Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

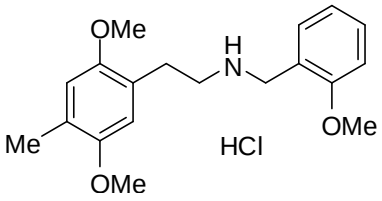
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

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

Report ID: D1010.2021.03

Chemical Formula: $C_{19}H_{25}NO_3$.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)	97.8 ± 0.7%

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

Expiration of certification: The property values are valid till 2 March 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: Off-white powder 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 reference material is recommended for qualitative analysis only.

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 ºC in a closed container in a dry, dark area.

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.
21 September 2022

This report supersedes any issued prior to 21 September 2022.

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

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 ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

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

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890 or 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	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 = 99.1%, s = 0.2% (10 sub samples in duplicate, April 2014)
	Re-analysis:	Mean = 99.2%, s = 0.06% (5 sub samples in duplicate, April 2015)
	Re-analysis:	Mean = 99.0%, s = 0.07% (5 sub samples in duplicate, March 2016)
	Re-analysis:	Mean = 98.7%, s = 0.2% (5 sub samples in duplicate, March 2017)
	Re-analysis:	Mean = 99.6%, s = 0.05% (5 sub samples in duplicate, March 2021)
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)
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.

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	190 °C (1 min), 6 °C/min to 300 °C (5.67 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °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 μL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	316.2 ($M+H^+$) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate/diethyl amine (16/4/0.5) Single spot observed, R_f = 0.2
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2925, 2828, 2781, 2680, 2641, 2484, 1601, 1587, 1509, 1496, 1466, 1457, 1439, 1402, 1257, 1215, 1051, 1034, 770 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance 400
	Field strength:	400 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 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, *J* = 5.6 Hz), 6.77 (1H, s), 6.82 (1H, s), 6.99 (1H, dt, *J* = 1.2, 7.2 Hz), 7.08 (1H, d, *J* = 8 Hz), 7.40 (1H, m), 7.51 (1H, dd, *J* = 1.6, 7.6 Hz), 9.31 (2H, s) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	101 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 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, 157.5ppm
Melting point:		159-164 °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 $C_{13}H_{17}NO_2$.HCl+ 1.4% water)