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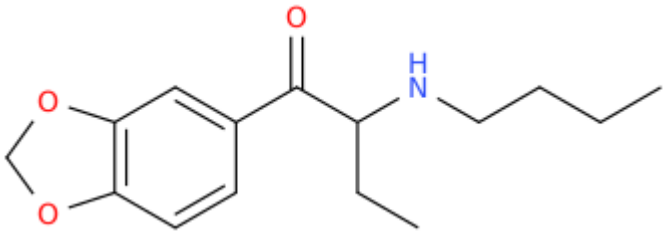
ANALYTICAL REPORT[1]

N-butyl-norbutylone (C15H21NO3)

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

Remark – other NPS detected:

Sample ID:	3058-22
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	23/05/2022
Report 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	1-(2H-1,3-benzodioxol-5-yl)-2-(butylamino)butan-1-one
Other names	
Formula (per base form)	C15H21NO3
M_w (g/mol)	263,34
Salt form/anions detected	HCl
StdInChIKey (per base form)	FCZRKVSLLXPJBA-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	>97% purity of a sample based on 1H NMR spectrum

[1] Approved by: dr. Sonja Klemenc
[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 µl 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 6.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 9.1 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 µl 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

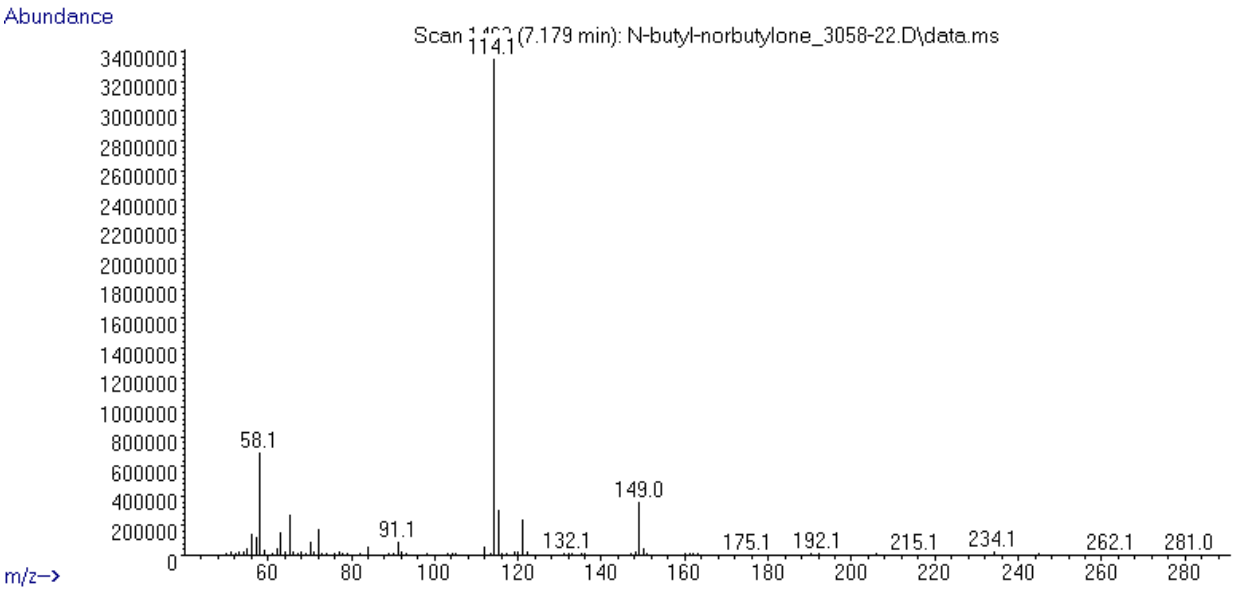
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	partially
H_2O	partially

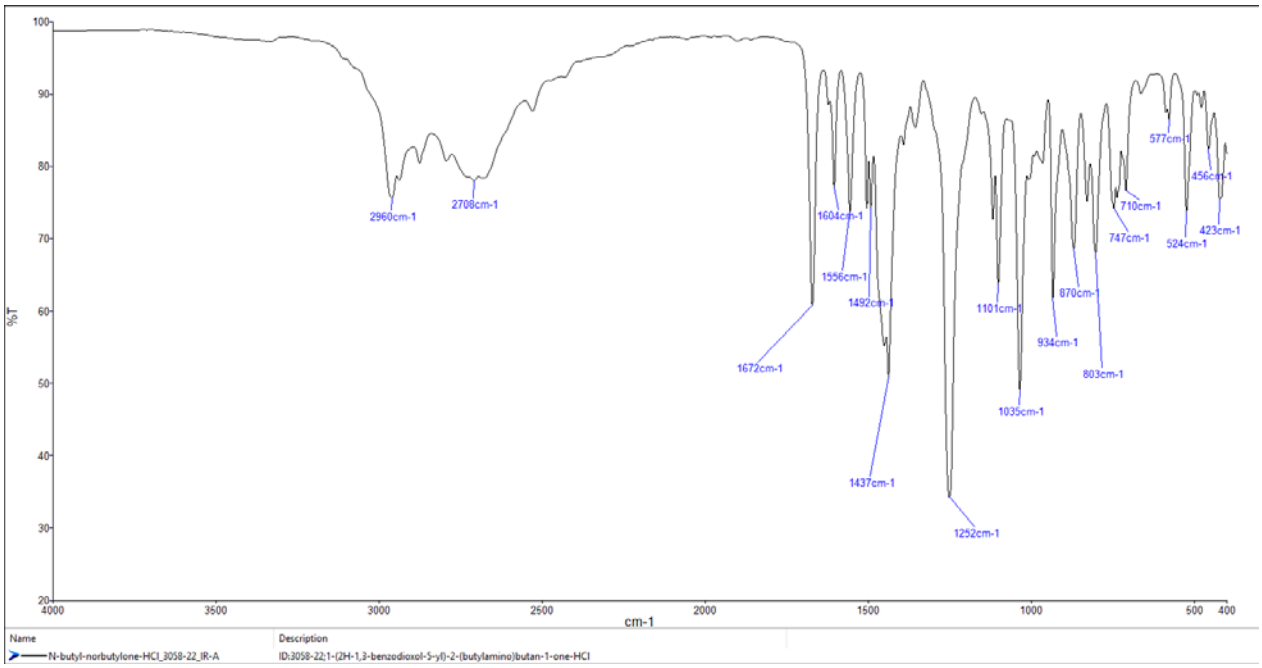
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 7,18 BP(1): 114; BP(2): 58,BP(3) :149,
HPLC-TOF	+	Exact mass (theoretical): 263,1521; measured value Δppm:0,81; formula:C15H21NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

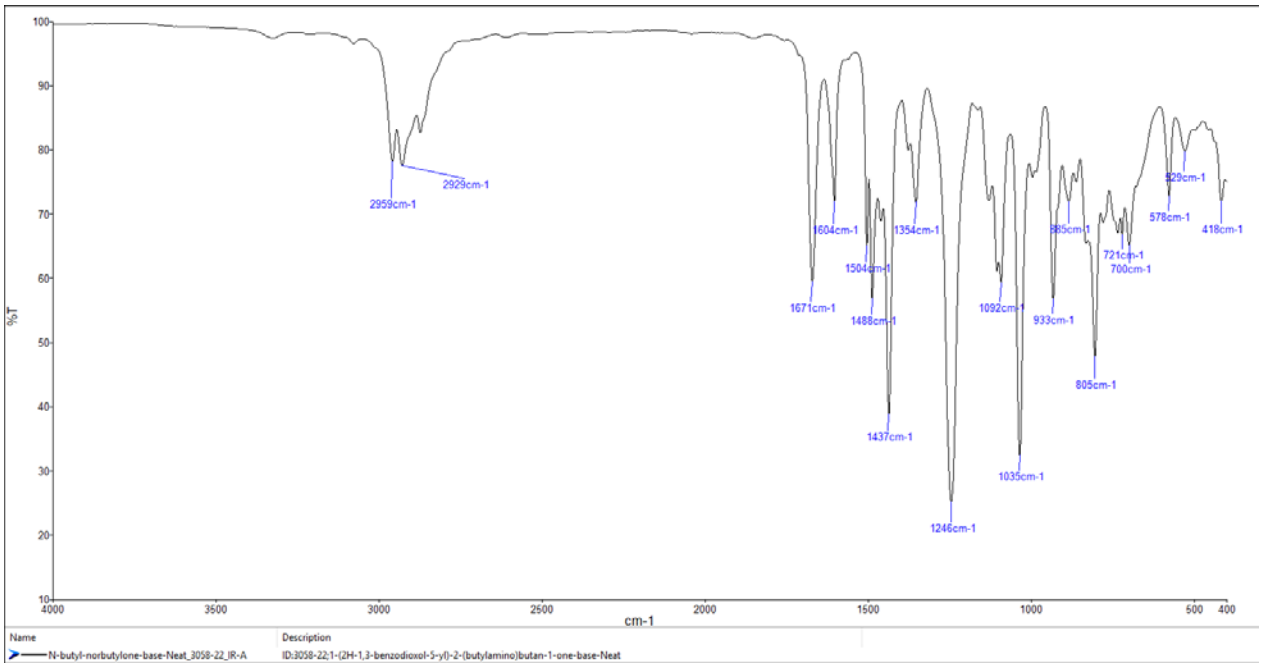
MS (EI)



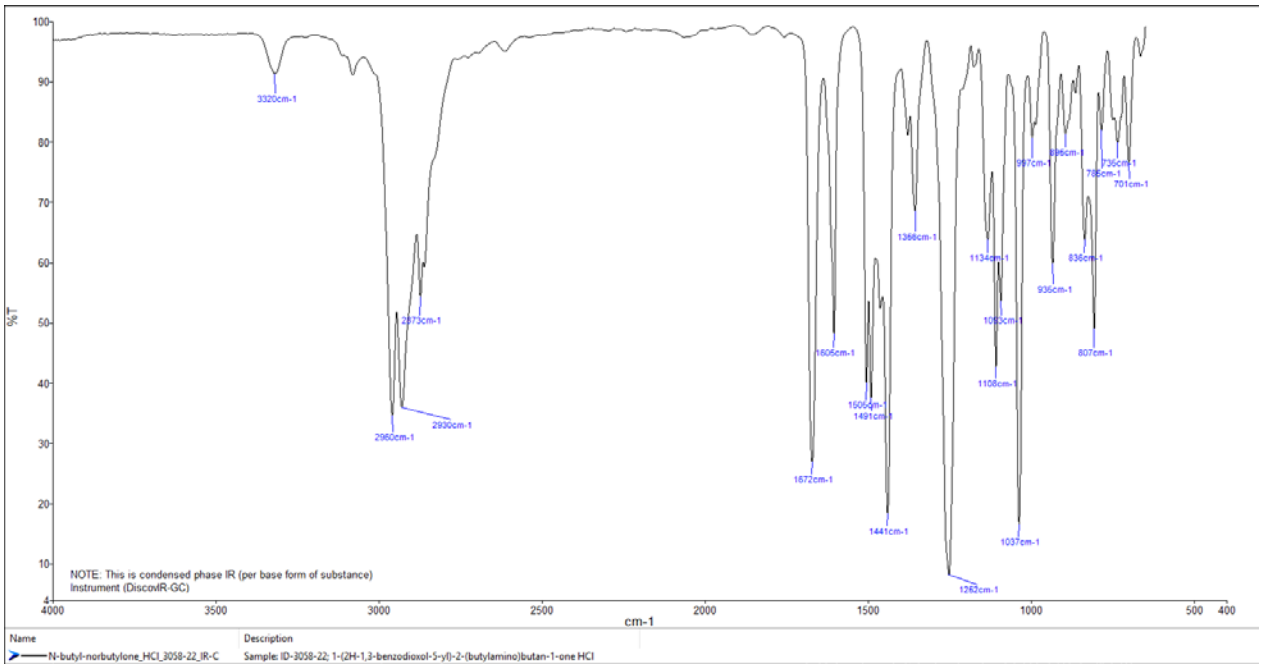
FTIR-ATR - direct measurement (sample as received)



FTIR-ATR (base neat)



IR (solid phase – after chromatographic separation)



TOF REPORT

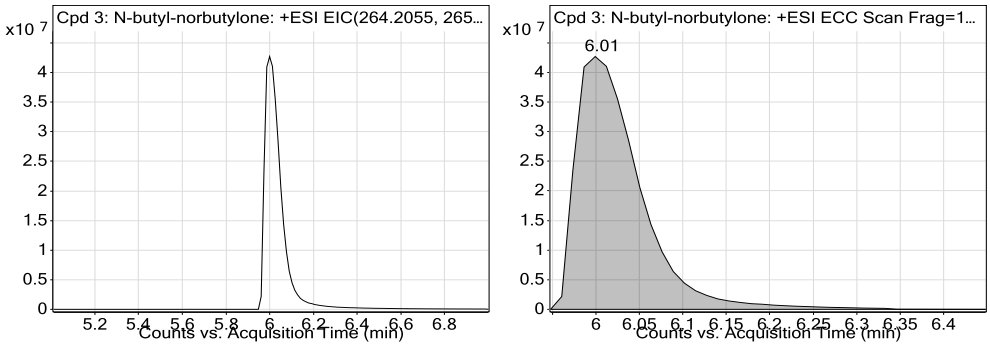
Data File	N-butyl-norbutylone_3058-22.d	**Sample Name**	ID-3058-22
Sample Type	Sample	**Position**	P1-B3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	**Acquired Time**	3/3/2022 1:38:18 PM
IRM Calibration Status	Success	**DA Method**	0-NPS in sorodne snovi.m
Comment	extract in MeOH		

Compound Table

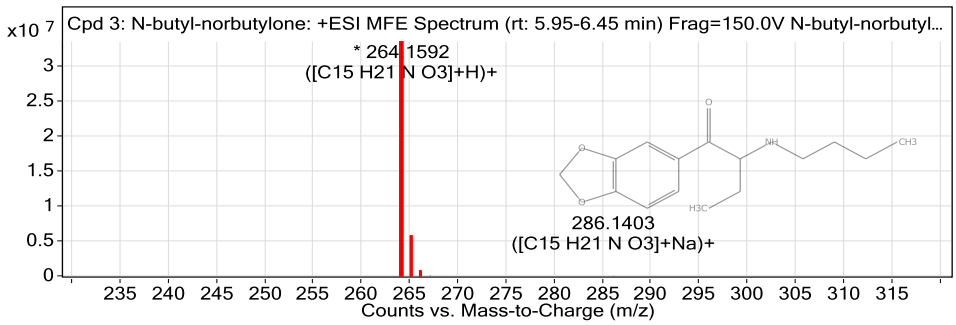
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 3: N-butyl-norbutylone	N-butyl-norbutylone	C15 H21 N O3	6.01	263.1519

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
N-butyl-norbutylone	264.1592	6.01	263.1519	6.01	C15 H21 N O3	263.1521	0.81

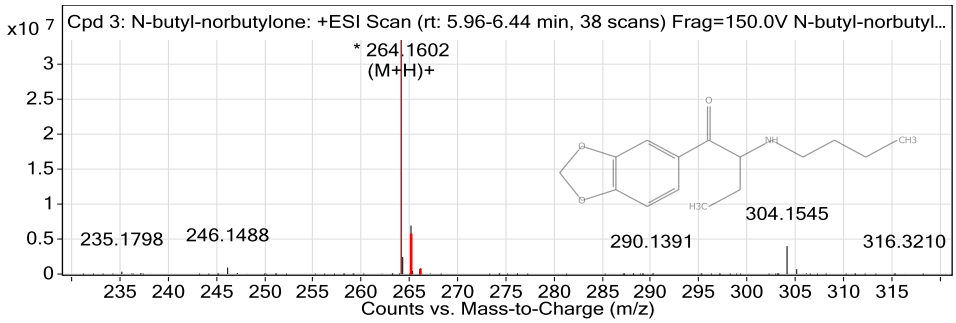
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

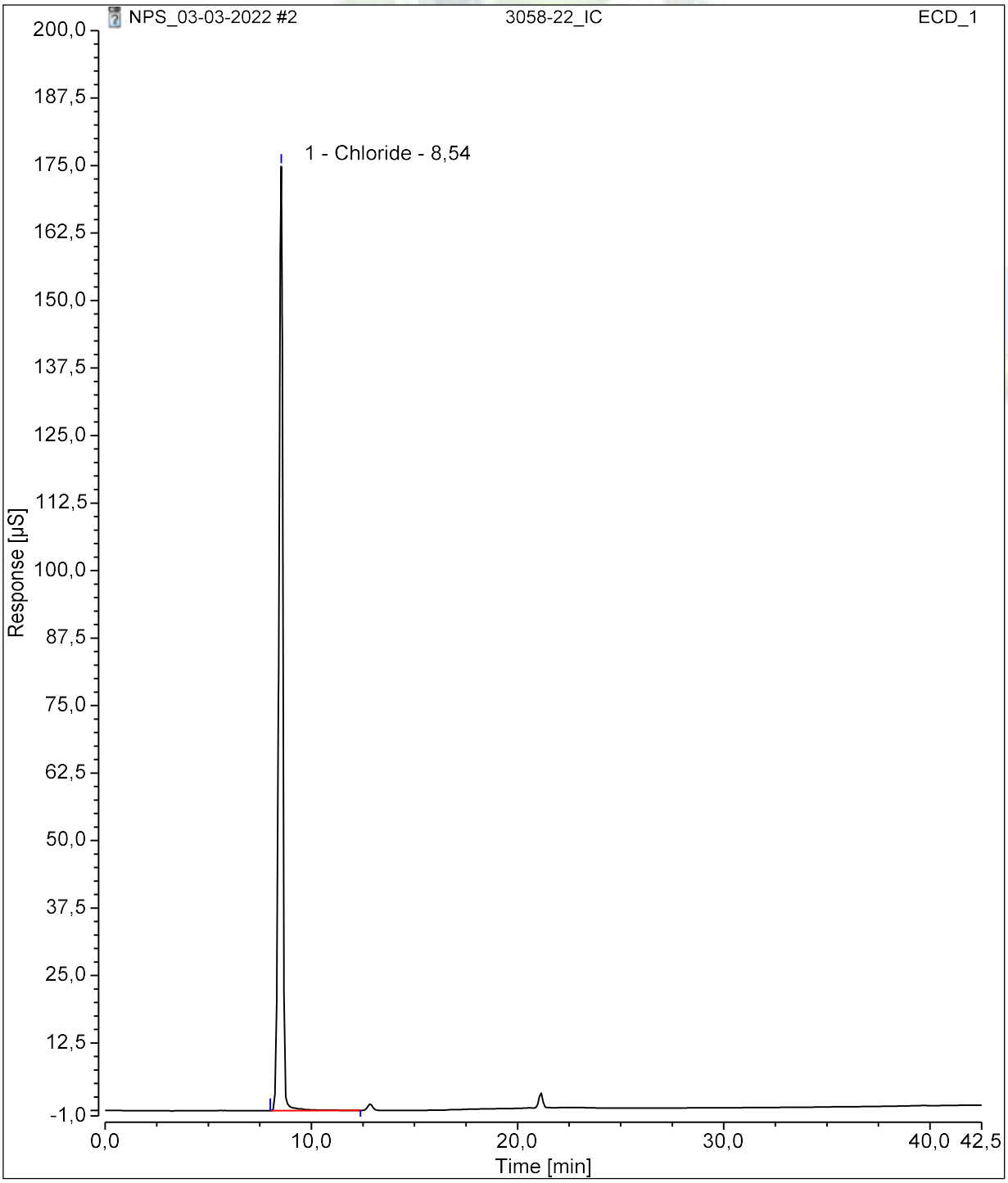
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
264.1592	1	33536184	C15 H21 N O3	(M+H)+
265.1625	1	5822412.97	C15 H21 N O3	(M+H)+
266.1656	1	620892.8	C15 H21 N O3	(M+H)+
267.1676	1	53423.75	C15 H21 N O3	(M+H)+
268.1739	1	6226.75	C15 H21 N O3	(M+H)+
286.1403	1	15978.92	C15 H21 N O3	(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**3058-22_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**Admin**
Inj. Date / Time:	**03-Mar-2022 / 12:03**	*Run Time:*	**43,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1	8,54	**Chloride**	BMB	38,606	174,856	n.a.
		TOTAL:		38,61	174,86	0,0



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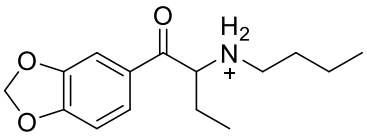
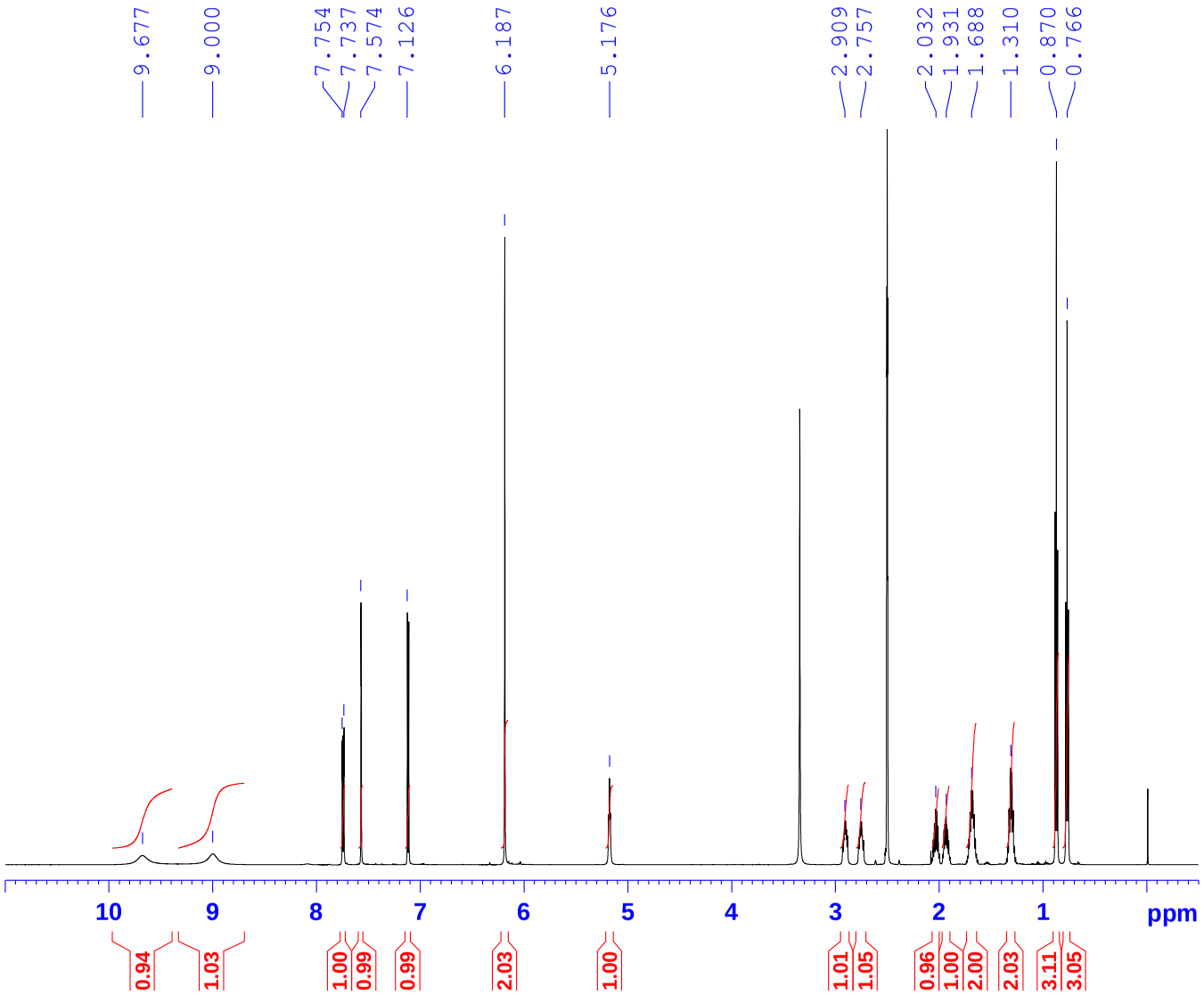
Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-21-460153 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**3058-22**
Received date:	March 14, 2022
Our notebook code:	NFL-3058-22
NMR sample preparation:	19.1 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 formula, exact mass, molecular weight:	 Chemical Formula: $C_{15}H_{22}NO_3^+$ Exact Mass: 264,1594 Molecular Weight: 264,3445
Chemical name:	*N*-protonated 1-(benzo[*d*][1,3]dioxol-5-yl)-2-(butylamino)butan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - Traces of acetone could be detected in the sample. ->97% purity of a sample 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:	April 3, 2022

NFL-3058-22
1H

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Current Data Parameters
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EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
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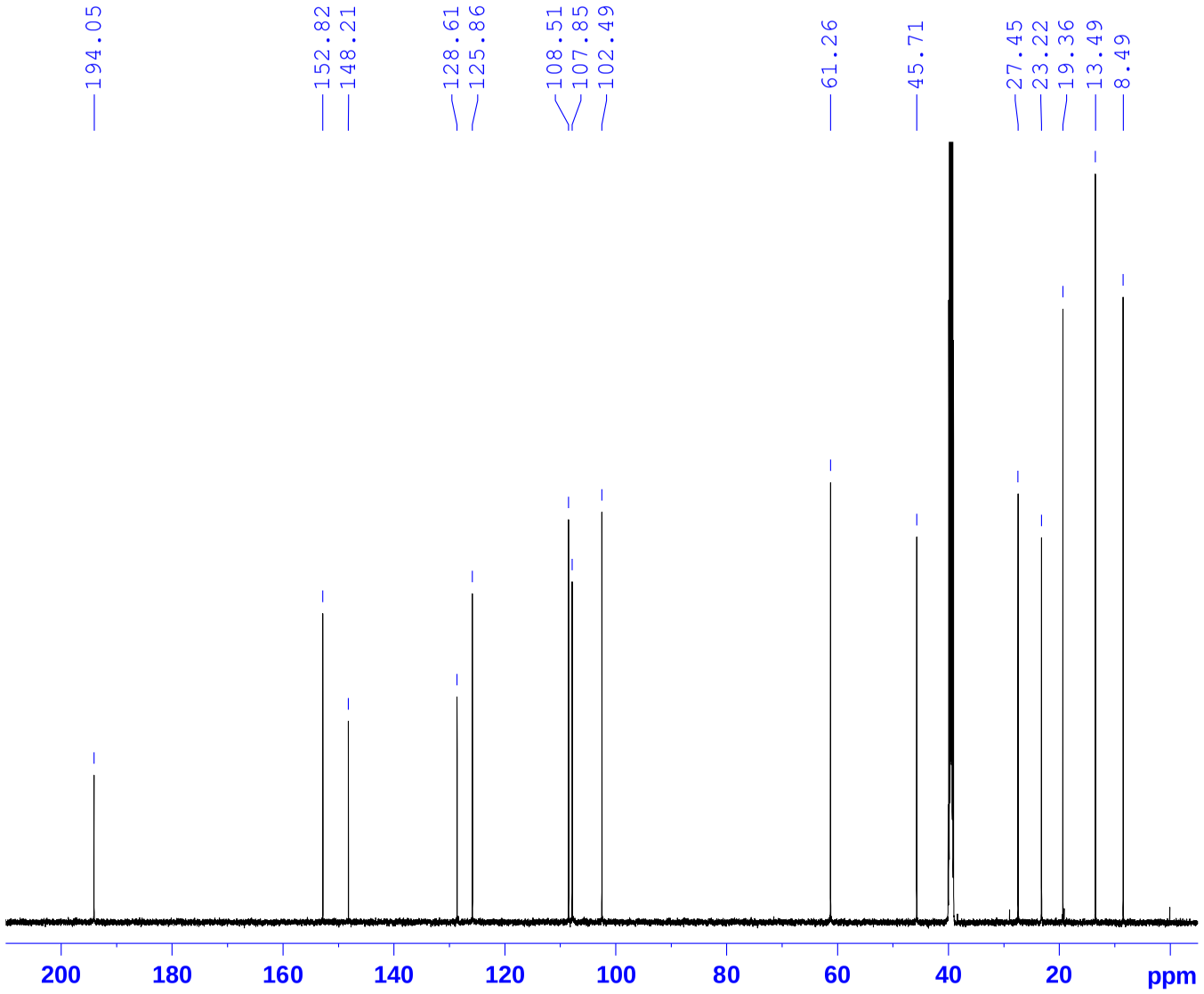
Time              23.39 h
INSTRUM        AV600 NEO
PROBHD   Z176567_0002 (
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH           11904.762 Hz
FIDRES         0.363304 Hz
AQ            2.7525120 sec
RG              59.4286
DW               42.000 usec
DE                8.79 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         600.1337058 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1         16.51399994 W

F2 - Processing parameters
SI                 65536
SF           600.1300051 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

NFL-3058-22
13C



Current Data Parameters
NAME NFL-3058-22
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220315
Time 1.22 h
INSTRUM AV600 NEO
PROBHD Z176567_0002 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3072
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 101.26000214 W
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 16.51399994 W
PLW12 0.33702001 W
PLW13 0.16952001 W

F2 - Processing parameters
SI 32768
SF 150.9028811 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40