Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Copper-Catalyzed Synthesis of Quinoline Derivatives *via* Tandem Knoevenagel Condensation, Amination and Cyclization

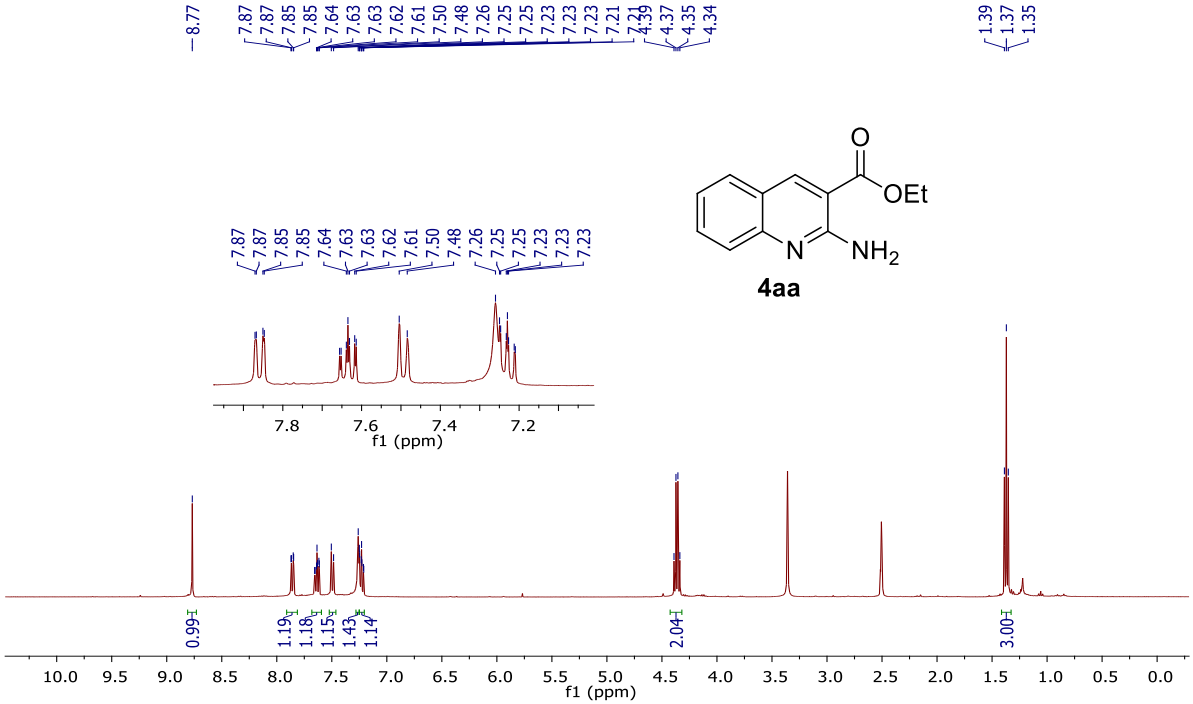
Shiv Dhiman, Hitesh Kumar Saini, Nitesh Kumar Nandwana, Dalip Kumar and Anil Kumar*

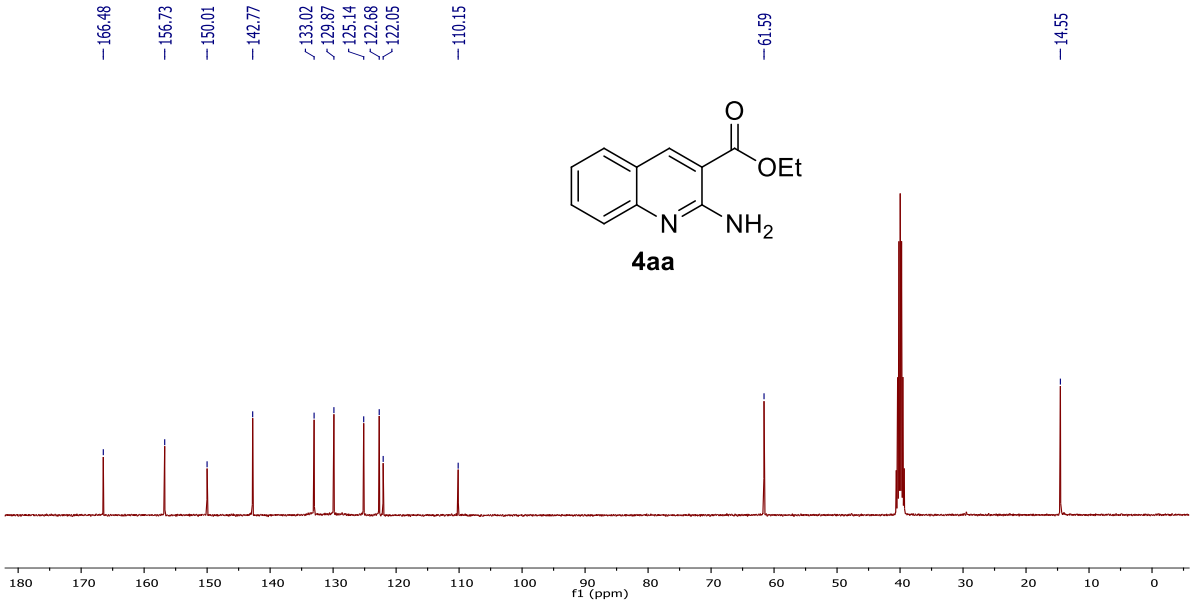
Department of Chemistry, Birla Institute of Technology and Science, Pilani 333 031, Rajasthan, India

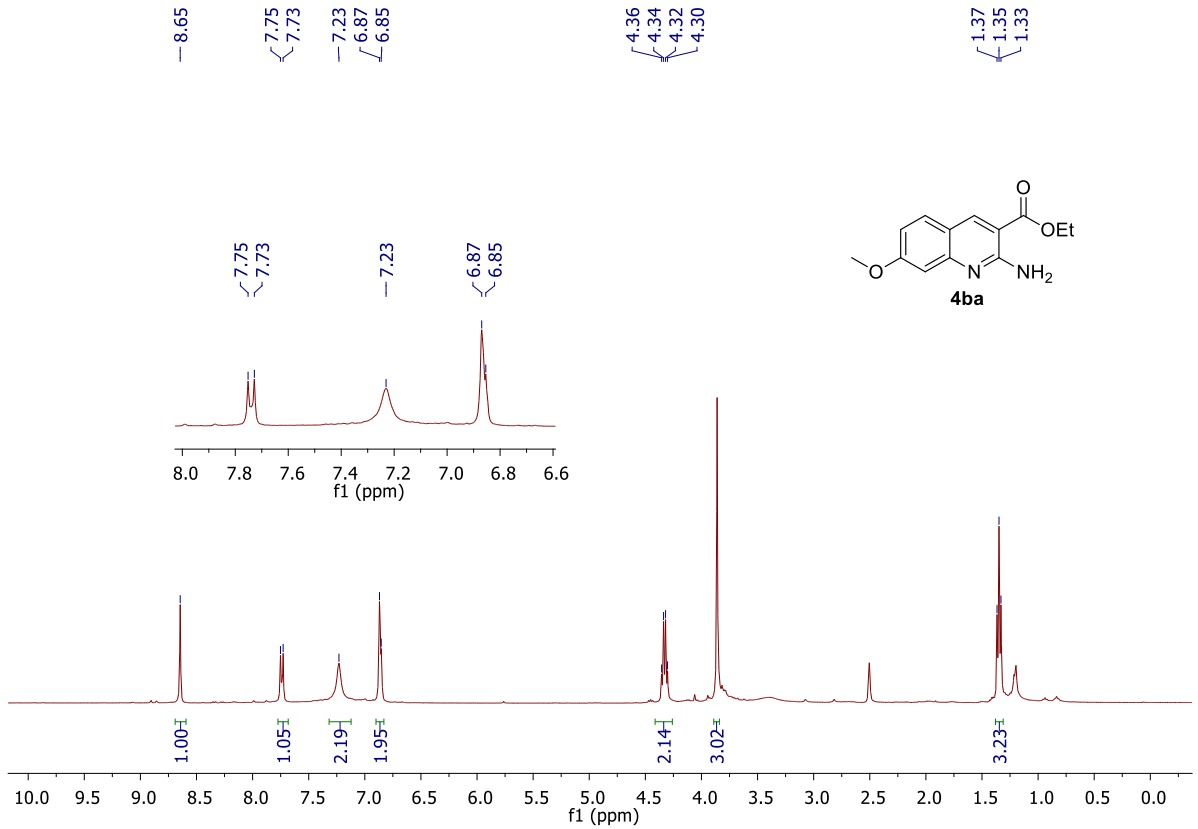
E-mail: anilkumar@pilani.bits-pilani.ac.in

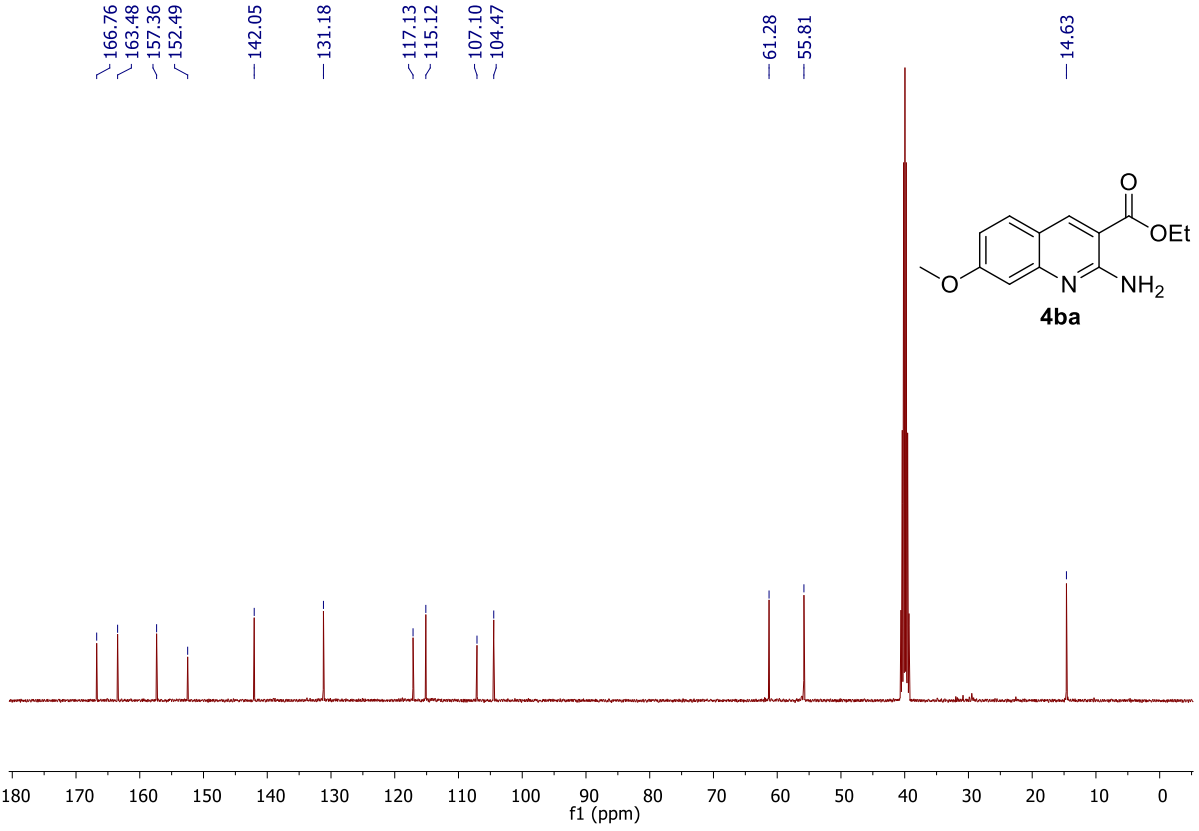
Contents

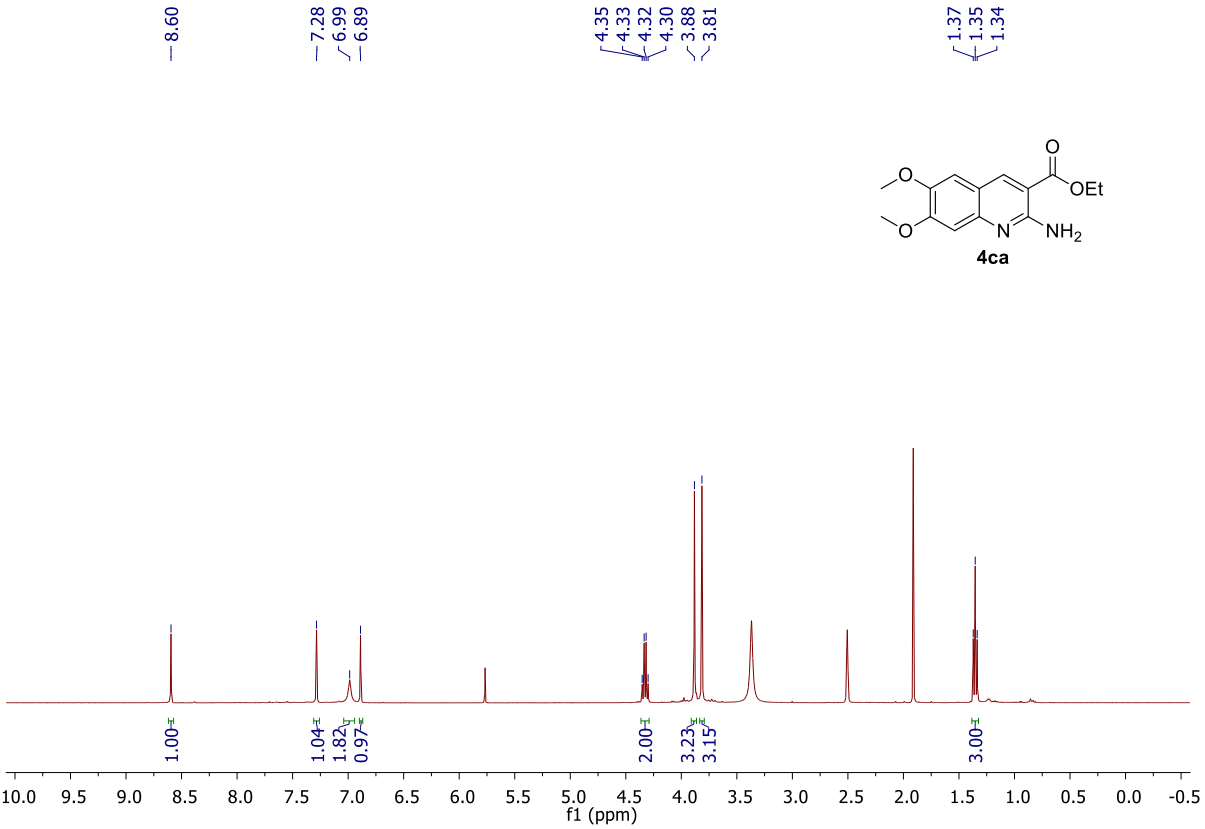
Copies of ^{1}H and ^{13}C NMR spectra of 4, 5, 6, 8, 9 & 10

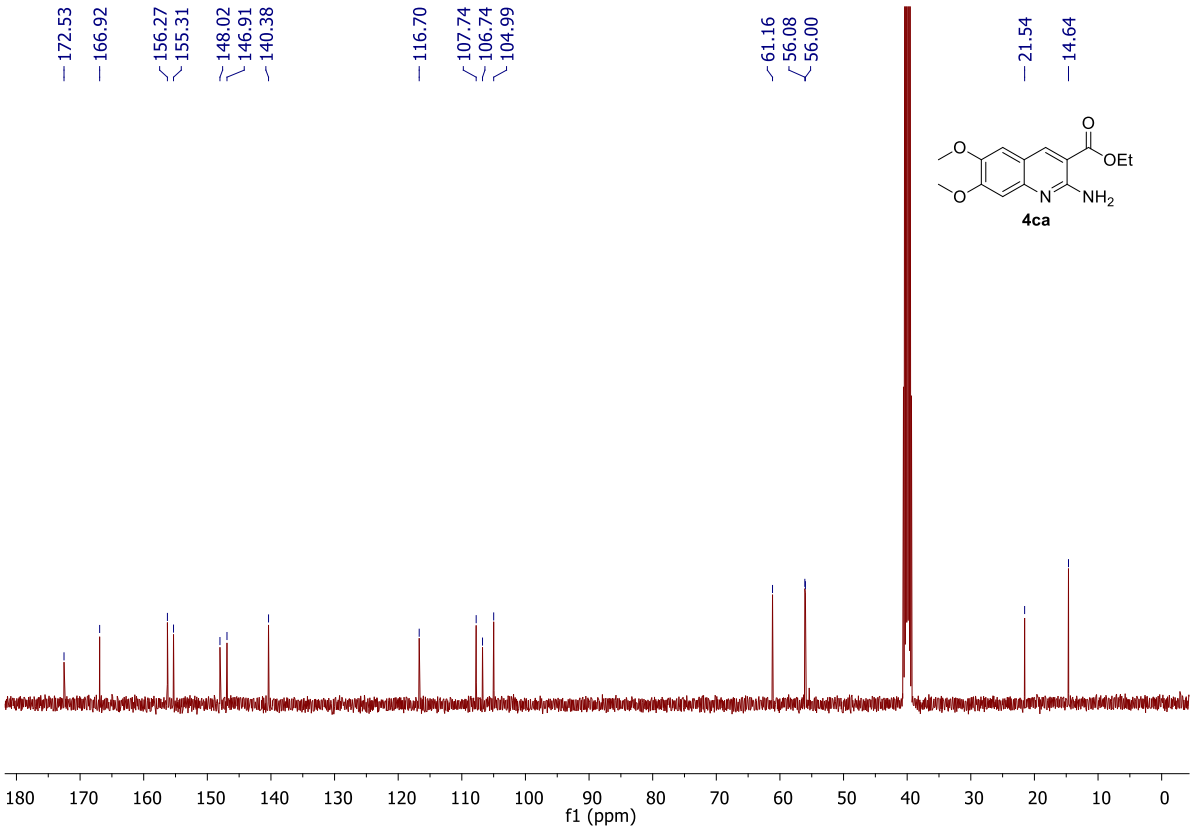


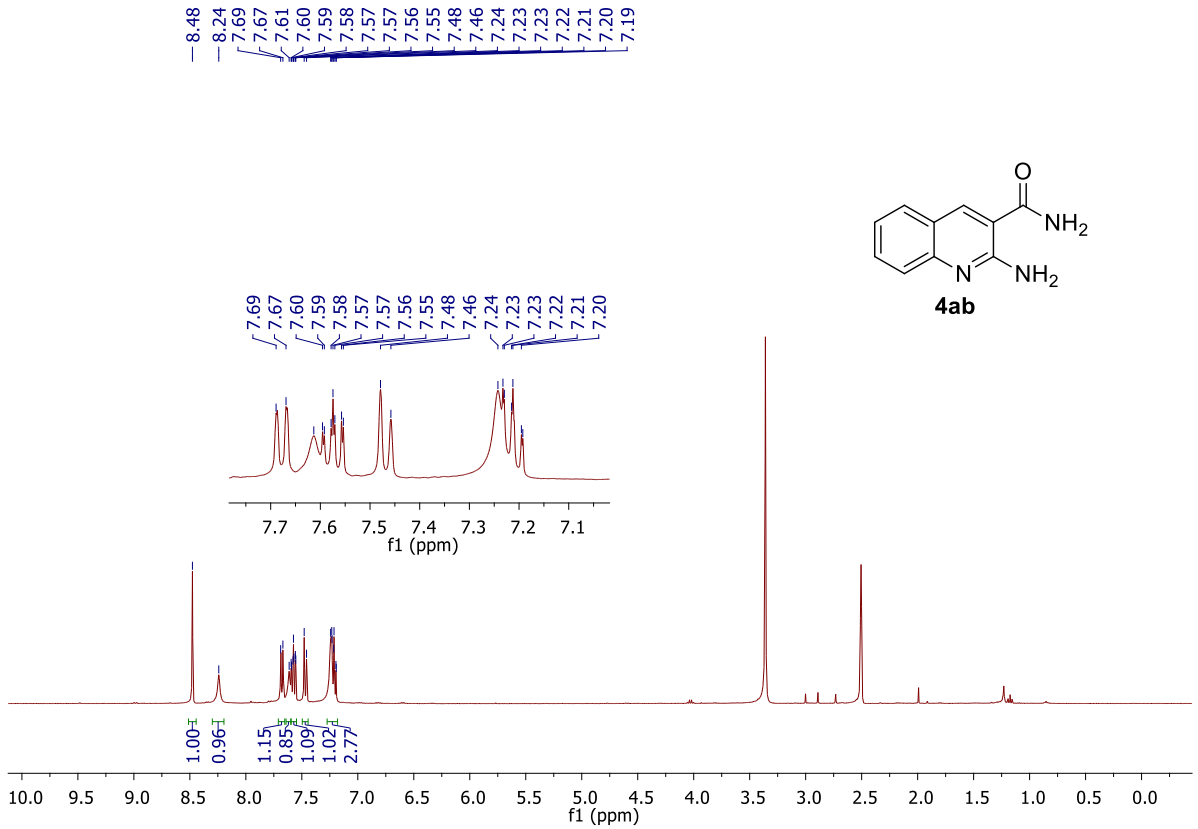


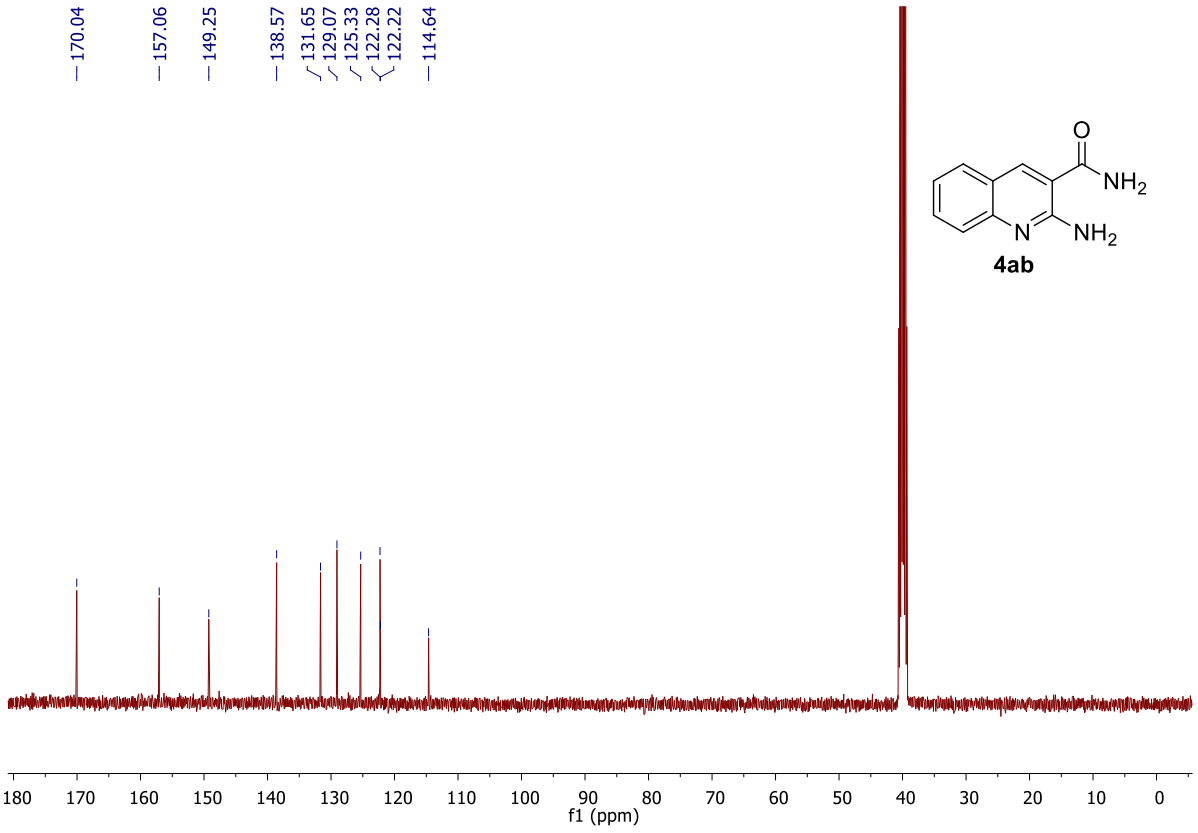

8.65
7.75
7.73
7.23
6.87
6.85
4.36
4.34
4.32
4.30
1.37
1.35
1.33
O
OEt
O
N
NH2
4ba
f1 (ppm)
1.00
1.05
2.19
1.95
2.14
3.02
3.23
f1 (ppm)

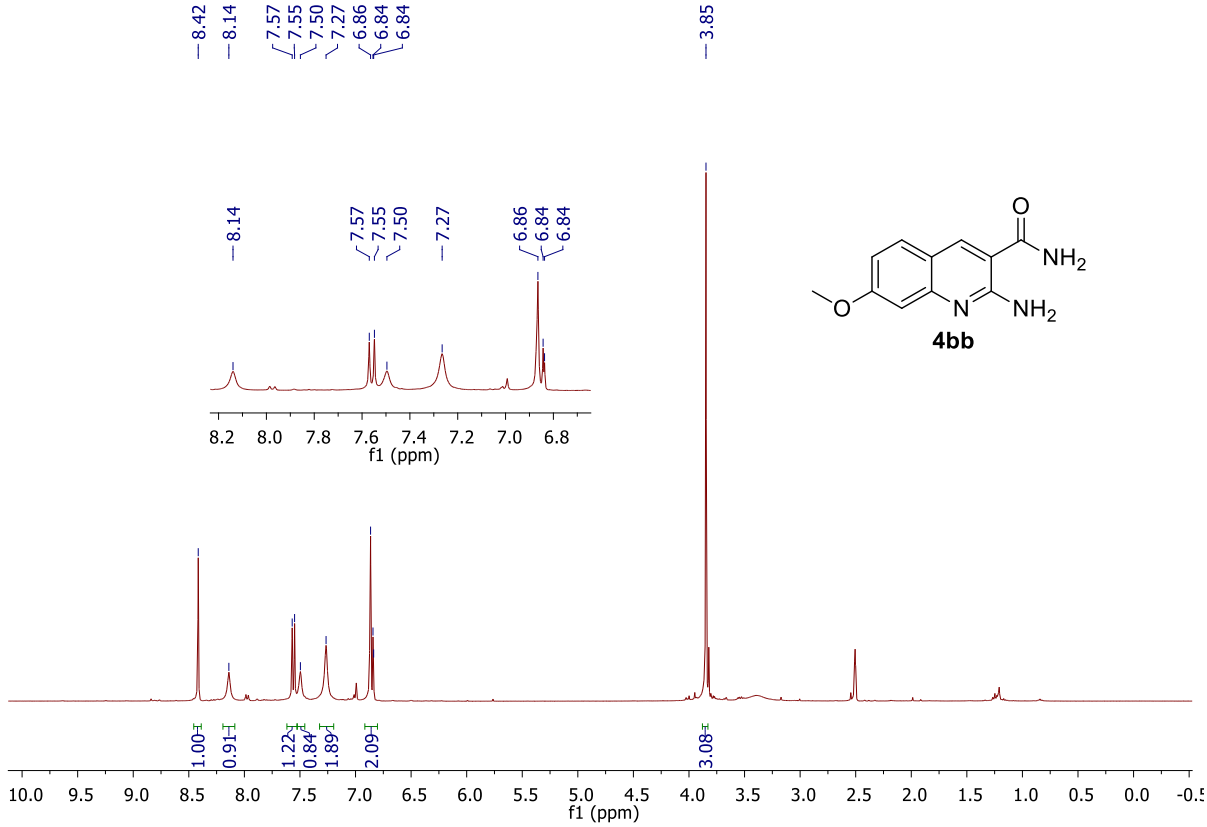

166.76
163.48
157.36
152.49
142.05
131.18
117.13
115.12
107.10
104.47
61.28
55.81
14.63
O
OEt
O
N
NH2
4ba
f1 (ppm)

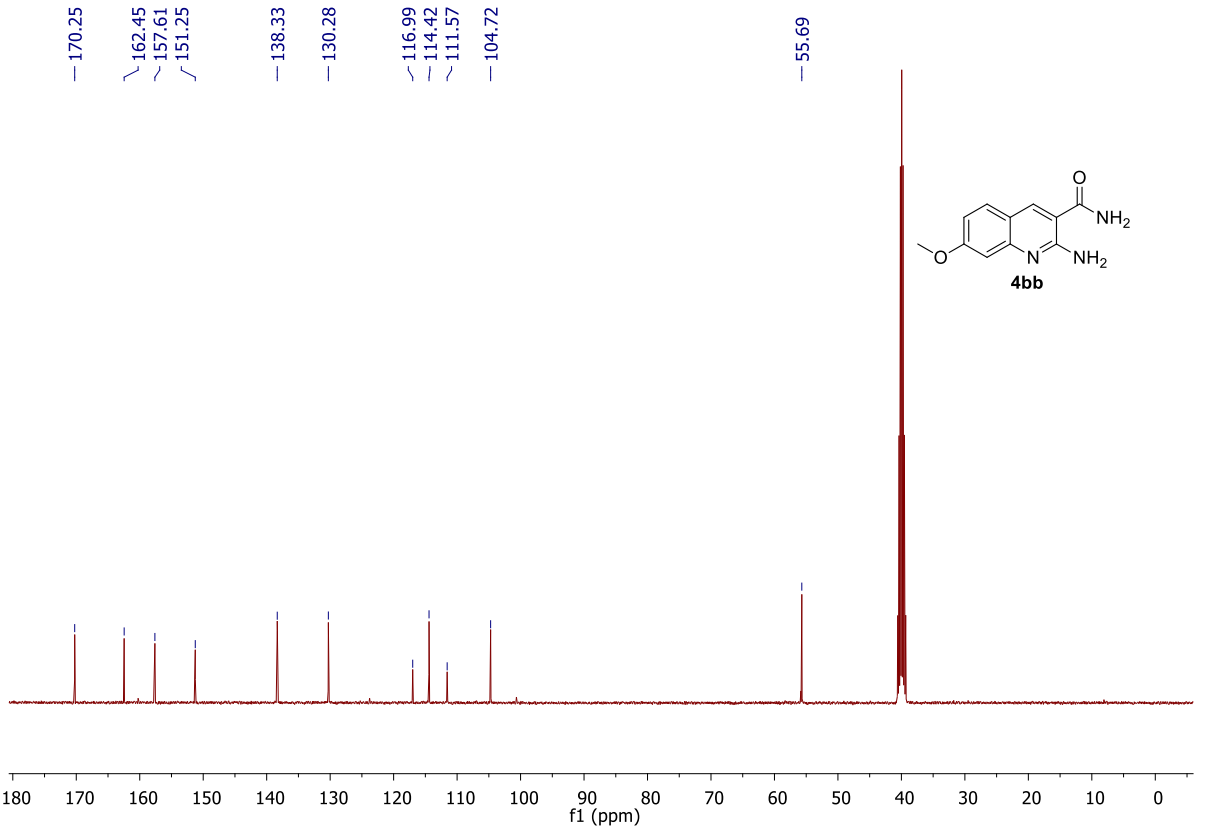

8.60
7.28
6.99
6.89
4.35
4.33
4.32
4.30
3.88
3.81
1.37
1.35
1.34
O
OEt
O
O
N
NH2
4ca
1.00
1.04
1.82
0.97
2.00
3.23
3.15
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

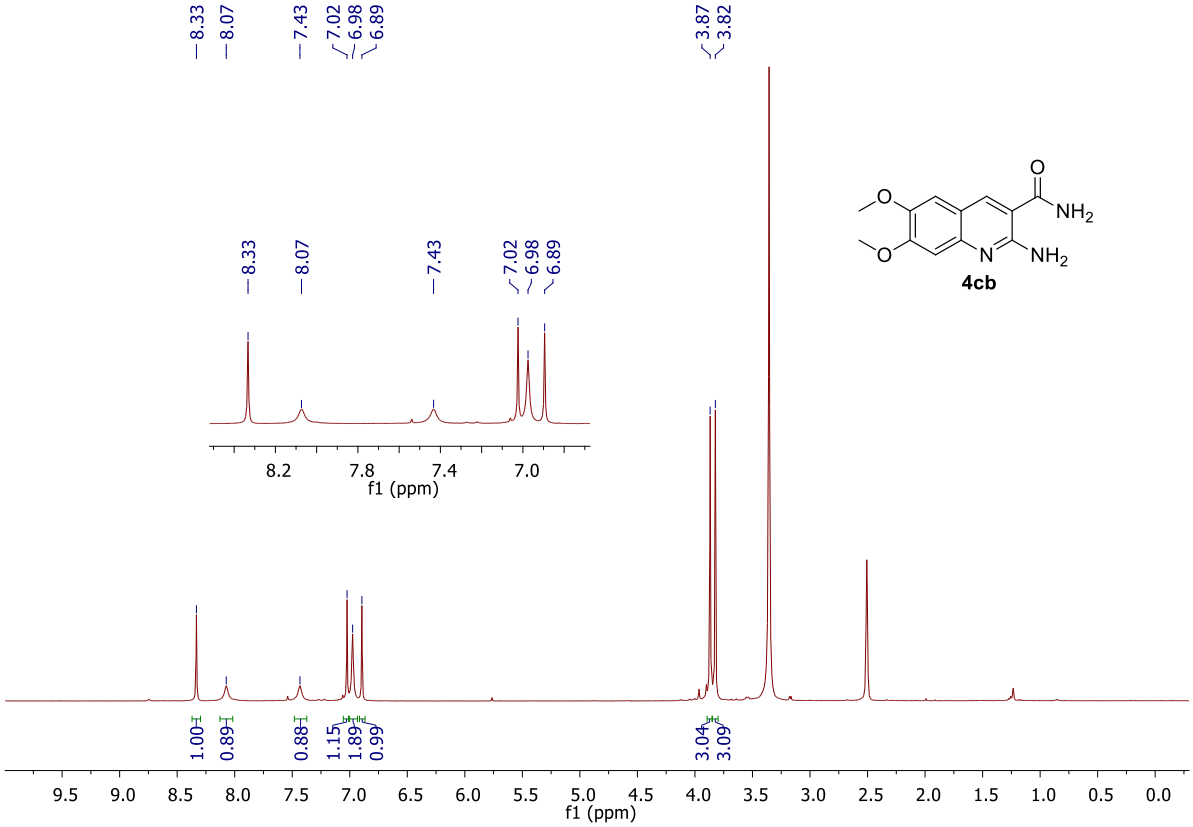

172.53
166.92
156.27
155.31
148.02
146.91
140.38
116.70
107.74
106.74
104.99
61.16
56.08
56.00
21.54
14.64
O
OEt
O
O
N
NH2
4ca
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

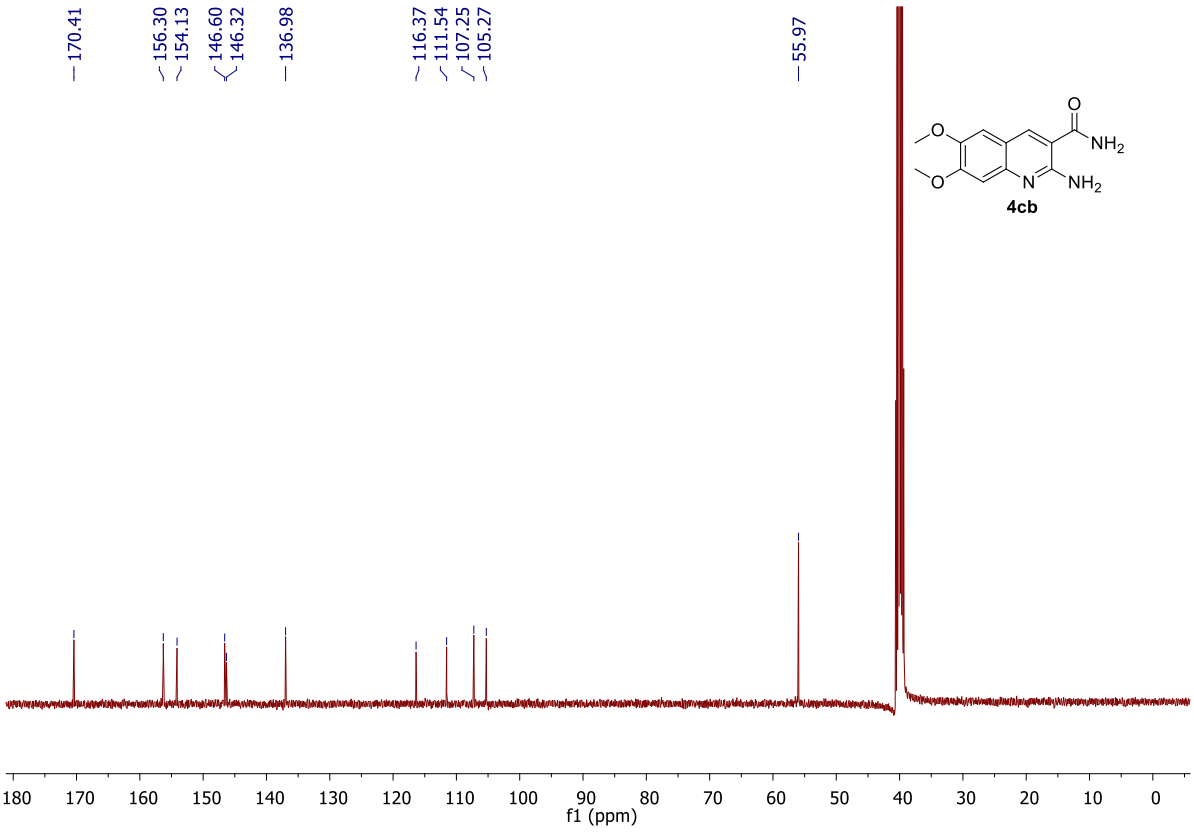

8.48
8.24
7.69
7.67
7.61
7.60
7.59
7.58
7.57
7.57
7.56
7.55
7.48
7.46
7.24
7.23
7.23
7.22
7.21
7.20
7.19
O
NH2
N
NH2
4ab
7.69
7.67
7.60
7.59
7.58
7.57
7.57
7.56
7.55
7.48
7.46
7.24
7.23
7.23
7.22
7.21
7.20
7.7 7.6 7.5 7.4 7.3 7.2 7.1
f1 (ppm)
1.00
0.96
1.15
0.85
1.09
1.02
2.77
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.04
157.06
149.25
138.57
131.65
129.07
125.33
122.28
122.22
114.64
O
NH2
N
NH2
4ab
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

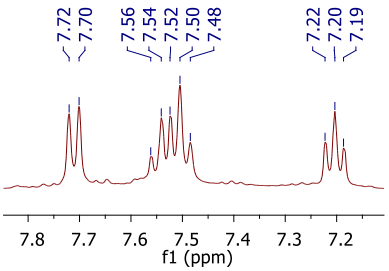

8.42
8.14
7.57
7.55
7.50
7.27
6.86
6.84
6.84
3.85
1.00
0.91
1.22
0.84
1.89
2.09
3.08
f1 (ppm)
O
NH2
N
NH2
O
4bb

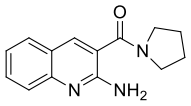

170.25
162.45
157.61
151.25
138.33
130.28
116.99
114.42
111.57
104.72
55.69
f1 (ppm)
O
NH2
N
NH2
O
4bb


8.33
8.07
7.43
7.02
6.98
6.89
3.87
3.82
1.00
0.89
0.88
1.15
1.89
0.99
3.04
3.09
f1 (ppm)
4cb

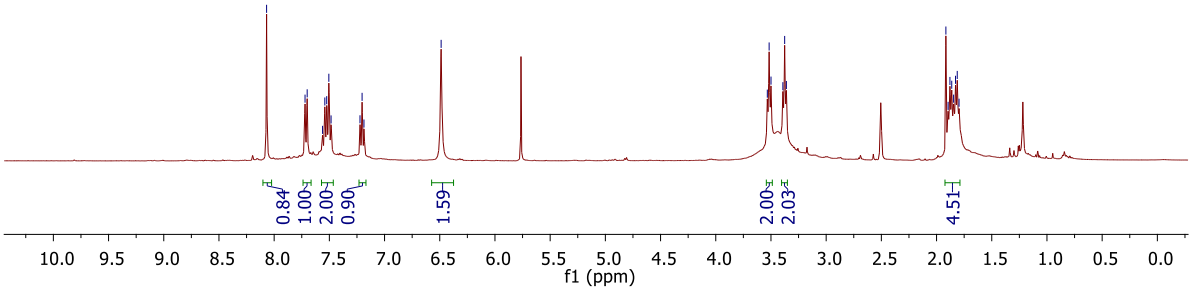

170.41
156.30
154.13
146.60
146.32
136.98
116.37
111.54
107.25
105.27
55.97
f1 (ppm)
4cb

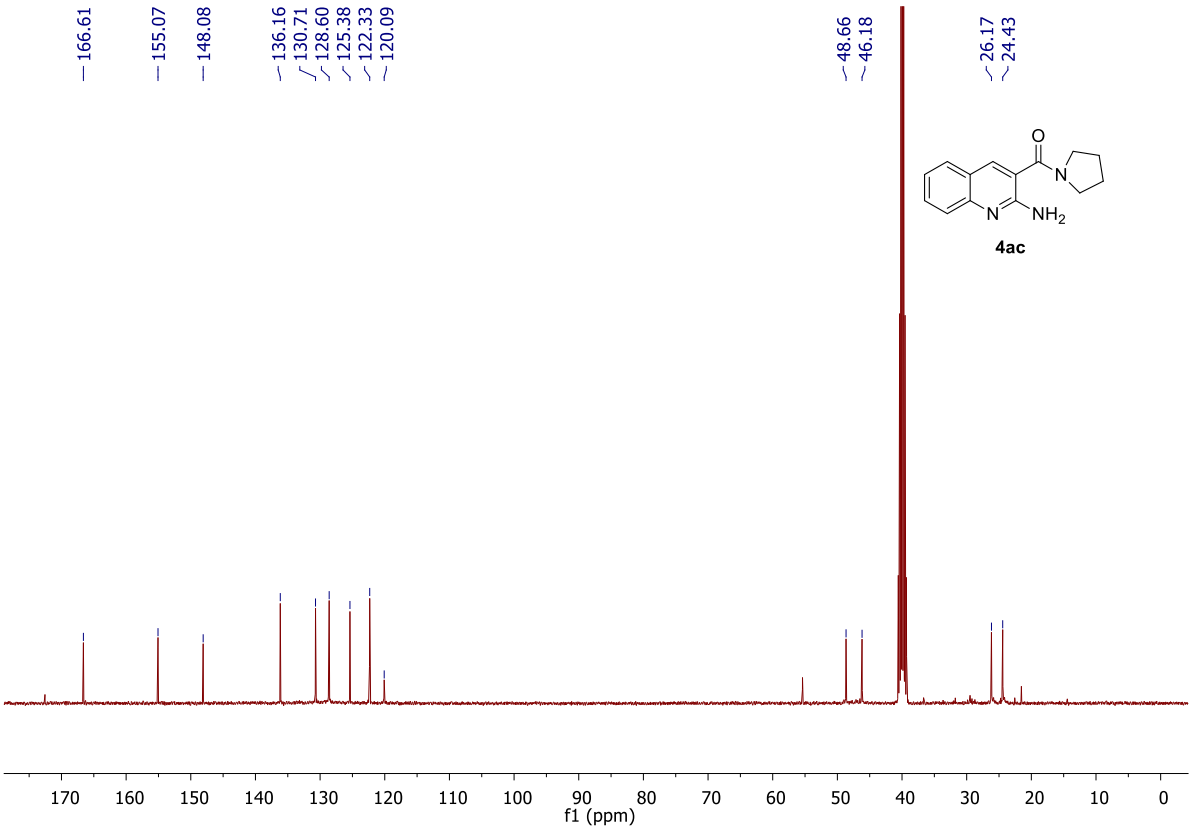

8.07
7.72
7.70
7.56
7.54
7.52
7.50
7.48
7.22
7.20
7.19
6.49
3.53
3.52
3.50
3.39
3.38
3.36
1.92
1.90
1.88
1.86
1.85
1.83
1.81
1.80


7.72
7.70
7.56
7.54
7.52
7.50
7.48
7.22
7.20
7.19
7.8
7.7
7.6
7.5
7.4
7.3
7.2
f1 (ppm)

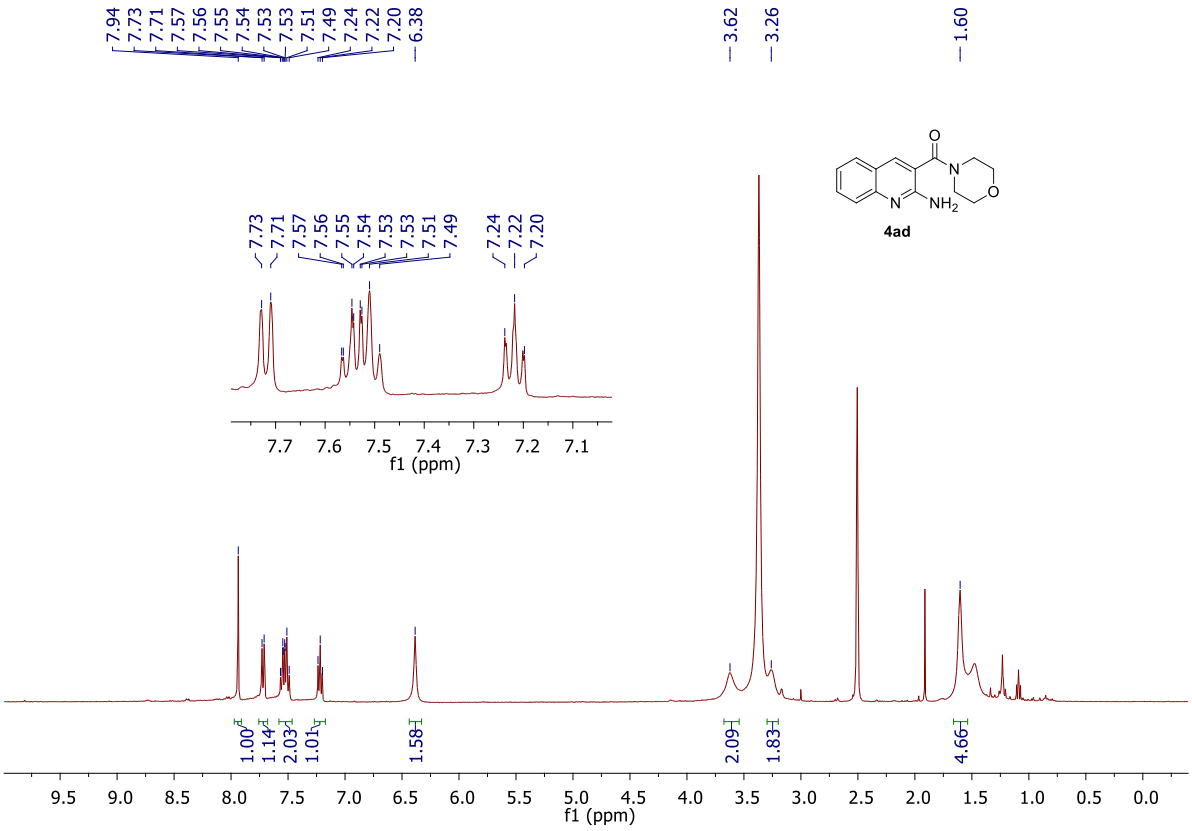

O
N
N
NH2

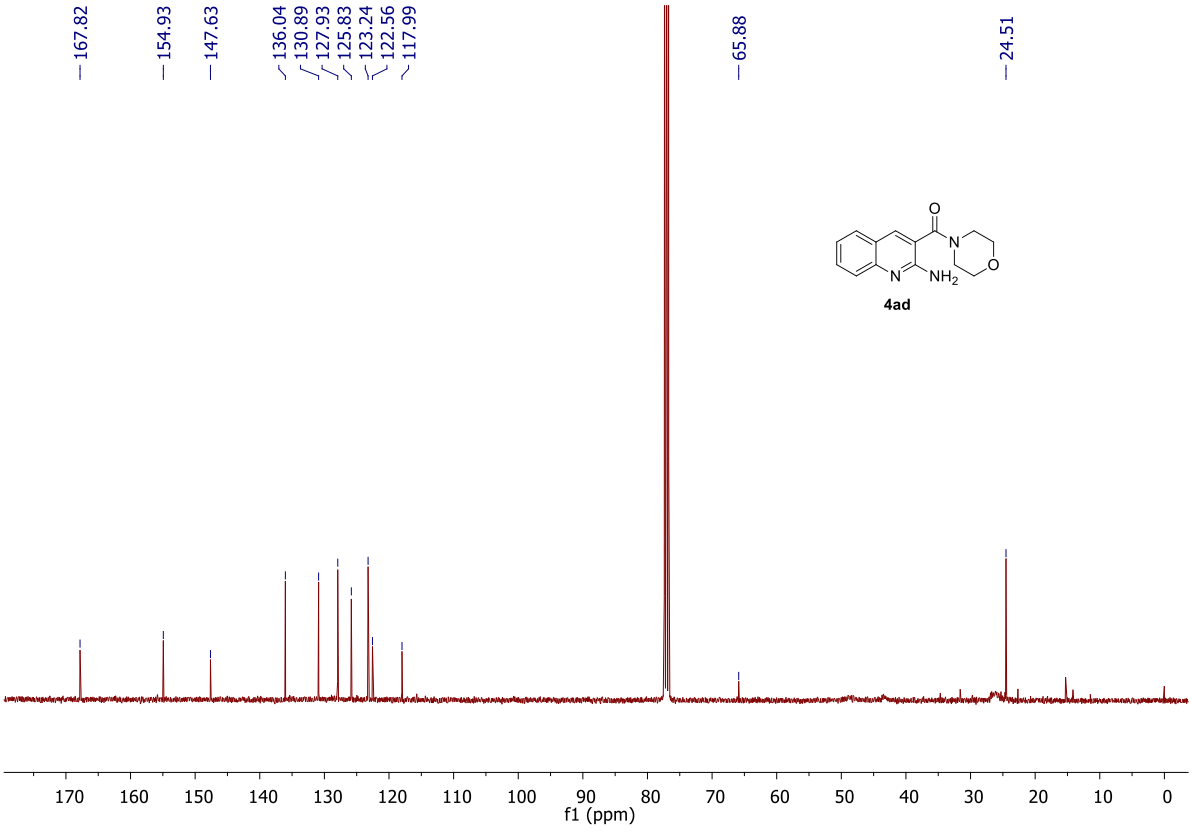
4ac

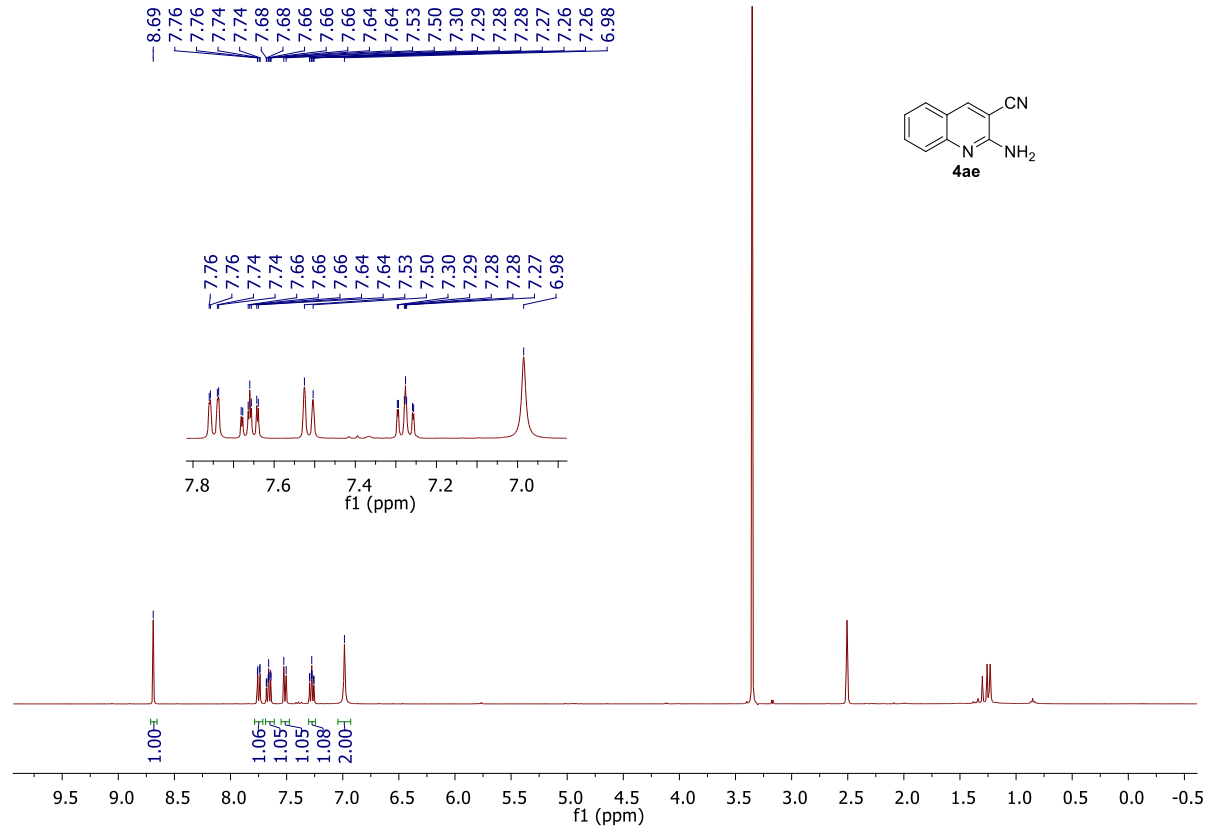

0.84
1.00
2.00
0.90
1.59
2.00
2.03
4.51
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

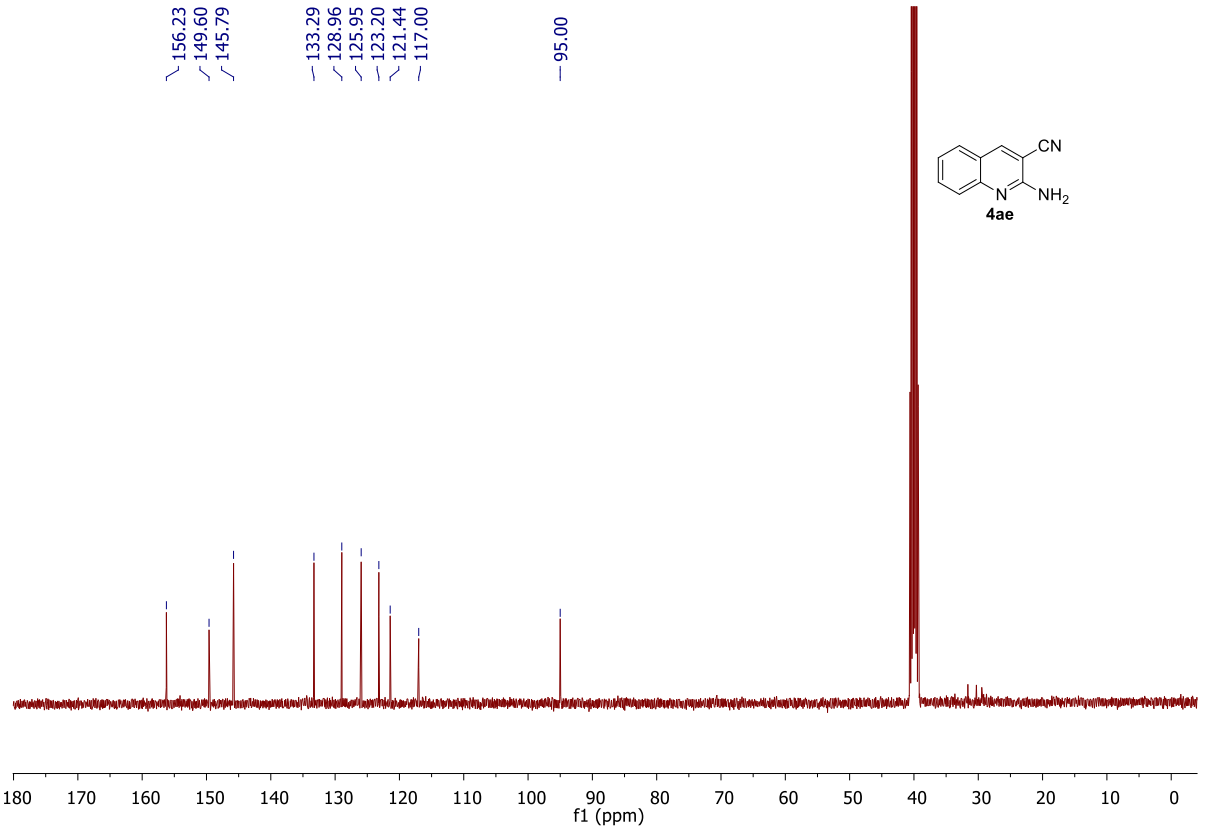

166.61
155.07
148.08
136.16
130.71
128.60
125.38
122.33
120.09
48.66
46.18
26.17
24.43
O
N
N
NH2
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

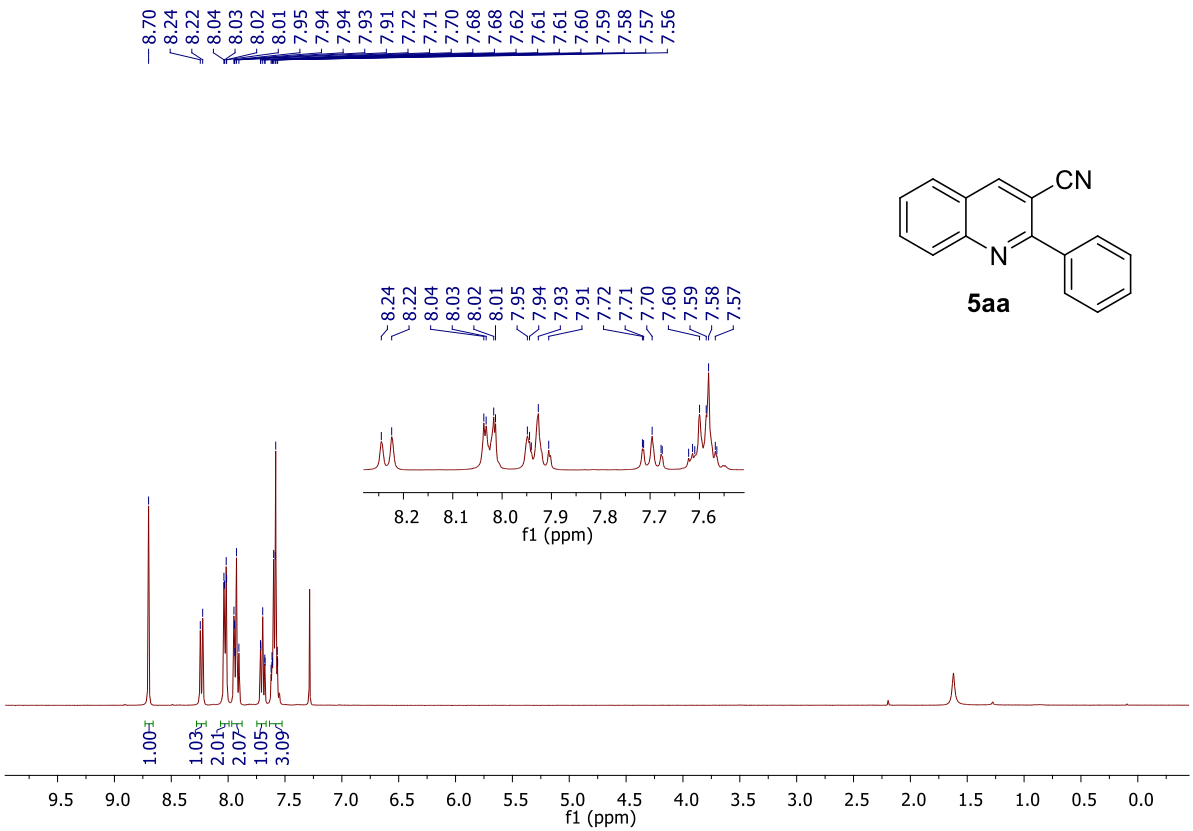
4ac

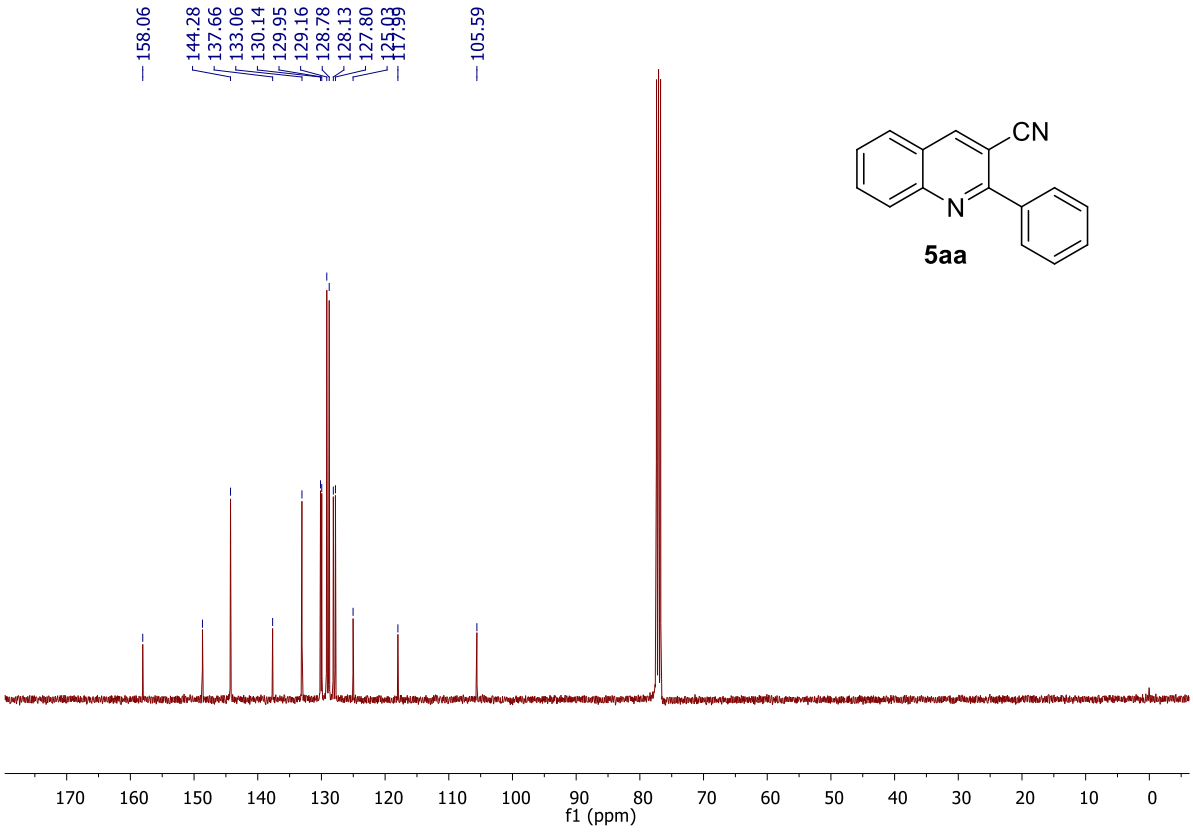

7.94
7.73
7.71
7.57
7.56
7.55
7.54
7.53
7.53
7.51
7.49
7.24
7.22
7.20
6.38
3.62
3.26
1.60
4ad
f1 (ppm)
1.00
1.14
2.03
1.01
1.58
2.09
1.83
4.66

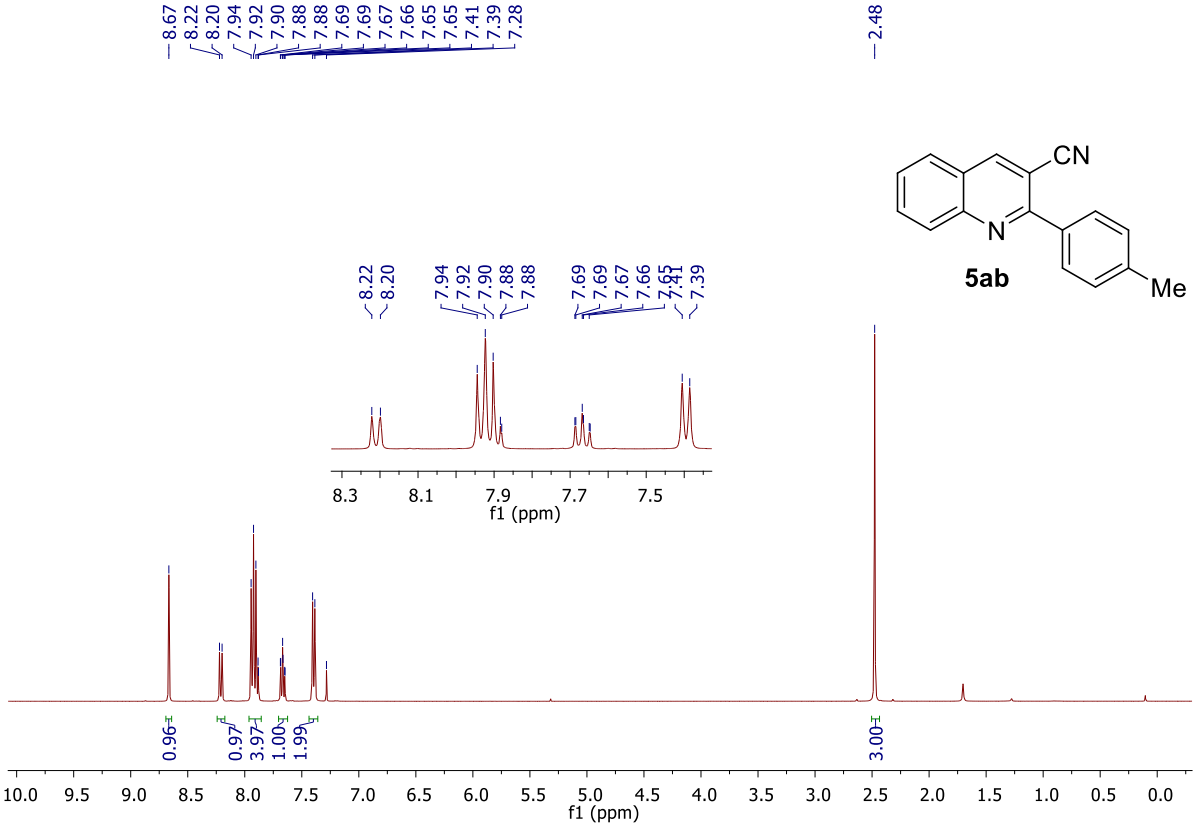

167.82
154.93
147.63
136.04
130.89
127.93
125.83
123.24
122.56
117.99
65.88
24.51
4ad
f1 (ppm)

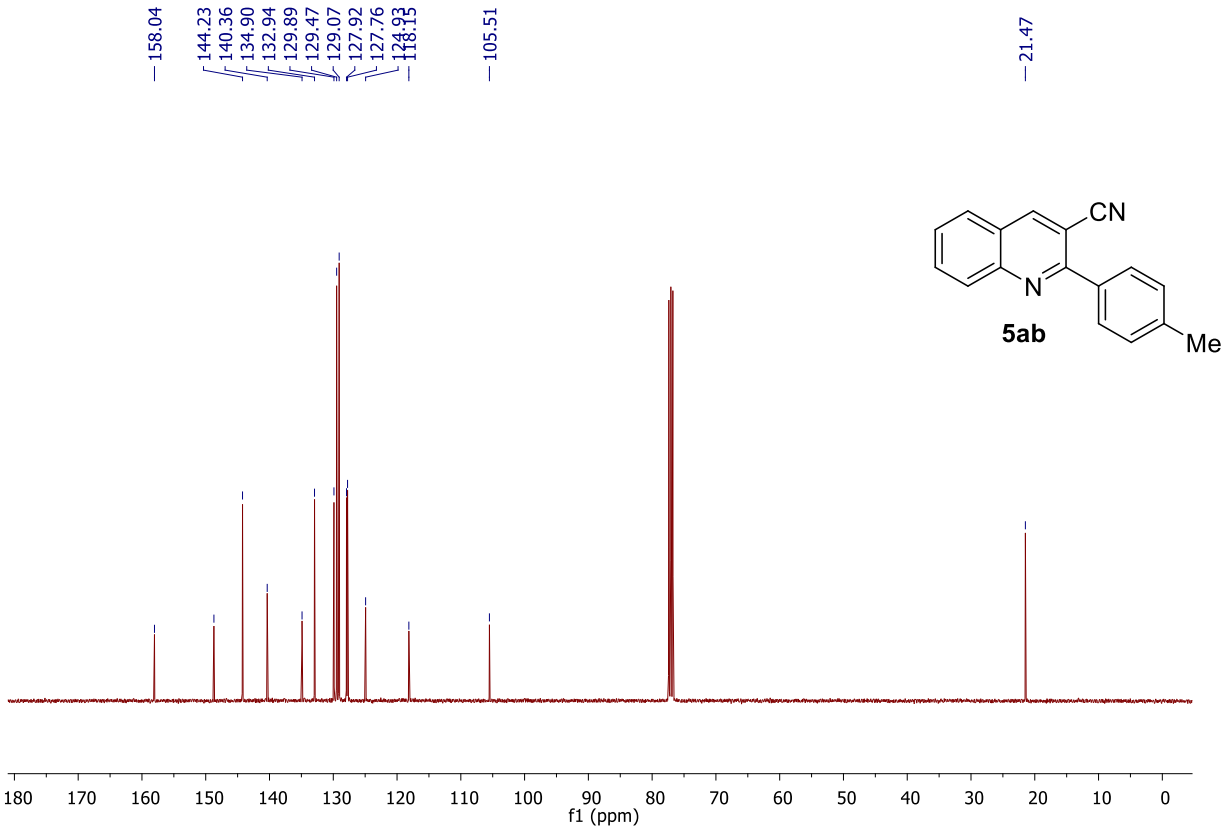

8.69
7.76
7.76
7.74
7.74
7.68
7.68
7.66
7.66
7.66
7.64
7.64
7.53
7.50
7.30
7.29
7.28
7.28
7.27
7.26
7.26
6.98
CN
NH2
N
4ae
7.8
7.6
7.4
7.2
7.0
f1 (ppm)
1.00
1.06
1.05
1.05
1.08
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

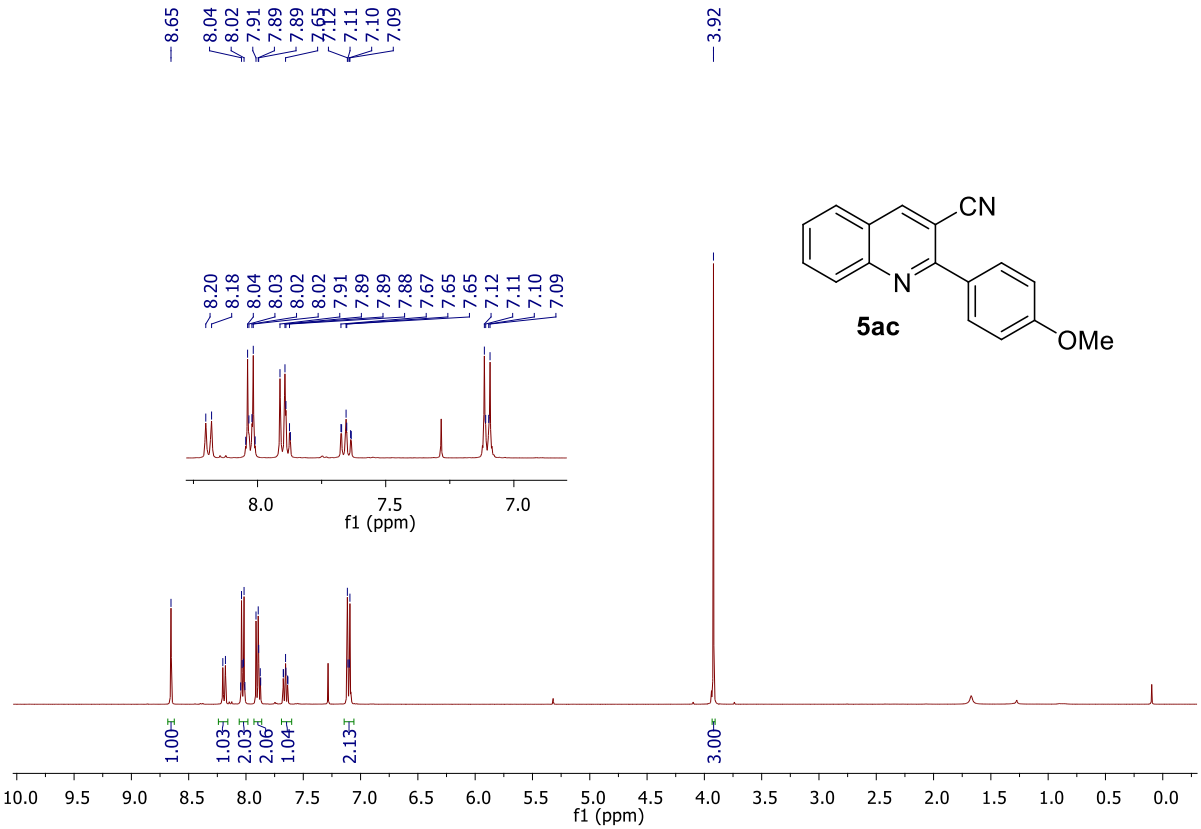

156.23
149.60
145.79
133.29
128.96
125.95
123.20
121.44
117.00
95.00
CN
NH2
N
4ae
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

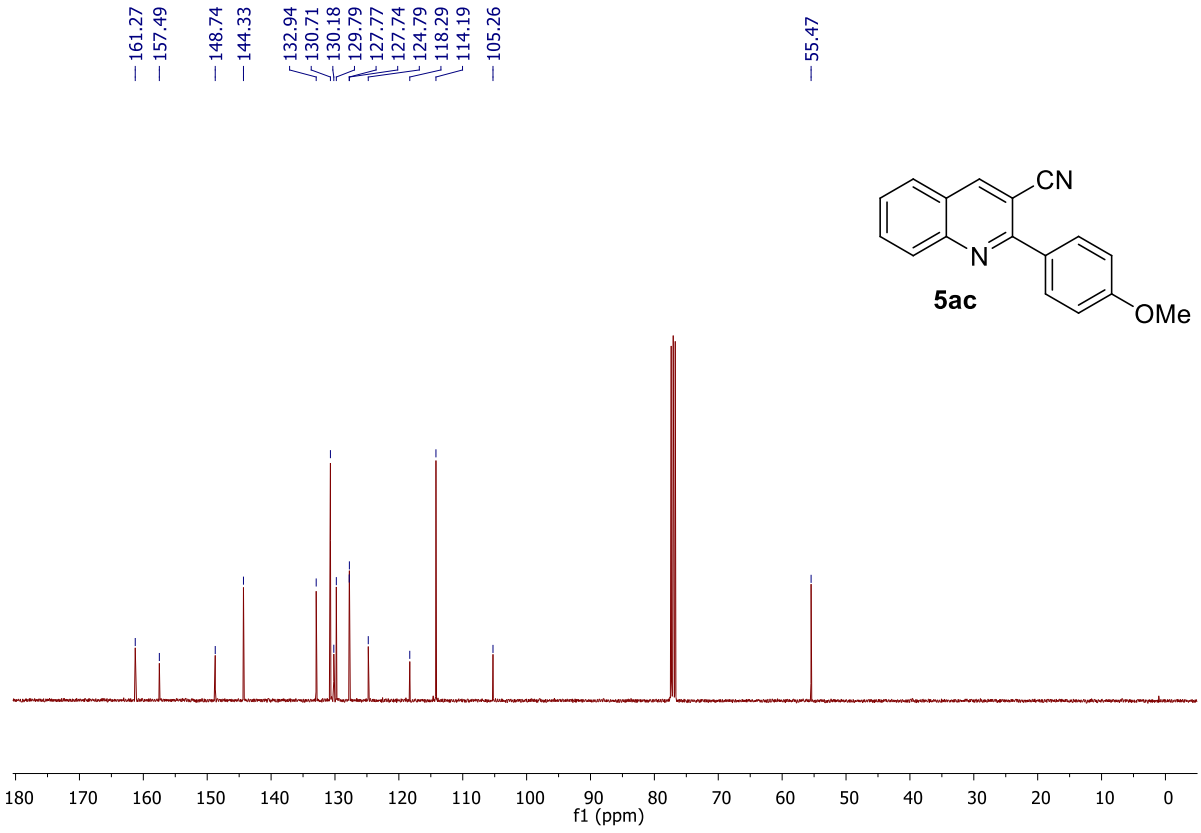

8.70
8.24
8.22
8.04
8.03
8.02
8.01
7.95
7.94
7.94
7.93
7.91
7.72
7.71
7.70
7.68
7.68
7.62
7.61
7.61
7.60
7.59
7.58
7.57
7.56
CN
N
5aa
8.24
8.22
8.04
8.03
8.02
8.01
7.95
7.94
7.93
7.91
7.72
7.71
7.70
7.60
7.59
7.58
7.57
8.2 8.1 8.0 7.9 7.8 7.7 7.6
f1 (ppm)
1.00
1.03
2.01
2.07
1.05
3.09
9.5 9.0 8.5 8.0 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
f1 (ppm)

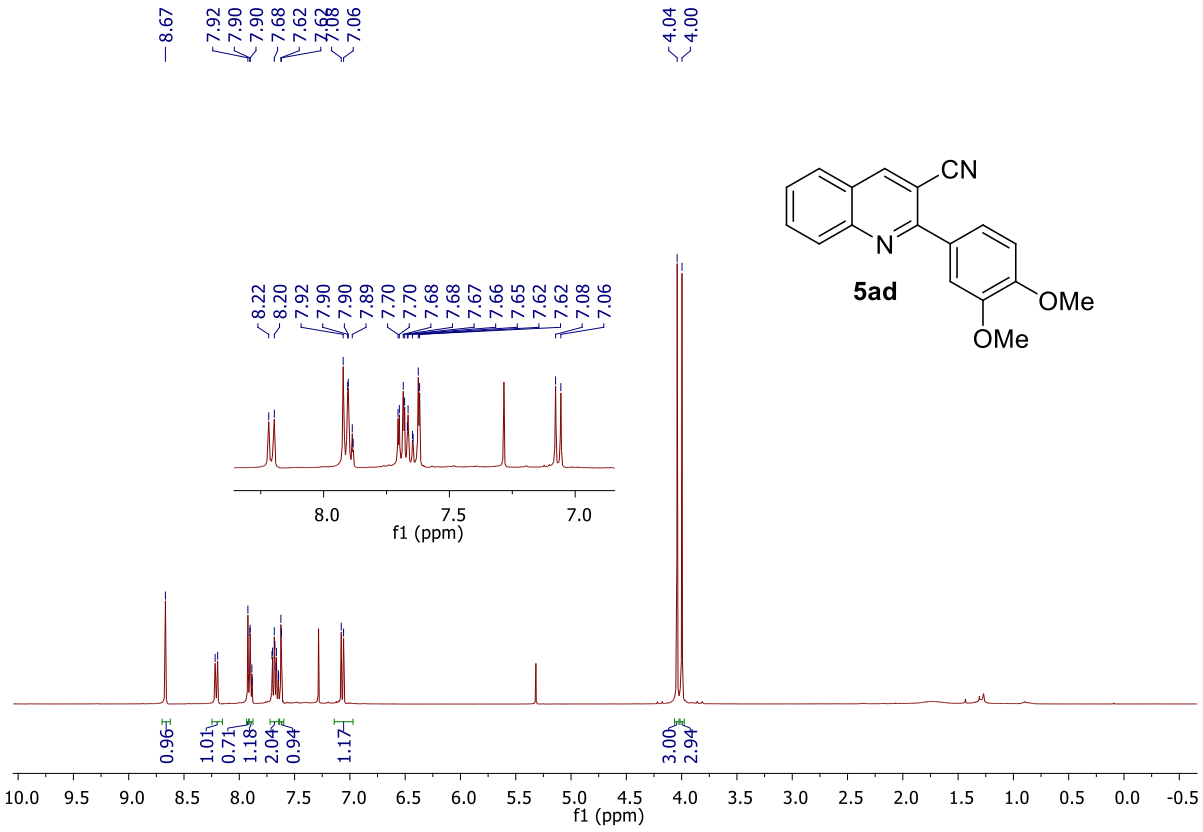

158.06
144.28
137.66
133.06
130.14
129.95
129.16
128.78
128.13
127.80
125.03
117.99
105.59
CN
N
5aa
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

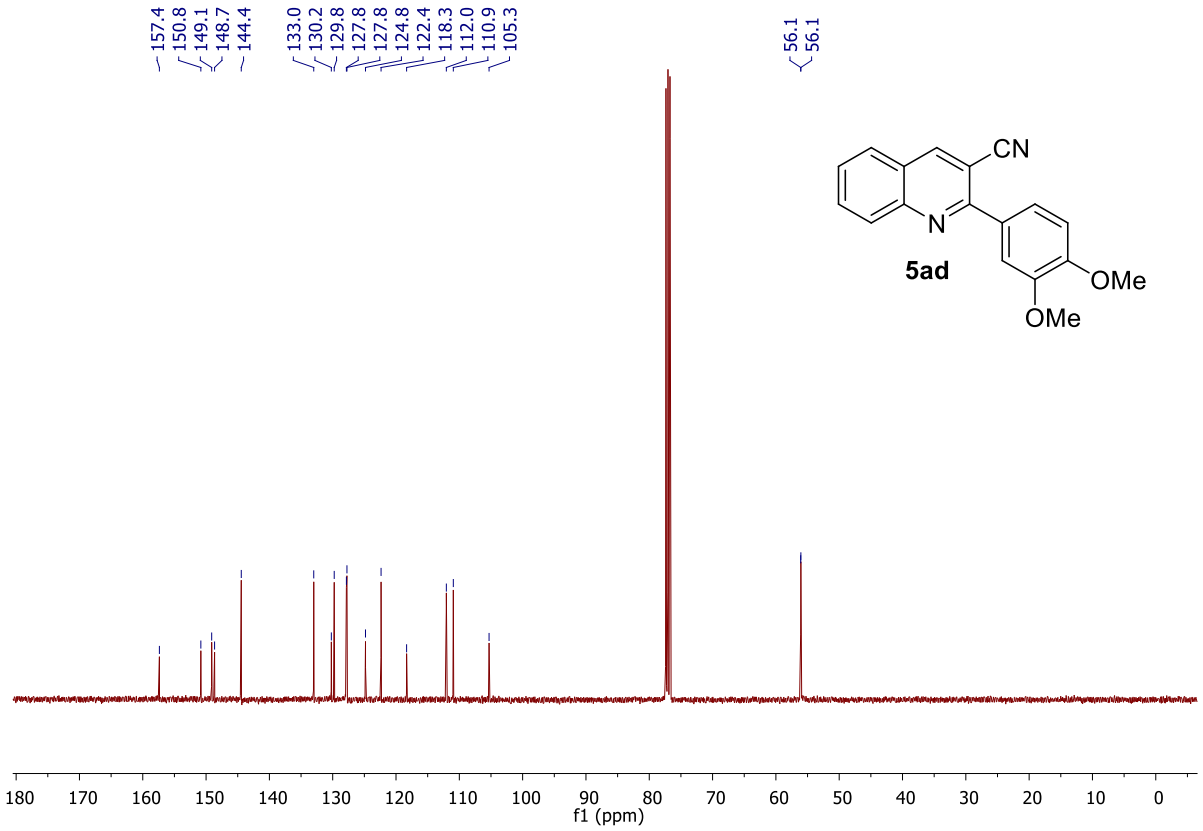

8.67
8.22
8.20
7.94
7.92
7.90
7.88
7.88
7.69
7.67
7.66
7.65
7.65
7.41
7.39
7.28
2.48
CN
N
5ab
Me
8.22
8.20
7.94
7.92
7.90
7.88
7.88
7.69
7.69
7.67
7.66
7.65
7.41
7.39
8.3
8.1
7.9
7.7
7.5
f1 (ppm)
0.96
0.97
3.97
1.00
1.99
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

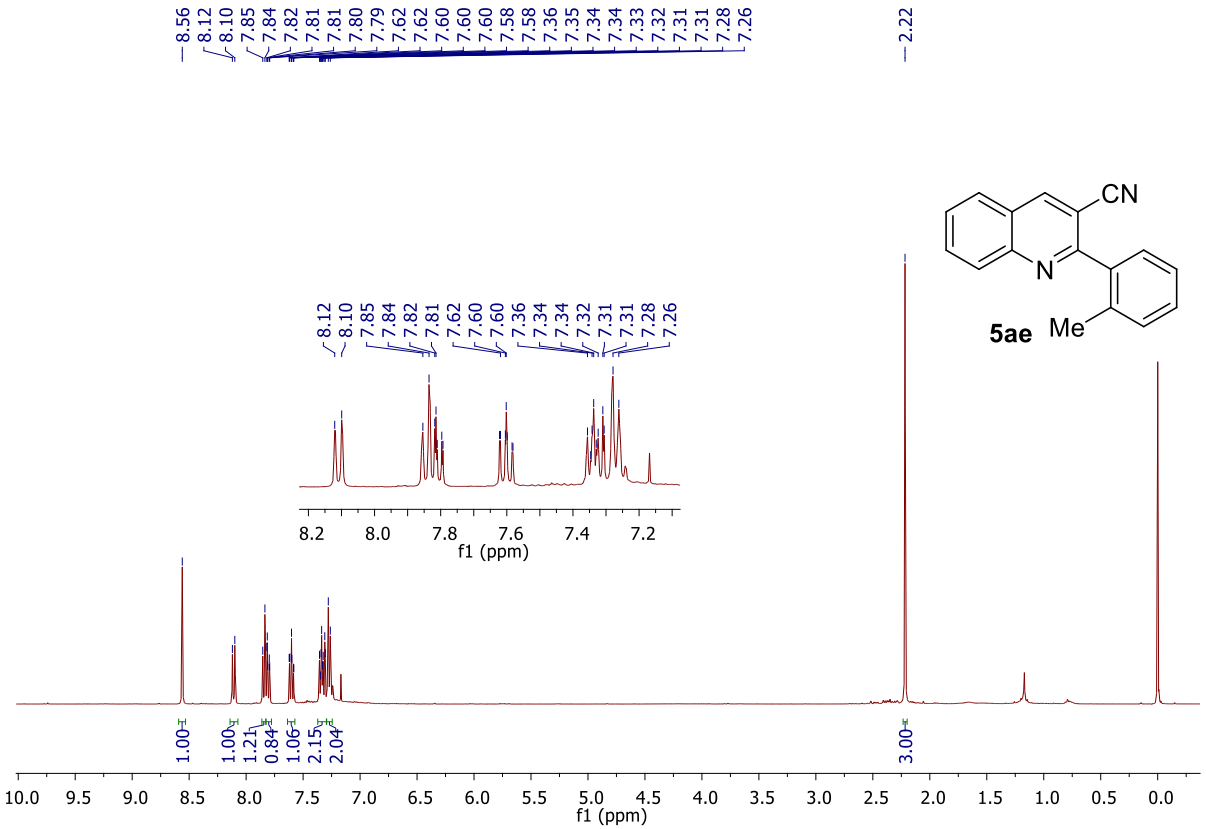

158.04
144.23
140.36
134.90
132.94
129.89
129.47
129.07
127.92
127.76
124.93
118.15
105.51
21.47
CN
N
5ab
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

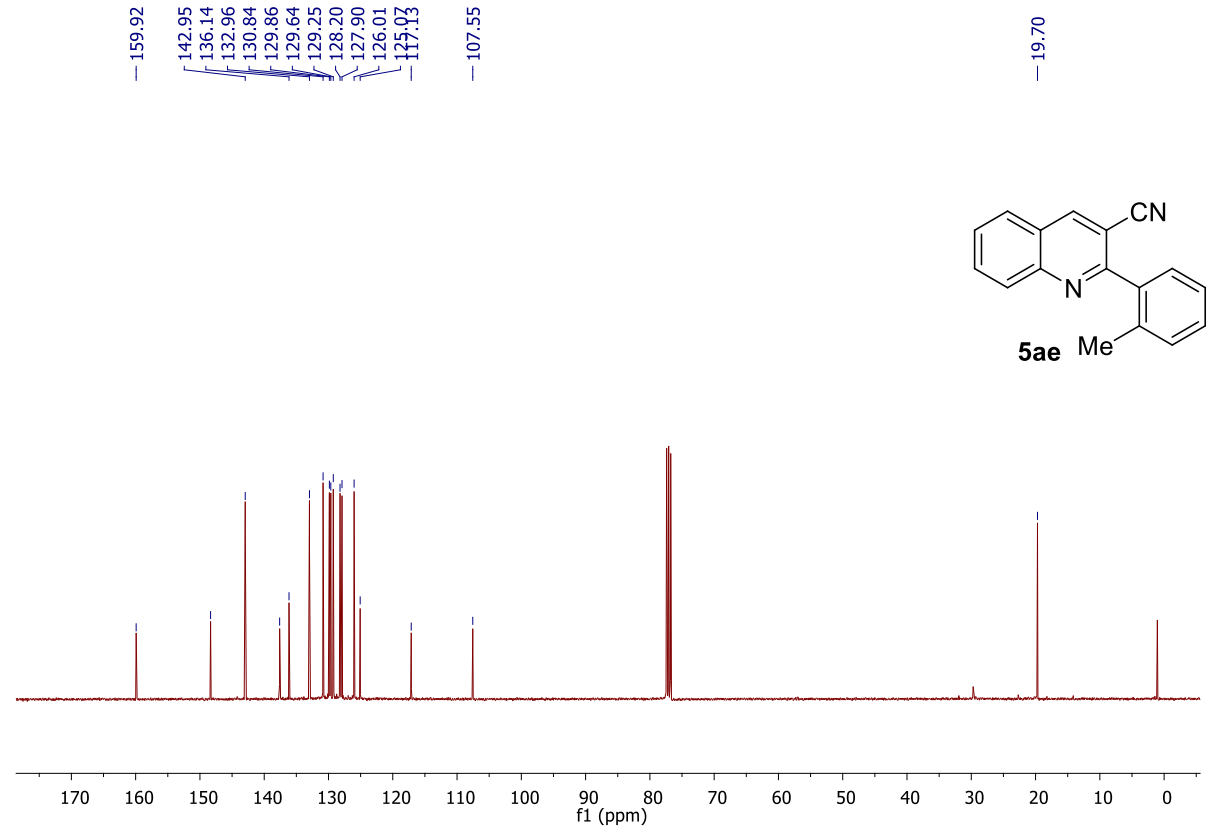

8.65
8.04
8.02
7.91
7.89
7.89
7.65
7.12
7.11
7.10
7.09
3.92
8.20
8.18
8.04
8.03
8.02
8.02
7.91
7.89
7.89
7.88
7.67
7.65
7.65
7.12
7.11
7.10
7.09
8.0
7.5
7.0
f1 (ppm)
CN
N
5ac
OMe
1.00
1.03
2.03
2.06
1.04
2.13
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

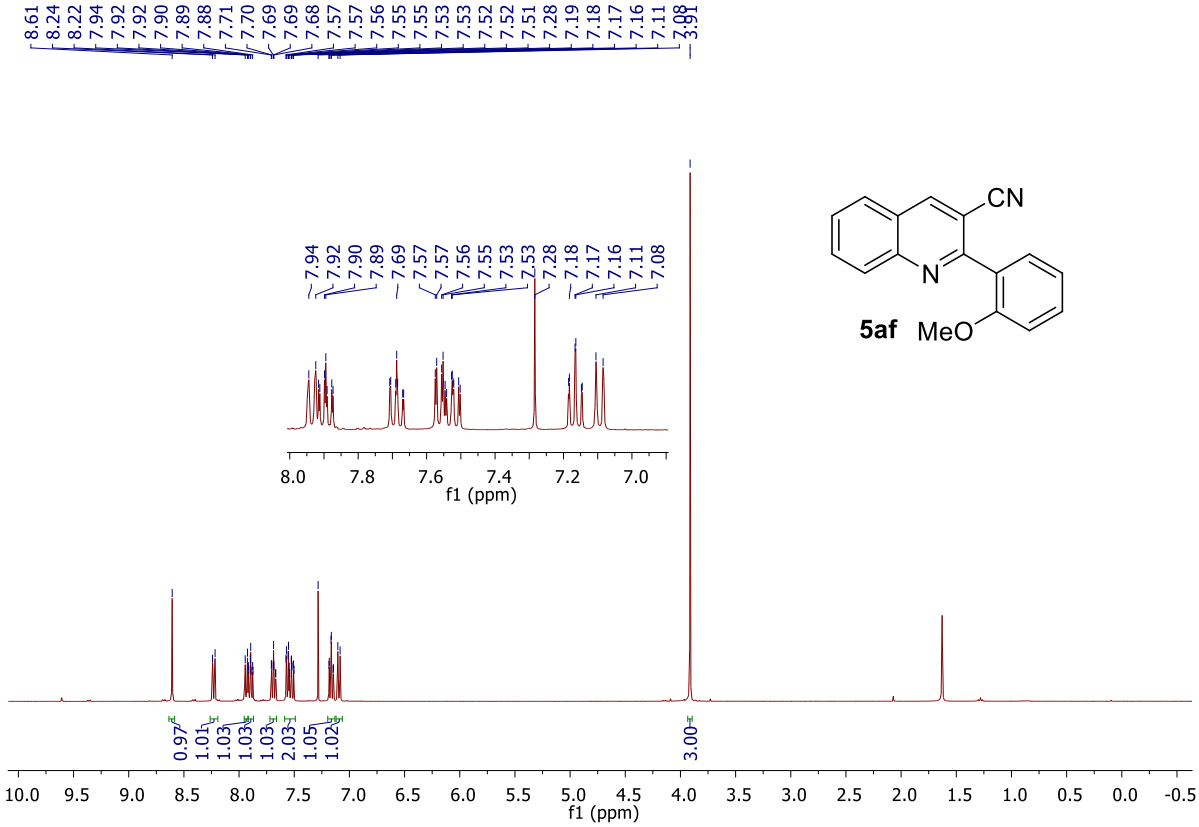

161.27
157.49
148.74
144.33
132.94
130.71
130.18
129.79
127.77
127.74
124.79
118.29
114.19
105.26
55.47
CN
N
5ac
OMe
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

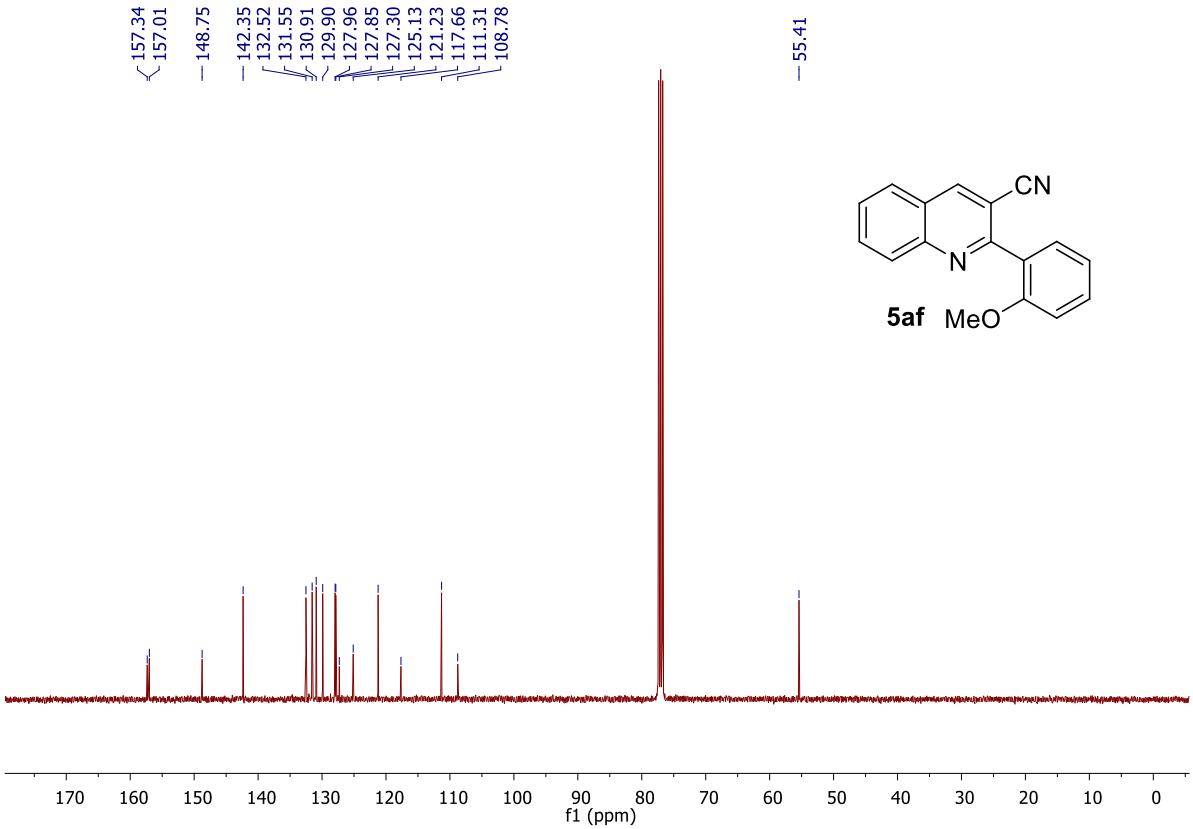

8.67
7.92
7.90
7.90
7.68
7.62
7.08
7.06
4.04
4.00
CN
N
OMe
OMe
5ad
8.22
8.20
7.92
7.90
7.90
7.89
7.70
7.70
7.68
7.68
7.67
7.66
7.65
7.62
7.62
7.08
7.06
8.0
7.5
7.0
f1 (ppm)
0.96
1.01
0.71
1.18
2.04
0.94
1.17
3.00
2.94
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

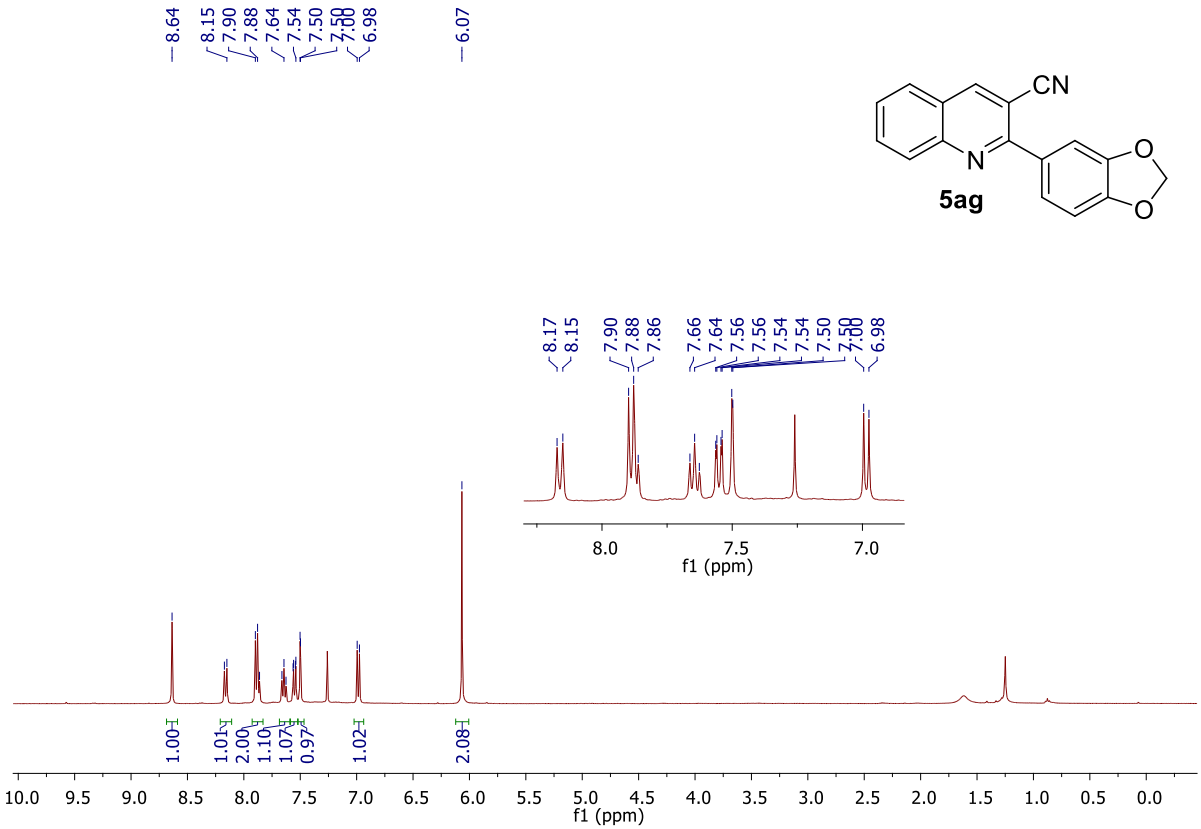

157.4
150.8
149.1
148.7
144.4
133.0
130.2
129.8
127.8
127.8
124.8
122.4
118.3
112.0
110.9
105.3
56.1
56.1
CN
N
OMe
OMe
5ad
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

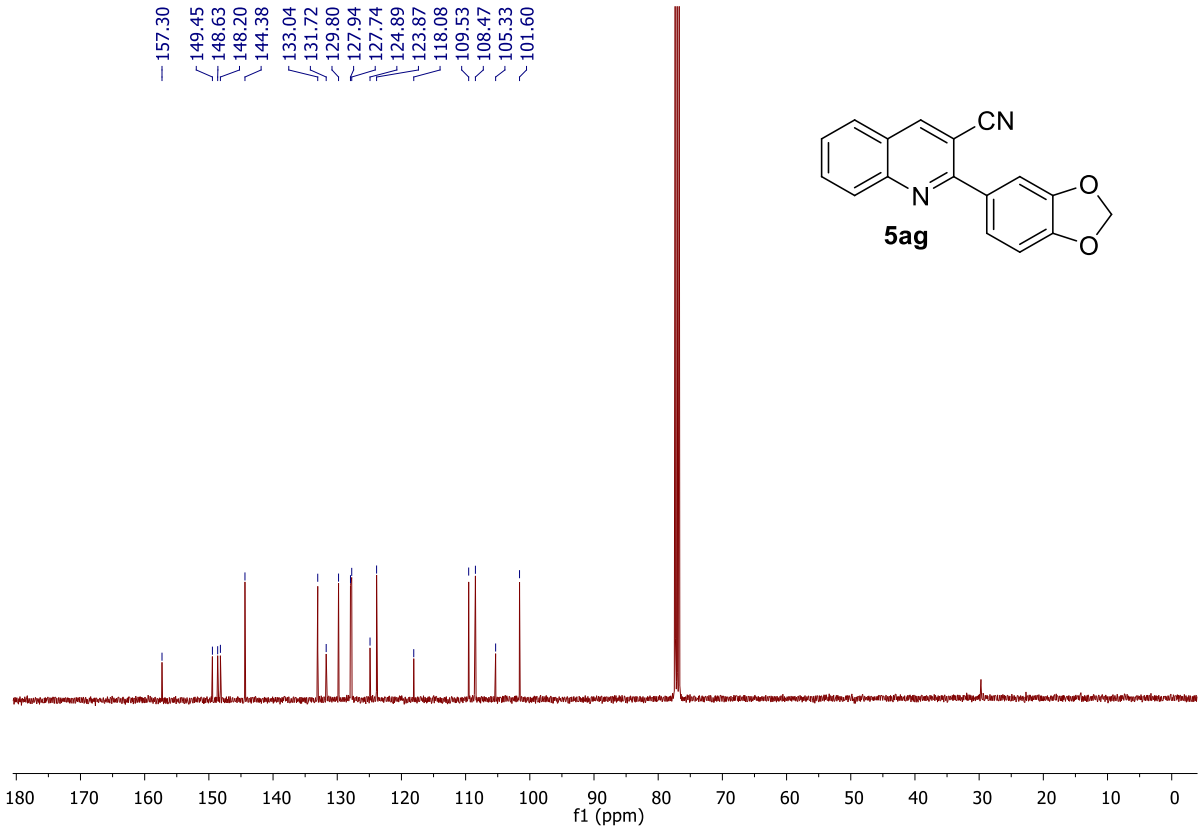

8.56
8.12
8.10
7.85
7.84
7.82
7.81
7.81
7.80
7.79
7.62
7.62
7.60
7.60
7.60
7.58
7.58
7.36
7.35
7.34
7.34
7.33
7.32
7.31
7.31
7.28
7.26
2.22
CN
N
Me
5ae
f1 (ppm)
1.00
1.00
1.21
0.84
1.06
2.15
2.04
3.00


159.92
142.95
136.14
132.96
130.84
129.86
129.64
129.25
128.20
127.90
126.01
125.07
117.13
107.55
19.70
CN
N
Me
5ae
f1 (ppm)

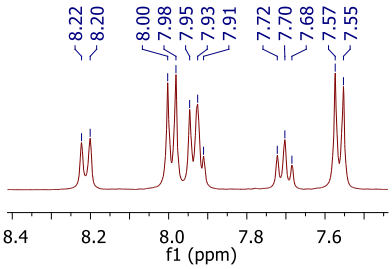

8.61
8.24
8.22
7.94
7.92
7.92
7.90
7.89
7.88
7.71
7.70
7.69
7.69
7.68
7.57
7.57
7.56
7.55
7.55
7.53
7.53
7.52
7.52
7.51
7.28
7.19
7.18
7.17
7.16
7.11
7.08
3.91
7.94
7.92
7.90
7.89
7.69
7.57
7.57
7.56
7.55
7.53
7.53
7.28
7.18
7.17
7.16
7.11
7.08
8.0
7.8
7.6
7.4
7.2
7.0
f1 (ppm)
CN
N
5af
MeO
0.97
1.01
1.03
1.03
1.03
2.03
1.05
1.02
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

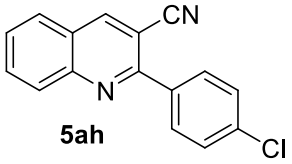

157.34
157.01
148.75
142.35
132.52
131.55
130.91
129.90
127.96
127.85
127.30
125.13
121.23
117.66
111.31
108.78
55.41
CN
N
5af MeO
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

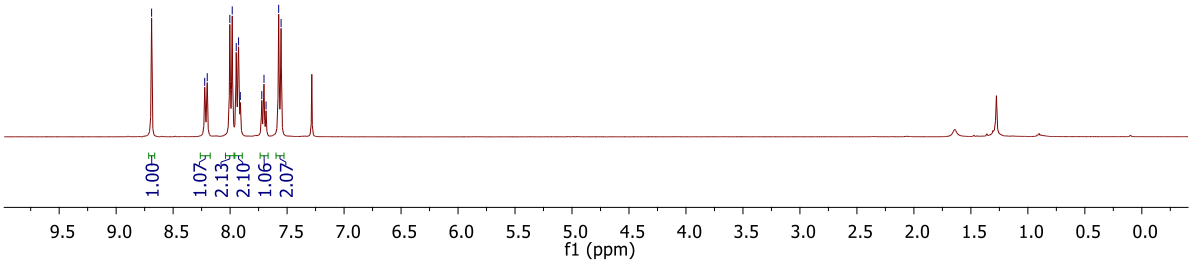

8.64
8.15
7.90
7.88
7.64
7.54
7.50
7.00
6.98
6.07
CN
N
O
O
5ag
8.17
8.15
7.90
7.88
7.86
7.66
7.64
7.56
7.56
7.54
7.54
7.50
7.00
6.98
8.0
7.5
7.0
f1 (ppm)
1.00
1.01
2.00
1.10
1.07
0.97
1.02
2.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

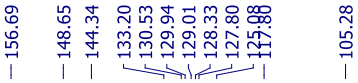

157.30
149.45
148.63
148.20
144.38
133.04
131.72
129.80
127.94
127.74
124.89
123.87
118.08
109.53
108.47
105.33
101.60
CN
N
O
O
5ag
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

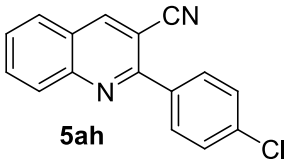

8.69
8.22
8.20
8.00
7.98
7.95
7.93
7.91
7.72
7.70
7.68
7.57
7.55

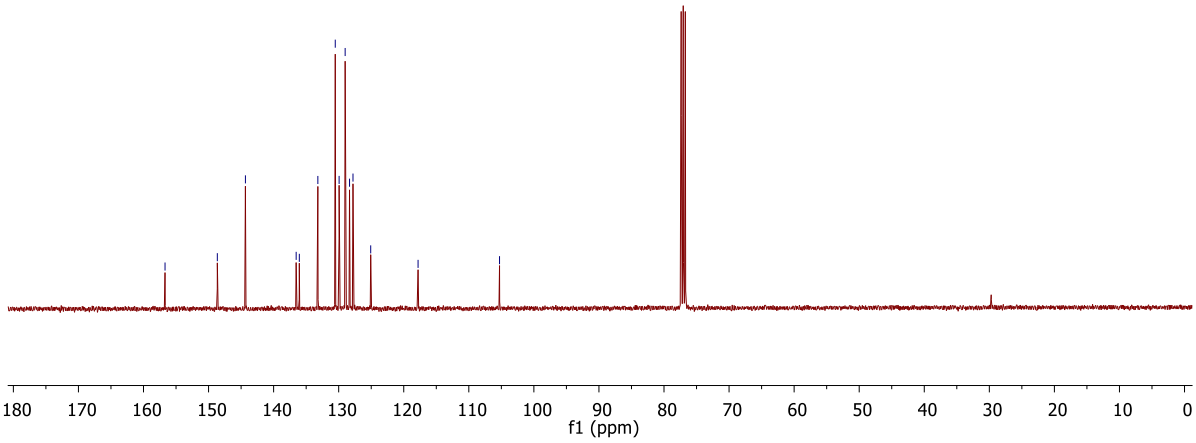

8.22
8.20
8.00
7.98
7.95
7.93
7.91
7.72
7.70
7.68
7.57
7.55
8.4
8.2
8.0
7.8
7.6
f1 (ppm)

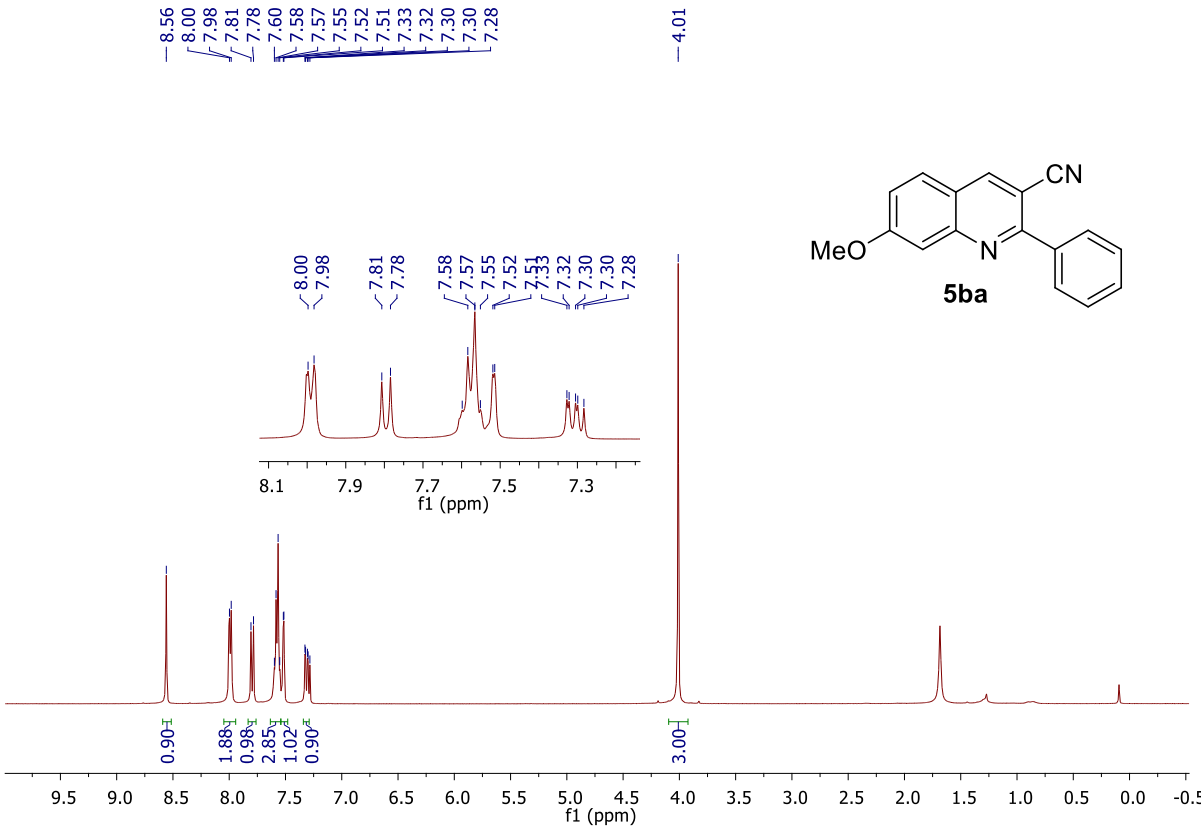

CN
N
Cl
5ah

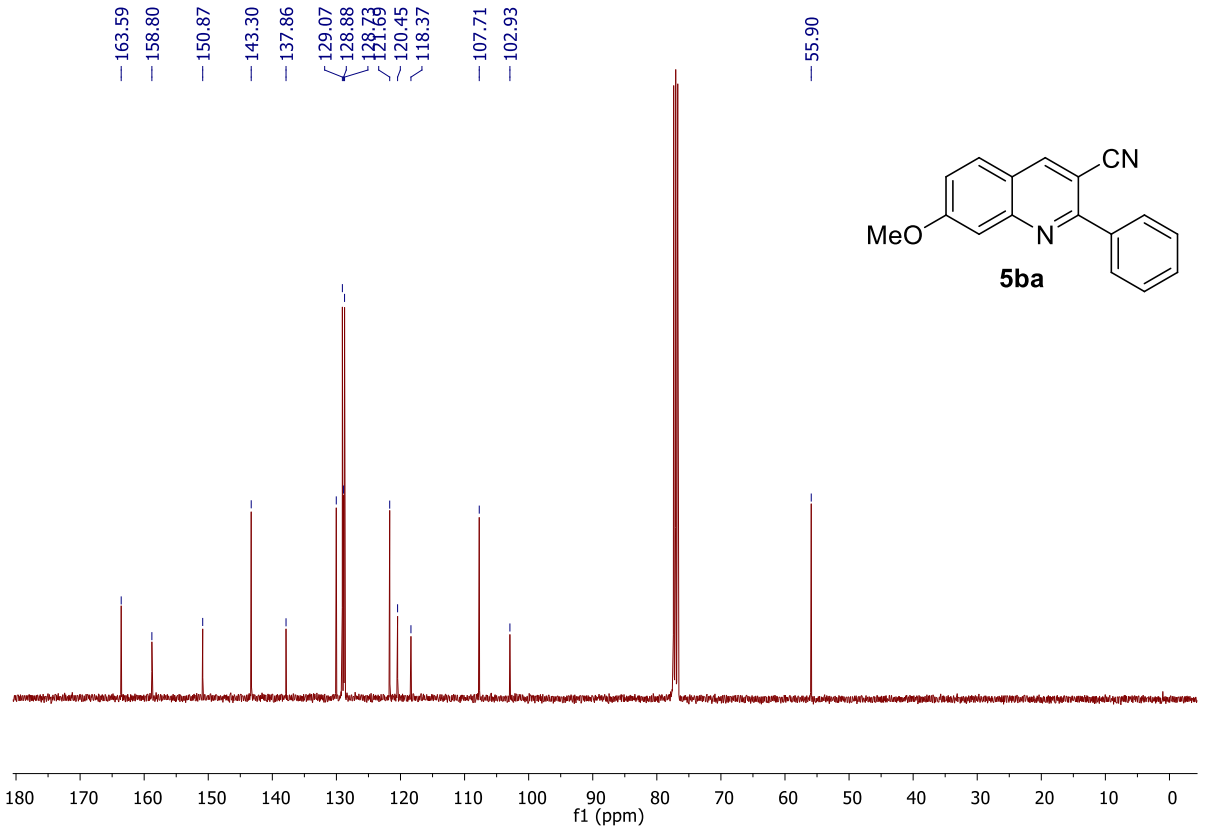

1.00
1.07
2.13
2.10
1.06
2.07
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

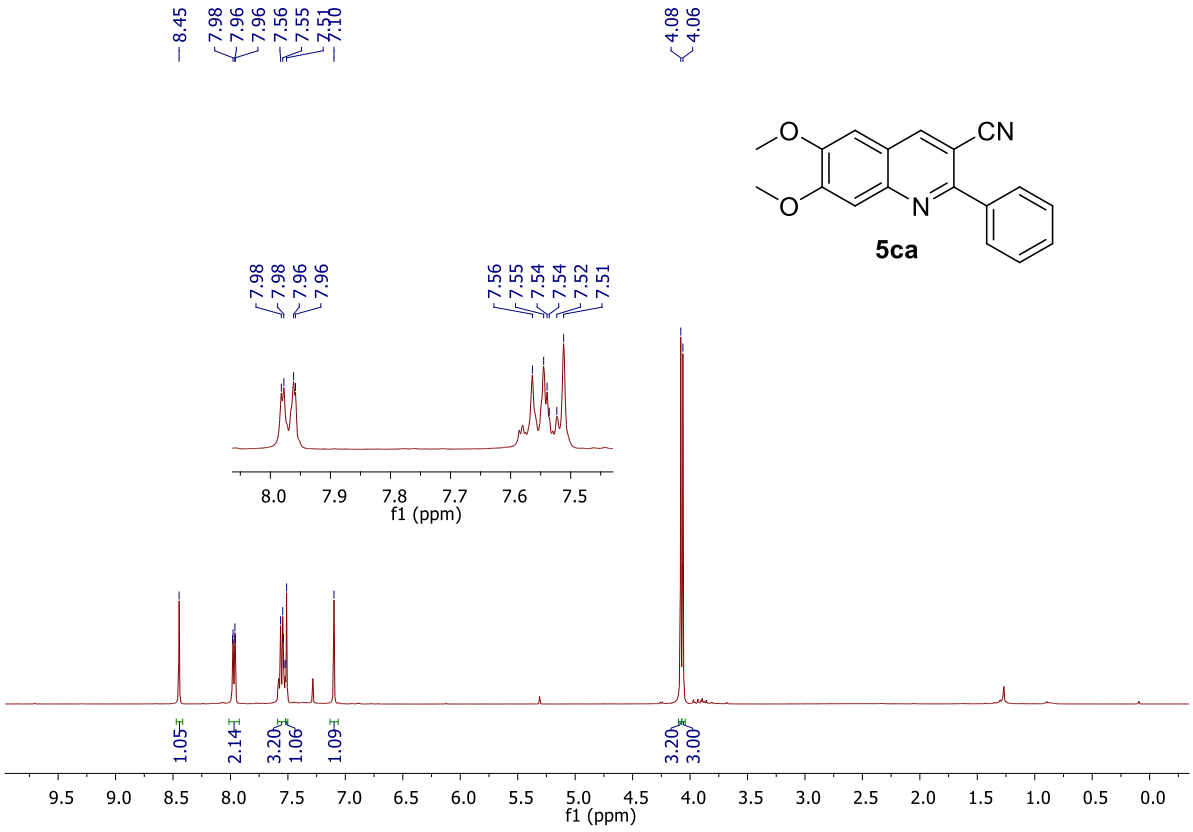

156.69
148.65
144.34
133.20
130.53
129.94
129.01
128.33
127.80
105.28

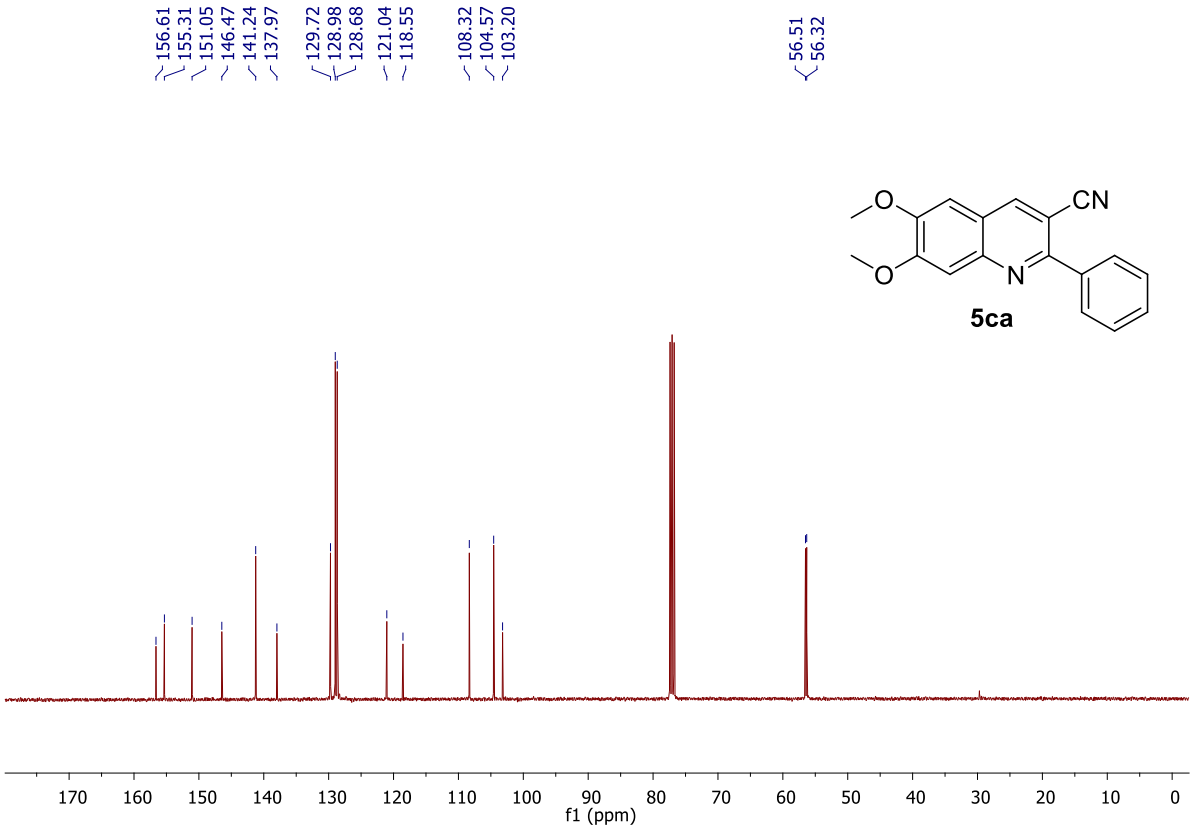

CN
N
Cl
5ah

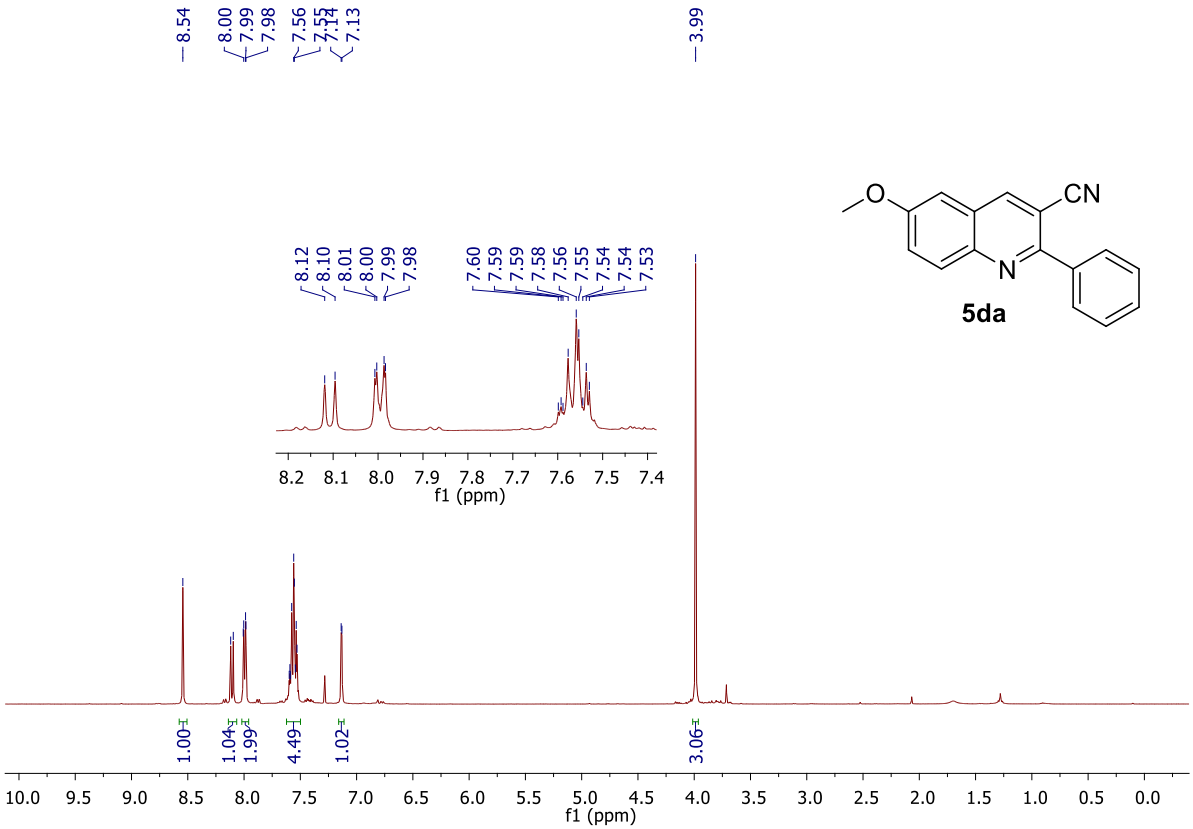

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

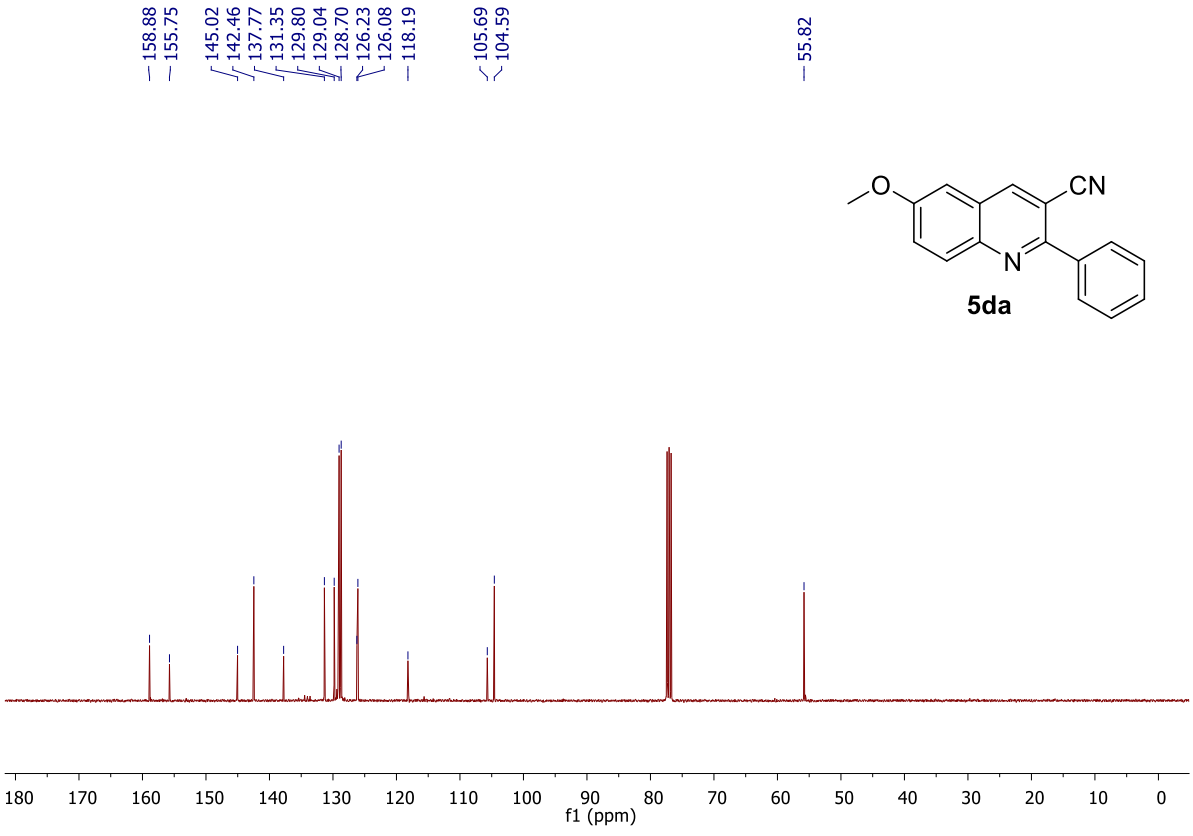

8.56
8.00
7.98
7.81
7.78
7.60
7.58
7.57
7.55
7.52
7.51
7.33
7.32
7.30
7.30
7.28
4.01
CN
MeO
N
5ba
f1 (ppm)
0.90
1.88
0.98
2.85
1.02
0.90
3.00

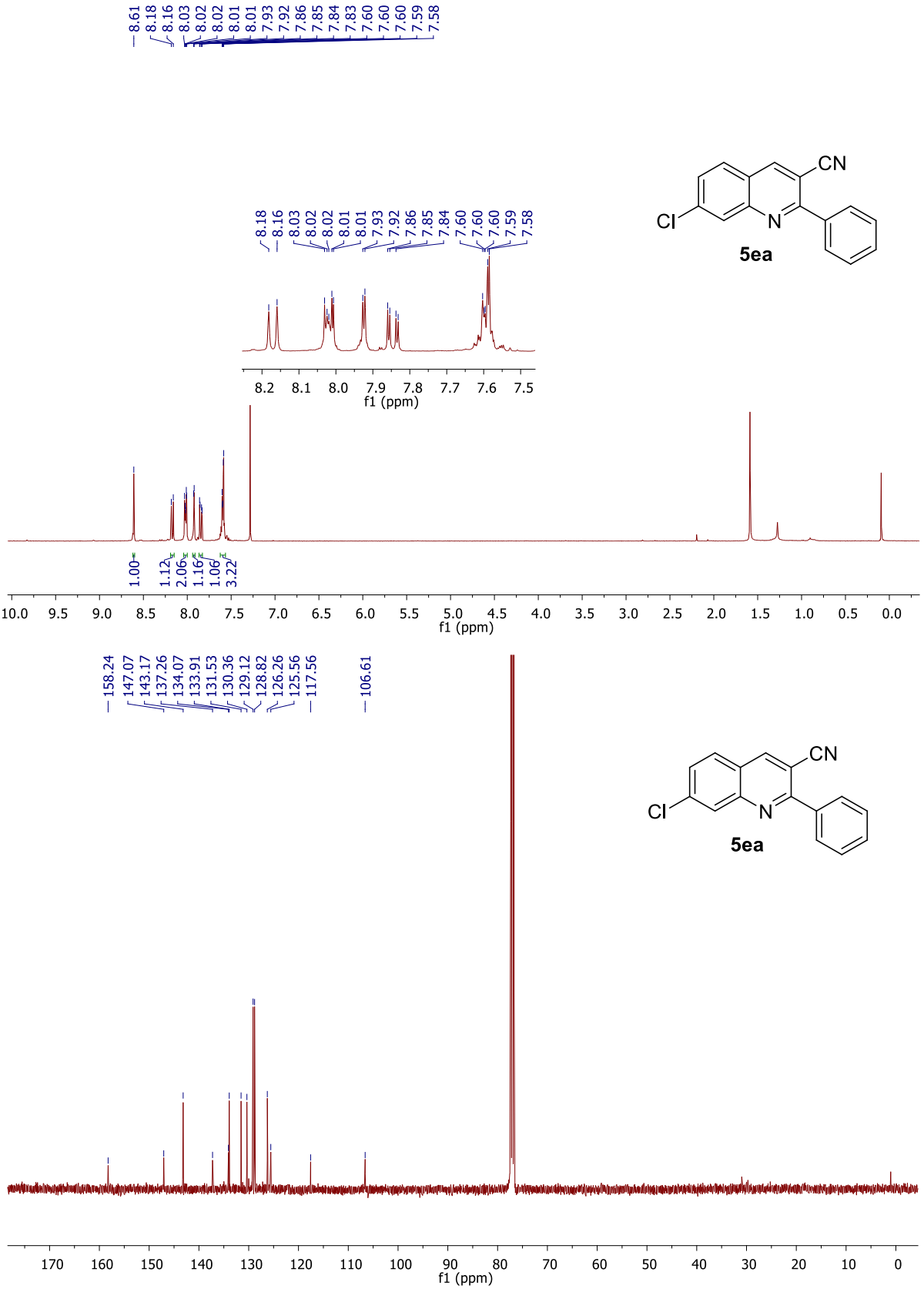

163.59
158.80
150.87
143.30
137.86
129.07
128.88
128.73
121.63
120.45
118.37
107.71
102.93
55.90
CN
MeO
N
5ba
f1 (ppm)

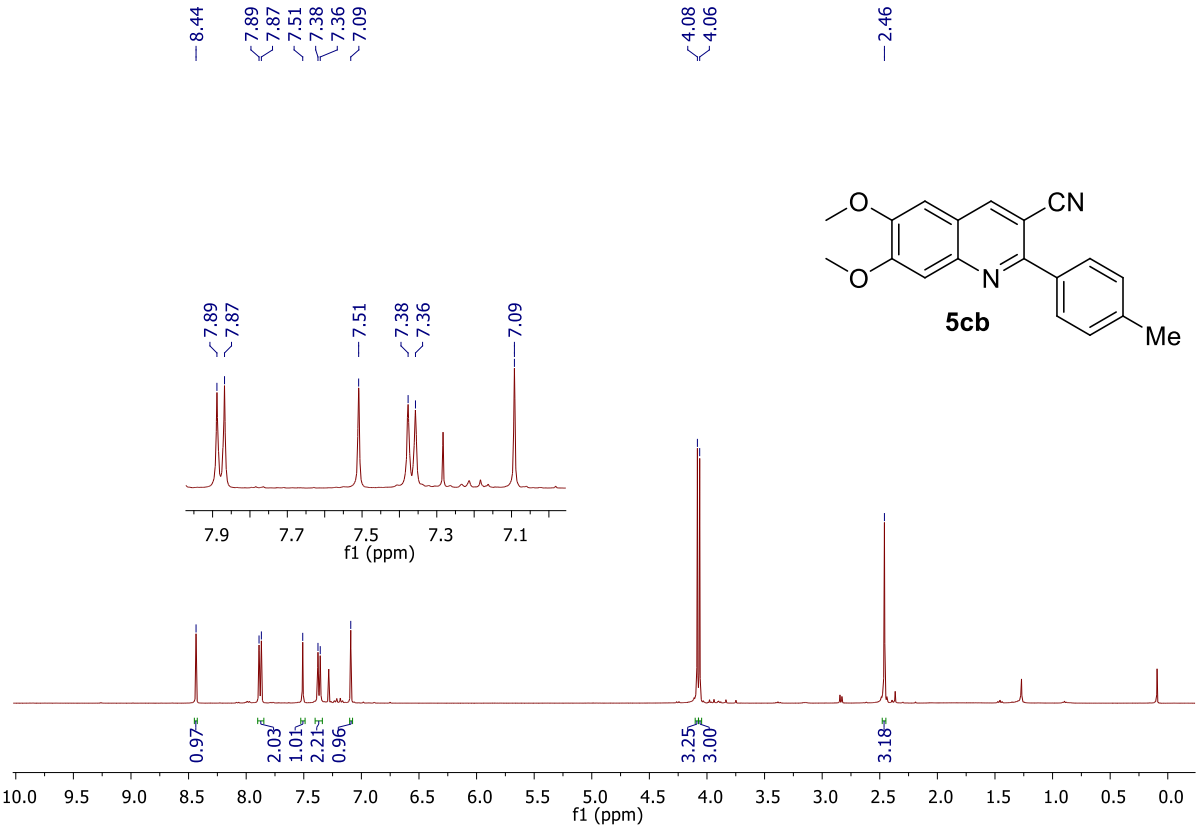

8.45
7.98
7.96
7.96
7.56
7.55
7.51
7.10
4.08
4.06
O
O
CN
N
5ca
7.98
7.98
7.96
7.96
7.56
7.55
7.54
7.54
7.52
7.51
8.0
7.9
7.8
7.7
7.6
7.5
f1 (ppm)
1.05
2.14
3.20
1.06
1.09
3.20
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

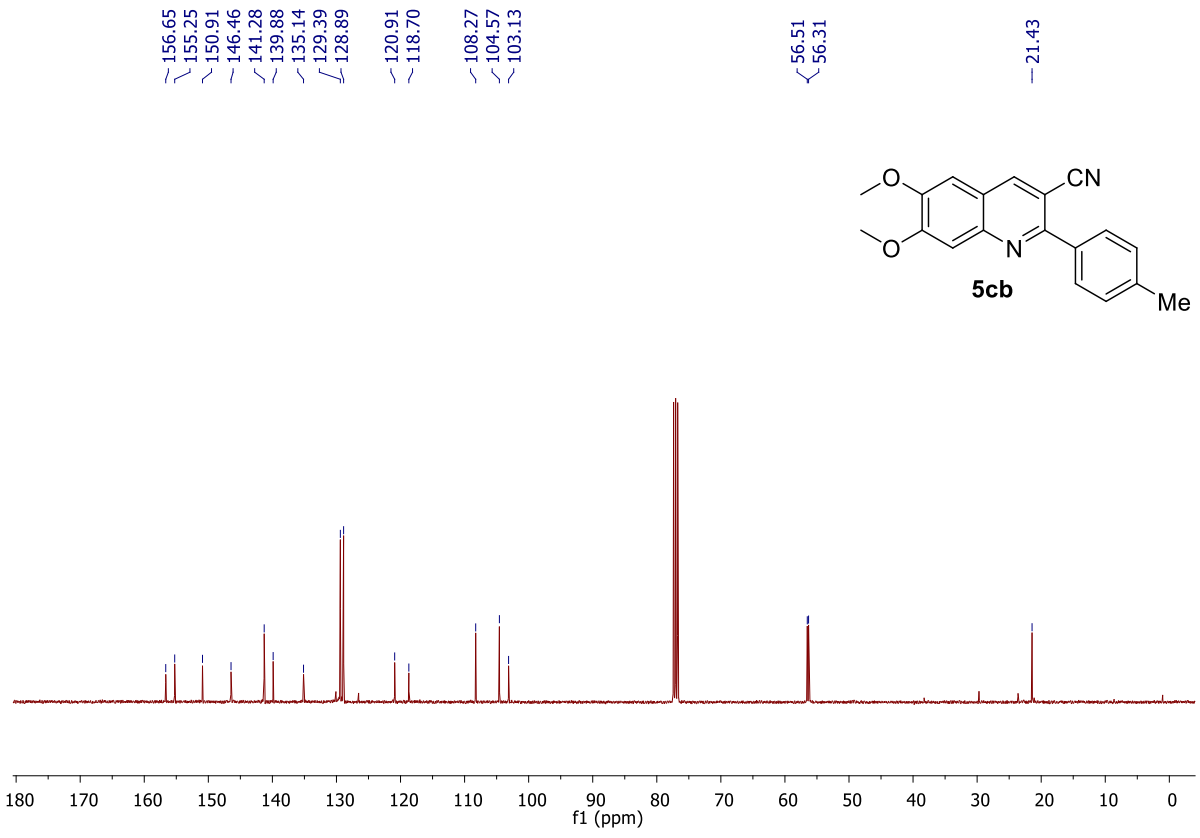

156.61
155.31
151.05
146.47
141.24
137.97
129.72
128.98
128.68
121.04
118.55
108.32
104.57
103.20
56.51
56.32
O
O
CN
N
5ca
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

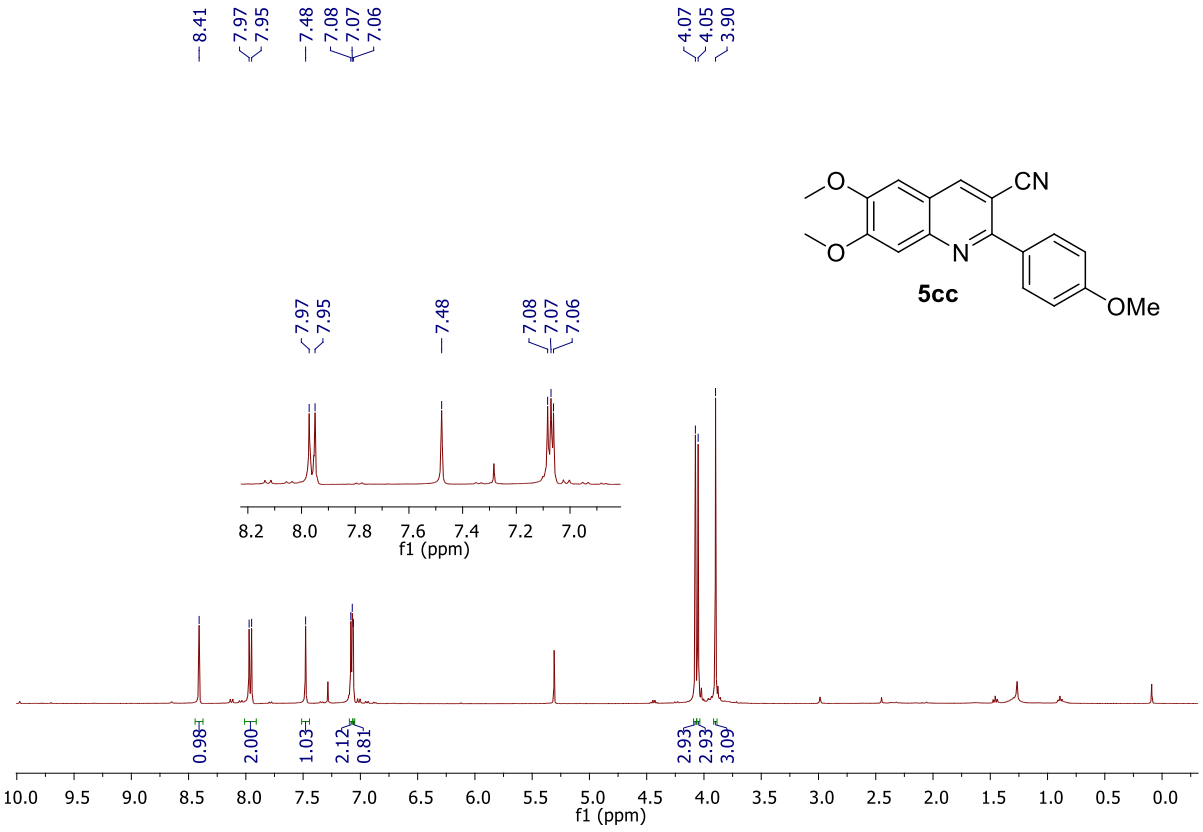

8.54
8.00
7.99
7.98
7.56
7.55
7.14
7.13
3.99
8.12
8.10
8.01
8.00
7.99
7.98
7.60
7.59
7.59
7.58
7.56
7.55
7.54
7.54
7.53
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
1.00
1.04
1.99
4.49
1.02
3.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
O
CN
N
5da

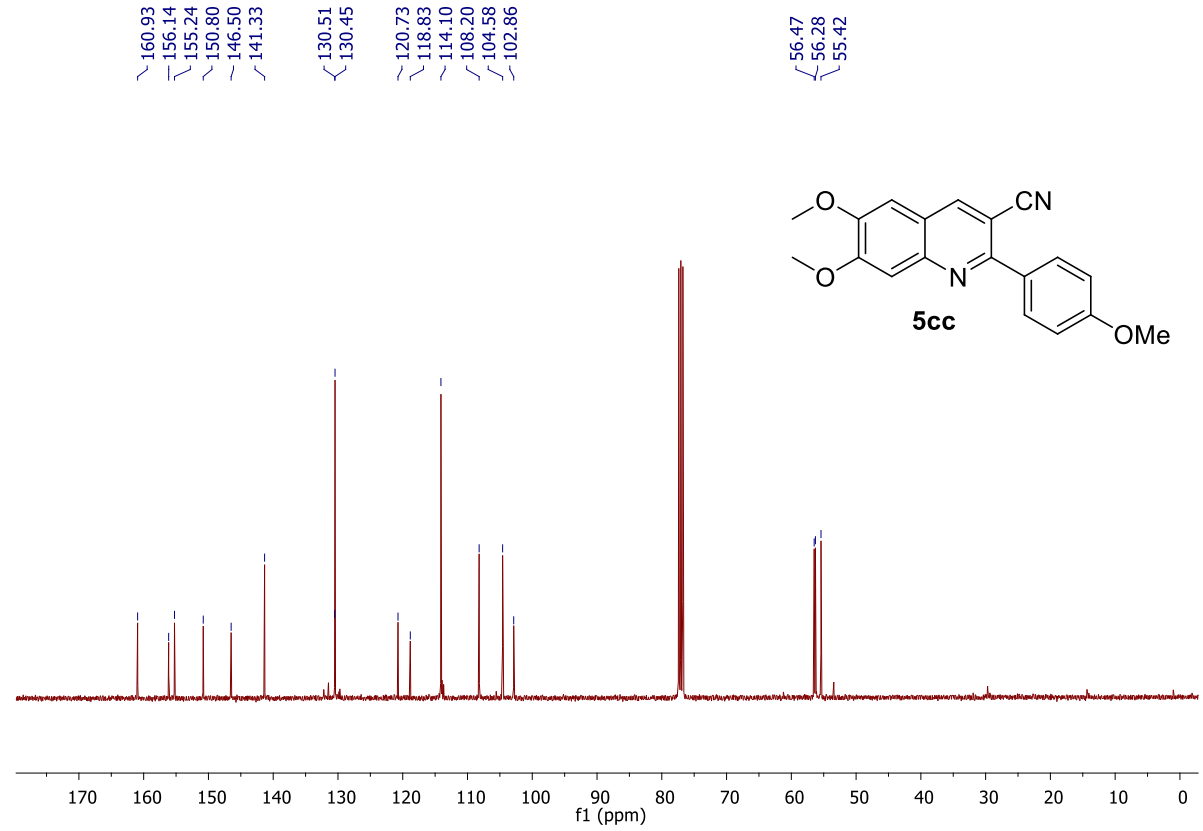

158.88
155.75
145.02
142.46
137.77
131.35
129.80
129.04
128.70
126.23
126.08
118.19
105.69
104.59
55.82
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
O
CN
N
5da

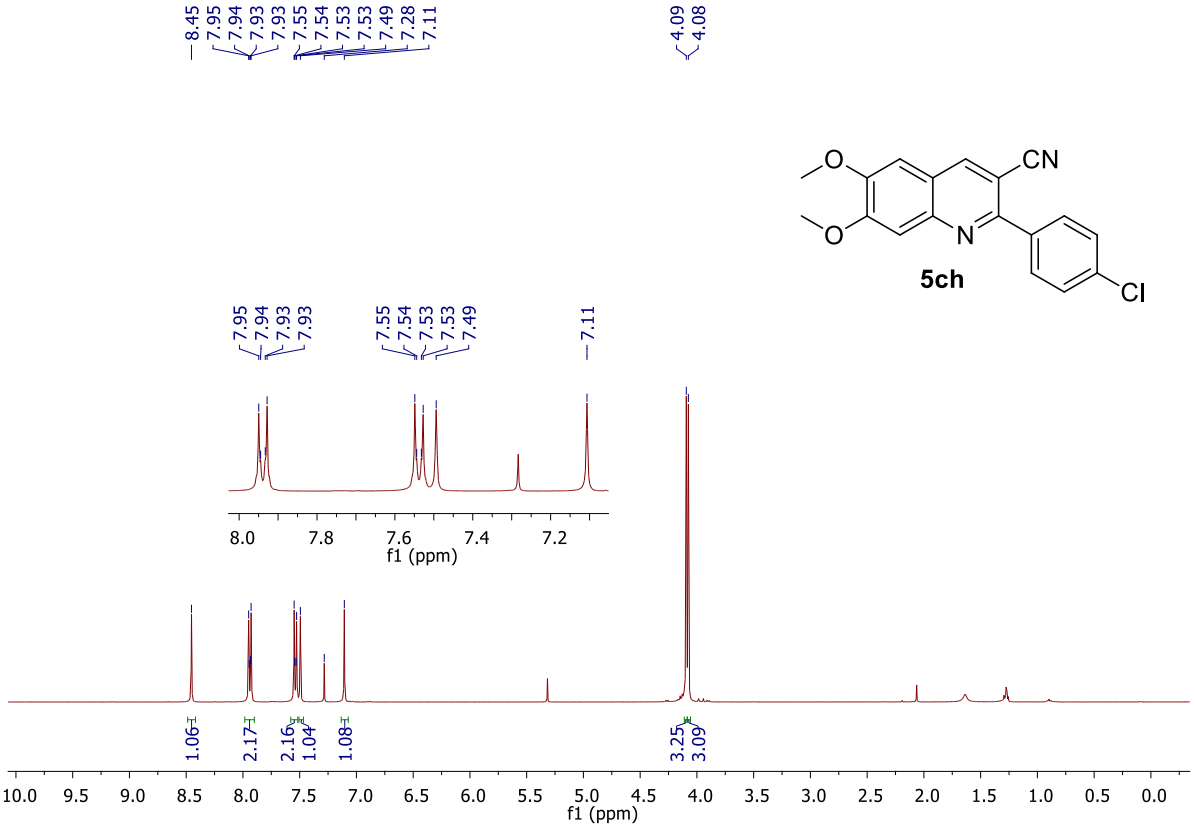

8.61
8.18
8.16
8.03
8.02
8.02
8.01
8.01
7.93
7.92
7.86
7.85
7.84
7.83
7.60
7.60
7.60
7.59
7.58
CN
Cl
N
5ea
f1 (ppm)
1.00
1.12
2.06
1.16
1.06
3.22
158.24
147.07
143.17
137.26
134.07
133.91
131.53
130.36
129.12
128.82
126.26
125.56
117.56
106.61

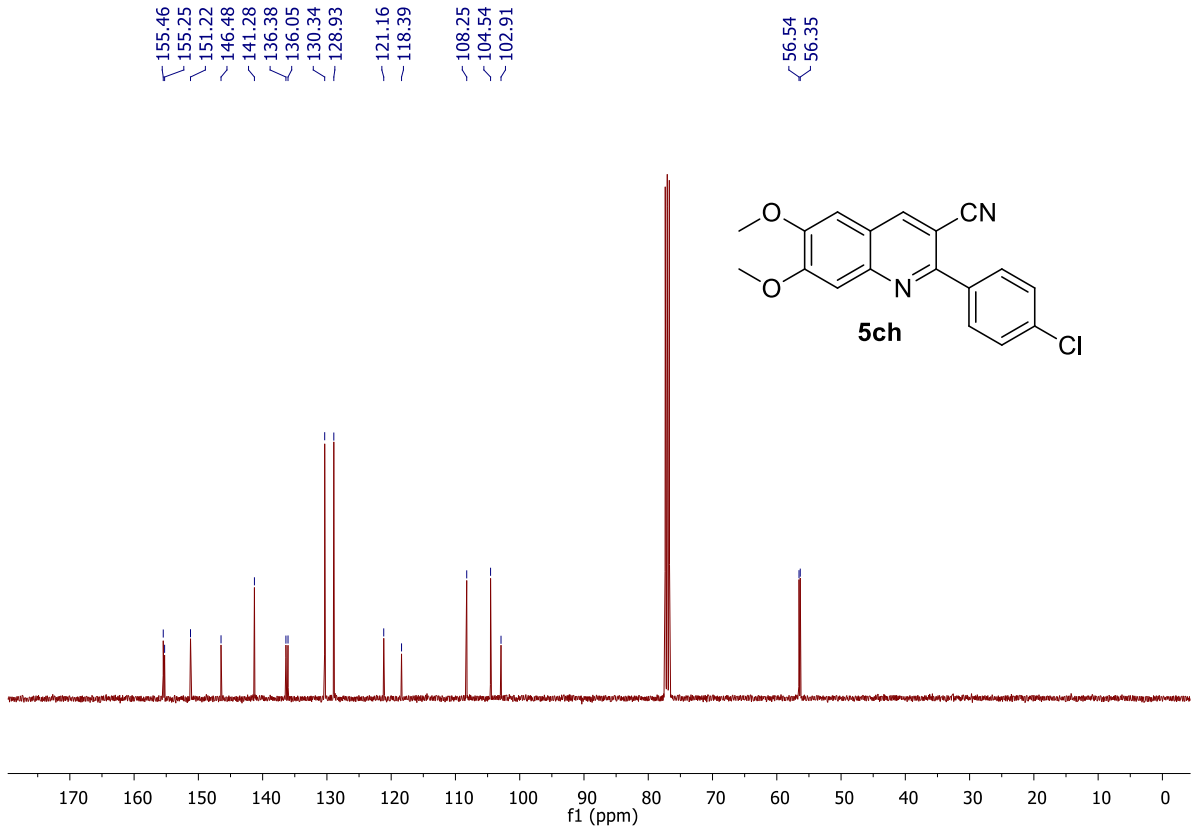

8.44
7.89
7.87
7.51
7.38
7.36
7.09
4.08
4.06
2.46
O
O
CN
N
Me
5cb
f1 (ppm)
0.97
2.03
1.01
2.21
0.96
3.25
3.00
3.18

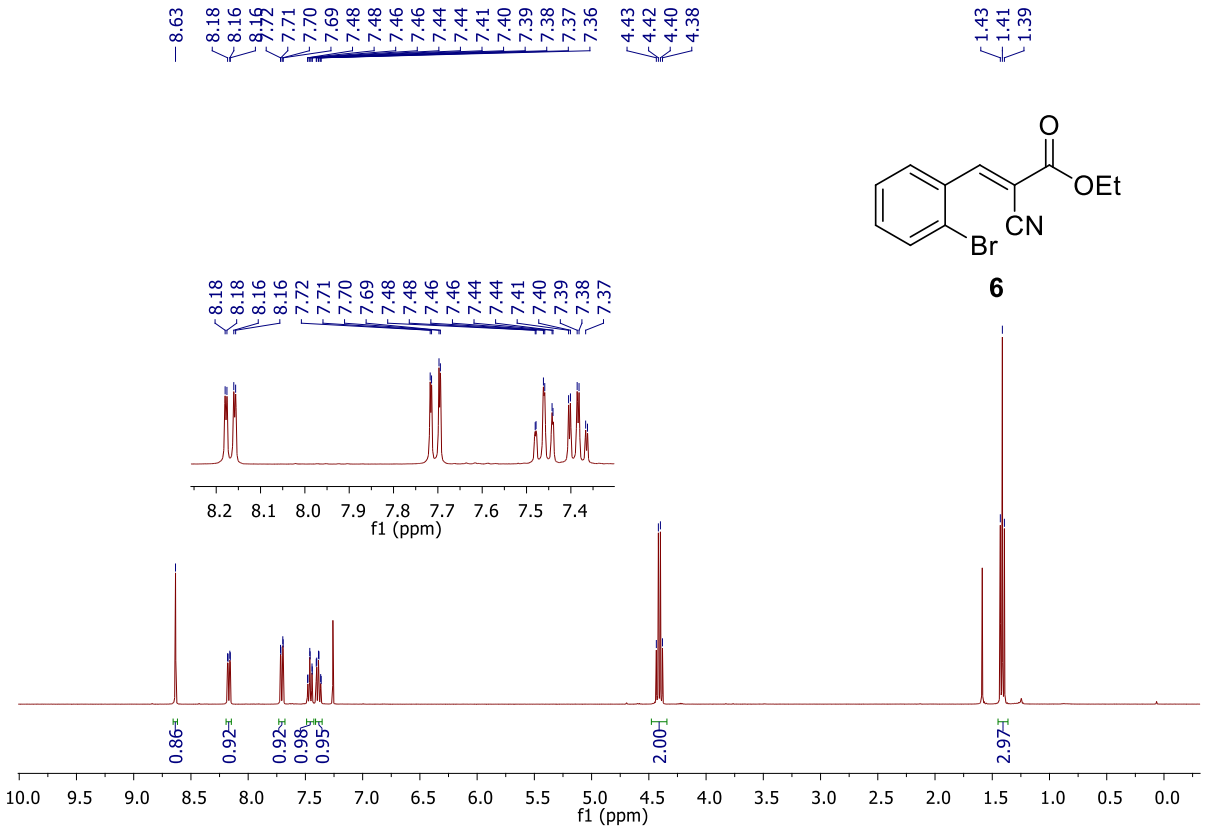

156.65
155.25
150.91
146.46
141.28
139.88
135.14
129.39
128.89
120.91
118.70
108.27
104.57
103.13
56.51
56.31
21.43
O
O
CN
N
Me
5cb
f1 (ppm)

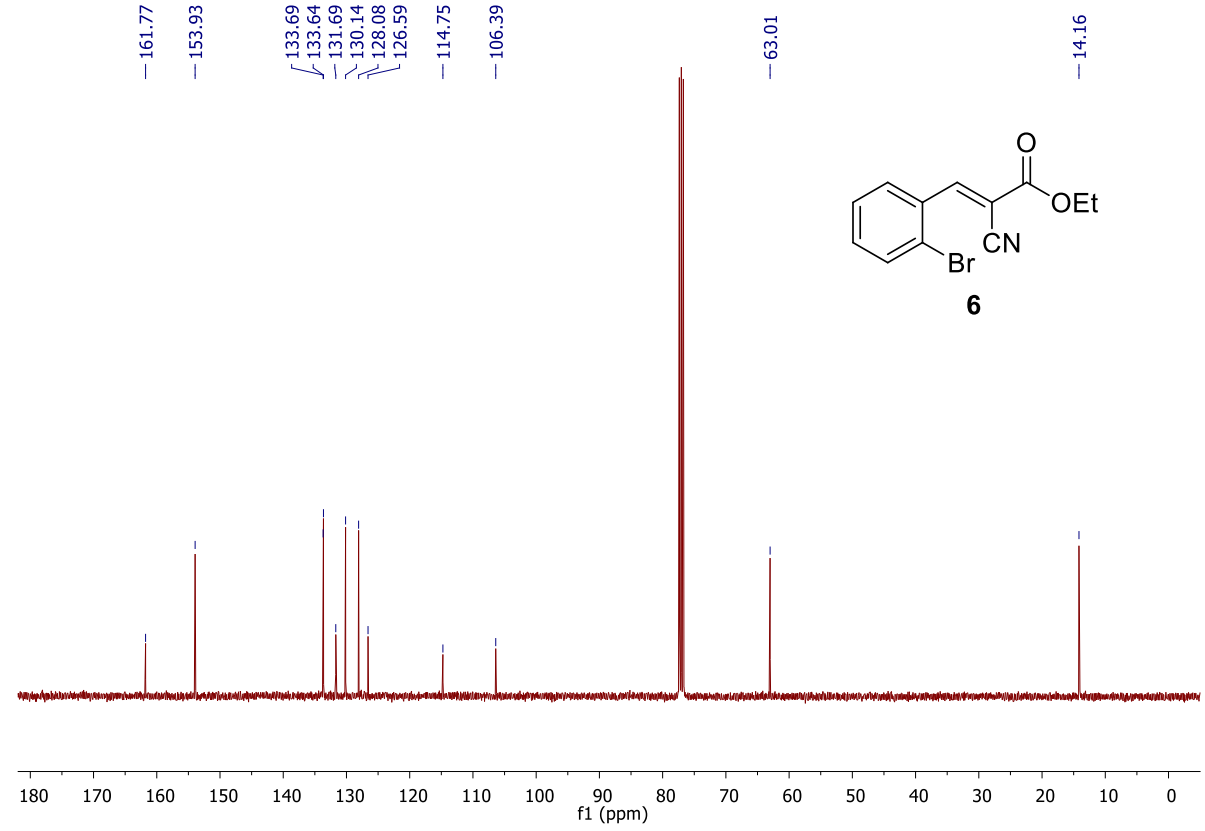

8.41
7.97
7.95
7.48
7.08
7.07
7.06
4.07
4.05
3.90
O
O
CN
N
OMe
5cc
0.98
2.00
1.03
2.12
0.81
2.93
2.93
3.09
f1 (ppm)

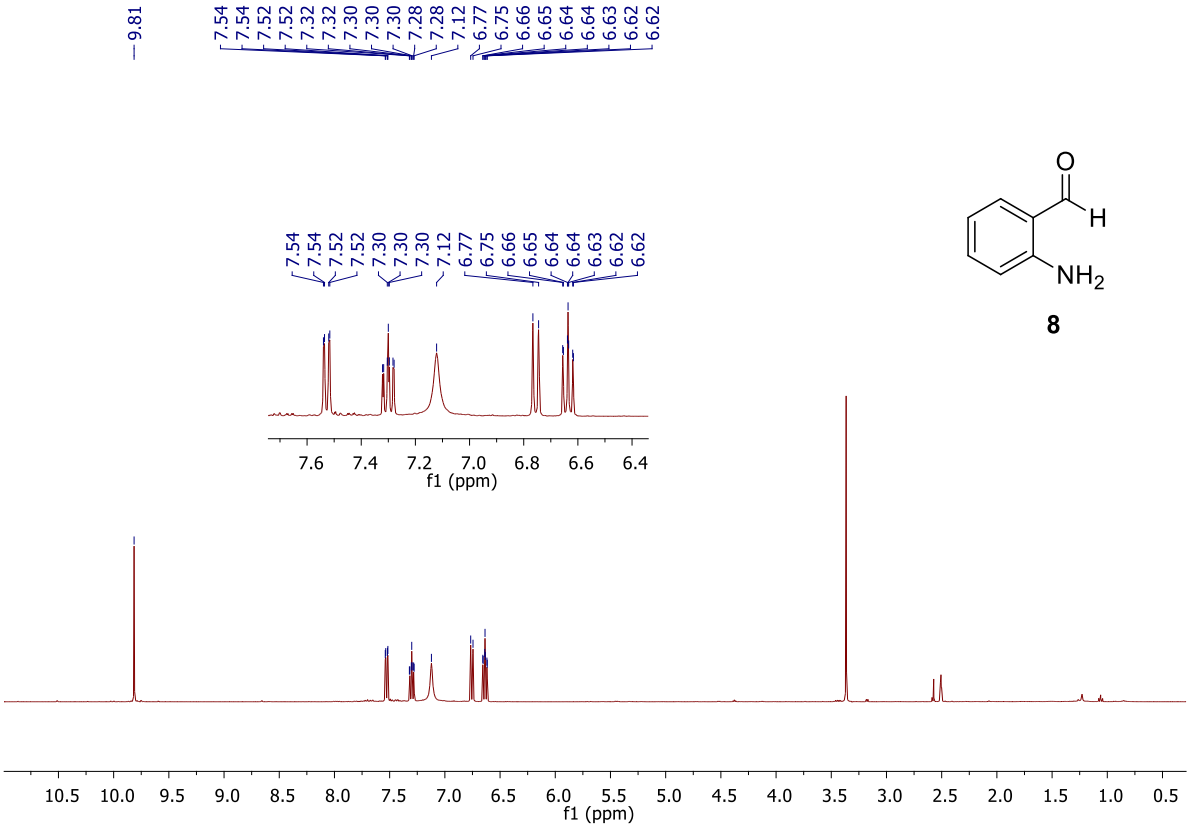

160.93
156.14
155.24
150.80
146.50
141.33
130.51
130.45
120.73
118.83
114.10
108.20
104.58
102.86
56.47
56.28
55.42
O
O
CN
N
OMe
5cc
f1 (ppm)

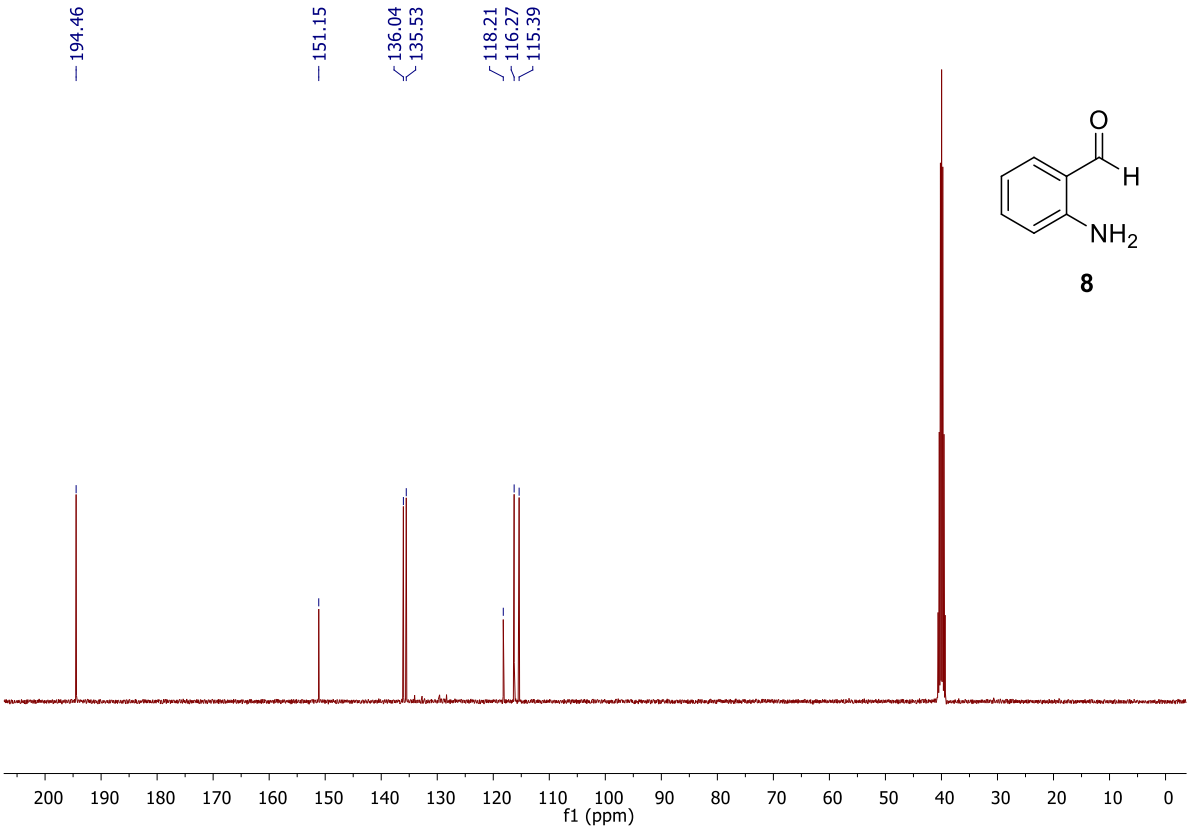

8.45
7.95
7.94
7.93
7.93
7.55
7.54
7.53
7.53
7.49
7.28
7.11
4.09
4.08
O
O
CN
N
Cl
5ch
7.95
7.94
7.93
7.93
7.55
7.54
7.53
7.53
7.49
7.11
8.0
7.8
7.6
7.4
7.2
f1 (ppm)
1.06
2.17
2.16
1.04
1.08
3.25
3.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

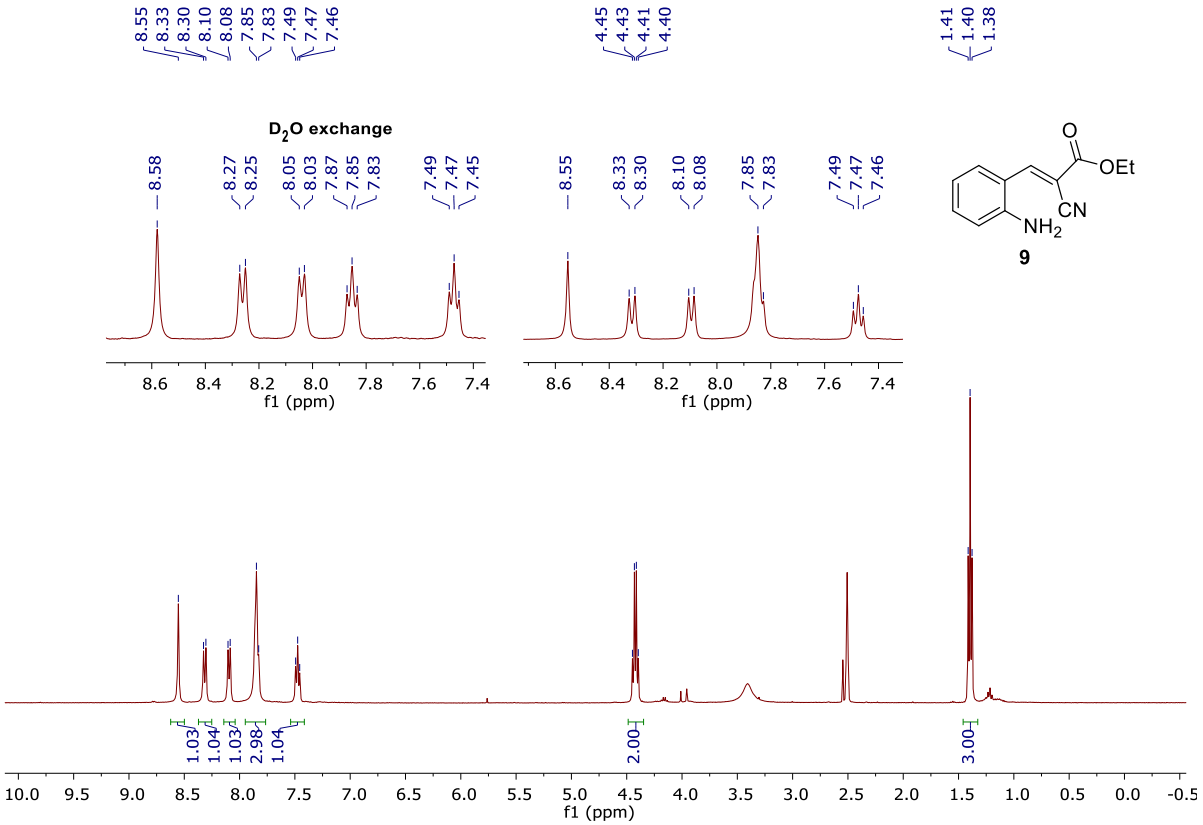

155.46
155.25
151.22
146.48
141.28
136.38
136.05
130.34
128.93
121.16
118.39
108.25
104.54
102.91
56.54
56.35
O
O
CN
N
Cl
5ch
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

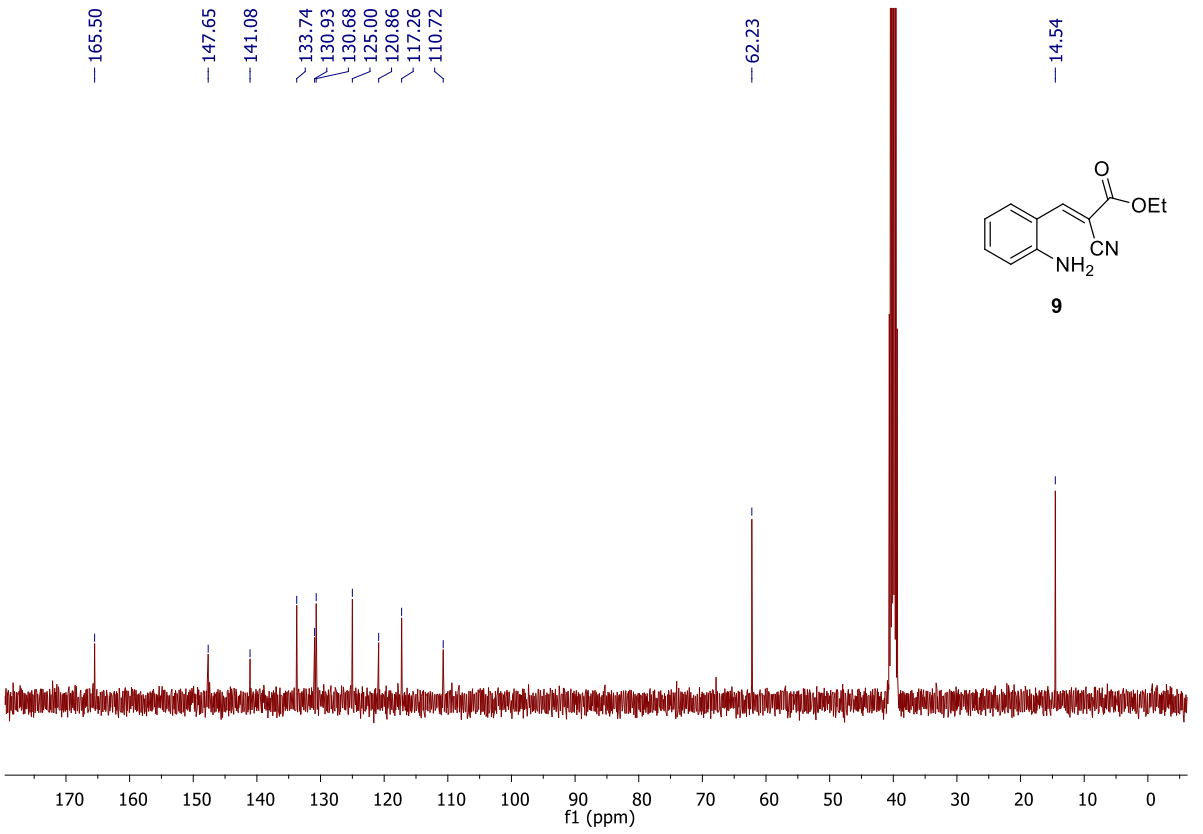

8.63
8.18
8.16
7.72
7.71
7.70
7.69
7.48
7.46
7.46
7.44
7.44
7.41
7.40
7.39
7.38
7.37
7.36
4.43
4.42
4.40
4.38
1.43
1.41
1.39
O
OEt
CN
Br
6
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
0.86
0.92
0.92
0.98
0.95
2.00
2.97
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

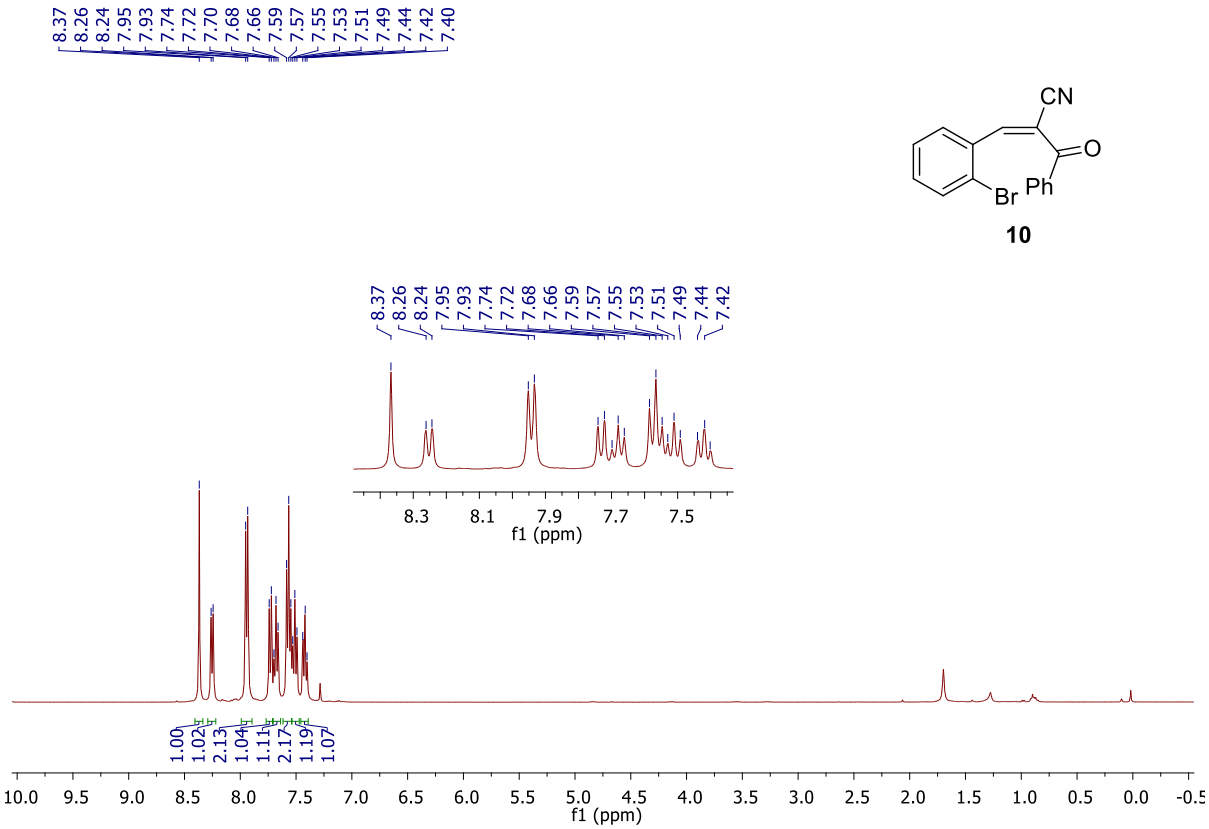

161.77
153.93
133.69
133.64
131.69
130.14
128.08
126.59
114.75
106.39
63.01
14.16
O
OEt
CN
Br
6
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

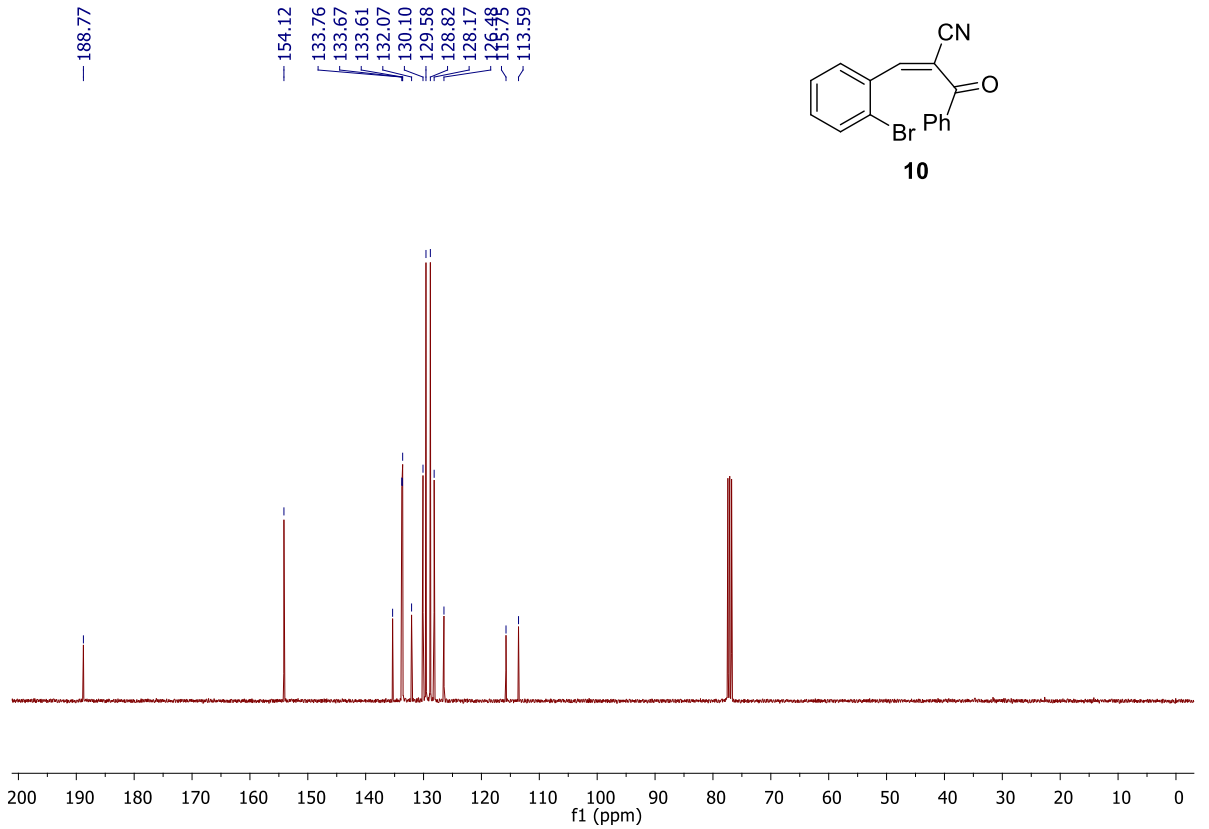

9.81
7.54
7.54
7.52
7.52
7.32
7.32
7.30
7.30
7.30
7.28
7.28
7.12
6.77
6.75
6.66
6.65
6.64
6.64
6.63
6.62
6.62
7.6
7.4
7.2
7.0
6.8
6.6
6.4
f1 (ppm)
O
H
NH2
8
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

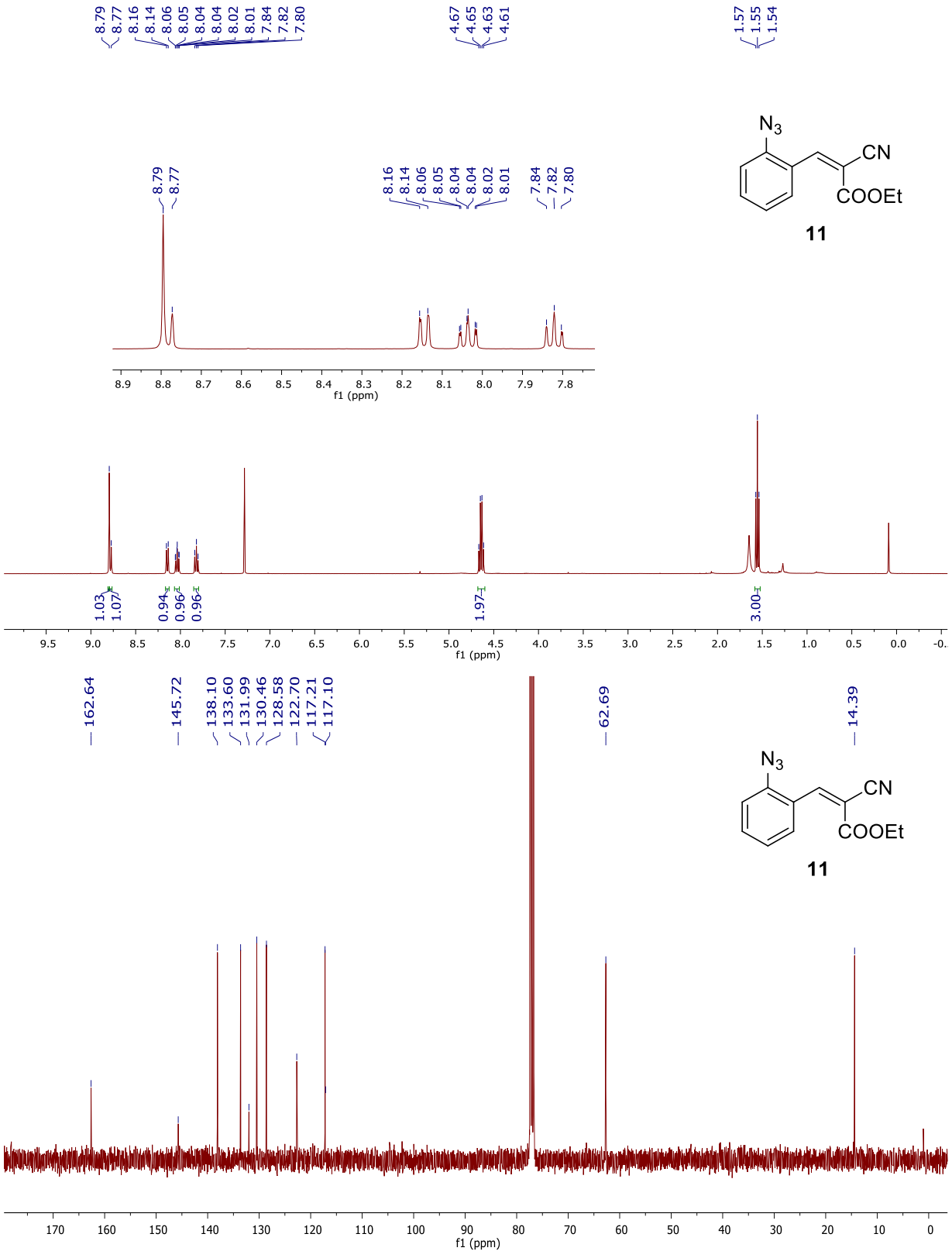

194.46
151.15
136.04
135.53
118.21
116.27
115.39
O
H
NH2
8
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.55
8.33
8.30
8.10
8.08
7.85
7.83
7.49
7.47
7.46
4.45
4.43
4.41
4.40
1.41
1.40
1.38
D2O exchange
8.58
8.27
8.25
8.05
8.03
7.87
7.85
7.83
7.49
7.47
7.45
8.55
8.33
8.30
8.10
8.08
7.85
7.83
7.49
7.47
7.46
f1 (ppm)
O
OEt
CN
NH2
9
1.03
1.04
1.03
2.98
1.04
2.00
3.00
f1 (ppm)

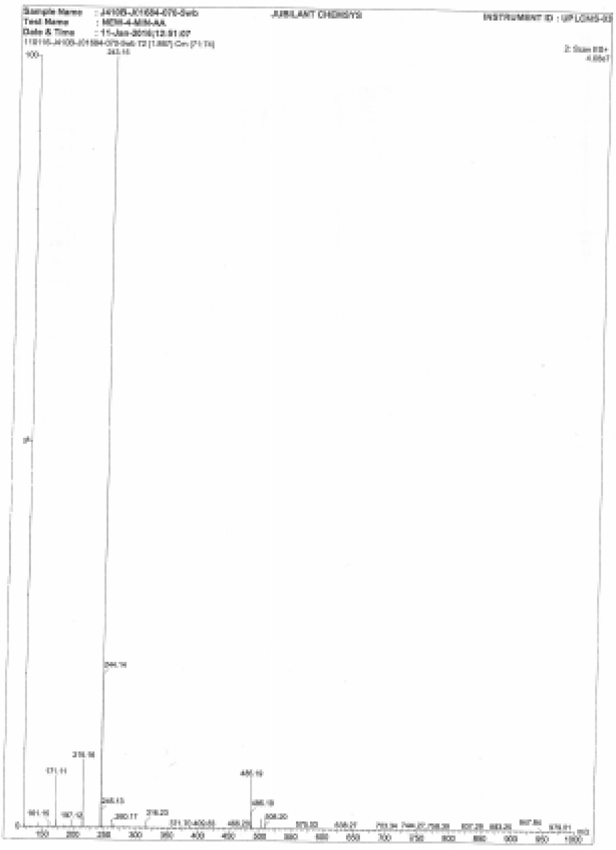

165.50
147.65
141.08
133.74
130.93
130.68
125.00
120.86
117.26
110.72
62.23
14.54
O
OEt
CN
NH2
9
f1 (ppm)

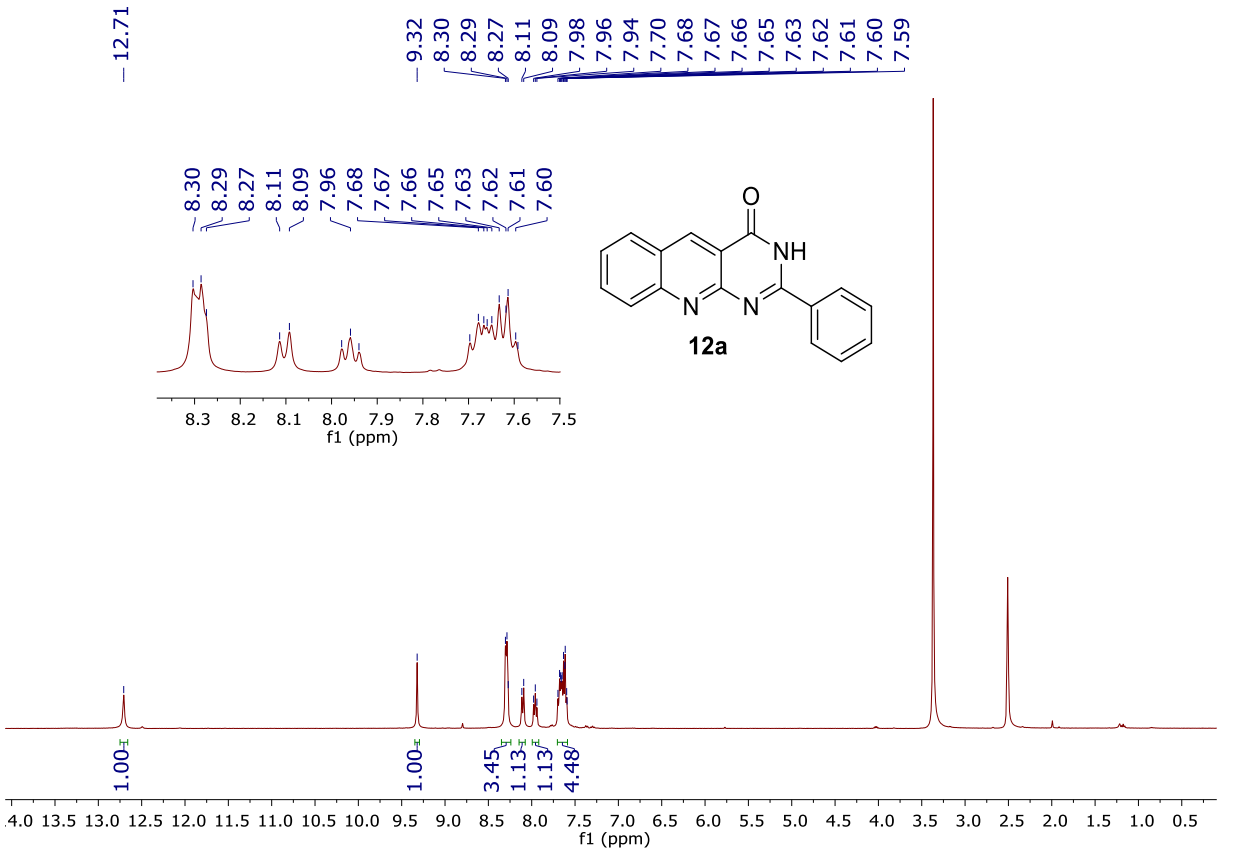

8.37
8.26
8.24
7.95
7.93
7.74
7.72
7.70
7.68
7.66
7.59
7.57
7.55
7.53
7.51
7.49
7.44
7.42
7.40
CN
O
Br
Ph
10
8.3
8.1
7.9
7.7
7.5
f1 (ppm)
1.00
1.02
2.13
1.04
1.11
2.17
1.19
1.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

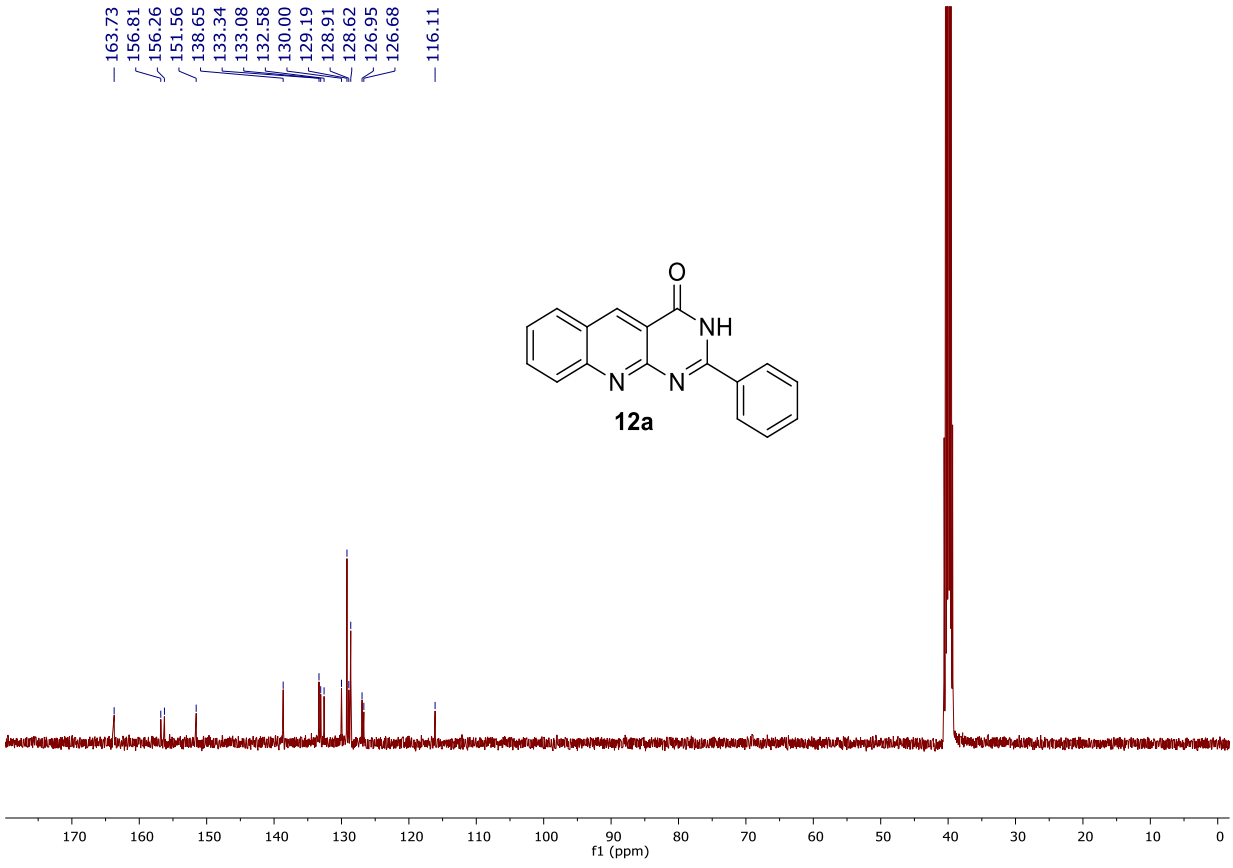

188.77
154.12
133.76
133.67
133.61
132.07
130.10
129.58
128.82
128.17
113.59
CN
O
Br
Ph
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

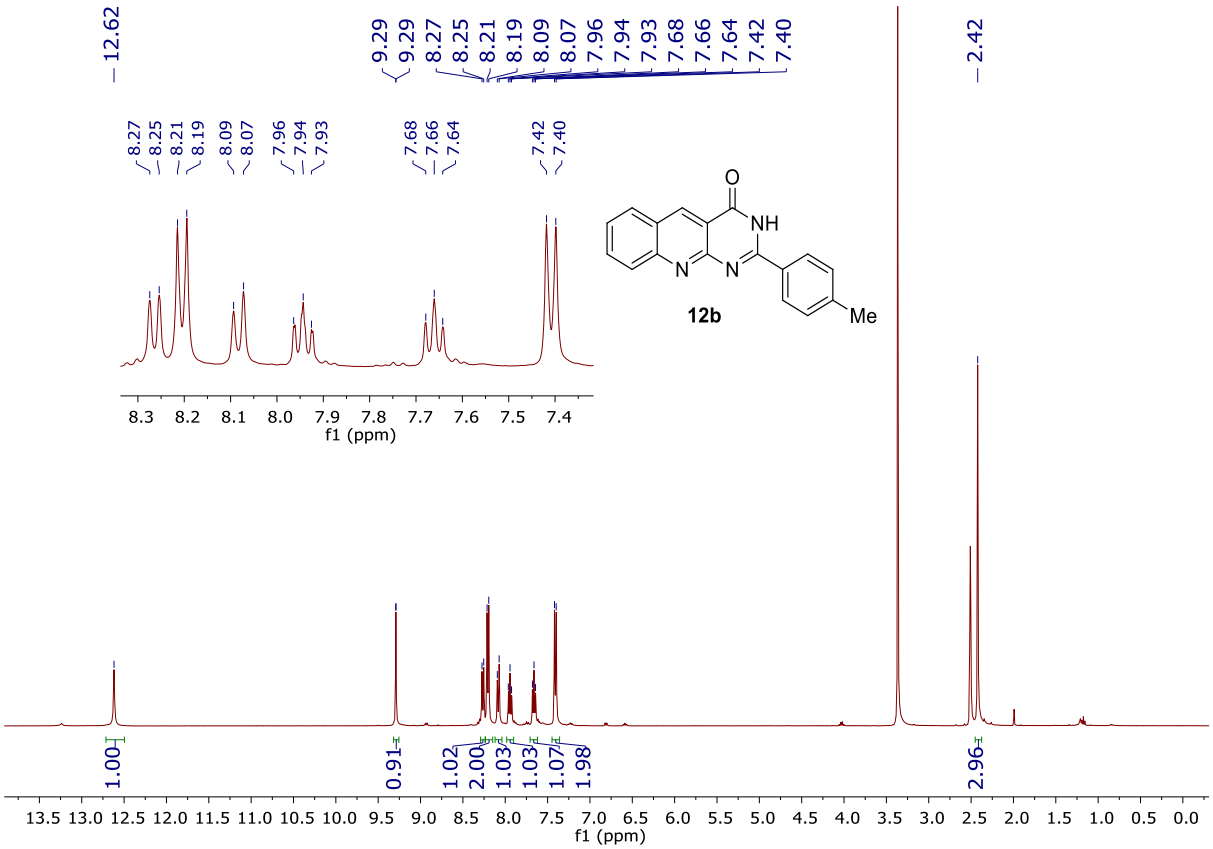

8.79
8.77
8.16
8.14
8.06
8.05
8.04
8.04
8.02
8.01
7.84
7.82
7.80
4.67
4.65
4.63
4.61
1.57
1.55
1.54
N3
CN
COOEt
11
f1 (ppm)
1.03
1.07
0.94
0.96
0.96
1.97
3.00
162.64
145.72
138.10
133.60
131.99
130.46
128.58
122.70
117.21
117.10
62.69
14.39

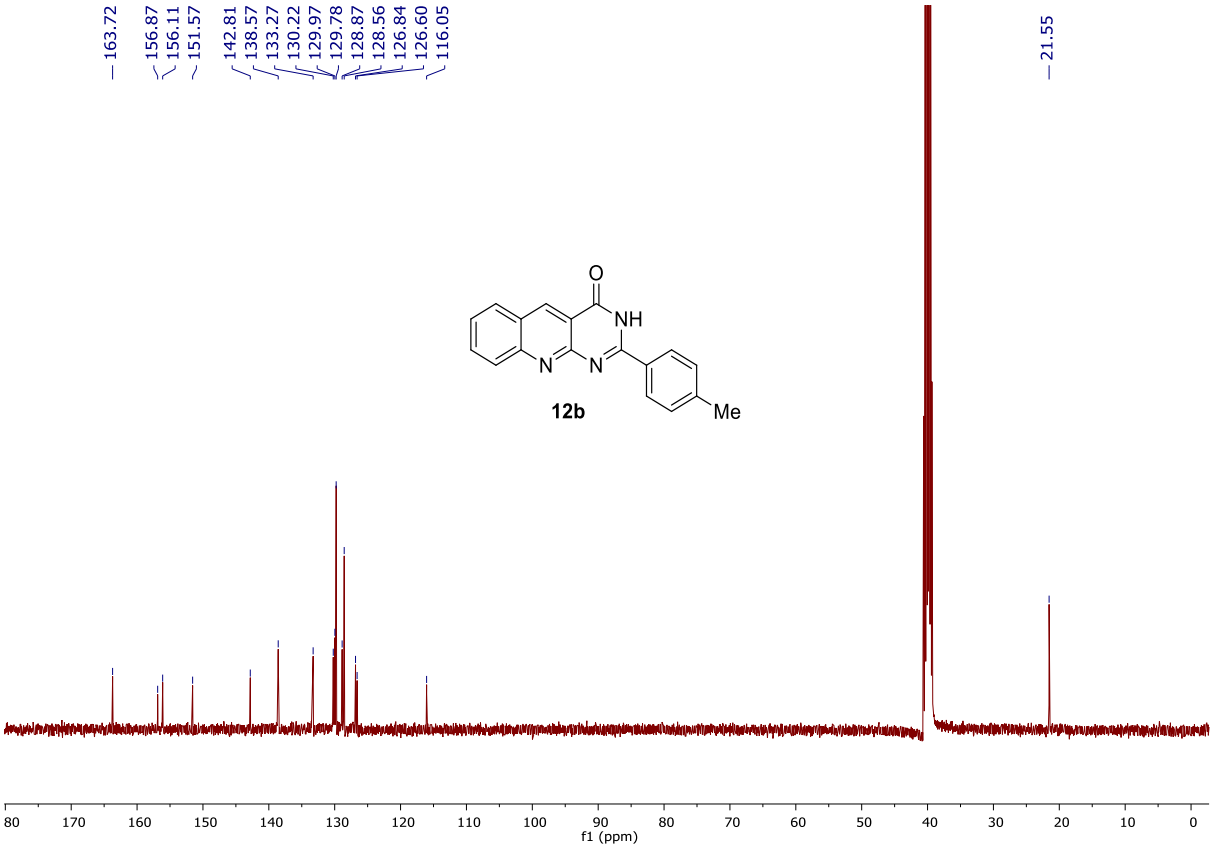
LC-MS of **11**

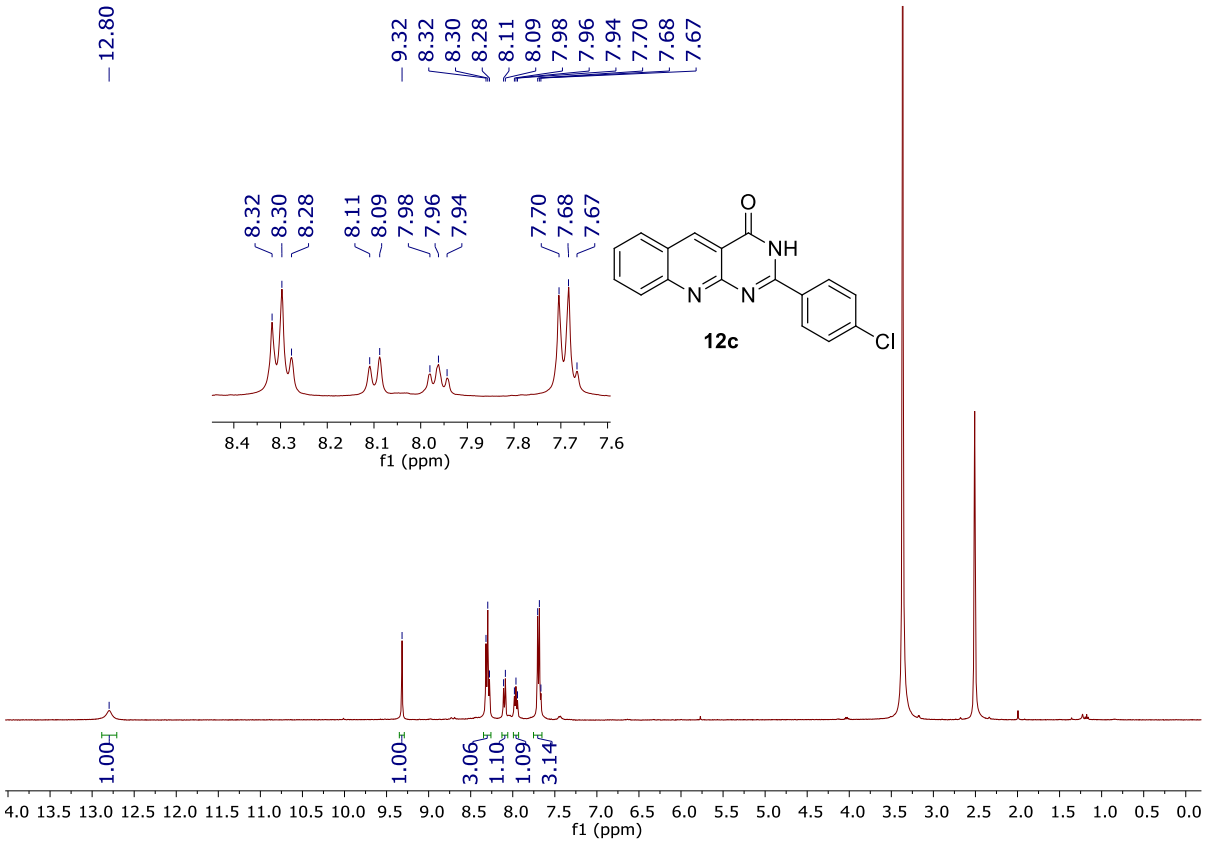


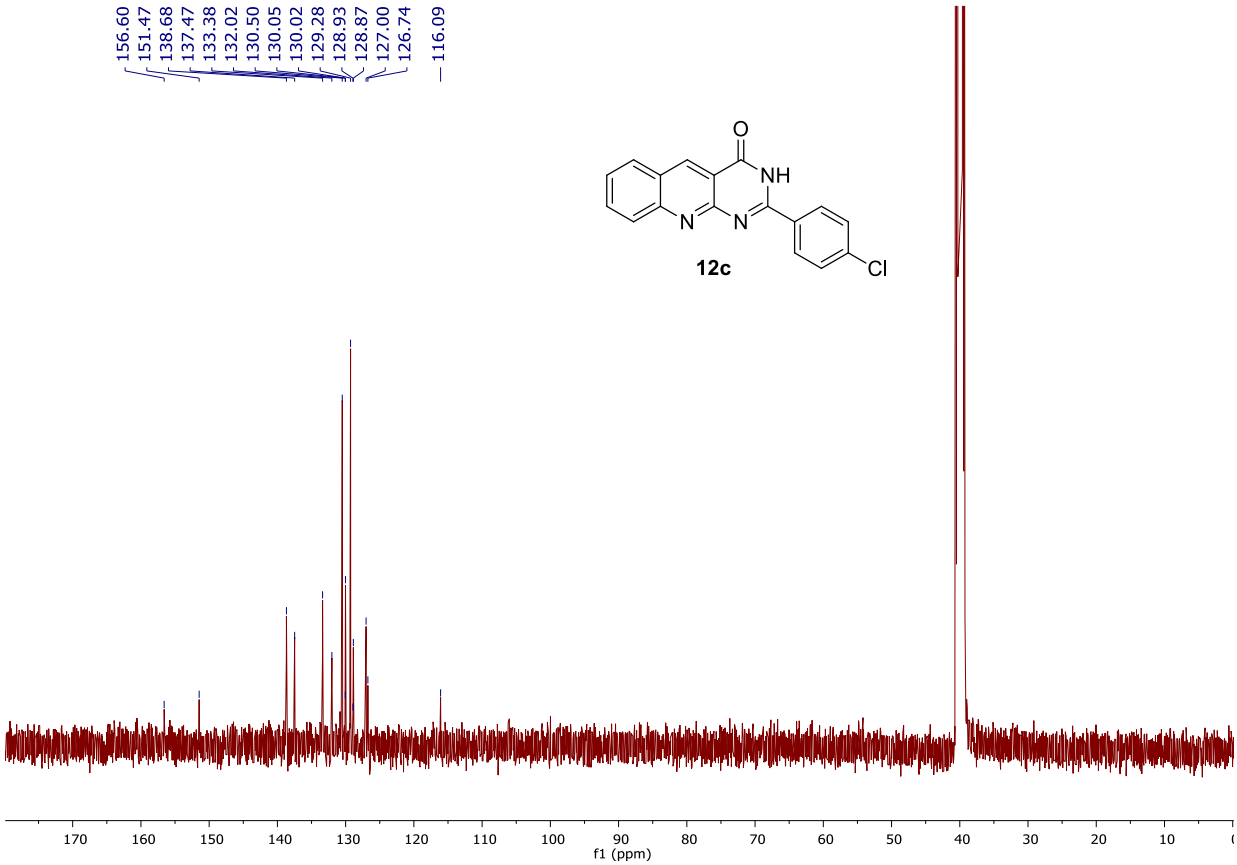

12.71
9.32
8.30
8.29
8.27
8.11
8.09
7.98
7.96
7.94
7.70
7.68
7.67
7.66
7.65
7.63
7.62
7.61
7.60
7.59
O
NH
N
N
12a
1.00
1.00
3.45
1.13
1.13
4.48
f1 (ppm)


163.73
156.81
156.26
151.56
138.65
133.34
133.08
132.58
130.00
129.19
128.91
128.62
126.95
126.68
116.11
O
NH
N
N
12a
f1 (ppm)


12.62
9.29
9.29
8.27
8.25
8.21
8.19
8.09
8.07
7.96
7.94
7.93
7.68
7.66
7.64
7.42
7.40
2.42
NH
N
N
O
Me
12b
f1 (ppm)
1.00
0.91
1.02
2.00
1.03
1.03
1.07
1.98
2.96


163.72
156.87
156.11
151.57
142.81
138.57
133.27
130.22
129.97
129.78
128.87
128.56
126.84
126.60
116.05
21.55
NH
N
N
O
Me
12b
f1 (ppm)


12.80
9.32
8.32
8.30
8.28
8.11
8.09
7.98
7.96
7.94
7.70
7.68
7.67
O
NH
N
N
Cl
12c
f1 (ppm)
1.00
1.00
3.06
1.10
1.09
3.14


156.60
151.47
138.68
137.47
133.38
132.02
130.50
130.05
130.02
129.28
128.93
128.87
127.00
126.74
116.09
O
NH
N
N
Cl
12c
f1 (ppm)

Crystal Structure Report for 10 (CCDC 1433055)

Table 1. Sample and crystal data

Identification code	vbchem114	
Chemical formula	CHBrNO	
Formula weight	122.94 g/mol	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.9523(11) Å	α = 83.854(10)°
	b = 10.8955(15) Å	β = 89.882(9)°
	c = 13.2918(18) Å	γ = 89.928(9)°
Volume	1289.0(3) $Å^3$	
Z	16	
Density (calculated)	2.534 g/cm^3	
Absorption coefficient	12.492 mm^{-1}	
F(000)	912	

Table 2. Data collection and structure refinement

Theta range for data collection	1.54 to 28.89°	
Index ranges	-12<=h<=12, -14<=k<=14, -17<=l<=17	
Reflections collected	41097	
Independent reflections	6642 [R(int) = 0.3862]	
Coverage of independent reflections	97.9%	
Absorption correction	multi-scan	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2014/7 (Sheldrick, 2014)	
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	6642 / 0 / 343	
Goodness-of-fit on F^2	1.410	
Final R indices	2552 data; I>2σ(I)	R1 = 0.1575, wR2 = 0.2795
	all data	R1 = 0.3435, wR2 = 0.3407
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.1000P)^2+0.6035P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	1.172 and -2.442 $eÅ^{-3}$	
R.M.S. deviation from mean	0.342 $eÅ^{-3}$	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2)

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
Br1	0.60369(15)	0.69560(14)	0.83172(11)	0.0500(5)
Br2	0.10329(15)	0.80468(14)	0.66867(11)	0.0506(5)
O1	0.9436(9)	0.6540(9)	0.9648(7)	0.054(3)
O2	0.4430(9)	0.8458(9)	0.5355(7)	0.051(3)
C26	0.8130(12)	0.9171(10)	0.5811(8)	0.023(3)
C11	0.4127(12)	0.4928(11)	0.8810(8)	0.025(3)
C24	0.6670(11)	0.9044(10)	0.5990(8)	0.022(3)
C27	0.9104(11)	0.0081(10)	0.6184(8)	0.021(3)
N2	0.5303(12)	0.0301(11)	0.7224(9)	0.053(3)
C10	0.3134(12)	0.5834(10)	0.9178(8)	0.025(3)
C28	0.0510(13)	0.9716(10)	0.6573(9)	0.027(3)
C1	0.1367(13)	0.7953(11)	0.9798(9)	0.031(3)
C6	0.2482(13)	0.8594(12)	0.9246(10)	0.037(3)
C25	0.5917(14)	0.9761(13)	0.6660(10)	0.040(3)
C7	0.0678(14)	0.6820(12)	0.9475(9)	0.034(3)
C8	0.1679(11)	0.5961(10)	0.9002(8)	0.022(3)
C12	0.5517(13)	0.5272(11)	0.8441(8)	0.027(3)
N1	0.0322(13)	0.4726(12)	0.7763(10)	0.058(4)
C16	0.3682(14)	0.3704(12)	0.8881(9)	0.038(3)
C29	0.1508(13)	0.0522(13)	0.6904(9)	0.037(3)
C9	0.0936(14)	0.5236(13)	0.8350(11)	0.042(3)
C32	0.8698(14)	0.1305(12)	0.6139(9)	0.039(3)
C18	0.7488(14)	0.6418(12)	0.5745(10)	0.039(3)
C23	0.5687(14)	0.8178(11)	0.5518(9)	0.033(3)
C31	0.9671(15)	0.2133(13)	0.6487(9)	0.040(3)
C17	0.6362(12)	0.7056(12)	0.5193(9)	0.030(3)
C15	0.4680(15)	0.2877(13)	0.8525(10)	0.044(3)
C13	0.6516(14)	0.4463(14)	0.8084(10)	0.041(3)
C30	0.1078(16)	0.1758(15)	0.6855(10)	0.053(4)
C2	0.0718(15)	0.8384(14)	0.0669(10)	0.048(4)

	x/a	y/b	z/c	U(eq)
C22	0.5754(15)	0.6607(13)	0.4332(10)	0.046(4)
C14	0.6106(16)	0.3280(15)	0.8148(9)	0.049(4)
C5	0.3053(15)	0.9665(12)	0.9598(12)	0.047(4)
C21	0.6369(18)	0.5529(14)	0.4033(11)	0.056(4)
C4	0.2500(18)	0.0126(14)	0.0420(13)	0.058(4)
C20	0.7503(18)	0.4885(14)	0.4563(12)	0.052(4)
C3	0.1345(18)	0.9483(16)	0.0979(12)	0.062(5)
C19	0.8027(16)	0.5314(13)	0.5392(12)	0.054(4)

Table 4. Bond lengths (Å).

Br1-C12	1.884(11)	Br2-C28	1.868(11)
O1-C7	1.169(13)	O2-C23	1.179(14)
C26-C24	1.333(14)	C26-C27	1.449(15)
C11-C12	1.374(15)	C11-C16	1.387(17)
C11-C10	1.451(15)	C24-C25	1.415(17)
C24-C23	1.480(16)	C27-C32	1.377(16)
C27-C28	1.403(16)	N2-C25	1.141(16)
C10-C8	1.328(15)	C28-C29	1.360(17)
C1-C6	1.382(16)	C1-C2	1.418(17)
C1-C7	1.485(17)	C6-C5	1.400(18)
C7-C8	1.482(16)	C8-C9	1.403(18)
C12-C13	1.374(17)	N1-C9	1.147(17)
C16-C15	1.386(17)	C29-C30	1.40(2)
C32-C31	1.371(17)	C18-C17	1.390(17)
C18-C19	1.420(18)	C23-C17	1.468(16)
C31-C30	1.396(19)	C17-C22	1.403(17)
C15-C14	1.423(19)	C13-C14	1.33(2)
C2-C3	1.42(2)	C22-C21	1.39(2)
C5-C4	1.34(2)	C21-C20	1.38(2)
C4-C3	1.41(2)	C20-C19	1.33(2)

Table 5. Bond angles (°)

C24-C26-C27	126.3(10)	C12-C11-C16	120.6(11)
C12-C11-C10	120.3(10)	C16-C11-C10	119.1(10)
C26-C24-C25	121.6(10)	C26-C24-C23	124.6(10)
C25-C24-C23	113.8(10)	C32-C27-C28	119.2(10)
C32-C27-C26	121.5(10)	C28-C27-C26	119.3(10)
C8-C10-C11	127.0(10)	C29-C28-C27	122.9(11)
C29-C28-Br2	117.8(10)	C27-C28-Br2	119.3(9)
C6-C1-C2	122.4(12)	C6-C1-C7	122.4(11)
C2-C1-C7	115.1(11)	C1-C6-C5	118.6(12)
N2-C25-C24	177.6(15)	O1-C7-C8	119.6(11)
O1-C7-C1	123.2(11)	C8-C7-C1	117.0(10)
C10-C8-C9	121.4(10)	C10-C8-C7	125.2(10)
C9-C8-C7	113.4(10)	C13-C12-C11	123.4(11)
C13-C12-Br1	117.6(9)	C11-C12-Br1	118.8(9)
C15-C16-C11	116.6(12)	C28-C29-C30	117.2(12)
N1-C9-C8	174.5(16)	C31-C32-C27	119.1(13)
C17-C18-C19	117.6(12)	O2-C23-C17	123.0(11)
O2-C23-C24	118.8(11)	C17-C23-C24	118.0(10)
C32-C31-C30	121.0(13)	C18-C17-C22	121.0(12)
C18-C17-C23	121.9(11)	C22-C17-C23	117.0(11)
C16-C15-C14	120.6(13)	C14-C13-C12	116.7(12)
C29-C30-C31	120.6(12)	C1-C2-C3	115.9(13)
C21-C22-C17	117.3(13)	C13-C14-C15	122.0(12)
C4-C5-C6	122.3(13)	C20-C21-C22	122.7(13)
C5-C4-C3	119.2(13)	C19-C20-C21	118.6(14)
C4-C3-C2	121.4(13)	C20-C19-C18	122.7(14)

Table 6. Anisotropic atomic displacement parameters (Å^2).

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	0.0527(9)	0.0466(10)	0.0521(10)	-0.0116(8)	0.0168(7)	-0.0195(7)
Br2	0.0509(9)	0.0492(10)	0.0538(10)	-0.0156(8)	-0.0135(7)	0.0197(7)
O1	0.022(5)	0.073(7)	0.073(7)	-0.028(6)	0.010(4)	-0.011(4)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O2	0.022(5)	0.065(7)	0.070(7)	-0.026(6)	-0.009(4)	0.014(4)
C26	0.029(6)	0.017(6)	0.023(6)	-0.007(5)	-0.002(5)	0.004(5)
C11	0.028(6)	0.030(8)	0.019(6)	-0.011(5)	0.001(5)	-0.004(5)
C24	0.029(6)	0.019(7)	0.020(6)	-0.012(5)	0.002(5)	0.005(5)
C27	0.024(6)	0.022(7)	0.019(6)	-0.013(5)	0.006(4)	0.003(5)
N2	0.042(7)	0.058(9)	0.064(9)	-0.033(7)	0.015(6)	0.004(6)
C10	0.038(7)	0.017(7)	0.022(7)	-0.006(5)	0.011(5)	-0.005(5)
C28	0.045(7)	0.012(6)	0.025(7)	-0.003(5)	0.014(5)	0.006(5)
C1	0.037(7)	0.034(8)	0.024(7)	-0.016(6)	-0.006(5)	0.009(6)
C6	0.034(7)	0.029(8)	0.046(8)	0.001(7)	0.009(6)	0.001(6)
C25	0.036(7)	0.048(10)	0.036(8)	-0.005(7)	-0.006(6)	-0.014(6)
C7	0.038(8)	0.032(8)	0.035(8)	-0.017(6)	0.001(6)	-0.001(6)
C8	0.025(6)	0.018(7)	0.022(6)	-0.005(5)	-0.001(5)	-0.004(5)
C12	0.043(7)	0.018(7)	0.022(7)	-0.012(5)	0.002(5)	-0.014(5)
N1	0.044(7)	0.065(9)	0.072(9)	-0.039(8)	-0.008(6)	-0.001(6)
C16	0.061(9)	0.024(8)	0.033(8)	-0.017(6)	-0.008(6)	0.005(6)
C29	0.033(7)	0.047(10)	0.033(8)	-0.011(7)	0.000(5)	-0.008(6)
C9	0.041(8)	0.034(9)	0.053(10)	-0.016(8)	0.017(7)	0.010(6)
C32	0.060(9)	0.034(9)	0.025(7)	-0.013(6)	0.016(6)	-0.014(7)
C18	0.042(8)	0.033(8)	0.043(8)	-0.004(7)	0.003(6)	0.005(6)
C23	0.046(8)	0.025(8)	0.028(7)	-0.013(6)	0.004(6)	-0.003(6)
C31	0.050(8)	0.043(9)	0.026(7)	-0.003(7)	-0.001(6)	-0.004(7)
C17	0.031(7)	0.040(8)	0.021(7)	-0.011(6)	0.003(5)	-0.007(6)
C15	0.055(9)	0.048(10)	0.029(8)	-0.010(7)	-0.006(6)	0.009(7)
C13	0.036(7)	0.047(10)	0.043(9)	-0.023(7)	0.002(6)	0.004(6)
C30	0.059(10)	0.069(12)	0.031(8)	-0.011(8)	0.013(7)	-0.037(8)
C2	0.052(9)	0.054(10)	0.041(9)	-0.025(8)	-0.008(6)	0.015(7)
C22	0.057(9)	0.050(10)	0.034(8)	-0.023(7)	0.008(6)	-0.022(7)
C14	0.056(9)	0.066(12)	0.023(8)	-0.005(7)	-0.006(6)	0.033(8)
C5	0.059(9)	0.021(8)	0.064(11)	-0.015(7)	-0.009(7)	-0.009(6)
C21	0.078(11)	0.047(10)	0.048(10)	-0.033(8)	0.015(8)	-0.016(8)
C4	0.076(11)	0.045(11)	0.058(11)	-0.026(9)	-0.014(8)	-0.005(8)
C20	0.077(11)	0.041(10)	0.043(9)	-0.021(8)	0.002(8)	0.004(8)
C3	0.078(11)	0.062(11)	0.052(10)	-0.039(9)	-0.018(8)	0.027(9)
C19	0.067(10)	0.032(9)	0.064(11)	-0.006(8)	0.014(8)	0.006(7)

References:

1. Shen, J.; Yang, D.; Liu, Y.; Qin, S.; Zhang, J.; Sun, J.; Liu, C.; Liu, C.; Zhao, X.; Chu, C.; Liu, R., *Org. Lett.* **2014,** *16*, 350-353.
2. Wang, K.; Nguyen, K.; Huang, Y.; Dömling, A., *J. Comb. Chem.* **2009,** *11*, 920-927.