

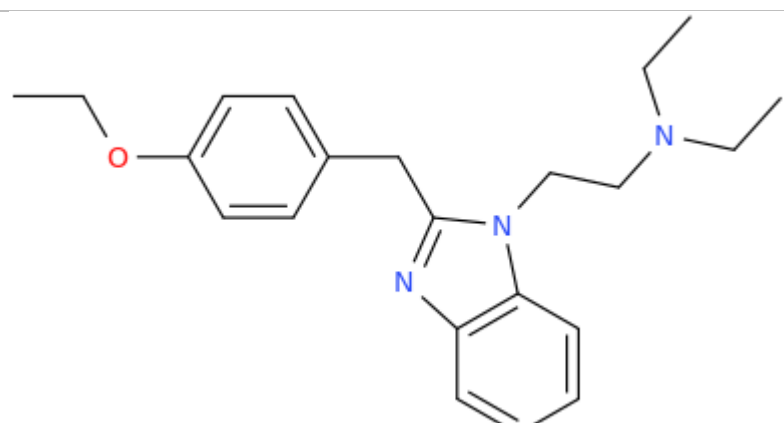
## ANALYTICAL REPORT

Etazene (C<sub>22</sub>H<sub>29</sub>N<sub>3</sub>O)

## (2-{2-[(4-ethoxyphenyl)methyl]-1H-1,3-benzodiazol-1-yl}ethyl)diethylamine

Remark – other NPS detected:

Sample ID:	2199-20
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of entry (DD/MM/YYYY) into NFL database:	18/12/2020
Report updates (if any) will be published here:	<a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a>

Substance identified - structure <sup>1</sup> (base form)	
Systematic name	(2-{2-[(4-ethoxyphenyl)methyl]-1H-1,3-benzodiazol-1-yl}ethyl)diethylamine
Other names	2-[(4-ethoxyphenyl)methyl]-N,N-diethyl-1H-benzimidazole-1-ethanamine
Formula (per base form)	C <sub>22</sub> H <sub>29</sub> N <sub>3</sub> O
M <sub>w</sub> (g/mol)	351,49
Salt form/anions detected	HCl
StdInChIKey (per base form)	BMLPNUNXHUGDOI-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	>98% purity of a sample based on 1H NMR

<sup>1</sup> 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 (N<sub>2</sub>) 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**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<sup>-1</sup>.

**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 <sub>2</sub> Cl <sub>2</sub>	partially
MeOH	soluble
H <sub>2</sub> O	soluble

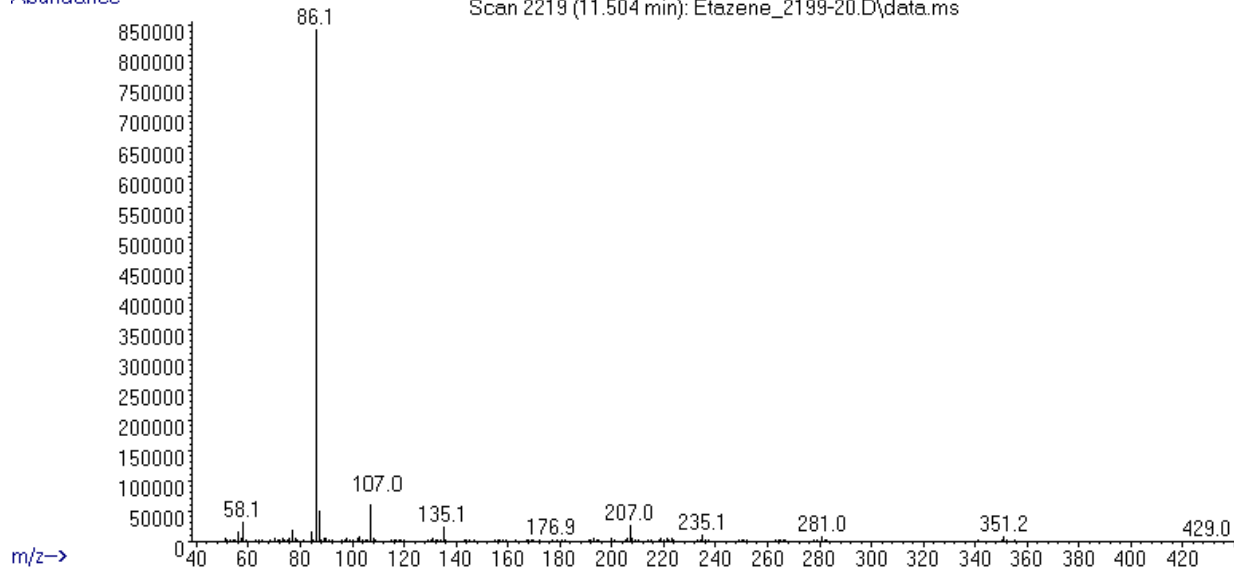
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 11,5 BP(1): 86; BP(2): 107,BP(3) :87,
HPLC-TOF	+	Exact mass (theoretical): 351,2311; measured value Δppm:-0,86; formula:C22H29N3O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

# ANALYTICAL RESULTS

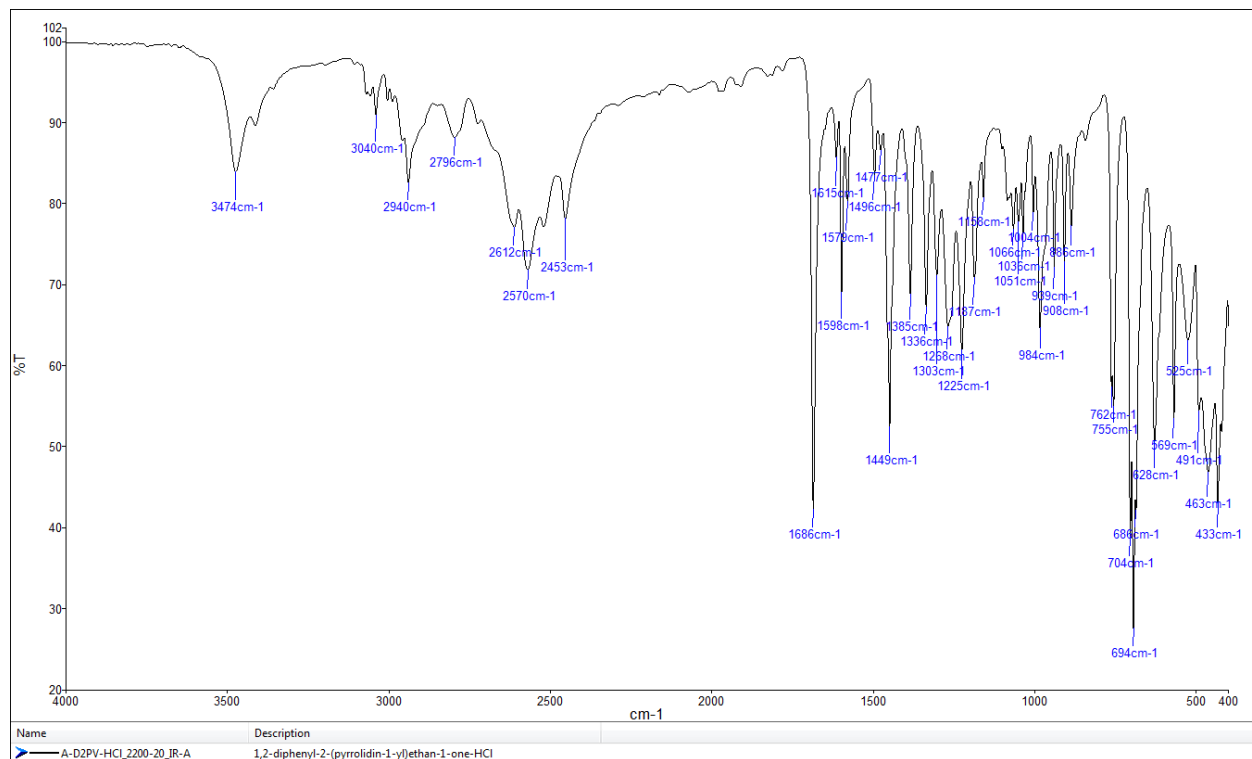
MS (EI)

Abundance

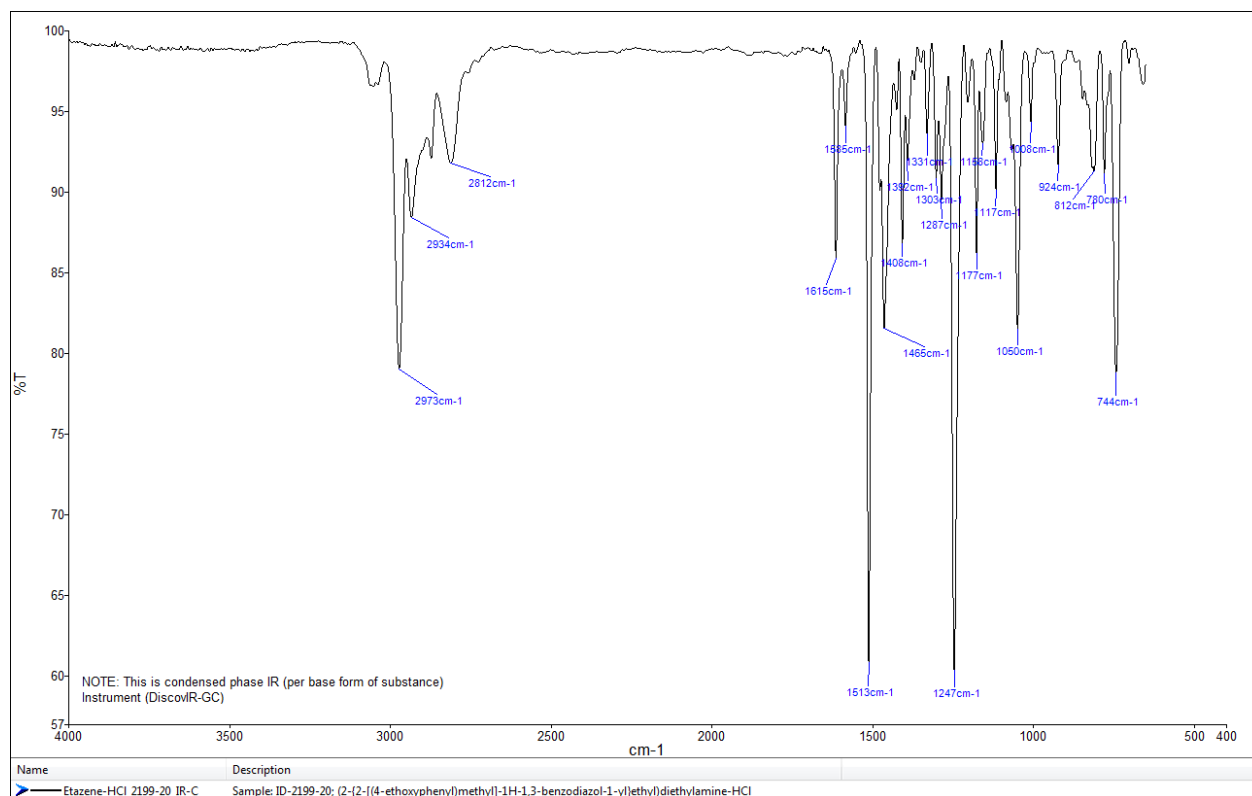
Scan 2219 (11.504 min): Etazene\_2199-20.D\data.ms



## FTIR-ATR - direct measurement (sample as received)



## IR (solid phase – after chromatographic separation)



# TOF REPORT

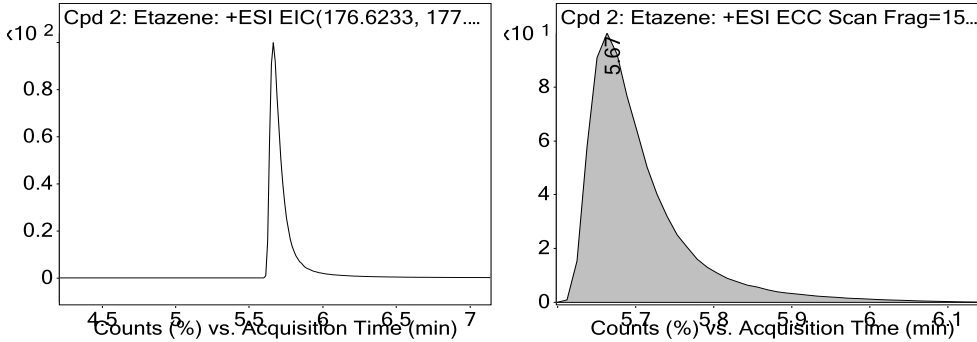
<b>Data File</b>	Etazene_2199_20.d	<b>Sample Name</b>	ID-2199-20
<b>Sample Type</b>	Sample	<b>Position</b>	P1-A4
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	general-15_01_2020-XDB-C18-ESI+.m	<b>Acquired Time</b>	11/13/2020 9:05:45 AM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	a-Drugs_NFL.m
<b>Comment</b>	MeOH		

## Compound Table

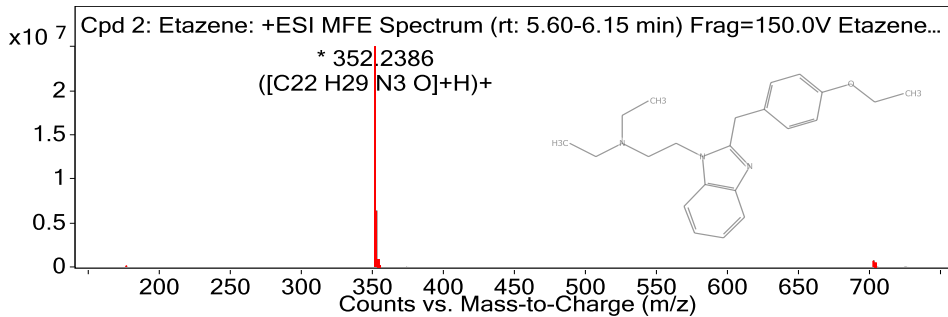
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: Etazene	Etazene	C22 H29 N3 O	5.67	351.2314

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Etazene	352.2386	5.67	351.2314	5.67	C22 H29 N3 O	351.2311	-0.86

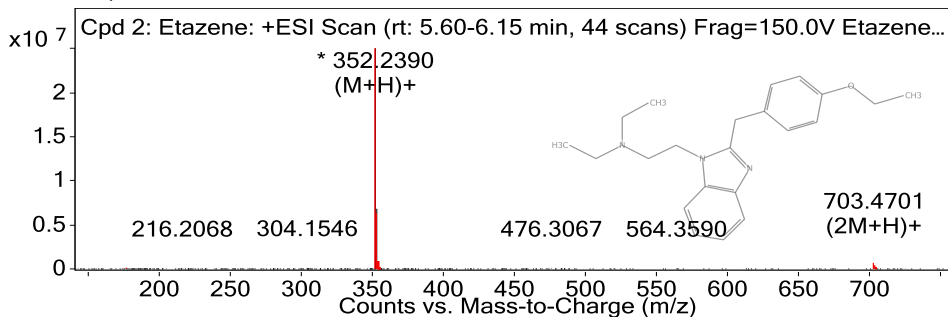
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
176.6232	2	76966.54	C22 H29 N3 O	(M+2H)+2
352.2386	1	25025364	C22 H29 N3 O	(M+H)+
353.2419	1	6401916.8	C22 H29 N3 O	(M+H)+
354.2455	1	813282.64	C22 H29 N3 O	(M+H)+
355.2477	1	62019.66	C22 H29 N3 O	(M+H)+
374.2204	1	21049.28	C22 H29 N3 O	(M+Na)+
703.4699	1	748285.13	C22 H29 N3 O	(2M+H)+
704.4728	1	367293	C22 H29 N3 O	(2M+H)+
705.4756	1	92330.67	C22 H29 N3 O	(2M+H)+
725.4504	1	38669.47	C22 H29 N3 O	(2M+Na)+

--- End Of Report ---

### Peak Integration Report

Sample Name:	2199_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	Admin
Inj. Date / Time:	16-Nov-2020 / 17:45	Run Time:	43,00

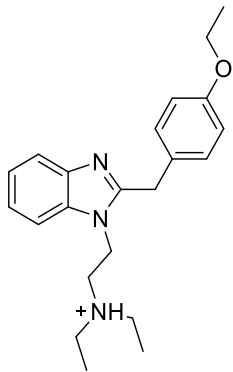
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height $\mu\text{S}$	Amount mg/L
1	8,61	Chloride	BMB	85,366	354,506	n.a.
TOTAL:				85,37	354,51	0,0



University  
of Ljubljana  
Faculty of Chemistry  
and Chemical Technology

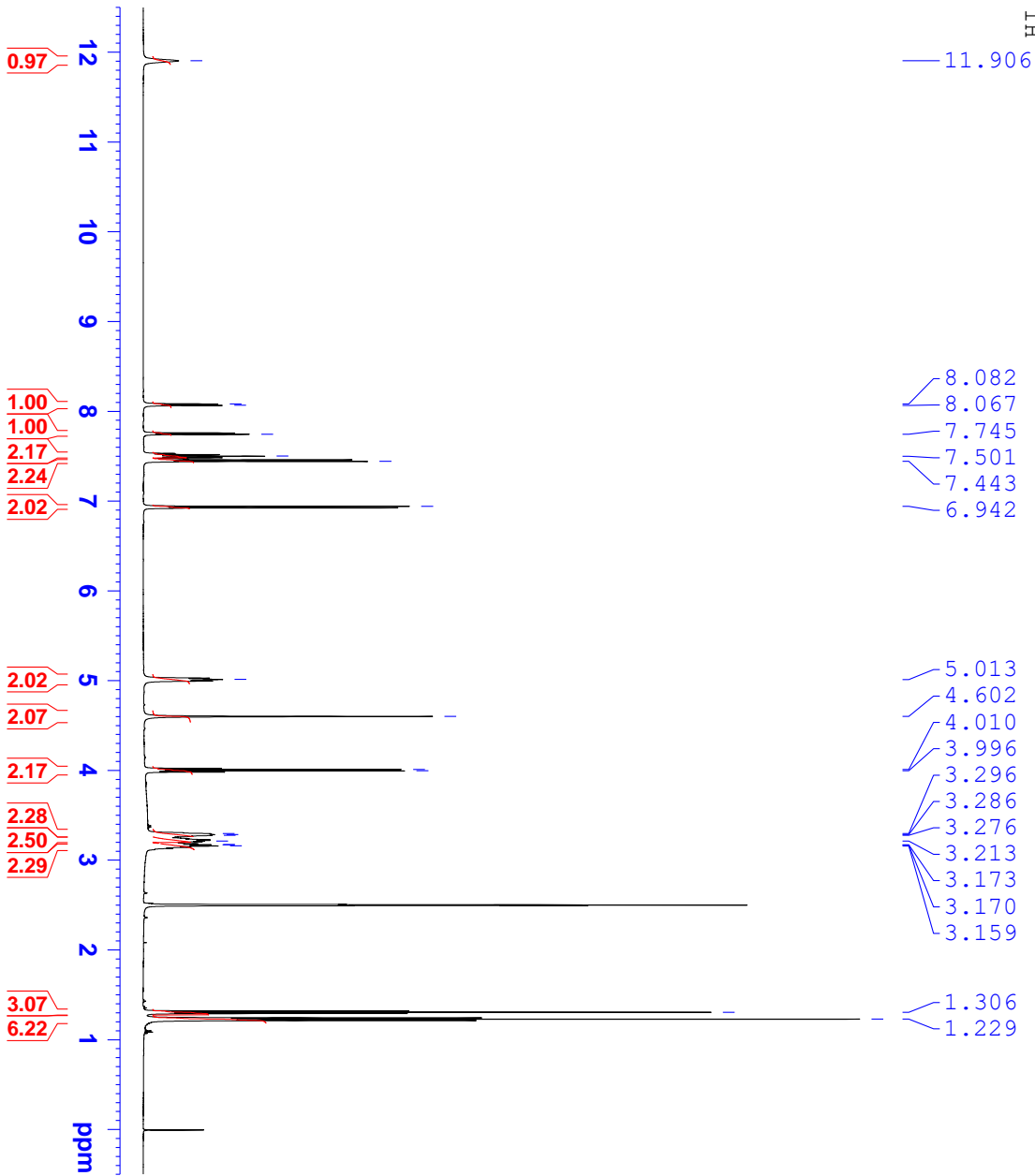


## R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	<b>2199-20</b>
Received date:	November 19, 2020
Our notebook code:	NFL-2199-20
NMR sample preparation:	18.4 mg dissolved in 0.7 mL DMSO- <i>d</i> <sub>6</sub>
NMR experiments:	<sup>1</sup> H, <sup>13</sup> C, <sup>1</sup> H- <sup>1</sup> H <i>gs</i> -COSY, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HSQC, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HMBC, <sup>1</sup> H- <sup>15</sup> N <i>gs</i> -HMBC
Proposed structure with formula, exact mass, molecular weight:	 <p>Chemical Formula: C<sub>22</sub>H<sub>30</sub>N<sub>3</sub>O<sup>+</sup> Exact Mass: 352,2383 Molecular Weight: 352,5015</p>
Chemical name:	<i>N</i> -protonated 2-(2-(4-ethoxybenzyl)-1 <i>H</i> -benzo[ <i>d</i> ]imidazol-1-yl)- <i>N,N</i> -diethylethan-1-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. ->98% purity of a sample based on <sup>1</sup> H NMR spectrum.
Supporting information:	Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra, <sup>1</sup> H and <sup>13</sup> C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	December 4, 2020



NFL-2199-20  
1H



Current Data Parameters  
 NAME NFL-2199-20  
 EXNO 1  
 PROCNO 1

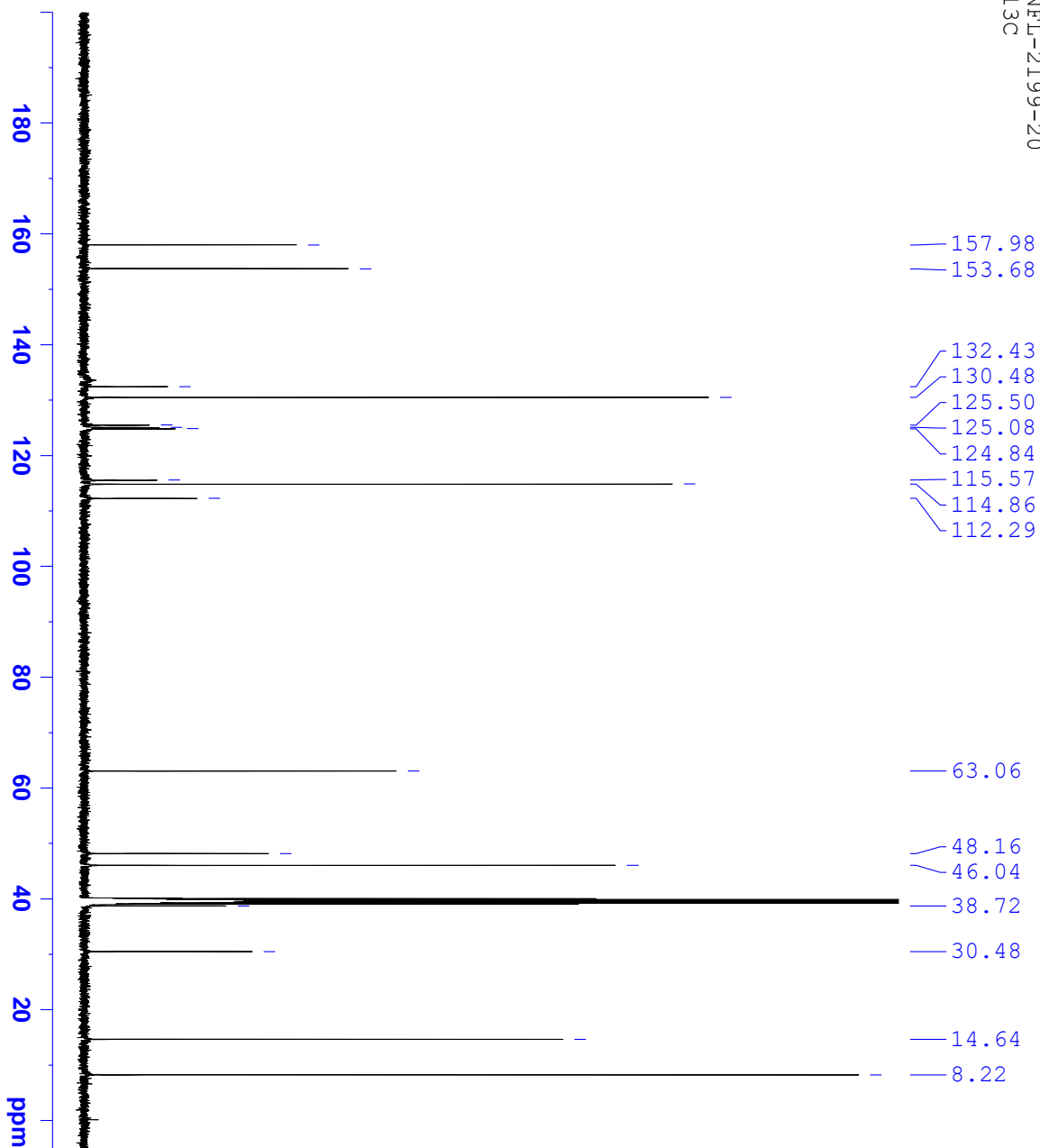
F2 - Acquisition Parameters  
 Date\_ 20201123  
 Time\_ 2.20

INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 32  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 80.6  
 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  
 PLW1 26.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 500.1300041 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

NFL-2199-20  
13C



Current Data Parameters  
NAME NFL-2199-20  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20201123  
Time 3.35

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

DS 4  
SMH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010048 sec  
RG 1820  
DM 16.800 usec  
DE 6.50 usec  
TE 296.0 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.70 usec  
PLW1 122.00000000 W

==== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG12 waltz16  
PCPD2 80.00 usec  
PLW2 26.00000000 W  
PLW12 0.30046001 W  
PLW13 0.15113001 W

F2 - Processing parameters  
SI 32768  
SF 125.7578470 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40