

**An unexpected product of the reaction between  
N-hydroxy-6-methyluracil-5-carboximidoyl chloride and thioureas**

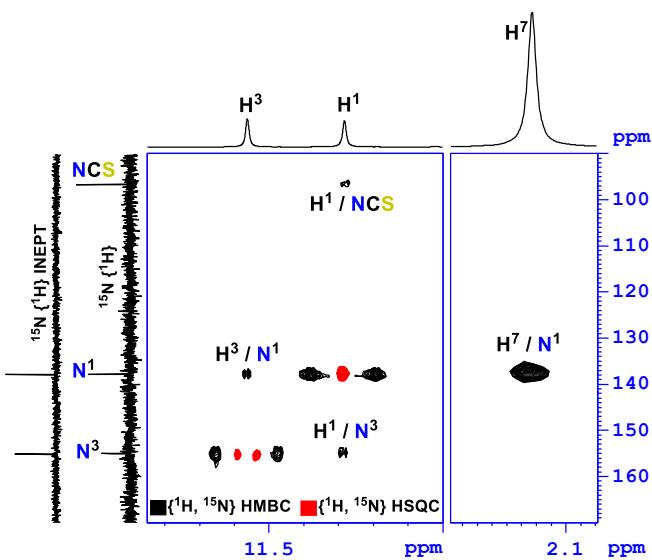
**Inna B. Chernikova, Alexander N. Lobov, Ekaterina S. Meshcheryakova,  
Leonard M. Khalilov and Marat S. Yunusov**

**EXPERIMENTAL SECTION**

<sup>1</sup>H and <sup>13</sup>C spectra were recorded on a Bruker Avance-III 500 MHz pulsed spectrometer with working frequencies of 500.13 MHz (<sup>1</sup>H), 125.76 MHz (<sup>13</sup>C) and 50.68 MHz (<sup>15</sup>N) using a 5 mm probe with PABBO Z-gradient at a constant sample temperature of 298K in DMSO-*d*<sub>6</sub>. Chemical shifts in the NMR spectra of <sup>1</sup>H, <sup>13</sup>C are given in ppm relative to the solvent signal ( $\delta_H$  2.50 and  $\delta_C$  39.52). Chemical shifts for <sup>15</sup>N nuclei are obtained from the <sup>15</sup>N{<sup>1</sup>H} spectrum with reverse discontinuous decoupling from protons and <sup>15</sup>N{<sup>1</sup>H} INEPT spectrum, and are confirmed by correlations in {<sup>1</sup>H, <sup>15</sup>N} HSQC and HMBC spectra, values are given in the ammonia scale. For <sup>15</sup>N nuclei, the calibration procedure was used according to the  $\chi$ -standard [S1,S2]. The mass-spectra were recorded on a high-resolution Agilent LC/QTOF 6530 time-of-flight liquid chromatomass spectrometer, electrospray ionization (IER) and chromatographic separation (eluent acetonitrile/water = 1:1 and 0.1% formic acid, flow rate 0.25 ml min<sup>-1</sup>, chromatographic column C18 Zorbax Extend-C18, 2.1 mm, 1.8  $\mu$ m) the capillary potential is 4 kV (the speed and temperature of the drying gas (nitrogen) is 10 dm<sup>3</sup> min<sup>-1</sup> and 325 °C, the pressure of the spraying gas is 4.2 atm). IR spectra of substances were taken on the device Shimadzu IR Prestige-21 in Nujol in the range of 400-4000 cm<sup>-1</sup>. The elemental analysis was performed on the EURO-3000 device. The melting point is determined in a glass capillary.

**The general method.** To a homogeneous mixture of compound **1** (0.10 g, 0.50 mmol) and **2a-f** (1.00 mmol) in MeOH (2.0 ml), one portion of Et<sub>3</sub>N (0.14 ml, 1.00 mmol) was added, and this was stirred at room temperature or at 0 °C for 1 hour. The precipitate was filtered, washed with MeOH, distilled water, dried (Table 1 of this article).

**5-Isothiocyanato-6-methylpyrimidine-2,4(1H,3H)-dione **3**.** White needles, m.p. >300 °C (MeOH), decomp. <sup>1</sup>H NMR,  $\delta$ , ppm: 2.12 s (3H, H<sup>7</sup>), 11.28 s (1H, H<sup>1</sup>), 11.56 s (1H, H<sup>3</sup>). <sup>13</sup>C{<sup>1</sup>H} NMR,  $\delta$ , ppm: 15.92 (C<sup>7</sup>), 105.19 (C<sup>5</sup>), 144.94 (N=C=S), 148.11 (C<sup>6</sup>), 149.47 (C<sup>2</sup>), 160.29 (C<sup>4</sup>). <sup>15</sup>N{<sup>1</sup>H} NMR,  $\delta$ , ppm: 95.53 (N=C=S), 137.73 (N<sup>1</sup>), 154.91 (N<sup>3</sup>). Mass-spectrum, m/z (%): 182.0022 (100) [M-H]<sup>-</sup> C<sub>6</sub>H<sub>4</sub>N<sub>3</sub>O<sub>2</sub>S. IR spectrum,  $\nu$ , cm<sup>-1</sup> (in vaseline oil) 1686-1737 (C=O), 1633-1653 (C=N). The data of the elemental analysis correspond to the formula C<sub>6</sub>H<sub>5</sub>N<sub>3</sub>O<sub>2</sub>S.



**Figure S1** Superposition of the  $\{^1\text{H}, ^{15}\text{N}\}$  HSQC and  $\{^1\text{H}, ^{15}\text{N}\}$  HMBC spectra of compound **3** in DMSO-d<sub>6</sub> solution.

**Table S1.** Crystal data and structure refinement for compound **3**.

Empirical formula	C <sub>6</sub> H <sub>5</sub> N <sub>3</sub> O <sub>2</sub> S
Formula weight	183.19
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	4.7912(2)
b/Å	7.2561(4)
c/Å	12.1180(6)
$\alpha/^\circ$	98.484(5)
$\beta/^\circ$	90.493(4)
$\gamma/^\circ$	106.926(5)
Volume/Å <sup>3</sup>	398.04(4)
Z	2
$\rho_{\text{calc}}/\text{cm}^3$	1.528
$\mu/\text{mm}^{-1}$	0.366
F(000)	188.0
Radiation	MoK $\alpha$ ( $\lambda = 0.71073$ )
2 $\Theta$ range for data collection/°	5.942 to 69.702
Index ranges	-7 $\leq$ h $\leq$ 7, -11 $\leq$ k $\leq$ 11, -19 $\leq$ l $\leq$ 19
Reflections collected	15221
Independent reflections	3283 [ $R_{\text{int}} = 0.0408$ ]
Data/restraints/parameters	3283/0/111
Goodness-of-fit on $F^2$	1.034
Final R indexes [ $I >= 2\sigma (I)$ ]	$R_1 = 0.0488$ , $wR_2 = 0.1306$
Final R indexes [all data]	$R_1 = 0.0712$ , $wR_2 = 0.1484$
Largest diff. peak/hole / e Å <sup>-3</sup>	0.45/-0.48

**Table S2.** Bond Lengths for compound 3.

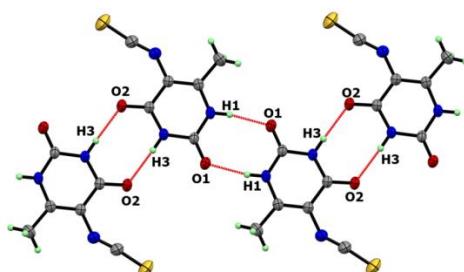
Bond	Length/Å	Bond	Length/Å
S1–C9	1.5666(15)	N3–C4	1.3758(15)
O2–C4	1.2329(14)	N8–C5	1.3807(16)
O1–C2	1.2282(14)	N8–C9	1.1853(18)
N1–C2	1.3652(15)	C4–C5	1.4452(17)
N1–C6	1.3711(16)	C6–C5	1.3632(17)
N3–C2	1.3681(14)	C6–C7	1.4885(18)

**Table S3.** Bond Angles for compound 3.

Bond Angle	Angle/°	Bond Angle	Angle/°
C2–N1–C6	124.12(10)	N3–C4–C5	115.01(10)
C2–N3–C4	125.84(10)	N1–C6–C7	116.46(11)
C9–N8–C5	141.66(13)	C5–C6–N1	118.83(11)
O1–C2–N1	122.77(10)	C5–C6–C7	124.69(12)
O1–C2–N3	121.82(11)	N8–C5–C4	117.87(11)
N1–C2–N3	115.40(10)	C6–C5–N8	121.44(11)
O2–C4–N3	121.35(11)	C6–C5–C4	120.64(11)
O2–C4–C5	123.59(11)	N8–C9–S1	173.51(13)

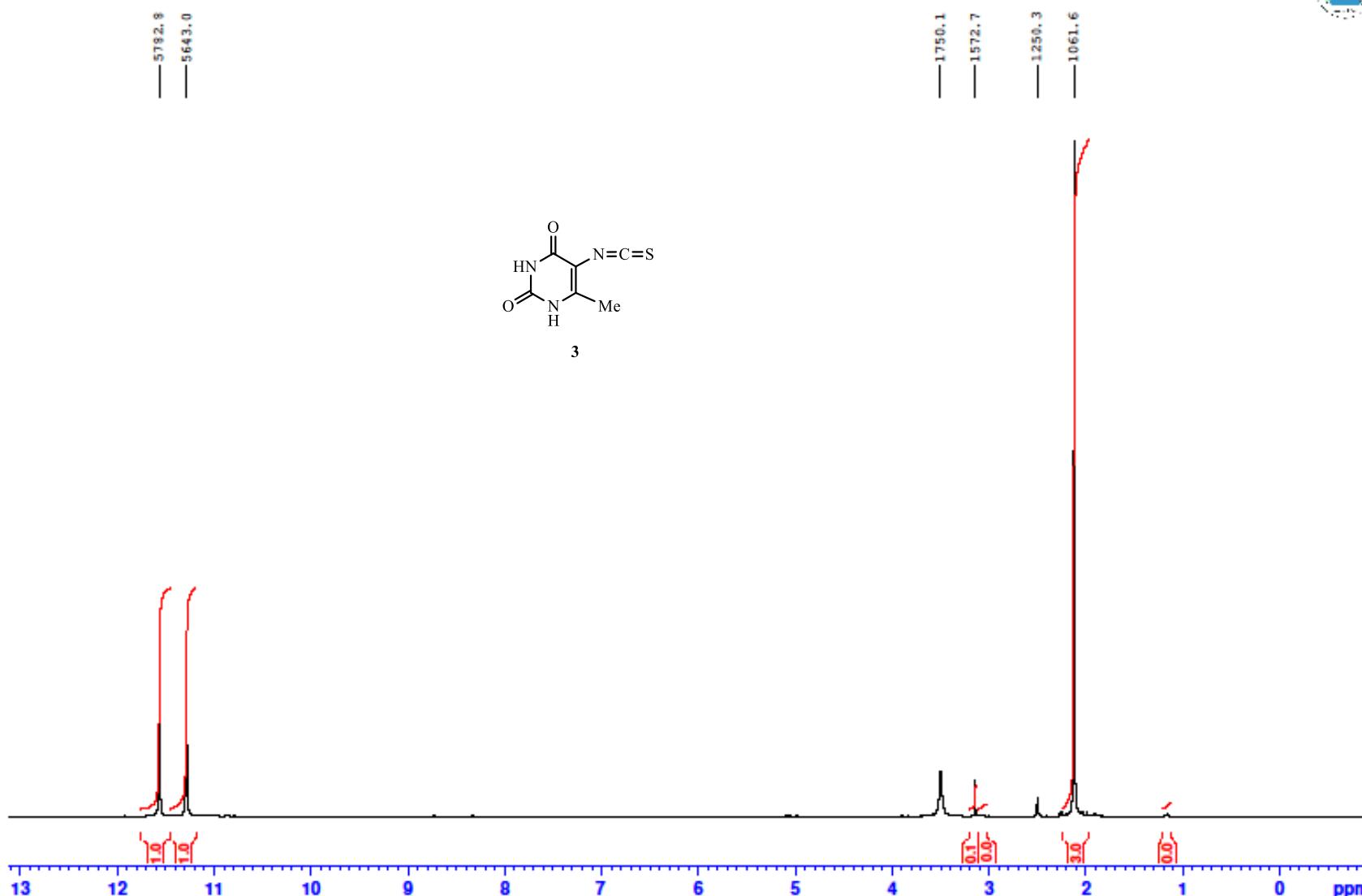
**Table S4.** Torsion Angles for compound 3.

Torsion Angle	Angle/°	Torsion Angle	Angle/°
O2–C4–C5–N8	2.95(19)	C2–N3–C4–C5	0.45(18)
O2–C4–C5–C6	-174.46(12)	C4–N3–C2–O1	178.25(12)
N1–C6–C5–N8	178.53(12)	C4–N3–C2–N1	-2.97(18)
N1–C6–C5–C4	-4.16(19)	C6–N1–C2–O1	-179.22(12)
N3–C4–C5–N8	-179.36(11)	C6–N1–C2–N3	2.01(17)
N3–C4–C5–C6	3.24(18)	C9–N8–C5–C4	10.7(3)
C2–N1–C6–C5	1.46(19)	C9–N8–C5–C6	-171.9(2)
C2–N1–C6–C7	-177.01(12)	C7–C6–C5–N8	-3.1(2)
C2–N3–C4–O2	178.20(12)	C7–C6–C5–C4	174.18(13)

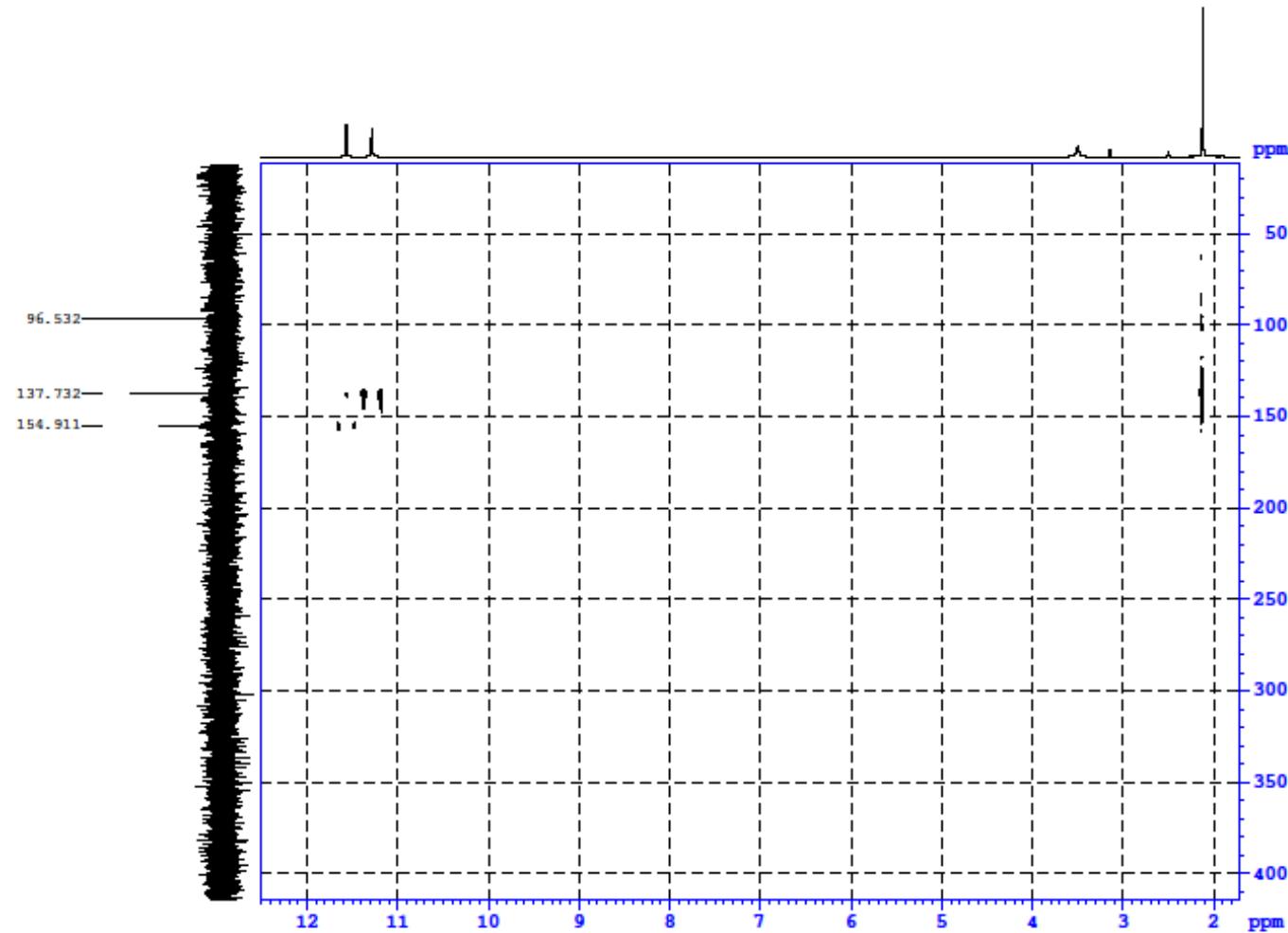
**Figure S2** Fragment of the crystal packaging of compound 3.

[S1] R. K. Harris, E. D. Becker, S. M. Cabral De Menezes, R. Goodfellow, P. Granger, *Pure Appl. Chem.*, 2001, **73**, 1795.

[S2] R. K. Harris, E. D. Becker, S. M. Cabral De Menezes, P. Granger, R. E. Hoffman, K. W. Zilm, *Pure Appl. Chem.*, 2008, **80**, 59.



<sup>1</sup>H NMR spectrum compound 3



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PROCNO 1

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SOLVENT DMSO  
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DS 16  
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AQ 0.300000 T  
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DR 6.50 t  
TR 298.0 F  
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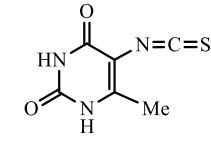
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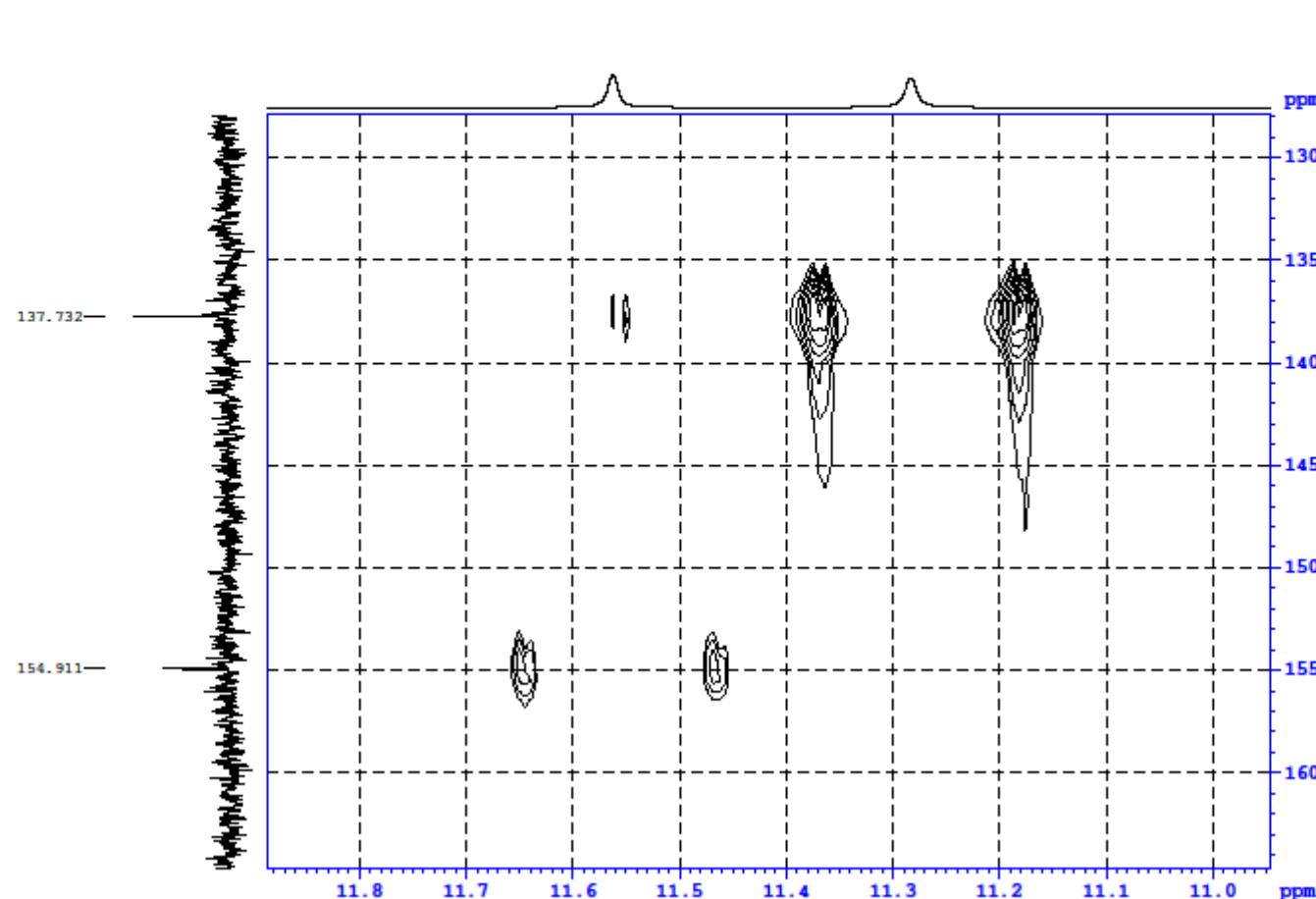
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F1 - Processing parameters  
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<sup>1</sup>H-<sup>15</sup>N HMBC NMR spectrum compound 3



Current Data Parameters  
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 EXPNO 21  
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 AQ 0.3400372 s  
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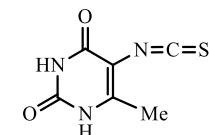
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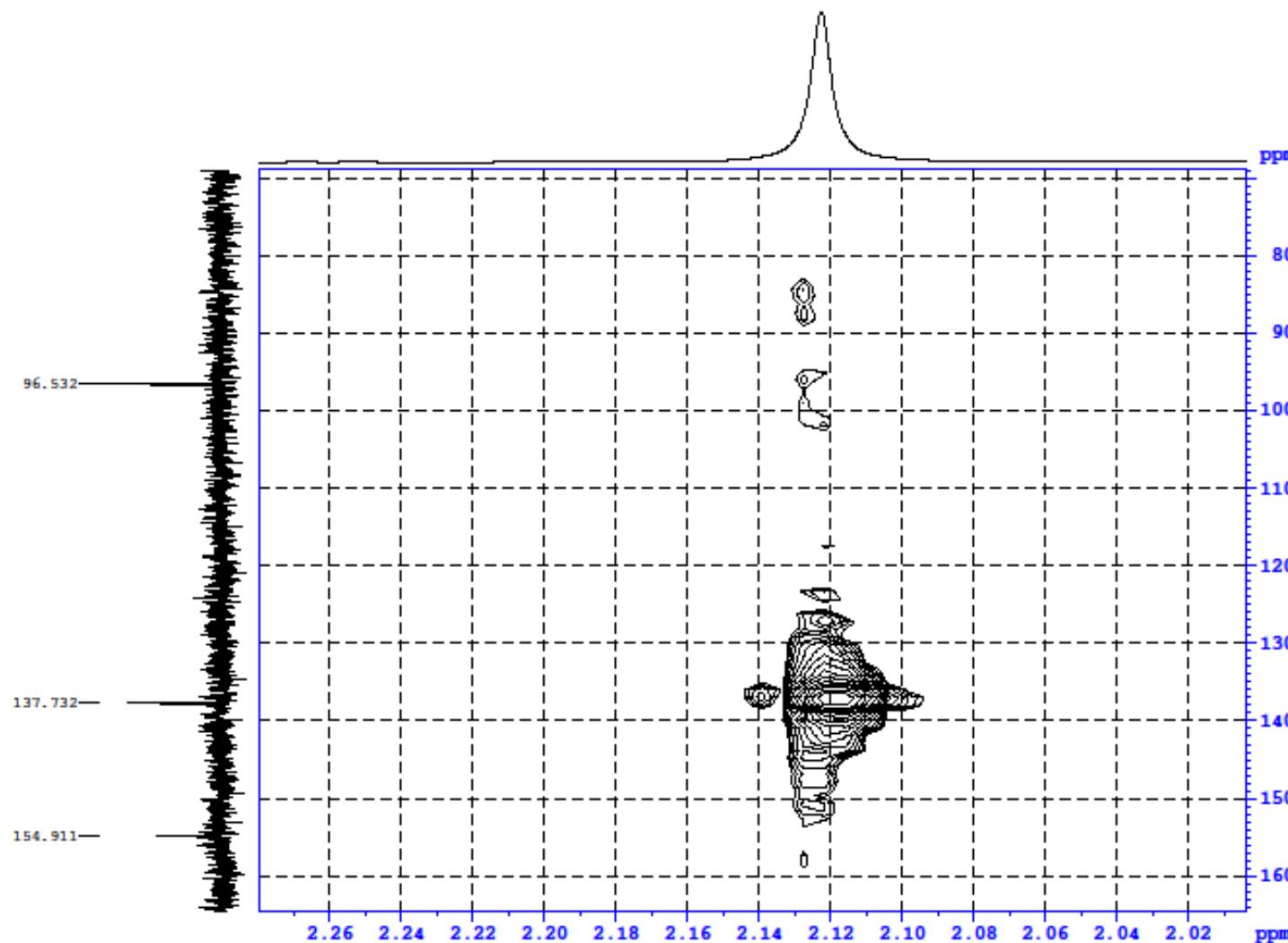
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F1 - Processing parameters  
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<sup>1</sup>H-<sup>15</sup>N HMBC NMR spectrum 1 of compound 3



Current Data Parameters  
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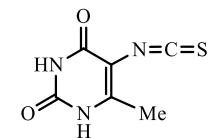
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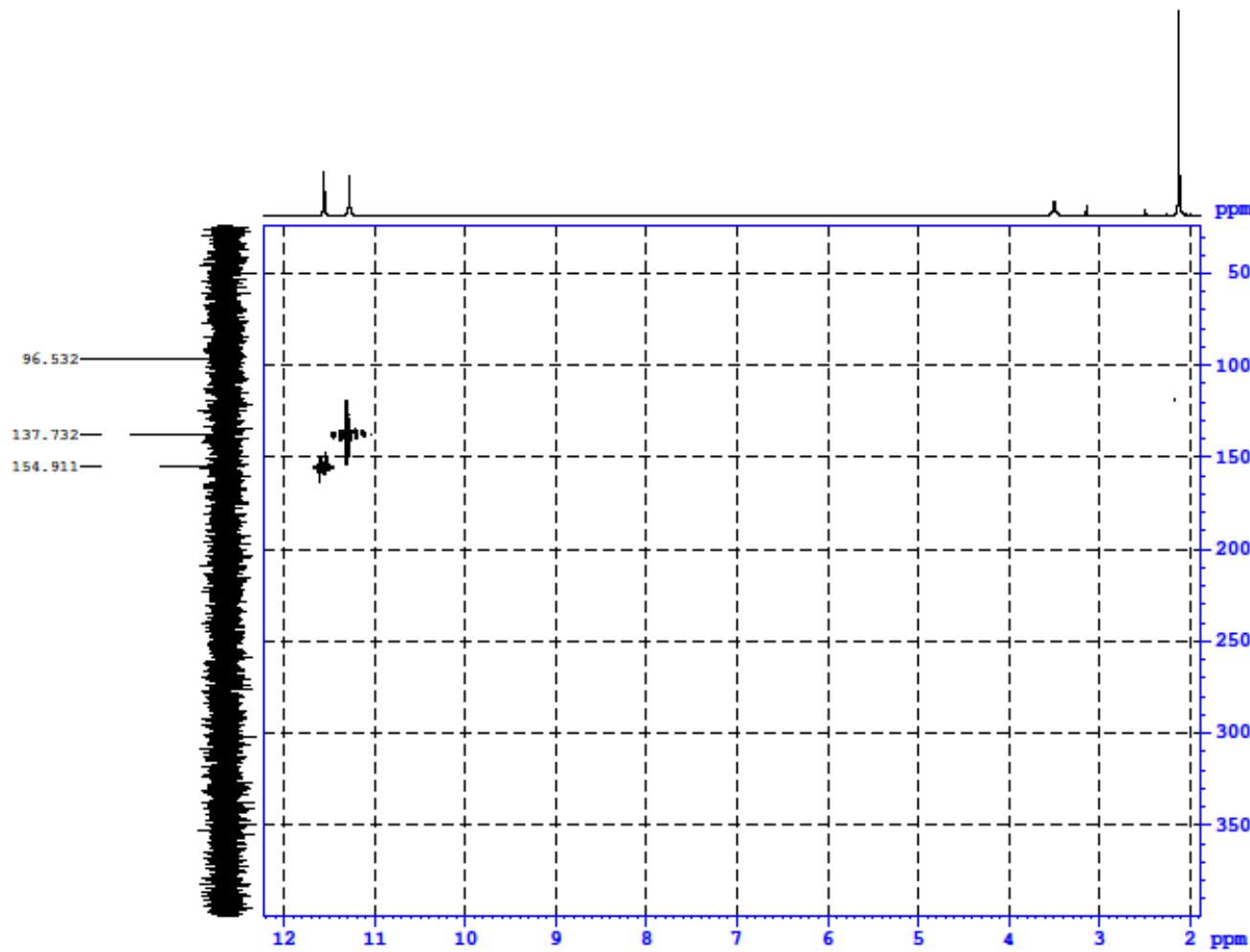
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<sup>1</sup>H-<sup>15</sup>N HMBC NMR spectrum 2 of compound 3

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 $\Delta\delta(1H)=12.02$ ppm;  $\Delta\delta(1H)=6.60$ ppm;  $\Delta\delta(15N)=499.98$ ppm;  $\Delta\delta(15N)=240.00$ ppm; Obs.Freq.: 500.13MHz; RF(15N)=50.60MHz; DI=0.4s; T=297.0K; Probe:BBQ; Exp.Time:13 min 26 sec; TimeDate: 09:23:53 23 May 2022.

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 AQ 0.3408372 sec  
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 TE 297.0 K  
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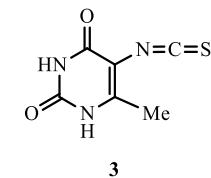
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 SFO2 50.6898957 MHz

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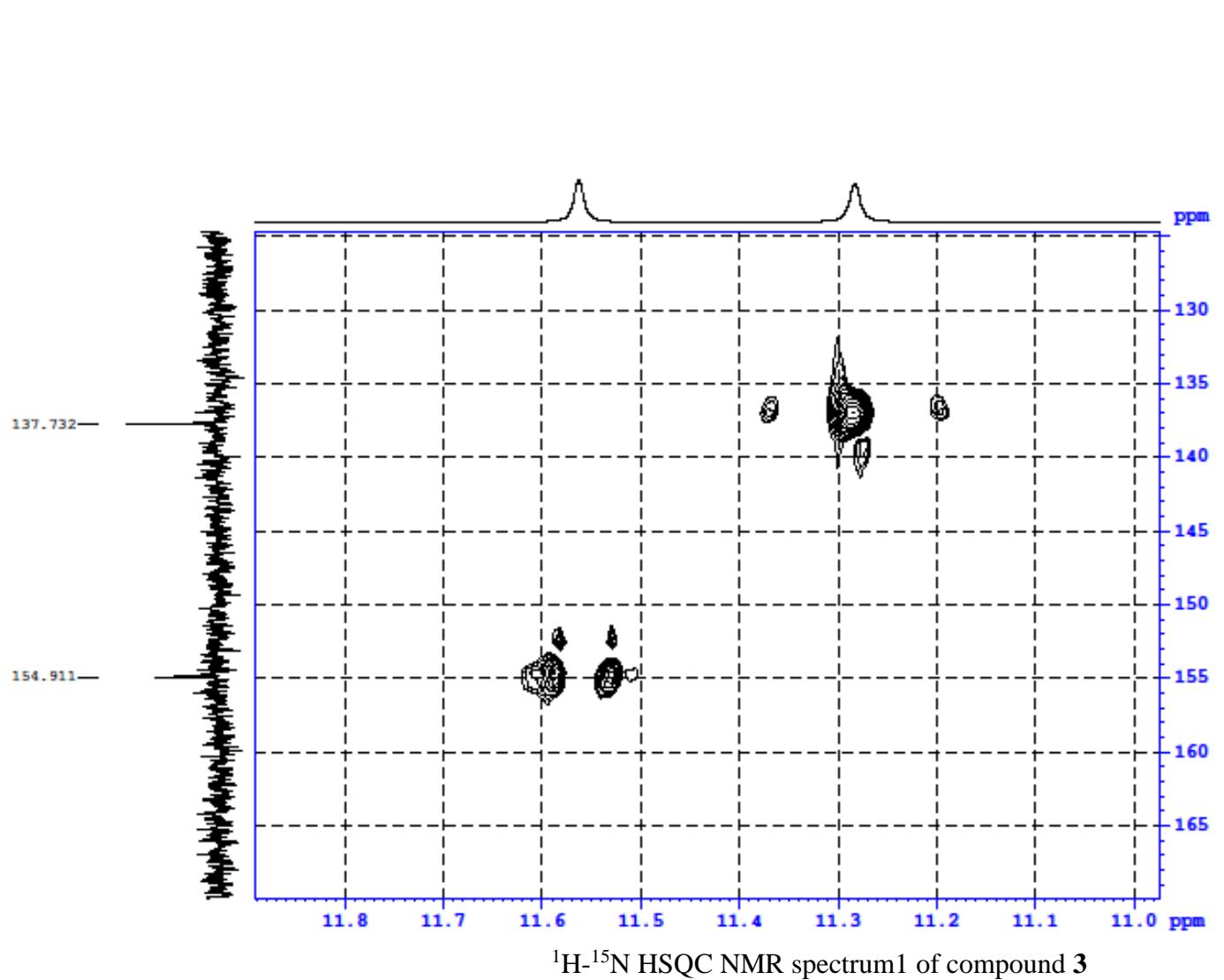
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$^1\text{H}$ - $^{15}\text{N}$  HSQC NMR spectrum of compound 3

Sp-957 Charnikova IB-957 180mg in DMSO, (1H, 15N) HSQC AV500 23.05.2022 LAN  
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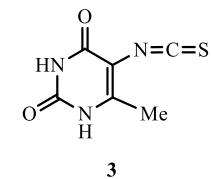
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 P2B 0 usc  
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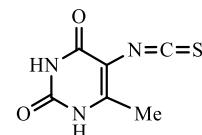
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 LB 0 Hz



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UIC RAS



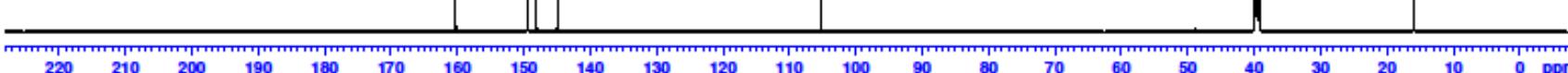
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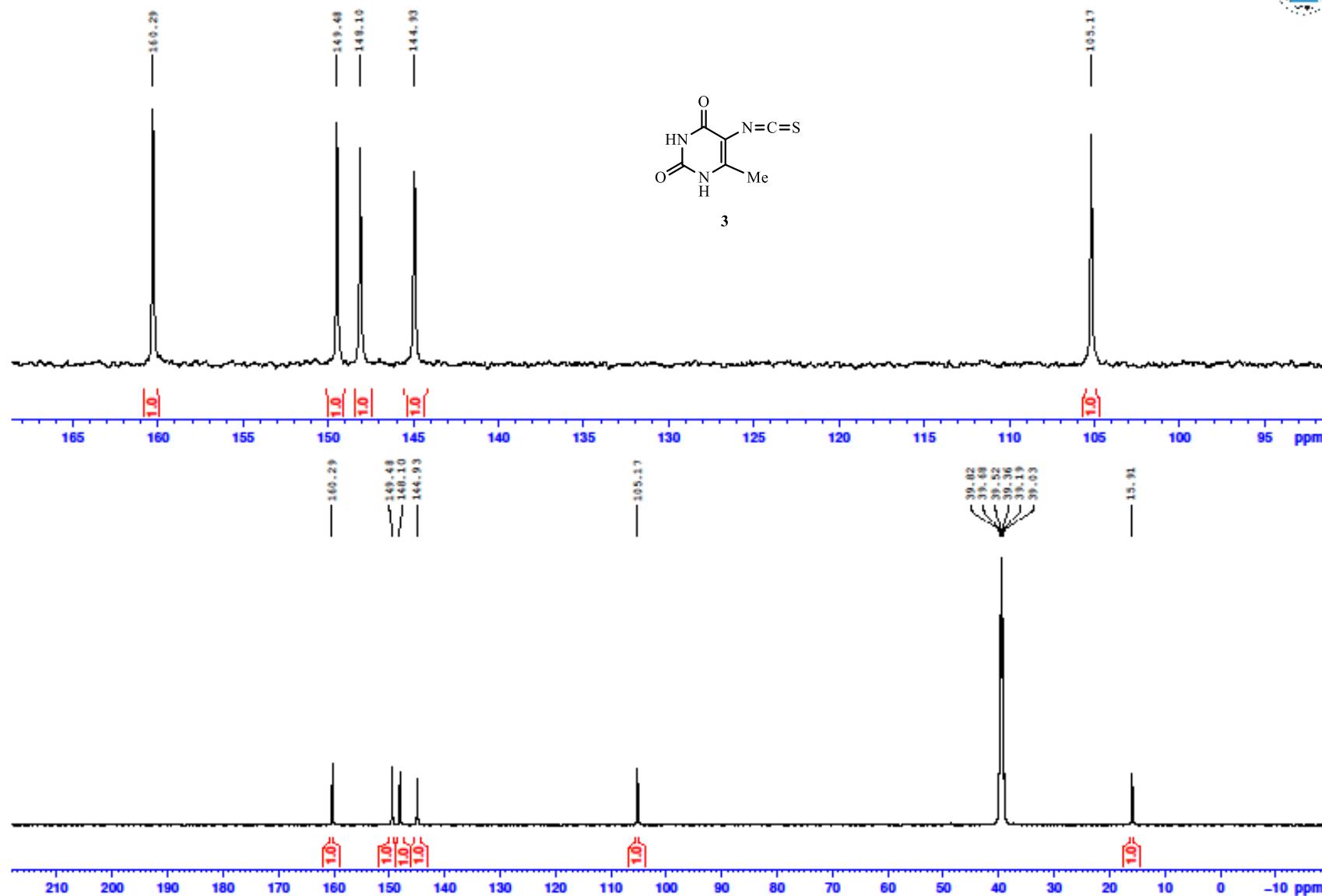
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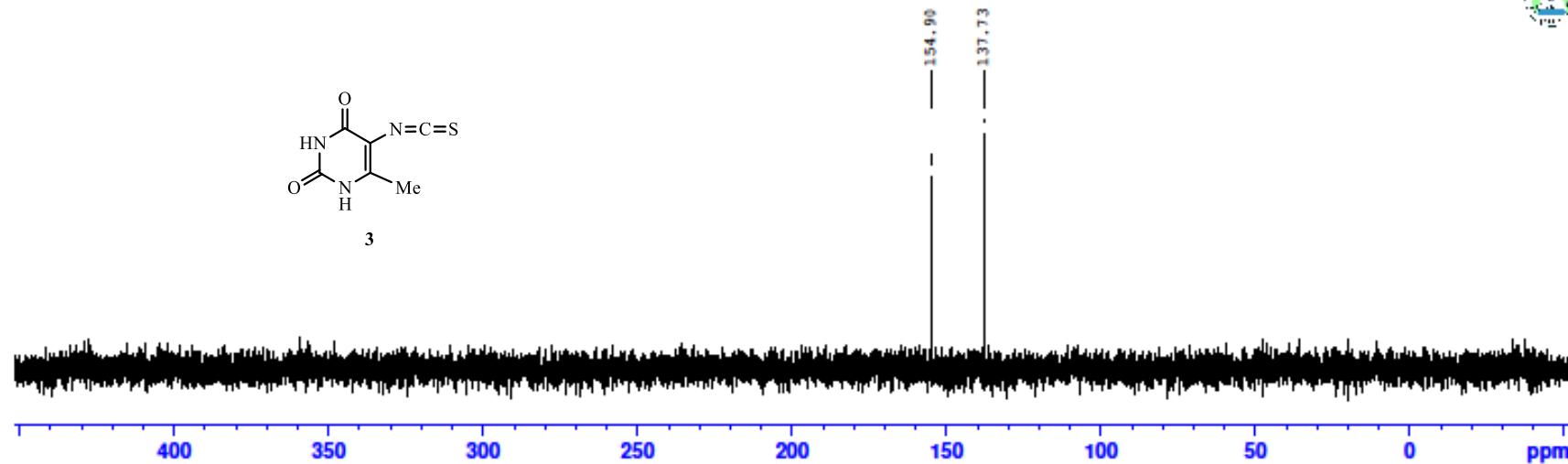
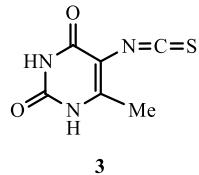
$^{13}\text{C}$  NMR spectrum of compound 3



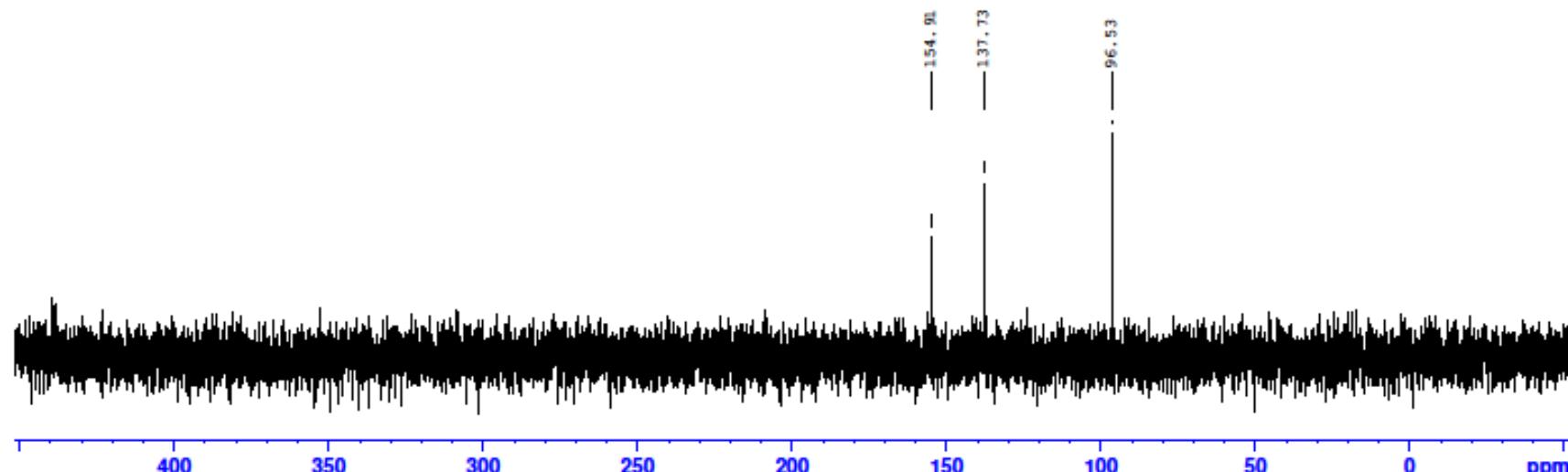
### <sup>13</sup>C NMR spectrum integrals for compound 3

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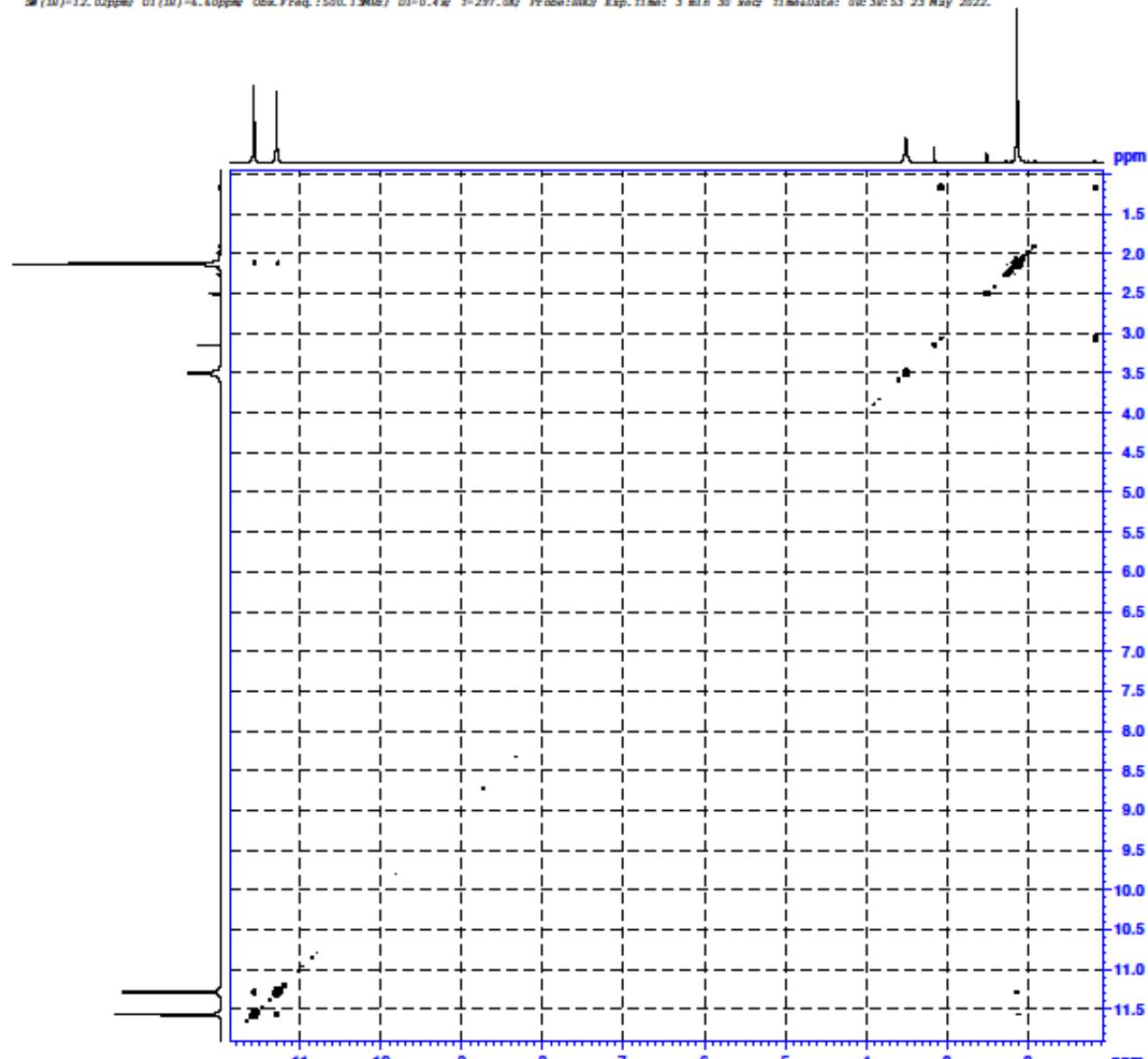
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Sp-957 Chernikova IB-957 180mg in DMSO,  $^{15}\text{N}$ ( $^1\text{H}$ ) inv.gated spectrum AV500 23.05.2022 LAN  
 $\Delta\text{f}$  ( $^{15}\text{N}$ )=503.20ppm  $\Delta\text{f}$  ( $^1\text{H}$ )=200.00ppm; Obs.Freq.:50.60MHz; DI=30.0s; T=296.5K; Probe:BBQ; Exp.Time: 34 min 47 sec; TimeDate: 10:07:53 23 May 2022.



$^{15}\text{N}$  NMR spectrum of compound 3



Current Data Parameters  
NAME vts-IB-957-DMSO  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Data\_ 20220523  
Time 0.38  
INSTRUM spect  
PROBHD 5 mm FABRO BB-  
PULPROG cosy90ppf  
TD 4096  
SOLVENT DMSO  
NS 1  
DS 16  
SWH 6009.615 Hz  
FIDRES 1.46715 Hz  
AC 0.3409372 sec  
RG 38.27  
DW 83.200 usec  
DE 6.50 usec  
TE 297.0 K  
D0 0.00000300 sec  
D1 0.40000001 sec  
D13 0.00000400 sec  
D16 0.00010000 sec  
INR 0.00016640 sec

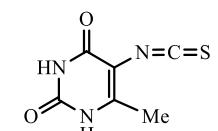
CHANNEL f1  
NUC1 1H  
F0 11.50 usec  
P1 11.50 usec  
P1W1 15.8489998 W  
SF01 500.1333009 MHz

GRADIENT CHANNEL  
GPNAM1 SINEQ10.100  
GPE1 10.00 %  
P1E 1000.00 usec

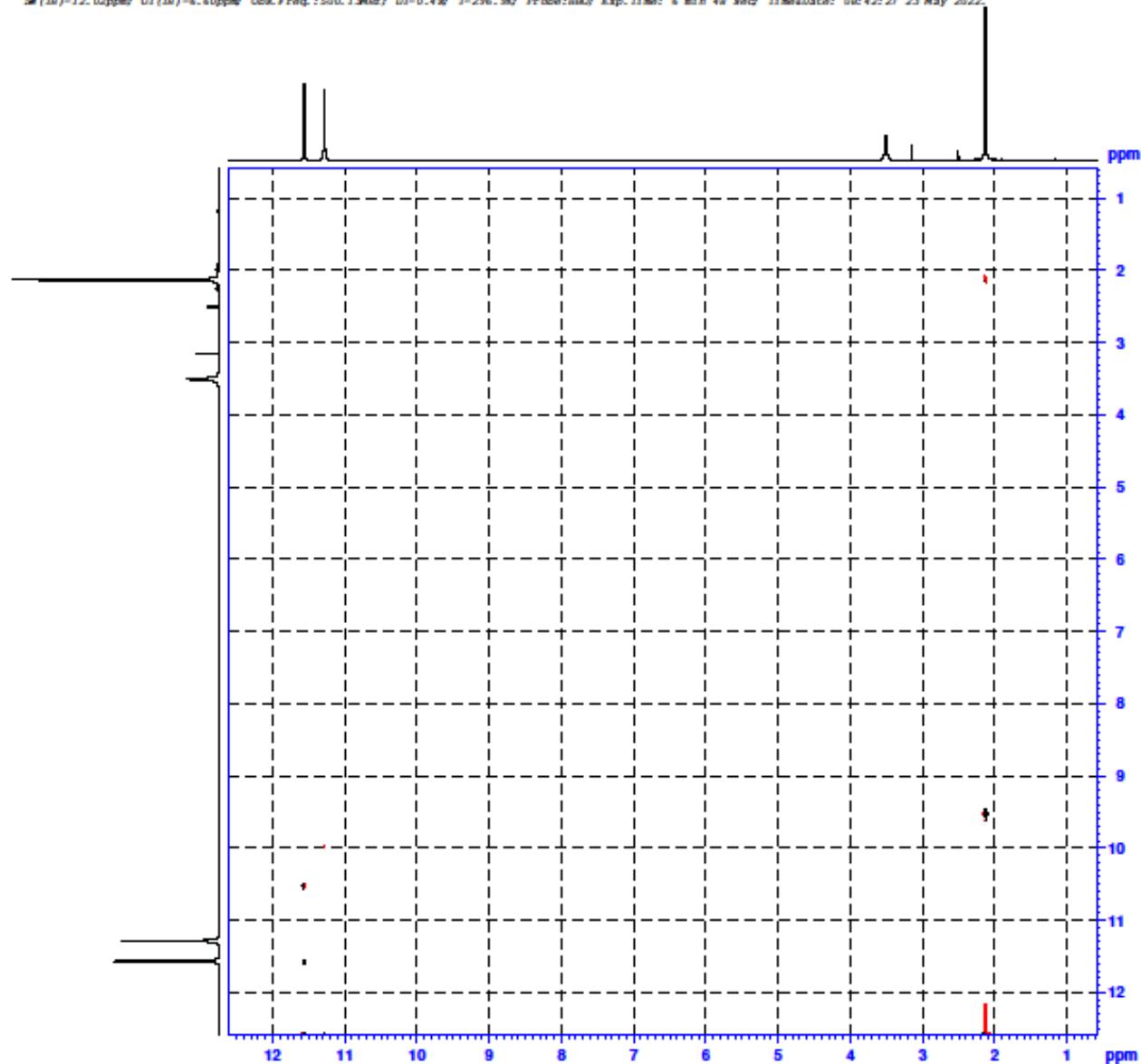
F1 - Acquisition parameters  
TD 256  
SF01 500.1333 MHz  
FIDRES 23.475060 Hz  
SW 12.016 ppm  
FnMDEK QF

F2 - Processing parameters  
SI 2048  
SF 500.1300053 MHz  
WNW QSIINE  
SSB 0 Hz  
LB 0 Hz  
GB 0  
PC 3.00

F1 - Processing parameters  
SI 2048  
MC2 QF  
SF 500.1300053 MHz  
WNW USER  
SSB 0 Hz  
LB 0 Hz  
GB 0



COSY NMR spectrum of compound 3



Current Data Parameters  
NAME vts-IB-957-DMSO  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20220523  
Time\_ 0.42  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG cosyqopenp1h  
TD 4096  
SOLVENT DMSO  
NS 1  
DS 16  
SWH 6009.615 Hz  
FIDRES 1.467191 Hz  
AQ 0.3408372 sec  
RG 201.51  
DW 83.200 usec  
DE 6.50 usec  
TE 296.9 K  
D1 0.00080001 sec  
D11 0.00000001 sec  
D12 0.00000400 sec  
D13 0.00010000 sec  
D14 0.00016640 sec  
IND 0

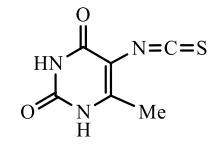
CHANNEL F1  
NUC1 1H  
P1 11.50 usec  
P2 23.00 usec  
P1M1 15.84899998 Hz  
SFO1 500.1333009 MHz

GRADIENT CHANNEL  
GPNAME1 SMDQ10.100  
GPNAME2 SMDQ10.100  
GP1 10.00 %  
GP2 20.00 %  
P16 1800.00 usec

F1 - Acquisition parameters  
TD 512  
SFO1 500.1333 MHz  
FIDRES 11.737536 Hz  
SW 12.016 ppm  
Pw10CR 7791

F2 - Processing parameters  
SI 2048  
SF 500.1300053 MHz  
WM QSIINE  
SSB 2  
LB 0 Hz  
GB 0  
PC 3.00

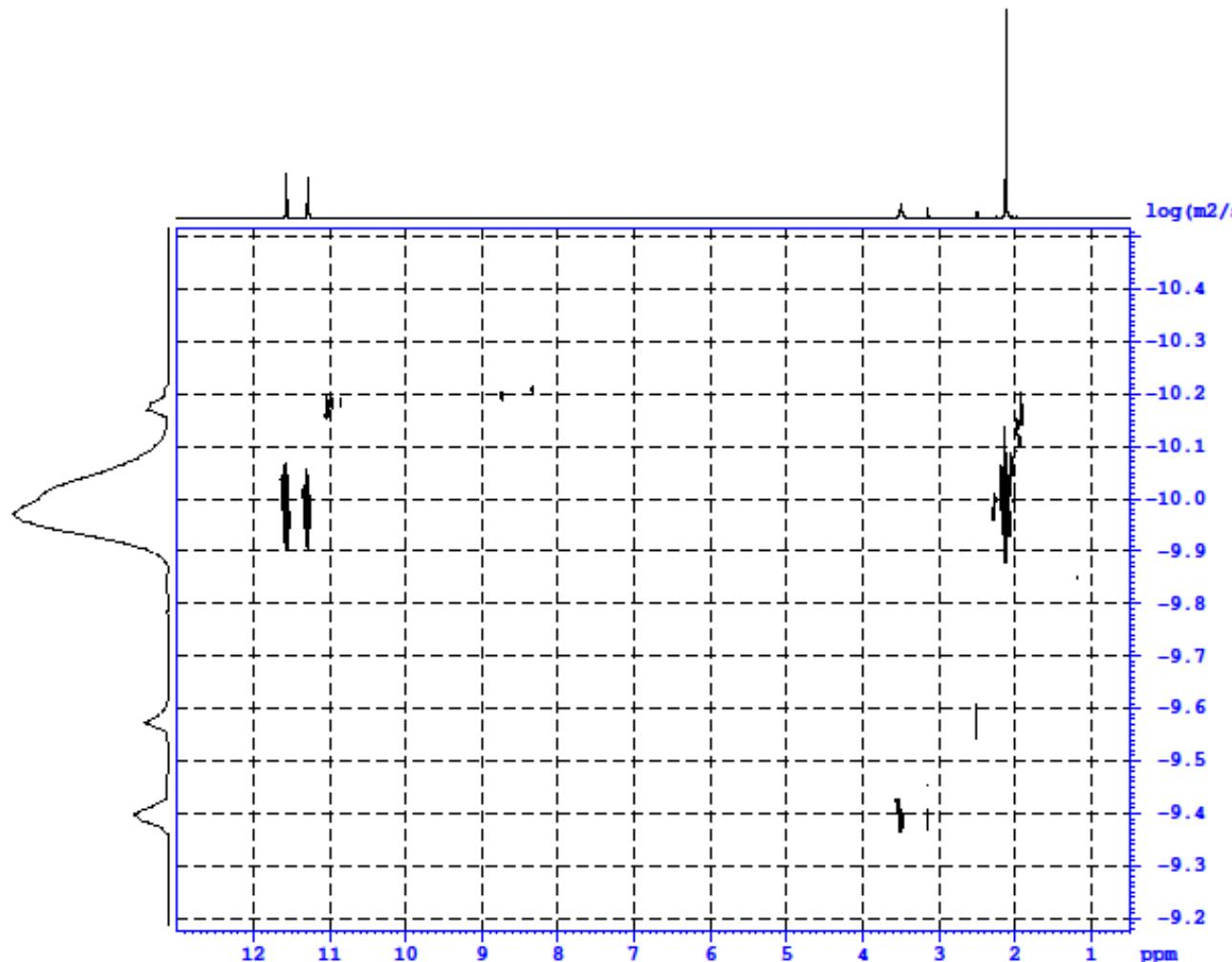
F1 - Processing parameters  
SI 2048  
MC2 TIP1  
SF 500.1300053 MHz  
WM 2  
SSB 0 Hz  
LB 0 Hz  
GB 0



COSY DQF NMR spectrum of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 1H) DOSY AV500 23.05.2022 LAN  
 $\Delta\delta$  (1H)=19.99ppm  $\Delta\delta$  (1H)=7.00ppm Obs.Freq.: 500.13MHz; DI=5.0x; T=296.4K; Probe:BBQ; Exp.Time: 6 min 20 sec; Time/Date: 10:48:27 23 May 2022.

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Current Data Parameters  
 NAME vta-IB-957-DMSO  
 EXPNO 61  
 PROCNO 1

R2 - Acquisition Parameter  
 Date 20220523  
 Time 10:48  
 INSTRUM spect  
 PROBHD 5 mm FABBO BB-  
 PULPROG 1eddpap2s  
 TD 8192  
 SOLVENT DMSO  
 NS 8  
 DS 4  
 SWH 10000.000 Hz  
 FIDRES 1.220703 Hz  
 AQ 0.4096500 sec  
 RG 56.39  
 DW 50.000 us  
 DE 6.50 us  
 TE 296.4 K  
 D1 5.00000000 sec  
 D16 0.00010000 sec  
 D20 0.05000000 sec  
 D21 0.00500000 sec

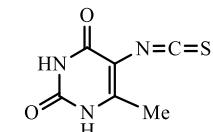
CHANNEL F1  
 NUC1 1H  
 P1 11.57 us  
 P2 23.14 us  
 P1M1 15.84899998 W  
 SF01 500.1335009 MHz

GRADIENT CHANNEL  
 GPNAM6 2M5Q10.100  
 GPNAM7 2M5Q10.100  
 GPNAM8 2M5Q10.100  
 GPZ6 100.00 %  
 GPZ7 -17.13 %  
 GPZ8 -13.17 %  
 P19 500.00 us  
 P30 2000.00 us

R1 - Acquisition parameter  
 TD 8  
 SF01 500.1335 MHz  
 FIDRES 1250.000000 Hz  
 SW 19.995 ppm  
 F1MODE QF

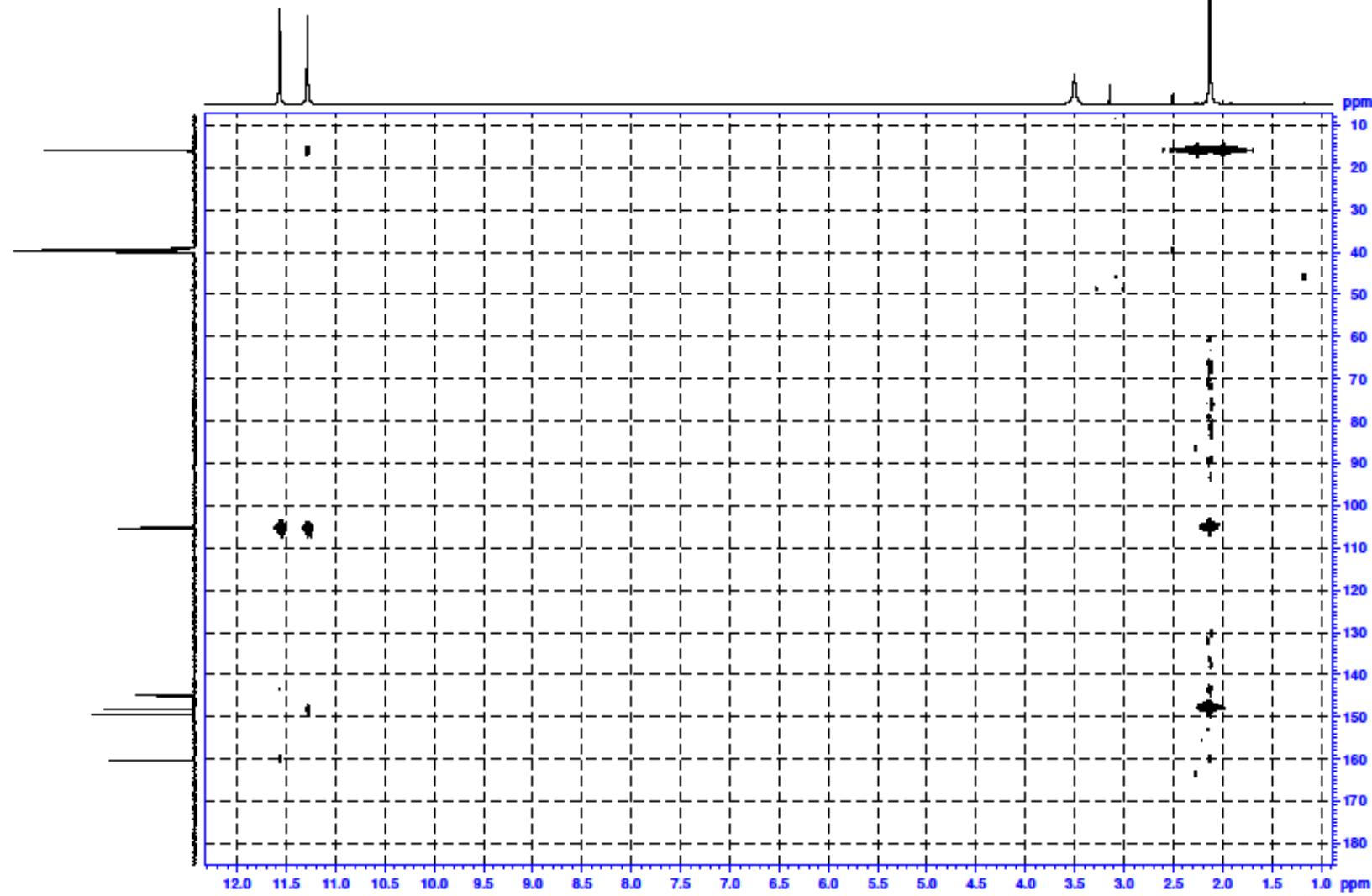
R2 - Processing parameters  
 SI 4096  
 SF 500.1300053 MHz  
 WMW 0 sec  
 SSB 0 Hz  
 LB 0 2.00 Hz  
 GB 0 3.00  
 PC

R1 - Processing parameters  
 SI 256  
 MC2 QF  
 SF 500.1300000 MHz  
 WMW 0 sec  
 SSB 0 Hz  
 LB 0 Hz  
 GB 0

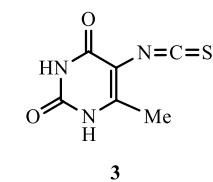
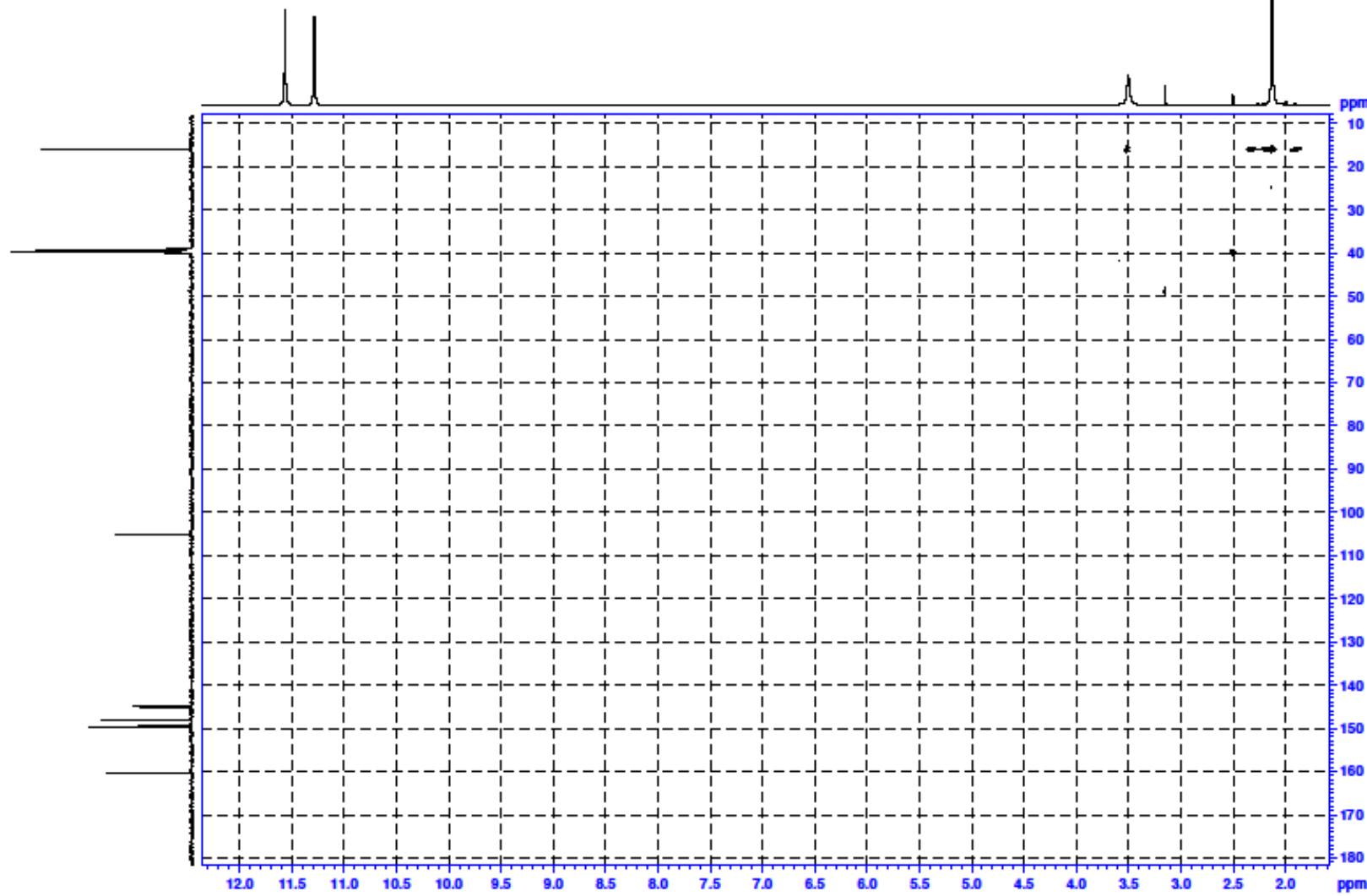


DOSY NMR spectrum of compound 3

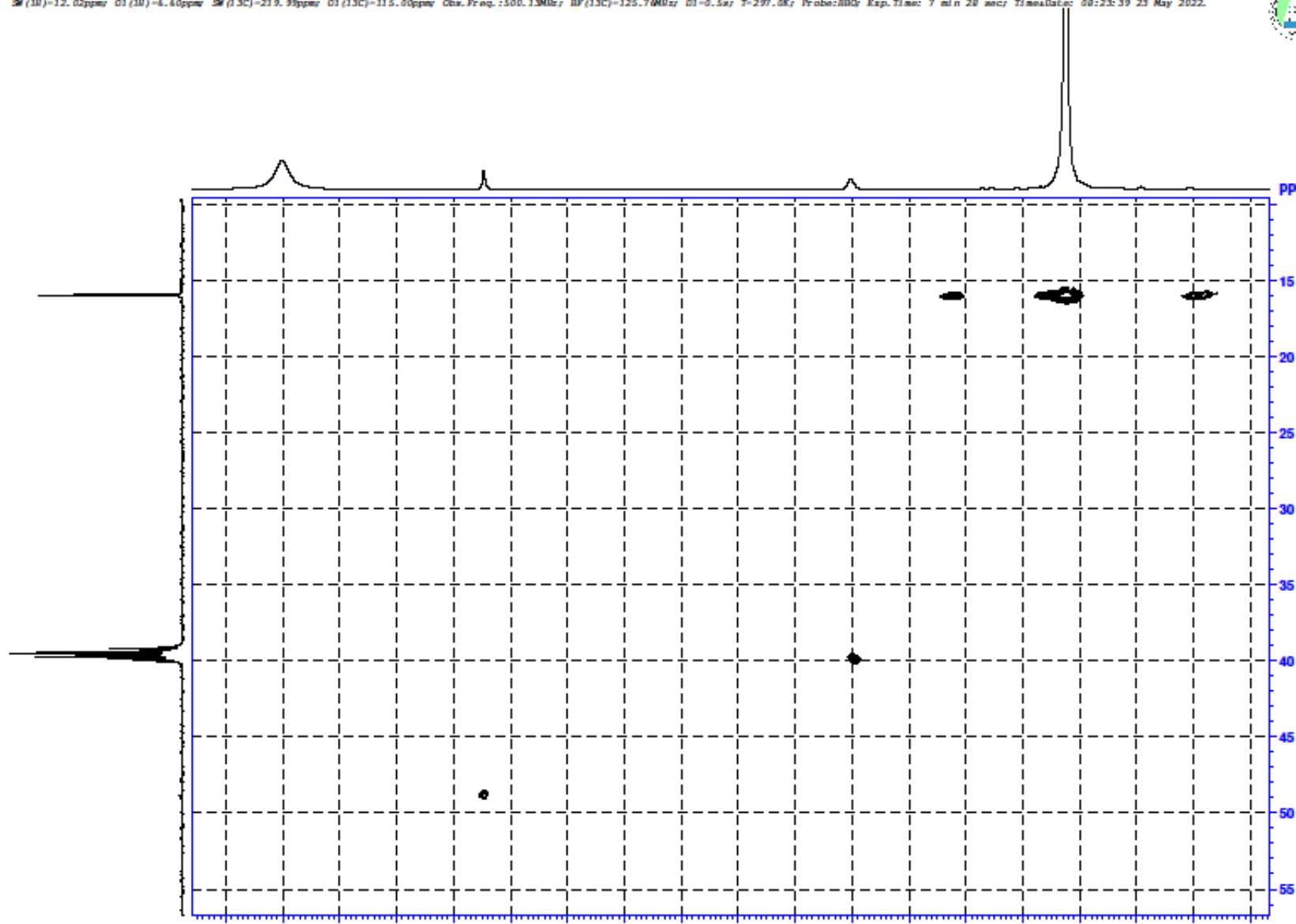
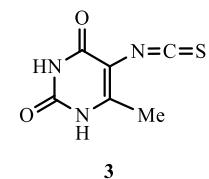
Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 13C) HMBC AV500 23.05.2022 LAN  
Sh(1H)=12.02ppm, D1(1H)=6.60ppm, Sh(13C)=220.00ppm, D1(13C)=115.00ppm; Obs.Freq.:500.13MHz; RF(13C)=125.70MHz; D1=0.4s; T=298.0K; Probe:BBQ; Exp.Time: 7 min 32 sec; Times:Date: 00:31:30 23 May 2022.

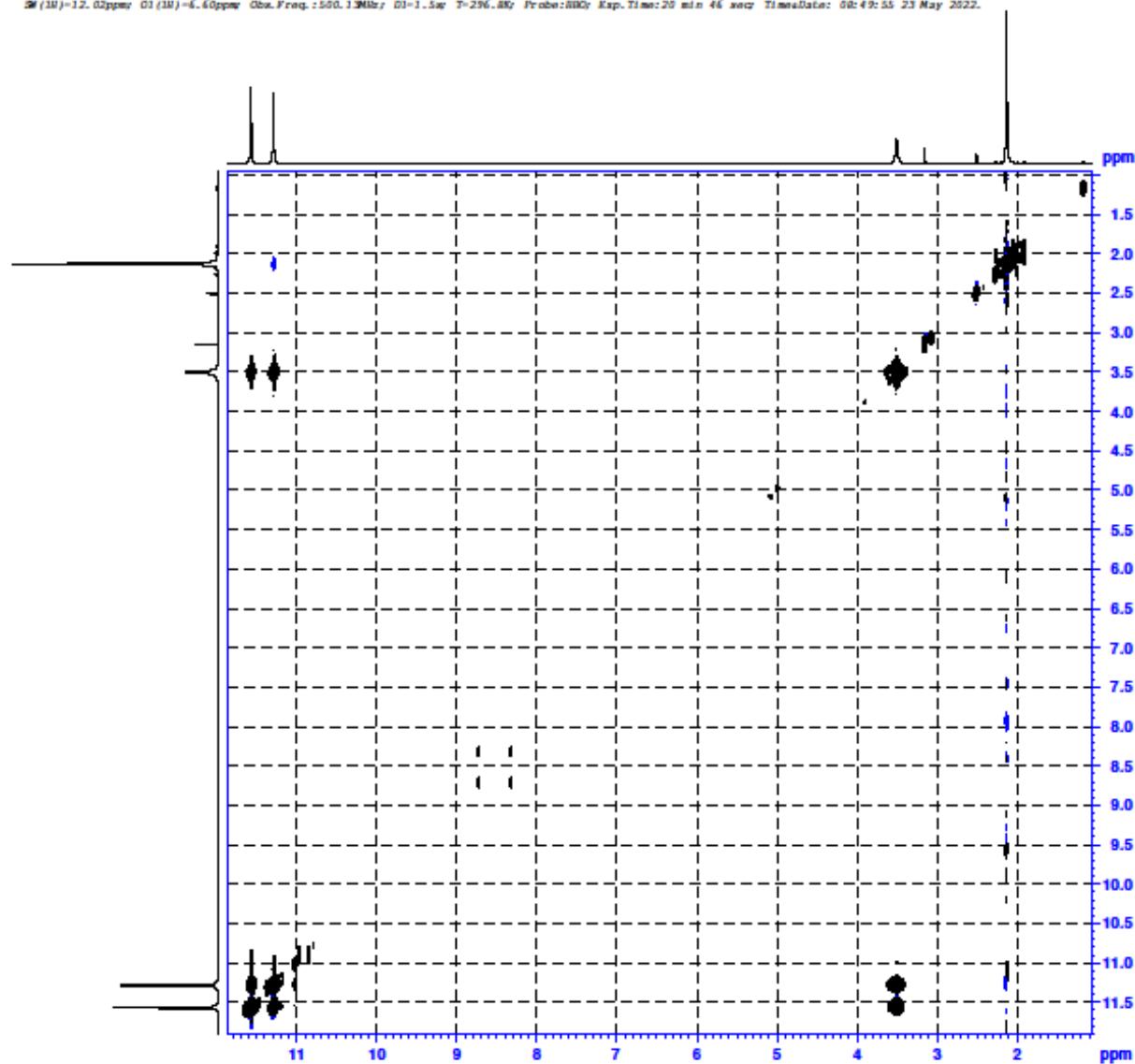


HMBC NMR spectrum of compound 3



HSQC NMR spectrum of compound 3





Current Data Parameters  
NAME: vta-IB-957-DMSO  
EXPNO: 9  
PROCNO: 1

F2 - Acquisition Parameters  
Date: 20220523  
Time: 0.49  
INSTRUM: spect  
PROBHD: 5 mm TBIRO BB-  
PULPROG: noesypph  
TD: 4096  
SOLVENT: DMSO  
TE: 29.8  
DS: 2  
SF: 6009.615 Hz  
FIDRES: 1.467191 Hz  
AQ: 0.3400372 sec  
RG: 65.36  
DW: 83.200 usec  
DE: 6.50 usec  
TE: 29.8 K  
D1: 0.0000000 sec  
D2: 1.5000000 sec  
D3: 0.0000000 sec  
D16: 0.00016640 sec

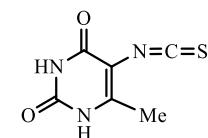
CHANNEL F1  
NUC1: 1H  
F1: 11.50 usec  
F2: 23.00 usec  
F1W1: 15.04899998 Hz  
SF01: 500.1333009 MHz

GRADIENT CHANNEL  
GPNAM1: SINE, 100  
GPZ1: 40.00 %  
P16: 1000.00 usec

F1 - Acquisition parameters  
TD: 256  
SF01: 500.1333 MHz  
FIDRES: 23.475060 Hz  
SW: 12.016 ppm  
PwMode: States-TPPI

F2 - Processing parameters  
SI: 1024  
SF: 500.1300053 MHz  
WM: QSGINE  
SSB: 2  
LB: 0 Hz  
GB: 0  
FC: 3.00

F1 - Processing parameters  
SI: 1024  
MC2: States-TPPI  
SF: 500.1300053 MHz  
WM: echo-antiecho  
SSB: 2  
LB: 0 Hz  
GB: 0



NOESY NMR spectrum of compound 3