ANALYTICAL REPORT

THF-F (C24H30N2O2)
N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]oxolane-2-carboxamide

Remark – other NPS detected: none

| Sample ID: | 1659-16 |
| Sample description: | powder - brown |
| Sample type: | test purchase /RESPONSE -purchasing |
| Date of sample receipt (M/D/Y): | 10/18/2016 |
| Date of entry (M/D/Y) into NFL database: | 1/13/2017 |
| Report updates (if any) will be published here: | http://www.policija.si/apps/nfl_response_web/seznam.php |

Substance identified - structure (base form)

Systematic name: N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]oxolane-2-carboxamide

Other names: TETRAHYDROFURAN-F; THF-F; THF-fentanyl; Tetrahydrofuranyl-fentanyl

Formula (per base form): C24H30N2O2

Mw (g/mol): 378.52

Salt form/anions detected: HCl

StdInChIKey (for base form): OHJNHKUFSKAANI-UHFFFAOYSA-N

Other NPS detected: none

Additional info (purity..): Sample is not pure by GC-MS, and TOF (impurity 4-Aminophenyl-1-phenethylpiperidine)

1 This report has been produced with the financial support of the Prevention of and Fight against Crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

2 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>01/03/2017</td>
<td>Some accidentally omitted analytical data were added (TOF, IC, NMR).</td>
</tr>
</tbody>
</table>

**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 °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 EI = 70 eV. GC-MS transfer line T= 235°C, source and quadrupole 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. **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))
   MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadrupole 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.

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

<table>
<thead>
<tr>
<th>Solubility in</th>
<th>result/remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₂Cl₂</td>
<td>soluble</td>
</tr>
<tr>
<td>MeOH</td>
<td>soluble</td>
</tr>
<tr>
<td>H₂O</td>
<td>soluble</td>
</tr>
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</table>

<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): 13,87</td>
</tr>
<tr>
<td></td>
<td></td>
<td>BP(1): 287; BP(2): 71, BP(3): 146,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 378,2307;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>measured value Δppm:-0.82;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>formula:C₂₄H₃₀N₂O₂</td>
</tr>
<tr>
<td>FTIR-ATR</td>
<td></td>
<td>direct measurement (sample as received)</td>
</tr>
<tr>
<td>FTIR (condensed phase)</td>
<td>+</td>
<td>always as base form</td>
</tr>
<tr>
<td>IC (anions)</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>NMR (in FKKT)</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>validation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>other</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
ANALYTICAL RESULTS

MS (EI)

Abundance

Scan 2532 (13.857 min) THF-F-HCl_1859-16.D preceded ms

m/z ->

71.0
105.0
146.1
189.1
216.1
244.1
287.1
341.0
376.2
FTIR-ATR - direct measurement (sample as received)

IR (condensed phase – after chromatographic separation)
### TOF REPORT

#### Data File
1659-16_TOF.d

#### Sample Name
ID_1659-16

#### Sample Type
Sample

#### Position
P1-A2

#### Instrument Name
6230B TOF LC-MS

#### User Name
TG

#### Acq Method
general-24_08_2016-XDB-C18-ESI-poz-soft.m

#### Acquired Time
9/1/2016 7:41:21 AM

#### IRM Calibration Status
Success

#### DA Method
Drugs NFL.m

#### Comment
extract in MeOH

### Compound Table

<table>
<thead>
<tr>
<th>Label</th>
<th>Compound Name</th>
<th>MFG Formula</th>
<th>Obs. RT</th>
<th>Obs. Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cpd 2: C24H30N2O2 (received as THF-F)</td>
<td>C24H30N2O2 (received as THF-F)</td>
<td>C24 H30 N2 O2</td>
<td>6.059</td>
<td>378.231</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Name</th>
<th>Obs. m/z</th>
<th>Obs. RT</th>
<th>Obs. Mass</th>
<th>DB RT</th>
<th>DB Formula</th>
<th>DB Mass</th>
<th>DB Mass Error (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C24H30N2O2 (received as THF-F)</td>
<td>379.2382</td>
<td>6.059</td>
<td>378.231</td>
<td>6.06</td>
<td>C24 H30 N2 O2</td>
<td>378.2307</td>
<td>-0.82</td>
</tr>
</tbody>
</table>

### Compound Chromatograms

#### MFE MS Zoomed Spectrum

- Obs. m/z: 379.2382
- Obs. RT: 6.059
- Obs. Mass: 378.231
- DB RT: 6.06
- DB Formula: C24 H30 N2 O2
- DB Mass: 378.2307
- DB Mass Error (ppm): -0.82

#### MS Zoomed Spectrum

- Obs. m/z: 379.2383
- Obs. RT: 6.059
- Obs. Mass: 378.231

### MS Spectrum Peak List

<table>
<thead>
<tr>
<th>Obs. m/z</th>
<th>Charge</th>
<th>Abund</th>
<th>Formula</th>
<th>Ion/Isotope</th>
</tr>
</thead>
<tbody>
<tr>
<td>379.2382</td>
<td>1</td>
<td>11645100</td>
<td>C24 H30 N2 O2</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>380.2416</td>
<td>1</td>
<td>3310979.68</td>
<td>C24 H30 N2 O2</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>381.2453</td>
<td>1</td>
<td>428654.54</td>
<td>C24 H30 N2 O2</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>382.2475</td>
<td>1</td>
<td>42542.50</td>
<td>C24 H30 N2 O2</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>383.2493</td>
<td>1</td>
<td>3813.50</td>
<td>C24 H30 N2 O2</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>401.2203</td>
<td>1</td>
<td>84530.29</td>
<td>C24 H30 N2 O2</td>
<td>(M+Na)+</td>
</tr>
<tr>
<td>402.2233</td>
<td>1</td>
<td>22627.24</td>
<td>C24 H30 N2 O2</td>
<td>(M+Na)+</td>
</tr>
<tr>
<td>403.2254</td>
<td>1</td>
<td>3414.82</td>
<td>C24 H30 N2 O2</td>
<td>(M+Na)+</td>
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</tbody>
</table>

--- End Of Report ---
### Peak Integration Report

<table>
<thead>
<tr>
<th>No.</th>
<th>Time min</th>
<th>Peak Name</th>
<th>Peak Type</th>
<th>Area µS/min</th>
<th>Height µS</th>
<th>Amount mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>7.77</td>
<td>Chloride</td>
<td>BMB</td>
<td>4.44</td>
<td>22.31</td>
<td>n.a.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>TOTAL:</td>
<td>4.44</td>
<td>22.31</td>
</tr>
</tbody>
</table>

**Sample Name:** ID_1659-16_IC  
**Injection Vol.:** 25.00  
**Injection Type:** Unknown  
**Dilution Factor:** 1.0000  
**Program:** ANIONI  
**Operator:** kemija  
**Inj. Date / Time:** 01-sep-2016 / 15:29  
**Run Time:** 42.00
Sample ID: 1659-16

Our notebook code: P-1659-16

NMR sample preparation: 15 mg dissolved in 0.7 mL CDCl₃

NMR experiments: ¹H, ¹³C, ¹H–¹H gs-COSY, ¹H–¹³C gs-HSQC, ¹H–¹³C gs-HMBC, ¹H–¹⁵N gs-HMBC.

Proposed structure:

Chemical name: 1-phenethyl-4-(N-phenyltetrahydrofuran-2-carboxamido)piperidin-1-ium cation

Comments: - Structure elucidation based on 1D and 2D NMR spectra
- Sample is not pure, as is evident by NMR it contains some minor impurities.

Supporting information: Copies of ¹H and ¹³C NMR spectra

Author: Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc

Date of report: January 11, 2017

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Current Data Parameters

NAME          p-1659-16
EXPNO                 1
PROCNO                1
F2 - Acquisition Parameters

WID          500.1300020 MHz
EM          30.90 µsec
SSB         0.30 Hz
LB                 0.30 Hz
PC                 1.00

--- CHANNEL 1 ---

110 1.0 sec
200 2.0 sec
500 5.0 sec
1000 1.0 sec
2000 2.0 sec
5000 5.0 sec
10000 1.0 sec
20000 2.0 sec

Solvent: CDCl3
6.4956 ppm

- Acquisition Parameters

PROCNO 1
EXONO 1
NAME p-1659-16