Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

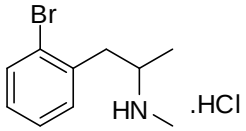
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1037: (±)-2-Bromomethamphetamine hydrochloride

Report ID: D1037.2021.02

Chemical Formula: $C_{10}H_{14}BrN.HCl$

Molecular Weight: 264.6 g/mol (HCl), 228.1 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-37	**23695-11-6 (HCl)** **4302-94-7 (base)**	**98.6 ± 1.9%**

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

IUPAC name: 1-(2-Bromophenyl)-N-methyl-2-propanamine hydrochloride

Expiration of certification: The property values are valid till 22 November 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. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: White powder prepared by synthesis, certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium 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 ºC 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 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 14 December 2021.

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 certified 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 quantitative NMR (qNMR), qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis. The purity value obtained by qNMR was determined using combination of the one proton doublet of doublets at 3.2 ppm and the four proton multiplets at 7.0-7.8 ppm were measured against a certified internal standard of maleic acid.

GC-FID:	Instrument:	Agilent 8890 or Varian CP-3800 or Agilent 6890
	Column:	HP - 5, VF-1MS or HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main componentas the free base:	
	Initial analysis:	Mean = 99.1%, s = 0.08% (10 sub samples in duplicate, October 2014)
	Re-analysis:	Mean = 99.0%, s = 0.10% (5 sub samples in duplicate, October 2015)
	Re-analysis:	Mean = 99.0%, s = 0.13% (5 sub samples in duplicate, October 2016)
	Re-analysis:	Mean = 99.0%, s = 0.13% (5 sub samples in duplicate, November 2021)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (September 2015, 2016 and November 2021)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (3.22 ppm) = 99.5%, s = 0.6% (3 sub samples, October 2014)
	Initial analysis:	Mean (7.0-7.8 ppm) = 100.0%, s = 0.7% (3 sub samples, October 2014)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (1 min), 30 °C/min to 300 °C (2 min)
	Injector:	250 °C,
	Split ratio:	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the parent compound 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 (10.2 min):	171 (3), 169 (3), 115 (4), 90 (5), 89 (5) 58 (100), 56 (10) *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:	230.3 ($M^{Br81}+H^+$), 228.2 ($M^{Br79}+H^+$) *m/z*
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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Ethanol, diethyl ether
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/NH_3 (200:3) Single spot observed, R_f = 0.50
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2725, 2460, 1473, 1443, 1389, 1194, 1075, 1045, 1027, 748, 660 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.26 (3H, d, *J* = 6.6 Hz), 2.72 (3H, s), 2.97 (1H, dd, *J* = 8.7, 13.8 Hz), 3.25 (1H, dd, *J* = 6.1, 13.8 Hz), 3.63 (1H, m), 7.23 (1H, m), 7.33-7.39 (2H, m), 7.65 (1H, d, *J* = 7.7 Hz) ppm
		Ethanol estimated at 0.1% mass fraction was observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D_2O
	Spectral data:	δ 14.8, 30.0, 38.9, 55.0, 124.2, 128.0, 129.4, 131.9, 133.1, 135.0 ppm
Melting point:		160-163 °C
Microanalysis:	Found:	C = 45.4%; H = 5.6%; N = 5.3%, Cl = 13.3%; Br = 30.2% (October, 2014)
	Calculated:	C = 45.4%; H = 5.7%; N = 5.3%, Cl = 13.4%; Br = 30.2% (Calculated for $C_{10}H_{14}BrN.HCl$)