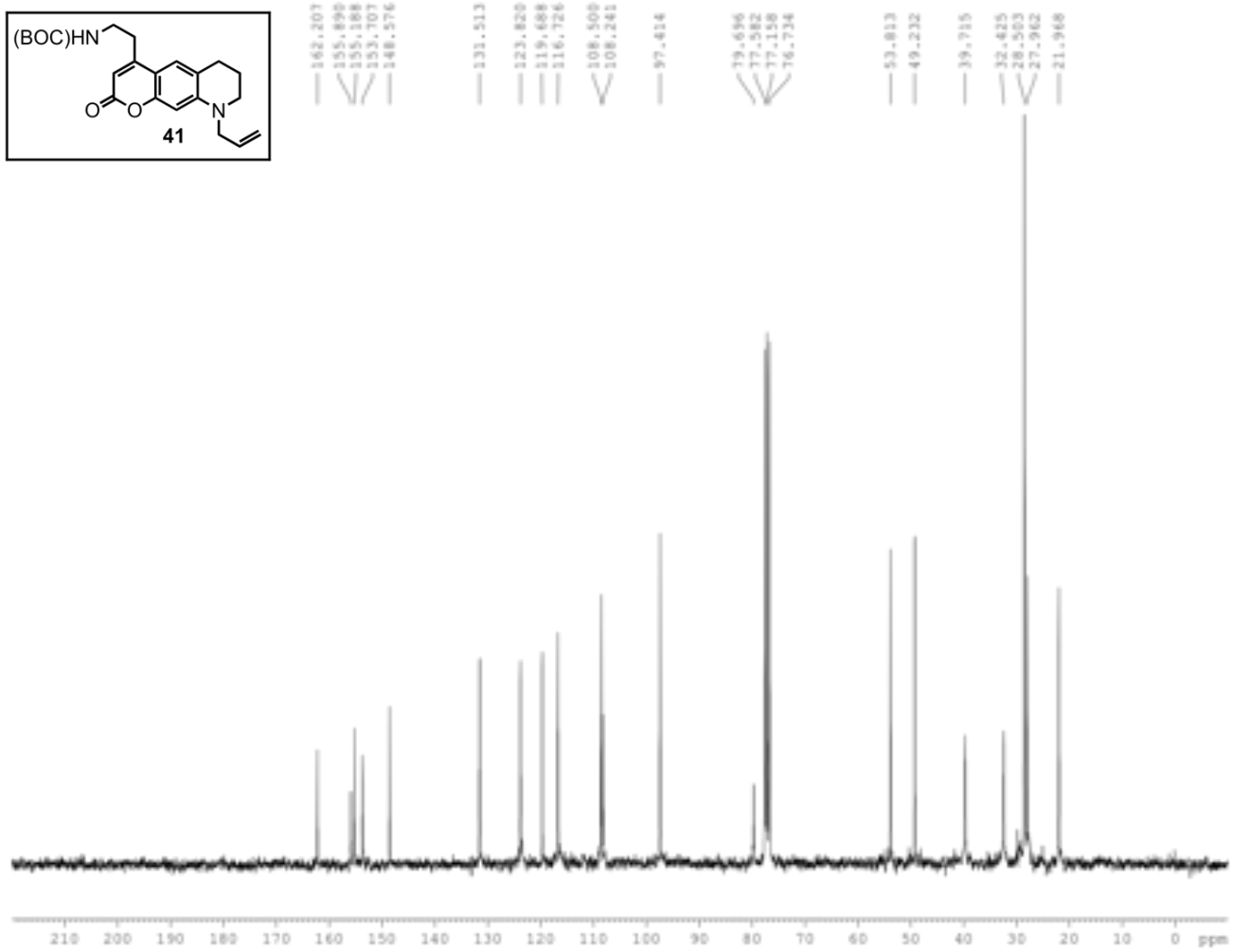

(BOC)HN
O
O
N
39
162.621
155.724
152.866
151.423
146.063
130.879
118.299
107.084
106.765
105.743
80.353
77.580
77.156
76.733
50.068
49.656
40.996
28.493
27.868
21.678
20.790
20.605
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

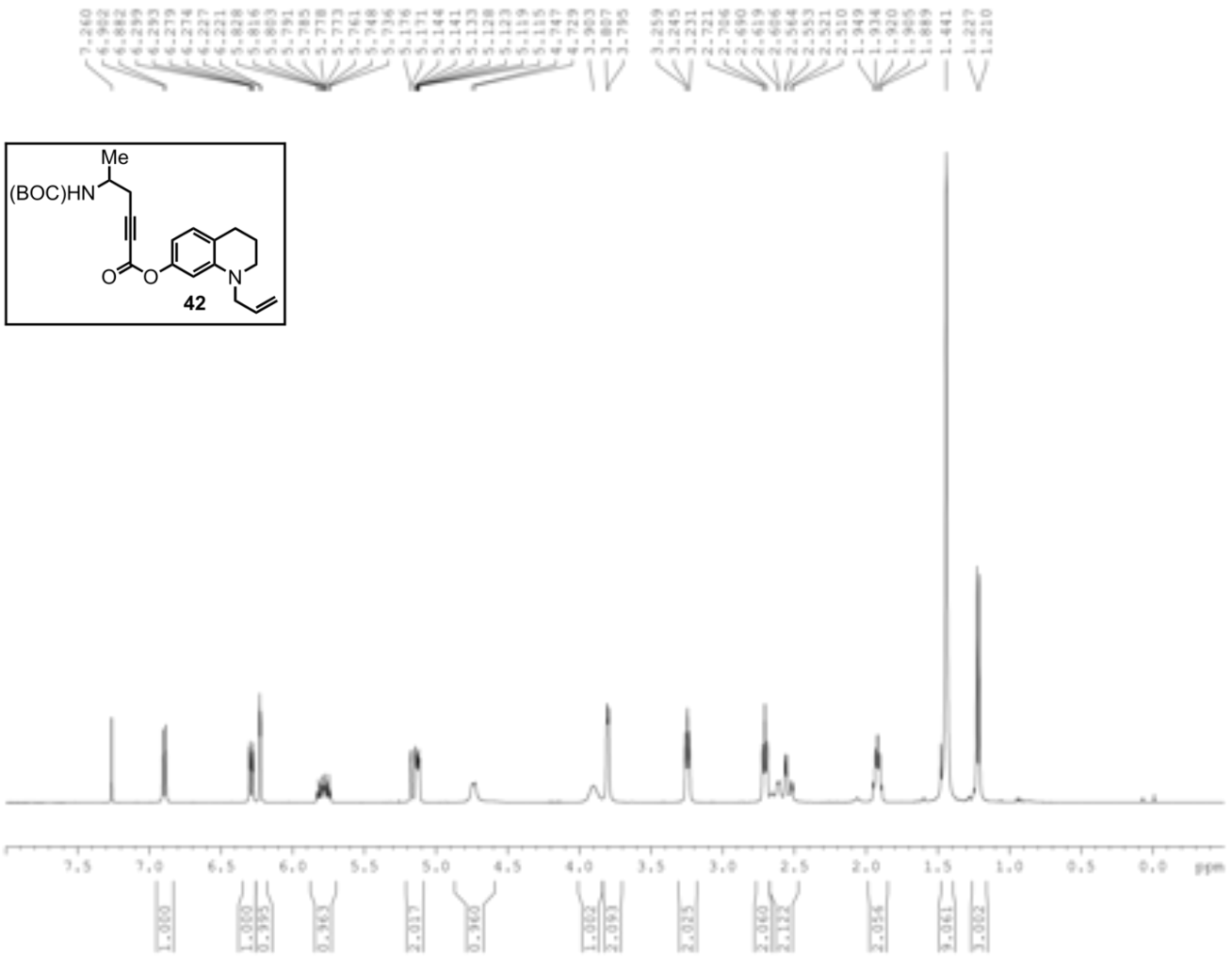

(BOC)HN
O
O
N
40

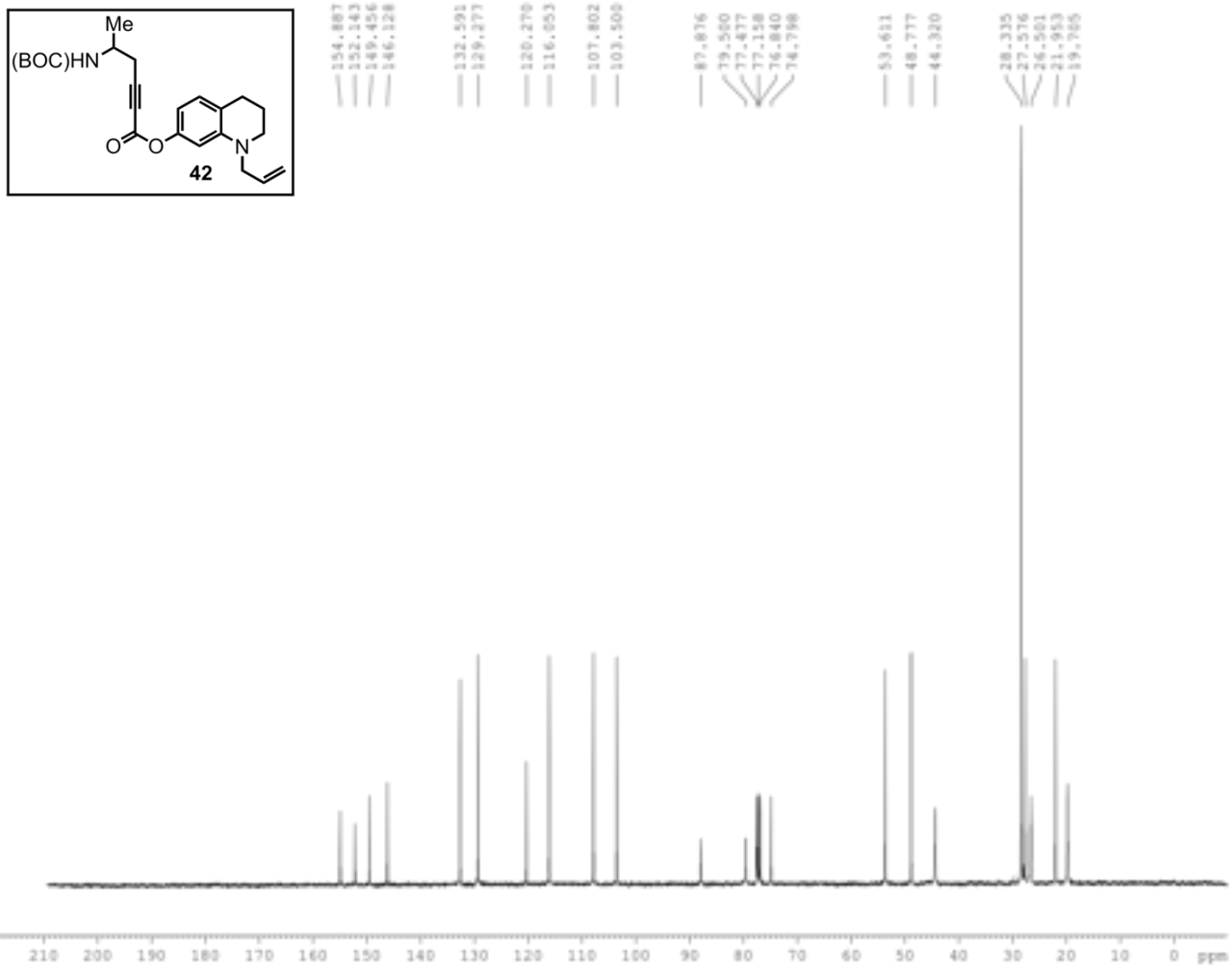

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
0.992
0.980
0.991
2.026
0.818
2.054
2.002
2.121
2.668
2.111
3.115
2.902

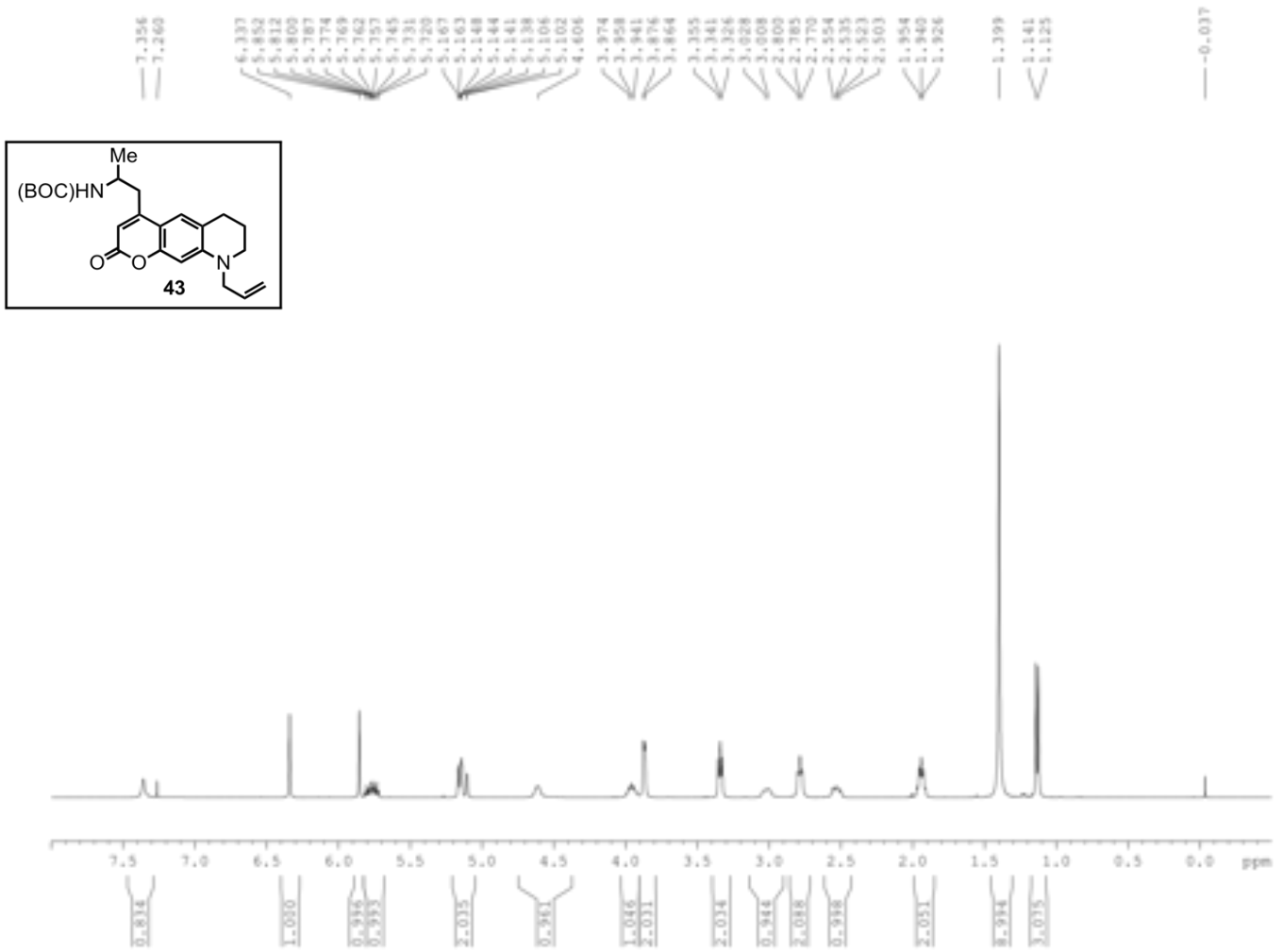

(BOC)HN
O
O
N
40
155.656
152.142
149.370
146.117
132.595
129.275
120.388
116.058
107.753
103.452
80.705
79.597
77.588
77.366
77.170
76.743
73.865
53.621
48.788
30.459
28.326
27.577
21.957
20.378
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

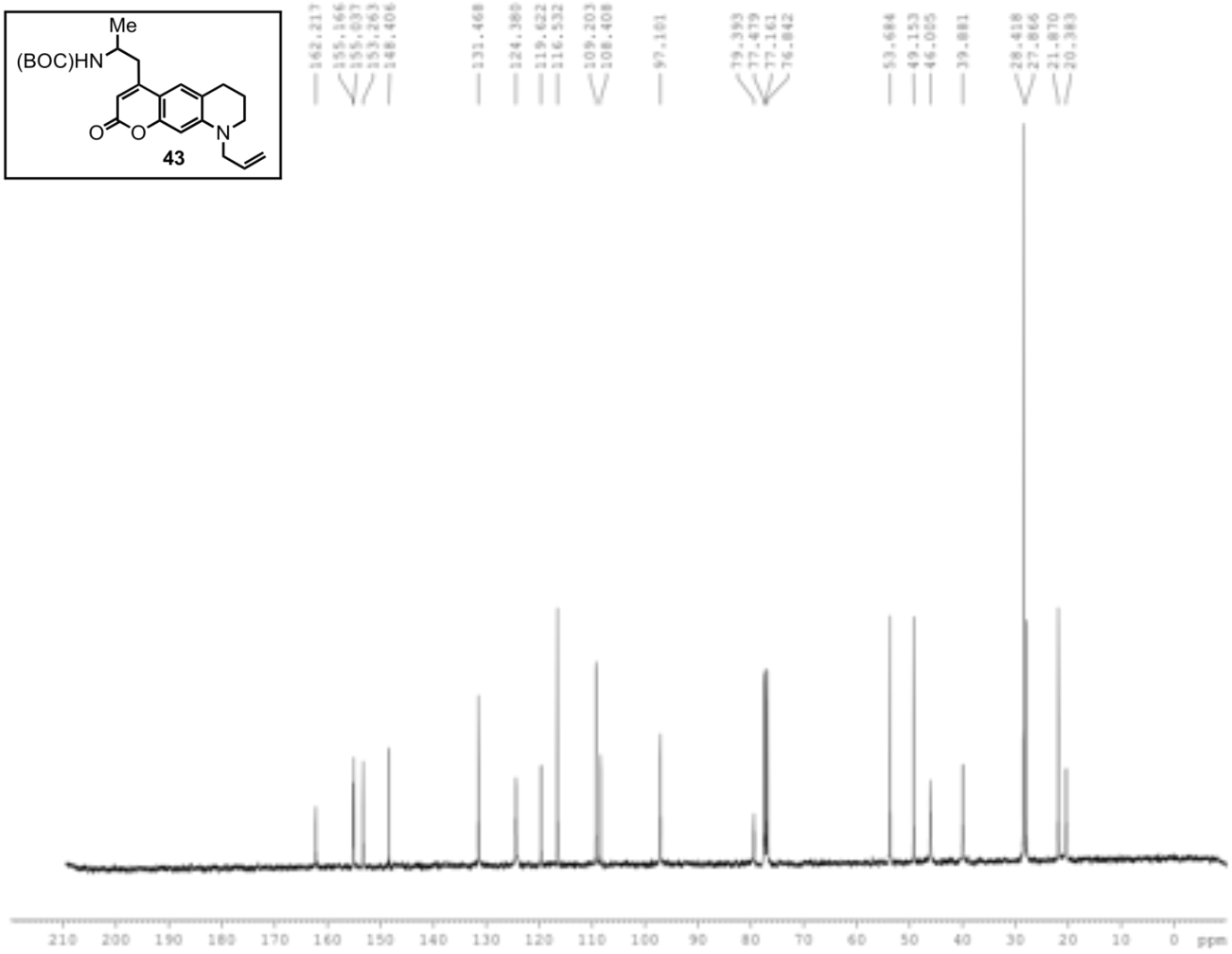

(BOC)HN
O
O
N
41
7.263
7.164
6.397
5.903
5.840
5.827
5.814
5.809
5.802
5.797
5.785
5.771
5.760
5.234
5.211
5.188
5.165
5.146
5.142
4.461
3.921
3.916
3.914
3.909
3.463
3.447
3.431
3.415
3.397
3.383
3.368
2.883
2.867
2.850
2.815
2.800
2.784
1.996
1.982
1.967
1.576
1.445
0.000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.000
1.000
0.939
0.994
2.034
0.995
2.054
4.010
4.011
2.061
9.025

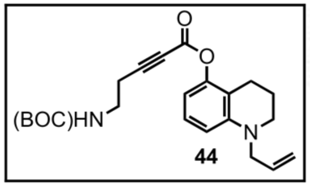

(BOC)HN
O
O
N
41
162.207
155.890
155.188
153.707
148.576
131.513
123.820
119.688
116.726
108.500
108.241
97.414
79.696
77.582
77.158
76.734
53.813
49.232
39.715
32.425
28.503
27.962
21.968
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

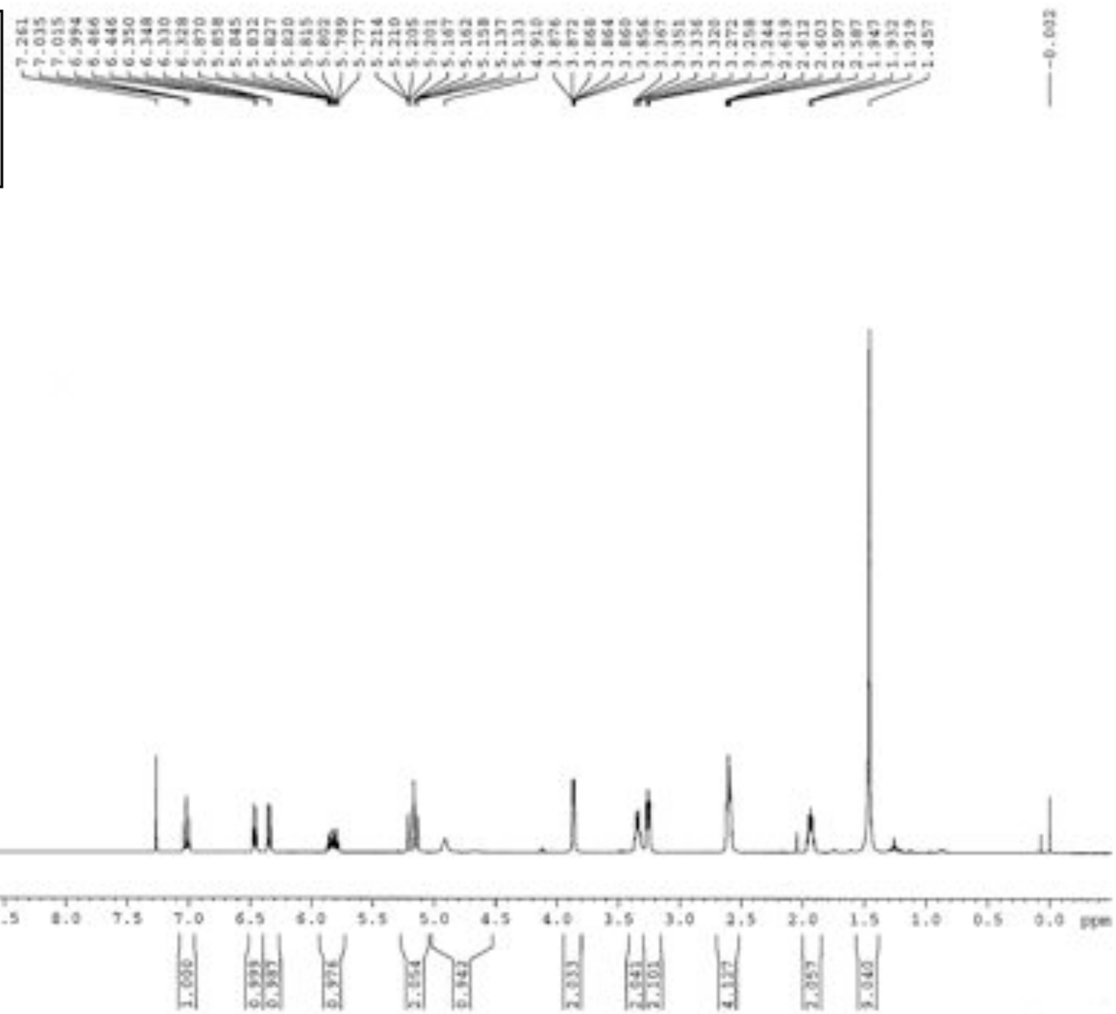

7.260
6.902
6.882
6.299
6.293
6.279
6.274
6.227
6.221
5.828
5.816
5.803
5.791
5.785
5.778
5.773
5.761
5.748
5.736
5.176
5.171
5.144
5.141
5.133
5.128
5.123
5.119
5.115
4.747
4.729
3.903
3.807
3.795
3.259
3.245
3.231
2.721
2.706
2.690
2.619
2.606
2.564
2.553
2.521
2.510
1.949
1.934
1.920
1.905
1.889
1.441
1.227
1.210
Me
(BOC)HN
O
O
N
42
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
ppm
1.000
1.000
0.995
0.963
2.017
0.960
1.002
2.093
2.035
2.060
2.122
2.054
9.061
3.002

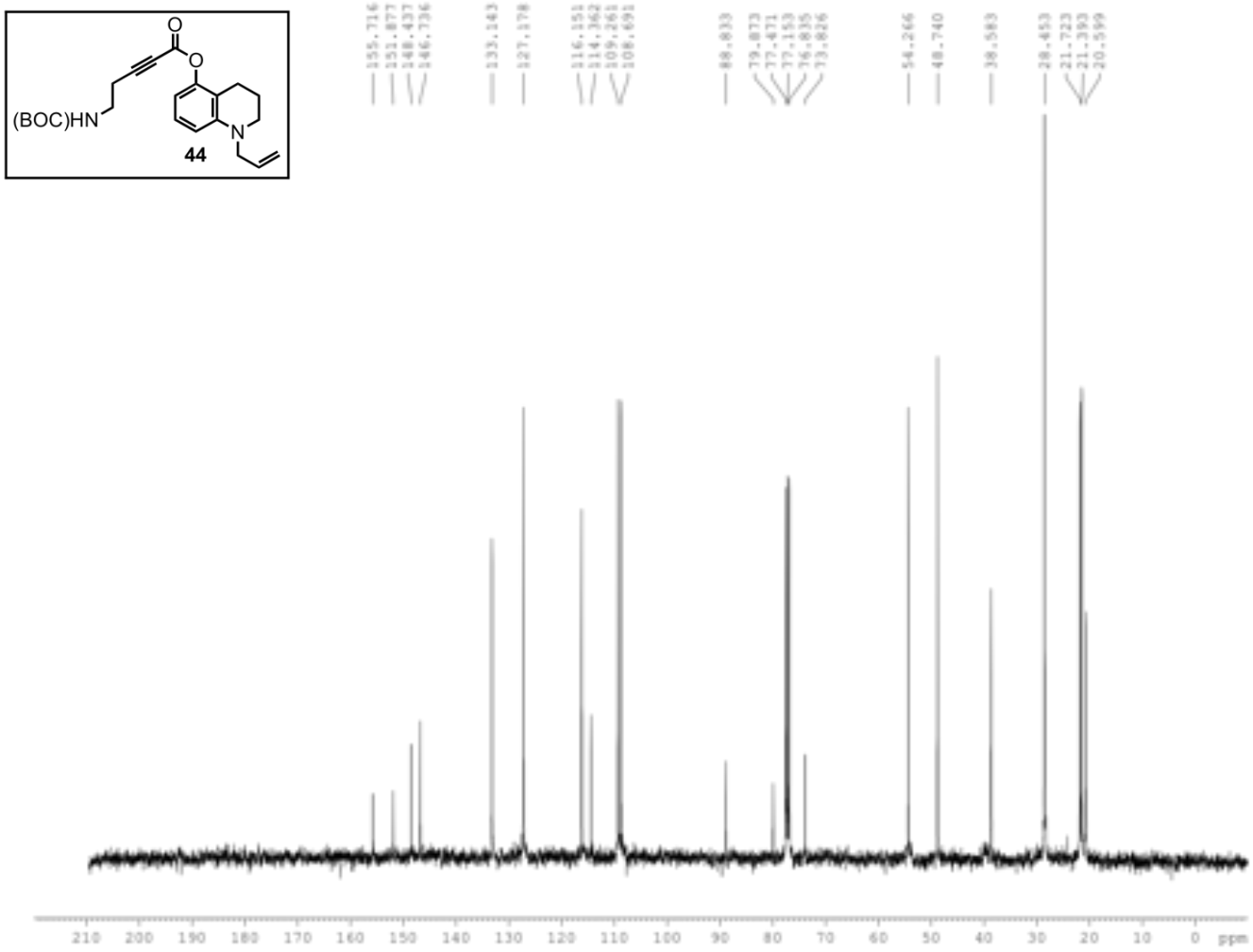

Me
(BOC)HN
O
O
N
42
154.887
152.143
149.456
146.128
132.591
129.277
120.270
116.053
107.802
103.500
87.876
79.560
77.477
77.158
76.840
74.798
53.611
48.777
44.320
28.335
27.576
26.501
21.953
19.705
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

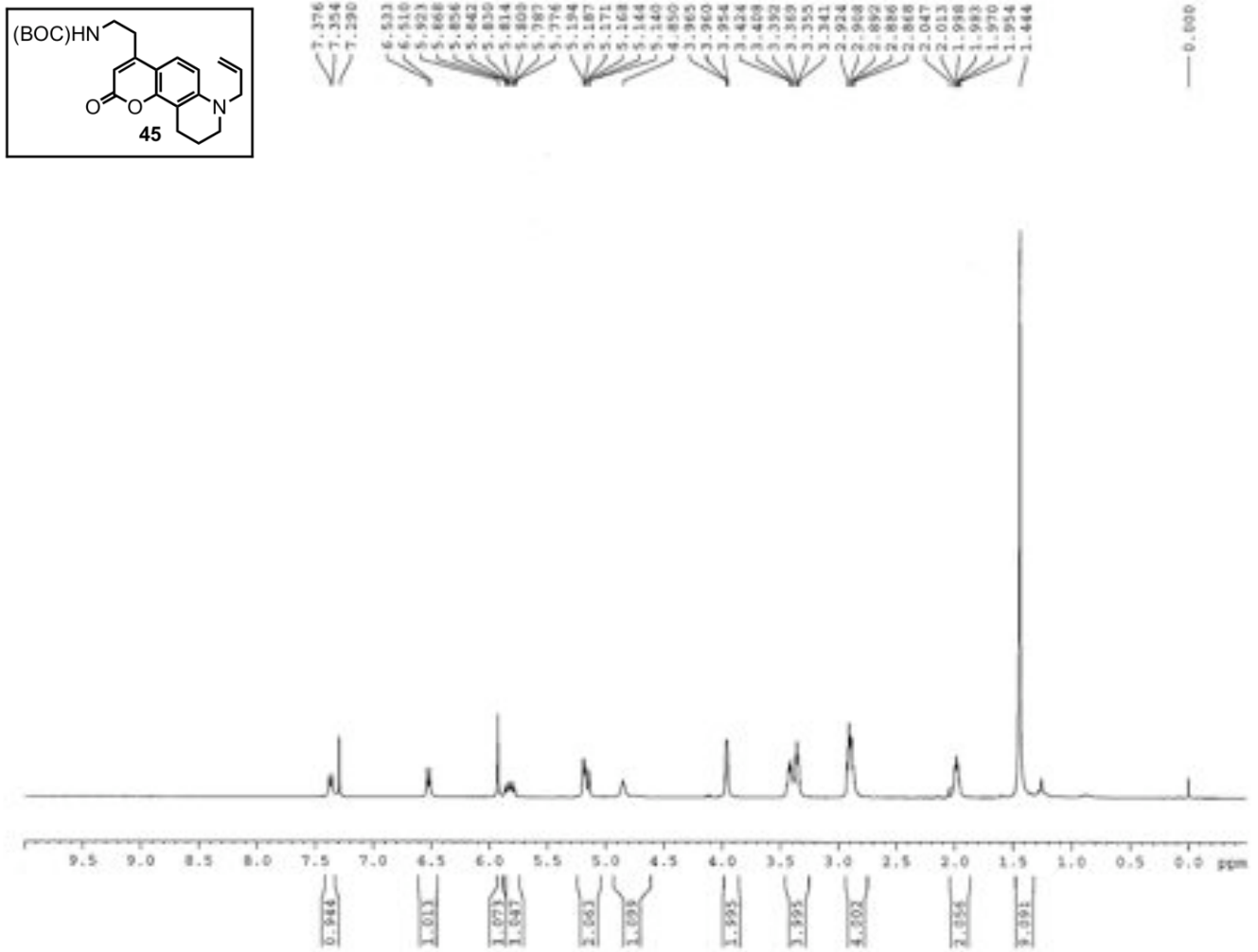

7.356
7.260
6.337
5.852
5.812
5.800
5.787
5.774
5.769
5.762
5.757
5.745
5.731
5.720
5.167
5.163
5.148
5.144
5.141
5.138
5.106
5.102
4.606
3.974
3.958
3.941
3.876
3.864
3.355
3.341
3.326
3.028
3.008
2.800
2.785
2.770
2.554
2.535
2.523
2.503
1.954
1.940
1.926
1.399
1.141
1.125
-0.037
Me
(BOC)HN
O
O
N
43
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
ppm
0.814
1.000
0.936
0.955
2.035
0.981
1.046
2.031
2.034
0.944
2.088
0.998
2.051
8.994
3.035

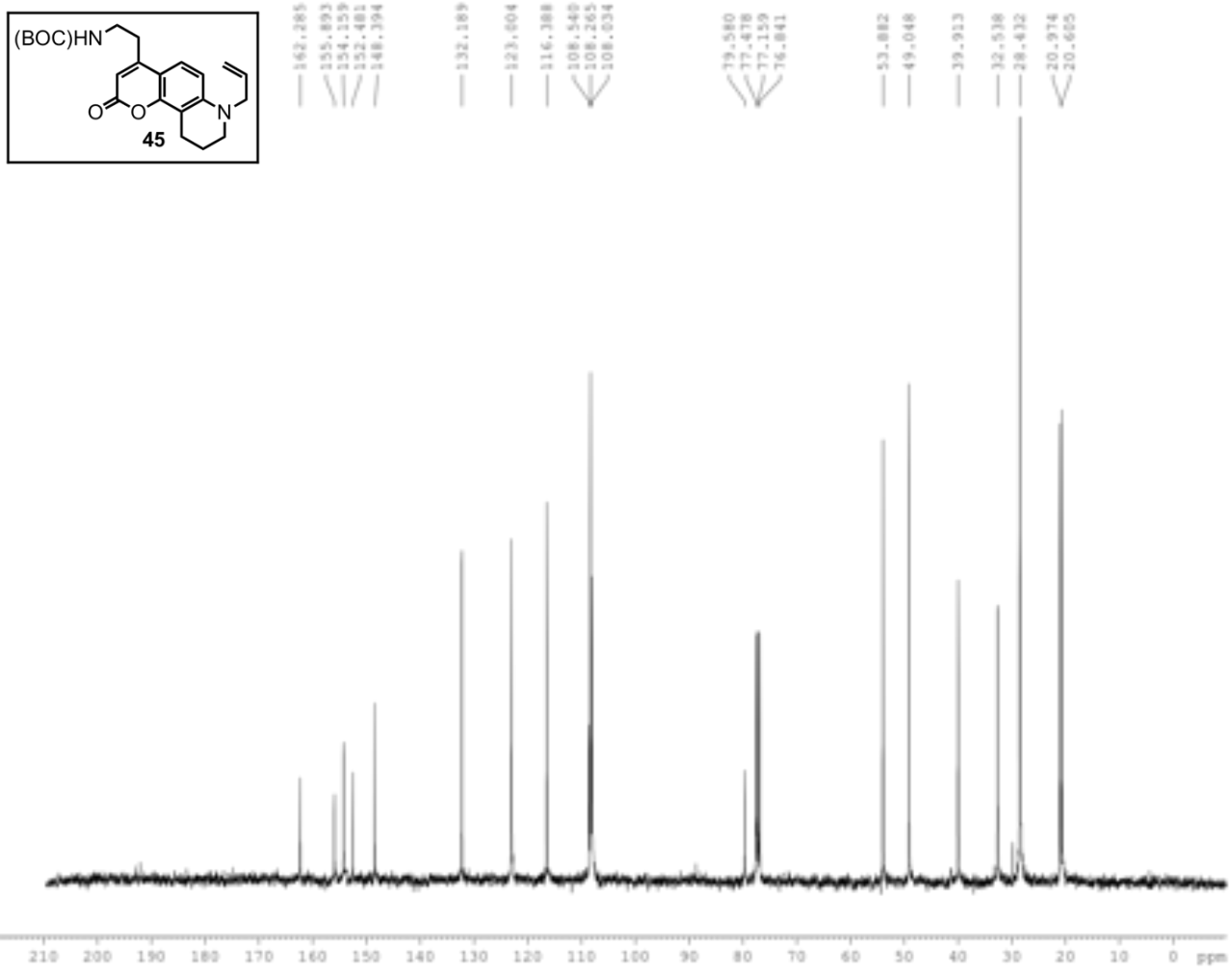

Me
(BOC)HN
O
O
N
43
162.217
155.166
155.037
153.263
148.406
131.468
124.380
119.622
116.532
109.203
108.408
97.101
79.393
77.479
77.161
76.842
53.684
49.153
46.005
39.881
28.418
27.866
21.870
20.383
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

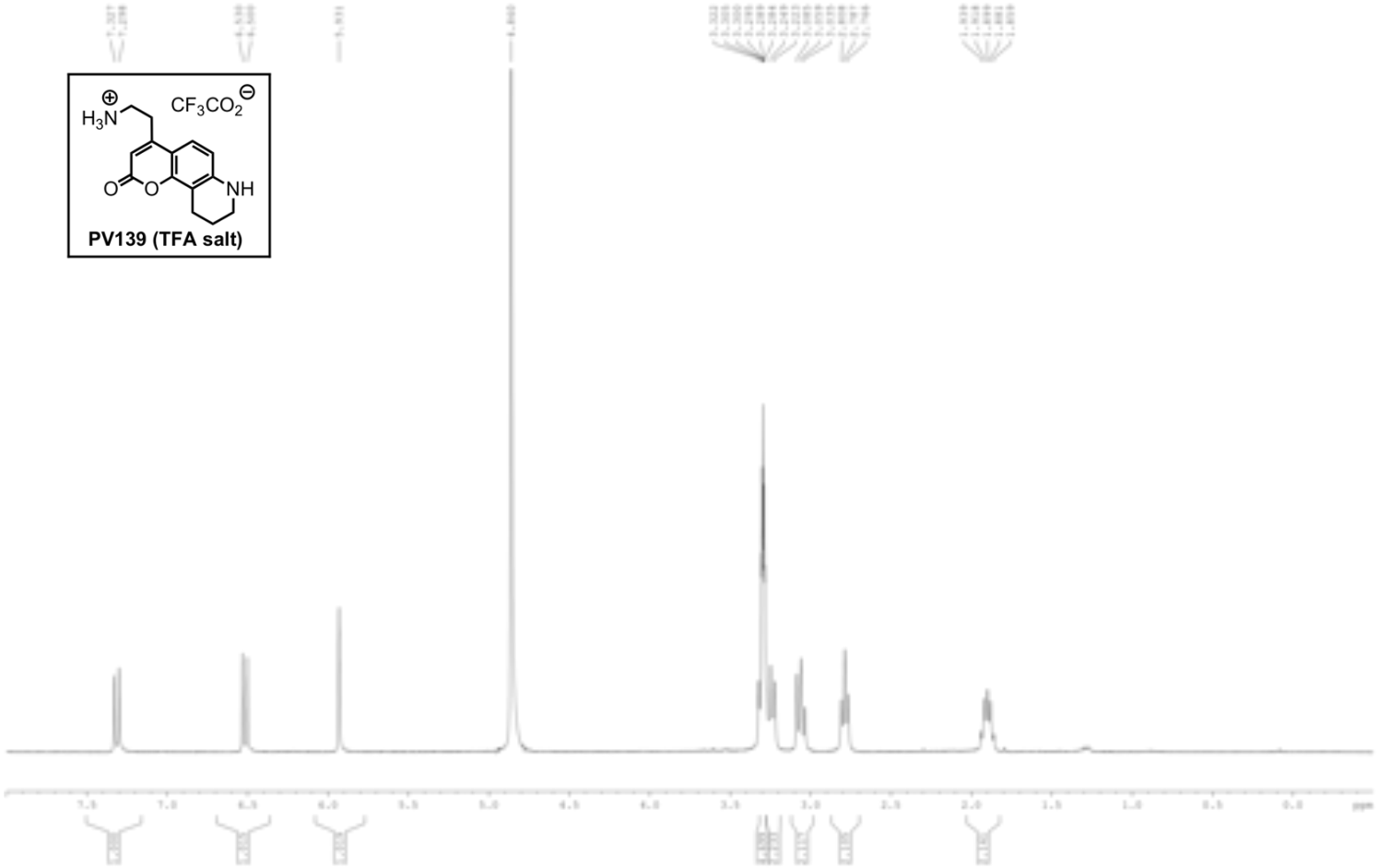

(BOC)HN
44

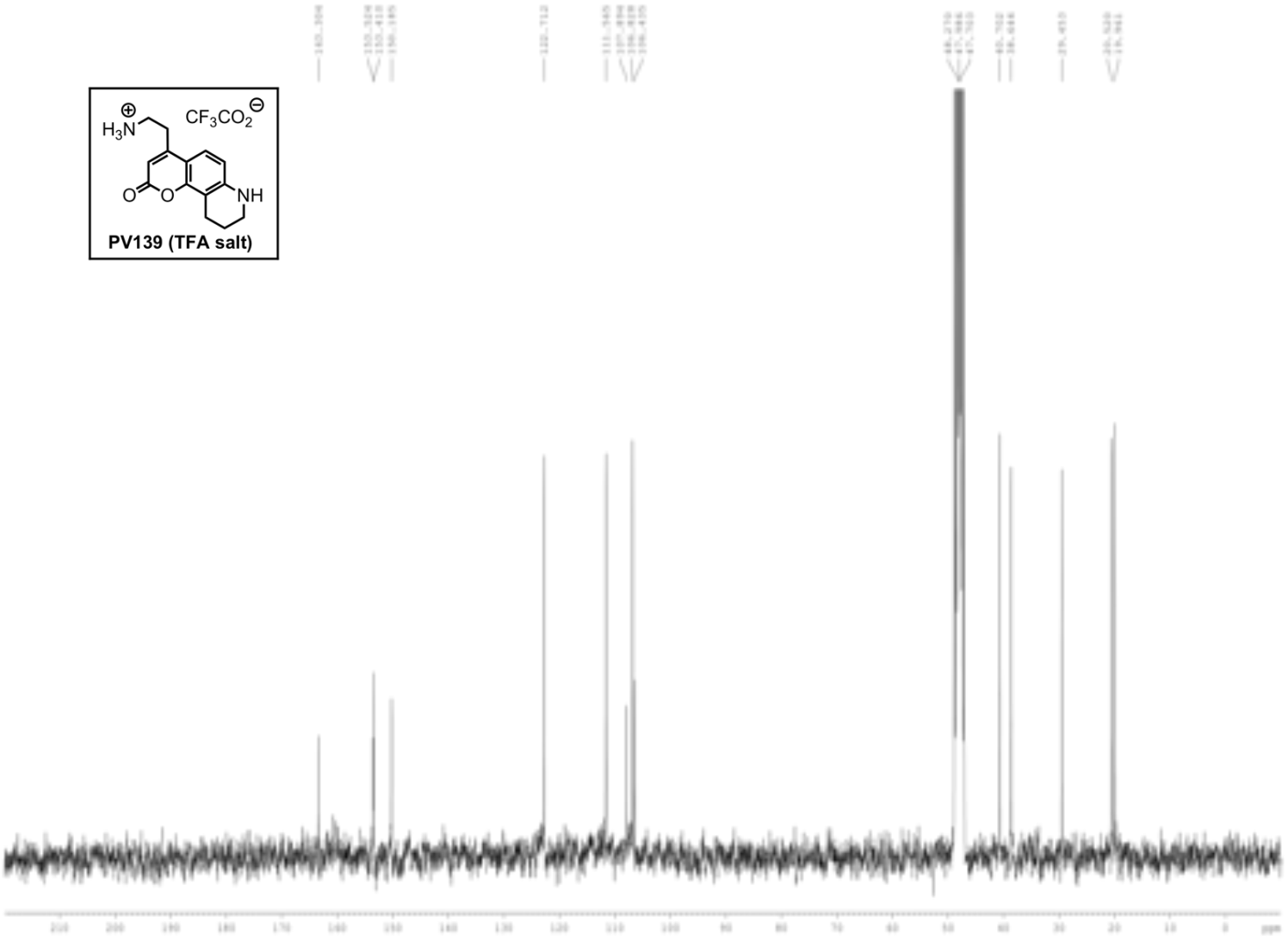



(BOC)HN
O
O
N
44
155.716
151.877
148.437
146.736
133.143
127.178
116.151
114.362
109.261
108.691
88.833
79.873
77.471
77.153
76.835
73.826
54.266
48.740
38.583
28.453
21.723
21.393
20.599
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


(BOC)HN
O
O
N
45
7.376
7.354
7.290
6.533
6.510
6.503
5.868
5.856
5.842
5.830
5.814
5.809
5.787
5.776
5.194
5.187
5.171
5.168
5.144
5.140
4.850
3.965
3.960
3.954
3.424
3.409
3.392
3.369
3.355
3.341
2.924
2.908
2.892
2.886
2.868
2.047
2.013
1.998
1.983
1.970
1.954
1.444
0.000
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 ppm
0.944
1.013
1.671
1.547
1.063
1.000
1.995
1.995
4.302
3.856
3.991


(BOC)HN
O
O
N
45
162.285
155.893
154.159
152.481
148.394
132.189
123.604
116.388
108.540
108.265
108.034
79.580
77.478
77.159
76.841
53.882
49.048
39.913
32.538
28.432
20.974
20.605
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


PV139 (TFA salt)


PV139 (TFA salt)