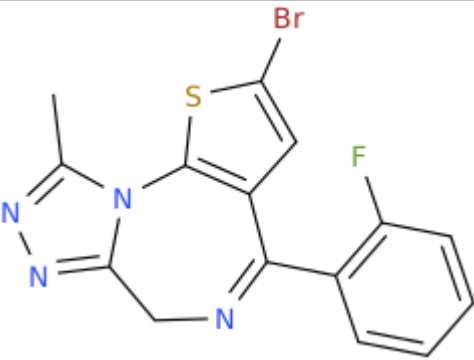


ANALYTICAL REPORT¹Flubrotizolam (C₁₅H₁₀BrFN₄S)4-bromo-7-(2-fluorophenyl)-13-methyl-3-thia-1,8,11,12-tetraazatricyclo[8.3.0.0^{2,6}]trideca-2(6),4,7,10,12-pentaene

Remark – other active cpd. detected

| | |
|-----------------------------|-----------------------|
| Sample ID: | 3006-21 |
| Sample description: | powder - brown |
| Sample type: | RM-reference material |
| Comments: | CAY Lot#0612268-1, |
| Date of entry (DD/MM/YYYY): | 29/10/2021 |

| | |
|---|--|
| Substance identified-structure ² (base form) |  |
| Systematic name: | 4-bromo-7-(2-fluorophenyl)-13-methyl-3-thia-1,8,11,12-tetraazatricyclo[8.3.0.0 ^{2,6}]trideca-2(6),4,7,10,12-pentaene |
| Other names: | 2-bromo-4-(2-fluorophenyl)-9-methyl-6H-thieno[3,2-f][1,2,4]triazolo[4,3-a][1,4]diazepine |
| Formula (per base form) | C ₁₅ H ₁₀ BrFN ₄ S |
| M _w (g/mol) | 377,24 |
| Salt form: | base |
| StdInChIKey (per base form) | VOZDBDBHBXLWCG-UHFFFAOYSA-N |
| Other active cpd. detected | |
| Add.info (purity..) | ≥98% |

¹ Approved by: Dr. Sonja Klemenc² Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

Report updates

| date | comments (explanation) |
|------|------------------------|
| | |
| | |
| | |
| | |

Supporting information

| Analytical technique: | applied | remarks |
|-------------------------|---------|---|
| GC-MS (EI ionization) | + | NFL GC-RT (min): 12,54 BP(1): 229; BP(2): 378,BP(3) :376, |
| FTIR-ATR | + | direct measurement |
| GC-IR (condensed phase) | + | always as base form |
| HPLC-TOF | + | exact mass theoretical: 375,9794 / measured Δ ppm: -0,45 |

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (9.258 min). Injection volume 1 μ l 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 μ m. 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 6.1 min, then heating at 50 °C/min up to 325 °C and finally 9.1 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. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1} ; resolution 4 cm^{-1}

3. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 μ l 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.

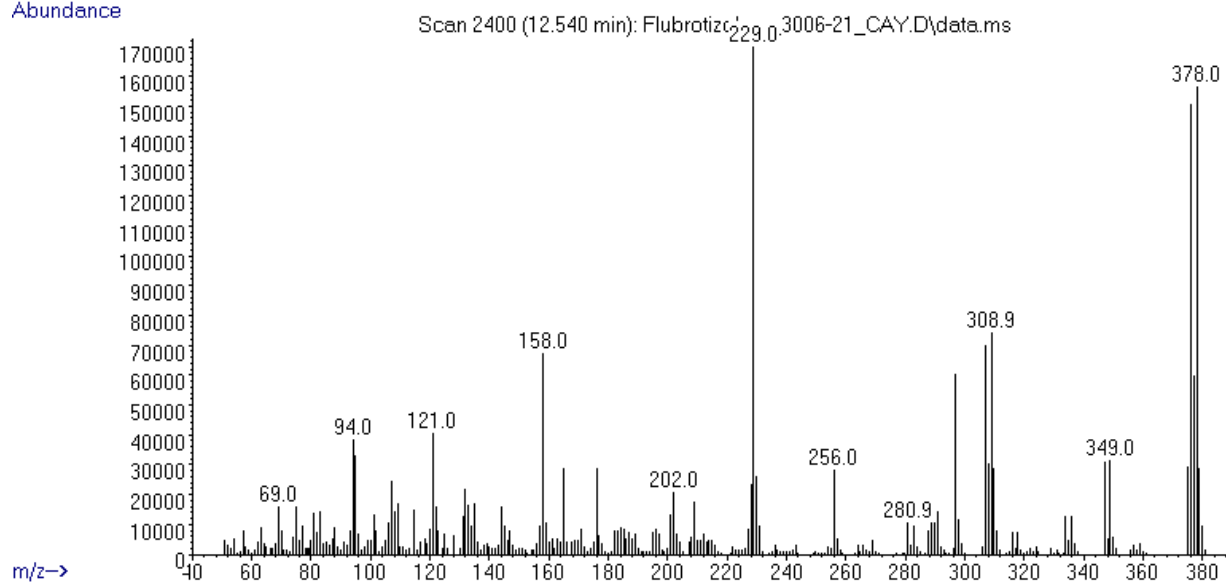
IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm^{-1} .

4. 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 μ l. 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.

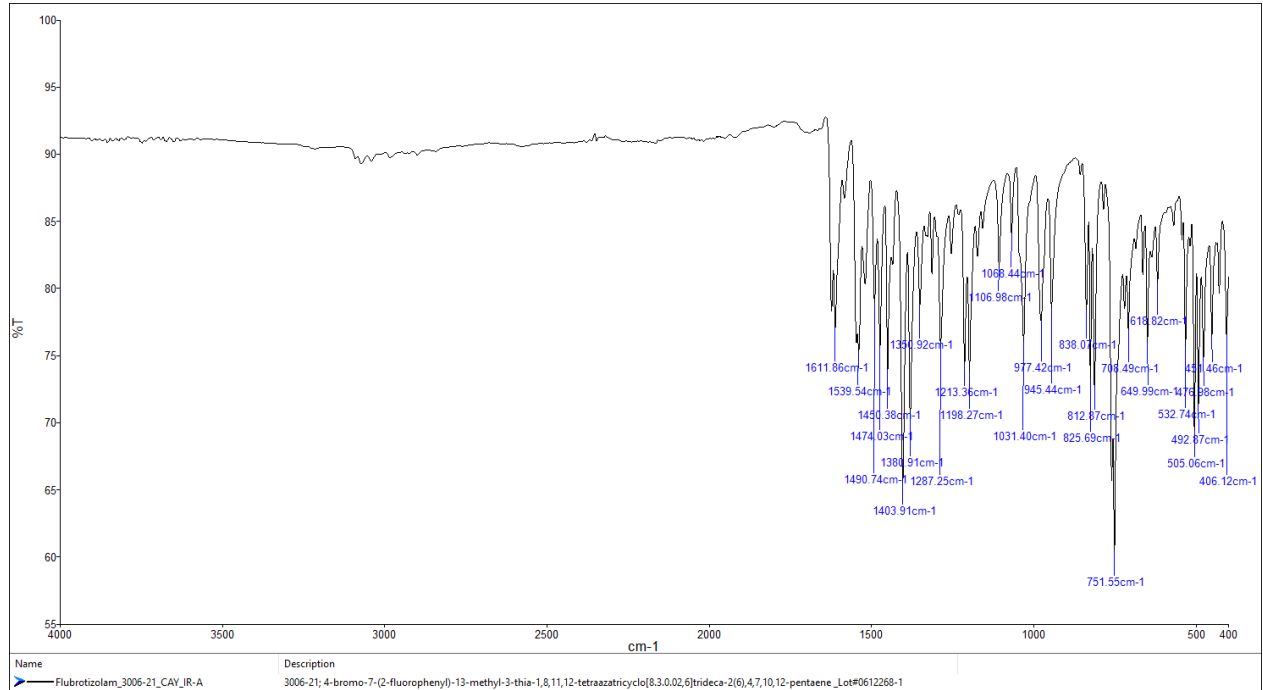
ANALYTICAL RESULTS

MS (EI)

Abundance



FTIR-ATR (direct measurement – sample as received)



IR- (condensed (solid) phase – after chromatographic separation) - spectrum per base form

