



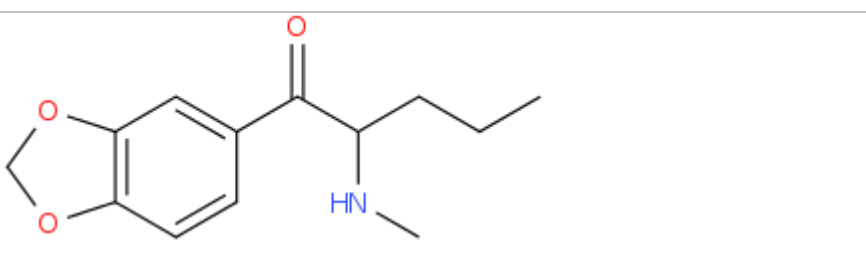
## ANALYTICAL REPORT<sup>1</sup>

### Pentylone (C<sub>13</sub>H<sub>17</sub>N<sub>3</sub>O<sub>3</sub>)

#### 1-(2H-1,3-benzodioxol-5-yl)-2-(methylamino)pentan-1-one

Remark – other NPS detected: **none**

Sample ID:	1533-16
Sample description:	crystallinic - white-off
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	2/18/2016
Date of entry (M/D/Y) into NFL database:	5/31/2016
Report updates (if any) will be published here:	<a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a>

Substance identified - structure <sup>2</sup> (base form)	
Systematic name	1-(2H-1,3-benzodioxol-5-yl)-2-(methylamino)pentan-1-one
Other names	β-keto-methylbenzodioxolylpentanamine; βk-{N-methyl-1-(1,3-benzodioxol-5-yl)-2-pentanamine; βk-MBDP
Formula (per base form)	C <sub>13</sub> H <sub>17</sub> N <sub>3</sub> O <sub>3</sub>
M <sub>w</sub> (g/mol)	235,28
Salt form/anions detected	chloride
StdInChIKey	DFMLLULIEUXXSA-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	none
Add.info (purity..)	pure by GC-MS, HPLC-TOF, NMR

<sup>1</sup> 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.

<sup>2</sup> 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 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235 °C, source and quadropole temperatures 280 °C and 180 °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 (N<sub>2</sub>) 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**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 °C. Chromatographic separation as above (**1**). Split MS : IR = 1: 9.

MSD source EI = 70 eV. GC-MS transfer line T= 235 °C, source and quadropole temperatures 280 °C and 180 °C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.

IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm<sup>-1</sup>.

**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

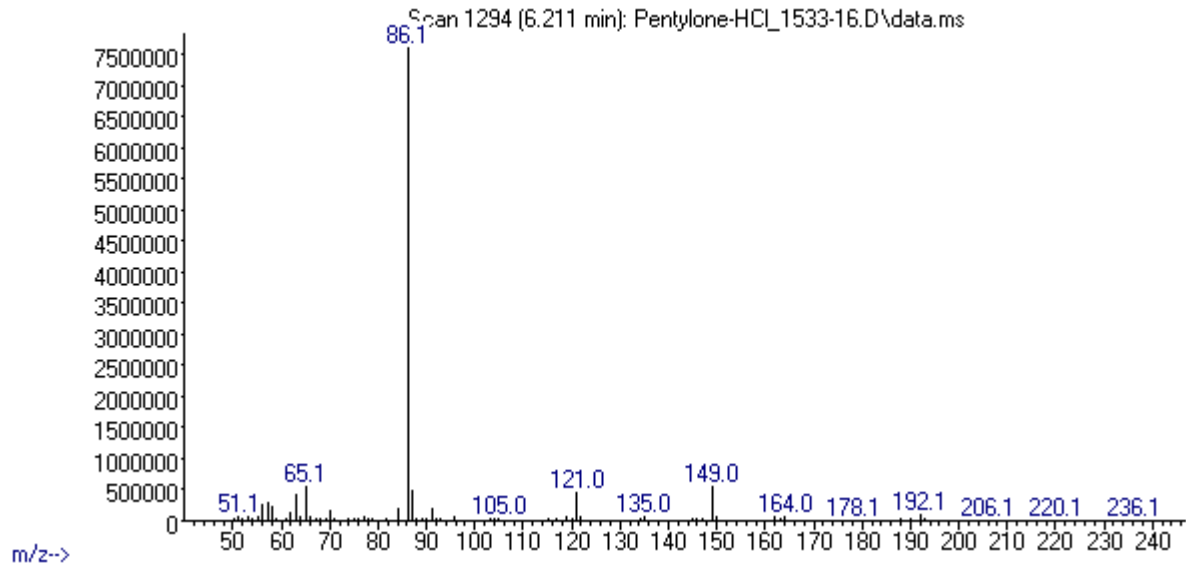
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	partially
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 6,21 BP(1): 86; BP(2): 149,BP(3) :65,
HPLC-TOF	+	Exact mass (theoretical): 235,1208; measured value Δppm:0,35; formula:C13H17NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		MS spectrum consistent by the one from SWGDRUG library and CAYMAN library
other		IR-condensed phase spectrum match the spectrum from Spectra Analysis library.

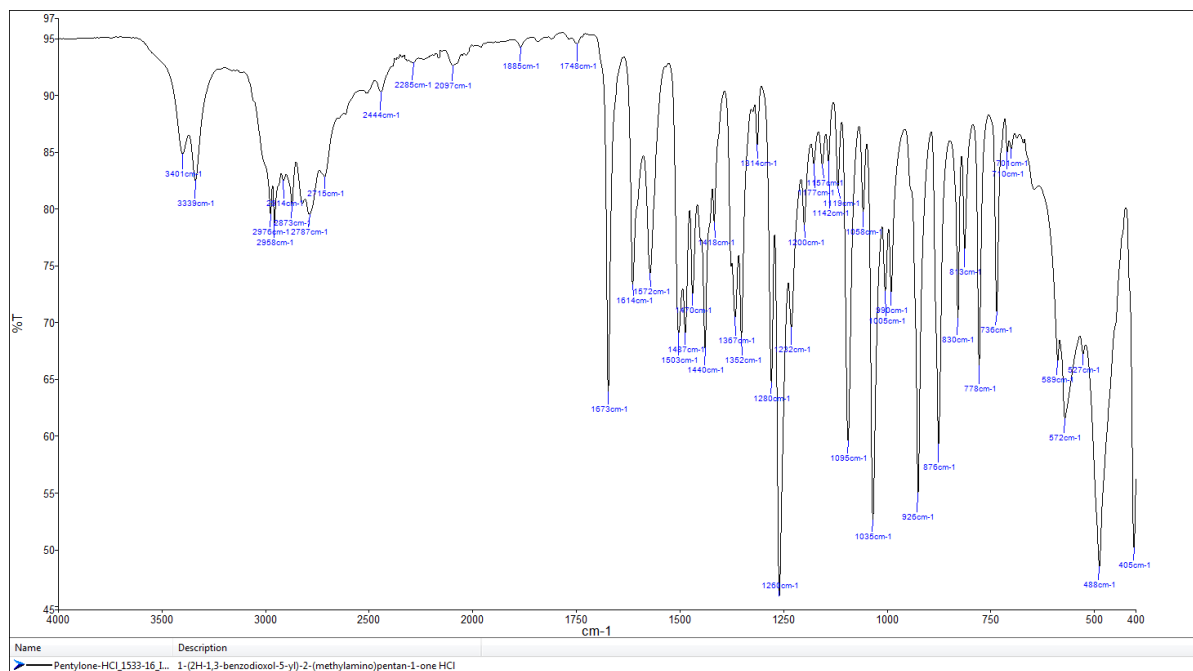
# ANALYTICAL RESULTS

MS (EI)

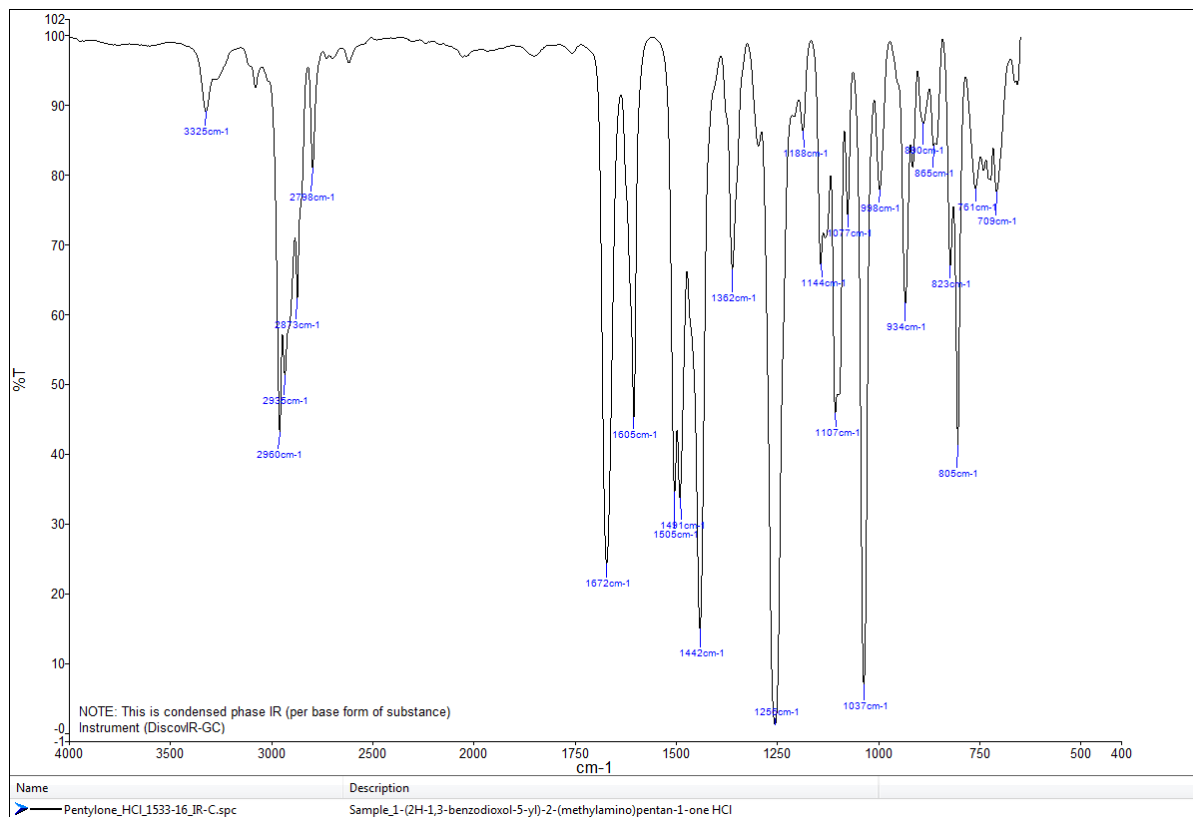
Abundance



## FTIR-ATR - direct measurement (sample as received)



## IR (condensed phase – after chromatographic separation)



# TOF REPORT

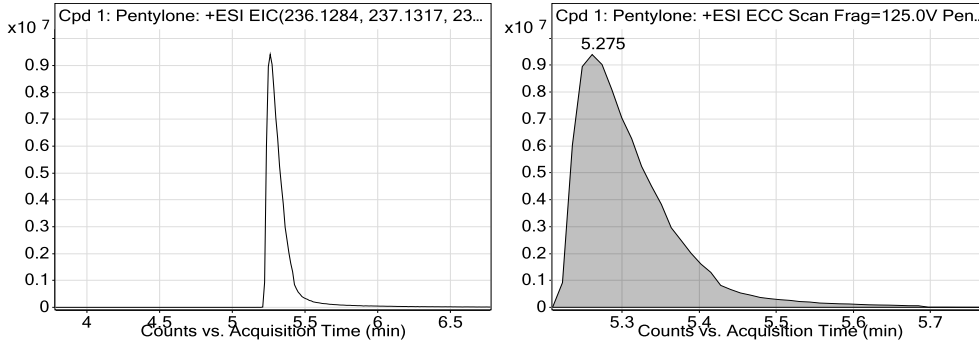
<b>Data File</b>	Pentylone_1533-16_TOF.d	<b>Sample Name</b>	ID_1533-16
<b>Sample Type</b>	Sample	<b>Position</b>	P1-D1
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	general-1512015-XDB-C18-ESI-poz-pod.m	<b>Acquired Time</b>	2/19/2016 9:49:15 AM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	Drugs_NFL.m
<b>Comment</b>	extract in MeOH		

## Compound Table

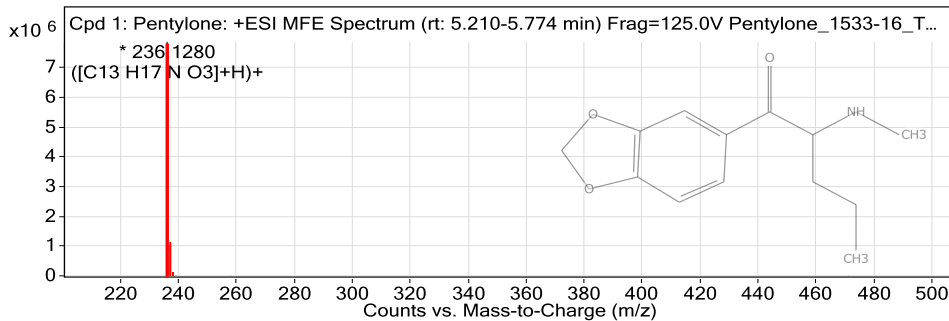
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Pentylone	Pentylone	C13 H17 N O3	5.275	235.1208

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Pentylone	236.128	5.275	235.1208	5.28	C13 H17 N O3	235.1208	0.35

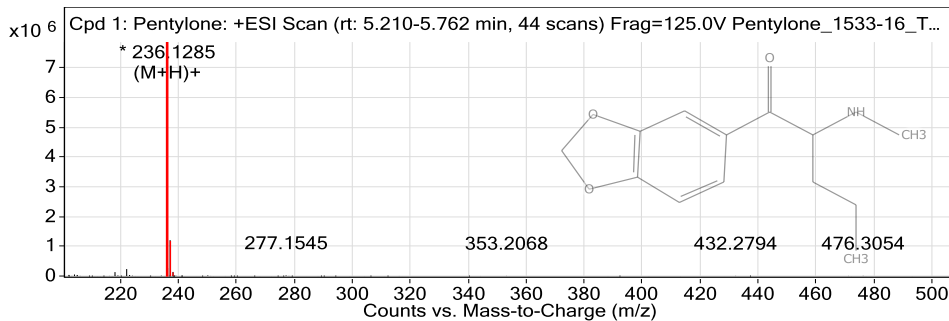
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

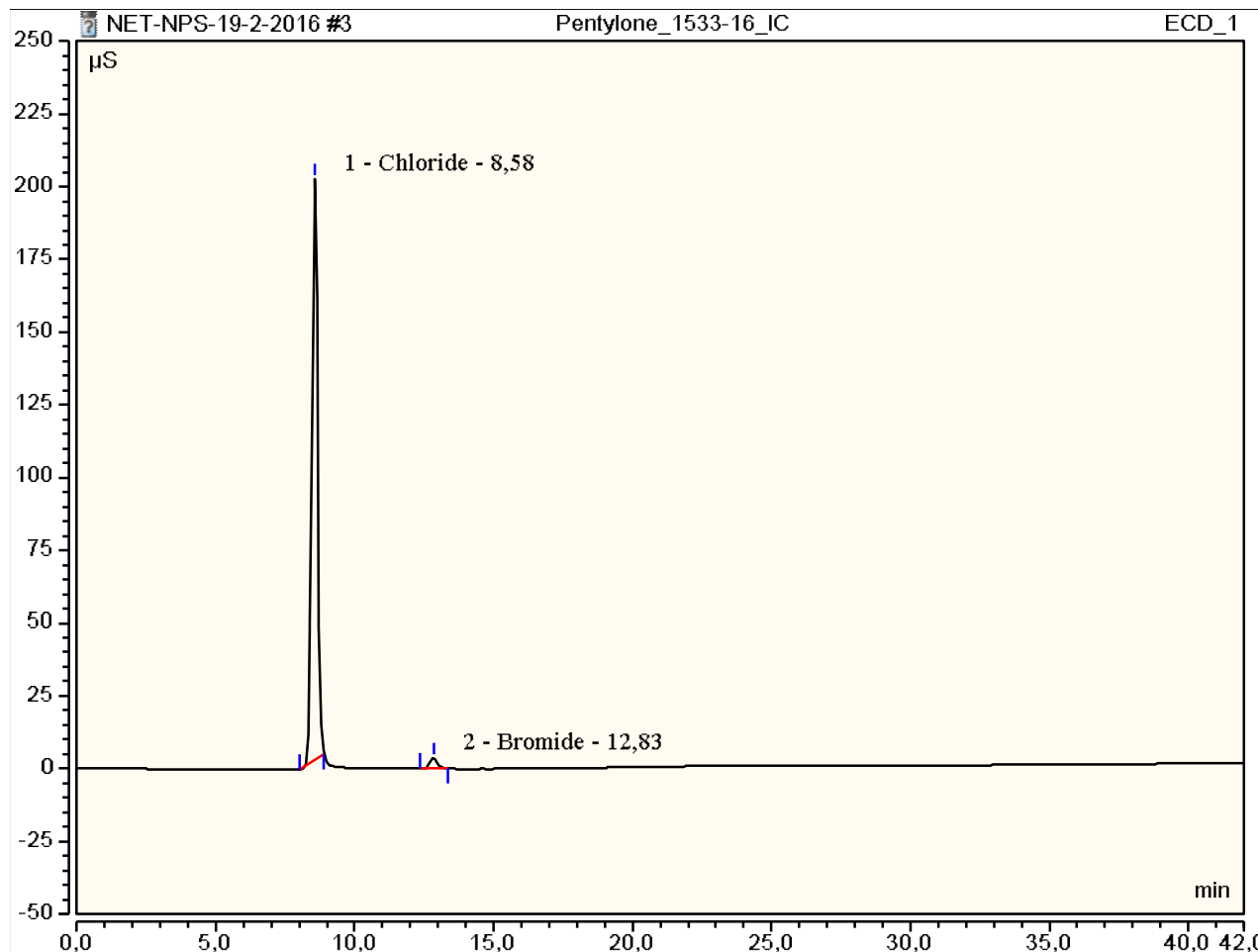
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
236.128	1	7868439.5	C13 H17 N O3	(M+H)+
237.1315	1	1043038.35	C13 H17 N O3	(M+H)+
238.1343	1	128958.09	C13 H17 N O3	(M+H)+
239.1369	1	10980.57	C13 H17 N O3	(M+H)+
240.1398	1	1226.17	C13 H17 N O3	(M+H)+
258.11	1	5768.07	C13 H17 N O3	(M+Na)+
259.1146	1	900.06	C13 H17 N O3	(M+Na)+
471.2479	1	4186.31	C13 H17 N O3	(2M+H)+
472.2513	1	1222.58	C13 H17 N O3	(2M+H)+

--- End Of Report ---

### Peak Integration Report

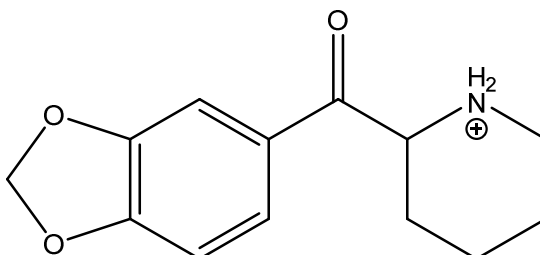
Sample Name:	Pentylone_1533-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	22-feb-2016 / 11:48	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height $\mu\text{S}$	Amount mg/L
1,00	8,58	Chloride	BMB	46,65	199,39	n.a.
2,00	12,83	Bromide	BMB	1,15	3,64	n.a.
TOTAL:				47,80	203,02	0,00





## REPORT

Sample ID:	<b>1533-16</b>
Our notebook code:	P-1533-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- $d_6$
NMR experiments:	$^1\text{H}$ , $^{13}\text{C}$ , $^1\text{H}$ - $^1\text{H}$ <i>gs</i> -COSY, $^1\text{H}$ - $^{13}\text{C}$ <i>gs</i> -HSQC, $^1\text{H}$ - $^{13}\text{C}$ <i>gs</i> -HMBC, $^1\text{H}$ - $^{15}\text{N}$ <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	1-(benzo[ <i>d</i> ][1,3]dioxol-5-yl)- <i>N</i> -methyl-1-oxopentan-2-aminium cation
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure according to NMR.
Supporting information:	Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	May 30, 2016



P-1533-16



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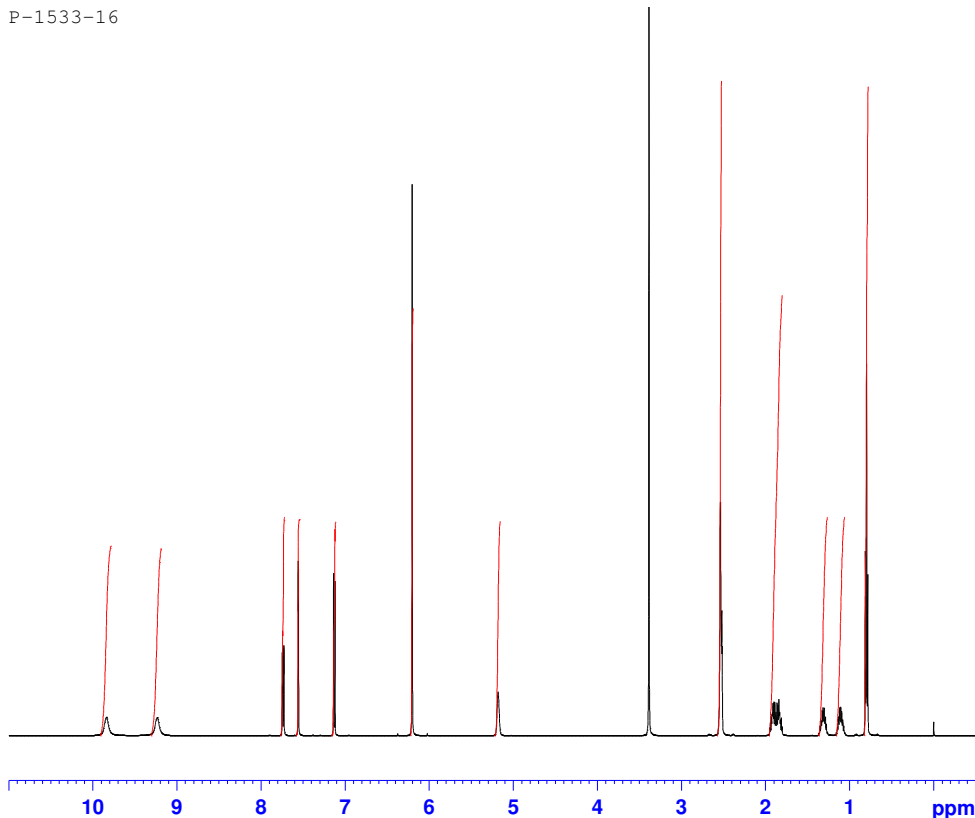
Current Data Parameters
NAME          P-1533-16
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20160425
Time          15.22
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            50.8
DW            50.000 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1          1H
P1            8.90 usec
PLW1          26.00000000 W

F2 - Processing parameters
SI            65536
SF            500.1299939 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

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P-1533-16



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Current Data Parameters
NAME          P-1533-16
EXPNO         3
PROCNO        1

F2 - Acquisition Parameters
Date_         20160425
Time          17.57
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            4096
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG            2050
DW            16.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          125.7703637 MHz
NUC1          13C
P1            9.00 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         0.32179001 W
PLW13         0.16186000 W

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

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