Australian Government
Department of Industry, Science and Resources

National Measurement Institute

CIPM MRA

NATA
WORLD RECOGNISED ACCREDITATION

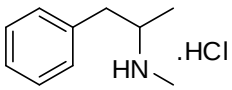
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D816g: (±)-Methamphetamine hydrochloride

Report ID: D816g.2021.03 (Bottled 210413)

Chemical Formula: $C_{10}H_{15}N.HCl$

Molecular Weight: 185.7 g/mol (HCl), 149.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-12	**300-42-5**	**99.8 ± 0.3%**

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

IUPAC name: *N*-Methyl-1-phenyl-2-propanamine hydrochloride (1:1)

Expiration of certification: The property values are valid till 8 April 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.

Description: White solid prepared by synthesis, 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%. 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 five years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from long term 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.
15 September 2022

This report supersedes any issued prior to 15 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 purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include 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

The purity value by qNMR was obtained using the three-proton doublet at 1.27 ppm measured against a certified internal standard of maleic acid.

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

GC-FID:	Instrument:	Agilent 6890/Varian CP-3800
	Column:	HP-1/VF-1MS, 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:	180 °C
	Detector Temp:	320 °
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the free base:	
	Initial analysis:	Mean = 99.8%, s = 0.03% (10 sub samples in duplicate, April 2014)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, April 2015)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, October 2016)
	Re-analysis:	Mean = 99.98%, s = 0.024% (5 sub samples in duplicate, April 2021)
Thermogravimetric analysis:		Non-volatile residue < 0.2% mass fraction (April 2014). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (May 2014, April 2015, October 2016 and April 2021)
qNMR:	Instrument:	Bruker Avance-III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.80 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (1.27 ppm) = 99.8%, s = 0.3% (5 sub samples, May 2014)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 250 °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 (8.0 min):	134 (3), 91 (14), 58 (100) *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
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	No solvents detected
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/ammonia (100/0.5) Single spot observed, R_f = 0.4. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3180, 2967, 2797, 2739, 2457, 2056, 1954, 1883, 1811, 1603, 1488, 1455, 1191, 1081, 1060, 916, 749, 701, 462 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.80 ppm)
	Spectral data:	δ 1.29 (3H, d, *J* = 6.6 Hz), 2.72 (3H, s), 2.92 (1H, dd, *J* = 8.0, 13.8 Hz), 3.09 (1H, dd, *J* = 6.3, 13.8 Hz), 3.56 (1H, m), 7.31-7.48 (5H, m) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-600
	Field strength:	151 MHz
	Solvent:	MeOH-*d*$_4$ (49.0 ppm)
	Spectral data:	δ 14.7, 30.0, 39.2, 56.8, 127.4, 129.0, 129.5, 136.3 ppm
Melting point:		135-138 °C
Microanalysis:	Found:	C = 64.6%; H = 8.7%; N = 7.5%; Cl = 19.2% (April 2014)
	Calculated:	C = 64.7%; H = 8.7%; N = 7.5%; Cl = 19.1% (Calculated for $C_{10}H_{15}N.HCl$)