ANALYTICAL REPORT

Proscaline (C13H21NO3)

2-(3,5-dimethoxy-4-propoxyphenyl)ethan-1-amine

Remark – other NPS detected: none

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1731-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample description:</td>
<td>powder - brown</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>11/14/2016</td>
</tr>
<tr>
<td>Date of entry (M/D/Y) into NFL database:</td>
<td>12/12/2016</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)

![Structure](structure.png)

Systematic name

2-(3,5-dimethoxy-4-propoxyphenyl)ethan-1-amine

Other names

2-(3,5-dimethoxy-4-propoxyphenyl)ethanamine, 4-propoxy-3,5-DMPEA

Formula (per base form)

C13H21NO3

\(M_w\) (g/mol)

239.32

Salt form/anions detected

HCl

StdInChIKey

HYWLMSUAZVDUFUW-UHFFFAOYSA-N

Compound Class

Phenethylamines

Other NPS detected

none

Add.info (purity..)

minor impurities by GC-MS, NMR

---

\(^1\) 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/](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>
<td></td>
</tr>
<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%, 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)
   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 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 (conded (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

### Solubility in

<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>
</tbody>
</table>

### Analytical technique:

<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): 5,93 BP(1): 167; BP(2): 168, BP(3) :210,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 239,1521; measured value Δppm:-0,74;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>formula:C13H21NO3</td>
</tr>
<tr>
<td>FTIR-ATR</td>
<td>+</td>
<td>direct measurement (sample as received)</td>
</tr>
<tr>
<td>GC-IR (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 SWGDRUG.L and ENFSI.L spectra (QM = 98); GC-IR condensed phase match with the spectrum of Proscaline obtained from FSI Zurich, Switzerland (cosine correlation &gt; 0.99).</td>
</tr>
<tr>
<td>other</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
FTIR-ATR - direct measurement (sample as received)

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

**Data File**: Proscaline_1731-16.d  
**Sample Name**: ID_1731-16  
**Sample Type**: Sample  
**Position**: P1-C2  
**Instrument Name**: 6230B TOF LC-MS  
**Acq Method**: general-10_10_2016-XDB-ESI-poz-soft.m  
**User Name**: TG  
**Acquired Time**: 11/16/2016 12:28:24 PM  
**Comment**: extract in MeOH

--- End Of Report ---

### Compound Table

<table>
<thead>
<tr>
<th>Cpd 2: Proscaline</th>
<th>Compound Name</th>
<th>MFG Formula</th>
<th>Obs. RT</th>
<th>Obs. Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Proscaline</td>
<td>C13 H21 N O3</td>
<td>5.87</td>
<td>239.1523</td>
<td></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>Proscaline</td>
<td>240.1596</td>
<td>5.87</td>
<td>239.1523</td>
<td>5.87</td>
<td>C13 H21 N O3</td>
<td>239.1521</td>
<td>-0.74</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>240.1596</td>
<td>1</td>
<td>3015598.5</td>
<td>C13 H21 N O3</td>
<td>[M+H]+</td>
</tr>
<tr>
<td>241.1632</td>
<td>1</td>
<td>401862.77</td>
<td>C13 H21 N O3</td>
<td>[M+H]+</td>
</tr>
<tr>
<td>242.1653</td>
<td>1</td>
<td>40369.67</td>
<td>C13 H21 N O3</td>
<td>[M+H]+</td>
</tr>
<tr>
<td>262.1417</td>
<td>1</td>
<td>10971.78</td>
<td>C13 H21 N O3</td>
<td>[M+Na]+</td>
</tr>
<tr>
<td>479.3117</td>
<td>1</td>
<td>1400294.5</td>
<td>C13 H21 N O3</td>
<td>[2M+H]+</td>
</tr>
<tr>
<td>480.3153</td>
<td>1</td>
<td>383177.18</td>
<td>C13 H21 N O3</td>
<td>[2M+H]+</td>
</tr>
<tr>
<td>481.3172</td>
<td>1</td>
<td>64843.93</td>
<td>C13 H21 N O3</td>
<td>[2M+H]+</td>
</tr>
<tr>
<td>482.3189</td>
<td>1</td>
<td>8563.03</td>
<td>C13 H21 N O3</td>
<td>[2M+H]+</td>
</tr>
<tr>
<td>501.2930</td>
<td>1</td>
<td>25561.17</td>
<td>C13 H21 N O3</td>
<td>[2M+Na]+</td>
</tr>
<tr>
<td>502.2964</td>
<td>1</td>
<td>7927.18</td>
<td>C13 H21 N O3</td>
<td>[2M+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>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>9.83</td>
<td>Chloride</td>
<td>BMB</td>
<td>14.53</td>
<td>52.14</td>
<td>n.a.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>TOTAL: 14.53</td>
<td>52.14</td>
<td>0.00</td>
</tr>
</tbody>
</table>

![Graph of the peak integration report](image-url)
## REPORT

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th><strong>1731-16</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Our notebook code:</td>
<td>P-1731-16</td>
</tr>
<tr>
<td>NMR sample preparation:</td>
<td>15 mg dissolved in 0.7 mL CDCl₃</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>
<tr>
<td>Proposed structure:</td>
<td><img src="image" alt="Chemical structure" /></td>
</tr>
<tr>
<td>Chemical name:</td>
<td>2-(3,5-dimethoxy-4-propoxyphenyl)ethan-1-aminium cation</td>
</tr>
</tbody>
</table>
| Comments:        | - Structure elucidation based on 1D and 2D NMR spectra  
                  - Sample contains a minor amount of impurities, according to the NMR. |
| Supporting information: | Copies of $^1$H and $^{13}$C NMR spectra |
| Author:          | Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc |
| Date of report:  | December 12, 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.
Current Data Parameters
NAME          P-1731-16
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Data_          20161207
Time               22.36
INSTRUM           spect
F2MOD  5 mm PABBO BB-
PULPROG        zg30
TD                65536
SOLVENT           CDCl3
NS                 16
DS                2
SMN           10000.000 Hz
FIDRES          0.152588 Hz
AQ               3.2765000 sec
RG                 57
DW                5.000 usec
TE                298.0 K
DI           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                 1H
P1                8.60 usec
PLW1        26.00000000 W

F2 - Processing parameters
SI                65536
SF          500.1300057 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

Current Data Parameters
NAME          P-1731-16
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Data_          20161208
Time               0.34
INSTRUM           spect
F2MOD  5 mm PABBO BB-
PULPROG        zg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                4
SMN            29761.904 Hz
FIDRES          0.454131 Hz
AQ               1.1010048 sec
RG                2050
DW                16.800 usec
TE                398.3 K
DI           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
PLWX        26.00000000 W
PLWY12        0.3046001 W
PLW13        0.1511301 W

F2 - Processing parameters
SI                32768
SF          125.7577880 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.40