NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY
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S podporo skladov Evropske unije na področju notranjih zadev

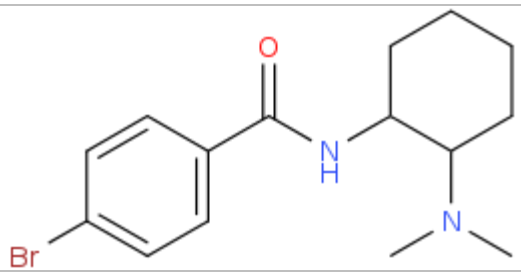
ANALYTICAL REPORT

U-47931E (C15H21BrN2O)

4-bromo-N-[2-(dimethylamino)cyclohexyl]benzamide

Remark – other NPS detected: none

Sample ID:	1869-17
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of sample receipt (M/D/Y):	10/18/2017
Date of entry (M/D/Y) into NFL database:	11/21/2017
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	4-bromo-N-[2-(dimethylamino)cyclohexyl]benzamide
Other names	Bromadoline
Formula (per base form)	C15H21BrN2O
M_w (g/mol)	325.25
Salt form/anions detected	maleate , chloride, sulphate
StdInChIKey (per base form)	UFDJFJYMMIZKLG-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	pure by GC-MS, HPLC-TOF and NMR

[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 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

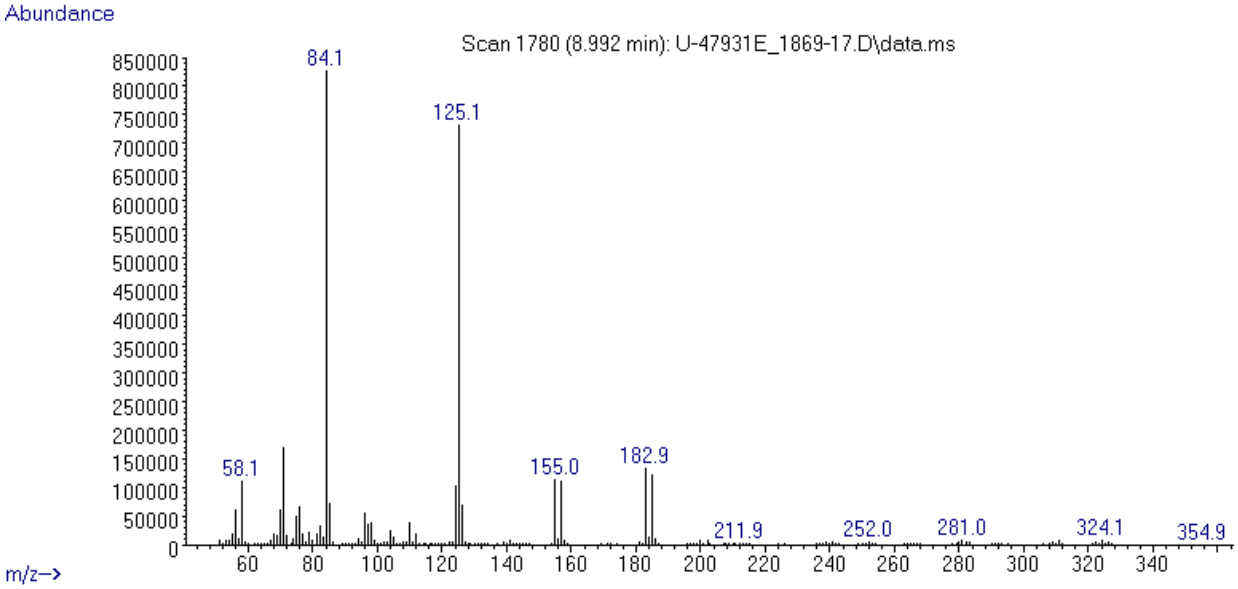
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	soluble

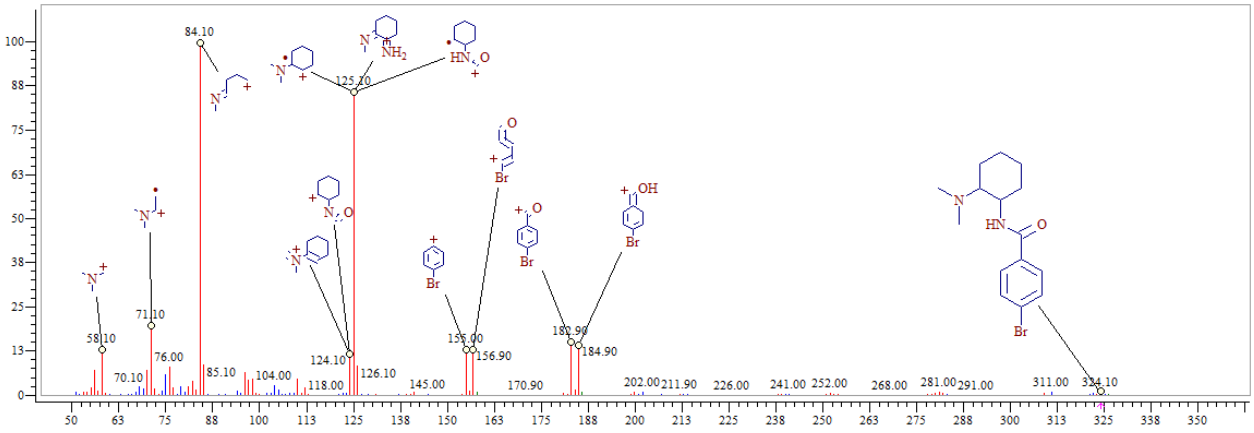
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8.99 BP(1): 84; BP(2): 125,BP(3) :71,
HPLC-TOF	+	Exact mass (theoretical): 324.0837; measured value Δppm:-0.68; formula:C15H21BrN2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

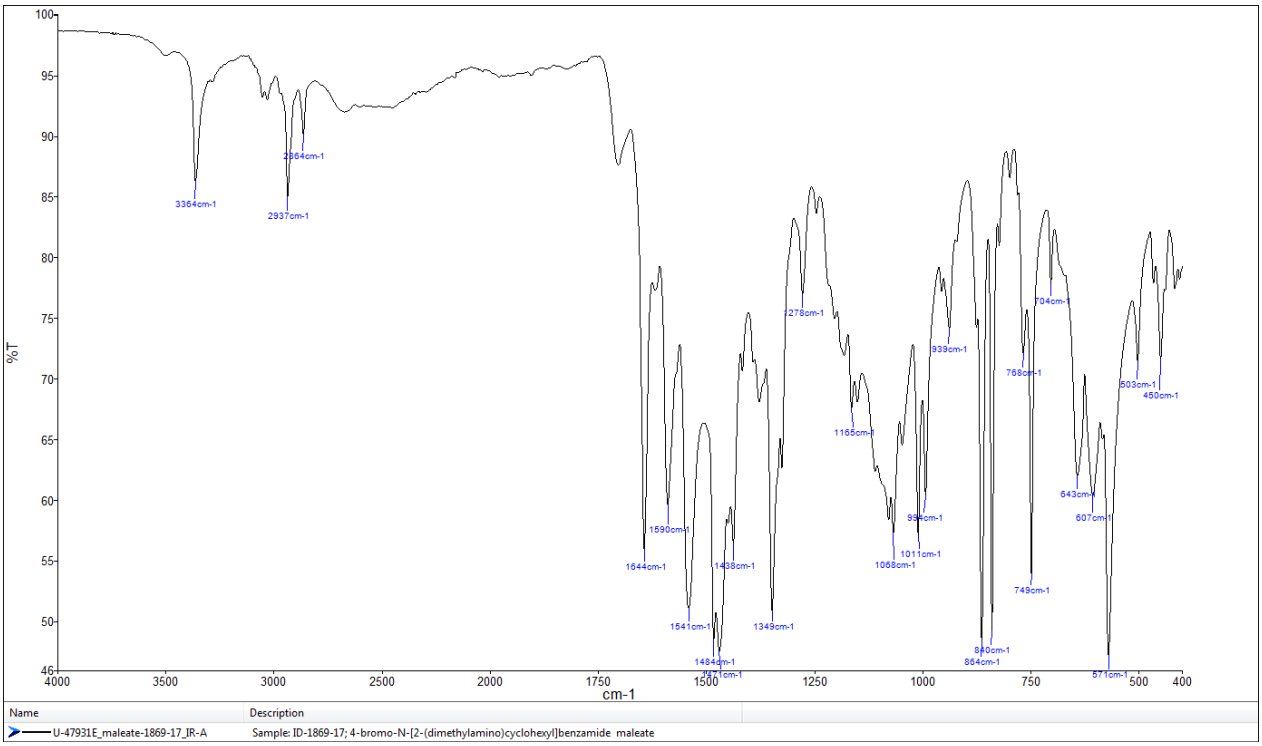
MS (EI)



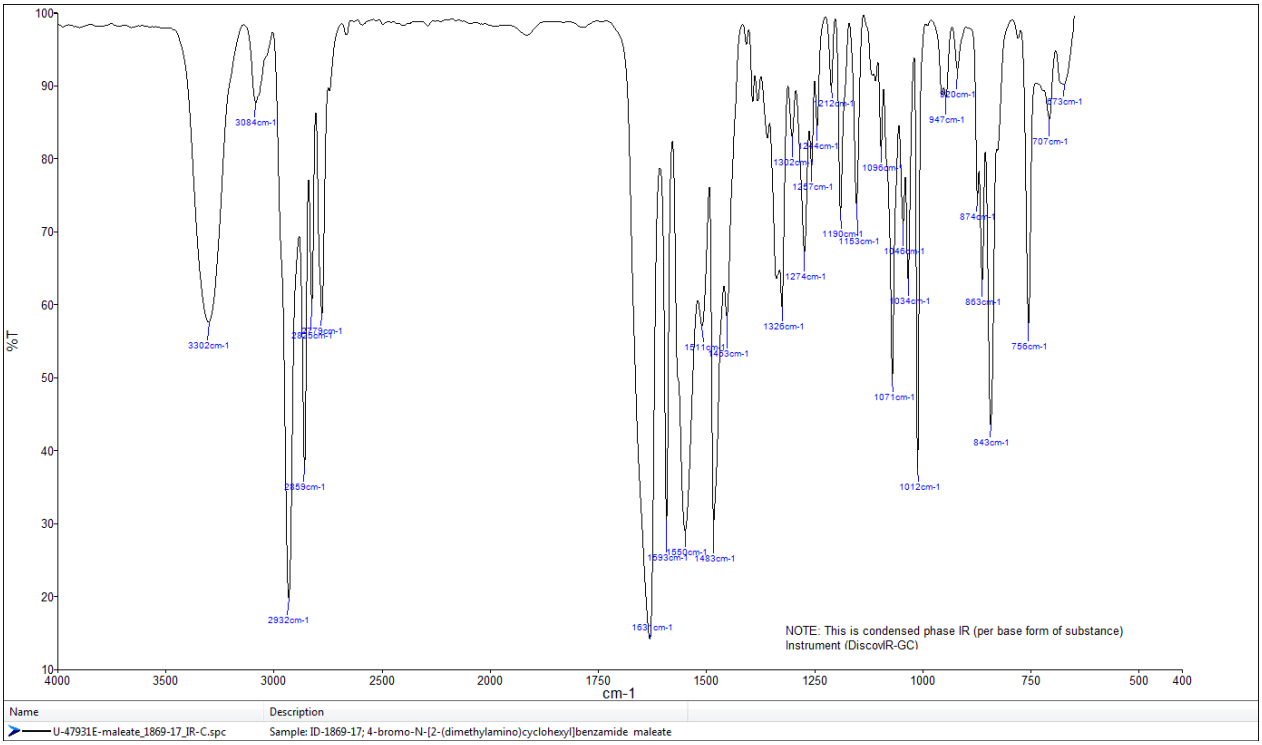
MS fragmentation (red labeled peaks explained – possible fragments)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

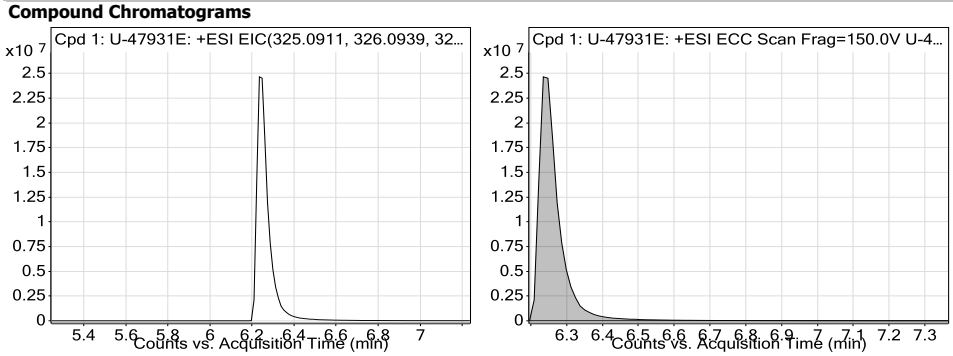
Data File	U-47931E_1869-17.d	**Sample Name**	1869-17
Sample Type	Sample	**Position**	P2-A8
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-19_07_2017-XDB-C18-ESI-final.m	**Acquired Time**	10/20/2017 10:07:53 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	MeOH		

Compound Table

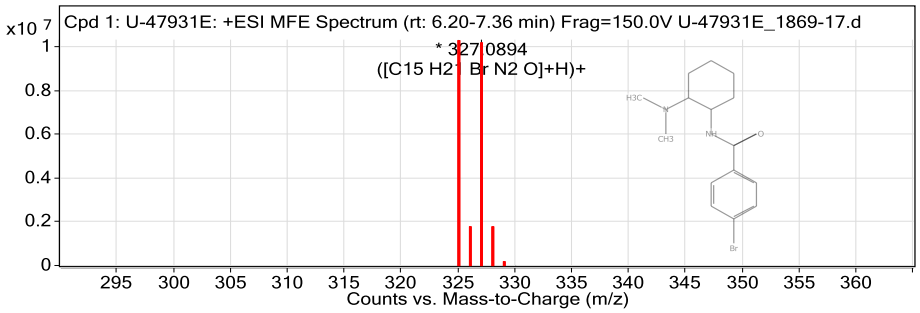
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: U-47931E	U-47931E	C15 H21 Br N2 O	6.25	324.084

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
U-47931E	325.0911	6.25	324.084	6.25	C15 H21 Br N2 O	324.0837	-0.68

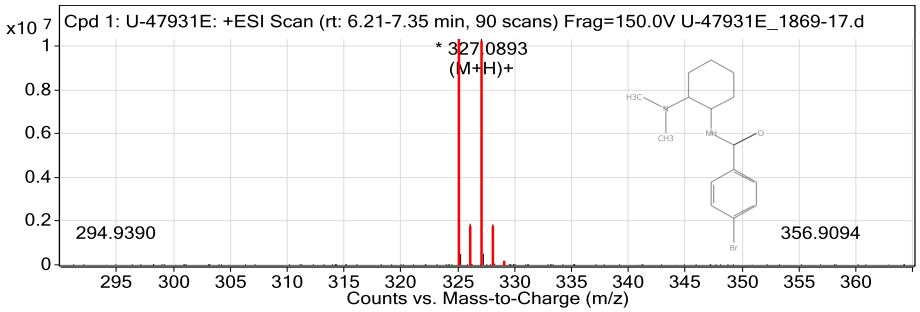
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

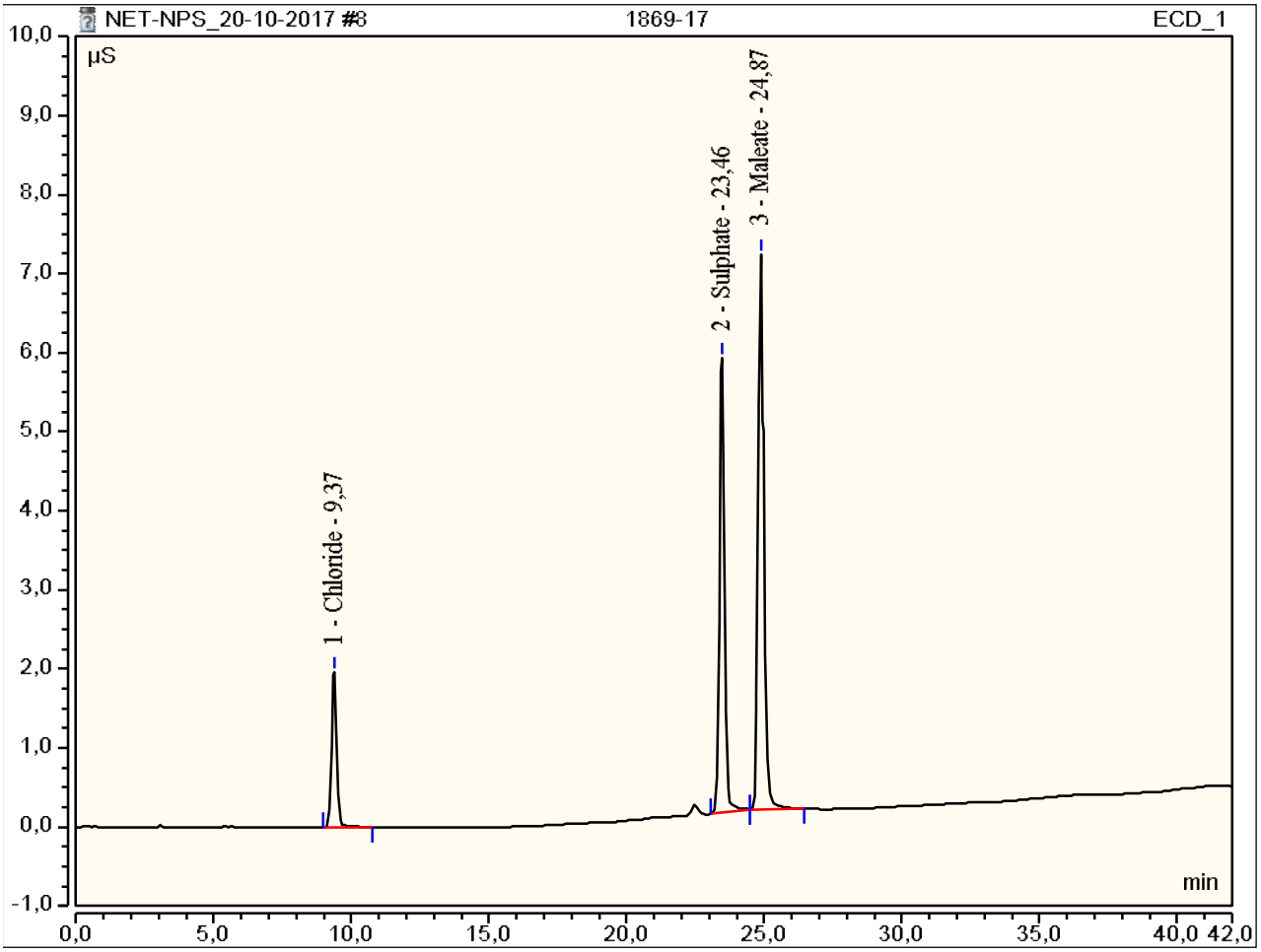
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
325.0911	1	10284081.58	C15 H21 Br N2 O	(M+H)+
326.0944	1	1697163.9	C15 H21 Br N2 O	(M+H)+
327.0894	1	10337141	C15 H21 Br N2 O	(M+H)+
328.0924	1	1670510.94	C15 H21 Br N2 O	(M+H)+
329.0954	1	138504.23	C15 H21 Br N2 O	(M+H)+
330.0984	1	8607.75	C15 H21 Br N2 O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**1869-17**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**20-okt-2017 / 13:53**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,37	**Chloride**	BMB	0,42	1,96	n.a.
2,00	23,46	**Sulphate**	BMb	1,20	5,75	n.a.
3,00	24,87	**Maleate**	bMB	1,73	7,03	n.a.
		TOTAL:		3,36	14,74	0,00



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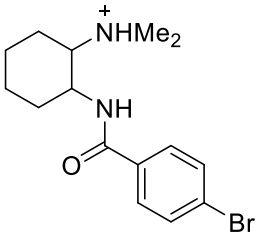
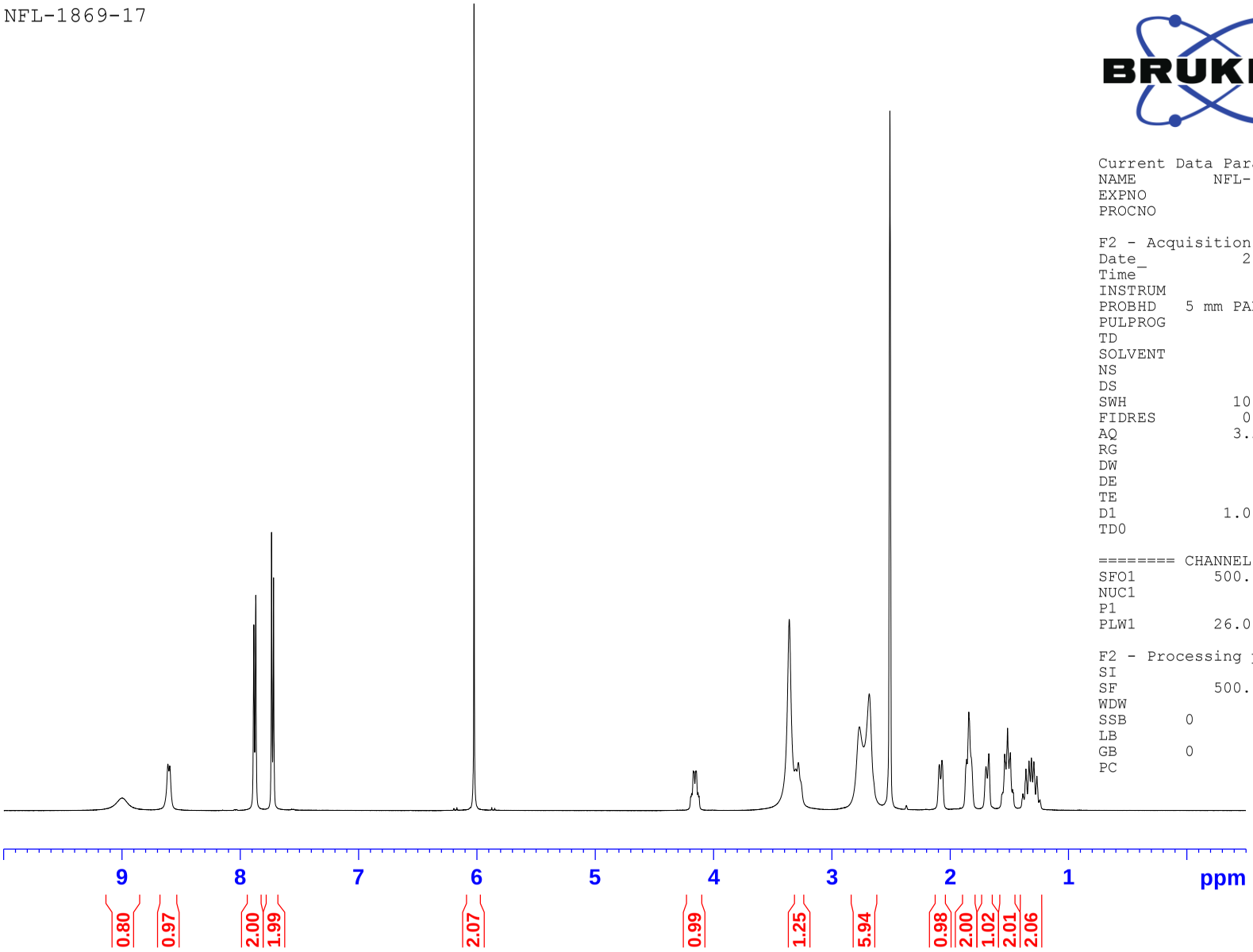
University
of Ljubljana

Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**1869-17**
Received date:	October 23, 2017
Our notebook code:	NFL-1869-17
NMR sample preparation:	15 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, chemical formula, exact mass, molecular weight:	 Chemical Formula: $C_{15}H_{22}BrN_2O^+$ Exact Mass: 325,0910 Molecular Weight: 326,2575
Chemical name:	2-(4-bromobenzamido)-*N*,*N*-dimethylcyclohexan-1-aminium ion
Comments:	- Structure elucidation based on 1D and 2D NMR spectra. - Compound is pure by NMR. - Counterion is maleate (recognized from 1D and 2D NMR spectra).
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Authors:	Marko Krivec, Martin Gazvoda, Janez Košmrlj
Date of report:	November 14, 2017

NFL-1869-17



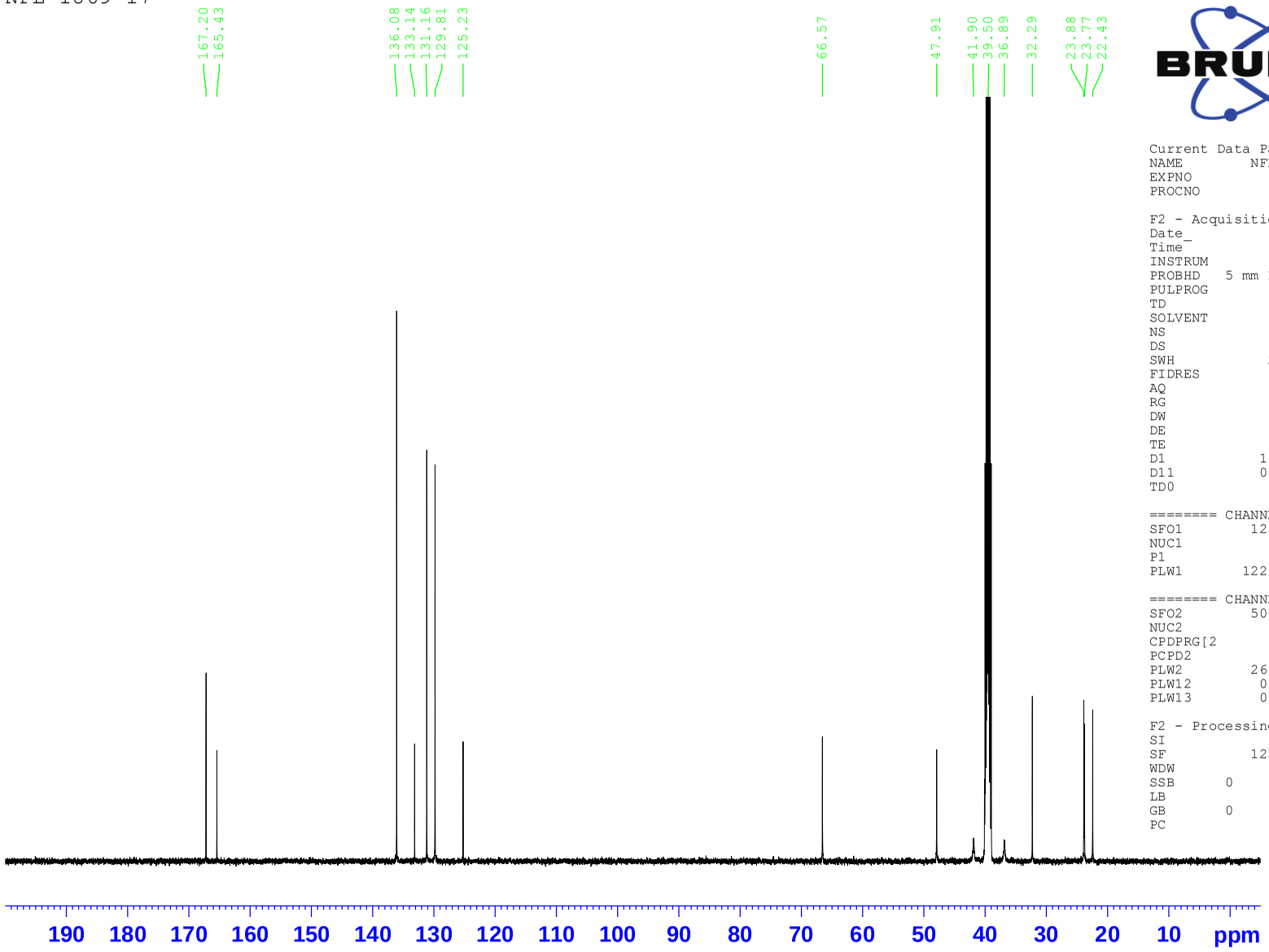
Current Data Parameters
NAME NFL-1869-17
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171027
Time 15.49
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 90.5
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
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-1869-17



BRUKER

```
Current Data Parameters
NAME         NFL-1869-17
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171027
Time              18.29
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          125.7577885 MHz
WDW                EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```