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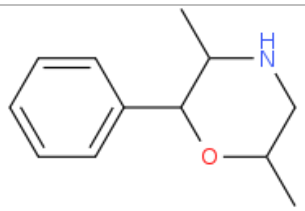
ANALYTICAL REPORT[1]

3,6-DMPM (C12H17NO)

3,6-dimethyl-2-phenylmorpholine

Remark – other NPS detected: none

Sample ID:	1412-16
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	1/6/2016
Date of entry (M/D/Y) into NFL database:	2/12/2016
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	3,6-dimethyl-2-phenylmorpholine
Other names	2-Phenyl-3,6-dimethylmorpholine
Formula (per base form)	C12H17NO
M_w (g/mol)	191,27
Salt form/anions detected	tartrate (and chloride ions)
StdInChIKey	FZEIVUHEODGHML-UHFFFAOYSA-N
Compound Class	Others
Other NPS detected	none
Add.info (purity..)	MS, TOF: two signals - diastereoisomers, NMR: 3 isomers

[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 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) 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 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

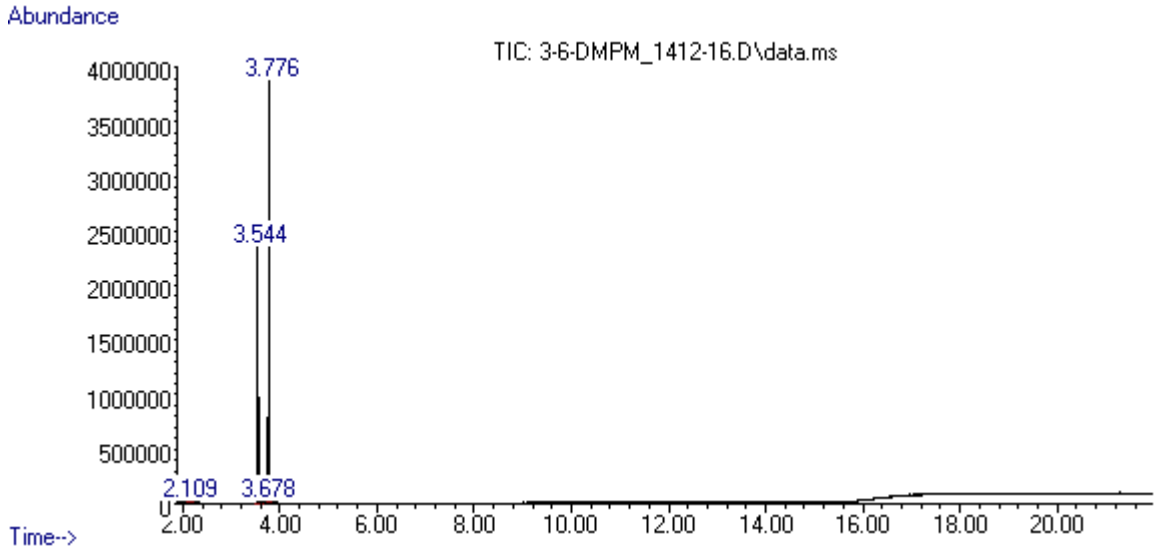
Supporting information

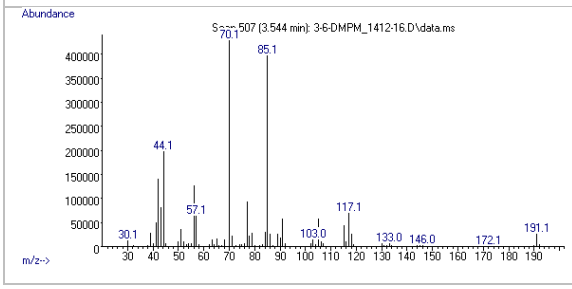
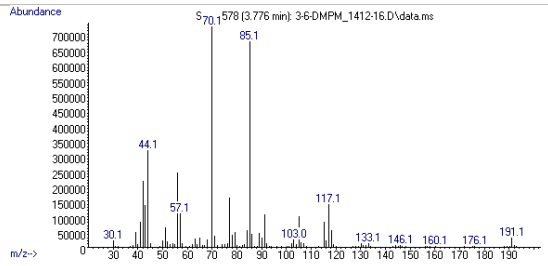
Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 3,78 (refers to the most intensive peak) BP(1): 70; BP(2): 85,BP(3) :44,
HPLC-TOF	+	Exact mass (theoretical): 366,505; measured value Δppm:-1,93; formula:C12H17NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

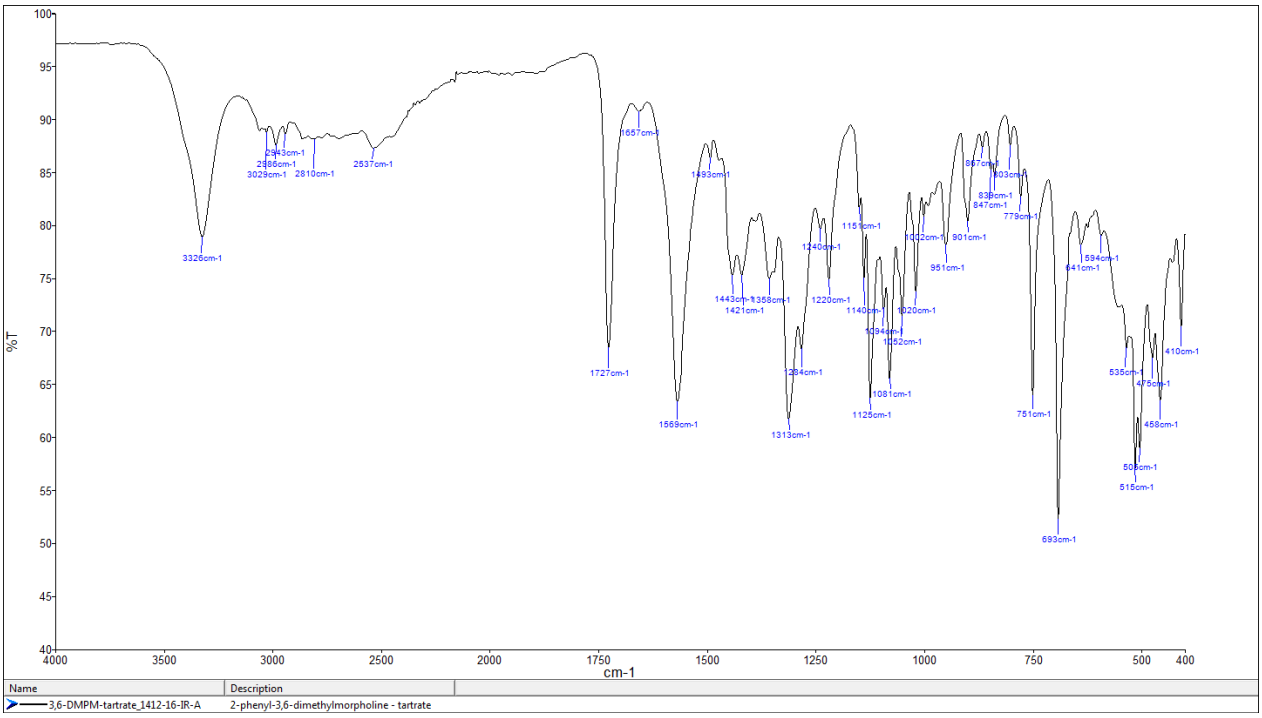
ANALYTICAL RESULTS

Chromatogram (

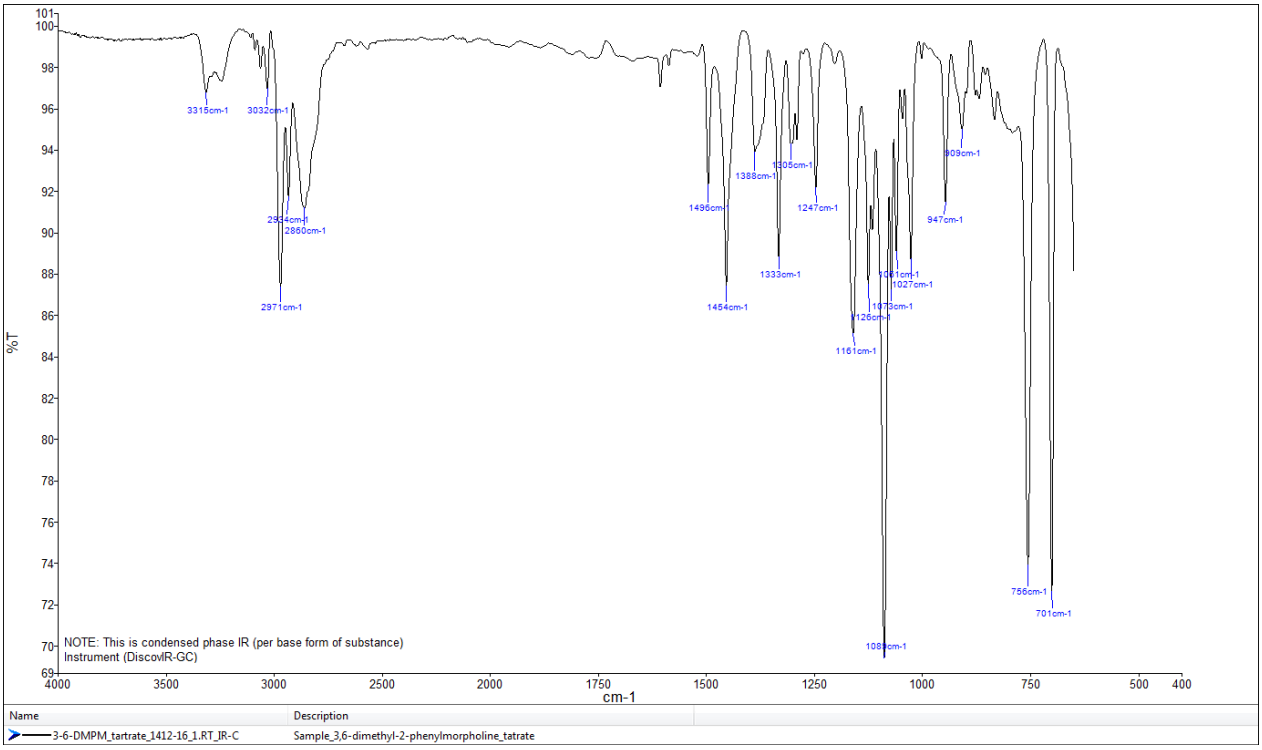


MS (EI): RT=5.54	MS(EI): RT=3.78
	

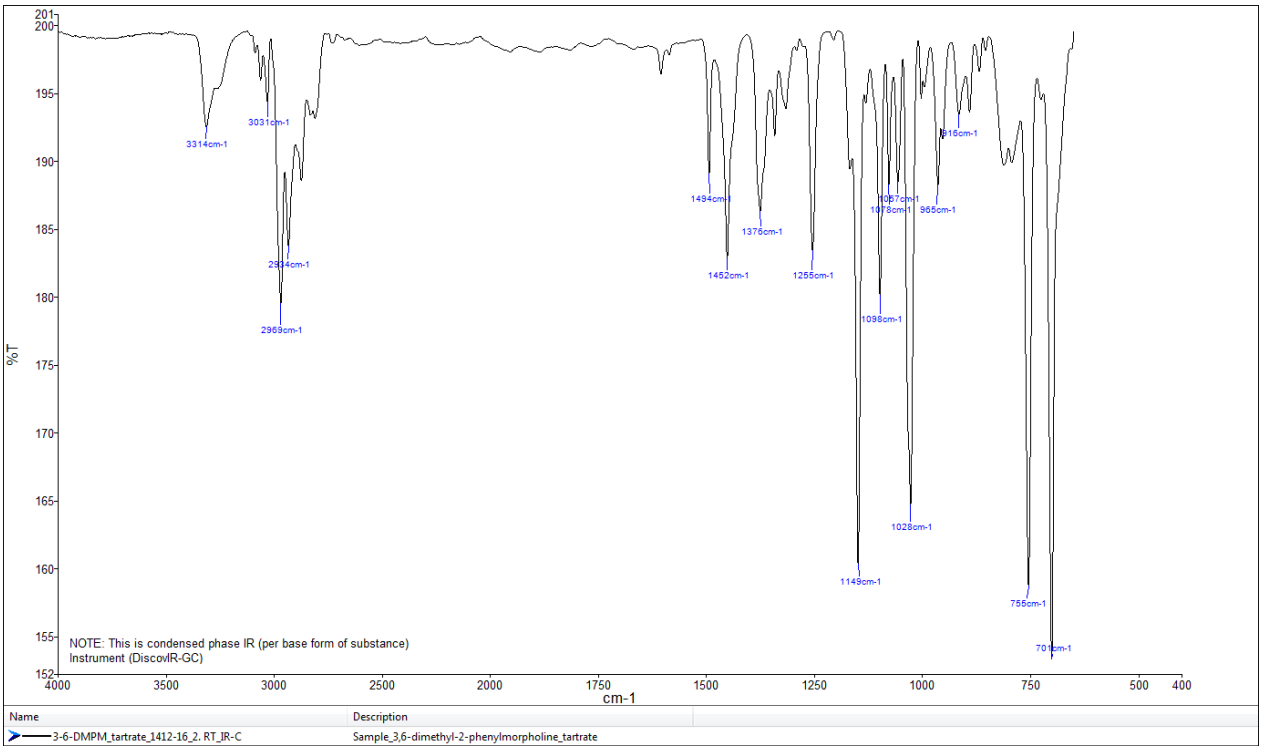
FTIR-ATR - direct measurement (sample as received, tartrate salt)



IR (condensed phase – after chromatographic separation at RT1)



IR-condensed phase (after chromatographic separation - at RT2)



TOF REPORT

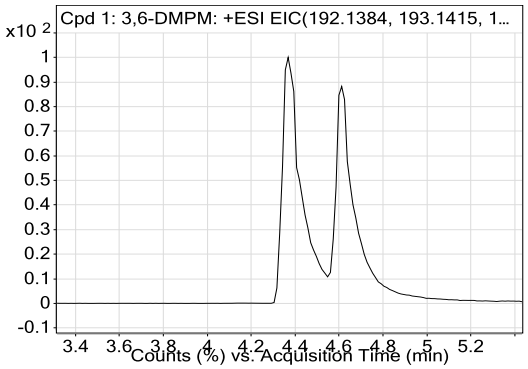
Data File	3-6-DMPM_1412-15_TOF.d	**Sample Name**	ID_1412-15
Sample Type	Sample	**Position**	P1-E1
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-1512015-XDB-C18-ESI-poz.m	**Acquired Time**	1/11/2016 1:14:56 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

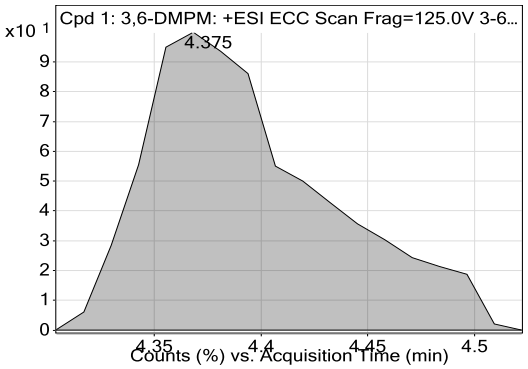
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: 3,6-DMPM	3,6-DMPM	C12 H17 N O	4.375	191.1314
Cpd 2: 3,6-DMPM	3,6-DMPM	C12 H17 N O	4.618	191.1317
Cpd 3: 3,6-DMPM	3,6-DMPM	C12 H17 N O	5.893	191.1314

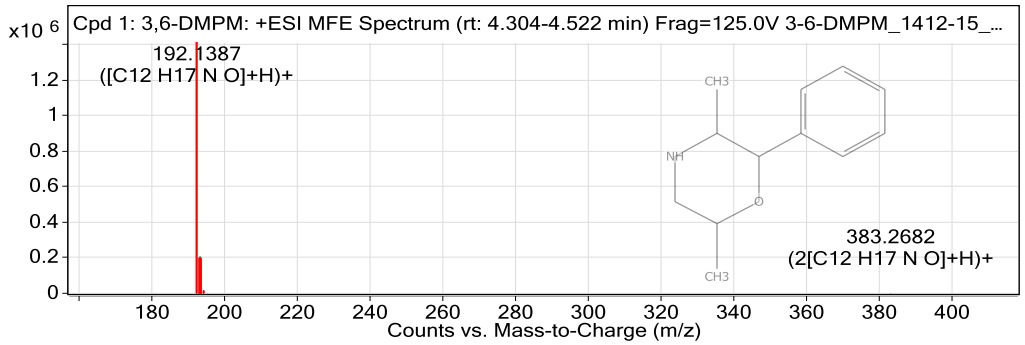
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)
3,6-DMPM	192.1387	4.375	191.1314	C12 H17 N O	191.131	-1.93

Compound Chromatograms

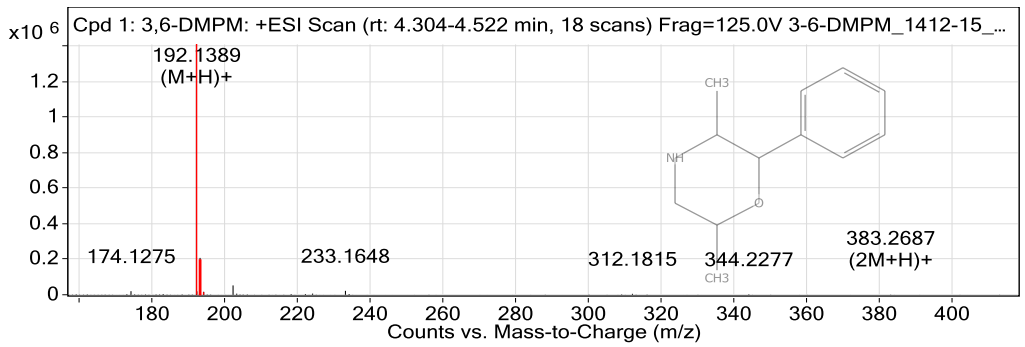




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



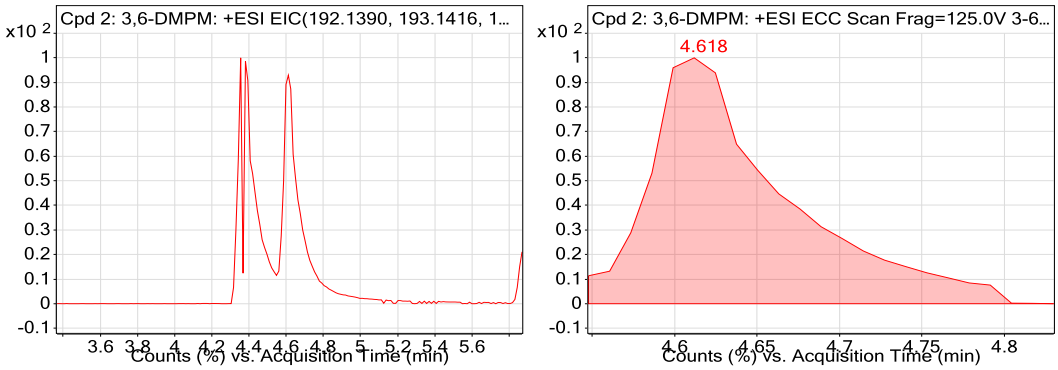
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
192.1387	1	1407233.75	C12 H17 N O	(M+H)+
193.142	1	207439.19	C12 H17 N O	(M+H)+
194.1439	1	16868.51	C12 H17 N O	(M+H)+
195.1388	1	1025.58	C12 H17 N O	(M+H)+
383.2682	1	1374.19	C12 H17 N O	(2M+H)+

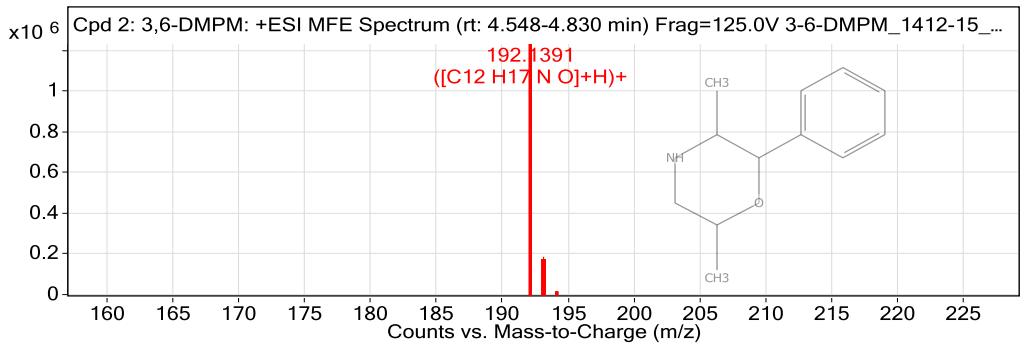
TOF REPORT

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)
3,6-DMPM	192.1391	4.618	191.1317	C12 H17 N O	191.131	-3.82

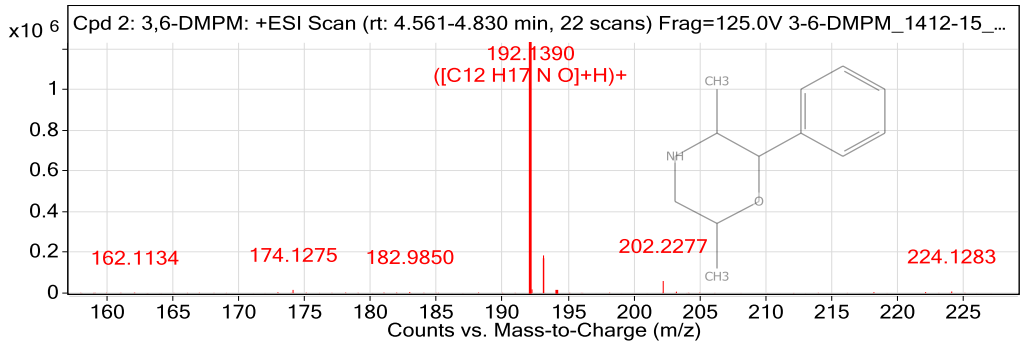
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



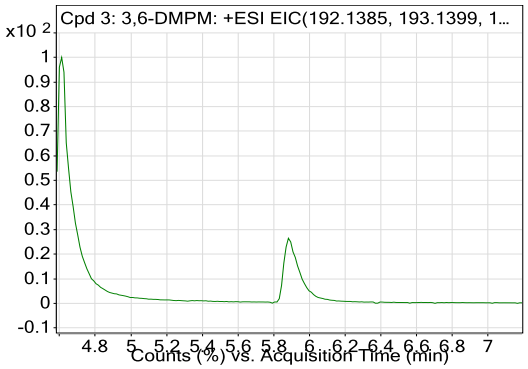
MS Spectrum Peak List

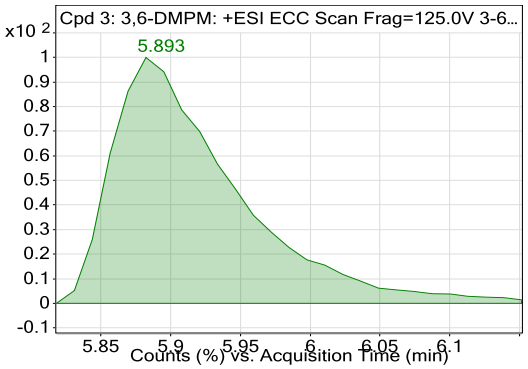
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
192.1391	1	1231525.13	C12 H17 N O	(M+H)+
193.142	1	186117.75	C12 H17 N O	(M+H)+
194.1437	1	15124.27	C12 H17 N O	(M+H)+

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)
3,6-DMPM	192.1388	5.893	191.1314	C12 H17 N O	191.131	-2.16

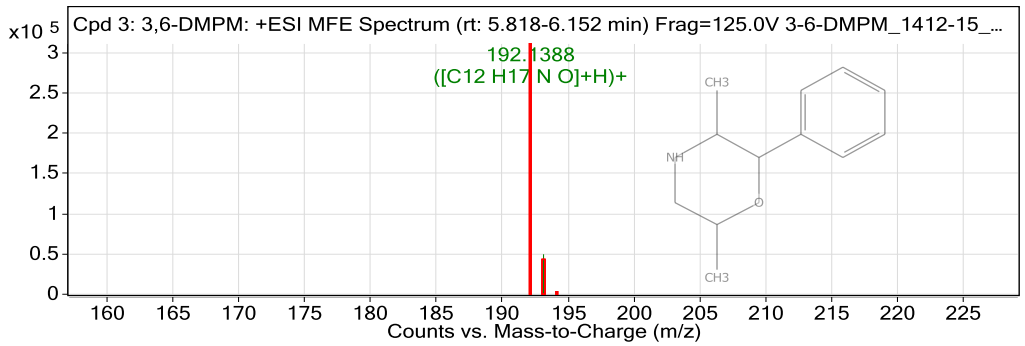
Compound Chromatograms

TOF REPORT

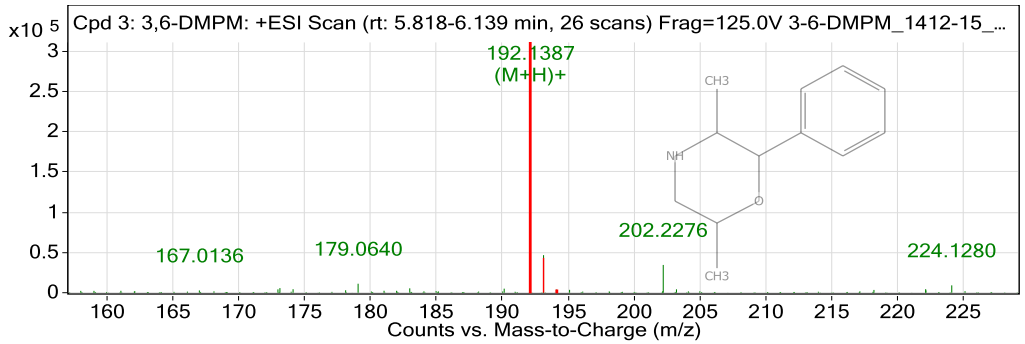




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

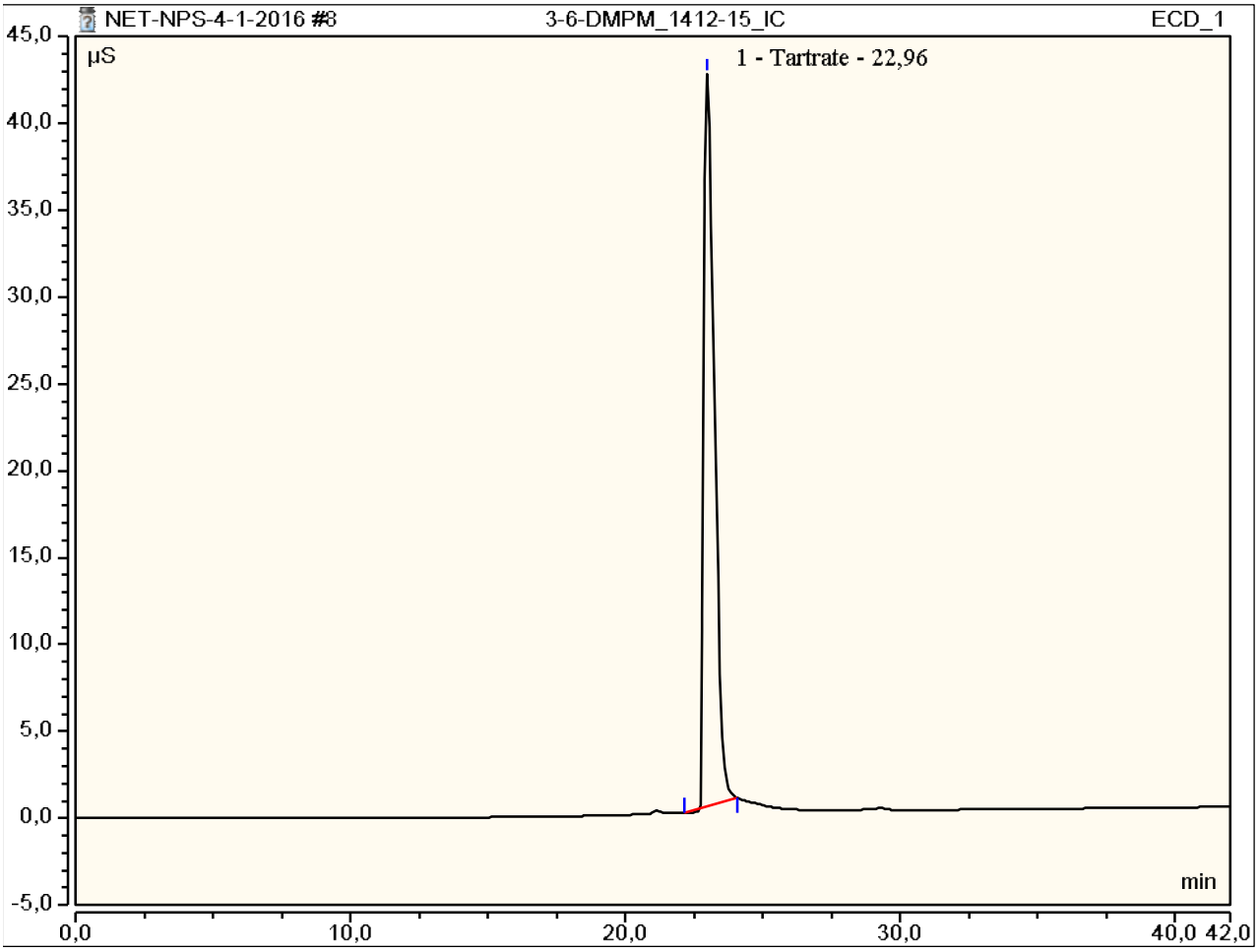
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
192.1388	1	310769.06	C12 H17 N O	(M+H)+
193.1416	1	49630.01	C12 H17 N O	(M+H)+
194.1406	1	3996.49	C12 H17 N O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**3-6-DMPM_1412-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**11-jan-2016 / 19:52**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	22,96	**Tartrate**	BMB	18,84	42,16	n.a.
		TOTAL:		18,84	42,16	0,00



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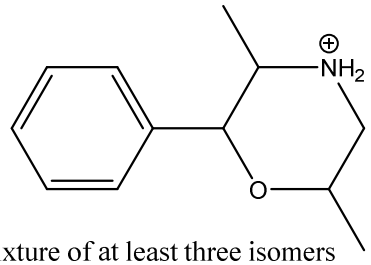
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1412-16**
Our notebook code:	P-1412-16
NMR sample preparation:	30 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:	 mixture of at least three isomers
Chemical name:	3,6-dimethyl-2-phenylmorpholin-4-ium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure according to NMR; it is a mixture of at least three isomers (possibly diastereoisomers), most probably contains tartaric anions and some additional impurities as evident by the redundant peaks in ^{13}C NMR (probably 76.8, 70.7, 49.8, 41.8 etc.). This mixture gives convoluted spectra therefore preventing a complete assignation of all peaks.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	February 16, 2016

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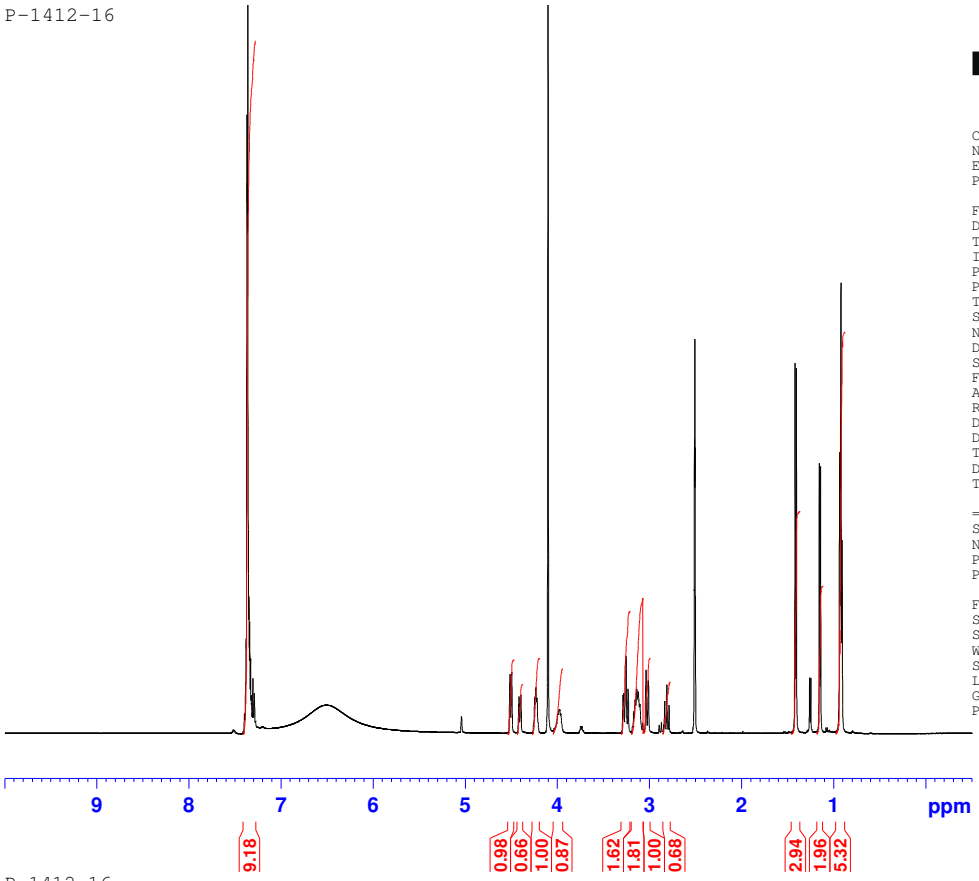
P-1412-16



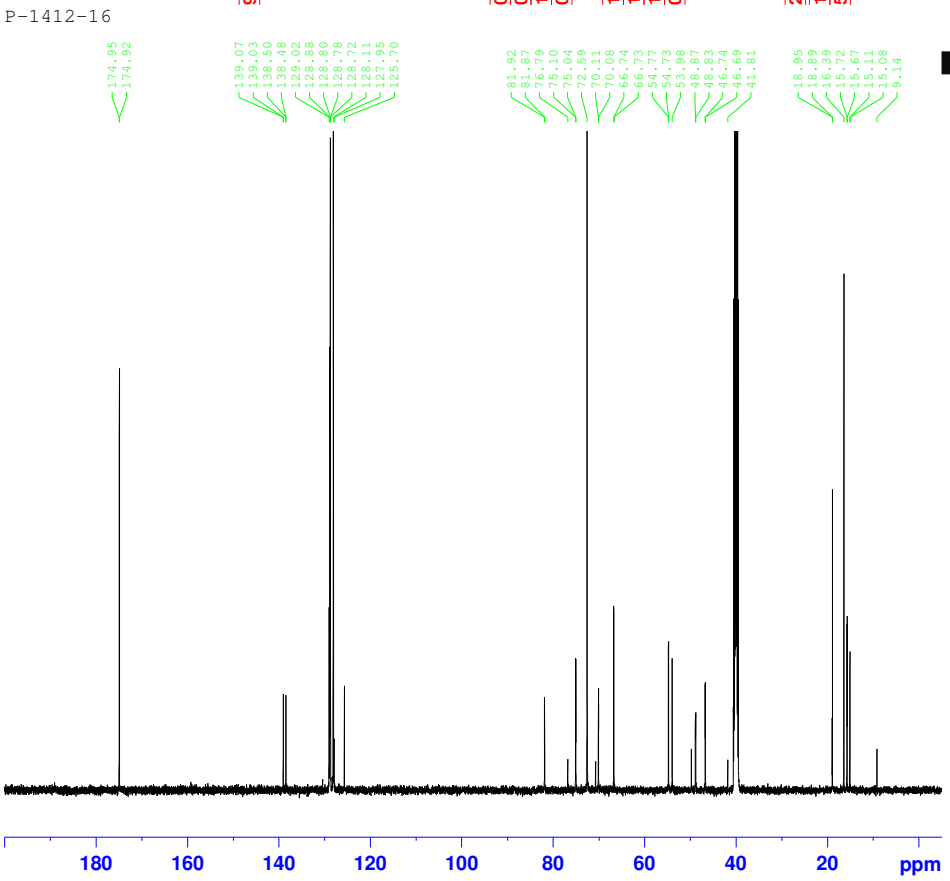

```
Current Data Parameters
NAME                  p-1412-16
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20160216
Time                      2.59
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                   DMSO
NS                         128
DS                           2
SWH                  10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                         50.8
DW                       50.000 usec
DE                        6.50 usec
TE                       300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                500.1330885 MHz
NUC1                        1H
P1                        8.90 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
```

P-1412-16




```
Current Data Parameters
NAME                  P-1412-16
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20160216
Time                      6.05
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                    1.1010048 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.7703637 MHz
NUC1                        13C
P1                        9.00 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                0.32179001 W
PLW13                0.16186000 W

F2 - Processing parameters
SI                       32768
SF                  125.7577885 MHz
WDW                         EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
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