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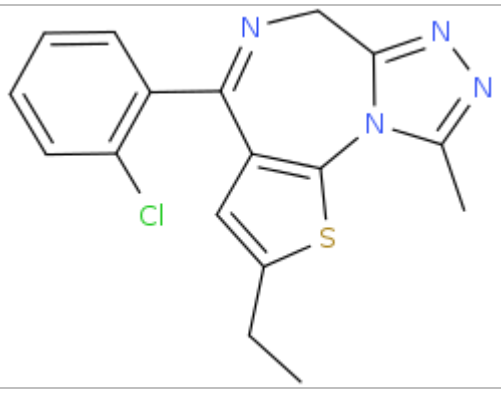
# ANALYTICAL REPORT

## 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: | 1835-17 |
| Sample description: | powder |
| Sample type: | seized /KP |
| Date of sample receipt (M/D/Y): | 6/10/2017 |
| Date of entry (M/D/Y) into NFL database: | 8/18/2017 |
| 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 | 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 | Etilaam, Etizola, Sedekopan, Etizest, Pasaden, Depas |
| Formula (per base form) | C17H15ClN4S |
| $M_w$ (g/mol) | 342,85 |
| Salt form/anions detected | base |
| StdInChIKey (per base form) | VMZUTJCNQWMAGF-UHFFFAOYSA-N |
| Other NPS detected | none |
| Additional info (purity..) | pure by GC-MS, HPLC-TOF |

[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 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

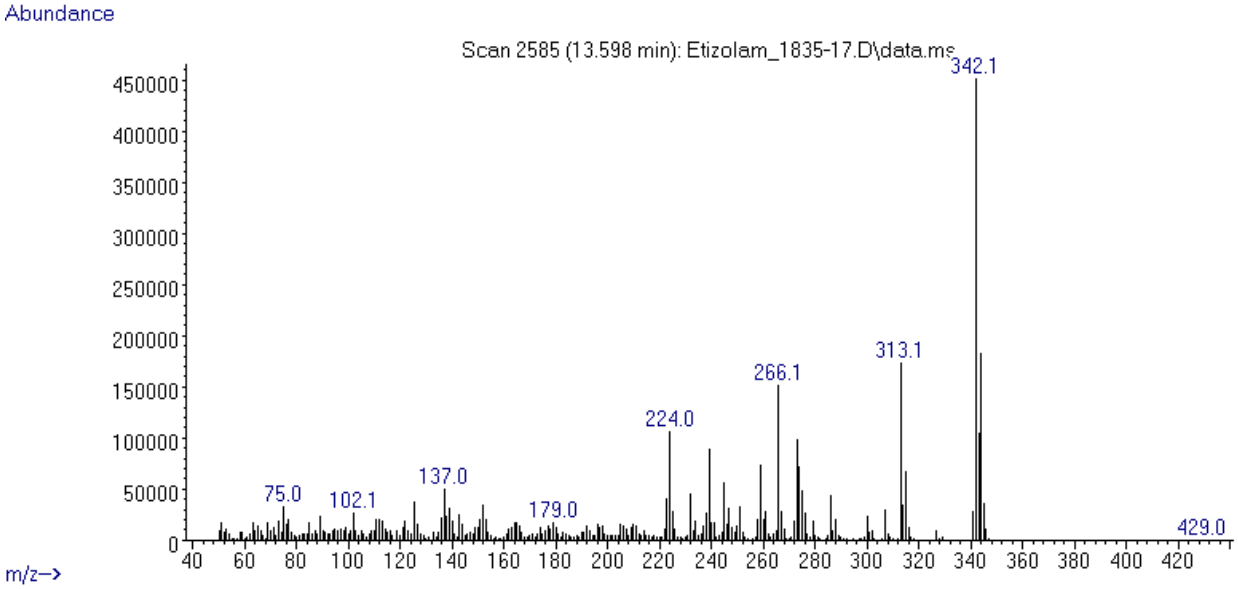
## Supporting information

| Solubility in | result/remark |
|---|---|
| $CH_2Cl_2$ | soluble |
| MeOH | soluble |
| $H_2O$ | not tested |

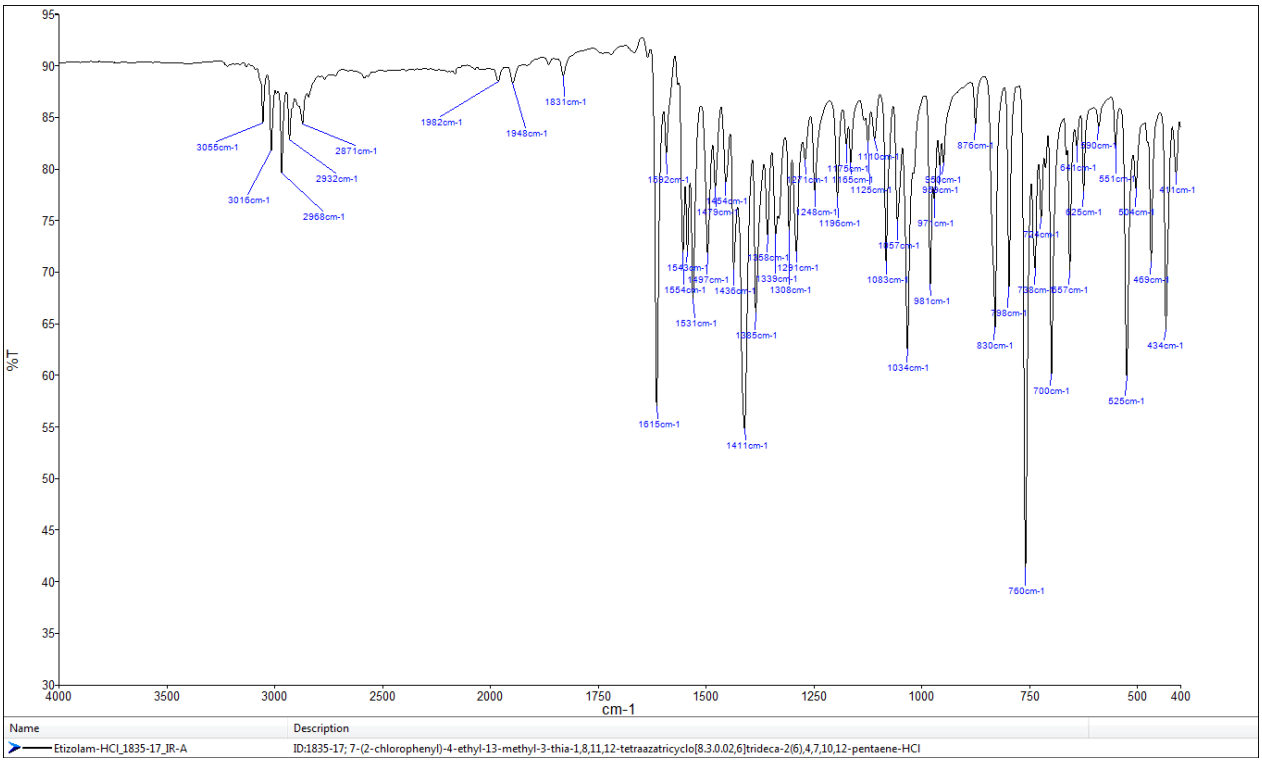
| Analytical technique: | applied | remarks |
|---|---|---|
| GC-MS (EI ionization) | + | NFL GC-RT (min): 13,6<br>BP(1): 342; BP(2): 344,BP(3) :313, |
| HPLC-TOF | + | Exact mass (theoretical): 342,0706;<br>measured value Δppm:-2,19;<br>formula:C17H15ClN4S |
| FTIR-ATR | + | direct measurement (sample as received) |
| FTIR (condensed phase) always as base form | + | |
| IC (anions) | + | |
| NMR (in FKKT) | - | |
| validation | | MS consistent by SWGDRUG.L and ENFSI15.L, DD2016.L |
| other | | IR-ATR spectrum in good agreement by Etizolam (Lot #17313) entry of SWGDRUG-IR library http://swgdrug.org/ir.htm |

## ANALYTICAL RESULTS

MS (EI)

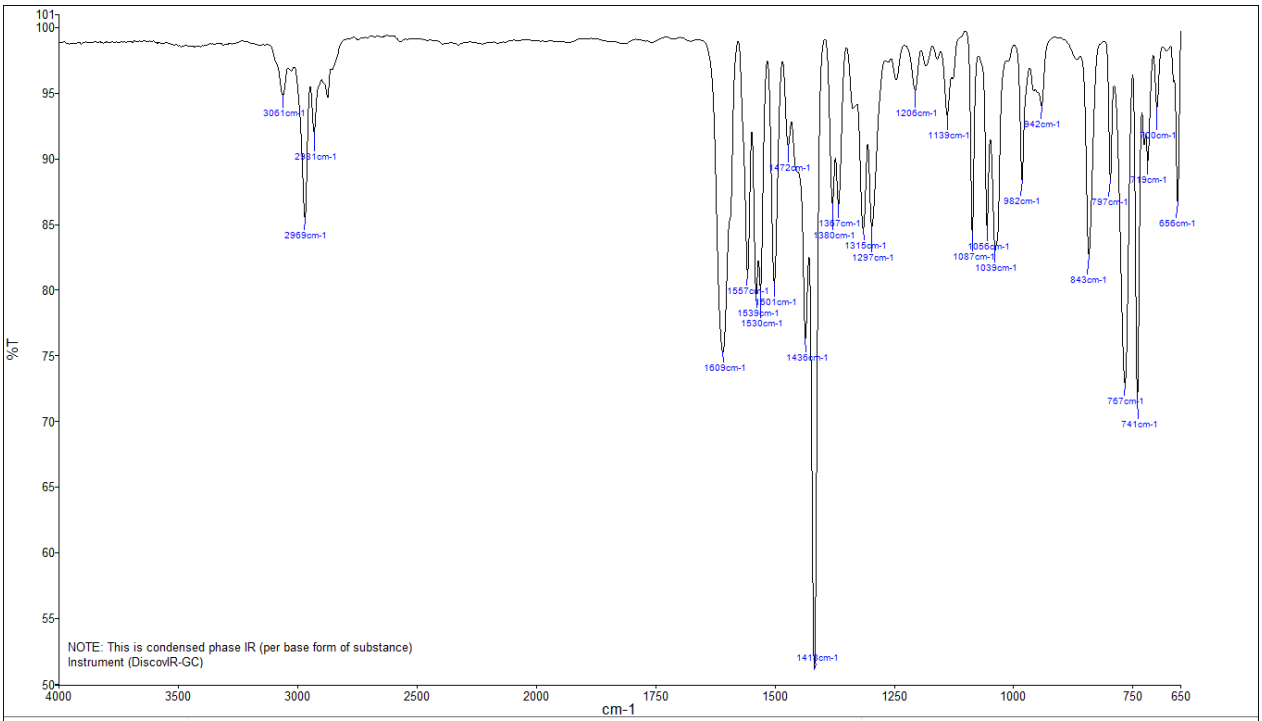


FTIR-ATR - direct measurement (sample as received)




| Name | Description |
|---|---|
| Etizolam-HCl_1835-17_IR-A | ID:1835-17; 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-HCl |

IR (condensed phase – after chromatographic separation)




| Name | Description |
|---|---|
| ETIZOLAM_1835-17_IR-C | ID=1835-17; 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 |

# TOF REPORT

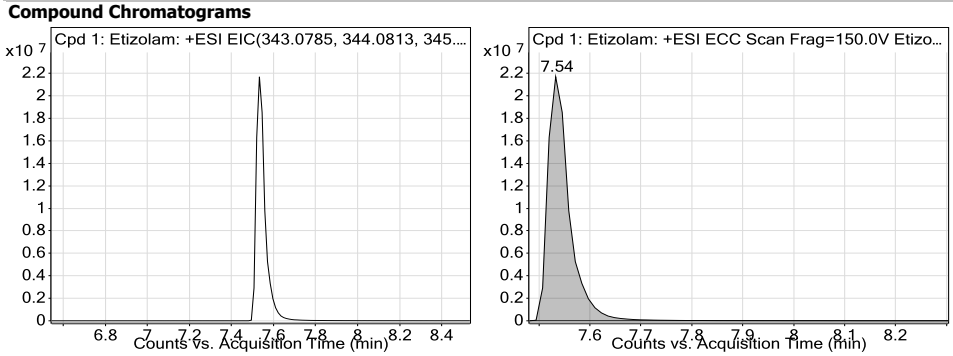
| | | | |
|---|---|---|---|
| **Data File** | Etizolam_1835-17.d | **Sample Name** | 1835-17 |
| **Sample Type** | Sample | **Position** | P1-C5 |
| **Instrument Name** | 6230B TOF LC-MS | **User Name** | TG |
| **Acq Method** | general-19_07_2017-XDB-C18-ESI-final.m | **Acquired Time** | 8/17/2017 1:11:44 PM |
| **IRM Calibration Status** | Success | **DA Method** | Drugs_NFL.m |
| **Comment** | MeOH | | |

**Compound Table**

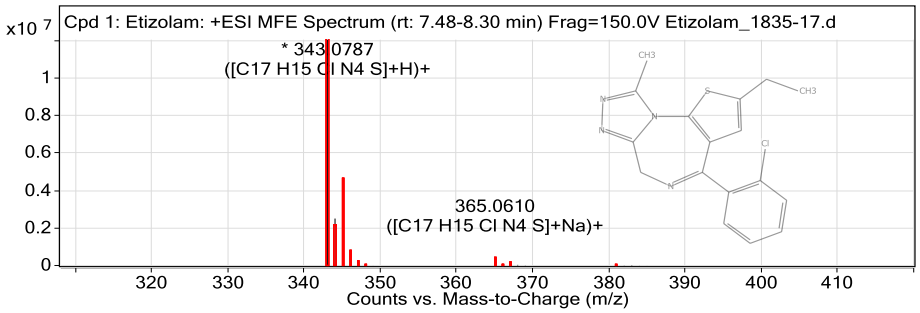
| Label | Compound Name | MFG Formula | Obs. RT | Obs. Mass |
|---|---|---|---|---|
| Cpd 1: Etizolam | Etizolam | C17 H15 Cl N4 S | 7.54 | 342.0713 |

| Name | *Obs. m/z* | Obs. RT | Obs. Mass | DB RT | DB Formula | DB Mass | DB Mass Error (ppm) |
|---|---|---|---|---|---|---|---|
| **Etizolam** | 343.0787 | 7.54 | 342.0713 | 7.53 | C17 H15 Cl N4 S | 342.0706 | -2.19 |

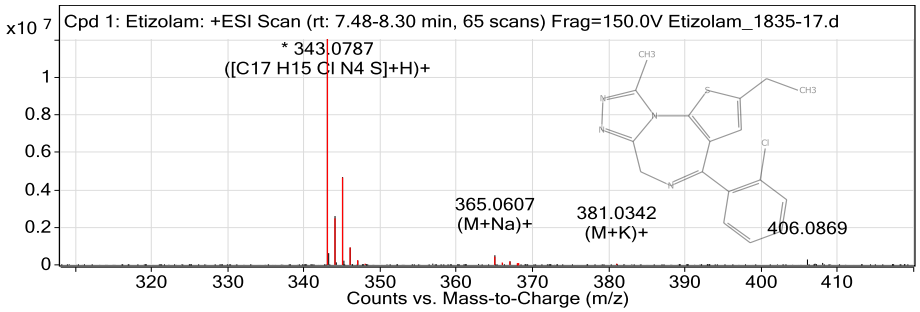
**Compound Chromatograms**




MFE MS Zoomed Spectrum




MS Zoomed Spectrum




**MS Spectrum Peak List**

| *Obs. m/z* | Charge | Abund | Formula | Ion/Isotope |
|---|---|---|---|---|
| 343.0787 | 1 | 12057925 | C17 H15 Cl N4 S | (M+H)+ |
| 344.0817 | 1 | 2505450.83 | C17 H15 Cl N4 S | (M+H)+ |
| 345.0763 | 1 | 4593646.57 | C17 H15 Cl N4 S | (M+H)+ |
| 346.079 | 1 | 841067.72 | C17 H15 Cl N4 S | (M+H)+ |
| 347.0756 | 1 | 210298.17 | C17 H15 Cl N4 S | (M+H)+ |
| 348.076 | 1 | 34849.52 | C17 H15 Cl N4 S | (M+H)+ |
| 365.061 | 1 | 462277.5 | C17 H15 Cl N4 S | (M+Na)+ |
| 366.0635 | 1 | 89916.37 | C17 H15 Cl N4 S | (M+Na)+ |
| 367.0581 | 1 | 168065.3 | C17 H15 Cl N4 S | (M+Na)+ |
| 381.0344 | 1 | 42638.49 | C17 H15 Cl N4 S | (M+K)+ |

--- End Of Report ---