NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY

Vodovodna 95
1000 Ljubljana
SLOVENIJA

T: +386 (0)1 428 44 93
E: nfl@policija.si
www.policija.si

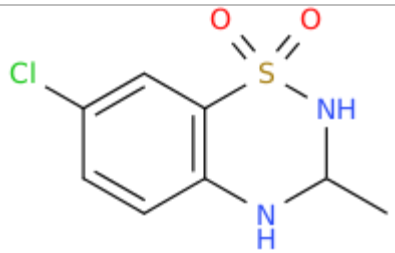
ANALYTICAL REPORT

IDRA-21 (C8H9ClN2O2S)

7-chloro-3-methyl-3,4-dihydro-2H-1λ⁶,2,4-benzothiadiazine-1,1-dione

Remark – other NPS detected: none

Sample ID:	1675-16
Sample description:	powder
Sample type:	collected /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	9/16/2017
Date of entry (M/D/Y) into NFL database:	10/26/2017
Report[1] updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[2] (base form)	
Systematic name	7-chloro-3-methyl-3,4-dihydro-2H-1λ⁶,2,4-benzothiadiazine-1,1-dione
Other names	7-chloro-3-methyl-3,4-dihydro-2H-benzo[e][1,2,4]thiadiazine 1,1-dioxide
Formula (per base form)	C8H9ClN2O2S
M_w (g/mol)	232,68
Salt form/anions detected	base
StdInChIKey (per base form)	VZRNTCHTJRLTMU-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	

[1] This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

[2] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Report updates

date	comments (explanation)

Instrumental methods (if applied) in NFL

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (9.258 min). Injection volume 1 ml and split mode (1:50). Injector temperature: 280 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 7.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300 until 6 min) amu.

2. HPLC-TOF (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; Injection volume 1 µl. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N2) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V.

3.FTIR-ATR (Perkin Elmer): scan range 4000-400 cm-1; resolution 4cm-1

4. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 ^{0}C. Chromatographic separation as above **(1).**
Split MS : IR = 1: 9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.
IR (condesed (solid) phase): IR scan range 4000 to 650, resolution 4 cm^{-1}.

5. **IC** (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30˚C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

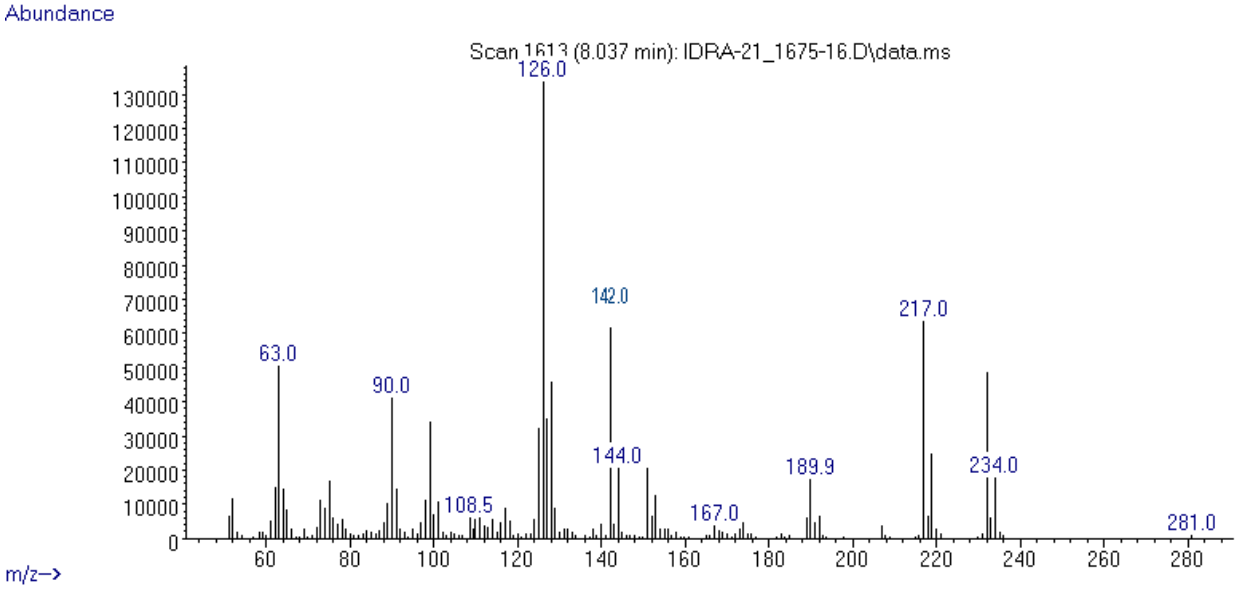
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	low (bad)

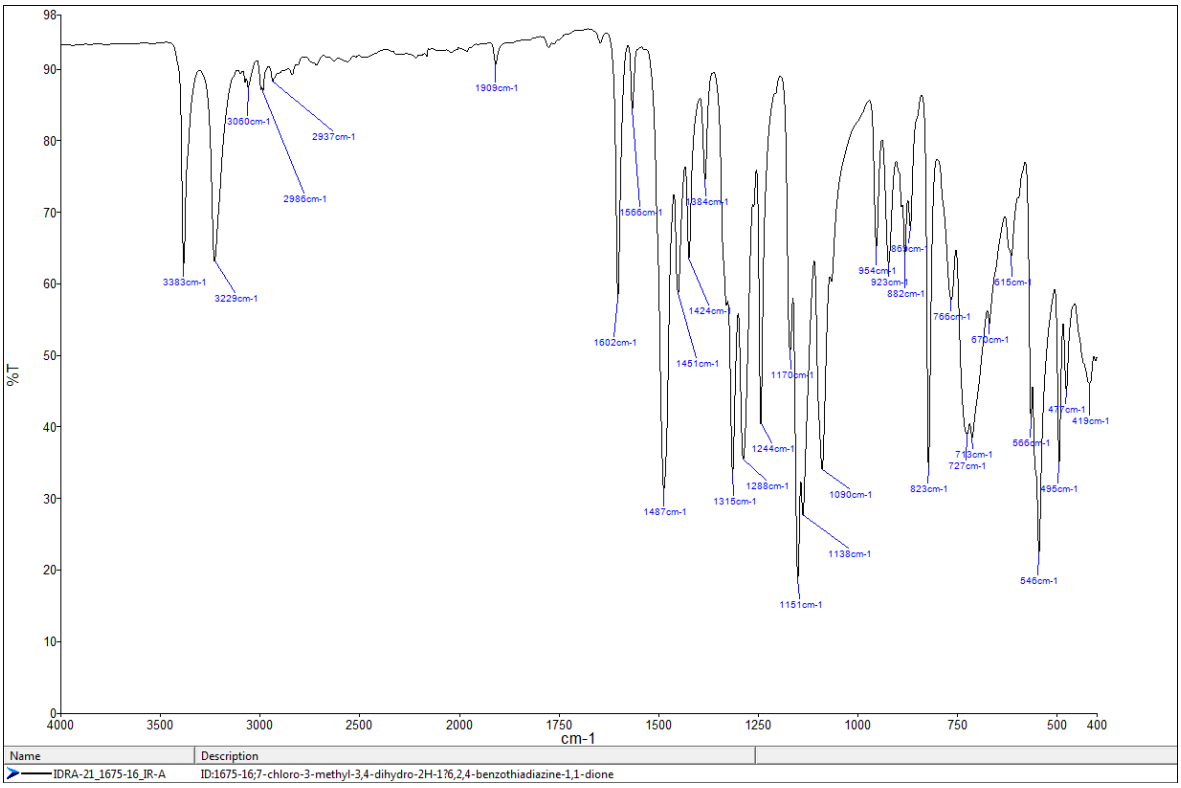
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8,05 BP(1): 126; BP(2):217,BP(3) :142,
HPLC-TOF	+	Exact mass (theoretical): 232,0073; measured value Δppm:-2,23; formula:C8H9ClN2O2S
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	base
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

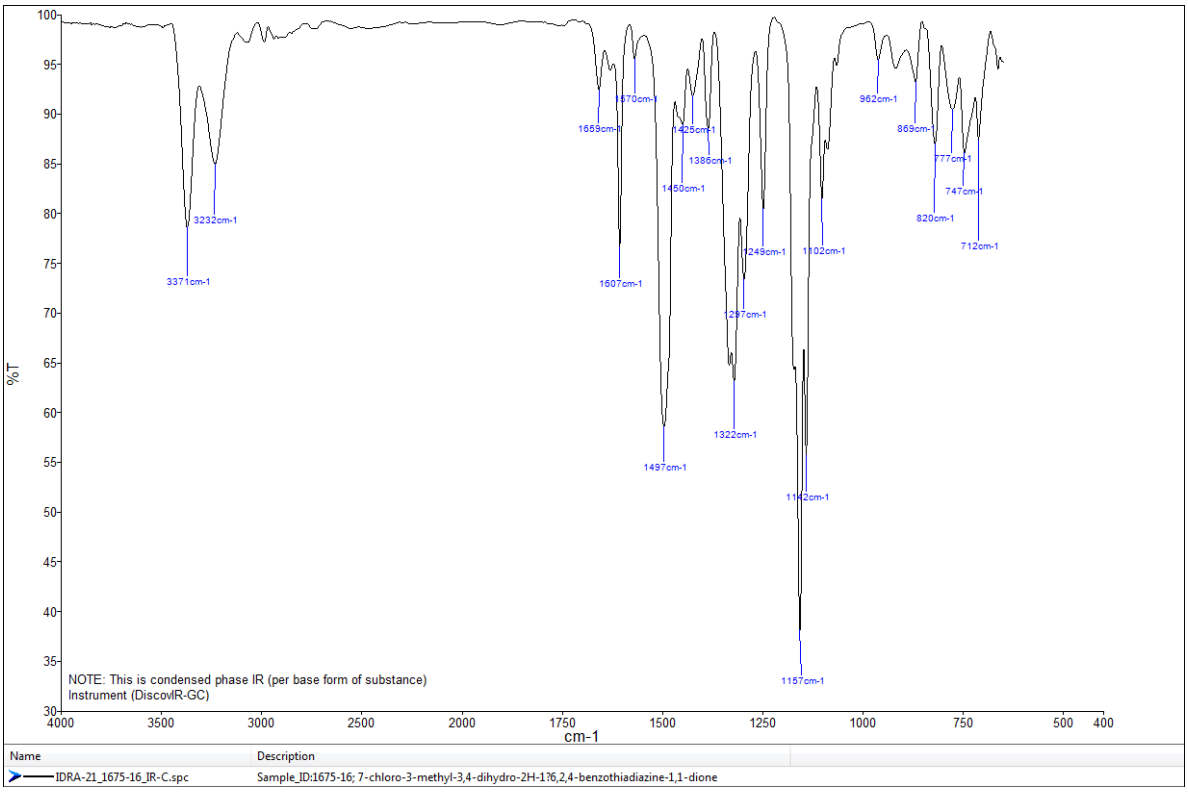
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)

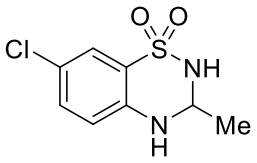


University
of Ljubljana

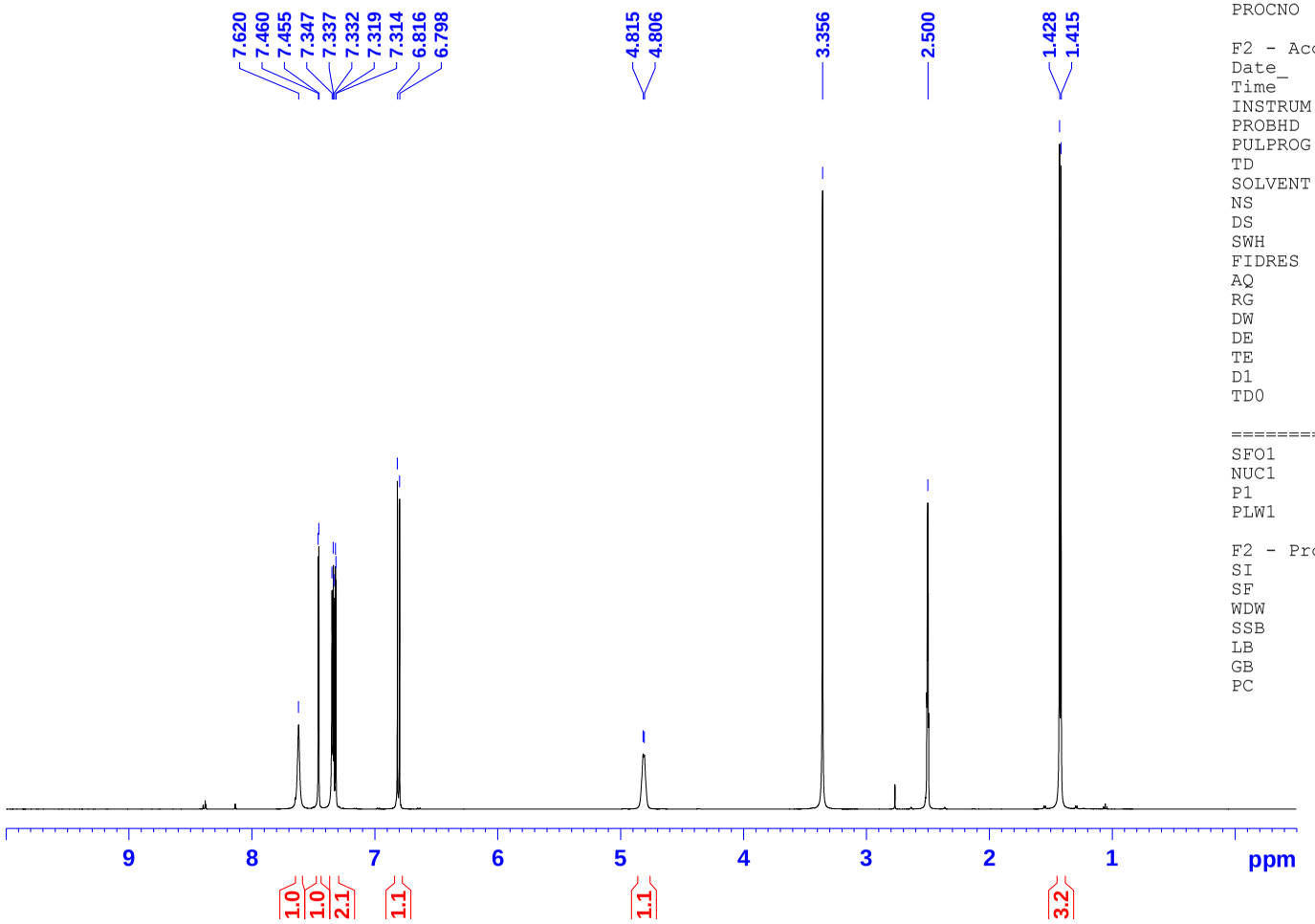
Faculty *of Chemistry and Chemical Technology*

Večna pot 113
P. O. Box 537
SI-1001 Ljubljana
Slovenia
Phone: +386 1 479 8558
janez.kosmrlj@fkkt.uni-lj.si

R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**1675-16**
Received date:	October 10, 2017
Our notebook code:	P-1675-16
NMR sample preparation:	14 mg dissolved in 0.7 mL DMSO-d_6
NMR experiments:	^{1}H, ^{13}C, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC, ^{1}H–^{15}N *gs*-HMBC
Proposed structure with atom numbering scheme, formula, exact mass, molecular weight:	 Chemical Formula: $C_8H_9ClN_2O_2S$ Exact Mass: 232,0073 Molecular Weight: 232,6820
Chemical name:	7-chloro-3-methyl-3,4-dihydro-2H-benzo[e][1,2,4]thiadiazine 1,1-dioxide (IDRA 21)
Comments:	- Structure elucidation based on 1D and 2D NMR spectra. - The result is consistent with the chemical formula proposed by HRMS.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Authors:	Martin Gazvoda, Marko Krivec, Janez Košmrlj
Date of report:	October 21, 2017

P-1675-16

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Current Data Parameters
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EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20171012
Time                      16.27
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                    zg30
TD                        65536
SOLVENT                    DMSO
NS                           16
DS                            2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                         80.6
DW                       50.000 usec
DE                        6.50 usec
TE                       296.0 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                500.1330885 MHz
NUC1                        1H
P1                        8.70 usec
PLW1                26.00000000 W

F2 - Processing parameters
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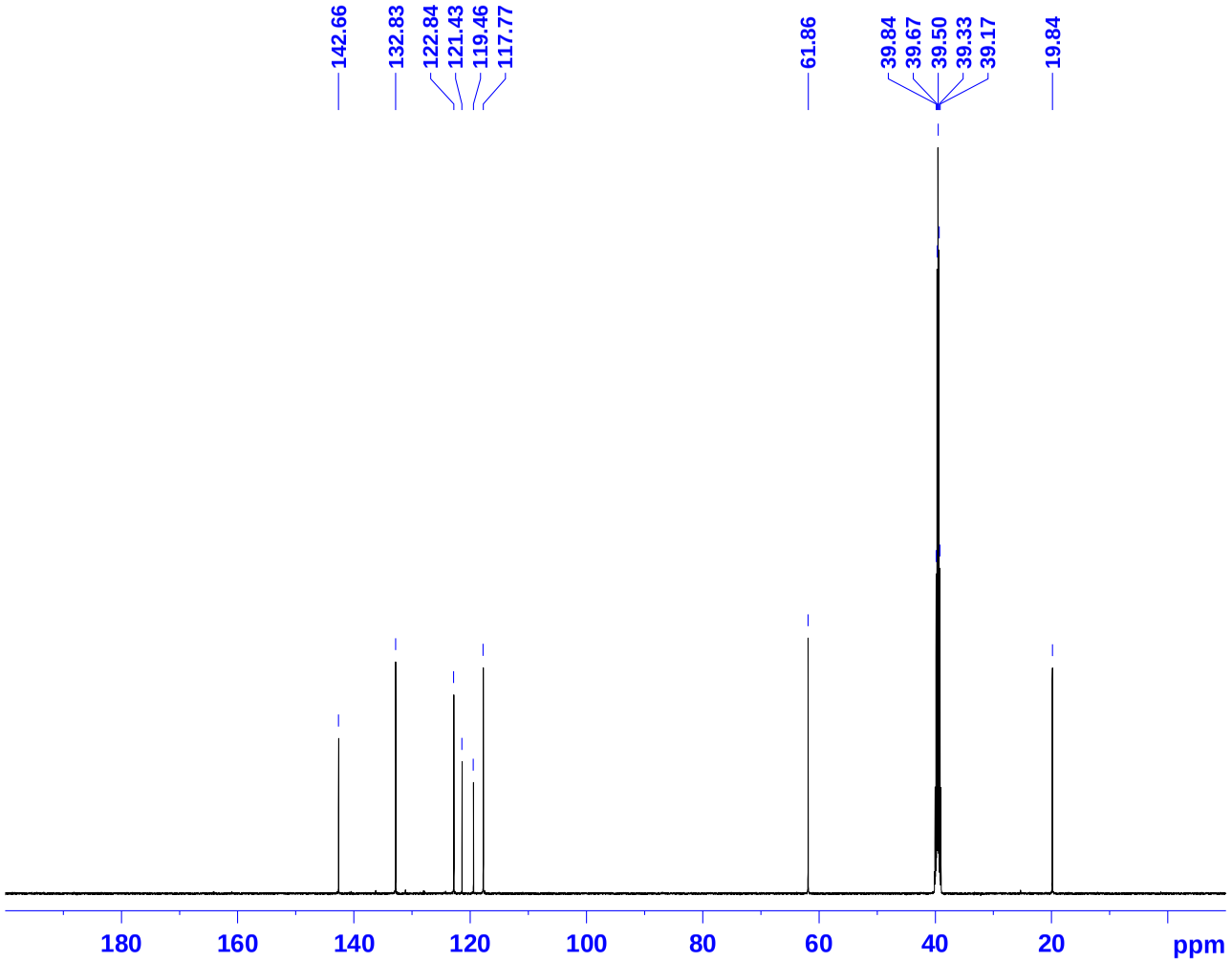
SF                  500.1300042 MHz
WDW                         EM
SSB                  0
LB                        0.05 Hz
GB                   0
PC                        1.00
```

P-1675-16




```
Current Data Parameters
NAME                  P-1675-16
EXPNO                        12
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20171012
Time                      18.39
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                    DMSO
NS                         3072
DS                            4
SWH                   29761.904 Hz
FIDRES                 0.454131 Hz
AQ                    1.1010048 sec
RG                          912
DW                       16.800 usec
DE                        6.50 usec
TE                       296.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                125.7703637 MHz
NUC1                        13C
P1                        8.70 usec
PLW1               122.00000000 W

======== CHANNEL f2 ========
SFO2                500.1320005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                26.00000000 W
PLW12                0.30046001 W
PLW13                0.15113001 W

F2 - Processing parameters
SI                        32768
SF                  125.7577885 MHz
WDW                          EM
SSB              0
LB                        1.00 Hz
GB               0
PC                        1.40
```