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EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

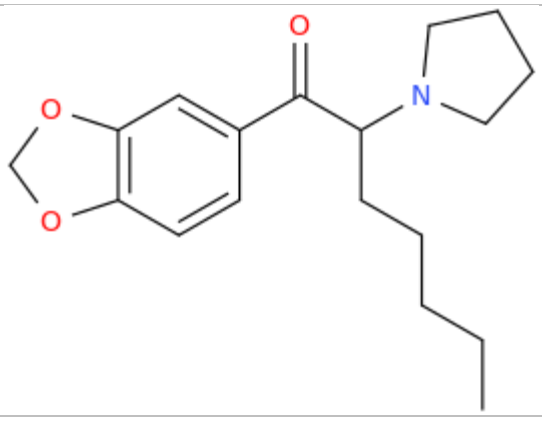
ANALYTICAL REPORT

MDPEP (C18H25NO3)

1-(2H-1,3-benzodioxol-5-yl)-2-(pyrrolidin-1-yl)heptan-1-one

Remark – other compounds detected: 5,6-dibromo-2H-1,3-benzodioxole; pyrrolidine

Sample ID:	2083-19
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of entry (DD/MM/YYYY) into NFL database:	16/10/2019
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[1] (base form)	
Systematic name	1-(2H-1,3-benzodioxol-5-yl)-2-(pyrrolidin-1-yl)heptan-1-one
Other names	1-(1,3-benzodioxol-5-yl)-2-pyrrolidin-1-yl-heptan-1-one; 3,4-methylenedioxy-PV8; 1-(benzo[d][1,3]dioxol-5-yl)-2-(pyrrolidin-1-yl)heptan-1-one
Formula (per base form)	C18H25NO3
M_w (g/mol)	303,4
Salt form/anions detected	HCl
StdInChIKey (per base form)	APQUWEZHVKBPTI-UHFFFAOYSA-N
Other NPS detected	5,6-dibromo-2H-1,3-benzodioxole; pyrrolidine
Additional info (purity..)	MDPEP and 5,6-dibromo-2H-1,3-benzodioxole and pyrolidine in ratio of 1:0.14:0.25 based on 1H NMR spectrum

[1] 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 KOH 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

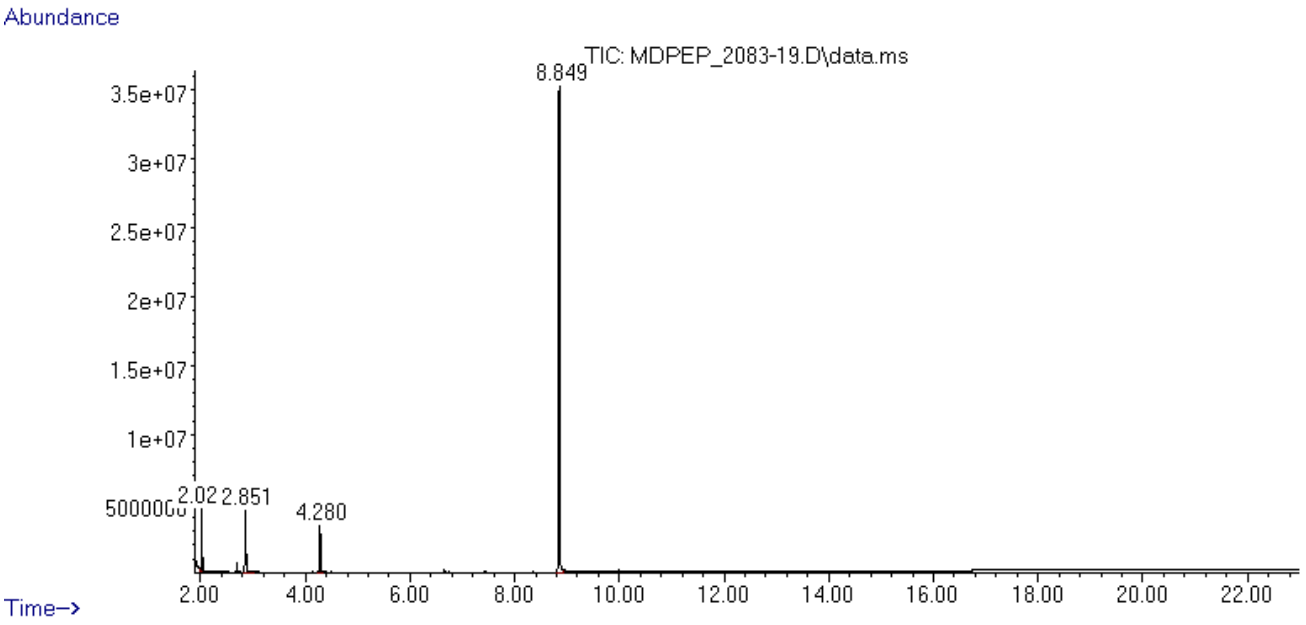
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	partially

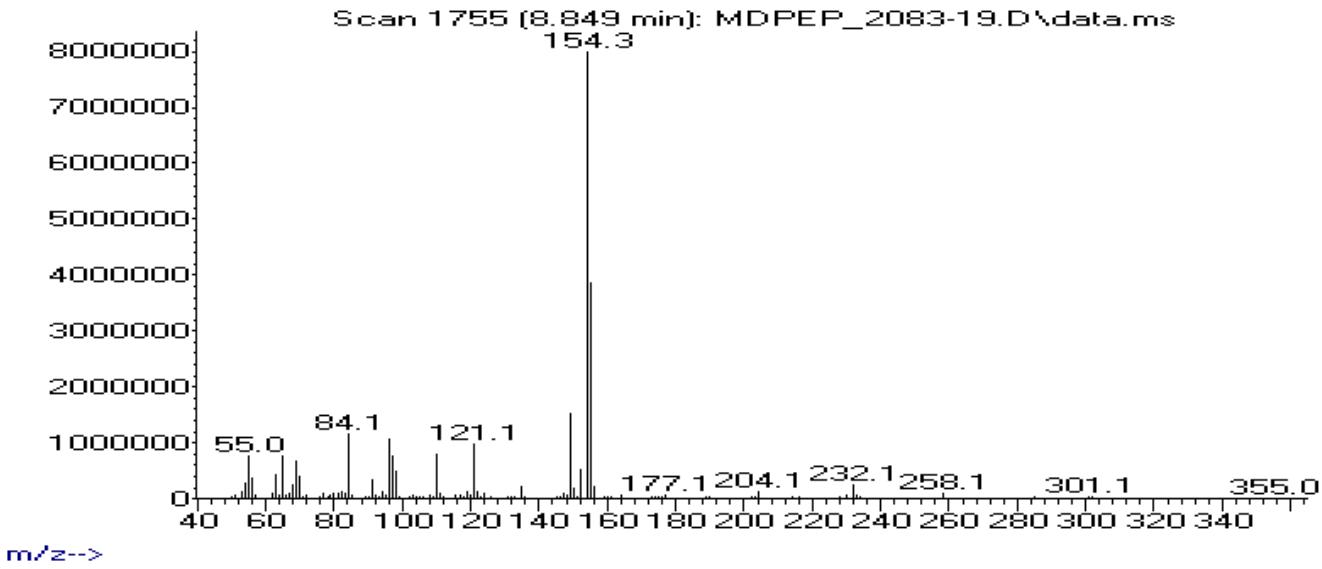
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8,85 BP(1): 154; BP(2): 155,BP(3) :149,
HPLC-TOF	+	Exact mass (theoretical): 303,1834; measured value Δppm:2,15; formula:C18H25NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		MS consistent with HIFS (RESPONSE database entry HIFS-ID-013)
other		

ANALYTICAL RESULTS

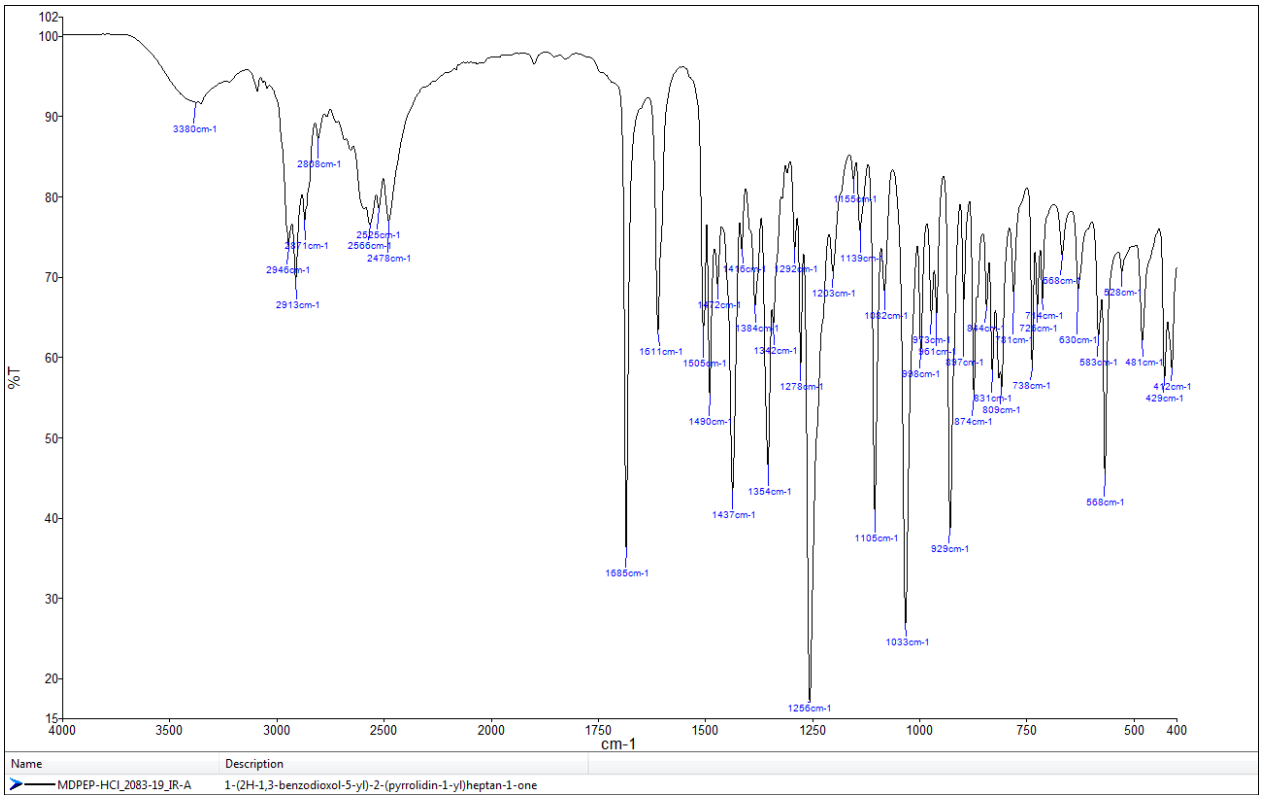
GC-Chromatogram (MDPEP RT=8.849 min)



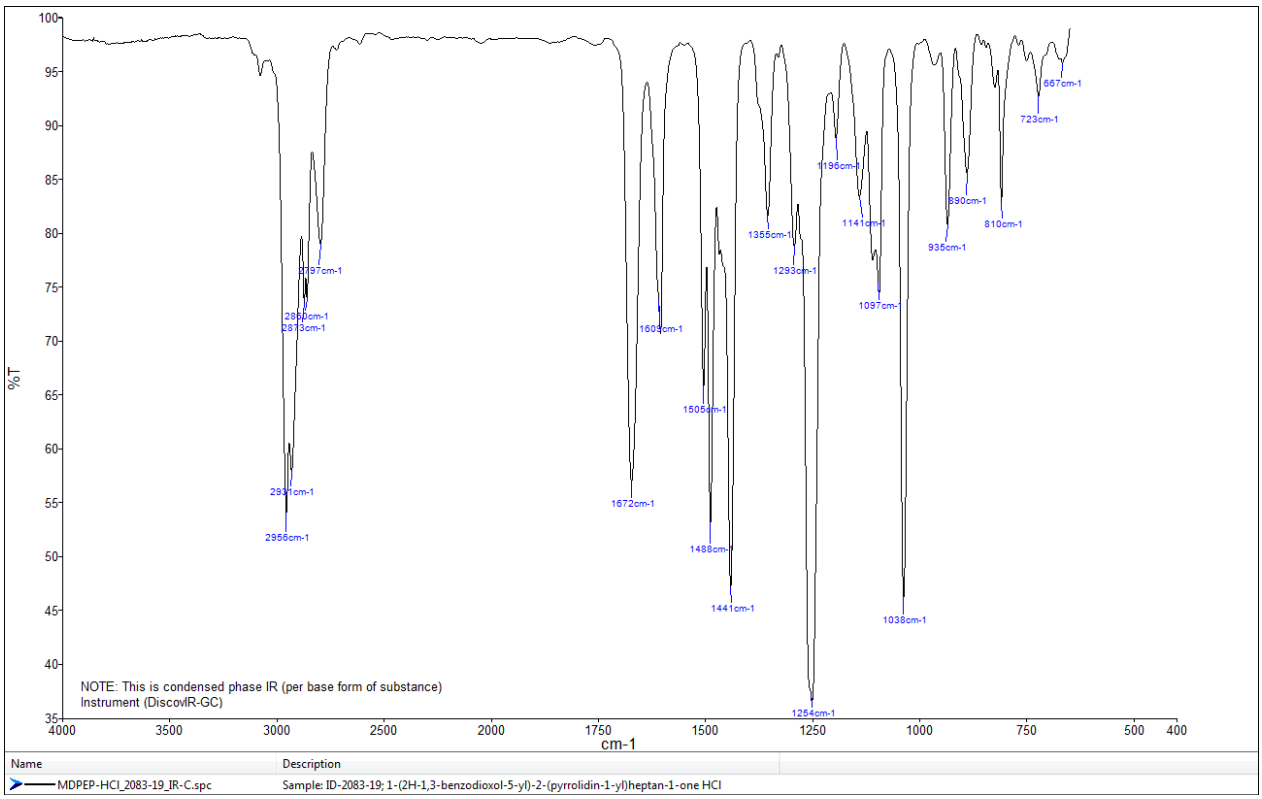
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

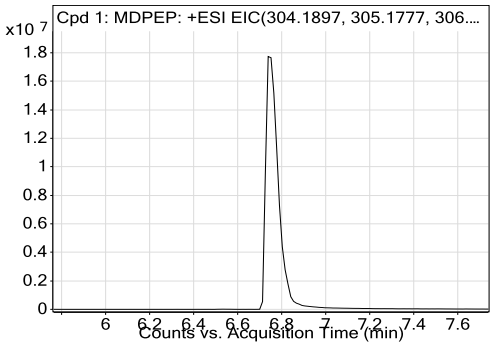
Data File	MDPEP_2083-2019.d	**Sample Name**	ID-2083-19
Sample Type	Sample	**Position**	P1-C4
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	**Acquired Time**	8/5/2019 1:37:23 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

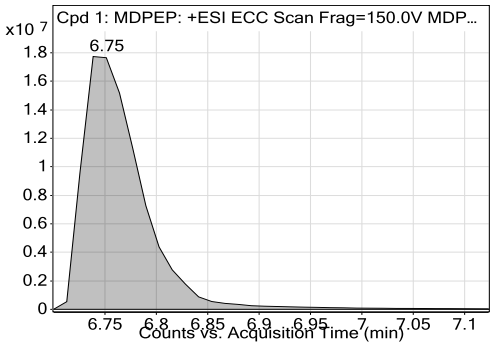
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: MDPEP	MDPEP	C18 H25 N O3	6.75	303.1828

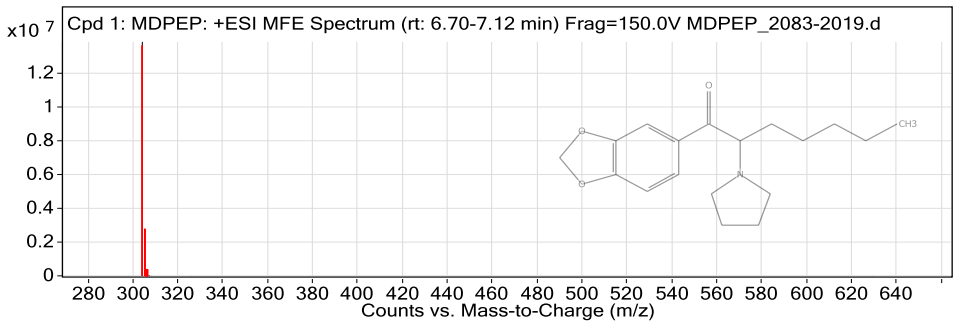
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
MDPEP	304.19	6.75	303.1828	6.75	C18 H25 N O3	303.1834	2.15

Compound Chromatograms

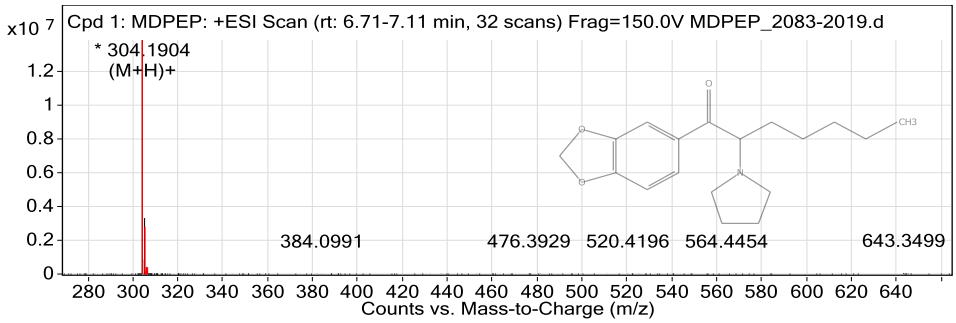




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

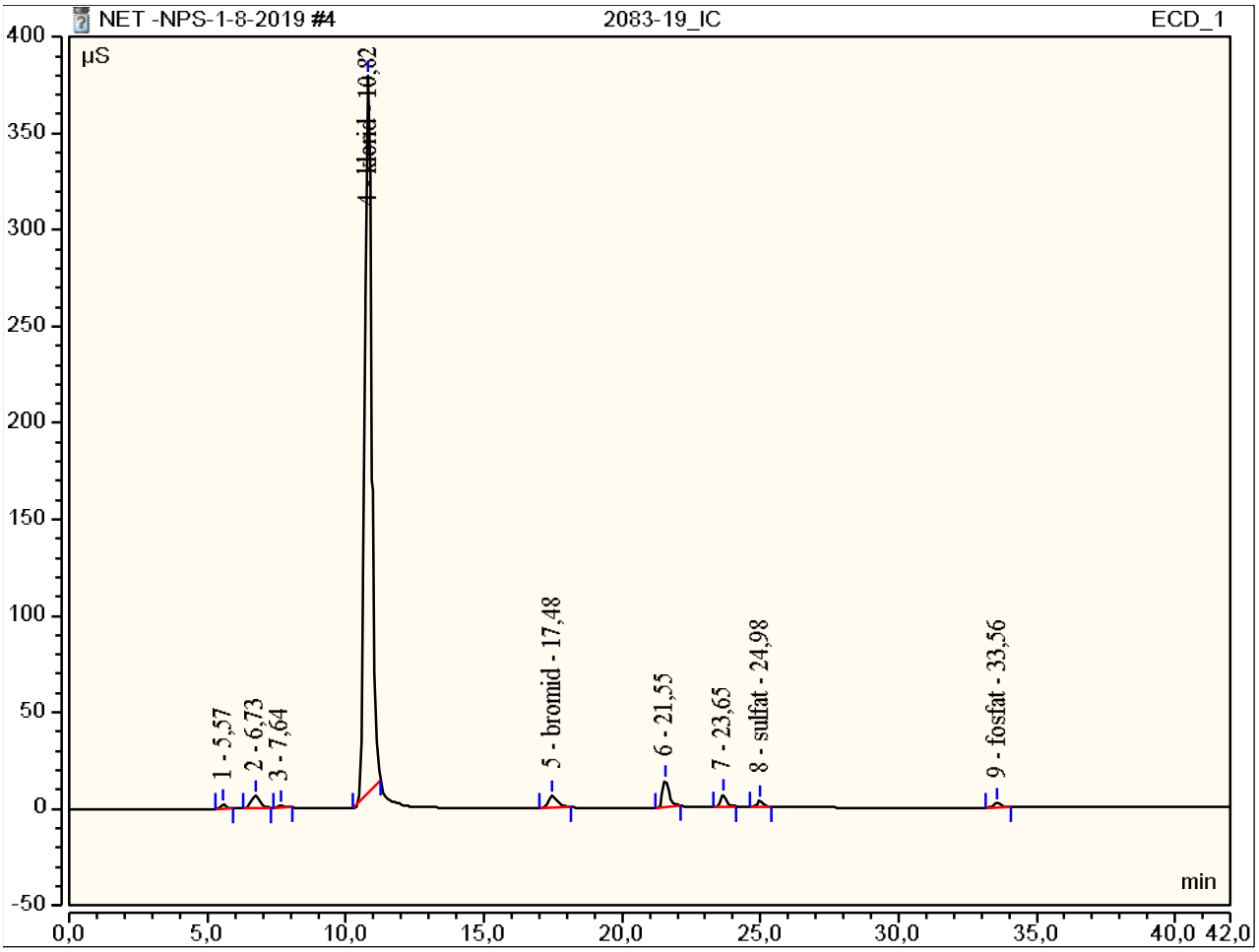
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
304.19	1	13882840	C18 H25 N O3	(M+H)+
305.1938	1	2472500.93	C18 H25 N O3	(M+H)+
306.1973	1	417936.66	C18 H25 N O3	(M+H)+
307.1993	1	44941.7	C18 H25 N O3	(M+H)+
308.2045	1	5616.69	C18 H25 N O3	(M+H)+
326.1725	1	3989.23	C18 H25 N O3	(M+Na)+
629.3547	1	4115.44	C18 H25 N O3	(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**2083-19_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**01-avg-2019 / 09:35**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
4,00	10,82	**klorid**	BMB	105,07	371,33	n.a.
5,00	17,48	**bromid**	BMB	2,36	6,25	n.a.
8,00	24,98	**sulfat**	BMB	0,71	3,00	n.a.
9,00	33,56	**fosfat**	BMB	0,66	2,25	n.a.
		TOTAL:		108,80	382,83	0,00



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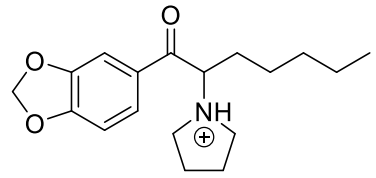
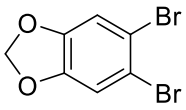
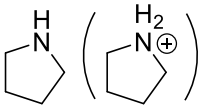
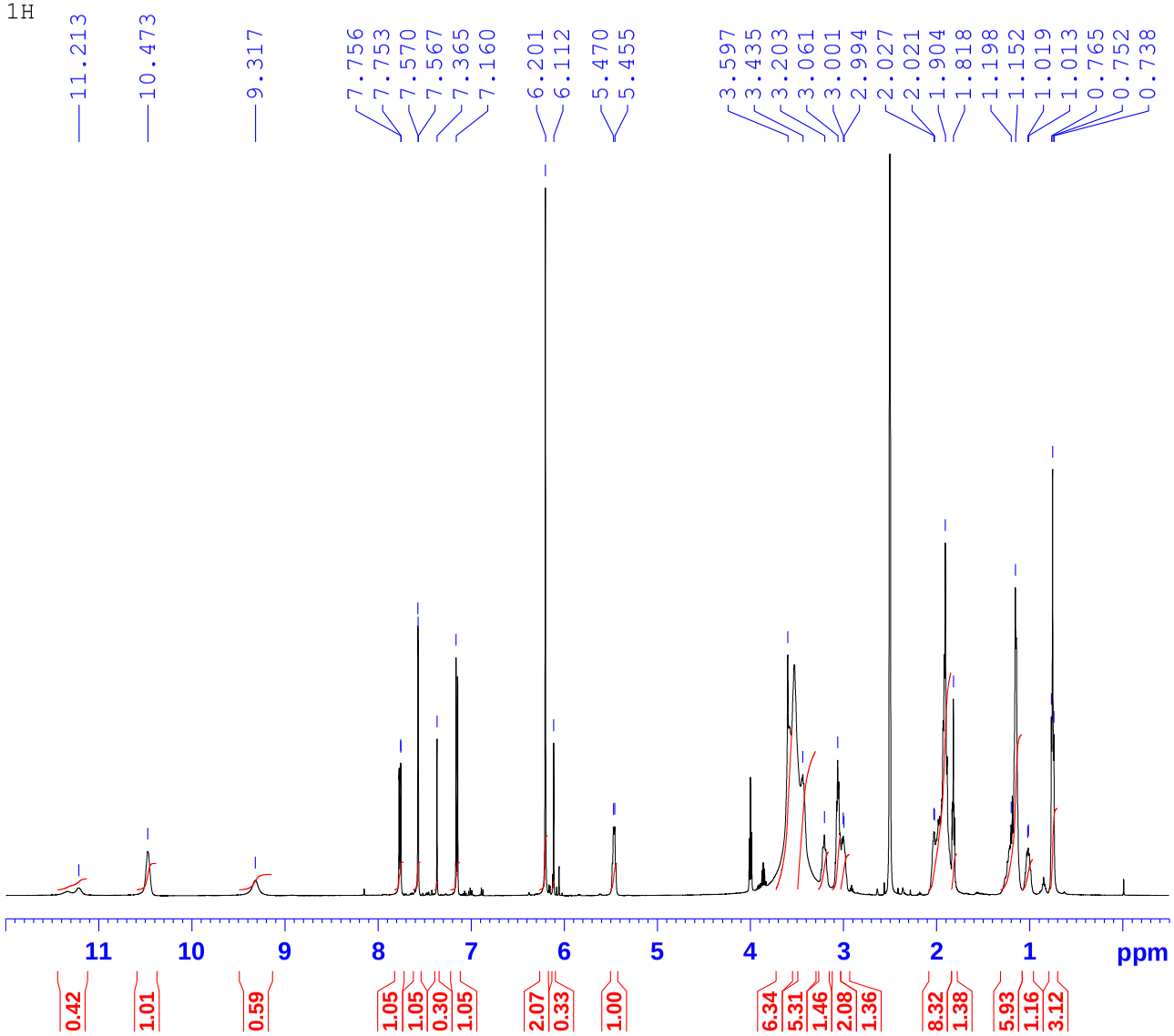
Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**2083-19**
Received date:	October 1, 2019
Our notebook code:	NFL-2083-19
NMR sample preparation:	23.8 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, *gs*-HSQC-TOCSY
Proposed structure with formula, exact mass, molecular weight:	**A**  Chemical Formula: $C_{18}H_{26}NO_3^+$ Exact Mass: 304,1907 Molecular Weight: 304,4095 **B**  Chemical Formula: $C_7H_4Br_2O_2$ Exact Mass: 277,8578 Molecular Weight: 279,9150 **C**  Chemical Formula: C_4H_9N Exact Mass: 71,0735 Molecular Weight: 71,1230
Chemical name:	*N*-protonated 1-(benzo[*d*][1,3]dioxol-5-yl)-2-(pyrrolidin-1-yl)heptan-1-one (**A**), 5,6-dibromobenzo[*d*][1,3]dioxole (**B**), pyrrolidine (**C**)
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - The sample consists of herein identified compounds **A**, **B** and **C** in molar ration 1 : 0.14 : 0.25, based on ^{1}H NMR spectrum.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra, ^{1}H and ^{13}C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	October 11, 2019

NFL-2083-19
1H

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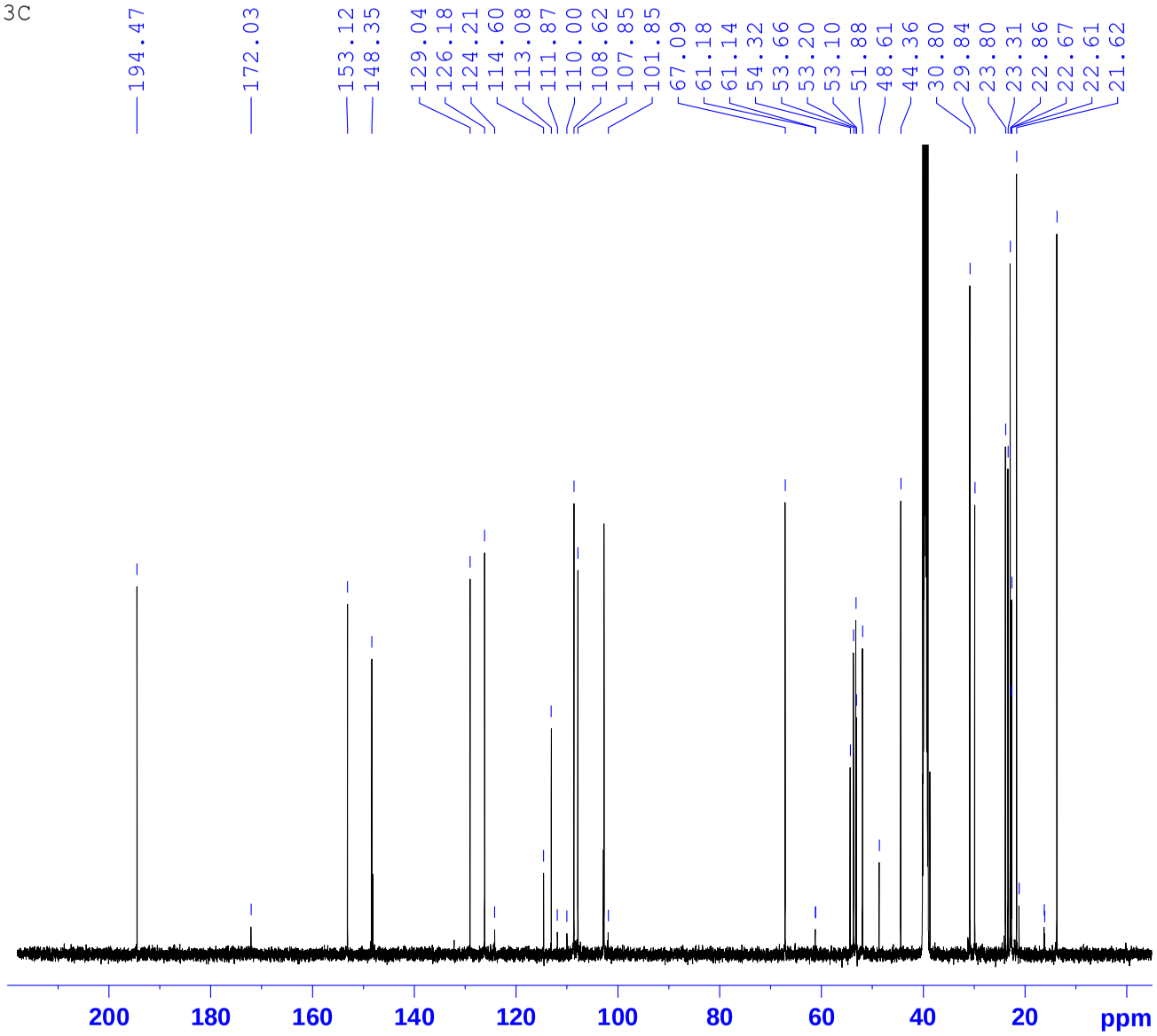
Current Data Parameters
NAME         NFL-2083-19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191001
Time              16.32
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT             DMSO
NS                    32
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                    64
DW               50.000 usec
DE                6.50 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

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

F2 - Processing parameters
SI                 65536
SF           500.1300045 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

NFL-2083-19
13C

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EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191001
Time               19.02
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                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          125.7578438 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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