NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY
Vodovodna 95, 1000 LJUBLJANA, SLOVENIA
E: nfl@policija.si

EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

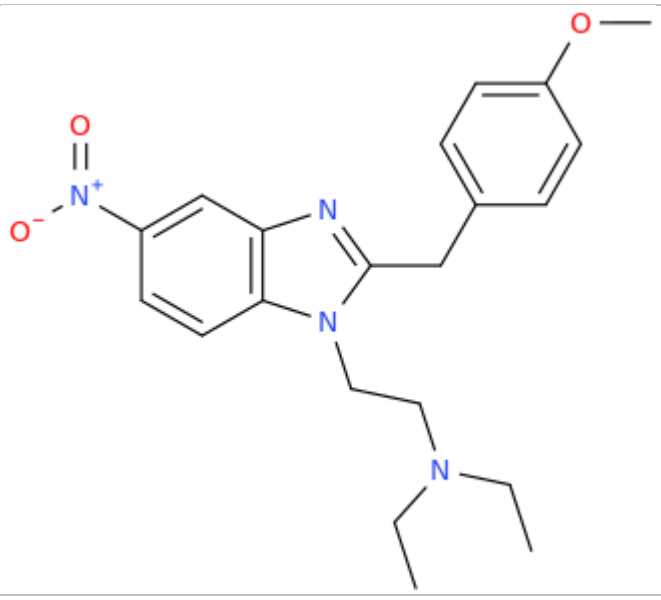
ANALYTICAL REPORT

Metonitazene (C21H26N4O3)

diethyl(2-{2-[(4-methoxyphenyl)methyl]-5-nitro-1H-1,3-benzodiazol-1-yl}ethyl)amine

Remark – other NPS detected:

Sample ID:	2198-20
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:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[1] (base form)	
Systematic name	diethyl(2-{2-[(4-methoxyphenyl)methyl]-5-nitro-1H-1,3-benzodiazol-1-yl}ethyl)amine
Other names	N,N-diethyl-2-[2-[(4-methoxyphenyl)methyl]-5-nitro-benzimidazol-1-yl]ethanamine
Formula (per base form)	C21H26N4O3
M_w (g/mol)	382,46
Salt form/anions detected	HCl
StdInChIKey (per base form)	HNGZTLMRQTVPBH-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	>97% purity of a sample based on 1H 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 µl and split mode (1:50). Injector temperature: 280 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 7.1 min, then 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 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 ^{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 (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

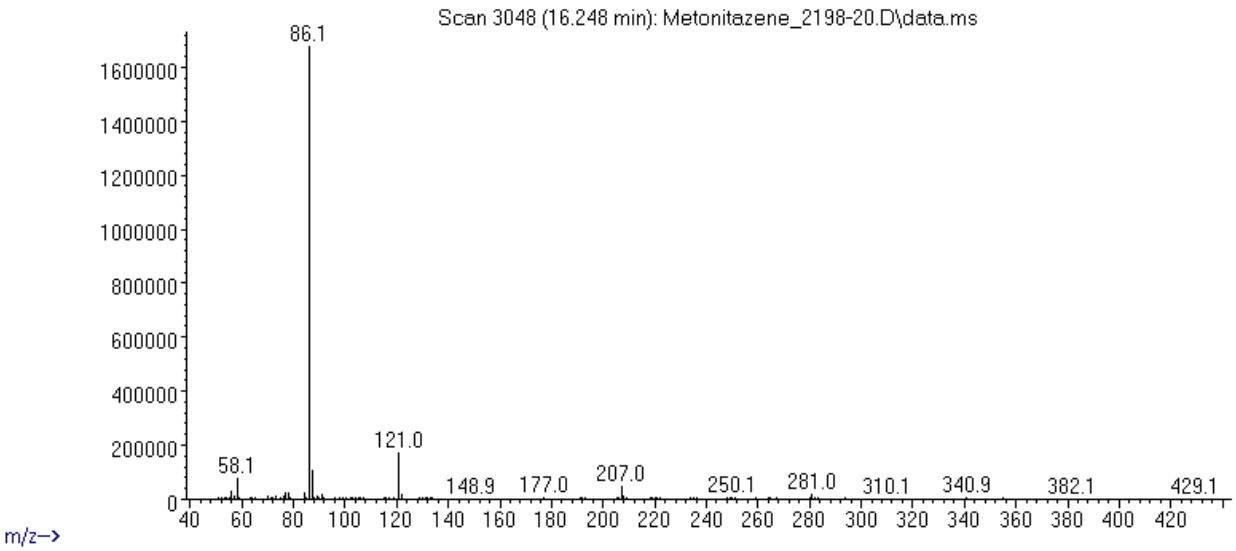
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	partially
H_2O	partially

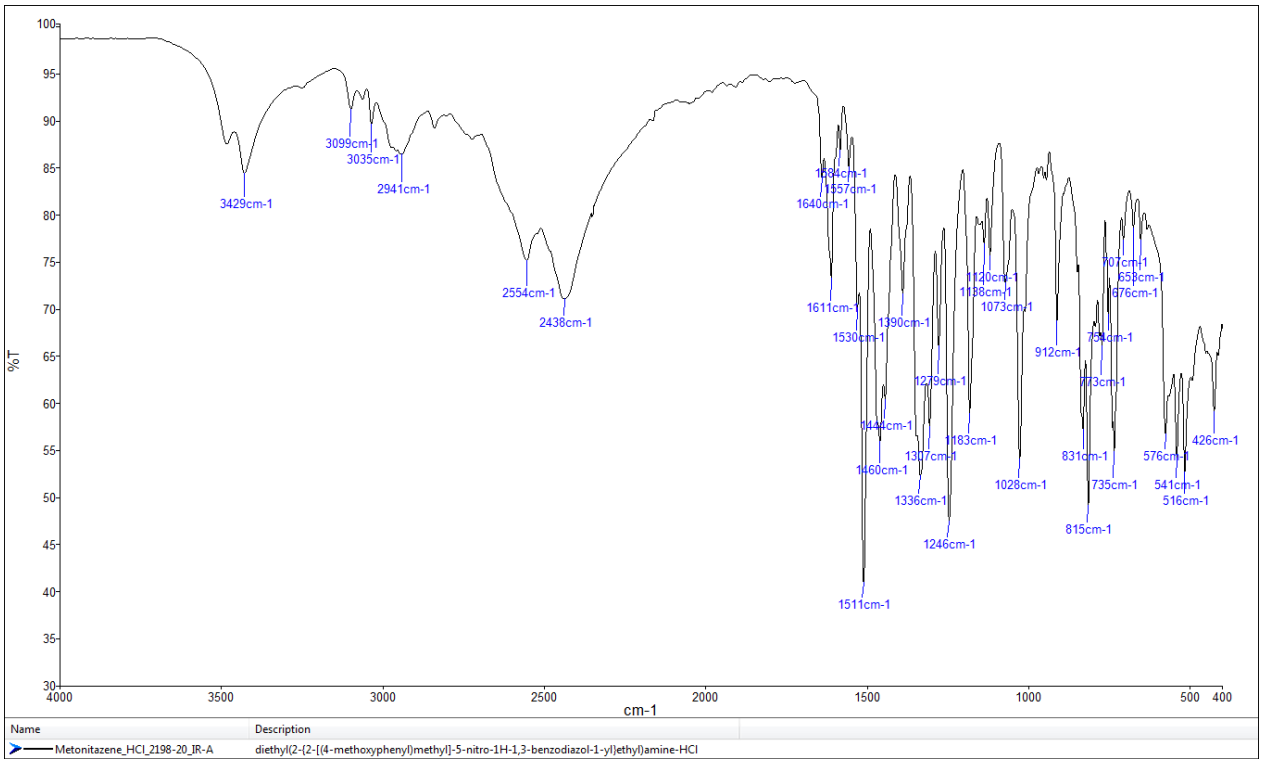
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 16,25 BP(1): 86; BP(2): 121,BP(3) :87,
HPLC-TOF	+	Exact mass (theoretical): 382,2005; measured value Δppm:-1,21; formula:C21H26N4O3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

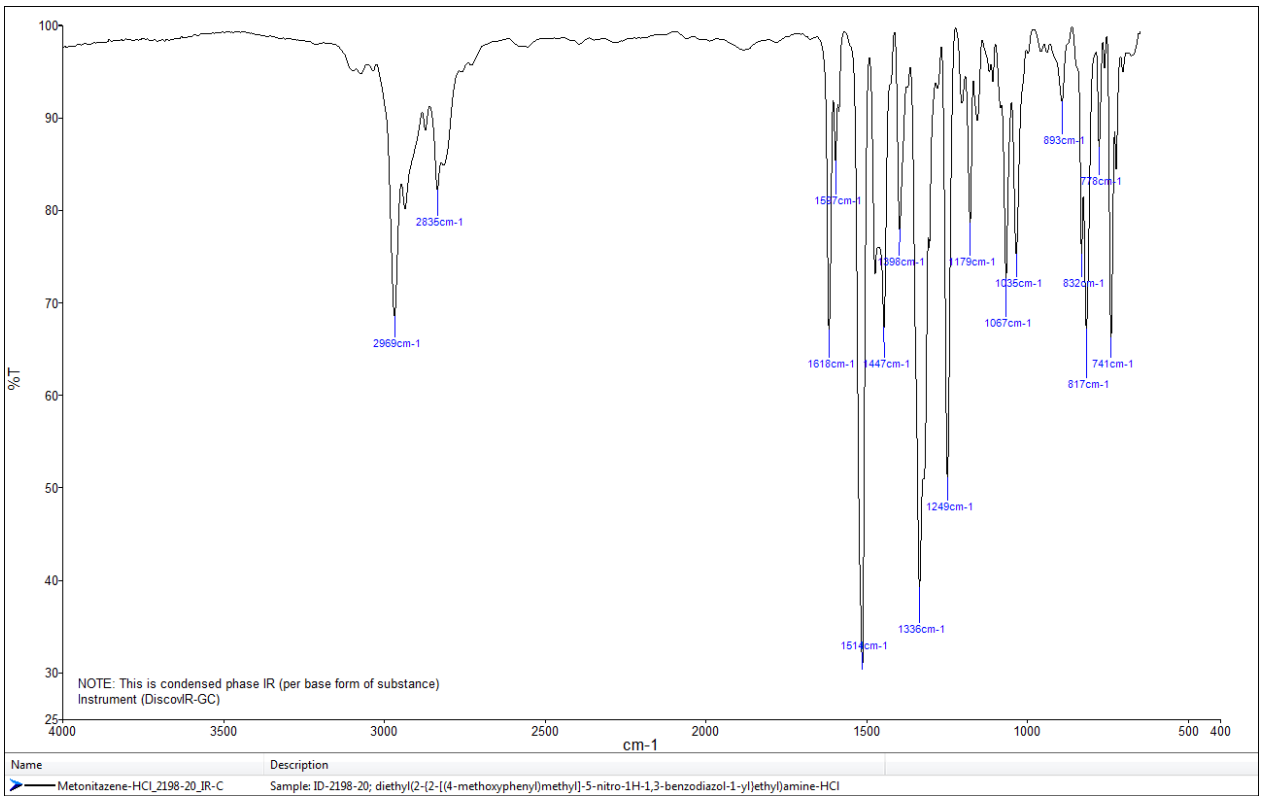
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

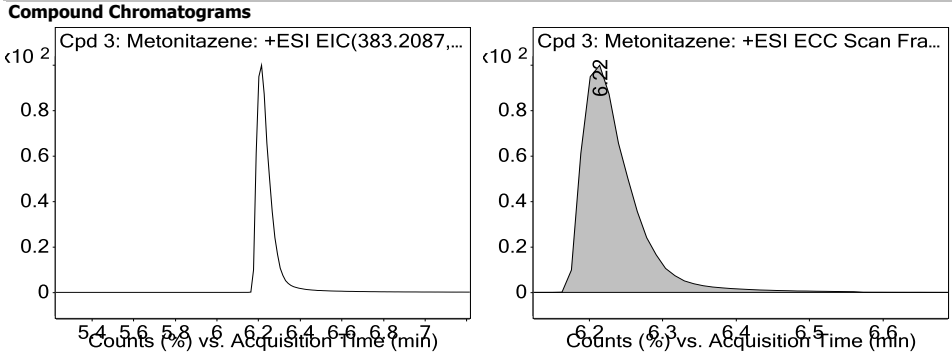
Data File	Metonitazene_2198_20.d	**Sample Name**	ID-2198-20
Sample Type	Sample	**Position**	P1-A3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	**Acquired Time**	11/13/2020 8:49:06 AM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

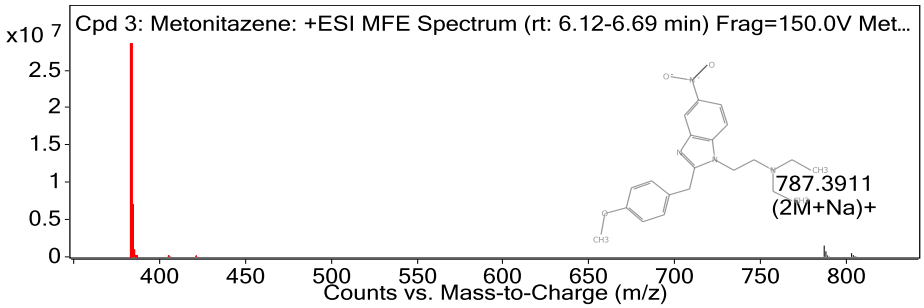
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 3: Metonitazene	Metonitazene	C21 H26 N4 O3	6.22	382.201

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Metonitazene	383.2082	6.22	382.201	6.22	C21 H26 N4 O3	382.2005	-1.21

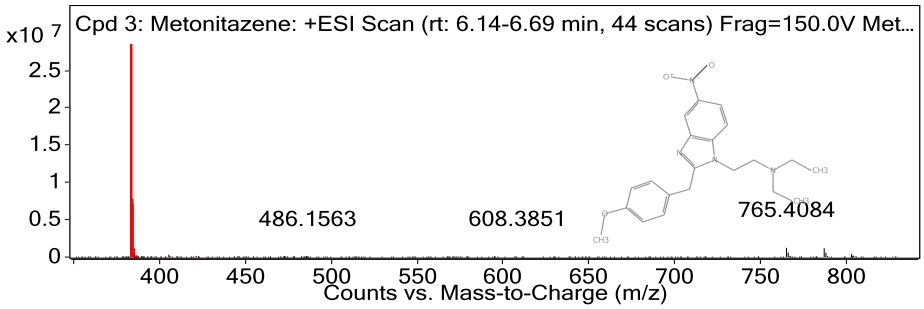
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

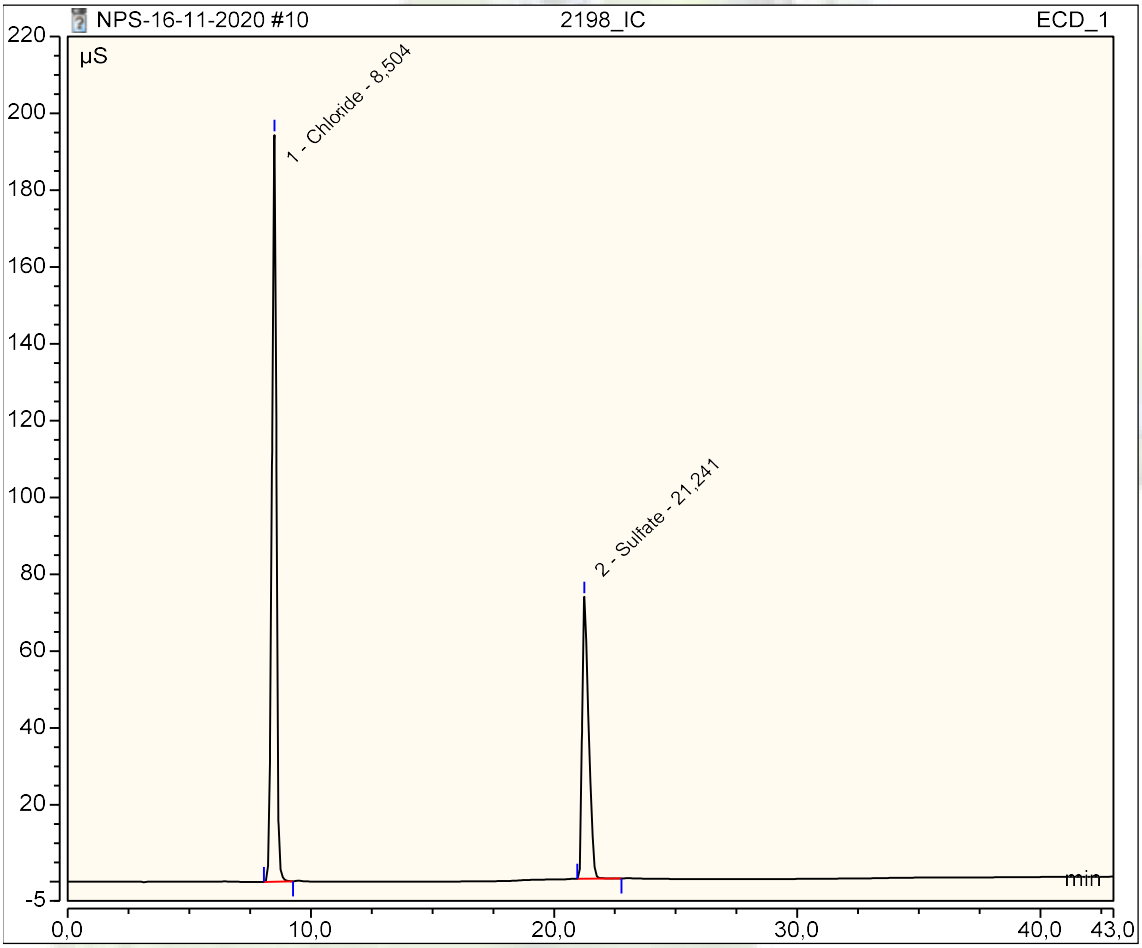
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
383.2082	1	28546910	C21 H26 N4 O3	(M+H)+
384.2112	1	7091395.8	C21 H26 N4 O3	(M+H)+
385.2146	1	999146.91	C21 H26 N4 O3	(M+H)+
386.217	1	97080.54	C21 H26 N4 O3	(M+H)+
405.1902	1	225291.98	C21 H26 N4 O3	(M+Na)+
787.3911	1	1515600.88		(2M+Na)+
788.3945	1	747517.72		(2M+Na)+
789.3966	1	190891.11		(2M+Na)+
803.365	1	489930.22		(2M+K)+
804.3677	1	232675.53		(2M+K)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**2198_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**Admin**
Inj. Date / Time:	**16-Nov-2020 / 16:55**	*Run Time:*	**43,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1	8,50	**Chloride**	BMB	41,370	194,345	n.a.
2	21,24	**Sulfate**	BMB	22,457	73,354	n.a.
TOTAL:				63,83	267,70	0,0

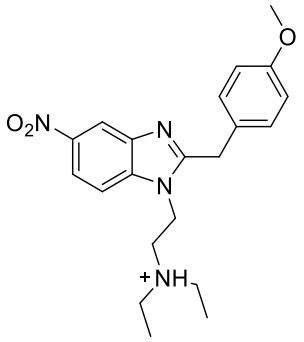


Večna pot 113
P. O. Box 537
SI-1001 Ljubljana
Slovenia
Phone: +386 1 479 8558
janez.kosmrlj@fkkt.uni-lj.si

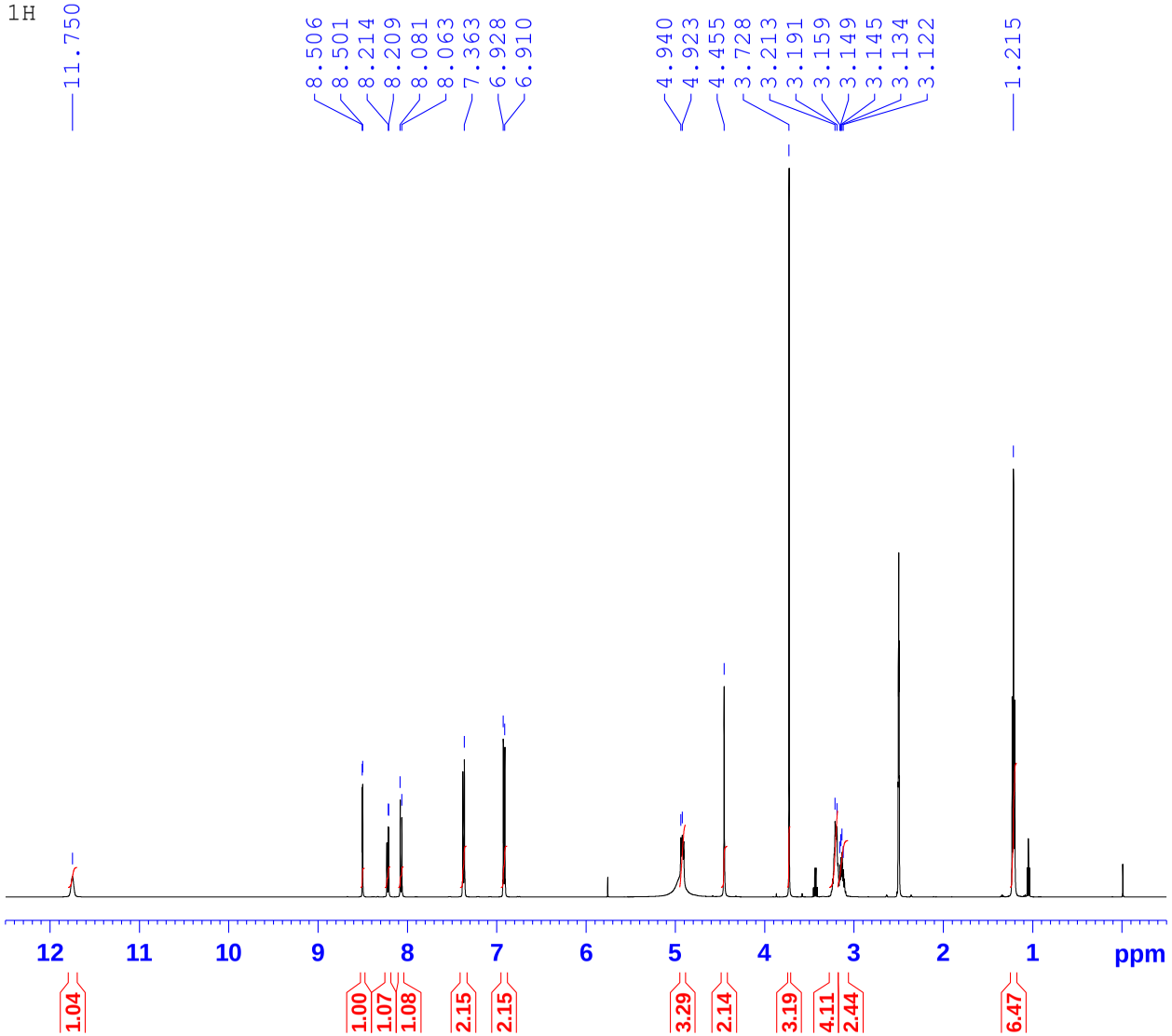
University
of Ljubljana

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:	**2198-20**
Received date:	November 19, 2020
Our notebook code:	NFL-2198-20
NMR sample preparation:	20.4 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 with formula, exact mass, molecular weight:	 Chemical Formula: $C_{21}H_{27}N_4O_3^+$ Exact Mass: 383,2078 Molecular Weight: 383,4715
Chemical name:	*N*-protonated *N*,*N*-diethyl-2-(2-(4-methoxybenzyl)-6-nitro-1*H*-benzo[*d*]imidazol-1-yl)ethan-1-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. ->97% 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 4, 2020

NFL-2198-20
1H

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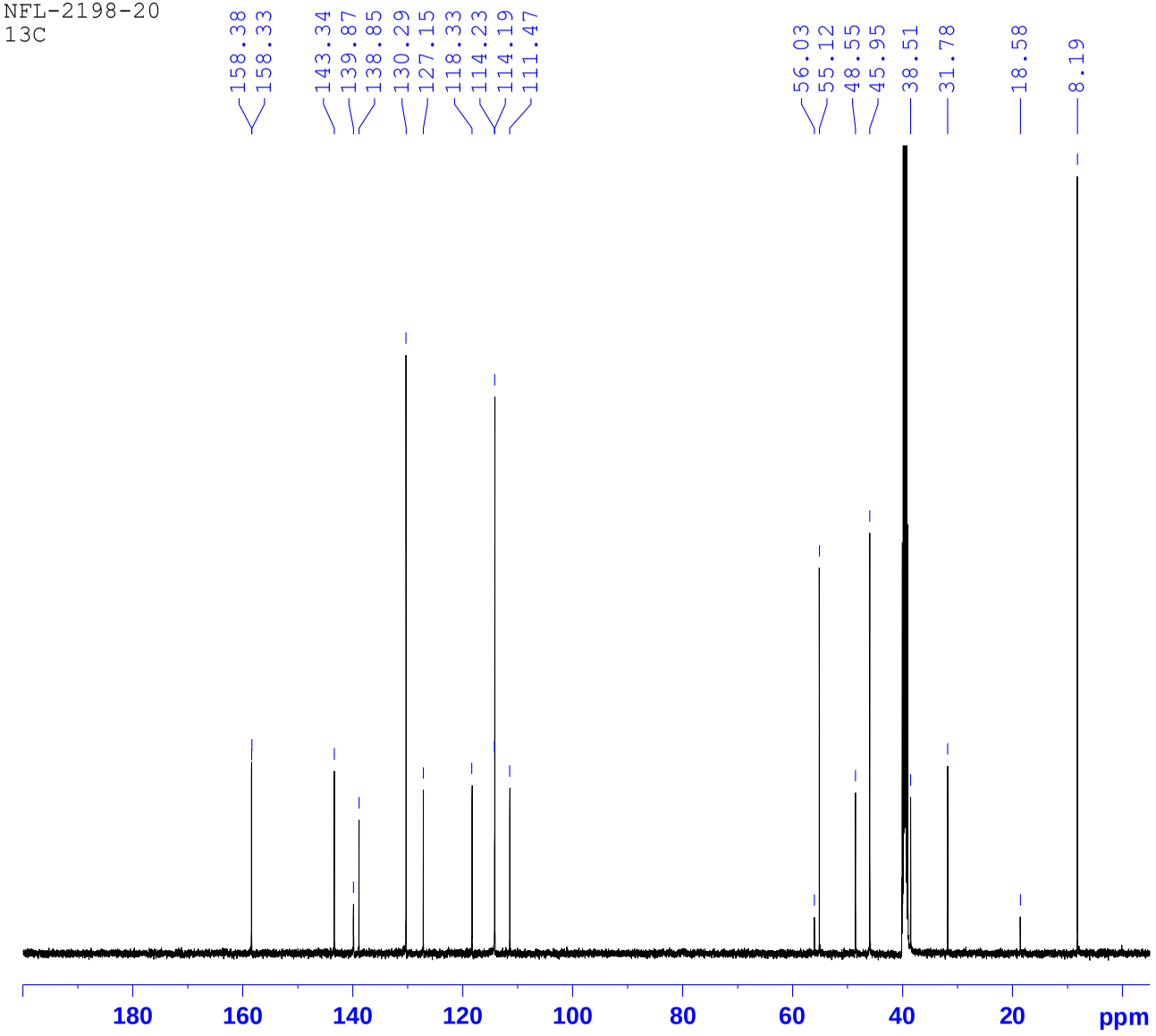
Current Data Parameters
NAME         NFL-2198-20
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201122
Time              21.01
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-2198-20
13C

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Current Data Parameters
NAME         NFL-2198-20
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201122
Time              22.16
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           1.00000000 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
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2        26.00000000 W
PLW12        0.30046001 W
PLW13        0.15113001 W

F2 - Processing parameters
SI                 32768
SF          125.7578452 MHz
WDW                EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```