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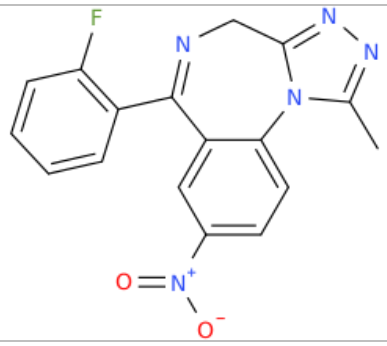
ANALYTICAL REPORT[1]

Flunitrazolam (C17H12FN5O2)

9-(2-fluorophenyl)-3-methyl-12-nitro-2,4,5,8-tetraazatricyclo[8.4.0.0^{2},^{6}]tetradeca-1(14),3,5,8,10,12-hexaene

Remark – other NPS detected: none

Sample ID:	1852-17
Sample description:	tablet - pink
Sample type:	collected /FSI Zurich, Switzerland
Date of sample receipt (M/D/Y):	10/11/2017
Date of entry (M/D/Y) into NFL database:	10/20/2017
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[2] (base form)	
Systematic name	9-(2-fluorophenyl)-3-methyl-12-nitro-2,4,5,8-tetraazatricyclo[8.4.0.0^{2},^{6}]tetradeca-1(14),3,5,8,10,12-hexaene
Other names	1-methyl-8-nitro-6-(2-fluorophenyl)-4H-[1,2,4]triazolo[4,3-a][1,4]benzodiazepine
Formula (per base form)	C17H12FN5O2
M_w (g/mol)	337,31
Salt form/anions detected	base
StdInChIKey (for base form)	RDLAGIOILLWVTM-UHFFFAOYSA-N
Other NPS detected	none
Add.info (purity..)	

[1] Acknowledgement: Sample was kindly provided by FSI Zurich, Switzerland. Masurements shown in this report were done in NFL.

[2] 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 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 ml 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 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

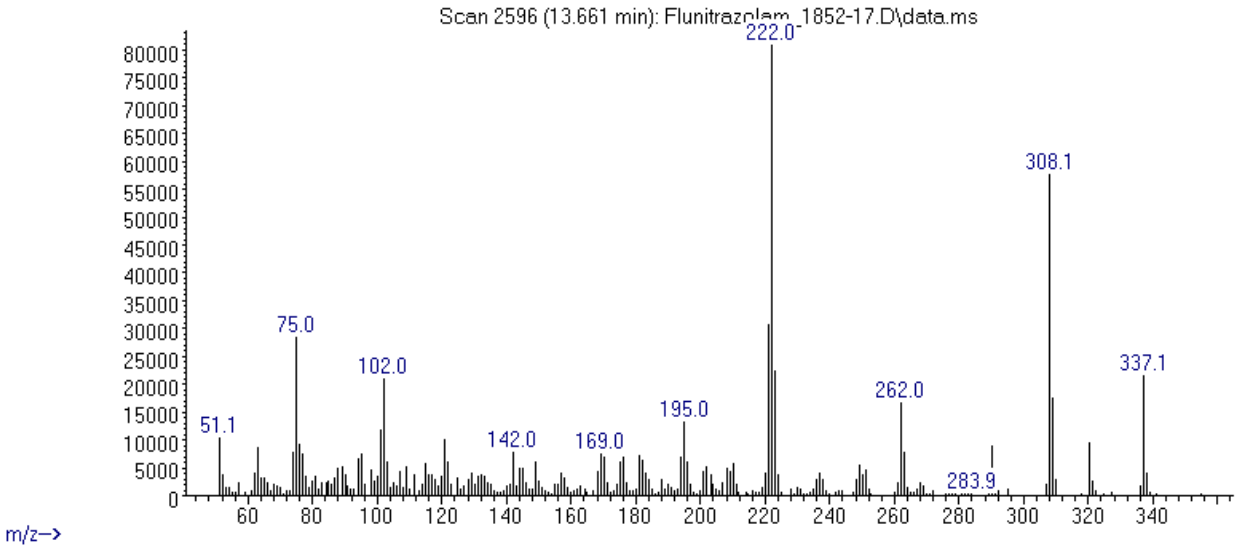
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	partially
H_2O	partially

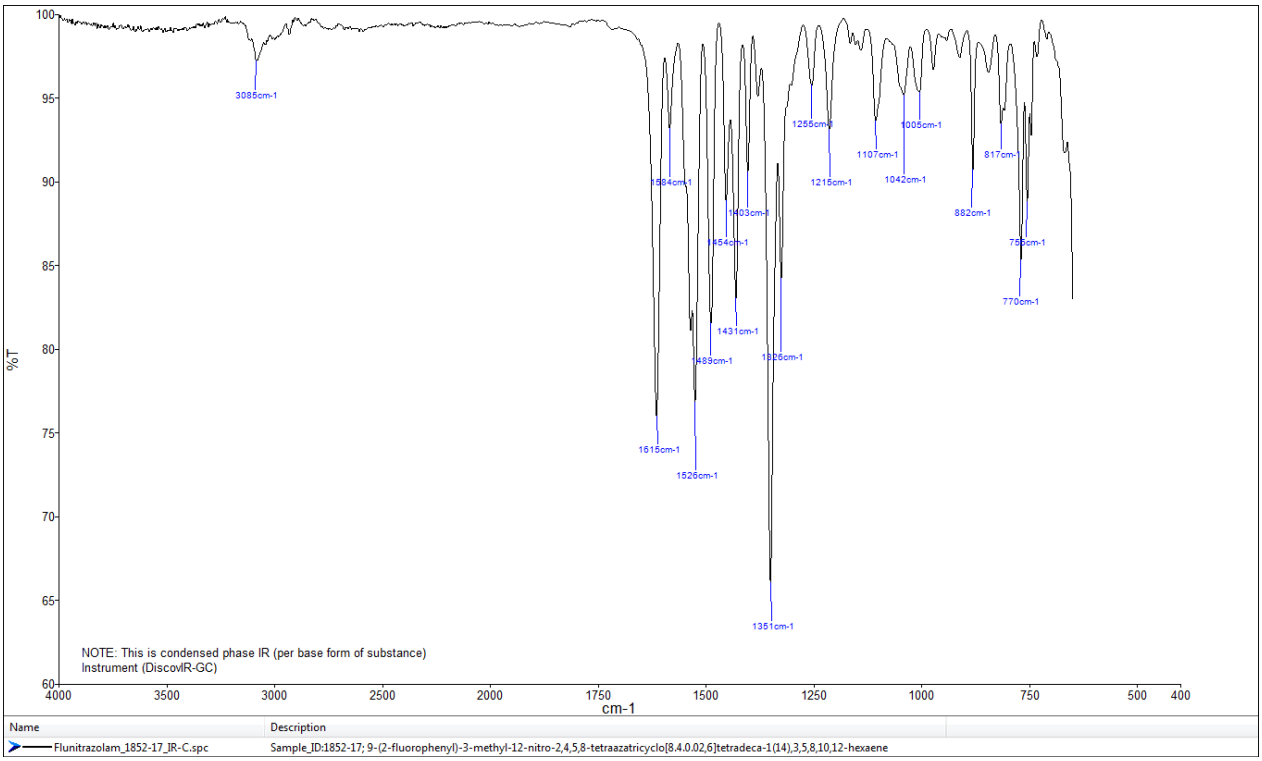
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,66 BP(1): 222; BP(2): 308,BP(3) :221,
HPLC-TOF	+	Exact mass (theoretical): 337,0975; measured value Δppm:-0,68; formula:C17H12FN5O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	-	
validation		
other		

ANALYTICAL RESULTS

MS (EI)



IR (condensed phase – after chromatographic separation)



TOF REPORT

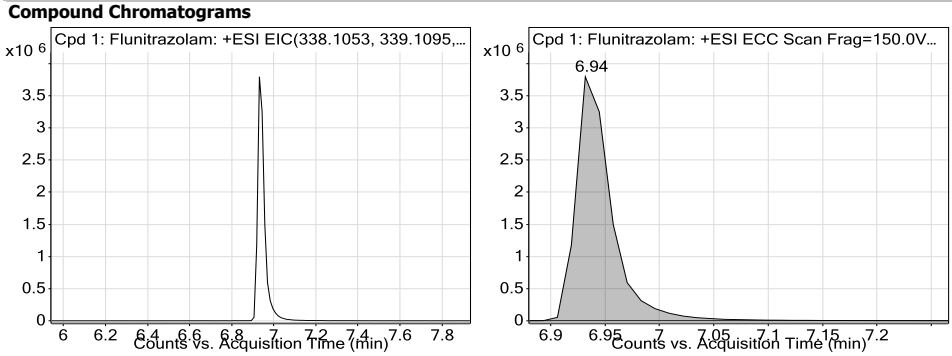
Data File	Flunitrazolam_1852-17.d	**Sample Name**	1852-17
Sample Type	Sample	**Position**	P1-A2
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-19_07_2017-XDB-C18-ESI-final.m	**Acquired Time**	10/17/2017 1:13:02 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	MeOH		

Compound Table

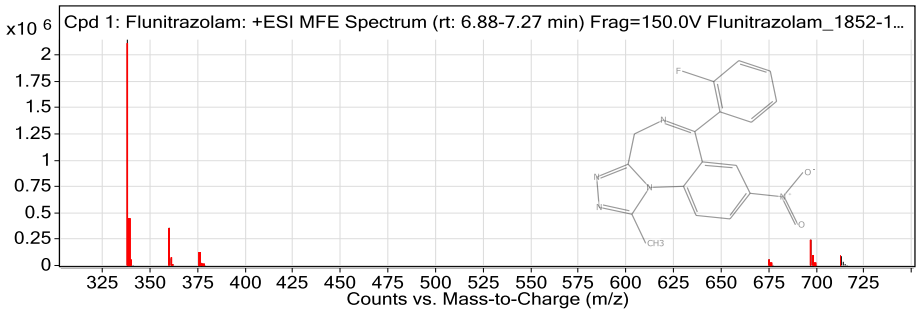
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Flunitrazolam	Flunitrazolam	C17 H12 F N5 O2	6.94	337.0977

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Flunitrazolam	338.1052	6.94	337.0977	6.94	C17 H12 F N5 O2	337.0975	-0.68

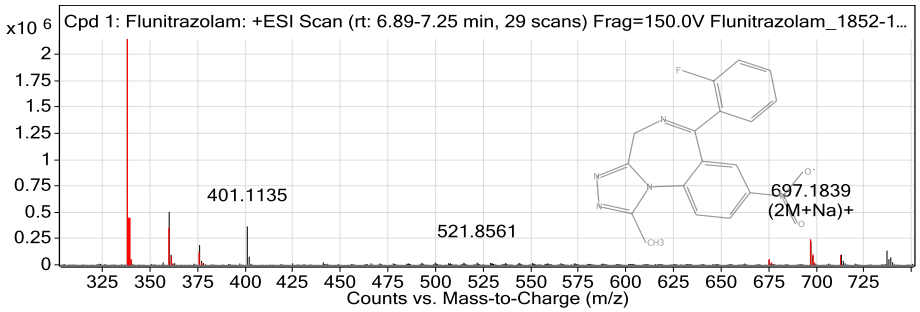
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
338.1052	1	2145349.75	C17 H12 F N5 O2	(M+H)+
339.1085	1	413179.87	C17 H12 F N5 O2	(M+H)+
340.1111	1	43317.56	C17 H12 F N5 O2	(M+H)+
360.0873	1	358489.5	C17 H12 F N5 O2	(M+Na)+
361.0899	1	67108.59	C17 H12 F N5 O2	(M+Na)+
376.0609	1	124971.88	C17 H12 F N5 O2	(M+K)+
675.2016	1	58614.44	C17 H12 F N5 O2	(2M+H)+
697.1841	1	245060.72	C17 H12 F N5 O2	(2M+Na)+
698.1867	1	94807.64	C17 H12 F N5 O2	(2M+Na)+
713.1576	1	88362.7	C17 H12 F N5 O2	(2M+K)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**1852-17**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**17-okt-2017 / 13:03**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount n.a.
		TOTAL:		0,00	0,00	0,00