ANALYTICAL REPORT

Ethylbenzyl-CYBINACA (C22H24N4O)

1-(4-cyanobutyl)-N-(1-phenylpropyl)-1H-indazole-3-carboxamide

Remark – other active cpd. detected none

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>2036-19</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample description:</td>
<td>powder - white</td>
</tr>
<tr>
<td>Sample type:</td>
<td>RM-reference material</td>
</tr>
<tr>
<td>Comments:</td>
<td>CAY Lot#0523449-4,</td>
</tr>
<tr>
<td>Date of entry (DD/MM/YYYY):</td>
<td>14/03/2019</td>
</tr>
</tbody>
</table>

Substance identified-structure\(^1\) (base form)

Systematic name: 1-(4-cyanobutyl)-N-(1-phenylpropyl)-1H-indazole-3-carboxamide

Other names: ethylbenzyl-4CN-BINACA; 4-cyano ethylbenzyl-BUTINACA

Formula (per base form) C22H24N4O

M\(_w\) (g/mol) 360.46

Salt form: base

StdInChIKey (per base form) IIBFPSPORLUHNT-UHFFFAOYSA-N

Other active cpd. detected none

Add.info (purity..) ≥98%

\(^1\) Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ DOI: 10.1021/ci100384d
Supporting information

<table>
<thead>
<tr>
<th>Analytical technique</th>
<th>applied</th>
<th>remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC-MS (EI ionization)</td>
<td>+</td>
<td>NFL GC-RT (min): 14,47 BP(1): 226; BP(2): 331, BP(3): 145,</td>
</tr>
<tr>
<td>FTIR-ATR</td>
<td>+</td>
<td>direct measurement</td>
</tr>
<tr>
<td>GC-IR (condensed phase)</td>
<td>+</td>
<td>always as base form</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>exact mass theoretical: 360,195 / measured Δppm: -1,63</td>
</tr>
</tbody>
</table>

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 El = 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))
   MSD source El = 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 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 (El)

Abundance

Scan 2736 (14.474 min): Ethylencyclopentadiene_2036-19_CAY_DBdate.ms

m/z→
FTIR-ATR – direct measurement

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