Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

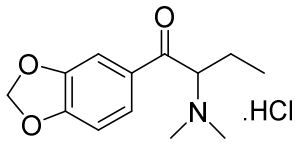
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1027: (±)-N-Methylbutylone hydrochloride

Report ID: D1027.2024.01

Chemical Formula: $C_{13}H_{17}NO_3$.HCl

Molecular Weight: 271.7 g/mol (HCl), 235.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-27	17763-12-1	99.8 ± 0.4%

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

IUPAC name: (±)-1-(1,3-Benzodioxol-5-yl)-2-(dimethylamino)-1-butanone hydrochloride

Expiration of certification: The property values are valid till 20 August 2034, ten 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: White powder sourced from an external supplier and 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 all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: In the absence of long term 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 ten 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.
28 August 2024

This report supersedes any issued prior to 28 August 2024

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 GC-FID, thermogravimetric analysis, Karl Fischer analysis, and ^{1}H NMR spectroscopy. Supporting evidence is provided by elemental microanalysis. 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.

GC-FID:	Instrument:	Varian CP-3800 OR Agilent 8890
	Column:	VF-1MS or HP-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	160 °C (15 min), 30 °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 = 99.9%, s = 0.01% (10 sub samples in duplicate, August 2014)
	Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, August 2015)
	Re-analysis:	Mean = 99.7%, s = 0.04% (7 sub samples in duplicate, August 2024)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (September 2014, July 2015, and August 2024)
Thermogravimetric analysis:		Volatiles content ≤ 0.1% and non-volatile residue < 0.2% mass fraction (August 2024)
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 (0.77 ppm) = 100.1%, s = 0.5% (5 sub samples, August 2014)
	Initial analysis:	Mean (6.09 ppm) = 100.2%, s = 0.5% (5 sub samples, August 2014)
	Initial analysis:	Mean (7.66 ppm) = 100.0%, s = 0.5% (5 sub samples, August 2014)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	160 °C (15 min), 30 °C/min to 300 °C (5 min)
	Injector:	180 °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 spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Free base (12.0 min):	149 (7), 121 (3), 86 (100), 71 (8), 65 (4) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Direct infusion at 10 µL/min
	Ionisation mode:	Electrospray positive ion
	Interface voltage:	3.5 kV
	Peak:	236 ($M+H^+$) *m/z*
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 0.78 (3H, t, *J* = 7.6 Hz), 2.08 (1H, m), 2.16 (1H, m), 2.86 (3H, s), 2.98 (3H, s), 5.11 (1H, dd, *J* = 3.9, 6.3 Hz), 6.09 (2H, m), 7.43 (1H, d, *J* = 8.3 Hz), 7.67 (1H, dd, *J* = 1.9, 8.3 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D_2O
	Spectral data:	δ 7.0, 22.1, 40.1, 43.6, 70.0, 102.7, 107.7, 108.5, 126.7, 128.0, 148.4, 153.8, 194.6 ppm
Melting point:		220-222 °C
Microanalysis:	Found:	C = 57.6%; H = 6.8%; N = 5.2%, Cl = 13.0 (September, 2014)
	Calculated:	C = 57.5%; H = 6.7%; N = 5.2%, Cl = 13.1 (Calculated for $C_{13}H_{17}NO_3$.HCl)