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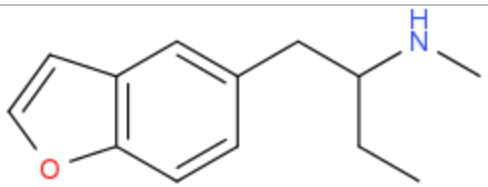
ANALYTICAL REPORT

5-MBPB (C13H17NO)

1-(benzofuran-5-yl)-N-methylbutan-2-amine

Remark – other NPS detected: none

Sample ID:	1260-15
Sample description:	powder - white-yellowish
Sample type:	test purchase (RESPONSE -purchasing)
Comments[1]:	
Date of entry:	9/2/2015

Substance identified-structure[2] (base form)	
Systematic name	1-(benzofuran-5-yl)-N-methylbutan-2-amine
Other names	
Formula (per base form)	C13H17NO
M_w (g/mol)	203,28
Salt form	HCl
StdInChIKey	CTEZPBCLIKEASW-UHFFFAOYSA-N
Compound Class	Arylalkylamines
Other NPS detected	none
Add.info (purity..)	pure by Gc, HPLC

[1] 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.

[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 (RT=9.53 min). Injection volume 1 ml and split mode (1:50) . Injector temperature: 280 ^{0}C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 293 ^{0}C at a rate of 18 ^{0}C/min, hold for 6.1 min, than heating at 50 ^{0}C/min up to 325 ^{0}C and finally 2.8 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 (40) to 550 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 4 cm^{-1}

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 (40) to 550 amu.
IR (condesed phase): IR scan range 4000 to 700, 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

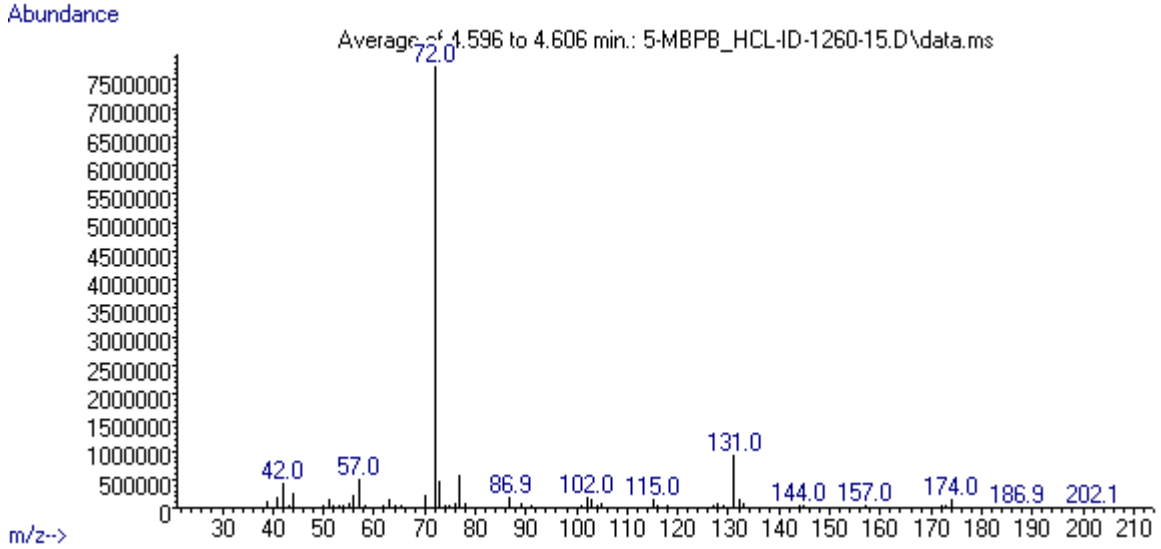
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
other	not tested

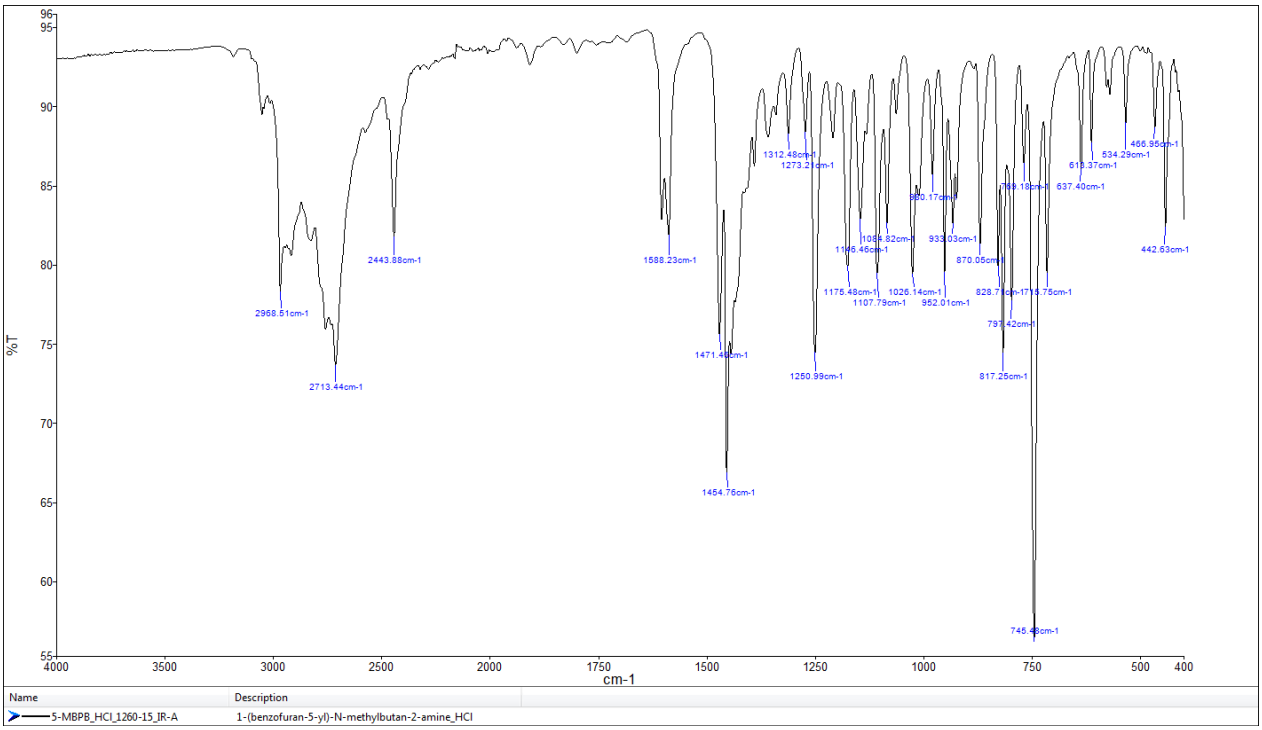
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,61 BP(1): 72; BP(2): 131,BP(3) :77,
HPLC-TOF	+	Exact mass (theoretical): 203,131; measured value Δppm:-0,22; formula:C13H17NO
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
FTIR-ATR	+	
NMR	+	
validation		
other		

ANALYTICAL RESULTS

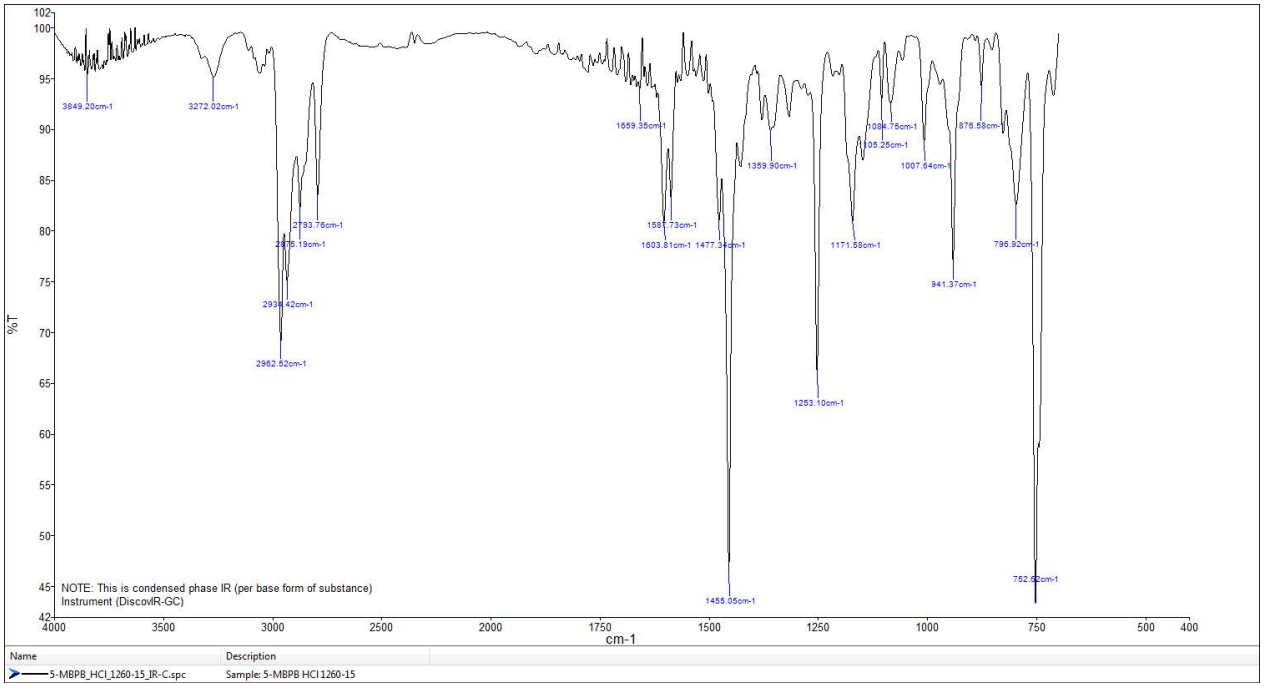
MS (EI)



FTIR-ATR



IR (condensed phase)



Target Compound Screening Report

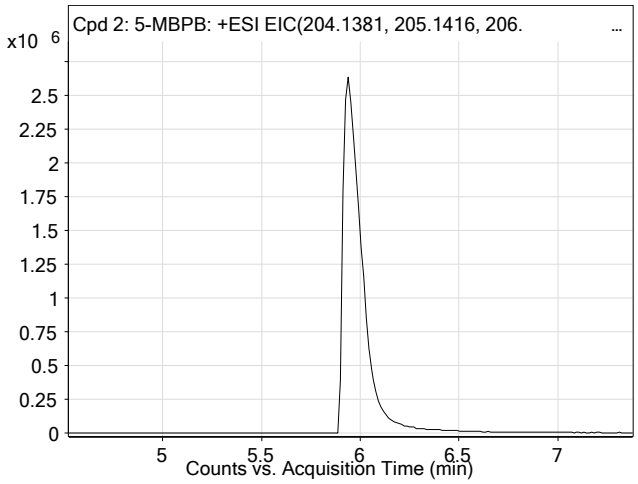
Data File	5-MBPB_1260-15_TOF.d	**Sample Name**	1260-15
Sample Type	Sample	**Position**	P1-B2
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	9/2/2015 12:57:59 PM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

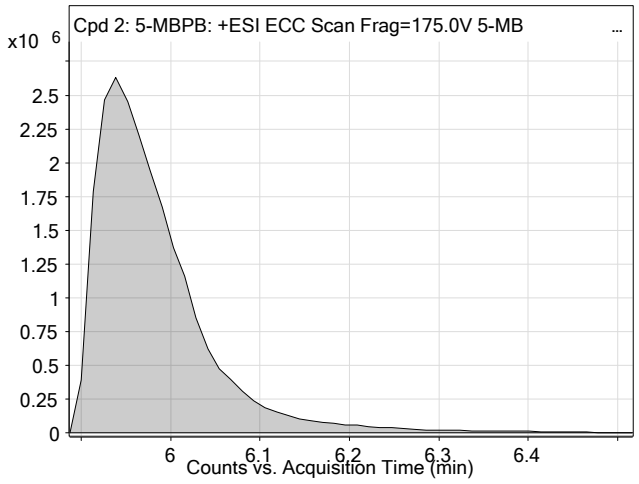
Compound Table

Label	Tgt Name	Obs. RT	Obs. Mass
Cpd 2: 5-MBPB	5-MBPB	5.951	203.1311

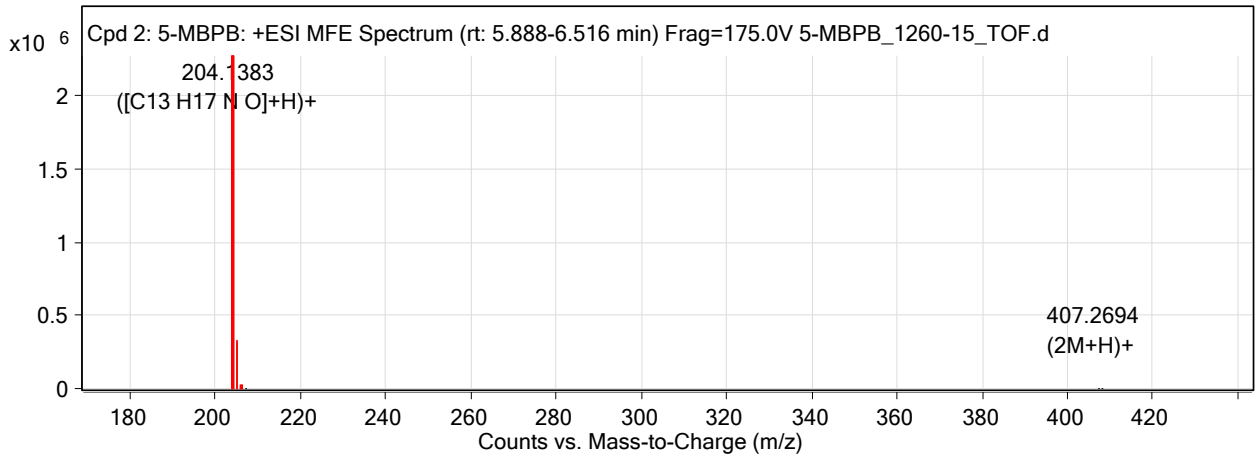
Name	Obs. *m/z*	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
5-MBPB	204.1383	5.951	203.1311	C13 H17 N O	203.131	-0.22	Find by Molecular Feature

Compound Chromatograms





MFE MS Zoomed Spectrum

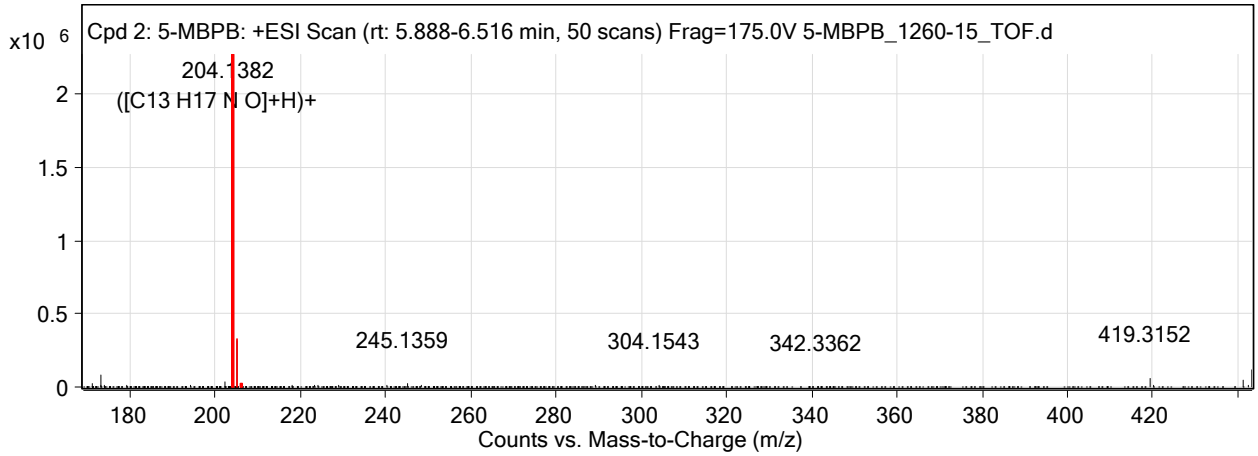


MS Spectrum Peak List

Obs. *m/z*	Charge	Abund	Formula	Ion/Isotope
204.1383	1	2275197	C13 H17 N O	(M+H)+
205.1418	1	312476.23	C13 H17 N O	(M+H)+
206.1444	1	24487.77	C13 H17 N O	(M+H)+
207.1477	1	1556.13	C13 H17 N O	(M+H)+
407.2694	1	3811.8		(2M+H)+
408.2727	1	1299.47		(2M+H)+

Agilent Technologies

MS Zoomed Spectrum

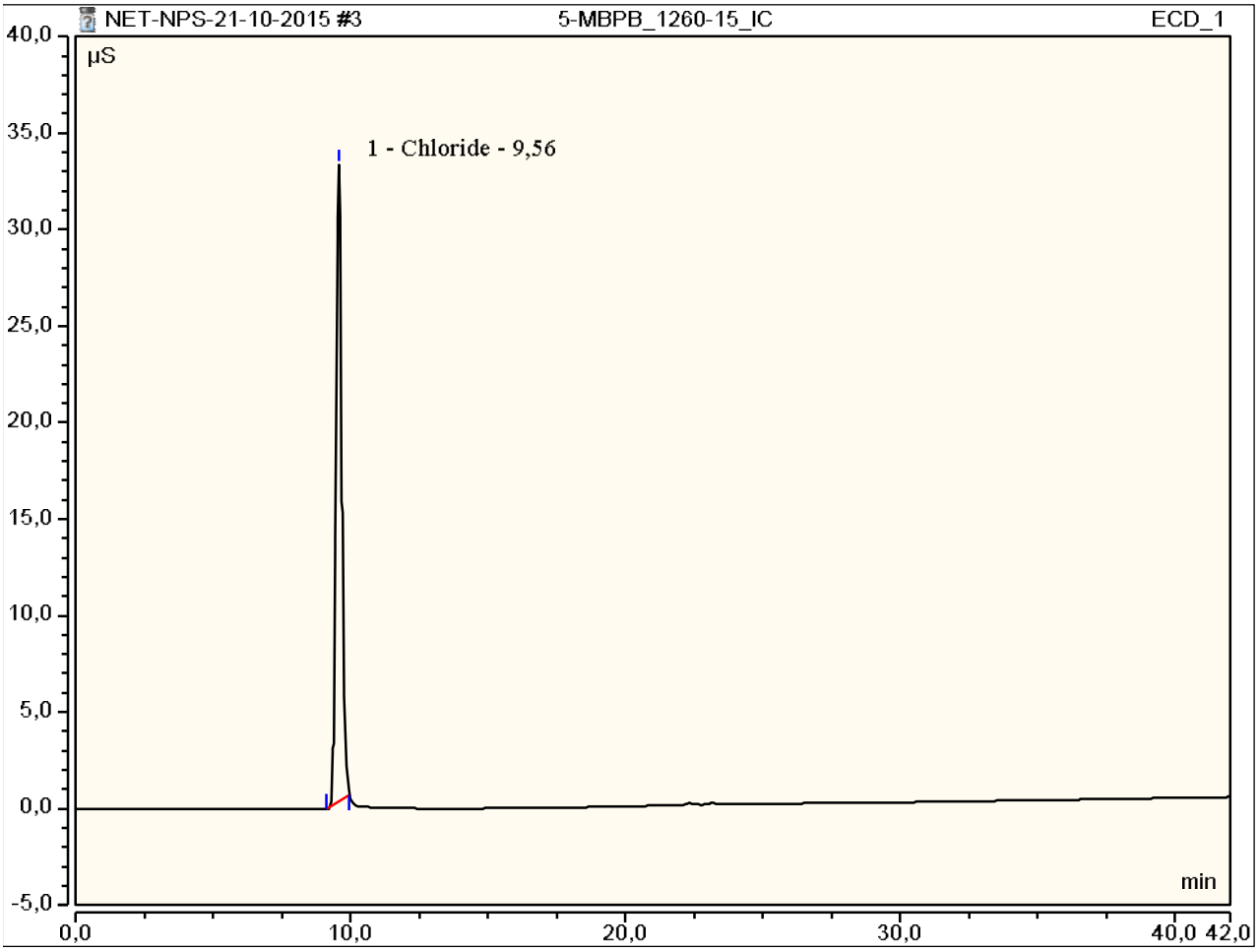


--- End Of Report ---

Peak Integration Report

Sample Name:	**5-MBPB_1260-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**21-okt-2015 / 15:48**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,56	**Chloride**	BMB	7,96	32,95	n.a.
		TOTAL:		7,96	32,95	0,00



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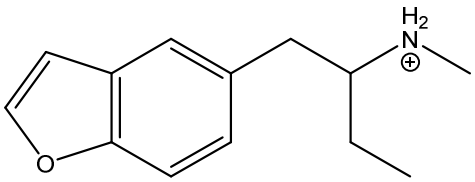
REPORT

Sample ID:	**1260-15**
Our notebook code:	P-1260-15
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.
Proposed structure:	
Chemical name:	1-(benzofuran-5-yl)-N-methylbutan-2-aminium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	November 3, 2015

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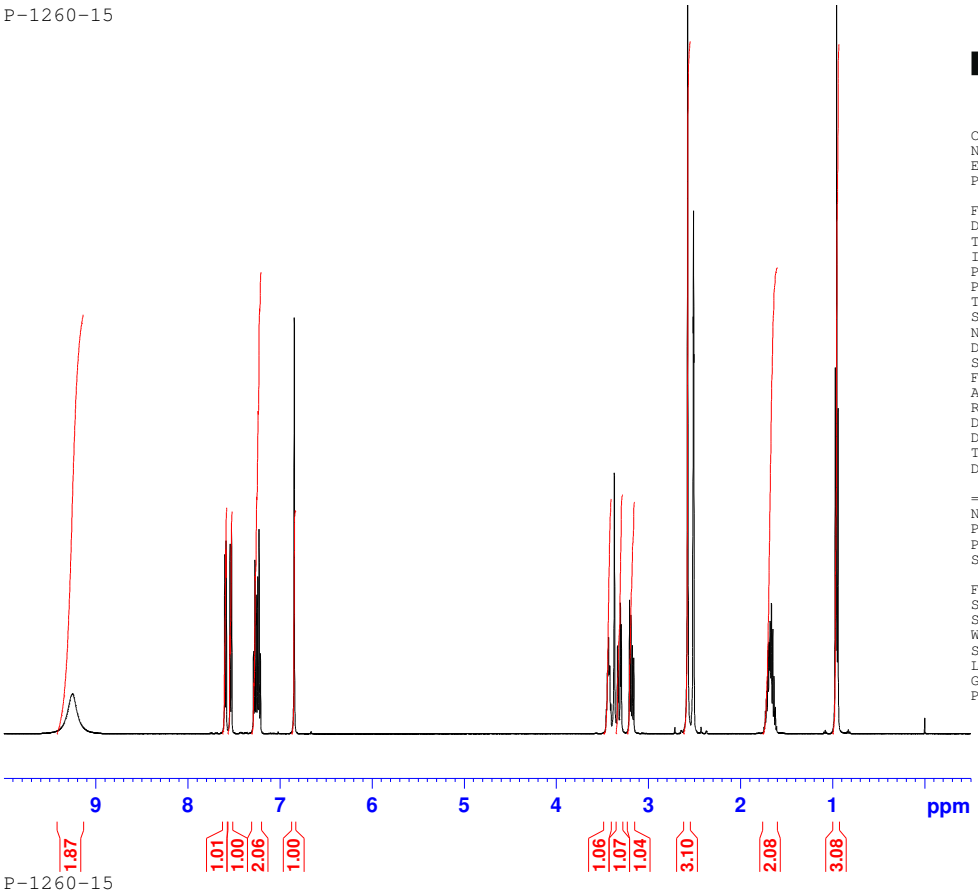
P-1260-15



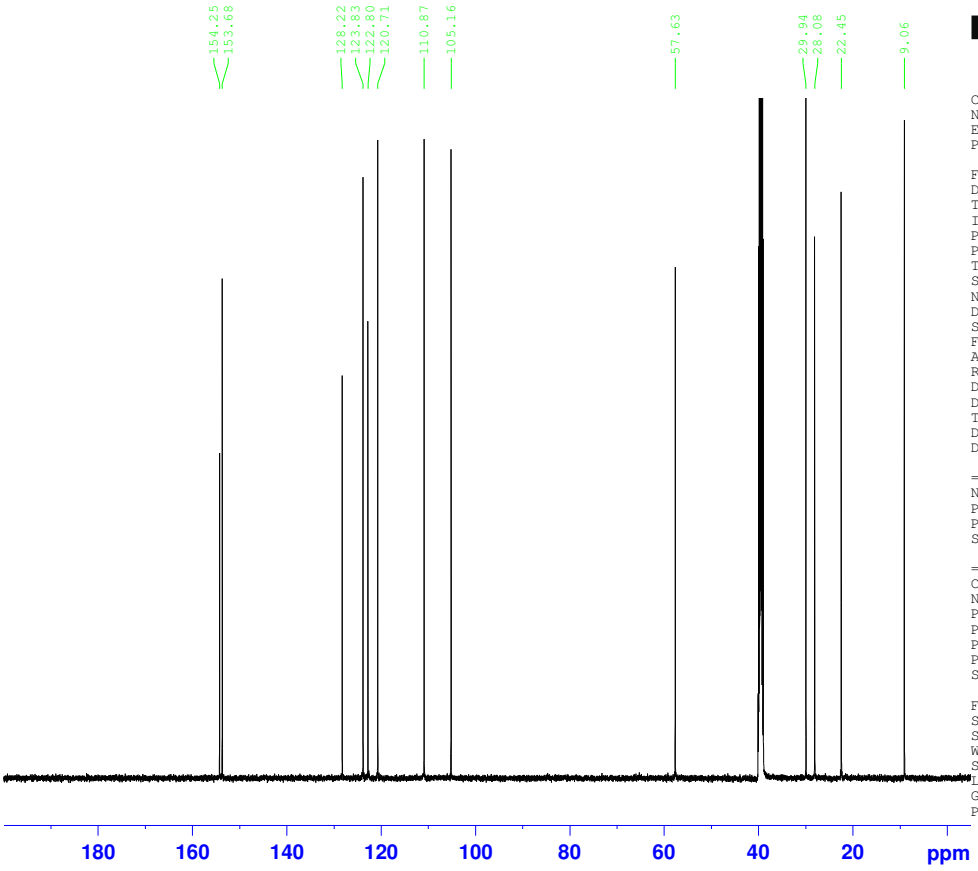
Current Data Parameters
NAME P-1260-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151031
Time 23.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 65536
SF 500.1300006 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1260-15



Current Data Parameters
NAME P-1260-15
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151101
Time 3.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 5120
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 122.00000000 W
SFO1 125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.32179001 W
PLW13 0.20595001 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578519 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40