ANALYTICAL REPORT

2-MeO-diphenidine (C20H25NO)
1-(1-(2-methoxyphenyl)-2-phenylethyl)piperidine

Remark – other NPS detected: none

Sample ID: 1195-15
Sample description: powder - white
Sample type: test purchase 7/22/2015
Comments:\nDate of entry into NFL database: 10/14/2015

Substance identified-structure\(^2\) (base form)

Systematic name 1-(1-(2-methoxyphenyl)-2-phenylethyl)piperidine

Other names

Formula (per base form) C20H25NO
\(M_w\) (g/mol) 295.42
Salt form HCl
StdInChIKey QXXCUXIRBSITD-UHFFFAOYSA-N

Compound Class Others

Other NPS detected none

Add.info (purity..) NMR: sample contains 7% to 8 % MeoH, GC and HPLC pure

\(^1\) 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
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, then heating at 50 °C/min up to 325 °C and finally 2.8 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 (40) to 550 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 AIS 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 (40) to 550 amu.
   IR (condensed phase): IR scan range 4000 to 650, resolution 4 cm⁻¹.
### 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>other</td>
<td>not tested</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): 8,25  
|                      |         | BP(1): 204; BP(2): 188; BP(3): 91, |
| HPLC-TOF           | +       | Exact mass (theoretical): 295,1936;  
|                     |         | measured value Δppm: -0,22;  
|                     |         | formula: C₂₀H₂₅NO |
| FTIR-ATR          | +       | direct measurement (note sample contains MeOH) |
| FTIR (condensed phase) always as base form | + | |
| NMR                | +       | validation |
| other              |         |         |
ANALYTICAL RESULTS

MS (EI)

Abundance

Scan 551 (8.246 min) 2MeO-diphenidline_1195-15_MS.D\data.ms

m/z->

40 60 80 100 120 140 160 180 200 220 240 260 280 300

1000000 2000000 3000000 4000000 5000000 6000000 7000000 8000000

65.0 91.0 121.0 152.0 170.0 222.0 294.1 294.1
FTIR-ATR - direct measurement (note! HCl form & sample contains 7% to 8% MeOH)

IR (condensed phase)
Target Compound Screening Report

Data File 2MeO-diphenidine_1195-15_TOF.d
Sample Type Sample
Instrument Name 6230B TOF LC-MS
Acq Method droge general-13-5-2015-XDB-ESI-poz.m
Acquired Time 7/27/2015 11:23:48 AM
IRM Calibration Status Success
DA Method Droge_Default.m
Comment extract in MeOH

Sample Name 2MeO-diphenidine
Position P1-D5
User Name TG

MFE MS Zoomed Spectrum

Compound Table

<table>
<thead>
<tr>
<th>Label</th>
<th>Tgt Name</th>
<th>Obs. RT</th>
<th>Obs. Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cpd 5: 2MeO-Diphenidine</td>
<td>2MeO-Diphenidine</td>
<td>6.545</td>
<td>295.1934</td>
</tr>
</tbody>
</table>

Name | Obs. m/z | Obs. RT | Obs. Mass | DB RT | DB Formula | DB Mass | DB Mass Error (ppm) | Find Cpd Method |
<table>
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<tr>
<th></th>
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</thead>
<tbody>
<tr>
<td>2MeO-Diphenidine</td>
<td>296.2009</td>
<td>6.545</td>
<td>295.1937</td>
<td>6.545</td>
<td>C20 H25 N O</td>
<td>295.1936</td>
<td>-0.22</td>
<td>Find by Molecular Feature</td>
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</tbody>
</table>

Compound Chromatograms

MFE 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>
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</thead>
<tbody>
<tr>
<td>296.2009</td>
<td>1</td>
<td>15138817</td>
<td>C20 H25 N O</td>
<td>M+H+</td>
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<tr>
<td>297.2044</td>
<td>1</td>
<td>3492598.58</td>
<td>C20 H25 N O</td>
<td>M+H+</td>
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<tr>
<td>298.2077</td>
<td>1</td>
<td>364991.07</td>
<td>C20 H25 N O</td>
<td>M+H+</td>
</tr>
<tr>
<td>299.2103</td>
<td>1</td>
<td>29507.91</td>
<td>C20 H25 N O</td>
<td>M+H+</td>
</tr>
<tr>
<td>300.2126</td>
<td>1</td>
<td>299.47</td>
<td>C20 H25 N O</td>
<td>M+H+</td>
</tr>
</tbody>
</table>

MS Zoomed Spectrum
Cpd 5: 2MeO-Diphenidine: +ESI Scan (rt: 6.498-7.178 min, 54 scans) Frag=175.0V 2MeO-diphenid...
Sample ID: 1195-15

Our notebook code: P-1195-15

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 with chemical name:

![Chemical structure](image)

1-(1-(2-methoxyphenyl)-2-phenylethyl)piperidin-1-ium

Comments: - Structure elucidation based on 1D and 2D NMR spectra
- Compound is not pure by NMR, containing approx. 7–8% of methanol (signals in ¹H NMR at 3.49 and 1.30 and in ¹³C NMR at 50.77), degrading the quality of spectra.

Supporting information: Copies of ¹H and ¹³C NMR spectra

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

Date of report: October 12, 2015

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