



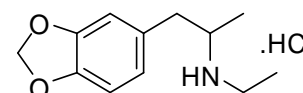
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D739c: (\pm)-N-Ethyl-3,4-methylenedioxyamphetamine hydrochloride

Report ID: D739c.2019.03

Chemical Formula: C₁₂H₁₇NO₂.HCl

Molecular Weight: 243.7 g/mol (HCl), 207.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
11-D-14	74341-78-9 (HCl)	99.8 \pm 0.5%

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-ethyl-2-propanamine hydrochloride (1:1)

Expiration of certification: The property values are valid till 19 June 2024, 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: Off-white solid 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%.

Stability: This material has demonstrated stability over a minimum period of five 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.

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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID: Instrument: Varian CP-3800/Agilent 7890
 Column: VF-1MS/HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 100 °C (1 min), 10 °C/min to 170 °C, 20 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Split ratio: 20/1
 Detector Temp: 320 °C
 Carrier: Helium

Relative mass fraction of the main component as the free base:
 Initial analysis: Mean = 99.9%, s = 0.06% (10 sub samples in duplicate, August 2011)
 Re-analysis: Mean = 99.6%, s = 0.03% (5 sub samples in duplicate, August 2012)
 Re-analysis: Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, June 2013)
 Re-analysis: Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, July 2016)
 Re-analysis: Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, June 2019)

GC-FID: Instrument: Varian CP-3800
 Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 100 °C (1 min), 10 °C/min to 170 °C, 20 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Split ratio: 20/1
 Detector Temp: 320 °C
 Carrier: Helium

Relative mass fraction of the main component as the free base:
 Initial analysis: Mean = 99.8%, s = 0.06% (10 sub samples in duplicate, August 2011)

Thermogravimetric analysis: Non-volatile residue < 0.2% mass fraction (August 2011). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.

Karl Fischer analysis: Moisture content < 0.1% mass fraction (August 2011, July 2013 and May 2019)
 Moisture content < 0.2% mass fraction (August 2012 and July 2016)

Spectroscopic and other characterisation data

GC-MS:	Instrument: Agilent 6890/5973 Column: TG-1MS, 30 m × 0.25 mm I.D. × 0.25 µm Program: 90 °C (1 min), 10 °C/min to 180 °C (7 min), 30 °C/min to 300 °C (3 min) Injector: 200 °C Transfer line temp: 280 °C Carrier: Helium, 1.0 mL/min Split ratio: 30/1 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 (10.2 min): 135 (8), 72 (100), 44 (14) <i>m/z</i>
LC/ESI -MS:	Instrument: Micromass Quatro LC Micro Operation: Positive ion mode, direct infusion at 20 µL/min Ionisation: ESI spray voltage at 3.5 kV positive ion EM voltage: 650 V Cone voltage: 28 V Peak: 208.3 (M ⁺ H ⁺) <i>m/z</i>
HS-GC-MS:	Instrument: Agilent 6890/5973/G1888 Column: DB-624, 30 m × 0.25 mm I.D. × 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: Isopropanol, acetone, diethyl ether
TLC:	Conditions: Kieselgel 60F254. Hexane/ethyl acetate/diethylamine (10/20/1) Single spot observed, R _f = 0.44. Visualisation with UV at 254 nm
IR:	Instrument: Biorad FTS300MX FT-IR Range: 4000-400cm ⁻¹ , KBr Peaks: 3181, 2972, 2789, 2471, 2393, 1603, 1489, 1444, 1246, 1039, 931, 803 cm ⁻¹
¹ H NMR:	Instrument: Bruker Avance-400 Field strength: 400 MHz Solvent: MeOH- <i>d</i> ₄ (3.31 ppm) Spectral data: δ 1.23 (3H, d, <i>J</i> = 6.4 Hz), 1.34 (3H, t, <i>J</i> = 7.2 Hz), 2.65 (1H, dd, <i>J</i> = 9.6, 13.2 Hz), 3.06-3.21 (3H, m), 3.44 (1H, d, <i>J</i> = 1.1 Hz), 5.94 (2H, m), 6.73 (1H, dd, <i>J</i> = 1.6, 8.0 Hz), 6.79 (1H, d, <i>J</i> = 1.6 Hz), 6.80 (1H, d, <i>J</i> = 8.0 Hz) ppm
¹³ C NMR:	Instrument: Bruker Gyro-300 Field strength: 75 MHz Solvent: MeOH- <i>d</i> ₄ (49.0 ppm) Spectral data: δ 11.8, 16.0, 40.0, 41.4, 56.6, 102.5, 109.4, 110.5, 123.7, 130.8, 148.3, 149.6 ppm
Melting point:	199-203 °C
Microanalysis:	Found: C = 59.4%; H = 7.6%; N = 5.8%; Cl = 14.4% (August, 2011) Calculated: C = 59.1%; H = 7.4%; N = 5.8%; Cl = 14.6% (Calculated for C ₁₂ H ₁₇ NO ₂ .HCl)