



**Australian Government**  
**National Measurement Institute**

**CERTIFIED REFERENCE MATERIAL**  
**CERTIFICATE OF ANALYSIS**

**Report ID: D758.2013.01**

Compound Name: **4-Bromo-2,5-dimethoxyphenethylamine. HCl**

Description: White powder

Collection Number: D758

Chemical Formula:  $C_{10}H_{14}BrNO_2 \cdot HCl$

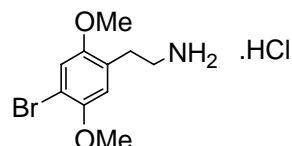
CAS Number: 56281-37-9 (HCl)

Structure:

Batch Number: 02-D-33

Molecular Weight: 296.6 (HCl), 260.1 (base)

Release Date: 30<sup>th</sup> November 2002



Names: (±)-4-Bromo-2,5-dimethoxybenzeneethamine. HCl  
nexus  
MFT  
BDMPEA

Purity (mass fraction):  $99.1 \pm 0.4$  % (95 % coverage interval)

Purity estimate obtained from a combination of traditional analytical techniques. 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 <sup>1</sup>H NMR. Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 6890N  
Column: HP-1 30 m × 0.32mm × 0.25 μm  
Program: 100 °C (2 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)  
Injector: 250 °C Detector Temp: 320 °C  
Carrier: Helium Split ratio: 20/1  
Initial analysis: 99.7 %, s = 0.04% (10 samples in duplicate, November 2002)  
Re-analysis: 99.5 %, s = 0.01% (5 sub samples in duplicate, September 2010)  
Re-analysis: 99.5 %, s = 0.01% (5 sub samples in duplicate, July 2013)

GC-FID: Instrument: Varian CP-3800  
Column: VF-1, 30 m x 0.32 mm I.D. x 0.25 μm  
Program: 100 °C (2 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)  
Injector: 250 °C Detector Temp: 320 °C  
Carrier: Helium Split ratio: 20/1  
Initial analysis: 99.7 %, s = 0.04% (10 samples in duplicate, November 2002)  
Re-analysis: 99.4 %, s = 0.05% (5 sub samples in duplicate, September 2007)

Thermogravimetric analysis: Volatile and non volatile content < 0.3 % total (2002)

Karl Fischer analysis: Moisture content 0.2% mass fraction (September 2007)  
Moisture content < 0.1% mass fraction (September 2010 & July 2013)

GC-MS: Instrument: Agilent 6890/5973  
 Column: Zebron ZB-5, 30 m x 0.25 mm I.D. x 0.30  $\mu$ m  
 Program: 130 °C (1 min), 20 °C/min to 250 °C (5 min) 30 °C/min to 300 °C (2 min)  
 Injector: 250 °C Transfer line temp: 280 °C  
 Carrier: Helium, 1.0 ml/min Split ratio: 20/1  
 The retention times of the free base is reported along with the major peaks in the mass spectrum. The latter are reported as mass to charge ratios (m/z) and (in brackets) as a percentage relative to the base peak.  
 6.7 min): 261 (M<sup>+</sup>, 11), 259 (M<sup>+</sup>, 11) 232 (97), 230 (100), 217 (25), 215 (26), 201 (11), 199 (10), 105 (16), 91, (16), 77 (30), 63 (12), 53 (12), 51 (11) m/z

TLC: Conditions: Kieselgel 60F<sub>254</sub>. methanol/conc NH<sub>3</sub> (200/3)  
 Single spot, R<sub>f</sub> = 0.26. visualisation with UV light (254 nm)

IR: Instrument: FT-IR, Biorad FTS3000MX  
 Range: 4000-400 cm<sup>-1</sup>, KBr  
 Peaks: 2938, 2900, 2699, 2606, 2444, 2364, 2038, 1606, 1500, 1490, 1435, 1389, 1305, 1211, 1117, 1049, 1029, 848, 775, 706 cm<sup>-1</sup>

<sup>1</sup>H NMR: Instrument: Bruker DMX600  
 Field strength: 600 MHz Solvent: CD<sub>3</sub>OD (3.3 ppm)  
 Spectral data:  $\delta$  2.94 (2H, t, *J* = 7.26 Hz), 3.13 (2H, t, *J* = 7.26 Hz), 3.81 (3H, s), 3.83 (3H, s), 6.95 (1H, s), 7.17 (1H, s) ppm

<sup>13</sup>C NMR: Instrument: Bruker DMX600  
 Field strength: 151 MHz Solvent: CD<sub>3</sub>OD (49 ppm)  
 Spectral data:  $\delta$  29.7, 40.6, 56.6, 57.4, 111.4, 116.3, 117.2, 126.2, 151.7, 153.4 ppm

Microanalysis: Found: C = 40.8%, H = 5.0%, N = 4.6% (October 2002)  
 Calc: C = 40.5%, H = 5.1%, N = 4.7% (Calculated for C<sub>10</sub>H<sub>14</sub>BrNO<sub>2</sub>.HCl)

Melting point: 240–241°C

**Expiration of certification**

The property values are valid till 19<sup>th</sup> July 2016, 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% confidence 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 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 20 °C 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: 29 July, 2013

Characterisation data and certified property values specified in this report supercede those in all reports issued prior to 29<sup>th</sup> July 2013



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