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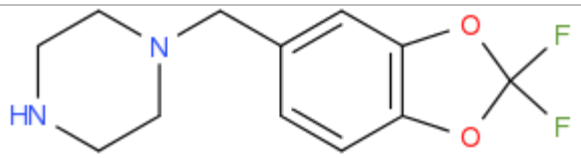
ANALYTICAL REPORT[1]

DB-MDBP (C12H14F2N2O2)

1-((2,2-difluorobenzo[D][1,3]dioxol-5-yl)methyl)piperazine

Remark – other NPS detected: none

Sample ID:	1252-15
Sample description:	powder - white-off
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	8/18/2015
Date of entry (M/D/Y) into NFL database:	8/19/2015
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-((2,2-difluorobenzo[D][1,3]dioxol-5-yl)methyl)piperazine
Other names	
Formula (per base form)	C12H14F2N2O2
M_w (g/mol)	256.25
Salt form	HCl
StdInChIKey	MPZINTHEMFDGTH-UHFFFAOYSA-N
Compound Class	Piperazine derivates
Other NPS detected	none
Add.info (purity..)	pure by GC, HPLC-TOF, compound is not pure by NMR (see analytical report) , pure by GC, HPLC-TOF, compound is not pure by NMR (see analytical report)

[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)
11/04/2018	Compound class corrected

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 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 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

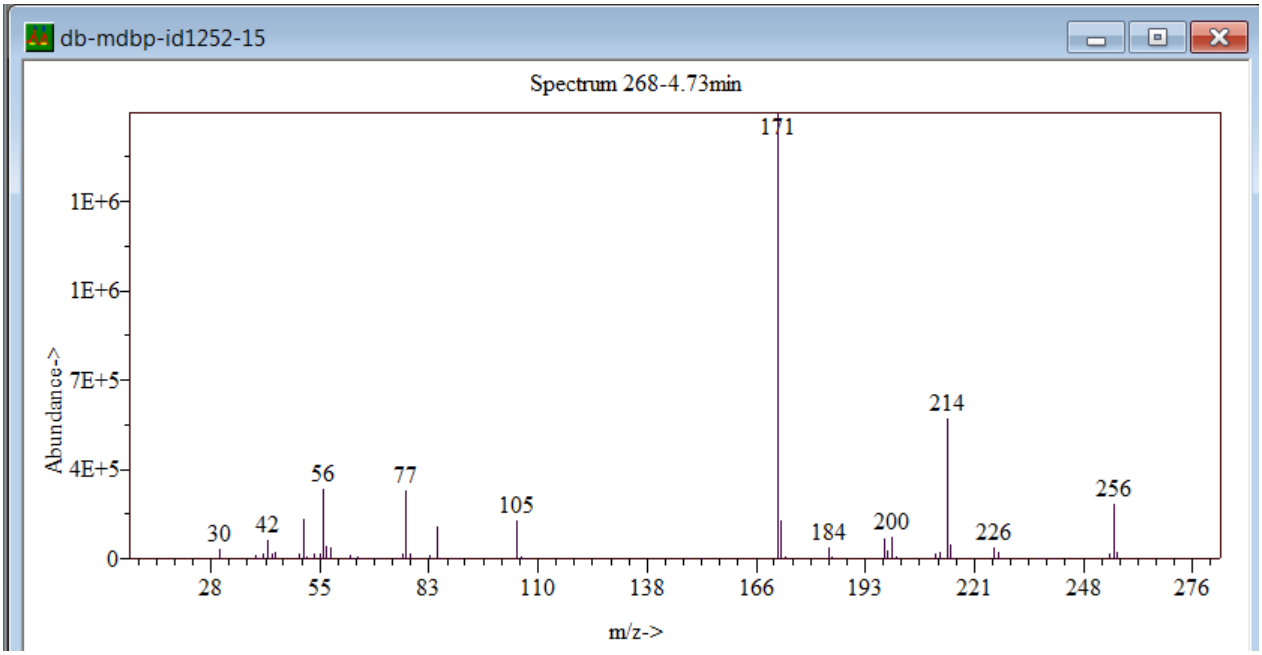
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	not tested

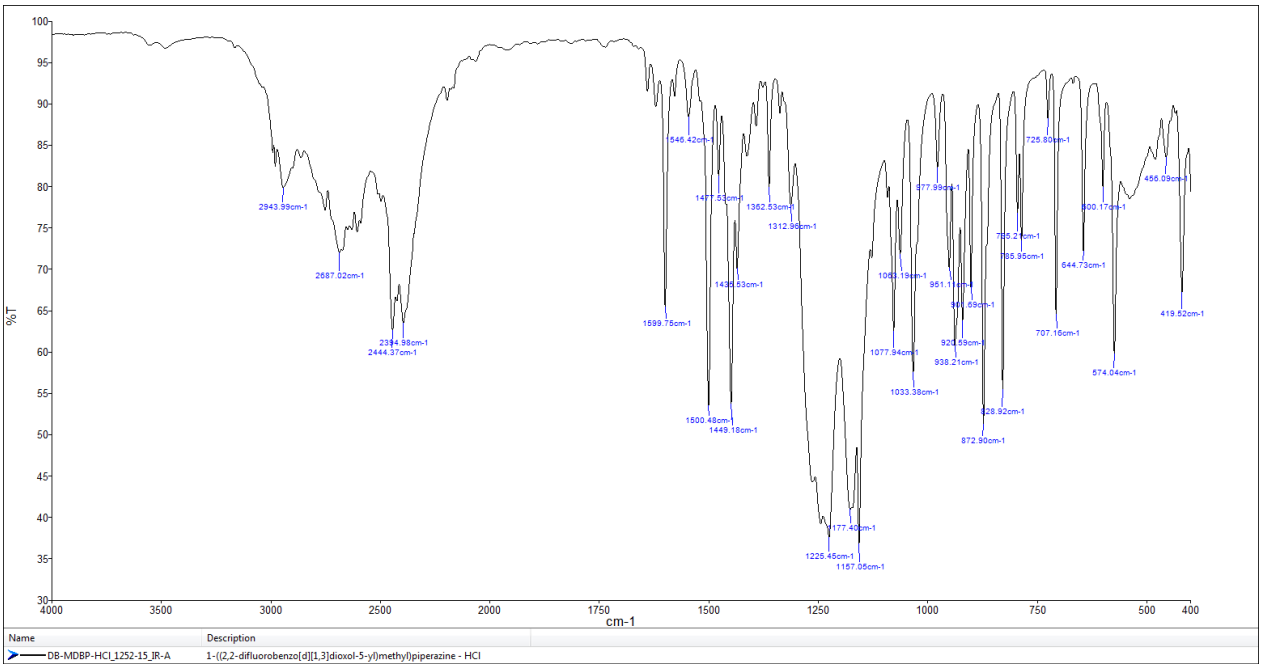
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4.73 BP(1): 171; BP(2): 214,BP(3) :56,
HPLC-TOF	+	Exact mass (theoretical): 256.1023; measured value Δppm:-0.13; formula:C12H14F2N2O2
FTIR-ATR	+	
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

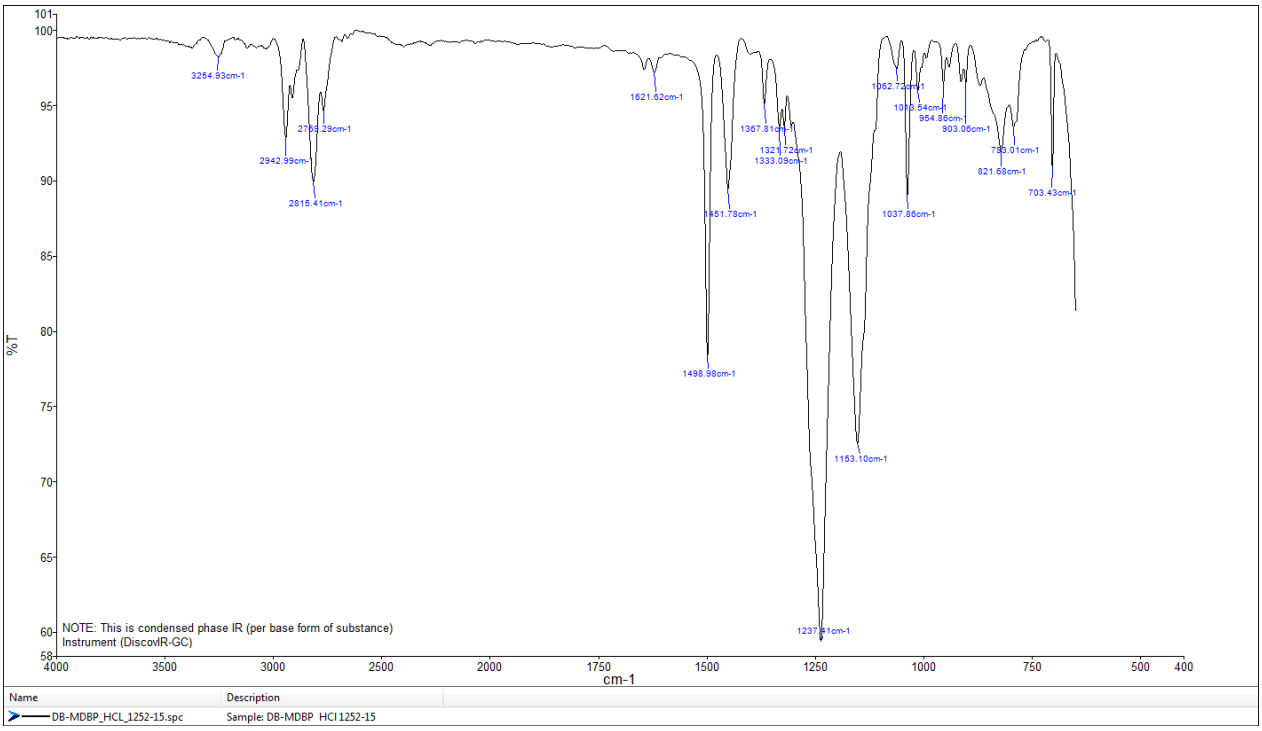
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase)



Target Compound Screening Report

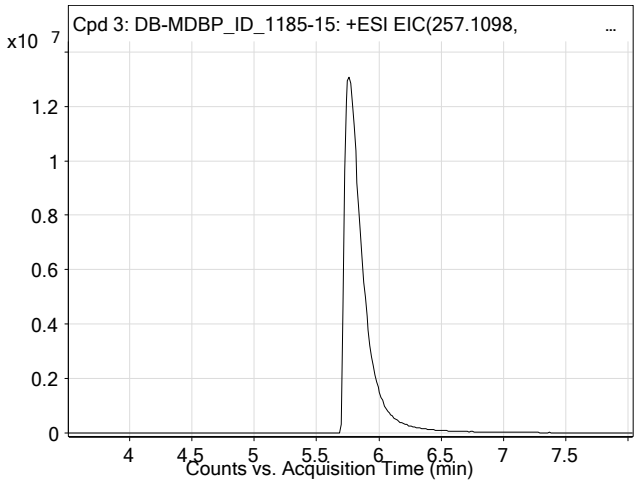
Data File	DB-MDBP_1252-15_TOF.d	**Sample Name**	DB-MDBP
Sample Type	Sample	**Position**	P2-F9
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	8/19/2015 2:39:50 PM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

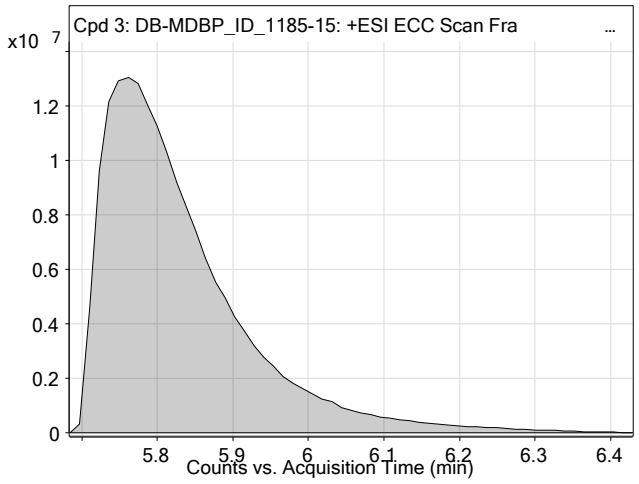
Compound Table

Label	Tgt Name	Obs. RT	Obs. Mass
Cpd 3: DB-MDBP_ID_1185-15	DB-MDBP_ID_1185-15	5.777	256.1024

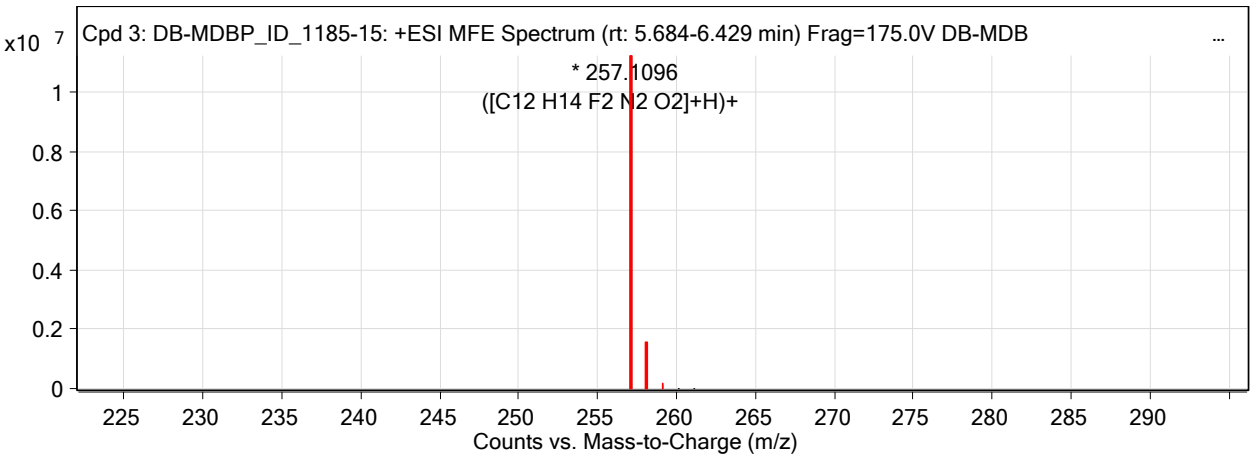
Name	Obs. *m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
DB-MDBP_ID_1185-15	257.1096	5.777	256.1024	5.884	C12 H14 F2 N2 O2	256.1023	-0.13	Find by Molecular Feature

Compound Chromatograms





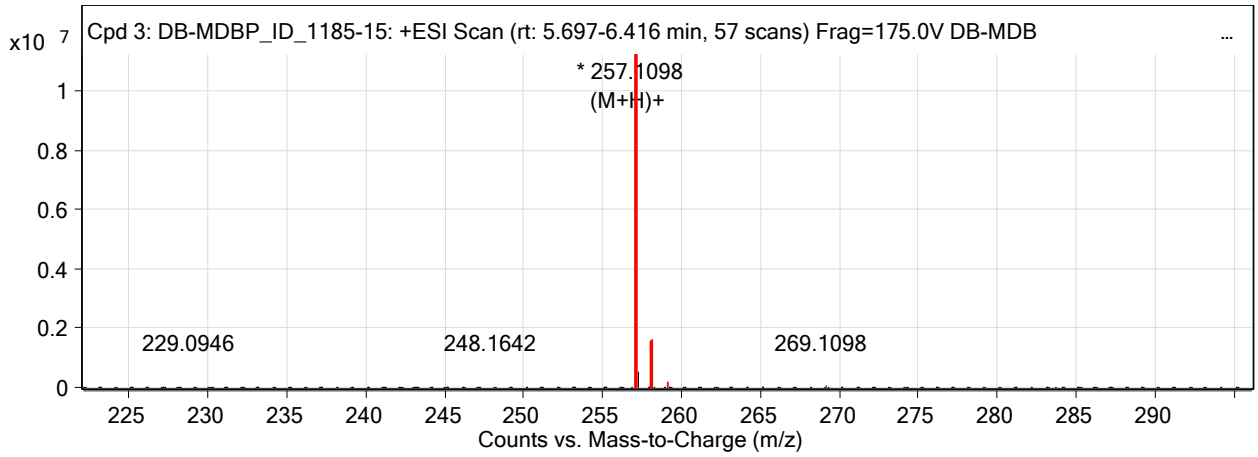
MFE MS Zoomed Spectrum



MS Spectrum Peak List

Obs. *m/z*	Charge	Abund	Formula	Ion/Isotope
257.1096	1	11205948	C12 H14 F2 N2 O2	(M+H)+
258.113	1	1505093.68	C12 H14 F2 N2 O2	(M+H)+
259.1155	1	130843.18	C12 H14 F2 N2 O2	(M+H)+
260.1178	1	9880.51	C12 H14 F2 N2 O2	(M+H)+
261.1243	1	179.47	C12 H14 F2 N2 O2	(M+H)+

MS Zoomed Spectrum

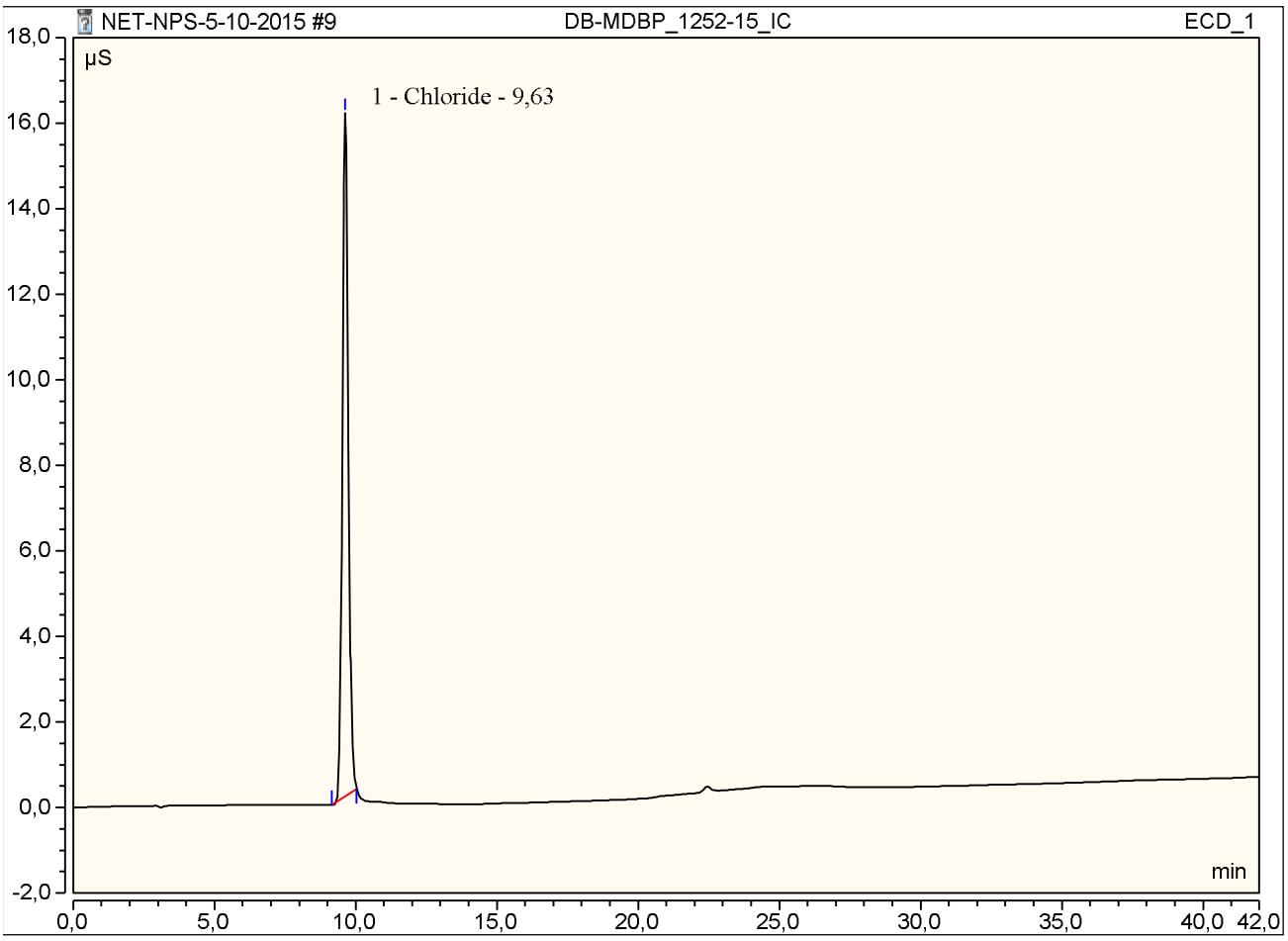


--- End Of Report ---

Peak Integration Report

Sample Name:	**DB-MDBP_1252-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**05-okt-2015 / 20:24**	*Run Time:*	**41,99**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,63	**Chloride**	BMB	3,83	15,96	n.a.
		TOTAL:		3,83	15,96	0,00



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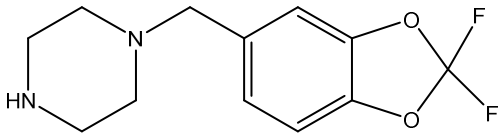
REPORT

Sample ID:	**1252-15**
Our notebook code:	P-1252-15
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-((2,2-difluorobenzo[d][1,3]dioxol-5-yl)methyl)piperazine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is not pure by NMR, it contains organic impurities (evident in ^{13}C NMR, signal at 191.1 and in ^{1}H NMR signals around 1.0 ppm) and also possibly some other impurities (inorganic cations?) that cause line broadening in ^{1}H NMR spectrum significantly diminishing its use for structural determination. - According to NMR spectra this sample is identical to the sample P-1185-15.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	November 19, 2015

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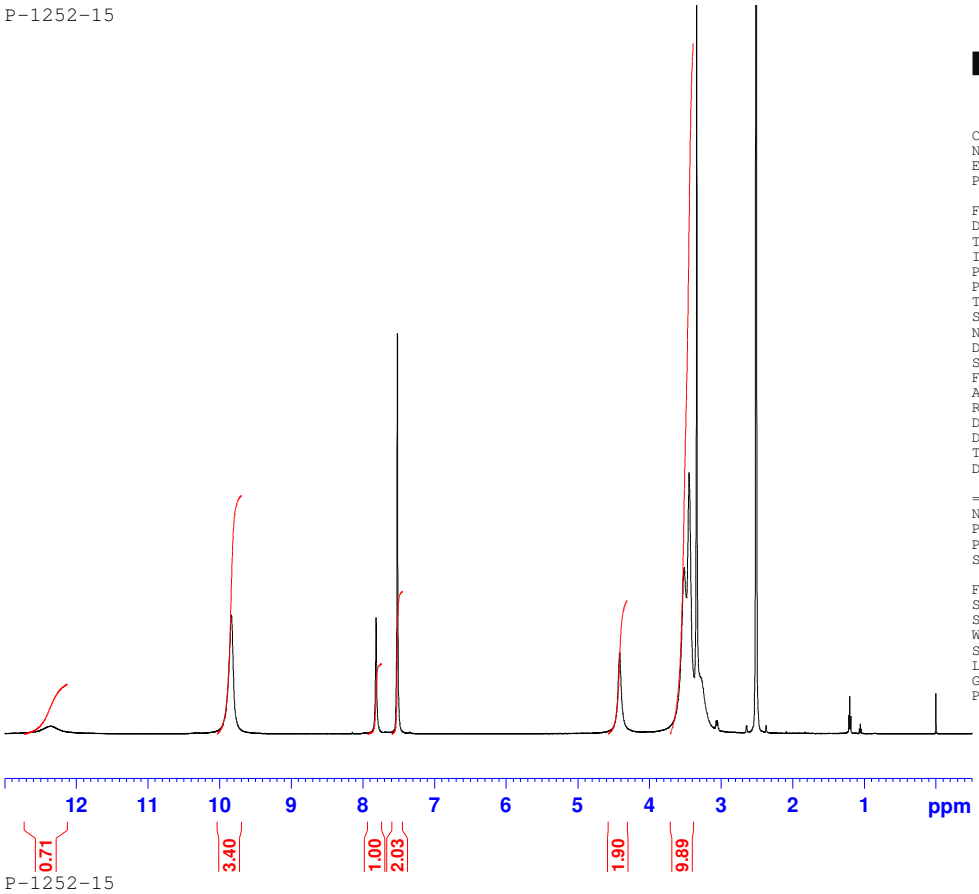
P-1252-15



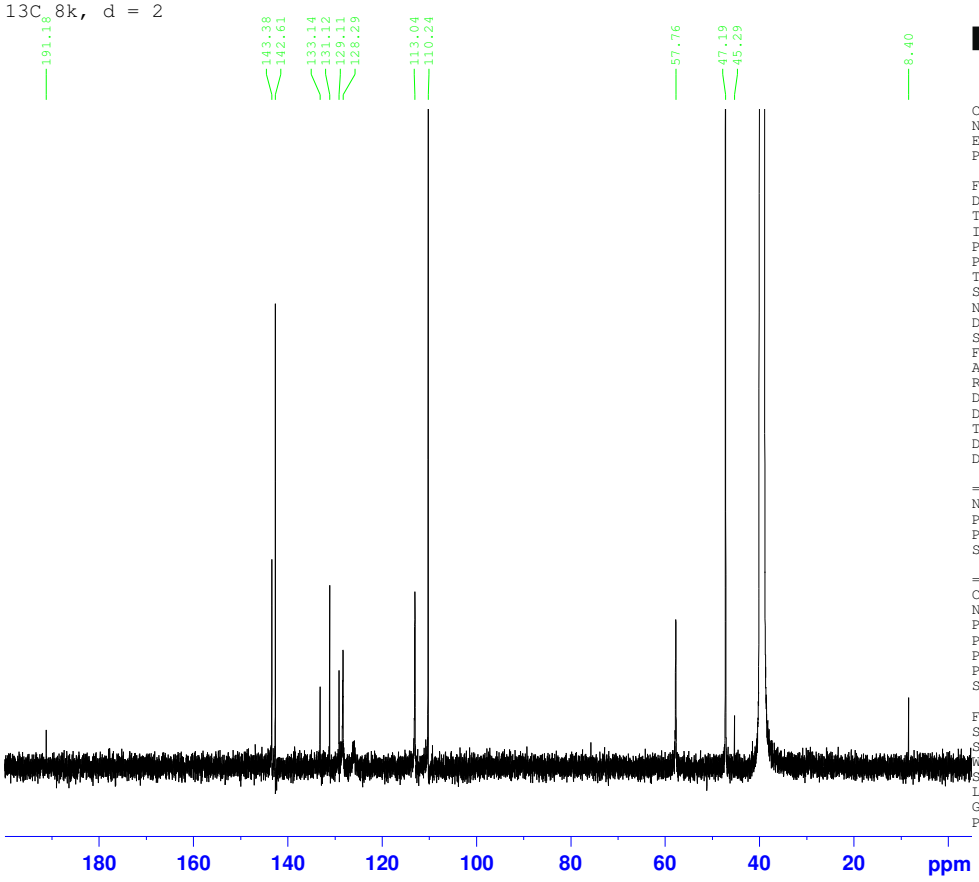

```
Current Data Parameters
NAME          P-1252-15
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20151021
Time               4.06
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                  101
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-1252-15
13C 8k, d = 2




```
Current Data Parameters
NAME          P-1252-15
EXPNO                21
PROCNO                1

F2 - Acquisition Parameters
Date_          20151031
Time              19.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 8192
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            2.00000000 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
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