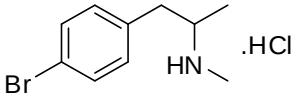
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D1028.2016.01

Compound Name: **(±)-4-Bromomethamphetamine hydrochloride**
Description: White solid
Collection Number: D1028
Batch Number: 14-D-28
Chemical Formula: $C_{10}H_{14}BrN.HCl$
Molecular Weight: 264.6 (HCl), 228.1 (base)
CAS Number: 30651-67-3
Release date: 13th October 2014
Structure:



Synonyms: (±)-4-Bromo-*N*-α-dimethyl-benzeneethanamine hydrochloride
(±)-*p*-Bromo-*N*,α-dimethyl-phenylethylamine hydrochloride

Purity (mass fraction): 99.6 ± 2.5% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, Karl Fischer analysis and ^{1}H NMR. The purity estimate by qNMR was obtained using a combination of the three-proton doublet at 1.23 ppm, the doublet of doublets at 3.00 ppm and the two-proton doublet at 7.53 ppm against a certified internal standard of maleic acid. Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800	
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 ºC/min to 300 ºC (3 min)	
	Injector:	180 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area of main component as the free base:		
	Initial analysis:	Mean = 99.7%, s = 0.05% (7 sub samples in duplicate, August 2014)	
	Re-analysis:	Mean = 99.8%, s = 0.04% (7 sub samples in duplicate, July 2015)	
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate (June 2016)	
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (September 2014) Moisture content 0.2% mass fraction (July 2015) Moisture content < 0.1% mass fraction (June 2016)	
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 (1.23 ppm) = 99.2%, s = 0.2% (5 sub samples, August 2014)	
	Initial analysis:	Mean (3.00 ppm) = 99.9%, s = 0.2% (5 sub samples, August 2014)	
	Initial analysis:	Mean (7.53 ppm) = 99.6%, s = 0.3% (5 sub samples, August 2014)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973	
	Column:	TG1-MS, 30 m x 0.25 mm I.D. x 0.25 µm	
	Program:	60 °C (1 min), 10 °C/min 100 °C, 15 °C/min to 300 °C (3 min)	
	Injector:	180 °C	Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 20/1
	The retention time of the free base is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.		
	Free base (10.5 min): 214 (1), 212 (1), 171 (5), 169 (5), 90 (7), 89 (7), 58 (100) 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:	2 V	
	Peak:	228 and 230 ($M+H^+$) m/z	
IR:	Instrument:	Bruker Alpha FT-IR	
	Range:	4000-400 cm^{-1}, neat	
	Peaks:	2963, 2723, 1716, 1475, 1070, 1012, 797 cm^{-1}	
^{1}H NMR:	Instrument:	Bruker Avance III-500	
	Field strength:	500 MHz	Solvent: D_2O (4.79 ppm)
	Spectral data:	δ 1.23 (3H, d, *J* = 6.6 Hz), 2.68 (3H, s), 2.82 (1H, dd, *J* = 8.2, 13.9 Hz), 3.02 (1H, dd, *J* = 6.1, 13.9 Hz), 3.49 (1H, m), 7.18 (2H, d, *J* = 8.8 Hz), 7.53 (2H, d, *J* = 8.4 Hz) ppm	
^{13}C NMR:	Instrument:	Bruker Avance III-500	
	Field strength:	126 MHz	Solvent: D_2O
	Spectral data:	δ 14.7, 29.9, 38.1, 56.1, 120.6, 131.3, 131.8, 134.8 ppm	
Melting point:		155-158 ºC	
Microanalysis:		Found: C = 45.6%; H = 5.7%; N = 5.3%; Br = 30.6; Cl = 13.3; (September, 2014) Calc: C = 45.4%; H = 5.7%; N = 5.3%; Br = 30.2; Cl = 13.4; (Calculated for $C_{10}H_{14}BrN.HCl$)	

Expiration of certification
The property values are valid till 15th June 2019, i.e. three 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 long-term stability of the compound in solution has not been examined.

This material has been given a shelf life of three years from the date of re-certification. 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.

In the absence of stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last 10 years.

Homogeneity assessment
The homogeneity of the material was assessed using purity assay by GC-FID on seven 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.

Metrological traceability
The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile content is directly traceable to mass through use of Karl Fischer analysis. 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).

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.

Intended use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 22 June, 2016.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 22nd June 2016.