



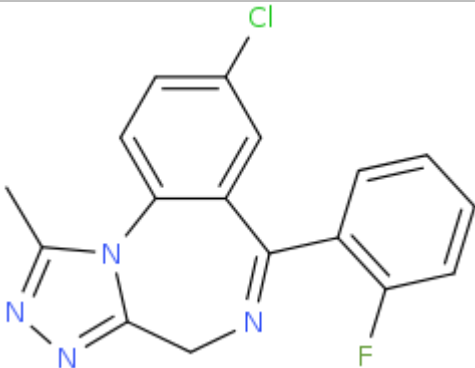
ANALYTICAL REPORT

Flualprazolam (C₁₇H₁₂ClFN₄)

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

Remark – other NPS detected:

Sample ID:	1903-18
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of sample receipt (M/D/Y):	1/3/2018
Date of entry (M/D/Y) into NFL database:	1/29/2018
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	12-chloro-9-(2-fluorophenyl)-3-methyl-2,4,5,8-tetraazatricyclo[8.4.0.0 ^{2,6}]tetradeca-1(14),3,5,8,10,12-hexaene
Other names	8-chloro-6-(2-fluorophenyl)-1-methyl-4H-benzo[f][1,2,4]triazolo[4,3-a][1,4]diazepine
Formula (per base form)	C ₁₇ H ₁₂ ClFN ₄
M _w (g/mol)	326,76
Salt form/anions detected	base
StdInChIKey (per base form)	MPZVLJCMGPYWQQ-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	>97% purity by 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 ml 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 ml 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 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

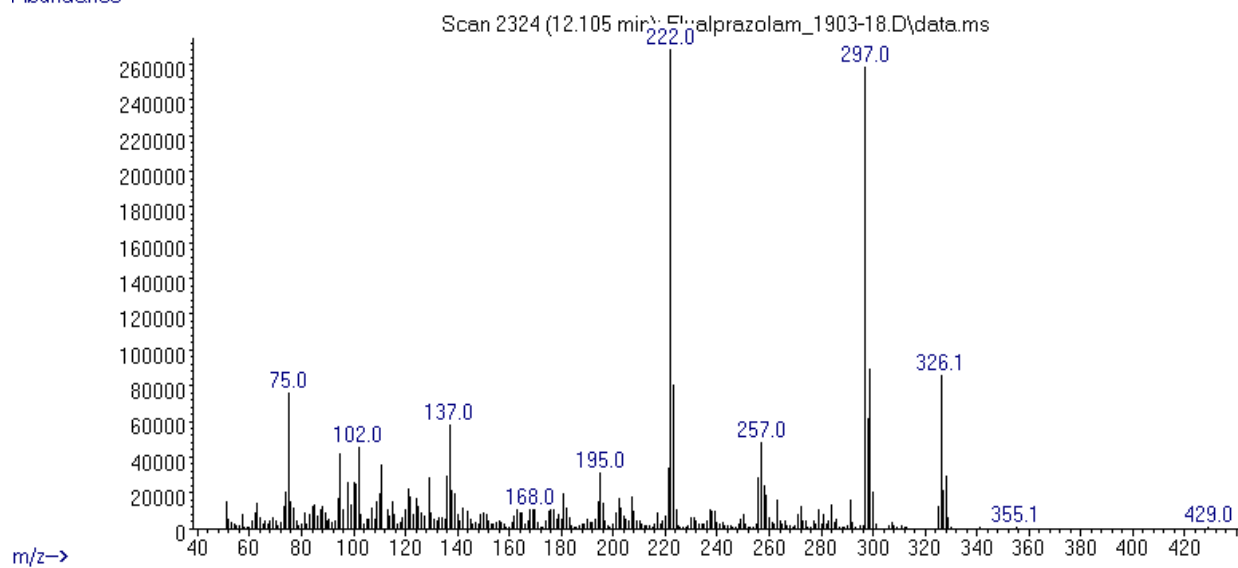
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 12,11 BP(1): 222; BP(2): 297,BP(3) :299,
HPLC-TOF	+	Exact mass (theoretical): 326,0735; measured value Δppm:0,61; formula:C17H12ClFN4
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

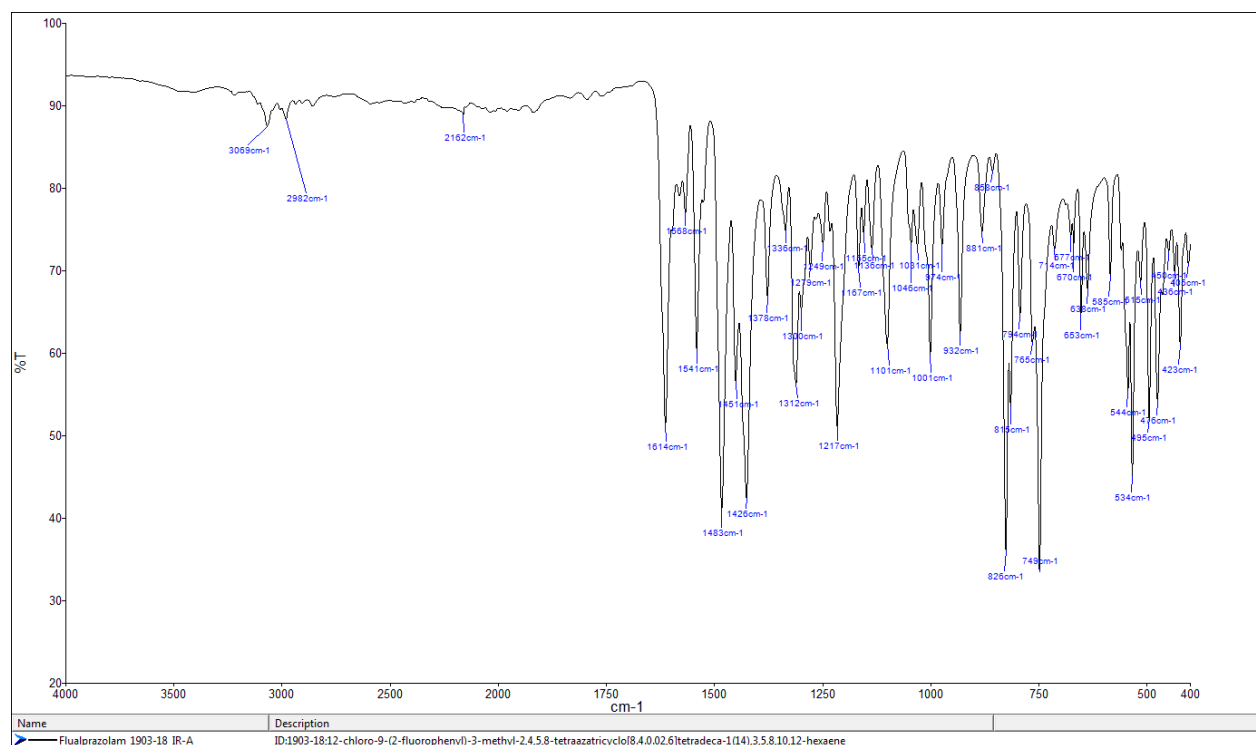
ANALYTICAL RESULTS

MS (EI)

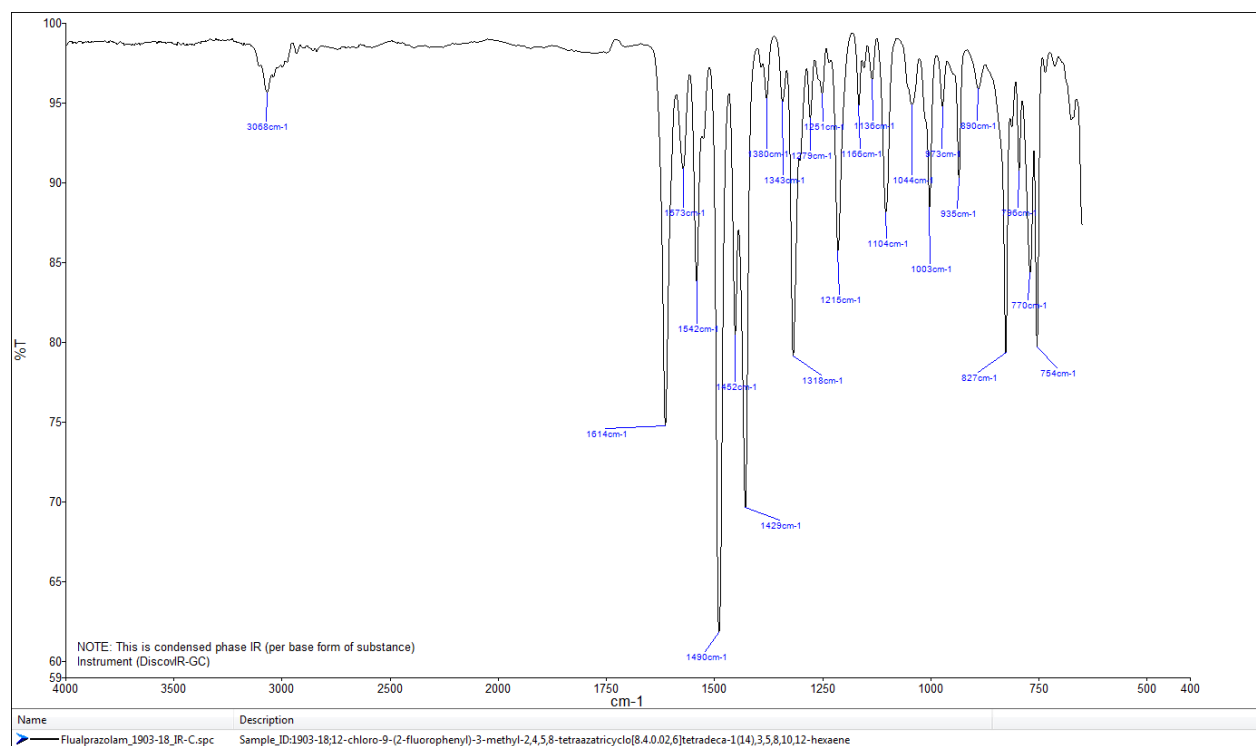
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

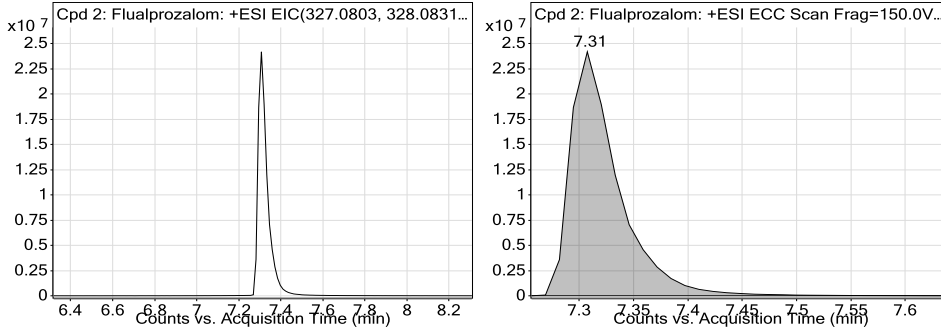
Data File	Flualprazolam_1903-18.d	Sample Name	1903-18
Sample Type	Sample	Position	P1-A6
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-04_12_2017-XDB-C18-ESI+.m	Acquired Time	1/4/2018 10:07:16 AM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	MeOH		

Compound Table

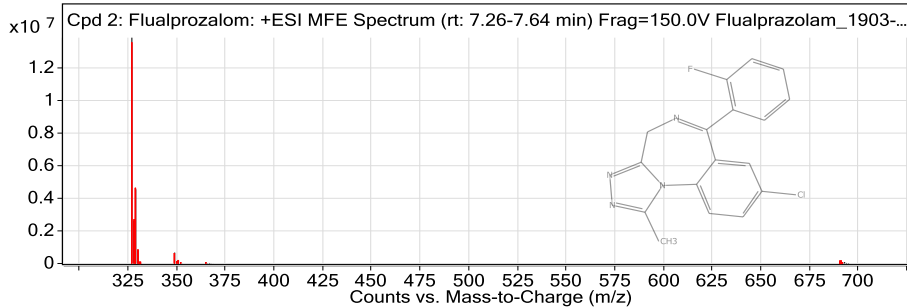
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: Flualprazolom	Flualprazolom	C17 H12 Cl F N4	7.31	326.0733

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Flualprazolom	327.0805	7.31	326.0733	7.31	C17 H12 Cl F N4	326.0735	0.61

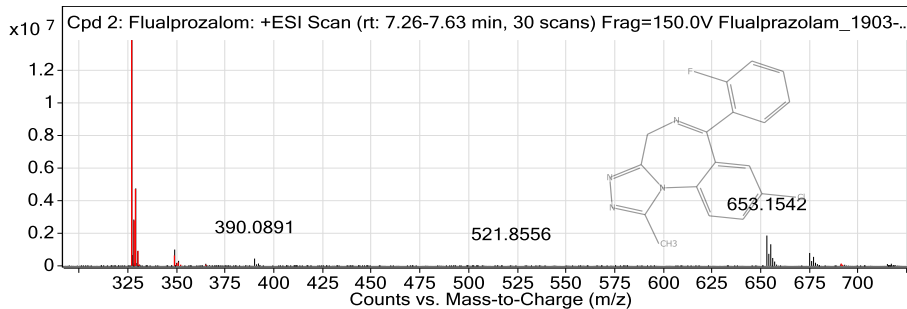
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

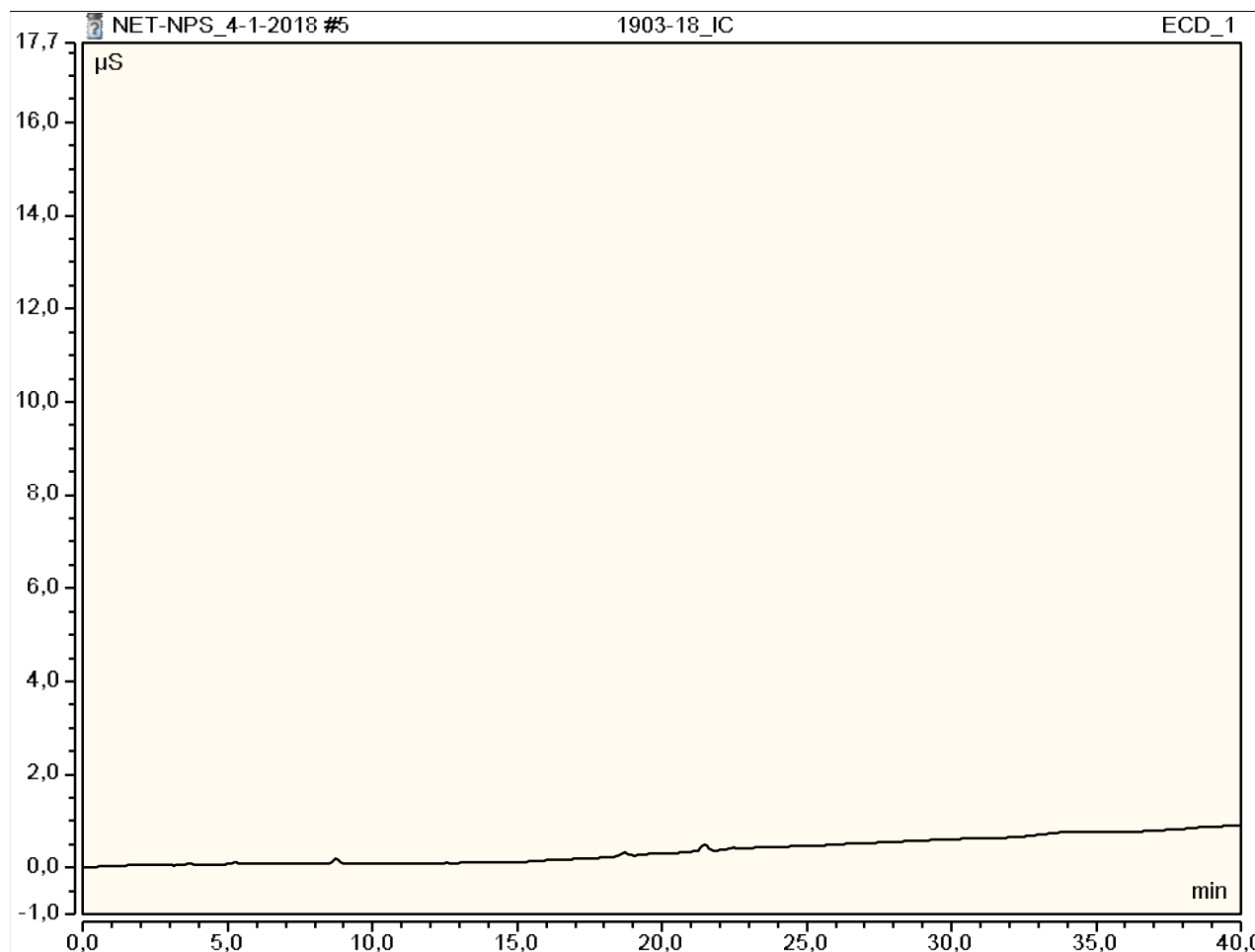
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
327.0805	1	13851375	C17 H12 Cl F N4	(M+H)+
328.0835	1	2622209.57	C17 H12 Cl F N4	(M+H)+
329.0781	1	4551712.42	C17 H12 Cl F N4	(M+H)+
330.0812	1	798314.03	C17 H12 Cl F N4	(M+H)+
349.0628	1	669216.25	C17 H12 Cl F N4	(M+Na)+
350.0654	1	122433.78	C17 H12 Cl F N4	(M+Na)+
351.0601	1	206334.45	C17 H12 Cl F N4	(M+Na)+
365.0363	1	78276.2	C17 H12 Cl F N4	(M+K)+
691.1104	1	114960.06	C17 H12 Cl F N4	(2M+K)+
693.1083	1	90368.58	C17 H12 Cl F N4	(2M+K)+

--- End Of Report ---

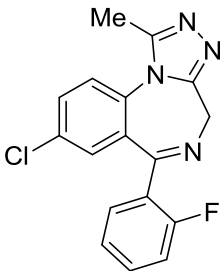
Peak Integration Report

Sample Name:	1903-18_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	04-jan-2018 / 11:57	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height μS	Amount n.a.
		TOTAL:		0,00	0,00	0,00



**R E P O R T**

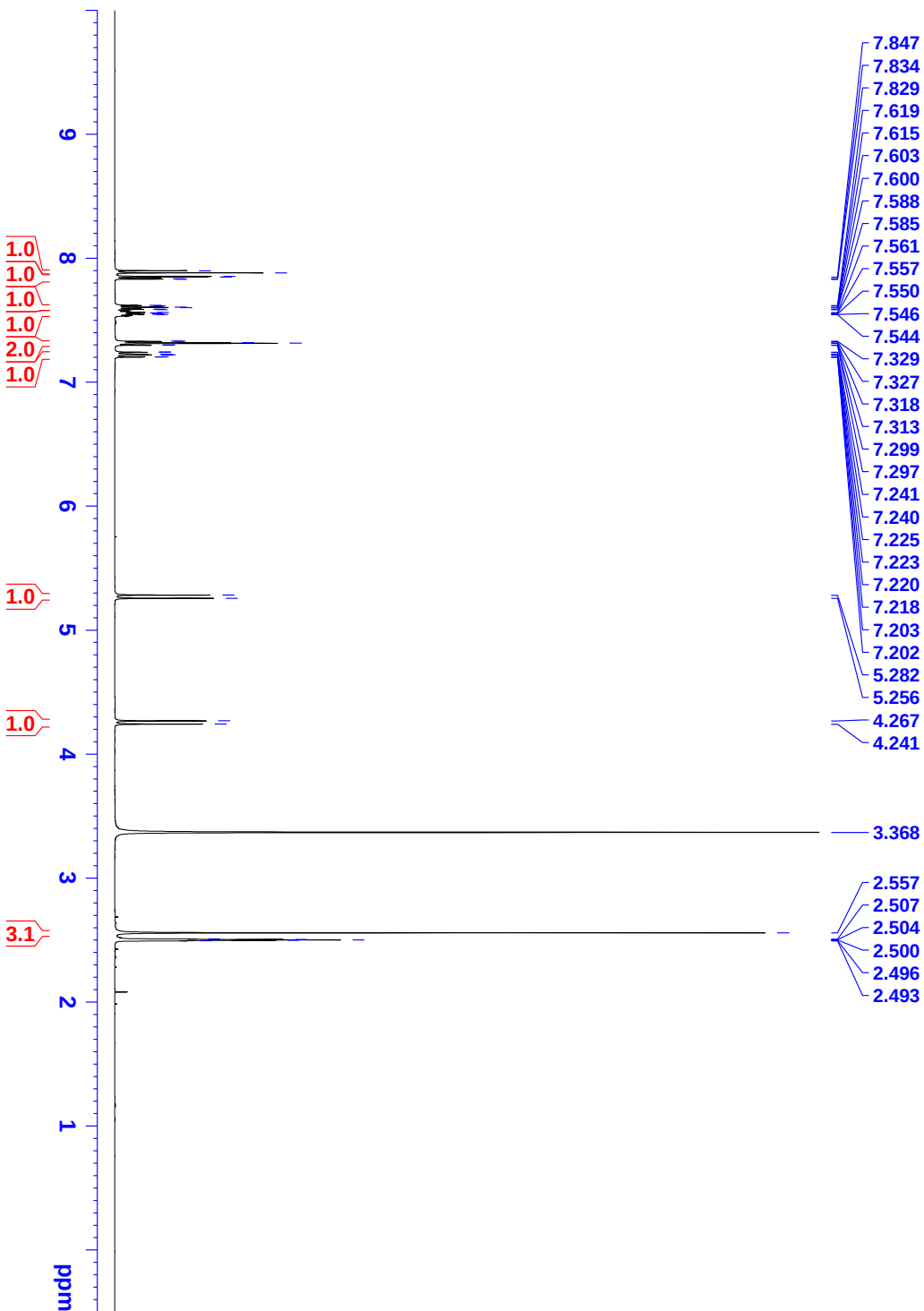
Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	1903-18
Received date:	January 11, 2018
Our notebook code:	NFL-1903-18
NMR sample preparation:	20.5 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC, ^{19}F
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $\text{C}_{17}\text{H}_{12}\text{ClFN}_4$ Exact Mass: 326,0735 Molecular Weight: 326,7594
Chemical name:	8-Chloro-6-(2-fluorophenyl)-1-methyl-4H-benzo[f][1,2,4]triazolo[4,3-a]diazepine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >97% purity of a sample based on ^1H NMR spectrum
Supporting information:	Copies of ^1H and ^{13}C NMR spectra, ^1H and ^{13}C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	January 26, 2018

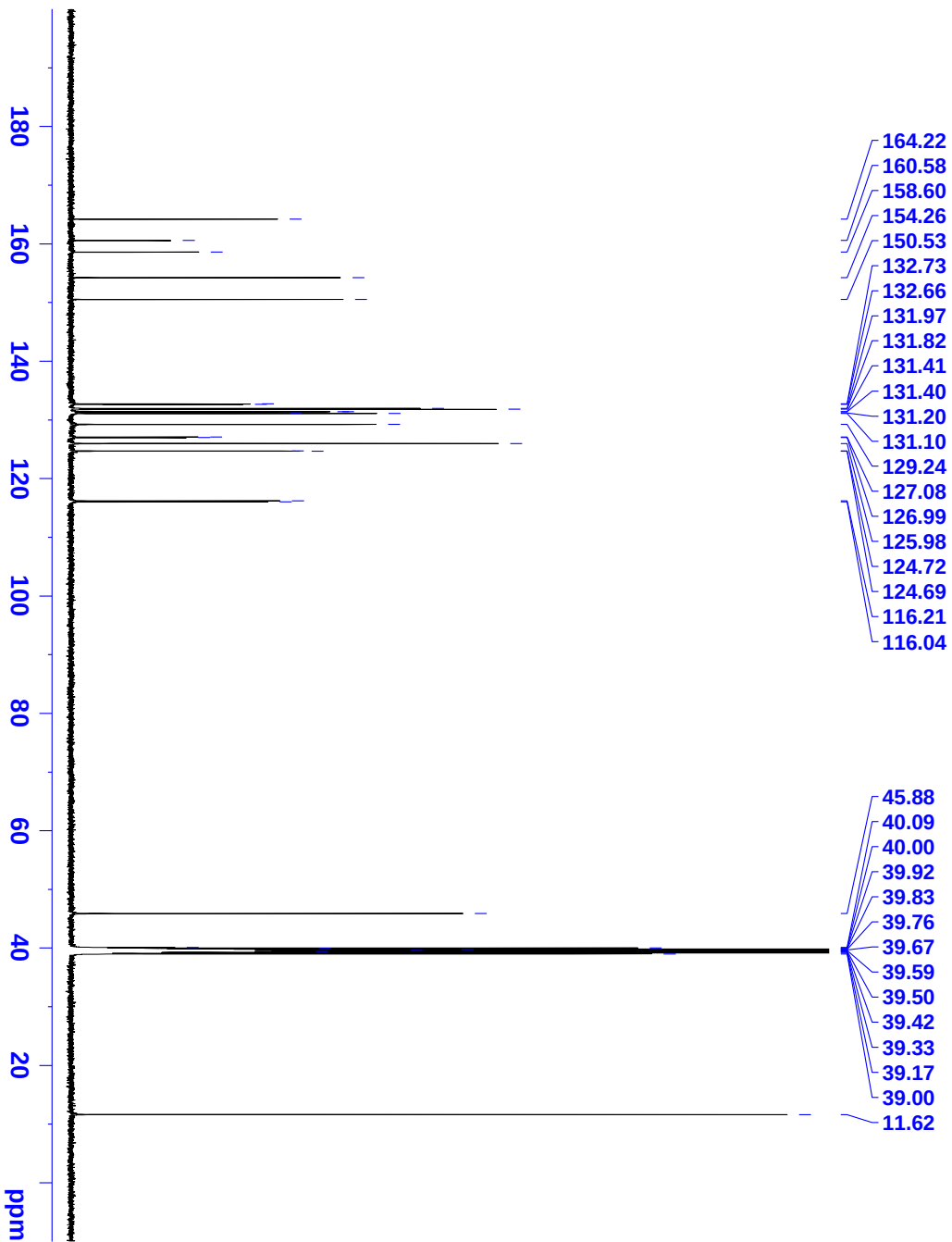
NFL-1903-18

Current Data Parameters
 NAME NFL-1903-18
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180125
 Time 23.00
 INSTRUM spect
 PROBHD 5 mm PABO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 80.6
 DW 50.000 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 500.130885 MHz
 NUC1 1H
 P1 8.70 usec
 PLW1 26.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 500.130045 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
NAME NFL-1903-18
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180126
Time_ 2.38
INSTRUM 5 mm PABBO BB-
PROBHD spect
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 2.00000000 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 125.757785 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40