



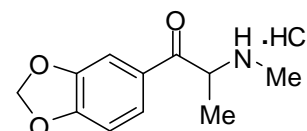
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D942: 3,4-Methylenedioxyamphetamine hydrochloride

Report ID: D942.2020.03

Chemical Formula: C<sub>11</sub>H<sub>13</sub>NO<sub>3</sub>.HCl

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



### Certified value (mass fraction of the free base)

Batch No.	CAS No.	Purity (mass fraction)
09-D-14	186028-79-5	84.4% ± 1.4%

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

### Reference value (mass fraction as the hydrochloride salt)

Batch No.	CAS No.	Purity (mass fraction)
09-D-14	186028-80-8	99.3%

The mass fraction of hydrogen chloride has not been measured and therefore the 1:1 stoichiometry cannot be confirmed.

**IUPAC name:** 1-(1,3-Benzodioxol-5-yl)-2-(methylamino)-1-propanone hydrochloride

**Expiration of certification:** The property values are valid till 12 August 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:** Off-white powder 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 °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. 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 ten 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 HPLC 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.

**Caution:** 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.



Dr Stephen R. Davies,  
Team Leader,  
Chemical Reference Materials, NMI.  
20 September 2022

This report supersedes any issued prior to 20 September 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.

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**Characterisation Report:**

The mass fraction of methamphetamine free base was obtained from a combination of quantitative nuclear magnetic resonance (qNMR) and HPLC-UV analysis. The purity value by qNMR was obtained using the three-proton singlet at 2.79 ppm measured against a certified internal standard of glycine.

Supporting evidence is provided by thermogravimetric analysis, Karl Fischer analysis, <sup>1</sup>H NMR spectroscopy, and elemental microanalysis.

QNMNR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D <sub>2</sub> O (4.79 ppm)
	Internal standard:	Glycine (100% m/m)
	Initial analysis:	Mean (2.79 ppm) = 84.4%, s = 0.06% (4 sub samples, August 2020)
HPLC:	Instrument:	Shimadzu or Waters HPLC
	Column:	Ascentis C-18, 2.7µm (4.6 mm I.D x 150 mm)
	Mobile Phase:	MeOH/Milli-Q water (35/65) 0.05% TFA was present in both aqueous and organic phases
	Flow Rate:	0.4 mL/min
	Detector:	Shimadzu ELSD-LTII or Waters 2424 ELSD
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.78%, s = 0.04% (5 sub samples in duplicate, July 2010)
	Re-analysis:	Mean = 99.50%, s = 0.1% (5 sub samples in duplicate, July 2011)
	Re-analysis:	Mean = 99.95%, s = 0.04% (5 sub samples in duplicate, July 2012)
	Re-analysis:	Mean = 99.85%, s = 0.09% (5 sub samples in duplicate, June 2015)
HPLC:	Instrument:	Waters Model 600E pump, 717 plus Autosampler
	Column:	Alltima C-18, 5 µm (4.6 mm I.D x 150 mm)
	Mobile Phase:	Acetonitrile/20mM NH <sub>4</sub> OAc/ NH <sub>4</sub> OH pH 9 buffer, 40/60
	Flow Rate:	1.0 mL/min
	Detector:	PDA max plot
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.55%, s = 0.1% (10 sub samples in duplicate, August 2009)
HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Ascentis C-18, 2.7 µm (4.6 mm I.D x 150 mm)
	Mobile Phase:	A = MilliQ water; B = Methanol 0.05% TFA was present in both aqueous and organic phases 0-15 min 30% B; 15-20 min 30-80% B; 20-25 min 80%B; 25-27 min 80-35%B.
	Flow Rate:	1.0 mL/min
	Detector:	PDA at 283 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.03% (5 sub samples in duplicate, May 2020)
Karl Fischer analysis:		Moisture content < 0.2 % mass fraction (May 2009 & 2010, August 2011, July 2012 and June 2015, May 2020)
Thermogravimetric analysis:		Volatile content not determined due to volatility of the material.

**Spectroscopic and other characterisation data:**

GC-MS:	Instrument:	Agilent 6890
	Column:	VF-1ms, 15 m × 0.25 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 200 °C (2 min), 40 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	300 °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 (11.2 min):	149 (10), 121 (5), 65 (6), 58 (100) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 µL/min
	Ionisation:	ESI spray voltage at 3.2 kV positive ion
	EM voltage:	650 V
	Cone voltage:	25 V
	Peak:	208 (M+H <sup>+</sup> , 100) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . MeOH/NH <sub>3</sub> (9/1) Single spot observed, R <sub>f</sub> = 0.8. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm <sup>-1</sup> , KBr powder
	Peaks:	2909, 2797, 2741, 1678, 1603, 1503, 1453, 1424, 1349, 1299, 1262, 1121, 1091, 1038, 992, 930, 887, 741 cm <sup>-1</sup>
<sup>1</sup> H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	DMSO- <i>d</i> <sub>6</sub> (2.50 ppm)
	Spectral data:	δ 1.44 (3H, d, <i>J</i> = 7.2 Hz), 2.55 (3H, s), 5.09 (1H, q, <i>J</i> = 7.1 Hz), 6.18 (2H, s), 7.12 (1H, d, <i>J</i> = 8.2 Hz), 7.53 (1H, d, <i>J</i> = 1.6 Hz), 7.69 (1H, dd, <i>J</i> = 1.8, 8.2 Hz), 9.49 (2H, br s) ppm
<sup>13</sup> C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	100 MHz
	Solvent:	DMSO- <i>d</i> <sub>6</sub> (39.52 ppm)
	Spectral data:	δ 15.7, 30.6, 57.9, 102.5, 107.9, 108.5, 125.8, 127.4, 148.2, 152.7, 194.3 ppm
Melting point:		244-246 °C
Microanalysis:	Found:	C = 54.4 %; H = 5.9 %; N = 5.7 % (April 2009)
	Calculated:	C = 54.2 %; H = 5.8 %; N = 5.8 % (Calculated for C <sub>11</sub> H <sub>13</sub> NO <sub>3</sub> .HCl)