

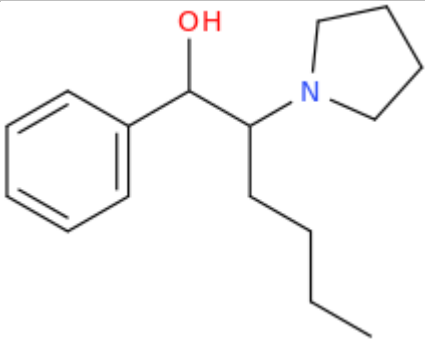
ANALYTICAL REPORT

BOH-PHP (C16H25NO)

1-phenyl-2-(pyrrolidin-1-yl)hexan-1-ol

Remark – other NPS detected: none

Sample ID:	2131-19
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	19/02/2020
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	1-phenyl-2-(pyrrolidin-1-yl)hexan-1-ol
Other names	
Formula (per base form)	C16H25NO
M _w (g/mol)	247,38
Salt form/anions detected	HCl
StdInChIKey (per base form)	GJHNPZVIGWPXBH-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>99% pure based on 1H NMR spectrum

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 µl 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 µl 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 KOH 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

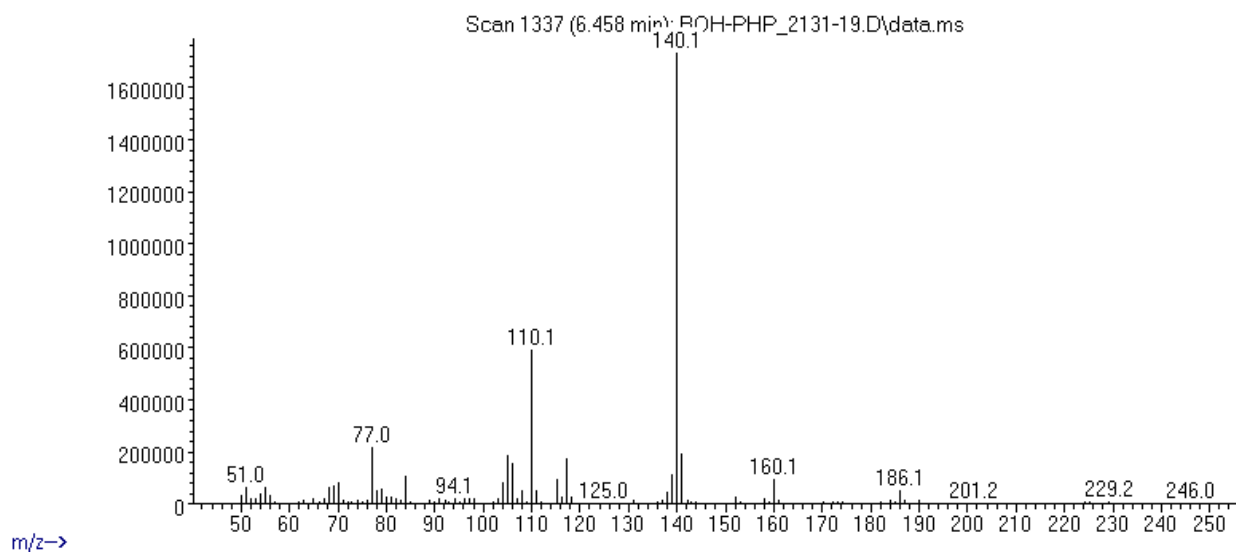
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 6,46 BP(1): 140; BP(2): 110,BP(3) :77,
HPLC-TOF	+	Exact mass (theoretical): 247,1936; measured value Δppm:-1,55; formula:C16H25NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

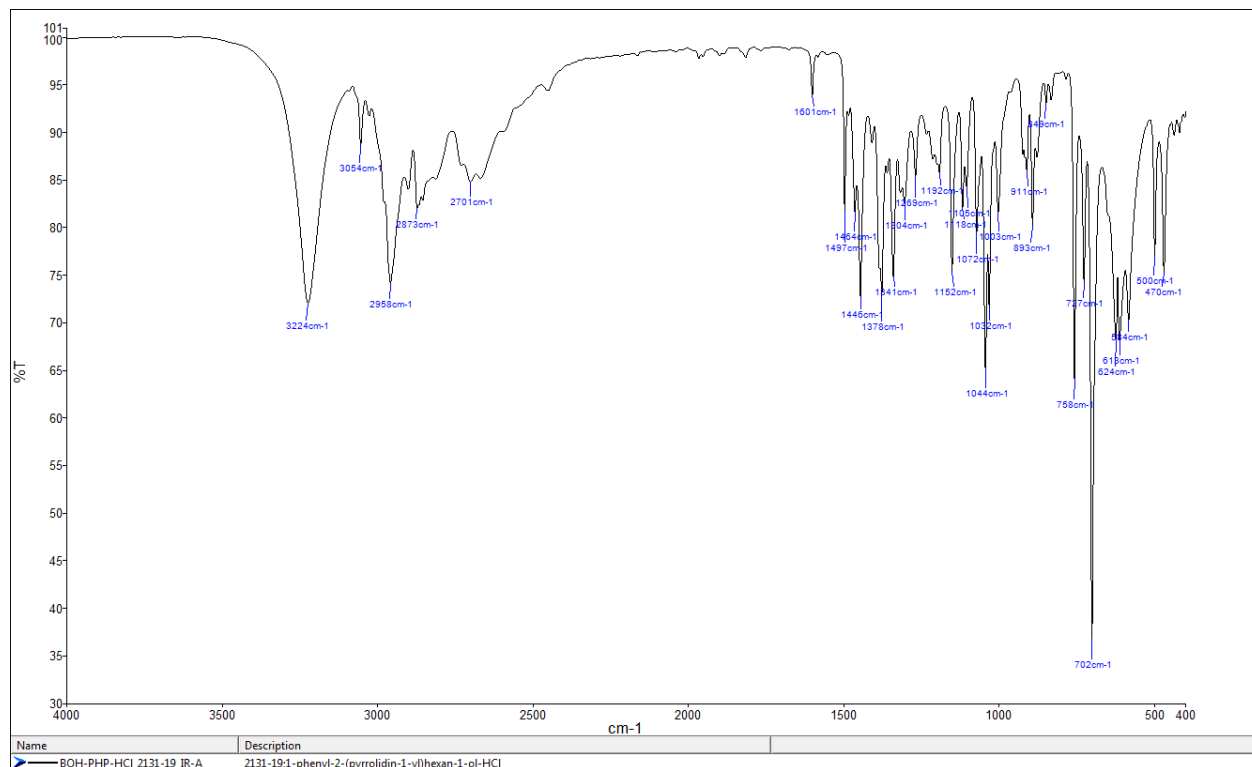
ANALYTICAL RESULTS

MS (EI)

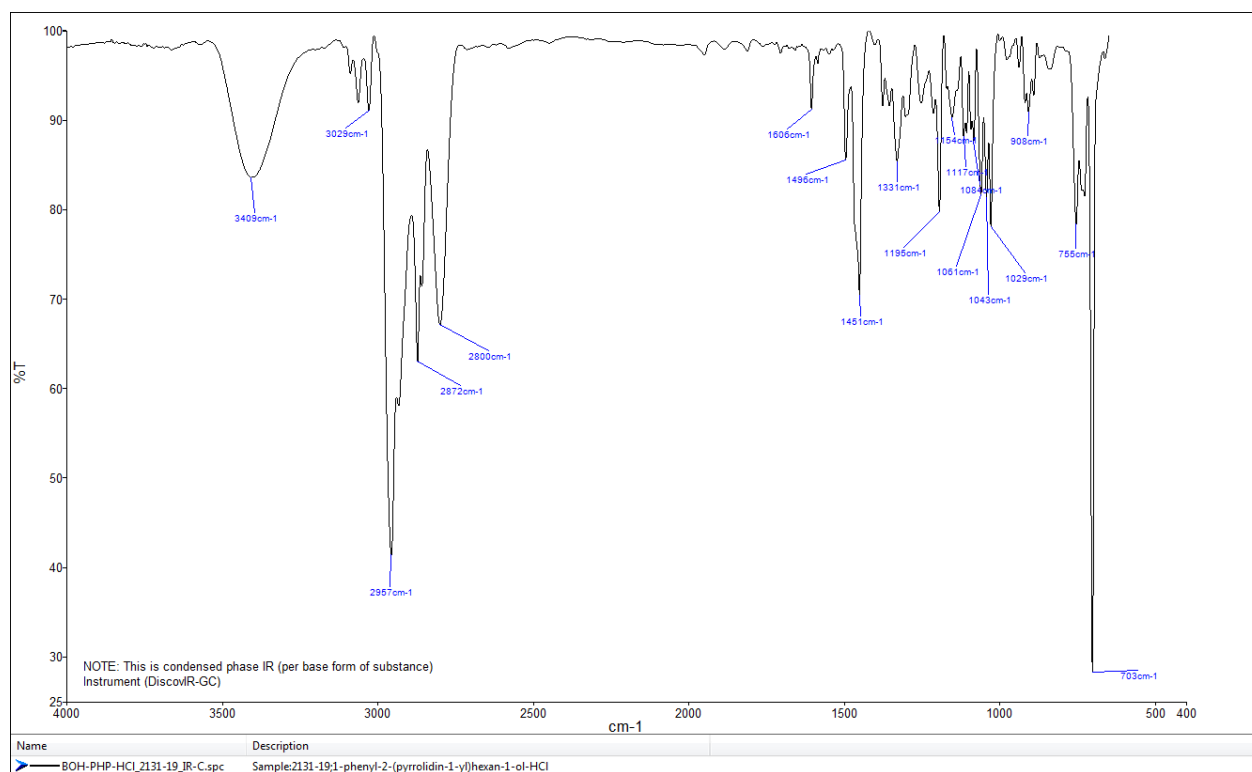
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

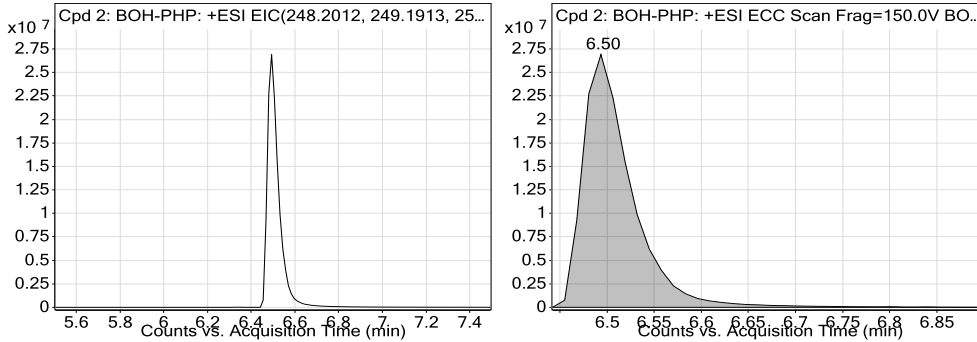
Data File	BOH-PHP_2131-19.d	Sample Name	ID-2131-19
Sample Type	Sample	Position	P1-D2
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-19_10_2019-XDB-C18-ESI+.m	Acquired Time	1/8/2020 12:47:12 PM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

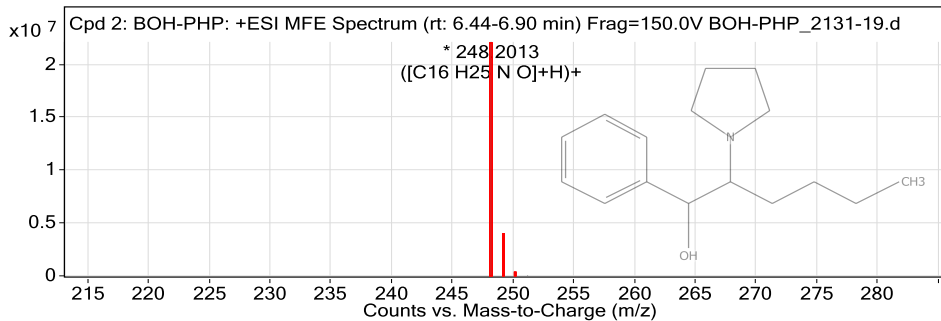
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: BOH-PHP	BOH-PHP	C16 H25 N O	6.5	247.194

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
BOH-PHP	248.2013	6.5	247.194	6.5	C16 H25 N O	247.1936	-1.55

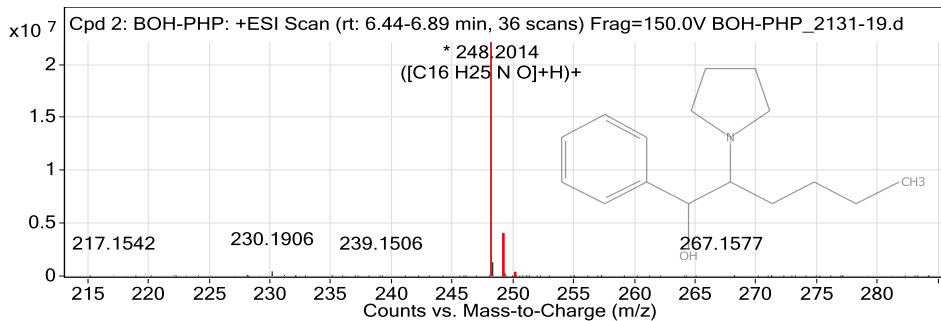
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

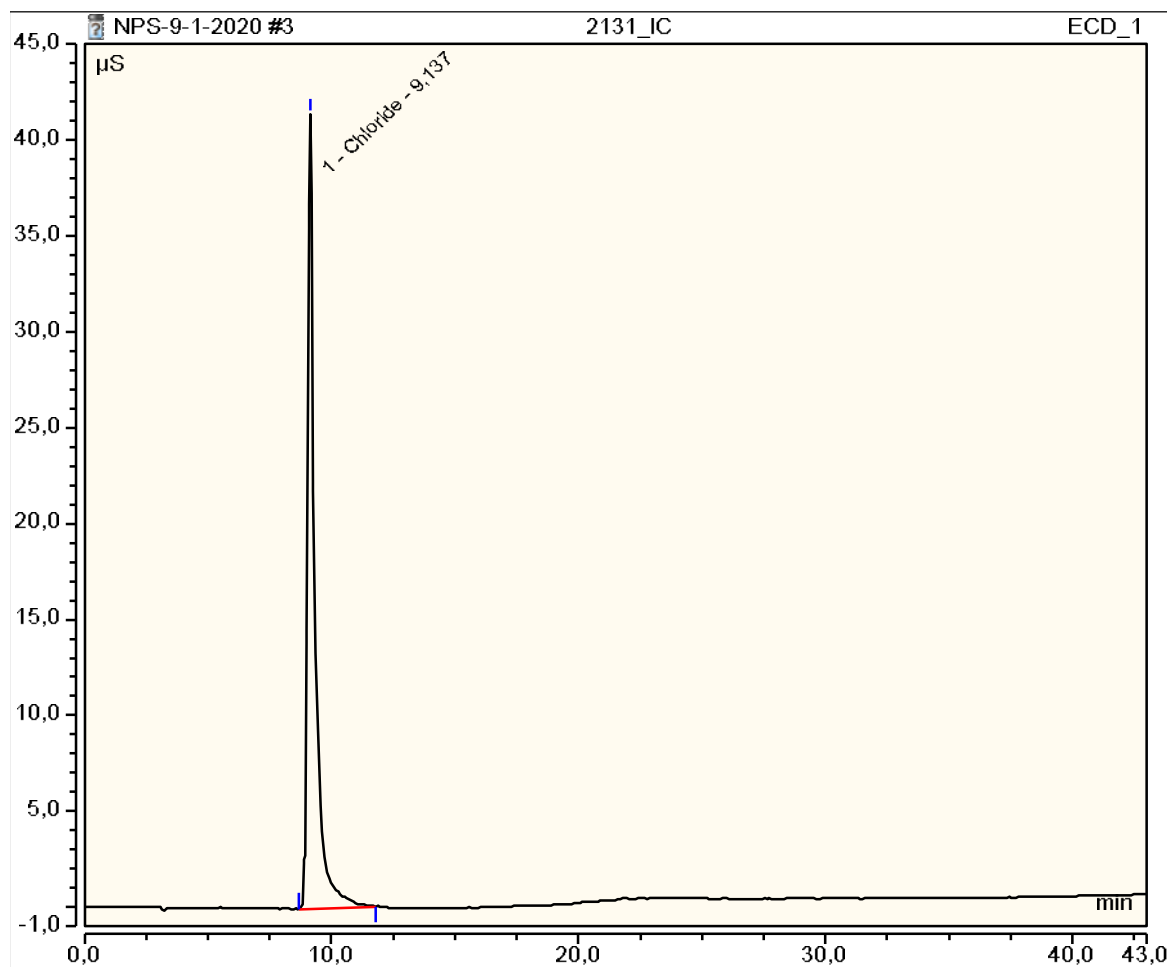
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
248.2013	1	22114486	C16 H25 N O	(M+H)+
249.2045	1	3826190.08	C16 H25 N O	(M+H)+
250.2078	1	352806.73	C16 H25 N O	(M+H)+
251.2102	1	24252.37	C16 H25 N O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	2131_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	09-jan-2020 / 08:52	Run Time:	43,00

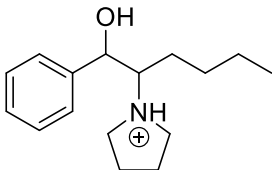
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height μS	Amount mg/L
1	9,14	Chloride	BMB	14,306	41,436	n.a.
TOTAL:				14,31	41,44	0,0



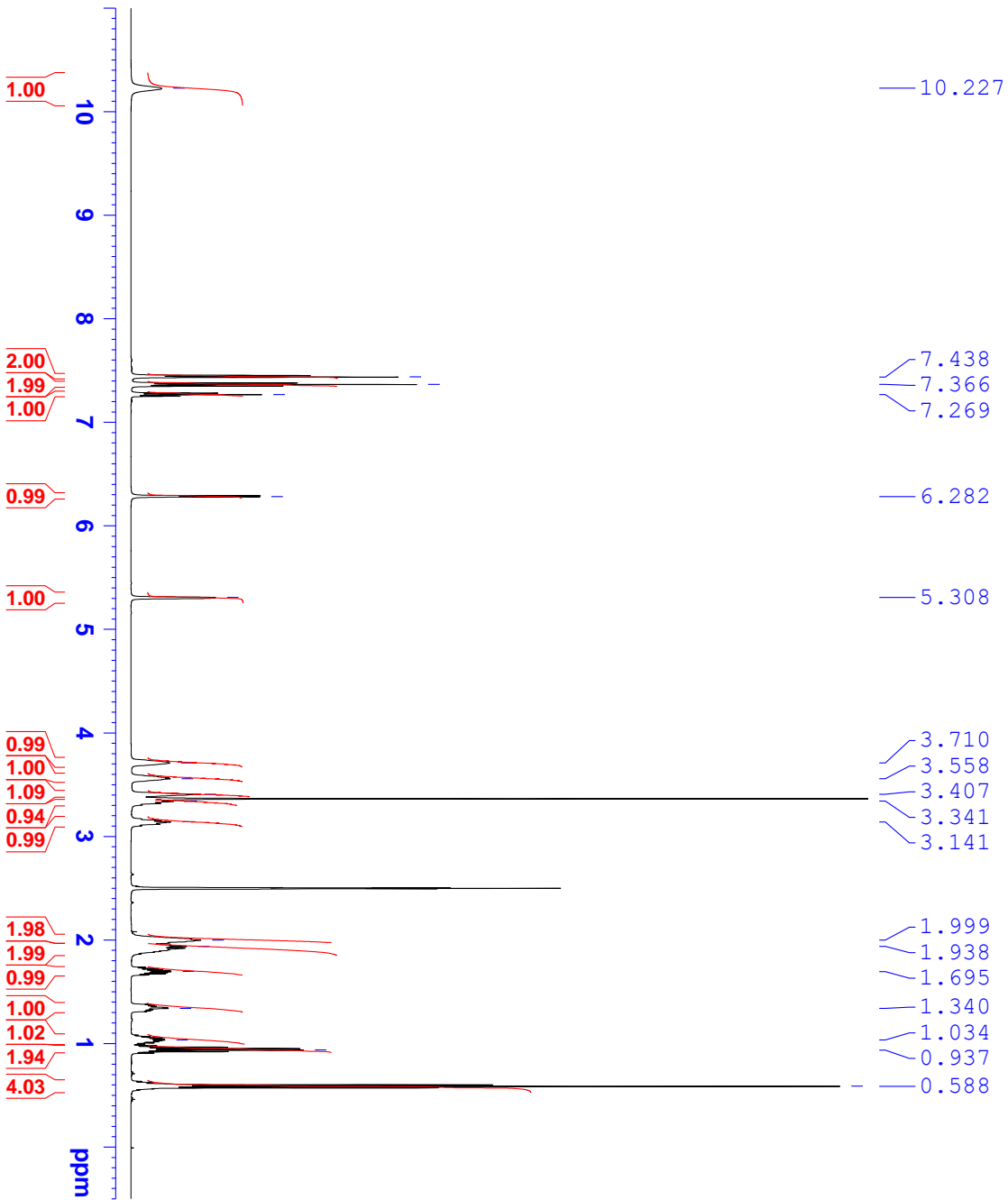
University
of Ljubljana
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2131-19
Received date:	January 23, 2020
Our notebook code:	NFL-2131-19
NMR sample preparation:	21.3 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: C₁₆H₂₆NO⁺ Exact Mass: 248,2009 Molecular Weight: 248,3895</p>
Chemical name:	<i>N</i> -protonated 1-phenyl-2-(pyrrolidin-1-yl)hexan-1-ol
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >99% 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:	February 17, 2020

NFL-2131-19
1H



Current Data Parameters
 NAME NFL-2131-19
 EXPNO 1
 PROCNO 1

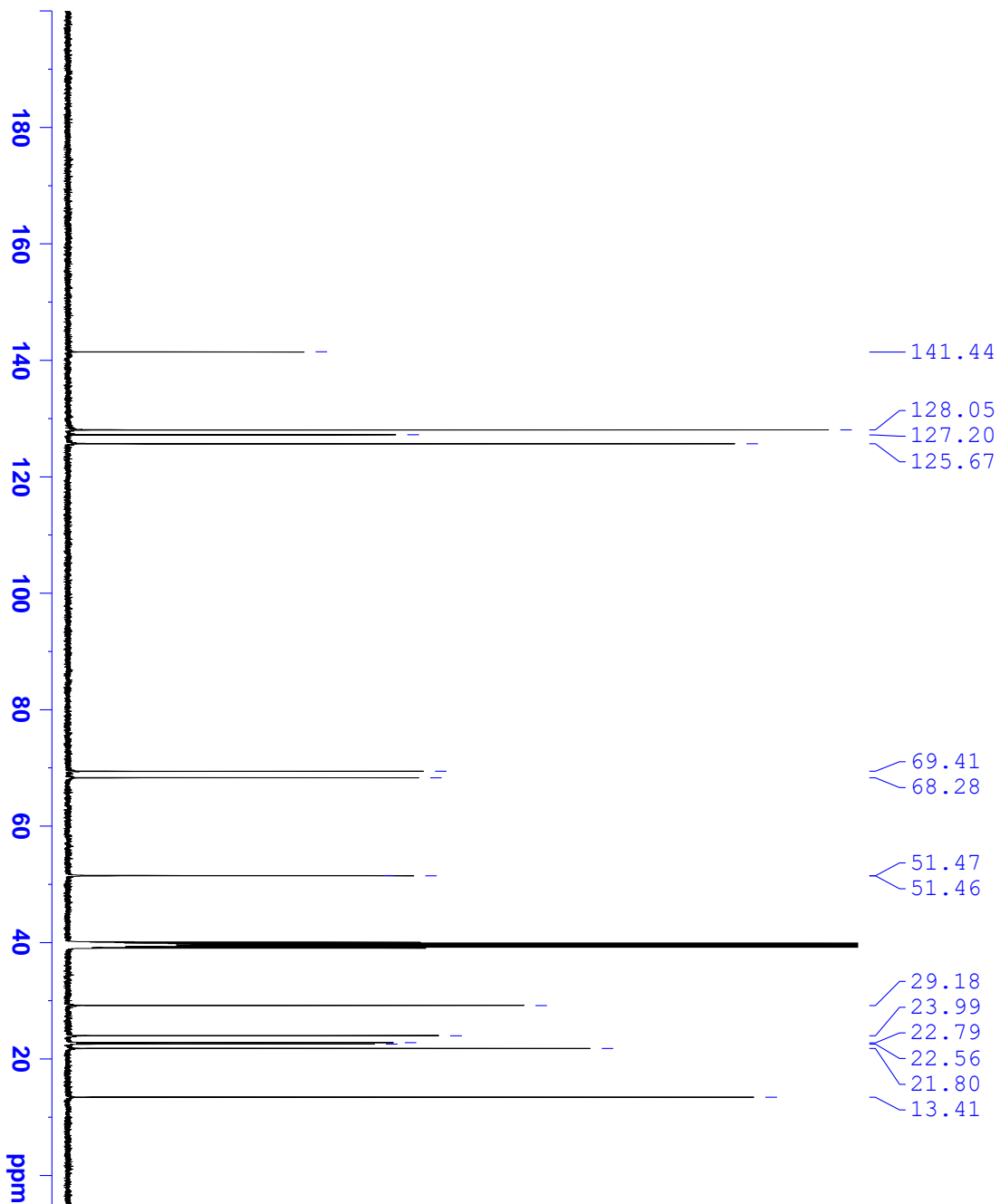
F2 - Acquisition Parameters

Date_ 20200124
 Time 17.16
 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 3.2767999 sec
 RG 57
 DW 50.000 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 8.70 usec
 PL1 26.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300043 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NFL-2131-19
13C



Current Data Parameters
 NAME NFL-2131-19
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20200124
 Time 18.32

INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 2048

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 296.0 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 8.70 usec
 PLW1 122.0000000 W

==== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CDPORG2 waltz16
 PCPD2 80.00 usec
 PLW2 26.0000000 W
 PLW12 0.30046001 W
 PLW13 0.15113001 W

F2 - Processing parameters
 SI 32768
 SF 125.7578463 MHz
 MDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40