Supplementary Material

Thio-glycomimetics with enhanced lipophilicity and their biological activity

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1. General information

All reagents and solvents were used as purchased without further purification. Unless otherwise stated, all reactions were carried out under inert atmosphere in oven-dried glassware with dried solvents. All solvents were dried and degassed by standard methods before use. Optical rotation was measured using a JASCO
P-2000 Digital Polarimeter. 1H NMR and 13C NMR spectra were recorded on 400MHz Bruker Avance.2D experiments (COSY and HSQC) were performed to enhance assignments. Chemical shifts (δ-scale) are reported in ppm with TMS (0 ppm) as internal standard for ^{1}H NMR and the residual solvent signals (CDCl3: 7.26, for H NMR and (CDCl3: 77.0 ppm) for ^{13} C NMR. Thin layer chromatography was performed on silica gel coated TLC plates and visualized under UV light (at 254nm); detection was executed by exposing to iodine (I2) vapor. The melting points (mp) were obtained on an ElectroThermal FARGO MP- 2D capillary melting point apparatus and were uncorrected. Chemical names were generated by ChemDraw Professional V.15.1.0.144 software.

Crystal Structure Report for compound 10

(1R,2R,5S,6R)-8-(((3S,5S,7S)-adamantan-1-yl)thio)-3,11-dioxa-7,9-diazatricyclo[4.3.1.12,5]undec-8-en-1-ol

A specimen of $C_{17}H_{24}N_2O_3S$ was used for the X-ray crystallographic analysis. The X-ray intensity data were measured. The total exposure time was 12.20 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a tetragonal unit cell yielded a total of 5025 reflections to a maximum θ angle of 26.00° (0.81 Å resolution), of which 2963 were independent (average redundancy 1.696, completeness = 94.0%, R_{int} = 2.16%, R_{sig} = 3.44%) and 2733 (92.24%) were greater than 2σ(F^2). The final cell constants of a = 11.730(7) Å, b = 11.730(7) Å, c = 24.056(15) Å, volume = 3310.(4) Å^3, are based upon the refinement of the XYZ-centroids of 9858 reflections above 20 σ(I) with 4.850° < 2θ < 64.14°. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.945. The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 41 21 2, with Z = 8 for the formula unit, $C_{17}H_{24}N_2O_3S$. The final anisotropic full-matrix least-squares refinement on F^2 with 304 variables converged at R1 = 3.03%, for the observed data and wR2 = 7.39% for all data. The goodness-of-fit was 1.060. The largest peak in the final difference electron density synthesis was 0.389 e^-/Å^3 and the largest hole was -0.167 e^-/Å^3 with an RMS deviation of 0.038 e^-/Å^3. On the basis of the final model, the calculated density was 1.350 g/cm^3 and F(000), 1440 e^-.

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Formula weight	336.44 g/mol	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	tetragonal	
Space group	P41212	
Unit cell dimensions	a = 11.730(7) Å	α=90°
	b = 11.730(7) Å	β=90°
	c = 24.056(15) Å	γ=90°
Volume	3310.(4) $Å^3$	
Z	8	
Density (calculated)	1.350 g/cm^3	
Absorption coefficient	0.212 mm^{-1}	
F(000)	1440	

Table 2. Data collection and structure refinement for compound 10. Theta range for data collection

1.93 to 26.00°	
Index ranges	-14<=h<=4, -9<=k<=12, -8<=l<=29
Reflections collected	5025
Independent reflections	2963 [R(int) = 0.0216]
Coverage of independent reflections	94.0%
Absorption correction	Multi-Scan
Structure solution technique	direct methods
Structure solution program	XT, VERSION 2014/4
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2014/7 (Sheldrick, 2014)
Function minimized	
Data / restraints / parameters	2963 / 0 / 304
Goodness-of-fit on F^2	1.060
Final R indices	2733 data; I>2σ(I) R1 = 0.0347, wR2 =
	all data 0.0739
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0395P)^2+0.4573P]$ where $P=(F_o^2+2F_c^2)/3$
Absolute structure parameter	-0.1(0)
Largest diff. peak and hole	0.389 and -0.167 eÅ^{-3}
R.M.S. deviation from mean	0.038 eÅ^{-3}

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters ($Å^2$) for compound 10.

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

	x/a	y/b	z/c	U(eq)
S1	0.60339(6)	0.13124(6)	0.47987(2)	0.01858(16)
O1	0.24982(16)	0.34650(16)	0.43494(7)	0.0198(4)
O2	0.53477(16)	0.46250(16)	0.41065(7)	0.0204(4)
O3	0.37126(17)	0.53325(16)	0.37394(7)	0.0244(4)
N1	0.5636(2)	0.2388(2)	0.38374(9)	0.0202(5)
N2	0.43146(18)	0.27116(18)	0.45642(8)	0.0150(5)
C1	0.5396(2)	0.9905(2)	0.46352(9)	0.0162(5)
C2	0.4123(2)	0.9864(2)	0.47898(11)	0.0218(6)
C3	0.3665(3)	0.8649(2)	0.46968(11)	0.0262(6)
C4	0.4332(3)	0.7812(3)	0.50683(11)	0.0285(7)
C5	0.5595(3)	0.7853(2)	0.49243(10)	0.0233(7)
C6	0.5775(3)	0.7517(3)	0.43119(10)	0.0224(6)
C7	0.5107(3)	0.8342(2)	0.39390(10)	0.0219(6)
C8	0.5560(2)	0.9564(2)	0.40222(9)	0.0177(6)
C9	0.3832(3)	0.8304(3)	0.40856(12)	0.0275(7)
C10	0.6051(3)	0.9068(2)	0.50105(10)	0.0214(6)
C11	0.5223(2)	0.2243(2)	0.43615(9)	0.0156(5)
C12	0.5012(2)	0.3111(2)	0.34460(10)	0.0204(6)
C13	0.3744(3)	0.2995(2)	0.35902(10)	0.0196(6)
C14	0.3650(2)	0.3448(2)	0.41847(9)	0.0160(5)
C15	0.4155(2)	0.4679(2)	0.41936(10)	0.0179(6)
C16	0.4533(3)	0.5221(3)	0.32924(11)	0.0296(7)
C17	0.5394(3)	0.4361(2)	0.35209(10)	0.0234(6)

Table 4. Bond lengths (Å) for compound 10.

S1-C11	1.789(3)	S1-C1	1.855(3)
O1-C14	1.408(3)	O1-H1O	0.81(3)
O2-C15	1.416(3)	O2-C17	1.443(3)
O3-C15	1.432(3)	O3-C16	1.449(3)
N1-C11	1.361(3)	N1-C12	1.463(3)
N1-H1N	0.81(3)	N2-C11	1.294(3)
N2-C14	1.480(3)	C1-C10	1.539(4)
C1-C8	1.540(3)	C1-C2	1.541(4)

C2-C3	1.539(4)	C2-H2A	0.95(3)
C2-H2B	0.98(3)	C3-C9	1.538(4)
C3-C4	1.541(4)	C3-H3	1.00(3)
C4-C5	1.523(4)	C4-H4A	0.97(3)
C4-H4B	1.02(3)	C5-C10	1.536(4)
C5-C6	1.540(3)	C5-H5	0.98(3)
C6-C7	1.535(4)	C6-H6A	0.99(3)
C6-H6B	0.99(3)	C7-C9	1.537(5)
C7-C8	1.542(4)	C7-H7	1.00(2)
C8-H8A	1.04(3)	C8-H8B	0.97(3)
C9-H9A	0.99(3)	C9-H9B	0.95(3)
C10-H10A	0.96(3)	C10-H10B	1.01(3)
C12-C13	1.534(4)	C12-C17	1.544(4)
C12-H12	0.98(3)	C13-C14	1.530(3)
C13-H13A	0.99(3)	C13-H13B	1.02(3)
C14-C15	1.561(4)	C15-H15	0.94(2)
C16-C17	1.529(4)	C16-H16A	1.01(3)
C16-H16B	1.01(4)	C17-H17	0.93(3)

Table 5. Bond angles (°) for compound 10.

C11-S1-C1	101.78(12)	C14-O1-H1O	110.(2)
C15-O2-C17	101.0(2)	C15-O3-C16	106.1(2)
C11-N1-C12	119.4(2)	C11-N1-H1N	119.(2)
C12-N1-H1N	121.(2)	C11-N2-C14	116.72(19)
C10-C1-C8	109.5(2)	C10-C1-C2	108.9(2)
C8-C1-C2	110.1(2)	C10-C1-S1	104.01(17)
C8-C1-S1	112.56(18)	C2-C1-S1	111.54(18)
C3-C2-C1	109.3(2)	C3-C2-H2A	112.1(18)
C1-C2-H2A	107.6(18)	C3-C2-H2B	109.8(17)
C1-C2-H2B	108.5(17)	H2A-C2-H2B	109.(2)
C9-C3-C2	109.8(2)	C9-C3-C4	108.8(2)
C2-C3-C4	109.2(2)	C9-C3-H3	107.6(18)
C2-C3-H3	108.(2)	C4-C3-H3	112.9(19)
C5-C4-C3	109.9(2)	C5-C4-H4A	108.1(17)
C3-C4-H4A	110.3(17)	C5-C4-H4B	111.1(18)
C3-C4-H4B	109.5(17)	H4A-C4-H4B	108.(2)
C4-C5-C10	109.8(2)	C4-C5-C6	110.0(2)

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C10-C5-C6	108.6(2)	C4-C5-H5	109.0(18)
C10-C5-H5	109.6(18)	C6-C5-H5	109.8(17)
C7-C6-C5	109.1(2)	C7-C6-H6A	107.3(18)
C5-C6-H6A	112.0(17)	C7-C6-H6B	109.1(17)
C5-C6-H6B	110.1(17)	H6A-C6-H6B	109.(3)
C6-C7-C9	110.1(2)	C6-C7-C8	109.5(2)
C9-C7-C8	109.5(2)	C6-C7-H7	110.2(17)
C9-C7-H7	109.3(17)	C8-C7-H7	108.2(17)
C1-C8-C7	108.8(2)	C1-C8-H8A	108.1(14)
C7-C8-H8A	110.4(16)	C1-C8-H8B	108.6(16)
C7-C8-H8B	111.4(17)	H8A-C8-H8B	109.(2)
C7-C9-C3	109.6(2)	C7-C9-H9A	108.9(17)
C3-C9-H9A	110.1(16)	C7-C9-H9B	110.3(19)
C3-C9-H9B	108.6(16)	H9A-C9-H9B	109.(2)
C5-C10-C1	109.8(2)	C5-C10-H10A	111.3(17)
C1-C10-H10A	108.1(16)	C5-C10-H10B	109.6(17)
C1-C10-H10B	108.4(16)	H10A-C10-H10B	110.(2)
N2-C11-N1	126.1(2)	N2-C11-S1	118.37(18)
N1-C11-S1	115.6(2)	N1-C12-C13	106.7(2)
N1-C12-C17	109.3(2)	C13-C12-C17	109.8(2)
N1-C12-H12	108.9(16)	C13-C12-H12	109.7(16)
C17-C12-H12	112.2(16)	C14-C13-C12	104.5(2)
C14-C13-H13A	110.6(17)	C12-C13-H13A	111.8(17)
C14-C13-H13B	108.6(14)	C12-C13-H13B	111.8(16)
H13A-C13-H13B	109.(2)	O1-C14-N2	109.89(19)
O1-C14-C13	109.7(2)	N2-C14-C13	109.6(2)
O1-C14-C15		N2-C14-C15	109.4(2)
C13-C14-C15		O2-C15-O3	105.6(2)
O2-C15-C14	109.4(2)	O3-C15-C14	110.3(2)
O2-C15-H15	109.0(16)	O3-C15-H15	110.1(15)
C14-C15-H15	112.3(16)	O3-C16-C17	103.4(2)
O3-C16-H16A	108.8(18)	C17-C16-H16A	113.3(18)
O3-C16-H16B	107.3(18)	C17-C16-H16B	113.5(19)
H16A-C16-H16B	110.(2)	O2-C17-C16	100.6(2)
O2-C17-C12	107.8(2)	C16-C17-C12	113.1(3)
O2-C17-H17	108.9(18)	C16-C17-H17	113.3(18)
C12-C17-H17	112.2(19)		

Table 6. Anisotropic atomic displacement parameters (Å^2) for compound 10.

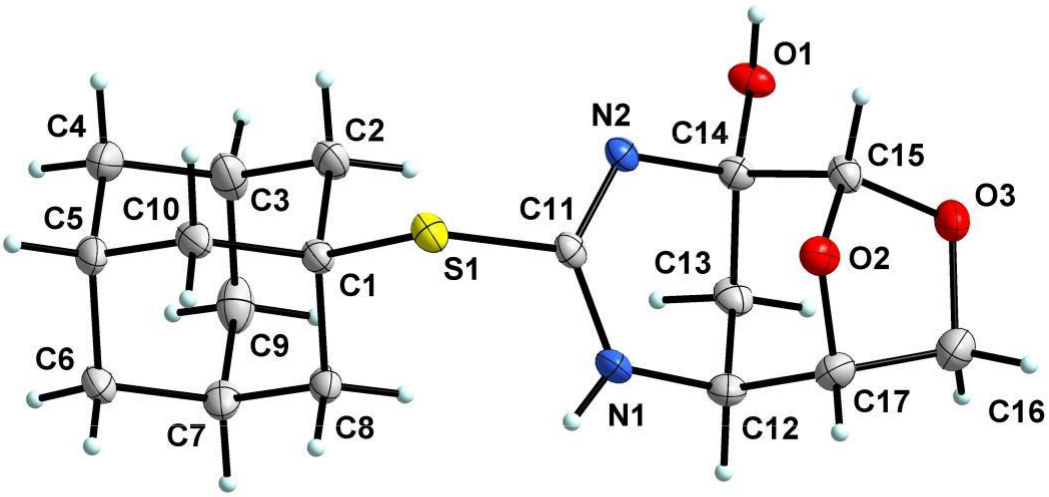
The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

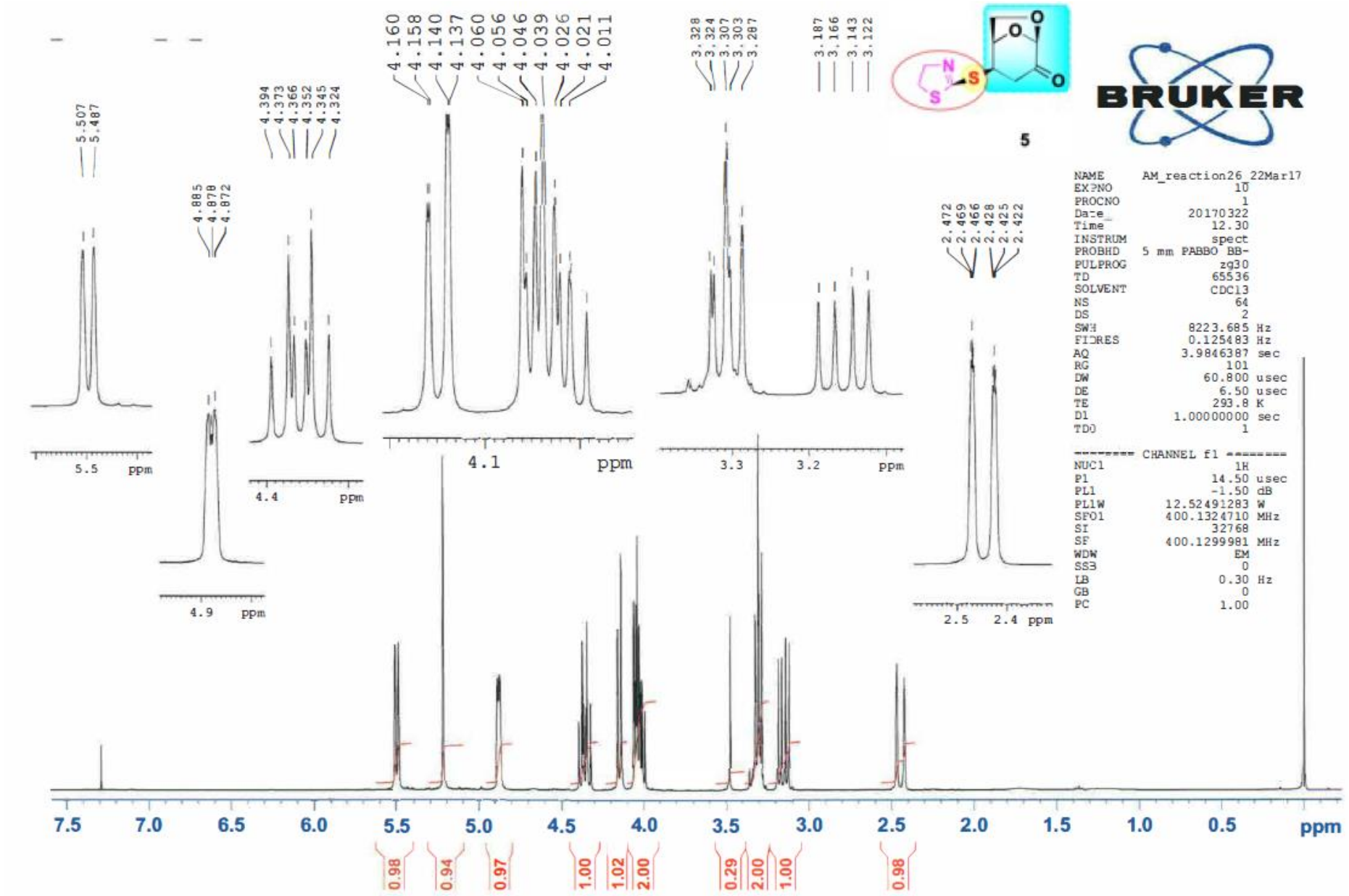
	U_{11}				U_{13}	U_{12}
S1	0.0188(4)	0.0169(4)	0.0201(3)	-0.0029(2)	-0.0033(2)	0.0030(3)
O1	0.0149(10)	0.0263(11)	0.0182(8)	-0.0076(8)	-0.0010(7)	0.0013(8)
O2	0.0206(10)	0.0185(10)	0.0222(8)	-0.0020(7)	0.0016(8)	-0.0028(9)
O3	0.0326(12)	0.0203(10)	0.0204(8)	0.0020(8)	-0.0003(8)	0.0074(9)
N1	0.0220(14)	0.0188(13)	0.0198(10)	-0.0012(9)	0.0049(9)	0.0070(11)
N2	0.0152(12)	0.0127(11)	0.0171(9)	-0.0030(8)	-0.0003(8)	0.0001(9)
C1	0.0180(14)	0.0147(14)	0.0159(10)	-0.0012(9)	-0.0004(10)	0.0023(11)
C2	0.0197(14)	0.0192(15)	0.0263(13)	0.0012(11)	0.0037(11)	0.0046(13)
C3	0.0212(16)	0.0184(15)	0.0391(15)	0.0027(11)	0.0053(12)	0.0019(14)
C4	0.042(2)	0.0187(16)	0.0251(13)	0.0029(11)	0.0101(12)	0.0031(14)
C5	0.0358(18)	0.0176(15)	0.0165(11)	0.0029(10)	-0.0001(11)	0.0086(13)
C6	0.0325(19)	0.0149(15)	0.0199(12)	-0.0019(10)	0.0019(11)	0.0061(13)
C7	0.0335(18)	0.0179(15)	0.0144(11)	-0.0013(10)	-0.0028(10)	0.0030(13)
C8	0.0235(16)	0.0147(14)	0.0148(10)	0.0008(10)	-0.0019(10)	0.0027(12)
C9	0.0332(19)	0.0176(16)	0.0316(14)	0.0011(11)	-0.0098(12)	-0.0027(14)
C10	0.0271(17)	0.0223(15)	0.0147(11)	-0.0008(10)	-0.0025(10)	0.0092(13)
C11	0.0174(14)	0.0110(13)	0.0183(11)	-0.0024(9)	-0.0001(9)	-0.0005(11)
C12	0.0271(16)	0.0197(14)	0.0145(11)	-0.0017(10)	0.0029(11)	0.0031(12)
C13	0.0239(16)	0.0178(14)	0.0171(11)	-0.0053(10)	-0.0028(11)	0.0014(13)
C14	0.0152(13)	0.0166(14)	0.0161(11)	-0.0025(9)	-0.0006(10)	-0.0001(11)
C15	0.0206(15)	0.0154(14)	0.0177(12)	-0.0020(10)	-0.0024(10)	0.0029(12)
C16	0.0416(19)	0.0234(17)	0.0238(13)	0.0049(12)	0.0057(13)	0.0025(15)
C17	0.0274(17)	0.0225(16)	0.0203(12)	0.0019(11)	0.0057(12)	-0.0006(13)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for compound 10

	x/a	y/b	z/c	U(eq)
H1N	0.616(3)	0.200(3)	0.3732(12)	0.021(8)
H1O	0.244(3)	0.373(3)	0.4660(14)	0.036(9)
H3	0.283(3)	-0.135(3)	0.4774(13)	0.040(9)
H2A	0.374(3)	0.041(3)	0.4566(11)	0.021(7)
H2B	0.404(2)	0.007(2)	0.5182(11)	0.019(7)
H5	0.601(3)	-0.268(3)	0.5167(12)	0.030(8)
H4A	0.425(2)	-0.198(3)	0.5457(11)	0.021(7)

	x/a	y/b	z/c	U(eq)
H4B	0.401(3)	-0.299(3)	0.5019(11)	0.026(8)
H7	0.521(2)	-0.187(2)	0.3541(10)	0.020(7)
H6A	0.549(3)	-0.326(3)	0.4232(12)	0.032(9)
H6B	0.660(3)	-0.244(3)	0.4215(12)	0.027(8)
H8A	0.643(3)	-0.040(2)	0.3933(10)	0.020(7)
H8B	0.515(2)	0.011(3)	0.3790(12)	0.021(7)
H9A	0.341(3)	-0.116(3)	0.3838(11)	0.027(8)
H9B	0.354(3)	-0.244(3)	0.4036(11)	0.023(8)
H10A	0.685(3)	-0.088(2)	0.4918(10)	0.014(7)
H10B	0.593(2)	-0.070(2)	0.5411(11)	0.023(7)
H12	0.515(2)	0.283(2)	0.3068(11)	0.018(7)
H13A	0.326(3)	0.345(3)	0.3335(12)	0.027(8)
H13B	0.348(2)	0.216(3)	0.3586(10)	0.017(7)
H15	0.401(2)	0.506(2)	0.4533(10)	0.008(6)
H17	0.613(3)	0.448(3)	0.3393(11)	0.025(8)
H16A	0.412(3)	0.494(3)	0.2948(12)	0.028(8)
H16B	0.487(3)	0.600(3)	0.3224(12)	0.036(9)

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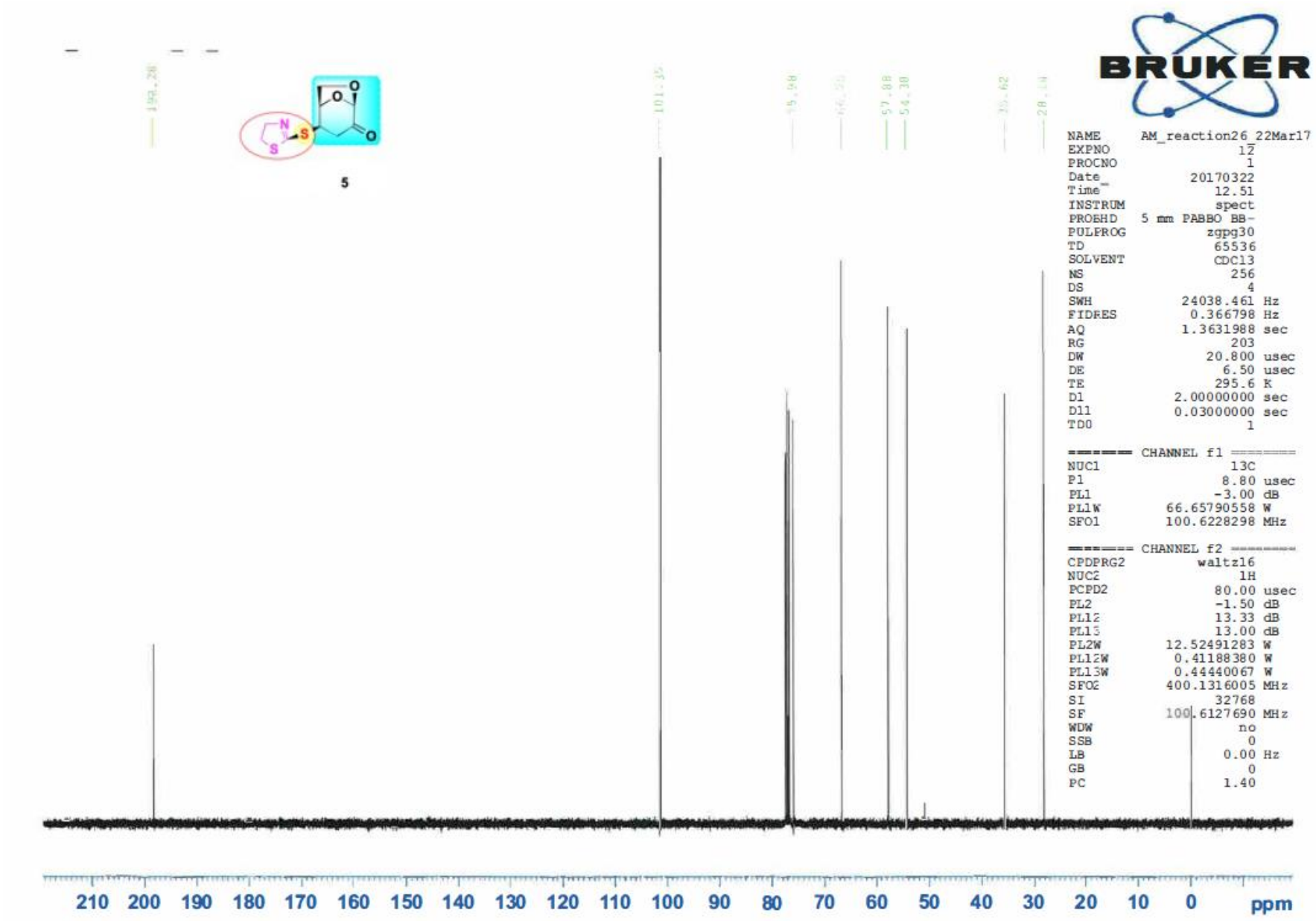
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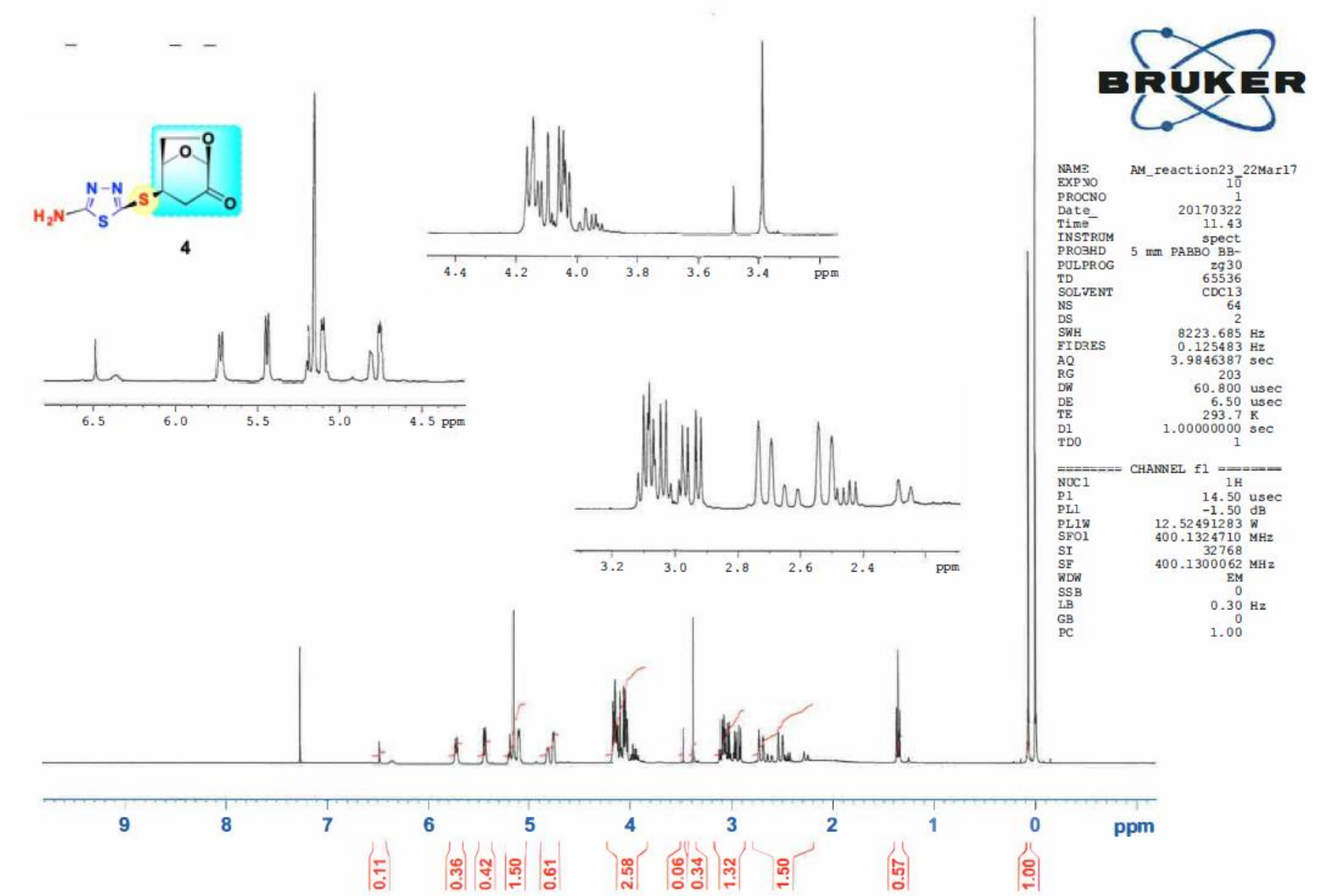
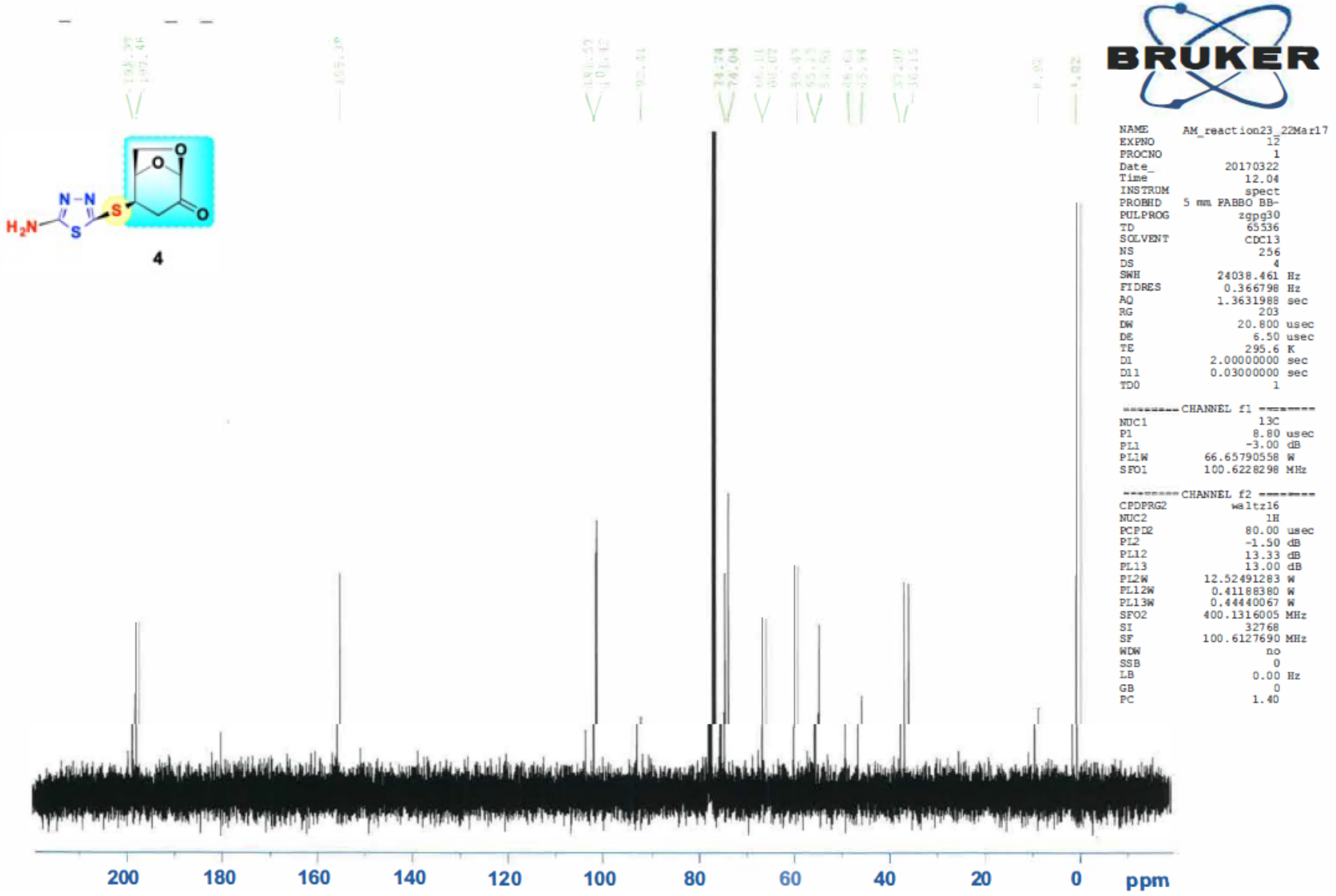
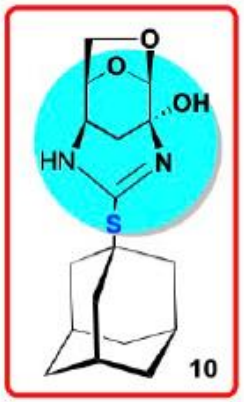
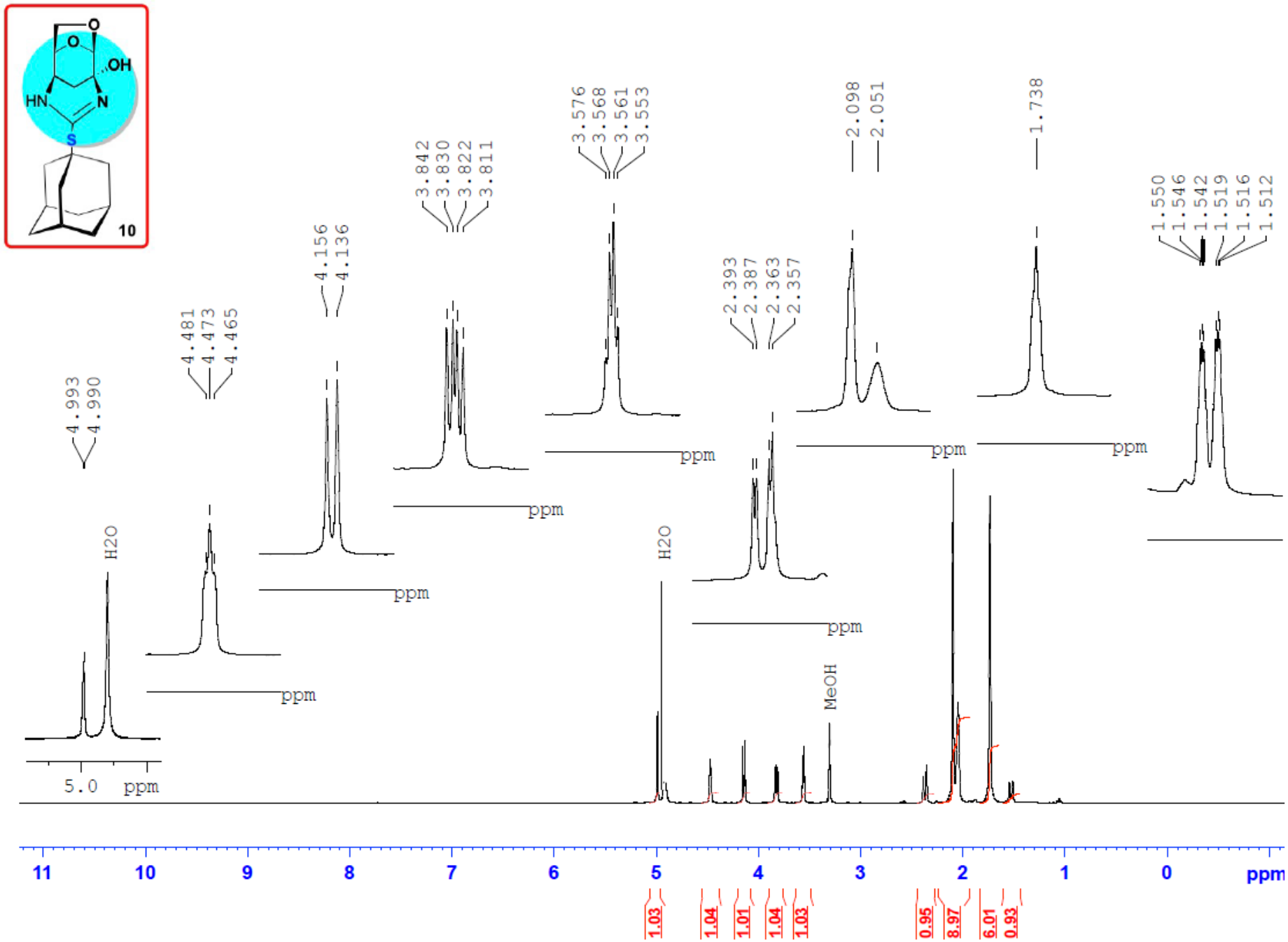
PL1 -3.00 dB
PL1W 66.65790558 W
SFO1 100.6228298 MHz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.50 dB
PL12 13.33 dB
PL13 13.00 dB
PL2W 12.52491283 W
PL12W 0.41188380 W
PL13W 0.44440067 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

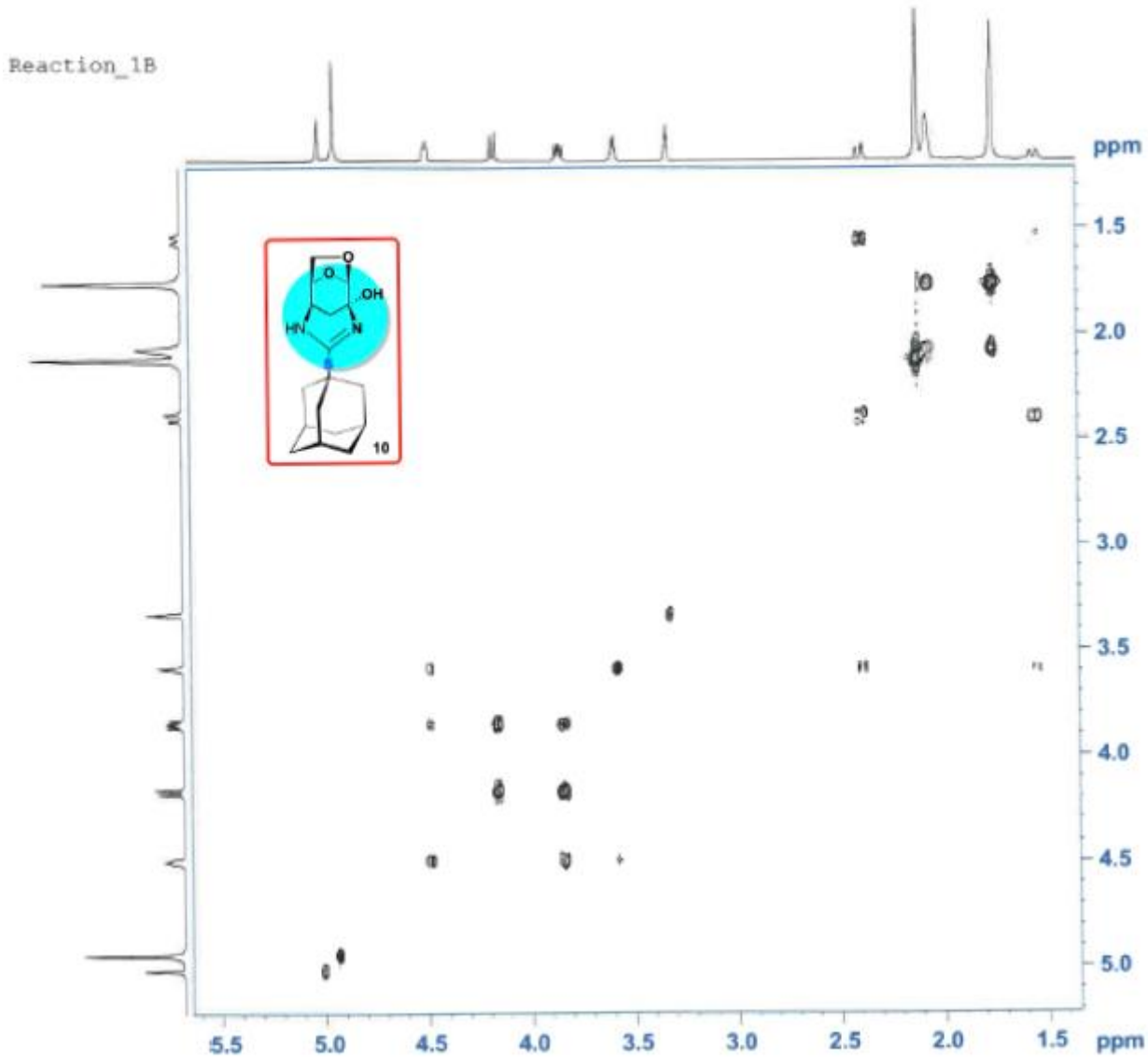

4
6.5 6.0 5.5 5.0 4.5 ppm
4.4 4.2 4.0 3.8 3.6 3.4 ppm
3.2 3.0 2.8 2.6 2.4 ppm
9 8 7 6 5 4 3 2 1 0 ppm
0.11 0.36 0.42 1.50 0.61 2.58 0.06 0.34 1.32 1.50 0.57 1.00
BRUKER
NAME AM_reaction23_22Mar17
EXPNO 10
PROCNO 1
Date_ 20170322
Time 11.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.50 usec
PL1 -1.50 dB
PL1W 12.52491283 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

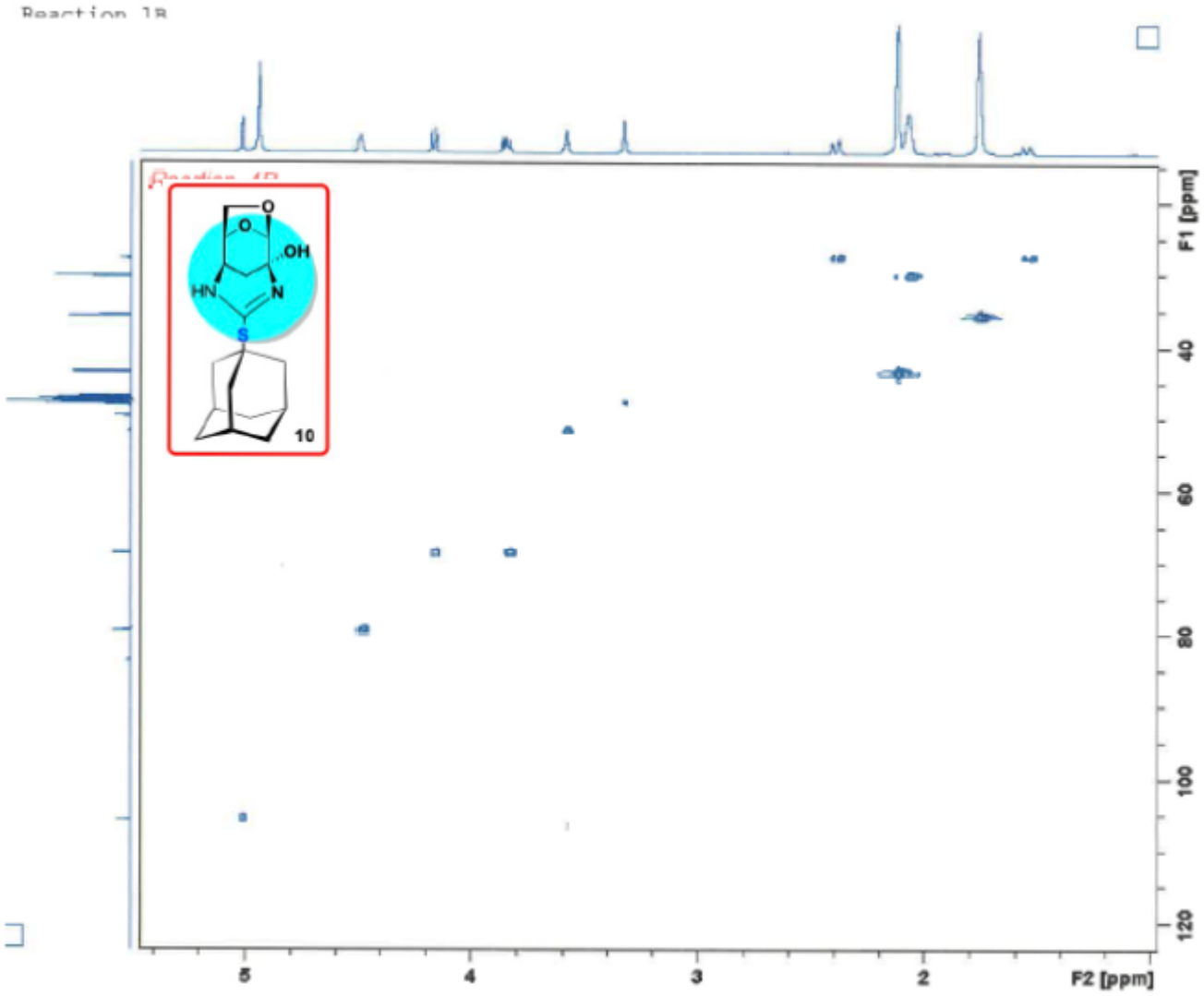

BRUKER
4
NAME AM_reaction23_22Mar17
EXPNO 12
PROCNO 1
Date_ 20170322
Time 12.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.80 usec
PL1 -3.00 dB
PL1W 66.65790558 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.50 dB
PL12 13.33 dB
PL13 13.00 dB
PL2W 12.52491283 W
PL12W 0.41188380 W
PL13W 0.44440067 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

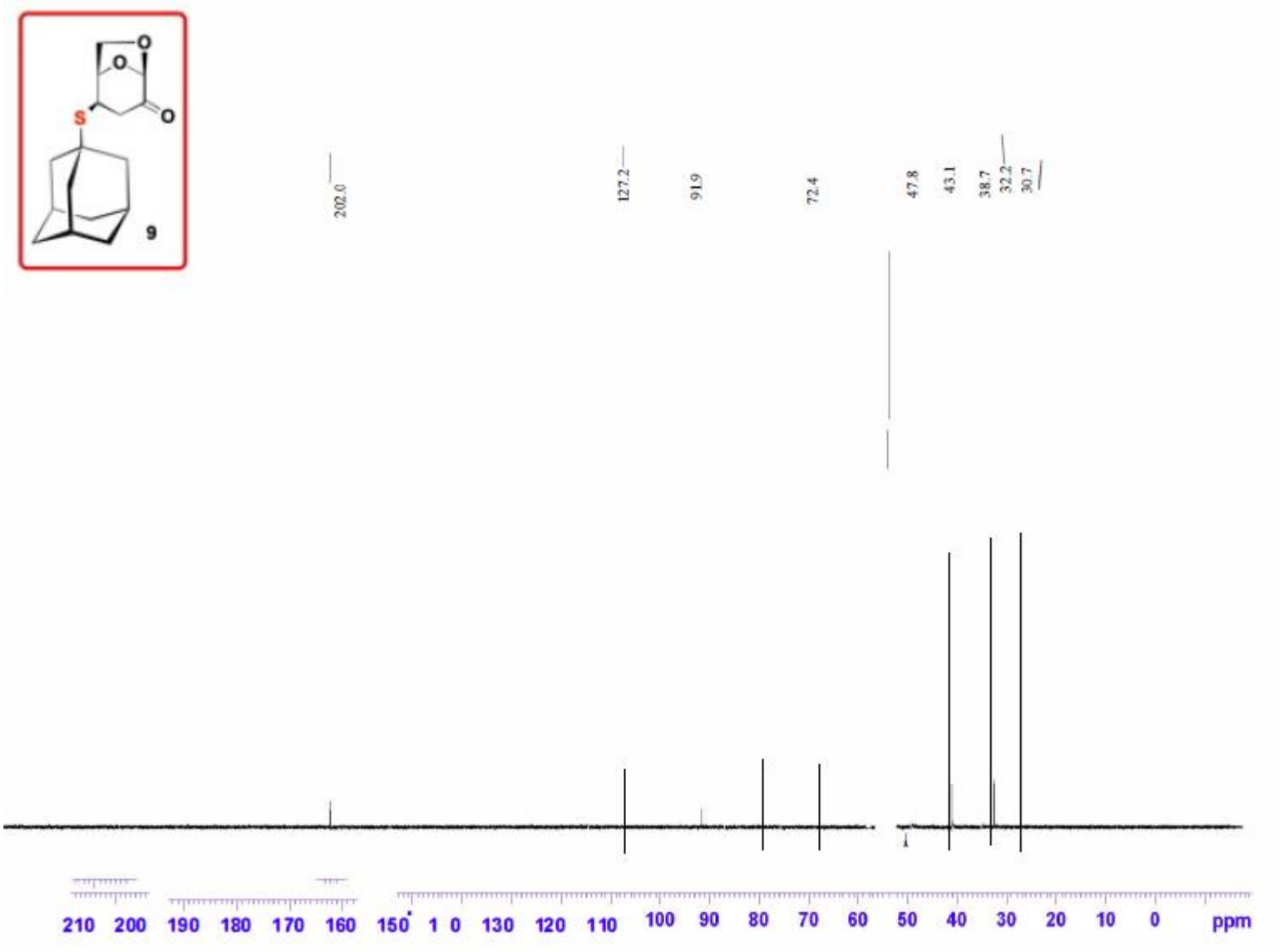

O
O
OH
HN
N
S
10

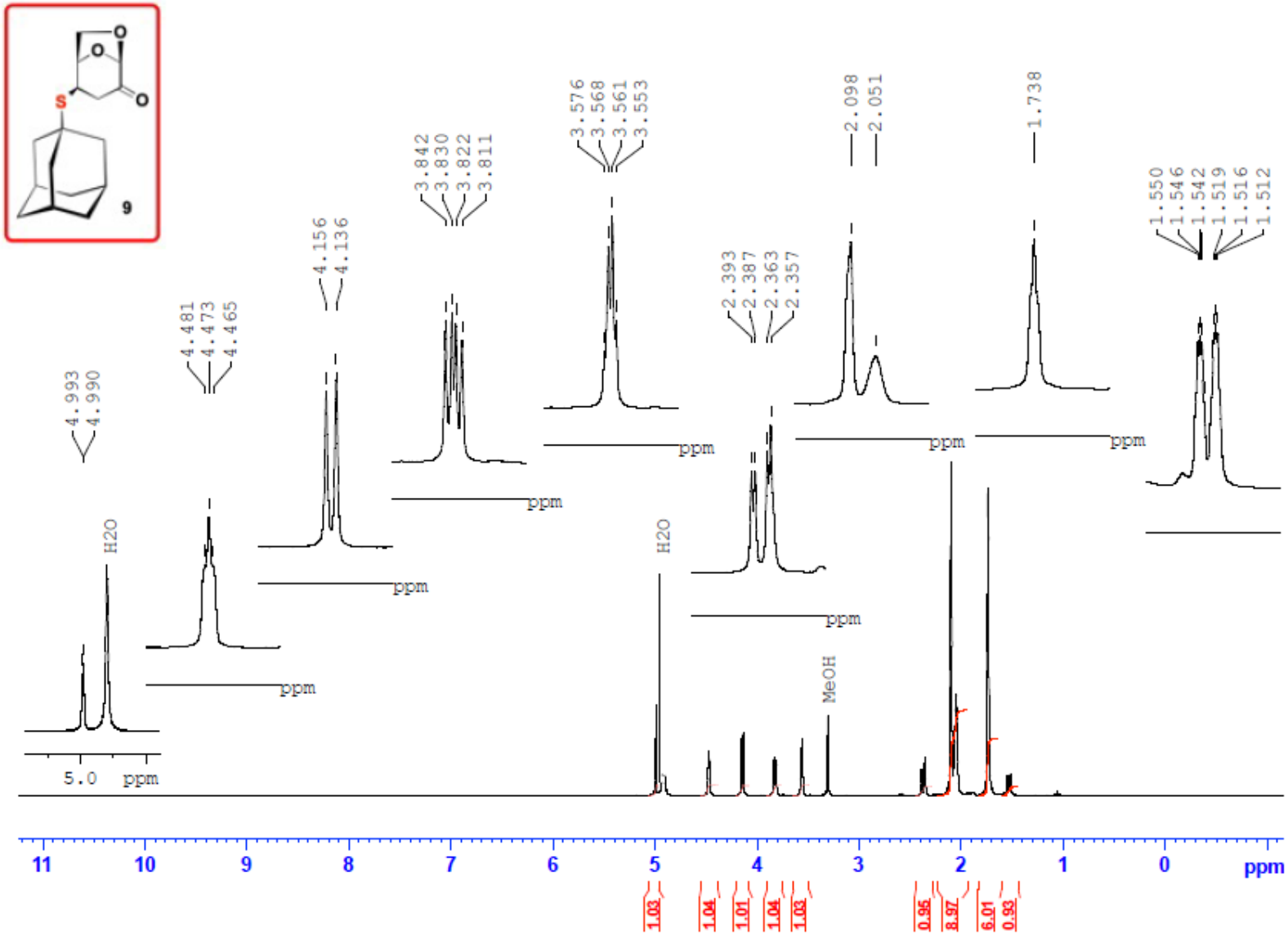
155.07
105.70
83.72
79.52
79.50
68.73
51.92
49.72
43.72
35.87
30.32
27.86
MeOD
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


O
O
OH
HN
N
S
10
4.993
4.990
4.481
4.473
4.465
4.156
4.136
3.842
3.830
3.822
3.811
3.576
3.568
3.561
3.553
2.393
2.387
2.363
2.357
2.098
2.051
1.738
1.550
1.546
1.542
1.519
1.516
1.512
H2O
H2O
MeOH
ppm
5.0
11
10
9
8
7
6
5
4
3
2
1
0
1.03
1.04
1.01
1.04
1.03
0.95
8.97
6.01
0.93


Reaction_1B
ppm
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
O
O
OH
HN
N
10




O
O
S
O
9
202.0
127.2
91.9
72.4
47.8
43.1
38.7
32.2
30.7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


9
4.993
4.990
4.481
4.473
4.465
4.156
4.136
3.842
3.830
3.822
3.811
3.576
3.568
3.561
3.553
2.393
2.387
2.363
2.357
2.098
2.051
1.738
1.550
1.546
1.542
1.519
1.516
1.512
H2O
H2O
MeOH
ppm
5.0
11
10
9
8
7
6
5
4
3
2
1
0
1.03
1.04
1.01
1.04
1.03
0.96
8.97
6.01
0.93

