ANALYTICAL REPORT

4F-CUMYL-5F-PINACA (C22H25F2N3O)

1-(5-fluoropentyl)-N-[2-(4-fluorophenyl)propan-2-yl]-1H-indazole-3-carboxamide

Remark – other active cpd. detected none

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1946-18</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample description:</td>
<td>powder - granulated - white</td>
</tr>
<tr>
<td>Sample type:</td>
<td>RM-reference material</td>
</tr>
<tr>
<td>Comments:</td>
<td>Chiron AS Lot20662,</td>
</tr>
<tr>
<td>Date of entry (DD/MM/YYYY):</td>
<td>01/06/2018</td>
</tr>
</tbody>
</table>

Substance identified-structure¹ (base form)

Systematic name: 1-(5-fluoropentyl)-N-[2-(4-fluorophenyl)propan-2-yl]-1H-indazole-3-carboxamide

Other names: SGT-65

Formula (per base form) C22H25F2N3O

Mₘ (g/mol) 385,46

Salt form: base

StdInChIKey (per base form) WAXVVLQFKMMDHR-UHFFFAOYSA-N

Other active cpd. detected none

Add.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

<table>
<thead>
<tr>
<th>date</th>
<th>comments (explanation)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Supporting information

Analytical technique: applied remarks
---
GC-MS (EI ionization) + NFL GC-RT (min): 11,76 BP(1): 233; BP(2): 145,BP(3) :370,  
FTIR-ATR + direct measurement  
GC-IR (condensed phase) + always as base form  
HPLC-TOF + exact mass theoretical: 385,1966 / measured Δppm: -1,61

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 0C for 1 min, followed by heating up to 190 0C at rate 8 0C/min, then heating up to 293 0C at a rate of 18 0C/min, hold for 7.1 min, then heating at 50 0C/min up to 325 0C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 2350C, source and quadropole temperatures 2800C and 1800C, 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 4cm⁻¹

3. 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 0C. Chromatographic separation as above (1). Split MS: IR = 1 : 9.  
MSD source EI = 70 eV. GC-MS transfer line T= 2350C, source and quadropole temperatures 2800C and 1800C, 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 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 AIS 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

Scan 2233 (11.768 min): 4F-CUMYL-5F,-INACA_1846-18_CHLID\data.ms

m/z →

10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380

63.1 109.0 145.0 174.0 139.0 252.1 238.0 320.1 372.1 233.1
FTIR-ATR

IR (condensed phase – after chromatographic separation)