Electronic Supplementary Information

Metal-free oxidative ring contraction of benzodiazepinones: an entry to quinoxalinones

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[c] Normandie Univ, France; UNIROUEN, Laboratoire SMS EA3233, 1 rue Tesniere, France, F-76821 Mont Saint Aignan (France)

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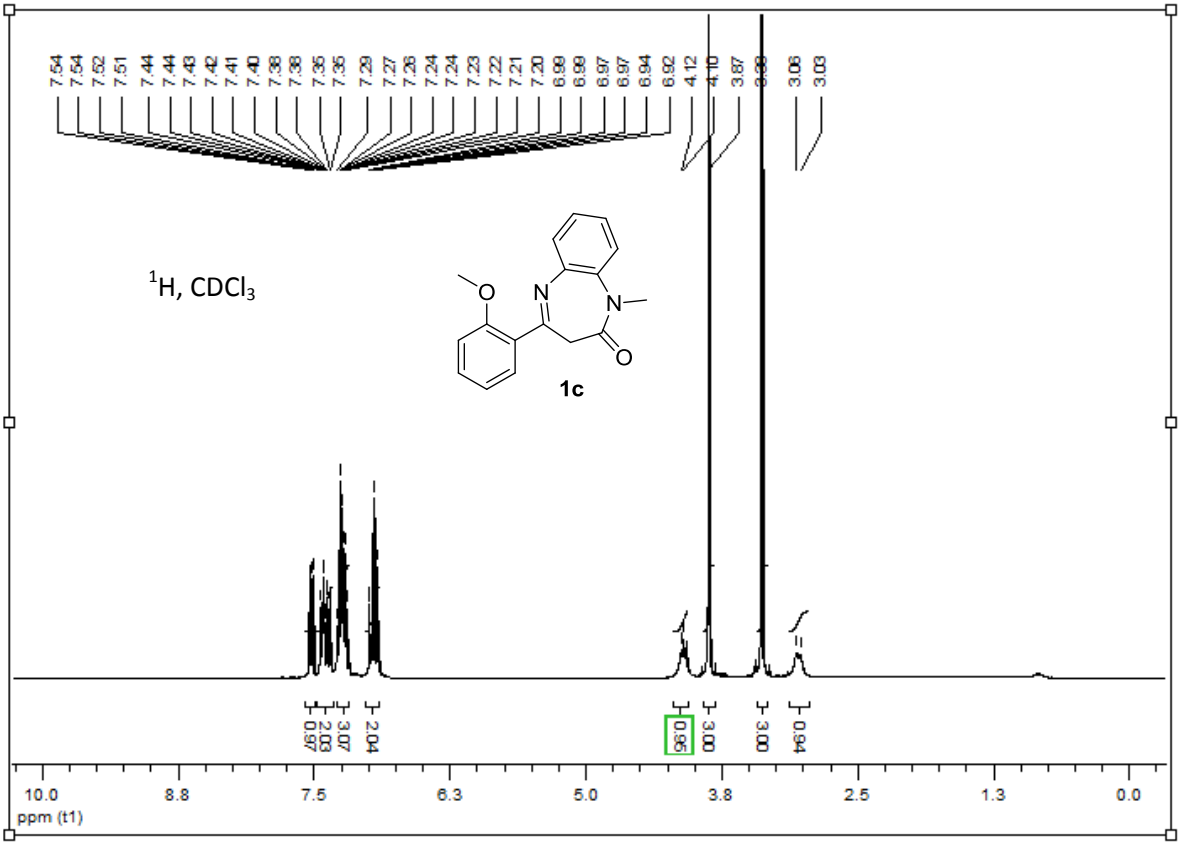
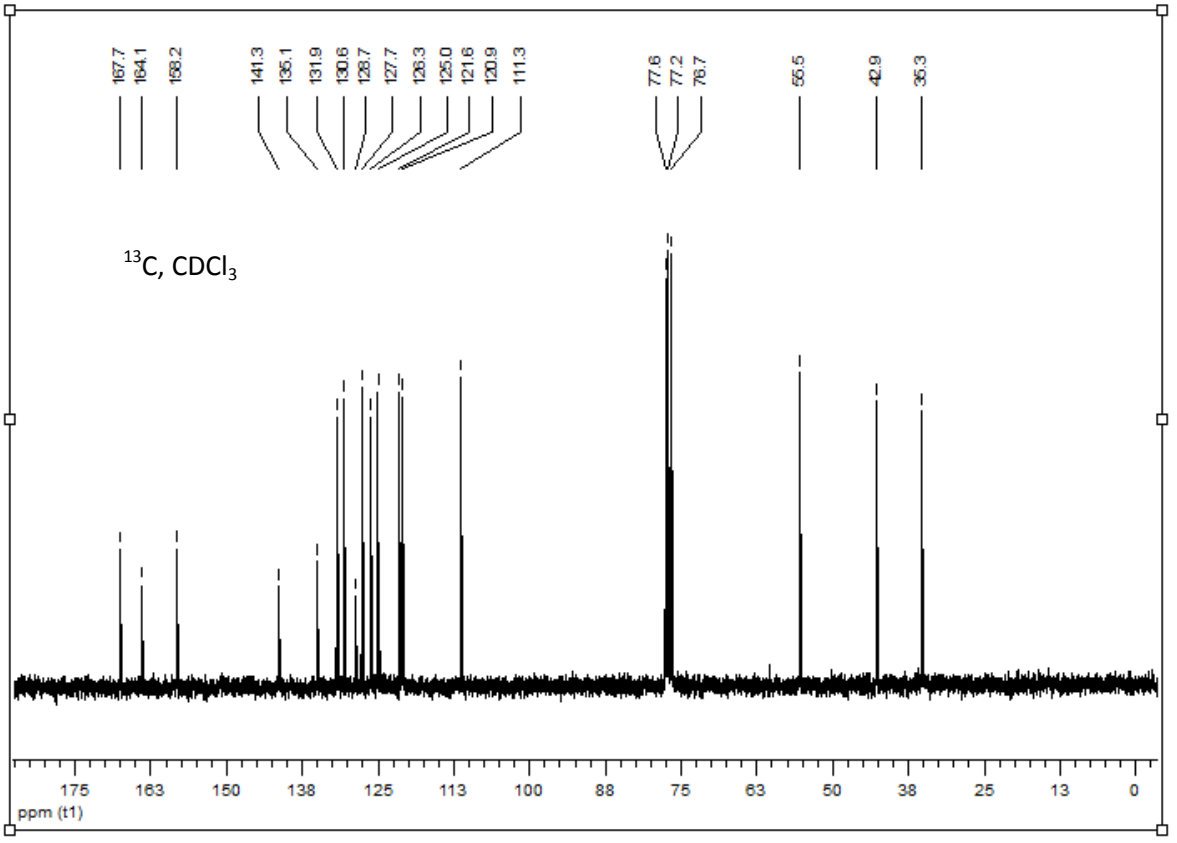
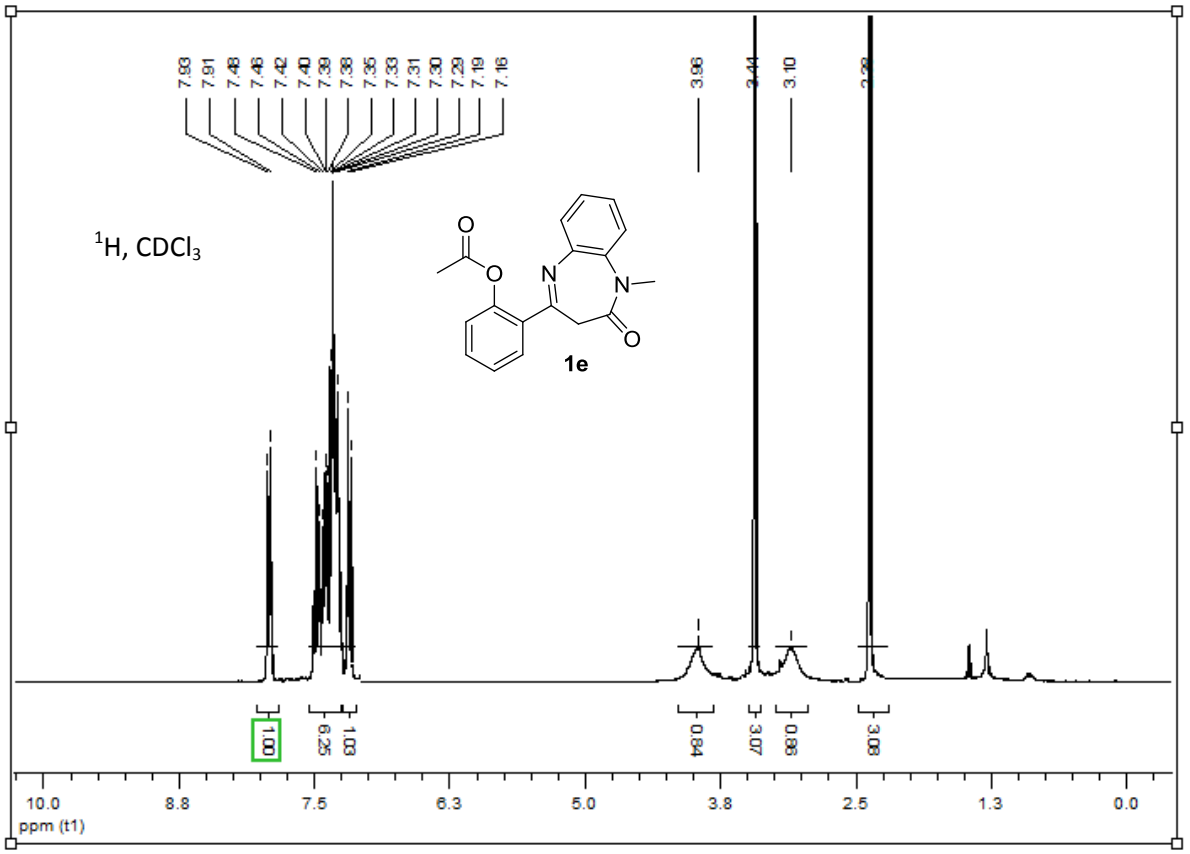
I. Copies of ^{1}H and ^{13}C NMR for compounds 1, 2, 3, 5, 6 and 8



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7.19
7.16
3.96
3.44
3.10
2.32
^{1}H, CDCl$_3$
O
O
N
N
O
1e
1.00
6.25
1.03
0.84
3.07
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8.8
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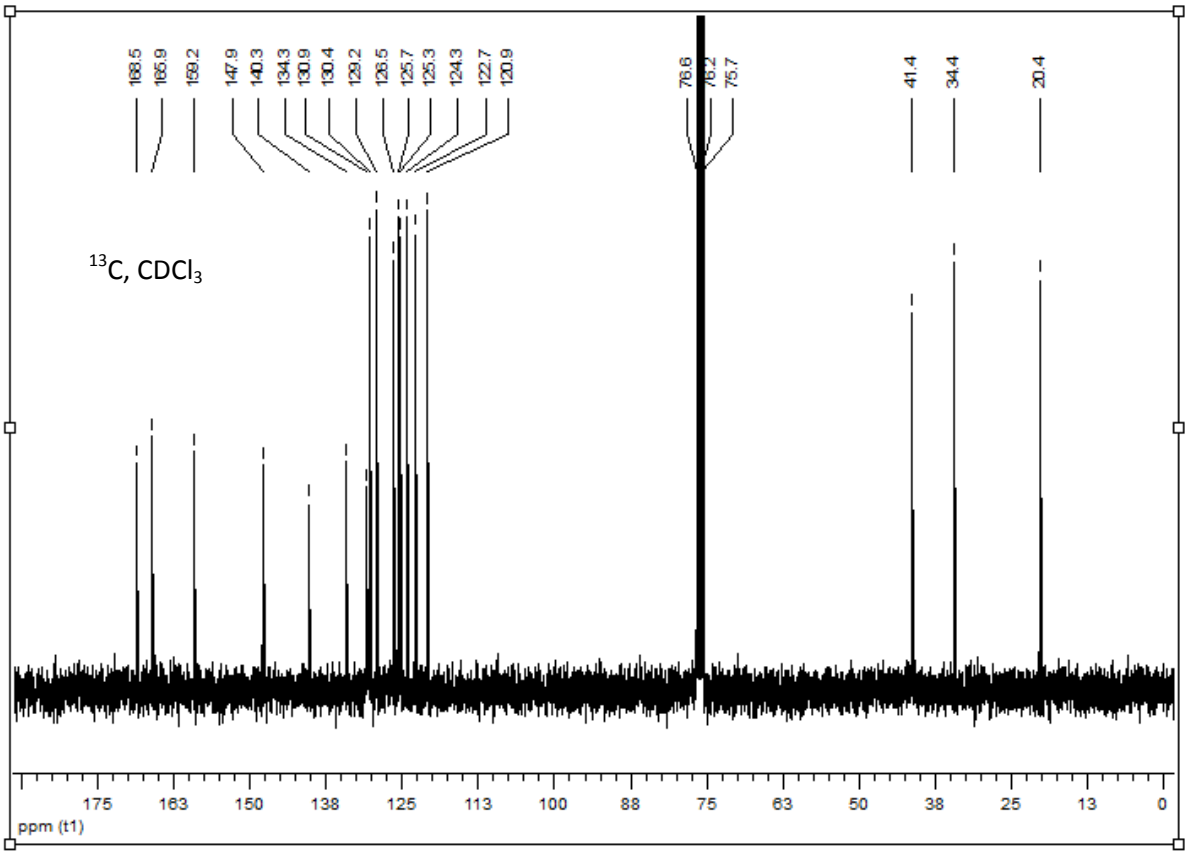
1.3
0.0
ppm (t1)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124.3
122.7
120.9
76.6
76.2
75.7
41.4
34.4
20.4
^{13}C, CDCl$_3$
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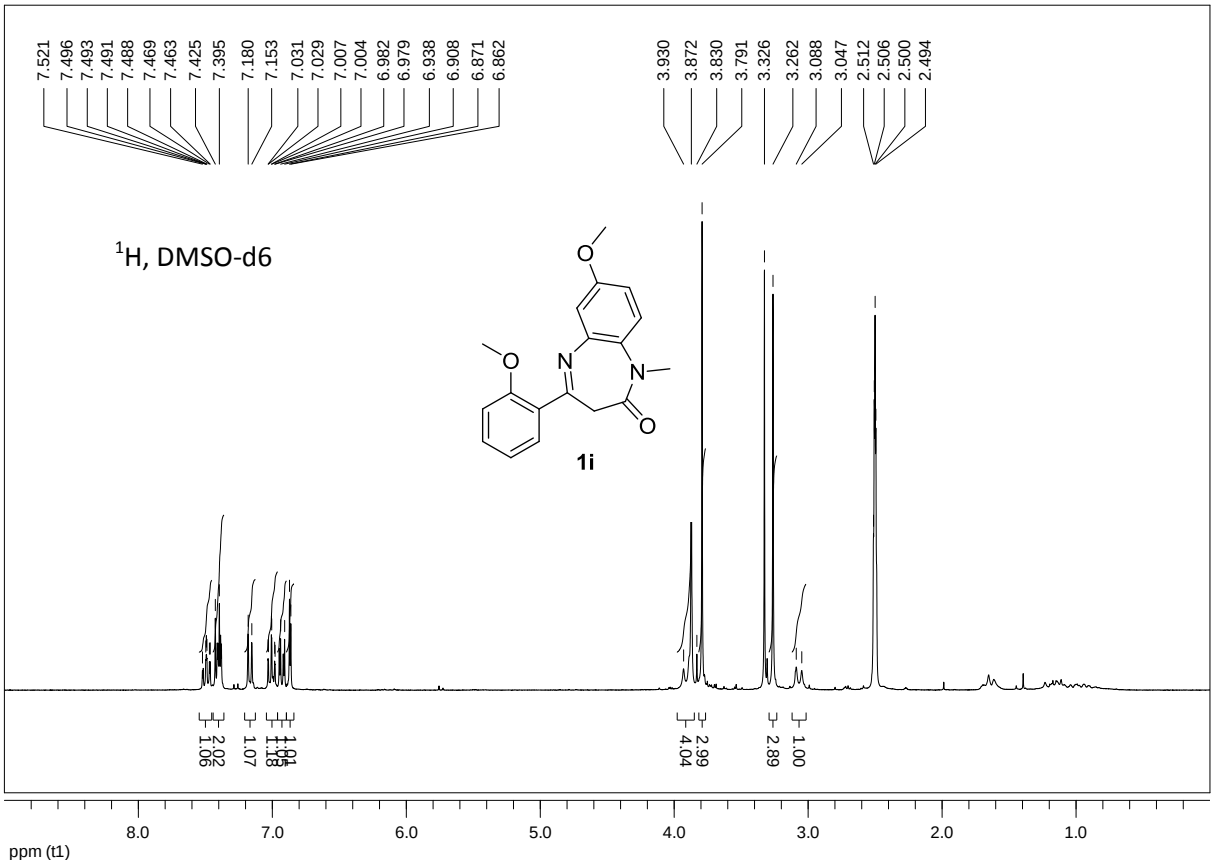
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)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7.029
7.007
7.004
6.982
6.979
6.938
6.908
6.871
6.862
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3.872
3.830
3.791
3.326
3.262
3.088
3.047
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2.500
2.494
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O
O
N
N
O
1i
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2.02
1.07
1.18
1.11
1.01
4.04
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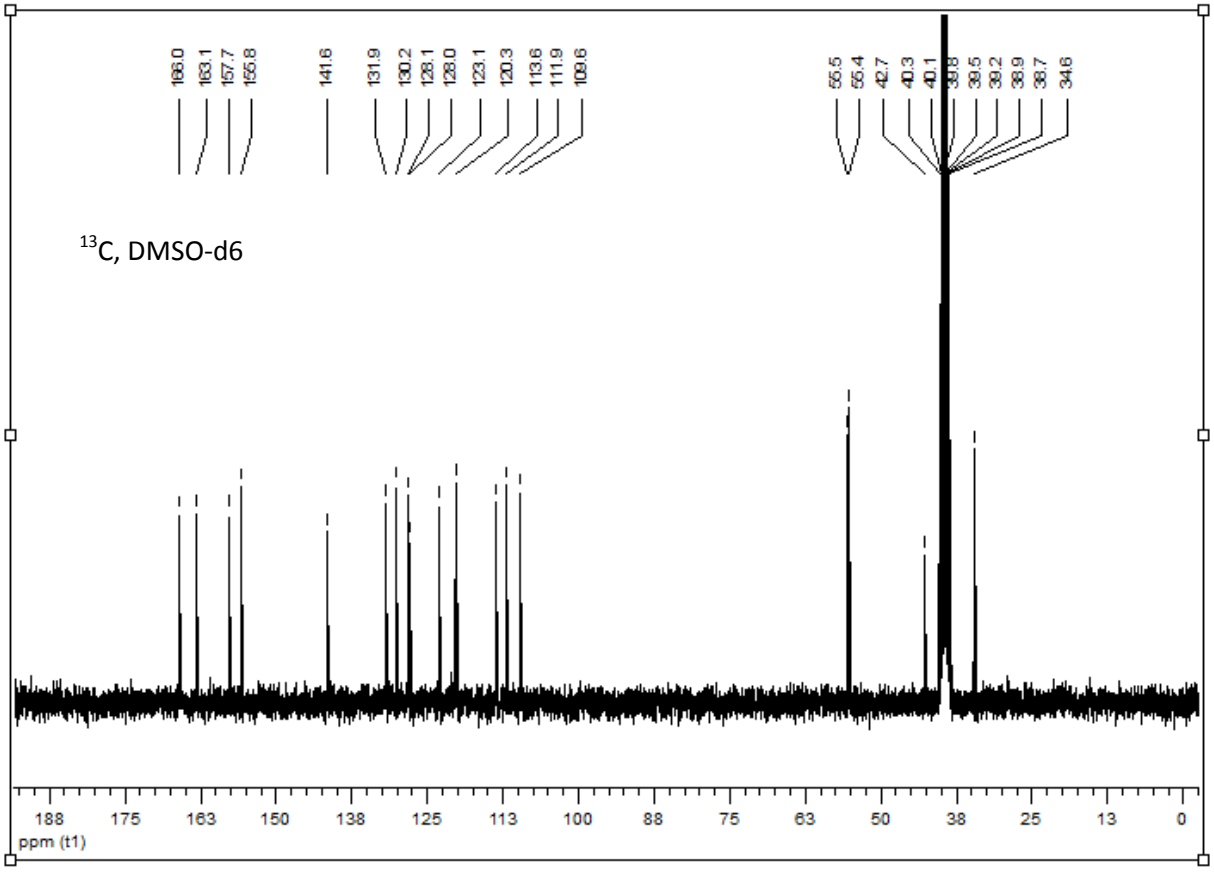
ppm (t1)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120.3
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111.9
109.6
55.5
55.4
42.7
40.3
40.1
39.8
39.5
39.2
38.9
38.7
34.6
13C, DMSO-d6
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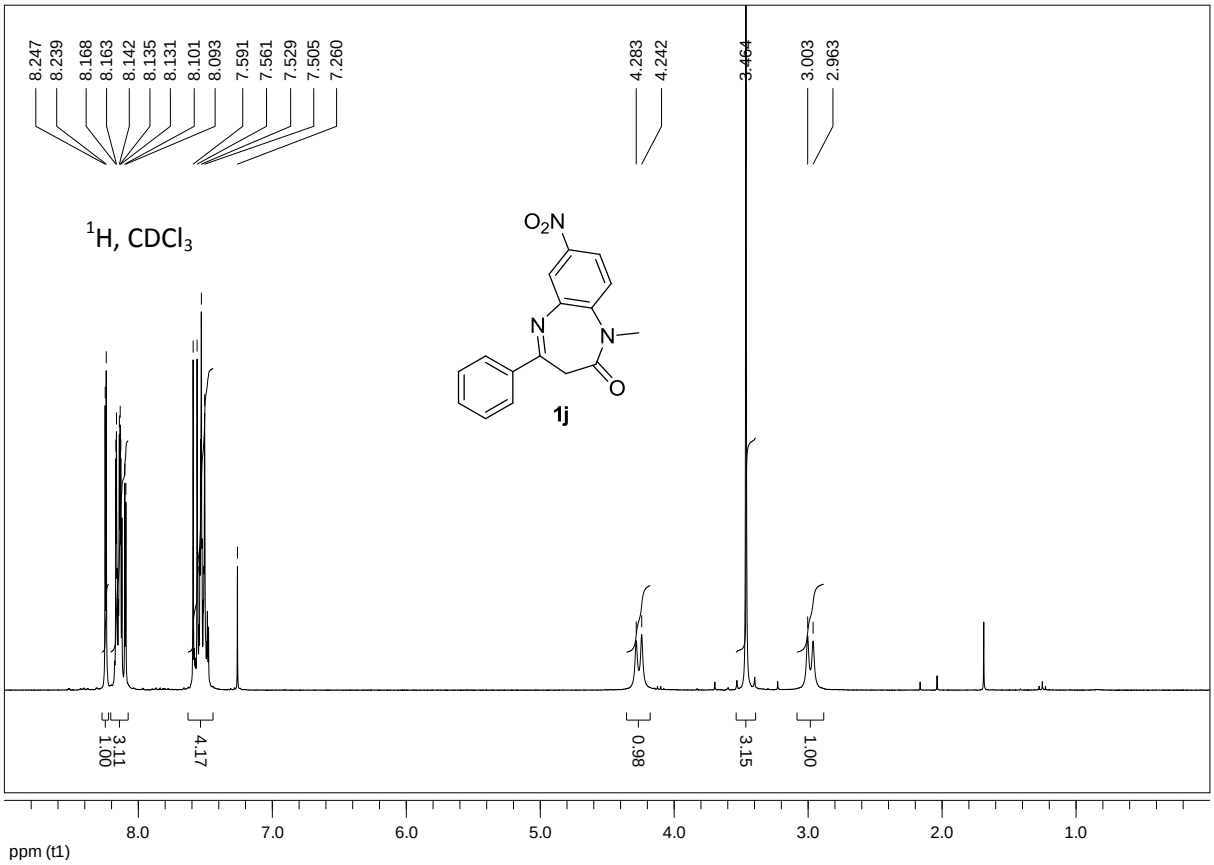
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)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7.260
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4.242
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3.003
2.963
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N
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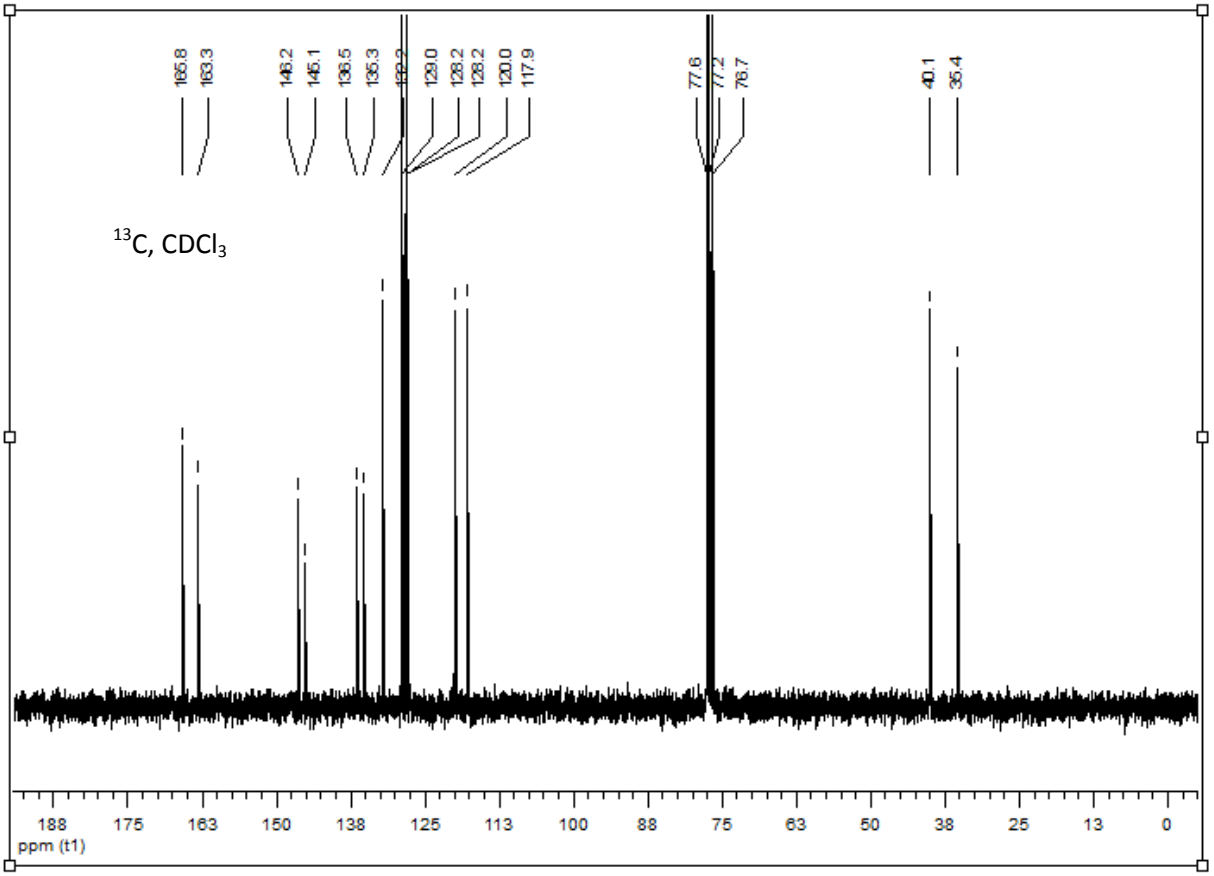
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77.6
77.2
76.7
40.1
35.4
^{13}C, CDCl$_3$
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175
163
150
138
125
113
100
88
75
63
50
38
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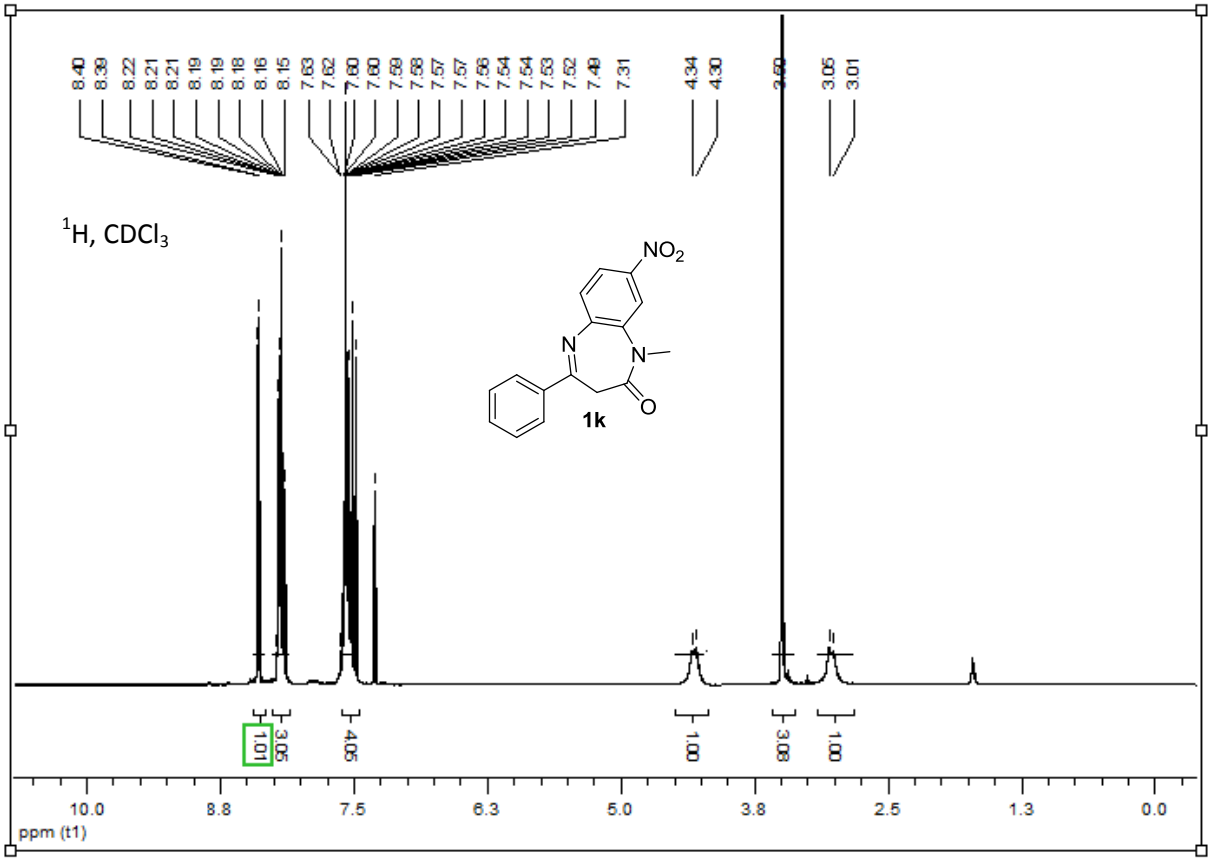
13
0
ppm (t1)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7.59
7.58
7.57
7.57
7.56
7.54
7.54
7.53
7.52
7.49
7.31
4.34
4.30
3.60
3.05
3.01
^{1}H, CDCl$_3$
NO$_2$
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N
O
1k
1.01
3.05
4.05
1.00
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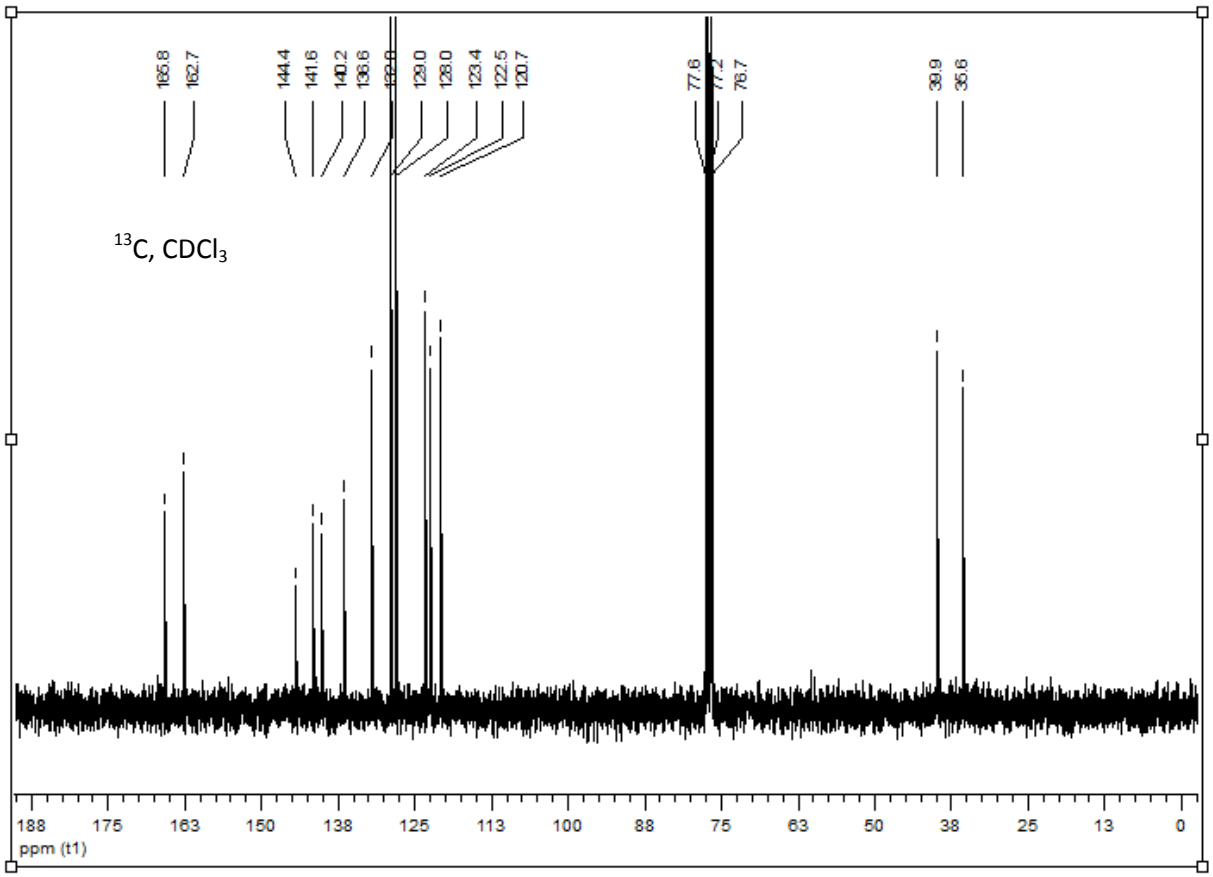
ppm (t1)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77.2
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39.9
35.6
^{13}C, CDCl$_3$
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163
150
138
125
113
100
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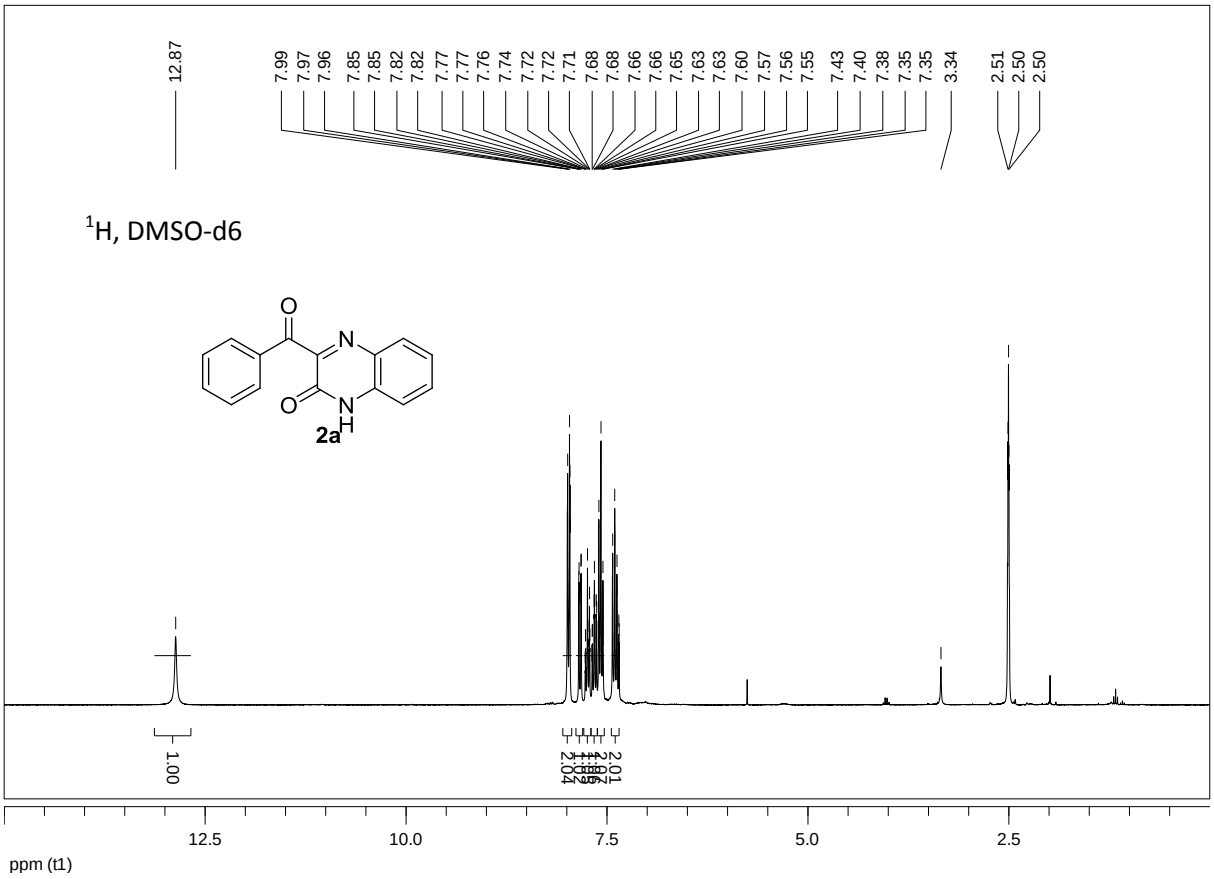
0
ppm (t1)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7.63
7.63
7.60
7.57
7.56
7.55
7.43
7.40
7.38
7.35
7.35
3.34
2.51
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2.50
1H, DMSO-d6
O
N
O
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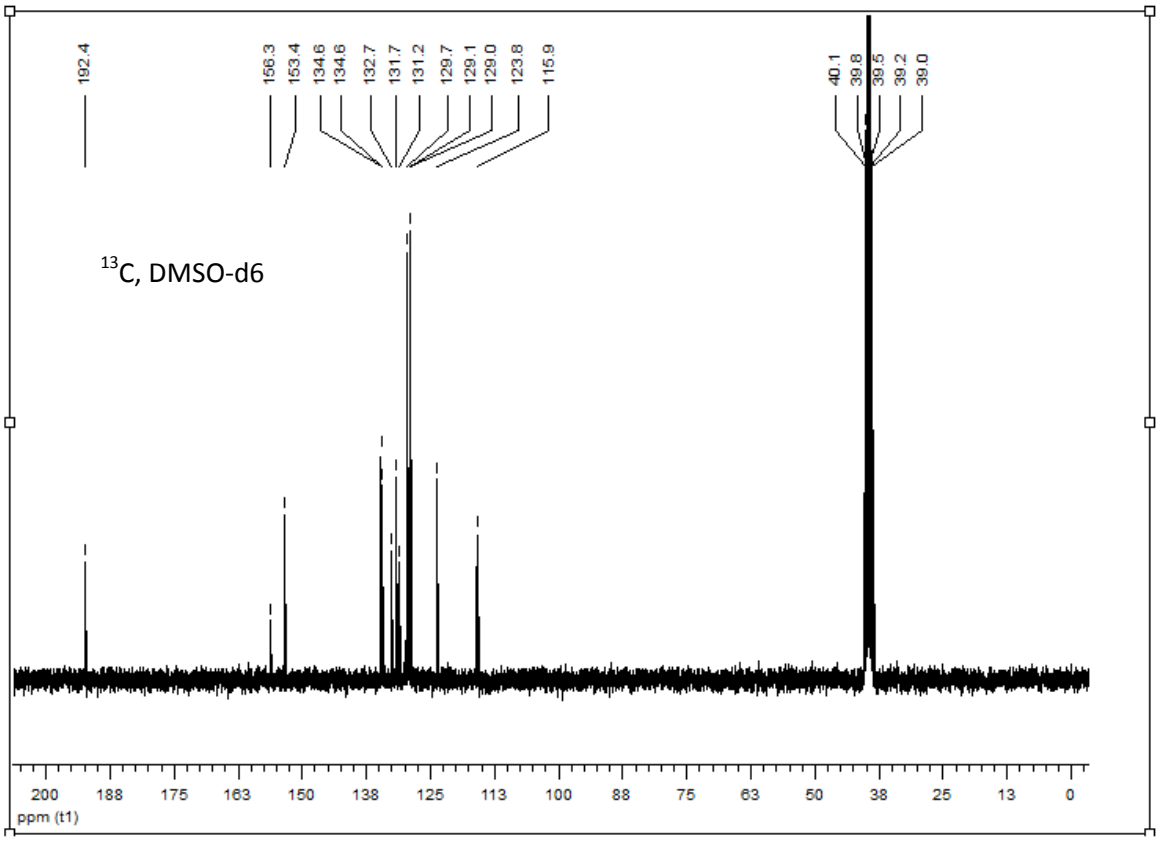
ppm (t1)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123.8
115.9
40.1
39.8
39.5
39.2
39.0
13C, DMSO-d6
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150
138
125
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100
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50
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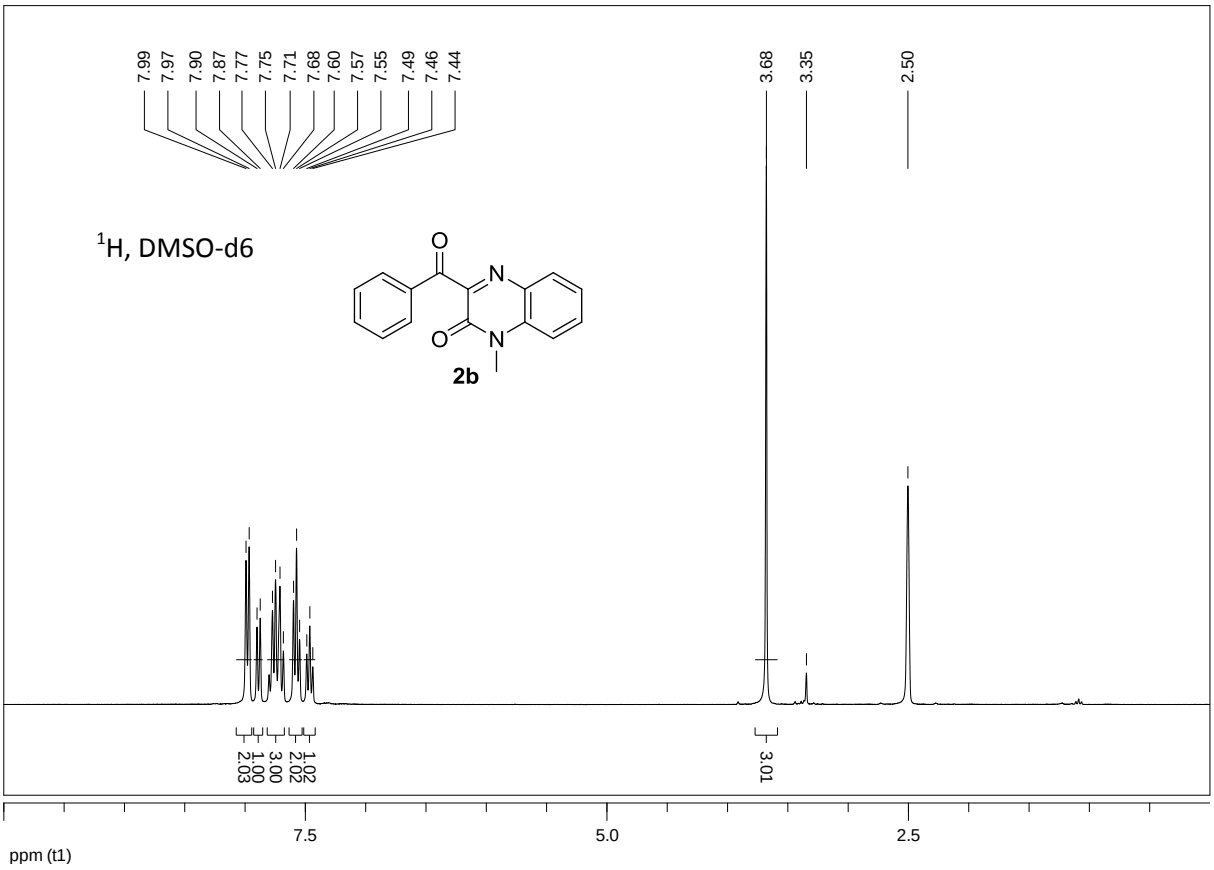
25
13
0
ppm (t1)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7.46
7.44
3.68
3.35
2.50
1H, DMSO-d6
O
N
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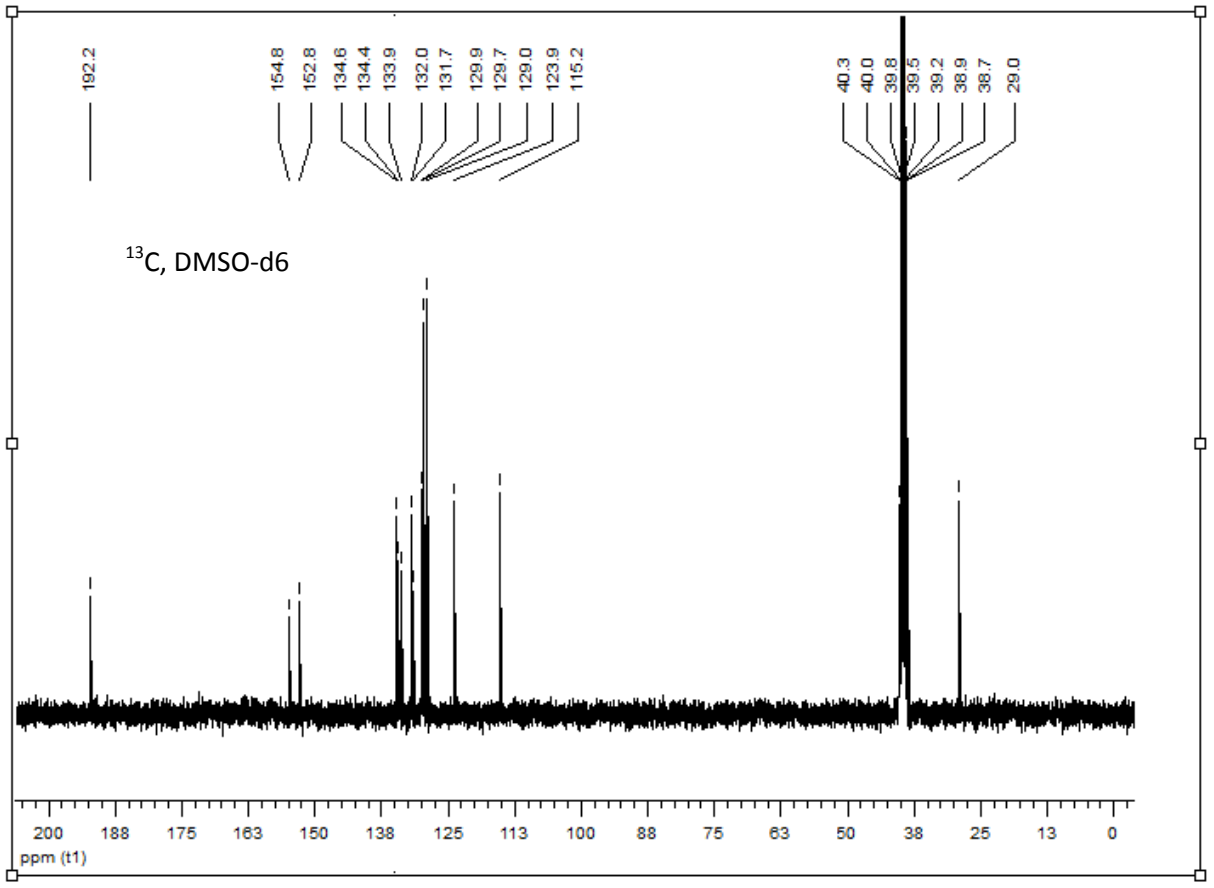
ppm (t1)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129.0
123.9
115.2
40.3
40.0
39.8
39.5
39.2
38.9
38.7
29.0
13C, DMSO-d6
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163
150
138
125
113
100
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50
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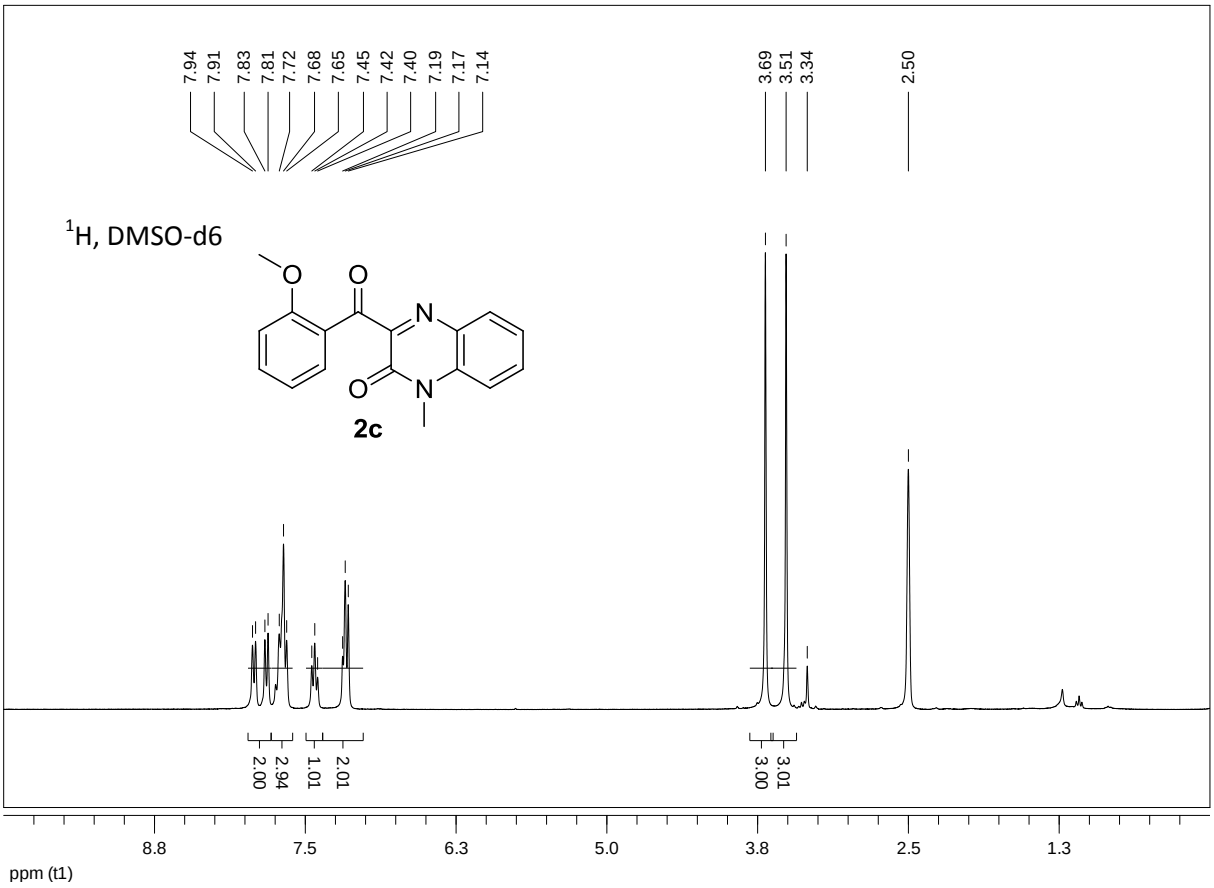
13
0
ppm (t1)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3.69
3.51
3.34
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1H, DMSO-d6
O
O
N
O
N
2c
2.00
2.94
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3.01
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6.3
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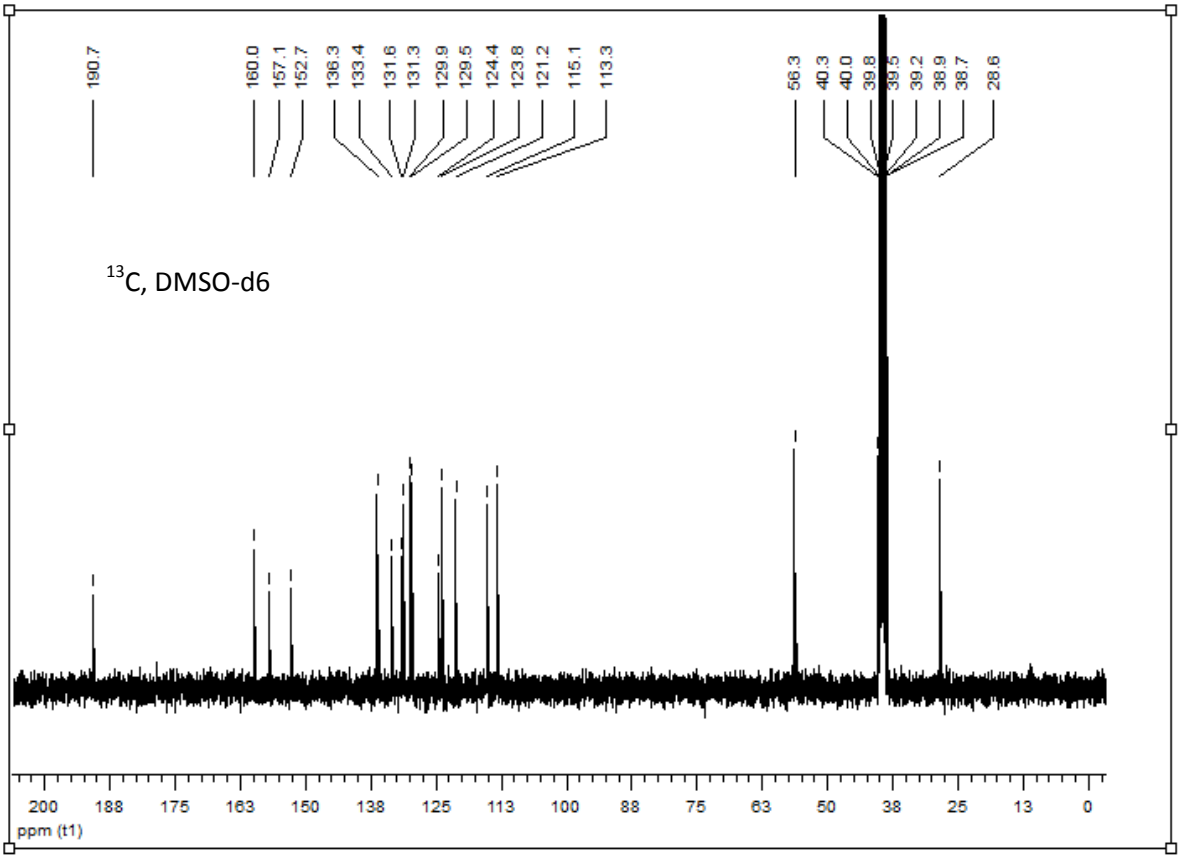
3.8
2.5
1.3
ppm (t1)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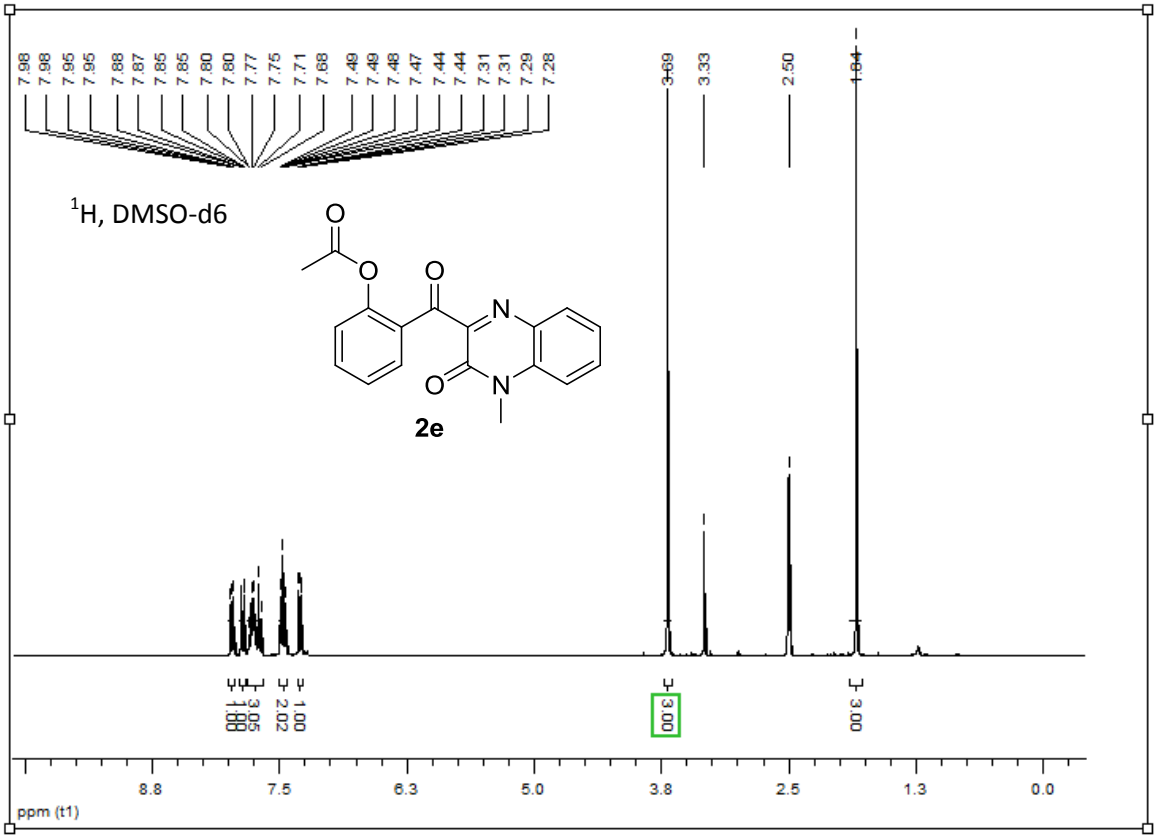
121.2
115.1
113.3
56.3
40.3
40.0
39.8
39.5
39.2
38.9
38.7
28.6
13C, DMSO-d6
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)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7.71
7.68
7.49
7.49
7.48
7.47
7.44
7.44
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7.31
7.29
7.28
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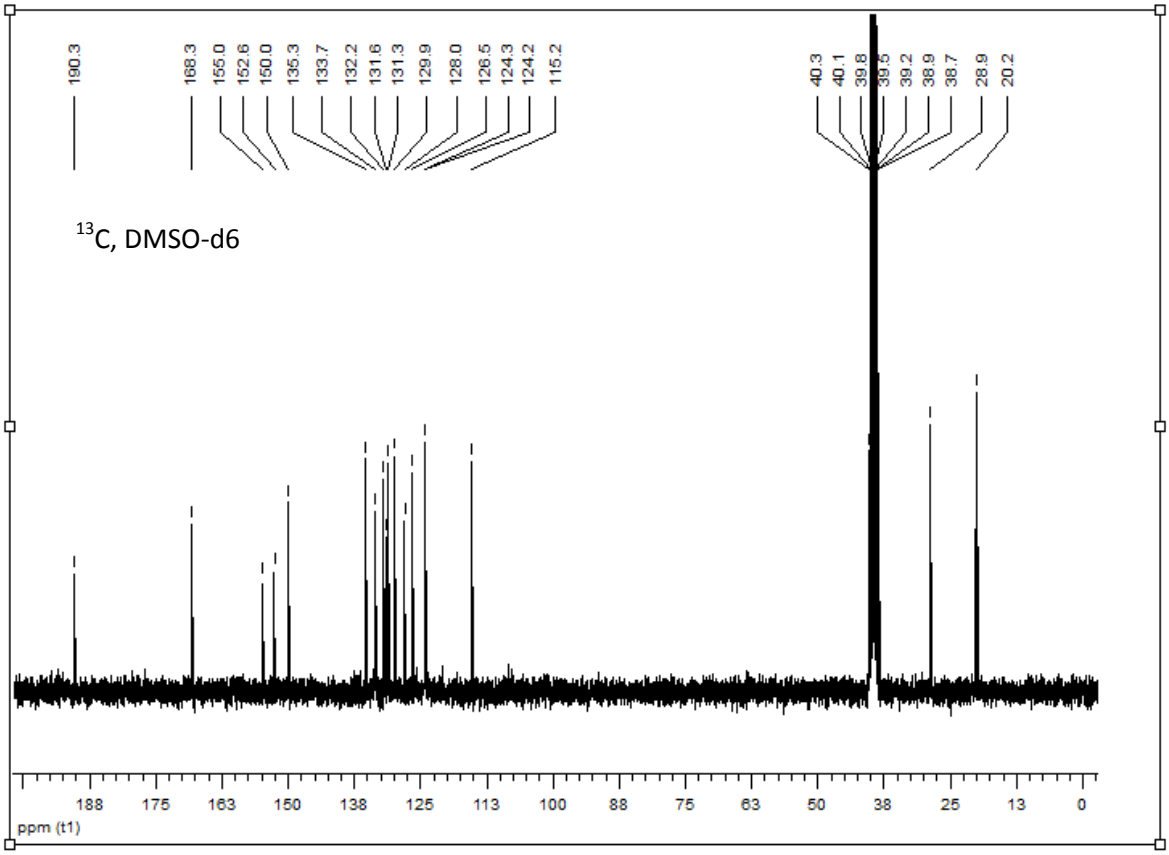
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124.3
124.2
115.2
40.3
40.1
39.8
39.5
39.2
38.9
38.7
28.9
20.2
13C, DMSO-d6
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163
150
138
125
113
100
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75
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50
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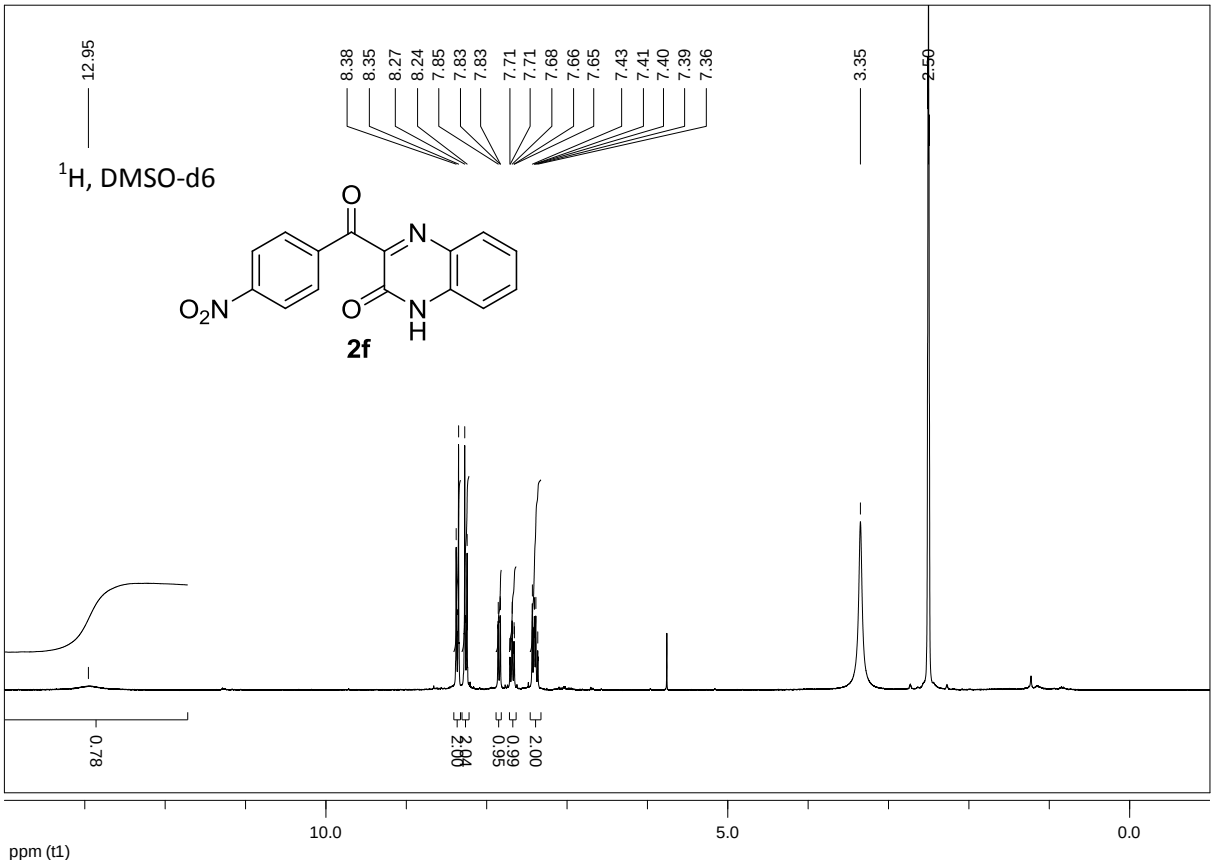
ppm (t1)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7.68
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7.65
7.43
7.41
7.40
7.39
7.36
3.35
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O
N
O_2N
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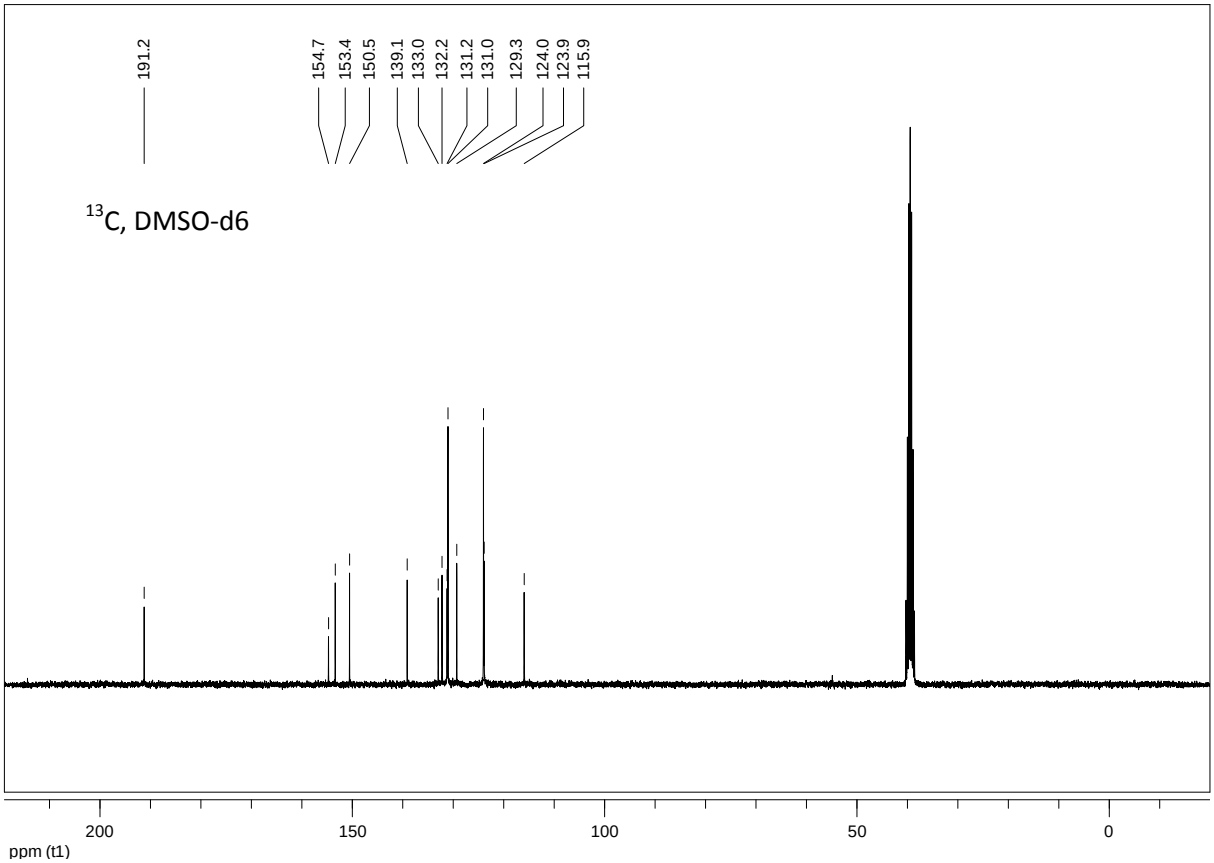
ppm (t1)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123.9
115.9
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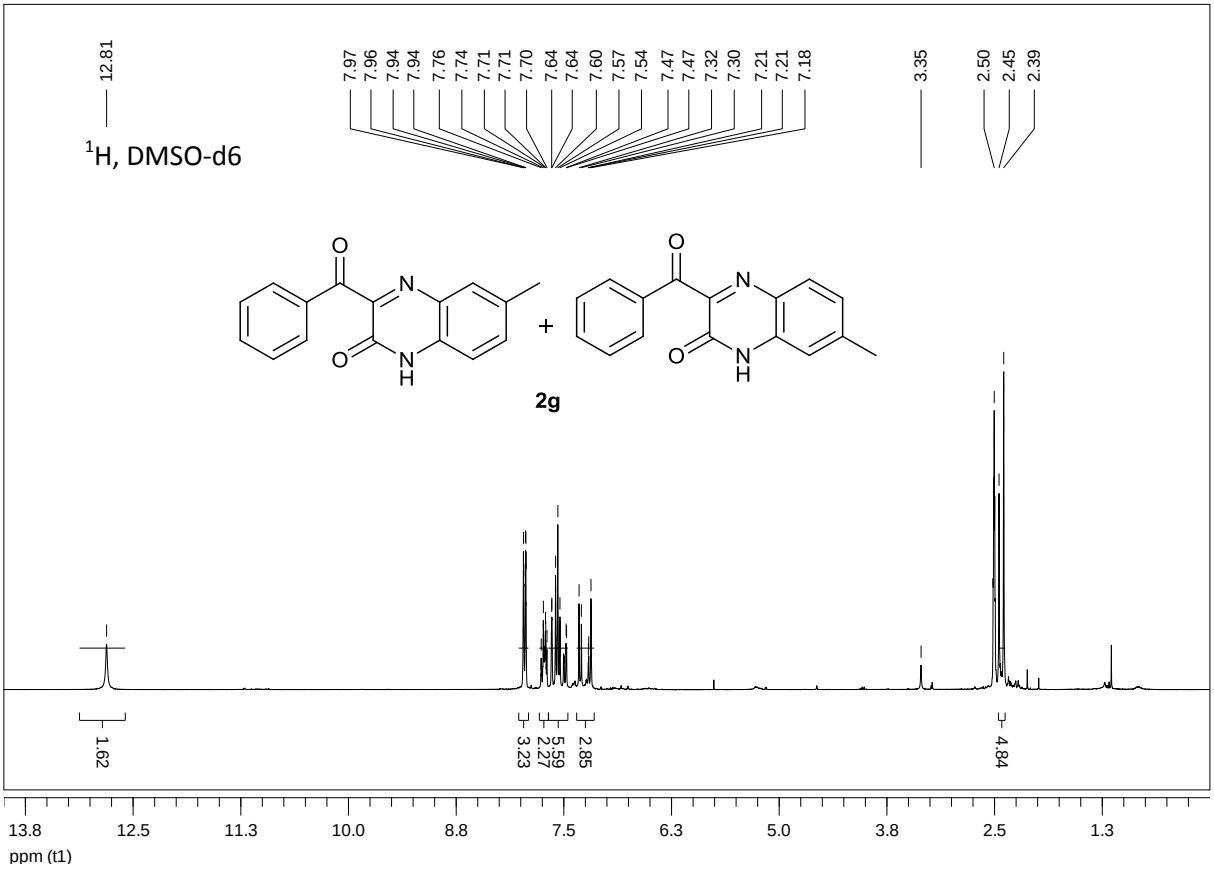
150
100
50
0
ppm (t1)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7.54
7.47
7.47
7.32
7.30
7.21
7.21
7.18
3.35
2.50
2.45
2.39
1H, DMSO-d6
2g
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3.23
2.27
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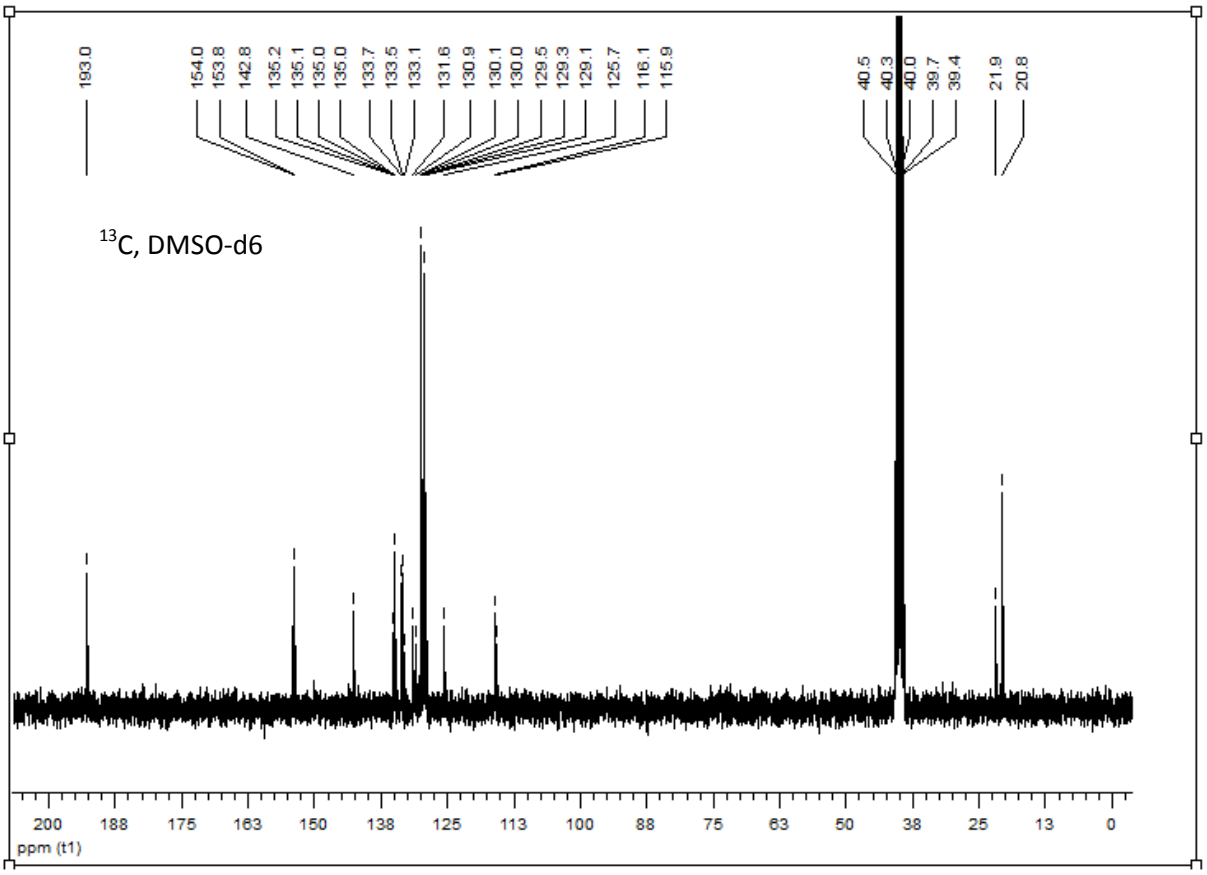
ppm (t1)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142.8
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135.0
135.0
133.7
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133.1
131.6
130.9
130.1
130.0
129.5
129.3
129.1
125.7
116.1
115.9
40.5
40.3
40.0
39.7
39.4
21.9
20.8
13C, DMSO-d6
200
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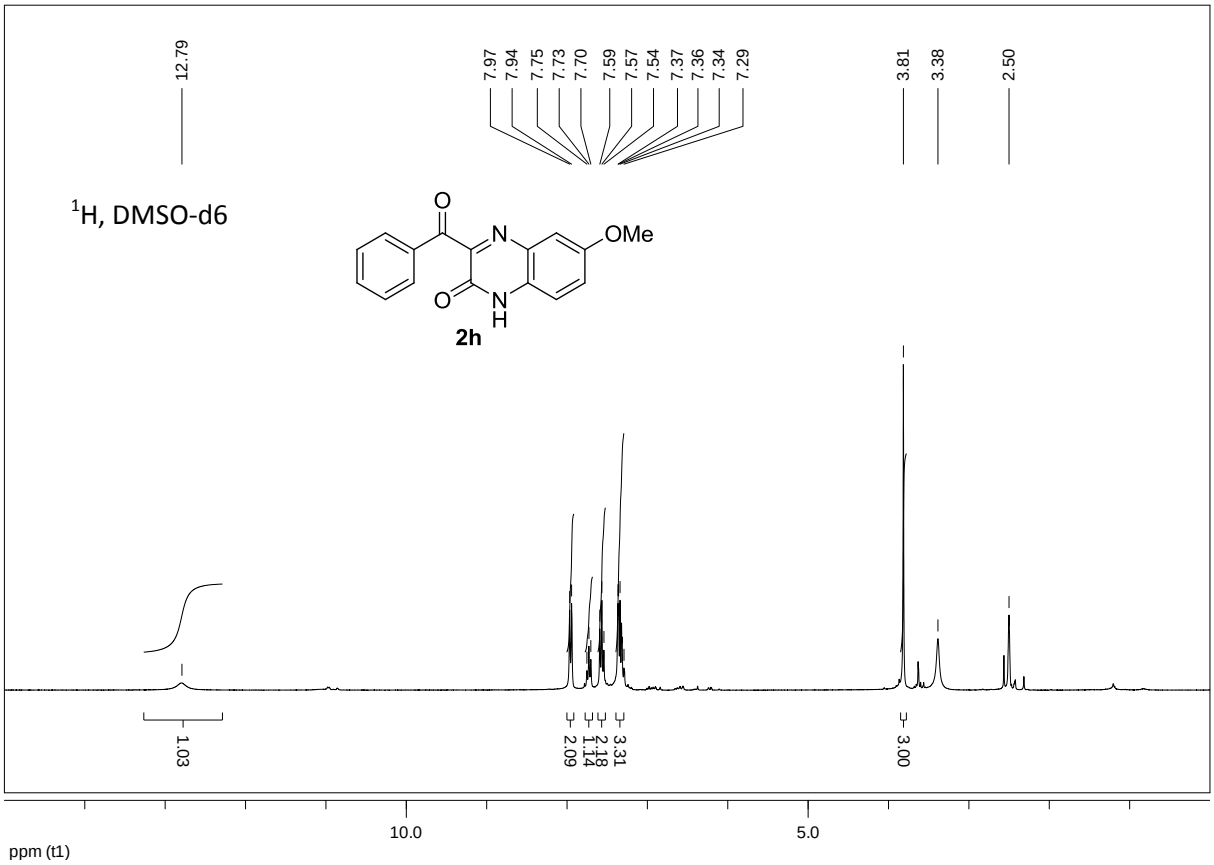
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)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7.57
7.54
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7.34
7.29
3.81
3.38
2.50
1H, DMSO-d6
O
N
OMe
O
N
H
2h
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2.09
1.14
2.18
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3.00
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5.0
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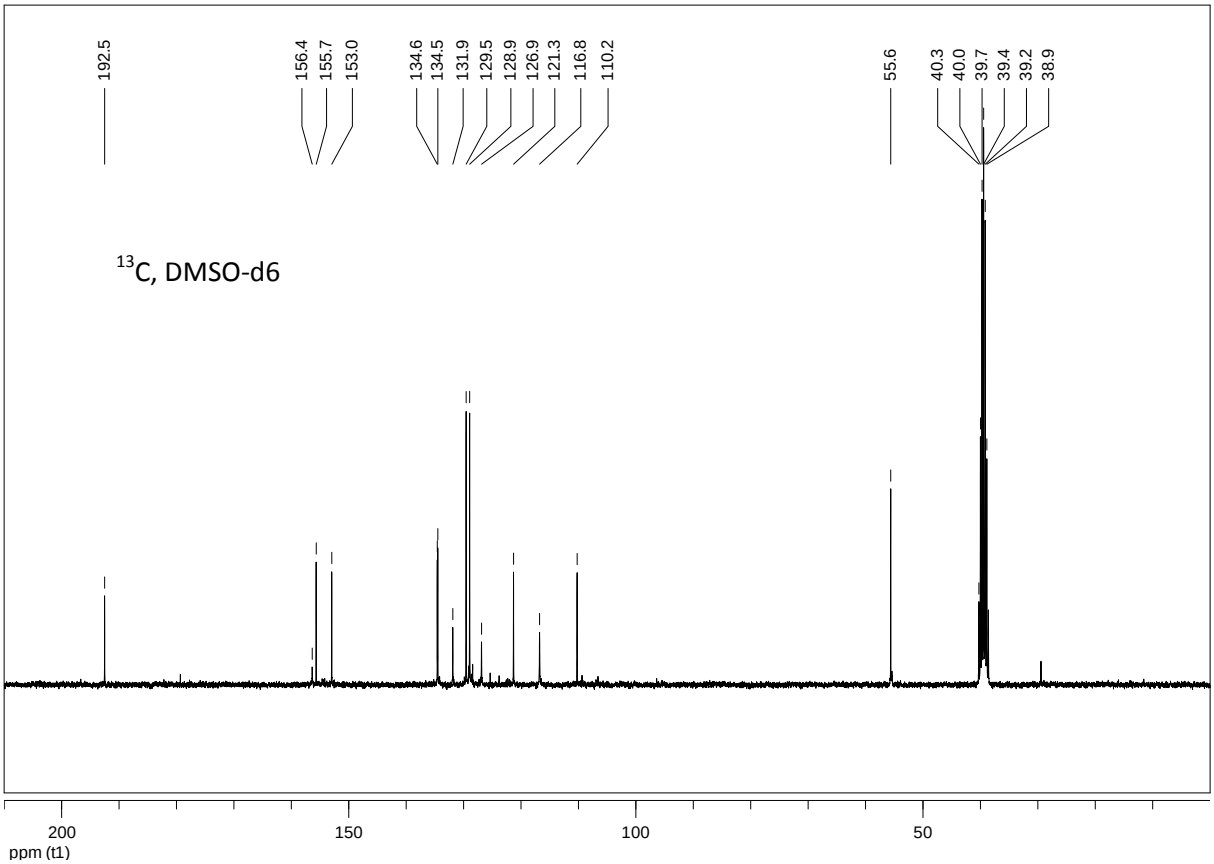
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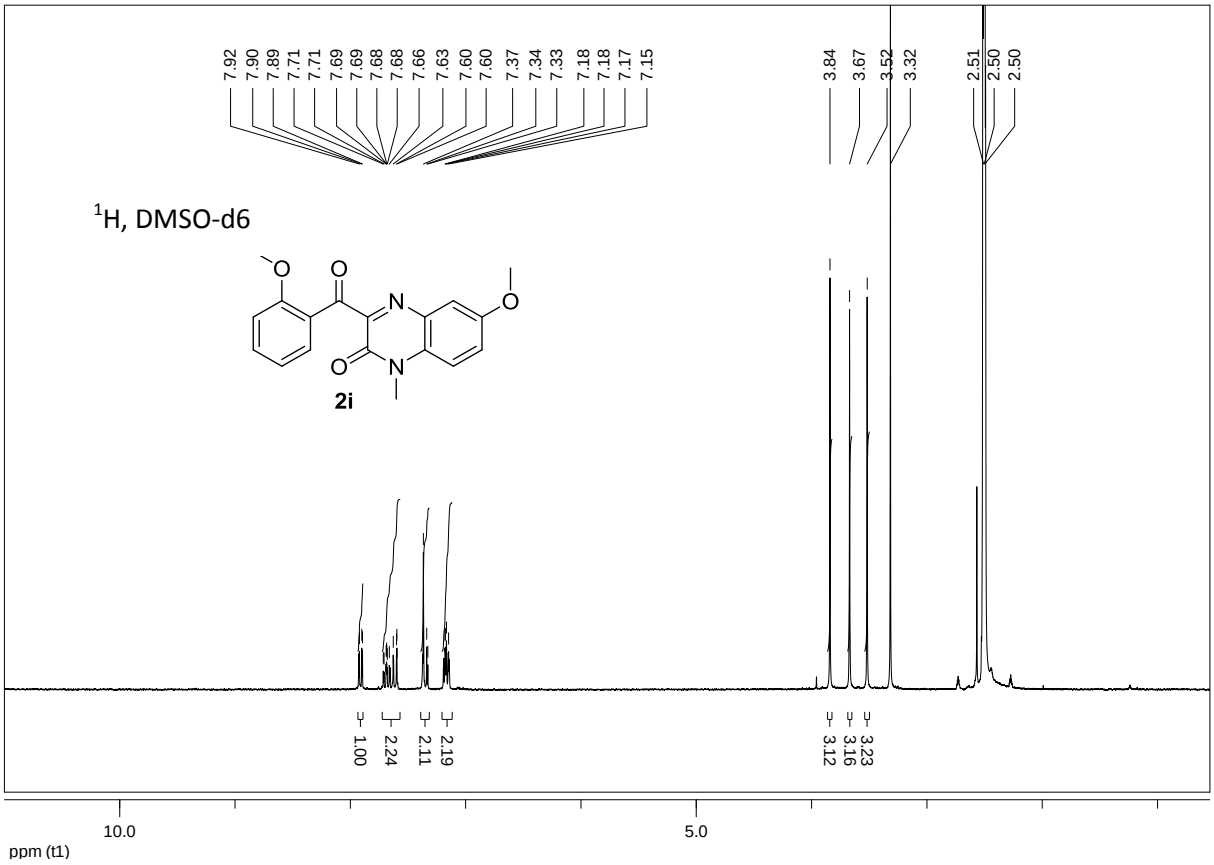
153.0
134.6
134.5
131.9
129.5
128.9
126.9
121.3
116.8
110.2
55.6
40.3
40.0
39.7
39.4
39.2
38.9
13C, DMSO-d6
200
150
100
50
ppm (t1)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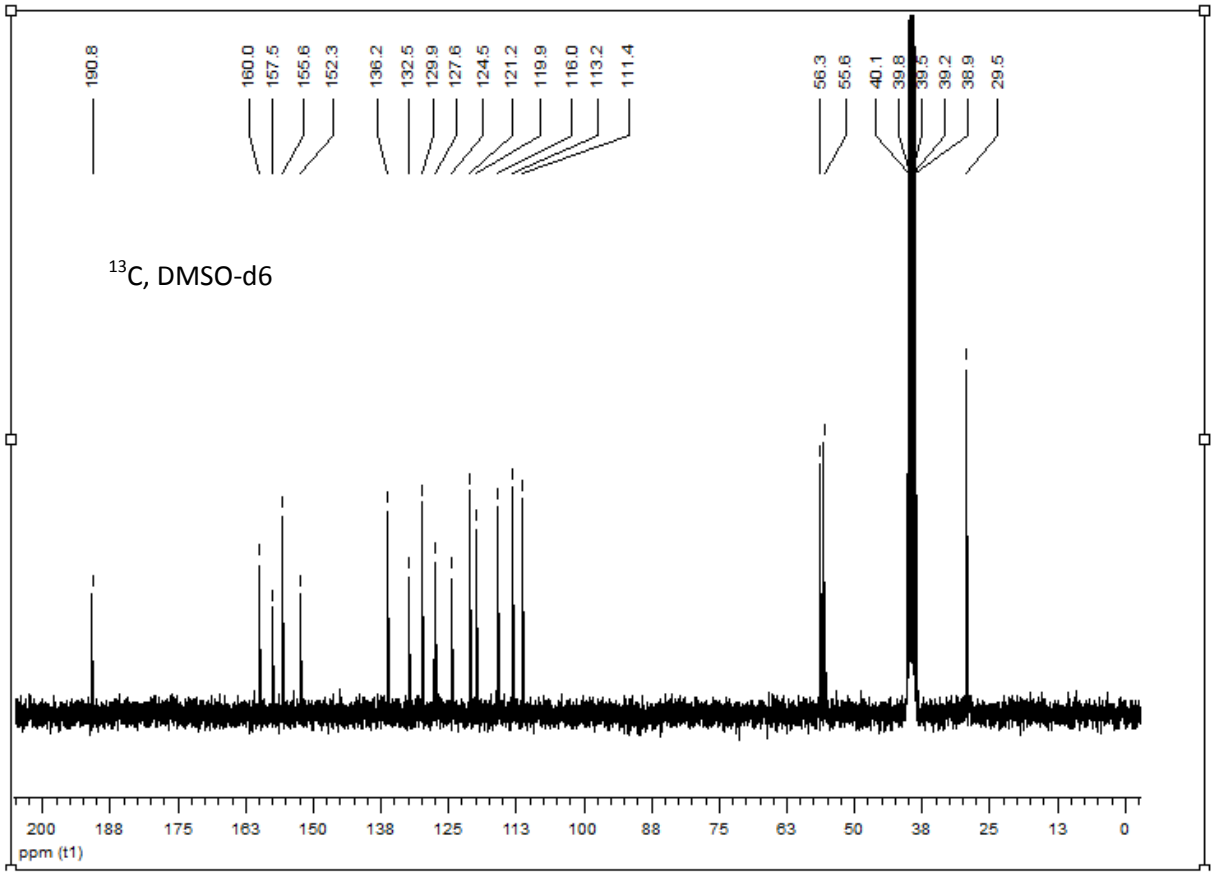
ppm (t1)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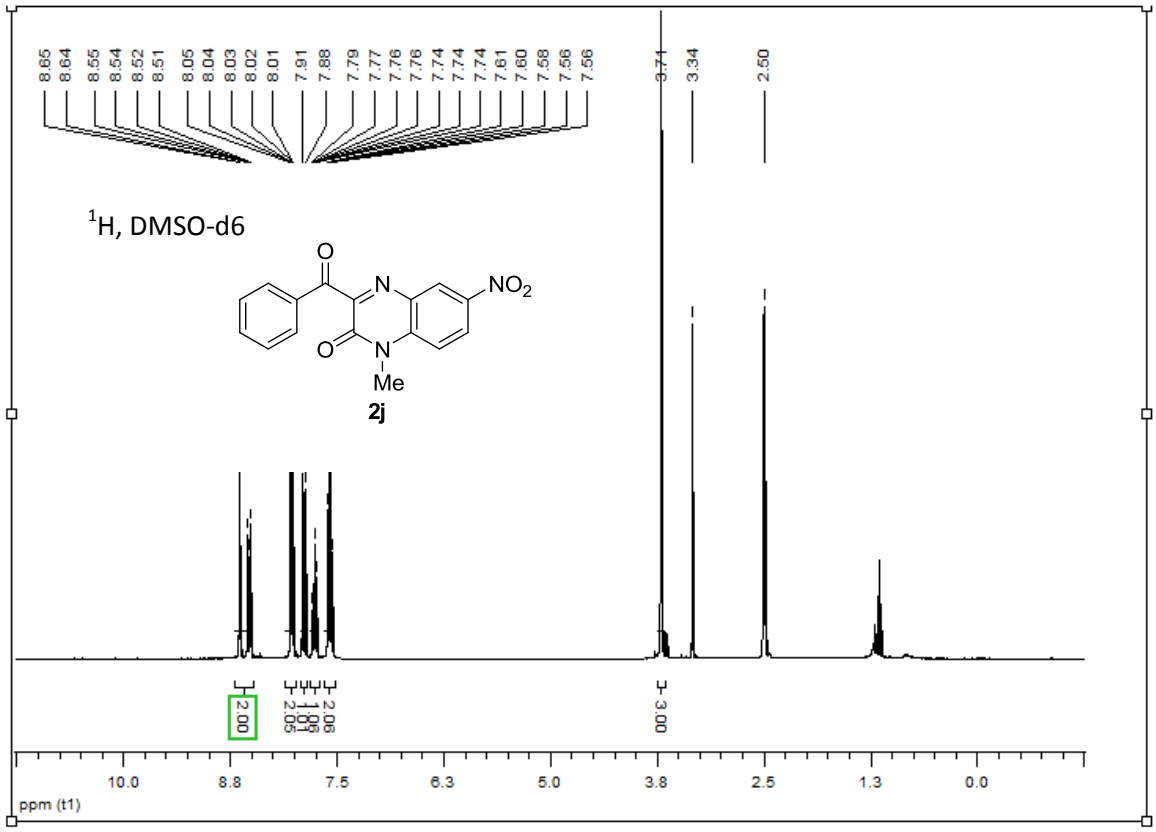
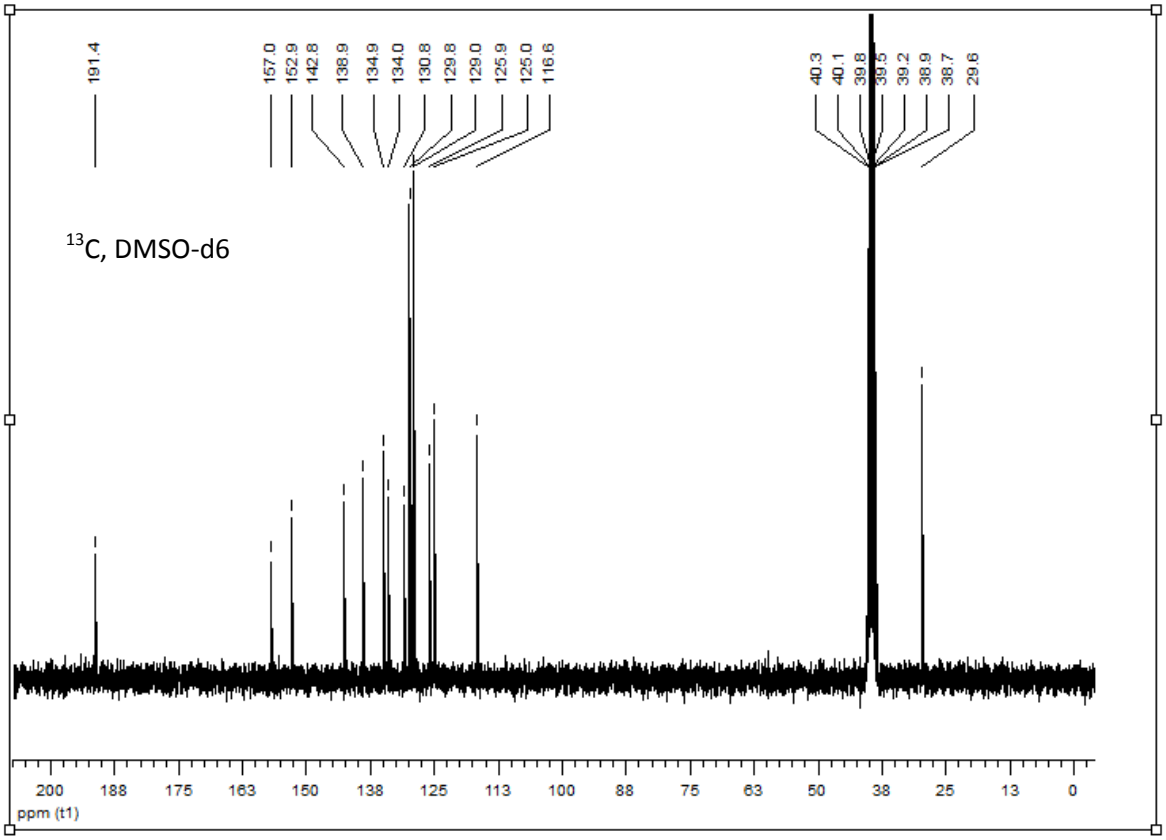
39.5
39.2
38.9
29.5
13C, DMSO-d6
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)


8.65
8.64
8.55
8.54
8.52
8.51
8.05
8.04
8.03
8.02
8.01
7.91
7.88
7.79
7.77
7.76
7.76
7.74
7.74
7.74
7.61
7.60
7.58
7.56
7.56
3.71
3.34
2.50
1H, DMSO-d6
O
N
NO2
O
N
Me
2j
2.00
2.06
1.98
2.06
3.00
10.0
8.8
7.5
6.3
5.0
3.8
2.5
1.3
0.0
ppm (t1)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157.0
152.9
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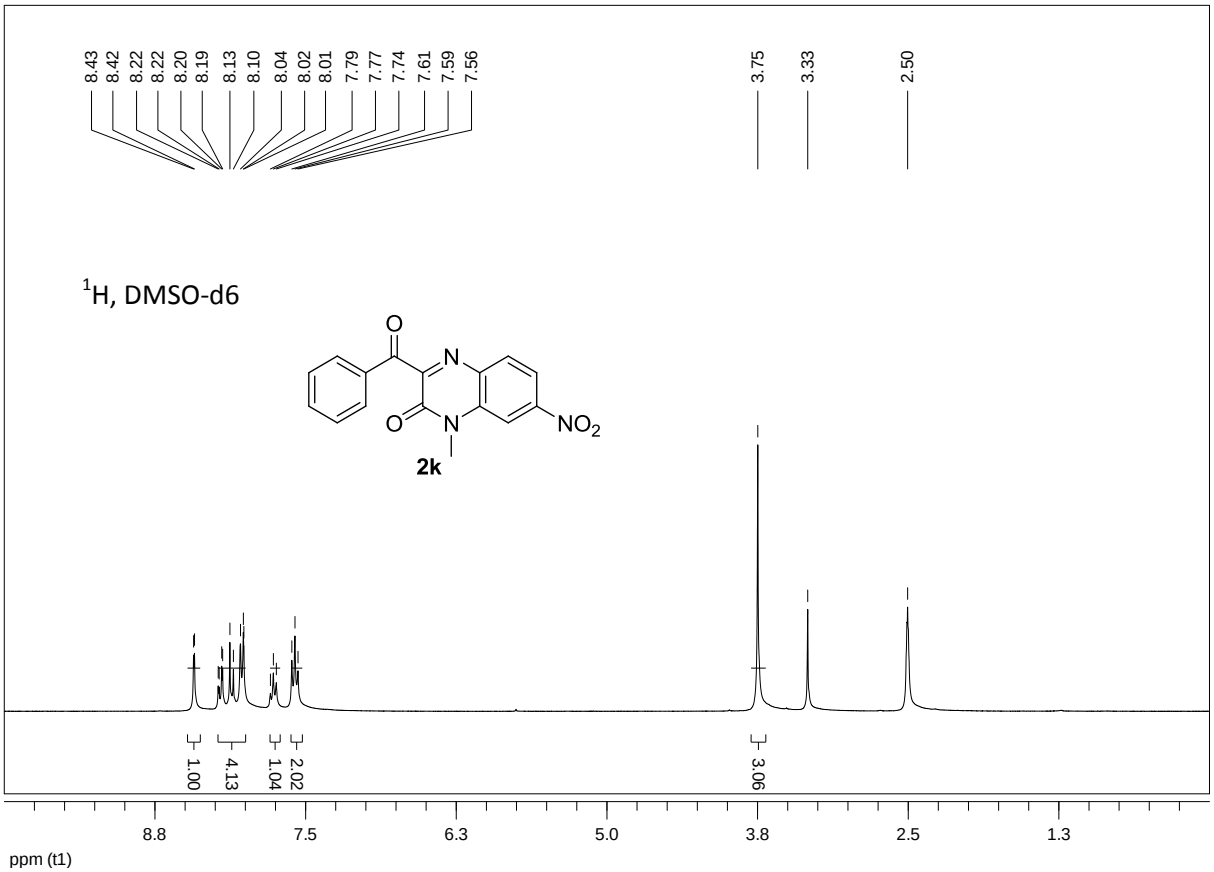
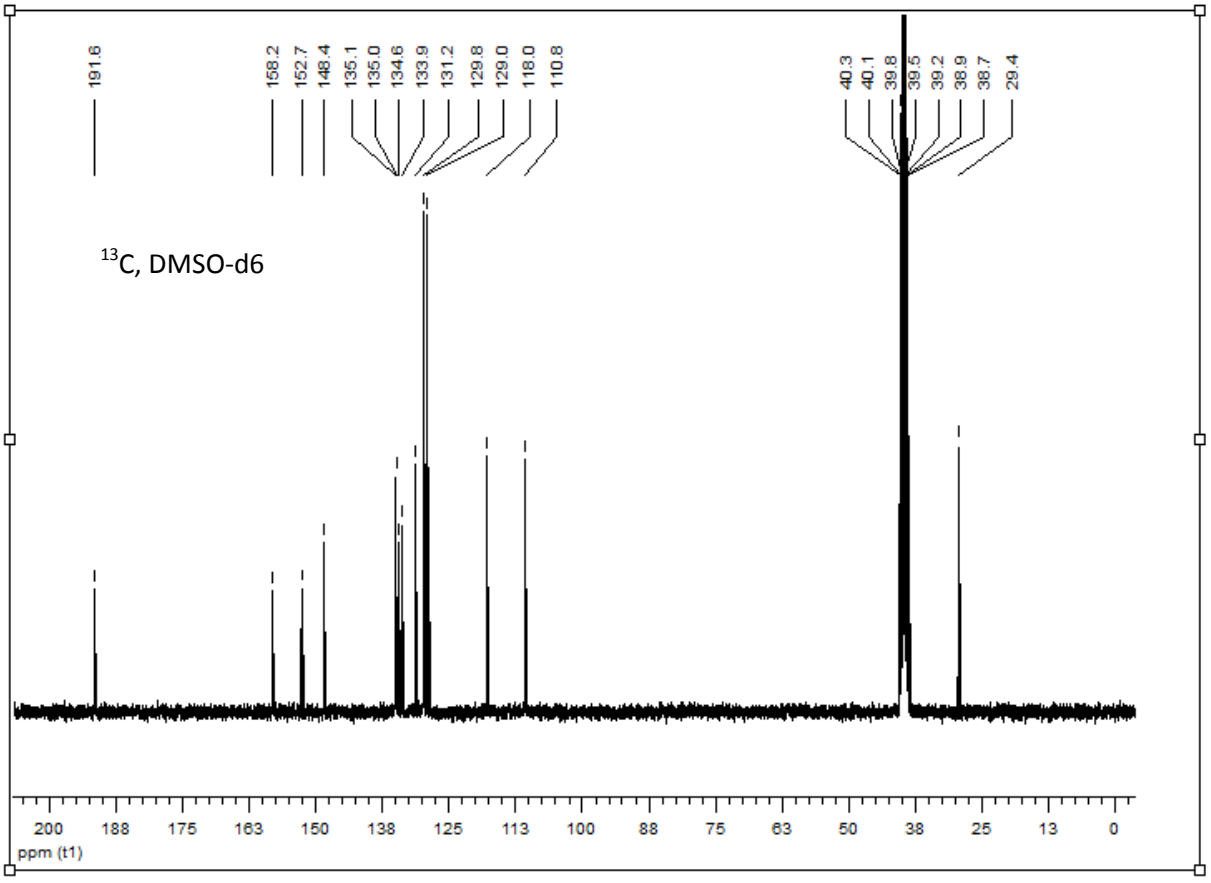
138.9
134.9
134.0
130.8
129.8
129.0
125.9
125.0
116.6
40.3
40.1
39.8
39.5
39.2
38.9
38.7
29.6
13C, DMSO-d6
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)


8.43
8.42
8.22
8.22
8.20
8.19
8.13
8.10
8.04
8.02
8.01
7.79
7.77
7.74
7.61
7.59
7.56
3.75
3.33
2.50
1H, DMSO-d6
O
N
O
N
NO2
2k
1.00
4.13
1.04
2.02
3.06
8.8
7.5
6.3
5.0
3.8
2.5
1.3
ppm (t1)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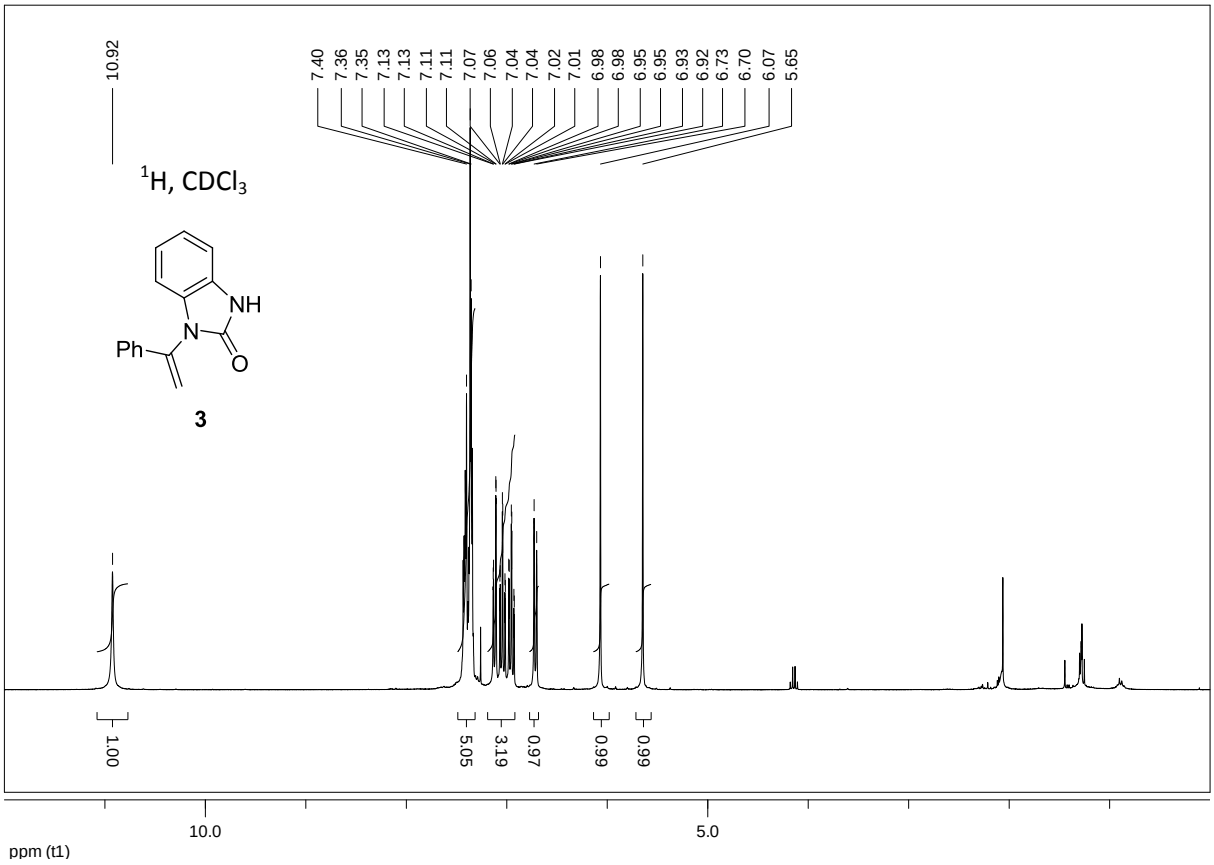
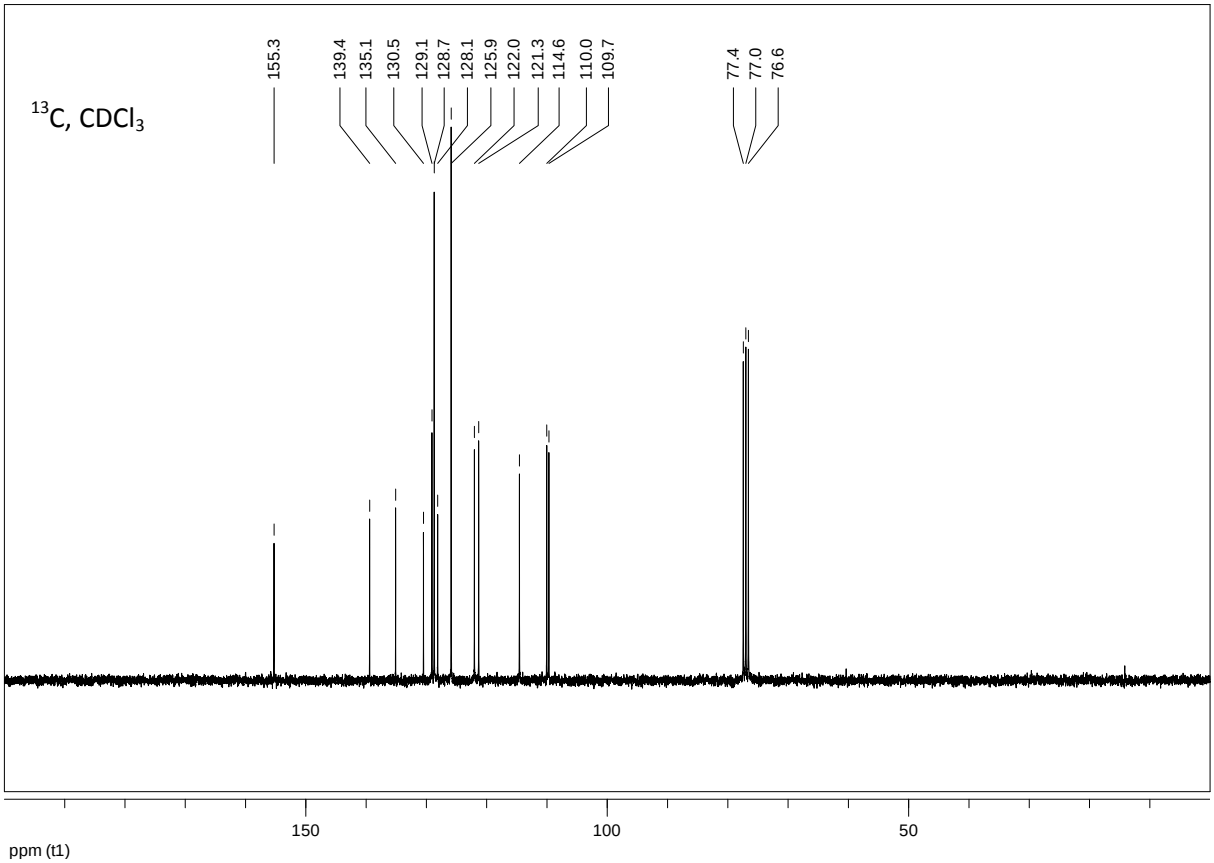
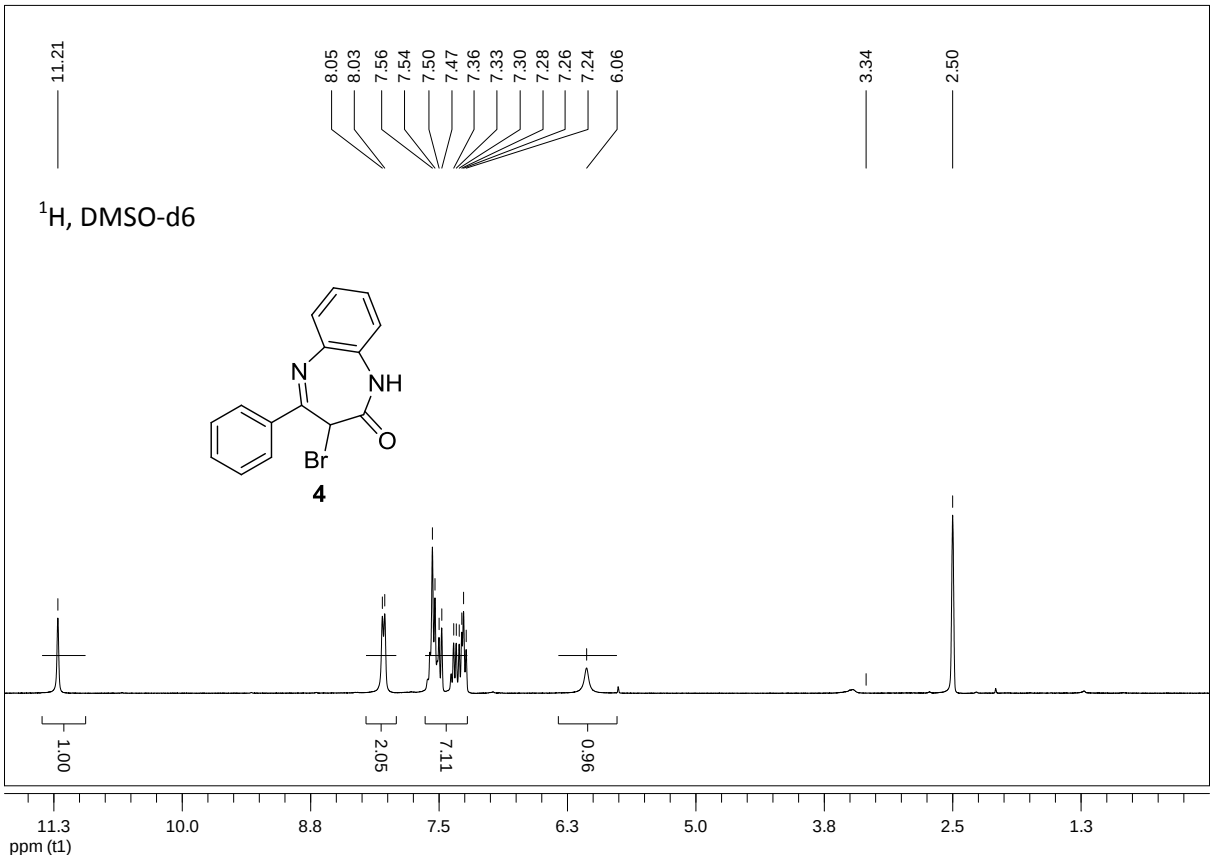
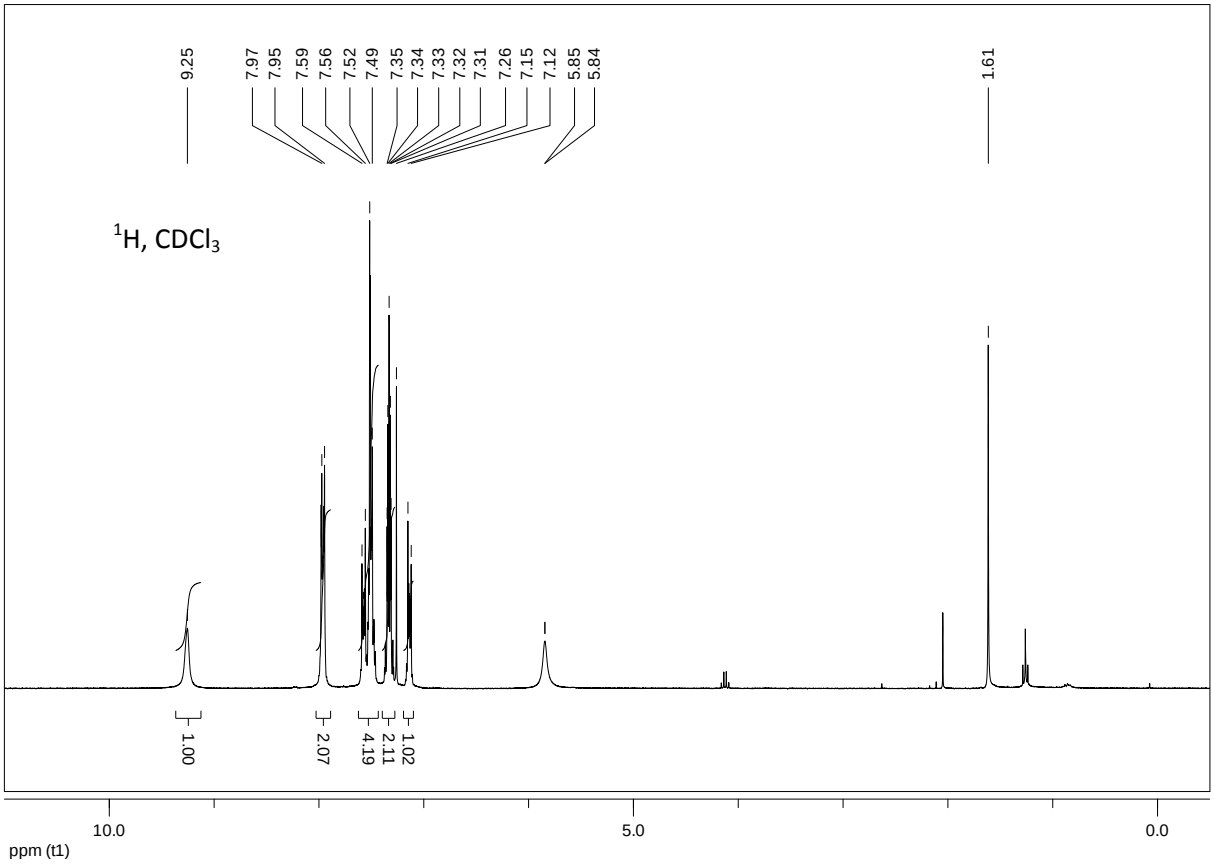
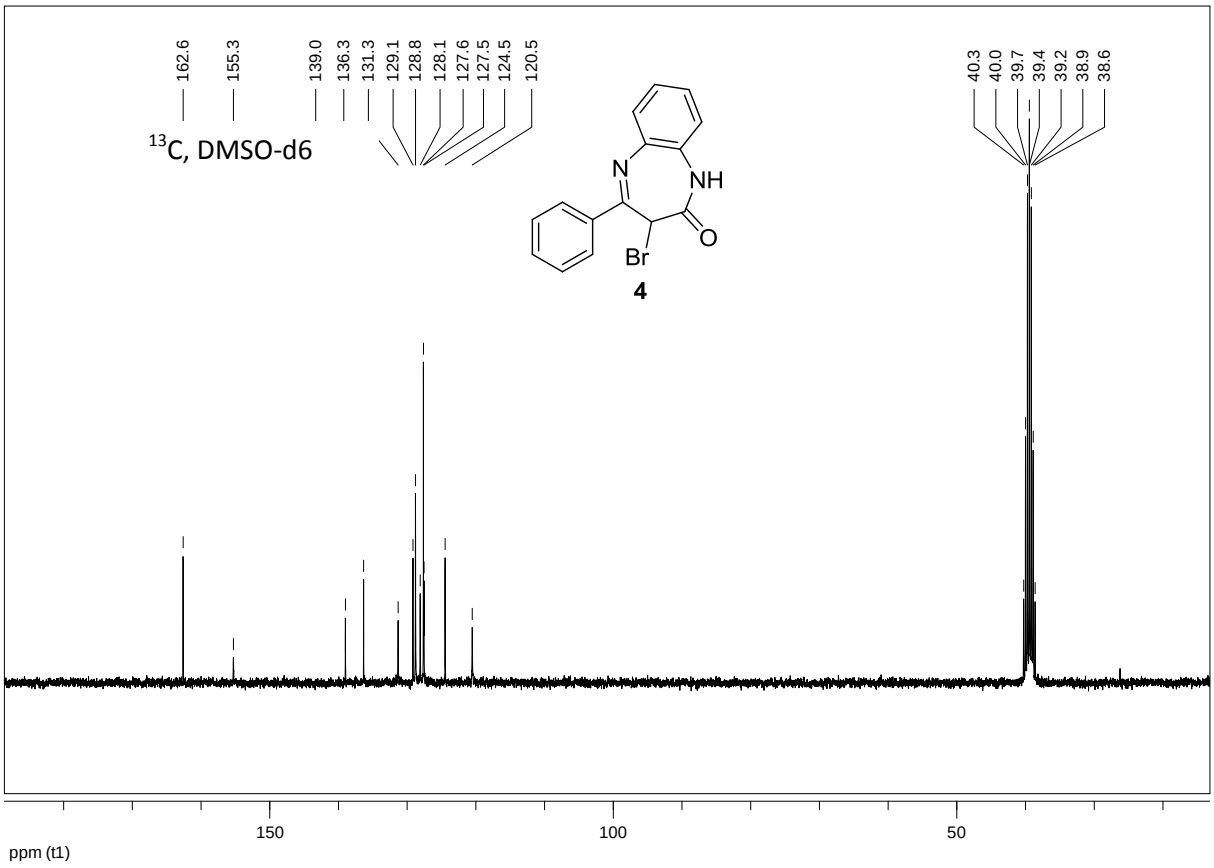
158.2
152.7
148.4
135.1
135.0
134.6
133.9
131.2
129.8
129.0
118.0
110.8
40.3
40.1
39.8
39.5
39.2
38.9
38.7
29.4
13C, DMSO-d6
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)

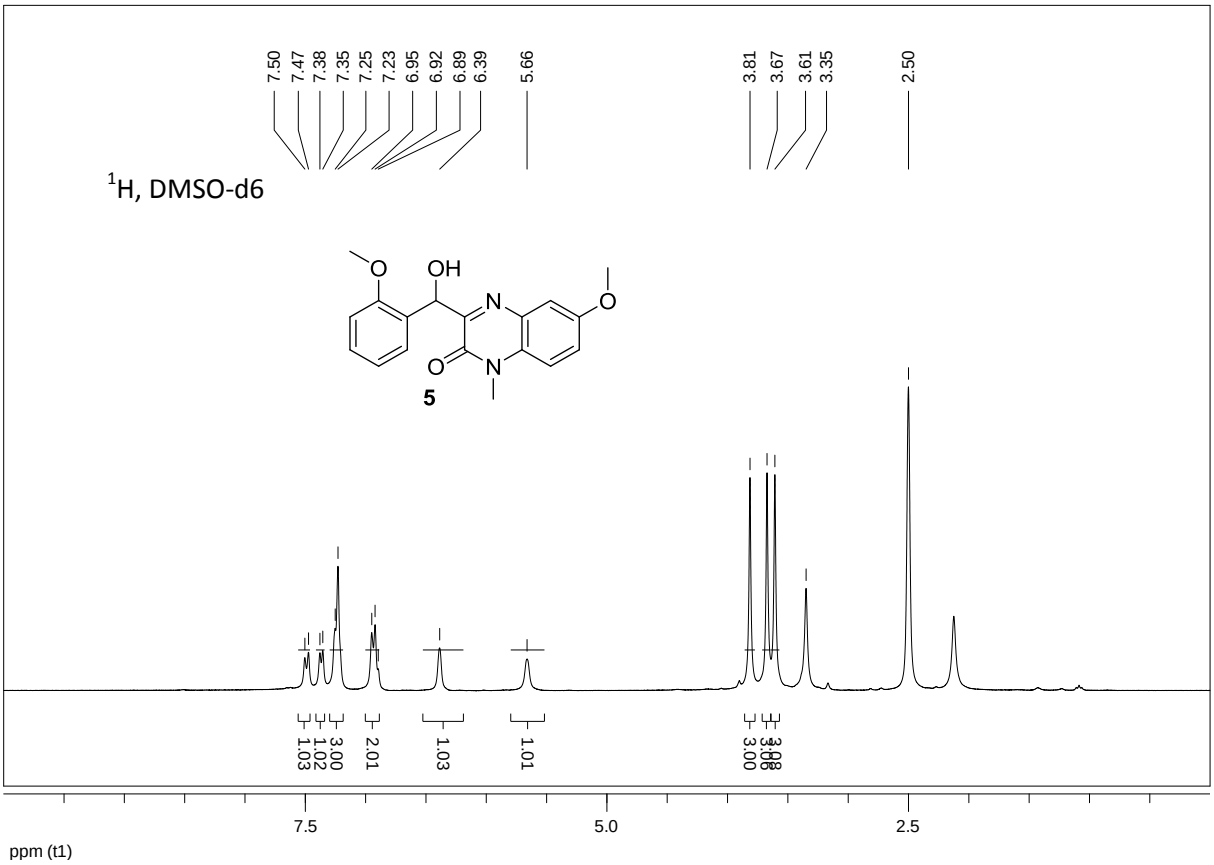

10.92
7.40
7.36
7.35
7.13
7.13
7.11
7.11
7.07
7.06
7.04
7.04
7.02
7.01
6.98
6.98
6.95
6.95
6.93
6.92
6.73
6.70
6.07
5.65
^{1}H, CDCl$_3$
NH
N
Ph
O
3
1.00
5.05
3.19
0.97
0.99
0.99
10.0
5.0
ppm (t1)

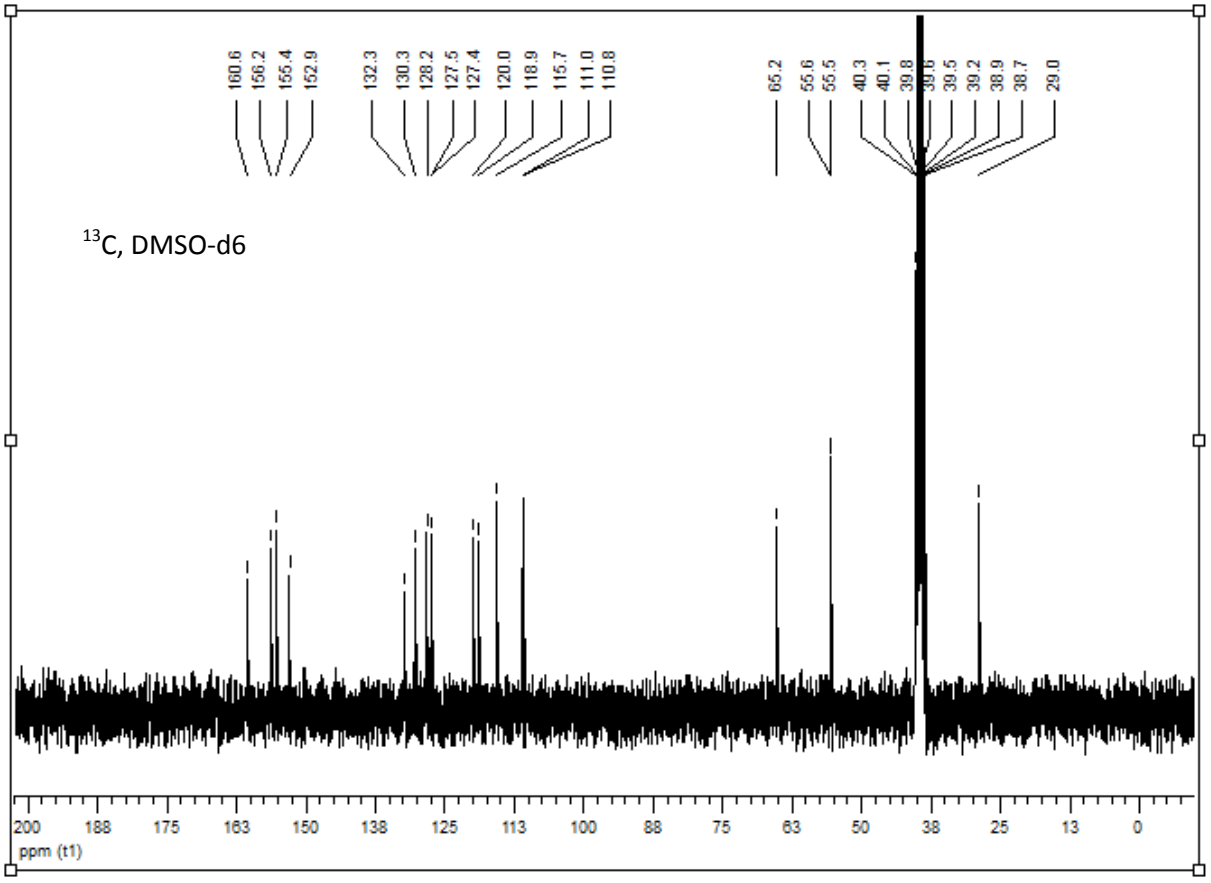

155.3
139.4
135.1
130.5
129.1
128.7
128.1
125.9
122.0
121.3
114.6
110.0
109.7
77.4
77.0
76.6
^{13}C, CDCl$_3$
150
100
50
ppm (t1)

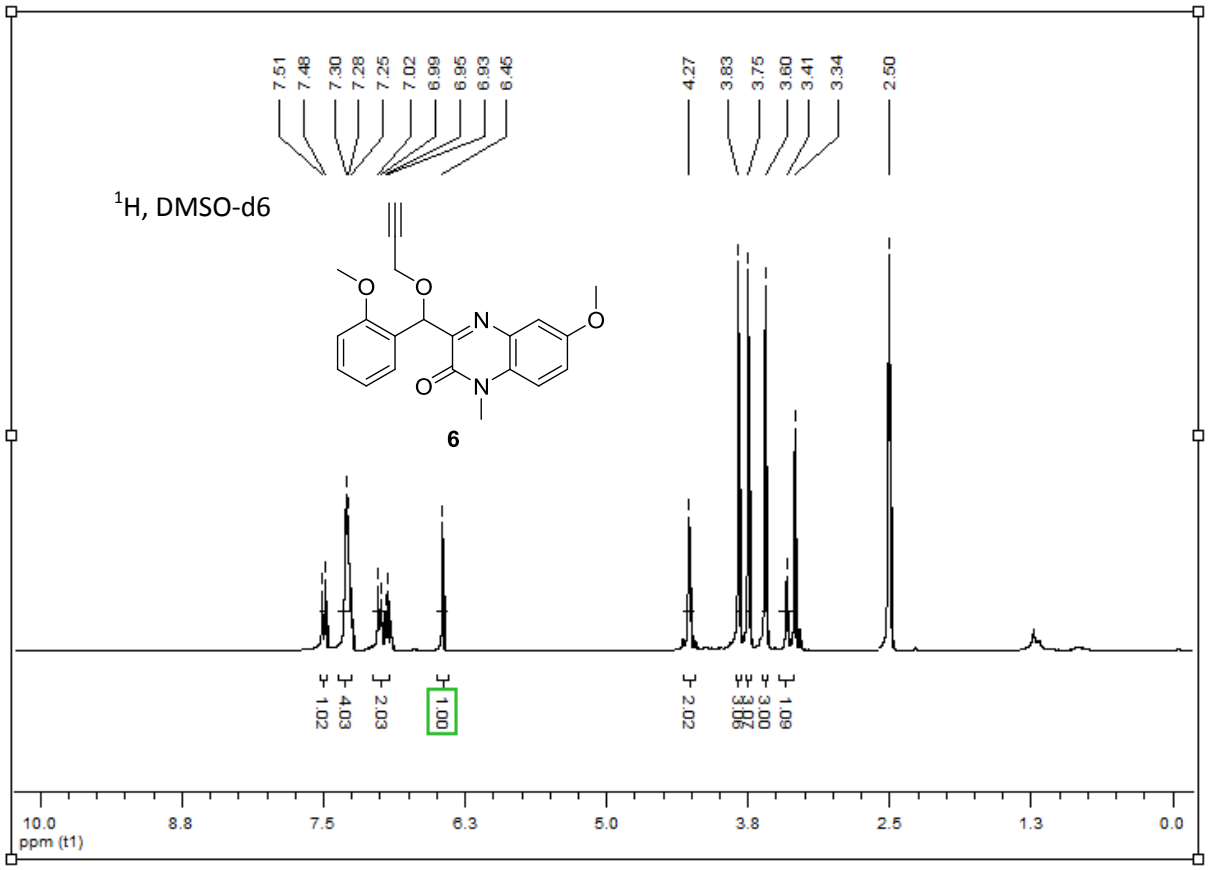

11.21
8.05
8.03
7.56
7.54
7.50
7.47
7.36
7.33
7.30
7.28
7.26
7.24
6.06
3.34
2.50
1H, DMSO-d6
N
NH
O
Br
4
1.00
2.05
7.11
0.96
11.3
10.0
8.8
7.5
6.3
5.0
3.8
2.5
1.3
ppm (t1)

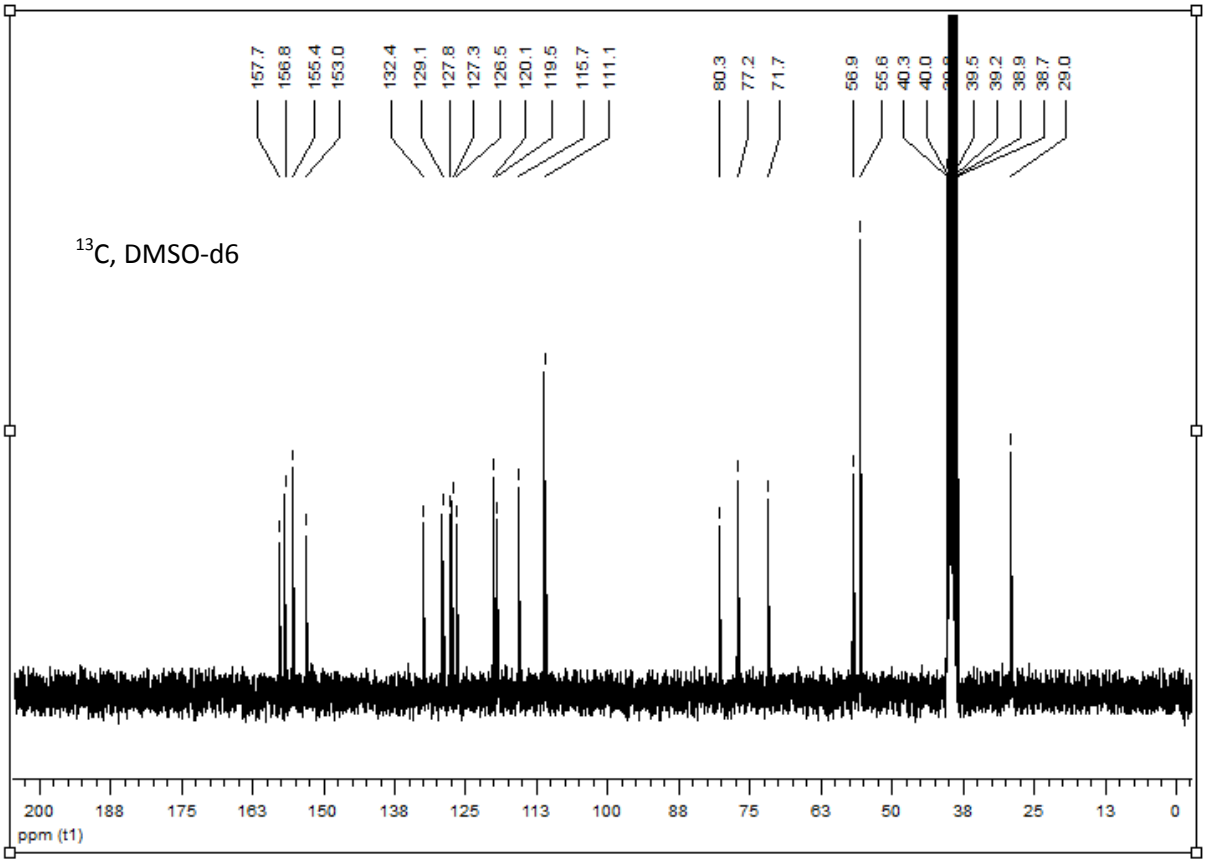

9.25
7.97
7.95
7.59
7.56
7.52
7.49
7.35
7.34
7.33
7.32
7.31
7.26
7.15
7.12
5.85
5.84
1.61
1H, CDCl3
1.00
2.07
4.19
2.11
1.02
10.0
5.0
0.0
ppm (t1)

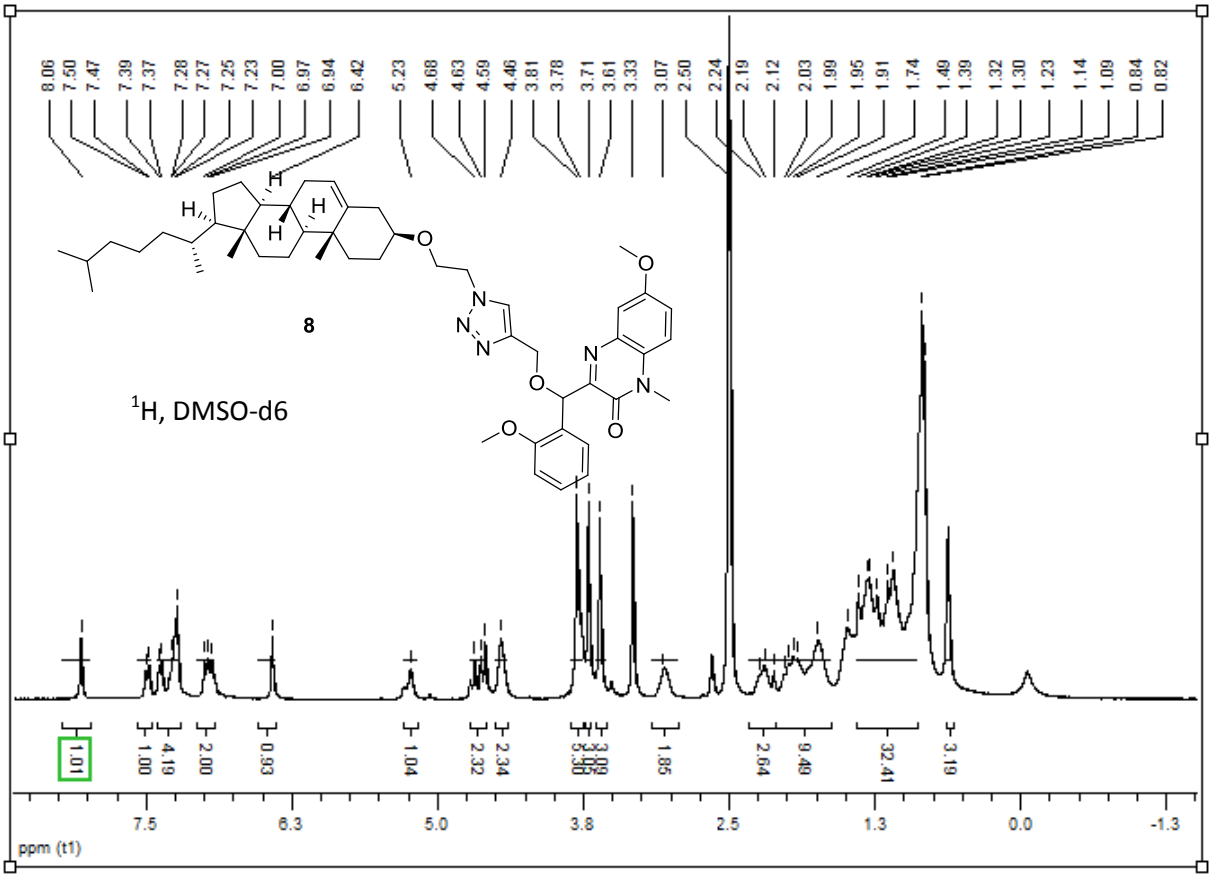

162.6
155.3
139.0
136.3
131.3
129.1
128.8
128.1
127.6
127.5
124.5
120.5
40.3
40.0
39.7
39.4
39.2
38.9
38.6
13C, DMSO-d6
N
NH
O
Br
4
150
100
50
ppm (t1)

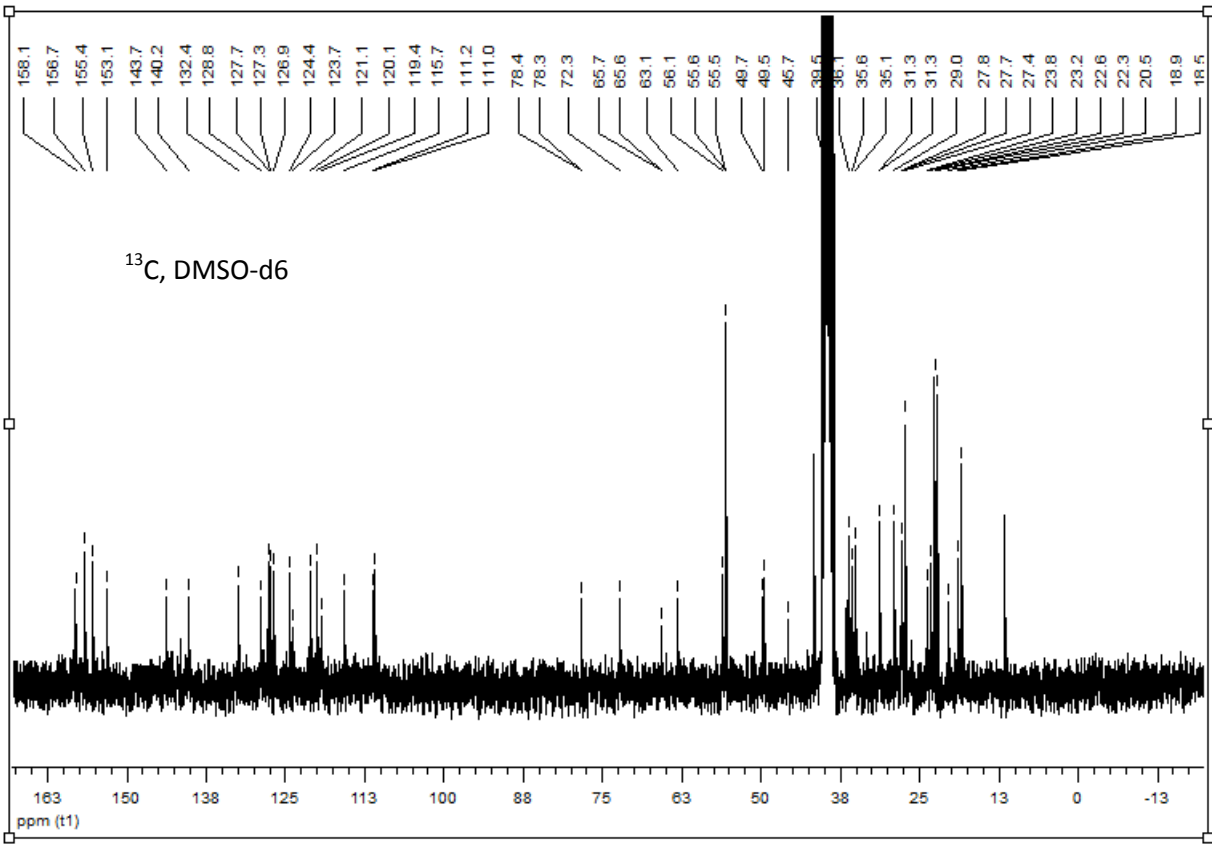

7.50
7.47
7.38
7.35
7.25
7.23
6.95
6.92
6.89
6.39
5.66
3.81
3.67
3.61
3.35
2.50
1H, DMSO-d6
O
OH
N
O
O
N
5
1.03
1.02
3.00
2.01
1.03
1.01
3.00
3.08
3.08
7.5
5.0
2.5
ppm (t1)


160.6
156.2
155.4
152.9
132.3
130.3
128.2
127.5
127.4
120.0
118.9
115.7
111.0
110.8
65.2
55.6
55.5
40.3
40.1
39.8
39.6
39.5
39.2
38.9
38.7
29.0
13C, DMSO-d6
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)

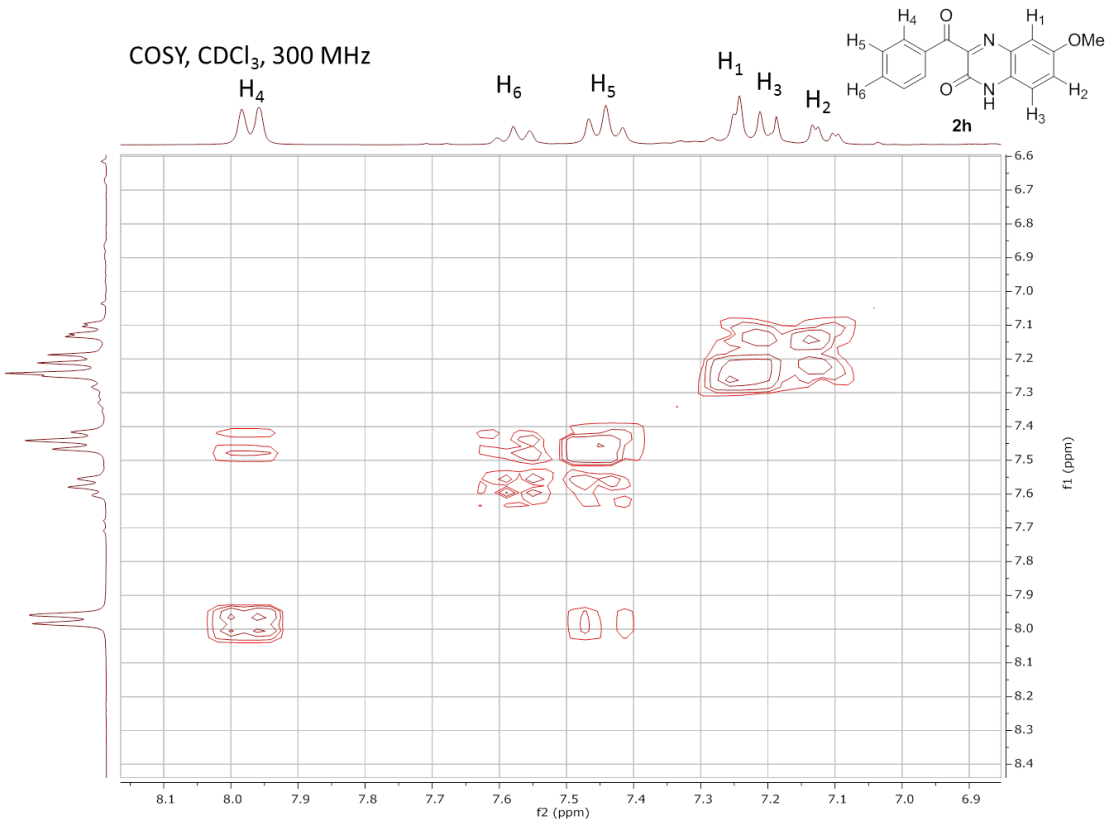

7.51
7.48
7.30
7.28
7.25
7.02
6.99
6.95
6.93
6.45
4.27
3.83
3.75
3.60
3.41
3.34
2.50
1H, DMSO-d6
O
O
N
O
O
N
6
1.02
4.03
2.03
1.00
2.02
3.06
3.00
1.09
10.0
8.8
7.5
6.3
5.0
3.8
2.5
1.3
0.0
ppm (t1)

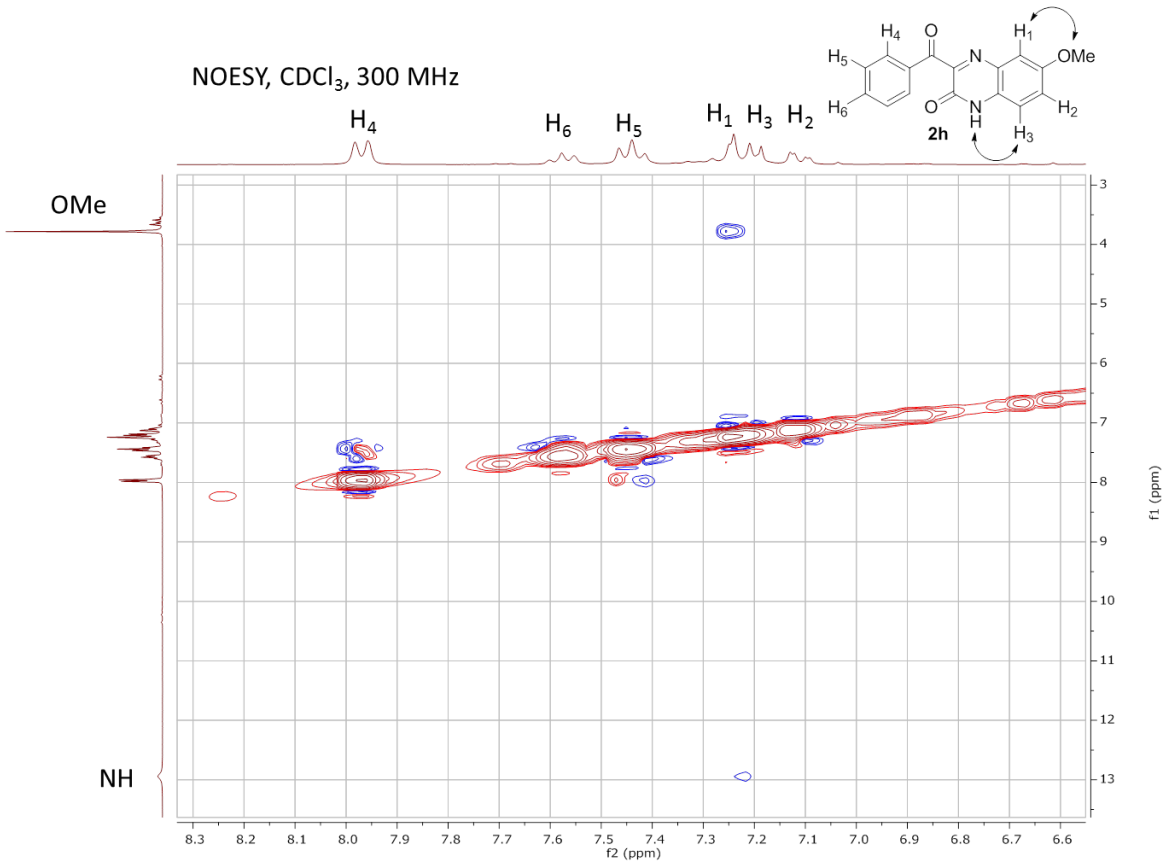

157.7
156.8
155.4
153.0
132.4
129.1
127.8
127.3
126.5
120.1
119.5
115.7
111.1
80.3
77.2
71.7
56.9
55.6
40.3
40.0
39.5
39.2
38.9
38.7
29.0
13C, DMSO-d6
200
188
175
163
150
138
125
113
100
88
75
63
50
38
25
13
0
ppm (t1)

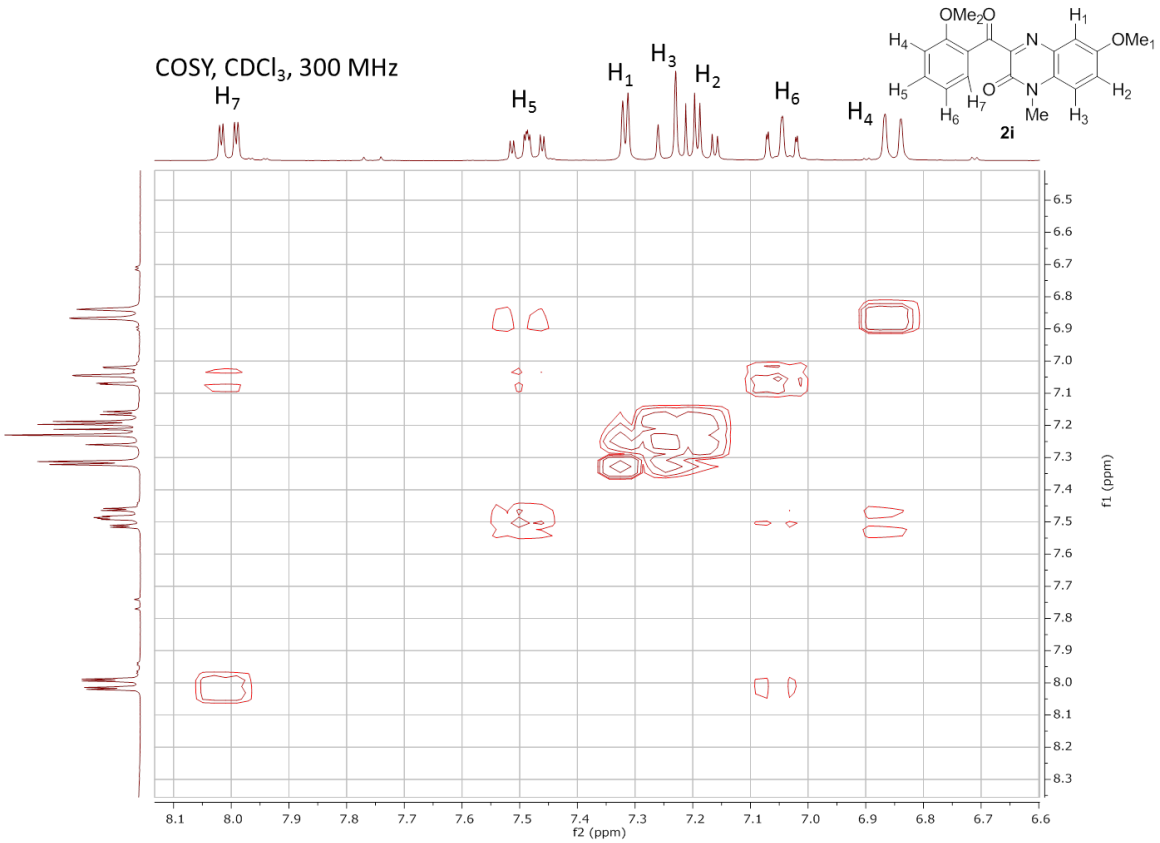

8.06
7.50
7.47
7.39
7.37
7.28
7.27
7.25
7.23
7.00
6.97
6.94
6.42
5.23
4.68
4.63
4.59
4.46
3.81
3.78
3.71
3.61
3.33
3.07
2.50
2.24
2.19
2.12
2.03
1.99
1.95
1.91
1.74
1.49
1.39
1.32
1.30
1.23
1.14
1.09
0.84
0.82
8
^{1}H, DMSO-d6
1.01
1.00
4.19
2.00
0.93
1.04
2.32
2.34
3.09
1.85
2.64
9.49
32.41
3.19
7.5
6.3
5.0
3.8
2.5
1.3
0.0
-1.3
ppm (t1)

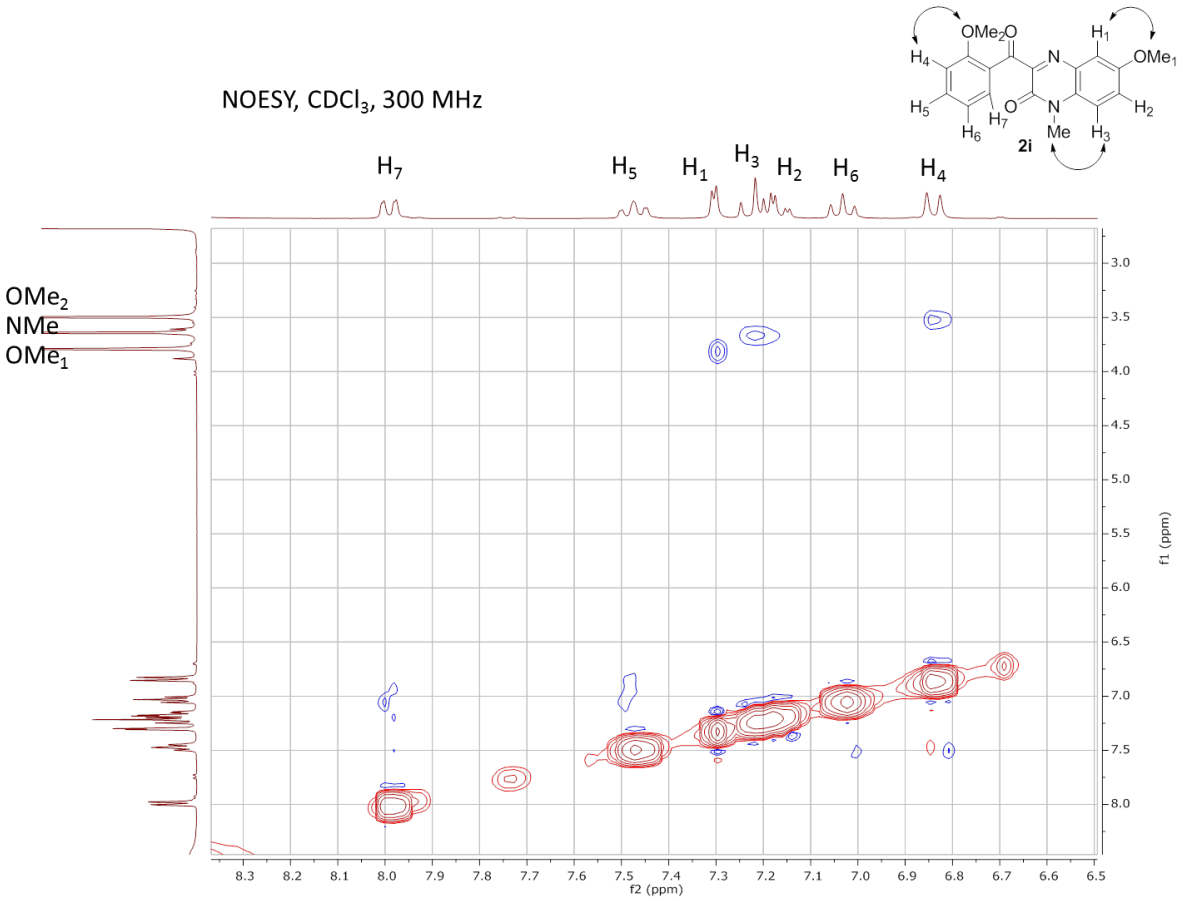

158.1
156.7
155.4
153.1
143.7
140.2
132.4
128.8
127.7
127.3
126.9
124.4
123.7
121.1
120.1
119.4
115.7
111.2
111.0
78.4
78.3
72.3
65.7
65.6
63.1
56.1
55.6
55.5
49.7
49.5
45.7
39.5
36.1
35.6
35.1
31.3
31.3
29.0
27.8
27.7
27.4
23.8
23.2
22.6
22.3
20.5
18.9
18.5
^{13}C, DMSO-d6
163
150
138
125
113
100
88
75
63
50
38
25
13
0
-13
ppm (t1)

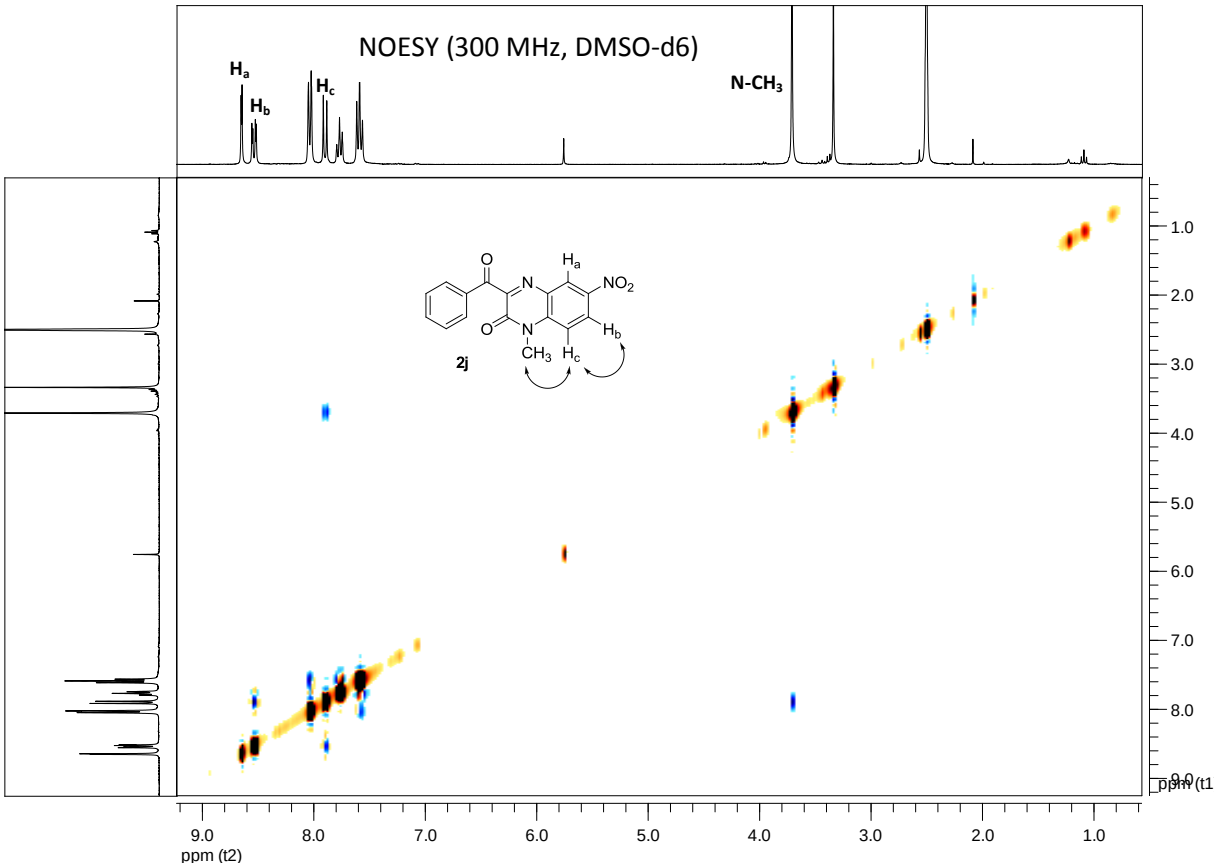
II. 2D-NMR experiments for compounds 2h-j



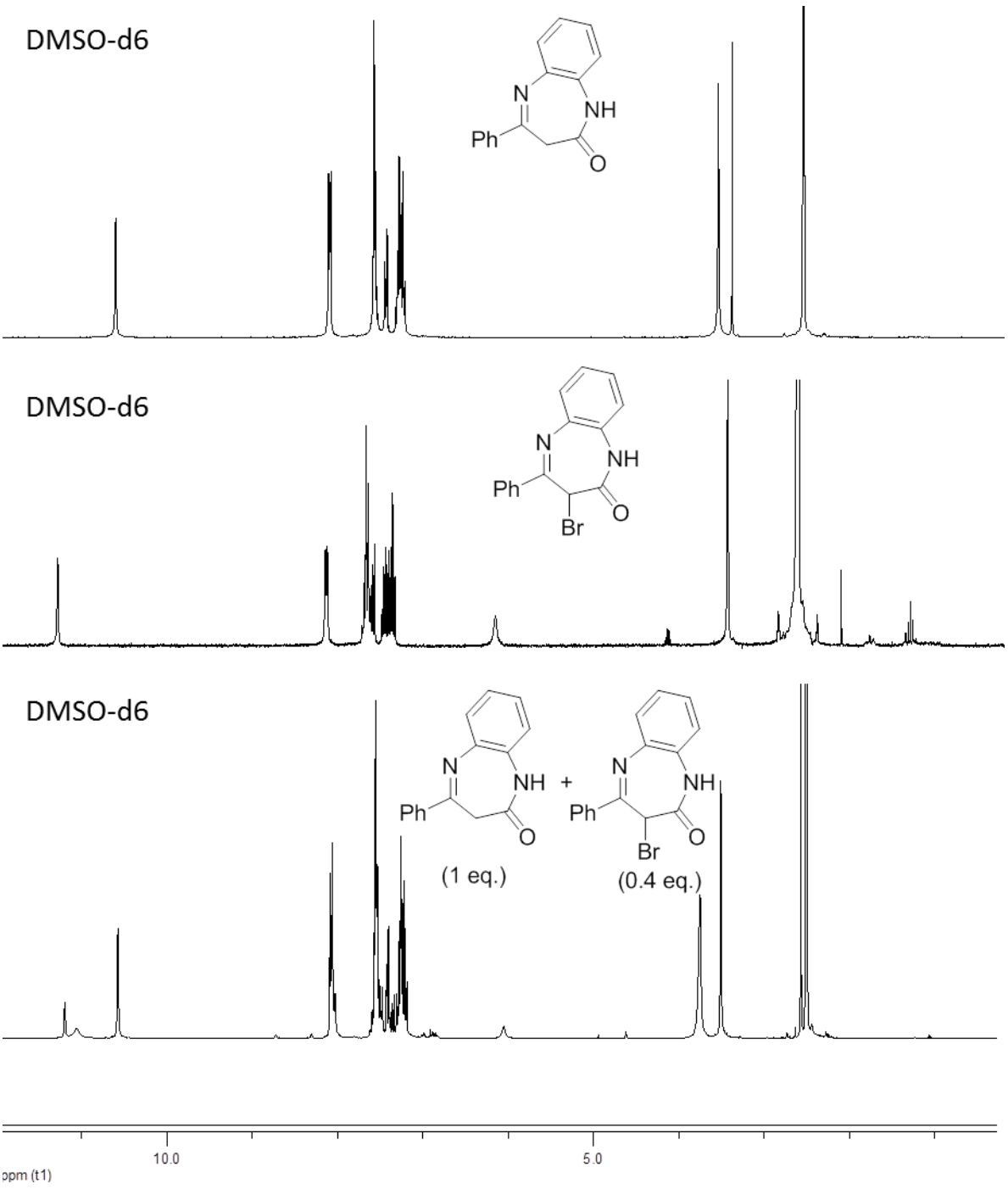



COSY, CDCl3, 300 MHz
H7
H5
H1
H3
H2
H6
H4
OMe2
H1
OMe1
H4
H5
H7
O
N
N
H2
H6
Me
H3
2i
f1 (ppm)
f2 (ppm)

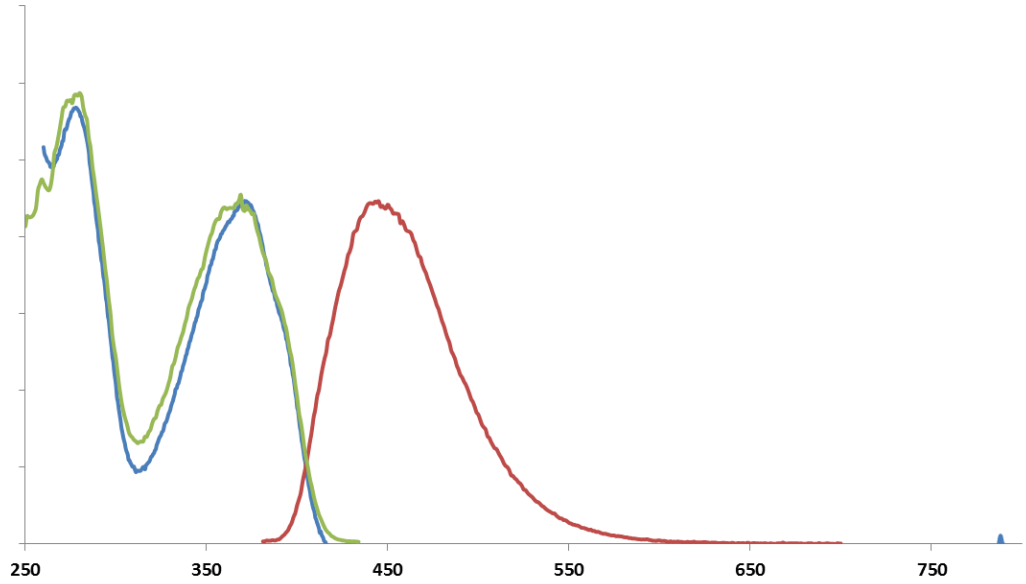

NOESY, CDCl3, 300 MHz
OMe2
H1
OMe1
H4
H5
H7
O
N
N
H2
H6
Me
H3
2i
H7
H5
H1
H3
H2
H6
H4
OMe2
NMe
OMe1
f1 (ppm)
f2 (ppm)


NOESY (300 MHz, DMSO-d6)
Ha
Hb
Hc
N-CH3
2j
ppm (t2)
ppm (t1

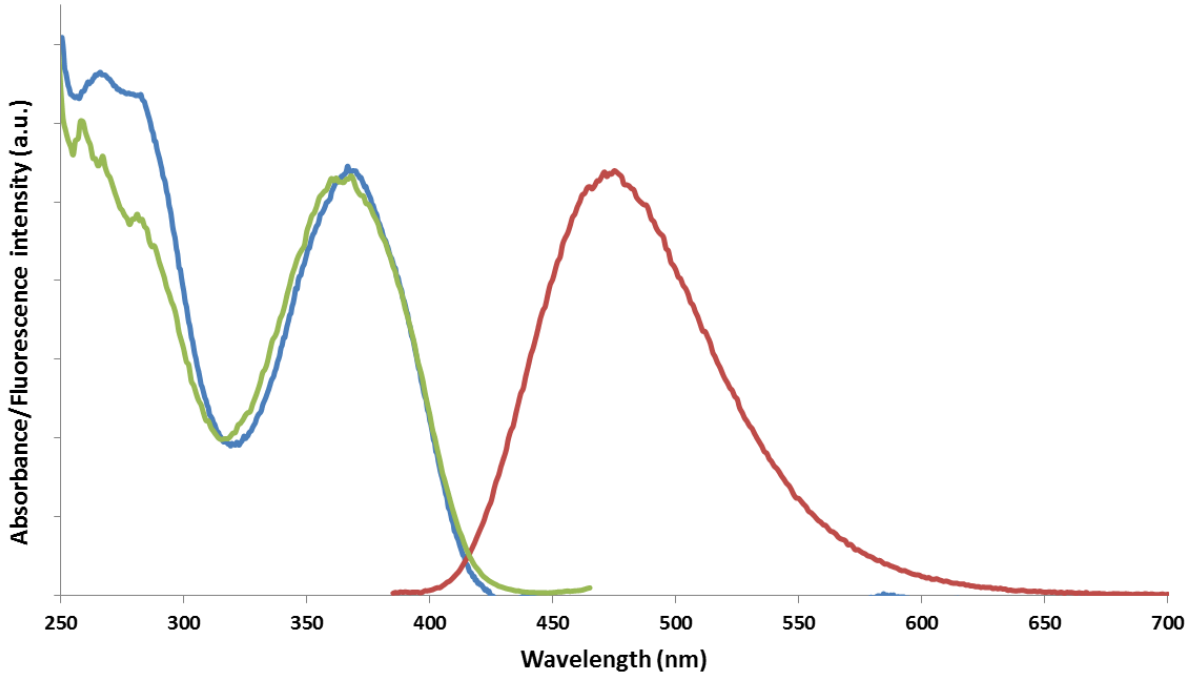
III. ^{1}H NMR study of the reaction between quinoxalinone 1a and NBS



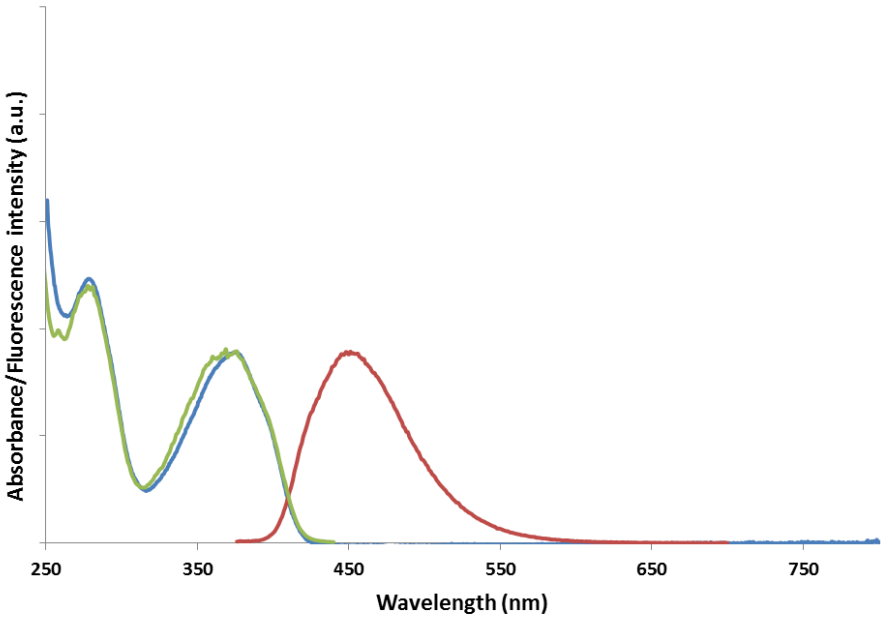
IV. Spectroscopic data for compounds 5 and 8



Normalized absorption (blue), emission (red), and excitation spectra (green)of quinoxalinone **5** in THF, at 25 °C.



Normalized absorption (blue), emission (red), and excitation spectra (green)of quinoxalinone **5** in PBS 7.4, at 25 °C.



Normalized absorption (blue), emission (red), and excitation spectra (green) of cholesterol conjugate **8** in THF, at 25 °C.

V. Crystallographic data for compound 2a

The crystal structure of 2a [$C_{15}H_{10}N_2O_2$] 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 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.

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 FO2. The program package WinGX3[3] was used for space group determination, structure solution and refinement.

DATA REFINEMENT

The non-standard space group $P2_1/n$ (n°14) was determined from systematic extinctions and relative Fo^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 2407 observed reflections and 172 variable parameters and converged with unweighted and weighted agreement factors of:

R1 = 0.0417, wR2 = 0.1096 for 2186 reflections with I>2σI and R1 = 0.0457, wR2 = 0.1131 for all data.

[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.

ANNEXE: CRYSTALLOGRAPHIC DATA

Table 1a: Atomic coordinates (x104) and equivalent isotropic displacement parameters ($Å^2$ x 10^3).

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
C(1)	10816(1)	1728(2)	7013(1)	33(1)
C(2)	11538(1)	170(3)	7454(1)	42(1)
C(3)	12031(1)	608(3)	8348(1)	48(1)
C(4)	11820(1)	2591(3)	8812(1)	50(1)
C(5)	11096(1)	4099(3)	8391(1)	46(1)
C(6)	10571(1)	3676(2)	7484(1)	35(1)
C(7)	9355(1)	4720(2)	6238(1)	35(1)
C(8)	9616(1)	2871(2)	5653(1)	34(1)
C(9)	8497(1)	6269(2)	5814(1)	37(1)
C(10)	7570(1)	5693(2)	6011(1)	35(1)
C(11)	7439(1)	3664(3)	6491(1)	45(1)
C(12)	6557(1)	3218(3)	6658(1)	57(1)
C(13)	5815(1)	4777(4)	6357(1)	63(1)
C(14)	5935(1)	6787(4)	5872(1)	66(1)
C(15)	6808(1)	7242(3)	5701(1)	49(1)
N(1)	9800(1)	5129(2)	7082(1)	39(1)
N(2)	10319(1)	1401(2)	6108(1)	37(1)
O(1)	8585(1)	7934(2)	5336(1)	64(1)
O(2)	9209(1)	2674(2)	4831(1)	42(1)

Table 1b: Hydrogen coordinates (x104) and equivalent isotropic displacement parameters (Å2 x 103).

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
H(2)	11686	-1156	7147	50
H(3)	12511	-435	8646	57
H(4)	12172	2894	9411	60
H(5)	10951	5410	8708	55
H(11)	7943	2604	6700	54
H(12)	6470	1853	6977	68
H(13)	5228	4480	6479	75
H(14)	5426	7833	5660	79
H(15)	6887	8600	5374	59
H(2A)	10465	197	5818	44

Table 2: Bond lengths (Å)

C(1)-N(2)	1.3812(16)
C(1)-C(2)	1.3916(19)
C(1)-C(6)	1.3977(19)
C(2)-C(3)	1.373(2)
C(2)-H(2)	0.93
C(3)-C(4)	1.390(2)
C(3)-H(3)	0.93
C(4)-C(5)	1.367(2)
C(4)-H(4)	0.93
C(5)-C(6)	1.4023(18)
C(5)-H(5)	0.93
C(6)-N(1)	1.3870(17)
C(7)-N(1)	1.2892(16)
C(7)-C(8)	1.4730(18)
C(7)-C(9)	1.5130(18)
C(8)-O(2)	1.2319(15)
C(8)-N(2)	1.3496(17)
C(9)-O(1)	1.2072(17)
C(9)-C(10)	1.4822(18)
C(10)-C(15)	1.3860(19)
C(10)-C(11)	1.3876(19)
C(11)-C(12)	1.386(2)
C(11)-H(11)	0.93
C(12)-C(13)	1.367(3)
C(12)-H(12)	0.93
C(13)-C(14)	1.379(3)
C(13)-H(13)	0.93
C(14)-C(15)	1.376(2)
C(14)-H(14)	0.93
C(15)-H(15)	0.93
N(2)-H(2A)	0.86

Table 3: Angles (°)

N(2)-C(1)-C(2)	121.53(12)	O(1)-C(9)-C(7)	119.69(12)
N(2)-C(1)-C(6)	118.07(12)	C(10)-C(9)-C(7)	118.34(11)
C(2)-C(1)-C(6)	120.40(12)	C(15)-C(10)-C(11)	119.12(13)
C(3)-C(2)-C(1)	119.36(13)	C(15)-C(10)-C(9)	118.49(12)
C(3)-C(2)-H(2)	120.3	C(11)-C(10)-C(9)	122.39(12)
C(1)-C(2)-H(2)	120.3	C(12)-C(11)-C(10)	119.95(14)
C(2)-C(3)-C(4)	120.81(14)	C(12)-C(11)-H(11)	120
C(2)-C(3)-H(3)	119.6	C(10)-C(11)-H(11)	120
C(4)-C(3)-H(3)	119.6	C(13)-C(12)-C(11)	120.26(16)
C(5)-C(4)-C(3)	120.19(13)	C(13)-C(12)-H(12)	119.9
C(5)-C(4)-H(4)	119.9	C(11)-C(12)-H(12)	119.9
C(3)-C(4)-H(4)	119.9	C(12)-C(13)-C(14)	120.25(15)
C(4)-C(5)-C(6)	120.25(14)	C(12)-C(13)-H(13)	119.9
C(4)-C(5)-H(5)	119.9	C(14)-C(13)-H(13)	119.9
C(6)-C(5)-H(5)	119.9	C(15)-C(14)-C(13)	119.89(16)
N(1)-C(6)-C(1)	121.22(11)	C(15)-C(14)-H(14)	120.1
N(1)-C(6)-C(5)	119.82(12)	C(13)-C(14)-H(14)	120.1
C(1)-C(6)-C(5)	118.91(12)	C(14)-C(15)-C(10)	120.53(15)
N(1)-C(7)-C(8)	125.20(12)	C(14)-C(15)-H(15)	119.7
N(1)-C(7)-C(9)	117.39(12)	C(10)-C(15)-H(15)	119.7
C(8)-C(7)-C(9)	117.41(11)	C(7)-N(1)-C(6)	117.78(11)
O(2)-C(8)-N(2)	123.79(12)	C(8)-N(2)-C(1)	123.46(11)
O(2)-C(8)-C(7)	122.46(12)	C(8)-N(2)-H(2A)	118.3
N(2)-C(8)-C(7)	113.74(11)	C(1)-N(2)-H(2A)	118.3
O(1)-C(9)-C(10)	121.96(12)		

Table 4: Anisotropic displacement parameters (Å² 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)	31(1)	37(1)	33(1)	-2(1)	8(1)	-2(1)
C(2)	39(1)	42(1)	44(1)	-2(1)	9(1)	5(1)
C(3)	40(1)	53(1)	45(1)	6(1)	3(1)	7(1)
C(4)	48(1)	61(1)	35(1)	-3(1)	0(1)	0(1)
C(5)	50(1)	49(1)	36(1)	-8(1)	8(1)	2(1)
C(6)	34(1)	38(1)	34(1)	-2(1)	9(1)	0(1)
C(7)	33(1)	35(1)	36(1)	0(1)	10(1)	0(1)
C(8)	31(1)	34(1)	35(1)	-2(1)	7(1)	-3(1)
C(9)	39(1)	35(1)	37(1)	1(1)	10(1)	2(1)
C(10)	35(1)	36(1)	32(1)	-2(1)	7(1)	0(1)
C(11)	44(1)	43(1)	47(1)	7(1)	9(1)	1(1)
C(12)	56(1)	59(1)	58(1)	11(1)	20(1)	-12(1)
C(13)	42(1)	76(1)	75(1)	-1(1)	24(1)	-7(1)
C(14)	41(1)	69(1)	90(1)	13(1)	21(1)	13(1)
C(15)	43(1)	47(1)	58(1)	12(1)	14(1)	6(1)
N(1)	40(1)	42(1)	37(1)	-5(1)	10(1)	5(1)
N(2)	38(1)	36(1)	34(1)	-7(1)	7(1)	4(1)
O(1)	49(1)	61(1)	88(1)	34(1)	27(1)	9(1)
O(2)	45(1)	42(1)	34(1)	-5(1)	1(1)	2(1)

Table 5: Torsion angles (°)

N(2)-C(1)-C(2)-C(3)	178.16(13)
C(6)-C(1)-C(2)-C(3)	-2.2(2)
C(1)-C(2)-C(3)-C(4)	-0.4(2)
C(2)-C(3)-C(4)-C(5)	2.0(2)
C(3)-C(4)-C(5)-C(6)	-1.0(2)
N(2)-C(1)-C(6)-N(1)	5.28(19)
C(2)-C(1)-C(6)-N(1)	-174.38(12)
N(2)-C(1)-C(6)-C(5)	-177.20(12)
C(2)-C(1)-C(6)-C(5)	3.1(2)
C(4)-C(5)-C(6)-N(1)	176.02(14)
C(4)-C(5)-C(6)-C(1)	-1.5(2)
N(1)-C(7)-C(8)-O(2)	-174.35(13)
C(9)-C(7)-C(8)-O(2)	5.41(19)
N(1)-C(7)-C(8)-N(2)	7.11(19)
C(9)-C(7)-C(8)-N(2)	-173.13(11)
N(1)-C(7)-C(9)-O(1)	99.06(17)
C(8)-C(7)-C(9)-O(1)	-80.72(17)
N(1)-C(7)-C(9)-C(10)	-80.36(16)
C(8)-C(7)-C(9)-C(10)	99.86(14)
O(1)-C(9)-C(10)-C(15)	-5.8(2)
C(7)-C(9)-C(10)-C(15)	173.64(13)
O(1)-C(9)-C(10)-C(11)	174.07(15)
C(7)-C(9)-C(10)-C(11)	-6.53(19)
C(15)-C(10)-C(11)-C(12)	-0.4(2)
C(9)-C(10)-C(11)-C(12)	179.77(14)
C(10)-C(11)-C(12)-C(13)	-0.3(3)
C(11)-C(12)-C(13)-C(14)	1.0(3)
C(12)-C(13)-C(14)-C(15)	-0.8(3)
C(13)-C(14)-C(15)-C(10)	0.1(3)
C(11)-C(10)-C(15)-C(14)	0.5(2)
C(9)-C(10)-C(15)-C(14)	-179.65(15)
C(8)-C(7)-N(1)-C(6)	-2.0(2)
C(9)-C(7)-N(1)-C(6)	178.28(11)
C(1)-C(6)-N(1)-C(7)	-4.44(19)
C(5)-C(6)-N(1)-C(7)	178.06(13)
O(2)-C(8)-N(2)-C(1)	175.40(12)
C(7)-C(8)-N(2)-C(1)	-6.08(18)
C(2)-C(1)-N(2)-C(8)	-179.85(13)
C(6)-C(1)-N(2)-C(8)	0.49(19)

Table 6: Calculated reflections from PowderCell*

h	k	l	2θ/°	d/Å	I/rel.	|F(hkl)|
-1	0	1	7.54	11.71	96.77	48.66
1	0	1	9.84	8.98	100.00	64.63
2	0	0	12.67	6.98	11.41	28.19
-2	0	2	15.12	5.86	2.33	15.26
0	1	1	16.93	5.23	61.42	62.12
-1	1	1	17.52	5.06	26.96	42.64
-1	0	3	17.70	5.01	23.08	56.39
-3	0	1	18.43	4.81	41.48	78.80
1	1	1	18.63	4.76	47.72	60.45
0	1	2	19.96	4.44	6.07	23.14
-1	1	2	19.97	4.44	72.17	79.84
-2	1	1	20.22	4.39	11.59	32.41
2	1	0	20.29	4.37	29.58	51.97
1	0	3	20.85	4.26	86.15	129.03
3	0	1	21.48	4.13	5.50	33.61
1	1	2	21.91	4.05	24.67	51.40
-2	1	2	21.93	4.05	79.70	92.44
-3	0	3	22.76	3.90	66.15	123.83
-1	1	3	23.80	3.74	7.55	31.01
0	1	3	24.23	3.67	21.43	53.19
0	0	4	24.42	3.64	14.95	63.39
3	1	0	24.84	3.58	12.86	42.30
-2	1	3	25.07	3.55	5.76	28.59
-3	1	2	25.41	3.50	72.53	102.89
4	0	0	25.49	3.49	2.80	28.71
1	1	3	26.26	3.39	23.12	60.16
-3	1	3	27.82	3.20	12.68	47.35
0	1	4	29.22	3.05	26.88	72.63
-2	1	4	29.23	3.05	12.67	49.90
-4	1	1	29.38	3.04	4.44	29.69
3	0	3	29.81	2.99	2.37	31.18
-4	1	2	29.92	2.98	39.17	89.93

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.