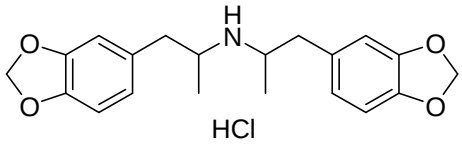
## CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

**Report ID: D896.2012.02**

Compound Name: ***N, N*-Di[1-(3,4-Methylenedioxyphenyl)-2-propyl]amine hydrochloride**
Description: White solid
Collection Number: D896 Batch Number: 05-D-13
Chemical Formula: $C_{20}H_{23}NO_4$.HCl Molecular Weight: 377.9 (341.4 base)
CAS Number: 67668-98-8 (base) Release date: 20th September 2005
Structure:




Synonyms: *N*-[2-(1,3-Benzodioxol-5-yl)-1-methylethyl]-α-methyl-1,3-benzodioxole-5-ethanamine HCl

Purity (mass fraction): 99.3 ± 1.3% (95% coverage interval)

The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and $^1$H NMR. Supporting evidence is provided by elemental microanalysis.

GC-FID:
- Instrument: Agilent 6890N
- Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
- Program: 180 °C (1 min), 30 °C/min to 250 °C (5 min), 30 °C/min to 300 °C (3 min)
- Injector: 250 °C Detector Temp: 320 °C
- Carrier: Helium Split ratio: 20/1

Relative peak area response of main component:
- Initial analysis: Mean = 99.5%, s = 0.02% (7 sub-samples in duplicate, August 2005)
- Re-analysis: Mean = 99.5%, s = 0.01% (5 sub-samples in duplicate, November 2007)
- Re-analysis: Mean = 99.5%, s = 0.01% (5 sub-samples in duplicate, November 2012)

Thermogravimetric analysis: Volatile content < 0.1% mass fraction. Non volatile residue was not determined (August 2005, November 2006 and December 2007)

Karl Fischer analysis: Moisture content < 0.3% mass fraction (November 2012)

**Spectroscopic and other characterisation data**

| | | | |
|---|---|---|---|
| GC-MS: | Instrument: | HP5890/5971A | |
| | Column: | ZB-5, 30 m × 0.25 mm I.D. × 0.20 μm | |
| | Program: | 220 °C (2 min), 10 °C/min to 290 °C (5 min), 10 °C/min to 300 °C | |
| | Injector: | 250 °C | Transfer line temp: 280 °C |
| | Carrier: | Helium, 1.0 ml/min | Split ratio: 20/1 |
| | The retention time of the free base is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.<br>8.9 min: 206 (98), 163 (100), 135 (48), 105 (33), 77 (27), 70 (14), 51 (11) m/z | | |
| TLC: | Conditions: | Kieselgel $60F_{254}$. Methanol/Conc $NH_3$ (200:3)<br>Single spot observed, $R_f$ = 0.69. Visualisation with UV at 254 nm. | |
| IR: | Instrument: | Biorad FTS300MX FT-IR | |
| | Range: | 4000-400$cm^{-1}$, KBr pellet | |
| | Peaks: | 2973, 2819, 2788, 2754, 2485, 2461, 2046, 1848, 1607, 1493, 1442, 1250, 1042, 933, 862, 806, 778 $cm^{-1}$ | |
| $^1$H NMR: | Instrument: | Bruker DMX-500 | |
| | Field strength: | 500 MHz | Solvent: $CD_3OD$ |
| | Spectral data: | δ 1.25 (6H, d, *J* = 6.6 Hz), 2.65 (2H, dd, *J* = 9.3, 13.4 Hz), 3.05 (2H, dd, *J* = 5.2, 13.6 Hz), 3.51 (2H, m), 5.94 (4H, s), 6.69 (2H, dd, *J* = 1.7, 8.0 Hz), 6.74 (2H, d, *J* = 1.6 Hz), 6.78 (2H, d, *J* = 8.0 Hz) ppm | |
| $^{13}$C NMR: | Instrument: | Bruker DMX-500 | |
| | Field strength: | 125 MHz | Solvent: $CD_3OD$ |
| | Spectral data: | δ 16.9, 40.2, 54.7, 102.6, 109.5, 110.3, 123.6, 130.8, 148.4, 149.6 ppm | |
| Melting point: | | 261-263 °C | |
| Microanalysis: | | Found: C = 63.3%; H = 6.3%; N = 3.7%<br>Calc: C = 63.6%; H = 6.4%; N = 3.7% Calculated for $C_{20}H_{23}NO_4$.HCl) | |

**Expiration of certification**
The property values are valid till 15th November 2017 i.e. five years from the date of re-certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body.

The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of five years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

**Homogeneity assessment**
The homogeneity of the material was assessed using purity assay by GC-FID on ten randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

**Metrological traceability**
The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis.

**Recommended storage**
When not in use this material should be stored at or below 25 ºC in a closed container in a dry, dark area.

**Intended use**
For ***in vitro*** laboratory analysis only.

**Caution**
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

**Legal notice**
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 20 June, 2017.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 20th June 2017.