ANALYTICAL REPORT
3,4-MDO-U-47700 (C17H24N2O3)
N-[2-(dimethylamino)cyclohexyl]-N-methyl-2H-1,3-benzodioxole-5-carboxamide

Remark – other active cpd. detected none

Sample ID: 2024-18
Sample description: powder - white
Sample type: RM-reference material
Comments: CAY Lot#0511960-14,
Date of entry (DD/MM/YYYY): 27/11/2018

Substance identified-structure¹ (base form)

Systematic name: N-[2-(dimethylamino)cyclohexyl]-N-methyl-2H-1,3-benzodioxole-5-carboxamide
Other names: 3,4-MDO-U-47700; 3,4-Methylenedioxy U-47700
Formula (per base form) C17H24N2O3
M_w (g/mol) 304.39
Salt form: HCl
StdInChIKey (per base form) UUAVKYBZVMWSM-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
Report updates

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

<table>
<thead>
<tr>
<th>Analytical technique</th>
<th>applied</th>
<th>remarks</th>
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<tbody>
<tr>
<td>GC-MS (EI ionization)</td>
<td>+</td>
<td>NFL GC-RT (min): 9.54 BP(1): 84; BP(2): 125, BP(3): 149,</td>
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<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>
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<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>exact mass theoretical: 304.1787 / measured Δppm: -0.94</td>
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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 0C 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 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 4cm⁻¹

3. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
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.
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 1876 (9.542 min): 3-4-MDO_UU_42700_2024-18_OAY_Digastorms

m/z→
FTIR-ATR – direct measurement

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