



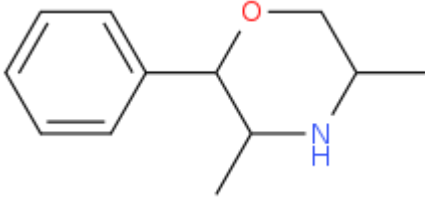
ANALYTICAL REPORT¹

PDM-35 (C₁₂H₁₇NO)

2-phenyl-3,5-dimethylmorpholine

Remark – other NPS detected: **none**

Sample ID:	1408-16
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:	2/10/2016
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	2-phenyl-3,5-dimethylmorpholine
Other names	5-Methylphenmetrazine
Formula (per base form)	C ₁₂ H ₁₇ NO
M _w (g/mol)	191,27
Salt form/anions detected	HCl
StdInChIKey	YKCSYIYQRSVLAK-UHFFFAOYSA-N
Compound Class	Others
Other NPS detected	none
Add.info (purity..)	HPLC TOF and GC-MS two major peaks for C ₁₂ H ₁₇ NO and same mass spectra; NMR: a mixture of two very similar compounds (probably diastereoisomers) in approx. molar ratio 3:2

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 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°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 °C. Chromatographic separation as above (**1**). Split MS : IR = 1:9.

MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.

IR (condensed 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

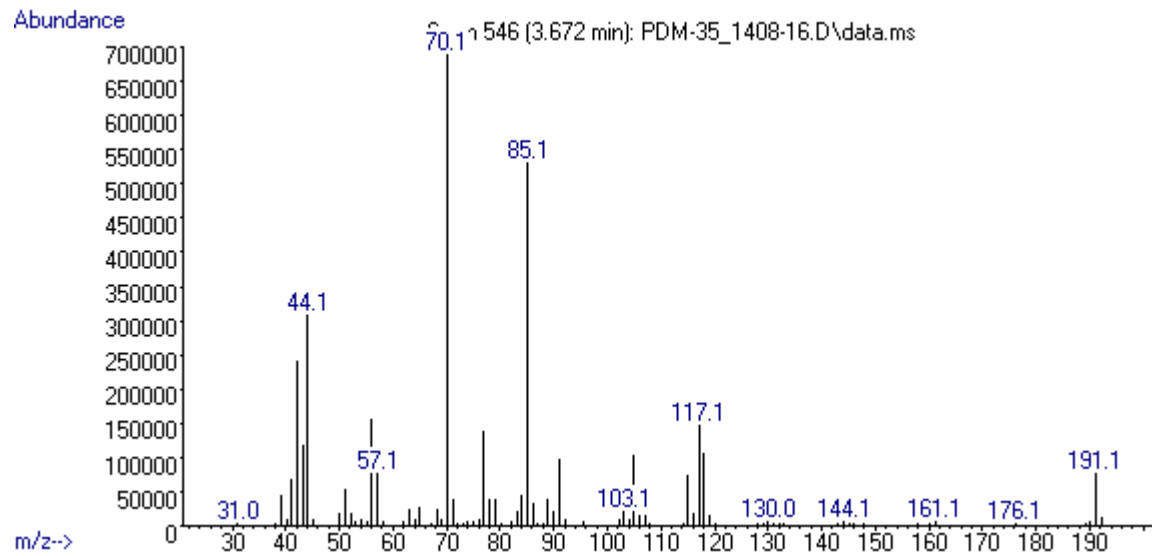
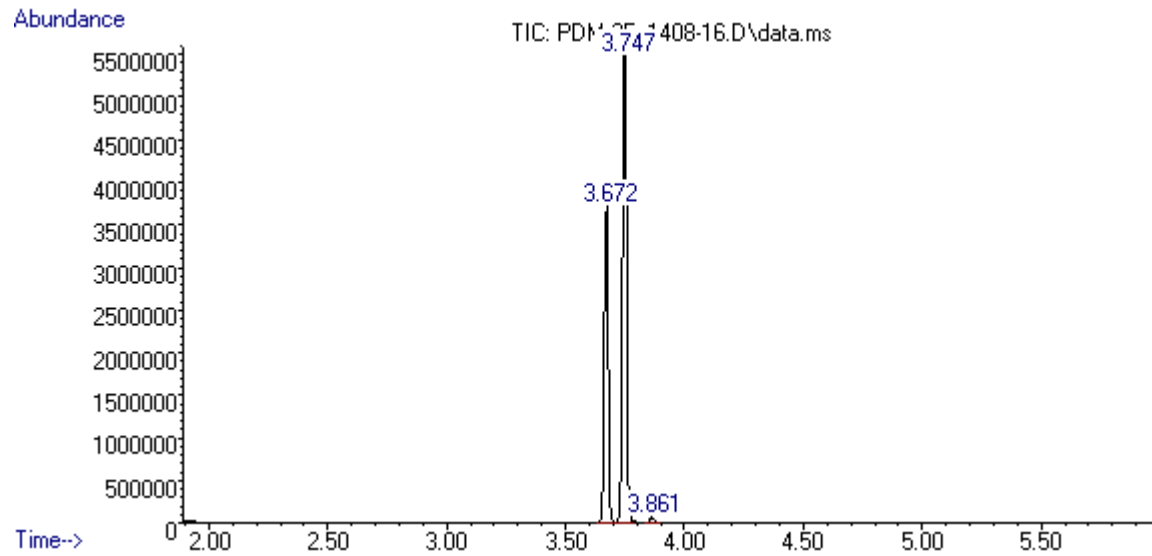
Supporting information

Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	partially

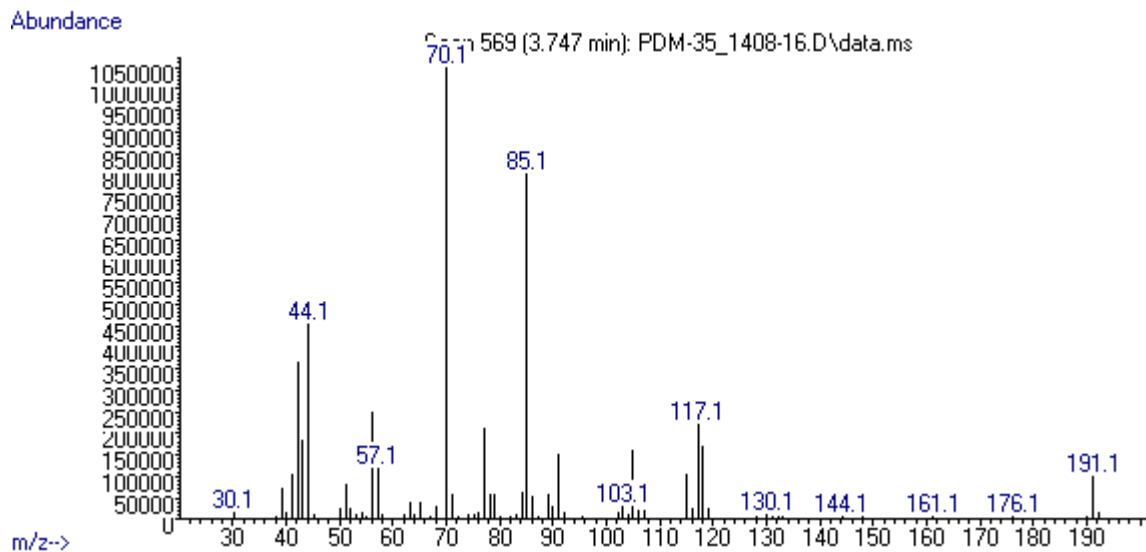
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 3,75 (major compound 2) BP(1): 70; BP(2): 85,BP(3) :44,
HPLC-TOF	+	Exact mass (theoretical): 191,131; measured value Δppm:-2,45; formula:C12H17NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	-	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

GC-MS (EI)



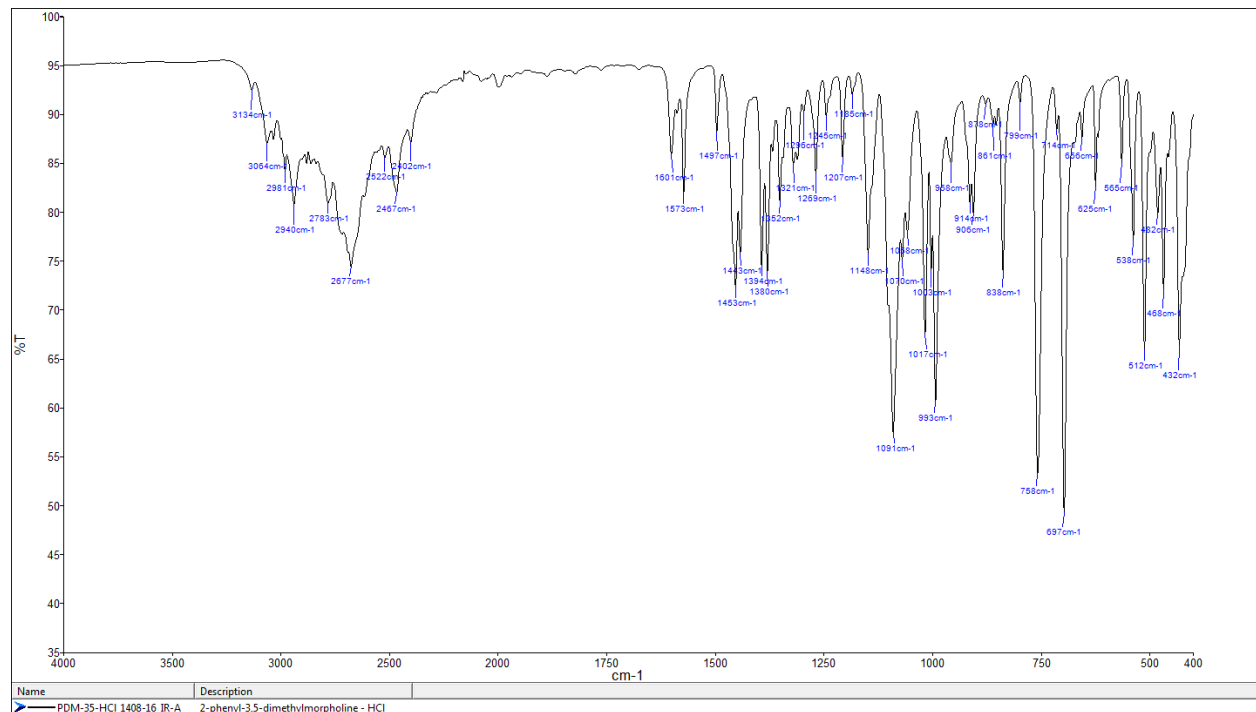
MS spectrum at 3.67 min (major compound 1)



MS spectrum at 3.75 min (major compound 2)

Remark: MS spectrum of minor compound at RT= 3.86 is practically the same as the two spectra shown above.

FTIR-ATR - direct measurement (sample as received)



TOF REPORT

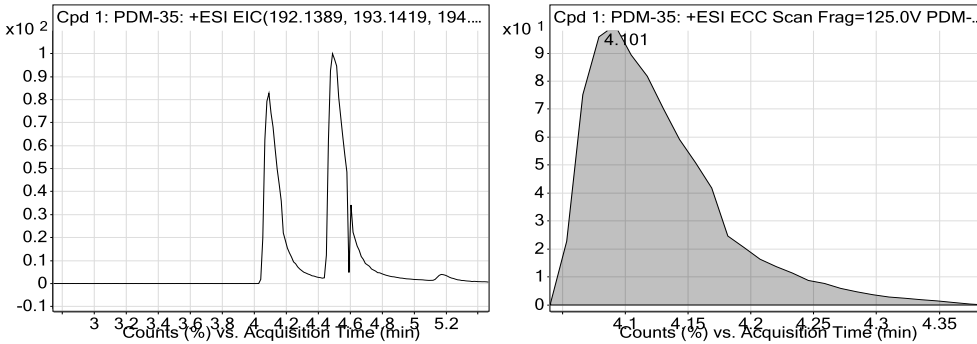
Data File	PDM-35_1408-15_TOF.d	Sample Name	ID_1408-15
Sample Type	Sample	Position	P1-D6
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-1512015-XDB-C18-ESI-poz.m	Acquired Time	1/11/2016 12:16:44 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

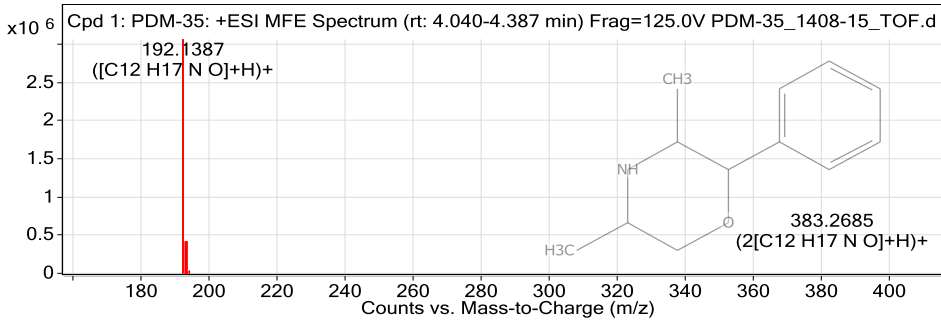
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: PDM-35	PDM-35	C12 H17 N O	4.101	191.1315
Cpd 2: PDM-35	PDM-35	C12 H17 N O	4.51	191.1312

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
PDM-35	192.1387	4.101	191.1315	4.101	C12 H17 N O	191.131	-2.45

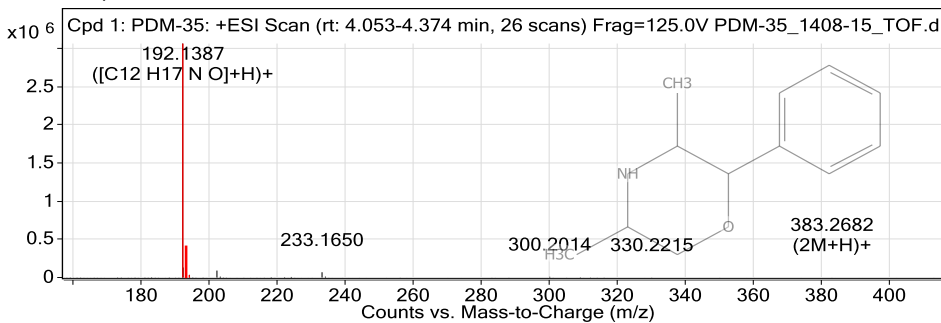
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



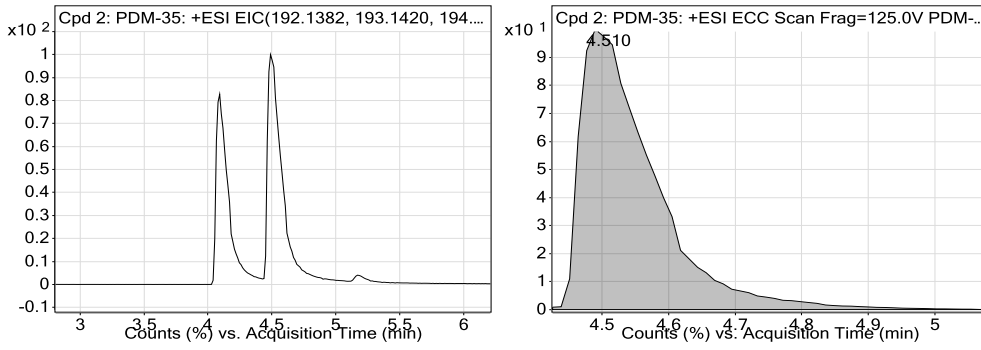
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
192.1387	1	3066859.5	C12 H17 N O	(M+H)+
193.1425	1	407815.4	C12 H17 N O	(M+H)+
194.1445	1	35390.01	C12 H17 N O	(M+H)+
195.145	1	1496.98	C12 H17 N O	(M+H)+
383.2685	1	1231.25	C12 H17 N O	(2M+H)+

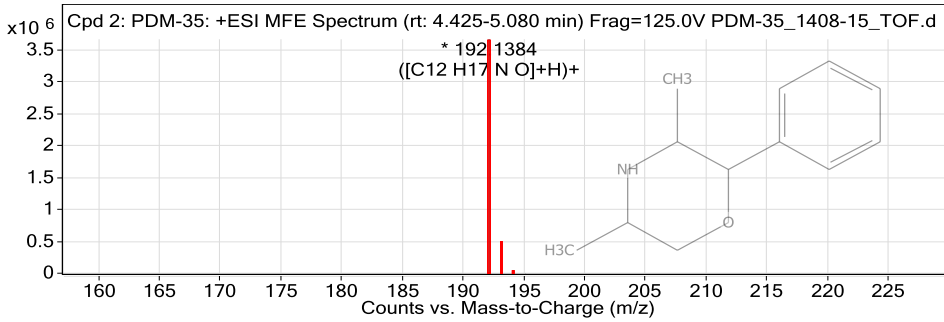
Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
PDM-35	192.1384	4.51	191.1312	4.51	C12 H17 N O	191.131	-1.13

Compound Chromatograms

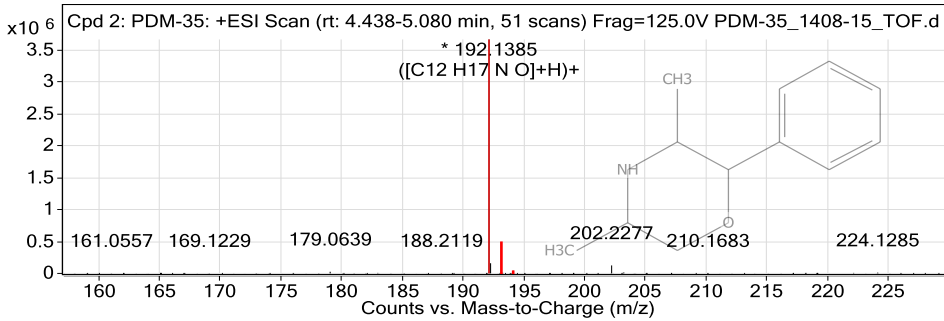
TOF REPORT



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

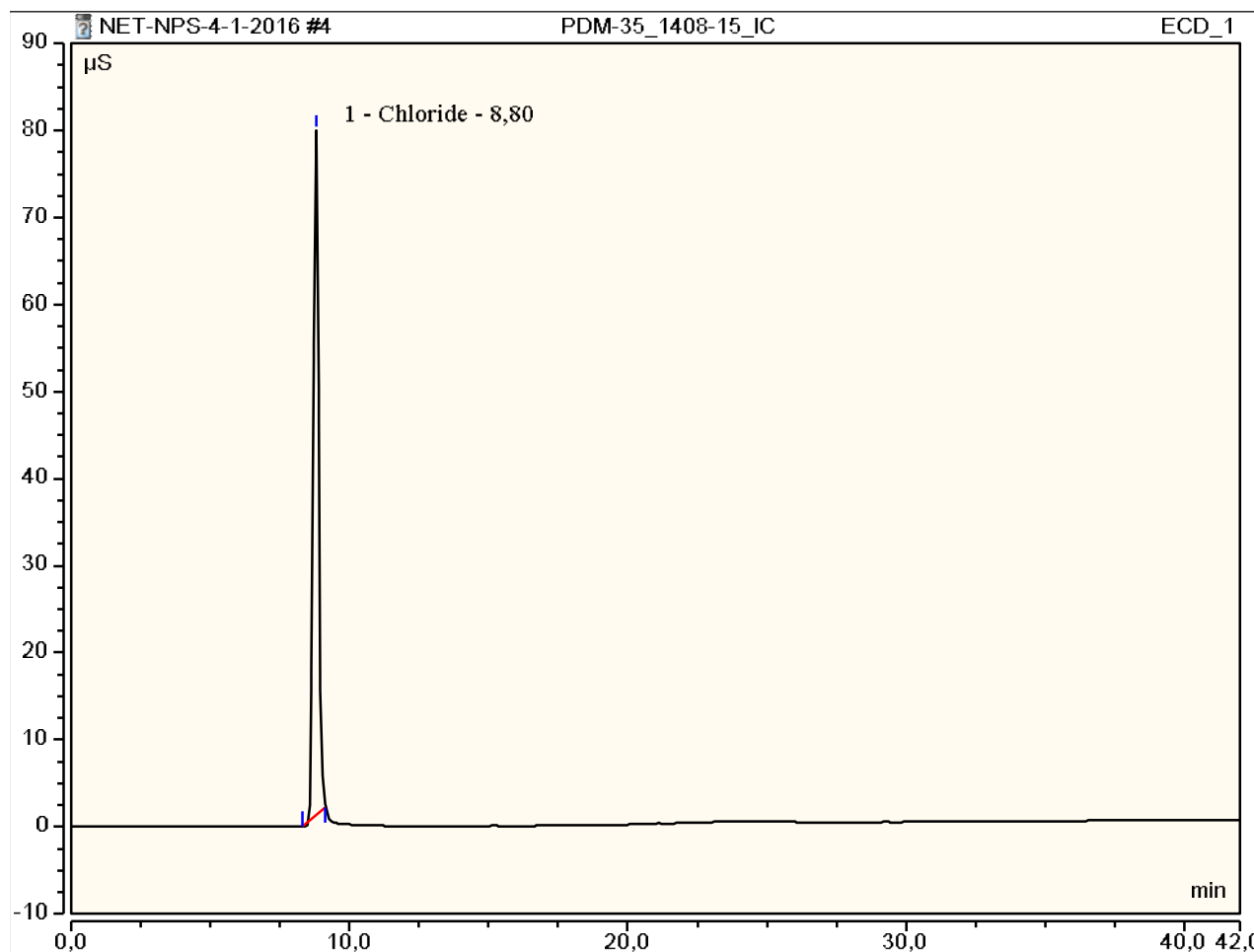
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
192.1384	1	3662285	C ₁₂ H ₁₇ N O	(M+H) ⁺
193.1424	1	479391.67	C ₁₂ H ₁₇ N O	(M+H) ⁺
194.1444	1	43062.67	C ₁₂ H ₁₇ N O	(M+H) ⁺
195.1459	1	1752.12	C ₁₂ H ₁₇ N O	(M+H) ⁺

--- End Of Report ---

Peak Integration Report

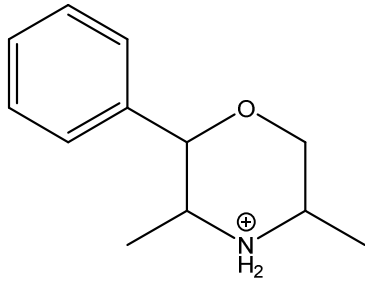
Sample Name:	PDM-35_1408-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	11-jan-2016 / 16:34	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	8,80	Chloride	BMB	17,21	78,70	n.a.
TOTAL:				17,21	78,70	0,00





REPORT

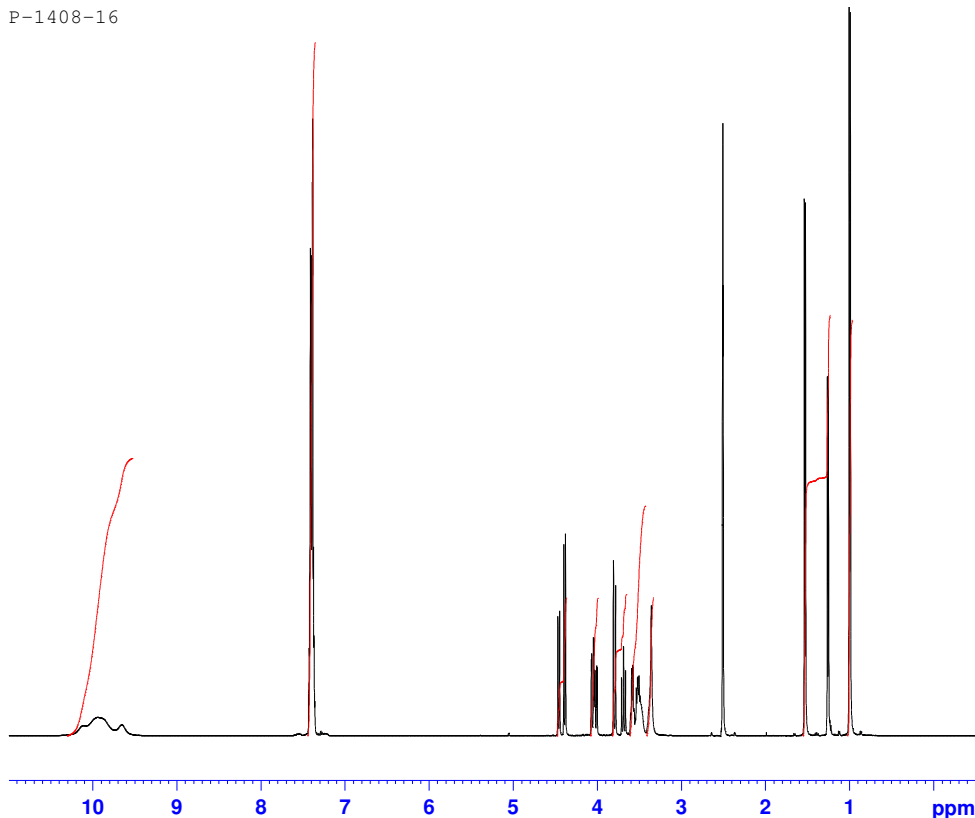
Sample ID:	1408-16
Our notebook code:	P-1408-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	3,5-dimethyl-2-phenylmorpholin-4-ium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - According to NMR the sample is a mixture of two very similar compounds (most probably diastereoisomers) in approx. molar ratio of 3 : 2. This is most evident from ^{13}C NMR where all (except one) signal are doubled.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 27, 2016

P-1408-16



Current Data Parameters
 NAME P-1408-16
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160124
 Time 9.43
 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 71.8
 DW 50.000 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1



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

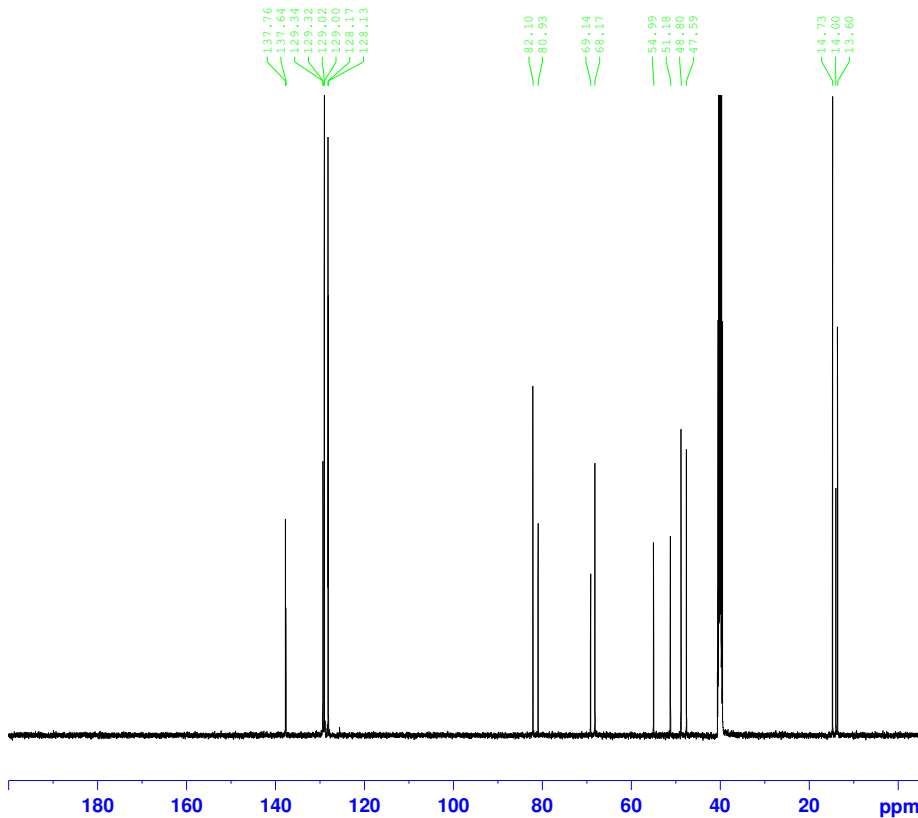
F2 - Processing parameters
 SI 65536
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

P-1408-16



Current Data Parameters
 NAME P-1408-16
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160124
 Time 12.23
 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 298.0 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