



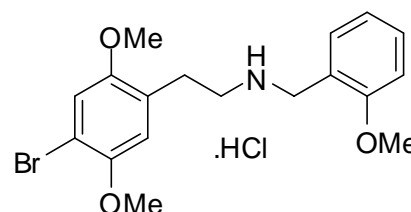
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D995: 2-(4-Bromo-2,5-dimethoxyphenyl)-N-[(2-methoxybenzyl)]ethylamine hydrochloride

Report ID: D995.2023.01

Chemical Formula: C₁₉H₂₂BrNO₃.HCl

Molecular Weight: 416.7 g/mol (HCl), 380.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
13-D-11	1026511-90-9 (base)	98.3 ± 1.4%

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

IUPAC name: 2-(4-bromo-2,5-dimethoxyphenyl)-N-(2-methoxybenzyl)ethanamine

Expiration of certification: The property values are valid till 14 June 2028, 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 solid 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%.

Stability: This material has demonstrated stability over a minimum period of three 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 HPLC with UV detection 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.



Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
21 June 2023

This report supersedes any issued prior to 21 June 2023.

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.

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 HPLC with UV detection, thermogravimetric analysis, Karl Fischer analysis and ¹H 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_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	X-bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 $^{\circ}\text{C}$
	Mobile Phase:	Acetonitrile/MilliQ water (50:50) The aqueous phase was buffered at pH 10.8 using 20 mM NH_4OAc and 25% aqueous ammonia.
	Flow rate:	1.0 mL/min
	Detector:	Waters PDA 2998 operating at 210 nm
	Relative mass fraction of the main component	
	Initial analysis:	Mean = 98.6%, s = 0.04% (10 sub samples in duplicate, June 2013)
	Re-analysis:	Mean = 98.7%, s = 0.06% (5 sub samples in duplicate, May 2014)
	Re-analysis:	Mean = 98.7%, s = 0.05% (5 sub samples in duplicate, June 2015)
	Re-analysis:	Mean = 98.6%, s = 0.02% (5 sub samples in duplicate, May 2016)
	Re-analysis:	Mean = 98.6%, s = 0.01% (5 sub samples in duplicate, April 2019)
	Re-analysis:	Mean = 98.5%, s = 0.03% (5 sub samples in duplicate, June 2023)
Thermogravimetric analysis:	Volatile content 0.3% and non volatile residue < 0.2% mass fraction (June 2013)	
Karl Fischer analysis:	Moisture content < 0.2% mass fraction (June 2013) Moisture content < 0.2% mass fraction (June 2014) Moisture content < 0.1% mass fraction (May 2015) Moisture content < 0.2% mass fraction (May 2016) Moisture content \leq 0.2% mass fraction (April 2019 and June 2023)	

Spectroscopic and other characterisation data

LC-MS:	Instrument:	Waters 2695 (HPLC)/Micromass Quatro
	Column:	X-Bridge C-18, 100 mm x 2.1 mm I.D. x 3.5 µm
	Column temp:	40 °C
	Solvent system:	2% formic acid [10% v/v], acetonitrile [50% v/v], MilliQ water [40% v/v]
	Flow rate:	1 mL/min
	Sample prep:	1000 µg/g in 2% formic acid [10% v/v], acetonitrile [50% v/v], MilliQ water [40% v/v]
	Injection volume:	30 µL
	Ionisation mode:	Electrospray negative ion
	Capillary voltage:	3.5 kV
	Cone voltage:	5 V
	Source temp:	130 °C
	Cone gas flow rate:	27 L/hr
		Desolvation gas temperature: 350 °C
		Desolvation gas flow rate: 740 L/hr
	The retention time of 25B-NBOMe is reported along with the major peak in the mass spectrum. The latter is reported as a mass/charge ratio.	
	10.85 min:	380.1 (M+H ⁺); 382.1 (M+H ⁺) <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:	Diethyl ether
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Hexane/ethyl acetate/diethyl amine (15/3/0.2) Single spot observed, R _f = 0.15. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm ⁻¹ , KBr powder
	Peaks:	3069, 3008, 2943, 2760, 2457, 2430, 1606, 1592, 1498, 1463, 1437, 1387, 1250, 1219, 1027, 870, 801, 752 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	MeOH- <i>d</i> ₄ (3.31 ppm)
	Spectral data:	δ 3.02 (2H, m), 3.22 (2H, m), 3.78 (3H, s), 3.83 (3H, s), 3.89 (3H, s), 4.25 (2H, s), 6.97 (1H, s), 7.02 (1H, dt, <i>J</i> = 1.5, 7.9 Hz), 7.09 (1H, bd, <i>J</i> = 7.9 Hz), 7.17 (1H, s), 7.39 (1H, dd, <i>J</i> = 1.5, 7.9 Hz), 7.45 (1H, ddd, <i>J</i> = 1.7, 7.6, 9.3 Hz) ppm Diethyl ether estimated at 0.2% mass fraction was observed in the ¹ H NMR.
¹³ C NMR:	Instrument:	Bruker Avance-400
	Field strength:	101 MHz
	Solvent:	MeOH- <i>d</i> ₄ (49.0 ppm)
	Spectral data:	δ 28.3, 47.8, 48.0, 56.2, 56.7, 57.4, 111.5, 112.1, 116.3, 117.2, 120.2, 122.0, 126.0, 132.7, 151.7, 153.2, 159.3 ppm
Melting point:		175-178 °C
Microanalysis:	Found:	C = 52.1%; H = 5.7%; N = 3.4%; Cl = 8.5%; Br = 19.4% (June 2013)
	Calculated:	C = 51.9%; H = 5.6%; N = 3.4%; Cl = 8.5%; Br = 19.2% (Calculated for C ₁₈ H ₂₂ BrNO ₃ .HCl)