CHEMISTRY
A EUROPEAN JOURNAL

Supporting Information

1,2,3-Triazolo[4,5,-*e*]furazano[3,4,-*b*]pyrazine 6-Oxide—A Fused Heterocycle with a Roving Hydrogen Forms a New Class of Insensitive Energetic Materials

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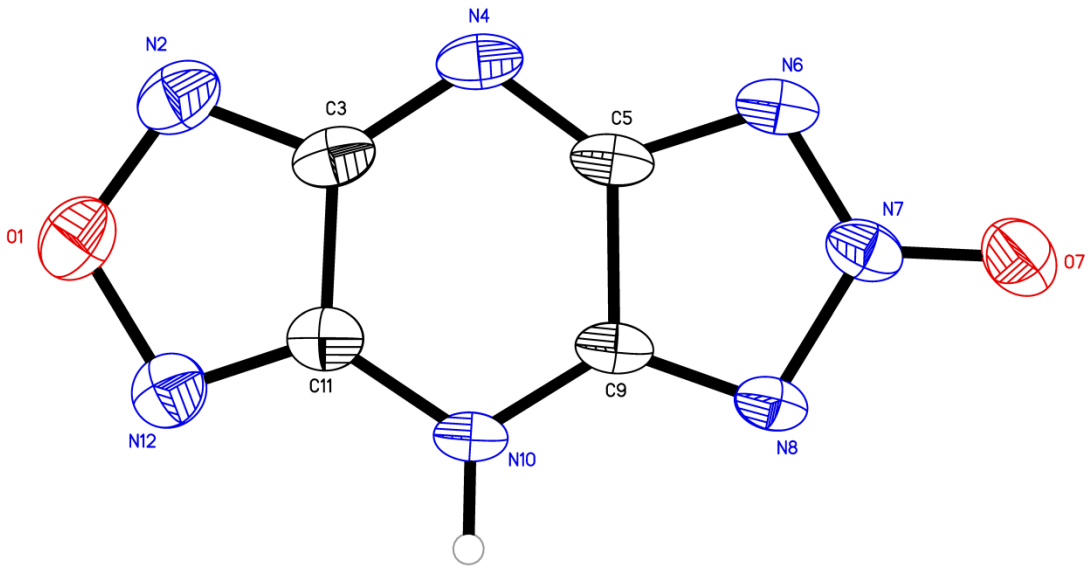
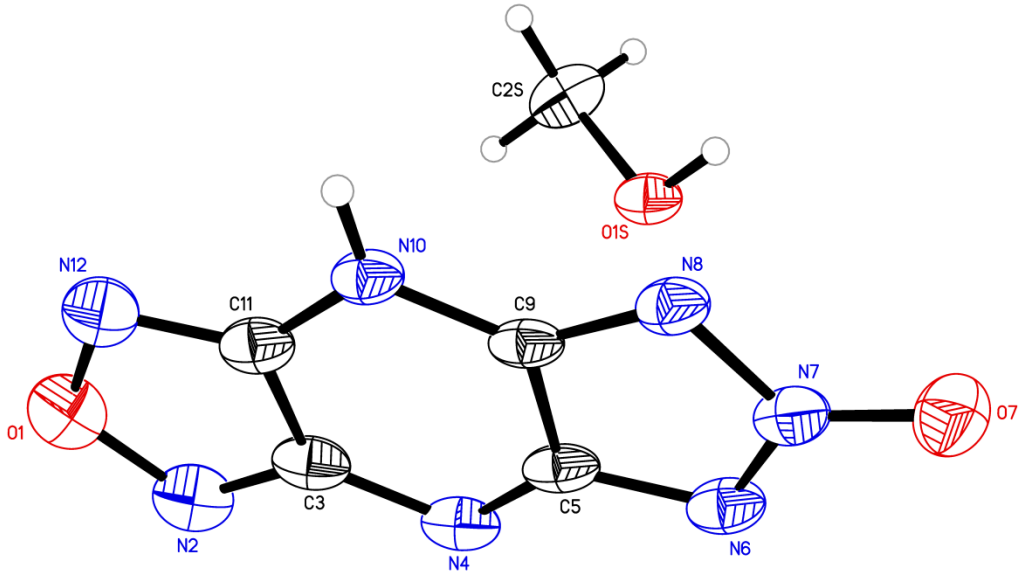
Single-crystal X-ray Diffraction Analysis of **5**.

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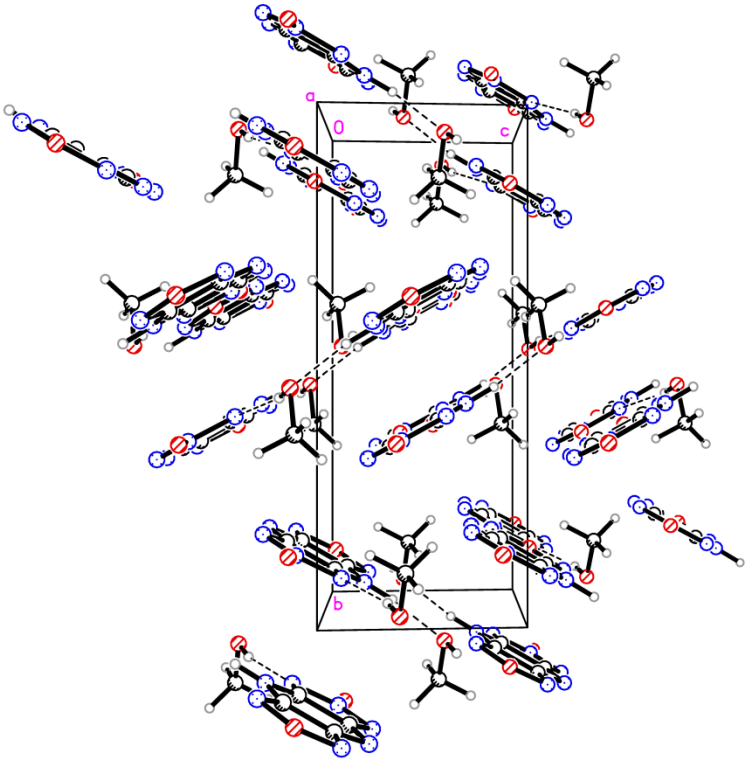
N6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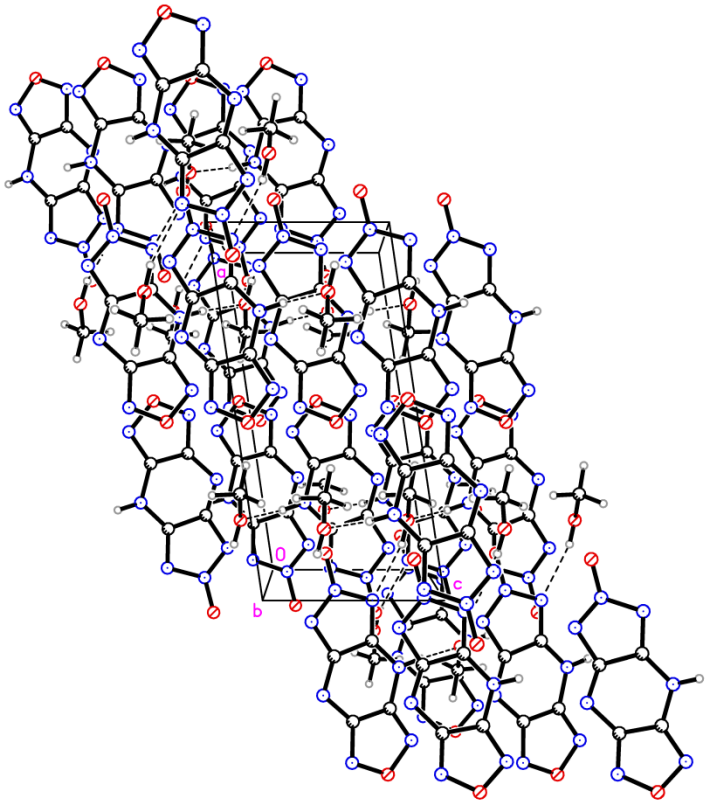
c
b


O
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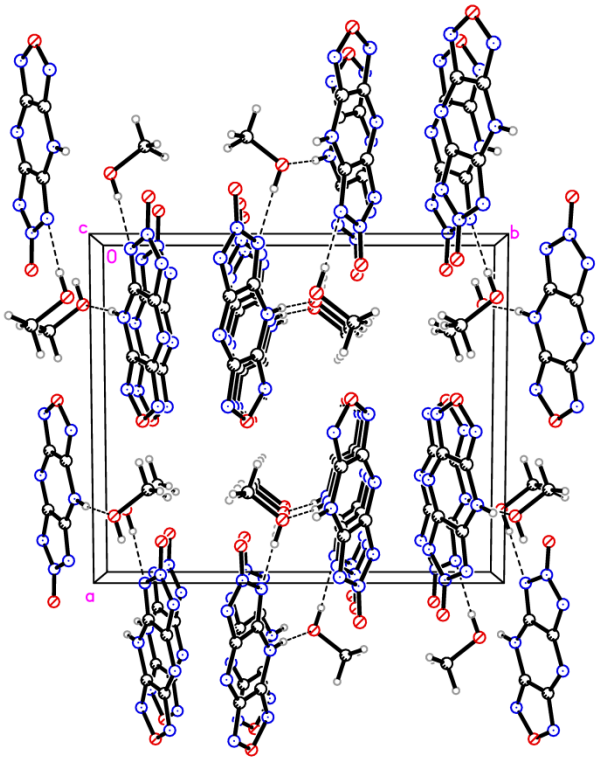
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b
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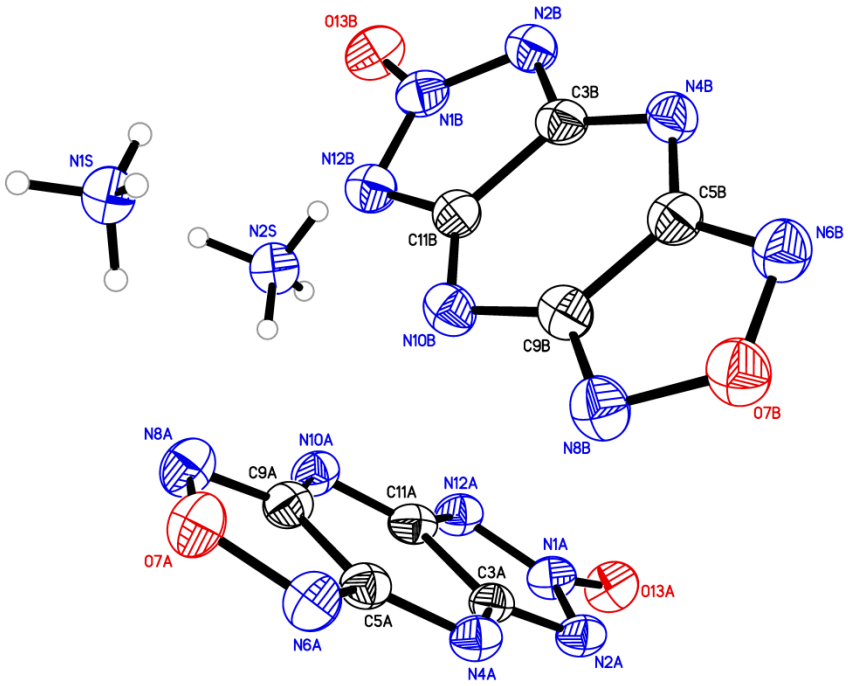
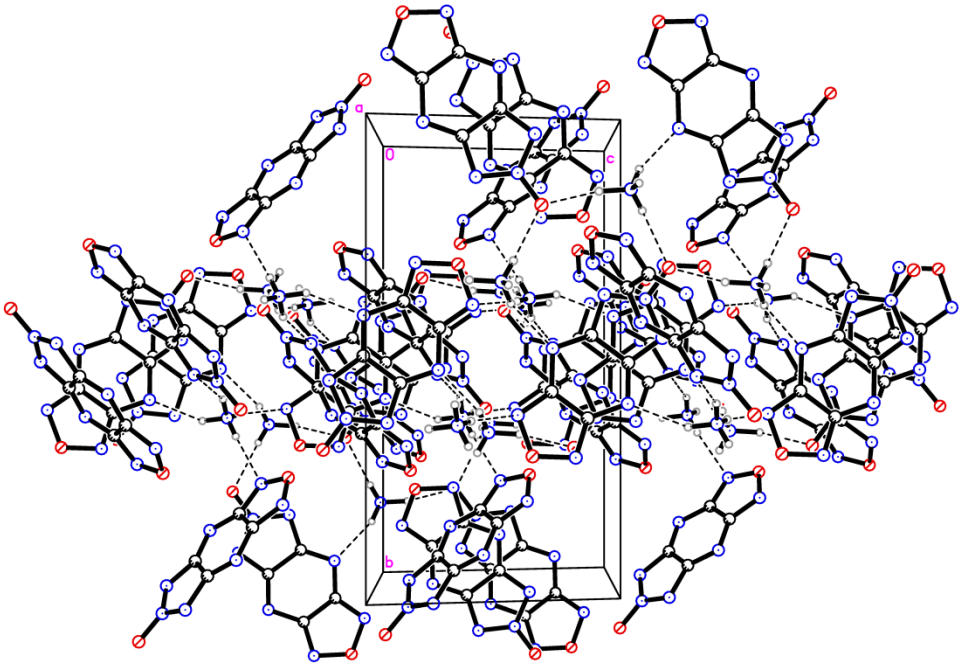
Single-crystal X-ray Diffraction Analysis of **6**

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c
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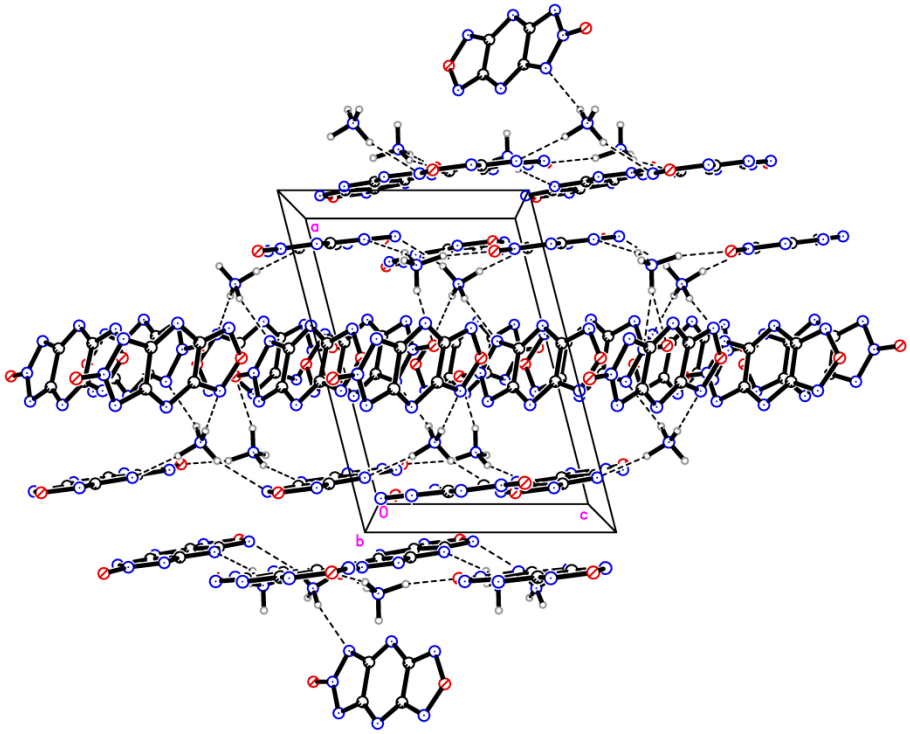
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c
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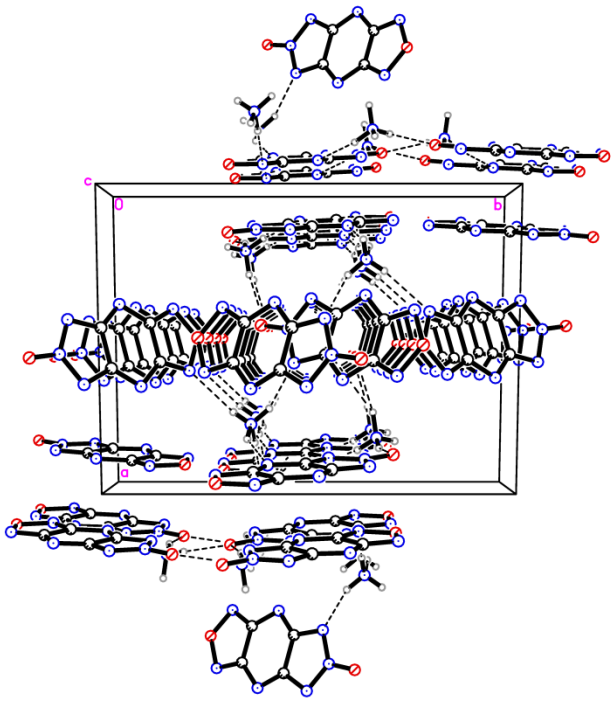
c
0
b
a

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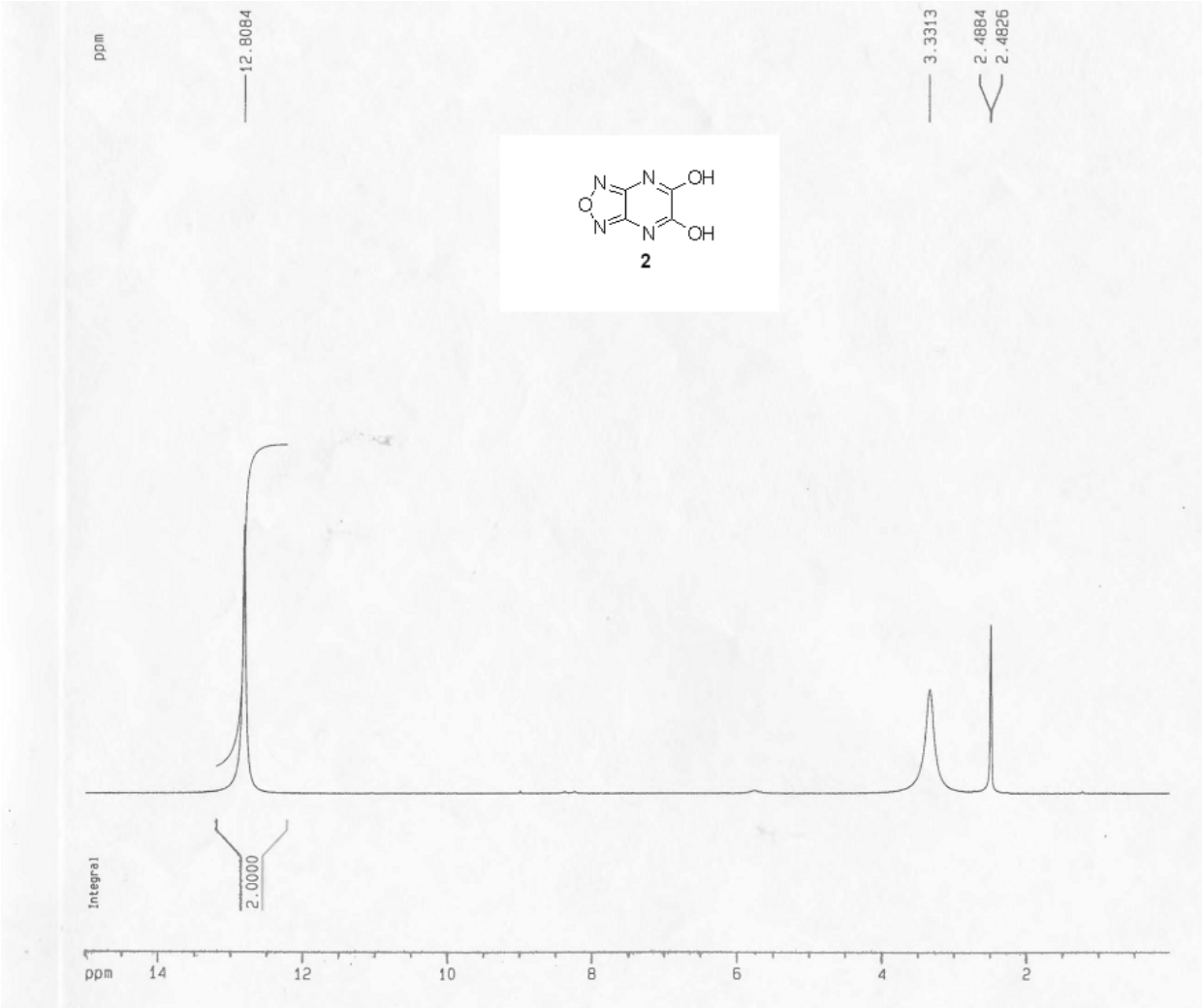
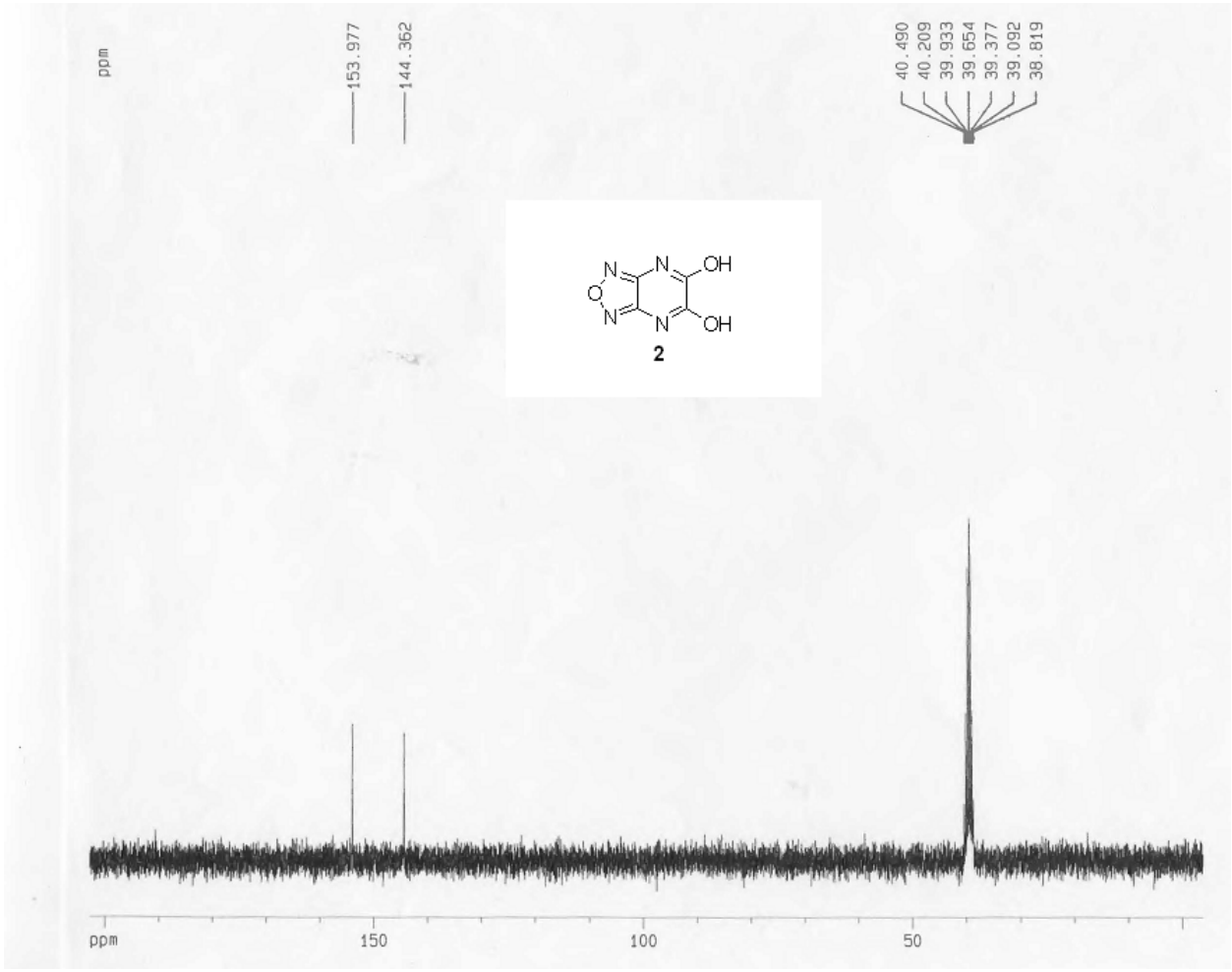
^{1}H, ^{13}C, ^{15}N NMR and IR Spectra and DSC of compounds **2**-**11**.

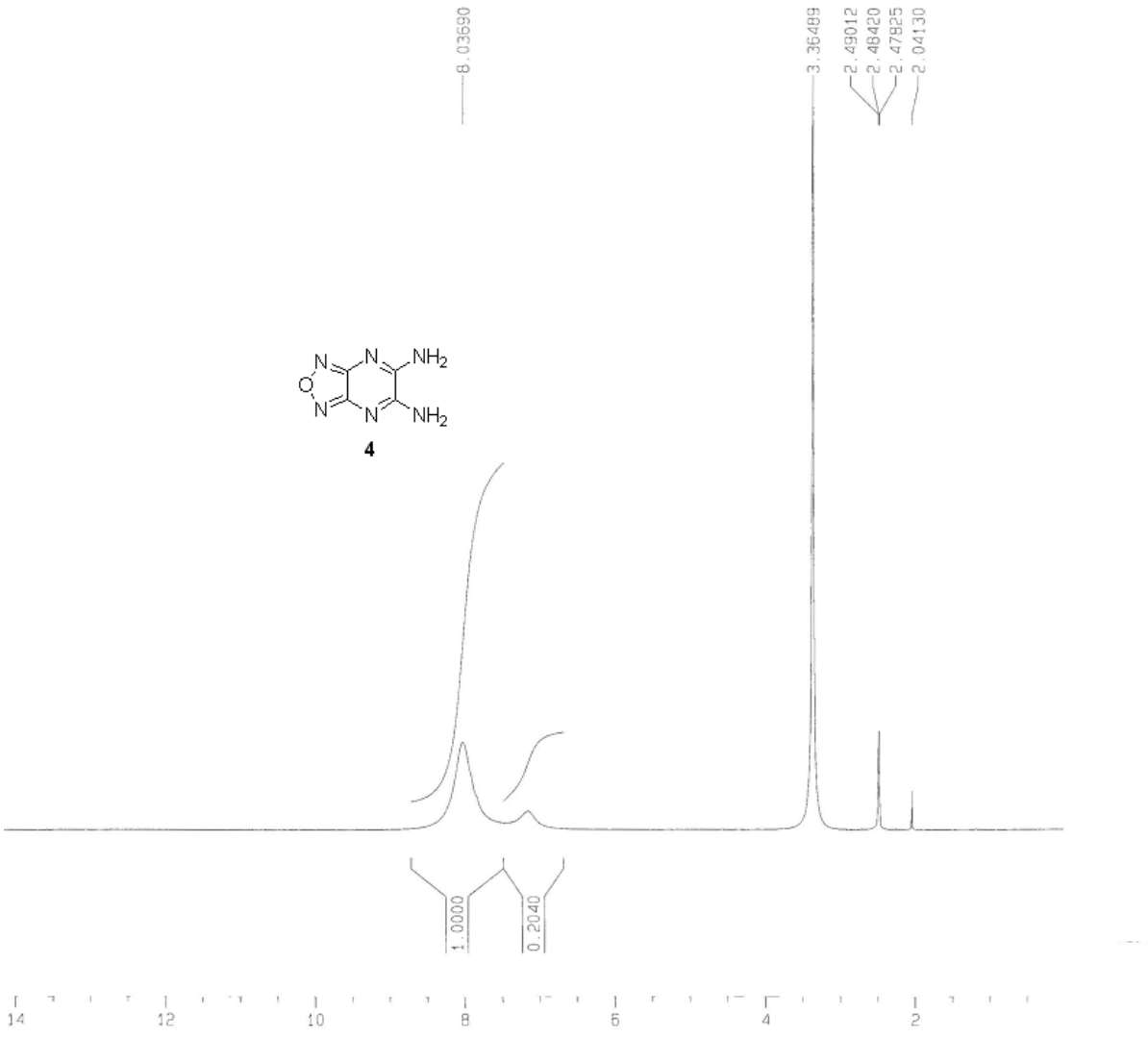
^{1}H NMR in [D_6]DMSO



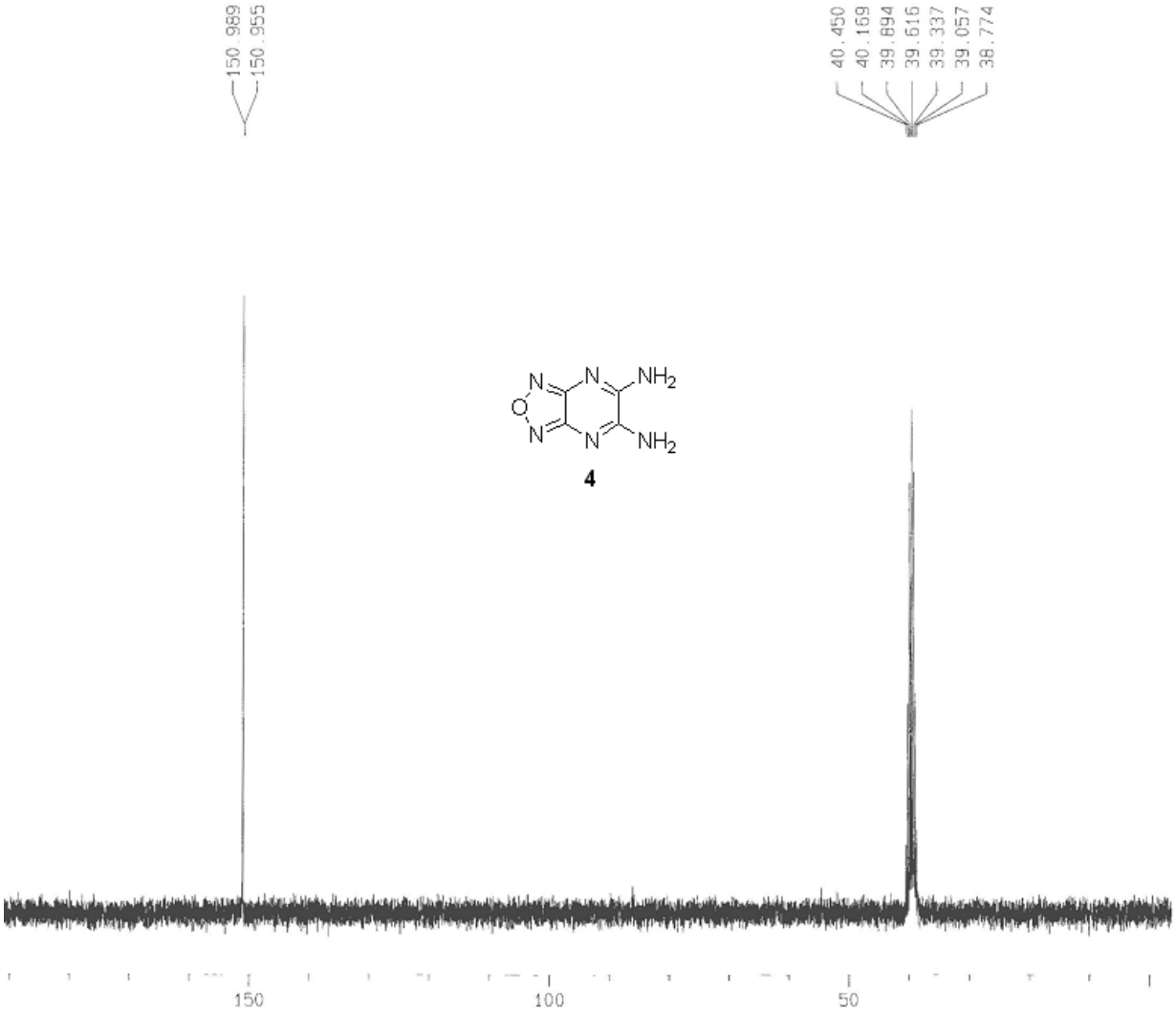
^{13}C NMR in [D$_6$]DMSO



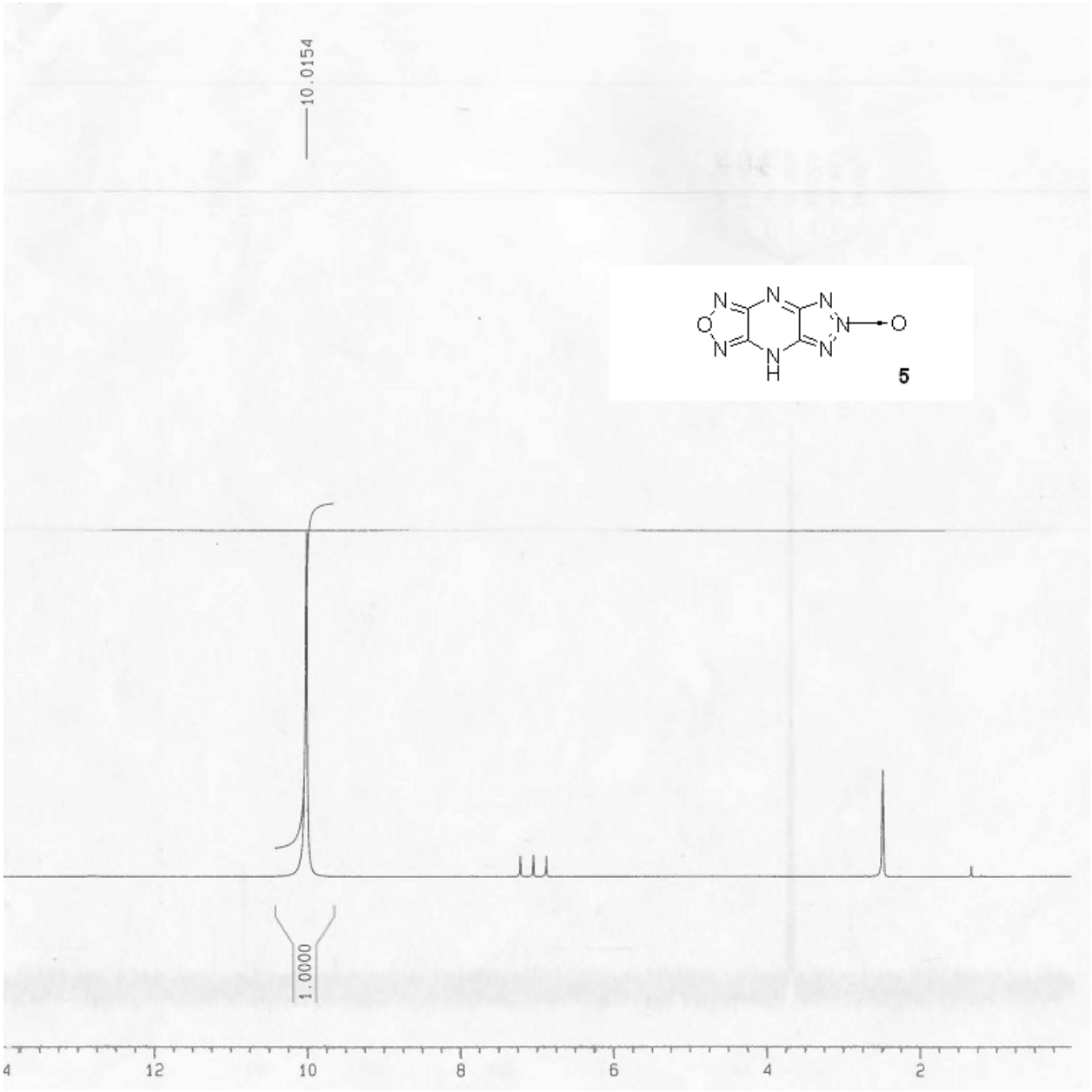
^{1}H NMR in [D$_6$]DMSO



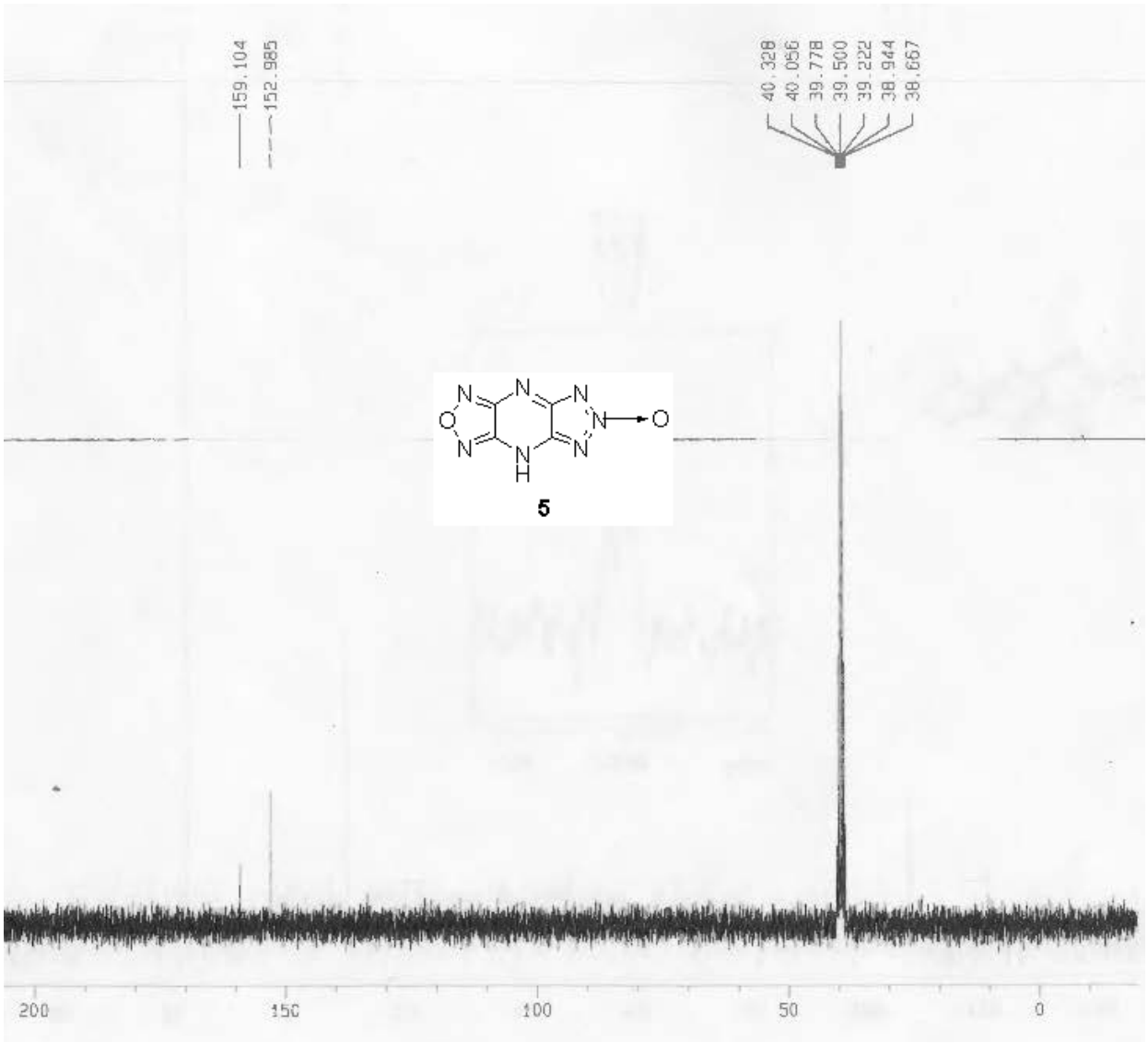
^{13}C NMR in [D$_6$]DMSO



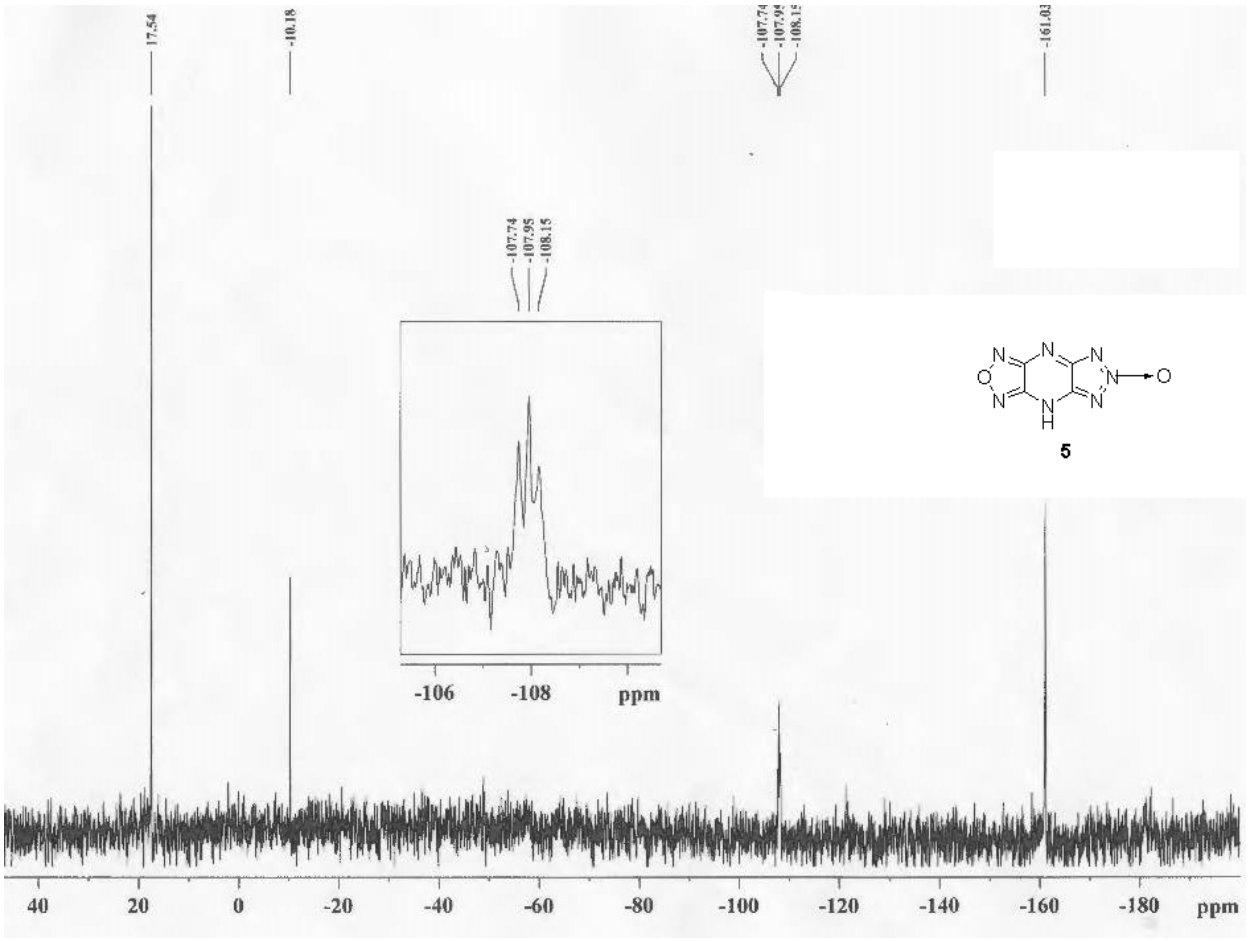
^{1}H NMR in [D$_6$]DMSO



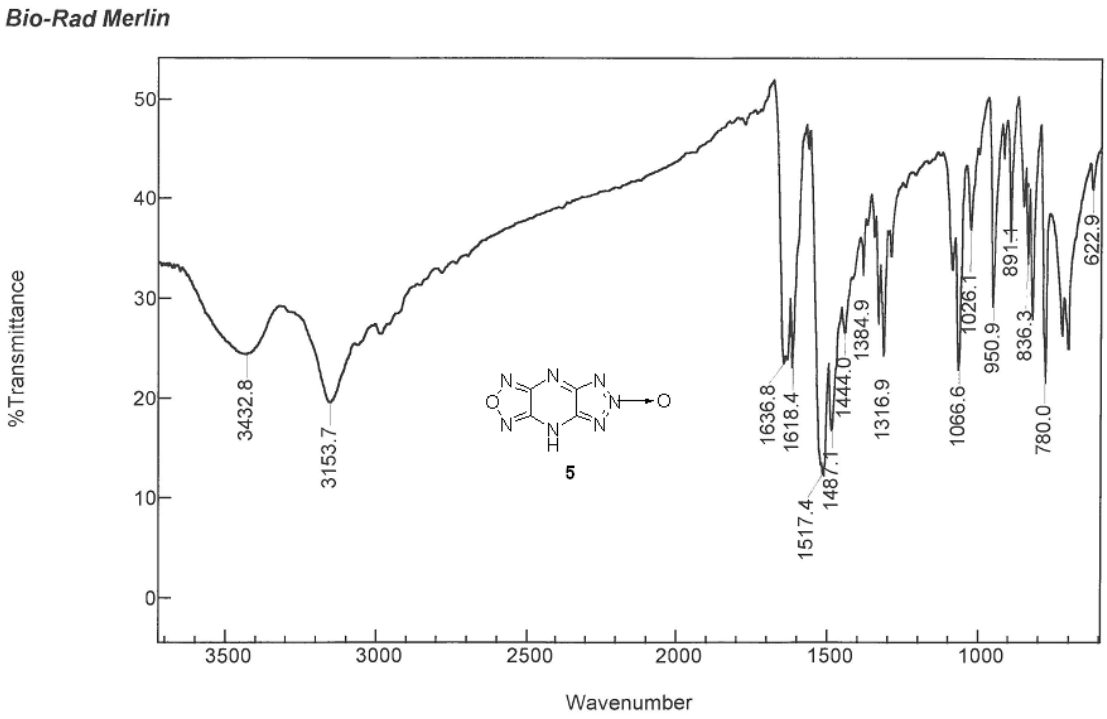
^{13}C NMR in [D$_6$]DMSO



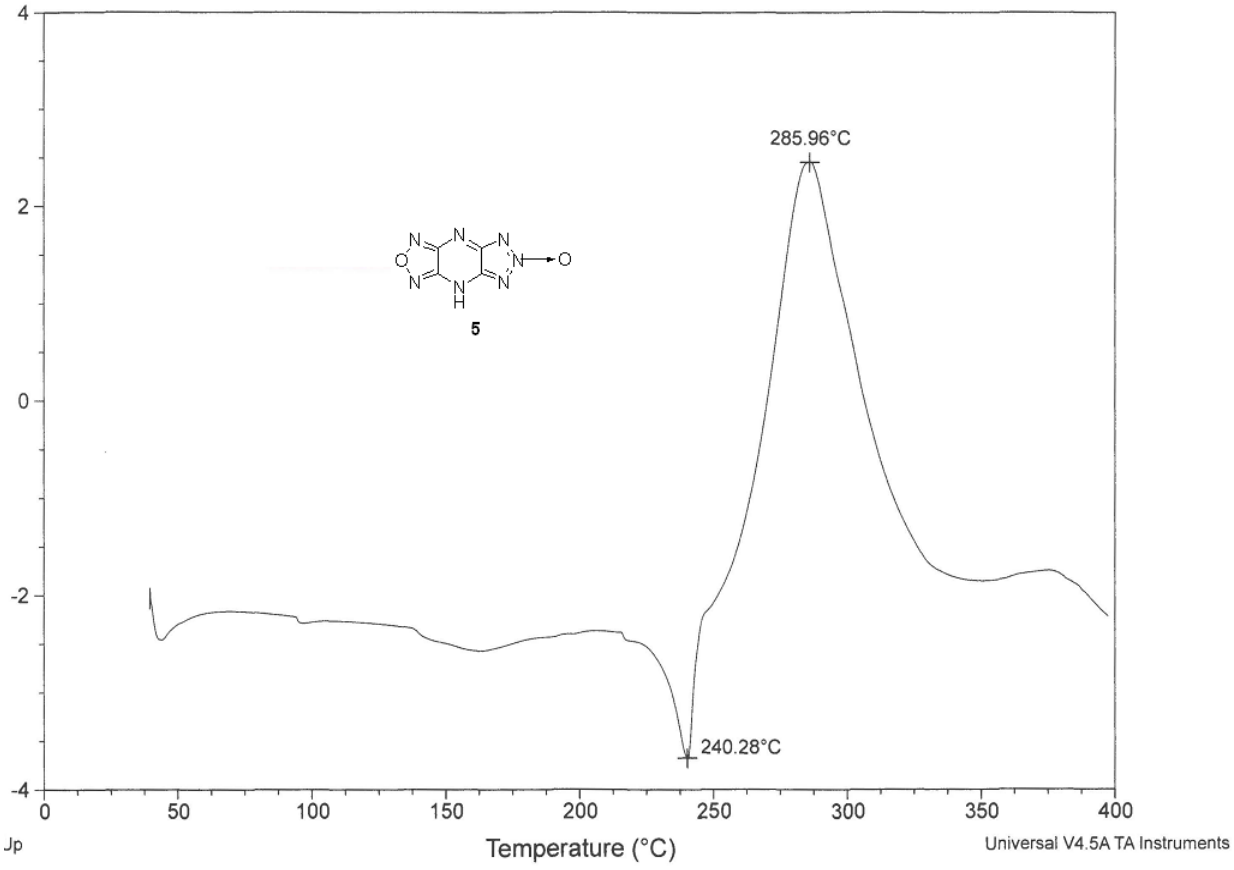
^{15}N NMR in [D$_6$]DMSO



IR Spectrum


Bio-Rad Merlin
%Transmittance
Wavenumber
3500
3000
2500
2000
1500
1000
50
40
30
20
10
0
3432.8
3153.7
1636.8
1618.4
1517.4
1487.1
1444.0
1384.9
1316.9
1066.6
1026.1
950.9
891.1
836.3
780.0
622.9
5

DSC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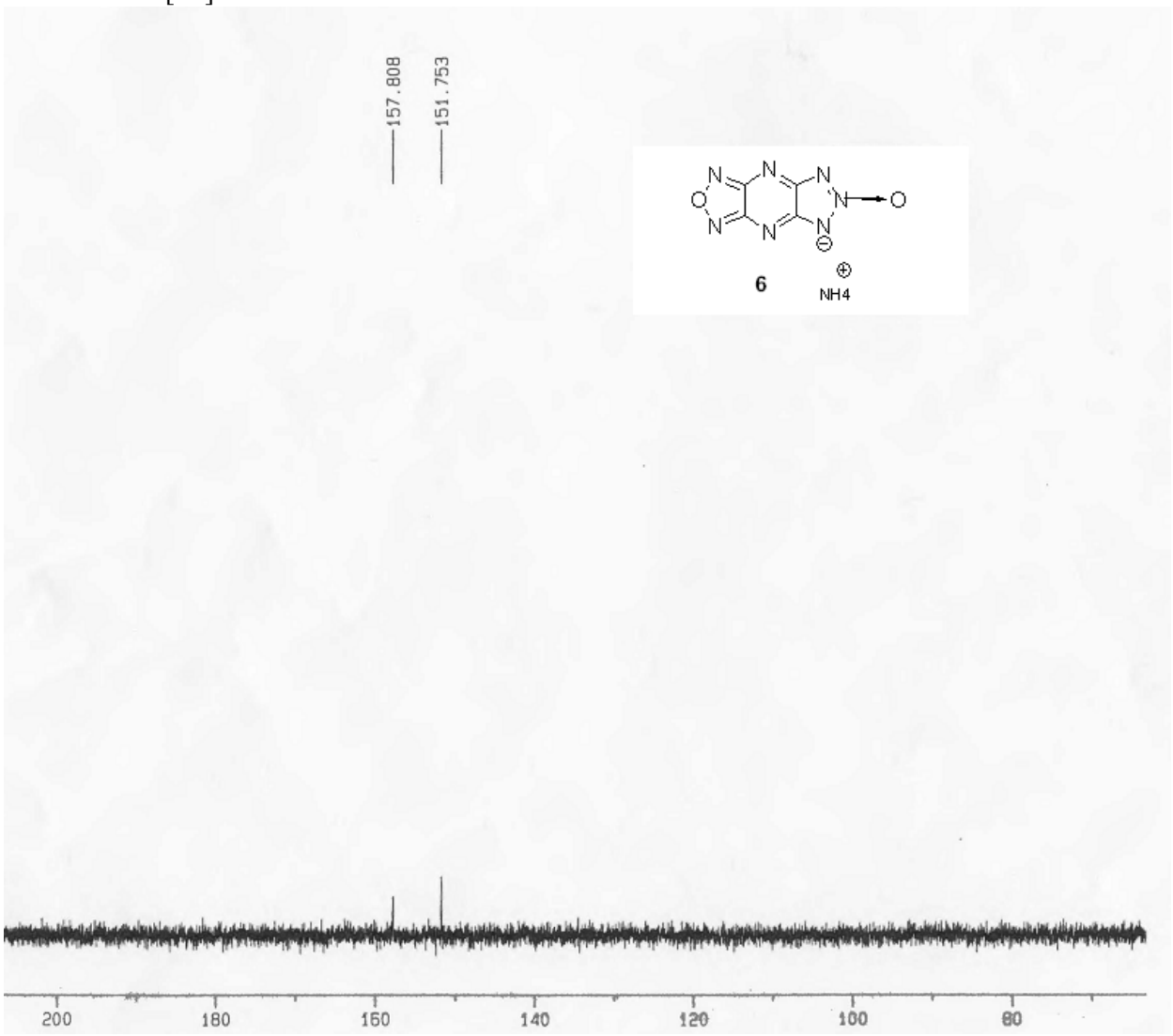
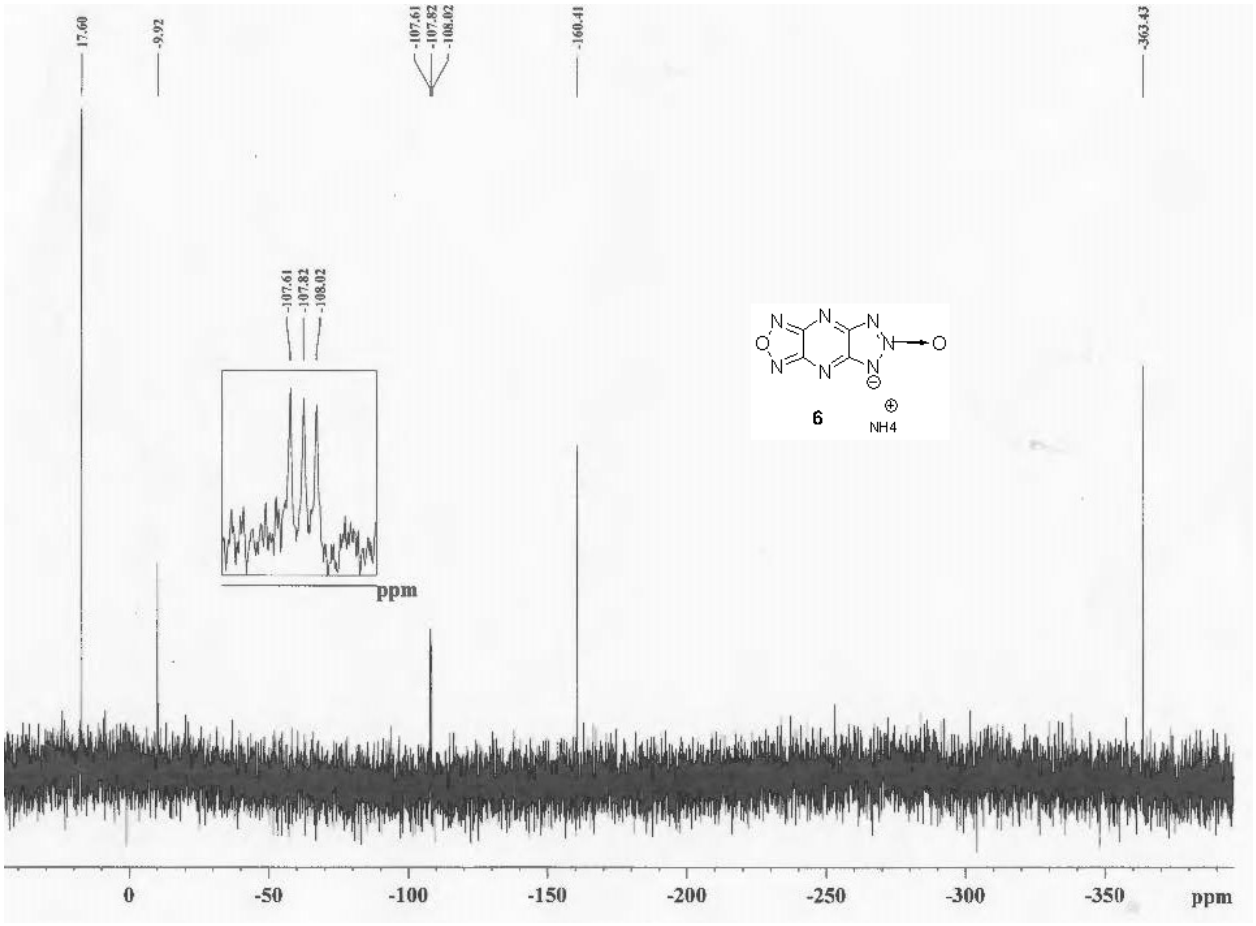
240.28°C
5
Jp
Temperature (°C)
Universal V4.5A TA Instruments

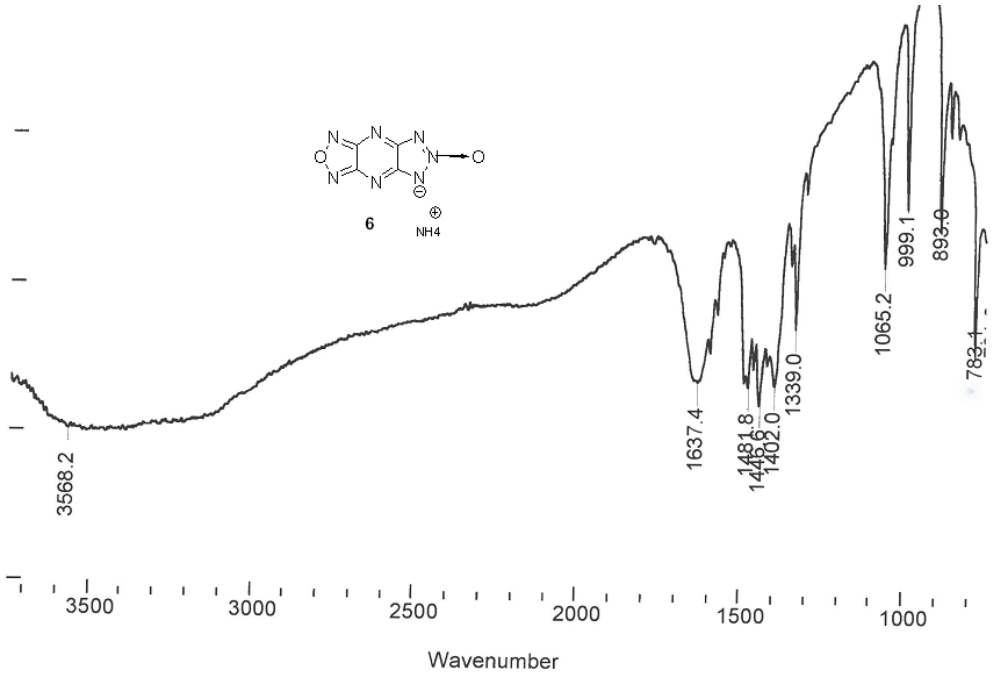
^{13}C NMR in [D$_6$]DMSO

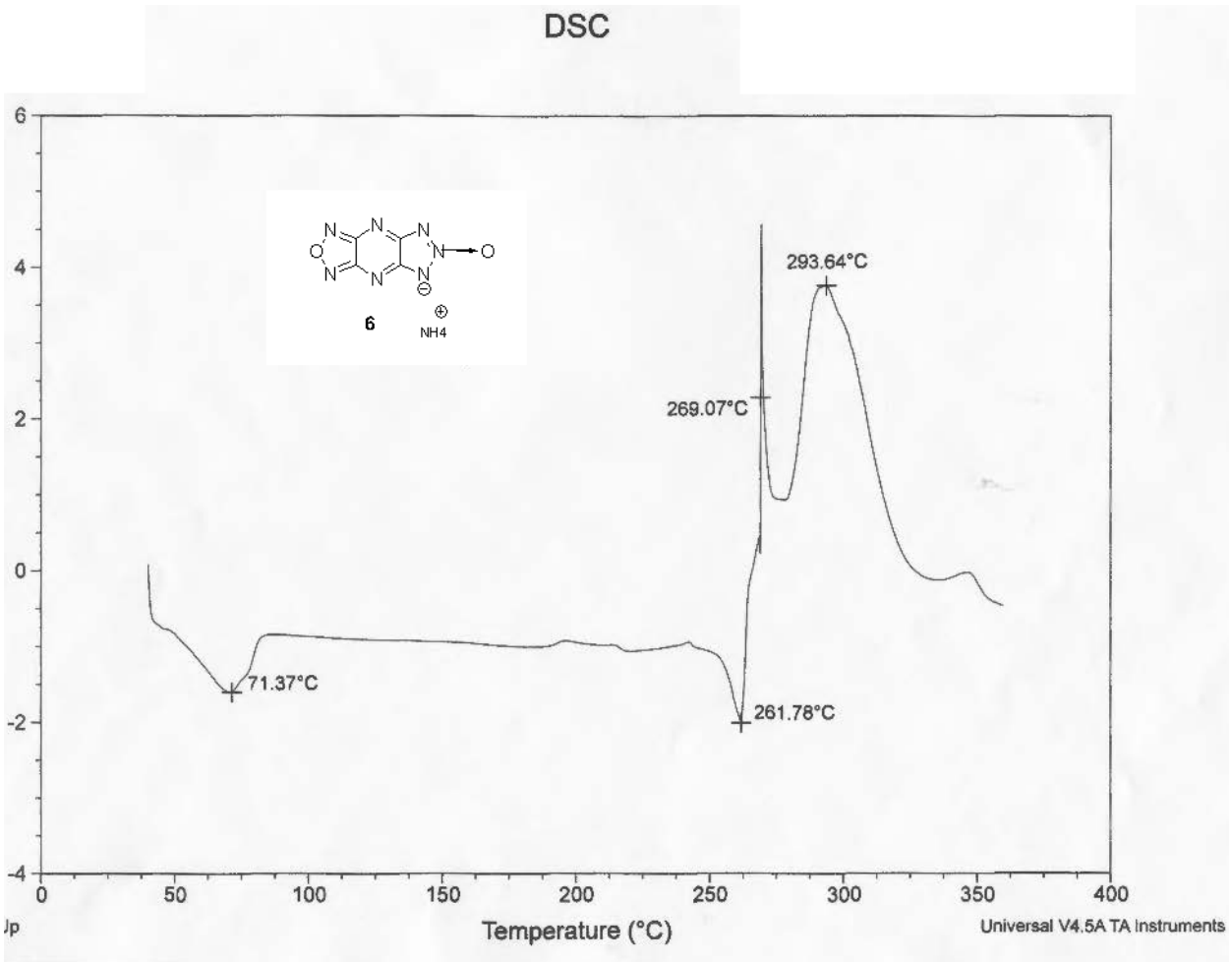


^{15}N NMR in [D$_6$]DMSO

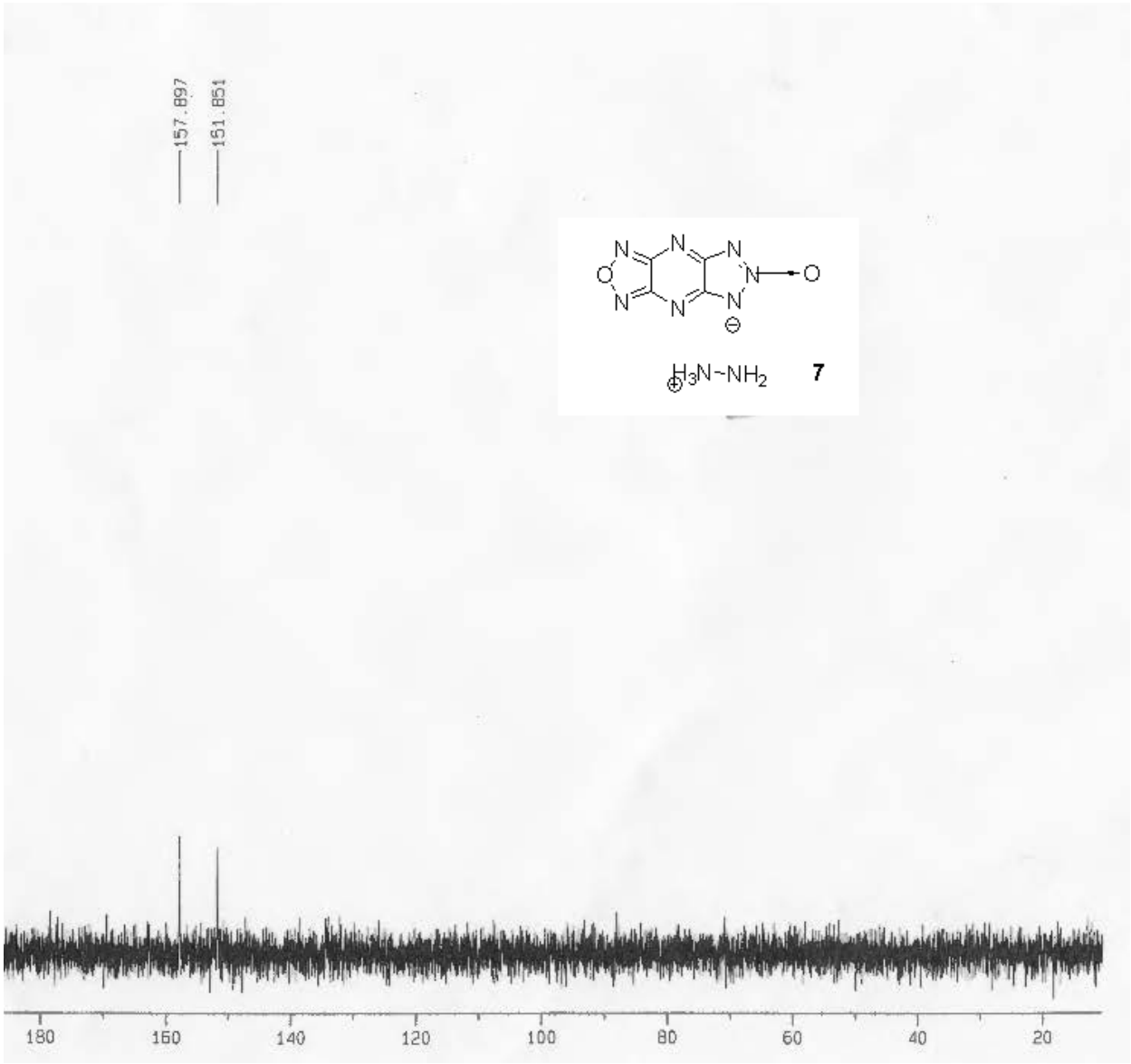


IR Spectrum

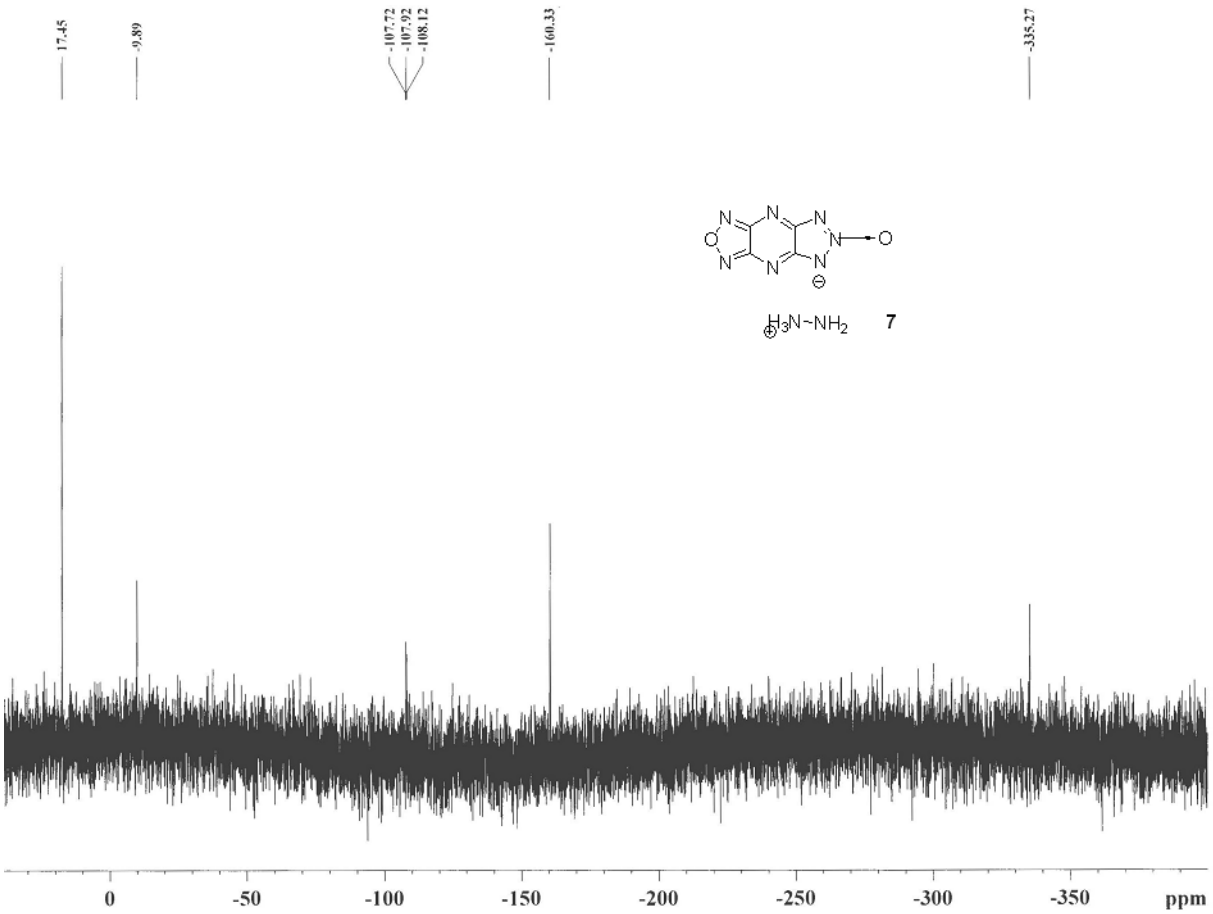

6
NH4
3568.2
1637.4
1481.8
1466.6
1402.0
1339.0
1065.2
999.1
893.0
3500
3000
2500
2000
1500
1000
Wavenumber


DSC
6
4
2
0
-2
-4
0
50
100
150
200
250
300
350
400
Temperature (°C)
293.64°C
269.07°C
71.37°C
261.78°C
6
NH4
Universal V4.5A TA Instruments

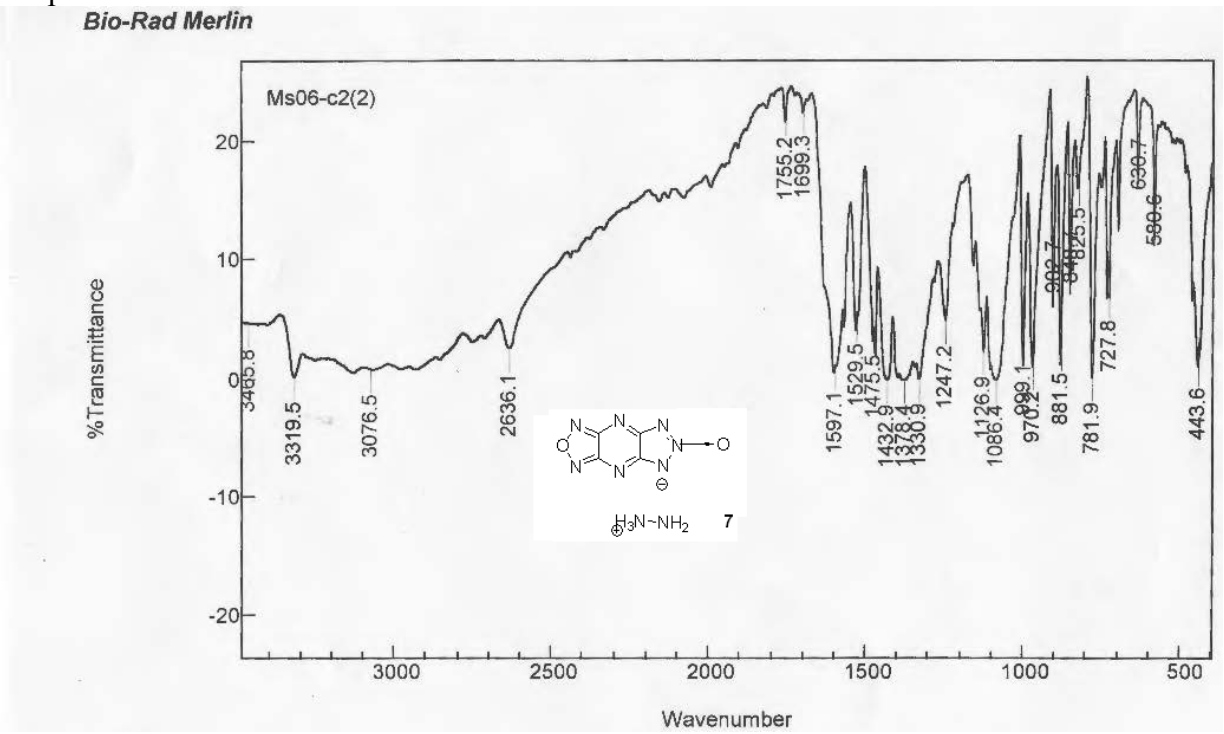
^{13}C NMR in D_2O



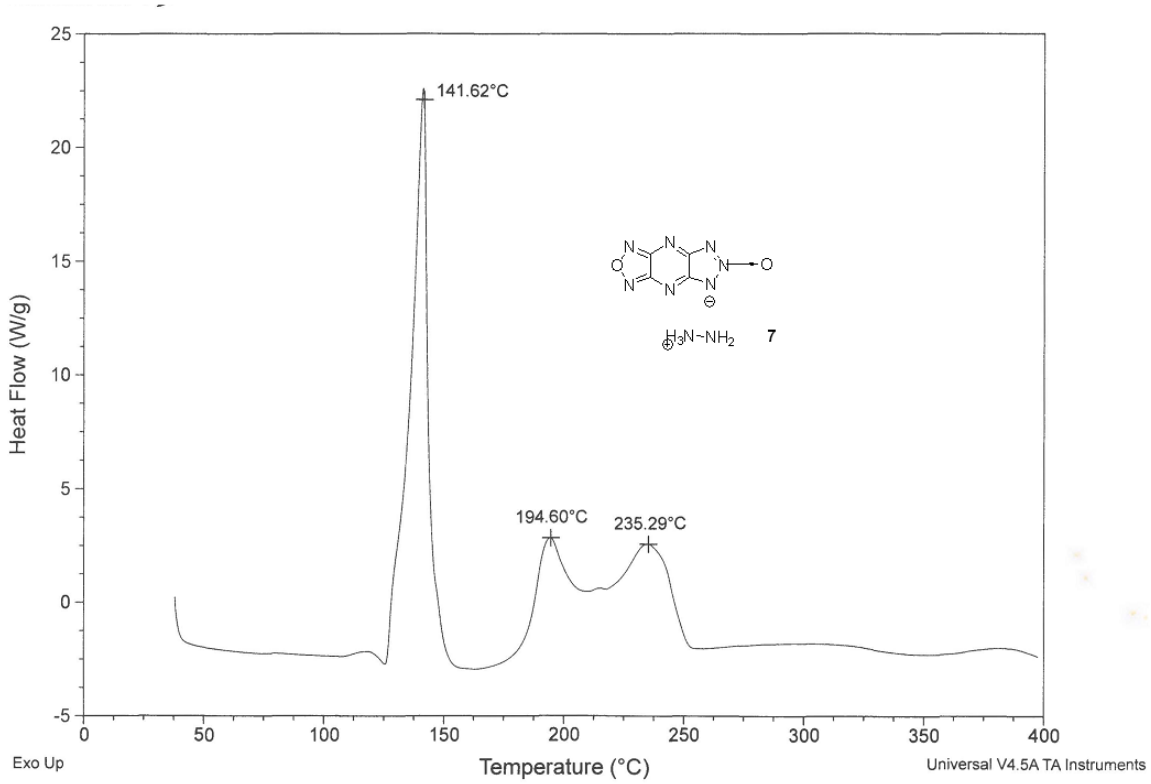
^{15}N NMR in [D$_6$]DMSO



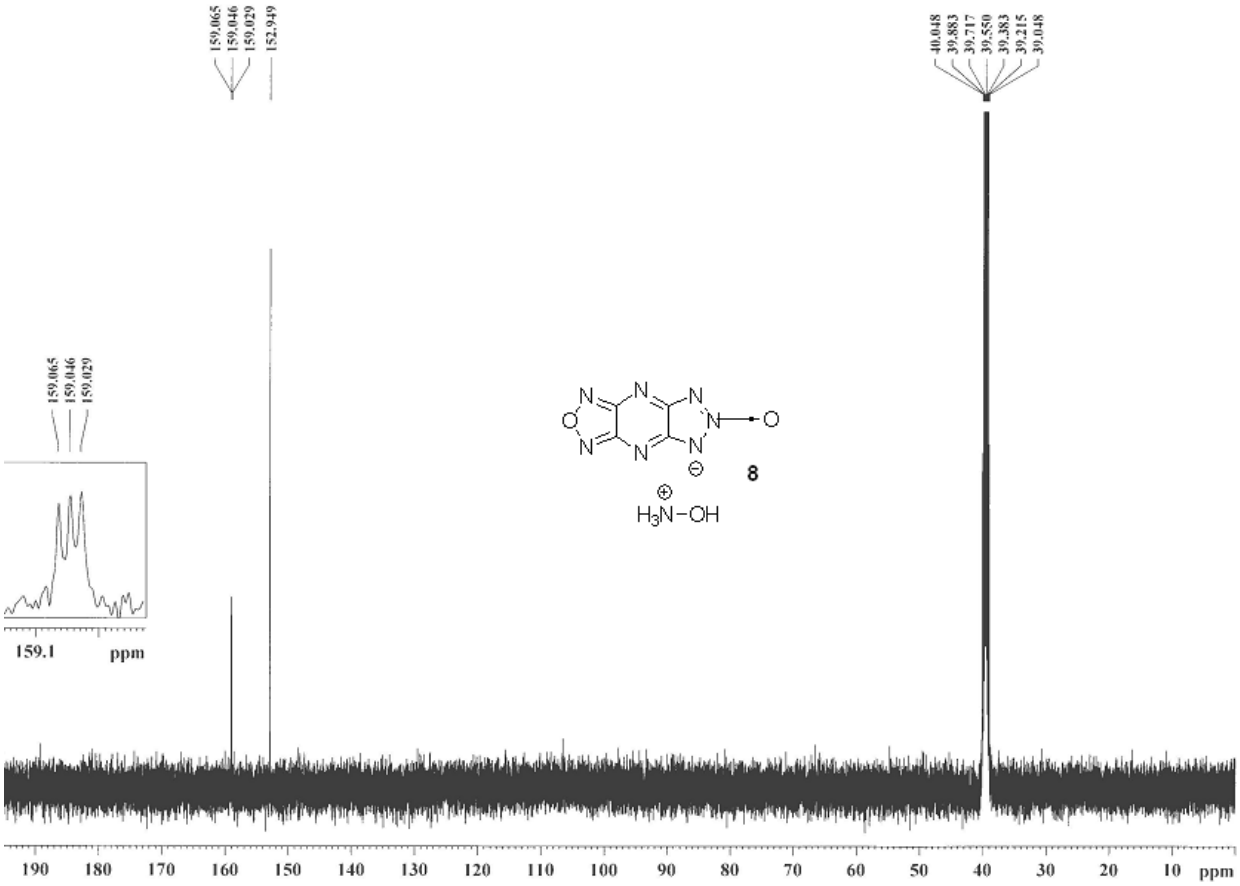
IR Spectrum


Bio-Rad Merlin
Ms06-c2(2)
%Transmittance
Wavenumber
20
10
0
-10
-20
3000
2500
2000
1500
1000
500
3465.8
3319.5
3076.5
2636.1
1755.2
1699.3
1597.1
1529.5
1475.5
1432.9
1378.4
1330.9
1247.2
1126.9
1086.4
881.5
781.9
727.8
630.7
580.6
443.6
$H_3N{\sim}NH_2$
7

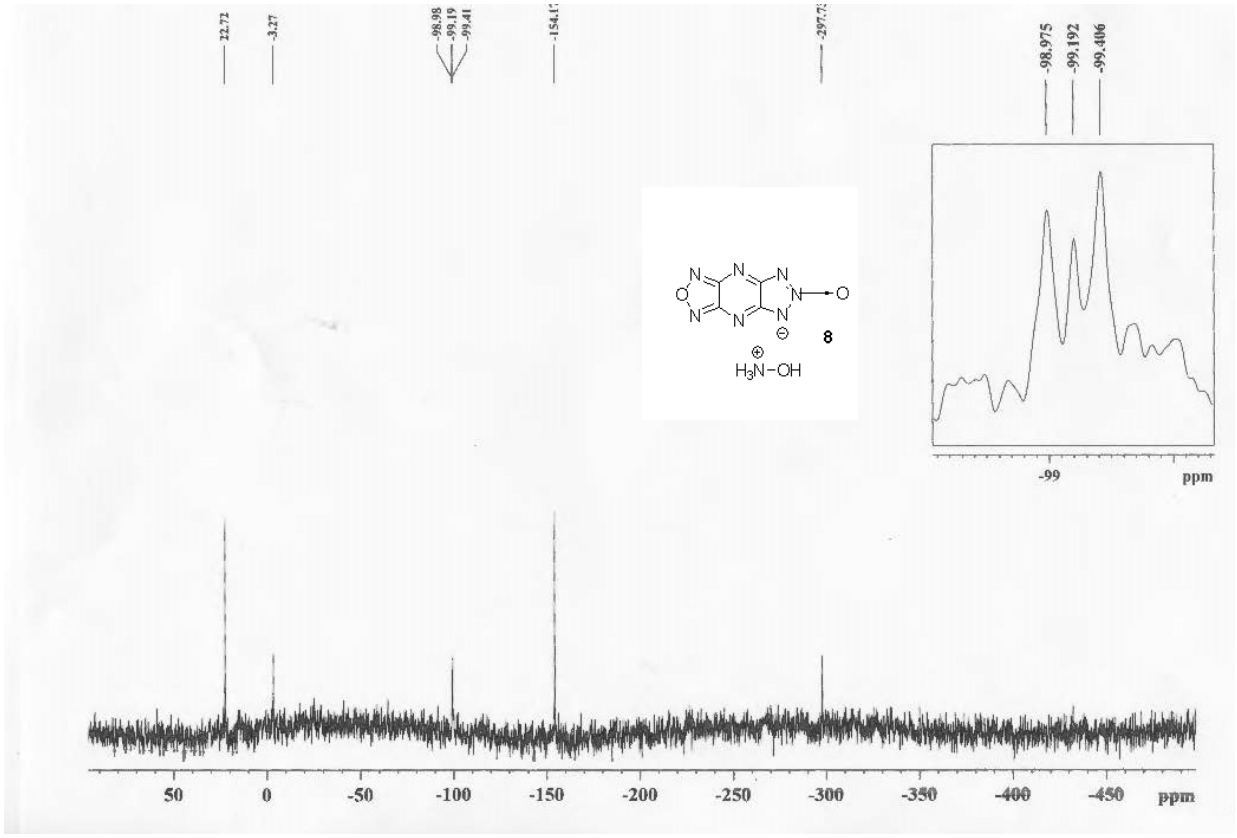
DSC


141.62°C
194.60°C
235.29°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.5A TA Instruments
7

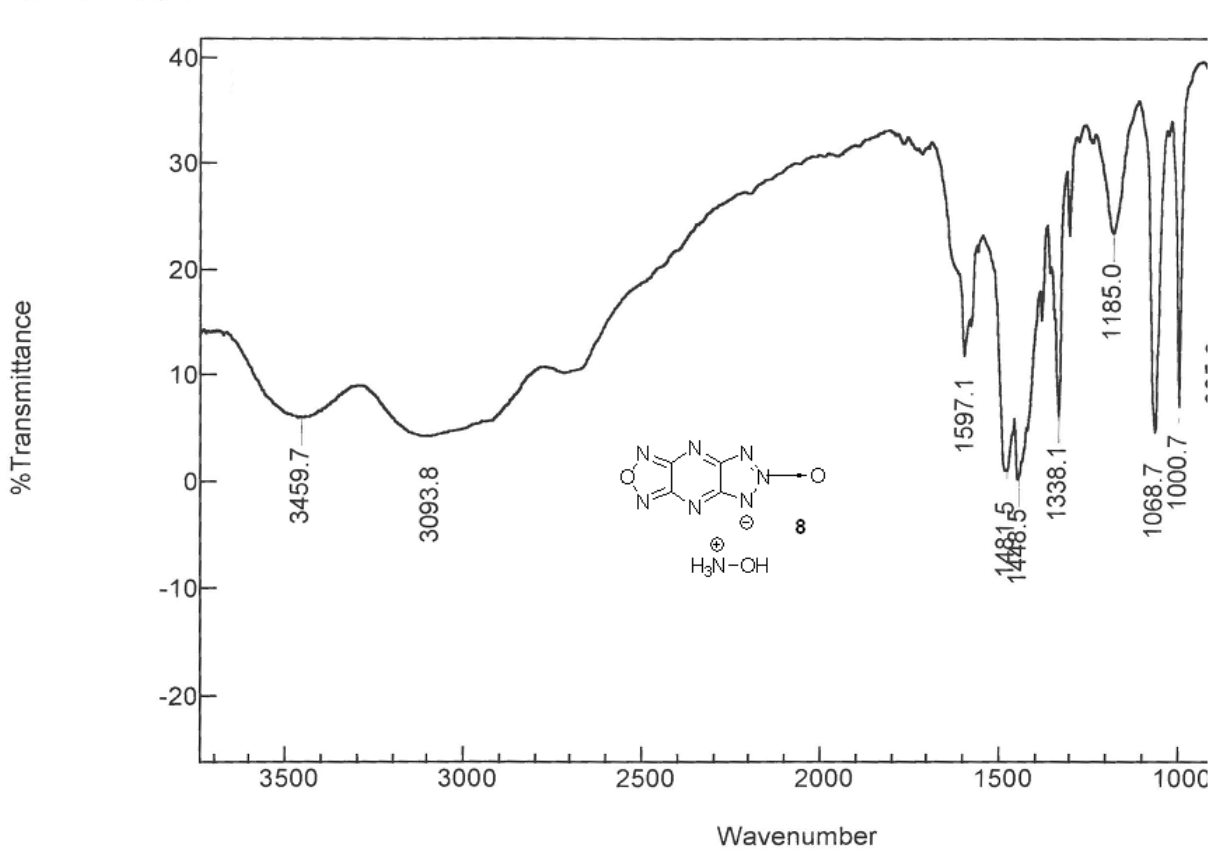
^{13}C NMR in [D$_6$]DMSO



^{15}N NMR in [D$_6$]DMSO



IR Spectrum



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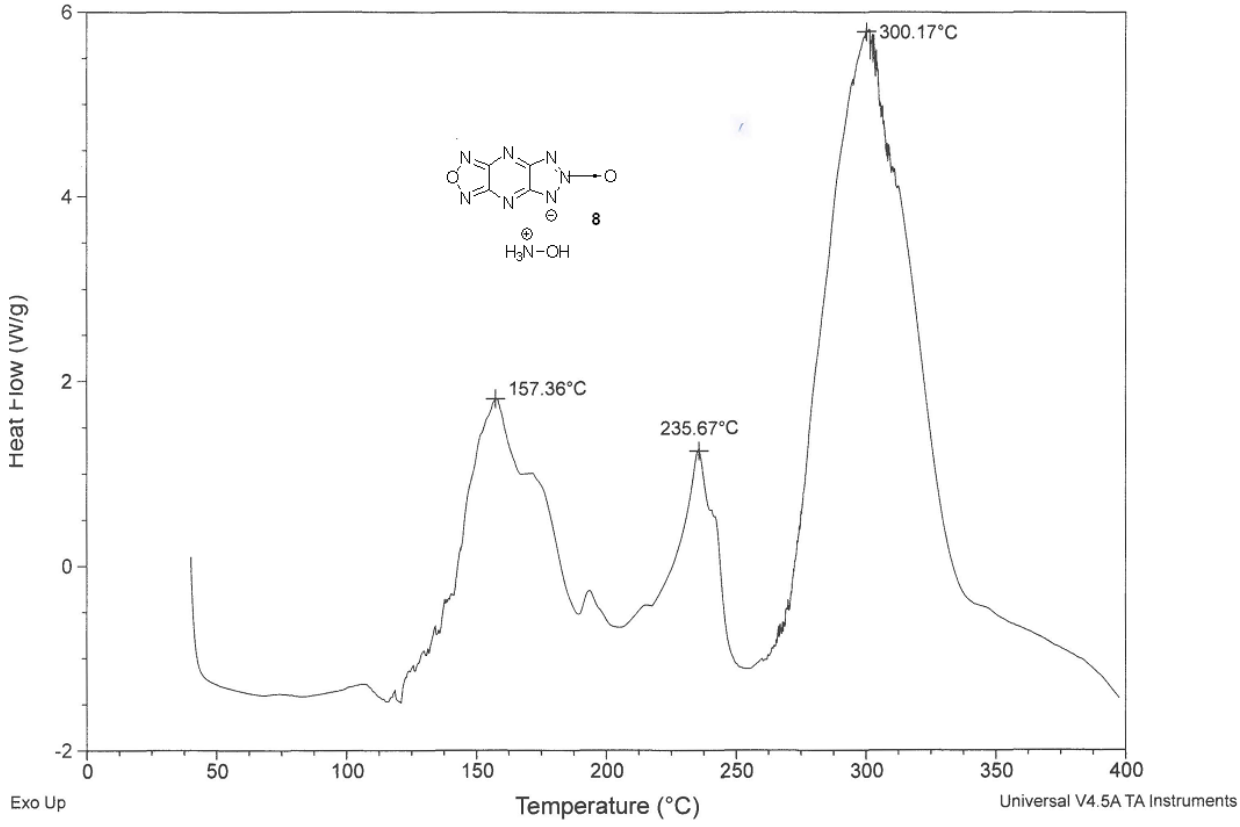
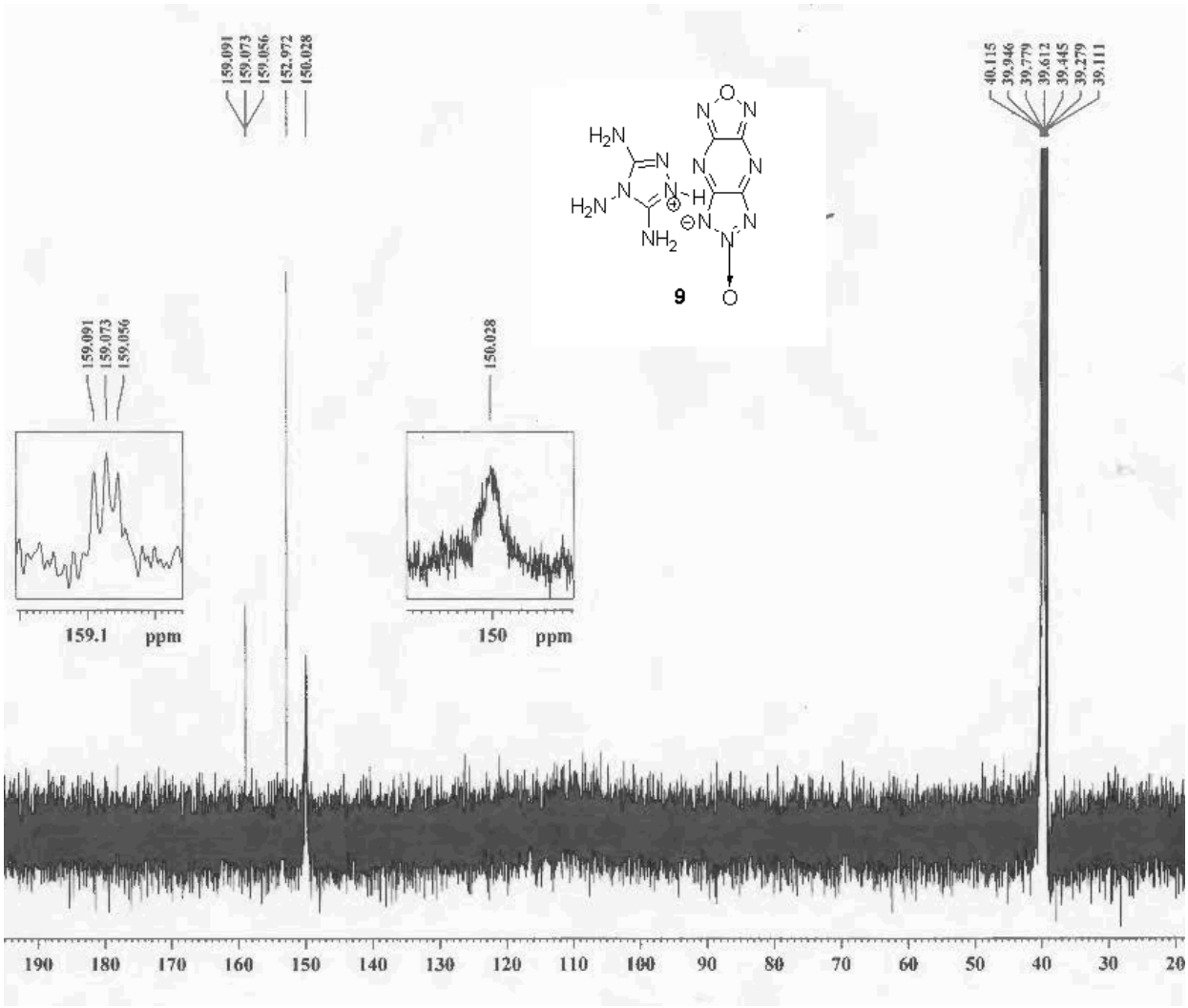
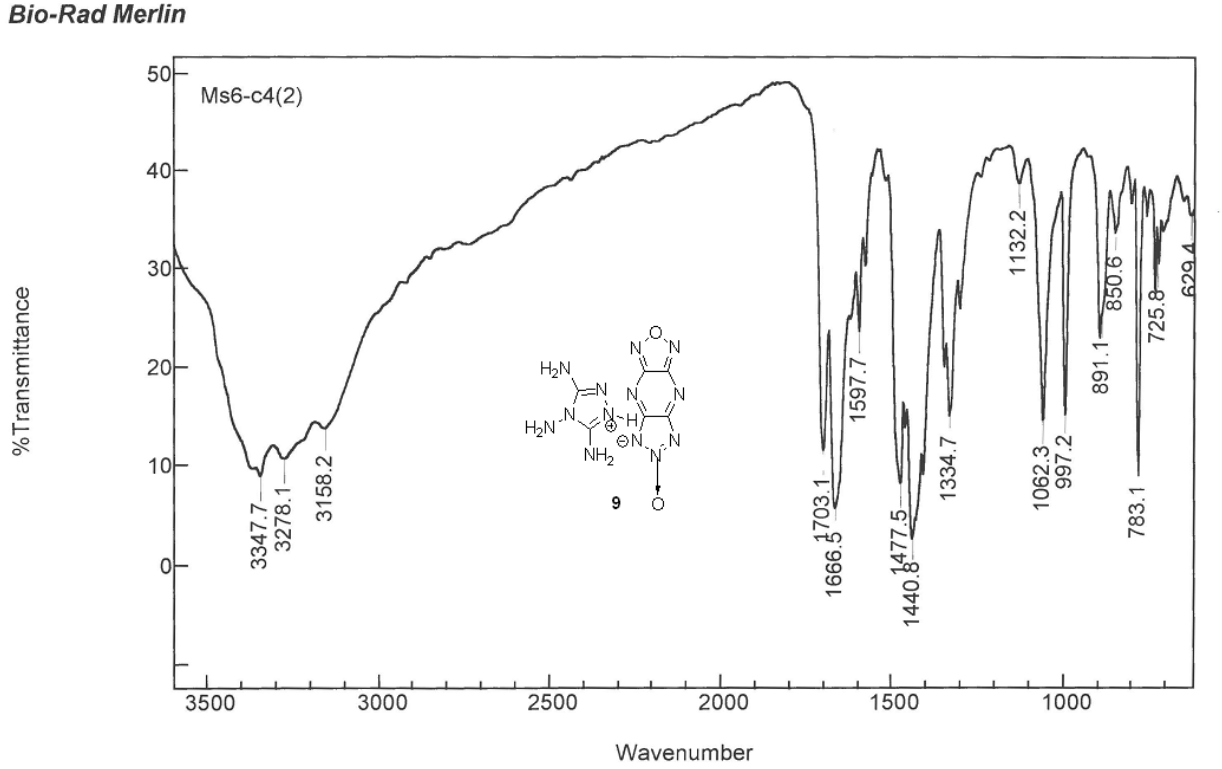


^{13}C NMR in [D$_6$]DMSO



IR Spectrum



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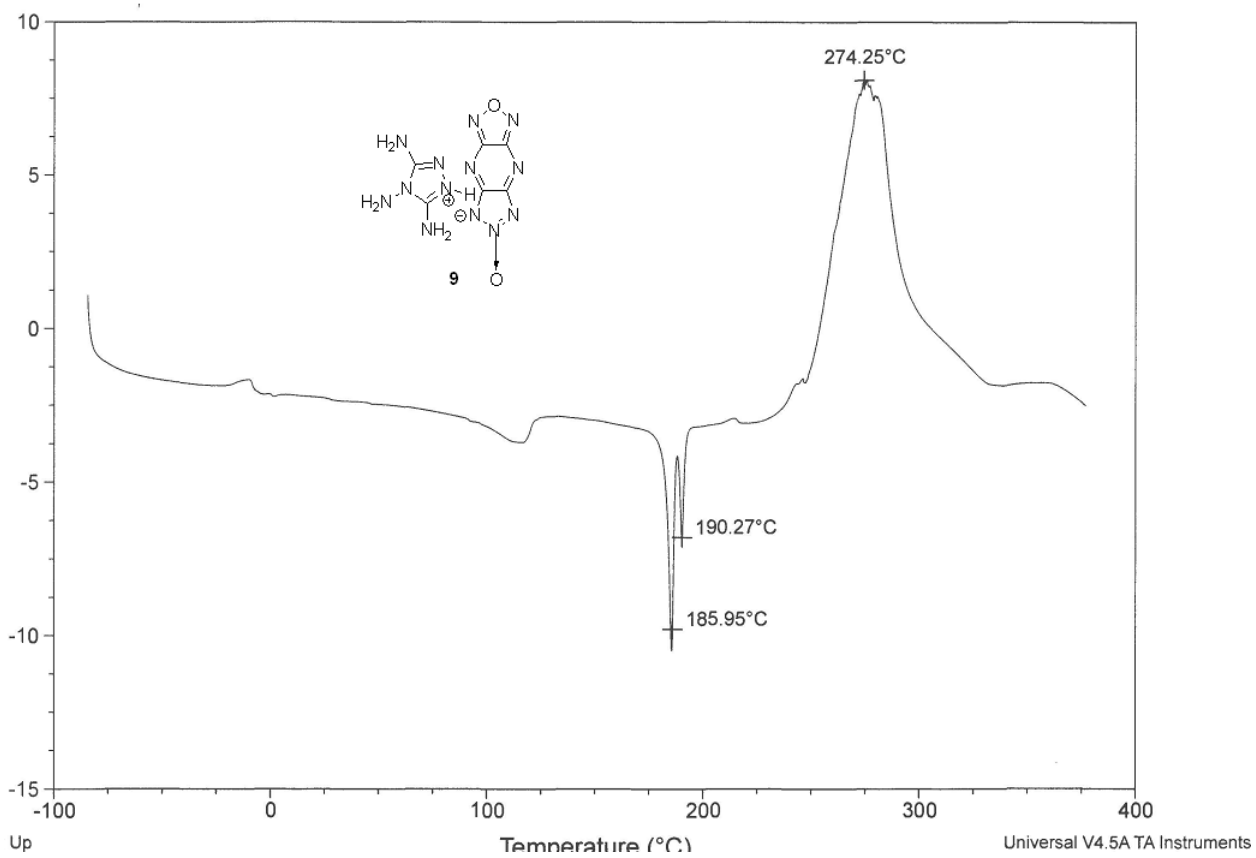
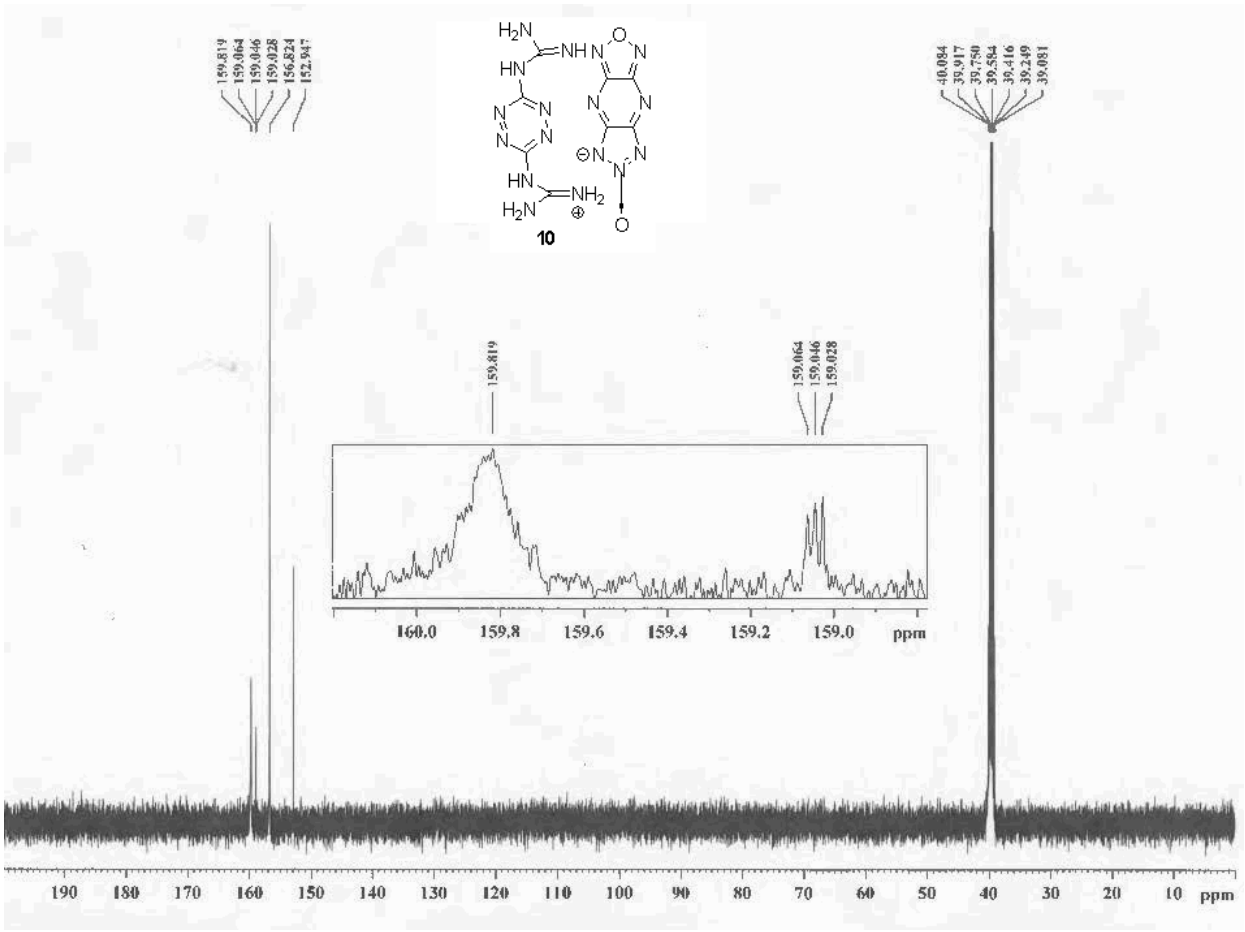
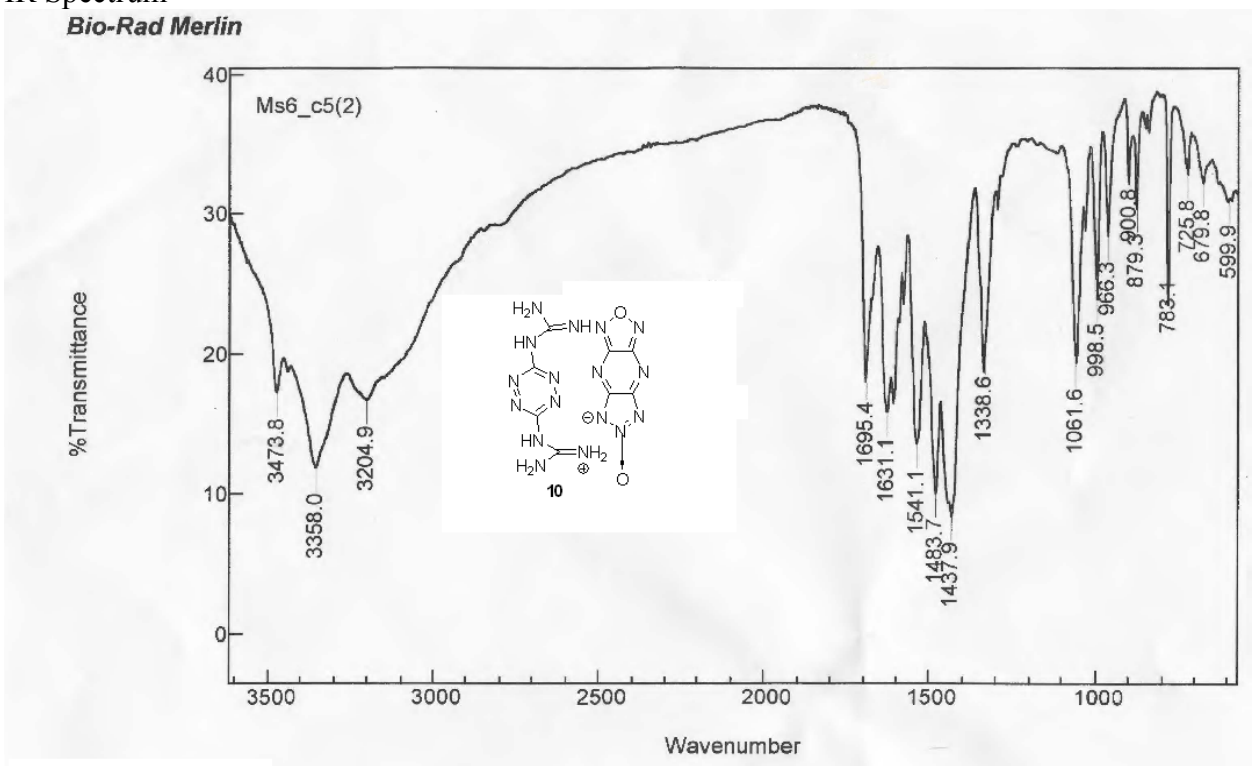


^{13}C NMR in [D$_6$]DMSO



IR Spectrum


Bio-Rad Merlin
Ms6_c5(2)
%Transmittance
Wavenumber
3473.8
3358.0
3204.9
1695.4
1631.1
1541.1
1483.7
1437.9
1338.6
1061.6
998.5
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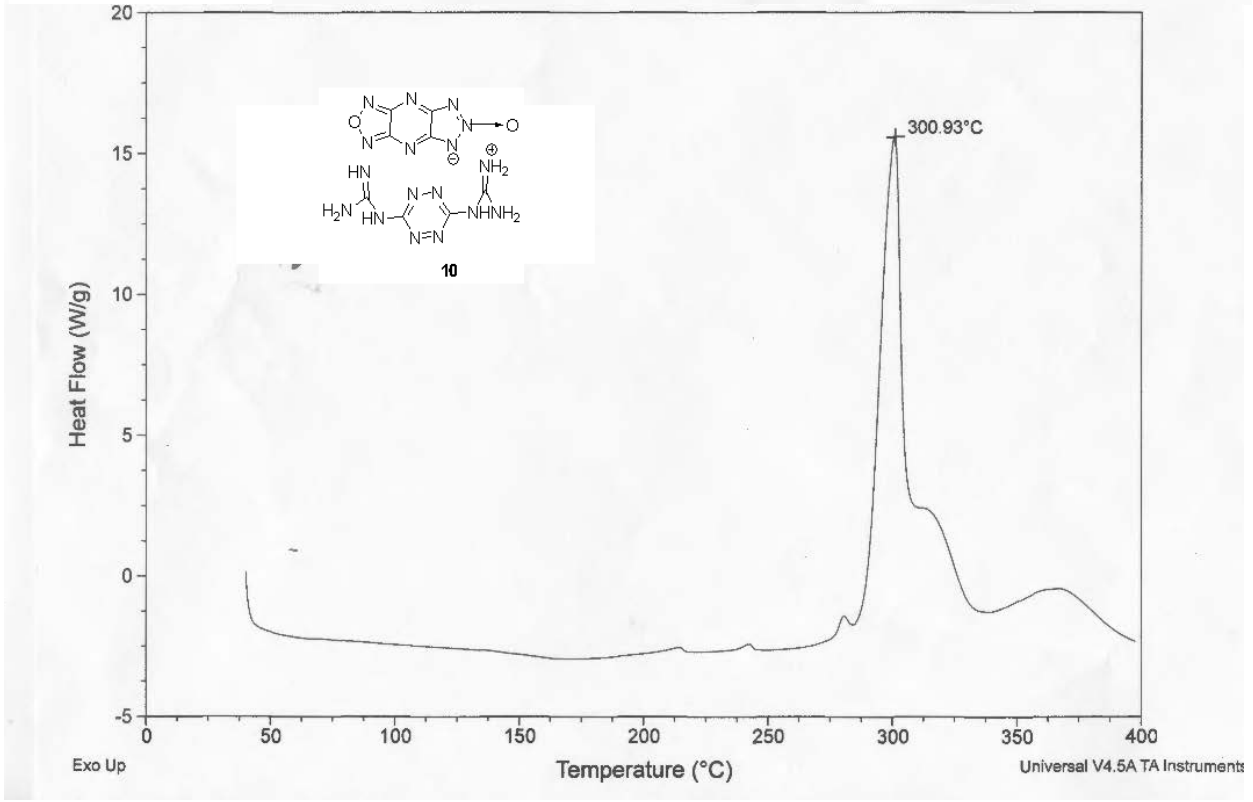
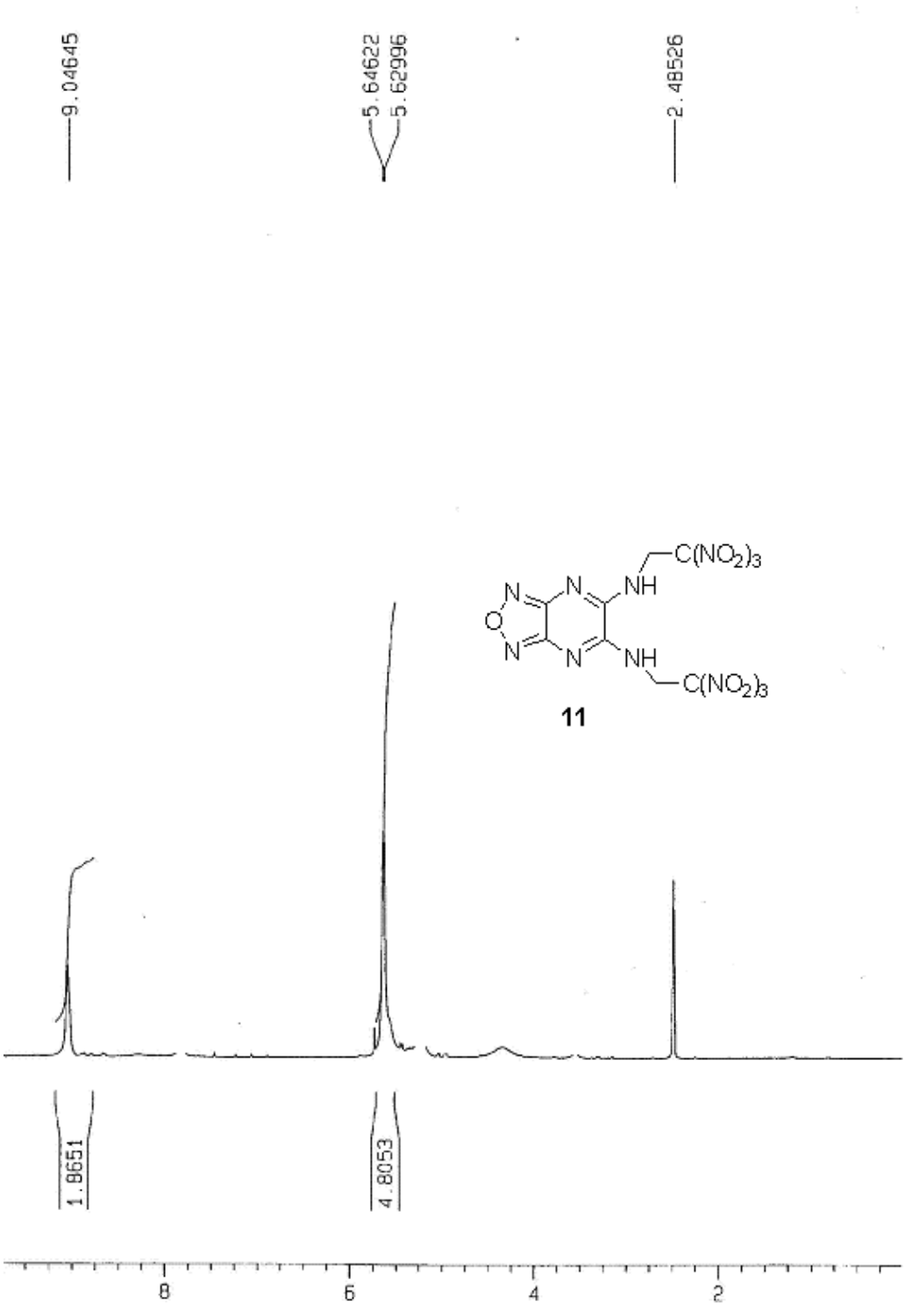
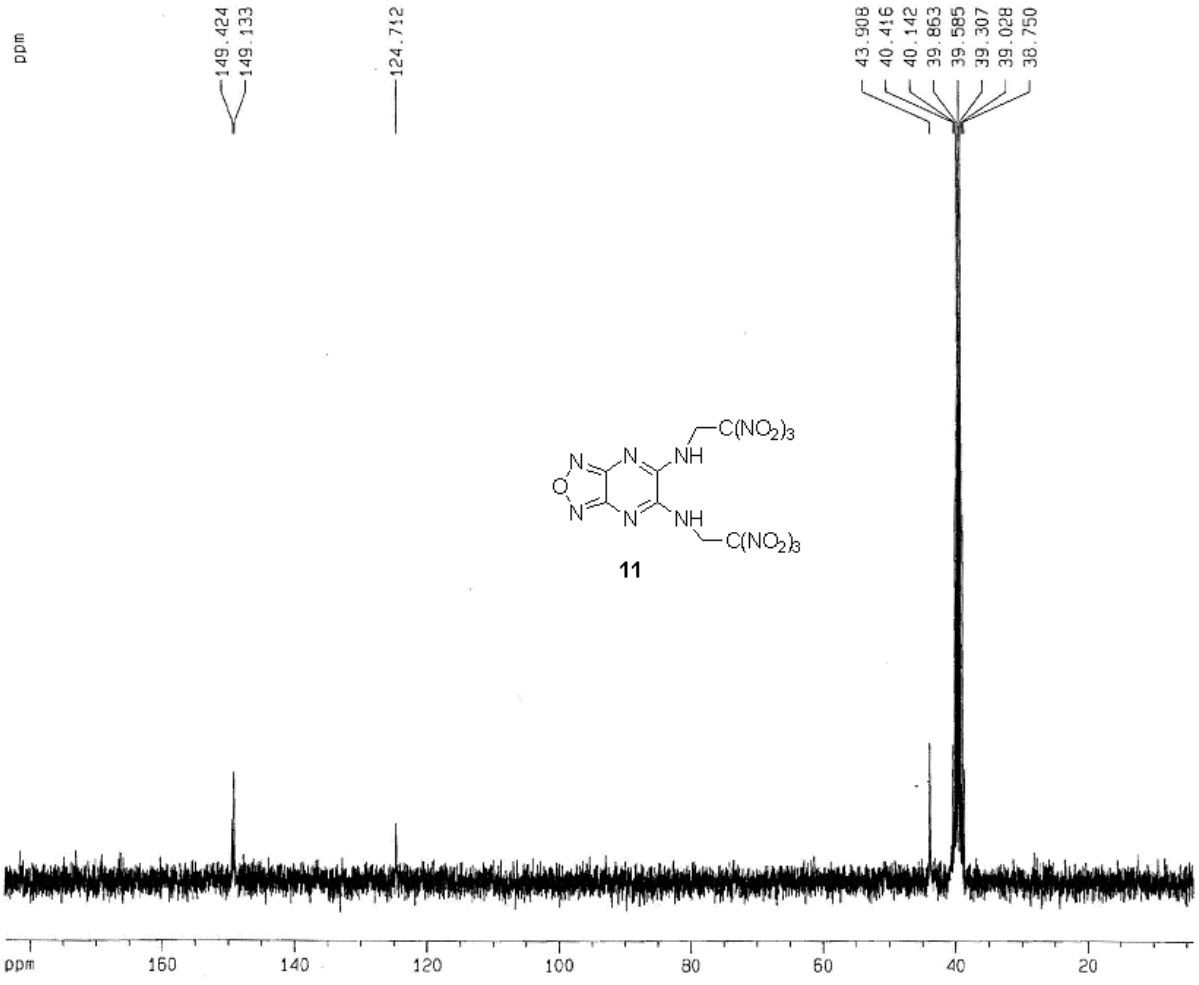
900.8
879.3
783.1
725.8
679.8
599.9
10


Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.5A TA Instruments
300.93°C
10

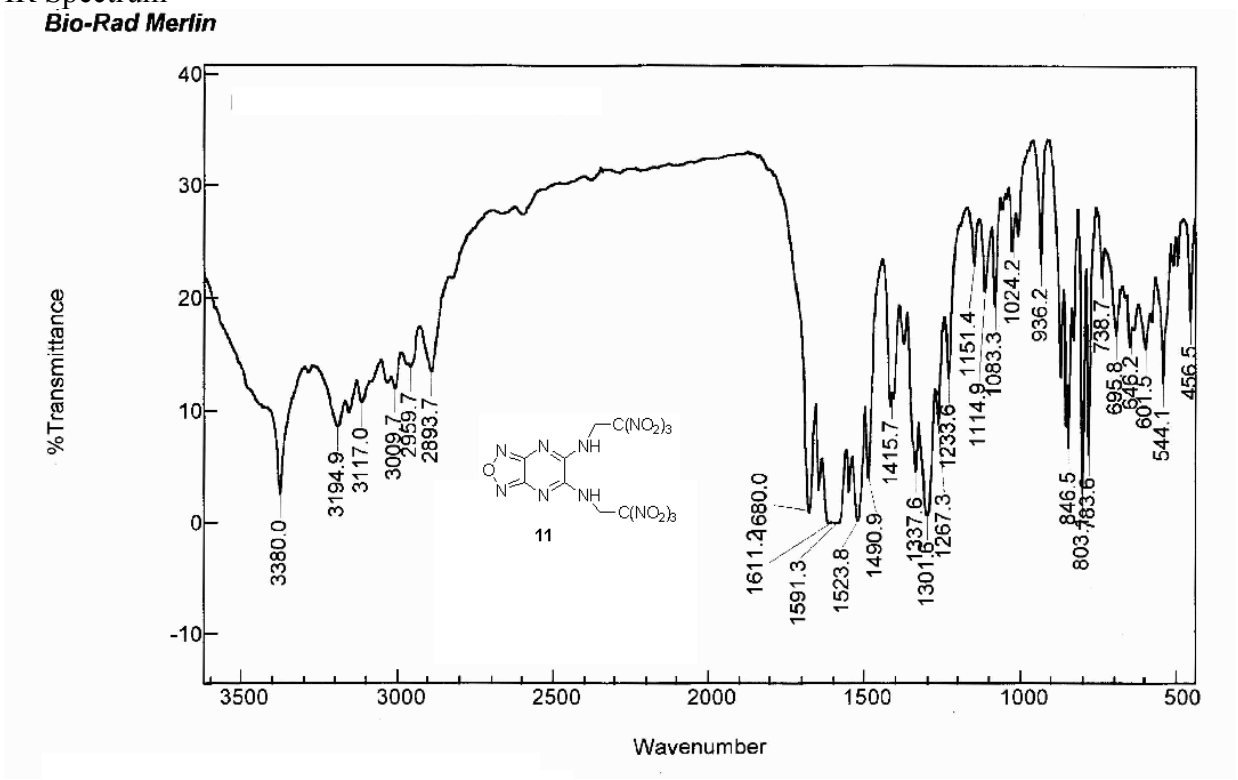
^{1}H NMR in [D$_6$]DMSO



^{13}C NMR in [D$_6$]DMSO



IR Spectrum



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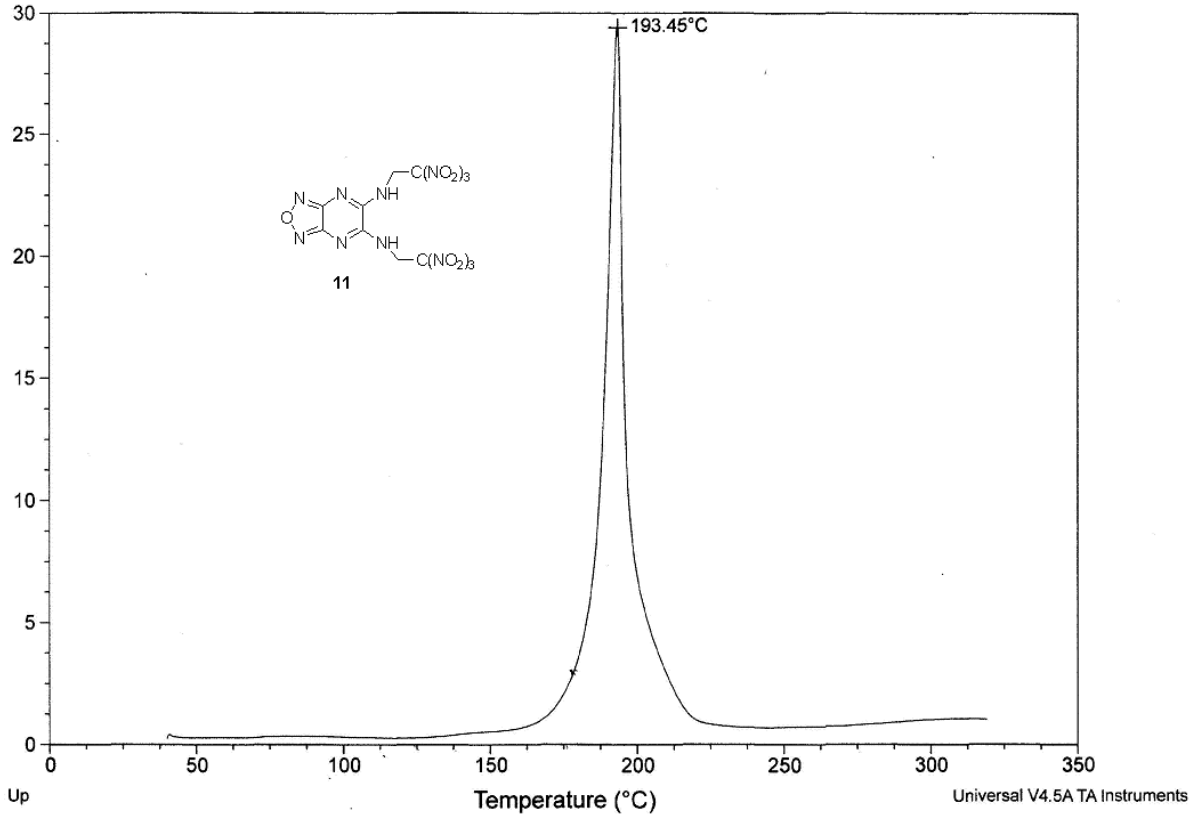

Table S1. Crystal data and structure refinement for **5**.

Empirical formula	$C_5H_5N_7O_3$	
Formula weight	211.16	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.4905(18) Å	α= 90°.
	b = 13.491(2) Å	β= 98.337(5)°.
	c = 5.4699(9) Å	γ = 90°.
Volume	839.0(2) $Å^3$	
Z	4	
Density (-123ºC)	1.672 Mg/m^3	
Density (20ºC)	1.630 Mg/m^3	
Absorption coefficient	0.141 mm^{-1}	
F(000)	432	
Crystal size	0.21 x 0.13 x 0.12 mm^3	
Theta range for data collection	1.79 to 26.54°.	
Index ranges	-14<=h<=14, -15<=k<=16, -6<=l<=6	
Reflections collected	7708	
Independent reflections	1725 [R_{int} = 0.0721]	
Completeness to theta = 26.54°	98.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9833 and 0.9710	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1725 / 0 / 138	
Goodness-of-fit on F^2	1.242	
Final R indices [I>2sigma(I)]	R_1 = 0.0452, wR_2 = 0.1087	
R indices (all data)	R_1 = 0.0535, wR_2 = 0.1112	
Largest diff. peak and hole	0.234 and -0.280 e.$Å^{-3}$	

Table S2. Bond lengths [Å] and angles [°] for **5**.

O(1)-N(2)	1.372(3)	O(1)-N(12)	1.403(3)
N(2)-C(3)	1.311(3)	C(3)-N(4)	1.370(3)
C(3)-C(11)	1.431(3)	N(4)-C(5)	1.297(3)
C(5)-N(6)	1.364(3)	C(5)-C(9)	1.478(3)
N(6)-N(7)	1.323(3)	O(7)-N(7)	1.220(2)
N(7)-N(8)	1.413(2)	N(8)-C(9)	1.311(3)
C(9)-N(10)	1.328(3)	N(10)-C(11)	1.378(3)
N(10)-H(10)	0.8800	C(11)-N(12)	1.300(3)
O(1S)-C(2S)	1.438(3)	O(1S)-H(1S)	0.8400
C(2S)-H(2SA)	0.9800	C(2S)-H(2SB)	0.9800
C(2S)-H(2SC)	0.9800		

N(2)-O(1)-N(12)	112.74(16)	C(3)-N(2)-O(1)	104.89(18)
N(2)-C(3)-N(4)	125.8(2)	N(2)-C(3)-C(11)	108.4(2)
N(4)-C(3)-C(11)	125.8(2)	C(5)-N(4)-C(3)	110.38(18)
N(4)-C(5)-N(6)	125.63(19)	N(4)-C(5)-C(9)	127.1(2)
N(6)-C(5)-C(9)	107.22(18)	N(7)-N(6)-C(5)	103.50(16)
O(7)-N(7)-N(6)	123.90(18)	O(7)-N(7)-N(8)	118.82(18)
N(6)-N(7)-N(8)	117.28(18)	C(9)-N(8)-N(7)	101.77(16)
N(8)-C(9)-N(10)	128.77(19)	N(8)-C(9)-C(5)	110.23(19)
N(10)-C(9)-C(5)	121.00(19)	C(9)-N(10)-C(11)	114.43(18)
C(9)-N(10)-H(10)	122.8	C(11)-N(10)-H(10)	122.8
N(12)-C(11)-N(10)	127.8(2)	N(12)-C(11)-C(3)	111.0(2)
N(10)-C(11)-C(3)	121.2(2)	C(11)-N(12)-O(1)	103.00(18)
C(2S)-O(1S)-H(1S)	109.5	O(1S)-C(2S)-H(2SA)	109.5
O(1S)-C(2S)-H(2SB)	109.5	H(2SA)-C(2S)-H(2SB)	109.5
O(1S)-C(2S)-H(2SC)	109.5	H(2SA)-C(2S)-H(2SC)	109.5
H(2SB)-C(2S)-H(2SC)	109.5		

Table S3. Crystal data and structure refinement for **6**.

Empirical formula	$C_4H_4N_8O_2$	
Formula weight	196.15	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.8394(7) Å	α= 90°.
	b = 15.5423(10) Å	β= 104.344(2)°.
	c = 8.4208(5) Å	γ = 90°.
Volume	1501.22(16) $Å^3$	
Z	8	
Density (-123ºC)	1.736 Mg/m^3	
Density (20ºC)	1.712 Mg/m^3	
Absorption coefficient	0.144 mm^{-1}	
F(000)	800	
Crystal size	0.31 x 0.28 x 0.03 mm^3	
Theta range for data collection	1.78 to 26.41°.	
Index ranges	-14<=h<=14, -19<=k<=17, -10<=l<=10	
Reflections collected	13217	
Independent reflections	3078 [R_{int} = 0.0407]	
Completeness to theta = 26.41°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9957 and 0.9567	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3078 / 0 / 277	
Goodness-of-fit on F^2	1.019	
Final R indices [I>2sigma(I)]	R_1 = 0.0442, wR_2 = 0.1087	
R indices (all data)	R_1 = 0.0644, wR_2 = 0.1206	
Largest diff. peak and hole	0.508 and -0.279 e.$Å^{-3}$	

Table S4. Bond lengths [Å] and angles [°] for **6**.

N(1A)-O(13A)	1.249(2)	N(1A)-N(12A)	1.331(2)
N(1A)-N(2A)	1.353(2)	N(2A)-C(3A)	1.351(3)
C(3A)-N(4A)	1.307(3)	C(3A)-C(11A)	1.481(3)
N(4A)-C(5A)	1.367(3)	C(5A)-N(6A)	1.308(3)
C(5A)-C(9A)	1.440(3)	N(6A)-O(7A)	1.385(2)
O(7A)-N(8A)	1.393(2)	N(8A)-C(9A)	1.311(3)
C(9A)-N(10A)	1.369(3)	N(10A)-C(11A)	1.306(3)
C(11A)-N(12A)	1.353(3)	N(1B)-O(13B)	1.221(2)
N(1B)-N(2B)	1.349(3)	N(1B)-N(12B)	1.349(3)
N(2B)-C(3B)	1.356(3)	C(3B)-N(4B)	1.309(3)
C(3B)-C(11B)	1.469(3)	N(4B)-C(5B)	1.376(3)
C(5B)-N(6B)	1.309(3)	C(5B)-C(9B)	1.432(3)
N(6B)-O(7B)	1.389(2)	O(7B)-N(8B)	1.388(3)
N(8B)-C(9B)	1.310(3)	C(9B)-N(10B)	1.368(3)
N(10B)-C(11B)	1.309(3)	C(11B)-N(12B)	1.361(3)
N(1S)-H(1SA)	0.79(3)	N(1S)-H(1SB)	0.88(3)
N(1S)-H(1SC)	0.90(3)	N(1S)-H(1SD)	0.94(3)
N(2S)-H(2SA)	0.80(3)	N(2S)-H(2SB)	0.92(3)
N(2S)-H(2SC)	1.01(3)	N(2S)-H(2SD)	0.92(3)
O(13A)-N(1A)-N(12A)	120.82(17)	O(13A)-N(1A)-N(2A)	119.45(17)
N(12A)-N(1A)-N(2A)	119.73(17)	C(3A)-N(2A)-N(1A)	101.74(16)
N(4A)-C(3A)-N(2A)	126.20(18)	N(4A)-C(3A)-C(11A)	125.58(19)
N(2A)-C(3A)-C(11A)	108.21(18)	C(3A)-N(4A)-C(5A)	110.05(17)
N(6A)-C(5A)-N(4A)	126.32(19)	N(6A)-C(5A)-C(9A)	109.45(19)
N(4A)-C(5A)-C(9A)	124.2(2)	C(5A)-N(6A)-O(7A)	104.75(17)
N(6A)-O(7A)-N(8A)	112.12(15)	C(9A)-N(8A)-O(7A)	104.58(17)
N(8A)-C(9A)-N(10A)	125.8(2)	N(8A)-C(9A)-C(5A)	109.1(2)
N(10A)-C(9A)-C(5A)	125.08(19)	C(11A)-N(10A)-C(9A)	109.81(18)
N(10A)-C(11A)-N(12A)	126.76(19)	N(10A)-C(11A)-C(3A)	125.23(19)
N(12A)-C(11A)-C(3A)	108.01(18)	N(1A)-N(12A)-C(11A)	102.30(16)
O(13B)-N(1B)-N(2B)	120.44(18)	O(13B)-N(1B)-N(12B)	120.74(18)
N(2B)-N(1B)-N(12B)	118.81(18)	N(1B)-N(2B)-C(3B)	102.51(17)
N(4B)-C(3B)-N(2B)	126.0(2)	N(4B)-C(3B)-C(11B)	125.8(2)
N(2B)-C(3B)-C(11B)	108.16(18)	C(3B)-N(4B)-C(5B)	109.31(18)
N(6B)-C(5B)-N(4B)	125.9(2)	N(6B)-C(5B)-C(9B)	109.6(2)
N(4B)-C(5B)-C(9B)	124.5(2)	C(5B)-N(6B)-O(7B)	104.65(18)
N(8B)-O(7B)-N(6B)	111.69(16)	C(9B)-N(8B)-O(7B)	105.03(17)
N(8B)-C(9B)-N(10B)	125.6(2)	N(8B)-C(9B)-C(5B)	109.0(2)
N(10B)-C(9B)-C(5B)	125.4(2)	C(11B)-N(10B)-C(9B)	109.06(18)
N(10B)-C(11B)-N(12B)	125.65(19)	N(10B)-C(11B)-C(3B)	125.9(2)
N(12B)-C(11B)-C(3B)	108.43(18)	N(1B)-N(12B)-C(11B)	102.08(17)
H(1SA)-N(1S)-H(1SB)	108(3)	H(1SA)-N(1S)-H(1SC)	108(3)