Supplementary Materials

Q-Tube®-Assisted Alkylation and Arylation of Xanthines and Other N-H-Containing Heterocycles in Water

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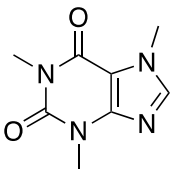
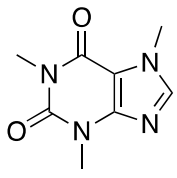


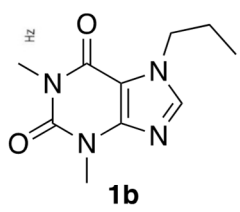
1a

7.51
3.99
3.58
3.40
0.82
3.00
2.92
3.01
f1 (ppm)



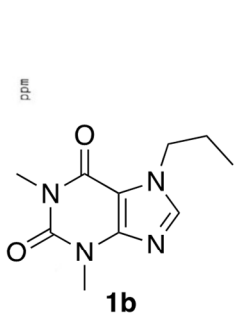
1a

155.81
152.10
149.08
141.80
107.97
77.80
77.48
77.16
34.01
30.15
28.33
f1 (ppm)


Hz
O
N
N
O
N
N

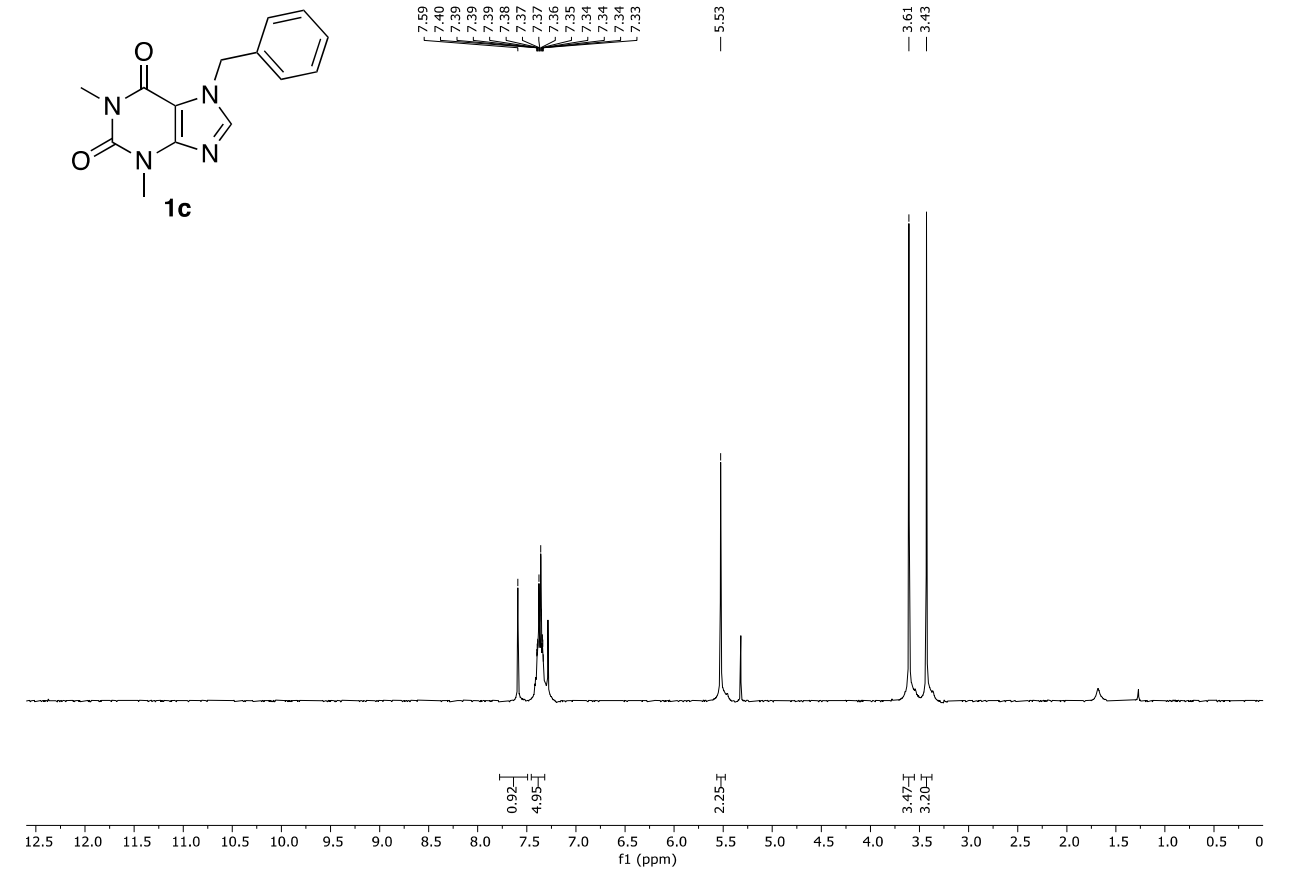
1b

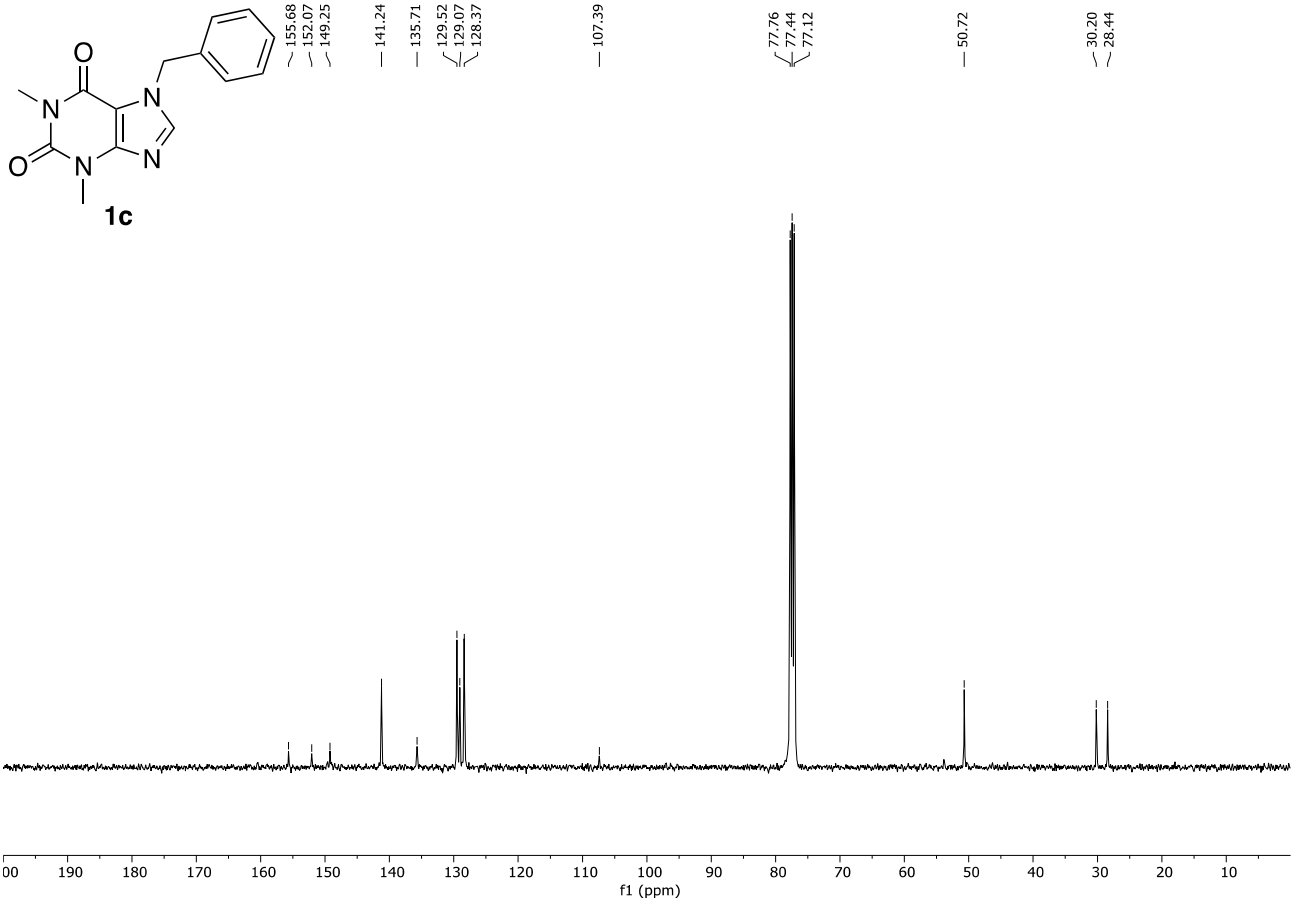
3011.45
2908.51
1703.16
1696.09
1689.07
1430.14
1357.24
776.30
768.93
761.66
754.49
747.26
740.17
493.88
379.36
371.97
364.59
Integral
0.9047
2.1663
3.0000
2.8601
2.2853
3.1827
ppm
9
8
7
6
5
4
3
2
1

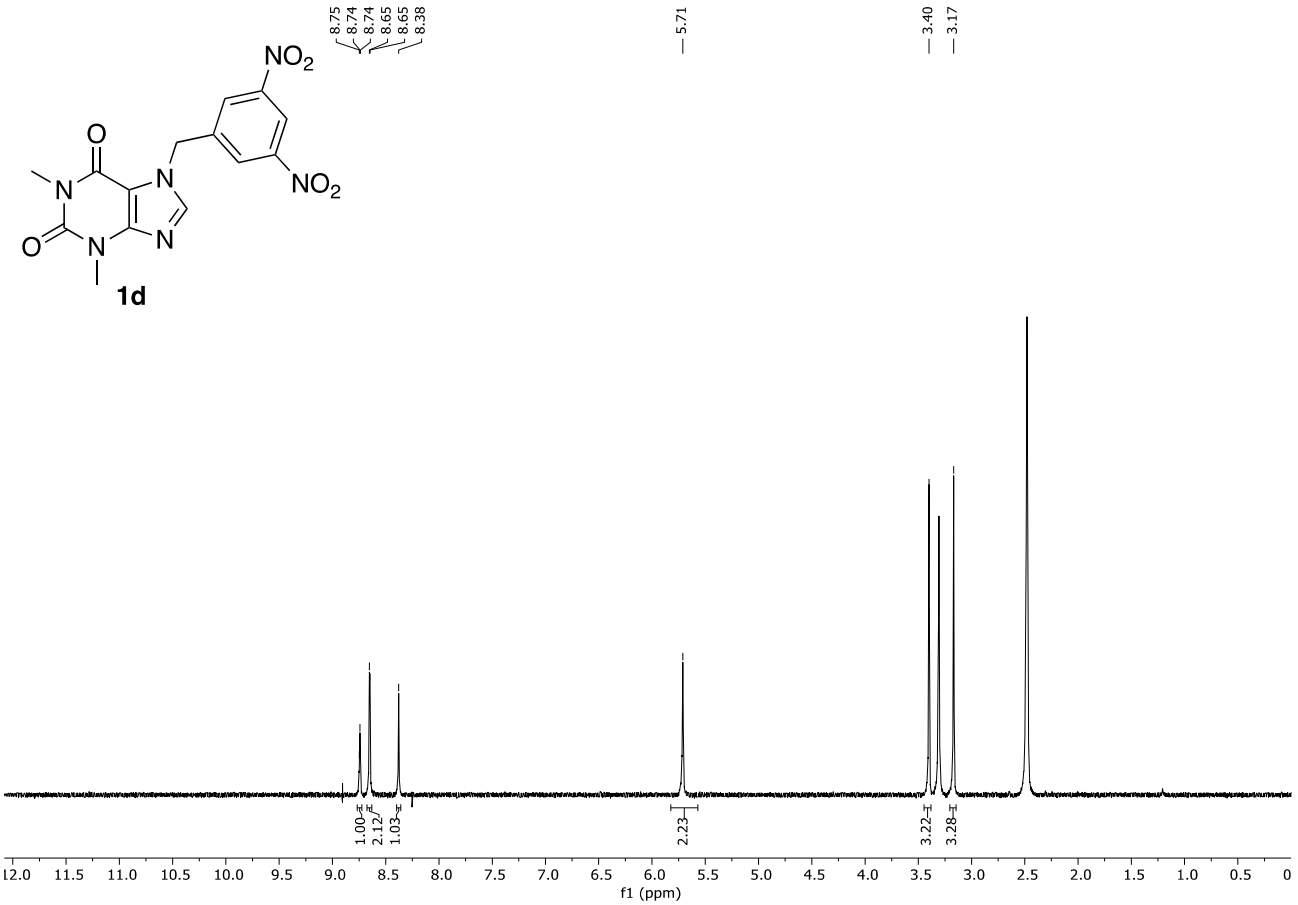

O
N
N
O
N
N

1b

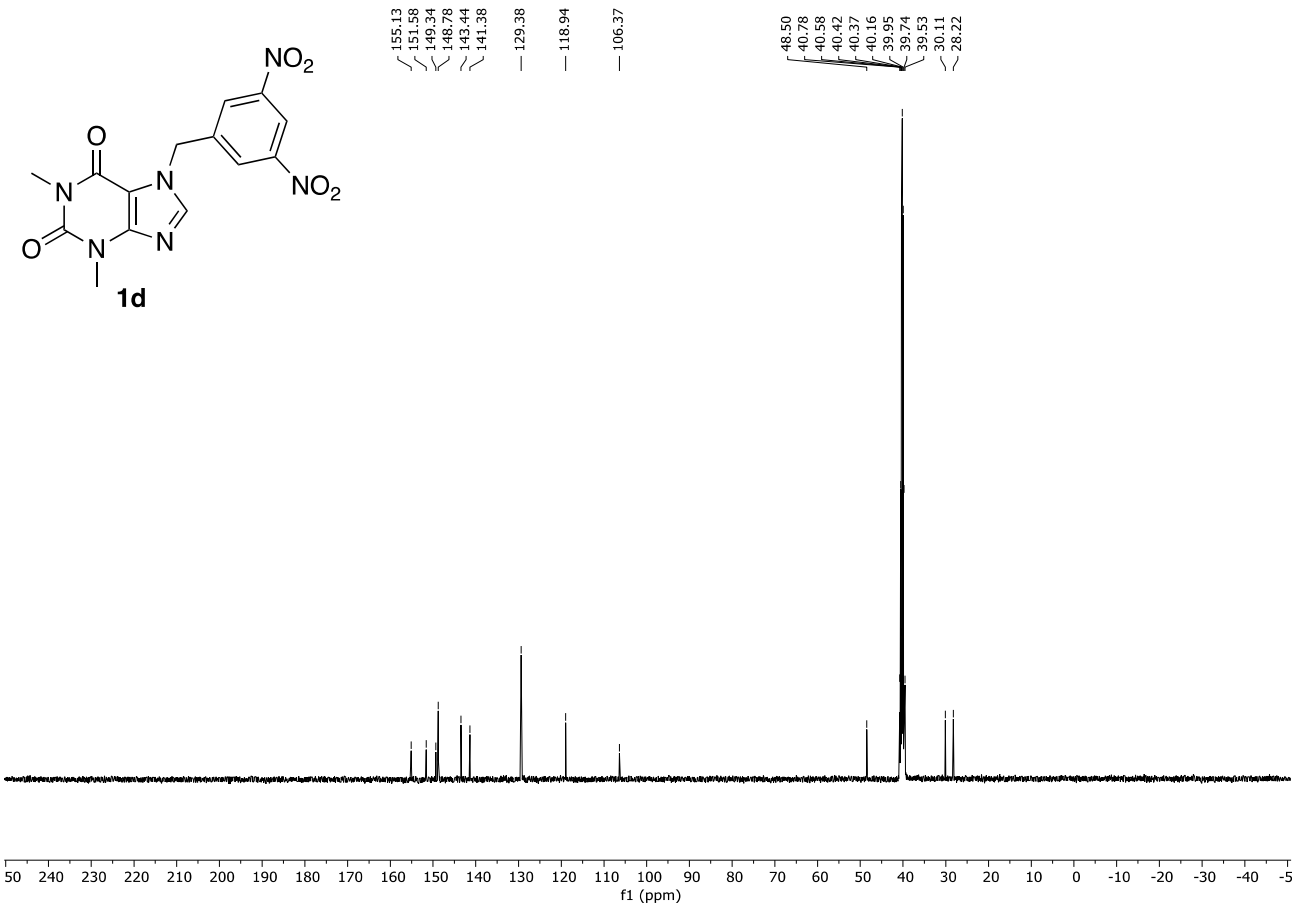
AI04
ppm
155.50876
152.08804
149.33023
141.24377
130.10008
107.37856
77.76799
77.45035
77.13260
49.22972
30.14214
28.35611
24.56569
11.19839
ppm
200
180
160
140
120
100
80
60
40
20


1c
7.59
7.40
7.39
7.39
7.39
7.38
7.37
7.37
7.36
7.35
7.34
7.34
7.34
7.33
5.53
3.61
3.43
0.92
4.95
2.25
3.47
3.20
f1 (ppm)

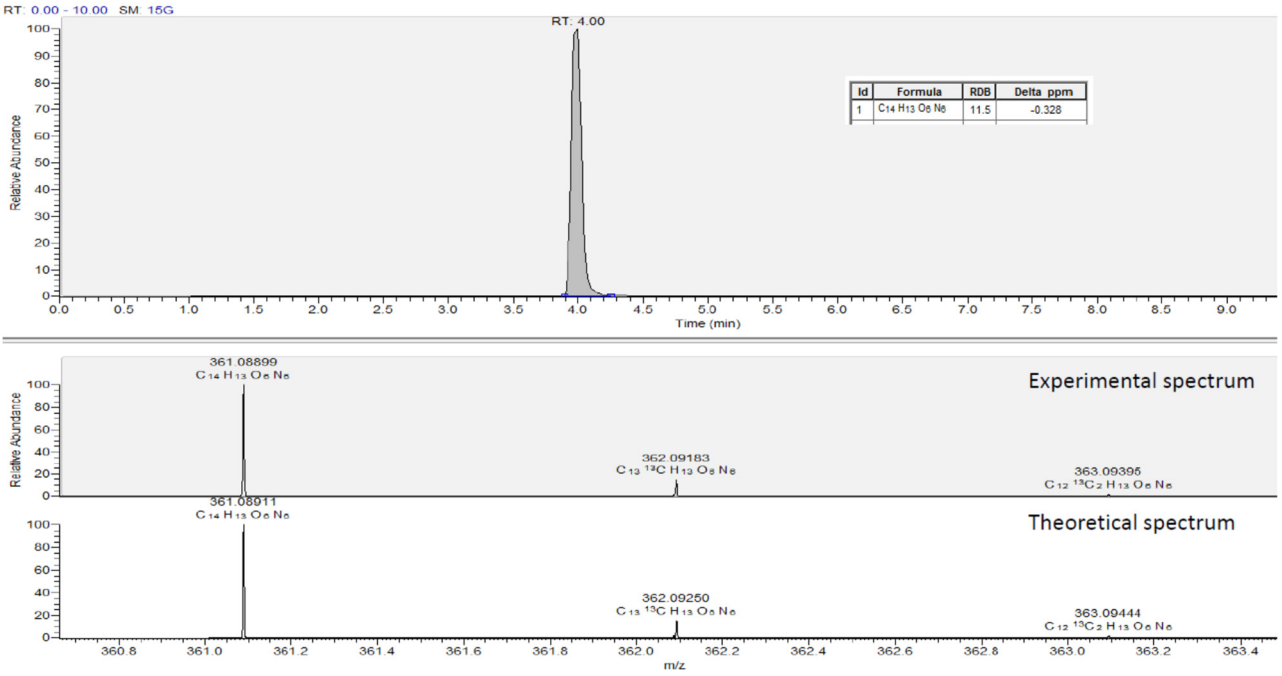

1c
155.68
152.07
149.25
141.24
135.71
129.52
129.07
128.37
107.39
77.76
77.44
77.12
50.72
30.20
28.44
f1 (ppm)

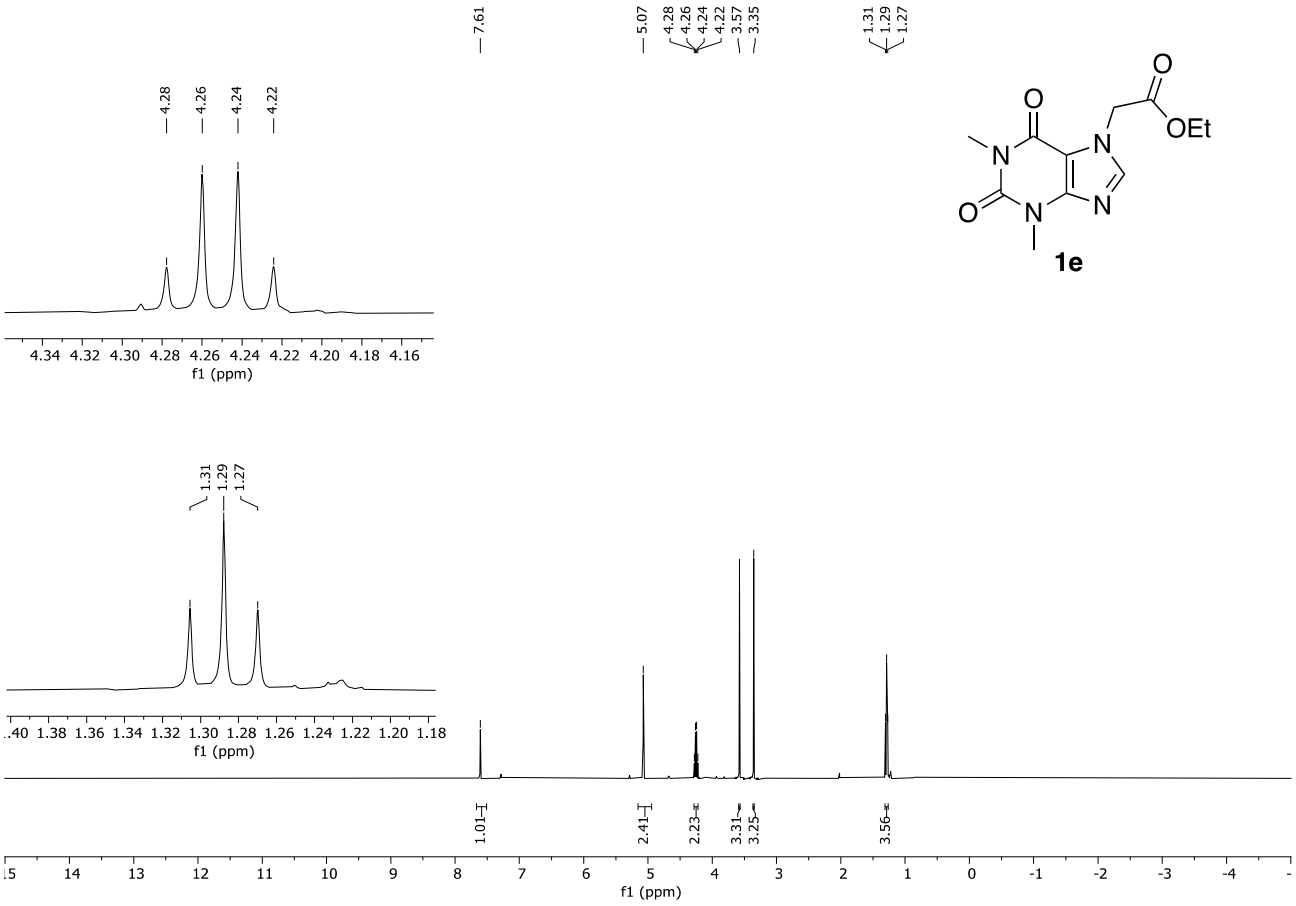

8.75
8.74
8.74
8.65
8.65
8.38
5.71
3.40
3.17
1.00
2.12
1.03
2.23
3.22
3.28
f1 (ppm)

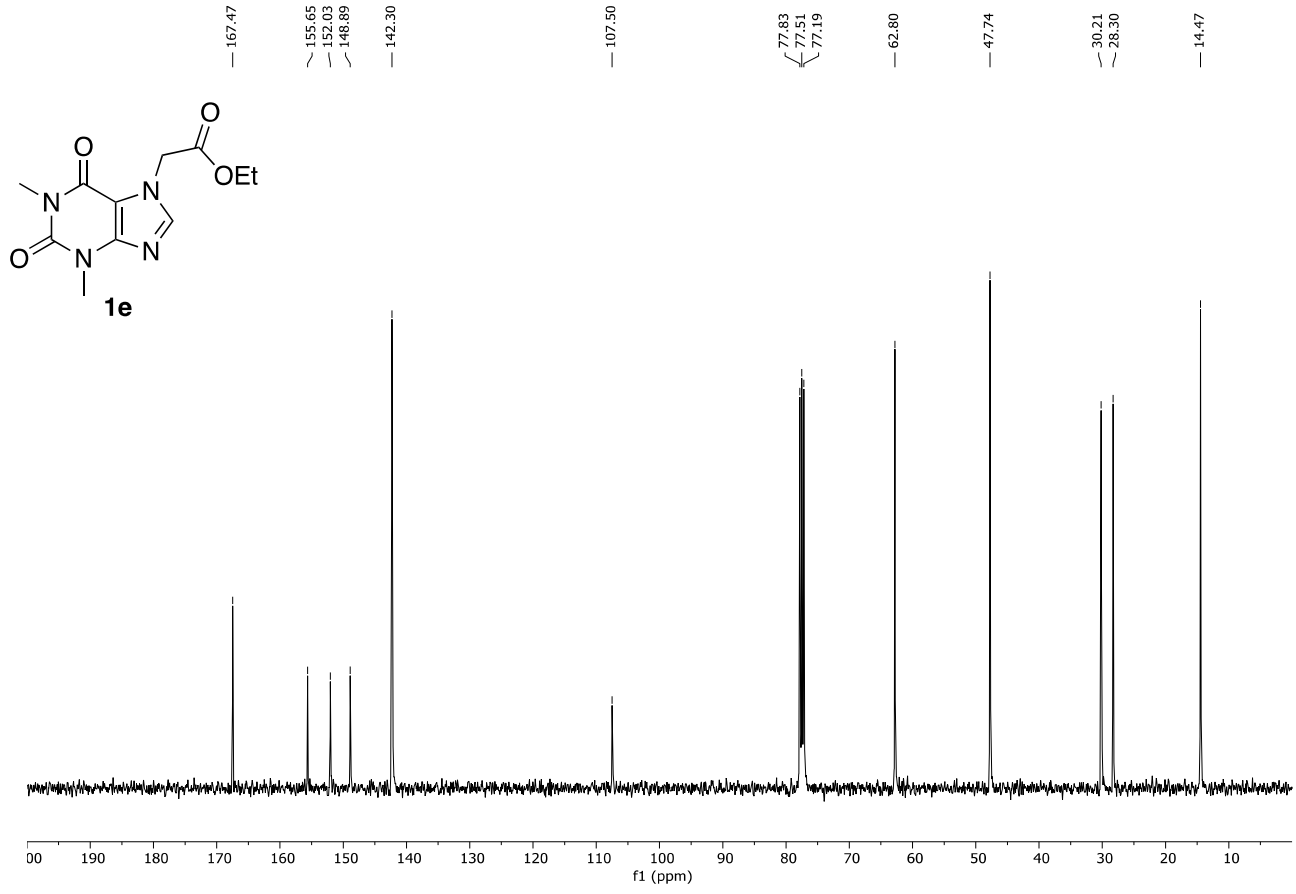
1d

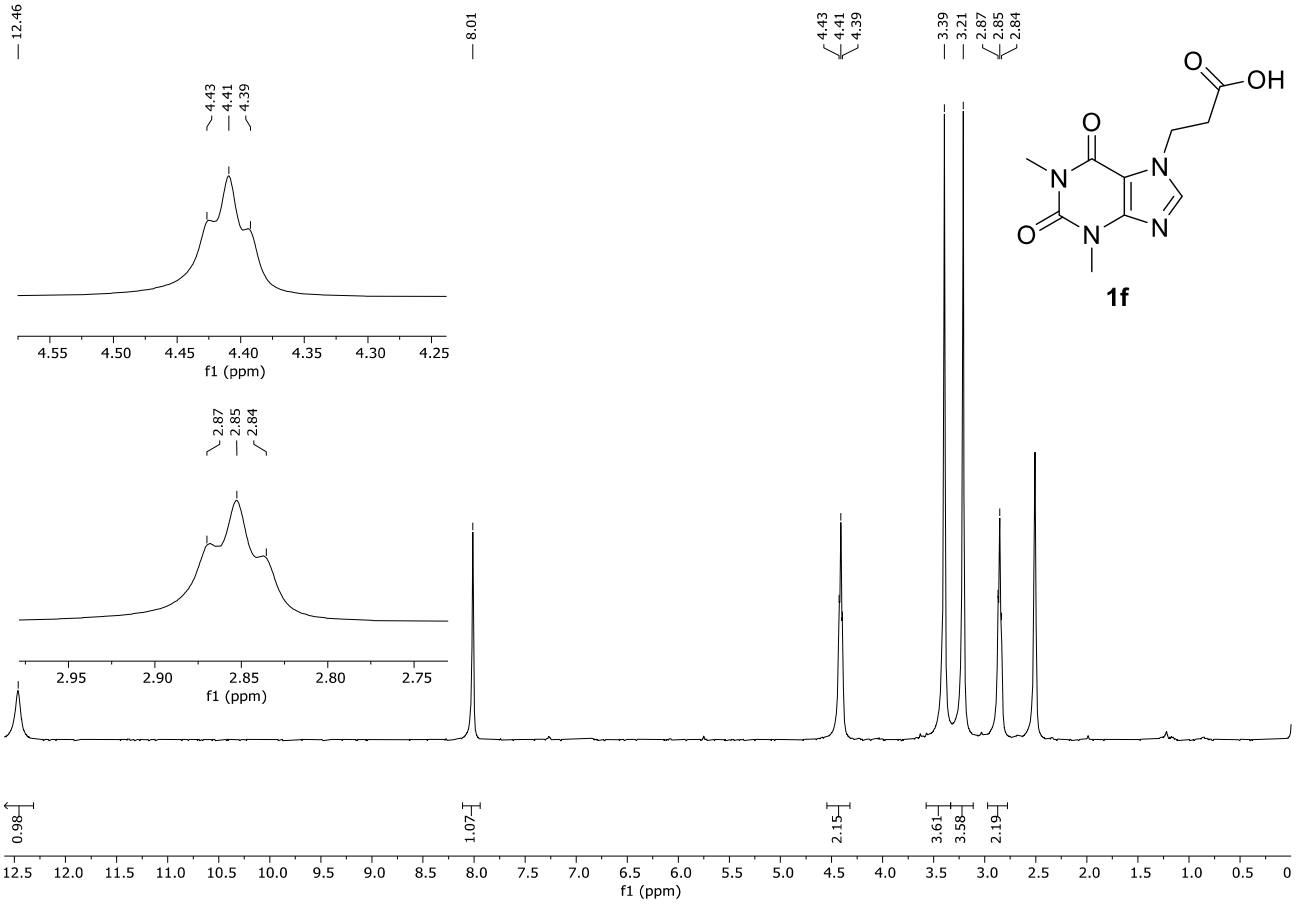

155.13
151.58
149.34
148.78
143.44
141.38
129.38
118.94
106.37
48.50
40.78
40.58
40.42
40.37
40.16
39.95
39.74
39.53
30.11
28.22
f1 (ppm)

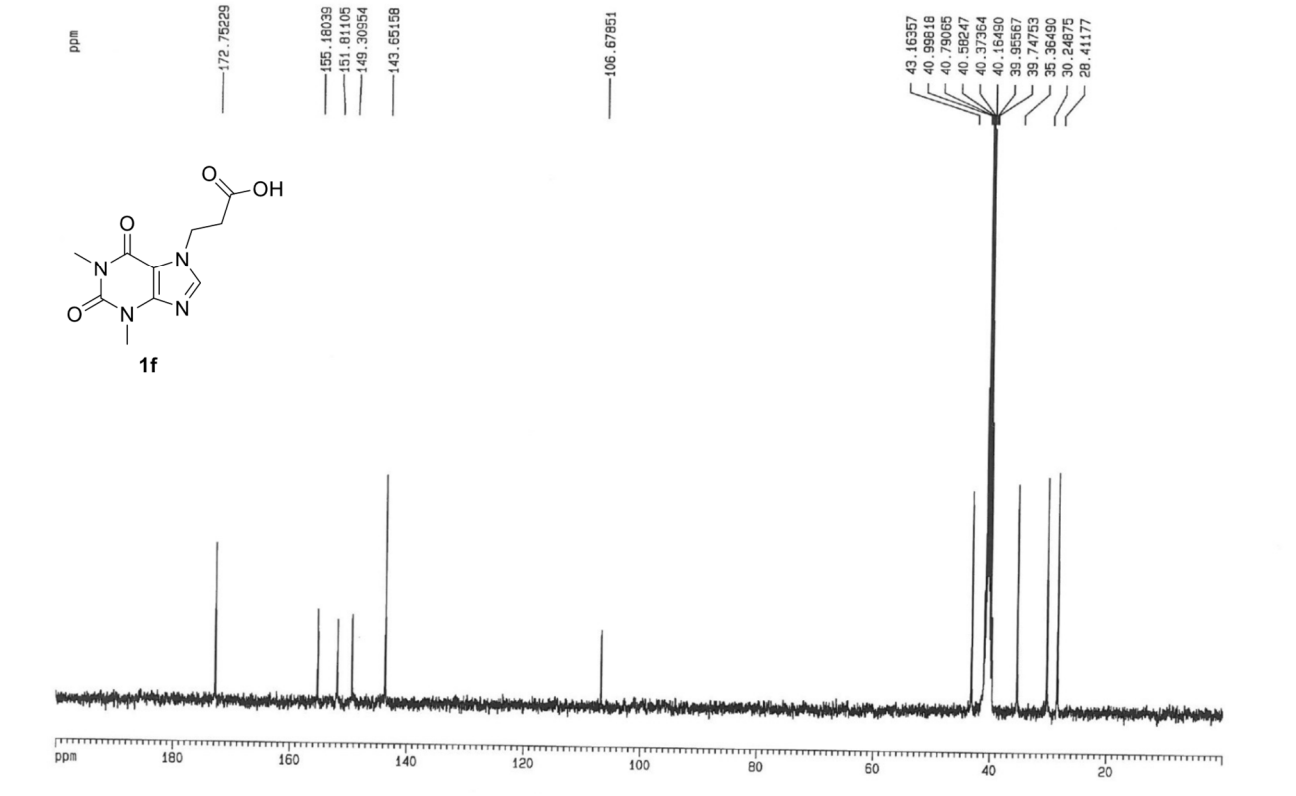
1d

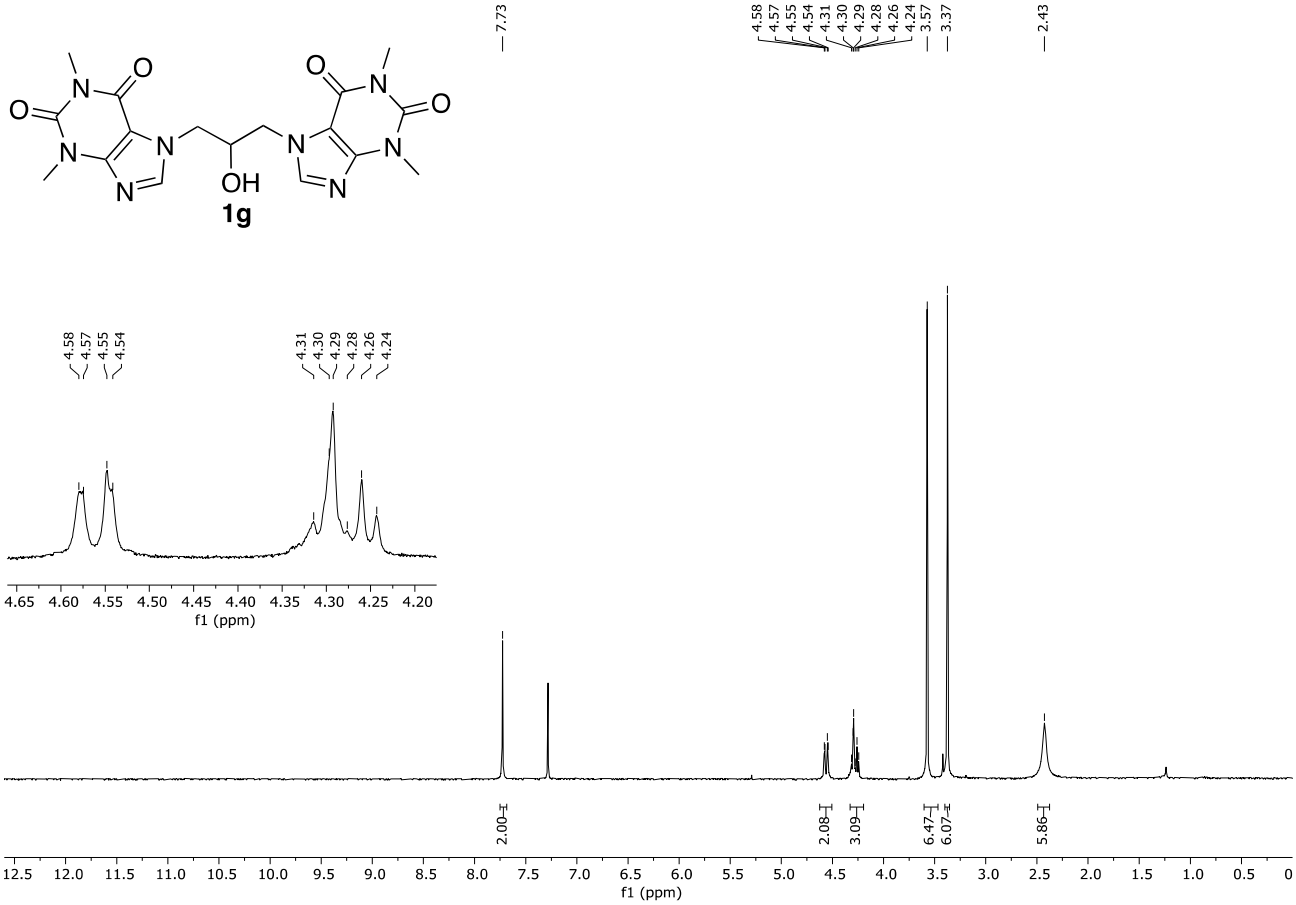

RT: 0.00 - 10.00 SM: 15G
RT: 4.00
Relative Abundance
Time (min)
Id | Formula | RDB | Delta ppm
1 | C14 H13 O6 N6 | 11.5 | -0.328
361.08899
C14 H13 O6 N6
362.09183
C13 13C H13 O6 N6
363.09395
C12 13C2 H13 O6 N6
Experimental spectrum
361.08911
C14 H13 O6 N6
362.09250
C13 13C H13 O6 N6
363.09444
C12 13C2 H13 O6 N6
Theoretical spectrum
m/z

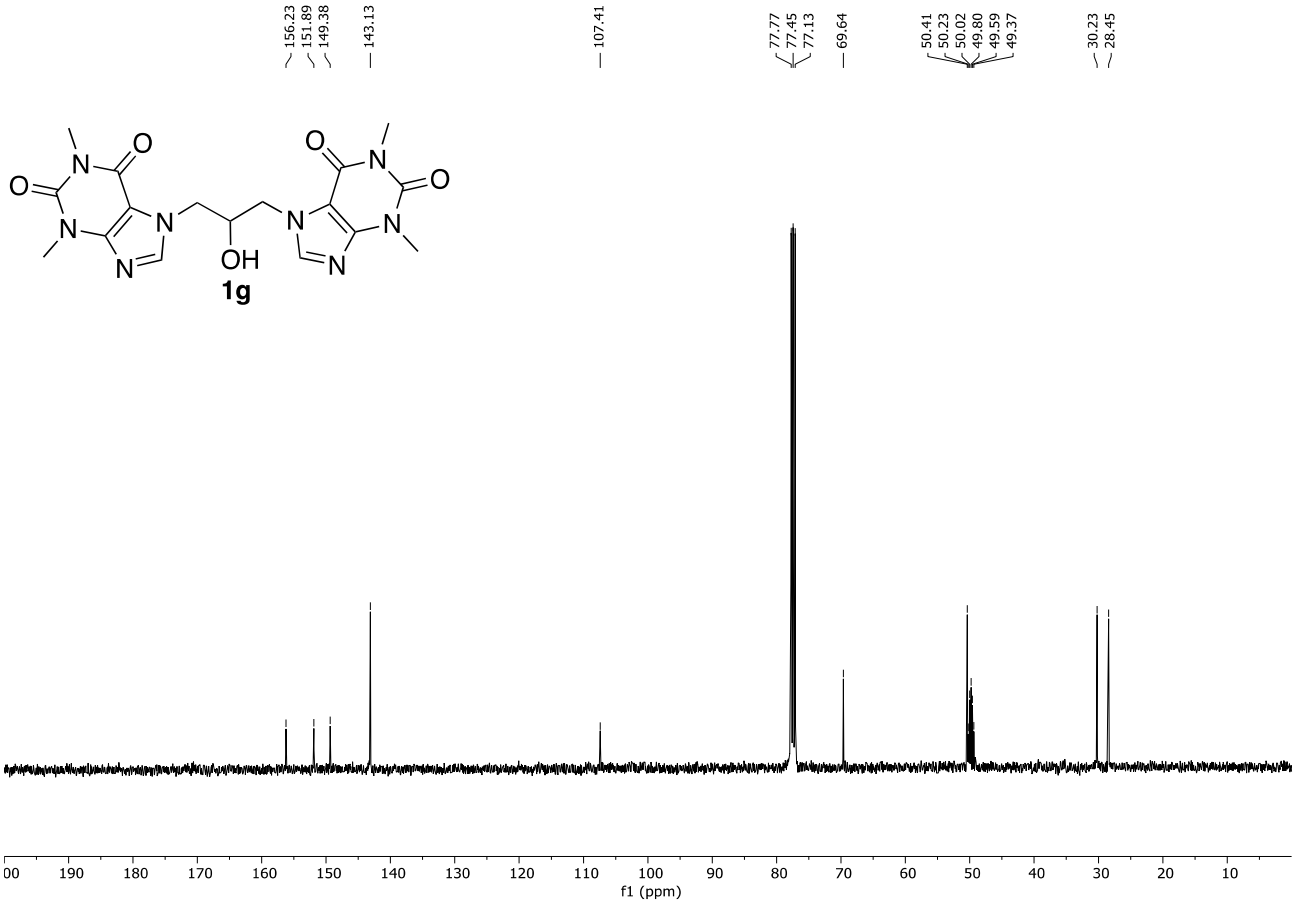

7.61
5.07
4.28
4.26
4.24
4.22
3.57
3.35
1.31
1.29
1.27
1.01
2.41
2.23
3.31
3.25
3.56
f1 (ppm)
1e

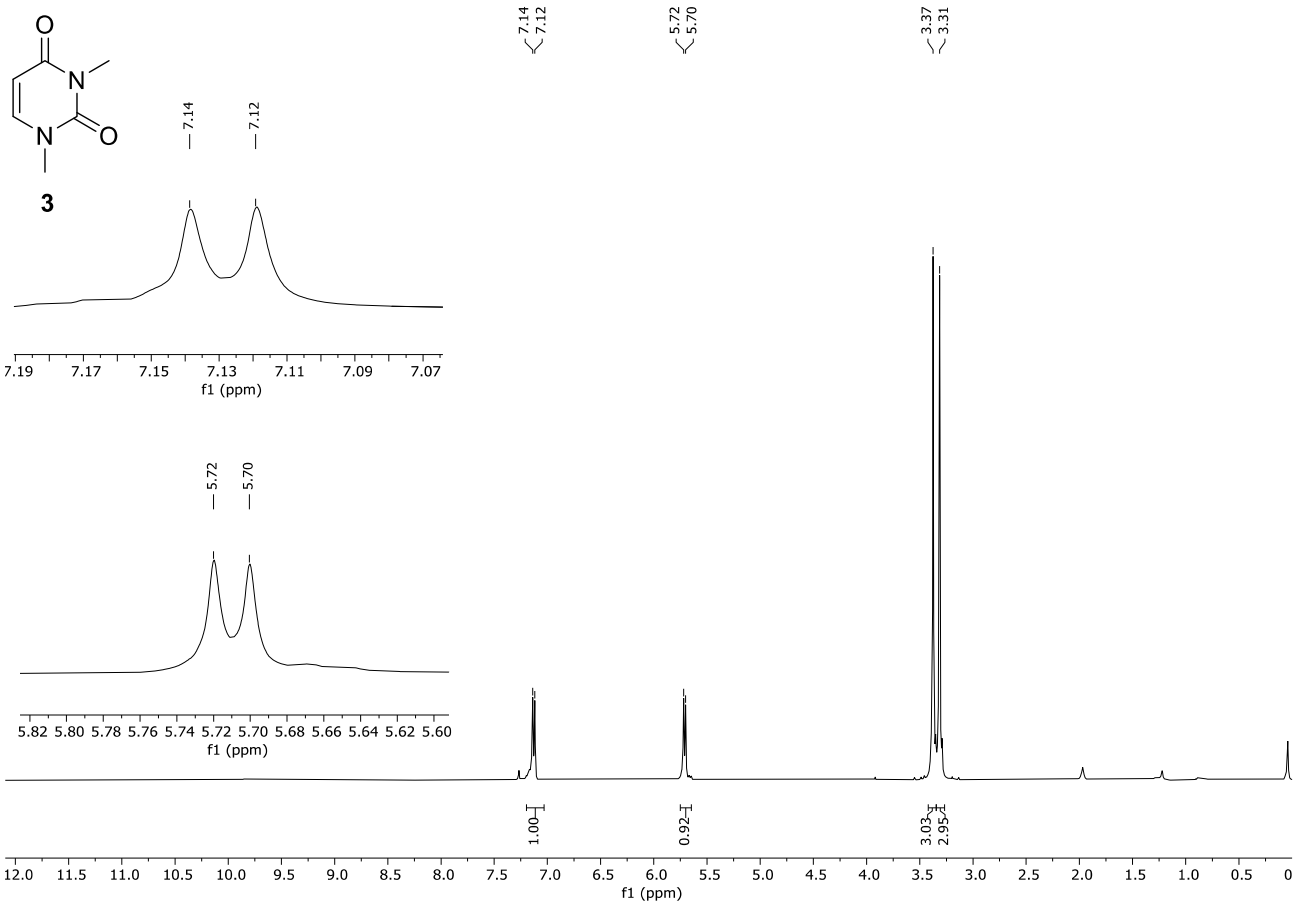

167.47
155.65
152.03
148.89
142.30
107.50
77.83
77.51
77.19
62.80
47.74
30.21
28.30
14.47
f1 (ppm)
1e

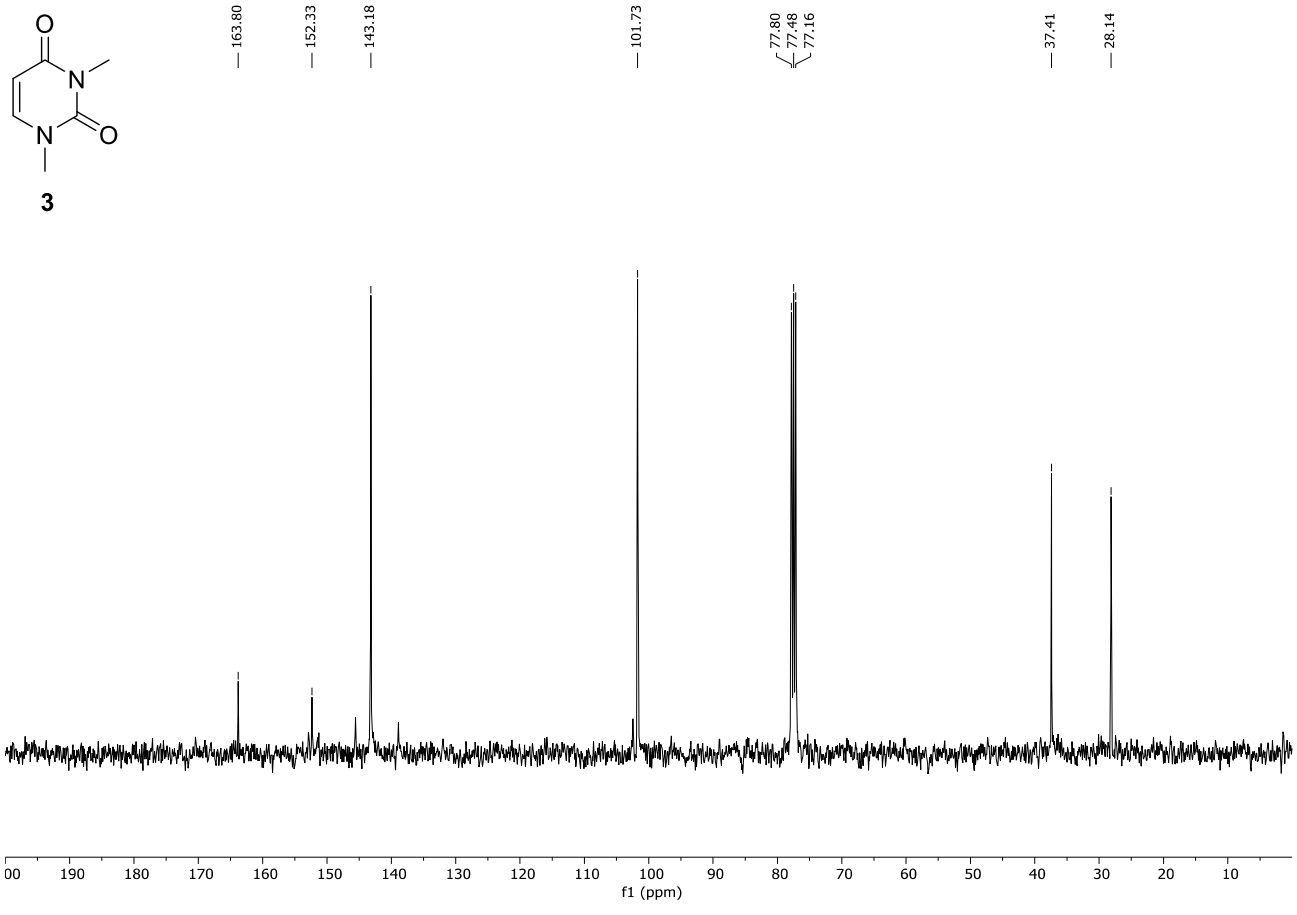

12.46
8.01
4.43
4.41
4.39
3.39
3.21
2.87
2.85
2.84
1f
f1 (ppm)
0.98
1.07
2.15
3.61
3.58
2.19

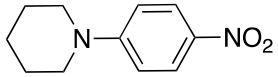

ppm
172.78229
155.18039
151.81105
149.30954
143.65158
106.67851
43.16357
40.99818
40.79065
40.58247
40.37364
40.16490
39.95567
39.74753
35.36490
30.24875
28.41177
1f
ppm


7.73
4.58
4.57
4.55
4.54
4.31
4.30
4.29
4.28
4.26
4.24
3.57
3.37
2.43
OH
1g
2.00
2.08
3.09
6.47
6.07
5.86
f1 (ppm)


156.23
151.89
149.38
143.13
107.41
77.77
77.45
77.13
69.64
50.41
50.23
50.02
49.80
49.59
49.37
30.23
28.45
OH
1g
f1 (ppm)

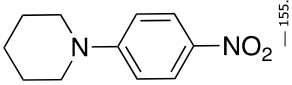

3
7.14
7.12
5.72
5.70
3.37
3.31
7.19 7.17 7.15 7.13 7.11 7.09 7.07
f1 (ppm)
5.82 5.80 5.78 5.76 5.74 5.72 5.70 5.68 5.66 5.64 5.62 5.60
f1 (ppm)
1.00
0.92
3.03
2.95
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
f1 (ppm)


3
163.80
152.33
143.18
101.73
77.80
77.48
77.16
37.41
28.14
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


N
NO2

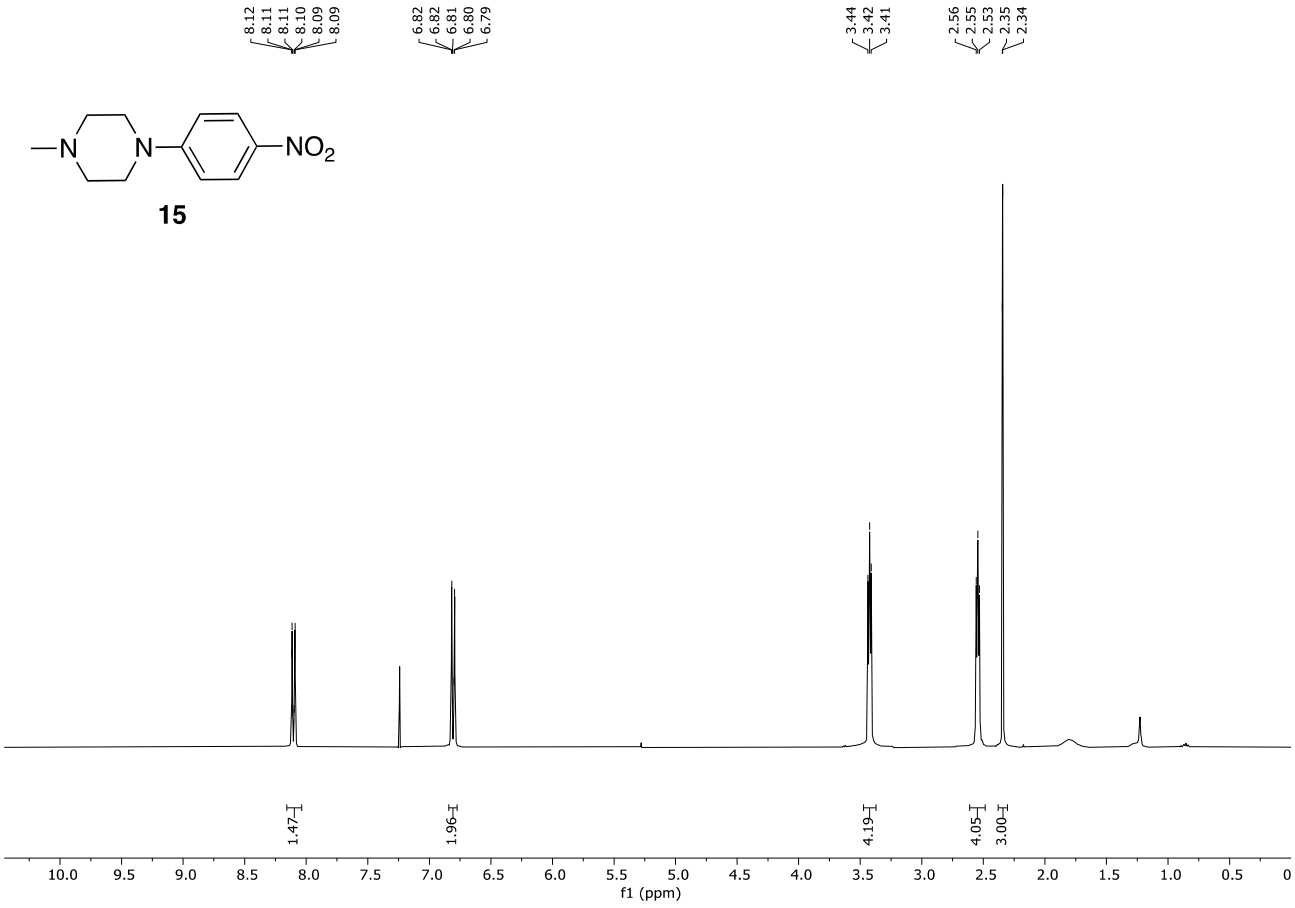
14

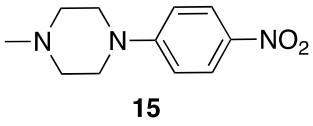
8.13
8.13
8.11
8.11
6.82
6.82
6.80
6.80
3.47
3.46
3.45
1.72
1.71
1.70
1.00
1.25
2.51
3.63
f1 (ppm)


N
NO2

14

155.27
137.81
126.58
112.72
77.77
77.45
77.13
48.80
25.68
24.65
f1 (ppm)

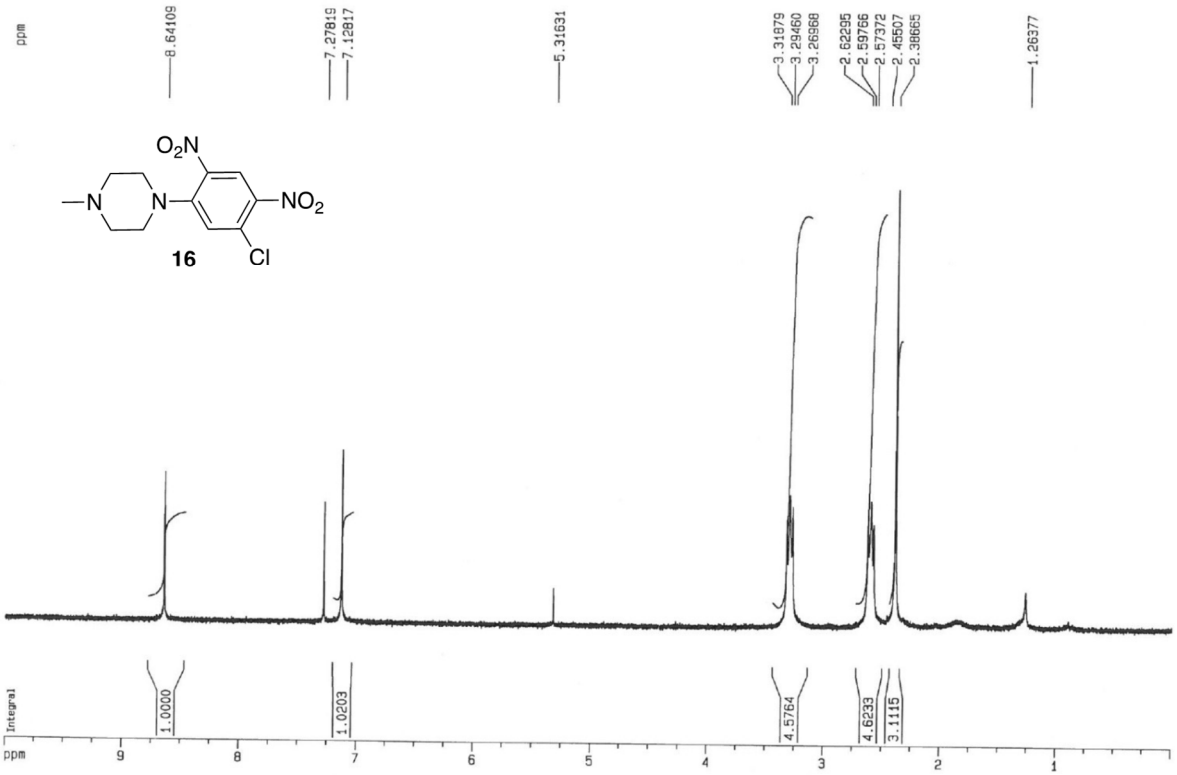

8.12
8.11
8.11
8.10
8.09
8.09
6.82
6.82
6.81
6.80
6.79
3.44
3.42
3.41
2.56
2.55
2.53
2.35
2.34
1.47
1.96
4.19
4.05
3.00
f1 (ppm)

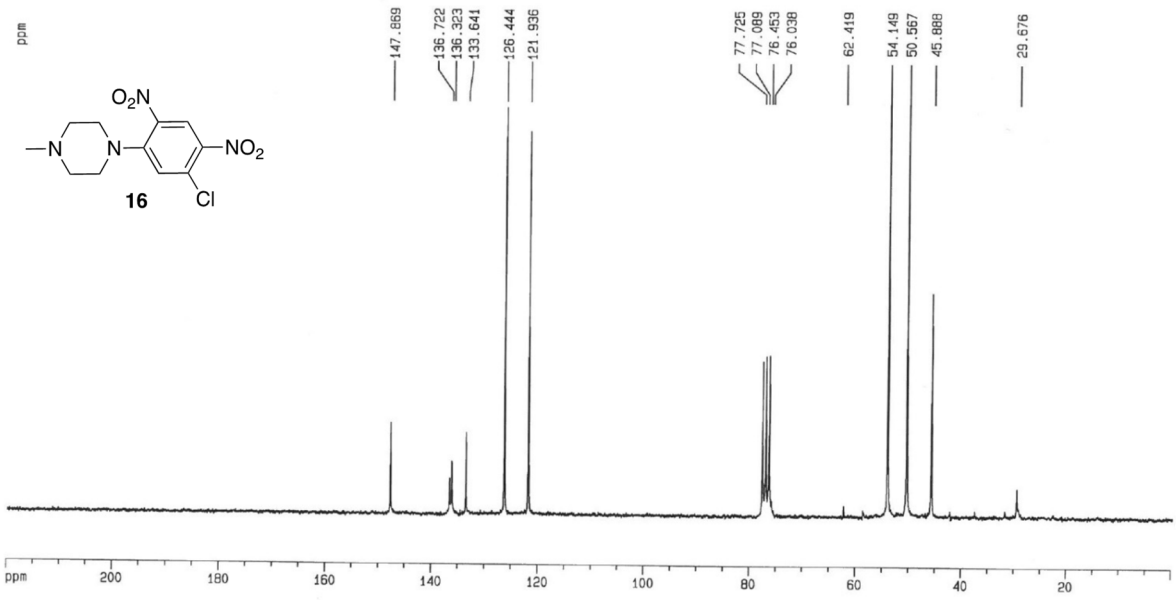


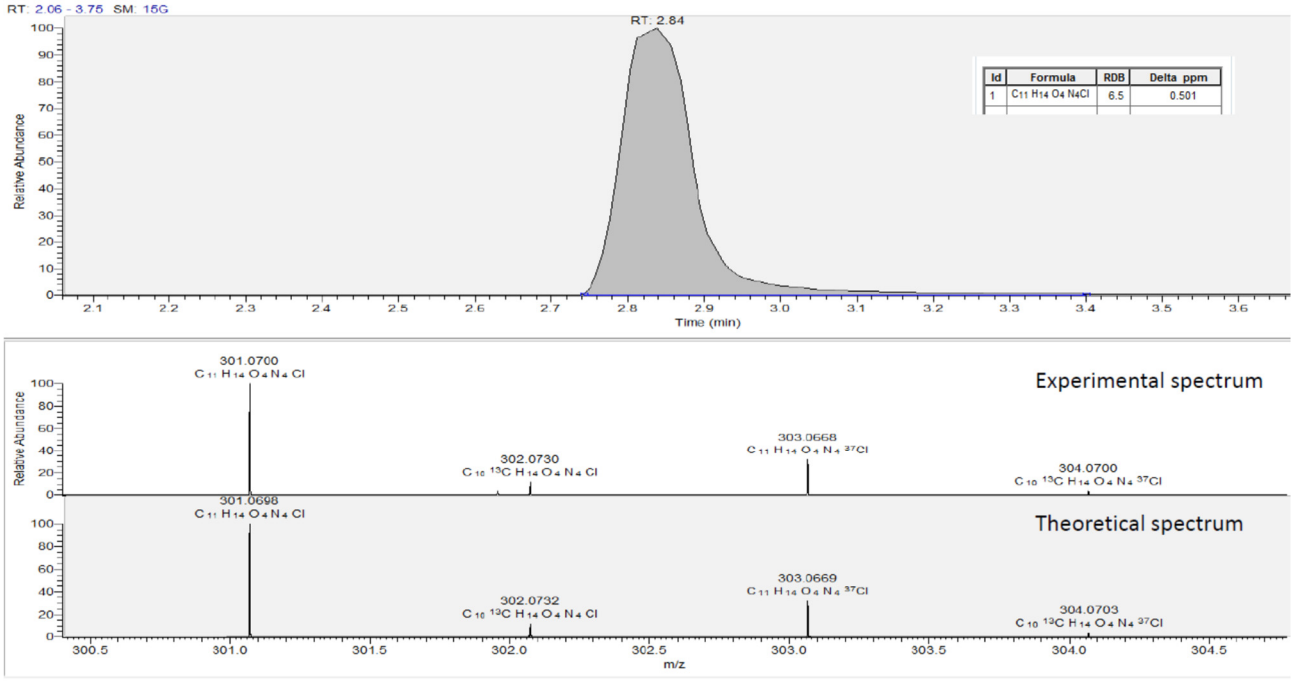
15

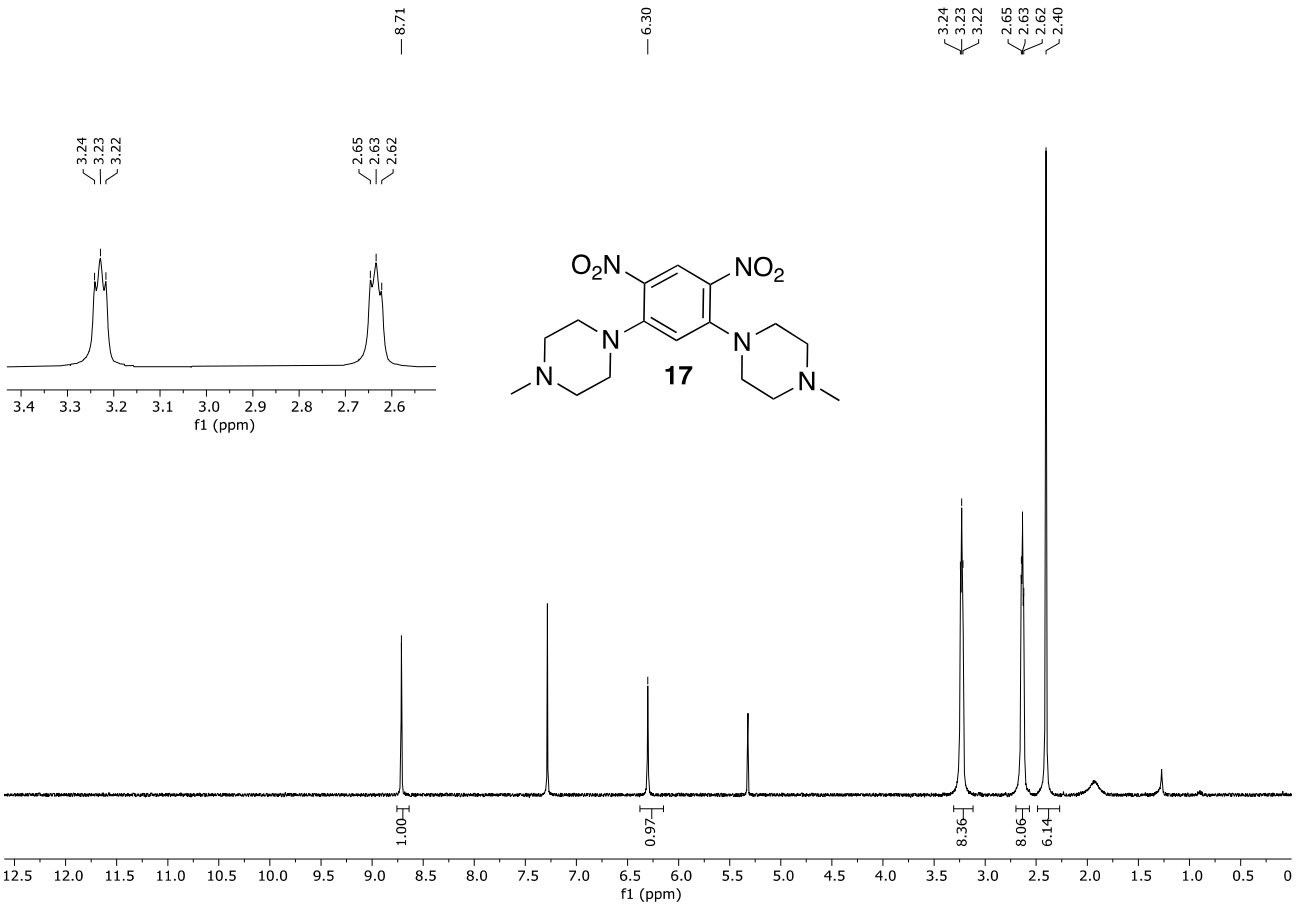
155.23
138.83
126.39
113.12
77.76
77.45
77.13
54.92
47.34
46.47
f1 (ppm)

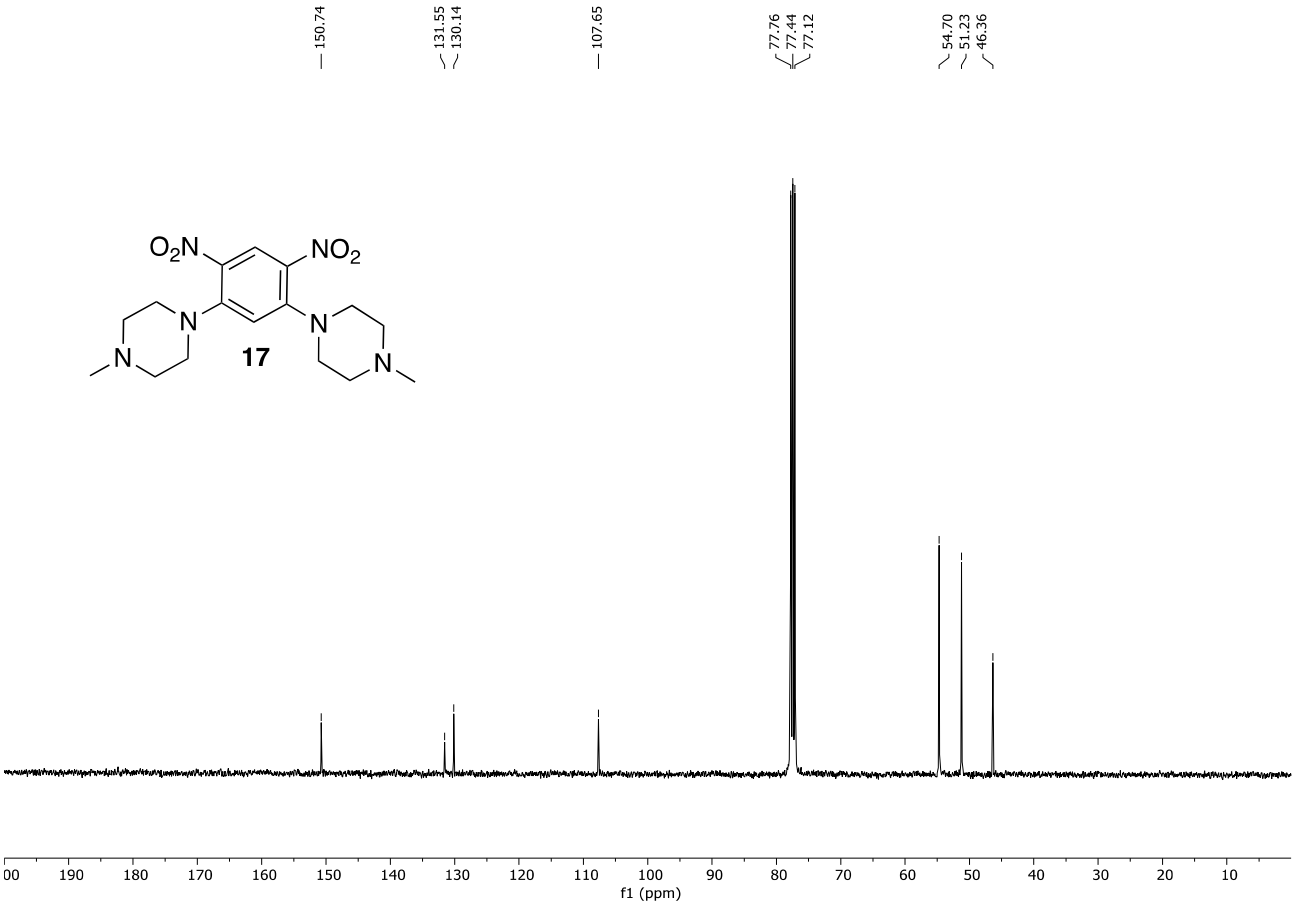
15

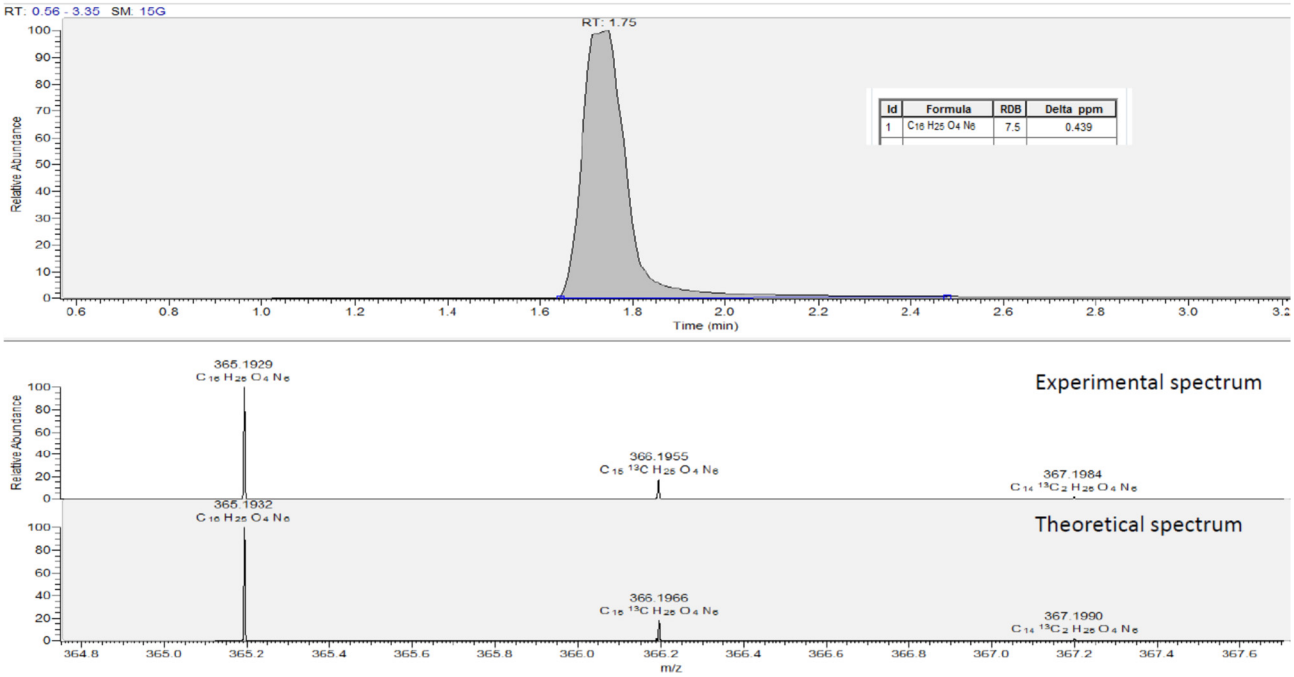

ppm
8.64109
7.27819
7.12817
5.31631
3.31879
3.29460
3.26968
2.62295
2.59766
2.57372
2.45507
2.38665
1.26377
O2N
NO2
N
N
Cl
16
Integral
1.0000
1.0203
4.5764
4.6233
3.1115
ppm
9
8
7
6
5
4
3
2
1

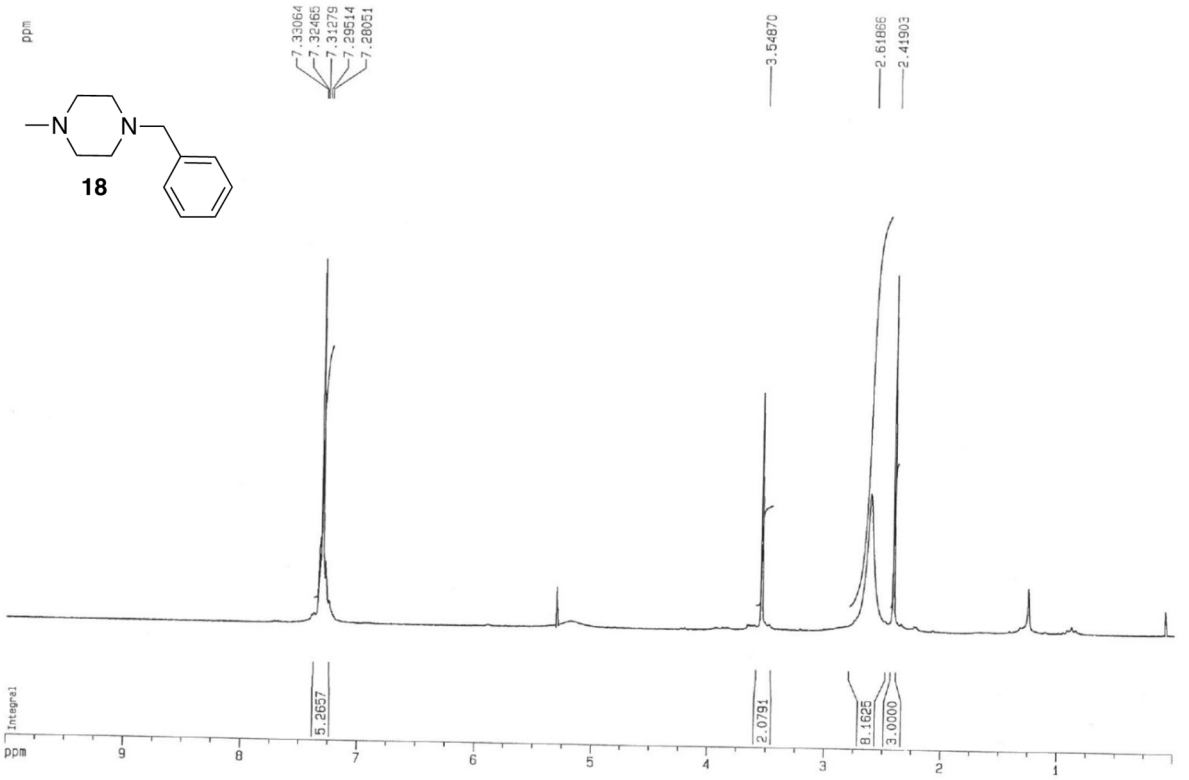

ppm
147.869
136.722
136.323
133.641
126.444
121.936
77.725
77.089
76.453
76.038
62.419
54.149
50.567
45.888
29.676
O2N
NO2
N
N
Cl
16
ppm
200
180
160
140
120
100
80
60
40
20


RT: 2.06 - 3.75 SM: 15G
RT: 2.84
Relative Abundance
Time (min)
Id | Formula | RDB | Delta ppm
1 | C11 H14 O4 N4Cl | 6.5 | 0.501
Experimental spectrum
301.0700
C11 H14 O4 N4 Cl
302.0730
C10 13C H14 O4 N4 Cl
303.0668
C11 H14 O4 N4 37Cl
304.0700
C10 13C H14 O4 N4 37Cl
Theoretical spectrum
301.0698
C11 H14 O4 N4 Cl
302.0732
C10 13C H14 O4 N4 Cl
303.0669
C11 H14 O4 N4 37Cl
304.0703
C10 13C H14 O4 N4 37Cl
m/z


8.71
6.30
3.24
3.23
3.22
2.65
2.63
2.62
2.40
O2N
NO2
N
N
N
N
17
1.00
0.97
8.36
8.06
6.14
f1 (ppm)


150.74
131.55
130.14
107.65
77.76
77.44
77.12
54.70
51.23
46.36
O2N
NO2
N
N
N
N
17
f1 (ppm)


RT: 0.56 - 3.35 SM: 15G
RT: 1.75
Relative Abundance
Time (min)
Id | Formula | RDB | Delta_ppm
1 | C16 H25 O4 N6 | 7.5 | 0.439
365.1929
C16 H25 O4 N6
366.1955
C15 13C H25 O4 N6
367.1984
C14 13C2 H25 O4 N6
Experimental spectrum
365.1932
C16 H25 O4 N6
366.1966
C15 13C H25 O4 N6
367.1990
C14 13C2 H25 O4 N6
Theoretical spectrum
m/z


ppm
7.33064
7.32465
7.31279
7.29514
7.28051
3.54870
2.61866
2.41903
18
Integral
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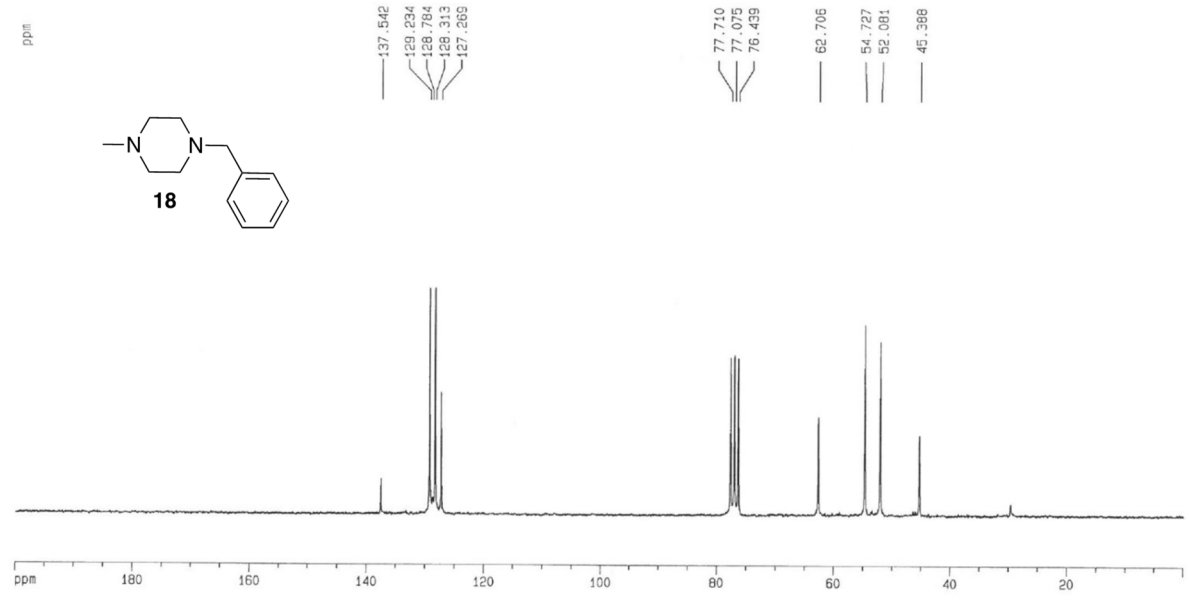
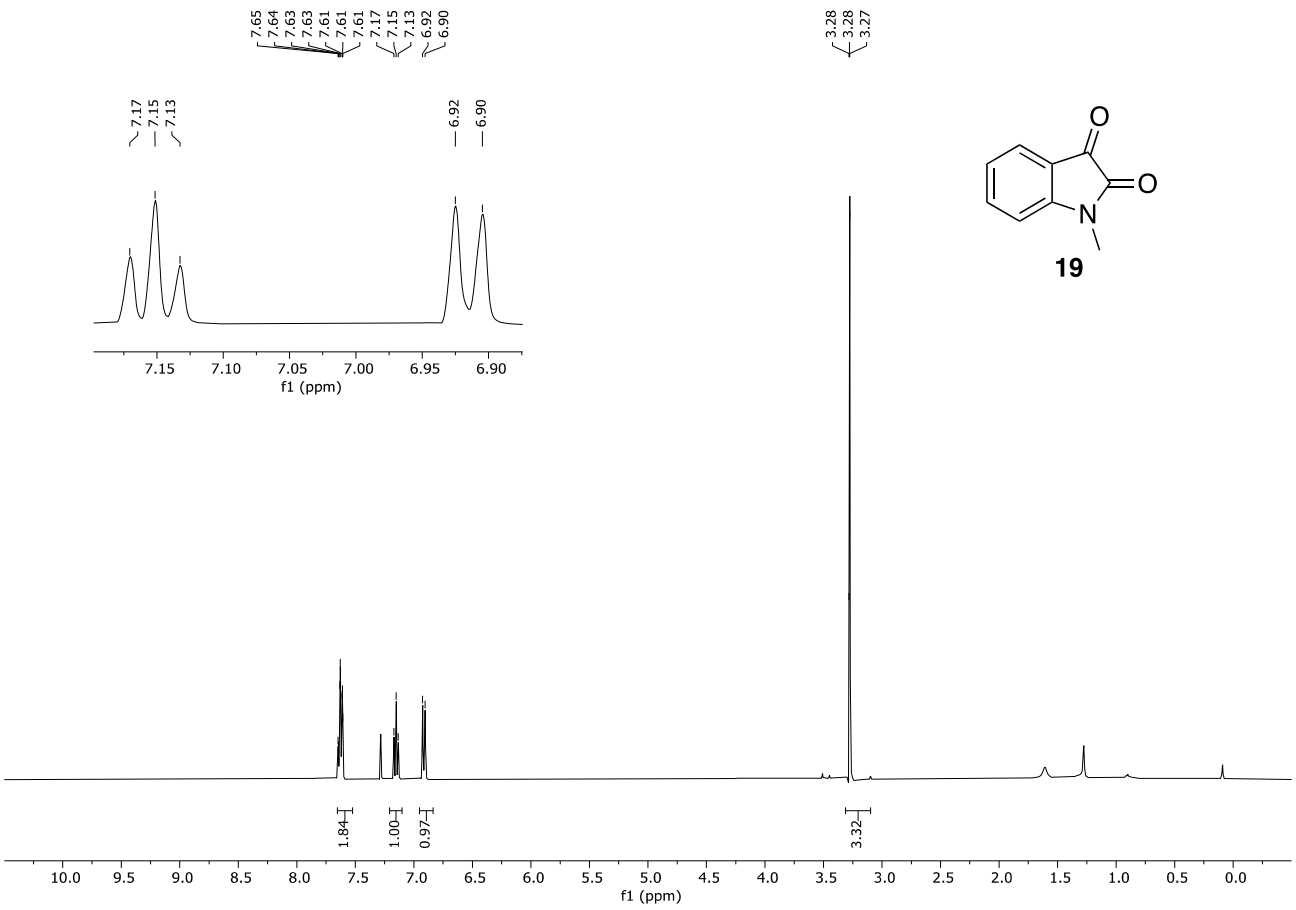
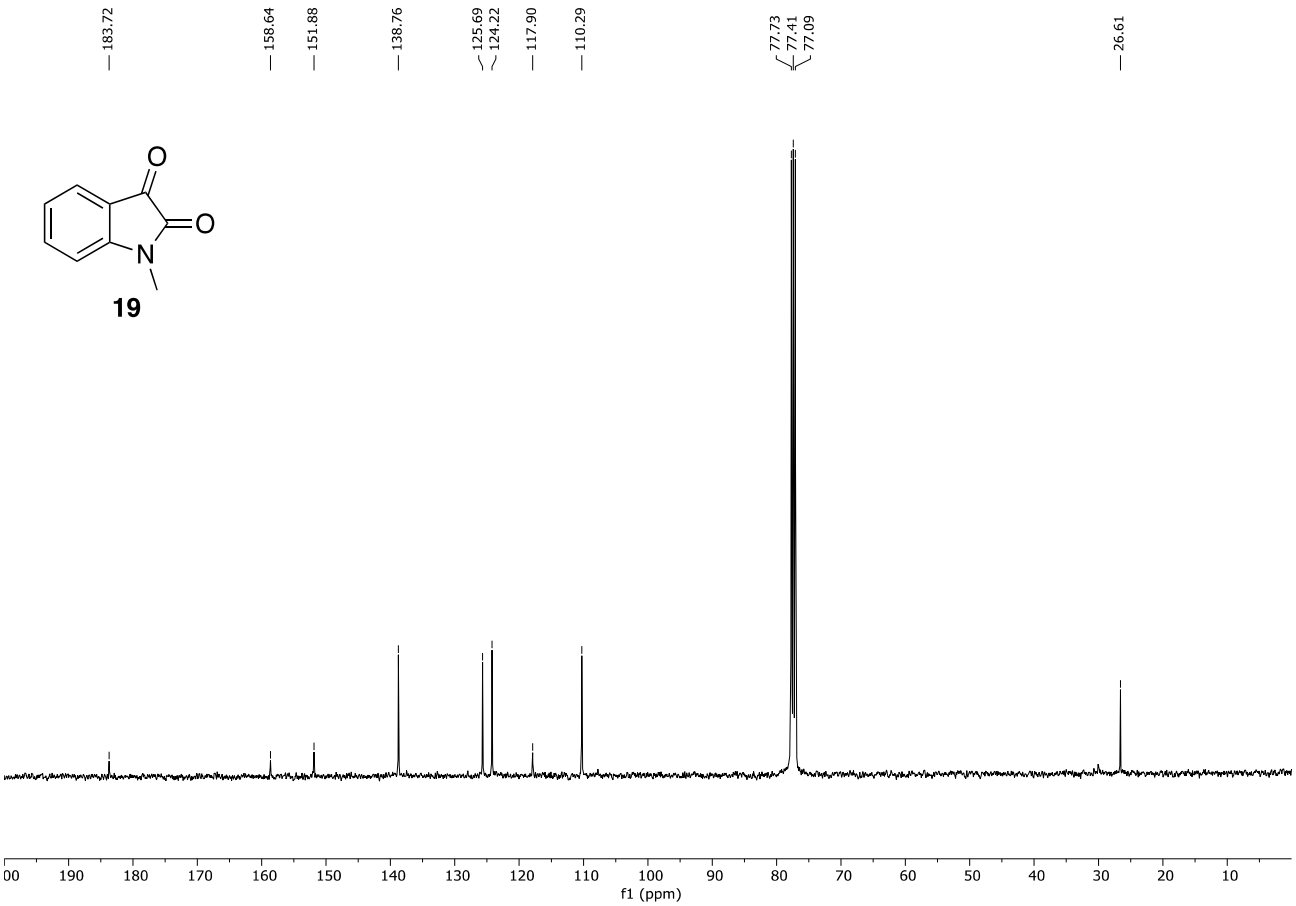
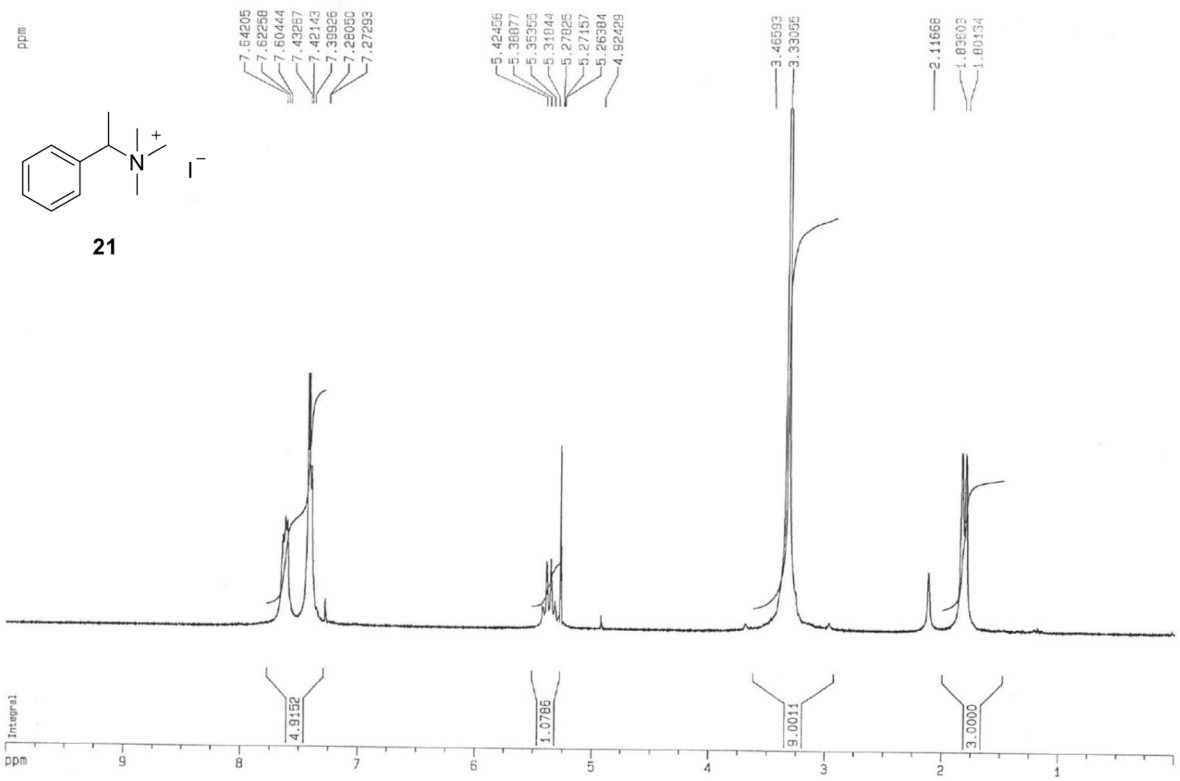
2.0791
8.1625
3.0000
ppm
9
8
7
6
5
4
3
2
1

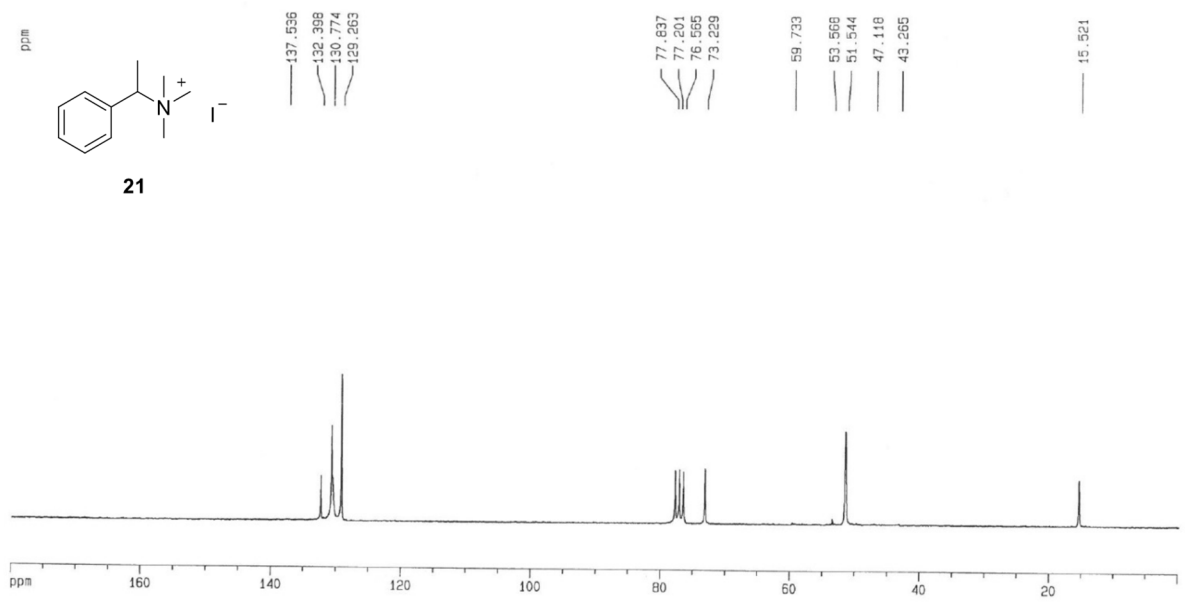

ppm
137.542
129.234
128.784
128.313
127.269
77.710
77.075
76.439
62.706
54.727
52.081
45.388
18
ppm
180
160
140
120
100
80
60
40
20


7.65
7.64
7.63
7.63
7.61
7.61
7.61
7.17
7.15
7.13
6.92
6.90
3.28
3.28
3.27
7.17
7.15
7.13
6.92
6.90
7.15
7.10
7.05
7.00
6.95
6.90
f1 (ppm)
O
O
N
19
1.84
1.00
0.97
3.32
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)


183.72
158.64
151.88
138.76
125.69
124.22
117.90
110.29
77.73
77.41
77.09
26.61
O
O
N
19
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


ppm
7.64205
7.62258
7.60444
7.43267
7.42143
7.39926
7.28060
7.27293
5.42456
5.38677
5.35355
5.31844
5.27825
5.27157
5.26384
4.92429
3.46593
3.33056
2.11668
1.83603
1.80134
Integral
4.9152
1.0786
9.0011
3.0000
ppm
9
8
7
6
5
4
3
2
1

21


ppm
137.536
132.398
130.774
129.263
77.837
77.201
76.565
73.229
59.733
53.568
51.544
47.118
43.265
15.521
ppm
160
140
120
100
80
60
40
20

21