



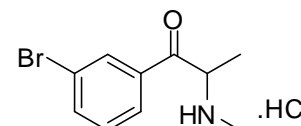
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1035: (\pm)-3-Bromomethcathinone hydrochloride

Report ID: D1035.2022.01

Chemical Formula: C₁₀H₁₂BrNO.HCl

Molecular Weight: 278.6 g/mol (HCl), 242.1 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-35	676487-42-6 (HCl) 486459-02-3 (free base)	97.3 \pm 2.0%

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

IUPAC name: 1-(3-Bromophenyl)-2-(methylamino)-1-propanone hydrochloride (1:1).

Expiration of certification: The property values are valid till 8 July 2027, 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. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

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 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 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 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.
12 July 2022

This report supersedes any issued prior to 12 July 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.

Characterisation Report:

The purity value was obtained by quantitative nuclear magnetic resonance (qNMR). A combination of the three proton singlet at 2.77 ppm, and the aromatic protons at 7.3-8.2 ppm were measured against a certified internal standard of maleic acid.

Supporting evidence is provided by GC-FID, Karl Fischer analysis, headspace GC-MS analysis of occluded solvents, and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1 or HP-5, 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 280 °C (10 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component as the <i>N</i> -acetyl derivative:	
	Initial analysis:	Mean = 99.3%, s = 0.01% (10 sub samples in duplicate, November 2014)
	Re-analysis:	Mean = 98.8%, s = 0.14% (7 sub samples in duplicate, October 2015)
	Re-analysis:	Mean = 98.9%, s = 0.24% (5 sub samples in duplicate, February 2017)
	Re-analysis:	Mean = 98.5%, s = 0.43% (5 sub samples in duplicate, February 2018)
	Re-analysis:	Mean = 98.5%, s = 0.27% (5 sub samples in duplicate, July 2022)
Karl Fischer analysis:	Moisture content 0.2% mass fraction (November 2014)	
	Moisture content 0.4% mass fraction (October 2015)	
	Moisture content 0.4% mass fraction (September 2016)	
	Moisture content 0.3% mass fraction (January 2018)	
	Moisture content 0.3% mass fraction (July 2022)	
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (2.77 ppm) = 98.6%, s = 0.1% (4 sub samples, November 2014)
	Initial analysis:	Mean (7.3-8.2 ppm) = 98.8%, s = 0.2% (4 sub samples, November 2014)

Spectroscopic and other characterisation data

GC-MS:	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 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 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 (11.4 min): 185 (5), 183 (5), 157 (11), 155 (11), 58 (100) <i>m/z</i>
ESI-MS:	Instrument: Micromass Quatro LC Micro Operation: Positive/Negative ion mode, direct infusion at 10 µL/min Ionisation: ESI spray voltage at 3.5 kV positive ion EM voltage: 650 V Cone voltage: 3 V Peak: 244.2 ($M^{Br81}+H^+$), 242.3 ($M^{Br79}+H^+$) <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: Ethanol, diethyl ether
TLC:	Conditions: Kieselgel 60F ₂₅₄ . Methanol/ammonia (200:3) Single spot observed, $R_f = 0.7$
IR:	Instrument: Bruker Alpha Platinum ATR Range: 4000-400 cm^{-1} , neat Peaks: 2902, 2792, 2702, 2439, 1692, 1564, 1413, 1246, 978, 899, 799, 728, 694, 670, 442 cm^{-1}
¹ H NMR:	Instrument: Bruker Avance III-500 Field strength: 500 MHz Solvent: D ₂ O (4.79 ppm) Spectral data: δ 1.57 (3H, d, $J = 7.3$ Hz), 2.78 (3H, s), 5.04 (1H, q, $J = 7.4$ Hz), 7.48 (1H, t, $J = 8.0$ Hz), 7.88 (1H, m), 7.94 (1H, m), 8.15 (1H, m) ppm
¹³ C NMR:	Instrument: Bruker Avance III-500 Field strength: 126 MHz Solvent: D ₂ O Spectral data: δ 15.0, 30.8, 59.6, 122.7, 127.6, 130.9, 131.5, 134.0, 137.9, 196.3 ppm
Melting point:	205-208 °C
Microanalysis:	Found: C = 43.1%; H = 4.6%; N = 5.0%; Br = 28.9%; Cl = 12.8% (November, 2014) Calculated: C = 43.1%; H = 4.7%; N = 5.0%; Br = 28.7%; Cl = 12.7% (Calculated for C ₁₀ H ₁₂ BrNO.HCl)