### ANALYTICAL REPORT

**3-MeO-PCE (C15H23NO)**

**N-ethyl-1-(3-methoxyphenyl)cyclohexan-1-amine**

#### Remark – other NPS detected: none

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>1732-16</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>11/14/2016</td>
</tr>
<tr>
<td>Date of entry (M/D/Y) into NFL database:</td>
<td>12/15/2016</td>
</tr>
</tbody>
</table>

#### Substance identified - structure (base form)

![Structure](image)

**Systematic name**  
N-ethyl-1-(3-methoxyphenyl)cyclohexan-1-amine

**Other names**  
3-Methoxyeticyclidine

**Formula (per base form)**  
C15H23NO

**M_w (g/mol)**  
233.36

**Salt form/anions detected**  
HCl

**StdInChIKey**  
OFGOOZLOGUNDFS-UHFFFAOYSA-N

**Compound Class**  
Arylcyclohexylamines

**Other NPS detected**  
none

**Add.info (purity..)**  
pure by GC-MS, HPLC-TOF, 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
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)
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>soluble</td>
</tr>
<tr>
<td>H₂O</td>
<td>soluble</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): 5,83&lt;br&gt;BP(1): 190; BP(2): 233; BP(3): 176,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 233.178; measured value Δppm: -1.72; formula: C₁₅H₂₃N₂O</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 ENFSI.L 2016 and SWGDRUG.L</td>
</tr>
<tr>
<td>other</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
ANALYTICAL RESULTS

MS (EI)

Scan 1205 (5.826 min): 3-MeO-PCE_1732-16.D.dat

Abundance

m/z ->
FTIR-ATR - direct measurement (sample as received)

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

**Data File**: 3-MeO_PCE_1732-16.d  
**Sample Name**: ID_1732-16  
**Sample Type**: Sample  
**Position**: P1-C3  
**Instrument Name**: 6230B TOF LC-MS  
**User Name**: TG  
**Acq Method**: general-10_10_2016-XDB-C18-ESI-poz-soft.m  
**Acquired Time**: 11/17/2016 12:27:57 PM  
**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 3: 3-MeO-PCE</td>
<td>3-MeO-PCE</td>
<td>C15 H23 N O</td>
<td>6.44</td>
<td>233.1784</td>
</tr>
</tbody>
</table>

### Compound Chromatograms

- **Obs. m/z**: 234.1856, 235.1891, 236.1923, 237.1950  
- **Obs. RT**: 6.44  
- **DB RT**: 6.44  
- **DB Formula**: C15 H23 N O  
- **DB Mass**: 233.178  
- **DB Mass Error (ppm)**: -1.72

### MFE MS Zoomed Spectrum

Cpd 3: 3-MeO-PCE: +ESI EIC(234.1857, 235.1889, ...)  

### MS Zoomed Spectrum

Cpd 3: 3-MeO-PCE: +ESI Scan (rt: 6.38-7.11 min, 58 scans)  

### 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>234.1856</td>
<td>1</td>
<td>19705530</td>
<td>C15 H23 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>235.1891</td>
<td>1</td>
<td>3216222.09</td>
<td>C15 H23 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>236.1923</td>
<td>1</td>
<td>256631.02</td>
<td>C15 H23 N O</td>
<td>(M+H)+</td>
</tr>
<tr>
<td>237.1951</td>
<td>1</td>
<td>18567.63</td>
<td>C15 H23 N O</td>
<td>(M+H)+</td>
</tr>
</tbody>
</table>

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

Sample Name: ID-1732-16
Injection Type: Unknown
Program: ANIONI
Inj. Date / Time: 16-nov-2016 / 14:45

<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>9.86</td>
<td>Chloride</td>
<td>BMB</td>
<td>15.94</td>
<td>57.02</td>
<td>n.a.</td>
</tr>
</tbody>
</table>

TOTAL:

15.94  57.02  0.00

No.  Time  Peak Name  Peak Type  Area  Height  Amount
  1    9.86 Chloride   BMB        15.94  57.02  n.a.

TOTAL:

15.94  57.02  0.00

Anions-report template/Integration

Chromeleon (c) Dionex 1996-2009
Version 7.2.0.3765
REPORT

Sample ID: 1732-16
Our notebook code: P-1732-16
NMR sample preparation: 15 mg dissolved in 0.7 mL CDCl₃
NMR experiments: ¹H, ¹³C, ¹H-¹H gs-COSY, ¹H-¹³C gs-HSQC, ¹H-¹³C gs-HMBC, ¹H-¹⁵N gs-HMBC.

Proposed structure:

![Proposed structure diagram]

Chemical name: N-ethyl-1-(3-methoxyphenyl)cyclohexan-1-aminium cation
Comments: - Structure elucidation based on 1D and 2D NMR spectra
- Sample is pure as evident by NMR.

Supporting information: Copies of ¹H and ¹³C NMR spectra
Author: Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report: December 13, 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.