Supplementary Information

Solution and solid-state fluorescence of 2-(2'-Hydroxyphenyl) 1,5-benzodiazepin-2-one (HBD) borate complexes

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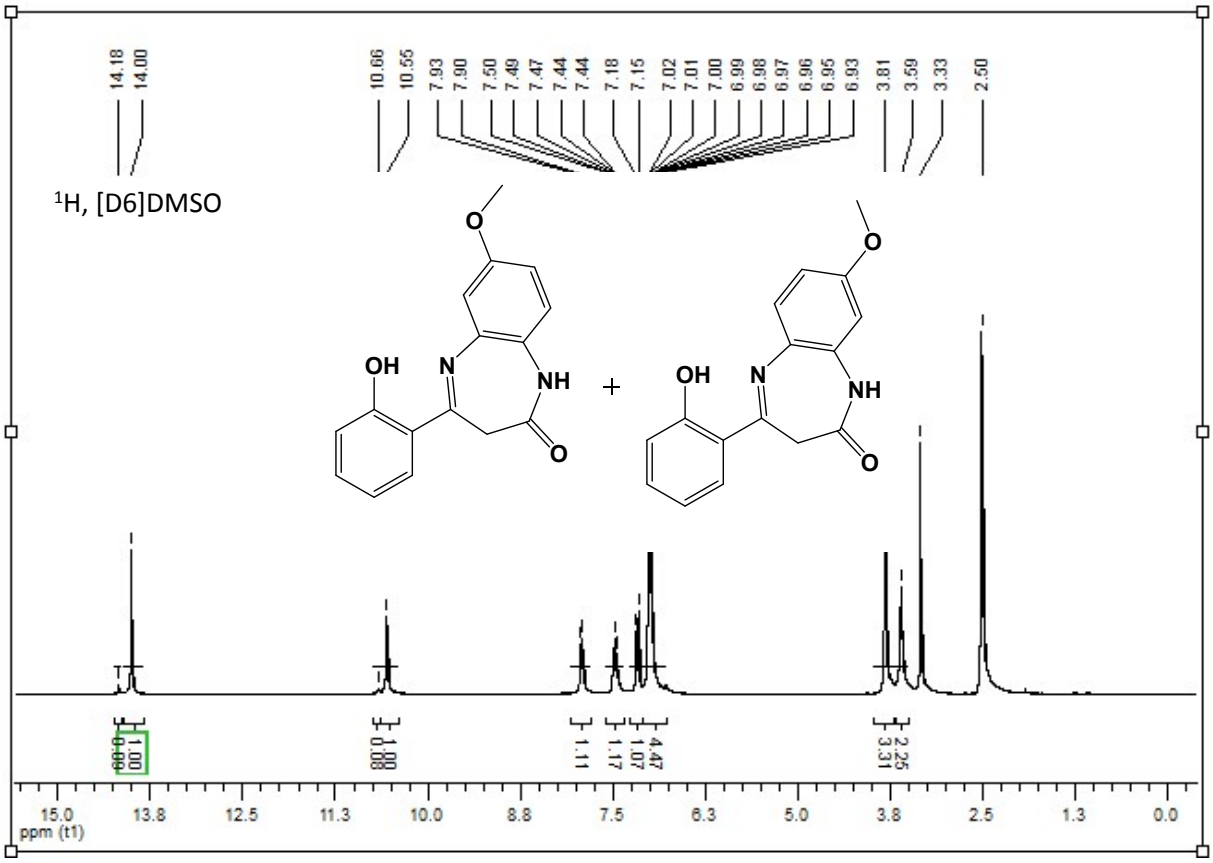
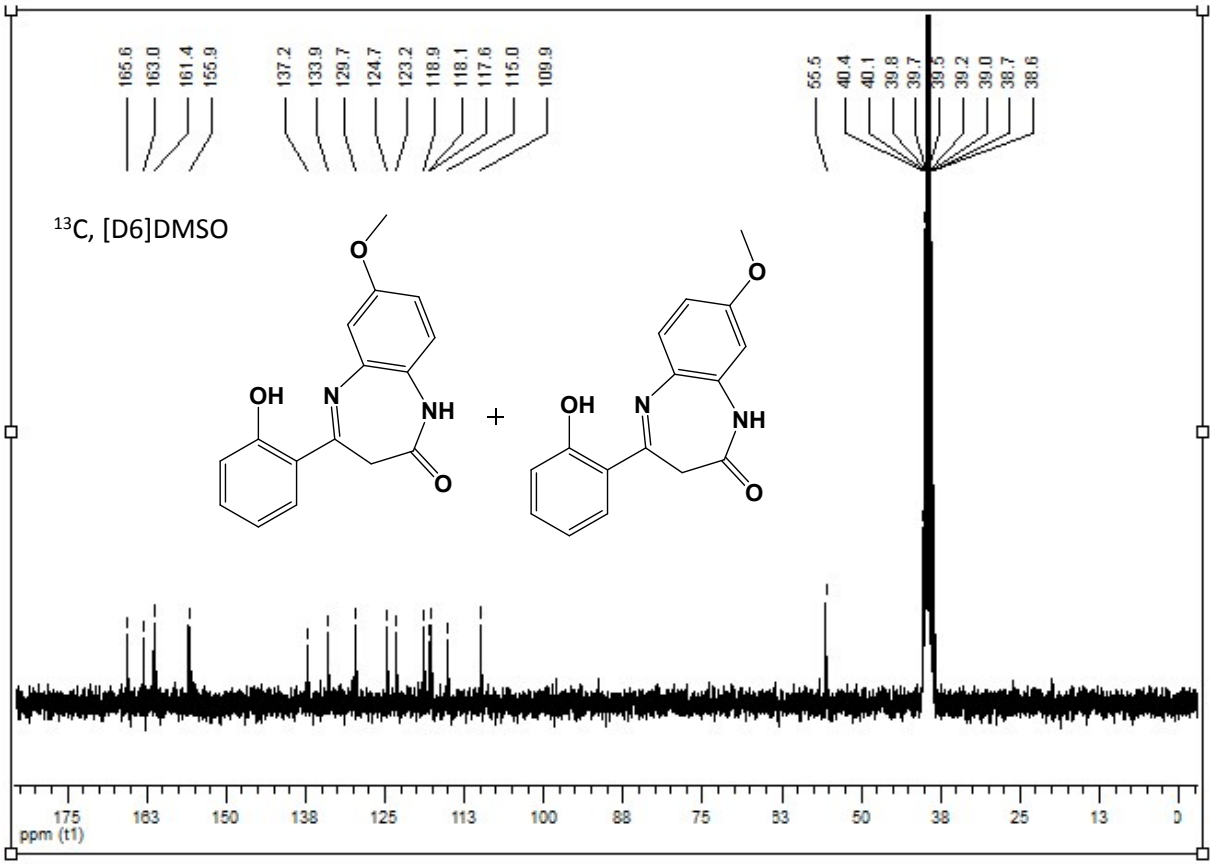
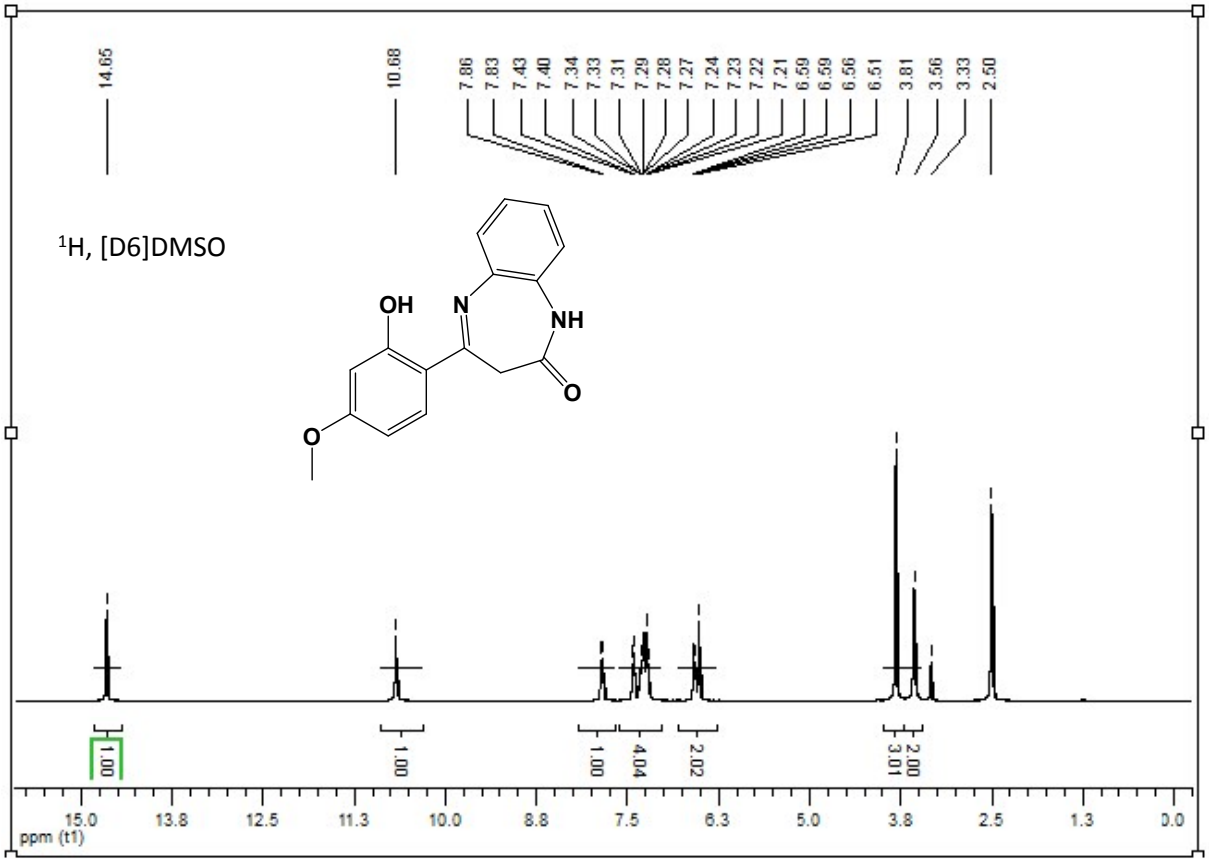
[c.] *Normandie Univ, CNRS, UNIROUEN, INSA Rouen, COBRA (UMR 6014), 76000 Rouen, (France).*

I. Copies of ^{1}H, and ^{13}C NMR



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7.24
7.23
7.22
7.21
6.59
6.59
6.56
6.51
3.81
3.56
3.33
2.50
1H, [D6]DMSO
OH
N
NH
O
O
1.00
1.00
1.00
4.04
2.02
3.01
2.00
15.0
13.8
12.5
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2.5
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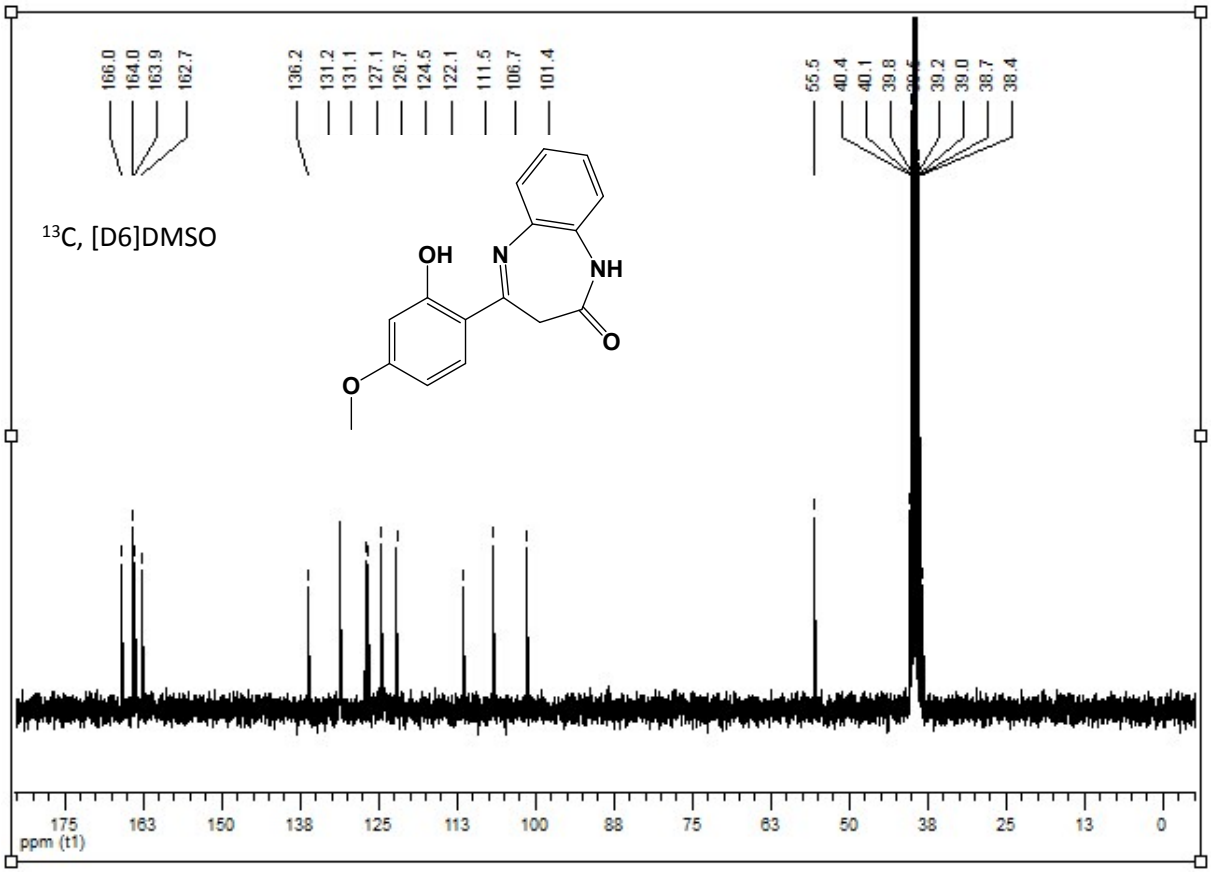
ppm (t1)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127.1
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111.5
106.7
101.4
55.5
40.4
40.1
39.8
39.2
39.0
38.7
38.4
13C, [D6]DMSO
OH
N
NH
O
O
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163
150
138
125
113
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88
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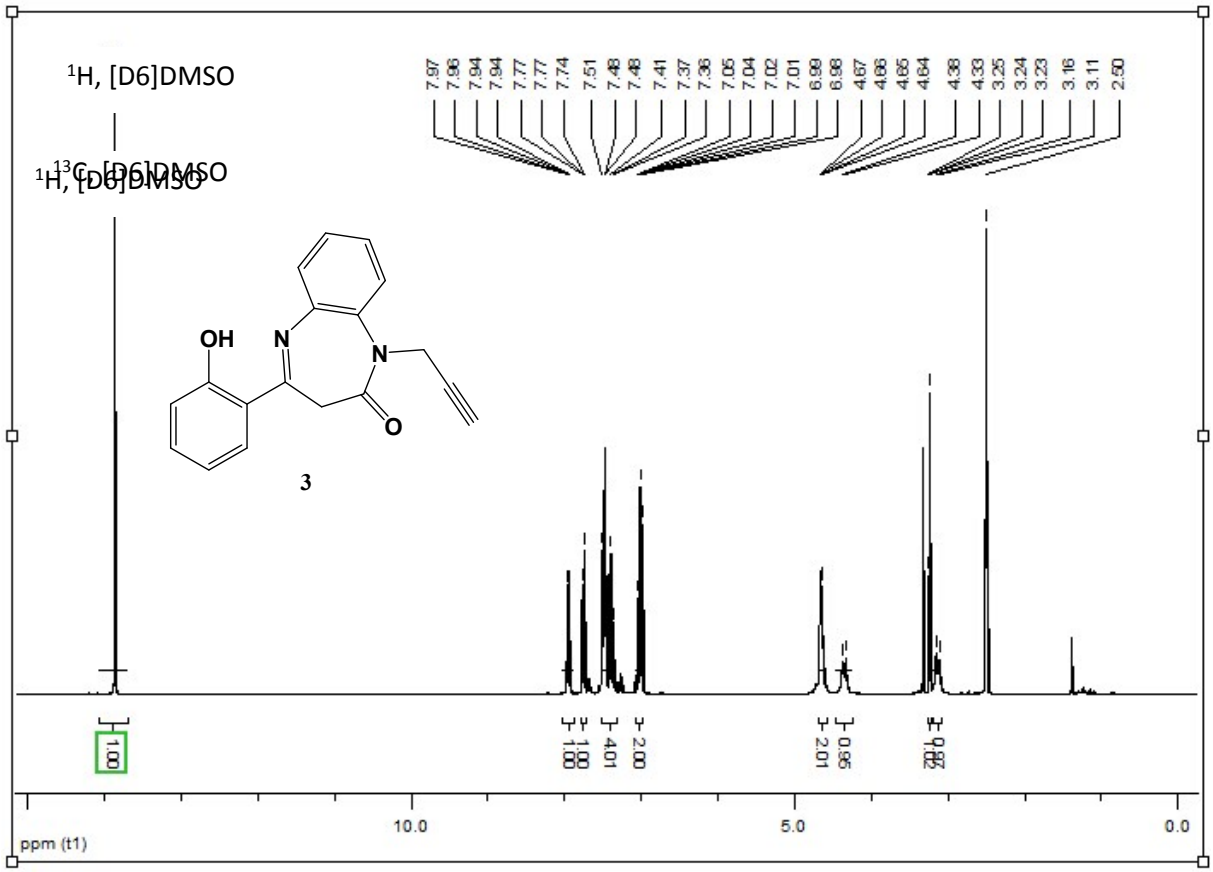
63
50
38
25
13
0
ppm (t1)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^{13}C, [D6]DMSO
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7.51
7.48
7.48
7.41
7.37
7.36
7.05
7.04
7.02
7.01
6.99
6.98
4.67
4.66
4.65
4.64
4.38
4.33
3.25
3.24
3.23
3.16
3.11
2.50
OH
N
N
O
3
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2.01
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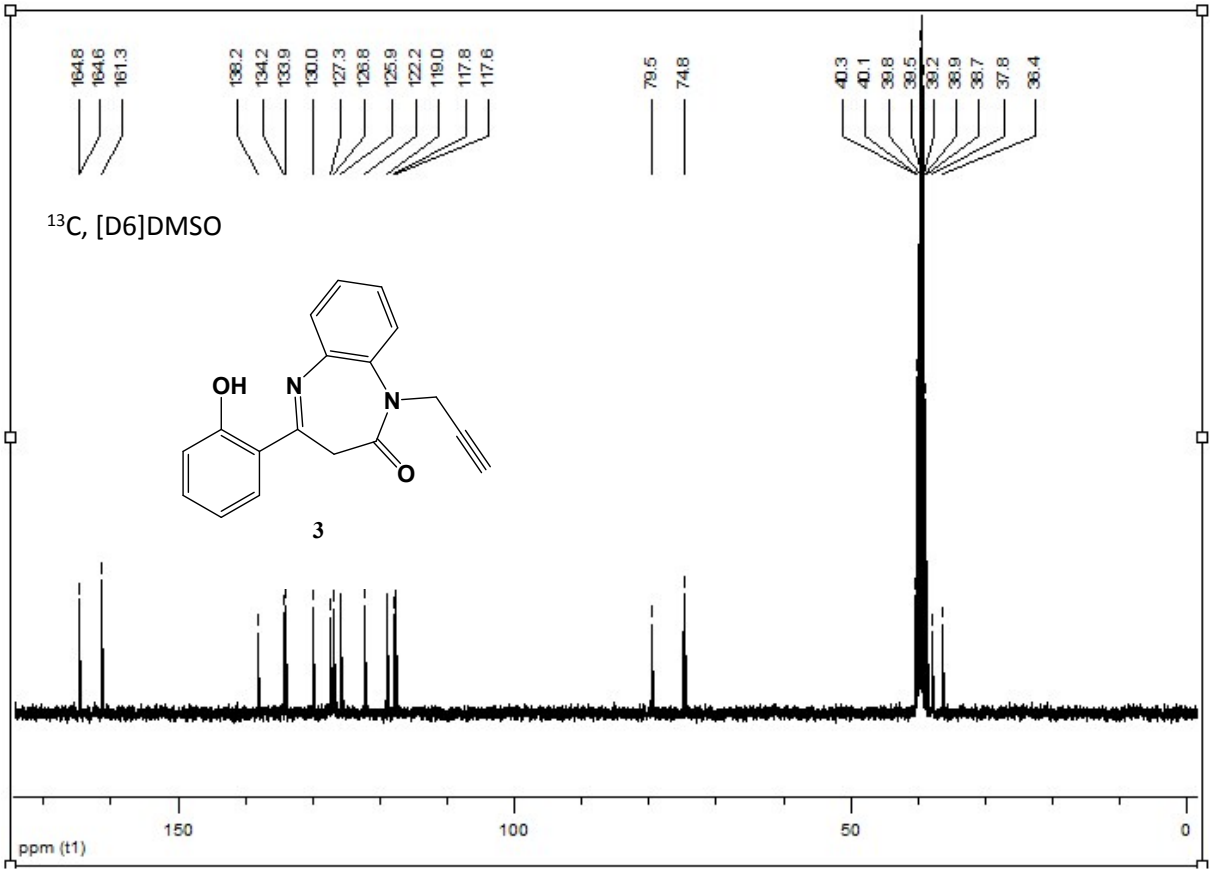
5.0
0.0
ppm (t1)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127.3
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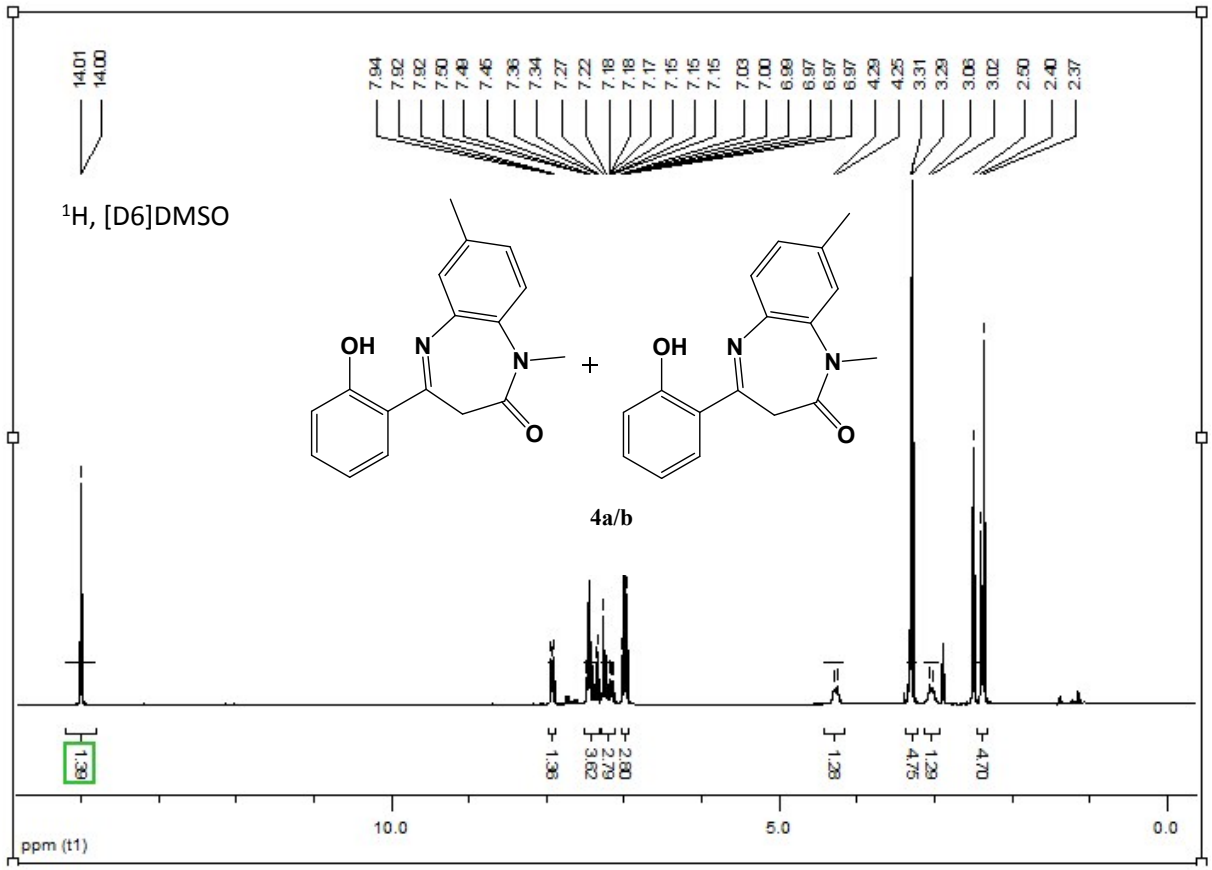
117.8
117.6
79.5
74.8
40.3
40.1
39.8
39.5
39.2
38.9
38.7
37.8
36.4
^{13}C, [D6]DMSO
OH
N
N
O
3
150
100
50
0
ppm (t1)



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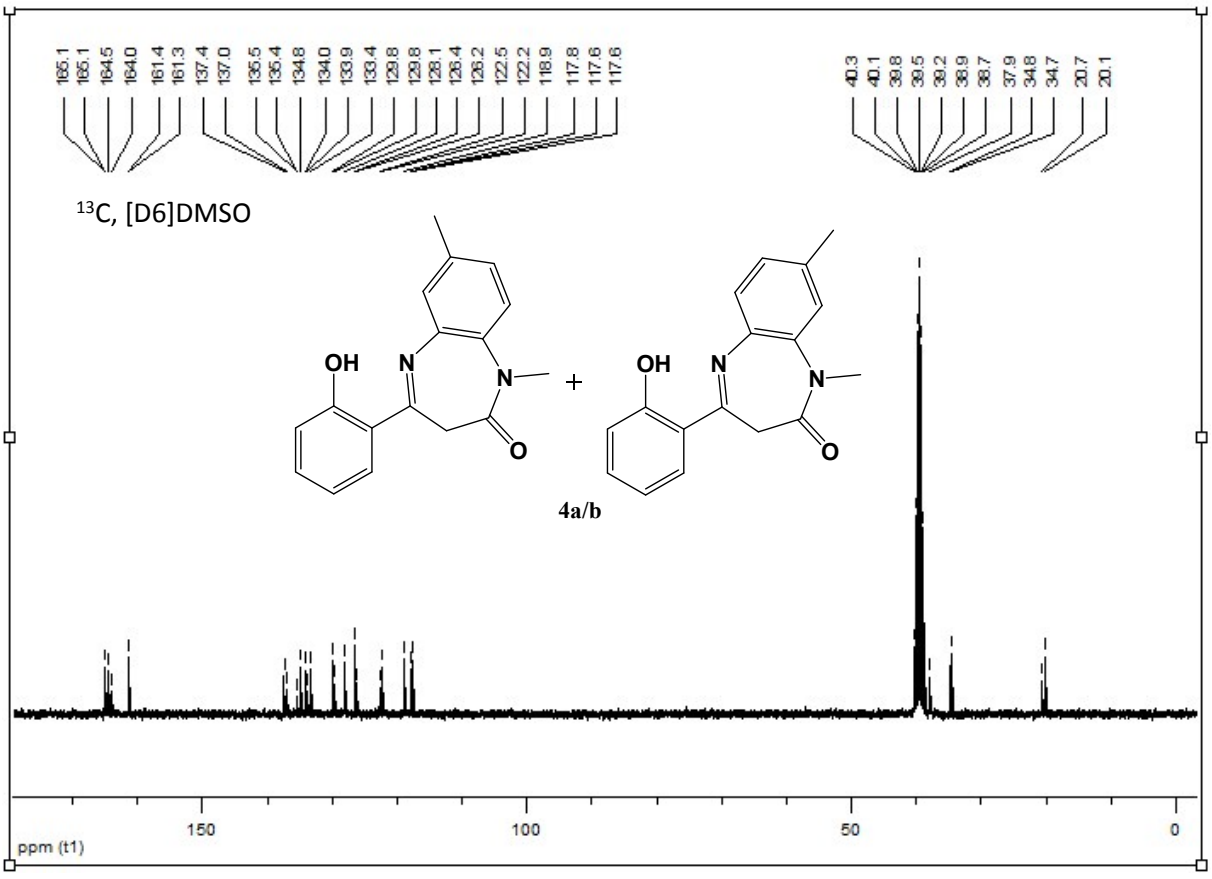
4a/b



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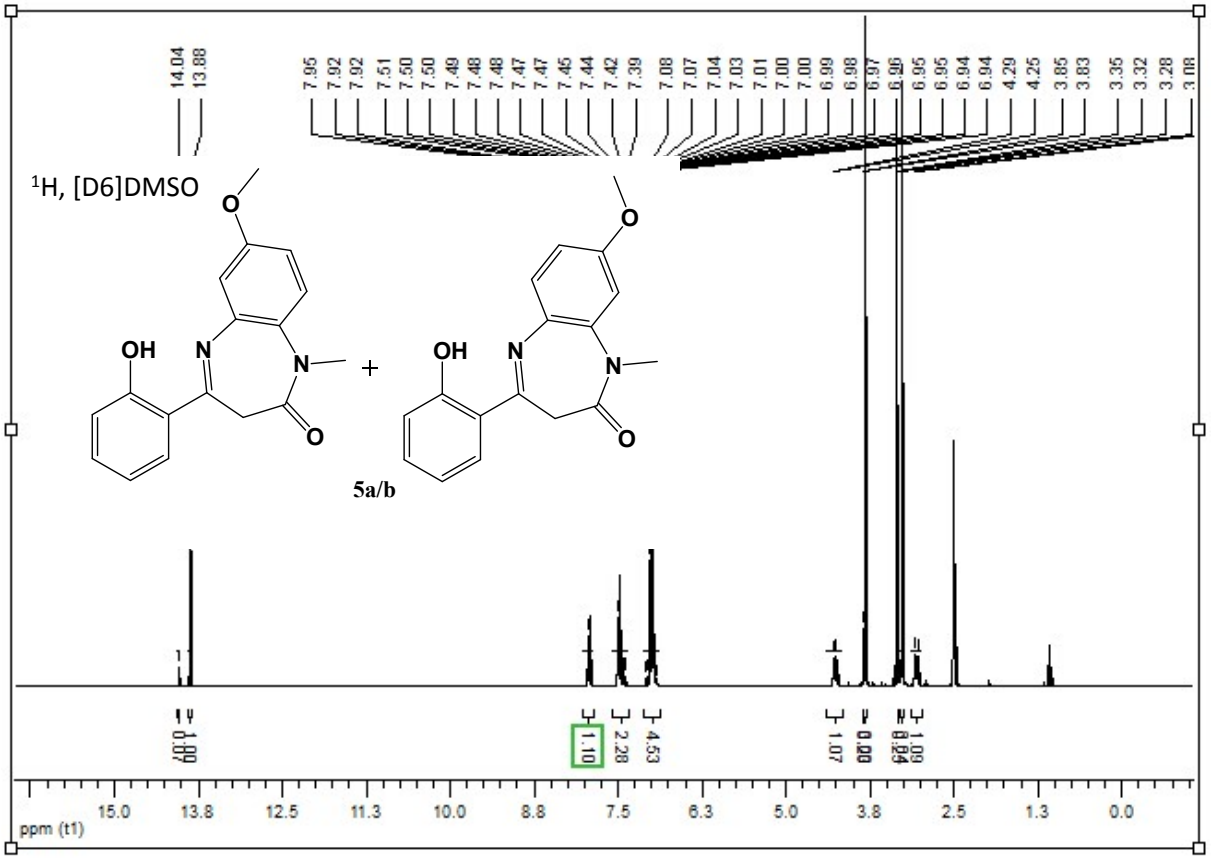
4a/b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7.92
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7.50
7.50
7.49
7.48
7.48
7.47
7.47
7.45
7.44
7.42
7.39
7.08
7.07
7.04
7.03
7.01
7.00
7.00
6.99
6.98
6.97
6.96
6.95
6.95
6.94
6.94
4.29
4.25
3.85
3.83
3.35
3.32
3.28
OH
N
N
O
+
5a/b
1.00
1.10
2.28
4.53
1.07
1.00
8.84
1.09
15.0
13.8
12.5
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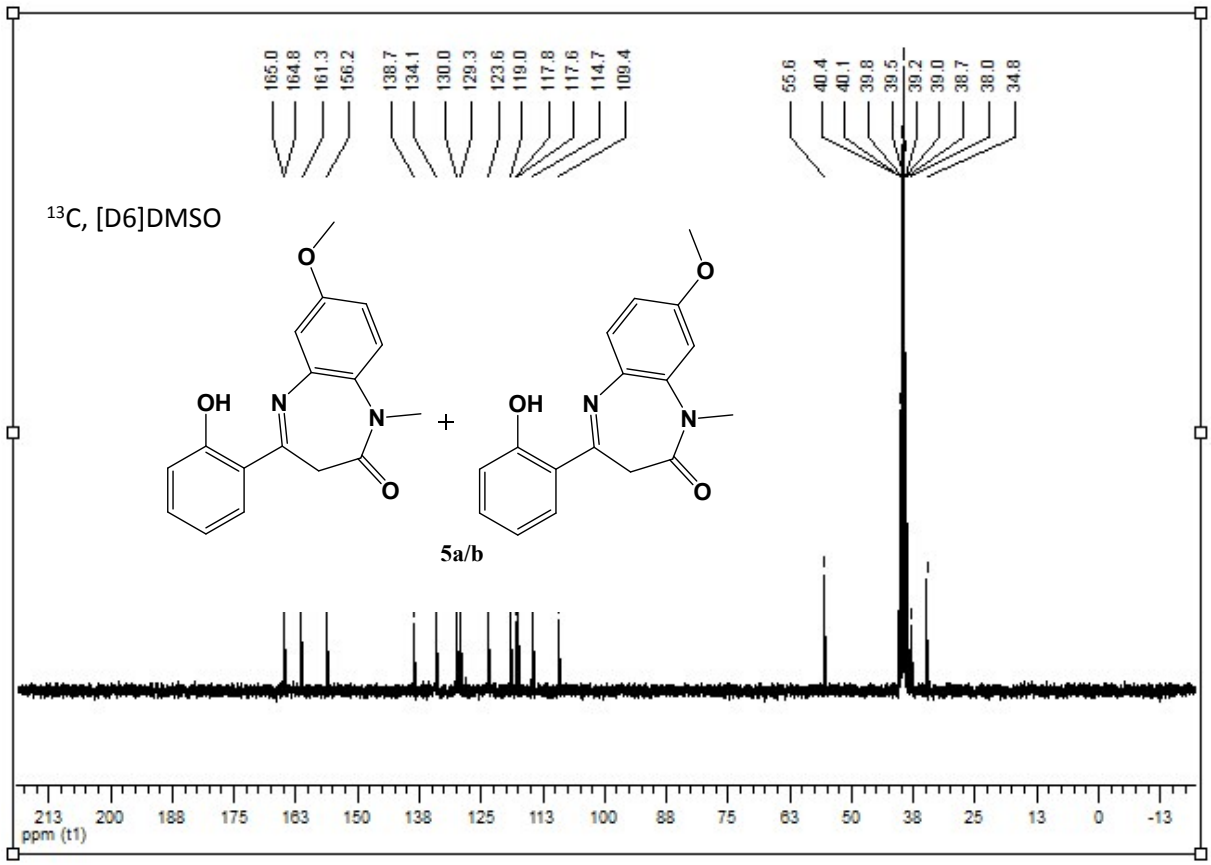
7.5
6.3
5.0
3.8
2.5
1.3
0.0
ppm (t1)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164.8
161.3
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130.0
129.3
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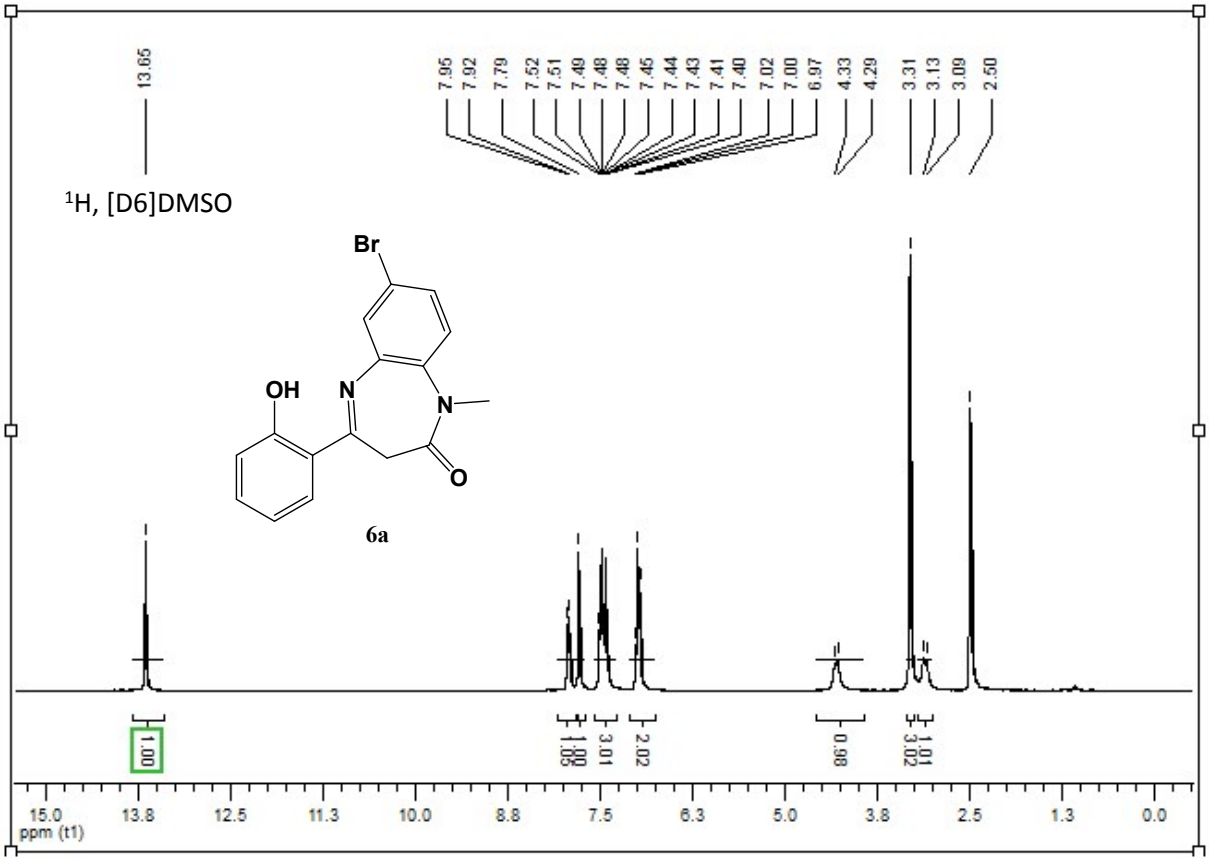
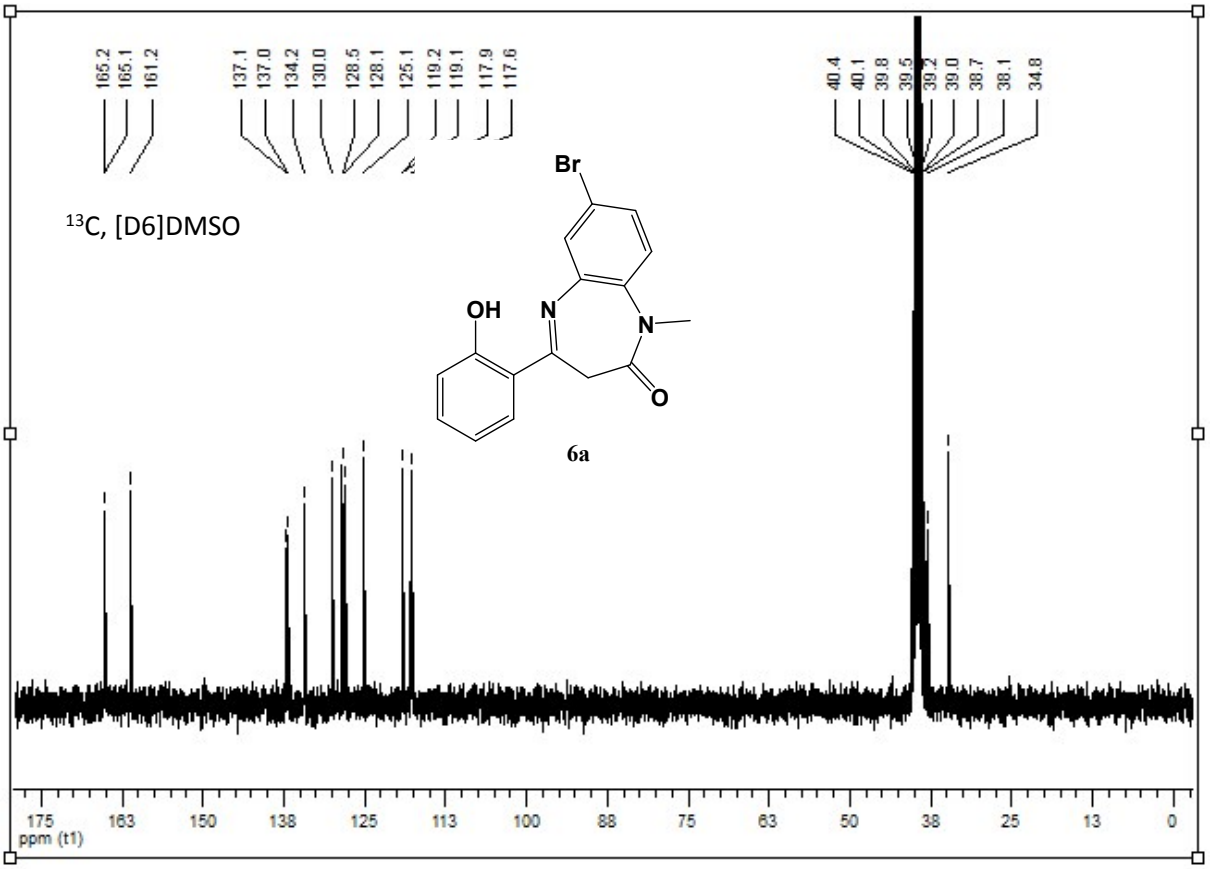
119.0
117.8
117.6
114.7
109.4
55.6
40.4
40.1
39.8
39.5
39.2
39.0
38.7
38.0
34.8
OH
N
N
O
+
5a/b
213
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
-13
ppm (t1)



^{1}H, [D6]DMSO

6a



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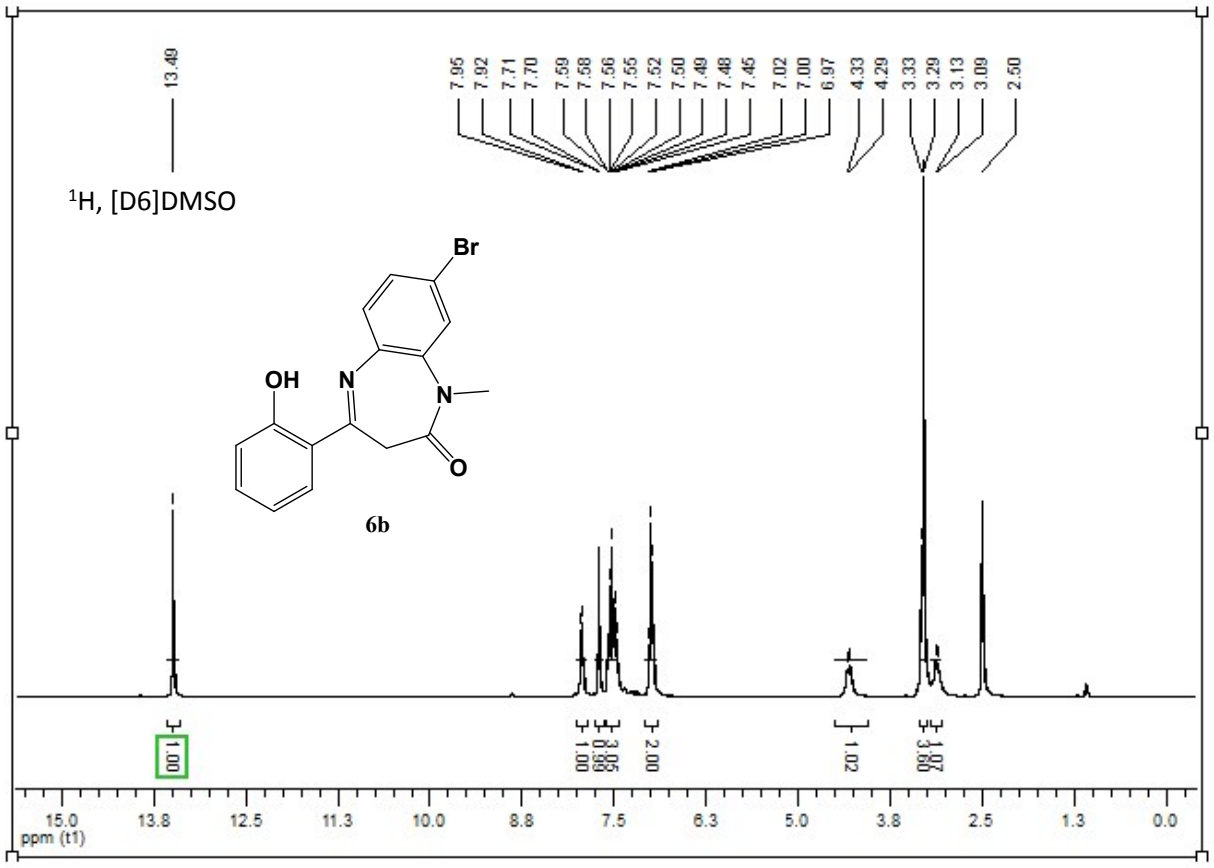
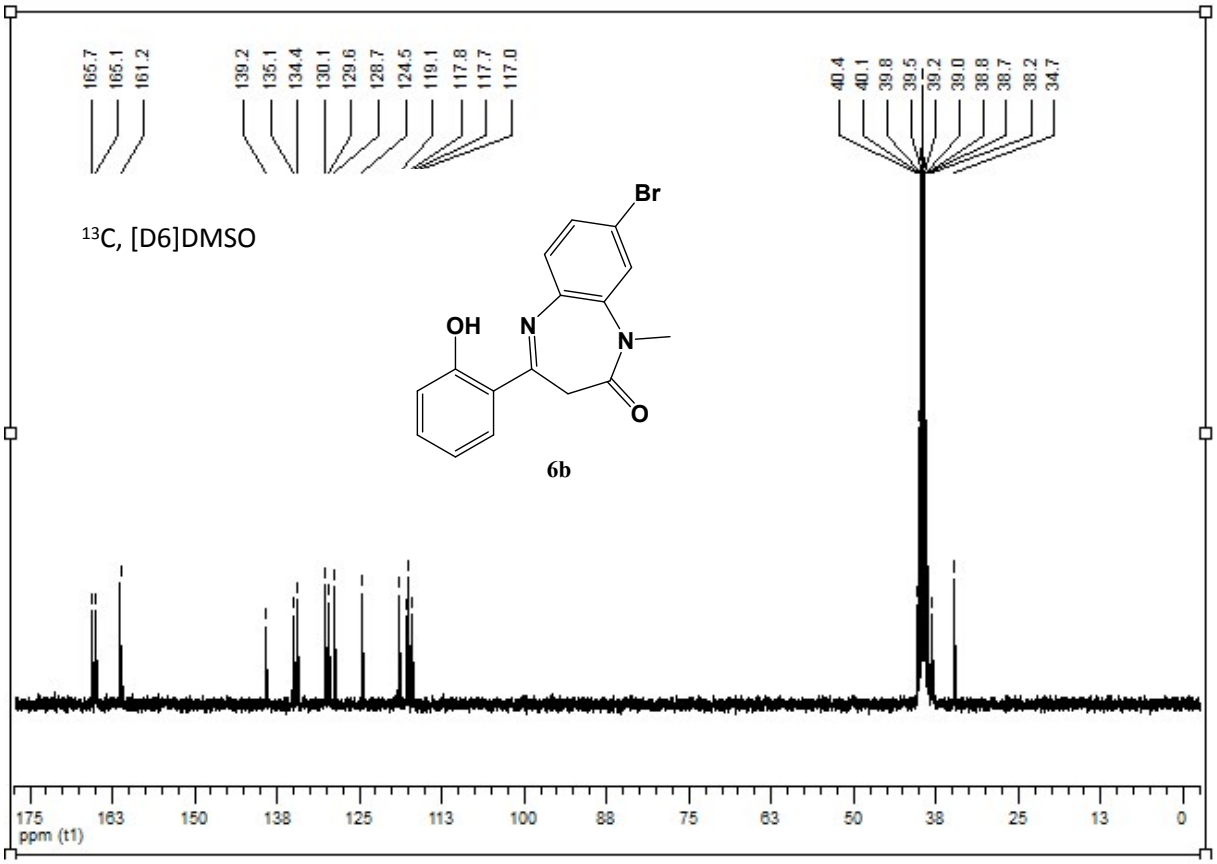
6a



^{1}H, [D6]DMSO

6b



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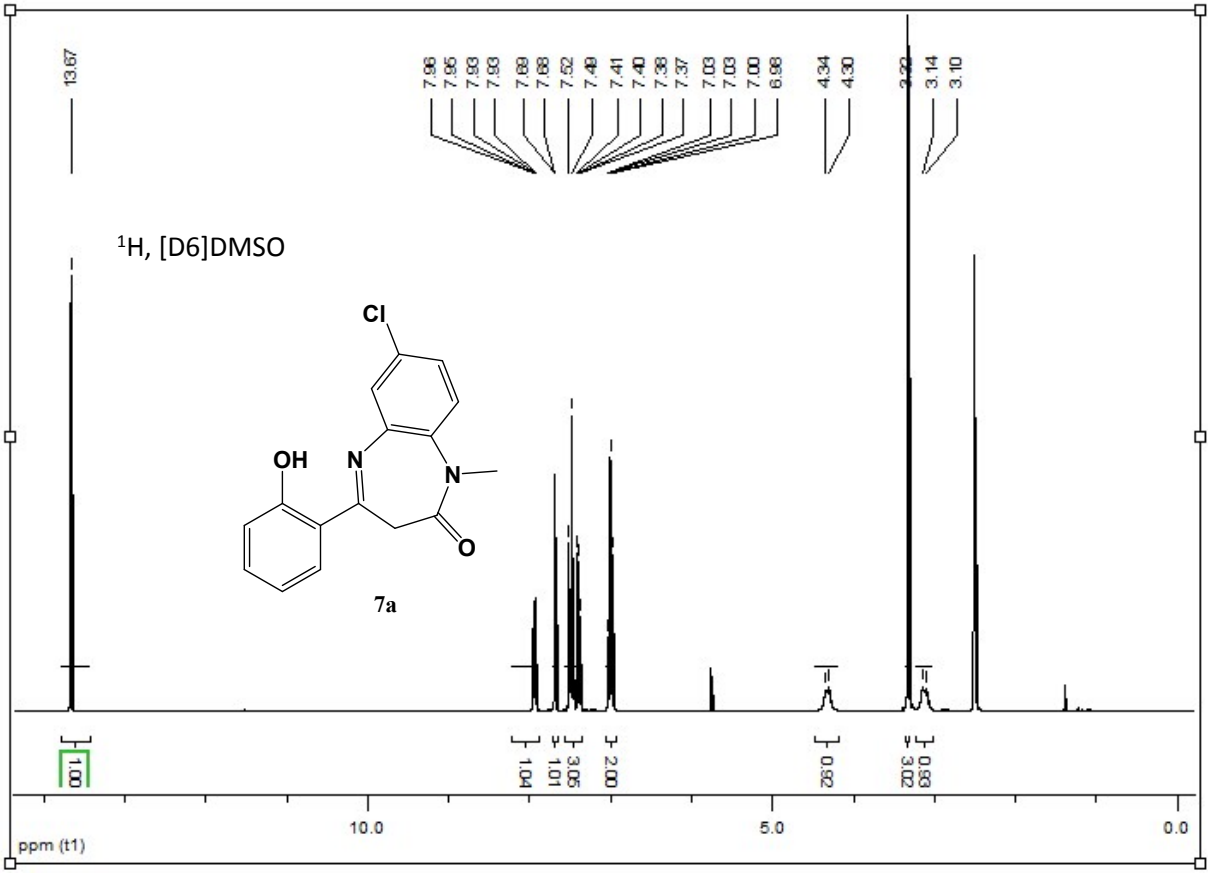
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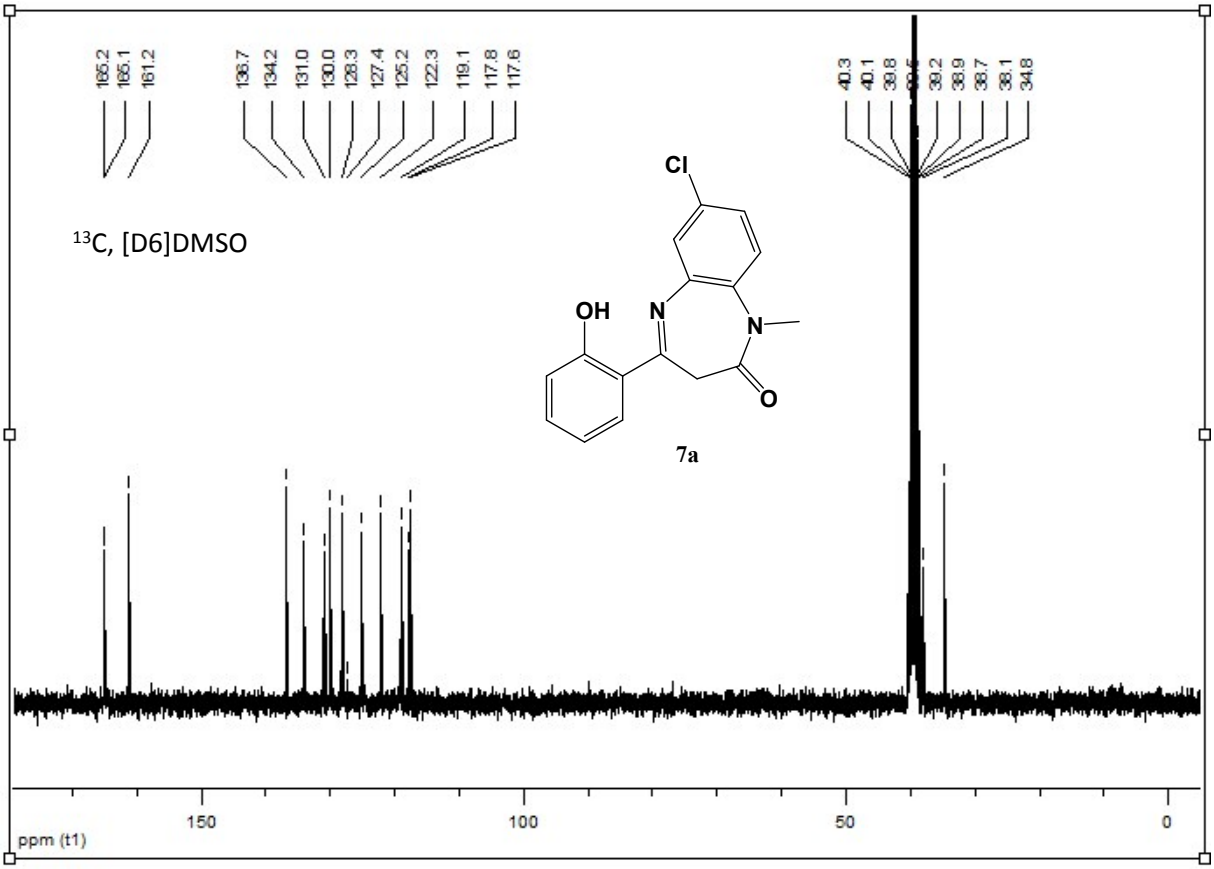
7.41
7.40
7.38
7.37
7.03
7.03
7.00
6.98
4.34
4.30
3.22
3.14
3.10
1H, [D6]DMSO
Cl
OH
N
N
O
7a
1.00
1.04
3.06
1.01
2.00
0.92
3.02
0.99
10.0
5.0
0.0
ppm (t1)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127.4
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122.3
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117.8
117.6
40.3
40.1
39.8
39.5
39.2
38.9
38.7
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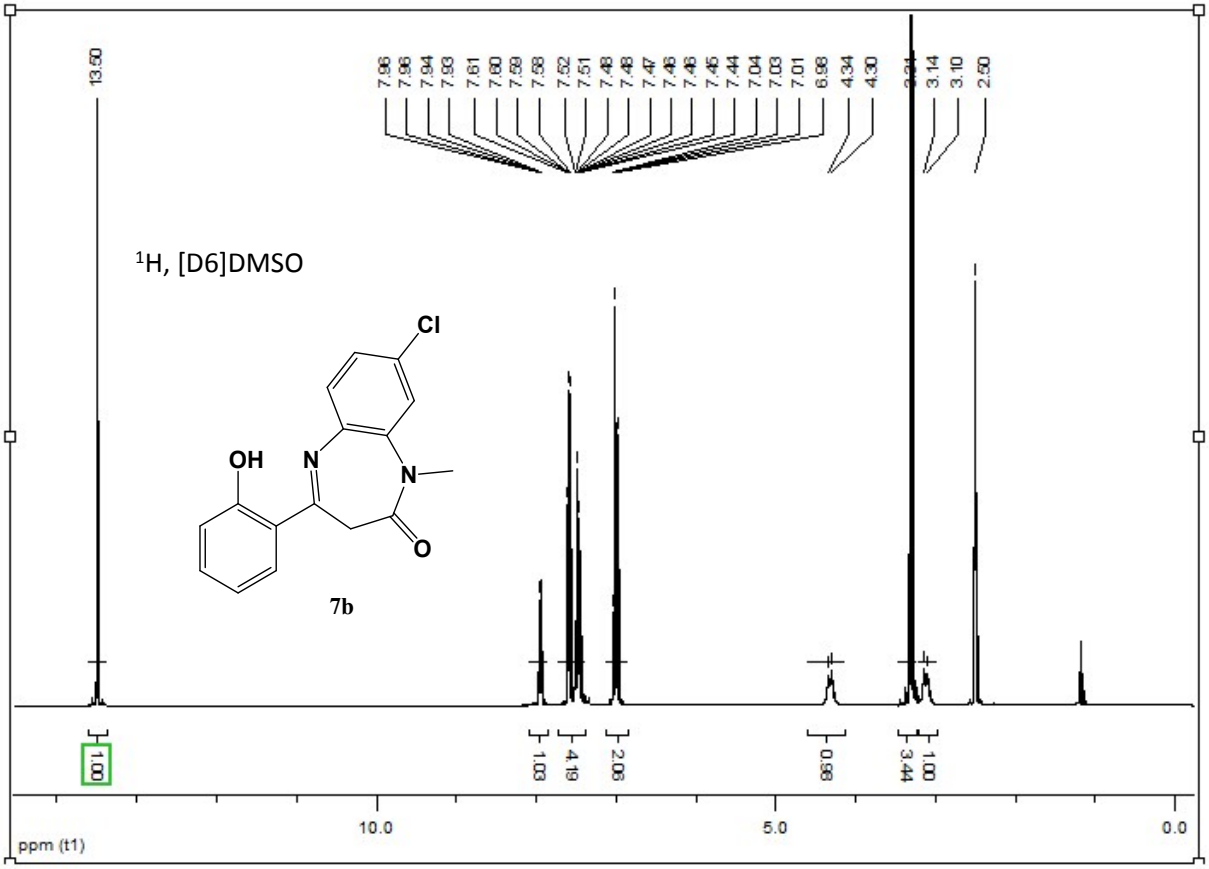
34.8
13C, [D6]DMSO
Cl
OH
N
N
O
7a
150
100
50
0
ppm (t1)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7.59
7.58
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7.51
7.48
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7.47
7.46
7.46
7.45
7.44
7.04
7.03
7.01
6.98
4.34
4.30
3.31
3.14
3.10
2.50
1H, [D6]DMSO
Cl
OH
N
N
O
7b
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1.03
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2.08
0.80
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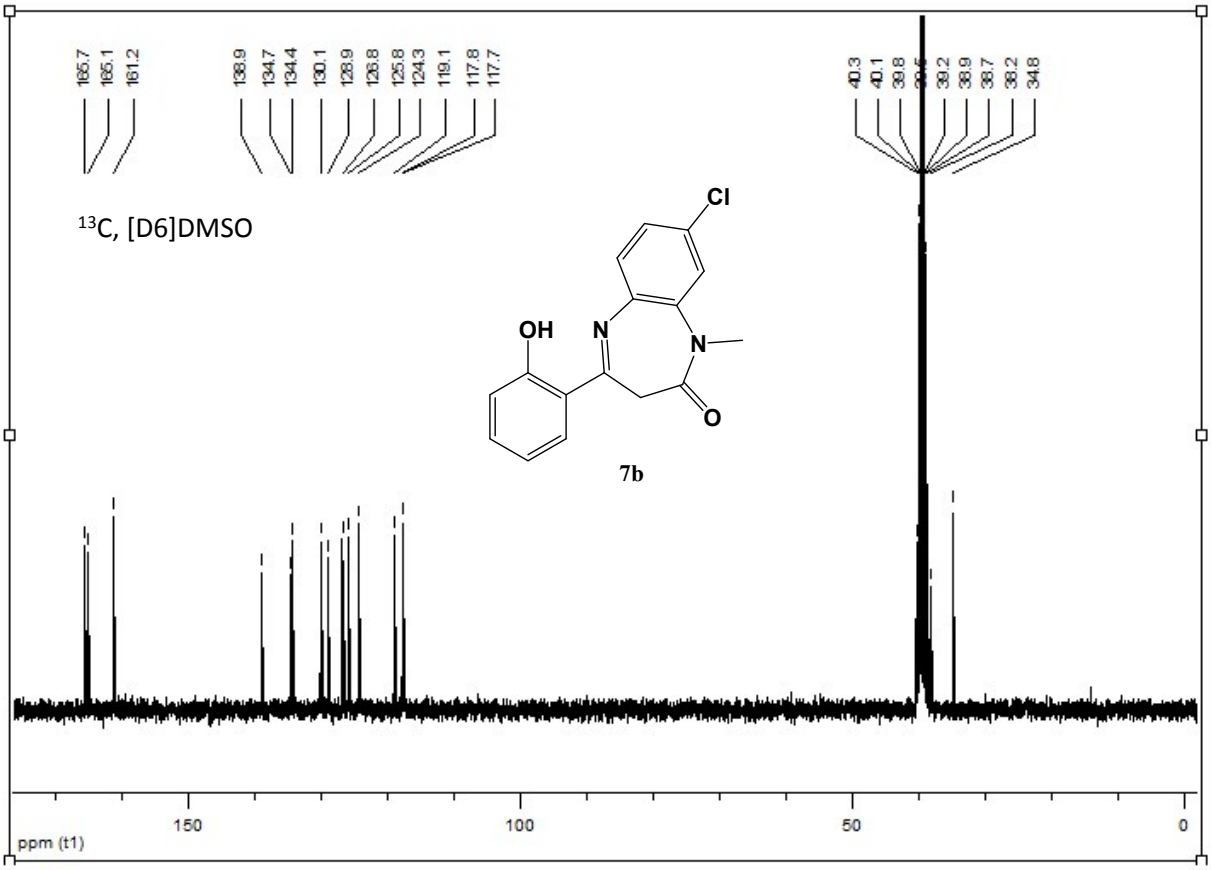
ppm (t1)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128.9
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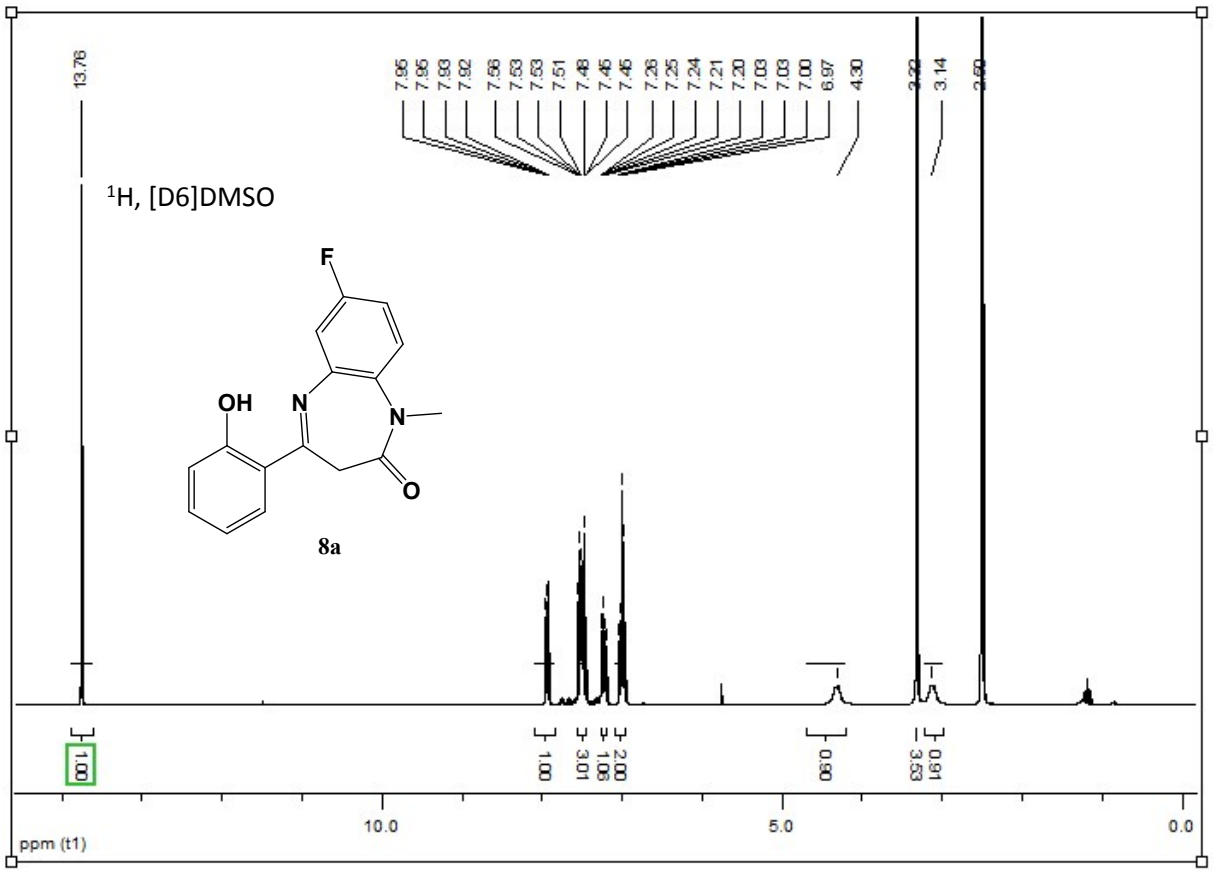
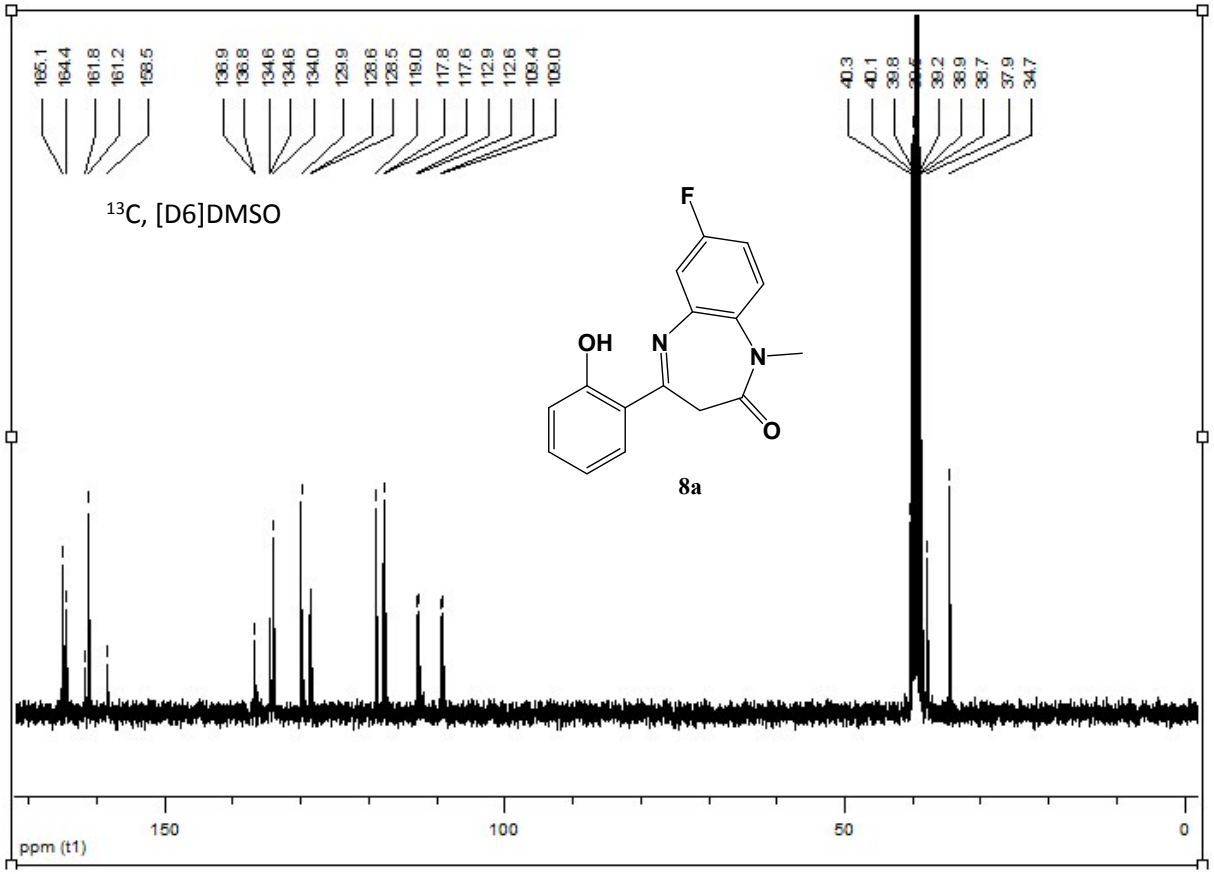
125.8
124.3
119.1
117.8
117.7
40.3
40.1
39.8
39.2
38.9
38.7
38.2
34.8
13C, [D6]DMSO
Cl
OH
N
N
O
7b
150
100
50
0
ppm (t1)



^{1}H, [D6]DMSO

8a



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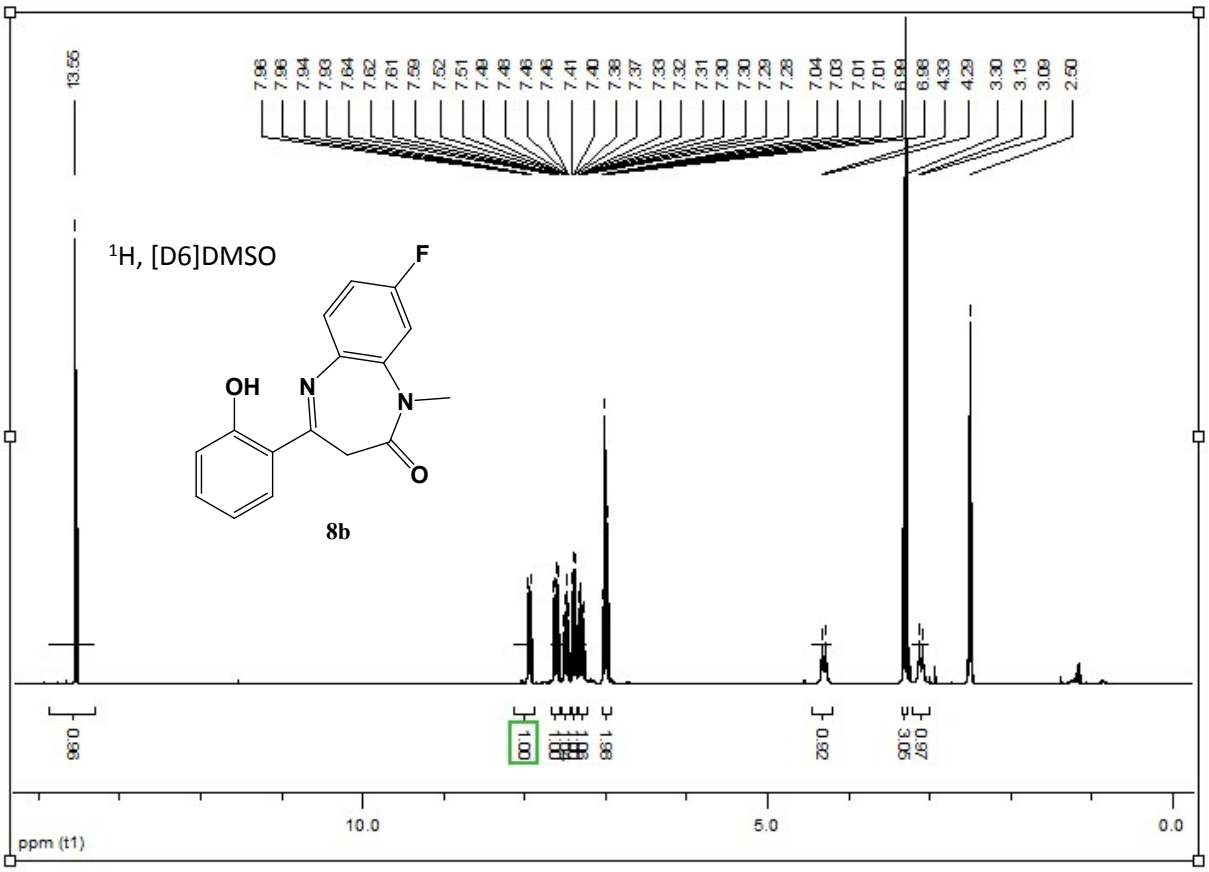
8a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7.61
7.59
7.52
7.51
7.49
7.48
7.46
7.46
7.41
7.40
7.38
7.37
7.33
7.32
7.31
7.30
7.30
7.29
7.28
7.04
7.03
7.01
7.01
6.99
6.98
4.33
4.29
3.30
3.13
3.09
2.50
1H, [D6]DMSO
F
OH
N
N
O
8b
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1.98
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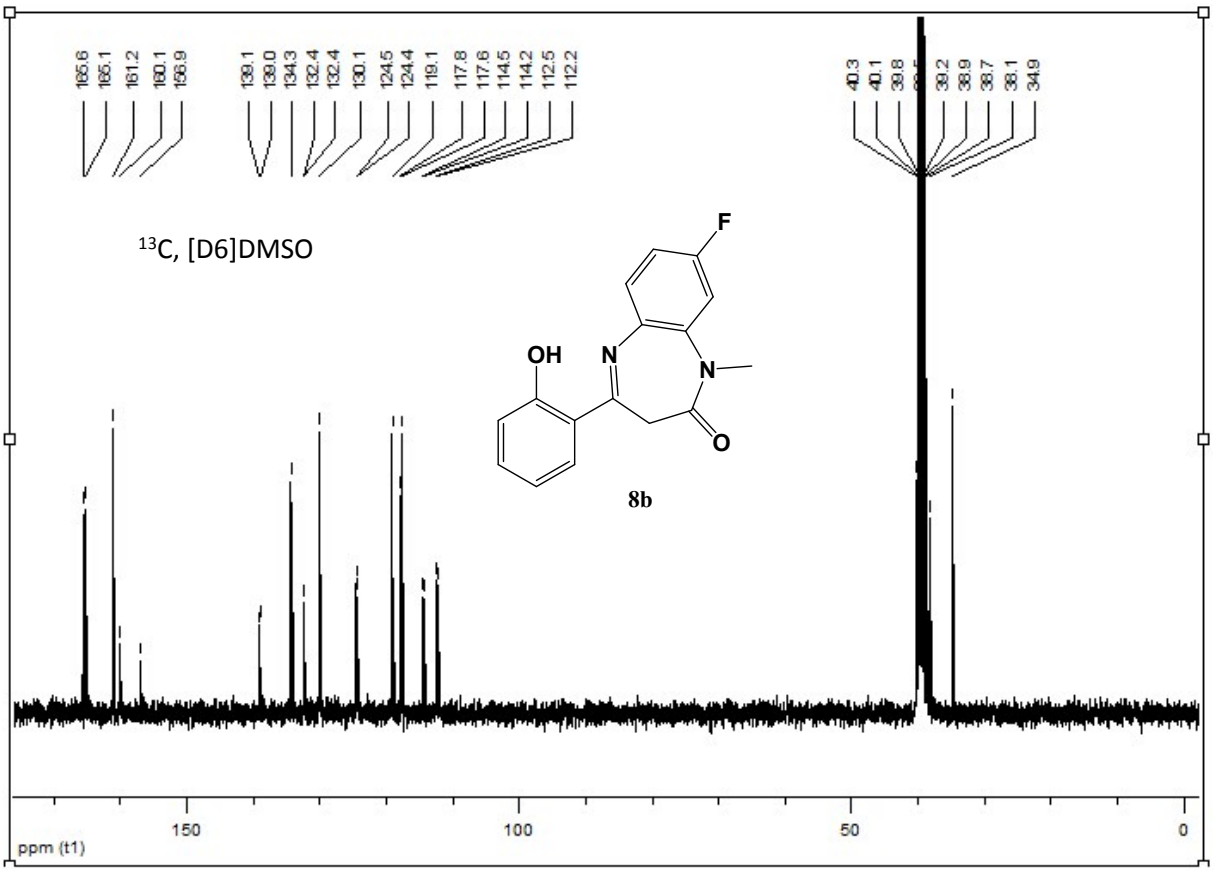
ppm (t1)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124.4
119.1
117.8
117.6
114.5
114.2
112.5
112.2
40.3
40.1
39.8
39.5
39.2
38.9
38.7
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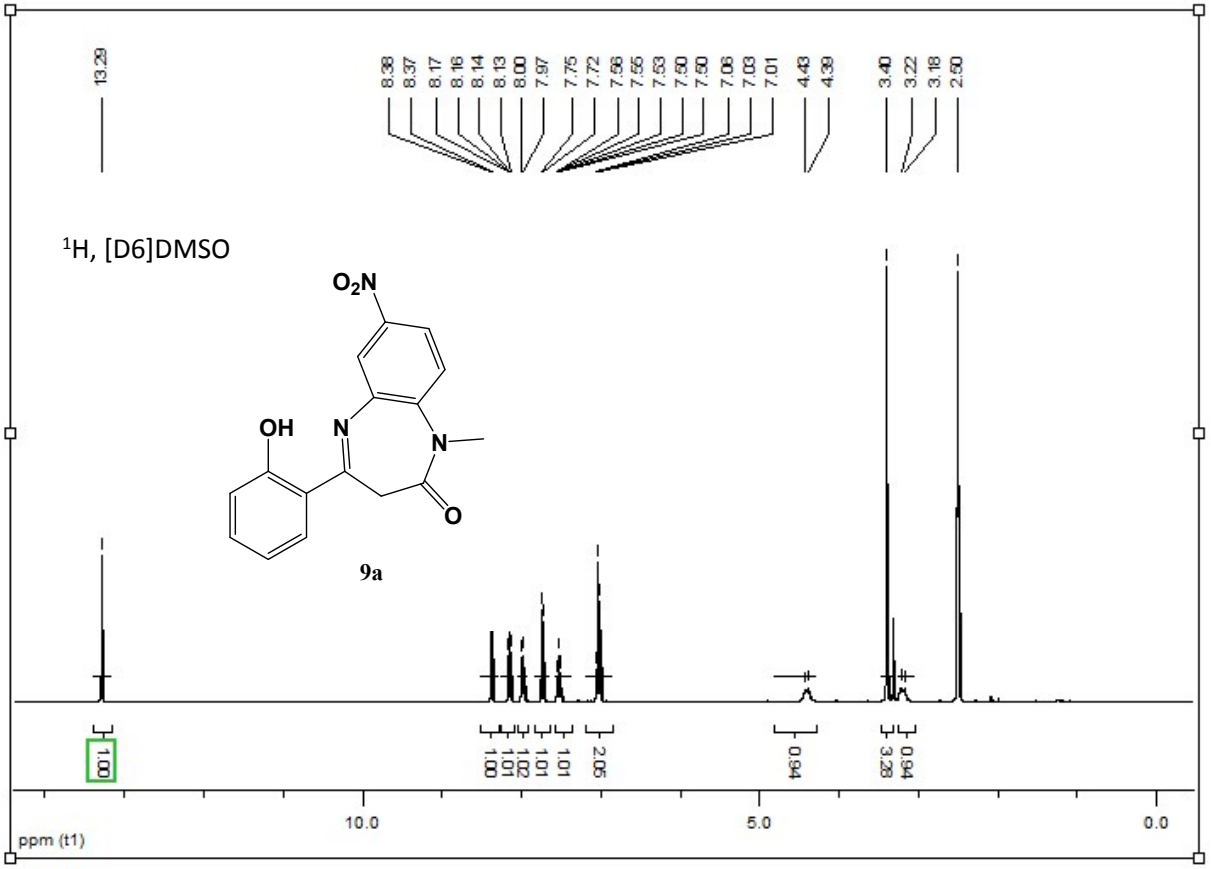
13C, [D6]DMSO
F
OH
N
N
O
8b
150
100
50
0
ppm (t1)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7.72
7.56
7.55
7.53
7.50
7.50
7.06
7.03
7.01
4.43
4.39
3.40
3.22
3.18
2.50
1H, [D6]DMSO
O2N
OH
N
N
O
9a
1.00
1.00
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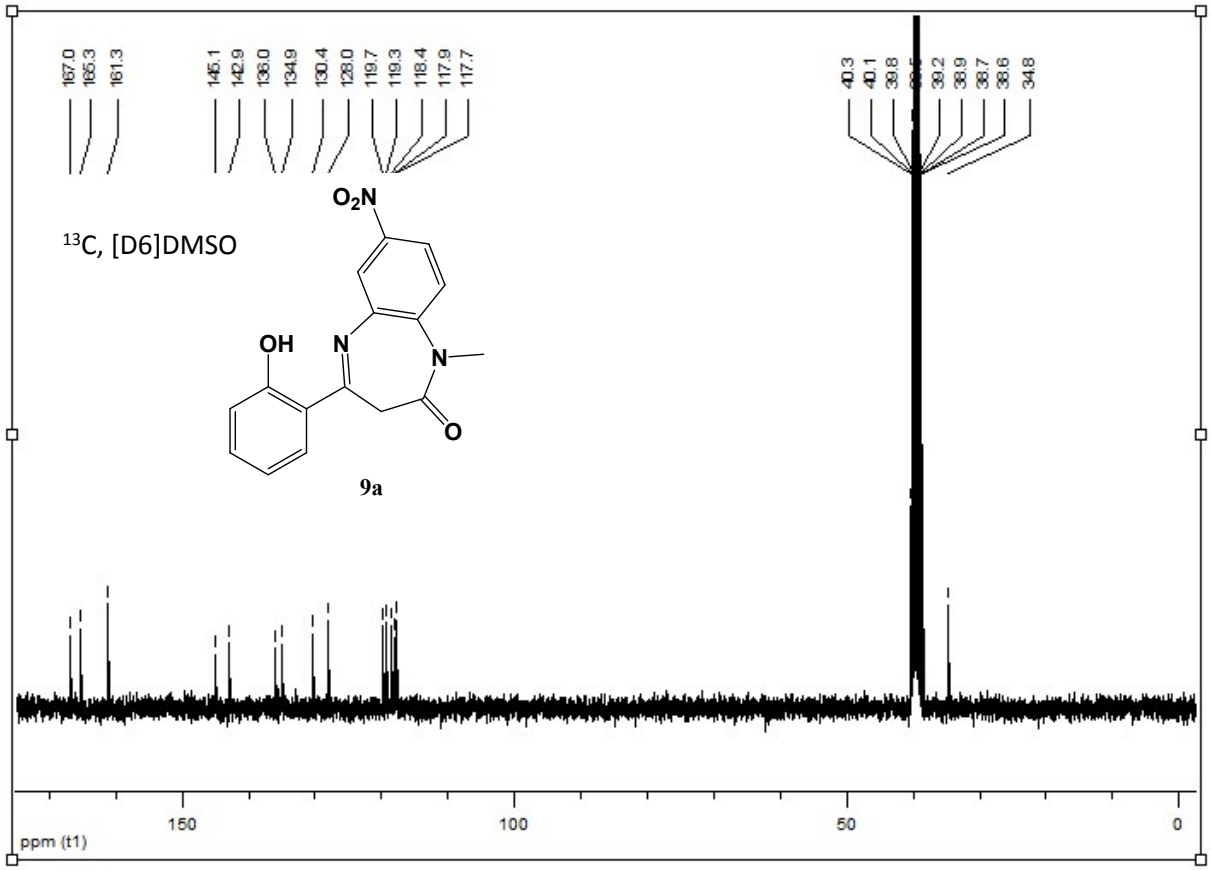
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134.9
130.4
128.0
119.7
119.3
118.4
117.9
117.7
40.3
40.1
39.8
39.5
39.2
38.9
38.7
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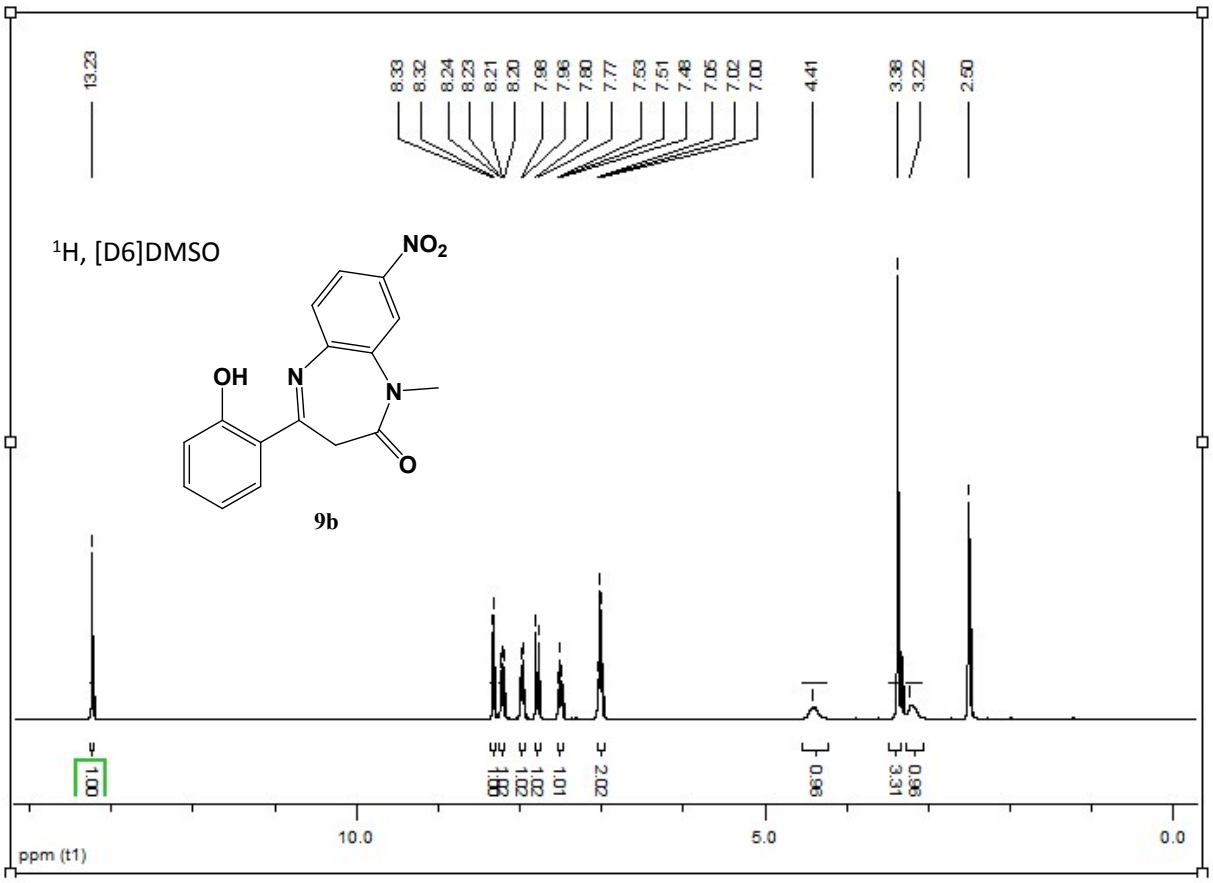
O2N
OH
N
N
O
9a
150
100
50
0
ppm (t1)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7.77
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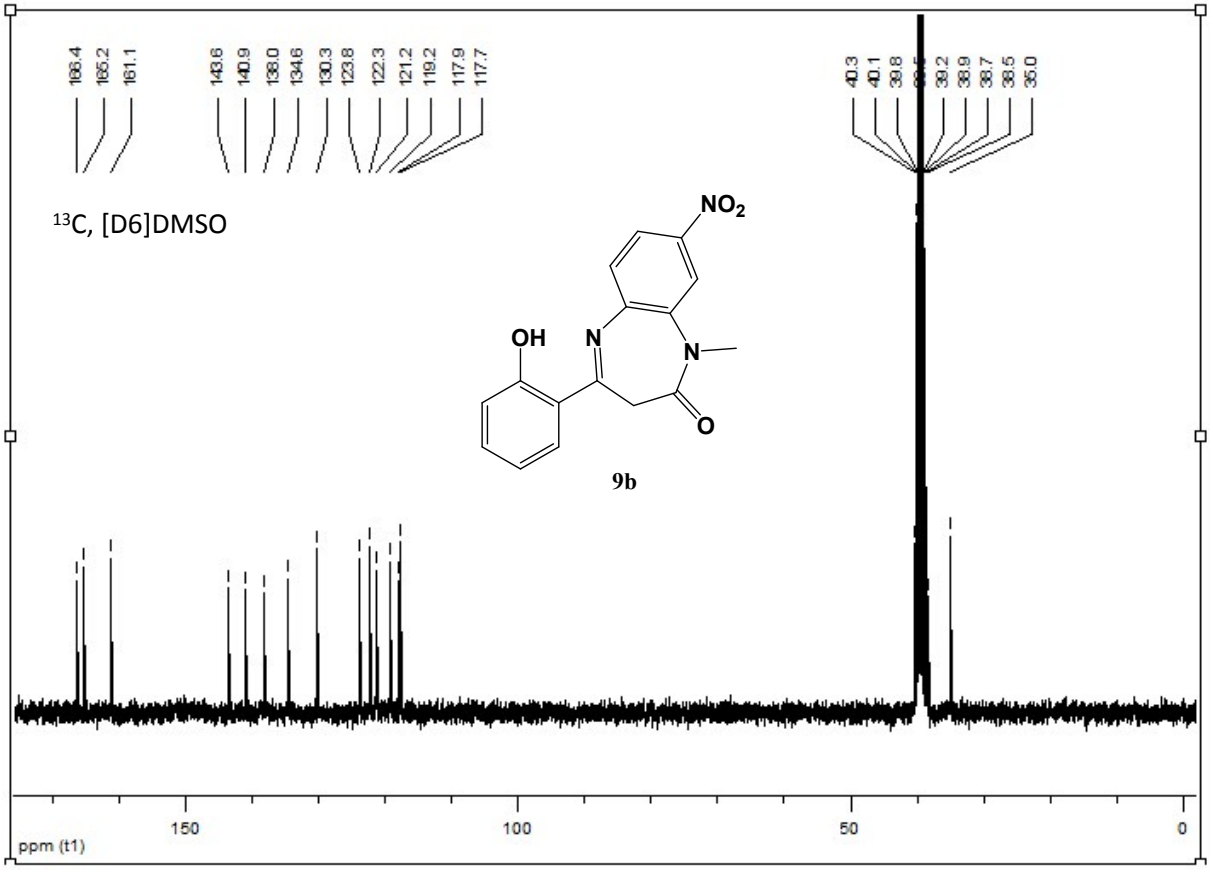
7.51
7.48
7.05
7.02
7.00
4.41
3.38
3.22
2.50
1H, [D6]DMSO
NO2
OH
N
N
O
9b
1.00
0.96
3.31
0.96
10.0
5.0
0.0
ppm (t1)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122.3
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117.9
117.7
40.3
40.1
39.8
39.5
39.2
38.9
38.7
38.5
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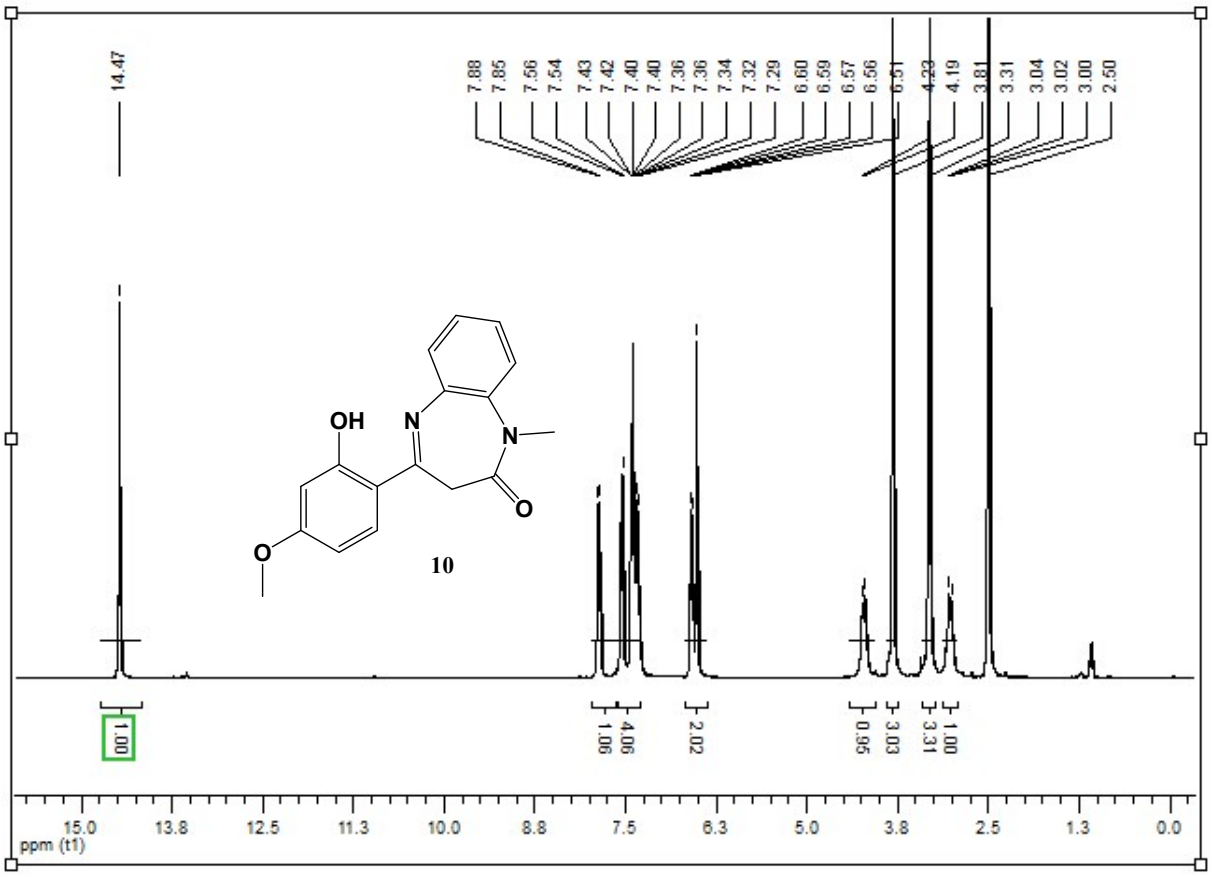
13C, [D6]DMSO
NO2
OH
N
N
O
9b
150
100
50
0
ppm (t1)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7.32
7.29
6.60
6.59
6.57
6.56
6.51
4.23
4.19
3.81
3.31
3.04
3.02
3.00
2.50
OH
N
N
O
O
10
1.00
1.06
4.06
2.02
0.96
3.03
3.31
1.00
15.0
13.8
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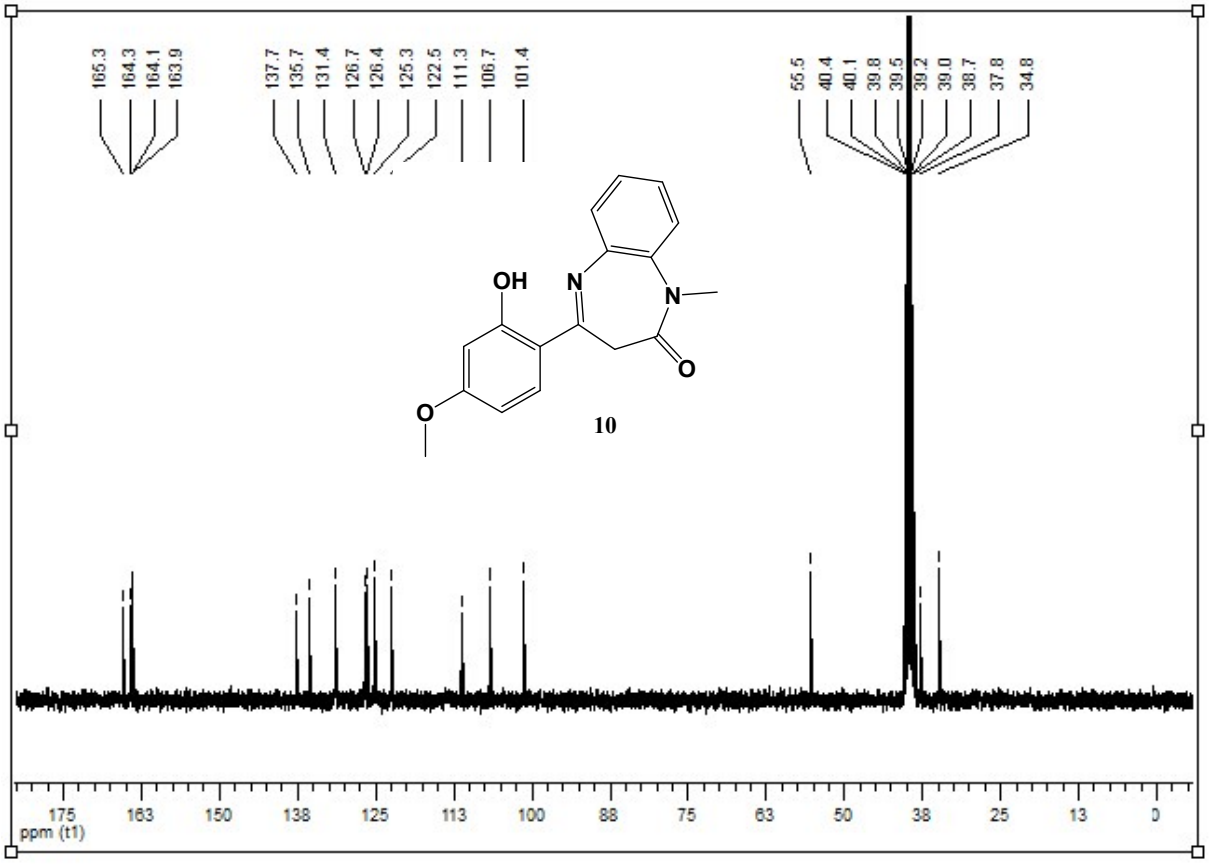
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164.1
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106.7
101.4
55.5
40.4
40.1
39.8
39.5
39.2
39.0
38.7
37.8
34.8
OH
N
N
O
O
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163
150
138
125
113
100
88
75
63
50
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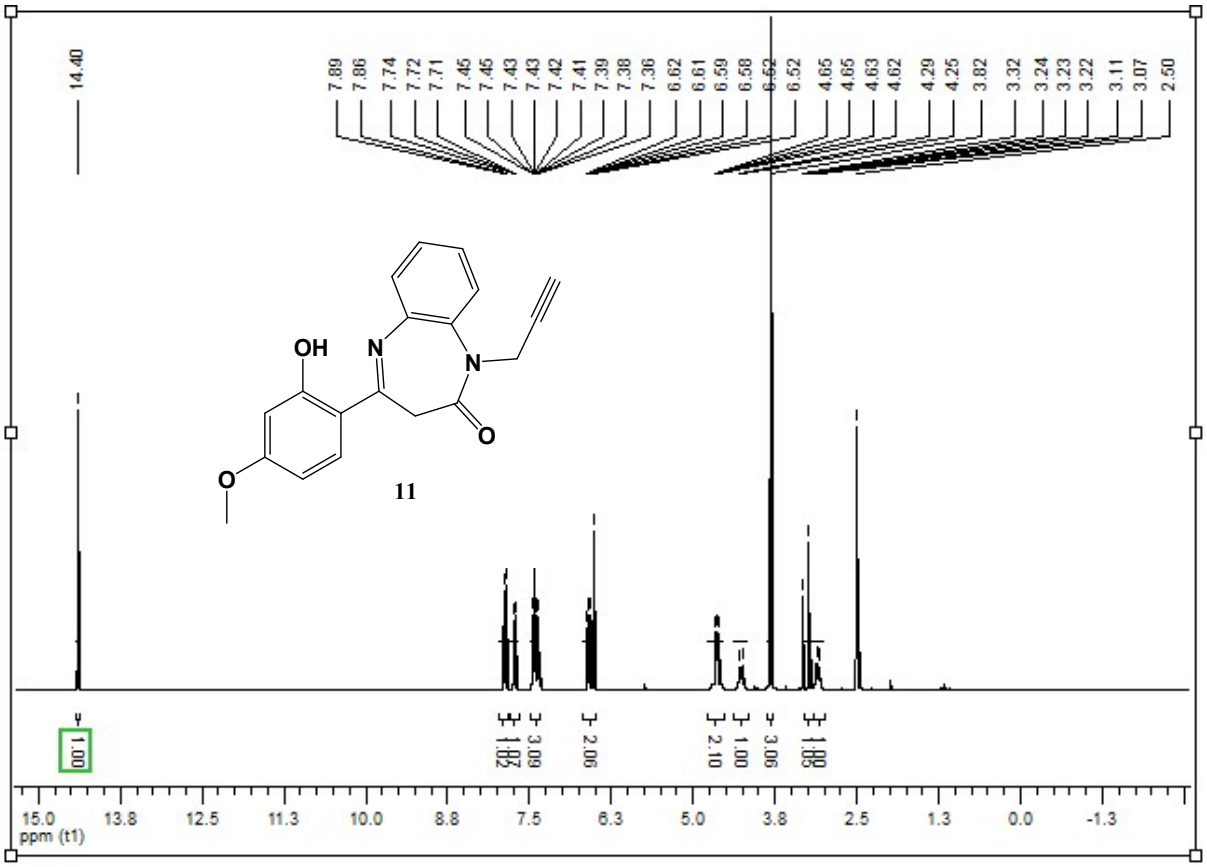
25
13
0
ppm (t1)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7.71
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7.45
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7.43
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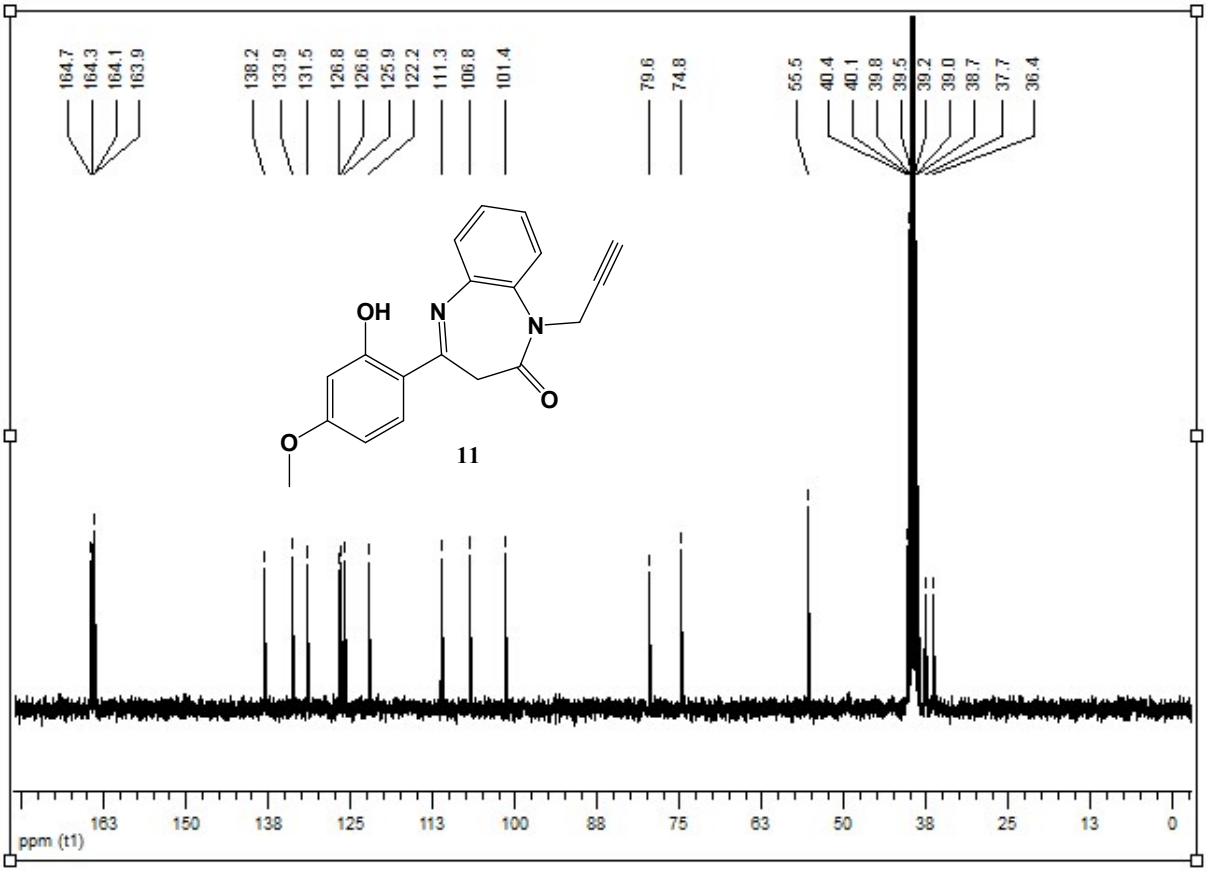
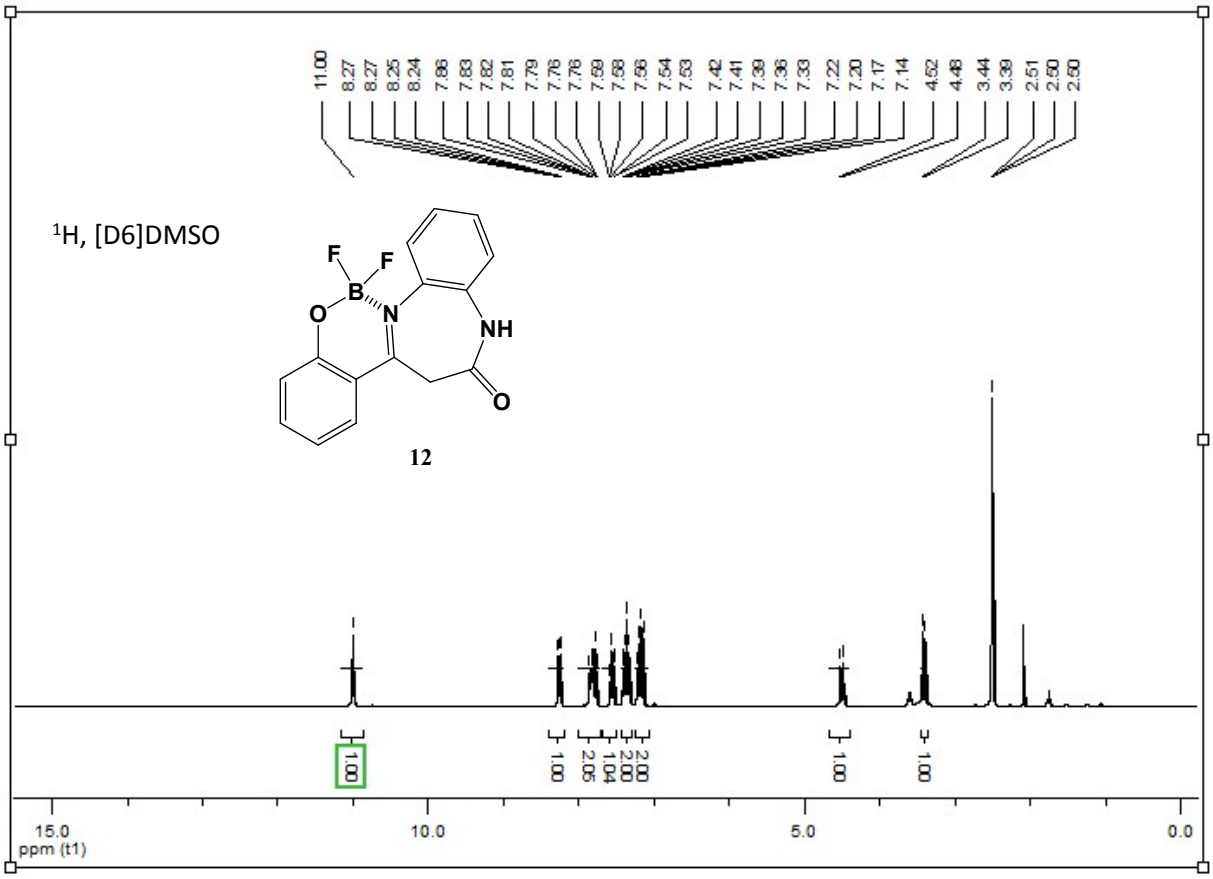
7.39
7.38
7.36
6.62
6.61
6.59
6.58
6.52
4.65
4.65
4.63
4.62
4.29
4.25
3.82
3.32
3.24
3.23
3.22
3.11
3.07
2.50
OH
N
N
O
O
11
1.00
1.02
3.09
2.06
2.10
1.00
3.06
1.00
15.0
13.8
12.5
11.3
10.0
8.8
7.5
6.3
5.0
3.8
2.5
1.3
0.0
-1.3
ppm (t1)


164.7
164.3
164.1
163.9
138.2
133.9
131.5
126.8
126.6
125.9
122.2
111.3
106.8
101.4
79.6
74.8
55.5
40.4
40.1
39.8
39.5
39.2
39.0
38.7
37.7
36.4
OH
N
N
O
O
11
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)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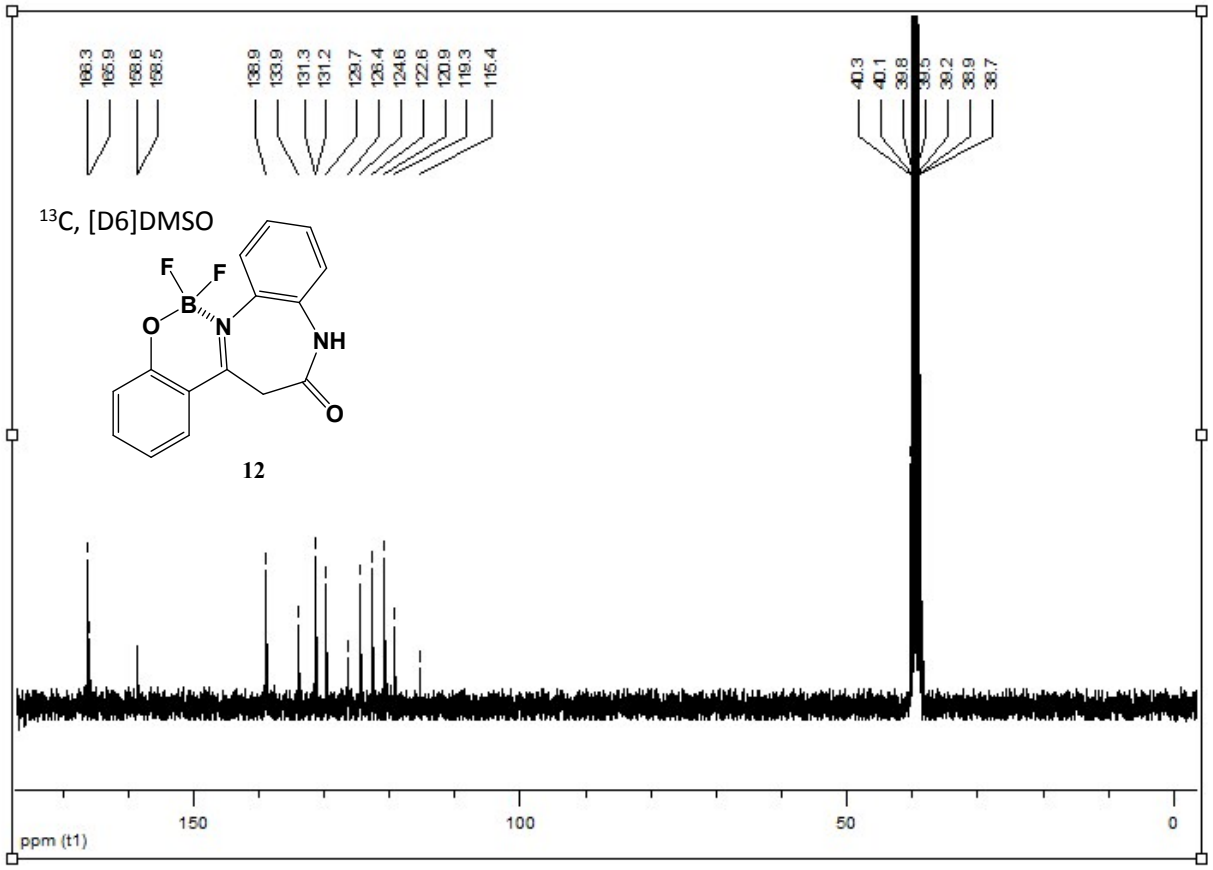
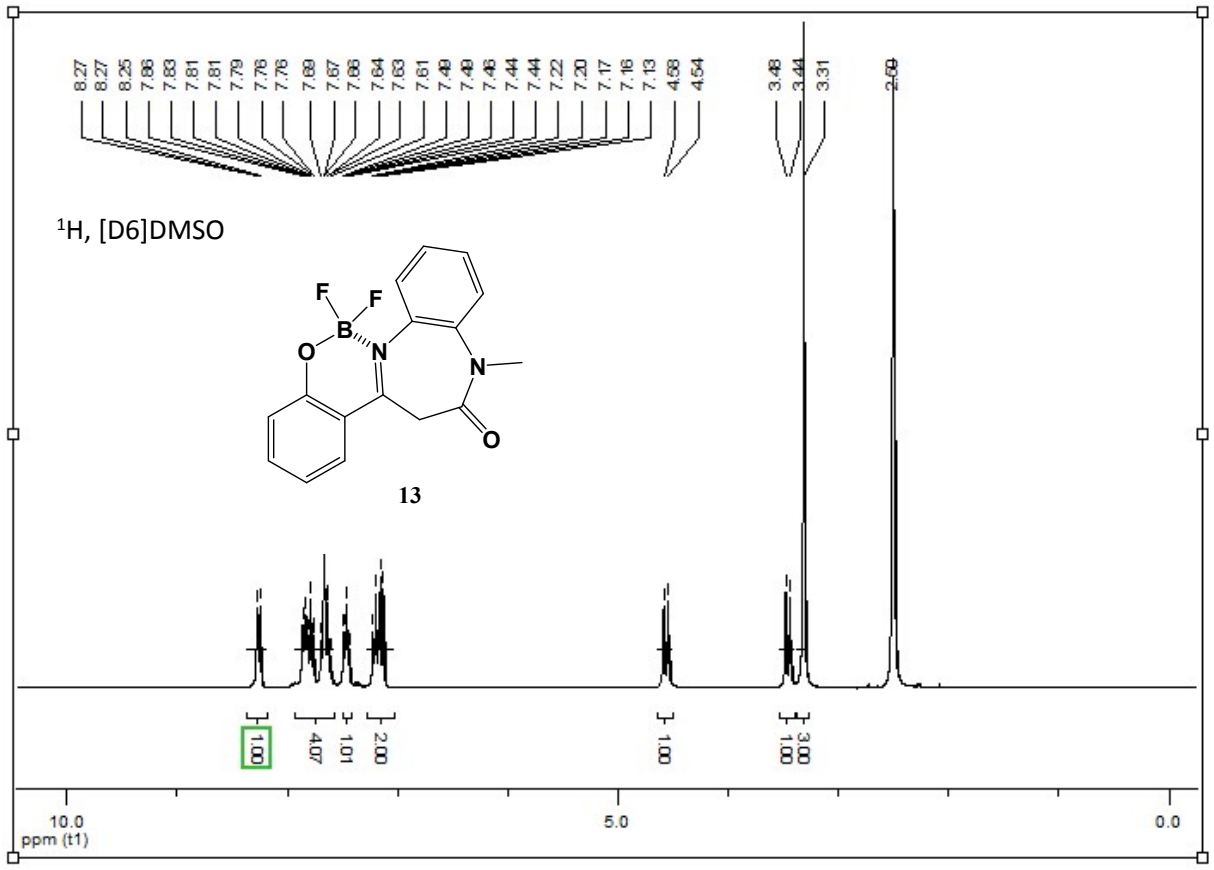
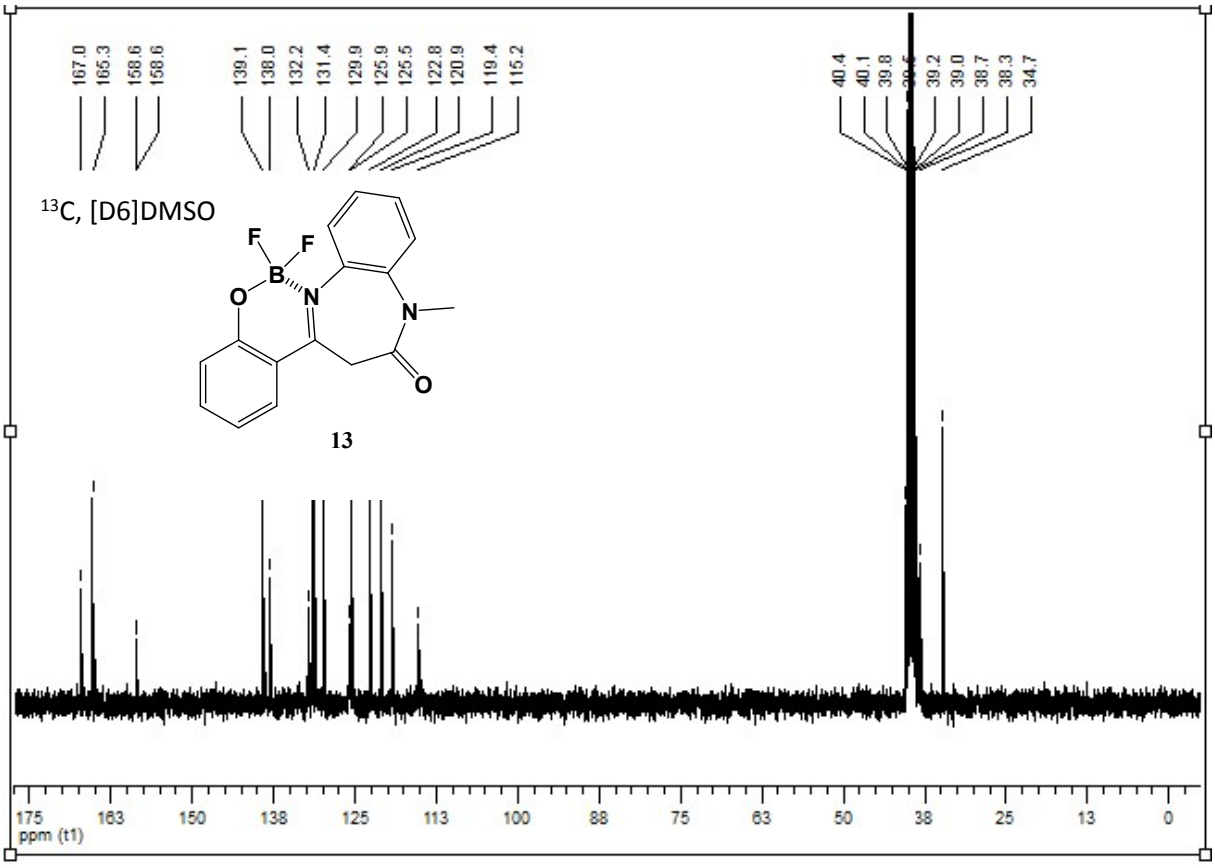
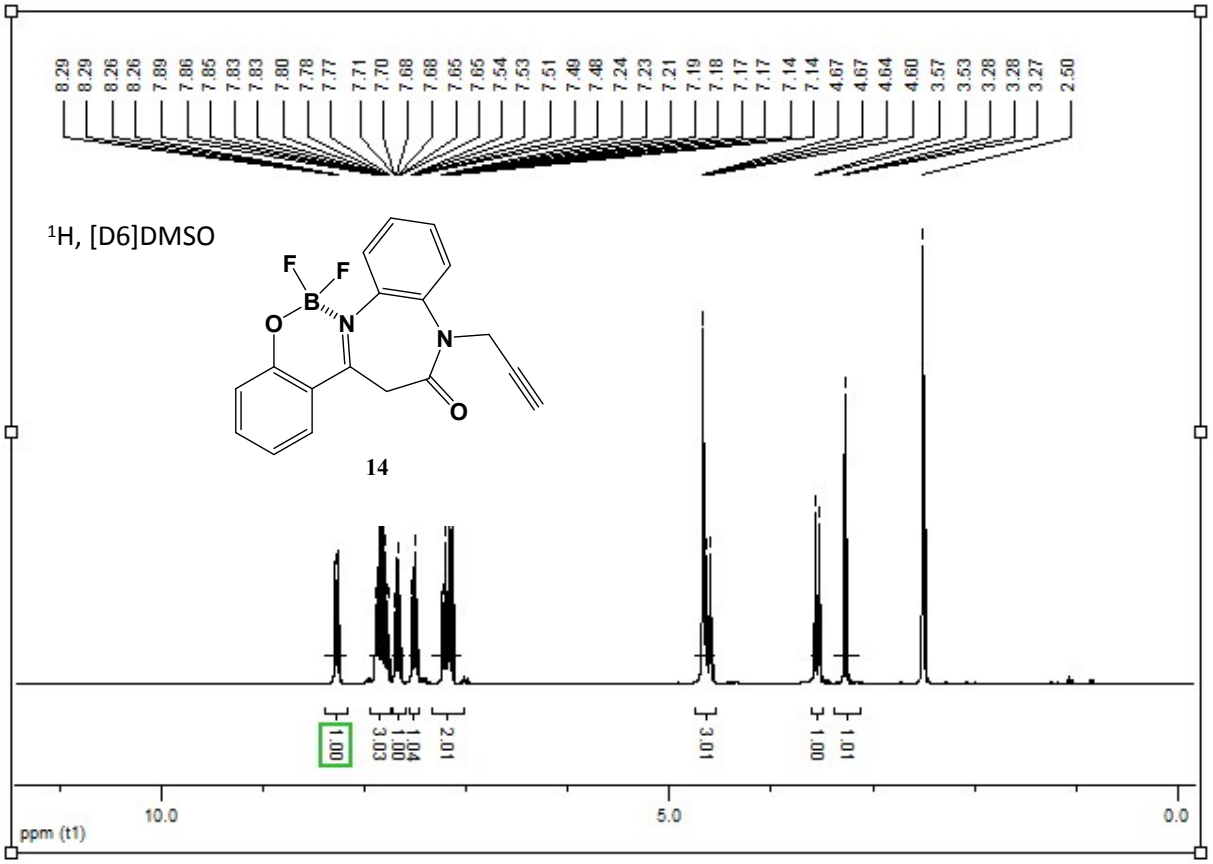
8.27
8.27
8.25
8.24
7.86
7.83
7.82
7.81
7.79
7.76
7.76
7.69
7.68
7.66
7.54
7.53
7.42
7.41
7.39
7.36
7.33
7.22
7.20
7.17
7.14
4.62
4.48
3.44
3.39
2.51
2.50
2.50
1H, [D6]DMSO
F
F
B
O
N
NH
O
12
1.00
1.00
2.05
1.04
2.00
2.22
1.00
3.00
15.0
10.0
5.0
0.0
ppm (t1)

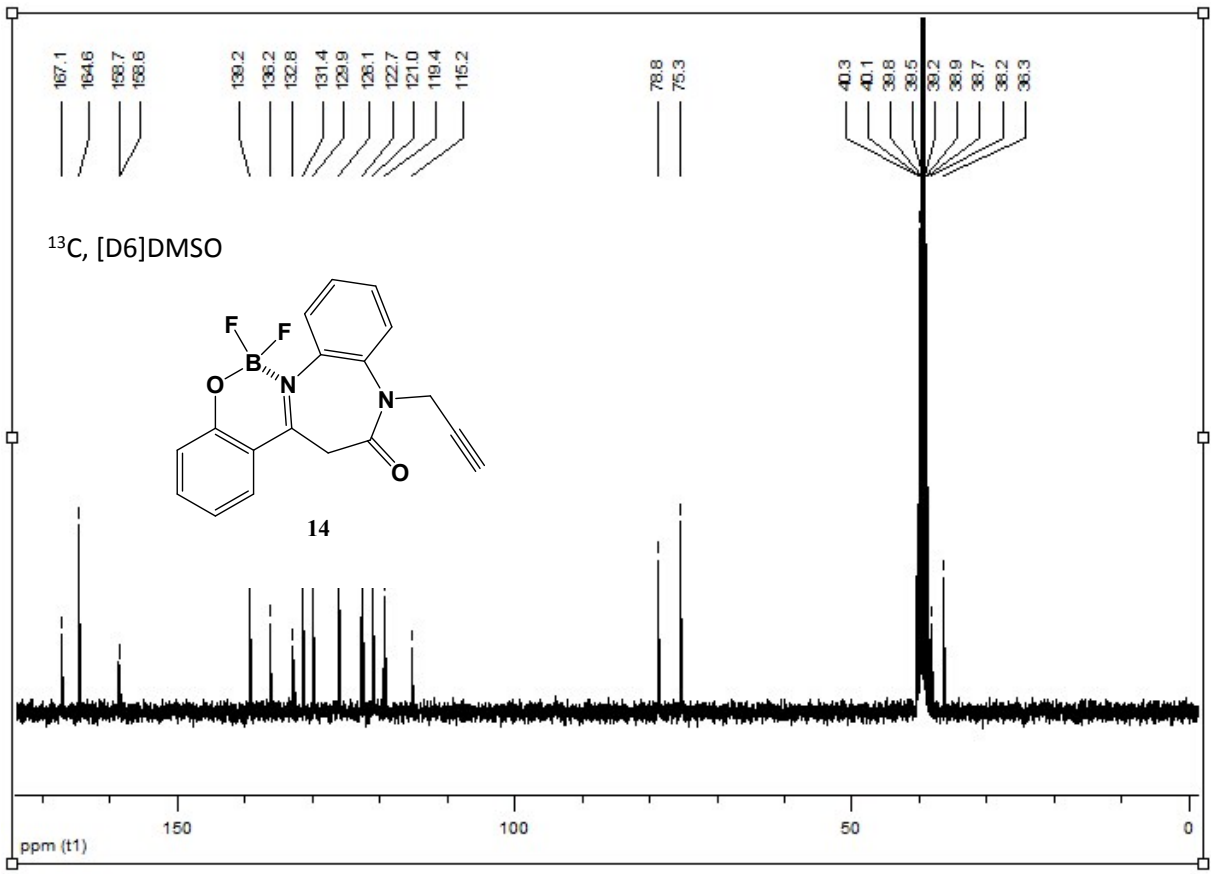

166.3
165.9
158.6
158.5
136.9
133.9
131.3
131.2
129.7
126.4
124.6
122.6
120.9
119.3
115.4
40.3
40.1
39.8
39.5
39.2
38.9
38.7
13C, [D6]DMSO
F
F
B
O
N
NH
O
12
150
100
50
0
ppm (t1)


8.27
8.27
8.25
7.86
7.83
7.81
7.81
7.79
7.76
7.76
7.69
7.67
7.66
7.64
7.63
7.61
7.49
7.49
7.46
7.44
7.44
7.22
7.20
7.17
7.16
7.13
4.58
4.54
3.48
3.44
3.31
2.69
1H, [D6]DMSO
F
F
B
O
N
N
O
13
1.00
4.07
1.01
2.00
1.00
3.00
10.0
ppm (t1)
5.0
0.0

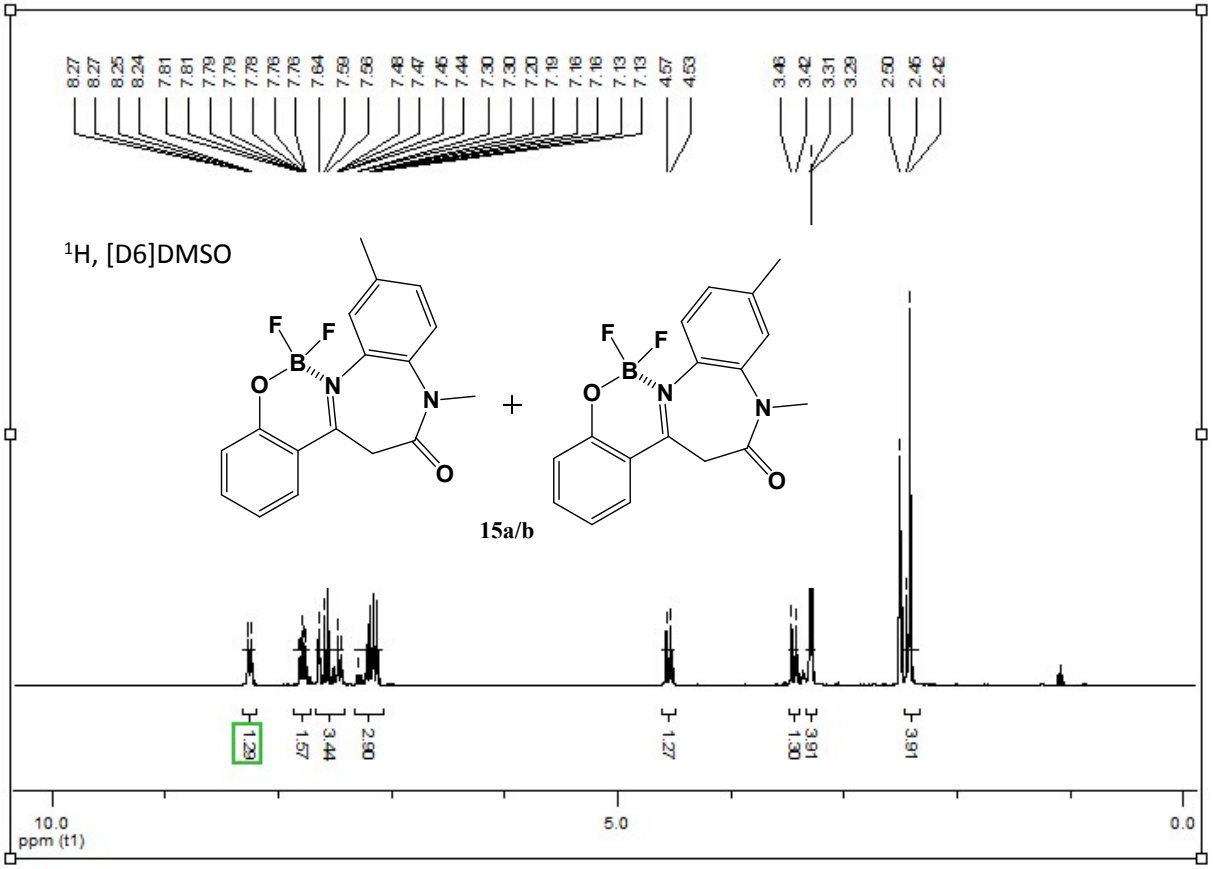

167.0
165.3
158.6
158.6
139.1
138.0
132.2
131.4
129.9
125.9
125.5
122.8
120.9
119.4
115.2
40.4
40.1
39.8
39.5
39.2
39.0
38.7
38.3
34.7
13C, [D6]DMSO
F
F
B
O
N
N
O
13
175
ppm (t1)
163
150
138
125
113
100
88
75
63
50
38
25
13
0

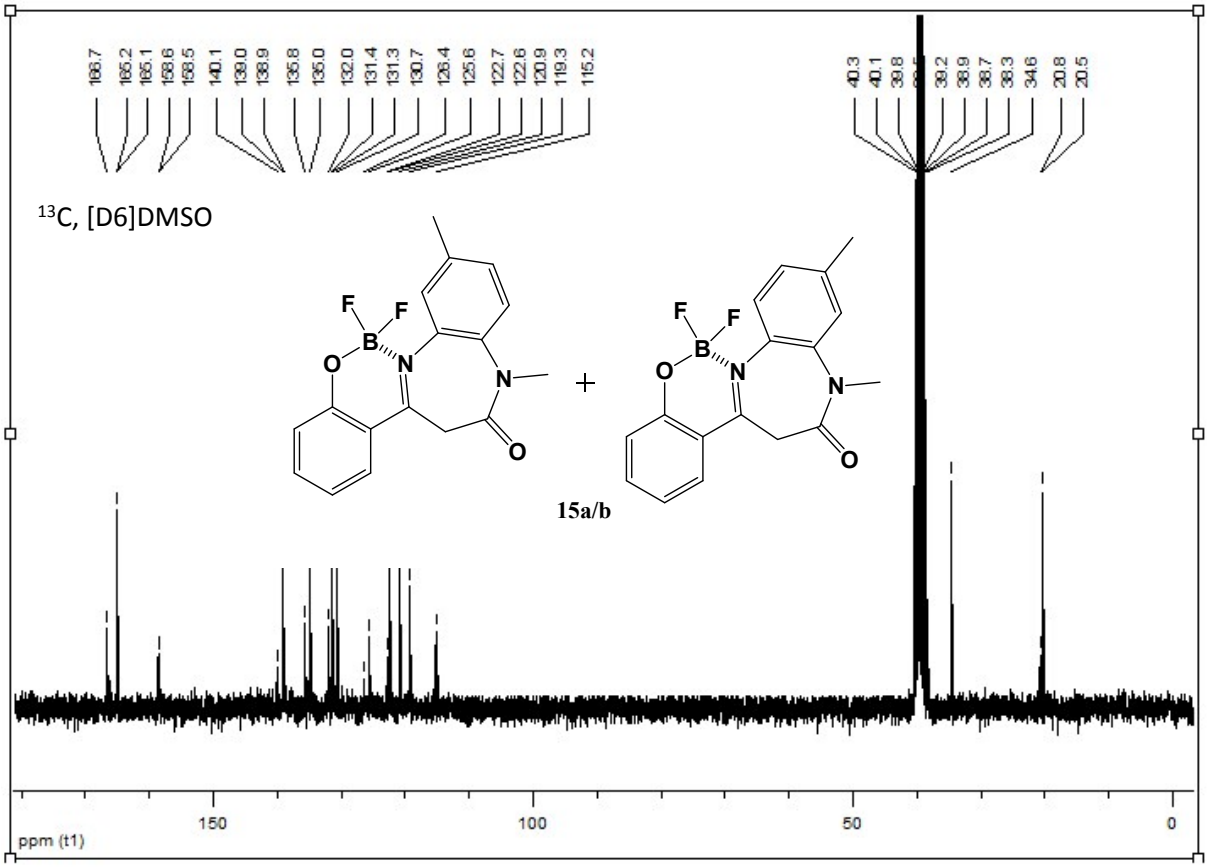

8.29
8.29
8.26
8.26
7.89
7.86
7.85
7.83
7.83
7.80
7.78
7.77
7.71
7.70
7.68
7.68
7.65
7.65
7.54
7.53
7.51
7.49
7.48
7.24
7.23
7.21
7.19
7.18
7.17
7.17
7.14
7.14
4.67
4.67
4.64
4.60
3.57
3.53
3.28
3.28
3.27
2.50
1H, [D6]DMSO
F
F
B
O
N
N
O
1.00
3.03
1.00
1.04
2.01
3.01
3.00
1.01
10.0
5.0
0.0
ppm (t1)

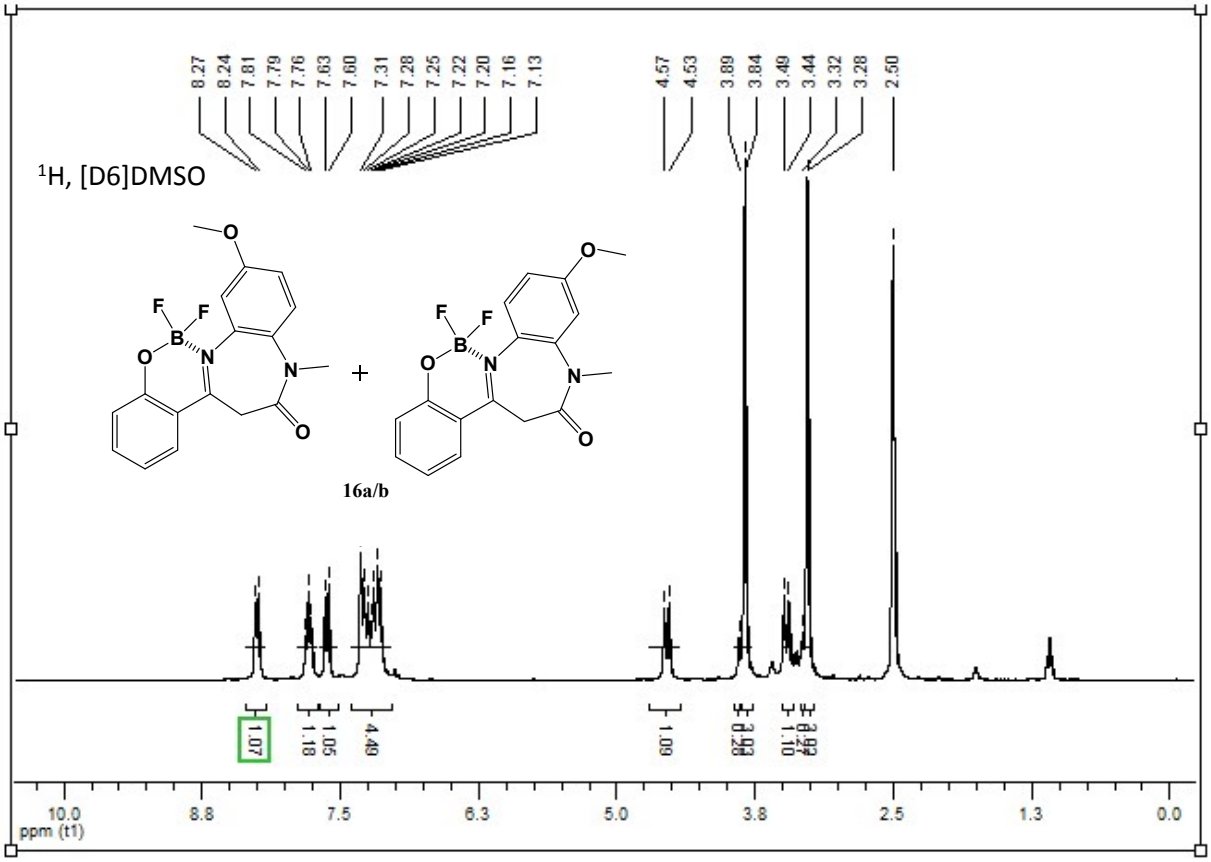
14

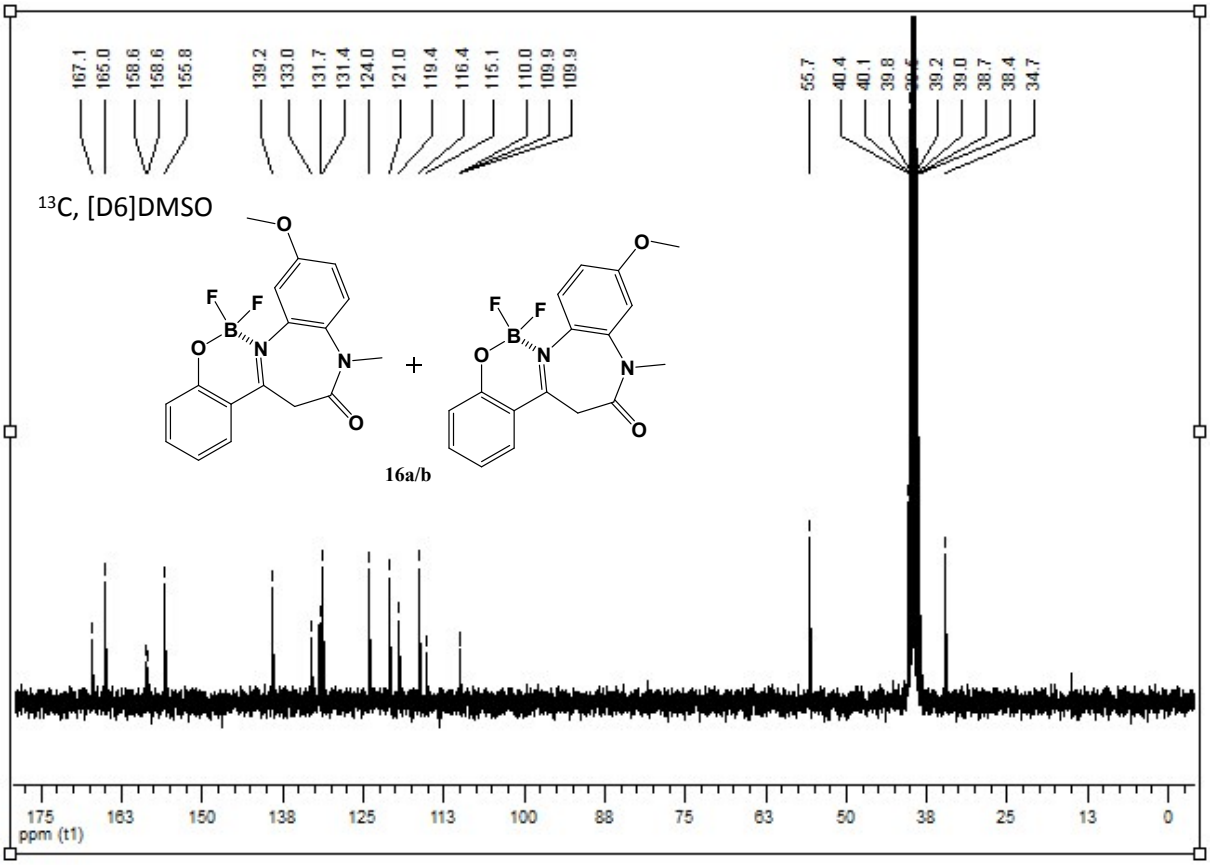

167.1
164.6
158.7
158.6
139.2
136.2
132.8
131.4
129.9
126.1
122.7
121.0
119.4
115.2
78.8
75.3
40.3
40.1
39.8
39.5
39.2
38.9
38.7
38.2
36.3
13C, [D6]DMSO
F
F
B
O
N
N
O
150
100
50
0
ppm (t1)

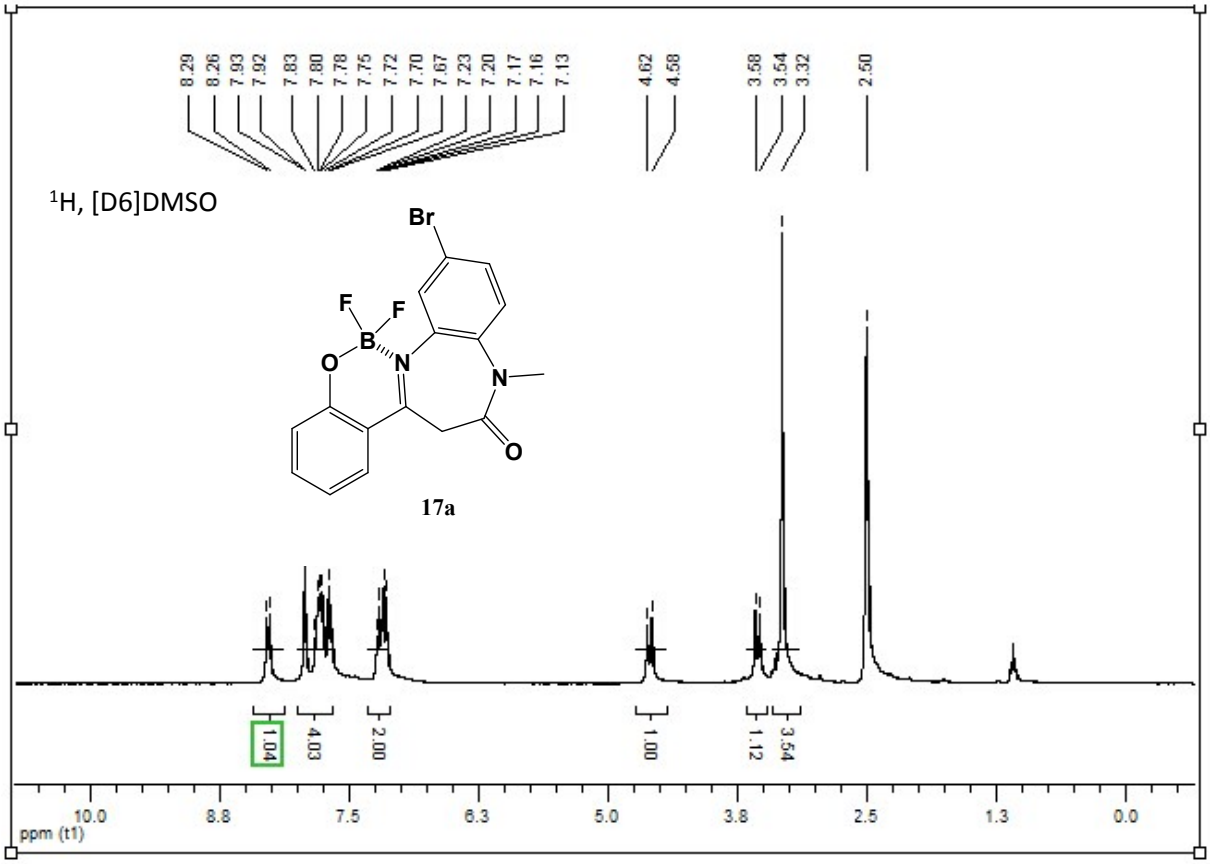
14


8.27
8.27
8.25
8.24
7.81
7.81
7.79
7.79
7.78
7.76
7.76
7.64
7.59
7.56
7.48
7.47
7.45
7.44
7.30
7.30
7.20
7.19
7.16
7.16
7.13
7.13
4.57
4.53
3.46
3.42
3.31
3.29
2.50
2.45
2.42
1H, [D6]DMSO
15a/b
1.28
1.57
3.44
2.90
1.27
1.30
3.91
3.91
10.0
ppm (t1)
5.0
0.0


166.7
165.2
165.1
158.6
158.5
140.1
139.0
138.9
135.8
135.0
132.0
131.4
131.3
130.7
126.4
125.6
122.7
122.6
120.9
119.3
115.2
40.3
40.1
39.8
39.2
38.9
38.7
38.3
34.6
20.8
20.5
13C, [D6]DMSO
15a/b
150
100
50
0
ppm (t1)

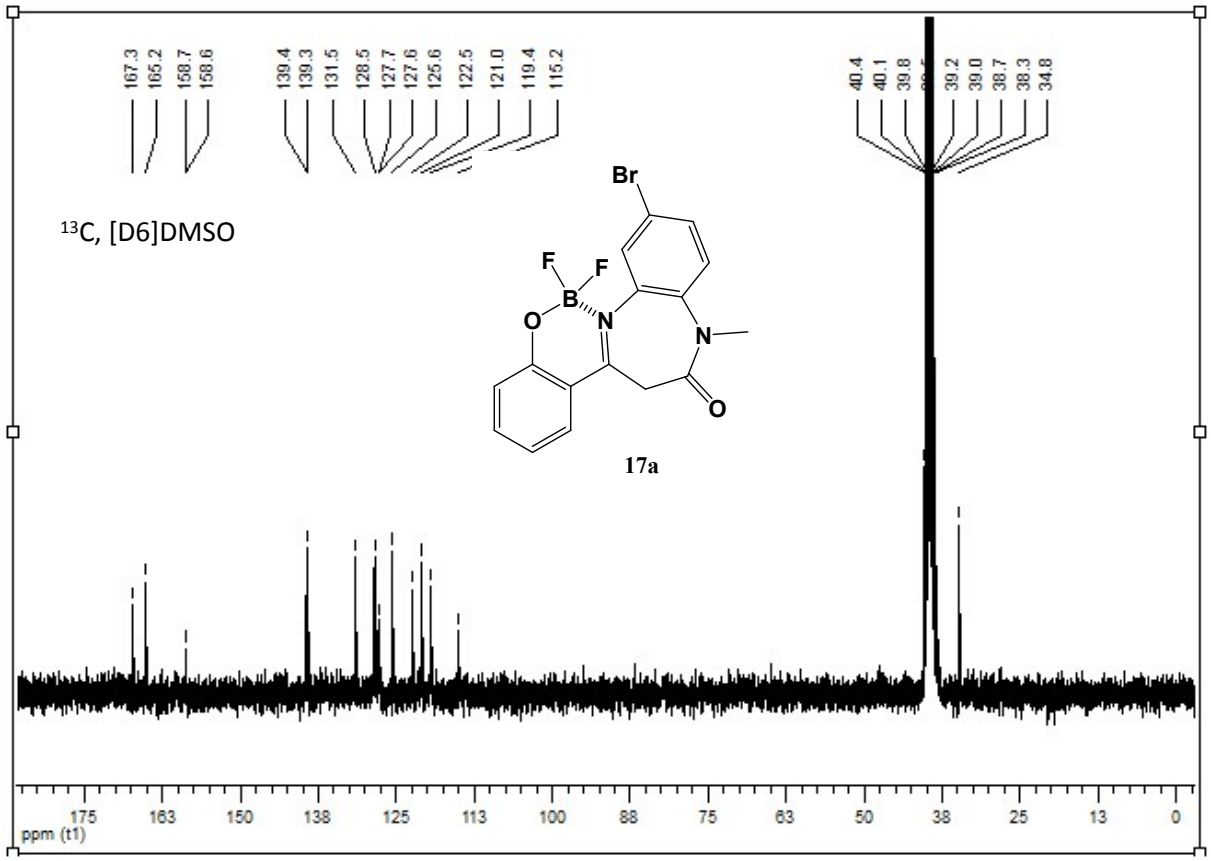

8.27
8.24
7.81
7.79
7.76
7.63
7.60
7.31
7.28
7.25
7.22
7.20
7.16
7.13
4.57
4.53
3.89
3.84
3.49
3.44
3.32
3.28
2.50
1H, [D6]DMSO
16a/b
1.07
1.18
1.05
4.49
1.09
3.00
3.00
1.10
3.02
10.0
8.8
7.5
6.3
5.0
3.8
2.5
1.3
0.0
ppm (t1)


167.1
165.0
158.6
158.6
155.8
139.2
133.0
131.7
131.4
124.0
121.0
119.4
116.4
115.1
110.0
109.9
109.9
55.7
40.4
40.1
39.8
39.2
39.0
38.7
38.4
34.7
13C, [D6]DMSO
16a/b
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)



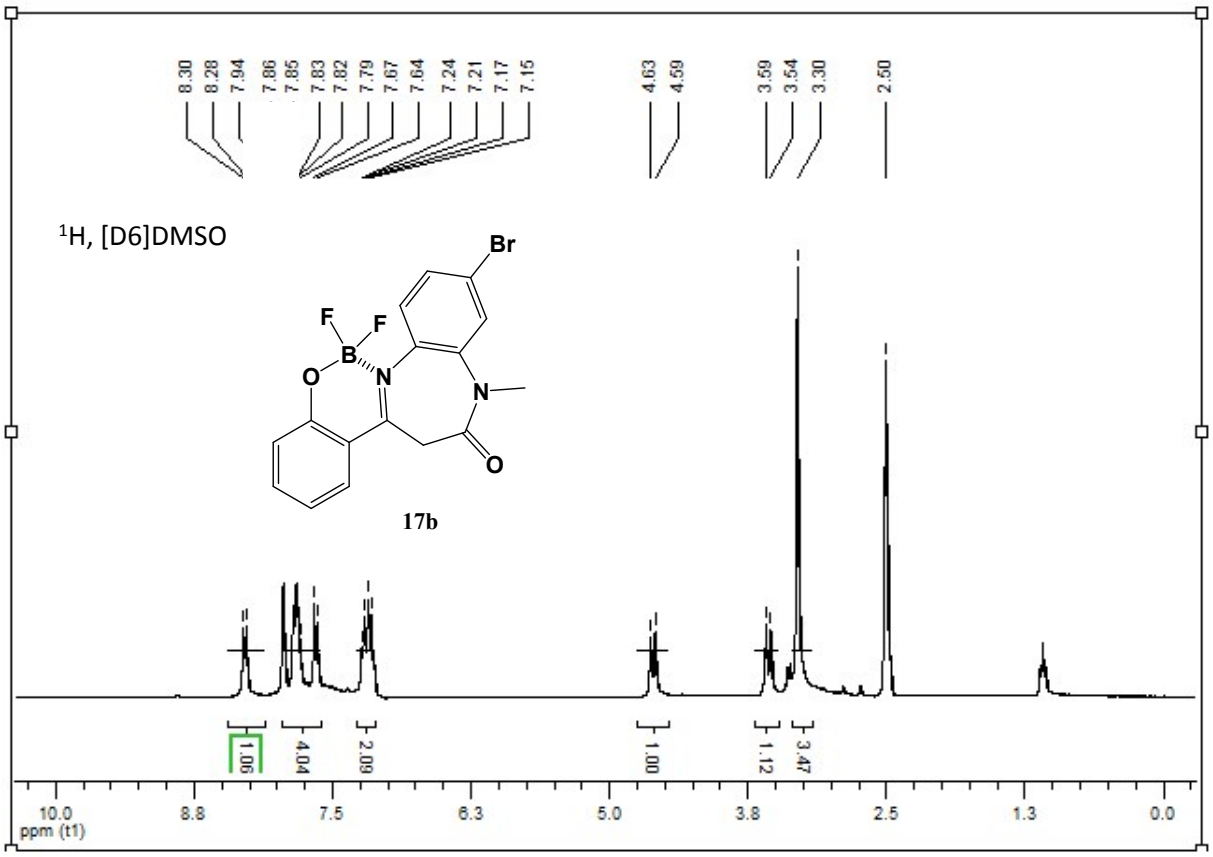
^{1}H, [D6]DMSO

17a

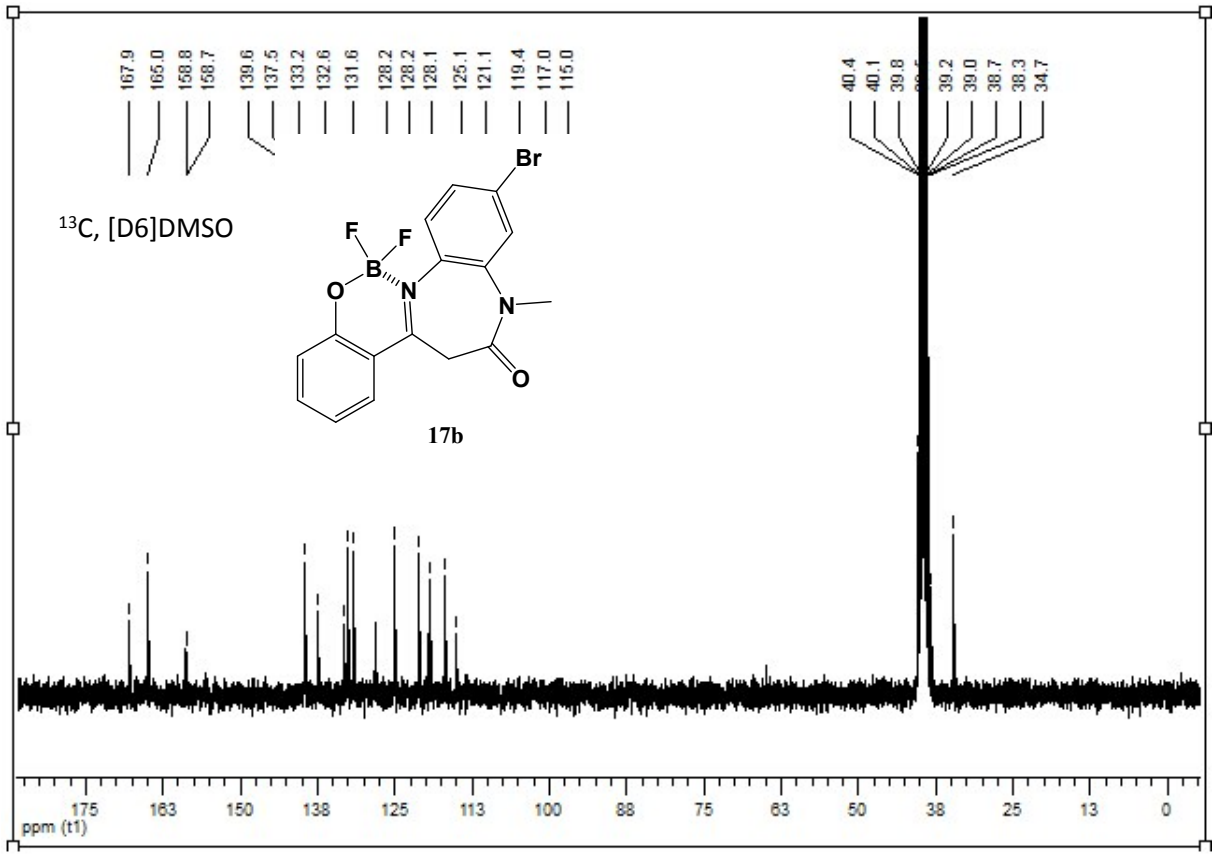


^{13}C, [D6]DMSO

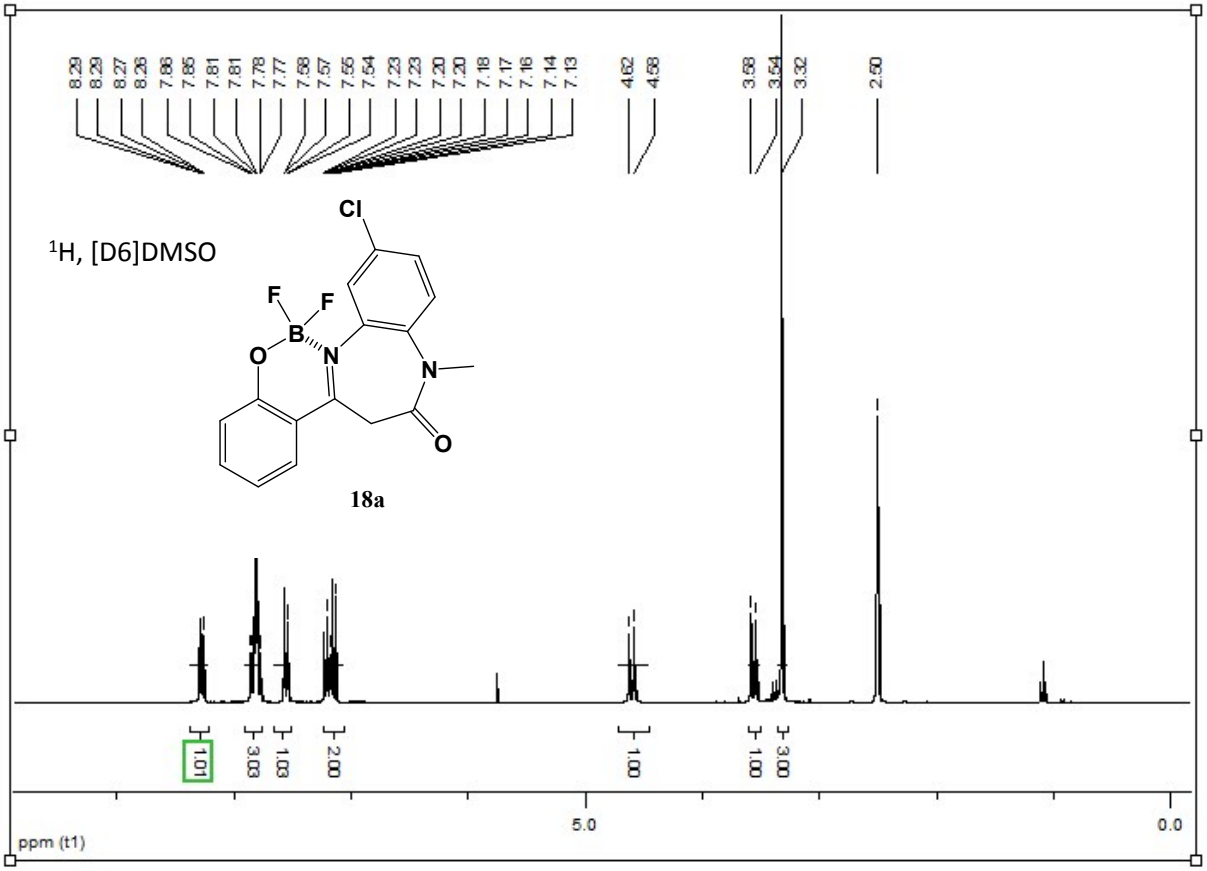
17a


8.30
8.28
7.94
7.86
7.85
7.83
7.82
7.79
7.67
7.64
7.24
7.21
7.17
7.15
4.63
4.59
3.59
3.54
3.30
2.50
1H, [D6]DMSO
Br
F
F
B
O
N
N
O
1.06
4.04
2.09
1.00
1.12
3.47
10.0
8.8
7.5
6.3
5.0
3.8
2.5
1.3
0.0
ppm (t1)

17b

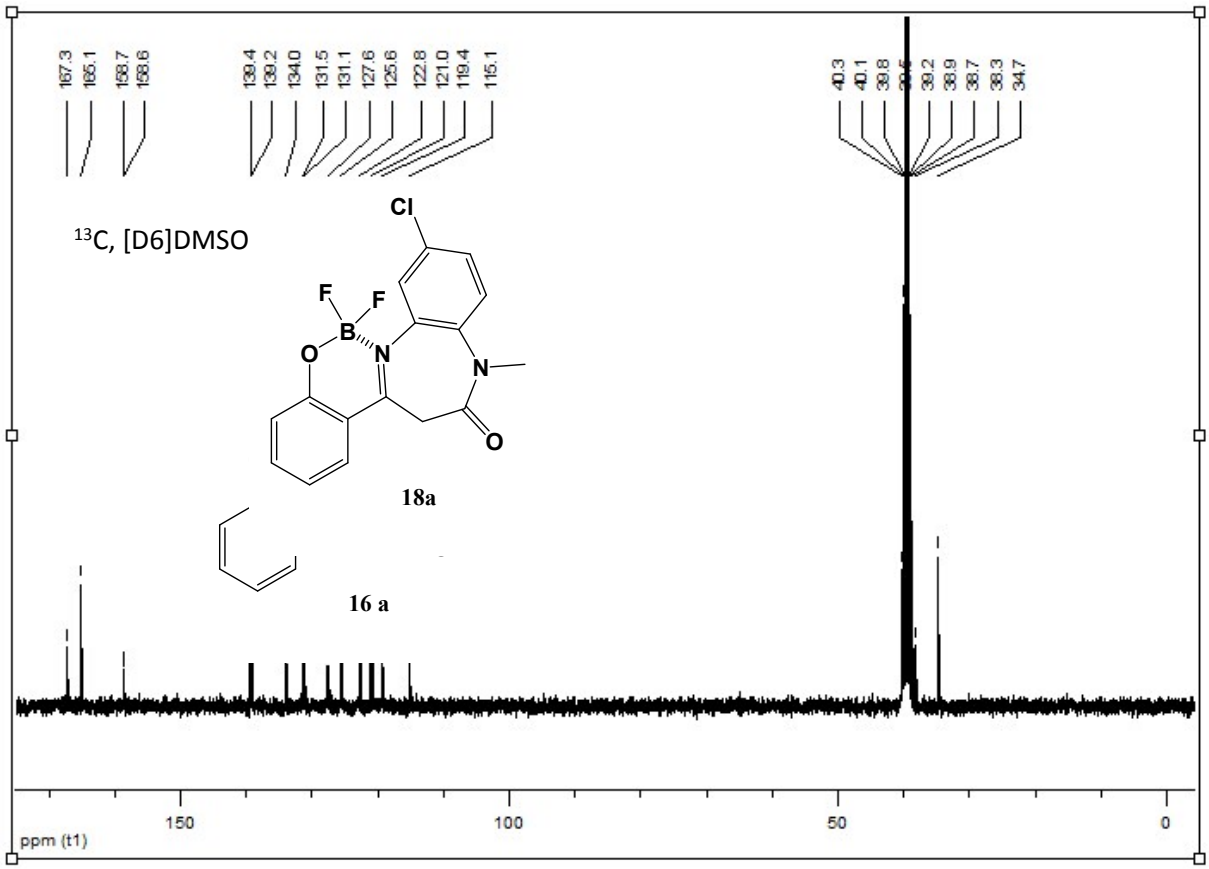

167.9
165.0
158.8
158.7
139.6
137.5
133.2
132.6
131.6
128.2
128.2
128.1
125.1
121.1
119.4
117.0
115.0
40.4
40.1
39.8
39.2
39.0
38.7
38.3
34.7
13C, [D6]DMSO
Br
F
F
B
O
N
N
O
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)

17b



^{1}H, [D6]DMSO

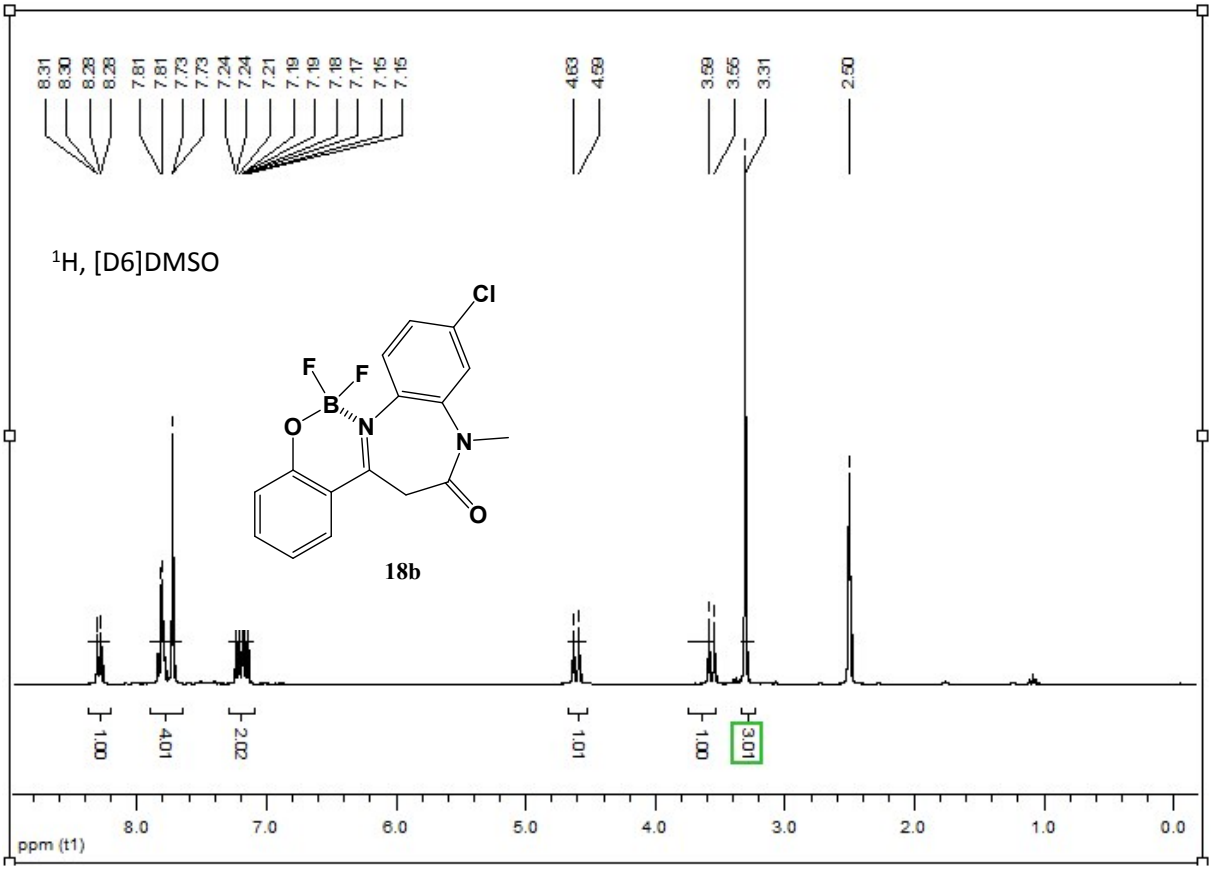
18a

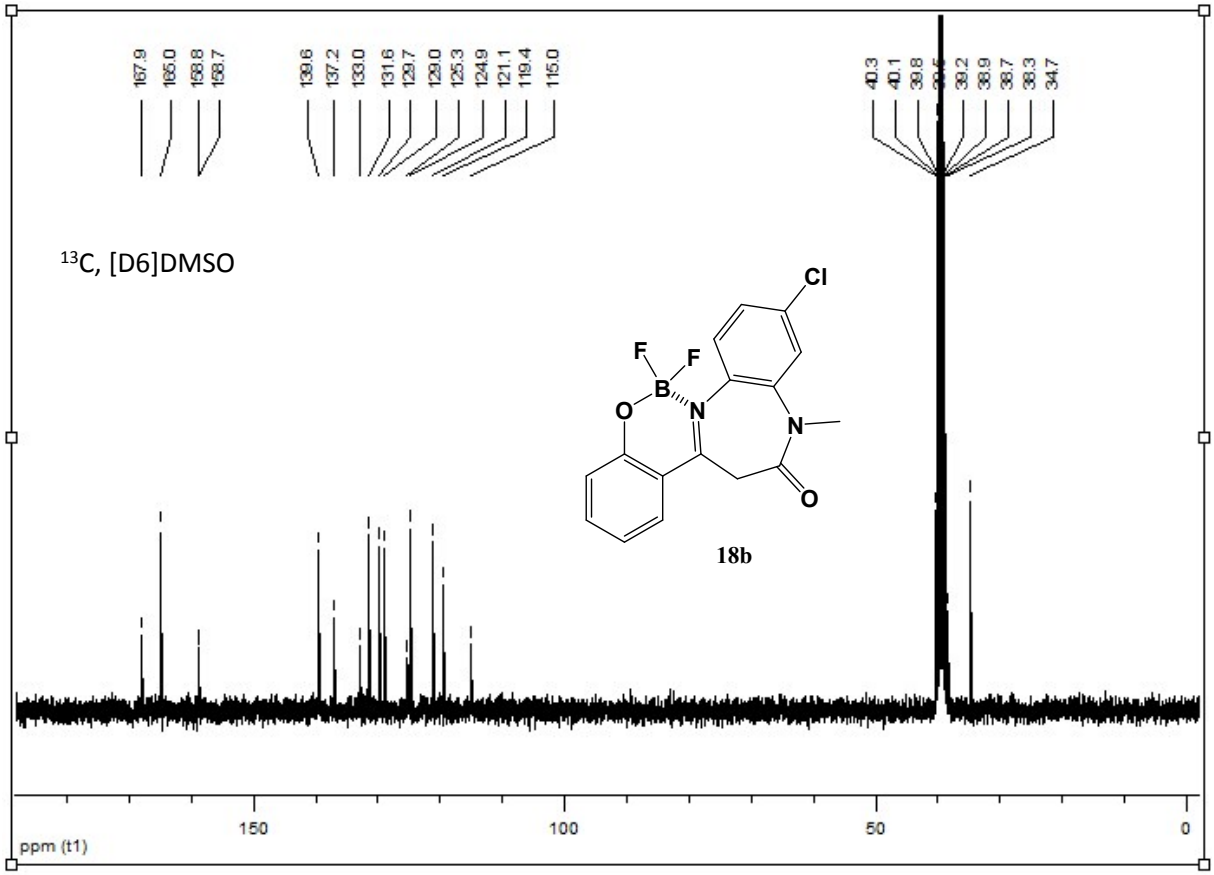


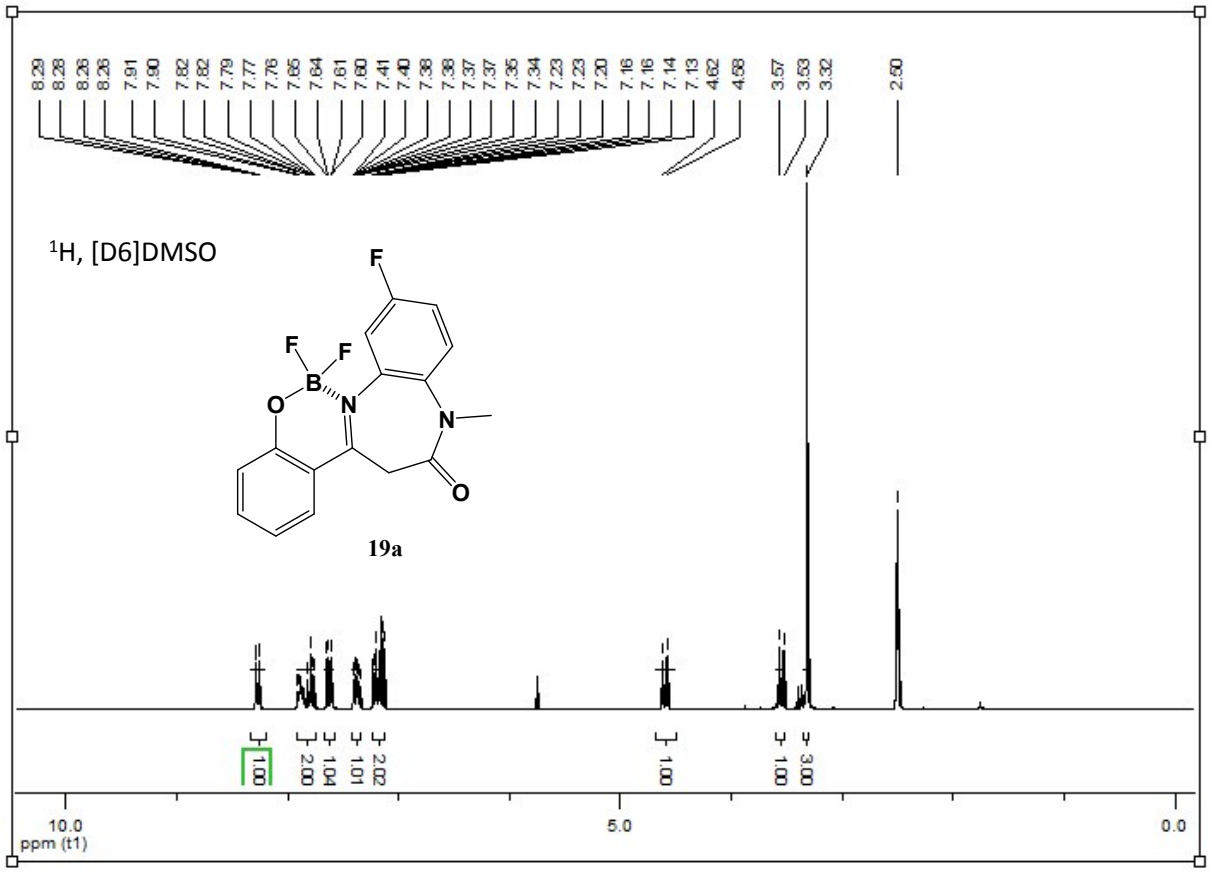
^{13}C, [D6]DMSO

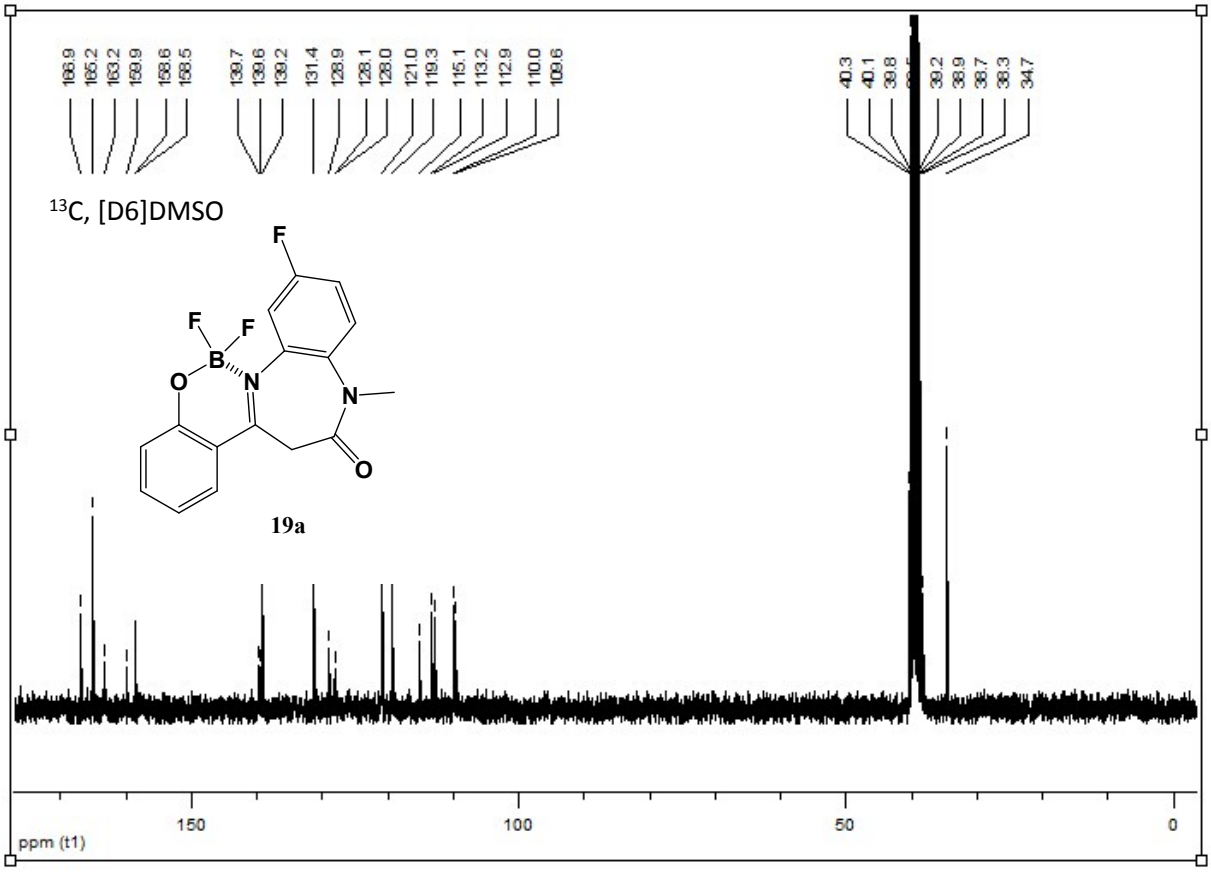
18a

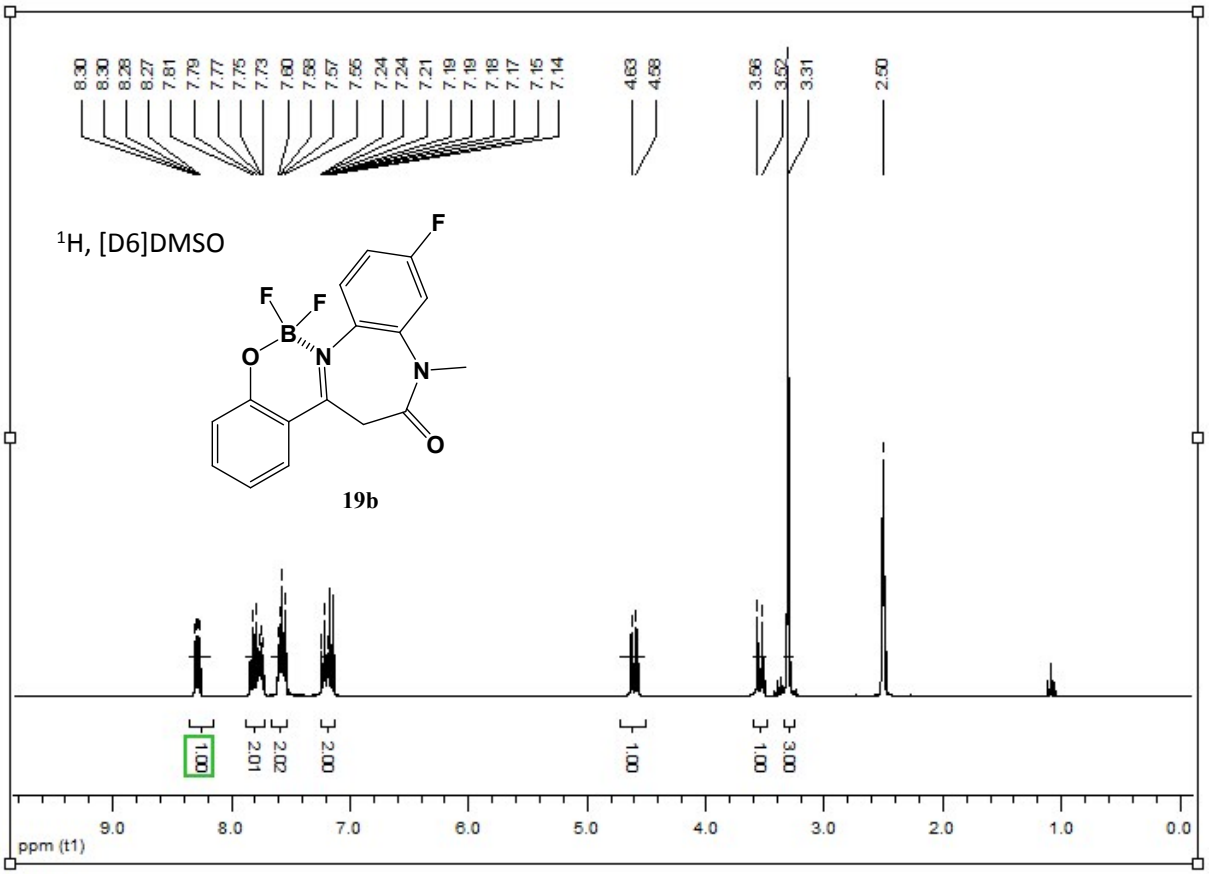
16 a


8.31
8.30
8.28
8.28
7.81
7.81
7.73
7.73
7.24
7.24
7.21
7.19
7.19
7.18
7.17
7.15
7.15
4.63
4.59
3.59
3.55
3.31
2.50
1H, [D6]DMSO
Cl
F
F
B
O
N
N
O
18b
1.00
4.01
2.02
1.01
1.00
3.01
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

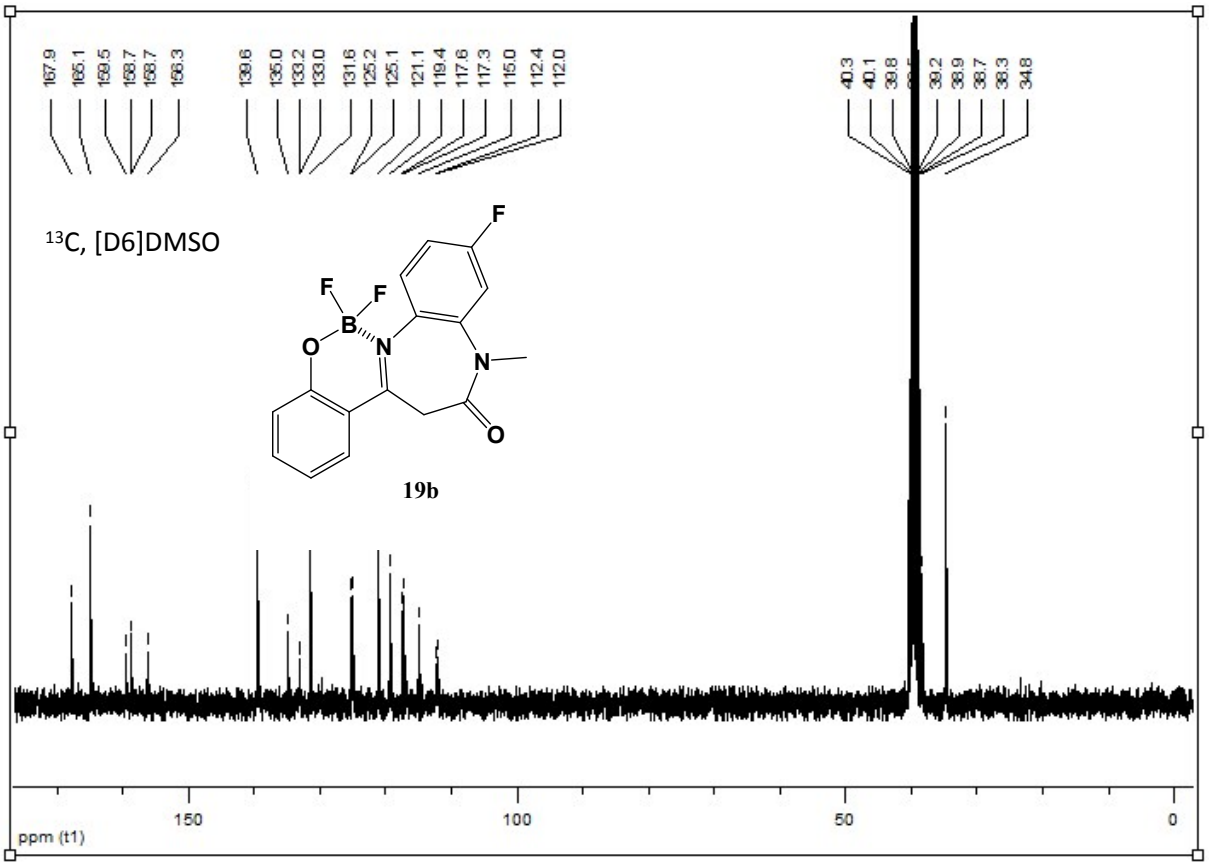

167.9
165.0
158.8
158.7
139.6
137.2
133.0
131.6
129.7
129.0
125.3
124.9
121.1
119.4
115.0
40.3
40.1
39.8
39.6
39.2
38.9
38.7
38.3
34.7
13C, [D6]DMSO
Cl
F
F
B
O
N
N
O
18b
150
100
50
0
ppm (t1)


8.29
8.28
8.26
8.26
7.91
7.90
7.82
7.82
7.79
7.77
7.76
7.65
7.64
7.61
7.60
7.41
7.40
7.38
7.38
7.37
7.37
7.35
7.34
7.23
7.23
7.20
7.16
7.16
7.14
7.13
4.62
4.58
3.57
3.53
3.32
2.50
1H, [D6]DMSO
F
F
F
B
O
N
N
O
19a
1.00
2.00
1.04
1.01
2.02
1.00
1.00
3.00
10.0
ppm (t1)
5.0
0.0

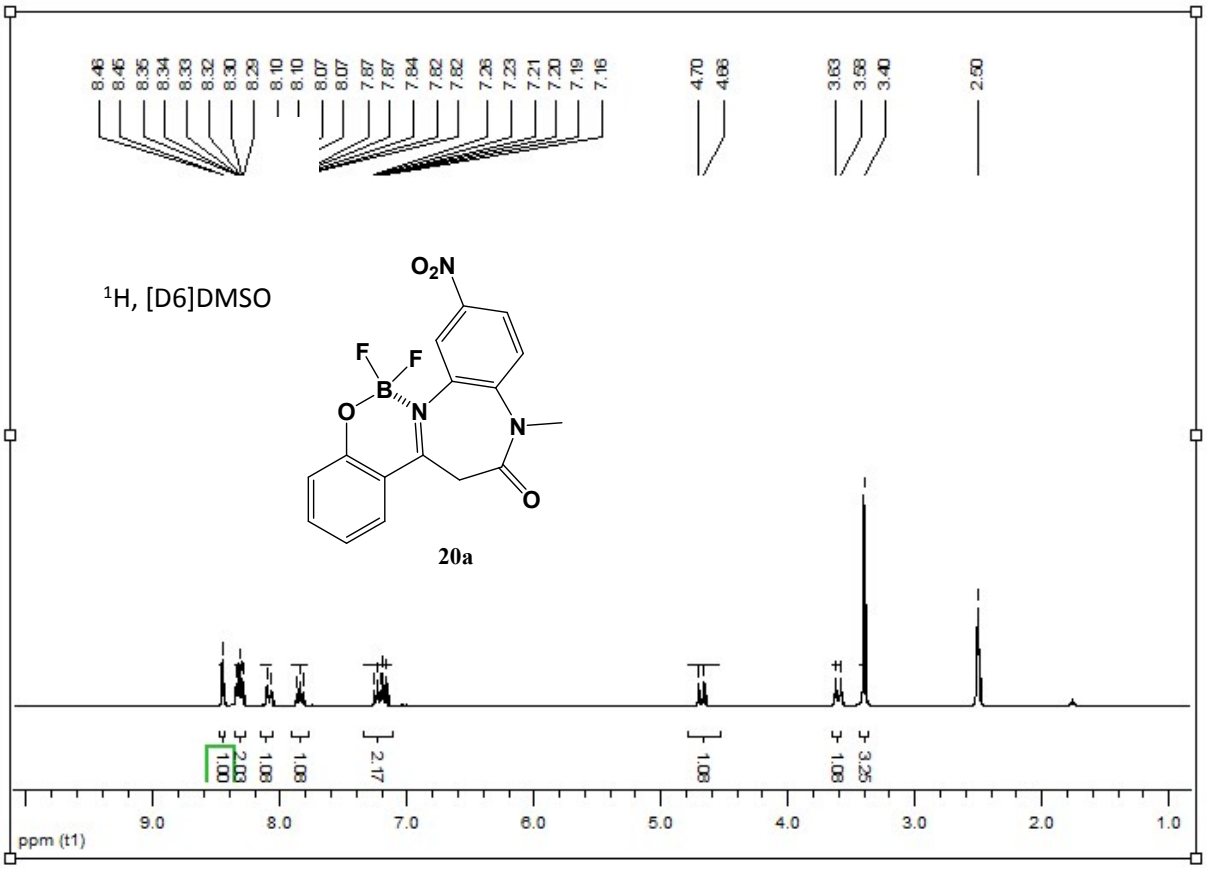

166.9
165.2
163.2
159.9
158.6
158.5
139.7
139.6
139.2
131.4
128.9
128.1
128.0
121.0
119.3
115.1
113.2
112.9
110.0
109.6
40.3
40.1
39.8
39.2
38.9
38.7
38.3
34.7
13C, [D6]DMSO
F
F
F
B
O
N
N
O
19a
150
100
50
0
ppm (t1)

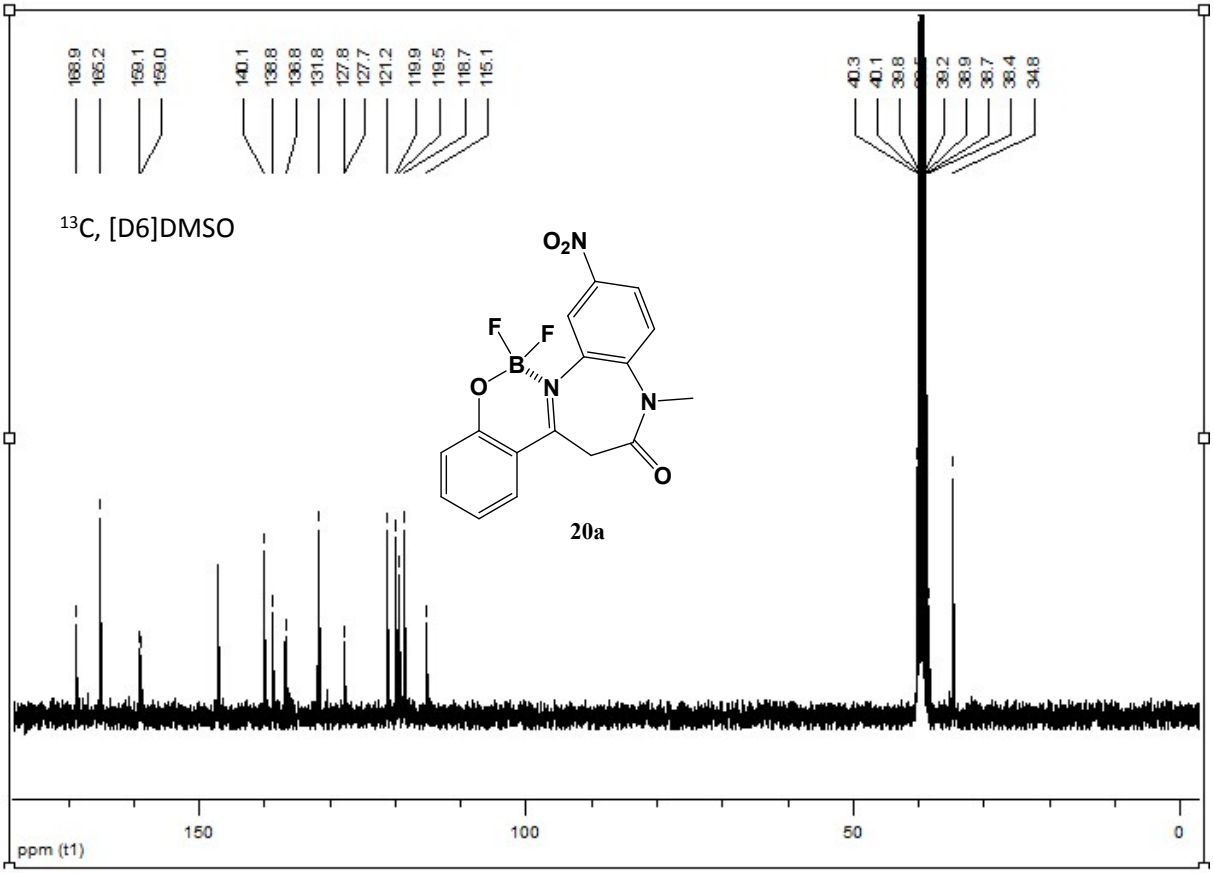

8.30
8.30
8.28
8.27
7.81
7.79
7.77
7.75
7.73
7.60
7.58
7.57
7.55
7.24
7.24
7.21
7.19
7.19
7.18
7.17
7.15
7.14
4.63
4.58
3.56
3.52
3.31
2.50
1H, [D6]DMSO
F
F
F
B
O
N
N
O
1.00
2.01
2.02
2.00
1.00
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

19b


167.9
165.1
159.5
158.7
158.7
156.3
139.6
135.0
133.2
133.0
131.6
125.2
125.1
121.1
119.4
117.6
117.3
115.0
112.4
112.0
40.3
40.1
39.8
39.2
38.9
38.7
38.3
34.8
13C, [D6]DMSO
F
F
F
B
O
N
N
O
150
100
50
0
ppm (t1)

19b


8.45
8.45
8.35
8.34
8.33
8.32
8.30
8.29
8.10
8.10
8.07
8.07
7.87
7.87
7.84
7.82
7.82
7.26
7.23
7.21
7.20
7.19
7.16
4.70
4.66
3.63
3.58
3.40
2.50
^{1}H, [D6]DMSO
O_2N
F
F
B
O
N
N
O
20a
1.00
2.08
3.08
1.08
2.17
1.08
3.08
3.26
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


168.9
165.2
159.1
159.0
140.1
138.8
136.8
131.8
127.8
127.7
121.2
119.9
119.5
118.7
115.1
40.3
40.1
39.8
39.2
38.9
38.7
38.4
34.8
^{13}C, [D6]DMSO
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B
O
N
N
O
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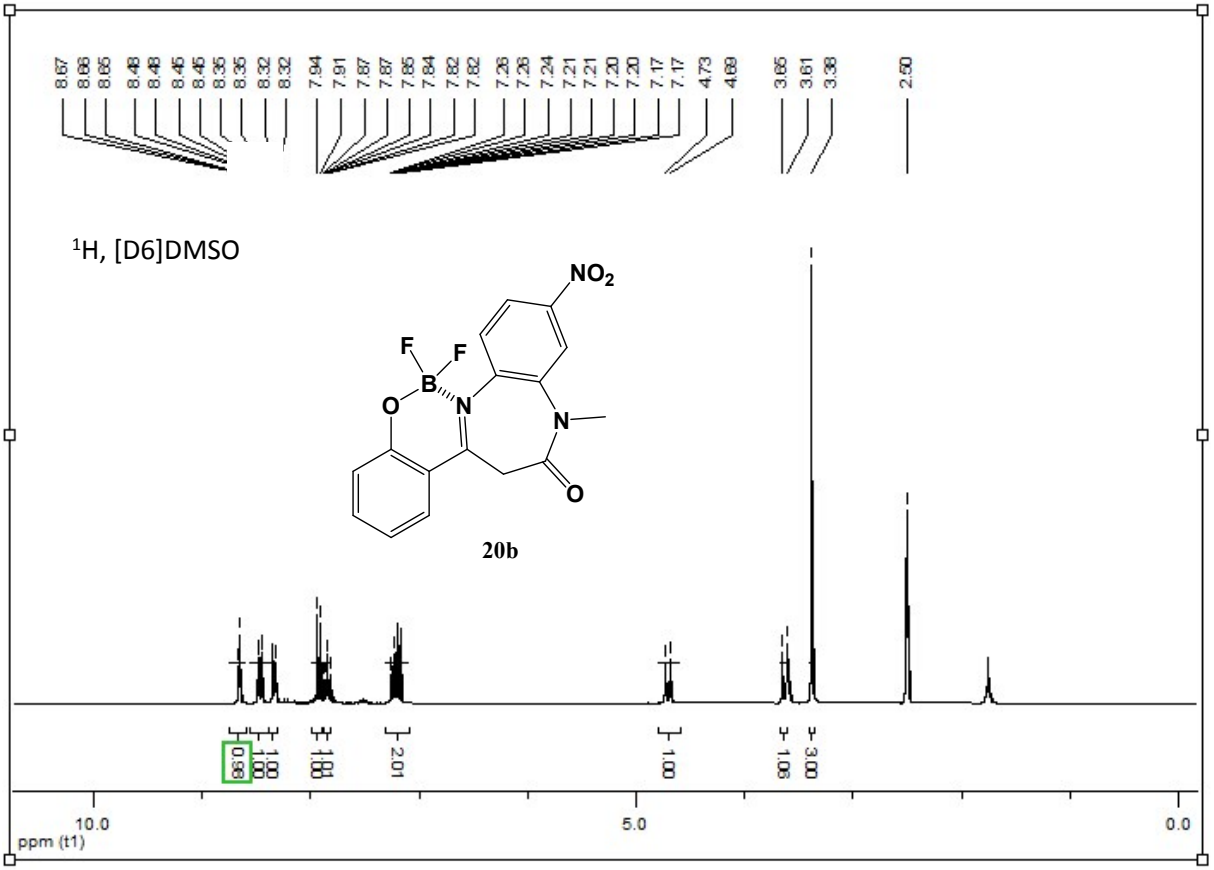
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7.82
7.26
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7.20
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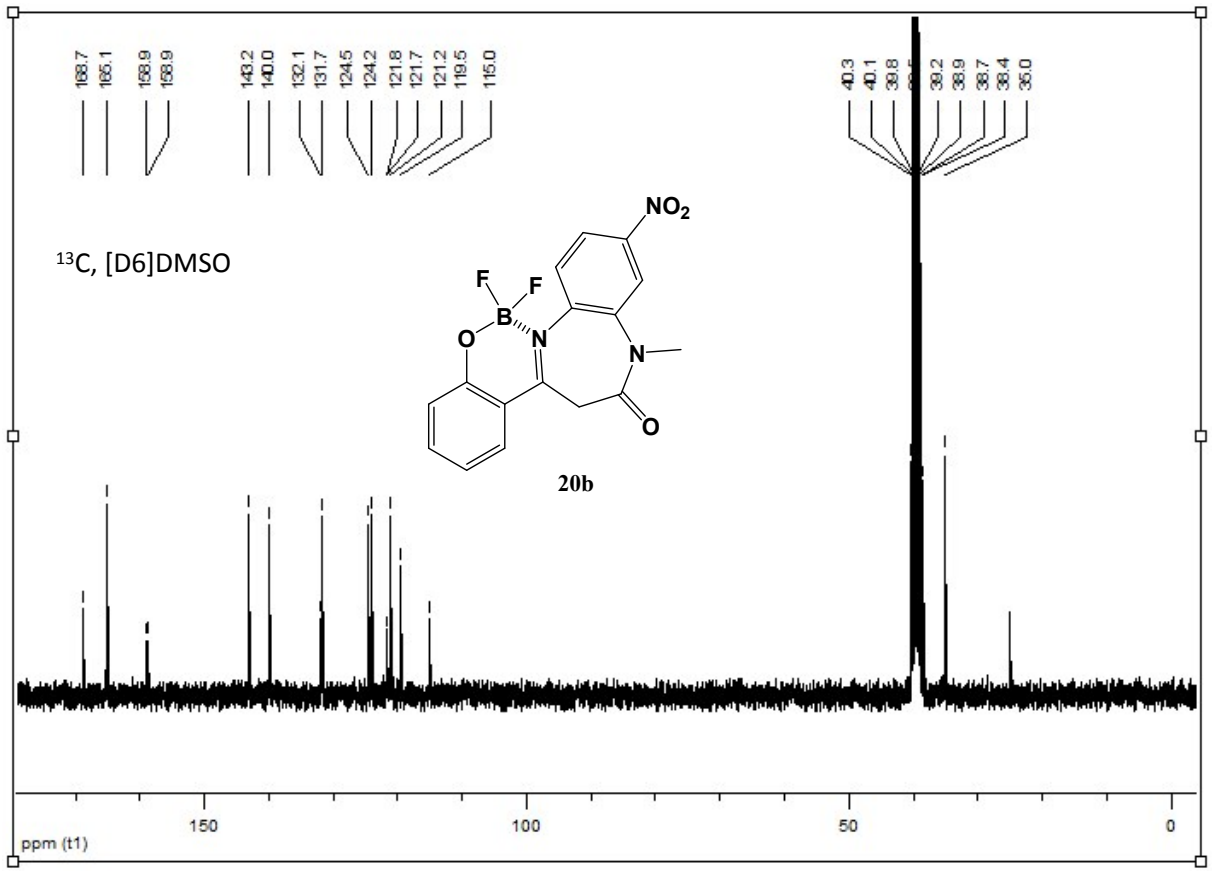
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119.5
115.0
40.3
40.1
39.8
39.2
38.9
38.7
38.4
35.0
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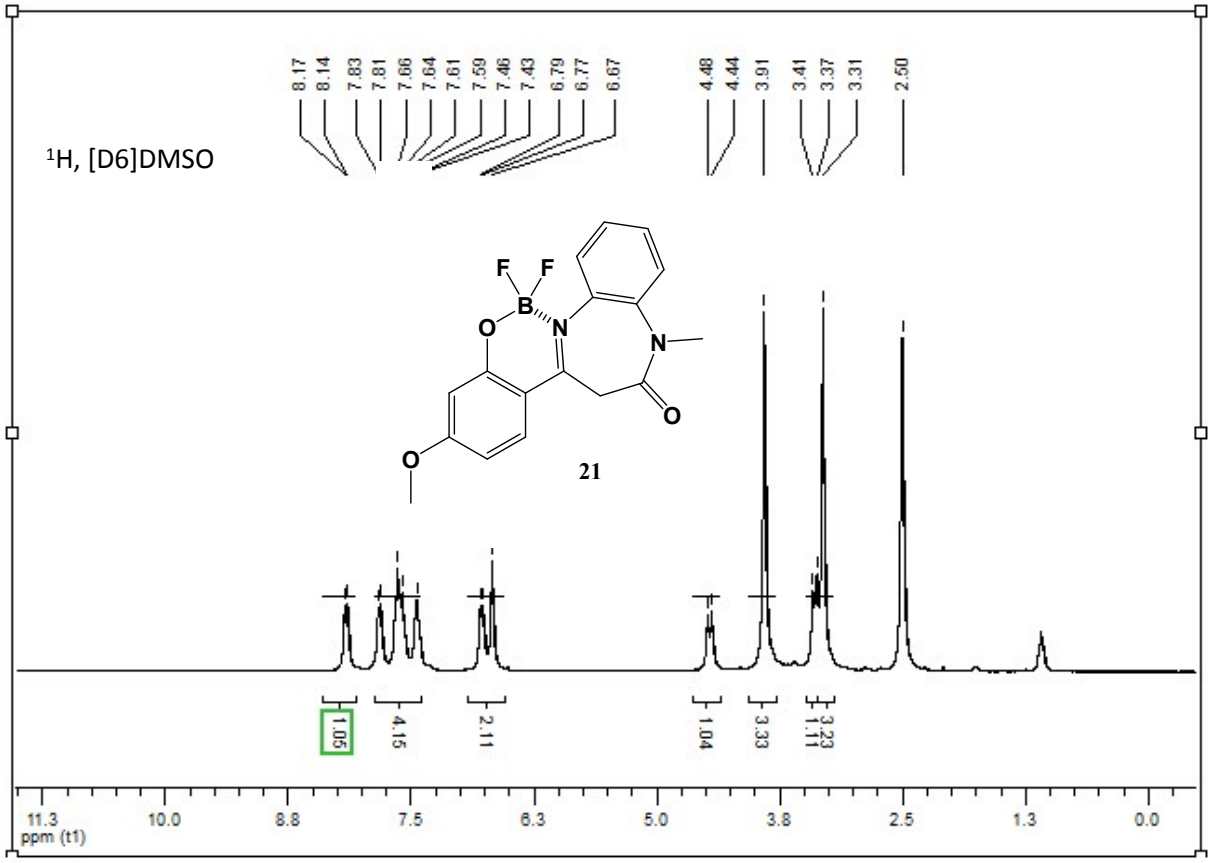
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4.48
4.44
3.91
3.41
3.37
3.31
2.50
1H, [D6]DMSO
F
F
B
O
N
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1.04
3.33
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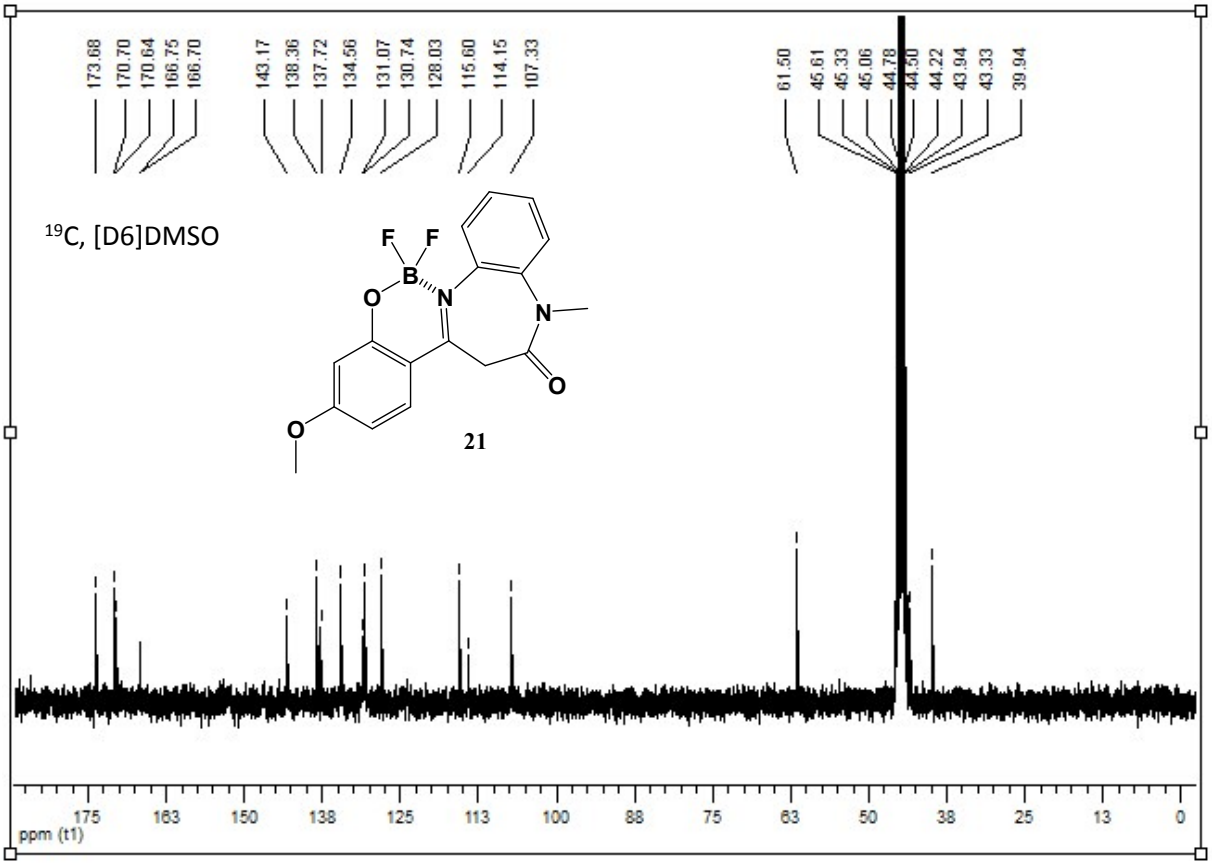
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107.33
61.50
45.61
45.33
45.06
44.78
44.50
44.22
43.94
43.33
39.94
19C, [D6]DMSO
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B
O
N
N
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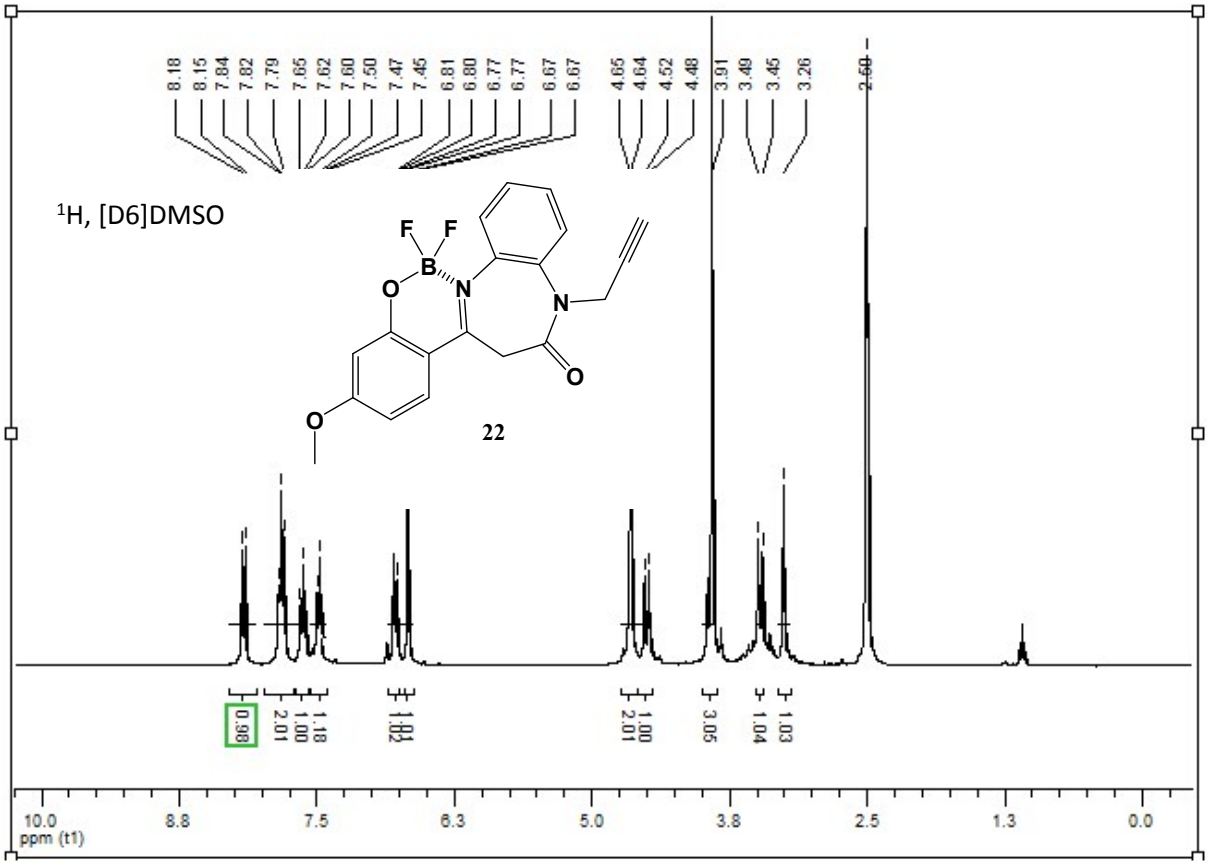
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3.26
2.50
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B
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N
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O
O
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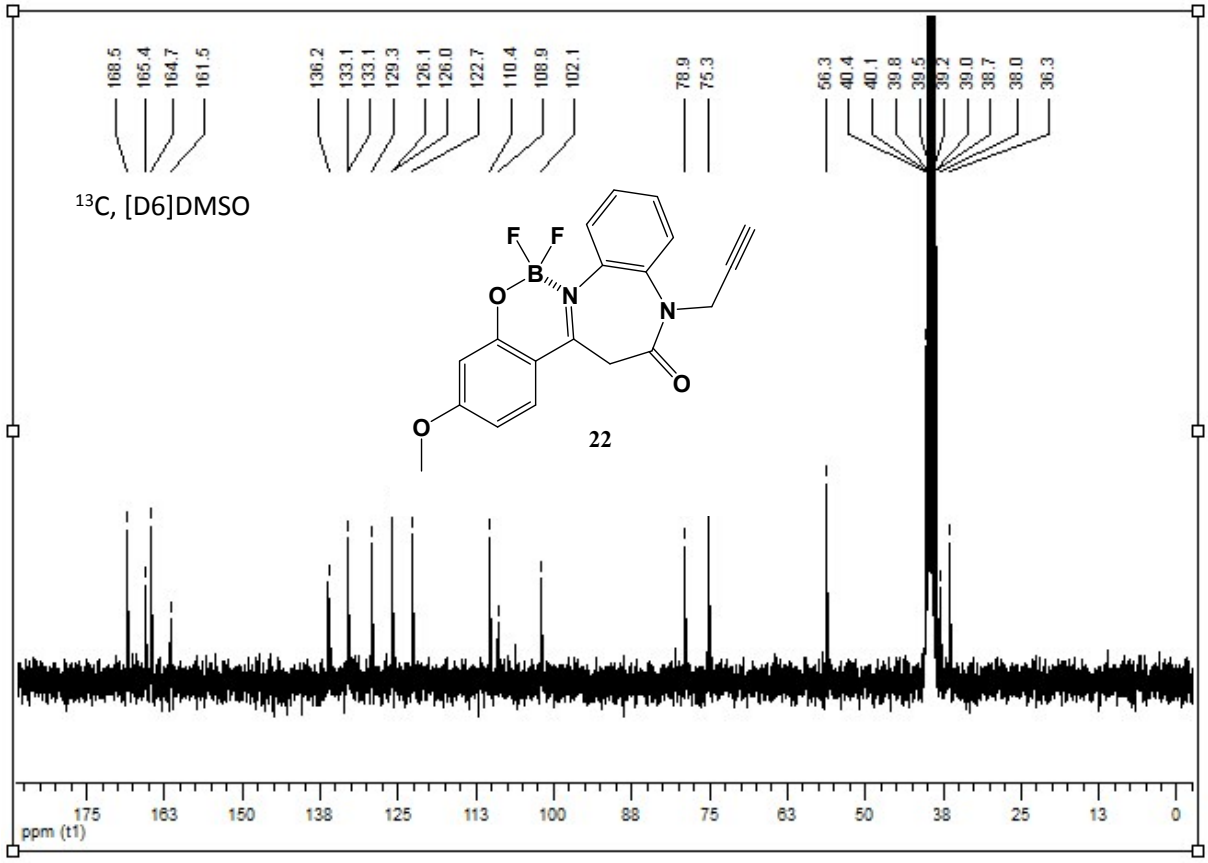
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78.9
75.3
56.3
40.4
40.1
39.8
39.5
39.2
39.0
38.7
38.0
36.3
[13]C, [D6]DMSO
F
F
B
O
N
N
O
O
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50
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25
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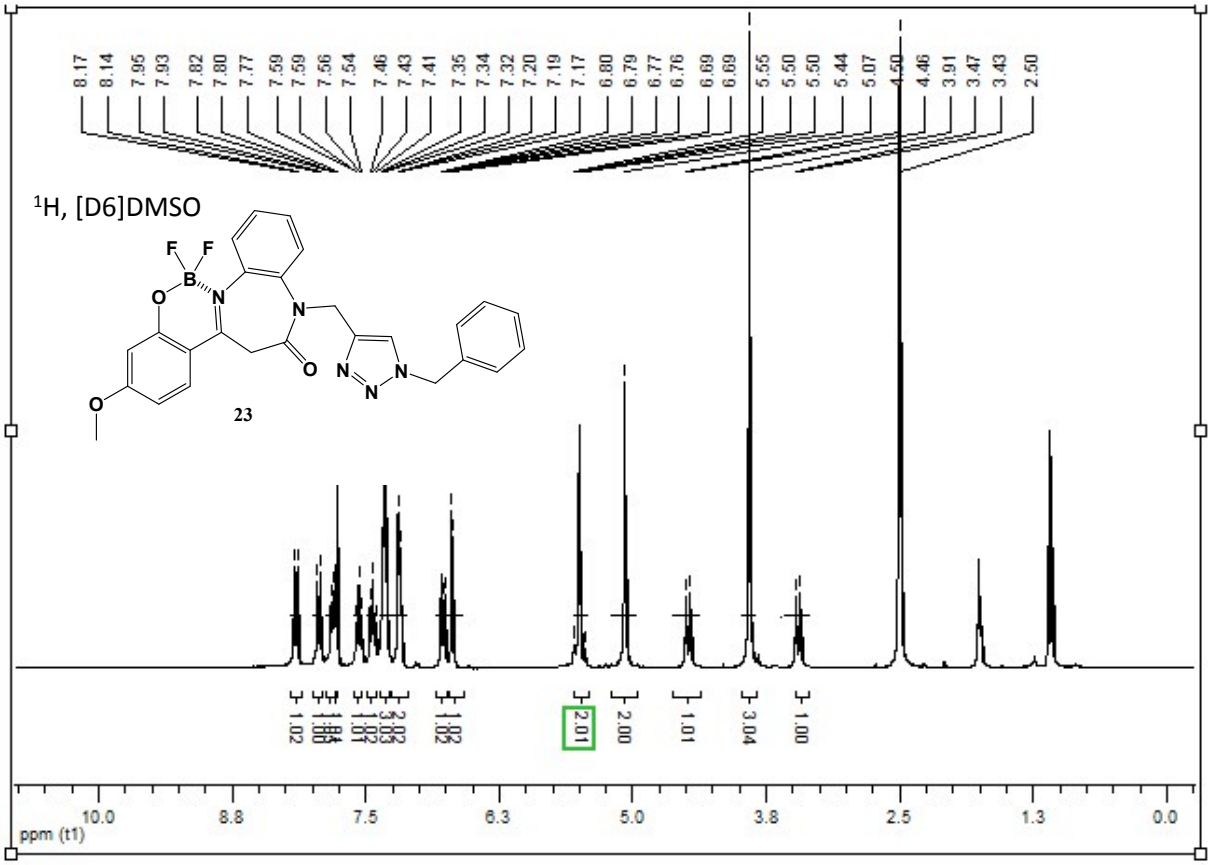
ppm (t1)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7.41
7.35
7.34
7.32
7.20
7.19
7.17
6.80
6.79
6.77
6.76
6.69
6.69
5.55
5.50
5.50
5.44
5.07
4.50
4.46
3.91
3.47
3.43
2.50
1H, [D6]DMSO
23
1.02
2.01
2.00
1.01
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6.3
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2.5
1.3
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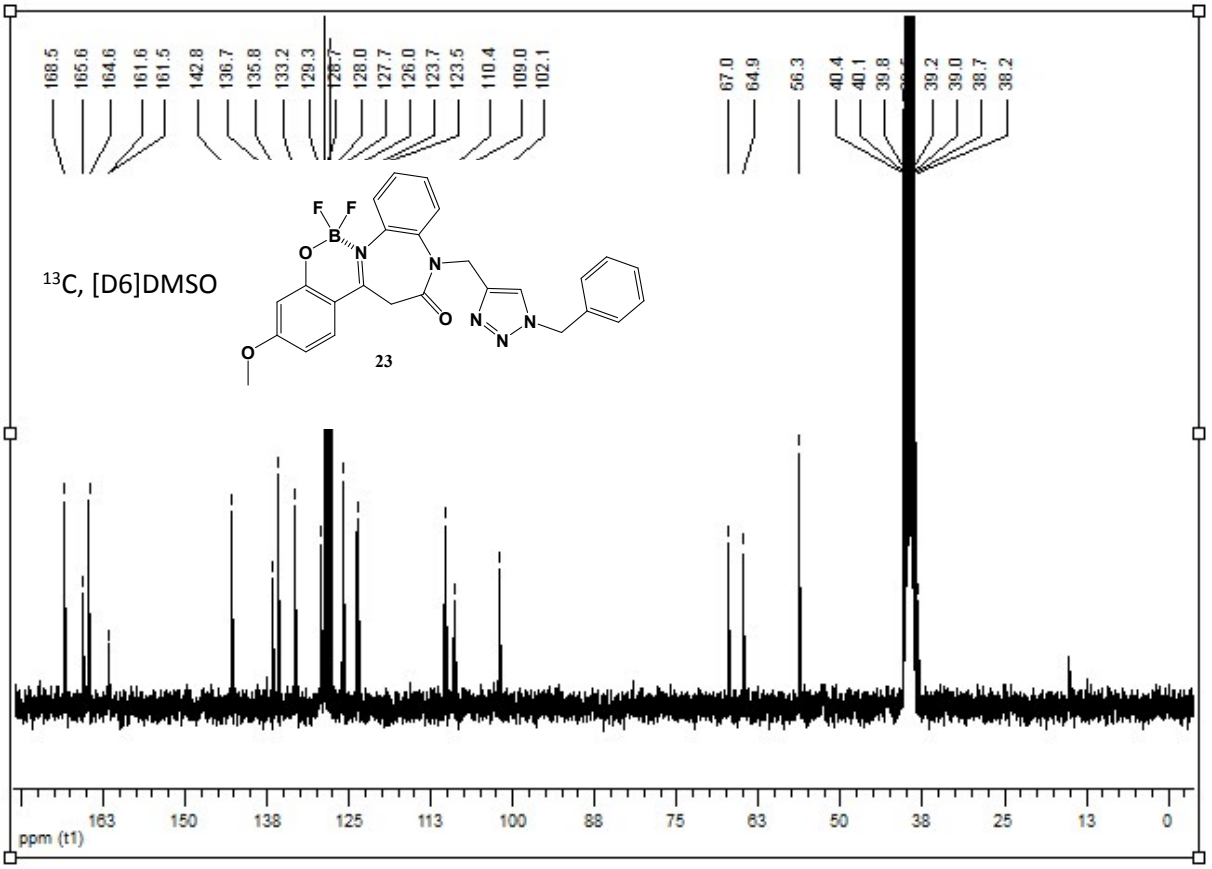
ppm (t1)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127.7
126.0
123.7
123.5
110.4
109.0
102.1
67.0
64.9
56.3
40.4
40.1
39.8
39.2
39.0
38.7
38.2
13C, [D6]DMSO
23
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)

II. Crystallographic data

DATA COLLECTION

The crystal structure of compound **13** [$C_{16}H_{13}BF_2N_2O_2$] has been determined from single crystal X-Ray diffraction. The chosen crystal was stuck on a glass fibre and mounted on the full three-circle goniometer of a Bruker SMART APEX diffractometer with a CCD area detector. Three sets of exposures (a total of 1800 frames) were recorded, corresponding to three ω scans (steps of 0.3°), for three different values of ϕ. The details of data collection are given in annexe 1.

The cell parameters and the orientation matrix of the crystal were preliminary determined by using SMART Software[1]. Data integration and global cell refinement were performed with SAINT Software[2]. Intensities were corrected for Lorentz, polarisation, decay and absorption effects (SAINT and SADABS Softwares) and reduced to F_O^2. The program package WinGX[3] was used for space group determination, structure solution and refinement.

DATA REFINEMENT

The standard space group $P2_1/c$ (n°14) was determined from systematic extinctions and relative F_O^2 of equivalent reflections. The structure was solved by direct methods[4]. Anisotropic displacement parameters were refined for all non-hydrogen atoms. Every Hydrogen atoms were located from subsequent difference Fourier syntheses and placed with geometrical constraints (SHELXL[5]). The final cycle of full-matrix least-square refinement on F^2 was based on 2887 observed reflections and 228 variable parameters and converged with unweighted and weighted agreement factors of:

R1 = 0.0357, wR2 = 0.0977 for 2458 reflections with $I>2\sigma I$ and R1 = 0.0420, wR2 = 0.1022 for all data.

The refinement data are given in annexe 1 table 2

CRYSTALLOGRAPHIC DATA AND STRUCTURAL DESCRIPTION

Crystallographic data

The crystal data are collected in Table 1. The full crystallographic parameters (atomic coordinates, bond length, angles and anisotropic displacements) are reported in annexe 2.

Chemical Formula	$C_{16}H_{13}BF_2N_2O_2$
Molecular Weight / $g.mol^{-1}$	314.09
Crystal System	Monoclinic
Space Group	P21/c
Z , Z' (asymmetric units per unit cell)	4,1
a / *Å*	10.173(1)
b / *Å*	7.374(1)
c / *Å*	19.011(2)
α / °	90
β / °	94.747(2)
γ / °	90
V / $Å^3$	1421.4(2)
d_{calc} / $g.cm^{-3}$	1.468
F(000) / e^-	648
Absorption coefficient μ (Mo$K\alpha_1$) / mm^{-1}	0.114

Structural description

The asymmetric unit is composed of one molecule of $C_{16}H_{13}N_2O_2B$ (figure1). The Fluorine atoms are presenting a disordered distribution on two crystallographic sites with 73/27 % of statistical occupancy.

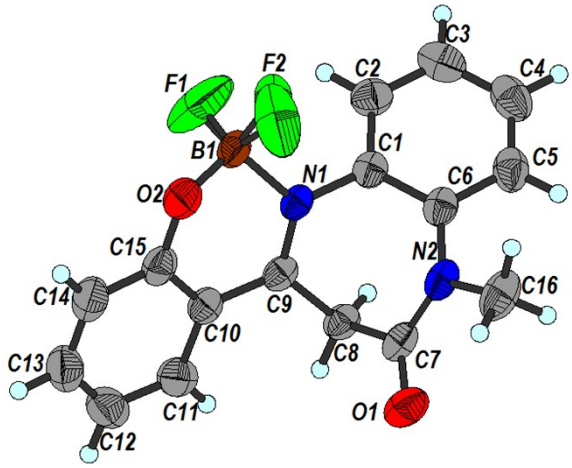

Figure 1: Asymmetric unit in ORTEP representation , with atom labels.

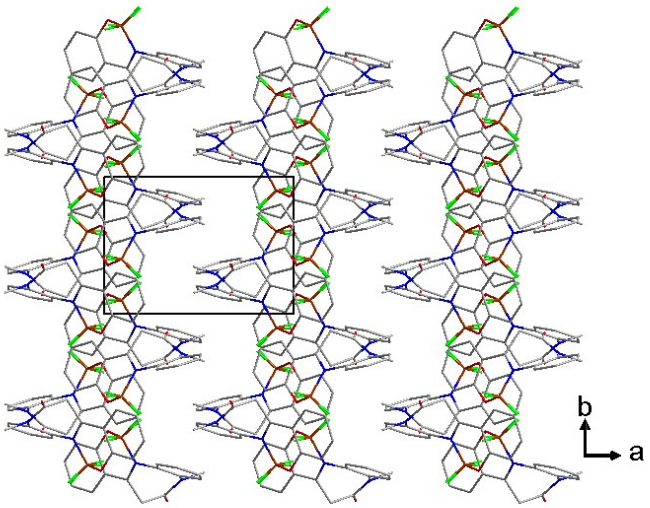

Figure 2: Projection along c.

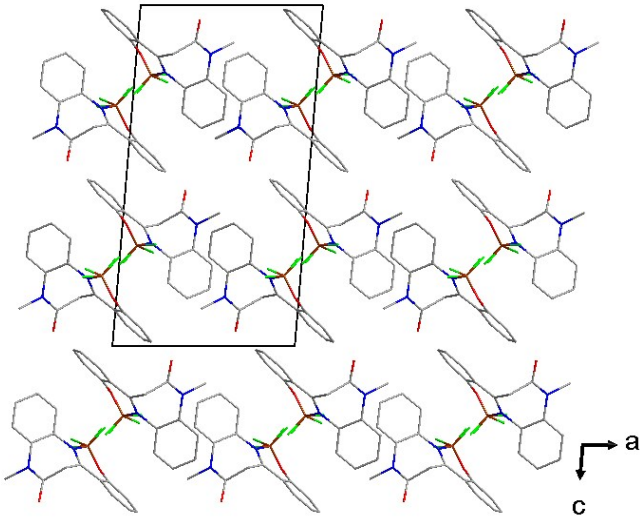

Figure 3: Projection along b

Sofwares :

(1)- SMART for WNT/2000 V5.622 (2001), Smart software reference manual, Bruker Advanced X Ray Solutions, Inc., Madison, Wisconsin, USA.

(2)- SAINT+ V6.02 (1999), Saint software reference manual, Bruker Advanced X Ray Solutions, Inc., Madison, Wisconsin, USA.

(3)-WinGX: Version 1.70.01: An integrated system of Windows Programs for the solution, refinement and analysis of Single Crystal X-Ray Diffraction Data, By LouisJ. Farrugia, Dept. of chemistry, University of Glasgow.

L. J. Farrugia (1999) J. Appl. Cryst. 32, 837-838.

(4)-include in WinGX suite : SIR 92: A. Altomare, G. Cascarano, & A. Gualardi (1993) J. Appl. Cryst. 26, 343-350; SHELXS-97: Sheldrick, G. M., (1990) Acta cryst, A46, 467.

(5)-include in WinGX suite: SHELXL-97 – a program for crystal structure refinement, G. M. .Sheldrick, University of Goettingen, Germany, 1997, release 97-2.

(6)-PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

ANNEXE 1 :

- Table 1 : DATA COLLECTION FOR COMPOUND 13

Date	**10/06/16**
Temperature / *K*	RT
Radiation	Mo-Kα_1 (λ = 0.71073Å)
Monochromator	Graphite
Collimator / *mm*	0.5
Generator set	50 kV 40mA
Crystal-detector distance / *mm*	60
Detector 2θ angle / °	-28
ω oscillations / °	-0.3
ω scan 1	χ = 54.7°, ϕ = 0°, -28° ≤ ω ≤ -208°
ω scan 2	χ = 54.7°, ϕ = 120°, -28° ≤ ω ≤ -208°
ω scan 3	χ = 54.7°, ϕ = 240°, -28° ≤ ω ≤ -208°
Time exposure / *s*	20
Total number of reflections	10887
Unique reflections [$F_o > 4.0\ \sigma(F_o)$]	2887 /2458
θ range / °	2.01 to 26.34
hkl range	-12≤h≤12, -9≤k≤9, -23≤l≤23
$R_{int} = \Sigma[\lvert F_O^2 - F_O^2(mean)\rvert] / \Sigma[F_O^2]$	0.018
Completeness to θ = 26.40 / *%*	99.7

- **Table 2 : REFINEMENT DATA FOR COMPOUND 13**

Number of reflections (n) (with $F_O > 4.0\ \sigma(F_O)$)	2458
Number of refined parameters (p) / restraints	228
Reflection / parameter ratio	
Final R indices [I>2sigma(I)]	R1=0.0357 wR2=0.0977
R indices (all data)	R1=0.0420, wR2=0.1022
Goodness of Fit indicator (Restrained GooF)	1.061
Maximum peak in Final Difference Map / $e^{-}Å^{-3}$	0.178
Maximum hole in Final Difference Map / $e^{-}Å^{-3}$	-0.173

$R_1 = \Sigma\ (\lvert\lvert F_O\rvert - \lvert F_C\rvert\rvert) / \Sigma\ \lvert F_O\rvert$

$wR_2 = [\ \Sigma\ [\ w\ (F_O^2 - F_C^2)^2]\ /\ \Sigma\ [\ w\ (F_O^2)^2\ ]\ ]^{1/2}$

$GooF = [\Sigma\ [\ w\ (F_O^2 - F_C^2)^2]\ /\ (n - p)\ ]^{1/2}$

ANNEXE 2 : CRYSTALLOGRAPHIC DATA

Table 1a: Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
C(1)	2820(1)	6230(2)	2426(1)	37(1)
C(2)	2780(1)	5949(2)	3147(1)	48(1)
C(3)	3888(2)	6221(2)	3601(1)	57(1)
C(4)	5053(2)	6749(2)	3335(1)	58(1)
C(5)	5106(1)	7040(2)	2626(1)	51(1)
C(6)	3991(1)	6802(2)	2159(1)	40(1)
C(7)	3186(1)	8205(2)	1050(1)	41(1)
C(8)	1992(1)	8767(2)	1431(1)	40(1)
C(9)	1207(1)	7079(2)	1513(1)	35(1)
C(10)	36(1)	6674(2)	1055(1)	39(1)
C(11)	-640(1)	8003(2)	636(1)	49(1)
C(12)	-1705(2)	7547(2)	184(1)	60(1)
C(13)	-2099(1)	5742(3)	125(1)	60(1)
C(14)	-1470(1)	4420(2)	527(1)	55(1)
C(15)	-416(1)	4878(2)	1010(1)	43(1)
C(16)	5256(1)	6561(2)	1098(1)	60(1)
B(1)	900(1)	3988(2)	2057(1)	42(1)
F(1)	1807(5)	2676(7)	2256(2)	60(1)
F(2)	56(2)	4253(5)	2594(2)	53(1)
F(1B)	230(30)	3960(40)	2549(9)	118(6)
F(2B)	1900(20)	2760(30)	2030(30)	109(8)
N(1)	1653(1)	5876(1)	1974(1)	35(1)
N(2)	4067(1)	7127(2)	1430(1)	43(1)
O(1)	3312(1)	8634(2)	442(1)	57(1)
O(2)	180(1)	3559(1)	1400(1)	51(1)

Table 1b: Hydrogen coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
H(2)	1999	5575	3324	57
H(3)	3854	6051	4084	68
H(4)	5807	6908	3640	69
H(5)	5895	7401	2454	61
H(8A)	2268	9282	1889	47
H(8B)	1472	9662	1157	47
H(11)	-360	9203	666	59
H(12)	-2163	8437	-82	72
H(13)	-2805	5431	-195	72
H(14)	-1744	3220	479	65
H(16A)	5027	6288	609	89
H(16B)	5624	5503	1333	89
H(16C)	5893	7524	1134	89

Table 2: Bond lengths (Å)

C(1)-C(2)	1.3901(18)	C(10)-C(11)	1.4060(19)
C(1)-C(6)	1.3979(17)	C(11)-C(12)	1.369(2)
C(1)-N(1)	1.4308(15)	C(11)-H(11)	0.93
C(2)-C(3)	1.376(2)	C(12)-C(13)	1.392(2)
C(2)-H(2)	0.93	C(12)-H(12)	0.93
C(3)-C(4)	1.382(2)	C(13)-C(14)	1.366(2)
C(3)-H(3)	0.93	C(13)-H(13)	0.93
C(4)-C(5)	1.371(2)	C(14)-C(15)	1.3938(19)
C(4)-H(4)	0.93	C(14)-H(14)	0.93
C(5)-C(6)	1.3919(19)	C(15)-O(2)	1.3380(17)
C(5)-H(5)	0.93	C(16)-N(2)	1.4703(16)
C(6)-N(2)	1.4147(18)	C(16)-H(16A)	0.96
C(7)-O(1)	1.2165(16)	C(16)-H(16B)	0.96
C(7)-N(2)	1.3605(17)	C(16)-H(16C)	0.96
C(7)-C(8)	1.5221(17)	B(1)-F(1B)	1.20(3)
C(8)-C(9)	1.4940(16)	B(1)-F(1)	1.369(5)
C(8)-H(8A)	0.97	B(1)-F(2B)	1.37(2)
C(8)-H(8B)	0.97	B(1)-F(2)	1.400(4)
C(9)-N(1)	1.3029(16)	B(1)-O(2)	1.4302(19)
C(9)-C(10)	1.4463(17)	B(1)-N(1)	1.6031(16)
C(10)-C(15)	1.4030(19)		

Table 3: Angles (°)

C(2)-C(1)-C(6)	119.89(12)	C(13)-C(12)-H(12)	120.2
C(2)-C(1)-N(1)	118.32(11)	C(14)-C(13)-C(12)	121.27(14)
C(6)-C(1)-N(1)	121.78(11)	C(14)-C(13)-H(13)	119.4
C(3)-C(2)-C(1)	120.46(13)	C(12)-C(13)-H(13)	119.4
C(3)-C(2)-H(2)	119.8	C(13)-C(14)-C(15)	119.69(15)
C(1)-C(2)-H(2)	119.8	C(13)-C(14)-H(14)	120.2
C(2)-C(3)-C(4)	119.61(14)	C(15)-C(14)-H(14)	120.2
C(2)-C(3)-H(3)	120.2	O(2)-C(15)-C(14)	118.55(13)
C(4)-C(3)-H(3)	120.2	O(2)-C(15)-C(10)	121.39(11)
C(5)-C(4)-C(3)	120.59(13)	C(14)-C(15)-C(10)	120.00(13)
C(5)-C(4)-H(4)	119.7	N(2)-C(16)-H(16A)	109.5
C(3)-C(4)-H(4)	119.7	N(2)-C(16)-H(16B)	109.5
C(4)-C(5)-C(6)	120.69(13)	H(16A)-C(16)-H(16B)	109.5
C(4)-C(5)-H(5)	119.7	N(2)-C(16)-H(16C)	109.5
C(6)-C(5)-H(5)	119.7	H(16A)-C(16)-H(16C)	109.5
C(5)-C(6)-C(1)	118.74(13)	H(16B)-C(16)-H(16C)	109.5
C(5)-C(6)-N(2)	119.62(11)	F(1B)-B(1)-F(1)	100.5(16)
C(1)-C(6)-N(2)	121.63(11)	F(1B)-B(1)-F(2B)	119.5(13)
O(1)-C(7)-N(2)	122.70(12)	F(1)-B(1)-F(2B)	19(2)
O(1)-C(7)-C(8)	122.18(12)	F(1B)-B(1)-F(2)	9.9(17)
N(2)-C(7)-C(8)	115.06(11)	F(1)-B(1)-F(2)	109.7(2)
C(9)-C(8)-C(7)	106.05(10)	F(2B)-B(1)-F(2)	129(2)
C(9)-C(8)-H(8A)	110.5	F(1B)-B(1)-O(2)	113.2(9)
C(7)-C(8)-H(8A)	110.5	F(1)-B(1)-O(2)	111.7(2)
C(9)-C(8)-H(8B)	110.5	F(2B)-B(1)-O(2)	98.3(18)
C(7)-C(8)-H(8B)	110.5	F(2)-B(1)-O(2)	111.52(16)
H(8A)-C(8)-H(8B)	108.7	F(1B)-B(1)-N(1)	113.5(12)
N(1)-C(9)-C(10)	119.22(11)	F(1)-B(1)-N(1)	108.9(2)
N(1)-C(9)-C(8)	118.55(11)	F(2B)-B(1)-N(1)	101.9(12)
C(10)-C(9)-C(8)	122.01(11)	F(2)-B(1)-N(1)	106.11(18)
C(15)-C(10)-C(11)	118.73(12)	O(2)-B(1)-N(1)	108.71(11)
C(15)-C(10)-C(9)	118.78(11)	C(9)-N(1)-C(1)	120.40(10)
C(11)-C(10)-C(9)	122.45(12)	C(9)-N(1)-B(1)	120.98(10)
C(12)-C(11)-C(10)	120.63(15)	C(1)-N(1)-B(1)	118.61(10)
C(12)-C(11)-H(11)	119.7	C(7)-N(2)-C(6)	122.14(10)
C(10)-C(11)-H(11)	119.7	C(7)-N(2)-C(16)	117.83(12)
C(11)-C(12)-C(13)	119.57(15)	C(6)-N(2)-C(16)	119.14(12)
C(11)-C(12)-H(12)	120.2	C(15)-O(2)-B(1)	119.95(11)

Table 4: Anisotropic displacement parameters ($Å^2$ x 10^3) The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2}$ U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	37(1)	30(1)	45(1)	-2(1)	6(1)	1(1)
C(2)	53(1)	46(1)	46(1)	2(1)	9(1)	3(1)
C(3)	72(1)	49(1)	48(1)	-2(1)	-5(1)	10(1)
C(4)	57(1)	43(1)	70(1)	-6(1)	-17(1)	4(1)
C(5)	40(1)	39(1)	74(1)	-1(1)	-1(1)	-2(1)
C(6)	37(1)	29(1)	53(1)	-2(1)	7(1)	-1(1)
C(7)	43(1)	36(1)	46(1)	-3(1)	13(1)	-11(1)
C(8)	41(1)	32(1)	46(1)	2(1)	9(1)	0(1)
C(9)	35(1)	33(1)	39(1)	-1(1)	14(1)	2(1)
C(10)	35(1)	43(1)	39(1)	-2(1)	12(1)	1(1)
C(11)	47(1)	53(1)	49(1)	3(1)	10(1)	7(1)
C(12)	48(1)	81(1)	50(1)	7(1)	5(1)	12(1)
C(13)	43(1)	90(1)	47(1)	-6(1)	4(1)	-6(1)
C(14)	47(1)	68(1)	50(1)	-7(1)	9(1)	-14(1)
C(15)	38(1)	50(1)	44(1)	-2(1)	13(1)	-4(1)
C(16)	48(1)	54(1)	81(1)	-7(1)	31(1)	-2(1)
B(1)	35(1)	34(1)	57(1)	7(1)	8(1)	-3(1)
F(1)	44(1)	34(1)	100(2)	10(1)	-6(1)	2(1)
F(2)	47(2)	59(1)	56(1)	11(1)	23(1)	-6(1)
F(1B)	177(15)	117(10)	66(6)	-17(6)	53(8)	-89(9)
F(2B)	71(7)	33(5)	220(20)	-15(9)	-37(10)	6(4)
N(1)	32(1)	32(1)	41(1)	-1(1)	11(1)	-1(1)
N(2)	36(1)	40(1)	55(1)	-2(1)	17(1)	-3(1)
O(1)	64(1)	62(1)	47(1)	3(1)	19(1)	-10(1)
O(2)	53(1)	40(1)	60(1)	1(1)	4(1)	-11(1)

Table 5: Torsion angles (°)

C(6)-C(1)-C(2)-C(3)	-0.4(2)	C(10)-C(9)-N(1)-B(1)	-1.82(16)
N(1)-C(1)-C(2)-C(3)	178.16(12)	C(8)-C(9)-N(1)-B(1)	-176.56(10)
C(1)-C(2)-C(3)-C(4)	-1.0(2)	C(2)-C(1)-N(1)-C(9)	130.07(12)
C(2)-C(3)-C(4)-C(5)	1.4(2)	C(6)-C(1)-N(1)-C(9)	-51.43(16)
C(3)-C(4)-C(5)-C(6)	-0.3(2)	C(2)-C(1)-N(1)-B(1)	-49.07(15)
C(4)-C(5)-C(6)-C(1)	-1.1(2)	C(6)-C(1)-N(1)-B(1)	129.43(12)
C(4)-C(5)-C(6)-N(2)	179.41(12)	F(1B)-B(1)-N(1)-C(9)	-100.6(15)
C(2)-C(1)-C(6)-C(5)	1.40(18)	F(1)-B(1)-N(1)-C(9)	148.3(2)
N(1)-C(1)-C(6)-C(5)	-177.08(11)	F(2B)-B(1)-N(1)-C(9)	130(2)
C(2)-C(1)-C(6)-N(2)	-179.08(11)	F(2)-B(1)-N(1)-C(9)	-93.69(17)
N(1)-C(1)-C(6)-N(2)	2.44(18)	O(2)-B(1)-N(1)-C(9)	26.37(15)
O(1)-C(7)-C(8)-C(9)	109.22(13)	F(1B)-B(1)-N(1)-C(1)	78.5(15)
N(2)-C(7)-C(8)-C(9)	-68.13(14)	F(1)-B(1)-N(1)-C(1)	-32.6(2)
C(7)-C(8)-C(9)-N(1)	73.19(13)	F(2B)-B(1)-N(1)-C(1)	-51(2)
C(7)-C(8)-C(9)-C(10)	-101.40(12)	F(2)-B(1)-N(1)-C(1)	85.44(16)
N(1)-C(9)-C(10)-C(15)	-14.00(16)	O(2)-B(1)-N(1)-C(1)	-154.50(10)
C(8)-C(9)-C(10)-C(15)	160.56(11)	O(1)-C(7)-N(2)-C(6)	172.79(12)
N(1)-C(9)-C(10)-C(11)	168.25(11)	C(8)-C(7)-N(2)-C(6)	-9.88(16)
C(8)-C(9)-C(10)-C(11)	-17.20(17)	O(1)-C(7)-N(2)-C(16)	3.74(19)
C(15)-C(10)-C(11)-C(12)	-1.05(19)	C(8)-C(7)-N(2)-C(16)	-178.93(11)
C(9)-C(10)-C(11)-C(12)	176.71(12)	C(5)-C(6)-N(2)-C(7)	-129.51(13)
C(10)-C(11)-C(12)-C(13)	-1.8(2)	C(1)-C(6)-N(2)-C(7)	50.98(17)
C(11)-C(12)-C(13)-C(14)	2.2(2)	C(5)-C(6)-N(2)-C(16)	39.39(17)
C(12)-C(13)-C(14)-C(15)	0.3(2)	C(1)-C(6)-N(2)-C(16)	-140.11(13)
C(13)-C(14)-C(15)-O(2)	179.70(12)	C(14)-C(15)-O(2)-B(1)	-156.93(12)
C(13)-C(14)-C(15)-C(10)	-3.20(19)	C(10)-C(15)-O(2)-B(1)	26.01(17)
C(11)-C(10)-C(15)-O(2)	-179.43(11)	F(1B)-B(1)-O(2)-C(15)	89.2(18)
C(9)-C(10)-C(15)-O(2)	2.73(17)	F(1)-B(1)-O(2)-C(15)	-158.1(2)
C(11)-C(10)-C(15)-C(14)	3.55(18)	F(2B)-B(1)-O(2)-C(15)	-143.6(18)
C(9)-C(10)-C(15)-C(14)	-174.29(11)	F(2)-B(1)-O(2)-C(15)	78.7(2)
C(10)-C(9)-N(1)-C(1)	179.06(10)	N(1)-B(1)-O(2)-C(15)	-37.92(15)
C(8)-C(9)-N(1)-C(1)	4.32(15)		

Table 6: Calculated reflections from PowderCell*

h	k	l	2θ/°	d/Å	I/rel.	|F(hkl)|
1	0	0	8.72	10.14	51.01	59.64
0	0	2	9.33	9.47	100.00	89.42
-1	0	2	12.24	7.23	40.24	74.62
0	1	1	12.87	6.87	16.86	35.95
1	0	2	13.30	6.65	10.34	41.16
1	1	0	14.84	5.96	36.99	61.55
0	1	2	15.21	5.82	50.92	74.05
-1	1	1	15.35	5.77	93.52	101.24
-1	1	2	17.17	5.16	3.18	20.95
2	0	0	17.48	5.07	7.46	46.22
1	1	2	17.94	4.94	5.45	28.67
-2	0	2	19.15	4.63	3.55	34.99
-1	1	3	19.96	4.45	59.56	105.81
-1	0	4	20.01	4.43	6.25	48.59
2	0	2	20.53	4.32	4.79	43.70
1	1	3	20.96	4.23	8.67	42.48
2	1	0	21.25	4.18	41.19	93.92
1	0	4	21.34	4.16	17.15	86.05
-2	1	1	21.45	4.14	5.88	35.82
2	1	1	22.08	4.02	3.94	30.23
0	1	4	22.29	3.99	20.84	70.20
-1	1	4	23.39	3.80	15.63	63.94
0	2	0	24.12	3.69	12.59	83.77
1	1	4	24.54	3.62	11.95	58.79
0	2	1	24.58	3.62	3.99	34.03
-2	0	4	24.62	3.61	60.15	187.14
-2	1	3	24.72	3.60	14.81	65.93
1	2	0	25.69	3.47	9.58	55.23
-1	2	1	25.99	3.43	10.19	57.67
1	2	1	26.25	3.39	60.12	141.56
2	1	3	26.35	3.38	2.07	26.35
-1	2	2	27.13	3.28	17.05	78.05
-2	1	4	27.47	3.24	6.77	49.81
0	2	3	28.00	3.18	3.26	35.28
3	1	1	29.79	3.00	3.56	39.41

Source: Cu-$K_{\alpha 1}$ (λ = 1.540598 Å)

Condition on reflections: I ≥ 2

Range (2θ): From 3° to 30°

*PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.