



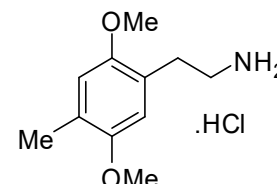
## CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

### NMIA D1009: 2,5-Dimethoxy-4-methylphenethylamine hydrochloride

Report ID: D1009.2026.02

Chemical Formula:  $C_{11}H_{17}ClNO_2 \cdot HCl$

Molecular Weight: 231.7 g/mol (HCl), 195.3 g/mol (base)



### Certified value

| Batch No. | CAS No.                               | Purity (mass fraction) |
|-----------|---------------------------------------|------------------------|
| 14-D-01   | 25505-65-1 (HCl)<br>24333-19-5 (base) | 99.8 ± 0.4%            |

The uncertainty has been calculated according to ISO 33405 and is stated at the 95% confidence limit ( $k = 2$ ).

**IUPAC name:** 2-(2,5-Dimethoxy-4-methylphenyl)ethanamine hydrochloride

**Expiration of certification:** The property values are valid till 25 June 2036, ten 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 material will be re-tested periodically to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

**Description:** Off-white powder prepared by synthesis and certified for identity and purity by NMI Australia. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top cap.

**Intended use:** This certified reference material is suitable for use as a primary calibrator.

**Instructions for use:** Equilibrate the bottled material to room temperature before opening.

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

**Metrological traceability:** The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%.

**Stability:** This material has demonstrated stability over a minimum period of ten years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. The long-term stability of the compound in solution has not been examined.

**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.

**Safety:** Treat as a hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,  
Team Leader,  
Chemical Reference Materials, NMI.  
14 July 2026

This report supersedes any issued prior to 14 July 2026.

NATA Accreditation No. 198 / Corporate Site No. 14214.

**Legal notice:** Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

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## Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and <sup>1</sup>H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100 \% - I_{\text{ORG}}) \times (100 \% - I_{\text{VOL}} - I_{\text{NVR}})$$

Equation 1

$I_{\text{ORG}}$  = Organic impurities of related structure,  $I_{\text{VOL}}$  = volatile impurities,  $I_{\text{NVR}}$  = non-volatile residue.

Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 8890 or 7890 or 6890  
 Column: HP-1MS or HP-1, 30 m × 0.32 mm I.D. × 0.25 μm  
 Program: 100 °C (1 min), 10 °C/min to 200 °C, 20 °C/min to 300 °C (5 min)  
 Or 100 °C (1 min), 10 °C/min to 200 °C, 30 °C/min to 300 °C (3 min) (2015)  
 Injector: 250 °C Detector Temp: 320 °C  
 Carrier: Helium Split ratio: 20/1  
 Relative mass fraction of the main component as the free base:  
 Initial analysis: Mean = 99.9%, s = 0.08% (10 sub samples in duplicate, February 2014)  
 Re-analysis: Mean = 99.9%, s = 0.15% (5 sub samples in duplicate, February 2015)  
 Re-analysis: Mean = 99.6%, s = 0.07% (5 sub samples in duplicate, February 2016)  
 Re-analysis: Mean = 99.8%, s = 0.003% (5 sub samples in duplicate, February 2017)  
 Re-analysis: Mean = 100%, s = 0.04% (5 sub samples in duplicate, February 2020)  
 Re-analysis: Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, June 2026)

GC-FID: Instrument: Agilent 7890  
 Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm  
 Program: 100 °C (1 min), 10 °C/min to 200 °C, 20 °C/min to 300 °C (5 min)  
 Injector: 250 °C Detector Temp: 320 °C  
 Carrier: Helium Split ratio: 20/1  
 Relative mass fraction of the main component as the free base:  
 Initial analysis: Mean = 99.9%, s = 0.1% (10 sub samples in duplicate, February 2014)

Karl Fischer analysis: Moisture content ≤ 0.3% mass fraction (February 2014, 2015, 2016, and 2017)  
 Moisture content ≤ 0.1% mass fraction (February 2020 and June 2026)

Thermogravimetric analysis: Non-volatile residue < 0.2% mass fraction (February 2014). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.

## Spectroscopic and other characterisation data

|                      |                                                                                                                                                                                                      |                                                                                                                                                     |
|----------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------|
| GC-MS:               | Parent compound:                                                                                                                                                                                     |                                                                                                                                                     |
|                      | Instrument:                                                                                                                                                                                          | HP6890/5973                                                                                                                                         |
|                      | Column:                                                                                                                                                                                              | TG-1MS, 30 m x 0.25 mm I.D. x 0.25 $\mu$ m                                                                                                          |
|                      | Program:                                                                                                                                                                                             | 100 °C (1 min), 10 °C/min to 200 °C, 20 °C/min to 300 °C (5 min)                                                                                    |
|                      | Injector:                                                                                                                                                                                            | 250 °C                                                                                                                                              |
|                      | Split ratio:                                                                                                                                                                                         | 20/1                                                                                                                                                |
|                      | Transfer line temp:                                                                                                                                                                                  | 280 °C                                                                                                                                              |
|                      | Carrier:                                                                                                                                                                                             | Helium                                                                                                                                              |
|                      | Scan range:                                                                                                                                                                                          | 50-550 <i>m/z</i>                                                                                                                                   |
|                      | The retention time of the free base is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak. |                                                                                                                                                     |
|                      | Free base (9.43 min):                                                                                                                                                                                | 195 ( $M^+$ , 22), 166 (100), 165 (20), 151 (64), 135 (25), 91 (16), 77 (10) <i>m/z</i>                                                             |
| TLC:                 | Conditions:                                                                                                                                                                                          | Kieselgel 60F <sub>254</sub> . Diethyl ether/TBME /diethylamine (45/45/10)                                                                          |
|                      | Single spot observed, $R_f$ = 0.3                                                                                                                                                                    |                                                                                                                                                     |
| IR:                  | Instrument:                                                                                                                                                                                          | Biorad FTS3000MX FT-IR                                                                                                                              |
|                      | Range:                                                                                                                                                                                               | 4000-400 $\text{cm}^{-1}$ , KBr powder                                                                                                              |
|                      | Peaks:                                                                                                                                                                                               | 3002, 2936, 2901, 2840, 2742, 2697, 2656, 2603, 2560, 2499, 2455 2036, 1605, 1512, 1464, 1402, 1314, 1211, 1045, 846, 804, 641 $\text{cm}^{-1}$     |
| <sup>1</sup> H NMR:  | Instrument:                                                                                                                                                                                          | Bruker Avance III-500                                                                                                                               |
|                      | Field strength:                                                                                                                                                                                      | 600 MHz                                                                                                                                             |
|                      | Solvent:                                                                                                                                                                                             | MeOH- <i>d</i> <sub>4</sub> (3.31 ppm)                                                                                                              |
|                      | Spectral data:                                                                                                                                                                                       | $\delta$ 2.18 (3H, s), 2.93 (2H, t, <i>J</i> = 7.2 Hz), 3.12 (2H, t, <i>J</i> = 7.2 Hz), 3.78 (3H, s), 3.80 (3H, s), 6.78 (1H, s), 6.81 (1H, s) ppm |
| <sup>13</sup> C NMR: | Instrument:                                                                                                                                                                                          | Bruker Avance III-500                                                                                                                               |
|                      | Field strength:                                                                                                                                                                                      | 151 MHz                                                                                                                                             |
|                      | Solvent:                                                                                                                                                                                             | MeOH- <i>d</i> <sub>4</sub> (49 ppm)                                                                                                                |
|                      | Spectral data:                                                                                                                                                                                       | $\delta$ 16.3, 29.8, 41.1, 56.3, 56.5, 114.2, 114.9, 123.4, 127.6, 152.6, 153.2 ppm                                                                 |
| Melting point:       | 212-214 °C                                                                                                                                                                                           |                                                                                                                                                     |
| Microanalysis:       | Found:                                                                                                                                                                                               | C = 57.1%; H = 8.0%; N = 6.1%; Cl% = 15.2% (February 2014)                                                                                          |
|                      | Calculated:                                                                                                                                                                                          | C = 57.0%; H = 7.8%; N = 6.0%; Cl% = 15.3% (Calculated for C <sub>11</sub> H <sub>17</sub> NO <sub>2</sub> .HCl)                                    |