



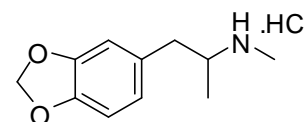
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D792f: (\pm)-3,4-Methylenedioxyamphetamine hydrochloride

Report ID: D792f.2021.03 (Bottled 200707)

Chemical Formula: $C_{11}H_{15}NO_2 \cdot HCl$

Molecular Weight: 229.7 g/mol (HCl), 193.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
20-D-01	64057-70-1 (HCl) 42542-10-9 (base)	99.8 \pm 0.3%

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*-methyl-2-propanamine hydrochloride

Expiration of certification: The property values are valid till 14 April 2024, 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.

Description: White powder prepared by synthesis, and 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. 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 three 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 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 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 ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\% - I_{\text{ORG}}) \times (100\% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qNMR and elemental microanalysis. The purity value by qNMR was obtained using the three-proton doublet at 1.29 ppm, the one-proton multiplet at 3.49 ppm, and the two-proton singlet at 5.97 ppm measured against a certified internal standard of sodium acetate.

GC-FID: Instrument: Agilent 7890
 Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 120 °C (1 min), 10 °C/min to 220 °C, 20 °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 = 100.0%, s = 0.04% (10 sub samples in duplicate, May 2020)
 Re-analysis: Mean = 100.0 %, s = 0.002% (5 sub samples in duplicate, April 2021)

Karl Fischer analysis: Moisture content ≤ 0.1% mass fraction (May 2020)
 Moisture content 0.2% mass fraction (March 2021)

Thermogravimetric analysis: Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (May2020)

qNMR: Instrument: Bruker Avance-III-500
 Field strength: 500 MHz
 Solvent: D₂O (4.79 ppm)
 Internal standard: Sodium acetate (60.1% mass fraction)

Initial analysis: Mean (1.29 ppm) = 100.1%, s = 0.1% (5 sub samples, May 2020)
 Initial analysis: Mean (3.49 ppm) = 99.97%, s = 0.3% (5 sub samples, May 2020)
 Initial analysis: Mean (5.97 ppm) = 100.3%, s = 0.2% (5 sub samples, May 2020)

Spectroscopic and other characterisation data

GC-MS:	Instrument: Agilent 6890/5973 Column: DB-5MS, 30 m x 0.25 mm I.D. x 0.25 µm Program: 120 °C (1 min), 10 °C/min to 220 °C, 20 °C/min to 300 °C (3 min) Injector: 250 °C Split ratio: 20/1 Transfer line temp: 280 °C Carrier: Helium Scan range: 50-550 <i>m/z</i>
	The retention time of the free base is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak. Parent (7.2 min): 136 (5), 135 (9), 105 (2), 77 (6), 58 (100), 56 (4), 51 (4) <i>m/z</i>
ESI-MS:	Instrument: Micromass Quatro LC Micro Operation: Positive ion mode, direct infusion at 10 µL/min Ionisation: ESI spray voltage at 2.5 kV positive ion EM voltage: 650 V Cone voltage: 20 V Peak: 194.1 (M+H ⁺) <i>m/z</i>
TLC:	Conditions: Kieselgel 60F ₂₅₄ . TBME/Diethyl ether/triethylamine (45:45:10) Single spot observed, R _f = 0.2. Visualisation with UV at 254 nm.
IR:	Instrument: Bruker Alpha Platinum ATR Range: 4000-400 cm ⁻¹ , neat Peaks: 2946, 2831, 2765, 2710, 2457, 1488, 1441, 1244, 1030, 930, 797 cm ⁻¹
¹ H NMR:	Instrument: Bruker Avance III-500 Field strength: 500 MHz Solvent: D ₂ O (4.79 ppm) Spectral data: δ 1.28 (3H, d, <i>J</i> = 6.6 Hz), 2.71 (3H, s), 2.84 (2H, dd, <i>J</i> = 7.7, 14.0 Hz), 3.05 (1H, dd, <i>J</i> = 6.6, 14.0 Hz), 3.49 (1H, m), 5.97 (2H, s), 6.79 (1H, dd, <i>J</i> = 1.6, 7.9 Hz), 6.85 (1H, d, <i>J</i> = 1.6 Hz), 6.89 (1H, d, <i>J</i> = 8.0 Hz) ppm Isopropanol estimated at 0.05% mass fraction was observed in the ¹ H NMR
¹³ C NMR:	Instrument: Bruker Avance III-500 Field strength: 126 MHz Solvent: D ₂ O Spectral data: δ 14.8, 29.9, 38.4, 56.5, 101.1, 108.7, 109.7, 122.8, 129.4, 146.3, 147.5 ppm
Melting point:	150-152 °C
Microanalysis:	Found: C = 57.6%; H = 7.0%; N = 6.0% (May, 2020) Calculated: C = 57.5%; H = 7.0%; N = 6.1% (Calculated for C ₁₁ H ₁₅ NO ₂ .HCl)