Australian Government
Department of Industry, Science and Resources

National Measurement Institute

CIPM MRA

NATA
WORLD RECOGNISED ACCREDITATION

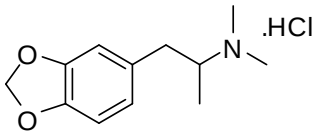
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D503: (±)-N,N-Dimethyl-3,4-methylenedioxyamphetamine hydrochloride

Report ID: D503.2020.03

Chemical Formula: $C_{12}H_{17}NO_2$.HCl

Molecular Weight: 243.7 g/mol (HCl salt), 207.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
97-000772	74341-79-0 (HCl) 74698-50-3 (base)	99.5 ± 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)-N,N-dimethyl-2-propanamine hydrochloride,

Expiration of certification: The property values are valid till 10 June 2030, i.e. 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 crystals prepared by synthesis or sourced from an external supplier, 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 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 measurement uncertainty at the 95% coverage 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 five 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 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.
14 September 2022

This report supersedes any issued prior to 14 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

CIPM MRA notice: This certificate is consistent with the capabilities that are included in Appendix C of the CIPM MRA drawn up by the CIPM. Under the CIPM MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C. The "CIPM MRA Logo" and this statement attest only to the measurement(s) applied for determining the certified values on the certificate (for details see http://www.bipm.org).

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. 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 elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (4 min)
	Injector:	180 °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 = 99.8%, s = 0.01% (5 sub samples in duplicate, January 2004)
	Re-analysis:	Mean = 99.7%, s = 0.04% (5 sub samples in duplicate, October 2007)
GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	130 °C (0.5 min), 8 °C/min to 240 °C (2min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °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 = 99.7%, s = 0.003% (5 sub samples in duplicate, October 2010)
	Re-analysis:	Mean = 99.7%, s = 0.04% (5 sub samples in duplicate, August 2015)
	Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, June 2020)
Thermogravimetric analysis:		Volatile content < 0.1% and non-volatile residue < 0.2% mass fraction (October 2004)
Karl Fischer analysis:		Moisture content 0.3% mass fraction (2 sub samples, October 2007) Moisture content 0.2% mass fraction (2 sub samples, October 2010) Moisture content < 0.1% mass fraction (2 sub samples, August 2015) Moisture content 0.1% mass fraction (2 sub samples, June 2020)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	Zebron ZB-5, 30 m x 0.25 mm I.D. x 0.30µm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (2 min)
	Injector:	180 °C
	Transfer line temp:	340 °C
	Carrier:	Helium, 1.3 mL/min
	Split ratio:	20/1
	The retention time of the free base is reported along with the major peaks in the mass spectra. The latter are reported as mass to charge ratios and (in brackets) as a percentage relative to the intensity of the base peak.	
	Free base (11.8 min):	207 (M^+, <1), 135 (4), 77 (4), 73 (5), 72 (100) *m/z*.
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3009, 2951, 2899, 2793, 2615, 2479, 1846, 1605, 1485, 1366, 1244, 1190, 1040, 931, 808, 638, 575 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.24 (3H, d, *J* = 6.7 Hz), 2.80 (1H, dd, *J* = 9.1, 13.7 Hz), 2.84 (6H, s), 3.02 (1H, dd, *J* = 6.0, 13.7 Hz), 3.64 (1H, m), 5.95 (2H, s), 6.79 (1H, dd, *J* = 1.5, 7.9 Hz), 6.85 (1H, d, *J* = 1.5 Hz), 6.87 (1H, d, *J* = 7.9 Hz) ppm
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	D_2O
	Spectral data:	δ 11.7, 36.5, 37.1, 40.5, 63.4, 101.2, 108.8, 109.5, 122.7, 129.3, 146.5, 147.7 ppm
Melting Point		167-170 °C
Microanalysis:	Found:	C = 59.3%, H = 7.4%, N = 5.7% (February 2004)
	Found:	C = 59.3%, H = 7.6%, N = 5.8% (September 2005)
	Calculated:	C = 59.1%, H = 7.4%, N = 5.8% (Calculated for $C_{12}H_{17}NO_2$.HCl)