ANALYTICAL REPORT

bk-IMP (C13H17NO)

1-(2,3-dihydro-1H-inden-5-yl)-N-methyl-1-oxopropan-2-amine

Remark – other NPS detected: none

Sample ID: 1867-17
Sample description: powder
Sample type: test purchase /ISF projekt (NFL-SI)
Date of sample receipt (M/D/Y): 10/18/2017
Date of entry (M/D/Y) into NFL database: 11/21/2017
Report updates (if any) will be published here: http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure\(^1\) (base form)

![Structure of bk-IMP](image)

Systematic name 1-(2,3-dihydro-1H-inden-5-yl)-N-methyl-1-oxopropan-2-amine

Other names bk-IMP; 1-(indan-5-yl)-2-(methylamino)propan-1-one

Formula (per base form) C13H17NO

M\(_w\) (g/mol) 203.29

Salt form/anions detected HCl

StdInChIKey (per base form) FHGNUKAARRYOMM-UHFFFAOYSA-N

Other NPS detected none

Additional info (purity..) pure by GC-MS, HPLC-TOF; by NMR 95%

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\(^1\) 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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</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-IR** condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 °C. Chromatographic separation as above (1). Split MS : IR = 1 : 9. 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): 5,5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>BP(1): 58; BP(2): 115, BP(3): 91,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 203,131;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>measured value Δppm: -0,51;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>formula: C₁₃H₁₇NO</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>
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<td></td>
</tr>
<tr>
<td>other</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
ANALYTICAL RESULTS

MS (El)

Abundance

Scan 1106 (5.502 min): bk-Imp-HCl_1867-17 DATA.mz
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 (Diacor R-OD)
TOF REPORT

Data File      bk-IMP_1867-17.d
Sample Name    1867-17
Sample Type    Sample
Instrument Name 6230B TOF LC-MS
Acq Method    general-19_07_2017-XDB-C18-ESI-final.m
IRM Calibration Status Success
Comment        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 3: bk-IMP</td>
<td>bk-IMP</td>
<td>C13 H17 N O</td>
<td>5.87</td>
<td>203.1311</td>
</tr>
</tbody>
</table>

Name | Obs. m/z | Obs. RT | Obs. Mass | DB RT | DB Formula | DB Mass | DB Mass Error (ppm) |
--- | --- | --- | --- | --- | --- | --- | --- |
bk-IMP | 204.1384 | 5.87 | 203.1311 | 5.87 | C13 H17 N O | 203.131 | -0.51 |

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>204.1384</td>
<td>1</td>
<td>20275416</td>
<td>C13 H17 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>205.1417</td>
<td>1</td>
<td>2862023.85</td>
<td>C13 H17 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>206.1451</td>
<td>1</td>
<td>207509.94</td>
<td>C13 H17 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>207.1478</td>
<td>1</td>
<td>8675.18</td>
<td>C13 H17 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>226.1204</td>
<td>1</td>
<td>123712.93</td>
<td>C13 H17 N O</td>
<td>(M+Na)+</td>
</tr>
<tr>
<td>227.1235</td>
<td>1</td>
<td>17743.57</td>
<td>C13 H17 N O</td>
<td>(M+Na)+</td>
</tr>
</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>
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</thead>
<tbody>
<tr>
<td>1.00</td>
<td>9.36</td>
<td>Chloride</td>
<td>BMB</td>
<td>2.49</td>
<td>11.63</td>
<td>n.a.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>TOTAL:</td>
<td>2.49</td>
<td>11.63</td>
</tr>
</tbody>
</table>

**Sample Name:** 1867-17  
**Injection Type:** Unknown  
**Dilution Factor:** 1,0000  
**Injection Date / Time:** 20-okt-2017 / 12:14  
**Program:** ANIONI  
**Operator:** kemija  
**Run Time:** 42.00

---

**Graph:**

[Graph showing peak integration report for Chloride at 9.36 minutes]
**REPORT**

<table>
<thead>
<tr>
<th>Contract No.</th>
<th>C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)</th>
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</thead>
<tbody>
<tr>
<td>Sample ID:</td>
<td><strong>1867-17</strong></td>
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<tr>
<td>Received date:</td>
<td>October 23, 2017</td>
</tr>
<tr>
<td>Our notebook code:</td>
<td>NFL-1867-17</td>
</tr>
<tr>
<td>NMR sample preparation:</td>
<td>15 mg dissolved in 0.7 mL DMSO-&lt;sub&gt;d&lt;/sub&gt;&lt;sup&gt;6&lt;/sup&gt;</td>
</tr>
<tr>
<td>NMR experiments:</td>
<td>&lt;sup&gt;1&lt;/sup&gt;H, &lt;sup&gt;13&lt;/sup&gt;C, &lt;sup&gt;1&lt;/sup&gt;H–&lt;sup&gt;1&lt;/sup&gt;H gs-COSY, &lt;sup&gt;1&lt;/sup&gt;H–&lt;sup&gt;13&lt;/sup&gt;C gs-HSQC, &lt;sup&gt;1&lt;/sup&gt;H–&lt;sup&gt;13&lt;/sup&gt;C gs-HMBC, &lt;sup&gt;1&lt;/sup&gt;H–&lt;sup&gt;15&lt;/sup&gt;N gs-HMBC</td>
</tr>
</tbody>
</table>

**Proposed structure, chemical formula, exact mass, molecular weight:**

![Chemical Structure Image]

Chemical Formula: C_{13}H_{18}NO<sup>+</sup>

Exact Mass: 204,1383

Molecular Weight: 204,2925

**Chemical name:** 1-(2,3-dihydro-1H-inden-5-yl)-N-methyl-1-oxopropan-2-aminium ion

**Comments:** - Structure elucidation based on 1D and 2D NMR spectra.
- Compound is 95% pure by NMR.

**Supporting information:** Copies of <sup>1</sup>H and <sup>13</sup>C NMR spectra

**Authors:** Marko Krivec, Martin Gazvoda, Janez Košmrlj

**Date of report:** November 14, 2017
### Current Data Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>NAME</td>
<td>NFL-1867-17</td>
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<tr>
<td>EXPNO</td>
<td>1</td>
</tr>
<tr>
<td>PROCNO</td>
<td>1</td>
</tr>
</tbody>
</table>

### F2 - Acquisition Parameters

- **Date**: 20171028
- **Time**: 11:10
- **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**: 64
- **DW**: 50.000 usec
- **DE**: 6.50 usec
- **TE**: 296.0 K
- **D1**: 1.00000000 sec
- **TD0**: 1

### CHANNEL f1

- **SFO1**: 500.1330885 MHz
- **NUC1**: 1H
- **P1**: 8.70 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
Current Data Parameters

NAME        NFL-1867-17
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20171028
Time              13.49
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                296.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 8.70 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        0.30046001 W
PLW13        0.15113001 W

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