ANALYTICAL REPORT

Cyclopentyl-F (C25H32N2O)

N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]cyclopentanecarboxamide

Remark – other NPS detected: none

Sample ID: 1661-16
Sample description: powder
Sample type: test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y): 8/30/2016
Date of entry (M/D/Y) into NFL database: 3/6/2017
Report updates (if any) will be published here:

Substance identified - structure (base form)

Systematic name: N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]cyclopentanecarboxamide
Other names: Cyclopentyfentanyl; Cyclopentyl fentanyl, CPF
Formula (per base form): C25H32N2O
M_w (g/mol): 376,54
Salt form/anions detected: oxalate
StdInChIKey (per base form): PEASFKSPITUZGT-UHFFFAOYSA-N
Other NPS detected: none
Additional info (purity..)

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
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 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. 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 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.

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

<table>
<thead>
<tr>
<th>date</th>
<th>comments (explanation)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18/05/2017</td>
<td>Empirical formula corrected.</td>
</tr>
</tbody>
</table>
## Supporting information

<table>
<thead>
<tr>
<th>Solubility in</th>
<th>result/remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₂Cl₂</td>
<td>partially</td>
</tr>
<tr>
<td>MeOH</td>
<td>soluble</td>
</tr>
<tr>
<td>H₂O</td>
<td>partially</td>
</tr>
</tbody>
</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): 13,74&lt;br&gt;BP(1): 285; BP(2): 69; BP(3): 189,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 376,2515; measured value Δppm: 0.56; formula: C₂₅H₃₂N₂O</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>MS consistent by the one published in EMCDDA EDND by Sweden</td>
</tr>
<tr>
<td>other</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
ANALYTICAL RESULTS

MS (EI)

Abundance

Scan 2609 (13.736 min): KEM-ID_1661-16.D

MS-explained (red and green labeled peaks)

MS-explained (red and green labeled peaks)
FTIR-ATR - direct measurement (sample as received)

IR (condensed phase – after chromatographic separation)

NOTE: This is condensed phase IR (as base form of substance) Instrument (Discover-GC)
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>C2SH32N2O (received as cyclopentyl-F)</td>
<td>C25H32N2O (received as cyclopentyl-F)</td>
<td>C25 H32 N2 O</td>
<td>7.12</td>
<td>376.2517</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>C2SH32N2O (received as cyclopentyl-F)</td>
<td>377.2589</td>
<td>7.12</td>
<td>376.2517</td>
<td>7.12</td>
<td>C25 H32 N2 O</td>
<td>376.2515</td>
<td>-0.56</td>
</tr>
</tbody>
</table>

Compound Chromatograms

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>377.2589</td>
<td>1</td>
<td>6255873</td>
<td>C25 H32 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>376.2522</td>
<td>1</td>
<td>1715430.4</td>
<td>C25 H32 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>379.2555</td>
<td>1</td>
<td>226304.78</td>
<td>C25 H32 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>380.2683</td>
<td>1</td>
<td>21549.87</td>
<td>C25 H32 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>381.2667</td>
<td>1</td>
<td>15893.89</td>
<td>C25 H32 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>399.2405</td>
<td>1</td>
<td>15039.89</td>
<td>C25 H32 N2 O</td>
<td>(M+Na)+</td>
</tr>
<tr>
<td>400.244</td>
<td>1</td>
<td>4571.63</td>
<td>C25 H32 N2 O</td>
<td>(M+Na)+</td>
</tr>
<tr>
<td>401.244</td>
<td>1</td>
<td>987.13</td>
<td>C25 H32 N2 O</td>
<td>(M+Na)+</td>
</tr>
</tbody>
</table>

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

**Sample Name:** 1661-16_IC  
**Injection Type:** Unknown  
**Program:** ANIONI  
**Operator:** kemija  
**Inj. Date / Time:** 01-sep-2016 / 10:31  
**Run Time:** 42,00

<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>21,59</td>
<td>Oxalate</td>
<td>BMB</td>
<td>2,78</td>
<td>11,05</td>
<td>n.a.</td>
</tr>
<tr>
<td><strong>TOTAL:</strong></td>
<td></td>
<td></td>
<td></td>
<td><strong>2,78</strong></td>
<td><strong>11,05</strong></td>
<td><strong>0,00</strong></td>
</tr>
</tbody>
</table>

**Chart:**

- Peak 1: Oxalate at 21.59 min

**Report Details:**

- **Injection Volume:** 25.00
- **Dilution Factor:** 1,0000
- **Injection Date / Time:** 01-sep-2016 / 10:31
- **Run Time:** 42.00

- **Program:** ANIONI
- **Operator:** kemija

**Sample Name:** 1661-16_IC

- **Peak Integration Report**

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Anions-report template/Integration

Anions-report template/Integration

Chromleon (c) Dionex 1996-2009  
Version 7.2.0.3765
**REPORT**

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1661-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Our notebook code:</td>
<td>P-1661-16</td>
</tr>
<tr>
<td>NMR sample preparation:</td>
<td>15 mg dissolved in 0.7 mL DMSO-(d_6)</td>
</tr>
<tr>
<td>NMR experiments:</td>
<td>(^1)H, (^13)C, (^1)H–(^1)H gs-COSY, (^1)H–(^13)C gs-HSQC, (^1)H–(^13)C gs-HMBC, (^1)H–(^15)N gs-HMBC.</td>
</tr>
</tbody>
</table>

Proposed structure:

![Proposed Structure Image]

Chemical name: \(N\)-(1-phenethylpiperidin-4-yl)-\(N\)-phenylcyclopentanecarboxamide

Comments:  
- Structure elucidation based on 1D and 2D NMR spectra  
- Sample is not pure as evident by NMR; it contains oxalate (signals in \(^1\)H NMR around 5 and in \(^13\)C NMR at 164.7 ppm) and also some other minor impurities.

Supporting information: Copies of \(^1\)H and \(^13\)C NMR spectra

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

Date of report: April 9, 2017

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Current Data Parameters

NAME: P-1661-16
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date: 20160912
Time: 18.19
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: DMSO
NS: 16
DS: 2
SWH: 10000.000 Hz
FIDRES: 0.152588 Hz
AQ: 3.2768500 sec
RG: 71.8
DW: 50.000 usec
DE: 6.50 usec
TE: 300.0 K
D1: 1.00000000 sec
TD0: 1

======== CHANNEL f1 ========
SFO1: 500.1330885 MHz
NUC1: 1H
P1: 8.90 usec
PLW1: 26.00000000 W

F2 - Processing parameters
SI: 65536
SF: 500.1300000 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

---

2.05 8.08 1.96 0.99 2.00 1.98 1.97 1.97 1.31 5.01 2.08 3.00
Current Data Parameters

NAME          P-1661-16
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160912
Time              22.02
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 4096
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG                 2050
DW               16.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.00 usec
PLW1       122.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        26.00000000 W
PLW12        0.32179001 W
PLW13        0.16186000 W

F2 - Processing parameters
SI                32768
SF          125.7577885 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40