



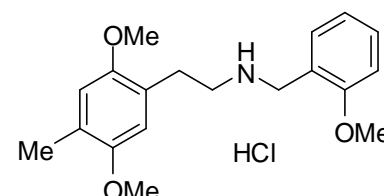
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D1010: 2-(2,5-Dimethoxy-4-methylphenyl)-N-(2-methoxybenzyl)ethanamine hydrochloride

Report ID: D1010.2025.01

Chemical Formula: C<sub>19</sub>H<sub>25</sub>NO<sub>3</sub>·HCl

Molecular Weight: 351.9 g/mol (HCl), 315.4 g/mol (base)



### Certified value

Batch No.	CAS No.	Purity estimate
14-D-02	1539266-35-7 (HCl) 1354632-02-2 (free base)	98.1 ± 0.7%

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

**Expiration of certification:** The property values are valid till 4 February 2035, 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.

**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 all impurities are quantified as a mass fraction and subtracted from 100%.

**Stability:** This material has demonstrated stability over a minimum period of five years.  
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.  
10 February 2025

This report supersedes any issued prior to 10 February 2025.

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 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.

$$\text{Purity} = (100\% - I_{\text{ORG}}) \times (100\% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

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

Supporting evidence is provided by elemental microanalysis.

**Warning:** This material is sensitive to the quality of the injection liner and has demonstrated degradation up to 1.2% under the GC conditions detailed below.

GC-FID:	Instrument:	Agilent 6890 or 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm (2014 - 2017) HP-5, 30 m × 0.32 mm I.D. × 0.25 μm (2021 - 2025)
	Program:	190 °C (1 min), 6 °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 = 99.8%, s = 0.08% (10 sub samples in duplicate, April 2014)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, April 2015)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, March 2016)
	Re-analysis:	Mean = 99.8%, s = 0.07% (5 sub samples in duplicate, March 2017)
	Re-analysis:	Mean = 99.7%, s = 0.04% (5 sub samples in duplicate, March 2021)
	Re-analysis:	Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, February 2025)

Karl Fischer analysis:	Moisture content 1.4% mass fraction (April 2014)
	Moisture content 1.9% mass fraction (April 2015)
	Moisture content 2.0% mass fraction (March 2016 and 2017)
	Moisture content 1.7% mass fraction (March 2021)
	Moisture content 1.5% mass fraction (March 2025)

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

GC-MS:	Instrument:	HP6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 $\mu$ m
	Program:	190 $^{\circ}$ C (1 min), 6 $^{\circ}$ C/min to 300 $^{\circ}$ C (5.67 min)
	Injector:	250 $^{\circ}$ C
	Split ratio:	20/1
	Transfer line temp:	280 $^{\circ}$ C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 $m/z$
	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 (11.1 min):	282 (8), 166 (24), 150 (35), 121 (100), 91 (28) $m/z$
	ESI-MS:	Instrument: Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 $\mu$ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	316.2 (M+H <sup>+</sup> ) $m/z$
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Hexane/ethyl acetate/diethyl amine (16/4/0.5) Single spot observed, R <sub>f</sub> = 0.2
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 $\text{cm}^{-1}$ , KBr powder
	Peaks:	2925, 2828, 2781, 2680, 2641, 2484, 1601, 1587, 1509, 1496, 1466, 1457, 1439, 1402, 1257, 1215, 1051, 1034, 770 $\text{cm}^{-1}$
<sup>1</sup> H NMR:	Instrument:	Bruker Avance 400
	Field strength:	400 MHz
	Solvent:	DMSO- <i>d</i> <sub>6</sub> (2.50 ppm)
	Spectral data:	$\delta$ 2.12 (3H, s), 2.95-3.02 (4H, m), 3.70 (3H, s), 3.72 (3H, s), 3.82 (3H, s), 4.11 (2H, t, <i>J</i> = 5.6 Hz), 6.77 (1H, s), 6.82 (1H, s), 6.99 (1H, dt, <i>J</i> = 1.2, 7.2 Hz), 7.08 (1H, d, <i>J</i> = 8 Hz), 7.40 (1H, m), 7.51 (1H, dd, <i>J</i> = 1.6, 7.6 Hz), 9.31 (2H, s) ppm
<sup>13</sup> C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	101 MHz
	Solvent:	DMSO- <i>d</i> <sub>6</sub> (39.52 ppm)
	Spectral data:	$\delta$ 16.1, 26.3, 44.6, 46.1, 55.6, 55.7, 55.9, 111.1, 112.8, 114.0, 119.7, 120.4, 122.6, 124.9, 130.7, 131.5, 150.6, 151.1, 157.5 ppm
Melting point:		159-164 $^{\circ}$ C
Microanalysis:	Found:	C = 63.7%; H = 7.5%; N = 3.9%; Cl% = 10.1% (April, 2014)
	Calculated:	C = 63.9%; H = 7.5%; N = 3.9%; Cl% = 9.9% (Calculated for C <sub>13</sub> H <sub>17</sub> NO <sub>2</sub> .HCl+ 1.4% water)