ANALYTICAL REPORT\(^1,2\)

**MN24 (C24H24N20)**

N-(naphthalen-1-yl)-1-pentyl-1H-indole-3-carboxamide

**Remark** – other NPS detected: none

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1457-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample description:</td>
<td>powder - brown</td>
</tr>
<tr>
<td>Sample type:</td>
<td>collected / Institute of Forensic medicine, University Freiburg, Germany</td>
</tr>
<tr>
<td>Date of sample receipt (M/D/Y):</td>
<td>1/14/2016</td>
</tr>
<tr>
<td>Date of entry (M/D/Y) into NFL database:</td>
<td>10/25/2016</td>
</tr>
</tbody>
</table>

**Substance identified - structure\(^3\) (base form)**

![Structure Image]

**Systematic name**

N-(naphthalen-1-yl)-1-pentyl-1H-indole-3-carboxamide

**Other names**

JWH-018 Carboxamide Derivative, MN-24, NNEI, AM-6527

**Formula (per base form)**

C24H24N20

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

356.47

**Salt form/anions detected**

base

**StdInChIKey**

GWCQNKRMTGYYIZ-UHFFFAOYSA-N

**Compound Class**

Cannabinoids

**Other NPS detected**

none

**Add.info (purity..)**

pure by GC-MS, minor impurities by NMR and HPLC- TOF

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\(^1\) 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/SEC/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.

\(^2\) Acknowledgement: Sample (not NMR confirmed) was kindly provided by the Institute of Forensic Medicine, University of Freiburg, Germany. Analytical results shown in this report were done in NFL and FKKT, Slovenia.

\(^3\) Created by OPSIN free tool: [http://opsin.ch.cam.ac.uk/](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>
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<td></td>
<td></td>
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<tr>
<td></td>
<td></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 6.1 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%, equilibrium 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
## 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>partially</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>
</table>
| GC-MS (EI ionization) | +       | NFL GC-RT (min): 18,12  
BP(1): 214; BP(2): 144, BP(3): 215, |
| HPLC-TOF             | +       | Exact mass (theoretical): 356,1889;  
measured value Δppm: 0,01;  
formula: C₂₄H₂₄N₂O |
| FTIR-ATR             | +       | direct measurement (sample as received) |
| FTIR (condensed phase) | always as base form | + |
| IC (anions)          | +       |        |
| NMR (in FKKT)        | +       |        |
| validation            |         | MS consistent by SWGDRUG.L and FTIR-ATR consistent by SWGDRUG-IR library entry |
| other                 |         |        |
ANALYTICAL RESULTS

MS (EI)

Abundance

Scan 3375 (18.119 min): JW21-18-Carboxamide-Deriv_1457-16.D\data.ms

m/z ->

0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420

100000 200000 300000 400000 500000 600000 700000 800000 900000 1000000 1100000 1200000

55.1 68.0 115.0 184.1 214.1 220.0 254.1 281.0 327.0 356.2 429.0
FTIR-ATR - direct measurement (sample as received)

IR (condensed phase – after chromatographic separation)

NOTE: This is condensed phase IR (per base form of substance) instrument (Shimadzu-IRPG)

Name | Description
--- | ---
JMH-038-carboxamide-decote,1457-16_IR-C | Sample, d: propyl-N (napthalen-1-yl-2H-indole-3-carboxamide)
JMH-038-carboxamide-decote,1457-16_IR-P | Sample, p: propyl-N (napthalen-1-yl-2H-indole-3-carboxamide)
TOF REPORT

Data File: JWH-018-Carboxamide-Derivate_1457-16_TOF.d
Sample Name: ID_1457-16
Sample Type: Sample
Instrument Name: 6230B TOF LC-MS
Acq Method: general-1512015-XDB-C18-ESI-poz-pod.m
IRM Calibration Status: Success
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 5: C38 H39 N3 O2</td>
<td>C38 H39 N3 O2</td>
<td></td>
<td>10.986</td>
<td>569.3041</td>
</tr>
</tbody>
</table>

Compound Chromatograms

MFE MS Zoomed Spectrum

MS Zoomed Spectrum

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>357.196</td>
<td>1</td>
<td>4987147.5</td>
<td>C24 H24 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>358.1997</td>
<td>1</td>
<td>1330453.35</td>
<td>C24 H24 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>359.2026</td>
<td>1</td>
<td>167737.25</td>
<td>C24 H24 N2 O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>713.3848</td>
<td>1</td>
<td>6949708.0</td>
<td>C24 H24 N2 O</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>714.3833</td>
<td>1</td>
<td>3766737.62</td>
<td>C24 H24 N2 O</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>715.3923</td>
<td>1</td>
<td>1048332.58</td>
<td>C24 H24 N2 O</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>716.3948</td>
<td>1</td>
<td>177534.46</td>
<td>C24 H24 N2 O</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>735.3672</td>
<td>1</td>
<td>335759.88</td>
<td>C24 H24 N2 O</td>
<td>(2M+Na)+</td>
</tr>
</tbody>
</table>

Obs. m/z: 713.3848
Ion: 713.3848
Charge: 1
Formula: C24 H24 N2 O
DB Mass: 356.1889
DB RT: 9.65
DB Formula: C24 H24 N2 O
DB Mass Error (ppm): -0.01

Obs. m/z: 713.3848
Ion: 713.3848
Charge: 1
Formula: C38 H39 N3 O2
DB Mass: 356.1889
DB RT: 9.645
DB Formula: C38 H39 N3 O2
DB Mass Error (ppm): -0.01
TOF REPORT

MFE MS Zoomed Spectrum

Ion/Isotope

(M+Na)+

--- End Of Report ---

C38 H39 N3 O2

--- End Of Report ---

Compound Chromatograms

MS Spectrum Peak List

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

**Sample Name:** JWH-018-Carboxamide_Derivate_1457-16_IC  
**Injection Volume:** 25.00 µL  
**Injection Type:** Unknown  
**Dilution Factor:** 1.0000  
**Program:** ANIONI  
**Operator:** kemija  
**Injection Date/Time:** 24-Jun-2016 / 20:58  
**Run Time:** 42.00 min

<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>8.06</td>
<td>Chloride</td>
<td>BMB</td>
<td>0.48</td>
<td>2.36</td>
<td>n.a.</td>
</tr>
<tr>
<td>TOTAL:</td>
<td></td>
<td></td>
<td></td>
<td>0.48</td>
<td>2.36</td>
<td>0.00</td>
</tr>
</tbody>
</table>

![Graph showing peak integration](image-url)
## Sample ID:
**1457-16**

Our notebook code: P-1457-16

NMR sample preparation: 15 mg dissolved in 0.7 mL CDCl$_3$

NMR experiments: $^1$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.

Proposed structure:

![Proposed structure](image)

Chemical name: $N$-(naphthalen-1-yl)-1-pentyl-$^1$H-indole-3-carboxamide

Comments:
- Structure elucidation based on 1D and 2D NMR spectra
- Sample is not pure as it contains some minor impurities that are evident from $^1$H and $^{13}$C NMR.

Supporting information: Copies of $^1$H and $^{13}$C NMR spectra

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

Date of report: October 21, 2016

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 publication are the sole responsibility of the Author and can in no way be taken to reflect the views of the European Commission.