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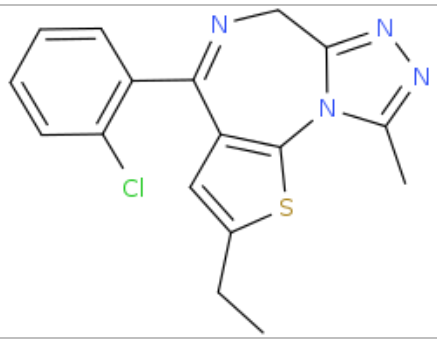
ANALYTICAL REPORT

Etizolam (C17H15ClN4S)

7-(2-chlorophenyl)-4-ethyl-13-methyl-3-thia-1,8,11,12-tetraazatricyclo[8.3.0.02,6]trideca-2(6),4,7,10,12-pentaene

Remark – other NPS detected: none

Sample ID:	1747-16
Sample description:	blotter
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	12/20/2016
Date of entry (M/D/Y) into NFL database:	2/17/2017
Report[1] 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	7-(2-chlorophenyl)-4-ethyl-13-methyl-3-thia-1,8,11,12-tetraazatricyclo[8.3.0.02,6]trideca-2(6),4,7,10,12-pentaene
Other names	
Formula (per base form)	C17H15ClN4S
M_w (g/mol)	342,85
Salt form/anions detected	not tested
StdInChIKey (for baze form)	VMZUTJCNQWMAGF-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	blotter

[1] This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

[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

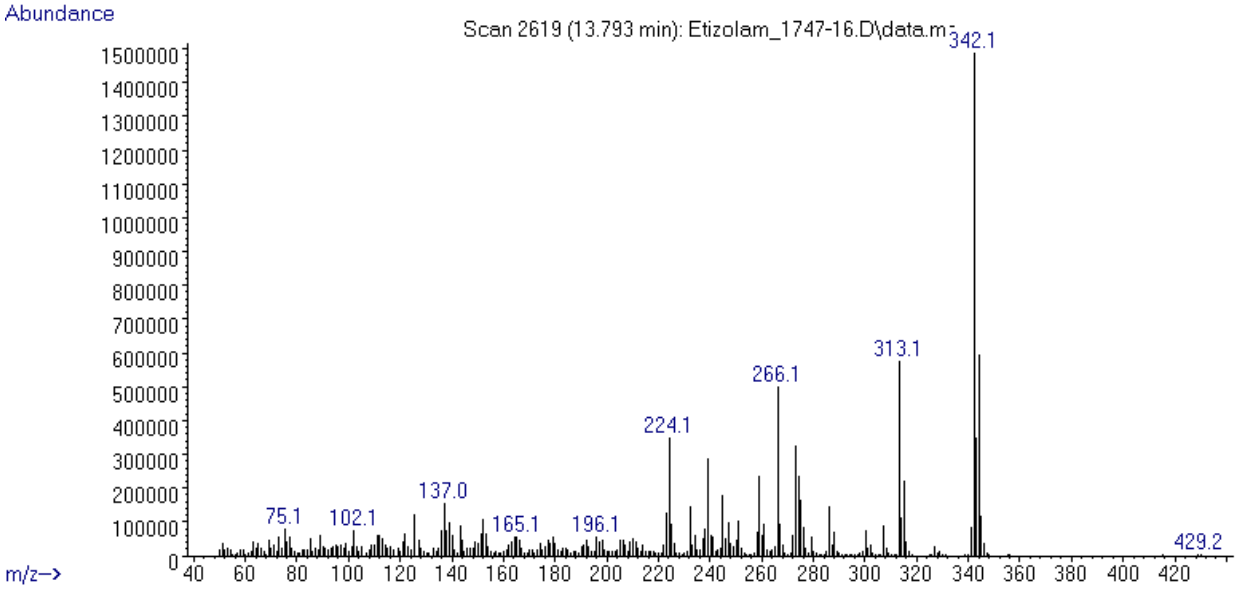
Supporting information

Solubility in	result/remark
CH_2Cl_2	na
MeOH	na
H_2O	na

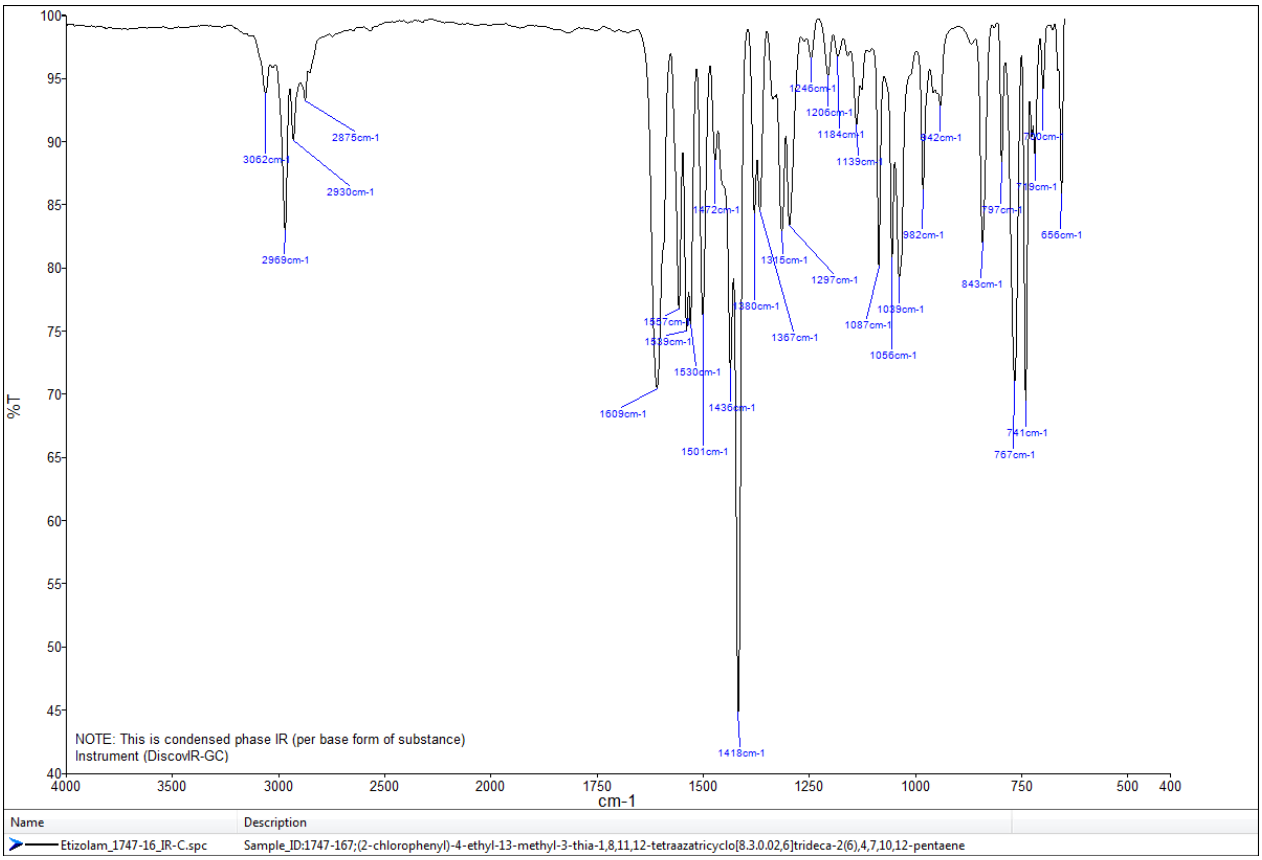
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,79 BP(1): 342; BP(2): 344,BP(3) :313,
HPLC-TOF	+	Exact mass (theoretical): 342,0706; measured value Δppm:0,64; formula:C17H15ClN4S
FTIR-ATR	-	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	-	
NMR (in FKKT)	-	
validation		MS consistent by SWGDRUG.L and ENFSI15.L,
other		

ANALYTICAL RESULTS

MS (EI)



IR (condensed phase – after chromatographic separation)



TOF REPORT

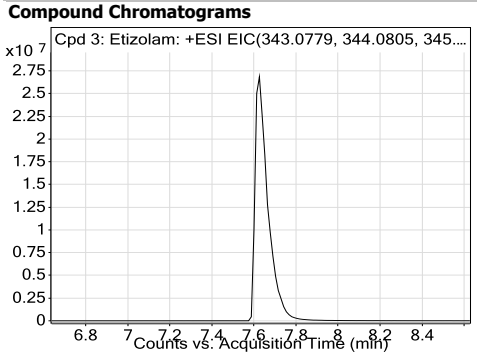
Data File	Etizolam_1747-16.d	**Sample Name**	ID_1747-16
Sample Type	Sample	**Position**	P1-C5
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-20_12_2016-XDB-C18-ESI-poz-soft.m	**Acquired Time**	12/29/2016 9:17:14 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

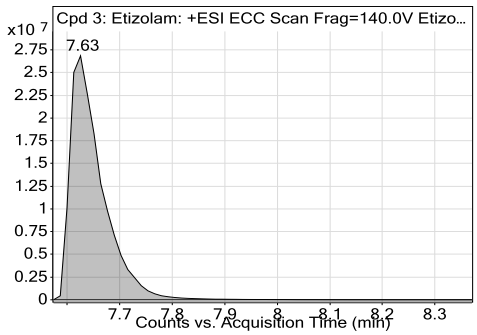
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 3: Etizolam	Etizolam	C17 H15 Cl N4 S	7.63	342.0704

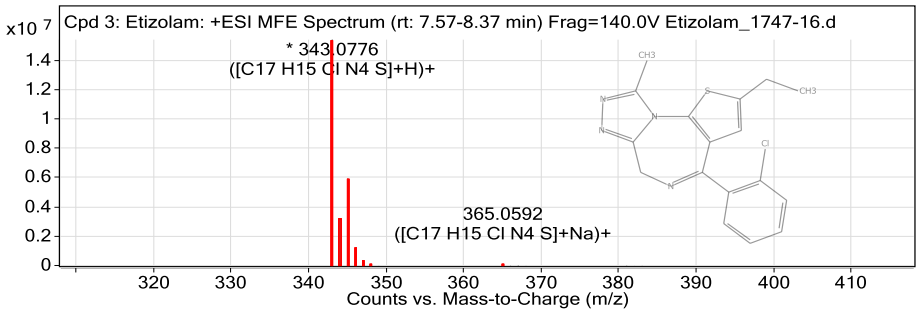
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Etizolam	343.0776	7.63	342.0704	7.63	C17 H15 Cl N4 S	342.0706	0.64

Compound Chromatograms

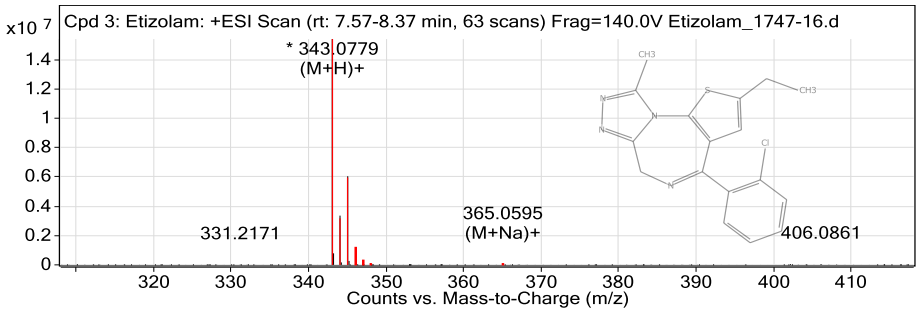




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
343.0776	1	15412519	C17 H15 Cl N4 S	(M+H)+
344.0805	1	3226151.14	C17 H15 Cl N4 S	(M+H)+
345.0751	1	5915644.65	C17 H15 Cl N4 S	(M+H)+
346.0779	1	1093399.77	C17 H15 Cl N4 S	(M+H)+
347.0744	1	277332.29	C17 H15 Cl N4 S	(M+H)+
348.0747	1	41353.69	C17 H15 Cl N4 S	(M+H)+
365.0592	1	50443.83	C17 H15 Cl N4 S	(M+Na)+
366.062	1	10156.64	C17 H15 Cl N4 S	(M+Na)+
367.0566	1	18751.96	C17 H15 Cl N4 S	(M+Na)+
381.0332	1	12269.21	C17 H15 Cl N4 S	(M+K)+

--- End Of Report ---