



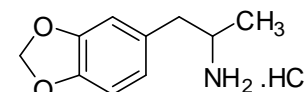
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D842c: (\pm)-3, 4-Methylenedioxyamphetamine hydrochloride

Report ID: D842c.2022.01

Chemical Formula: C₁₀H₁₃NO₂.HCl

Molecular Weight: 215.7 g/mol (HCl) 179.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
13-D-31	6292-91-7 (HCl)	99.3 \pm 0.9%

The uncertainty is stated at the 95% confidence limit.

IUPAC name: 1-(1,3-Benzodioxol-5-yl)-2-propanaminium chloride

Expiration of certification: The property values are valid till 20 July 2030, i.e. eight 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 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 °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 five years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from short term 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.

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.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
24 August 2022

This report supersedes any issued prior to 24 August 2022.

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

Legal notice: Neither NMIA as a representative of the Commonwealth of Australia, nor any person acting on NMIA's behalf, assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this document.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified 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 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

The purity value by QNMR was obtained using a combination of the three proton doublet at 1.30 ppm, the one proton multiplet at 3.57 and the two proton singlet at 5.97 ppm against a certified internal standard of sodium acetate.

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

GC-FID:	Instrument:	Varian CP-3800 or Agilent 7890
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 200 °C, 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 = 99.6%, s = 0.03% (10 sub samples in duplicate, December 2013)
	Re-analysis:	Mean = 99.6%, s = 0.04% (7 sub samples in duplicate, November 2014)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, October 2017)
	Re-analysis:	Mean = 99.7%, s = 0.03% (5 sub samples in duplicate, July 2022)

Thermogravimetric analysis: Non-volatile residue < 0.2% mass fraction (January 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 (January, November 2014, August 2017 and June 2022)

QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O (4.70 ppm)
	Internal standard:	Sodium acetate (60.1% mass fraction)
	Initial analysis:	Mean (1.30 ppm) = 98.8%, s = 0.1% (5 sub samples, February 2014)
	Initial analysis:	Mean (3.57 ppm) = 99.0%, s = 0.1% (5 sub samples, February 2014)
	Initial analysis:	Mean (5.97 ppm) = 99.4%, s = 0.1% (5 sub samples, February 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 Injector: 180 °C Transfer line temp: 280 °C Carrier: Helium, 1.0 mL/min Split ratio: 20/1
	The retention times 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 (11.8 min): 179 (M ⁺ , 2), 136 (48), 135 (21), 77 (12), 44 (100) <i>m/z</i>
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: Diethyl ether and isopropanol
TLC:	Conditions: Kieselgel 60F ₂₅₄ . MeOH/NH ₃ (200/3) 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: 2822, 2941, 2710, 2604, 2037, 1611, 1499, 1443, 1252, 1196, 1098, 1041, 937, 805, 776, 633, 605 cm ⁻¹
¹ H NMR:	Instrument: Bruker Ascend-500 Field strength: 500 MHz Solvent: MeOH-d ₄ (3.31 ppm) Spectral data: δ 1.26 (3H, d, <i>J</i> = 6.7 Hz), 2.74 (1H, dd, <i>J</i> = 8.1, 13.7 Hz), 2.89 (1H, dd, <i>J</i> = 6.5, 13.7 Hz), 3.47 (1H, m), 5.94 (2H, s), 6.72 (1H, dd, <i>J</i> = 1.6, 7.9 Hz), 6.77 (1H, d, <i>J</i> = 1.5 Hz), 6.80 (1H, d, <i>J</i> = 7.9 Hz) ppm Diethyl ether, isopropanol and acetic acid estimated at a total of 0.1% mass fraction was observed in the ¹ H NMR
¹³ C NMR:	Instrument: Bruker DMX600 Field strength: 150 MHz Solvent: MeOH-d ₄ (49.0 ppm) Spectral data: δ 18.3, 41.4, 50.4, 102.5, 109.5, 110.4, 123.6, 131.0, 148.4, 149.6 ppm
Melting point:	181-183 °C
Microanalysis:	Found: C = 55.8%; H = 6.7%; N = 6.5%; Cl = 16.4% (February, 2014) Calculated: C = 55.7%; H = 6.5%; N = 6.5%; Cl = 16.4% (Calculated for C ₁₀ H ₁₃ NO ₂ .HCl)