

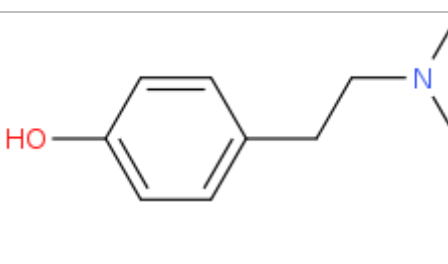
ANALYTICAL REPORT

Hordenine (C₁₀H₁₅NO)

4-[2-(dimethylamino)ethyl]phenol

Remark – other NPS detected:

Sample ID:	1890-17
Sample description:	powder
Sample type:	seized /KP
Date of sample receipt (M/D/Y):	10/2/2017
Date of entry (M/D/Y) into NFL database:	1/10/2018
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-[2-(dimethylamino)ethyl]phenol
Other names	p-(2-Dimethylaminoethyl)phenol; N,N-dimethyltyramine
Formula (per base form)	C ₁₀ H ₁₅ NO
M _w (g/mol)	165,24
Salt form/anions detected	HCl
StdInChIKey (per base form)	KUBCEEMXQZUPDQ-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	>98% purity by 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 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 7.1 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 (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 °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 (30 until 6 min.) to 550 (300) amu.

IR (condensed (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

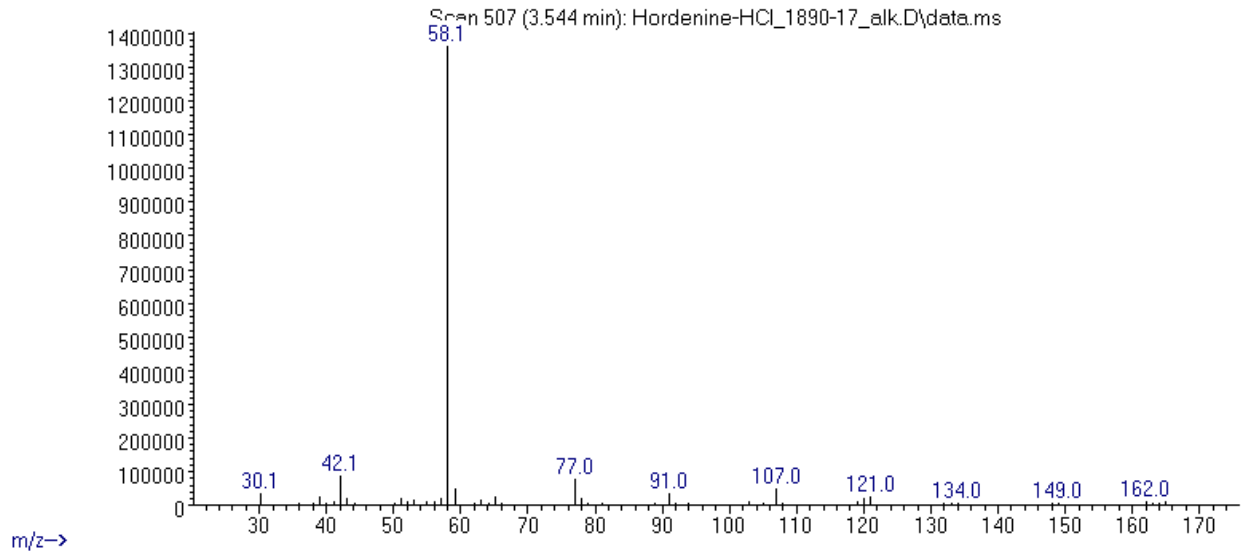
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 3,54 BP(1): 58; BP(2): 42,BP(3) :77,
HPLC-TOF	+	Exact mass (theoretical): 165,1154; measured value Δppm:0,02; formula:C10H15NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

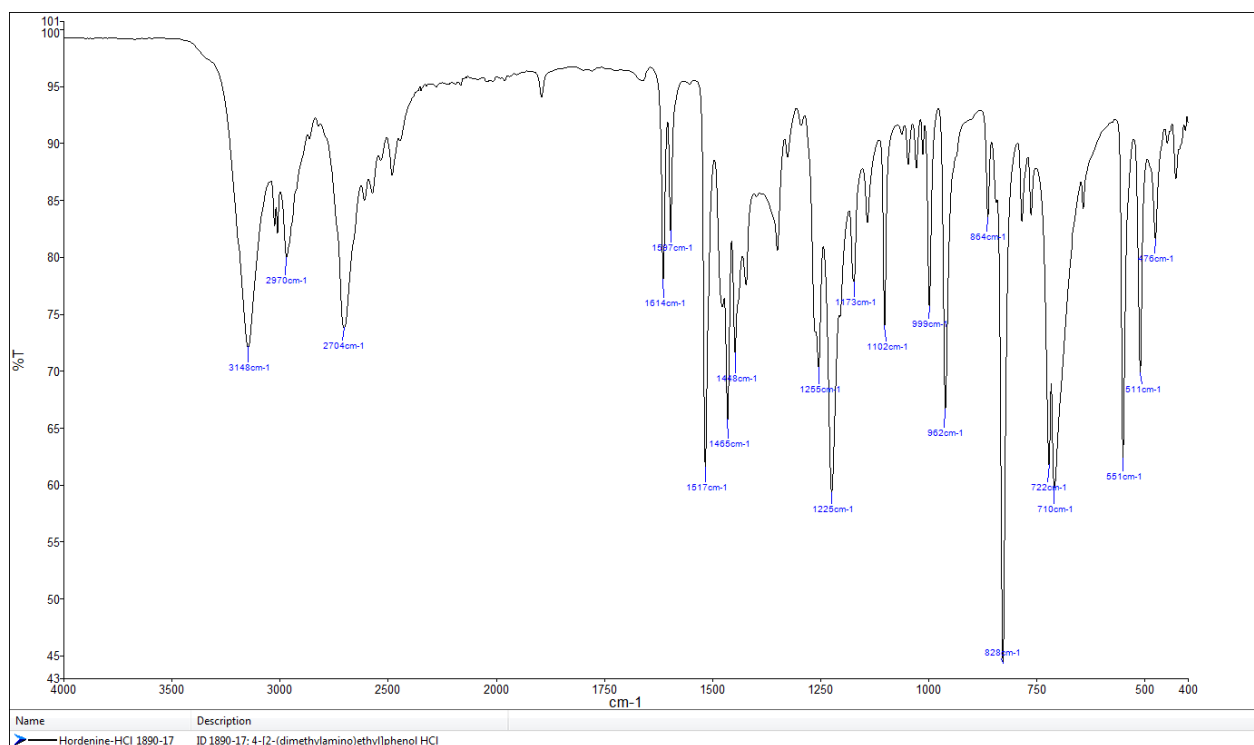
ANALYTICAL RESULTS

MS (EI)

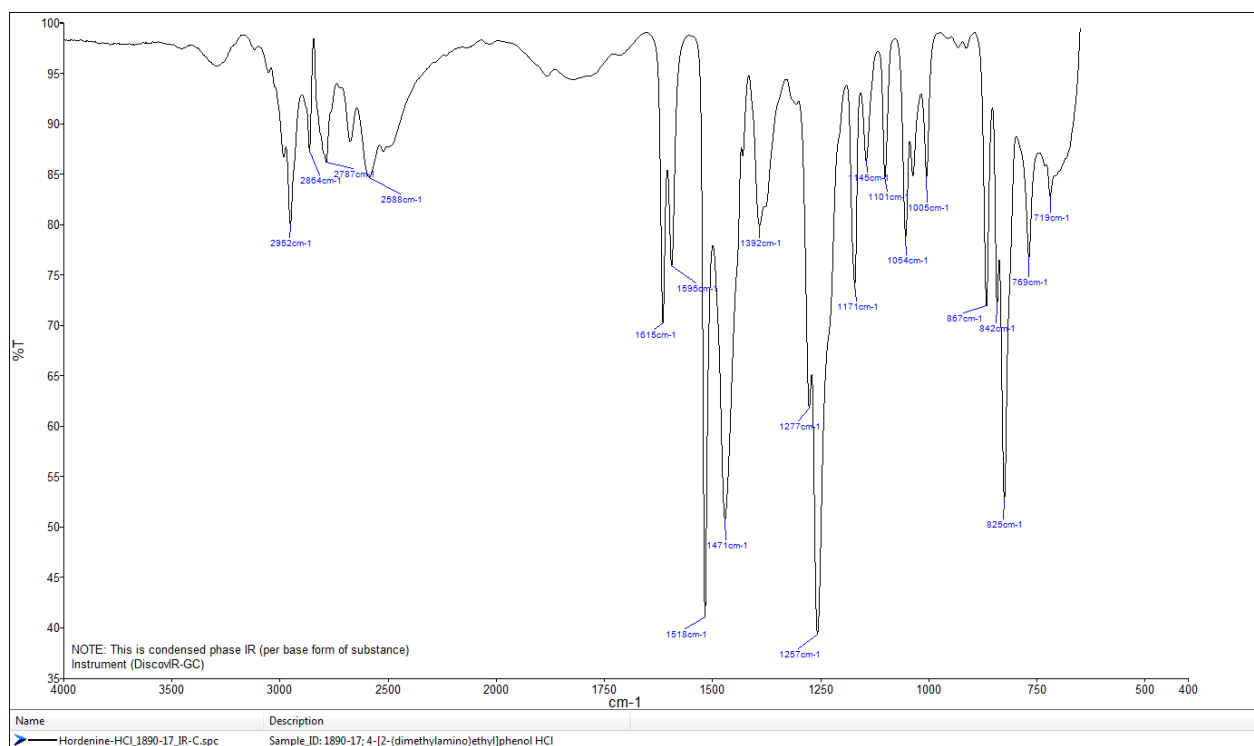
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

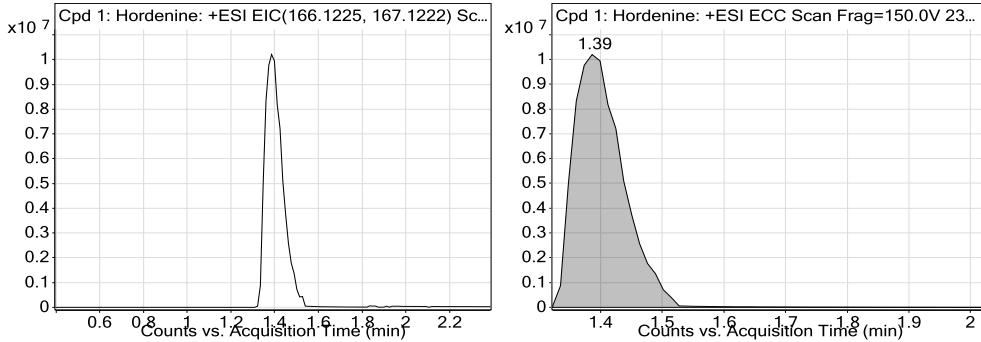
Data File	233-5217-2017_17.d	Sample Name	eks. in MeOH
Sample Type	Sample	Position	P1-D4
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-19_07_2017-XDB-C18-ESI-final.m	Acquired Time	11/10/2017 1:48:00 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	MeOH		

Compound Table

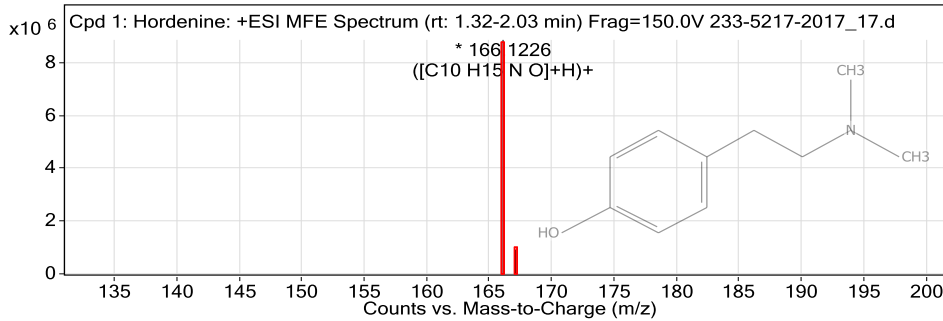
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Hordenine	Hordenine	C10 H15 N O	1.39	165.1154

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Hordenine	166.1226	1.39	165.1154	1.39	C10 H15 N O	165.1154	0.02

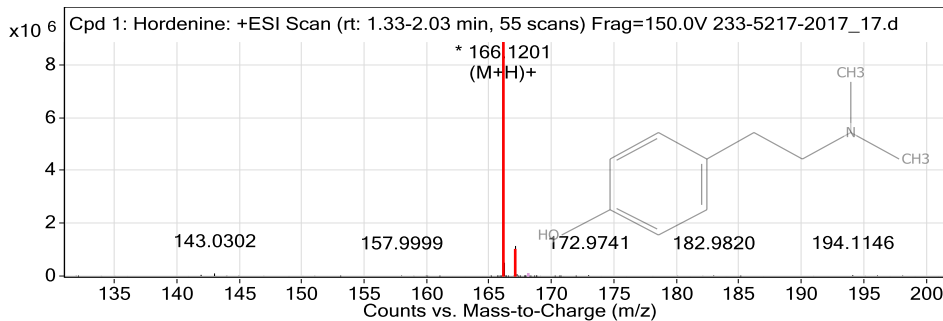
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

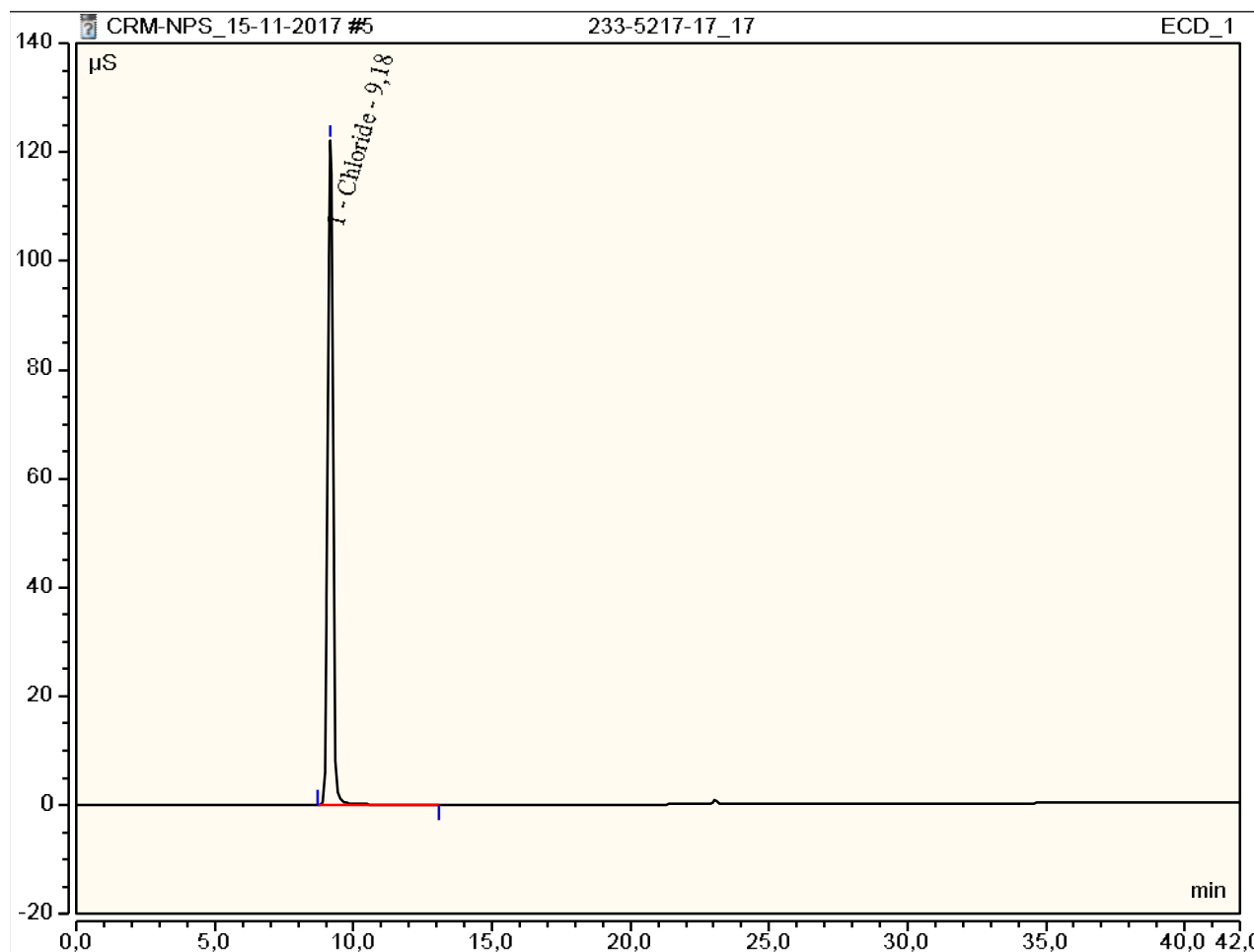
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
166.1226	1	8859580	C10 H15 N O	(M+H)+
167.126	1	917256.23	C10 H15 N O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	233-5217-17_17	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	15-nov-2017 / 12:55	Run Time:	42,00

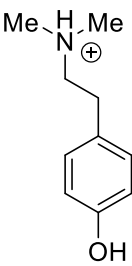
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	9,18	Chloride	BMB	25,62	122,16	n.a.
TOTAL:				25,62	122,16	0,00



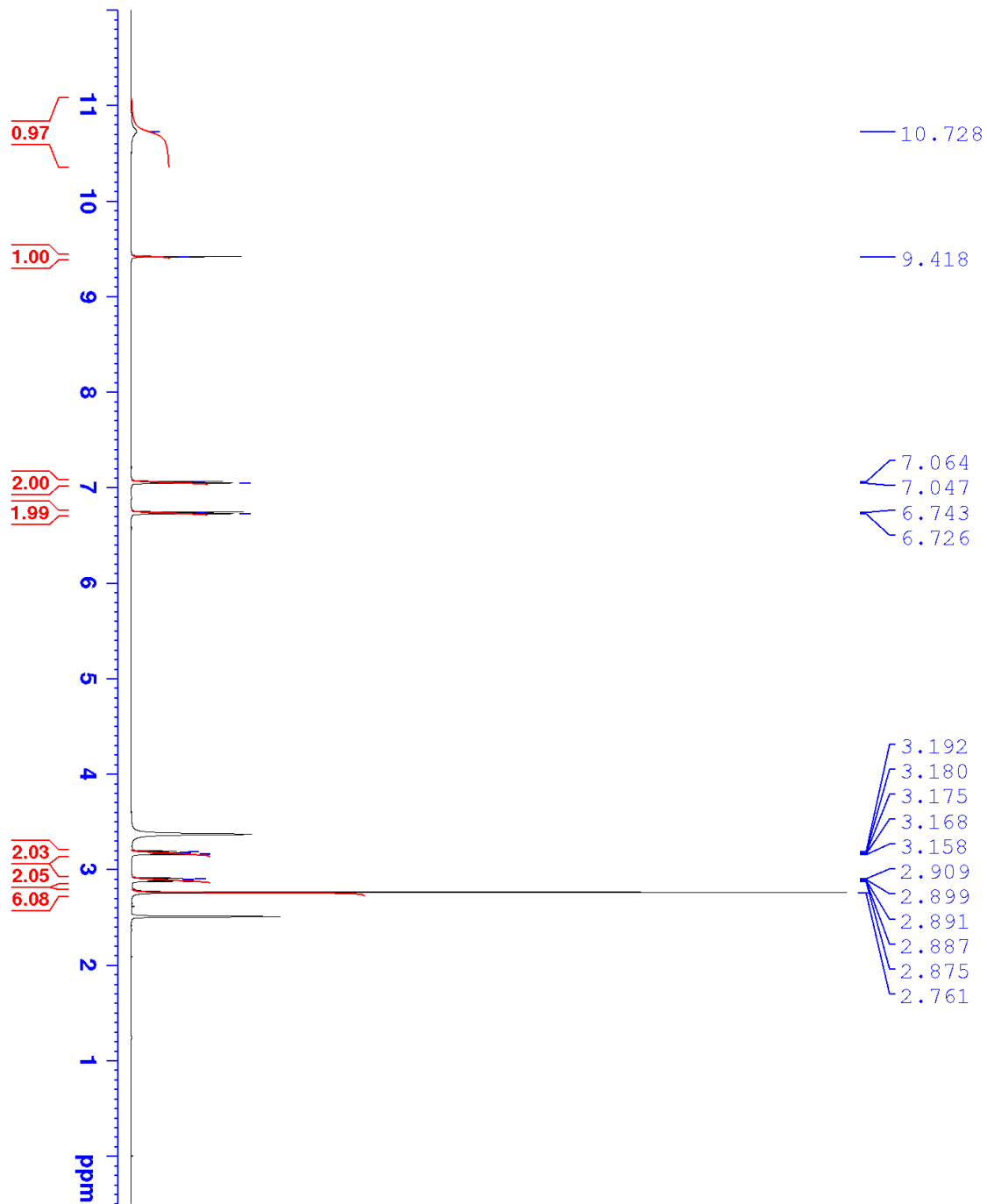
University
of Ljubljana
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	1890-17
Received date:	December 8, 2017
Our notebook code:	NFL-1890-17
NMR sample preparation:	16.2 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC
Proposed structure with formula, exact mass, molecular weight:	 <p>Chemical Formula: C₁₀H₁₆NO⁺ Exact Mass: 166,1226 Molecular Weight: 166,2435</p>
Chemical name:	<i>N</i> -protonated 4-(2-(dimethylamino)ethyl)phenol
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >98% purity of a sample based on ¹ H NMR spectrum
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra, ¹ H and ¹³ C FIDs
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	December 22, 2017

NFL-1890-17



Current Data Parameters
NAME NFL-1890-17
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171209
Time 7.01

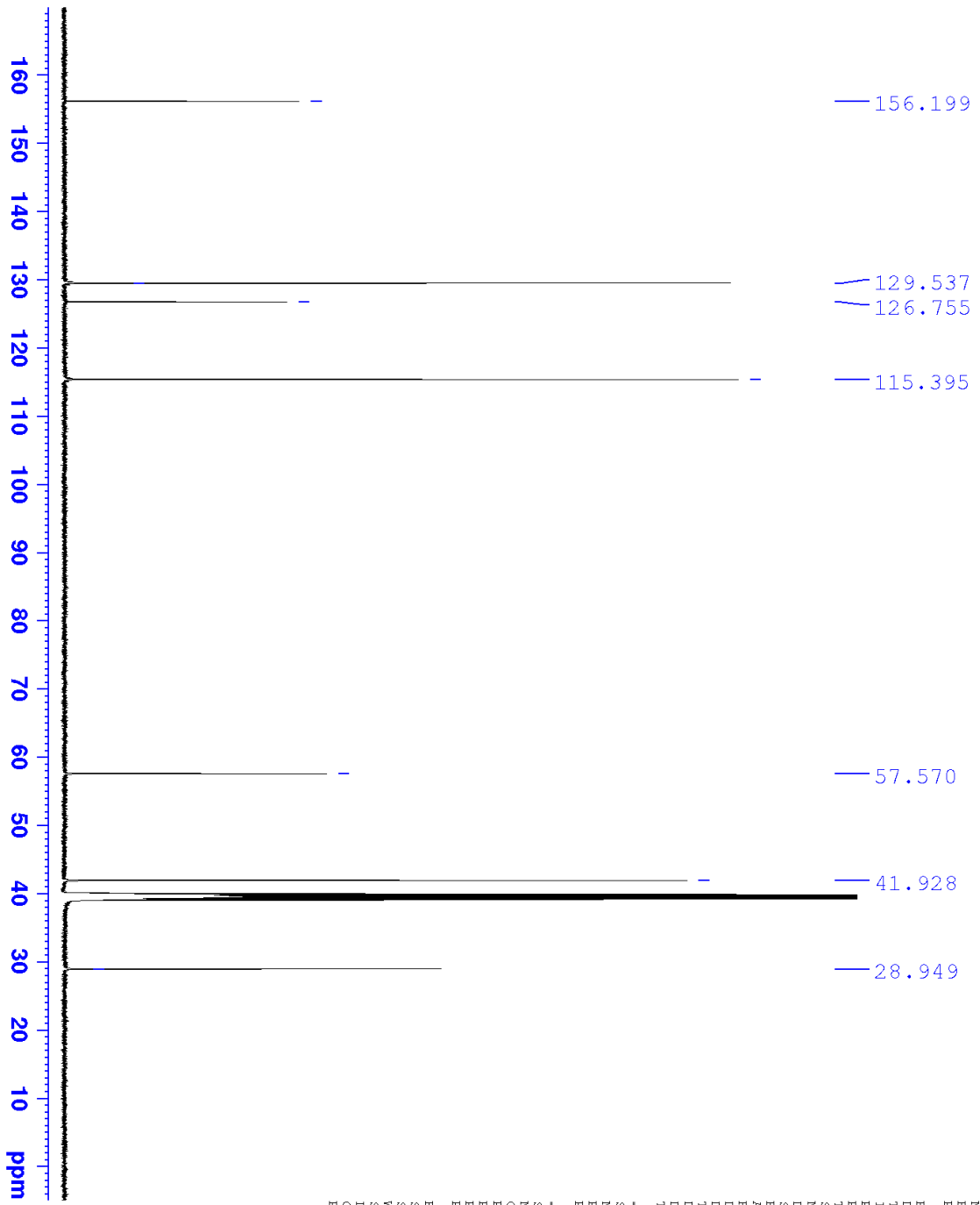
INSTRUM spect
PROBHD 5 mm PABBO BH-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2

SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.276799 sec
RG 80.6
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 8.70 usec
PLW1 26.0000000 W

F2 - Processing parameters
SI 65536
SF 500.1300010 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-1890-17



Current Data Parameters
 NAME NFL-1890-17
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171209
 Time 9.35

INSTRUM spect
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 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 299.1 K
 D1 2.0000000 sec
 D11 0.030000000 sec
 TD0 1

----- CHANNEL f1 -----
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 8.70 usec
 P1M1 122.00000000 W

----- CHANNEL f2 -----
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRG12 waltz16
 PCPD2 80.00 usec
 P1M2 26.000000000 W
 P1M3 0.30046001 W
 P1M13 0.15113001 W

F2 - Processing parameters
 SI 32768
 SF 125.7578516 MHz
 MDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40