ANALYTICAL REPORT

AKB-57 (C23H30N2O2)
adamantan-1-yl 1-pentyl-1H-indazole-3-carboxylate

Remark – other NPS detected: none

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1521-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample description:</td>
<td>powder - white</td>
</tr>
<tr>
<td>Sample type:</td>
<td>test purchase /RESPONSE -purchasing</td>
</tr>
<tr>
<td>Date of sample receipt (M/D/Y):</td>
<td>2/4/2016</td>
</tr>
<tr>
<td>Date of entry (M/D/Y) into NFL database:</td>
<td>2/12/2016</td>
</tr>
<tr>
<td>Report updates (if any) will be published here:</td>
<td><a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a></td>
</tr>
</tbody>
</table>

Substance identified - structure^1 (base form)

Systematic name
adamantan-1-yl 1-pentyl-1H-indazole-3-carboxylate

Other names

Formula (per base form)
C23H30N2O2

Mw (g/mol)
366,31

Salt form/anions detected
base

StdInChIKey
KCCVWUAAHDXNNQ-UHFFFAOYSA-N

Compound Class
Cannabinoids

Other NPS detected
none

Add.info (purity..)
pure by GC-MS, HPLC-TOF and NMR

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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/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></td>
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<tr>
<td></td>
<td></td>
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<tr>
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<td></td>
</tr>
</tbody>
</table>

**Instrumental methods** (if applied) in NFL

1. **GC-MS** (Agilent): GC-method is RT locked to tetracosane (RT=9.53 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 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, than 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) 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 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>low (bad)</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): 13,31 BP(1): 135; BP(2): 231; BP(3): 215,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 366,2307; measured value Δppm: 0,29; formula: C₂₃H₃₀N₂O₂</td>
</tr>
<tr>
<td>FTIR-ATR</td>
<td>+</td>
<td>direct measurement (sample as received)</td>
</tr>
<tr>
<td>FTIR (condensed phase) always as base form</td>
<td>+</td>
<td></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>
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</tr>
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</table>
ANALYTICAL RESULTS

MS (EI)

Abundance

m/z ->

67.1 92.1 175.0 203.1 231.1 265.2 295.1 322.2 366.2

S_1\text{35.1}^{3535} (13.312 \text{ min})$; AKB-57_1521-16.D\text{data.ns}$
FTIR-ATR - direct measurement (sample as received)

IR (condensed phase)
Data File: AKB-57-1521-16_TOF.d
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

Sample Name: ID_1521-16
Position: P1-F3
User Name: TG
Acquired Time: 2/4/2016 1:35:40 PM
DA Method: Drugs_NFL.m

----- End Of Report -----

Compound Table

<table>
<thead>
<tr>
<th>Label</th>
<th>Compound Name</th>
<th>Obs. RT</th>
<th>Obs. Mass</th>
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</thead>
<tbody>
<tr>
<td>Cpd 2: AKB-57</td>
<td>AKB-57</td>
<td>11.827</td>
<td>366.2306</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>AKB-57</td>
<td>733.4684</td>
<td>11.827</td>
<td>366.2306</td>
<td>11.83</td>
<td>C23 H30 N2 O2</td>
<td>366.2307</td>
<td>0.29</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>367.2379</td>
<td>1</td>
<td>6582520.5</td>
<td>(M+H)+</td>
<td></td>
</tr>
<tr>
<td>368.2414</td>
<td>1</td>
<td>1718753.91</td>
<td>(M+H)+</td>
<td></td>
</tr>
<tr>
<td>369.2444</td>
<td>1</td>
<td>221731.49</td>
<td>(M+H)+</td>
<td></td>
</tr>
<tr>
<td>733.4684</td>
<td>1</td>
<td>7818806.5</td>
<td>C23 H30 N2 O2</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>734.4719</td>
<td>1</td>
<td>4135649.75</td>
<td>C23 H30 N2 O2</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>735.4759</td>
<td>1</td>
<td>1138462.55</td>
<td>C23 H30 N2 O2</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>736.4784</td>
<td>1</td>
<td>190574.06</td>
<td>C23 H30 N2 O2</td>
<td>(2M+H)+</td>
</tr>
<tr>
<td>755.4505</td>
<td>1</td>
<td>4890680.5</td>
<td>(2M+Na)+</td>
<td></td>
</tr>
<tr>
<td>756.4538</td>
<td>1</td>
<td>259594.14</td>
<td>(2M+Na)+</td>
<td></td>
</tr>
<tr>
<td>757.4577</td>
<td>1</td>
<td>712630.88</td>
<td>(2M+Na)+</td>
<td></td>
</tr>
</tbody>
</table>

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

### Sample Details
- **Sample Name:** AKB-57_1521-16_IC
- **Injection Volume:** 25.00 µl
- **Injection Type:** Unknown
- **Dilution Factor:** 1.0000
- **Program:** ANIONI
- **Operator:** kemija
- **Injection Date/Time:** 04-feb-2016 / 13:32
- **Run Time:** 42.00 min

### Peak Information

<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 (n.a.)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TOTAL:</td>
<td></td>
<td></td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

### Graph

![Graph showing the integration of peaks with time and concentration](image-url)
## Sample ID:

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1521-16</th>
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</table>

## Our notebook code:

<table>
<thead>
<tr>
<th>Our notebook code:</th>
<th>P-1521-16</th>
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</thead>
</table>

## NMR sample preparation:

<table>
<thead>
<tr>
<th>NMR sample preparation:</th>
<th>15 mg dissolved in 0.7 mL DMSO-d$_6$</th>
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</thead>
</table>

## NMR experiments:

<table>
<thead>
<tr>
<th>NMR experiments:</th>
<th>$^1$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.</th>
</tr>
</thead>
</table>

## Proposed structure:

![Chemical structure](image.png)

## Chemical name:

<table>
<thead>
<tr>
<th>Chemical name:</th>
<th>adamantan-1-yl 1-pentyl-1H-indazole-3-carboxylate</th>
</tr>
</thead>
</table>

## Comments:

| Comments: | - Structure elucidation based on 1D and 2D NMR spectra  
- Sample is pure according to NMR. |
|------------|------------------------------------------------------|

## Supporting information:

<table>
<thead>
<tr>
<th>Supporting information:</th>
<th>Copies of $^1$H and $^{13}$C NMR spectra</th>
</tr>
</thead>
</table>

## Author:

<table>
<thead>
<tr>
<th>Author:</th>
<th>Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc</th>
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</thead>
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## Date of report:

<table>
<thead>
<tr>
<th>Date of report:</th>
<th>February 12, 2016</th>
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P-1521-16

Current Data Parameters
NAME P-1521-16
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160211
Time 21.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010244 sec
RG 2050
DW 16.800 usec
TE 300.0 K
D1 1.00000000 sec
D0 1

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

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

P-1521-16

Current Data Parameters
NAME P-1521-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160211
Time 20.11
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 80.6
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D0 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
PC 1.00

P-1521-16

Current Data Parameters
NAME P-1521-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160211
Time 21.32
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 80.6
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D0 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
PC 1.00