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

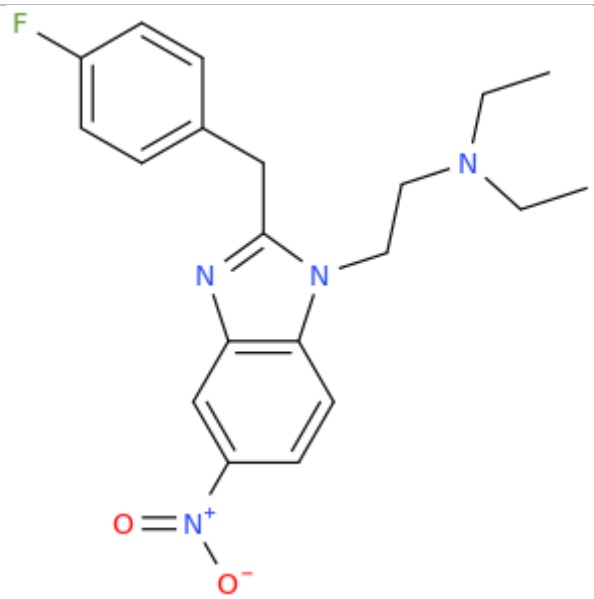
ANALYTICAL REPORT

Fluonitazene (C20H23FN402)

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

Remark – other NPS detected: none

Sample ID:	2184-20
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of entry (DD/MM/YYYY) into NFL database:	20/10/2020
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-fluorophenyl)methyl]-5-nitro-1H-1,3-benzodiazol-1-yl}ethyl)amine
Other names	N,N-diethyl-2-(2-(4-fluorobenzyl)-5-nitro-1H-benzo[d]imidazol-1-yl)ethan-1-amine
Formula (per base form)	C20H23FN402
M_w (g/mol)	370,43
Salt form/anions detected	HCl
StdInChIKey (per base form)	ZTWHIDCAGRMKTC-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>99% 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

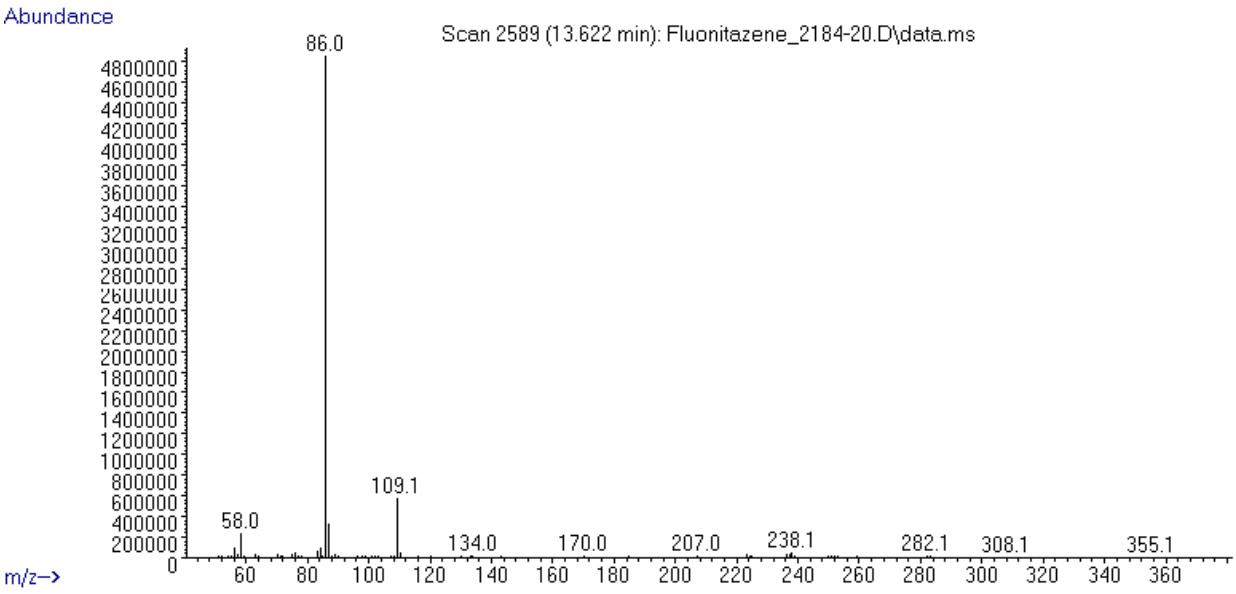
Supporting information

Solubility in	result/remark
CH_2Cl_2	not soluble
MeOH	soluble
H_2O	partially

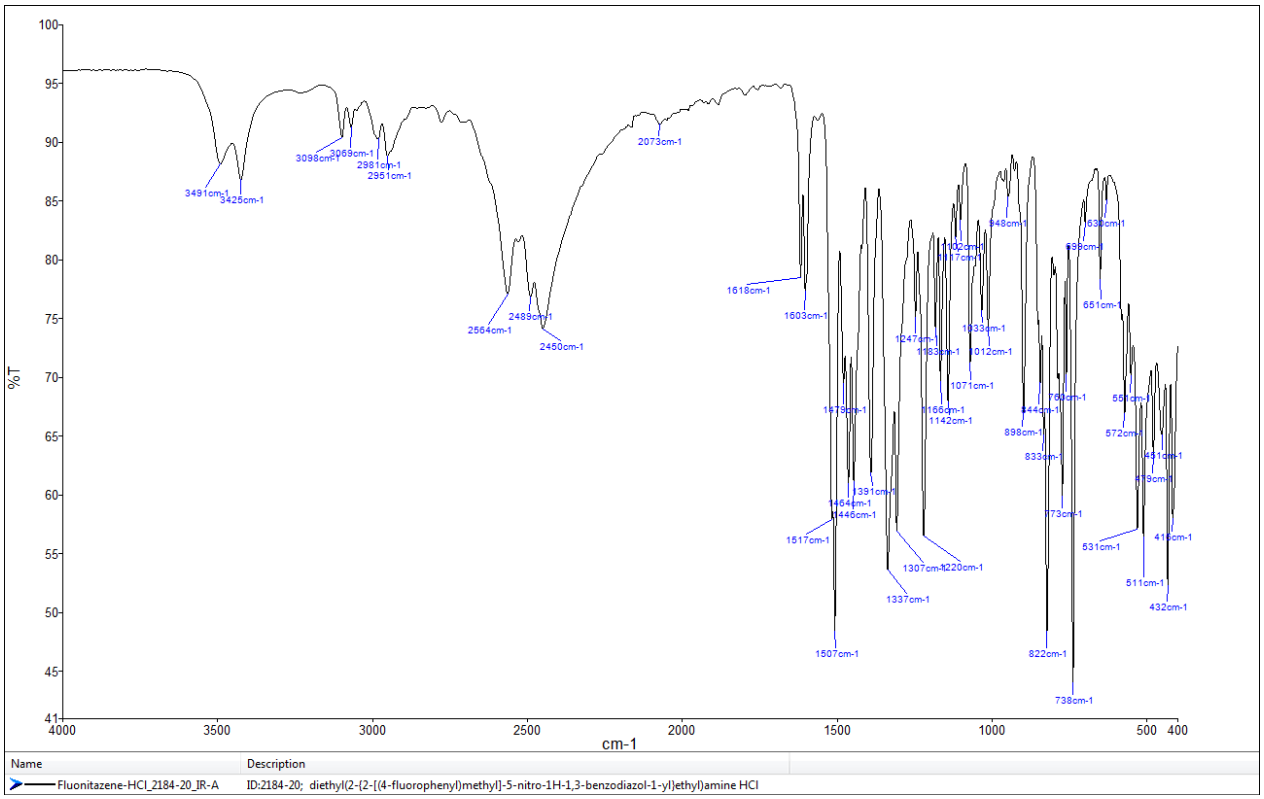
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,62 BP(1): 86; BP(2): 109,BP(3) :87,
HPLC-TOF	+	Exact mass (theoretical): 370,1805; measured value Δppm:-1,06; formula:C20H23FN402
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

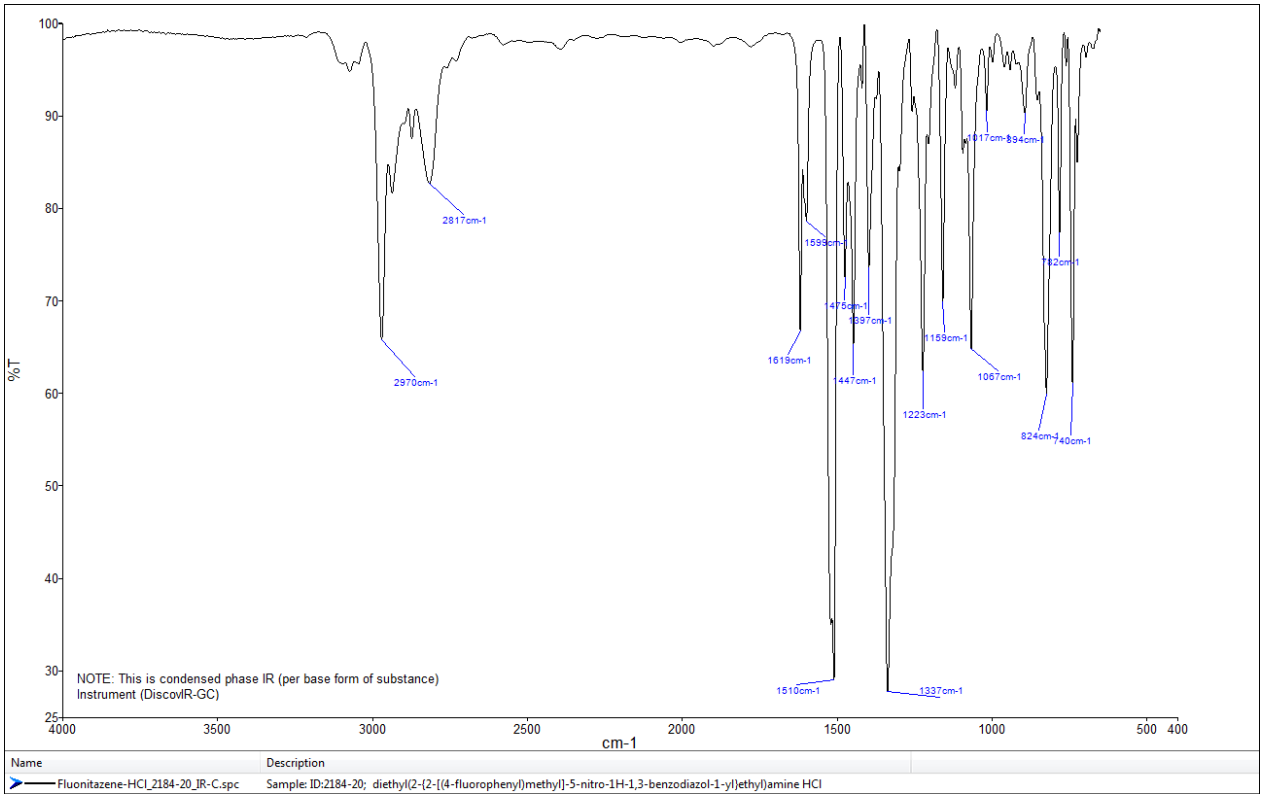
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

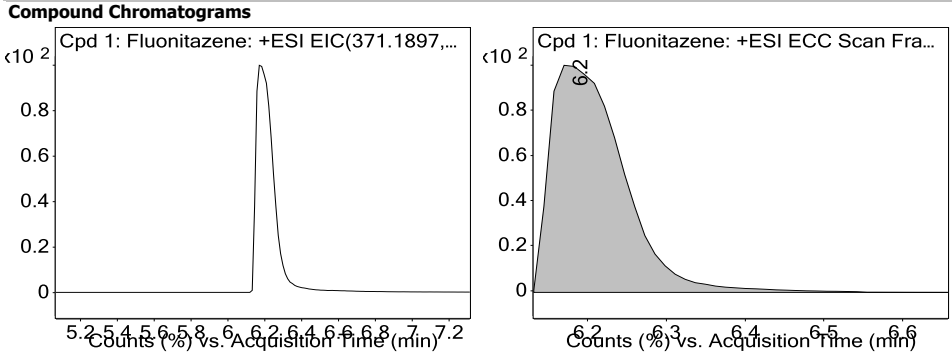
Data File	Fluonitazene_2184-20.d	**Sample Name**	ID-2184-20
Sample Type	Sample	**Position**	P2-A1
Instrument Name	6230B TOF LC-MS	**User Name**	
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	**Acquired Time**	9/2/2020 7:23:50 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment			

Compound Table

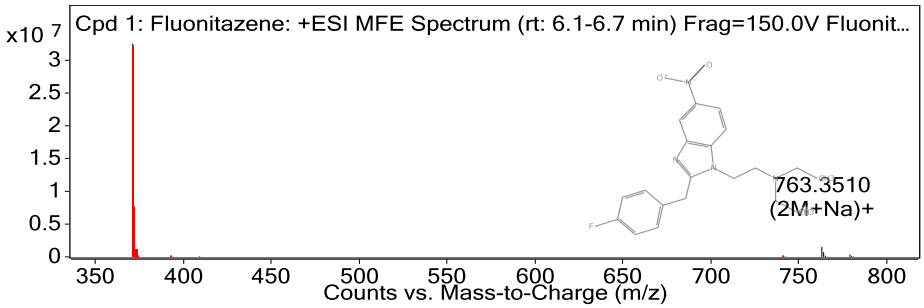
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Fluonitazene	Fluonitazene	C20 H23 F N4 O2	6.2	370.1809

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Fluonitazene	371.1883	6.2	370.1809	6.2	C20 H23 F N4 O2	370.1805	-1.06

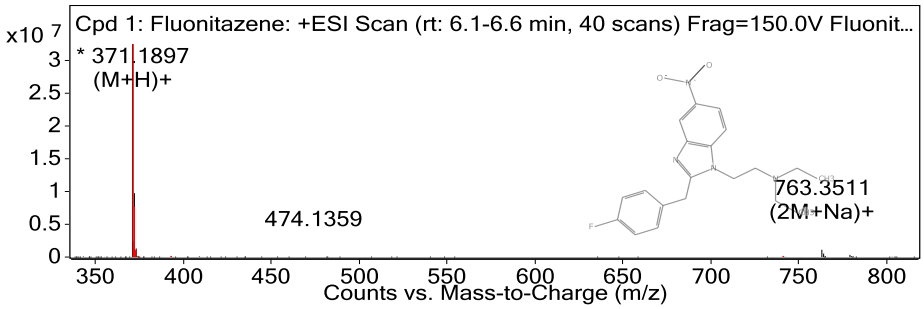
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

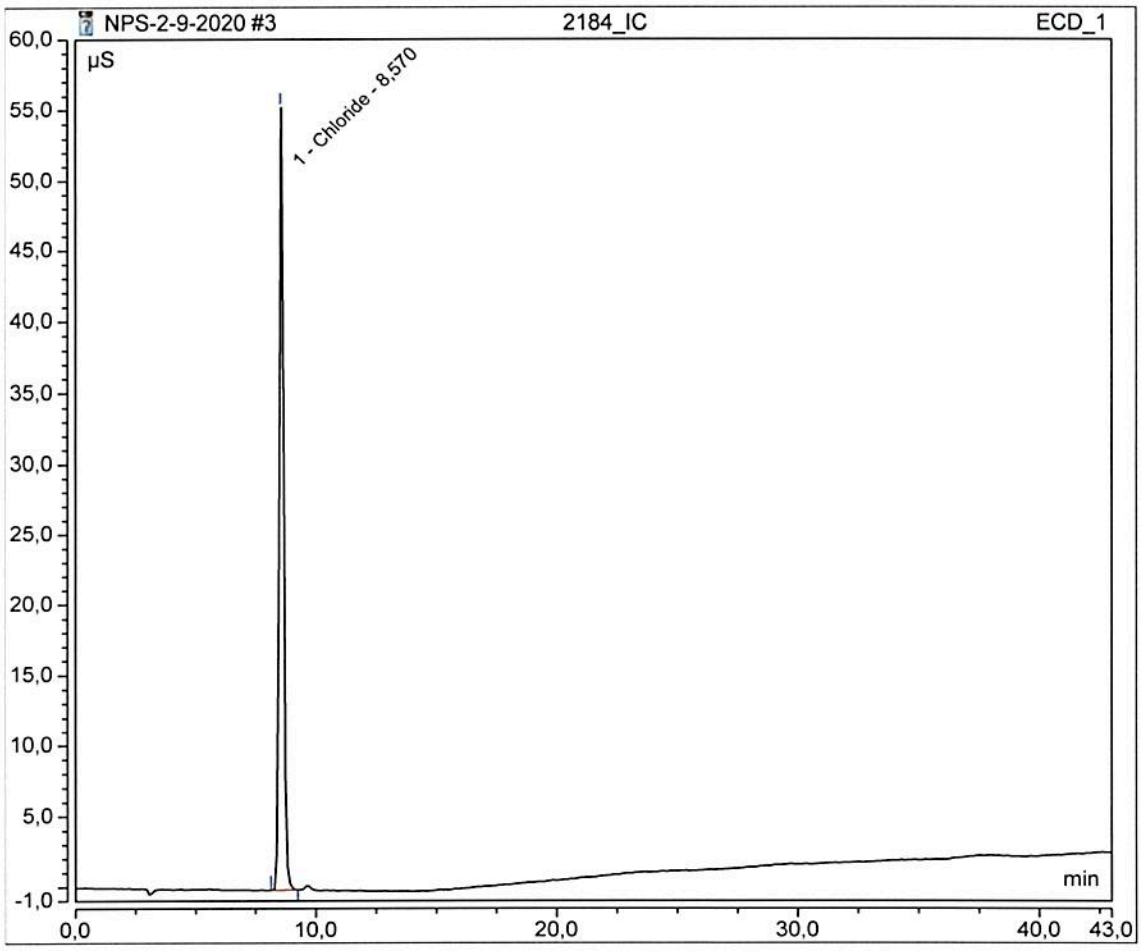
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
371.1883	1	32432198	C20 H23 F N4 O2	(M+H)+
372.1913	1	7481579.87	C20 H23 F N4 O2	(M+H)+
373.1943	1	944445.85	C20 H23 F N4 O2	(M+H)+
393.1701	1	157265.14	C20 H23 F N4 O2	(M+Na)+
741.3687	1	158068.19	C20 H23 F N4 O2	(2M+H)+
763.351	1	1522194.5		(2M+Na)+
764.3544	1	722812.11		(2M+Na)+
765.3567	1	172748.47		(2M+Na)+
779.3249	1	342313.5		(2M+K)+
780.3274	1	152397.25		(2M+K)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**2184_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**02-sep-2020 / 10:30**	*Run Time:*	**43,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1	8,57	Chloride	BMB	11,480	55,431	n.a.
		TOTAL:		11,48	55,43	0,0



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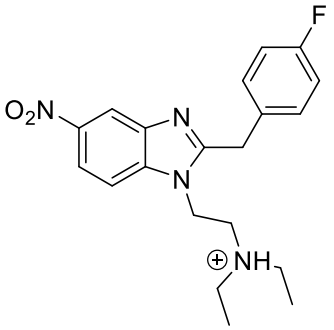
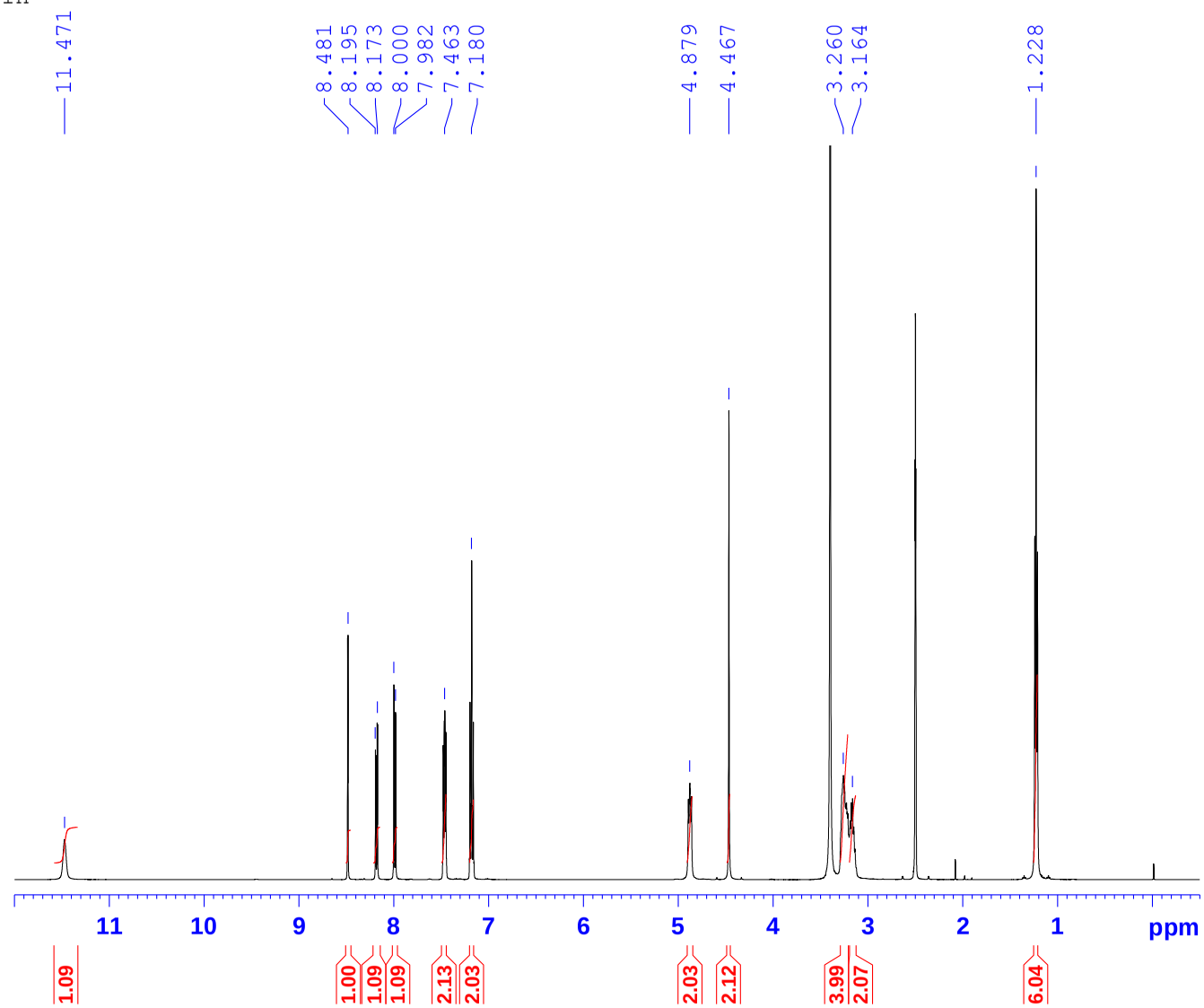
of Ljubljana

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:	**2184-20**
Received date:	September 10, 2020
Our notebook code:	NFL-2184-20
NMR sample preparation:	20.3 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, ^{19}F
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{20}H_{24}FN_4O_2^+$ Exact Mass: 371,1878 Molecular Weight: 371,4359
Chemical name:	*N*-protonated *N*,*N*-diethyl-2-(2-(4-fluorobenzyl)-5-nitro-1*H*-benzo[*d*]imidazol-1-yl)ethan-1-amine
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:	October 15, 2020

NFL-2184-20
1H

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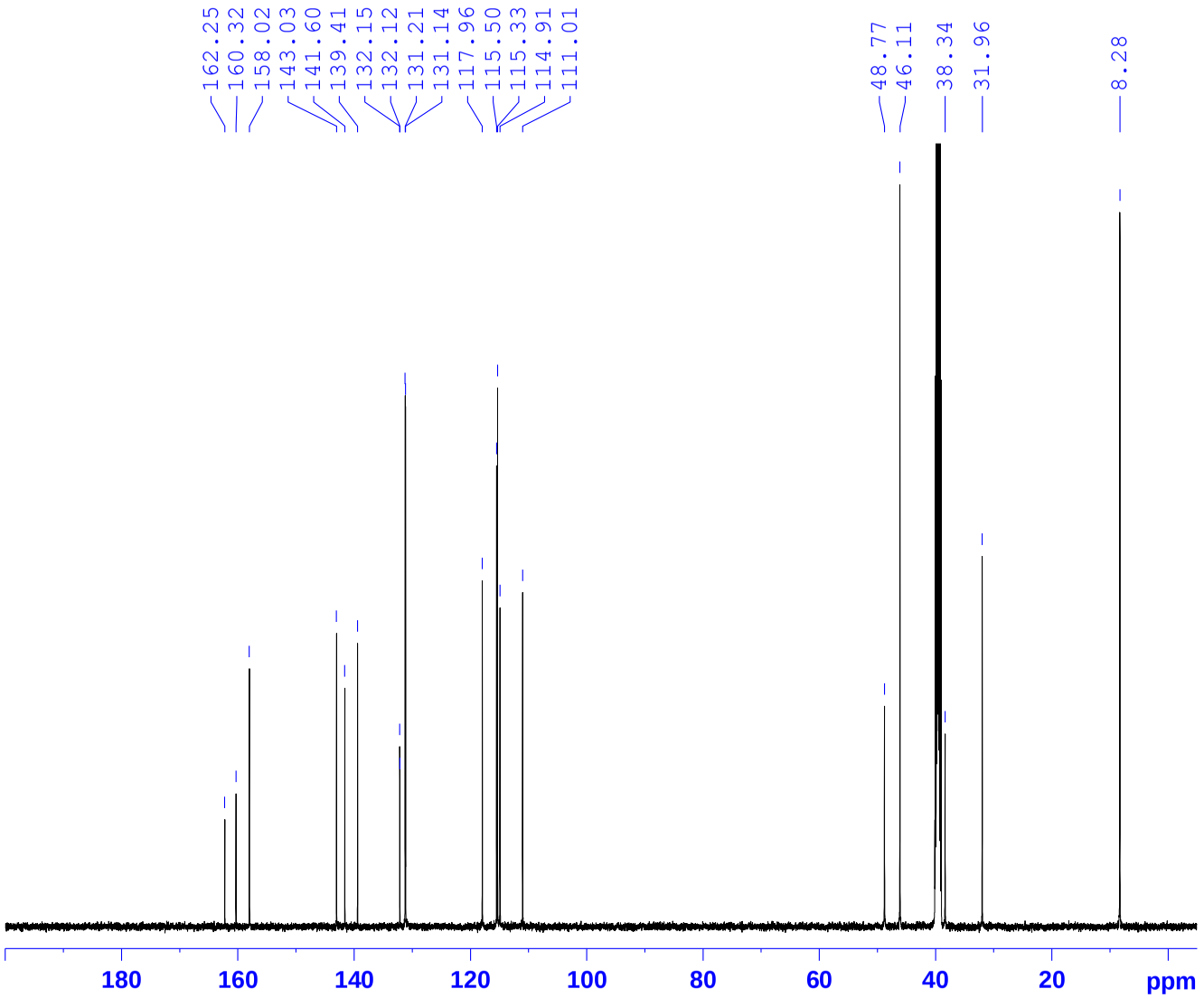
Current Data Parameters
NAME         NFL-2184-20
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200924
Time              23.35
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           500.1300048 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

NFL-2184-20
13C




```
Current Data Parameters
NAME         NFL-2184-20
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200925
Time                2.09
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                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           125.7578409 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```