



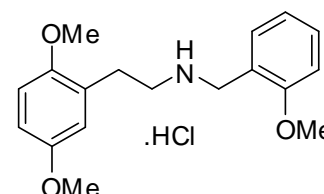
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D998: 2-(2,5-Dimethoxyphenyl)-N-[(2-methoxybenzyl)]ethylamine hydrochloride

Report ID: D998.2021.02

Chemical Formula:  $C_{18}H_{23}NO_3 \cdot HCl$

Molecular Weight: 337.8 g/mol (HCl), 301.4 g/mol (base)



### Certified value

Batch No.	CAS No.	Purity (mass fraction)
13-D-14	919797-16-3 (base)	98.8 ± 1.1%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit ( $k = 2$ ).

**Synonyms:** 25H-NBOMe  
2,5-Dimethoxy-N-[(2-methoxyphenyl)methyl]benzenethanamine hydrochloride  
2-(2,5-Dimethoxyphenyl)-N-[(2-methoxyphenyl)methyl]ethanamine hydrochlorides

**Expiration of certification:** The property values are valid till 7 December 2026, 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.

**Description:** White crystals prepared by synthesis, and certified for identity and purity by NMIA. 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

**Stability:** 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. 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.  
21 September 2022

This report supersedes any issued prior to 21 September 2022.

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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 and Agilent 8890
	Column:	VF-1MS or HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 240 °C (3 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.6%, s = 0.07% (10 sub samples in duplicate, July 2013) [VF-1]
	Initial analysis:	Mean = 99.2%, s = 0.08% (10 sub samples in duplicate, June 2013) [HP-5]
	Re-analysis:	Mean = 99.5%, s = 0.03% (7 sub samples in duplicate, June 2014)
	Re-analysis:	Mean = 99.6%, s = 0.05% (5 sub samples in duplicate, June 2015)
	Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, May 2016)
HPLC:	Re-analysis:	Mean = 98.9%, s = 0.2% (5 sub samples in duplicate, April 2019)
	Re-analysis:	Mean = 99.3%, s = 0.03% (5 sub samples in duplicate, December 2021)
	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Waters Xbridge C-18, 2.7 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/MilliQ water (50:50)
		The aqueous phase was buffered at pH 10.8 using 20mM NH <sub>4</sub> OAc
	Flow rate:	1.0 mL/min
	Detector:	SPD-M20A PDA operating at 210 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.5%, s = 0.03% (7 sub samples in duplicate, July 2013)
	Karl Fischer analysis:	Moisture content < 0.4% mass fraction (July 2013, June 2014, June 2015, May 2016, May 2019 and November 2021)
	Thermogravimetric analysis:	Non volatile residue < 0.2% mass fraction (July 2013). 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:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	60 °C (1 min), 15 °C/min to 280 °C (6 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 <i>m/z</i>
The retention times of the parent compound are 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.		
Parent (15.5 min): 270 (5), 207 (5), 150 (46), 121 (100), 91 (28), 77 (5), 65 (5) <i>m/z</i>		
LC/ESI-MS:	Instrument:	Waters Acquity UPLC/TQD
	Operation:	Positive ion mode, direct infusion at 5 µL/min
	Ionisation:	ESI spray voltage at 3.0 kV positive ion
	Capillary voltage:	3.5 kV
	Cone voltage:	30 V
	Peak:	302.2 (M+H <sup>+</sup> ) <i>m/z</i>
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 µm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	<i>t</i> -Butylmethyl ether
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . NH <sub>3</sub> /methanol (3/200) Single spot observed, R <sub>f</sub> = 0.63 Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm <sup>-1</sup> , KBr powder
	Peaks:	2935, 2834, 2763, 2684, 1589, 1501, 1458, 1298, 1223, 1038, 934, 854, 814, 769, 703 cm <sup>-1</sup>
<sup>1</sup> H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	DMSO (2.50 ppm)
	Spectral data:	δ 2.95-3.05 (4H, m), 3.69 (3H, s), 3.71 (3H, s), 3.83 (3H, s), 4.11 (2H, s), 6.78 (1H, s), 6.79 (1H, dd, <i>J</i> = 3.1, 10.0 Hz), 6.90 (1H, m), 6.98 (1H, dt, <i>J</i> = 0.9, 7.5 Hz), 7.07 (1H, dd, <i>J</i> = 0.7, 8.5 Hz), 7.40 (1H, ddd, <i>J</i> = 1.7, 7.5, 8.3 Hz), 7.52 (1H, dd, <i>J</i> = 1.6, 7.5 Hz) ppm <i>t</i> -Butylmethyl ether estimated at 0.03%, ethanol estimated at 0.1% and acetic acid estimated at 0.1% mass fraction were observed in the <sup>1</sup> H NMR.
<sup>13</sup> C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	101 MHz
	Solvent:	DMSO (39.5 ppm)
	Spectral data:	δ 26.4, 44.6, 45.9, 55.4, 55.6, 55.8, 111.1, 111.8, 112.3, 116.2, 119.7, 120.3, 126.1, 130.7, 131.5, 151.2, 153.1, 157.5 ppm
Melting point:		118-120 °C
Microanalysis:	Found:	C = 64.2%; H = 7.3%; N = 4.2%; Cl% = 10.3% (July, 2013)
	Calculated:	C = 64.0%; H = 7.2%; N = 4.2%; Cl% = 10.5% (Calculated for C <sub>18</sub> H <sub>23</sub> NO <sub>3</sub> ·HCl)