



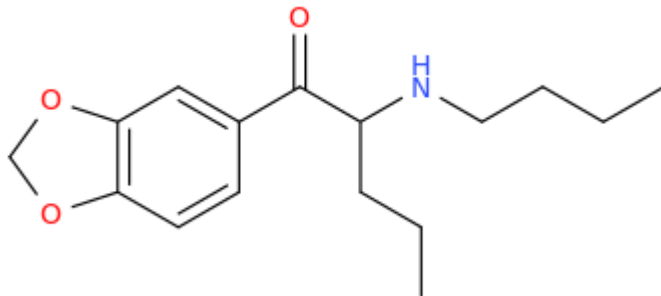
ANALYTICAL REPORT

N-butyl-norpentylone (C₁₆H₂₃N₃O₃)

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

Remark – other NPS detected: 5,6-dibromo-2H-1,3-benzodioxole

Sample ID:	2068-19
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	13/08/2019
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-(butylamino)pentan-1-one
Other names	β-Keto-3,4-methylenedioxy-α-propyl-N-butyl-phenethylamine, N-butylpentylone, BK-BUTYL-K, BK-BBDP
Formula (per base form)	C ₁₆ H ₂₃ N ₃ O ₃
M _w (g/mol)	277,36
Salt form/anions detected	HCl
StdInChIKey (per base form)	SEIQPCGGBMTHCB-UHFFFAOYSA-N
Other NPS detected	5,6-dibromo-2H-1,3-benzodioxole
Additional info (purity..)	BK-BBDP and 5,6-dibromo-2H-1,3-benzodioxole in ratio of 1:0.74 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 °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 7.1 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 (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 °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⁻¹.

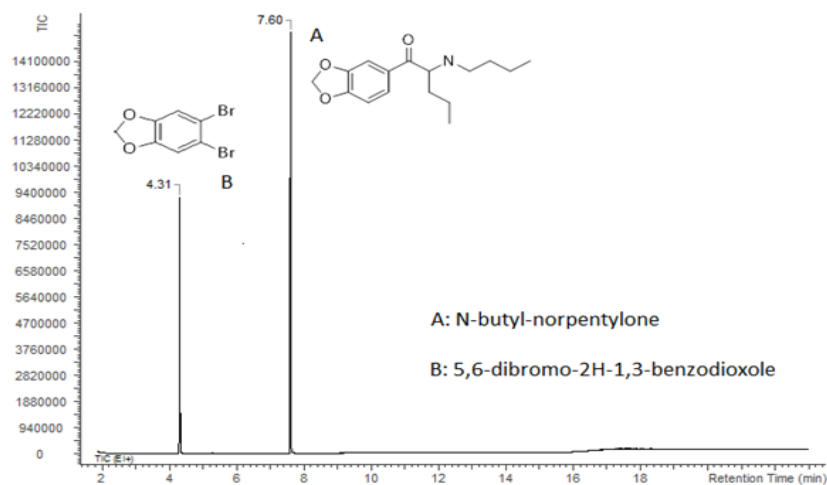
5. IC (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM KOH from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30°C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

Supporting information

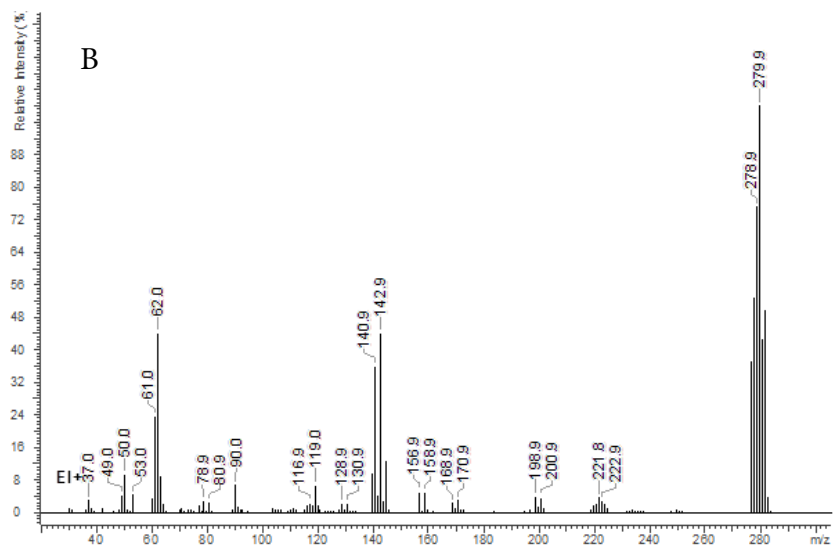
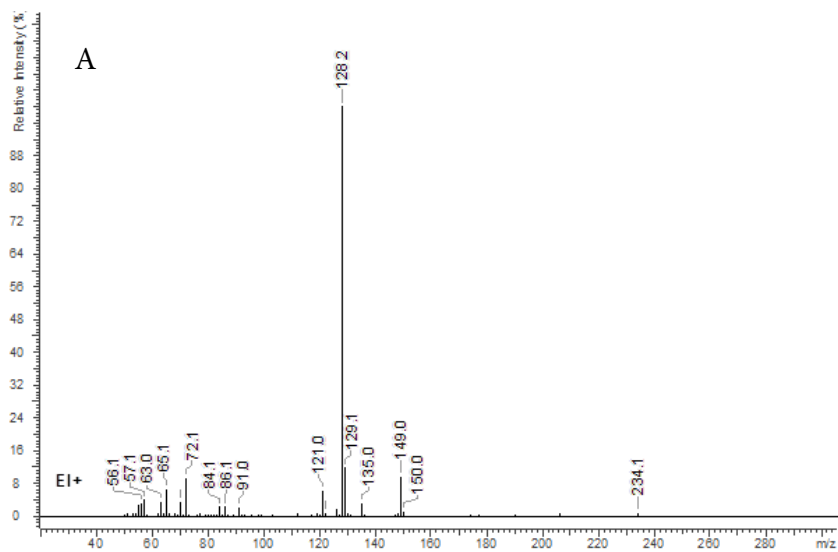
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 7,6 BP(1): 128; BP(2): 129,BP(3) :149,
HPLC-TOF	+	Exact mass (theoretical): 277,1678; measured value Δppm:-0,1; formula:C16H23NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

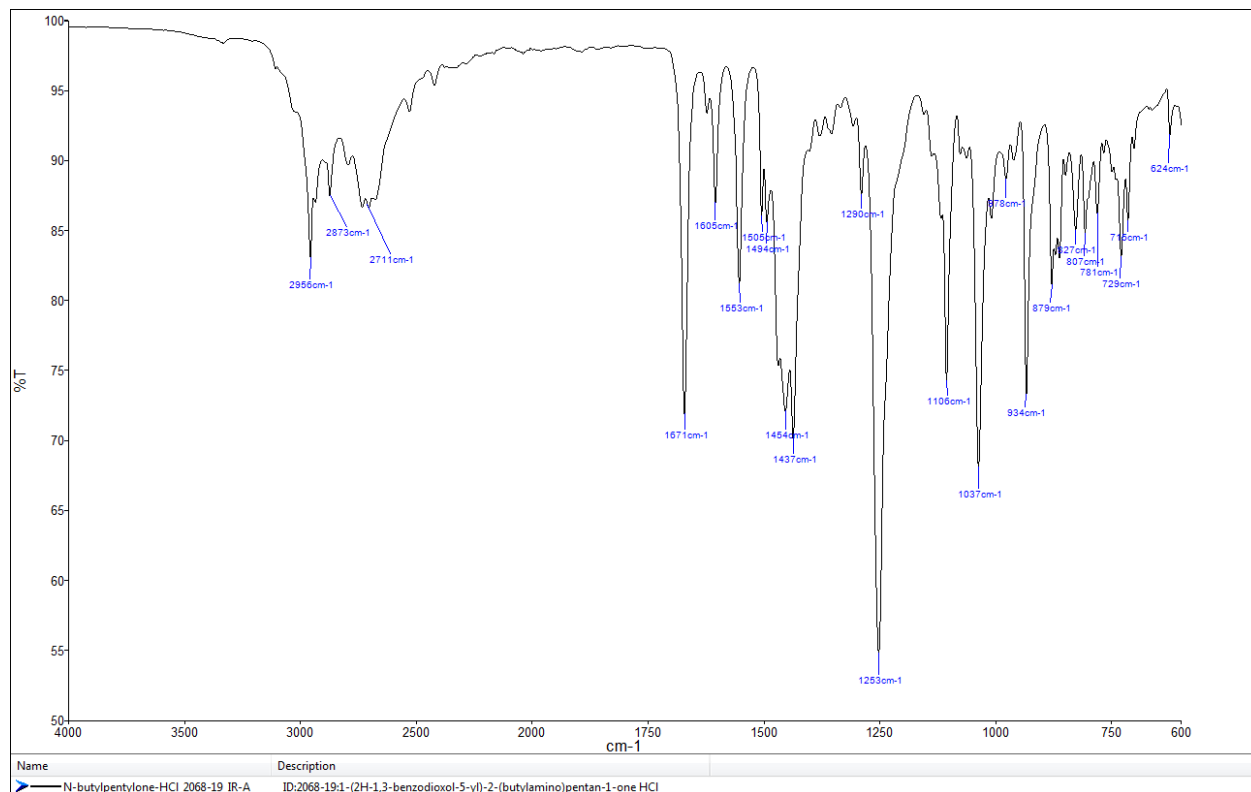
ANALYTICAL RESULTS



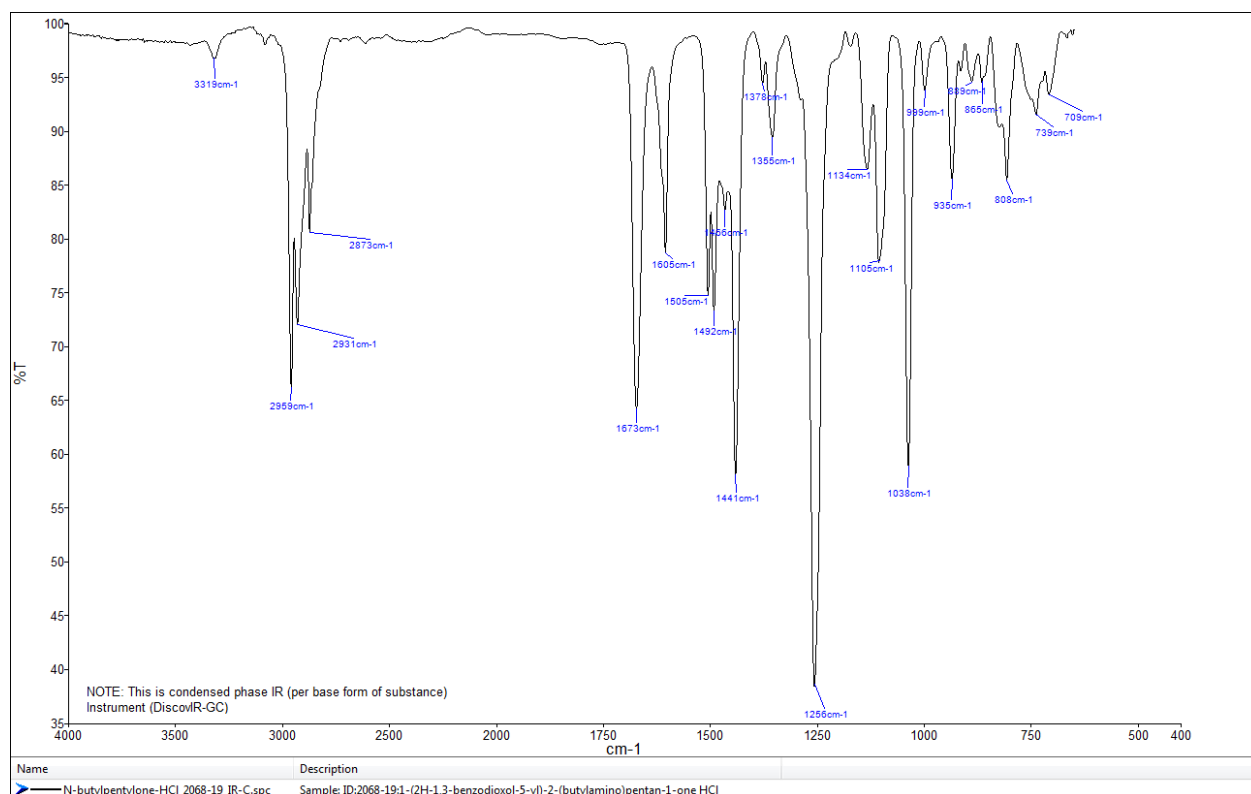
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

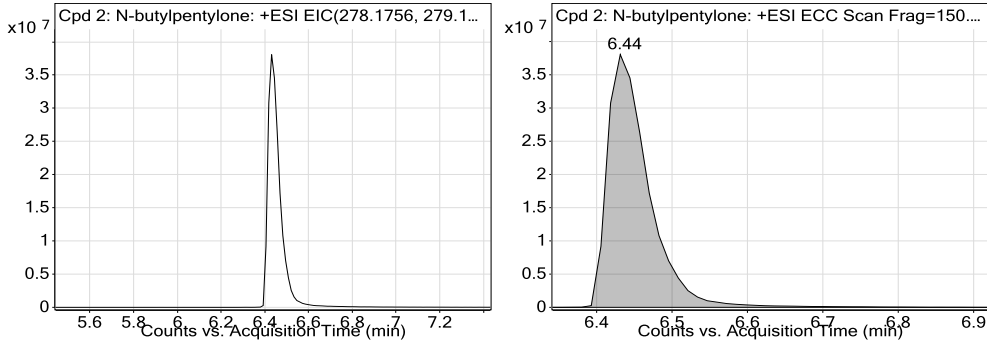
Data File	N-butylpentylone_2068-19.d	Sample Name	ID-2068-19
Sample Type	Sample	Position	P1-A7
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	Acquired Time	5/13/2019 2:06:19 PM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

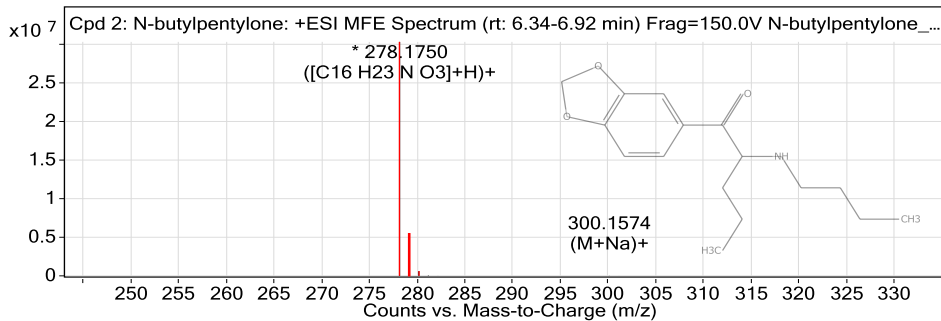
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 2: N-butylpentylone	N-butylpentylone	6.44	277.1678

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
N-butylpentylone	278.175	6.44	277.1678	6.44	C16 H23 N O3	277.1678	-0.1

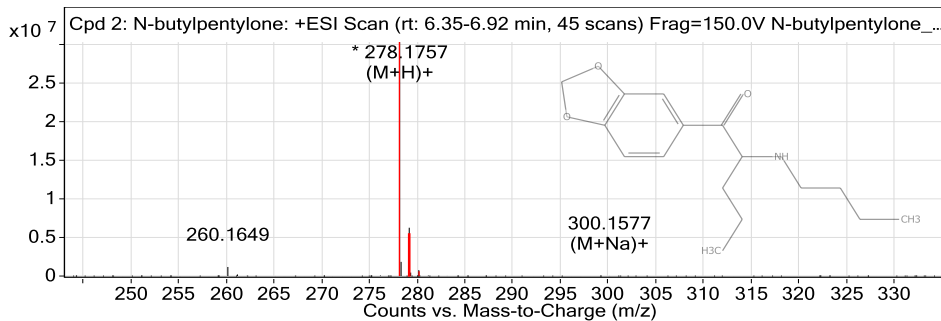
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

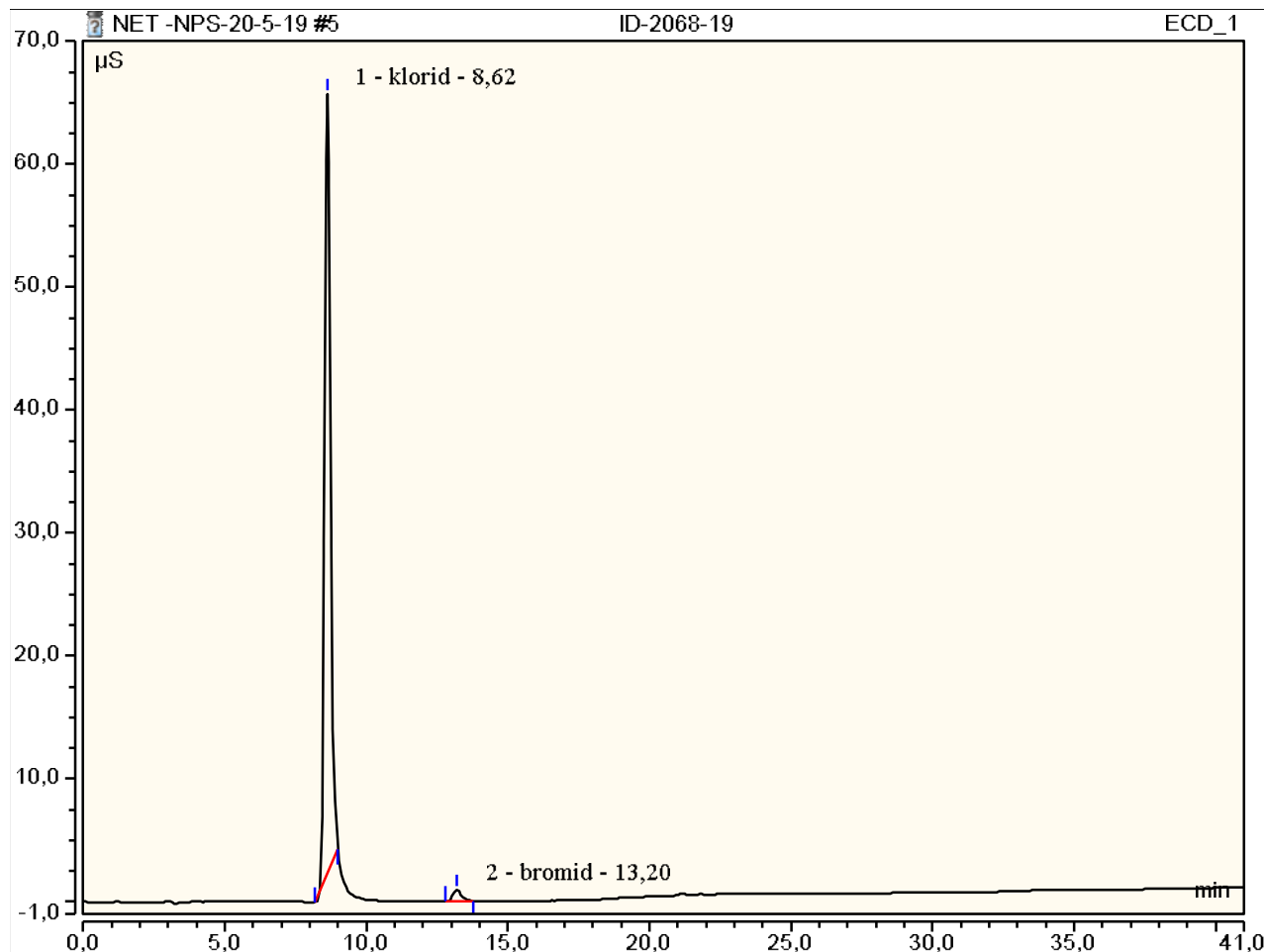
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
278.175	1	30235650	C16 H23 N O3	(M+H)+
279.1786	1	5495746.97	C16 H23 N O3	(M+H)+
280.1816	1	605382.48	C16 H23 N O3	(M+H)+
281.1838	1	53127.76	C16 H23 N O3	(M+H)+
282.1876	1	5974.11	C16 H23 N O3	(M+H)+
300.1574	1	14170.18		(M+Na)+
301.1609	1	4126.74		(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	ID-2068-19	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	20-maj-2019 / 12:07	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	8,62	klorid	BMB	14,64	63,39	n.a.
2,00	13,20	bromid	BMB	0,32	0,97	n.a.
TOTAL:				14,97	64,36	0,00



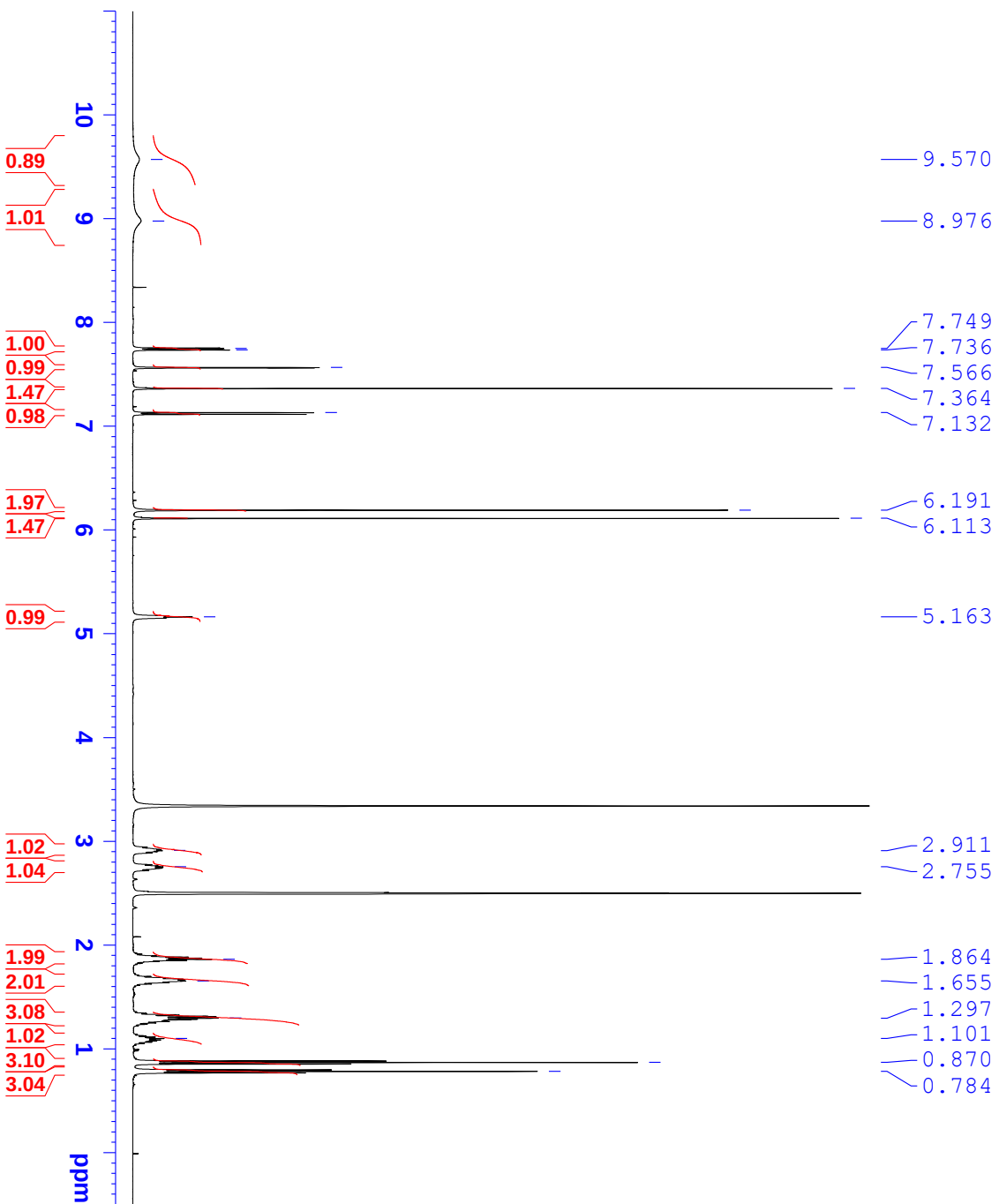
University
of Ljubljana
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2068-19
Received date:	July 30, 2019
Our notebook code:	NFL-2068-19
NMR sample preparation:	19.8 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC
Proposed structure with formula, exact mass, molecular weight:	A Chemical Formula: C₁₆H₂₄NO₃⁺ Exact Mass: 278,1751 Molecular Weight: 278,3715 B Chemical Formula: C₇H₄Br₂O₂ Exact Mass: 277,8578 Molecular Weight: 279,9150
Chemical name:	<i>N</i> -protonated 1-(benzo[<i>d</i>][1,3]dioxol-5-yl)-2-(butylamino)pentan-1-one (A), 5,6-dibromobenzo[<i>d</i>][1,3]dioxole (B)
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - The sample consists of herein identified compounds A and B in the molar ratio of 1:0.74, based on ¹H 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:	August 12, 2019

NFL-2068-19
1H



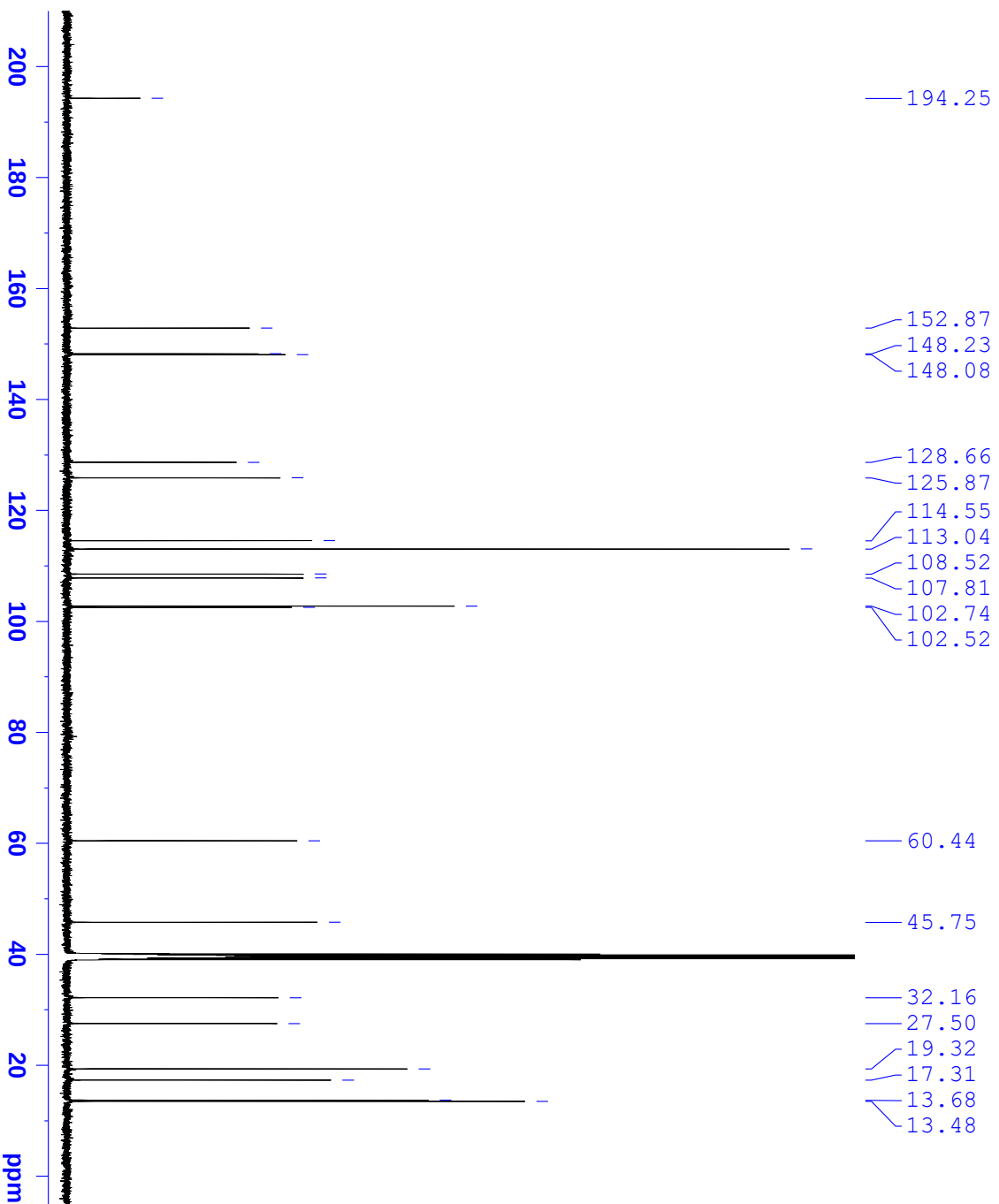
Current Data Parameters
NAME NFL-2068-19
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190801
Time_ 16.35
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 3.2767999 sec
RG 90.5
DW 50.000 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.130885 MHz
NUC1 1H
P1 8.70 usec
PLW1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.130043 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-2068-19
13C



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Current Data Parameters
NAME                NFL-2068-19
EXPNO               2
PROCNO             1

F2 - Acquisition Parameters
Date_              20190801
Time_              18.27
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                 2050
DW                 16.800 usec
DE                 6.50 usec
TE                 298.6 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
CPDPRG2            waltz16
PCPD2              80.00 usec
PLW2                26.00000000 W
PLW12              0.30046001 W
PLW13              0.15113001 W

F2 - Processing parameters
SI                 32768
SF                 125.7578494 MHz
WDW                EM
SSB                0
LB                 1.00 Hz
GB                 0
PC                 1.40
  
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