

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

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Leonard M. Khalilov and Marat S. Yunusov**

EXPERIMENTAL SECTION

^1H and ^{13}C spectra were recorded on a Bruker Avance-III 500 MHz pulsed spectrometer with working frequencies of 500.13 MHz (^1H), 125.76 MHz (^{13}C) and 50.68 MHz (^{15}N) using a 5 mm probe with PABBO Z-gradient at a constant sample temperature of 298K in $\text{DMSO-}d_6$. Chemical shifts in the NMR spectra of ^1H , ^{13}C are given in ppm relative to the solvent signal (δ_{H} 2.50 and δ_{C} 39.52). Chemical shifts for ^{15}N nuclei are obtained from the $^{15}\text{N}\{^1\text{H}\}$ spectrum with reverse discontinuous decoupling from protons and $^{15}\text{N}\{^1\text{H}\}$ INEPT spectrum, and are confirmed by correlations in $\{^1\text{H}, ^{15}\text{N}\}$ HSQC and HMBC spectra, values are given in the ammonia scale. For ^{15}N nuclei, the calibration procedure was used according to the χ -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 $^{-1}$, chromatographic column C18 Zorbax Extend-C18, 2.1 mm, 1.8 μm) the capillary potential is 4 kV (the speed and temperature of the drying gas (nitrogen) is 10 dm 3 min $^{-1}$ and 325 $^{\circ}\text{C}$, 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 $^{-1}$. 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 $_3\text{N}$ (0.14 ml, 1.00 mmol) was added, and this was stirred at room temperature or at 0 $^{\circ}\text{C}$ 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 $^{\circ}\text{C}$ (MeOH), decomp. ^1H NMR, δ , ppm: 2.12 s (3H, H 7), 11.28 s (1H, H 1), 11.56 s (1H, H 3). $^{13}\text{C}\{^1\text{H}\}$ NMR, δ , ppm: 15.92 (C 7), 105.19 (C 5), 144.94 (N=C=S), 148.11 (C 6), 149.47 (C 2), 160.29 (C 4). $^{15}\text{N}\{^1\text{H}\}$ NMR, δ , ppm: 95.53 (N=C=S), 137.73 (N 1), 154.91 (N 3). Mass-spectrum, m/z (%): 182.0022 (100) $[\text{M-H}]^-$ C $_6\text{H}_4\text{N}_3\text{O}_2\text{S}$. IR spectrum, ν , cm $^{-1}$ (in vaseline oil) 1686-1737 (C=O), 1633-1653 (C=N). The data of the elemental analysis correspond to the formula C $_6\text{H}_5\text{N}_3\text{O}_2\text{S}$.

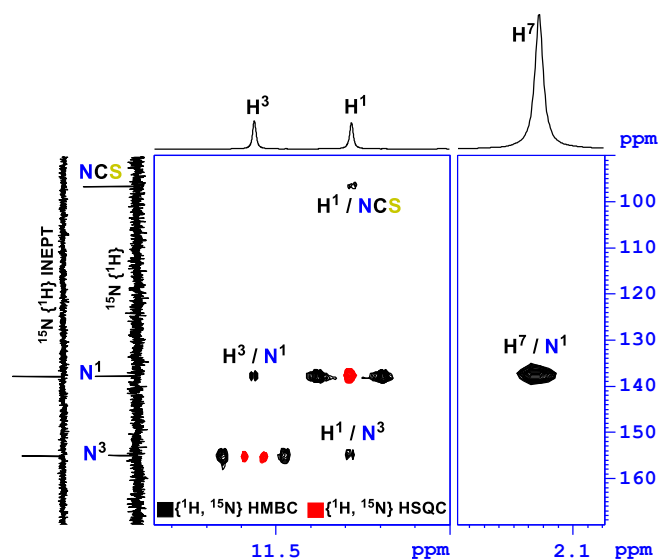


Figure S1 Superposition of the $\{^1\text{H}, ^{15}\text{N}\}$ HSQC and $\{^1\text{H}, ^{15}\text{N}\}$ HMBC spectra of compound **3** in DMSO- d_6 solution.

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

Empirical formula	C ₆ H ₅ N ₃ O ₂ 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 ³	398.04(4)
Z	2
$\rho_{\text{calc}}/\text{cm}^3$	1.528
μ/mm^{-1}	0.366
F(000)	188.0
Radiation	MoK α ($\lambda = 0.71073$)
2 θ range for data collection/ $^\circ$	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 [$I \geq 2\sigma(I)$]	$R_1 = 0.0488$, $wR_2 = 0.1306$
Final R indexes [all data]	$R_1 = 0.0712$, $wR_2 = 0.1484$
Largest diff. peak/hole / e Å ⁻³	0.45/-0.48

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

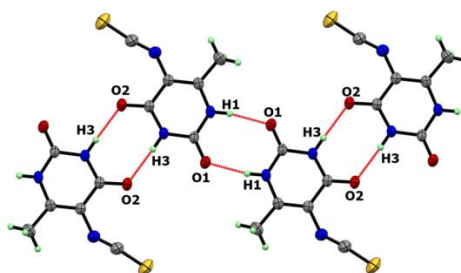
Bond	Length/Å	Bond	Length/Å
S1–C9	1.5666(15)	N3–C4	1.3758(15)
O2–C4	1.2329(14)	N8–C5	1.3807(16)
O1–C2	1.2282(14)	N8–C9	1.1853(18)
N1–C2	1.3652(15)	C4–C5	1.4452(17)
N1–C6	1.3711(16)	C6–C5	1.3632(17)
N3–C2	1.3681(14)	C6–C7	1.4885(18)

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)

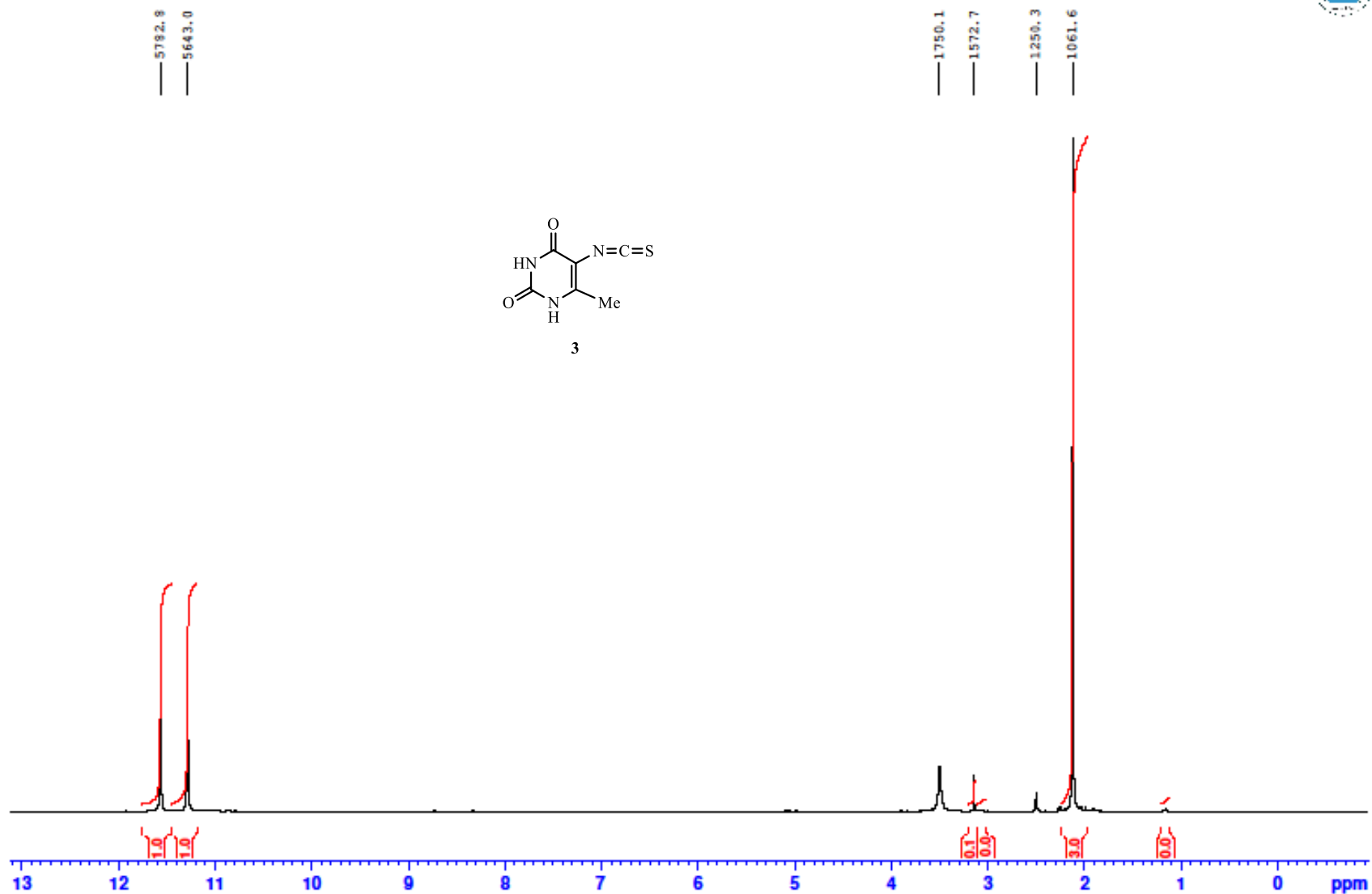
**Figure S2** Fragment of the crystal packaging of compound **3**.

- [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.

Sp-957 Chernikova IB-957 180mg in DMSO, 1H AV500 23.05.2022 LAN

SW (1H)=12.98ppm; D1 (1H)=6.00ppm; Obs.Freq.:500.13MHz; D1=30.0s; T=296.15K; Probe:BB0; Exp.Time: 5 min 35 sec; TimeDate: 08:15:11 23 May 2022.

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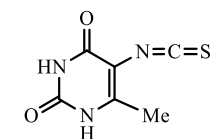
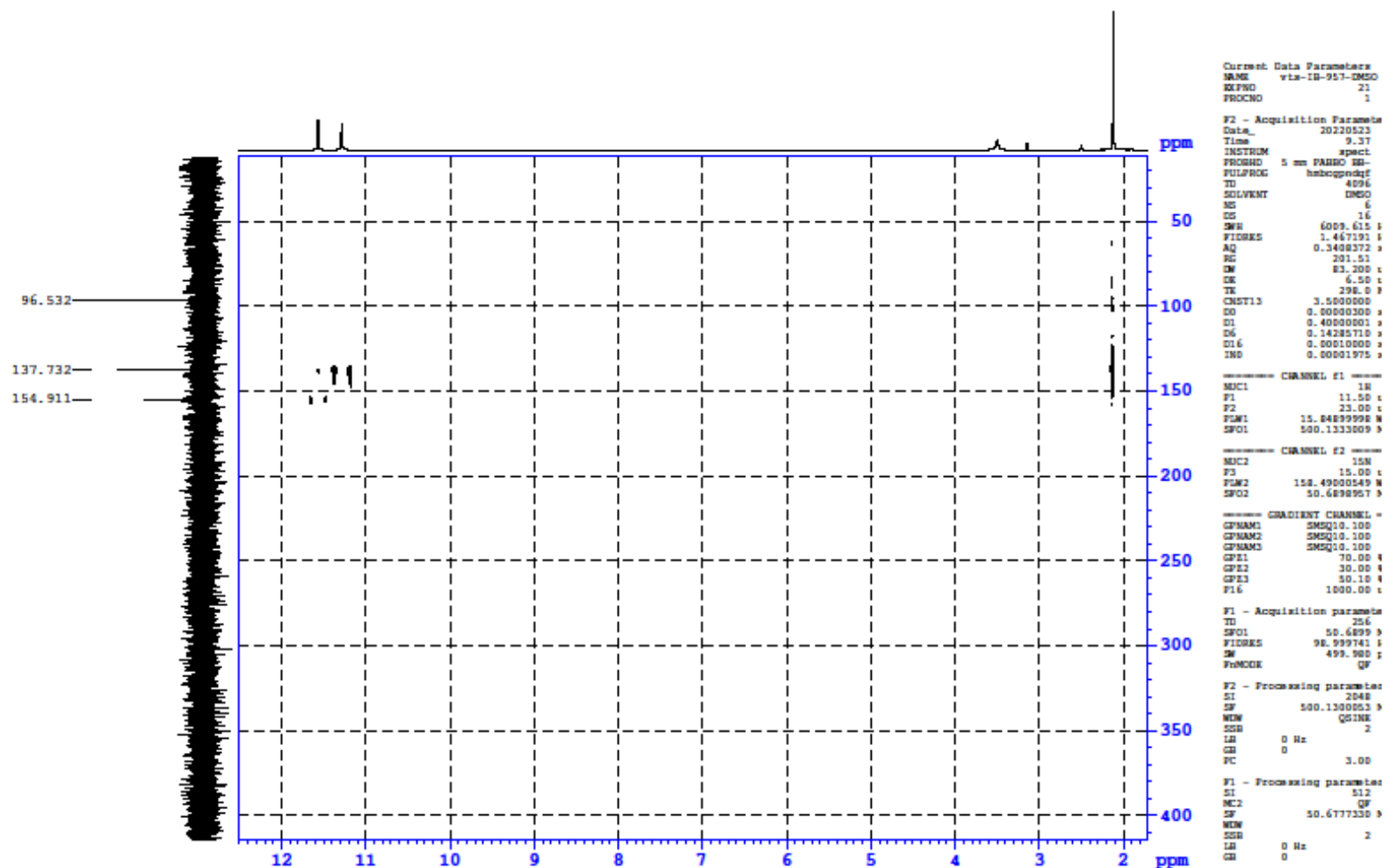


¹H NMR spectrum compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 15N) HMBC AV500 23.05.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (IIC RAS), 2022

SW (1H)=12.02ppm; CI (1H)=6.60ppm; SW (15N)=499.90ppm; CI (15N)=240.00ppm; Obs.Freq.:500.13MHz; IF (15N)=50.60MHz; DI=0.4x; T=298.0K; Probe:BBQ; Exp.Time:23 min 11 sec; TimeDate: 09:37:13 23 May



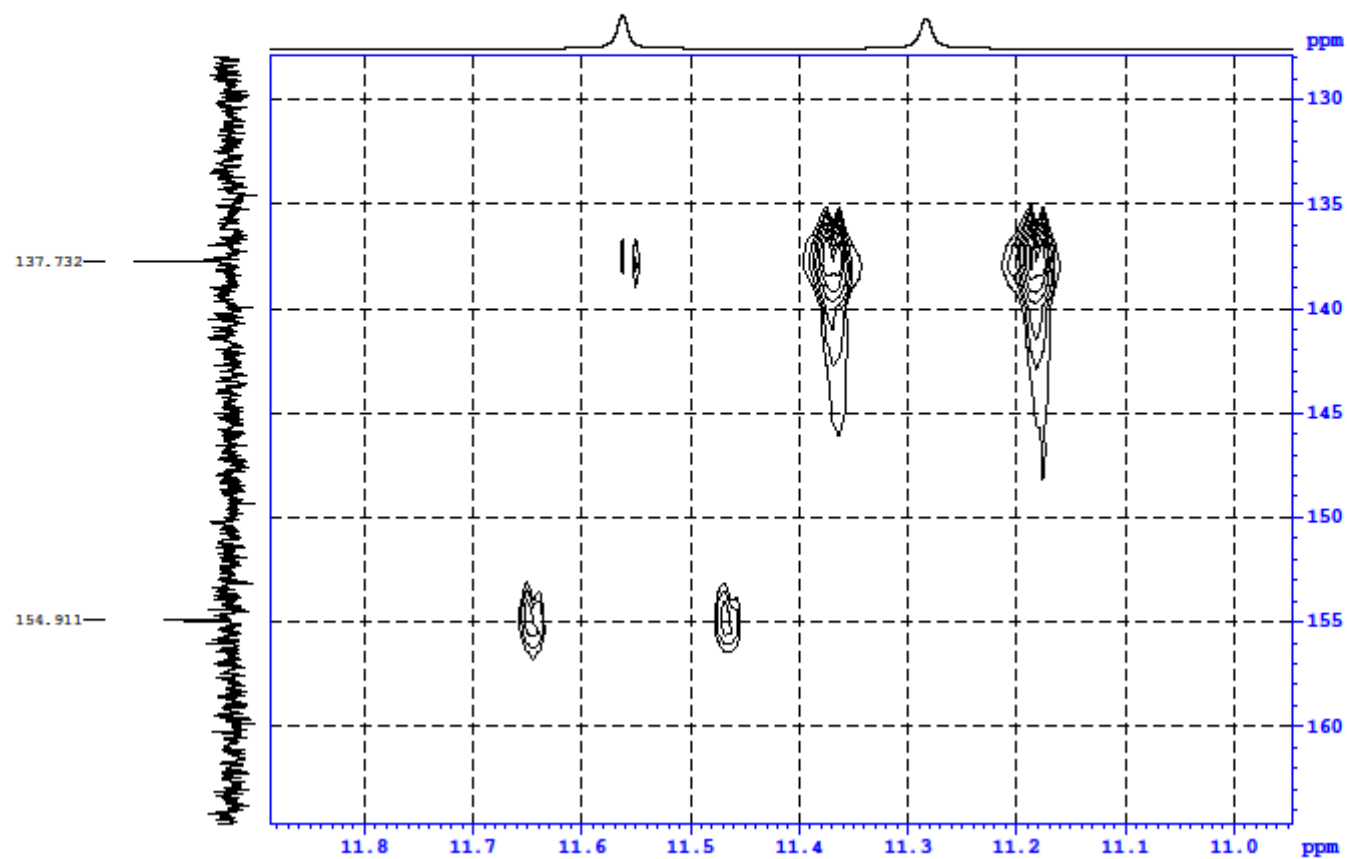
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^1H - ^{15}N HMBC NMR spectrum compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 15N) HMBC AV500 23.05.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

SW (1H)=12.02ppm CI (1H)=6.40ppm SW (15N)=499.90ppm CI (15N)=240.00ppm Obs.Freq.:500.13MHz SF (15N)=50.60MHz CI=0.4s T=298.0K; Probe:BBQ Exp.Time:23 min 11 sec TimeDate: 09:37:13 23 May



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PROCNO 1

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PULPROG hmcgpgndqf
TD 4096
SOLVENT DMSO
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SWH 6009.615 s
FIDRES 1.467191 s
AQ 0.3408372 s
RG 201.51
RM 81.200 s
CQ 6.50 s
TE 298.0 s
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DO 0.0000000 s
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D2 0.1408371 s
D16 0.0001000 s
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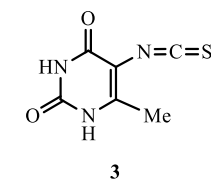
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GPE3 50.10 s
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F1 - Acquisition parameters
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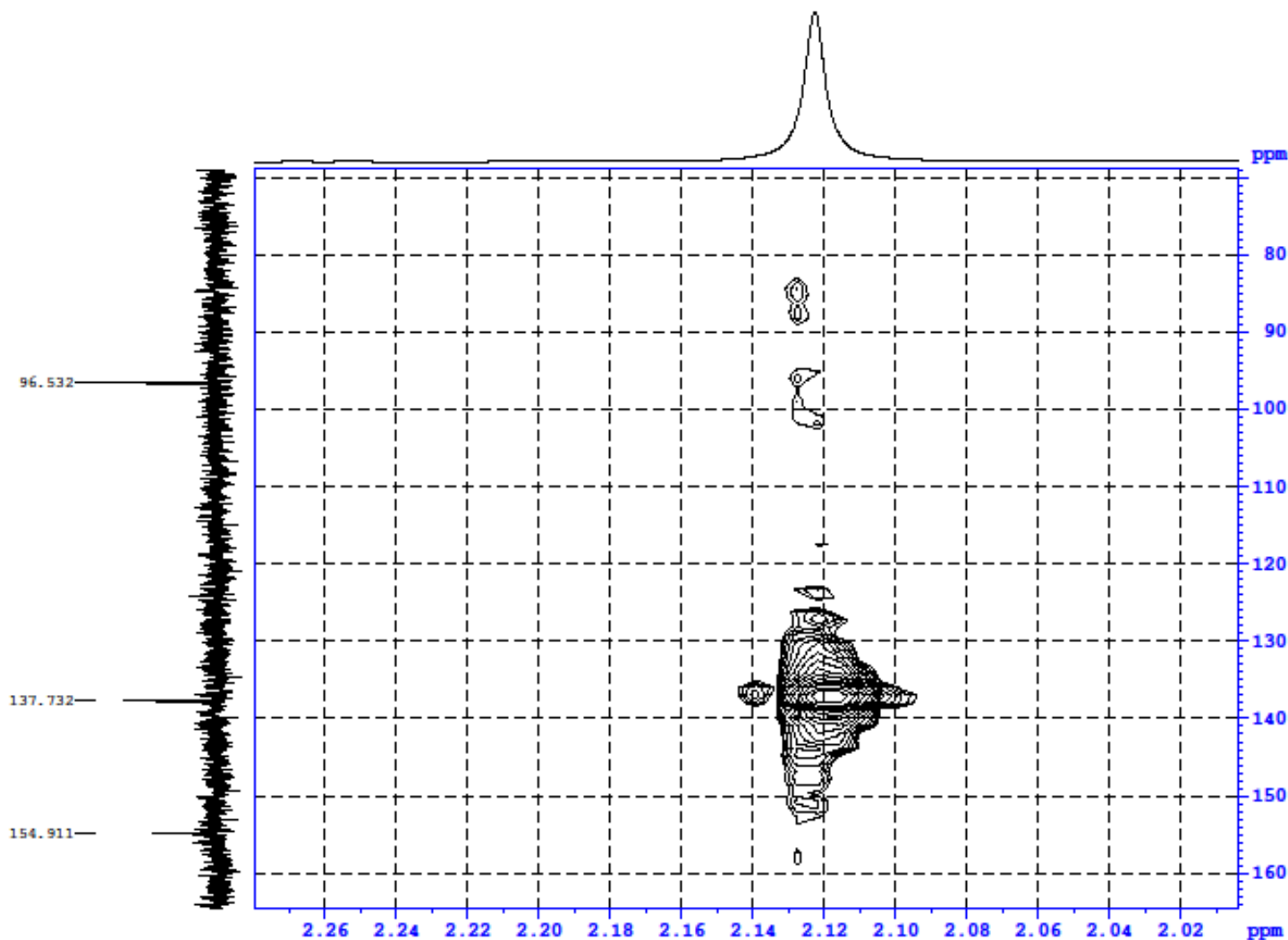


¹H-¹⁵N HMBC NMR spectrum 1 of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, [1H, 15N] HMBC AV500 23.05.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

SW (1H)=12.02ppm; CI (1H)=6.60ppm; SW (15N)=499.98ppm; CI (15N)=240.00ppm; Obs.Freq.:500.13MHz; RF (15N)=50.6MHz; DI=0.4; T=298.0K; Probe:BBQ; Exp.Time:23 min 11 sec; TimeDate: 09:37:13 23 May



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PROCNO 1

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TD 4096
SOLVENT DMSO
NS 6
DS 16
SWH 6009.615
FIDRES 1.467191
AQ 0.3488372
RG 201.51
SN 83.200
OR 6.50
TE 298.0
CHST13 3.5000000
SFO 0.0000000
D1 0.4000000
D6 0.1428571
D16 0.0001000
IND 0.0001975

CHANNEL F1
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F2 23.00
PLW1 15.84899998
SFO1 500.1333009

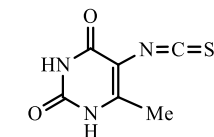
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F1 - Processing parameters
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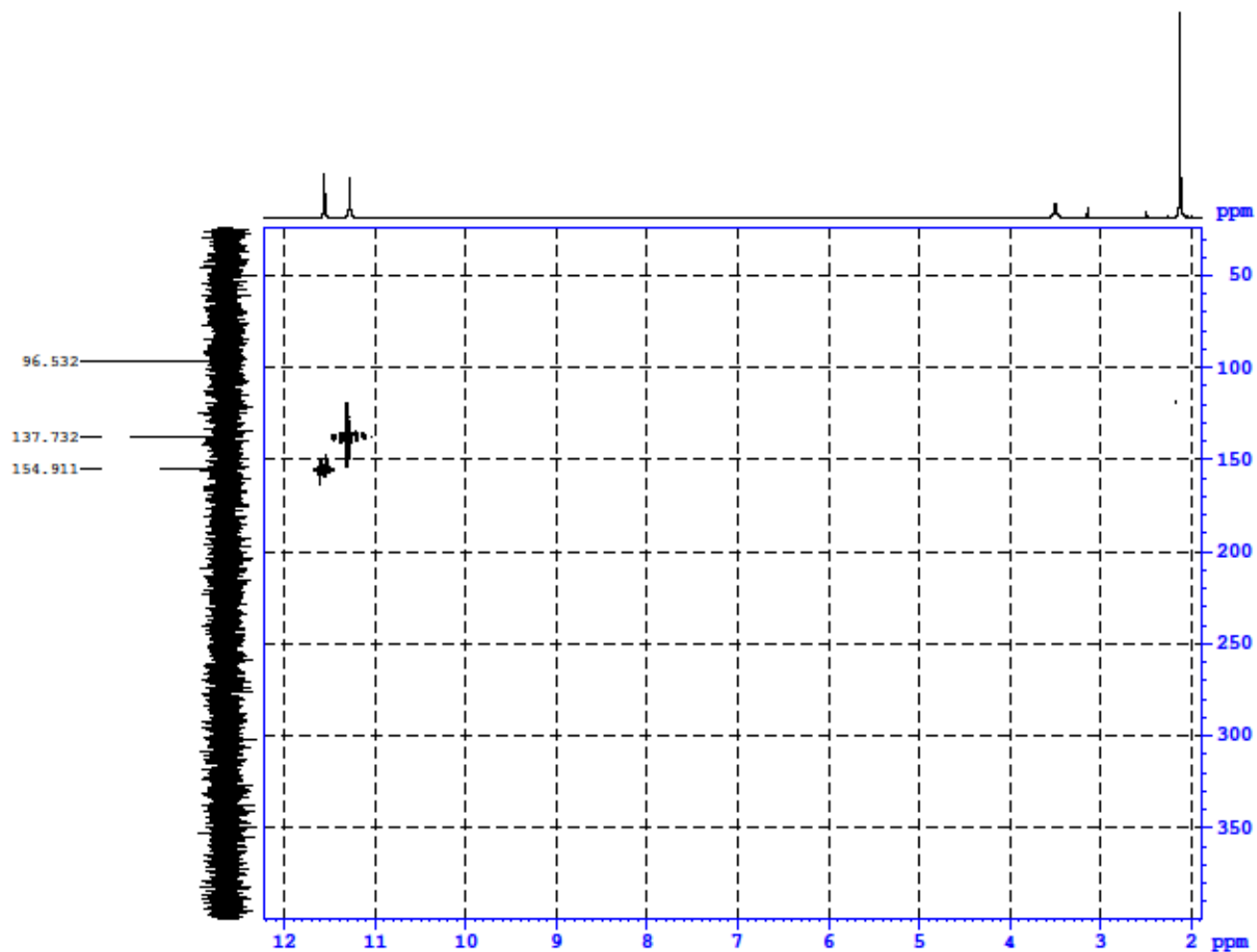
¹H-¹⁵N HMBC NMR spectrum 2 of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 15N) HSQC AV500 23.05.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2



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PROCNO 1

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SOLVENT DMSO
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FIDRES 1.467191 Hz
AQ 0.3408372 sec
RG 201.51
SW 83.200 uHz
DE 6.50 uHz
TE 297.0 K
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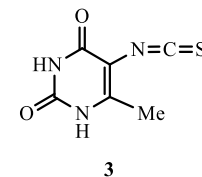
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CHANNEL F2
CPDPRG2 gcp
NUC2 15N
P3 15.00 uHz
P4 30.00 uHz
PCPD2 191.00 uHz
P1W2 158.49000549 W
P1W12 1.00000000 W
SFO2 50.6898957 MHz

GRADIENT CHANNEL
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GPHAM2 SMSQ10.100
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GP22 8.10 %
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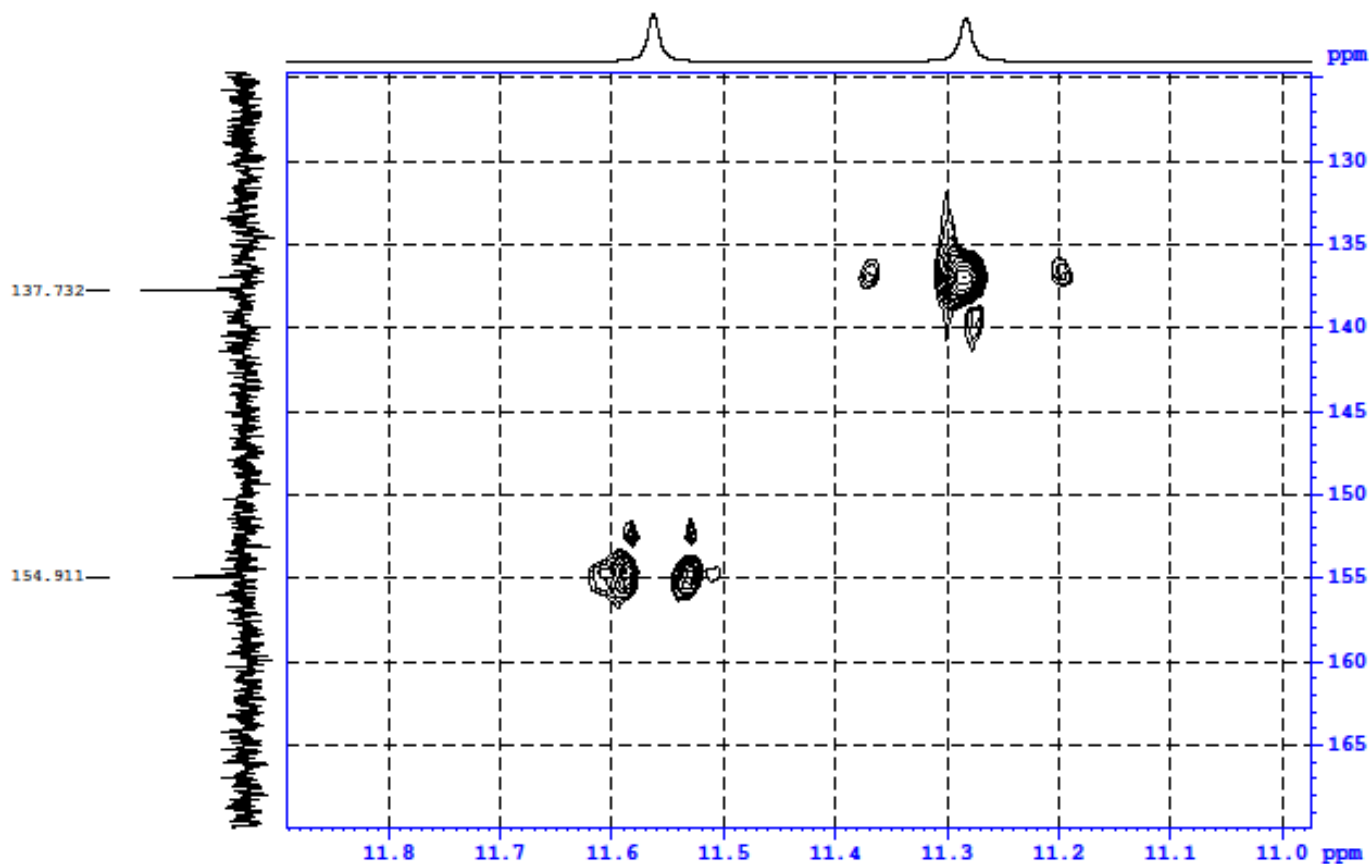
^1H - ^{15}N HSQC NMR spectrum of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 15N) HSQC AV500 23.05.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2



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



^1H - ^{15}N HSQC NMR spectrum1 of compound **3**

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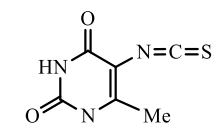
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FIDRES 1.467191 Hz
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RG 201.51
DM 83.200 sec
DE 6.50 sec
TE 297.0 K
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D1 0.40000001 sec
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P2 23.00 sec
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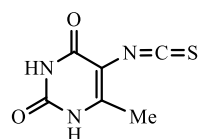
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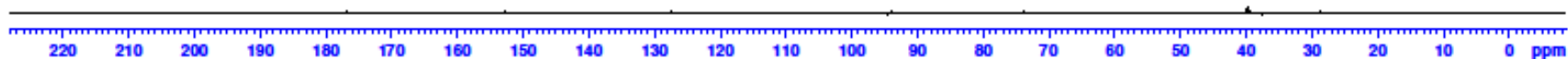
3

Sp-957 Chernikova IB-957 180mg in DMSO, $^{13}\text{C}\{^1\text{H}\}$ dept90 AV500 23.05.2022 LAN
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3



Sp-957 Chernikova IB-957 180mg in DMSO, $^{13}\text{C}\{^1\text{H}\}$ dept135 AV500 23.05.2022 LAN

δ (13C)=236.63ppm; δ (13C)=110.00ppm; Obs.Freq.:125.76MHz; δ 1=1.0s; T=297.2K; Probe:BBQ; Exp.Time: 1 min 27 sec; TimeDate: 08:19:07 23 May 2022.

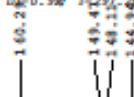


15.91



Sp-957 Chernikova IB-957 180mg in DMSO, $^{13}\text{C}\{^1\text{H}\}$ com AV500 23.05.2022 LAN

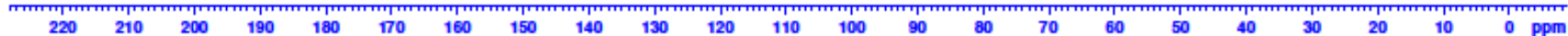
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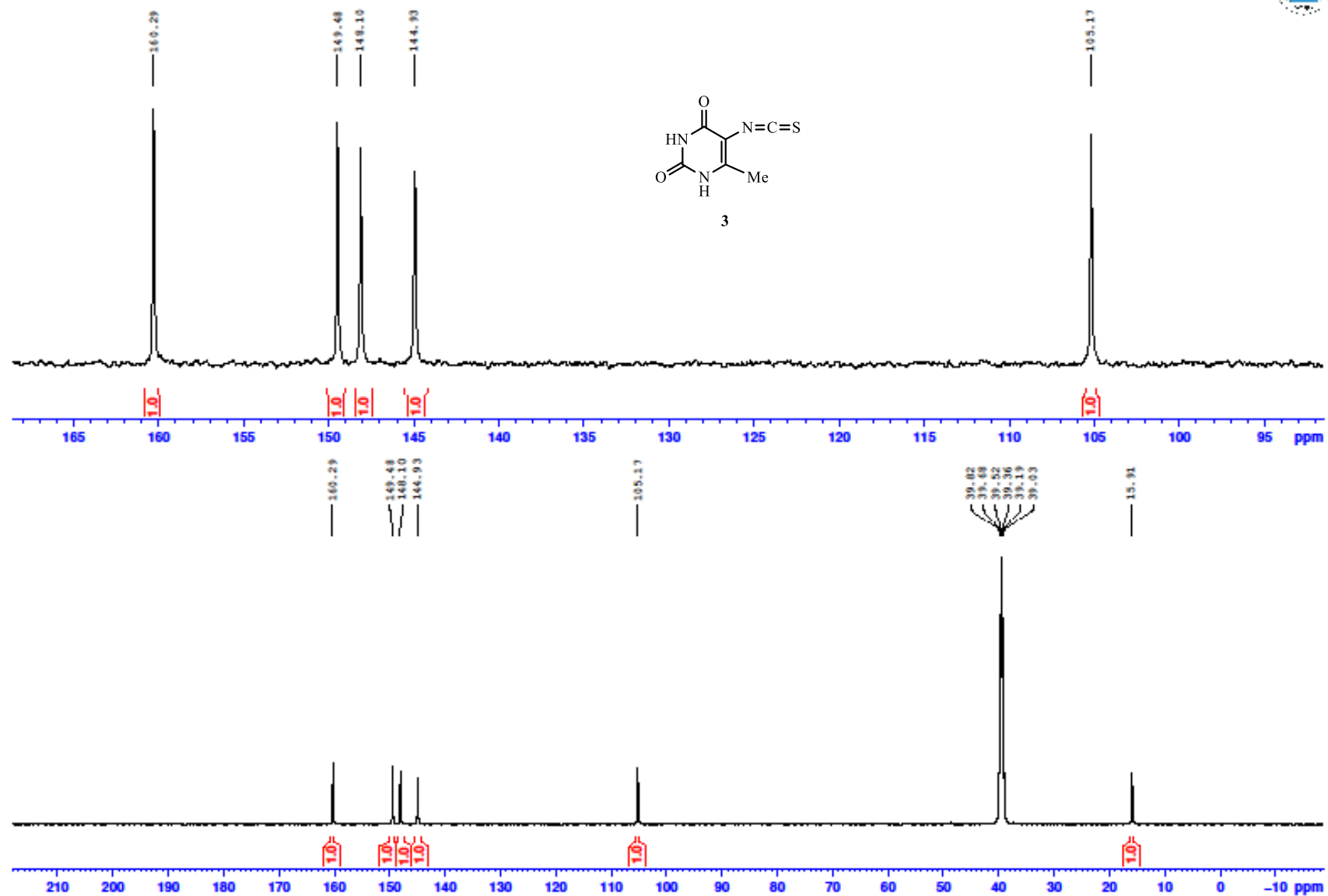
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15.92



^{13}C NMR spectrum of compound 3

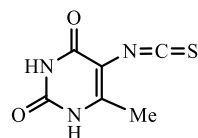


¹³C NMR spectrum integrals for compound 3

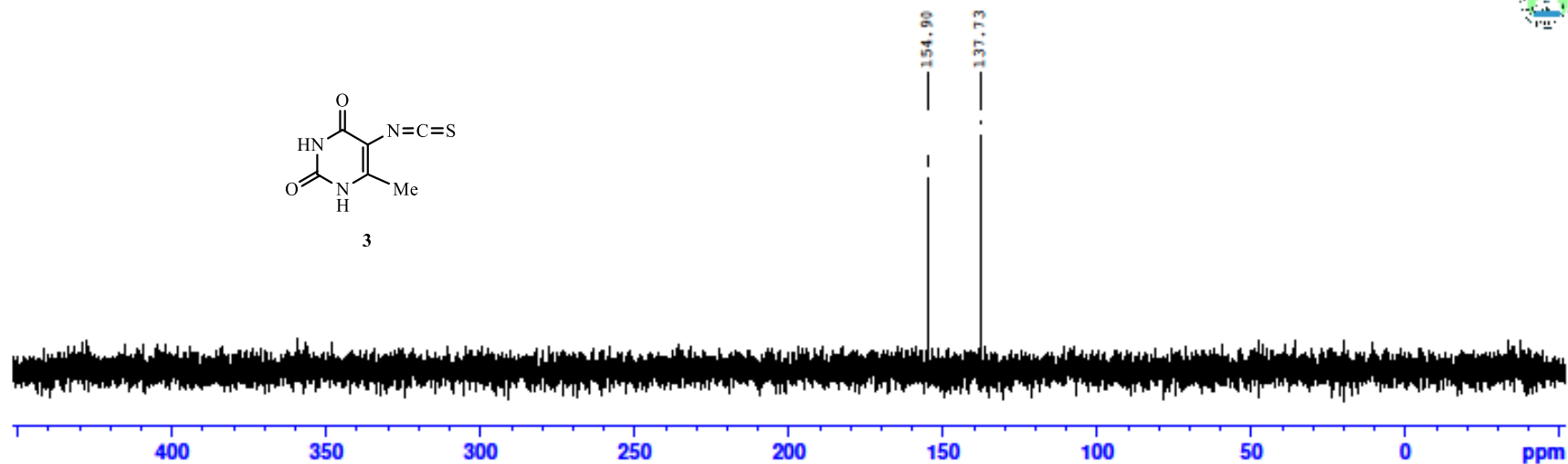
Sp-957 Chernikova IB-957 180mg in DMSO, 15N{1H} inapt AV500 23.05.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC)

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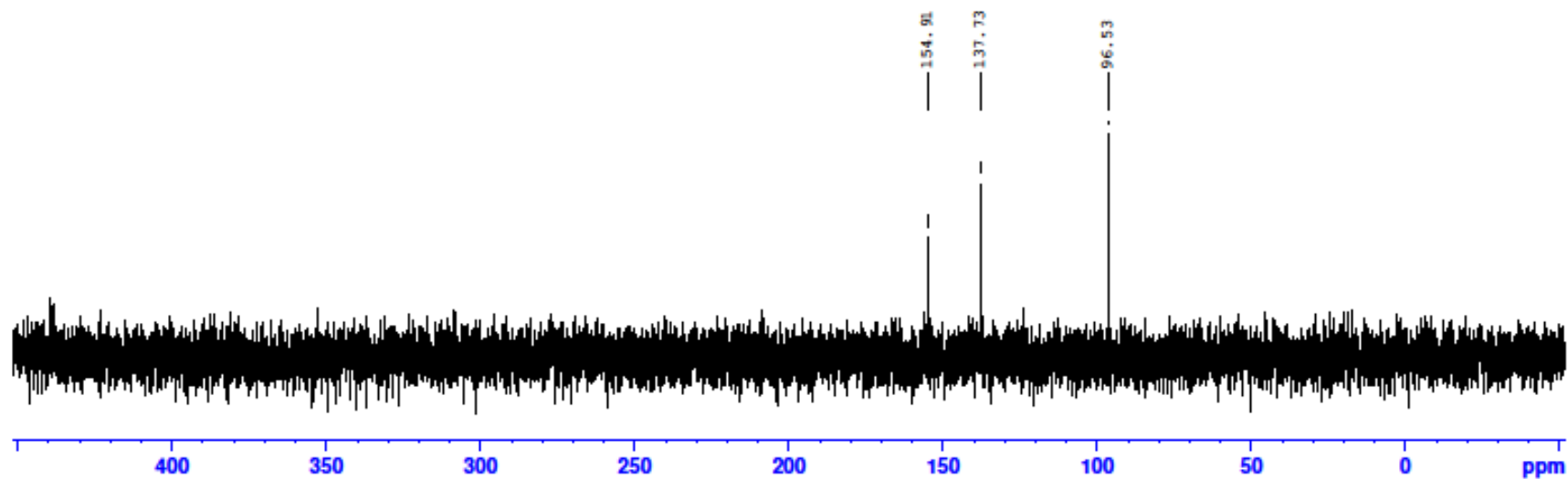


3

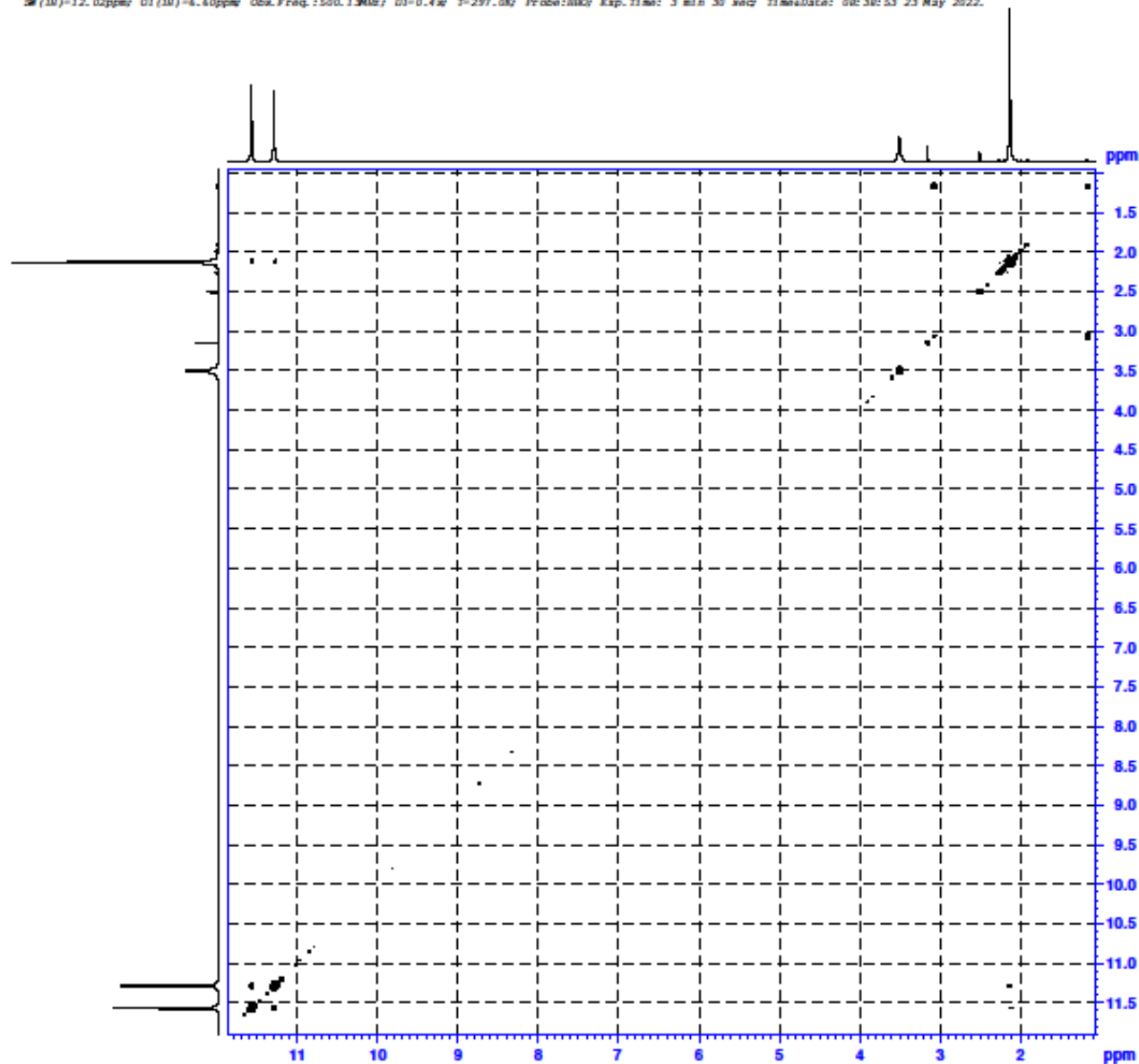


Sp-957 Chernikova IB-957 180mg in DMSO, 15N{1H} inv.gated spectrum AV500 23.05.2022 LAN

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¹⁵N NMR spectrum of compound 3



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 PROCNO 1

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 FIDRES 1.467191 Hz
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 RG 38.27
 DW 83.200 usec
 DE 6.50 usec
 TE 297.0 K
 D0 0.0000000 sec
 D1 0.40000001 sec
 D13 0.00000400 sec
 D16 0.00010000 sec
 TMO 0.00016640 sec

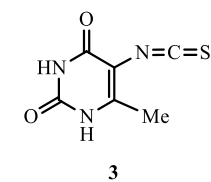
===== CHANNEL f1 =====
 NUC1 1H
 P0 11.50 usec
 P1 11.50 usec
 P1M1 15.84899998 W
 SFO1 500.1333009 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SMCQ10.100
 GPE1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 256
 SFO1 500.1333 MHz
 FIDRES 23.475060 Hz
 SW 12.016 ppm
 FWHM 0.7

F2 - Processing parameters
 SI 2048
 SF 500.1300013 MHz
 WCN QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 3.00

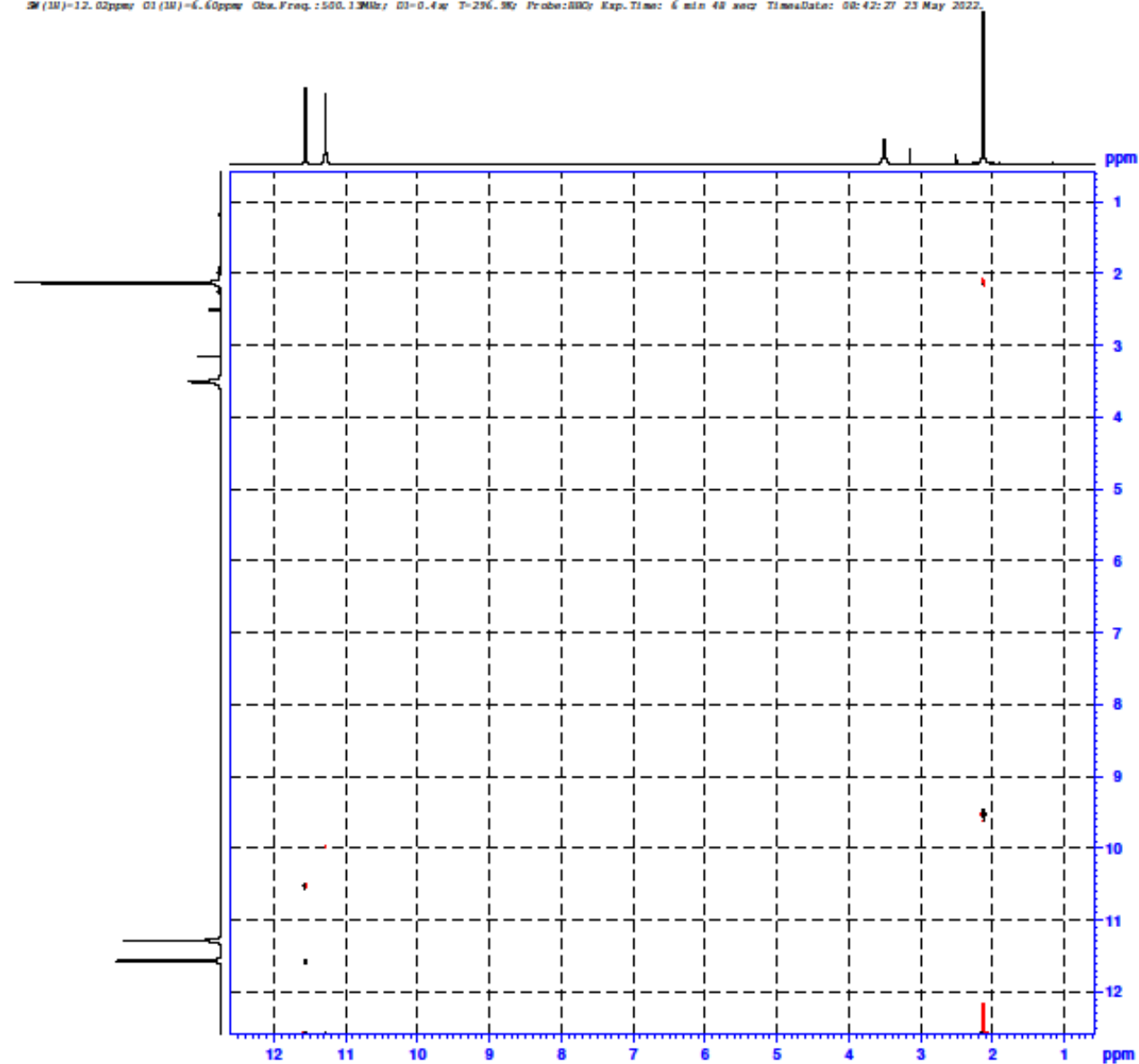
F1 - Processing parameters
 SI 2048
 MC2 QF
 SF 500.1300013 MHz
 WCN USER
 SSB 0
 LB 0 Hz
 GB 0



COSY NMR spectrum of compound 3

Sp-957 Charnikova IB-957 180mg in DMSO, {1H, 1H} COSY-DQF AV500 23.05.2022 IAN
 SW (1H)=12.02ppm D1 (1H)=6.60ppm Obs.Freq.:500.13MHz D1=0.4s T=296.9K Probe:BOO Exp.Time: 6 min 48 sec TimeDate: 00:42:27 23 May 2022.

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

F2 - Acquisition Parameters
 Date_ 20220523
 Time 8.42
 INSTRUM spect
 PROBE 5 mm FARGO S8-
 PULPROG cosygpmph
 TD 4096
 SOLVENT DMSO
 NS 1
 DS 16
 SWH 6009.615 Hz
 FIDRES 1.467191 Hz
 AQ 0.3408172 sec
 RG 201.51
 DM 83.200 umsec
 DE 6.50 umsec
 TE 296.9 K
 D0 0.00006856 sec
 D1 0.40000001 sec
 D13 0.00000400 sec
 D16 0.00010000 sec
 IN0 0.00016640 sec

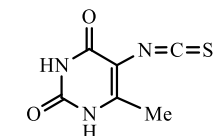
===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 umsec
 P2 23.00 umsec
 PLW1 15.84899998 W
 SFO1 500.133009 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SMSQ10.100
 GPNAM2 SMSQ10.100
 GPE1 10.00 %
 GPE2 20.00 %
 P16 1000.00 umsec

F1 - Acquisition parameters
 TD 512
 SFO1 500.1333 MHz
 FIDRES 11.737530 Hz
 SW 12.016 ppm
 FREQ001 TFP1

F2 - Processing parameters
 SI 2048
 SF 500.130053 MHz
 WDW QGWIN
 SSR 2
 LB 0 Hz
 GB 0
 PC 3.00

F1 - Processing parameters
 SI 2048
 MC2 TFP1
 SF 500.130053 MHz
 WDW
 SSR 2
 LB 0 Hz
 GB 0



3

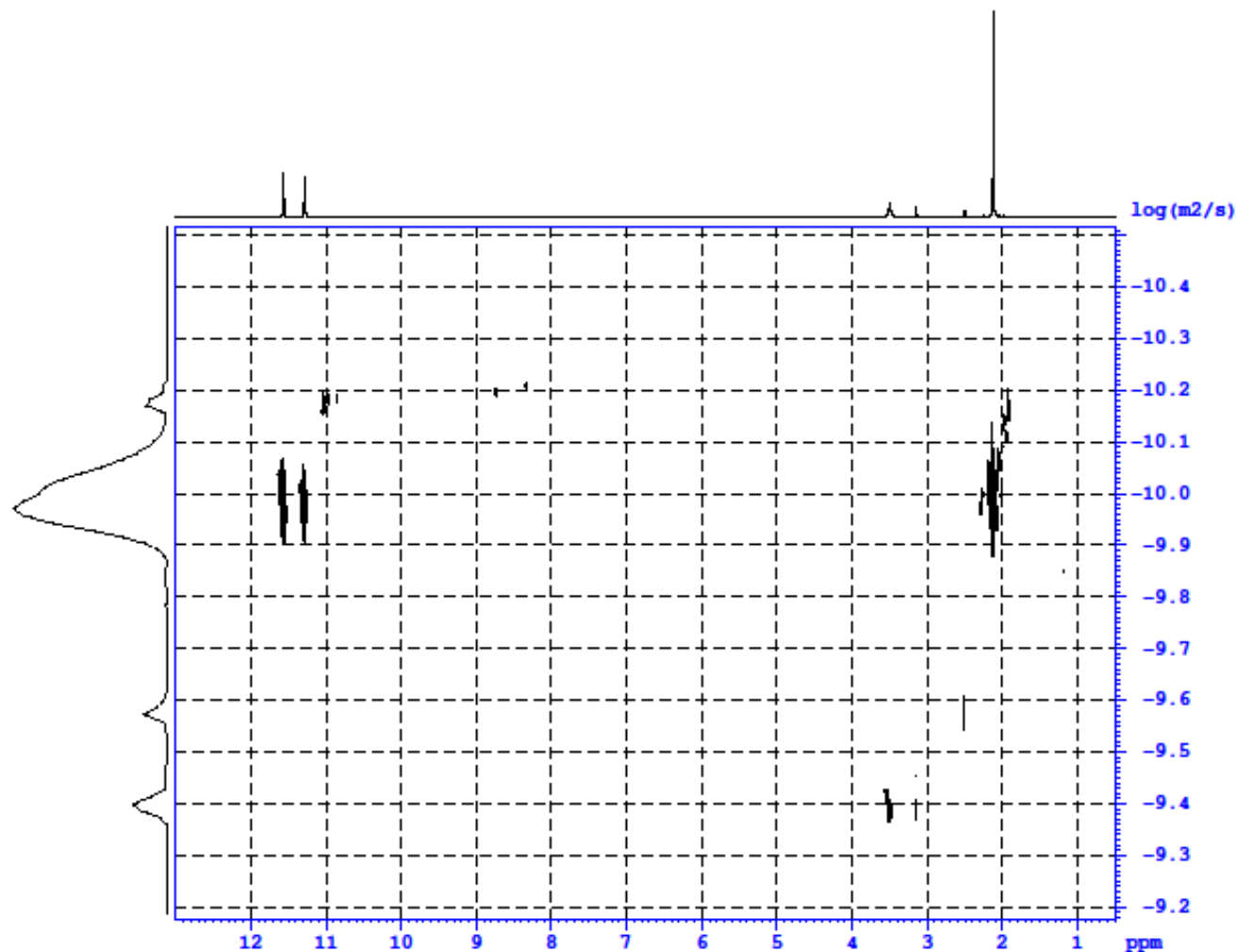
COSY DQF NMR spectrum of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 1H) DOSY AV500 23.05.2022 LAN

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SW(1H)=19.99ppm; OI(1H)=7.00ppm; Obs.Freq.:500.13MHz; DI=5.0s; T=296.4K; Probe:BBQ; Exp.Time: 6 min 20 sec; TimeDate: 10:48:27 23 May 2022.



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

F2 - Acquisition Parameter
Date_ 20220523
Time 10.48
INSTRUM spect
PROBHD 5 mm PABBO HD
PULPROG ledppp2a
TD 8192
SOLVENT DMSO
NS 8
DS 4
SWH 10000.000 Hz
FIDRES 1.220703 Hz
AQ 0.4096500 sec
RG 56.39
DM 50.000 sec
DE 6.50 sec
TE 296.4 K
D1 5.0000000 sec
D16 0.00010000 sec
D20 0.05000000 sec
D21 0.00500000 sec

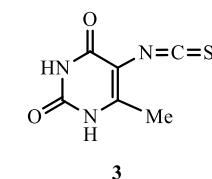
CHANNEL F1
NUC1 1H
P1 11.57 sec
P2 23.14 sec
P1M1 15.8489998 W
SFO1 500.1335009 MHz

GRADIENT CHANNEL
GPHAM6 SMSQ10.100
GPHAM7 SMSQ10.100
GPHAM8 SMSQ10.100
GPR6 100.00 %
GPR7 -17.13 %
GPR8 -13.17 %
P19 500.00 sec
P30 2000.00 sec

F1 - Acquisition parameter
TD 8
SFO1 500.1335 MHz
FIDRES 1250.000000 Hz
SW 19.995 pp
FMODE QF

F2 - Processing parameters
SI 4096
SF 500.1300053 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 3.00

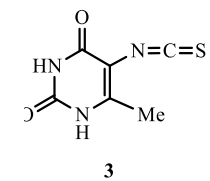
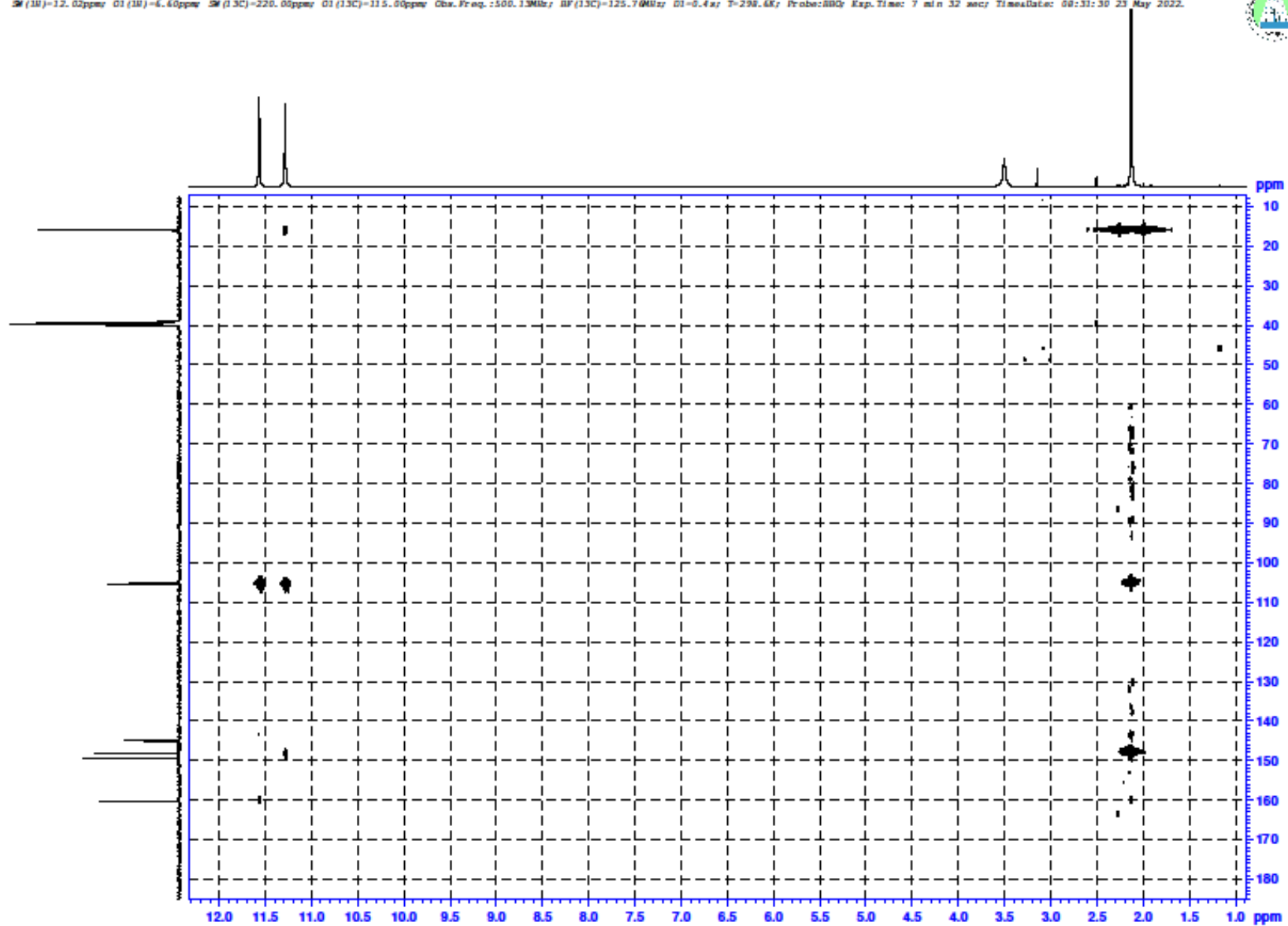
F1 - Processing parameters
SI 256
MC2 QF
SF 500.1300000 MHz
WDW #.
SSB 0
LB 0 Hz
GB 0



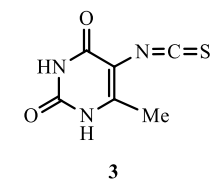
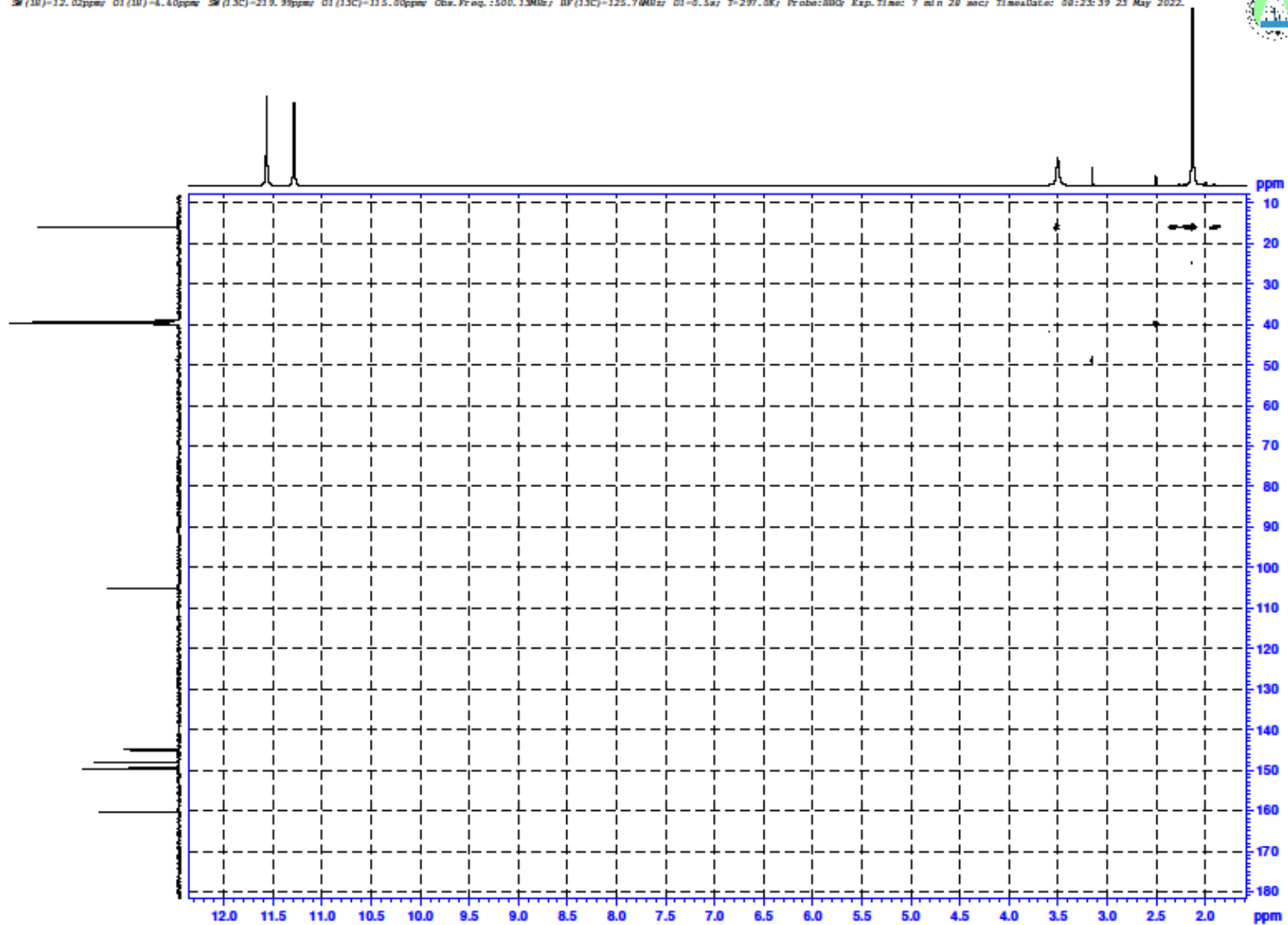
DOSY NMR spectrum of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, (1H, 13C) HMBC AV500 23.05.2022 LAN
 2H (1H)=12.02ppm; 01 (1H)=6.60ppm; 2H (13C)=220.00ppm; 01 (13C)=115.00ppm; Obs.Freq.:500.13MHz; RF(13C)=125.70MHz; Q1=0.4s; T=298.6K; Probe:BBQ; Exp.Time: 7 min 32 sec; Time/date: 09:31:30 23 May 2022.

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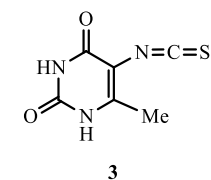
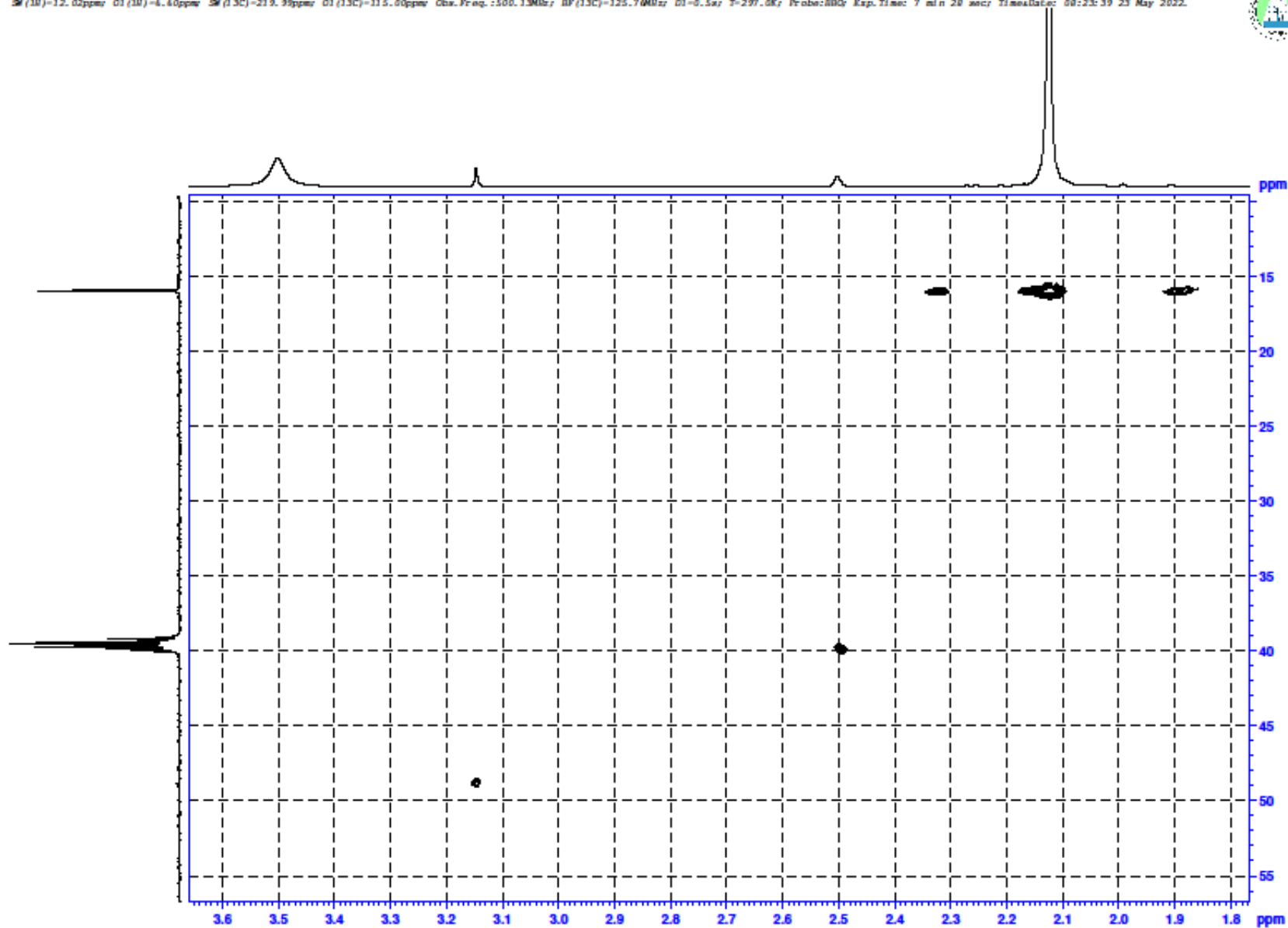
HMBC NMR spectrum of compound **3**



HSQC NMR spectrum of compound 3

Sp-957 Chernikova IB-957 180mg in DMSO, {1H, 13C} HSQC AV500 23.05.2022 LAN
 2H (1H)=12.02ppm; 01 (1H)=6.40ppm; 2H (13C)=219.90ppm; 01 (13C)=115.90ppm; Obs.Freq.:500.13MHz; RF (13C)=125.76MHz; D1=0.5s; T=297.0K; Probe:BBQ; Exp.Time: 7 min 28 sec; TimeDate: 00:23:39 23 May 2022.

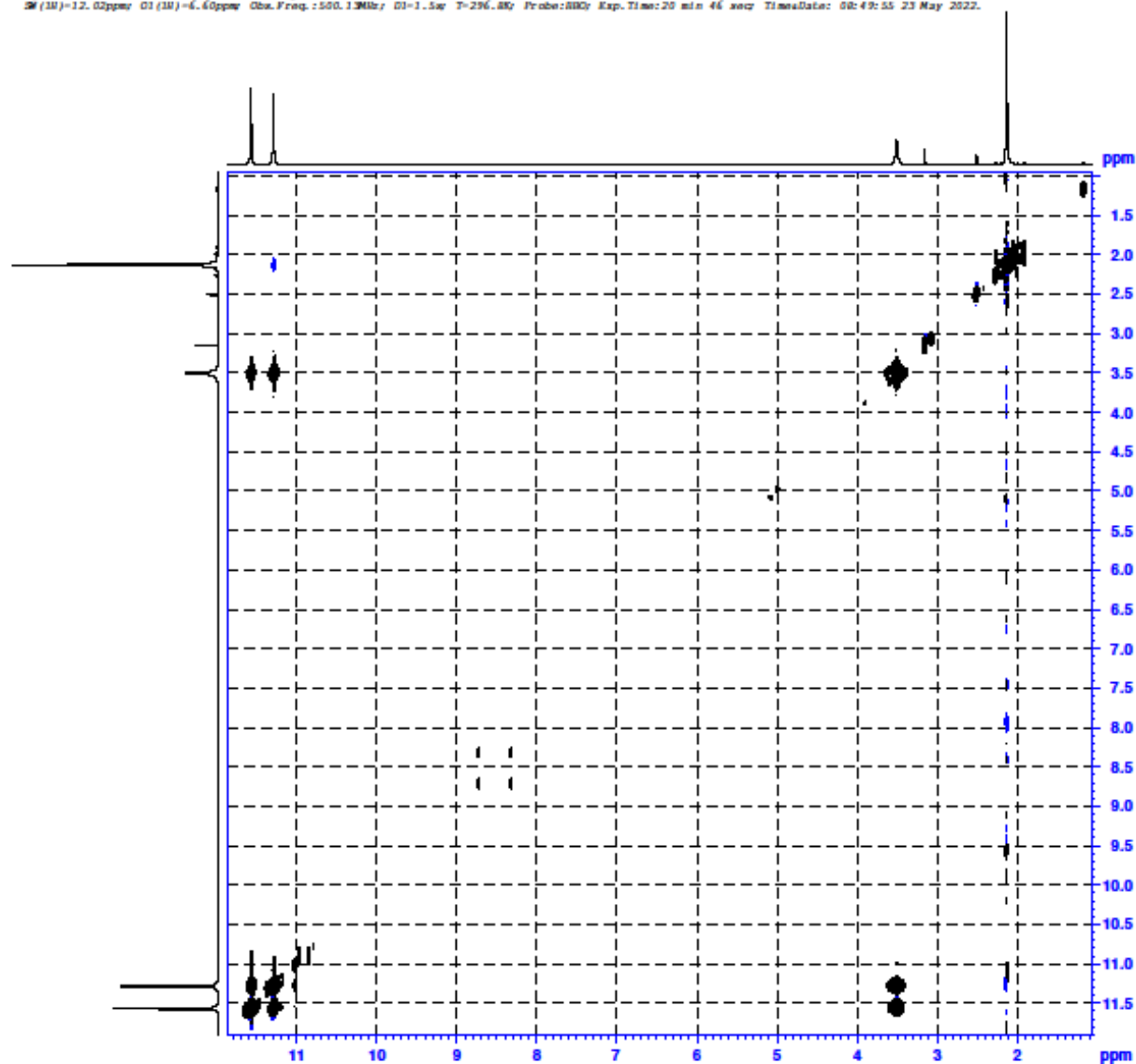
Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2022



HSQC NMR spectrum1 of compound 3

Sp-957 Charnikova IB-957 180mg in DMSO, ¹H, ¹H NOESY AV500 23.05.2022 LAN
 SW (1H)=12.02ppm D1 (1H)=6.60ppm Obs.Freq.:500.13MHz D1=1.5s T=296.8K Probe:BBQ Exp.Time:20 min 46 sec TimeDate: 08:49:55 23 May 2022.

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

F2 - Acquisition Parameters
 Date_ 20220523
 Time 8.49
 INSTRUM spect
 PULPROG zgpg30
 FULPROG noesypph
 TD 4096
 SOLVENT DMSO
 NS 2
 DS 16
 SSB 6009.615 Hz
 FIDRES 1.467191 Hz
 AQ 0.3400372 sec
 RG 65.36
 DW 83.200 usec
 DE 6.50 usec
 TE 296.8 K
 D0 0.00006856 sec
 D1 1.50000000 sec
 D8 0.50000000 sec
 D16 0.00010000 sec
 INO 0.00016640 sec

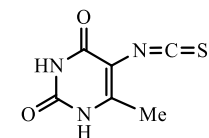
===== CHANNEL F1 =====
 NUC1 1H
 P1 11.50 usec
 P2 23.00 usec
 FWH 15.84899998 W
 SFO1 500.133009 MHz

===== GRADIENT CHANNEL =====
 GPM1 SINE 100
 GFE1 40.00 A
 F16 1000.00 usec

F1 - Acquisition parameters
 TD 256
 SFO1 500.1333 MHz
 FIDRES 23.475060 Hz
 SW 12.016 ppm
 FWH 15.84899998 W

F2 - Processing parameters
 SI 1024
 SF 500.130053 MHz
 WDM QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 3.00

F1 - Processing parameters
 SI 1024
 MC2 States-TFPI
 SF 500.130053 MHz
 WDM echo-antlecho
 SSB 2
 LB 0 Hz
 GB 0



3

NOESY NMR spectrum of compound 3